# research communications

# Synthesis and crystal structure of poly[[di-$\mu_3$-tetrathioantimonato-tris[(cyclam)cobalt(II)]] acetonitrile disolvate dihydrate] (cyclam = 1,4,8,11-tetraazacyclotetradecane)

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Reaction of Co(ClO$_4$)$_2$·6H$_2$O with cyclam (cyclam = 1,4,8,11-tetraazacyclotetradecane) and Na$_3$SbS$_4$·9H$_2$O (Schlippesches salt) in a mixture of acetonitrile and water leads to the formation of crystals of the title compound with the composition $\{[Co_3(SbS_4)_2(C_{10}H_{24}N_4)_3]·2CH_3CN·2H_2O\}_n$ or $\{[(Co\text{-}cyclam)_3(SbS_4)_2]·2(acetonitrile)·2H_2O\}_n$. The crystal structure of the title compound consists of three crystallographically independent [Co-cyclam]$^{2+}$ cations, which are located on centers of inversion, one [SbS$_4$]$^{3-}$ anion, one water and one acetonitrile molecule that occupy general positions. The acetonitrile molecule is disordered over two orientations and was refined using a split model. The Co$^{II}$ cations are coordinated by four N atoms of the cyclam ligand and two trans-S atoms of the tetrathioantimonate anion within slightly distorted octahedra. The unique [SbS$_4$]$^{3-}$ anion is coordinated to all three crystallographically independent Co$^{II}$ cations and this unit, with its symmetry-related counterparts, forms rings composed of six Co-cyclam cations and six tetrathioantimonate anions that are further condensed into layers. These layers are perfectly stacked onto each other so that channels are formed in which acetontrile solvate molecules that are hydrogen bonded to the anions are embedded. The water solvate molecules are located between the layers and are connected to the cyclam ligands and the [SbS$_4$]$^{3-}$ anions via intermolecular N—H···O and O—H···S hydrogen bonding.
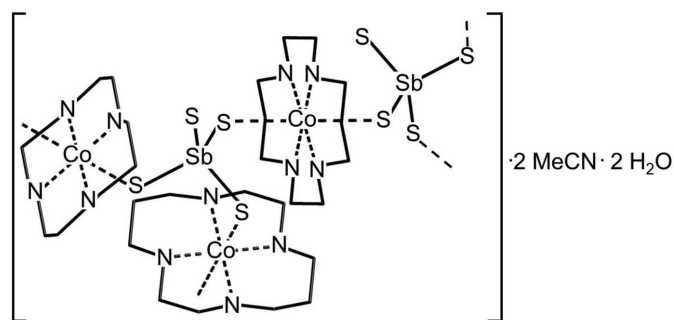
## 1. Chemical context

Inorganic–organic chalcogenidometallates are an important class of compounds and many such compounds have been reported in the literature (Sheldrick & Wachhold, 1988; Bensch et al., 1997; Dehnen & Melullis, 2007; Wang et al., 2016; Zhou, 2016; Zhu & Dai, 2017; Nie et al., 2017). A large part of this family of compounds consists of thioantimonates, which exhibit a variety of coordination numbers that can lead to networks of different dimensionality (Jia et al., 2004; Powell et al., 2005; Spetzler et al., 2004; Zhang et al., 2007; Liu & Zhou, 2011; Engelke et al., 2004; Puls et al., 2006). Moreover, some of them have potential for applications, for example in the field of superionic conductors (Zhou et al., 2019) or as photoconductive materials (Pienack et al., 2008a). For these reasons, we have explored such compounds over many years (Schaefer et al., 2003; Stähler et al., 2001; Schur et al., 1998, 2001; Kiebach et al., 2004; Spetzler et al., 2004; Lühmann et al., 2008; Pienack et al., 2008b). In the beginning, we synthesized new thioantimonates using elemental antimony, sulfur and amine molecules under solvothermal conditions but later we found that
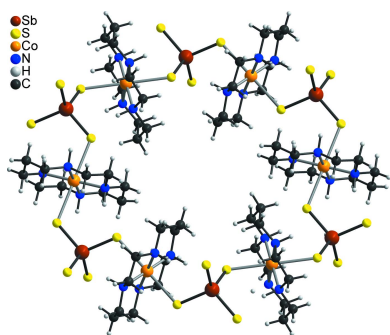
many of these compounds are also available under solvo-thermal conditions if Schlippesches salt ($Na_3SbS_4·9H_2O$) or $NaSbS_3$ are used as reactants (Anderer *et al.*, 2014, 2016; Danker *et al.*, 2020). In this case, different $SbS_x$ species are present in solution, because Schlippesches salt is unstable and forms different reactive species such as $[SbS_3O]^{3-}$, $HS^-$, $[S_2O_3]^{2-}$ or $[SbS_4]^{3-}$ anions (Rammelsberg, 1841; Long & Bowen, 1970; Mosselmanns *et al.*, 2000; Planer-Friedrich & Scheinost, 2011; Planer-Friedrich & Wilson, 2012; Anderer *et al.*, 2014). In addition, a variety of complex redox and condensation reactions occur, generating polymeric thioanti-monate(III) anions, which are found in the structures of the reaction products. To prevent the reduction of $Sb^V$ to $Sb^{III}$, a different synthesis strategy is required and the reaction temperature must be reduced to slow down the decomposition of Schlippesches salt. Using an aqueous solution of $Na_3SbS_4·9H_2O$ and adding a solution of late transition-metal cations leads to immediate precipitation of sulfides or hydro-xides, even when chelating amine molecules are added. To solve the problem we developed a two-solution strategy: an organic solution contains the transition-metal cations and the chelating amine molecule and a second solution comprises Schlippesches salt. In the organic solution, the transition-metal complex is already generated *in situ* and mixing the two solutions leads to nucleation and successive growth of the product. A challenge is the integration of transition-metal cations into a thioantimonate(V) network, despite the $[SbS_4]^{3-}$ anion offering four possible binding sites. In the course of this project we became interested in cyclam (cyclam = 1,4,8,11-tetraazacyclotetradecane), which is a tetradentate ligand. This means that in an octahedral coordination of a transition-metal cation, two coordination sites are provided to which thioantimonate(V) anions can coordinate, which, depending on the nature of the anion, can lead to the formation of the desired thioantimonate(V) networks.

are stacked in such a way that large pores are formed. Between the layers, water molecules are embedded. At first glance, the arrangement of the building blocks in the crystal structure of the Zn compound looks similar, but in this case the $Zn^{II}$ cation is disordered above and below the $N_4$ plane in a 1:1 ratio, which means that it is in a fivefold coordination of the four N atoms of the cyclam ligand and one S atom of the $[SbS_4]^{3-}$ anions in a square-pyramidal geometry. The structural difference between the Cu and Zn coordinations was reproduced by DFT calculations (Danker *et al.*, 2021). In the course of our systematic work we tried to prepare a similar compound with cobalt using the same synthetic approach. This led to crystals of the title compound, which were characterized by single-crystal X-ray diffraction.

## 2. Structural commentary

The asymmetric unit of the title compound consists of three crystallographically independent $Co^{II}$ cations and three independent cyclam ligands that are located on centers of inversion, as well as one $[SbS_4]^{3-}$ anion, one water and one acetonitrile molecule that occupy general positions (Fig. 1). The acetonitrile molecule is disordered over two orientations and was refined using a split model (see *Refinement*). The $Co^{II}$ cations are six-coordinate being bound to the four N atoms of cyclam ligand that are located in the equatorial plane and two *trans*-S atoms of two inversion-related tetrathioantimonate anions that occupy the apical positions (Fig. 2). The $Co-N$ bond lengths are very similar for the three crystallographically independent $Co^{II}$ cations whereas significant differences are found for the $Co-S$ bond lengths (Table 1). These changes, however, do not correlate with the $Sb-S$ distances (Table 1). The angles around the Co centers prove that the octahedra are slightly distorted (see supporting information). The cyclam ligands are in the *trans*-III($S,S,R,R$) configuration, which is the



In this context, we have reported on two new polymeric thioantimonates with the composition $[(Cu-cyclam)_3(SbS_4)_2]·20H_2O$ and $[(Zn-cyclam)_3(SbS_4)_2]·8H_2O$ (Danker *et al.*, 2021). In the crystal structure of the Cu compound, the copper cations are sixfold coordinated by the four N atoms of the cyclam ligand and two *trans*-sulfur atoms of the $[SbS_4]^{3-}$ anions within slightly distorted octahedra. The copper cations are linked by the anions into rings by corner-sharing $SbS_4$ and $CuN_4S_2$ units, which are condensed into layers. These layers

**Figure 1**
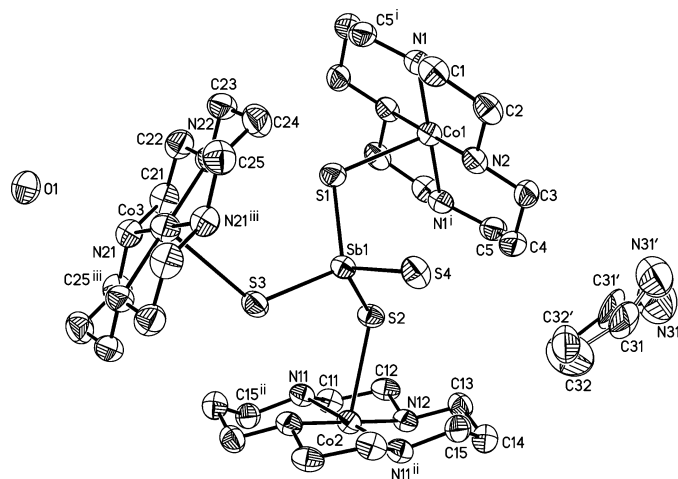Crystal structure of the title compound with labeling and displacement ellipsoids drawn at the 50% probability level. The hydrogen atoms are omitted for clarity and the disordering of the acetonitrile ligands is shown with full and open bonds. Symmetry codes for the generation of equivalent atoms: (i) $-x + 1$, $-y$, $-z + 2$; (ii) $-x + 1$, $-y + 1$, $-z + 1$; (iii) $-x + 2$, $-y$, $-z + 1$.

**Table 1**
Selected bond lengths (Å).

| Sb1—S4 | 2.3195 (13) | S1—Co1 | 2.7258 (12) |
|---|---|---|---|
| Sb1—S1 | 2.3200 (12) | S2—Co2 | 2.6932 (11) |
| Sb1—S3 | 2.3221 (12) | S3—Co3 | 2.7821 (12) |
| Sb1—S2 | 2.3382 (11) | | |

most stable arrangement for the first row transition-metal cation-centered cyclam complexes (Bosnich *et al.*, 1965).

The Sb—S bond lengths in the tetrathioantimonate anion (Table 1) are comparable and correspond to those observed in other compounds with this anion. From the S—Sb—S bond angles it is obvious that the tetrahedron is only slightly distorted (see supporting information). The $[SbS_4]^{3-}$ anion shows the rare tridentate coordination mode and is linked to each of the three crystallographically independent $Co^{II}$ cations and with inversion-related counterparts, forming rings



**Figure 3**
Crystal structure of the title compound with a view of an 24-membered ring composed of six Co cations and six $[SbS_4]^{3-}$ anions.

composed of six $[SbS_4]^{3-}$ anions and six $[Co(cyclam]^{2+}$ cations (Fig. 3). These rings are condensed into layers parallel to the *bc* plane (Fig. 4). This layer topology is identical to that in
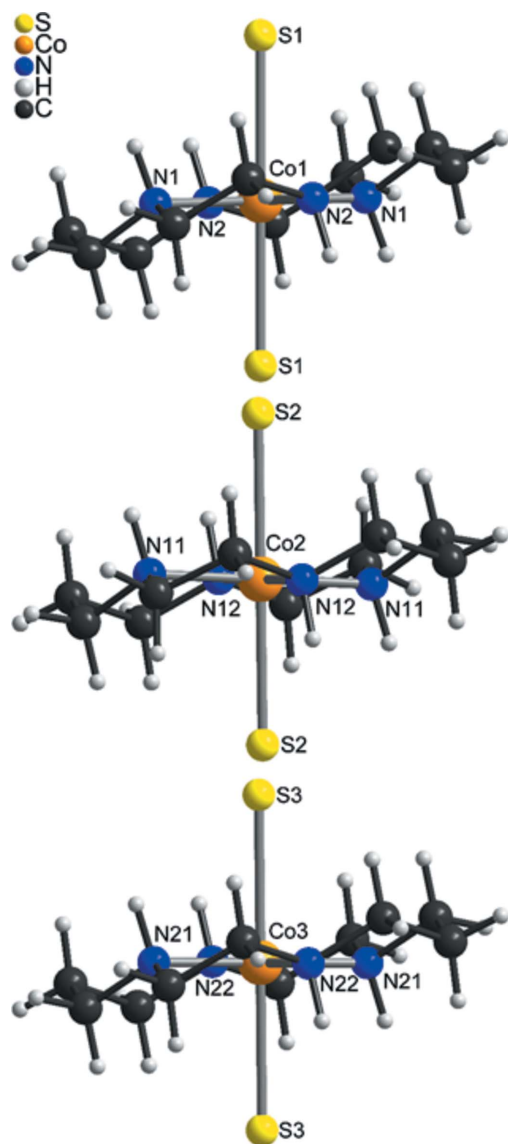


**Figure 2**
Crystal structure of the title compound with a view of the coordination sphere of the three crystallographically independent Co cations.
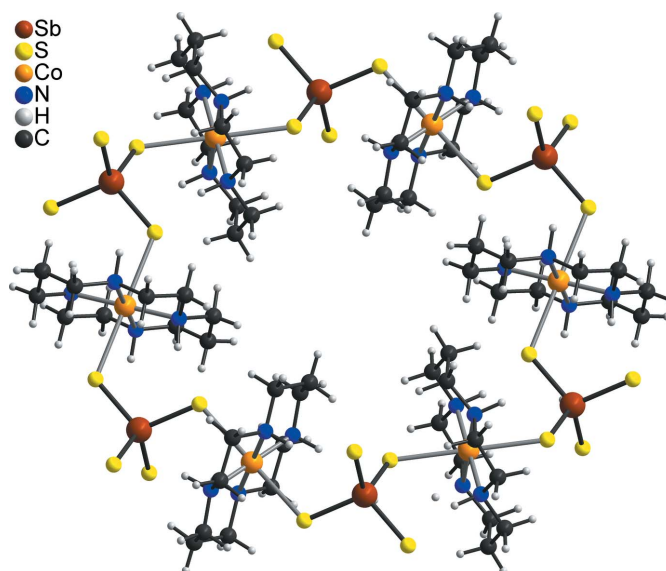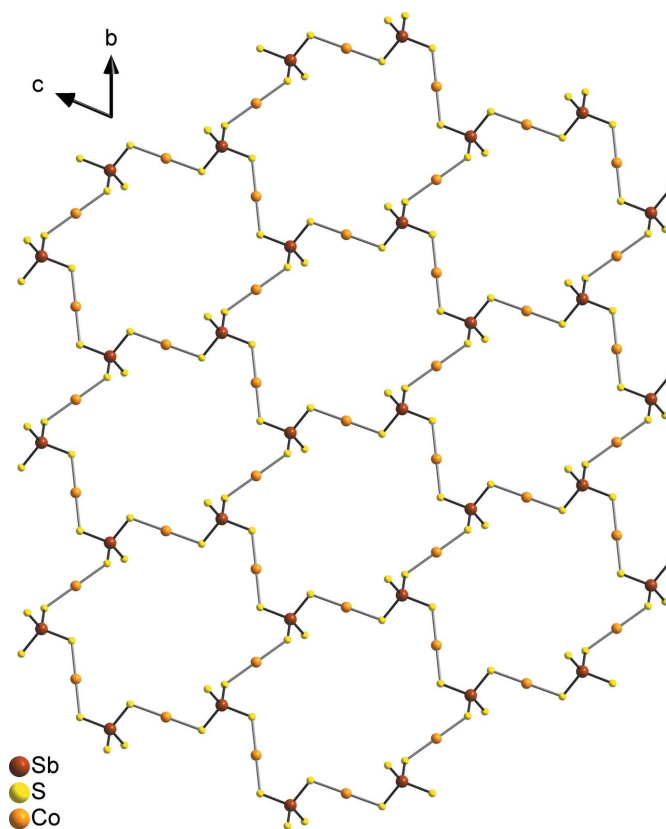


**Figure 4**
Crystal structure of the title compound with a view of the $Co_3(SbS_4)_2$ network along the crystallographic *a* axis. The cyclam ligands are not shown for clarity.

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|---|---|---|---|
| N1—H1···S2[i] | 1.00 | 2.48 | 3.442 (4) | 161 |
| C1—H1B···S4[ii] | 0.99 | 2.95 | 3.858 (5) | 152 |
| N2—H2···S4 | 1.00 | 2.49 | 3.448 (4) | 159 |
| N11—H11···O1[iii] | 1.00 | 2.23 | 3.151 (6) | 153 |
| N12—H12···S3[iv] | 1.00 | 2.43 | 3.378 (4) | 157 |
| N21—H21···O1 | 1.00 | 2.08 | 2.920 (6) | 141 |
| N22—H22···S1 | 1.00 | 2.35 | 3.290 (4) | 156 |
| O1—H1C···S2[iii] | 0.84 | 2.49 | 3.276 (4) | 157 |
| O1—H1D···S4[v] | 0.84 | 2.46 | 3.280 (4) | 166 |
| C32—H32B···S4 | 0.98 | 2.81 | 3.71 (4) | 154 |
| C32′—H32F···S4 | 0.98 | 2.88 | 3.85 (5) | 172 |

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

$[Cu(cyclam)_3[SbS_4]_2]\cdot 20H_2O$ but the two compounds are not isotypic (Danker *et al.*, 2021). The layers are stacked perfectly onto each other, forming channels extending along the a-axis direction (Fig. 5).

## 3. Supramolecular features

Within the channels are embedded acetonitrile solvate molecules that are disordered and hydrogen bonded to the tetrathioantimonate anion (Fig. 5). The C—H···S angles are close to linear, indicating that this is a significant interaction (Table 2). Water molecules are located between the layers and are connected to the $[SbS_4]^{3-}$ anions *via* intermolecular O—H···S hydrogen bonding, which is classed as strong because the angles are close to linearity and relatively short H···S distances are observed (Table 2). These water molecules also act as acceptors for N—H···O hydrogen bonding to the cyclam ligands (Table 2). The layers are linked by additional C—H···S and N—H···S hydrogen bonds between the cyclam
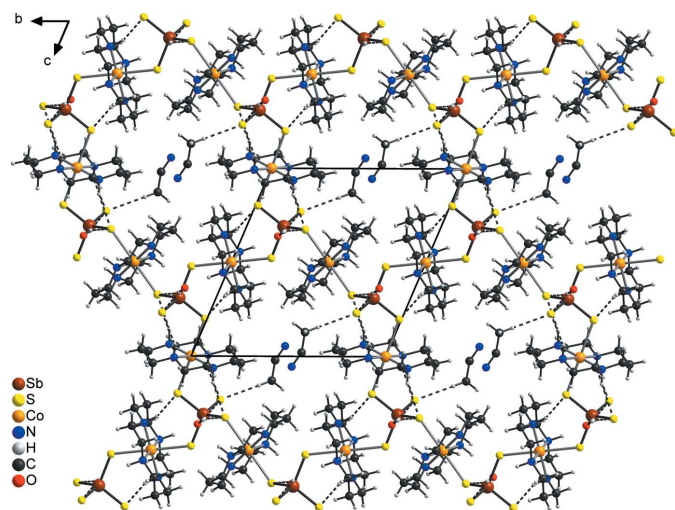


**Figure 5**
Crystal structure of the title compound with a view in the *a*-axis direction with intermolecular hydrogen bonding shown as dashed lines. The disorder of the acetonitrile molecules is omitted for clarity.

ligands and the tetrathioantimonate anions. There are additional H···S contacts but at distances close to van der Waals contacts with angles ranging between about 110 and 125°.

## 4. Database survey

A search for structures of cobalt-centered cylam complexes in the Cambridge Structural Database (CSD version 5.42, last update November 2020; Groom *et al.*, 2016) gave 152 hits, in four of which the cobalt cations are in an $N_4S_2$ coordination. In one of these structures (Refcode: NIMVIQ; Zeisler *et al.*, 2013), a thiostannate acts as anion but none of them contains thioantimonate anions. The same results are obtained if the search is expanded to any transition-metal cation. Therefore, only the Cu and Zn compounds mentioned above have been published (Danker *et al.*, 2021).

However, 21 structures with $Co^{II}$ and tetrathioantimonate anions have been published and in two of these structures, the cobalt cations are linked to a tetrathioantimonate anion, *viz.* $[Co(diethylenetriamine)_2][Co(tris(2-aminomethyl)amine)-SbS_4]_2\cdot 4H_2O$ (Engelke *et al.*, 2008) and $[Co(diethylenetriamine)_2][Co(tris(2-aminomethyl)amine)SbS_4]_2\cdot 0.5H_2O$ (Lichte, *et al.*, 2009).

## 5. Synthesis and crystallization

### Synthesis of $Na_3SbS_4\cdot 9H_2O$

$Na_3SbS_4\cdot 9H_2O$ was synthesized by adding 16.6 g (0.213 mol) of $Na_2S\cdot xH_2O$ (technical grade, purchased from Acros Organics) to 58 mL of demineralized water. This solution was heated to 323 K for 1 h. Afterwards, 19.6 g (0.058 mol) of $Sb_2S_3$ (98%, purchased from Alfa Aesar) and 3.69 g (0.115 mol) of sulfur (min. 99%, purchased from Alfa Aesar) were added and the reaction mixture was heated to 343 K for 6 h. The reaction mixture was filtered and the filtrate was stored overnight, leading to the formation of slightly yellow crystals, which were filtered off, washed with small amounts of water and dried under vacuum (yield about 30% based on $Sb_2S_3$).

### Synthesis of the title compound

16 mg (0.044 mmol) of $Co(ClO_4)_2\cdot 6H_2O$ (purchased from Alfa Aesar) and 16 mg (0.08 mmol) of cyclam (purchased from Strem Chemicals) were dissolved in 2 mL of acetonitrile (purchased from Merck). To this solution, a solution of 50 mg (0.14 mmol) of $Na_3SbS_4\cdot 9H_2O$ dissolved in 1 mL of $H_2O$ was added. Within 3d a few colorless crystals of the title compound were obtained, which were always contaminated with an additional and unknown phase that is amorphous to X-rays. This additional phase is also present if the reaction conditions are varied slightly. Therefore, one of the colorless crystals was selected for structure determination.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All non-hydrogen atoms were refined anisotropically. The C- and N-bound H atoms were

**Table 3**
Experimental details.

| Crystal data | |
|---|---|
| Chemical formula | $[Co_3(SbS_4)_2(C_{10}H_{24}N_4)_3]\cdot 2C_2H_3N\cdot 2H_2O$ |
| $M_r$ | 1395.90 |
| Crystal system, space group | Triclinic, $P\bar{1}$ |
| Temperature (K) | 200 |
| $a, b, c$ (Å) | 8.7292 (3), 12.9680 (5), 13.8936 (5) |
| $\alpha, \beta, \gamma$ (°) | 66.218 (3), 77.035 (3), 83.321 (3) |
| $V$ (Å$^3$) | 1401.93 (9) |
| $Z$ | 1 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 2.16 |
| Crystal size (mm) | $0.15 \times 0.10 \times 0.07$ |
| | |
| Data collection | |
| Diffractometer | Stoe IPDS2 |
| Absorption correction | Numerical (*X-RED* and *X-SHAPE*; Stoe, 2008) |
| $T_{min}, T_{max}$ | 0.649, 0.774 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 15304, 6098, 5078 |
| $R_{int}$ | 0.030 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.639 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.047, 0.126, 1.06 |
| No. of reflections | 6098 |
| No. of parameters | 321 |
| No. of restraints | 87 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}, \Delta\rho_{min}$ (e Å$^{-3}$) | 0.81, −1.56 |

Computer programs: *X-AREA* (Stoe, 2008), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg, 1999) and *publCIF* (Westrip, 2010).

located in the difference map but were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip) and were refined isotropically with $U_{iso}(H) = 1.2U_{eq}(C,N)$ (1.5 for methyl H atoms) using a riding model. The O-bound H atoms were located in the difference map, their bond lengths were set to ideal values and finally they were refined isotropically with $U_{iso}(H) = 1.5U_{eq}(O)$ using a riding model. The acetontrile molecule is disordered over two orientations and was refined using a split model (ratio: 1:1) with restraints for the geometry and the components of the anisotropic displacement parameters.

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# supporting information

## Synthesis and crystal structure of poly[[di-$\mu_3$-tetrathioantimonato-tris-[(cyclam)cobalt(II)]] acetonitrile disolvate dihydrate] (cyclam = 1,4,8,11-tetra-azacyclotetradecane)

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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: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2010).

**Poly[[di-$\mu_3$-tetrathioantimonato-tris-[(cyclam)cobalt(II)]] acetonitrile disolvate dihydrate]**

*Crystal data*

| | |
|---|---|
| [Co$_3$(SbS$_4$)$_2$(C$_{10}$H$_{24}$N$_4$)$_3$]·2C$_2$H$_3$N·2H$_2$O | $Z = 1$ |
| $M_r = 1395.90$ | $F(000) = 711$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.653$ Mg m$^{-3}$ |
| $a = 8.7292$ (3) Å | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $b = 12.9680$ (5) Å | Cell parameters from 15304 reflections |
| $c = 13.8936$ (5) Å | $\theta = 1.6$–27.0° |
| $\alpha = 66.218$ (3)° | $\mu = 2.16$ mm$^{-1}$ |
| $\beta = 77.035$ (3)° | $T = 200$ K |
| $\gamma = 83.321$ (3)° | Block, red |
| $V = 1401.93$ (9) Å$^3$ | 0.15 × 0.10 × 0.07 mm |

*Data collection*

| | |
|---|---|
| Stoe IPDS-2 diffractometer | 6098 independent reflections |
| $\omega$ scans | 5078 reflections with $I > 2\sigma(I)$ |
| Absorption correction: numerical (X-Red and X-Shape; Stoe, 2008) | $R_{int} = 0.030$ |
| | $\theta_{max} = 27.0°$, $\theta_{min} = 1.6°$ |
| $T_{min} = 0.649$, $T_{max} = 0.774$ | $h = -11{\rightarrow}11$ |
| 15304 measured reflections | $k = -16{\rightarrow}16$ |
| | $l = -17{\rightarrow}17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: mixed |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | $w = 1/[\sigma^2(F_o^2) + (0.084P)^2 + 0.1597P]$ |
| $wR(F^2) = 0.126$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{max} = 0.047$ |
| 6098 reflections | $\Delta\rho_{max} = 0.81$ e Å$^{-3}$ |
| 321 parameters | $\Delta\rho_{min} = -1.56$ e Å$^{-3}$ |
| 87 restraints | |

Extinction correction: *SHELXL2016/6*
(Sheldrick 2015b),
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0136 (11)

*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* | $U_{iso}*/U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Sb1 | 0.73888 (3) | 0.19084 (2) | 0.69041 (2) | 0.03283 (12) | |
| S1 | 0.64905 (15) | 0.01167 (10) | 0.80137 (9) | 0.0414 (3) | |
| S2 | 0.51359 (13) | 0.30840 (9) | 0.67415 (9) | 0.0380 (3) | |
| S3 | 0.86596 (14) | 0.19636 (10) | 0.52265 (9) | 0.0416 (3) | |
| S4 | 0.90553 (14) | 0.24527 (12) | 0.76901 (10) | 0.0461 (3) | |
| Co1 | 0.500000 | 0.000000 | 1.000000 | 0.0350 (2) | |
| N1 | 0.6290 (4) | −0.1388 (3) | 1.0629 (3) | 0.0392 (8) | |
| H1 | 0.583701 | −0.171109 | 1.141506 | 0.047* | |
| C1 | 0.7871 (6) | −0.1025 (5) | 1.0528 (4) | 0.0461 (11) | |
| H1A | 0.847663 | −0.085872 | 0.979114 | 0.055* | |
| H1B | 0.843932 | −0.162998 | 1.103578 | 0.055* | |
| C2 | 0.7700 (6) | 0.0016 (5) | 1.0773 (4) | 0.0465 (11) | |
| H2A | 0.720658 | −0.016978 | 1.153611 | 0.056* | |
| H2B | 0.874555 | 0.032869 | 1.063722 | 0.056* | |
| N2 | 0.6701 (5) | 0.0849 (3) | 1.0071 (3) | 0.0391 (8) | |
| H2 | 0.736736 | 0.114228 | 0.933522 | 0.047* | |
| C3 | 0.6268 (7) | 0.1831 (4) | 1.0369 (4) | 0.0465 (11) | |
| H3A | 0.723689 | 0.218535 | 1.033103 | 0.056* | |
| H3B | 0.567730 | 0.157340 | 1.111845 | 0.056* | |
| C4 | 0.5275 (7) | 0.2697 (4) | 0.9642 (4) | 0.0502 (12) | |
| H4A | 0.584251 | 0.290889 | 0.889012 | 0.060* | |
| H4B | 0.515488 | 0.338203 | 0.980459 | 0.060* | |
| C5 | 0.3653 (6) | 0.2311 (4) | 0.9738 (4) | 0.0462 (11) | |
| H5A | 0.309991 | 0.205729 | 1.049576 | 0.055* | |
| H5B | 0.304013 | 0.295721 | 0.930767 | 0.055* | |
| Co2 | 0.500000 | 0.500000 | 0.500000 | 0.0342 (2) | |
| N11 | 0.3450 (4) | 0.4238 (3) | 0.4693 (3) | 0.0375 (8) | |
| H11 | 0.350102 | 0.342674 | 0.518595 | 0.045* | |
| C11 | 0.1850 (5) | 0.4666 (4) | 0.5023 (4) | 0.0422 (10) | |
| H11A | 0.105455 | 0.411439 | 0.515475 | 0.051* | |
| H11B | 0.160604 | 0.538662 | 0.445200 | 0.051* | |
| C12 | 0.1825 (5) | 0.4840 (4) | 0.6041 (4) | 0.0422 (10) | |
| H12A | 0.083455 | 0.523577 | 0.622556 | 0.051* | |
| H12B | 0.188964 | 0.410337 | 0.664261 | 0.051* | |
| N12 | 0.3180 (4) | 0.5518 (3) | 0.5857 (3) | 0.0370 (8) | |

| | | | | |
|---|---|---|---|---|
| H12 | 0.291146 | 0.630110 | 0.537533 | 0.044* |
| C13 | 0.3355 (6) | 0.5600 (4) | 0.6854 (4) | 0.0433 (10) |
| H13A | 0.348209 | 0.483093 | 0.739725 | 0.052* |
| H13B | 0.238416 | 0.594863 | 0.713526 | 0.052* |
| C14 | 0.4744 (7) | 0.6285 (5) | 0.6695 (5) | 0.0500 (12) |
| H14A | 0.471829 | 0.639111 | 0.736564 | 0.060* |
| H14B | 0.464296 | 0.703873 | 0.612383 | 0.060* |
| C15 | 0.6317 (6) | 0.5751 (4) | 0.6397 (4) | 0.0435 (10) |
| H15A | 0.717003 | 0.616669 | 0.643320 | 0.052* |
| H15B | 0.637578 | 0.496295 | 0.691914 | 0.052* |
| Co3 | 1.000000 | 0.000000 | 0.500000 | 0.0441 (2) |
| N21 | 0.8192 (5) | −0.0196 (3) | 0.4448 (3) | 0.0409 (9) |
| H21 | 0.851395 | −0.081969 | 0.418885 | 0.049* |
| C21 | 0.6910 (6) | −0.0636 (5) | 0.5384 (5) | 0.0480 (11) |
| H21A | 0.610940 | −0.097814 | 0.520863 | 0.058* |
| H21B | 0.639594 | −0.001829 | 0.559812 | 0.058* |
| C22 | 0.7616 (6) | −0.1511 (4) | 0.6284 (4) | 0.0484 (12) |
| H22A | 0.680551 | −0.178519 | 0.694862 | 0.058* |
| H22B | 0.805027 | −0.216022 | 0.609610 | 0.058* |
| N22 | 0.8886 (5) | −0.0960 (3) | 0.6442 (3) | 0.0401 (9) |
| H22 | 0.833782 | −0.042725 | 0.677357 | 0.048* |
| C23 | 0.9780 (7) | −0.1761 (4) | 0.7226 (4) | 0.0477 (12) |
| H23A | 1.027418 | −0.235259 | 0.696736 | 0.057* |
| H23B | 0.904885 | −0.213090 | 0.791464 | 0.057* |
| C24 | 1.1039 (7) | −0.1201 (5) | 0.7413 (4) | 0.0549 (13) |
| H24A | 1.148426 | −0.175116 | 0.802670 | 0.066* |
| H24B | 1.055165 | −0.056814 | 0.761253 | 0.066* |
| C25 | 1.2359 (6) | −0.0757 (5) | 0.6455 (4) | 0.0496 (12) |
| H25A | 1.320806 | −0.049907 | 0.666612 | 0.060* |
| H25B | 1.279912 | −0.137189 | 0.621601 | 0.060* |
| O1 | 0.7600 (5) | −0.1823 (3) | 0.3619 (3) | 0.0554 (9) |
| H1C | 0.704099 | −0.205901 | 0.333522 | 0.08 (2)* |
| H1D | 0.850839 | −0.202701 | 0.339072 | 0.10 (3)* |
| N31 | 0.748 (3) | 0.4588 (19) | 1.0545 (11) | 0.116 (7) | 0.5 |
| C31 | 0.815 (5) | 0.457 (4) | 0.9743 (13) | 0.101 (7) | 0.5 |
| C32 | 0.908 (4) | 0.469 (4) | 0.8707 (12) | 0.090 (7) | 0.5 |
| H32A | 0.899961 | 0.547550 | 0.820009 | 0.135* | 0.5 |
| H32B | 0.869880 | 0.419111 | 0.844728 | 0.135* | 0.5 |
| H32C | 1.018580 | 0.449588 | 0.876821 | 0.135* | 0.5 |
| N31′ | 0.814 (3) | 0.4114 (18) | 1.0734 (10) | 0.112 (7) | 0.5 |
| C31′ | 0.833 (5) | 0.452 (4) | 0.9817 (11) | 0.093 (7) | 0.5 |
| C32′ | 0.854 (5) | 0.488 (4) | 0.8670 (12) | 0.122 (12) | 0.5 |
| H32D | 0.943593 | 0.537807 | 0.831978 | 0.183* | 0.5 |
| H32E | 0.758528 | 0.529186 | 0.843259 | 0.183* | 0.5 |
| H32F | 0.873030 | 0.422251 | 0.847836 | 0.183* | 0.5 |

*Atomic displacement parameters (Å²)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sb1 | 0.03142 (17) | 0.03401 (17) | 0.02939 (17) | −0.00144 (10) | −0.00454 (10) | −0.00920 (11) |
| S1 | 0.0463 (6) | 0.0347 (5) | 0.0360 (6) | −0.0044 (4) | 0.0013 (5) | −0.0103 (5) |
| S2 | 0.0336 (5) | 0.0376 (5) | 0.0357 (6) | 0.0022 (4) | −0.0059 (4) | −0.0085 (4) |
| S3 | 0.0441 (6) | 0.0424 (6) | 0.0312 (5) | 0.0026 (5) | −0.0021 (4) | −0.0111 (5) |
| S4 | 0.0384 (6) | 0.0608 (7) | 0.0406 (6) | −0.0107 (5) | −0.0073 (5) | −0.0189 (6) |
| Co1 | 0.0331 (4) | 0.0364 (4) | 0.0342 (4) | −0.0022 (3) | −0.0069 (3) | −0.0119 (3) |
| N1 | 0.0383 (19) | 0.041 (2) | 0.0338 (19) | 0.0015 (16) | −0.0068 (15) | −0.0103 (16) |
| C1 | 0.036 (2) | 0.055 (3) | 0.044 (3) | 0.002 (2) | −0.0090 (19) | −0.016 (2) |
| C2 | 0.038 (2) | 0.061 (3) | 0.039 (3) | −0.008 (2) | −0.0099 (19) | −0.016 (2) |
| N2 | 0.040 (2) | 0.045 (2) | 0.0311 (18) | −0.0077 (16) | −0.0064 (15) | −0.0122 (16) |
| C3 | 0.060 (3) | 0.043 (3) | 0.041 (3) | −0.014 (2) | −0.007 (2) | −0.018 (2) |
| C4 | 0.065 (3) | 0.040 (2) | 0.040 (3) | −0.010 (2) | 0.000 (2) | −0.013 (2) |
| C5 | 0.053 (3) | 0.038 (2) | 0.038 (2) | 0.003 (2) | −0.001 (2) | −0.011 (2) |
| Co2 | 0.0295 (4) | 0.0378 (4) | 0.0331 (4) | −0.0022 (3) | −0.0049 (3) | −0.0118 (3) |
| N11 | 0.0363 (19) | 0.0381 (19) | 0.0348 (19) | −0.0025 (15) | −0.0086 (15) | −0.0095 (16) |
| C11 | 0.032 (2) | 0.047 (3) | 0.044 (3) | −0.0011 (18) | −0.0105 (19) | −0.013 (2) |
| C12 | 0.030 (2) | 0.042 (2) | 0.043 (3) | −0.0043 (17) | −0.0024 (18) | −0.006 (2) |
| N12 | 0.0352 (18) | 0.0370 (18) | 0.0337 (19) | 0.0011 (15) | −0.0061 (15) | −0.0093 (15) |
| C13 | 0.044 (2) | 0.048 (3) | 0.035 (2) | 0.003 (2) | −0.0039 (19) | −0.016 (2) |
| C14 | 0.057 (3) | 0.050 (3) | 0.046 (3) | −0.004 (2) | −0.008 (2) | −0.022 (2) |
| C15 | 0.049 (3) | 0.043 (2) | 0.043 (3) | −0.003 (2) | −0.015 (2) | −0.017 (2) |
| Co3 | 0.0412 (5) | 0.0448 (5) | 0.0438 (5) | −0.0033 (4) | −0.0067 (4) | −0.0149 (4) |
| N21 | 0.0363 (19) | 0.042 (2) | 0.046 (2) | 0.0010 (16) | −0.0086 (16) | −0.0195 (18) |
| C21 | 0.035 (2) | 0.055 (3) | 0.058 (3) | −0.004 (2) | −0.006 (2) | −0.027 (3) |
| C22 | 0.046 (3) | 0.044 (3) | 0.052 (3) | −0.009 (2) | 0.003 (2) | −0.020 (2) |
| N22 | 0.042 (2) | 0.0364 (19) | 0.037 (2) | −0.0024 (16) | −0.0023 (16) | −0.0117 (16) |
| C23 | 0.056 (3) | 0.039 (2) | 0.037 (2) | 0.003 (2) | −0.002 (2) | −0.009 (2) |
| C24 | 0.067 (3) | 0.060 (3) | 0.042 (3) | 0.011 (3) | −0.020 (3) | −0.022 (2) |
| C25 | 0.044 (3) | 0.058 (3) | 0.051 (3) | 0.007 (2) | −0.019 (2) | −0.023 (2) |
| O1 | 0.054 (2) | 0.060 (2) | 0.061 (2) | −0.0025 (18) | −0.0136 (19) | −0.031 (2) |
| N31 | 0.150 (16) | 0.126 (13) | 0.073 (4) | −0.026 (10) | 0.011 (6) | −0.052 (5) |
| C31 | 0.141 (15) | 0.097 (11) | 0.071 (4) | −0.041 (10) | 0.014 (6) | −0.045 (5) |
| C32 | 0.117 (15) | 0.094 (14) | 0.066 (4) | −0.033 (11) | 0.002 (6) | −0.041 (7) |
| N31′ | 0.162 (19) | 0.106 (13) | 0.070 (3) | −0.046 (12) | −0.019 (5) | −0.026 (5) |
| C31′ | 0.114 (13) | 0.100 (13) | 0.070 (3) | −0.052 (11) | −0.017 (5) | −0.026 (5) |
| C32′ | 0.18 (3) | 0.12 (2) | 0.070 (3) | −0.07 (2) | −0.020 (6) | −0.026 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Sb1—S4 | 2.3195 (13) | N12—H12 | 1.0000 |
| Sb1—S1 | 2.3200 (12) | C13—C14 | 1.510 (8) |
| Sb1—S3 | 2.3221 (12) | C13—H13A | 0.9900 |
| Sb1—S2 | 2.3382 (11) | C13—H13B | 0.9900 |
| S1—Co1 | 2.7258 (12) | C14—C15 | 1.514 (7) |
| S2—Co2 | 2.6932 (11) | C14—H14A | 0.9900 |

| | | | |
|---|---|---|---|
| S3—Co3 | 2.7821 (12) | C14—H14B | 0.9900 |
| Co1—N2[i] | 1.990 (4) | C15—H15A | 0.9900 |
| Co1—N2 | 1.990 (4) | C15—H15B | 0.9900 |
| Co1—N1 | 1.993 (4) | Co3—N22 | 1.976 (4) |
| Co1—N1[i] | 1.993 (4) | Co3—N22[iii] | 1.976 (4) |
| N1—C1 | 1.468 (6) | Co3—N21 | 1.985 (4) |
| N1—C5[i] | 1.470 (7) | Co3—N21[iii] | 1.985 (4) |
| N1—H1 | 1.0000 | N21—C21 | 1.472 (7) |
| C1—C2 | 1.504 (8) | N21—C25[iii] | 1.488 (6) |
| C1—H1A | 0.9900 | N21—H21 | 1.0000 |
| C1—H1B | 0.9900 | C21—C22 | 1.506 (8) |
| C2—N2 | 1.481 (6) | C21—H21A | 0.9900 |
| C2—H2A | 0.9900 | C21—H21B | 0.9900 |
| C2—H2B | 0.9900 | C22—N22 | 1.486 (7) |
| N2—C3 | 1.476 (6) | C22—H22A | 0.9900 |
| N2—H2 | 1.0000 | C22—H22B | 0.9900 |
| C3—C4 | 1.511 (8) | N22—C23 | 1.468 (6) |
| C3—H3A | 0.9900 | N22—H22 | 1.0000 |
| C3—H3B | 0.9900 | C23—C24 | 1.506 (9) |
| C4—C5 | 1.517 (8) | C23—H23A | 0.9900 |
| C4—H4A | 0.9900 | C23—H23B | 0.9900 |
| C4—H4B | 0.9900 | C24—C25 | 1.510 (8) |
| C5—H5A | 0.9900 | C24—H24A | 0.9900 |
| C5—H5B | 0.9900 | C24—H24B | 0.9900 |
| Co2—N11[ii] | 1.975 (4) | C25—H25A | 0.9900 |
| Co2—N11 | 1.975 (4) | C25—H25B | 0.9900 |
| Co2—N12 | 1.985 (4) | O1—H1C | 0.8400 |
| Co2—N12[ii] | 1.985 (4) | O1—H1D | 0.8400 |
| N11—C15[ii] | 1.475 (6) | N31—C31 | 1.145 (15) |
| N11—C11 | 1.486 (6) | C31—C32 | 1.442 (17) |
| N11—H11 | 1.0000 | C32—H32A | 0.9800 |
| C11—C12 | 1.514 (7) | C32—H32B | 0.9800 |
| C11—H11A | 0.9900 | C32—H32C | 0.9800 |
| C11—H11B | 0.9900 | N31′—C31′ | 1.145 (15) |
| C12—N12 | 1.470 (6) | C31′—C32′ | 1.442 (16) |
| C12—H12A | 0.9900 | C32′—H32D | 0.9800 |
| C12—H12B | 0.9900 | C32′—H32E | 0.9800 |
| N12—C13 | 1.474 (6) | C32′—H32F | 0.9800 |
| | | | |
| S4—Sb1—S1 | 109.86 (5) | H12A—C12—H12B | 108.4 |
| S4—Sb1—S3 | 110.64 (5) | C12—N12—C13 | 111.3 (4) |
| S1—Sb1—S3 | 110.81 (5) | C12—N12—Co2 | 108.0 (3) |
| S4—Sb1—S2 | 110.32 (5) | C13—N12—Co2 | 119.6 (3) |
| S1—Sb1—S2 | 105.36 (4) | C12—N12—H12 | 105.6 |
| S3—Sb1—S2 | 109.73 (4) | C13—N12—H12 | 105.6 |
| Sb1—S1—Co1 | 112.07 (5) | Co2—N12—H12 | 105.6 |
| Sb1—S2—Co2 | 122.01 (4) | N12—C13—C14 | 112.7 (4) |
| Sb1—S3—Co3 | 119.94 (5) | N12—C13—H13A | 109.0 |

| | | | |
|---|---|---|---|
| N2$^i$—Co1—N2 | 180.00 (19) | C14—C13—H13A | 109.0 |
| N2$^i$—Co1—N1 | 93.50 (17) | N12—C13—H13B | 109.0 |
| N2—Co1—N1 | 86.50 (17) | C14—C13—H13B | 109.0 |
| N2$^i$—Co1—N1$^i$ | 86.50 (17) | H13A—C13—H13B | 107.8 |
| N2—Co1—N1$^i$ | 93.50 (17) | C13—C14—C15 | 113.7 (4) |
| N1—Co1—N1$^i$ | 180.0 | C13—C14—H14A | 108.8 |
| N2$^i$—Co1—S1 | 89.02 (12) | C15—C14—H14A | 108.8 |
| N2—Co1—S1 | 90.98 (12) | C13—C14—H14B | 108.8 |
| N1—Co1—S1 | 88.50 (12) | C15—C14—H14B | 108.8 |
| N1$^i$—Co1—S1 | 91.50 (12) | H14A—C14—H14B | 107.7 |
| N2$^i$—Co1—S1$^i$ | 90.98 (12) | N11$^{ii}$—C15—C14 | 111.5 (4) |
| N2—Co1—S1$^i$ | 89.02 (12) | N11$^{ii}$—C15—H15A | 109.3 |
| N1—Co1—S1$^i$ | 91.50 (12) | C14—C15—H15A | 109.3 |
| N1$^i$—Co1—S1$^i$ | 88.50 (12) | N11$^{ii}$—C15—H15B | 109.3 |
| S1—Co1—S1$^i$ | 180.0 | C14—C15—H15B | 109.3 |
| C1—N1—C5$^i$ | 111.8 (4) | H15A—C15—H15B | 108.0 |
| C1—N1—Co1 | 107.1 (3) | N22—Co3—N22$^{iii}$ | 180.00 (19) |
| C5$^i$—N1—Co1 | 119.1 (3) | N22—Co3—N21 | 86.93 (17) |
| C1—N1—H1 | 106.0 | N22$^{iii}$—Co3—N21 | 93.07 (17) |
| C5$^i$—N1—H1 | 106.0 | N22—Co3—N21$^{iii}$ | 93.07 (17) |
| Co1—N1—H1 | 106.0 | N22$^{iii}$—Co3—N21$^{iii}$ | 86.93 (17) |
| N1—C1—C2 | 108.2 (4) | N21—Co3—N21$^{iii}$ | 180.0 |
| N1—C1—H1A | 110.1 | N22—Co3—S3$^{iii}$ | 87.74 (11) |
| C2—C1—H1A | 110.1 | N22$^{iii}$—Co3—S3$^{iii}$ | 92.26 (11) |
| N1—C1—H1B | 110.1 | N21—Co3—S3$^{iii}$ | 87.89 (12) |
| C2—C1—H1B | 110.1 | N21$^{iii}$—Co3—S3$^{iii}$ | 92.11 (12) |
| H1A—C1—H1B | 108.4 | N22—Co3—S3 | 92.26 (11) |
| N2—C2—C1 | 107.9 (4) | N22$^{iii}$—Co3—S3 | 87.74 (11) |
| N2—C2—H2A | 110.1 | N21—Co3—S3 | 92.11 (12) |
| C1—C2—H2A | 110.1 | N21$^{iii}$—Co3—S3 | 87.89 (12) |
| N2—C2—H2B | 110.1 | S3$^{iii}$—Co3—S3 | 180.00 (5) |
| C1—C2—H2B | 110.1 | C21—N21—C25$^{iii}$ | 111.9 (4) |
| H2A—C2—H2B | 108.4 | C21—N21—Co3 | 106.4 (3) |
| C3—N2—C2 | 111.7 (4) | C25$^{iii}$—N21—Co3 | 119.8 (3) |
| C3—N2—Co1 | 118.9 (3) | C21—N21—H21 | 105.9 |
| C2—N2—Co1 | 107.0 (3) | C25$^{iii}$—N21—H21 | 105.9 |
| C3—N2—H2 | 106.2 | Co3—N21—H21 | 105.9 |
| C2—N2—H2 | 106.2 | N21—C21—C22 | 107.7 (4) |
| Co1—N2—H2 | 106.2 | N21—C21—H21A | 110.2 |
| N2—C3—C4 | 112.0 (4) | C22—C21—H21A | 110.2 |
| N2—C3—H3A | 109.2 | N21—C21—H21B | 110.2 |
| C4—C3—H3A | 109.2 | C22—C21—H21B | 110.2 |
| N2—C3—H3B | 109.2 | H21A—C21—H21B | 108.5 |
| C4—C3—H3B | 109.2 | N22—C22—C21 | 107.0 (4) |
| H3A—C3—H3B | 107.9 | N22—C22—H22A | 110.3 |
| C3—C4—C5 | 114.6 (4) | C21—C22—H22A | 110.3 |
| C3—C4—H4A | 108.6 | N22—C22—H22B | 110.3 |
| C5—C4—H4A | 108.6 | C21—C22—H22B | 110.3 |

| | | | |
|---|---|---|---|
| C3—C4—H4B | 108.6 | H22A—C22—H22B | 108.6 |
| C5—C4—H4B | 108.6 | C23—N22—C22 | 112.3 (4) |
| H4A—C4—H4B | 107.6 | C23—N22—Co3 | 119.9 (3) |
| N1$^{i}$—C5—C4 | 112.6 (4) | C22—N22—Co3 | 106.9 (3) |
| N1$^{i}$—C5—H5A | 109.1 | C23—N22—H22 | 105.6 |
| C4—C5—H5A | 109.1 | C22—N22—H22 | 105.6 |
| N1$^{i}$—C5—H5B | 109.1 | Co3—N22—H22 | 105.6 |
| C4—C5—H5B | 109.1 | N22—C23—C24 | 112.5 (4) |
| H5A—C5—H5B | 107.8 | N22—C23—H23A | 109.1 |
| N11$^{ii}$—Co2—N11 | 180.0 | C24—C23—H23A | 109.1 |
| N11$^{ii}$—Co2—N12 | 93.73 (16) | N22—C23—H23B | 109.1 |
| N11—Co2—N12 | 86.27 (16) | C24—C23—H23B | 109.1 |
| N11$^{ii}$—Co2—N12$^{ii}$ | 86.27 (16) | H23A—C23—H23B | 107.8 |
| N11—Co2—N12$^{ii}$ | 93.73 (16) | C23—C24—C25 | 113.8 (5) |
| N12—Co2—N12$^{ii}$ | 180.0 | C23—C24—H24A | 108.8 |
| N11$^{ii}$—Co2—S2$^{ii}$ | 86.16 (11) | C25—C24—H24A | 108.8 |
| N11—Co2—S2$^{ii}$ | 93.84 (11) | C23—C24—H24B | 108.8 |
| N12—Co2—S2$^{ii}$ | 91.31 (11) | C25—C24—H24B | 108.8 |
| N12$^{ii}$—Co2—S2$^{ii}$ | 88.69 (11) | H24A—C24—H24B | 107.7 |
| N11$^{ii}$—Co2—S2 | 93.84 (11) | N21$^{iii}$—C25—C24 | 111.6 (4) |
| N11—Co2—S2 | 86.16 (11) | N21$^{iii}$—C25—H25A | 109.3 |
| N12—Co2—S2 | 88.69 (11) | C24—C25—H25A | 109.3 |
| N12$^{ii}$—Co2—S2 | 91.31 (11) | N21$^{iii}$—C25—H25B | 109.3 |
| S2$^{ii}$—Co2—S2 | 180.0 | C24—C25—H25B | 109.3 |
| C15$^{ii}$—N11—C11 | 111.4 (4) | H25A—C25—H25B | 108.0 |
| C15$^{ii}$—N11—Co2 | 118.1 (3) | H1C—O1—H1D | 102.1 |
| C11—N11—Co2 | 108.6 (3) | N31—C31—C32 | 172 (5) |
| C15$^{ii}$—N11—H11 | 106.0 | C31—C32—H32A | 109.4 |
| C11—N11—H11 | 106.0 | C31—C32—H32B | 109.5 |
| Co2—N11—H11 | 106.0 | H32A—C32—H32B | 109.5 |
| N11—C11—C12 | 107.6 (4) | C31—C32—H32C | 109.5 |
| N11—C11—H11A | 110.2 | H32A—C32—H32C | 109.5 |
| C12—C11—H11A | 110.2 | H32B—C32—H32C | 109.5 |
| N11—C11—H11B | 110.2 | N31′—C31′—C32′ | 172 (5) |
| C12—C11—H11B | 110.2 | C31′—C32′—H32D | 109.5 |
| H11A—C11—H11B | 108.5 | C31′—C32′—H32E | 109.5 |
| N12—C12—C11 | 108.2 (4) | H32D—C32′—H32E | 109.5 |
| N12—C12—H12A | 110.1 | C31′—C32′—H32F | 109.4 |
| C11—C12—H12A | 110.1 | H32D—C32′—H32F | 109.5 |
| N12—C12—H12B | 110.1 | H32E—C32′—H32F | 109.5 |
| C11—C12—H12B | 110.1 | | |

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

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| N1—H1···S2$^{i}$ | 1.00 | 2.48 | 3.442 (4) | 161 |

| | | | | |
|---|---|---|---|---|
| C1—H1*B*···S4<sup>iv</sup> | 0.99 | 2.95 | 3.858 (5) | 152 |
| N2—H2···S4 | 1.00 | 2.49 | 3.448 (4) | 159 |
| N11—H11···O1<sup>v</sup> | 1.00 | 2.23 | 3.151 (6) | 153 |
| N12—H12···S3<sup>ii</sup> | 1.00 | 2.43 | 3.378 (4) | 157 |
| N21—H21···O1 | 1.00 | 2.08 | 2.920 (6) | 141 |
| N22—H22···S1 | 1.00 | 2.35 | 3.290 (4) | 156 |
| O1—H1*C*···S2<sup>v</sup> | 0.84 | 2.49 | 3.276 (4) | 157 |
| O1—H1*D*···S4<sup>iii</sup> | 0.84 | 2.46 | 3.280 (4) | 166 |
| C32—H32*B*···S4 | 0.98 | 2.81 | 3.71 (4) | 154 |
| C32′—H32*F*···S4 | 0.98 | 2.88 | 3.85 (5) | 172 |

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