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Dichloro(di-2-pyridylamine)gold(III) chloride

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A new gold(III) complex of di-2-pyridylamine (DPA), $[AuCl_2(C_{19}H_9N_3)]Cl$, has been synthesized and characterized by ESI–MS, ^{1}H NMR spectroscopy, elemental analysis and X-ray structural analysis. The compound possesses crystallographically imposed mirror symmetry. The DPA ligand coordinates to the gold(III) centre in a bidentate mode. The distance between the metal centre and the Cl^- anion is 3.037 (2) Å. The cations are linked into layers parallel to the *bc* plane through N—H···Cl and C—H···Cl hydrogen-bond interactions. The title complex is more cytotoxic than cisplatin against A-549 and HCT-116 tumour cell lines.

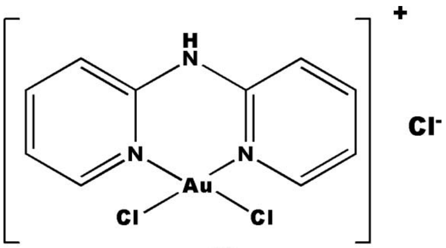
Key indicators

Single-crystal X-ray study
T = 273 K
Mean *σ*(C–C) = 0.009 Å
R factor = 0.031
wR factor = 0.082
Data-to-parameter ratio = 15.7

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

Comment

Gold(III) is isoelectronic with platinum(II), and so its complexes have long been evaluated as potential anticancer agents (Shaw, 1999). However, because of their high reductive potential, gold(III) complexes are not very stable under physiological conditions (Messori & Marcon, 2004). The essential prerequisite for any further pharmaceutical and pharmacological evaluation of gold(III) complexes is to stabilize them. Therefore, the selection of suitable ligands to achieve stable complexes is of great importance. Di-2-pyridylamine (DPA) is a very popular ligand in coordination chemistry (Fuchita *et al.*, 1998). In this paper, we report the synthesis and crystal structure of the title gold(III) complex, (I). UV-vis and ^{1}H NMR data show that the complex exhibits reasonable stability at physiologically relevant conditions.



(I)

The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are reported in Table 1. The compound possesses crystallographically imposed mirror symmetry. In the cation, the gold(III) centre is coordinated by two N atoms of DPA and two Cl atoms. The Au—N and Au—Cl bond lengths fall within the expected ranges for gold(III) complexes (Messori *et al.*, 2002). The AuN_2Cl_2 coordination has a distorted square-planar geometry, the maximum displacement from the mean plane being 0.182 (4) Å for N1. The dihedral angle formed by the pyridine ring with the

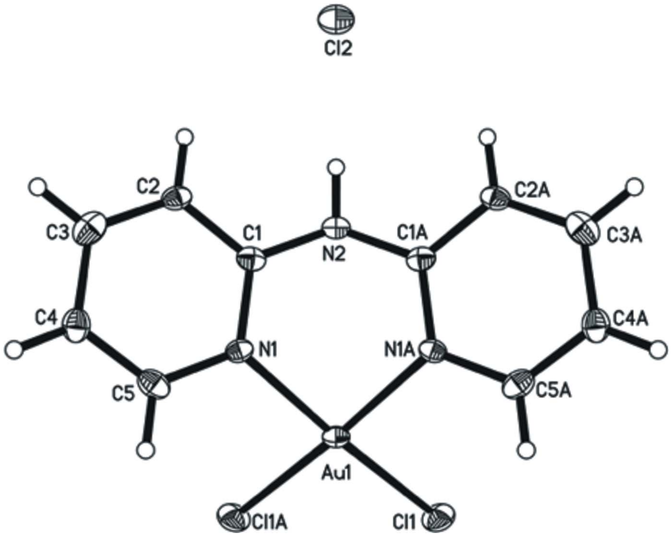


Figure 1
The molecular structure of the title complex. Displacement ellipsoids are drawn at the 30% probability level. Atoms labelled with the suffix A are related by the symmetry operator (x, $\frac{1}{2} - y$, z).

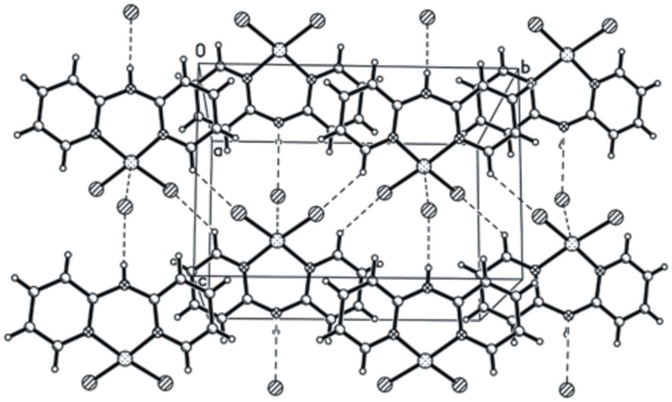


Figure 2
Packing diagram of the title complex, viewed approximately along the *a* axis. Dashed lines indicate hydrogen bonds.

coordination plane is 47.74 (14)°. The distance between the metal centre and anion Cl1 is 3.037 (2) Å.

Both independent Cl atoms are engaged in the formation of intermolecular N—H···Cl and C—H···Cl hydrogen-bond interactions (Table 2), linking complex cations into layers parallel to the *bc* plane (Figs. 2 and 3), where the separation between two adjacent layers is 3.3 Å.

Biological activity data showed that the title complex has promising *in vitro* cytotoxicity against A-549 and HCT-116 tumour cell lines.

Experimental

A mixture of di-2-pyridylamine (34 mg, 0.2 mmol) in tetrahydrofuran (5 ml) and $HAuCl_4{\cdot}H_2O$ (80 mg, 0.19 mmol) in H_2O (2 ml) was stirred at room temperature for 12 h, and the yellow precipitate which was obtained was then filtered off. Red crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a CH_3CN solution (yield 58%). Elemental analysis, calculated for $C_{10}H_9AuCl_3N_3$: C 25.31, H 1.91, N 8.86%; found: C 25.96, H 1.12, N 8.48%.

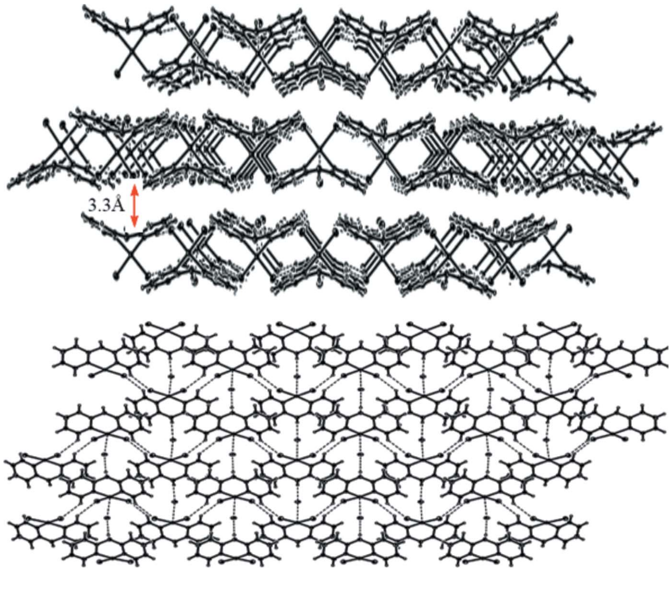


Figure 3
Packing diagrams for (I), showing the layers formed by hydrogen bonds (dashed lines), viewed along the *c* axis (top) and along the *a* axis (bottom).

Crystal data

$[AuCl_2(C_{10}H_9N_3)]Cl$
$M_r = 474.52$
Orthorhombic, *Pnma*
$a = 12.549$ (4) Å
$b = 12.313$ (4) Å
$c = 8.200$ (2) Å
$V = 1267.0$ (7) Å^3
$Z = 4$
$D_x = 2.488$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 12.22$ mm^{-1}
$T = 273$ (2) K
Block, red
0.3 × 0.22 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.043$, $T_{max} = 0.141$
6226 measured reflections
1299 independent reflections
1238 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.082$
$S = 1.10$
1299 reflections
83 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 3.0733P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.50$ e Å^{-3}
$\Delta\rho_{min} = -1.61$ e Å^{-3}
Extinction correction: *SHELXTL* (Bruker, 2000)
Extinction coefficient: 0.0010 (3)

Table 1
Selected geometric parameters (Å, °).

Au1—N1	2.015 (5)	Au1—Cl1	2.2628 (16)
N1^i—Au1—N1	87.0 (3)	N1—Au1—Cl1	174.11 (13)
N1^i—Au1—Cl1	92.18 (15)	Cl1—Au1—Cl1^i	88.02 (9)

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···Cl2	0.90	2.34	3.162 (7)	153
C5—H5···Cl1$^{\rm ii}$	0.93	2.74	3.457 (6)	135

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

All H atoms were positioned geometrically, with C—H = 0.93 Å and N—H = 0.90 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N). The maximum and minimum electron-density peaks in the final difference map were 0.96 and 1.32 Å, respectively, from atom Au1.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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