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Crystal structure of bis(1,10-phenanthroline-*N*,*N*')silver(I) mononitrate, $[Ag(C_{12}H_8N_2)_2][NO_3]$

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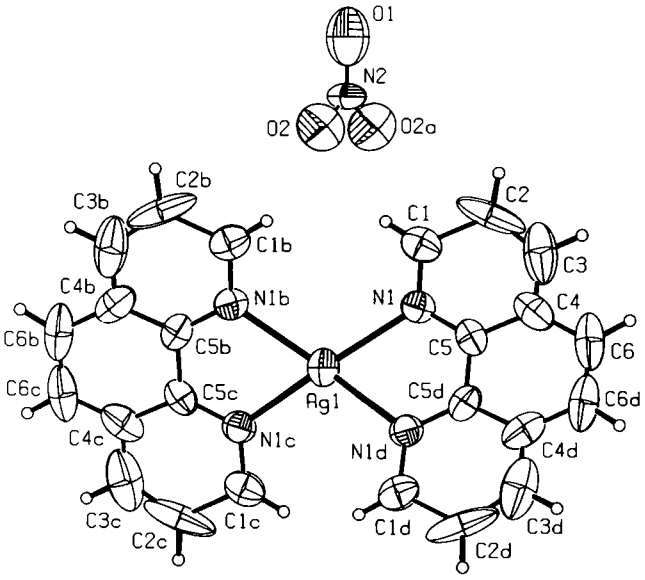

Abstract

$C_{24}H_{16}AgN_5O_3$, orthorhombic, *Fddd* (no. 70), $a = 3.668(1)$ Å, $b = 30.046(9)$ Å, $c = 38.88(1)$ Å, $V = 4284.2$ Å^3, $Z = 8$, $R_{gt}(F) = 0.064$, $wR_{ref}(F^2) = 0.198$, $T = 273$ K.

Source of material

The title complex was synthesized under hydrothermal conditions. A mixture of $AgNO_3$ (0.0170 g, 0.1 mmol), H_2bpndc (0.0270 g, 0.1 mmol), 1,10-phenanthroline (0.0198 g, 0.1 mmol), 0.05 ml Et_3N and 6 ml deionized water was sealed in a Teflon-lined stainless vessel (25 ml) and heated at 140 °C for 72 h under autogenous pressure, then cooled slowly to room temperature. Colorless and column single crystals were obtained by filtration.

Discussion

Coordination compounds of cationic silver(I) containing N-donor ligands have extensively been described as producing functional supramolecular arrays of different dimensionality in the solid [1-3]. This fact being related with the coordinative versatility of the silver atom to adopt several coordination numbers and with the variety of N-donor ligands and counterions [4].

In the crystal structure of title compound, the silver(I) adopts a distorted tetrahedron coordination environment, defined by four nitrogen atoms from two *o*-phenanthroline molecules. Due to the presence of two large *o*-phenanthroline molecules, no free space in the silver coordination sphere is left and the inorganic NO_3^- anion is moved off from the closest silver environment to acting as counter ion. In the $[Ag(phen)_2]^+$ cations, the Ag—N bond lengths are 2.304(6) Å, and two *o*-phenanthroline ligands from the one cations do not lie in the same plane and are twisted by the small angle of 40.3°. Such an arrangement of *o*-phenanthroline molecules around the silver atom was also found for the structure of $Ag(phen)_2[CF_3COO] \cdot H_2O$ [1]. As it can be expected, there is a π-π stacking interaction between phen ligands of the neighbor $[Ag(phen)_2]^+$ cations with a phen-phen interplanar separation of about 3.3 Å – 3.5 Å. Through those π-π interactions, the adjacent mononuclear units are linked into an infinite rows parallel to the *b*,*c* plane. The intermolecular C–H···O hydrogen bonds (d(C···O = 3.190 Å – 3.369 Å) between NO_3^- anions and adjacent *o*-phenanthroline molecules connect those infinite rows into 3D supramolecular architecture.

Table 1. Data collection and handling.

Crystal:	colorless, columned, size 0.12 × 0.16 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.79 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.18°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4808, 946
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 403
$N(param)_{refined}$:	87
Programs:	SHELXS-97 [5], SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	32*h*	0.8294	0.1590	0.2019	0.114
H(3)	32*h*	0.8481	0.2966	0.1967	0.191
H(6)	32*h*	1.0057	0.3351	0.1526	0.163
H(2)	32*h*	0.7247	0.2347	0.2284	0.209

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	8*a*		⅛	⅛	⅛	0.134(2)	0.072(1)	0.107(1)	0	0	0
N(1)	32*h*		0.990(2)	0.1869(2)	0.1576(2)	0.083(5)	0.085(4)	0.073(4)	−0.004(3)	0.001(3)	−0.004(3)
C(5)	32*h*		1.054(2)	0.2261(3)	0.1423(2)	0.051(5)	0.077(5)	0.093(5)	0.003(4)	−0.013(4)	−0.019(4)
C(1)	32*h*		0.871(3)	0.1854(3)	0.1901(2)	0.072(5)	0.123(7)	0.091(7)	−0.004(5)	−0.013(5)	−0.016(5)
C(3)	32*h*		0.888(4)	0.2689(4)	0.1867(5)	0.10(1)	0.094(8)	0.28(2)	0.028(8)	−0.07(1)	−0.04(1)
C(4)	32*h*		1.008(3)	0.2682(4)	0.1564(3)	0.106(9)	0.13(1)	0.103(7)	0.021(6)	−0.011(6)	−0.044(6)
C(6)	32*h*		1.059(4)	0.3088(3)	0.1412(4)	0.10(1)	0.062(5)	0.24(2)	0.005(6)	−0.04(1)	−0.018(6)
C(2)	32*h*		0.811(3)	0.2319(8)	0.2061(3)	0.046(6)	0.38(2)	0.096(7)	0.04(1)	−0.011(5)	−0.13(1)
N(2)	16*g*	0.5	⅝	⅛	0.2670(6)	0.20(3)	0.11(2)	0.05(1)	−0.01(2)	0	0
O(1)	16*g*	0.5	⅝	⅛	0.2983(6)	0.17(2)	0.12(2)	0.25(4)	−0.03(2)	0	0
O(2)	32*h*	0.5	0.894(5)	0.1140(8)	0.2508(6)	0.27(3)	0.18(2)	0.16(2)	0.05(2)	0.01(2)	−0.01(2)

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