8-Methyl-5-oxo-5,6-dihydrodipyrido[1,2-a:3′,2′-e]-pyrimidin-11-ium chloride trihydrate 120 K

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Key indicators

Single-crystal X-ray study

$T = 120$ K

Mean σ(C–C) = 0.003 Å

Disorder in solvent or counterion

R factor = 0.032

wR factor = 0.090

Data-to-parameter ratio = 15.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the structure of the title salt, $C_{12}H_{10}N_3O^+ \cdot Cl^- \cdot 3H_2O$, the only species which do not coincide with the mirror plane in the space group $P6_3/m$ are one complete water molecule, one H atom of another water molecule and two H atoms of the methyl group of the cation. Consequently, apart from the two H atoms of the methyl group, the cation is completely planar. The hydrogen bonding between the water molecules and, through Cl^- anions, to the cations, although extensive, can be modelled satisfactorily only by treating one H atom in each of the two types of water molecules as disordered.

Comment

The title compound, (I), is an example of a more elaborate, potentially biologically active, pyridine-containing compound of the type provided by 2-chloronicotinoyl chloride, (II), as precursor, undergoing cyclization with a dinucleophilic reagent, in this case 2-amino-4-methylpyridine, (III). The asymmetric unit in the structure of (I) is shown in Fig. 1. Bond lengths and bond angles within the cation of (I) are summarized in Table 1. Particularly notable are the C4a—C5 and C10a—N2 distances of 1.468 (2) and 1.429 (2) Å, respectively, and the bond angles N6—C5—C4a [114.00 (14)°] and C6a—N6—C5 [126.04 (14)°]. The somewhat extreme variation in bond lengths and more especially bond angles suggests that the cation is not altogether an aromatic species. This is certainly true of the lactam ring defined by C4a/C5/N6/C6a/N2/C10a. In the refinement of the structure of (I) described here, the only atoms which are not coincident with a mirror plane of the space group $P6_3/m$ are two H atoms of the methyl group, one H atom of the water molecule involving O2 and the entire water molecule involving O3. As a consequence the cation, with the exception of the methyl H atoms, is completely flat and the entire structure can be described in terms of well defined layers parallel to (001) at $z = \frac{1}{4}$ and $\frac{3}{4}$ such as that shown in Fig. 2. Adjacent layers are related to one another by the operation of crystallographic centres of symmetry. The layers are also connected to one another by hydrogen bonding as described later. A further consequence of the mirror symmetry is that both water molecules must be present in two distinct orientations which must be present in equal numbers resulting, therefore, in disorder. For the water molecule involving O2, H1W is ordered but H2W is distributed over two mirror-plane-related sites both of occupancy 0.5. The atom H3W is common to both orientations of the water molecule involving O3 and is accompanied by H4W in one orientation and H5W in the other. Thus, while the H3W site is fully occupied, H4W and H5W both have occupancies of 0.5. The disorder of the H atoms has serious implications for the disposition of the hydrogen bonds in which they are
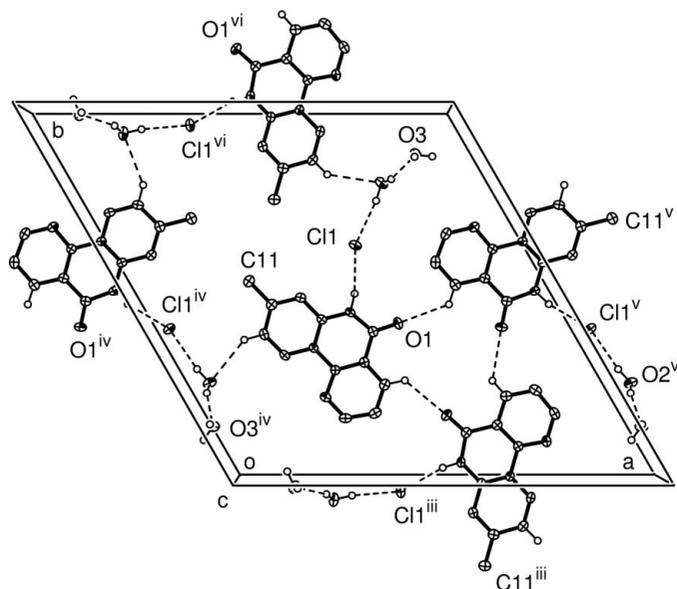
organic papers
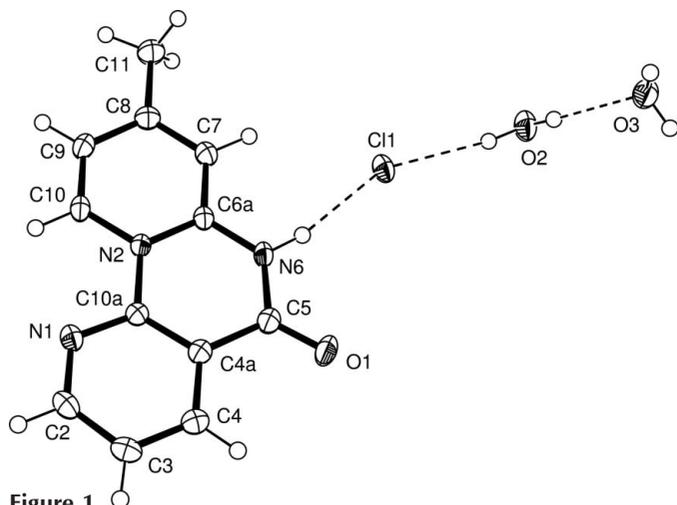
Figure 1
The asymmetric unit of (I), together with other H atoms to complete the molecules. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines represent hydrogen bonds.
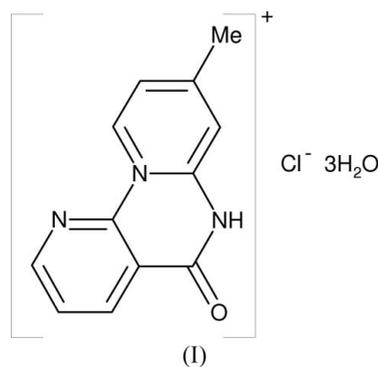

Figure 2
A layer of ions at $z = \frac{1}{4}$. Displacement ellipsoids are drawn at the 50% probability level and H atoms involved in hydrogen-bond formation (dashed lines) are shown as small spheres of arbitrary radii. Selected atoms are labelled. [Symmetry codes: (iii) $1 - y, x - y, z$; (iv) $y - x, 1 - x$, z; (v) $1 + y - x, 1 - x, z$; (vi) $1 - y, 1 + x - y, z$.]

involved (Table 2). As shown in Fig. 3, the majority of the hydrogen bonds given in Table 2 interconnect the water molecules to form infinite tube-like columns propagated in the direction of c. These constitute spines of connectivity with branches at $z = \frac{1}{4}$ and $\frac{3}{4}$ to Cl1 atoms which, by means of the N6—H6···Cl1 hydrogen bonds, extend the linkage to the cations. The surface of the aqueous 'tube' comprises six chains of the form shown in Fig. 4 connected to one another by hydrogen bonds of the form O3—H3W···O3^i [symmetry code (i) $1 + x - y, x, 1 - z$]. Notable here is the polarity, in the example shown in Fig. 4 in the positive direction of z, of the donor to acceptor alignment of the hydrogen bonds involved in the propagation of the chain in the [001] direction. Fig. 4
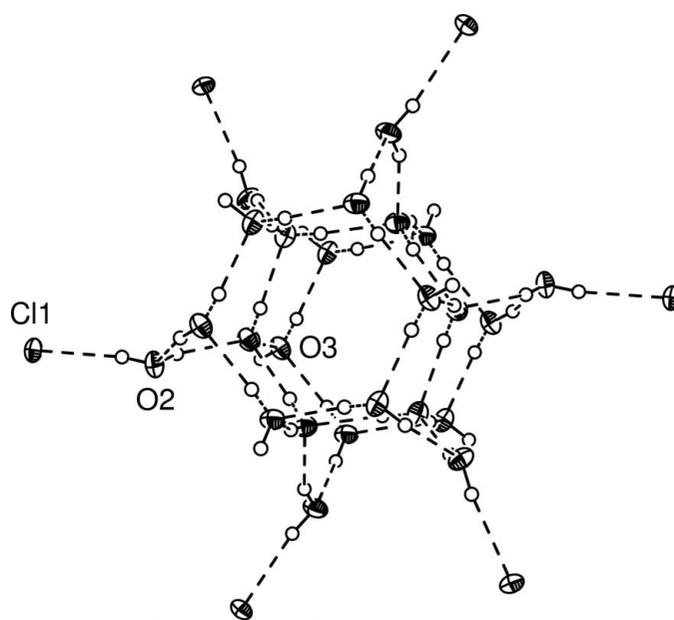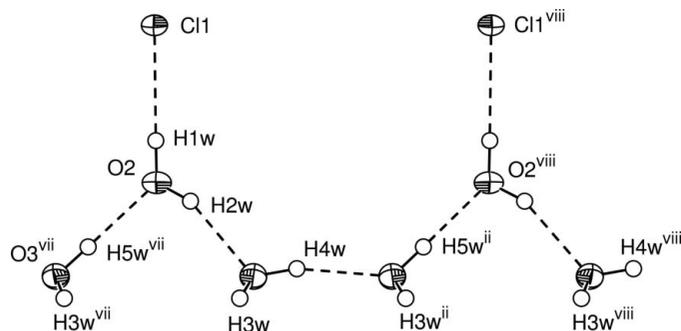

Figure 3
Hydrogen-bonds (dashed lines) within a column of water molecules and the attached Cl⁻ anions in the structure of (I). The view is approximately down c, with a running left to right across the page. The H atoms shown have been chosen so as to provide an ordered arrangement. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Selected atoms are labelled. The unit cell outline has been omitted for clarity.

shows an arbitrary choice of mutually compatible H atoms consistent with only one of the two possible polarities of the chain. This ordered arrangement is clearly incompatible, as far as the H atoms are concerned, with the mirror planes upon which the O2 atoms lie and which relate the O3 atoms to one another. Clearly chains of this and of the opposite polarity must be distributed throughout the structure in equal numbers in order to bring about the observed disorder. Weak C—H···O contacts, also given in Table 2, provide, as shown in Fig. 2, inter-cation connectivity within the layers.

(I)

Experimental

To a solution of 2-chloronicotinoyl chloride, (II) (1.0 g, 5.68 mmol), in anhydrous tetrahydrofuran (30 ml) were successively added, with stirring, 2-amino-4-methylpyridine, (III) (1.1 ml, 5.68 mmol), and

Figure 4
A more detailed view, with an arbitrary choice of mutually compatible H atoms, of the hydrogen-bond connectivity between O2 and O3 along the length of a single chain within the column of Fig. 3. The c axis runs from left to right across the page. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Selected atoms are labelled. The unit cell outline has been omitted for clarity. [Symmetry codes: (ii) $x, y, \frac{3}{2} - z$; (vii) $x, y, \frac{1}{2} - z$; (viii) $x, y, 1 + z$.]

triethylamine (1.63 ml, 11.36 mmol) at room temperature. The reaction mixture was stirred for 8 h at room temperature, quenched with water (20 ml), and ethyl acetate (15 ml) was added. The organic layer was collected, washed with saturated sodium bicarbonate solution (2 × 20 ml), dried over sodium sulfate and rotary evaporated. The residue was purified by chromatography, with hexane–ethyl acetate (7:3) as eluant. The sample used in the crystallographic study was recrystallized from ethanol (m.p. 535–536 K). ^{1}H NMR [400.00 MHz (FIDRES ±0.15 Hz), DMSO-d_6]: δ 9.80 (1H, d, J = 7.3 Hz, H10), 9.15 (1H, dd, J = 2.0 and 4.8 Hz, H2), 8.80 (1H, dd, J = 2.0 and 8.0 Hz, H4), 8.03 (1H, dd, J = 4.8 and 8.0 Hz, H3), 7.71 (1H, s, H7), 7.64 (1H, dd, J = 2.0 and 7.2 Hz, H9), 2.65 (3H, s, CH$_3$). ^{13}C NMR (100.0 MHz, DMSO-d_6): δ 159.4 (C5), 157.9 (C10), 154.3 (C2), 147.0 (C6a), 146.1 (C10a), 137.9 (C4), 129.9 (C9), 126.7 (C3), 120.7 (C4a), 116.7 (C8), 114.8 (C7), 21.8 (CH$_3$). IR (cm^{-1}, KBr disk): ν_{max} 3080 (NH), 1712 (C=O).

Crystal data

C$_{12}$H$_{10}$N$_3$O$^+$·Cl$^-$·3H$_2$O	Mo $K\alpha$ radiation
M_r = 301.73	Cell parameters from 3270
Hexagonal, $P6_3/m$	reflections
a = 19.4419 (3) Å	θ = 2.9–27.5°
c = 6.5498 (1) Å	μ = 0.28 mm^{-1}
V = 2144.05 (6) Å^3	T = 120 (2) K
Z = 6	Block, colourless
D_x = 1.402 Mg m^{-3}	0.24 × 0.12 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer	1783 independent reflections
φ and ω scans	1600 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	R_{int} = 0.036
(*SADABS*; Sheldrick, 2003)	θ_{max} = 27.5°
T_{min} = 0.649, T_{max} = 1.000	$h = -22 \rightarrow 25$
16137 measured reflections	$k = -25 \rightarrow 25$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.6158P]$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	
$wR(F^2)$ = 0.090	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.15	$(\Delta/\sigma)_{max}$ = 0.001
1783 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
118 parameters	$\Delta\rho_{min}$ = -0.23 e Å^{-3}
H-atom parameters constrained	

Table 1
Summary of geometric parameters (Å, °) in the cation of (I).

Parameter^a	Min.	Max.^{b}
C$_4$—C$_3^c$	1.498 (2)	
C$_3$—C$_3$	1.351 (2)	1.468 (2)
C$_3$—N$_3$	1.358 (2)	1.429 (2)
C$_3$—N$_2$	1.323 (2)	1.333 (2)
C$_3$—O$_1^d$	1.216 (2)	
X—Y—Z^e	114.00 (14)	126.04 (14)

Notes: (a) bond type indicated by atoms with subscripts corresponding to the atom connectivities; (b) only present for multiple occurrences; (c) C11—C8; (d) C5—O1; (e) internal angles of the ring system.

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N6—H6$\cdots$Cl1	0.88	2.23	3.0976 (15)	168
O2—H1$W\cdots$Cl1	0.86	2.25	3.1061 (13)	172
O2—H2$W\cdots$O3	0.82	1.94	2.7108 (13)	157
O3—H3$W\cdots$O3^i	0.81	1.93	2.7263 (9)	172
O3—H4$W\cdots$O3^{ii}	0.86	1.86	2.7042 (19)	165
O3—H5$W\cdots$O2	0.85	1.86	2.7108 (13)	177
C4—H4$\cdots$O1^{iii}	0.95	2.50	3.179 (2)	128
C9—H9$\cdots$O2^{iv}	0.95	2.54	3.357 (2)	144

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

Initial positions for the H atoms of the water molecules were obtained from difference maps, revised to provide a realistic hydrogen-bonding scheme and the geometry of the water molecules idealized to provide O—H distances and H—O—H angles in the ranges 0.81–0.86 Å and 105–111°, respectively. All other H atoms were placed in calculated positions, with C—H distances set at 0.95 and 0.98 Å for aryl and methyl H atoms, respectively, and the N—H distance set to 0.88 Å for the H atom of the NH group, placed as for an aryl H atom. The H atoms of the methyl group, H11A and H11B, were placed in positions appropriate to the mirror plane site symmetry of the group whose orientation was therefore fixed accordingly. In all cases, the H atoms were then refined using a riding model, with U_{iso}(H) = 1.3U_{eq}(C,N,O).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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supporting information

8-Methyl-5-oxo-5,6-dihydrodipyrido[1,2-*a*:3′,2′-e]pyrimidin-11-ium chloride trihydrate 120 K

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8-Methyl-5-oxo-5,6-dihydrodipyrido[1,2 - a:3′,2′-e]pyrimidin-11-ium chloride trihydrate

Crystal data

$C_{12}H_{10}N_3O^+·Cl^-·3H_2O$	Melting point = 535–536 K
$M_r = 301.73$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hexagonal, $P6_3/m$	Cell parameters from 3270 reflections
$a = 19.4419$ (3) Å	$\theta = 2.9$–27.5°
$c = 6.5498$ (1) Å	$\mu = 0.28$ mm^{-1}
$V = 2144.05$ (6) Å^3	$T = 120$ K
$Z = 6$	Block, colourless
$F(000) = 948$	$0.24 \times 0.12 \times 0.10$ mm
$D_x = 1.402$ Mg m^{-3}	

Data collection

Bruker–Nonius KappaCCD diffractometer	$T_{min} = 0.649$, $T_{max} = 1.000$
Radiation source: Bruker-Nonius FR591 rotating anode	16137 measured reflections
	1783 independent reflections
10 cm confocal mirrors monochromator	1600 reflections with $I > 2\sigma(I)$
Detector resolution: 9.091 pixels mm^{-1}	$R_{int} = 0.036$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -22 \rightarrow 25$
	$k = -25 \rightarrow 25$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: geom and difmap
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.090$	$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.6158P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	
1783 reflections	$(\Delta/\sigma)_{max} = 0.001$
118 parameters	$\Delta\rho_{max} = 0.25$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. Unit cell determined with *DIRAX* (Duisenberg, 1992; Duisenberg *et al.* 2000) but refined with the *DENZO/COLLECT HKL* package.
Refs as: Duisenberg, A. J. *M*. (1992). J. Appl. Cryst. 25, 92–96. Duisenberg, A. J. *M*., Hooft, *R*. W. W., Schreurs, A. *M. M.* & Kroon, J. (2000). J. Appl. Cryst. 33, 893–898.

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}	Occ. (<1)
Cl1	0.59489 (2)	0.63371 (2)	0.2500	0.02589 (14)	
N1	0.31683 (9)	0.21861 (8)	0.2500	0.0233 (3)	
N2	0.35594 (8)	0.35325 (8)	0.2500	0.0183 (3)	
N6	0.49140 (8)	0.45024 (8)	0.2500	0.0222 (3)	
H6	0.5271	0.5011	0.2500	0.029*	
O1	0.58999 (7)	0.42027 (8)	0.2500	0.0368 (4)	
O2	0.74225 (8)	0.80351 (8)	0.2500	0.0312 (3)	
H1W	0.7048	0.7548	0.2500	0.041*	
H2W	0.7687	0.8119	0.3541	0.041*	0.50
O3	0.85472 (5)	0.87284 (5)	0.54356 (15)	0.0294 (2)	
H3W	0.8927	0.8697	0.5071	0.038*	
H4W	0.8471	0.8648	0.6729	0.038*	0.50
H5W	0.8202	0.8503	0.4499	0.038*	0.50
C2	0.33541 (11)	0.16126 (10)	0.2500	0.0272 (4)	
H2	0.2932	0.1077	0.2500	0.035*	
C3	0.41286 (11)	0.17494 (11)	0.2500	0.0268 (4)	
H3	0.4231	0.1320	0.2500	0.035*	
C4	0.47436 (11)	0.25274 (11)	0.2500	0.0242 (4)	
H4	0.5280	0.2644	0.2500	0.032*	
C4A	0.45646 (10)	0.31379 (10)	0.2500	0.0212 (4)	
C5	0.51927 (10)	0.39755 (10)	0.2500	0.0242 (4)	
C6A	0.41393 (9)	0.43090 (9)	0.2500	0.0189 (3)	
C7	0.39353 (10)	0.49036 (10)	0.2500	0.0225 (4)	
H7	0.4342	0.5445	0.2500	0.029*	
C8	0.31520 (11)	0.47143 (10)	0.2500	0.0234 (4)	
C9	0.25658 (10)	0.39012 (11)	0.2500	0.0238 (4)	
H9	0.2020	0.3753	0.2500	0.031*	
C10	0.27715 (10)	0.33322 (10)	0.2500	0.0217 (4)	
H10	0.2369	0.2788	0.2500	0.028*	
C10A	0.37690 (10)	0.29249 (9)	0.2500	0.0193 (3)	
C11	0.29336 (11)	0.53520 (11)	0.2500	0.0299 (4)	
H11A	0.2354	0.5108	0.2500	0.039*	

H11B	0.3152	0.5682	0.1278	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0230 (2)	0.0188 (2)	0.0271 (2)	0.00393 (16)	0.000	0.000
N1	0.0208 (7)	0.0186 (7)	0.0260 (8)	0.0065 (6)	0.000	0.000
N2	0.0153 (6)	0.0180 (6)	0.0201 (7)	0.0071 (5)	0.000	0.000
N6	0.0151 (6)	0.0168 (7)	0.0301 (8)	0.0044 (5)	0.000	0.000
O1	0.0155 (6)	0.0294 (7)	0.0634 (10)	0.0097 (5)	0.000	0.000
O2	0.0219 (6)	0.0267 (7)	0.0346 (7)	0.0044 (5)	0.000	0.000
O3	0.0230 (4)	0.0340 (5)	0.0285 (5)	0.0123 (4)	0.0006 (4)	−0.0004 (4)
C2	0.0300 (9)	0.0173 (8)	0.0299 (10)	0.0086 (7)	0.000	0.000
C3	0.0356 (10)	0.0232 (8)	0.0251 (9)	0.0173 (8)	0.000	0.000
C4	0.0260 (9)	0.0268 (9)	0.0231 (8)	0.0156 (7)	0.000	0.000
C4A	0.0203 (8)	0.0224 (8)	0.0208 (8)	0.0107 (7)	0.000	0.000
C5	0.0196 (8)	0.0234 (8)	0.0291 (9)	0.0103 (7)	0.000	0.000
C6A	0.0168 (7)	0.0182 (8)	0.0186 (8)	0.0064 (6)	0.000	0.000
C7	0.0212 (8)	0.0185 (8)	0.0253 (9)	0.0082 (7)	0.000	0.000
C8	0.0260 (9)	0.0245 (8)	0.0218 (8)	0.0143 (7)	0.000	0.000
C9	0.0169 (8)	0.0266 (9)	0.0270 (9)	0.0101 (7)	0.000	0.000
C10	0.0157 (7)	0.0207 (8)	0.0231 (8)	0.0049 (6)	0.000	0.000
C10A	0.0208 (8)	0.0182 (8)	0.0177 (8)	0.0089 (6)	0.000	0.000
C11	0.0294 (9)	0.0284 (9)	0.0367 (10)	0.0179 (8)	0.000	0.000

Geometric parameters (Å, º)

N1—C10A	1.323 (2)	C3—C4	1.382 (3)
N1—C2	1.333 (2)	C3—H3	0.9500
N2—C6A	1.359 (2)	C4—C4A	1.394 (2)
N2—C10	1.379 (2)	C4—H4	0.9500
N2—C10A	1.429 (2)	C4A—C10A	1.387 (2)
N6—C6A	1.358 (2)	C4A—C5	1.468 (2)
N6—C5	1.378 (2)	C6A—C7	1.397 (2)
N6—H6	0.8800	C7—C8	1.376 (2)
O1—C5	1.216 (2)	C7—H7	0.9500
O2—H1W	0.8587	C8—C9	1.413 (2)
O2—H2W	0.8196	C8—C11	1.498 (2)
O3—H3W	0.8060	C9—C10	1.351 (2)
O3—H4W	0.8605	C9—H9	0.9500
O3—H5W	0.8516	C10—H10	0.9500
C2—C3	1.392 (3)	C11—H11A	0.9800
C2—H2	0.9500	C11—H11B	0.9800
C10A—N1—C2	116.55 (15)	O1—C5—C4A	124.43 (16)
C6A—N2—C10	120.06 (14)	N6—C5—C4A	114.00 (14)
C6A—N2—C10A	119.81 (13)	N6—C6A—N2	119.78 (14)
C10—N2—C10A	120.13 (13)	N6—C6A—C7	120.36 (14)

C6A—N6—C5	126.04 (14)	N2—C6A—C7	119.86 (14)
C6A—N6—H6	117.0	C8—C7—C6A	120.84 (15)
C5—N6—H6	117.0	C8—C7—H7	119.6
H1W—O2—H2W	108.7	C6A—C7—H7	119.6
H3W—O3—H4W	111.0	C7—C8—C9	117.70 (15)
H3W—O3—H5W	105.4	C7—C8—C11	120.82 (16)
H4W—O3—H5W	126.6	C9—C8—C11	121.47 (16)
N1—C2—C3	124.05 (16)	C10—C9—C8	120.84 (15)
N1—C2—H2	118.0	C10—C9—H9	119.6
C3—C2—H2	118.0	C8—C9—H9	119.6
C4—C3—C2	118.06 (16)	C9—C10—N2	120.70 (15)
C4—C3—H3	121.0	C9—C10—H10	119.7
C2—C3—H3	121.0	N2—C10—H10	119.7
C3—C4—C4A	118.98 (16)	N1—C10A—C4A	124.85 (15)
C3—C4—H4	120.5	N1—C10A—N2	115.84 (14)
C4A—C4—H4	120.5	C4A—C10A—N2	119.31 (14)
C10A—C4A—C4	117.50 (15)	C8—C11—H11A	109.5
C10A—C4A—C5	121.07 (15)	H11Bⁱ—C11—H11B	109.5
C4—C4A—C5	121.43 (15)	C8—C11—H11B	109.5
O1—C5—N6	121.57 (16)	H11A—C11—H11B	109.5

Symmetry code: (i) x, y, $-z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···Cl1	0.88	2.23	3.0976 (15)	168
O2—H1W···Cl1	0.86	2.25	3.1061 (13)	172
O2—H2W···O3	0.82	1.94	2.7108 (13)	157
O3—H3W···O3ⁱⁱ	0.81	1.93	2.7263 (9)	172
O3—H4W···O3ⁱⁱⁱ	0.86	1.86	2.7042 (19)	165
O3—H5W···O2	0.85	1.86	2.7108 (13)	177
C4—H4···O1ⁱᵛ	0.95	2.50	3.179 (2)	128
C7—H7···Cl1	0.95	2.71	3.4904 (17)	140
C9—H9···O2ᵛ	0.95	2.54	3.357 (2)	144

Symmetry codes: (ii) $x-y+1$, x, $-z+1$; (iii) x, y, $-z+3/2$; (iv) $-y+1$, $x-y$, z; (v) $-x+y$, $-x+1$, z.