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Di-μ-chlorido-bis{chlorido[4-nitro-*N*-(pyridin-2-ylmethylidene-κN)aniline-κN]mercury(II)}

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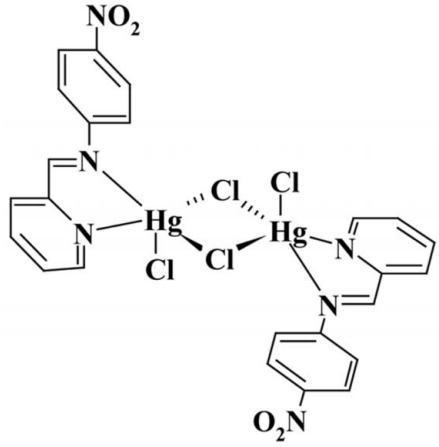
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.007 Å; R factor = 0.028; wR factor = 0.064; data-to-parameter ratio = 17.7.

In the centrosymmetric dinuclear title complex, $[Hg_2Cl_4(C_{12}H_9N_3O_2)_2]$, the Hg^{II} ion is in a distorted square-pyramidal coordination environment formed by the N atoms of the diimine ligand, two bridging Cl atoms and one terminal Cl atom. One of the bridging Hg—Cl bonds is significantly longer than the other.

Related literature

For background to diimine complexes and related structures, see: Dehghanpour *et al.* (2007); Mahmoudi *et al.* (2009).



Experimental

Crystal data

$[Hg_2Cl_4(C_{12}H_9N_3O_2)_2]$
M_r = 997.42
Monoclinic, $P2_1/c$
a = 8.9731 (2) Å
b = 7.8439 (3) Å
c = 20.1403 (7) Å
β = 98.155 (2)°
V = 1403.22 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 11.35 mm^{-1}
T = 150 K
0.18 × 0.16 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.115, T_{max} = 0.222
11428 measured reflections
3197 independent reflections
2639 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.064
S = 1.06
3197 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.94 e Å^{-3}
$\Delta\rho_{min}$ = −1.56 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hg1—N1	2.323 (4)	Hg1—Cl2	2.5161 (12)
Hg1—Cl1	2.3940 (11)	Hg1—Cl2^{i}	2.8068 (11)
Hg1—N2	2.442 (4)		

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

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

We are grateful to Bu-Ali Sina and Alzahra Universities for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2347).

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supplementary materials

Acta Cryst. (2011). E**67**, m327 [**doi:10.1107/S1600536811004703**]

Di-μ-chlorido-bis{chlorido[4-nitro-*N*-(pyridin-2-ylmethylidene-κN)aniline-κN]mercury(II)}

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Comment

In our ongoing studies on the synthesis, structural and spectroscopic characterization of transition metal complexes with diimine ligands (Dehghanpour *et al.,* 2007; Mahmoudi *et al.,* 2009), we report herein the crystal structure of the title complex. The title compound was prepared by the reaction of $HgCl_2$ with (4-nitrophenyl)pyridin-2-ylmethyleneamine.

The molecluar structure of the title complex is shown in Fig. 1. The unique Hg^{II} ion in is in a distorted square pyramidal coordination environment formed by a bis-chelating ligand, two bridging Cl atoms and one terminal Cl atom.

Experimental

The title complex was prepared by the reaction of $HgCl_2$ (22.7 mg, 0.1 mmol) and (4-nitrophenyl)pyridin-2-ylmethyleneamine (27.2 mg, 0.1mmol) in 15 ml acetonitrile at room temperature. The solution was then concentrated under vacuum, and diffusion of diethyl ether vapor into the concentrated solution gave yellow crystals of the title compound in 60% yield.

Refinement

The H- atom positions were calculated and refined in a riding model approximatiom with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

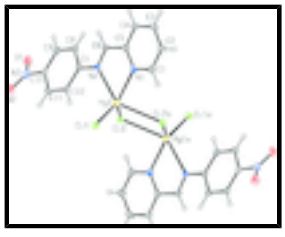


Fig. 1. A view of the structure of the title complex, with displacement ellipsoids drawn at the 50% probability level. Symmetry code: (*a*) -*x* + 1, -*y* + 1, -*z* + 1.

Di-μ-chlorido-bis{chlorido[4-nitro-*N*-(pyridin-2-ylmethylidene- κ*N*)aniline-κ*N*]mercury(II)}

Crystal data

$[Hg_2Cl_4(C_{12}H_9N_3O_2)_2]$	$F(000) = 928$
$M_r = 997.42$	$D_x = 2.361$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 6448 reflections
$a = 8.9731$ (2) Å	$\theta = 2.6–27.5°$
$b = 7.8439$ (3) Å	$\mu = 11.35$ mm^{-1}

c = 20.1403 (7) Å	T = 150 K
β = 98.155 (2)°	Block, colourless
V = 1403.22 (8) Å^3	0.18 × 0.16 × 0.14 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	3197 independent reflections
Radiation source: fine-focus sealed tube	2639 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.055
Detector resolution: 9 pixels mm^{-1}	θ_{max} = 27.6°, θ_{min} = 2.8°
φ scans and ω scans with κ offsets	h = −11→11
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)	k = −9→10
T_{min} = 0.115, T_{max} = 0.222	l = −25→26
11428 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.028	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.064	H-atom parameters constrained
S = 1.06	$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 1.6835P]$ where $P = (F_o^2 + 2F_c^2)/3$
3197 reflections	$(\Delta/\sigma)_{max}$ = 0.001
181 parameters	$\Delta\rho_{max}$ = 0.94 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −1.56 e Å^{-3}

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	0.641245 (19)	0.62356 (2)	0.448116 (9)	0.02542 (8)

Cl1	0.83299 (13)	0.45830 (16)	0.40698 (7)	0.0314 (3)
Cl2	0.60508 (13)	0.60053 (14)	0.56941 (6)	0.0270 (3)
O1	1.0314 (4)	1.2850 (5)	0.72381 (19)	0.0428 (9)
O2	1.1963 (4)	1.0918 (5)	0.7078 (2)	0.0440 (10)
N1	0.5013 (4)	0.7782 (5)	0.36266 (19)	0.0238 (8)
N2	0.6929 (4)	0.9273 (5)	0.4652 (2)	0.0216 (8)
N3	1.0753 (5)	1.1652 (5)	0.6919 (2)	0.0319 (10)
C1	0.4077 (5)	0.7090 (6)	0.3123 (2)	0.0265 (10)
H1A	0.3985	0.5884	0.3101	0.032*
C2	0.3234 (5)	0.8064 (7)	0.2632 (3)	0.0308 (11)
H2A	0.2589	0.7531	0.2277	0.037*
C3	0.3344 (5)	0.9808 (7)	0.2663 (3)	0.0312 (11)
H3A	0.2769	1.0502	0.2335	0.037*
C4	0.4315 (5)	1.0539 (6)	0.3187 (2)	0.0275 (10)
H4A	0.4404	1.1744	0.3222	0.033*
C5	0.5152 (5)	0.9497 (6)	0.3657 (2)	0.0212 (9)
C6	0.6209 (5)	1.0210 (6)	0.4202 (3)	0.0228 (10)
H6A	0.6364	1.1408	0.4221	0.027*
C7	0.7901 (5)	0.9952 (6)	0.5209 (2)	0.0206 (9)
C8	0.7776 (5)	1.1611 (6)	0.5433 (2)	0.0239 (10)
H8A	0.7039	1.2351	0.5203	0.029*
C9	0.8717 (5)	1.2189 (6)	0.5988 (2)	0.0265 (10)
H9A	0.8641	1.3324	0.6145	0.032*
C10	0.9778 (5)	1.1072 (6)	0.6314 (2)	0.0242 (10)
C11	0.9934 (5)	0.9425 (6)	0.6092 (2)	0.0257 (10)
H11A	1.0688	0.8696	0.6316	0.031*
C12	0.8981 (5)	0.8855 (6)	0.5542 (2)	0.0241 (10)
H12A	0.9058	0.7717	0.5388	0.029*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.03088 (12)	0.01758 (11)	0.02789 (12)	0.00384 (7)	0.00446 (8)	0.00096 (8)
Cl1	0.0311 (6)	0.0273 (6)	0.0363 (7)	0.0085 (5)	0.0069 (5)	−0.0012 (5)
Cl2	0.0332 (6)	0.0244 (6)	0.0233 (6)	−0.0052 (4)	0.0035 (5)	−0.0015 (5)
O1	0.037 (2)	0.046 (2)	0.043 (2)	−0.0073 (18)	0.0020 (17)	−0.017 (2)
O2	0.0277 (19)	0.055 (3)	0.045 (2)	0.0031 (17)	−0.0075 (16)	−0.0044 (19)
N1	0.0249 (19)	0.022 (2)	0.025 (2)	0.0021 (16)	0.0057 (16)	0.0018 (17)
N2	0.0210 (18)	0.0192 (19)	0.025 (2)	0.0007 (15)	0.0039 (15)	0.0047 (16)
N3	0.026 (2)	0.037 (3)	0.033 (3)	−0.0090 (18)	0.0047 (18)	−0.003 (2)
C1	0.029 (2)	0.026 (3)	0.024 (3)	−0.003 (2)	0.0052 (19)	0.001 (2)
C2	0.029 (2)	0.035 (3)	0.026 (3)	−0.004 (2)	−0.004 (2)	−0.004 (2)
C3	0.033 (3)	0.036 (3)	0.023 (3)	0.012 (2)	0.000 (2)	0.006 (2)
C4	0.035 (3)	0.021 (2)	0.026 (3)	0.004 (2)	0.003 (2)	0.001 (2)
C5	0.026 (2)	0.021 (2)	0.017 (2)	0.0034 (19)	0.0048 (18)	0.0020 (19)
C6	0.027 (2)	0.013 (2)	0.029 (3)	0.0024 (18)	0.0064 (19)	−0.001 (2)
C7	0.019 (2)	0.019 (2)	0.024 (3)	0.0006 (17)	0.0067 (18)	0.0006 (19)
C8	0.025 (2)	0.022 (2)	0.024 (3)	0.0018 (18)	0.0044 (19)	0.0039 (19)

C9	0.025 (2)	0.020 (2)	0.035 (3)	−0.0029 (19)	0.009 (2)	−0.006 (2)
C10	0.020 (2)	0.026 (3)	0.027 (3)	−0.0062 (18)	0.0036 (18)	−0.002 (2)
C11	0.024 (2)	0.023 (2)	0.030 (3)	0.004 (2)	0.0027 (19)	0.004 (2)
C12	0.022 (2)	0.020 (2)	0.031 (3)	−0.0005 (18)	0.0053 (19)	−0.001 (2)

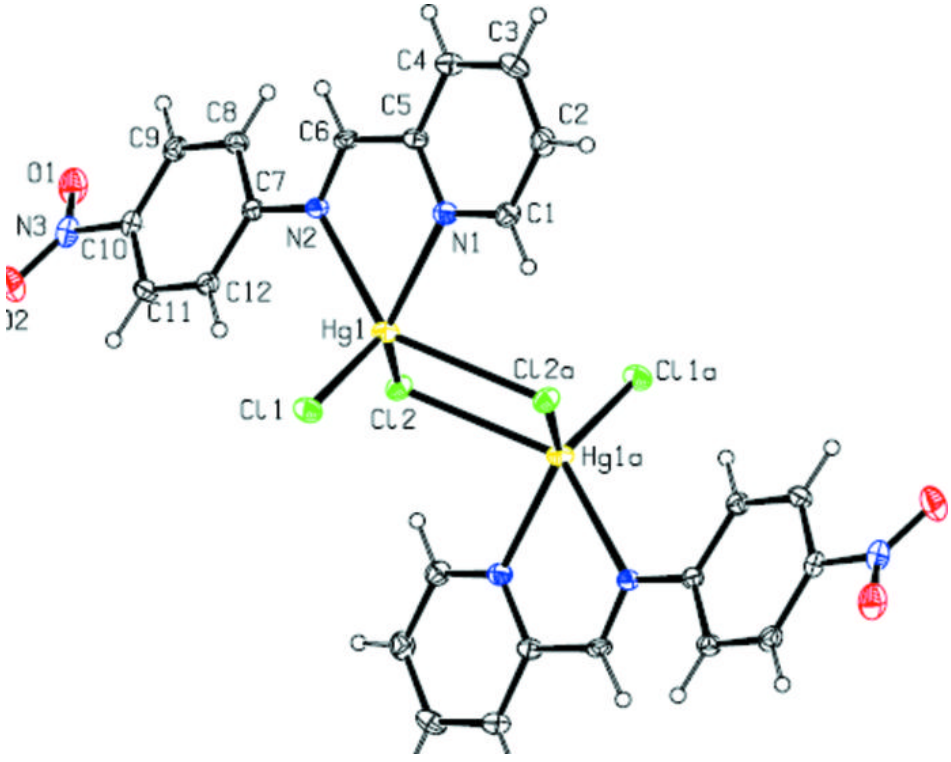
Geometric parameters (Å, °)

Hg1—N1	2.323 (4)	C3—C4	1.392 (7)
Hg1—Cl1	2.3940 (11)	C3—H3A	0.9500
Hg1—N2	2.442 (4)	C4—C5	1.388 (6)
Hg1—Cl2	2.5161 (12)	C4—H4A	0.9500
Hg1—Cl2^{i}	2.8068 (11)	C5—C6	1.457 (6)
Cl2—Hg1^{i}	2.8068 (11)	C6—H6A	0.9500
O1—N3	1.233 (5)	C7—C8	1.387 (6)
O2—N3	1.231 (5)	C7—C12	1.394 (6)
N1—C1	1.337 (6)	C8—C9	1.380 (7)
N1—C5	1.352 (6)	C8—H8A	0.9500
N2—C6	1.272 (6)	C9—C10	1.388 (7)
N2—C7	1.423 (6)	C9—H9A	0.9500
N3—C10	1.468 (6)	C10—C11	1.381 (6)
C1—C2	1.386 (7)	C11—C12	1.375 (7)
C1—H1A	0.9500	C11—H11A	0.9500
C2—C3	1.372 (7)	C12—H12A	0.9500
C2—H2A	0.9500		
N1—Hg1—Cl1	111.46 (10)	C4—C3—H3A	120.7
N1—Hg1—N2	70.57 (13)	C5—C4—C3	119.6 (5)
Cl1—Hg1—N2	116.47 (9)	C5—C4—H4A	120.2
N1—Hg1—Cl2	128.76 (9)	C3—C4—H4A	120.2
Cl1—Hg1—Cl2	119.68 (4)	N1—C5—C4	121.2 (4)
N2—Hg1—Cl2	88.91 (9)	N1—C5—C6	117.6 (4)
N1—Hg1—Cl2^{i}	84.28 (9)	C4—C5—C6	121.3 (4)
Cl1—Hg1—Cl2^{i}	102.07 (4)	N2—C6—C5	121.8 (4)
N2—Hg1—Cl2^{i}	139.38 (9)	N2—C6—H6A	119.1
Cl2—Hg1—Cl2^{i}	82.50 (4)	C5—C6—H6A	119.1
Hg1—Cl2—Hg1^{i}	97.50 (4)	C8—C7—C12	120.4 (4)
C1—N1—C5	118.8 (4)	C8—C7—N2	122.6 (4)
C1—N1—Hg1	124.5 (3)	C12—C7—N2	117.0 (4)
C5—N1—Hg1	116.7 (3)	C9—C8—C7	120.2 (4)
C6—N2—C7	122.6 (4)	C9—C8—H8A	119.9
C6—N2—Hg1	113.3 (3)	C7—C8—H8A	119.9
C7—N2—Hg1	124.0 (3)	C8—C9—C10	118.4 (4)
O2—N3—O1	123.8 (4)	C8—C9—H9A	120.8
O2—N3—C10	118.1 (4)	C10—C9—H9A	120.8
O1—N3—C10	118.1 (4)	C11—C10—C9	122.2 (4)
N1—C1—C2	122.6 (5)	C11—C10—N3	118.9 (4)
N1—C1—H1A	118.7	C9—C10—N3	119.0 (4)
C2—C1—H1A	118.7	C12—C11—C10	119.0 (4)

C3—C2—C1	119.2 (5)	C12—C11—H11A	120.5
C3—C2—H2A	120.4	C10—C11—H11A	120.5
C1—C2—H2A	120.4	C11—C12—C7	119.8 (4)
C2—C3—C4	118.6 (4)	C11—C12—H12A	120.1
C2—C3—H3A	120.7	C7—C12—H12A	120.1
N1—Hg1—Cl2—Hg1^{i}	76.43 (12)	C1—N1—C5—C6	−178.4 (4)
Cl1—Hg1—Cl2—Hg1^{i}	−99.66 (5)	Hg1—N1—C5—C6	2.7 (5)
N2—Hg1—Cl2—Hg1^{i}	140.20 (9)	C3—C4—C5—N1	−1.8 (7)
Cl2^{i}—Hg1—Cl2—Hg1^{i}	0.0	C3—C4—C5—C6	178.4 (4)
Cl1—Hg1—N1—C1	68.2 (4)	C7—N2—C6—C5	−176.3 (4)
N2—Hg1—N1—C1	179.9 (4)	Hg1—N2—C6—C5	1.9 (5)
Cl2—Hg1—N1—C1	−108.1 (3)	N1—C5—C6—N2	−3.2 (6)
Cl2^{i}—Hg1—N1—C1	−32.5 (3)	C4—C5—C6—N2	176.7 (4)
Cl1—Hg1—N1—C5	−112.9 (3)	C6—N2—C7—C8	22.2 (7)
N2—Hg1—N1—C5	−1.3 (3)	Hg1—N2—C7—C8	−155.9 (3)
Cl2—Hg1—N1—C5	70.7 (3)	C6—N2—C7—C12	−159.7 (4)
Cl2^{i}—Hg1—N1—C5	146.3 (3)	Hg1—N2—C7—C12	22.3 (5)
N1—Hg1—N2—C6	−0.3 (3)	C12—C7—C8—C9	−0.2 (7)
Cl1—Hg1—N2—C6	104.6 (3)	N2—C7—C8—C9	177.9 (4)
Cl2—Hg1—N2—C6	−132.5 (3)	C7—C8—C9—C10	−0.2 (7)
Cl2^{i}—Hg1—N2—C6	−55.3 (4)	C8—C9—C10—C11	1.2 (7)
N1—Hg1—N2—C7	177.8 (3)	C8—C9—C10—N3	−177.9 (4)
Cl1—Hg1—N2—C7	−77.2 (3)	O2—N3—C10—C11	23.7 (6)
Cl2—Hg1—N2—C7	45.7 (3)	O1—N3—C10—C11	−156.4 (4)
Cl2^{i}—Hg1—N2—C7	122.8 (3)	O2—N3—C10—C9	−157.1 (4)
C5—N1—C1—C2	−0.4 (7)	O1—N3—C10—C9	22.7 (6)
Hg1—N1—C1—C2	178.4 (3)	C9—C10—C11—C12	−1.8 (7)
N1—C1—C2—C3	−0.8 (7)	N3—C10—C11—C12	177.3 (4)
C1—C2—C3—C4	0.7 (7)	C10—C11—C12—C7	1.4 (7)
C2—C3—C4—C5	0.5 (7)	C8—C7—C12—C11	−0.5 (7)
C1—N1—C5—C4	1.7 (6)	N2—C7—C12—C11	−178.7 (4)
Hg1—N1—C5—C4	−177.2 (3)		

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

Fig. 1


C3
C4
C5
C2
C6
C8
C9
C7
O1
N3
C10
N2
N1
C1
O2
C11
C12
Hg1
Cl2a
Cl1a
Cl1
Cl2
Hg1a