Crystal structure of a copper–mefenamate complex solvated with diglyme and water

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1. Chemical context

Mefenamic acid is a non-steroidal anti-inflammatory drug (NSAID) that is synthesized through reaction of 2-chlorobenzoic acid and 2,3-dimethylaniline in the presence of a copper catalyst (Trinus et al., 1977). Subsequently, pharmacopoeia specifications for mefenamic acid specify a maximum limit of 10 ppm for the quantity of copper present in the final drug product (British Pharmacopoeia, 2017). In exploring strategies to ensure removal of copper from the crude reaction mixture, a new copper–mefenamate complex was isolated. The crystal structure of a copper–mefenamate complex solvated with water and diglyme is reported.

2. Structural commentary

The complex [Cu₂(mefenamate)₄(H₂O)₂]·2(diglyme) crystallizes in the space group P2₁/n, with a [Cu₂(RCO₂)₄(H₂O)₂]
The paddlewheel motif that is typical for coordination of four carboxylate groups to two CuII cations (Chong et al., 2022). Within the asymmetric unit (Fig. 1a), the planes of the 2,3-dimethylphenyls from the two mefenamate molecules are 42.61 (1)° apart. A water molecule occupies each of the apical positions of the paddlewheel motif, which is hydrogen bonded to a diglyme molecule (Fig. 1a). The diglyme molecule is oriented such that it fits between the 2,3-dimethylphenyl units of the two mefenamate molecules in the asymmetric unit and is hydrogen bonded to the coordinated water via the diglyme outer oxygen positions (Fig. 1a). A distorted square-pyramidal geometry is adopted by each CuII cation in the paddlewheel motif (Fig. 1b), with equatorial Cu—O distances of 1.968 (1), 1.961 (1), 1.954 (1), and 1.969 (1) Å between CuII and the carboxylate moieties. The axial Cu—O distance, between the copper(II) cation and water molecule, is 2.108 (1) Å. The distance between the two CuII cations is 2.6126 (4) Å. There is an intramolecular bond between the amine and carboxylate groups of the mefenamate, with O—C distances of 1.86 (3) and 1.87 (2) Å for mefenamate units A and B, respectively (Table 1).

3. Supramolecular features
There are no obvious interactions, such as π–π stacking, between neighbouring paddlewheel units within the packed structure. The paddlewheel units interact through edge-to-face interactions of the phenyl groups of the mefanamate ligands (Fig. 1c). In the global packing of the structure, the paddlewheel units are arranged as 2D sheets along the crystallographic ab plane, with symmetry-equivalent sheets repeating throughout the crystallographic c axis at a distance corresponding to c. A second 2D arrangement is intercalated halfway between the symmetry-equivalent sheets.

4. Database survey
There are three other similar copper–mefenamate paddlewheel structures in the CSD (version 5.43, November 2021; Groom et al., 2016), with different solvents occupying the apical positions. Two entries, MPANCU10 (Yatsimirskii et al., 1979) and MPANCU20 (Mys‘kiv et al., 1982), are with N,N-dimethylformamide (DMF) and one entry, SUTPIG (Facchin et al., 1998), has dimethyl sulfoxide (DMSO) occupying the apical position. The DMF analogue also crystallizes in a monoclinic space group (Table 2). The cell volume of [Cu2(mefenamate)4(H2O)2]·2H2O is larger than the DMF analogue (3026.535 Å³), to accommodate the larger diglyme molecule. The axial Cu—O distance in [Cu2(mefenamate)4(H2O)2]·2diglyme is shorter than those

| D—H—A | D—H | H—A | D—A | D—H—A |
|--------|-----|-----|-----|-------|
| N10A—H10DE2A | 0.86 (3) | 1.86 (3) | 2.604 (2) | 143 (2) |
| N10B—H10CE2B | 0.89 (2) | 1.87 (2) | 2.6065 (18) | 139 (2) |

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

Figure 1
Views of [Cu2(mefenamate)4(H2O)2]·2diglyme as an ORTEP representation with ellipsoids set to 50% probability: (a) asymmetric unit with hydrogen bonds highlighted (dashed blue lines), (b) a single paddlewheel unit of the complex, and (c) neighbouring units with edge-to-face interactions highlighted (dashed red lines).
in structures MPANCU20 and SUTP1G (Table 2). This may be attributed to the higher polarity of water (1.000) compared to DMF and DMSO (0.386 and 0.444, respectively; Reichardt & Welton, 2011). In the DMSO analogue, the 2,3-dimethylphenyls from the two mefenamate molecules within the asymmetric unit are almost coplanar, the planes are 9.06° apart, and the methyl groups of the DMSO point away from the 2,3-dimethylphenyls. For the DMF analogue, the two 2,3-dimethylphenyls are 70.22° apart, and the methyl groups of the DMSO point away from the 2,3-dimethylphenyls.

Three polymorphic forms are known for mefenamic acid, with significant differences between the forms in the C9−N10−C16 torsion angle \( \tau_3 \) (Fig. 1a; SeethaLekshmi & Guru Row, 2012). The larger torsion angle \( \tau_3 \) observed with the copper complex (Table 2) is more consistent with those of XYANAC03 (Yang et al., 2017). The increased torsional angle can be explained by the location of the dimethylphenyl group with respect to the diglyme group. The phenyl group needs to rotate to ensure a more planar packing arrangement with the diglyme molecule. In comparison to other polymorphs, the metastable form II suffers from significant disorder around the diglyme molecule. In comparison to other polymorphs, the 2,3-dimethylphenyls from the two mefenamate molecules within the asymmetric unit are almost coplanar, the planes are 9.06° apart, and the methyl groups of the DMSO point away from the 2,3-dimethylphenyls. For the DMF analogue, the two 2,3-dimethylphenyls are 70.22° apart.

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5. Synthesis and crystallization

Chemicals were purchased from commercial suppliers and used as received without further purification. Deionized water was obtained from an in-house Milli-Q (Millipore) purification system. A solution was prepared comprising mefenamic acid (25.0 g), diglyme (281.6 g), water (74.7 g) and copper (II) acetate (7.3 g). An aliquot (4 mL) of this solution was removed and mefenamic acid (0.4 g) added to generate a slurry. The mixture was filtered and the filtrate stored in the dark at room temperature for two weeks, after which large green block-shaped crystals of the complex had formed.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The diglyme moiety was found to be disordered over two positions. Initial isotropic refinement of the diglyme allowed the residual electron density to be observed. Using the functionality in OLEX2, the atoms were moved to ensure that they overlapped the electron density in a

### Table 2

**Comparison of selected geometries (Å, °).**

Coordinates are unavailable for entry MPANCU10. There are two values per structure, corresponding to the two mefenamate units in the asymmetric unit as denoted \( A \) and \( B \) in our atom-numbering scheme.

|                           | This work | MPANCU20 | SUTP1G |
|---------------------------|-----------|----------|--------|
| Space group               | \( P2_1/n \) | \( P2_1/n \) | \( P2_1/n \) |
| \( O1−C3−C4−C9 \)         | 171.1 (2)  | 170.98   | 153 (1) |
| \( C4−C9−N10−C11 \)       | 174.3 (2)  | 171.2 (2) | 171 (1) |
| \( O1−C3−C4−C9 \)         | 171.1 (2)  | -166.40  | 171 (1) |
| \( C4−C9−N10−C11 \)       | 174.3 (2)  | -155.25  | 172 (1) |
| \( Cu−O_mefenanmate \)    | 1.961 (1)  | 1.9737   | 107 (2) |
| \( Cu−O_{solvent} \)      | 2.108 (1)  | 1.954 (1)| 135 (2) |
| \( Cu−..Cu \)             | 2.6126 (4) | 2.1561   | 1.949 (7)|

### Table 3

**Experimental details.**

| Crystal data | Chemical formula | \( [\text{Cu}_2(C_15H_{14}NO_2)_4(H_2O)_2] \cdot 2C_6H_{14}O_3 \) |
|--------------|------------------|-------------------------------------------------------------|
| \( M_w \)    | 1392.54          |                                                              |
| Crystal system, space group | Monoclinic, \( P2_1/n \) |                                                              |
| Temperature (K) | 105               |                                                              |
| \( \beta (°) \)  | 94.791 (1)       |                                                              |
| \( V (A^3) \)   | 3539.25 (12)     |                                                              |
| \( Z \)          | 2                |                                                              |
| Radiation type  | Cu Kα            |                                                              |
| \( \mu (mm^{-1}) \) | 0.2 × 0.15 × 0.1 |                                                              |
| Crystal size (mm) | 1.30              |                                                              |

| Data collection | Diffractometer | Absorption correction |
|-----------------|----------------|-----------------------|
|                 | Bruker Photon100 CMOS | Multi-scan (SADABS; Bruker, 2016) |

| \( T_{\text{min}}, T_{\text{max}} \) | 0.781, 0.881 | 209288, 6424, 5989 |
| No. of measured, independent and observed \( | F > 2\sigma(F) \) | reflections | 0.039 |
| \( R_{\text{int}} \) (\( sin \theta/\lambda_{\text{max}} \) (Å⁻¹)) | 0.603 |

| Refinement | \( R(F^2 > 2\sigma(F^2)) \), \( wR(F^2) \), \( S \) | 0.033, 0.089, 1.06 |
| No. of reflections | 6424 | |
| No. of parameters | 477 |
| No. of restraints | 410 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| \( \Delta \rho_{\text{max}}, \Delta \rho_{\text{min}} \) (e Å⁻³) | 0.28, −0.40 |

Computer programs: APEX3 and SAINST (Bruker, 2016), SHELXTL (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b) and OLEX2 (Dolomanov et al., 2009).

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zigzag bonding pattern usually observed for alkyl chains. Distance restraints were applied to ensure the molecular integrity. Using the SPLIT function, the alkyl chain was duplicated and rotated to align with the remaining electron density. This model was refined isotropically before applying EADP restraints to the atoms and refining anisotropically. This provided a stable refined structure. The water hydrogen atoms were added from the difference map and refined with ideal DFIX restraints in place. C-bound hydrogen atoms were placed geometrically and a riding model applied [C—H = 0.95–0.99 Å; \( U_{	ext{iso}}(H) = 1.2–1.5 U_{eq}(C) \)].

All data underpinning this publication are openly available from the University of Strathclyde KnowledgeBase at https://doi.org/10.15129/39f97ad1-8173-4999-b0b6-41c6ae923fe6.

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Computing details

Data collection: APEX3 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

Tetrakis[µ-2-(2,3-dimethylanilino)benzoato-κ²O:O’]bis[aquacopper(II)]–1-methoxy-2-(2-methoxyethoxy)ethane (1/2)

Crystal data

\[\text{[Cu}_2\text{C}_{15}\text{H}_{14}\text{NO}_2\text{][H}_2\text{O} \cdot 2\text{C}_6\text{H}_{14}\text{O}_3] \]

\[M_r = 1392.54 \]

Monoclinic, \(P_2_1/n\)

\(a = 15.5420 (3) \ \AA\)

\(b = 14.0010 (3) \ \AA\)

\(c = 16.3217 (3) \ \AA\)

\(\beta = 94.791 (1)\)°

\(V = 3539.25 (12) \ \AA^3\)

\(Z = 2\)

\(F(000) = 1468\)

\(D_{\text{c}} = 1.307 \ \text{Mg m}^{-3}\)

Copper \(K\alpha\) radiation, \(\lambda = 1.54178 \ \AA\)

Cell parameters from 9398 reflections

\(\theta = 4.9–68.2°\)

\(\mu = 1.30 \ \text{mm}^{-1}\)

\(T = 105 \ \text{K}\)

Block, clear green

\(0.2 \times 0.15 \times 0.1 \ \text{mm}\)

Data collection

Bruker Photon100 CMOS diffractometer

Radiation source: Incoatec microfocus Cu source

\(\phi\) and \(\omega\) scans

Absorption correction: multi-scan (SADABS; Bruker, 2016)

\(T_{\text{min}} = 0.781, T_{\text{max}} = 0.881\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.033 \)

\(wR(F^2) = 0.089\)

\(S = 1.06\)

6424 reflections

477 parameters

410 restraints

Primary atom site location: dual

Hydrogen site location: mixed

\(H\) atoms treated by a mixture of independent and constrained refinement

\(w = 1/[(\sigma^2(F^2) + (0.0435P)^2 + 2.0003P)]\)

where \(P = (F_o^2 + 2F_c^2)/3\)
$\langle \Delta / \sigma \rangle_{\text{max}} = 0.001$
$\Delta \rho_{\text{max}} = 0.28\ \text{e}\ \text{Å}^{-3}$

Special details

**Experimental.** The X-ray intensities were collected on a Bruker D8 Venture diffractometer using a Photon 100 Detector. The data were reduced using APEX3 and absorption correction applied using SADABS (Bruker, 2016). The crystal structure was solved and refined using SHELXT and SHELXL via the Olex2 refinement package (Dolomanov et al., 2009). Non-hydrogen atom positions were refined anisotropically.

**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.** RIGU restraint applied. Diglyme disorder modelled using DFIX and SADI restraints. The ADPs for both diglyme parts were constrained using EADP constraint.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{Å}^2$)

|      | x (Å)            | y (Å)            | z (Å)            | U(eq)/Å² | Occ. (<1) |
|------|------------------|------------------|------------------|----------|-----------|
| Cu1  | 0.54253 (2)      | 0.42405 (2)      | 0.48032 (2)      | 0.02442 (8) |           |
| O1B  | 0.51049 (8)      | 0.45091 (8)      | 0.36430 (7)      | 0.0329 (3) |           |
| O2B  | 0.43845 (8)      | 0.58088 (8)      | 0.39902 (7)      | 0.0326 (3) |           |
| O1A  | 0.43605 (8)      | 0.35049 (8)      | 0.48736 (7)      | 0.0351 (3) |           |
| O2A  | 0.36511 (7)      | 0.48032 (8)      | 0.52427 (7)      | 0.0331 (3) |           |
| O10S | 0.62229 (8)      | 0.30515 (9)      | 0.46326 (8)      | 0.0376 (3) |           |
| N10B | 0.37240 (10)     | 0.69540 (10)     | 0.28457 (9)      | 0.0362 (3) |           |
| O2S1 | 0.7835 (9)       | 0.2813 (9)       | 0.5321 (12)      | 0.0540 (12) | 0.666 (3) |
| C3B  | 0.46922 (10)     | 0.52671 (11)     | 0.34617 (9)      | 0.0265 (3) |           |
| C9B  | 0.41162 (10)     | 0.63824 (11)     | 0.22997 (9)      | 0.0285 (3) |           |
| C3A  | 0.37072 (10)     | 0.39031 (11)     | 0.51303 (9)      | 0.0284 (3) |           |
| C4A  | 0.29745 (11)     | 0.32888 (12)     | 0.53288 (10)     | 0.0308 (3) |           |
| C4B  | 0.45728 (10)     | 0.55460 (11)     | 0.25802 (9)      | 0.0269 (3) |           |
| O5S1 | 0.73581 (16)     | 0.15001 (18)     | 0.40989 (17)     | 0.0438 (6) | 0.666 (3) |
| C11B | 0.33426 (11)     | 0.78619 (12)     | 0.27016 (10)     | 0.0312 (3) |           |
| C8B  | 0.40932 (11)     | 0.65984 (13)     | 0.14555 (10)     | 0.0345 (4) |           |
| H8B  | 0.39274          | 0.7152           | 0.1250           | 0.041    |           |
| C5B  | 0.49629 (11)     | 0.49735 (13)     | 0.20199 (10)     | 0.0348 (4) |           |
| H5B  | 0.5262           | 0.4414           | 0.2212           | 0.042    |           |
| C5A  | 0.30263 (12)     | 0.23152 (12)     | 0.51507 (10)     | 0.0349 (4) |           |
| H5A  | 0.3501           | 0.2088           | 0.4875           | 0.042    |           |
| N10A | 0.21962 (12)     | 0.45875 (13)     | 0.59054 (14)     | 0.0588 (5) |           |
| C16A | 0.17474 (12)     | 0.58519 (13)     | 0.67658 (11)     | 0.0367 (4) |           |
| O8S1 | 0.5611 (3)       | 0.1715 (4)       | 0.3438 (5)       | 0.0604 (14) | 0.666 (3) |
| C16B | 0.36286 (11)     | 0.86224 (12)     | 0.32109 (10)     | 0.0322 (3) |           |
| C15A | 0.10932 (12)     | 0.64349 (13)     | 0.70386 (11)     | 0.0388 (4) |           |
| C6B  | 0.49276 (13)     | 0.51950 (15)     | 0.11960 (11)     | 0.0447 (4) |           |
| H6B  | 0.5192           | 0.4792           | 0.0821           | 0.054    |           |
| C7B  | 0.44963 (13)     | 0.60229 (15)     | 0.09242 (11)     | 0.0429 (4) |           |
| H7B  | 0.4481           | 0.6193           | 0.0360           | 0.052    |           |
| C17A | 0.26827 (12)     | 0.60465 (15)     | 0.70369 (12)     | 0.0441 (4) |           |
|     | x     | y     | z     | U (10^4) |
|-----|-------|-------|-------|----------|
| H17A| 0.2948| 0.6400| 0.6605| 0.066    |
| H17B| 0.2987| 0.5440| 0.7139| 0.066    |
| H17C| 0.2720| 0.6426| 0.7543| 0.066    |
| C6A | 0.24089(12)| 0.16743(13)| 0.53636(11)| 0.0400(4) |
| H6A | 0.2446| 0.1018| 0.5222| 0.048    |
| C9A | 0.22586(11)| 0.36315(13)| 0.57261(12)| 0.0395(4) |
| C17B| 0.43678(13)| 0.85116(14)| 0.38602(11)| 0.0418(4) |
| H17D| 0.4145| 0.8498| 0.4404| 0.063    |
| H17E| 0.4766| 0.9051| 0.3830| 0.063    |
| H17F| 0.4674| 0.7914| 0.3770| 0.063    |
| C11A| 0.15206(12)| 0.50948(14)| 0.62317(13)| 0.0453(4) |
| C12B| 0.26720(12)| 0.79882(15)| 0.20959(12)| 0.0439(4) |
| H12B| 0.2476| 0.7465| 0.1760| 0.053    |
| C14A| 0.02351(12)| 0.62150(15)| 0.68039(12)| 0.0447(4) |
| H14A| −0.0210| 0.6595| 0.7002| 0.054    |
| C18A| 0.13044(14)| 0.72995(17)| 0.75676(13)| 0.0536(5) |
| H18A| 0.1576| 0.7096| 0.8103| 0.080    |
| H18B| 0.0773| 0.7651| 0.7647| 0.080    |
| H18C| 0.1703| 0.7714| 0.7297| 0.080    |
| C15B| 0.32210(14)| 0.95162(14)| 0.30921(12)| 0.0453(4) |
| C14B| 0.25653(16)| 0.96269(16)| 0.24775(13)| 0.0561(6) |
| H14B| 0.2299| 1.0234| 0.2395| 0.067    |
| C7A | 0.17319(12)| 0.20009(14)| 0.57892(11)| 0.0422(4) |
| H7A | 0.1319| 0.1567| 0.5964| 0.051    |
| C8A | 0.16519(13)| 0.29586(15)| 0.59604(13)| 0.0473(5) |
| H8A | 0.1177| 0.3170| 0.6243| 0.057    |
| C13A| 0.22881(14)| 0.88734(18)| 0.19800(13)| 0.0561(6) |
| H13B| 0.1835| 0.8962| 0.1559| 0.067    |
| C6S1| 0.6846(5)| 0.0737(6)| 0.3762(5)| 0.0517(16)| 0.666(3) |
| H6SA| 0.6530| 0.0435| 0.4196| 0.062    |
| H6SB| 0.7220| 0.0247| 0.3535| 0.062    |
| C13A| 0.00184(13)| 0.54565(16)| 0.62903(15)| 0.0534(5) |
| H13A| −0.0572| 0.5313| 0.6143| 0.064    |
| C12A| 0.06577(13)| 0.49027(16)| 0.59881(16)| 0.0569(6) |
| H12A| 0.0509| 0.4395| 0.5617| 0.068    |
| C3S1| 0.8407(4)| 0.2105(3)| 0.5096(3)| 0.0564(12)| 0.666(3) |
| H3SA| 0.8808| 0.1936| 0.5577| 0.068    |
| H3SB| 0.8753| 0.2356| 0.4661| 0.068    |
| C7S1| 0.6224(2)| 0.1110(2)| 0.3099(2)| 0.0487(8) |
| H7SA| 0.6538| 0.1472| 0.2697| 0.058    |
| H7SB| 0.5922| 0.0570| 0.2808| 0.058    |
| C18B| 0.3508(2)| 1.03494(17)| 0.36428(18)| 0.0787(8) |
| H18D| 0.3400| 1.0200| 0.4212| 0.118    |
| H18E| 0.3182| 1.0922| 0.3463| 0.118    |
| H18F| 0.4126| 1.0464| 0.3609| 0.118    |
| C1S1| 0.8212(6)| 0.3611(4)| 0.5730(4)| 0.0698(15)| 0.666(3) |
| H1SA| 0.8572| 0.3952| 0.5361| 0.105    |
| H1SB| 0.8571| 0.3400| 0.6219| 0.105    |

* indicates that the final parameter is anisotropic.
### Atomic displacement parameters (Å²)

|     | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|-----|------------|------------|------------|------------|------------|------------|
| Cu1 | 0.03002 (14) | 0.02114 (13) | 0.02236 (13) | 0.00405 (8) | 0.00367 (9) | -0.00087 (8) |
| O1B | 0.0443 (6) | 0.0296 (6) | 0.0245 (5) | 0.0110 (5) | 0.0006 (5) | -0.0010 (4) |
| O2B | 0.0447 (7) | 0.0288 (6) | 0.0245 (5) | 0.0108 (5) | 0.0040 (5) | 0.0000 (4) |
| O1A | 0.0371 (6) | 0.0271 (6) | 0.0420 (6) | 0.0001 (5) | 0.0089 (5) | -0.0062 (5) |
| O2A | 0.0346 (6) | 0.0259 (6) | 0.0394 (6) | -0.0001 (5) | 0.0078 (5) | -0.0027 (5) |
| O10S | 0.0403 (7) | 0.0311 (6) | 0.0403 (7) | 0.0141 (5) | -0.0020 (5) | -0.0086 (5) |
| N10B | 0.0532 (9) | 0.0289 (7) | 0.0262 (7) | 0.0110 (6) | 0.0012 (6) | 0.0013 (6) |
| O2S1 | 0.045 (4) | 0.046 (2) | 0.066 (2) | 0.0094 (16) | -0.020 (2) | -0.0047 (19) |
| C3B | 0.0281 (7) | 0.0246 (7) | 0.0268 (7) | -0.0014 (6) | 0.0016 (6) | -0.0018 (6) |
|    |     |     |     |     |     |     |
|----|-----|-----|-----|-----|-----|-----|
| C9B| 0.0307 (8) | 0.0270 (8) | 0.0273 (7) | −0.0023 (6) | −0.0005 (6) | −0.0005 (6) |
| C3A| 0.0351 (8) | 0.0281 (8) | 0.0213 (7) | 0.0004 (6) | −0.0015 (6) | −0.0016 (6) |
| C4A| 0.0350 (8) | 0.0298 (8) | 0.0272 (8) | −0.0032 (6) | 0.0004 (6) | −0.0011 (6) |
| C4B| 0.0295 (7) | 0.0265 (7) | 0.0243 (7) | −0.0008 (6) | 0.0000 (6) | −0.0008 (6) |
| O5S1| 0.0477 (11) | 0.0355 (14) | 0.0483 (16) | 0.0125 (10) | 0.0050 (10) | −0.0008 (10) |
| C11B| 0.0338 (8) | 0.0309 (8) | 0.0296 (8) | 0.0056 (6) | 0.0063 (6) | 0.0038 (6) |
| C8B| 0.0415 (9) | 0.0342 (9) | 0.0269 (8) | 0.0021 (7) | −0.0024 (7) | 0.0022 (7) |
| C5B| 0.0409 (9) | 0.0349 (9) | 0.0281 (8) | 0.0064 (7) | 0.0003 (7) | −0.0022 (7) |
| C5A| 0.0436 (9) | 0.0321 (8) | 0.0397 (9) | −0.0008 (6) | 0.0000 (6) | −0.0008 (6) |
| N10A| 0.0418 (9) | 0.0365 (9) | 0.1029 (16) | −0.0080 (7) | 0.0353 (10) | −0.0175 (9) |
| C16A| 0.0372 (9) | 0.0356 (9) | 0.0461 (11) | 0.0061 (8) | 0.0093 (8) | −0.0007 (9) |
| C11A| 0.0384 (9) | 0.0359 (9) | 0.0645 (12) | −0.0005 (8) | 0.0205 (9) | −0.0007 (9) |
| C12B| 0.0419 (10) | 0.0491 (11) | 0.0396 (10) | 0.0082 (8) | −0.0038 (8) | −0.0009 (8) |
| C14A| 0.0396 (10) | 0.0490 (11) | 0.0474 (10) | 0.0120 (8) | 0.0141 (8) | 0.0103 (9) |
| C18A| 0.0523 (12) | 0.0632 (13) | 0.0456 (11) | 0.0199 (10) | 0.0059 (9) | −0.0103 (10) |
| C15B| 0.0596 (12) | 0.0338 (9) | 0.0444 (10) | 0.0148 (8) | 0.0160 (9) | 0.0014 (8) |
| C14B| 0.0713 (14) | 0.0515 (12) | 0.0467 (11) | 0.0362 (11) | 0.0115 (10) | 0.0074 (9) |
| C7A| 0.0441 (10) | 0.0414 (10) | 0.0403 (10) | −0.0143 (8) | −0.0004 (8) | 0.0007 (8) |
| C8A| 0.0400 (10) | 0.0432 (10) | 0.0604 (12) | −0.0096 (8) | 0.0142 (9) | −0.0069 (9) |
| C13B| 0.0518 (12) | 0.0723 (14) | 0.0433 (11) | 0.0313 (11) | −0.0020 (9) | 0.0054 (10) |
| C6S1| 0.071 (2) | 0.033 (2) | 0.051 (3) | 0.0145 (16) | 0.006 (2) | −0.0009 (9) |
| C13A| 0.0347 (10) | 0.0564 (13) | 0.0707 (14) | −0.0004 (9) | 0.0128 (9) | 0.0044 (11) |
| C12A| 0.0415 (10) | 0.0480 (12) | 0.0837 (16) | −0.0075 (9) | 0.0195 (10) | −0.0120 (11) |
| C3S1| 0.0391 (17) | 0.053 (3) | 0.075 (3) | 0.0136 (18) | −0.0069 (19) | 0.0068 (19) |
| C7S1| 0.067 (2) | 0.0370 (16) | 0.042 (2) | 0.0056 (13) | 0.0058 (14) | −0.0119 (14) |
| C18B| 0.123 (2) | 0.0385 (12) | 0.0743 (17) | 0.0224 (14) | 0.0046 (16) | −0.0143 (11) |
| C1S1| 0.0702 (18) | 0.057 (3) | 0.076 (4) | 0.001 (3) | −0.032 (3) | −0.005 (2) |
| C4S1| 0.058 (2) | 0.041 (2) | 0.067 (3) | 0.0176 (15) | −0.0042 (19) | 0.0105 (18) |
| C9S2| 0.057 (2) | 0.0484 (16) | 0.059 (4) | 0.0007 (19) | −0.001 (2) | −0.009 (3) |
| O2S2| 0.045 (4) | 0.046 (2) | 0.066 (2) | 0.0094 (16) | −0.020 (2) | −0.0047 (19) |
| C3S2| 0.0391 (17) | 0.053 (3) | 0.075 (3) | 0.0136 (18) | −0.0069 (19) | 0.0068 (19) |
| C1S2| 0.0702 (18) | 0.057 (3) | 0.076 (4) | 0.001 (3) | −0.032 (3) | −0.005 (2) |
| C4S2| 0.058 (2) | 0.041 (2) | 0.067 (3) | 0.0176 (15) | −0.0042 (19) | 0.0105 (18) |
| O5S2| 0.0477 (11) | 0.0355 (14) | 0.0483 (16) | 0.0125 (10) | 0.0050 (10) | −0.0008 (10) |
| C6S2| 0.071 (2) | 0.033 (2) | 0.051 (3) | 0.0145 (16) | 0.006 (2) | −0.0009 (2) |
| C7S2| 0.067 (2) | 0.0370 (16) | 0.042 (2) | 0.0056 (13) | 0.0058 (14) | −0.0119 (14) |
| O8S2| 0.057 (2) | 0.045 (2) | 0.075 (2) | 0.0121 (18) | −0.018 (2) | −0.0332 (19) |
| C9S1| 0.057 (2) | 0.0484 (16) | 0.059 (4) | 0.0007 (19) | −0.001 (2) | −0.009 (3) |
### Geometric parameters (Å, °)

| Bond/Distance                  | Value (Å, °)       |
|--------------------------------|-------------------|
| Cu1—Cu1i                      | 2.6126 (4)        |
| Cu1—O1B                       | 1.9539 (11)       |
| Cu1—O2B'                      | 1.9682 (11)       |
| Cu1—O1A                       | 1.9608 (12)       |
| Cu1—O2A'                      | 1.9689 (11)       |
| Cu1—O10S                      | 2.1078 (11)       |
| O1B—C3B                       | 1.2624 (19)       |
| O2B—Cu1i                      | 1.9682 (11)       |
| O2B—C3B                       | 1.2715 (19)       |
| O1A—C3A                       | 1.260 (2)         |
| O2A—Cu1i                      | 1.9690 (11)       |
| O2A—C3A                       | 1.278 (2)         |
| O10S—H10A                     | 0.956 (3)         |
| O10S—H10B                     | 0.956 (3)         |
| N10B—C9B                      | 1.377 (2)         |
| N10B—C11B                     | 1.414 (2)         |
| N10B—H10C                     | 0.88 (2)          |
| O2S1—C3S1                     | 1.401 (10)        |
| O2S1—C1S1                     | 1.404 (10)        |
| C3B—C4B                       | 1.487 (2)         |
| C4B—C5B                       | 1.392 (2)         |
| C3A—C4A                       | 1.484 (2)         |
| C4A—C5A                       | 1.397 (2)         |
| C4A—C9A                       | 1.418 (2)         |
| C4B—C5B                       | 1.392 (2)         |
| O5S1—C6S1                     | 1.416 (8)         |
| O5S1—C4S1                     | 1.428 (5)         |
| C11B—C16B                     | 1.400 (2)         |
| C11B—C12B                     | 1.387 (2)         |
| C8B—H8B                       | 0.9500            |
| C8B—C7B                       | 1.373 (3)         |
| C5B—H5B                       | 0.9500            |
| C5B—C6B                       | 1.377 (2)         |
| C5A—H5A                       | 0.9500            |
| C5A—C6A                       | 1.379 (3)         |
| N10A—C9A                      | 1.375 (2)         |
| N10A—C11A                     | 1.409 (2)         |
| N10A—H10D                     | 0.86 (2)          |
| C16A—C15A                     | 1.405 (2)         |
| C16A—C17A                     | 1.509 (3)         |
| C16A—C11A                     | 1.399 (3)         |
| O8S1—C7S1                     | 1.421 (7)         |
| O8S1—C9S1                     | 1.383 (8)         |
| C16B—C17B                     | 1.505 (2)         |
| C16B—C15B                     | 1.409 (2)         |
## Supporting Information

| Bond / Angle / Distance | Value (with errors) | Description |
|-------------------------|---------------------|-------------|
| C15A—C14A              | 1.391 (3)           | C1S2—H1SF  | 0.9800 |
| C15A—C18A              | 1.507 (3)           | C4S2—H4SC  | 0.9900 |
| C6B—H6B                | 0.9500              | C4S2—H4SD  | 0.9900 |
| C6B—C7B                | 1.393 (3)           | C4S2—O5S2  | 1.397 (12)|
| C7B—H7B                | 0.9500              | O5S2—C6S2  | 1.407 (17)|
| C17A—H17A              | 0.9800              | C6S2—H6SC  | 0.9900 |
| C17A—H17B              | 0.9800              | C6S2—H6SD  | 0.9900 |
| C17A—H17C              | 0.9800              | C6S2—C7S2  | 1.471 (17)|
| C6A—H6A                | 0.9500              | C7S2—H7SC  | 0.9900 |
| C6A—C7A                | 1.388 (3)           | C7S2—H7SD  | 0.9900 |
| C9A—C8A                | 1.408 (3)           | C7S2—O8S2  | 1.426 (14)|
| C17B—H17D              | 0.9800              | C9S1—H9SA  | 0.9800 |
| C17B—H17E              | 0.9800              | C9S1—H9SB  | 0.9800 |
| C17B—H17F              | 0.9800              | C9S1—H9SC  | 0.9800 |
| O1B—Cu1—Cu1i           | 89.34 (3)           | C9B—N10B—C11B | 128.08 (14)| 109.5 |
| O1B—Cu1—O2B            | 168.92 (5)          | C9B—N10B—C11B | 128.08 (14)| 109.5 |
| O1B—Cu1—O1A            | 90.57 (5)           | C9B—N10B—C11B | 128.08 (14)| 119.6 |
| O1B—Cu1—O2A            | 87.70 (5)           | C9B—N10B—C11B | 128.08 (14)| 119.6 |
| O1B—Cu1—O10S           | 97.42 (5)           | C9B—N10B—C11B | 128.08 (14)| 120.5 |
| O2B—Cu1—Cu1i           | 79.58 (3)           | C9B—N10B—C11B | 128.08 (14)| 119.4 |
| O2B—Cu1—O2A            | 90.74 (5)           | C9B—N10B—C11B | 128.08 (14)| 121.2 |
| O2B—Cu1—O10S           | 93.65 (5)           | C9B—N10B—C11B | 128.08 (14)| 119.4 |
| O1A—Cu1—Cu1i           | 88.07 (3)           | C9B—N10B—C11B | 128.08 (14)| 119.6 |
| O1A—Cu1—O2B            | 88.85 (5)           | C9B—N10B—C11B | 128.08 (14)| 120.6 |
| O1A—Cu1—O2A            | 168.82 (5)          | C9B—N10B—C11B | 128.08 (14)| 119.6 |
| O1A—Cu1—O10S           | 95.82 (5)           | C9B—N10B—C11B | 128.08 (14)| 119.3 |
| O2A—Cu1—Cu1i           | 80.87 (3)           | C9B—N10B—C11B | 128.08 (14)| 121.4 |
| O2A—Cu1—O10S           | 95.36 (5)           | C9B—N10B—C11B | 128.08 (14)| 119.3 |
| O10S—Cu1—Cu1i          | 172.15 (4)          | C9B—N10B—C11B | 128.08 (14)| 120.1 |
| C3B—O1B—Cu1            | 118.18 (10)         | C9B—N10B—C11B | 128.08 (14)| 119.7 |
| C3B—O2B—Cu1            | 128.64 (10)         | C9B—N10B—C11B | 128.08 (14)| 120.1 |
| C3A—O1A—Cu1            | 119.63 (10)         | C9B—N10B—C11B | 128.08 (14)| 119.8 |
| C3A—O2A—Cu1            | 127.34 (10)         | C9B—N10B—C11B | 128.08 (14)| 119.8 |
| Cu1—O10S—H10A          | 128.2 (12)          | C9B—N10B—C11B | 128.08 (14)| 109.8 |
| Cu1—O10S—H10B          | 126.1 (13)          | C9B—N10B—C11B | 128.08 (14)| 108.3 |
| H10A—O10S—H10B         | 104.9 (7)           | C9B—N10B—C11B | 128.08 (14)| 109.8 |
| C9B—N10B—C11B          | 128.08 (14)         | C9B—N10B—C11B | 128.08 (14)| 109.8 |
| C9B—N10B—H10C          | 113.6 (14)          | C9B—N10B—C11B | 128.08 (14)| 119.9 |
| C11B—N10B—H10C         | 116.1 (15)          | C9B—N10B—C11B | 128.08 (14)| 120.1 |
| C3S1—O2S1—C1S1         | 116.0 (10)          | C9B—N10B—C11B | 128.08 (14)| 119.9 |
| O1B—C3B—O2B            | 123.71 (14)         | C9B—N10B—C11B | 128.08 (14)| 120.2 |
| O1B—C3B—C4B            | 117.85 (13)         | C9B—N10B—C11B | 128.08 (14)| 119.6 |
| O2B—C3B—C4B            | 118.43 (13)         | C9B—N10B—C11B | 128.08 (14)| 120.2 |
| N10B—C9B—C4B           | 120.34 (14)         | C9B—N10B—C11B | 128.08 (14)| 119.6 |
| N10B—C9B—C8B           | 122.13 (15)         | C9B—N10B—C11B | 128.08 (14)| 120.4 |
| C8B—C9B—C4B            | 117.53 (15)         | C9B—N10B—C11B | 128.08 (14)| 119.6 |
| O1A—C3A—O2A            | 123.41 (15)         | C9B—N10B—C11B | 128.08 (14)| 120.4 |

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| Bond                  | Angle (°) (RMS) | Bond                  | Angle (°) (RMS) |
|----------------------|-----------------|----------------------|-----------------|
| O1A—C3A—C4A         | 118.05 (14)     | C4S1—C3S1—H3SA      | 109.4           |
| O2A—C3A—C4A         | 118.53 (14)     | C4S1—C3S1—H3SB      | 109.4           |
| C5A—C4A—C3A         | 117.47 (15)     | O8S1—C7S1—C6S1      | 110.1 (5)       |
| C5A—C4A—C9A         | 119.10 (16)     | O8S1—C7S1—H7SA      | 109.6           |
| C9B—C4B—C3B         | 123.33 (15)     | O8S1—C7S1—H7SB      | 109.6           |
| C5B—C4B—C3B         | 117.28 (14)     | C6S1—C7S1—H7SB      | 109.6           |
| C5B—C4B—C9B         | 119.64 (14)     | H7SA—C7S1—H7SB      | 108.1           |
| C6S1—O5S1—C4S1      | 113.8 (4)       | C15B—C18B—H18D      | 109.5           |
| C16B—C11B—N10B      | 118.49 (15)     | C15B—C18B—H18E      | 109.5           |
| C12B—C11B—N10B      | 120.86 (16)     | C15B—C18B—H18F      | 109.5           |
| C12B—C11B—C16B      | 120.58 (16)     | H18D—C18B—H18E      | 109.5           |
| C9B—C8B—H8B         | 119.5           | H18D—C18B—H18F      | 109.5           |
| C7B—C8B—C9B         | 121.08 (16)     | O2S1—C1S1—H1SA      | 109.5           |
| C7B—C8B—H8B         | 119.5           | C3S1—C4S1—H3SA      | 109.5           |
| C6B—C5B—H5B         | 121.86 (16)     | C3S1—C4S1—H3SB      | 109.5           |
| C6B—C5B—C4B         | 119.1           | C3S1—C4S1—H3SC      | 109.5           |
| C4A—C5A—H5A         | 119.0           | C3S1—C4S1—H3SD      | 109.5           |
| C6A—C5A—C4A         | 121.92 (17)     | O5S1—C4S1—C3S1      | 108.0 (4)       |
| C6A—C5A—H5A         | 119.0           | O5S1—C4S1—C3S2      | 112.4 (15)      |
| C9A—N10A—C11A       | 129.73 (17)     | O2S2—C3S2—C4S2      | 112.4 (15)      |
| C9A—N10A—H10D       | 111.9 (16)      | O2S2—C3S2—H3SC      | 109.1           |
| C11A—N10A—H10D      | 118.3 (16)      | H3SC—C3S2—H3SD      | 107.9           |
| C15A—C16A—C17A      | 120.49 (17)     | C3S2—C4S2—H4SC      | 109.5           |
| C11A—C16A—C15A      | 119.10 (17)     | C3S2—C4S2—H4SD      | 109.5           |
| C11A—C16A—C17A      | 120.41 (16)     | H9SD—C9S2—H9SE      | 109.5           |
| C9S1—O8S1—C7S1      | 112.6 (8)       | H9SD—C9S2—H9SF      | 109.5           |
| C11B—C16B—C17B      | 121.71 (15)     | H9SE—C9S2—H9SF      | 109.5           |
| C11B—C16B—C15B      | 118.49 (16)     | O8S2—C9S2—H9SD      | 109.5           |
| C15B—C16B—C17B      | 119.78 (17)     | O8S2—C9S2—H9SE      | 109.5           |
| C16A—C15A—C18A      | 121.28 (17)     | O8S2—C9S2—H9SF      | 109.5           |
| C14A—C15A—C16A      | 119.14 (18)     | C3S2—O2S2—C1S2      | 121 (2)         |
| C14A—C15A—C18A      | 119.57 (17)     | O2S2—C3S2—H3SC      | 109.1           |
| C5B—C6B—H6B         | 120.7           | O2S2—C3S2—H3SD      | 109.1           |
| C5B—C6B—C7B         | 118.52 (17)     | O2S2—C3S2—C4S2      | 112.4 (15)      |
| C7B—C6B—H6B         | 120.7           | H3SC—C3S2—H3SD      | 107.9           |
| C8B—C7B—C6B         | 121.35 (16)     | C4S2—C3S2—H3SC      | 109.1           |
| C8B—C7B—H7B         | 119.3           | C4S2—C3S2—H3SD      | 109.1           |
| C6B—C7B—H7B         | 119.3           | O2S2—C1S2—H1SD      | 109.5           |
| C16A—C17A—H17A      | 109.5           | O2S2—C1S2—H1SE      | 109.5           |
| C16A—C17A—H17B      | 109.5           | O2S2—C1S2—H1SF      | 109.5           |
| C16A—C17A—H17C      | 109.5           | H1SD—C1S2—H1SE      | 109.5           |
| H17A—C17A—H17B      | 109.5           | H1SD—C1S2—H1SF      | 109.5           |
| H17A—C17A—H17C      | 109.5           | C3S2—C4S2—H4SC      | 109.5           |
| H17B—C17A—H17C      | 109.5           | C3S2—C4S2—H4SD      | 109.5           |
| C5A—C6A—H6A         | 120.6           | C3S2—C4S2—H4SD      | 109.5           |
| C5A—C6A—C7A         | 118.74 (17)     | H4SC—C4S2—H4SD      | 108.1           |
| Bond | Angle (°) | Bond | Angle (°) |
|------|----------|------|----------|
| C7A—C6A—H6A | 120.6 | O5S2—C4S2—C3S2 | 110.6 (9) |
| N10A—C9A—C4A | 119.92 (16) | O5S2—C4S2—H4SC | 109.5 |
| N10A—C9A—C8A | 122.18 (17) | O5S2—C4S2—H4SD | 109.5 |
| C8A—C9A—C4A | 117.82 (17) | O5S2—C6S2—C7S2 | 111.7 (14) |
| C16B—C17B—H17D | 109.5 | O5S2—C6S2—H6SC | 109.3 |
| C16B—C17B—H17E | 109.5 | O5S2—C6S2—H6SD | 109.3 |
| C16B—C17B—H17F | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |
| H17D—C17B—H17E | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |
| H17D—C17B—H17F | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |
| H17E—C17B—H17F | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |
| C16A—C11A—N10A | 117.43 (17) | C4S2—O5S2—C6S2 | 114.8 (10) |
| C12A—C11A—N10A | 121.68 (19) | C4S2—O5S2—C6S2 | 114.8 (10) |
| C12A—C11A—C16A | 120.71 (18) | C4S2—O5S2—C6S2 | 114.8 (10) |
| C11B—C12B—H12B | 119.9 | C4S2—O5S2—C6S2 | 114.8 (10) |
| C13B—C12B—C11B | 120.15 (19) | C4S2—O5S2—C6S2 | 114.8 (10) |
| C13B—C12B—H12B | 119.9 | C4S2—O5S2—C6S2 | 114.8 (10) |
| C15A—C14A—H14A | 119.4 | C4S2—O5S2—C6S2 | 114.8 (10) |
| C13A—C14A—C15A | 121.22 (18) | C4S2—O5S2—C6S2 | 114.8 (10) |
| C13A—C14A—H14A | 119.4 | C4S2—O5S2—C6S2 | 114.8 (10) |
| C15A—C18A—H18A | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |
| C15A—C18A—H18B | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |
| C15A—C18A—H18C | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |
| H18A—C18A—H18B | 109.5 | C4S2—O5S2—C6S2 | 114.8 (10) |

Cu1—O1B—C3B—O2B | −8.6 (2) | C5B—C6B—C7B—C8B | −1.7 (3) |
Cu1—O1B—C3B—C4B | 170.41 (10) | C5A—C4A—C9A—N10A | −179.92 (18) |
Cu1—O2B—C3B—O1B | 9.2 (2) | C5A—C4A—C9A—C8A | 3.2 (3) |
Cu1—O2B—C3B—C4B | −169.80 (10) | C5A—C6A—C7A—C8A | 3.2 (3) |
Cu1—O1A—C3A—O2A | 10.2 (2) | N10A—C9A—C8A—C7A | −178.9 (2) |
Cu1—O1A—C3A—O4A | −168.37 (10) | N10A—C9A—C8A—C7A | −178.9 (2) |
Cu1—O2A—C3A—O1A | −8.7 (2) | C16A—C15A—C14A—C13A | −2.0 (3) |
Cu1—O2A—C3A—C4A | 169.93 (10) | C16A—C15A—C14A—C13A | −2.0 (3) |
O1B—C3B—C4B—C9B | 179.72 (14) | C16B—C15B—C14B—C13B | 1.0 (3) |
O1B—C3B—C4B—C5B | −3.1 (2) | C16B—C15B—C14B—C13B | 1.0 (3) |
O2B—C3B—C4B—C9B | −1.2 (2) | C15A—C16A—C11A—N10A | 173.66 (18) |
O2B—C3B—C4B—C5B | 176.01 (15) | C15A—C16A—C11A—N10A | 173.66 (18) |
O1A—C3A—C4A—C5A | −5.4 (2) | C15A—C14A—C13A—C12A | −0.8 (3) |
O1A—C3A—C4A—C9A | 171.06 (16) | C17A—C16A—C15A—C14A | −177.50 (17) |
O2A—C3A—C4A—C5A | 175.93 (14) | C17A—C16A—C15A—C14A | −177.50 (17) |
O2A—C3A—C4A—C9A | −7.6 (2) | C17A—C16A—C15A—C14A | −177.50 (17) |
N10B—C9B—C4B—C3B | −3.1 (2) | C17A—C16A—C15A—C14A | −177.50 (17) |
N10B—C9B—C4B—C5B | 179.74 (15) | C6A—C7A—C8A—C9A | −1.2 (3) |
N10B—C9B—C8B—C7B | 179.34 (15) | C6A—C7A—C8A—C9A | −1.2 (3) |
N10B—C11B—C16B—C17B | 4.4 (2) | C9A—C10A—C11A—C12A | 144.7 (2) |
N10B—C11B—C16B—C15B | −177.08 (16) | C9A—C10A—C11A—C12A | 144.7 (2) |
N10B—C11B—C12B—C13B | 177.97 (18) | C17B—C16B—C15B—C14B | 177.61 (19) |
O2S1—C3S1—C4S1—O5S1 | 58.7 (11) | C17B—C16B—C15B—C14B | 177.61 (19) |
C3B—C4B—C5B—C6B | −176.76 (17) | C11A—C10A—C9A—C4A | 174.3 (2) |
### Hydrogen-bond geometry (Å, °)

| D—H···A      | D—H  | H···A  | D···A  | D—H···A |
|--------------|-------|--------|--------|---------|
| N10A—H10D···O2A | 0.86 (3) | 1.86 (3) | 2.604 (2) | 143 (2) |
| N10B—H10C···O2B | 0.89 (2) | 1.87 (2) | 2.6065 (18) | 139 (2) |

Symmetry code: (i) −x+1, −y+1, −z+1.

#### C9B—N10B—C11B—C16B

-124.92 (18) C11A—N10A—C9A—C8A  -8.9 (4)

#### C9B—N10B—C11B—C12B

58.1 (3) C11A—C16A—C15A—C14A  3.2 (3)

#### C9B—C4B—C5B—C6B

0.5 (3) C11A—C16A—C15A—C18A  -175.80 (18)

#### C9B—C8B—C7B—C6B

1.3 (3) C12B—C11B—C16B—C17B  -178.61 (17)

#### C3A—C4A—C5A—C6A

175.39 (15) C12B—C11B—C16B—C15B  -0.1 (3)

#### C3A—C4A—C9A—N10A

3.7 (3) C14A—C13A—C12A—C11A  2.4 (4)

#### C3A—C4A—C9A—C8A

-173.24 (17) C18A—C15A—C14A—C13A  177.00 (19)

#### C4A—C5A—C6A—C7A

-2.0 (3) C15B—C14B—C13B—C12B  0.0 (4)

#### C4A—C9A—C8A—C7A

-2.0 (3) C6S1—O5S1—C4S1—C3S1  -178.1 (5)

#### C4B—C9B—C8B—C7B

0.0 (3) C18B—C15B—C14B—C13B  -179.0 (2)

#### C4B—C5B—C6B—C7B

0.8 (3) C1S1—O2S1—C3S1—C4S1  171.1 (12)

#### O5S1—C6S1—C7S1—O8S1

-67.3 (7) C4S1—O5S1—C6S1—C7S1  175.2 (4)

#### C11B—N10B—C9B—C4B

171.23 (16) O2S2—C3S2—C4S2—O5S2  -64 (2)

#### C11B—N10B—C9B—C8B

-8.1 (3) C3S2—C4S2—O5S2—C6S2  176.1 (11)

#### C11B—C16B—C15B—C14B

-0.9 (3) C1S2—O2S2—C3S2—C4S2  -163 (3)

#### C11B—C16B—C15B—C18B

179.1 (2) C4S2—O5S2—C6S2—C7S2  -164.7 (10)

#### C11B—C12B—C13B—C14B

-1.0 (3) O5S2—C6S2—C7S2—O8S2  68.1 (17)

#### C8B—C9B—C4B—C3B

176.22 (15) C6S2—C7S2—O8S2—C9S2  171.6 (16)

#### C8B—C9B—C4B—C5B

-0.9 (2) C9S1—O8S1—C7S1—C6S1  172.0 (6)