

2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline-1-ium tetrachloridoaurate(III)

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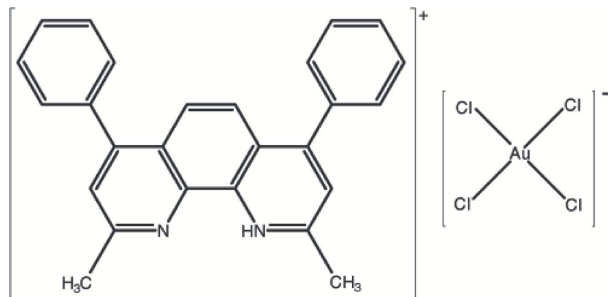
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.023; wR factor = 0.054; data-to-parameter ratio = 20.5.

Both the cation and anion of the title compound, $(\text{C}_{26}\text{H}_{21}\text{N}_2)[\text{AuCl}_4]$, are disposed about a plane of mirror symmetry. The 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline ring is oriented at a dihedral angle of $44.2(1)^\circ$ with respect to the planar phenyl ring systems. The Au^{III} atom has a square-planar environment defined by four Cl atoms. The crystal structure is stabilized by $\text{C}-\text{H}\cdots\pi$ and $\text{Au}\cdots\pi$ ring-metal (3.551 Å) interactions. In the crystal structure, the molecules stack along the c axis via $\text{N}-\text{H}\cdots\text{N}$ hydrogen-bond interactions.

Related literature

For general background to proton-transfer systems and their structures, see: Abedi *et al.* (2008); Amani *et al.* (2008); Calleja *et al.* (2001); Hasan *et al.* (1999); Hojjat Kashani *et al.* (2008); Johnson & Steed (1998); Karaca *et al.* (2009); Yap *et al.* (1995); Zhang *et al.* (2006).



Experimental

Crystal data

$(\text{C}_{26}\text{H}_{21}\text{N}_2)[\text{AuCl}_4]$
 $M_r = 700.22$
Orthorhombic, $Pnma$
 $a = 13.5195(10)$ Å
 $b = 22.9565(17)$ Å
 $c = 7.5556(6)$ Å

$V = 2345.0(3)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 6.75$ mm⁻¹
 $T = 150$ K
 $0.20 \times 0.06 \times 0.04$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
 $T_{\text{min}} = 0.345$, $T_{\text{max}} = 0.774$

24337 measured reflections
3265 independent reflections
2835 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.054$
 $S = 1.03$
3265 reflections
159 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.85$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.60$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H}\text{N1}\cdots\text{N1}^{\text{i}}$	0.76 (5)	2.28 (5)	2.646 (3)	111 (4)
$\text{C1}-\text{H1B}\cdots\text{Cg3}^{\text{ii}}$	0.96	2.76	3.574 (3)	143

Symmetry codes: (i) $x, -y + \frac{1}{2}, z$; (ii) $x + \frac{1}{2}, y, -z + \frac{1}{2}$. Cg3 is the centroid of the C8–C13 ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2496).

References

- Abedi, A., Bahrami Shabestari, A. & Amani, V. (2008). *Acta Cryst.* **E64**, o990.
Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Amani, V., Rahimi, R. & Khavasi, H. R. (2008). *Acta Cryst.* **E64**, m1143–m1144.
Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Calleja, M., Johnson, K., Belcher, W. J. & Steed, W. (2001). *Inorg. Chem.* **40**, 4978–4985.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hasan, M., Kozhevnikov, I. V., Siddiqui, M. R. H., Steiner, A. & Winterton, N. (1999). *Inorg. Chem.* **38**, 5637–5641.
Hojjat Kashani, L., Yousefi, M., Amani, V. & Khavasi, H. R. (2008). *Acta Cryst.* **E64**, m840–m841.
Johnson, K. & Steed, J. W. (1998). *Chem. Commun.* pp. 1479–1480.
Karaca, S., Akkurt, M., Safari, N., Amani, V., Büyükgüngör, O. & Abedi, A. (2009). *Acta Cryst.* **E65**, m235.

metal-organic compounds

Sheldrick, G. M. (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Yap, G. P. A., Rheingold, A. R., Das, P. & Crabtree, R. H. (1995). *Inorg. Chem.* **34**, 3474–3476.

Zhang, X.-P., Yang, G. & Ng, S. W. (2006). *Acta Cryst.* **E62**, m2018–m2020.

supplementary materials

Acta Cryst. (2009). E65, m479-m480 [doi:10.1107/S1600536809011994]

2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline-1-ium tetrachloridoaurate(III)

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Comment

In recent years, there has been considerable interest in proton transfer systems and their structures (Amani *et al.*, 2008; Abedi *et al.*, 2008; Karaca *et al.*, 2009). Several proton transfer systems using H₂AuCl₄ with proton acceptor molecules, such as [EMI][AuCl₄] and [BMI]₂[AuCl₄].2H₂O (Hasan *et al.*, 1999), [H₂bipy][AuCl₄][Cl] (Zhang *et al.*, 2006), [H₇O₃][15-crown-5][AuCl₄] and [H₅O₂][benzo-15-crown-5]₂[AuCl₄] (Johnson & Steed, 1998), [H₅O₂]₂[12-crown-4]₂[AuCl₄]₂, [H₃O][18-crown-6][AuCl₄] and [H₃O][4-nitrobenzo-18-crown-6][AuCl₄] (Calleja *et al.*, 2001), [DPPy.H][AuCl₄] (Yap *et al.*, 1995) and [H₂DA18C6][AuCl₄].2H₂O (Hojjat Kashani *et al.*, 2008) [where EMI is 1-ethyl-3-methylimidazolium, BMI is 1-butyl-3-methylimidazolium, H₂bipy is 2, 2'-bipyridinium, DPPy.H is 2,6-diphenylpyridinium and H₂DA18C6 is 1,10-diazonia-18-crown-6] have been synthesized and characterized by single-crystal X-ray diffraction methods. We report herein the synthesis and crystal structure of the title compound, (I).

Both the cation and anion of the title compound (Fig. 1) are disposed about a plane of mirror symmetry. The 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline ring is oriented at a dihedral angle of 44.2 (1)° with respect to the planar phenyl ring systems. The Au ion has a square-planar environment defined by four Cl atoms. In AuCl₄ anion, the Au—Cl bond lengths and Cl—Au—Cl bond angles are normal ranges. In the crystal structure, there exist ring-metal interactions [Cg2...Au1(*x*, *y*, *z*) = 3.551 Å and Cg2...Au1(*x*, 1/2 - *y*, *z*) = 3.551 Å, where Cg2 is a centroid of the central benzene ring (C5–C7/C5b–C7b) of the cation molecule]. For C1—H1B...Cg3 interaction, C1...Cg3 = 3.574 Å, where Cg3 is a centroid of the phenyl ring (C8–C13) (Table 2). View of the packing of (I) down the *c*-axis is given in Fig. 2.

Experimental

For the preparation of the title compound, a solution of 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (0.15 g, 0.44 mmol) in HCCl₃ (10 ml) was added to a solution of H₂AuCl₄.3H₂O, (0.17 g, 0.44 mmol) in ethanol (5 ml) and the resulting yellow solution was stirred for 15 min at 313 K. This solution was left to evaporate slowly at room temperature. After one week, yellow prismatic crystals of the title compound were isolated [yield; 0.23 g; 72.1%; m. p. 495 K].

Refinement

All H atoms were seen in the difference electron density map. The H atom HN1 bound to atom N1 were refined isotropically. Since the anion group of the title molecule has symmetrically two parts, the site occupation factor of the HN1 atom were fixed at the value of 0.5. The other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 and 0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$. The highest residual peak is located 0.87 Å from atom Au1 and the deepest hole is located 0.57 Å from atom Au1.

Figures

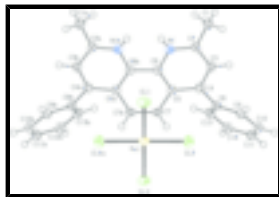


Fig. 1. ORTEP drawing of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level [Symmetry codes: (a and b) $x, 1/2 - y, z$].

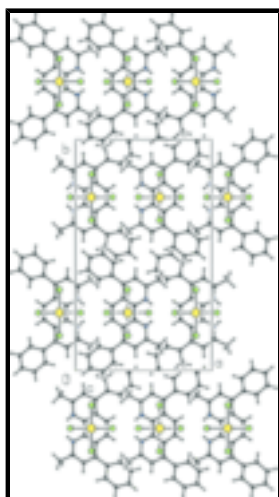


Fig. 2. The packing of the title compound viewed down c axis.

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Crystal data

(C₂₆H₂₁N₂)[AuCl₄]

$M_r = 700.22$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 13.5195$ (10) Å

$b = 22.9565$ (17) Å

$c = 7.5556$ (6) Å

$V = 2345.0$ (3) Å³

$Z = 4$

$F_{000} = 1352$

$D_x = 1.983$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Cell parameters from 5918 reflections

$\theta = 2.8$ – 27.6°

$\mu = 6.75$ mm⁻¹

$T = 150$ K

Prism, yellow

$0.20 \times 0.06 \times 0.04$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: sealed tube

Monochromator: graphite

$T = 150$ K

φ and ω scans

Absorption correction: multi-scan

3265 independent reflections

2835 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

$\theta_{\text{max}} = 29.2^\circ$

$\theta_{\text{min}} = 1.8^\circ$

$h = -18 \rightarrow 18$

(SADABS; Sheldrick, 2003)

$T_{\min} = 0.345$, $T_{\max} = 0.774$

24337 measured reflections

$k = -31 \rightarrow 31$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.023$

$wR(F^2) = 0.054$

$S = 1.03$

3265 reflections

159 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 1.1102P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.85 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.60 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.51546 (16)	0.19238 (10)	0.0058 (3)	0.0203 (7)	
C1	0.61127 (19)	0.10789 (13)	-0.0819 (4)	0.0271 (8)	
C2	0.52022 (19)	0.13465 (11)	-0.0049 (3)	0.0202 (8)	
C3	0.4389 (2)	0.10119 (12)	0.0488 (4)	0.0219 (8)	
C4	0.3533 (2)	0.12606 (12)	0.1132 (3)	0.0200 (7)	
C5	0.35028 (19)	0.18757 (11)	0.1303 (3)	0.0181 (7)	
C6	0.43300 (19)	0.21879 (11)	0.0701 (3)	0.0190 (7)	
C7	0.27048 (18)	0.22031 (10)	0.2068 (3)	0.0182 (7)	
C8	0.26628 (19)	0.08820 (11)	0.1485 (3)	0.0199 (7)	
C9	0.2791 (2)	0.03400 (11)	0.2301 (4)	0.0237 (8)	
C10	0.2004 (2)	-0.00427 (12)	0.2445 (4)	0.0272 (8)	
C11	0.1082 (2)	0.01067 (13)	0.1803 (4)	0.0279 (8)	
C12	0.0937 (2)	0.06484 (13)	0.1013 (4)	0.0263 (8)	
C13	0.1724 (2)	0.10337 (12)	0.0840 (3)	0.0230 (8)	

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Au1	0.38322 (1)	0.25000	0.60223 (2)	0.0219 (1)	
C11	0.53754 (7)	0.25000	0.48086 (15)	0.0343 (3)	
C12	0.38270 (5)	0.15042 (4)	0.60263 (10)	0.0336 (2)	
C13	0.22961 (7)	0.25000	0.72842 (14)	0.0298 (3)	
HN1	0.556 (4)	0.212 (2)	-0.029 (7)	0.009 (14)*	0.500
H1A	0.59680	0.09310	-0.19790	0.0410*	
H1B	0.63310	0.07660	-0.00740	0.0410*	
H1C	0.66240	0.13680	-0.08990	0.0410*	
H3	0.44280	0.06080	0.04060	0.0260*	
H7	0.21760	0.20050	0.25760	0.0220*	
H9	0.34070	0.02370	0.27490	0.0280*	
H10	0.20980	-0.04040	0.29790	0.0330*	
H11	0.05590	-0.01540	0.18980	0.0340*	
H12	0.03130	0.07520	0.06010	0.0310*	
H13	0.16280	0.13930	0.02960	0.0280*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0164 (11)	0.0215 (11)	0.0230 (12)	-0.0001 (9)	0.0006 (9)	0.0006 (9)
C1	0.0232 (14)	0.0249 (14)	0.0333 (16)	0.0047 (11)	0.0024 (12)	-0.0025 (12)
C2	0.0215 (13)	0.0200 (13)	0.0191 (13)	0.0039 (10)	-0.0020 (10)	-0.0022 (10)
C3	0.0242 (14)	0.0194 (13)	0.0220 (13)	0.0028 (10)	-0.0005 (10)	-0.0008 (10)
C4	0.0204 (12)	0.0205 (12)	0.0192 (12)	-0.0002 (10)	-0.0012 (10)	0.0019 (10)
C5	0.0176 (11)	0.0199 (12)	0.0167 (12)	0.0000 (10)	-0.0030 (9)	0.0014 (9)
C6	0.0186 (12)	0.0205 (13)	0.0180 (12)	0.0000 (10)	-0.0032 (9)	0.0014 (10)
C7	0.0174 (11)	0.0204 (12)	0.0168 (12)	-0.0035 (9)	0.0008 (9)	-0.0002 (10)
C8	0.0207 (12)	0.0198 (12)	0.0193 (12)	-0.0009 (10)	0.0004 (10)	-0.0022 (10)
C9	0.0226 (12)	0.0229 (13)	0.0256 (14)	0.0033 (10)	0.0005 (11)	0.0017 (11)
C10	0.0325 (15)	0.0208 (13)	0.0284 (15)	0.0018 (11)	0.0057 (12)	0.0040 (11)
C11	0.0292 (14)	0.0266 (14)	0.0280 (15)	-0.0071 (12)	0.0058 (12)	-0.0028 (12)
C12	0.0218 (13)	0.0292 (15)	0.0278 (14)	0.0010 (11)	-0.0015 (11)	-0.0031 (12)
C13	0.0246 (14)	0.0210 (13)	0.0233 (13)	0.0025 (10)	-0.0018 (11)	-0.0004 (10)
Au1	0.0177 (1)	0.0292 (1)	0.0187 (1)	0.0000	-0.0022 (1)	0.0000
C11	0.0186 (4)	0.0474 (6)	0.0370 (6)	0.0000	0.0020 (4)	0.0000
C12	0.0326 (4)	0.0297 (4)	0.0386 (4)	0.0049 (3)	0.0006 (3)	0.0074 (3)
C13	0.0212 (4)	0.0383 (6)	0.0298 (5)	0.0000	0.0043 (4)	0.0000

Geometric parameters (\AA , $^\circ$)

Au1—C12 ⁱ	2.2860 (9)	C8—C13	1.404 (4)
Au1—C13	2.2852 (10)	C8—C9	1.399 (4)
Au1—C11	2.2790 (10)	C9—C10	1.384 (4)
Au1—C12	2.2860 (9)	C10—C11	1.381 (4)
N1—C2	1.329 (3)	C11—C12	1.393 (4)
N1—C6	1.359 (3)	C12—C13	1.390 (4)
N1—HN1	0.76 (5)	C1—H1B	0.9600
C1—C2	1.494 (4)	C1—H1C	0.9600

C2—C3	1.401 (4)	C1—H1A	0.9600
C3—C4	1.379 (4)	C3—H3	0.9300
C4—C5	1.419 (4)	C7—H7	0.9300
C4—C8	1.487 (4)	C9—H9	0.9300
C5—C6	1.404 (4)	C10—H10	0.9300
C5—C7	1.436 (3)	C11—H11	0.9300
C6—C6 ⁱ	1.433 (4)	C12—H12	0.9300
C7—C7 ⁱ	1.363 (3)	C13—H13	0.9300
Au1…C7 ⁱ	3.423 (2)	C13…C7	3.135 (4)
Au1…C7	3.423 (2)	C3…H10 ^{xii}	3.1000
C11…C13 ⁱⁱ	3.4011 (15)	C3…H9	2.8000
C11…C7 ⁱⁱⁱ	3.520 (3)	C5…H13	2.8700
C11…C12	3.2333 (11)	C7…H13	2.7100
C11…C6	3.485 (3)	C8…H10 ^{xii}	2.8900
C11…C13 ^{iv}	3.4011 (15)	C8…H7	2.7900
C11…C7 ^v	3.520 (3)	C9…H3	2.7100
C11…C12 ⁱ	3.2333 (11)	C9…H1B ^{ix}	3.0400
C11…C6 ⁱ	3.485 (3)	C10…H1B ^{ix}	2.8700
C12…C11	3.2333 (11)	C11…H1B ^{ix}	2.9200
C12…C3 ^{vi}	3.636 (3)	C13…H10 ^{xii}	3.0500
C12…C13	3.2269 (11)	C13…H7	2.6600
C12…C2 ^{vi}	3.519 (2)	HN1…C13 ⁱⁱⁱ	2.92 (5)
C13…C11 ^{vii}	3.4011 (15)	HN1…H1C	2.2900
C13…C12	3.2269 (11)	HN1…C13 ^v	2.92 (5)
C13…C11 ^{viii}	3.4011 (15)	HN1…N1 ⁱ	2.28 (5)
C13…C12 ⁱ	3.2269 (11)	H1B…C11 ⁱⁱⁱ	2.9200
C11…H13 ^v	3.0500	H1B…C10 ⁱⁱⁱ	2.8700
C11…H13 ⁱⁱⁱ	3.0500	H1B…C9 ⁱⁱⁱ	3.0400
C12…H12 ⁱⁱⁱ	2.9200	H1C…HN1	2.2900
C12…H1C ^{ix}	3.0000	H1C…C13 ^v	2.9500
C13…HN1 ^x	2.92 (5)	H1C…C12 ⁱⁱⁱ	3.0000
C13…HN1 ^{ix}	2.92 (5)	H1C…C13 ⁱⁱⁱ	2.9500
C13…H1C ^x	2.9500	H3…H9	2.4000
C13…H1C ^{ix}	2.9500	H3…C9	2.7100
N1…N1 ⁱ	2.646 (3)	H7…C13	2.6600
N1…HN1 ⁱ	2.28 (5)	H7…C8	2.7900
C2…C12 ⁱⁱⁱ	3.585 (4)	H7…H13	2.3400
C2…C12 ^{xi}	3.519 (2)	H9…H3	2.4000
C3…C12 ⁱⁱⁱ	3.474 (4)	H9…C3	2.8000
C3…C12 ^{xi}	3.636 (3)	H10…C13 ^{xiii}	3.0500
C6…C11	3.485 (3)	H10…C3 ^{xiii}	3.1000
C6…C11	3.485 (3)	H10…C8 ^{xiii}	2.8900

supplementary materials

C7...C13	3.135 (4)	H12...C12 ^{ix}	2.9200
C7...Au1	3.423 (2)	H13...C7	2.7100
C7...Au1	3.423 (2)	H13...H7	2.3400
C7...C11 ^{ix}	3.520 (3)	H13...C11 ^x	3.0500
C7...C11 ^x	3.520 (3)	H13...C11 ^{ix}	3.0500
C12...C3 ^{ix}	3.474 (4)	H13...C5	2.8700
C12...C2 ^{ix}	3.585 (4)		
C11—Au1—C13	179.07 (4)	C8—C9—C10	120.3 (3)
C11—Au1—C12 ⁱ	90.19 (2)	C9—C10—C11	120.6 (3)
C12—Au1—C13	89.81 (2)	C10—C11—C12	120.0 (3)
C12—Au1—C12 ⁱ	179.62 (3)	C11—C12—C13	120.0 (3)
C12 ⁱ —Au1—C13	89.81 (2)	C8—C13—C12	120.1 (2)
C11—Au1—C12	90.19 (2)	H1A—C1—H1C	109.00
C2—N1—C6	120.4 (2)	C2—C1—H1A	109.00
C6—N1—HN1	117 (4)	C2—C1—H1B	109.00
C2—N1—HN1	123 (4)	C2—C1—H1C	109.00
C1—C2—C3	122.3 (2)	H1A—C1—H1B	109.00
N1—C2—C3	119.4 (2)	H1B—C1—H1C	109.00
N1—C2—C1	118.3 (2)	C2—C3—H3	119.00
C2—C3—C4	122.3 (3)	C4—C3—H3	119.00
C3—C4—C8	119.0 (2)	C7 ⁱ —C7—H7	119.00
C5—C4—C8	122.9 (2)	C5—C7—H7	119.00
C3—C4—C5	117.9 (2)	C8—C9—H9	120.00
C6—C5—C7	117.5 (2)	C10—C9—H9	120.00
C4—C5—C7	125.4 (2)	C9—C10—H10	120.00
C4—C5—C6	117.1 (2)	C11—C10—H10	120.00
C5—C6—C6 ⁱ	120.7 (2)	C12—C11—H11	120.00
N1—C6—C6 ⁱ	116.5 (2)	C10—C11—H11	120.00
N1—C6—C5	122.8 (2)	C11—C12—H12	120.00
C5—C7—C7 ⁱ	121.6 (2)	C13—C12—H12	120.00
C4—C8—C13	120.6 (2)	C12—C13—H13	120.00
C9—C8—C13	119.1 (2)	C8—C13—H13	120.00
C4—C8—C9	120.1 (2)		
C6—N1—C2—C1	178.1 (2)	C7—C5—C6—N1	175.7 (2)
C6—N1—C2—C3	0.8 (4)	C7—C5—C6—C6 ⁱ	-5.8 (3)
C2—N1—C6—C5	1.0 (4)	C4—C5—C7—C7 ⁱ	-175.3 (2)
C2—N1—C6—C6 ⁱ	-177.6 (2)	C6—C5—C7—C7 ⁱ	5.9 (3)
N1—C2—C3—C4	-0.1 (4)	N1—C6—C6 ⁱ —N1 ⁱ	0.0 (3)
C1—C2—C3—C4	-177.3 (3)	N1—C6—C6 ⁱ —C5 ⁱ	178.6 (2)
C2—C3—C4—C5	-2.2 (4)	C5—C6—C6 ⁱ —N1 ⁱ	-178.6 (2)
C2—C3—C4—C8	173.2 (2)	C5—C6—C6 ⁱ —C5 ⁱ	0.0 (4)
C3—C4—C5—C6	3.7 (3)	C5—C7—C7 ⁱ —C5 ⁱ	0.0 (4)
C3—C4—C5—C7	-175.1 (2)	C4—C8—C9—C10	-172.4 (2)
C8—C4—C5—C6	-171.5 (2)	C13—C8—C9—C10	0.9 (4)

C8—C4—C5—C7	9.6 (4)	C4—C8—C13—C12	173.2 (2)
C3—C4—C8—C9	42.3 (3)	C9—C8—C13—C12	-0.1 (4)
C3—C4—C8—C13	-130.8 (3)	C8—C9—C10—C11	-0.6 (4)
C5—C4—C8—C9	-142.5 (3)	C9—C10—C11—C12	-0.4 (5)
C5—C4—C8—C13	44.4 (3)	C10—C11—C12—C13	1.3 (4)
C4—C5—C6—N1	-3.2 (3)	C11—C12—C13—C8	-1.0 (4)
C4—C5—C6—C6 ⁱ	175.3 (2)		

Symmetry codes: (i) $x, -y+1/2, z$; (ii) $x+1/2, y, -z+3/2$; (iii) $x+1/2, y, -z+1/2$; (iv) $x+1/2, -y+1/2, -z+3/2$; (v) $x+1/2, -y+1/2, -z+1/2$; (vi) $x, y, z+1$; (vii) $x-1/2, y, -z+3/2$; (viii) $x-1/2, -y+1/2, -z+3/2$; (ix) $x-1/2, y, -z+1/2$; (x) $x-1/2, -y+1/2, -z+1/2$; (xi) $x, y, z-1$; (xii) $-x+1/2, -y, z-1/2$; (xiii) $-x+1/2, -y, z+1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—HN1 ⁱ —N1 ⁱ	0.76 (5)	2.28 (5)	2.646 (3)	111 (4)
C1—H1B ⁱⁱⁱ —Cg3 ⁱⁱⁱ	0.96	2.76	3.574 (3)	143

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

Fig. 1

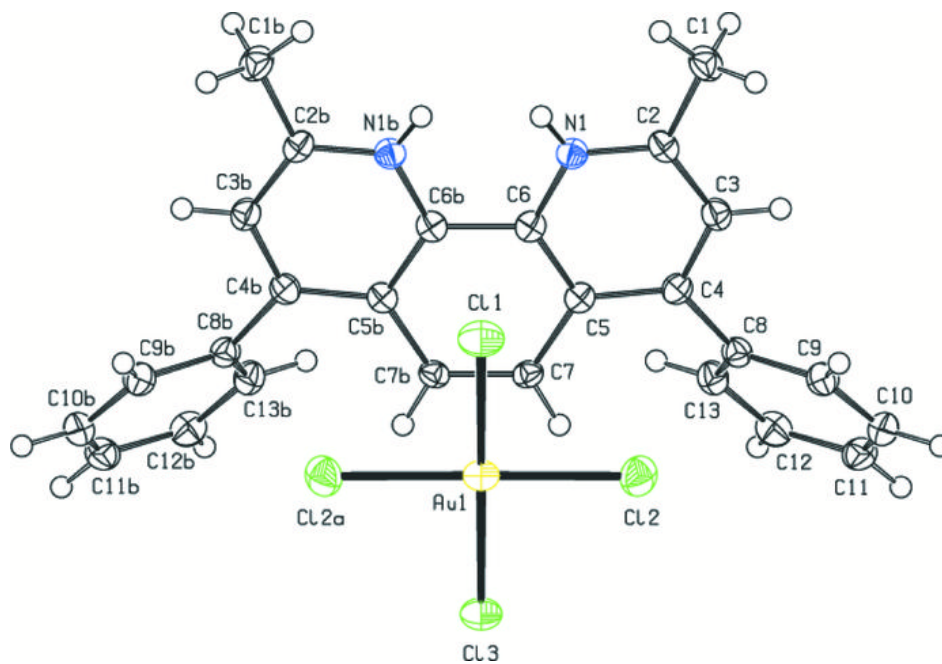


Fig. 2

