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# Crystal structure of 8-iodoquinolinium tetrachloridoaurate(III)

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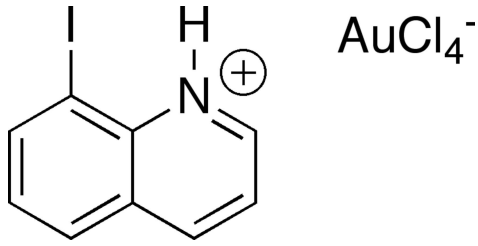
The structure of the title salt, $(C_9H_7IN)[AuCl_4]$, is comprised of planar 8-iodoquinolinium cations (r.m.s. deviation = 0.05 Å) and square-planar tetrachloridoaurate(III) anions. The asymmetric unit contains one 8-iodoquinolinium cation and two halfs of $[AuCl_4]^-$ anions, in each case with the central $Au^{III}$ atom located on an inversion center. Intermolecular halogen–halogen contacts were found between centrosymmetric pairs of I [3.6178 (4) Å] and Cl atoms [3.1484 (11), 3.3762 (13), and 3.4935 (12) Å]. Intermolecular N—H···Cl and C—H···Cl hydrogen bonding is also found in the structure. These interactions lead to the formation of a three-dimensional network. Additionally, there is an intramolecular N—H···I hydrogen bond between the aromatic iminium and iodine. There are no aurophilic interactions or short contacts between I and Au atoms, and there are no notable $\pi$-stacking interactions between the aromatic cations.

**Keywords:** crystal structure; 8-iodoquinolinium cation; tetrachloridoaurate anion; salt structure.

**CCDC reference**: 1438910

## 1. Related literature

There are only two reported structures containing the 8-iodoquinolinium cation, *viz*. 8-iodoquinolinium chloride dihydrate (Son & Hoefelmeyer, 2008*a*) and 8-iodoquinolinium triiodide tetrahydrofuran solvate (Son & Hoefelmeyer, 2008*b*). Recently, the zwitterionic 8-iodoquinoline *N*-oxide was also reported (Hwang *et al.*, 2014).




## 2. Experimental

### 2.1. Crystal data

$(C_9H_7IN)[AuCl_4]$
$M_r$ = 594.82
Triclinic, $P\overline{1}$
$a$ = 7.6299 (5) Å
$b$ = 7.8609 (5) Å
$c$ = 11.7125 (7) Å
$\alpha$ = 80.160 (1)°
$\beta$ = 78.143 (1)°
$\gamma$ = 85.178 (1)°
$V$ = 676.52 (7) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 13.92 mm$^{-1}$
$T$ = 100 K
0.16 × 0.11 × 0.04 mm

### 2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.174, $T_{max}$ = 0.573
6855 measured reflections
2482 independent reflections
2407 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.040
$S$ = 1.04
2482 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.94 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H99···Cl3$^{i}$ | 0.80 (5) | 2.62 (5) | 3.287 (3) | 142 (4) |
| N1—H99···I1 | 0.80 (5) | 2.81 (5) | 3.264 (3) | 118 (4) |
| C2—H2···Cl1$^{ii}$ | 0.93 | 2.79 | 3.493 (4) | 133 |
| C3—H3···Cl1$^{iii}$ | 0.93 | 2.81 | 3.722 (4) | 168 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrea *et al.*, 2006); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

## Acknowledgements

Purchase of the X-ray diffractometer was made possible by funding from the National Science Foundation (grant No. EPS-0554609).

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5236).

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# supporting information

*Acta Cryst.* (2015). E**71**, m261–m262 [https://doi.org/10.1107/S2056989015022574]

## Crystal structure of 8-iodoquinolinium tetrachloridoaurate(III)

**Benard O. Onserio, Sem Raj Tamang and James D. Hoefelmeyer**

### S1. Synthesis and crystallization

In a 4 ml vial, $HAuCl_4·3H_2O$ (0.12 g, 0.33 mmol), 8-iodoquinoline (0.10 g, 0.39 mmol) and acetonitrile (2 ml) were combined and sonicated for 30 minutes. The 4 ml vial was placed in a 20 ml vial with 5 ml diethylether. Diffusion of the ether vapor into the solution within the smaller vial gave yellow-green crystals, mostly with a cuboid-like form.

### S2. Refinement

C-bound H atoms were placed in ideal positions and refined as riding atoms (C—H = 0.93 Å; $U_{iso}$(H) = 1.2$U_{eq}$(H)). The H atom bound to the N atom was located from a difference map and refined freely. The highest remaining electron density peak was located 0.20 Å from H6. A transmission factor of 0.62 was calculated using the ratio of $T_{min}$ (0.4593) to $T_{max}$ (0.7452) taken from the absorption correction output file, whereas experimental $T_{min}$ (0.174) and $T_{max}$ (0.573) give a transmission factor of 0.30.

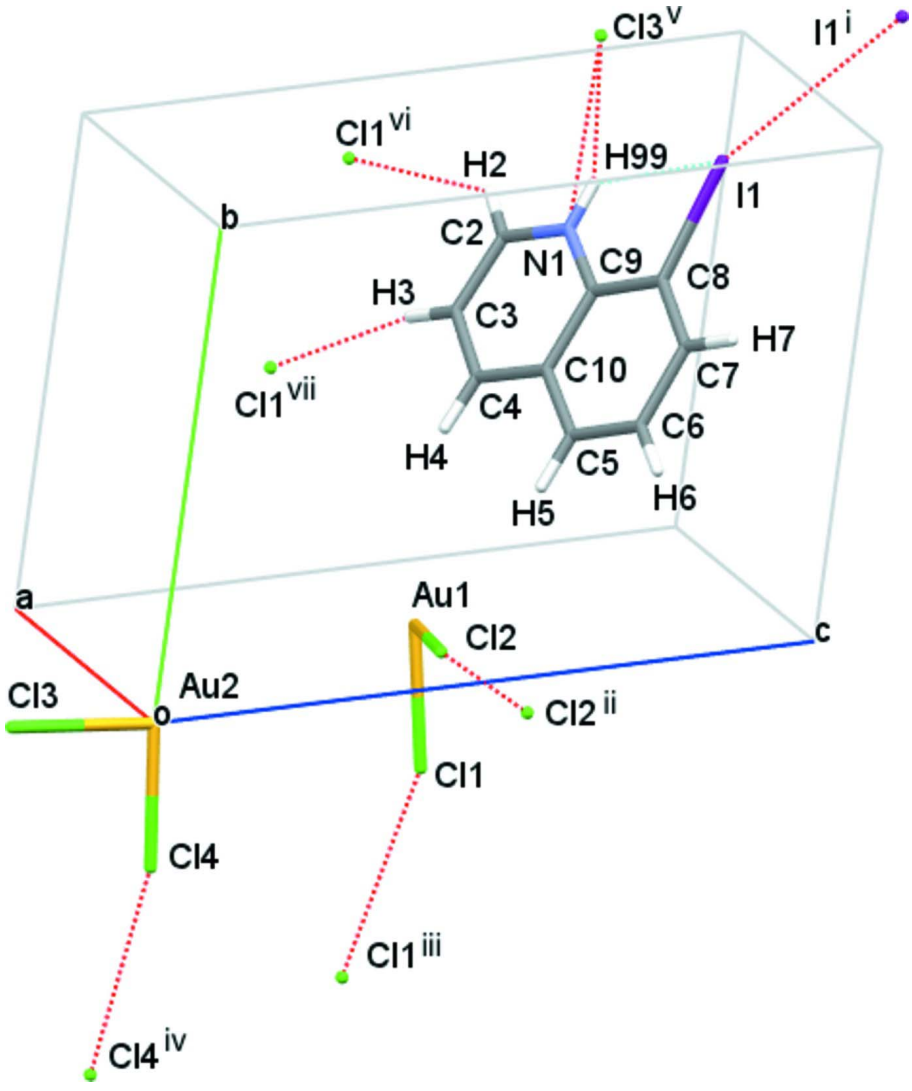


**Figure 1**

The expanded asymmetric unit of the crystal shown with intermolecular halogen···halogen contacts and hydrogen bonds as dashed lines. [Symmetry codes: (i) 1 - *x*, 2 - *y*, 2 - *z*; (ii) 2 - *x*, -*y*, 1 - *z*; (iii) 1 - *x*, -1 - *y*, 1 - *z*; (iv) -*x*, -*y* - 1, -*z*; (v) *x* + 1, y + 1, *z* + 1; (vi) *x* + 1, *y* + 1, *z*; (vii) -*x* + 2, -*y*, -*z* + 1.]

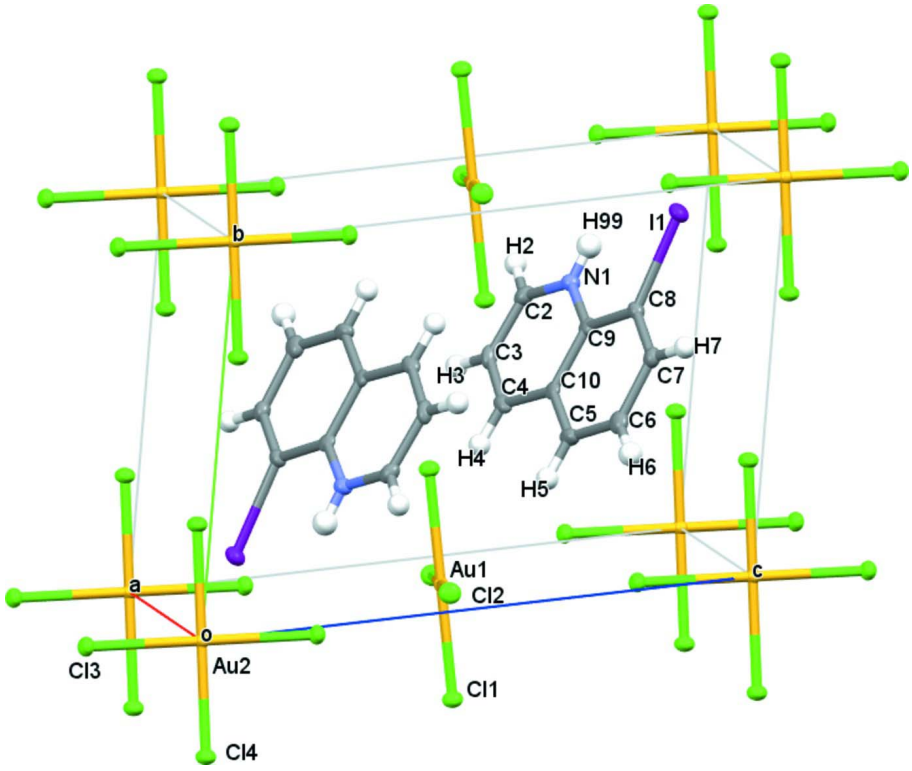


**Figure 2**

The centrosymmetric unit cell of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

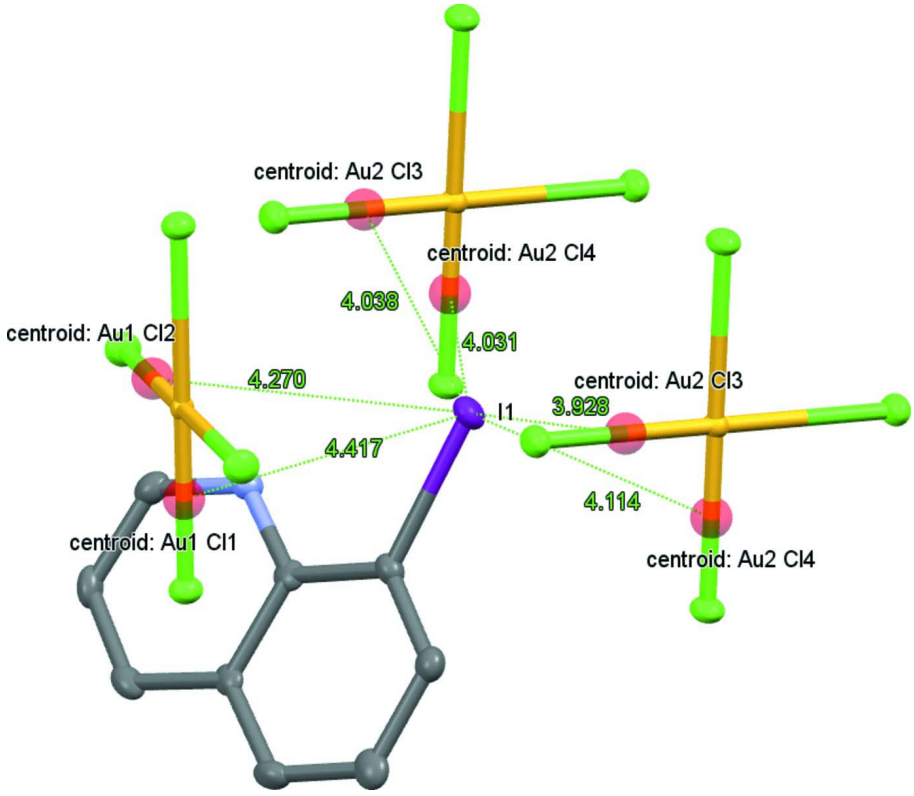


**Figure 3**

Examination of the nearest distances (Å) between iodine and Au—Cl bond centroids. These distances are beyond the sum of the van der Waals radii of the atoms.

### 8-Iodoquinolinium tetrachloridoaurate(III)

*Crystal data*

$(C_9H_7IN)[AuCl_4]$
$M_r$ = 594.82
Triclinic, $P\overline{1}$
$a$ = 7.6299 (5) Å
$b$ = 7.8609 (5) Å
$c$ = 11.7125 (7) Å
$\alpha$ = 80.160 (1)°
$\beta$ = 78.143 (1)°
$\gamma$ = 85.178 (1)°
$V$ = 676.52 (7) Å$^3$
$Z$ = 2
$F(000)$ = 536
$D_x$ = 2.920 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 5508 reflections
$\theta$ = 2.6–25.6°
$\mu$ = 13.92 mm$^{-1}$
$T$ = 100 K
Plate, light green
0.16 × 0.11 × 0.04 mm

*Data collection*

Bruker APEXII CCD diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.174, $T_{max}$ = 0.573
6855 measured reflections
2482 independent reflections
2407 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 1.8°
$h$ = −9→9
$k$ = −9→9
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.016$
$wR(F^2) = 0.040$
$S = 1.04$
2482 reflections
152 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.019P)^2 + 0.5573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.19$ e Å$^{-3}$
$\Delta\rho_{min} = -0.94$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| I1 | 0.54637 (3) | 0.86196 (3) | 0.88748 (2) | 0.01671 (7) |
| Au2 | 0.0000 | 0.0000 | 0.0000 | 0.01009 (6) |
| Au1 | 0.5000 | 0.0000 | 0.5000 | 0.00870 (6) |
| Cl3 | 0.07524 (11) | 0.00792 (10) | −0.20000 (7) | 0.01714 (17) |
| Cl2 | 0.20893 (10) | 0.01686 (10) | 0.47699 (7) | 0.01590 (17) |
| Cl4 | 0.00376 (12) | −0.29552 (11) | 0.02554 (8) | 0.01672 (18) |
| C8 | 0.5841 (5) | 0.6220 (4) | 0.8290 (3) | 0.0137 (7) |
| C7 | 0.4426 (5) | 0.5185 (5) | 0.8453 (3) | 0.0167 (8) |
| H7 | 0.3321 | 0.5507 | 0.8887 | 0.020* |
| C6 | 0.4621 (5) | 0.3624 (5) | 0.7968 (3) | 0.0201 (8) |
| H6 | 0.3655 | 0.2923 | 0.8092 | 0.024* |
| C5 | 0.6244 (5) | 0.3160 (5) | 0.7317 (3) | 0.0173 (8) |
| H5 | 0.6359 | 0.2155 | 0.6982 | 0.021* |
| C10 | 0.7737 (5) | 0.4170 (4) | 0.7144 (3) | 0.0136 (7) |
| C4 | 0.9432 (5) | 0.3733 (5) | 0.6493 (3) | 0.0163 (7) |
| H4 | 0.9589 | 0.2741 | 0.6142 | 0.020* |
| N1 | 0.9009 (4) | 0.6662 (4) | 0.7494 (3) | 0.0146 (6) |
| C9 | 0.7541 (5) | 0.5716 (5) | 0.7656 (3) | 0.0134 (7) |
| Cl1 | 0.49806 (11) | −0.29443 (10) | 0.53814 (8) | 0.01473 (17) |
| C3 | 1.0864 (5) | 0.4757 (5) | 0.6369 (3) | 0.0172 (8) |
| H3 | 1.1983 | 0.4458 | 0.5942 | 0.021* |
| C2 | 1.0612 (5) | 0.6228 (5) | 0.6887 (3) | 0.0166 (8) |
| H2 | 1.1569 | 0.6925 | 0.6812 | 0.020* |
| H99 | 0.890 (6) | 0.755 (6) | 0.776 (4) | 0.026 (12)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.01797 (12) | 0.01529 (12) | 0.01717 (12) | 0.00269 (9) | −0.00223 (9) | −0.00651 (9) |
| Au2 | 0.01106 (10) | 0.00938 (10) | 0.01044 (10) | −0.00048 (7) | −0.00355 (7) | −0.00150 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Au1 | 0.00681 (9) | 0.01063 (10) | 0.00881 (10) | 0.00010 (7) | −0.00208 (7) | −0.00155 (7) |
| Cl3 | 0.0245 (4) | 0.0160 (4) | 0.0109 (4) | −0.0024 (3) | −0.0030 (3) | −0.0017 (3) |
| Cl2 | 0.0086 (4) | 0.0191 (4) | 0.0211 (4) | 0.0000 (3) | −0.0055 (3) | −0.0036 (3) |
| Cl4 | 0.0233 (4) | 0.0104 (4) | 0.0166 (4) | −0.0010 (3) | −0.0040 (4) | −0.0020 (3) |
| C8 | 0.0163 (17) | 0.0134 (17) | 0.0117 (17) | 0.0026 (14) | −0.0041 (14) | −0.0028 (13) |
| C7 | 0.0180 (18) | 0.0184 (18) | 0.0124 (17) | −0.0002 (14) | −0.0022 (14) | −0.0001 (14) |
| C6 | 0.025 (2) | 0.0210 (19) | 0.0133 (18) | 0.0035 (16) | −0.0067 (15) | 0.0009 (15) |
| C5 | 0.027 (2) | 0.0131 (17) | 0.0139 (18) | −0.0024 (15) | −0.0092 (15) | −0.0016 (14) |
| C10 | 0.0198 (18) | 0.0127 (17) | 0.0077 (16) | 0.0013 (14) | −0.0048 (14) | 0.0014 (13) |
| C4 | 0.0235 (19) | 0.0133 (17) | 0.0130 (17) | 0.0058 (14) | −0.0080 (15) | −0.0021 (14) |
| N1 | 0.0164 (15) | 0.0125 (15) | 0.0153 (15) | 0.0006 (12) | −0.0041 (12) | −0.0024 (12) |
| C9 | 0.0177 (17) | 0.0125 (17) | 0.0098 (16) | 0.0008 (13) | −0.0067 (14) | 0.0027 (13) |
| Cl1 | 0.0150 (4) | 0.0115 (4) | 0.0176 (4) | −0.0005 (3) | −0.0037 (3) | −0.0014 (3) |
| C3 | 0.0138 (17) | 0.0234 (19) | 0.0117 (17) | 0.0055 (15) | −0.0005 (14) | −0.0007 (14) |
| C2 | 0.0159 (18) | 0.0173 (18) | 0.0159 (18) | −0.0029 (14) | −0.0053 (14) | 0.0026 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| I1—C8 | 2.093 (3) | C6—H6 | 0.9300 |
| Au2—Cl3 | 2.2857 (8) | C5—C10 | 1.404 (5) |
| Au2—Cl3$^{i}$ | 2.2857 (8) | C5—H5 | 0.9300 |
| Au2—Cl4$^{i}$ | 2.2894 (8) | C10—C4 | 1.407 (5) |
| Au2—Cl4 | 2.2895 (8) | C10—C9 | 1.429 (5) |
| Au1—Cl1$^{ii}$ | 2.2817 (8) | C4—C3 | 1.381 (5) |
| Au1—Cl1 | 2.2817 (8) | C4—H4 | 0.9300 |
| Au1—Cl2$^{ii}$ | 2.2818 (8) | N1—C2 | 1.331 (5) |
| Au1—Cl2 | 2.2818 (8) | N1—C9 | 1.360 (5) |
| C8—C7 | 1.369 (5) | N1—H99 | 0.80 (4) |
| C8—C9 | 1.418 (5) | C3—C2 | 1.377 (5) |
| C7—C6 | 1.422 (5) | C3—H3 | 0.9300 |
| C7—H7 | 0.9300 | C2—H2 | 0.9300 |
| C6—C5 | 1.371 (5) | | |
| | | | |
| Cl3—Au2—Cl3$^{i}$ | 180.0 | C6—C5—C10 | 121.4 (3) |
| Cl3—Au2—Cl4$^{i}$ | 90.15 (3) | C6—C5—H5 | 119.3 |
| Cl3$^{i}$—Au2—Cl4$^{i}$ | 89.85 (3) | C10—C5—H5 | 119.3 |
| Cl3—Au2—Cl4 | 89.85 (3) | C5—C10—C4 | 123.3 (3) |
| Cl3$^{i}$—Au2—Cl4 | 90.15 (3) | C5—C10—C9 | 118.8 (3) |
| Cl4$^{i}$—Au2—Cl4 | 180.0 | C4—C10—C9 | 117.9 (4) |
| Cl1$^{ii}$—Au1—Cl1 | 180.0 | C3—C4—C10 | 120.9 (3) |
| Cl1$^{ii}$—Au1—Cl2$^{ii}$ | 90.54 (3) | C3—C4—H4 | 119.6 |
| Cl1—Au1—Cl2$^{ii}$ | 89.46 (3) | C10—C4—H4 | 119.6 |
| Cl1$^{ii}$—Au1—Cl2 | 89.46 (3) | C2—N1—C9 | 123.7 (3) |
| Cl1—Au1—Cl2 | 90.54 (3) | C2—N1—H99 | 118 (3) |
| Cl2$^{ii}$—Au1—Cl2 | 180.0 | C9—N1—H99 | 118 (3) |
| C7—C8—C9 | 119.9 (3) | N1—C9—C8 | 122.7 (3) |
| C7—C8—I1 | 120.2 (3) | N1—C9—C10 | 117.9 (3) |
| C9—C8—I1 | 119.8 (3) | C8—C9—C10 | 119.4 (3) |

| | | | |
|---|---|---|---|
| C8—C7—C6 | 121.0 (3) | C2—C3—C4 | 119.0 (3) |
| C8—C7—H7 | 119.5 | C2—C3—H3 | 120.5 |
| C6—C7—H7 | 119.5 | C4—C3—H3 | 120.5 |
| C5—C6—C7 | 119.5 (4) | N1—C2—C3 | 120.6 (3) |
| C5—C6—H6 | 120.3 | N1—C2—H2 | 119.7 |
| C7—C6—H6 | 120.3 | C3—C2—H2 | 119.7 |

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*+1, −*y*, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H99···Cl3$^{iii}$ | 0.80 (5) | 2.62 (5) | 3.287 (3) | 142 (4) |
| N1—H99···I1 | 0.80 (5) | 2.81 (5) | 3.264 (3) | 118 (4) |
| C2—H2···Cl1$^{iv}$ | 0.93 | 2.79 | 3.493 (4) | 133 |
| C3—H3···Cl1$^{v}$ | 0.93 | 2.81 | 3.722 (4) | 168 |

Symmetry codes: (iii) *x*+1, *y*+1, *z*+1; (iv) *x*+1, *y*+1, *z*; (v) −*x*+2, −*y*, −*z*+1.