Crystal structure of bis-5-(pyridin-2-yl)pyrazine-2-carboxylic acid silver(I) triflate – acetonitrile (1:2), $[Ag(C_{10}H_7N_3O_2)_2](CF_3SO_3)\cdot 2CH_3CN$, $C_{24}H_{20}AgF_3N_8O_7S_2$

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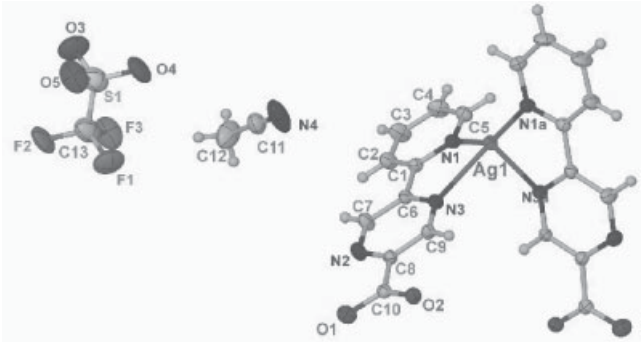

Abstract

$C_{24}H_{20}AgF_3N_8O_7S_2$, monoclinic, *C*2/*c* (no. 15), $a = 31.864(5)$ Å, $b = 6.7191(10)$ Å, $c = 14.052(2)$ Å, $\beta = 97.006(3)°$, $V = 2986.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0606$, $wR_{ref}(F^2) = 0.1637$, $T = 293$ K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.3×0.3×0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.91 cm^{-1}
Diffractometer, scan mode:	Bruker APEX II CCD, ω
$2\theta_{max}$:	49.99°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7732, 2616
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1922
$N(param)_{refined}$:	217
Programs:	SHELX [6], PLATON [7]

Source of material

The starting material 5-(2-pyridyl)-2-cyanopyrazine was obtained commercially. To a mixed solvent of 4 ml acetonitrile and deionized water the 5-(2-pyridyl)-2-cyanopyrazine (18.2 mg, 0.1 mmol) and $AgCF_3SO_3$ (26 mg, 0.1 mmol) were added with stirring at room temperature for one day. A cloudy solution resulted which was heated, and then filtrated. The clear filtrate was kept in air for one week at room temperature to yield colourless block-shaped crystals (25.1 mg, 66% yield). The 5-(pyridin-2-yl)pyrazine-2-carboxylate ligand of the title complex is believed to be the in-situ hydrated product.

Experimental details

The triflate is disordered with the O atoms and F atoms having equal positional occupanies at the same positions, respectively. All H atoms were in the difference electron density maps. Nevertheless, the hydrogen atoms were placed into idealized positions and allowed to ride on the carrier atoms. $U_{iso}(H) = 1.2U_{eq}(C)_{aryl}$.

Discussion

Carboxylate-, pyridyl- and triazole-based ligands have been widely employed to link metal ions to construct subtile metal organic frameworks (MOFs) [1, 2]. A numerous 2,2'-bipyridine derivatives together with their various metal complexes have also been synthesized and well characterized [3, 4]. Herein, we report the structure of a new mononuclear silver(I) complex using a ligand containing a 2,2'-bipyridine fragment with an additional 2-carboxylate acid group bonded to the 2-pyridyl carbon atom. As shown in the figure, the two ligands around the central Ag(I) ion are in a *syn*-relationship with four N atoms coordinating the Ag(I) center, forming a distorted N4-coordination geometry. The 2-pyridyl N1 atoms and the 2-pyrazinyl N3 atoms exhibit the Ag1–N1 bond lengths of 2.207(7) and 2.432(7) Å, respectively. The Ag–N bond lengths are similar to those (2.196(2)-2.685(2) Å) in the mononuclear structure of $[Ag(C_{10}H_6N_4)_2]BF_4$ derived from 5-(2-pyridyl)-2-cyanopyrazine [5]. Along the *b* axis, the mononuclear units are connected through $\pi\cdots\pi$ interactions between N3-containg pyrazinyl ring and N1^{iii}-containing pyridyl ring with the centroid···centroid distance of 3.933(5) Å, leading to an infinite chain motif. The formed chains are stacked along the *c* direction (iii: $x, y+1, z$). Except for the O–H···O interactions between the carboxylate acide (D···A = 2.110(5) Å, D–H···A 179°) to connect the upper and nether neighbouring chains together. The triflate anions and the acetonitrile molecules are embedded in the structure without strong bonding interactions to their neighbours.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	8*f*	–0.0383	0.9427	–0.0479	0.111
H(2B)	8*f*	–0.1538	0.0213	0.0767	0.069
H(3A)	8*f*	–0.1797	–0.2591	0.1424	0.086
H(4A)	8*f*	–0.1378	–0.4300	0.2593	0.078
H(5A)	8*f*	–0.0703	–0.3181	0.3057	0.061
H(7A)	8*f*	–0.1251	0.2702	0.0146	0.066
H(9A)	8*f*	0.0007	0.4733	0.1594	0.051
H(12A)	8*f*	–0.221(1)	–0.13(2)	–0.264(3)	0.3(1)
H(12C)	8*f*	–0.229(1)	–0.287(6)	–0.196(7)	0.21(8)
H(12B)	8*f*	–0.231(1)	–0.08(1)	–0.170(5)	0.13(5)

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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)	4*e*		0	–0.0030(1)	$\frac{1}{4}$	0.0339(4)	0.0428(4)	0.0492(4)	0	–0.0018(3)	0
O(1)	8*f*		–0.0509(2)	0.8398(8)	–0.0377(4)	0.077(4)	0.063(3)	0.082(4)	–0.001(3)	0.012(3)	0.010(3)
O(2)	8*f*		0.0061(2)	0.7909(6)	0.0742(3)	0.050(3)	0.050(3)	0.054(3)	–0.009(2)	–0.001(2)	0.004(2)
N(1)	8*f*		–0.0709(2)	–0.0874(8)	0.2207(4)	0.036(3)	0.037(3)	0.050(3)	–0.002(2)	0.006(2)	0.001(2)
N(2)	8*f*		–0.0843(2)	0.4851(8)	0.0145(4)	0.045(3)	0.054(4)	0.064(4)	–0.003(3)	–0.006(3)	0.013(3)
N(3)	8*f*		–0.0406(2)	0.2598(7)	0.1592(4)	0.038(3)	0.040(3)	0.049(3)	–0.003(2)	–0.002(2)	0.005(2)
C(1)	8*f*		–0.0963(2)	0.0141(9)	0.1545(4)	0.035(3)	0.041(3)	0.048(3)	–0.002(3)	0.006(3)	–0.004(3)
C(2)	8*f*		–0.1370(2)	–0.049(1)	0.1238(6)	0.043(4)	0.051(5)	0.077(5)	–0.005(3)	–0.004(4)	0.005(3)
C(3)	8*f*		–0.1523(2)	–0.216(1)	0.1628(7)	0.044(4)	0.060(5)	0.108(7)	–0.019(4)	–0.003(4)	0.006(5)
C(4)	8*f*		–0.1277(2)	–0.317(1)	0.2312(6)	0.056(5)	0.044(4)	0.097(6)	–0.015(4)	0.013(4)	0.009(4)
C(5)	8*f*		–0.0872(2)	–0.248(1)	0.2584(5)	0.050(4)	0.045(4)	0.058(4)	0.000(3)	0.009(3)	0.006(3)
C(6)	8*f*		–0.0785(2)	0.2006(9)	0.1177(4)	0.035(3)	0.039(3)	0.044(3)	0.001(3)	0.002(3)	–0.001(3)
C(7)	8*f*		–0.0992(2)	0.315(1)	0.0445(5)	0.037(4)	0.055(4)	0.070(5)	–0.006(3)	–0.010(3)	0.011(4)
C(8)	8*f*		–0.0471(2)	0.5450(8)	0.0602(4)	0.043(4)	0.038(4)	0.037(3)	0.002(3)	0.006(3)	–0.001(2)
C(9)	8*f*		–0.0256(2)	0.4298(9)	0.1307(4)	0.043(4)	0.037(3)	0.044(4)	–0.010(3)	–0.004(3)	–0.002(3)
C(10)	8*f*		–0.0284(2)	0.7378(9)	0.0325(4)	0.044(4)	0.040(4)	0.041(3)	0.007(3)	0.009(3)	0.002(3)
S(1)	8*f*	0.5	–0.2674(1)	–0.1557(6)	–0.4929(3)	0.115(3)	0.117(3)	0.183(4)	0.003(2)	–0.018(3)	0.008(3)
C(13)	8*f*	0.5	–0.2674(1)	–0.1557(6)	–0.4929(3)	0.115(3)	0.117(3)	0.183(4)	0.003(2)	–0.018(3)	0.008(3)
O(3)	8*f*	0.5	–0.2420(3)	0.0138(9)	–0.4600(8)	0.138(6)	0.088(5)	0.211(9)	–0.045(4)	–0.013(6)	–0.034(5)
F(1)	8*f*	0.5	–0.2420(3)	0.0138(9)	–0.4600(8)	0.138(6)	0.088(5)	0.211(9)	–0.045(4)	–0.013(6)	–0.034(5)
O(4)	8*f*	0.5	–0.2918(2)	–0.114(1)	–0.5798(6)	0.097(5)	0.145(6)	0.155(6)	0.029(4)	–0.060(4)	0.042(5)
F(2)	8*f*	0.5	–0.2918(2)	–0.114(1)	–0.5798(6)	0.097(5)	0.145(6)	0.155(6)	0.029(4)	–0.060(4)	0.042(5)
O(5)	8*f*	0.5	–0.2934(2)	–0.204(1)	–0.4199(6)	0.101(5)	0.213(8)	0.160(7)	0.007(5)	0.068(5)	0.060(6)
F(3)	8*f*	0.5	–0.2934(2)	–0.204(1)	–0.4199(6)	0.101(5)	0.213(8)	0.160(7)	0.007(5)	0.068(5)	0.060(6)
C(11)	8*f*		–0.1732(3)	–0.174(2)	–0.1657(7)	0.075(7)	0.091(7)	0.081(6)	0.002(5)	–0.010(5)	0.007(5)
N(4)	8*f*		–0.1396(3)	–0.173(2)	–0.1409(7)	0.089(7)	0.158(9)	0.138(9)	–0.021(6)	–0.022(6)	0.069(7)
C(12)	8*f*		–0.2154(4)	–0.167(3)	–0.199(1)	0.073(8)	0.18(2)	0.14(1)	0.001(9)	–0.005(7)	–0.05(1)

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