

# Bis(2-amino-3,5-dichloropyridinium) hexachloridostannate(IV) dihydrate

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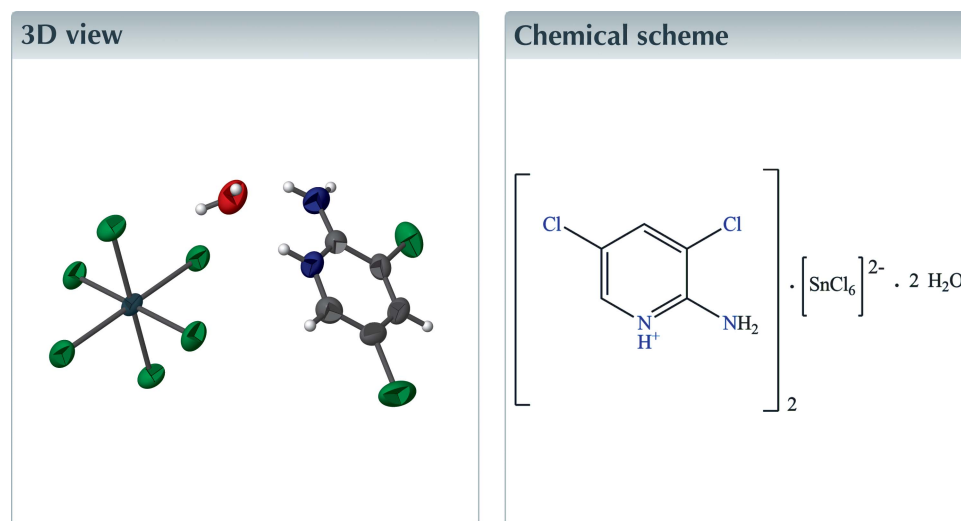
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Keywords: crystal structure; hexachloridostannate(IV); pyridinium; X-ray diffraction.

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Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

The title hybrid compound,  $(C_5H_5N_2Cl_2)_2[SnCl_6] \cdot 2H_2O$ , was synthesized and its structure was identified by single-crystal X-ray diffraction. The structure is non-polymeric (0D) in terms of containing isolated  $[SnCl_6]^{2-}$  polyhedra. The special position (0,0,0) of the  $Sn^{IV}$  atom in the crystal structure gives rise to a stacking structure with alternating cationic and anionic layers parallel to (001). The water molecules are intercalated between these layers, which are linked by cation–anion hydrogen bonds and dominant non-covalent interactions. The stability of the three-dimensional network for this compound is also discussed.



## Structure description

Bis(2-amino-3,5-dichloropyridinium) hexachloridostannate(IV) dihydrate,  $(C_5H_5N_2Cl_2)_2[SnCl_6] \cdot 2H_2O$ , crystallizes in the triclinic space group  $P\bar{1}$  (Fig. 1). The tin(IV) atom is hexacoordinated by chlorine atoms, generating a weakly distorted octahedron. The Sn–Cl bond lengths range from 2.4162 (5) to 2.4389 (5) Å while the Cl–Sn–Cl angles have a deviation of about  $\pm 1^\circ$  [89.277 (19)–90.723 (19)°], see Table 1. These values are comparable to those of the same anion associated with other types of cations (Bouchene *et al.*, 2018). The absence of larger distortions can probably be attributed to the fact that the hexachloridostannate(IV) anions are free, *i.e.* none of the chloride ions are bridging, although they do accept N–H...Cl, O–H...Cl and C–H...Cl hydrogen bonds (Table 2).

In the cation, we note an increase in C1–C2 and C2–C14 bond lengths and a decrease in C1–N2 bond lengths (Table 1). This phenomenon is due to resonance-assisted hydrogen bonding, commonly observed for this kind of molecule (Bertolasi *et al.*, 1998). The C–N–C angle is 124.32 (17)°. This large angle can be attributed to the protonation of the N atom. These values are comparable with those of the same cation associated with other types of anions (Ghallab *et al.*, 2020). The intermolecular interactions in the title

Table 1

Selected geometric parameters (Å, °).

Sn1—Cl1	2.4162 (5)	C1—N2	1.315 (3)
Sn1—Cl2	2.4389 (5)	C2—C3	1.356 (3)
Sn1—Cl3	2.4253 (5)	C2—Cl4	1.713 (2)
N1—C1	1.345 (3)	C3—C4	1.393 (3)
N1—C5	1.350 (3)	C4—C5	1.348 (3)
C1—C2	1.417 (3)	C4—Cl5	1.726 (2)
Cl1—Sn1—Cl2	90.722 (19)	N2—C1—N1	119.49 (18)
Cl1 <sup>i</sup> —Sn1—Cl2	89.278 (19)	N2—C1—C2	124.50 (19)
Cl1—Sn1—Cl2 <sup>i</sup>	89.277 (19)	C1—C2—Cl4	117.52 (16)
Cl1—Sn1—Cl3	89.906 (19)	C3—C2—C1	120.82 (18)
Cl1—Sn1—Cl3 <sup>i</sup>	90.093 (19)	C3—C2—Cl4	121.66 (15)
Cl1 <sup>i</sup> —Sn1—Cl3	90.093 (19)	C2—C3—C4	119.71 (18)
Cl3 <sup>i</sup> —Sn1—Cl2	89.81 (2)	C3—C4—Cl5	120.22 (16)
Cl3—Sn1—Cl2	90.19 (2)	C5—C4—C3	119.70 (19)
Cl3—Sn1—Cl2 <sup>i</sup>	89.81 (2)	C5—C4—Cl5	120.08 (18)
C1—N1—C5	124.32 (17)	C4—C5—N1	119.4 (2)
N1—C1—C2	116.00 (18)		

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

compound were analysed using *PLATON* (Spek, 2020), which shows that the structural cohesion in the crystal structure is ensured by N—H···O, N—H···Cl, O—H···Cl and C—H···Cl hydrogen bonds (Fig. 2a, Table 2). We also note the presence of Cl···Cl halogen bonds (Fig. 2a), and of  $\pi$ -stacking interactions between centrosymmetrically related aromatic rings of the cations as well as Y—X···Cg interactions (Fig. 2b).

### Synthesis and crystallization

Tin(II) chloride dihydrate (2.25 mmol) was mixed with 2-amino-3,5-dichloropyridine (3.3 mmol) in 1:2 molar ratio and a few drops of hydrochloric acid in an aliquot of distilled water were added. After stirring, the mixture was refluxed for one h at 343 K. After two weeks of slow solvent evaporation, single crystals suitable for X-ray analysis were obtained.

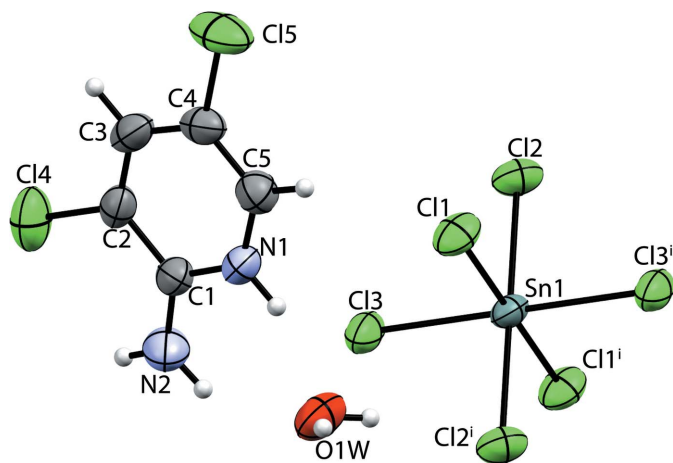


Figure 1

The molecular components in the crystal structure of the title compound, showing displacement ellipsoids at the 30% probability level [symmetry code: (i)  $-x + 2, -y, -z$ ].

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1···O1W	0.86	1.86	2.685 (2)	160
O1W—H1WA···Cl1 <sup>ii</sup>	0.85	2.67	3.296 (2)	131
O1W—H1WB···Cl2	0.85	2.47	3.301 (2)	168
N2—H2A···Cl3 <sup>iii</sup>	0.86	2.78	3.381 (2)	129
N2—H2A···O1W	0.86	2.38	3.065 (3)	137
N2—H2B···Cl2 <sup>iv</sup>	0.86	2.67	3.435 (2)	149
C3—H3···Cl3 <sup>v</sup>	0.93	2.77	3.695 (2)	177
C5—H5···Cl2 <sup>ii</sup>	0.93	2.80	3.615 (2)	147

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

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

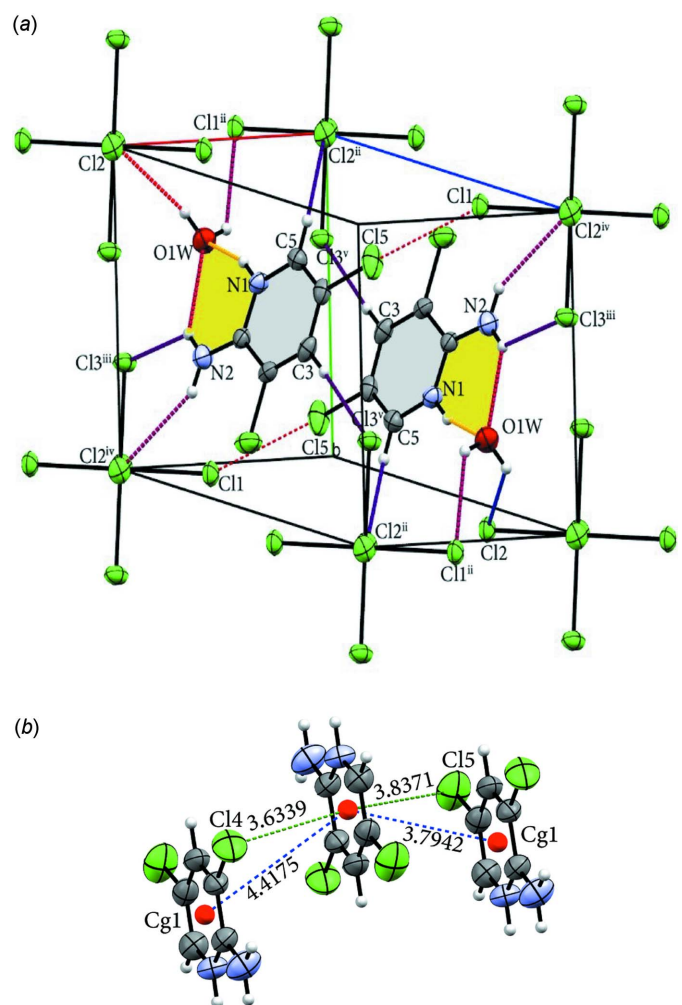


Figure 2

(a) Hydrogen bonds [yellow, purple and violet dashed lines; symmetry codes: (ii)  $-x + 1, -y, -z$ ; (iii)  $x, y + 1, z$ ; (iv)  $-x + 2, -y + 1, -z$ ; (v)  $x, y + 1, z + 1$ ] and halogen bonds (red dashed lines) in the title compound. (b) A view of the  $\pi$ -stacking interactions [blue dashed lines; symmetry codes: (i)  $1 - x, 1 - y, 1 - z$ ; (ii)  $2 - x, 1 - y, 1 - z$ ] and C—Cl···Cg [green dashed lines; symmetry operations: (i)  $2 - x, 1 - y, 1 - z$ ; (ii)  $1 - x, 1 - y, 1 - z$ ] interactions.

Table 3

Experimental details.

Crystal data	
Chemical formula	(C <sub>5</sub> H <sub>5</sub> Cl <sub>2</sub> N <sub>2</sub> ) <sub>2</sub> [SnCl <sub>6</sub> ]·2H <sub>2</sub> O
<i>M<sub>r</sub></i>	695.44
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.4624 (2), 8.4715 (2), 10.1324 (2)
$\alpha$ , $\beta$ , $\gamma$ (°)	101.434 (1), 90.043 (1), 107.554 (1)
<i>V</i> (Å <sup>3</sup> )	597.34 (2)
<i>Z</i>	1
Radiation type	Mo <i>K</i> α
$\mu$ (mm <sup>-1</sup> )	2.20
Crystal size (mm)	0.17 × 0.13 × 0.11
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2016)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.716, 0.785
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	13446, 3617, 3320
<i>R<sub>int</sub></i>	0.017
(sin $\theta$ /λ) <sub>max</sub> (Å <sup>-1</sup> )	0.714
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.025, 0.057, 1.02
No. of reflections	3617
No. of parameters	125
H-atom treatment	H-atom parameters constrained
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )	0.43, -0.55

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

## Acknowledgements

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## full crystallographic data

*IUCrData* (2022). 7, x220191 [https://doi.org/10.1107/S2414314622001912]

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*Crystal data*

$(C_5H_5Cl_2N_2)_2[SnCl_6] \cdot 2H_2O$

$M_r = 695.44$

Triclinic,  $P\bar{1}$

$a = 7.4624$  (2) Å

$b = 8.4715$  (2) Å

$c = 10.1324$  (2) Å

$\alpha = 101.434$  (1)°

$\beta = 90.043$  (1)°

$\gamma = 107.554$  (1)°

$V = 597.34$  (2) Å<sup>3</sup>

$Z = 1$

$F(000) = 338$

$D_x = 1.933$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 8759 reflections

$\theta = 2.9$ – $30.9$ °

$\mu = 2.20$  mm<sup>-1</sup>

$T = 296$  K

Block, clear light white

$0.17 \times 0.13 \times 0.11$  mm

*Data collection*

Bruker APEXII CCD

diffractometer

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

$T_{\min} = 0.716$ ,  $T_{\max} = 0.785$

13446 measured reflections

3617 independent reflections

3320 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 30.5$ °,  $\theta_{\min} = 3.6$ °

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 12$

$l = -14 \rightarrow 14$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.057$

$S = 1.02$

3617 reflections

125 parameters

0 restraints

Primary atom site location: iterative

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0221P)^2 + 0.2999P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.43$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.55$  e Å<sup>-3</sup>

Extinction correction: SHELXL (Sheldrick

2015),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0164 (12)

*Special details*

**Refinement.** Approximate positions for all H atoms were first obtained from difference Fourier maps. H atoms were then placed in idealized positions and refined using the riding-atom approximation: C—H = 0.93 Å and N—H = 0.86 Å, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ . H atoms of the water molecule were located in a difference Fourier map and the water molecule geometry was eventually idealized, with O—H = 0.85 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	1.000000	0.000000	0.000000	0.03672 (7)
Cl1	0.75548 (7)	-0.01363 (7)	0.15744 (5)	0.05267 (13)
Cl2	0.79968 (8)	0.04433 (7)	-0.17040 (5)	0.05363 (13)
Cl3	0.87570 (9)	-0.30370 (6)	-0.08206 (5)	0.05831 (15)
O1W	0.6070 (3)	0.3039 (2)	0.02149 (16)	0.0668 (5)
H1WA	0.488075	0.275909	0.008232	0.100*
H1WB	0.646535	0.225509	-0.021598	0.100*
N1	0.6643 (3)	0.3678 (2)	0.29104 (16)	0.0475 (4)
H1	0.636615	0.323122	0.206756	0.057*
C1	0.7864 (3)	0.5252 (2)	0.32410 (19)	0.0423 (4)
C2	0.8268 (3)	0.5946 (2)	0.4640 (2)	0.0440 (4)
C3	0.7460 (3)	0.5045 (3)	0.55683 (19)	0.0500 (5)
H3	0.773367	0.551264	0.648414	0.060*
C4	0.6218 (3)	0.3417 (3)	0.5144 (2)	0.0485 (5)
C5	0.5824 (3)	0.2753 (3)	0.3814 (2)	0.0504 (5)
H5	0.499435	0.166763	0.352231	0.061*
N2	0.8609 (3)	0.6033 (3)	0.2280 (2)	0.0635 (5)
H2A	0.831417	0.553756	0.144754	0.076*
H2B	0.939027	0.703933	0.248386	0.076*
Cl4	0.97856 (10)	0.79620 (8)	0.51057 (8)	0.0752 (2)
Cl5	0.51633 (12)	0.22629 (11)	0.63144 (8)	0.0814 (2)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.04012 (10)	0.03823 (10)	0.02500 (8)	0.00279 (7)	0.00185 (6)	0.00539 (6)
Cl1	0.0505 (3)	0.0630 (3)	0.0385 (2)	0.0095 (2)	0.0146 (2)	0.0090 (2)
Cl2	0.0537 (3)	0.0674 (3)	0.0377 (2)	0.0142 (2)	-0.0065 (2)	0.0131 (2)
Cl3	0.0850 (4)	0.0373 (2)	0.0378 (2)	0.0008 (2)	0.0014 (2)	0.00182 (18)
O1W	0.0687 (10)	0.0721 (11)	0.0450 (8)	0.0080 (8)	-0.0052 (7)	0.0001 (8)
N1	0.0607 (10)	0.0453 (9)	0.0327 (7)	0.0142 (8)	0.0013 (7)	0.0026 (6)
C1	0.0504 (10)	0.0414 (9)	0.0383 (9)	0.0185 (8)	0.0079 (8)	0.0083 (7)
C2	0.0461 (10)	0.0422 (9)	0.0421 (9)	0.0178 (8)	-0.0021 (8)	-0.0014 (8)
C3	0.0571 (12)	0.0667 (13)	0.0328 (8)	0.0331 (10)	-0.0002 (8)	0.0038 (8)
C4	0.0546 (12)	0.0595 (12)	0.0439 (10)	0.0288 (10)	0.0119 (9)	0.0216 (9)
C5	0.0538 (12)	0.0443 (10)	0.0524 (11)	0.0132 (9)	0.0055 (9)	0.0112 (9)
N2	0.0852 (14)	0.0542 (11)	0.0511 (10)	0.0171 (10)	0.0201 (10)	0.0175 (9)
Cl4	0.0688 (4)	0.0496 (3)	0.0897 (5)	0.0096 (3)	-0.0104 (3)	-0.0128 (3)
Cl5	0.0971 (5)	0.1009 (5)	0.0742 (4)	0.0474 (4)	0.0359 (4)	0.0562 (4)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Sn1—Cl1	2.4162 (5)	C1—C2	1.417 (3)
Sn1—Cl1 <sup>i</sup>	2.4162 (5)	C1—N2	1.315 (3)
Sn1—Cl2	2.4389 (5)	C2—C3	1.356 (3)

Sn1—C12 <sup>i</sup>	2.4389 (5)	C2—C14	1.713 (2)
Sn1—C13	2.4253 (5)	C3—H3	0.9300
Sn1—C13 <sup>i</sup>	2.4253 (5)	C3—C4	1.393 (3)
O1W—H1WA	0.8499	C4—C5	1.348 (3)
O1W—H1WB	0.8496	C4—C15	1.726 (2)
N1—H1	0.8600	C5—H5	0.9300
N1—C1	1.345 (3)	N2—H2A	0.8600
N1—C5	1.350 (3)	N2—H2B	0.8600
Cl1—Sn1—Cl1 <sup>i</sup>	180.0	N1—C1—C2	116.00 (18)
Cl1—Sn1—Cl2	90.722 (19)	N2—C1—N1	119.49 (18)
Cl1 <sup>i</sup> —Sn1—Cl2	89.278 (19)	N2—C1—C2	124.50 (19)
Cl1—Sn1—Cl2 <sup>i</sup>	89.277 (19)	C1—C2—C14	117.52 (16)
Cl1 <sup>i</sup> —Sn1—Cl2 <sup>i</sup>	90.723 (19)	C3—C2—C1	120.82 (18)
Cl1—Sn1—Cl3	89.906 (19)	C3—C2—C14	121.66 (15)
Cl1—Sn1—Cl3 <sup>i</sup>	90.093 (19)	C2—C3—H3	120.1
Cl1 <sup>i</sup> —Sn1—Cl3 <sup>i</sup>	89.907 (19)	C2—C3—C4	119.71 (18)
Cl1 <sup>i</sup> —Sn1—Cl3	90.093 (19)	C4—C3—H3	120.1
Cl2 <sup>i</sup> —Sn1—Cl2	180.0	C3—C4—C15	120.22 (16)
Cl3 <sup>i</sup> —Sn1—Cl2	89.81 (2)	C5—C4—C3	119.70 (19)
Cl3 <sup>i</sup> —Sn1—Cl2 <sup>i</sup>	90.19 (2)	C5—C4—C15	120.08 (18)
Cl3—Sn1—Cl2	90.19 (2)	N1—C5—H5	120.3
Cl3—Sn1—Cl2 <sup>i</sup>	89.81 (2)	C4—C5—N1	119.4 (2)
Cl3—Sn1—Cl3 <sup>i</sup>	180.0	C4—C5—H5	120.3
H1WA—O1W—H1WB	109.5	C1—N2—H2A	120.0
C1—N1—H1	117.8	C1—N2—H2B	120.0
C1—N1—C5	124.32 (17)	H2A—N2—H2B	120.0
C5—N1—H1	117.8		
N1—C1—C2—C3	0.4 (3)	C5—N1—C1—C2	-0.6 (3)
N1—C1—C2—C14	-178.93 (15)	C5—N1—C1—N2	178.8 (2)
C1—N1—C5—C4	0.3 (3)	N2—C1—C2—C3	-179.0 (2)
C1—C2—C3—C4	0.0 (3)	N2—C1—C2—C14	1.7 (3)
C2—C3—C4—C5	-0.3 (3)	Cl4—C2—C3—C4	179.31 (16)
C2—C3—C4—C15	-179.42 (16)	Cl5—C4—C5—N1	179.25 (16)
C3—C4—C5—N1	0.1 (3)		

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

#### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 $\cdots$ O1W	0.86	1.86	2.685 (2)	160
O1W—H1WA $\cdots$ Cl1 <sup>ii</sup>	0.85	2.67	3.296 (2)	131
O1W—H1WB $\cdots$ Cl2	0.85	2.47	3.301 (2)	168
N2—H2A $\cdots$ Cl3 <sup>iii</sup>	0.86	2.78	3.381 (2)	129
N2—H2A $\cdots$ O1W	0.86	2.38	3.065 (3)	137
N2—H2B $\cdots$ Cl4	0.86	2.61	2.986 (2)	108

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N2—H2B···Cl2 <sup>iv</sup>	0.86	2.67	3.435 (2)	149
C3—H3···Cl3 <sup>v</sup>	0.93	2.77	3.695 (2)	177
C5—H5···Cl2 <sup>ii</sup>	0.93	2.80	3.615 (2)	147

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