Crystal structure of poly[(μ_3-di-2-pyrimidylsulfide-κ^3*S*:*N*,*N*′)-silver(I)] pentafluoropropionate, $C_{11}H_6AgF_5N_4O_2S$

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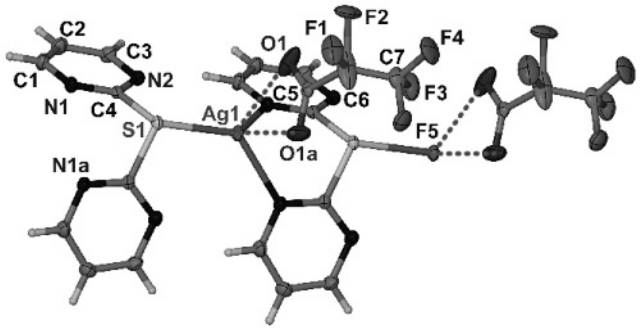

Abstract

$C_{11}H_6AgF_5N_4O_2S$, monoclinic, $P2_1/m$ (No. 11), a = 5.962(3) Å, b = 13.088(6) Å, c = 9.339(4) Å, β = 105.449(5)°, V = 702.4 Å^3, Z = 2, $R_{gt(F)}$ = 0.0214, $wR_{ref(F^2)}$ = 0.0556, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.15×0.20×0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	16.58 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII CCD, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6718, 1291
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1189
$N(param)_{refined}$:	133
Programs:	SHELX [5]

Source of material

All reagents and solvents from commercial sources were used without further purification. Di-2-pyrimidylsulfide (*DprS*) was synthesized according to reported procedures [1]. At room temperature, di-2-pyrimidylsulfide (*DprS*, 19 mg, 0.1 mmol) and $AgC_2F_5CO_2$ (27 mg, 0.1 mmol) were dissolved in 2 mL of methanol followed by addition of 3 mL of mixed solvent of acetonitrile and deionized water with stirring at room temperature. The colorless solution was filtered and then left to stand in air. After five days, colourless block-shaped crystals of the title compound were deposited.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. H atoms bonded to C atoms were positioned geometrically and allowed to ride on their parent atoms,with d(C–H) = 0.93 Å and U_{iso} (H) = 1.2 U_{eq} (C).

Discussion

Pyridyl-based building blocks are widely used in construction various supramolecules of transition metal complexes with distinct topological structures [2]. Among them, pyridyl sulfide derivatives are of great interest due to their versatile linkage behavior in coordination supramolecular assemblies [3, 4]. As a continuation work of the study on the coordination chemistry of di-2-pyrimidylsulfide [4], herein we report one new allomorphism of Ag(I) complex. In the mononuclear complex, the ligand takes a κ^3-*S*:*N*,*N*′-chelating mode with the pair of 2-pyrimidyl rings of each ligand exhibit a dihedral angle of 70.78(4)° (Figure). Each silver(I) center in the complex is surrounded by two N atoms from one di-2-pyrimidylsulfide ligand (Ag–N distance of 2.387(2) Å) and one S atom from another ligand (Ag–S distance of 2.510(2) Å). Furthermore, each Ag1 ion is weakly connected to O1 and O1a (symmetry code x, $-y$+0.5, z) atoms from one pentafluoropropionate anion with Ag···O distances of 2.655(3) Å. The mononuclear Ag(I) and a pair of the μ_3-bridging di-2-pyrimidyl sulfide ligands are alternately arranged and link together, forming a V-shaped chain-structure along *a* axis. The formed chains are stacked along *bc* plane and interconnect through weak C2–H2···O1b (symmetry code $-x$, $-y$, $-z$+1) interactions to form a three-dimensional framework, herein, the C2···O1b distance is 3.108(2) Å and the C2–H2···O1b angle equals 128.45°.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*f*	–0.5672	0.0427	0.3116	0.023
H(2A)	4*f*	–0.3586	–0.0667	0.2013	0.025
H(3A)	4*f*	0.0371	–0.0396	0.2445	0.021

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	2*e*		0.48014(4)	$\frac{1}{4}$	0.51798(3)	0.0098(2)	0.0186(2)	0.0161(2)	0	0.0047(1)	0
S(1)	2*e*		0.0653(1)	$\frac{1}{4}$	0.53168(9)	0.0096(4)	0.0152(4)	0.0141(4)	0	0.0029(3)	0
F(1)	2*e*		0.8022(4)	$\frac{1}{4}$	1.0959(2)	0.031(1)	0.072(2)	0.019(1)	0	0.012(1)	0
F(2)	4*f*	0.5	0.9401(6)	0.3755(2)	1.0148(3)	0.060(3)	0.011(2)	0.025(2)	0.002(2)	–0.004(2)	–0.008(1)
F(3)	2*e*		1.2488(4)	$\frac{1}{4}$	0.9196(3)	0.022(1)	0.060(2)	0.025(1)	0	0.008(1)	0
F(4)	2*e*		1.2803(4)	$\frac{1}{4}$	1.1558(2)	0.031(1)	0.057(2)	0.018(1)	0	–0.010(1)	0
F(5)	4*f*	0.5	1.1413(6)	0.1248(2)	1.0301(3)	0.027(2)	0.030(2)	0.032(2)	0.010(1)	0.001(2)	0.001(1)
O(1)	4*f*		0.6948(3)	0.1662(1)	0.7772(3)	0.028(1)	0.028(1)	0.081(2)	–0.0105(9)	0.029(1)	–0.025(1)
N(1)	4*f*		–0.3066(3)	0.1330(2)	0.4086(2)	0.0101(9)	0.017(1)	0.019(1)	–0.0001(8)	0.0045(8)	–0.0003(8)
N(2)	4*f*		0.0560(3)	0.0849(1)	0.3664(2)	0.014(1)	0.016(1)	0.018(1)	0.0018(8)	0.0068(8)	0.0023(8)
C(1)	4*f*		–0.4087(4)	0.0535(2)	0.3254(3)	0.012(1)	0.019(1)	0.027(1)	–0.002(1)	0.005(1)	0.000(1)
C(2)	4*f*		–0.2859(5)	–0.0125(2)	0.2600(3)	0.020(1)	0.017(1)	0.025(1)	–0.003(1)	0.005(1)	–0.003(1)
C(3)	4*f*		–0.0508(5)	0.0051(2)	0.2850(3)	0.019(1)	0.015(1)	0.021(1)	0.0014(9)	0.010(1)	–0.0013(9)
C(4)	4*f*		–0.0799(4)	0.1445(2)	0.4211(2)	0.013(1)	0.014(1)	0.012(1)	–0.0003(9)	0.002(1)	0.0032(9)
C(5)	2*e*		0.7475(5)	$\frac{1}{4}$	0.8329(4)	0.011(2)	0.018(2)	0.015(2)	0	0.007(1)	0
C(6)	2*e*		0.9036(9)	$\frac{1}{4}$	0.9917(5)	0.027(2)	0.141(6)	0.013(2)	0	0.007(2)	0
C(7)	4*f*	0.5	1.1455(9)	0.2261(4)	1.0254(5)	0.025(2)	0.029(7)	0.015(2)	–0.001(2)	0.005(2)	–0.000(2)

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