Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Variable temperature studies of tetrapyridinesilver(I) hexafluorophosphate and tetrapyridinesilver(I) hexafluoroantimonate

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Received 6 June 2024
Accepted 18 November 2024

Edited by L. Suescun, Universidad de la República, Uruguay

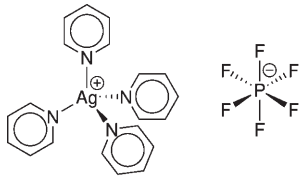
Keywords: crystal structure; tetrapyridinesilver(I) hexafluoroantimonate.

Supporting information: this article has supporting information at journals.iucr.org/e

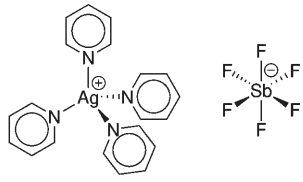
As part of a larger study into phase transitions, the structures of tetrapyridinesilver(I) hexafluorophosphate, $[Ag(C_5H_5N)_4](PF_6)$ or $AgPy_4PF_6$, and tetrapyridinesilver(I) hexafluoroantimonate, $[Ag(C_5H_5N)_4](SbF_6)$ or $AgPy_4SbF_6$, were determined at 300 and 100 K from single-crystal X-ray diffraction. The compounds are isostructural, crystallizing in the space group $I\overline{4}$ with $Z' = 1/4$. Over the temperature range studied no evidence of a phase change was found. The dihedral angles between the pyridine rings are compared with similar cations from the literature and discussed.

1. Chemical context

Barluenga's reagent, IPy_2BF_4, and some of its derivatives have been shown to exhibit phase transitions (Kim *et al.*, 2014; Morgan *et al.*, 2018). This study was recently extended to silver complexes of the form AgL_2X where L is a pyridine-based ligand and X is an anion with a single charge (Fleming, 2021; Thompson *et al.*, 2023).



I



II

While making and studying the parent compound $AgPy_2PF_6$ and the related $AgPy_2SbF_6$, the side-products $AgPy_4PF_6$ (**I**) and $AgPy_4SbF_6$ (**II**) crystallized. Although these have not been previously reported, a number of isostructural species are known including $CuPy_4PF_6$ (Coles *et al.*, 2008), $AgPy_4ClO_4$, $CuPy_4ClO_4$ (Nilsson & Oskarsson, 1981, 1982), $CuPy_4I$ (Al Shamaileh & Al-Far, 2016), $LiPy_4PF_6$ (Jalil *et al.*, 2017) and $LiPy_4ClO_4$ (Harvey *et al.*, 1992). All of these crystallize in the space group $I\overline{4}$ with both the cation and the anion occupying a position on a fourfold rotoinversion axis.

2. Structural commentary

Single-crystal X-ray diffraction data were collected initially at 300 K, before the crystals were cooled at 200 K h^{-1} to 100 K where a similar dataset was collected. In both cases, aside from the expected unit-cell contraction and some peak broadening (presumably caused by strain), there was no evidence of a change in phase over the temperature range studied (Figs. 1

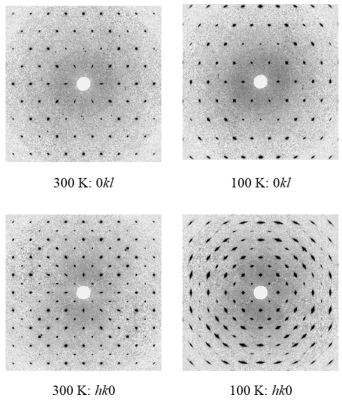


Table 1
Selected geometric parameters (Å, °) for **I** at 300 K.

Ag1—N1^{i}	2.322 (3)	C13—C14	1.356 (6)
Ag1—N1^{ii}	2.322 (3)	C14—C15	1.362 (6)
Ag1—N1^{iii}	2.322 (3)	P1—F1^{iv}	1.572 (2)
Ag1—N1	2.322 (3)	P1—F1^{v}	1.572 (2)
N1—C11	1.318 (5)	P1—F1^{vi}	1.572 (2)
N1—C15	1.331 (4)	P1—F2^{iv}	1.556 (4)
C11—C12	1.354 (6)	P1—F1	1.572 (2)
C12—C13	1.363 (6)	P1—F2	1.556 (4)
N1^{i}—Ag1—N1^{ii}	106.90 (8)	F1^{iv}—P1—F1^{vi}	179.7 (2)
N1^{i}—Ag1—N1^{iii}	114.75 (16)	F1^{v}—P1—F1^{vi}	90.00
N1^{ii}—Ag1—N1^{iii}	106.90 (8)	F1^{iv}—P1—F2^{iv}	90.16 (12)
N1^{i}—Ag1—N1	106.90 (8)	F1^{v}—P1—F2^{iv}	89.84 (12)
N1^{ii}—Ag1—N1	114.75 (16)	F1^{vi}—P1—F2^{iv}	90.16 (12)
N1^{iii}—Ag1—N1	106.90 (8)	F1^{iv}—P1—F1	90.00
Ag1—N1—C11	121.2 (2)	F1^{v}—P1—F1	179.7 (2)
Ag1—N1—C15	120.9 (3)	F1^{vi}—P1—F1	90.00
C11—N1—C15	117.5 (3)	F2^{iv}—P1—F1	89.84 (12)
N1—C11—C12	122.5 (4)	F1^{iv}—P1—F2	89.84 (12)
C11—C12—C13	119.8 (4)	F1^{v}—P1—F2	90.16 (12)
C12—C13—C14	118.3 (4)	F1^{vi}—P1—F2	89.84 (12)
C13—C14—C15	118.9 (4)	F2^{iv}—P1—F2	179.99
C14—C15—N1	122.9 (4)	F1—P1—F2	90.16 (12)
F1^{iv}—P1—F1^{v}	90.00		

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

Table 2
Selected geometric parameters (Å, °) for **I** at 100 K.

Ag1—N1^{i}	2.310 (2)	C13—C14	1.381 (5)
Ag1—N1^{ii}	2.310 (2)	C14—C15	1.392 (4)
Ag1—N1^{iii}	2.310 (2)	P1—F1^{iv}	1.6047 (15)
Ag1—N1	2.310 (2)	P1—F1^{v}	1.6047 (15)
N1—C11	1.346 (3)	P1—F1^{vi}	1.6047 (15)
N1—C15	1.343 (3)	P1—F2^{iv}	1.584 (4)
C11—C12	1.381 (4)	P1—F1	1.6047 (15)
C12—C13	1.389 (4)	P1—F2	1.584 (4)
N1^{i}—Ag1—N1^{ii}	105.36 (5)	F1^{iv}—P1—F1^{vi}	179.88 (12)
N1^{i}—Ag1—N1^{iii}	105.36 (5)	F1^{v}—P1—F1^{vi}	90.00
N1^{ii}—Ag1—N1^{iii}	118.04 (10)	F1^{iv}—P1—F2^{iv}	90.06 (6)
N1^{i}—Ag1—N1	118.04 (10)	F1^{v}—P1—F2^{iv}	89.94 (6)
N1^{ii}—Ag1—N1	105.36 (5)	F1^{vi}—P1—F2^{iv}	90.06 (6)
N1^{iii}—Ag1—N1	105.36 (5)	F1^{iv}—P1—F1	90.00
Ag1—N1—C11	121.60 (16)	F1^{v}—P1—F1	179.88 (12)
Ag1—N1—C15	120.02 (17)	F1^{vi}—P1—F1	90.00
C11—N1—C15	117.5 (2)	F2^{iv}—P1—F1	89.94 (6)
N1—C11—C12	122.9 (2)	F1^{iv}—P1—F2	89.94 (6)
C11—C12—C13	119.2 (3)	F1^{v}—P1—F2	90.06 (6)
C12—C13—C14	118.5 (3)	F1^{vi}—P1—F2	89.94 (6)
C13—C14—C15	119.0 (2)	F2^{iv}—P1—F2	179.99
C14—C15—N1	122.9 (2)	F1—P1—F2	90.06 (6)
F1^{iv}—P1—F1^{v}	90.00		

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

and 2). Both **I** and **II** crystallize with $Z' = 1/4$, with the silver atom at the centre of the cation and the phosphorus/antimony and one fluorine all lying on the fourfold rotoinversion axis with the other atoms on general positions (Figs. 3 and 4). In both cases, the structure forms a close-packed, body-centred arrangement of cations with the anions located in the voids. At 300 K, the voids in **I** are 118 Å^3 contracting to 101 Å^3 at 100 K; for **II** the void of 134 Å^3 contracts to 120 Å^3 showing a similar lattice contraction to **I**. Geometric parameters are given for **I** and **II** at 300 and 100 K in Tables 1–4.

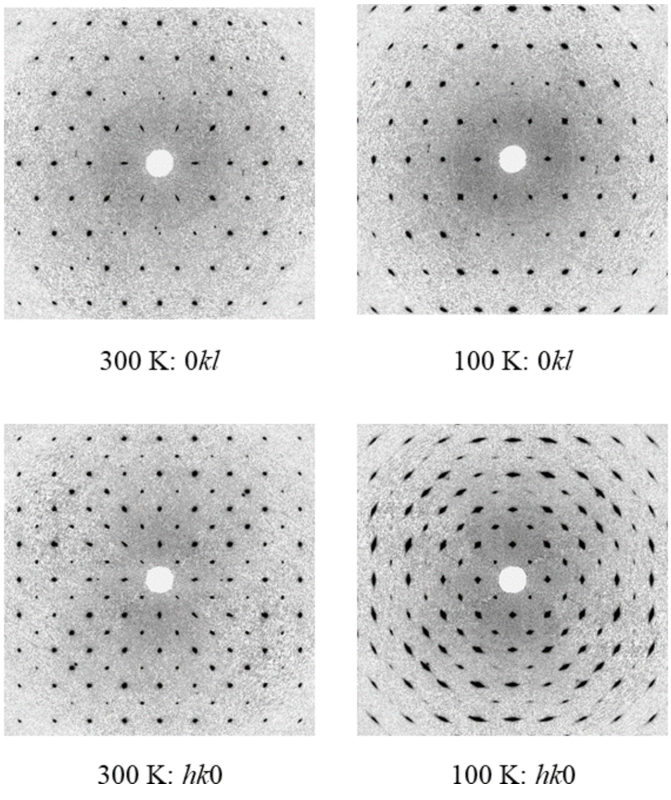


Figure 1
Selected reciprocal lattice sections reconstructed to 1.5 Å for the zero order layers for **I** at 300 K and 100 K.

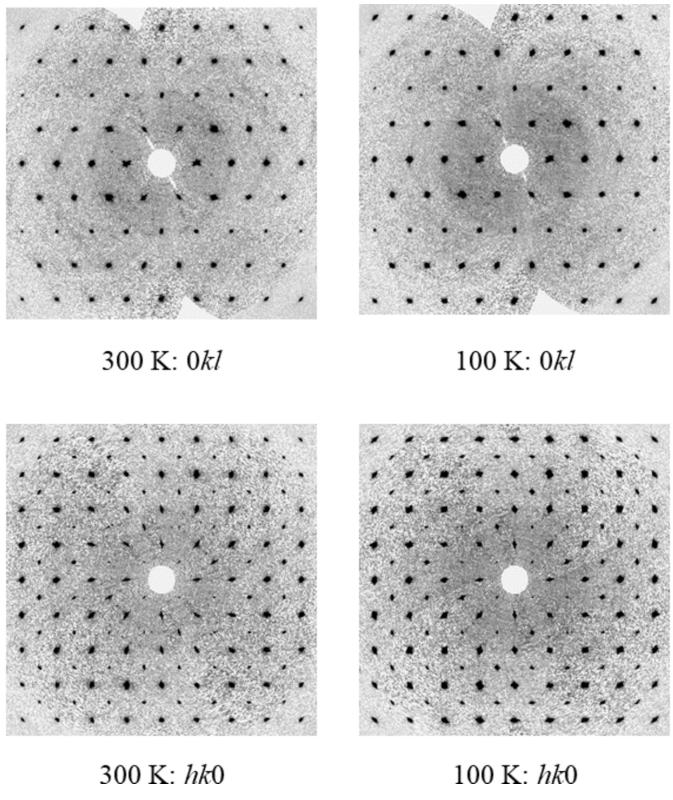


Figure 2
Selected reciprocal lattice sections reconstructed to 1.5 Å for the zero order layers for **II** at 300 K and 100 K.

Table 3
Selected geometric parameters (Å, °) for **II** at 300 K.

Ag1—N1^{i}	2.324 (7)	C13—C14	1.370 (18)
Ag1—N1^{ii}	2.324 (7)	C14—C15	1.366 (15)
Ag1—N1^{iii}	2.324 (7)	Sb1—F2^{iv}	1.872 (8)
Ag1—N1	2.324 (7)	Sb1—F1^{v}	1.849 (5)
N1—C11	1.328 (12)	Sb1—F1^{iv}	1.849 (5)
N1—C15	1.347 (10)	Sb1—F1^{vi}	1.849 (5)
C11—C12	1.342 (15)	Sb1—F1	1.849 (5)
C12—C13	1.368 (17)	Sb1—F2	1.872 (8)
N1^{i}—Ag1—N1^{ii}	108.10 (18)	F2^{iv}—Sb1—F1^{iv}	92.0 (5)
N1^{i}—Ag1—N1^{iii}	108.10 (18)	F1^{v}—Sb1—F1^{iv}	176.1 (10)
N1^{ii}—Ag1—N1^{iii}	112.2 (4)	F2^{iv}—Sb1—F1^{vi}	88.0 (5)
N1^{i}—Ag1—N1	112.2 (4)	F1^{v}—Sb1—F1^{vi}	90.07 (3)
N1^{ii}—Ag1—N1	108.10 (18)	F1^{iv}—Sb1—F1^{vi}	90.07 (3)
N1^{iii}—Ag1—N1	108.10 (18)	F2^{iv}—Sb1—F1	88.0 (5)
Ag1—N1—C11	121.3 (5)	F1^{v}—Sb1—F1	90.07 (3)
Ag1—N1—C15	121.0 (6)	F1^{iv}—Sb1—F1	90.07 (3)
C11—N1—C15	117.6 (8)	F1^{vi}—Sb1—F1	176.1 (10)
N1—C11—C12	123.2 (9)	F2^{iv}—Sb1—F2	179.99
C11—C12—C13	120.1 (11)	F1^{v}—Sb1—F2	88.0 (5)
C12—C13—C14	117.4 (10)	F1^{iv}—Sb1—F2	88.0 (5)
C13—C14—C15	120.3 (9)	F1^{vi}—Sb1—F2	92.0 (5)
C14—C15—N1	121.3 (9)	F1—Sb1—F2	92.0 (5)
F2^{iv}—Sb1—F1^{v}	92.0 (5)		

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

Table 4
Selected geometric parameters (Å, °) for **II** at 100 K.

Ag1—N1^{i}	2.305 (2)	C13—C14	1.384 (4)
Ag1—N1^{ii}	2.305 (2)	C14—C15	1.385 (4)
Ag1—N1^{iii}	2.305 (2)	Sb1—F2^{iv}	1.8809 (17)
Ag1—N1	2.305 (2)	Sb1—F1^{v}	1.8776 (13)
N1—C11	1.338 (3)	Sb1—F1^{iv}	1.8776 (13)
N1—C15	1.348 (3)	Sb1—F1^{vi}	1.8776 (13)
C11—C12	1.386 (4)	Sb1—F1	1.8776 (13)
C12—C13	1.383 (4)	Sb1—F2	1.8809 (17)
N1^{i}—Ag1—N1^{ii}	107.70 (5)	F2^{iv}—Sb1—F1^{iv}	90.62 (7)
N1^{i}—Ag1—N1^{iii}	107.70 (5)	F1^{v}—Sb1—F1^{iv}	178.75 (14)
N1^{ii}—Ag1—N1^{iii}	113.08 (11)	F2^{iv}—Sb1—F1^{vi}	89.38 (7)
N1^{i}—Ag1—N1	113.08 (11)	F1^{v}—Sb1—F1^{vi}	90.01
N1^{ii}—Ag1—N1	107.70 (5)	F1^{iv}—Sb1—F1^{vi}	90.01
N1^{iii}—Ag1—N1	107.70 (5)	F2^{iv}—Sb1—F1	89.38 (7)
Ag1—N1—C11	121.63 (17)	F1^{v}—Sb1—F1	90.01
Ag1—N1—C15	120.93 (17)	F1^{iv}—Sb1—F1	90.01
C11—N1—C15	117.1 (2)	F1^{vi}—Sb1—F1	178.75 (14)
N1—C11—C12	123.3 (2)	F2^{iv}—Sb1—F2	179.99
C11—C12—C13	118.9 (3)	F1^{v}—Sb1—F2	89.38 (7)
C12—C13—C14	118.5 (2)	F1^{iv}—Sb1—F2	89.38 (7)
C13—C14—C15	119.0 (2)	F1^{vi}—Sb1—F2	90.62 (7)
C14—C15—N1	123.1 (3)	F1—Sb1—F2	90.62 (7)
F2^{iv}—Sb1—F1^{v}	90.62 (7)		

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

3. Database survey

Searching the Cambridge Structural Database (CSD V. 5.45 with March 2024 and June 2024 updates; Groom *et al.*, 2016) using CONQUEST (Bruno *et al.*, 2002) for *X*Py$_4$ structures in the space group $I\overline{4}$ yields ten structures with three-dimensional coordinates deposited. For these, the *X*—N distance and the dihedral angles between the pyridine rings have been determined and plotted (Fig. 5). Since *X* occupies a special position, two pyridine–pyridine angles are the same and the third is related, such that as one decreases, the other increases. There is a general correlation between the *X*—N length and the dihedral angle; however, it is noticeable that the two compounds discussed herein exhibit larger dihedral angles than those previously reported and on cooling to 100 K, **I** (AgPy$_4$PF$_6$) shows a marked increase that is not seen for the SbF$_6$ analogue.

It is also worth pointing out that the Cu—N distance for CuPy$_4$I (YAGMAX, marked with an asterisk in Fig. 5; Al Shamaileh & Al-Far, 2016) is considerably shorter than the values for the other Cu structures plotted. Indeed, the value of 1.903 (4) Å is outside the interquartile range of 2.008–2.054 Å determined from a simple CSD search for a four-coordinate copper with four pyridine derived ligands. This structure was deposited in the CSD as a private communication, so while it is possible there is an error (*e.g.* the wrong atom type) more information is not readily available and the structure is included in the plot in Fig. 5 for completeness.

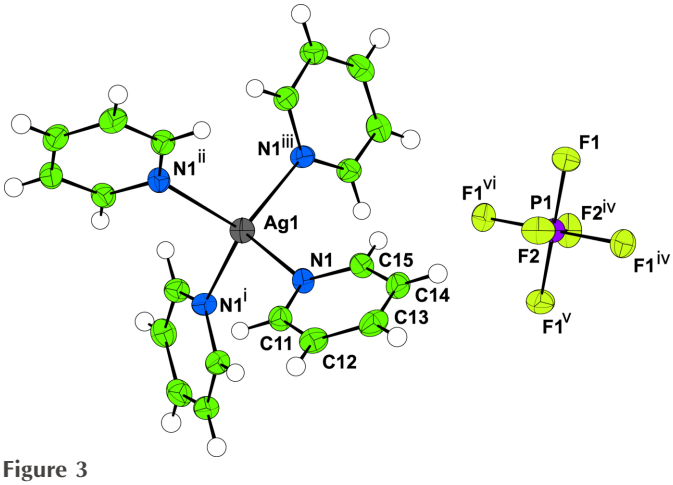


Figure 3
The molecular structure of **I** at 100 K. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) $y, 1-x, 1-z$; (ii) $1-x, 1-y, z$; (iii) $1-y, x, 1-z$; (iv) $y-\frac{1}{2}, \frac{1}{2}-x, \frac{1}{2}-z$; (v) $-x, 1-y, z$; (vi) $\frac{1}{2}-y, \frac{1}{2}+x, \frac{1}{2}-z$.

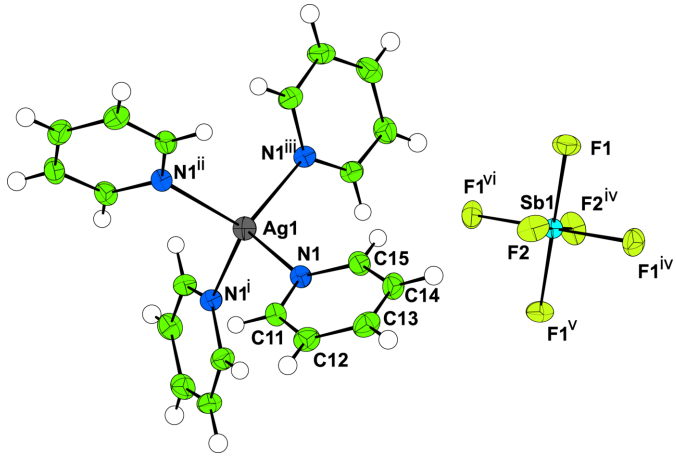


Figure 4
The molecular structure of **II** at 100 K. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) $y, 1-x, 1-z$; (ii) $1-x, 1-y, z$; (iii) $1-y, x, 1-z$; (iv) $y-\frac{1}{2}, \frac{1}{2}-x, \frac{1}{2}-z$; (v) $-x, 1-y, z$; (vi) $\frac{1}{2}-y, \frac{1}{2}+x, \frac{1}{2}-z$.

Table 5
Experimental details.

	I at 300 K	**I** at 100 K	**II** at 300 K	**II** at 100 K
Crystal data				
Chemical formula	$[Ag(C_5H_5N)_4](PF_6)$	$[Ag(C_5H_5N)_4](PF_6)$	$[Ag(C_5H_5N)_4](SbF_6)$	$[Ag(C_5H_5N)_4](SbF_6)$
M_r	569.24	569.23	660.01	660.01
Crystal system, space group	Tetragonal, $I\overline{4}$	Tetragonal, $I\overline{4}$	Tetragonal, $I\overline{4}$	Tetragonal, $I\overline{4}$
Temperature (K)	300	100	300	100
a, c (Å)	13.2831 (3), 6.6894 (7)	13.4408 (2), 6.1851 (1)	13.3825 (1), 6.8847 (1)	13.3461 (1), 6.5798 (1)
V (Å^3)	1180.28 (13)	1117.37 (4)	1232.99 (3)	1171.98 (3)
Z	2	2	2	2
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	8.06	8.52	15.60	16.42
Crystal size (mm)	0.23 × 0.06 × 0.05	0.23 × 0.06 × 0.05	0.25 × 0.20 × 0.05	0.25 × 0.20 × 0.05
Data collection				
Diffractometer	Oxford Diffraction SuperNova A	Oxford Diffraction SuperNova A	Oxford Diffraction SuperNova A	Oxford Diffraction SuperNova A
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2022)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2022)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2022)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.499, 1.000	0.153, 0.627	0.012, 0.394	0.093, 0.623
No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections	12427, 1240, 1154	10819, 1162, 1153	5859, 1286, 1281	5473, 1219, 1215
R_{int}	0.044	0.054	0.053	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.629	0.628	0.629
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.065, 0.86	0.021, 0.028, 1.01	0.048, 0.132, 1.01	0.017, 0.045, 1.02
No. of reflections	1240	1162	1286	1219
No. of parameters	73	73	73	73
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.30	0.58, −0.75	0.27, −0.73	0.20, −0.30
Absolute structure	Parsons *et al.* (2013), 469 Friedel Pairs	Parsons *et al.* (2013), 456 Friedel Pairs	Parsons *et al.* (2013), 471 Friedel Pairs	Parsons *et al.* (2013), 469 Friedel Pairs
Absolute structure parameter	−0.020 (5)	−0.028 (4)	−0.022 (17)	−0.006 (5)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SUPERFLIP* (Palatinus & Chapuis, 2007), *CRYSTALS* (Betteridge *et al.*, 2003) and *CAMERON* (Watkin *et al.*, 1996).

4. Synthesis and crystallization

For the synthesis of $AgPy_4PF_6$ (**I**), silver nitrate (1.5 g, 8.83 mmol, 1 eq.) and potassium hexafluorophosphate (1.63 g, 8.83 mmol, 1 eq.) were dissolved in deionized water (15 ml). Pyridine (2.0 ml, 24.7 mmol, 2.8 eq.) was added dropwise and the solution was stirred for 1 h. The precipitate was collected by suction filtration, washed with copious amounts of deionized water and dried for two days in a desiccator. Crystals were grown by vapour diffusion using DCM as the solvent and petroleum ether as the anti-solvent. Similar crystals (with a statistically indistinguishable unit cell) were also found using MeOH as the anti-solvent.

$AgPy_2SbF_6$ (**II**) was synthesized directly from $AgSbF_6$ (0.8 g, 2.33 mmol, 1 eq.) which was dissolved in deionized water (10 ml). Pyridine (0.53 ml, 6.52 mmol, 2.8 eq.) was added dropwise and the solution was stirred for 1 h. The precipitate was collected by suction filtration, washed with copious amounts of deionized water and dried for two days in a desiccator. Crystals were grown by solvent evaporation of DCM. Similar crystals (with a statistically indistinguishable unit cell) were also grown by vapour diffusion using DCM as the solvent and EtOH as the anti-solvent.

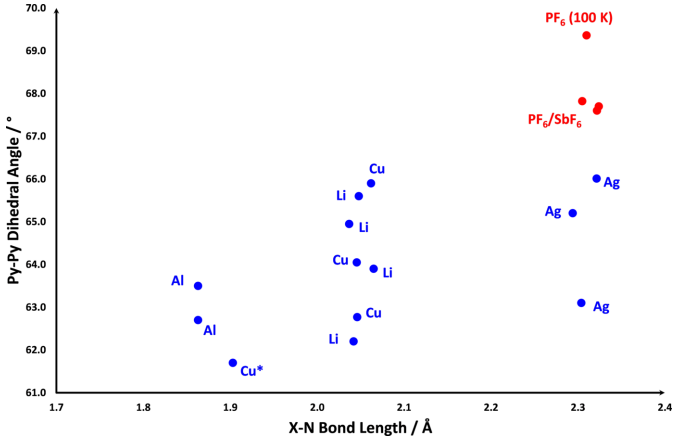


Figure 5
Scattergram plotting the dihedral angle between pyridine rings against the X—N bond distance for XPy_4 cations. Data points shown in blue are from literature values taken from the CSD while those in red are from this study. Each point is labelled with X, and the asterisk denotes an unusually short Cu—N distance as discussed herein.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. Both **I** and **II** crystallized from a mixed phase mixture with the $AgPy_2X$ analogue. Suitable crystals were isolated and mounted on a MiTeGen loop using

perfluoropolyether oil and placed in the N_2 stream of an Oxford CryoSystems CryoStream unit (Cosier & Glazer, 1986) at 300 K. Diffraction data were measured using a (Rigaku) Oxford Diffraction SuperNova A diffractometer ($K\alpha$ radiation, λ = 1.54184 Å). Raw frame images were processed using *CrysAlis PRO* (Rigaku OD, 2022). In both cases, the reflections were indexed using the same orientation matrix at both temperatures to enable comparison.

Examination of the symmetry equivalents and systematic absences suggested possible space groups of *I*4, $I\overline{4}$ and *I*4/*m;* however, structure solution with charge flipping using *SUPERFLIP* (Palatinus & Chapuis, 2007) suggested the space group was $I\overline{4}$. Once the symmetry and unit-cell contents had been confirmed, the crystal faces were indexed and an absorption correction applied.

The initial solution of the structure of **I** from the data collected at 300 K located all non-hydrogen atoms. Subsequent full-matrix least-squares refinement was carried out using the *CRYSTALS* program suite (Betteridge *et al.*, 2003). Coordinates and anisotropic displacement parameters of all non-hydrogen atoms were refined. The hydrogen atoms were all visible in the difference map, but were repositioned geometrically (Cooper *et al.*, 2010). Initially they were refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H distance = 0.93 Å), and U_{iso}(H) (1.2 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

The structure of **I** from the 300 K data was then refined against the data collected at 100 K: initially the non-hydrogen atoms were refined, then the hydrogen atoms with restraints before including them in the model with riding constraints. Similarly and to give a comparable set of structures, the structure of **I** at 300 K was refined against the data collected on **II** at 300 K. The model was modified to replace phosphorus with antimony and it was refined as above. Finally, the model for **II** at 300 K was refined against the data collected at 100 K (also as above). It is of note that the *R*-indices for **II** refined against the 300 K data are higher than those for the structure refined against the 100 K data. This is thought to be due to the fact that the data are weaker at the higher temperature and therefore noisier, something that is reflected in the internal agreement factors (5.3% and 300 K and 2.8% at 100 K).

Towards the end of each refinement, a modified Sheldrick weighting scheme was applied as per the details below (Watkin, 1994). In each case, the Flack *x* parameter (Flack, 1983) was included in the refinement and the absolute structure determined by analysis of the Bijvoet pairs (Thompson *et al.*, 2009; Parsons *et al.*, 2013). The void and dihedral angle calculations were carried out using *PLATON* (van der Sluis & Spek 1990; Spek, 2003, Spek 2009; Spek, 2015) and displacement ellipsoid plots were drawn with *CAMERON* (Watkin *et al.*, 1996).

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Variable temperature studies of tetrapyridinesilver(I) hexafluorophosphate and tetrapyridinesilver(I) hexafluoroantimonate

Alice G. McNelly, Kirsten E. Christensen and Amber L. Thompson

Computing details

Tetrapyridinesilver(I) hexafluorophosphate (I-300K)

Crystal data

$[Ag(C_5H_5N)_4](PF_6)$
M_r = 569.24
Tetragonal, $I\bar{4}$
Hall symbol: I -4
a = 13.2831 (3) Å
c = 6.6894 (7) Å
V = 1180.28 (13) Å^3
Z = 2
$F(000)$ = 568
D_x = 1.602 Mg m^{-3}
Melting point: not measured K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3328 reflections
θ = 4.7–73.0°
μ = 8.06 mm^{-1}
T = 300 K
Needle, clear_pale_colourless
0.23 × 0.06 × 0.05 mm

Data collection

Oxford Diffraction SuperNova A diffractometer
Focussing mirrors monochromator
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2022)
T_{min} = 0.499, T_{max} = 1.000
12427 measured reflections
1240 independent reflections
1154 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.044
θ_{max} = 76.0°, θ_{min} = 4.7°
h = −15→16
k = −16→16
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.065
S = 0.86
1240 reflections
73 parameters
0 restraints
Primary atom site location: other
Hydrogen site location: difference Fourier map
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.03P)^2 + 1.19P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.0001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Parsons *et al.* (2013), 469 Friedel Pairs
Absolute structure parameter: −0.020 (5)

Special details

Refinement. Reflections are selected by the following conditions :- Minimum value of SQRTW is 0.00 Minimum value of RATIO is -3.00

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.5000	0.5000	0.5000	0.0932
N1	0.3770 (2)	0.5809 (2)	0.6871 (5)	0.0789
C11	0.4011 (3)	0.6450 (3)	0.8296 (7)	0.0917
C12	0.3323 (4)	0.6830 (3)	0.9585 (8)	0.1104
C13	0.2339 (4)	0.6548 (4)	0.9433 (8)	0.1110
C14	0.2075 (3)	0.5911 (3)	0.7940 (8)	0.1002
C15	0.2804 (3)	0.5557 (3)	0.6692 (7)	0.0875
P1	0.0000	0.5000	0.2500	0.0703
F1	−0.0213 (2)	0.38358 (17)	0.2493 (5)	0.1276
F2	0.0000	0.5000	0.4827 (7)	0.1263
H111	0.4693	0.6662	0.8390	0.1101*
H121	0.3514	0.7252	1.0625	0.1420*
H131	0.1855	0.6797	1.0331	0.1380*
H141	0.1398	0.5717	0.7759	0.1199*
H151	0.2620	0.5117	0.5652	0.1029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0809 (2)	0.0809 (2)	0.1176 (4)	0.0000	0.0000	0.0000
N1	0.0643 (15)	0.0762 (17)	0.096 (2)	−0.0041 (12)	0.0023 (14)	−0.0071 (15)
C11	0.074 (2)	0.090 (2)	0.110 (3)	−0.0056 (18)	−0.008 (2)	−0.020 (2)
C12	0.117 (3)	0.108 (3)	0.106 (4)	0.010 (2)	−0.003 (3)	−0.026 (3)
C13	0.107 (3)	0.103 (3)	0.122 (4)	0.023 (2)	0.028 (3)	0.005 (3)
C14	0.068 (2)	0.097 (3)	0.135 (4)	−0.0037 (19)	0.012 (2)	0.011 (3)
C15	0.069 (2)	0.082 (2)	0.111 (3)	−0.0105 (17)	−0.0020 (19)	−0.009 (2)
P1	0.0686 (5)	0.0686 (5)	0.0738 (10)	0.0000	0.0000	0.0000
F1	0.152 (2)	0.0723 (14)	0.158 (2)	−0.0152 (15)	−0.001 (2)	−0.0115 (15)
F2	0.176 (3)	0.132 (3)	0.0702 (18)	0.007 (2)	0.0000	0.0000

Geometric parameters (Å, º)

Ag1—N1^{i}	2.322 (3)	C13—H131	0.940
Ag1—N1^{ii}	2.322 (3)	C14—C15	1.362 (6)
Ag1—N1^{iii}	2.322 (3)	C14—H141	0.944
Ag1—N1	2.322 (3)	C15—H151	0.940
N1—C11	1.318 (5)	P1—F1^{iv}	1.572 (2)
N1—C15	1.331 (4)	P1—F1^{v}	1.572 (2)
C11—C12	1.354 (6)	P1—F1^{vi}	1.572 (2)
C11—H111	0.951	P1—F2^{iv}	1.556 (4)
C12—C13	1.363 (6)	P1—F1	1.572 (2)
C12—H121	0.929	P1—F2	1.556 (4)
C13—C14	1.356 (6)		
N1^{i}—Ag1—N1^{ii}	106.90 (8)	C15—C14—H141	120.3

N1^{i}—Ag1—N1^{iii}	114.75 (16)	C14—C15—N1	122.9 (4)
N1^{ii}—Ag1—N1^{iii}	106.90 (8)	C14—C15—H151	118.9
N1^{i}—Ag1—N1	106.90 (8)	N1—C15—H151	118.2
N1^{ii}—Ag1—N1	114.75 (16)	F1^{iv}—P1—F1^{v}	90.00
N1^{iii}—Ag1—N1	106.90 (8)	F1^{iv}—P1—F1^{vi}	179.7 (2)
Ag1—N1—C11	121.2 (2)	F1^{v}—P1—F1^{vi}	90.00
Ag1—N1—C15	120.9 (3)	F1^{iv}—P1—F2^{iv}	90.16 (12)
C11—N1—C15	117.5 (3)	F1^{v}—P1—F2^{iv}	89.84 (12)
N1—C11—C12	122.5 (4)	F1^{vi}—P1—F2^{iv}	90.16 (12)
N1—C11—H111	118.1	F1^{iv}—P1—F1	90.00
C12—C11—H111	119.4	F1^{v}—P1—F1	179.7 (2)
C11—C12—C13	119.8 (4)	F1^{vi}—P1—F1	90.00
C11—C12—H121	121.2	F2^{iv}—P1—F1	89.84 (12)
C13—C12—H121	118.9	F1^{iv}—P1—F2	89.84 (12)
C12—C13—C14	118.3 (4)	F1^{v}—P1—F2	90.16 (12)
C12—C13—H131	120.8	F1^{vi}—P1—F2	89.84 (12)
C14—C13—H131	120.9	F2^{iv}—P1—F2	179.99
C13—C14—C15	118.9 (4)	F1—P1—F2	90.16 (12)
C13—C14—H141	120.8		

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

Tetrapyridinesilver(I) hexafluorophosphate (I-100K)

Crystal data

$[Ag(C_5H_5N)_4](PF_6)$
$M_r = 569.23$
Tetragonal, $I\bar{4}$
Hall symbol: I -4
$a = 13.4408$ (2) Å
$c = 6.1851$ (1) Å
$V = 1117.37$ (4) Å^3
$Z = 2$
$F(000) = 567.998$
$D_x = 1.692$ Mg m^{-3}
Melting point: not measured K
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 8381 reflections
$\theta = 4.6–75.8°$
$\mu = 8.52$ mm^{-1}
$T = 100$ K
Needle, clear_pale_colourless
0.23 × 0.06 × 0.05 mm

Data collection

Oxford Diffraction SuperNova A diffractometer
Focussing mirrors monochromator
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2022)
$T_{min} = 0.153$, $T_{max} = 0.627$
10819 measured reflections
1162 independent reflections
1153 reflections with $I > 2.0\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 75.9°$, $\theta_{min} = 4.7°$
$h = -15\rightarrow16$
$k = -16\rightarrow16$
$l = -7\rightarrow7$

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.028$
$S = 1.01$
1162 reflections
73 parameters
0 restraints
Primary atom site location: other
Hydrogen site location: difference Fourier map
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F) + (0.02P)^2 + 0.0P]$, where $P = (\max(F_o,0) + 2F_c)/3$

$(\Delta/\sigma)_{max}$ = 0.0001
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

Absolute structure: Parsons *et al.* (2013), 456 Friedel Pairs
Absolute structure parameter: −0.028 (4)

Special details

Refinement. Reflections are selected by the following conditions :- Minimum value of SQRTW is 0.00 Minimum value of RATIO is -3.00

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.5000	0.5000	0.5000	0.0318
N1	0.37821 (15)	0.58293 (16)	0.6922 (3)	0.0292
C11	0.40105 (19)	0.64517 (19)	0.8554 (4)	0.0310
C12	0.33091 (17)	0.68141 (16)	0.9983 (8)	0.0375
C13	0.23219 (19)	0.65286 (19)	0.9749 (7)	0.0393
C14	0.2074 (2)	0.5906 (2)	0.8057 (5)	0.0368
C15	0.28217 (19)	0.55778 (19)	0.6672 (4)	0.0328
P1	0.0000	0.5000	0.2500	0.0269
F1	−0.02173 (13)	0.38260 (11)	0.2497 (3)	0.0383
F2	0.0000	0.5000	0.5060 (6)	0.0413
H111	0.4677	0.6654	0.8686	0.0365*
H121	0.3499	0.7242	1.1087	0.0446*
H131	0.1831	0.6757	1.0704	0.0470*
H141	0.1426	0.5711	0.7848	0.0442*
H151	0.2655	0.5165	0.5510	0.0385*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.03228 (12)	0.03228 (12)	0.03097 (15)	0.0000	0.0000	0.0000
N1	0.0253 (9)	0.0323 (10)	0.0301 (10)	−0.0012 (7)	−0.0007 (7)	−0.0011 (8)
C11	0.0294 (11)	0.0327 (12)	0.0307 (11)	−0.0001 (9)	−0.0018 (9)	−0.0023 (9)
C12	0.0424 (11)	0.0341 (9)	0.0359 (10)	0.0042 (8)	0.0015 (19)	−0.0028 (19)
C13	0.0393 (11)	0.0373 (11)	0.0414 (19)	0.0091 (9)	0.0107 (13)	0.0070 (13)
C14	0.0279 (11)	0.0363 (12)	0.0463 (15)	0.0003 (9)	0.0033 (10)	0.0079 (10)
C15	0.0304 (11)	0.0317 (11)	0.0364 (11)	−0.0029 (9)	−0.0020 (10)	0.0013 (10)
P1	0.0291 (3)	0.0291 (3)	0.0223 (6)	0.0000	0.0000	0.0000
F1	0.0416 (8)	0.0310 (7)	0.0422 (8)	−0.0018 (6)	−0.0014 (7)	−0.0054 (6)
F2	0.0609 (11)	0.0387 (9)	0.0243 (9)	−0.0006 (8)	−0.0000	−0.0000

Geometric parameters (Å, º)

Ag1—N1^{i}	2.310 (2)	C13—H131	0.938
Ag1—N1^{ii}	2.310 (2)	C14—C15	1.392 (4)
Ag1—N1^{iii}	2.310 (2)	C14—H141	0.919
Ag1—N1	2.310 (2)	C15—H151	0.935
N1—C11	1.346 (3)	P1—F1^{iv}	1.6047 (15)
N1—C15	1.343 (3)	P1—F1^{v}	1.6047 (15)

C11—C12	1.381 (4)	P1—F1^{vi}	1.6047 (15)
C11—H111	0.939	P1—F2^{iv}	1.584 (4)
C12—C13	1.389 (4)	P1—F1	1.6047 (15)
C12—H121	0.929	P1—F2	1.584 (4)
C13—C14	1.381 (5)		

N1^{i}—Ag1—N1^{ii}	105.36 (5)	C15—C14—H141	120.5
N1^{i}—Ag1—N1^{iii}	105.36 (5)	C14—C15—N1	122.9 (2)
N1^{ii}—Ag1—N1^{iii}	118.04 (10)	C14—C15—H151	119.2
N1^{i}—Ag1—N1	118.04 (10)	N1—C15—H151	117.9
N1^{ii}—Ag1—N1	105.36 (5)	F1^{iv}—P1—F1^{v}	90.00
N1^{iii}—Ag1—N1	105.36 (5)	F1^{iv}—P1—F1^{vi}	179.88 (12)
Ag1—N1—C11	121.60 (16)	F1^{v}—P1—F1^{vi}	90.00
Ag1—N1—C15	120.02 (17)	F1^{iv}—P1—F2^{iv}	90.06 (6)
C11—N1—C15	117.5 (2)	F1^{v}—P1—F2^{iv}	89.94 (6)
N1—C11—C12	122.9 (2)	F1^{vi}—P1—F2^{iv}	90.06 (6)
N1—C11—H111	117.5	F1^{iv}—P1—F1	90.00
C12—C11—H111	119.6	F1^{v}—P1—F1	179.88 (12)
C11—C12—C13	119.2 (3)	F1^{vi}—P1—F1	90.00
C11—C12—H121	120.1	F2^{iv}—P1—F1	89.94 (6)
C13—C12—H121	120.7	F1^{iv}—P1—F2	89.94 (6)
C12—C13—C14	118.5 (3)	F1^{v}—P1—F2	90.06 (6)
C12—C13—H131	121.1	F1^{vi}—P1—F2	89.94 (6)
C14—C13—H131	120.4	F2^{iv}—P1—F2	179.99
C13—C14—C15	119.0 (2)	F1—P1—F2	90.06 (6)
C13—C14—H141	120.5		

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

Tetrapyridinesilver(I) hexafluoroantimonate (II-300K)

Crystal data

$[Ag(C_5H_5N)_4](SbF_6)$
M_r = 660.01
Tetragonal, $I\bar{4}$
Hall symbol: I -4
a = 13.3825 (1) Å
c = 6.8847 (1) Å
V = 1232.99 (3) Å^3
Z = 2
F(000) = 640

D_x = 1.778 Mg m^{-3}
Melting point: not measured K
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 4713 reflections
θ = 4.7–75.2°
μ = 15.60 mm^{-1}
T = 300 K
Block, clear_pale_colourless
0.25 × 0.20 × 0.05 mm

Data collection

Oxford Diffraction SuperNova A diffractometer
Focussing mirrors monochromator
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2022)
T_{min} = 0.012, T_{max} = 0.394
5859 measured reflections

1286 independent reflections
1281 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.053
θ_{max} = 75.4°, θ_{min} = 4.7°
h = −16→16
k = −16→16
l = −6→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.132$
$S = 1.00$
1286 reflections
73 parameters
0 restraints
Primary atom site location: other
Hydrogen site location: difference Fourier map
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + ($
$0.11P)^2 + 0.34P]$,
where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.73$ e Å^{-3}
Absolute structure: Parsons *et al.* (2013), 471
Friedel Pairs
Absolute structure parameter: −0.022 (17)

Special details

Refinement. Reflections are selected by the following conditions :- Minimum value of SQRTW is 0.00 Minimum value of RATIO is -3.00

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.5000	0.5000	0.5000	0.1126
N1	0.3792 (5)	0.5787 (5)	0.6882 (11)	0.0930
C11	0.4043 (6)	0.6419 (8)	0.8286 (16)	0.1051
C12	0.3377 (9)	0.6818 (10)	0.9521 (19)	0.1247
C13	0.2384 (9)	0.6589 (9)	0.9356 (19)	0.1200
C14	0.2110 (7)	0.5962 (8)	0.788 (2)	0.1162
C15	0.2815 (6)	0.5573 (7)	0.6658 (17)	0.1041
Sb1	0.0000	0.5000	0.2500	0.0747
F1	−0.0202 (7)	0.3634 (4)	0.241 (2)	0.1602
F2	0.0000	0.5000	0.5220 (11)	0.1412
H111	0.4712	0.6581	0.8442	0.1228*
H121	0.3593	0.7260	1.0471	0.1460*
H131	0.1927	0.6855	1.0236	0.1440*
H141	0.1443	0.5789	0.7713	0.1399*
H151	0.2615	0.5158	0.5654	0.1219*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0990 (5)	0.0990 (5)	0.1398 (11)	0.0000	0.0000	0.0000
N1	0.078 (3)	0.091 (3)	0.110 (5)	−0.006 (3)	−0.003 (2)	−0.003 (3)
C11	0.086 (4)	0.108 (5)	0.122 (6)	−0.008 (4)	−0.009 (4)	−0.014 (4)
C12	0.128 (7)	0.133 (8)	0.114 (7)	0.001 (6)	−0.006 (6)	−0.024 (6)
C13	0.118 (6)	0.116 (6)	0.125 (7)	0.011 (5)	0.024 (6)	0.002 (5)
C14	0.084 (4)	0.128 (6)	0.137 (12)	−0.005 (4)	0.010 (5)	0.018 (6)
C15	0.086 (4)	0.103 (5)	0.123 (6)	−0.013 (4)	−0.010 (4)	−0.002 (4)
Sb1	0.0768 (3)	0.0768 (3)	0.0707 (3)	0.0000	0.0000	0.0000
F1	0.225 (8)	0.083 (2)	0.173 (6)	−0.021 (3)	0.012 (9)	−0.013 (5)
F2	0.188 (12)	0.148 (10)	0.088 (3)	0.025 (10)	0.0000	0.0000

Geometric parameters (Å, º)

Ag1—N1^{i}	2.324 (7)	C13—H131	0.932
Ag1—N1^{ii}	2.324 (7)	C14—C15	1.366 (15)
Ag1—N1^{iii}	2.324 (7)	C14—H141	0.929
Ag1—N1	2.324 (7)	C15—H151	0.926
N1—C11	1.328 (12)	Sb1—F2^{iv}	1.872 (8)
N1—C15	1.347 (10)	Sb1—F1^{v}	1.849 (5)
C11—C12	1.342 (15)	Sb1—F1^{iv}	1.849 (5)
C11—H111	0.927	Sb1—F1^{vi}	1.849 (5)
C12—C13	1.368 (17)	Sb1—F1	1.849 (5)
C12—H121	0.928	Sb1—F2	1.872 (8)
C13—C14	1.370 (18)		
N1^{i}—Ag1—N1^{ii}	108.10 (18)	C15—C14—H141	119.6
N1^{i}—Ag1—N1^{iii}	108.10 (18)	C14—C15—N1	121.3 (9)
N1^{ii}—Ag1—N1^{iii}	112.2 (4)	C14—C15—H151	119.1
N1^{i}—Ag1—N1	112.2 (4)	N1—C15—H151	119.6
N1^{ii}—Ag1—N1	108.10 (18)	F2^{iv}—Sb1—F1^{v}	92.0 (5)
N1^{iii}—Ag1—N1	108.10 (18)	F2^{iv}—Sb1—F1^{iv}	92.0 (5)
Ag1—N1—C11	121.3 (5)	F1^{v}—Sb1—F1^{iv}	176.1 (10)
Ag1—N1—C15	121.0 (6)	F2^{iv}—Sb1—F1^{vi}	88.0 (5)
C11—N1—C15	117.6 (8)	F1^{v}—Sb1—F1^{vi}	90.07 (3)
N1—C11—C12	123.2 (9)	F1^{iv}—Sb1—F1^{vi}	90.07 (3)
N1—C11—H111	118.5	F2^{iv}—Sb1—F1	88.0 (5)
C12—C11—H111	118.3	F1^{v}—Sb1—F1	90.07 (3)
C11—C12—C13	120.1 (11)	F1^{iv}—Sb1—F1	90.07 (3)
C11—C12—H121	119.6	F1^{vi}—Sb1—F1	176.1 (10)
C13—C12—H121	120.3	F2^{iv}—Sb1—F2	179.99
C12—C13—C14	117.4 (10)	F1^{v}—Sb1—F2	88.0 (5)
C12—C13—H131	119.9	F1^{iv}—Sb1—F2	88.0 (5)
C14—C13—H131	122.7	F1^{vi}—Sb1—F2	92.0 (5)
C13—C14—C15	120.3 (9)	F1—Sb1—F2	92.0 (5)
C13—C14—H141	120.1		

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

Tetrapyridinesilver(I) hexafluoroantimonate (II-100K)

Crystal data

$[Ag(C_5H_5N)_4](SbF_6)$
M_r = 660.01 _
Tetragonal, $I\overline{4}$
Hall symbol: I -4
a = 13.3461 (1) Å
c = 6.5798 (1) Å
V = 1171.98 (3) Å^3
Z = 2
F(000) = 640

D_x = 1.870 Mg m^{-3}
Melting point: not measured K
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 4749 reflections
θ = 4.7–75.5°
μ = 16.42 mm^{-1}
T = 100 K
Block, clear_pale_colourless
0.25 × 0.20 × 0.05 mm

Data collection

Oxford Diffraction SuperNova A diffractometer
Focussing mirrors monochromator
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2022)
T_{min} = 0.093, T_{max} = 0.623
5473 measured reflections
1219 independent reflections
1215 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.028
θ_{max} = 75.9°, θ_{min} = 4.7°
h = −16→16
k = −16→16
l = −6→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.045
S = 1.02
1219 reflections
73 parameters
0 restraints
Primary atom site location: other
Hydrogen site location: difference Fourier map
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.43P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.0001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Parsons *et al.* (2013), 469 Friedel Pairs
Absolute structure parameter: −0.006 (5)

Special details

Refinement. ? Reflections are selected by the following conditions :- Minimum value of SQRTW is 0.00 Minimum value of RATIO is -3.00

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.5000	0.5000	0.5000	0.0290
N1	0.38043 (16)	0.58038 (16)	0.6931 (3)	0.0259
C11	0.40561 (19)	0.64332 (19)	0.8429 (4)	0.0278
C12	0.3366 (2)	0.6848 (2)	0.9760 (4)	0.0330
C13	0.2365 (2)	0.6601 (2)	0.9552 (5)	0.0351
C14	0.2089 (2)	0.5962 (2)	0.7993 (4)	0.0338
C15	0.2824 (2)	0.5584 (2)	0.6721 (4)	0.0292
Sb1	0.0000	0.5000	0.2500	0.0191
F1	−0.02296 (12)	0.36121 (10)	0.2469 (3)	0.0356
F2	0.0000	0.5000	0.5359 (3)	0.0370
H111	0.4727	0.6606	0.8569	0.0342*
H121	0.3572	0.7282	1.0784	0.0430*
H131	0.1887	0.6854	1.0453	0.0441*
H141	0.1420	0.5786	0.7805	0.0439*
H151	0.2632	0.5160	0.5661	0.0370*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.02868 (12)	0.02868 (12)	0.02968 (18)	0.0000	0.0000	0.0000
N1	0.0238 (10)	0.0259 (10)	0.0279 (10)	−0.0013 (8)	0.0004 (7)	0.0000 (7)
C11	0.0238 (11)	0.0280 (12)	0.0315 (12)	−0.0017 (9)	−0.0022 (9)	0.0000 (10)

C12	0.0371 (13)	0.0308 (12)	0.0311 (13)	0.0035 (10)	0.0002 (11)	−0.0049 (11)
C13	0.0343 (13)	0.0341 (13)	0.0368 (16)	0.0070 (10)	0.0092 (10)	0.0053 (10)
C14	0.0224 (11)	0.0371 (13)	0.0419 (17)	−0.0015 (10)	0.0019 (9)	0.0095 (11)
C15	0.0265 (12)	0.0303 (12)	0.0308 (11)	−0.0036 (9)	−0.0024 (10)	0.0024 (10)
Sb1	0.02040 (10)	0.02040 (10)	0.01656 (12)	0.0000	0.0000	0.0000
F1	0.0408 (7)	0.0227 (6)	0.0433 (8)	−0.0020 (5)	0.0021 (9)	−0.0044 (8)
F2	0.0568 (16)	0.0371 (13)	0.0171 (9)	0.0069 (13)	0.0000	0.0000

Geometric parameters (Å, º)

Ag1—N1^{i}	2.305 (2)	C13—H131	0.934
Ag1—N1^{ii}	2.305 (2)	C14—C15	1.385 (4)
Ag1—N1^{iii}	2.305 (2)	C14—H141	0.931
Ag1—N1	2.305 (2)	C15—H151	0.934
N1—C11	1.338 (3)	Sb1—F2^{iv}	1.8809 (17)
N1—C15	1.348 (3)	Sb1—F1^{v}	1.8776 (13)
C11—C12	1.386 (4)	Sb1—F1^{iv}	1.8776 (13)
C11—H111	0.929	Sb1—F1^{vi}	1.8776 (13)
C12—C13	1.383 (4)	Sb1—F1	1.8776 (13)
C12—H121	0.929	Sb1—F2	1.8809 (17)
C13—C14	1.384 (4)		

N1^{i}—Ag1—N1^{ii}	107.70 (5)	C15—C14—H141	120.4
N1^{i}—Ag1—N1^{iii}	107.70 (5)	C14—C15—N1	123.1 (3)
N1^{ii}—Ag1—N1^{iii}	113.08 (11)	C14—C15—H151	118.6
N1^{i}—Ag1—N1	113.08 (11)	N1—C15—H151	118.3
N1^{ii}—Ag1—N1	107.70 (5)	F2^{iv}—Sb1—F1^{v}	90.62 (7)
N1^{iii}—Ag1—N1	107.70 (5)	F2^{iv}—Sb1—F1^{iv}	90.62 (7)
Ag1—N1—C11	121.63 (17)	F1^{v}—Sb1—F1^{iv}	178.75 (14)
Ag1—N1—C15	120.93 (17)	F2^{iv}—Sb1—F1^{vi}	89.38 (7)
C11—N1—C15	117.1 (2)	F1^{v}—Sb1—F1^{vi}	90.01
N1—C11—C12	123.3 (2)	F1^{iv}—Sb1—F1^{vi}	90.01
N1—C11—H111	118.1	F2^{iv}—Sb1—F1	89.38 (7)
C12—C11—H111	118.6	F1^{v}—Sb1—F1	90.01
C11—C12—C13	118.9 (3)	F1^{iv}—Sb1—F1	90.01
C11—C12—H121	120.7	F1^{vi}—Sb1—F1	178.75 (14)
C13—C12—H121	120.4	F2^{iv}—Sb1—F2	179.99
C12—C13—C14	118.5 (2)	F1^{v}—Sb1—F2	89.38 (7)
C12—C13—H131	120.7	F1^{iv}—Sb1—F2	89.38 (7)
C14—C13—H131	120.8	F1^{vi}—Sb1—F2	90.62 (7)
C13—C14—C15	119.0 (2)	F1—Sb1—F2	90.62 (7)
C13—C14—H141	120.6		

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