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

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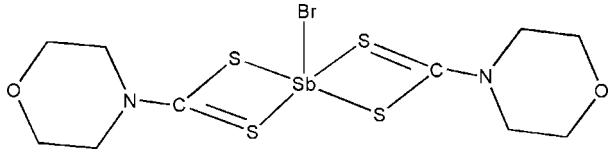
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.015 Å; R factor = 0.052; wR factor = 0.160; data-to-parameter ratio = 16.8.

In the title compound, $[SbBr(C_5H_8NOS_2)_2]$, both organic ligands bond to Sb in an S,S'-bidentate mode, although one of the Sb—S bond lengths is much longer than the other three. A bromide ion completes the very distorted trigonal–bipyramidal geometry about the Sb atom.

Related literature

For background, see: Sheng *et al.* (1999).



Experimental

Crystal data

$[SbBr(C_5H_8NOS_2)_2]$
M_r = 526.15
Triclinic, $P\overline{1}$
a = 6.428 (2) Å
b = 11.175 (4) Å
c = 13.241 (4) Å
α = 71.060 (4)°
β = 81.796 (4)°
γ = 76.924 (3)°
V = 873.8 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.34 mm^{-1}
T = 293 (2) K
0.48 × 0.46 × 0.43 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.230, T_{max} = 0.257
(expected range = 0.138–0.155)
4592 measured reflections
3044 independent reflections
2616 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.160
S = 1.03
3044 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.02 e Å^{-3}
$\Delta\rho_{min}$ = −1.04 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sb1—S3	2.469 (2)	Sb1—S4	2.909 (2)
Sb1—S1	2.542 (2)	Sb1—Br1	2.8087 (14)
Sb1—S2	2.621 (2)		
S3—Sb1—S1	89.32 (8)	S2—Sb1—Br1	150.27 (6)
S3—Sb1—S2	92.18 (8)	S3—Sb1—S4	66.17 (7)
S1—Sb1—S2	69.49 (7)	S1—Sb1—S4	139.75 (7)
S3—Sb1—Br1	83.76 (6)	S2—Sb1—S4	79.63 (7)
S1—Sb1—Br1	80.99 (6)	Br1—Sb1—S4	124.28 (6)

Data collection: *SMART* (Bruker 1998); cell refinement: *SAINT* (Bruker 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 1998); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2665).

References

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supplementary materials

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

Y. Sun, Y. Li, X. Cui and X. Lang

Comment

Dialkyl-substituted dithiocarbamate anions have proved to be highly versatile chelating agents for the separation of metals as metal chelates using gas chromatography. Some dialkyl-substituted dithiocarbamate salts have also shown interesting biological effects which include anti-alkylation or anti-HIV properties (Sheng *et al.*, 1999). Here, we report the synthesis and structure of the title compound, (I).

The Sb atom is five-coordinated with a distorted trigonal bipyramidal geometry (Table 1, Fig. 1). Around the central Sb atom, atoms S1, S3, S4 occupy the equatorial plane, while Br1 and S2 lie in axial sites. The axial bond angle [150.27 (6)°] deviates from linearity by over 29°. The sum of the S3—Sb1—S4 [66.17 (7)°], S3—Sb1—S1 [89.32 (8)°] and S1—Sb1—S4 [139.75 (7)°] bond angles is 295.2°, which shows that these atoms have large deviations from ideal trigonal bipyramidal geometry. The C—S bonds associated with the strong Sb—S bonds are significantly longer than that associated with the weak Sb—S bonds, suggesting some delocation in the system.

In the crystal, a two-dimensional chain network arises from intermolecular weak Sb···*S* and *S*···S contacts (Fig. 2).

Experimental

Morpholinyldithiocarbamate (371 mg, 2 mmol) was added to a stirring solution containing tribromoantimony (362 mg, 1 mmol) in ethanol (80 ml). After stirring for 8 h at room temperature, a yellow solution was obtained and then filtered. The resulting solution was evaporated under vacuum until the title compound was obtaibed as a yellow solid, which was recrystallized from methanol/dichloromethane (2:1 *v*/*v*) to give yellow blocks of (I); yield 76%, m.p. 507 K. Anal. Calcd (%) for $C_{10}H_{16}BrN_2O_2S_4Sb$: C 22.83; H 3.06; N 5.32; Found: C 22.71; H 3.19; N 5.49.

Refinement

The H atoms were positioned geometrically (C—H = 0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

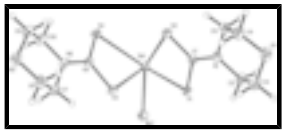

Fig. 1. The molecular structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level.

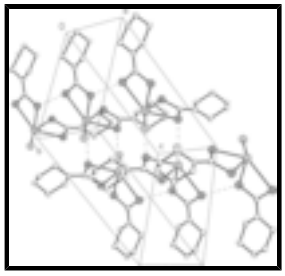

Fig. 2. A packing diagram for (I). H atoms have been omitted for clarity.

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

Crystal data

$[SbBr(C_5H_8NOS_2)_2]$	$Z = 2$
$M_r = 526.15$	$F_{000} = 512$
Triclinic, $P\overline{1}$	$D_x = 2.000$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.428$ (2) Å	Cell parameters from 2859 reflections
$b = 11.175$ (4) Å	$\theta = 2.9$–28.0°
$c = 13.241$ (4) Å	$\mu = 4.34$ mm^{-1}
$\alpha = 71.060$ (4)°	$T = 293$ (2) K
$\beta = 81.796$ (4)°	Block, yellow
$\gamma = 76.924$ (3)°	0.48 × 0.46 × 0.43 mm
$V = 873.8$ (5) Å^3	

Data collection

Bruker SMART CCD diffractometer	3044 independent reflections
Radiation source: fine-focus sealed tube	2616 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 293$(2) K	$\theta_{max} = 25.0^\circ$
ω scans	$\theta_{min} = 2.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -4 \rightarrow 7$
$T_{min} = 0.230$, $T_{max} = 0.257$	$k = -13 \rightarrow 13$
4592 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H-atom parameters constrained
$wR(F^2) = 0.160$	$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2 + 5.9643P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
3044 reflections	$\Delta\rho_{max}$ = 2.02 e Å^{-3}
181 parameters	$\Delta\rho_{min}$ = −1.04 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}
Sb1	−0.00045 (9)	0.46615 (5)	0.32671 (4)	0.0291 (2)
Br1	0.16432 (17)	0.69319 (9)	0.27216 (8)	0.0455 (3)
N1	0.2808 (12)	0.1526 (7)	0.5928 (6)	0.0327 (16)
N2	0.2041 (11)	0.3082 (7)	0.0529 (6)	0.0326 (16)
O1	0.5164 (13)	−0.0596 (7)	0.7403 (6)	0.0519 (18)
O2	0.3961 (12)	0.1478 (7)	−0.0739 (5)	0.0477 (17)
S1	0.2747 (4)	0.3940 (2)	0.46525 (18)	0.0347 (5)
S2	0.0126 (4)	0.2235 (2)	0.43813 (19)	0.0402 (6)
S3	0.2845 (3)	0.4096 (2)	0.19460 (18)	0.0336 (5)
S4	−0.1470 (3)	0.3730 (2)	0.17329 (19)	0.0375 (5)
C1	0.1961 (13)	0.2443 (8)	0.5102 (7)	0.0299 (18)
C2	0.4261 (16)	0.1714 (9)	0.6596 (8)	0.041 (2)
H2A	0.3463	0.1874	0.7234	0.049*
H2B	0.4890	0.2461	0.6204	0.049*
C3	0.6012 (17)	0.0538 (9)	0.6913 (9)	0.047 (2)
H3A	0.6928	0.0451	0.6282	0.056*
H3B	0.6882	0.0647	0.7406	0.056*
C4	0.3955 (18)	−0.0798 (9)	0.6673 (9)	0.048 (3)
H4A	0.3447	−0.1602	0.6996	0.057*
H4B	0.4857	−0.0857	0.6031	0.057*
C5	0.2063 (16)	0.0308 (9)	0.6381 (8)	0.042 (2)
H5A	0.1272	0.0182	0.5864	0.050*
H5B	0.1108	0.0329	0.7015	0.050*
C6	0.1182 (13)	0.3570 (7)	0.1320 (6)	0.0270 (17)
C7	0.4269 (14)	0.3060 (9)	0.0091 (8)	0.038 (2)
H7A	0.5094	0.3198	0.0588	0.046*
H7B	0.4328	0.3750	−0.0579	0.046*

C8	0.5227 (16)	0.1772 (9)	−0.0101 (8)	0.041 (2)
H8A	0.6655	0.1798	−0.0452	0.049*
H8B	0.5349	0.1099	0.0582	0.049*
C9	0.1872 (17)	0.1407 (10)	−0.0235 (8)	0.046 (2)
H9A	0.1969	0.0745	0.0455	0.055*
H9B	0.1049	0.1163	−0.0670	0.055*
C10	0.0739 (17)	0.2665 (11)	−0.0077 (9)	0.049 (3)
H10A	0.0500	0.3311	−0.0767	0.059*
H10B	−0.0643	0.2572	0.0311	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.0306 (3)	0.0284 (3)	0.0290 (3)	−0.0039 (2)	−0.0028 (2)	−0.0107 (2)
Br1	0.0604 (7)	0.0351 (5)	0.0466 (6)	−0.0170 (5)	−0.0012 (5)	−0.0156 (4)
N1	0.035 (4)	0.030 (4)	0.032 (4)	−0.009 (3)	−0.007 (3)	−0.004 (3)
N2	0.027 (4)	0.036 (4)	0.042 (4)	−0.005 (3)	−0.006 (3)	−0.020 (3)
O1	0.069 (5)	0.037 (4)	0.047 (4)	−0.010 (3)	−0.025 (4)	0.000 (3)
O2	0.058 (4)	0.050 (4)	0.043 (4)	−0.010 (3)	0.000 (3)	−0.026 (3)
S1	0.0436 (13)	0.0311 (11)	0.0342 (11)	−0.0125 (9)	−0.0104 (9)	−0.0099 (9)
S2	0.0489 (14)	0.0360 (12)	0.0401 (13)	−0.0165 (10)	−0.0160 (10)	−0.0067 (10)
S3	0.0260 (11)	0.0441 (12)	0.0407 (12)	−0.0111 (9)	−0.0009 (9)	−0.0243 (10)
S4	0.0253 (11)	0.0490 (13)	0.0432 (13)	−0.0070 (10)	−0.0045 (9)	−0.0200 (11)
C1	0.027 (4)	0.033 (4)	0.032 (4)	−0.009 (3)	−0.001 (3)	−0.011 (4)
C2	0.053 (6)	0.034 (5)	0.039 (5)	−0.005 (4)	−0.015 (4)	−0.013 (4)
C3	0.047 (6)	0.043 (5)	0.053 (6)	−0.010 (5)	−0.023 (5)	−0.009 (5)
C4	0.065 (7)	0.033 (5)	0.045 (6)	−0.014 (5)	−0.021 (5)	−0.001 (4)
C5	0.046 (6)	0.034 (5)	0.044 (5)	−0.017 (4)	−0.005 (4)	−0.002 (4)
C6	0.028 (4)	0.023 (4)	0.031 (4)	−0.008 (3)	−0.008 (3)	−0.007 (3)
C7	0.035 (5)	0.041 (5)	0.042 (5)	−0.011 (4)	−0.004 (4)	−0.016 (4)
C8	0.044 (5)	0.045 (5)	0.035 (5)	−0.004 (4)	−0.003 (4)	−0.017 (4)
C9	0.055 (6)	0.045 (6)	0.050 (6)	−0.011 (5)	−0.011 (5)	−0.025 (5)
C10	0.044 (6)	0.063 (7)	0.055 (6)	−0.007 (5)	−0.013 (5)	−0.036 (5)

Geometric parameters (Å, °)

Sb1—S3	2.469 (2)	C2—H2A	0.9700
Sb1—S1	2.542 (2)	C2—H2B	0.9700
Sb1—S2	2.621 (2)	C3—H3A	0.9700
Sb1—S4	2.909 (2)	C3—H3B	0.9700
Sb1—Br1	2.8087 (14)	C4—C5	1.517 (14)
N1—C1	1.317 (11)	C4—H4A	0.9700
N1—C5	1.458 (11)	C4—H4B	0.9700
N1—C2	1.464 (12)	C5—H5A	0.9700
N2—C6	1.330 (11)	C5—H5B	0.9700
N2—C7	1.464 (11)	C7—C8	1.518 (13)
N2—C10	1.474 (12)	C7—H7A	0.9700
O1—C3	1.414 (12)	C7—H7B	0.9700
O1—C4	1.423 (12)	C8—H8A	0.9700

O2—C8	1.408 (12)	C8—H8B	0.9700
O2—C9	1.419 (12)	C9—C10	1.495 (14)
S1—C1	1.748 (9)	C9—H9A	0.9700
S2—C1	1.716 (9)	C9—H9B	0.9700
S3—C6	1.740 (8)	C10—H10A	0.9700
S4—C6	1.702 (8)	C10—H10B	0.9700
C2—C3	1.510 (13)		
S3—Sb1—S1	89.32 (8)	O1—C4—H4A	109.7
S3—Sb1—S2	92.18 (8)	C5—C4—H4A	109.7
S1—Sb1—S2	69.49 (7)	O1—C4—H4B	109.7
S3—Sb1—Br1	83.76 (6)	C5—C4—H4B	109.7
S1—Sb1—Br1	80.99 (6)	H4A—C4—H4B	108.2
S2—Sb1—Br1	150.27 (6)	N1—C5—C4	110.0 (8)
S3—Sb1—S4	66.17 (7)	N1—C5—H5A	109.7
S1—Sb1—S4	139.75 (7)	C4—C5—H5A	109.7
S2—Sb1—S4	79.63 (7)	N1—C5—H5B	109.7
Br1—Sb1—S4	124.28 (6)	C4—C5—H5B	109.7
C1—N1—C5	122.2 (8)	H5A—C5—H5B	108.2
C1—N1—C2	123.5 (7)	N2—C6—S4	123.2 (6)
C5—N1—C2	113.3 (7)	N2—C6—S3	118.3 (6)
C6—N2—C7	123.8 (7)	S4—C6—S3	118.5 (5)
C6—N2—C10	122.0 (7)	N2—C7—C8	110.1 (7)
C7—N2—C10	113.7 (7)	N2—C7—H7A	109.6
C3—O1—C4	110.0 (7)	C8—C7—H7A	109.6
C8—O2—C9	110.7 (7)	N2—C7—H7B	109.6
C1—S1—Sb1	87.9 (3)	C8—C7—H7B	109.6
C1—S2—Sb1	86.0 (3)	H7A—C7—H7B	108.1
C6—S3—Sb1	94.4 (3)	O2—C8—C7	111.4 (8)
C6—S4—Sb1	80.8 (3)	O2—C8—H8A	109.3
N1—C1—S2	122.9 (6)	C7—C8—H8A	109.3
N1—C1—S1	120.7 (6)	O2—C8—H8B	109.3
S2—C1—S1	116.3 (5)	C7—C8—H8B	109.3
N1—C2—C3	110.4 (7)	H8A—C8—H8B	108.0
N1—C2—H2A	109.6	O2—C9—C10	111.6 (9)
C3—C2—H2A	109.6	O2—C9—H9A	109.3
N1—C2—H2B	109.6	C10—C9—H9A	109.3
C3—C2—H2B	109.6	O2—C9—H9B	109.3
H2A—C2—H2B	108.1	C10—C9—H9B	109.3
O1—C3—C2	111.6 (8)	H9A—C9—H9B	108.0
O1—C3—H3A	109.3	N2—C10—C9	109.7 (8)
C2—C3—H3A	109.3	N2—C10—H10A	109.7
O1—C3—H3B	109.3	C9—C10—H10A	109.7
C2—C3—H3B	109.3	N2—C10—H10B	109.7
H3A—C3—H3B	108.0	C9—C10—H10B	109.7
O1—C4—C5	109.9 (9)	H10A—C10—H10B	108.2

Fig. 1

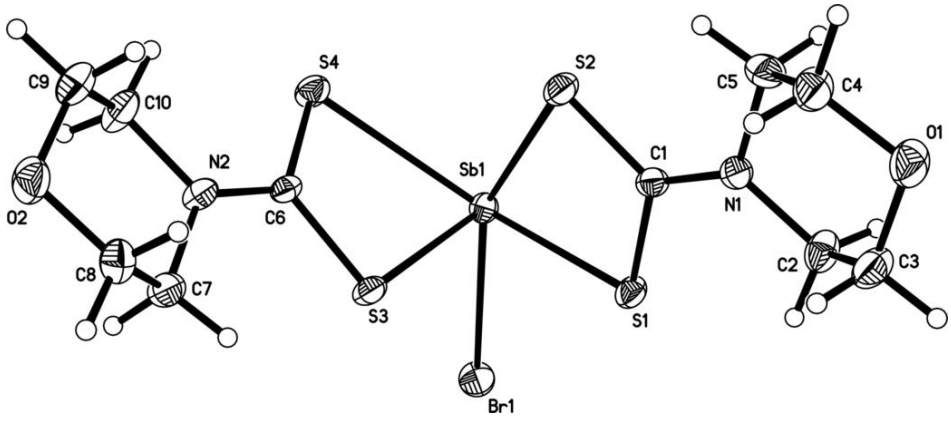

Fig. 2