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(4,5-Diazafluoren-9-one-κ^2N,N')bis(thiocyanato-κS)mercury(II)

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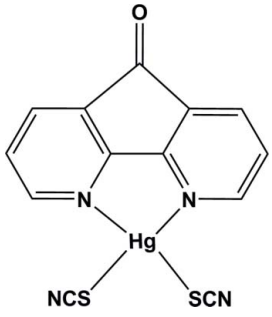
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.048; wR factor = 0.118; data-to-parameter ratio = 19.6.

In the title compound, $[Hg(NCS)_2(C_{11}H_6N_2O)]$, the Hg^{II} atom, lying on a twofold rotation axis, is four-coordinated in a distorted tetrahedral geometry by an N,N'-bidentate diazafluoren-9-one ligand and two thiocyanate anions. In the crystal, intermolecular C—H···N and C—H···O hydrogen bonds are effective in the stabilization of the structure.

Related literature

For general background to metal complexes with diazafluoren-9-one ligands, see: Biju & Rajasekharan (2008); Kulkarni *et al.* (2002); Menon & Rajasekharan (1998); Shi *et al.* (1995); Wu & Xu (2004); Zhang *et al.* (2004). For related structures, see: Ravikumar & Lakshmi (1994); Safari *et al.* (2009). For the synthesis of the ligand, see: Henderson *et al.* (1984).



Experimental

Crystal data

$[Hg(NCS)_2(C_{11}H_6N_2O)]$
M_r = 498.95
Monoclinic, $C2/c$
a = 10.570 (2) Å
b = 16.112 (3) Å
c = 8.3390 (17) Å
β = 94.35 (3)°
V = 1416.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 11.17 mm^{-1}
T = 298 K
0.45 × 0.30 × 0.25 mm

Data collection

Stoe IPDS-2T diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)
T_{min} = 0.023, T_{max} = 0.059
4787 measured reflections
1903 independent reflections
1737 reflections with $I > 2\sigma(I)$
R_{int} = 0.098

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.118
S = 1.09
1903 reflections
97 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 4.61 e Å^{-3}
$\Delta\rho_{min}$ = −1.82 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hg1—S1	2.4098 (17)	Hg1—N1	2.483 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···N2^{i}	0.93	2.56	3.276 (10)	134
C4—H4···O1^{ii}	0.93	2.59	3.366 (8)	142

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (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: HY2412).

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supporting information

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(4,5-Diazafluoren-9-one-κ^2N,N')bis(thiocyanato-κS)mercury(II)

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S1. Comment

Henderson *et al.* (1984) first reported the synthesis of diazafluoren-9-one (dafone) and Ravikumar & Lakshmi (1994) determined the structure of this compound. Dafone is a bidentate ligand and numerous complexes with dafone have been prepared, such as that of cobalt (Shi *et al.*, 1995), copper (Kulkarni *et al.*, 2002; Menon & Rajasekharan, 1998), zinc (Zhang *et al.*, 2004), manganese (Wu & Xu, 2004) and silver (Biju & Rajasekharan, 2008). For further investigation of the dafone complexes, we synthesized the title compound.

The asymmetric unit of the title compound (Fig. 1) contains a half molecule. The Hg^{II} atom, lying on a twofold rotation axis, is four-coordinated in a distorted tetrahedral geometry by an N,N′-bidentate dafone ligand and two thiocyanate anions. The Hg—N and Hg—S bond lengths (Table 1) and angles are within normal range as observed in $[Hg(SCN)_2(dm4bt)]$ (dm4bt = 2,2′-dimethyl-4,4′-bi-1,3-thiazole) (Safari *et al.*, 2009). In the crystal, intermolecular C—H···O and C—H···N hydrogen bonds stabilize the structure (Table 2, Fig. 2).

S2. Experimental

For the preparation of the title compound, a solution of dafone (0.13 g, 0.71 mmol) in methanol (20 ml) was added to a solution of $Hg(SCN)_2$ (0.23 g, 0.71 mmol) in methanol (20 ml) at room temperature and the yellow powder was formed. Crystals suitable for X-ray diffraction were obtained by methanol vapor diffusion into a DMSO solution of the complex. The crystals were isolated after one week (yield: 0.28 g, 79%).

S3. Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The highest residual electron density was found at 0.90 Å from Hg1 atom and the deepest hole at 0.84 Å from Hg1 atom.

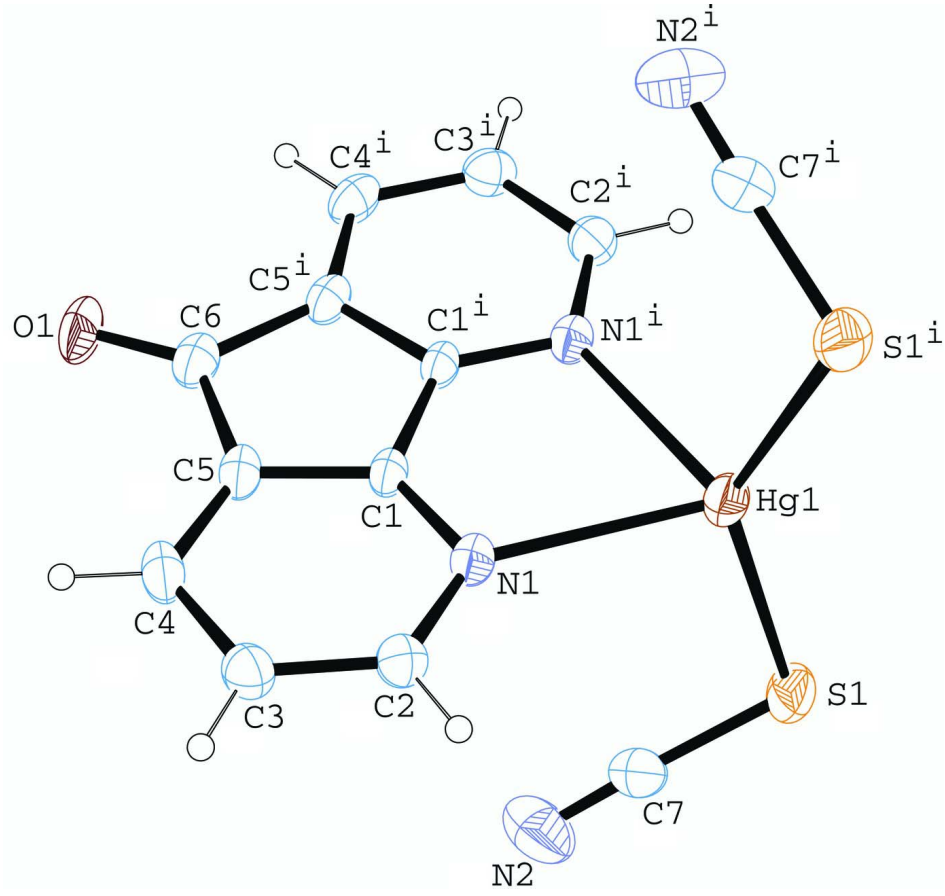

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (i) -*x*+1, *y*, -*z*+1/2.]

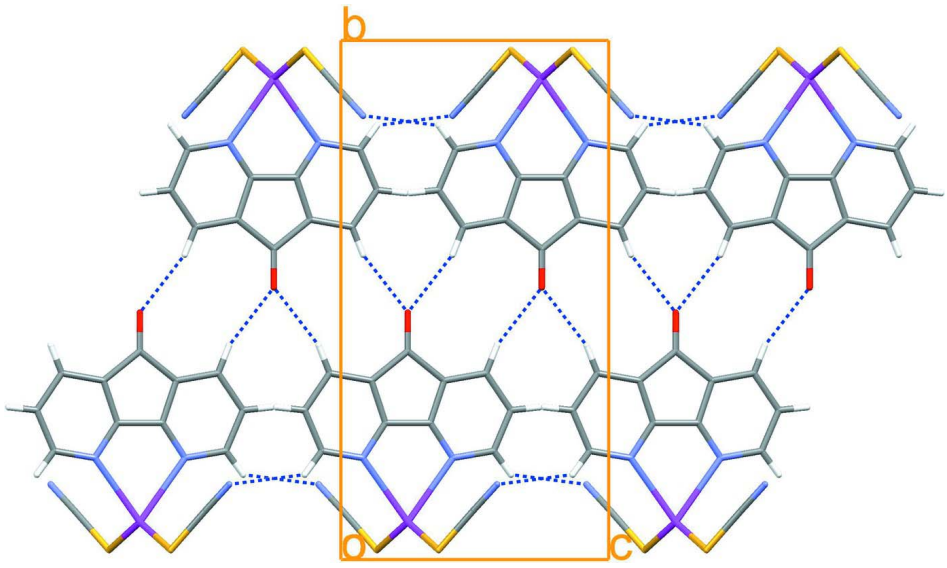

Figure 2

The packing diagram of the title compound. Intermolecular C—H···O and C—H···N hydrogen bonds are shown as blue dashed lines.

(4,5-Diazafluoren-9-one-κ^2N,N')bis(thiocyanato- κS)mercury(II)

Crystal data

$[Hg(NCS)_2(C_{11}H_6N_2O)]$
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Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 10.570$ (2) Å
$b = 16.112$ (3) Å
$c = 8.3390$ (17) Å
$\beta = 94.35$ (3)°
$V = 1416.1$ (5) Å^3
$Z = 4$
$F(000) = 928$
$D_x = 2.340$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1903 reflections
$\theta = 2.3$–29.1°
$\mu = 11.17$ mm^{-1}
$T = 298$ K
Block, yellow
0.45 × 0.30 × 0.25 mm

Data collection

Stoe IPDS-2T diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)
$T_{min} = 0.023$, $T_{max} = 0.059$
4787 measured reflections
1903 independent reflections
1737 reflections with $I > 2\sigma(I)$
$R_{int} = 0.098$
$\theta_{max} = 29.1°$, $\theta_{min} = 2.3°$
$h = -14\rightarrow14$
$k = -19\rightarrow22$
$l = -11\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.118$
$S = 1.09$
1903 reflections
97 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
$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 4.61$ e Å^{-3}
$\Delta\rho_{min} = -1.82$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C7	0.7118 (6)	0.0950 (5)	0.0048 (8)	0.0493 (13)
Hg1	0.5000	0.072315 (18)	0.2500	0.04567 (15)
N1	0.4449 (4)	0.1960 (3)	0.0815 (6)	0.0365 (9)
C1	0.4721 (4)	0.2635 (3)	0.1663 (6)	0.0320 (9)
C2	0.3956 (5)	0.2094 (4)	−0.0707 (7)	0.0422 (11)
H2	0.3735	0.1637	−0.1350	0.051*
C5	0.4558 (5)	0.3449 (3)	0.1142 (7)	0.0377 (10)
C3	0.3766 (6)	0.2879 (4)	−0.1349 (7)	0.0453 (12)
H3	0.3438	0.2937	−0.2409	0.054*
C4	0.4063 (5)	0.3591 (4)	−0.0419 (8)	0.0456 (12)
H4	0.3935	0.4124	−0.0827	0.055*
S1	0.68555 (17)	0.01847 (11)	0.1348 (2)	0.0576 (4)
O1	0.5000	0.4758 (4)	0.2500	0.0586 (17)
C6	0.5000	0.4016 (6)	0.2500	0.0432 (17)
N2	0.7356 (8)	0.1448 (6)	−0.0831 (10)	0.083 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.049 (3)	0.058 (4)	0.041 (3)	0.004 (3)	0.005 (2)	−0.002 (3)
Hg1	0.0541 (2)	0.03049 (19)	0.0543 (2)	0.000	0.01618 (14)	0.000
N1	0.0374 (19)	0.029 (2)	0.043 (2)	0.0004 (16)	0.0032 (16)	0.0006 (17)
C1	0.0334 (19)	0.022 (2)	0.040 (3)	0.0006 (16)	0.0044 (17)	0.0012 (16)
C2	0.046 (3)	0.040 (3)	0.040 (3)	0.004 (2)	0.002 (2)	0.001 (2)
C5	0.035 (2)	0.028 (2)	0.050 (3)	0.0014 (18)	0.0046 (19)	0.005 (2)
C3	0.046 (3)	0.047 (3)	0.043 (3)	0.007 (2)	0.005 (2)	0.007 (2)
C4	0.046 (3)	0.035 (3)	0.056 (3)	0.005 (2)	0.006 (2)	0.015 (2)
S1	0.0623 (9)	0.0412 (8)	0.0715 (11)	0.0171 (7)	0.0201 (8)	0.0072 (7)
O1	0.072 (4)	0.023 (3)	0.081 (5)	0.000	0.007 (4)	0.000
C6	0.038 (3)	0.033 (4)	0.060 (5)	0.000	0.011 (3)	0.000
N2	0.077 (4)	0.104 (7)	0.070 (5)	0.003 (4)	0.021 (3)	0.025 (4)

Geometric parameters (Å, º)

C7—N2	1.128 (11)	C2—H2	0.9300
C7—S1	1.679 (8)	C5—C4	1.385 (8)
Hg1—S1	2.4098 (17)	C5—C6	1.502 (8)
Hg1—N1	2.483 (5)	C3—C4	1.407 (9)
N1—C1	1.316 (7)	C3—H3	0.9300
N1—C2	1.352 (7)	C4—H4	0.9300
C1—C5	1.388 (6)	O1—C6	1.195 (12)

C1—C1^{i}	1.473 (10)	C6—C5^{i}	1.502 (8)
C2—C3	1.382 (8)		
N2—C7—S1	176.4 (8)	C3—C2—H2	118.5
S1^{i}—Hg1—S1	137.80 (9)	C4—C5—C1	118.7 (5)
S1^{i}—Hg1—N1^{i}	103.08 (11)	C4—C5—C6	132.9 (5)
S1—Hg1—N1^{i}	110.60 (12)	C1—C5—C6	108.4 (5)
S1^{i}—Hg1—N1	110.60 (12)	C2—C3—C4	120.9 (5)
S1—Hg1—N1	103.08 (11)	C2—C3—H3	119.6
N1^{i}—Hg1—N1	73.2 (2)	C4—C3—H3	119.6
C1—N1—C2	115.2 (5)	C5—C4—C3	115.8 (5)
C1—N1—Hg1	109.0 (3)	C5—C4—H4	122.1
C2—N1—Hg1	135.7 (4)	C3—C4—H4	122.1
N1—C1—C5	126.5 (5)	C7—S1—Hg1	99.9 (2)
N1—C1—C1^{i}	124.4 (3)	O1—C6—C5^{i}	127.5 (4)
C5—C1—C1^{i}	109.1 (3)	O1—C6—C5	127.5 (4)
N1—C2—C3	122.9 (6)	C5^{i}—C6—C5	105.0 (7)
N1—C2—H2	118.5		
S1^{i}—Hg1—N1—C1	97.7 (3)	N1—C1—C5—C6	−179.7 (4)
S1—Hg1—N1—C1	−108.1 (3)	C1^{i}—C1—C5—C6	−0.8 (6)
N1^{i}—Hg1—N1—C1	−0.3 (2)	N1—C2—C3—C4	−1.1 (9)
S1^{i}—Hg1—N1—C2	−82.2 (5)	C1—C5—C4—C3	0.0 (8)
S1—Hg1—N1—C2	71.9 (5)	C6—C5—C4—C3	179.5 (5)
N1^{i}—Hg1—N1—C2	179.8 (6)	C2—C3—C4—C5	0.6 (9)
C2—N1—C1—C5	−0.5 (8)	S1^{i}—Hg1—S1—C7	150.1 (3)
Hg1—N1—C1—C5	179.5 (4)	N1^{i}—Hg1—S1—C7	−69.2 (3)
C2—N1—C1—C1^{i}	−179.2 (6)	N1—Hg1—S1—C7	7.5 (3)
Hg1—N1—C1—C1^{i}	0.8 (7)	C4—C5—C6—O1	0.7 (7)
C1—N1—C2—C3	1.1 (8)	C1—C5—C6—O1	−179.7 (3)
Hg1—N1—C2—C3	−179.0 (4)	C4—C5—C6—C5^{i}	−179.3 (7)
N1—C1—C5—C4	0.0 (8)	C1—C5—C6—C5^{i}	0.3 (3)
C1^{i}—C1—C5—C4	178.9 (5)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···N2^{ii}	0.93	2.56	3.276 (10)	134
C4—H4···O1^{iii}	0.93	2.59	3.366 (8)	142

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