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Crystal structure of the magnesium salt of the herbicide 2,4-D: pentaaqua[(2,4-dichlorophenoxy)-acetato-κO]magnesium (2,4-dichlorophenoxy)-acetate hemihydrate

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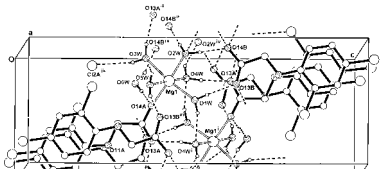
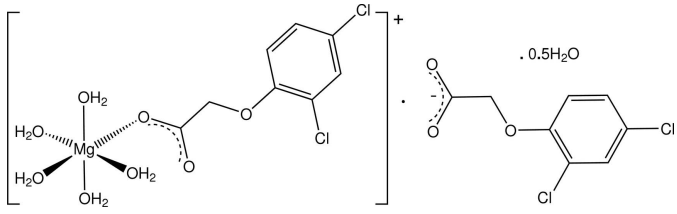
In the crystal structure of the title magnesium salt of the phenoxy herbicide (2,4-dichlorophenoxy)acetic acid (2,4-D), $[Mg(C_8H_5Cl_2O_3)(H_2O)_5](C_8H_5Cl_2O_3)\cdot 0.5H_2O$, the discrete cationic MgO_6 complex unit comprises a carboxylate O-donor from a monodentate 2,4-D anionic ligand and five water molecules, resulting in a slightly distorted octahedral coordination sphere. The free 2,4-D anions are linked to the complex units through duplex water–carboxylate O—H···O hydrogen bonds through the coordinating water molecules. In the crystal, inter-unit O—H···O hydrogen-bonding interactions involving coordinating water molecules as well as the solvent water molecule (occupancy 0.5) with carboxylate O-atom acceptors, give a layered structure lying parallel to (001), in which π–π ligand–cation interactions [minimum ring centroid separation = 3.6405 (17) Å] and a short O—H···Cl interaction are also found.

1. Chemical context

The phenoxyacetic acids comprise an important group of chemicals which has among its members those ring-substituted representatives having selective herbicidal activity, *e.g.* the commercial but in some cases, now prohibited (2,4-dichlorophenoxy)acetic acid (2,4-D), (2,4,5-trichlorophenoxy)acetic acid (2,4,5-T) and (4-chloro-2-methylphenoxy)acetic acid (MCPA) (O'Neil, 2002; Zumdahl, 2010; Cobb & Reade, 2011). Of interest have also been the structures of the metal complexes with these acids, including those with magnesium in which the monoanionic phenoxyacetate ligands (L) display a variety of coordination modes, all based on an octahedral MgO_6 metal stereochemistry. These include discrete monomeric {$[MgL_2(H_2O)_4]$ [L = 2-(2-fluorophenoxy)acetate (Kennard *et al.*, 1986) and L = $MCPA^-$ (Smith *et al.*, 1981)] and $[MgL(H_2O)_5]\cdot L$ [L = $2,4,5\text{-}T^-$ (Smith *et al.*, 1982)]} or polymeric $\{[MgL_2(H_2O)_2]\}_n$ [L = phenoxyacetate, (4-chlorophenoxy)acetate or (4-fluorophenoxy)acetate] (Smith *et al.*, 1980; Smith, 2012)}. The title complex, $[Mg(C_8H_5Cl_2O_3)(H_2O)_5](C_8H_5Cl_2O_3)\cdot 0.5H_2O$, was obtained from the reaction of 2,4-D with $MgCO_3$ in aqueous ethanol and its crystal structure is reported herein.





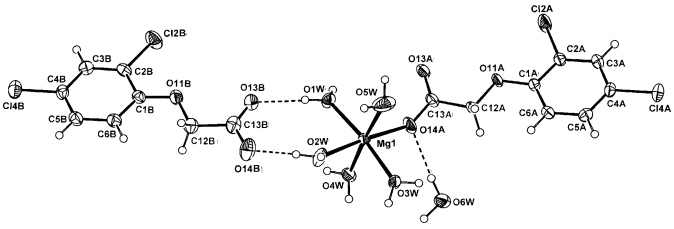


Figure 1
Molecular configuration and atom-naming scheme for the title compound, with displacement ellipsoids drawn at the 40% probability level. Inter-species hydrogen bonds are shown as dashed lines.

2. Structural commentary

In the title complex (Fig. 1), the discrete MgO_6 complex units have, as expected, essentially octahedral stereochemistry [Mg—O bond length range = 2.031 (2)–2.094 (2) Å], comprising a carboxylate O-donor from a monodentate 2,4-D$^-$ ligand and five water molecules. The free 2,4-D$^-$ counter-anion is linked to the complex unit through an unusual duplex water–carboxylate O—H···O hydrogen-bonding association involving the coordinating water molecules O1*W* and O2*W* (Table 1), giving a cyclic ring motif incorporating the Mg^{2+} cation [graph set $R_2^2(8)$]. Except for the presence of the hemihydrate molecule of solvation, the title complex is very similar to that of the Mg complex with the analogous phenoxy herbicide, (2,4,5-trichlorophenoxy)acetic acid (Smith *et al.*, 1982).

In the present complex, both 2,4-D species are essentially planar [defining torsion angles for the oxoacetic acid side chain (C1*A*/*B*—O11*A*/*B*—C12*A*/*B*—C13*A*/*B* and O11*A*/*B*—C12*A*/*B*—C13*A*/*B*—O14*A*/*B*) being 179.0 (2) and 174.8 (2)° (ligand *A*), and 175.7 (2) and 178.7 (2)° (anion *B*), respectively]. This contrasts with the parent acid 2,4-D (Smith *et al.*, 1976), in which the oxoacetic acid side chain adopts a synclinal conformation (benzene ring to carboxyl group dihedral angle = 75.2°).

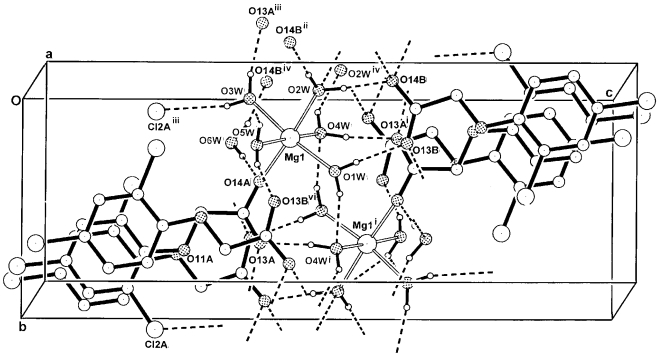


Figure 2
The two-dimensional hydrogen-bonded structure of the title compound in the unit cell, viewed down the *a* axis. Non-associative H atoms have been omitted. For symmetry codes, see Table 1.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11*W*···O13*B*	0.86 (3)	1.92 (3)	2.772 (3)	171 (3)
O1*W*—H12*W*···O4*W*^{i}	0.87 (3)	2.09 (3)	2.939 (3)	165 (3)
O2*W*—H21*W*···O14*B*	0.88 (2)	1.75 (2)	2.623 (3)	176 (3)
O2*W*—H22*W*···O14*B*^{ii}	0.87 (3)	1.88 (3)	2.754 (3)	173 (3)
O3*W*—H31*W*···O13*A*^{iii}	0.87 (2)	1.80 (2)	2.656 (3)	167 (3)
O3*W*—H32*W*···Cl2*A*^{iii}	0.87 (3)	2.50 (3)	3.345 (2)	165 (3)
O4*W*—H41*W*···O13*A*^{i}	0.89 (2)	1.77 (2)	2.652 (3)	172 (4)
O4*W*—H42*W*···O2*W*^{iv}	0.88 (2)	2.19 (3)	2.980 (3)	151 (3)
O5*W*—H51*W*···O6*W*^{v}	0.90 (5)	1.90 (6)	2.543 (5)	127 (4)
O5*W*—H52*W*···O13*B*^{vi}	0.88 (4)	1.86 (4)	2.708 (4)	162 (4)
O6*W*—H61*W*···O14*B*^{iv}	0.91 (6)	1.77 (6)	2.654 (5)	162 (7)
O6*W*—H62*W*···O14*A*	0.90 (6)	2.12 (5)	3.006 (5)	168 (5)

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

3. Supramolecular features

In the crystal of the title compound, inter-unit O—H···O hydrogen-bonding interactions (Table 1) involving all coordinating water molecules, as well as the hemihydrate solvent molecule, with carboxylate O-atom acceptors, give a layered structure lying parallel (001) (Fig. 2). Within these layers, weak π–π interactions between centrosymmetrically related 2,4-D ligand–anion species $A\cdots B^{i}$ are also found. The 2,4-D$^-$ molecules lie parallel to (10$\bar{1}$) and have a minimum ring centroid

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Mg(C_8H_5Cl_2O_3)(H_2O)_5]$-$(C_8H_5Cl_2O_3)\cdot 0.5H_2O$
M_r	563.44
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	200
a, *b*, *c* (Å)	7.3551 (6), 7.6579 (5), 20.7878 (14)
α, β, γ (°)	91.266 (6), 94.341 (6), 94.250 (6)
V (Å^3)	1163.84 (14)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.59
Crystal size (mm)	0.40 × 0.12 × 0.10
Data collection	
Diffractometer	Oxford Diffraction Gemini-S CCD detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min}, T_{max}	0.970, 0.980
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7636, 4575, 3458
R_{int}	0.029
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.050, 0.107, 1.04
No. of reflections	4575
No. of parameters	334
No. of restraints	12
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.69, −0.51

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012), and *PLATON* (Spek, 2009).

separation of 3.6405 (17) Å. A short O3*W*—H···Cl2*A*^{iii} interaction [3.345 (2) Å] is also observed [for symmetry codes (i) and (iii), see: Table 1].

4. Synthesis and crystallization

The title compound was synthesized by the addition of excess $MgCO_3$ to 15 ml of a hot aqueous solution of (2,4-dichlorophenoxy)acetic acid (0.1 mmol) in ethanol–water (1:10 *v*/*v*). After completion of the reaction, excess $MgCO_3$ was removed by filtration and the solution was allowed to evaporate at room temperature, providing colourless prisms of the title compound from which a specimen was cleaved for the X-ray analysis.

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms on all water molecules were located in difference Fourier maps. Their positional parameters were refined with restraints [O—H = 0.90 (2) Å], with U_{iso}(H) = 1.5U_{eq}(O). Other H atoms were included in the refinement at calculated positions (aromatic C—H = 0.95 Å or methylene 0.99 Å), with U_{iso}(H) = 1.2U_{eq}(C), using a riding-model approximation. The site-occupancy factor for the water molecule of solvation was determined as 0.502 (4) and was subsequently fixed at 0.50.

Acknowledgements

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supporting information

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Crystal structure of the magnesium salt of the herbicide 2,4-D: pentaaqua[(2,4-dichlorophenoxy)acetato-*κO*]magnesium (2,4-dichlorophenoxy)acetate hemihydrate

Graham Smith

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009).

Pentaaqua[(2,4-dichlorophenoxy)acetato-*κO*]magnesium (2,4-dichlorophenoxy)acetate hemihydrate

Crystal data

$[Mg(C_8H_5Cl_2O_3)(H_2O)_5](C_8H_5Cl_2O_3)\cdot 0.5H_2O$
$M_r = 563.44$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.3551\ (6)$ Å
$b = 7.6579\ (5)$ Å
$c = 20.7878\ (14)$ Å
$\alpha = 91.266\ (6)°$
$\beta = 94.341\ (6)°$
$\gamma = 94.250\ (6)°$
$V = 1163.84\ (14)$ Å^3
$Z = 2$
$F(000) = 578$
$D_x = 1.608$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1889 reflections
$\theta = 3.5–27.2°$
$\mu = 0.59$ mm^{-1}
$T = 200$ K
Lath, colourless
0.40 × 0.12 × 0.10 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
$T_{min} = 0.970$, $T_{max} = 0.980$
7636 measured reflections
4575 independent reflections
3458 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.3°$
$h = -8\rightarrow 9$
$k = -9\rightarrow 8$
$l = -16\rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.107$
$S = 1.04$
4575 reflections
334 parameters
12 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 0.6822P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 0.69$ e Å^{-3}
	$\Delta\rho_{min} = -0.51$ 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 e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}	Occ. (<1)
Cl2A	0.32241 (15)	1.10346 (11)	0.21550 (4)	0.0581 (3)	
Cl4A	0.09192 (10)	0.77979 (11)	−0.01112 (4)	0.0416 (3)	
Mg1	0.64072 (13)	0.28185 (12)	0.42864 (4)	0.0272 (3)	
O1W	0.7607 (3)	0.4533 (3)	0.50028 (11)	0.0392 (8)	
O2W	0.7909 (3)	0.0906 (3)	0.47054 (11)	0.0428 (8)	
O3W	0.5253 (3)	0.0856 (3)	0.36448 (10)	0.0384 (7)	
O4W	0.4292 (3)	0.2243 (3)	0.48859 (10)	0.0354 (7)	
O5W	0.8325 (4)	0.3474 (5)	0.36472 (14)	0.0803 (14)	
O11A	0.3737 (3)	0.7518 (2)	0.26146 (9)	0.0311 (6)	
O13A	0.5235 (3)	0.7438 (3)	0.38384 (10)	0.0394 (8)	
O14A	0.4784 (3)	0.4541 (3)	0.38457 (11)	0.0523 (9)	
C1A	0.3065 (4)	0.7496 (4)	0.19790 (13)	0.0269 (9)	
C2A	0.2754 (4)	0.9101 (4)	0.17018 (13)	0.0291 (9)	
C3A	0.2087 (4)	0.9207 (4)	0.10640 (14)	0.0314 (9)	
C4A	0.1726 (4)	0.7682 (4)	0.06967 (13)	0.0299 (9)	
C5A	0.2025 (4)	0.6081 (4)	0.09524 (14)	0.0339 (10)	
C6A	0.2687 (4)	0.5993 (4)	0.15926 (14)	0.0322 (10)	
C12A	0.3942 (4)	0.5832 (4)	0.28841 (13)	0.0308 (9)	
C13A	0.4716 (4)	0.5993 (4)	0.35773 (14)	0.0322 (10)	
Cl2B	1.16172 (14)	0.63544 (11)	0.80594 (4)	0.0560 (3)	
Cl4B	1.41057 (11)	0.24035 (12)	0.99966 (4)	0.0459 (3)	
O11B	1.1112 (3)	0.3112 (3)	0.73144 (10)	0.0377 (7)	
O13B	0.9449 (3)	0.3560 (3)	0.61359 (11)	0.0479 (8)	
O14B	0.9418 (3)	0.0723 (4)	0.58839 (11)	0.0584 (10)	
C1B	1.1820 (4)	0.2850 (4)	0.79283 (14)	0.0304 (10)	
C2B	1.2150 (4)	0.4327 (4)	0.83429 (15)	0.0324 (10)	
C3B	1.2847 (4)	0.4191 (4)	0.89740 (14)	0.0341 (10)	
C4B	1.3236 (4)	0.2574 (4)	0.91982 (14)	0.0324 (10)	
C5B	1.2948 (4)	0.1096 (4)	0.88026 (15)	0.0354 (10)	
C6B	1.2253 (4)	0.1242 (4)	0.81655 (15)	0.0356 (10)	

C12B	1.0691 (4)	0.1584 (4)	0.69131 (14)	0.0375 (11)	
C13B	0.9786 (4)	0.2045 (5)	0.62639 (15)	0.0412 (11)	
O6W	0.1369 (6)	0.2203 (6)	0.3500 (2)	0.0411 (17)	0.500
H3A	0.18810	1.03100	0.08830	0.0380*	
H5A	0.17800	0.50420	0.06930	0.0410*	
H6A	0.28850	0.48840	0.17700	0.0390*	
H11W	0.828 (4)	0.423 (5)	0.5331 (13)	0.0590*	
H12A	0.47650	0.51830	0.26280	0.0370*	
H12W	0.713 (5)	0.549 (3)	0.5108 (17)	0.0590*	
H13A	0.27360	0.51580	0.28600	0.0370*	
H21W	0.838 (5)	0.089 (5)	0.5105 (10)	0.0640*	
H22W	0.869 (4)	0.035 (5)	0.4497 (16)	0.0640*	
H31W	0.511 (5)	−0.028 (2)	0.3660 (17)	0.0580*	
H32W	0.464 (4)	0.108 (5)	0.3286 (12)	0.0580*	
H41W	0.451 (4)	0.245 (5)	0.5307 (9)	0.0530*	
H42W	0.371 (4)	0.120 (3)	0.4861 (17)	0.0530*	
H51W	0.912 (6)	0.275 (6)	0.382 (3)	0.1210*	
H52W	0.900 (6)	0.447 (4)	0.363 (2)	0.1210*	
H3B	1.30550	0.52030	0.92500	0.0410*	
H5B	1.32220	−0.00140	0.89630	0.0430*	
H6B	1.20730	0.02270	0.78900	0.0430*	
H12B	0.98600	0.07460	0.71280	0.0450*	
H13B	1.18280	0.10110	0.68470	0.0450*	
H61W	0.126 (10)	0.131 (7)	0.378 (3)	0.0620*	0.500
H62W	0.230 (7)	0.296 (7)	0.365 (3)	0.0620*	0.500

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2A	0.1102 (8)	0.0252 (4)	0.0356 (5)	0.0087 (5)	−0.0197 (5)	0.0018 (3)
Cl4A	0.0416 (5)	0.0557 (5)	0.0268 (4)	0.0087 (4)	−0.0071 (3)	0.0001 (4)
Mg1	0.0359 (5)	0.0197 (5)	0.0251 (5)	−0.0003 (4)	−0.0017 (4)	0.0002 (4)
O1W	0.0516 (14)	0.0262 (12)	0.0373 (13)	0.0055 (10)	−0.0121 (10)	−0.0075 (10)
O2W	0.0436 (13)	0.0400 (14)	0.0434 (14)	0.0202 (11)	−0.0164 (10)	−0.0166 (11)
O3W	0.0561 (14)	0.0208 (11)	0.0348 (13)	0.0009 (10)	−0.0169 (10)	−0.0010 (10)
O4W	0.0464 (13)	0.0290 (12)	0.0309 (12)	0.0021 (10)	0.0042 (10)	0.0036 (10)
O5W	0.082 (2)	0.095 (3)	0.0573 (19)	−0.0509 (18)	0.0218 (16)	−0.0094 (17)
O11A	0.0489 (12)	0.0209 (10)	0.0232 (10)	0.0040 (9)	−0.0023 (8)	0.0071 (8)
O13A	0.0628 (15)	0.0246 (12)	0.0295 (12)	−0.0012 (10)	−0.0020 (10)	0.0065 (9)
O14A	0.0907 (19)	0.0218 (12)	0.0419 (14)	0.0064 (12)	−0.0159 (12)	0.0106 (10)
C1A	0.0289 (15)	0.0283 (16)	0.0234 (15)	0.0018 (12)	0.0010 (11)	0.0055 (12)
C2A	0.0371 (17)	0.0261 (16)	0.0238 (15)	0.0014 (13)	0.0007 (12)	0.0020 (12)
C3A	0.0342 (16)	0.0314 (17)	0.0296 (16)	0.0079 (13)	0.0013 (12)	0.0088 (13)
C4A	0.0266 (15)	0.0413 (18)	0.0216 (15)	0.0028 (13)	−0.0005 (11)	0.0024 (13)
C5A	0.0363 (17)	0.0318 (17)	0.0324 (17)	−0.0004 (13)	−0.0006 (13)	−0.0040 (13)
C6A	0.0386 (17)	0.0265 (16)	0.0312 (17)	0.0007 (13)	0.0016 (13)	0.0066 (13)
C12A	0.0438 (18)	0.0188 (15)	0.0302 (16)	0.0040 (13)	0.0015 (13)	0.0070 (12)
C13A	0.0425 (18)	0.0249 (16)	0.0299 (17)	0.0063 (13)	0.0025 (13)	0.0065 (13)

Cl2B	0.0899 (7)	0.0302 (5)	0.0493 (5)	0.0140 (4)	0.0029 (5)	0.0068 (4)
Cl4B	0.0388 (5)	0.0653 (6)	0.0332 (4)	0.0054 (4)	−0.0018 (3)	0.0070 (4)
O11B	0.0491 (13)	0.0354 (13)	0.0278 (12)	0.0029 (10)	−0.0017 (9)	0.0037 (9)
O13B	0.0471 (14)	0.0538 (16)	0.0409 (14)	−0.0040 (12)	−0.0052 (10)	0.0117 (12)
O14B	0.0687 (17)	0.0742 (19)	0.0330 (14)	0.0209 (14)	−0.0021 (11)	−0.0153 (13)
C1B	0.0288 (16)	0.0316 (17)	0.0314 (17)	0.0016 (13)	0.0049 (12)	0.0051 (13)
C2B	0.0362 (17)	0.0245 (16)	0.0380 (18)	0.0055 (13)	0.0075 (13)	0.0051 (13)
C3B	0.0339 (17)	0.0333 (18)	0.0351 (18)	0.0024 (13)	0.0040 (13)	−0.0014 (13)
C4B	0.0259 (16)	0.0419 (19)	0.0298 (17)	0.0047 (13)	0.0018 (12)	0.0058 (14)
C5B	0.0335 (17)	0.0345 (18)	0.0389 (18)	0.0049 (14)	0.0021 (13)	0.0091 (14)
C6B	0.0411 (18)	0.0278 (17)	0.0380 (18)	0.0039 (14)	0.0017 (13)	0.0024 (14)
C12B	0.0383 (18)	0.0406 (19)	0.0339 (18)	0.0043 (14)	0.0042 (13)	0.0005 (14)
C13B	0.0361 (18)	0.060 (2)	0.0287 (18)	0.0055 (17)	0.0094 (13)	−0.0006 (17)
O6W	0.044 (3)	0.032 (3)	0.047 (3)	0.000 (2)	0.003 (2)	0.004 (2)

Geometric parameters (Å, º)

Mg1—O1W	2.065 (2)	O14B—C13B	1.270 (5)
Mg1—O4W	2.094 (2)	O6W—H61W	0.91 (6)
Mg1—O5W	2.053 (3)	O6W—H62W	0.90 (6)
Mg1—O14A	2.031 (2)	C1A—C6A	1.388 (4)
Mg1—O2W	2.067 (2)	C1A—C2A	1.396 (4)
Mg1—O3W	2.076 (2)	C2A—C3A	1.385 (4)
Cl2A—C2A	1.736 (3)	C3A—C4A	1.379 (4)
Cl4A—C4A	1.745 (3)	C4A—C5A	1.373 (4)
Cl2B—C2B	1.734 (3)	C5A—C6A	1.387 (4)
Cl4B—C4B	1.744 (3)	C12A—C13A	1.508 (4)
O11A—C1A	1.375 (3)	C3A—H3A	0.9500
O11A—C12A	1.432 (3)	C5A—H5A	0.9500
O13A—C13A	1.243 (4)	C6A—H6A	0.9500
O14A—C13A	1.258 (4)	C12A—H12A	0.9900
O1W—H12W	0.87 (3)	C12A—H13A	0.9900
O1W—H11W	0.86 (3)	C1B—C6B	1.386 (4)
O2W—H22W	0.87 (3)	C1B—C2B	1.402 (4)
O2W—H21W	0.88 (2)	C2B—C3B	1.381 (4)
O3W—H31W	0.870 (16)	C3B—C4B	1.375 (4)
O3W—H32W	0.87 (3)	C4B—C5B	1.378 (4)
O4W—H42W	0.88 (2)	C5B—C6B	1.393 (4)
O4W—H41W	0.89 (2)	C12B—C13B	1.520 (4)
O5W—H51W	0.90 (5)	C3B—H3B	0.9500
O5W—H52W	0.88 (4)	C5B—H5B	0.9500
O11B—C1B	1.366 (4)	C6B—H6B	0.9500
O11B—C12B	1.423 (4)	C12B—H13B	0.9900
O13B—C13B	1.235 (4)	C12B—H12B	0.9900
O1W—Mg1—O2W	87.62 (10)	C4A—C5A—C6A	119.5 (3)
O1W—Mg1—O3W	173.07 (10)	C1A—C6A—C5A	121.2 (3)
O1W—Mg1—O4W	87.94 (9)	O11A—C12A—C13A	111.3 (2)

O1W—Mg1—O5W	93.97 (12)	O14A—C13A—C12A	113.2 (3)
O1W—Mg1—O14A	96.53 (10)	O13A—C13A—C12A	121.7 (3)
O2W—Mg1—O3W	86.18 (9)	O13A—C13A—O14A	125.1 (3)
O2W—Mg1—O4W	90.97 (9)	C2A—C3A—H3A	121.00
O2W—Mg1—O5W	93.47 (12)	C4A—C3A—H3A	121.00
O2W—Mg1—O14A	175.40 (10)	C6A—C5A—H5A	120.00
O3W—Mg1—O4W	89.06 (9)	C4A—C5A—H5A	120.00
O3W—Mg1—O5W	89.51 (12)	C5A—C6A—H6A	119.00
O3W—Mg1—O14A	89.56 (10)	C1A—C6A—H6A	119.00
O4W—Mg1—O5W	175.23 (12)	O11A—C12A—H12A	109.00
O4W—Mg1—O14A	87.21 (10)	O11A—C12A—H13A	109.00
O5W—Mg1—O14A	88.23 (12)	C13A—C12A—H13A	109.00
C1A—O11A—C12A	115.3 (2)	H12A—C12A—H13A	108.00
Mg1—O14A—C13A	146.3 (2)	C13A—C12A—H12A	109.00
H11W—O1W—H12W	108 (3)	O11B—C1B—C2B	117.1 (3)
Mg1—O1W—H11W	124 (3)	O11B—C1B—C6B	124.8 (3)
Mg1—O1W—H12W	122 (2)	C2B—C1B—C6B	118.0 (3)
H21W—O2W—H22W	102 (3)	Cl2B—C2B—C1B	119.0 (2)
Mg1—O2W—H21W	127 (2)	Cl2B—C2B—C3B	119.6 (2)
Mg1—O2W—H22W	123 (2)	C1B—C2B—C3B	121.4 (3)
Mg1—O3W—H31W	134 (2)	C2B—C3B—C4B	119.2 (3)
Mg1—O3W—H32W	122 (2)	Cl4B—C4B—C3B	119.2 (2)
H31W—O3W—H32W	103 (3)	Cl4B—C4B—C5B	119.8 (2)
Mg1—O4W—H41W	119 (2)	C3B—C4B—C5B	121.0 (3)
Mg1—O4W—H42W	120 (2)	C4B—C5B—C6B	119.5 (3)
H41W—O4W—H42W	104 (3)	C1B—C6B—C5B	120.8 (3)
H51W—O5W—H52W	103 (4)	O11B—C12B—C13B	110.8 (3)
Mg1—O5W—H51W	93 (3)	O13B—C13B—C12B	122.0 (3)
Mg1—O5W—H52W	128 (3)	O14B—C13B—C12B	113.0 (3)
C1B—O11B—C12B	116.2 (2)	O13B—C13B—O14B	125.0 (3)
H61W—O6W—H62W	109 (6)	C2B—C3B—H3B	120.00
C2A—C1A—C6A	117.7 (3)	C4B—C3B—H3B	120.00
O11A—C1A—C6A	124.6 (3)	C4B—C5B—H5B	120.00
O11A—C1A—C2A	117.6 (2)	C6B—C5B—H5B	120.00
Cl2A—C2A—C1A	120.1 (2)	C5B—C6B—H6B	120.00
C1A—C2A—C3A	121.7 (3)	C1B—C6B—H6B	120.00
Cl2A—C2A—C3A	118.2 (2)	O11B—C12B—H12B	110.00
C2A—C3A—C4A	118.8 (3)	O11B—C12B—H13B	109.00
C3A—C4A—C5A	121.1 (3)	C13B—C12B—H13B	110.00
Cl4A—C4A—C3A	119.3 (2)	H12B—C12B—H13B	108.00
Cl4A—C4A—C5A	119.6 (2)	C13B—C12B—H12B	109.00

O1W—Mg1—O14A—C13A	56.7 (4)	C2A—C3A—C4A—C5A	−0.3 (4)
O3W—Mg1—O14A—C13A	−126.6 (4)	C3A—C4A—C5A—C6A	0.6 (5)
O4W—Mg1—O14A—C13A	144.3 (4)	Cl4A—C4A—C5A—C6A	179.2 (2)
O5W—Mg1—O14A—C13A	−37.1 (4)	C4A—C5A—C6A—C1A	−0.4 (4)
C12A—O11A—C1A—C2A	176.9 (3)	O11A—C12A—C13A—O13A	−5.8 (4)
C12A—O11A—C1A—C6A	−3.6 (4)	O11A—C12A—C13A—O14A	174.8 (2)

C1A—O11A—C12A—C13A	179.0 (2)	O11B—C1B—C2B—Cl2B	0.9 (4)
Mg1—O14A—C13A—O13A	−60.7 (5)	O11B—C1B—C2B—C3B	179.4 (3)
Mg1—O14A—C13A—C12A	118.7 (3)	C6B—C1B—C2B—Cl2B	−179.9 (2)
C12B—O11B—C1B—C2B	−176.6 (3)	C6B—C1B—C2B—C3B	−1.4 (4)
C12B—O11B—C1B—C6B	4.3 (4)	O11B—C1B—C6B—C5B	−179.1 (3)
C1B—O11B—C12B—C13B	175.7 (2)	C2B—C1B—C6B—C5B	1.7 (4)
C6A—C1A—C2A—C3A	0.2 (4)	Cl2B—C2B—C3B—C4B	178.9 (2)
O11A—C1A—C6A—C5A	−179.4 (3)	C1B—C2B—C3B—C4B	0.4 (5)
C2A—C1A—C6A—C5A	0.1 (4)	C2B—C3B—C4B—Cl4B	−179.7 (2)
O11A—C1A—C2A—C3A	179.7 (3)	C2B—C3B—C4B—C5B	0.3 (4)
C6A—C1A—C2A—Cl2A	−179.2 (2)	Cl4B—C4B—C5B—C6B	180.0 (2)
O11A—C1A—C2A—Cl2A	0.3 (4)	C3B—C4B—C5B—C6B	0.0 (5)
Cl2A—C2A—C3A—C4A	179.3 (2)	C4B—C5B—C6B—C1B	−1.1 (5)
C1A—C2A—C3A—C4A	−0.1 (5)	O11B—C12B—C13B—O13B	−1.9 (4)
C2A—C3A—C4A—Cl4A	−179.0 (2)	O11B—C12B—C13B—O14B	178.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11*W*···O13*B*	0.86 (3)	1.92 (3)	2.772 (3)	171 (3)
O1*W*—H12*W*···O4*W*^{i}	0.87 (3)	2.09 (3)	2.939 (3)	165 (3)
O2*W*—H21*W*···O14*B*	0.88 (2)	1.75 (2)	2.623 (3)	176 (3)
O2*W*—H22*W*···O14*B*^{ii}	0.87 (3)	1.88 (3)	2.754 (3)	173 (3)
O3*W*—H31*W*···O13*A*^{iii}	0.87 (2)	1.80 (2)	2.656 (3)	167 (3)
O3*W*—H32*W*···Cl2*A*^{iii}	0.87 (3)	2.50 (3)	3.345 (2)	165 (3)
O4*W*—H41*W*···O13*A*^{i}	0.89 (2)	1.77 (2)	2.652 (3)	172 (4)
O4*W*—H42*W*···O2*W*^{iv}	0.88 (2)	2.19 (3)	2.980 (3)	151 (3)
O5*W*—H51*W*···O6*W*^{v}	0.90 (5)	1.90 (6)	2.543 (5)	127 (4)
O5*W*—H52*W*···O13*B*^{vi}	0.88 (4)	1.86 (4)	2.708 (4)	162 (4)
O6*W*—H61*W*···O14*B*^{iv}	0.91 (6)	1.77 (6)	2.654 (5)	162 (7)
O6*W*—H62*W*···O14*A*	0.90 (6)	2.12 (5)	3.006 (5)	168 (5)

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