organic compounds

3,10-*C-meso*-3,5,7,7,10,12,14,14-Octa-methyl-4,11-diaza-1,8-diazoniacyclo-tetradecane bis(perchlorate)

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The structure determination of the title salt, $C_{18}H_{42}N_4{}^{2+}$·$2ClO_4{}^-$, reveals that protonation has occurred at diagonally opposite amine N atoms. Intramolecular N—H···N hydrogen bonds stabilize the conformation of the dication. In the crystal, the dications are bridged by perchlorate ions *via* N—H···O hydrogen bonds into supramolecular chains propagating along the *c* axis and weak C—H···O interactions cross-link the chains.

Related literature

For background to macrocycles and for related structures, see: Benson *et al.* (2006); Roy *et al.* (2006, 2008); Hazari *et al.* (2008). For the synthesis, see: Curtis *et al.* (1969); Bembi *et al.* (1989).
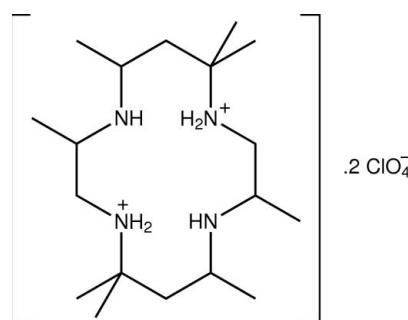
Experimental

Crystal data

$C_{18}H_{42}N_4{}^{2+}$·$2ClO_4{}^-$	$b = 16.297$ (3) Å
$M_r = 513.46$	$c = 17.754$ (5) Å
Monoclinic, $P2_1/c$	$\beta = 102.088$ (5)°
$a = 8.868$ (2) Å	$V = 2508.9$ (10) Å^3

‡ Additional correspondence author, e-mail: tapashir@yahoo.com.

$Z = 4$	$T = 98$ K
Mo $K\alpha$ radiation	$0.35 \times 0.10 \times 0.03$ mm
$\mu = 0.31$ mm^{-1}	

Data collection

Rigaku AFC12/SATURN724 diffractometer	54635 measured reflections
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	4387 independent reflections
$T_{min} = 0.695$, $T_{max} = 1$	4279 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.078$	2 restraints
$wR(F^2) = 0.240$	H-atom parameters constrained
$S = 1.19$	$\Delta\rho_{max} = 0.85$ e Å^{-3}
4387 reflections	$\Delta\rho_{min} = -0.73$ e Å^{-3}
295 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2n···N11	0.92	2.07	2.841 (4)	140
N8—H9n···N4	0.92	1.95	2.763 (5)	146
N1—H1n···O2	0.92	2.37	2.963 (4)	122
N1—H1n···O7^i	0.92	2.41	3.169 (4)	140
N1—H2n···N4	0.92	2.56	2.929 (4)	105
N4—H4n···O6	0.88	2.38	3.212 (4)	158
N8—H8n···O1	0.92	2.02	2.937 (5)	177
N11—H11n···O6	0.88	2.24	3.087 (5)	162
C2—H2a···O1^{ii}	0.99	2.57	3.109 (5)	114
C9—H9a···O5^{iii}	0.99	2.57	3.498 (6)	155
C12a—H12b···O8	0.98	2.56	3.487 (5)	158

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

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5587).

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supporting information

3,10-*C*-*meso*-3,5,7,7,10,12,14,14-Octamethyl-4,11-diaza-1,8-diazoniacyclotetradecane bis(perchlorate)

Tapashi G. Roy, Saroj K. S. Hazari, Kanak K. Barua, Provi Palit and Edward R. T. Tiekink

S1. Comment

The title salt (I) was characterized during studies of macrocyclic ligands of this type as well as their transition metal complexes (Benson *et al.*, 2006; Roy *et al.*, 2006; Hazari *et al.*, 2008; Roy *et al.*, 2008). The asymmetric unit of (I) comprises one di-protonated macrocyclic ligand and two perchlorate anions, Fig. 1. The observed conformation and pattern of protonation (*i.e.* at the diagonally opposite N1 and N8 atoms) in the di-cation conforms to expectation (Hazari *et al.*, 2008). The presence of N–H⋯N hydrogen bonds within the cavity is noted, Table 1. The remaining N–H groups form N–H⋯O hydrogen bonds to perchlorate-O atoms, Table 1. The perchlorate ions serve to bridge the cations to form a supramolecular chain along the *c* axis and connections between these, *i.e. via* C–H⋯O contacts, lead to the formation of a supramolecular layer in the *ac* plane. Layers stack along the *b* axis, Fig. 3.

S2. Experimental

The compound 3,10-*C*-*meso*-3,5,7,7,10,12,14,14—octamethyl-4,11-diaza-1,8-diazoniacyclotetradecadiene (Curtis *et al.*, 1969), on reduction with NaBH$_4$, yields an isomeric mixture of saturated macrocycles, the Me$_8$[14] anes, which have been resolved into three distinct isomers (Bembi *et al.*, 1989). During synthesis of Fe(III) complex of one of the isomers, L_C (Bembi *et al.*, 1989), this isomeric ligand on heating with slightly acidic methanolic solution of Fe$_2$(ClO$_4$)$_3$.6H$_2$O in the ratio of 1:2, followed by cooling and slow evaporation at room temperature for a few days produced yellow-orange prisms of (I).

S3. Refinement

All N– and C-bound H atoms were allowed to ride on their parent atoms at N–H and C–H distances of 0.88–0.92 Å and 0.92–1.00 Å, respectively, and with U_{iso}(H) values of 1.2–1.5U_{eq}(parent atom).
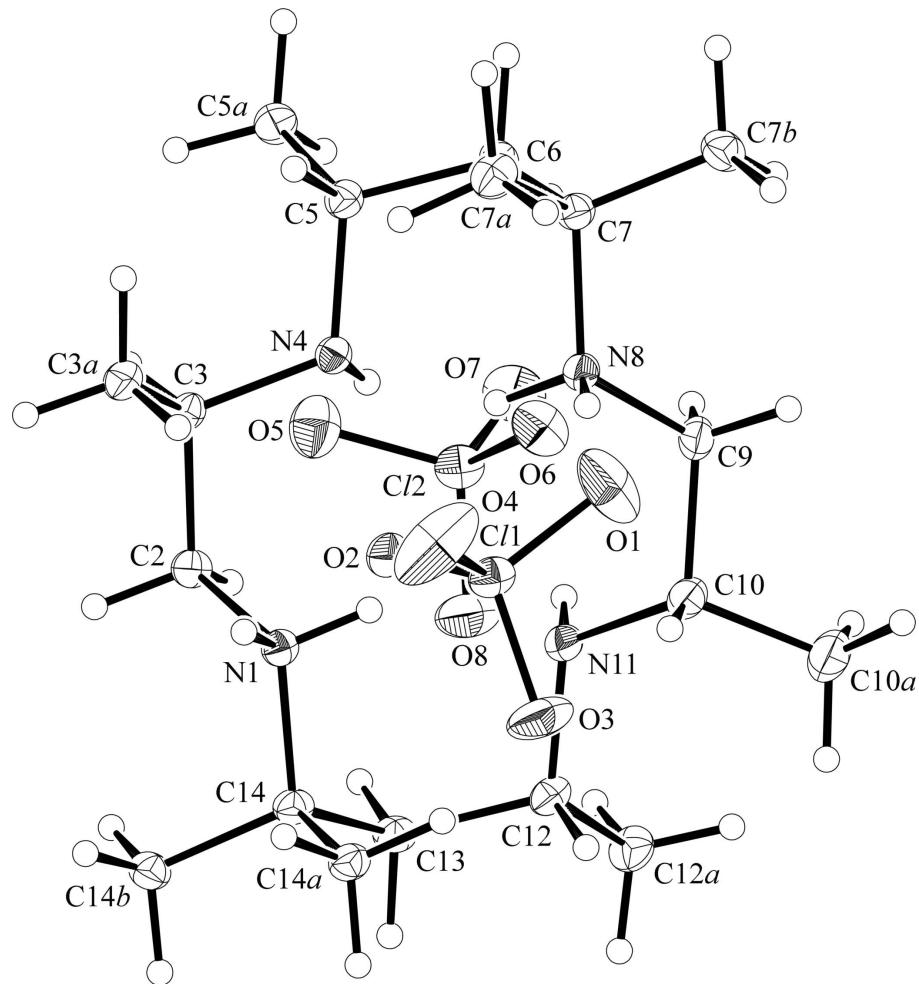
Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.
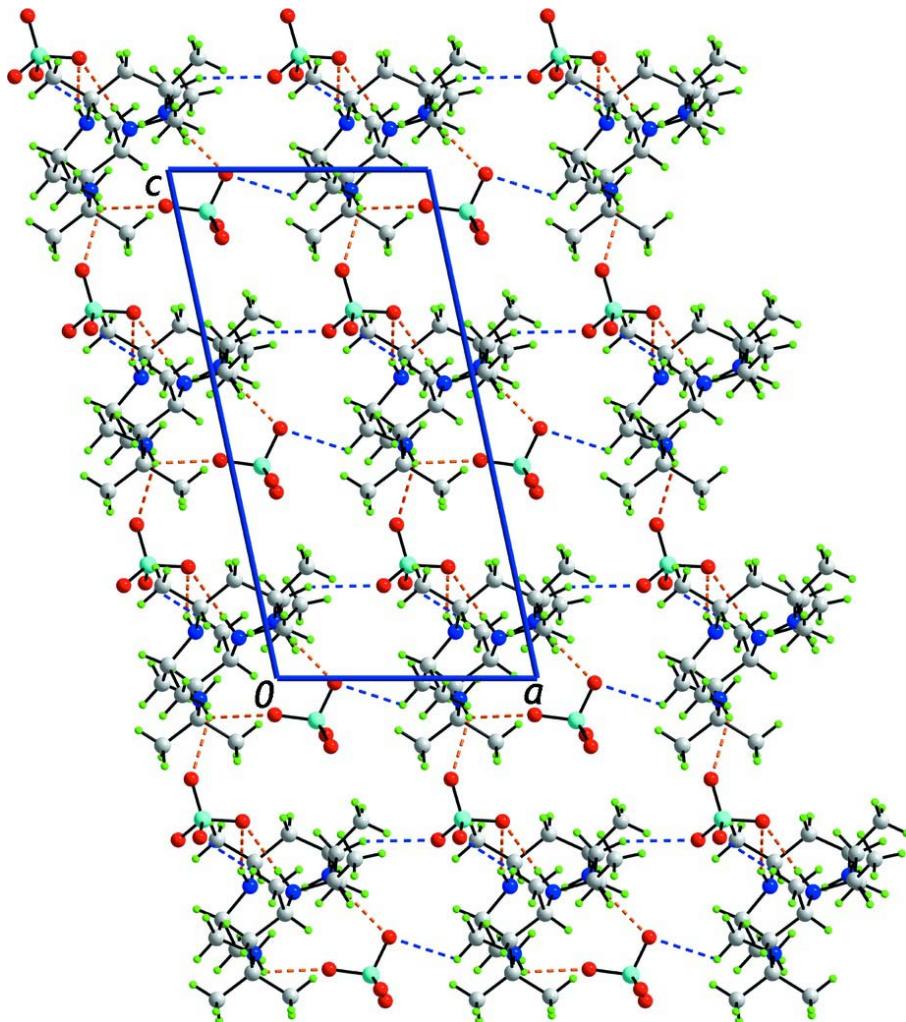
Figure 2

Supramolecular array in (I) showing N–H···O hydrogen bonds and C–H···O contacts as orange and blue dashed lines, respectively. Colour code: Cl (cyan), O (red), N (blue), C (grey) & H (green).
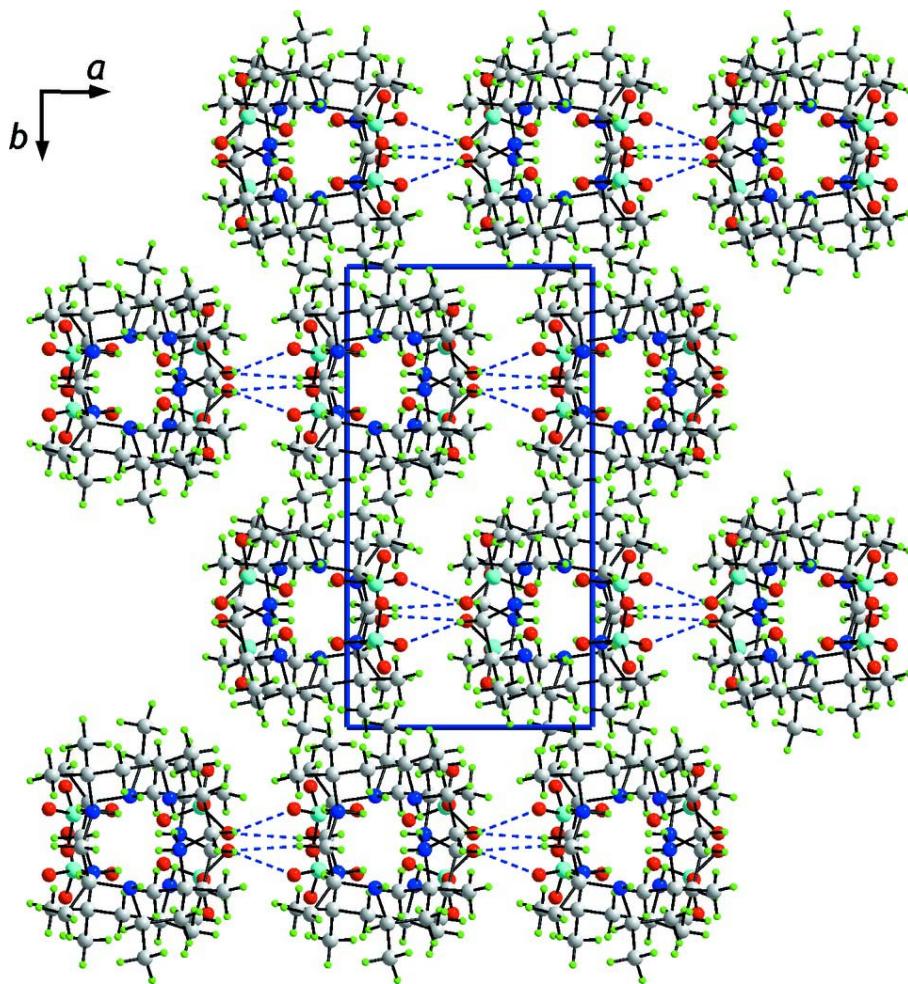
Figure 3

Stacking of layers along the *b* axis in (I). The C–H···O contacts are shown as blue dashed lines. Colour code: Cl (cyan), O (red), N (blue), C (grey) & H (green).

3,10-*C-meso*-3,5,7,7,10,12,14,14-Octamethyl-4,11-diaza-1,8- diazoniacyclotetradecane bis(perchlorate)

Crystal data
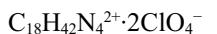
$C_{18}H_{42}N_4{}^{2+}·2ClO_4{}^-$
$M_r = 513.46$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.868\ (2)$ Å
$b = 16.297\ (3)$ Å
$c = 17.754\ (5)$ Å
$\beta = 102.088\ (5)°$
$V = 2508.9\ (10)$ Å^3
$Z = 4$

$F(000) = 1104$
$D_x = 1.359$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7813 reflections
$\theta = 2.4–30.4°$
$\mu = 0.31$ mm^{-1}
$T = 98$ K
Prism, yellow-orange
$0.35 \times 0.10 \times 0.03$ mm

Data collection

Rigaku AFC12K/SATURN724 diffractometer	54635 measured reflections
Radiation source: fine-focus sealed tube	4387 independent reflections
Graphite monochromator	4279 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.055$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.4°$
$T_{min} = 0.695$, $T_{max} = 1$	$h = -10 \rightarrow 10$
	$k = -19 \rightarrow 19$
	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.078$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.240$	
$S = 1.19$	H-atom parameters constrained
4387 reflections	$w = 1/[\sigma^2(F_o^2) + (0.128P)^2 + 4.0207P]$
295 parameters	where $P = (F_o^2 + 2F_c^2)/3$
2 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.85$ e Å^{-3}
	$\Delta\rho_{min} = -0.73$ e Å^{-3}

Special details

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

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	−0.11642 (10)	0.18918 (6)	0.58280 (6)	0.0337 (3)
Cl2	0.40297 (12)	0.32683 (6)	0.27709 (6)	0.0372 (3)
O1	−0.2173 (4)	0.1783 (4)	0.5090 (2)	0.0900 (17)
O2	0.0398 (3)	0.18064 (16)	0.57180 (16)	0.0351 (6)
O3	−0.1414 (4)	0.2682 (2)	0.6113 (2)	0.0641 (11)
O4	−0.1502 (6)	0.1294 (3)	0.6341 (3)	0.0955 (17)
O5	0.5140 (4)	0.2692 (2)	0.3177 (2)	0.0593 (9)
O6	0.2511 (4)	0.2976 (2)	0.28141 (18)	0.0506 (8)
O7	0.4141 (4)	0.3316 (2)	0.19821 (18)	0.0551 (9)
O8	0.4289 (4)	0.4058 (2)	0.3127 (2)	0.0608 (10)
N1	0.3158 (3)	0.26608 (17)	0.54123 (17)	0.0251 (6)
H1N	0.2939	0.2296	0.5770	0.030*
H2N	0.2306	0.2683	0.5015	0.030*
N4	0.2753 (3)	0.15534 (18)	0.40857 (16)	0.0253 (6)
H4N	0.2973	0.1951	0.3791	0.030*
N8	−0.0379 (4)	0.18381 (18)	0.38755 (18)	0.0280 (7)

H8N	−0.0926	0.1801	0.4261	0.034*
H9N	0.0646	0.1883	0.4110	0.034*
N11	0.1144 (4)	0.35008 (19)	0.42023 (18)	0.0292 (7)
H11N	0.1560	0.3470	0.3795	0.035*
C2	0.4467 (4)	0.2319 (2)	0.5101 (2)	0.0287 (8)
H2A	0.5392	0.2281	0.5523	0.034*
H2B	0.4708	0.2698	0.4707	0.034*
C3	0.4093 (4)	0.1473 (2)	0.4745 (2)	0.0276 (8)
H3	0.5000	0.1284	0.4539	0.033*
C3A	0.3788 (5)	0.0843 (2)	0.5324 (2)	0.0347 (9)
H3A1	0.3546	0.0313	0.5066	0.052*
H3A2	0.2915	0.1022	0.5542	0.052*
H3A3	0.4706	0.0786	0.5738	0.052*
C5	0.2380 (4)	0.0798 (2)	0.3613 (2)	0.0284 (8)
H5	0.2272	0.0334	0.3966	0.034*
C5A	0.3644 (4)	0.0571 (3)	0.3177 (2)	0.0352 (9)
H5A1	0.4621	0.0487	0.3547	0.053*
H5A2	0.3763	0.1016	0.2823	0.053*
H5A3	0.3356	0.0064	0.2885	0.053*
C6	0.0830 (4)	0.0920 (2)	0.3062 (2)	0.0298 (8)
H6A	0.0640	0.0441	0.2713	0.036*
H6B	0.0910	0.1409	0.2742	0.036*
C7	−0.0589 (4)	0.1032 (2)	0.3428 (2)	0.0277 (8)
C7A	−0.0691 (5)	0.0366 (2)	0.4014 (2)	0.0371 (9)
H7A1	−0.1593	0.0465	0.4239	0.056*
H7A2	0.0244	0.0374	0.4421	0.056*
H7A3	−0.0790	−0.0170	0.3759	0.056*
C7B	−0.2054 (4)	0.1054 (3)	0.2809 (2)	0.0359 (9)
H7B1	−0.1974	0.1487	0.2436	0.054*
H7B2	−0.2937	0.1165	0.3046	0.054*
H7B3	−0.2199	0.0523	0.2544	0.054*
C9	−0.0841 (5)	0.2612 (2)	0.3447 (2)	0.0359 (9)
H9A	−0.1952	0.2585	0.3205	0.043*
H9B	−0.0259	0.2667	0.3031	0.043*
C10	−0.0545 (5)	0.3363 (2)	0.3968 (3)	0.0388 (10)
H10	−0.0939	0.3240	0.4444	0.047*
C10A	−0.1490 (5)	0.4068 (3)	0.3556 (4)	0.0603 (15)
H10A	−0.1333	0.4557	0.3884	0.091*
H10B	−0.2585	0.3919	0.3444	0.091*
H10C	−0.1163	0.4184	0.3072	0.091*
C12	0.1636 (4)	0.4273 (2)	0.4622 (2)	0.0303 (8)
H12	0.0878	0.4406	0.4950	0.036*
C12A	0.1744 (5)	0.5016 (2)	0.4101 (2)	0.0382 (9)
H12A	0.0739	0.5111	0.3759	0.057*
H12B	0.2516	0.4907	0.3792	0.057*
H12C	0.2045	0.5503	0.4421	0.057*
C13	0.3223 (4)	0.4157 (2)	0.5151 (2)	0.0307 (8)
H13A	0.3547	0.4691	0.5398	0.037*

H13B	0.3964	0.4017	0.4824	0.037*
C14	0.3374 (4)	0.3508 (2)	0.5788 (2)	0.0283 (8)
C14A	0.2108 (4)	0.3575 (2)	0.6251 (2)	0.0303 (8)
H14A	0.1096	0.3539	0.5902	0.045*
H14B	0.2198	0.4103	0.6522	0.045*
H14C	0.2217	0.3127	0.6627	0.045*
C14B	0.4963 (4)	0.3570 (2)	0.6320 (2)	0.0324 (8)
H14D	0.5052	0.3157	0.6728	0.049*
H14E	0.5095	0.4119	0.6550	0.049*
H14F	0.5761	0.3474	0.6023	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0281 (5)	0.0333 (5)	0.0438 (6)	−0.0019 (3)	0.0170 (4)	−0.0056 (4)
Cl2	0.0403 (6)	0.0380 (6)	0.0367 (6)	−0.0025 (4)	0.0157 (4)	−0.0013 (4)
O1	0.0288 (18)	0.183 (5)	0.060 (2)	0.005 (2)	0.0121 (16)	−0.045 (3)
O2	0.0232 (13)	0.0397 (15)	0.0448 (16)	0.0018 (11)	0.0129 (12)	−0.0050 (12)
O3	0.050 (2)	0.0422 (19)	0.108 (3)	−0.0004 (15)	0.033 (2)	−0.0312 (19)
O4	0.105 (4)	0.058 (2)	0.156 (4)	0.017 (2)	0.099 (3)	0.041 (3)
O5	0.054 (2)	0.059 (2)	0.064 (2)	0.0037 (16)	0.0102 (17)	0.0140 (17)
O6	0.0411 (18)	0.065 (2)	0.0484 (18)	−0.0130 (15)	0.0167 (14)	−0.0025 (15)
O7	0.066 (2)	0.064 (2)	0.0416 (18)	−0.0099 (17)	0.0256 (16)	0.0037 (15)
O8	0.059 (2)	0.048 (2)	0.079 (2)	−0.0058 (16)	0.0246 (19)	−0.0237 (17)
N1	0.0215 (14)	0.0253 (15)	0.0294 (15)	−0.0008 (11)	0.0076 (12)	−0.0010 (12)
N4	0.0254 (15)	0.0241 (15)	0.0266 (15)	0.0005 (12)	0.0060 (12)	0.0015 (11)
N8	0.0244 (15)	0.0257 (16)	0.0331 (16)	0.0010 (11)	0.0038 (12)	−0.0012 (12)
N11	0.0270 (16)	0.0243 (15)	0.0351 (17)	0.0005 (12)	0.0034 (13)	−0.0022 (13)
C2	0.0209 (16)	0.0318 (19)	0.0351 (19)	−0.0021 (14)	0.0100 (14)	−0.0056 (15)
C3	0.0215 (17)	0.0288 (19)	0.0335 (19)	0.0040 (14)	0.0077 (14)	−0.0035 (15)
C3A	0.042 (2)	0.0269 (19)	0.0326 (19)	0.0072 (16)	0.0011 (16)	0.0004 (15)
C5	0.0285 (18)	0.0262 (18)	0.0306 (18)	0.0004 (14)	0.0066 (15)	0.0007 (14)
C5A	0.033 (2)	0.040 (2)	0.033 (2)	0.0050 (16)	0.0093 (16)	−0.0067 (16)
C6	0.0291 (19)	0.0305 (19)	0.0285 (18)	−0.0015 (15)	0.0031 (15)	−0.0012 (14)
C7	0.0265 (18)	0.0245 (18)	0.0322 (18)	−0.0002 (14)	0.0063 (15)	−0.0017 (14)
C7A	0.037 (2)	0.029 (2)	0.047 (2)	−0.0034 (16)	0.0130 (18)	0.0046 (17)
C7B	0.030 (2)	0.039 (2)	0.037 (2)	−0.0044 (16)	0.0045 (16)	−0.0062 (17)
C9	0.0295 (19)	0.0266 (19)	0.046 (2)	0.0020 (15)	−0.0040 (17)	0.0040 (16)
C10	0.029 (2)	0.028 (2)	0.057 (3)	0.0031 (15)	0.0028 (18)	−0.0041 (18)
C10A	0.038 (2)	0.036 (2)	0.098 (4)	0.0065 (19)	−0.009 (3)	−0.004 (2)
C12	0.0304 (19)	0.0231 (18)	0.039 (2)	0.0022 (14)	0.0106 (16)	0.0008 (15)
C12A	0.042 (2)	0.0270 (19)	0.045 (2)	−0.0008 (16)	0.0084 (18)	0.0078 (16)
C13	0.0320 (19)	0.0232 (18)	0.037 (2)	−0.0056 (14)	0.0078 (16)	−0.0025 (15)
C14	0.0271 (18)	0.0242 (18)	0.0342 (19)	−0.0027 (14)	0.0077 (15)	−0.0041 (14)
C14A	0.0298 (19)	0.0287 (18)	0.0336 (19)	0.0019 (14)	0.0095 (15)	−0.0044 (15)
C14B	0.0285 (19)	0.032 (2)	0.035 (2)	−0.0031 (15)	0.0044 (15)	−0.0068 (15)

Geometric parameters (Å, º)

Cl1—O4	1.408 (4)	C6—C7	1.542 (5)
Cl1—O3	1.418 (3)	C6—H6A	0.9900
Cl1—O1	1.435 (4)	C6—H6B	0.9900
Cl1—O2	1.445 (3)	C7—C7B	1.516 (5)
Cl2—O7	1.427 (3)	C7—C7A	1.520 (5)
Cl2—O8	1.431 (3)	C7A—H7A1	0.9800
Cl2—O5	1.440 (4)	C7A—H7A2	0.9800
Cl2—O6	1.446 (3)	C7A—H7A3	0.9800
N1—C2	1.493 (4)	C7B—H7B1	0.9800
N1—C14	1.528 (4)	C7B—H7B2	0.9800
N1—H1N	0.9200	C7B—H7B3	0.9800
N1—H2N	0.9200	C9—C10	1.524 (6)
N4—C5	1.489 (4)	C9—H9A	0.9900
N4—C3	1.489 (4)	C9—H9B	0.9900
N4—H4N	0.8800	C10—C10A	1.517 (6)
N8—C9	1.485 (5)	C10—H10	1.0000
N8—C7	1.526 (4)	C10A—H10A	0.9800
N8—H8N	0.9200	C10A—H10B	0.9800
N8—H9N	0.9200	C10A—H10C	0.9800
N11—C12	1.481 (5)	C12—C13	1.531 (5)
N11—C10	1.485 (5)	C12—C12A	1.539 (5)
N11—H11N	0.8800	C12—H12	1.0000
C2—C3	1.524 (5)	C12A—H12A	0.9800
C2—H2A	0.9900	C12A—H12B	0.9800
C2—H2B	0.9900	C12A—H12C	0.9800
C3—C3A	1.518 (5)	C13—C14	1.533 (5)
C3—H3	1.0000	C13—H13A	0.9900
C3A—H3A1	0.9800	C13—H13B	0.9900
C3A—H3A2	0.9800	C14—C14B	1.526 (5)
C3A—H3A3	0.9800	C14—C14A	1.529 (5)
C5—C6	1.523 (5)	C14A—H14A	0.9800
C5—C5A	1.534 (5)	C14A—H14B	0.9800
C5—H5	1.0000	C14A—H14C	0.9800
C5A—H5A1	0.9800	C14B—H14D	0.9800
C5A—H5A2	0.9800	C14B—H14E	0.9800
C5A—H5A3	0.9800	C14B—H14F	0.9800
O4—Cl1—O3	109.1 (3)	C7B—C7—C6	110.4 (3)
O4—Cl1—O1	109.2 (3)	C7A—C7—C6	112.0 (3)
O3—Cl1—O1	108.9 (3)	N8—C7—C6	107.4 (3)
O4—Cl1—O2	111.1 (2)	C7—C7A—H7A1	109.5
O3—Cl1—O2	111.18 (19)	C7—C7A—H7A2	109.5
O1—Cl1—O2	107.27 (19)	H7A1—C7A—H7A2	109.5
O7—Cl2—O8	110.3 (2)	C7—C7A—H7A3	109.5
O7—Cl2—O5	110.3 (2)	H7A1—C7A—H7A3	109.5
O8—Cl2—O5	109.6 (2)	H7A2—C7A—H7A3	109.5

O7—Cl2—O6	109.1 (2)	C7—C7B—H7B1	109.5
O8—Cl2—O6	109.7 (2)	C7—C7B—H7B2	109.5
O5—Cl2—O6	107.8 (2)	H7B1—C7B—H7B2	109.5
C2—N1—C14	117.5 (3)	C7—C7B—H7B3	109.5
C2—N1—H1N	107.9	H7B1—C7B—H7B3	109.5
C14—N1—H1N	107.9	H7B2—C7B—H7B3	109.5
C2—N1—H2N	107.9	N8—C9—C10	112.2 (3)
C14—N1—H2N	107.9	N8—C9—H9A	109.2
H1N—N1—H2N	107.2	C10—C9—H9A	109.2
C5—N4—C3	114.7 (3)	N8—C9—H9B	109.2
C5—N4—H4N	109.0	C10—C9—H9B	109.2
C3—N4—H4N	107.0	H9A—C9—H9B	107.9
C9—N8—C7	118.3 (3)	N11—C10—C10A	116.3 (4)
C9—N8—H8N	107.7	N11—C10—C9	109.1 (3)
C7—N8—H8N	107.7	C10A—C10—C9	107.9 (4)
C9—N8—H9N	107.7	N11—C10—H10	107.7
C7—N8—H9N	107.7	C10A—C10—H10	107.7
H8N—N8—H9N	107.1	C9—C10—H10	107.7
C12—N11—C10	116.3 (3)	C10—C10A—H10A	109.5
C12—N11—H11N	109.5	C10—C10A—H10B	109.5
C10—N11—H11N	109.5	H10A—C10A—H10B	109.5
N1—C2—C3	111.9 (3)	C10—C10A—H10C	109.5
N1—C2—H2A	109.2	H10A—C10A—H10C	109.5
C3—C2—H2A	109.2	H10B—C10A—H10C	109.5
N1—C2—H2B	109.2	N11—C12—C13	109.9 (3)
C3—C2—H2B	109.2	N11—C12—C12A	114.5 (3)
H2A—C2—H2B	107.9	C13—C12—C12A	107.5 (3)
N4—C3—C3A	111.4 (3)	N11—C12—H12	108.3
N4—C3—C2	108.4 (3)	C13—C12—H12	108.3
C3A—C3—C2	112.7 (3)	C12A—C12—H12	108.3
N4—C3—H3	108.1	C12—C12A—H12A	109.5
C3A—C3—H3	108.1	C12—C12A—H12B	109.5
C2—C3—H3	108.1	H12A—C12A—H12B	109.5
C3—C3A—H3A1	109.5	C12—C12A—H12C	109.5
C3—C3A—H3A2	109.5	H12A—C12A—H12C	109.5
H3A1—C3A—H3A2	109.5	H12B—C12A—H12C	109.5
C3—C3A—H3A3	109.5	C12—C13—C14	117.7 (3)
H3A1—C3A—H3A3	109.5	C12—C13—H13A	107.9
H3A2—C3A—H3A3	109.5	C14—C13—H13A	107.9
N4—C5—C6	108.5 (3)	C12—C13—H13B	107.9
N4—C5—C5A	112.6 (3)	C14—C13—H13B	107.9
C6—C5—C5A	111.4 (3)	H13A—C13—H13B	107.2
N4—C5—H5	108.1	C14B—C14—N1	110.2 (3)
C6—C5—H5	108.1	C14B—C14—C14A	110.4 (3)
C5A—C5—H5	108.1	N1—C14—C14A	105.1 (3)
C5—C5A—H5A1	109.5	C14B—C14—C13	109.7 (3)
C5—C5A—H5A2	109.5	N1—C14—C13	108.5 (3)
H5A1—C5A—H5A2	109.5	C14A—C14—C13	112.8 (3)

C5—C5A—H5A3	109.5	C14—C14A—H14A	109.5
H5A1—C5A—H5A3	109.5	C14—C14A—H14B	109.5
H5A2—C5A—H5A3	109.5	H14A—C14A—H14B	109.5
C5—C6—C7	116.9 (3)	C14—C14A—H14C	109.5
C5—C6—H6A	108.1	H14A—C14A—H14C	109.5
C7—C6—H6A	108.1	H14B—C14A—H14C	109.5
C5—C6—H6B	108.1	C14—C14B—H14D	109.5
C7—C6—H6B	108.1	C14—C14B—H14E	109.5
H6A—C6—H6B	107.3	H14D—C14B—H14E	109.5
C7B—C7—C7A	110.3 (3)	C14—C14B—H14F	109.5
C7B—C7—N8	110.5 (3)	H14D—C14B—H14F	109.5
C7A—C7—N8	106.0 (3)	H14E—C14B—H14F	109.5
C14—N1—C2—C3	180.0 (3)	C7—N8—C9—C10	−179.3 (3)
C5—N4—C3—C3A	−63.2 (4)	C12—N11—C10—C10A	49.5 (5)
C5—N4—C3—C2	172.2 (3)	C12—N11—C10—C9	171.8 (3)
N1—C2—C3—N4	62.0 (4)	N8—C9—C10—N11	70.5 (4)
N1—C2—C3—C3A	−61.8 (4)	N8—C9—C10—C10A	−162.3 (4)
C3—N4—C5—C6	169.6 (3)	C10—N11—C12—C13	152.2 (3)
C3—N4—C5—C5A	−66.6 (4)	C10—N11—C12—C12A	−86.7 (4)
N4—C5—C6—C7	−63.7 (4)	N11—C12—C13—C14	−61.2 (4)
C5A—C5—C6—C7	171.8 (3)	C12A—C12—C13—C14	173.6 (3)
C9—N8—C7—C7B	−37.0 (4)	C2—N1—C14—C14B	−44.5 (4)
C9—N8—C7—C7A	−156.5 (3)	C2—N1—C14—C14A	−163.4 (3)
C9—N8—C7—C6	83.6 (4)	C2—N1—C14—C13	75.7 (4)
C5—C6—C7—C7B	−173.8 (3)	C12—C13—C14—C14B	−171.8 (3)
C5—C6—C7—C7A	−50.5 (4)	C12—C13—C14—N1	67.7 (4)
C5—C6—C7—N8	65.6 (4)	C12—C13—C14—C14A	−48.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2n···N11	0.92	2.07	2.841 (4)	140
N8—H9n···N4	0.92	1.95	2.763 (5)	146
N1—H1n···O2	0.92	2.37	2.963 (4)	122
N1—H1n···O7[i]	0.92	2.41	3.169 (4)	140
N1—H2n···N4	0.92	2.56	2.929 (4)	105
N4—H4n···O6	0.88	2.38	3.212 (4)	158
N8—H8n···O1	0.92	2.02	2.937 (5)	177
N11—H11n···O6	0.88	2.24	3.087 (5)	162
C2—H2a···O1[ii]	0.99	2.57	3.109 (5)	114
C9—H9a···O5[iii]	0.99	2.57	3.498 (6)	155
C12a—H12b···O8	0.98	2.56	3.487 (5)	158

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