

Tetramethylammonium chlorodiphenylthiocyanatoantimonate(III)

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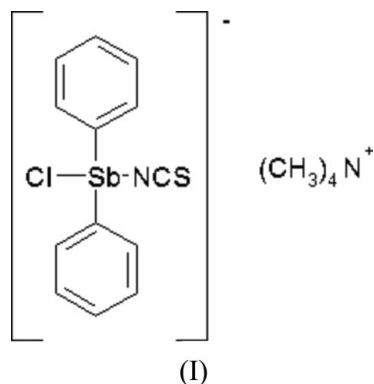
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å
 R factor = 0.039
 wR factor = 0.100
Data-to-parameter ratio = 17.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, $(\text{C}_4\text{H}_{12}\text{N})\text{[Sb}(\text{C}_6\text{H}_5)_2\text{Cl}(\text{NCS})]$, contains two cations and two anions in the asymmetric unit. The Sb atom exhibits a distorted pseudo-trigonal–bipyramidal coordination, with the phenyl groups and the lone pair of electrons in equatorial positions and N-bonded thiocyanate and Cl^- ligands in axial positions.

Comment

There are only a few examples in the literature of diorganoantimonate(III) anionic complex species, such as diphenylantimony complexes with different cations, *e.g.* $(\text{Me}_4\text{N})[\text{Ph}_2\text{SbCl}_2]$ (Hall & Sowerby, 1988), $[(\text{Cp})_2\text{Co}][\text{Ph}_2\text{SbCl}_2]$ (Calderazzo *et al.*, 1991) and $(\text{Et}_4\text{N})[\text{Ph}_2\text{SbI}_2]$ (Sheldrick & Martin, 1991), where the anions have substantially the same distorted pseudo-trigonal–bipyramidal coordination around Sb, with phenyl groups in the equatorial positions and halides in the two axial positions. The compound $\text{K}[\text{Ph}_2\text{Sb}(\text{SCN})_2]$ has also been characterized and (on the basis of IR spectroscopic results) probably contains monomeric anions having both N- and S-bonded thiocyanate groups (Forster *et al.*, 1995*a*). We succeeded in the preparation of the title mixed chloride–thiocyanate diphenyl–antimony(III) monomeric anionic complex, (I), and its crystal structure is reported here.



The anion of (I) has a pseudo-trigonal–bipyramidal structure, with the phenyl groups in equatorial positions and Cl^- and thiocyanate ions in the two axial positions. The thiocyanate groups in the two independent anions are N-bonded in a monodentate fashion, with angles at C1 and C1*a* of 179.1 (5) and 178.8 (5)°, respectively. The C–N bond lengths are short [C1–N1 = 1.117 (7) Å and C1*a*–N1*a* = 1.081 (8) Å] and close to the values for a triple bond (C≡N 1.16 Å and C=N = 1.30 Å; Emsley, 1991), whereas the S1–C1 and S1*a*–C1*a* bond lengths of 1.617 (5) and 1.625 (6) Å, respectively, are closer to the value observed for C=S double bonds (*ca*

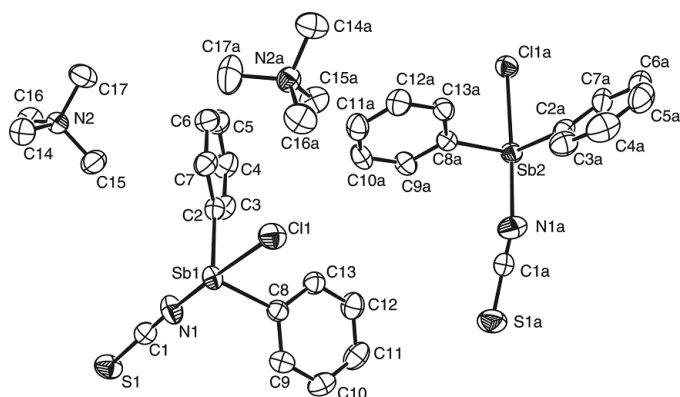


Figure 1
A view of the two independent cations and anions of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

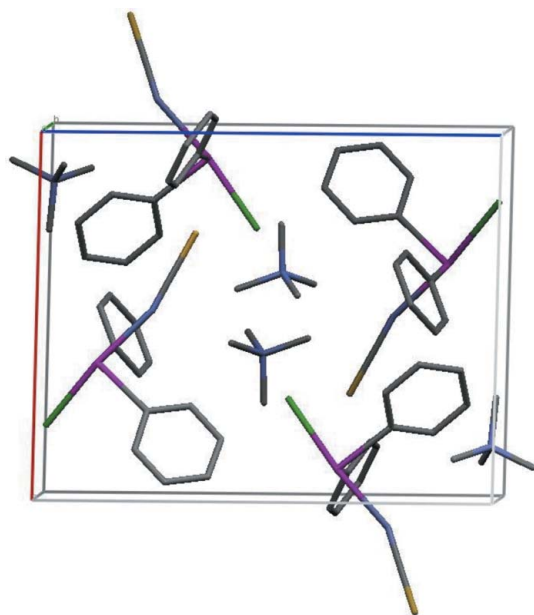


Figure 2
A packing diagram for (I), viewed approximately down the *b* axis. H atoms have been omitted.

1.55 Å) than that for single bonds (*ca* 1.82 Å; Emsley *et al.*, 1991). The angles at the coordinated N atoms are significantly different, at 154.6 (4)° for Sb1–N1–C1 and 162.1 (5)° for Sb2–N1a–C1a, while those between Cl[–] and thiocyanate are 178.7 (1) and 170.1 (1)° for Cl1–Sb1–N1 and Cl1a–Sb2–N1a, respectively. The Sb–C bond distances of 2.143 (4) and 2.145 (5) Å are in agreement with those found in the polymeric structure of Ph₂Sb(NCS) [2.133 (6) and 2.143 (6) Å; Forster *et al.*, 1995b], whereas the Sb–N distances of 2.365 (5) and 2.355 (6) Å are at the upper limit of those found for the bridging thiocyanates in the same compound [2.304 (5), 2.273 (6) and 2.364 (6) Å].

Experimental

Ph₂SbCl was obtained by the reorganization in the absence of solvent of a Ph₃Sb–SbCl₃ 2:1 molar ratio mixture (Nunn *et al.*, 1983). The

resulting crude viscous yellow oil (3.60 g, nominally 11.5 mmol) was dissolved in absolute ethanol (50 ml). After filtration, the resulting clear solution (10 ml) was treated with (Me₄N)NCS (0.32 g, 2.42 mmol; Bertazzi & Barbieri, 1969) in absolute ethanol (20 ml). After allowing the reaction vessel to stand at room temperature for 3 d, small white crystals of (I) were obtained. These were recrystallized from absolute EtOH (m.p. 416–418 K). Analysis, found: C 46.10, H 5.15, N 6.45; calculated for C₁₇H₂₂N₂SbClS: C 46.03, H 5.00, N 6.31; IR (cm^{–1}, Nujoll): 2045 (*vs*, *ν* CN), 470 (*w*, *δ* NCS).

Crystal data

(C₄H₁₂N)[Sb(C₆H₅)₂Cl(NCS)]

M_r = 443.65

Triclinic, *P* $\bar{1}$

a = 11.245 (2) Å

b = 12.354 (3) Å

c = 15.226 (3) Å

α = 112.05 (2)°

β = 92.83 (3)°

γ = 86.44 (3)°

V = 1955.9 (8) Å³

Z = 4

D_x = 1.507 Mg m^{–3}

Mo *K*α radiation

Cell parameters from 25 reflections

θ = 3.2–26°

μ = 1.65 mm^{–1}

T = 293 (2) K

Prism, white

0.36 × 0.32 × 0.26 mm

Data collection

Enraf–Nonius CAD-4

diffractometer

ω scans

Absorption correction: ψ scan
(North *et al.*, 1968)

T_{min} = 0.546, *T_{max}* = 0.648

7524 measured reflections

7175 independent reflections

6983 reflections with *I* > 2 σ (*I*)

R_{int} = 0.030

θ_{\max} = 26.0°

h = –13 → 13

k = –15 → 14

l = 0 → 18

3 standard reflections

frequency: 180 min

intensity decay: none

Refinement

Refinement on *F*²

R [*F*² > 2 σ (*F*²)] = 0.039

wR(*F*²) = 0.100

S = 1.29

7175 reflections

405 parameters

H-atom parameters constrained

w = 1/[$\sigma^2(F_o^2) + 3.4332P$]

where *P* = (*F_o*² + 2*F_c*²)/3

($\Delta\sigma$)_{max} = 0.010

$\Delta\rho_{\max}$ = 0.61 e Å^{–3}

$\Delta\rho_{\min}$ = –0.70 e Å^{–3}

Table 1

Selected geometric parameters (Å, °).

Sb1–C8	2.143 (4)	Sb2–C2a	2.143 (5)
Sb1–C2	2.145 (5)	Sb2–C8a	2.147 (4)
Sb1–N1	2.365 (5)	Sb2–N1a	2.355 (6)
Sb1–Cl1	2.5981 (15)	Sb2–Cl1a	2.6105 (15)
S1–C1	1.617 (5)	S1a–C1a	1.625 (5)
N1–C1	1.117 (6)	N1a–C1a	1.080 (7)
C8–Sb1–C2	97.84 (17)	C2a–Sb2–C8a	98.31 (17)
C8–Sb1–N1	84.25 (17)	C2a–Sb2–N1a	86.1 (2)
C2–Sb1–N1	86.19 (17)	C8a–Sb2–N1a	84.23 (18)
C8–Sb1–Cl1	88.83 (12)	C2a–Sb2–Cl1a	88.35 (13)
C2–Sb1–Cl1	88.66 (12)	C8a–Sb2–Cl1a	88.41 (12)
N1–Sb1–Cl1	170.74 (12)	N1a–Sb2–Cl1a	170.06 (14)
Cl1–N1–Sb1	154.6 (4)	Cl1a–N1a–Sb2	162.1 (5)
N1–C1–S1	179.1 (5)	N1a–C1a–S1a	178.8 (5)

H atoms were treated as riding, with *Csp*³–H 0.96 and *Csp*²–H 0.93 Å, and *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD-4* (Harms & Wocadlo, 1995), and *PSI* and *EAC* in *CAD-4 EXPRESS*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular

graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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