

catena-Poly[[silver- μ -piperazine- κ^2 N:N'] perchlorate]

Ju-Tao Liu^a and Seik Weng Ng^{b*}

^aCollege of Life Science, Dalian Nationalities University, Dalian 116600, People's Republic of China, and ^bDepartment of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
 T = 295 K
 Mean σ (C–C) = 0.014 Å
 Disorder in main residue
 R factor = 0.057
 wR factor = 0.188
 Data-to-parameter ratio = 13.4

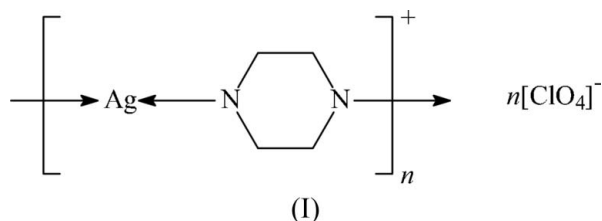
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\{[\text{Ag}(\text{C}_4\text{H}_{10}\text{N}_2)]\text{ClO}_4\}_n$, the piperazine ligand links adjacent Ag atoms into a linear polycationic chain; the Ag atom shows a linear coordination $[\text{Ag}—\text{N} = 2.158(8)$ and $2.172(8)$ Å; $\text{N}—\text{Ag}—\text{N} = 173.9(3)^\circ$]. The anions lie on crystallographic twofold axes and the piperazine rings on centres of inversion. The anion is hydrogen bonded to the chain.

Received 20 July 2006
 Accepted 24 July 2006

Comment

Silver(I) salts afford isolable complexes with piperazine; those whose crystal structures have been reported are the 2:1 silver 4-chlorobenzoate adduct (Yang *et al.*, 2005), the 1:2 silver trifluoroacetate adduct (Brammer *et al.*, 2002) and the 1:2 silver tetrafluoroborate adduct (Carlucci *et al.*, 1995). In the tetrafluoroborate, Ag is coordinated by the N atoms of four ligands in a tetrahedral geometry.



A solution study on the interaction of silver perchlorate with piperazine rules out direct interaction between the metal ion and the ligand in acetonitrile (Grzejdzia, 1993). This observation is in accord with the crystal structure study on the silver tetrafluoroborate adduct as the perchlorate anion possesses nucleophilic properties similar to that of the tetrafluoroborate. However, the interaction in the solid state for the piperazine adduct is a normal coordination bond, as found in the present study (Scheme I, Fig. 1). The interaction gives rise to a linear chain $[\text{Ag}—\text{N} = 2.158(8)$ and $2.172(8)$ Å; $\text{N}—\text{Ag}—\text{N} = 173.9(3)^\circ$]; the anions surround the chain and interact through hydrogen bonds (Table 1). The anions lie on twofold axes whereas the piperazine rings lie on centres of inversion.

Experimental

Silver perchlorate (41 mg, 0.2 mmol) and piperazine (20 mg, 0.2 mmol) were dissolved in ammonium hydroxide (20%, 10 ml). The solution was filtered and then kept away from light for about 14 d. Colourless crystals of the adduct were obtained in 40% yield.

Crystal data

[Ag(C₄H₁₀N₂)]ClO₄
M_r = 293.46
 Monoclinic, *P*2₁/*c*
a = 11.502 (8) Å
b = 6.032 (4) Å
c = 16.359 (8) Å
 β = 131.47 (3)°
V = 850.5 (10) Å³

Z = 4
D_x = 2.292 Mg m⁻³
 Mo *K*α radiation
 μ = 2.66 mm⁻¹
T = 295 (2) K
 Block, colourless
 0.26 × 0.24 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID IP
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
T_{min} = 0.325, *T_{max}* = 0.618

7985 measured reflections
 1954 independent reflections
 1556 reflections with *I* > 2σ(*I*)
R_{int} = 0.041
 θ_{\max} = 27.5°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.057
wR(*F*²) = 0.188
S = 1.21
 1954 reflections
 146 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 8.1397P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 $\Delta\rho_{\max} = 1.36 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.98 \text{ e } \text{Å}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1	0.85	2.29	3.03 (2)	144
N2—H2...O5	0.85	2.36	3.13 (2)	150
N2—H2...O6 ⁱ	0.85	2.43	3.15 (2)	143

Symmetry code: (i) $-x + 1, y, -z + \frac{3}{2}$.

The two perchlorate ions were disordered about a twofold rotation axis. All the O atoms of perchlorate ions were refined with an occupation factor of 0.50. The Cl—O lengths were restrained to within 0.01 Å of each other and the O...O distances to 0.02 Å of each other. Additionally, the displacement parameters were restrained to be nearly isotropic. In the final difference Fourier map, a large peak was found near atom O1. Attempts to allow the chlorine atoms of the perchlorate ions to refine off the symmetry element did not lead to any improvement. The C- and N-bound H atoms were positioned geometrically (C—H = 0.97 Å and N—H = 0.85 Å) and were included

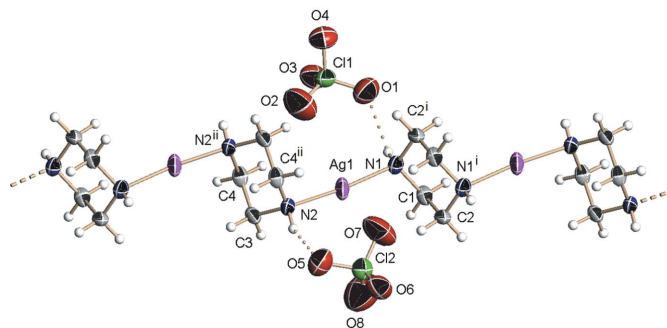


Figure 1

Part of a polymeric chain of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. Only one component of each disordered anion is shown. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $-x, 1 - y, 1 - z$.]

in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The authors thank Tianjin University for the diffraction measurements and Dalian Nationalities University and the University of Malaya for supporting this study.

References

- Brammer, L., Burgard, M. D., Eddleston, M. D., Rodger, C. S., Rath, N. P. & Adams, H. (2002). *CrystEngComm*, **4**, 239–248.
 Carlucci, L., Ciani, G., Prosperio, D. M. & Sironi, A. (1995). *Inorg. Chem.* **34**, 5698–5700.
 Grzejdziak, A. (1993). *Elektrokhimiya*, **29**, 886–891. (In Russian.)
 Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Rigaku (2004). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
 Rigaku/MS (2004). *CrystalStructure*. Rigaku/MS Inc. The Woodlands, Texas, USA.
 Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
 Yang, S., Zhou, Y. & Zhu, H.-L. (2005). *Acta Cryst.* **E61**, m219–m220.