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2,2′,2″,2‴-(3,6-Dioxaoctane-1,8-diyldinitrilo)tetrabenzimidazolium tetrakis(perchlorate) dihydrate

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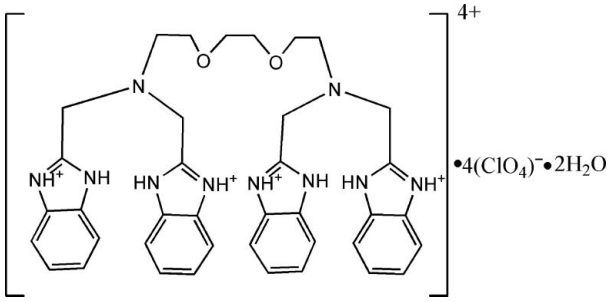
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.015 Å; R factor = 0.078; wR factor = 0.225; data-to-parameter ratio = 12.7.

In the title crystal structure, $C_{38}H_{44}N_{10}O_2^{4+}\cdot 4ClO_4^-\cdot 2H_2O$, components are linked into a two-dimensional framework by a combination of N—H···O, C—H···O, O—H···O and N—H···N hydrogen bonds. In addition, weak π–π stacking interactions and anion–π noncovalent interactions between perchlorate anions and heteroaromatic imidazole rings [O···Cg = 3.328 (10) and 3.386 (11) Å; Cg is the centroid of an imidazole ring] consolidate the crystal structure.

Related literature

For related literature, see: Hendriks *et al.* (1982); Li *et al.* (2005); Qiu *et al.* (2005); Zhang *et al.* (2005); Zhou *et al.* (2007); Gamez *et al.* (2007); Kim *et al.* (2004); Bruker (2001).



Experimental

Crystal data

$C_{38}H_{44}N_{10}O_2^{4+}\cdot 4ClO_4^-\cdot 2H_2O$
M_r = 1106.66
Monoclinic, $P2_1$
a = 10.6093 (7) Å
b = 17.6593 (12) Å
c = 14.0164 (8) Å
β = 112.238 (1)°
V = 2430.7 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 292 (2) K
0.20 × 0.10 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.925, T_{max} = 0.987
23871 measured reflections
8332 independent reflections
4522 reflections with $I > 2\sigma(I)$
R_{int} = 0.117

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.225
S = 0.88
8332 reflections
655 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 3891 Friedel pairs
Flack parameter: 0.00 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
O19—H19B···O16^{i}	0.84	2.61	2.962 (16)	107 (3)
C4—H4A···O3^{ii}	0.97	2.57	3.446 (11)	150
C18—H18···O12^{iii}	0.93	2.53	3.339 (14)	146
C28—H28···O1^{iv}	0.93	2.52	3.394 (12)	156
O20—H20B···O6^{iv}	0.86	2.07	2.861 (9)	153
O20—H20A···O12^{iv}	0.89	2.10	2.887 (14)	146
O20—H20A···O13^{iv}	0.89	2.32	3.123 (14)	150
O19—H19B···O9^{i}	0.84	2.49	2.991 (11)	119 (3)
C23—H23A···O18^{v}	0.97	2.33	3.206 (13)	150
N2—H2C···O4	0.86	1.95	2.799 (9)	170
N8—H8A···O14	0.86	2.14	2.958 (14)	158
N3—H3C···O19	0.86	1.91	2.746 (9)	165
N4—H4C···O19	0.86	2.05	2.859 (10)	156
N5—H5C···O17	0.86	2.15	3.006 (15)	173
N10—H10A···O7	0.86	1.94	2.786 (9)	168
C5—H5A···O1	0.97	2.26	2.969 (10)	129
N7—H7A···O20	0.86	2.02	2.855 (10)	164
N9—H9A···O20	0.86	2.03	2.875 (10)	168
C31—H31A···O2	0.97	2.32	2.990 (10)	126

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

Table 2
π–π stacking interaction geometry (°, Å).

$Cg1$ is the centroid defined by N2/N3/C6/C7/C12; $Cg2$ is the centroid defined by N9/N10/C32/C33/C38; $Cg3$ is the centroid defined by C7–C12; $Cg4$ is the centroid defined by C15–C20; $Cg5$ is the centroid defined by C25–C30; $Cg6$ is the centroid defined by C33–C38.

Cg_i	Cg_j	Dihedral angle	Centroid distance	Interplanar spacing
$Cg1$	$Cg4^{vi}$	3.1 (1)	4.020 (5)	3.489 (5)
$Cg1$	$Cg5^{vii}$	3.8 (1)	3.941 (5)	3.472 (5)
$Cg2$	$Cg4^{viii}$	2.5 (1)	3.931 (5)	3.535 (5)
$Cg3$	$Cg5^{vii}$	2.9 (1)	3.867 (6)	3.467 (6)
$Cg4$	$Cg6^{i}$	2.3 (1)	3.767 (6)	3.510 (6)

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z$; (vi) $x-1, y, z$; (vii) $-x+1, y-\frac{1}{2}, -z+1$; (viii) $-x+2, y+\frac{1}{2}, -z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2522).

References

Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gamez, P., Mooibroek, T. J., Teat, S. J. & Reedijk, J. (2007). *Acc. Chem. Res.* **40**, 435–444.
Hendriks, H.-M.-J., Birker, J.-M.-W.-L., Van, R.-J., Verschoor, G.-C. & Reedijk, A. (1982). *J. Am. Chem. Soc.* **104**, 3607–3617.
Kim, D., Tarakeshwar, P. & Kim, K. S. (2004). *J. Phys. Chem. A*, **108**, 1250–1258.
Li, J., Meng, X.-G. & Liao, Z.-R. (2005). *Acta Cryst.* E**61**, o3421–o3423.
Qiu, J.-H., Liao, Z.-R., Meng, X.-G., Zhu, L., Wang, Z.-M. & Yu, K.-B. (2005). *Polyhedron*, **24**, 1617–1623.
Sheldrick, G. M. (1997). *SADABS* (Version 2.10), *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Zhang, Y., Liao, Z.-R., Li, D.-F. & Meng, X.-G. (2005). *Acta Cryst.* E**61**, o3396–o3397.
Zhou, C.-S., Hu, S. & Meng, X.-G. (2007). *Acta Cryst.* E**63**, o2800.

supplementary materials

Acta Cryst. (2007). E**63**, o4334-o4335 [**doi:10.1107/S160053680704980X**]

2,2',2'',2'''-(3,6-Dioxaoctane-1,8-diyldinitrilo)tetrabenzimidazolium tetrakis(perchlorate) dihydrate

C.-S. Zhou, Y.-M. Pei and X.-G. Meng

Comment

We recently reported the unexpectedly obtained crystal structure of a polybenzimidazolyl-containing ligand, H_4EGTB·4NO_3 (EGTB = μ_2-*N*,*N*, *N*', *N*'-tetrakis(Benzimidazol-2-ylmethyl)- 3,6- dioxaoctane-1,8-diamine), as part of our further research work on the crystallization behavior of EGTB with other anions (Zhou *et al.* 2007, Zhang *et al.*,2005; Li *et al.*, 2005; Qiu *et al.*, 2005), we reported here another organic salt formed by EGTB and ClO_4^{-1}, namely 2,2',2'',2'''[N,*N*,*N*',*N*'- (3,6- dioxaoctane-1,8-diyldinitrilo)tetramethylene] tetrakis(benzimidazolium) tetra-perchlorate, $(C_{38}H_{42}N_{10}O_2)^{4+}$·4($ClO_4^-$)·2($H_2O$), (I).

Unlike the earlier reported structure (Zhou *et al.*, 2007) which crystallize in the centrosymmetric space group $P2_1/c$, the title compound crystallize in $P2_1$ space group with four perchlorate anions and two water solvent molecules in the asymmetric unit. The dihedral angles between the two benzimidazole(abbr bzim) groups on each end of EGTB are 2.8 (3) and 3.0 (3)°, respectively. Two water molecules occupy the inner sphere formed by the end-on bzim groups. The four perchlorate anions lie outside of the end-on bzim groups.

In the supramolecular structure of (I), by a combination of a series of H-bonds (Table 1), the EGTB tetra-cations, perchlorate anions and water molecules are interlinked into a two-dimensional network running parallel to the [101] direction (Fig.2). The two-dimensional H-bonding network is consolidated by weak π–π stacking interactions listed in Table 2 except for *Cg*1—*Cg*4 interaction. By a combination of *Cg*1—*Cg*5 and *Cg*3—*Cg*5 interactions, only half of the adjacent EGTB tetra-cations are interlinked into a one-dimensional chain running parallel to the [010] direction, which is generated by the 2~1~ screw axis along (1/2, *y*, 1/2). Similarly, combinations of *Cg*2—*Cg*4 and *Cg*4—*Cg*6 interactions also link the other half of EGTB tetra-cations into a second one-dimensional chain, which is also produced by the 2~1~ screw axis, but this time along (1, *y*, 0) direction.

Another weak π–π stacking interactions, *Cg*1—*Cg*4, along the [100] direction link the adjacent two-dimensional network into a three-dimensional network. Through careful analysis using *PLATON* (Spek, 2003) we found that in the crystal structure anion-π interactions (Kim *et al.*, 2004; Gamez *et al.*, 2007) are also present between Cl1-containing perchlorate anion and hetero-aromatic imidazole ring at (*x* − 1, *y*,*z*) defined by N4/N5/C14/C15/C20, *Cg*7, and between Cl2-containing perchlorate anion and imidazole ring at (1 + *x*, *y*, *z*) defined by N7/N8/C24/C25/C30, *Cg*8, respectively. The related parameters for the former is: O3···*Cg*7 = 3.323 (10) Å, Cl1···*Cg*7 = 3.938 (4)Å and Cl1—O3···*Cg*7 = 104.6 (4)°; for the latter, O10···*Cg*8 = 3.386 (11) Å, Cl2···*Cg*8 = 3.869Å and Cl2—O10···*Cg*8 = 99.8 (5)°. The two anion-π interactions further strengthened the three-dimensional network along the [100] direction as *Cg*1—*Cg*4 interaction.

Experimental

All the reagents and solvents were used as obtained without further purification. EGTB was prepared according to literature procedure (Hendriks *et al.*, 1982). The title organic salt was obtained by crystallizing EGTB with $NaClO_4$ (molar ratio: 1/4). The mixture was stirred for half an hour at room temperature. The resulting pale-yellow solution was kept in air for one week. Crystals of (I) suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation of the solution at the bottom of the vessel.

Refinement

In the refinement the systematic absences permitted $P2_1$ and $P2_1/m$ as possible space groups: in view of the unit-cell volumes, $P2_1$ was selected and this choice was confirmed by the successful structure analysis. Careful search using PLATOM (Spek, 2003) for possible additional symmetry revealed none. The non-centrosymmetric space group may be caused by a slight disorder of the perchlorate anions (indicated by the larger than normal anisotropic displacement parameters of the O atoms of these anions. Owing to the poor quality of the crystal selected for diffraction with, only about 54% observed intensities were above the threshold of $2\sigma(I)$ within the 0–25.1° range. We attempted to select better crystals for diffraction, but failed every time. Conventional least squares refinement of the structural model against the defined data sets within the 0–25.1° range converged only at $R_1 = 0.079$, wR= 0.188 and Goof = 0.878. The title compound is racemic in solution but spontaneously racemized upon crystallization. The absolute configuration of the molecules in the crystal selected was readily determined and the configuration has no chemical significance.

All the H atoms bonded to carbon atoms were located at their geometrical positions with C–H = 0.97 Å(methylene) and 0.93 Å(aromatic), $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms bonded to imine N and water O atoms were located on the difference Fourier maps and then restrained at their more suitable positions to form decent hydrogen bonds, and the $U_{iso}(H)$ values were set 1.2 times of $U_{eq}(N)$ or 1.5 times of $U_{eq}(O)$ of their carrier atoms.

Figures

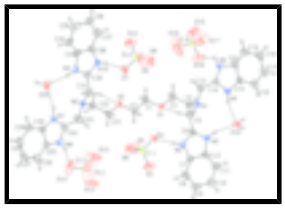


Fig. 1. Molecular structure of showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

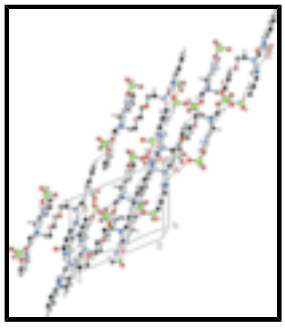


Fig. 2. Part of the crystal structure, showing the formation of the two-dimensional network along the [101] direction. Hydrogen bonding are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif have been omitted.

2,2',2'',2'''-(3,6-Dioxaoctane-1,8-diyldinitrilo)tetrabenzimidazolium tetrakis(perchlorate) dihydrate

Crystal data

$C_{38}H_{44}N_{10}O_2^{4+}\cdot 4ClO_4^-\cdot 2H_2O$	F_{000} = 1148
M_r = 1106.66	D_x = 1.512 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2334 reflections
a = 10.6093 (7) Å	θ = 2.4–18.9º
b = 17.6593 (12) Å	μ = 0.33 mm^{-1}
c = 14.0164 (8) Å	T = 292 (2) K
β = 112.238 (1)º	Plate, pale yellow
V = 2430.7 (3) Å^3	0.20 × 0.10 × 0.04 mm
Z = 2	

Data collection

Bruker SMART Apex CCD area-detector diffractometer	8332 independent reflections
Radiation source: fine focus sealed Siemens Mo tube	4522 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.117
T = 292(2) K	θ_{max} = 25.1º
0.3° wide ω exposures scans	θ_{min} = 2.0º
Absorption correction: multi-scan SADABS, (Sheldrick,1997)	h = −12→12
T_{min} = 0.925, T_{max} = 0.987	k = −21→20
23871 measured reflections	l = −16→16

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.078	$w = 1/[\sigma^2(F_o^2) + (0.1264P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.225	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.88	$\Delta\rho_{max}$ = 0.47 e Å^{-3}
8332 reflections	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
655 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983); 3891 Friedels
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.00 (1)
Secondary atom site location: constr	

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8375 (10)	0.5386 (5)	0.2636 (8)	0.068 (3)
H1A	0.8500	0.5827	0.2270	0.081*
H1B	0.9153	0.5344	0.3282	0.081*
C2	0.7120 (11)	0.5461 (5)	0.2834 (9)	0.084 (4)
H2A	0.6336	0.5460	0.2189	0.101*
H2B	0.7033	0.5040	0.3249	0.101*
C3	0.9468 (9)	0.4579 (6)	0.1822 (7)	0.074 (3)
H3A	1.0238	0.4482	0.2459	0.089*
H3B	0.9680	0.5012	0.1484	0.089*
C4	0.9174 (8)	0.3879 (5)	0.1115 (6)	0.049 (2)
H4A	1.0031	0.3696	0.1104	0.058*
H4B	0.8807	0.3484	0.1415	0.058*
C5	0.6814 (8)	0.4116 (5)	−0.0072 (6)	0.053 (2)
H5A	0.6771	0.4497	0.0413	0.063*
H5B	0.6305	0.4302	−0.0762	0.063*
C6	0.6202 (7)	0.3423 (5)	0.0094 (6)	0.0444 (19)
C7	0.4869 (8)	0.2607 (5)	0.0463 (6)	0.051 (2)
C8	0.3923 (9)	0.2222 (7)	0.0761 (7)	0.072 (3)
H8	0.3365	0.2482	0.1025	0.087*
C9	0.3845 (12)	0.1475 (7)	0.0656 (9)	0.088 (3)
H9	0.3205	0.1218	0.0838	0.106*
C10	0.4687 (13)	0.1054 (7)	0.0282 (10)	0.103 (4)
H10	0.4630	0.0529	0.0258	0.124*
C11	0.5610 (11)	0.1423 (6)	−0.0053 (9)	0.087 (3)
H11	0.6148	0.1165	−0.0336	0.104*
C12	0.5662 (8)	0.2200 (5)	0.0069 (6)	0.051 (2)
C13	0.8745 (9)	0.4541 (4)	−0.0490 (7)	0.054 (2)
H13A	0.9179	0.4959	−0.0034	0.065*
H13B	0.7986	0.4743	−0.1070	0.065*
C14	0.9730 (8)	0.4188 (5)	−0.0863 (6)	0.052 (2)
C15	1.0851 (8)	0.3332 (5)	−0.1394 (6)	0.052 (2)
C16	1.1323 (9)	0.2655 (6)	−0.1684 (6)	0.063 (2)
H16	1.0947	0.2186	−0.1647	0.076*

C17	1.2346 (10)	0.2732 (7)	−0.2015 (7)	0.075 (3)
H17	1.2688	0.2302	−0.2217	0.090*
C18	1.2916 (9)	0.3435 (8)	−0.2068 (7)	0.076 (3)
H18	1.3636	0.3462	−0.2292	0.091*
C19	1.2434 (10)	0.4084 (6)	−0.1797 (7)	0.072 (3)
H19	1.2789	0.4556	−0.1855	0.086*
C20	1.1401 (9)	0.4011 (5)	−0.1433 (6)	0.054 (2)
C21	0.5964 (8)	0.6323 (6)	0.3502 (7)	0.067 (3)
H21A	0.5743	0.5913	0.3872	0.080*
H21B	0.5226	0.6376	0.2835	0.080*
C22	0.6151 (8)	0.7050 (5)	0.4104 (6)	0.049 (2)
H22A	0.6441	0.7427	0.3749	0.059*
H22B	0.5275	0.7185	0.4105	0.059*
C23	0.6805 (9)	0.6522 (6)	0.5864 (7)	0.065 (3)
H23A	0.7610	0.6439	0.6482	0.078*
H23B	0.6528	0.6040	0.5514	0.078*
C24	0.5673 (8)	0.6842 (6)	0.6146 (6)	0.060 (3)
C25	0.3984 (9)	0.6921 (5)	0.6691 (7)	0.057 (2)
C26	0.2955 (11)	0.6777 (8)	0.7078 (8)	0.084 (3)
H26	0.2759	0.6292	0.7240	0.101*
C27	0.2268 (10)	0.7403 (10)	0.7196 (7)	0.087 (4)
H27	0.1564	0.7335	0.7430	0.104*
C28	0.2569 (10)	0.8130 (7)	0.6985 (7)	0.073 (3)
H28	0.2085	0.8540	0.7089	0.087*
C29	0.3612 (10)	0.8249 (6)	0.6611 (7)	0.068 (3)
H29	0.3808	0.8734	0.6446	0.082*
C30	0.4348 (8)	0.7624 (5)	0.6492 (6)	0.049 (2)
C31	0.8552 (8)	0.7013 (5)	0.5346 (6)	0.053 (2)
H31A	0.8696	0.6588	0.4960	0.064*
H31B	0.9093	0.6930	0.6070	0.064*
C32	0.9000 (8)	0.7742 (5)	0.4989 (6)	0.0464 (19)
C33	0.9223 (8)	0.8947 (5)	0.4705 (6)	0.054 (2)
C34	0.9053 (10)	0.9726 (6)	0.4576 (8)	0.082 (3)
H34	0.8423	0.9994	0.4757	0.098*
C35	0.9884 (15)	1.0067 (7)	0.4162 (10)	0.105 (4)
H35	0.9798	1.0587	0.4053	0.126*
C36	1.0852 (12)	0.9688 (8)	0.3891 (9)	0.091 (3)
H36	1.1408	0.9957	0.3634	0.109*
C37	1.0986 (9)	0.8904 (7)	0.4008 (7)	0.077 (3)
H37	1.1620	0.8634	0.3833	0.092*
C38	1.0100 (8)	0.8547 (5)	0.4406 (6)	0.052 (2)
N1	0.8241 (6)	0.3996 (4)	0.0059 (5)	0.0447 (16)
N2	0.5245 (6)	0.3361 (4)	0.0467 (5)	0.0481 (16)
H2C	0.4900	0.3733	0.0682	0.058*
N3	0.6475 (6)	0.2724 (4)	−0.0183 (5)	0.0490 (17)
H3C	0.7048	0.2621	−0.0465	0.059*
N4	0.9816 (7)	0.3442 (4)	−0.1022 (5)	0.0531 (18)
H4C	0.9321	0.3093	−0.0914	0.064*
N5	1.0680 (7)	0.4538 (4)	−0.1112 (5)	0.064 (2)

H5C	1.0822	0.5019	−0.1077	0.077*
N6	0.7107 (6)	0.7059 (3)	0.5189 (5)	0.0434 (16)
N7	0.5389 (7)	0.7554 (4)	0.6168 (5)	0.0516 (17)
H7A	0.5797	0.7922	0.6003	0.062*
N8	0.4872 (7)	0.6437 (4)	0.6483 (5)	0.066 (2)
H8A	0.4901	0.5953	0.6559	0.079*
N9	0.8522 (7)	0.8416 (5)	0.5056 (6)	0.063 (2)
H9A	0.7887	0.8515	0.5278	0.075*
N10	0.9971 (6)	0.7786 (4)	0.4604 (5)	0.0474 (16)
H10A	1.0429	0.7416	0.4499	0.057*
O1	0.8256 (5)	0.4728 (3)	0.2036 (4)	0.0609 (16)
O2	0.7188 (6)	0.6166 (3)	0.3374 (5)	0.0661 (18)
O19	0.7934 (8)	0.2223 (4)	−0.1313 (6)	0.100 (3)
H19A	0.768 (6)	0.238 (5)	−0.2037 (9)	0.150*
H19B	0.830 (2)	0.1797 (8)	−0.125 (3)	0.150*
O20	0.6623 (8)	0.8939 (4)	0.5927 (6)	0.088 (2)
H20A	0.6179	0.9113	0.5288	0.132*
H20B	0.6727	0.9261	0.6413	0.132*
Cl1	0.3179 (2)	0.46640 (13)	0.14581 (17)	0.0578 (6)
O3	0.2512 (8)	0.3941 (5)	0.1316 (7)	0.114 (3)
O4	0.4116 (7)	0.4666 (4)	0.0956 (5)	0.081 (2)
O5	0.2205 (8)	0.5251 (5)	0.1046 (6)	0.105 (3)
O6	0.3892 (7)	0.4803 (5)	0.2531 (5)	0.097 (2)
Cl3	0.5559 (3)	0.43523 (16)	0.6731 (2)	0.0792 (8)
O11	0.6518 (17)	0.3873 (9)	0.7353 (9)	0.259 (9)
O12	0.4397 (12)	0.3910 (8)	0.6297 (9)	0.195 (6)
O13	0.5693 (12)	0.4723 (7)	0.5901 (9)	0.180 (5)
O14	0.5236 (12)	0.4885 (6)	0.7338 (9)	0.175 (5)
Cl4	0.9707 (3)	0.65978 (16)	−0.1340 (2)	0.0781 (8)
O15	0.9452 (13)	0.7175 (5)	−0.2014 (9)	0.161 (4)
O16	0.9602 (15)	0.6803 (14)	−0.0491 (10)	0.294 (12)
O17	1.0903 (10)	0.6235 (8)	−0.1090 (14)	0.214 (7)
O18	0.8586 (9)	0.6074 (5)	−0.1794 (8)	0.135 (3)
Cl2	1.2108 (2)	0.64119 (14)	0.38154 (17)	0.0629 (6)
O7	1.1187 (8)	0.6449 (4)	0.4331 (5)	0.090 (2)
O8	1.3126 (9)	0.5877 (6)	0.4296 (7)	0.132 (4)
O9	1.1380 (8)	0.6221 (6)	0.2768 (5)	0.112 (3)
O10	1.2701 (9)	0.7118 (6)	0.3893 (8)	0.134 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.074 (6)	0.057 (6)	0.084 (7)	−0.009 (5)	0.045 (6)	−0.033 (5)
C2	0.108 (8)	0.057 (6)	0.125 (9)	−0.049 (6)	0.086 (8)	−0.055 (6)
C3	0.059 (5)	0.089 (8)	0.070 (6)	0.012 (5)	0.019 (5)	−0.036 (6)
C4	0.049 (5)	0.037 (4)	0.063 (5)	0.005 (4)	0.025 (4)	−0.003 (4)
C5	0.055 (5)	0.058 (6)	0.049 (5)	0.016 (4)	0.024 (4)	0.007 (4)
C6	0.032 (4)	0.061 (5)	0.046 (4)	0.005 (4)	0.022 (4)	−0.004 (4)

C7	0.060 (5)	0.044 (5)	0.050 (5)	−0.008 (4)	0.022 (4)	−0.002 (4)
C8	0.056 (5)	0.108 (9)	0.063 (6)	−0.004 (6)	0.033 (5)	−0.012 (6)
C9	0.102 (8)	0.062 (7)	0.112 (8)	−0.027 (7)	0.054 (7)	0.006 (7)
C10	0.119 (10)	0.077 (8)	0.147 (12)	−0.038 (7)	0.089 (10)	−0.041 (8)
C11	0.100 (8)	0.054 (6)	0.137 (9)	−0.019 (6)	0.079 (7)	−0.025 (6)
C12	0.045 (5)	0.047 (5)	0.070 (5)	−0.006 (4)	0.031 (4)	0.000 (4)
C13	0.078 (6)	0.029 (4)	0.069 (5)	0.000 (4)	0.042 (5)	0.000 (4)
C14	0.060 (5)	0.056 (6)	0.049 (5)	−0.004 (4)	0.030 (4)	−0.001 (4)
C15	0.044 (5)	0.063 (6)	0.056 (5)	−0.005 (4)	0.028 (4)	−0.008 (4)
C16	0.061 (6)	0.076 (7)	0.061 (5)	0.011 (5)	0.033 (5)	0.002 (5)
C17	0.067 (6)	0.099 (9)	0.062 (6)	0.016 (6)	0.028 (5)	−0.007 (6)
C18	0.048 (5)	0.119 (10)	0.065 (6)	0.006 (6)	0.029 (5)	−0.007 (6)
C19	0.069 (6)	0.085 (8)	0.070 (6)	−0.028 (6)	0.036 (5)	−0.002 (5)
C20	0.060 (5)	0.063 (6)	0.046 (5)	0.012 (5)	0.029 (4)	0.008 (4)
C21	0.037 (4)	0.096 (8)	0.079 (6)	−0.012 (5)	0.036 (4)	−0.016 (6)
C22	0.048 (5)	0.048 (5)	0.051 (5)	0.009 (4)	0.020 (4)	−0.009 (4)
C23	0.055 (5)	0.080 (7)	0.066 (6)	0.016 (5)	0.030 (5)	0.017 (5)
C24	0.052 (5)	0.088 (8)	0.048 (5)	0.003 (5)	0.028 (4)	0.009 (5)
C25	0.062 (6)	0.060 (6)	0.061 (5)	−0.008 (5)	0.035 (5)	−0.013 (4)
C26	0.078 (7)	0.117 (10)	0.070 (7)	−0.024 (7)	0.042 (6)	−0.005 (6)
C27	0.053 (6)	0.161 (13)	0.057 (6)	−0.022 (7)	0.033 (5)	−0.019 (7)
C28	0.061 (6)	0.108 (9)	0.056 (6)	−0.002 (6)	0.029 (5)	−0.016 (6)
C29	0.072 (6)	0.065 (7)	0.074 (6)	−0.003 (5)	0.034 (5)	−0.009 (5)
C30	0.052 (5)	0.054 (6)	0.043 (4)	−0.007 (4)	0.021 (4)	−0.009 (4)
C31	0.043 (5)	0.053 (6)	0.053 (5)	0.017 (4)	0.007 (4)	−0.004 (4)
C32	0.044 (5)	0.048 (5)	0.037 (4)	0.006 (4)	0.004 (4)	0.003 (4)
C33	0.046 (5)	0.046 (6)	0.061 (5)	−0.009 (4)	0.011 (4)	−0.019 (4)
C34	0.069 (6)	0.071 (8)	0.105 (8)	−0.038 (6)	0.032 (6)	−0.040 (6)
C35	0.117 (10)	0.064 (8)	0.124 (10)	−0.034 (8)	0.033 (9)	−0.016 (7)
C36	0.081 (8)	0.079 (9)	0.116 (9)	−0.046 (7)	0.040 (7)	−0.029 (7)
C37	0.045 (5)	0.113 (10)	0.071 (6)	−0.027 (6)	0.020 (5)	−0.027 (6)
C38	0.038 (4)	0.065 (6)	0.053 (5)	−0.014 (4)	0.019 (4)	−0.015 (4)
N1	0.045 (4)	0.048 (4)	0.046 (4)	−0.004 (3)	0.022 (3)	−0.009 (3)
N2	0.042 (4)	0.052 (4)	0.049 (4)	0.011 (3)	0.016 (3)	0.001 (3)
N3	0.052 (4)	0.040 (4)	0.070 (4)	0.001 (3)	0.039 (4)	−0.002 (3)
N4	0.059 (4)	0.049 (5)	0.060 (4)	−0.005 (4)	0.032 (4)	0.010 (3)
N5	0.079 (5)	0.058 (5)	0.069 (5)	−0.019 (4)	0.043 (4)	0.001 (4)
N6	0.044 (4)	0.040 (4)	0.054 (4)	0.007 (3)	0.029 (3)	0.005 (3)
N7	0.063 (4)	0.042 (4)	0.066 (4)	0.003 (4)	0.043 (4)	−0.007 (3)
N8	0.081 (5)	0.047 (4)	0.075 (5)	−0.006 (4)	0.035 (4)	0.009 (4)
N9	0.061 (4)	0.062 (5)	0.071 (5)	−0.009 (4)	0.032 (4)	−0.034 (4)
N10	0.041 (4)	0.047 (4)	0.057 (4)	0.003 (3)	0.021 (3)	−0.012 (3)
O1	0.062 (3)	0.057 (4)	0.081 (4)	−0.018 (3)	0.048 (3)	−0.032 (3)
O2	0.071 (4)	0.062 (4)	0.079 (4)	−0.018 (3)	0.044 (3)	−0.036 (3)
O19	0.125 (6)	0.077 (5)	0.146 (7)	−0.017 (4)	0.103 (6)	−0.019 (5)
O20	0.128 (6)	0.059 (4)	0.101 (5)	0.014 (4)	0.070 (5)	−0.010 (4)
Cl1	0.0509 (11)	0.0680 (15)	0.0636 (14)	−0.0052 (12)	0.0321 (11)	−0.0039 (12)
O3	0.098 (6)	0.095 (6)	0.149 (8)	−0.040 (5)	0.049 (5)	−0.005 (5)
O4	0.100 (5)	0.063 (4)	0.111 (5)	−0.011 (4)	0.077 (4)	−0.014 (4)

O5	0.102 (6)	0.096 (6)	0.113 (6)	0.029 (5)	0.037 (5)	−0.013 (5)
O6	0.100 (5)	0.123 (6)	0.066 (4)	−0.018 (5)	0.029 (4)	−0.016 (4)
Cl3	0.0831 (18)	0.0698 (17)	0.0872 (18)	−0.0061 (15)	0.0349 (15)	0.0067 (15)
O11	0.304 (17)	0.240 (15)	0.138 (10)	0.206 (14)	−0.023 (10)	−0.004 (10)
O12	0.175 (10)	0.255 (14)	0.174 (10)	−0.145 (10)	0.087 (8)	−0.010 (10)
O13	0.172 (10)	0.213 (12)	0.169 (10)	−0.068 (9)	0.081 (8)	0.066 (9)
O14	0.219 (12)	0.120 (8)	0.164 (9)	0.052 (8)	0.047 (9)	−0.060 (7)
Cl4	0.0834 (18)	0.0796 (19)	0.0801 (17)	−0.0092 (15)	0.0411 (15)	0.0030 (15)
O15	0.239 (13)	0.061 (6)	0.171 (10)	−0.026 (7)	0.065 (9)	0.006 (6)
O16	0.221 (14)	0.54 (4)	0.143 (11)	−0.006 (19)	0.093 (11)	−0.115 (16)
O17	0.085 (7)	0.169 (11)	0.37 (2)	0.028 (7)	0.071 (9)	0.010 (12)
O18	0.125 (7)	0.129 (7)	0.143 (8)	−0.052 (6)	0.041 (6)	0.018 (6)
Cl2	0.0575 (13)	0.0731 (17)	0.0657 (14)	−0.0020 (13)	0.0317 (12)	−0.0057 (12)
O7	0.106 (5)	0.086 (5)	0.110 (5)	0.018 (5)	0.078 (5)	−0.003 (5)
O8	0.099 (6)	0.189 (10)	0.112 (6)	0.085 (7)	0.045 (5)	0.025 (6)
O9	0.100 (5)	0.172 (9)	0.056 (4)	−0.001 (6)	0.021 (4)	−0.019 (5)
O10	0.114 (7)	0.125 (8)	0.163 (9)	−0.060 (6)	0.052 (6)	−0.019 (7)

Geometric parameters (Å, °)

C1—O1	1.413 (10)	C24—N7	1.295 (11)
C1—C2	1.464 (11)	C24—N8	1.327 (11)
C1—H1A	0.9700	C25—C30	1.359 (12)
C1—H1B	0.9700	C25—N8	1.383 (11)
C2—O2	1.445 (10)	C25—C26	1.413 (13)
C2—H2A	0.9700	C26—C27	1.368 (16)
C2—H2B	0.9700	C26—H26	0.9300
C3—O1	1.450 (10)	C27—C28	1.383 (16)
C3—C4	1.541 (11)	C27—H27	0.9300
C3—H3A	0.9700	C28—C29	1.408 (13)
C3—H3B	0.9700	C28—H28	0.9300
C4—N1	1.450 (10)	C29—C30	1.399 (13)
C4—H4A	0.9700	C29—H29	0.9300
C4—H4B	0.9700	C30—N7	1.349 (10)
C5—C6	1.445 (11)	C31—N6	1.466 (9)
C5—N1	1.469 (10)	C31—C32	1.520 (12)
C5—H5A	0.9700	C31—H31A	0.9700
C5—H5B	0.9700	C31—H31B	0.9700
C6—N2	1.310 (9)	C32—N9	1.312 (11)
C6—N3	1.358 (10)	C32—N10	1.335 (10)
C7—C12	1.372 (11)	C33—C38	1.354 (11)
C7—C8	1.400 (12)	C33—C34	1.390 (14)
C7—N2	1.389 (10)	C33—N9	1.397 (11)
C8—C9	1.326 (15)	C34—C35	1.365 (15)
C8—H8	0.9300	C34—H34	0.9300
C9—C10	1.407 (15)	C35—C36	1.393 (16)
C9—H9	0.9300	C35—H35	0.9300
C10—C11	1.396 (14)	C36—C37	1.394 (16)
C10—H10	0.9300	C36—H36	0.9300

C11—C12	1.381 (13)	C37—C38	1.411 (12)
C11—H11	0.9300	C37—H37	0.9300
C12—N3	1.398 (10)	C38—N10	1.389 (11)
C13—N1	1.454 (9)	N2—H2C	0.8600
C13—C14	1.473 (11)	N3—H3C	0.8600
C13—H13A	0.9700	N4—H4C	0.8600
C13—H13B	0.9700	N5—H5C	0.8600
C14—N5	1.337 (10)	N7—H7A	0.8600
C14—N4	1.344 (11)	N8—H8A	0.8600
C15—C20	1.342 (12)	N9—H9A	0.8600
C15—N4	1.395 (10)	N10—H10A	0.8600
C15—C16	1.415 (12)	O19—H19A	0.9900
C16—C17	1.339 (13)	O19—H19B	0.837
C16—H16	0.9300	O20—H20A	0.8943
C17—C18	1.395 (16)	O20—H20B	0.8618
C17—H17	0.9300	Cl1—O4	1.420 (6)
C18—C19	1.367 (14)	Cl1—O5	1.423 (8)
C18—H18	0.9300	Cl1—O6	1.426 (7)
C19—C20	1.380 (12)	Cl1—O3	1.437 (8)
C19—H19	0.9300	Cl3—O11	1.357 (10)
C20—N5	1.384 (10)	Cl3—O13	1.389 (9)
C21—O2	1.404 (9)	Cl3—O12	1.390 (10)
C21—C22	1.508 (12)	Cl3—O14	1.396 (9)
C21—H21A	0.9700	Cl4—O16	1.288 (12)
C21—H21B	0.9700	Cl4—O17	1.344 (10)
C22—H22A	0.9700	Cl4—O15	1.346 (10)
C22—H22B	0.9700	Cl4—O18	1.449 (8)
C22—N6	1.475 (10)	Cl2—O10	1.382 (10)
C23—N6	1.458 (10)	Cl2—O8	1.400 (8)
C23—C24	1.509 (12)	Cl2—O9	1.416 (7)
C23—H23A	0.9700	Cl2—O7	1.420 (7)
C23—H23B	0.9700		
O1—C1—C2	107.7 (7)	C25—C26—H26	122.4
O1—C1—H1A	110.3	C26—C27—C28	123.2 (9)
C2—C1—H1A	110.2	C26—C27—H27	118.4
O1—C1—H1B	110.1	C28—C27—H27	118.4
C2—C1—H1B	110.1	C27—C28—C29	119.8 (10)
H1A—C1—H1B	108.5	C27—C28—H28	120.1
O2—C2—C1	108.0 (7)	C29—C28—H28	120.1
O2—C2—H2A	110.1	C30—C29—C28	118.6 (10)
C1—C2—H2A	110.0	C30—C29—H29	120.7
O2—C2—H2B	110.1	C28—C29—H29	120.7
C1—C2—H2B	110.2	N7—C30—C25	108.6 (8)
H2A—C2—H2B	108.4	N7—C30—C29	132.6 (9)
O1—C3—C4	107.4 (7)	C25—C30—C29	118.8 (8)
O1—C3—H3A	110.2	N6—C31—C32	110.1 (6)
C4—C3—H3A	110.2	N6—C31—H31A	109.6
O1—C3—H3B	110.3	C32—C31—H31A	109.6
C4—C3—H3B	110.2	N6—C31—H31B	109.7

H3A—C3—H3B	108.5	C32—C31—H31B	109.7
N1—C4—C3	115.8 (7)	H31A—C31—H31B	108.1
N1—C4—H4A	108.3	N9—C32—N10	110.7 (8)
C3—C4—H4A	108.4	N9—C32—C31	124.6 (8)
N1—C4—H4B	108.3	N10—C32—C31	124.6 (7)
C3—C4—H4B	108.3	C38—C33—C34	123.1 (10)
H4A—C4—H4B	107.4	C38—C33—N9	106.2 (8)
C6—C5—N1	111.3 (6)	C34—C33—N9	130.5 (9)
C6—C5—H5A	109.4	C35—C34—C33	114.8 (11)
N1—C5—H5A	109.3	C35—C34—H34	122.5
C6—C5—H5B	109.4	C33—C34—H34	122.7
N1—C5—H5B	109.4	C34—C35—C36	124.4 (12)
H5A—C5—H5B	108.0	C34—C35—H35	117.9
N2—C6—N3	108.6 (7)	C36—C35—H35	117.7
N2—C6—C5	126.7 (7)	C37—C36—C35	119.9 (11)
N3—C6—C5	124.5 (6)	C37—C36—H36	120.0
C12—C7—C8	118.9 (8)	C35—C36—H36	120.1
C12—C7—N2	106.5 (7)	C36—C37—C38	115.9 (10)
C8—C7—N2	134.6 (9)	C36—C37—H37	122.1
C9—C8—C7	118.1 (10)	C38—C37—H37	122.0
C9—C8—H8	120.9	C33—C38—N10	108.2 (8)
C7—C8—H8	121.0	C33—C38—C37	121.8 (10)
C8—C9—C10	123.0 (10)	N10—C38—C37	129.9 (9)
C8—C9—H9	118.5	C13—N1—C4	112.8 (6)
C10—C9—H9	118.5	C13—N1—C5	114.0 (6)
C11—C10—C9	120.2 (11)	C4—N1—C5	114.9 (6)
C11—C10—H10	119.9	C6—N2—C7	110.1 (7)
C9—C10—H10	119.9	C6—N2—H2C	124.9
C12—C11—C10	115.0 (9)	C7—N2—H2C	125.0
C12—C11—H11	122.5	C6—N3—C12	108.1 (6)
C10—C11—H11	122.5	C6—N3—H3C	125.9
C7—C12—C11	124.6 (8)	C12—N3—H3C	126.0
C7—C12—N3	106.7 (7)	C14—N4—C15	108.1 (7)
C11—C12—N3	128.8 (8)	C14—N4—H4C	125.9
N1—C13—C14	111.1 (6)	C15—N4—H4C	126.0
N1—C13—H13A	109.4	C14—N5—C20	109.7 (7)
C14—C13—H13A	109.4	C14—N5—H5C	125.2
N1—C13—H13B	109.4	C20—N5—H5C	125.1
C14—C13—H13B	109.5	C31—N6—C23	109.9 (6)
H13A—C13—H13B	108.0	C31—N6—C22	115.2 (6)
N5—C14—N4	107.8 (7)	C23—N6—C22	115.3 (6)
N5—C14—C13	127.1 (8)	C24—N7—C30	108.9 (7)
N4—C14—C13	125.2 (7)	C24—N7—H7A	125.6
C20—C15—N4	107.9 (8)	C30—N7—H7A	125.5
C20—C15—C16	122.4 (8)	C24—N8—C25	108.7 (8)
N4—C15—C16	129.7 (9)	C24—N8—H8A	125.6
C17—C16—C15	115.8 (10)	C25—N8—H8A	125.7
C17—C16—H16	122.1	C32—N9—C33	108.2 (7)
C15—C16—H16	122.1	C32—N9—H9A	126.0

C16—C17—C18	122.2 (10)	C33—N9—H9A	125.8
C16—C17—H17	119.0	C32—N10—C38	106.7 (7)
C18—C17—H17	118.8	C32—N10—H10A	126.7
C19—C18—C17	121.1 (9)	C38—N10—H10A	126.6
C19—C18—H18	119.4	C1—O1—C3	112.7 (6)
C17—C18—H18	119.5	C21—O2—C2	111.8 (7)
C18—C19—C20	117.2 (9)	H19A—O19—H19B	107.0
C18—C19—H19	121.4	H20A—O20—H20B	115.2
C20—C19—H19	121.4	O4—Cl1—O5	109.5 (5)
C15—C20—N5	106.6 (7)	O4—Cl1—O6	109.4 (4)
C15—C20—C19	121.2 (9)	O5—Cl1—O6	108.5 (5)
N5—C20—C19	132.1 (9)	O4—Cl1—O3	109.5 (5)
O2—C21—C22	107.9 (7)	O5—Cl1—O3	110.3 (5)
O2—C21—H21A	110.2	O6—Cl1—O3	109.6 (5)
C22—C21—H21A	110.1	O11—Cl3—O13	122.8 (10)
O2—C21—H21B	110.1	O11—Cl3—O12	104.8 (11)
C22—C21—H21B	110.1	O13—Cl3—O12	104.6 (7)
H21A—C21—H21B	108.4	O11—Cl3—O14	109.1 (7)
N6—C22—C21	118.7 (7)	O13—Cl3—O14	108.7 (9)
N6—C23—C24	108.5 (8)	O12—Cl3—O14	105.3 (8)
N6—C23—H23A	109.9	O16—Cl4—O17	107.2 (11)
C24—C23—H23A	110.0	O16—Cl4—O15	111.8 (13)
N6—C23—H23B	110.0	O17—Cl4—O15	117.6 (9)
C24—C23—H23B	110.0	O16—Cl4—O18	104.0 (10)
H23A—C23—H23B	108.4	O17—Cl4—O18	110.3 (8)
N7—C24—N8	109.2 (8)	O15—Cl4—O18	105.2 (6)
N7—C24—C23	125.8 (9)	O10—Cl2—O8	109.0 (6)
N8—C24—C23	124.8 (10)	O10—Cl2—O9	110.5 (6)
C30—C25—N8	104.5 (7)	O8—Cl2—O9	110.9 (6)
C30—C25—C26	124.4 (9)	O10—Cl2—O7	107.4 (6)
N8—C25—C26	131.0 (10)	O8—Cl2—O7	109.9 (5)
C27—C26—C25	115.1 (11)	O9—Cl2—O7	109.2 (5)
C27—C26—H26	122.5		
O1—C1—C2—O2	−175.3 (8)	N9—C33—C38—C37	−179.8 (7)
O1—C3—C4—N1	72.2 (10)	C36—C37—C38—C33	−3.2 (13)
N1—C5—C6—N2	148.7 (7)	C36—C37—C38—N10	179.5 (9)
N1—C5—C6—N3	−35.9 (10)	C14—C13—N1—C4	80.6 (8)
C12—C7—C8—C9	0.6 (13)	C14—C13—N1—C5	−145.9 (7)
N2—C7—C8—C9	−179.2 (10)	C3—C4—N1—C13	62.4 (9)
C7—C8—C9—C10	1.4 (17)	C3—C4—N1—C5	−70.6 (9)
C8—C9—C10—C11	−3.5 (19)	C6—C5—N1—C13	155.6 (6)
C9—C10—C11—C12	3.3 (17)	C6—C5—N1—C4	−71.9 (8)
C8—C7—C12—C11	−0.6 (14)	N3—C6—N2—C7	1.0 (8)
N2—C7—C12—C11	179.3 (9)	C5—C6—N2—C7	177.0 (7)
C8—C7—C12—N3	178.5 (7)	C12—C7—N2—C6	0.4 (9)
N2—C7—C12—N3	−1.6 (9)	C8—C7—N2—C6	−179.8 (9)
C10—C11—C12—C7	−1.4 (16)	N2—C6—N3—C12	−2.0 (8)
C10—C11—C12—N3	179.7 (10)	C5—C6—N3—C12	−178.1 (7)
N1—C13—C14—N5	−158.3 (7)	C7—C12—N3—C6	2.2 (9)

N1—C13—C14—N4	22.2 (12)	C11—C12—N3—C6	−178.7 (10)
C20—C15—C16—C17	−0.9 (13)	N5—C14—N4—C15	−0.7 (9)
N4—C15—C16—C17	−180.0 (8)	C13—C14—N4—C15	178.8 (8)
C15—C16—C17—C18	0.2 (13)	C20—C15—N4—C14	1.5 (9)
C16—C17—C18—C19	−1.1 (15)	C16—C15—N4—C14	−179.4 (9)
C17—C18—C19—C20	2.4 (14)	N4—C14—N5—C20	−0.3 (9)
N4—C15—C20—N5	−1.6 (9)	C13—C14—N5—C20	−179.8 (8)
C16—C15—C20—N5	179.2 (8)	C15—C20—N5—C14	1.2 (9)
N4—C15—C20—C19	−178.4 (8)	C19—C20—N5—C14	177.5 (9)
C16—C15—C20—C19	2.4 (13)	C32—C31—N6—C23	−158.1 (7)
C18—C19—C20—C15	−3.1 (13)	C32—C31—N6—C22	69.5 (8)
C18—C19—C20—N5	−178.9 (9)	C24—C23—N6—C31	149.0 (7)
O2—C21—C22—N6	−67.4 (10)	C24—C23—N6—C22	−78.8 (9)
N6—C23—C24—N7	−25.9 (12)	C21—C22—N6—C31	71.0 (9)
N6—C23—C24—N8	159.2 (8)	C21—C22—N6—C23	−58.8 (9)
C30—C25—C26—C27	3.4 (14)	N8—C24—N7—C30	−2.1 (10)
N8—C25—C26—C27	179.6 (9)	C23—C24—N7—C30	−177.7 (7)
C25—C26—C27—C28	−1.8 (14)	C25—C30—N7—C24	0.7 (10)
C26—C27—C28—C29	1.2 (15)	C29—C30—N7—C24	−176.8 (9)
C27—C28—C29—C30	−1.9 (13)	N7—C24—N8—C25	2.6 (10)
N8—C25—C30—N7	0.8 (9)	C23—C24—N8—C25	178.3 (8)
C26—C25—C30—N7	177.9 (8)	C30—C25—N8—C24	−2.1 (9)
N8—C25—C30—C29	178.7 (7)	C26—C25—N8—C24	−178.8 (9)
C26—C25—C30—C29	−4.2 (13)	N10—C32—N9—C33	−0.6 (8)
C28—C29—C30—N7	−179.4 (8)	C31—C32—N9—C33	176.9 (7)
C28—C29—C30—C25	3.3 (12)	C38—C33—N9—C32	1.6 (9)
N6—C31—C32—N9	33.5 (10)	C34—C33—N9—C32	176.5 (9)
N6—C31—C32—N10	−149.4 (7)	N9—C32—N10—C38	−0.6 (8)
C38—C33—C34—C35	−2.7 (14)	C31—C32—N10—C38	−178.1 (7)
N9—C33—C34—C35	−176.8 (9)	C33—C38—N10—C32	1.6 (8)
C33—C34—C35—C36	−0.8 (16)	C37—C38—N10—C32	179.3 (8)
C34—C35—C36—C37	2.2 (18)	C2—C1—O1—C3	−177.9 (8)
C35—C36—C37—C38	−0.2 (15)	C4—C3—O1—C1	−176.7 (7)
C34—C33—C38—N10	−177.4 (8)	C22—C21—O2—C2	177.9 (8)
N9—C33—C38—N10	−1.9 (9)	C1—C2—O2—C21	173.9 (9)
C34—C33—C38—C37	4.8 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O19—H19B···O16^{i}	0.84	2.61	2.962 (16)	107 (3)
C4—H4A···O3^{ii}	0.97	2.57	3.446 (11)	150
C18—H18···O12^{iii}	0.93	2.53	3.339 (14)	146
C28—H28···O1^{iv}	0.93	2.52	3.394 (12)	156
O20—H20B···O6^{iv}	0.86	2.07	2.861 (9)	153
O20—H20A···O12^{iv}	0.89	2.10	2.887 (14)	146
O20—H20A···O13^{iv}	0.89	2.32	3.123 (14)	150

O19—H19B···O9^{i}	0.84	2.49	2.991 (11)	119 (3)
C23—H23A···O18^{v}	0.97	2.33	3.206 (13)	150
N2—H2C···O4	0.86	1.95	2.799 (9)	170
N8—H8A···O14	0.86	2.14	2.958 (14)	158
N3—H3C···O19	0.86	1.91	2.746 (9)	165
N4—H4C···O19	0.86	2.05	2.859 (10)	156
N5—H5C···O17	0.86	2.15	3.006 (15)	173
N10—H10A···O7	0.86	1.94	2.786 (9)	168
C5—H5A···O1	0.97	2.26	2.969 (10)	129
N7—H7A···O20	0.86	2.02	2.855 (10)	164
N9—H9A···O20	0.86	2.03	2.875 (10)	168
C31—H31A···O2	0.97	2.32	2.990 (10)	126

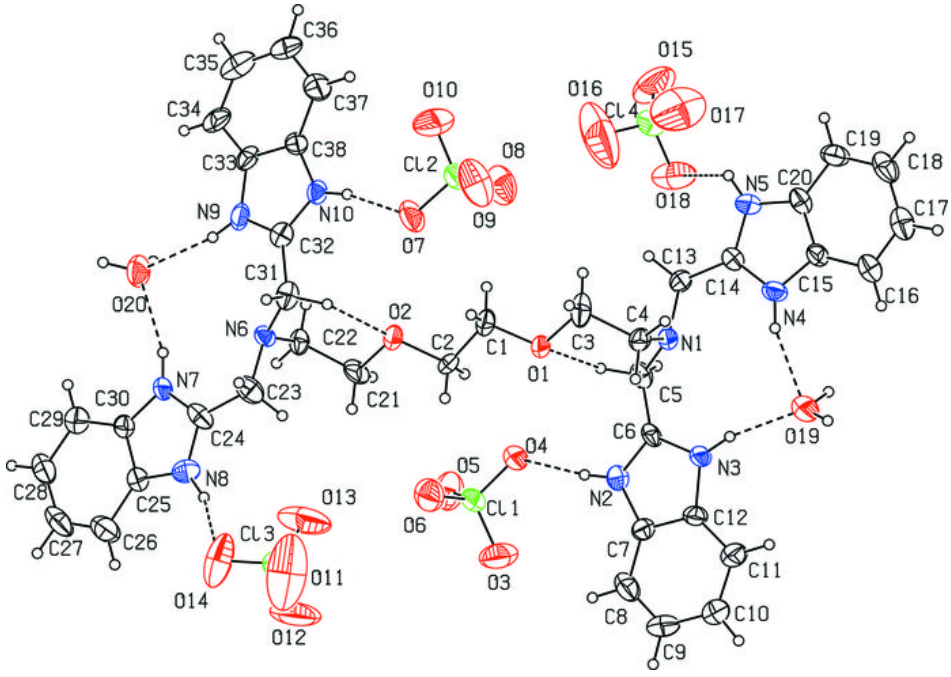
Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*; (ii) *x*+1, *y*, *z*; (iii) *x*+1, *y*, *z*−1; (iv) −*x*+1, *y*+1/2, −*z*+1; (v) *x*, *y*, *z*+1.

Table 2 π–π stacking interactions in structure of (I)

Cg_i	Cg_j	Dihedral angle(°)	Centroid distance (Å)	*ca* Interplanar spacing (Å)
*Cg*1	*Cg*4^{vi}	3.1 (1)	4.020 (5)	3.489 (5)
*Cg*1	*Cg*5^{vii}	3.8 (1)	3.941 (5)	3.472 (5)
*Cg*2	*Cg*4^{viii}	2.5 (1)	3.931 (5)	3.535 (5)
*Cg*3	*Cg*5^{vii}	2.9 (1)	3.867 (6)	3.467 (6)
*Cg*4	*Cg*6^{i}	2.3 (1)	3.767 (6)	3.510 (6)

*Cg*1 is the centroid defined by N2/N3/C6/C7/C12; *Cg*2 is the centroid defined by N9/N10/C32/C33/C38; *Cg*3 is the centroid defined by C7 to C12; *Cg*4 is the centroid defined by C15 to C20; *Cg*5 is the centroid defined by C25 to C30; *Cg*6 is the centroid defined by C33 to C38; symmetry code: (vi) *x* − 1, *y*, *z*; (vii) 1 − *x*, *y* − 1/2, 1 − *z*; (viii) 2 − *x*, 1/2 + *y*, −*z*.

Fig. 1


C36
C35
C34
C37
C33
C38
N9
N10
C32
C31
O20
N6
C22
O2
C2
C1
O1
C3
C4
N1
C5
C6
N3
C12
C7
C11
C8
C10
C9
C30
N7
C23
C21
C29
C24
C28
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C25
C27
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O12
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O10
O8
O9
O7
Cl1
O4
O5
O6
O3
N2
Cl4
O15
O16
O17
O18
N5
C20
C19
C18
C17
C16
C15
C14
C13
N4
O19

Fig. 2