Poly[bis(μ₂-N,N-dimethylformamide-κ²O:O)bis(μ₄-
thiophene-2,5-dicarboxylato-κ⁴O:O':O'':O'''')-
dicobalt(II)]

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The asymmetric unit of the title three-dimensional metal–organic hybrid compound, [Co₂(C₆H₂O₄S)₂(C₃H₇NO)]ₙ, comprises two cobalt(II) cations, one residing on a twofold axis and the other on a centre of inversion, one thiophene-2,5-dicarboxylate (tdc²⁻) ligand and one coordinating dimethylformamide (DMF) solvent molecule. Both of the cobalt(II) cations exhibit an octahedral coordination environment from the four carboxyl O atoms of the tdc²⁻ anions in a μ₄-κ¹:κ¹:κ¹:κ¹ fashion and two O atoms from DMF. A pair of carboxyl O atoms and one DMF molecule connect the adjacent cobalt(II) cations into an infinite chain, leading to a rod-spacer framework with rhombus-window channels, yet no residual solvent-accessible voids are present because the coordinating DMF molecules are oriented into the potential channels.

Structure description

Facing the timetable for a carbon-neutral future, electrochemical redox reactions are the cornerstones of large-scale storage and chemical conversion of renewable clean energy in the future, in which electrocatalytic water splitting plays a central role (Seh et al., 2017; Cheng et al., 2022). Metal–organic frameworks (MOFs), a class of crystalline and highly porous frameworks usually constructed from 3d metal ions and organic ligands (Yin et al., 2015), provide great opportunities for the preparation of new electrocatalysts for water splitting. Benefitting from outstanding designability and regulation for the composition and structure of MOFs, 3d-metal-based electrocatalysts with excellent electrocatalyst performance can be obtained from both highly stable MOFs and nanocomposites derived from the thermal or chemical reaction of the MOF precursor (Zhu et al., 2018). In a previous study, we discovered alkali-induced in situ formation of amorphous NiₓFe₁₋ₓ...
from a linear $[M_3(COO)_6]$-based MOF template for overall electrochemical water splitting (Yin et al., 2015).

In parallel work, thiophene-2,5-dicarboxylic acid (H$_2$tdc) and the cobalt ion were chosen to construct MOFs for potential electrochemical applications. The H$_2$tdc ligand is a typical di-topic linker comparable to terephthalic acid that has potential electrochemical applications. The H$_2$tdc ligand is a second on an inversion centre), one full tdc$^{2-}$ ligand, and one coordinating DMF molecule Each of the cobalt(II) cations exhibits an octahedral coordination geometry by the four carboxyl O atoms from the tdc$^{2-}$ anions in a $\mu_2$-$\kappa^2$-$\kappa'$-$\kappa'$ fashion and two O atoms from DMF. The calculated continuous shape measures (CShM) value for Co1 and Co2 are 0.338 and 0.240, respectively, indicating only quite a small coordination distortion from a regular octahedron. A pair of carboxyl and one DMF link adjacent cobalt(II) cations into infinite chains via $C-H\cdot\cdot\cdot O$ hydrogen bonds (Table 1, Fig. 2).

In particular, the DMF ligand adopts a $\mu_2$-bridging mode to link adjacent metal ions. Compared to its usual role as a terminally bound ligand, such coordination behaviour is rare but has been observed in some known MOFs (Fritzsche et al., 2019). As a result, a rod–spacer framework with rhombus-linkage of neighbouring chains. However, no solvent-accessible voids were noted because the coordinating DMF molecule is oriented into the channels and fully occupies any potential void space. The compound is thermally stable up to 260°C under an N$_2$ atmosphere by thermogravimetric analysis. Thermogravimetric analysis: the mass of the compound remains stable until 250°C, followed by an obvious mass loss of 23.7% corresponding to the loss of coordinating DMF (calculated 26.8%) in the range of 250–310°C, and then thermal decomposition of the framework with residuals of 34.3% from 400–800°C, much higher than the theoretical data for decomposition products of Co$_3$O$_4$ (calculated 27.7%) or CoO (calculated 26.0%), suggesting the formation of carbon- and sulfur-rich nanocomposites.

### Synthesis and crystallization

A solution of H$_2$tdc (0.2 mmol, 34.4 mg) and CoCl$_2$·6H$_2$O (0.2 mmol, 47.6 mg) in DMF (dimethyl formamide, 15 ml) was stirred in air with a magnetic stirrer, generating a purple transparent solution after stirring for 5 min. The reaction solution was transferred to a hydrothermal reaction vessel containing 25 ml of a polytetrafluoroethylene liner, followed by heating at 140°C for 48 h. The reaction vessel was cooled to room temperature at a rate of 10°C per hour. The precipitate was washed and filtered to obtain a large amount of light-
purple block-shaped crystals of the title compound with a yield of about 60% (based on Co). The obtained crystals are insoluble in common organic solvents of DMF, CH3OH, C2H5OH, CH2Cl2 and acetone. IR (KBr pellets, cm\(^{-1}\)): 3446 (bm), 2943 (vs), 1654(s), 1532(s), 1370(vs), 1106(s), 1010(s), 771(m), 674(w). Elemental analysis (%), calculated: C, 39.63; H, 3.33; N, 5.14; S, 11.76; found: C, 38.83; H, 3.76; N, 4.95; S, 12.02.

**Refinement**

Crystal data, data collection and structure refinement details are summarized in Table 2. Atoms C3 and C4 of the thiazole ring and the C atoms of the coordinating DMF are disordered over two sets of sites with occupancy ratios of 0.550 (17):0.450 (17) nd 0.855 (5):0.145 (5), respectively. Disorder treatment and restraints for the displacement parameters of the thiazole ring and coordinated DMF were applied. Disorder was treated as follows: two adjacent carbon atoms C3, C4 in the thiazole ring were split into two parts, and the C7, C8, C9 atoms in the DMF were split into two positions also, followed by SIMU restraints for these atoms and subsequent refinements, resulting in lower, acceptable R-factors and refinement.

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**References**

Brandenburg, K. & Putz, H. (2019). DIAMOND. Crystal Impact GbR, Bonn, Germany.

Bruker (2016). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.

Cheng, Y., Yin, Z., Ma, W. M., He, Z. X., Yao, X. & Lv, W. Y. (2022). Inorg. Chem. 61, 3327–3336.

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**Table 2**

| Crystal data                                      | [Co(C6H2O4S)(C3H7NO)] |
|--------------------------------------------------|-----------------------|
| Chemical formula                                 | [Co(C12H16O10S)(C3H7NO)] |
| Crystal system, space group                      | Monoclinic, \(C2/c\) |
| Temperature (K)                                  | 298 (2) |
| \(a\), \(b\), \(c\) (\(\text{\AA}\))          | 11.610 (2), 18.046 (4), 11.496 (2) |
| \(\beta\) (\(^\circ\))                         | 102.35 (5) |
| \(V\) (\(\text{\AA}^3\))                      | 2352.9 (9) |
| \(Z\)                                            | 8 |
| Radiation type                                   | Mo K\(\alpha\) |
| \(\mu\) (mm\(^{-1}\))                          | 1.64 |
| Crystal size (mm)                                | 0.24 \(\times\) 0.15 \(\times\) 0.11 |

**Data collection**

| Diffractometer                                  | Bruker APEXI CCD |
|-------------------------------------------------|------------------|
| Absorption correction                           | Multi-scan (SADABS; Bruker, 2016) |
| \(\text{\(T_{min}\)}}, \(\text{\(T_{max}\)}\) | 0.656, 0.746 |
| No. of measured, independent and observed \([I > 2\sigma(I)]\) reflections | 12568, 2928, 2285 |
| \(R_{int}\)                                     | 0.054 |
| \(\text{\(\sin \theta\)}\)\(_{\text{\(max\)}}\) (\(\text{\AA}^{-1}\)) | 0.667 |

**Refinement**

| \(R[F^2 > 2\sigma(F^2)]\), \(wR(F^2)\), \(S\)  | 0.040, 0.089, 1.10 |
| No. of reflections                               | 2928 |
| No. of parameters                                | 203 |
| No. of restraints                                | 90 |
| H-atom treatment                                 | H-atom parameters constrained |
| \(\Delta \rho_{\text{max}}\), \(\Delta \rho_{\text{min}}\) (e \(\text{\AA}^{-3}\)) | 0.48, –0.44 |

**Computer programs:** APEX2 and SAINT (Bruker, 2016), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), OLEX2 (Dolomanov et al., 2009) and DIAMOND (Brandenburg & Putz, 2019).

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). J. Appl. Cryst. 42, 339–341.

Fritzsche, J., Ettlinger, R., Grzywa, M., Jantz, S. G., Kalytta-Mewes, A., Bunzen, H., Hoppe, H. A. & Volkmer, D. (2019). Dalton Trans. 48, 15236–15246.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). Acta Cryst. B72, 171–179.

Seh, Z. W., Kibsgaard, J., Dickens, C. F., Chorkendorff, I., Norskov, J. K. & Jaramillo, T. F. (2017). Science, 355, aad4998. https://doi.org/10.1126/science.aad4998.

Sheldrick, G. M. (2015a). Acta Cryst. A71, 3–8.

Sheldrick, G. M. (2015b). Acta Cryst. C71, 3–8.

Tan, Y. X., He, Y. P., Zhang, Y., Zheng, Y. J. & Zhang, J. (2013). CrystEngComm, 15, 6009–6014.

Yin, Z., Zhou, Y. L., Zeng, M. H. & Kurmoo, M. (2015). Dalton Trans. 44, 5258–5275.

Zhu, B. J., Zou, R. Q. & Xu, Q. (2018). Adv. Energy Mater. 8, 1801193.
full crystallographic data

**Poly[bis(µ₂-Ν,Ν-dimethylformamide-κ²O:O)bis(µ₄-thiophene-2,5-dicarboxylato-κ⁴O:O′:O″:O‴)dicobalt(II)]]**

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**Crystal data**

\[\text{[Co(C}_6\text{H}_2\text{O}_4\text{S})(\text{C}_3\text{H}_7\text{NO})]\]

- \(M_r = 302.16\)
- Monoclinic, \(C_2/c\)
- \(a = 11.610 (2) \text{ Å}\)
- \(b = 18.046 (4) \text{ Å}\)
- \(c = 11.496 (2) \text{ Å}\)
- \(β = 102.35 (3)°\)
- \(V = 2352.9 (9) \text{ Å}^3\)
- \(Z = 8\)
- \(F(000) = 1224\)
- \(D_x = 1.706 \text{ Mg m}^{-3}\)
- Mo Ka radiation, \(λ = 0.71073 \text{ Å}\)
- Cell parameters from 4391 reflections
- \(θ = 3.6–27.9°\)
- \(µ = 1.64 \text{ mm}^{-1}\)
- \(T = 298 \text{ K}\)
- Block, clear dark violet
- \(0.24 \times 0.15 \times 0.11 \text{ mm}\)

**Data collection**

- Bruker APEXII CCD diffractometer
- \(ω\) scans
- Absorption correction: multi-scan (SADABS; Bruker, 2016)
- \(T_{\text{min}} = 0.656, T_{\text{max}} = 0.746\)
- 12568 measured reflections

**Refinement**

- Refinement on \(F^2\)
- Least-squares matrix: full
- \(R[F^2 > 2σ(F^2)] = 0.040\)
- \(wR(F^2) = 0.089\)
- \(S = 1.10\)
- 2928 reflections
- 203 parameters
- 90 restraints

- Primary atom site location: difference Fourier map
- Hydrogen site location: inferred from neighbouring sites
- H-atom parameters constrained
- \(w = 1/[σ^2(F_o^2) + (0.0336P)^2 + 4.208P]\)
- \[(Δ/σ)_{\text{max}} < 0.001\]
- \(Δρ_{\text{max}} = 0.48 \text{ e Å}^{-3}\)
- \(Δρ_{\text{min}} = -0.43 \text{ e Å}^{-3}\)

**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.
Refinement. The single-crystal diffraction data were collected on a Bruker APEX-II CCD diffractometer (Mo-Kα, λ = 0.71073 Å), with the APEX-II software for data reduction and analysis (Bruker 2016). The dataset of a selected single-crystal of (I) were collected at 298 K. The structure was solved by direct methods and refined by full-matrix least-squares method on F2 using SHELX algorithms in Olex2 (Sheldrick 2008; Dolomanov et al., 2009). All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were generated geometrically.

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

|   | x   | y   | z   | Uiso*/Ueq | Occ. (<1) |
|---|-----|-----|-----|-----------|-----------|
| Co1 | 0.0000 | 0.22585 (3) | 0.2500 | 0.01571 (14) |           |
| Co2 | 0.2500 | 0.2500 | 0.5000 | 0.01521 (13) |           |
| S1  | 0.28427 (7) | 0.49423 (4) | 0.30550 (7) | 0.0294 (2) |           |
| O1  | 0.11164 (18) | 0.30921 (11) | 0.22411 (18) | 0.0282 (5) |           |
| O2  | 0.24100 (19) | 0.34160 (11) | 0.39173 (18) | 0.0292 (5) |           |
| O3  | 0.21863 (18) | 0.67965 (10) | 0.12859 (17) | 0.0238 (4) |           |
| O4  | 0.38032 (18) | 0.64477 (11) | 0.26243 (18) | 0.0266 (5) |           |
| O5  | 0.06101 (17) | 0.22609 (11) | 0.44616 (17) | 0.0226 (4) |           |
| N1  | −0.0669 (3) | 0.2096 (2) | 0.5674 (3) | 0.0460 (8) |           |
| C1  | 0.1779 (2) | 0.35367 (14) | 0.2909 (2) | 0.0203 (6) |           |
| C2  | 0.1840 (3) | 0.42955 (16) | 0.2402 (3) | 0.0288 (7) |           |
| C3A | 0.1295 (10) | 0.4528 (5) | 0.1270 (9) | 0.035 (2) | 0.550 (17) |
| H3A | 0.0808 | 0.4227 | 0.0719 | 0.043* | 0.550 (17) |
| C4A | 0.1552 (10) | 0.5261 (5) | 0.1043 (9) | 0.035 (2) | 0.550 (17) |
| H4A | 0.1262 | 0.5500 | 0.0323 | 0.042* | 0.550 (17) |
| C3B | 0.1167 (12) | 0.5369 (7) | 0.1402 (12) | 0.039 (2) | 0.450 (17) |
| H3B | 0.0643 | 0.5678 | 0.0901 | 0.047* | 0.450 (17) |
| C4B | 0.0916 (11) | 0.4632 (7) | 0.1631 (12) | 0.036 (2) | 0.450 (17) |
| H4B | 0.0209 | 0.4396 | 0.1302 | 0.043* | 0.450 (17) |
| C5  | 0.2283 (3) | 0.55903 (16) | 0.2002 (3) | 0.0285 (7) |           |
| C6  | 0.2810 (3) | 0.63407 (15) | 0.1970 (2) | 0.0205 (6) |           |
| C7A | −0.0030 (3) | 0.2499 (2) | 0.5112 (3) | 0.0309 (9) | 0.855 (5) |
| H7A | −0.0058 | 0.3010 | 0.5208 | 0.037* | 0.855 (5) |
| C8A | −0.0681 (5) | 0.1296 (3) | 0.5558 (5) | 0.0716 (17) | 0.855 (5) |
| H8AA | −0.1193 | 0.1088 | 0.6026 | 0.107* | 0.855 (5) |
| H8AB | −0.0962 | 0.1164 | 0.4737 | 0.107* | 0.855 (5) |
| H8AC | 0.0103 | 0.1107 | 0.5832 | 0.107* | 0.855 (5) |
| C9A | −0.1409 (5) | 0.2434 (4) | 0.6420 (5) | 0.0772 (19) | 0.855 (5) |
| H9AA | −0.1810 | 0.2052 | 0.6758 | 0.116* | 0.855 (5) |
| H9AB | −0.0922 | 0.2714 | 0.7047 | 0.116* | 0.855 (5) |
| H9AC | −0.1978 | 0.2756 | 0.5941 | 0.116* | 0.855 (5) |
| C7B | 0.008 (2) | 0.1836 (16) | 0.497 (2) | 0.044 (4) | 0.145 (5) |
| H7B | 0.0183 | 0.1328 | 0.4896 | 0.052* | 0.145 (5) |
| C9B | −0.078 (3) | 0.291 (2) | 0.591 (3) | 0.072 (5) | 0.145 (5) |
| H9BA | −0.1340 | 0.2977 | 0.6413 | 0.107* | 0.145 (5) |
| H9BB | −0.0030 | 0.3101 | 0.6308 | 0.107* | 0.145 (5) |
| H9BC | −0.1051 | 0.3165 | 0.5175 | 0.107* | 0.145 (5) |
| C8B | −0.118 (3) | 0.154 (2) | 0.628 (3) | 0.085 (6) | 0.145 (5) |
| H8BA | −0.1682 | 0.1761 | 0.6743 | 0.128* | 0.145 (5) |
### Data Reports

|     | 0.1631 | 0.1202 | 0.5712 | 0.128* | 0.145 (5) |
|-----|--------|--------|--------|--------|-----------|
| H8BB| 0.0562 | 0.1265 | 0.6802 | 0.128* | 0.145 (5) |

### Atomic Displacement Parameters (Å²)

|     | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|-----|----------|----------|----------|----------|----------|----------|
| Co1 | 0.0156 (3)| 0.0107 (3)| 0.0203 (3)| 0.000    | 0.0029 (2)| 0.000    |
| Co2 | 0.0192 (3)| 0.0098 (2)| 0.0164 (2)| −0.0001 (2)| 0.0033 (2)| −0.00102 (18)|
| S1  | 0.0331 (4)| 0.0171 (4)| 0.0303 (4)| −0.0084 (3)| −0.0102 (3)| 0.0082 (3)|
| O1  | 0.0318 (12)| 0.0204 (11)| 0.0297 (11)| −0.0118 (9)| 0.0004 (9)| 0.0050 (8)|
| O2  | 0.0403 (13)| 0.0151 (10)| 0.0280 (11)| −0.0063 (9)| −0.0020 (9)| 0.0069 (8)|

### Geometric Parameters (Å, °)

|       | 2.0485 (19)| 2.0484 (19)| 2.0438 (19)| 2.0437 (19)| 2.215 (2)| 2.215 (2)| 2.0585 (19)| 2.0585 (19)| 2.0397 (18)| 2.0396 (18)| 2.192 (2)| 2.192 (2)| 1.705 (3)| 1.709 (3)| 1.254 (3)| 1.251 (3)|
|-------|-------------|-------------|-------------|-------------|----------|----------|-------------|-------------|-------------|-------------|----------|----------|---------|---------|---------|---------|---------|---------|
| Co1—O1 | 2.0485 (19) | 2.0484 (19) | 2.0438 (19) | 2.0437 (19) | 2.215 (2) | 2.215 (2) | 2.0585 (19) | 2.0585 (19) | 2.0397 (18) | 2.0396 (18) | 2.192 (2) | 2.192 (2) | 1.705 (3) | 1.709 (3) | 1.254 (3) | 1.251 (3) |
| Bond                     | Distance (Å) | Angle (°) |
|--------------------------|--------------|-----------|
| O3—Co2^vi                | 2.0396 (18)  |           |
| O3—Co2                   | 1.255 (3)    |            |
| O4—Co1^vii               | 2.0438 (19)  |           |
| O4—C6                     | 1.250 (3)    |            |
| O5—C7A                    | 1.239 (4)    |            |
| O5—C7B                    | 1.21 (3)     |            |
| N1—C7A                   | 1.305 (5)    |            |
| N1—C8A                   | 1.449 (6)    |            |
| N1—C9A                   | 1.469 (6)    |            |
| N1—C7B                   | 1.39 (3)     |            |
| N1—C9B                   | 1.50 (4)     |            |
| O1—Co1—O1^i              | 85.50 (13)   | 110.2 (5) |
| O1—Co1—O5^i              | 94.13 (8)    | 123.9 (5) |
| O1—Co1—O5                | 94.13 (8)    | 123.5    |
| O1—Co1—O5^i              | 85.71 (8)    | 113.1 (6) |
| O1—Co1—O5                | 85.71 (8)    | 123.5    |
| O4—Co1—O1^i              | 175.30 (8)   | 123.8    |
| O4—Co1—O1                | 93.14 (9)    | 112.4 (6) |
| O4—Co1—O1                | 93.14 (9)    | 123.8    |
| O4—Co1—O1                | 175.30 (8)   | 123.5    |
| O4^iv—Co1—O1             | 88.56 (12)   | 123.5    |
| O4—Co1—O5^i              | 90.25 (8)    | 112.9 (8) |
| O4—Co1—O5                | 90.25 (8)    | 124.0    |
| O4—Co1—O5                | 89.91 (8)    | 124.0    |
| O5—Co1—O5                | 179.78 (11)  | 110.4 (4) |
| O2—Co2—O2                | 180.00 (9)   | 124.1 (4) |
| O2—Co2—O2                | 86.04 (8)    | 109.1 (5) |
| O2—Co2—O5^iv             | 86.04 (8)    | 126.4 (5) |
| O2—Co2—O5                | 93.96 (8)    | 122.9 (2) |
| O2—Co2—O5                | 93.96 (8)    | 115.2 (2) |
| O3—Co2—O2                | 86.82 (8)    | 127.6 (3) |
| O3—Co2—O2^v              | 86.83 (8)    | 117.2 (2) |
| O3—Co2—O2                | 93.17 (8)    | 125.7 (4) |
| O3—Co2—O2                | 93.18 (8)    | 117.1    |
| O3—Co2—O3^a              | 180.0        | 117.1    |
| O3—Co2—O5^v              | 90.24 (8)    | 109.5    |
| O3—Co2—O5^v              | 89.76 (8)    | 109.5    |
| O3—Co2—O5                | 89.76 (8)    | 109.5    |
| O3—Co2—O5                | 90.24 (8)    | 109.5    |
| O5—Co2—O5                | 180.0        | 109.5    |
| C2—S1—C5                 | 92.08 (15)   | 109.5    |
| C1—O1—Co1                | 134.86 (19)  | 109.5    |
| C1—O2—Co2                | 130.09 (18)  | 109.5    |
| C6—O3—Co2^vi             | 133.92 (19)  | 109.5    |
| C6—O4—Co1^vii            | 128.48 (18)  | 109.5    |
| Co2—O5—Co1               | 111.77 (9)   | 109.5    |
C7A—O5—Co1 120.9 (2)  H9AB—C9A—H9AC 109.5
C7A—O5—Co2 117.1 (2)  O5—C7B—N1 121 (2)
C7B—O5—Co1 114.2 (11)  O5—C7B—H7B 119.6
C7B—O5—Co2 124.6 (11)  N1—C7B—H7B 119.6
C7A—N1—C8A 120.3 (4)  N1—C9B—H9BA 109.5
C7A—N1—C9A 121.5 (4)  N1—C9B—H9BB 109.5
C7B—N1—C8B 121.7 (18)  H9BA—C9B—H9BB 109.5
C7B—N1—C8B 115 (2)  H9BA—C9B—H9BC 109.5
C8B—N1—C9B 123 (2)  H9BB—C9B—H9BC 109.5
O1—C1—C2 114.9 (2)  N1—C8B—H8BA 109.5
O2—C1—O1 128.1 (2)  N1—C8B—H8BB 109.5
O2—C1—C2 116.9 (2)  N1—C8B—H8BC 109.5
C1—C2—S1 122.7 (2)  H8BA—C8B—H8BB 109.5
C3A—C2—S1 109.7 (4)  H8BA—C8B—H8BC 109.5
C3A—C2—C3A 126.4 (4)  H8BB—C8B—H8BC 109.5
Co1—O1—C1—O2 38.9 (5)  C1—C2—C3A—C4A −177.8 (5)
Co1—O1—C1—C2 −143.4 (2)  C1—C2—C4A—C3B 173.3 (6)
Co1—O4—C6—O3 −20.0 (5)  C2—S1—C5—C4A −14.2 (6)
Co1—O4—C6—C5 160.7 (2)  C2—S1—C5—C3B 16.9 (8)
Co1—O5—C7A—N1 99.3 (4)  C2—S1—C5—C6 −176.5 (3)
Co1—O5—C7B—N1 −110.7 (16)  C2—S3A—C4A—C5 −0.6 (10)
Co2—O2—C1—O1 −8.1 (5)  C3A—C4A—C5—S1 10.9 (9)
Co2—O2—C1—C2 174.2 (2)  C3A—C4A—C5—C6 173.0 (5)
Co2—O3—C6—O4 49.8 (4)  C4A—C5—C6—O3 32.4 (8)
Co2—O3—C6—C5 −130.8 (2)  C4A—C5—C6—O4 −148.1 (7)
Co2—O5—C7A—N1 −118.5 (4)  C3B—C5—C6—O3 −3.5 (10)
Co2—O5—C7B—N1 105.9 (18)  C3B—C5—C6—O4 176.0 (9)
S1—C2—C3A—C4A −10.0 (9)  C4B—C3B—C5—S1 −12.7 (11)
S1—C2—C4B—C3B 12.8 (10)  C4B—C3B—C5—C6 −178.7 (6)
S1—C5—C6—O3 −167.7 (2)  C5—S1—C2—C1 −177.9 (3)
S1—C5—C6—O4 11.8 (4)  C5—S1—C2—C3A 13.7 (6)
O1—C1—C2—S1 −170.0 (2)  C5—S1—C2—C4B −17.2 (8)
O1—C1—C2—C3A −3.6 (8)  C5—S1—C2—C4B 0.0 (12)
O1—C1—C2—C4B 31.9 (9)  C8A—N1—C7A—O5 −0.7 (6)
O2—C1—C2—S1 8.0 (4)  C9A—N1—C7A—O5 −179.5 (4)
O2—C1—C2—C3A 174.4 (7)  C9B—N1—C7B—O5 −5 (3)
O2—C1—C2—C4B −150.1 (9)  C8B—N1—C7B—O5 −176 (2)

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

**Hydrogen-bond geometry (Å, °)**

| D—H···A   | D—H | H···A | D···A   | D—H···A   |
|-----------|-----|-------|---------|-----------|
| C8A—H8AB···O4iii | 0.96 | 2.44  | 3.308 (6) | 150       |

IUCrData (2022). 7, x220775 data-5
C9A—H9A···O1\textsuperscript{vii} & 0.96 & 2.60 & 3.347 (6) & 135 \\
C9B—H9BB···O3\textsuperscript{v} & 0.96 & 2.58 & 3.42 (4) & 146 \\

Symmetry codes: (iii) \(x-1/2, y-1/2, z\); (v) \(x, -y+1, z+1/2\); (viii) \(x-1/2, -y+1/2, z+1/2\).