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Diiodidobis[(*E*)-1-(2-pyridylmethylene)-thiosemicarbazone-*κS*]mercury(II)

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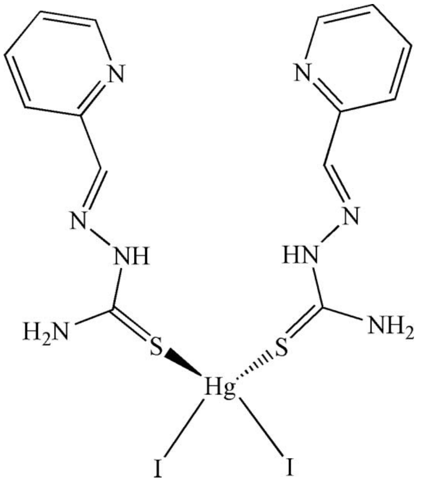
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.012 Å; R factor = 0.042; wR factor = 0.110; data-to-parameter ratio = 16.5.

The reaction of mercury(II) iodide with (*E*)-1-(2-pyridylmethylene)thiosemicarbazone afforded the title complex, $[HgI_2(C_7H_8N_4S)_2]$. The Hg^{II} centre, which lies on a twofold rotation axis, is coordinated by two S atoms and two Cl atoms in a distorted tetrahedral coordination geometry. N—H···I and N—H···N hydrogen bonds stabilize the crystal structure.

Related literature

For related literature, see: French & Blanz (1970); Klayman *et al.* (1979).



Experimental

Crystal data

$[HgI_2(C_7H_8N_4S)_2]$
M_r = 814.86
Monoclinic, $C2/c$
a = 18.973 (5) Å
b = 7.0494 (17) Å
c = 17.454 (4) Å
β = 103.137 (3)°
V = 2273.4 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 9.69 mm^{-1}
T = 298 (2) K
0.30 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.159, T_{max} = 0.444 (expected range = 0.136–0.380)
5534 measured reflections
2048 independent reflections
1888 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.110
S = 1.06
2048 reflections
124 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.96 e Å^{-3}
$\Delta\rho_{min}$ = −2.79 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···I1^{i}	0.86	3.13	3.978 (7)	169
N1—H1A···N4^{ii}	0.86	2.06	2.910 (9)	169

Symmetry codes: (i) $-x+2, y, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: BT2394).

References

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French, F. A. & Blanz, E. Jr (1970). *J. Med. Chem.* **13**, 1117–1124.
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supplementary materials

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Diiodidobis[(*E*)-1-(2-pyridylmethylene)thiosemicarbazone-*κS*]mercury(II)

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Comment

Heterocyclic thiosemicarbazones, as well as their metal complexes, are currently under discussion because of their biological activity (Klayman *et al.*, 1979). A number of studies dealing with complex formation properties and structures of thiosemicarbazones are published (French *et al.*, 1970). (*E*)-1-(1-(pyridin-2-yl)methylene) thiosemicarbazone is synthesized by 1-(pyridin-2-yl)methylene and thiosemicarbazide forming an insoluble complex.

The Hg(II) center is coordinated by two S atoms and two Cl atoms in a distorted tetrahedral coordination geometry. N—H···I and N—H···N hydrogen bonds stabilize the crystal structure.

Experimental

An methanol solution (10 ml) of HgI2 (45.4 mg, 0.10 mmol) was slowly diffused into a ethanol solution (10 ml) of (*E*)-1-(1-(pyridin-2-yl)methylene)thiosemicarbazide (36.0 mg, 0.20 mmol). Yellow single crystals of (I) were obtained after the solution was allowed to stand at room temperature for one week.

Refinement

All H atoms were included in calculated positions with N—H = 0.86, C—H = 0.93 and were included in the final cycles of refinement using a riding model with U_{iso}(H) = 1.2U_{eq}(N,*C*).

Figures

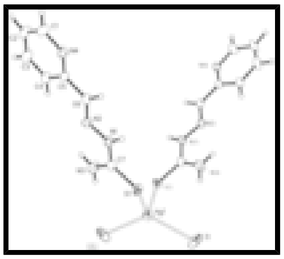

Fig. 1. The diagram of the complex with atom numbering, showing 30% probability displacement ellipsoids [Symmetry code: (i) −*x* + 2, *y*, −*z* + 1/2].

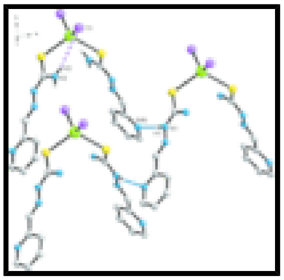

Fig. 2. A diagram showing the N—H···I and N—H···N hydrogen bonding arrangement [Symmetry codes: (i) −*x* + 2, *y*, −*z* + 1/2; (ii) −*x* + 3/2, *y* + 1/2, −*z* + 1/2].

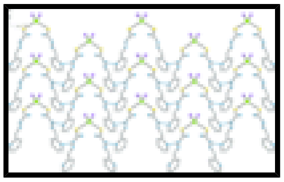

Fig. 3. A diagram showing crystal structure (view along the *c* axis).

Diiodidobis[(*E*)-1-(2-pyridylmethylene)thiosemicarbazone-κ*S*]mercury(II)

Crystal data

$[HgI_2(C_7H_8N_4S)_2]$

$M_r = 814.86$

Monoclinic, $C2/c$

Hall symbol: -C2yc

$a = 18.973$ (5) Å

$b = 7.0494$ (17) Å

$c = 17.454$ (4) Å

$\beta = 103.137$ (3)°

$V = 2273.4$ (9) Å^3

$Z = 4$

$F_{000} = 1496$

$D_x = 2.381$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 3233 reflections

$\theta = 2.4$–27.8°

$\mu = 9.69$ mm^{-1}

$T = 298$ (2) K

Block, yellow

0.30 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 1997)

$T_{min} = 0.159$, $T_{max} = 0.444$

5534 measured reflections

2048 independent reflections

1888 reflections with $I > 2\sigma(I)$

$R_{int} = 0.030$

$\theta_{max} = 25.3$°

$\theta_{min} = 2.9$°

$h = -22 \rightarrow 22$

$k = -8 \rightarrow 8$

$l = -16 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.110$

$S = 1.06$

2048 reflections

124 parameters

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 22.4472P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 2.96$ e Å^{-3}

$\Delta\rho_{min} = -2.79$ e Å^{-3}

Extinction correction: SHELXTL (Bruker, 2001), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00154 (15)
Secondary atom site location: difference Fourier map	

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	1.0000	1.20675 (6)	0.2500	0.0413 (2)
N1	0.8487 (3)	0.6917 (8)	0.2868 (4)	0.0335 (13)
H1A	0.8223	0.6719	0.2405	0.040*
N2	0.8486 (3)	0.5658 (9)	0.3465 (3)	0.0327 (13)
N3	0.9329 (4)	0.8690 (11)	0.3721 (4)	0.0516 (19)
H3A	0.9334	0.7850	0.4079	0.062*
H3B	0.9600	0.9678	0.3822	0.062*
N4	0.7574 (3)	0.1331 (9)	0.3603 (4)	0.0353 (14)
C1	0.7530 (5)	−0.0075 (13)	0.4107 (5)	0.049 (2)
H1	0.7198	−0.1046	0.3947	0.058*
C2	0.7963 (5)	−0.0124 (14)	0.4856 (5)	0.056 (2)
H2	0.7919	−0.1119	0.5192	0.067*
C3	0.8452 (5)	0.1268 (15)	0.5106 (5)	0.053 (2)
H3	0.8743	0.1246	0.5613	0.064*
C4	0.8511 (5)	0.2720 (12)	0.4596 (5)	0.0402 (18)
H4	0.8844	0.3693	0.4751	0.048*
C5	0.8067 (4)	0.2707 (10)	0.3849 (4)	0.0295 (14)
C6	0.8098 (4)	0.4184 (10)	0.3269 (4)	0.0315 (15)
H6	0.7832	0.4048	0.2754	0.038*
C7	0.8907 (4)	0.8463 (11)	0.3019 (4)	0.0348 (16)
S1	0.88321 (11)	1.0065 (3)	0.22652 (11)	0.0399 (5)
I1	0.96424 (3)	1.36465 (12)	0.10320 (4)	0.0687 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.0375 (3)	0.0448 (3)	0.0401 (3)	0.000	0.00548 (18)	0.000
N1	0.038 (3)	0.035 (3)	0.024 (3)	−0.007 (3)	−0.001 (3)	0.006 (2)
N2	0.033 (3)	0.037 (3)	0.024 (3)	−0.003 (3)	−0.002 (2)	0.003 (3)
N3	0.057 (4)	0.049 (4)	0.038 (4)	−0.021 (3)	−0.012 (3)	0.007 (3)

N4	0.036 (3)	0.040 (3)	0.028 (3)	−0.005 (3)	0.002 (3)	0.006 (3)
C1	0.049 (5)	0.049 (5)	0.045 (5)	−0.013 (4)	0.005 (4)	0.009 (4)
C2	0.069 (6)	0.059 (5)	0.039 (5)	−0.008 (5)	0.009 (4)	0.026 (4)
C3	0.060 (5)	0.068 (6)	0.026 (4)	−0.007 (5)	−0.002 (4)	0.014 (4)
C4	0.046 (4)	0.048 (4)	0.024 (4)	−0.009 (4)	0.000 (3)	0.003 (3)
C5	0.031 (4)	0.033 (4)	0.024 (3)	0.000 (3)	0.006 (3)	0.003 (3)
C6	0.032 (4)	0.040 (4)	0.021 (3)	−0.001 (3)	0.003 (3)	0.002 (3)
C7	0.031 (4)	0.045 (4)	0.027 (4)	−0.002 (3)	0.002 (3)	0.000 (3)
S1	0.0424 (10)	0.0455 (11)	0.0285 (9)	−0.0139 (8)	0.0011 (8)	0.0061 (8)
I1	0.0479 (4)	0.0830 (5)	0.0693 (5)	−0.0013 (3)	0.0009 (3)	0.0441 (4)

Geometric parameters (Å, °)

Hg1—S1	2.5803 (19)	N4—C5	1.348 (10)
Hg1—S1^{i}	2.5803 (19)	C1—C2	1.377 (13)
Hg1—I1^{i}	2.7340 (8)	C1—H1	0.9300
Hg1—I1	2.7340 (8)	C2—C3	1.353 (14)
N1—C7	1.340 (10)	C2—H2	0.9300
N1—N2	1.369 (8)	C3—C4	1.378 (12)
N1—H1A	0.8600	C3—H3	0.9300
N2—C6	1.275 (9)	C4—C5	1.382 (11)
N3—C7	1.312 (10)	C4—H4	0.9300
N3—H3A	0.8600	C5—C6	1.462 (10)
N3—H3B	0.8600	C6—H6	0.9300
N4—C1	1.341 (11)	C7—S1	1.715 (8)
S1—Hg1—S1^{i}	113.68 (10)	C3—C2—H2	119.8
S1—Hg1—I1^{i}	113.13 (5)	C1—C2—H2	119.8
S1^{i}—Hg1—I1^{i}	93.02 (4)	C2—C3—C4	118.7 (8)
S1—Hg1—I1	93.02 (4)	C2—C3—H3	120.6
S1^{i}—Hg1—I1	113.13 (5)	C4—C3—H3	120.6
I1^{i}—Hg1—I1	131.95 (4)	C3—C4—C5	118.9 (8)
C7—N1—N2	118.8 (6)	C3—C4—H4	120.6
C7—N1—H1A	120.6	C5—C4—H4	120.6
N2—N1—H1A	120.6	N4—C5—C4	122.4 (7)
C6—N2—N1	115.1 (6)	N4—C5—C6	115.2 (6)
C7—N3—H3A	120.0	C4—C5—C6	122.4 (7)
C7—N3—H3B	120.0	N2—C6—C5	120.4 (6)
H3A—N3—H3B	120.0	N2—C6—H6	119.8
C1—N4—C5	117.7 (7)	C5—C6—H6	119.8
N4—C1—C2	121.9 (8)	N3—C7—N1	119.4 (7)
N4—C1—H1	119.0	N3—C7—S1	124.3 (6)
C2—C1—H1	119.0	N1—C7—S1	116.3 (5)
C3—C2—C1	120.4 (8)	C7—S1—Hg1	108.7 (3)
C7—N1—N2—C6	−177.2 (7)	N4—C5—C6—N2	172.4 (7)
C5—N4—C1—C2	0.7 (13)	C4—C5—C6—N2	−7.7 (11)
N4—C1—C2—C3	0.0 (16)	N2—N1—C7—N3	3.0 (11)
C1—C2—C3—C4	−0.5 (16)	N2—N1—C7—S1	−175.4 (5)

C2—C3—C4—C5	0.3 (15)	N3—C7—S1—Hg1	24.2 (8)
C1—N4—C5—C4	−0.9 (11)	N1—C7—S1—Hg1	−157.5 (5)
C1—N4—C5—C6	179.1 (7)	S1^{i}—Hg1—S1—C7	52.4 (3)
C3—C4—C5—N4	0.4 (13)	I1^{i}—Hg1—S1—C7	−52.0 (3)
C3—C4—C5—C6	−179.5 (8)	I1—Hg1—S1—C7	169.3 (3)
N1—N2—C6—C5	178.3 (6)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3B···I1^{i}	0.86	3.13	3.978 (7)	169
N1—H1A···N4^{ii}	0.86	2.06	2.910 (9)	169

Symmetry codes: (i) −*x*+2, *y*, −*z*+1/2; (ii) −*x*+3/2, *y*+1/2, −*z*+1/2.

Fig. 1

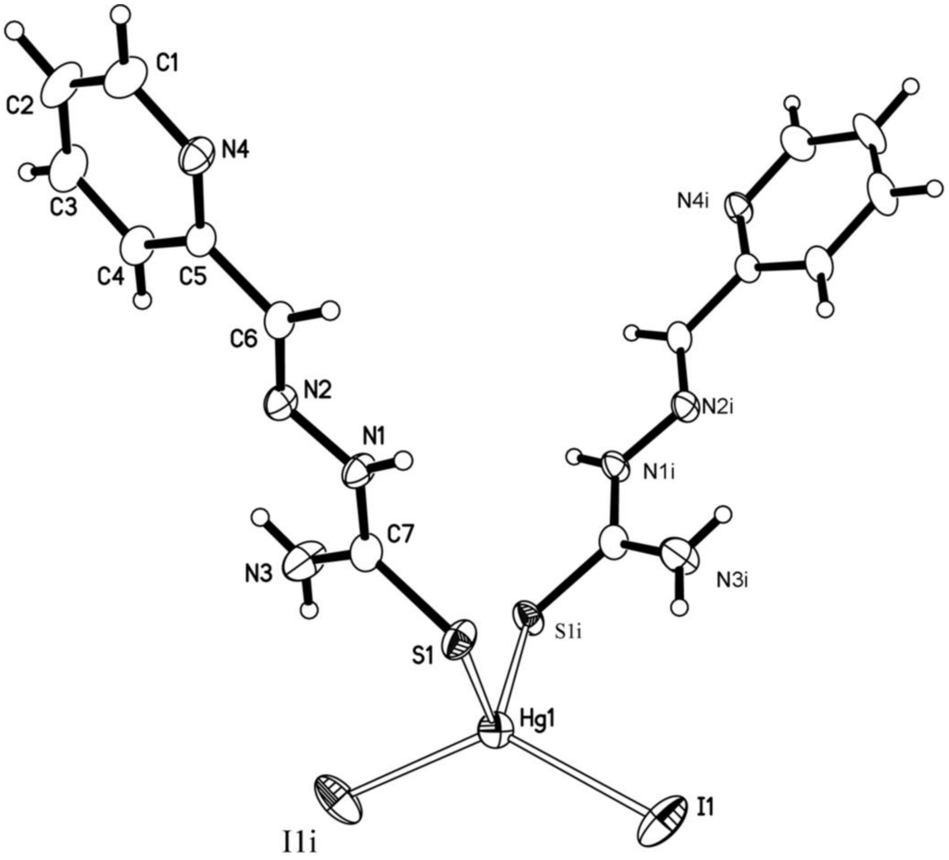

Fig. 2

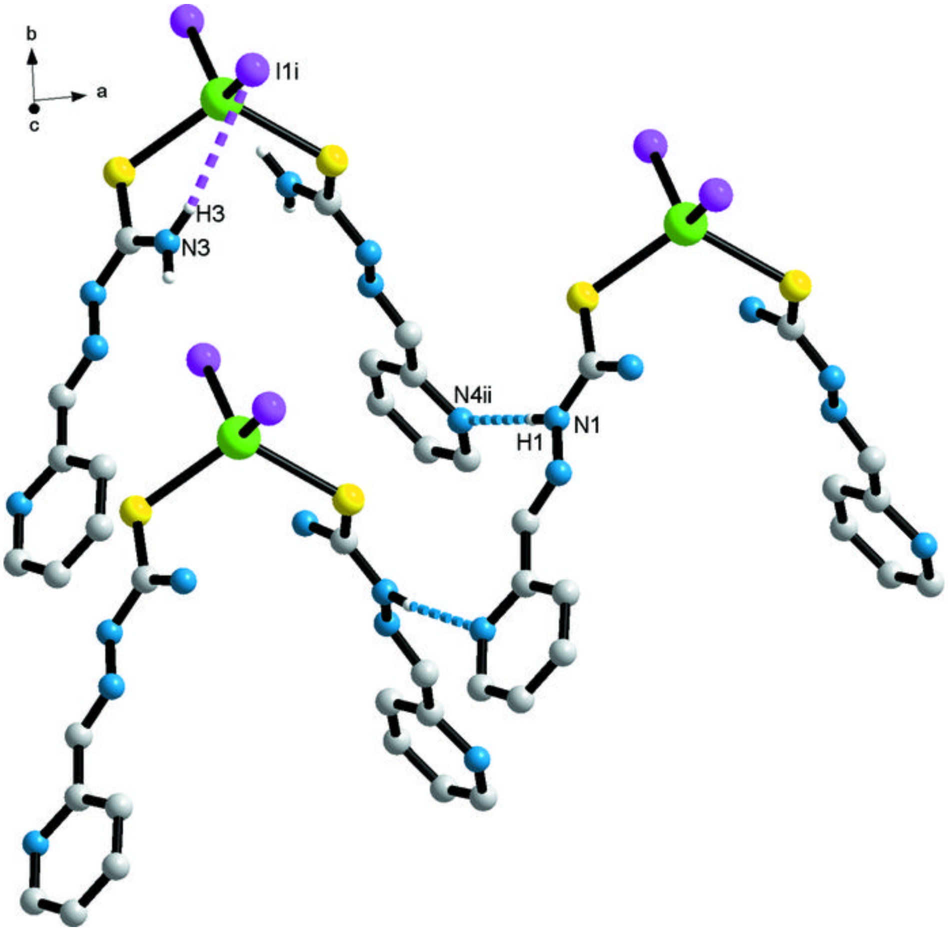

Fig. 3