# Chloridodimethyl(thiosemicarbazide)-tin(IV) chloride

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In the title salt, $[Sn(CH_3)_2Cl(CH_4N_3S)]Cl$, the $Sn^{IV}$ atom is five-coordinated in a distorted trigonal-bipyramidal geometry with two methyl groups and one S atom in the equatorial plane, and one N atom and one Cl atom occupying the apical positions. In the crystal, molecules are linked by intermolecular N—H⋯S hydrogen bonds with set graph-motif $C(4)$ along [010]. N—H⋯Cl hydrogen bonds with graph-set motif $D(2)$ and $D_3^3(10)$ link cations and anions.

## Related literature

For a related structure, see: Delgado *et al.* (2009). For graph-set motifs, see: Bernstein *et al.* (1995). For the biological activity of organotin(IV) complexes, see: Davies & Smith (1982).
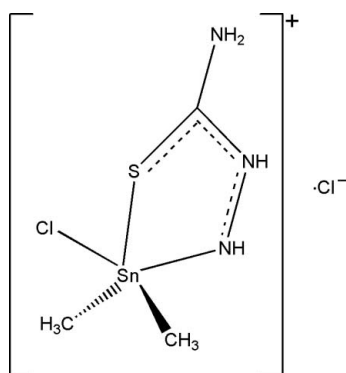
## Experimental

### Crystal data

$[Sn(CH_3)_2Cl(CH_4N_3S)]Cl$
$M_r = 309.79$
Monoclinic, $P2/c$
$a = 13.4980\ (12)$ Å
$b = 6.2470\ (5)$ Å
$c = 12.7160\ (13)$ Å
$\beta = 108.871\ (10)°$

$V = 1014.60\ (16)$ Å$^3$
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 3.19$ mm$^{-1}$
$T = 293$ K
$0.13 \times 0.10 \times 0.09$ mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan
  (*SORTAV*; Blessing, 1995)
$T_{min} = 0.613$, $T_{max} = 0.809$

4452 measured reflections
2915 independent reflections
2475 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.103$
$S = 1.14$
2915 reflections

91 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 1.04$ e Å$^{-3}$
$\Delta\rho_{min} = -1.52$ e Å$^{-3}$

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|---|---|---|---|
| N1—H1⋯Cl2$^i$ | 0.86 | 2.3555 | 3.147 (4) | 153.17 |
| N2—H2⋯S$^{ii}$ | 0.86 | 2.5549 | 3.327 (3) | 149.90 |

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

## References

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# supporting information

## Chloridodimethyl(thiosemicarbazide)tin(IV) chloride

**Laura Cortes C., Ana E. Burgos C. and Coco K. Y. A. Okio**

### S1. Comment

Organotin(IV) complexes have been extensively studied due to the diversity of structures that such compounds can form and to their potential biological activities as well as their wide industrial and agricultural applications (Davies & Smith, 1982). In the framework of our research for new organotin(IV) compounds (Delgado *et al.*, 2009), we report here the crystal structure of the title compound (I). The asymmetric unit is formed by one cation and one anion. The Sn atom is five-coordinate in a distorted trigonal–bipyramidal geometry. The distorted trigonal-bipyramidal coordination polyhedron has two methyl groups and one S atom in the equatorial plane, the N2 and Cl1 atom occupying the apical positions. In the crystal, molecules are linked by intermolecular N—H···S hydrogen bonds with set graph-motif C(4) along [010]. N—H··· Cl hydrogen bond linking cations and anions with set graph-motif D(2) and $D_3^3(10)$ , Table 1 and Fig.2. The C1-S and C1-N1 bond distances are quite shorter than the ones reported for C-S and C-N single bonds (1.755 (4), 1.366 (6)) (Delgado *et al.*, 2009), suggesting the delocalization of the C=S double bond on the SCN moiety.

### S2. Experimental

Compound (I) was obtained by reacting dimethyltin (IV) dichloride (220 mg, 1 mmol) with thiosemicarbazide (68 mg, 0.75 mmol) in methanol under reflux for 3 h. Colourless crystals suitable for X-ray analysis were grown by slow solvent evaporation.

### S3. Refinement

H atoms were positioned geometrically, with C—H, N—H distances of 0.96 and 0.86Å respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.5U_{eq}(C)$ and $1.2U_{eq}(N)$
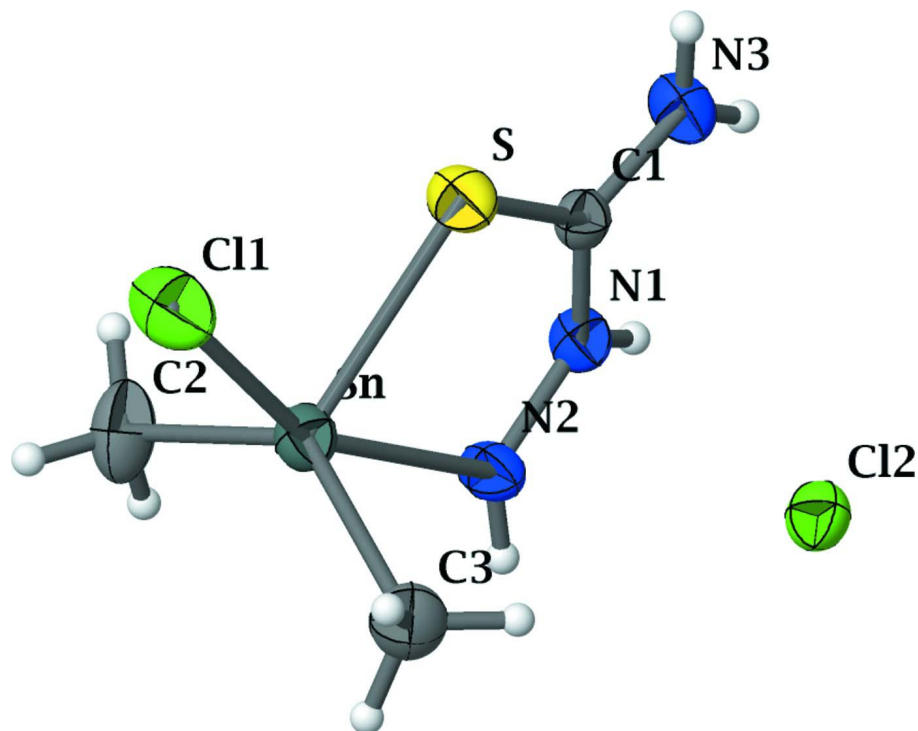
**Figure 1**

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.
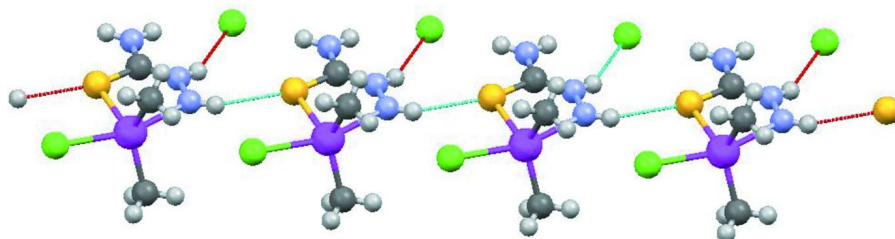


**Figure 2**

Unit-cell packing diagram for (I). Hydrogen bonds are shown as dashed lines.

**Chloridodimethyl(thiosemicarbazide)tin(IV) chloride**

*Crystal data*

[Sn(CH$_3$)$_2$Cl(CH$_4$N$_3$S)]Cl
$M_r$ = 309.79
Monoclinic, $P2/c$
Hall symbol: -P 2yc
$a$ = 13.4980 (12) Å
$b$ = 6.2470 (5) Å
$c$ = 12.7160 (13) Å
$\beta$ = 108.871 (10)°
$V$ = 1014.60 (16) Å$^3$
$Z$ = 4

$F(000)$ = 596
$D_x$ = 2.028 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 1915 reflections
$\theta$ = 1.0–30.0°
$\mu$ = 3.19 mm$^{-1}$
$T$ = 293 K
Prism, colorless
0.13 × 0.10 × 0.09 mm

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 4452 measured reflections |
| Radiation source: fine-focus sealed tube | 2915 independent reflections |
| Graphite monochromator | 2475 reflections with $I > 2\sigma(I)$ |
| $\pi$ scans | $R_{int} = 0.020$ |
| Absorption correction: multi-scan (*SORTAV*; Blessing, 1995) | $\theta_{max} = 30.0°$, $\theta_{min} = 1.6°$ |
| | $h = -18 \rightarrow 18$ |
| | $k = -7 \rightarrow 8$ |
| $T_{min} = 0.613$, $T_{max} = 0.809$ | $l = -17 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.037$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.103$ | |
| $S = 1.14$ | H-atom parameters constrained |
| 2915 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 1.1794P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 91 parameters | |
| 0 restraints | $(\Delta/\sigma)_{max} = 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 1.04$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -1.52$ e Å$^{-3}$ |

*Special details*

**Experimental**. Absorption correction: multi-scan from symmetry-related measurements (SORTAV; Blessing, 1995)

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Sn | 0.18008 (2) | 0.64590 (4) | 0.54686 (2) | 0.03327 (10) |
| Cl1 | 0.10951 (9) | 0.27486 (19) | 0.52290 (13) | 0.0570 (3) |
| Cl2 | 0.37205 (8) | 0.89418 (16) | 0.34229 (9) | 0.0384 (2) |
| S | 0.34762 (8) | 0.45496 (15) | 0.57559 (10) | 0.0394 (2) |
| N1 | 0.4153 (3) | 0.8592 (5) | 0.6156 (3) | 0.0304 (6) |
| H1 | 0.4647 | 0.9508 | 0.6420 | 0.036* |
| N2 | 0.3105 (2) | 0.9290 (5) | 0.5800 (3) | 0.0348 (7) |
| H2 | 0.2937 | 1.0621 | 0.5707 | 0.042* |
| N3 | 0.5392 (3) | 0.6073 (5) | 0.6316 (3) | 0.0371 (7) |
| H3AN | 0.5859 | 0.7063 | 0.6494 | 0.045* |
| H3BN | 0.5575 | 0.4762 | 0.6282 | 0.045* |
| C1 | 0.4390 (3) | 0.6566 (6) | 0.6093 (3) | 0.0291 (7) |
| C2 | 0.1509 (4) | 0.7230 (10) | 0.6955 (4) | 0.0548 (12) |
| H2A | 0.1548 | 0.8753 | 0.7060 | 0.082* |
| H2B | 0.2022 | 0.6549 | 0.7569 | 0.082* |

| | | | |
|---|---|---|---|
| H2C | 0.0823 | 0.6738 | 0.6913 | 0.082* |
| C3 | 0.1119 (4) | 0.7551 (9) | 0.3820 (4) | 0.0471 (10) |
| H3A | 0.0640 | 0.8695 | 0.3806 | 0.071* |
| H3B | 0.0749 | 0.6396 | 0.3362 | 0.071* |
| H3C | 0.1658 | 0.8060 | 0.3542 | 0.071* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sn | 0.03237 (15) | 0.03147 (15) | 0.03651 (16) | −0.00300 (10) | 0.01187 (11) | −0.00248 (10) |
| Cl1 | 0.0403 (5) | 0.0344 (5) | 0.0951 (9) | −0.0086 (5) | 0.0201 (6) | −0.0063 (6) |
| Cl2 | 0.0369 (5) | 0.0377 (5) | 0.0413 (5) | −0.0039 (4) | 0.0138 (4) | 0.0036 (4) |
| S | 0.0352 (5) | 0.0245 (4) | 0.0595 (6) | −0.0027 (4) | 0.0168 (4) | −0.0017 (4) |
| N1 | 0.0299 (15) | 0.0266 (14) | 0.0342 (15) | −0.0046 (12) | 0.0097 (12) | −0.0039 (12) |
| N2 | 0.0285 (15) | 0.0259 (14) | 0.0494 (19) | 0.0002 (13) | 0.0117 (13) | 0.0017 (14) |
| N3 | 0.0316 (16) | 0.0367 (17) | 0.0444 (19) | −0.0017 (14) | 0.0141 (14) | −0.0064 (14) |
| C1 | 0.0322 (18) | 0.0308 (17) | 0.0262 (15) | 0.0006 (14) | 0.0121 (13) | 0.0015 (13) |
| C2 | 0.062 (3) | 0.067 (3) | 0.046 (3) | −0.017 (3) | 0.031 (2) | −0.010 (2) |
| C3 | 0.042 (2) | 0.057 (3) | 0.038 (2) | −0.002 (2) | 0.0072 (17) | 0.002 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Sn—C2 | 2.107 (4) | N3—C1 | 1.325 (5) |
| Sn—C3 | 2.112 (4) | N3—H3AN | 0.8600 |
| Sn—N2 | 2.434 (3) | N3—H3BN | 0.8600 |
| Sn—S | 2.4771 (11) | C2—H2A | 0.9600 |
| Sn—Cl1 | 2.4870 (12) | C2—H2B | 0.9600 |
| S—C1 | 1.718 (4) | C2—H2C | 0.9600 |
| N1—C1 | 1.315 (5) | C3—H3A | 0.9600 |
| N1—N2 | 1.408 (4) | C3—H3B | 0.9600 |
| N1—H1 | 0.8600 | C3—H3C | 0.9600 |
| N2—H2 | 0.8600 | | |
| | | | |
| C2—Sn—C3 | 132.0 (2) | C1—N3—H3BN | 120.0 |
| C2—Sn—N2 | 90.75 (17) | H3AN—N3—H3BN | 120.0 |
| C3—Sn—N2 | 89.75 (16) | N1—C1—N3 | 117.6 (3) |
| C2—Sn—S | 113.85 (17) | N1—C1—S | 123.5 (3) |
| C3—Sn—S | 112.70 (14) | N3—C1—S | 118.9 (3) |
| N2—Sn—S | 75.51 (8) | Sn—C2—H2A | 109.5 |
| C2—Sn—Cl1 | 98.48 (16) | Sn—C2—H2B | 109.5 |
| C3—Sn—Cl1 | 98.80 (15) | H2A—C2—H2B | 109.5 |
| N2—Sn—Cl1 | 157.72 (8) | Sn—C2—H2C | 109.5 |
| S—Sn—Cl1 | 82.21 (4) | H2A—C2—H2C | 109.5 |
| C1—S—Sn | 103.38 (13) | H2B—C2—H2C | 109.5 |
| C1—N1—N2 | 121.2 (3) | Sn—C3—H3A | 109.5 |
| C1—N1—H1 | 119.4 | Sn—C3—H3B | 109.5 |
| N2—N1—H1 | 119.4 | H3A—C3—H3B | 109.5 |
| N1—N2—Sn | 115.2 (2) | Sn—C3—H3C | 109.5 |

| | | | |
|---|---|---|---|
| N1—N2—H2 | 122.4 | H3A—C3—H3C | 109.5 |
| Sn—N2—H2 | 122.4 | H3B—C3—H3C | 109.5 |
| C1—N3—H3AN | 120.0 | | |
| | | | |
| C2—Sn—S—C1 | −79.8 (2) | S—Sn—N2—N1 | −9.3 (2) |
| C3—Sn—S—C1 | 88.0 (2) | Cl1—Sn—N2—N1 | −9.7 (4) |
| N2—Sn—S—C1 | 4.48 (15) | N2—N1—C1—N3 | 172.0 (3) |
| Cl1—Sn—S—C1 | −175.69 (14) | N2—N1—C1—S | −8.9 (5) |
| C1—N1—N2—Sn | 13.1 (4) | Sn—S—C1—N1 | 0.0 (4) |
| C2—Sn—N2—N1 | 105.2 (3) | Sn—S—C1—N3 | 179.2 (3) |
| C3—Sn—N2—N1 | −122.9 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H⋯*A* | *D*—H | H⋯*A* | *D*⋯*A* | *D*—H⋯*A* |
|---|---|---|---|---|
| N1—H1⋯Cl2[i] | 0.86 | 2.3555 | 3.147 (4) | 153.17 |
| N2—H2⋯S[ii] | 0.86 | 2.5549 | 3.327 (3) | 149.90 |

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