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1,4,10,16,21-Pentaaza-7,13,24-triazoniabicyclo-[8.8.8]hexacosane monohydrate clathrate trichloride hexahydrate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 14.2

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

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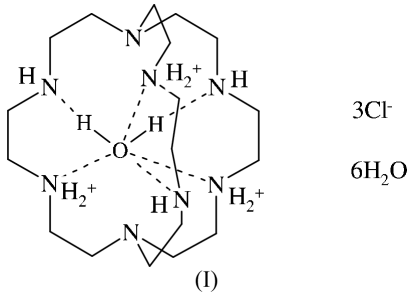
The title compound, $C_{18}H_{59}N_8^{3+}\cdot 3\,Cl^-\cdot 7H_2O$, is a chloride salt of a triprotonated cryptand. One of the water molecules is located inside the crypt and makes six hydrogen bonds to the crypt (two as donor and four as acceptor). The coordination geometry about the bound water molecule is trigonal prismatic.

Comment

The small cryptand 1,4,7,10,13,16,21,24-octaazabicyclo[8.8.8]-hexacosane is a versatile host for cations (Coyle *et al.*, 1997, 2004; McKee *et al.*, 2001) and also, in the hexaprotonated form, for anions (Dietrich *et al.*, 1996; Hossain *et al.*, 2002). In this paper, we report the structure, (I), of a triprotonated form of the cryptand, which encapsulates a neutral water molecule as guest in the cavity. A similar complex formed from the tetraprotonated ligand has been reported recently (Hossain *et al.*, 2002).



(I)

The structure of the $[C_{18}H_{59}N_8(H_2O)]^{3+}$ cation is shown in Fig. 1. H atoms bonded to O or N atoms were located in difference maps. The highest residual peaks are in the region of the chloride anions and not close to the amines; the protonated amines are N3*B*, N4*A* and N4*C*. The water molecule makes six hydrogen bonds to the cryptand (Table 1), two as donor (to N3*A* and N3*C*), and four as acceptor (from the three protonated amines and from N4*B*). These hydrogen bonds cover a wide range [2.680 (2)–3.022 (2) Å]; the shortest are those where the encapsulated water molecule acts as donor to the neutral amines N3*A* and N3*C*, and the longest is that with neutral amine N4*B* as donor. Taken in isolation, it is surprising that the shortest hydrogen bonds are not the charge-assisted interactions involving the protonated amines; however, this is likely to be due to mutual constraints within the extended hydrogen-bond network (Fig. 2). All the secondary amine groups, protonated or not, make two hydrogen bonds, one to the central water molecule (O1) and the other to either a chloride anion or a water molecule of crystallization (Table 1). Within each of the three clefts

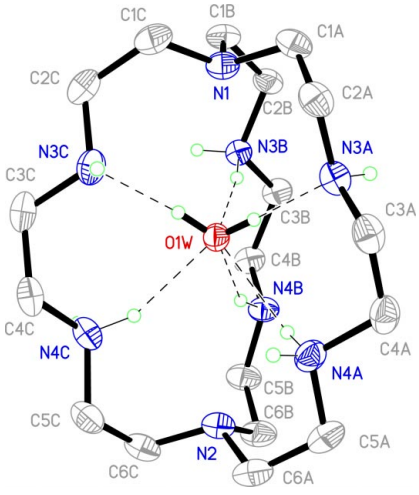


Figure 1
A perspective view of the $[C_{18}H_{59}N_8(H_2O)]^{3+}$ cation. H atoms bonded to C atoms have been omitted for clarity and displacement ellipsoids are drawn at the 50% probability level. The protonated N atoms are N3*B*, N4*A* and N4*C*.

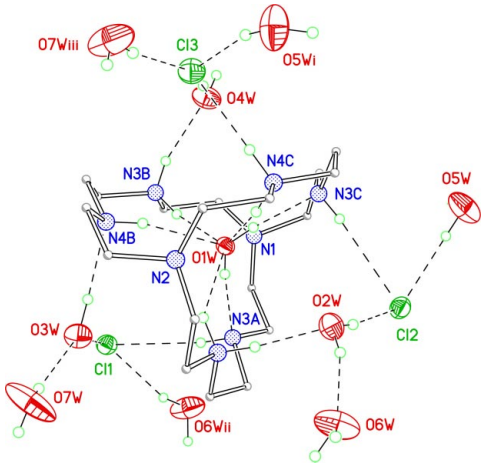


Figure 2
The hydrogen bonding about the cryptate (dashed lines); displacement ellipsoids for the Cl and O atoms are drawn at the 50% probability level. [Symmetry codes: (i) $x-\frac{1}{2}, y, \frac{1}{2}-z$; (ii) $\frac{5}{2}-x, y-\frac{1}{2}, z$; (iii) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$.]

formed by the cryptand is an amine–chloride–water–amine hydrogen-bonded chain and the hydrogen bonding extends in three dimensions throughout the crystal structure.

The coordination geometry at the encapsulated water O atom (O1*W*) is close to trigonal prismatic (Fig. 2). The arrangement of the six hydrogen bonds is remarkably similar to that observed for the encapsulated water molecule in the tetraprotonated analogue $[C_{18}H_{60}N_8(H_2O)]^{4+}$, although in that case (Hossain *et al.*, 2002) the geometry was described as tetrahedral.

Experimental

The unsubstituted cryptand was prepared as described previously (Smith *et al.*, 1993). The protonated product was obtained in an attempt at part-tosylation of the amine N atoms. To one millimole of free cryptand in dichloromethane (50 ml) was added 4-methoxybenzene sulfonyl chloride (4 mmol) and triethylamine (4 mmol). The mixture was stirred overnight and evaporated to dryness. The white solid obtained was redissolved and subjected to alumina chromatography using as eluant dichloromethane/1% MeOH, which had been treated with ammonia gas beforehand. The crystals were obtained on slow evaporation of the eluant.

Crystal data

$C_{18}H_{45}N_8^{3+}\cdot 3Cl^-\cdot 7H_2O$
$M_r = 606.08$
Orthorhombic, *Pbca*
$a = 14.2710\ (6)$ Å
$b = 14.9228\ (6)$ Å
$c = 29.8597\ (12)$ Å
$V = 6359.0\ (4)$ Å^3
$Z = 8$
$D_x = 1.266$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 824 reflections
$\theta = 2.4$–$27.5°$
$\mu = 0.34$ mm^{-1}
$T = 150\ (2)$ K
Block, colourless
0.36 × 0.30 × 0.29 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.826$, $T_{max} = 0.907$
43 473 measured reflections
5604 independent reflections
4323 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0°$
$h = -16 \rightarrow 16$
$k = -17 \rightarrow 17$
$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.102$
$S = 1.03$
5604 reflections
394 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 5.7025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3*A*—H3*A*4···Cl1	0.86 (3)	2.55 (3)	3.4000 (19)	172 (2)
N3*B*—H3*B*3···O4*W*	0.86 (3)	1.96 (3)	2.787 (3)	161 (3)
N3*B*—H3*B*4···O1*W*	0.96 (3)	1.91 (3)	2.859 (2)	170 (2)
N3*C*—H3*C*3···Cl2	0.80 (3)	2.62 (3)	3.420 (2)	173 (3)
N4*A*—H4*A*3···O2*W*	0.89 (3)	1.86 (3)	2.743 (3)	170 (3)
N4*A*—H4*A*4···O1*W*	0.91 (3)	2.19 (3)	2.954 (2)	141 (2)
N4*B*—H4*B*4···O1*W*	0.87 (3)	2.21 (3)	3.022 (2)	154 (2)
N4*C*—H4*C*3···O1*W*	0.90 (3)	2.04 (3)	2.888 (2)	156 (2)
N4*C*—H4*C*4···Cl3	0.93 (3)	2.22 (3)	3.144 (2)	173 (2)
O1*W*—H1*WA*···N3*C*	0.845 (9)	1.842 (11)	2.680 (2)	171 (3)
O1*W*—H1*WB*···N3*A*	0.838 (10)	1.861 (10)	2.696 (2)	174 (3)
O2*W*—H2*WA*···O6*W*	0.833 (10)	1.908 (12)	2.709 (3)	161 (3)
O2*W*—H2*WB*···Cl2	0.831 (9)	2.314 (11)	3.1240 (18)	165 (2)
O3*W*—H3*WA*···Cl1	0.844 (10)	2.313 (10)	3.1531 (19)	174 (2)
O3*W*—H3*WB*···N4*B*	0.851 (10)	1.968 (10)	2.818 (3)	177 (2)
O4*W*—H4*WB*···Cl2^{iv}	0.837 (9)	2.328 (11)	3.1410 (19)	164 (2)
O4*W*—H4*WA*···Cl3	0.835 (9)	2.295 (11)	3.1087 (19)	165 (2)
O5*W*—H5*WA*···Cl3^{v}	0.851 (10)	2.423 (12)	3.257 (2)	166 (2)
O5*W*—H5*WB*···Cl2	0.850 (10)	2.340 (10)	3.187 (2)	174 (2)
O6*W*—H6*WA*···O7*W*^{vi}	0.867 (10)	1.893 (11)	2.734 (3)	163 (2)
O6*W*—H6*WB*···Cl1^{vii}	0.847 (10)	2.319 (11)	3.147 (2)	166 (2)
O7*W*—H7*WA*···Cl3^{viii}	0.832 (10)	2.408 (10)	3.228 (2)	169 (2)
O7*W*—H7*WB*···O3*W*	0.824 (10)	1.941 (11)	2.743 (3)	164 (3)

Symmetry codes: (iv) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+\frac{1}{2}, y, -z+\frac{1}{2}$; (vi) $-x+2, -y+1, -z+1$; (vii) $-x+\frac{5}{2}, y+\frac{1}{2}, z$; (viii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

H atoms bonded to C atoms were placed at calculated positions and refined using a riding model. The C—H distances were constrained to 0.99 Å and were refined with U_{iso}(H) = 1.2U_{eq}(C). H atoms bonded to O or N were located in difference maps and assigned a common U_{iso}(H) value of 0.05 Å^2; their coordinates were refined freely, except that DFIX restraints were applied to the O—H distances [0.84 (2) Å] and H—O—H angles [by restraining the H···H distances to 1.37 (1) Å] (Sheldrick, 2001).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Coyle, J., Downard, A. J., Harding, C. J., Herbst-Irmer, R., McKee, V. & Nelson, J. (2004). *J. Chem. Soc. Dalton Trans.* pp. 2357–2363.

Coyle, J., Drew, M. G. B., Nelson, J. & Town, R. M. (1997). *J. Chem. Soc. Dalton Trans.* pp. 112–116.

Dietrich, B., Dilworth, B., Lehn, J.-M., Souchez, J.-P., Cesario, M., Guilhem, J. & Pascard, C. (1996). *Helv. Chim. Acta,* **79**, 569–586.

Hossain, M. A., Llinares, J. M., Alcock, N. W., Powell, D. & Bowman-James, K. (2002). *J. Supramol. Chem.* **2**, 143–149.

McKee, V., Nelson, J., Speed, D. & Town, R. M. (2001). *J. Chem. Soc. Dalton Trans.* pp. 3641–3646.

Sheldrick, G. M. (2001). *SADABS* and *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.

Smith, P. H., Barr, M. E., Brainard, J. R., Ford, D. K., Frieser, H., Muralidharan, S., Reilly, S. D., Ryan, R. R., Silks, L. A. & Yu, W. (1993). *J. Org. Chem.* **58**, 7939–7941.

supporting information

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4323 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = -16\rightarrow16$
$k = -17\rightarrow17$
$l = -35\rightarrow35$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.102$
$S = 1.03$
5604 reflections
394 parameters
21 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 5.7025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	1.13477 (13)	0.07746 (12)	0.30336 (6)	0.0301 (4)
C1A	1.23306 (15)	0.09037 (16)	0.31705 (8)	0.0345 (5)
H1A1	1.2745	0.0747	0.2916	0.041*
H1A2	1.2475	0.0490	0.3420	0.041*
C2A	1.25378 (16)	0.18517 (15)	0.33170 (8)	0.0336 (5)
H2A1	1.3222	0.1922	0.3360	0.040*
H2A2	1.2339	0.2272	0.3079	0.040*
N3A	1.20534 (13)	0.20831 (12)	0.37365 (6)	0.0287 (4)
C3A	1.23589 (16)	0.29603 (15)	0.39027 (8)	0.0337 (5)
H3A1	1.2257	0.3414	0.3666	0.040*
H3A2	1.3039	0.2937	0.3966	0.040*
C4A	1.18507 (16)	0.32482 (16)	0.43195 (8)	0.0348 (5)
H4A1	1.1897	0.2766	0.4546	0.042*
H4A2	1.2160	0.3788	0.4443	0.042*
N4A	1.08415 (14)	0.34515 (14)	0.42357 (7)	0.0315 (4)
C5A	1.03864 (18)	0.39006 (17)	0.46258 (8)	0.0393 (6)
H5A1	1.0713	0.4471	0.4691	0.047*
H5A2	1.0437	0.3512	0.4893	0.047*
C6A	0.93677 (18)	0.40858 (16)	0.45281 (8)	0.0395 (6)
H6A1	0.9071	0.4349	0.4797	0.047*
H6A2	0.9323	0.4531	0.4283	0.047*
N2	0.88543 (13)	0.32694 (12)	0.43985 (6)	0.0323 (4)
C1B	1.10709 (17)	−0.01725 (15)	0.30762 (8)	0.0336 (5)
H1B1	1.1584	−0.0558	0.2963	0.040*
H1B2	1.0510	−0.0284	0.2889	0.040*
C2B	1.08563 (15)	−0.04221 (14)	0.35556 (7)	0.0299 (5)
H2B1	1.0722	−0.1072	0.3574	0.036*
H2B2	1.1409	−0.0295	0.3746	0.036*
N3B	1.00330 (13)	0.00944 (12)	0.37246 (6)	0.0255 (4)
C3B	0.97707 (16)	−0.01609 (14)	0.41909 (7)	0.0316 (5)
H3B1	1.0315	−0.0062	0.4390	0.038*
H3B2	0.9616	−0.0807	0.4199	0.038*
C4B	0.89437 (16)	0.03693 (14)	0.43656 (7)	0.0314 (5)
H4B1	0.8437	0.0359	0.4139	0.038*
H4B2	0.8703	0.0077	0.4640	0.038*

N4B	0.91826 (13)	0.13050 (12)	0.44682 (6)	0.0283 (4)
C5B	0.84034 (17)	0.17591 (16)	0.46944 (8)	0.0368 (5)
H5B1	0.8251	0.1437	0.4975	0.044*
H5B2	0.7842	0.1744	0.4499	0.044*
C6B	0.86466 (17)	0.27241 (16)	0.48021 (8)	0.0382 (6)
H6B1	0.8116	0.3000	0.4965	0.046*
H6B2	0.9199	0.2734	0.5003	0.046*
C1C	1.11905 (17)	0.11018 (17)	0.25743 (7)	0.0378 (6)
H1C1	1.1369	0.0625	0.2360	0.045*
H1C2	1.1603	0.1623	0.2519	0.045*
C2C	1.01835 (17)	0.13746 (16)	0.24855 (8)	0.0362 (5)
H2C1	1.0108	0.1523	0.2164	0.043*
H2C2	0.9762	0.0867	0.2556	0.043*
N3C	0.99193 (13)	0.21537 (13)	0.27584 (6)	0.0307 (4)
C3C	0.89795 (16)	0.24844 (16)	0.26544 (8)	0.0360 (5)
H3C1	0.8523	0.1993	0.2698	0.043*
H3C2	0.8956	0.2663	0.2335	0.043*
C4C	0.86957 (17)	0.32736 (16)	0.29407 (8)	0.0363 (5)
H4C1	0.9193	0.3735	0.2925	0.044*
H4C2	0.8115	0.3539	0.2816	0.044*
N4C	0.85314 (14)	0.30325 (14)	0.34199 (7)	0.0334 (4)
C5C	0.81360 (18)	0.37909 (17)	0.36888 (8)	0.0415 (6)
H5C1	0.7534	0.3985	0.3556	0.050*
H5C2	0.8574	0.4305	0.3680	0.050*
C6C	0.79792 (17)	0.35121 (17)	0.41688 (8)	0.0396 (6)
H6C1	0.7675	0.4011	0.4332	0.048*
H6C2	0.7548	0.2993	0.4175	0.048*
Cl1	1.23502 (4)	0.04805 (4)	0.453342 (19)	0.03510 (15)
Cl2	1.14708 (4)	0.38858 (4)	0.264667 (19)	0.03771 (15)
Cl3	0.70182 (5)	0.15299 (4)	0.35678 (2)	0.04588 (17)
O1W	1.01762 (10)	0.19982 (10)	0.36441 (5)	0.0255 (3)
O2W	1.04341 (13)	0.45163 (12)	0.35126 (6)	0.0432 (4)
O3W	1.07898 (13)	0.16730 (12)	0.49848 (6)	0.0453 (4)
O4W	0.85106 (13)	0.02680 (13)	0.31466 (6)	0.0483 (5)
O5W	1.07215 (18)	0.32483 (16)	0.16931 (8)	0.0751 (7)
O6W	1.08022 (16)	0.59849 (17)	0.40159 (8)	0.0756 (7)
O7W	1.05245 (18)	0.2838 (2)	0.56842 (8)	0.1029 (11)
H3A4	1.2180 (19)	0.1704 (19)	0.3943 (9)	0.050*
H3B3	0.956 (2)	0.0014 (19)	0.3552 (9)	0.050*
H3B4	1.0153 (19)	0.073 (2)	0.3710 (9)	0.050*
H3C3	1.028 (2)	0.2553 (19)	0.2708 (9)	0.050*
H4A3	1.0781 (19)	0.3785 (19)	0.3989 (10)	0.050*
H4A4	1.0491 (19)	0.2955 (19)	0.4179 (9)	0.050*
H4B4	0.9305 (19)	0.1608 (18)	0.4225 (9)	0.050*
H4C3	0.907 (2)	0.2844 (18)	0.3547 (9)	0.050*
H4C4	0.8114 (19)	0.2555 (19)	0.3447 (9)	0.050*
H1WA	1.0089 (16)	0.1985 (18)	0.3364 (4)	0.050*
H1WB	1.0757 (8)	0.1995 (19)	0.3688 (8)	0.050*

H2WA	1.0517 (19)	0.5031 (10)	0.3613 (8)	0.050*
H2WB	1.0712 (17)	0.4454 (16)	0.3270 (5)	0.050*
H3WA	1.1221 (12)	0.1345 (16)	0.4883 (8)	0.050*
H3WB	1.0317 (11)	0.1555 (18)	0.4821 (8)	0.050*
H4WB	0.8399 (17)	−0.0070 (15)	0.2929 (6)	0.050*
H4WA	0.8038 (12)	0.0557 (16)	0.3226 (8)	0.050*
H5WA	1.0970 (18)	0.2752 (11)	0.1618 (8)	0.050*
H5WB	1.0951 (18)	0.3390 (16)	0.1946 (5)	0.050*
H6WA	1.0473 (15)	0.6399 (14)	0.4145 (8)	0.050*
H6WB	1.1258 (13)	0.5906 (18)	0.4193 (7)	0.050*
H7WA	1.0955 (13)	0.2937 (17)	0.5865 (7)	0.050*
H7WB	1.0673 (17)	0.2436 (14)	0.5508 (7)	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0306 (10)	0.0307 (10)	0.0291 (10)	−0.0004 (8)	0.0069 (8)	−0.0009 (8)
C1A	0.0268 (12)	0.0366 (13)	0.0400 (13)	0.0039 (10)	0.0091 (10)	0.0006 (10)
C2A	0.0245 (11)	0.0360 (13)	0.0403 (13)	−0.0024 (10)	0.0044 (10)	0.0062 (10)
N3A	0.0268 (10)	0.0250 (10)	0.0342 (10)	−0.0025 (8)	−0.0003 (8)	0.0062 (8)
C3A	0.0259 (11)	0.0296 (12)	0.0457 (14)	−0.0074 (10)	−0.0051 (10)	0.0053 (10)
C4A	0.0362 (13)	0.0302 (12)	0.0380 (13)	−0.0054 (10)	−0.0108 (10)	0.0021 (10)
N4A	0.0355 (11)	0.0274 (10)	0.0316 (10)	−0.0038 (9)	−0.0045 (9)	−0.0049 (8)
C5A	0.0526 (15)	0.0337 (13)	0.0315 (12)	−0.0062 (11)	−0.0003 (11)	−0.0099 (10)
C6A	0.0517 (16)	0.0299 (13)	0.0368 (13)	0.0018 (11)	0.0086 (11)	−0.0084 (10)
N2	0.0332 (10)	0.0296 (10)	0.0343 (10)	0.0040 (8)	0.0057 (8)	−0.0037 (8)
C1B	0.0366 (13)	0.0282 (12)	0.0361 (13)	0.0033 (10)	0.0061 (10)	−0.0076 (10)
C2B	0.0291 (12)	0.0237 (11)	0.0368 (12)	0.0031 (9)	0.0015 (10)	−0.0031 (9)
N3B	0.0270 (10)	0.0246 (10)	0.0250 (10)	−0.0008 (8)	0.0011 (7)	0.0004 (7)
C3B	0.0412 (13)	0.0254 (11)	0.0281 (12)	−0.0003 (10)	0.0035 (10)	0.0041 (9)
C4B	0.0368 (13)	0.0283 (12)	0.0293 (12)	−0.0048 (10)	0.0079 (10)	0.0021 (9)
N4B	0.0311 (10)	0.0264 (10)	0.0274 (10)	−0.0003 (8)	0.0090 (8)	0.0012 (8)
C5B	0.0374 (13)	0.0366 (13)	0.0365 (13)	0.0026 (11)	0.0145 (11)	0.0003 (10)
C6B	0.0425 (14)	0.0393 (13)	0.0329 (13)	0.0058 (11)	0.0137 (11)	−0.0030 (10)
C1C	0.0444 (14)	0.0420 (14)	0.0270 (12)	−0.0012 (11)	0.0099 (10)	−0.0019 (10)
C2C	0.0457 (14)	0.0400 (13)	0.0229 (11)	−0.0074 (11)	−0.0002 (10)	−0.0007 (10)
N3C	0.0295 (10)	0.0341 (11)	0.0286 (10)	−0.0066 (8)	−0.0040 (8)	0.0033 (8)
C3C	0.0346 (13)	0.0431 (14)	0.0303 (12)	−0.0074 (11)	−0.0109 (10)	0.0073 (10)
C4C	0.0333 (13)	0.0392 (13)	0.0365 (13)	0.0005 (10)	−0.0099 (10)	0.0110 (11)
N4C	0.0270 (10)	0.0343 (11)	0.0391 (11)	0.0057 (9)	−0.0024 (9)	0.0047 (9)
C5C	0.0373 (14)	0.0363 (14)	0.0509 (15)	0.0130 (11)	−0.0040 (12)	−0.0005 (11)
C6C	0.0335 (13)	0.0377 (14)	0.0477 (15)	0.0116 (11)	0.0071 (11)	−0.0037 (11)
Cl1	0.0375 (3)	0.0296 (3)	0.0382 (3)	0.0002 (2)	−0.0041 (2)	−0.0002 (2)
Cl2	0.0420 (3)	0.0367 (3)	0.0344 (3)	−0.0069 (3)	0.0041 (3)	0.0001 (2)
Cl3	0.0439 (4)	0.0460 (4)	0.0478 (4)	0.0002 (3)	−0.0007 (3)	−0.0037 (3)
O1W	0.0232 (7)	0.0299 (8)	0.0235 (7)	0.0006 (6)	−0.0008 (6)	−0.0002 (6)
O2W	0.0471 (11)	0.0473 (11)	0.0352 (10)	0.0039 (9)	0.0029 (8)	−0.0024 (8)
O3W	0.0444 (11)	0.0413 (10)	0.0502 (11)	0.0038 (9)	−0.0013 (9)	−0.0097 (8)

O4W	0.0420 (11)	0.0548 (12)	0.0481 (11)	0.0077 (9)	−0.0074 (9)	−0.0153 (9)
O5W	0.0931 (18)	0.0682 (15)	0.0641 (15)	0.0318 (13)	−0.0317 (13)	−0.0160 (12)
O6W	0.0529 (13)	0.0848 (17)	0.0891 (17)	0.0239 (12)	−0.0207 (12)	−0.0399 (14)
O7W	0.0830 (18)	0.161 (3)	0.0648 (16)	0.0622 (18)	−0.0364 (13)	−0.0654 (17)

Geometric parameters (Å, º)

N1—C1C	1.473 (3)	N4B—C5B	1.467 (3)
N1—C1B	1.473 (3)	N4B—H4B4	0.87 (3)
N1—C1A	1.474 (3)	C5B—C6B	1.516 (3)
C1A—C2A	1.510 (3)	C5B—H5B1	0.99
C1A—H1A1	0.99	C5B—H5B2	0.99
C1A—H1A2	0.99	C6B—H6B1	0.99
C2A—N3A	1.472 (3)	C6B—H6B2	0.99
C2A—H2A1	0.99	C1C—C2C	1.517 (3)
C2A—H2A2	0.99	C1C—H1C1	0.99
N3A—C3A	1.466 (3)	C1C—H1C2	0.99
N3A—H3A4	0.86 (3)	C2C—N3C	1.469 (3)
C3A—C4A	1.503 (3)	C2C—H2C1	0.99
C3A—H3A1	0.99	C2C—H2C2	0.99
C3A—H3A2	0.99	N3C—C3C	1.463 (3)
C4A—N4A	1.493 (3)	N3C—H3C3	0.80 (3)
C4A—H4A1	0.99	C3C—C4C	1.511 (3)
C4A—H4A2	0.99	C3C—H3C1	0.99
N4A—C5A	1.492 (3)	C3C—H3C2	0.99
N4A—H4A3	0.89 (3)	C4C—N4C	1.494 (3)
N4A—H4A4	0.91 (3)	C4C—H4C1	0.99
C5A—C6A	1.508 (4)	C4C—H4C2	0.99
C5A—H5A1	0.99	N4C—C5C	1.498 (3)
C5A—H5A2	0.99	N4C—H4C3	0.90 (3)
C6A—N2	1.473 (3)	N4C—H4C4	0.93 (3)
C6A—H6A1	0.99	C5C—C6C	1.509 (3)
C6A—H6A2	0.99	C5C—H5C1	0.99
N2—C6C	1.470 (3)	C5C—H5C2	0.99
N2—C6B	1.484 (3)	C6C—H6C1	0.99
C1B—C2B	1.510 (3)	C6C—H6C2	0.99
C1B—H1B1	0.99	O1W—H1WA	0.845 (9)
C1B—H1B2	0.99	O1W—H1WB	0.838 (10)
C2B—N3B	1.493 (3)	O2W—H2WA	0.833 (10)
C2B—H2B1	0.99	O2W—H2WB	0.831 (9)
C2B—H2B2	0.99	O3W—H3WA	0.844 (10)
N3B—C3B	1.491 (3)	O3W—H3WB	0.851 (10)
N3B—H3B3	0.86 (3)	O4W—H4WB	0.837 (9)
N3B—H3B4	0.96 (3)	O4W—H4WA	0.835 (9)
C3B—C4B	1.514 (3)	O5W—H5WA	0.851 (10)
C3B—H3B1	0.99	O5W—H5WB	0.850 (10)
C3B—H3B2	0.99	O6W—H6WA	0.867 (10)
C4B—N4B	1.470 (3)	O6W—H6WB	0.847 (10)

C4B—H4B1	0.99	O7W—H7WA	0.832 (10)
C4B—H4B2	0.99	O7W—H7WB	0.824 (10)
C1C—N1—C1B	110.95 (18)	H3B1—C3B—H3B2	107.8
C1C—N1—C1A	111.10 (18)	N4B—C4B—C3B	112.81 (18)
C1B—N1—C1A	110.90 (18)	N4B—C4B—H4B1	109.0
N1—C1A—C2A	112.90 (18)	C3B—C4B—H4B1	109.0
N1—C1A—H1A1	109.0	N4B—C4B—H4B2	109.0
C2A—C1A—H1A1	109.0	C3B—C4B—H4B2	109.0
N1—C1A—H1A2	109.0	H4B1—C4B—H4B2	107.8
C2A—C1A—H1A2	109.0	C5B—N4B—C4B	111.03 (18)
H1A1—C1A—H1A2	107.8	C5B—N4B—H4B4	107.1 (18)
N3A—C2A—C1A	111.99 (18)	C4B—N4B—H4B4	111.5 (18)
N3A—C2A—H2A1	109.2	N4B—C5B—C6B	111.28 (19)
C1A—C2A—H2A1	109.2	N4B—C5B—H5B1	109.4
N3A—C2A—H2A2	109.2	C6B—C5B—H5B1	109.4
C1A—C2A—H2A2	109.2	N4B—C5B—H5B2	109.4
H2A1—C2A—H2A2	107.9	C6B—C5B—H5B2	109.4
C3A—N3A—C2A	110.97 (17)	H5B1—C5B—H5B2	108.0
C3A—N3A—H3A4	106.5 (19)	N2—C6B—C5B	113.23 (19)
C2A—N3A—H3A4	111.0 (19)	N2—C6B—H6B1	108.9
N3A—C3A—C4A	113.07 (18)	C5B—C6B—H6B1	108.9
N3A—C3A—H3A1	109.0	N2—C6B—H6B2	108.9
C4A—C3A—H3A1	109.0	C5B—C6B—H6B2	108.9
N3A—C3A—H3A2	109.0	H6B1—C6B—H6B2	107.7
C4A—C3A—H3A2	109.0	N1—C1C—C2C	113.33 (18)
H3A1—C3A—H3A2	107.8	N1—C1C—H1C1	108.9
N4A—C4A—C3A	112.63 (18)	C2C—C1C—H1C1	108.9
N4A—C4A—H4A1	109.1	N1—C1C—H1C2	108.9
C3A—C4A—H4A1	109.1	C2C—C1C—H1C2	108.9
N4A—C4A—H4A2	109.1	H1C1—C1C—H1C2	107.7
C3A—C4A—H4A2	109.1	N3C—C2C—C1C	111.01 (19)
H4A1—C4A—H4A2	107.8	N3C—C2C—H2C1	109.4
C5A—N4A—C4A	112.36 (18)	C1C—C2C—H2C1	109.4
C5A—N4A—H4A3	110.6 (18)	N3C—C2C—H2C2	109.4
C4A—N4A—H4A3	110.1 (18)	C1C—C2C—H2C2	109.4
C5A—N4A—H4A4	105.8 (17)	H2C1—C2C—H2C2	108.0
C4A—N4A—H4A4	113.3 (17)	C3C—N3C—C2C	112.62 (18)
H4A3—N4A—H4A4	104 (2)	C3C—N3C—H3C3	107 (2)
N4A—C5A—C6A	110.53 (19)	C2C—N3C—H3C3	108 (2)
N4A—C5A—H5A1	109.5	N3C—C3C—C4C	112.88 (18)
C6A—C5A—H5A1	109.5	N3C—C3C—H3C1	109.0
N4A—C5A—H5A2	109.5	C4C—C3C—H3C1	109.0
C6A—C5A—H5A2	109.5	N3C—C3C—H3C2	109.0
H5A1—C5A—H5A2	108.1	C4C—C3C—H3C2	109.0
N2—C6A—C5A	112.25 (19)	H3C1—C3C—H3C2	107.8
N2—C6A—H6A1	109.2	N4C—C4C—C3C	113.35 (19)
C5A—C6A—H6A1	109.2	N4C—C4C—H4C1	108.9

N2—C6A—H6A2	109.2	C3C—C4C—H4C1	108.9
C5A—C6A—H6A2	109.2	N4C—C4C—H4C2	108.9
H6A1—C6A—H6A2	107.9	C3C—C4C—H4C2	108.9
C6C—N2—C6A	109.95 (19)	H4C1—C4C—H4C2	107.7
C6C—N2—C6B	110.14 (19)	C4C—N4C—C5C	112.98 (19)
C6A—N2—C6B	109.85 (18)	C4C—N4C—H4C3	110.0 (17)
N1—C1B—C2B	111.89 (18)	C5C—N4C—H4C3	109.4 (17)
N1—C1B—H1B1	109.2	C4C—N4C—H4C4	111.5 (17)
C2B—C1B—H1B1	109.2	C5C—N4C—H4C4	106.8 (17)
N1—C1B—H1B2	109.2	H4C3—N4C—H4C4	106 (2)
C2B—C1B—H1B2	109.2	N4C—C5C—C6C	110.89 (19)
H1B1—C1B—H1B2	107.9	N4C—C5C—H5C1	109.5
N3B—C2B—C1B	110.65 (18)	C6C—C5C—H5C1	109.5
N3B—C2B—H2B1	109.5	N4C—C5C—H5C2	109.5
C1B—C2B—H2B1	109.5	C6C—C5C—H5C2	109.5
N3B—C2B—H2B2	109.5	H5C1—C5C—H5C2	108.1
C1B—C2B—H2B2	109.5	N2—C6C—C5C	112.66 (19)
H2B1—C2B—H2B2	108.1	N2—C6C—H6C1	109.1
C3B—N3B—C2B	112.42 (17)	C5C—C6C—H6C1	109.1
C3B—N3B—H3B3	109.2 (18)	N2—C6C—H6C2	109.1
C2B—N3B—H3B3	110.0 (18)	C5C—C6C—H6C2	109.1
C3B—N3B—H3B4	109.8 (16)	H6C1—C6C—H6C2	107.8
C2B—N3B—H3B4	110.6 (16)	H1WA—O1W—H1WB	107.5 (19)
H3B3—N3B—H3B4	104 (2)	H2WA—O2W—H2WB	110 (2)
N3B—C3B—C4B	112.58 (18)	H3WA—O3W—H3WB	104.6 (19)
N3B—C3B—H3B1	109.1	H4WB—O4W—H4WA	112 (2)
C4B—C3B—H3B1	109.1	H5WA—O5W—H5WB	106.9 (19)
N3B—C3B—H3B2	109.1	H6WA—O6W—H6WB	103.7 (18)
C4B—C3B—H3B2	109.1	H7WA—O7W—H7WB	111 (2)

C1C—N1—C1A—C2A	79.9 (2)	C3B—C4B—N4B—C5B	−172.04 (19)
C1B—N1—C1A—C2A	−156.24 (18)	C4B—N4B—C5B—C6B	179.86 (19)
N1—C1A—C2A—N3A	67.6 (2)	C6C—N2—C6B—C5B	76.9 (2)
C1A—C2A—N3A—C3A	172.63 (19)	C6A—N2—C6B—C5B	−161.8 (2)
C2A—N3A—C3A—C4A	177.26 (18)	N4B—C5B—C6B—N2	61.0 (3)
N3A—C3A—C4A—N4A	−68.4 (2)	C1B—N1—C1C—C2C	81.7 (2)
C3A—C4A—N4A—C5A	−169.05 (19)	C1A—N1—C1C—C2C	−154.39 (19)
C4A—N4A—C5A—C6A	−179.51 (19)	N1—C1C—C2C—N3C	65.4 (3)
N4A—C5A—C6A—N2	55.4 (3)	C1C—C2C—N3C—C3C	175.10 (18)
C5A—C6A—N2—C6C	−161.48 (19)	C2C—N3C—C3C—C4C	178.80 (19)
C5A—C6A—N2—C6B	77.2 (2)	N3C—C3C—C4C—N4C	−69.6 (3)
C1C—N1—C1B—C2B	−157.38 (19)	C3C—C4C—N4C—C5C	−172.82 (19)
C1A—N1—C1B—C2B	78.6 (2)	C4C—N4C—C5C—C6C	179.7 (2)
N1—C1B—C2B—N3B	63.6 (2)	C6A—N2—C6C—C5C	77.8 (2)
C1B—C2B—N3B—C3B	177.49 (18)	C6B—N2—C6C—C5C	−161.0 (2)
C2B—N3B—C3B—C4B	179.29 (18)	N4C—C5C—C6C—N2	62.9 (3)
N3B—C3B—C4B—N4B	−72.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3*A*—H3*A*4···Cl1	0.86 (3)	2.55 (3)	3.4000 (19)	172 (2)
N3*B*—H3*B*3···O4*W*	0.86 (3)	1.96 (3)	2.787 (3)	161 (3)
N3*B*—H3*B*4···O1*W*	0.96 (3)	1.91 (3)	2.859 (2)	170 (2)
N3*C*—H3*C*3···Cl2	0.80 (3)	2.62 (3)	3.420 (2)	173 (3)
N4*A*—H4*A*3···O2*W*	0.89 (3)	1.86 (3)	2.743 (3)	170 (3)
N4*A*—H4*A*4···O1*W*	0.91 (3)	2.19 (3)	2.954 (2)	141 (2)
N4*B*—H4*B*4···O1*W*	0.87 (3)	2.21 (3)	3.022 (2)	154 (2)
N4*C*—H4*C*3···O1*W*	0.90 (3)	2.04 (3)	2.888 (2)	156 (2)
N4*C*—H4*C*4···Cl3	0.93 (3)	2.22 (3)	3.144 (2)	173 (2)
O1*W*—H1*WA*···N3*C*	0.85 (1)	1.84 (1)	2.680 (2)	171 (3)
O1*W*—H1*WB*···N3*A*	0.84 (1)	1.86 (1)	2.696 (2)	174 (3)
O2*W*—H2*WA*···O6*W*	0.83 (1)	1.91 (1)	2.709 (3)	161 (3)
O2*W*—H2*WB*···Cl2	0.83 (1)	2.31 (1)	3.1240 (18)	165 (2)
O3*W*—H3*WA*···Cl1	0.84 (1)	2.31 (1)	3.1531 (19)	174 (2)
O3*W*—H3*WB*···N4*B*	0.85 (1)	1.97 (1)	2.818 (3)	177 (2)
O4*W*—H4*WB*···Cl2^{i}	0.84 (1)	2.33 (1)	3.1410 (19)	164 (2)
O4*W*—H4*WA*···Cl3	0.84 (1)	2.30 (1)	3.1087 (19)	165 (2)
O5*W*—H5*WA*···Cl3^{ii}	0.85 (1)	2.42 (1)	3.257 (2)	166 (2)
O5*W*—H5*WB*···Cl2	0.85 (1)	2.34 (1)	3.187 (2)	174 (2)
O6*W*—H6*WA*···O7*W*^{iii}	0.87 (1)	1.89 (1)	2.734 (3)	163 (2)
O6*W*—H6*WB*···Cl1^{iv}	0.85 (1)	2.32 (1)	3.147 (2)	166 (2)
O7*W*—H7*WA*···Cl3^{v}	0.83 (1)	2.41 (1)	3.228 (2)	169 (2)
O7*W*—H7*WB*···O3*W*	0.82 (1)	1.94 (1)	2.743 (3)	164 (3)

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+1/2; (ii) *x*+1/2, *y*, −*z*+1/2; (iii) −*x*+2, −*y*+1, −*z*+1; (iv) −*x*+5/2, *y*+1/2, *z*; (v) *x*+1/2, −*y*+1/2, −*z*+1.