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# Aqua{2-[(3,5-dichloro-2-oxidobenzylidene)amino]-3-(4-hydroxyphenyl)propionato-$\kappa^3O^1,N,O^2$}copper(II) sesquihydrate

**Zheng Liu,* Yong Liao Wang and Li Wang**

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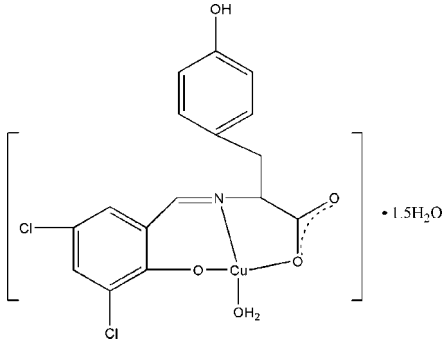
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.008 Å; some non-H atoms missing; disorder in main residue; $R$ factor = 0.048; $wR$ factor = 0.143; data-to-parameter ratio = 10.8.

In the title compound, $[Cu(C_{16}H_{11}Cl_2NO_4)(H_2O)]\cdot1.5H_2O$, the $Cu^{II}$ atom is coordinated by two O atoms and one N atom from the 2-[(3,5-dichloro-2-oxidobenzylidene)amino]-3-(4-hydroxyphenyl)propionate ligand, and by the O atom from a water molecule in a square-planar coordination. There are two formula units in the asymmetric unit. Molecules are further assembled into a three-dimensional network through C—H···Cl contacts, a Cu···Cl weak interaction [3.161 (2) Å], O—H···O and C—H···O hydrogen bonds. The three water molecules of the asymmetric unit are distributed over five positions with one full and two approximately half occupancies, while a tyrosine side chain in one of the complex molecules is disordered over two positions [occupancies 0.507 (5) and 0.493 (5)].

## Related literature

For related literature, see: Casella & Gullotti (1986); Guthrie *et al.* (1980); Wang *et al.* (1994); Zhang *et al.* (2003).




## Experimental

### Crystal data

$[Cu(C_{16}H_{11}Cl_2NO_4)(H_2O)]\cdot1.5H_2O$
$M_r$ = 460.74
Triclinic, $P\overline{1}$
$a$ = 10.006 (2) Å
$b$ = 13.899 (2) Å
$c$ = 14.565 (2) Å
$\alpha$ = 70.469 (2)°
$\beta$ = 87.427 (3)°
$\gamma$ = 73.121 (3)°
$V$ = 1823.8 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.53 mm$^{-1}$
$T$ = 293 (2) K
0.40 × 0.18 × 0.09 mm

### Data collection

Bruker SMART 1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.580, $T_{max}$ = 0.875
9183 measured reflections
6315 independent reflections
3605 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.143
$S$ = 1.02
6315 reflections
583 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.36 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O12$^{i}$ | 0.82 | 1.88 | 2.643 (10) | 154 |
| O8—H8···O7$^{ii}$ | 0.82 | 1.70 | 2.515 (9) | 175 |
| O5—H33···O6$^{iii}$ | 0.85 | 1.89 | 2.714 (6) | 164 |
| O13—H42···Cl1$^{iv}$ | 0.85 | 2.77 | 3.555 (9) | 154 |
| C26—H26···O3$^{v}$ | 0.93 | 2.44 | 3.365 (7) | 173 |

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

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: CS2072).

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Zhang, S. H., Jiang, Y. M., Xiao, Y. & Zhou, Z. Y. (2003). *Chin. J. Inorg. Chem.* **19**, 517–520.

# supporting information

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# Aqua{2-[(3,5-dichloro-2-oxidobenzylidene)amino]-3-(4-hydroxyphenyl)-propionato-$\kappa^3O^1,N,O^2$}copper(II) sesquihydrate

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## S1. Comment

Schiff base complexes containing amino acids have been studied for many years(Casella & Gullotti, 1986; Wang *et al.*, 1994; Zhang *et al.*, 2003). Schiff bases have played an important role in the development of coordination chemistry as they form stable complexes with most of the transition metals.

In the asymmetric unit there are two $Cu^{II}$ ions, two *L* ligand anions, two ligating water molecules and three water hydrate molecules (Fig. 1). The $Cu^{II}$ cations are coordinated by two O atoms and one N atom from ligand *L*, then the O atom from an $H_2O$ coordinate to $Cu^{II}$. These four atoms form a square-planar coordination centered at the cuprate(II) ions (Fig. 1). The coordination in these two complexes in the asymmetric unit does not seem to be alike. There is a Cu···Cl distance of 3.161 (2) Å indicating a possible weak interaction between the two complexes in the asymmetric unit. Fig. 2 shows that the tyrosine in one of the complexes has two different orientations in this crystal giving a disordered structure. Weak contacts of C–H···Cl and Cu···Cl types and O–H···O hydrogen bonds construct a 3-D network running along the *a* axis (Fig.3).

## S2. Experimental

An ethanol solution (5 ml) containing 3,5-dichloro-2-hydroxy-benzaldehyde (0.096 g, 0.5 mmol) was added to an aqueous solution containing 2-amino-3-(4-hydroxy-phenyl)-propionic acid (0.222 g, 2 mmol) and sodium hydroxide (0.091 g, 0.5 mmol). After stirring for 1 h, an aqueous solution of copper chloride (0.1 g, 0.5 mmol) was added to the resulting solution and stirred for 2 h. The green solution was filtered. After 10 days, green block crystals were obtained by slow evaporation of the filtrate (yield: 45.2%, based on Cu).

## S3. Refinement

Water H atoms were located in a difference Fourier map and were allowed to ride on the O atom, with $U_{iso}$(H) = 1.5$U_{eq}$(O). All other H atoms were positioned geometrically and refined as riding, with C–H = 0.93 Å and with $U_{iso}$(H) = 1.2 $U_{eq}$(C).

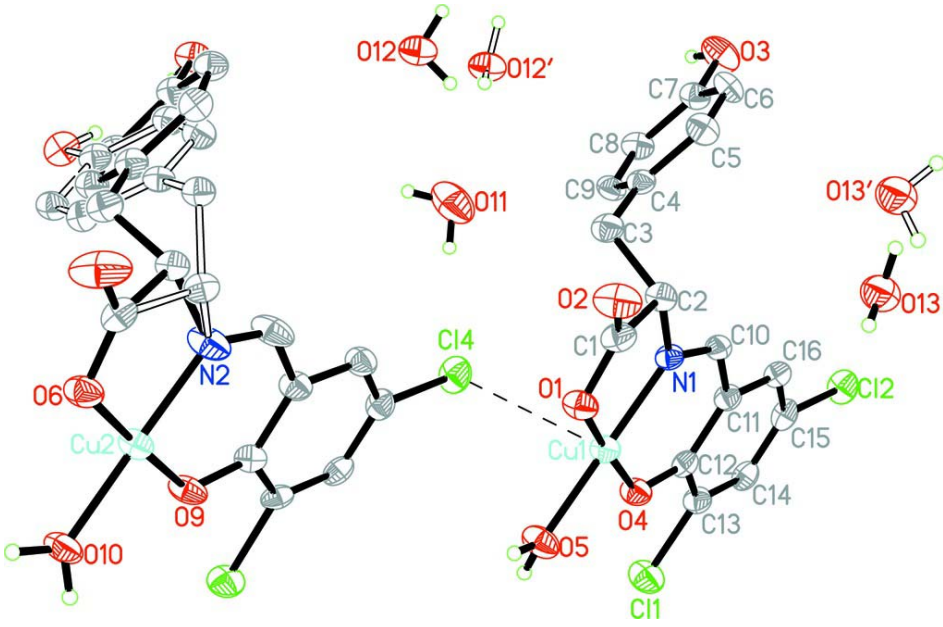


**Figure 1**

A view of the title complex showing 30% probability displacement ellipsoids. H atoms, except for the water molecules, are not shown.

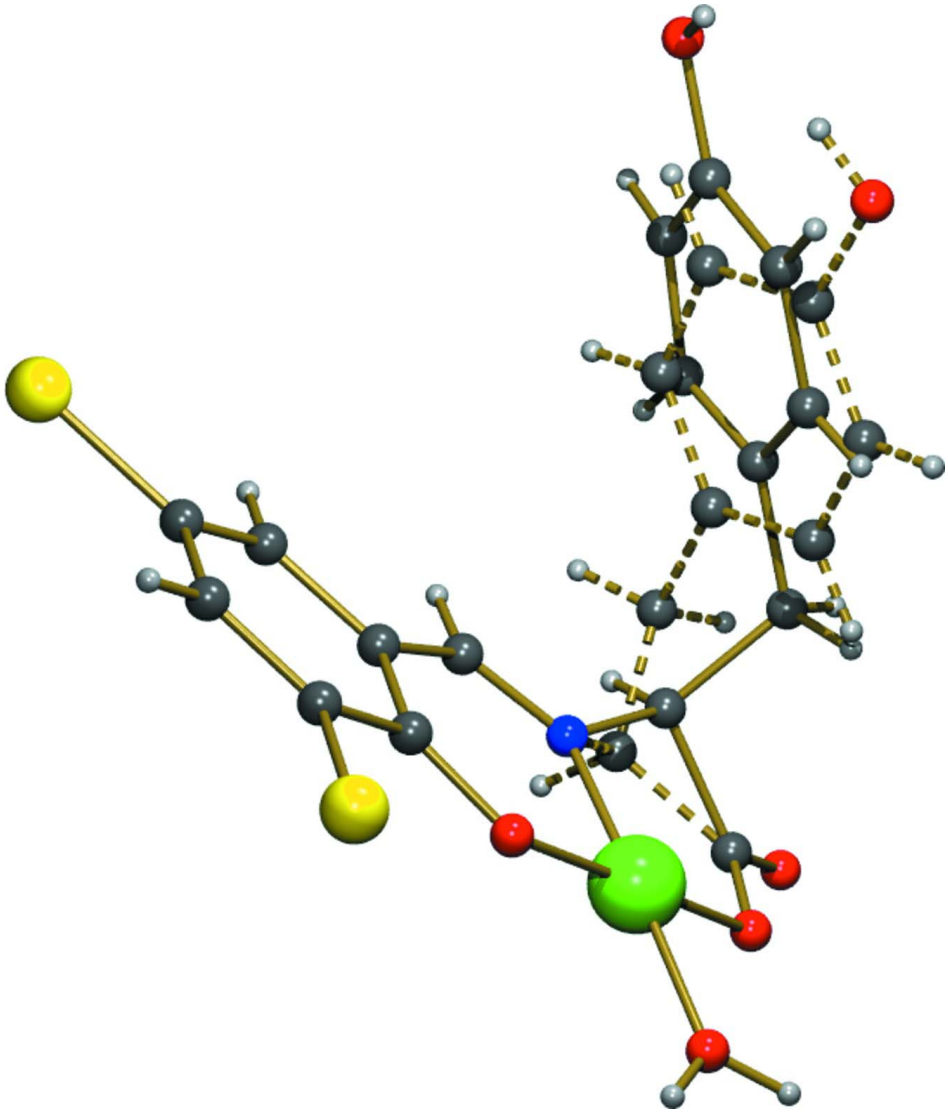

**Figure 2**

The disordered tyrosine view in one of the complexes.

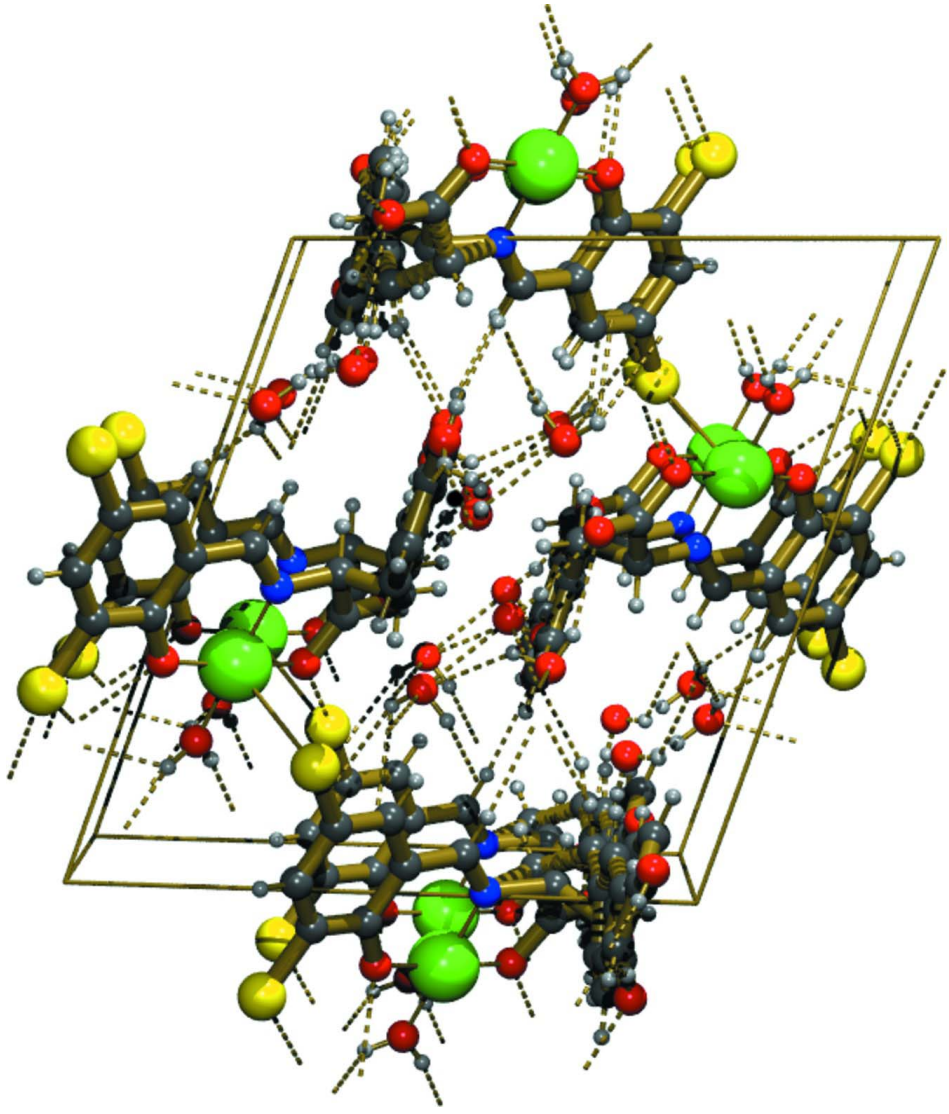

**Figure 3**

The three-dimensional network of Cu···Cl weak interactions and hydrogen bonds shown in dotted lines.

## Aqua{2-[(3,5-dichloro-2-oxidobenzylidene)amino]-3-(4-hydroxyphenyl)propionato- $\kappa^3O^1,N,O^2$}copper(II) sesquihydrate

*Crystal data*

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Triclinic, $P\overline{1}$
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$c = 14.565$ (2) Å
$\alpha = 70.469$ (2)°
$\beta = 87.427$ (3)°
$\gamma = 73.121$ (3)°
$V = 1823.8$ (5) Å$^3$
$Z = 4$
$F(000) = 936$
$D_x = 1.678$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2332 reflections
$\theta = 2.3$–25.2°
$\mu = 1.53$ mm$^{-1}$
$T = 293$ K
Plate, green
0.40 × 0.18 × 0.09 mm

*Data collection*

Bruker SMART 1000 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.580, $T_{max}$ = 0.875
9183 measured reflections
6315 independent reflections
3605 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 1.5°
$h$ = −11→11
$k$ = −12→16
$l$ = −16→17

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.143
$S$ = 1.02
6315 reflections
583 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
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.58 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.36 e Å$^{-3}$

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cu1 | 0.46267 (7) | 0.15476 (5) | 0.36588 (4) | 0.0406 (2) | |
| Cu2 | −0.01499 (6) | 0.64742 (5) | −0.11028 (4) | 0.0407 (2) | |
| Cl1 | 0.87069 (17) | −0.11542 (12) | 0.34408 (11) | 0.0668 (5) | |
| Cl2 | 1.07017 (15) | −0.16022 (11) | 0.69630 (11) | 0.0578 (4) | |
| Cl3 | 0.39713 (16) | 0.37698 (11) | −0.12877 (10) | 0.0580 (4) | |
| Cl4 | 0.58637 (15) | 0.33274 (11) | 0.22575 (11) | 0.0597 (4) | |
| N1 | 0.5002 (4) | 0.1767 (3) | 0.4841 (3) | 0.0349 (10) | |
| N2 | 0.0214 (5) | 0.6696 (4) | 0.0075 (3) | 0.0556 (13) | |
| O1 | 0.2819 (4) | 0.2595 (3) | 0.3616 (2) | 0.0447 (9) | |
| O2 | 0.1508 (4) | 0.3508 (3) | 0.4497 (3) | 0.0621 (12) | |
| O3 | 0.8857 (4) | 0.3483 (4) | 0.7126 (3) | 0.0699 (12) | |
| H3 | 0.9506 | 0.3622 | 0.6795 | 0.105* | |
| O4 | 0.6311 (4) | 0.0441 (3) | 0.3785 (2) | 0.0454 (9) | |
| O5 | 0.4122 (4) | 0.1358 (3) | 0.2489 (2) | 0.0552 (10) | |
| H33 | 0.3333 | 0.1676 | 0.2174 | 0.083* | |
| H34 | 0.4672 | 0.1072 | 0.2129 | 0.083* | |
| O6 | −0.1909 (4) | 0.7570 (3) | −0.1170 (3) | 0.0508 (10) | |
| O7 | −0.3148 (4) | 0.8569 (3) | −0.0328 (4) | 0.0801 (15) | |
| O8 | 0.4986 (7) | 0.8913 (6) | 0.0835 (5) | 0.051 (2) | 0.507 (5) |
| H8 | 0.5564 | 0.8836 | 0.0429 | 0.077* | 0.507 (5) |
| O8′ | 0.4246 (7) | 0.9296 (6) | −0.1086 (6) | 0.049 (2) | 0.493 (5) |
| H8′ | 0.4912 | 0.9095 | −0.0691 | 0.074* | 0.493 (5) |
| O9 | 0.1554 (3) | 0.5378 (3) | −0.0977 (2) | 0.0459 (9) | |
| O10 | −0.0682 (4) | 0.6254 (3) | −0.2247 (2) | 0.0547 (10) | |
| H35 | −0.1283 | 0.6714 | −0.2690 | 0.082* | |

| | | | | | |
|---|---|---|---|---|---|
| H36 | −0.0357 | 0.5731 | −0.2452 | 0.082* | |
| O11 | 0.8440 (5) | 0.4610 (4) | 0.3037 (3) | 0.1056 (18) | |
| H37 | 0.8272 | 0.5183 | 0.2548 | 0.158* | |
| H38 | 0.8233 | 0.4217 | 0.2763 | 0.158* | |
| O12 | 0.8592 (8) | 0.6491 (7) | 0.3412 (6) | 0.066 (3) | 0.522 (10) |
| H39 | 0.8529 | 0.6666 | 0.3922 | 0.098* | 0.522 (10) |
| H40 | 0.8643 | 0.5832 | 0.3637 | 0.098* | 0.522 (10) |
| O13 | 0.4751 (9) | 0.0597 (6) | 0.7508 (5) | 0.077 (3) | 0.523 (6) |
| H41 | 0.4422 | 0.0976 | 0.7865 | 0.116* | 0.523 (6) |
| H42 | 0.4076 | 0.0581 | 0.7184 | 0.116* | 0.523 (6) |
| O12′ | 0.9281 (9) | 0.5554 (7) | 0.4185 (7) | 0.066 (4) | 0.478 (10) |
| H39′ | 0.8473 | 0.5526 | 0.4064 | 0.098* | 0.478 (10) |
| H40′ | 0.9155 | 0.5923 | 0.4561 | 0.098* | 0.478 (10) |
| O13′ | 0.3267 (10) | 0.1743 (7) | 0.8023 (6) | 0.077 (3) | 0.477 (6) |
| H41′ | 0.3386 | 0.1669 | 0.8620 | 0.116* | 0.477 (6) |
| H42′ | 0.2932 | 0.1248 | 0.8019 | 0.116* | 0.477 (6) |
| C1 | 0.2626 (6) | 0.2932 (4) | 0.4349 (4) | 0.0468 (14) | |
| C2 | 0.3934 (5) | 0.2649 (4) | 0.5020 (4) | 0.0404 (13) | |
| H2 | 0.3698 | 0.2435 | 0.5705 | 0.048* | |
| C3 | 0.4401 (5) | 0.3661 (4) | 0.4761 (4) | 0.0456 (14) | |
| H3A | 0.4656 | 0.3846 | 0.4087 | 0.055* | |
| H3B | 0.3613 | 0.4241 | 0.4809 | 0.055* | |
| C4 | 0.5605 (5) | 0.3567 (4) | 0.5389 (4) | 0.0380 (13) | |
| C5 | 0.5444 (6) | 0.3550 (4) | 0.6350 (4) | 0.0486 (15) | |
| H5A | 0.4579 | 0.3564 | 0.6614 | 0.058* | |
| C6 | 0.6528 (6) | 0.3515 (4) | 0.6913 (4) | 0.0474 (14) | |
| H6 | 0.6388 | 0.3506 | 0.7551 | 0.057* | |
| C7 | 0.7826 (6) | 0.3492 (4) | 0.6543 (4) | 0.0446 (14) | |
| C8 | 0.8034 (6) | 0.3492 (4) | 0.5601 (4) | 0.0450 (14) | |
| H8A | 0.8907 | 0.3471 | 0.5344 | 0.054* | |
| C9 | 0.6932 (6) | 0.3523 (4) | 0.5041 (4) | 0.0429 (14) | |
| H9 | 0.7083 | 0.3513 | 0.4409 | 0.051* | |
| C10 | 0.6014 (5) | 0.1182 (4) | 0.5469 (4) | 0.0380 (13) | |
| H10A | 0.6016 | 0.1323 | 0.6050 | 0.046* | |
| C11 | 0.7153 (5) | 0.0322 (4) | 0.5356 (4) | 0.0383 (12) | |
| C12 | 0.7253 (5) | 0.0025 (4) | 0.4506 (4) | 0.0395 (13) | |
| C13 | 0.8490 (6) | −0.0788 (4) | 0.4477 (4) | 0.0452 (14) | |
| C14 | 0.9516 (5) | −0.1270 (4) | 0.5211 (4) | 0.0433 (14) | |
| H14 | 1.0306 | −0.1798 | 0.5158 | 0.052* | |
| C15 | 0.9376 (5) | −0.0971 (4) | 0.6025 (4) | 0.0438 (14) | |
| C16 | 0.8219 (5) | −0.0183 (4) | 0.6104 (4) | 0.0411 (13) | |
| H16 | 0.8141 | 0.0017 | 0.6658 | 0.049* | |
| C17 | −0.2082 (5) | 0.7943 (4) | −0.0462 (4) | 0.0420 (13) | |
| C18 | −0.073 (2) | 0.7774 (14) | 0.0135 (14) | 0.038 (4) | 0.507 (5) |
| H18 | −0.0932 | 0.7705 | 0.0814 | 0.045* | 0.507 (5) |
| C19 | −0.0263 (11) | 0.8773 (8) | −0.0352 (8) | 0.045 (3) | 0.507 (5) |
| H19A | −0.0256 | 0.8894 | −0.1047 | 0.053* | 0.507 (5) |
| H19B | −0.0973 | 0.9373 | −0.0259 | 0.053* | 0.507 (5) |

| | | | | | |
|---|---|---|---|---|---|
| C20 | 0.1122 (19) | 0.8796 (15) | −0.0029 (15) | 0.042 (4) | 0.507 (5) |
| C21 | 0.140 (2) | 0.8750 (15) | 0.0908 (16) | 0.044 (4) | 0.507 (5) |
| H21 | 0.0745 | 0.8647 | 0.1374 | 0.053* | 0.507 (5) |
| C22 | 0.2662 (11) | 0.8858 (8) | 0.1162 (8) | 0.046 (3) | 0.507 (5) |
| H22 | 0.2784 | 0.8920 | 0.1767 | 0.055* | 0.507 (5) |
| C23 | 0.3739 (14) | 0.8875 (10) | 0.0528 (10) | 0.042 (3) | 0.507 (5) |
| C24 | 0.347 (6) | 0.887 (5) | −0.039 (5) | 0.044 (9) | 0.507 (5) |
| H24 | 0.4177 | 0.8887 | −0.0834 | 0.053* | 0.507 (5) |
| C25 | 0.218 (2) | 0.8852 (13) | −0.0675 (12) | 0.044 (4) | 0.507 (5) |
| H25 | 0.2023 | 0.8875 | −0.1307 | 0.053* | 0.507 (5) |
| C18′ | −0.101 (2) | 0.7389 (15) | 0.0366 (14) | 0.036 (4) | 0.493 (5) |
| H18′ | −0.1411 | 0.6948 | 0.0915 | 0.043* | 0.493 (5) |
| C19′ | −0.0505 (10) | 0.8163 (8) | 0.0707 (8) | 0.038 (3) | 0.493 (5) |
| H19C | −0.1274 | 0.8811 | 0.0598 | 0.046* | 0.493 (5) |
| H19D | −0.0285 | 0.7845 | 0.1405 | 0.046* | 0.493 (5) |
| C20′ | 0.0742 (19) | 0.8468 (15) | 0.0232 (13) | 0.032 (4) | 0.493 (5) |
| C21′ | 0.0869 (12) | 0.8771 (8) | −0.0768 (9) | 0.040 (3) | 0.493 (5) |
| H21′ | 0.0139 | 0.8811 | −0.1167 | 0.048* | 0.493 (5) |
| C22′ | 0.2054 (19) | 0.9017 (13) | −0.1196 (11) | 0.042 (4) | 0.493 (5) |
| H22′ | 0.2126 | 0.9191 | −0.1867 | 0.050* | 0.493 (5) |
| C23′ | 0.313 (5) | 0.900 (5) | −0.062 (5) | 0.040 (8) | 0.493 (5) |
| C24′ | 0.3069 (15) | 0.8696 (10) | 0.0395 (12) | 0.042 (3) | 0.493 (5) |
| H24′ | 0.3785 | 0.8676 | 0.0792 | 0.051* | 0.493 (5) |
| C25′ | 0.188 (2) | 0.8424 (17) | 0.0773 (17) | 0.046 (5) | 0.493 (5) |
| H25′ | 0.1842 | 0.8192 | 0.1448 | 0.055* | 0.493 (5) |
| C26 | 0.1228 (6) | 0.6130 (5) | 0.0709 (4) | 0.0566 (16) | |
| H26 | 0.1228 | 0.6290 | 0.1280 | 0.068* | |
| C27 | 0.2371 (5) | 0.5265 (4) | 0.0609 (4) | 0.0420 (13) | |
| C28 | 0.2481 (5) | 0.4970 (4) | −0.0244 (4) | 0.0371 (12) | |
| C29 | 0.3733 (5) | 0.4155 (4) | −0.0259 (4) | 0.0386 (13) | |
| C30 | 0.4740 (5) | 0.3672 (4) | 0.0480 (4) | 0.0422 (13) | |
| H30 | 0.5542 | 0.3153 | 0.0431 | 0.051* | |
| C31 | 0.4552 (6) | 0.3966 (4) | 0.1310 (4) | 0.0452 (14) | |
| C32 | 0.3407 (5) | 0.4747 (4) | 0.1372 (4) | 0.0456 (14) | |
| H32 | 0.3307 | 0.4943 | 0.1928 | 0.055* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cu1 | 0.0356 (4) | 0.0389 (4) | 0.0449 (4) | −0.0035 (3) | −0.0044 (3) | −0.0167 (3) |
| Cu2 | 0.0304 (4) | 0.0523 (4) | 0.0438 (4) | −0.0084 (3) | 0.0012 (3) | −0.0247 (3) |
| Cl1 | 0.0668 (11) | 0.0601 (10) | 0.0635 (10) | 0.0104 (8) | −0.0035 (8) | −0.0319 (8) |
| Cl2 | 0.0417 (9) | 0.0507 (9) | 0.0644 (10) | −0.0008 (7) | −0.0143 (7) | −0.0070 (7) |
| Cl3 | 0.0579 (10) | 0.0565 (9) | 0.0624 (9) | −0.0029 (8) | 0.0133 (8) | −0.0366 (8) |
| Cl4 | 0.0463 (9) | 0.0514 (9) | 0.0650 (10) | −0.0010 (7) | −0.0144 (7) | −0.0080 (7) |
| N1 | 0.032 (2) | 0.029 (2) | 0.042 (2) | −0.003 (2) | 0.000 (2) | −0.014 (2) |
| N2 | 0.034 (3) | 0.070 (3) | 0.063 (3) | 0.014 (2) | −0.008 (2) | −0.045 (3) |
| O1 | 0.034 (2) | 0.049 (2) | 0.051 (2) | −0.0024 (17) | −0.0083 (17) | −0.0244 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2 | 0.031 (2) | 0.069 (3) | 0.090 (3) | 0.005 (2) | −0.010 (2) | −0.046 (2) |
| O3 | 0.053 (3) | 0.105 (4) | 0.062 (3) | −0.022 (3) | −0.009 (2) | −0.039 (3) |
| O4 | 0.044 (2) | 0.041 (2) | 0.049 (2) | 0.0006 (17) | −0.0049 (19) | −0.0213 (18) |
| O5 | 0.045 (2) | 0.066 (3) | 0.053 (2) | 0.002 (2) | −0.0085 (18) | −0.031 (2) |
| O6 | 0.043 (2) | 0.052 (2) | 0.060 (2) | −0.0029 (19) | −0.0139 (19) | −0.030 (2) |
| O7 | 0.033 (2) | 0.079 (3) | 0.144 (4) | 0.008 (2) | −0.013 (3) | −0.075 (3) |
| O8 | 0.032 (4) | 0.060 (5) | 0.066 (5) | −0.006 (4) | −0.005 (4) | −0.033 (4) |
| O8′ | 0.026 (4) | 0.054 (5) | 0.067 (6) | −0.013 (4) | 0.007 (4) | −0.021 (4) |
| O9 | 0.030 (2) | 0.062 (2) | 0.054 (2) | −0.0033 (18) | −0.0003 (18) | −0.039 (2) |
| O10 | 0.054 (3) | 0.062 (3) | 0.053 (2) | −0.007 (2) | −0.0147 (19) | −0.032 (2) |
| O11 | 0.103 (4) | 0.138 (5) | 0.078 (3) | −0.016 (4) | 0.013 (3) | −0.057 (3) |
| O12 | 0.060 (6) | 0.073 (7) | 0.078 (7) | −0.020 (5) | 0.012 (5) | −0.042 (6) |
| O13 | 0.090 (7) | 0.076 (6) | 0.069 (6) | −0.022 (5) | −0.004 (5) | −0.030 (5) |
| O12′ | 0.060 (6) | 0.073 (8) | 0.078 (7) | −0.020 (5) | 0.012 (5) | −0.042 (6) |
| O13′ | 0.090 (8) | 0.076 (7) | 0.069 (6) | −0.022 (6) | −0.004 (5) | −0.030 (5) |
| C1 | 0.041 (4) | 0.040 (3) | 0.060 (4) | −0.009 (3) | −0.005 (3) | −0.020 (3) |
| C2 | 0.030 (3) | 0.043 (3) | 0.045 (3) | −0.002 (3) | −0.007 (2) | −0.017 (3) |
| C3 | 0.043 (3) | 0.037 (3) | 0.052 (3) | −0.001 (3) | −0.008 (3) | −0.017 (3) |
| C4 | 0.038 (3) | 0.030 (3) | 0.043 (3) | −0.002 (2) | −0.005 (3) | −0.016 (2) |
| C5 | 0.037 (3) | 0.061 (4) | 0.052 (3) | −0.014 (3) | 0.010 (3) | −0.024 (3) |
| C6 | 0.047 (4) | 0.064 (4) | 0.034 (3) | −0.014 (3) | 0.001 (3) | −0.021 (3) |
| C7 | 0.040 (3) | 0.047 (3) | 0.047 (3) | −0.005 (3) | −0.008 (3) | −0.020 (3) |
| C8 | 0.032 (3) | 0.050 (3) | 0.057 (4) | −0.006 (3) | 0.004 (3) | −0.027 (3) |
| C9 | 0.048 (4) | 0.044 (3) | 0.040 (3) | −0.009 (3) | 0.006 (3) | −0.023 (3) |
| C10 | 0.045 (3) | 0.032 (3) | 0.036 (3) | −0.008 (3) | −0.003 (3) | −0.012 (2) |
| C11 | 0.035 (3) | 0.032 (3) | 0.044 (3) | −0.006 (2) | −0.002 (2) | −0.011 (2) |
| C12 | 0.040 (3) | 0.030 (3) | 0.045 (3) | −0.008 (3) | 0.000 (3) | −0.009 (2) |
| C13 | 0.049 (4) | 0.035 (3) | 0.050 (3) | −0.005 (3) | 0.006 (3) | −0.017 (3) |
| C14 | 0.036 (3) | 0.028 (3) | 0.054 (3) | 0.001 (2) | −0.002 (3) | −0.007 (3) |
| C15 | 0.028 (3) | 0.036 (3) | 0.055 (3) | −0.004 (3) | −0.006 (3) | −0.002 (3) |
| C16 | 0.042 (3) | 0.036 (3) | 0.046 (3) | −0.012 (3) | 0.001 (3) | −0.013 (3) |
| C17 | 0.027 (3) | 0.035 (3) | 0.061 (4) | −0.007 (3) | 0.000 (3) | −0.014 (3) |
| C18 | 0.027 (9) | 0.041 (12) | 0.047 (10) | −0.005 (7) | −0.002 (7) | −0.020 (8) |
| C19 | 0.032 (7) | 0.043 (7) | 0.061 (7) | −0.008 (5) | −0.008 (6) | −0.021 (6) |
| C20 | 0.031 (12) | 0.039 (11) | 0.054 (12) | −0.002 (7) | −0.002 (9) | −0.018 (8) |
| C21 | 0.032 (11) | 0.048 (12) | 0.053 (9) | −0.013 (9) | 0.005 (7) | −0.017 (8) |
| C22 | 0.041 (7) | 0.046 (7) | 0.053 (7) | −0.011 (5) | 0.003 (6) | −0.023 (6) |
| C23 | 0.034 (8) | 0.040 (7) | 0.054 (8) | −0.006 (6) | −0.002 (7) | −0.021 (6) |
| C24 | 0.04 (3) | 0.040 (16) | 0.06 (2) | −0.007 (17) | 0.006 (15) | −0.022 (15) |
| C25 | 0.036 (9) | 0.045 (9) | 0.052 (10) | −0.004 (7) | −0.009 (11) | −0.023 (9) |
| C18′ | 0.024 (9) | 0.038 (12) | 0.047 (10) | −0.003 (7) | 0.012 (6) | −0.022 (8) |
| C19′ | 0.032 (6) | 0.040 (7) | 0.045 (6) | −0.009 (5) | 0.000 (5) | −0.019 (5) |
| C20′ | 0.024 (9) | 0.036 (11) | 0.042 (10) | −0.007 (6) | 0.004 (7) | −0.021 (8) |
| C21′ | 0.030 (7) | 0.042 (7) | 0.048 (7) | −0.008 (5) | −0.003 (6) | −0.016 (6) |
| C22′ | 0.034 (8) | 0.046 (9) | 0.049 (9) | −0.009 (6) | −0.004 (9) | −0.021 (9) |
| C23′ | 0.03 (2) | 0.039 (14) | 0.05 (2) | −0.004 (15) | 0.003 (14) | −0.022 (15) |
| C24′ | 0.034 (9) | 0.042 (8) | 0.049 (9) | −0.002 (7) | −0.004 (8) | −0.021 (6) |
| C25′ | 0.038 (13) | 0.049 (12) | 0.049 (9) | −0.005 (9) | 0.000 (9) | −0.022 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C26 | 0.040 (4) | 0.076 (4) | 0.060 (4) | 0.005 (3) | −0.003 (3) | −0.048 (3) |
| C27 | 0.036 (3) | 0.043 (3) | 0.047 (3) | −0.001 (3) | 0.006 (3) | −0.024 (3) |
| C28 | 0.028 (3) | 0.043 (3) | 0.046 (3) | −0.012 (3) | 0.006 (3) | −0.022 (3) |
| C29 | 0.036 (3) | 0.033 (3) | 0.054 (3) | −0.011 (3) | 0.015 (3) | −0.025 (3) |
| C30 | 0.036 (3) | 0.026 (3) | 0.060 (4) | −0.003 (2) | 0.006 (3) | −0.014 (3) |
| C31 | 0.039 (3) | 0.039 (3) | 0.050 (3) | −0.008 (3) | −0.009 (3) | −0.008 (3) |
| C32 | 0.045 (4) | 0.045 (3) | 0.044 (3) | −0.003 (3) | −0.001 (3) | −0.020 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cu1—O4 | 1.890 (3) | C8—C9 | 1.383 (7) |
| Cu1—N1 | 1.917 (4) | C8—H8A | 0.9300 |
| Cu1—O5 | 1.919 (3) | C9—H9 | 0.9300 |
| Cu1—O1 | 1.953 (3) | C10—C11 | 1.441 (7) |
| Cu2—O9 | 1.896 (3) | C10—H10A | 0.9300 |
| Cu2—N2 | 1.909 (4) | C11—C16 | 1.406 (7) |
| Cu2—O10 | 1.915 (3) | C11—C12 | 1.422 (7) |
| Cu2—O6 | 1.946 (4) | C12—C13 | 1.423 (7) |
| Cl1—C13 | 1.733 (5) | C13—C14 | 1.366 (7) |
| Cl2—C15 | 1.756 (5) | C14—C15 | 1.371 (7) |
| Cl3—C29 | 1.738 (5) | C14—H14 | 0.9300 |
| Cl4—C31 | 1.755 (5) | C15—C16 | 1.374 (7) |
| N1—C10 | 1.278 (6) | C16—H16 | 0.9300 |
| N1—C2 | 1.463 (6) | C17—C18′ | 1.49 (2) |
| N2—C26 | 1.279 (6) | C17—C18 | 1.56 (2) |
| N2—C18′ | 1.47 (2) | C18—C19 | 1.527 (17) |
| N2—C18 | 1.55 (2) | C18—H18 | 0.9800 |
| O1—C1 | 1.290 (6) | C19—C20 | 1.50 (2) |
| O2—C1 | 1.229 (6) | C19—H19A | 0.9700 |
| O3—C7 | 1.361 (6) | C19—H19B | 0.9700 |
| O3—H3 | 0.8200 | C20—C21 | 1.38 (3) |
| O4—C12 | 1.301 (6) | C20—C25 | 1.39 (3) |
| O5—H33 | 0.8499 | C21—C22 | 1.39 (3) |
| O5—H34 | 0.8498 | C21—H21 | 0.9300 |
| O6—C17 | 1.285 (6) | C22—C23 | 1.388 (18) |
| O7—C17 | 1.220 (6) | C22—H22 | 0.9300 |
| O8—C23 | 1.366 (14) | C23—C24 | 1.38 (6) |
| O8—H8 | 0.8200 | C24—C25 | 1.39 (5) |
| O8′—C23′ | 1.38 (7) | C24—H24 | 0.9300 |
| O8′—H8′ | 0.8200 | C25—H25 | 0.9300 |
| O9—C28 | 1.303 (6) | C18′—C19′ | 1.54 (2) |
| O10—H35 | 0.8500 | C18′—H18′ | 0.9800 |
| O10—H36 | 0.8500 | C19′—C20′ | 1.499 (18) |
| O11—H37 | 0.8504 | C19′—H19C | 0.9700 |
| O11—H38 | 0.8502 | C19′—H19D | 0.9700 |
| O12—H39 | 0.8501 | C20′—C21′ | 1.39 (2) |
| O12—H40 | 0.8501 | C20′—C25′ | 1.39 (3) |
| O13—H41 | 0.8500 | C21′—C22′ | 1.39 (2) |

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|---|---|---|---|
| O13—H42 | 0.8500 | C21′—H21′ | 0.9300 |
| O12′—H39′ | 0.8500 | C22′—C23′ | 1.39 (4) |
| O12′—H40′ | 0.8500 | C22′—H22′ | 0.9300 |
| O13′—H41′ | 0.8500 | C23′—C24′ | 1.39 (7) |
| O13′—H42′ | 0.8500 | C24′—C25′ | 1.39 (3) |
| C1—C2 | 1.543 (7) | C24′—H24′ | 0.9300 |
| C2—C3 | 1.532 (7) | C25′—H25′ | 0.9300 |
| C2—H2 | 0.9800 | C26—C27 | 1.442 (7) |
| C3—C4 | 1.494 (7) | C26—H26 | 0.9300 |
| C3—H3A | 0.9700 | C27—C32 | 1.401 (7) |
| C3—H3B | 0.9700 | C27—C28 | 1.423 (7) |
| C4—C9 | 1.392 (7) | C28—C29 | 1.429 (7) |
| C4—C5 | 1.395 (7) | C29—C30 | 1.358 (7) |
| C5—C6 | 1.368 (7) | C30—C31 | 1.389 (7) |
| C5—H5A | 0.9300 | C30—H30 | 0.9300 |
| C6—C7 | 1.379 (7) | C31—C32 | 1.354 (7) |
| C6—H6 | 0.9300 | C32—H32 | 0.9300 |
| C7—C8 | 1.379 (7) | | |

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| O4—Cu1—N1 | 94.31 (16) | C15—C16—H16 | 119.7 |
| O4—Cu1—O5 | 88.98 (15) | C11—C16—H16 | 119.7 |
| N1—Cu1—O5 | 176.21 (16) | O7—C17—O6 | 125.8 (5) |
| O4—Cu1—O1 | 174.80 (15) | O7—C17—C18′ | 116.7 (9) |
| N1—Cu1—O1 | 84.68 (15) | O6—C17—C18′ | 116.2 (9) |
| O5—Cu1—O1 | 91.87 (15) | O7—C17—C18 | 116.8 (9) |
| O9—Cu2—N2 | 94.16 (16) | O6—C17—C18 | 116.0 (9) |
| O9—Cu2—O10 | 89.46 (15) | C19—C18—N2 | 118.3 (12) |
| N2—Cu2—O10 | 174.82 (18) | C19—C18—C17 | 105.5 (12) |
| O9—Cu2—O6 | 177.43 (15) | N2—C18—C17 | 102.0 (11) |
| N2—Cu2—O6 | 83.96 (16) | C19—C18—H18 | 110.2 |
| O10—Cu2—O6 | 92.31 (15) | N2—C18—H18 | 110.2 |
| C10—N1—C2 | 120.7 (4) | C17—C18—H18 | 110.2 |
| C10—N1—Cu1 | 125.7 (3) | C20—C19—C18 | 118.6 (13) |
| C2—N1—Cu1 | 113.5 (3) | C20—C19—H19A | 107.7 |
| C26—N2—C18′ | 118.4 (9) | C18—C19—H19A | 107.7 |
| C26—N2—C18 | 120.1 (8) | C20—C19—H19B | 107.7 |
| C26—N2—Cu2 | 126.6 (4) | C18—C19—H19B | 107.7 |
| C18′—N2—Cu2 | 112.6 (8) | H19A—C19—H19B | 107.1 |
| C18—N2—Cu2 | 112.7 (8) | C21—C20—C25 | 117.7 (17) |
| C1—O1—Cu1 | 114.9 (3) | C21—C20—C19 | 122.2 (17) |
| C7—O3—H3 | 109.5 | C25—C20—C19 | 120.0 (17) |
| C12—O4—Cu1 | 127.0 (3) | C20—C21—C22 | 120.7 (17) |
| Cu1—O5—H33 | 124.3 | C20—C21—H21 | 119.6 |
| Cu1—O5—H34 | 126.9 | C22—C21—H21 | 119.6 |
| H33—O5—H34 | 107.4 | C23—C22—C21 | 121.1 (14) |
| C17—O6—Cu2 | 115.6 (3) | C23—C22—H22 | 119.5 |
| C23′—O8′—H8′ | 109.5 | C21—C22—H22 | 119.5 |
| C28—O9—Cu2 | 126.2 (3) | O8—C23—C24 | 123 (3) |

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|---|---|---|---|
| Cu2—O10—H35 | 124.6 | O8—C23—C22 | 118.9 (12) |
| Cu2—O10—H36 | 130.1 | C24—C23—C22 | 118 (3) |
| H35—O10—H36 | 105.1 | C23—C24—C25 | 121 (6) |
| H39—O12—H40 | 103.0 | C23—C24—H24 | 119.4 |
| H41—O13—H42 | 108.7 | C25—C24—H24 | 119.4 |
| H39′—O12′—H40′ | 105.2 | C24—C25—C20 | 121 (4) |
| H41′—O13′—H42′ | 106.2 | C24—C25—H25 | 119.4 |
| O2—C1—O1 | 124.1 (5) | C20—C25—H25 | 119.4 |
| O2—C1—C2 | 119.7 (5) | N2—C18′—C17 | 109.7 (13) |
| O1—C1—C2 | 116.0 (5) | N2—C18′—C19′ | 107.5 (12) |
| N1—C2—C3 | 112.2 (4) | C17—C18′—C19′ | 113.0 (13) |
| N1—C2—C1 | 108.1 (4) | N2—C18′—H18′ | 108.8 |
| C3—C2—C1 | 106.6 (4) | C17—C18′—H18′ | 108.8 |
| N1—C2—H2 | 109.9 | C19′—C18′—H18′ | 108.8 |
| C3—C2—H2 | 109.9 | C20′—C19′—C18′ | 116.6 (12) |
| C1—C2—H2 | 109.9 | C20′—C19′—H19C | 108.1 |
| C4—C3—C2 | 114.6 (4) | C18′—C19′—H19C | 108.1 |
| C4—C3—H3A | 108.6 | C20′—C19′—H19D | 108.1 |
| C2—C3—H3A | 108.6 | C18′—C19′—H19D | 108.1 |
| C4—C3—H3B | 108.6 | H19C—C19′—H19D | 107.3 |
| C2—C3—H3B | 108.6 | C21′—C20′—C25′ | 115.3 (15) |
| H3A—C3—H3B | 107.6 | C21′—C20′—C19′ | 122.7 (14) |
| C9—C4—C5 | 116.4 (5) | C25′—C20′—C19′ | 122.0 (16) |
| C9—C4—C3 | 122.0 (5) | C20′—C21′—C22′ | 122.1 (14) |
| C5—C4—C3 | 121.5 (5) | C20′—C21′—H21′ | 119.0 |
| C6—C5—C4 | 121.7 (5) | C22′—C21′—H21′ | 119.0 |
| C6—C5—H5A | 119.2 | C23′—C22′—C21′ | 120 (3) |
| C4—C5—H5A | 119.2 | C23′—C22′—H22′ | 120.2 |
| C5—C6—C7 | 120.6 (5) | C21′—C22′—H22′ | 120.2 |
| C5—C6—H6 | 119.7 | O8′—C23′—C22′ | 117 (5) |
| C7—C6—H6 | 119.7 | O8′—C23′—C24′ | 122 (3) |
| O3—C7—C8 | 122.3 (5) | C22′—C23′—C24′ | 121 (5) |
| O3—C7—C6 | 118.2 (5) | C25′—C24′—C23′ | 116 (3) |
| C8—C7—C6 | 119.6 (5) | C25′—C24′—H24′ | 122.0 |
| C7—C8—C9 | 119.2 (5) | C23′—C24′—H24′ | 122.0 |
| C7—C8—H8A | 120.4 | C24′—C25′—C20′ | 125.8 (19) |
| C9—C8—H8A | 120.4 | C24′—C25′—H25′ | 117.1 |
| C8—C9—C4 | 122.4 (5) | C20′—C25′—H25′ | 117.1 |
| C8—C9—H9 | 118.8 | N2—C26—C27 | 125.2 (5) |
| C4—C9—H9 | 118.8 | N2—C26—H26 | 117.4 |
| N1—C10—C11 | 125.7 (5) | C27—C26—H26 | 117.4 |
| N1—C10—H10A | 117.2 | C32—C27—C28 | 120.9 (5) |
| C11—C10—H10A | 117.2 | C32—C27—C26 | 117.3 (5) |
| C16—C11—C12 | 120.5 (5) | C28—C27—C26 | 121.7 (5) |
| C16—C11—C10 | 117.2 (5) | O9—C28—C27 | 125.5 (5) |
| C12—C11—C10 | 122.3 (5) | O9—C28—C29 | 119.9 (4) |
| O4—C12—C11 | 124.6 (5) | C27—C28—C29 | 114.5 (5) |
| O4—C12—C13 | 120.0 (5) | C30—C29—C28 | 123.9 (5) |

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|---|---|---|---|
| C11—C12—C13 | 115.4 (5) | C30—C29—Cl3 | 118.6 (4) |
| C14—C13—C12 | 123.4 (5) | C28—C29—Cl3 | 117.6 (4) |
| C14—C13—Cl1 | 119.0 (4) | C29—C30—C31 | 119.0 (5) |
| C12—C13—Cl1 | 117.7 (4) | C29—C30—H30 | 120.5 |
| C13—C14—C15 | 119.7 (5) | C31—C30—H30 | 120.5 |
| C13—C14—H14 | 120.2 | C32—C31—C30 | 120.7 (5) |
| C15—C14—H14 | 120.2 | C32—C31—Cl4 | 121.0 (4) |
| C14—C15—C16 | 120.6 (5) | C30—C31—Cl4 | 118.2 (4) |
| C14—C15—Cl2 | 119.1 (4) | C31—C32—C27 | 120.9 (5) |
| C16—C15—Cl2 | 120.3 (4) | C31—C32—H32 | 119.6 |
| C15—C16—C11 | 120.5 (5) | C27—C32—H32 | 119.6 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O12$^{i}$ | 0.82 | 1.88 | 2.643 (10) | 154 |
| O8—H8···O7$^{ii}$ | 0.82 | 1.70 | 2.515 (9) | 175 |
| O5—H33···O6$^{iii}$ | 0.85 | 1.89 | 2.714 (6) | 164 |
| O13—H42···Cl1$^{iv}$ | 0.85 | 2.77 | 3.555 (9) | 154 |
| C26—H26···O3$^{v}$ | 0.93 | 2.44 | 3.365 (7) | 173 |

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