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Crystallographic report

Chlorobis(pyrrolinedithiocarbamato)antimony(III)

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The X-ray crystal structure of $Sb(S_2CN(CH_2)_4)_2Cl$ features a five-coordinate geometry for antimony within a ClS_4 donor set, provides evidence for a stereochemical influence exerted by the lone pair of electrons on antimony, and shows no evidence for molecular aggregation. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; antimony; dithiocarbamate

COMMENT

Whereas the main group binary 1,1-dithiolate compounds have been well characterized crystallographically (as have their organometallic derivatives), structures of mixed 1,1-dithiolate/halide species are less common, e.g. see Ref. 1. In this context, whereas the structure of the xanthate compound, $Sb(S_2COEt)_2Br$, is polymeric owing to Sb—Br bridging,[2] no analogous antimony monohalide structure containing dithiocarbamate ligands has been structurally characterized. The crystallography (see below) of the title compound, $Sb(S_2CN(CH_2)_4)_2Cl$ (**I**;[3] Fig. 1), reveals a five-coordinate geometry for antimony defined by a ClS_4 donor set. Each of the dithiocarbamate ligands coordinates in an anisobidentate fashion with the asymmetry in the S(3), S(4) ligand being significantly greater than for the other. The coordination geometry is based on a highly distorted octahedron with one of the positions being occupied by a stereochemically active lone pair of electrons that is projected to occupy a position approximately *trans* to the Sb—S(3) bond. There are no hypervalent interactions in the crystal lattice involving the antimony atom thereby precluding intermolecular association, even weak, in the crystal lattice. This result is consistent with the stronger coordination mode of the dithiocarbamate compared with the xanthate ligand.[4] Thus, the Lewis acidity of the metal centre in **I** is reduced, negating the imperative to form additional interactions as found in the xanthate structure.[2]

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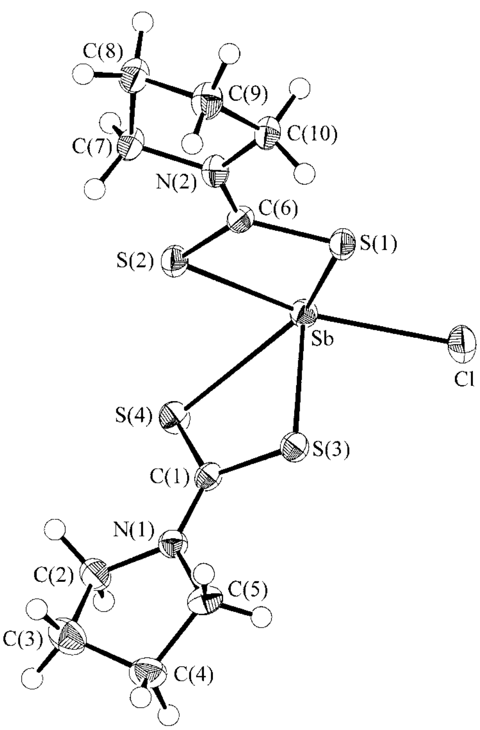

Figure 1. Molecular structure of **I**. Key geometric parameters: Sb—S(1) 2.5500(7), Sb—S(2) 2.6074(7), Sb—S(3) 2.4648(7), Sb—S(4) 2.9324(7), Sb—Cl 2.6323(7), S(1)—C(6) 1.742(3), S(2)—C(6) 1.718(3), C(6)—N(2) 1.307(3), S(3)—C(1) 1.751(3), S(4)—C(1) 1.693(3), C(1)—N(1) 1.305(3) Å; S(1)—Sb—S(2) 69.74(2), S(1)—Sb—S(3) 91.22(2), S(1)—Sb—S(4) 138.59(2), S(1)—Sb—Cl 81.89(2), S(2)—Sb—S(3) 91.51(3), S(2)—Sb—S(4) 76.24(2), S(2)—Sb—Cl 151.25(2), S(3)—Sb—S(4) 66.40(2), S(3)—Sb—Cl 84.31(2), S(4)—Sb—Cl 126.62(2)°.

CRYSTALLOGRAPHY

Crystals of **I**[4] were obtained from the slow evaporation of an acetonitrile/chloroform (1/1) solution; m.p. 203–207 °C. IR (KBr): ν(C—S) 998 and ν(C—N) 1430 cm^{-1}. Intensity data for **I** were collected at 183 K on a Bruker AXS SMART CCD diffractometer for a pale-yellow block 0.08 × 0.18 × 0.57 mm^3. $C_{10}H_{16}ClN_2S_4Sb$, $M = 449.7$, triclinic, $P\bar{1}$, $a = 6.2859(5)$, $b = 10.3205(8)$, $c = 13.2834(11)$ Å, $\alpha = 111.500(1)$, $\beta = 91.557(2)$, $\gamma = 102.728(2)$ °, $V = 776.53(11)$ Å^3, $Z = 2$, 4424 unique data (θ_{max} 30.0 °), $R = 0.033$ (all data), $wR = 0.087$ (all data), $\rho_{max} = 1.13\ e^{-}$ Å^{-3} (near Sb). Programs used: teXsan, DIRDIF, SHELXL, and ORTEP. CCDC deposition number: 191093.

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REFERENCES

1. Tiekink ERT and Winter G. *Rev. Inorg. Chem.* 1992; **12**: 183.
2. Gable RW, Hoskins BF, Steen RJ, Tiekink ERT and Winter G. *Inorg. Chim. Acta* 1983; **74**: 15.
3. Srivastava TN and Bhargava A. *J. Indian Chem. Soc.* 1979; **56**: 103.
4. Hoskins BF, Tiekink ERT and Winter G. *Inorg. Chim. Acta* 1985; **105**: 171.