Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1′-(*p*-Phenylenedimethylene)-dipyridinium trichloridoiodidomercurate(II)

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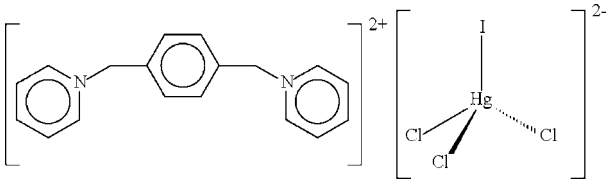
Received 30 May 2007; accepted 5 June 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.013 Å; disorder in main residue; R factor = 0.042; wR factor = 0.113; data-to-parameter ratio = 16.9.

The Hg atom in the title compound, $(C_{18}H_{18}N_2)[HgCl_{2.75}I_{1.25}]$, is coordinated by four halogen atoms in a tetrahedral geometry. Two of the four halogen atoms are each disordered between I and Cl, with the I:Cl ratios being 0.5793 (15):0.4208 (15) and 0.6708 (15):0.3292 (15). The two independent cations lie on different inversion centres.

Related literature

For related literature of tetrahalidomercurates, see: Wang *et al.* (2007). For the synthesis of the organic component, see: Sindelar *et al.* (2004).



Experimental

Crystal data

$(C_{18}H_{18}N_2)[HgCl_{2.75}I_{1.25}]$
M_r = 719.05
Monoclinic, $P2_1/c$
a = 14.2901 (9) Å
b = 9.4764 (6) Å
c = 16.151 (1) Å
β = 98.785 (1)°
V = 2161.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 9.25 mm^{-1}
T = 295 (2) K
0.20 × 0.15 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.089, T_{max} = 0.400
(expected range = 0.073–0.330)
15087 measured reflections
3792 independent reflections
3097 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
S = 1.05
3792 reflections
224 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hg1—Cl1	2.371 (13)	Hg1—Cl4	2.593 (2)
Hg1—Cl2	2.347 (13)	Hg1—I1	2.742 (3)
Hg1—Cl3	2.530 (2)	Hg1—I2	2.755 (2)

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

The authors thank the National Natural Science Foundation of China (grant No. 20671083), the Henan Province Excellent Young Foundation (grant No. 0612002800), Zhengzhou University and the University of Malaya for supporting this work. We thank Central China Normal University for the diffraction measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2390).

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Westrip, S. P. (2007). *publCIF*. In preparation.

  doi:10.1107/S1600536807027481

supplementary materials

Acta Cryst. (2007). E**63**, m1896 [**doi:10.1107/S1600536807027481**]

1,1'-(*p*-Phenylenedimethylene)dipyridinium trichloridoiodidomercurate(II)

Z.-H. He, M.-S. Tang, Y.-Y. Niu, X.-R. Lv and S. W. Ng

Comment

The preceding study reports the structure of a tetrahedral dibromodichloromercurate(II), which was isolated as a 1,2-ethanedipyridinium salt (Wang *et al.*, 2007). Replacing the cation by α,α'-4-xylyldipyridinium furnishes a similar tetrahalomercurate. The anion of the salt is of composed 2.75 chlorines and 1.25 iodines (Fig. 1); the metal atom shows tetrahedral coordination. Selected bond distances are given in Table 1.

Experimental

The salt was synthesized from the reaction of α,α'-4-xylyldipyridinium dichloride (0.033 g, 0.1 mmol) in methanol (5 ml) and mercuric iodide (0.091 g, 0.2 mmol) in DMF (10 ml). The mixture was set aside for the formation of colorless crystals in 30% yield after several days. The organic reactant was synthesized by using a literature method (Sindelar *et al.*, 2004).

Refinement

The four halogens lie in general positions. Initial attempts to refine the structure with either four iodines or four chlorines gave unacceptably high *R*-indices and large peaks/holes. The four halogen atoms were then refined as four (I+Cl) mixtures, with same displacement parameters. This led to a formulation consisting of approximately 1.25 I and 2.75 C l atoms. The use of a restraint that fixed the number of I and Cl atoms as exactly 1.25 I and 2.75 C l led to the occupancy of I1 as 0.6 and that of I2 as 0.7; the occupancies of I3 and I4 were nearly zero. As such, the Cl3 and Cl4 atoms were each assigned full occupancy, so that only the I1/Cl1 and I2/Cl2 halogen atoms were disordered.

The anion is $[HgCl_{2.75}I_{1.25}]^{2-}$, but because it has nearly integral numbers of chlorine and iodine atoms, it is regarded as $[HgCl_3I]$ for the purpose of naming the compound. The formulation is, however, in poor agreement with CH&N elemental analysis, so that the synthesis probably yielded a range of tetrahalogenmercurates. Other formulations led to somewhat larger peaks/deep holes.

The disorder affected the cation; the pyridyl ring was refined as a rigid hexagon (C—C = C—N = 1.39 Å). C-bound H atoms were generated geometrically (C–H 0.93 Å), and were included in the refinement in the riding-model approximation, with U(H) set to 1.2U_{eq}(C).

The final difference Fourier map had a large peak at 0.91 Å from Hg1, but was otherwise featureless.

Figures

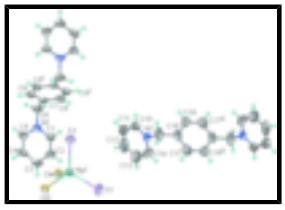


Fig. 1. The molecular structure of $[C_{18}H_{18}N_2][HgCl_{2.75}I_{1.25}]$. Displacement ellipsoids are drawn at the 50% probability level. Labels X1 and X2 denote the disordered I and Cl atoms (I1, I2, Cl1 and Cl2). Hydrogen atoms are drawn as spheres of arbitrary radius. [Symmetry code (i): $1-x, 1-y, 2-z$; (ii) $2-x, -1-y, 2-z$.]

1,1'-(*p*-Phenylenedimethylene)dipyridinium trichloridoiodidomercurate(II)

Crystal data

$(C_{18}H_{18}N_2)[HgCl_{2.75}I_{1.25}]$	F_{000} = 1332
M_r = 719.05	D_x = 2.210 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4098 reflections
a = 14.2901 (9) Å	θ = 2.5–22.8º
b = 9.4764 (6) Å	μ = 9.25 mm^{-1}
c = 16.151 (1) Å	T = 295 (2) K
β = 98.785 (1)º	Block, colourless
V = 2161.5 (2) Å^3	0.20 × 0.15 × 0.12 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	3792 independent reflections
Radiation source: fine-focus sealed tube	3097 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 295(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −16→16
T_{min} = 0.089, T_{max} = 0.400	k = −11→11
15087 measured reflections	l = −19→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H-atom parameters constrained
$wR(F^2)$ = 0.113	$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 2.8764P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001

3792 reflections	$\Delta\rho_{max}$ = 1.10 e Å^{-3}
224 parameters	$\Delta\rho_{min}$ = −0.57 e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Hg1	0.74880 (2)	0.42222 (4)	0.68910 (2)	0.06253 (16)	
I1	0.8501 (3)	0.1938 (3)	0.6478 (3)	0.0731 (6)	0.5793 (15)
Cl1	0.8430 (17)	0.2326 (13)	0.6529 (17)	0.077 (4)	0.4208 (15)
I2	0.6700 (2)	0.3885 (4)	0.83326 (16)	0.0613 (5)	0.6708 (15)
Cl2	0.6851 (19)	0.386 (3)	0.8131 (13)	0.058 (4)	0.3292 (15)
Cl3	0.62488 (15)	0.4902 (2)	0.56647 (12)	0.0617 (5)	
Cl4	0.84520 (15)	0.6549 (2)	0.69693 (14)	0.0646 (5)	
N1	0.3671 (4)	0.3819 (5)	0.7986 (2)	0.0523 (16)	
C1	0.4004 (4)	0.2569 (4)	0.7687 (3)	0.063 (2)	
H1	0.4048	0.1761	0.8017	0.075*	
C2	0.4271 (4)	0.2529 (5)	0.6895 (3)	0.071 (2)	
H2	0.4494	0.1693	0.6695	0.085*	
C3	0.4205 (5)	0.3738 (6)	0.6402 (2)	0.071 (3)	
H3	0.4384	0.3711	0.5873	0.085*	
C4	0.3871 (5)	0.4987 (5)	0.6701 (3)	0.078 (3)	
H4	0.3827	0.5796	0.6372	0.094*	
C5	0.3604 (4)	0.5028 (4)	0.7493 (3)	0.069 (2)	
H5	0.3381	0.5863	0.7693	0.082*	
C6	0.3432 (7)	0.3870 (9)	0.8825 (5)	0.065 (2)	
H6A	0.3274	0.2928	0.8994	0.078*	
H6B	0.2879	0.4464	0.8827	0.078*	
C7	0.4251 (6)	0.4449 (8)	0.9450 (4)	0.054 (2)	
C8	0.4980 (7)	0.3591 (8)	0.9794 (5)	0.060 (2)	
H8	0.4966	0.2638	0.9655	0.072*	
C9	0.4264 (7)	0.5878 (9)	0.9654 (5)	0.063 (2)	
H9	0.3772	0.6468	0.9424	0.076*	
N2	0.8607 (4)	−0.1805 (4)	0.9319 (4)	0.0560 (16)	
C10	0.8258 (5)	−0.0683 (7)	0.9734 (4)	0.100 (4)	
H10	0.7900	−0.0854	1.0159	0.120*	
C11	0.8443 (7)	0.0696 (5)	0.9515 (5)	0.113 (5)	
H11	0.8209	0.1447	0.9792	0.136*	
C12	0.8977 (6)	0.0953 (5)	0.8880 (6)	0.104 (4)	
H12	0.9101	0.1875	0.8733	0.125*	
C13	0.9327 (6)	−0.0170 (7)	0.8465 (5)	0.119 (5)	
H13	0.9684	0.0002	0.8040	0.143*	
C14	0.9142 (5)	−0.1549 (6)	0.8685 (4)	0.093 (3)	
H14	0.9375	−0.2300	0.8407	0.111*	
C15	0.8358 (7)	−0.3242 (10)	0.9509 (8)	0.092 (3)	
H15A	0.7977	−0.3652	0.9019	0.110*	

H15B	0.7980	−0.3230	0.9959	0.110*
C16	0.9223 (7)	−0.4144 (8)	0.9763 (7)	0.067 (3)
C17	0.9533 (8)	−0.5048 (10)	0.9197 (7)	0.078 (3)
H17	0.9219	−0.5087	0.8649	0.094*
C18	0.9696 (8)	−0.4109 (9)	1.0562 (7)	0.074 (3)
H18	0.9491	−0.3501	1.0949	0.089*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.0675 (3)	0.0560 (2)	0.0639 (2)	0.00375 (15)	0.00949 (17)	0.00970 (15)
I1	0.0721 (11)	0.0430 (16)	0.1092 (11)	0.0162 (12)	0.0297 (8)	0.0095 (15)
Cl1	0.092 (6)	0.017 (5)	0.129 (8)	0.030 (4)	0.041 (5)	0.017 (5)
I2	0.0767 (13)	0.0561 (7)	0.0568 (13)	−0.0089 (8)	0.0283 (9)	0.0031 (9)
Cl2	0.087 (9)	0.049 (5)	0.050 (8)	−0.001 (5)	0.050 (5)	0.002 (5)
Cl3	0.0677 (13)	0.0627 (12)	0.0528 (11)	0.0040 (10)	0.0031 (9)	0.0056 (9)
Cl4	0.0588 (13)	0.0624 (12)	0.0726 (13)	−0.0014 (10)	0.0106 (10)	0.0147 (10)
N1	0.070 (5)	0.047 (3)	0.043 (3)	−0.010 (3)	0.017 (3)	−0.007 (3)
C1	0.074 (6)	0.067 (5)	0.049 (4)	−0.007 (4)	0.013 (4)	0.002 (4)
C2	0.079 (6)	0.071 (6)	0.066 (6)	−0.002 (5)	0.024 (5)	−0.007 (5)
C3	0.083 (7)	0.081 (6)	0.050 (5)	−0.019 (5)	0.013 (5)	0.002 (5)
C4	0.118 (8)	0.073 (6)	0.044 (5)	−0.004 (6)	0.011 (5)	0.012 (4)
C5	0.091 (7)	0.061 (5)	0.053 (5)	0.003 (5)	0.008 (4)	−0.002 (4)
C6	0.085 (6)	0.065 (5)	0.049 (5)	−0.020 (5)	0.025 (4)	−0.006 (4)
C7	0.078 (6)	0.049 (4)	0.036 (4)	−0.014 (4)	0.018 (4)	−0.005 (3)
C8	0.098 (7)	0.041 (4)	0.044 (4)	−0.008 (5)	0.019 (4)	0.003 (3)
C9	0.083 (7)	0.056 (5)	0.053 (5)	0.004 (4)	0.015 (5)	0.002 (4)
N2	0.052 (4)	0.043 (4)	0.072 (4)	0.003 (3)	0.008 (3)	0.006 (3)
C10	0.133 (11)	0.102 (9)	0.075 (7)	0.034 (7)	0.043 (7)	0.014 (6)
C11	0.185 (15)	0.066 (7)	0.088 (8)	0.033 (8)	0.021 (9)	−0.011 (6)
C12	0.099 (9)	0.046 (6)	0.160 (13)	0.006 (5)	−0.004 (8)	0.011 (7)
C13	0.138 (12)	0.088 (8)	0.148 (12)	0.012 (8)	0.073 (10)	0.049 (8)
C14	0.124 (9)	0.058 (6)	0.109 (8)	0.010 (6)	0.057 (7)	−0.001 (6)
C15	0.068 (6)	0.060 (6)	0.150 (10)	0.003 (5)	0.022 (6)	0.031 (6)
C16	0.063 (6)	0.038 (4)	0.098 (7)	−0.013 (4)	0.010 (5)	0.018 (4)
C17	0.092 (8)	0.056 (6)	0.084 (7)	−0.008 (5)	0.006 (6)	0.008 (5)
C18	0.087 (7)	0.051 (5)	0.088 (7)	0.006 (5)	0.025 (6)	0.007 (5)

Geometric parameters (Å, °)

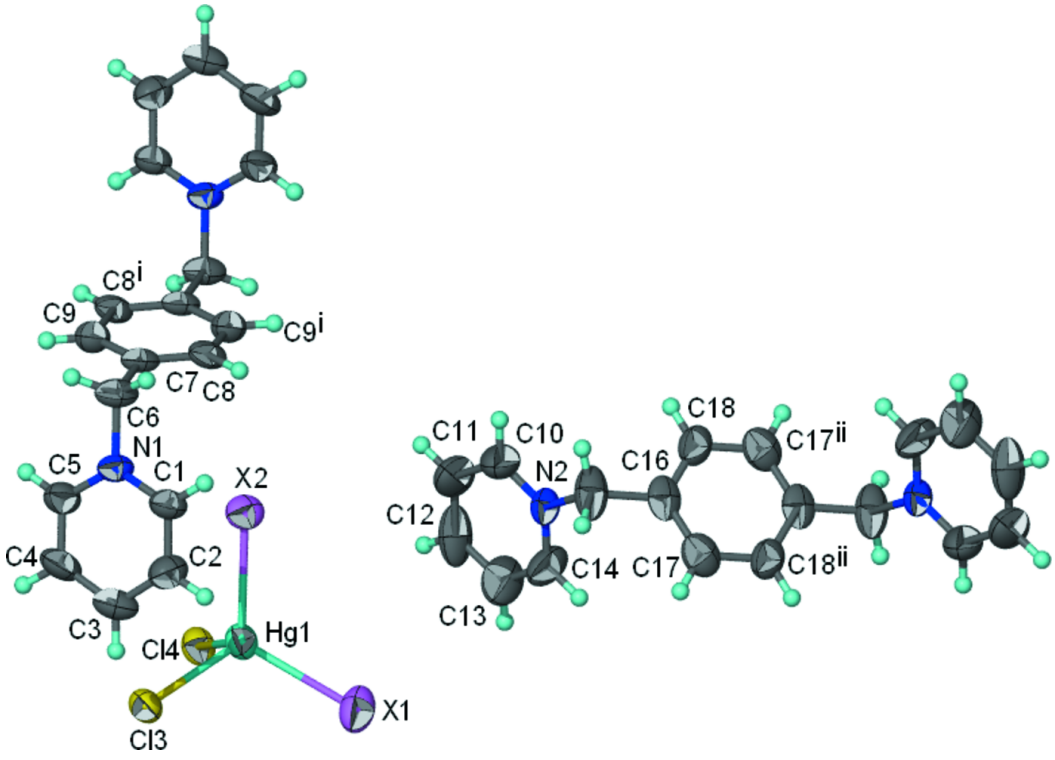
Hg1—Cl1	2.371 (13)	C8—H8	0.93
Hg1—Cl2	2.347 (13)	C9—C8^{i}	1.386 (13)
Hg1—Cl3	2.530 (2)	C9—H9	0.93
Hg1—Cl4	2.593 (2)	N2—C10	1.39
Hg1—I1	2.742 (3)	N2—C14	1.39
Hg1—I2	2.755 (2)	N2—C15	1.452 (10)
N1—C1	1.39	C10—C11	1.39
N1—C5	1.39	C10—H10	0.93

N1—C6	1.447 (8)	C11—C12	1.39
C1—C2	1.39	C11—H11	0.93
C1—H1	0.93	C12—C13	1.39
C2—C3	1.39	C12—H12	0.93
C2—H2	0.93	C13—C14	1.39
C3—C4	1.39	C13—H13	0.93
C3—H3	0.93	C14—H14	0.93
C4—C5	1.39	C15—C16	1.508 (13)
C4—H4	0.93	C15—H15A	0.97
C5—H5	0.93	C15—H15B	0.97
C6—C7	1.527 (12)	C16—C18	1.361 (15)
C6—H6A	0.97	C16—C17	1.375 (14)
C6—H6B	0.97	C17—C18^{ii}	1.369 (14)
C7—C8	1.371 (12)	C17—H17	0.93
C7—C9	1.393 (10)	C18—C17^{ii}	1.369 (14)
C8—C9^{i}	1.386 (13)	C18—H18	0.93
Cl2—Hg1—Cl1	114.2 (9)	C9—C7—C6	119.2 (8)
Cl2—Hg1—Cl3	112.9 (7)	C7—C8—C9^{i}	121.0 (8)
Cl1—Hg1—Cl3	110.8 (7)	C7—C8—H8	119.5
Cl2—Hg1—Cl4	110.7 (7)	C9^{i}—C8—H8	119.5
Cl1—Hg1—Cl4	109.8 (6)	C8^{i}—C9—C7	119.1 (8)
Cl3—Hg1—Cl4	97.16 (7)	C8^{i}—C9—H9	120.5
Cl2—Hg1—I1	112.4 (7)	C7—C9—H9	120.5
Cl1—Hg1—I1	2.9 (6)	C10—N2—C14	120.0
Cl3—Hg1—I1	110.05 (11)	C10—N2—C15	119.9 (6)
Cl4—Hg1—I1	112.76 (10)	C14—N2—C15	120.0 (6)
Cl2—Hg1—I2	2.0 (8)	N2—C10—C11	120.0
Cl1—Hg1—I2	116.2 (6)	N2—C10—H10	120.0
Cl3—Hg1—I2	111.32 (9)	C11—C10—H10	120.0
Cl4—Hg1—I2	109.89 (9)	C12—C11—C10	120.0
I1—Hg1—I2	114.39 (12)	C12—C11—H11	120.0
C1—N1—C5	120.0	C10—C11—H11	120.0
C1—N1—C6	119.7 (5)	C11—C12—C13	120.0
C5—N1—C6	120.2 (5)	C11—C12—H12	120.0
C2—C1—N1	120.0	C13—C12—H12	120.0
C2—C1—H1	120.0	C14—C13—C12	120.0
N1—C1—H1	120.0	C14—C13—H13	120.0
C1—C2—C3	120.0	C12—C13—H13	120.0
C1—C2—H2	120.0	C13—C14—N2	120.0
C3—C2—H2	120.0	C13—C14—H14	120.0
C4—C3—C2	120.0	N2—C14—H14	120.0
C4—C3—H3	120.0	N2—C15—C16	111.8 (7)
C2—C3—H3	120.0	N2—C15—H15A	109.3
C5—C4—C3	120.0	C16—C15—H15A	109.3
C5—C4—H4	120.0	N2—C15—H15B	109.3
C3—C4—H4	120.0	C16—C15—H15B	109.3
C4—C5—N1	120.0	H15A—C15—H15B	107.9

C4—C5—H5	120.0	C18—C16—C17	118.9 (9)
N1—C5—H5	120.0	C18—C16—C15	120.8 (10)
N1—C6—C7	111.4 (7)	C17—C16—C15	120.4 (10)
N1—C6—H6A	109.4	C18^{ii}—C17—C16	120.0 (10)
C7—C6—H6A	109.4	C18^{ii}—C17—H17	120.0
N1—C6—H6B	109.4	C16—C17—H17	120.0
C7—C6—H6B	109.4	C16—C18—C17^{ii}	121.1 (10)
H6A—C6—H6B	108.0	C16—C18—H18	119.4
C8—C7—C9	119.9 (8)	C17^{ii}—C18—H18	119.4
C8—C7—C6	120.9 (7)		
C6—N1—C1—C2	−177.4 (6)	C15—N2—C10—C11	−175.7 (7)
C6—N1—C5—C4	177.4 (6)	C15—N2—C14—C13	175.6 (7)
C1—N1—C6—C7	96.1 (7)	C10—N2—C15—C16	−124.6 (8)
C5—N1—C6—C7	−81.2 (8)	C14—N2—C15—C16	59.7 (12)
N1—C6—C7—C8	−82.8 (9)	N2—C15—C16—C18	81.6 (12)
N1—C6—C7—C9	95.7 (9)	N2—C15—C16—C17	−100.2 (11)
C9—C7—C8—C9^{i}	−0.1 (13)	C18—C16—C17—C18^{ii}	0.0 (15)
C6—C7—C8—C9^{i}	178.4 (7)	C15—C16—C17—C18^{ii}	−178.3 (8)
C8—C7—C9—C8^{i}	0.1 (13)	C17—C16—C18—C17^{ii}	0.0 (15)
C6—C7—C9—C8^{i}	−178.4 (7)	C15—C16—C18—C17^{ii}	178.3 (8)

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

Fig. 1


C8i
C9
C9i
C7
C8
C6
N1
C5
C1
X2
C4
C2
C3
Cl4
Hg1
Cl3
X1
C11
C10
C18
N2
C16
C17ii
C12
C14
C17
C18ii
C13