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Bis(2-amino-3-methylpyridine-κN^1)-dichloridomercury(II)

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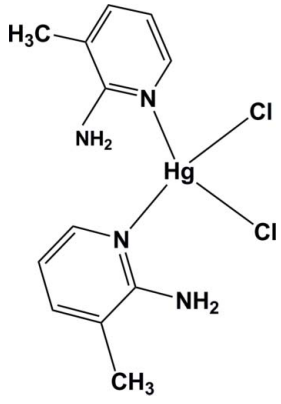
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.013 Å; R factor = 0.047; wR factor = 0.116; data-to-parameter ratio = 15.5.

In the title compound, $[HgCl_2(C_6H_8N_2)_2]$, the two independent Hg^{II} cations are each located on a twofold rotation axis and coordinated by two pyridine N atoms from two 2-amino-3-methylpyridine ligands and two Cl^- anions in a distorted tetrahedral geometry. An intramolecular N—H···Cl hydrogen bond occurs in each independent complex molecule. Intermolecular N—H···Cl hydrogen bonds occur in the crystal structure.

Related literature

For coordination modes of 2-amino-3-methylpyridine (ampy), see: Arab Ahmadi *et al.* (2011); Tadjarodi *et al.* (2010); Amani Komaei *et al.* (1999); Ziegler *et al.* (2000); Castillo *et al.* (2001); Chen *et al.* (2005). For proton-transfer compounds incorporating ampy, see: Carnevale *et al.* (2010).



Experimental

Crystal data

$[HgCl_2(C_6H_8N_2)_2]$
M_r = 487.78
Monoclinic, $P2_1/c$
a = 16.495 (3) Å
b = 6.6320 (13) Å
c = 16.273 (3) Å
β = 119.56 (3)°
V = 1548.5 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 10.28 mm^{-1}
T = 298 K
0.30 × 0.30 × 0.27 mm

Data collection

Stoe IPDS 2T diffractometer
Absorption correction: numerical [shape of crystal determined optically (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)]
T_{min} = 0.149, T_{max} = 0.168
5454 measured reflections
2912 independent reflections
2493 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.116
S = 1.03
2912 reflections
188 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 2.40 e Å^{-3}
$\Delta\rho_{min}$ = −2.67 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl1	0.86 (2)	2.58 (4)	3.420 (9)	164 (10)
N2—H2B···Cl2^{i}	0.87 (2)	2.72 (7)	3.420 (8)	139 (8)
N4—H4A···Cl1^{ii}	0.86 (2)	2.70 (7)	3.409 (8)	140 (9)
N4—H4B···Cl2	0.87 (2)	2.59 (4)	3.424 (9)	162 (9)

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); 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: XU5594).

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supplementary materials

Acta Cryst. (2012). E**68**, m1099 [doi:10.1107/S1600536812032126]

Bis(2-amino-3-methylpyridine-κN^1)dichloridomercury(II)

Azadeh Tadjarodi, Keyvan Bijanzad and Behrouz Notash

Comment

2-Amino-3-methylpyridine (ampy) is capable of coordinating to metals not only through the nitrogen atom of the pyridyl group (Arab Ahmadi *et al.*, 2011; Tadjarodi *et al.*, 2010; Amani Komaei *et al.*, 1999; Ziegler *et al.*, 2000; Castillo *et al.*, 2001) but also *via* the nitrogen atom of the amino group (Chen *et al.*, 2005). So far, different structures of proton-transfer compounds, $[(ampyH)_2CoX_4]$ (X = Cl, Br) have been reported (Carnevale *et al.* (2010).

We report herein the synthesis and molecular structure of the title compound, $[Hg(ampy)_2Cl_2]$. The asymmetric unit of the title compound consists of two half of one mercury, one ampy and one chloride atom. The coordination sphere of the mononuclear complex consists of two chloride ions and two pyridyl nitrogen atoms from two ampy ligands in a distorted tetrahedral geometry (Fig. 1). In the crystal structure of $[Hg(ampy)_2Cl_2]$, there are several intermolecular N–H···Cl hydrogen bond interactions which stabilized crystal structure (Fig. 2 & Table 1).

Experimental

An ethanolic solution of 2-amino-3-methylpyridine (10 mmol) was added to a solution of $HgCl_2$ (5 mmol) in ethanol (10 ml) and stirred for 10 min at 50°C. Slow evaporation of the resulting filtrate gave the colorless crystals suitable for X-ray analysis (decomposition > 240 °C).

Refinement

Hydrogen atoms attached to nitrogen atoms were found in difference Fourier map. H2A, H2B, H4A and H4B were refined with Uiso(H) = 1.5 Ueq(N). H2A, H2B, H4A and H4B were refined with distance restraints of N—H 0.86 (2), 0.87 (2), 0.86 (2) and 0.87 (2), respectively. H atoms attached to C atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å (CH), with C—H = 0.96 Å (CH_3), and U_{iso}(H) = 1.2,1.5U_{eq}(C).

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA* (Stoe & Cie, 2005); data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); 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).

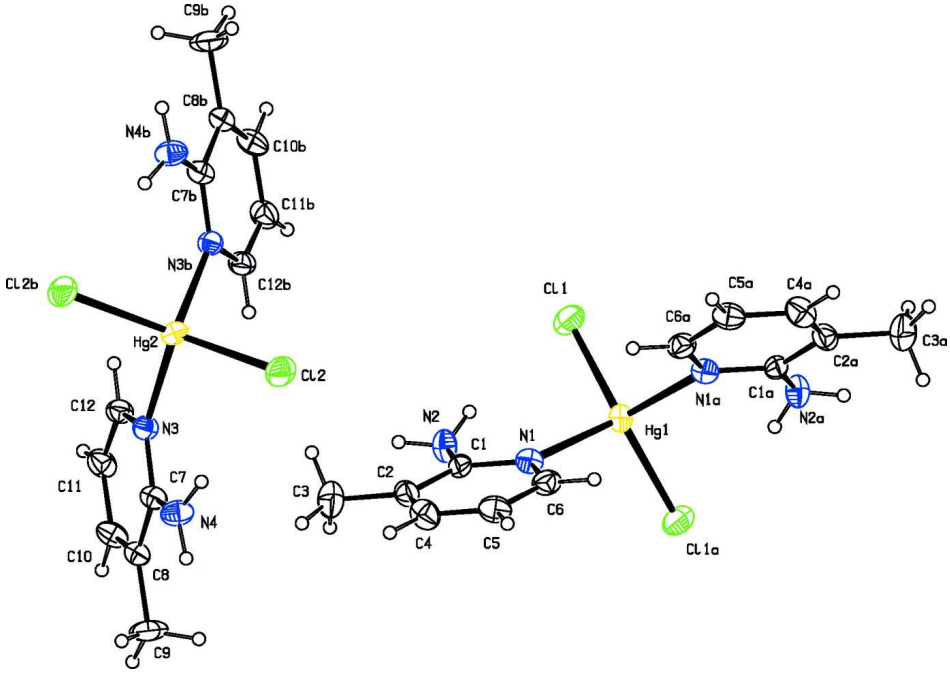

Figure 1

The molecular structure of $[Hg(ampy)_2Cl_2]$ with displacement ellipsoids drawn at 30% probability level.

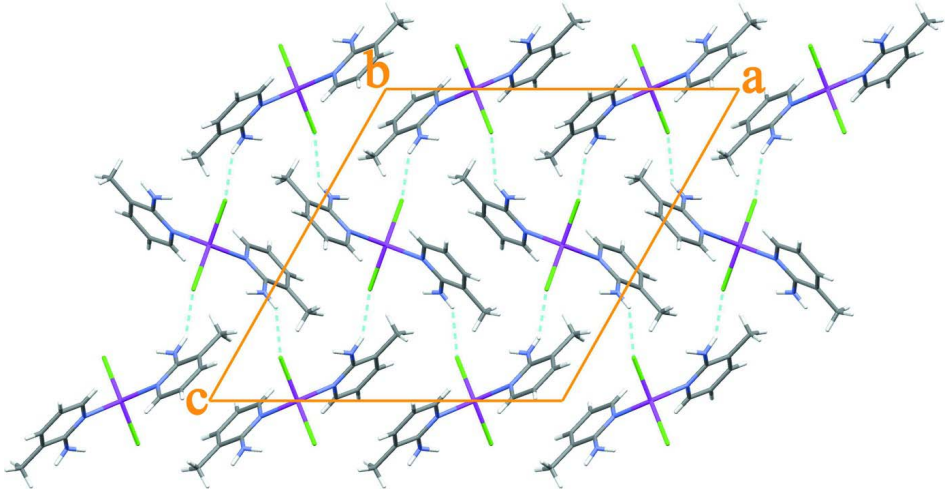

Figure 2

The packing diagram of the title compound showing hydrogen bonding as blue dashed lines.

Bis(2-amino-3-methylpyridine-κN^1)dichloridomercury(II)

Crystal data

$[HgCl_2(C_6H_8N_2)_2]$
$M_r = 487.78$
Monoclinic, $P2/c$
Hall symbol: -P 2yc
$a = 16.495$ (3) Å
$b = 6.6320$ (13) Å
$c = 16.273$ (3) Å
$\beta = 119.56$ (3)°
$V = 1548.5$ (7) Å^3
$Z = 4$
$F(000) = 920$
$D_x = 2.092$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2912 reflections

θ = 2.5–26.0°
μ = 10.28 mm^{-1}
T = 298 K

Block, colorless
0.30 × 0.30 × 0.27 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
rotation method scans
Absorption correction: numerical [shape of crystal determined optically (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)]
T_{min} = 0.149, T_{max} = 0.168

5454 measured reflections
2912 independent reflections
2493 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 26.0°, θ_{min} = 2.5°
h = −16→20
k = −7→8
l = −19→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.116
S = 1.03
2912 reflections
188 parameters
4 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 2.40 e Å^{-3}
$\Delta\rho_{min}$ = −2.67 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0064 (5)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Hg1	0.0000	0.19701 (7)	0.2500	0.04474 (19)
Hg2	0.5000	0.76184 (6)	0.2500	0.04007 (19)
Cl1	0.13755 (15)	0.0118 (3)	0.36667 (16)	0.0565 (6)
Cl2	0.35836 (16)	0.9352 (4)	0.22391 (16)	0.0590 (6)
N1	0.0500 (5)	0.4038 (9)	0.1715 (4)	0.0407 (15)
N2	0.1527 (6)	0.1618 (12)	0.1736 (6)	0.0497 (17)
N3	0.4556 (4)	0.5485 (9)	0.1232 (4)	0.0359 (14)
N4	0.3477 (6)	0.7778 (10)	0.0184 (5)	0.0472 (19)
C1	0.1163 (5)	0.3496 (11)	0.1495 (5)	0.0362 (16)
C2	0.1430 (6)	0.4814 (13)	0.1001 (5)	0.0441 (19)
C3	0.2151 (7)	0.4171 (18)	0.0751 (8)	0.069 (3)

H3A	0.2217	0.5192	0.0370	0.104*
H3B	0.1962	0.2928	0.0404	0.104*
H3C	0.2737	0.3980	0.1318	0.104*
C4	0.1028 (7)	0.6659 (13)	0.0756 (6)	0.051 (2)
H4	0.1206	0.7549	0.0432	0.061*
C5	0.0361 (7)	0.7229 (12)	0.0981 (6)	0.049 (2)
H5	0.0088	0.8499	0.0813	0.059*
C6	0.0103 (6)	0.5893 (13)	0.1459 (5)	0.0454 (19)
H6	−0.0352	0.6267	0.1609	0.055*
C7	0.3884 (6)	0.5942 (11)	0.0346 (5)	0.0373 (16)
C8	0.3618 (6)	0.4525 (12)	−0.0402 (5)	0.0403 (17)
C9	0.2853 (7)	0.5040 (17)	−0.1374 (6)	0.066 (3)
H9A	0.2783	0.3968	−0.1801	0.100*
H9B	0.3005	0.6267	−0.1581	0.100*
H9C	0.2280	0.5215	−0.1364	0.100*
C10	0.4071 (8)	0.2741 (12)	−0.0193 (6)	0.050 (2)
H10	0.3904	0.1785	−0.0668	0.060*
C11	0.4775 (7)	0.2304 (13)	0.0704 (6)	0.049 (2)
H11	0.5096	0.1087	0.0833	0.058*
C12	0.4993 (6)	0.3695 (12)	0.1401 (5)	0.0405 (17)
H12	0.5458	0.3394	0.2012	0.049*
H2A	0.158 (7)	0.107 (14)	0.224 (4)	0.061*
H4A	0.301 (5)	0.788 (14)	−0.038 (4)	0.061*
H2B	0.202 (4)	0.161 (15)	0.168 (7)	0.061*
H4B	0.348 (7)	0.845 (14)	0.064 (5)	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.0379 (3)	0.0561 (3)	0.0446 (3)	0.000	0.0238 (2)	0.000
Hg2	0.0326 (3)	0.0476 (3)	0.0330 (3)	0.000	0.0108 (2)	0.000
Cl1	0.0393 (11)	0.0617 (13)	0.0566 (12)	−0.0032 (9)	0.0147 (10)	0.0151 (9)
Cl2	0.0442 (11)	0.0714 (14)	0.0564 (12)	0.0166 (10)	0.0210 (10)	−0.0068 (10)
N1	0.043 (4)	0.041 (4)	0.036 (3)	0.001 (3)	0.018 (3)	0.001 (2)
N2	0.048 (4)	0.051 (4)	0.063 (5)	0.006 (3)	0.037 (4)	0.001 (3)
N3	0.036 (3)	0.036 (3)	0.034 (3)	0.007 (3)	0.016 (3)	0.001 (2)
N4	0.049 (5)	0.039 (4)	0.040 (4)	0.008 (3)	0.012 (3)	0.003 (3)
C1	0.029 (4)	0.046 (4)	0.033 (3)	−0.007 (3)	0.014 (3)	−0.009 (3)
C2	0.045 (5)	0.054 (5)	0.039 (4)	−0.015 (4)	0.025 (4)	−0.009 (3)
C3	0.055 (6)	0.085 (8)	0.081 (7)	−0.007 (5)	0.044 (6)	0.003 (5)
C4	0.065 (6)	0.043 (4)	0.052 (5)	−0.015 (4)	0.034 (4)	−0.003 (4)
C5	0.058 (6)	0.042 (5)	0.042 (4)	0.002 (4)	0.020 (4)	0.002 (3)
C6	0.040 (4)	0.049 (5)	0.039 (4)	0.009 (4)	0.014 (4)	0.001 (3)
C7	0.045 (4)	0.037 (4)	0.030 (3)	−0.006 (3)	0.018 (3)	−0.001 (3)
C8	0.042 (4)	0.049 (4)	0.031 (4)	0.002 (3)	0.019 (3)	0.002 (3)
C9	0.054 (6)	0.087 (7)	0.029 (4)	0.010 (5)	−0.002 (4)	−0.007 (4)
C10	0.066 (6)	0.050 (5)	0.036 (4)	−0.001 (4)	0.027 (4)	−0.009 (3)
C11	0.058 (6)	0.049 (5)	0.045 (5)	0.012 (4)	0.030 (5)	0.004 (3)
C12	0.042 (4)	0.044 (4)	0.032 (3)	0.005 (3)	0.016 (3)	0.004 (3)

Geometric parameters (Å, º)

Hg1—N1	2.287 (7)	C2—C3	1.497 (14)
Hg1—N1^{i}	2.287 (7)	C3—H3A	0.9600
Hg1—Cl1^{i}	2.452 (2)	C3—H3B	0.9600
Hg1—Cl1	2.452 (2)	C3—H3C	0.9600
Hg2—N3	2.303 (6)	C4—C5	1.373 (15)
Hg2—N3^{ii}	2.303 (6)	C4—H4	0.9300
Hg2—Cl2^{ii}	2.446 (2)	C5—C6	1.378 (13)
Hg2—Cl2	2.446 (2)	C5—H5	0.9300
N1—C1	1.356 (11)	C6—H6	0.9300
N1—C6	1.360 (10)	C7—C8	1.425 (10)
N2—C1	1.354 (11)	C8—C10	1.351 (12)
N2—H2A	0.86 (2)	C8—C9	1.498 (11)
N2—H2B	0.87 (2)	C9—H9A	0.9600
N3—C12	1.345 (10)	C9—H9B	0.9600
N3—C7	1.350 (9)	C9—H9C	0.9600
N4—C7	1.352 (10)	C10—C11	1.375 (13)
N4—H4A	0.86 (2)	C10—H10	0.9300
N4—H4B	0.87 (2)	C11—C12	1.365 (12)
C1—C2	1.397 (11)	C11—H11	0.9300
C2—C4	1.355 (13)	C12—H12	0.9300
N1—Hg1—N1^{i}	106.3 (3)	C2—C3—H3C	109.5
N1—Hg1—Cl1^{i}	108.60 (16)	H3A—C3—H3C	109.5
N1^{i}—Hg1—Cl1^{i}	106.35 (17)	H3B—C3—H3C	109.5
N1—Hg1—Cl1	106.35 (17)	C2—C4—C5	120.7 (8)
N1^{i}—Hg1—Cl1	108.60 (16)	C2—C4—H4	119.6
Cl1^{i}—Hg1—Cl1	119.88 (11)	C5—C4—H4	119.6
N3—Hg2—N3^{ii}	104.2 (3)	C4—C5—C6	119.0 (8)
N3—Hg2—Cl2^{ii}	107.38 (18)	C4—C5—H5	120.5
N3^{ii}—Hg2—Cl2^{ii}	106.20 (16)	C6—C5—H5	120.5
N3—Hg2—Cl2	106.20 (16)	N1—C6—C5	121.3 (9)
N3^{ii}—Hg2—Cl2	107.38 (18)	N1—C6—H6	119.4
Cl2^{ii}—Hg2—Cl2	123.91 (13)	C5—C6—H6	119.4
C1—N1—C6	119.2 (7)	N3—C7—N4	118.5 (6)
C1—N1—Hg1	123.3 (5)	N3—C7—C8	120.6 (7)
C6—N1—Hg1	117.5 (6)	N4—C7—C8	120.9 (7)
C1—N2—H2A	119 (7)	C10—C8—C7	117.7 (7)
C1—N2—H2B	106 (7)	C10—C8—C9	122.6 (8)
H2A—N2—H2B	116 (10)	C7—C8—C9	119.7 (7)
C12—N3—C7	119.4 (6)	C8—C9—H9A	109.5
C12—N3—Hg2	117.3 (5)	C8—C9—H9B	109.5
C7—N3—Hg2	123.3 (5)	H9A—C9—H9B	109.5
C7—N4—H4A	112 (7)	C8—C9—H9C	109.5
C7—N4—H4B	120 (7)	H9A—C9—H9C	109.5
H4A—N4—H4B	120 (10)	H9B—C9—H9C	109.5
N2—C1—N1	117.8 (7)	C8—C10—C11	121.6 (8)
N2—C1—C2	121.4 (7)	C8—C10—H10	119.2
N1—C1—C2	120.7 (7)	C11—C10—H10	119.2

C4—C2—C1	119.1 (8)	C12—C11—C10	118.6 (8)
C4—C2—C3	121.1 (8)	C12—C11—H11	120.7
C1—C2—C3	119.8 (8)	C10—C11—H11	120.7
C2—C3—H3A	109.5	N3—C12—C11	122.1 (7)
C2—C3—H3B	109.5	N3—C12—H12	118.9
H3A—C3—H3B	109.5	C11—C12—H12	118.9
N1^{i}—Hg1—N1—C1	152.6 (6)	C1—C2—C4—C5	−0.5 (12)
Cl1^{i}—Hg1—N1—C1	−93.3 (5)	C3—C2—C4—C5	179.4 (8)
Cl1—Hg1—N1—C1	37.0 (6)	C2—C4—C5—C6	−0.2 (13)
N1^{i}—Hg1—N1—C6	−27.8 (5)	C1—N1—C6—C5	−0.1 (11)
Cl1^{i}—Hg1—N1—C6	86.3 (5)	Hg1—N1—C6—C5	−179.8 (6)
Cl1—Hg1—N1—C6	−143.4 (5)	C4—C5—C6—N1	0.5 (13)
N3^{ii}—Hg2—N3—C12	−31.0 (5)	C12—N3—C7—N4	−177.6 (8)
Cl2^{ii}—Hg2—N3—C12	81.4 (6)	Hg2—N3—C7—N4	2.9 (10)
Cl2—Hg2—N3—C12	−144.2 (6)	C12—N3—C7—C8	1.8 (12)
N3^{ii}—Hg2—N3—C7	148.5 (7)	Hg2—N3—C7—C8	−177.7 (6)
Cl2^{ii}—Hg2—N3—C7	−99.1 (6)	N3—C7—C8—C10	−1.2 (13)
Cl2—Hg2—N3—C7	35.3 (6)	N4—C7—C8—C10	178.3 (9)
C6—N1—C1—N2	−178.2 (7)	N3—C7—C8—C9	178.3 (8)
Hg1—N1—C1—N2	1.4 (9)	N4—C7—C8—C9	−2.3 (13)
C6—N1—C1—C2	−0.6 (10)	C7—C8—C10—C11	−0.9 (15)
Hg1—N1—C1—C2	179.0 (5)	C9—C8—C10—C11	179.7 (10)
N2—C1—C2—C4	178.4 (8)	C8—C10—C11—C12	2.1 (16)
N1—C1—C2—C4	0.9 (11)	C7—N3—C12—C11	−0.5 (13)
N2—C1—C2—C3	−1.5 (11)	Hg2—N3—C12—C11	179.0 (7)
N1—C1—C2—C3	−179.0 (7)	C10—C11—C12—N3	−1.5 (15)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl1	0.86 (2)	2.58 (4)	3.420 (9)	164 (10)
N2—H2*B*···Cl2^{iii}	0.87 (2)	2.72 (7)	3.420 (8)	139 (8)
N4—H4*A*···Cl1^{iv}	0.86 (2)	2.70 (7)	3.409 (8)	140 (9)
N4—H4*B*···Cl2	0.87 (2)	2.59 (4)	3.424 (9)	162 (9)

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