Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tris(morpholine-4-dithiocarboxylato-κ^2S,S')antimony(III)

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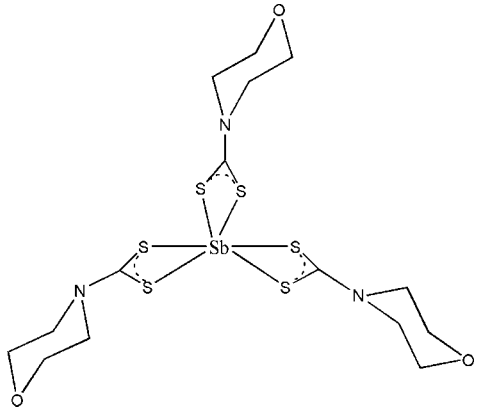
Received 27 October 2007; accepted 7 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; R factor = 0.044; wR factor = 0.111; data-to-parameter ratio = 16.0.

In the title compound, $[Sb(C_5H_8NOS_2)_3]$, the Sb^{III} ion adopts a distorted pentagonal–pyramidal geometry because of its stereochemically active lone pair of electrons. In the crystal structure, the molecules are associated into dimers by short intermolecular Sb···S contacts [3.4111 (17) Å].

Related literature

For descriptions of the versatile coordination modes of dithiocarbamates, see: Xu *et al.* (2001); Bardaji *et al.* (1994).



Experimental

Crystal data

$[Sb(C_5H_8NOS_2)_3]$
M_r = 608.48
Monoclinic, $P2_1/c$
a = 12.8891 (13) Å
b = 20.009 (2) Å
c = 9.0073 (9) Å
β = 94.734 (2)°
V = 2315.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.76 mm^{-1}
T = 298 (2) K
0.59 × 0.20 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.424, T_{max} = 0.872
11465 measured reflections
4056 independent reflections
2716 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.111
S = 1.01
4056 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sb1—S1	2.4742 (16)	Sb1—S5	2.8015 (18)
Sb1—S4	2.6826 (16)	Sb1—S2	2.8307 (16)
Sb1—S6	2.6876 (17)	Sb1—S3	2.8329 (17)
S1—Sb1—S4	88.42 (5)	S6—Sb1—S2	138.11 (5)
S1—Sb1—S6	90.02 (6)	S5—Sb1—S2	77.50 (5)
S4—Sb1—S6	73.60 (5)	S1—Sb1—S3	83.97 (5)
S1—Sb1—S5	83.44 (6)	S4—Sb1—S3	64.39 (5)
S4—Sb1—S5	137.49 (5)	S6—Sb1—S3	137.66 (5)
S6—Sb1—S5	64.78 (5)	S5—Sb1—S3	154.16 (5)
S1—Sb1—S2	67.35 (5)	S2—Sb1—S3	76.84 (5)
S4—Sb1—S2	136.27 (5)		

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 Science Foundation of China (grant No. 20771053), Shandong Province Science Foundation and the State Key Laboratory of Crystalline Material, Shandong University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2460).

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 doi:10.1107/S1600536807056516

supplementary materials

Acta Cryst. (2007). E**63**, m2989 [**doi:10.1107/S1600536807056516**]

Tris(morpholine-4-dithiocarboxylato-$\kappa^2 S,S'$)antimony(III)

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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). As a contribution to the chemistry of main-group metal complexes with dithiocarbamates, we present here the synthesis and crystal structure of the title compound (I).

In (I) (Fig. 1), the Sb^{III} ion is coordinated by the six S atoms [Sb—S 2.474 (2)–2.833 (2) Å] belonging to three morpholine-4-dithiocarboxylate ligands. The resulting SbS_6 coordination polyhedron approximates to a pentagonal pyramid with atoms Sb1 and S2—S6 at the base of the pyramid and S1 in the apical position. Three of the five equatorial Sb—S bonds are long (to S2, S3 and S5), two are short (to S4 and S6), while the apical donor atom forms the strongest bond [Sb1—S1=2.474 (2)]. The Sb^{III} lone pair of electrons may project in a direction roughly *trans* to the Sb1—S1 bond. The short intermolecular distance Sb1···$S3^{i}$ of 3.411 (2) Å suggests a presence of Sb···S interactions (sSymmetry code as in Fig. 2), which lead to the dimeric associations in the crystal (Fig. 2).

Experimental

The title compound were prepared by reaction of antimony tribromide (36.1 mg, 0.1 mmol) with the corresponding sodium dithiocarbamate (36.3 mg, 0.3 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 methanol/dichloromethane (1:2 *v*/*v*) solution over a period of two weeks (yield 90%. m.p. 435 Kk).

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.97 Å for methylene H atoms, and with $U_{iso}(H) = 1.2U_{eq}(C)$ for C-bound H atoms.

Figures

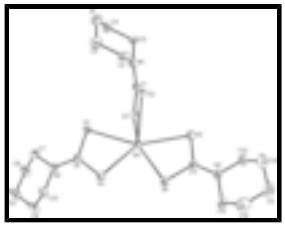

Fig. 1. The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering schems. H atoms have been omitted for clarity.

Fig. 2. Crystal packing of (I), showing the Sb···S interactions as dashed lines. [Symmetry code: (i) −*x*,-*y*,-*z*.]

Tris(morpholine-4-dithiocarboxylato-κ^2*S*,*S*')antimony(III)

Crystal data

$[Sb(C_5H_8NOS_2)_3]$	F_{000} = 1224
M_r = 608.48	D_x = 1.746 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2654 reflections
a = 12.8891 (13) Å	θ = 2.5–25.3º
b = 20.009 (2) Å	μ = 1.76 mm^{-1}
c = 9.0073 (9) Å	T = 298 (2) K
β = 94.734 (2)º	Block, yellow
V = 2315.0 (4) Å^3	0.59 × 0.20 × 0.08 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4056 independent reflections
Radiation source: fine-focus sealed tube	2716 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.064
T = 298(2) K	θ_{max} = 25.0º
phi and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −15→12
T_{min} = 0.424, T_{max} = 0.872	k = −23→22
11465 measured reflections	l = −10→10

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H-atom parameters constrained
$wR(F^2)$ = 0.111	$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
4056 reflections	$\Delta\rho_{max}$ = 0.88 e Å^{-3}
253 parameters	$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Sb1	0.15201 (3)	0.046775 (19)	0.10064 (5)	0.03166 (16)
N1	0.3477 (3)	−0.1181 (2)	−0.0361 (6)	0.0331 (12)
N2	0.0485 (4)	0.1343 (2)	−0.3489 (6)	0.0398 (14)
N3	0.3273 (4)	0.1164 (2)	0.5276 (6)	0.0485 (15)
O1	0.4047 (4)	−0.1910 (2)	−0.2841 (5)	0.0554 (13)
O2	−0.1021 (4)	0.2012 (2)	−0.5360 (6)	0.0685 (16)
O3	0.4797 (4)	0.1579 (3)	0.7463 (6)	0.0753 (17)
S1	0.32072 (12)	0.01042 (7)	0.01547 (19)	0.0345 (4)
S2	0.16824 (12)	−0.09393 (8)	0.07939 (19)	0.0388 (4)
S3	0.07604 (12)	0.02040 (7)	−0.19772 (19)	0.0352 (4)
S4	0.15521 (13)	0.15062 (7)	−0.08730 (19)	0.0391 (4)
S5	0.24492 (15)	0.01171 (8)	0.3820 (2)	0.0473 (5)
S6	0.24826 (15)	0.14712 (8)	0.2552 (2)	0.0478 (5)
C1	0.2839 (4)	−0.0739 (3)	0.0153 (6)	0.0281 (14)
C2	0.4407 (5)	−0.1006 (3)	−0.1099 (8)	0.0446 (18)
H2A	0.5011	−0.1227	−0.0608	0.054*
H2B	0.4522	−0.0527	−0.1046	0.054*
C3	0.4259 (6)	−0.1222 (4)	−0.2688 (8)	0.056 (2)
H3A	0.3687	−0.0971	−0.3186	0.067*
H3B	0.4882	−0.1118	−0.3176	0.067*
C4	0.3158 (5)	−0.2091 (3)	−0.2104 (7)	0.0483 (18)
H4A	0.3059	−0.2571	−0.2180	0.058*
H4B	0.2549	−0.1878	−0.2600	0.058*
C5	0.3251 (5)	−0.1894 (3)	−0.0484 (7)	0.0404 (17)
H5A	0.2605	−0.1992	−0.0046	0.048*
H5B	0.3804	−0.2147	0.0050	0.048*
C6	0.0875 (4)	0.1039 (3)	−0.2247 (7)	0.0310 (14)
C7	−0.0106 (5)	0.0991 (3)	−0.4706 (8)	0.0520 (19)
H7A	−0.0212	0.0530	−0.4424	0.062*
H7B	0.0281	0.0995	−0.5585	0.062*
C8	−0.1123 (6)	0.1322 (3)	−0.5040 (9)	0.061 (2)
H8A	−0.1492	0.1103	−0.5888	0.073*
H8B	−0.1536	0.1271	−0.4194	0.073*
C9	−0.0489 (5)	0.2341 (3)	−0.4159 (9)	0.060 (2)
H9A	−0.0891	0.2308	−0.3298	0.072*
H9B	−0.0425	0.2811	−0.4402	0.072*
C10	0.0562 (5)	0.2058 (3)	−0.3781 (8)	0.0462 (18)
H10A	0.0992	0.2131	−0.4600	0.055*
H10B	0.0888	0.2281	−0.2907	0.055*
C11	0.2771 (5)	0.0935 (3)	0.4024 (7)	0.0341 (15)
C12	0.3629 (6)	0.0738 (3)	0.6512 (8)	0.059 (2)
H12A	0.3596	0.0275	0.6199	0.070*
H12B	0.3180	0.0794	0.7316	0.070*

C13	0.4711 (6)	0.0911 (4)	0.7045 (9)	0.069 (2)
H13A	0.4935	0.0631	0.7890	0.083*
H13B	0.5165	0.0824	0.6261	0.083*
C14	0.4465 (7)	0.1995 (4)	0.6268 (11)	0.088 (3)
H14A	0.4951	0.1950	0.5507	0.105*
H14B	0.4501	0.2454	0.6614	0.105*
C15	0.3438 (6)	0.1874 (3)	0.5596 (8)	0.058 (2)
H15A	0.3332	0.2127	0.4677	0.070*
H15B	0.2931	0.2026	0.6260	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.0340 (3)	0.0308 (2)	0.0303 (3)	−0.00067 (18)	0.00391 (18)	−0.00238 (19)
N1	0.026 (3)	0.033 (3)	0.042 (3)	−0.001 (2)	0.007 (2)	−0.003 (2)
N2	0.046 (3)	0.030 (3)	0.041 (4)	0.000 (2)	−0.011 (3)	0.004 (3)
N3	0.075 (4)	0.031 (3)	0.035 (4)	−0.014 (3)	−0.017 (3)	0.003 (3)
O1	0.072 (3)	0.052 (3)	0.044 (3)	0.004 (3)	0.017 (3)	−0.010 (2)
O2	0.071 (4)	0.049 (3)	0.080 (4)	−0.004 (3)	−0.030 (3)	0.017 (3)
O3	0.084 (4)	0.076 (4)	0.062 (4)	−0.025 (3)	−0.022 (3)	0.005 (3)
S1	0.0309 (9)	0.0300 (8)	0.0432 (11)	−0.0040 (7)	0.0070 (8)	0.0009 (7)
S2	0.0384 (10)	0.0340 (9)	0.0459 (12)	−0.0059 (7)	0.0146 (8)	−0.0017 (8)
S3	0.0397 (9)	0.0282 (8)	0.0366 (10)	−0.0016 (7)	−0.0041 (8)	−0.0003 (7)
S4	0.0501 (10)	0.0301 (8)	0.0351 (11)	−0.0022 (7)	−0.0086 (8)	−0.0017 (7)
S5	0.0728 (13)	0.0321 (9)	0.0356 (11)	−0.0122 (8)	−0.0032 (10)	0.0030 (8)
S6	0.0751 (13)	0.0305 (9)	0.0350 (11)	−0.0057 (8)	−0.0128 (10)	0.0035 (8)
C1	0.024 (3)	0.030 (3)	0.030 (4)	0.000 (3)	−0.001 (3)	0.003 (3)
C2	0.035 (4)	0.044 (4)	0.056 (5)	0.004 (3)	0.012 (4)	−0.009 (4)
C3	0.058 (5)	0.060 (5)	0.053 (5)	0.003 (4)	0.021 (4)	0.003 (4)
C4	0.061 (5)	0.045 (4)	0.037 (5)	−0.002 (3)	−0.010 (4)	−0.003 (3)
C5	0.051 (4)	0.026 (3)	0.045 (5)	0.007 (3)	0.008 (3)	−0.003 (3)
C6	0.026 (3)	0.036 (3)	0.031 (4)	0.008 (3)	−0.004 (3)	−0.001 (3)
C7	0.073 (5)	0.042 (4)	0.038 (5)	0.004 (4)	−0.014 (4)	−0.008 (3)
C8	0.069 (5)	0.047 (4)	0.062 (6)	−0.013 (4)	−0.028 (4)	0.012 (4)
C9	0.059 (5)	0.035 (4)	0.082 (6)	0.007 (3)	−0.009 (5)	0.012 (4)
C10	0.052 (4)	0.037 (4)	0.047 (5)	−0.009 (3)	−0.013 (4)	0.011 (3)
C11	0.045 (4)	0.032 (3)	0.026 (4)	0.000 (3)	0.005 (3)	−0.002 (3)
C12	0.086 (6)	0.045 (4)	0.042 (5)	−0.019 (4)	−0.019 (4)	0.010 (4)
C13	0.072 (6)	0.085 (7)	0.047 (6)	−0.008 (5)	−0.015 (4)	0.009 (5)
C14	0.103 (8)	0.057 (5)	0.098 (8)	−0.035 (5)	−0.020 (6)	0.002 (5)
C15	0.082 (6)	0.043 (4)	0.045 (5)	−0.010 (4)	−0.019 (4)	−0.006 (3)

Geometric parameters (Å, °)

Sb1—S1	2.4742 (16)	C2—H2A	0.9700
Sb1—S4	2.6826 (16)	C2—H2B	0.9700
Sb1—S6	2.6876 (17)	C3—H3A	0.9700
Sb1—S5	2.8015 (18)	C3—H3B	0.9700
Sb1—S2	2.8307 (16)	C4—C5	1.507 (9)

Sb1—S3	2.8329 (17)	C4—H4A	0.9700
Sb1—S3^{i}	3.4111 (17)	C4—H4B	0.9700
N1—C1	1.316 (7)	C5—H5A	0.9700
N1—C5	1.459 (7)	C5—H5B	0.9700
N1—C2	1.460 (7)	C7—C8	1.477 (9)
N2—C6	1.335 (7)	C7—H7A	0.9700
N2—C10	1.460 (7)	C7—H7B	0.9700
N2—C7	1.462 (7)	C8—H8A	0.9700
N3—C11	1.334 (7)	C8—H8B	0.9700
N3—C12	1.446 (8)	C9—C10	1.482 (8)
N3—C15	1.461 (8)	C9—H9A	0.9700
O1—C3	1.409 (8)	C9—H9B	0.9700
O1—C4	1.418 (8)	C10—H10A	0.9700
O2—C9	1.397 (8)	C10—H10B	0.9700
O2—C8	1.419 (7)	C12—C13	1.478 (9)
O3—C13	1.390 (9)	C12—H12A	0.9700
O3—C14	1.399 (9)	C12—H12B	0.9700
S1—C1	1.753 (6)	C13—H13A	0.9700
S2—C1	1.691 (6)	C13—H13B	0.9700
S3—C6	1.696 (6)	C14—C15	1.431 (9)
S4—C6	1.729 (6)	C14—H14A	0.9700
S5—C11	1.695 (6)	C14—H14B	0.9700
S6—C11	1.722 (6)	C15—H15A	0.9700
C2—C3	1.492 (9)	C15—H15B	0.9700
S1—Sb1—S4	88.42 (5)	N1—C5—C4	109.3 (5)
S1—Sb1—S6	90.02 (6)	N1—C5—H5A	109.8
S4—Sb1—S6	73.60 (5)	C4—C5—H5A	109.8
S1—Sb1—S5	83.44 (6)	N1—C5—H5B	109.8
S4—Sb1—S5	137.49 (5)	C4—C5—H5B	109.8
S6—Sb1—S5	64.78 (5)	H5A—C5—H5B	108.3
S1—Sb1—S2	67.35 (5)	N2—C6—S3	122.5 (4)
S4—Sb1—S2	136.27 (5)	N2—C6—S4	119.1 (4)
S6—Sb1—S2	138.11 (5)	S3—C6—S4	118.4 (3)
S5—Sb1—S2	77.50 (5)	N2—C7—C8	109.7 (6)
S1—Sb1—S3	83.97 (5)	N2—C7—H7A	109.7
S4—Sb1—S3	64.39 (5)	C8—C7—H7A	109.7
S6—Sb1—S3	137.66 (5)	N2—C7—H7B	109.7
S5—Sb1—S3	154.16 (5)	C8—C7—H7B	109.7
S2—Sb1—S3	76.84 (5)	H7A—C7—H7B	108.2
S1—Sb1—S3^{i}	139.61 (4)	O2—C8—C7	112.4 (6)
S4—Sb1—S3^{i}	121.70 (5)	O2—C8—H8A	109.1
S6—Sb1—S3^{i}	122.16 (5)	C7—C8—H8A	109.1
S5—Sb1—S3^{i}	88.99 (5)	O2—C8—H8B	109.1
S2—Sb1—S3^{i}	72.27 (4)	C7—C8—H8B	109.1
S3—Sb1—S3^{i}	86.11 (5)	H8A—C8—H8B	107.9
C1—N1—C5	123.8 (5)	O2—C9—C10	112.3 (6)
C1—N1—C2	124.0 (5)	O2—C9—H9A	109.2

C5—N1—C2	111.4 (5)	C10—C9—H9A	109.2
C6—N2—C10	124.8 (5)	O2—C9—H9B	109.2
C6—N2—C7	123.1 (5)	C10—C9—H9B	109.2
C10—N2—C7	112.0 (5)	H9A—C9—H9B	107.9
C11—N3—C12	123.4 (5)	N2—C10—C9	110.0 (5)
C11—N3—C15	123.5 (5)	N2—C10—H10A	109.7
C12—N3—C15	112.9 (5)	C9—C10—H10A	109.7
C3—O1—C4	111.2 (5)	N2—C10—H10B	109.7
C9—O2—C8	110.4 (5)	C9—C10—H10B	109.7
C13—O3—C14	110.6 (6)	H10A—C10—H10B	108.2
C1—S1—Sb1	92.21 (19)	N3—C11—S5	121.3 (5)
C1—S2—Sb1	81.9 (2)	N3—C11—S6	119.7 (4)
C6—S3—Sb1	85.7 (2)	S5—C11—S6	118.9 (4)
C6—S4—Sb1	90.0 (2)	N3—C12—C13	110.1 (6)
C11—S5—Sb1	86.5 (2)	N3—C12—H12A	109.6
C11—S6—Sb1	89.7 (2)	C13—C12—H12A	109.6
N1—C1—S2	123.7 (4)	N3—C12—H12B	109.6
N1—C1—S1	118.0 (4)	C13—C12—H12B	109.6
S2—C1—S1	118.3 (3)	H12A—C12—H12B	108.2
N1—C2—C3	108.8 (5)	O3—C13—C12	111.4 (7)
N1—C2—H2A	109.9	O3—C13—H13A	109.3
C3—C2—H2A	109.9	C12—C13—H13A	109.3
N1—C2—H2B	109.9	O3—C13—H13B	109.3
C3—C2—H2B	109.9	C12—C13—H13B	109.3
H2A—C2—H2B	108.3	H13A—C13—H13B	108.0
O1—C3—C2	112.7 (6)	O3—C14—C15	116.0 (7)
O1—C3—H3A	109.1	O3—C14—H14A	108.3
C2—C3—H3A	109.1	C15—C14—H14A	108.3
O1—C3—H3B	109.1	O3—C14—H14B	108.3
C2—C3—H3B	109.1	C15—C14—H14B	108.3
H3A—C3—H3B	107.8	H14A—C14—H14B	107.4
O1—C4—C5	112.6 (5)	C14—C15—N3	111.2 (6)
O1—C4—H4A	109.1	C14—C15—H15A	109.4
C5—C4—H4A	109.1	N3—C15—H15A	109.4
O1—C4—H4B	109.1	C14—C15—H15B	109.4
C5—C4—H4B	109.1	N3—C15—H15B	109.4
H4A—C4—H4B	107.8	H15A—C15—H15B	108.0

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

Fig. 1

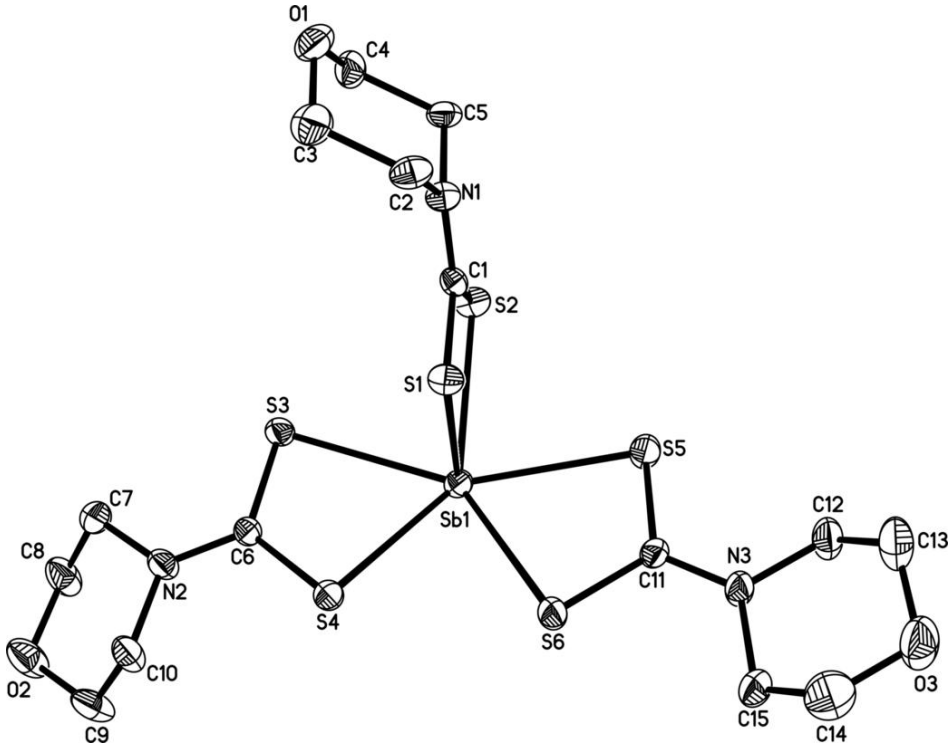
O1
C4
C5
C3
C2
N1
C1
S2
S1
S3
S5
Sb1
C7
C8
C6
N2
S4
S6
C11
N3
C12
C13
O2
C10
C9
C15
C14
O3

Fig. 2