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Dichloridotriphenylantimony(V)–bis(pyrrolidin-1-ylthiocarbonyl) disulfide (1/1)

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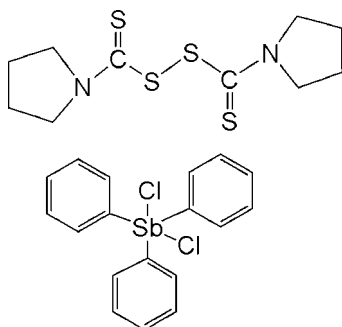
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}–\text{C}) = 0.007$ Å; R factor = 0.031; wR factor = 0.092; data-to-parameter ratio = 16.6.

The asymmetric unit of the title compound, $[\text{Sb}(\text{C}_6\text{H}_5)_3\text{Cl}_2] \cdot \text{C}_{10}\text{H}_{16}\text{N}_2\text{S}_4$, comprises a bis(pyrrolidinylthiocarbonyl) molecule and a dichlorotriphenylantimony(V) complex. In the Sb complex, the central atom is coordinated by three C atoms of the three phenyl ligands and two Cl atoms in a slightly distorted trigonal-bipyramidal geometry. The thiocarbonyl units, connected *via* the disulfide bond, are approximately perpendicular to each other. The molecules are connected by weak $\text{C}–\text{H} \cdots \text{S}$ and $\text{C}–\text{H} \cdots \text{Cl}$ hydrogen-bonding interactions into two one-dimensional supramolecular chains.

Related literature

For related structures, see: Williams *et al.* (1983); Feng Li *et al.* (2006). For discussion on $\text{C}–\text{H} \cdots \text{S}$ interactions, see: Srinivasan *et al.* (2007). For related literature, see: Kumar *et al.* (1990).



Experimental

Crystal data

 $[\text{Sb}(\text{C}_6\text{H}_5)_3\text{Cl}_2] \cdot \text{C}_{10}\text{H}_{16}\text{N}_2\text{S}_4$
 $M_r = 716.48$

 Monoclinic, $P2_1/n$
 $a = 14.8138$ (18) Å

 $b = 13.6440$ (13) Å
 $c = 16.316$ (3) Å
 $\beta = 105.509$ (2)°
 $V = 3177.7$ (8) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 1.32$ mm⁻¹
 $T = 298$ (2) K
 $0.50 \times 0.42 \times 0.39$ mm

Data collection

 Siemens SMART CCD
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.558$, $T_{\max} = 0.627$

 14597 measured reflections
 5560 independent reflections
 4333 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.092$
 $S = 1.00$
 5560 reflections

 335 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.67$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.59$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D–H \cdots A$	$D–H$	$H \cdots A$	$D \cdots A$	$D–H \cdots A$
$\text{C15}–\text{H15} \cdots \text{S4}^i$	0.93	2.91	3.594 (5)	132
$\text{C13}–\text{H13} \cdots \text{Cl2}^{ii}$	0.93	2.83	3.509 (4)	131
$\text{C8}–\text{H8A} \cdots \text{Cl1}^{iii}$	0.97	2.84	3.719 (6)	151

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x, -y + 2, -z + 1$; (iii) $-x, -y + 2, -z$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2063).

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supplementary materials

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Dichloridotriphenylantimony(V)-bis(pyrrolidin-1-ylthiocarbonyl) disulfide (1/1)

L. Quan, H. Yin, J. Zhai and D. Wang

Comment

In the title complex, the antimony atom is coordinated by three C atoms of three phenyl ligands and two Cl atoms in a slightly distorted trigonal-bipyramidal geometry. Atoms Cl1, Cl2 of the complex lie in axial positions (Fig. 1), with the axial angle Cl1—Sb1—Cl2 178.65 (4)°, deviating substantially from the linear value of 180°. The distances Sb—Cl also vary merely with the role they play in the structure: Sb1—Cl1 = 2.4720 (9) Å and Sb1—Cl2 = 2.4792 (10) Å. In the bis(pyrrolidinylthiocarbonyl) molecule of the title compound, the thiocarbonyl moieties, connected *via* the disulfide bond (S1—S3 = 2.0029 (17) Å), are approximately perpendicular to each other. A Newman projection, calculated with *PLATON*, with a view along the disulfide bond results in a dihedral angle of 87° for C1—S1—S3—C6 (Spek, 2003).

The Dipyrrolidylthiuram disulfide molecule, (which is another name of the organic part in our structure) shows a planar model with crystallographic inversion symmetry in the midpoint of the S—S bond (Williams *et al.*, 1983).

In the similar structure bis(*N,N*-dicyclohexylthiocarbonyl) disulfide, a crystallographic twofold axis passes through the midpoint of the S—S bond (Li *et al.*, 2006). The disulfide S—S distance is close to the distances observed in free (uncoordinated) disulfides (Kumar *et al.*, 1990).

The S atoms of bis(pyrrolidinylthiocarbonyl) and the Cl atoms of dichlorotriphenylantimony play a significant role in the crystal packing, linking the complex molecules by weak C—H···S and C—H···Cl (Table 1) hydrogen bonds to form two one-dimensional supramolecular chains (Fig. 2).

Experimental

Chlorotriphenylantimony (0.2 mmol) was dissolved in benzene (15 ml) and bis(pyrrolidinylthiocarbonyl) (0.2 mmol) dissolved in methanol was added with stirring at room temperature for eight hours and then filtered. Orange crystals suitable for X-ray analysis were obtained by slow evaporation of a petroleum/dichloromethane (1:2 *v/v*) solution over a period of twenty days (yield 85%. m.p. 432k). Anal. Calcd (%) for C₂₈H₃₁N₂S₄Cl₂Sb (Mr = 716.48): C, 46.94; H, 4.36; N, 3.91. Found (%): C, 46.89; H, 4.31; N, 3.87.

Refinement

The H atoms bound to C of pyrrolidine were located in a difference map and were refined as riding on their respective C atoms with distances C—H = 0.97 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The other H atoms were constraint at calculated positions (riding mode), with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Figures

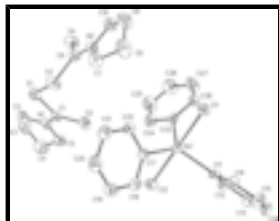


Fig. 1. The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

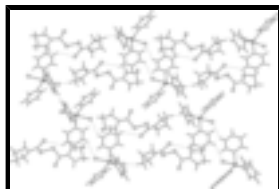


Fig. 2. Crystal packing of the title compound, showing catemer chains, linked by weak C15—H15...S4, C13—H13...Cl2, C8—H8A...Cl1 hydrogen bonding contacts, indicated by dashed lines. Symmetry codes are given in Table 1.

Dichloridotriphenylantimony(V)-bis(pyrrolidin-1-ylthiocarbonyl) disulfide (1/1)

Crystal data

[Sb(C₆H₅)₃Cl₂]·C₁₀H₁₆N₂S₄

$M_r = 716.48$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 14.8138$ (18) Å

$b = 13.6440$ (13) Å

$c = 16.316$ (3) Å

$\beta = 105.509$ (2)°

$V = 3177.7$ (8) Å³

$Z = 4$

$F_{000} = 1448$

$D_x = 1.498$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 7025 reflections

$\theta = 2.2$ – 27.8 °

$\mu = 1.32$ mm⁻¹

$T = 298$ (2) K

Block, colorless

$0.50 \times 0.42 \times 0.39$ mm

Data collection

Siemens SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.558$, $T_{\max} = 0.627$

14597 measured reflections

5560 independent reflections

4333 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.041$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.0$ °

$h = -17 \rightarrow 10$

$k = -16 \rightarrow 16$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.031$$

$$wR(F^2) = 0.092$$

$$S = 1.00$$

5560 reflections

335 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 1.9404P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.67 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.59 \text{ e } \text{\AA}^{-3}$$

Extinction correction: SHELXL

Extinction coefficient: 0.0048 (3)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sb1	0.018116 (16)	0.991080 (17)	0.234502 (14)	0.03657 (11)
Cl1	-0.07709 (7)	1.08719 (7)	0.11362 (6)	0.0508 (3)
Cl2	0.11117 (8)	0.89132 (8)	0.35457 (6)	0.0569 (3)
N1	0.6750 (2)	0.8384 (2)	0.3780 (2)	0.0516 (8)
N2	0.4034 (2)	1.0375 (2)	0.0796 (2)	0.0534 (9)
S1	0.59570 (9)	0.82805 (9)	0.21567 (7)	0.0664 (3)
S2	0.50821 (9)	0.92203 (9)	0.34589 (8)	0.0665 (3)
S3	0.46930 (9)	0.86974 (9)	0.14467 (7)	0.0704 (4)
S4	0.58682 (9)	1.05180 (10)	0.13631 (8)	0.0742 (4)
C1	0.5950 (3)	0.8643 (3)	0.3226 (2)	0.0479 (10)
C2	0.7541 (3)	0.7877 (3)	0.3599 (3)	0.0660 (12)
H2A	0.7718	0.8181	0.3127	0.079*
H2B	0.7396	0.7192	0.3467	0.079*
C3	0.8311 (4)	0.7985 (4)	0.4418 (3)	0.0879 (17)
H3A	0.8735	0.7429	0.4505	0.106*
H3B	0.8667	0.8580	0.4415	0.106*
C4	0.7802 (4)	0.8025 (5)	0.5087 (3)	0.0972 (19)
H4A	0.8174	0.8356	0.5591	0.117*
H4B	0.7654	0.7371	0.5243	0.117*
C5	0.6926 (3)	0.8589 (4)	0.4697 (3)	0.0685 (13)
H5A	0.6410	0.8362	0.4910	0.082*
H5B	0.7017	0.9285	0.4812	0.082*
C6	0.4843 (3)	0.9968 (3)	0.1173 (2)	0.0531 (11)
C7	0.3104 (3)	0.9904 (3)	0.0625 (3)	0.0732 (14)
H7A	0.3029	0.9567	0.1126	0.088*
H7B	0.3015	0.9438	0.0161	0.088*
C8	0.2428 (4)	1.0747 (4)	0.0389 (4)	0.100 (2)
H8A	0.1850	1.0541	-0.0012	0.120*
H8B	0.2283	1.1018	0.0889	0.120*
C9	0.2927 (4)	1.1471 (5)	-0.0001 (4)	0.104 (2)
H9A	0.2703	1.2128	0.0061	0.124*
H9B	0.2828	1.1334	-0.0602	0.124*
C10	0.3956 (4)	1.1383 (3)	0.0462 (3)	0.0739 (14)
H10A	0.4345	1.1476	0.0076	0.089*

supplementary materials

H10B	0.4133	1.1858	0.0920	0.089*
C11	-0.0708 (2)	1.0332 (3)	0.3075 (2)	0.0377 (8)
C12	-0.0319 (3)	1.0717 (3)	0.3882 (2)	0.0496 (10)
H12	0.0328	1.0757	0.4095	0.059*
C13	-0.0893 (3)	1.1037 (3)	0.4366 (3)	0.0589 (11)
H13	-0.0632	1.1315	0.4897	0.071*
C14	-0.1851 (3)	1.0950 (3)	0.4067 (3)	0.0589 (11)
H14	-0.2235	1.1151	0.4403	0.071*
C15	-0.2239 (3)	1.0566 (3)	0.3274 (3)	0.0610 (11)
H15	-0.2886	1.0508	0.3075	0.073*
C16	-0.1680 (3)	1.0268 (3)	0.2772 (2)	0.0495 (10)
H16	-0.1950	1.0024	0.2230	0.059*
C17	-0.0118 (2)	0.8611 (3)	0.1610 (2)	0.0384 (8)
C18	-0.0404 (3)	0.7789 (3)	0.1959 (3)	0.0503 (10)
H18	-0.0478	0.7808	0.2507	0.060*
C19	-0.0580 (3)	0.6933 (3)	0.1490 (3)	0.0607 (12)
H19	-0.0773	0.6374	0.1723	0.073*
C20	-0.0469 (3)	0.6908 (3)	0.0685 (3)	0.0650 (12)
H20	-0.0581	0.6329	0.0373	0.078*
C21	-0.0195 (3)	0.7731 (3)	0.0336 (3)	0.0611 (11)
H21	-0.0127	0.7709	-0.0214	0.073*
C22	-0.0018 (3)	0.8593 (3)	0.0794 (2)	0.0491 (10)
H22	0.0166	0.9152	0.0556	0.059*
C23	0.1365 (3)	1.0813 (3)	0.2432 (2)	0.0428 (9)
C24	0.2261 (3)	1.0430 (4)	0.2705 (3)	0.0642 (12)
H24	0.2351	0.9765	0.2825	0.077*
C25	0.3018 (3)	1.1049 (4)	0.2797 (4)	0.0844 (16)
H25	0.3622	1.0795	0.2967	0.101*
C26	0.2892 (4)	1.2026 (4)	0.2643 (4)	0.0859 (16)
H26	0.3408	1.2437	0.2714	0.103*
C27	0.2006 (4)	1.2404 (3)	0.2382 (3)	0.0757 (14)
H27	0.1923	1.3072	0.2278	0.091*
C28	0.1236 (3)	1.1801 (3)	0.2271 (2)	0.0505 (10)
H28	0.0635	1.2060	0.2089	0.061*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.03922 (16)	0.03827 (16)	0.03499 (15)	0.00222 (11)	0.01473 (11)	0.00279 (10)
Cl1	0.0587 (6)	0.0493 (6)	0.0422 (5)	0.0073 (5)	0.0095 (4)	0.0092 (4)
Cl2	0.0678 (7)	0.0605 (6)	0.0419 (5)	0.0145 (6)	0.0138 (5)	0.0138 (4)
N1	0.050 (2)	0.0453 (19)	0.0531 (19)	-0.0006 (17)	0.0033 (16)	0.0008 (15)
N2	0.059 (2)	0.0451 (19)	0.0507 (19)	-0.0064 (18)	0.0047 (17)	-0.0006 (15)
S1	0.0762 (8)	0.0609 (7)	0.0545 (6)	0.0119 (6)	0.0046 (6)	-0.0034 (5)
S2	0.0606 (7)	0.0570 (7)	0.0807 (8)	0.0096 (6)	0.0170 (6)	0.0004 (6)
S3	0.0749 (8)	0.0524 (7)	0.0655 (7)	-0.0139 (6)	-0.0136 (6)	0.0068 (5)
S4	0.0652 (8)	0.0727 (8)	0.0780 (8)	-0.0193 (7)	0.0074 (6)	0.0031 (6)
C1	0.053 (2)	0.034 (2)	0.054 (2)	-0.0035 (18)	0.0078 (19)	0.0020 (16)

C2	0.057 (3)	0.057 (3)	0.080 (3)	0.005 (2)	0.012 (2)	-0.004 (2)
C3	0.059 (3)	0.083 (4)	0.104 (4)	0.013 (3)	-0.010 (3)	-0.005 (3)
C4	0.085 (4)	0.111 (5)	0.076 (4)	0.005 (4)	-0.014 (3)	0.017 (3)
C5	0.072 (3)	0.072 (3)	0.055 (3)	-0.003 (3)	0.005 (2)	0.006 (2)
C6	0.066 (3)	0.048 (2)	0.040 (2)	-0.008 (2)	0.005 (2)	-0.0023 (17)
C7	0.059 (3)	0.066 (3)	0.085 (3)	-0.008 (3)	0.001 (3)	-0.005 (2)
C8	0.069 (4)	0.085 (4)	0.128 (5)	0.006 (3)	-0.001 (3)	-0.011 (4)
C9	0.095 (5)	0.091 (4)	0.116 (5)	0.031 (4)	0.014 (4)	0.029 (4)
C10	0.089 (4)	0.053 (3)	0.078 (3)	0.002 (3)	0.019 (3)	0.001 (2)
C11	0.039 (2)	0.047 (2)	0.0280 (17)	-0.0044 (18)	0.0112 (15)	0.0047 (15)
C12	0.047 (2)	0.059 (3)	0.045 (2)	0.001 (2)	0.0155 (18)	-0.0031 (18)
C13	0.066 (3)	0.068 (3)	0.046 (2)	-0.002 (2)	0.022 (2)	-0.011 (2)
C14	0.057 (3)	0.067 (3)	0.064 (3)	0.006 (2)	0.035 (2)	-0.008 (2)
C15	0.044 (2)	0.075 (3)	0.069 (3)	-0.001 (2)	0.025 (2)	-0.010 (2)
C16	0.047 (2)	0.060 (3)	0.041 (2)	-0.002 (2)	0.0133 (18)	-0.0061 (18)
C17	0.0344 (19)	0.038 (2)	0.0434 (19)	0.0001 (16)	0.0112 (16)	0.0024 (15)
C18	0.049 (2)	0.051 (2)	0.057 (2)	-0.001 (2)	0.0257 (19)	0.0049 (19)
C19	0.051 (3)	0.049 (3)	0.087 (3)	-0.011 (2)	0.026 (2)	0.004 (2)
C20	0.066 (3)	0.048 (3)	0.079 (3)	-0.002 (2)	0.017 (3)	-0.016 (2)
C21	0.078 (3)	0.059 (3)	0.050 (2)	0.003 (3)	0.024 (2)	-0.009 (2)
C22	0.065 (3)	0.045 (2)	0.043 (2)	0.003 (2)	0.0250 (19)	0.0023 (17)
C23	0.039 (2)	0.047 (2)	0.047 (2)	-0.0026 (18)	0.0181 (17)	-0.0019 (17)
C24	0.047 (3)	0.057 (3)	0.091 (3)	0.005 (2)	0.023 (2)	0.010 (2)
C25	0.038 (3)	0.081 (4)	0.131 (5)	0.000 (3)	0.017 (3)	0.013 (3)
C26	0.053 (3)	0.076 (4)	0.125 (5)	-0.022 (3)	0.018 (3)	0.004 (3)
C27	0.066 (3)	0.044 (3)	0.115 (4)	-0.010 (2)	0.021 (3)	0.004 (3)
C28	0.045 (2)	0.042 (2)	0.066 (3)	0.0014 (19)	0.017 (2)	0.0033 (19)

Geometric parameters (Å, °)

Sb1—C11	2.079 (4)	C10—H10A	0.9700
Sb1—C23	2.116 (4)	C10—H10B	0.9700
Sb1—C17	2.120 (3)	C11—C12	1.392 (5)
Sb1—Cl1	2.4720 (9)	C11—C16	1.394 (5)
Sb1—Cl2	2.4792 (10)	C12—C13	1.377 (5)
N1—C1	1.332 (5)	C12—H12	0.9300
N1—C2	1.457 (5)	C13—C14	1.377 (6)
N1—C5	1.476 (5)	C13—H13	0.9300
N2—C6	1.315 (5)	C14—C15	1.371 (6)
N2—C10	1.473 (5)	C14—H14	0.9300
N2—C7	1.477 (6)	C15—C16	1.372 (5)
S1—C1	1.817 (4)	C15—H15	0.9300
S1—S3	2.0029 (17)	C16—H16	0.9300
S2—C1	1.636 (4)	C17—C18	1.375 (5)
S3—C6	1.818 (4)	C17—C22	1.379 (5)
S4—C6	1.648 (5)	C18—C19	1.382 (6)
C2—C3	1.515 (6)	C18—H18	0.9300
C2—H2A	0.9700	C19—C20	1.368 (6)
C2—H2B	0.9700	C19—H19	0.9300

supplementary materials

C3—C4	1.484 (8)	C20—C21	1.369 (6)
C3—H3A	0.9700	C20—H20	0.9300
C3—H3B	0.9700	C21—C22	1.381 (6)
C4—C5	1.496 (7)	C21—H21	0.9300
C4—H4A	0.9700	C22—H22	0.9300
C4—H4B	0.9700	C23—C28	1.378 (5)
C5—H5A	0.9700	C23—C24	1.384 (6)
C5—H5B	0.9700	C24—C25	1.380 (6)
C7—C8	1.507 (7)	C24—H24	0.9300
C7—H7A	0.9700	C25—C26	1.360 (7)
C7—H7B	0.9700	C25—H25	0.9300
C8—C9	1.476 (8)	C26—C27	1.367 (7)
C8—H8A	0.9700	C26—H26	0.9300
C8—H8B	0.9700	C27—C28	1.378 (6)
C9—C10	1.514 (7)	C27—H27	0.9300
C9—H9A	0.9700	C28—H28	0.9300
C9—H9B	0.9700		
C11—Sb1—C23	116.22 (14)	C8—C9—H9B	110.4
C11—Sb1—C17	119.09 (14)	C10—C9—H9B	110.4
C23—Sb1—C17	124.66 (14)	H9A—C9—H9B	108.6
C11—Sb1—C11	89.80 (10)	N2—C10—C9	103.2 (4)
C23—Sb1—C11	91.75 (10)	N2—C10—H10A	111.1
C17—Sb1—C11	90.23 (9)	C9—C10—H10A	111.1
C11—Sb1—C12	90.17 (10)	N2—C10—H10B	111.1
C23—Sb1—C12	89.47 (10)	C9—C10—H10B	111.1
C17—Sb1—C12	88.61 (9)	H10A—C10—H10B	109.1
C11—Sb1—C12	178.65 (4)	C12—C11—C16	119.1 (3)
C1—N1—C2	127.4 (3)	C12—C11—Sb1	118.7 (3)
C1—N1—C5	121.4 (4)	C16—C11—Sb1	122.2 (3)
C2—N1—C5	111.2 (3)	C13—C12—C11	119.9 (4)
C6—N2—C10	122.6 (4)	C13—C12—H12	120.0
C6—N2—C7	126.3 (4)	C11—C12—H12	120.0
C10—N2—C7	111.1 (4)	C12—C13—C14	120.4 (4)
C1—S1—S3	103.49 (15)	C12—C13—H13	119.8
C6—S3—S1	104.86 (16)	C14—C13—H13	119.8
N1—C1—S2	125.6 (3)	C15—C14—C13	119.9 (4)
N1—C1—S1	110.2 (3)	C15—C14—H14	120.1
S2—C1—S1	124.1 (2)	C13—C14—H14	120.1
N1—C2—C3	103.4 (4)	C14—C15—C16	120.6 (4)
N1—C2—H2A	111.1	C14—C15—H15	119.7
C3—C2—H2A	111.1	C16—C15—H15	119.7
N1—C2—H2B	111.1	C15—C16—C11	120.0 (4)
C3—C2—H2B	111.1	C15—C16—H16	120.0
H2A—C2—H2B	109.0	C11—C16—H16	120.0
C4—C3—C2	103.9 (4)	C18—C17—C22	120.6 (4)
C4—C3—H3A	111.0	C18—C17—Sb1	119.3 (3)
C2—C3—H3A	111.0	C22—C17—Sb1	120.1 (3)
C4—C3—H3B	111.0	C17—C18—C19	119.6 (4)
C2—C3—H3B	111.0	C17—C18—H18	120.2

H3A—C3—H3B	109.0	C19—C18—H18	120.2
C3—C4—C5	105.1 (4)	C20—C19—C18	120.0 (4)
C3—C4—H4A	110.7	C20—C19—H19	120.0
C5—C4—H4A	110.7	C18—C19—H19	120.0
C3—C4—H4B	110.7	C19—C20—C21	120.3 (4)
C5—C4—H4B	110.7	C19—C20—H20	119.9
H4A—C4—H4B	108.8	C21—C20—H20	119.9
N1—C5—C4	103.3 (4)	C20—C21—C22	120.5 (4)
N1—C5—H5A	111.1	C20—C21—H21	119.7
C4—C5—H5A	111.1	C22—C21—H21	119.7
N1—C5—H5B	111.1	C17—C22—C21	119.0 (4)
C4—C5—H5B	111.1	C17—C22—H22	120.5
H5A—C5—H5B	109.1	C21—C22—H22	120.5
N2—C6—S4	125.1 (3)	C28—C23—C24	120.2 (4)
N2—C6—S3	111.2 (3)	C28—C23—Sb1	119.1 (3)
S4—C6—S3	123.7 (3)	C24—C23—Sb1	120.5 (3)
N2—C7—C8	103.8 (4)	C25—C24—C23	119.0 (4)
N2—C7—H7A	111.0	C25—C24—H24	120.5
C8—C7—H7A	111.0	C23—C24—H24	120.5
N2—C7—H7B	111.0	C26—C25—C24	120.8 (5)
C8—C7—H7B	111.0	C26—C25—H25	119.6
H7A—C7—H7B	109.0	C24—C25—H25	119.6
C9—C8—C7	104.2 (5)	C25—C26—C27	120.0 (5)
C9—C8—H8A	110.9	C25—C26—H26	120.0
C7—C8—H8A	110.9	C27—C26—H26	120.0
C9—C8—H8B	110.9	C26—C27—C28	120.5 (4)
C7—C8—H8B	110.9	C26—C27—H27	119.7
H8A—C8—H8B	108.9	C28—C27—H27	119.7
C8—C9—C10	106.6 (4)	C27—C28—C23	119.5 (4)
C8—C9—H9A	110.4	C27—C28—H28	120.3
C10—C9—H9A	110.4	C23—C28—H28	120.3

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C15—H15 \cdots S4 ⁱ	0.93	2.91	3.594 (5)	132
C13—H13 \cdots C12 ⁱⁱ	0.93	2.83	3.509 (4)	131
C8—H8A \cdots C11 ⁱⁱⁱ	0.97	2.84	3.719 (6)	151

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, -y+2, -z+1$; (iii) $-x, -y+2, -z$.

Fig. 1

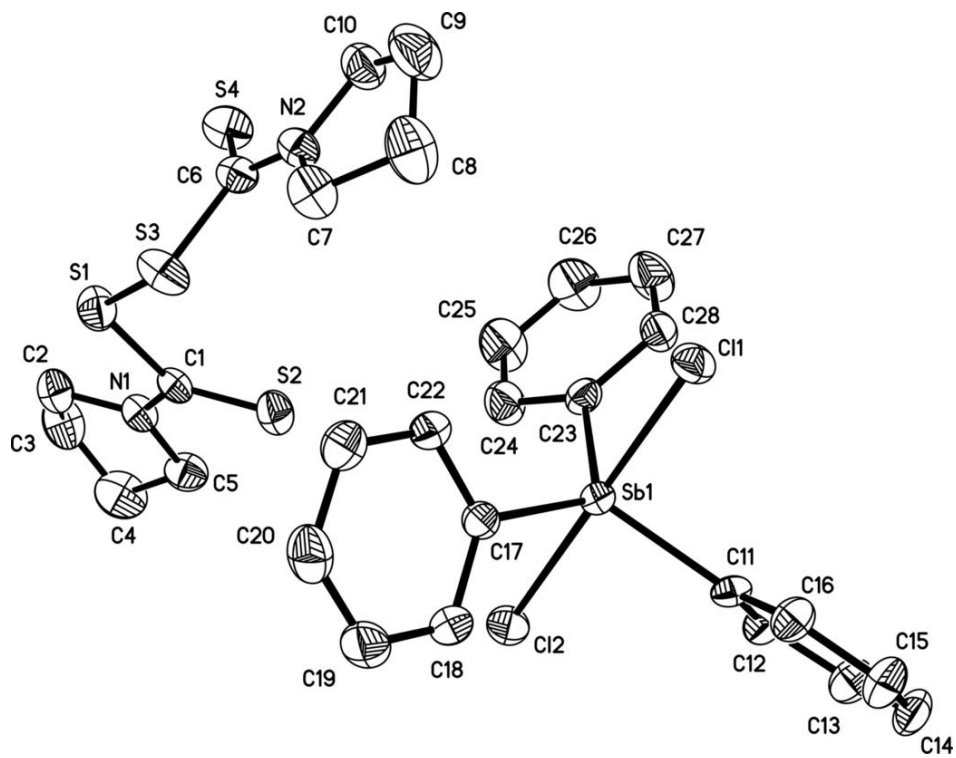


Fig. 2

