

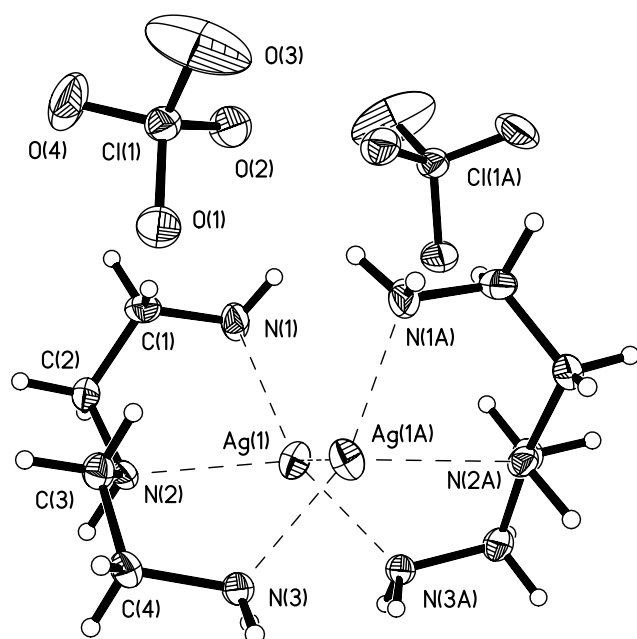
Crystal structure of diethylenetriaminesilver(I) perchlorate, $\text{Ag}(\text{C}_4\text{H}_{13}\text{N}_3)(\text{ClO}_4)$

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Abstract

$\text{C}_4\text{H}_{13}\text{AgClN}_3\text{O}_4$, orthorhombic, *Pccn* (No. 56), $a = 17.730(7)$ Å, $b = 7.162(3)$ Å, $c = 15.891(7)$ Å, $V = 2017.9$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.047$, $wR_{\text{ref}}(F^2) = 0.152$, $T = 298$ K.

Source of material

With stirring to a dien (1 mmol, 0.10 g) solution in 30% ammonium solution (5 ml) was added a AgClO_4 (1 mmol, 0.20 g) solution in 30% ammonium solution (5 ml), where dien is diethylenetriamine. The resulting clear solution was stood still in air for overnight. Colorless prism crystals suitable for X-ray determination were deposited at the bottom of the flask. The product was washed with water/methanol (1 : 1, v/v) twice, and dried in vacuum under CaCl_2 (yield 95%). Elemental analysis: found – C, 15.5%; H, 4.3%; N, 13.0%; $\text{C}_4\text{H}_{13}\text{N}_3\text{O}_4\text{ClAg}$ requires C, 15.5%; H, 4.2%; N, 13.5%.

Experimental details

The H atom attached to the secondary nitrogen atom was located from difference Fourier maps and refined isotropically. All the other hydrogen atoms were added on calculated positions.

Discussion

The title complex crystallizes primarily in a mononuclear form, in which the silver atom is coordinated in a Y-shape fashion

[$\angle \text{N1–Ag1–N2} = 76.0(2)^\circ$, $\angle \text{N2–Ag1–N3A} = 126.9(2)^\circ$ and $\angle \text{N1–Ag1–N3A} = 157.0(3)^\circ$] with the coordination of two nitrogen atoms from a dien and the third coordinate N atom from another dien. In each dien molecule, three nitrogen atoms respectively coordinate to two different Ag atoms with two N atoms joining one Ag atom and one end-nitrogen atom joining a different silver atom, forming a dimer. The $\text{Ag}\cdots\text{Ag}$ distance 3.035(2) Å in the dimer is shorter than the van der Waals radii of two silver atoms (3.44 Å), indicating some weak metal-metal interaction. The Ag–N distances of silver and two primary amine nitrogens ($d(\text{Ag1–N1}) = 2.200(7)$ Å and $d(\text{Ag1–N3A}) = 2.166(6)$ Å) are comparable to those found in other Ag-amine containing complexes. However, the Ag–N (secondary amine) distance ($d(\text{Ag1–N2}) = 2.543(6)$ Å) is much longer than those in the similar complexes we reported previously. Weak hydrogen contacts (from 2.962(4) Å to 3.348(8) Å) between nitrogen atoms from diens and oxygen atoms from perchlorates link the discrete dimers along *b*-axis into a one-dimensional chain.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.13 × 0.17 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	22.52 cm ^{−1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{\text{max}}$:	53.08°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	10599, 2030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 815
$N(\text{param})_{\text{refined}}$:	122
Programs:	SHELXTL [2], SHELXTL-plus [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1A)	8e	0.7275	0.0357	0.198	0.108
H(1B)	8e	0.6953	−0.1398	0.2271	0.108
H(3C)	8e	0.6895	0.3471	0.4955	0.072
H(3D)	8e	0.6872	0.5489	0.4977	0.072
H(1C)	8e	0.5977	−0.0024	0.1763	0.087
H(1D)	8e	0.6246	0.1911	0.2114	0.087
H(2A)	8e	0.5227	0.0721	0.2855	0.092
H(2B)	8e	0.5723	−0.099	0.3126	0.092
H(3A)	8e	0.5278	0.345	0.3548	0.075
H(3B)	8e	0.6081	0.3908	0.3194	0.075
H(4A)	8e	0.5811	0.5714	0.4387	0.082
H(4B)	8e	0.5725	0.3892	0.4925	0.082
H(1)	8e	0.571(3)	0.080(8)	0.420(4)	0.03(2)

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Ag(1)	8e	0.73904(4)	0.0399(1)	0.36349(5)	0.0615(5)	0.0741(6)	0.0844(6)	−0.0079(4)	−0.0149(4)	−0.0022(4)
Cl(1)	8e	0.6341(1)	0.5440(4)	0.0932(2)	0.064(2)	0.073(2)	0.057(2)	0.010(1)	−0.000(1)	0.003(1)
N(1)	8e	0.6961(4)	−0.016(1)	0.2360(5)	0.050(5)	0.139(8)	0.082(6)	−0.016(5)	0.007(4)	−0.058(5)
N(2)	8e	0.6013(3)	0.138(1)	0.3706(4)	0.045(4)	0.066(5)	0.048(5)	0.011(4)	0.008(4)	0.003(4)
N(3)	8e	0.6791(3)	0.449(1)	0.4645(4)	0.047(4)	0.080(5)	0.052(4)	0.018(4)	0.000(3)	−0.003(4)
O(1)	8e	0.6279(4)	0.6000(9)	0.1777(4)	0.088(5)	0.119(7)	0.071(5)	0.005(4)	0.004(4)	−0.036(4)
O(2)	8e	0.6858(4)	0.396(1)	0.0863(4)	0.087(5)	0.109(6)	0.083(5)	0.029(4)	0.004(4)	−0.007(4)
O(3)	8e	0.6579(8)	0.679(1)	0.0424(7)	0.48(2)	0.084(7)	0.19(1)	0.05(1)	0.15(1)	0.072(8)
O(4)	8e	0.5676(5)	0.477(2)	0.0692(6)	0.080(6)	0.43(2)	0.109(7)	0.057(8)	−0.037(5)	−0.13(1)
C(1)	8e	0.6211(5)	0.059(1)	0.2240(5)	0.079(7)	0.093(8)	0.046(6)	−0.016(6)	0.000(5)	−0.008(5)
C(2)	8e	0.5737(5)	0.033(1)	0.2984(6)	0.045(5)	0.119(8)	0.065(7)	−0.005(5)	−0.003(5)	−0.014(6)
C(3)	8e	0.5814(5)	0.335(1)	0.3662(5)	0.053(5)	0.067(7)	0.068(6)	0.010(5)	−0.001(5)	0.002(5)
C(4)	8e	0.5993(4)	0.445(1)	0.4454(6)	0.051(6)	0.088(7)	0.065(6)	0.016(5)	0.010(5)	−0.005(6)

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