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Di-μ-chlorido-bis(chlorido{2-[4-(dimethylamino)benzylideneamino]-pyridine-κN}mercury(II))

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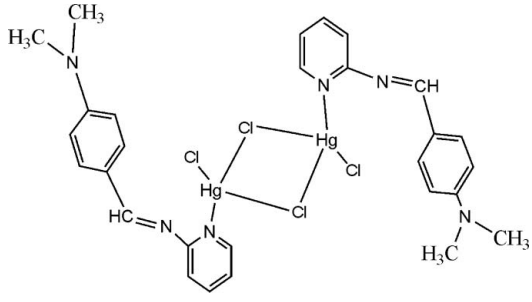
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.020 Å; R factor = 0.069; wR factor = 0.205; data-to-parameter ratio = 14.5.

The title complex, $[Hg_2Cl_4(C_{14}H_{15}N_3)_2]$, has a centre of symmetry at the centre of the four-membered ring formed by the two Hg and two bridging Cl atoms. Each Hg^{II} atom is four-coordinated in a distorted tetrahedral coordination geometry by one N atom from the pyridyl ring of a Schiff base ligand, two bridging Cl atoms and one terminal Cl atom. The Hg···Hg distance is 3.774 (2) Å. In the crystal structure, the molecules are linked into a two-dimensional network by intermolecular C—H···Cl hydrogen bonds.

Related literature

For biological activity, see: Yang *et al.* (2000). For synthesis of related compounds, see: Mondal *et al.* (2001); Tarafder *et al.* (2002).



Experimental

Crystal data

$[Hg_2Cl_4(C_{14}H_{15}N_3)_2]$
M_r = 993.56
Triclinic, $P\overline{1}$
a = 8.9474 (16) Å
b = 9.0853 (17) Å
c = 10.202 (2) Å
α = 73.597 (1)°
β = 79.807 (2)°
γ = 79.619 (2)°
V = 775.5 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 10.26 mm^{-1}
T = 298 (2) K
0.28 × 0.27 × 0.27 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.161, T_{max} = 0.168
(expected range = 0.060–0.063)
3911 measured reflections
2660 independent reflections
2239 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.205
S = 1.05
2660 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 3.62 e Å^{-3}
$\Delta\rho_{min}$ = −4.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Hg1—N1	2.173 (12)	Hg1—Cl1	2.655 (4)
Hg1—Cl2	2.344 (5)	Hg1—Cl1^{i}	2.785 (4)
N1—Hg1—Cl2	153.4 (3)	N1—Hg1—Cl1^{i}	89.9 (3)
N1—Hg1—Cl1	99.5 (3)	Cl2—Hg1—Cl1^{i}	100.32 (19)
Cl2—Hg1—Cl1	104.57 (16)	Cl1—Hg1—Cl1^{i}	92.18 (11)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···Cl2^{ii}	0.93	2.82	3.626 (18)	145
C5—H5···Cl1^{i}	0.93	2.80	3.433 (16)	127
C8—H8···Cl1^{iii}	0.93	2.80	3.628 (15)	148

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2406).

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supplementary materials

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Di-μ-chlorido-bis(chlorido{2-[4-(dimethylamino)benzylideneamino]pyridine-κN}mercury(II))

Q. Wang and D. Wang

Comment

Schiff bases have been intensively investigated recently owing to their strong coordination capability and diverse biological activities, such as antibacterial, antitumor activities *etc* (Yang *et al.*, 2000; Mondal *et al.*, 2001; Tarafder *et al.*, 2002). We report here the synthesis and crystal structure of the title compound, a new mercury(II) complex, with a monodentate Schiff base ligand derived from the condensation of *p*-dimethylaminobenzaldehyde and 2-aminopyridine.

The title complex (Fig.1) possesses a crystallographically imposed center of symmetry. The two crystallographically equivalent mercury atoms are bridged by two Cl atoms. Each Hg^{II} atom is four-coordinated in a distorted tetrahedral coordination geometry by one N atom from the pyridyl ring of a Schiff base ligand, two bridging Cl atoms and one terminal Cl atom. There is significant distortion from tetrahedral geometry, the angles about the metal ranging from 89.9 (3)–153.4 (3)° (Table 1).

As seen in Fig. 2, the molecules are linked into a two-dimensional framework by intermolecular C—H···Cl hydrogen bonds (Table 2).

Experimental

p-dimethylaminobenzaldehyde (1 mmol, 149.2 mg) in hot absolute ethanol (10 ml) was added dropwise to a absolute ethanol solution (5 ml) of 2-aminopyridine (1 mmol, 94.1 mg). The mixture was heated under reflux with stirring for 4 h. An absolute ethanol solution (5 ml) of mercury chloride (1 mmol, 217.2 mg) was then added dropwise, and the mixture was stirred at 323 K for another 8 h. The solution was kept at room temperature for about two weeks, after which large red-brown block-shaped crystals of the title complex suitable for X-ray diffraction analysis were obtained.

Refinement

All H-atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 (methylene) or 0.93 Å (aromatic) and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

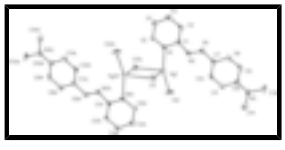

Fig. 1. The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Atoms labelled with the suffix A are generated by the symmetry operation ($-x$, $2-y$, $1-z$). H atoms have been omitted for clarity.

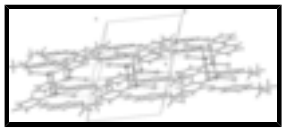

Fig. 2. The crystal packing of the title complex, viewed approximately along the *a* axis.

Di-μ-Chlorido-bis({2-[4-(dimethylamino)benzylideneamino]pyridine-κ*N*} chloridomercury(II))

Crystal data

$[Hg_2Cl_4(C_{14}H_{15}N_3)_2]$	$Z = 1$
$M_r = 993.56$	$F_{000} = 468$
Triclinic, $P\overline{1}$	$D_x = 2.127$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.9474$ (16) Å	Cell parameters from 2345 reflections
$b = 9.0853$ (17) Å	$\theta = 2.1$–27.9º
$c = 10.202$ (2) Å	$\mu = 10.26$ mm^{-1}
$\alpha = 73.597$ (1)º	$T = 298$ (2) K
$\beta = 79.807$ (2)º	Block, red-brown
$\gamma = 79.619$ (2)º	0.28 × 0.27 × 0.27 mm
$V = 775.5$ (3) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	2660 independent reflections
Radiation source: fine-focus sealed tube	2239 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.037$
$T = 298(2)$ K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 7$
$T_{min} = 0.161$, $T_{max} = 0.168$	$k = -10 \rightarrow 10$
3911 measured reflections	$l = -12 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.070$	H-atom parameters constrained
$wR(F^2) = 0.205$	$w = 1/[\sigma^2(F_o^2) + (0.095P)^2 + 4.2002P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
2660 reflections	$\Delta\rho_{max} = 3.62$ e Å^{-3}
183 parameters	$\Delta\rho_{min} = -4.39$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.05073 (6)	0.79012 (6)	0.59293 (5)	0.0476 (3)
Cl1	0.2155 (4)	1.0226 (4)	0.4919 (5)	0.0523 (9)
Cl2	0.0013 (8)	0.7687 (6)	0.8306 (5)	0.0838 (16)
N1	0.0817 (12)	0.6988 (12)	0.4132 (12)	0.038 (2)
N2	0.2356 (12)	0.5164 (12)	0.5504 (12)	0.040 (3)
N3	0.6090 (15)	0.1517 (14)	1.0596 (15)	0.053 (3)
C1	0.1761 (14)	0.5656 (14)	0.4248 (13)	0.033 (3)
C2	0.2015 (17)	0.4915 (16)	0.3185 (18)	0.050 (4)
H2	0.2641	0.3967	0.3268	0.061*
C3	0.1337 (19)	0.5597 (19)	0.2026 (19)	0.055 (4)
H3	0.1528	0.5130	0.1299	0.066*
C4	0.0369 (17)	0.6973 (18)	0.1926 (18)	0.050 (4)
H4	−0.0126	0.7421	0.1150	0.060*
C5	0.0144 (16)	0.7676 (16)	0.2981 (16)	0.045 (3)
H5	−0.0473	0.8628	0.2907	0.054*
C6	0.3453 (15)	0.4021 (15)	0.5702 (15)	0.042 (3)
H6	0.3847	0.3553	0.4989	0.050*
C7	0.4084 (15)	0.3450 (14)	0.6964 (16)	0.040 (3)
C8	0.5328 (15)	0.2215 (15)	0.7054 (16)	0.044 (3)
H8	0.5696	0.1829	0.6288	0.053*
C9	0.5995 (15)	0.1581 (15)	0.8230 (18)	0.047 (4)
H9	0.6802	0.0774	0.8253	0.057*
C10	0.5470 (16)	0.2139 (15)	0.9402 (16)	0.043 (3)
C11	0.4236 (17)	0.3374 (17)	0.9313 (17)	0.050 (4)
H11	0.3871	0.3774	1.0072	0.059*
C12	0.3575 (16)	0.3986 (16)	0.8127 (17)	0.047 (3)
H12	0.2761	0.4785	0.8105	0.056*
C13	0.7362 (17)	0.0248 (18)	1.073 (2)	0.061 (5)
H13A	0.7170	−0.0526	1.0333	0.091*
H13B	0.7456	−0.0203	1.1692	0.091*
H13C	0.8296	0.0639	1.0265	0.091*
C14	0.546 (2)	0.199 (2)	1.184 (2)	0.076 (5)
H14A	0.5378	0.3096	1.1656	0.113*

H14B	0.6120	0.1511	1.2538	0.113*
H14C	0.4461	0.1677	1.2165	0.113*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.0615 (4)	0.0461 (4)	0.0394 (4)	−0.0006 (3)	−0.0138 (3)	−0.0178 (3)
Cl1	0.0460 (18)	0.0465 (18)	0.067 (3)	−0.0028 (14)	−0.0047 (17)	−0.0234 (18)
Cl2	0.139 (5)	0.088 (3)	0.038 (3)	−0.039 (3)	−0.005 (3)	−0.026 (2)
N1	0.040 (5)	0.037 (5)	0.042 (7)	−0.008 (4)	−0.007 (5)	−0.018 (5)
N2	0.046 (6)	0.034 (5)	0.037 (7)	−0.003 (5)	−0.005 (5)	−0.007 (5)
N3	0.061 (7)	0.041 (6)	0.059 (9)	−0.001 (6)	−0.024 (7)	−0.011 (6)
C1	0.044 (6)	0.033 (6)	0.025 (6)	−0.013 (5)	−0.007 (5)	−0.003 (5)
C2	0.054 (8)	0.039 (7)	0.064 (11)	−0.002 (6)	−0.004 (8)	−0.028 (7)
C3	0.064 (9)	0.052 (8)	0.065 (11)	−0.017 (7)	−0.008 (8)	−0.035 (8)
C4	0.051 (8)	0.053 (8)	0.056 (10)	−0.012 (7)	−0.019 (7)	−0.019 (7)
C5	0.050 (8)	0.044 (7)	0.043 (9)	0.000 (6)	−0.014 (7)	−0.015 (6)
C6	0.043 (7)	0.045 (7)	0.034 (8)	−0.003 (6)	0.002 (6)	−0.011 (6)
C7	0.041 (7)	0.031 (6)	0.048 (9)	−0.003 (5)	−0.008 (6)	−0.007 (6)
C8	0.038 (6)	0.039 (7)	0.051 (9)	0.003 (5)	−0.005 (6)	−0.012 (6)
C9	0.035 (6)	0.030 (6)	0.075 (11)	−0.001 (5)	−0.008 (7)	−0.011 (7)
C10	0.050 (7)	0.035 (6)	0.050 (9)	−0.014 (6)	−0.018 (7)	−0.008 (6)
C11	0.055 (8)	0.044 (7)	0.054 (10)	0.001 (6)	−0.011 (7)	−0.022 (7)
C12	0.042 (7)	0.037 (7)	0.058 (10)	0.010 (6)	−0.018 (7)	−0.011 (7)
C13	0.050 (8)	0.049 (8)	0.071 (12)	0.004 (7)	−0.028 (8)	0.010 (8)
C14	0.095 (14)	0.061 (10)	0.067 (13)	0.007 (10)	−0.032 (11)	−0.009 (9)

Geometric parameters (Å, °)

Hg1—N1	2.173 (12)	C5—H5	0.93
Hg1—Cl2	2.344 (5)	C6—C7	1.42 (2)
Hg1—Cl1	2.655 (4)	C6—H6	0.93
Hg1—Cl1^{i}	2.785 (4)	C7—C12	1.38 (2)
Hg1—N2	2.825 (10)	C7—C8	1.426 (17)
Cl1—Hg1^{i}	2.785 (4)	C8—C9	1.37 (2)
N1—C1	1.334 (16)	C8—H8	0.93
N1—C5	1.354 (19)	C9—C10	1.40 (2)
N2—C6	1.291 (16)	C9—H9	0.93
N2—C1	1.399 (17)	C10—C11	1.421 (19)
N3—C10	1.36 (2)	C11—C12	1.37 (2)
N3—C14	1.45 (2)	C11—H11	0.93
N3—C13	1.462 (18)	C12—H12	0.93
C1—C2	1.40 (2)	C13—H13A	0.96
C2—C3	1.36 (2)	C13—H13B	0.96
C2—H2	0.93	C13—H13C	0.96
C3—C4	1.38 (2)	C14—H14A	0.96
C3—H3	0.93	C14—H14B	0.96
C4—C5	1.37 (2)	C14—H14C	0.96

C4—H4	0.93		
N1—Hg1—Cl2	153.4 (3)	C4—C5—H5	119.9
N1—Hg1—Cl1	99.5 (3)	N2—C6—C7	122.8 (13)
Cl2—Hg1—Cl1	104.57 (16)	N2—C6—H6	118.6
N1—Hg1—Cl1^{i}	89.9 (3)	C7—C6—H6	118.6
Cl2—Hg1—Cl1^{i}	100.32 (19)	C12—C7—C6	125.1 (12)
Cl1—Hg1—Cl1^{i}	92.18 (11)	C12—C7—C8	116.9 (14)
N1—Hg1—N2	52.0 (4)	C6—C7—C8	118.0 (13)
Cl2—Hg1—N2	108.4 (3)	C9—C8—C7	122.1 (14)
Cl1—Hg1—N2	108.2 (2)	C9—C8—H8	118.9
Cl1^{i}—Hg1—N2	138.5 (3)	C7—C8—H8	118.9
Hg1—Cl1—Hg1^{i}	87.82 (11)	C8—C9—C10	120.4 (12)
C1—N1—C5	121.5 (12)	C8—C9—H9	119.8
C1—N1—Hg1	113.7 (9)	C10—C9—H9	119.8
C5—N1—Hg1	124.8 (9)	N3—C10—C9	121.9 (13)
C6—N2—C1	120.2 (12)	N3—C10—C11	120.5 (14)
C6—N2—Hg1	156.2 (11)	C9—C10—C11	117.6 (14)
C1—N2—Hg1	81.9 (7)	C12—C11—C10	121.1 (14)
C10—N3—C14	122.0 (13)	C12—C11—H11	119.5
C10—N3—C13	122.3 (15)	C10—C11—H11	119.5
C14—N3—C13	115.5 (14)	C11—C12—C7	121.9 (12)
N1—C1—C2	119.5 (13)	C11—C12—H12	119.1
N1—C1—N2	112.3 (11)	C7—C12—H12	119.1
C2—C1—N2	128.2 (12)	N3—C13—H13A	109.5
C3—C2—C1	119.3 (13)	N3—C13—H13B	109.5
C3—C2—H2	120.3	H13A—C13—H13B	109.5
C1—C2—H2	120.3	N3—C13—H13C	109.5
C2—C3—C4	120.2 (15)	H13A—C13—H13C	109.5
C2—C3—H3	119.9	H13B—C13—H13C	109.5
C4—C3—H3	119.9	N3—C14—H14A	109.5
C5—C4—C3	119.2 (15)	N3—C14—H14B	109.5
C5—C4—H4	120.4	H14A—C14—H14B	109.5
C3—C4—H4	120.4	N3—C14—H14C	109.5
N1—C5—C4	120.2 (13)	H14A—C14—H14C	109.5
N1—C5—H5	119.9	H14B—C14—H14C	109.5

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···Cl2^{ii}	0.93	2.82	3.626 (18)	145
C5—H5···Cl1^{i}	0.93	2.80	3.433 (16)	127
C8—H8···Cl1^{iii}	0.93	2.80	3.628 (15)	148

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

Fig. 1

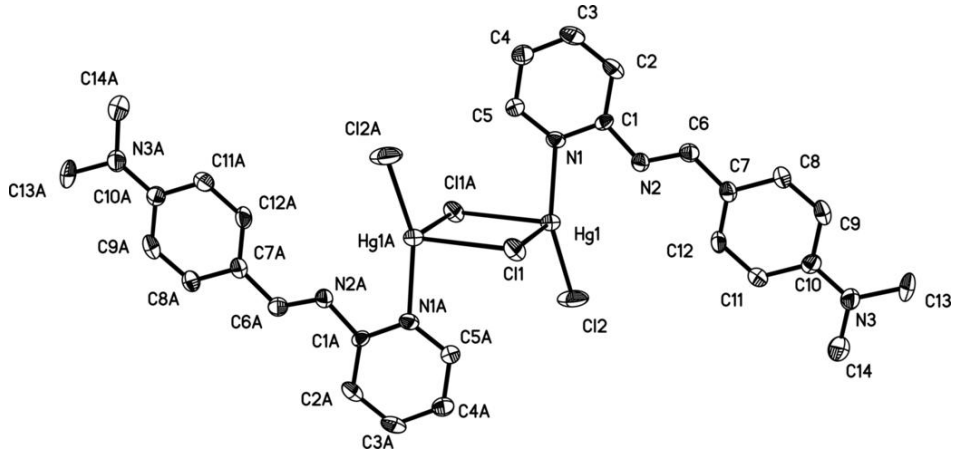
C3
C4
C2
C14A
C5
C1
C6
Cl2A
N3A
N1
C11A
C13A
C10A
Cl1A
N2
C7
C8
C12A
C9
Hg1A
Hg1
C9A
C7A
C12
Cl1
N2A
C8A
C10
C11
C13
N1A
C6A
Cl2
N3
C1A
C5A
C14
C2A
C4A
C3A

Fig. 2