

Diiodidobis(thioacetamide- κ S)-cadmium(II)

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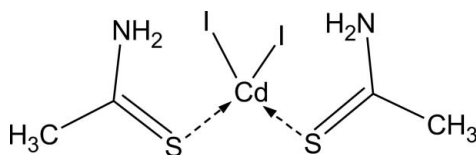
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.029; wR factor = 0.055; data-to-parameter ratio = 26.8.

Thioacetamide complexes are used for the synthesis of metal sulfide powders. In the title molecular complex, $[\text{CdI}_2(\text{C}_2\text{H}_5\text{NS})_2]$, the Cd^{II} atom is located on a twofold rotation axis and is tetrahedrally coordinated by two iodine atoms and two thioacetamide molecules. The $\text{Cd}-\text{I}$ distance is 2.7615 (17) Å and the $\text{Cd}-\text{S}$ distance is 2.5586 (17) Å.

Related literature

For the synthesis of metal sulfide powders, see: Trindade *et al.* (1994); Grau & Akinc (1997). For the structures of Zn, Cd and Hg halide complexes with thioacetamide, see: Rolies & De Ranter (1977*a,b*, 1978).



Experimental

Crystal data

$[\text{CdI}_2(\text{C}_2\text{H}_5\text{NS})_2]$
 $M_r = 516.49$
 Orthorhombic, $Pbcn$
 $a = 10.369$ (9) Å
 $b = 9.809$ (6) Å
 $c = 12.792$ (8) Å

$V = 1301.1$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 6.70$ mm⁻¹
 $T = 293$ (2) K
 $0.4 \times 0.4 \times 0.4$ mm

Data collection

Enraf–Nonius CAD4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\text{min}} = 0.069$, $T_{\text{max}} = 0.086$
 (expected range = 0.055–0.069)

1418 measured reflections
 1418 independent reflections
 1115 reflections with $I > 2\sigma(I)$
 1 standard reflections
 frequency: 120 min
 intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.055$
 $S = 1.03$
 1418 reflections

53 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.49$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.58$ e Å⁻³

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); 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: RK2044).

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supplementary materials

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Diiodidobis(thioacetamide- κ S)cadmium(II)

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Comment

Thioacetamide complexes are used for synthesis of metal sulfide powders (Trindade *et al.*, 1994; Grau & Akinc, 1997). Several structures of metal complexes with thioacetamide are reported. Cadmium chloride complex with thioacetamide $\text{CdCl}_2 \cdot \text{CH}_3\text{CSNH}_2$ contains double chains of cadmium atoms with bridging chlorine atoms (Rolies & De Ranter, 1978). Two chains are shared through tridentate bridging chlorine atoms. Every cadmium atom is coordinated by five chlorine atoms and one sulfur atom of terminal thioacetamide molecule. In the zinc chloride complex with thioacetamide $\text{ZnCl}_2 \cdot 2\text{CH}_3\text{CSNH}_2$, zinc is tetrahedrally coordinated by two chlorine atoms and two thioacetamide molecules (Rolies & De Ranter, 1977*a*). The mercury chloride complex with thioacetamide $\text{HgCl}_2 \cdot 2\text{CH}_3\text{CSNH}_2$ is polymeric; adjacent mercury atoms linked through two bridging chlorine atoms (Rolies & De Ranter, 1977*b*). In this work, we report the crystal structure of diiodidobis(thioacetamide)cadmium $[\text{Cd}(\text{CH}_3\text{CSNH}_2)_2\text{I}_2]$. The complex is molecular, cadmium is located on a twofold axis and tetrahedrally coordinated by two chlorine atoms and two thioacetamide molecules. Cd—I distance is 2.7615 (17) Å and Cd—S1 is 2.5586 (17) Å.

Experimental

Cadmium iodide (1.0 g) and thioacetamide (0.4 g) were dissolved in water (6 ml) at room temperature. The solution was layered on a liquid perfluorinated hydrocarbon (1-methyldecahydronaphthalene). In 0.5 h colorless crystals formed and felt in 1-methyldecahydronaphthalene which served as protective environment. Yield 20%.

Refinement

H atoms were positioned geometrically and refined using a riding model (including free rotation about C—C bond), with C—H = 0.96 Å, N—H = 0.86 Å, $U_{\text{iso}}(\text{H}) = 1.2$ (for NH_2 and 1.5 (for CH_3) times U_{eq} of the parent atom.

Figures

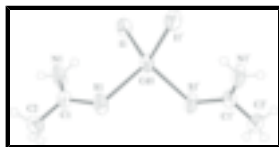


Fig. 1. ORTEP-3 (Farrugia, 1997) view of the title complex, with the atom numbering scheme. Displacement ellipsoids are presented with 50% probability level. H atoms are drawn as spheres of arbitrary radius. Symmetry code: (i) $-x + 1, y, -z + 3/2$.

Diiodidobis(thioacetamide- κ S)cadmium(II)

Crystal data

$[\text{Cd}(\text{I})_2(\text{C}_2\text{H}_5\text{NS})_2]$

$F_{000} = 936$

supplementary materials

$M_r = 516.49$

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

$a = 10.369 (9) \text{ \AA}$

$b = 9.809 (6) \text{ \AA}$

$c = 12.792 (8) \text{ \AA}$

$V = 1301.1 (16) \text{ \AA}^3$

$Z = 4$

$D_x = 2.637 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 13\text{--}14^\circ$

$\mu = 6.70 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Prism, colourless

$0.4 \times 0.4 \times 0.4 \text{ mm}$

Data collection

Enraf–Nonius CAD4
diffractometer

ω scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.069$, $T_{\max} = 0.086$

1418 measured reflections

1418 independent reflections

1115 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0$

$\theta_{\max} = 27.0^\circ$

$\theta_{\min} = 2.9^\circ$

$h = -13 \rightarrow 0$

$k = 0 \rightarrow 12$

$l = 0 \rightarrow 16$

1 standard reflections

every 120 min

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.055$

$S = 1.03$

1418 reflections

53 parameters

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0215P)^2 + 0.5736P]$$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.49 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.58 \text{ e \AA}^{-3}$

Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
II	0.71665 (3)	1.06035 (4)	0.70992 (3)	0.04723 (12)
Cd1	0.5000	0.90519 (6)	0.7500	0.03851 (14)
S1	0.44664 (13)	0.74390 (15)	0.59888 (10)	0.0504 (4)
C1	0.5480 (4)	0.7604 (5)	0.4977 (4)	0.0379 (11)
N1	0.6340 (4)	0.8524 (5)	0.4899 (3)	0.0656 (15)
H1A	0.6821	0.8560	0.4352	0.079*
H1B	0.6439	0.9110	0.5392	0.079*
C2	0.5367 (6)	0.6619 (6)	0.4086 (4)	0.0641 (17)
H2A	0.5244	0.7114	0.3446	0.096*
H2B	0.4643	0.6028	0.4201	0.096*
H2C	0.6140	0.6085	0.4039	0.096*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.03944 (19)	0.0613 (2)	0.04098 (19)	-0.01312 (17)	0.00435 (14)	-0.00932 (16)
Cd1	0.0327 (2)	0.0548 (3)	0.0280 (2)	0.000	0.0044 (2)	0.000
S1	0.0453 (7)	0.0710 (10)	0.0348 (7)	-0.0217 (7)	0.0110 (6)	-0.0112 (7)
C1	0.035 (2)	0.051 (3)	0.028 (2)	0.001 (2)	0.001 (2)	0.001 (2)
N1	0.066 (3)	0.091 (4)	0.040 (3)	-0.035 (3)	0.028 (2)	-0.020 (3)
C2	0.093 (5)	0.051 (3)	0.048 (3)	-0.007 (3)	0.016 (3)	-0.012 (3)

Geometric parameters (\AA , $^\circ$)

II—Cd1	2.7615 (17)	N1—H1A	0.8607
Cd1—S1	2.5586 (17)	N1—H1B	0.8607
S1—C1	1.675 (5)	C2—H2A	0.9600
C1—N1	1.272 (6)	C2—H2B	0.9600
C1—C2	1.499 (7)	C2—H2C	0.9600
S1—Cd1—S1 ⁱ	103.61 (9)	C1—N1—H1B	120.0
S1—Cd1—II	112.11 (4)	H1A—N1—H1B	119.9
S1—Cd1—II ⁱ	107.77 (5)	C1—C2—H2A	109.5
II—Cd1—II ⁱ	113.11 (6)	C1—C2—H2B	109.5
C1—S1—Cd1	112.85 (19)	H2A—C2—H2B	109.5
N1—C1—C2	116.9 (4)	C1—C2—H2C	109.5
N1—C1—S1	124.7 (4)	H2A—C2—H2C	109.5
C2—C1—S1	118.4 (4)	H2B—C2—H2C	109.5
C1—N1—H1A	120.0		

Symmetry codes: (i) $-x+1, y, -z+3/2$.

Fig. 1

