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Key indicators

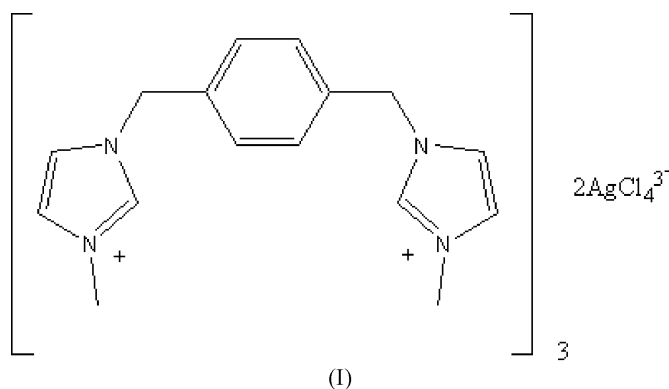
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.051
 wR factor = 0.132
Data-to-parameter ratio = 18.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3,3'-(*p*-Phenylenedimethylene)bis(1-methyl-
imidazolium)—tetrachloroargentate(I) (3/2)

The title compound, $(\text{C}_{16}\text{H}_{20}\text{N}_4)_3[\text{AgCl}_4]_2$, exists as discrete 3,3'-(1,4-phenyldimethylene)bis(1-methylimidazolium) cations and tetrachloroargentate(I) anions, which are linked by $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds [$\text{C}\cdots\text{Cl} = 3.453(5)-3.711(7)\text{ \AA}$] and $\pi-\pi$ interactions. The asymmetric unit contains one anion, one cation in a general position, and half of a cation which lies on an inversion centre.

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Comment

The most common *N*-heterocyclic carbenes (NHC) are imidazol-2-ylenes containing various *N*-substituents. In recent years, numerous chelating NHC precursors containing flexible or rigid chains have been synthesized and structurally investigated. They have attracted considerable attention because of their ability to coordinate very strongly to transition metals and main-group elements and their increasing use in organometallic chemistry and homogeneous catalysis (Herrmann & Kocher, 1997; Bourissou *et al.*, 2000; Chen & Lin, 2000; Douthwaite *et al.*, 2001; Scholl *et al.*, 1999; Lee *et al.*, 2004). We have synthesized a series of NHC precursors and metal carbene complexes, and studied their photophysical properties (Liu *et al.*, 2003). The structure of a new carbene precursor complex, *viz.* the title compound, (I), is presented here.



The asymmetric unit of (I) is shown in Fig. 1. It consists of one and a half $\text{C}_{16}\text{H}_{20}\text{N}_4^{2+}$ cations and one $[\text{AgCl}_4]^{3-}$ anion. In the tetrachloroargentate(I) anion, the coordination of Ag^{I} by the four Cl atoms can be considered as tetrahedral, with $\text{Cl}-\text{Ag}-\text{Cl}$ bond angles in the range $104.58(7)-116.14(7)^\circ$. The four $\text{Ag}-\text{Cl}$ bond distances are almost equal, ranging from $2.539(1)$ to $2.591(2)\text{ \AA}$, with an average value of 2.574 \AA (Table 1). All the $\text{C}_{16}\text{H}_{20}\text{N}_4^{2+}$ cations adopt a *trans* configuration with respect to the 1,4-phenyldimethylene group. The dihedral angles between pairs of these rings are $73.67(1)^\circ$ (N4/C13/N3/C14/C15 and C6-C11), $72.77(1)^\circ$ (N1/C2/N2/C4/C3

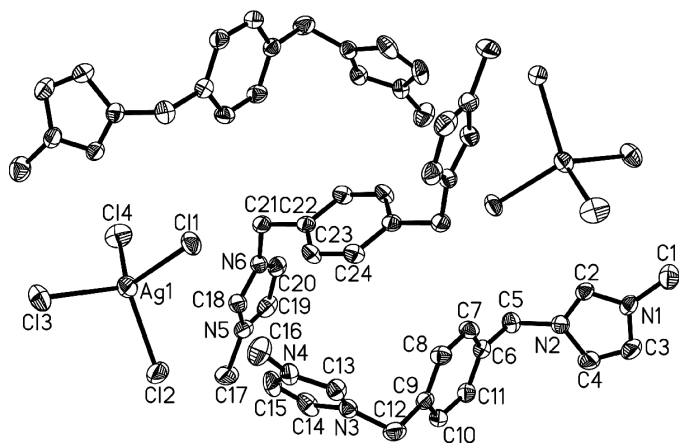


Figure 1
The structure of (I), showing 35% probability displacement ellipsoids; the atom-numbering scheme for the contents of the asymmetric unit is shown. H atoms have been omitted for clarity.

and C6–C11), 8.26 (1)° (N1/C2/N2/C4/C3 and N4/C13/N3/C14/C15) and 68.98 (1)° [N5/C18/N6/C20/C19 and C22–C24/C22ⁱ–C24ⁱ; symmetry code (i): $-x + 2, -y, -z + 1$].

In the crystal structure, the $C_{16}H_{20}N_4^{2+}$ cations and $[AgCl_4]^{3-}$ anions are linked by C–H...Cl hydrogen bonds; the C...Cl distances range from 3.453 (5) to 3.711 (7) Å (Table 2). Atom Cl2 is involved in a short C...Cl contact [3.453 (3) Å] and also in a long Ag...Cl distance [2.591 (2) Å]. This can be attributed to a relatively strong hydrogen bond existing between the ionic pair through atom Cl2 and the H atom at C12 of the imidazolium cation. In addition, there exists a weak π – π interaction between N1/C2/N2/C4/C3 rings of the cations at (x, y, z) and $(\frac{3}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z)$, with a centroid...centroid distance of 3.688 (1) Å and a perpendicular distance of 3.551 (1) Å.

Experimental

1-Methylimidazole (5.0 g, 60.98 mmol) was reacted with 1,4-dichloromethylbenzene (5.34 g, 30.49 mmol) in tetrahydrofuran under reflux to give the corresponding imidazole salt. Compound (I) was synthesized by the reaction of the imidazole salt (1.0 g, 5.45 mmol) with silver oxide (2.53 g, 10.9 mmol) in methanol (50 ml) at 333 K. Colourless single crystals of the title compound were obtained by recrystallization from dimethyl sulfoxide and diethyl ether (yield 40%).

Crystal data

$(C_{16}H_{20}N_4)_3[AgCl_4]_2$
 $M_r = 1304.42$
 Monoclinic, $P2_1/n$
 $a = 9.111$ (3) Å
 $b = 18.921$ (7) Å
 $c = 16.536$ (5) Å
 $\beta = 92.368$ (6)°
 $V = 2848.2$ (17) Å³
 $Z = 2$

$D_x = 1.521$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 962 reflections
 $\theta = 3.3$ – 24.0 °
 $\mu = 1.11$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.689, T_{max} = 0.838$
 13 029 measured reflections

5799 independent reflections
 3614 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.043$
 $\theta_{max} = 26.4$ °
 $h = -11 \rightarrow 10$
 $k = -22 \rightarrow 23$
 $l = -20 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.132$
 $S = 1.07$
 5799 reflections
 319 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 2.3376P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.97$ e Å⁻³
 $\Delta\rho_{min} = -0.40$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ag1–Cl1	2.539 (1)	N2–C5	1.482 (6)
Ag1–Cl3	2.576 (2)	N3–C12	1.475 (6)
Ag1–Cl4	2.589 (2)	N4–C16	1.463 (6)
Ag1–Cl2	2.591 (2)	C5–C6	1.514 (7)
N1–C1	1.479 (6)	C9–C12	1.511 (7)
Cl1–Ag1–Cl3	115.34 (5)	Cl4–Ag1–Cl2	116.14 (7)
Cl1–Ag1–Cl4	106.04 (6)	C2–N1–C1	125.9 (4)
Cl1–Ag1–Cl2	107.27 (5)	C2–N2–C5	126.3 (4)
Cl3–Ag1–Cl4	107.83 (6)	Cl3–N3–C12	126.5 (4)
Cl3–Ag1–Cl2	104.58 (7)	Cl3–N4–C16	126.0 (4)

Table 2

Hydrogen-bond geometry (Å, °).

D–H...A	D–H	H...A	D...A	D–H...A
C1–H1C...Cl4 ⁱ	0.96	2.81	3.703 (6)	154
C5–H5A...Cl4 ⁱⁱ	0.97	2.68	3.617 (5)	162
C5–H5B...Cl1 ⁱ	0.97	2.76	3.643 (5)	151
C12–H12A...Cl1 ⁱⁱⁱ	0.97	2.65	3.536 (6)	152
C12–H12B...Cl2 ^{iv}	0.97	2.62	3.487 (6)	148
C14–H14...Cl3 ⁱⁱⁱ	0.93	2.64	3.529 (5)	160
C15–H15...Cl2 ^v	0.93	2.53	3.453 (5)	171
C16–H16C...Cl3 ^{iv}	0.96	2.73	3.627 (6)	157
C17–H17A...Cl3 ⁱⁱⁱ	0.96	2.80	3.732 (7)	165
C17–H17B...Cl1 ^{vi}	0.96	2.73	3.557 (7)	145
C17–H17C...Cl2 ^v	0.96	2.82	3.711 (7)	154
C18–H18...Cl2 ^v	0.93	2.73	3.548 (6)	148
C20–H20...Cl3 ^{vii}	0.93	2.60	3.487 (6)	161

Symmetry codes: (i) $-x + 2, -y, -z + 1$; (ii) $-x + 1, -y, -z + 1$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (iv) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (v) x, y, z ; (vi) $x - 1, y, z$; (vii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

H atoms were placed in calculated positions, with C–H distances in the range 0.93–0.97 Å, and included in the final cycles of refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier atom})$ (1.5 U_{eq} for methyl H atoms). A PLATON (Spek, 2003) analysis showed no solvent-accessible voids in the structure.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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