Acta Crystallographica Section C
**Crystal Structure
Communications**
ISSN 0108-2701

Dichlorobis(trimethylenethiourea-*κS*)-antimony(III) chloride

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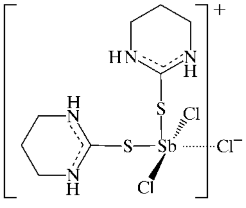
Received 19 September 2001
Accepted 5 December 2001
Online 31 January 2002

In the title compound, $[SbCl_2(C_4H_8N_2S)_2]Cl$, the coordination around the Sb atom can be described as distorted pseudo-octahedral. Both rings of the trimethylenethiourea ligands [alternatively 3,4,5,6-tetrahydropyrimidine-2(1*H*)-thione] adopt an envelope conformation. The molecules are connected into dimers in the *ab* plane by two intermolecular hydrogen bonds. The dimers are arranged into infinite one-dimensional chains along the *a* axis as a result of the Cl^- ions forming intermolecular hydrogen bonds with three NH groups.

Comment

Cyclic monothiones act exclusively as monodentate ligands with several metals. The complexes can be neutral molecules or cations. Some examples of cationic complexes of the ligands are tetrakis[1-methyl-2(3*H*)-imidazolinethione]zinc(II) nitrate monohydrate (Nowell *et al.*, 1979), tris(ethylenethiourea-*S*)tellurium(II) perchlorate and tris(trimethylenethiourea-*S*)tellurium(II) perchlorate (Foust, 1980). The ability of imidazolidine-2-thione (trimethylenethiourea) to reduce metallic salts and form complexes is demonstrated by the formation of tetrakis(imidazolidine-2-thione)copper(I) nitrate (Raper, 1985) and bis[bis(imidazolidine-2-thione)(μ-imidazolidine-2-thione)copper(I)] diperchlorate (Raper *et al.*, 1992) when it is reacted with copper(II) nitrate and copper(II) perchlorate, respectively. Our interest in the structures of antimony(III) halides complexed with *S*-donor ligands led us to investigate the title complex, (I) (Fig. 1).

The average thioamide N—C [1.314 (7) Å] and C—C [1.479 (12) Å] bond lengths of the ligands are shorter compared with the values in the free ligand (Dias & Truter, 1964), while the average S—C [1.751 (6) Å] and $N—C_{methylene}$ [1.456 (9) Å] bond lengths are longer (Table 1). The longer S—C bond length results from the reduction in the π-electron density of the exocyclic S—C bond for the S atom coordinated to the metal atom. This reduction contributes to an increased π-electron density of the thioamide N—C bonds, resulting in the shortening of the N—C bond lengths. The average S—C—N [119.2 (4)°], N—C—N [121.5 (5)°], C—N—C [122.7 (5)°] and N—C—C [109.9 (6)°] bond angles are also comparable with the reported values in the free ligand (Dias & Truter, 1964), while the average C—C—C bond angle is slightly bigger. Of the two Sb—Cl bonds, the Sb1—Cl2 bond, where Cl2 is involved in intermolecular hydrogen bonding, is longer than Sb1—Cl1. This behaviour is also observed both in the Sb—Cl bond lengths reported by Razak *et al.* (1999) for $[SbCl_3\{[(C_6H_5)_2PO]_2CH_2\}]$ and in the Bi—Cl bond lengths reported in the structures of $[BiCl_3(pptu)_3]$ and $[\{BiCl_3(deimdt)_2\}_2]$ [pptu is 1-phenyl-3-(2-pyridyl)-2-thiourea and deimdt is *N,N′*-diethylimidazolidine-2-thione; Battaglia *et al.*, 1978].



(I)

The coordination around the Sb1 atom can be described as distorted octahedral. The basal plane is occupied by atoms S2, Cl1 and Cl2 from the cation and the Cl3 anion at a longer distance [Sb1···Cl3 = 3.010 (2) Å and S2—Sb1···Cl3 = 176.7 (1)°], which is less than the sum of the contact radii of Sb and Cl. The Sb1···Cl3 short contact has a lengthening effect on the Sb1—S2 bond compared with that of Sb1—S1. The apical positions of the octahedron are occupied by the S1 atom and the lone-pair electrons on Sb1.

The two trimethylenethiourea ring moieties adopt an envelope conformation, with atoms C3 and C7 deviating by 0.338 (9) and 0.262 (11) Å, respectively, from the mean plane through each of the two rings.

In the crystal, all the thiourea NH groups are involved in intermolecular hydrogen bonding (Table 2). N3—H3*A*···Cl2(1 − *x*, 1 − *y*, −*z*) hydrogen bonds interconnect molecules into dimers situated in the *ab* plane (Fig. 2). These dimers are arranged into infinite one-dimensional chains along

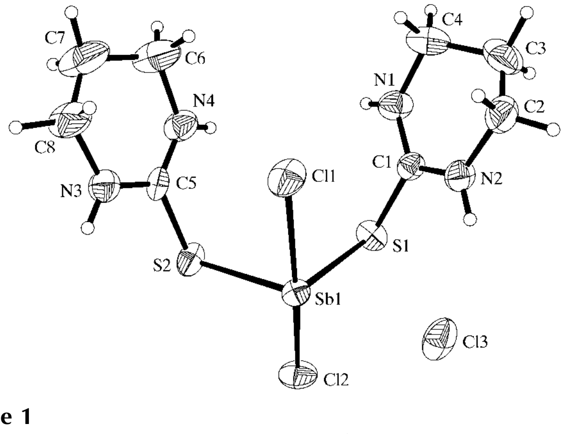


Figure 1
The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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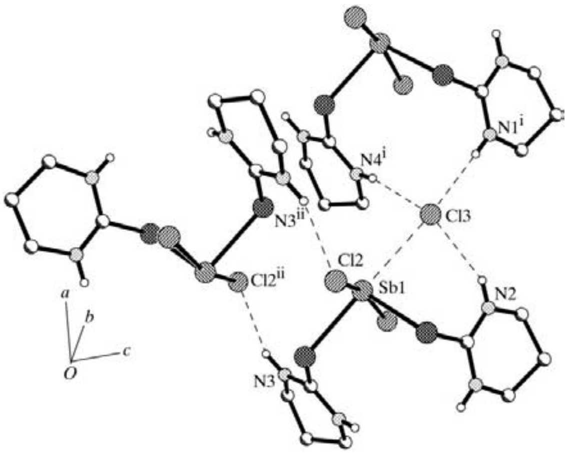

Figure 2

Packing diagram showing the hydrogen-bonding and secondary Sb···Cl interactions. [Symmetry codes: (i) $1 + x, y, z$; (ii) $1 - x, 1 - y, -z$.]

the *a* axis as a result of the Cl3 atoms forming intermolecular hydrogen bonds with the remaining NH groups; N2—H2*A*···Cl3, N1—H1*A*···Cl3($x - 1, y, z$) and N4—H4*A*···Cl3($x - 1, y, z$).

Experimental

Solutions of antimony trichloride (0.320 g, 1.40 mmol) and trimethylenethiourea (0.325 g, 0.28 mmol) in a 1:2 molar ratio in acetonitrile were mixed in a 50 ml flask and stirred for about 30 min. The solution was then filtered and left to evaporate slowly. After three days, single crystals were collected (yield 62%), washed with hexane and dried before being subjected to X-ray crystallographic analysis.

Crystal data

$[SbCl_2(C_4H_8N_2S)_2]Cl$
$M_r = 460.47$
Triclinic, $P\bar{1}$
$a = 7.5103$ (4) Å
$b = 10.3304$ (5) Å
$c = 12.1206$ (6) Å
$\alpha = 71.358$ (1)°
$\beta = 84.252$ (1)°
$\gamma = 73.612$ (1)°
$V = 854.78$ (7) Å^3
$Z = 2$
$D_x = 1.789$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5944 reflections
$\theta = 1.8$–29.4°
$\mu = 2.32$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.20 × 0.16 × 0.14 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.655$, $T_{\max} = 0.738$
4916 measured reflections
2981 independent reflections
2799 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.069$
$\theta_{\max} = 25.0°$
$h = -7 \rightarrow 8$
$k = -12 \rightarrow 9$
$l = -14 \rightarrow 14$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.158$
$S = 1.09$
2981 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0841P)^2 + 0.1942P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.55$ e Å^{-3}
$\Delta\rho_{\min} = -1.79$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.054 (5)

Table 1

Selected geometric parameters (Å, °).

Sb1—S1	2.481 (1)	N2—C2	1.465 (8)
Sb1—S2	2.555 (2)	N3—C5	1.330 (7)
Sb1—Cl1	2.514 (1)	N3—C8	1.453 (9)
Sb1—Cl2	2.670 (2)	N4—C5	1.306 (7)
S1—C1	1.757 (5)	N4—C6	1.481 (9)
S2—C5	1.745 (6)	C2—C3	1.492 (11)
N1—C1	1.316 (7)	C3—C4	1.517 (11)
N1—C4	1.462 (8)	C6—C7	1.461 (13)
N2—C1	1.303 (7)	C7—C8	1.490 (12)
S1—Sb1—Cl1	92.41 (5)	Cl1—Sb1—S2	92.43 (5)
S1—Sb1—Cl2	76.62 (5)	Cl1—Sb1—Cl2	163.60 (6)
S1—Sb1—S2	92.59 (5)	C1—S1—Sb1	104.01 (18)
S2—Sb1—Cl2	76.18 (5)	C5—S2—Sb1	104.18 (18)

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl3^{i}	0.86	2.37	3.213 (6)	169
N2—H2*A*···Cl3	0.86	2.44	3.227 (5)	152
N3—H3*A*···Cl2^{ii}	0.86	2.40	3.203 (6)	156
N4—H4*A*···Cl3^{i}	0.86	2.37	3.168 (6)	155

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

After checking their presence in a difference map, all the H atoms were fixed geometrically and allowed to ride on their attached atoms (N—H = 0.86 Å and C—H = 0.97 Å). The highest peak and the deepest hole were found near the Sb1 atom at distances of 0.94 and 1.12 Å, respectively.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

The authors would like to thank the Malaysian Government and both Universiti Kebangsaan Malaysia and Universiti Sains Malaysia for research grants (IRPA Nos. 09-02-02-0163 and 305/PFIZIK/610961, respectively). AU thanks Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TR1006). Services for accessing these data are described at the back of the journal.

References

Battaglia, L. P., Corradi, A. B., Nardelli, M. & Tani, M. E. V. (1978). *J. Chem. Soc. Dalton Trans.* pp. 583–587.
Dias, H. W. & Truter, M. R. (1964). *Acta Cryst.* **17**, 937–943.
Foust, A. S. (1980). *Inorg. Chem.* **19**, 1050–1055.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Nowell, I. W., Cox, A. G. & Raper, E. S. (1979). *Acta Cryst.* B**35**, 3047–3050.
Raper, E. S. (1985). *Coord. Chem. Rev.* **61**, 115–184.
Raper, E. S., Wilson, J. D. & Clegg, W. (1992). *Inorg. Chim. Acta*, **194**, 51–55.
Razak, I. A., Fun, H.-K., Yamin, B. M., Chinnakali, K., Zakaria, H. & Ismail, N. B. (1999). *Acta Cryst.* C**55**, 172–174.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.