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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.017$ Å
 R factor = 0.039
 wR factor = 0.080
Data-to-parameter ratio = 19.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Poly[[μ -aqua-tetra- μ -thiocyanato-cadmium(II)-
mercury(II)] sulfolane solvate]

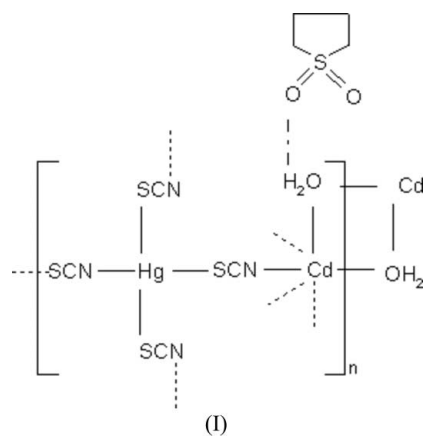
The title complex, $\{[\text{CdHg}(\text{SCN})_4(\text{H}_2\text{O})] \cdot \text{C}_4\text{H}_8\text{O}_2\text{S}\}_n$, comprises slightly distorted CdN_4O_2 octahedra and HgS_4 tetrahedra where the donor atoms are derived from ambidentate thiocyanate ligands and, in the case of cadmium, also from water molecules. Pairs of Cd atoms are bridged by two water molecules, forming a parallelogram, and each water molecule is hydrogen bonded to a sulfolane molecule. Pairs of Cd and Hg atoms are connected by $-\text{SCN}-$ bridges, which leads to the formation of a three-dimensional $-\text{Cd}-\text{NCS}-\text{Hg}-$ network.

Received 10 August 2006

Accepted 23 August 2006

Comment

Cadmium mercury thiocyanate (CMTC) and its Lewis-base adducts are known to exist in a variety of polymeric structures. CMTC comprises distorted CdN_4 and HgS_4 tetrahedra (Yuan *et al.*, 1997). Each of the three Lewis-base adducts of CMTC contain distorted CdN_4O_2 octahedra and HgS_4 tetrahedra (Zhou *et al.*, 2000; Guo *et al.*, 2001; Wang *et al.*, 2002). In the Ph_3P adduct (Wang *et al.*, 2005), the Cd and Hg atoms exist within a distorted N_4S trigonal-bipyramidal and a distorted S_2P_2 tetrahedral geometry, respectively. In the present work, the sulfolane adduct of CMTC, (I), has been characterized.



In (I), the Cd^{II} and Hg^{II} atoms are within a distorted N_4O_2 octahedral and S_4 tetrahedral geometry, respectively (Fig. 1). The $\text{Cd}-\text{N}$ and $\text{Cd}-\text{O}$ bond lengths are shorter and longer than the sum of Shannon's ionic radii (2.41 and 2.30 Å, respectively; Shannon, 1976). From the bond-angle data (Table 1), there are significant distortions from the ideal octahedral geometry. The $\text{Hg}-\text{S}$ bond lengths are a little longer than the sum of the single-bond covalent radii (2.52 Å; Pauling, 1960), and the $\text{S}-\text{Hg}-\text{S}$ angles deviate from the ideal tetrahedral angle. A parallelogram is formed by two Cd atoms and two bridging water molecules. The sulfolane mol-

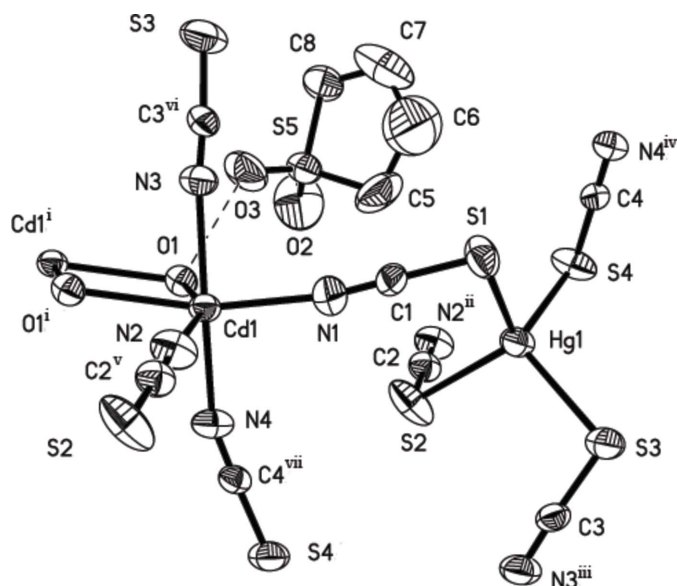


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. The dashed line indicates a hydrogen bond. [Symmetry codes: (i) $2 - x, -y, 1 - z$; (ii) $x - 1, y, z$; (iii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (iv) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; (v) $1 + x, y, z$; (vi) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (vii) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$]

ecule is not directly coordinated to a metal atom, rather it is hydrogen bonded to a coordinated water molecule; see Table 2 for parameters. The C—N—Cd and C—S—Hg angles (close to 180 and 90°, respectively) show that the C—N—Cd and C—S—Hg groups are quasilinear and significantly bent, respectively. The —SCN— bridges connect the Cd and Hg atoms, forming an infinite three-dimensional network.

Experimental

To a crystalline powder of CMTC (0.632 g) was added a mixed solvent comprising water (10 ml) and sulfolane (3 ml). The mixture was heated and stirred until CMTC dissolved at about 333 K. The colourless solution was allowed to stand at about 313 K. After several hours, transparent crystals of (I) suitable for X-ray structure analysis were obtained. Analysis calculated for $C_8H_{10}CdHgN_4O_3S_5$: C 14.06, H 1.47, N 8.20%; found: C 14.18, H 1.53, N 8.15%.

Crystal data

$[CdHg(NCS)_4(H_2O)] \cdot C_4H_8O_2S$
 $M_r = 683.49$
 Monoclinic, $P2_1/n$
 $a = 8.7219$ (7) Å
 $b = 17.3525$ (19) Å
 $c = 12.2719$ (14) Å
 $\beta = 92.457$ (9)°
 $V = 1855.6$ (3) Å³
 $Z = 4$
 $D_x = 2.447$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 9.98$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.12 \times 0.09 \times 0.08$ mm

Data collection

Bruker P4 diffractometer
 ω scans
 Absorption correction: ψ scan
 (XSCANS; Bruker,1996)
 $T_{min} = 0.296, T_{max} = 0.449$
 4929 measured reflections
 3843 independent reflections
 2711 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.026$
 $\theta_{max} = 26.5^\circ$
 3 standard reflections
 every 97 reflections
 intensity decay: none

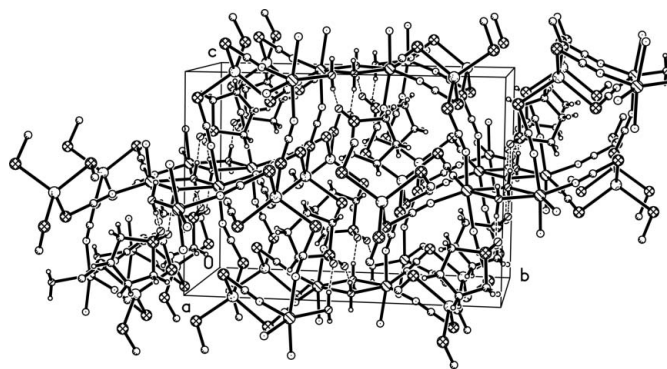


Figure 2
Packing diagram for (I), showing the three-dimensional network. Dashed lines indicate hydrogen bonds.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.080$
 $S = 1.01$
 3843 reflections
 201 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0311P)^2 + 0.2895P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 1.46$ e Å⁻³
 $\Delta\rho_{min} = -1.09$ e Å⁻³
 Extinction correction: SHELXTL
 Extinction coefficient: 0.00413 (15)

Table 1

Selected geometric parameters (Å, °).

Hg1—S1	2.547 (2)	Cd1—O1	2.390 (5)
Hg1—S2	2.537 (2)	Cd1—O1 ⁱ	2.470 (5)
Hg1—S3	2.543 (3)	Cd1—N2	2.248 (8)
Hg1—S4	2.555 (2)	Cd1—N3	2.282 (7)
Cd1—N1	2.248 (8)	Cd1—N4	2.273 (6)
S1—Hg1—S2	112.00 (9)	N1—Cd1—N4	93.0 (3)
S1—Hg1—S3	111.41 (10)	N2—Cd1—N3	101.7 (3)
S1—Hg1—S4	105.94 (8)	N2—Cd1—N4	92.9 (2)
S2—Hg1—S3	106.22 (9)	N3—Cd1—N4	165.1 (3)
S2—Hg1—S4	110.56 (8)	Hg1—S1—C1	95.1 (3)
S3—Hg1—S4	110.79 (10)	Hg1—S2—C2	93.9 (3)
O1—Cd1—O1 ⁱ	73.28 (19)	Hg1—S3—C3	103.2 (3)
O1—Cd1—N1	103.4 (3)	Hg1—S4—C4	97.3 (3)
O1—Cd1—N2	155.1 (2)	Cd1—N1—C1	170.0 (7)
O1—Cd1—N3	83.1 (2)	Cd1—N2—C2 ⁱⁱ	173.9 (7)
O1—Cd1—N4	82.4 (2)	Cd1—N3—C3 ⁱⁱⁱ	156.6 (8)
O1 ⁱ —Cd1—N1	166.8 (2)	Cd1—N4—C4 ^{iv}	161.7 (7)
O1 ⁱ —Cd1—N2	83.5 (2)	Cd1—O1—Cd1 ⁱ	106.72 (19)
O1 ⁱ —Cd1—N3	80.2 (2)	S1—C1—N1	179.3 (9)
O1 ⁱ —Cd1—N4	99.1 (2)	S2—C2—N2 ^v	179.0 (8)
N1—Cd1—N2	101.2 (3)	S3—C3—N3 ^{vi}	176.0 (8)
N1—Cd1—N3	86.8 (3)	S4—C4—N4 ^{vii}	178.0 (8)

Cd1ⁱ—O1—Cd1—O1ⁱ 0.0

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

Table 2

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1A...O2 ^{viii}	0.97	1.77	2.727 (9)	170
O1—H1B...O3	0.97	1.72	2.680 (8)	170

Symmetry code: (viii) $-x + 1, -y, -z + 1$.

All H atoms of the sulfolane solvent molecule were placed in geometrically calculated positions and refined using a riding model, with C–H = 0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The two water H atoms were located in a difference Fourier map and refined independently with isotropic displacement parameters. The highest peak and deepest hole were located 0.79 and 0.42 Å, respectively, from atom S3.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by the grants (Nos. 60476020 and 50323006) of the National Natural Science Foundation of China.

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