$S = 0.75$
2779 reflections
137 parameters
$w = 1/[\sigma^2(F) + 0.012134F^2]$
$(\Delta/\sigma)_{max} = 0.050$

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn	0.21397 (7)	0.05703 (5)	0.21018 (5)	0.0452 (2)
I1	0.15287 (10)	0.25475 (6)	0.11184 (7)	0.0689 (3)
I2	0.39800 (9)	0.10823 (7)	0.37307 (6)	0.0652 (3)
C1	0.3067 (10)	−0.0480 (8)	0.0984 (8)	0.048 (2)
C2	0.4418 (11)	−0.0857 (9)	0.1186 (8)	0.052 (2)
C3	0.4997 (11)	−0.1533 (10)	0.0453 (9)	0.057 (3)
C4	0.4238 (14)	−0.1785 (10)	−0.0473 (9)	0.065 (3)
C5	0.2888 (15)	−0.1368 (12)	−0.0700 (10)	0.071 (4)
C6	0.2286 (13)	−0.0709 (11)	0.0033 (9)	0.066 (3)
C7	0.0211 (10)	0.0007 (8)	0.2654 (8)	0.048 (2)
C8	−0.0365 (17)	0.0561 (14)	0.3463 (14)	0.095 (6)
C9	−0.1633 (18)	0.0181 (17)	0.3787 (14)	0.093 (6)
C10	−0.2352 (14)	−0.0676 (15)	0.3287 (14)	0.086 (5)
C11	−0.1762 (13)	−0.1205 (12)	0.2485 (12)	0.074 (4)
C12	−0.0478 (12)	−0.0887 (9)	0.2163 (9)	0.055 (3)

Table 2. *Selected geometric parameters (Å, °)*

I1—Sn	2.7097 (10)	I2—Sn	2.6749 (10)
C1—Sn	2.140 (10)	C7—Sn	2.133 (9)
I2—Sn—I1	105.17 (3)	C1—Sn—I1	106.7 (3)
C1—Sn—I2	111.5 (3)	C7—Sn—I1	105.5 (3)
C7—Sn—I2	110.3 (3)	C7—Sn—C1	116.7 (4)
C2—C1—Sn	120.1 (7)	C6—C1—Sn	118.0 (8)
C8—C7—Sn	120.3 (9)	C12—C7—Sn	119.2 (8)
I1···Sn^{i}	4.3885 (10)	I2···I1^{iii}	4.0745 (12)
I1···I2^{ii}	4.0745 (12)	I2···I1^{iv}	4.2490 (11)
I1···I2^{i}	4.2490 (11)	I2···I2^{v}	4.4550 (11)

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

The structure was solved by heavy-atom methods and refinement was by full-matrix least-squares methods. All non-H atoms were refined anisotropically. H atoms were placed in calculated positions (C—H = 0.95 Å) and refined riding upon the C atom to which they are attached with a common group U_{iso}. All computations were performed on the SUN SPARCserver (UNIX operating system) of the University of Aberdeen.

Data collection: *Nicolet P3 Software* (Nicolet XRD Corporation, 1980). Cell refinement: *Nicolet P3 Software*. Data reduction: *RDNIC* (Howie, 1980). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *PLOTAID* (Cradwick, 1970) and *ORTEX* (McArdle, 1994). Software used to prepare material for publication: *XPUB* (Low, 1980).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1048). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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catena-Poly[silver-μ-(di-2-pyridyl ketone)-*N*,*O*:*N'*,*O*] Tetrafluoroborate

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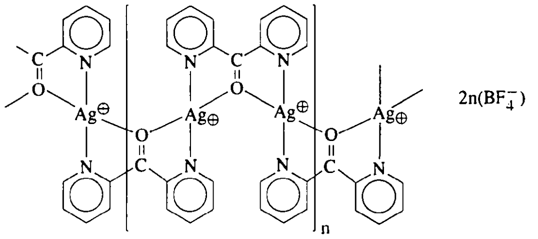
Abstract

The title complex, $[Ag(C_{11}H_8N_2O)]^+_n.n(BF_4)^-$, contains polymeric [silver(di-2-pyridyl ketone)]^+_n zigzag chains, counterbalanced by BF_4^- anions. The Ag atoms reside on inversion centers, the di-2-pyridyl ketone ligands lie with their carbonyl groups along crystallographic twofold axes, and the BF_4^- ions also reside on inversion centers (and are disordered). The AgN_2O_2 moiety is precisely planar, with Ag—N 2.180 (3) and Ag—O 2.552 (1) Å, and N—Ag—O 69.35 (9)°.

Comment

We have described previously *catena*-poly[(nitrato-*O*,*O'*)silver(I)-μ-(di-2-pyridyl ketone-*N*:*N'*)] (Sommerer, Westcott & Abboud, 1994), a metal-containing extended structure consisting of Ag(dpk)$^+$ chains which adopt an extended spiral structure (dpk is the bidentate ligand di-2-pyridyl ketone). A prominent feature of this structure is the presence of a bidentate NO_3^- anion which has a pronounced effect on the tertiary structure of the polymer. In order to explore the relationship between the chemistry of the anion and the three-dimensional structure further, the nitrate anion was replaced with BF_4^-, a potentially non-coordinating anion. We describe here the resulting structure, (I).



(I)

The crystals were found to consist of Ag(dpk)$^+$ zigzag chains (Fig. 1) counterbalanced by BF_4^- anions. The Ag atoms reside on centers of inversion and the C7—O bond lies along a twofold axis. The Ag coordination is distorted square planar; the AgN_2O_2 moiety is precisely planar with Ag—O [2.552 (1) Å] being significantly longer than Ag—N [2.180 (3) Å]. The dihedral angle between the two pyridine rings is 48.6 (5)°; the dihedral angle between the pyridine ring and the carbonyl plane (C6, C7, O, C6^{ii}) is 27.2 (4)° with a twist angle (torsion angle) for C5—C6—C7—C6^{ii} of 29.4 (4)° [symmetry code: (ii) $-x, y, \frac{1}{2} - z$].

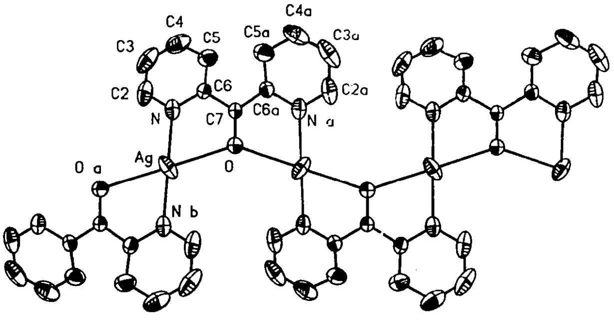


Fig. 1. Displacement ellipsoid drawing of the extended structure of the cation of the title compound with ellipsoids drawn at the 50% probability level. The symmetry operation for O*a* and N*b* from O and N is $-x, 1 - y, -z$, and for N*a* from N is $-x, y, \frac{1}{2} - z$.

The Ag—O distance is roughly 0.10 Å longer than the Ag—O bond formed by direct coordination in analogous species (Aoyama, Yamanaka, Ohba & Saito, 1990) and 0.20 Å longer than that reported for a species containing Ag—μ-O$_{oxo}$ bonds (Colsman *et al.*, 1990). The Ag—N distances are unremarkable and within ±0.10 Å of those reported for related Ag species (Sommerer, Westcott & Abboud, 1994; Housecroft, 1992; Nilsson & Oskarsson, 1982).

The five-membered chelate ring adopts an envelope conformation with the O atom occupying the flap position. The O atom is 0.41 (1) Å from the plane of the envelope containing atoms Ag—N—C6—C7. The torsion angle of the envelope, Ag—N—C6—C7, is −2.2 (3)°, and is the smallest in the ring.

The B atom of the unique BF_4^- anion also lies on an inversion center and the four F atoms are distorted over eight sites. The BF_4^- ions reside between $[Ag(dpk)^+]_n$ cation chains with the shortest Ag···B contact being 4.113 (3) Å. A weak long-range interaction may exist between the Ag and the F atoms with Ag···F3 and Ag···F4 (where F4 requires the transformation $\frac{1}{2} - x, \frac{1}{2} - y, -z$) distances of 3.175 (6) and 3.193 (7) Å, respectively. The Ag···Ag separation within the plane of the extended chain is 4.858 (1) Å. The shortest distance between two Ag atoms in successive layers is 8.226 (1) Å, with the perpendicular distance between Ag and the plane of coordination of the adjacent layer being 7.583 (1) Å.

We observe in the present structure that the carbonyl O atom plays a prominent role in forming a μ-oxo bridge between the Ag$^+$ centers and ultimately helps to maintain the planarity of the extended structure. This is significantly different from what was found in *catena*-poly[(nitrato-*O*,*O*′)silver(I)-μ-(di-2-pyridyl ketone-*N*:*N*′)], where the dpk ligand again bridges separate Ag$^+$ cations by coordinating through the pyridine N atoms but requires a dramatic rotation of the pyridine rings around the C6—C7 bonds leading to a complete break in planarity, resulting in a dihedral angle of 111.3 (3)°. Hence, incorporation of a non-coordinating anion appears to enhance the coordination opportunity for the carbonyl of dpk, which ultimately leads to a significantly different tertiary structure for Ag(dpk)$^+$.

Experimental

The title compound was prepared when silver tetrafluroborate (prepared from silver metal and fluroboric acid) was combined in a 1:1 stoichiometric ratio with dpk in 70 ml of H_2O. The resulting mixture was filtered and slow evaporation of the yellow filtrate gave clear yellow crystals, suitable for X-ray analysis, within 10 d.

Crystal data

$[Ag(C_{11}H_8N_2O)](BF_4)$
$M_r = 378.87$
Monoclinic
$C2/c$
$a = 12.129 (1)$ Å
$b = 11.116 (1)$ Å
$c = 9.716 (1)$ Å
$\beta = 93.95 (1)°$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10–11°$
$\mu = 1.58$ mm^{-1}
$T = 298$ K
Block

$V = 1306.9\,(2)$ Å^3
$Z = 4$
$D_x = 1.926$ Mg m^{-3}
$D_m = 1.94$ Mg m^{-3}
D_m measured by floatation
0.47 × 0.36 × 0.25 mm
Yellow

Data collection

Siemens *P3m/V* diffractometer
ω scans
Absorption correction: analytical
$T_{min} = 0.606$, $T_{max} = 0.701$
2080 measured reflections
1900 independent reflections
1524 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.0097$
$\theta_{max} = 30°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 15$
$l = -13 \rightarrow 13$
4 standard reflections monitored every 96 reflections intensity decay: 1%

Refinement

Refinement on F
$R = 0.0456$
$wR = 0.0584$
$S = 2.06$
1524 reflections
113 parameters
H atoms riding
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.99$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Ag	0	1/2	0	0.0738 (2)
O	0	0.4297 (3)	1/4	0.083 (2)
N	−0.0600 (2)	0.3156 (3)	0.0100 (2)	0.0538 (9)
C2	−0.1087 (4)	0.2609 (5)	−0.0997 (4)	0.0760 (15)
C3	−0.1541 (5)	0.1486 (6)	−0.0960 (6)	0.095 (2)
C4	−0.1519 (4)	0.0903 (5)	0.0238 (6)	0.088 (2)
C5	−0.1008 (4)	0.1424 (3)	0.1404 (4)	0.0665 (13)
C6	−0.0535 (3)	0.2546 (3)	0.1299 (3)	0.0454 (8)
C7	0	0.3201 (3)	1/4	0.0463 (12)
B	1/4	1/4	0	0.083 (3)
F1	0.3036 (9)	0.2937 (11)	−0.0843 (10)	0.159 (5)
F2	0.1471 (6)	0.1914 (6)	−0.0649 (7)	0.105 (3)
F3	0.2007 (5)	0.3287 (6)	0.0941 (6)	0.092 (2)
F4	0.2929 (6)	0.1536 (6)	0.0851 (6)	0.103 (2)

Table 2. *Selected geometric parameters (Å, °)*

Ag—O	2.552 (1)	C2—C3	1.366 (8)
Ag—N	2.180 (3)	C3—C4	1.331 (8)
O—C7	1.217 (5)	C4—C5	1.381 (7)
N—C2	1.330 (5)	C5—C6	1.380 (5)
N—C6	1.345 (4)	C6—C7	1.486 (3)
O—Ag—N	69.35 (9)	C5—C4—C3	119.5
O—Ag—N^i	110.65 (9)	C6—C5—C4	119.0 (4)
C7—O—Ag	107.85 (7)	C7—C6—N	115.4 (3)
C2—N—C6	117.4 (3)	C7—C6—C5	123.1 (3)
C2—N—Ag	121.7 (3)	N—C6—C5	121.3 (3)
C6—N—Ag	120.8 (2)	O—C7—C6	119.3 (2)
C3—C2—N	123.7 (4)	C6—C7—C6^{ii}	121.3 (3)
C4—C3—C2	119.0 (5)		

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

The H atoms were placed in calculated idealized positions (C—H 0.96 Å) and given fixed displacement parameters (0.08 Å^2).

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1132). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Polymeric [*p*-(2-Hydroxy-5-methylphenylazo)benzoato]trimethyltin

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Abstract

The presence of bidentate bridging carboxylate ligands leads to the formation of a polymeric structure in the title compound, *catena*-poly[trimethyltin-μ-[*p*-(2-hydroxy-5-methylphenylazo)benzoato-*O*:*O'*]], $[Sn(C_{14}H_{11}N_2O_3)(CH_3)_3]_n$. The Sn atom is five-coordinate and has distorted trigonal bipyramidal geometry with the three methyl groups defining the trigonal plane.

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