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[Bis(2-pyridylmethyl)amine]dichloridomercury(II)

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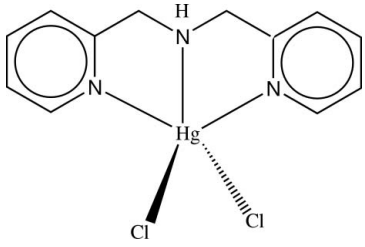
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.008 Å; disorder in main residue; R factor = 0.027; wR factor = 0.062; data-to-parameter ratio = 20.7.

The Hg atom in the title complex, $[HgCl_2(C_{12}H_{13}N_3)]$, adopts a square-pyramidal geometry, being ligated by three N atoms of the tridentate bis(2-pyridylmethyl)amine ligand and two Cl atoms, with one of the latter occupying the apical position. Disorder is noted in the amine portion of the ligand and this was modelled over two sites, with the major component having a site-occupancy factor of 0.794 (14).

Related literature

For general background, see: Ojida *et al.* (2004); Kirin *et al.* (2005); Storr *et al.* (2005); Tamamura *et al.* (2006); Kim *et al.* (2007); Lee *et al.* (2007). For related literature, see: Addison *et al.* (1984).



Experimental

Crystal data

$[HgCl_2(C_{12}H_{13}N_3)]$
M_r = 470.74
Monoclinic, $P2_1/n$
a = 8.4083 (6) Å
b = 12.8278 (11) Å
c = 13.3457 (12) Å
β = 90.462 (2)°
V = 1439.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 11.05 mm^{-1}
T = 295 (2) K
0.18 × 0.15 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.143, T_{max} = 0.185
15603 measured reflections
3580 independent reflections
2547 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.061
S = 1.01
3580 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Hg—Cl1	2.4336 (12)	Hg—N8	2.445 (5)
Hg—Cl2	2.4579 (14)	Hg—N15	2.405 (4)
Hg—N1	2.394 (4)		
Cl1—Hg—Cl2	118.63 (5)	N1—Hg—N15	133.99 (12)
N1—Hg—N8	67.82 (15)	N8—Hg—N15	68.04 (14)

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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: TK2241).

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[Bis(2-pyridylmethyl)amine]dichloridomercury(II)

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S1. Comment

Transition metal complexes with bis(2-pyridylmethyl)amine (dpa) or substituted-dpa ligands continue to be of interest in many fields of chemistry (Kirin *et al.*, 2005; Storr *et al.*, 2005; Tamamura *et al.*, 2006). Recently, we reported Cu(II) (Lee *et al.*, 2007) and Zn(II) (Kim *et al.*, 2007) halide complexes with the dpa ligand, and Zn(dpa)Cl_2 was proposed as a blue fluorescent material. In this work, as an extension of a study on fluorescent chemosensors (Ojida *et al.*, 2004), we prepared a Hg(II) complex of dpa, Hg(dpa)Cl_2 (I), and its structure and properties were investigated. The Hg(II) atom is 5-coordinated by the three N atoms of the tridentate di(picolyl)amine ligand and two Cl atoms. The coordination geometry is based on a square pyramid with the basal plane defined by three N atoms and one Cl, with the other Cl atom occupying the apical position. The calculated trigonality index, $\tau = 0.03$, indicates that the Hg atom is in a square pyramidal geometry (Addison *et al.*, 1984). Hg(dpa)Cl_2 exhibits an intense blue emission at 425 nm in DMF solution upon excitation at 400 nm.

S2. Experimental

All of the reagents and solvents were purchased from either Aldrich and used without further purification. A mixture of mercuric chloride (1.35 g, 5 mmol) and bis(2-pyridylmethyl)amine (0.99 g, 5 mmol) in methanol (20 ml) was stirred for 8 h at room temperature under a nitrogen atmosphere. The precipitates were filtered off and recrystallized from methanol in a 63% yield. ^{1}H-NMR for dpa in (I) (d_6-DMSO, p.p.m.): δ: 8.51 (d, 2H), 7.96 (t, 2H), 7.52 (m, 4H), 4.98 (s, 1H), 4.08 (s, 4H).

S3. Refinement

The C and N-bound H atoms were included in the riding model approximation with C—H = 0.93–0.97 Å and N—H = 0.91 Å, and with U_{iso}(H) = 1.2U_{eq}(C and N). Disorder was noted in the structure and this modelled so that two sites were resolved for the N8—H atoms. From refinement, the major component of the disorder had a site occupancy factor = 0.794 (14). The maximum and minimum residual electron denisty peaks were located 0.85 and 0.78 Å, respectively, from the Hg atom.

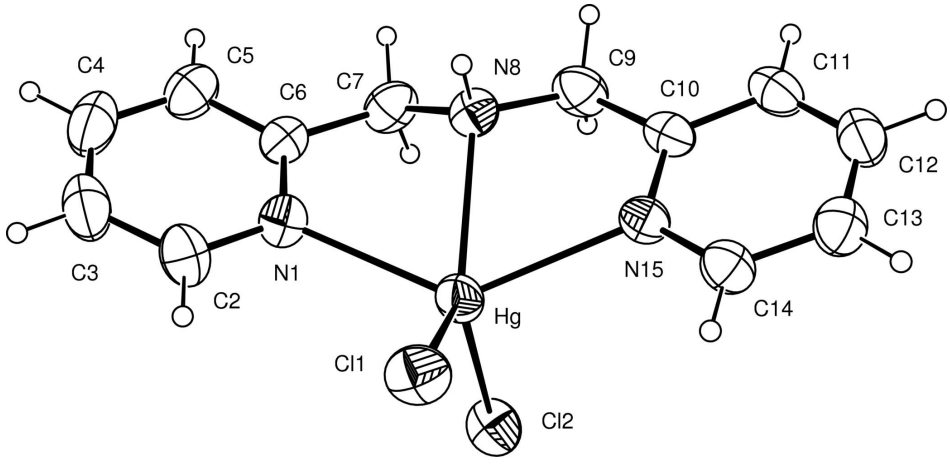

Figure 1

Molecular structure of (I), showing the atom-numbering scheme and 30% probability ellipsoids. Only the major component of the disordered atoms is shown for clarity.

[Bis(2-pyridylmethyl)amine]dichloridomercury(II)

Crystal data

$[HgCl_2(C_{12}H_{13}N_3)]$
$M_r = 470.74$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.4083\ (6)$ Å
$b = 12.8278\ (11)$ Å
$c = 13.3457\ (12)$ Å
$\beta = 90.462\ (2)°$
$V = 1439.4\ (2)$ Å^3
$Z = 4$
$F(000) = 880$
$D_x = 2.172$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4387 reflections
$\theta = 2.2–24.5°$
$\mu = 11.05$ mm^{-1}
$T = 295$ K
Block, orange
0.18 × 0.15 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.143$, $T_{max} = 0.185$
15603 measured reflections
3580 independent reflections
2547 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -11\rightarrow11$
$k = -12\rightarrow17$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.061$
$S = 1.02$
3580 reflections
173 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 0.7364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.22$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Hg	0.87912 (2)	0.305769 (14)	0.873548 (12)	0.06174 (8)	
Cl1	1.04268 (15)	0.36276 (12)	0.73426 (9)	0.0791 (3)	
Cl2	0.63148 (16)	0.21091 (10)	0.83674 (11)	0.0794 (4)	
N1	1.0536 (5)	0.1777 (3)	0.9449 (3)	0.0636 (10)	
C2	1.1638 (6)	0.1270 (4)	0.8923 (4)	0.0799 (14)	
H2	1.1708	0.1399	0.8239	0.096*	
C3	1.2665 (7)	0.0573 (5)	0.9348 (5)	0.0911 (17)	
H3	1.3425	0.0235	0.8963	0.109*	
C4	1.2548 (7)	0.0381 (5)	1.0354 (6)	0.0959 (18)	
H4	1.3227	−0.0093	1.0665	0.115*	
C5	1.1423 (6)	0.0898 (4)	1.0893 (4)	0.0792 (14)	
H5	1.1332	0.0779	1.1578	0.095*	
C6	1.0426 (5)	0.1592 (3)	1.0422 (3)	0.0576 (10)	
C7	0.9172 (6)	0.2192 (4)	1.0981 (4)	0.0727 (13)	
H7A	0.815	0.1847	1.0914	0.087*	
H7B	0.9447	0.2228	1.1688	0.087*	
N8	0.9085 (9)	0.3260 (5)	1.0549 (3)	0.0594 (19)	0.794 (14)
H8	1.0033	0.358	1.0671	0.071*	0.794 (14)
N8A	0.807 (3)	0.2914 (15)	1.0593 (11)	0.048 (7)	0.206 (14)
H8A	0.7101	0.2599	1.0615	0.057*	0.206 (14)
C9	0.7863 (6)	0.3898 (4)	1.0952 (3)	0.0746 (14)	
H9A	0.8204	0.4161	1.1601	0.09*	
H9B	0.6918	0.3478	1.1053	0.09*	
C10	0.7454 (5)	0.4802 (4)	1.0282 (3)	0.0590 (11)	
C11	0.6733 (5)	0.5697 (4)	1.0649 (4)	0.0707 (13)	
H11	0.6523	0.5763	1.1329	0.085*	
C12	0.6337 (6)	0.6482 (4)	0.9995 (4)	0.0766 (14)	
H12	0.5842	0.7081	1.0229	0.092*	
C13	0.6666 (6)	0.6383 (4)	0.9010 (4)	0.0742 (13)	
H13	0.6408	0.6912	0.856	0.089*	
C14	0.7391 (5)	0.5482 (4)	0.8685 (3)	0.0669 (12)	
H14	0.7622	0.5413	0.8007	0.08*	
N15	0.7774 (4)	0.4704 (3)	0.9307 (2)	0.0563 (8)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg	0.07430 (13)	0.06556 (13)	0.04548 (10)	0.01047 (9)	0.00744 (7)	0.00080 (8)
Cl1	0.0815 (8)	0.0984 (10)	0.0575 (6)	−0.0078 (7)	0.0157 (6)	0.0109 (6)

Cl2	0.0726 (8)	0.0803 (9)	0.0857 (9)	−0.0067 (6)	0.0138 (6)	−0.0032 (6)
N1	0.061 (2)	0.064 (2)	0.066 (2)	0.0019 (18)	0.0042 (18)	0.0070 (18)
C2	0.078 (3)	0.077 (4)	0.085 (3)	0.010 (3)	0.020 (3)	−0.004 (3)
C3	0.074 (4)	0.069 (4)	0.130 (5)	0.010 (3)	0.010 (3)	−0.020 (4)
C4	0.075 (4)	0.072 (4)	0.140 (6)	0.014 (3)	−0.019 (4)	0.006 (4)
C5	0.077 (3)	0.076 (3)	0.084 (3)	0.000 (3)	−0.009 (3)	0.017 (3)
C6	0.056 (3)	0.053 (2)	0.064 (3)	−0.007 (2)	−0.004 (2)	0.008 (2)
C7	0.073 (3)	0.086 (4)	0.059 (3)	0.001 (3)	0.000 (2)	0.017 (3)
N8	0.051 (4)	0.067 (4)	0.060 (3)	−0.003 (3)	0.004 (2)	0.003 (2)
N8A	0.046 (14)	0.053 (12)	0.045 (9)	−0.007 (9)	−0.002 (7)	0.009 (7)
C9	0.089 (4)	0.082 (4)	0.053 (3)	0.000 (3)	0.011 (2)	−0.003 (2)
C10	0.058 (3)	0.063 (3)	0.056 (2)	−0.009 (2)	0.0045 (19)	−0.012 (2)
C11	0.068 (3)	0.076 (3)	0.068 (3)	−0.005 (3)	0.010 (2)	−0.022 (3)
C12	0.058 (3)	0.062 (3)	0.109 (4)	0.002 (2)	−0.001 (3)	−0.021 (3)
C13	0.068 (3)	0.064 (3)	0.090 (4)	−0.003 (3)	−0.003 (3)	0.003 (3)
C14	0.074 (3)	0.061 (3)	0.066 (3)	−0.006 (2)	0.006 (2)	−0.001 (2)
N15	0.060 (2)	0.058 (2)	0.0507 (19)	−0.0047 (17)	0.0061 (16)	−0.0016 (16)

Geometric parameters (Å, º)

Hg—Cl1	2.4336 (12)	C7—H7A	0.97
Hg—Cl2	2.4579 (14)	C7—H7B	0.97
Hg—N1	2.394 (4)	N8—C9	1.423 (7)
Hg—N8	2.445 (5)	N8—H8	0.91
Hg—N15	2.405 (4)	N8A—C9	1.362 (18)
Hg—N8A	2.563 (17)	N8A—H8A	0.91
N1—C6	1.325 (6)	C9—C10	1.503 (7)
N1—C2	1.336 (6)	C9—H9A	0.97
C2—C3	1.364 (7)	C9—H9B	0.97
C2—H2	0.93	C10—N15	1.336 (5)
C3—C4	1.370 (8)	C10—C11	1.389 (6)
C3—H3	0.93	C11—C12	1.372 (7)
C4—C5	1.365 (8)	C11—H11	0.93
C4—H4	0.93	C12—C13	1.351 (7)
C5—C6	1.372 (6)	C12—H12	0.93
C5—H5	0.93	C13—C14	1.379 (7)
C6—C7	1.508 (7)	C13—H13	0.93
C7—N8A	1.407 (18)	C14—N15	1.337 (6)
C7—N8	1.488 (7)	C14—H14	0.93
Cl1—Hg—Cl2	118.63 (5)	H7A—C7—H7B	108.4
N1—Hg—N8	67.82 (15)	C9—N8—C7	114.6 (5)
N1—Hg—N15	133.99 (12)	C9—N8—Hg	111.6 (3)
N8—Hg—N15	68.04 (14)	C7—N8—Hg	106.9 (3)
N1—Hg—Cl1	99.30 (10)	C9—N8—H8	107.8
N15—Hg—Cl1	100.55 (9)	C7—N8—H8	107.8
Cl1—Hg—N8	132.16 (19)	Hg—N8—H8	107.8
N1—Hg—Cl2	104.77 (10)	C9—N8A—C7	124.5 (15)

N15—Hg—Cl2	101.26 (9)	C9—N8A—Hg	107.8 (10)
N8—Hg—Cl2	109.21 (19)	C7—N8A—Hg	104.1 (10)
N1—Hg—N8A	73.5 (4)	C9—N8A—H8A	106.4
N15—Hg—N8A	70.7 (4)	C7—N8A—H8A	106.4
Cl1—Hg—N8A	154.1 (6)	Hg—N8A—H8A	106.4
N8—Hg—N8A	22.0 (5)	N8A—C9—C10	122.3 (7)
Cl2—Hg—N8A	87.2 (6)	N8—C9—C10	112.4 (4)
C6—N1—C2	118.8 (4)	N8A—C9—H9A	126.8
C6—N1—Hg	117.8 (3)	N8—C9—H9A	109.1
C2—N1—Hg	123.4 (3)	C10—C9—H9A	109.1
N1—C2—C3	122.7 (5)	N8—C9—H9B	109.1
N1—C2—H2	118.6	C10—C9—H9B	109.1
C3—C2—H2	118.6	H9A—C9—H9B	107.9
C2—C3—C4	118.4 (5)	N15—C10—C11	120.9 (5)
C2—C3—H3	120.8	N15—C10—C9	117.4 (4)
C4—C3—H3	120.8	C11—C10—C9	121.7 (4)
C5—C4—C3	119.1 (5)	C12—C11—C10	119.1 (5)
C5—C4—H4	120.5	C12—C11—H11	120.5
C3—C4—H4	120.5	C10—C11—H11	120.5
C4—C5—C6	119.8 (5)	C13—C12—C11	120.0 (5)
C4—C5—H5	120.1	C13—C12—H12	120
C6—C5—H5	120.1	C11—C12—H12	120
N1—C6—C5	121.3 (5)	C12—C13—C14	118.6 (5)
N1—C6—C7	116.6 (4)	C12—C13—H13	120.7
C5—C6—C7	122.1 (4)	C14—C13—H13	120.7
N8A—C7—C6	127.9 (8)	N15—C14—C13	122.4 (4)
N8—C7—C6	108.1 (4)	N15—C14—H14	118.8
N8—C7—H7A	110.1	C13—C14—H14	118.8
C6—C7—H7A	110.1	C10—N15—C14	119.0 (4)
N8A—C7—H7B	118.6	C10—N15—Hg	117.8 (3)
N8—C7—H7B	110.1	C14—N15—Hg	122.9 (3)
C6—C7—H7B	110.1		