Crystal structure of $\mu_3$-tetrathioantimonato-tris-[(cyclam)zinc(II)] tetrathioantimonate acetonitrile disolvate dihydrate showing Zn disorder over the cyclam ring planes (cyclam = 1,4,8,11-tetraazacyclotetradecane)

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Reaction of Zn(ClO₄)₂·6H₂O with cyclam (cyclam = 1,4,8,11-tetraazacyclotetradecane, C₆₁H₆₄N₄) and Na₃SbS₄ in an acetonitrile/water mixture led to the formation of crystals of the title compound, [Zn₃(SbS₄)(C₆₁H₆₄N₄)₃](SbS₄)·2CH₃CN·2H₂O or [(Zn-cyclam)₃(SbS₄)₂](H₂O)₂(acetonitrile)₂. The set-up of the crystal structure is similar to that of [(Zn-cyclam)(SbS₄)₃]8H₂O reported recently [Danker et al. (2021). Dalton Trans. 50, 18107–18117]. The crystal structure of the title compound consists of three crystallographically independent Zn²⁺ cations (each disordered around centers of inversion), three centrosymmetric cyclam ligands, one SbS₄³⁻ anion, one water and one acetonitrile molecule occupying general positions. The acetonitrile molecule is equally disordered over two sets of sites. Each Zn²⁺ cation is bound to four nitrogen atoms of a cyclam ligand and one sulfur atom of the SbS₄³⁻ anion within a distorted square-pyramidal coordination. The cation disorder of the [Zn(cyclam)]²⁺ complexes is discussed in detail and is also observed in other compounds, where identical ligands are located above and below the [Zn(cyclam)]²⁺ plane. In the title compound, the building units are arranged in layers parallel to the bc plane forming pores in which the acetonitrile solvate molecules are located. Intermolecular C—H···S hydrogen bonding links these units to the SbS₄³⁻ anions. Between the layers, additional water solvate molecules are present that act as acceptor and donor groups for intermolecular N—H···O and O—H···S hydrogen bonding.

1. Chemical context

For several years, chalcogenidometallates and chalcogenides with inorganic and/or organic cations have been investigated intensively because several of them show promising physical properties (Feng et al., 2021; Lokhande et al., 2019; Thiele et al., 2017; Feng et al., 2016; Si et al., 2016; Bensch & Kanatzidis, 2012). Hence, numerous such compounds have been reported in the literature (Sheldrick & Wachhold, 1998; Bensch et al., 1997; Dehnen & Melullis, 2007; Wang et al., 2016; Zhou, 2016; Zhu & Dai, 2017; Nie et al., 2017). An important class of chalcogenidometallates are represented by thioantimonates, which exhibit a pronounced structural variability with different coordination numbers of the Sb⁵⁺ atom and networks of different dimensionality (Spetzler et al., 2004; Jia et al., 2004; Powell et al., 2005; Engelke et al., 2004; Zhang et al., 2007; Liu & Zhou, 2011), with some of them having potential for future applications (Zhou et al., 2019).
For several years, we have been interested in the syntheses and structural behaviors of thioantimonate(V) compounds (Stähler et al., 2001; Schur et al., 2001; Pienack et al., 2008). In the early stages of these studies, such compounds were prepared at elevated temperatures under solvothermal conditions but subsequently, new synthetic approaches using soluble precursors such as Na₃SbS₄·9H₂O were developed, which allowed the synthesis of new thioantimonates at room temperature (Anderer et al., 2016). The major advantage of this route is that, under these conditions, thioantimonate compounds containing Sb^{V} atoms can be prepared selectively, which is otherwise difficult to achieve. In most cases, we used transition-metal complexes (TMCs) as counter-cations. In this context, cyclam (cyclam = 1,4,8,11-tetraazaacyclotetradecane) became of interest as a ligand. The formed complex cations are in a fourfold coordination environment and provide additional coordination sites for thioantimonate anions, which became of interest as a ligand. The formed complex cations are octahedrally coordinated by the four N atoms of the cyclam ligand and by two S atoms of the tetrathioantimonate anion. Following this synthetic approach, we reacted cyclam with Na₃SbS₄·9H₂O and different transition-metal salts, which led to the formation of compounds with compositions: [(Cu-cyclam)₃(SbS₄)₂]·2H₂O, [(Zn-cyclam)₃(SbS₄)₂]·8H₂O (Danker et al., 2021) and [(Co-cyclam)₃(SbS₄)₂]·(H₂O)₂(acetonitrile)₂ (Näther et al., 2022). In the crystal structure of the cobalt and copper compounds, the metal cations are octahedrally coordinated by the four N atoms of the cyclam ligand and by two S atoms of the tetrathioantimonate(V) anions in *trans*-positions. Each of the SbS₄^{V} anions coordinates to three crystallographically independent [M(cyclam)]⁺⁺ cations (M = Cu and Co), linking the cations and anions into layers. Within these layers, channels are formed in which water or acetonitrile solvent molecules are located. These layers are connected into a three-dimensional network by intermolecular hydrogen bonding via water molecules. In the case of M = Zn, a different coordination is observed, because this cation is shifted out of the N₄ plane of the cyclam ligand and because of the center of inversion is disordered over both ring planes (Danker et al., 2021). In this context, it is noted that such a disorder in Zn–cyclam complexes has already been observed in other, different compounds, but the structural consequences were not discussed in detail (see Database survey).

Figure 1
Part of the crystal structure of the title compound with labeling and displacement ellipsoids drawn at the 50% probability level. The hydrogen atoms were omitted for clarity; the disorder of the acetonitrile solvent molecule and the Zn^{2+} cations is shown with full and open bonds. [Symmetry codes: (i) −x + 1, −y, −z + 1; (ii) −x + 1, −y + 1, −z; (iii) −x, −y + 1, −z + 1.]

Table 1
Selected bond lengths (Å).

| Bond | Length (Å) |
|------|------------|
| Zn1—S1 | 2.4071 (2) |
| Zn2—N1 | 2.179 (2) |
| S3—Zn3 | 2.3622 (3) |
| Sb1—S3 | 2.3522 (6) |
| Sb1—S2 | 2.3214 (7) |
| Sb1—S1 | 2.3398 (6) |
| Sb1—S4 | 2.3049 (7) |
| Sb1—S8 | 2.093 (2) |

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

To find more examples of similar compounds, additional syntheses were performed. From an acetonitrile/water mixture, crystals of another Zn^{1+}–cyclam tetrathioantimonate(V) compound with composition [(Zn-cyclam)₃(SbS₄)₂(CH₃CN)₂(H₂O)](SbS₄) were obtained. Likewise, in this compound disorder of the Zn^{2+} cations is observed and the structural consequences are discussed in this contribution.

2. Structural commentary
The asymmetric unit of the title compound consists of three half cyclam ligands (completed by inversion symmetry), one SbS₄^{V} anion, one water solvent molecule, one disordered acetonitrile solvent molecule and three Zn^{2+} cations that are disordered around centers of inversion (Fig. 1). In contrast to [(Cu-cyclam)₃(SbS₄)₂]·2H₂O (Danker et al., 2021) and [(Co-cyclam)₃(SbS₄)₂](H₂O)₂(acetonitrile)₂ (Näther et al., 2022), in both of which the cations are located at the center of the cyclam ligand and have an octahedral coordination, in the title compound the Zn^{2+} cations are shifted out of the N₄ plane of the ligand by 0.4318 (6) Å (Zn1), 0.3751 (6) Å (Zn2) and 0.4998 (7) Å (Zn3). This means that each Zn^{2+} cation is in a...
fivefold coordination defined by the four N atoms of the cyclam ligand in the basal plane and one S atom of the SbS₄³⁻ anions in the apical position (Fig. 2, Table 1). The Zn–S distances to the Zn²⁺ cation on the other side of the N₄ plane are 3.2748 (8) Å (Zn1), 3.2063 (9) Å (Zn2) and 3.4234 (9) Å (Zn3), which are much too long for a significant interaction. Because all of the Zn²⁺ cations are disordered around centers of inversion, the connectivity within the crystal structure is difficult to define. In principle, the SbS₄³⁻ anions can coordinate to one, two or three [Zn(cyclam)]²⁺ cations (Fig. 3). If the disorder were not present and the Zn²⁺ cations were located on centers of inversion in the planes of the cyclam ligands, layers would be formed (Fig. 4A) like in [(Cu-cyclam)₃(SbS₄)₂]·20H₂O (Danker et al., 2021) or [(Co-cyclam)₃(SbS₄)₂](H₂O)₂(acetonitrile)₂ (Na¨ther et al., 2022) reported recently. In the case of [(Zn-cyclam)₃(SbS₄)₂](H₂O)₂(acetonitrile)₂, one can argue that each of the SbS₄³⁻ anions acts as a tri-coordinating ligand like in the Cu and Co compounds and is connected to each of the [Zn(cyclam)]²⁺ cations, forming [(Zn-cyclam)_3·(SbS₄)]³⁺ moieties. However, in this case, an equivalent amount of non-coordinating SbS₄³⁻ anions must be present for charge balance as well as for the correct ratio between Zn-cyclam cations and tetrathioantimonate anions (Fig. 4B). Alternatively, the anion can coordinate to two cations forming [(Zn-cyclam)_2(SbS₄)]⁺ cations. Then, an equivalent amount of [(Zn-cyclam)(SbS₄)]⁻ anions must be present to have the correct ratio between Zn-cyclam and the tetrathioantimonate anions (Fig. 4C). The arrangement with [(Zn-

cyclam)_3(SbS₄)]³⁺ cations and an SbS₄³⁻ anion appears to be more likely because of the higher charge, but this is in fact difficult to prove. This possibility can also not be verified from the Sb–S bond lengths because they are very similar for the thioantimonate anions, which is expected because they are averaged over the whole crystal structure (Table 1).

It is noted that such a cation disorder is also observed in other compounds containing [Zn(cyclam)]²⁺ cations, which includes [(Zn-cyclam)_3(SbS₄)₂]·8H₂O (Danker et al., 2021) and other compounds where identical anions are located above and below the N₄ plane of the [Zn(cyclam)]²⁺ cations (see Database survey). The reason for this disorder is still unclear. For [(Cu-cyclam)_3(SbS₄)₂]·20H₂O and [(Zn-cyclam)_3(SbS₄)₂]·8H₂O, DFT calculations were performed, which reasonably reproduced the octahedral coordination for the Cu²⁺ and the square-pyramidal coordination for the Zn²⁺ cations (Danker et al., 2021). Moreover, these calculations also revealed that in the Cu compound, the attractive dispersion interactions between the cyclam ligand and the SbS₄³⁻ anion contribute to the environment of the metal cation, which might be the reason for the different behavior of the Cu²⁺ and
the Zn²⁺ cations. Also, for very large cations it might be possible that they are shifted out of the center of the cyclam ring, because there is not enough space available within the ring plane. To examine whether the size of Zn²⁺ might be a reason for the shift out of the N₄ plane, we analyzed the ionic radii (Shannon, 1976) and found no significant differences for octahedrally coordinated Zn²⁺ (r = 0.74 Å), Co²⁺bas (r = 0.745 Å), Co²⁺bas (r = 0.65 Å) and Cu²⁺ (r = 0.73 Å). One may argue that in [(Co(cyclam)₃(SbS₄)₂(H₂O)₃(acetonitrile)₂], for which the spin state is not known, Co²⁺ is ordered because it adopts the low-spin state with a smaller ionic radius compared to Zn²⁺, and larger than for Co²⁺bas. Hence, the ionic radius is most probably not the driving force of the disorder of Zn²⁺. We also checked many other transition-metal cations in the form of their cyclam complexes, and there were no indications for metal disorder except in some of the Zn compounds, which suggests that such a disorder is limited to Zn²⁺ cations. Even for these compounds, only about 10% show disorder (see Database survey). A possible explanation for these observations might be the ligand field stabilization energy, which is zero for Zn²⁺ (electronic configuration 3d¹⁰), while it is reasonably large for Co²⁺ (3d⁷) and Cu²⁺ (3d⁹), resulting in a preference of the position of these cations within the N₄ plane. Because not all [Zn(cyclam)²⁺ complexes show disorder, secondary effects (sterical demands, packing) may also be responsible for the disorder.

3. Supramolecular features

The cations and anions are arranged into layers parallel to the bc plane in such a way that channels are formed in which the disordered acetonitrile solvate molecules are located. The latter are hydrogen-bonded to the tetrathioantimonate anions by intermolecular C—H···S interactions (Figs. 4 and 5, Table 2). One of the C—H···S angles is close to linearity, which indicates that this is a relatively strong interaction. The water molecules are located between the layers and are hydrogen-bonded to the tetrathioantimonate anions via comparatively strong intermolecular O—H···S interactions (Table 2). The water molecules also act as acceptors for strong N—H···O hydrogen bonding involving the NH hydrogen atoms of the cyclam ligands (Fig. 5, Table 2). The NH groups are also hydrogen-bonded to the S atoms of the tetrathioantimonate(V) groups. There are additional C—H···S interactions, but according to the the corresponding angles, it seems that these are only weak (Table 2).

4. Database survey

A search for structures of Zn²⁺—cyclam complexes in the Cambridge Structural Database (CSD version 5.42, last update November 2020; Groom et al., 2016) led to 34 hits but none of them contains SbS₄³⁻ anions. However, as mentioned above, one compound with composition [(Zn(cyclam)₃)(SbS₄)₂]·8H₂O has already been reported (Danker et al., 2021) but so far is not included in the database.

In one of the other structures, two [Zn(cyclam)²⁺ cations are linked by oxalate anions into a centrosymmetric dimer, which means that both O atoms are on the same side of one cyclam ring (FIHYEB; Jo et al., 2005). In all remaining structures, the Zn²⁺ cations seem to be sixfold coordinated with one monocoordinating donor atom at each side of the cyclam ring; however, for five of them no atomic coordinates are given (HEGNEM10, HEGNOW, HEGNOW10, VUSDUI20 and WARJAD). For these hits, it is difficult to decide whether disorder is present or not. In some of the entries, the Zn
disorder is mentioned in the database and this includes structures with the following refcodes: CUZHUA (Kato & Ito, 1985), in which the Zn$^{2+}$ cations are coordinated by methyl-carbonato anions from both sites, DITZIP (Ito et al., 1984), in which the cations are linked to two thiocyanate anions and HEGNEM, HEGNOW and VUSDUI10 (Porai-Koshits et al., 1994), in which chloride, bromide and iodine anions are located on each side of the cyclam ligand. It should be noted that, for the first structure determination of ZnCl$_2$(cyclam) (VUSDUI; Antsyshkina et al., 1991), no disorder is mentioned. One can assume that the disorder was overlooked and the Zn$^{2+}$ cation forced to be situated at the center of inversion. For the remaining structures, the two Zn—X bond lengths ($X = O, Cl, Br, I$) are identical in each case, which points to ordered structures. Nonetheless, in some cases the Zn$^{2+}$ cations are located on special positions and because no anisotropic displacement parameters are available in the corresponding CIFs, one cannot decide whether there are hints of disorder.

5. Synthesis and crystallization

**Synthesis of Na$_3$SbS$_4$·9H$_2$O (Schlippe’s salt)**

Na$_3$SbS$_4$·9H$_2$O was synthesized by adding 16.6 g (0.213 mol) of Na$_3$S·xH$_2$O (technical grade, purchased from Acros Organics) to 58 ml of demineralized water. This solution was heated to 333 K for 1 h and afterwards 19.6 g (0.058 mol) of Sb$_2$S$_3$ (98%, purchased from Alfa Aesar) and 3.69 g (0.115 mol) of sulfur (min. 99%, purchased from Alfa Aesar) were added. The reaction mixture was then heated to 343 K for 6 h, filtered off and the filtrate was stored overnight at room temperature. Light-yellow-colored crystals formed overnight, which were filtered off, washed with small amounts of water and dried in vacuo.

**Synthesis of tris(cyclam-zinc(II)-bis-tetrathioantimonate)-bis water-bis-acetonitrile solvate**

Single crystals of the title compound were serendipitously obtained by dissolving 10 mg (0.274 mmol) of Zn(ClO$_4$)$_2$·6H$_2$O (purchased from Alfa Aesar) and 10 mg (0.05 mmol) of cyclam (purchased from Strem Chemicals) in 2 ml of acetonitrile (purchased from Merck) to which 20 mg (0.14 mmol) of Na$_3$SbS$_4$·9H$_2$O dissolved in 1 ml of water were added. After storing this mixture for 3 d at room temperature, a few colorless crystals of the title compound were obtained.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The C—H and N—H hydrogen atoms were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip) and were refined with $U_{	ext{iso}}$(H) = 1.2 $U_{	ext{eq}}$(C,N) (1.5 for methyl H atoms) using a riding model. The water hydrogen atoms were located in a difference-Fourier map, and their bond lengths set to ideal values with $U_{	ext{eq}}$(H) = 1.5 $U_{	ext{eq}}$(O) using a riding model. The acetonitrile molecule was modeled as being equally disordered over two sets of sites and was refined using a split model with restraints for the geometry and the components of the anisotropic displacement parameters. Each of the three Zn$^{2+}$ cations was found to be disordered around a center of inversion and thus was refined with half occupancy.

The crystal structure was alternatively refined in space group $P1$ but the disorder remained the same. There were also no hints of superstructure reflections, and in the diffraction pattern diffuse scattering was not observed.

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Crystal structure of $\mu_3$-tetrathioantimonato-tris[(cyclam)zinc(II)] tetrathioantimonate acetonitrile disolvate dihydrate showing Zn disorder over the cyclam ring planes (cyclam = 1,4,8,11-tetraazacyclotetradecane)

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Computing details

Data collection: *X-AREA* (Stoe, 2008); cell refinement: *X-AREA* (Stoe, 2008); data reduction: *X-AREA* (Stoe, 2008); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2010).

$\mu_3$-Tetrathioantimonato-tris[(cyclam)zinc(II)] tetrathioantimonate acetonitrile disolvate dihydrate

**Crystal data**

$[\text{Zn}_3(\text{SbS}_4)(\text{C}_{10}\text{H}_{24}\text{N}_4)_3](\text{SbS}_4)\cdot2\text{CH}_3\text{CN}\cdot2\text{H}_2\text{O}$

$M_r = 1415.22$

Triclinic, $P\overline{1}$

$a = 8.7856$ (3) Å

$b = 13.1738$ (6) Å

$c = 14.0096$ (6) Å

$\alpha = 67.018$ (3)°

$\beta = 77.677$ (3)°

$\gamma = 84.220$ (3)°

$V = 1458.10$ (11) Å³

$Z = 1$

$F(000) = 720$

$D_x = 1.612$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 14357 reflections

$\theta = 1.6$–27.0°

$\mu = 2.46$ mm⁻¹

$T = 200$ K

Block, colorless

$0.16 \times 0.12 \times 0.09$ mm

**Data collection**

Stoe IPDS-2

ω scans

Absorption correction: numerical

(X-RED and X-SHAPE; Stoe, 2008)

$T_{min} = 0.562, T_{max} = 0.781$

14357 measured reflections

6303 independent reflections

5594 reflections with $I > 2\sigma(I)$

$R_{int} = 0.038$

$\theta_{max} = 27.0$°, $\theta_{min} = 1.6$°

$h = -10$–11

$k = -15$–16

$l = -17$–17

**Refinement**

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.077$

$S = 1.02$

6303 reflections

327 parameters

75 restraints

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/\sigma^2(F_c^2) + (0.0533P)^2$

where $P = (F_c^2 + 2F_S^2)/3$

$(\Delta\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.81$ e Å⁻³

$\Delta\rho_{min} = -0.80$ e Å⁻³
**Special details**

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

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

|   | x    | y    | z    | Uiso*/Ueq | Occ. (<1) |
|---|------|------|------|-----------|-----------|
| Sb1 | 0.25921 (2) | 0.30825 (2) | 0.30750 (2) | 0.02516 (6) |
| S1  | 0.48271 (7)  | 0.19527 (5)  | 0.31911 (5)  | 0.03152 (13) |
| S2  | 0.35056 (8)  | 0.48478 (5)  | 0.20520 (5)  | 0.03581 (15) |
| S3  | 0.13698 (8)  | 0.29796 (5)  | 0.47540 (5)  | 0.03425 (14) |
| S4  | 0.09240 (8)  | 0.25656 (7)  | 0.22862 (6)  | 0.04413 (17) |
| Zn1 | 0.49252 (6)  | 0.03467 (5)  | 0.47770 (4)  | 0.02883 (13) |
| N1  | 0.3114 (2)   | 0.05124 (19) | 0.58800 (17) | 0.0335 (5)   |
| H1  | 0.269523     | 0.126482     | 0.549863     | 0.040*       |
| C1  | 0.1818 (3)   | −0.0192 (2)  | 0.6033 (2)   | 0.0351 (6)   |
| H1A | 0.081605     | 0.016393     | 0.622325     | 0.042*       |
| H1B | 0.191006     | −0.090967    | 0.661824     | 0.042*       |
| N2  | 0.3388 (2)   | −0.08173 (19)| 0.46890 (18) | 0.0336 (5)   |
| H2  | 0.356027     | −0.158230    | 0.519687     | 0.040*       |
| C3  | 0.3665 (3)   | −0.0776 (2)  | 0.3609 (2)   | 0.0368 (6)   |
| H3A | 0.361440     | 0.000038     | 0.311335     | 0.044*       |
| H3B | 0.283808     | −0.118224    | 0.353400     | 0.044*       |
| C4  | 0.5251 (4)   | −0.1280 (2)  | 0.3323 (2)   | 0.0425 (6)   |
| H4A | 0.535867     | −0.200472    | 0.389591     | 0.051*       |
| H4B | 0.527222     | −0.141658    | 0.267327     | 0.051*       |
| C5  | 0.6657 (3)   | −0.0594 (2)  | 0.3143 (2)   | 0.0378 (6)   |
| H5A | 0.760284     | −0.092999    | 0.284689     | 0.045*       |
| H5B | 0.651608     | 0.015681     | 0.261789     | 0.045*       |
| Zn2 | 0.47865 (8)  | 0.48973 (6)  | 0.02914 (4)  | 0.03109 (15) |
| N11 | 0.3674 (3)   | 0.64170 (19)| −0.06257 (18)| 0.0355 (5)   |
| H11 | 0.420952     | 0.668281     | −0.138314    | 0.043*       |
| C11 | 0.2113 (3)   | 0.6021 (3)   | −0.0510 (2)  | 0.0435 (7)   |
| H11A| 0.151576     | 0.660530     | −0.098613    | 0.052*       |
| H11B| 0.154414     | 0.583893     | 0.022484     | 0.052*       |
| C12 | 0.2264 (3)   | 0.5006 (3)   | −0.0783 (2)  | 0.0424 (7)   |
| H12A| 0.121747     | 0.471917     | −0.068014    | 0.051*       |
| H12B| 0.276978     | 0.519890     | −0.153222    | 0.051*       |
| N12 | 0.3206 (3)   | 0.4154 (2)   | −0.00964 (17)| 0.0364 (5)   |
| H12 | 0.245116     | 0.382658     | 0.058312     | 0.044*       |
| C13 | 0.3676 (4)   | 0.3214 (3)   | −0.0419 (2)  | 0.0435 (6)   |
| H13A| 0.426065     | 0.348879     | −0.115575    | 0.052*       |
| H13B| 0.273100     | 0.285028     | −0.039931    | 0.052*       |
| Atomic displacement parameters ($\AA^2$) |
|----------------------------------------|
| $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
| Sb1   | 0.02327 (9) | 0.02630 (9) | 0.02246 (9) | 0.00062 (6) | $-0.00337$ (6) | $-0.00633$ (6) |
| S1    | 0.0276 (3)  | 0.0294 (3)  | 0.0303 (3)  | 0.0036 (2)  | $-0.0045$ (2)  | $-0.0051$ (2)  |
| S2    | 0.0441 (4)  | 0.0264 (3)  | 0.0292 (3)  | $-0.0021$ (2) | 0.0042 (3)  | $-0.0079$ (2)  |
|      |          |          |          |          |          |          |
|------|----------|----------|----------|----------|----------|----------|
| S3   | 0.0395 (3) | 0.0293 (3) | 0.0262 (3) | 0.0066 (2) | 0.0003 (2) | −0.0074 (2) |
| S4   | 0.0317 (3) | 0.0659 (5) | 0.0360 (3) | −0.0125 (3) | −0.0068 (3) | −0.0177 (3) |
| Zn1  | 0.0220 (2) | 0.0361 (3) | 0.0293 (3) | −0.0022 (3) | −0.0033 (2) | −0.0137 (2) |
| N1   | 0.0293 (10)| 0.0340 (11)| 0.0308 (11)| −0.0014 (8) | −0.0038 (8) | −0.0063 (9) |
| C1   | 0.0227 (11)| 0.0336 (13)| 0.0389 (14)| −0.0004 (9) | −0.0004 (10)| −0.0059 (11)|
| C2   | 0.0226 (11)| 0.0369 (13)| 0.0439 (14)| −0.0024 (10)| −0.0098 (10)| −0.0091 (11)|
| N2   | 0.0276 (10)| 0.0355 (11)| 0.0341 (11)| 0.0024 (8)  | −0.0095 (8) | −0.0083 (9) |
| C3   | 0.0419 (14)| 0.0341 (13)| 0.0390 (14)| −0.0048 (11)| −0.0140 (11)| −0.0146 (11)|
| C4   | 0.0517 (16)| 0.0396 (15)| 0.0423 (15)| −0.0002 (12)| −0.0094 (13)| −0.0219 (13)|
| C5   | 0.0395 (14)| 0.0383 (14)| 0.0327 (13)| 0.0030 (11)| −0.0001 (11)| −0.0148 (11)|
| Zn2  | 0.0277 (4) | 0.0299 (3) | 0.0374 (4) | 0.0002 (3)  | −0.0105 (3) | −0.0125 (4) |
| N11  | 0.0339 (11)| 0.0384 (12)| 0.0301 (11)| 0.0003 (9)  | −0.0026 (9) | −0.0106 (9) |
| C11  | 0.0305 (13)| 0.0547 (17)| 0.0378 (14)| 0.0074 (12) | −0.0101 (11)| −0.0097 (13)|
| C12  | 0.0294 (13)| 0.0606 (18)| 0.0354 (14)| −0.0069 (12)| −0.0106 (11)| −0.0127 (13)|
| N12  | 0.0360 (11)| 0.0434 (13)| 0.0283 (10)| −0.0075 (9) | −0.0054 (9) | −0.0108 (9) |
| C13  | 0.0527 (17)| 0.0447 (16)| 0.0352 (14)| −0.0171 (13)| −0.0045 (12)| −0.0155 (12)|
| C14  | 0.0658 (19)| 0.0319 (13)| 0.0358 (14)| −0.0117 (13)| −0.0005 (13)| −0.0110 (11)|
| C15  | 0.0533 (17)| 0.0306 (13)| 0.0364 (14)| 0.0036 (12) | −0.0011 (12)| −0.0097 (11)|
| Zn3  | 0.0258 (3) | 0.0273 (3) | 0.0292 (3) | −0.0013 (2) | −0.0054 (2) | −0.0074 (2) |
| N21  | 0.0390 (12)| 0.0287 (11)| 0.0401 (12)| 0.0007 (9)  | −0.0036 (10)| −0.0128 (9) |
| C21  | 0.0372 (14)| 0.0399 (15)| 0.0529 (17)| −0.0132 (12)| 0.0103 (12)| −0.0199 (13)|
| C22  | 0.0271 (13)| 0.0532 (17)| 0.0628 (19)| −0.0058 (12)| −0.0031 (12)| −0.0319 (15)|
| N22  | 0.0302 (11)| 0.0382 (12)| 0.0505 (14)| 0.0054 (9)  | −0.0093 (10)| −0.0198 (11)|
| C23  | 0.0439 (16)| 0.0516 (17)| 0.0567 (18)| 0.0151 (13) | −0.0262 (14)| −0.0276 (15)|
| C24  | 0.071 (2)  | 0.0530 (18)| 0.0367 (15)| 0.0161 (16) | −0.0234 (15)| −0.0185 (14)|
| C25  | 0.0580 (18)| 0.0358 (14)| 0.0307 (13)| 0.0060 (13) | −0.0015 (12)| −0.0062 (11)|
| O1   | 0.0433 (11)| 0.0527 (13)| 0.0576 (13)| −0.0048 (9) | −0.0073 (10)| −0.0303 (11)|
| N31  | 0.222 (18)| 0.123 (11)| 0.059 (5)  | −0.092 (12) | 0.002 (8)  | −0.029 (7)  |
| C31  | 0.197 (16)| 0.087 (12)| 0.067 (6)  | −0.097 (12) | 0.008 (9)  | −0.036 (6)  |
| C32  | 0.24 (3)  | 0.102 (16)| 0.060 (6)  | −0.043 (14) | −0.024 (11)| −0.030 (7)  |
| N31′ | 0.25 (2)  | 0.146 (15)| 0.112 (13) | −0.031 (14) | 0.020 (12) | −0.070 (12) |
| C31′ | 0.183 (16)| 0.110 (19)| 0.112 (12) | −0.049 (15) | 0.007 (11) | −0.067 (12) |
| C32′ | 0.18 (2)  | 0.119 (19)| 0.109 (13) | −0.085 (17) | 0.025 (13) | −0.071 (13) |

**Geometric parameters (Å, †)**

|      |          |          |          |          |          |          |
|------|----------|----------|----------|----------|----------|----------|
| Sb1—S4 | 2.3049 (7) |          |          |          |          |          |
| Sb1—S2 | 2.3214 (7) |          |          |          |          |          |
| Sb1—S3 | 2.3252 (6) |          |          |          |          |          |
| Sb1—S1 | 2.3358 (6) |          |          |          |          |          |
| S1—Zn1 | 2.4071 (8) |          |          |          |          |          |
| S2—Zn2 | 2.4614 (9) |          |          |          |          |          |
| S3—Zn3 | 2.4300 (8) |          |          |          |          |          |
| Zn1—N1 | 2.028 (2)  |          |          |          |          |          |
| Zn1—N2 | 2.039 (2)  |          |          |          |          |          |
| Zn1—N2 | 2.196 (2)  |          |          |          |          |          |
| Zn2—N2 | 2.200 (2)  |          |          |          |          |          |
| N1—C1  | 1.468 (3)  |          |          |          |          |          |
N1—C5$^i$ 1.471 (4) Zn3—N21$^{iii}$ 2.207 (2)
N1—H1 1.0000 N21—C21 1.466 (4)
C1—C2 1.521 (4) N21—C25$^{iii}$ 1.471 (4)
C1—H1A 0.9900 N21—H21 1.0000
C1—H1B 0.9900 C21—C22 1.515 (5)
C2—N2 1.474 (3) C21—H21A 0.9900
C2—H2A 0.9900 C21—H21B 0.9900
C2—H2B 0.9900 C22—N22 1.471 (4)
N2—C3 1.461 (4) C22—H22A 0.9900
N2—H2 1.0000 C22—H22B 0.9900
C3—C4 1.525 (4) N22—C23 1.462 (4)
C3—H3A 0.9900 C31—C32 1.41 (2)
C3—H3B 0.9900 C32—H32A 0.9800
C4—C5 1.524 (4) C32—H32B 0.9800
C4—H4A 0.9900 C32—H32C 0.9800
C4—H4B 0.9900 C32—H32D 0.9800
C5—H5A 0.9900 C32—H32E 0.9800
C5—H5B 0.9900 C32—H32F 0.9800
Zn2—N12 2.048 (2) C13—N12—Zn2 120.73 (18)
Zn2—N11$^i$ 2.052 (2) C12—N12—Zn2 109.48 (18)
Zn2—N12$^{ii}$ 2.172 (2) C12—N12—H12 103.4
Zn2—N11 2.179 (2) N12—C13—C14 112.6 (2)
N11—C11 1.470 (4) N12—C13—H13A 109.1
N11—C15$^i$ 1.471 (4) C14—C13—H13A 109.1
N11—H11 1.0000 C13—C14—H14A 108.2
C11—C12 1.513 (5) C13—C14—H14B 108.2
C11—H11A 0.9900 N12—C13—H13B 109.1
C11—H11B 0.9900 N1—C15—N12 109.1
N12—C12—C13 1.476 (3) C14—C13—H13B 107.8
C12—N12—Zn1 96.40 (9) C13—C14—H13B 107.8
N1—Zn1—N2$^i$ 115.03 (3) C14—C13—H13B 107.8
N1—Zn1—N2 110.73 (2) C13—C14—H13B 107.8
N2$^i$—Zn1—N2 111.23 (3) C13—C14—H13B 107.8
N1—Zn1—N1$^i$ 110.73 (2) C13—C14—H13B 107.8
N2$^i$—Zn1—N1$^i$ 110.73 (2) C13—C14—H13B 107.8
N2—Zn1—N1$^i$ 110.73 (2) C13—C14—H13B 107.8
S4—Sb1—S2 119.36 (3) N12—C13—C14 112.6 (2)
S4—Sb1—S3 111.23 (3) C13—N12—Zn2 120.73 (18)
S2—Sb1—S3 110.64 (3) C12—N12—Zn2 109.1
S4—Sb1—S1 109.55 (2) N12—C13—N12 109.1
S2—Sb1—S1 110.37 (3) C13—N12—H12 103.4
S3—Sb1—S1 104.65 (2) C13—N12—H12 103.4
Sb1—S1—Zn1 108.53 (3) C14—C13—H13A 109.1
Sb1—S2—Zn2 115.03 (3) C14—C13—H13A 109.1
Sb1—S3—Zn3 115.03 (3) C14—C13—H13A 109.1
N1—Zn1—N2$^i$ 96.40 (9) C14—C13—H13A 109.1
N1—Zn1—N2 83.55 (9) H13A—C13—H13B 107.8
N2$^i$—Zn1—N2 83.55 (9) H13A—C13—H13B 107.8
N1—Zn1—N1$^i$ 115.90 (4) C13—C14—C15 116.4 (2)
N2$^i$—Zn1—N1$^i$ 115.90 (4) C13—C14—H14A 108.2
N2—Zn1—N1$^i$ 115.90 (4) C13—C14—H14B 108.2

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| Bond | Angle | Symmetry | Notes  |
|------|-------|----------|--------|
| N1—Zn1—S1 | 106.20 (7) |  |  |
| N2—Zn1—S1 | 99.42 (7) |  |  |
| N2—Zn1—S1 | 103.76 (6) | N1ii—C15—C14 | 112.9 (2) |
| N1—Zn1—S1 | 97.60 (6) | N1i—C15—H15A | 109.0 |
| C1—N1—C5i | 113.9 (2) | C14—C15—H15A | 109.0 |
| C1—N1—Zn1 | 111.15 (17) | N1i—C15—H15B | 109.0 |
| C5i—N1—Zn1 | 122.20 (17) | C14—C15—H15B | 109.0 |
| C1—N1—H1 | 102.0 | H15A—C15—H15B | 107.8 |
| C5i—N1—H1 | 102.0 | N21—Zn3—N22iii | 95.73 (10) |
| Zn1—N1—H1 | 102.0 | N21—Zn3—N22 | 82.91 (10) |
| N1—C1—C2 | 109.7 (2) | N22iii—Zn3—N22 | 152.15 (4) |
| N1—C1—H1A | 109.7 | N21—Zn3—N21ii | 152.13 (4) |
| C2—C1—H1A | 109.7 | N22iii—Zn3—N21iii | 82.31 (10) |
| N1—C1—H1B | 109.7 | N22—Zn3—N21iii | 86.13 (9) |
| C2—C1—H1B | 109.7 | N22—Zn3—S3 | 109.80 (7) |
| H1A—C1—H1B | 109.7 | N22iii—Zn3—S3 | 103.39 (7) |
| N2—C2—C1 | 110.8 (2) | N22—Zn3—S3 | 103.20 (6) |
| N2—C2—H2A | 110.8 | N21iii—Zn3—S3 | 97.61 (6) |
| C1—C2—H2A | 109.7 | C21—N21—C25iii | 114.4 (2) |
| N2—C2—H2B | 109.7 | C21—N21—Zn3 | 111.54 (18) |
| C1—C2—H2B | 109.7 | C25iii—N21—Zn3 | 122.87 (19) |
| H2A—C2—H2B | 108.2 | C21—N21—H21 | 101.2 |
| C3—N2—C2 | 113.9 (2) | C25iii—N21—H21 | 101.2 |
| C3—N2—Zn1 | 109.35 (17) | Zn3—N21—H21 | 101.2 |
| C2—N2—Zn1 | 100.71 (17) | N21—C21—C22 | 109.0 (2) |
| C3—N2—H2 | 110.8 | N21—C21—H21A | 109.9 |
| C2—N2—H2 | 110.8 | C22—C21—H21A | 109.9 |
| Zn1—N2—H2 | 110.8 | N21—C21—H21B | 109.9 |
| N2—C3—C4 | 111.4 (2) | C22—C21—H21B | 109.9 |
| N2—C3—H3A | 109.3 | H21A—C21—H21B | 108.3 |
| C4—C3—H3A | 109.3 | N22—C22—C21 | 108.9 (2) |
| N2—C3—H3B | 109.3 | N22—C22—H22A | 109.9 |
| C4—C3—H3B | 109.3 | C21—C22—H22A | 109.9 |
| H3A—C3—H3B | 108.0 | N22—C22—H22B | 109.9 |
| C5—C4—C3 | 115.7 (2) | C21—C22—H22B | 109.9 |
| C5—C4—H4A | 108.3 | H22A—C22—H22B | 108.3 |
| C3—C4—H4A | 110.8 | C23—N22—C22 | 113.9 (2) |
| C5—C4—H4B | 110.8 | C23—N22—Zn3 | 111.25 (19) |
| C3—C4—H4B | 108.3 | C22—N22—Zn3 | 99.07 (18) |
| C4—C5—H5A | 110.1 | Zn3—N22—H22 | 110.7 |
| N1—C5—C4 | 112.4 (2) | C22—N22—H22 | 110.7 |
| N1—C5—H5A | 109.1 | N22—C23—C24 | 111.6 (2) |
| C4—C5—H5B | 109.1 | N22—C23—H23A | 109.3 |
| C4—C5—H5B | 109.1 | C24—C23—H23A | 109.3 |
| H5A—C5—H5B | 107.9 | N22—C23—H23B | 109.3 |
| N12—Zn2—N11ii | 95.95 (10) | C24—C23—H23B | 109.3 |
| N12—Zn2—N12iii | 159.15 (4) | H23A—C23—H23B | 108.0 |
N11ii—Zn2—N12ii 83.98 (10) C25—C24—C23 116.2 (3)
N12—Zn2—N11 83.92 (9) C25—C24—H24A 108.2
N11ii—Zn2—N11 159.22 (4) C23—C24—H24A 108.2
N12ii—Zn2—N11 88.86 (9) C23—C24—H24B 108.2
N11ii—Zn2—S2 103.12 (7) H24A—C24—H24B 107.4
N12ii—Zn2—S2 97.41 (7) N21iii—C25—C24 111.9 (2)
N11—Zn2—S2 97.13 (7) N21iii—C25—H25A 109.2
N12—Zn2—S2 102.88 (7) N21iii—C25—H25B 109.2
N11—Zn2—S2 103.67 (7) H24A—C24—H25A 107.9
N12—Zn2—S2 102.88 (7) H24A—C24—H25B 109.2
N11—Zn2—S2 102.88 (7) H24A—C24—H25B 109.2
C11—N11—C15ii 114.2 (2) C24—C25—H25A 109.2
C11—N11—Zn2 100.67 (17) N21iii—C25—H25B 109.2
C15ii—N11—Zn2 111.02 (17) C24—C25—H25B 107.9
C11—N11—H11 110.2 N21iii—C25—H25B 109.2
C15ii—N11—H11 110.2 N21iii—C25—H25B 109.2
C11—N11—H11 112.2 Zn2—N11—H11 174 (4)
C15ii—N11—H11 112.2 N21iii—C25—C24 111.9 (2)
N11—C11—C12 109.5 (2) C31—C32—C32A 109.5
N11—C11—H11A 109.8 C31—C32—C32B 109.5
C12—C11—H11A 109.8 H32A—C32—C32B 109.5
N11—C11—H11B 109.8 C31—C32—C32C 109.5
C12—C11—H11B 109.8 H32A—C32—C32C 109.5
H11A—C11—H11B 109.8 H32A—C32—C32C 109.5
N12—C12—C11 109.4 (2) N21iii—C31—C32 171 (3)
N12—C12—H12A 109.8 C31′—C32′—H32D 109.5
C11—C12—H12A 109.8 C31′—C32′—H32E 109.5
N12—C12—H12B 109.8 H32D—C32′—H32E 109.5
C11—C12—H12B 109.8 C31′—C32′—H32F 109.5
H12A—C12—H12B 109.8 H32D—C32′—H32F 109.5
C13—N12—C12 114.1 (2) H32E—C32′—H32F 109.5

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

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N1—H1···S3 | 1.00 | 2.39 | 3.380 (2) | 172 |
| N2—H2···S1i | 1.00 | 2.78 | 3.400 (2) | 121 |
| N2—H2···O1ii | 1.00 | 2.26 | 3.133 (3) | 146 |
| C3—H3···S1 | 0.99 | 2.93 | 3.626 (3) | 128 |
| C5—H5···S1 | 0.99 | 2.95 | 3.590 (3) | 123 |
| N11—H1···S1ii | 1.00 | 2.49 | 3.433 (2) | 157 |
| N11—H1···S2ii | 1.00 | 2.97 | 3.545 (2) | 117 |
| C11—H11···S4iv | 0.99 | 2.96 | 3.872 (3) | 154 |
| N12—H12···S4 | 1.00 | 2.50 | 3.475 (2) | 166 |
| C13—H13···S2ii | 0.99 | 2.81 | 3.490 (3) | 126 |
| C15—H15···S2ii | 0.99 | 2.82 | 3.495 (3) | 126 |
| N21—H21···S2 | 1.00 | 2.29 | 3.287 (2) | 172 |
| N22—H22···S3ii | 1.00 | 2.86 | 3.518 (2) | 124 |
| N22—H22···O1 | 1.00 | 2.18 | 2.940 (3) | 131 |
| C23—H23···S3 | 0.99 | 3.01 | 3.670 (3) | 125 |
|             | d | e | f | g |
|-------------|---|---|---|---|
| C25—H25A···S3 | 0.99 | 2.86 | 3.547 (3) | 127 |
| O1—H1O···S1\textsuperscript{vi} | 0.84 | 2.52 | 3.286 (2) | 152 |
| O1—H2O···S4\textsuperscript{iii} | 0.84 | 2.47 | 3.305 (2) | 173 |
| C32—H32A···S4 | 0.98 | 2.96 | 3.92 (3) | 170 |
| C32’—H32D···S4 | 0.98 | 2.89 | 3.66 (3) | 136 |

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