Crystal structure of di(4-aminopyridine)silver(I) trifluoromethylsulfate, $Ag(C_5H_6N_2)_2(CF_3SO_3)$

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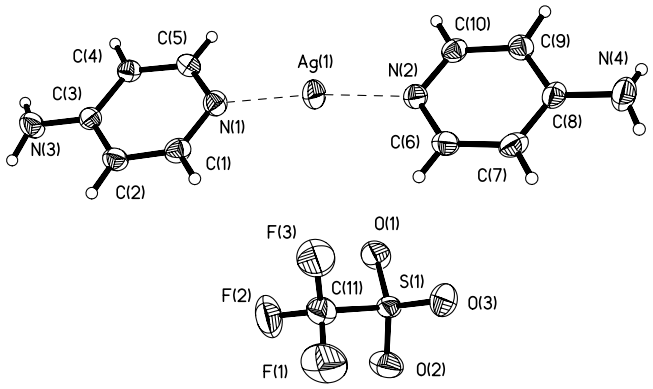

Abstract

$C_{11}H_{12}AgF_3N_4O_3S$, monoclinic, $P12_1/n1$ (No. 14), a = 6.901(1) Å, b = 11.106(2) Å, c = 20.958(4) Å, β = 94.799(3)°, V = 1600.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.033, $wR_{ref}(F^2)$ = 0.081, T = 293 K.

Source of material

The title compound was prepared by dissolving 4-aminopyridine (1 mmol, 94 mg) and silver(I) trifluoromethylsulfate (0.5 mmol, 128 mg) in acetonitrile. After keeping the mixture in air for three days, the colorless block crystals were formed. The products were isolated and washed with acetonitrile for three times and dried in a vacuum using $CaCl_2$ (yield: 59%). Reagents and solvents used were of commercially available reagent quality.

Experimental details

All H atoms were refined isotropically. The U_{aniso} values for O(1), O(2), O(3), F(1), F(2) and F(3) atoms are a little large, but we did not try to split them.

Discussion

The title compound is a mononuclear silver(I) complex. The central silver(I) atom is linearly coordinated by two nitrogen atoms from two different 4-aminopyridine ligands at the distances of d(Ag1—N1) = 2.114(2) Å and d(Ag1—N2) = 2.118(2) Å, which is quite close to both d(Ag—N) = 2.122(3) Å in the similar bis(4-aminopyridine)silver(I) nitrate (**I**) [3] and to d(Ag1—N1) = 2.111(4) Å /d(Ag1—N3) = 2.119(4) Å in the similar bis(4-aminopyridine)silver(I) trifluoroacetate (**II**) [4]. The bond angle of Ag(1) is close to 180° (∠(N–Ag–N) = 173.47(8)°), indicating a slightly distorted linear coordination of Ag(1). This angle is similar to that in **I** [3] (174.43(14)°), but much larger than that in **II** [4] (166.51(15)°). The two aromatic rings joined by Ag(1) are nearly parallel, the diahedral angle is 4.6(1)°. Weak interactions between silver(I) atoms and a great number of hydrogen bonds join the complex into a three-dimensional network.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.22 × 0.28 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.39 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, φ/ω
$2\theta_{max}$:	56.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9921, 3921
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3183
$N(param)_{refined}$:	256
Programs:	SHELXTL [3], SHELXTL-plus [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.368(4)	0.302(3)	0.013(1)	0.051(8)
H(2)	4*e*	0.408(5)	0.451(3)	–0.058(2)	0.060(9)
H(4)	4*e*	0.354(4)	0.209(3)	–0.199(1)	0.056(8)
H(5)	4*e*	0.302(4)	0.072(3)	–0.124(1)	0.037(7)
H(6)	4*e*	0.200(6)	0.027(3)	0.159(2)	0.08(1)
H(7)	4*e*	0.162(5)	–0.096(3)	0.241(2)	0.068(9)
H(9)	4*e*	0.120(5)	–0.371(3)	0.113(2)	0.07(1)
H(10)	4*e*	0.178(4)	–0.230(3)	0.040(1)	0.052(8)
H(1N3)	4*e*	0.387(5)	0.417(3)	–0.222(2)	0.08(1)
H(1N4)	4*e*	0.038(6)	–0.394(4)	0.220(2)	0.09(2)
H(2N3)	4*e*	0.405(5)	0.514(3)	–0.170(2)	0.07(1)
H(2N4)	4*e*	0.056(5)	–0.296(3)	0.266(2)	0.06(1)

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

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	4*e*	0.26799(4)	0.03375(2)	0.01789(1)	0.0683(2)	0.0545(1)	0.0494(1)	0.0038(1)	0.0091(1)	0.0176(1)
S(1)	4*e*	–0.2518(1)	0.21855(6)	0.12993(3)	0.0734(5)	0.0421(3)	0.0345(3)	0.0049(3)	0.0070(3)	–0.0049(2)
O(1)	4*e*	–0.2714(5)	0.1574(2)	0.0703(1)	0.154(3)	0.062(1)	0.042(1)	–0.008(2)	0.008(1)	–0.014(1)
O(2)	4*e*	–0.4074(4)	0.3005(2)	0.1401(1)	0.067(2)	0.062(1)	0.112(2)	0.005(1)	0.015(1)	–0.017(1)
O(3)	4*e*	–0.1952(4)	0.1432(2)	0.1836(1)	0.120(2)	0.080(2)	0.045(1)	0.008(2)	0.009(1)	0.016(1)
N(1)	4*e*	0.3283(3)	0.1695(2)	–0.0483(1)	0.048(1)	0.047(1)	0.044(1)	0.003(1)	0.0026(9)	0.0067(9)
N(2)	4*e*	0.1988(3)	–0.0862(2)	0.0912(1)	0.048(1)	0.046(1)	0.040(1)	0.006(1)	0.0053(9)	0.0043(9)
N(3)	4*e*	0.4126(4)	0.4381(2)	–0.1816(1)	0.092(2)	0.042(1)	0.043(1)	0.001(1)	0.012(1)	0.004(1)
N(4)	4*e*	0.0878(5)	–0.3277(4)	0.2356(2)	0.078(2)	0.081(2)	0.054(2)	0.007(2)	0.019(2)	0.023(2)
F(1)	4*e*	–0.0087(4)	0.3829(3)	0.1754(2)	0.103(2)	0.117(2)	0.138(2)	–0.033(2)	–0.008(2)	–0.048(2)
F(2)	4*e*	–0.0716(4)	0.3910(2)	0.0753(1)	0.125(2)	0.080(2)	0.140(2)	–0.006(1)	0.028(2)	0.047(2)
F(3)	4*e*	0.1138(4)	0.2546(2)	0.1150(2)	0.081(2)	0.103(2)	0.192(3)	0.028(1)	0.054(2)	0.024(2)
C(1)	4*e*	0.3604(4)	0.2844(3)	–0.0306(1)	0.054(2)	0.055(2)	0.036(1)	0.004(1)	0.005(1)	–0.002(1)
C(2)	4*e*	0.3889(4)	0.3754(2)	–0.0727(1)	0.057(2)	0.039(1)	0.043(1)	0.002(1)	0.005(1)	–0.005(1)
C(3)	4*e*	0.3850(4)	0.3514(2)	–0.1383(1)	0.042(1)	0.041(1)	0.040(1)	0.004(1)	0.005(1)	0.003(1)
C(4)	4*e*	0.3535(4)	0.2314(2)	–0.1567(1)	0.045(1)	0.043(1)	0.036(1)	0.005(1)	0.002(1)	–0.001(1)
C(5)	4*e*	0.3258(4)	0.1465(2)	–0.1113(1)	0.045(1)	0.038(1)	0.045(1)	0.003(1)	–0.000(1)	–0.000(1)
C(6)	4*e*	0.1886(5)	–0.0511(3)	0.1521(1)	0.060(2)	0.045(2)	0.047(1)	0.008(1)	0.008(1)	–0.003(1)
C(7)	4*e*	0.1544(4)	–0.1266(3)	0.2006(1)	0.053(2)	0.065(2)	0.037(1)	0.014(1)	0.008(1)	–0.003(1)
C(8)	4*e*	0.1237(4)	–0.2485(3)	0.1885(1)	0.035(1)	0.060(2)	0.044(1)	0.010(1)	0.008(1)	0.013(1)
C(9)	4*e*	0.1345(4)	–0.2858(3)	0.1254(1)	0.045(2)	0.047(1)	0.051(1)	0.002(1)	0.004(1)	0.001(1)
C(10)	4*e*	0.1705(4)	–0.2038(2)	0.0794(1)	0.046(2)	0.055(2)	0.036(1)	0.006(1)	0.004(1)	–0.001(1)
C(11)	4*e*	–0.0433(5)	0.3158(3)	0.1236(2)	0.069(2)	0.058(2)	0.083(2)	0.008(2)	0.015(2)	0.001(2)

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