Diacridinium hexachloridoplatinate(IV) dihydrate

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The asymmetric unit of the title compound, $(C_{13}H_{10}N)_2$-$[PtCl_6]\cdot 2H_2O$, contains a protonated acridine cation, one half of a $[PtCl_6]^{2-}$ dianionic complex and a solvent water molecule. The octahedral $[PtCl_6]^{2-}$ dianion is located on an inversion centre. π–π interactions between neighboring acridinium cations produce stacks along the a axis; the shortest distance between the centroids of the six-membered rings within the cations is 3.553 (9) Å. In the crystal, two independent intermolecular O—H···Cl hydrogen bonds, both involving the same Cl atom of the anion as acceptor, give rise to chains also running along the a axis; in addition each water molecule, as a hydrogen-bond acceptor, is linked to the acridinium N—H group.

Related literature

For related acridinium salts, see: Hafiz (2006); Veldhuizen *et al.* (1997). For the crystal structures of $[PtCl_6]^{2-}$ complexes, see: Karaca *et al.* (2009); Yousefi *et al.* (2007); Zordan & Brammer (2004).
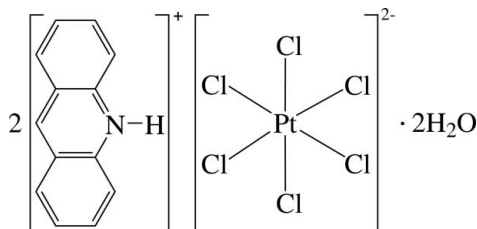
Experimental

Crystal data

$(C_{13}H_{10}N)_2[PtCl_6]\cdot 2H_2O$
$M_r = 804.26$

Triclinic, $P\bar{1}$
$a = 7.4781$ (8) Å
$b = 9.8973$ (10) Å
$c = 10.7226$ (12) Å
$\alpha = 70.675$ (2)°
$\beta = 71.505$ (2)°
$\gamma = 77.862$ (2)°

$V = 705.29$ (13) Å^3
$Z = 1$
Mo $K\alpha$ radiation
$\mu = 5.57$ mm^{-1}
$T = 200$ K
$0.24 \times 0.20 \times 0.06$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.577$, $T_{max} = 1.000$

4295 measured reflections
2725 independent reflections
2536 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.174$
$S = 1.21$
2725 reflections

169 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 1.99$ e Å^{-3}
$\Delta\rho_{min} = -2.96$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.88	1.94	2.782 (16)	161
O1—H1A···Cl1^{ii}	0.84	2.74	3.485 (12)	149
O1—H1B···Cl1	0.84	2.62	3.342 (12)	145

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x, -y + 1, -z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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supporting information

Diacridinium hexachloridoplatinate(IV) dihydrate

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S1. Comment

The asymmetric unit of the title compound, $(C_{13}H_{10}N)_2[PtCl_6].2H_2O$, contains a protonated acridine cation, one half of a $[PtCl_6]^{2-}$ dianionic complex and a solvent water molecule (Fig. 1). The anion occupies a special position in the inversion centre; the Pt—Cl bond lengths, 2.370 (3), 2.377 (3) and 2.379 (3) Å, are similar to those found in other $PtCl_6$ salts, i.e. $(C_{13}H_{10}N)_2[PtCl_6].2C_2H_6OS$ (Karaca *et al.*, 2009), $(C_{14}H_{13}N_2)_2[PtCl_6]$ (Yousefi *et al.*, 2007) and $(HPyX-3)_2[PtCl_6].2H_2O$ (X = Br or I) (Zordan & Brammer, 2004).

   The essentially planar acridinium cations [maximum deviation from the least-squares plane is equal to 0.025 (17) Å], are stacked in columns along the a-axis (Fig. 2); the shortest distance between the centroids of the six-membered rings in neighboring cations in the stack is equal to 3.553 (9) Å. Two independent O-H···Cl bonds, both involving atom Cl1 of the anion as acceptor (Table 1), give rise to the chains also running along the a-axis; in addition each water molecule, as an H-bond acceptor is linked to the acridinium N-H group (Fig. 2).

S2. Experimental

To a solution of K_2PtCl_6 (0.1999 g, 0.411 mmol) in H_2O (20 ml) was added acridine (0.1548 g, 0.864 mmol) and the mixture was refluxed for 7 h. The precipitate was then separated by filtration, washed with water and acetone, and dried at 50 °C, to give an orange powder (0.2198 g). Crystals suitable for X-ray analysis were obtained by slow evaporation from a CH_3CN solution.

S3. Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms [C—H = 0.95 Å, N—H = 0.88 Å and $U_{iso}(H) = 1.2U_{eq}(C, N)$]. The H atoms of the solvent water molecule were located from difference maps then allowed to ride on their parent O atom in the final cycles of refinement [O—H = 0.84 Å; $U_{iso}(H) = 1.5U_{eq}(O)$]. The highest peak (1.99 e Å^{-3}) and the deepest hole (-2.96 e Å^{-3}) in the difference Fourier map are located 0.60 and 0.84 Å from the Cl3 and Pt1 atoms, respectively.
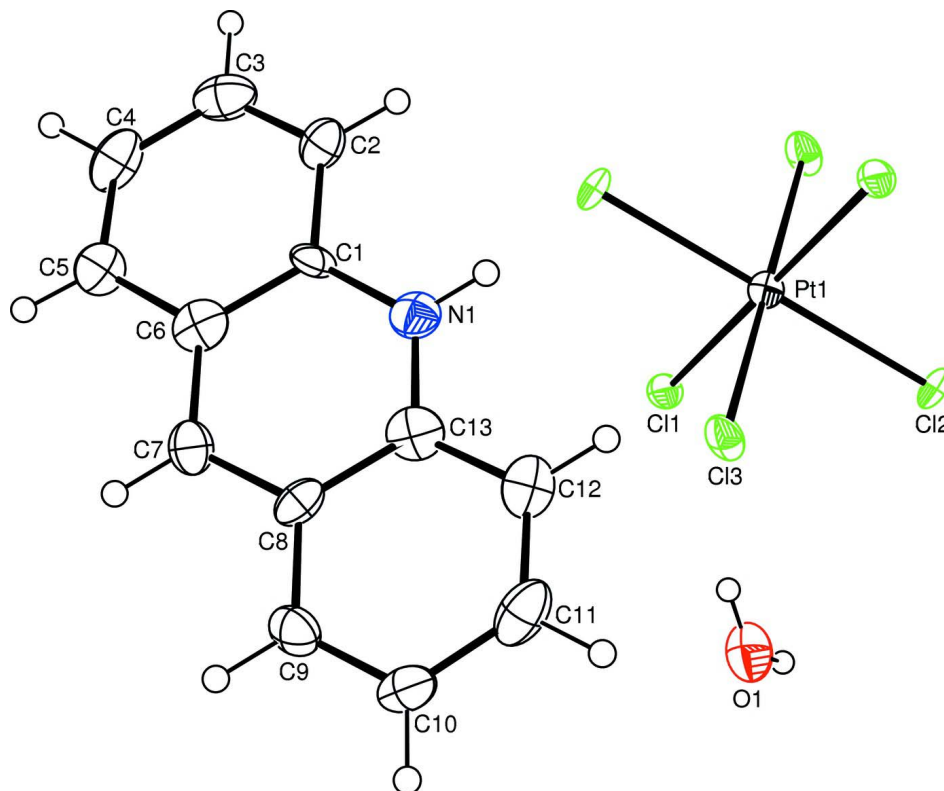
Figure 1

The structure of the title compound, with displacement ellipsoids drawn at the 50% probability level; H atoms are shown as small circles of arbitrary radius. Unlabelled atoms are related to the reference atoms by the (1-x, 1-y, -z) symmetry transformation.
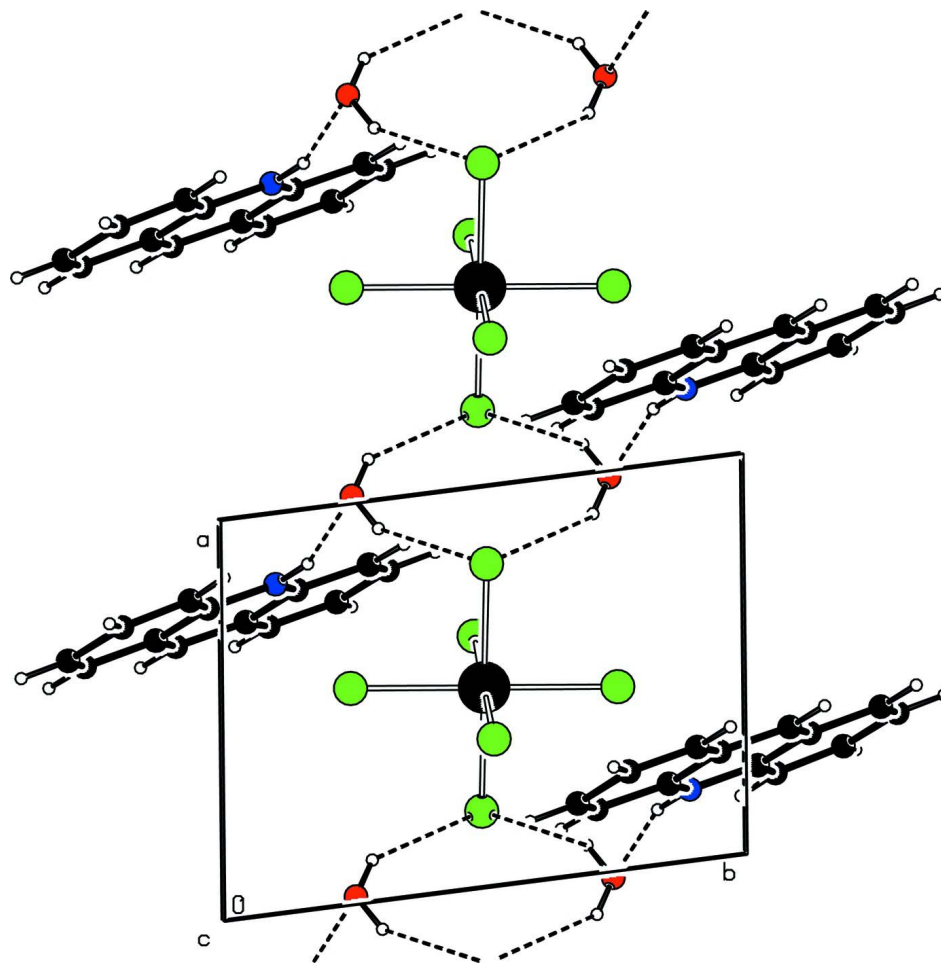
Figure 2

Packing diagram for the crystal of the title compound viewed down the c-axis; H-bonds are drawn as dashed lines.

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Crystal data

$(C_{13}H_{10}N)_2[PtCl_6]\cdot 2H_2O$	$Z = 1$
$M_r = 804.26$	$F(000) = 390$
Triclinic, $P\bar{1}$	$D_x = 1.894$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.4781$ (8) Å	Cell parameters from 2945 reflections
$b = 9.8973$ (10) Å	$\theta = 2.6$–26.0°
$c = 10.7226$ (12) Å	$\mu = 5.57$ mm^{-1}
$\alpha = 70.675$ (2)°	$T = 200$ K
$\beta = 71.505$ (2)°	Plate, orange
$\gamma = 77.862$ (2)°	$0.24 \times 0.20 \times 0.06$ mm
$V = 705.29$ (13) Å^3	

Data collection

Bruker SMART 1000 CCD diffractometer	φ and ω scans
Radiation source: fine-focus sealed tube	Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
Graphite monochromator	$T_{min} = 0.577$, $T_{max} = 1.000$

4295 measured reflections	$\theta_{max} = 26.0°$, $\theta_{min} = 2.1°$
2725 independent reflections	$h = -9 \rightarrow 8$
2536 reflections with $I > 2\sigma(I)$	$k = -12 \rightarrow 12$
$R_{int} = 0.029$	$l = -13 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.059$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.174$	
$S = 1.21$	H-atom parameters constrained
2725 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 22.3646P]$
169 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 1.99$ e Å^{-3}
	$\Delta\rho_{min} = -2.96$ 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.

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 > 2sigma(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}
Pt1	0.5000	0.5000	0.0000	0.0289 (2)
Cl1	0.1916 (4)	0.4938 (3)	0.1558 (3)	0.0282 (7)
Cl2	0.3696 (4)	0.5171 (3)	−0.1801 (3)	0.0289 (7)
Cl3	0.5401 (5)	0.2446 (3)	0.0506 (3)	0.0315 (7)
N1	0.8227 (15)	0.1042 (13)	0.3453 (12)	0.035 (3)
H1	0.8630	0.1656	0.2645	0.042*
C1	0.8023 (17)	0.1451 (13)	0.4598 (13)	0.027 (3)
C2	0.842 (2)	0.2841 (15)	0.4478 (15)	0.035 (3)
H2	0.8838	0.3500	0.3600	0.043*
C3	0.820 (2)	0.3208 (17)	0.5619 (18)	0.047 (4)
H3	0.8465	0.4130	0.5553	0.056*
C4	0.758 (2)	0.2231 (18)	0.6908 (16)	0.046 (4)
H4	0.7445	0.2515	0.7699	0.055*
C5	0.716 (2)	0.0926 (16)	0.7086 (16)	0.040 (3)
H5	0.6727	0.0301	0.7982	0.048*
C6	0.7368 (19)	0.0490 (15)	0.5907 (16)	0.037 (3)
C7	0.694 (2)	−0.0880 (15)	0.6011 (14)	0.036 (3)
H7	0.6495	−0.1540	0.6883	0.043*
C8	0.7195 (18)	−0.1244 (15)	0.4794 (14)	0.032 (3)
C9	0.678 (2)	−0.2605 (15)	0.4860 (15)	0.038 (3)
H9	0.6376	−0.3289	0.5724	0.045*

C10	0.698 (2)	−0.2930 (16)	0.3666 (16)	0.040 (3)
H10	0.6665	−0.3824	0.3696	0.049*
C11	0.765 (2)	−0.1910 (17)	0.2399 (17)	0.047 (4)
H11	0.7788	−0.2149	0.1582	0.057*
C12	0.810 (2)	−0.0609 (17)	0.2278 (15)	0.040 (3)
H12	0.8583	0.0039	0.1405	0.048*
C13	0.784 (2)	−0.0259 (14)	0.3493 (16)	0.037 (3)
O1	0.0214 (17)	0.2503 (13)	0.0838 (11)	0.054 (3)
H1A	−0.0687	0.3001	0.0522	0.081*
H1B	0.1071	0.2824	0.0970	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.0362 (4)	0.0291 (4)	0.0234 (4)	−0.0105 (3)	−0.0078 (3)	−0.0060 (3)
Cl1	0.0281 (16)	0.0351 (17)	0.0217 (14)	−0.0124 (13)	0.0031 (12)	−0.0124 (13)
Cl2	0.0365 (17)	0.0379 (17)	0.0221 (15)	−0.0061 (13)	−0.0158 (13)	−0.0125 (13)
Cl3	0.049 (2)	0.0154 (14)	0.0234 (15)	−0.0030 (13)	−0.0029 (13)	−0.0041 (12)
N1	0.024 (6)	0.043 (7)	0.039 (7)	−0.006 (5)	−0.008 (5)	−0.012 (5)
C1	0.023 (6)	0.027 (6)	0.028 (7)	−0.013 (5)	0.003 (5)	−0.007 (5)
C2	0.036 (8)	0.036 (8)	0.038 (8)	0.004 (6)	−0.018 (6)	−0.012 (6)
C3	0.042 (9)	0.035 (8)	0.072 (12)	−0.001 (7)	−0.024 (8)	−0.018 (8)
C4	0.049 (9)	0.055 (10)	0.046 (9)	0.014 (8)	−0.017 (7)	−0.036 (8)
C5	0.038 (8)	0.042 (8)	0.040 (8)	−0.001 (6)	−0.008 (6)	−0.016 (7)
C6	0.028 (7)	0.034 (7)	0.052 (9)	0.010 (6)	−0.017 (6)	−0.017 (7)
C7	0.036 (8)	0.035 (7)	0.032 (7)	0.003 (6)	−0.012 (6)	−0.006 (6)
C8	0.024 (7)	0.039 (8)	0.039 (8)	0.002 (5)	−0.015 (6)	−0.019 (6)
C9	0.040 (8)	0.027 (7)	0.042 (8)	−0.003 (6)	−0.016 (7)	0.000 (6)
C10	0.043 (8)	0.037 (8)	0.051 (9)	−0.006 (6)	−0.019 (7)	−0.017 (7)
C11	0.056 (10)	0.048 (9)	0.051 (10)	0.015 (8)	−0.027 (8)	−0.031 (8)
C12	0.032 (8)	0.047 (9)	0.039 (8)	0.010 (6)	−0.007 (6)	−0.020 (7)
C13	0.034 (8)	0.024 (7)	0.053 (9)	0.012 (6)	−0.018 (7)	−0.012 (6)
O1	0.052 (7)	0.064 (8)	0.039 (6)	−0.005 (6)	−0.008 (5)	−0.012 (6)

Geometric parameters (Å, º)

Pt1—Cl2[i]	2.370 (3)	C5—C6	1.42 (2)
Pt1—Cl2	2.370 (3)	C5—H5	0.9500
Pt1—Cl3[i]	2.377 (3)	C6—C7	1.42 (2)
Pt1—Cl3	2.377 (3)	C7—C8	1.412 (19)
Pt1—Cl1[i]	2.379 (3)	C7—H7	0.9500
Pt1—Cl1	2.379 (3)	C8—C13	1.42 (2)
N1—C13	1.362 (18)	C8—C9	1.418 (19)
N1—C1	1.370 (17)	C9—C10	1.38 (2)
N1—H1	0.8800	C9—H9	0.9500
C1—C6	1.411 (19)	C10—C11	1.41 (2)
C1—C2	1.424 (18)	C10—H10	0.9500
C2—C3	1.34 (2)	C11—C12	1.36 (2)

C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.40 (2)	C12—C13	1.40 (2)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.33 (2)	O1—H1A	0.8400
C4—H4	0.9500	O1—H1B	0.8400
Cl2^i—Pt1—Cl2	180.0	C3—C4—H4	118.2
Cl2^i—Pt1—Cl3^i	89.44 (11)	C4—C5—C6	118.4 (15)
Cl2—Pt1—Cl3^i	90.56 (11)	C4—C5—H5	120.8
Cl2^i—Pt1—Cl3	90.56 (11)	C6—C5—H5	120.8
Cl2—Pt1—Cl3	89.44 (11)	C1—C6—C7	119.4 (13)
Cl3^i—Pt1—Cl3	180.00 (16)	C1—C6—C5	118.8 (13)
Cl2^i—Pt1—Cl1^i	90.27 (11)	C7—C6—C5	121.8 (14)
Cl2—Pt1—Cl1^i	89.73 (11)	C8—C7—C6	118.7 (13)
Cl3^i—Pt1—Cl1^i	90.55 (11)	C8—C7—H7	120.6
Cl3—Pt1—Cl1^i	89.45 (11)	C6—C7—H7	120.6
Cl2^i—Pt1—Cl1	89.73 (11)	C7—C8—C13	120.8 (13)
Cl2—Pt1—Cl1	90.27 (11)	C7—C8—C9	120.3 (13)
Cl3^i—Pt1—Cl1	89.45 (11)	C13—C8—C9	118.9 (12)
Cl3—Pt1—Cl1	90.55 (11)	C10—C9—C8	119.7 (13)
Cl1^i—Pt1—Cl1	180.00 (10)	C10—C9—H9	120.1
C13—N1—C1	123.9 (12)	C8—C9—H9	120.1
C13—N1—H1	118.0	C9—C10—C11	118.7 (13)
C1—N1—H1	118.0	C9—C10—H10	120.6
N1—C1—C6	119.2 (11)	C11—C10—H10	120.6
N1—C1—C2	120.8 (12)	C12—C11—C10	123.9 (14)
C6—C1—C2	119.9 (12)	C12—C11—H11	118.1
C3—C2—C1	119.3 (14)	C10—C11—H11	118.1
C3—C2—H2	120.4	C11—C12—C13	117.3 (15)
C1—C2—H2	120.4	C11—C12—H12	121.4
C2—C3—C4	119.9 (14)	C13—C12—H12	121.4
C2—C3—H3	120.0	N1—C13—C12	120.7 (14)
C4—C3—H3	120.0	N1—C13—C8	117.9 (13)
C5—C4—C3	123.7 (14)	C12—C13—C8	121.4 (13)
C5—C4—H4	118.2	H1A—O1—H1B	125.9
C13—N1—C1—C6	−0.4 (19)	C6—C7—C8—C13	−1 (2)
C13—N1—C1—C2	−178.9 (12)	C6—C7—C8—C9	180.0 (12)
N1—C1—C2—C3	179.7 (13)	C7—C8—C9—C10	177.9 (13)
C6—C1—C2—C3	1 (2)	C13—C8—C9—C10	−1 (2)
C1—C2—C3—C4	0 (2)	C8—C9—C10—C11	2 (2)
C2—C3—C4—C5	−1 (2)	C9—C10—C11—C12	−1 (2)
C3—C4—C5—C6	1 (2)	C10—C11—C12—C13	−2 (2)
N1—C1—C6—C7	0.3 (19)	C1—N1—C13—C12	−179.3 (12)
C2—C1—C6—C7	178.8 (12)	C1—N1—C13—C8	−0.1 (19)
N1—C1—C6—C5	−179.8 (12)	C11—C12—C13—N1	−178.5 (13)
C2—C1—C6—C5	−1.3 (19)	C11—C12—C13—C8	2 (2)
C4—C5—C6—C1	0 (2)	C7—C8—C13—N1	0.6 (19)

C4—C5—C6—C7	−179.7 (14)	C9—C8—C13—N1	179.9 (12)
C1—C6—C7—C8	0.2 (19)	C7—C8—C13—C12	179.8 (13)
C5—C6—C7—C8	−179.7 (13)	C9—C8—C13—C12	−1 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1ⁱⁱ	0.88	1.94	2.782 (16)	161
O1—H1*A*···Cl1ⁱⁱⁱ	0.84	2.74	3.485 (12)	149
O1—H1*B*···Cl1	0.84	2.62	3.342 (12)	145

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