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

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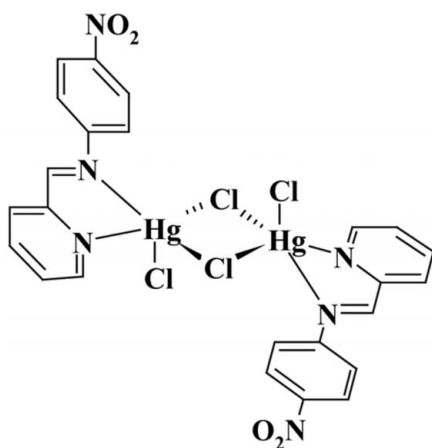
Received 3 February 2011; accepted 7 February 2011

Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.028; wR factor = 0.064; data-to-parameter ratio = 17.7.

In the centrosymmetric dinuclear title complex, $[\text{Hg}_2\text{Cl}_4(\text{C}_{12}\text{H}_9\text{N}_3\text{O}_2)_2]$, 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

$[\text{Hg}_2\text{Cl}_4(\text{C}_{12}\text{H}_9\text{N}_3\text{O}_2)_2]$
 $M_r = 997.42$
 Monoclinic, $P2_1/c$
 $a = 8.9731$ (2) Å
 $b = 7.8439$ (3) Å
 $c = 20.1403$ (7) Å
 $\beta = 98.155$ (2)°

$V = 1403.22$ (8) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 11.35$ mm⁻¹
 $T = 150$ K
 $0.18 \times 0.16 \times 0.14$ mm

Data collection

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

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
 $\Delta\rho_{\text{max}} = 0.94$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.56$ 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*.

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

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

Sadegh Salehzadeh, Saeed Dehghanpour, Mehdi Khalaj and Mohammad Rahimishakiba

S1. 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 HgCl₂ with (4-nitrophenyl)pyridin-2-ylmethyleamine.

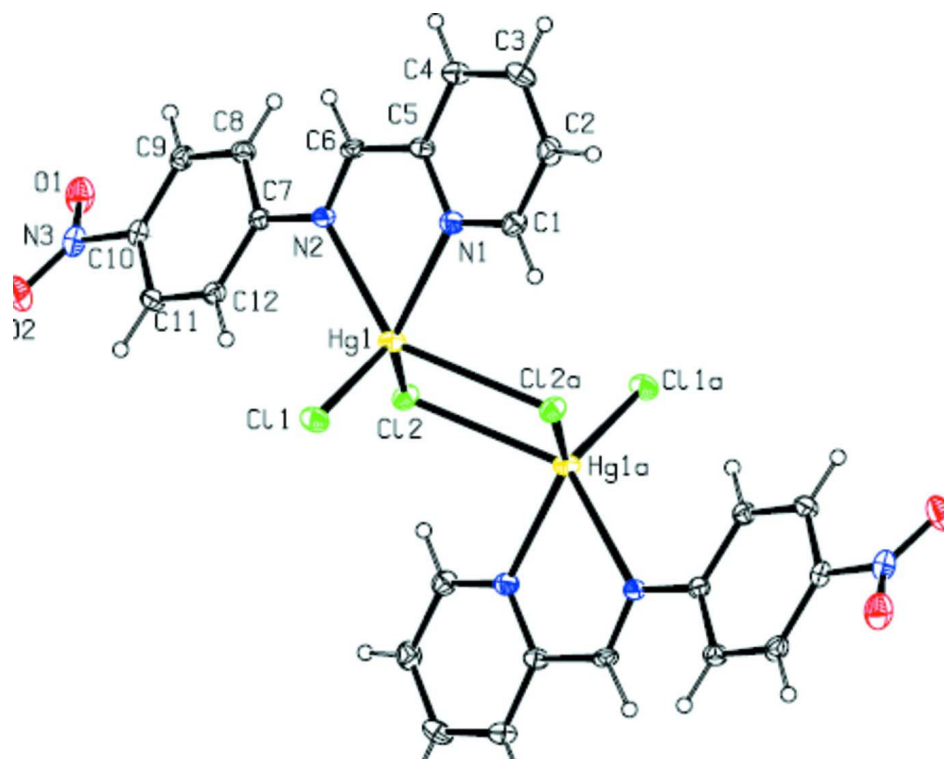
The molecular structure of the title complex is shown in Fig. 1. The unique Hg^{II} ion is in a distorted square pyramidal coordination environment formed by a bis-chelating ligand, two bridging Cl atoms and one terminal Cl atom.

S2. Experimental

The title complex was prepared by the reaction of HgCl₂ (22.7 mg, 0.1 mmol) and (4-nitrophenyl)pyridin-2-ylmethyleamine (27.2 mg, 0.1 mmol) 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.

S3. Refinement

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

**Figure 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-ylmethylene- κ N)aniline- κ N]mercury(II)}

Crystal data

$[\text{Hg}_2\text{Cl}_4(\text{C}_{12}\text{H}_9\text{N}_3\text{O}_2)_2]$

$M_r = 997.42$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1\ ybc$

$a = 8.9731\ (2)\ \text{\AA}$

$b = 7.8439\ (3)\ \text{\AA}$

$c = 20.1403\ (7)\ \text{\AA}$

$\beta = 98.155\ (2)^\circ$

$V = 1403.22\ (8)\ \text{\AA}^3$

$Z = 2$

$F(000) = 928$

$D_x = 2.361\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 6448 reflections

$\theta = 2.6\text{--}27.5^\circ$

$\mu = 11.35\ \text{mm}^{-1}$

$T = 150\ \text{K}$

Block, colourless

$0.18 \times 0.16 \times 0.14\ \text{mm}$

Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 9 pixels mm^{-1}

φ scans and ω scans with κ offsets

Absorption correction: multi-scan

(*SORTAV*; Blessing, 1995)

$T_{\min} = 0.115, T_{\max} = 0.222$

11428 measured reflections

3197 independent reflections

2639 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.055$

$\theta_{\max} = 27.6^\circ, \theta_{\min} = 2.8^\circ$

$h = -11 \rightarrow 11$

$k = -9 \rightarrow 10$

$l = -25 \rightarrow 26$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.064$
 $S = 1.06$
 3197 reflections
 181 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 1.6835P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.94 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.56 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å²)

	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)
C11	0.0311 (6)	0.0273 (6)	0.0363 (7)	0.0085 (5)	0.0069 (5)	-0.0012 (5)
C12	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—C11	2.3940 (11)	C3—H3A	0.9500
Hg1—N2	2.442 (4)	C4—C5	1.388 (6)
Hg1—C12	2.5161 (12)	C4—H4A	0.9500
Hg1—C12 ⁱ	2.8068 (11)	C5—C6	1.457 (6)
C12—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—C11	111.46 (10)	C4—C3—H3A	120.7
N1—Hg1—N2	70.57 (13)	C5—C4—C3	119.6 (5)
C11—Hg1—N2	116.47 (9)	C5—C4—H4A	120.2
N1—Hg1—C12	128.76 (9)	C3—C4—H4A	120.2
C11—Hg1—C12	119.68 (4)	N1—C5—C4	121.2 (4)
N2—Hg1—C12	88.91 (9)	N1—C5—C6	117.6 (4)
N1—Hg1—C12 ⁱ	84.28 (9)	C4—C5—C6	121.3 (4)
C11—Hg1—C12 ⁱ	102.07 (4)	N2—C6—C5	121.8 (4)
N2—Hg1—C12 ⁱ	139.38 (9)	N2—C6—H6A	119.1
C12—Hg1—C12 ⁱ	82.50 (4)	C5—C6—H6A	119.1
Hg1—C12—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—C12—Hg1 ⁱ	76.43 (12)	C1—N1—C5—C6	-178.4 (4)
C11—Hg1—C12—Hg1 ⁱ	-99.66 (5)	Hg1—N1—C5—C6	2.7 (5)
N2—Hg1—C12—Hg1 ⁱ	140.20 (9)	C3—C4—C5—N1	-1.8 (7)
C12 ⁱ —Hg1—C12—Hg1 ⁱ	0.0	C3—C4—C5—C6	178.4 (4)
C11—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)
C12—Hg1—N1—C1	-108.1 (3)	N1—C5—C6—N2	-3.2 (6)
C12 ⁱ —Hg1—N1—C1	-32.5 (3)	C4—C5—C6—N2	176.7 (4)
C11—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)
C12—Hg1—N1—C5	70.7 (3)	C6—N2—C7—C12	-159.7 (4)
C12 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)
C11—Hg1—N2—C6	104.6 (3)	N2—C7—C8—C9	177.9 (4)
C12—Hg1—N2—C6	-132.5 (3)	C7—C8—C9—C10	-0.2 (7)
C12 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)
C11—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 code: (i) $-x+1, -y+1, -z+1$.