metal-organic compounds

catena-Poly[[(2-amino-5-chlorobenzene-sulfonato-κ²N,O)silver(I)]-μ-2,5-di-methylpyrazine-κ²N:N′]

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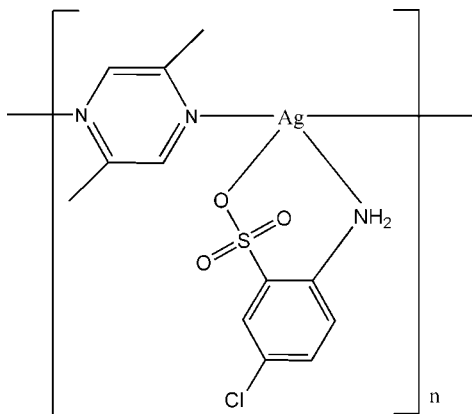Correspondence e-mail: yangjinnenu@yahoo.com.cn

The title compound, $[Ag(C_6H_5ClNO_3S)(C_6H_8N_2)]_n$, has a chain structure, where the Ag^{I} cation is four-coordinated by three N atoms from two different 2,5-dimethylpyrazine molecules and an NH$_2$ group of a 2-amino-5-chlorobenzene-sulfonate anion, and one sulfonate O atom. N—H⋯O hydrogen bonds stabilize the structure.

Related literature

For studies on silver sulfonates, see Liu *et al.* (2007).

For related literature, see: Cote & Shimizu (2003); Li *et al.* (2006).

Experimental

Crystal data

$[Ag(C_6H_5ClNO_3S)(C_6H_8N_2)]$
M_r = 422.63
Orthorhombic, $Pna2_1$
a = 14.671 (3) Å
b = 11.947 (2) Å
c = 8.2025 (16) Å

V = 1437.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.75 mm^{-1}
T = 293 (2) K
0.32 × 0.23 × 0.06 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.653, T_{max} = 0.906

11475 measured reflections
2935 independent reflections
2638 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.053
S = 1.05
2935 reflections
200 parameters
4 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 1172 Friedel pairs
Flack parameter: −0.03 (3)

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—HN1⋯O1^{i}	0.841 (10)	2.146 (12)	2.968 (3)	165 (3)
N1—HN2⋯O2	0.840 (10)	2.34 (3)	2.890 (4)	124 (3)

Symmetry code: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2528).

References

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supplementary materials

catena-Poly[[(2-amino-5-chlorobenzenesulfonato-*κ²N,O*)silver(I)]-*μ*-2,5-dimethylpyrazine-*κ²N:N'*]

H.-Y. Liu, J.-C. Ma and J. Yang

Comment

The design and synthesis of silver(I) sulfonates have attracted intense interests of chemists (Cote & Shimizu, 2003). Generally, the structure motifs of most silver(I) sulfonates observed is a two-dimensional layer, which is similar to that of metal phosphonates (Liu *et al.*, 2007). So far, some silver(I) sulfonate compounds modified by nitrogen-based secondary ligands have been reported (Li *et al.*, 2006). Herein, we present a new sulfonate coordination polymer, namely [Ag(dmp)(*L*)] where dmp = 2,5-dimethylpyrazine and HL= 2-amino-5-chlorobenzenesulfonic acid.

In the title compound the Ag^I cation is four-coordinated by three N atoms from two different 2,5-dimethylprazine molecules and a –NH$_2$ group of 2-amino-5-chlorobenzenesulfonate anion, and one sulfonate O atom (Fig. 1). The Ag—O (sulfonate) distance is near to that in a related compound (Liu *et al.*, 2007). The dmp ligand links two neighboring Ag^I atoms, forming a chain structure. The *L* ligands are attached on one side of the chains in a chelating mode. Finally, the molecules are linked through N—H···O hydrogen bonds (Table 1).

Experimental

An aqueous solution (8 ml) of 2-amino-5-chlorobenzenesulfonic acid (1 mmol) was added to solid Ag$_2$CO$_3$ (0.5 mmol) and stirred for several minutes until no further CO$_2$ was given off. The 2,5-dimethylprazine (1 mmol) was then added and a precipitate was formed. The precipitate was dissolved by ammonium hydroxide. Crystals were obtained by evaporation of the solution for several days at room temperature.

Refinement

H atoms bonded to N atom were located in a difference map and refined with distance restraints of N–H = 0.85±0.01 Å and H···H = 1.3±0.01 Å, and with U_{iso}(H) = 1.2U_{eq}(N). H atoms of C atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding, with U_{iso}(H)=1.2U_{eq}(C). The methyl groups were allowed to rotate but not to tip.
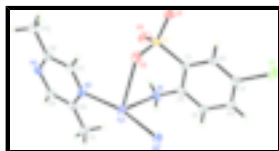
Figures

Fig. 1. The structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code: (i) 0.5 − *x*, *y* − 1/2, 1/2 + *z*.

supplementary materials

Fig. 2. View of the chain structure.

***catena*-Poly[[(2-amino-5-chlorobenzenesulfonato-κ^2N,O)silver(I)]-µ- 2,5-dimethylpyrazine-$\kappa^2N:N'$]**

Crystal data

[Ag(C$_6$H$_5$ClNO$_3$S)(C$_6$H$_8$N$_2$)]	$F_{000} = 840$
$M_r = 422.63$	$D_x = 1.953$ Mg m^{-3}
Orthorhombic, *Pna*2$_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 10124 reflections
$a = 14.671$ (3) Å	$\theta = 3.0–27.5º$
$b = 11.947$ (2) Å	$\mu = 1.75$ mm^{-1}
$c = 8.2025$ (16) Å	$T = 293$ (2) K
$V = 1437.7$ (5) Å^3	Platelet, colorless
$Z = 4$	$0.32 \times 0.23 \times 0.06$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer	2935 independent reflections
Radiation source: fine-focus sealed tube	2638 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{max} = 27.5º$
$T = 293(2)$ K	$\theta_{min} = 3.0º$
ω scans	$h = -18 \rightarrow 19$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -15 \rightarrow 15$
$T_{min} = 0.653$, $T_{max} = 0.906$	$l = -10 \rightarrow 8$
11475 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.025$	$w = 1/[\sigma^2(F_o^2) + (0.0213P)^2 + 0.3336P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.053$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
2935 reflections	$\Delta\rho_{min} = -0.33$ e Å^{-3}
200 parameters	Extinction correction: none
4 restraints	Absolute structure: Flack (1983), 1172 Friedel pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: −0.03 (3)

Secondary atom site location: difference Fourier map

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.243272 (14)	0.646505 (19)	0.76476 (5)	0.03722 (8)
S1	0.07812 (5)	0.76842 (6)	1.01266 (12)	0.03172 (17)
Cl1	0.08916 (7)	0.48754 (8)	1.52741 (16)	0.0547 (3)
O3	0.08442 (16)	0.7030 (2)	0.8637 (3)	0.0456 (6)
O1	−0.01398 (14)	0.77287 (18)	1.0795 (3)	0.0389 (6)
N2	0.24774 (17)	0.7932 (2)	0.5917 (4)	0.0280 (6)
C10	0.3268 (2)	0.8382 (3)	0.5426 (4)	0.0305 (7)
N1	0.28661 (17)	0.7479 (2)	1.0269 (4)	0.0348 (6)
O2	0.12086 (18)	0.87723 (19)	1.0001 (4)	0.0523 (8)
C1	0.14351 (19)	0.6928 (2)	1.1575 (4)	0.0261 (6)
C6	0.23964 (19)	0.6899 (3)	1.1469 (4)	0.0291 (7)
C7	0.1702 (2)	0.8401 (3)	0.5413 (4)	0.0312 (7)
H7	0.1152	0.8104	0.5772	0.037*
C2	0.09859 (17)	0.6303 (2)	1.2728 (6)	0.0287 (6)
H2	0.0353	0.6322	1.2789	0.034*
C3	0.1481 (2)	0.5643 (3)	1.3803 (4)	0.0316 (7)
C4	0.2415 (2)	0.5588 (3)	1.3718 (5)	0.0355 (8)
H4	0.2737	0.5136	1.4439	0.043*
C5	0.28739 (18)	0.6210 (2)	1.2549 (7)	0.0340 (7)
H5	0.3506	0.6169	1.2485	0.041*
C11	0.4135 (2)	0.7864 (3)	0.5991 (6)	0.0501 (10)
H11A	0.4174	0.7917	0.7157	0.075*
H11B	0.4641	0.8250	0.5508	0.075*
H11C	0.4148	0.7091	0.5673	0.075*
C8	0.1686 (2)	0.9322 (3)	0.4367 (4)	0.0280 (7)
N3	0.24766 (18)	0.9781 (2)	0.3873 (4)	0.0288 (6)
C12	0.0810 (2)	0.9820 (3)	0.3801 (6)	0.0438 (9)
H12A	0.0731	1.0544	0.4291	0.066*
H12B	0.0314	0.9342	0.4113	0.066*
H12C	0.0821	0.9896	0.2636	0.066*

C9	0.3255 (2)	0.9311 (3)	0.4415 (4)	0.0335 (8)
H9	0.3807	0.9624	0.4097	0.040*
HN1	0.3438 (7)	0.752 (2)	1.029 (4)	0.027 (8)*
HN2	0.272 (2)	0.8154 (13)	1.015 (8)	0.070 (16)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.04065 (13)	0.03172 (11)	0.03929 (14)	0.00103 (10)	0.0046 (2)	0.01531 (14)
S1	0.0169 (3)	0.0350 (4)	0.0432 (5)	0.0006 (3)	0.0003 (4)	0.0113 (4)
Cl1	0.0565 (6)	0.0521 (5)	0.0554 (6)	0.0058 (4)	0.0073 (5)	0.0249 (5)
O3	0.0299 (13)	0.0725 (19)	0.0345 (14)	0.0059 (12)	−0.0044 (11)	0.0053 (13)
O1	0.0198 (10)	0.0398 (13)	0.0572 (18)	0.0037 (9)	0.0060 (10)	0.0130 (11)
N2	0.0311 (13)	0.0232 (13)	0.0299 (15)	−0.0038 (11)	0.0025 (12)	0.0020 (11)
C10	0.0270 (15)	0.0314 (17)	0.0332 (19)	−0.0036 (12)	−0.0011 (14)	0.0031 (13)
N1	0.0170 (12)	0.0399 (17)	0.0477 (19)	−0.0027 (11)	0.0030 (14)	−0.0003 (15)
O2	0.0325 (13)	0.0378 (15)	0.087 (2)	−0.0034 (10)	0.0017 (15)	0.0254 (15)
C1	0.0207 (14)	0.0237 (15)	0.0340 (18)	0.0016 (12)	−0.0033 (13)	−0.0013 (12)
C6	0.0209 (14)	0.0313 (16)	0.0353 (18)	−0.0039 (13)	0.0010 (14)	−0.0038 (13)
C7	0.0286 (16)	0.0260 (16)	0.039 (2)	−0.0036 (13)	0.0015 (15)	0.0016 (13)
C2	0.0229 (11)	0.0259 (13)	0.0374 (17)	−0.0007 (10)	0.000 (2)	−0.0027 (15)
C3	0.0365 (17)	0.0261 (16)	0.0322 (18)	0.0014 (13)	−0.0031 (15)	0.0021 (13)
C4	0.0373 (18)	0.0335 (18)	0.0358 (19)	0.0115 (14)	−0.0049 (16)	−0.0030 (15)
C5	0.0207 (12)	0.0389 (16)	0.0424 (18)	0.0078 (11)	−0.008 (2)	−0.007 (2)
C11	0.0356 (19)	0.052 (2)	0.063 (3)	0.0027 (16)	−0.0062 (19)	0.0296 (19)
C8	0.0298 (15)	0.0229 (16)	0.0314 (17)	0.0013 (13)	−0.0028 (13)	−0.0026 (12)
N3	0.0326 (14)	0.0246 (13)	0.0293 (15)	−0.0002 (11)	0.0032 (12)	0.0032 (11)
C12	0.0335 (18)	0.038 (2)	0.060 (3)	0.0036 (15)	−0.0027 (18)	0.0071 (17)
C9	0.0277 (15)	0.0351 (19)	0.038 (2)	−0.0035 (14)	0.0037 (15)	0.0085 (14)

Geometric parameters (Å, °)

Ag1—N3[i]	2.253 (3)	C7—C8	1.396 (4)
Ag1—N2	2.256 (3)	C7—H7	0.9300
Ag1—N1	2.549 (3)	C2—C3	1.388 (5)
Ag1—O3	2.559 (2)	C2—H2	0.9300
S1—O2	1.447 (2)	C3—C4	1.372 (5)
S1—O3	1.453 (3)	C4—C5	1.388 (6)
S1—O1	1.459 (2)	C4—H4	0.9300
S1—C1	1.774 (3)	C5—H5	0.9300
Cl1—C3	1.746 (3)	C11—H11A	0.9600
N2—C7	1.334 (4)	C11—H11B	0.9600
N2—C10	1.341 (4)	C11—H11C	0.9600
C10—C9	1.385 (4)	C8—N3	1.345 (4)
C10—C11	1.488 (5)	C8—C12	1.491 (4)
N1—C6	1.387 (5)	N3—C9	1.347 (4)
N1—HN1	0.841 (10)	N3—Ag1[ii]	2.253 (3)
N1—HN2	0.840 (10)	C12—H12A	0.9600

C1—C2	1.374 (5)	C12—H12B	0.9600
C1—C6	1.413 (4)	C12—H12C	0.9600
C6—C5	1.397 (6)	C9—H9	0.9300
N3^{i}—Ag1—N2	166.54 (11)	C1—C2—C3	119.7 (3)
N3^{i}—Ag1—N1	91.92 (10)	C1—C2—H2	120.2
N2—Ag1—N1	98.89 (10)	C3—C2—H2	120.2
N3^{i}—Ag1—O3	98.52 (9)	C4—C3—C2	121.2 (3)
N2—Ag1—O3	91.20 (9)	C4—C3—Cl1	120.3 (3)
N1—Ag1—O3	80.45 (8)	C2—C3—Cl1	118.5 (3)
O2—S1—O3	113.29 (18)	C3—C4—C5	119.6 (3)
O2—S1—O1	113.28 (15)	C3—C4—H4	120.2
O3—S1—O1	113.24 (15)	C5—C4—H4	120.2
O2—S1—C1	105.71 (16)	C4—C5—C6	120.7 (3)
O3—S1—C1	104.77 (15)	C4—C5—H5	119.7
O1—S1—C1	105.53 (15)	C6—C5—H5	119.7
S1—O3—Ag1	117.81 (13)	C10—C11—H11A	109.5
C7—N2—C10	118.4 (3)	C10—C11—H11B	109.5
C7—N2—Ag1	119.8 (2)	H11A—C11—H11B	109.5
C10—N2—Ag1	121.7 (2)	C10—C11—H11C	109.5
N2—C10—C9	119.2 (3)	H11A—C11—H11C	109.5
N2—C10—C11	118.6 (3)	H11B—C11—H11C	109.5
C9—C10—C11	122.1 (3)	N3—C8—C7	119.5 (3)
C6—N1—Ag1	103.7 (2)	N3—C8—C12	119.2 (3)
C6—N1—HN1	120 (2)	C7—C8—C12	121.4 (3)
Ag1—N1—HN1	107 (2)	C8—N3—C9	117.5 (3)
C6—N1—HN2	116 (4)	C8—N3—Ag1^{ii}	123.3 (2)
Ag1—N1—HN2	107 (4)	C9—N3—Ag1^{ii}	118.0 (2)
HN1—N1—HN2	102.0 (15)	C8—C12—H12A	109.5
C2—C1—C6	120.5 (3)	C8—C12—H12B	109.5
C2—C1—S1	118.6 (2)	H12A—C12—H12B	109.5
C6—C1—S1	120.8 (3)	C8—C12—H12C	109.5
N1—C6—C5	119.7 (3)	H12A—C12—H12C	109.5
N1—C6—C1	121.8 (3)	H12B—C12—H12C	109.5
C5—C6—C1	118.4 (3)	N3—C9—C10	122.9 (3)
N2—C7—C8	122.4 (3)	N3—C9—H9	118.5
N2—C7—H7	118.8	C10—C9—H9	118.5
C8—C7—H7	118.8		
O2—S1—O3—Ag1	63.33 (19)	Ag1—N1—C6—C1	−67.9 (3)
O1—S1—O3—Ag1	−165.89 (13)	C2—C1—C6—N1	176.9 (3)
C1—S1—O3—Ag1	−51.40 (18)	S1—C1—C6—N1	1.8 (5)
N3^{i}—Ag1—O3—S1	92.60 (17)	C2—C1—C6—C5	0.8 (5)
N2—Ag1—O3—S1	−96.73 (17)	S1—C1—C6—C5	−174.3 (3)
N1—Ag1—O3—S1	2.08 (16)	C10—N2—C7—C8	1.6 (5)
N3^{i}—Ag1—N2—C7	113.6 (4)	Ag1—N2—C7—C8	178.8 (2)
N1—Ag1—N2—C7	−103.3 (2)	C6—C1—C2—C3	0.4 (5)
O3—Ag1—N2—C7	−22.8 (3)	S1—C1—C2—C3	175.6 (3)
N3^{i}—Ag1—N2—C10	−69.3 (5)	C1—C2—C3—C4	−1.1 (5)

N1—Ag1—N2—C10	73.8 (3)	C1—C2—C3—Cl1	179.1 (3)
O3—Ag1—N2—C10	154.3 (3)	C2—C3—C4—C5	0.7 (5)
C7—N2—C10—C9	−0.2 (5)	Cl1—C3—C4—C5	−179.5 (3)
Ag1—N2—C10—C9	−177.4 (2)	C3—C4—C5—C6	0.5 (6)
C7—N2—C10—C11	179.7 (3)	N1—C6—C5—C4	−177.4 (3)
Ag1—N2—C10—C11	2.6 (4)	C1—C6—C5—C4	−1.3 (5)
N3ⁱ—Ag1—N1—C6	−40.2 (2)	N2—C7—C8—N3	−1.8 (5)
N2—Ag1—N1—C6	147.8 (2)	N2—C7—C8—C12	179.0 (3)
O3—Ag1—N1—C6	58.1 (2)	C7—C8—N3—C9	0.5 (5)
O2—S1—C1—C2	136.8 (3)	C12—C8—N3—C9	179.7 (3)
O3—S1—C1—C2	−103.3 (3)	C7—C8—N3—Ag1ⁱⁱ	−166.4 (2)
O1—S1—C1—C2	16.5 (3)	C12—C8—N3—Ag1ⁱⁱ	12.8 (4)
O2—S1—C1—C6	−48.0 (3)	C8—N3—C9—C10	0.9 (5)
O3—S1—C1—C6	71.9 (3)	Ag1ⁱⁱ—N3—C9—C10	168.5 (3)
O1—S1—C1—C6	−168.3 (3)	N2—C10—C9—N3	−1.1 (5)
Ag1—N1—C6—C5	108.1 (3)	C11—C10—C9—N3	179.0 (4)

Symmetry codes: (i) −x+1/2, y−1/2, z+1/2; (ii) −x+1/2, y+1/2, z−1/2.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—HN1···O1ⁱⁱⁱ	0.841 (10)	2.146 (12)	2.968 (3)	165 (3)
N1—HN2···O2	0.840 (10)	2.34 (3)	2.890 (4)	124 (3)

Symmetry codes: (iii) x+1/2, −y+3/2, z.

Fig. 1

Fig. 2