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

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In the title compound, [$SbBr(C_5H_8NS_2)_2$], the Sb^{III} ion is coordinated by the four S atoms [Sb—S = 2.4616 (11)–2.9194 (13) Å] from two pyrrolidine-1-dithiocarboxylate ligands and a Br atom [Sb—Br = 2.8159 (7) Å] in a distorted trigonal–bipyramidal geometry. In the crystal structure, the molecules are associated into dimers with short intermolecular Sb⋯S contacts of 3.7350 (13) Å.

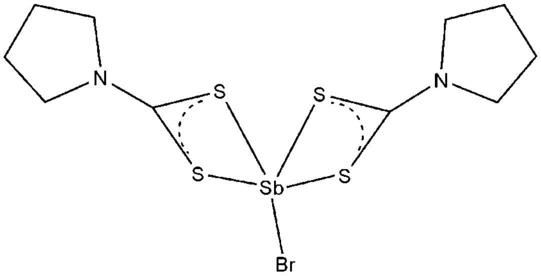
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.027
wR factor = 0.073
Data-to-parameter ratio = 17.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Dithiocarbamates have been known as effective ligands for transition metal ions; they 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 report here the synthesis and crystal structure of the title compound, (I).



(I)

In (I) (Fig. 1), the Sb^{III} ion is coordinated by the four S atoms from two pyrrolidine-1-dithiocarboxylate ligands and a Br atom in a distorted trigonal–bipyramidal geometry

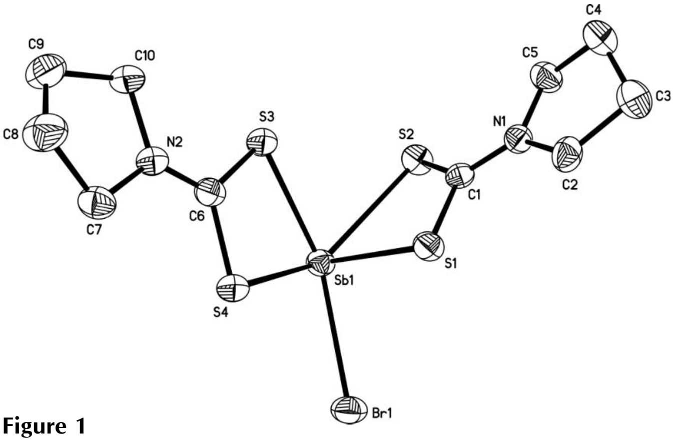


Figure 1
The molecular structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

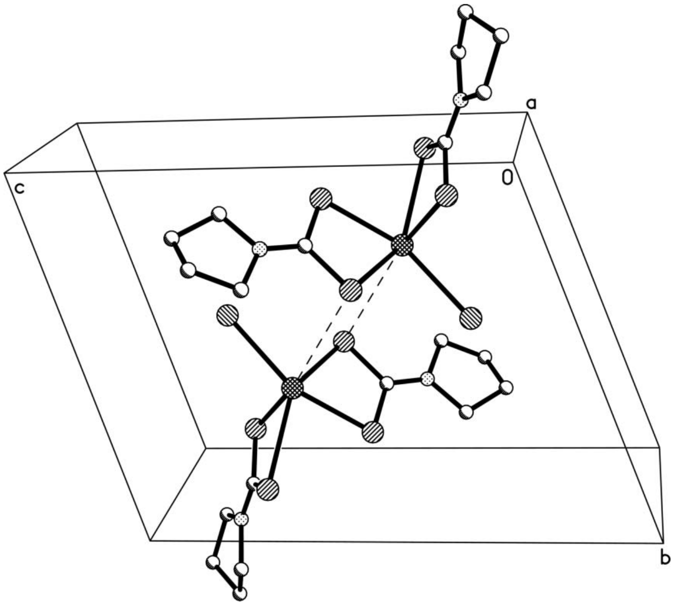


Figure 2
A portion of the crystal packing, showing the intemolecular Sb$\cdots$S interactions as dashed lines. H atoms have been omitted for clarity.

(Table 1). The short intermolecular distance Sb1$\cdots$S4^{i} of 3.7350 (13) Å suggests the presence of Sb$\cdots$S interactions [symmetry code: (i) $1 - x, 1 - y, 1 - z$], which lead to dimeric associations in the crystal structure (Fig. 2).

Experimental

The title compound was prepared by the reaction of antimony tribromide (0.1 mmol) with sodium dithiocarbamate (0.2 mmol) in absolute acetone (30 ml). After stirring for 5 h at room temperature, a yellow paste was obtained. It was dissolved in 10 ml of ethanol, and dichloromethane (10 ml) was added. The resulting mixture was filtered and recrystallized from ethanol/dichloromethane (1:1) to give yellow crystals (yield 80%, m.p. 430 K).

Crystal data

$[\mathrm{SbBr(C_5H_8NS_2)_2}]$
$M_r = 494.19$
Triclinic, $P\bar{1}$
$a = 6.4655$ (15) Å
$b = 10.471$ (2) Å
$c = 13.394$ (3) Å
$\alpha = 110.716$ (3)°
$\beta = 92.361$ (4)°
$\gamma = 102.869$ (3)°
$V = 819.6$ (3) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 4.62$ mm^{-1}
$T = 298$ (2) K
$0.30 \times 0.13 \times 0.07$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{\min} = 0.338$, $T_{\max} = 0.738$
4258 measured reflections
2846 independent reflections
2398 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.00$
2846 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.57$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sb1—S1	2.4616 (11)	Sb1—Br1	2.8159 (7)
Sb1—S4	2.5606 (12)	Sb1—S2	2.9194 (13)
Sb1—S3	2.5980 (11)		
S1—Sb1—S4	91.05 (4)	S3—Sb1—Br1	151.34 (3)
S1—Sb1—S3	91.90 (4)	S1—Sb1—S2	66.56 (3)
S4—Sb1—S3	69.88 (4)	S4—Sb1—S2	139.34 (3)
S1—Sb1—Br1	84.31 (3)	S3—Sb1—S2	77.13 (4)
S4—Sb1—Br1	81.77 (3)	Br1—Sb1—S2	125.99 (3)

All H atoms were positioned geometrically with methylene C—H distances of 0.97 Å, and treated as riding on their parent atoms, with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$.

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*.

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