Tris(*N*-cyclohexyl-*N*-methyldithiocarbamato-*S*)-antimony(III)

Ibrahim Baba,[a] Sukeria Ibrahim,[a] Yang Farina,[a] A. Hamid Othman,[a] Ibrahim Abdul Razak,[b] Hoong-Kun Fun[b] and Seik Weng Ng[c]*

[a]School of Chemical Sciences, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia, [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]Institute of Postgraduate Studies, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail:
h1nswen@umcsd.um.edu.my

Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.026
wR factor = 0.054
Data-to-parameter ratio = 38.2

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

In the title compound, [Sb(C$_8$H$_{14}$NS$_2$)$_3$], the dithiocarbamate groups chelate to the Sb atom in an anisobidentate manner [Sb—S = 2.530 (1) and 2.975 (1) Å]. The Sb atom lies on a threefold axis and the lone pair is also stereochemically active.

Comment

The electron lone-pair is stereochemically active in antimony(III) and bismuth(III) tri(diethyldithiocarbamates); the dithiocarbamate group coordinates to metal atom in an anisobidentate manner and the covalent is shorter than the dative distance (Raston & White, 1976). In the title compound, (I), the dithiocarbamate anions chelate to the Sb atom in an anisobidentate manner and the lone pair is also stereochemically active; the Sb atom exists in a distorted octahedral environment.

(I)
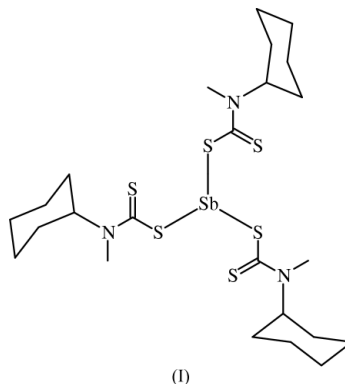
The conformation of the dithiocabamate ligand appears to be governed by two interactions [C2···S1 = 2.943 (2) Å and C3···S2 = 3.026 (2) Å] that are characterized by H···S [H···S1 = 2.40 Å and H···S2 = 2.53 Å] distances that significantly shorter than the sum of Pauling's van der Waals radii (3.05 Å).

Experimental

An ethanol solution of carbon disulfide was added to a solution of cyclohexylmethylamine in ethanol at 277 K followed by an aqueous solution of concentrated ammonia. The solid ammonium dithiocarbamate was isolated and this was reacted with antimony(III) trichloride in ethanol (3/1 molar stoichiometry) at 277 K. The solid product was collected and recrystallized from ethanol (m.p. 483–484 K). Elemental analysis (calculated in parenthesis) for C$_{24}$H$_{42}$N$_3$S$_6$Sb: C 42.07 (42.02), H 5.90 (6.12), N 6.15 (6.12), S 28.58% (28.01%).
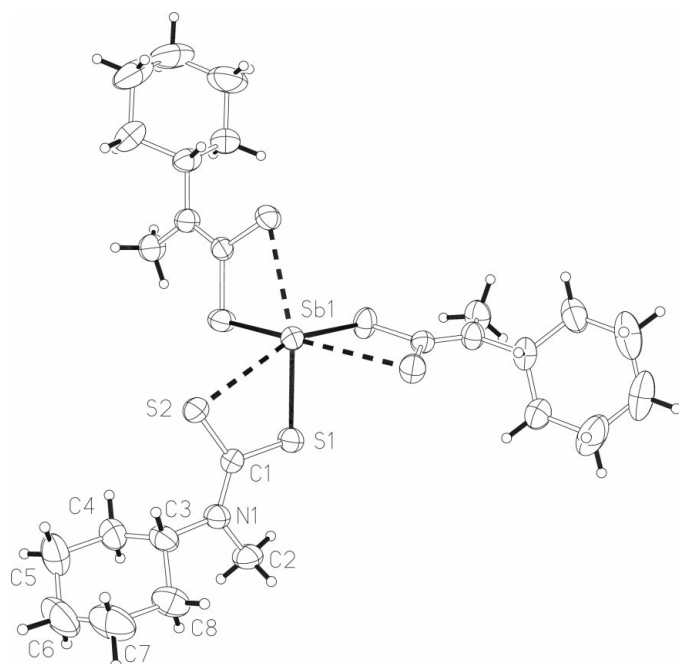
metal-organic papers

Figure 1
*ORTEP*II (Johnson, 1976) plot of (I) at the 50% probability level. H atoms are shown as circles of arbitrary radii.

Crystal data

[Sb(C$_8$H$_{14}$NS$_2$)$_3$]	Cell parameters from 8192
$M_r = 686.72$	reflections
Hexagonal, $P6_3$	$\theta = 2.7$–33.2°
$a = 13.8948$ (4) Å	$\mu = 1.28$ mm^{-1}
$V = 1586.19$ (8) Å^3	$T = 298$ (2) K
$Z = 2$	Block, yellow
$D_x = 1.438$ Mg m^{-3}	$0.62 \times 0.28 \times 0.24$ mm
Mo $K\alpha$ radiation	

Data collection

Siemens CCD area-detector	3970 independent reflections
diffractometer	3486 reflections with $(I) > 2\sigma(I)$
ω scans	$R_{int} = 0.029$
Absorption correction: empirical	$\theta_{max} = 33.18°$
(*SADABS*; Sheldrick, 1996)	$h = -19 \rightarrow 21$
$T_{min} = 0.504$, $T_{max} = 0.748$	$k = -21 \rightarrow 9$
14 627 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0249P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.026$	$+ 0.0788P]$
$wR(F^2) = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
3970 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
104 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
H-atom parameters constrained	Absolute structure: Flack &
	Schwarzenbach (1988)
	Flack parameter = -0.02 (1)

Table 1
Selected geometric parameters (Å, °).

Sb1—S1	2.530 (1)	Sb1—S2	2.975 (1)
S1—Sb1—S1^i	87.1 (1)	S1^i—Sb1—S2	150.3 (1)
S1—Sb1—S2	64.7 (1)	S1^{ii}—Sb1—S2	82.3 (1)
S1^i—Sb1—S1^{ii}	87.1 (1)		

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

Of the 3970 reflections, 2118 were unique reflections and 1582 were Friedel pairs.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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