Crystal structure of 1,2-ethylenediaminesilver(I) bis(4-nitrobenzoato)-silver(I), $C_{16}H_{16}Ag_2N_4O_8$

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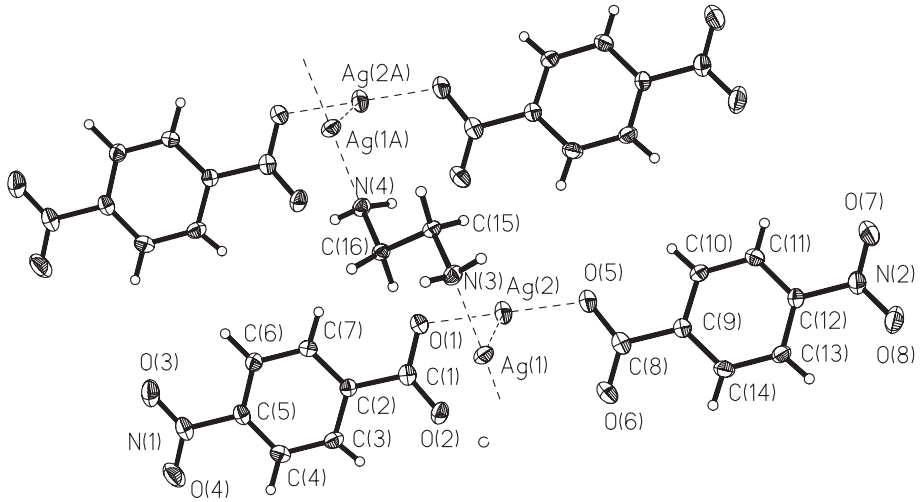

Abstract

$C_{16}H_{16}Ag_2N_4O_8$, triclinic, $P\bar{1}$ (No. 2), a = 6.346(3) Å, b = 7.022(3) Å, c = 23.04(1) Å, α = 82.638(7)°, β = 83.169(6)°, γ = 67.880(6)°, V = 940.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.070, T = 298 K.

Source of material

Ag_2O (0.5 , 116 mg) and 4-nitrobenzoic acid (= Hnbc; 1 mmol, 167 mg) were dissolved in ammonium solution (10 ml), stirring for ca. 10 min. 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 prism crystals were crystallized, isolated, washed with water for three times, and dried in a vacuum desiccator under drying $CaCl_2$ (yield 71%). Analysis: found – C, 31.79%; H, 2.77%; N, 9.05%; calc. for $C_{16}H_{16}Ag_2N_4O_8$ – C, 31.60%; H, 2.65%; N, 9.05%.

Discussion

The coordination chemistry of the coinage metals has been the subject of investigation for decades. Historically, interest in this area grew out of the diverse structural motifs displayed by these superficially similar monovalent cations. More recently, interest has been renewed by practical concerns. Silver ion can bind to a large number of sites on a peptide, including the amino nitrogen at the *N*-terminus, basic groups on the side chain, and the carbonyl oxygens of the peptide bonds [1]. We have been interested in the investigation on silver(I) complexes with various organic ligands containing N and/or O atoms.

The asymmetric unit consists of two Ag^+ cations, two 4-nitrobenzoate anions (nbc) and one ethylenediamine (en). The Ag(1) ion is linear coordinated by two nitrogen atoms from two en molecules. The Ag—N distances are 2.129(3) Å and 2.130(3) Å and the N–Ag–N angle is 178.4(1)°. The Ag(2) ion also has a linear coordination, being coordinated by two oxygen atoms from two nbc. The Ag—O distances are 2.144(3) Å and 2.093(3) Å and the O–Ag–O angle is 178.0(1)°. Ag(1) and Ag(2) form ligand-unsupported Ag···Ag interaction with a distance of 3.267 Å. The ethylenediamine ligands link Ag(1) ions to form a coordination polymer chain with $Ag(nbc)_2$ fragments attached via Ag···Ag interactions. In addition, there are a variety of N–H···O and C–H···O hydrogen bonds [d(N3···O2) = 3.063(3) Å; d(N3···O6) = 3.173(3) Å; d(N4···O2) = 3.025(3) Å; d(N4···O6) = 2.950(3) Å; d(C16···O1) = 3.398(3) Å] which extend the one-dimensional chain into three-dimensional supramolecular array.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.10 × 0.25 × 0.42 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	21.38 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.82°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5388, 3696
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2916
$N(param)_{refined}$:	335
Programs:	SHELXTL [2], SHELXTL-plus [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	0.397(7)	0.238(6)	0.430(2)	0.06(1)
H(2)	2*i*	0.281(7)	0.166(6)	0.528(2)	0.06(1)
H(3)	2*i*	–0.365(7)	0.367(6)	0.474(2)	0.06(1)
H(4)	2*i*	–0.210(6)	0.417(5)	0.378(2)	0.04(1)
H(5)	2*i*	0.400(6)	0.673(6)	0.031(2)	0.04(1)
H(6)	2*i*	0.525(7)	0.753(6)	–0.063(2)	0.05(1)
H(7)	2*i*	1.054(6)	0.794(6)	–0.004(2)	0.05(1)
H(8)	2*i*	0.928(7)	0.716(6)	0.092(2)	0.06(1)
H(9)	2*i*	–0.261(7)	1.223(6)	0.275(2)	0.06(1)
H(10)	2*i*	–0.199(7)	1.223(6)	0.217(2)	0.05(1)
H(11)	2*i*	0.483(7)	0.708(6)	0.310(2)	0.05(1)
H(12)	2*i*	0.574(8)	0.695(7)	0.251(2)	0.08(2)
H(13)	2*i*	–0.497(6)	1.140(6)	0.228(2)	0.04(1)
H(14)	2*i*	–0.270(6)	0.954(6)	0.204(2)	0.05(1)
H(15)	2*i*	0.602(6)	0.971(6)	0.325(2)	0.05(1)
H(16)	2*i*	0.797(6)	0.791(5)	0.299(2)	0.037(9)

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*	0.15091(4)	0.96519(5)	0.26372(1)	0.0307(2)	0.0515(2)	0.0484(2)	–0.0202(1)	–0.0043(1)	–0.0011(1)
Ag(2)	2*i*	0.21981(5)	0.52819(5)	0.21427(1)	0.0579(2)	0.0573(2)	0.0379(2)	–0.0226(2)	0.0059(1)	0.0008(1)
N(1)	2*i*	–0.1364(7)	0.2093(5)	0.5692(2)	0.075(3)	0.046(2)	0.035(2)	–0.032(2)	0.006(2)	–0.007(2)
N(2)	2*i*	0.8848(6)	0.8368(5)	–0.1016(2)	0.055(2)	0.056(2)	0.041(2)	–0.026(2)	0.005(2)	–0.000(2)
N(3)	2*i*	–0.1950(5)	1.1451(5)	0.2478(2)	0.035(2)	0.039(2)	0.045(2)	–0.019(2)	–0.001(2)	0.001(2)
N(4)	2*i*	0.4944(5)	0.7780(5)	0.2804(2)	0.035(2)	0.039(2)	0.042(2)	–0.020(2)	–0.002(1)	0.001(2)
O(1)	2*i*	0.0781(4)	0.4177(4)	0.2946(1)	0.051(2)	0.059(2)	0.037(2)	–0.022(1)	–0.003(1)	0.002(1)
O(2)	2*i*	0.4030(5)	0.3928(4)	0.3289(1)	0.048(2)	0.057(2)	0.053(2)	–0.029(1)	0.008(1)	–0.004(1)
O(3)	2*i*	–0.3409(6)	0.2478(5)	0.5781(1)	0.079(2)	0.087(3)	0.052(2)	–0.039(2)	0.018(2)	–0.006(2)
O(4)	2*i*	–0.0027(6)	0.1416(6)	0.6071(1)	0.102(3)	0.098(3)	0.036(2)	–0.045(2)	–0.012(2)	0.011(2)
O(5)	2*i*	0.3623(5)	0.6256(5)	0.1349(1)	0.066(2)	0.089(2)	0.040(2)	–0.042(2)	0.000(1)	0.008(2)
O(6)	2*i*	0.6924(5)	0.5761(4)	0.1715(1)	0.068(2)	0.059(2)	0.036(2)	–0.018(2)	–0.009(1)	0.002(1)
O(7)	2*i*	0.7730(6)	0.8445(6)	–0.1423(1)	0.096(3)	0.123(3)	0.041(2)	–0.063(2)	–0.007(2)	0.009(2)
O(8)	2*i*	1.0633(6)	0.8678(6)	–0.1089(1)	0.071(2)	0.099(3)	0.062(2)	–0.053(2)	0.015(2)	–0.005(2)
C(1)	2*i*	0.2113(6)	0.3836(5)	0.3355(2)	0.047(2)	0.028(2)	0.041(2)	–0.013(2)	0.003(2)	–0.005(2)
C(2)	2*i*	0.1185(6)	0.3291(5)	0.3959(2)	0.038(2)	0.030(2)	0.036(2)	–0.016(2)	–0.001(2)	–0.003(2)
C(3)	2*i*	0.2640(7)	0.2493(6)	0.4405(2)	0.035(2)	0.047(2)	0.051(2)	–0.019(2)	–0.008(2)	–0.002(2)
C(4)	2*i*	0.1826(7)	0.2095(6)	0.4973(2)	0.051(2)	0.044(2)	0.042(2)	–0.021(2)	–0.013(2)	0.000(2)
C(5)	2*i*	–0.0457(6)	0.2511(5)	0.5084(2)	0.055(2)	0.035(2)	0.033(2)	–0.023(2)	0.001(2)	–0.006(2)
C(6)	2*i*	–0.1953(7)	0.3261(6)	0.4656(2)	0.040(2)	0.048(2)	0.040(2)	–0.021(2)	0.001(2)	–0.004(2)
C(7)	2*i*	–0.1119(6)	0.3655(6)	0.4088(2)	0.038(2)	0.050(2)	0.037(2)	–0.016(2)	–0.008(2)	–0.003(2)
C(8)	2*i*	0.5663(7)	0.6237(6)	0.1309(2)	0.056(2)	0.033(2)	0.038(2)	–0.013(2)	0.001(2)	–0.004(2)
C(9)	2*i*	0.6492(6)	0.6852(5)	0.0705(2)	0.038(2)	0.030(2)	0.037(2)	–0.010(2)	–0.000(2)	–0.005(2)
C(10)	2*i*	0.5272(6)	0.7032(6)	0.0229(2)	0.036(2)	0.050(2)	0.044(2)	–0.023(2)	–0.006(2)	–0.001(2)
C(11)	2*i*	0.5988(7)	0.7537(6)	–0.0331(2)	0.045(2)	0.049(2)	0.036(2)	–0.019(2)	–0.011(2)	–0.001(2)
C(12)	2*i*	0.8006(6)	0.7879(5)	–0.0419(2)	0.040(2)	0.035(2)	0.035(2)	–0.016(2)	0.002(2)	–0.001(2)
C(13)	2*i*	0.9264(6)	0.7747(6)	0.0042(2)	0.033(2)	0.046(2)	0.048(2)	–0.018(2)	–0.008(2)	0.002(2)
C(14)	2*i*	0.8526(7)	0.7219(6)	0.0603(2)	0.044(2)	0.050(2)	0.041(2)	–0.017(2)	–0.015(2)	0.002(2)
C(15)	2*i*	–0.3395(6)	1.0318(6)	0.2373(2)	0.029(2)	0.045(2)	0.039(2)	–0.017(2)	–0.005(2)	–0.002(2)
C(16)	2*i*	0.6444(6)	0.8855(6)	0.2907(2)	0.032(2)	0.038(2)	0.036(2)	–0.017(2)	–0.005(2)	0.001(2)

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