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

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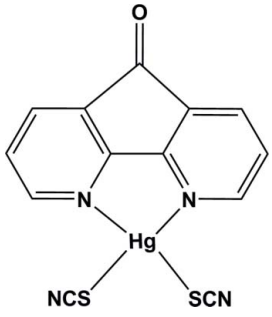
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.048; wR factor = 0.118; data-to-parameter ratio = 19.6.

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).

References

Biju, A. R. & Rajasekharan, M. V. (2008). *Polyhedron*, **27**, 2065–2068.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Henderson, L. J. Jr, Fronczek, F. R. & Cherry, W. R. (1984). *J. Am. Chem. Soc.* **106**, 5876–5879.
Kulkarni, P., Padhye, S., Sinn, E., Anson, C. E. & Powell, A. K. (2002). *Inorg. Chim. Acta*, **332**, 167–175.
Menon, S. & Rajasekharan, M. V. (1998). *Polyhedron*, **17**, 2463–2476.
Ravikumar, K. & Lakshmi, N. V. (1994). *Z. Kristallogr.* **209**, 56–57.
Safari, N., Amani, V., Abedi, A., Notash, B. & Ng, S. W. (2009). *Acta Cryst.* E**65**, m372.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shi, X.-H., You, X.-Z., Li, C. & Xiong, R.-G. (1995). *Transition Met. Chem.* **20**, 191–195.
Stoe & Cie (2005). *X-AREA*, *X-SHAPE* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.
Wu, Z.-Y. & Xu, D.-J. (2004). *Acta Cryst.* E**60**, m839–m841.
Zhang, R.-L., Zhao, J.-S., Yang, S.-Y. & Ng, S. W. (2004). *Acta Cryst.* E**60**, m262–m263.

supplementary materials

Acta Cryst. (2011). E**67**, m418 [**doi:10.1107/S1600536811008142**]

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

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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).

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%).

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.

Figures

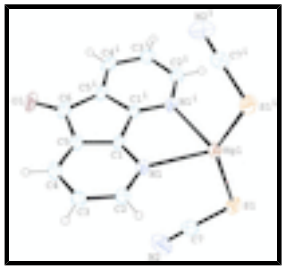

Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (i) -*x*+1, *y*, -*z*+1/2.]

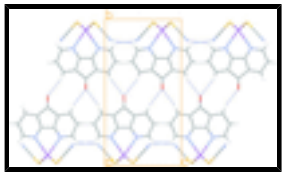

Fig. 2. The packing diagram of the title compound. Intermolecular C—H···O and C—H···N hydrogen bonds are shown as blue dashed lines.

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

Crystal data

$[Hg(NCS)_2(C_{11}H_6N_2O)]$	$F(000) = 928$
$M_r = 498.95$	$D_x = 2.340$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 1903 reflections
a = 10.570 (2) Å	θ = 2.3–29.1°
b = 16.112 (3) Å	μ = 11.17 mm^{-1}
c = 8.3390 (17) Å	*T* = 298 K
β = 94.35 (3)°	Block, yellow
V = 1416.1 (5) Å^3	0.45 × 0.30 × 0.25 mm
Z = 4	

Data collection

Stoe IPDS-2T diffractometer	1903 independent reflections
Radiation source: fine-focus sealed tube	1737 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.098$
ω scans	$\theta_{max} = 29.1°$, $\theta_{min} = 2.3°$
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2005)	$h = -14 \rightarrow 14$
$T_{min} = 0.023$, $T_{max} = 0.059$	$k = -19 \rightarrow 22$
4787 measured reflections	$l = -11 \rightarrow 9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.118$	H-atom parameters constrained
$S = 1.09$	$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
1903 reflections	$(\Delta/\sigma)_{max} < 0.001$
97 parameters	$\Delta\rho_{max} = 4.61$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -1.82$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	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 (Å^2)

	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, °)

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 codes: (i) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

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*.

Fig. 1

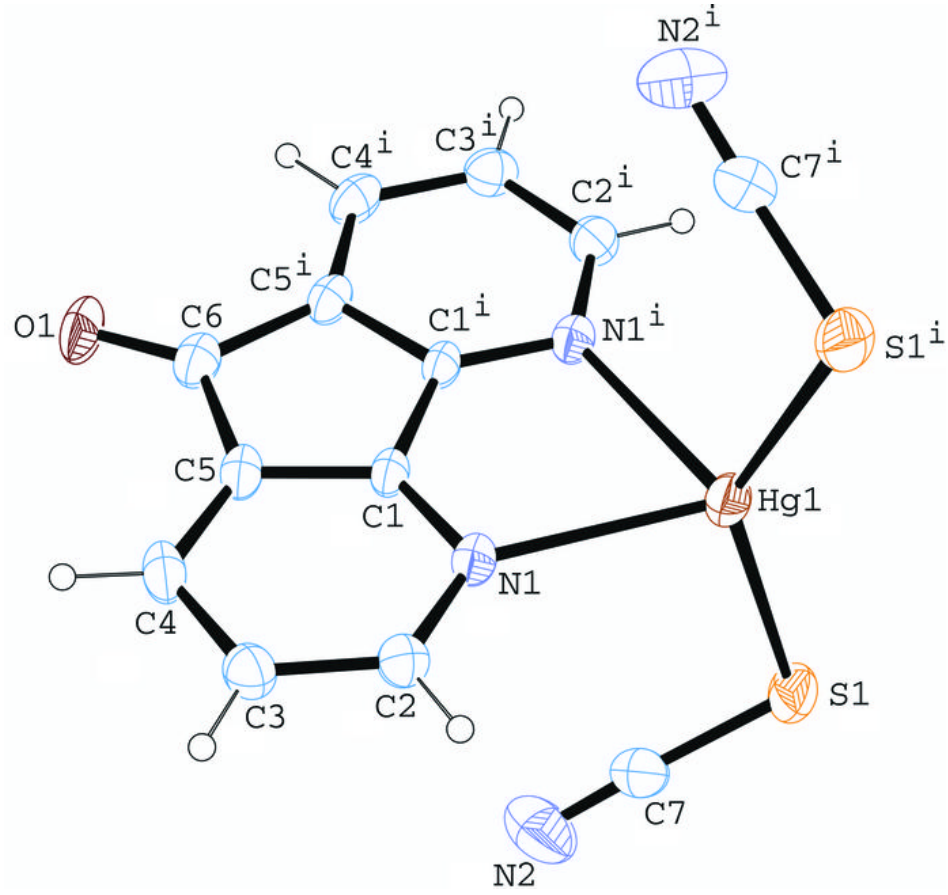
N2i
C7i
C4i
C3i
C2i
S1i
C5i
O1
C6
C1i
N1i
C5
C1
Hg1
N1
C4
S1
C3
C2
C7
N2

Fig. 2