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# 2,2′,2″-Nitrilotris(ethylammonium) tetrathioantimonate

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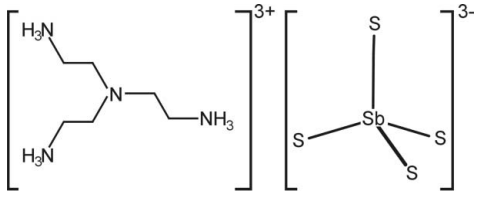
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.015 Å; $R$ factor = 0.025; $wR$ factor = 0.065; data-to-parameter ratio = 12.9.

The crystal structure of the title compound, $(C_6H_{21}N_4)[SbS_4]$, consists of discrete tetrahedral $[SbS_4]^{3-}$ anions and discrete tris(2-ethylammonium)amine cations. There are two crystallographically independent cations and anions in the asymmetric unit, all of them located in general positions. The cations and anions are connected by N—H···S hydrogen bonding.

## Related literature

For structures of related compounds, see: Stähler & Bensch (2002); Graf & Schäfer (1976); Graf *et al.* (1969); Mereiter *et al.* (1979); Schimek *et al.* (1996); Bensch & Dürichen (1996); Schur *et al.* (1998); Schur & Bensch (2000); Schaefer *et al.* (2003); Jia *et al.* (2004); Jia, Zhao *et al.* (2005); Jia, Zhu *et al.* (2005); Stähler *et al.* (2002); Alyea *et al.* (1995).




## Experimental

### Crystal data

$(C_6H_{21}N_4)[SbS_4]$
$M_r$ = 399.26
Orthorhombic, $Pca2_1$
$a$ = 19.7872 (13) Å
$b$ = 10.8571 (10) Å
$c$ = 13.5055 (8) Å
$V$ = 2901.4 (4) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 2.46 mm$^{-1}$
$T$ = 293 (2) K
0.2 × 0.1 × 0.1 mm

### Data collection

Stoe AEDII diffractometer
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998)
$T_{min}$ = 0.738, $T_{max}$ = 0.772
6750 measured reflections
3571 independent reflections
2898 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
4 standard reflections
frequency: 2 h
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
$S$ = 1.03
3571 reflections
277 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.55 e Å$^{-3}$
Absolute structure: Flack (1983), 544 Friedel pairs
Flack parameter: 0.04 (5)

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H1*N*···S1$^{i}$ | 0.89 | 2.48 | 3.336 (7) | 161 |
| N3—H4*N*···S6 | 0.89 | 2.46 | 3.327 (9) | 164 |
| N3—H5*N*···S4$^{ii}$ | 0.89 | 2.54 | 3.383 (8) | 158 |
| N3—H6*N*···S8$^{ii}$ | 0.89 | 2.48 | 3.307 (9) | 154 |
| N4—H7*N*···S6$^{iii}$ | 0.89 | 2.38 | 3.217 (10) | 157 |
| N4—H8*N*···S7$^{iv}$ | 0.89 | 2.39 | 3.240 (9) | 159 |
| N12—H12*N*···S3$^{v}$ | 0.89 | 2.41 | 3.259 (9) | 160 |
| N12—H13*N*···S2$^{i}$ | 0.89 | 2.36 | 3.184 (10) | 154 |
| N13—H14*N*···S4 | 0.89 | 2.49 | 3.316 (8) | 155 |
| N13—H15*N*···S8 | 0.89 | 2.62 | 3.425 (8) | 152 |
| N13—H16*N*···S2$^{vi}$ | 0.89 | 2.38 | 3.267 (8) | 172 |
| N14—H19*N*···S5$^{vii}$ | 0.89 | 2.59 | 3.410 (9) | 155 |

Symmetry codes: (i) $-x+1, -y+1, z+\frac{1}{2}$; (ii) $-x+1, -y, z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y, z$; (iv) $-x+\frac{1}{2}, y, z+\frac{1}{2}$; (v) $x-\frac{1}{2}, -y+1, z$; (vi) $-x+\frac{3}{2}, y, z+\frac{1}{2}$; (vii) $x, y+1, z$.

Data collection: *DIF4* (Stoe & Cie, 1992); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2025).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, m1751-m1752 [ **doi:10.1107/S160053680702394X** ]

# 2,2',2''-Nitrilotris(ethylammonium) tetrathioantimonate

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## Comment

During the last decade numerous thioantimonate(III) compounds were synthesized under solvothermal conditions. An intriguing observation is that in the overwhelming cases Sb(III)$S_x$ ($x$ = 3 − 6) are observed and the number of compounds containing Sb(V)$S_4$ is comparably low. Some examples for thioantimonate(V) compounds are $(C_3H_{10}N)[NiSbS_4(C_6H_{18}N_4)]$ (Stähler & Bensch, 2002), $K_3SbS_4$ (Graf & Schäfer, 1976), $(NH_4)_3SbS_4$ (Graf *et al.*, 1969), $Na_3SbS_4 \times 9\ H_2O$ (Mereiter *et al.*, 1979), $KAg_2SbS_4$ (Schimek *et al.*, 1996), $Rb_3SbS_4$ (Bensch & Dürichen, 1996), $Cr(en)_3SbS_4$ (Schur *et al.*, 1998), $[Ni(en)_3]_2SbS_4NO_3$ (en = ethylenediamine) (Schur & Bensch, 2000), $[Mn(C_6H_{14}N_2)_3]_2[Mn(C_6H_{14}N_2)_2(SbS_4)_2] \times 6\ H_2O$ and [Mn(tren)(trenH)]$SbS_4$ (tren = tris(2-ethyl)amine)(Schaefer *et al.*, 2003), $[Ni(en)_3(enH)]SbS_4$ (Jia *et al.*, 2004), $[Sm(en)_4]SbS_4 \times 0.5$ en (Jia, Zhu *et al.*, 2005), $[Ln(en)_3(H_2O)_x(\mu_{3-x}SbS_4)]$ (Ln = La, $x$ = 0; Ln = Nd, $x$ = 1) and $[Ln(en)_4]SbS_4 \times 0.5$en (Ln = Eu, Dy, Yb) (Jia, Zhao *et al.*, 2005. There is also one example where Sb(III) and Sb(V) species coexist in a common anion, *i.e.*, in $[Ni(dien)_2]_2Sb_4S_9$ (*R*. Stähler *et al.*, 2002). During our ongoing work in the field of solvothermal syntheses of new thioantimonates the title compound was prepared. In the crystal structure of $(C_6H_{21}N_4)SbS_4$, discrete tetrathioantimonate anions and tris(2-ethylammonium)amine cations coexist. There are two crystallographically independent cations and anions in the asymmetric unit, all of them located in general positions (Fig. 1). The tetrathioantimonate anions show slightly distorted tetrahedral geometry (Table 1), but the values are in the range reported for the above mentioned compounds containing this anion. The tetrathioantimonate anions and the organic cations are arranged in layers, which are parallel to the a b plane (Fig. 2). These layers are interconnected by intermolecular N—H···S hydrogen bonding (Table 2). The Sb(1)$S_4$ anion has two such S···H contacts to the first and four contacts to the second crystallographically independent cation, whereas for the Sb(2)$S_4$ anion it is the other way around.

## Experimental

A mixture of 0.5 mmol $MnSb_2S_4$, 0.5 mmol $(NH_4)_2MoS_4$, 3 mmol S and 5 ml concentrated tris(2-aminoethyl)amine were heated at 413 K for 7 d followed by cooling to room temperature. The product was identified by X-ray powder diffraction and contained two different phases in approximately equivalent amount: red crystals of [Mn(tren)(trenH)]$SbS_4$ (tren = tris(2-ethyl)amine)(Schaefer *et al.*, 2003), and yellow crystals of the title compound.

## Refinement

All hydrogen atoms were positioned with idealized geometry (amine H atoms allowed to rotate but not to tip) and were refined with fixed isotropic displacement parameters ($U_{iso}$(H) = 1.2 $U_{eq}$(C)) or 1.5 $U_{eq}$(N)) using a riding model with d(C—H) = 0.97 Å and d(N—H) = 0.89 Å. In this structure a pseudo mirror plane and a pseudo center of symmetry are found, but refinement of the structure in the centrosymmetric space group *Pnma* was not successful and leads to pronounced disorder. The absolue structure was determined on the basis of 544 Friedel pairs.

## Figures

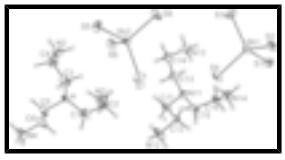

Fig. 1. : Crystal structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level.

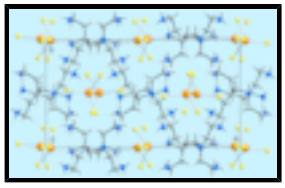

Fig. 2. Crystal structure of the title compound with view along the *c* axis (hydrogen bonding is not shown for clarity).

## 2,2',2''-Nitrilotris(ethylammonium) tetrathioantimonate

*Crystal data*

| | |
|---|---|
| $(C_6H_{21}N_4)[SbS_4]$ | $F_{000}$ = 1600 |
| $M_r$ = 399.26 | $D_x$ = 1.828 Mg m$^{-3}$ |
| Orthorhombic, *Pca*2$_1$ | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: P 2c -2ac | Cell parameters from 132 reflections |
| *a* = 19.7872 (13) Å | θ = 13–20º |
| *b* = 10.8571 (10) Å | μ = 2.46 mm$^{-1}$ |
| *c* = 13.5055 (8) Å | *T* = 293 (2) K |
| *V* = 2901.4 (4) Å$^3$ | Block, orange |
| *Z* = 8 | 0.2 × 0.1 × 0.1 mm |

*Data collection*

| | |
|---|---|
| Stoe AEDII diffractometer | $R_{int}$ = 0.023 |
| Radiation source: fine-focus sealed tube | $\theta_{max}$ = 27.0º |
| Monochromator: graphite | $\theta_{min}$ = 1.9º |
| *T* = 293(2) K | *h* = −25→14 |
| Phi scans | *k* = −13→1 |
| Absorption correction: numerical (X-Shape; Stoe & Cie, 1998) | *l* = −17→1 |
| $T_{min}$ = 0.738, $T_{max}$ = 0.772 | 4 standard reflections |
| 6750 measured reflections | every 2 h min |
| 3571 independent reflections | intensity decay: none |
| 2898 reflections with *I* > 2σ(*I*) | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | $w = 1/[\sigma^2(F_o^2) + (0.0261P)^2 + 3.7053P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.065$ | $(\Delta/\sigma)_{max} = 0.001$ |
| $S = 1.03$ | $\Delta\rho_{max} = 0.88$ e Å$^{-3}$ |
| 3571 reflections | $\Delta\rho_{min} = -0.55$ e Å$^{-3}$ |
| 277 parameters | Extinction correction: none |
| 1 restraint | Absolute structure: Flack (1983), 544 Friedel pairs |
| Primary atom site location: structure-invariant direct methods | Flack parameter: 0.04 (5) |
| Secondary atom site location: difference Fourier map | |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on $F^2$, conventional *R*-factors *R* are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma($F^2$) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Sb1 | 0.72266 (4) | 0.49351 (4) | 0.64960 (3) | 0.0204 (2) |
| S1 | 0.75959 (10) | 0.6794 (2) | 0.7180 (2) | 0.0305 (5) |
| S2 | 0.7352 (2) | 0.50371 (16) | 0.4781 (4) | 0.0289 (9) |
| S3 | 0.78342 (12) | 0.3293 (3) | 0.7154 (3) | 0.0388 (6) |
| S4 | 0.60811 (12) | 0.4725 (2) | 0.6887 (3) | 0.0318 (5) |
| Sb2 | 0.52627 (4) | 0.00637 (4) | 0.83030 (3) | 0.0196 (2) |
| S5 | 0.48991 (11) | −0.1799 (2) | 0.7655 (3) | 0.0338 (5) |
| S6 | 0.5162 (2) | −0.00107 (17) | 1.0015 (4) | 0.0319 (10) |
| S7 | 0.46215 (11) | 0.1664 (2) | 0.7672 (2) | 0.0366 (6) |
| S8 | 0.64036 (11) | 0.0315 (2) | 0.7919 (3) | 0.0338 (5) |
| N1 | 0.2974 (6) | −0.0028 (5) | 1.0178 (10) | 0.023 (3) |
| C1 | 0.2972 (4) | 0.1250 (6) | 0.9832 (6) | 0.0345 (17) |
| H1A | 0.2898 | 0.1259 | 0.9122 | 0.041* |
| H1B | 0.2599 | 0.1684 | 1.0140 | 0.041* |
| C2 | 0.3586 (5) | 0.1882 (9) | 1.0049 (9) | 0.034 (3) |
| H2A | 0.3548 | 0.2732 | 0.9833 | 0.040* |
| H2B | 0.3954 | 0.1503 | 0.9686 | 0.040* |
| N2 | 0.3745 (4) | 0.1851 (7) | 1.1154 (6) | 0.0354 (18) |
| H1N | 0.3463 | 0.2350 | 1.1475 | 0.053* |
| H2N | 0.4169 | 0.2099 | 1.1252 | 0.053* |
| H3N | 0.3697 | 0.1086 | 1.1379 | 0.053* |
| C3 | 0.3304 (4) | −0.0849 (7) | 0.9525 (7) | 0.0362 (18) |

| | | | | |
|---|---|---|---|---|
| H3A | 0.3690 | −0.0425 | 0.9242 | 0.043* |
| H3B | 0.2997 | −0.1038 | 0.8987 | 0.043* |
| C4 | 0.3538 (5) | −0.1994 (9) | 0.9943 (10) | 0.035 (3) |
| H4A | 0.3803 | −0.2422 | 0.9446 | 0.042* |
| H4B | 0.3148 | −0.2502 | 1.0091 | 0.042* |
| N3 | 0.3965 (4) | −0.1871 (7) | 1.0885 (7) | 0.039 (2) |
| H4N | 0.4338 | −0.1444 | 1.0751 | 0.059* |
| H5N | 0.4078 | −0.2617 | 1.1102 | 0.059* |
| H6N | 0.3727 | −0.1482 | 1.1348 | 0.059* |
| C5 | 0.2287 (4) | −0.0471 (7) | 1.0443 (6) | 0.0316 (16) |
| H5A | 0.1979 | −0.0278 | 0.9905 | 0.038* |
| H5B | 0.2299 | −0.1359 | 1.0515 | 0.038* |
| C6 | 0.2022 (8) | 0.0076 (8) | 1.1366 (16) | 0.045 (4) |
| H6A | 0.2329 | −0.0116 | 1.1904 | 0.054* |
| H6B | 0.2010 | 0.0964 | 1.1294 | 0.054* |
| N4 | 0.1343 (4) | −0.0365 (7) | 1.1625 (8) | 0.0404 (17) |
| H7N | 0.1091 | −0.0411 | 1.1080 | 0.061* |
| H8N | 0.1153 | 0.0156 | 1.2051 | 0.061* |
| H9N | 0.1373 | −0.1108 | 1.1901 | 0.061* |
| N11 | 0.5448 (7) | 0.5007 (4) | 0.9564 (11) | 0.024 (3) |
| C11 | 0.4766 (4) | 0.4589 (7) | 0.9377 (6) | 0.0307 (15) |
| H11A | 0.4466 | 0.4905 | 0.9885 | 0.037* |
| H11B | 0.4750 | 0.3696 | 0.9399 | 0.037* |
| C12 | 0.4534 (6) | 0.5041 (5) | 0.8357 (12) | 0.021 (2) |
| H12A | 0.4578 | 0.5930 | 0.8322 | 0.025* |
| H12B | 0.4818 | 0.4683 | 0.7847 | 0.025* |
| N12 | 0.3826 (4) | 0.4685 (7) | 0.8190 (8) | 0.0431 (18) |
| H11N | 0.3811 | 0.3936 | 0.7924 | 0.065* |
| H12N | 0.3632 | 0.5222 | 0.7780 | 0.065* |
| H13N | 0.3605 | 0.4683 | 0.8764 | 0.065* |
| C13 | 0.5806 (4) | 0.4188 (7) | 1.0288 (6) | 0.0324 (17) |
| H13A | 0.5505 | 0.3994 | 1.0833 | 0.039* |
| H13B | 0.6195 | 0.4617 | 1.0555 | 0.039* |
| C14 | 0.6045 (5) | 0.2953 (9) | 0.9772 (11) | 0.037 (3) |
| H14A | 0.6290 | 0.2453 | 1.0248 | 0.044* |
| H14B | 0.5653 | 0.2490 | 0.9556 | 0.044* |
| N13 | 0.6473 (3) | 0.3202 (7) | 0.8936 (6) | 0.0348 (18) |
| H14N | 0.6240 | 0.3614 | 0.8480 | 0.052* |
| H15N | 0.6619 | 0.2495 | 0.8681 | 0.052* |
| H16N | 0.6825 | 0.3653 | 0.9130 | 0.052* |
| C15 | 0.5440 (4) | 0.6289 (6) | 0.9983 (6) | 0.0311 (16) |
| H15A | 0.5366 | 0.6247 | 1.0692 | 0.037* |
| H15B | 0.5069 | 0.6747 | 0.9692 | 0.037* |
| C16 | 0.6113 (4) | 0.6975 (10) | 0.9776 (10) | 0.032 (3) |
| H16A | 0.6069 | 0.7840 | 0.9942 | 0.039* |
| H16B | 0.6474 | 0.6626 | 1.0172 | 0.039* |
| N14 | 0.6263 (4) | 0.6837 (8) | 0.8743 (7) | 0.051 (2) |
| H17N | 0.6168 | 0.6071 | 0.8553 | 0.077* |
| H18N | 0.6699 | 0.6991 | 0.8639 | 0.077* |

| | | | | |
|---|---|---|---|---|
| H19N | 0.6014 | 0.7365 | 0.8394 | 0.077* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sb1 | 0.0172 (4) | 0.0241 (4) | 0.0197 (5) | 0.00031 (15) | 0.0006 (4) | −0.0019 (3) |
| S1 | 0.0332 (10) | 0.0250 (9) | 0.0333 (11) | −0.0025 (9) | −0.0050 (12) | −0.0036 (9) |
| S2 | 0.0218 (15) | 0.042 (2) | 0.023 (2) | 0.0020 (7) | 0.0040 (17) | 0.0018 (7) |
| S3 | 0.0500 (13) | 0.0341 (11) | 0.0321 (12) | 0.0173 (10) | −0.0011 (14) | 0.0031 (10) |
| S4 | 0.0169 (10) | 0.0494 (9) | 0.0290 (12) | −0.0009 (10) | 0.0043 (8) | −0.0011 (14) |
| Sb2 | 0.0160 (4) | 0.0236 (4) | 0.0192 (5) | −0.00037 (14) | −0.0011 (4) | 0.0003 (3) |
| S5 | 0.0403 (12) | 0.0253 (9) | 0.0357 (12) | −0.0075 (10) | −0.0061 (13) | −0.0007 (10) |
| S6 | 0.0282 (17) | 0.050 (2) | 0.017 (2) | −0.0088 (8) | −0.0049 (17) | 0.0021 (7) |
| S7 | 0.0477 (12) | 0.0282 (10) | 0.0340 (12) | 0.0110 (10) | −0.0064 (15) | −0.0007 (9) |
| S8 | 0.0183 (10) | 0.0532 (10) | 0.0298 (12) | −0.0053 (11) | 0.0017 (9) | −0.0123 (14) |
| N1 | 0.008 (4) | 0.041 (6) | 0.020 (6) | 0.000 (2) | −0.004 (4) | 0.000 (2) |
| C1 | 0.038 (4) | 0.038 (4) | 0.028 (4) | −0.008 (4) | −0.009 (4) | 0.014 (3) |
| C2 | 0.045 (5) | 0.019 (4) | 0.036 (6) | 0.004 (4) | 0.021 (5) | 0.011 (4) |
| N2 | 0.039 (4) | 0.028 (3) | 0.039 (5) | 0.001 (3) | −0.006 (3) | −0.001 (3) |
| C3 | 0.027 (4) | 0.048 (5) | 0.033 (5) | 0.003 (3) | 0.003 (4) | −0.008 (4) |
| C4 | 0.041 (5) | 0.029 (5) | 0.034 (6) | 0.001 (4) | 0.002 (5) | −0.008 (4) |
| N3 | 0.043 (4) | 0.024 (3) | 0.051 (5) | 0.008 (3) | −0.007 (4) | −0.001 (3) |
| C5 | 0.023 (4) | 0.042 (4) | 0.030 (4) | −0.012 (4) | 0.000 (4) | −0.004 (4) |
| C6 | 0.020 (6) | 0.069 (9) | 0.047 (10) | −0.004 (4) | −0.001 (7) | −0.006 (5) |
| N4 | 0.029 (4) | 0.063 (4) | 0.029 (4) | −0.005 (4) | 0.000 (3) | 0.004 (5) |
| N11 | 0.028 (5) | 0.017 (5) | 0.029 (6) | 0.0012 (19) | −0.012 (5) | −0.001 (2) |
| C11 | 0.020 (4) | 0.045 (4) | 0.028 (4) | 0.000 (4) | −0.001 (3) | 0.000 (4) |
| C12 | 0.018 (5) | 0.025 (4) | 0.019 (5) | −0.005 (2) | −0.007 (5) | 0.006 (3) |
| N12 | 0.020 (3) | 0.083 (4) | 0.027 (4) | −0.009 (4) | −0.006 (3) | 0.003 (5) |
| C13 | 0.036 (4) | 0.039 (4) | 0.022 (4) | −0.003 (3) | −0.004 (4) | 0.000 (3) |
| C14 | 0.037 (5) | 0.023 (5) | 0.051 (7) | 0.001 (4) | −0.003 (5) | 0.006 (4) |
| N13 | 0.037 (4) | 0.033 (3) | 0.035 (4) | 0.000 (3) | −0.010 (3) | −0.007 (3) |
| C15 | 0.024 (4) | 0.038 (4) | 0.031 (4) | 0.011 (3) | −0.007 (3) | −0.006 (3) |
| C16 | 0.018 (4) | 0.037 (5) | 0.042 (7) | 0.000 (4) | −0.005 (4) | −0.001 (5) |
| N14 | 0.050 (5) | 0.036 (4) | 0.068 (6) | 0.000 (4) | 0.024 (4) | −0.001 (4) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Sb1—S3 | 2.326 (3) | C6—H6B | 0.9700 |
| Sb1—S2 | 2.333 (5) | N4—H7N | 0.8900 |
| Sb1—S1 | 2.337 (3) | N4—H8N | 0.8900 |
| Sb1—S4 | 2.338 (3) | N4—H9N | 0.8900 |
| Sb2—S7 | 2.314 (3) | N11—C11 | 1.445 (14) |
| Sb2—S5 | 2.319 (3) | N11—C13 | 1.499 (13) |
| Sb2—S6 | 2.323 (5) | N11—C15 | 1.503 (9) |
| Sb2—S8 | 2.332 (3) | C11—C12 | 1.532 (17) |
| N1—C3 | 1.414 (13) | C11—H11A | 0.9700 |
| N1—C1 | 1.463 (9) | C11—H11B | 0.9700 |
| N1—C5 | 1.486 (13) | C12—N12 | 1.472 (13) |

| | | | |
|---|---|---|---|
| C1—C2 | 1.427 (13) | C12—H12A | 0.9700 |
| C1—H1A | 0.9700 | C12—H12B | 0.9700 |
| C1—H1B | 0.9700 | N12—H11N | 0.8900 |
| C2—N2 | 1.525 (14) | N12—H12N | 0.8900 |
| C2—H2A | 0.9700 | N12—H13N | 0.8900 |
| C2—H2B | 0.9700 | C13—C14 | 1.583 (12) |
| N2—H1N | 0.8900 | C13—H13A | 0.9700 |
| N2—H2N | 0.8900 | C13—H13B | 0.9700 |
| N2—H3N | 0.8900 | C14—N13 | 1.437 (15) |
| C3—C4 | 1.441 (13) | C14—H14A | 0.9700 |
| C3—H3A | 0.9700 | C14—H14B | 0.9700 |
| C3—H3B | 0.9700 | N13—H14N | 0.8900 |
| C4—N3 | 1.534 (15) | N13—H15N | 0.8900 |
| C4—H4A | 0.9700 | N13—H16N | 0.8900 |
| C4—H4B | 0.9700 | C15—C16 | 1.551 (12) |
| N3—H4N | 0.8900 | C15—H15A | 0.9700 |
| N3—H5N | 0.8900 | C15—H15B | 0.9700 |
| N3—H6N | 0.8900 | C16—N14 | 1.434 (16) |
| C5—C6 | 1.48 (2) | C16—H16A | 0.9700 |
| C5—H5A | 0.9700 | C16—H16B | 0.9700 |
| C5—H5B | 0.9700 | N14—H17N | 0.8900 |
| C6—N4 | 1.469 (16) | N14—H18N | 0.8900 |
| C6—H6A | 0.9700 | N14—H19N | 0.8900 |
| S3—Sb1—S2 | 111.14 (11) | C6—N4—H7N | 109.5 |
| S3—Sb1—S1 | 110.43 (13) | C6—N4—H8N | 109.5 |
| S2—Sb1—S1 | 108.56 (10) | H7N—N4—H8N | 109.5 |
| S3—Sb1—S4 | 109.88 (11) | C6—N4—H9N | 109.5 |
| S2—Sb1—S4 | 109.39 (14) | H7N—N4—H9N | 109.5 |
| S1—Sb1—S4 | 107.34 (10) | H8N—N4—H9N | 109.5 |
| S7—Sb2—S5 | 110.24 (12) | C11—N11—C13 | 111.6 (7) |
| S7—Sb2—S6 | 110.23 (12) | C11—N11—C15 | 110.3 (9) |
| S5—Sb2—S6 | 108.60 (11) | C13—N11—C15 | 107.9 (9) |
| S7—Sb2—S8 | 111.17 (11) | N11—C11—C12 | 109.6 (9) |
| S5—Sb2—S8 | 108.58 (10) | N11—C11—H11A | 109.8 |
| S6—Sb2—S8 | 107.95 (14) | C12—C11—H11A | 109.8 |
| C3—N1—C1 | 113.5 (10) | N11—C11—H11B | 109.8 |
| C3—N1—C5 | 111.7 (7) | C12—C11—H11B | 109.8 |
| C1—N1—C5 | 112.4 (8) | H11A—C11—H11B | 108.2 |
| C2—C1—N1 | 112.9 (8) | N12—C12—C11 | 109.8 (10) |
| C2—C1—H1A | 109.0 | N12—C12—H12A | 109.7 |
| N1—C1—H1A | 109.0 | C11—C12—H12A | 109.7 |
| C2—C1—H1B | 109.0 | N12—C12—H12B | 109.7 |
| N1—C1—H1B | 109.0 | C11—C12—H12B | 109.7 |
| H1A—C1—H1B | 107.8 | H12A—C12—H12B | 108.2 |
| C1—C2—N2 | 111.5 (8) | C12—N12—H11N | 109.5 |
| C1—C2—H2A | 109.3 | C12—N12—H12N | 109.5 |
| N2—C2—H2A | 109.3 | H11N—N12—H12N | 109.5 |
| C1—C2—H2B | 109.3 | C12—N12—H13N | 109.5 |
| N2—C2—H2B | 109.3 | H11N—N12—H13N | 109.5 |

| | | | |
|---|---|---|---|
| H2A—C2—H2B | 108.0 | H12N—N12—H13N | 109.5 |
| C2—N2—H1N | 109.5 | N11—C13—C14 | 110.9 (8) |
| C2—N2—H2N | 109.5 | N11—C13—H13A | 109.5 |
| H1N—N2—H2N | 109.5 | C14—C13—H13A | 109.5 |
| C2—N2—H3N | 109.5 | N11—C13—H13B | 109.5 |
| H1N—N2—H3N | 109.5 | C14—C13—H13B | 109.5 |
| H2N—N2—H3N | 109.5 | H13A—C13—H13B | 108.1 |
| N1—C3—C4 | 116.6 (9) | N13—C14—C13 | 111.2 (7) |
| N1—C3—H3A | 108.1 | N13—C14—H14A | 109.4 |
| C4—C3—H3A | 108.1 | C13—C14—H14A | 109.4 |
| N1—C3—H3B | 108.1 | N13—C14—H14B | 109.4 |
| C4—C3—H3B | 108.1 | C13—C14—H14B | 109.4 |
| H3A—C3—H3B | 107.3 | H14A—C14—H14B | 108.0 |
| C3—C4—N3 | 115.2 (8) | C14—N13—H14N | 109.5 |
| C3—C4—H4A | 108.5 | C14—N13—H15N | 109.5 |
| N3—C4—H4A | 108.5 | H14N—N13—H15N | 109.5 |
| C3—C4—H4B | 108.5 | C14—N13—H16N | 109.5 |
| N3—C4—H4B | 108.5 | H14N—N13—H16N | 109.5 |
| H4A—C4—H4B | 107.5 | H15N—N13—H16N | 109.5 |
| C4—N3—H4N | 109.5 | N11—C15—C16 | 111.6 (8) |
| C4—N3—H5N | 109.5 | N11—C15—H15A | 109.3 |
| H4N—N3—H5N | 109.5 | C16—C15—H15A | 109.3 |
| C4—N3—H6N | 109.5 | N11—C15—H15B | 109.3 |
| H4N—N3—H6N | 109.5 | C16—C15—H15B | 109.3 |
| H5N—N3—H6N | 109.5 | H15A—C15—H15B | 108.0 |
| C6—C5—N1 | 113.4 (8) | N14—C16—C15 | 107.6 (9) |
| C6—C5—H5A | 108.9 | N14—C16—H16A | 110.2 |
| N1—C5—H5A | 108.9 | C15—C16—H16A | 110.2 |
| C6—C5—H5B | 108.9 | N14—C16—H16B | 110.2 |
| N1—C5—H5B | 108.9 | C15—C16—H16B | 110.2 |
| H5A—C5—H5B | 107.7 | H16A—C16—H16B | 108.5 |
| N4—C6—C5 | 113.2 (11) | C16—N14—H17N | 109.5 |
| N4—C6—H6A | 108.9 | C16—N14—H18N | 109.5 |
| C5—C6—H6A | 108.9 | H17N—N14—H18N | 109.5 |
| N4—C6—H6B | 108.9 | C16—N14—H19N | 109.5 |
| C5—C6—H6B | 108.9 | H17N—N14—H19N | 109.5 |
| H6A—C6—H6B | 107.7 | H18N—N14—H19N | 109.5 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H1N···S1$^{i}$ | 0.89 | 2.48 | 3.336 (7) | 161 |
| N3—H4N···S6 | 0.89 | 2.46 | 3.327 (9) | 164 |
| N3—H5N···S4$^{ii}$ | 0.89 | 2.54 | 3.383 (8) | 158 |
| N3—H6N···S8$^{ii}$ | 0.89 | 2.48 | 3.307 (9) | 154 |
| N4—H7N···S6$^{iii}$ | 0.89 | 2.38 | 3.217 (10) | 157 |
| N4—H8N···S7$^{iv}$ | 0.89 | 2.39 | 3.240 (9) | 159 |
| N12—H12N···S3$^{v}$ | 0.89 | 2.41 | 3.259 (9) | 160 |

| | | | | |
|---|---|---|---|---|
| N12—H13N···S2$^{i}$ | 0.89 | 2.36 | 3.184 (10) | 154 |
| N13—H14N···S4 | 0.89 | 2.49 | 3.316 (8) | 155 |
| N13—H15N···S8 | 0.89 | 2.62 | 3.425 (8) | 152 |
| N13—H16N···S2$^{vi}$ | 0.89 | 2.38 | 3.267 (8) | 172 |
| N14—H19N···S5$^{vii}$ | 0.89 | 2.59 | 3.410 (9) | 155 |

Symmetry codes: (i) −*x*+1, −*y*+1, *z*+1/2; (ii) −*x*+1, −*y*, *z*+1/2; (iii) *x*−1/2, −*y*, *z*; (iv) −*x*+1/2, *y*, *z*+1/2; (v) *x*−1/2, −*y*+1, *z*; (vi) −*x*+3/2, *y*, *z*+1/2; (vii) *x*, *y*+1, *z*.

**Fig. 1**

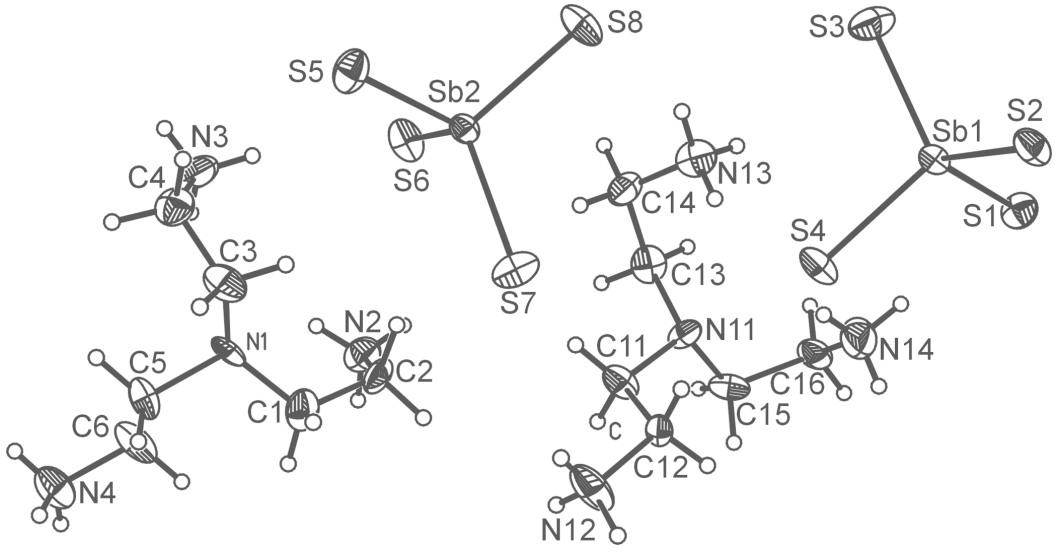
S8
S5
Sb2
S6
S7
S3
Sb1
S2
S4
S1
N3
C4
C3
N2
N1
C5
C2
C1
C6
N4
N13
C14
C13
N11
C11
C16
N14
C15
C12
N12

**Fig. 2**