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Key indicators

Single-crystal X-ray study

T = 291 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.022

wR factor = 0.061

Data-to-parameter ratio = 17.8

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

4,4'-Bipyridinium tetrachloroaurate(III) chloride

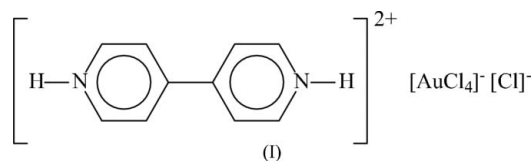
The reaction of gold(III) chloride and 4,4'-bipyridine yields the compound $(\text{C}_{10}\text{H}_8\text{N}_2)[\text{AuCl}_3]_2$, which is converted to the ionic title compound $(\text{C}_{10}\text{H}_{10}\text{N}_2)[\text{AuCl}_4]\text{Cl}$ upon recrystallization from DMSO. The Au^{III} ion in the tetrachloroaurate anion exhibits a square-planar geometry; this anion is linked to the bipyridinium cation by an $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bond to form chains which extend parallel to the *b* axis. The bipyridinium cation exhibits 2 symmetry, whereas the tetrachloroaurate anion shows $2/m$ symmetry, and the chloride anion is located on a position with 222 symmetry.

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Comment

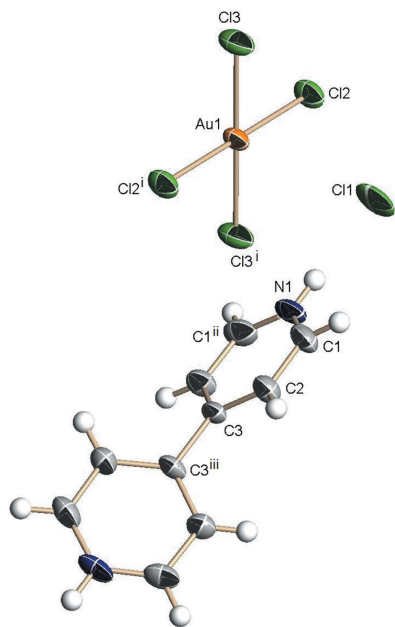
The 4,4'-bipyridine spacer heterocycle is used to bind to metals in an enormous range of complexes. However, with gold compounds, the number of gold–bipyridine adducts is small, and is limited to complexes having mostly also a phosphine ligand (Adams & Strähle, 1982; Brandys & Jennings, 2000; Li *et al.*, 2002; Lin *et al.*, 2004). The reaction of potassium tetrachloroaurate(III) with the heterocycle yields a compound in which potassium is linked to it (Song *et al.*, 2004). In our case, the reaction yielded the expected bis(trichlorogold)/4,4'-bipyridine adduct as a microcrystalline material. However, our attempts to recrystallize this product to grow larger crystals led to an unexpected compound, (I), in which the heterocycle is protonated at both N atoms. The positive charges of the dication are balanced by one tetrachloroaurate(III) and by a free chloride ion (Fig. 1).



The Au ion in the tetrachloro anion shows the common square-planar coordination, a geometry that is also found, for example, in pyridinium tetrachloroaurate (Adams & Strähle, 1982). The cation and the tetrachloroaurate anion are linked by an $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bond, with a donor–acceptor distance of 2.995 (6) Å and a linear $\text{N}-\text{H}\cdots\text{Cl}$ angle, into a chain that extends parallel to the *b* axis. Fig. 2 shows the packing of the structural units.

Experimental

The chemicals used were of technical grade and purchased from a commercial chemical company. A dichloromethane (5 ml) solution of 4,4'-bipyridine (10 mg, 0.05 mmol) was layered with a methanol

**Figure 1**

ORTEP (Johnson, 1976) plot of the formula unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, y, \frac{3}{2} - z$; (iii) $1 - x, 2 - y, z$.]

(5 ml) solution of potassium tetrachloroaurate (18 mg, 0.05 mmol) to furnish yellow microcrystals of $(C_{10}H_{10}N_2)[AuCl_4]Cl$ after one week in 90% yield. Elemental analysis (calculated): C 15.90, H 1.06, N 3.71%; found: C 16.17, H 1.10, N 3.80%. As only microcrystalline material was obtained with THF as solvent, recrystallization was attempted with DMSO. The original crystals were dissolved in the latter, and diethyl ether was diffused into the solution to afford yellow prismatic crystals of the title compound.

Crystal data

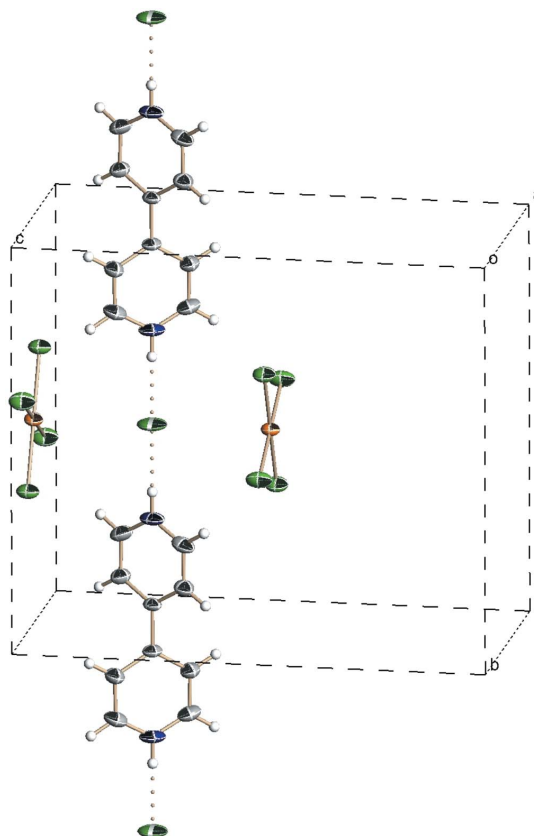
$(C_{10}H_{10}N_2)[AuCl_4]Cl$ $Z = 4$
 $M_r = 532.42$ $D_x = 2.379 \text{ Mg m}^{-3}$
 Orthorhombic, *Ibam* Mo $K\alpha$ radiation
 $a = 7.8216$ (5) Å $\mu = 10.78 \text{ mm}^{-1}$
 $b = 12.9012$ (9) Å $T = 291$ (2) K
 $c = 14.732$ (1) Å Prism, yellow
 $V = 1486.5$ (2) Å³ $0.32 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker APEX area-detector 4351 measured reflections
 diffractometer 891 independent reflections
 φ and ω scans 794 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{\text{int}} = 0.032$
 (*SADABS*; Sheldrick, 1996) $\theta_{\text{max}} = 27.5^\circ$
 $T_{\text{min}} = 0.105, T_{\text{max}} = 0.222$
 (expected range = 0.055–0.116)

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 1.1366P]$
 $R[F^2 > 2\sigma(F^2)] = 0.022$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.061$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 $S = 1.15$ $\Delta\rho_{\text{max}} = 0.97 \text{ e } \text{Å}^{-3}$
 891 reflections $\Delta\rho_{\text{min}} = -1.69 \text{ e } \text{Å}^{-3}$
 50 parameters
 H atoms treated by a mixture of independent and constrained refinement

**Figure 2**

A partial packing plot of the structural units in the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Table 1

Selected geometric parameters (Å, °).

Au1—Cl2	2.281 (1)	C2—C3	1.394 (4)
Au1—Cl3	2.282 (1)	C3—C3 ⁱ	1.48 (1)
N1—C1	1.340 (5)	N1—H1 _n	0.85 (1)
C1—C2	1.365 (5)		
Cl2—Au1—Cl2 ⁱⁱ	180	Cl2—Au1—Cl3 ⁱⁱ	90.51 (4)
Cl2—Au1—Cl3	89.49 (4)	Cl3—Au1—Cl3 ⁱⁱ	180

Symmetry codes: (i) $-x + 1, -y + 2, z$; (ii) $-x + 1, -y + 1, -z + 1$.

The two independent carbon-bound H atoms were positioned geometrically ($C-H = 0.93$ Å), and were included in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The iminium H atom was located in a difference Fourier map and was refined with a distance restraint of $N-H = 0.85$ (1) Å; its displacement parameter was refined freely. The deepest hole in the final difference Fourier map is 0.93 Å from atom Au1.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINTE* (Bruker, 2004); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2004); software used to prepare material for publication: *SHELXL97*.

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