Crystal structure of ethylenediaminesilver(I) perchlorate, $C_2H_8N_2Ag(ClO_4)$

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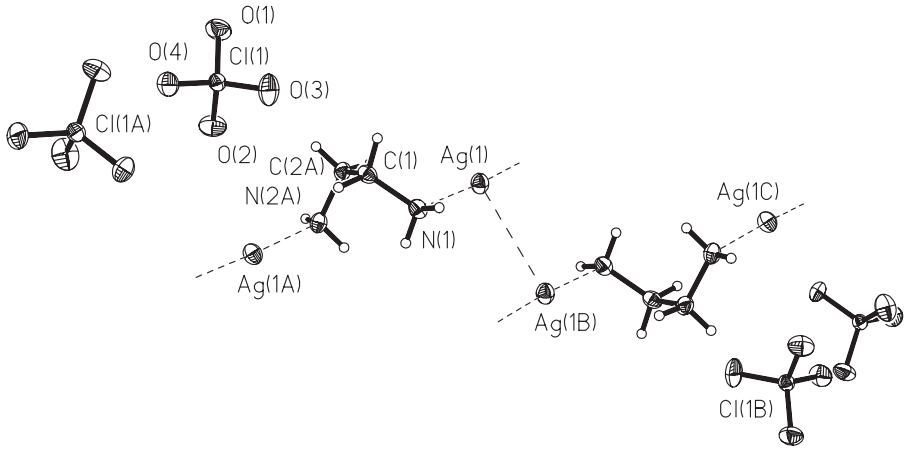

Abstract

$C_2H_8AgClN_2O_4$, monoclinic, $P12_1/n1$ (No. 14), $a = 8.676(3)$ Å, $b = 9.869(3)$ Å, $c = 8.822(3)$ Å, $\beta = 111.240(4)°$, $V = 704.1$ Å^3, $Z = 4$, $R_{gt}(F) = 0.032$, $wR_{ref}(F^2) = 0.063$, $T = 293$ K.

Source of material

All reagents and solvents were used as obtained without further purification. With stirring to an (1 mmol, 0.06 g) solution in 30% ammonium solution (5 ml) was added a $AgClO_4$ (1 mmol, 0.20 g) solution in 30% ammonium solution (5 ml). The resulting clear solution was stood still in air for 3 days. Colorless prism crystals suitable for X-ray determination were deposited at the bottom of the flask. The product was washed with water for twice, and dried in vacuum under $CaCl_2$ (yield 65%). Elemental analysis: found – C, 9.11%; H, 3.10%; N, 10.36%; calc. for $C_2H_8N_2O_4ClAg$ – C, 8.98%; H, 3.02%; N, 10.48%.

Discussion

Preparation of noble metal nanocomposites is very important in the development of optical materials, polymer conductors, catalyst carriers, and other applications. Formation of metallic silver particles from silver salts has been in the focus of interest since the discovery of photography. To achieve optimal performance, it is fundamental to control the size and polydispersity of the particles of silver compounds. It has been shown that a great deal of silver(I) complexes act as excellent templates for nanoparticle fabrication [1-6]. We have been interested in the study of silver(I) complexes with various organic ligands in recent years.

There are crystallographically distinct one Ag ion, one ethylenediamine molecule and one perchlorate anion in the crystal structure. The Ag ions are coordinated by nitrogen atom of the amine [d(Ag—N) = 2.151(2) Å] to form one-dimensional chains and these chains have further a ligand unsupported Ag···Ag interaction [Ag···Ag 3.379(5) Å], resulting in a two-dimensional layer with (4,4) topology. The four-connected nodes are provided by pairs of Ag ions and connectors are provided by ethylenediamine ligands. The perchlorate groups residue between layers via weak interaction of oxygen and Ag ion [d(Ag1···O3) = 2.829(3) Å] to furnish final three-dimensional supramolecular array.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.14 × 0.18 × 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	32.02 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.82°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3907, 1413
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1052
$N(param)_{refined}$:	123
Programs:	SHELXTL [7], SHELXTL-plus [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.733(5)	0.136(5)	0.325(6)	0.05(2)
H(2)	4*e*	0.692(6)	–0.006(6)	0.385(6)	0.07(2)
H(3)	4*e*	1.224(6)	0.134(5)	0.829(6)	0.05(2)
H(4)	4*e*	1.156(6)	0.026(6)	0.881(6)	0.06(2)
H(5)	4*e*	0.543(5)	0.125(4)	0.500(5)	0.03(1)
H(6)	4*e*	0.487(6)	0.179(5)	0.328(6)	0.06(2)
H(7)	4*e*	1.027(6)	0.154(5)	0.995(6)	0.06(1)
H(8)	4*e*	1.200(6)	0.199(5)	1.057(6)	0.06(2)

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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*	0.93162(4)	0.09156(4)	0.62484(4)	0.0428(2)	0.0452(2)	0.0424(2)	0.0042(2)	0.0080(2)	–0.0016(2)
Cl(1)	4*e*	0.1022(1)	0.3094(1)	0.3853(1)	0.0353(5)	0.0340(6)	0.0326(6)	0.0006(4)	0.0102(4)	0.0002(5)
N(1)	4*e*	0.7141(5)	0.0949(4)	0.4071(5)	0.050(2)	0.035(2)	0.035(2)	0.001(2)	0.012(2)	–0.001(2)
N(2)	4*e*	1.1399(5)	0.1016(5)	0.8503(5)	0.037(2)	0.032(2)	0.049(2)	–0.001(2)	0.013(2)	0.003(2)
O(1)	4*e*	0.0965(4)	0.3449(4)	0.5392(4)	0.082(2)	0.075(3)	0.038(2)	0.002(2)	0.022(2)	–0.005(2)
O(2)	4*e*	0.1973(4)	0.4076(3)	0.3399(4)	0.068(2)	0.058(2)	0.069(2)	–0.026(2)	0.034(2)	–0.002(2)
O(3)	4*e*	0.1759(4)	0.1807(4)	0.3945(5)	0.076(2)	0.040(2)	0.093(3)	0.017(2)	0.017(2)	–0.002(2)
O(4)	4*e*	–0.0615(3)	0.3063(4)	0.2650(4)	0.033(2)	0.092(3)	0.045(2)	–0.005(2)	–0.002(1)	0.003(2)
C(1)	4*e*	0.5732(5)	0.1687(5)	0.4224(6)	0.038(3)	0.045(3)	0.033(2)	–0.006(2)	0.008(2)	0.003(2)
C(2)	4*e*	1.1131(6)	0.1871(5)	0.9752(6)	0.032(2)	0.049(3)	0.037(3)	–0.005(2)	0.009(2)	0.001(2)

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