Crystal structure of ethylenediaminesilver(I) di(3,5-dinitrobenzoato)-silver(I) dihydrate, $Ag_2(C_2H_8N_2)(C_7H_3N_2O_6)_2 \cdot 2H_2O$

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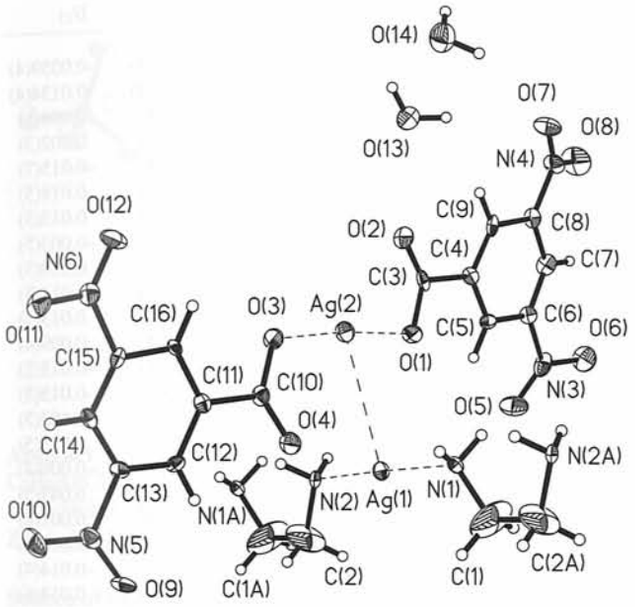

Abstract

$C_{16}H_{18}Ag_2N_6O_{14}$, triclinic, $P\bar{1}$ (No. 2), $a = 6.978(2)$ Å, $b = 12.919(5)$ Å, $c = 15.029(5)$ Å, $\alpha = 84.320(6)°$, $\beta = 76.810(5)°$, $\gamma = 79.612(6)°$, $V = 1295.1$ Å^3, $Z = 2$, $R_{gt(F)} = 0.065$, $wR_{ref(F^2)} = 0.091$, $T = 298$ K.

Source of material

Ag_2O (0.5 mmol, 116 mg) and 3,5-dinitrobenzoic acid (1 mmol, 212 mg) were dissolved in ammonium solution (10 ml), stirring for ca. 10 min and ethylenediamine (1 mmol, 60 mg) was added to obtain a clear solution. After standing in air for two days with the ammonium gas escaping, large colorless prismatic crystals were crystallized, isolated, washed with water for three times, and dried in a vacuum desiccator under drying $CaCl_2$ (yield 73%). Elemental analysis: found – C, 26.45%; H, 2.55%; N, 11.26%; calc. for $C_{16}H_{18}Ag_2N_6O_{14}$ – C, 26.18%; H, 2.47%; N, 11.45%.

Experimental details

All the H atoms were added on calculated positions. Due to a little disorder, the U_{ij} values of C1 and C2 atoms are quite large, we do not try to split them.

Discussion

The coordination chemistry of the coinage metals has been the subject of investigation for decades [1]. Historically, the interest in this area grew out of the diverse structural motifs displayed by these superficially similar monovalent cations. More recently, the interest has been renewed by practical concerns. Such coinage metal complexes can be used as potential precursors to metal films via CVD (chemical vapor deposition) processes [2,3]. An alternative method for separating olefins and paraffins is chemical adsorption using silver(I) compounds [4], which form complexes with unsaturated compounds such as olefins and acetylenes but not with saturated compounds such as paraffins [5]. We are 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 two Ag ions, two 3,5-dinitrobenzoate anions, one ethylenediamine molecule, and two crystal water molecules. The Ag(1) ion has a linear coordination, being coordinated by two nitrogen atoms from two amine. The Ag—N distances are 2.148(5) Å and 2.166(5) Å and the N–Ag–N angle is 175.7(2)°. The Ag(2) ion also has a linear coordination, being coordinated by two oxygen atoms from two dnbc. The Ag—O distances are 2.146(5) Å and 2.097(6)Å and the O–Ag–O angle is 174.5(3)°. Ag(1) and Ag(2) form ligand-unsupported Ag···Ag interaction with a distance of 3.177(5) Å. The amine ligands bridge Ag(1) ions to form a coordination polymer chain with $Ag(dnbc)_2$ (where dnbc is 3,5-dinitrobenzoate anion) coordination fragment attached via Ag···Ag interaction. In addition, there are a variety of O–H···O and C–H···O hydrogen bonds [d(O13···O14) = 2.729(4) Å; d(O13···O14)= 2.775(4) Å, d(O14···O(13), 2.775(4), d(C2···O7) = 2.980(4) Å; d(C9···O2) = 2.826(4) Å] which extend the two-dimensional layer into three-dimensional supramolecular array.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.08 × 0.13 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	15.89 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6823, 4780
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1816
$N(param)_{refined}$:	343
Programs:	SHELXTL [6], SHELXTL-plus [7]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.4410	0.6461	0.7127	0.044
H(1B)	2*i*	0.4618	0.7189	0.6327	0.044
H(2A)	2*i*	–0.2403	0.7045	0.6328	0.043
H(2B)	2*i*	–0.2601	0.6516	0.7235	0.043
H(1)	2*i*	0.3876	0.0554	0.6562	0.080
H(2)	2*i*	0.4221	0.0499	0.5563	0.080
H(3)	2*i*	0.8304	0.0656	0.4822	0.080
H(4)	2*i*	0.8091	–0.0447	0.5112	0.080
H(1C)	2*i*	0.5560	0.7741	0.7477	0.080
H(1D)	2*i*	0.3717	0.8487	0.7210	0.080
H(2C)	2*i*	–0.4753	0.8030	0.7154	0.080
H(2D)	2*i*	–0.3271	0.8157	0.7764	0.080
H(5)	2*i*	0.0622	0.4133	0.8676	0.050
H(7)	2*i*	0.1552	0.1496	1.0232	0.077
H(9)	2*i*	0.3487	0.1561	0.7561	0.062
H(12)	2*i*	0.0756	0.8809	0.3784	0.050
H(14)	2*i*	0.2133	0.8690	0.1057	0.048
H(16)	2*i*	0.3451	0.6081	0.2649	0.043

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)	2*i*	0.08760(8)	0.70971(5)	0.68482(4)	0.0297(4)	0.0560(5)	0.0597(5)	–0.0053(3)	–0.0132(3)	–0.0059(4)
Ag(2)	2*i*	0.2149(1)	0.50890(6)	0.56557(5)	0.0873(6)	0.0553(5)	0.0472(5)	–0.0112(4)	–0.0181(4)	0.0134(4)
N(1)	2*i*	0.3920(8)	0.7102(5)	0.6904(4)	0.024(3)	0.037(4)	0.045(4)	–0.005(3)	–0.004(3)	0.006(3)
N(2)	2*i*	–0.2245(8)	0.7094(5)	0.6899(4)	0.033(4)	0.045(4)	0.034(4)	–0.009(3)	–0.016(3)	0.002(3)
N(3)	2*i*	–0.016(1)	0.347(1)	1.0416(6)	0.077(7)	0.13(1)	0.037(6)	–0.025(7)	–0.006(5)	–0.015(7)
N(4)	2*i*	0.369(2)	0.0254(7)	0.8965(7)	0.096(7)	0.045(6)	0.069(7)	–0.011(5)	–0.019(6)	0.018(5)
N(5)	2*i*	0.043(1)	1.0002(7)	0.2336(7)	0.065(5)	0.043(6)	0.077(7)	–0.012(5)	–0.022(5)	0.013(5)
N(6)	2*i*	0.390(1)	0.6688(8)	0.0937(6)	0.048(5)	0.074(7)	0.047(5)	–0.007(5)	–0.004(4)	–0.003(5)
O(1)	2*i*	0.1683(9)	0.4467(5)	0.7052(4)	0.088(5)	0.042(4)	0.056(4)	–0.018(4)	–0.016(3)	0.010(3)
O(2)	2*i*	0.295(1)	0.3009(5)	0.6357(4)	0.098(5)	0.074(5)	0.026(4)	–0.014(4)	–0.007(3)	0.011(3)
O(3)	2*i*	0.242(1)	0.5825(5)	0.4340(4)	0.119(6)	0.043(4)	0.057(4)	–0.024(4)	–0.037(4)	0.013(3)
O(4)	2*i*	0.1638(9)	0.7387(5)	0.4975(4)	0.095(5)	0.071(5)	0.044(4)	–0.002(4)	–0.011(4)	0.009(4)
O(5)	2*i*	–0.132(1)	0.4282(7)	1.0331(5)	0.115(7)	0.081(6)	0.094(6)	–0.008(5)	0.018(5)	–0.015(5)
O(6)	2*i*	0.017(1)	0.3048(7)	1.1135(5)	0.173(9)	0.146(8)	0.041(5)	0.013(6)	–0.020(5)	–0.015(5)
O(7)	2*i*	0.480(1)	–0.0085(6)	0.8330(6)	0.099(6)	0.056(5)	0.101(7)	–0.004(4)	0.012(5)	0.002(5)
O(8)	2*i*	0.312(2)	–0.0245(7)	0.9691(6)	0.213(9)	0.096(7)	0.092(6)	–0.027(6)	–0.016(6)	0.055(5)
O(9)	2*i*	0.010(1)	1.0510(5)	0.2992(6)	0.102(6)	0.025(4)	0.111(7)	0.004(4)	–0.037(5)	–0.009(4)
O(10)	2*i*	0.007(1)	1.0343(6)	0.1604(6)	0.126(7)	0.080(6)	0.094(6)	–0.003(5)	–0.026(5)	0.043(5)
O(11)	2*i*	0.395(1)	0.7197(6)	0.0184(5)	0.120(6)	0.086(5)	0.044(4)	–0.026(4)	–0.003(4)	0.001(4)
O(12)	2*i*	0.450(1)	0.5745(7)	0.1033(5)	0.144(8)	0.058(5)	0.092(6)	0.043(5)	–0.030(5)	–0.011(4)
O(13)	2*i*	0.391(1)	0.0861(6)	0.6033(5)	0.138(7)	0.093(6)	0.073(5)	–0.017(5)	–0.025(5)	–0.014(4)
O(14)	2*i*	0.761(1)	0.0206(7)	0.5115(5)	0.127(7)	0.119(7)	0.116(6)	–0.020(5)	–0.027(5)	0.013(6)
C(1)	2*i*	0.416(3)	0.782(2)	0.750(2)	0.10(2)	0.37(4)	0.75(6)	0.01(2)	–0.15(2)	–0.34(4)
C(2)	2*i*	–0.340(2)	0.809(1)	0.715(2)	0.06(1)	0.20(2)	0.49(4)	0.02(1)	–0.09(2)	–0.10(2)
C(3)	2*i*	0.225(1)	0.3480(8)	0.7017(7)	0.040(5)	0.046(7)	0.070(7)	–0.019(5)	–0.033(5)	0.022(5)
C(4)	2*i*	0.211(1)	0.2930(8)	0.7970(5)	0.036(5)	0.053(6)	0.035(5)	–0.005(4)	0.001(4)	0.002(5)
C(5)	2*i*	0.116(1)	0.3422(7)	0.8726(6)	0.041(5)	0.036(5)	0.048(6)	–0.010(4)	–0.007(4)	–0.001(4)
C(6)	2*i*	0.096(1)	0.2882(8)	0.9617(6)	0.053(6)	0.050(7)	0.045(6)	–0.012(5)	–0.003(4)	0.002(5)
C(7)	2*i*	0.173(1)	0.1863(9)	0.9663(6)	0.075(7)	0.074(8)	0.047(6)	–0.041(6)	–0.005(5)	0.015(6)
C(8)	2*i*	0.276(1)	0.1328(8)	0.8919(6)	0.055(6)	0.059(7)	0.029(5)	–0.019(5)	0.000(4)	0.018(5)
C(9)	2*i*	0.285(1)	0.1913(8)	0.8085(5)	0.040(5)	0.084(8)	0.041(5)	–0.022(5)	–0.022(4)	0.004(5)
C(10)	2*i*	0.202(1)	0.6837(9)	0.4317(6)	0.060(6)	0.066(8)	0.029(5)	–0.007(6)	–0.003(4)	0.002(5)
C(11)	2*i*	0.207(1)	0.7378(7)	0.3368(5)	0.034(5)	0.047(6)	0.042(5)	–0.003(4)	–0.006(4)	0.003(4)
C(12)	2*i*	0.129(1)	0.8416(6)	0.3273(5)	0.061(6)	0.033(5)	0.032(5)	–0.021(4)	–0.005(4)	–0.003(4)
C(13)	2*i*	0.132(1)	0.8859(6)	0.2405(7)	0.067(6)	0.022(5)	0.063(7)	–0.019(5)	–0.021(5)	0.012(5)
C(14)	2*i*	0.211(1)	0.8363(7)	0.1618(6)	0.027(4)	0.033(5)	0.060(6)	–0.008(4)	–0.011(4)	0.002(5)
C(15)	2*i*	0.289(1)	0.7340(7)	0.1749(5)	0.038(5)	0.065(7)	0.030(5)	–0.015(5)	–0.010(4)	–0.009(5)
C(16)	2*i*	0.291(1)	0.6792(6)	0.2600(5)	0.026(4)	0.035(5)	0.047(5)	0.003(4)	–0.014(4)	–0.002(4)

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