

Poly[disilver(I)(Ag—Ag)- μ -4-chlorobenzoato- μ -piperazine]Song Yang, Ying Zou and
Hai-liang Zhu*Department of Chemistry, Fuyang Normal
College, Fuyang Anhui 236041, People's
Republic of ChinaCorrespondence e-mail:
hailiang_zhu@163.com

Key indicators

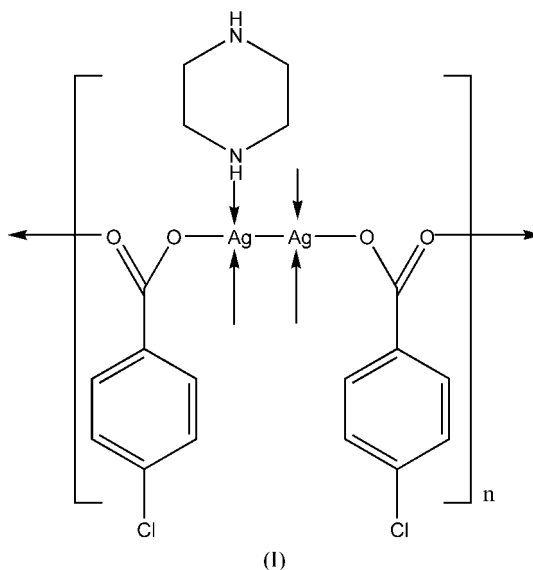
Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.012$ Å
 R factor = 0.035
 wR factor = 0.101
Data-to-parameter ratio = 14.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The 2/1 adduct of 4-chlorobenzoato-silver(I) and piperazine, $[\text{Ag}_2(\text{ClC}_6\text{H}_4\text{COO})_2(\text{C}_4\text{H}_{10}\text{N}_2)]_n$, adopts a chain structure in which the Ag atom is linked to one chlorobenzoate anion, one piperazine heterocycle and another Ag atom. The two Ag atoms exist in tetrahedral environments.

Received 9 November 2004
Accepted 20 December 2004
Online 8 January 2005

Comment

N-Heterocycles afford a number of adducts with silver(I) carboxylates; with some, such as piperazine and silver(I) trifluoroacetate (Brammer *et al.*, 2002), the piperazine and the trifluoroacetate units both engage in coordination, whereas in the phenanthroline complex, the anion does not participate, as the Ag atom is already chelated by two heterocycles (Paramonov *et al.*, 2003). Interest in this area grew out of the diverse structural motifs displayed by these superficially similar monovalent cations. We report here the crystal structure of (I), a new piperazine complex of silver 4-chlorobenzoate.



Just like the adduct with trifluoroacetate and piperazine (Brammer *et al.*, 2002), the heterocyclic ligand uses both N-donor sites and both O donors of the carboxylate anion to bind to Ag atoms in (I). Furthermore, two Ag atoms are joined by an Ag—Ag bond, with each Ag atom in a tetrahedral environment. The μ_2 bridging modes of the heterocycle and the anion lead to the formation of a network structure. For the adduct with trifluoroacetate, the Ag atom is coordinated by two pyridyl N atoms and two trifluoroacetate anions, but all Ag atoms are crystallographically independent (Brammer *et al.*, 2002).

Experimental

The reagents were commercial products and were used without further purification. Silver 4-chlorobenzoate (2 mmol, 0.52 g) and piperazine (1 mmol, 86 mg) were dissolved in dilute aqueous ammonia (20 ml) and the mixture stirred for about 10 min to obtain a clear solution. The solution was left to stand for 1 day to allow the ammonia gas to escape. Large brown crystals separated from the solution; these were collected, washed three times with water and dried in a vacuum desiccator using CaCl₂. The compound was isolated in 77% yield.

Crystal data

[Ag₂(C₇H₄ClO₂)₂(C₄H₁₀N₂)]
M_r = 612.98
 Orthorhombic, *Pna*2₁
a = 11.818 (4) Å
b = 7.422 (3) Å
c = 24.425 (9) Å
V = 2142.4 (14) Å³
Z = 4
D_x = 1.900 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 3794 reflections
 $\theta = 2.9\text{--}26.3^\circ$
 $\mu = 2.10\text{ mm}^{-1}$
T = 298 (2) K
 Block, brown
 0.32 × 0.11 × 0.07 mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.553, *T_{max}* = 0.867
 10524 measured reflections

3736 independent reflections
 2996 reflections with *I* > 2σ(*I*)
R_{int} = 0.045
 $\theta_{\text{max}} = 25.0^\circ$
h = -13 → 14
k = -8 → 8
l = -29 → 29

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.035
wR(*F*²) = 0.101
S = 1.01
 3736 reflections
 254 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\rho)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.56\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.69\text{ e \AA}^{-3}$
 Absolute structure: Flack (1983), with 1799 Friedel pairs
 Flack parameter = 0.00 (3)

Table 1

Selected geometric parameters (Å, °).

Ag1–N1	2.165 (6)	Ag2–O2	2.160 (5)
Ag1–O1	2.168 (5)	Ag2–N2	2.168 (6)
Ag1–Ag2	3.1716 (11)		
N1–Ag1–O1	166.5 (2)	C1–O1–Ag1	106.2 (5)
N1–Ag1–Ag2	82.51 (15)	C8–O2–Ag2	109.0 (5)
O1–Ag1–Ag2	90.05 (15)	C15–N1–Ag1	116.3 (5)
O2–Ag2–N2	166.0 (2)	C18 ⁱ –N1–Ag1	112.0 (4)
O2–Ag2–Ag1	89.63 (16)	C17–N2–Ag2	113.8 (6)
N2–Ag2–Ag1	82.59 (16)	C16–N2–Ag2	112.9 (5)

Symmetry code: (i) *x*, 1 + *y*, *z*.

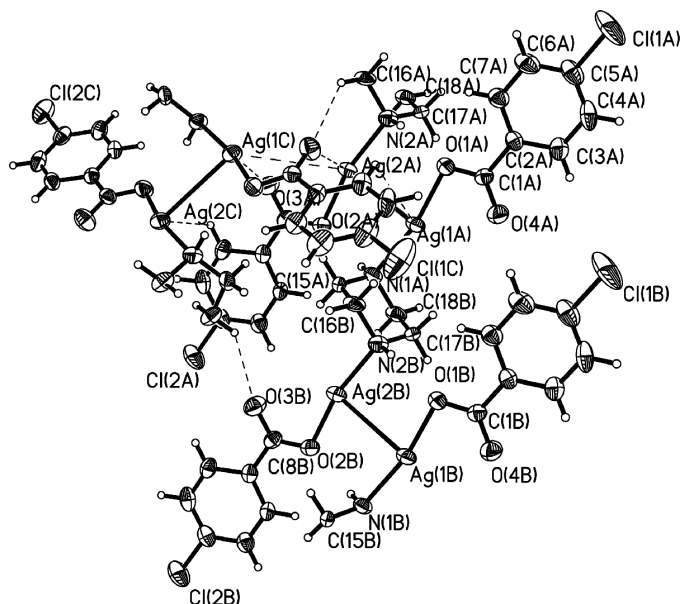


Figure 1

The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds. [Symmetry codes: (A) *x*, *y* – 1, *z*; (B) *x*, *y*, *z*; (C) *x*, *y* + 1, *z*.]

All H atoms were positioned geometrically (C–H 0.93 Å for the aromatic H atoms, C–H 0.97 Å and N–H 0.91 Å for the piperazine H atoms), and were refined as riding, with *U_{iso}*(H) = 1.2*U_{eq}*(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

The authors thank the Education Office of Anhui Province, China, for research grants 2004kJ300zd and 2004kJ302.

References

Bruker (2002). *SADABS*, *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Brammer, L., Burgard, M. D., Eddleston, M. D., Rodger, C. S., Rath, N. P. & Adams, H. (2002). *CrystEngComm*, **4**, 239–241.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Paramonov, S. E., Kuzmina, N. P. & Troyanov, S. I. (2003). *Polyhedron*, **22**, 837–841.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.