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2-Amino-5-chloropyridin-1-ium barbiturate dihydrate

Thankappan Ramalakshmi Anantheeswary,[a] Sundaramoorthy Gomathi,[a]* Jeyaraman Selvaraj Nirmalram[b] and Franc Perdih[c]

[a]Department of Chemistry, Periyar Maniammai Institute of Science & Technology, Thanjavur, Tamilnadu-613403, India, [b]Department of Chemistry, Government Arts and Science College, Karambakudi, Pudukottai, Tamilnadu-622302, India, and [c]Faculty of Chemistry and Chemical Technology, University of Ljubljana, Slovenia. *Correspondence e-mail: gomathichemist@pmu.edu

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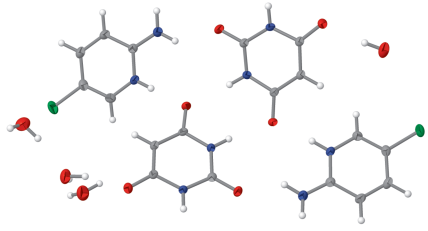
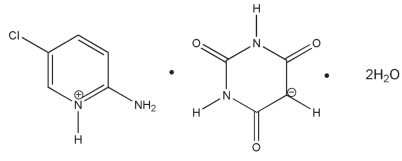
Structural data: full structural data are available from iucrdata.iucr.org

In the title hydrated salt, $C_5H_6ClN_2^+\cdot C_4H_3N_2O_3^-\cdot 2H_2O$, the asymmetric unit contains two cations, two anions and four water molecules of crystallization. In the extended structure, the barbiturate (2,4,6-trioxo-1,3-diazinan-5-ide) anions form a wave-like supramolecular [001] chain decorated by the cations, mediated by numerous N—H···O hydrogen bonds. Adjacent chains are cross-linked by [010] chains of water molecules featuring four- and six-membered rings and five-membered carboxylate aggregates *via* O—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

The crystal structure of 2-amino-5-chloropyridine, $C_5H_5ClN_2$ (Kvick & Backéus, 1974) and its diverse salts with dicarboxylic acids (Jayanalina *et al.*, 2015), aromatic acids (Hanif *et al.*, 2020) and other inorganic anions such as nitrate (Zaouali Zgolli *et al.*, 2009), phosphate (Akriche & Rzaigui, 2005), sulfonate (Jagan & Boopathi, 2020) and trifluoroacetate (Hemamalini & Fun, 2010) have been extensively studied, highlighting its diverse hydrogen-bonding interactions. Barbiturates derived from barbituric acid ($C_4H_4N_2O_3$) play a significant role in biological systems (Hueso Ureña *et al.*, 2003). Research on barbiturate salts and co-crystals demonstrate that hydrogen bonding is a key driving force for structure and property modulation.

Hydrogen bonding not only increases the reactivity and electrophilicity of barbiturates (Bauer & Spange, 2010), but also governs their supramolecular organization in host–guest systems and polymeric assemblies, particularly those involving Hamilton-type receptors (Chang & Hamilton, 1988). Thus hydrogen bonding acts as the primary driving force behind barbiturate self-assembly and functional behaviour and indicates its significance in supramolecular chemistry, materials science and biomimetic design. As

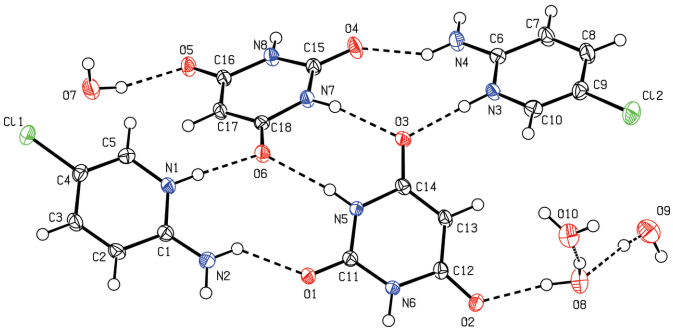


Figure 1
The molecular structure of (**I**) with displacement ellipsoids drawn at the 50% probability level.

part of our studies in this area, we now report the synthesis and structure of the title hydrated salt, $C_5H_6ClN_2^+\cdot C_4H_3N_2O_3^-\cdot 2H_2O$ (**I**).

The asymmetric unit of (**I**) consists of two crystallographically independent 2-amino-5-chloropyridinium cations, two barbiturate anions and four water molecules of crystallization in space group $P2_1/c$. The proton acceptance from the barbituric acid occurs at the pyridine ring N atoms of the cations (Fig. 1) which is evident from the widening of the C1—N1—C5 and C6—N3—C10 bond angles [122.67 (13) and 122.84 (14)°, respectively] compared to the unprotonated molecule, in which the bond angle is around 118° (Anantheeswary *et al.*, 2024). The deprotonation of the barbituric acid occurs from the active methylene groups (atoms C13 and C17) driven by the electron withdrawing carbonyl group at both sides and this is supported by the sp^2 hybridization at these atoms in (**I**) implied by the C—C—C bond angles [C12—C13—C14 = 121.55 (14); C16—C17—C18 = 121.61 (14)°]. Overall, both barbiturate anions display a nearly planar six-membered ring: the deviations of the atoms from the mean plane are small (±0.025 Å and ±0.013 Å for the O1 and O4 anions, respectively). These structural parameters align well with known data for barbiturate systems (Gelbrich *et al.*, 2015) and indicate that the barbiturate anion in (**I**) adopts a stabilized, delocalized and nearly planar conformation.

In the extended structure of (**I**), the anions are linked by pairwise N—H···O hydrogen bonds into wave-like [001] ribbons, with the O1 and O4 anions alternating in the chains. Atoms N5 and N7 (donors) and carbonyl oxygen atoms O3 and O6 (acceptors) form one pairwise linkage and atoms N6 and N8 (donors) and O1 and O4 (acceptors) the other, which leads to two distinct $R_2^2(8)$ ring motifs (Table 1). The [001] chains are decorated by the cations: the C1 cation links to atoms O1, O5 and O6 in the anions and the C6 cation to O2, O3 and O4 *via* strong N—H···O hydrogen bonds to render $R_3^2(10)$ ring motifs. Taken together, these hydrogen bonds lead to propagate a wave-like supramolecular ribbon as shown in Fig. 2. Adjacent anion/cation supramolecular sheets are connected by four- and six-membered [010] tape-like arrays of water molecules which form various O_w—$H_w\cdots O_w$ and O_w—$H_w\cdots O_c$ (w = water, c = carbonyl) hydrogen bonds. This arrangement has a close resemblance to the water tape *T*4(2) and *T*6(2) motifs in the systematic classification of hydrated organic crystal structures reported in the literature (Infantes & Motherwell, 2002). In addition, the interaction between the

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6	0.87	1.85	2.7207 (18)	176
N2—H2*A*···O1	0.89	2.02	2.7461 (18)	138
N2—H2*B*···O5^{i}	0.89	1.87	2.7610 (17)	180
N3—H3*A*···O3	0.86	1.83	2.6863 (18)	177
N4—H4*A*···O4	0.87	2.00	2.7248 (18)	139
N4—H4*B*···O2^{ii}	0.89	1.97	2.8606 (17)	173
N5—H5*A*···O6	0.87	2.02	2.8786 (17)	173
N6—H6···O4^{i}	0.89	2.03	2.9005 (17)	170
N7—H7*A*···O3	0.87	2.06	2.8985 (17)	163
N8—H8*A*···O1^{ii}	0.88	2.01	2.8762 (16)	172
O7—H7*B*···O5	0.86	1.92	2.7581 (16)	164
O7—H7*C*···O2^{iii}	0.82	2.13	2.9155 (18)	160
O8—H8*B*···O10	0.86	1.89	2.739 (2)	167
O8—H8*C*···O2	0.89	1.91	2.7810 (16)	165
O9—H9*A*···O8	0.86	1.93	2.7864 (18)	168
O10—H10*B*···O7^{iii}	0.87	1.96	2.8287 (18)	175
C10—H10···O7^{iii}	0.95	2.44	3.321 (2)	153

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

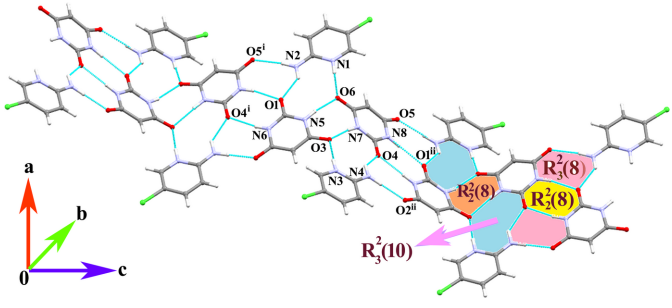


Figure 2
A view of a wave-like supramolecular chain built up from N—H···O hydrogen bonds [symmetry codes: (i) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$].

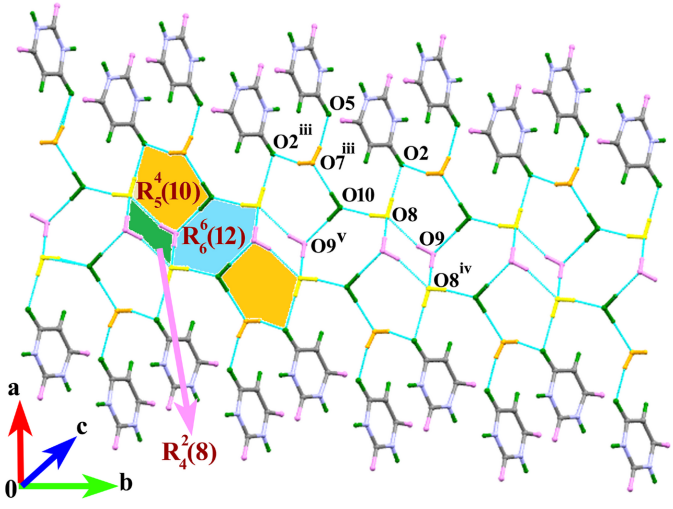


Figure 3
A water chain extending along the *c*-axis direction cross-linking the supramolecular chains [symmetry code: (iii) $1-x, \frac{1}{2}+y, 3/2-z$].

water molecules O7, O9 and O10 and the carbonyl O2 atom of the anion leads to an $R_5^4(10)$ ring motif. The existence of the four- and six-membered water loops and five-membered carbonyl–water loops generates two different *DDAA* (*D* = donor, *A* = acceptor) hydrogen-bonded arrays with ring motifs of $R_5^4(10)$, $R_6^6(12)$ and $R_5^4(10)$ and $R_5^4(10)$, $R_4^2(8)$ and $R_5^4(10)$, along the *a* axis direction as shown in Fig. 3.

A possible offset aromatic π–π stacking interaction between barbiturate rings [$Cg1\cdots Cg2^{vi}$; symmetry code: (vi) $1-x, -\frac{1}{2}+y, 3/2-z$; *Cg*1 = C11–C14/N5/N6 centroid; *Cg*2 = C15–C18/N7/N8 centroid] occurs with centroid-to-centroid and perpendicular distances of 3.7978 (9) and 3.4530 (6) Å, respectively. However, the large slippage angle of 30.5° suggests that this interaction is weak.

Synthesis and crystallization

The title compound was synthesized by mixing 20 ml ethanol: water (1:1 *v*/*v*) solutions of 2-amino-5-chloropyridine (0.25 mmol) and barbituric acid (0.25 mmol) and the resulting clear solution was then warmed over a water bath for 20 min at 353 K. The solution was then allowed to cool to room temperature and after a few days, colourless crystals of (**I**) were separated out from the mother liquor.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_5H_6ClN_2^+\cdot C_4H_3N_2O_3^-\cdot 2H_2O$
M_r	292.68
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	18.8228 (10), 6.9495 (4), 19.3563 (10)
β (°)	99.817 (5)
V (Å^3)	2494.9 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.33
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	SuperNova, Dual, Cu at home/near, Atlas
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2019)
T_{min}, T_{max}	0.812, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13946, 6542, 5147
R_{int}	0.023
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.712
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.108, 1.04
No. of reflections	6542
No. of parameters	343
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.30

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020), *Mercury* (Macrae *et al.*, 2020), *POVRay* (Cason, 2004), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2026). **11**, x260351 [https://doi.org/10.1107/S2414314626003512]

2-Amino-5-chloropyridin-1-ium barbiturate dihydrate

Thankappan Ramalakshmi Anantheeswary, Sundaramoorthy Gomathi, Jeyaraman Selvaraj Nirmalram and Franc Perdih

2-Amino-5-chloropyridin-1-ium 2,4,6-trioxo-1,3-diazinan-5-ide dihydrate

Crystal data

$C_5H_6ClN_2^+·C_4H_3N_2O_3^-·2H_2O$
M_r = 292.68
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 18.8228 (10) Å
b = 6.9495 (4) Å
c = 19.3563 (10) Å
β = 99.817 (5)°
V = 2494.9 (2) Å^3
Z = 8
$F(000)$ = 1216
D_x = 1.558 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4971 reflections
θ = 3.6–30.0°
μ = 0.33 mm^{-1}
T = 150 K
Prism, yellow
0.30 × 0.20 × 0.20 mm

Data collection

SuperNova, Dual, Cu at home/near, Atlas diffractometer
Radiation source: micro-focus sealed X-ray tube
SuperNova (Mo) X-ray Source monochromator
Detector resolution: 10.4933 pixels mm^{-1}
ωw scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2019)
T_{min} = 0.812, T_{max} = 1.000
13946 measured reflections
6542 independent reflections
5147 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 30.4°, θ_{min} = 2.6°
h = −24→24
k = −9→6
l = −18→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.04
6542 reflections
343 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
W = 1/[$\Sigma^2(FO^2)$ + (0.0476P)^2 + 0.6984P] WHERE P = (FO^2 + 2FC^2)/3
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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.
The H atoms of the $–NH_2$ groups were located from a difference Fourier map and refined freely. The other H atoms were placed geometrically and refined using a riding model.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.87257 (2)	0.58875 (6)	0.79957 (2)	0.0262 (1)
Cl2	0.01456 (2)	0.65022 (7)	0.64189 (2)	0.0331 (1)
O1	0.48161 (6)	0.29532 (19)	0.55832 (5)	0.0241 (3)
O2	0.24377 (6)	0.15226 (18)	0.50080 (5)	0.0232 (3)
O3	0.33892 (6)	0.45699 (17)	0.71542 (5)	0.0209 (3)
N5	0.40902 (7)	0.3677 (2)	0.63664 (6)	0.0172 (3)
N6	0.36206 (6)	0.2313 (2)	0.52973 (6)	0.0180 (3)
O4	0.39978 (6)	0.39067 (19)	0.89731 (6)	0.0272 (4)
O5	0.63610 (6)	0.23191 (18)	0.95562 (5)	0.0229 (3)
O6	0.53956 (6)	0.46773 (18)	0.73026 (5)	0.0216 (3)
C11	0.42087 (8)	0.2982 (2)	0.57385 (7)	0.0178 (4)
C12	0.29255 (8)	0.2292 (2)	0.54502 (7)	0.0178 (4)
C13	0.28334 (8)	0.3118 (2)	0.60855 (7)	0.0190 (4)
C14	0.34145 (8)	0.3827 (2)	0.65576 (7)	0.0173 (4)
N7	0.47177 (7)	0.4344 (2)	0.81589 (6)	0.0189 (4)
N8	0.51837 (7)	0.3158 (2)	0.92568 (6)	0.0199 (4)
C15	0.46008 (8)	0.3809 (2)	0.88069 (8)	0.0192 (4)
C16	0.58734 (8)	0.2981 (2)	0.90937 (8)	0.0178 (4)
C17	0.59555 (8)	0.3545 (2)	0.84172 (7)	0.0190 (4)
C18	0.53785 (8)	0.4195 (2)	0.79346 (7)	0.0177 (4)
N1	0.67547 (7)	0.4856 (2)	0.70029 (6)	0.0192 (4)
N2	0.62458 (7)	0.3966 (2)	0.58823 (7)	0.0261 (4)
C1	0.68229 (8)	0.4424 (2)	0.63372 (8)	0.0196 (4)
C2	0.75242 (8)	0.4493 (2)	0.61612 (8)	0.0217 (5)
C3	0.81029 (8)	0.4945 (2)	0.66553 (8)	0.0220 (4)
C4	0.79994 (8)	0.5347 (2)	0.73472 (8)	0.0199 (4)
C5	0.73289 (8)	0.5300 (2)	0.75058 (8)	0.0199 (4)
N3	0.20843 (7)	0.53881 (19)	0.74635 (6)	0.0188 (4)
N4	0.25572 (7)	0.4593 (2)	0.86099 (7)	0.0244 (4)
C6	0.19906 (8)	0.5027 (2)	0.81288 (8)	0.0193 (4)
O7	0.77655 (6)	0.2806 (2)	0.93547 (6)	0.0325 (4)
C7	0.12861 (9)	0.5152 (3)	0.82787 (8)	0.0253 (5)
C8	0.07259 (9)	0.5605 (3)	0.77660 (8)	0.0265 (5)
C9	0.08531 (9)	0.5963 (2)	0.70812 (8)	0.0224 (5)
C10	0.15318 (9)	0.5862 (2)	0.69438 (8)	0.0217 (4)
O8	0.09636 (6)	0.2133 (2)	0.49087 (6)	0.0346 (4)
O9	−0.00582 (7)	0.1255 (2)	0.57464 (7)	0.0385 (4)

O10	0.11145 (7)	0.6003 (2)	0.47080 (7)	0.0395 (4)
H5A	0.44601	0.40394	0.66680	0.0210*
H6	0.36970	0.18200	0.48947	0.0220*
H13	0.23625	0.31983	0.61984	0.0230*
H7A	0.43332	0.46700	0.78672	0.0230*
H8A	0.51061	0.27394	0.96639	0.0240*
H17	0.64191	0.34797	0.82868	0.0230*
H1	0.63257	0.47895	0.71181	0.0230*
H2	0.75892	0.42205	0.56951	0.0260*
H2A	0.58118	0.38803	0.60077	0.0310*
H2B	0.62828	0.35503	0.54527	0.0310*
H3	0.85722	0.49913	0.65372	0.0260*
H5	0.72573	0.55782	0.79696	0.0240*
H3A	0.25032	0.51691	0.73631	0.0230*
H4A	0.29875	0.44901	0.85025	0.0290*
H4B	0.24985	0.43301	0.90465	0.0290*
H7	0.12025	0.49171	0.87417	0.0300*
H8	0.02506	0.56796	0.78680	0.0320*
H10	0.16226	0.61216	0.64850	0.0260*
H7B	0.73332	0.24406	0.93811	0.0490*
H7C	0.77982	0.39306	0.94911	0.0490*
H8B	0.09589	0.33708	0.48869	0.0520*
H8C	0.14319	0.18508	0.50149	0.0520*
H9A	0.03030	0.14603	0.55342	0.0580*
H9B	−0.02290	0.01903	0.55642	0.0580*
H10A	0.07720	0.68196	0.45889	0.0590*
H10B	0.14490	0.65296	0.50189	0.0590*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0180 (2)	0.0300 (2)	0.0287 (2)	−0.0023 (2)	−0.0017 (1)	−0.0038 (2)
Cl2	0.0236 (2)	0.0411 (3)	0.0310 (2)	0.0059 (2)	−0.0059 (2)	0.0034 (2)
O1	0.0148 (5)	0.0390 (7)	0.0196 (5)	−0.0032 (5)	0.0062 (4)	−0.0073 (5)
O2	0.0161 (5)	0.0331 (7)	0.0197 (5)	−0.0034 (5)	0.0015 (4)	−0.0030 (5)
O3	0.0169 (5)	0.0315 (7)	0.0152 (5)	0.0011 (5)	0.0052 (4)	−0.0026 (5)
N5	0.0130 (6)	0.0243 (7)	0.0144 (5)	−0.0011 (5)	0.0026 (4)	−0.0022 (5)
N6	0.0146 (6)	0.0263 (7)	0.0136 (5)	−0.0008 (5)	0.0040 (4)	−0.0027 (5)
O4	0.0157 (5)	0.0467 (8)	0.0205 (5)	0.0053 (5)	0.0072 (4)	0.0080 (5)
O5	0.0160 (5)	0.0336 (7)	0.0186 (5)	0.0033 (5)	0.0018 (4)	0.0047 (5)
O6	0.0168 (5)	0.0346 (7)	0.0138 (5)	0.0001 (5)	0.0042 (4)	0.0025 (5)
C11	0.0160 (7)	0.0216 (8)	0.0163 (7)	0.0001 (6)	0.0038 (5)	−0.0005 (6)
C12	0.0151 (7)	0.0221 (8)	0.0166 (7)	0.0003 (6)	0.0035 (5)	0.0039 (6)
C13	0.0139 (7)	0.0253 (9)	0.0184 (7)	0.0010 (6)	0.0048 (5)	0.0017 (6)
C14	0.0173 (7)	0.0211 (8)	0.0145 (6)	0.0016 (6)	0.0057 (5)	0.0030 (6)
N7	0.0149 (6)	0.0278 (8)	0.0141 (6)	0.0025 (5)	0.0028 (4)	0.0031 (5)
N8	0.0165 (6)	0.0299 (8)	0.0139 (6)	0.0022 (6)	0.0047 (5)	0.0056 (6)
C15	0.0156 (7)	0.0250 (8)	0.0177 (7)	0.0004 (6)	0.0050 (5)	0.0013 (6)

C16	0.0149 (7)	0.0200 (8)	0.0190 (7)	0.0005 (6)	0.0044 (5)	0.0002 (6)
C17	0.0140 (7)	0.0250 (8)	0.0187 (7)	0.0000 (6)	0.0052 (5)	0.0007 (6)
C18	0.0162 (7)	0.0207 (8)	0.0170 (7)	−0.0028 (6)	0.0055 (5)	−0.0030 (6)
N1	0.0150 (6)	0.0256 (7)	0.0180 (6)	−0.0013 (5)	0.0059 (5)	−0.0010 (6)
N2	0.0182 (7)	0.0417 (9)	0.0190 (6)	−0.0050 (6)	0.0047 (5)	−0.0073 (6)
C1	0.0186 (7)	0.0216 (8)	0.0190 (7)	−0.0008 (6)	0.0045 (6)	0.0000 (6)
C2	0.0211 (8)	0.0256 (9)	0.0202 (7)	−0.0011 (6)	0.0091 (6)	−0.0025 (7)
C3	0.0165 (7)	0.0230 (8)	0.0283 (8)	−0.0016 (6)	0.0090 (6)	−0.0021 (7)
C4	0.0172 (7)	0.0187 (8)	0.0228 (7)	−0.0017 (6)	0.0010 (6)	−0.0005 (6)
C5	0.0204 (8)	0.0221 (8)	0.0172 (7)	−0.0002 (6)	0.0030 (5)	−0.0018 (6)
N3	0.0155 (6)	0.0226 (7)	0.0193 (6)	0.0012 (5)	0.0061 (5)	0.0011 (5)
N4	0.0171 (7)	0.0359 (9)	0.0206 (6)	0.0036 (6)	0.0040 (5)	0.0044 (6)
C6	0.0181 (7)	0.0214 (8)	0.0189 (7)	0.0005 (6)	0.0046 (5)	−0.0012 (6)
O7	0.0206 (6)	0.0332 (7)	0.0451 (7)	−0.0004 (5)	0.0095 (5)	−0.0077 (6)
C7	0.0195 (8)	0.0368 (10)	0.0211 (7)	0.0022 (7)	0.0080 (6)	0.0024 (7)
C8	0.0168 (8)	0.0342 (10)	0.0295 (8)	0.0017 (7)	0.0070 (6)	0.0007 (8)
C9	0.0189 (8)	0.0225 (8)	0.0245 (8)	0.0024 (6)	0.0000 (6)	0.0009 (7)
C10	0.0240 (8)	0.0225 (8)	0.0187 (7)	0.0006 (6)	0.0040 (6)	0.0021 (6)
O8	0.0204 (6)	0.0406 (8)	0.0424 (7)	0.0008 (6)	0.0046 (5)	0.0027 (6)
O9	0.0387 (8)	0.0413 (8)	0.0380 (7)	0.0019 (6)	0.0135 (6)	0.0028 (6)
O10	0.0297 (7)	0.0445 (9)	0.0434 (7)	0.0032 (6)	0.0039 (6)	−0.0042 (7)

Geometric parameters (Å, º)

Cl1—C4	1.7322 (16)	C1—C2	1.419 (2)
Cl2—C9	1.7249 (17)	N2—H2A	0.8900
O1—C11	1.2309 (19)	C2—C3	1.358 (2)
O2—C12	1.2624 (18)	N2—H2B	0.8900
O3—C14	1.2732 (17)	C3—C4	1.414 (2)
N5—C11	1.3611 (18)	C4—C5	1.349 (2)
N5—C14	1.388 (2)	C2—H2	0.9500
N6—C11	1.3596 (19)	C3—H3	0.9500
N6—C12	1.3899 (19)	N3—C10	1.358 (2)
O4—C15	1.2332 (19)	N3—C6	1.3527 (19)
N5—H5A	0.8700	N4—C6	1.326 (2)
O5—C16	1.2551 (19)	C5—H5	0.9500
O6—C18	1.2741 (17)	N3—H3A	0.8600
N6—H6	0.8900	N4—H4B	0.8900
C12—C13	1.3944 (19)	N4—H4A	0.8700
C13—C14	1.391 (2)	C6—C7	1.408 (2)
N7—C15	1.3616 (19)	C7—C8	1.356 (2)
N7—C18	1.389 (2)	C8—C9	1.409 (2)
N8—C15	1.357 (2)	C9—C10	1.350 (2)
N8—C16	1.393 (2)	C7—H7	0.9500
C13—H13	0.9500	O7—H7C	0.8200
N7—H7A	0.8700	O7—H7B	0.8600
N8—H8A	0.8800	C8—H8	0.9500
C16—C17	1.400 (2)	C10—H10	0.9500

C17—C18	1.382 (2)	O8—H8B	0.8600
N1—C1	1.3503 (19)	O8—H8C	0.8900
N1—C5	1.361 (2)	O9—H9A	0.8600
N2—C1	1.316 (2)	O9—H9B	0.8600
C17—H17	0.9500	O10—H10A	0.8600
N1—H1	0.8700	O10—H10B	0.8700

C11—N5—C14	124.19 (13)	C1—N1—H1	118.00
C11—N6—C12	124.41 (12)	C1—N2—H2A	122.00
C11—N5—H5A	118.00	C1—N2—H2B	121.00
C14—N5—H5A	118.00	C1—C2—C3	120.39 (14)
C11—N6—H6	117.00	H2A—N2—H2B	117.00
C12—N6—H6	119.00	C2—C3—C4	119.29 (14)
O1—C11—N6	122.12 (13)	C3—C4—C5	119.74 (14)
N5—C11—N6	116.26 (13)	Cl1—C4—C3	120.67 (12)
O1—C11—N5	121.62 (13)	Cl1—C4—C5	119.58 (12)
O2—C12—N6	117.54 (12)	N1—C5—C4	120.16 (14)
O2—C12—C13	125.94 (14)	C1—C2—H2	120.00
N6—C12—C13	116.52 (13)	C3—C2—H2	120.00
C12—C13—C14	121.55 (14)	C2—C3—H3	120.00
O3—C14—C13	126.36 (14)	C4—C3—H3	120.00
N5—C14—C13	116.88 (12)	C6—N3—C10	122.84 (14)
O3—C14—N5	116.75 (13)	N1—C5—H5	120.00
C15—N7—C18	124.15 (13)	C4—C5—H5	120.00
C15—N8—C16	124.54 (12)	C10—N3—H3A	120.00
C14—C13—H13	119.00	C6—N3—H3A	117.00
C12—C13—H13	119.00	C6—N4—H4A	121.00
C15—N7—H7A	115.00	H4A—N4—H4B	119.00
C18—N7—H7A	120.00	C6—N4—H4B	120.00
C15—N8—H8A	117.00	N3—C6—C7	117.72 (14)
C16—N8—H8A	119.00	N3—C6—N4	119.42 (14)
O4—C15—N8	122.03 (14)	N4—C6—C7	122.86 (14)
N7—C15—N8	116.25 (13)	C6—C7—C8	120.38 (15)
O4—C15—N7	121.72 (14)	C7—C8—C9	119.60 (16)
O5—C16—N8	117.85 (13)	C8—C9—C10	119.63 (15)
O5—C16—C17	125.85 (14)	Cl2—C9—C8	120.28 (13)
N8—C16—C17	116.29 (13)	Cl2—C9—C10	120.09 (12)
C16—C17—C18	121.61 (14)	N3—C10—C9	119.82 (14)
O6—C18—C17	126.11 (14)	C6—C7—H7	120.00
N7—C18—C17	117.09 (12)	C8—C7—H7	120.00
O6—C18—N7	116.80 (13)	H7B—O7—H7C	106.00
C1—N1—C5	122.67 (13)	C7—C8—H8	120.00
C16—C17—H17	119.00	C9—C8—H8	120.00
C18—C17—H17	119.00	N3—C10—H10	120.00
N1—C1—N2	119.38 (14)	C9—C10—H10	120.00
N1—C1—C2	117.72 (14)	H8B—O8—H8C	103.00
N2—C1—C2	122.90 (14)	H9A—O9—H9B	103.00
C5—N1—H1	119.00	H10A—O10—H10B	109.00

C14—N5—C11—O1	176.94 (14)	C16—C17—C18—O6	−177.54 (14)
C14—N5—C11—N6	−3.5 (2)	C16—C17—C18—N7	2.7 (2)
C11—N5—C14—O3	−177.60 (13)	C5—N1—C1—N2	178.47 (14)
C11—N5—C14—C13	3.6 (2)	C5—N1—C1—C2	−1.7 (2)
C12—N6—C11—O1	179.22 (14)	C1—N1—C5—C4	0.9 (2)
C12—N6—C11—N5	−0.4 (2)	N1—C1—C2—C3	1.2 (2)
C11—N6—C12—O2	−175.86 (14)	N2—C1—C2—C3	−178.93 (14)
C11—N6—C12—C13	3.7 (2)	C1—C2—C3—C4	0.0 (2)
O2—C12—C13—C14	176.03 (14)	C2—C3—C4—Cl1	178.85 (11)
N6—C12—C13—C14	−3.5 (2)	C2—C3—C4—C5	−0.8 (2)
C12—C13—C14—O3	−178.55 (14)	Cl1—C4—C5—N1	−179.26 (11)
C12—C13—C14—N5	0.1 (2)	C3—C4—C5—N1	0.4 (2)
C18—N7—C15—O4	−177.42 (14)	C10—N3—C6—N4	−179.28 (14)
C18—N7—C15—N8	2.4 (2)	C10—N3—C6—C7	0.1 (2)
C15—N7—C18—O6	177.04 (14)	C6—N3—C10—C9	−0.9 (2)
C15—N7—C18—C17	−3.2 (2)	N3—C6—C7—C8	0.5 (3)
C16—N8—C15—O4	178.69 (14)	N4—C6—C7—C8	179.89 (17)
C16—N8—C15—N7	−1.1 (2)	C6—C7—C8—C9	−0.4 (3)
C15—N8—C16—O5	−178.53 (14)	C7—C8—C9—Cl2	178.93 (15)
C15—N8—C16—C17	0.7 (2)	C7—C8—C9—C10	−0.4 (3)
O5—C16—C17—C18	177.64 (14)	Cl2—C9—C10—N3	−178.31 (11)
N8—C16—C17—C18	−1.6 (2)	C8—C9—C10—N3	1.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6	0.87	1.85	2.7207 (18)	176
N2—H2*A*···O1	0.89	2.02	2.7461 (18)	138
N2—H2*B*···O5^{i}	0.89	1.87	2.7610 (17)	180
N3—H3*A*···O3	0.86	1.83	2.6863 (18)	177
N4—H4*A*···O4	0.87	2.00	2.7248 (18)	139
N4—H4*B*···O2^{ii}	0.89	1.97	2.8606 (17)	173
N5—H5*A*···O6	0.87	2.02	2.8786 (17)	173
N6—H6···O4^{i}	0.89	2.03	2.9005 (17)	170
N7—H7*A*···O3	0.87	2.06	2.8985 (17)	163
N8—H8*A*···O1^{ii}	0.88	2.01	2.8762 (16)	172
O7—H7*B*···O5	0.86	1.92	2.7581 (16)	164
O7—H7*C*···O2^{iii}	0.82	2.13	2.9155 (18)	160
O8—H8*B*···O10	0.86	1.89	2.739 (2)	167
O8—H8*C*···O2	0.89	1.91	2.7810 (16)	165
O9—H9*A*···O8	0.86	1.93	2.7864 (18)	168
O10—H10*B*···O7^{iii}	0.87	1.96	2.8287 (18)	175
C10—H10···O7^{iii}	0.95	2.44	3.321 (2)	153

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