Crystal structure of 3,14-dimethyl-2,13-diaza-6,17-diazoniatricyclo[16.4.0.0^{7,12}]docosane bis(perchlorate) from synchrotron X-ray data

Dohyun Moon,[a] Sunghwan Jeon[b] and Jong-Ha Choi[b]*

[a]Pohang Accelerator Laboratory, POSTECH, Pohang 37673, Republic of Korea, and [b]Department of Chemistry, Andong National University, Andong 36729, Republic of Korea. *Correspondence e-mail: jhchoi@anu.ac.kr

The crystal structure of the title salt, $C_{20}H_{42}N_4^{2+} \cdot 2ClO_4^-$, has been determined using synchrotron radiation at 220 (2) K. The structure determination reveals that protonation has occurred at diagonally opposite amine N atoms. The asymmetric unit comprises one half of the organic dication, which lies about a center of inversion, and one perchlorate anion. The macrocyclic dication adopts the most stable endodentate *trans*-III conformation. The crystal structure is stabilized by intramolecular N—H···N, and intermolecular N—H···O and C–H···O hydrogen bonds involving the macrocycle N—H and C—H groups as donors and the O atoms of perchlorate anions as acceptors, giving rise to a three-dimensional network.

1. Chemical context

The macrocyclic compound, 3,14-dimethyl-2,6,13,17-tetraazatricyclo(16.4.0.0^{7,12})docosane ($C_{20}H_{40}N_4$) contains a cyclam backbone with two cyclohexane subunits and two methyl groups are also attached to carbon atoms 3 and 14 of the propyl chains that bridge opposite pairs of N atoms in the structure. The macrocycle is basic and readily captures two or four protons to form the $[C_{20}H_{42}N_4]^{2+}$ dication or the $[C_{20}H_{44}N_4]^{4+}$ tetracation in which all of the N—H bonds are generally available for hydrogen-bond formation (Moon *et al.*, 2021).

Previously, the crystal structures of $[Cu(C_{20}H_{40}N_4)](NO_3)_2 \cdot 3H_2O$, $[Cu(C_{20}H_{40}N_4)](NO_3)_2$, $[Cu(C_{20}H_{40}N_4)](ClO_4)_2$ and $[Cu(C_{20}H_{40}N_4)(H_2O)_2](BF_4)_2 \cdot 2H_2O$ were reported together with $[Zn(C_{20}H_{40}N_4)(OCOCH_3)_2]$. In these structures, the copper(II) or zinc(II) cations have tetragonally distorted octahedral environments with the four N atoms of the macrocyclic ligand in equatorial positions and the O atoms of the counter-anions, water molecules or acetato ligands in axial positions (Choi *et al.*, 2006, 2007, 2012*a*,*b*; Ross *et al.*, 2012). In these Cu^{II} and Zn^{II} complexes, the macrocyclic ligands adopt their most stable *trans*-III configurations. The crystal structures of $(C_{20}H_{40}N_4) \cdot 2(C_{11}H_{10}O)$ (Choi *et al.*, 2012*c*), $(C_{20}H_{40}N_4) \cdot 2(NO_2OH)$ (Moon *et al.*, 2020), $[C_{20}H_{42}N_4](SO_4) \cdot 2MeOH$ (White *et al.*, 2015), $[C_{20}H_{42}N_4]Br_2 \cdot 2H_2O$ (Moon *et al.*, 2021) and $[C_{20}H_{44}N_4]Br_4 \cdot 4H_2O$ (Moon *et al.*, 2021) have also been determined.

We report here the preparation of a new dicationic compound, $[C_{20}H_{42}N_4](ClO_4)_2$, (I) and its structural characterization by synchrotron single-crystal X-ray diffraction.

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$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
$N1—H1N1\cdots O3^i$	0.86 (2)	2.22 (2)	3.007 (2)	152.4 (18)
$N2—H2A\cdots O1$	0.90	2.09	2.970 (2)	164
$N2—H2A\cdots O2$	0.90	2.56	3.239 (2)	132
$N2—H2B\cdots N1^{ii}$	0.90	2.29	2.9846 (16)	134
$N2—H2B\cdots N1$	0.90	2.39	2.8230 (17)	109
$C7—H7A\cdots O2^{iii}$	0.98	2.57	3.423 (3)	145

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 2, -z + 1$; (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.
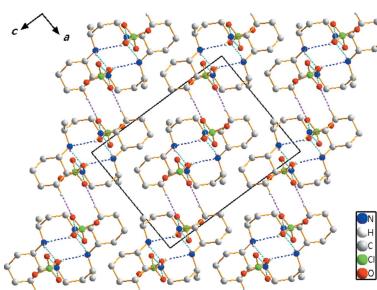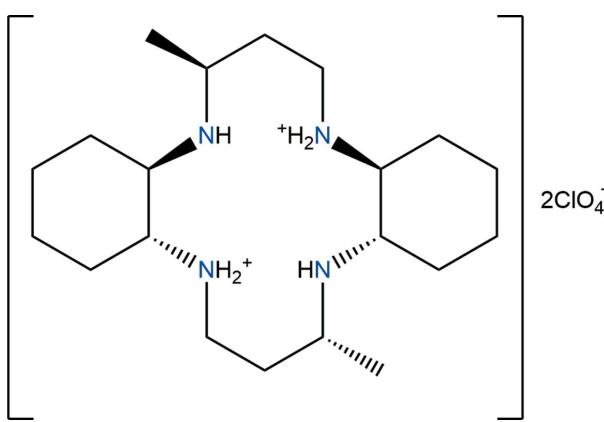
2. Structural commentary

An ellipsoid plot of the molecular components in (I) with the atom-numbering scheme is shown in Fig. 1. The asymmetric unit consists of one half of the macrocyclic dication, which lies about a center of inversion, and one perchlorate anion. The four N atoms are coplanar, and the two methyl substituents are *anti* with respect to the macrocyclic plane as a result of the molecular inversion symmetry. The $[C_{20}H_{42}N_4]^{2+}$ dication adopts an endodentate conformation and *trans*-III configuration along the center of the macrocyclic cavity. The endo conformation of the dication may be due to the intramolecular $N—H\cdots N$ hydrogen-bonding interaction. Within the centrosymmetric diprotonated amine unit, the C—C and N—C bond lengths range from 1.5173 (18) to 1.5368 (18) Å and from 1.4795 (16) to 1.5044 (16) Å, respectively. The range of N—C—C and C—N—C angles is 108.89 (11) to 113.50 (11)° and 113.46 (11) to 114.61 (11)°, respectively. The bond lengths and angles within the dication are comparable to those found in the free ligand or other cations in $(C_{20}H_{40}N_4)\cdot 2C_{11}H_{10}O$ (Choi *et al.*, 2012*c*), $[C_{20}H_{42}N_4](SO_4)\cdot 2MeOH$ (White *et al.*, 2015) and $[C_{20}H_{42}N_4][Fe\{HB(pz)_3\}(CN)_3]_2\cdot 2H_2O\cdot 2MeOH$ (Kim *et*

al., 2004; pz = pyrazolyl). The protonation of the N atoms may depend on the location of the neighboring counter-anions involved in hydrogen bonding. The bond-length difference can be noticed for several N—C bonds. The N—C bond length involving the non-protonated N1 atom is shorter than that involving protonated N2 atom, *e.g.* N1—C2 [1.4817 (18) Å] and N1—C3 [1.4795 (16) Å] are slightly shorter than N2—C8 [1.5044 (16) Å] and N2—C9 [1.4952 (18) Å]. Each of the two hydrogen atoms of N2 and N2′ ($-x + 1, -y + 2, -z + 1$) is involved in hydrogen bonding with both of the two remaining nitrogen atoms (Table 1). The intramolecular hydrogen bonding plays a substantial role in maintaining the endodentate geometry of the diprotonated macrocyclic cation. The Cl—O bond distances in the tetrahedral ClO_4^- anion vary from 1.4218 (19) to 1.4529 (16) Å, and the O—Cl—O angles vary from 106.45 (10) to 110.51 (12)°. The distorted geometry of the ClO_4^- anion undoubtedly results from its involvement in hydrogen-bonding interactions with the organic cation.

3. Supramolecular features

Three $N—H\cdots O$, $C—H\cdots O$ and $N—H\cdots N$ hydrogen-bonding interactions occur in the crystal structure (Table 1). The O atoms of the perchlorate anions serve as hydrogen-bond
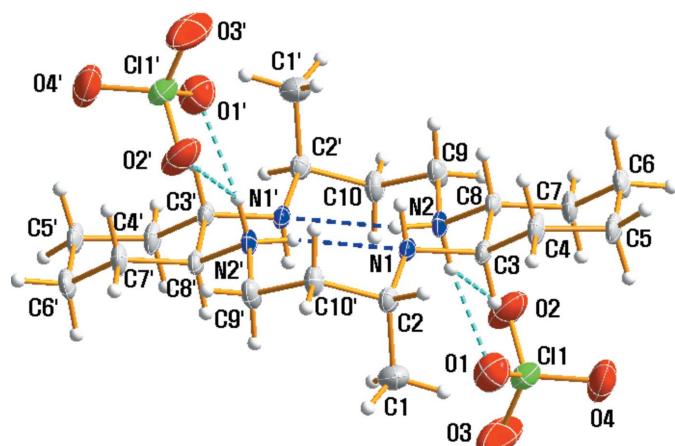
Figure 1
The molecular structure of compound (I), drawn with displacement ellipsoids at the 50% probability level. Dashed lines represent hydrogen-bonding interactions and primed atoms are related by the symmetry operation ($-x + 1, -y + 2, -z + 1$).
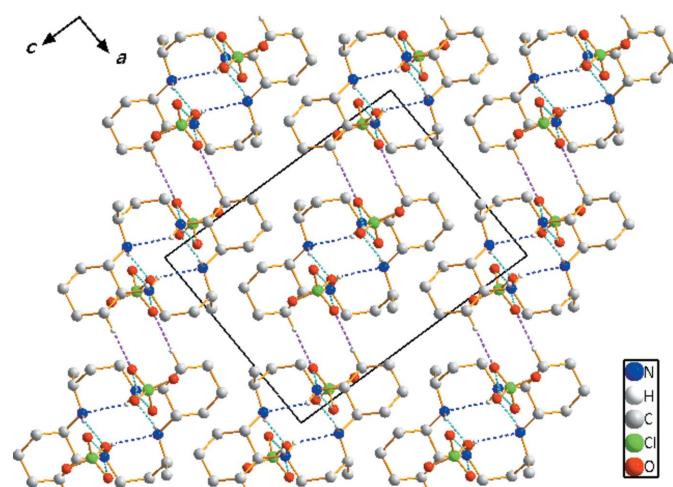
Figure 2
Crystal packing in compound (I), viewed perpendicular to the *ac* plane. Dashed lines represent $N—H\cdots O$ (cyan), $N—H\cdots N$ (blue) and $C—H\cdots O$ (purple) hydrogen-bonding interactions, respectively.

acceptors. The ClO_4^- anions are connected to the $[C_{20}H_{42}N_4]^{2+}$ dication by N—H···O hydrogen bonds. The macrocyclic dication is linked to a neighboring ClO_4^- anion through a very weak C—H···O hydrogen bond. The extensive array of these contacts generates a three-dimensional network structure (Fig. 2), and these hydrogen-bonding interactions help to stabilize the crystal structure.

4. Database survey

A search of the Cambridge Structural (Version 5.42, Update 1, February 2021; Groom *et al.*, 2016) indicated 121 hits for organic and transition-metal compounds containing the macrocycles $(C_{20}H_{40}N_4)$, $[C_{20}H_{42}N_4]^{2+}$ or $[C_{20}H_{44}N_4]^{4+}$. The crystal structures of $(C_{20}H_{40}N_4)\cdot2C_{11}H_{10}O$ (Choi *et al.*, 2012*c*), $[C_{20}H_{42}N_4](SO_4)\cdot2MeOH$ (White *et al.*, 2015), $[C_{20}H_{42}N_4]Br_2\cdot2H_2O$ (Moon *et al.*, 2021), $[C_{20}H_{44}N_4]Cl_4\cdot4H_2O$ (Moon *et al.*, 2018) and $[C_{20}H_{44}N_4]Br_4\cdot4H_2O$ (Moon *et al.*, 2021) were reported previously and commented on in the *Chemical context* section.

5. Synthesis and crystallization

Commercially available *trans*-1,2-cyclohexanediamine and methyl vinyl ketone (Sigma–Aldrich) were used as provided. All chemicals were reagent grade and used without further purification. As a starting material, macrocycle 3,14-dimethyl-2,6,13,17-tetraazatricyclo(16.4.0.0^{7,12})docosane, *L*, was prepared according to a published procedure (Kang *et al.*, 1991). Macrocycle *L* (0.034 g, 0.1 mmol) was suspended in methanol (20 mL) and the pH was adjusted to 3.0 with 0.5 *M* $HClO_4$. The mixture was stirred magnetically for 30 min and the resulting solution was filtered. The neat filtrate was allowed to stand for one week to give block-like colorless crystals of (I) suitable for X-ray structural analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All non-hydrogen atoms were refined anisotropically. All C-bound H atoms and the hydrogen atoms of the diprotonated amine (H2*A* and H2*B*) were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.97–0.98 Å and an N—H distance of 0.99 Å, and with U_{iso}(H) values of 1.5 and 1.2 times, respectively, that of the parent atoms. The one N-bound H atom (H1*N*1) of the amine was assigned based on a difference-Fourier map, and a U_{iso}(H) value of $1.5U_{eq}$(N1).

Acknowledgements

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{42}N_4^{2+}\cdot2ClO_4^-$
M_r	537.47
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	220
a, b, c (Å)	10.689 (2), 8.4450 (17), 14.020 (3)
β (°)	92.90 (3)
V (Å^3)	1263.9 (4)
Z	2
Radiation type	Synchrotron, $\lambda = 0.630$ Å
μ (mm^{-1})	0.22
Crystal size (mm)	$0.08 \times 0.08 \times 0.08$
Data collection	
Diffractometer	Rayonix MX225HS CCD area detector
Absorption correction	Empirical (using intensity measurements) (*HKL3000sm SCALEPACK*; Otwinowski *et al.*, 2003)
T_{min}, T_{max}	0.957, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12842, 3549, 3164
R_{int}	0.063
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.696
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.172, 1.11
No. of reflections	3549
No. of parameters	158
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.86, −0.44

Computer programs: *PAL BL2D-SMDC* (Shin *et al.*, 2016), *HKL3000sm* (Otwinowski & Minor, 1997), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *DIAMOND 4* (Putz & Brandenburg, 2014) and *publCIF* (Westrip, 2010).

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Crystal structure of 3,14-dimethyl-2,13-diaza-6,17-diazoniatricyclo-[16.4.0.0^{7,12}]docosane bis(perchlorate) from synchrotron X-ray data

Dohyun Moon, Sunghwan Jeon and Jong-Ha Choi

Computing details

Data collection: *PAL BL2D-SMDC* (Shin *et al.*, 2016); cell refinement: *HKL3000sm* (Otwinowski & Minor, 1997); data reduction: *HKL3000sm* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXT2018* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *DIAMOND 4* (Putz & Brandenburg, 2014); software used to prepare material for publication: *publCIF* (Westrip, 2010).

3,14-Dimethyl-2,13-diaza-6,17-diazoniatricyclo[16.4.0.0^{7,12}]docosane bis(perchlorate)

Crystal data
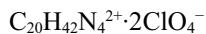
$C_{20}H_{42}N_4{}^{2+}\cdot 2ClO_4{}^-$	$F(000) = 576$
$M_r = 537.47$	$D_x = 1.412$ Mg m^{-3}
Monoclinic, $P2_1/n$	Synchrotron radiation, $\lambda = 0.630$ Å
$a = 10.689$ (2) Å	Cell parameters from 41946 reflections
$b = 8.4450$ (17) Å	$\theta = 0.4–33.6°$
$c = 14.020$ (3) Å	$\mu = 0.22$ mm^{-1}
$\beta = 92.90$ (3)°	$T = 220$ K
$V = 1263.9$ (4) Å^3	Block, colorless
$Z = 2$	$0.08 \times 0.08 \times 0.08$ mm

Data collection

Rayonix MX225HS CCD area detector diffractometer	12842 measured reflections
	3549 independent reflections
Radiation source: PLSII 2D bending magnet	3164 reflections with $I > 2\sigma(I)$
ω scan	$R_{int} = 0.063$
Absorption correction: empirical (using intensity measurements) (*HKL3000sm* Scalepack; Otwinowski *et al.*, 2003)	$\theta_{max} = 26.0°$, $\theta_{min} = 2.5°$
	$h = -14 \rightarrow 14$
	$k = -11 \rightarrow 11$
	$l = -19 \rightarrow 19$
$T_{min} = 0.957$, $T_{max} = 1.000$	

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.055$	
$wR(F^2) = 0.172$	$w = 1/[\sigma^2(F_o^2) + (0.1016P)^2 + 0.4023P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	
3549 reflections	$(\Delta/\sigma)_{max} < 0.001$
158 parameters	$\Delta\rho_{max} = 0.86$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.44$ e Å^{-3}

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.35546 (11)	1.04262 (14)	0.58019 (8)	0.0179 (2)
H1N1	0.3881 (18)	1.135 (3)	0.5913 (14)	0.027*
N2	0.58037 (10)	0.87373 (14)	0.61832 (7)	0.0189 (2)
H2A	0.556980	0.771653	0.612584	0.023*
H2B	0.556374	0.923391	0.563613	0.023*
C1	0.17869 (17)	0.8824 (2)	0.51236 (13)	0.0357 (4)
H1A	0.104402	0.890989	0.470004	0.054*
H1B	0.158772	0.823502	0.569045	0.054*
H1C	0.243896	0.827482	0.479816	0.054*
C2	0.22417 (13)	1.04715 (17)	0.54095 (10)	0.0208 (3)
H2	0.171026	1.086474	0.591704	0.025*
C3	0.37355 (13)	0.95865 (16)	0.67248 (9)	0.0186 (3)
H3	0.339745	0.850008	0.664720	0.022*
C4	0.30834 (15)	1.03951 (18)	0.75469 (10)	0.0254 (3)
H4A	0.337406	1.149317	0.760396	0.030*
H4B	0.217795	1.041505	0.739748	0.030*
C5	0.33416 (16)	0.95505 (19)	0.85002 (10)	0.0270 (3)
H5A	0.296287	1.015186	0.900932	0.032*
H5B	0.295502	0.849816	0.847313	0.032*
C6	0.47429 (16)	0.9383 (2)	0.87300 (10)	0.0284 (3)
H6A	0.488076	0.877269	0.932053	0.034*
H6B	0.511244	1.043474	0.883362	0.034*
C7	0.53896 (14)	0.85522 (19)	0.79198 (9)	0.0258 (3)
H7A	0.629365	0.849658	0.807106	0.031*
H7B	0.506976	0.746930	0.784522	0.031*
C8	0.51363 (13)	0.94746 (16)	0.69916 (9)	0.0187 (3)
H8	0.546260	1.056300	0.708844	0.022*
C9	0.71994 (13)	0.8814 (2)	0.63136 (9)	0.0257 (3)
H9A	0.747588	0.810782	0.683724	0.031*
H9B	0.744648	0.989510	0.649456	0.031*
C10	0.78531 (13)	0.83475 (19)	0.54199 (9)	0.0239 (3)
H10A	0.750585	0.733364	0.519265	0.029*
H10B	0.874193	0.817560	0.559494	0.029*
Cl1	0.54918 (4)	0.43816 (5)	0.65415 (3)	0.03296 (16)
O1	0.45977 (15)	0.55690 (19)	0.61926 (13)	0.0495 (4)
O2	0.67029 (15)	0.5118 (2)	0.65563 (14)	0.0618 (5)
O3	0.5462 (2)	0.3046 (2)	0.59233 (17)	0.0762 (6)
O4	0.51936 (16)	0.3923 (2)	0.74864 (12)	0.0598 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0238 (5)	0.0176 (5)	0.0127 (5)	−0.0015 (4)	0.0047 (4)	0.0024 (4)
N2	0.0246 (5)	0.0218 (6)	0.0109 (4)	0.0014 (4)	0.0056 (4)	0.0023 (4)
C1	0.0388 (8)	0.0307 (8)	0.0374 (9)	−0.0110 (7)	−0.0011 (6)	−0.0010 (7)
C2	0.0229 (6)	0.0235 (7)	0.0165 (6)	0.0007 (5)	0.0065 (4)	0.0003 (5)
C3	0.0262 (6)	0.0175 (6)	0.0128 (5)	0.0000 (5)	0.0073 (4)	0.0028 (4)
C4	0.0347 (7)	0.0271 (7)	0.0155 (6)	0.0061 (6)	0.0121 (5)	0.0040 (5)
C5	0.0407 (8)	0.0275 (7)	0.0139 (6)	0.0017 (6)	0.0128 (5)	0.0031 (5)
C6	0.0427 (8)	0.0326 (8)	0.0105 (6)	−0.0001 (6)	0.0061 (5)	0.0000 (5)
C7	0.0349 (7)	0.0315 (7)	0.0113 (5)	0.0049 (6)	0.0060 (5)	0.0046 (5)
C8	0.0265 (6)	0.0194 (6)	0.0106 (5)	−0.0004 (5)	0.0063 (4)	0.0006 (4)
C9	0.0242 (6)	0.0387 (8)	0.0146 (6)	0.0006 (6)	0.0047 (4)	0.0006 (5)
C10	0.0264 (6)	0.0291 (7)	0.0168 (6)	0.0062 (5)	0.0063 (5)	0.0030 (5)
Cl1	0.0328 (2)	0.0270 (3)	0.0393 (3)	−0.00471 (14)	0.00435 (17)	0.00776 (14)
O1	0.0454 (8)	0.0462 (9)	0.0564 (9)	0.0068 (6)	−0.0015 (7)	0.0120 (7)
O2	0.0423 (8)	0.0658 (11)	0.0770 (12)	−0.0215 (8)	0.0003 (8)	0.0287 (10)
O3	0.1111 (16)	0.0318 (8)	0.0893 (14)	−0.0193 (10)	0.0399 (12)	−0.0118 (9)
O4	0.0577 (9)	0.0755 (12)	0.0463 (9)	−0.0126 (9)	0.0036 (7)	0.0284 (9)

Geometric parameters (Å, º)

N1—C3	1.4795 (16)	C5—C6	1.523 (2)
N1—C2	1.4817 (18)	C5—H5A	0.9800
N1—H1N1	0.86 (2)	C5—H5B	0.9800
N2—C9	1.4952 (18)	C6—C7	1.530 (2)
N2—C8	1.5044 (16)	C6—H6A	0.9800
N2—H2A	0.9000	C6—H6B	0.9800
N2—H2B	0.9000	C7—C8	1.5290 (18)
C1—C2	1.521 (2)	C7—H7A	0.9800
C1—H1A	0.9700	C7—H7B	0.9800
C1—H1B	0.9700	C8—H8	0.9900
C1—H1C	0.9700	C9—C10	1.5173 (18)
C2—C10^i	1.5314 (19)	C9—H9A	0.9800
C2—H2	0.9900	C9—H9B	0.9800
C3—C8	1.5278 (19)	C10—H10A	0.9800
C3—C4	1.5368 (18)	C10—H10B	0.9800
C3—H3	0.9900	Cl1—O3	1.4218 (19)
C4—C5	1.528 (2)	Cl1—O4	1.4315 (16)
C4—H4A	0.9800	Cl1—O2	1.4354 (15)
C4—H4B	0.9800	Cl1—O1	1.4529 (16)
C3—N1—C2	114.61 (11)	H5A—C5—H5B	108.0
C3—N1—H1N1	103.8 (13)	C5—C6—C7	111.23 (13)
C2—N1—H1N1	114.3 (13)	C5—C6—H6A	109.4
C9—N2—C8	113.46 (11)	C7—C6—H6A	109.4
C9—N2—H2A	108.9	C5—C6—H6B	109.4

C8—N2—H2A	108.9	C7—C6—H6B	109.4
C9—N2—H2B	108.9	H6A—C6—H6B	108.0
C8—N2—H2B	108.9	C8—C7—C6	109.34 (13)
H2A—N2—H2B	107.7	C8—C7—H7A	109.8
C2—C1—H1A	109.5	C6—C7—H7A	109.8
C2—C1—H1B	109.5	C8—C7—H7B	109.8
H1A—C1—H1B	109.5	C6—C7—H7B	109.8
C2—C1—H1C	109.5	H7A—C7—H7B	108.3
H1A—C1—H1C	109.5	N2—C8—C3	109.71 (11)
H1B—C1—H1C	109.5	N2—C8—C7	111.10 (11)
N1—C2—C1	110.99 (12)	C3—C8—C7	111.66 (11)
N1—C2—C10ⁱ	108.89 (11)	N2—C8—H8	108.1
C1—C2—C10ⁱ	112.81 (13)	C3—C8—H8	108.1
N1—C2—H2	108.0	C7—C8—H8	108.1
C1—C2—H2	108.0	N2—C9—C10	112.70 (11)
C10ⁱ—C2—H2	108.0	N2—C9—H9A	109.1
N1—C3—C8	109.08 (10)	C10—C9—H9A	109.1
N1—C3—C4	113.50 (11)	N2—C9—H9B	109.1
C8—C3—C4	108.65 (12)	C10—C9—H9B	109.1
N1—C3—H3	108.5	H9A—C9—H9B	107.8
C8—C3—H3	108.5	C9—C10—C2ⁱ	116.25 (13)
C4—C3—H3	108.5	C9—C10—H10A	108.2
C5—C4—C3	112.33 (12)	C2ⁱ—C10—H10A	108.2
C5—C4—H4A	109.1	C9—C10—H10B	108.2
C3—C4—H4A	109.1	C2ⁱ—C10—H10B	108.2
C5—C4—H4B	109.1	H10A—C10—H10B	107.4
C3—C4—H4B	109.1	O3—Cl1—O4	110.51 (12)
H4A—C4—H4B	107.9	O3—Cl1—O2	110.20 (14)
C6—C5—C4	111.15 (12)	O4—Cl1—O2	110.28 (11)
C6—C5—H5A	109.4	O3—Cl1—O1	110.37 (13)
C4—C5—H5A	109.4	O4—Cl1—O1	108.95 (11)
C6—C5—H5B	109.4	O2—Cl1—O1	106.45 (10)
C4—C5—H5B	109.4		
C3—N1—C2—C1	−67.02 (15)	C9—N2—C8—C7	−65.28 (15)
C3—N1—C2—C10ⁱ	168.21 (11)	N1—C3—C8—N2	−53.86 (14)
C2—N1—C3—C8	173.99 (10)	C4—C3—C8—N2	−178.05 (10)
C2—N1—C3—C4	−64.72 (15)	N1—C3—C8—C7	−177.48 (11)
N1—C3—C4—C5	−177.06 (12)	C4—C3—C8—C7	58.32 (15)
C8—C3—C4—C5	−55.52 (16)	C6—C7—C8—N2	177.49 (12)
C3—C4—C5—C6	54.72 (17)	C6—C7—C8—C3	−59.68 (16)
C4—C5—C6—C7	−55.07 (17)	C8—N2—C9—C10	−169.40 (12)
C5—C6—C7—C8	57.14 (16)	N2—C9—C10—C2ⁱ	71.50 (17)
C9—N2—C8—C3	170.77 (11)		

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1*N*1⋯O3[ii]	0.86 (2)	2.22 (2)	3.007 (2)	152.4 (18)
N2—H2*A*⋯O1	0.90	2.09	2.970 (2)	164
N2—H2*A*⋯O2	0.90	2.56	3.239 (2)	132
N2—H2*B*⋯N1[i]	0.90	2.29	2.9846 (16)	134
N2—H2*B*⋯N1	0.90	2.39	2.8230 (17)	109
C7—H7*A*⋯O2[iii]	0.98	2.57	3.423 (3)	145

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) *x*, *y*+1, *z*; (iii) −*x*+3/2, *y*+1/2, −*z*+3/2.