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Crystallographic report

Dimeric chlorobis(hexamethylenedithiocarbamato) antimony(III) di-chloroform solvate

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The structure of $Sb(S_2CN(CH_2)_6)_2Cl$, is dimeric owing to Sb–Cl bridges and features a distorted six-coordinate geometry for antimony; there are two solvent chloroform molecules per dimer. The geometry is based on a capped octahedron as a result of the presence of a stereochemically active lone pair of electrons. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; antimony; dithiocarbamate

COMMENT

The dimeric title compound, $[Sb(S_2CN(CH_2)_6)_2Cl]_2$, was characterized as its di-chloroform solvate and is disposed about a center of inversion; Fig. 1. The antimony atom exists within a Cl_2S_4 donor set in a distorted octahedral geometry. Evidence for the presence of a stereochemically active lone-pair of electrons is readily found as one Sb–S bond distance is considerably longer than the other three Sb–S distances. Thus, the lone-pair projects out through the triangular face defined by the S2 and chlorine atoms, resulting in elongation of the associated distances. The dimeric structure contrasts with those found in closely related species, i.e. mononuclear $Sb(S_2CN(CH_2)_4)_2Cl$[1] and polymeric $[Sb(S_2COEt)_2Br]_\infty$.[2]

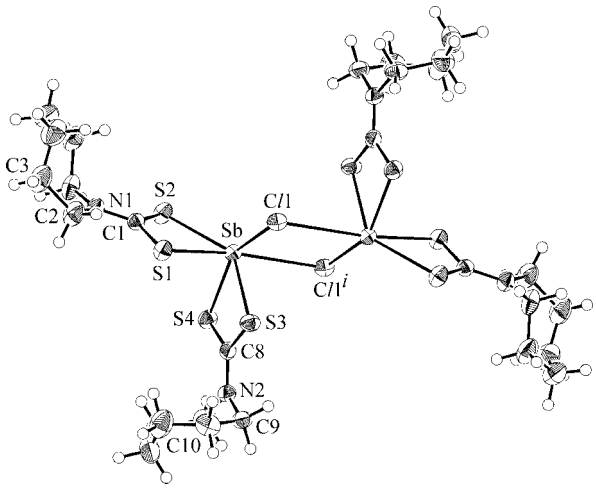

Figure 1. Molecular structure of $[Sb(S_2CN(CH_2)_6)_2Cl]_2$; the solvent chloroform molecules are not shown. Key geometric parameters: Sb–S1 2.5175(6), Sb–S2 2.8548(6), Sb–S3 2.5476(6), Sb–S4 2.5748(6), Sb–Cl1 2.8314(7), Sb–Cl^i-3.0928(7) Å; symmetry operation *i*: $-x$, $1-y$, $-z$.

EXPERIMENTAL

$[Sb(S_2CN(CH_2)_6)_2Cl]_2$ was obtained from the 1 : 2 reaction between $SbCl_3$ and the sodium salt of the ligand as per the literature method.[1] Crystals were isolated from the slow evaporation of an acetonitrile/chloroform (1/3) solution of the compound; m.p. 422–423 K. IR (KBr): ν(C–S) 974 and ν(C–N) 1510 cm^{-1}. Data were collected at 223(2) K on a Bruker AXS SMART CCD for a crystal of dimensions $0.16 \times 0.18 \times 0.47$ mm^3. $C_{14}H_{24}ClN_2S_4Sb{\cdot}CHCl_3$, $M = 625.16$, triclinic, $P\bar{1}$, $a = 9.9352(5)$, $b = 11.0154(5)$, $c = 11.8993(6)$ Å, $\alpha = 110.935(1)$, $\beta = 94.871(1)$, $\gamma = 98.678(1)°$, $V = 1188.7(1)$ Å^3, $Z = 2$, 6823 unique data (θ_{max} 30.0°), 6002 data with $I \geq 2\sigma(I)$, $R = 0.031$ (obs. data), $wR = 0.082$ (all data). Programs used: teXsan, DIRDIF, SHELXL-97 and ORTEP. CCDC deposition number: 230129.

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REFERENCES

1. Lai CS, Tiekink ERT. *Appl. Organometal. Chem.* 2003; **17**: 195.
2. Gable RW, Hoskins BF, Steen RJ, Tiekink ERT, Winter G. *Inorg. Chim. Acta* 1983; **74**: 15.