Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Chloridobis(pyrrolidine-1-dithiocarboxylato-κ^2S,S')antimony(III)

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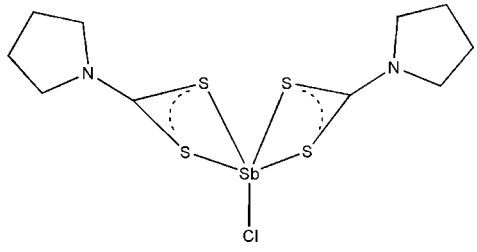
Received 12 September 2007; accepted 29 October 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.027; wR factor = 0.060; data-to-parameter ratio = 17.0.

In the title compound, $[Sb(C_5H_8NS_2)_2Cl]$, the Sb^{III} ion is coordinated by the four S atoms belonging to two pyrrolidine-1-dithiocarboxylate ligands and a Cl atom in a distorted trigonal–bipyramidal geometry. The crystal structure is stabilized by intermolecular Sb···S interactions of 3.689 (1) Å.

Related literature

For details of the versatile coordination modes of dithiocarbamates, see: Bardaji *et al.* (1994) and Xu *et al.* (2001). For the crystal structure of an isomer of the title compound, see: Zhai *et al.* (2007).



Experimental

Crystal data

$[Sb(C_5H_8NS_2)_2Cl]$
M_r = 449.69
Triclinic, $P\overline{1}$
a = 6.367 (2) Å
b = 10.368 (3) Å
c = 13.414 (4) Å
α = 111.451 (3)°
β = 91.950 (3)°
γ = 102.334 (3)°
V = 799.0 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.40 mm^{-1}
T = 298 (2) K
0.43 × 0.22 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.540, T_{max} = 0.748
4147 measured reflections
2764 independent reflections
2300 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.060
S = 1.06
2764 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

We acknowledge the National Natural Foundation of China (Grant No. 20771053), Shandong Province Science Foundation and the State Key Laboratory of Crystal Material, Shandong University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2025).

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 doi:10.1107/S1600536807053962

supplementary materials

Acta Cryst. (2007). E**63**, m2871 [**doi:10.1107/S1600536807053962**]

Chloridobis(pyrrolidine-1-dithiocarboxylato-κ^2S,S')antimony(III)

J. Zhai, H. Yin, F. Li and D. Wang

Comment

Dithiocarbamates have been known as effective ligands for transition metal ions, which can form chelates (Xu *et al.*, 2001) or act as bridging ligands (Bardaji *et al.*, 1994). We have reported a similar compound, $C_{10}H_{16}N_2S_4SbBr$ (Zhai *et al.*, 2007). As part of our continuing studies on the chemistry of main-group metal complexes with dithiocarbamates, we have recently described the crystal structure of a similar compound, $[SbBr(C_5H_8NS_2]_2$, (Zhai *et al.*, 2007). Herein we report the crystal structure of the title compound, bis(pyrrolidine-1-dithiocarboxylato-κ^2S,S') chloridoantimony(III) (Fig. 1).

In the title compound (Fig. 1), the Sb^{III} ion is coordinated by the four S atoms [Sb—S; 2.466 (1)–2.942 (1) Å] from two pyrrolidine-1-dithiocarboxylate ligands and a chloride atom in a distorted trigonal-bipyramid geometry, with S2, Cl in the axial sites and S1, S3, S4, Sb occupying the equatorial plane. The angles at Sb confirm that the complex has a distorted trigonal-bipyramid configuration. The short intermolecular distance $Sb\cdots S1^{i}$ of 3.689 (1) Å suggests a presence of Sb···S interactions (Symmetry code as in Fig. 2).

Experimental

The title compound were prepared by reaction of antimony trichloride (22.8 mg, 0.1 mmol) with the corresponding sodium dithiocarbamate (33.8 mg, 0.2 mmol), in absolute acetone. After stirring for 5 h at room temperature, the yellow paste was obtained and then filtered. Yellow crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol/dichloromethane (1:2 *v*/*v*) solution over a period of two weeks (yield 85%. m.p. 421k).

Refinement

All methylene H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å, and with U_{iso}(H) = 1.2Ueq(C).

Figures

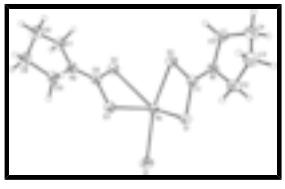

Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

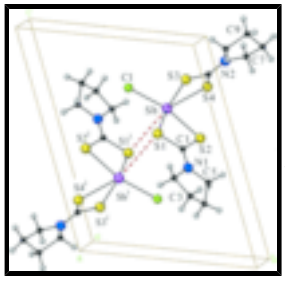

Fig. 2. Sb···S interactions (dashed lines) in the title compound. [Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*.]

Chloridobis(pyrrolidine-1-dithiocarboxylato- κ^2S,S')antimony(III)

Crystal data

$[Sb(C_5H_8NS_2)_2Cl]$	$Z = 2$
$M_r = 449.69$	$F_{000} = 444$
Triclinic, $P\overline{1}$	$D_x = 1.869$ Mg m^{-3}
Hall symbol: -p_1	Melting point: 421 K
$a = 6.367$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.368$ (3) Å	Cell parameters from 2270 reflections
$c = 13.414$ (4) Å	$\theta = 2.2$–27.2º
$\alpha = 111.451$ (3)º	$\mu = 2.40$ mm^{-1}
$\beta = 91.950$ (3)º	$T = 298$ (2) K
$\gamma = 102.334$ (3)º	Block, yellow
$V = 799.0$ (4) Å^3	0.43 × 0.22 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2764 independent reflections
Radiation source: fine-focus sealed tube	2300 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{max} = 25.0$º
$T = 298(2)$ K	$\theta_{min} = 1.6$º
φ and ω scans	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -12 \rightarrow 12$
$T_{min} = 0.540$, $T_{max} = 0.748$	$l = -15 \rightarrow 14$
4147 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	H-atom parameters constrained
$wR(F^2) = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2 + 0.5878P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2764 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
163 parameters	$\Delta\rho_{min} = -0.39$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > \sigma(F^2)$ 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}
Sb	0.47864 (4)	0.69539 (3)	0.67024 (2)	0.04236 (10)
Cl	0.55811 (19)	0.51311 (11)	0.75268 (10)	0.0617 (3)
N1	0.8661 (5)	0.7553 (3)	0.4252 (2)	0.0407 (7)
N2	0.6570 (5)	1.1281 (3)	0.9245 (2)	0.0422 (8)
S1	0.79220 (18)	0.62807 (11)	0.56520 (8)	0.0470 (3)
S2	0.57692 (18)	0.84792 (11)	0.55290 (9)	0.0494 (3)
S3	0.73312 (16)	0.87619 (11)	0.82510 (8)	0.0461 (3)
S4	0.32233 (16)	0.95228 (11)	0.78277 (9)	0.0492 (3)
C1	0.7584 (6)	0.7455 (4)	0.5043 (3)	0.0409 (9)
C2	1.0227 (7)	0.6709 (4)	0.3792 (3)	0.0491 (10)
H2A	0.9501	0.5719	0.3370	0.059*
H2B	1.1267	0.6746	0.4354	0.059*
C3	1.1315 (8)	0.7439 (5)	0.3083 (4)	0.0653 (13)
H3A	1.2543	0.8217	0.3491	0.078*
H3B	1.1805	0.6766	0.2476	0.078*
C4	0.9570 (7)	0.7985 (5)	0.2707 (4)	0.0607 (12)
H4A	0.8640	0.7239	0.2087	0.073*
H4B	1.0193	0.8785	0.2511	0.073*
C5	0.8312 (7)	0.8447 (4)	0.3655 (3)	0.0493 (10)
H5A	0.8865	0.9454	0.4097	0.059*
H5B	0.6785	0.8272	0.3416	0.059*
C6	0.5738 (6)	1.0000 (4)	0.8510 (3)	0.0373 (9)
C7	0.5537 (7)	1.2488 (4)	0.9503 (4)	0.0542 (11)
H7A	0.4385	1.2397	0.9946	0.065*
H7B	0.4950	1.2553	0.8851	0.065*
C8	0.7370 (8)	1.3764 (5)	1.0113 (4)	0.0682 (13)

H8A	0.6835	1.4527	1.0620	0.082*
H8B	0.8160	1.4119	0.9623	0.082*
C9	0.8783 (8)	1.3204 (5)	1.0696 (4)	0.0726 (14)
H9A	0.8228	1.3188	1.1357	0.087*
H9B	1.0257	1.3790	1.0872	0.087*
C10	0.8697 (6)	1.1707 (4)	0.9909 (3)	0.0513 (11)
H10A	0.9873	1.1702	0.9468	0.062*
H10B	0.8773	1.1074	1.0285	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb	0.04020 (17)	0.03755 (16)	0.04356 (17)	0.00320 (11)	−0.00365 (11)	0.01315 (12)
Cl	0.0679 (8)	0.0477 (6)	0.0738 (8)	0.0122 (5)	0.0030 (6)	0.0298 (6)
N1	0.0409 (19)	0.0416 (18)	0.0417 (19)	0.0116 (15)	−0.0013 (15)	0.0180 (15)
N2	0.0419 (19)	0.0383 (18)	0.0404 (19)	0.0063 (15)	0.0001 (15)	0.0105 (15)
S1	0.0551 (7)	0.0439 (6)	0.0477 (6)	0.0167 (5)	0.0046 (5)	0.0214 (5)
S2	0.0574 (7)	0.0480 (6)	0.0487 (6)	0.0223 (5)	0.0068 (5)	0.0201 (5)
S3	0.0399 (6)	0.0441 (6)	0.0479 (6)	0.0109 (5)	−0.0091 (5)	0.0112 (5)
S4	0.0333 (5)	0.0490 (6)	0.0586 (7)	0.0080 (5)	−0.0051 (5)	0.0149 (5)
C1	0.041 (2)	0.034 (2)	0.040 (2)	0.0037 (17)	−0.0081 (18)	0.0107 (17)
C2	0.046 (2)	0.048 (2)	0.059 (3)	0.014 (2)	0.007 (2)	0.025 (2)
C3	0.064 (3)	0.074 (3)	0.077 (3)	0.030 (3)	0.026 (3)	0.041 (3)
C4	0.063 (3)	0.071 (3)	0.061 (3)	0.021 (2)	0.014 (2)	0.036 (3)
C5	0.053 (3)	0.052 (2)	0.050 (3)	0.013 (2)	0.001 (2)	0.027 (2)
C6	0.033 (2)	0.041 (2)	0.037 (2)	0.0022 (17)	0.0027 (16)	0.0185 (18)
C7	0.057 (3)	0.043 (2)	0.056 (3)	0.016 (2)	0.007 (2)	0.010 (2)
C8	0.084 (4)	0.046 (3)	0.063 (3)	0.006 (3)	0.006 (3)	0.012 (2)
C9	0.079 (4)	0.056 (3)	0.060 (3)	−0.006 (3)	−0.010 (3)	0.009 (2)
C10	0.044 (2)	0.056 (3)	0.041 (2)	0.000 (2)	−0.0083 (19)	0.012 (2)

Geometric parameters (Å, °)

Sb—S1	2.555 (1)	C3—C4	1.499 (6)
Sb—S2	2.614 (1)	C3—H3A	0.9700
Sb—S3	2.466 (1)	C3—H3B	0.9700
Sb—S4	2.942 (1)	C4—C5	1.509 (6)
Sb—S1^{i}	3.689 (2)	C4—H4A	0.9700
Sb—Cl	2.636 (1)	C4—H4B	0.9700
N1—C1	1.306 (5)	C5—H5A	0.9700
N1—C2	1.469 (5)	C5—H5B	0.9700
N1—C5	1.473 (4)	C7—C8	1.505 (6)
N2—C6	1.312 (5)	C7—H7A	0.9700
N2—C7	1.473 (5)	C7—H7B	0.9700
N2—C10	1.482 (5)	C8—C9	1.505 (6)
S1—C1	1.739 (4)	C8—H8A	0.9700
S2—C1	1.719 (4)	C8—H8B	0.9700
S3—C6	1.747 (4)	C9—C10	1.513 (6)

S4—C6	1.696 (4)	C9—H9A	0.9700
C2—C3	1.519 (5)	C9—H9B	0.9700
C2—H2A	0.9700	C10—H10A	0.9700
C2—H2B	0.9700	C10—H10B	0.9700
S3—Sb—S1	91.14 (4)	C3—C4—C5	105.1 (3)
S3—Sb—S2	91.50 (4)	C3—C4—H4A	110.7
S1—Sb—S2	69.56 (3)	C5—C4—H4A	110.7
S3—Sb—Cl	84.64 (4)	C3—C4—H4B	110.7
S1—Sb—Cl	82.23 (4)	C5—C4—H4B	110.7
S2—Sb—Cl	151.46 (4)	H4A—C4—H4B	108.8
S3—Sb—S4	66.25 (4)	N1—C5—C4	103.4 (3)
S1—Sb—S4	138.73 (3)	N1—C5—H5A	111.1
S2—Sb—S4	76.71 (4)	C4—C5—H5A	111.1
Cl—Sb—S4	126.19 (4)	N1—C5—H5B	111.1
S3—Sb—S1^{i}	164.90 (3)	C4—C5—H5B	111.1
S1—Sb—S1^{i}	77.01 (4)	H5A—C5—H5B	109.0
S2—Sb—S1^{i}	93.03 (4)	N2—C6—S4	122.8 (3)
Cl—Sb—S1^{i}	84.50 (4)	N2—C6—S3	117.2 (3)
S4—Sb—S1^{i}	128.84 (3)	S4—C6—S3	120.0 (2)
C1—N1—C2	124.6 (3)	N2—C7—C8	103.3 (3)
C1—N1—C5	123.4 (3)	N2—C7—H7A	111.1
C2—N1—C5	111.8 (3)	C8—C7—H7A	111.1
C6—N2—C7	124.5 (3)	N2—C7—H7B	111.1
C6—N2—C10	124.3 (3)	C8—C7—H7B	111.1
C7—N2—C10	111.2 (3)	H7A—C7—H7B	109.1
C1—S1—Sb	87.21 (14)	C7—C8—C9	104.1 (4)
C1—S2—Sb	85.70 (13)	C7—C8—H8A	110.9
C6—S3—Sb	93.81 (13)	C9—C8—H8A	110.9
C6—S4—Sb	79.43 (13)	C7—C8—H8B	110.9
N1—C1—S2	121.6 (3)	C9—C8—H8B	110.9
N1—C1—S1	121.4 (3)	H8A—C8—H8B	109.0
S2—C1—S1	117.0 (2)	C8—C9—C10	104.5 (4)
N1—C2—C3	102.8 (3)	C8—C9—H9A	110.8
N1—C2—H2A	111.2	C10—C9—H9A	110.8
C3—C2—H2A	111.2	C8—C9—H9B	110.8
N1—C2—H2B	111.2	C10—C9—H9B	110.8
C3—C2—H2B	111.2	H9A—C9—H9B	108.9
H2A—C2—H2B	109.1	N2—C10—C9	103.4 (3)
C4—C3—C2	103.9 (3)	N2—C10—H10A	111.1
C4—C3—H3A	111.0	C9—C10—H10A	111.1
C2—C3—H3A	111.0	N2—C10—H10B	111.1
C4—C3—H3B	111.0	C9—C10—H10B	111.1
C2—C3—H3B	111.0	H10A—C10—H10B	109.0
H3A—C3—H3B	109.0		
S3—Sb—S1—C1	−95.47 (13)	Sb—S2—C1—S1	−6.72 (19)
S2—Sb—S1—C1	−4.29 (12)	Sb—S1—C1—N1	−173.1 (3)
Cl—Sb—S1—C1	−179.90 (13)	Sb—S1—C1—S2	6.9 (2)

S4—Sb—S1—C1	−41.48 (14)	C1—N1—C2—C3	−168.9 (4)
S1^{i}—Sb—S1—C1	93.98 (13)	C5—N1—C2—C3	15.7 (5)
S3—Sb—S2—C1	95.01 (13)	N1—C2—C3—C4	−31.5 (5)
S1—Sb—S2—C1	4.35 (13)	C2—C3—C4—C5	36.4 (5)
Cl—Sb—S2—C1	13.47 (16)	C1—N1—C5—C4	−169.2 (4)
S4—Sb—S2—C1	160.16 (13)	C2—N1—C5—C4	6.3 (4)
S1^{i}—Sb—S2—C1	−70.58 (13)	C3—C4—C5—N1	−26.3 (5)
S1—Sb—S3—C6	140.29 (12)	C7—N2—C6—S4	−5.7 (5)
S2—Sb—S3—C6	70.71 (12)	C10—N2—C6—S4	175.7 (3)
Cl—Sb—S3—C6	−137.62 (12)	C7—N2—C6—S3	174.7 (3)
S4—Sb—S3—C6	−4.06 (12)	C10—N2—C6—S3	−3.9 (5)
S1^{i}—Sb—S3—C6	178.18 (14)	Sb—S4—C6—N2	174.1 (3)
S3—Sb—S4—C6	4.24 (12)	Sb—S4—C6—S3	−6.34 (18)
S1—Sb—S4—C6	−57.83 (13)	Sb—S3—C6—N2	−172.9 (3)
S2—Sb—S4—C6	−93.42 (12)	Sb—S3—C6—S4	7.5 (2)
Cl—Sb—S4—C6	67.61 (13)	C6—N2—C7—C8	−163.2 (4)
S1^{i}—Sb—S4—C6	−176.51 (12)	C10—N2—C7—C8	15.6 (4)
C2—N1—C1—S2	−179.7 (3)	N2—C7—C8—C9	−32.2 (5)
C5—N1—C1—S2	−4.8 (5)	C7—C8—C9—C10	37.3 (5)
C2—N1—C1—S1	0.3 (5)	C6—N2—C10—C9	−174.1 (4)
C5—N1—C1—S1	175.2 (3)	C7—N2—C10—C9	7.1 (4)
Sb—S2—C1—N1	173.3 (3)	C8—C9—C10—N2	−27.1 (5)

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

Fig. 1

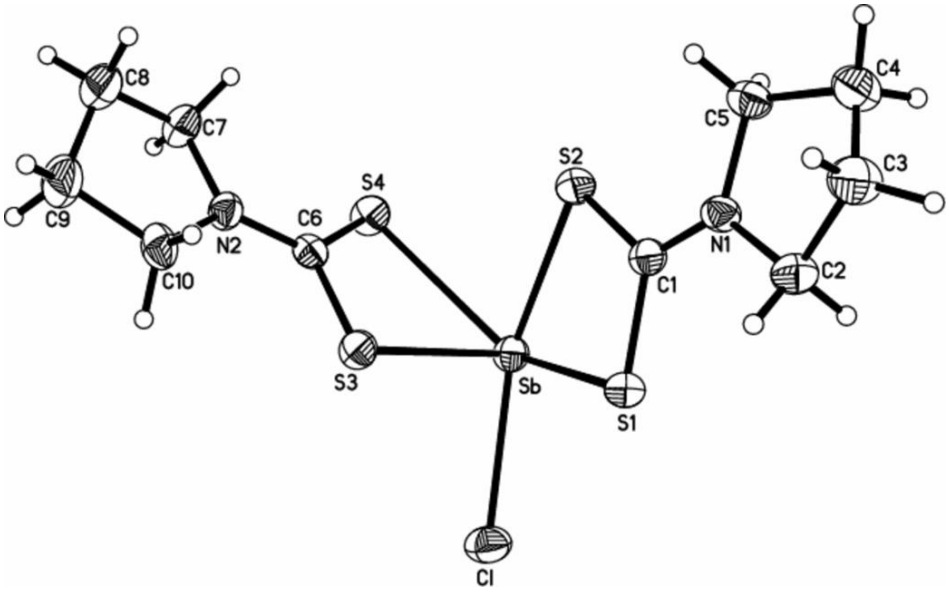

Fig. 2