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Crystal structure of poly[(μ_2 -4-amino-1,5-naphthalenedisulfonato- κ^4 O, $N:O'$, N')bis(μ_2 -hexamethylenetetramino- $\kappa^2 N;N'$)silver(I)], $\{C_{22}H_{30}Ag_2N_9O_6S_2\}_n$

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Abstract

$C_{22}H_{30}Ag_2N_9O_6S_2$, monoclinic, $P2_1/c$ (no. 14), $a = 10.4629(10)$ Å, $b = 10.9785(8)$ Å, $c = 12.3483(15)$ Å, $\beta = 109.766(12)^\circ$, $V = 1334.8(2)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0662$, $wR_{ref}(F^2) = 0.1356$, $T = 293(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixed solution of Ag_2CO_3 (0.2757 g, 1 mmol) and 4-amino-1,5-naphthalene disulfonic acid (0.1562 g, 0.5 mmol) in H_2O/DMF (3:2 v/v) reacted for several minutes. An aqueous solution of ammonia was added in order to make it clear.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.21 \times 0.15 \times 0.13$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.68 mm^{-1}
Diffractometer, scan mode:	XtaLAB Pro, ω
$\theta_{\max}$, completeness:	29.7° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12927, 3348, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2804
$N(\text{param})_{\text{refined}}$:	190
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4], Diamond [5]

The hexamethylenetetramine (0.1362 g, 1 mmol) was put into solution at the same time. After two weeks, the colourless crystals were obtained.

Experimental details

The data were collected via CrysAlis^{Pro} 1.171.39.7e [1], solved by SHELXT [2] program with WinGX [3], refined by SHELXL [4]. The structural graphics were finished by Diamond [5]. Most importantly, the N1 atom is disordered.

Comment

In recent years, due to the enormous applications such as magnetism [6], photoluminescence [7] and so on, the coordination compounds containing metal ion have attracted extensive attention.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
C1	0.6023(7)	0.4133(7)	0.6338(5)	0.0497(18)
C2	0.5401(9)	0.4552(9)	0.7090(6)	0.063(2)
H1	0.5704(01)	0.4266(36)	0.7843(43)	0.076*
C3	0.4350(8)	0.5378(8)	0.6765(6)	0.056(2)
H2	0.3971(74)	0.5642(17)	0.7305(13)	0.068*
C4	0.3846(6)	0.5822(7)	0.5658(5)	0.0455(16)
C5	0.4445(6)	0.5428(6)	0.4833(5)	0.0365(13)
C6	0.1984(7)	0.2979(5)	0.4737(5)	0.0382(13)
H3	0.2771(98)	0.3468(14)	0.5142(93)	0.046*
H4	0.1210(86)	0.3304(82)	0.4906(64)	0.046*
C7	0.0505(6)	0.2307(5)	0.2876(5)	0.0354(13)
H5	0.0322(11)	0.2336(54)	0.2052(00)	0.043*
H6	−0.0275(20)	0.2643(77)	0.3026(40)	0.043*
C8	0.2895(6)	0.2510(5)	0.3225(5)	0.0355(12)
H7	0.2727(53)	0.2533(62)	0.2402(87)	0.043*
H8	0.3703(35)	0.2987(35)	0.3602(12)	0.043*
C9	0.1003(7)	0.1010(6)	0.4530(6)	0.0447(15)
H9	0.0234(01)	0.1342(00)	0.4702(33)	0.054*
H10	0.1132(02)	0.0175(63)	0.4803(56)	0.054*
C10	0.1913(6)	0.0533(5)	0.3037(6)	0.0387(13)
H11	0.2064(62)	−0.0305(83)	0.3296(63)	0.046*
H12	0.1746(33)	0.0543(07)	0.2215(18)	0.046*
C11	0.3370(6)	0.1222(6)	0.4861(6)	0.0439(15)
H13	0.4179(08)	0.1689(30)	0.5258(92)	0.053*
H14	0.3527(99)	0.0386(93)	0.5130(96)	0.053*
Ag1	0.1163(35)	0.4966(65)	0.2814(45)	0.0529(19)
N1 ^a	0.7014(9)	0.3537(9)	0.6806(9)	0.037(2)
H15 ^a	0.7264(57)	0.3412(27)	0.7536(16)	0.044*
H16 ^a	0.7475(93)	0.3232(34)	0.6413(03)	0.044*
N2	0.1719(4)	0.3061(4)	0.3466(4)	0.0304(9)
N3	0.2219(6)	0.1719(5)	0.5142(4)	0.0438(13)
N4	0.0695(5)	0.1018(4)	0.3266(5)	0.0355(11)
N5	0.3129(5)	0.1252(5)	0.3624(5)	0.0372(11)
O1	0.2225(7)	0.7002(9)	0.6447(7)	0.112(3)
O2	0.2711(7)	0.7918(6)	0.4875(8)	0.094(2)
O3	0.1266(5)	0.6136(6)	0.4581(5)	0.0692(16)
S1	0.2401(2)	0.6807(2)	0.5364(2)	0.0655(6)

^aOccupancy: 0.5.

As is shown in the left part of figure, the silver atom is four-coordinated by an oxygen atom and a nitrogen atom from 4-amino-1,5-naphthalenedisulfonic acid, and two nitrogen atoms from two hexamethylenetetramine ligands desperately, the bond lengths of Ag–N and Ag–O in a normal range [8]. Thus, this crystal structure is 3D and there is weak intramolecular interaction such as NH···O (*cf.* right part of the figure). Symmetry codes: A = −*x*, 0.5 + *y*, 0.5 − *z*; B = 1 − *x*, 1 − *y*, 1 − *z*; C = −*x*, −0.5 + *y*, 0.5 − *z*.

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