Crystal structure of ethylenediaminesilver(I) 4-nitrobenzoate hemi-hydrate, $Ag(C_2H_8N_2)(C_7H_4NO_4) \cdot 0.5H_2O$

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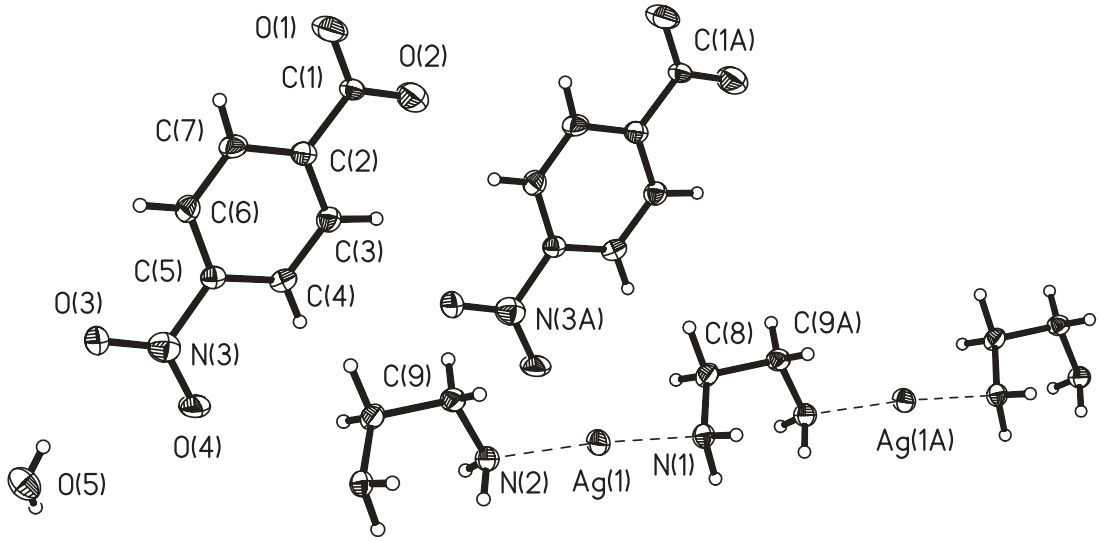

Abstract

$C_9H_{13}AgN_3O_{4.50}$, monoclinic, *C*12/*c*1 (No. 15), $a = 8.331(2)$ Å, $b = 11.645(2)$ Å, $c = 23.947(5)$ Å, $\beta = 94.845(3)°$, $V = 2314.8$ Å^3, $Z = 8$, $R_{gt}(F) = 0.030$, $wR_{ref}(F^2) = 0.072$, $T = 298$ K.

Source of material

Ag_2O (0.5 mmol, 116 mg) and 4-nitrobenzoate (1 mmol, 167 mg) were dissolved in a 1 : 1 solution of acetonitrile and concentrated ammonium solution (v/v, 10 ml), stirring for ca. 10 min ethylenediamine (1 mmol, 60 mg) was added to obtain a clear solution. After standing still the solution in air for two days with the ammium gas escaping large colorless prism crystals were crystallized, isolated, washed with water for three times, and dried in a vacuum desiccator under drying $CaCl_2$ (yield 45%). Elemental analysis: found – C, 31.70%; H, 3.90%; N, 12.14%; calc. for $C_9H_{13}AgN_3O_{4.5}$ – C, 31.51%; H, 3.82%; N, 12.25%.

Discussion

Coordination chemistry of coinage metal(I) monovalent ions have received considerable attention in the past three decades. The research on different uses and ideas of various silver(I) compounds is being in the ascendant. We have been interested in the investigation on silver(I) complexes with various organic ligands containing N and/or O atoms. Reported here is a silver(I)carboxylato complex with ethylenediamine.

The title complex crystallizes with the asymmetric unit consisting of one Ag ions, one 4-nitrobenzoate anion, one ethylenediamine molecule, and half a crystal water molecule. The Ag(1) ion is coordinated by two nitrogen atoms from two ethylenediamines. The Ag—N distances are 2.138(3) Å and 2.148(3) Å and the N–Ag–N angle is 173.1(2)°, indicating a linear coordination of Ag(1). Besides, there is a ligand unsupported Ag—Ag bond with a *d*(Ag···Ag) distance of 2.934(3) Å. The nitrobenzoate anion is pendent and functions as a counterion to maintain charge balance. The amine ligands bridge adjacent Ag ions to form one-dimensional chain and the chains are linked by Ag—Ag bonds to form two-dimensional layer with (4,4) topology. The four-connected nodes are provided by pairs of Ag ions. In addition, there are a variety of hydrogen bonds N–H···O, O–H···O and C–H···O [*d*(N1···O3) = 3.070(3) Å; *d*(N1···O4) = 2.968(4) Å; *d*(N2···O5) = 3.060(2) Å; *d*(N2···O4) = 3.022(4) Å; *d*(O5···O3) = 2.803(1) Å, *d*(C3···O2) = 2.714(0) Å; *d*(C4···O4) = 2.784(7) Å; *d*(C7···O1) = 2.726(8) Å] which extend the two-dimensional layer into three-dimensional supramolecular array.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.20 × 0.30 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.55 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.72°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6470, 2309
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1862
$N(param)_{refined}$:	211
Programs:	SHELXTL [1], SHELXTL-plus [2]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*f*	0.334(5)	0.016(3)	0.572(2)	0.05(1)
H(2)	8*f*	0.287(4)	0.135(3)	0.649(2)	0.039(9)
H(3)	8*f*	–0.020(4)	0.320(3)	0.552(1)	0.040(9)
H(4)	8*f*	0.020(5)	0.194(3)	0.479(2)	0.06(1)
H(5)	8*f*	1.230(4)	–0.104(3)	0.669(2)	0.04(1)
H(6)	8*f*	1.207(4)	–0.143(3)	0.724(2)	0.040(9)
H(7)	8*f*	0.739(6)	0.102(4)	0.728(2)	0.08(2)
H(8)	8*f*	0.836(4)	0.186(3)	0.719(2)	0.05(1)
H(9)	8*f*	1.008(4)	–0.256(3)	0.684(1)	0.035(9)
H(10)	8*f*	1.011(4)	–0.219(2)	0.625(2)	0.037(9)
H(11)	8*f*	0.642(4)	0.129(3)	0.634(1)	0.033(9)
H(12)	8*f*	0.771(3)	0.210(2)	0.628(1)	0.023(8)
H(13)	8*f*	0.000(6)	0.492(3)	0.725(2)	0.05(1)

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)	8*f*	0.97424(3)	0.00715(2)	0.68864(1)	0.0430(2)	0.0342(2)	0.0402(2)	0.0154(1)	–0.0026(1)	–0.0033(1)
N(1)	8*f*	1.1547(3)	–0.1241(2)	0.6862(1)	0.032(2)	0.030(1)	0.037(2)	0.006(1)	0.000(1)	–0.001(1)
N(2)	8*f*	0.7937(4)	0.1327(3)	0.7014(1)	0.039(2)	0.030(1)	0.038(2)	0.010(1)	–0.004(1)	–0.003(1)
N(3)	8*f*	0.1047(4)	0.3182(3)	0.6592(1)	0.054(2)	0.055(2)	0.045(2)	–0.007(2)	0.008(2)	0.001(2)
O(1)	8*f*	0.1338(4)	0.0425(2)	0.4281(1)	0.086(2)	0.060(2)	0.035(2)	0.009(2)	–0.001(1)	–0.010(1)
O(2)	8*f*	0.3045(4)	–0.0546(3)	0.4803(1)	0.091(2)	0.084(2)	0.055(2)	0.037(2)	–0.008(2)	–0.020(2)
O(3)	8*f*	0.0006(3)	0.3947(2)	0.6519(1)	0.045(1)	0.048(1)	0.046(2)	0.015(1)	–0.000(1)	–0.007(1)
O(4)	8*f*	0.1955(3)	0.3020(2)	0.7027(1)	0.073(2)	0.066(2)	0.032(2)	0.020(1)	–0.016(1)	–0.011(1)
O(5)	4*e*	0	0.5259(4)	3/4	0.068(3)	0.040(2)	0.049(3)	0	0.015(2)	0
C(1)	8*f*	0.2074(4)	0.0237(2)	0.4732(1)	0.038(2)	0.030(2)	0.027(2)	–0.000(1)	–0.000(1)	–0.004(1)
C(2)	8*f*	0.1769(4)	0.0964(3)	0.5209(1)	0.036(2)	0.037(2)	0.031(2)	–0.003(1)	0.003(1)	–0.002(1)
C(3)	8*f*	0.2584(4)	0.0736(3)	0.5722(1)	0.037(2)	0.034(2)	0.037(2)	0.003(1)	–0.000(1)	0.001(1)
C(4)	8*f*	0.2311(4)	0.1445(3)	0.6171(2)	0.035(2)	0.035(2)	0.032(2)	–0.003(1)	–0.004(1)	0.003(1)
C(5)	8*f*	0.1270(3)	0.2376(2)	0.6103(1)	0.030(2)	0.032(2)	0.029(2)	–0.006(1)	0.003(1)	0.001(1)
C(6)	8*f*	0.0454(4)	0.2569(3)	0.5578(1)	0.039(2)	0.038(2)	0.037(2)	0.009(2)	0.001(2)	0.004(1)
C(7)	8*f*	0.0692(4)	0.1859(3)	0.5134(1)	0.043(2)	0.046(2)	0.026(2)	0.001(2)	–0.003(1)	0.004(2)
C(8)	8*f*	1.0820(4)	–0.2317(3)	0.6631(2)	0.029(2)	0.034(2)	0.042(2)	0.002(1)	–0.004(2)	–0.000(1)
C(9)	8*f*	0.7014(4)	0.1768(3)	0.6509(2)	0.036(2)	0.030(2)	0.037(2)	0.002(1)	–0.002(2)	–0.004(1)

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