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# Diaqua[$\mu$-11,23-di-*tert*-butyl-3,7,15,19-tetraazatricyclo[19.3.1.1$^{9,13}$]tetracosa-1(25),2,6,9,11,13(26),14,19,21,23-dodecaene-25,26-diolato-$\kappa^4N^3,N^7,O^{25},O^{26}$:$\kappa^4N^{15},N^{19},O^{25},O^{26}$]dicopper(II) bis(perchlorate)

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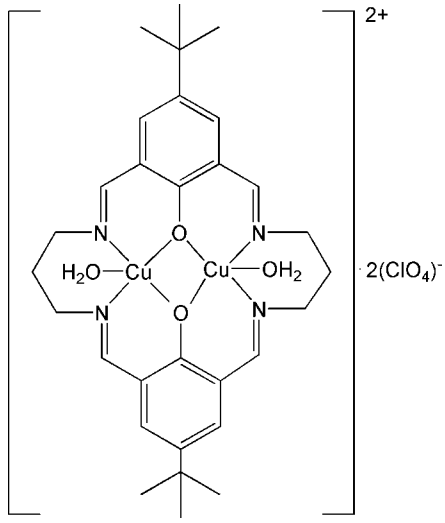
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.006 Å; disorder in main residue; $R$ factor = 0.039; $wR$ factor = 0.115; data-to-parameter ratio = 11.7.

In the dinuclear title complex, $[Cu_2(C_{30}H_{38}N_4O_2)(H_2O)_2](ClO_4)_2$, the coordination cation has crystallographically imposed twofold rotational symmetry. The Cu$^{II}$ ion is five-coordinated by two N and two O atoms from the macrocylic ligand and one O atom from a water molecule, forming a square-pyramidal $N_2O_3$ geometry with the water molecule in the apical position. The distance between the two Cu$^{II}$ atoms is 3.0930 (5) Å. Hydrogen bonds between water molecules and between water molecules and perchlorate anions assemble two cations and four anions into discrete supermolecules of $S_4$ symmetry. Intramolecular O—H···N hydrogen bonds are also observed. The perchlorate anion and the *tert*-butyl group are disordered over two positions, with occupancies of the major positions of 0.527 (11) and 0.592 (9), respectively.

## Related literature

For the synthesis of the magnesium precursor, see: Mohanta *et al.* (1997). For the synthesis of 4-*tert*-butyl-2,6-diformylphenol, see: Lindoy *et al.* (1998). For similar copper(II) and nickel(II) complexes, see: Bai *et al.* (2007); Chen *et al.* (2005); Nanda *et al.* (1994). For the preparation of similar macrocyclic ligands, see: Thompson *et al.* (1996); Pilkington & Robson (1970); Zhou *et al.* (2005).




## Experimental

### Crystal data

$[Cu_2(C_{30}H_{38}N_4O_2)(H_2O)_2](ClO_4)_2$
$M_r$ = 848.66
Tetragonal, $P\bar{4}2_1c$
$a$ = 18.9013 (4) Å
$c$ = 9.9174 (4) Å
$V$ = 3543.08 (18) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.42 mm$^{-1}$
$T$ = 296 K
0.38 × 0.36 × 0.32 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.615, $T_{max}$ = 0.660
19006 measured reflections
3489 independent reflections
2942 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
$S$ = 1.02
3489 reflections
298 parameters
125 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$
Absolute structure: Flack (1983), 1527 Friedel pairs
Flack parameter: 0.31 (3)

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

| | | | |
|---|---|---|---|
| Cu1—N1 | 1.939 (4) | Cu1—O1 | 1.964 (3) |
| Cu1—N2 | 1.945 (4) | Cu1—O2 | 2.707 (5) |
| Cu1—O1$^{i}$ | 1.954 (3) | | |

Symmetry code: (i) $-x, -y+2, z$.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2$B$···O2$^{ii}$ | 0.82 (2) | 2.07 (4) | 2.821 (6) | 153 (8) |
| O2—H2$A$···O3 | 0.81 (2) | 2.26 (3) | 2.806 (8) | 125 (2) |
| O2—H2$A$···O3′ | 0.81 (2) | 2.49 (4) | 2.947 (10) | 117 (3) |
| O2—H2$A$···N1 | 0.81 (2) | 2.55 (3) | 3.197 (6) | 138 (3) |

Symmetry code: (ii) $y-1, -x+1, -z+1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 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: GK2504).

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# supporting information

*Acta Cryst.* (2012). E**68**, m1060–m1061 [https://doi.org/10.1107/S1600536812031248]

## Diaqua[$\mu$-11,23-di-*tert*-butyl-3,7,15,19-tetraazatricyclo-[19.3.1.1$^{9,13}$]tetracosa-1(25),2,6,9,11,13(26),14,19,21,23-dodecaene-25,26-diolato-$\kappa^4N^3,N^7,O^{25},O^{26}$:$\kappa^4N^{15},N^{19},O^{25},O^{26}$]dicopper(II) bis(perchlorate)

**Qiang Xu, Zhaodong Wang, Jiahong He, Zhongrong Song, Fengming Chen, Jiangping Meng and Chengbo Hu**

### S1. Comment

Dinuclear heterometallic and homometallic transition metal complexes have been well studied with a series of macrocyclic liagnds based on the first reported condensation reaction between 2,6-diformyl-4-R-phenol (*R*= $CH_3$, Cl, F, *n*-butyl) and alkylenediamine by stepwise template reaction (Thompson *et al.*, 1996; Pilkington & Robson, 1970; Zhou *et al.*, 2005). Several tetranuclear as well as trinuclear nickel(II) and copper(II) complexes have been structurally characterized (Mohanta *et al.*,1997; Nanda *et al.*,1994). In addition, Mohanta *et al.* (1997) reported a protonated macrocyclic magnesium compound of composition $[Mg_2(L^1H_4)_2(NO_3)_2](NO_3)_26H_2O$ by a template reaction. The transmetalation reaction of the magnesium precursor with copper(II) perchlorate in the presence of triethylamine resulted in the formation of a dinuclear copper(II) complex (Mohanta *et al.*,1997). Herein, we synthesized a similar magnesium precursor by a template reaction involving 4-*tert*-butyl-2,6-diformylphenol, 1,3-diaminopropane, magnesium acetate, and magnesium nitrate.The transmetalation reaction of the new magnesium precursor with copper(II) perchlorate leads to a new dinuclear copper(II) complex.

The structure of the cation the title compound is shown in Fig.1. In the cation, each copper(II) is coordinated by two O atoms and two N atoms from the macrocylic ligand and one O from water molecule, forming a square pyramidal $\{N_2O_3\}$ geometry. In $\{N_2O_3\}$, the $N_2O_2$ donor sets from the macrocyclic ligand occupy the basal plane of the pyramid and the O atom from the water molecule locates in the apical position. The distance of the apical O atom and the copper atom [Cu1–O2: 2.707 (5) Å] is longer than the basal donors [ranging from 1.938 (4) to 1.964 (3) Å] due to the Jahn-Teller effect. Fig. 2 shows the crystal packing of the title compound along the *b* axis.

### S2. Experimental

4-*tert*-Butyl-2,6-diformylphenol was synthesized according to the reported literature (Lindoy *et al.*,1998). The magnesium precursor was prepared according to the reported method by Mohanta (Mohanta *et al.*,1997). The title complex was obtained by the following procedures: $Cu(ClO_4)_26H_2O$ (0.185 g, 0.5 mmol) and magnesium precursor (0.136 g, 0.1 mmol) were dissolved in $CH_3OH$ (15 ml) and the solution was filtered and left to stand at room temperature. Blue block single crystals suitable for X-ray analysis were obtained by slow evaporation over a period of two weeks.

### S3. Refinement

The H atoms bonded to C atoms were positioned geometrically and refined using a riding model, with C–H = 0.93-0.96 Å, and with $U_{iso}$(H) = 1.2 or 1.5 times $U_{eq}$(C). The H atoms bonded to O1W were located in Fourier difference maps and

refined with restraints imposed on O-H and H···H distances [O-H= 0.83 (1) Å, H···H. 1.35 (1) Å]. Restraints were also imposed on Cl-O, O···O, C-C and C···C distances of disordered perchlorate and tert-butyl groups [Cl-O 1.44 (1) Å, O···O 2.35 (1) Å, C-C 1. 50 (1) Å, C···C 2.35 (1) Å]. The crystal was refined as an inversion twin with the ratio of the two twin domains of 0.31 (3):0.69 (3).

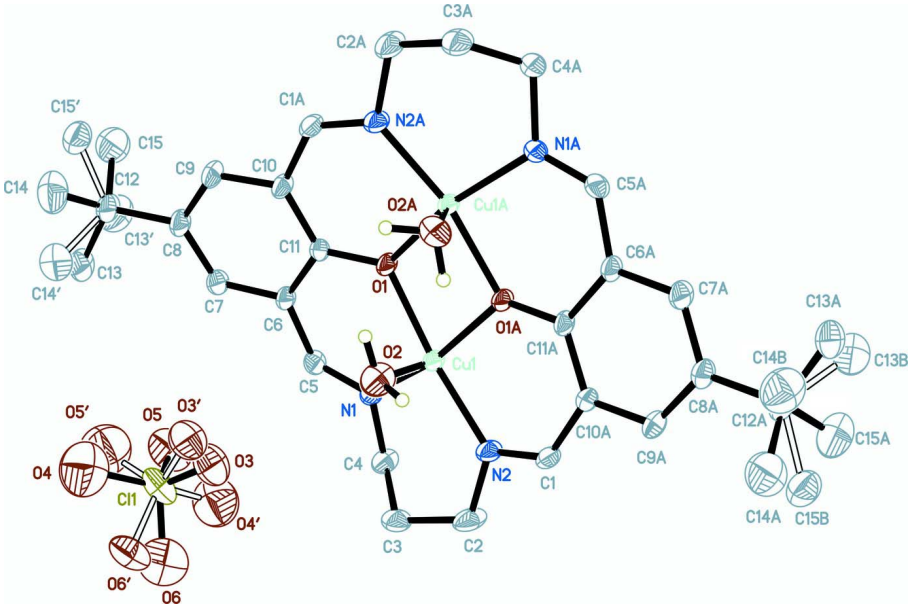


**Figure 1**

Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. The H atoms attached to C atoms were omitted for clarity. Atoms with the A label are generated by the ′-x, -y+2, z′ symmetry operation. *tert*-Butyl group and perchlorate anion are disordered.

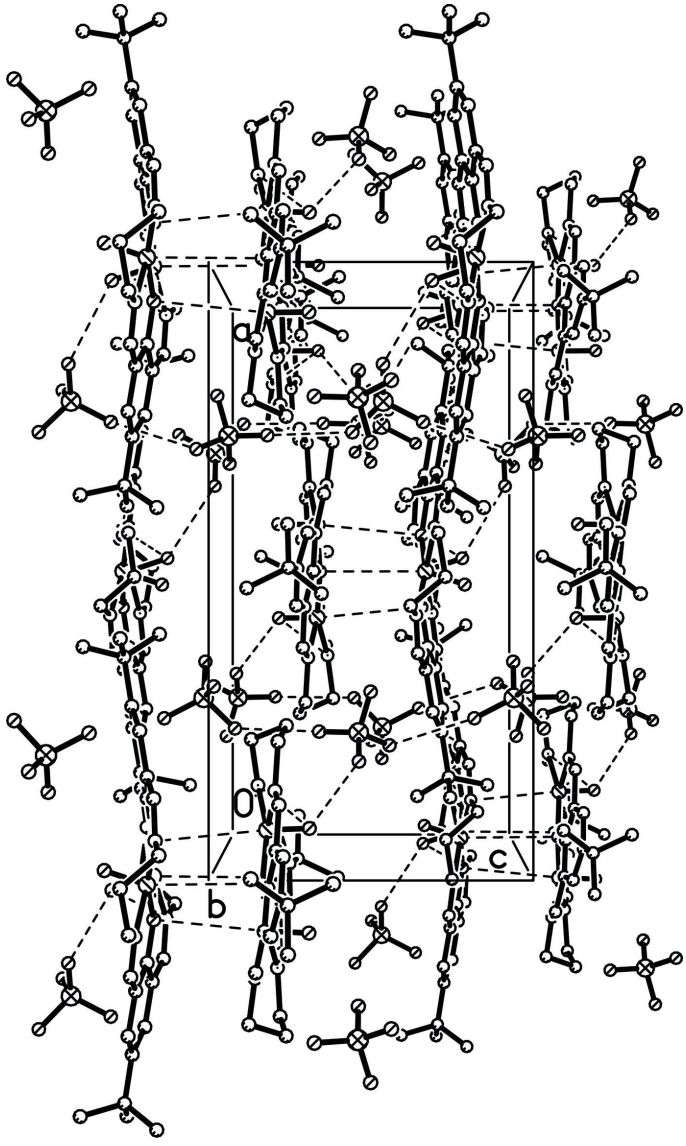


**Figure 2**

View of the crystal packing along the *b* axis. For the sake of clarity, H atoms and minor position of the disordered groups have been omitted.

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*Crystal data*

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$c$ = 9.9174 (4) Å
$V$ = 3543.08 (18) Å$^3$
$Z$ = 4
$F(000)$ = 1752
$D_x$ = 1.591 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 7958 reflections
$\theta$ = 2.3–29.4°
$\mu$ = 1.42 mm$^{-1}$

$T$ = 296 K
Block, blue

0.38 × 0.36 × 0.32 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.615, $T_{max}$ = 0.660

19006 measured reflections
3489 independent reflections
2942 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 1.5°
$h$ = −18→23
$k$ = −22→23
$l$ = −12→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
$S$ = 1.02
3489 reflections
298 parameters
125 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 4.6836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$
Absolute structure: Flack (1983), 1527 Friedel pairs
Absolute structure parameter: 0.31 (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.

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cu1 | 0.08182 (2) | 0.99986 (3) | 0.18030 (5) | 0.03063 (16) | |
| O1 | −0.00045 (19) | 0.93667 (12) | 0.1690 (3) | 0.0329 (6) | |
| O2 | 0.0833 (2) | 0.9539 (2) | 0.4388 (5) | 0.0614 (10) | |
| N1 | 0.14955 (18) | 0.92478 (19) | 0.1474 (4) | 0.0314 (9) | |
| N2 | 0.14970 (19) | 1.0758 (2) | 0.2082 (4) | 0.0389 (10) | |
| C1 | 0.1312 (2) | 1.1411 (2) | 0.2233 (5) | 0.0361 (10) | |
| H1 | 0.1678 | 1.1728 | 0.2406 | 0.043* | |
| C2 | 0.2279 (3) | 1.0650 (3) | 0.2171 (8) | 0.0657 (19) | |
| H2A′ | 0.2493 | 1.0819 | 0.1343 | 0.079* | |
| H2B′ | 0.2461 | 1.0936 | 0.2904 | 0.079* | |
| C3 | 0.2500 (2) | 0.9908 (3) | 0.2390 (6) | 0.0504 (13) | |
| H3A | 0.2311 | 0.9744 | 0.3244 | 0.061* | |
| H3B | 0.3012 | 0.9889 | 0.2446 | 0.061* | |
| C4 | 0.2254 (2) | 0.9415 (3) | 0.1278 (5) | 0.0431 (11) | |
| H4A | 0.2322 | 0.9639 | 0.0408 | 0.052* | |
| H4B | 0.2530 | 0.8983 | 0.1296 | 0.052* | |

| | | | | | |
|---|---|---|---|---|---|
| C5 | 0.1332 (2) | 0.8591 (2) | 0.1536 (5) | 0.0347 (10) | |
| H5 | 0.1698 | 0.8272 | 0.1384 | 0.042* | |
| C6 | 0.0651 (2) | 0.8288 (2) | 0.1813 (5) | 0.0318 (9) | |
| C7 | 0.0649 (2) | 0.7551 (2) | 0.2042 (5) | 0.0398 (11) | |
| H7 | 0.1073 | 0.7305 | 0.1974 | 0.048* | |
| C8 | 0.0046 (3) | 0.71818 (19) | 0.2360 (4) | 0.0393 (9) | |
| C9 | −0.0578 (3) | 0.7557 (2) | 0.2404 (5) | 0.0394 (11) | |
| H9 | −0.0995 | 0.7316 | 0.2599 | 0.047* | |
| C10 | −0.0611 (3) | 0.8288 (2) | 0.2168 (5) | 0.0330 (11) | |
| C11 | 0.0005 (3) | 0.86673 (17) | 0.1894 (4) | 0.0297 (7) | |
| C12 | 0.0062 (3) | 0.6385 (2) | 0.2598 (5) | 0.0538 (12) | |
| C13 | 0.0775 (4) | 0.6074 (4) | 0.2618 (11) | 0.059 (3) | 0.592 (9) |
| H13A | 0.0741 | 0.5574 | 0.2771 | 0.089* | 0.592 (9) |
| H13B | 0.1047 | 0.6287 | 0.3328 | 0.089* | 0.592 (9) |
| H13C | 0.1004 | 0.6159 | 0.1768 | 0.089* | 0.592 (9) |
| C14 | −0.0277 (6) | 0.6227 (6) | 0.3911 (9) | 0.085 (4) | 0.592 (9) |
| H14A | −0.0269 | 0.5725 | 0.4067 | 0.127* | 0.592 (9) |
| H14B | −0.0759 | 0.6389 | 0.3898 | 0.127* | 0.592 (9) |
| H14C | −0.0024 | 0.6463 | 0.4619 | 0.127* | 0.592 (9) |
| C15 | −0.0368 (6) | 0.6024 (6) | 0.1554 (11) | 0.086 (4) | 0.592 (9) |
| H15A | −0.0357 | 0.5522 | 0.1705 | 0.129* | 0.592 (9) |
| H15B | −0.0178 | 0.6127 | 0.0678 | 0.129* | 0.592 (9) |
| H15C | −0.0848 | 0.6188 | 0.1603 | 0.129* | 0.592 (9) |
| C13′ | 0.0409 (8) | 0.6032 (9) | 0.1429 (13) | 0.084 (6) | 0.408 (9) |
| H13D | 0.0419 | 0.5530 | 0.1577 | 0.126* | 0.408 (9) |
| H13E | 0.0883 | 0.6206 | 0.1335 | 0.126* | 0.408 (9) |
| H13F | 0.0147 | 0.6132 | 0.0622 | 0.126* | 0.408 (9) |
| C14′ | 0.0491 (8) | 0.6226 (10) | 0.3793 (13) | 0.085 (6) | 0.408 (9) |
| H14D | 0.0500 | 0.5724 | 0.3938 | 0.127* | 0.408 (9) |
| H14E | 0.0289 | 0.6455 | 0.4567 | 0.127* | 0.408 (9) |
| H14F | 0.0964 | 0.6395 | 0.3657 | 0.127* | 0.408 (9) |
| C15′ | −0.0633 (5) | 0.6048 (7) | 0.2754 (15) | 0.064 (4) | 0.408 (9) |
| H15D | −0.0572 | 0.5550 | 0.2896 | 0.096* | 0.408 (9) |
| H15E | −0.0908 | 0.6124 | 0.1953 | 0.096* | 0.408 (9) |
| H15F | −0.0873 | 0.6251 | 0.3514 | 0.096* | 0.408 (9) |
| Cl1 | 0.22950 (8) | 0.78567 (9) | 0.46056 (14) | 0.0669 (4) | |
| O3 | 0.1844 (6) | 0.8458 (5) | 0.4620 (12) | 0.086 (4) | 0.527 (11) |
| O4 | 0.2055 (8) | 0.7328 (7) | 0.5596 (13) | 0.160 (7) | 0.527 (11) |
| O5 | 0.2295 (6) | 0.7511 (6) | 0.3334 (8) | 0.102 (4) | 0.527 (11) |
| O6 | 0.2988 (5) | 0.8071 (8) | 0.4988 (16) | 0.153 (6) | 0.527 (11) |
| O3′ | 0.1632 (4) | 0.8212 (7) | 0.4726 (13) | 0.082 (4) | 0.473 (11) |
| O4′ | 0.2672 (7) | 0.8119 (8) | 0.3419 (11) | 0.142 (7) | 0.473 (11) |
| O5′ | 0.2189 (8) | 0.7114 (4) | 0.4411 (19) | 0.164 (7) | 0.473 (11) |
| O6′ | 0.2725 (5) | 0.7960 (6) | 0.5766 (10) | 0.084 (4) | 0.473 (11) |
| H2B | 0.054 (4) | 0.932 (4) | 0.482 (8) | 0.101* | |
| H2A | 0.115 (3) | 0.937 (3) | 0.395 (3) | 0.101* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cu1 | 0.0211 (2) | 0.0202 (2) | 0.0506 (3) | 0.0005 (3) | −0.00050 (19) | −0.0018 (3) |
| O1 | 0.0256 (12) | 0.0176 (11) | 0.0556 (16) | 0.0010 (15) | 0.002 (2) | 0.0020 (10) |
| O2 | 0.051 (2) | 0.056 (2) | 0.078 (3) | 0.0051 (18) | 0.013 (2) | −0.008 (2) |
| N1 | 0.0249 (17) | 0.0275 (18) | 0.042 (2) | 0.0022 (15) | 0.0023 (15) | −0.0005 (15) |
| N2 | 0.0228 (17) | 0.031 (2) | 0.063 (3) | −0.0002 (15) | −0.0023 (17) | −0.0026 (18) |
| C1 | 0.030 (2) | 0.027 (2) | 0.052 (3) | −0.0061 (17) | −0.004 (2) | −0.0028 (19) |
| C2 | 0.026 (2) | 0.037 (3) | 0.134 (6) | 0.001 (2) | −0.013 (3) | −0.016 (3) |
| C3 | 0.027 (2) | 0.049 (3) | 0.076 (3) | 0.002 (2) | −0.009 (2) | −0.003 (3) |
| C4 | 0.030 (2) | 0.033 (2) | 0.066 (3) | 0.0016 (19) | 0.006 (2) | −0.006 (2) |
| C5 | 0.029 (2) | 0.029 (2) | 0.046 (3) | 0.0074 (18) | 0.0041 (19) | 0.0023 (19) |
| C6 | 0.034 (2) | 0.019 (2) | 0.043 (2) | 0.0015 (17) | −0.001 (2) | 0.0013 (19) |
| C7 | 0.036 (2) | 0.024 (2) | 0.060 (3) | 0.0062 (19) | −0.002 (2) | 0.003 (2) |
| C8 | 0.046 (2) | 0.0224 (17) | 0.049 (2) | 0.001 (2) | 0.000 (2) | 0.0035 (16) |
| C9 | 0.047 (3) | 0.022 (2) | 0.049 (3) | −0.006 (2) | 0.004 (2) | 0.0032 (19) |
| C10 | 0.034 (3) | 0.022 (2) | 0.043 (3) | −0.0023 (17) | 0.0053 (19) | 0.0008 (17) |
| C11 | 0.0305 (17) | 0.0183 (15) | 0.040 (2) | 0.002 (2) | −0.004 (2) | 0.0004 (14) |
| C12 | 0.054 (3) | 0.0230 (18) | 0.085 (3) | 0.001 (2) | −0.005 (3) | 0.010 (2) |
| C13 | 0.062 (4) | 0.031 (4) | 0.085 (5) | 0.013 (3) | 0.003 (4) | 0.010 (3) |
| C14 | 0.097 (6) | 0.066 (5) | 0.091 (6) | 0.013 (4) | 0.012 (4) | 0.020 (4) |
| C15 | 0.095 (6) | 0.061 (5) | 0.102 (6) | −0.006 (4) | −0.015 (5) | −0.001 (4) |
| C13′ | 0.092 (8) | 0.068 (7) | 0.092 (8) | −0.002 (5) | 0.011 (5) | −0.008 (5) |
| C14′ | 0.088 (7) | 0.075 (7) | 0.091 (7) | −0.001 (5) | −0.008 (5) | 0.015 (5) |
| C15′ | 0.070 (6) | 0.048 (6) | 0.075 (6) | −0.007 (4) | 0.002 (5) | 0.007 (4) |
| Cl1 | 0.0723 (9) | 0.0795 (10) | 0.0488 (7) | 0.0325 (8) | −0.0097 (7) | −0.0036 (7) |
| O3 | 0.088 (5) | 0.075 (5) | 0.095 (5) | 0.031 (4) | −0.004 (4) | 0.003 (4) |
| O4 | 0.169 (8) | 0.154 (8) | 0.157 (8) | 0.000 (5) | 0.014 (5) | 0.012 (5) |
| O5 | 0.105 (6) | 0.110 (6) | 0.091 (5) | 0.033 (4) | 0.002 (4) | −0.021 (4) |
| O6 | 0.142 (8) | 0.159 (8) | 0.157 (8) | −0.004 (5) | −0.012 (5) | 0.004 (5) |
| O3′ | 0.076 (6) | 0.082 (6) | 0.087 (6) | 0.019 (4) | 0.002 (4) | −0.001 (4) |
| O4′ | 0.137 (8) | 0.151 (8) | 0.137 (8) | 0.011 (5) | 0.013 (5) | 0.013 (5) |
| O5′ | 0.169 (9) | 0.151 (8) | 0.173 (9) | −0.002 (5) | 0.002 (5) | −0.011 (5) |
| O6′ | 0.072 (5) | 0.098 (6) | 0.082 (5) | 0.022 (4) | −0.026 (4) | −0.008 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cu1—N1 | 1.939 (4) | C10—C1$^{i}$ | 1.444 (6) |
| Cu1—N2 | 1.945 (4) | C12—C15′ | 1.467 (8) |
| Cu1—O1$^{i}$ | 1.954 (3) | C12—C14′ | 1.468 (8) |
| Cu1—O1 | 1.964 (3) | C12—C13 | 1.471 (7) |
| Cu1—O2 | 2.707 (5) | C12—C14 | 1.482 (7) |
| O1—C11 | 1.338 (4) | C12—C15 | 1.482 (8) |
| O1—Cu1$^{i}$ | 1.954 (3) | C12—C13′ | 1.489 (9) |
| O2—H2B | 0.82 (2) | C13—H13A | 0.9600 |
| O2—H2A | 0.81 (2) | C13—H13B | 0.9600 |
| N1—C5 | 1.282 (6) | C13—H13C | 0.9600 |

| | | | |
|---|---|---|---|
| N1—C4 | 1.481 (6) | C14—H14A | 0.9600 |
| N2—C1 | 1.291 (6) | C14—H14B | 0.9600 |
| N2—C2 | 1.494 (6) | C14—H14C | 0.9600 |
| C1—C10$^{i}$ | 1.444 (6) | C15—H15A | 0.9600 |
| C1—H1 | 0.9300 | C15—H15B | 0.9600 |
| C2—C3 | 1.480 (7) | C15—H15C | 0.9600 |
| C2—H2A′ | 0.9700 | C13′—H13D | 0.9600 |
| C2—H2B′ | 0.9700 | C13′—H13E | 0.9600 |
| C3—C4 | 1.516 (7) | C13′—H13F | 0.9600 |
| C3—H3A | 0.9700 | C14′—H14D | 0.9600 |
| C3—H3B | 0.9700 | C14′—H14E | 0.9600 |
| C4—H4A | 0.9700 | C14′—H14F | 0.9600 |
| C4—H4B | 0.9700 | C15′—H15D | 0.9600 |
| C5—C6 | 1.435 (6) | C15′—H15E | 0.9600 |
| C5—H5 | 0.9300 | C15′—H15F | 0.9600 |
| C6—C7 | 1.411 (6) | Cl1—O5 | 1.421 (6) |
| C6—C11 | 1.418 (6) | Cl1—O3 | 1.421 (6) |
| C7—C8 | 1.372 (7) | Cl1—O6′ | 1.422 (7) |
| C7—H7 | 0.9300 | Cl1—O6 | 1.422 (7) |
| C8—C9 | 1.378 (7) | Cl1—O3′ | 1.426 (7) |
| C8—C12 | 1.525 (5) | Cl1—O5′ | 1.431 (7) |
| C9—C10 | 1.403 (6) | Cl1—O4′ | 1.462 (8) |
| C9—H9 | 0.9300 | Cl1—O4 | 1.472 (8) |
| C10—C11 | 1.395 (7) | | |

| | | | |
|---|---|---|---|
| N1—Cu1—N2 | 97.38 (14) | C13—C12—C14 | 107.7 (6) |
| N1—Cu1—O1$^{i}$ | 163.64 (15) | C13—C12—C15 | 109.2 (6) |
| N2—Cu1—O1$^{i}$ | 94.27 (13) | C14—C12—C15 | 106.5 (6) |
| N1—Cu1—O1 | 93.91 (13) | C15′—C12—C13′ | 106.4 (7) |
| N2—Cu1—O1 | 168.30 (15) | C14′—C12—C13′ | 107.1 (7) |
| O1$^{i}$—Cu1—O1 | 75.33 (11) | C15′—C12—C8 | 115.3 (7) |
| C11—O1—Cu1$^{i}$ | 127.5 (3) | C14′—C12—C8 | 109.8 (8) |
| C11—O1—Cu1 | 125.6 (3) | C13—C12—C8 | 114.4 (5) |
| Cu1$^{i}$—O1—Cu1 | 104.28 (11) | C14—C12—C8 | 109.1 (6) |
| H2B—O2—H2A | 125 (8) | C15—C12—C8 | 109.7 (6) |
| C5—N1—C4 | 116.5 (4) | C13′—C12—C8 | 109.3 (8) |
| C5—N1—Cu1 | 122.8 (3) | C12—C13—H13A | 109.5 |
| C4—N1—Cu1 | 120.3 (3) | C12—C13—H13B | 109.5 |
| C1—N2—C2 | 113.0 (4) | H13A—C13—H13B | 109.5 |
| C1—N2—Cu1 | 122.9 (3) | C12—C13—H13C | 109.5 |
| C2—N2—Cu1 | 124.1 (3) | H13A—C13—H13C | 109.5 |
| N2—C1—C10$^{i}$ | 128.3 (4) | H13B—C13—H13C | 109.5 |
| N2—C1—H1 | 115.9 | C12—C14—H14A | 109.5 |
| C10$^{i}$—C1—H1 | 115.9 | C12—C14—H14B | 109.5 |
| C3—C2—N2 | 114.7 (4) | H14A—C14—H14B | 109.5 |
| C3—C2—H2A′ | 108.6 | C12—C14—H14C | 109.5 |
| N2—C2—H2A′ | 108.6 | H14A—C14—H14C | 109.5 |
| C3—C2—H2B′ | 108.6 | H14B—C14—H14C | 109.5 |

| | | | |
|---|---|---|---|
| N2—C2—H2B′ | 108.6 | C12—C15—H15A | 109.5 |
| H2A′—C2—H2B′ | 107.6 | C12—C15—H15B | 109.5 |
| C2—C3—C4 | 112.8 (5) | H15A—C15—H15B | 109.5 |
| C2—C3—H3A | 109.0 | C12—C15—H15C | 109.5 |
| C4—C3—H3A | 109.0 | H15A—C15—H15C | 109.5 |
| C2—C3—H3B | 109.0 | H15B—C15—H15C | 109.5 |
| C4—C3—H3B | 109.0 | C12—C13′—H13D | 109.5 |
| H3A—C3—H3B | 107.8 | C12—C13′—H13E | 109.5 |
| N1—C4—C3 | 109.5 (4) | H13D—C13′—H13E | 109.5 |
| N1—C4—H4A | 109.8 | C12—C13′—H13F | 109.5 |
| C3—C4—H4A | 109.8 | H13D—C13′—H13F | 109.5 |
| N1—C4—H4B | 109.8 | H13E—C13′—H13F | 109.5 |
| C3—C4—H4B | 109.8 | C12—C14′—H14D | 109.5 |
| H4A—C4—H4B | 108.2 | C12—C14′—H14E | 109.5 |
| N1—C5—C6 | 127.7 (4) | H14D—C14′—H14E | 109.5 |
| N1—C5—H5 | 116.1 | C12—C14′—H14F | 109.5 |
| C6—C5—H5 | 116.1 | H14D—C14′—H14F | 109.5 |
| C7—C6—C11 | 119.2 (4) | H14E—C14′—H14F | 109.5 |
| C7—C6—C5 | 115.3 (4) | C12—C15′—H15D | 109.5 |
| C11—C6—C5 | 125.6 (4) | C12—C15′—H15E | 109.5 |
| C8—C7—C6 | 122.8 (4) | H15D—C15′—H15E | 109.5 |
| C8—C7—H7 | 118.6 | C12—C15′—H15F | 109.5 |
| C6—C7—H7 | 118.6 | H15D—C15′—H15F | 109.5 |
| C7—C8—C9 | 117.2 (3) | H15E—C15′—H15F | 109.5 |
| C7—C8—C12 | 121.5 (5) | O5—Cl1—O3 | 112.1 (6) |
| C9—C8—C12 | 121.3 (5) | O5—Cl1—O6 | 111.6 (6) |
| C8—C9—C10 | 122.6 (4) | O3—Cl1—O6 | 108.8 (7) |
| C8—C9—H9 | 118.7 | O6′—Cl1—O3′ | 111.6 (6) |
| C10—C9—H9 | 118.7 | O6′—Cl1—O5′ | 108.9 (7) |
| C11—C10—C9 | 120.2 (4) | O3′—Cl1—O5′ | 110.5 (6) |
| C11—C10—C1$^{i}$ | 124.9 (4) | O6′—Cl1—O4′ | 109.0 (6) |
| C9—C10—C1$^{i}$ | 114.9 (4) | O3′—Cl1—O4′ | 109.7 (6) |
| O1—C11—C10 | 121.7 (4) | O5′—Cl1—O4′ | 107.0 (7) |
| O1—C11—C6 | 120.2 (4) | O5—Cl1—O4 | 106.3 (6) |
| C10—C11—C6 | 118.0 (3) | O3—Cl1—O4 | 110.5 (6) |
| C15′—C12—C14′ | 108.7 (7) | O6—Cl1—O4 | 107.4 (6) |

| | | | |
|---|---|---|---|
| N1—Cu1—O1—C11 | 23.1 (3) | C5—C6—C7—C8 | 178.0 (5) |
| N2—Cu1—O1—C11 | −141.7 (7) | C6—C7—C8—C9 | 2.3 (7) |
| O1$^{i}$—Cu1—O1—C11 | −169.4 (3) | C6—C7—C8—C12 | 179.9 (5) |
| N1—Cu1—O1—Cu1$^{i}$ | −174.27 (14) | C7—C8—C9—C10 | −1.4 (7) |
| N2—Cu1—O1—Cu1$^{i}$ | 21.0 (8) | C12—C8—C9—C10 | −178.9 (5) |
| O1$^{i}$—Cu1—O1—Cu1$^{i}$ | −6.78 (17) | C8—C9—C10—C11 | −0.9 (8) |
| N2—Cu1—N1—C5 | 163.2 (4) | C8—C9—C10—C1$^{i}$ | 178.9 (4) |
| O1$^{i}$—Cu1—N1—C5 | −61.8 (7) | Cu1$^{i}$—O1—C11—C10 | 3.0 (5) |
| O1—Cu1—N1—C5 | −13.7 (4) | Cu1—O1—C11—C10 | 161.7 (3) |
| N2—Cu1—N1—C4 | −10.2 (4) | Cu1$^{i}$—O1—C11—C6 | −177.9 (3) |
| O1$^{i}$—Cu1—N1—C4 | 124.9 (5) | Cu1—O1—C11—C6 | −19.3 (5) |

| | | | |
|---|---|---|---|
| O1—Cu1—N1—C4 | 172.9 (3) | C9—C10—C11—O1 | −178.7 (4) |
| N1—Cu1—N2—C1 | 173.9 (4) | C1$^{i}$—C10—C11—O1 | 1.5 (7) |
| O1$^{i}$—Cu1—N2—C1 | 5.4 (4) | C9—C10—C11—C6 | 2.3 (6) |
| O1—Cu1—N2—C1 | −21.5 (10) | C1$^{i}$—C10—C11—C6 | −177.5 (5) |
| N1—Cu1—N2—C2 | −7.4 (5) | C7—C6—C11—O1 | 179.5 (4) |
| O1$^{i}$—Cu1—N2—C2 | −175.8 (5) | C5—C6—C11—O1 | 0.7 (7) |
| O1—Cu1—N2—C2 | 157.3 (7) | C7—C6—C11—C10 | −1.4 (7) |
| C2—N2—C1—C10$^{i}$ | 178.0 (5) | C5—C6—C11—C10 | 179.8 (5) |
| Cu1—N2—C1—C10$^{i}$ | −3.2 (7) | C7—C8—C12—C15′ | −172.3 (8) |
| C1—N2—C2—C3 | 163.5 (5) | C9—C8—C12—C15′ | 5.1 (9) |
| Cu1—N2—C2—C3 | −15.3 (8) | C7—C8—C12—C14′ | 64.5 (9) |
| N2—C2—C3—C4 | 59.5 (7) | C9—C8—C12—C14′ | −118.0 (8) |
| C5—N1—C4—C3 | −125.1 (4) | C7—C8—C12—C13 | 7.0 (8) |
| Cu1—N1—C4—C3 | 48.7 (5) | C9—C8—C12—C13 | −175.5 (6) |
| C2—C3—C4—N1 | −77.9 (6) | C7—C8—C12—C14 | 127.7 (7) |
| C4—N1—C5—C6 | 175.2 (5) | C9—C8—C12—C14 | −54.8 (7) |
| Cu1—N1—C5—C6 | 1.6 (7) | C7—C8—C12—C15 | −116.1 (7) |
| N1—C5—C6—C7 | −169.8 (5) | C9—C8—C12—C15 | 61.4 (8) |
| N1—C5—C6—C11 | 9.0 (9) | C7—C8—C12—C13′ | −52.6 (9) |
| C11—C6—C7—C8 | −0.9 (8) | C9—C8—C12—C13′ | 124.9 (8) |

Symmetry code: (i) −*x*, −*y*+2, *z*.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2*B*···O2$^{ii}$ | 0.82 (2) | 2.07 (4) | 2.821 (6) | 153 (8) |
| O2—H2*A*···O3 | 0.81 (2) | 2.26 (3) | 2.806 (8) | 125 (2) |
| O2—H2*A*···O3′ | 0.81 (2) | 2.49 (4) | 2.947 (10) | 117 (3) |
| O2—H2*A*···N1 | 0.81 (2) | 2.55 (3) | 3.197 (6) | 138 (3) |

Symmetry code: (ii) *y*−1, −*x*+1, −*z*+1.