Crystal structure of 1,2-diaminopropanesilver(I) 3,5-dinitrobenzoate hydrate, $Ag(C_3H_{10}N_2)(C_7H_3N_2O_6) \cdot 1.5H_2O$

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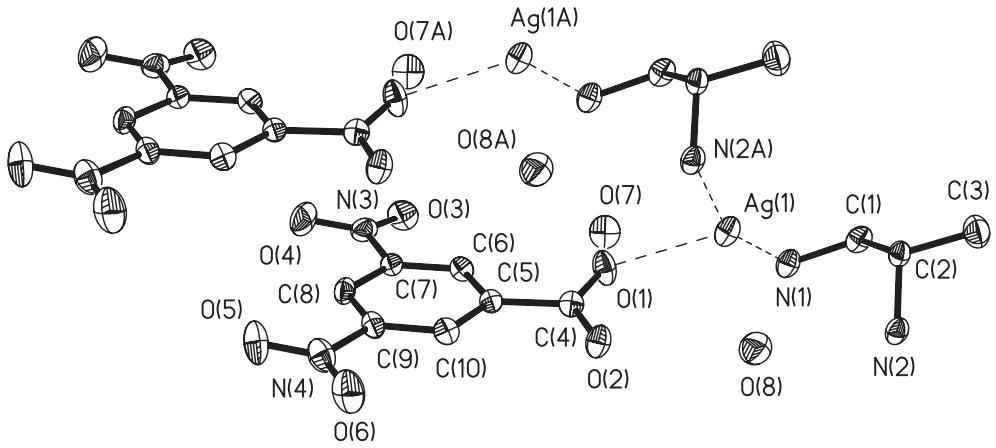

Abstract

$C_{10}H_{16}AgN_4O_{7.50}$, monoclinic, *C*12/*c*1 (No. 15), $a = 28.123(8)$ Å, $b = 5.241(2)$ Å, $c = 24.723(7)$ Å, $\beta = 122.958(4)°$, $V = 3057.9$ Å^3, $Z = 8$, $R_{gt}(F) = 0.037$, $wR_{ref}(F^2) = 0.070$, $T = 298$ K.

Source of material

Ag_2O (0.5 mmol, 116 mg) and 3,5-dinitrobenzoic acid (= dnbc; 1 mmol, 212 mg) were dissolved in ammonium solution (10 ml), stirring for ca. 10 min 1,2-diaminopropane (= dapn; 1 mmol, 74 mg) was added to obtain a clear solution. After standing still the solution 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 35%). Elemental analysis: found – C, 29.00%; H, 3.92%; N, 13.11%; calc. for $C_{10}H_{16}AgN_4O_{7.5}$ – C, 28.59%; H, 3.84%; N, 13.34%.

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, interest has been renewed by practical concerns. Despite major advances described above, 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.

The asymmetric unit of the title complex consists of one Ag ion, one 3,5-dinitrobenzoate (dnbc) anion, one 1,2-diaminopropane (dapn) molecule, and 1.5 crystal water molecules. The Ag ion is coordinated by two nitrogen atoms from two dapn and one carboxylate oxygen atoms from dnbc. The Ag—O bond distance is 2.483(3) Å and Ag—N distances are 2.169(3) Å and 2.194(3) Å. The N–Ag–N angle is 156.9(1)° and N–Ag–O angles are 108.0(1)° and 91.1(1)° which indicate the coordination of Ag is closer to T-shape rather than triangle. The Ag ions are linked by dapn to form one-dimensional chain. The chains are further linked by O–H···O [d(O7···O7) = 2.874(2) Å; d(O7···O1) = 2.718(2) Å; d(O8···O2) = 2.952(2) Å] and C–H···O [d(C6···O3) = 2.730(2) Å; d(C8···O4) = 2.692(2) Å] hydrogen bonds to result in 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:	10.22 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	53°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8165, 3164
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1904
$N(param)_{refined}$:	268
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)	8*f*	0.129(2)	0.389(7)	0.413(2)	0.06(1)
H(2)	8*f*	0.153(2)	0.547(7)	0.465(2)	0.05(1)
H(3)	8*f*	0.037(1)	0.290(5)	0.336(1)	0.05(1)
H(4)	8*f*	–0.0073(7)	0.134(7)	0.331(1)	0.08(2)
H(5)	8*f*	0.114(1)	0.198(7)	0.485(2)	0.05(1)
H(6)	8*f*	0.097(1)	0.462(6)	0.497(2)	0.04(1)
H(7)	8*f*	0.014(1)	0.497(6)	0.401(1)	0.034(8)
H(8)	8*f*	–0.036(2)	0.143(8)	0.410(2)	0.11(2)

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(9)	8*f*	0.028(2)	0.00(1)	0.454(2)	0.13(2)
H(10)	8*f*	0.011(2)	0.242(8)	0.477(2)	0.09(2)
H(11)	8*f*	0.222(1)	1.372(5)	0.379(1)	0.032(9)
H(12)	8*f*	0.224(1)	1.885(5)	0.266(1)	0.033(9)
H(13)	8*f*	0.098(1)	1.373(5)	0.196(1)	0.032(9)
H(14)	8*f*	0.257(2)	1.154(5)	0.482(2)	0.19(4)
H(15)	8*f*	0.2122(9)	0.994(7)	0.4359(9)	0.07(1)
H(16)	8*f*	0.023(2)	0.72(1)	0.252(3)	0.11(2)

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*	0.08178(1)	0.84411(5)	0.38566(1)	0.0591(2)	0.0404(2)	0.0609(2)	–0.0049(2)	0.0271(2)	0.0022(2)
N(1)	8*f*	0.1243(2)	0.5005(7)	0.4392(2)	0.036(2)	0.051(2)	0.048(2)	–0.012(2)	0.012(2)	–0.005(2)
N(2)	8*f*	0.0282(1)	0.1847(6)	0.3591(1)	0.043(2)	0.045(2)	0.039(2)	–0.012(2)	0.015(2)	–0.004(2)
N(3)	8*f*	0.2836(1)	1.7440(7)	0.3840(2)	0.051(2)	0.055(2)	0.063(2)	–0.012(2)	0.034(2)	–0.018(2)
N(4)	8*f*	0.1290(2)	1.7633(7)	0.1565(2)	0.057(3)	0.069(2)	0.057(2)	0.009(2)	0.033(2)	0.020(2)
O(1)	8*f*	0.1449(1)	1.0630(5)	0.3592(1)	0.051(2)	0.081(2)	0.043(2)	–0.015(1)	0.019(1)	0.016(1)
O(2)	8*f*	0.0826(1)	1.0242(5)	0.2539(1)	0.039(2)	0.051(2)	0.053(2)	–0.007(1)	0.014(1)	0.006(1)
O(3)	8*f*	0.3075(1)	1.6284(6)	0.4346(1)	0.059(2)	0.087(2)	0.046(2)	–0.011(2)	0.017(2)	–0.007(2)
O(4)	8*f*	0.3003(1)	1.9445(6)	0.3743(1)	0.073(2)	0.065(2)	0.090(2)	–0.036(2)	0.040(2)	–0.017(2)
O(5)	8*f*	0.1494(1)	1.9563(6)	0.1510(2)	0.085(2)	0.078(2)	0.091(2)	0.002(2)	0.050(2)	0.039(2)
O(6)	8*f*	0.0860(1)	1.6615(6)	0.1128(1)	0.069(2)	0.099(2)	0.051(2)	–0.002(2)	0.015(2)	0.029(2)
O(7)	8*f*	0.2440(1)	0.9967(7)	0.4748(1)	0.050(2)	0.084(2)	0.059(2)	0.005(2)	0.015(2)	0.011(2)
O(8)	4*e*	0	0.664(1)	1/4	0.081(4)	0.062(3)	0.074(3)	0	0.043(4)	0
C(1)	8*f*	0.0957(2)	0.3652(9)	0.4651(2)	0.052(3)	0.046(3)	0.032(2)	–0.004(2)	0.011(2)	0.001(2)
C(2)	8*f*	0.0340(2)	0.3224(7)	0.4147(2)	0.046(2)	0.037(2)	0.043(2)	–0.004(2)	0.023(2)	0.004(2)
C(3)	8*f*	0.0070(3)	0.167(1)	0.4424(3)	0.080(4)	0.079(4)	0.068(4)	–0.019(4)	0.046(3)	–0.001(3)
C(4)	8*f*	0.1249(2)	1.1216(7)	0.3015(2)	0.034(2)	0.043(2)	0.045(2)	0.006(2)	0.019(2)	0.005(2)
C(5)	8*f*	0.1549(1)	1.3365(6)	0.2903(2)	0.035(2)	0.038(2)	0.039(2)	0.004(2)	0.020(2)	0.003(2)
C(6)	8*f*	0.2052(2)	1.4341(7)	0.3401(2)	0.038(2)	0.048(2)	0.038(2)	0.003(2)	0.019(2)	0.004(2)
C(7)	8*f*	0.2308(2)	1.6344(7)	0.3300(2)	0.045(2)	0.037(2)	0.045(2)	–0.000(2)	0.028(2)	–0.001(2)
C(8)	8*f*	0.2068(2)	1.7470(7)	0.2701(2)	0.046(3)	0.036(2)	0.067(3)	–0.005(2)	0.038(2)	0.001(2)
C(9)	8*f*	0.1566(2)	1.6436(7)	0.2208(2)	0.043(2)	0.044(2)	0.049(2)	0.008(2)	0.030(2)	0.012(2)
C(10)	8*f*	0.1306(2)	1.4418(7)	0.2295(2)	0.034(2)	0.049(2)	0.042(2)	0.002(2)	0.018(2)	0.004(2)

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