

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

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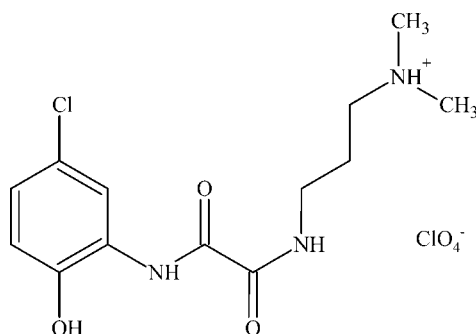
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.054; wR factor = 0.179; data-to-parameter ratio = 14.2.

In the title compound, $\text{C}_{13}\text{H}_{19}\text{ClN}_3\text{O}_3^+\cdot\text{ClO}_4^-$, 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 $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into centrosymmetric dimers and these dimers are assembled into chains along the a -axis direction, also through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The perchlorate anions are linked to the hydroxy groups of the cations by $\text{O}-\text{H}\cdots\text{O}$ 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

$\text{C}_{13}\text{H}_{19}\text{ClN}_3\text{O}_3^+\cdot\text{ClO}_4^-$
 $M_r = 400.21$
 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) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.39$ mm⁻¹
 $T = 296$ K
 $0.52 \times 0.28 \times 0.13$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

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
 $\Delta\rho_{\text{max}} = 0.56$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O5}$	0.89 (4)	1.80 (4)	2.693 (3)	174 (3)
$\text{N2}-\text{H2A}\cdots\text{O2}^{\text{i}}$	0.88 (3)	2.12 (3)	2.905 (3)	149 (2)
$\text{N3A}-\text{H3A}\cdots\text{O3}^{\text{ii}}$	0.91	2.04	2.814 (6)	142
$\text{N3B}-\text{H3B}\cdots\text{O3}^{\text{ii}}$	0.91	2.13	2.905 (8)	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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 Tao, R.-J., Zang, S.-Q., Mei, C.-Z., Wang, Q.-L., Lou, B.-Y., Niu, J.-Y., Cheng, Y.-X. & Liao, D.-Z. (2003). *J. Cluster Sci.* **14**, 459–469.

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

X.-T. Yue, X.-W. Li and Z.-Y. Wu

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 (H₃chdpoxd). Unexpectedly, the perchlorate salt of the ligand, (H₄chdpoxd)ClO₄, **I**, was obtained.

The title compound consists of a H₄chdpoxd⁺ 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 parallel to *ab* 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.

Experimental

The ligand, H₃chdpoxd, was prepared according to the method proposed by Tao *et al.*, (2003). To a solution of H₃chdpoxd (0.0299 g, 0.1 mmol) in methanol (10 ml) were added sequentially piperidine (0.2 mmol) and a solution of Cu(ClO₄)₂·6H₂O (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 chdpoxd³⁻ as a bridge ligand and 2,9-dimethyl-1,10-phenanthroline as a terminal ligand, the colourless crystals of the title compound, (H₄chdpoxd)ClO₄, unexpectedly precipitated on the seventh day, after the solution had been left to stand at room temperature.

Anal. Calcd for C₁₃H₁₉Cl₂N₃O₇ (%): C, 39.01; H, 4.79; N, 10.50. Found: C, 38.49, H, 4.72, N, 11.06.

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 instruction 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

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(aromatic), 0.97 (methylene) and 0.96 (methyl) and N—H = 0.91 Å, and refined using riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}$, or $1.5 U_{\text{eq}}$ for methyl groups.

Figures

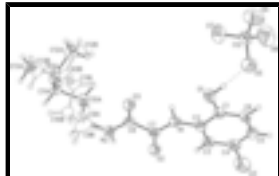


Fig. 1. The structure of the title compound. The displacement ellipsoids are drawn at the 30% probability levels and H atoms are shown as small spheres of arbitrary radii. Two positions of a disordered group are depicted in different styles. Dotted lines indicate hydrogen bonds.

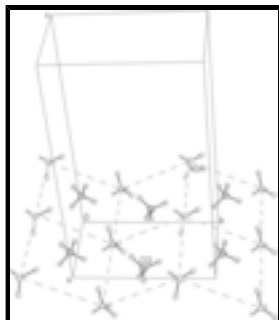


Fig. 2. A folded layer structure parallel to ab plane formed by the positively charged ammonium groups and the perchlorate ions with a quadrilateral pattern.

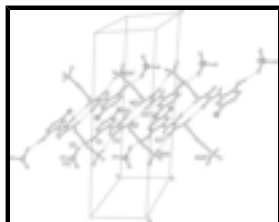


Fig. 3. A view of a hydrogen bonding in the title compound. Symmetry codes: i = $-x + 1, -y + 1, -z + 1$; ii = $x - 1, y, z$.

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

Crystal data

$\text{C}_{13}\text{H}_{19}\text{ClN}_3\text{O}_3^+ \cdot \text{ClO}_4^-$

$M_r = 400.21$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 6.7423\ (5)\ \text{\AA}$

$b = 12.8169\ (10)\ \text{\AA}$

$c = 21.6454\ (17)\ \text{\AA}$

$\beta = 98.275\ (1)^\circ$

$V = 1851.0\ (2)\ \text{\AA}^3$

$Z = 4$

$F(000) = 832$

$D_x = 1.436\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2302 reflections

$\theta = 2.5\text{--}23.6^\circ$

$\mu = 0.39\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colourless

$0.52 \times 0.28 \times 0.13\ \text{mm}$

Data collection

Bruker APEX area-detector
diffractometer

4204 independent reflections

Radiation source: fine-focus sealed tube graphite	2526 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.028$
φ and ω scans	$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -8 \rightarrow 7$
$T_{\text{min}} = 0.823$, $T_{\text{max}} = 0.951$	$k = -16 \rightarrow 16$
10727 measured reflections	$l = -28 \rightarrow 27$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.054$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.179$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0933P)^2 + 0.3342P]$
4204 reflections	where $P = (F_o^2 + 2F_c^2)/3$
297 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
16 restraints	$\Delta\rho_{\text{max}} = 0.56 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.26 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C11	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)	

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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)
C12	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 (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	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)
C12	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 (\AA , $^\circ$)

C11—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

supplementary materials

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—C11	119.8 (3)	C11B—C10B—H10C	109.2
C3—C4—C11	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—C12—O4	113.6 (2)
H10A—C10A—H10B	107.8	O7—C12—O6	107.9 (3)
N3A—C11A—C10A	111.6 (3)	O4—C12—O6	107.4 (2)
N3A—C11A—H11A	109.3	O7—C12—O5	111.86 (19)
C10A—C11A—H11A	109.3	O4—C12—O5	109.62 (19)
N3A—C11A—H11B	109.3	O6—C12—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—C11	178.9 (2)	C9A—C10A—C11A—N3A	-170.2 (5)
C3—C4—C5—C6	0.4 (5)	C10A—C11A—N3A—C13A	172.5 (5)
C11—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 (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots O5	0.89 (4)	1.80 (4)	2.693 (3)	174 (3)

supplementary materials

N2—H2A···O2 ⁱ	0.88 (3)	2.12 (3)	2.905 (3)	149 (2)
N3A—H3A···O3 ⁱⁱ	0.91	2.04	2.814 (6)	142.
N3B—H3B···O3 ⁱⁱ	0.91	2.13	2.905 (18)	143.

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

Fig. 2

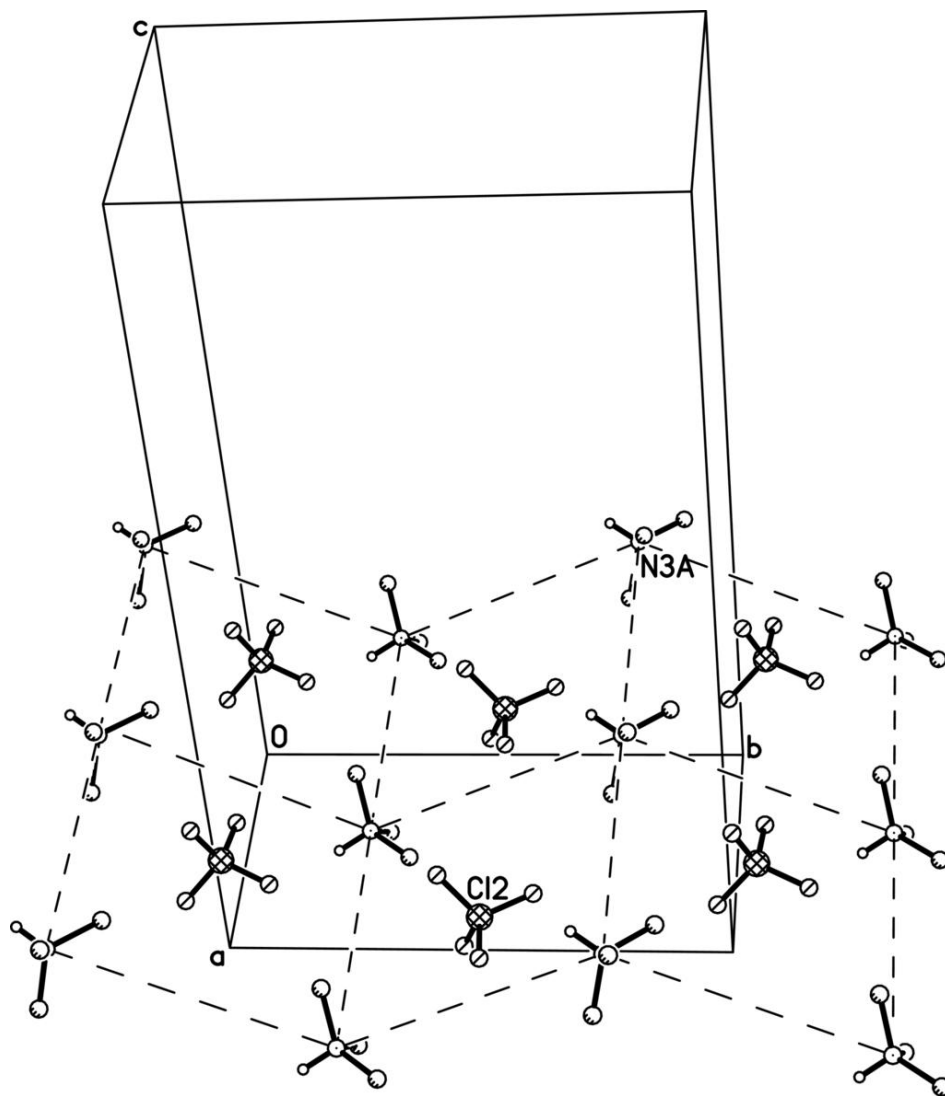


Fig. 3

