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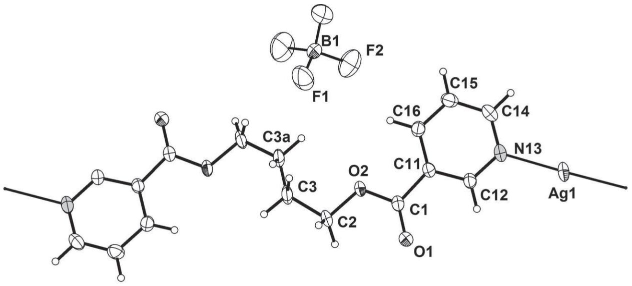
Crystal structure of *catena*-poly[(μ$_2$-butane-1,4-diyl-bis(pyridine-3-carboxylato-κ*N*)) silver(I)] tetrafluoroborate, $C_{16}H_{16}AgN_2O_4BF_4$



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Abstract

$C_{16}H_{16}AgN_2O_4BF_4$, monoclinic, $C2/c$ (no. 15), $a = 17.416(2)$ Å, $b = 13.1378(14)$ Å, $c = 7.8246(10)$ Å, $\beta = 96.566(10)°$, $V = 1778.6(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.025$, $wR_{ref}(F^2) = 0.065$, $T = 173(2)$ K.

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A part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.33 \times 0.29 \times 0.27$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	12.0 cm^{-1}
Diffractometer, scan mode:	Bruker IPDS II, ω-scans
$2\theta_{max}$, completeness:	51.2°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6065, 1665, 0.040
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1628
$N(param)_{refined}$:	138
Programs:	SHELX [4], Stoe X-AREA [5], PLATON [6], Absorption correction [7]

Source of material

A solution of $AgBF_4$ (19.46 mg, 0.1 mmol) in water was slowly added to a solution of the ligand, butane-1,4-diyl bis (pyridine-3-carboxylate) (L) (30.03 mg, 0.1 mmol) in THF (4 ml). Colourless crystals suitable for X-ray were obtained after a few days (85%).

Experimental details

All H atoms were located in the difference Fourier map, but refined with fixed individual displacement parameters, using a riding mode with C—H distances of 0.95 Å (for aromatic rings), 0.99 Å (for CH_2), with $U_{iso}(H)$ values of $1.2U_{eq}(C)$ (for CH in aromatic and CH_2).

Discussion

The design of polymeric organic-inorganic materials with novel topologies and structural motifs is of current interest in the field of coordination chemistry [1] . This paper forms part of our continuing study of the synthesis, structural characterization and physical properties of coordination polymers [2].

In the title compound the molecule lies on a twofold rotation axis which passes through the mid point of the C3—C3′ bond, giving one half molecule per asymmetric unit. The Ag atom exists in a linear AgN_2 coordination geometry; it lies on an two-fold axis. The bond distances and angles Ag—N and N—Ag$\cdots$NA($A = -x, y, 1/2 - z$) are 2.151(2) Å and 167.46(11)° respectively (*cf.* the figure). The ligand has a gauche conformation and links two Ag atoms using its two pyridinyl groups. In the structure of $\{[Ag(L)]\}_n$ linear one-dimensional chains

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

Atom	x	y	z	U_{iso}*/U_{eq}
B1	0.5000	0.1947(4)	0.2500	0.0355(9)
F1	0.49888(13)	0.1296(2)	0.3902(3)	0.0736(7)
F2	0.43400(13)	0.2507(2)	0.2252(3)	0.0808(7)
Ag1	0.0000	0.41148(2)	0.2500	0.03182(14)
O1	0.24415(10)	0.39577(13)	0.7796(2)	0.0287(4)
O2	0.35544(10)	0.38911(17)	0.6619(2)	0.0383(5)
C1	0.27884(13)	0.38919(17)	0.6561(3)	0.0228(4)
C2	0.39934(15)	0.3959(2)	0.8319(3)	0.0387(6)
H2A[a]	0.4034	0.3283	0.8880	0.046*
H2B[a]	0.3742	0.4437	0.9061	0.046*
H2C[a]	0.3759	0.3516	0.9142	0.046*
H2D[a]	0.3993	0.4668	0.8747	0.046*
C3[a]	0.4811(3)	0.4359(4)	0.7999(6)	0.0320(11)
H3A[a]	0.4762	0.5036	0.7442	0.038*
H3B[a]	0.5140	0.4431	0.9111	0.038*
C3′[a]	0.4817(3)	0.3616(5)	0.8148(6)	0.0302(10)
H3′1[a]	0.5120	0.3592	0.9298	0.036*
H3′2[a]	0.4811	0.2925	0.7643	0.036*
C11	0.24134(13)	0.38121(17)	0.4755(3)	0.0215(4)
C12	0.16232(13)	0.39673(17)	0.4456(3)	0.0236(4)
H12	0.1345	0.4101	0.5407	0.028*
N13	0.12357(12)	0.39359(15)	0.2868(3)	0.0265(4)
C14	0.16398(15)	0.37346(19)	0.1540(3)	0.0296(5)
H14	0.1374	0.3719	0.0410	0.036*
C15	0.24237(15)	0.35503(19)	0.1754(3)	0.0296(5)
H15	0.2688	0.3398	0.0788	0.036*
C16	0.28246(13)	0.35883(17)	0.3387(3)	0.0244(4)
H16	0.3365	0.3465	0.3563	0.029*

[a] Occupancy: 0.50.

are formed. Weak interactions are observed between F and Ag atoms. The most obvious difference between the ligand of the title compound and the uncoordinated ligand [3] is the angle between the planes of pyridine rings (24.73(11)° and 44.71(19)°) respectively, which is a consequence of the flexible organic components of the title compound. The C3 atom and H-atoms related to C2 atoms are disordered (site-occupancy factor of 0.5). The BF_4^- anion lies on an two-fold axis.

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References

1. Blake, A. J.; Champness, N. R.; Chung, S. S.; Li, W. S.; Schröder, M.: In situ ligand synthesis and construction of an unprecedented three-dimensional array with silver(I): a new approach to inorganic crystal engineering. Chem. Commun. (1997) 1675–1676.
2. Vallejos, J.; Brito, I.; Cárdenas, A.; Bolte, M.; Conejeros, S.; Alemany, P.; Llanos, J.: Self-Assembly of discrete metallocycles versus coordination polymers based on Cu(I) and Ag(I) ions and flexible ligands: structural diversification and luminescent properties. Polymers **8** (2016) 2–16.
3. Brito, I.; Vallejos, J.; López-Rodríguez, M.; Cárdenas, A.: Ethane-1,2-diylbis(pyridine-3-carboxylate). Acta Crystallogr. **E66** (2010) o114.
4. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
5. Stoe & Cie. X-AREA. Stoe & Cie, Darmstadt, Germany, 2001.
6. Spek, A. L.: Structure validation in chemical crystallography. Acta Crystallogr. **D65** (2009) 148–155.
7. Blessing, R. H.: An empirical correction for absorption anisotropy. Acta Crystallogr. **A51** (1995) 33–38.