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# *catena*-Poly[[[tetrakis(4-methylpyridine-$\kappa N$)copper(II)]-$\mu$-sulfato-$\kappa^2 O$:$O'$] 4.393-hydrate]

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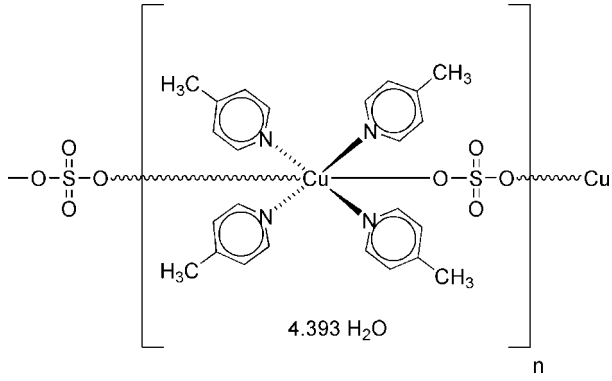
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.005 Å; disorder in solvent or counterion; $R$ factor = 0.046; $wR$ factor = 0.118; data-to-parameter ratio = 19.3.

The structure of the title compound, $\{[Cu(SO_4)(C_6H_7N)_4]\cdot 4.393H_2O\}_n$, consists of $Cu^{2+}$ ions surrounded in a square-planar fashion by 4-methylpyridine ligands, forming two crystallographically independent $Cu\{H_3C(C_5H_4N)\}_4$ units that are both located on crystallographic inversion centers. The Cu(4-methylpyridine)$_4$ units are, in turn, connected with each other *via* bridging sulfate anions, leading to the formation of infinite $[Cu\{H_3C(C_5H_4N)\}_4SO_4]_n$ zigzag chains along [001]. The completed coordination spheres of the $Cu^{2+}$ ions are slightly distorted octahedral. The axial Cu—O bonds are elongated [average length = 2.42 (4) Å] compared to the equatorial Cu—N bonds [average length = 2.043 (2) Å]. The interstitial space between the chains is filled with uncoordinated water molecules that consolidate the structure through O—H···O hydrogen bonding. One of the five crystallographically independent solvent water molecules is partially occupied with an occupancy factor of 0.396 (4). Due to hydrogen bonding between symmetry-equivalent water molecules across inversion centers, several of the water H atoms are disordered in 1:1 ratios over mutually exclusive positions. The crystal under investigation was found to be non-merohedrally twinned in a 0.789 (1):0.211 (1) ratio by a 180° rotation around the reciprocal $b$ axis.

## Related literature

For the structures of related binuclear copper(II) complexes, see: Shahid *et al.* (2008, 2009).




## Experimental

### Crystal data

$[Cu(SO_4)(C_6H_7N)_4]\cdot 4.393H_2O$
$M_r$ = 611.27
Triclinic, $P\overline{1}$
$a$ = 10.4688 (12) Å
$b$ = 11.6327 (14) Å
$c$ = 12.8300 (15) Å
$\alpha$ = 78.672 (3)°
$\beta$ = 87.609 (3)°
$\gamma$ = 67.571 (3)°
$V$ = 1415.2 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.90 mm$^{-1}$
$T$ = 100 K
0.60 × 0.45 × 0.40 mm

### Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*TWINABS*; Bruker, 2008)
$T_{min}$ = 0.607, $T_{max}$ = 0.746
16814 measured reflections
6963 independent reflections
6170 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.118
$S$ = 1.06
6963 reflections
361 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.61 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H5$A$···O6$^{i}$ | 0.85 | 2.04 | 2.887 (4) | 173 |
| O5—H5$B$···O4$^{ii}$ | 0.85 | 2.01 | 2.843 (4) | 168 |
| O6—H6$A$···O6$^{i}$ | 0.84 | 2.34 | 2.983 (6) | 133 |
| O6—H6$B$···O4$^{ii}$ | 0.85 | 1.92 | 2.762 (4) | 172 |
| O6—H6$C$···O8 | 0.85 | 1.98 | 2.804 (5) | 163 |
| O7—H7$A$···O5 | 0.84 | 2.16 | 2.906 (4) | 148 |
| O7—H7$B$···O3$^{iii}$ | 0.85 | 2.13 | 2.923 (4) | 157 |
| O8—H8$A$···O3$^{ii}$ | 0.84 | 2.42 | 2.788 (4) | 107 |
| O8—H8$B$···O6 | 0.85 | 2.04 | 2.804 (5) | 149 |
| O8—H8$C$···O8$^{iv}$ | 0.85 | 1.87 | 2.710 (7) | 176 |
| O9—H9$A$···O6 | 0.84 | 2.48 | 3.210 (7) | 145 |
| O9—H9$B$···O3$^{ii}$ | 0.84 | 2.22 | 3.063 (7) | 175 |
| O9—H9$B$···O4$^{ii}$ | 0.84 | 2.71 | 3.275 (7) | 126 |

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2009); data reduction: *CELL NOW* (Bruker, 2009) and *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2458).

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# supporting information

*Acta Cryst.* (2011). E**67**, m375–m376 [doi:10.1107/S1600536811006325]

## *catena*-Poly[[[tetrakis(4-methylpyridine-$\kappa N$)copper(II)]-$\mu$-sulfato-$\kappa^2 O:O'$] 4.393-hydrate]

**Naveed Alam, Muhammad Shahid, Muhammad Mazhar, Saad Al-Jassabi, Matthias Zeller and Allen D. Hunter**

### S1. Comment

In relation to our previous work on the structural chemistry of copper complexes (Shahid *et al.*, 2008; 2009) the title compound was prepared as the unintended product of the reaction of $CuSO_4{\cdot}5H_2O$ with pyrolidine in acetone and 4-methylpyridine.

In the title structure (Fig. 1), two crystallographically independent $Cu^{2+}$ ions are each located on an inversion center. Their coordination environments are distorted octahedral, with a $CuN_4O_2$ set of ligating atoms, composed of four N atoms from four 4-methylpyridine groups and two O atoms of two sulfate anions. The equatorial plane is made up of four N atoms of the 4-methylpyridine ligands, N1, N1$^{i}$, N2 and N2$^{i}$ for Cu1 and N3, N3$^{ii}$, N4 and N4$^{ii}$ for Cu2 (symmetry operators (i) -*x* + 1, -*y* + 2, -*z* + 1; (ii) -*x* + 1, -*y* + 2, -*z*.), with distances ranging between 2.041 (2) and 2.046 (2) Å. The O atoms O1, O1$^{i}$, O2 and O2$^{ii}$ of the sulfate anion are bonded at the axial positions of Cu1 and Cu2, respectively, resulting in considerably longer Cu—O bonds of 2.393 (2) Å for Cu1—O1 and 2.443 (2) Å for Cu2—O2.

The bridging sulfate ions connect the Cu(4-methylpyridine)$_4$ units to form infinite $[Cu\{H_3C(C_5H_4N)\}_4SO_4]_n$ zigzag chains along the [001] direction of the crystal (Fig. 2). The shape of the zigzag chain follows the coordination geometry of the copper and sulfate ions: Cu—O—S angles are close to 180° (169.62 (13) for S1—O1—Cu1 and 172.99 (1) for S1—O2—Cu2), the O—Cu—O angles are exactly 180° (due to the location of the copper ions on inversion centers). The angles of the zigzag chain, represented by the Cu—S—Cu angles, thus follow closely the tetrahedral sulfate O—S—O angles and are 111.73 ° (the O—S—O angles range between 108.76 (12) and 110.04 (13)°).

Parallel zigzag chains interdigitate as shown in Fig. 3, but interstitial space is left between neighboring molecules. Along the *a*-axis neighboring sulfate ions are connected through O—H···O hydrogen bonds mediated by the water molecules O5 and O7 to form infinite $\{O\cdots H_2O\cdots H_2O\cdots O\cdots H_2O\cdots H_2O\cdots\}_n$ chains. Parallel pairs of these chains are connected with each other through additional O—H···O hydrogen bonds mediated by the water molecules of O6 and O8. Due to hydrogen bonding between symmetry equivalent water molecules across inversion centers, the H atoms of O6 and O8 are partially disordered over mutually exclusive positions (see refinement section for details). The connection of the sulfate ions with these water molecules creates flat strands made up of slightly wobbly single molecule layers of tightly hydrogen-bonded water and sulfate molecules about seven to eight Å wide that stretch infinitely parallel to the *a*-axis (Figs. 3 and 4). The last of the five crystallographically independent water solvate molecules (O9) is not part of these strands but is located about three Å away and is only weakly hydrogen bonded with the other water molecules (Fig. 4). It is only partially occupied with a refined occupancy factor of 0.396 (4). For details and numerical values of the hydrogen bonding geometries, including symmetry operators, see: Table 1.

## S2. Experimental

$CuSO_4{\cdot}5H_2O$ (0.30 g, 1.21 mmol) was added directly to a stirred solution of pyrolidine (0.5 g, 2.43 mmole) in 20 ml acetone. The contents were stirred until complete dissolution of the salt to which about 30 ml of 4-methylpyridine was added and stirring was continued for another hour. Insoluble matter was removed by filtration and slow evaporation of the reaction mixture at room temperature gave the title compound as blue crystals after three weeks. Yield 60% (0.44 g), m.p. 373 K. Elemental analysis: calculated (found): C 47.15(47.66), H 6.06(5.92), N 9.20(8.95)%.

## S3. Refinement

The crystal under investigation was found to be non-merohedrally twinned by a 180° rotation around the reciprocal *b*-axis. The orientation matrices for the two components were identified using the program *CELL NOW* (Bruker, 2008), and the two components were integrated using *SAINT*, resulting in a total of 23387 reflections. 6633 reflections (4611 unique ones) involved component 1 only (mean I/$\sigma$ = 12.2), 6573 reflections (3653 unique ones) involved component 2 only (mean I/$\sigma$ = 5.8), and 10181 reflections (5801 unique ones) involved both components (mean I/$\sigma$ = 9.4). The exact twin matrix identified by the integration program was found to be -1.00018 0.00039 0.00025, -0.83074 0.99991 - 0.32879, 0.00016 - 0.00153 - 0.99973. The structure was solved using direct methods with only the non-overlapping reflections of component 1. The structure was refined using the HKLF5 routine with all reflections of component 1 (including the overlapping ones) below a *d*-spacing threshold of 0.75 Å, resulting in a BASF value of 0.211 (1). The $R_{int}$ value given is for all reflections before the cutoff at d = 0.75 Å and is based on agreement between observed single and composite intensities and those calculated from refined unique intensities and twin fractions (*TWINABS*; Bruker, 2008).

Hydrogen atoms of the water molecules are partially disordered over mutually exclusive positions due to hydrogen bonding between symmetry equivalent water molecules across inversion centers. The H atoms in question are H6A and H8C, which are each located close to a crystallographic inversion center between pairs of symmetry equivalent atoms of O6 and O8. Both H atoms were thus refined as 50% occupied. For both water molecules O6 and O8 a second half occupied hydrogen atom is located in a position in which it hydrogen-bonds with the a neighboring water molecule of O8 and O6, respectively, thus again creating a pair of close by half occupied H atoms (H6C and H8B) in mutually exclusive positions. The water solvate molecule of O9 is only partially occupied with a refined occupancy factor of 0.396 (4). Water hydrogen atoms were located in difference density Fourier maps, assigned occupancies as described above and their positions were refined with an O—H distance of 0.84 (1) Å and H···H distances of 1.30 (1) Å. In the final refinement cycles the water H atoms were set to ride on their carrying oxygen atoms. All other hydrogen atoms were immediately placed in calculated positions and all H atoms were refined with an isotropic displacement parameter $U_{iso}$ of 1.5 (methyl, hydroxyl) or 1.2 times (aromatic) that of $U_{eq}$ of the adjacent carbon or oxygen atom.

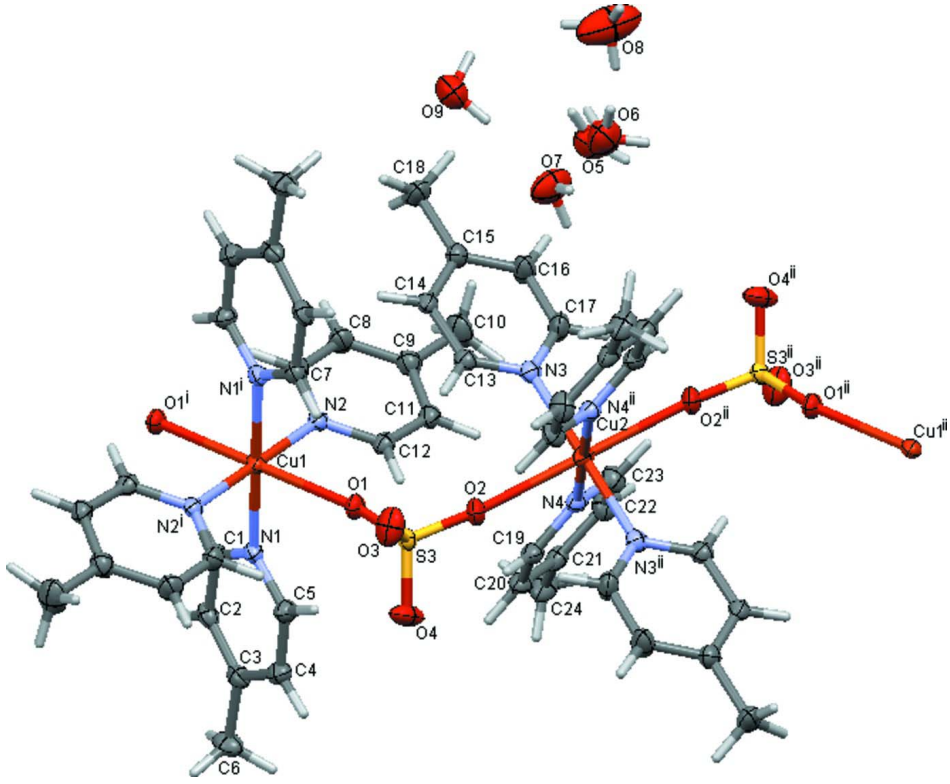


**Figure 1**

View of the molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level with atom labeling. Symmetry transformations used to generate equivalent atoms: (i) -*x* + 1,-*y* + 2,-*z* + 1; (ii) -*x* + 1,-*y* + 2,-*z*. For the water molecules all disordered H atoms are shown.

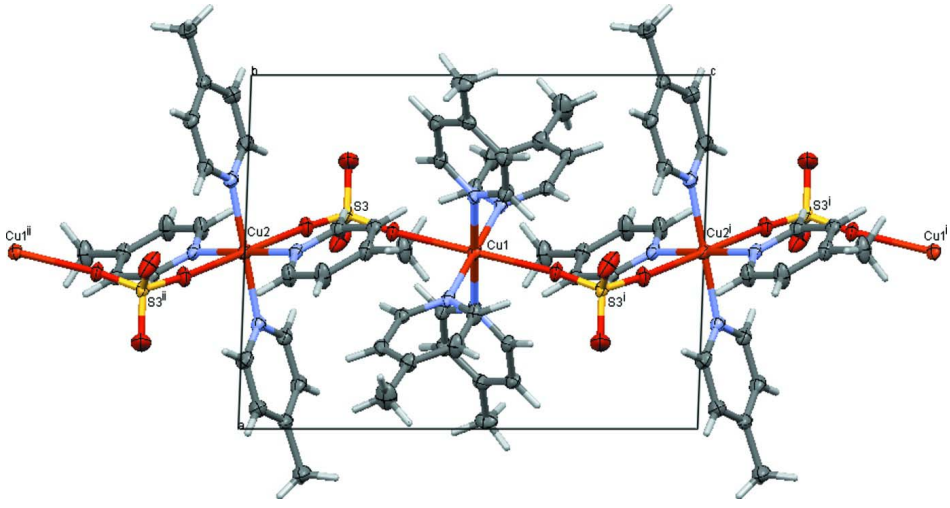


**Figure 2**

View of the polymeric zigzag chains along the [001] direction. Symmetry transformations used to generate equivalent atoms: (i) -*x* + 1, -*y* + 2,-*z* + 1; (ii) -*x* + 1,-*y* + 2,-*z*.

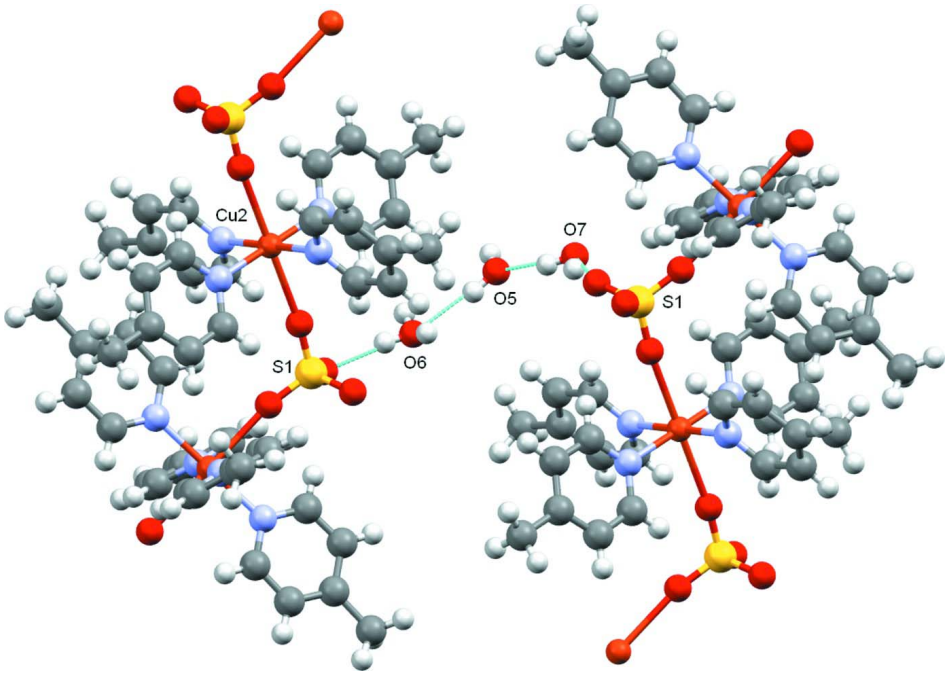


**Figure 3**

Interdigitation between parallel zigzag chains and hydrogen bonding. View is down the *c* axis along the direction of the polymeric chains shown in Fig. 2. Note the interdigitation of tolyl groups of parallel chains along the *a* axis and the water filled areas between chains along the *b* axis. Hydrogen bonds are indicated by dashed light blue lines. Bands of H–bonded water molecules stretch infinitely parallel to the *a* axis.

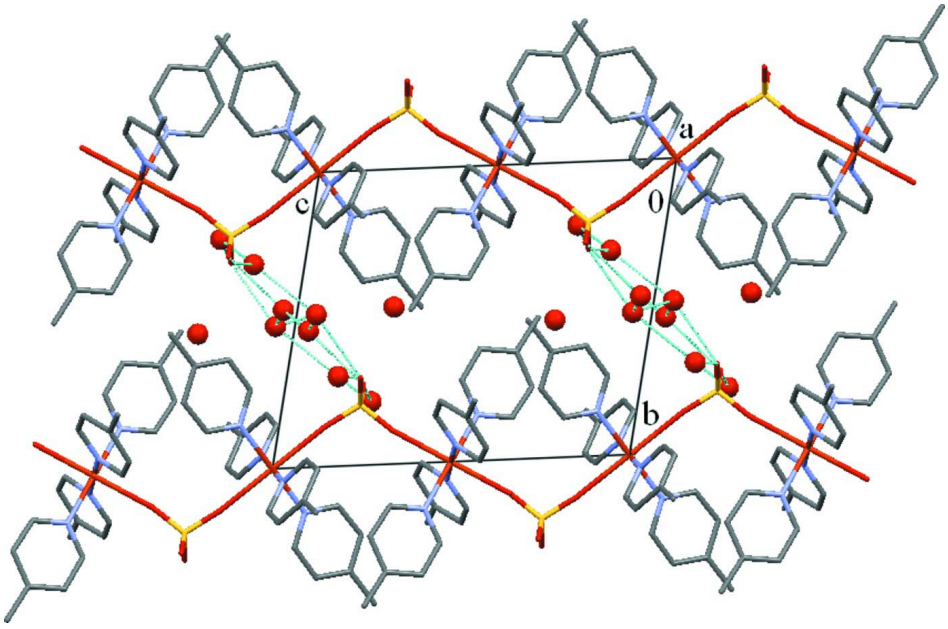


**Figure 4**

Packing and hydrogen bonding in the structure of the title compound. View is down the *a* axis. Hydrogen bonds are indicated by dashed light blue lines (H atoms omitted for clarity). Layers of H bonded water molecules stretch infinitely parallel to the *a*-axis. The single water molecule O9 that is not part of the water cluster is clearly visible in this view.

**_catena_-Poly[[[tetrakis(4-methylpyridine-$\kappa N$)copper(II)]-$\mu$-sulfato-$\kappa^2O$:$O'$] 4.392-hydrate]**

*Crystal data*

$[Cu(SO_4)(C_6H_7N)_4]\cdot 4.393H_2O$
$M_r = 611.27$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 10.4688$ (12) Å
$b = 11.6327$ (14) Å
$c = 12.8300$ (15) Å
$\alpha = 78.672$ (3)°
$\beta = 87.609$ (3)°
$\gamma = 67.571$ (3)°
$V = 1415.2$ (3) Å$^3$
$Z = 2$
$F(000) = 642$
$D_x = 1.434$ Mg m$^{-3}$
Melting point: 373 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4676 reflections
$\theta = 2.3$–$30.4°$
$\mu = 0.90$ mm$^{-1}$
$T = 100$ K
Block, blue
0.60 × 0.45 × 0.40 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*TWINABS*; Bruker, 2008)
$T_{min} = 0.607$, $T_{max} = 0.746$
16814 measured reflections
6963 independent reflections
6170 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.6°$
$h = -13\rightarrow13$
$k = -15\rightarrow15$
$l = 0\rightarrow17$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.118$
$S = 1.06$
6963 reflections
361 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 1.4896P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.68$ e Å$^{-3}$
$\Delta\rho_{min} = -0.61$ e Å$^{-3}$

*Special details*

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

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

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.7599 (3) | 1.0000 (3) | 0.5809 (2) | 0.0204 (5) | |
| H1 | 0.7594 | 0.9263 | 0.6281 | 0.024* | |
| C2 | 0.8641 (3) | 1.0426 (3) | 0.5935 (2) | 0.0231 (5) | |

| | | | | |
|---|---|---|---|---|
| H2 | 0.9329 | 0.9984 | 0.6488 | 0.028* |
| C3 | 0.8680 (3) | 1.1497 (3) | 0.5255 (2) | 0.0230 (5) |
| C4 | 0.7680 (3) | 1.2070 (3) | 0.4436 (2) | 0.0254 (6) |
| H4 | 0.7692 | 1.2784 | 0.3932 | 0.030* |
| C5 | 0.6665 (3) | 1.1597 (3) | 0.4355 (2) | 0.0242 (6) |
| H5 | 0.5990 | 1.1999 | 0.3789 | 0.029* |
| C6 | 0.9755 (3) | 1.2030 (3) | 0.5399 (3) | 0.0313 (6) |
| H6F | 0.9338 | 1.2775 | 0.5729 | 0.047* |
| H6E | 1.0100 | 1.2278 | 0.4704 | 0.047* |
| H6D | 1.0523 | 1.1385 | 0.5856 | 0.047* |
| C7 | 0.6771 (3) | 0.7248 (3) | 0.5052 (2) | 0.0259 (6) |
| H7 | 0.6453 | 0.7125 | 0.5754 | 0.031* |
| C8 | 0.7656 (4) | 0.6209 (3) | 0.4678 (2) | 0.0318 (7) |
| H8 | 0.7943 | 0.5392 | 0.5123 | 0.038* |
| C9 | 0.8136 (3) | 0.6346 (3) | 0.3647 (2) | 0.0272 (6) |
| C10 | 0.9073 (4) | 0.5230 (3) | 0.3195 (3) | 0.0451 (9) |
| H10A | 0.8548 | 0.4736 | 0.3050 | 0.068* |
| H10B | 0.9834 | 0.4695 | 0.3709 | 0.068* |
| H10C | 0.9449 | 0.5532 | 0.2533 | 0.068* |
| C11 | 0.7700 (3) | 0.7568 (3) | 0.3053 (2) | 0.0224 (5) |
| H11 | 0.8015 | 0.7715 | 0.2353 | 0.027* |
| C12 | 0.6808 (3) | 0.8575 (3) | 0.3476 (2) | 0.0212 (5) |
| H12 | 0.6517 | 0.9402 | 0.3048 | 0.025* |
| C13 | 0.4334 (3) | 0.8651 (3) | 0.2020 (2) | 0.0216 (5) |
| H13 | 0.3870 | 0.9490 | 0.2138 | 0.026* |
| C14 | 0.4296 (3) | 0.7646 (3) | 0.2792 (2) | 0.0208 (5) |
| H14 | 0.3811 | 0.7806 | 0.3424 | 0.025* |
| C15 | 0.4963 (3) | 0.6413 (3) | 0.2645 (2) | 0.0253 (6) |
| C16 | 0.5674 (4) | 0.6247 (3) | 0.1714 (2) | 0.0341 (7) |
| H16 | 0.6166 | 0.5416 | 0.1585 | 0.041* |
| C17 | 0.5667 (3) | 0.7287 (3) | 0.0975 (2) | 0.0310 (6) |
| H17 | 0.6156 | 0.7150 | 0.0342 | 0.037* |
| C18 | 0.4934 (4) | 0.5303 (3) | 0.3466 (2) | 0.0371 (8) |
| H18A | 0.5735 | 0.5000 | 0.3960 | 0.056* |
| H18B | 0.4967 | 0.4620 | 0.3112 | 0.056* |
| H18C | 0.4082 | 0.5569 | 0.3860 | 0.056* |
| C19 | 0.7394 (3) | 1.0233 (3) | 0.0928 (2) | 0.0228 (5) |
| H19 | 0.6692 | 1.0967 | 0.1105 | 0.027* |
| C20 | 0.8748 (3) | 0.9916 (3) | 0.1272 (2) | 0.0258 (6) |
| H20 | 0.8958 | 1.0428 | 0.1676 | 0.031* |
| C21 | 0.9792 (3) | 0.8852 (3) | 0.1023 (2) | 0.0256 (6) |
| C22 | 0.9428 (3) | 0.8158 (3) | 0.0406 (2) | 0.0291 (6) |
| H22 | 1.0113 | 0.7430 | 0.0207 | 0.035* |
| C23 | 0.8054 (3) | 0.8539 (3) | 0.0084 (2) | 0.0272 (6) |
| H23 | 0.7822 | 0.8067 | −0.0349 | 0.033* |
| C24 | 1.1264 (3) | 0.8448 (3) | 0.1420 (2) | 0.0328 (7) |
| H24A | 1.1478 | 0.9201 | 0.1405 | 0.049* |
| H24B | 1.1893 | 0.7918 | 0.0962 | 0.049* |

| | | | | | |
|---|---|---|---|---|---|
| H24C | 1.1378 | 0.7964 | 0.2150 | 0.049* | |
| Cu1 | 0.5000 | 1.0000 | 0.5000 | 0.01857 (11) | |
| Cu2 | 0.5000 | 1.0000 | 0.0000 | 0.02031 (11) | |
| N1 | 0.6596 (2) | 1.0589 (2) | 0.50462 (17) | 0.0197 (4) | |
| N2 | 0.6332 (2) | 0.8438 (2) | 0.44667 (17) | 0.0196 (4) | |
| N3 | 0.5002 (2) | 0.8479 (2) | 0.11114 (17) | 0.0204 (4) | |
| N4 | 0.7039 (2) | 0.9544 (2) | 0.03565 (17) | 0.0208 (4) | |
| O1 | 0.4432 (2) | 1.11465 (19) | 0.32076 (14) | 0.0241 (4) | |
| O2 | 0.4297 (2) | 1.1372 (2) | 0.13099 (15) | 0.0257 (4) | |
| O3 | 0.2438 (2) | 1.2743 (2) | 0.21897 (18) | 0.0387 (6) | |
| O4 | 0.4641 (3) | 1.2969 (2) | 0.20710 (19) | 0.0389 (6) | |
| O5 | 0.7247 (3) | 0.3043 (2) | 0.1438 (2) | 0.0501 (7) | |
| H5A | 0.7007 | 0.3519 | 0.0826 | 0.075* | |
| H5B | 0.6529 | 0.2956 | 0.1714 | 0.075* | |
| O6 | 0.3674 (3) | 0.5172 (3) | 0.0548 (2) | 0.0568 (7) | |
| H6A | 0.4103 | 0.5011 | −0.0008 | 0.085* | 0.50 |
| H6B | 0.4034 | 0.4467 | 0.0972 | 0.085* | |
| H6C | 0.2905 | 0.5147 | 0.0399 | 0.085* | 0.50 |
| O7 | 0.9960 (3) | 0.2146 (3) | 0.2479 (2) | 0.0567 (8) | |
| H7A | 0.9293 | 0.2632 | 0.2054 | 0.085* | |
| H7B | 1.0552 | 0.2449 | 0.2228 | 0.085* | |
| O8 | 0.1306 (4) | 0.4630 (4) | 0.0397 (4) | 0.0971 (15) | |
| H8A | 0.1109 | 0.4722 | 0.1027 | 0.146* | |
| H8B | 0.1842 | 0.5027 | 0.0261 | 0.146* | 0.50 |
| H8C | 0.0507 | 0.4844 | 0.0125 | 0.146* | 0.50 |
| O9 | 0.2000 (7) | 0.5327 (6) | 0.2704 (5) | 0.045 (2) | 0.393 (8) |
| H9A | 0.2311 | 0.5640 | 0.2151 | 0.067* | 0.393 (8) |
| H9B | 0.2166 | 0.4593 | 0.2590 | 0.067* | 0.393 (8) |
| S3 | 0.39467 (7) | 1.20521 (6) | 0.21937 (5) | 0.01904 (13) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0229 (13) | 0.0205 (12) | 0.0186 (12) | −0.0087 (11) | −0.0004 (9) | −0.0046 (10) |
| C2 | 0.0201 (12) | 0.0294 (14) | 0.0220 (13) | −0.0095 (11) | −0.0012 (10) | −0.0099 (11) |
| C3 | 0.0187 (12) | 0.0275 (14) | 0.0274 (14) | −0.0106 (11) | 0.0047 (10) | −0.0131 (11) |
| C4 | 0.0276 (14) | 0.0269 (14) | 0.0247 (14) | −0.0145 (12) | 0.0019 (11) | −0.0035 (11) |
| C5 | 0.0278 (14) | 0.0285 (14) | 0.0198 (13) | −0.0162 (12) | −0.0028 (10) | −0.0002 (10) |
| C6 | 0.0259 (14) | 0.0341 (16) | 0.0411 (17) | −0.0166 (13) | 0.0008 (12) | −0.0126 (13) |
| C7 | 0.0371 (16) | 0.0245 (14) | 0.0176 (12) | −0.0145 (12) | −0.0017 (11) | −0.0015 (10) |
| C8 | 0.0478 (19) | 0.0231 (14) | 0.0235 (14) | −0.0138 (14) | −0.0010 (13) | −0.0013 (11) |
| C9 | 0.0316 (15) | 0.0267 (15) | 0.0241 (14) | −0.0111 (12) | −0.0026 (11) | −0.0056 (11) |
| C10 | 0.062 (2) | 0.0287 (17) | 0.0363 (19) | −0.0067 (17) | 0.0047 (17) | −0.0091 (14) |
| C11 | 0.0238 (13) | 0.0278 (14) | 0.0184 (12) | −0.0128 (11) | 0.0005 (10) | −0.0046 (10) |
| C12 | 0.0202 (12) | 0.0258 (13) | 0.0190 (12) | −0.0118 (11) | −0.0009 (10) | −0.0014 (10) |
| C13 | 0.0212 (13) | 0.0235 (13) | 0.0222 (13) | −0.0106 (11) | −0.0007 (10) | −0.0044 (10) |
| C14 | 0.0221 (12) | 0.0275 (14) | 0.0178 (12) | −0.0145 (11) | 0.0002 (10) | −0.0051 (10) |
| C15 | 0.0333 (15) | 0.0262 (14) | 0.0187 (13) | −0.0148 (12) | −0.0032 (11) | −0.0017 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.053 (2) | 0.0220 (14) | 0.0234 (14) | −0.0103 (14) | 0.0054 (13) | −0.0057 (11) |
| C17 | 0.0429 (18) | 0.0297 (15) | 0.0183 (13) | −0.0117 (14) | 0.0062 (12) | −0.0053 (11) |
| C18 | 0.062 (2) | 0.0293 (16) | 0.0227 (15) | −0.0231 (16) | 0.0065 (14) | −0.0013 (12) |
| C19 | 0.0259 (13) | 0.0262 (14) | 0.0201 (12) | −0.0157 (11) | −0.0012 (10) | −0.0010 (10) |
| C20 | 0.0290 (14) | 0.0341 (15) | 0.0208 (13) | −0.0211 (13) | −0.0035 (10) | −0.0006 (11) |
| C21 | 0.0212 (13) | 0.0377 (16) | 0.0175 (12) | −0.0147 (12) | −0.0016 (10) | 0.0032 (11) |
| C22 | 0.0232 (14) | 0.0358 (16) | 0.0252 (14) | −0.0078 (12) | −0.0004 (11) | −0.0059 (12) |
| C23 | 0.0280 (14) | 0.0338 (15) | 0.0223 (13) | −0.0134 (13) | −0.0044 (11) | −0.0065 (12) |
| C24 | 0.0235 (14) | 0.0469 (18) | 0.0278 (15) | −0.0162 (14) | −0.0024 (11) | −0.0009 (13) |
| Cu1 | 0.0212 (2) | 0.0220 (2) | 0.0169 (2) | −0.01266 (19) | −0.00015 (16) | −0.00454 (18) |
| Cu2 | 0.0198 (2) | 0.0240 (2) | 0.0176 (2) | −0.0110 (2) | −0.00355 (16) | 0.00135 (18) |
| N1 | 0.0229 (11) | 0.0248 (11) | 0.0158 (10) | −0.0127 (9) | 0.0005 (8) | −0.0060 (8) |
| N2 | 0.0237 (11) | 0.0233 (11) | 0.0163 (10) | −0.0141 (9) | −0.0028 (8) | −0.0025 (8) |
| N3 | 0.0213 (11) | 0.0236 (11) | 0.0178 (10) | −0.0103 (9) | −0.0041 (8) | −0.0027 (9) |
| N4 | 0.0221 (11) | 0.0242 (11) | 0.0172 (10) | −0.0119 (9) | −0.0015 (8) | 0.0003 (8) |
| O1 | 0.0311 (10) | 0.0295 (10) | 0.0130 (8) | −0.0145 (9) | −0.0030 (7) | −0.0002 (7) |
| O2 | 0.0331 (11) | 0.0319 (11) | 0.0149 (9) | −0.0143 (9) | 0.0015 (8) | −0.0069 (8) |
| O3 | 0.0250 (11) | 0.0461 (14) | 0.0315 (12) | −0.0004 (10) | 0.0011 (9) | −0.0046 (10) |
| O4 | 0.0571 (15) | 0.0289 (11) | 0.0373 (13) | −0.0282 (11) | −0.0223 (11) | 0.0077 (9) |
| O5 | 0.0516 (16) | 0.0386 (14) | 0.0540 (16) | −0.0131 (12) | −0.0121 (13) | −0.0011 (12) |
| O6 | 0.077 (2) | 0.0500 (17) | 0.0409 (15) | −0.0260 (16) | −0.0023 (14) | 0.0007 (13) |
| O7 | 0.0577 (17) | 0.0562 (17) | 0.0492 (16) | −0.0260 (15) | −0.0123 (13) | 0.0174 (14) |
| O8 | 0.066 (2) | 0.089 (3) | 0.123 (3) | −0.044 (2) | −0.044 (2) | 0.050 (2) |
| O9 | 0.049 (4) | 0.044 (4) | 0.045 (4) | −0.017 (3) | 0.001 (3) | −0.017 (3) |
| S3 | 0.0231 (3) | 0.0207 (3) | 0.0147 (3) | −0.0099 (2) | −0.0032 (2) | −0.0026 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.343 (3) | C19—N4 | 1.342 (3) |
| C1—C2 | 1.386 (4) | C19—C20 | 1.388 (4) |
| C1—H1 | 0.9500 | C19—H19 | 0.9500 |
| C2—C3 | 1.387 (4) | C20—C21 | 1.386 (4) |
| C2—H2 | 0.9500 | C20—H20 | 0.9500 |
| C3—C4 | 1.389 (4) | C21—C22 | 1.391 (4) |
| C3—C6 | 1.509 (4) | C21—C24 | 1.508 (4) |
| C4—C5 | 1.386 (4) | C22—C23 | 1.388 (4) |
| C4—H4 | 0.9500 | C22—H22 | 0.9500 |
| C5—N1 | 1.348 (4) | C23—N4 | 1.341 (4) |
| C5—H5 | 0.9500 | C23—H23 | 0.9500 |
| C6—H6F | 0.9800 | C24—H24A | 0.9800 |
| C6—H6E | 0.9800 | C24—H24B | 0.9800 |
| C6—H6D | 0.9800 | C24—H24C | 0.9800 |
| C7—N2 | 1.352 (4) | Cu1—N1$^{i}$ | 2.041 (2) |
| C7—C8 | 1.373 (4) | Cu1—N1 | 2.041 (2) |
| C7—H7 | 0.9500 | Cu1—N2 | 2.046 (2) |
| C8—C9 | 1.397 (4) | Cu1—N2$^{i}$ | 2.046 (2) |
| C8—H8 | 0.9500 | Cu1—O1 | 2.3926 (18) |
| C9—C11 | 1.385 (4) | Cu1—O1$^{i}$ | 2.3926 (18) |

| | | | |
|---|---|---|---|
| C9—C10 | 1.503 (4) | Cu2—N4$^{ii}$ | 2.042 (2) |
| C10—H10A | 0.9800 | Cu2—N4 | 2.042 (2) |
| C10—H10B | 0.9800 | Cu2—N3 | 2.042 (2) |
| C10—H10C | 0.9800 | Cu2—N3$^{ii}$ | 2.042 (2) |
| C11—C12 | 1.382 (4) | Cu2—O2$^{ii}$ | 2.443 (2) |
| C11—H11 | 0.9500 | Cu2—O2 | 2.443 (2) |
| C12—N2 | 1.348 (3) | O1—S3 | 1.4726 (19) |
| C12—H12 | 0.9500 | O2—S3 | 1.464 (2) |
| C13—N3 | 1.344 (3) | O3—S3 | 1.473 (2) |
| C13—C14 | 1.387 (4) | O4—S3 | 1.485 (2) |
| C13—H13 | 0.9500 | O5—H5A | 0.8515 |
| C14—C15 | 1.382 (4) | O5—H5B | 0.8498 |
| C14—H14 | 0.9500 | O6—H6A | 0.8408 |
| C15—C16 | 1.388 (4) | O6—H6B | 0.8470 |
| C15—C18 | 1.507 (4) | O6—H6C | 0.8472 |
| C16—C17 | 1.381 (4) | O7—H7A | 0.8445 |
| C16—H16 | 0.9500 | O7—H7B | 0.8477 |
| C17—N3 | 1.336 (4) | O8—H8A | 0.8431 |
| C17—H17 | 0.9500 | O8—H8B | 0.8459 |
| C18—H18A | 0.9800 | O8—H8C | 0.8456 |
| C18—H18B | 0.9800 | O9—H9A | 0.8436 |
| C18—H18C | 0.9800 | O9—H9B | 0.8448 |

| | | | |
|---|---|---|---|
| N1—C1—C2 | 122.6 (3) | C22—C21—C24 | 121.1 (3) |
| N1—C1—H1 | 118.7 | C23—C22—C21 | 119.3 (3) |
| C2—C1—H1 | 118.7 | C23—C22—H22 | 120.3 |
| C1—C2—C3 | 120.0 (3) | C21—C22—H22 | 120.3 |
| C1—C2—H2 | 120.0 | N4—C23—C22 | 123.0 (3) |
| C3—C2—H2 | 120.0 | N4—C23—H23 | 118.5 |
| C2—C3—C4 | 117.3 (2) | C22—C23—H23 | 118.5 |
| C2—C3—C6 | 121.7 (3) | C21—C24—H24A | 109.5 |
| C4—C3—C6 | 121.0 (3) | C21—C24—H24B | 109.5 |
| C5—C4—C3 | 119.7 (3) | H24A—C24—H24B | 109.5 |
| C5—C4—H4 | 120.1 | C21—C24—H24C | 109.5 |
| C3—C4—H4 | 120.1 | H24A—C24—H24C | 109.5 |
| N1—C5—C4 | 122.7 (3) | H24B—C24—H24C | 109.5 |
| N1—C5—H5 | 118.7 | N1$^{i}$—Cu1—N1 | 180.00 (11) |
| C4—C5—H5 | 118.7 | N1$^{i}$—Cu1—N2 | 91.28 (9) |
| C3—C6—H6F | 109.5 | N1—Cu1—N2 | 88.72 (9) |
| C3—C6—H6E | 109.5 | N1$^{i}$—Cu1—N2$^{i}$ | 88.72 (9) |
| H6F—C6—H6E | 109.5 | N1—Cu1—N2$^{i}$ | 91.28 (9) |
| C3—C6—H6D | 109.5 | N2—Cu1—N2$^{i}$ | 179.999 (1) |
| H6F—C6—H6D | 109.5 | N1$^{i}$—Cu1—O1 | 90.27 (8) |
| H6E—C6—H6D | 109.5 | N1—Cu1—O1 | 89.73 (8) |
| N2—C7—C8 | 122.9 (3) | N2—Cu1—O1 | 90.38 (8) |
| N2—C7—H7 | 118.5 | N2$^{i}$—Cu1—O1 | 89.62 (8) |
| C8—C7—H7 | 118.5 | N1$^{i}$—Cu1—O1$^{i}$ | 89.73 (8) |
| C7—C8—C9 | 120.4 (3) | N1—Cu1—O1$^{i}$ | 90.27 (8) |

| | | | |
|---|---|---|---|
| C7—C8—H8 | 119.8 | N2—Cu1—O1$^{i}$ | 89.62 (8) |
| C9—C8—H8 | 119.8 | N2$^{i}$—Cu1—O1$^{i}$ | 90.38 (8) |
| C11—C9—C8 | 116.6 (3) | O1—Cu1—O1$^{i}$ | 180.0 |
| C11—C9—C10 | 121.3 (3) | N4$^{ii}$—Cu2—N4 | 180.0 |
| C8—C9—C10 | 122.1 (3) | N4$^{ii}$—Cu2—N3 | 89.63 (9) |
| C9—C10—H10A | 109.5 | N4—Cu2—N3 | 90.37 (9) |
| C9—C10—H10B | 109.5 | N4$^{ii}$—Cu2—N3$^{ii}$ | 90.37 (9) |
| H10A—C10—H10B | 109.5 | N4—Cu2—N3$^{ii}$ | 89.63 (9) |
| C9—C10—H10C | 109.5 | N3—Cu2—N3$^{ii}$ | 179.999 (1) |
| H10A—C10—H10C | 109.5 | N4—Cu2—O2$^{ii}$ | 88.90 (8) |
| H10B—C10—H10C | 109.5 | N4$^{ii}$—Cu2—O2$^{ii}$ | 91.11 (8) |
| C12—C11—C9 | 120.2 (3) | N3—Cu2—O2$^{ii}$ | 88.72 (8) |
| C12—C11—H11 | 119.9 | N3$^{ii}$—Cu2—O2$^{ii}$ | 91.28 (8) |
| C9—C11—H11 | 119.9 | N4—Cu2—O2 | 91.11 (8) |
| N2—C12—C11 | 123.1 (3) | N4$^{ii}$—Cu2—O2 | 88.90 (8) |
| N2—C12—H12 | 118.5 | N3—Cu2—O2 | 91.28 (8) |
| C11—C12—H12 | 118.5 | N3$^{ii}$—Cu2—O2 | 88.72 (8) |
| N3—C13—C14 | 122.4 (3) | O2$^{ii}$—Cu2—O2 | 180.000 (1) |
| N3—C13—H13 | 118.8 | C1—N1—C5 | 117.5 (2) |
| C14—C13—H13 | 118.8 | C1—N1—Cu1 | 120.11 (18) |
| C15—C14—C13 | 120.2 (3) | C5—N1—Cu1 | 122.30 (18) |
| C15—C14—H14 | 119.9 | C12—N2—C7 | 116.8 (2) |
| C13—C14—H14 | 119.9 | C12—N2—Cu1 | 119.50 (19) |
| C14—C15—C16 | 116.9 (3) | C7—N2—Cu1 | 123.72 (19) |
| C14—C15—C18 | 121.5 (3) | C17—N3—C13 | 117.5 (2) |
| C16—C15—C18 | 121.7 (3) | C17—N3—Cu2 | 122.05 (19) |
| C17—C16—C15 | 120.1 (3) | C13—N3—Cu2 | 120.41 (19) |
| C17—C16—H16 | 120.0 | C23—N4—C19 | 117.7 (2) |
| C15—C16—H16 | 120.0 | C23—N4—Cu2 | 122.75 (19) |
| N3—C17—C16 | 122.9 (3) | C19—N4—Cu2 | 119.52 (19) |
| N3—C17—H17 | 118.5 | S3—O1—Cu1 | 169.60 (13) |
| C16—C17—H17 | 118.5 | H5A—O5—H5B | 107.6 |
| C15—C18—H18A | 109.5 | H6A—O6—H6B | 101.2 |
| C15—C18—H18B | 109.5 | H6A—O6—H6C | 101.0 |
| H18A—C18—H18B | 109.5 | H6B—O6—H6C | 100.4 |
| C15—C18—H18C | 109.5 | H7A—O7—H7B | 97.7 |
| H18A—C18—H18C | 109.5 | H8A—O8—H8B | 100.6 |
| H18B—C18—H18C | 109.5 | H8A—O8—H8C | 100.5 |
| N4—C19—C20 | 122.5 (3) | H8B—O8—H8C | 126.9 |
| N4—C19—H19 | 118.7 | H9A—O9—H9B | 100.6 |
| C20—C19—H19 | 118.7 | O2—S3—O1 | 109.72 (12) |
| C21—C20—C19 | 119.9 (3) | O2—S3—O3 | 109.57 (13) |
| C21—C20—H20 | 120.0 | O1—S3—O3 | 110.06 (13) |
| C19—C20—H20 | 120.0 | O2—S3—O4 | 109.34 (14) |
| C20—C21—C22 | 117.5 (3) | O1—S3—O4 | 108.76 (12) |
| C20—C21—C24 | 121.4 (3) | O3—S3—O4 | 109.37 (15) |
| | | | |
| N1—C1—C2—C3 | −0.3 (4) | C11—C12—N2—C7 | 0.1 (4) |

| | | | |
|---|---|---|---|
| C1—C2—C3—C4 | −2.6 (4) | C11—C12—N2—Cu1 | −179.4 (2) |
| C1—C2—C3—C6 | 176.9 (3) | C8—C7—N2—C12 | −0.1 (4) |
| C2—C3—C4—C5 | 2.7 (4) | C8—C7—N2—Cu1 | 179.4 (2) |
| C6—C3—C4—C5 | −176.8 (3) | N1$^{i}$—Cu1—N2—C12 | 109.81 (19) |
| C3—C4—C5—N1 | 0.1 (5) | N1—Cu1—N2—C12 | −70.19 (19) |
| N2—C7—C8—C9 | −0.7 (5) | O1—Cu1—N2—C12 | 19.53 (19) |
| C7—C8—C9—C11 | 1.5 (4) | O1$^{i}$—Cu1—N2—C12 | −160.47 (19) |
| C7—C8—C9—C10 | −178.2 (3) | N1$^{i}$—Cu1—N2—C7 | −69.7 (2) |
| C8—C9—C11—C12 | −1.5 (4) | N1—Cu1—N2—C7 | 110.3 (2) |
| C10—C9—C11—C12 | 178.3 (3) | O1—Cu1—N2—C7 | −160.0 (2) |
| C9—C11—C12—N2 | 0.7 (4) | O1$^{i}$—Cu1—N2—C7 | 20.0 (2) |
| N3—C13—C14—C15 | 0.1 (4) | C16—C17—N3—C13 | 1.0 (5) |
| C13—C14—C15—C16 | 1.2 (4) | C16—C17—N3—Cu2 | −179.6 (3) |
| C13—C14—C15—C18 | −179.7 (3) | C14—C13—N3—C17 | −1.2 (4) |
| C14—C15—C16—C17 | −1.4 (5) | C14—C13—N3—Cu2 | 179.34 (19) |
| C18—C15—C16—C17 | 179.4 (3) | N4$^{ii}$—Cu2—N3—C17 | 109.5 (2) |
| C15—C16—C17—N3 | 0.4 (5) | N4—Cu2—N3—C17 | −70.5 (2) |
| N4—C19—C20—C21 | 0.1 (4) | N4$^{ii}$—Cu2—N3—C13 | −71.1 (2) |
| C19—C20—C21—C22 | 1.6 (4) | N4—Cu2—N3—C13 | 108.9 (2) |
| C19—C20—C21—C24 | −177.5 (3) | C22—C23—N4—C19 | 2.9 (4) |
| C20—C21—C22—C23 | −1.1 (4) | C22—C23—N4—Cu2 | −174.0 (2) |
| C24—C21—C22—C23 | 178.1 (3) | C20—C19—N4—C23 | −2.3 (4) |
| C21—C22—C23—N4 | −1.2 (5) | C20—C19—N4—Cu2 | 174.7 (2) |
| C2—C1—N1—C5 | 3.2 (4) | N3—Cu2—N4—C23 | 71.8 (2) |
| C2—C1—N1—Cu1 | −174.3 (2) | N3$^{ii}$—Cu2—N4—C23 | −108.2 (2) |
| C4—C5—N1—C1 | −3.1 (4) | N3—Cu2—N4—C19 | −105.1 (2) |
| C4—C5—N1—Cu1 | 174.3 (2) | N3$^{ii}$—Cu2—N4—C19 | 74.9 (2) |
| N2—Cu1—N1—C1 | −73.2 (2) | N1$^{i}$—Cu1—O1—S3 | 97.0 (7) |
| N2$^{i}$—Cu1—N1—C1 | 106.8 (2) | N1—Cu1—O1—S3 | −83.0 (7) |
| O1—Cu1—N1—C1 | −163.6 (2) | N2—Cu1—O1—S3 | −171.7 (7) |
| O1$^{i}$—Cu1—N1—C1 | 16.4 (2) | N2$^{i}$—Cu1—O1—S3 | 8.3 (7) |
| N2—Cu1—N1—C5 | 109.4 (2) | Cu1—O1—S3—O2 | −173.0 (7) |
| N2$^{i}$—Cu1—N1—C5 | −70.6 (2) | Cu1—O1—S3—O3 | −52.3 (7) |
| O1—Cu1—N1—C5 | 19.1 (2) | Cu1—O1—S3—O4 | 67.4 (7) |
| O1$^{i}$—Cu1—N1—C5 | −160.9 (2) | | |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H5*A*···O6$^{iii}$ | 0.85 | 2.04 | 2.887 (4) | 173 |
| O5—H5*B*···O4$^{iv}$ | 0.85 | 2.01 | 2.843 (4) | 168 |
| O6—H6*A*···O6$^{iii}$ | 0.84 | 2.34 | 2.983 (6) | 133 |
| O6—H6*B*···O4$^{iv}$ | 0.85 | 1.92 | 2.762 (4) | 172 |
| O6—H6*C*···O8 | 0.85 | 1.98 | 2.804 (5) | 163 |
| O7—H7*A*···O5 | 0.84 | 2.16 | 2.906 (4) | 148 |
| O7—H7*B*···O3$^{v}$ | 0.85 | 2.13 | 2.923 (4) | 157 |

| | | | | |
|---|---|---|---|---|
| O8—H8*A*···O3$^{iv}$ | 0.84 | 2.42 | 2.788 (4) | 107 |
| O8—H8*B*···O6 | 0.85 | 2.04 | 2.804 (5) | 149 |
| O8—H8*C*···O8$^{vi}$ | 0.85 | 1.87 | 2.710 (7) | 176 |
| O9—H9*A*···O6 | 0.84 | 2.48 | 3.210 (7) | 145 |
| O9—H9*B*···O3$^{iv}$ | 0.84 | 2.22 | 3.063 (7) | 175 |
| O9—H9*B*···O4$^{iv}$ | 0.84 | 2.71 | 3.275 (7) | 126 |

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