

# Synthesis and supramolecular networks of 5,6-dioxo-1,10-phenanthroline-2,9-dicarboxylic acid dihydrate and its first coordination compound *cis*-diaquachlorido(5,6-dioxo-1,10-phenanthroline-2,9-dicarboxylic acid- $\kappa^4 O^2, N, N', O^9$ )-manganese(II) chloride dihydrate

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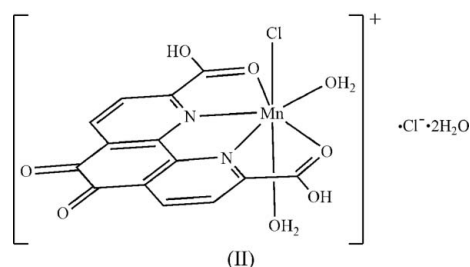
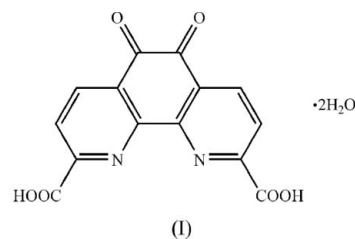
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5,6-Dioxo-1,10-phenanthroline-2,9-dicarboxylic acid dihydrate ( $H_2pdda \cdot 2H_2O$ ),  $C_{14}H_6N_2O_6 \cdot 2H_2O$ , was obtained by carbonylation of 1,10-phenanthroline-2,9-dicarboxylic acid. Its first coordination compound,  $[MnCl(C_{14}H_6N_2O_6)(H_2O)_2]Cl \cdot 2H_2O$ , in which the  $H_2pdda$  ligand remains protonated, was synthesized in aqueous acetic acid.  $H_2pdda$  chelates one water molecule *via* hydrogen bonds in  $H_2pdda \cdot 2H_2O$ , whereas in the coordination compound it chelates one heptacoordinate  $Mn^{II}$  atom *via* coordination bonds. The N atoms in  $H_2pdda \cdot 2H_2O$  and the Cl atoms in the coordination compound are also involved in hydrogen bonds. Extensive hydrogen bonding results in supramolecular networks in both compounds.

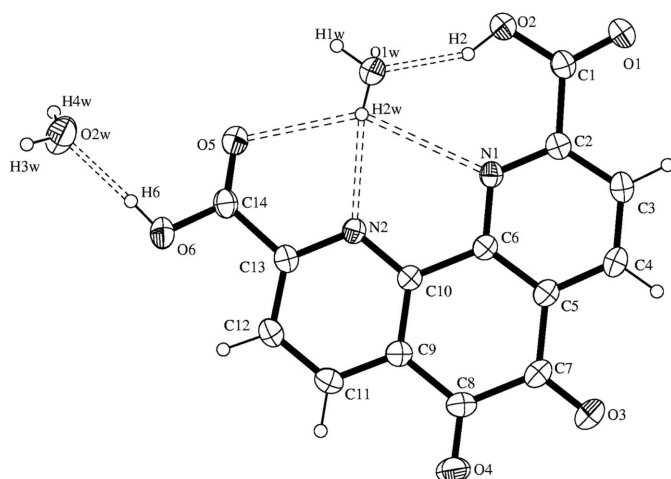
## Comment

1,10-Phenanthroline is among the most extensively studied chelators and its numerous derivatives have played key roles in coordination and supramolecular chemistry (Bencini & Lippolis, 2010; Luman & Castellano, 2004). 1,10-Phenanthroline-5,6-dione (Ma *et al.*, 2010; Braband *et al.*, 2010; Dong *et al.*, 2010; Liu *et al.*, 2008; Wang *et al.*, 2006; Zheng *et al.*, 2010; Patel *et al.*, 2010; Goswami & Chakrabarty, 2010; Si *et al.*, 2009; Ettetdgui & Neumann, 2009; Zhao *et al.*, 2008; Bodige & MacDonnell, 1997; Lenaerts *et al.*, 2005; Cardinaels *et al.*, 2008; Beaudoin & Obare, 2008) and 1,10-phenanthroline-2,9-dicarboxylic acid (Miao *et al.*, 2010; Chen *et al.*, 2010; Williams *et al.*, 2009; Harbuzaru *et al.*, 2009; Dean *et al.*, 2008; Fan *et al.*, 2008; Melton *et al.*, 2006; Garas & Vagg, 2000) are two important derivatives, whereas 5,6-dioxo-1,10-phenanthro-

line-2,9-dicarboxylic acid ( $H_2pdda$ ), which combines both the 5,6-dicarbonyl and 2,9-dicarboxyl groups, has received little attention (Lamarque *et al.*, 2010; Echavarren & Porcel, 2006; Garas & Vagg, 2000; Evangelista & Pollak, 1986). No coordination compound of  $H_2pdda$  has been reported. Continuing our interest in polypyridine compounds and their coordination behaviour (Wu *et al.*, 1997, 2002, 2005), we discuss here the synthesis and structure of  $H_2pdda \cdot 2H_2O$  (I), and its first coordination compound, *viz.*  $[MnCl(H_2pdda)(H_2O)_2]Cl \cdot 2H_2O$  (II).



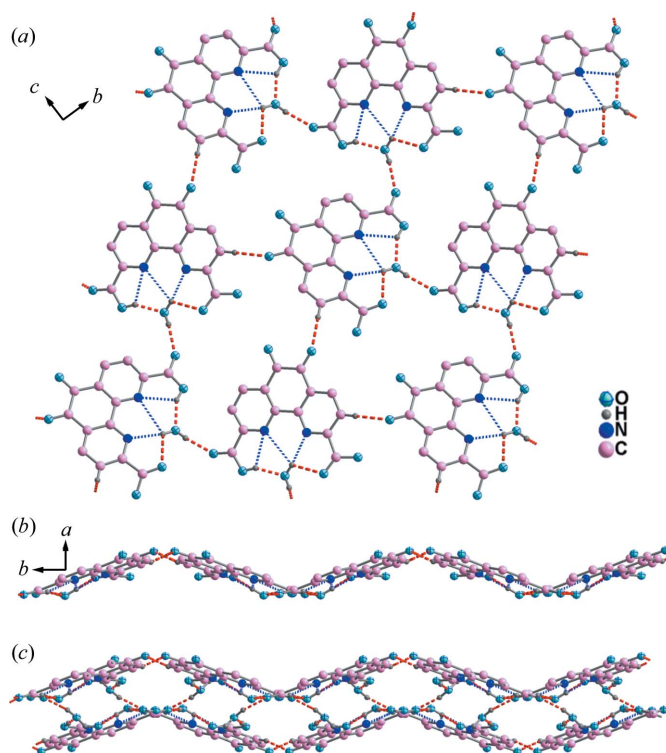
Two previous procedures for obtaining (I) were reported (Evangelista & Pollak, 1986; Garas & Vagg, 2000) using 2,9-dimethyl-1,10-phenanthroline-5,6-dione as the reactant, but the yields were not high and the handling procedures were somewhat complicated. Here we have employed a new synthetic route with 1,10-phenanthroline-2,9-dicarboxylic acid as the starting material. The main advantages of this route are the improved yield and simpler product separation. The normal carbonylation procedure for 1,10-phenanthroline-type compounds includes oxidation with  $H_2SO_4/HNO_3/KBr$  (or  $NaBr$ ), neutralization of the resulting acidic solution, extraction of the quinone with dichloromethane, drying and distilling of the organic solution (Ma *et al.*, 2010; Braband *et al.*, 2010; Dong *et al.*, 2010; Liu *et al.*, 2008; Wang *et al.*, 2006; Zheng *et al.*, 2010; Patel *et al.*, 2010; Goswami & Chakrabarty, 2010; Si *et al.*, 2009; Ettetdgui & Neumann, 2009; Zhao *et al.*, 2008; Bodige & MacDonnell, 1997; Lenaerts *et al.*, 2005; Cardinaels *et al.*, 2008; Beaudoin & Obare, 2008). For 1,10-phenanthroline-2,9-dicarboxylic acid, the solution remains acidic after oxidation and, on pouring into cold water, the product precipitated directly with a high yield. This is reasonable since the two carboxyl groups should make  $H_2pdda$  less soluble in acidic solution. The proton NMR spectrum of (I) shows three kinds of protons, including the carboxyl protons, and their integration areas are fully consistent with the structure of  $H_2pdda$ . Interestingly, the X-ray structural analysis confirms that  $H_2pdda$  preferentially exists as the dihydrate, just like the compounds reported previously without X-ray analysis (Evangelista & Pollak, 1986; Garas & Vagg, 2000).



**Figure 1**  
The asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level. Dashed lines represent hydrogen bonds.

Complex (II) is the first reported coordination compound of  $H_2pdda$  and it is noteworthy that this crystalline solid was obtained in acetic acid/water solution. The acidic solution favours the neutral form of  $H_2pdda$ , which is supported by the IR spectrum of the complex since the characteristic strong band around  $1600\text{ cm}^{-1}$ , corresponding to the asymmetric stretching of a deprotonated carboxyl group (Nakamoto, 2009), was not observed. The X-ray diffraction data for both (I) and (II) are of high quality and the H atoms are generally visible in the difference Fourier maps. Therefore, we believe the detailed description of the structures (specifically the hydrogen bonding) is reliable.

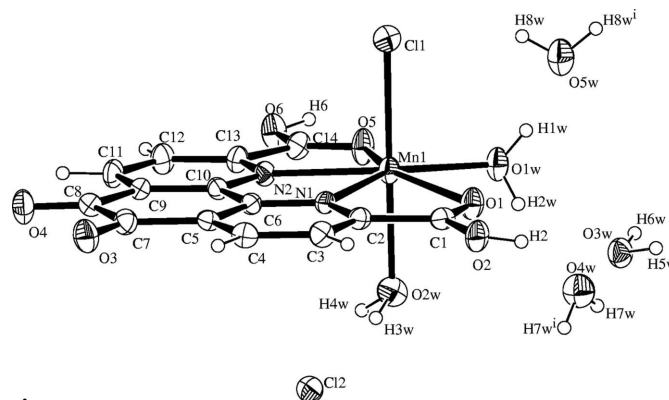
The asymmetric unit of (I) is shown in Fig. 1 and consists of one  $H_2pdda$  molecule and two water molecules. The lengths of the C7–O3 and C8–O4 carbonyl bonds are 1.202 (3) and 1.210 (3) Å, respectively, comparable to the corresponding bond lengths in 1,10-phenanthroline-5,6-dione which range from 1.207 (3) to 1.214 (3) Å (Calderazzo *et al.*, 1999). This reflects the fact that the two carboxyl groups at the 2- and 9-positions do not have a significant influence on the carbonyl groups. The locations of the carboxyl H atoms which are clearly visible in the difference Fourier map are also confirmed by the C–O bond lengths: the C1=O1 and C14=O5 double-bond lengths are 1.219 (3) and 1.200 (3) Å, respectively, whereas the C1–O2 and C14–O6 single-bond lengths are significantly longer at 1.298 (3) and 1.305 (3) Å, respectively. The two carboxyl groups are approximately coplanar with the phenanthroline unit, particularly that including atom C1; the N1–C2–C1–O1 and N2–C13–C14–O5 torsion angles are 176.5 (2) and 15.3 (4)°, respectively. It is interesting that the O1W water molecule is situated such that the O atom and its attached H atom (H2W) look to be chelated by two N atoms and two carboxyl groups through O–H···O and O–H···N hydrogen bonds (Table 1, and Figs. 1 and 2). This water molecule donates the other H atom (H1W) to carboxyl atom O1 in an adjacent  $H_2pdda$  molecule (symmetry code:  $x, -y + \frac{5}{2}, z - \frac{1}{2}$ ). These classical hydrogen bonds, together with nonclassical intermolecular C–H···O hydrogen bonds



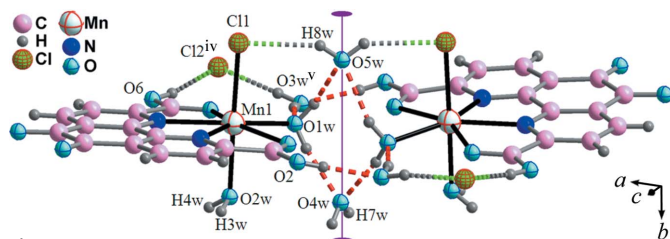
**Figure 2**  
Three views of the hydrogen-bonding networks in (I). The  $H_2pdda$  layer and the O1W water molecules are shown in (a), viewed down the  $a$  axis. These layers are bridged by O2W water molecules to form the double layers shown in (b) and (c) viewed down the  $c$  axis.

(Steiner, 1996) between C12 and O4( $x, -y + \frac{3}{2}, z - \frac{1}{2}$ ), lead to the formation of a corrugated layer parallel to the (100) plane, as shown in Fig. 2. The other water molecule (O2W) forms three hydrogen bonds with three  $H_2pdda$  molecules. These hydrogen bonds further link two adjacent antiparallel layers, giving rise to a double-layer network (Fig. 2b).

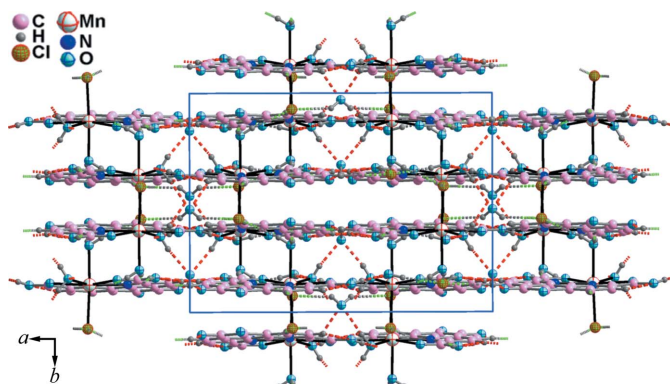
The molecular structure of (II) is shown in Fig. 3. The  $Mn^{II}$  atom is seven coordinate, ligated by a tetradentate  $H_2pdda$  molecule, two water molecules and one chloride ion, forming the pentagonal–bipyramidal geometry. The  $H_2pdda$  ligand remains protonated and binds to the  $Mn^{II}$  atom in a planar



**Figure 3**  
The molecular structure of (II), with displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (i)  $-x + 1, y, -z + \frac{1}{2}$ .]


**Figure 4**

The dimer composed of two twofold-axis-related molecules in (II), formed by hydrogen bonds (dashed bonds). The twofold axis which bisects water molecules O4W and O5W is shown. Symmetry codes are as in Table 2.


**Figure 5**

The pillar-layer-like hydrogen-bond network in (II).

$\text{N}_2\text{O}_2$  chelation manner. The rigid nature of the ligand ensures that the Mn–O(carboxyl) bond lengths [2.430 (3) and 2.461 (3) Å for O5 and O1, respectively] are longer than the Mn–O(water) bond lengths [2.204 (3) and 2.248 (3) Å for O1W and O2W, respectively], which are comparable to the Mn–N bond lengths [2.295 (3) and 2.298 (3) Å for N1 and N2, respectively]. The Mn–Cl bond length [2.4945 (15) Å] falls into the expected range. Inspection of the bond lengths for  $\text{H}_2\text{pdda}$  in (I) and (II) reveals no remarkable difference in the corresponding data, indicative of similar environments for the  $\text{H}_2\text{pdda}$  molecule in the two compounds. This is reasonable if the  $\text{H}_2\text{pdda}$  molecule in (I) is considered to ‘chelate’ the water molecule in a similar fashion to the binding of  $\text{H}_2\text{pdda}$  in (II), where the tetradentate  $\text{H}_2\text{pdda}$  chelates the  $\text{Mn}^{\text{II}}$  atom. The discrepancy in binding guests and bonding nature does not induce a significant change in the structure of  $\text{H}_2\text{pdda}$ .

There are also extensive hydrogen bonds (Table 2) in (II). As shown in Fig. 4, water molecules O4W or O5W are located on the same twofold axis. They connect two symmetry-related manganese complexes *via* hydrogen bonds to form a dimer. The O3W water molecule and the  $\text{Cl}^-$  counter-ion are also involved in the formation of the dimer. Continuous hydrogen-bonding linkage among the dimers throughout the crystal lattice leads to a pillar-layer-like three-dimensional supra-molecular network, as illustrated in Fig. 5.

## Experimental

For the synthesis of (I), cold concentrated  $\text{H}_2\text{SO}_4$  (20 ml) and then concentrated  $\text{HNO}_3$  (10 ml) were added dropwise to a three-necked

reaction flask loaded with 1,10-phenanthroline-2,9-dicarboxylic acid (De Cian *et al.*, 2007) (2.68 g, 10 mmol) and potassium bromide (1.78 g, 15 mmol) in an ice–salt bath. After stirring for a further 30 min, the mixture was heated to 358 K and refluxed for 4 h. The condenser tube was then removed and the solution allowed to cool to room temperature. The solution was poured into ice water (200 ml). The yellow precipitate was collected and washed (yield 2.70 g, 8.1 mmol, 81%). Recrystallization from acetone afforded crystals (m.p. 478 K) suitable for X-ray crystallographic analysis.  $^1\text{H}$  NMR ( $d_6$ -DMSO):  $\delta$  13.70 (*br*, 2H, COOH), 8.60 (*d*, 2H,  $J = 8.0$  Hz, 3-H), 8.27 (*d*, 2H,  $J = 8.0$  Hz, 4-H). IR (KBr,  $\nu$   $\text{cm}^{-1}$ ): 3514 (*s*), 3092 (*m*), 1927 (*m*), 1701 (*vs*), 1630 (*m*), 1570 (*s*), 1431 (*m*), 1385 (*s*), 1294 (*m*), 1229 (*m*), 1155 (*m*), 992 (*m*), 931 (*m*), 726 (*m*), 531 (*s*).

For the synthesis of (II), a solution of  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  (19.8 mg, 0.1 mmol) in water (5 ml) was added dropwise to a solution of  $\text{H}_2\text{pdda} \cdot 2\text{H}_2\text{O}$  (33.4 mg, 0.1 mmol) in glacial acetic acid (10 ml). The mixture was stirred for 2 h at 358 K and the resulting clear solution was evaporated for two weeks at room temperature to yield yellow crystals. IR (KBr,  $\nu$   $\text{cm}^{-1}$ ): 3414 (*b s*), 2466 (*m*), 1876 (*m*), 1703 (*vs*), 1608 (*m*), 1575 (*s*), 1468 (*m*), 1389 (*m*), 1262 (*s*), 1242 (*m*), 936 (*m*), 812 (*m*), 714 (*m*), 528 (*m*).

## Compound (I)

### Crystal data

$\text{C}_{14}\text{H}_6\text{N}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$   
 $M_r = 334.24$   
 Monoclinic,  $P2_1/c$   
 $a = 7.206$  (2) Å  
 $b = 15.968$  (6) Å  
 $c = 13.055$  (3) Å  
 $\beta = 115.049$  (13)°

$V = 1360.9$  (7) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.14$   $\text{mm}^{-1}$   
 $T = 296$  K  
 $0.20 \times 0.13 \times 0.12$  mm

### Data collection

Bruker APEXII CCD area-detector diffractometer 6770 measured reflections  
 2517 independent reflections  
 Absorption correction: multi-scan (SADABS; Bruker, 2002) 1610 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$   
 $T_{\text{min}} = 0.973$ ,  $T_{\text{max}} = 0.984$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$  217 parameters  
 $wR(F^2) = 0.138$  H-atom parameters constrained  
 $S = 1.03$   $\Delta\rho_{\text{max}} = 0.18$   $\text{e} \text{ \AA}^{-3}$   
 2517 reflections  $\Delta\rho_{\text{min}} = -0.29$   $\text{e} \text{ \AA}^{-3}$

## Compound (II)

### Crystal data

$[\text{MnCl}(\text{C}_{14}\text{H}_6\text{N}_2\text{O}_6)(\text{H}_2\text{O})_2]\text{Cl} \cdot 2\text{H}_2\text{O}$   
 $M_r = 496.11$   
 Orthorhombic,  $Pbcn$   
 $a = 17.195$  (6) Å  
 $b = 12.411$  (5) Å  
 $c = 17.569$  (6) Å

$V = 3750$  (2) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.05$   $\text{mm}^{-1}$   
 $T = 296$  K  
 $0.42 \times 0.40 \times 0.30$  mm

### Data collection

Bruker APEXII CCD area-detector diffractometer 17437 measured reflections  
 3372 independent reflections  
 Absorption correction: multi-scan (SADABS; Bruker, 2002) 2021 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.082$   
 $T_{\text{min}} = 0.667$ ,  $T_{\text{max}} = 0.744$

**Table 1**

Hydrogen-bond geometry (Å, °) for (I).

D—H...A	D—H	H...A	D...A	D—H...A
C12—H12...O4 <sup>i</sup>	0.93	2.45	3.367 (1)	170
O1W—H1W...O1 <sup>ii</sup>	0.84	2.03	2.865 (3)	170
O2—H2...O1W	0.82	1.81	2.561 (2)	152
O1W—H2W...O5	0.83	2.44	2.917 (3)	117
O1W—H2W...N2	0.83	2.15	2.968 (3)	169
O2W—H3W...O1 <sup>iii</sup>	0.86	1.96	2.805 (3)	169
O2W—H4W...O3 <sup>iv</sup>	0.86	2.53	3.053 (3)	120
O6—H6...O2W	0.82	1.76	2.574 (3)	176

Symmetry codes: (i)  $x, -y + \frac{3}{2}, z - \frac{1}{2}$ ; (ii)  $x, -y + \frac{5}{2}, z - \frac{1}{2}$ ; (iii)  $-x, y - \frac{1}{2}, -z + \frac{1}{2}$ ; (iv)  $x, y, z - 1$ .

**Refinement**

$R[F^2 > 2\sigma(F^2)] = 0.046$  265 parameters  
 $wR(F^2) = 0.121$  H-atom parameters constrained  
 $S = 0.98$   $\Delta\rho_{\max} = 0.48 \text{ e \AA}^{-3}$   
 3372 reflections  $\Delta\rho_{\min} = -0.33 \text{ e \AA}^{-3}$

H atoms attached to C atoms were positioned geometrically and allowed to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . H atoms bound to O atoms were located from difference Fourier maps and treated in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . The method described by Cooper *et al.* (2010) was applied to assist the location of the H atoms, especially H3W and H4W, for more rational hydrogen bonds and short contacts.

For both compounds, data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TP3010). Services for accessing these data are described at the back of the journal.

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**Table 2**

Hydrogen-bond geometry (Å, °) for (II).

D—H...A	D—H	H...A	D...A	D—H...A
C11—H11...Cl2 <sup>i</sup>	0.93	2.89	3.799 (3)	166
C12—H12...O6 <sup>ii</sup>	0.93	2.47	3.283 (5)	146
C4—H4...Cl1 <sup>iii</sup>	0.93	2.72	3.640 (4)	171
O5W—H8W...Cl1	0.82	2.45	3.1625 (16)	145
O4W—H7W...O4 <sup>iv</sup>	0.82	2.22	2.947 (3)	149
O3W—H6W...O5 <sup>v</sup>	0.81	2.69	3.230 (4)	125
O3W—H6W...O1W <sup>v</sup>	0.81	2.10	2.888 (4)	164
O3W—H5W...Cl2 <sup>vi</sup>	0.83	2.40	3.217 (3)	169
O2W—H4W...Cl1 <sup>vii</sup>	0.82	2.51	3.315 (3)	172
O2W—H3W...Cl2	0.84	2.53	3.371 (3)	172
O1W—H2W...O4W	0.82	1.94	2.751 (4)	172
O1W—H1W...O5W	0.85	2.08	2.858 (5)	151
O6—H6...Cl2 <sup>iv</sup>	0.82	2.19	3.004 (3)	170
O2—H2...O3W	0.82	1.81	2.625 (4)	174

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

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