



The first structural characterization of the protonated azacyclam ligand in *catena*-poly[[[(perchlorato)copper(II)]- μ -3-(3-carboxypropyl)-1,5,8,12-tetraaza-3-azoniacyclotetradecane] bis(perchlorate)]

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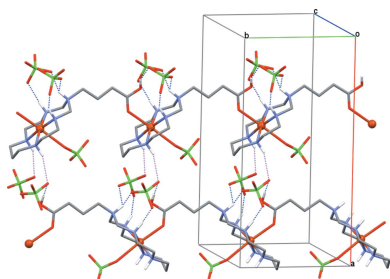
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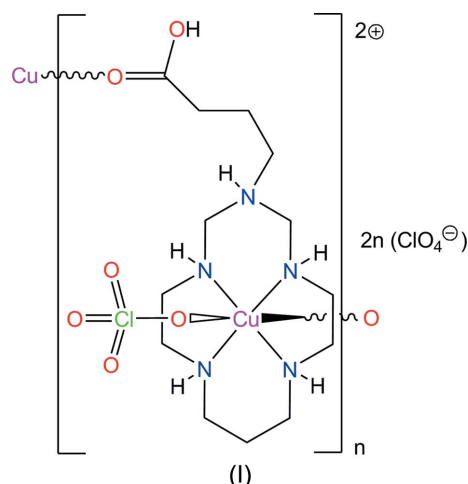
The asymmetric unit of the title compound, *catena*-poly[[[(perchlorato- κ O)copper(II)]- μ -3-(3-carboxypropyl)-1,5,8,12-tetraaza-3-azoniacyclotetradecane- $\kappa^4N^1,N^5,N^8,N^{12}$] bis(perchlorate)], $\{[\text{Cu}(\text{C}_{13}\text{H}_{30}\text{N}_5\text{O}_2)(\text{ClO}_4)](\text{ClO}_4)_2\}_n$, (I), consists of a macrocyclic cation, one coordinated perchlorate anion and two perchlorate ions as counter-anions. The metal ion is coordinated in a tetragonally distorted octahedral geometry by the four secondary N atoms of the macrocyclic ligand, the mutually *trans* O atoms of the perchlorate anion and the carbonyl O atom of the protonated carboxylic acid group of a neighbouring cation. The average equatorial Cu–N bond lengths [2.01 (6) Å] are significantly shorter than the axial Cu–O bond lengths [2.379 (8) Å for carboxylate and average 2.62 (7) Å for disordered perchlorate]. The coordinated macrocyclic ligand in (I) adopts the most energetically favourable *trans*-III conformation with an equatorial orientation of the substituent at the protonated distal 3-position N atom in a six-membered chelate ring. The coordination of the carboxylic acid group of the cation to a neighbouring complex unit results in the formation of infinite chains running along the *b*-axis direction, which are crosslinked by N–H \cdots O hydrogen bonds between the secondary amine groups of the macrocycle and O atoms of the perchlorate counter-anions to form sheets lying parallel to the (001) plane. Additionally, the extended structure of (I) is consolidated by numerous intra- and interchain C–H \cdots O contacts.

1. Chemical context

Because of their exceptionally high thermodynamic stability and kinetic inertness (Melson, 1979; Yatsimirskii & Lampeka, 1985), transition-metal complexes of the macrocycles 1,4,8,11-tetraazacyclotetradecane (cyclam), N^3,N^{10} -disubstituted 1,3,5,8,10,12-hexaazacyclotetradecane (diazacyclam) and, to a lesser extent, N^3 -substituted 1,3,5,8,12-pentaazacyclotetradecane (azacyclam) are popular building units for the assembly of metal–organic frameworks (MOFs), demonstrating many promising applications (Lampeka & Tsymbal, 2004; Suh & Moon, 2007; Suh *et al.*, 2012; Stackhouse & Ma, 2018). Two latter types of the Cu^{II} and Ni^{II} complexes are readily obtainable *via* template-directed Mannich condensation of bis(ethylenediamine) or 3,7-diazanonane-1,9-diamine complexes, respectively, with formaldehyde and primary amines (Rosokha *et al.*, 1993; Costisor & Linert, 2000). The use of primary amines bearing a carboxylic acid function as locking fragments in these template reactions allows for the preparation of complexes of carboxyl-functionalized diaza-



cyclams, as was shown for the Ni^{II} and Cu^{II} complexes of diazacyclam substituted with 3-carboxypropyl groups (Lu *et al.*, 2005; Ou *et al.*, 2005). Such compounds are of particular interest because they can self-polymerize due to the coordination of the donor group of the substituent to the metal ion of another molecule, thus forming coordination polymers without using additional bridging ligands, the most popular of which are carboxylates (Rao *et al.*, 2004). Indeed, the Cu^{II} complex of this diazacyclam ligand possesses a self-polymeric structure (Ou *et al.*, 2005), whereas the Ni^{II} complex does not form a polymer (Lu *et al.*, 2005). Data on the polymeric compounds of the given type formed by the complexes of functionalized azacyclam are not available in the literature so far.



Another issue of interest is the acid–base properties of the noncoordinated distal N atom present in the macrocyclic backbones of aza- and diazacyclams. Its likely protonation was postulated first based on the solution properties of the Ni^{II} compounds (Rosokha *et al.*, 1993; Tsymbal *et al.*, 1995; Hay *et al.*, 1997) and was further confirmed by X-ray structural analysis of the diethyl-substituted Ni^{II} diazacyclam complex (Jiang *et al.*, 2006), while such a possibility for the Cu^{II} complexes has not been reported yet.

Herein, we describe the synthesis and the crystal structure of the title Cu^{II} complex, (I), with a protonated azacyclam ligand bearing a carboxylic acid group, namely, *catenapoly*[[[(perchlorato- κ O)copper(II)]- μ -3-(3-carboxypropyl)-1,5,8,12-tetraaza-3-azoniacyclotetradecane- κ^4 N¹,N⁵,N⁸,N¹²] bis(perchlorate)], $[[\text{Cu}(\text{H}_2\text{L})(\text{ClO}_4)](\text{ClO}_4)_2]_n$, which is the first example of azacyclam ligand with a carboxylic acid group.

2. Structural commentary

The Cu^{II} ion in the complex cation in (I) is coordinated by four secondary amine N atoms of the azamacrocyclic ligand in a square-planar fashion and by O atoms from the perchlorate anion and the carboxylic acid group of a neighbouring cation in the axial positions, resulting in a tetragonally distorted octahedral geometry (Table 1 and Fig. 1). The Cu^{II} ion is displaced by 0.075 Å from the mean plane of the N₄ donor atoms (r.m.s. deviation = 0.005 Å) towards the O2 atom of the

Table 1
Selected bond lengths and angles (Å, °).

Distances		Bite angles	
Cu1–N1	2.005 (19)	N1–Cu1–N2	84.4 (9)
Cu1–N2	1.99 (2)	N4–Cu1–N5	88.0 (9)
Cu1–N4	2.071 (19)	N1–Cu1–N5	93.9 (4)
Cu1–N5	1.99 (2)	N2–Cu1–N4	93.4 (4)
Cu1–O2	2.379 (8)		
Cu1–O3	2.544 (16)		
Cu1–O3X	2.687 (18)		

carboxylate group. The equatorial Cu–N bond lengths are significantly shorter than the axial Cu–O bond lengths (Table 1), which can be attributed to a large Jahn–Teller distortion.

The macrocyclic ligand in (I) adopts the most energetically favourable *trans*-III (*R,R,S,S*) conformation (Bosnich *et al.*, 1965), with the five-membered chelate rings in *gauche* [average bite angle = 86.2 (18)°] and the six-membered chelate rings in *chair* [average bite angle = 93.6 (2)°] conformations. The methylene group of the substituent at the noncoordinated N3 atom in the six-membered chelate ring is oriented equatorially. Such an arrangement of the substituent, in contrast to an axial orientation, is relatively uncommon and only a few examples of such Cu^{II} complexes with aza- and diazacyclam ligands have been described so far (Shin *et al.*, 2010, 2012; Tsymbal *et al.*, 2010; Husain *et al.*, 2012; Xia *et al.*, 2014).

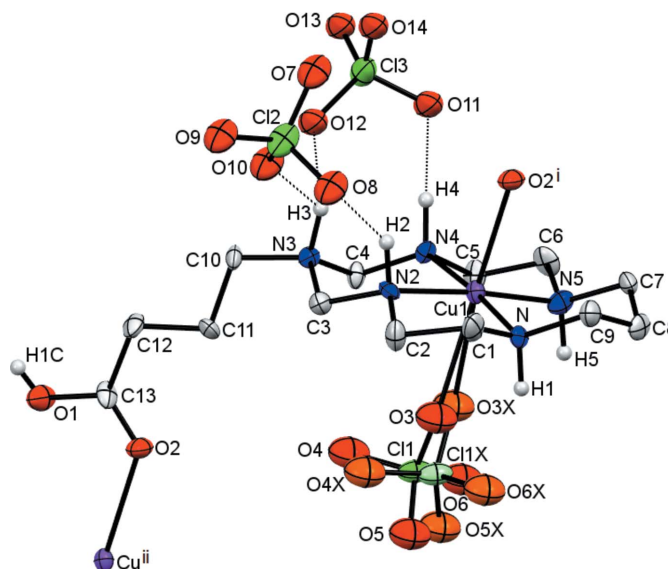


Figure 1
View of the asymmetric unit of (I), expanded to show the linking atoms O2ⁱ and Cu1ⁱⁱ forming the [010] polymeric chains, with displacement ellipsoids drawn at the 30% probability level. H atoms attached to C atoms have been omitted for clarity. The coordinated perchlorate anion (Cl1O₄) is equally disordered over two sets of sites and is shown in different shades; the minor-disorder components of the perchlorate counter-anions with site occupancies of 20 (Cl2O₄) and 22% (Cl3O₄) have been omitted for clarity. Dashed lines represent hydrogen bonds. [Symmetry codes: (i) *x*, *y* + 1, *z*; (ii) *x*, *y* – 1, *z*.]

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N2—H2···O8	1.00	2.35	3.15 (3)	136
N3—H3···O10	1.00	2.23	2.85 (2)	119
N3—H3···O12	1.00	2.43	2.99 (2)	115
N4—H4···O11	1.00	2.23	3.08 (3)	143
O1—H1C···O7 ⁱ	0.84	2.39	3.13 (4)	147
N2—H2···O1 ⁱⁱ	1.00	2.47	3.20 (3)	129
N4—H4···O1 ⁱⁱ	1.00	2.43	3.13 (3)	126
N1—H1···O7 ⁱⁱⁱ	1.00	2.40	3.29 (3)	148
N5—H5···O14 ⁱⁱⁱ	1.00	2.35	3.26 (3)	151
C2—H2A···O3 ⁱⁱⁱ	0.99	2.63	3.15 (4)	113
C2—H2B···O8	0.99	2.64	3.26 (4)	120
C3—H3A···O3 ⁱⁱⁱ	0.99	2.65	3.23 (3)	118
C3—H3B···O11 ^{iv}	0.99	2.57	3.42 (3)	144
C4—H4A···O4 ⁱⁱⁱ	0.99	2.44	3.40 (3)	163
C4—H4B···O8 ^v	0.99	2.61	3.48 (3)	147
C5—H5B···O5 ^v	0.99	2.65	3.29 (3)	122
C7—H7A···O2 ⁱⁱ	0.99	2.64	3.23 (3)	118
C7—H7A···O4 ^{vi}	0.99	2.65	3.26 (3)	120
C7—H7A···O9 ^{vii}	0.99	2.64	3.44 (3)	138
C8—H8B···O5 ^{vi}	0.99	2.65	3.56 (3)	153
C9—H9B···O2 ⁱⁱ	0.99	2.58	3.20 (3)	120
C10—H10B···O10	0.99	2.60	3.15 (3)	115
C10—H10A···O12	0.99	2.56	3.18 (3)	121
C11—H11B···O9 ^v	0.99	2.47	3.40 (4)	157
C11—H11A···O13 ^{iv}	0.99	2.44	3.43 (3)	172
N1—H1···O6X ⁱⁱⁱ	1.00	2.46	3.36 (3)	149
N5—H5···O3X ⁱⁱⁱ	1.00	2.36	2.88 (3)	112
C3—H3A···O4X ⁱⁱⁱ	0.99	2.52	3.45 (4)	156
C4—H4A···O3X ⁱⁱⁱ	0.99	2.47	3.14 (3)	125
C5—H5A···O3X ⁱⁱⁱ	0.99	2.13	2.83 (4)	127
C6—H6B···O4X ^v	0.99	2.61	3.48 (4)	147
C8—H8B···O5X ^{vi}	0.99	2.65	3.59 (3)	157
C12—H12B···O5X ⁱ	0.99	2.62	3.19 (3)	117
C12—H12A···O6X ⁱ	0.99	2.52	3.25 (3)	130

Symmetry codes: (i) $x, y - 1, z$; (ii) $x, y + 1, z$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, z$; (iv) $-x + \frac{1}{2}, y - \frac{1}{2}, z - \frac{1}{2}$; (v) $-x + \frac{1}{2}, y - \frac{1}{2}, z + \frac{1}{2}$; (vi) $x - \frac{1}{2}, -y + \frac{3}{2}, z$; (vii) $-x + \frac{1}{2}, y + \frac{1}{2}, z + \frac{1}{2}$.

The formation of the azonia N3H⁺ group in (I) leads to clear changes in the C—N—C angles compared to the nonprotonated ones. The sum of these angles in the latter case (345–354°) is much larger than the canonical value for an *sp*³-hybridized N atom (*ca* 327°), thus indicating their partial *sp*² character (Tsybmal *et al.*, 2010; Andriichuk *et al.*, 2019), while in (I) this parameter equals 335 (2)°, demonstrating an *sp*²-to-*sp*³ transformation of the noncoordinated N atom upon protonation.

The C—O bond lengths in the carboxylic acid group of the substituent differ considerably [1.318 (13) and 1.198 (13) Å for C13—O1 and C13—O2, respectively], thus confirming its protonated form and the lack of delocalization. Interestingly, it is coordinated to the Cu^{II} ion *via* O2, the carbonyl O atom, which is analogous to the situation observed in a bis(3-carboxypropyl)-substituted diazacyclam polymeric complex (Ou *et al.*, 2005).

Three disordered perchlorate anions in the title compound counterbalance the charge of the complex cations. The Cl1O₄ anion is completely disordered over two positions with site occupancies of 50% and is weakly coordinated to the metal ion (Table 1). Two remaining counter-anions are partially disordered with the retention of the positions of the central Cl

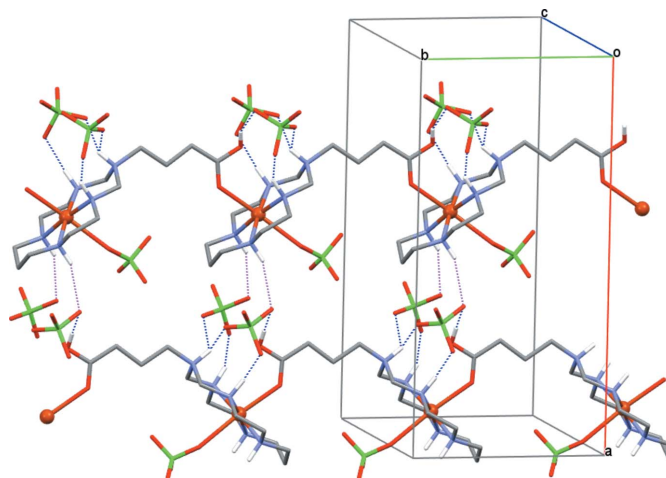


Figure 2
The packing in (I), showing [010] polymeric chains crosslinked by N—H···O hydrogen bonds to form sheets lying parallel to the (001) plane. H atoms at C atoms of the macrocyclic ligands have been omitted, as has one disorder component of the perchlorate anions coordinated to Cu^{II} and the minor-disorder components of the noncoordinated perchlorate counter-anions. Intra- (blue) and interchain (purple) N—H···O hydrogen bonds are shown as dashed lines.

atoms, with site occupancies of the major components of 80 (Cl2O₄) and 78% (Cl3O₄). Because of the low partial population, the minor components of these perchlorate anions were not considered further in the analysis of the hydrogen-bonding network.

3. Supramolecular features

The inter-cationic coordination of the carboxylic acid group of the substituent in the macrocycle to the metal ion results in the formation of one-dimensional polymeric chains running along the *b*-axis direction (Fig. 2). These chains are further reinforced by hydrogen bonding between secondary amine groups of the macrocycle acting as proton donors and O atoms of the perchlorate anions as proton acceptors [N2—H2···O8(Cl2) and N4—H4···O11(Cl3)]. Additionally, the azonia group of the macrocycle forms a bifurcated hydrogen bond with both noncoordinated perchlorate anions [N3—H3⁺···O10(Cl2),-O12(Cl3)], so that each perchlorate anion is fixed in a chain in a ditopic manner (Fig. 2 and Table 2). In addition, weak hydrogen bonding exists between the carboxylic acid group as the proton donor and an O atom of one of the perchlorate ions [O1—H1C···O7(Cl2)(*x, y - 1, z*)], as well as between secondary amine groups of the macrocycle and an O atom of the carboxylic acid group as the proton acceptor [N2—H2(N4—H4)···O1(*x, y + 1, z*)]. Hydrogen bonding of the secondary amine groups of the macrocycle and the O atoms of perchlorate anions not involved in above-mentioned intra-chain interactions [N1—H1···O7(Cl2)($x - \frac{1}{2}, -y + \frac{1}{2}, z$) and N5—H5···O14(Cl3)($x - \frac{1}{2}, -y + \frac{1}{2}, z$)] results in the formation of sheets lying parallel to the (001) plane (Fig. 2), with a distance between them of 6.82 Å. There are also numerous intra- and interchain C—H···O contacts between methylene

groups of the macrocycle and the O atoms of the anions (Table 2), and these latter interactions are responsible for the formation of the three-dimensional structure of (I).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, last update February 2019; Groom *et al.*, 2016) indicated that among 76 Cu^{II} complexes of *N*³,*N*¹⁰-disubstituted diazacyclam ligands, 37 compounds are formed by the ligands bearing alkyl substituents decorated with potentially coordinating groups (hydroxy, imidazolyl, thienyl, amine, nitrile or carboxyl) and many of them were investigated as building blocks for the construction of MOFs by using additional carboxylate or metalocyanide linkers. At the same time, there are only two examples demonstrating self-polymerization (coordination of the substituent in the macrocycle with a neighbouring metal ion), namely, those with diazacyclam ligands containing 2-propionitrile (refcode CAGHOI; Liu *et al.*, 2002) or 3-carboxypropyl (WAMWIR; Ou *et al.*, 2005) donor groups. Among the Cu^{II} complexes of *N*³-substituted azacyclam ligands only one complex with the 3-picolyl substituent that is potentially able to coordinate has been described (NOLDAW; Andriichuk *et al.*, 2019), thus the title compound (I) is the second example of a [Cu(azacyclam)]²⁺ cation of this kind described so far.

5. Synthesis and crystallization

All chemicals and solvents used in this work were purchased from Sigma–Aldrich and were used without further purification. The starting Cu^{II} complexes with an open-chain tetraamine, [Cu(2,3,2-*tet*)](ClO₄)₂ (2,3,2-*tet* = 3,7-diazanonane-1,9-diamine), was prepared according to a published method (Maloshtan & Lampeka, 1996). Compound (I) was prepared as follows. A mixture of [Cu(2,3,2-*tet*)](ClO₄)₂ (200 mg, 0.46 mmol), 4-aminobutanoic acid (49 mg, 0.47 mmol) and 30% aqueous formaldehyde (0.24 ml, 3.2 mmol) in methanol (40 ml) was refluxed for 24 h. After cooling and filtration, the solution was kept in a refrigerator overnight. The violet crystalline precipitate was filtered off, washed with methanol (5 ml) and recrystallized from a 1:1 (*v/v*) water–ethanol solvent mixture (10 ml) containing 0.5 *M* perchloric acid (yield 84 mg, 28%). Analysis calculated (%) for C₁₃H₃₀Cl₃CuN₅O₁₄: C 24.01, H 4.65, N 10.76; found: C 24.17, H 4.51, N 10.92. Violet blocks of (I) suitable for X-ray diffraction analysis were selected from the sample resulting from the synthesis.

Safety note: perchlorate salts of metal complexes are potentially explosive and should be handled with care.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms in (I) were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H = 0.99 Å, N–H = 1.00 Å and carboxylate O–H = 0.84 Å, with *U*_{iso}(H) values of 1.2 or

Table 3
Experimental details.

Crystal data	
Chemical formula	[Cu(C ₁₃ H ₃₀ N ₅ O ₂)(ClO ₄)](ClO ₄) ₂
<i>M</i> _r	650.31
Crystal system, space group	Orthorhombic, <i>Pna</i> 2 ₁
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	18.990 (4), 9.3640 (19), 13.636 (3)
<i>V</i> (Å ³)	2424.9 (8)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
<i>μ</i> (mm ^{−1})	1.31
Crystal size (mm)	0.18 × 0.14 × 0.12
Data collection	
Diffractometer	Bruker X8 APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2007)
<i>T</i> _{min} , <i>T</i> _{max}	0.799, 0.859
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	60778, 4458, 2787
<i>R</i> _{int}	0.150
(sin θ/λ) _{max} (Å ^{−1})	0.604
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.070, 0.215, 1.03
No. of reflections	4458
No. of parameters	315
No. of restraints	161
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ^{−3})	1.06, −1.45
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.50 (9)

Computer programs: *APEX2* (Bruker, 2007), *SAINT* (Bruker, 2007), *SHELXS2014* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

1.5*U*_{eq} of the parent atoms. The crystal of (I) chosen for data collection was found to crystallize as an inversion twin.

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supporting information

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Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE* (Bruker, 2007); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

catena-Poly[[[(perchlorato- $\kappa\kappa$ O)copper(II)]- μ -3-(3-carboxypropyl)-1,5,8,12-tetraaza-3-azoniacyclotetradecane- κ^4 N¹,N²,N⁴,N⁵] bis(perchlorate)]

Crystal data

[Cu(C₁₃H₃₀N₅O₂)(ClO₄)](ClO₄)₂
 $M_r = 650.31$
 Orthorhombic, *Pna*2₁
 $a = 18.990$ (4) Å
 $b = 9.3640$ (19) Å
 $c = 13.636$ (3) Å
 $V = 2424.9$ (8) Å³
 $Z = 4$
 $F(000) = 1340$

$D_x = 1.781$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 4345 reflections
 $\theta = 2.1$ – 24.5°
 $\mu = 1.31$ mm⁻¹
 $T = 100$ K
 Block, violet
 0.18 × 0.14 × 0.12 mm

Data collection

Bruker X8 APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2007)
 $T_{\min} = 0.799$, $T_{\max} = 0.859$
 60778 measured reflections

4458 independent reflections
 2787 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.150$
 $\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 2.2^\circ$
 $h = -22 \rightarrow 22$
 $k = -11 \rightarrow 11$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.070$
 $wR(F^2) = 0.215$
 $S = 1.03$
 4458 reflections
 315 parameters

161 restraints
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1002P)^2 + 10.5107P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 1.06 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.44 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Refined as an inversion twin

Absolute structure parameter: 0.50 (9)

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. Refined as a 2-component inversion twin

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Cu1	0.08430 (6)	0.21078 (13)	0.8220 (4)	0.0290 (4)	
Cl3	0.3273 (4)	0.1883 (8)	1.0262 (4)	0.068 (3)	
Cl2	0.3260 (4)	0.1803 (8)	0.6130 (4)	0.059 (2)	
O7	0.3630 (8)	0.3036 (16)	0.6503 (11)	0.068 (3)	0.8
O8	0.2596 (8)	0.224 (2)	0.5737 (13)	0.068 (3)	0.8
O9	0.3677 (8)	0.1169 (16)	0.5374 (11)	0.068 (3)	0.8
O10	0.3152 (10)	0.0807 (18)	0.6902 (10)	0.068 (3)	0.8
O7X	0.3906 (14)	0.238 (5)	0.578 (3)	0.068 (3)	0.2
O8X	0.275 (2)	0.292 (4)	0.622 (4)	0.068 (3)	0.2
O9X	0.300 (3)	0.076 (4)	0.543 (3)	0.068 (3)	0.2
O10X	0.335 (2)	0.112 (5)	0.7053 (19)	0.068 (3)	0.2
O11	0.2616 (8)	0.2416 (19)	1.0611 (13)	0.067 (3)	0.78
O12	0.3169 (9)	0.0676 (17)	0.9647 (11)	0.067 (3)	0.78
O13	0.3769 (7)	0.1662 (17)	1.1023 (10)	0.067 (3)	0.78
O14	0.3598 (8)	0.2996 (16)	0.9616 (10)	0.067 (3)	0.78
O11X	0.2692 (17)	0.281 (4)	1.051 (3)	0.067 (3)	0.22
O12X	0.311 (2)	0.114 (4)	0.937 (2)	0.067 (3)	0.22
O13X	0.336 (2)	0.085 (4)	1.104 (2)	0.067 (3)	0.22
O14X	0.3902 (16)	0.271 (4)	1.016 (3)	0.067 (3)	0.22
N4	0.1446 (10)	0.116 (2)	0.9306 (14)	0.028 (4)	
H4	0.1868	0.1769	0.9436	0.033*	
N5	0.0237 (12)	0.291 (2)	0.9273 (17)	0.039 (6)	
H5	-0.0196	0.2300	0.9302	0.047*	
N1	0.0240 (11)	0.2873 (19)	0.7134 (16)	0.030 (5)	
H1	-0.0194	0.2268	0.7106	0.036*	
N2	0.1386 (11)	0.1191 (19)	0.7138 (16)	0.031 (5)	
H2	0.1800	0.1829	0.7023	0.038*	
N3	0.2112 (4)	-0.0353 (9)	0.8184 (18)	0.027 (2)	
H3	0.2441	0.0479	0.8184	0.033*	
C5	0.0944 (15)	0.124 (3)	1.0203 (19)	0.041 (7)	
H5A	0.0566	0.0512	1.0139	0.049*	
H5B	0.1209	0.1040	1.0814	0.049*	
C6	0.0638 (16)	0.267 (3)	1.0239 (17)	0.042 (7)	
H6A	0.1014	0.3393	1.0312	0.051*	
H6B	0.0313	0.2752	1.0803	0.051*	

C7	-0.0001 (15)	0.442 (3)	0.914 (2)	0.039 (6)	
H7A	0.0413	0.5058	0.9123	0.046*	
H7B	-0.0304	0.4701	0.9694	0.046*	
C8	-0.0406 (6)	0.4551 (12)	0.819 (3)	0.040 (3)	
H8A	-0.0790	0.3834	0.8197	0.047*	
H8B	-0.0629	0.5506	0.8177	0.047*	
C9	0.0008 (17)	0.436 (3)	0.726 (2)	0.044 (7)	
H9A	-0.0287	0.4647	0.6695	0.053*	
H9B	0.0425	0.4997	0.7277	0.053*	
C1	0.0607 (16)	0.268 (3)	0.622 (2)	0.051 (9)	
H1A	0.0951	0.3467	0.6126	0.061*	
H1B	0.0268	0.2706	0.5668	0.061*	
C2	0.0999 (17)	0.121 (3)	0.625 (2)	0.045 (8)	
H2A	0.0656	0.0410	0.6228	0.054*	
H2B	0.1320	0.1114	0.5677	0.054*	
C3	0.1686 (14)	-0.026 (3)	0.728 (2)	0.036 (6)	
H3A	0.1297	-0.0967	0.7319	0.043*	
H3B	0.1983	-0.0513	0.6711	0.043*	
C4	0.1670 (14)	-0.027 (2)	0.9140 (17)	0.031 (6)	
H4A	0.1254	-0.0903	0.9083	0.037*	
H4B	0.1957	-0.0603	0.9701	0.037*	
C10	0.2553 (5)	-0.1691 (11)	0.821 (3)	0.034 (3)	
H10A	0.2849	-0.1678	0.8808	0.041*	
H10B	0.2871	-0.1697	0.7635	0.041*	
C11	0.2116 (6)	-0.3044 (11)	0.820 (3)	0.037 (3)	
H11A	0.1824	-0.3073	0.7604	0.045*	
H11B	0.1797	-0.3048	0.8778	0.045*	
Cl1	0.4733 (6)	0.6298 (13)	0.8275 (11)	0.049 (2)	0.5
O5	0.4266 (13)	0.681 (3)	0.7565 (17)	0.092 (3)	0.5
O4	0.5273 (11)	0.731 (2)	0.8438 (19)	0.092 (3)	0.5
O3	0.5054 (7)	0.5048 (18)	0.7937 (18)	0.092 (3)	0.5
O6	0.4378 (13)	0.605 (2)	0.9148 (16)	0.092 (3)	0.5
Cl1X	0.4594 (6)	0.5922 (13)	0.8170 (11)	0.049 (2)	0.5
O5X	0.4124 (13)	0.682 (3)	0.8729 (18)	0.092 (3)	0.5
O6X	0.4188 (12)	0.494 (2)	0.7562 (18)	0.092 (3)	0.5
O3X	0.5033 (7)	0.507 (2)	0.8825 (18)	0.092 (3)	0.5
O4X	0.5041 (13)	0.679 (2)	0.7532 (18)	0.092 (3)	0.5
O2	0.1552 (4)	-0.5798 (9)	0.819 (2)	0.052 (2)	
O1	0.2542 (5)	-0.6903 (9)	0.823 (3)	0.085 (5)	
H1C	0.2935	-0.6775	0.7958	0.128*	
C13	0.2181 (6)	-0.5702 (11)	0.821 (3)	0.037 (3)	
C12	0.2587 (6)	-0.4350 (12)	0.824 (3)	0.041 (3)	
H12A	0.2915	-0.4324	0.7675	0.049*	
H12B	0.2871	-0.4323	0.8847	0.049*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0320 (7)	0.0255 (7)	0.0296 (7)	0.0037 (5)	0.0012 (18)	0.0012 (18)
Cl3	0.045 (5)	0.090 (6)	0.069 (5)	0.014 (5)	-0.011 (4)	-0.046 (5)
Cl2	0.050 (5)	0.071 (4)	0.057 (5)	0.008 (4)	0.009 (4)	0.027 (4)
O7	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O8	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O9	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O10	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O7X	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O8X	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O9X	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O10X	0.060 (7)	0.072 (7)	0.071 (6)	0.002 (5)	0.015 (5)	0.023 (5)
O11	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
O12	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
O13	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
O14	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
O11X	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
O12X	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
O13X	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
O14X	0.046 (5)	0.080 (7)	0.074 (6)	-0.006 (5)	0.004 (4)	-0.037 (5)
N4	0.025 (9)	0.038 (11)	0.020 (8)	0.006 (9)	-0.003 (8)	-0.003 (8)
N5	0.033 (13)	0.046 (17)	0.039 (12)	0.002 (10)	-0.007 (11)	0.009 (11)
N1	0.031 (13)	0.017 (12)	0.041 (12)	0.007 (8)	-0.009 (10)	0.006 (9)
N2	0.034 (11)	0.017 (9)	0.043 (11)	-0.004 (8)	-0.007 (9)	-0.004 (9)
N3	0.027 (4)	0.024 (5)	0.031 (5)	0.000 (4)	-0.002 (11)	0.010 (11)
C5	0.053 (16)	0.048 (17)	0.022 (14)	0.013 (13)	0.016 (11)	0.004 (12)
C6	0.064 (19)	0.048 (17)	0.015 (11)	0.004 (14)	-0.005 (11)	-0.012 (10)
C7	0.036 (15)	0.026 (14)	0.054 (16)	0.000 (13)	0.009 (13)	-0.015 (12)
C8	0.033 (6)	0.028 (6)	0.057 (8)	0.011 (5)	-0.008 (17)	0.015 (16)
C9	0.040 (17)	0.032 (15)	0.061 (17)	0.008 (14)	-0.005 (15)	-0.001 (13)
C1	0.042 (16)	0.046 (17)	0.06 (2)	0.020 (13)	-0.021 (14)	-0.008 (14)
C2	0.052 (16)	0.043 (17)	0.039 (18)	0.021 (13)	-0.003 (12)	-0.006 (14)
C3	0.033 (15)	0.031 (14)	0.044 (15)	0.001 (13)	0.000 (12)	-0.003 (12)
C4	0.036 (15)	0.026 (14)	0.030 (13)	0.010 (13)	0.009 (11)	0.007 (11)
C10	0.029 (5)	0.022 (5)	0.052 (7)	0.007 (4)	0.015 (16)	0.002 (17)
C11	0.035 (6)	0.027 (6)	0.050 (7)	-0.001 (5)	-0.006 (17)	-0.017 (15)
Cl1	0.045 (4)	0.057 (6)	0.045 (3)	0.018 (4)	-0.009 (6)	-0.015 (7)
O5	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)
O4	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)
O3	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)
O6	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)
Cl1X	0.045 (4)	0.057 (6)	0.045 (3)	0.018 (4)	-0.009 (6)	-0.015 (7)
O5X	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)
O6X	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)
O3X	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)
O4X	0.089 (6)	0.080 (7)	0.106 (9)	0.026 (6)	0.000 (8)	-0.004 (7)

O2	0.029 (4)	0.032 (5)	0.093 (7)	-0.006 (4)	-0.010 (14)	0.007 (13)
O1	0.046 (5)	0.026 (5)	0.184 (14)	0.005 (4)	-0.035 (18)	0.014 (17)
C13	0.044 (7)	0.020 (6)	0.045 (7)	0.003 (5)	0.018 (16)	0.013 (15)
C12	0.025 (6)	0.035 (6)	0.063 (8)	0.002 (5)	0.017 (16)	0.008 (17)

Geometric parameters (Å, °)

Cu1—N5	1.99 (2)	C6—H6A	0.9900
Cu1—N2	1.99 (2)	C6—H6B	0.9900
Cu1—N1	2.005 (19)	C7—C8	1.51 (4)
Cu1—N4	2.071 (19)	C7—H7A	0.9900
Cu1—O2 ⁱ	2.379 (8)	C7—H7B	0.9900
Cl3—O13	1.417 (12)	C8—C9	1.50 (5)
Cl3—O12	1.422 (12)	C8—H8A	0.9900
Cl3—O11	1.426 (12)	C8—H8B	0.9900
Cl3—O14X	1.429 (15)	C9—H9A	0.9900
Cl3—O12X	1.435 (15)	C9—H9B	0.9900
Cl3—O11X	1.446 (14)	C1—C2	1.57 (4)
Cl3—O13X	1.447 (15)	C1—H1A	0.9900
Cl3—O14	1.497 (13)	C1—H1B	0.9900
Cl2—O10	1.421 (13)	C2—H2A	0.9900
Cl2—O7X	1.424 (15)	C2—H2B	0.9900
Cl2—O10X	1.424 (15)	C3—H3A	0.9900
Cl2—O9	1.430 (13)	C3—H3B	0.9900
Cl2—O8	1.431 (13)	C4—H4A	0.9900
Cl2—O8X	1.437 (15)	C4—H4B	0.9900
Cl2—O7	1.443 (13)	C10—C11	1.514 (14)
Cl2—O9X	1.448 (15)	C10—H10A	0.9900
N4—C4	1.42 (3)	C10—H10B	0.9900
N4—C5	1.55 (3)	C11—C12	1.515 (15)
N4—H4	1.0000	C11—H11A	0.9900
N5—C7	1.50 (3)	C11—H11B	0.9900
N5—C6	1.54 (3)	Cl1—O6	1.387 (17)
N5—H5	1.0000	Cl1—O3	1.398 (17)
N1—C1	1.44 (4)	Cl1—O5	1.399 (17)
N1—C9	1.47 (3)	Cl1—O4	1.411 (17)
N1—H1	1.0000	Cl1X—O5X	1.446 (19)
N2—C2	1.42 (3)	Cl1X—O3X	1.456 (19)
N2—C3	1.49 (3)	Cl1X—O6X	1.461 (19)
N2—H2	1.0000	Cl1X—O4X	1.463 (19)
N3—C3	1.47 (3)	O2—C13	1.198 (13)
N3—C10	1.507 (13)	O2—Cu1 ⁱⁱ	2.379 (8)
N3—C4	1.55 (3)	O1—C13	1.318 (13)
N3—H3	1.0000	O1—H1C	0.8400
C5—C6	1.46 (4)	C13—C12	1.483 (16)
C5—H5A	0.9900	C12—H12A	0.9900
C5—H5B	0.9900	C12—H12B	0.9900

N5—Cu1—N2	175.2 (9)	N5—C7—H7A	109.7
N5—Cu1—N1	93.9 (4)	C8—C7—H7A	109.7
N2—Cu1—N1	84.4 (9)	N5—C7—H7B	109.7
N5—Cu1—N4	88.0 (9)	C8—C7—H7B	109.7
N2—Cu1—N4	93.4 (4)	H7A—C7—H7B	108.2
N1—Cu1—N4	175.6 (8)	C9—C8—C7	116.4 (10)
N5—Cu1—O2 ⁱ	91.8 (9)	C9—C8—H8A	108.2
N2—Cu1—O2 ⁱ	92.8 (8)	C7—C8—H8A	108.2
N1—Cu1—O2 ⁱ	90.8 (8)	C9—C8—H8B	108.2
N4—Cu1—O2 ⁱ	93.1 (8)	C7—C8—H8B	108.2
O13—C13—O12	114.1 (7)	H8A—C8—H8B	107.3
O13—C13—O11	112.8 (7)	N1—C9—C8	112 (2)
O12—C13—O11	110.7 (7)	N1—C9—H9A	109.3
O14X—C13—O12X	110.8 (9)	C8—C9—H9A	109.3
O14X—C13—O11X	109.5 (9)	N1—C9—H9B	109.3
O12X—C13—O11X	109.1 (9)	C8—C9—H9B	109.3
O14X—C13—O13X	109.6 (9)	H9A—C9—H9B	108.0
O12X—C13—O13X	109.0 (9)	N1—C1—C2	109 (2)
O11X—C13—O13X	108.7 (9)	N1—C1—H1A	110.0
O13—C13—O14	105.1 (7)	C2—C1—H1A	110.0
O12—C13—O14	105.3 (7)	N1—C1—H1B	110.0
O11—C13—O14	108.3 (7)	C2—C1—H1B	110.0
O7X—C12—O10X	111.1 (9)	H1A—C1—H1B	108.3
O10—C12—O9	110.0 (7)	N2—C2—C1	106 (2)
O10—C12—O8	109.8 (7)	N2—C2—H2A	110.6
O9—C12—O8	109.7 (7)	C1—C2—H2A	110.6
O7X—C12—O8X	109.7 (9)	N2—C2—H2B	110.6
O10X—C12—O8X	110.0 (9)	C1—C2—H2B	110.6
O10—C12—O7	109.5 (7)	H2A—C2—H2B	108.7
O9—C12—O7	108.5 (7)	N3—C3—N2	111.8 (18)
O8—C12—O7	109.4 (7)	N3—C3—H3A	109.2
O7X—C12—O9X	108.9 (9)	N2—C3—H3A	109.2
O10X—C12—O9X	108.6 (9)	N3—C3—H3B	109.2
O8X—C12—O9X	108.4 (9)	N2—C3—H3B	109.2
C4—N4—C5	110.6 (19)	H3A—C3—H3B	107.9
C4—N4—Cu1	117.0 (15)	N4—C4—N3	110.0 (17)
C5—N4—Cu1	101.8 (14)	N4—C4—H4A	109.7
C4—N4—H4	109.0	N3—C4—H4A	109.7
C5—N4—H4	109.0	N4—C4—H4B	109.7
Cu1—N4—H4	109.0	N3—C4—H4B	109.7
C7—N5—C6	113 (2)	H4A—C4—H4B	108.2
C7—N5—Cu1	116.2 (19)	N3—C10—C11	113.0 (8)
C6—N5—Cu1	106.1 (15)	N3—C10—H10A	109.0
C7—N5—H5	107.0	C11—C10—H10A	109.0
C6—N5—H5	107.0	N3—C10—H10B	109.0
Cu1—N5—H5	107.0	C11—C10—H10B	109.0
C1—N1—C9	111 (2)	H10A—C10—H10B	107.8
C1—N1—Cu1	108.6 (16)	C10—C11—C12	110.6 (9)

C9—N1—Cu1	115.0 (19)	C10—C11—H11A	109.5
C1—N1—H1	107.2	C12—C11—H11A	109.5
C9—N1—H1	107.2	C10—C11—H11B	109.5
Cu1—N1—H1	107.2	C12—C11—H11B	109.5
C2—N2—C3	108.6 (19)	H11A—C11—H11B	108.1
C2—N2—Cu1	111.3 (16)	O6—C11—O3	110.9 (9)
C3—N2—Cu1	119.6 (17)	O6—C11—O5	110.0 (8)
C2—N2—H2	105.4	O3—C11—O5	109.7 (9)
C3—N2—H2	105.4	O6—C11—O4	109.2 (9)
Cu1—N2—H2	105.4	O3—C11—O4	107.1 (8)
C3—N3—C10	112 (2)	O5—C11—O4	109.8 (8)
C3—N3—C4	113.6 (8)	O5X—C11X—O3X	110.4 (8)
C10—N3—C4	109 (2)	O5X—C11X—O6X	110.0 (8)
C3—N3—H3	107.4	O3X—C11X—O6X	107.8 (8)
C10—N3—H3	107.4	O5X—C11X—O4X	110.3 (8)
C4—N3—H3	107.4	O3X—C11X—O4X	109.7 (8)
C6—C5—N4	108 (2)	O6X—C11X—O4X	108.7 (8)
C6—C5—H5A	110.1	C13—O2—Cu1 ⁱⁱ	128.7 (7)
N4—C5—H5A	110.1	C13—O1—H1C	109.5
C6—C5—H5B	110.1	O2—C13—O1	117.1 (10)
N4—C5—H5B	110.1	O2—C13—C12	125.7 (10)
H5A—C5—H5B	108.4	O1—C13—C12	117.2 (10)
C5—C6—N5	108 (2)	C13—C12—C11	112.4 (9)
C5—C6—H6A	110.2	C13—C12—H12A	109.1
N5—C6—H6A	110.2	C11—C12—H12A	109.1
C5—C6—H6B	110.2	C13—C12—H12B	109.1
N5—C6—H6B	110.2	C11—C12—H12B	109.1
H6A—C6—H6B	108.5	H12A—C12—H12B	107.8
N5—C7—C8	110 (2)		
C4—N4—C5—C6	-171 (2)	C10—N3—C3—N2	-166.3 (16)
Cu1—N4—C5—C6	-45 (2)	C4—N3—C3—N2	70.0 (19)
N4—C5—C6—N5	60 (3)	C2—N2—C3—N3	178 (2)
C7—N5—C6—C5	-170 (2)	Cu1—N2—C3—N3	-52 (2)
Cu1—N5—C6—C5	-42 (3)	C5—N4—C4—N3	174.6 (18)
C6—N5—C7—C8	-178 (2)	Cu1—N4—C4—N3	59 (2)
Cu1—N5—C7—C8	58 (3)	C3—N3—C4—N4	-75.1 (19)
N5—C7—C8—C9	-68 (2)	C10—N3—C4—N4	159.7 (17)
C1—N1—C9—C8	177 (2)	C3—N3—C10—C11	-62 (3)
Cu1—N1—C9—C8	-59 (3)	C4—N3—C10—C11	64 (3)
C7—C8—C9—N1	69 (2)	N3—C10—C11—C12	-180 (3)
C9—N1—C1—C2	166 (2)	Cu1 ⁱⁱ —O2—C13—O1	2 (6)
Cu1—N1—C1—C2	38 (3)	Cu1 ⁱⁱ —O2—C13—C12	-177 (3)
C3—N2—C2—C1	172 (2)	O2—C13—C12—C11	-2 (6)
Cu1—N2—C2—C1	38 (3)	O1—C13—C12—C11	179 (3)
N1—C1—C2—N2	-51 (3)	C10—C11—C12—C13	-179 (3)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots O8	1.00	2.35	3.15 (3)	136
N3—H3 \cdots O10	1.00	2.23	2.85 (2)	119
N3—H3 \cdots O12	1.00	2.43	2.99 (2)	115
N4—H4 \cdots O11	1.00	2.23	3.08 (3)	143
O1—H1C \cdots O7 ⁱⁱ	0.84	2.39	3.13 (4)	147
N2—H2 \cdots O1 ⁱ	1.00	2.47	3.20 (3)	129
N4—H4 \cdots O1 ⁱ	1.00	2.43	3.13 (3)	126
N1—H1 \cdots O7 ⁱⁱⁱ	1.00	2.40	3.29 (3)	148
N5—H5 \cdots O14 ⁱⁱⁱ	1.00	2.35	3.26 (3)	151
C2—H2A \cdots O3 ⁱⁱⁱ	0.99	2.63	3.15 (4)	113
C2—H2B \cdots O8	0.99	2.64	3.26 (4)	120
C3—H3A \cdots O3 ⁱⁱⁱ	0.99	2.65	3.23 (3)	118
C3—H3B \cdots O11 ^{iv}	0.99	2.57	3.42 (3)	144
C4—H4A \cdots O4 ⁱⁱⁱ	0.99	2.44	3.40 (3)	163
C4—H4B \cdots O8 ^v	0.99	2.61	3.48 (3)	147
C5—H5B \cdots O5 ^v	0.99	2.65	3.29 (3)	122
C7—H7A \cdots O2 ⁱ	0.99	2.64	3.23 (3)	118
C7—H7A \cdots O4 ^{vi}	0.99	2.65	3.26 (3)	120
C7—H7A \cdots O9 ^{vii}	0.99	2.64	3.44 (3)	138
C8—H8B \cdots O5 ^{vi}	0.99	2.65	3.56 (3)	153
C9—H9B \cdots O2 ⁱ	0.99	2.58	3.20 (3)	120
C10—H10B \cdots O10	0.99	2.60	3.15 (3)	115
C10—H10A \cdots O12	0.99	2.56	3.18 (3)	121
C11—H11B \cdots O9 ^v	0.99	2.47	3.40 (4)	157
C11—H11A \cdots O13 ^{iv}	0.99	2.44	3.43 (3)	172
N1—H1 \cdots O6X ⁱⁱⁱ	1.00	2.46	3.36 (3)	149
N5—H5 \cdots O3X ⁱⁱⁱ	1.00	2.36	2.88 (3)	112
C3—H3A \cdots O4X ⁱⁱⁱ	0.99	2.52	3.45 (4)	156
C4—H4A \cdots O3X ⁱⁱⁱ	0.99	2.47	3.14 (3)	125
C5—H5A \cdots O3X ⁱⁱⁱ	0.99	2.13	2.83 (4)	127
C6—H6B \cdots O4X ^v	0.99	2.61	3.48 (4)	147
C8—H8B \cdots O5X ^{vi}	0.99	2.65	3.59 (3)	157
C12—H12B \cdots O5X ⁱⁱ	0.99	2.62	3.19 (3)	117
C12—H12A \cdots O6X ⁱⁱ	0.99	2.52	3.25 (3)	130

Symmetry codes: (i) $x, y+1, z$; (ii) $x, y-1, z$; (iii) $x-1/2, -y+1/2, z$; (iv) $-x+1/2, y-1/2, z-1/2$; (v) $-x+1/2, y-1/2, z+1/2$; (vi) $x-1/2, -y+3/2, z$; (vii) $-x+1/2, y+1/2, z+1/2$.