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# (3-{[*N*-(5-Chloro-2-hydroxyphenyl)-oxamoyl]amino}propyl)dimethylazanium perchlorate

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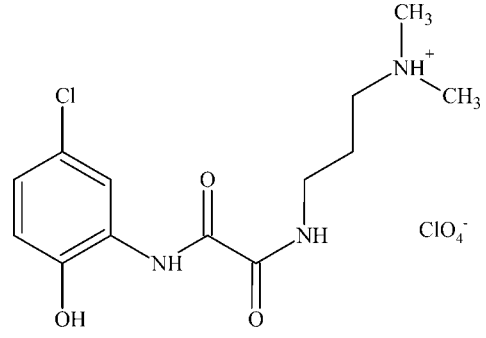
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.054; $wR$ factor = 0.179; data-to-parameter ratio = 14.2.

In the title compound, $C_{13}H_{19}ClN_3O_3^+\cdot ClO_4^-$, the 3-(dimethylammonio)propyl group of the cation is disordered over two sets of sites with occupancies 0.772 (6) and 0.228 (6). The cations are joined by pairs of N—H···O hydrogen bonds into centrosymmetric dimers and these dimers are assembled into chains along the *a*-axis direction, also through N—H···O hydrogen bonds. The perchlorate anions are linked to the hydroxy groups of the cations by O—H···O hydrogen bonds. The positively charged ammonium groups and the anions give rise to folded layers parallel to the *ab* plane.

## Related literature

For DNA binding of oxamide complexes, see: Li *et al.* (2010). For the synthesis, see: Tao *et al.* (2003).




## Experimental

### Crystal data

$C_{13}H_{19}ClN_3O_3^+\cdot ClO_4^-$
$M_r$ = 400.21
Monoclinic, $P2_1/c$
$a$ = 6.7423 (5) Å
$b$ = 12.8169 (10) Å
$c$ = 21.6454 (17) Å
$\beta$ = 98.275 (1)°
$V$ = 1851.0 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.39 mm$^{-1}$
$T$ = 296 K
0.52 × 0.28 × 0.13 mm

### Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.823, $T_{max}$ = 0.951
10727 measured reflections
4204 independent reflections
2526 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.179
$S$ = 1.03
4204 reflections
297 parameters
16 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.56 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O5 | 0.89 (4) | 1.80 (4) | 2.693 (3) | 174 (3) |
| N2—H2$A$···O2$^{i}$ | 0.88 (3) | 2.12 (3) | 2.905 (3) | 149 (2) |
| N3$A$—H3$A$···O3$^{ii}$ | 0.91 | 2.04 | 2.814 (6) | 142 |
| N3$B$—H3$B$···O3$^{ii}$ | 0.91 | 2.13 | 2.905 (18) | 143 |

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2031).

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# supporting information

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# (3-{[*N*-(5-Chloro-2-hydroxyphenyl)oxamoyl]amino}propyl)dimethylazanium perchlorate

**Xi-Teng Yue, Xiao-Wen Li and Zhi-Yong Wu**

## S1. Comment

Aimed for the DNA-binding study of a series of asymmetric *N*,*N*-disubstituted oxamide complexes (Li *et al.*, 2010), we attempted to prepare a binuclear copper(II) complex with *N*-(5-chloro-2-hydroxyphenyl)-*N*′-(3-(dimethylammonio)-propyl) oxamide ($H_3$chdpoxd). Unexpectedly, the perchlorate salt of the ligand, ($H_4$chdpoxd)$ClO_4$, **I**, was obtained.

The title compound consists of a $H_4$chdpoxd$^+$ cations and a ClO4$^-$ anions (Fig. 1). In the cation, the oxamide group adopts transoid conformation. The benzene ring is nearly parallel to the oxamide plane, with a dihedral angle of 5.39 (15)°. As for the alkyl substituent, the torsion angles of C8—N2—C9A—C10A and C8—N2—C9B—C10B are 86.1 (6)° and 117.6 (19)°, respectively.

In the crystal, the positively charged ammonium N atoms (N3A and N3B) together with the perchlorate anions form a folded layer structure with a quadrilateral pattern (Fig. 2). Such a charge-balanced layer is paralled to *a0b* plane. Cations related by an inversion center are linked by the hydrogen bonds involving oxamide groups (N2—H2A···O2, Table 3) to form a dimer. These dimers form chains parallel to *a* direction through the hydrogen bonds involving the ammonium groups (Fig. 3). The perchlorate ions append to the chains through the hydrogen bonds with phenolic hydroxy groups.

## S2. Experimental

The ligand, $H_3$chdpoxd, was prepared according to the method proposed by Tao *et al.*, (2003). To a solution of $H_3$chdpoxd (0.0299 g, 0.1 mmol) in methanol (10 ml) were added sequentially piperidine (0.2 mmol) and a solution of $Cu(ClO_4)_2.6H_2O$ (0.0742 g, 0.2 mmol) in methanol (10 ml). The mixture was intensively stirred until the solution became clear, and then 2,9-dimethyl-1,10-phenanthroline (dmphen, 0.0432 g, 0.4 mmol) in methanol (10 ml) was added. Stirring of the reaction mixture was continued at 333 K for 2 h. Although our original goal was to prepare a dinuclear copper(II) complex with chdpoxd$^{3-}$ as a bridge ligand and 2,9-dimethyl-1,10-phenanthroline as a terminal ligand, the colourless crystals of the title compound, ($H_4$chdpoxd)$ClO_4$, unexpectedly precipitated on the seventh day, after the solution had been left to stand at room temperature.

Anal. Calcd for $C_{13}H_{19}Cl_2N_3O_7$ (%): C, 39.01; H, 4.79; N, 10.50. Found: C, 38.49, H, 4.72, N, 11.06.

## S3. Refinement

The 3-(dimethylamminio)propyl group of the cation is disordered over two sets of positions, suffixed with A and B. The occupancies were refined freely to 0.772 (6) and 0.228 (6), respectively. The bond lengths C9A—C10A, C10A—C11A and C10B—C11B were restrained to 1.54 Å with DFIX instructionto to avoid the unreasonable geometries. The H atoms on the phenolic hydroxyl and the oxamide group were found in a difference Fourier map and then refined freely except for the restrain on N1—H1A bond length of 0.86 Å. Other H atoms were placed in calculated positions, with C—H = 0.93 (aromatic), 0.97 (methylene) and 0.96 (methyl) and N—H = 0.91 Å, and refined using riding model, with $U_{iso}$(H) =

1.2 $U_{eq}$, or 1.5 $U_{eq}$ for methyl groups.

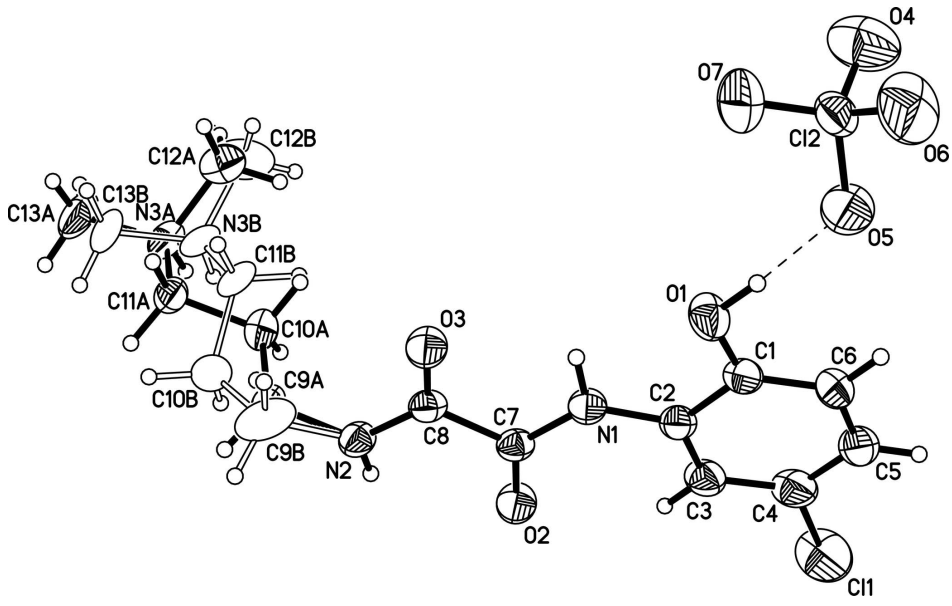


**Figure 1**

The structure of the title compound. The displacement ellipsoids are drawn at the 30% probability levels and H atoms are shown as small spheres of arbitrary radii. Two position of a disordered group are depicted in different styles. Dotted line indicate hydrogen bond.

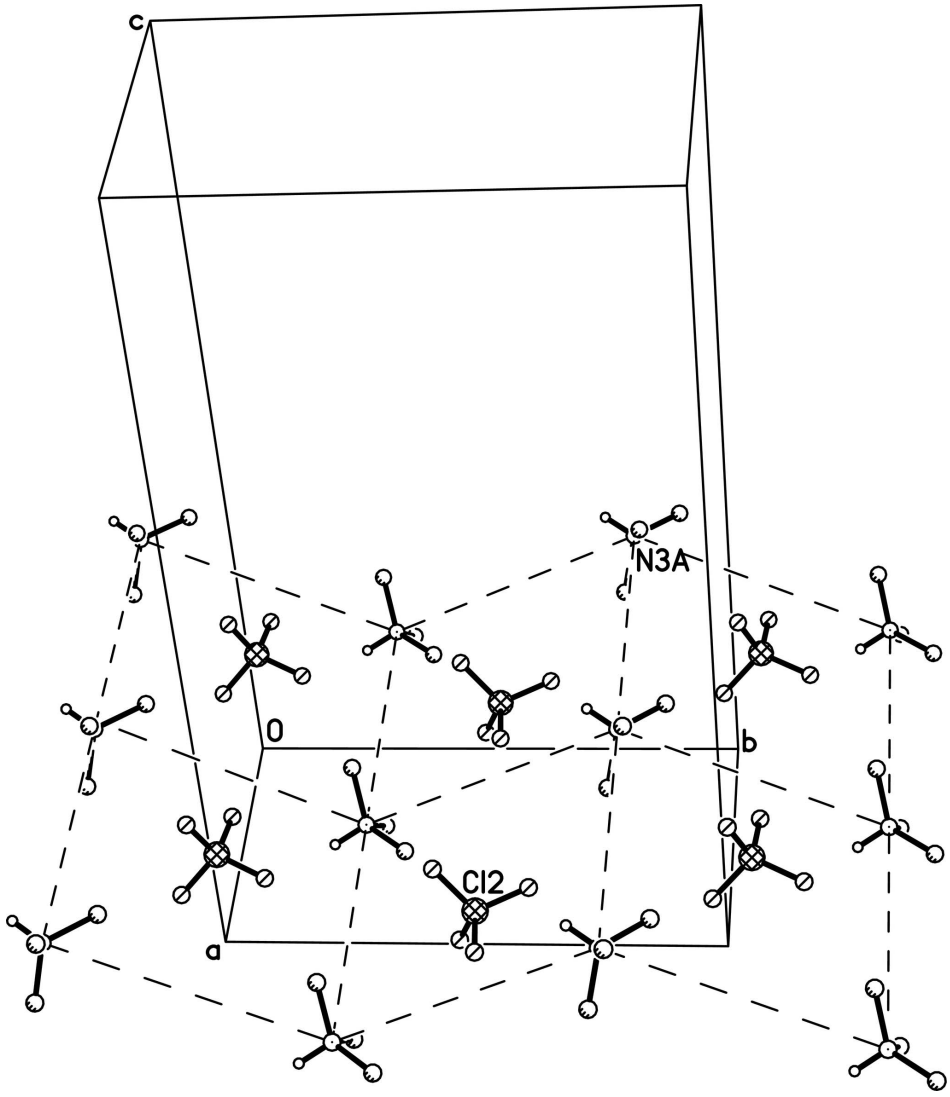


**Figure 2**

A folded layer structure parallel to *a0b* plane formed by the positively charged ammonium groups and the perchlorate ions with a quadrilateral pattern.

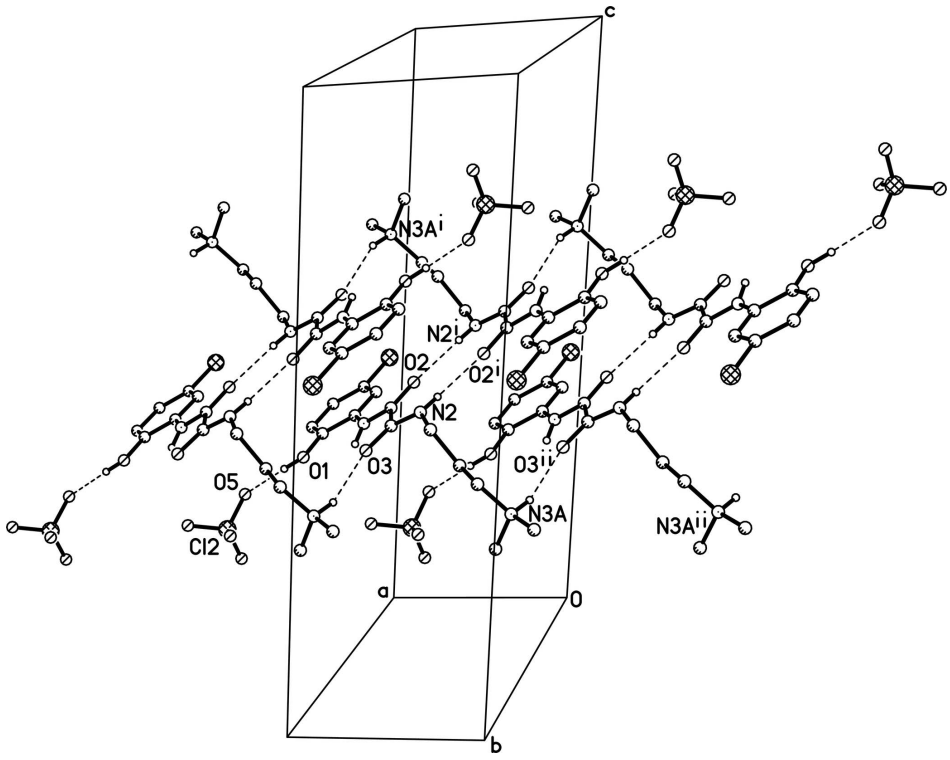


**Figure 3**

A view of a hydrogen bonding in the title compound. Symmetry codes: i = -*x* + 1, -*y* + 1, -*z* + 1; ii = *x* - 1, *y*, *z*.

**(3-{[*N*-(5-Chloro-2-hydroxyphenyl)oxamoyl]amino}propyl)dimethylazanium perchlorate**

*Crystal data*

$C_{13}H_{19}ClN_3O_3^+\cdot ClO_4^-$
$M_r$ = 400.21
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 6.7423 (5) Å
$b$ = 12.8169 (10) Å
$c$ = 21.6454 (17) Å
$\beta$ = 98.275 (1)°
$V$ = 1851.0 (2) Å$^3$
$Z$ = 4
$F(000)$ = 832
$D_x$ = 1.436 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2302 reflections
$\theta$ = 2.5–23.6°
$\mu$ = 0.39 mm$^{-1}$
$T$ = 296 K
Block, colourless
0.52 × 0.28 × 0.13 mm

*Data collection*

Bruker APEX area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.823, $T_{max}$ = 0.951
10727 measured reflections
4204 independent reflections
2526 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028
$\theta_{max}$ = 27.6°, $\theta_{min}$ = 1.9°
$h$ = −8→7
$k$ = −16→16
$l$ = −28→27

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.179$
$S = 1.03$
4204 reflections
297 parameters
16 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
$w = 1/[\sigma^2(F_o^2) + (0.0933P)^2 + 0.3342P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cl1 | 1.0226 (3) | 0.10172 (8) | 0.45657 (6) | 0.1407 (6) | |
| O1 | 1.2592 (3) | 0.49690 (16) | 0.35643 (11) | 0.0730 (6) | |
| O2 | 0.7001 (3) | 0.45689 (16) | 0.47116 (10) | 0.0706 (6) | |
| O3 | 0.8146 (3) | 0.69101 (15) | 0.40187 (9) | 0.0674 (5) | |
| N1 | 0.9394 (3) | 0.49367 (17) | 0.41071 (10) | 0.0555 (5) | |
| C1 | 1.2142 (4) | 0.4024 (2) | 0.37909 (12) | 0.0548 (6) | |
| C2 | 1.0414 (4) | 0.39935 (19) | 0.40791 (11) | 0.0521 (6) | |
| C3 | 0.9810 (5) | 0.3065 (2) | 0.43123 (13) | 0.0663 (8) | |
| H3 | 0.8656 | 0.3031 | 0.4501 | 0.080* | |
| C4 | 1.0967 (6) | 0.2179 (2) | 0.42594 (15) | 0.0814 (10) | |
| C5 | 1.2658 (6) | 0.2206 (2) | 0.39791 (14) | 0.0791 (9) | |
| H5 | 1.3401 | 0.1603 | 0.3947 | 0.095* | |
| C6 | 1.3251 (5) | 0.3137 (2) | 0.37437 (13) | 0.0665 (8) | |
| H6 | 1.4401 | 0.3164 | 0.3553 | 0.080* | |
| C7 | 0.7847 (3) | 0.5170 (2) | 0.43988 (11) | 0.0488 (6) | |
| C8 | 0.7207 (4) | 0.6308 (2) | 0.43042 (11) | 0.0499 (6) | |
| N2 | 0.5596 (3) | 0.65522 (19) | 0.45564 (10) | 0.0549 (5) | |
| C9A | 0.4556 (10) | 0.7539 (4) | 0.4449 (3) | 0.0573 (15) | 0.772 (6) |
| H9A | 0.5504 | 0.8093 | 0.4403 | 0.069* | 0.772 (6) |
| H9B | 0.3859 | 0.7706 | 0.4798 | 0.069* | 0.772 (6) |
| C10A | 0.3053 (5) | 0.7430 (3) | 0.3848 (2) | 0.0621 (11) | 0.772 (6) |
| H10A | 0.3783 | 0.7337 | 0.3498 | 0.075* | 0.772 (6) |
| H10B | 0.2245 | 0.6811 | 0.3879 | 0.075* | 0.772 (6) |
| C11A | 0.1694 (6) | 0.8358 (3) | 0.37263 (17) | 0.0574 (11) | 0.772 (6) |

| | | | | | |
|---|---|---|---|---|---|
| H11A | 0.1131 | 0.8526 | 0.4102 | 0.069* | 0.772 (6) |
| H11B | 0.2467 | 0.8954 | 0.3622 | 0.069* | 0.772 (6) |
| N3A | 0.0037 (8) | 0.8157 (4) | 0.3207 (2) | 0.0601 (14) | 0.772 (6) |
| H3A | −0.0604 | 0.7570 | 0.3308 | 0.072* | 0.772 (6) |
| C12A | 0.0819 (11) | 0.7936 (7) | 0.2601 (3) | 0.0734 (19) | 0.772 (6) |
| H12A | 0.1502 | 0.8541 | 0.2477 | 0.110* | 0.772 (6) |
| H12B | −0.0281 | 0.7768 | 0.2284 | 0.110* | 0.772 (6) |
| H12C | 0.1733 | 0.7358 | 0.2658 | 0.110* | 0.772 (6) |
| C13A | −0.1473 (9) | 0.9010 (5) | 0.3133 (3) | 0.0913 (19) | 0.772 (6) |
| H13A | −0.2004 | 0.9103 | 0.3517 | 0.137* | 0.772 (6) |
| H13B | −0.2540 | 0.8832 | 0.2807 | 0.137* | 0.772 (6) |
| H13C | −0.0850 | 0.9646 | 0.3026 | 0.137* | 0.772 (6) |
| C9B | 0.482 (3) | 0.7601 (11) | 0.4581 (12) | 0.098 (11) | 0.228 (6) |
| H9C | 0.5740 | 0.8071 | 0.4416 | 0.118* | 0.228 (6) |
| H9D | 0.4825 | 0.7783 | 0.5016 | 0.118* | 0.228 (6) |
| C10B | 0.2703 (17) | 0.7816 (12) | 0.4234 (5) | 0.071 (4) | 0.228 (6) |
| H10C | 0.1797 | 0.7278 | 0.4340 | 0.085* | 0.228 (6) |
| H10D | 0.2233 | 0.8481 | 0.4371 | 0.085* | 0.228 (6) |
| C11B | 0.2654 (18) | 0.7841 (11) | 0.3535 (5) | 0.064 (4) | 0.228 (6) |
| H11C | 0.3377 | 0.8453 | 0.3427 | 0.077* | 0.228 (6) |
| H11D | 0.3351 | 0.7231 | 0.3411 | 0.077* | 0.228 (6) |
| N3B | 0.061 (2) | 0.7862 (15) | 0.3176 (6) | 0.059 (5) | 0.228 (6) |
| H3B | −0.0068 | 0.7323 | 0.3324 | 0.071* | 0.228 (6) |
| C12B | 0.067 (4) | 0.763 (2) | 0.2495 (8) | 0.109 (13) | 0.228 (6) |
| H12D | 0.1776 | 0.7996 | 0.2359 | 0.164* | 0.228 (6) |
| H12E | −0.0557 | 0.7849 | 0.2251 | 0.164* | 0.228 (6) |
| H12F | 0.0847 | 0.6892 | 0.2441 | 0.164* | 0.228 (6) |
| C13B | −0.059 (3) | 0.8810 (14) | 0.3249 (9) | 0.078 (6) | 0.228 (6) |
| H13D | −0.0671 | 0.8921 | 0.3683 | 0.118* | 0.228 (6) |
| H13E | −0.1916 | 0.8723 | 0.3024 | 0.118* | 0.228 (6) |
| H13F | 0.0036 | 0.9402 | 0.3085 | 0.118* | 0.228 (6) |
| Cl2 | 1.62273 (11) | 0.56840 (6) | 0.24890 (4) | 0.0702 (3) | |
| O4 | 1.5678 (6) | 0.5285 (3) | 0.18955 (14) | 0.1317 (11) | |
| O5 | 1.5772 (4) | 0.4944 (2) | 0.29410 (13) | 0.1029 (9) | |
| O6 | 1.8348 (4) | 0.5793 (3) | 0.25838 (18) | 0.1327 (12) | |
| O7 | 1.5414 (6) | 0.6642 (3) | 0.25755 (16) | 0.1554 (15) | |
| H1 | 1.365 (5) | 0.491 (3) | 0.3362 (15) | 0.081 (10)* | |
| H1A | 0.955 (5) | 0.544 (2) | 0.3857 (14) | 0.107 (13)* | |
| H2A | 0.501 (4) | 0.601 (2) | 0.4705 (13) | 0.059 (8)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.2476 (16) | 0.0511 (5) | 0.1406 (10) | 0.0012 (7) | 0.0861 (10) | 0.0201 (5) |
| O1 | 0.0726 (13) | 0.0608 (12) | 0.0946 (15) | −0.0003 (10) | 0.0425 (12) | 0.0074 (10) |
| O2 | 0.0703 (12) | 0.0647 (12) | 0.0842 (14) | 0.0021 (9) | 0.0365 (11) | 0.0226 (10) |
| O3 | 0.0708 (12) | 0.0570 (11) | 0.0813 (13) | 0.0003 (9) | 0.0340 (10) | 0.0185 (9) |
| N1 | 0.0588 (13) | 0.0515 (13) | 0.0598 (13) | −0.0011 (10) | 0.0205 (11) | 0.0115 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.0622 (15) | 0.0564 (15) | 0.0465 (13) | −0.0016 (12) | 0.0105 (12) | 0.0009 (11) |
| C2 | 0.0611 (15) | 0.0497 (14) | 0.0460 (13) | 0.0003 (11) | 0.0094 (12) | 0.0019 (11) |
| C3 | 0.089 (2) | 0.0538 (16) | 0.0597 (16) | −0.0027 (14) | 0.0243 (15) | 0.0055 (13) |
| C4 | 0.136 (3) | 0.0487 (17) | 0.0644 (18) | 0.0009 (17) | 0.030 (2) | 0.0056 (13) |
| C5 | 0.120 (3) | 0.0584 (18) | 0.0604 (17) | 0.0236 (18) | 0.0167 (18) | −0.0043 (14) |
| C6 | 0.0768 (19) | 0.0693 (19) | 0.0543 (15) | 0.0127 (15) | 0.0123 (14) | −0.0092 (14) |
| C7 | 0.0443 (12) | 0.0567 (15) | 0.0457 (12) | −0.0036 (11) | 0.0073 (10) | 0.0086 (11) |
| C8 | 0.0474 (13) | 0.0581 (14) | 0.0444 (13) | −0.0010 (11) | 0.0066 (11) | 0.0090 (11) |
| N2 | 0.0482 (12) | 0.0598 (14) | 0.0583 (13) | 0.0049 (10) | 0.0139 (10) | 0.0147 (11) |
| C9A | 0.055 (4) | 0.053 (3) | 0.065 (3) | 0.014 (2) | 0.015 (2) | 0.015 (2) |
| C10A | 0.057 (2) | 0.056 (2) | 0.073 (3) | 0.0112 (17) | 0.009 (2) | 0.001 (2) |
| C11A | 0.060 (2) | 0.050 (2) | 0.064 (2) | 0.0094 (17) | 0.0180 (18) | 0.0115 (17) |
| N3A | 0.045 (3) | 0.055 (3) | 0.081 (3) | 0.004 (2) | 0.011 (2) | 0.022 (2) |
| C12A | 0.078 (4) | 0.071 (4) | 0.067 (3) | −0.015 (3) | −0.004 (3) | 0.005 (3) |
| C13A | 0.071 (4) | 0.079 (4) | 0.124 (5) | 0.033 (3) | 0.015 (3) | 0.042 (3) |
| C9B | 0.042 (11) | 0.15 (2) | 0.102 (19) | −0.002 (12) | 0.004 (11) | 0.062 (15) |
| C10B | 0.067 (9) | 0.081 (10) | 0.070 (9) | 0.012 (7) | 0.030 (7) | 0.024 (8) |
| C11B | 0.071 (9) | 0.049 (8) | 0.073 (9) | 0.011 (7) | 0.010 (7) | 0.034 (7) |
| N3B | 0.048 (9) | 0.061 (11) | 0.067 (9) | 0.005 (7) | 0.007 (6) | 0.037 (7) |
| C12B | 0.15 (2) | 0.10 (2) | 0.076 (14) | −0.075 (18) | 0.012 (13) | −0.003 (12) |
| C13B | 0.070 (13) | 0.057 (10) | 0.115 (15) | 0.032 (10) | 0.034 (12) | 0.037 (10) |
| Cl2 | 0.0760 (5) | 0.0676 (5) | 0.0745 (5) | 0.0045 (3) | 0.0358 (4) | 0.0035 (3) |
| O4 | 0.178 (3) | 0.127 (3) | 0.0937 (19) | −0.032 (2) | 0.032 (2) | −0.0124 (18) |
| O5 | 0.0856 (16) | 0.113 (2) | 0.119 (2) | 0.0062 (14) | 0.0467 (15) | 0.0427 (16) |
| O6 | 0.0826 (17) | 0.139 (3) | 0.188 (3) | −0.0109 (17) | 0.0595 (19) | 0.024 (2) |
| O7 | 0.220 (4) | 0.114 (3) | 0.148 (3) | 0.087 (3) | 0.081 (3) | 0.015 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.733 (3) | N3A—C13A | 1.487 (6) |
| O1—C1 | 1.357 (3) | N3A—C12A | 1.507 (7) |
| O1—H1 | 0.89 (4) | N3A—H3A | 0.9100 |
| O2—C7 | 1.220 (3) | C12A—H12A | 0.9600 |
| O3—C8 | 1.221 (3) | C12A—H12B | 0.9600 |
| N1—C2 | 1.396 (3) | C12A—H12C | 0.9600 |
| N1—C7 | 1.329 (3) | C13A—H13A | 0.9600 |
| N2—C8 | 1.321 (3) | C13A—H13B | 0.9600 |
| C7—C8 | 1.527 (4) | C13A—H13C | 0.9600 |
| N1—H1A | 0.857 (10) | C9B—C10B | 1.537 (10) |
| C1—C6 | 1.372 (4) | C9B—H9C | 0.9700 |
| C1—C2 | 1.399 (4) | C9B—H9D | 0.9700 |
| C2—C3 | 1.377 (4) | C10B—C11B | 1.508 (7) |
| C3—C4 | 1.392 (4) | C10B—H10C | 0.9700 |
| C3—H3 | 0.9300 | C10B—H10D | 0.9700 |
| C4—C5 | 1.366 (5) | C11B—N3B | 1.483 (10) |
| C5—C6 | 1.379 (4) | C11B—H11C | 0.9700 |
| C5—H5 | 0.9300 | C11B—H11D | 0.9700 |
| C6—H6 | 0.9300 | N3B—C13B | 1.480 (10) |

| | | | |
|---|---|---|---|
| N2—C9B | 1.447 (10) | N3B—C12B | 1.513 (10) |
| N2—C9A | 1.448 (4) | N3B—H3B | 0.9100 |
| N2—H2A | 0.88 (3) | C12B—H12D | 0.9600 |
| C9A—C10A | 1.535 (6) | C12B—H12E | 0.9600 |
| C9A—H9A | 0.9700 | C12B—H12F | 0.9600 |
| C9A—H9B | 0.9700 | C13B—H13D | 0.9600 |
| C10A—C11A | 1.501 (4) | C13B—H13E | 0.9600 |
| C10A—H10A | 0.9700 | C13B—H13F | 0.9600 |
| C10A—H10B | 0.9700 | Cl2—O7 | 1.369 (3) |
| C11A—N3A | 1.489 (6) | Cl2—O4 | 1.383 (3) |
| C11A—H11A | 0.9700 | Cl2—O6 | 1.422 (3) |
| C11A—H11B | 0.9700 | Cl2—O5 | 1.428 (2) |

| | | | |
|---|---|---|---|
| C1—O1—H1 | 110 (2) | H11A—C11A—H11B | 108.0 |
| C7—N1—C2 | 129.8 (2) | C13A—N3A—C11A | 112.4 (4) |
| C7—N1—H1A | 108 (3) | C13A—N3A—C12A | 111.4 (5) |
| C2—N1—H1A | 121 (3) | C11A—N3A—C12A | 111.8 (5) |
| O1—C1—C6 | 124.0 (3) | C13A—N3A—H3A | 107.0 |
| O1—C1—C2 | 115.4 (2) | C11A—N3A—H3A | 107.0 |
| C6—C1—C2 | 120.5 (3) | C12A—N3A—H3A | 107.0 |
| C3—C2—N1 | 124.0 (2) | N2—C9B—C10B | 117.7 (11) |
| C3—C2—C1 | 119.8 (2) | N2—C9B—H9C | 107.9 |
| N1—C2—C1 | 116.2 (2) | C10B—C9B—H9C | 107.9 |
| C2—C3—C4 | 118.4 (3) | N2—C9B—H9D | 107.9 |
| C2—C3—H3 | 120.8 | C10B—C9B—H9D | 107.9 |
| C4—C3—H3 | 120.8 | H9C—C9B—H9D | 107.2 |
| C5—C4—C3 | 121.9 (3) | C11B—C10B—C9B | 112.3 (12) |
| C5—C4—Cl1 | 119.8 (3) | C11B—C10B—H10C | 109.2 |
| C3—C4—Cl1 | 118.3 (3) | C9B—C10B—H10C | 109.2 |
| C4—C5—C6 | 119.4 (3) | C11B—C10B—H10D | 109.2 |
| C4—C5—H5 | 120.3 | C9B—C10B—H10D | 109.2 |
| C6—C5—H5 | 120.3 | H10C—C10B—H10D | 107.9 |
| C1—C6—C5 | 119.9 (3) | N3B—C11B—C10B | 114.2 (10) |
| C1—C6—H6 | 120.0 | N3B—C11B—H11C | 108.7 |
| C5—C6—H6 | 120.0 | C10B—C11B—H11C | 108.7 |
| O2—C7—N1 | 125.7 (2) | N3B—C11B—H11D | 108.7 |
| O2—C7—C8 | 122.1 (2) | C10B—C11B—H11D | 108.7 |
| N1—C7—C8 | 112.2 (2) | H11C—C11B—H11D | 107.6 |
| O3—C8—N2 | 125.2 (2) | C13B—N3B—C11B | 116.1 (13) |
| O3—C8—C7 | 120.9 (2) | C13B—N3B—C12B | 111.0 (12) |
| N2—C8—C7 | 113.8 (2) | C11B—N3B—C12B | 110.6 (12) |
| C8—N2—C9B | 124.0 (14) | C13B—N3B—H3B | 106.1 |
| C8—N2—C9A | 123.2 (4) | C11B—N3B—H3B | 106.1 |
| C8—N2—H2A | 113.7 (18) | C12B—N3B—H3B | 106.1 |
| C9B—N2—H2A | 122 (2) | N3B—C12B—H12D | 109.5 |
| C9A—N2—H2A | 120.9 (18) | N3B—C12B—H12E | 109.5 |
| N2—C9A—C10A | 107.6 (3) | H12D—C12B—H12E | 109.5 |
| N2—C9A—H9A | 110.2 | N3B—C12B—H12F | 109.5 |

| | | | |
|---|---|---|---|
| C10A—C9A—H9A | 110.2 | H12D—C12B—H12F | 109.5 |
| N2—C9A—H9B | 110.2 | H12E—C12B—H12F | 109.5 |
| C10A—C9A—H9B | 110.2 | N3B—C13B—H13D | 109.5 |
| H9A—C9A—H9B | 108.5 | N3B—C13B—H13E | 109.5 |
| C11A—C10A—C9A | 112.9 (3) | H13D—C13B—H13E | 109.5 |
| C11A—C10A—H10A | 109.0 | N3B—C13B—H13F | 109.5 |
| C9A—C10A—H10A | 109.0 | H13D—C13B—H13F | 109.5 |
| C11A—C10A—H10B | 109.0 | H13E—C13B—H13F | 109.5 |
| C9A—C10A—H10B | 109.0 | O7—Cl2—O4 | 113.6 (2) |
| H10A—C10A—H10B | 107.8 | O7—Cl2—O6 | 107.9 (3) |
| N3A—C11A—C10A | 111.6 (3) | O4—Cl2—O6 | 107.4 (2) |
| N3A—C11A—H11A | 109.3 | O7—Cl2—O5 | 111.86 (19) |
| C10A—C11A—H11A | 109.3 | O4—Cl2—O5 | 109.62 (19) |
| N3A—C11A—H11B | 109.3 | O6—Cl2—O5 | 106.11 (18) |
| C10A—C11A—H11B | 109.3 | | |

| | | | |
|---|---|---|---|
| C7—N1—C2—C1 | −172.9 (3) | O2—C7—C8—N2 | −4.1 (4) |
| C7—N1—C2—C3 | 7.4 (4) | N1—C7—C8—N2 | 176.1 (2) |
| O1—C1—C2—C3 | 178.7 (2) | O3—C8—N2—C9B | −6.1 (9) |
| C6—C1—C2—C3 | −0.6 (4) | C7—C8—N2—C9B | 173.8 (8) |
| O1—C1—C2—N1 | −1.1 (3) | O3—C8—N2—C9A | 9.3 (5) |
| C6—C1—C2—N1 | 179.7 (2) | C7—C8—N2—C9A | −170.8 (3) |
| N1—C2—C3—C4 | −179.5 (3) | C8—N2—C9A—C10A | 86.1 (6) |
| C1—C2—C3—C4 | 0.8 (4) | C9B—N2—C9A—C10A | −176 (8) |
| C2—C3—C4—C5 | −0.7 (5) | N2—C9A—C10A—C11A | 172.9 (4) |
| C2—C3—C4—Cl1 | 178.9 (2) | C9A—C10A—C11A—N3A | −170.2 (5) |
| C3—C4—C5—C6 | 0.4 (5) | C10A—C11A—N3A—C13A | 172.5 (5) |
| Cl1—C4—C5—C6 | −179.2 (2) | C10A—C11A—N3A—C12A | −61.4 (6) |
| O1—C1—C6—C5 | −178.9 (3) | C8—N2—C9B—C10B | 117.6 (19) |
| C2—C1—C6—C5 | 0.3 (4) | C9A—N2—C9B—C10B | 27 (5) |
| C4—C5—C6—C1 | −0.2 (5) | N2—C9B—C10B—C11B | −73 (3) |
| C2—N1—C7—O2 | 0.1 (4) | C9B—C10B—C11B—N3B | 169.9 (13) |
| C2—N1—C7—C8 | 179.9 (2) | C10B—C11B—N3B—C13B | 65.8 (18) |
| O2—C7—C8—O3 | 175.8 (2) | C10B—C11B—N3B—C12B | −166.5 (16) |
| N1—C7—C8—O3 | −4.0 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O5 | 0.89 (4) | 1.80 (4) | 2.693 (3) | 174 (3) |
| N2—H2*A*···O2$^{i}$ | 0.88 (3) | 2.12 (3) | 2.905 (3) | 149 (2) |
| N3*A*—H3*A*···O3$^{ii}$ | 0.91 | 2.04 | 2.814 (6) | 142 |
| N3*B*—H3*B*···O3$^{ii}$ | 0.91 | 2.13 | 2.905 (18) | 143 |

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