Crystal structure of 1,2-diaminopropanesilver(I) 3,5-dinitrobenzoate — ammonia (1:1), $Ag(C_3H_{10}N_2)(C_7H_3N_2O_6) \cdot NH_3$

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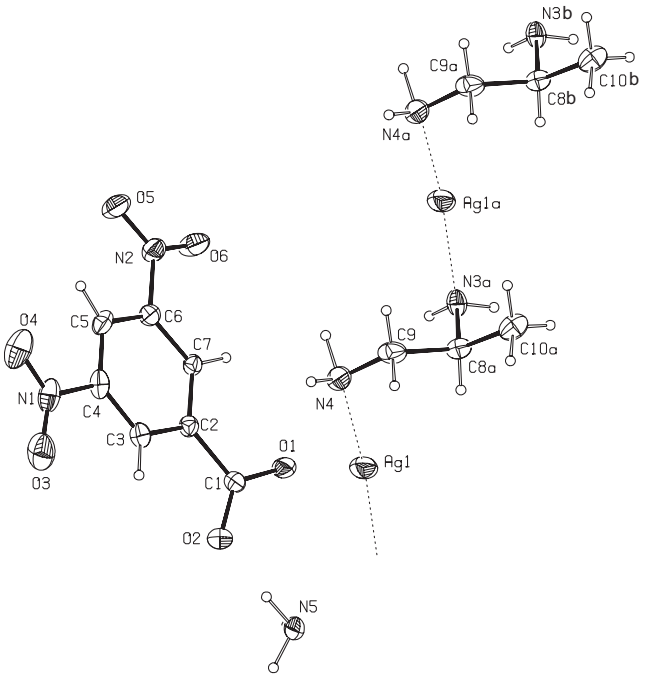

Abstract

$C_{10}H_{16}AgN_5O_6$, triclinic, $P\bar{1}$ (No. 2), $a = 5.640(5)$ Å, $b = 9.499(8)$ Å, $c = 14.84(2)$ Å, $\alpha = 84.24(1)°$, $\beta = 79.05(2)°$, $\gamma = 72.731(8)°$, $V = 744.8$ Å^3, $Z = 2$, $R_{gt}(F) = 0.044$, $wR_{ref}(F^2) = 0.111$, $T = 298$ K.

Source of material

All reagents and solvents were used as obtained without further purification. To an ammonium solution (10 ml) of Ag_2O (116 mg, 0.5 mmol) and 3,5-dinitrobenzoic acid (212 mg, 1.0 mmol) was added an acetonitrile solution (5 ml) of 1,2-diaminopropane (74 mg, 1 mmol) with stirring. The resulting clear solution was stood still for 1 week to vapor most of the ammonium gas, large colorless prismatic crystals of the title complex were deposited and collected by filtration, washed with acetonitrile and diethyl ether and dried in a vacuum desiccator over silica gel (yield 32%). Elemental analysis: found – C, 29.51%; H, 4.00%; N, 16.96%; calc. for $C_{10}H_{16}AgN_5O_6$ – C, 29.29%; H, 3.93%; N, 17.08%.

Discussion

Coordination compounds of the coinage monovalent ions have received considerable attention in the last thirty years. In particular, the study of their biological activity is of importance. Silver is by far the less investigated coinage metal in coordination chemistry, which possibly be attributed to the poor solubility of silver(I) compounds in common solvents and the sensitivity toward photode composition [1]. On the other hand, it has been found that many factors such as the nature of the ligands, solvents, counter-anions, etc., appear to modulate the stereochemistry of silver complexes. Our previous studies on the coordination of various silver(I) salts to a macrocyclic Schiff base have clearly shown the versatility [2,3]. As a continuation of these studies, we have synthesized and characterized silver(I)carboxylic complex of 1,2-diaminopropane, which is a single-brand helical complex.

The title complex crystallizes with two crystallographically equivalent [Ag(pren)](dnbc)(NH_3) symmetric units per unit cell (where pren is 1,2-diaminopropane and dnbc is 3,5-dinitrobenzoate anion), and one crystal lattice ammonia molecule. There is one silver(I) atom in each symmetric unit, which is two coordinated to two nitrogen atoms from different pren ligands. The average silver-nitrogen bond length (2.151(5) Å) is in the normal distance of Ag—N (primary amine). The N–Ag–N angle of 163.5(2)° is significantly deviated from 180°, indicating a larger geometry strain around silver atom. All pren molecules in the complex are in *trans*-conformation, which join the silver atoms along *a*-axis to form a one-dimensional helical chain. All the oxygen atoms from the ligand groups in the title complex do not participate in coordinating, but some of them contribute to the formation of a great deal of weak interactions. The non-coordinated molecular NH_3 join the neighboring silver atoms (d(N—Ag) = 3.240(5) Å and 2.955(5) Å), forming a double single-helical chain.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.18 × 0.23 × 0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.90 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	54.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4256, 2991
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2029
$N(param)_{refined}$:	263
Programs:	SHELXTL [4], SHELXTL-plus [5]

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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*	0.585(9)	0.626(5)	0.111(3)	0.06(1)
H(2)	2*i*	–0.046(8)	0.837(4)	0.011(3)	0.05(1)
H(3)	2*i*	–0.034(7)	0.851(4)	0.278(3)	0.032(9)
H(4)	2*i*	0.708(8)	0.428(5)	0.422(3)	0.05(1)
H(5)	2*i*	0.620(8)	0.343(4)	0.488(3)	0.05(1)
H(6)	2*i*	0.15(2)	0.330(8)	0.229(6)	0.14(3)
H(7)	2*i*	–0.07(1)	0.425(6)	0.266(3)	0.07(1)
H(8)	2*i*	0.998(7)	0.218(3)	0.427(2)	0.026(8)
H(9)	2*i*	–0.155(8)	0.224(4)	0.275(3)	0.04(1)
H(10)	2*i*	0.119(9)	0.139(5)	0.291(3)	0.06(1)
H(11)	2*i*	0.93(1)	–0.006(7)	0.408(4)	0.11(2)
H(12)	2*i*	0.705(8)	0.092(4)	0.491(3)	0.05(1)
H(13)	2*i*	0.64(1)	0.078(6)	0.400(4)	0.09(2)
H(14)	2*i*	1.019(2)	0.627(5)	0.412(3)	0.10(2)
H(15)	2*i*	0.780(8)	0.661(5)	0.381(2)	0.10(2)
H(16)	2*i*	0.809(9)	0.738(3)	0.455(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)	2*i*	0.33444(7)	0.37125(4)	0.36665(3)	0.0464(2)	0.0713(3)	0.1052(4)	–0.0091(2)	–0.0171(2)	0.0160(2)
N(1)	2*i*	0.432(1)	0.6838(5)	–0.0454(3)	0.118(4)	0.067(3)	0.040(2)	–0.032(3)	–0.008(3)	–0.002(2)
N(2)	2*i*	–0.3293(8)	0.9602(4)	0.1560(3)	0.063(2)	0.056(2)	0.068(3)	–0.017(2)	–0.023(2)	0.013(2)
N(3)	2*i*	0.6594(7)	0.3469(4)	0.4289(3)	0.062(2)	0.055(2)	0.036(2)	–0.016(2)	0.008(2)	–0.007(2)
N(4)	2*i*	0.0855(8)	0.3495(5)	0.2788(3)	0.049(2)	0.094(3)	0.042(2)	0.002(2)	0.001(2)	–0.002(2)
N(5)	2*i*	0.8494(8)	0.6451(5)	0.4335(3)	0.058(2)	0.099(3)	0.036(2)	–0.033(2)	–0.010(2)	0.010(2)
O(1)	2*i*	0.3069(5)	0.7054(4)	0.3659(2)	0.046(2)	0.111(2)	0.040(2)	–0.020(2)	–0.012(1)	0.002(2)
O(2)	2*i*	0.6705(6)	0.6317(4)	0.2700(2)	0.044(2)	0.079(2)	0.072(2)	–0.013(2)	–0.015(2)	0.010(2)
O(3)	2*i*	0.656(1)	0.6213(5)	–0.0557(3)	0.118(4)	0.096(3)	0.060(2)	–0.013(3)	0.017(2)	–0.015(2)
O(4)	2*i*	0.313(1)	0.7077(5)	–0.1063(3)	0.169(5)	0.118(3)	0.037(2)	–0.039(3)	–0.028(3)	–0.013(2)
O(5)	2*i*	–0.4326(7)	0.9950(4)	0.0895(3)	0.079(2)	0.087(2)	0.092(3)	–0.023(2)	–0.048(2)	0.024(2)
O(6)	2*i*	–0.4260(7)	1.0005(4)	0.2321(3)	0.062(2)	0.098(3)	0.067(2)	0.003(2)	–0.026(2)	–0.014(2)
C(1)	2*i*	0.4361(7)	0.6863(4)	0.2879(3)	0.040(2)	0.053(2)	0.058(3)	–0.014(2)	–0.007(2)	0.009(2)
C(2)	2*i*	0.3004(7)	0.7334(4)	0.2071(2)	0.047(2)	0.045(2)	0.036(2)	–0.018(2)	–0.008(2)	–0.001(2)
C(3)	2*i*	0.4227(9)	0.6891(4)	0.1185(3)	0.057(3)	0.045(2)	0.047(3)	–0.018(2)	–0.001(2)	–0.003(2)
C(4)	2*i*	0.297(1)	0.7313(4)	0.0465(3)	0.080(3)	0.047(2)	0.045(3)	–0.024(2)	0.002(2)	0.002(2)
C(5)	2*i*	0.0501(9)	0.8187(5)	0.0558(3)	0.073(3)	0.053(2)	0.044(2)	–0.025(2)	–0.025(2)	0.009(2)
C(6)	2*i*	–0.0660(8)	0.8640(4)	0.1435(3)	0.050(2)	0.046(2)	0.052(2)	–0.020(2)	–0.015(2)	0.004(2)
C(7)	2*i*	0.0528(7)	0.8216(4)	0.2180(3)	0.046(2)	0.051(2)	0.033(2)	–0.019(2)	–0.007(2)	0.001(2)
C(8)	2*i*	0.8664(8)	0.2135(5)	0.4032(3)	0.046(2)	0.053(2)	0.055(3)	–0.005(2)	–0.015(2)	–0.008(2)
C(9)	2*i*	–0.0248(9)	0.2241(6)	0.3041(3)	0.043(2)	0.085(3)	0.056(3)	0.002(2)	–0.005(2)	–0.023(2)
C(10)	2*i*	0.773(1)	0.0790(6)	0.4290(5)	0.084(4)	0.056(3)	0.072(4)	–0.015(3)	–0.033(3)	–0.007(2)

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References

1. Omary, M. A.; Webb, T. R.; Assefa, Z.; Shankle G. E.; Patterson, H. H.: Crystal Structure, Electronic Structure, and Temperature-Dependent Raman Spectra of $Tl[Ag(CN)_2]$: Evidence for Ligand-Unsupported Argentophilic Interactions. Inorg. Chem. **37** (1998) 1380-1386.
2. Zhu, H.-L.; Tong, Y.-X.; Long, L.-S.; Tong, M.-L. Chen, X.-M.: Anion-Controlled Formation of Silver(I) Complexes of a Hexaazamacrocyclic Schiff Base: Synthesis, Structures and Electrochemistry. Supramol. Chem. **11** (1999) 119-133.
3. Zhu, H.-L.; Tong Y.-X.; Chen, X.-M.: Influence of ligand backbones and counter ions on structures of helical silver(I) complexes with di-Schiff bases derived from phthalaldehydes and diamine. J. Chem. Soc., Dalton Trans. (2000) 4182-4186.
4. Sheldrick, G. M.: Siemens SHELXTL (Version 5.0). Siemens Industrial Automation, Inc., Analytical Instrumentation, USA 1995.
5. Sheldrick, G. M.: SHELXTL-plus. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison Wisconsin, USA 1991.