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Poly[[hexa-μ-aqua-diaquabis(μ_4-dihydrogen benzene-1,2,4,5-tetracarboxylato)magnesiumdisodium] dihydrate]

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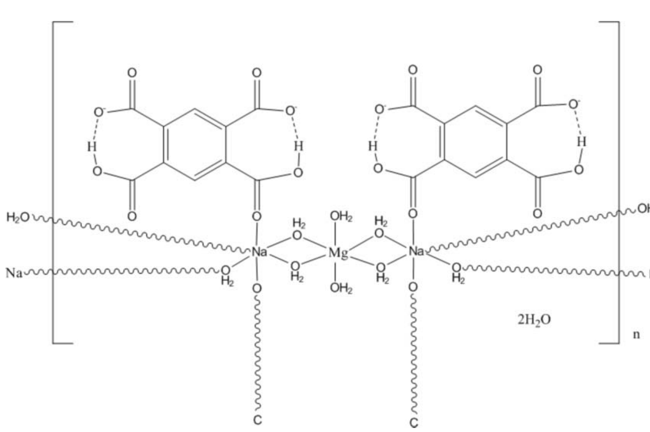
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.089; data-to-parameter ratio = 11.8.

The asymmetric unit of the title compound, $\{[MgNa_2(C_{10}H_4O_8)_2(H_2O)_8]\cdot 2H_2O\}_n$, contains one octahedrally coordinated Mg^{II} atom (site symmetry $2/m$), one octahedrally coordinated Na^{I} atom (site symmetry 2) and one half of the dihydrogen benzene-1,2,4,5-tetracarboxylate (btec) ligand, the second half of the ligand being generated by a twofold rotation axis. The basic framework of the title compound features infinite (–Na–Na–Mg–)$_n$ chains along [10$\overline{1}$] with the metal cations bridged by the coordinating water molecules. The chains are isolated from each other by μ_4-bridging btec ligands, which form intermolecular O—H···O hydrogen bonds to uncoordinated water molecules and the coordinated water molecules of a neighbouring chain. In each btec ligand, there are also intramolecular O—H···O hydrogen bonds.

Related literature

For structures based on the H_4btec ligand, see: Gong & Zhang (2011); Liu *et al.* (2009, 2010); Zhang *et al.* (2007).



Experimental

Crystal data

$[MgNa_2(C_{10}H_4O_8)_2(H_2O)_8]\cdot 2H_2O$	V = 1502.3 (5) Å^3
M_r = 754.71	Z = 2
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
a = 7.3335 (13) Å	μ = 0.20 mm^{-1}
b = 20.155 (4) Å	T = 296 K
c = 10.4450 (18) Å	0.20 × 0.05 × 0.05 mm
β = 103.325 (3)°	

Data collection

Bruker APEXII CCD area-detector diffractometer	4088 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.961, T_{max} = 0.990	1440 independent reflections
	1272 reflections with $I > 2\sigma(I)$
	R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030	122 parameters
$wR(F^2)$ = 0.089	H-atom parameters constrained
S = 1.08	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
1440 reflections	$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	1.06	1.35	2.3827 (17)	163
O5—H5···O7	0.90	1.83	2.7313 (14)	173
O6—H6···O4^{i}	0.90	1.97	2.8519 (15)	167
O7—H7···O2^{ii}	0.86	1.91	2.7699 (14)	173
O8—H8···O3^{iii}	0.85	1.94	2.7779 (14)	172
O9—H9···O4^{ii}	0.85	1.91	2.7559 (14)	171

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2296).

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supplementary materials

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Poly[[hexa-μ-aqua-diaquabis(μ_4-dihydrogen benzene-1,2,4,5-tetracarboxylato)magnesiumdisodium] dihydrate]

Dan Zhao, Peng Liang, Yan-Feng Li, Sen Qiu and Jun-Ran Ren

Comment

In recent years, much attention has been paid to coordination polymer materials based on covalent interactions or supramolecular contacts, and huge numbers of novel compounds with interesting structures and topologies have been reported. As part of this research benzene-1,2,4,5-tetracarboxylate (btec) can be used as a ligand to form various supramolecular architectures with its four rigid carboxyl groups (Gong *et al.*, 2011; Liu *et al.*, 2009; Liu *et al.*, 2010; Zhang *et al.*, 2007). In order to enrich this family of compounds, we used the hydrothermal method to synthesise the title compound, a new sodium(I)-magnesium(II) complex, that is, $Na_2Mg(btec)_2(H_2O)_8.2(H_2O)$, where btec = benzene-1,2,4,5-tetracarboxylate, and we determined its structure by single-crystal X-ray diffraction.

As shown in Fig. 1, the asymmetric unit of the title compound contains one octahedrally coordinated magnesium atom, one octahedrally coordinated sodium atom and half a benzene-1,2,4,5-tetracarboxylate (btec) ligand. Each btec ligands contains two intramolecular O–H···O hydrogen bonds, with the H atoms bonded to atoms O3 and O2, and connects two Na atoms in a μ_2– manner. Each Na atom is coordinated by two *cis* carboxylate oxygen atoms from two btec ligands and by four water molecules, while each Mg is coordinated by six water molecules. The Na–O bond distances range from 2.2669 (12) to 2.6146 (18) Å, while the Mg–O bond lengths are slightly longer ranging from 2.0301 (14) to 2.1008 (14) Å. Furthermore, NaO_6 octahedra and MgO_6 octahedra are connected *via* coordinated water molecules to form a one-dimensional infinite $(–Na–Na–Mg–)_n$ chain, as shown in Fig. 2. O—H···O hydrogen bonds link the coordinated and uncoordinated water molecules to neighbouring btec ligands (Table 1).

Experimental

A mixture of 1,2,4,5-benzene-tetracarboxylic (0.2 g), Na_2CO3 (0.1 g), MgO(0.05 g) and H_2O (15 ml) was heated at 448 K for 7 d in a sealed 25 ml Teflon-lined stainless steel vessel under autogenous pressure. After cooling to room temperature at a rate of 5 C h^{-1}, colourless prismatic crystals were obtained in low yield.

Refinement

The H atoms of C atoms were positioned geometrically and refined with a riding model, with C—H = 0.93 Å and U_{iso}(H) = $1.2U_{eq}$(C). The water H atoms were located in difference Fourier maps, and then refined with a riding model, with U_{iso}(H) = $1.5U_{eq}$(O).

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication:

SHELXTL (Sheldrick, 2008).

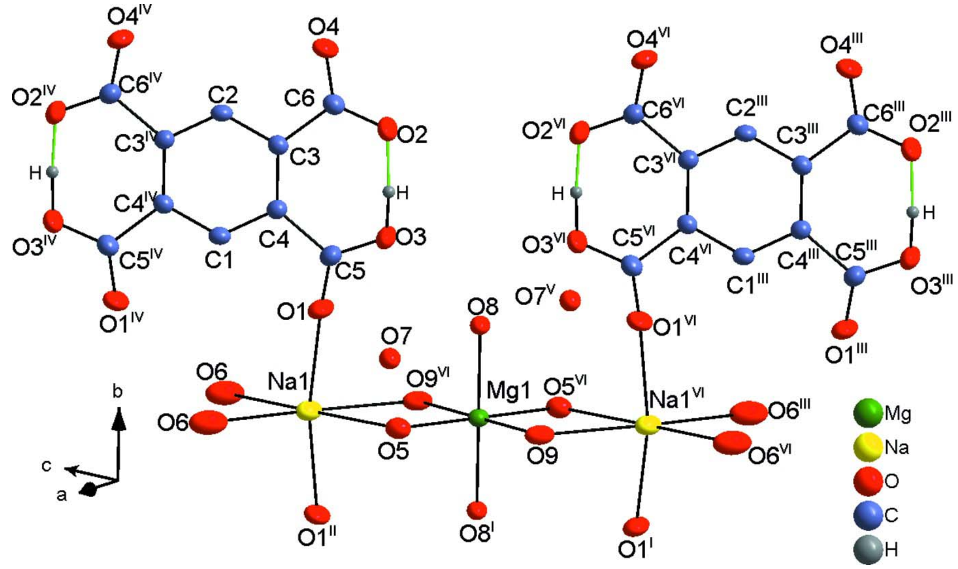

Figure 1

A view of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and hydrogen atoms are omitted for clarity, except for intramolecular hydrogen bonding H atoms in the btec ligands (indicated as pea green lines). [Symmetry codes: (i) -*x*, -*y*, -*z*; (ii) *x*, -*y*, *z*; (iii) *x* - 1, *y*, *z* - 1; (iv) -*x* + 1, *y*, -*z* + 1; (v) *x* - 1, *y*, *z*; (vi) -*x*, *y*, -*z*].

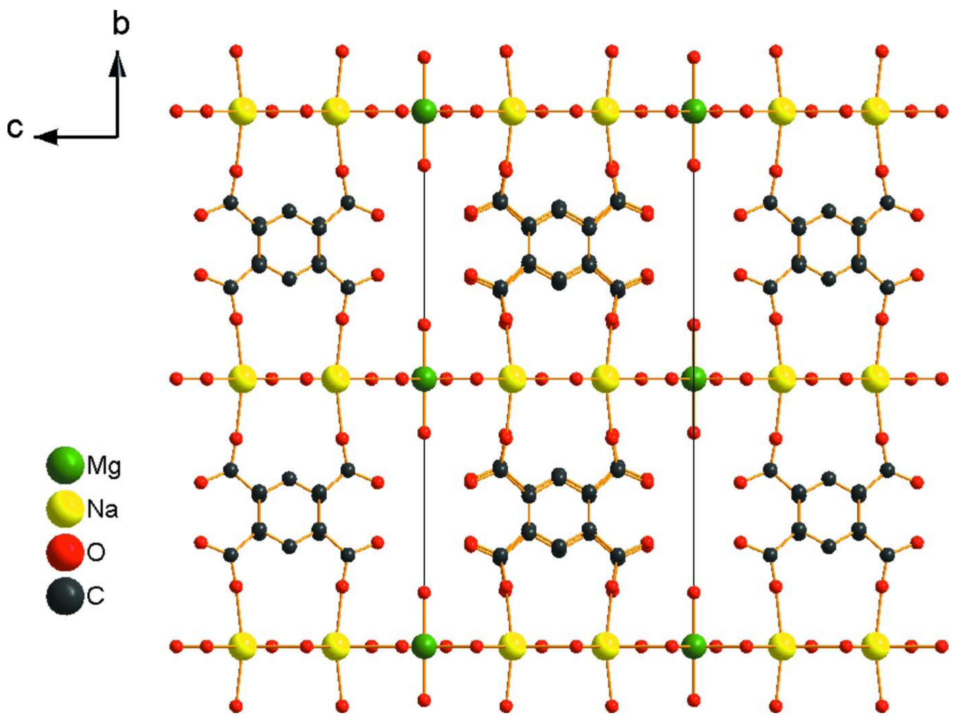

Figure 2

View of the crystal structure of the title compound along the *a*-axis.

Poly[[hexa-μ-aqua-diaquabis(μ_4-dihydrogen benzene-1,2,4,5-tetracarboxylato)magnesium(II)disodium] dihydrate]

Crystal data

$[MgNa_2(C_{10}H_4O_8)_2(H_2O)_8]\cdot 2H_2O$	$F(000) = 780$
$M_r = 754.71$	$D_x = 1.668$ Mg m^{-3}
Monoclinic, $C2/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2y	Cell parameters from 2031 reflections
$a = 7.3335$ (13) Å	$\theta = 2.9$–27.8°
$b = 20.155$ (4) Å	$\mu = 0.20$ mm^{-1}
$c = 10.4450$ (18) Å	$T = 296$ K
$\beta = 103.325$ (3)°	Prism, colourless
$V = 1502.3$ (5) Å^3	0.20 × 0.05 × 0.05 mm
$Z = 2$	

Data collection

Bruker APEXII CCD area-detector diffractometer	4088 measured reflections
Radiation source: fine-focus sealed tube	1440 independent reflections
Graphite monochromator	1272 reflections with $I > 2\sigma(I)$
Detector resolution: 83.33 pixels mm^{-1}	$R_{int} = 0.022$
ω scans	$\theta_{max} = 25.5°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -8\rightarrow 8$
$T_{min} = 0.961$, $T_{max} = 0.990$	$k = -24\rightarrow 22$
	$l = -11\rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 0.7471P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.089$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
1440 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}
122 parameters	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.0033 (7)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Mg1	0.0000	0.0000	0.0000	0.0220 (2)

Na1	0.31699 (13)	0.0000	0.32732 (8)	0.0356 (3)
O1	0.30603 (18)	0.11183 (5)	0.30357 (11)	0.0464 (3)
C1	0.5000	0.18685 (9)	0.5000	0.0233 (4)
H1	0.5000	0.1407	0.5000	0.028*
O2	0.20225 (19)	0.31210 (6)	0.17351 (10)	0.0487 (4)
C2	0.5000	0.32051 (9)	0.5000	0.0221 (4)
H2	0.5000	0.3667	0.5000	0.027*
O3	0.20002 (18)	0.19389 (6)	0.17286 (10)	0.0481 (4)
H3	0.2132	0.2456	0.1607	0.072*
C3	0.39709 (18)	0.28869 (6)	0.38857 (12)	0.0211 (3)
O4	0.31030 (15)	0.39579 (5)	0.30165 (10)	0.0339 (3)
C4	0.39692 (18)	0.21864 (6)	0.38884 (12)	0.0214 (3)
O5	0.28991 (19)	0.0000	0.08188 (14)	0.0288 (3)
H5	0.3530	0.0340	0.0564	0.043*
C5	0.2960 (2)	0.17086 (7)	0.28270 (14)	0.0283 (3)
O6	0.3317 (3)	0.0000	0.56419 (17)	0.0483 (5)
H6	0.2884	−0.0368	0.5963	0.073*
C6	0.29702 (19)	0.33570 (7)	0.28103 (13)	0.0258 (3)
O7	0.5000	0.09605 (7)	0.0000	0.0311 (3)
H7	0.4367	0.1223	−0.0590	0.047*
O8	0.0000	0.10073 (7)	0.0000	0.0322 (4)
H8	−0.0681	0.1262	−0.0560	0.048*
O9	0.0320 (2)	0.0000	−0.19461 (14)	0.0314 (4)
H9	0.0897	0.0325	−0.2191	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mg1	0.0265 (5)	0.0161 (4)	0.0222 (5)	0.000	0.0032 (4)	0.000
Na1	0.0521 (6)	0.0188 (4)	0.0328 (5)	0.000	0.0034 (4)	0.000
O1	0.0678 (8)	0.0176 (6)	0.0428 (7)	−0.0038 (5)	−0.0098 (6)	−0.0044 (5)
C1	0.0259 (10)	0.0152 (9)	0.0275 (10)	0.000	0.0037 (8)	0.000
O2	0.0697 (8)	0.0256 (6)	0.0343 (6)	0.0023 (5)	−0.0222 (6)	0.0038 (5)
C2	0.0262 (9)	0.0136 (9)	0.0258 (10)	0.000	0.0046 (8)	0.000
O3	0.0683 (8)	0.0244 (6)	0.0350 (6)	−0.0028 (5)	−0.0224 (6)	−0.0032 (5)
C3	0.0216 (7)	0.0186 (7)	0.0225 (7)	0.0007 (5)	0.0038 (5)	0.0014 (5)
O4	0.0478 (7)	0.0177 (5)	0.0335 (6)	0.0032 (4)	0.0040 (5)	0.0050 (4)
C4	0.0215 (6)	0.0185 (7)	0.0231 (7)	−0.0010 (5)	0.0031 (5)	−0.0012 (5)
O5	0.0280 (7)	0.0221 (7)	0.0363 (8)	0.000	0.0070 (6)	0.000
C5	0.0318 (8)	0.0214 (7)	0.0282 (7)	−0.0012 (6)	0.0000 (6)	−0.0036 (6)
O6	0.0713 (12)	0.0291 (8)	0.0533 (11)	0.000	0.0321 (9)	0.000
C6	0.0282 (7)	0.0211 (7)	0.0263 (7)	0.0011 (5)	0.0028 (6)	0.0028 (6)
O7	0.0357 (8)	0.0214 (7)	0.0304 (8)	0.000	−0.0046 (6)	0.000
O8	0.0419 (9)	0.0162 (7)	0.0299 (8)	0.000	−0.0094 (6)	0.000
O9	0.0469 (9)	0.0185 (7)	0.0324 (8)	0.000	0.0166 (7)	0.000

Geometric parameters (Å, º)

Mg1—O8	2.0301 (14)	O2—C6	1.2691 (17)
Mg1—O8^{i}	2.0302 (14)	O2—H3	1.3505

Mg1—O9	2.0992 (14)	C2—C3	1.3895 (15)
Mg1—O9^{i}	2.0992 (14)	C2—C3^{iv}	1.3895 (15)
Mg1—O5^{i}	2.1008 (14)	C2—H2	0.9300
Mg1—O5	2.1008 (14)	O3—C5	1.2865 (17)
Mg1—Na1^{i}	3.6645 (10)	O3—H3	1.0579
Mg1—Na1	3.6646 (10)	C3—C4	1.4119 (18)
Na1—O1	2.2669 (12)	C3—C6	1.5225 (18)
Na1—O1^{ii}	2.2669 (12)	O4—C6	1.2301 (17)
Na1—O6	2.4515 (19)	C4—C5	1.5250 (18)
Na1—O5	2.5252 (16)	O5—H5	0.9007
Na1—O6^{iii}	2.564 (2)	O6—Na1^{iii}	2.564 (2)
Na1—O9^{i}	2.6146 (18)	O6—H6	0.9011
Na1—Na1^{iii}	3.9692 (17)	O7—H7	0.8628
O1—C5	1.2088 (18)	O8—H8	0.8481
C1—C4	1.3876 (15)	O9—Na1^{i}	2.6146 (17)
C1—C4^{iv}	1.3876 (15)	O9—H9	0.8510
C1—H1	0.9300		

O8—Mg1—O8^{i}	180.0	O1^{ii}—Na1—Mg1	84.21 (4)
O8—Mg1—O9	90.0	O6—Na1—Mg1	144.34 (6)
O8^{i}—Mg1—O9	90.0	O5—Na1—Mg1	33.73 (3)
O8—Mg1—O9^{i}	90.0	O6^{iii}—Na1—Mg1	140.26 (5)
O8^{i}—Mg1—O9^{i}	90.0	O9^{i}—Na1—Mg1	34.15 (3)
O9—Mg1—O9^{i}	180.00 (8)	O1—Na1—Na1^{iii}	95.68 (4)
O8—Mg1—O5^{i}	90.0	O1^{ii}—Na1—Na1^{iii}	95.68 (4)
O8^{i}—Mg1—O5^{i}	90.0	O6—Na1—Na1^{iii}	38.69 (5)
O9—Mg1—O5^{i}	86.23 (6)	O5—Na1—Na1^{iii}	143.24 (5)
O9^{i}—Mg1—O5^{i}	93.77 (6)	O6^{iii}—Na1—Na1^{iii}	36.70 (4)
O8—Mg1—O5	90.0	O9^{i}—Na1—Na1^{iii}	148.88 (5)
O8^{i}—Mg1—O5	90.0	Mg1—Na1—Na1^{iii}	176.97 (4)
O9—Mg1—O5	93.77 (6)	C5—O1—Na1	175.97 (11)
O9^{i}—Mg1—O5	86.23 (6)	C4—C1—C4^{iv}	125.01 (18)
O5^{i}—Mg1—O5	180.0	C4—C1—H1	117.5
O8—Mg1—Na1^{i}	90.0	C4^{iv}—C1—H1	117.5
O8^{i}—Mg1—Na1^{i}	90.0	C6—O2—H3	115.2
O9—Mg1—Na1^{i}	44.36 (4)	C3—C2—C3^{iv}	125.01 (17)
O9^{i}—Mg1—Na1^{i}	135.64 (4)	C3—C2—H2	117.5
O5^{i}—Mg1—Na1^{i}	41.87 (4)	C3^{iv}—C2—H2	117.5
O5—Mg1—Na1^{i}	138.13 (4)	C5—O3—H3	114.6
O8—Mg1—Na1	90.0	C2—C3—C4	117.42 (12)
O8^{i}—Mg1—Na1	90.0	C2—C3—C6	114.02 (12)
O9—Mg1—Na1	135.64 (4)	C4—C3—C6	128.56 (11)
O9^{i}—Mg1—Na1	44.36 (4)	C1—C4—C3	117.57 (12)
O5^{i}—Mg1—Na1	138.13 (4)	C1—C4—C5	113.34 (13)
O5—Mg1—Na1	41.87 (4)	C3—C4—C5	129.09 (11)
Na1^{i}—Mg1—Na1	180.0	Mg1—O5—Na1	104.40 (6)
O1—Na1—O1^{ii}	167.72 (7)	Mg1—O5—H5	114.8
O1—Na1—O6	95.89 (4)	Na1—O5—H5	112.0
O1^{ii}—Na1—O6	95.89 (4)	O1—C5—O3	120.93 (13)

O1—Na1—O5	84.05 (4)	O1—C5—C4	119.43 (13)
O1^{ii}—Na1—O5	84.05 (4)	O3—C5—C4	119.64 (12)
O6—Na1—O5	178.07 (7)	Na1—O6—Na1^{iii}	104.61 (6)
O1—Na1—O6^{iii}	93.16 (4)	Na1—O6—H6	116.0
O1^{ii}—Na1—O6^{iii}	93.16 (4)	Na1^{iii}—O6—H6	103.9
O6—Na1—O6^{iii}	75.39 (6)	O4—C6—O2	121.94 (13)
O5—Na1—O6^{iii}	106.54 (6)	O4—C6—C3	118.58 (12)
O1—Na1—O9^{i}	86.35 (4)	O2—C6—C3	119.48 (12)
O1^{ii}—Na1—O9^{i}	86.35 (4)	Mg1—O8—H8	127.2
O6—Na1—O9^{i}	110.19 (6)	Mg1—O9—Na1^{i}	101.49 (6)
O5—Na1—O9^{i}	67.88 (5)	Mg1—O9—H9	117.8
O6^{iii}—Na1—O9^{i}	174.41 (6)	Na1^{i}—O9—H9	109.5
O1—Na1—Mg1	84.21 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2	1.06	1.35	2.3827 (17)	163
O5—H5···O7	0.90	1.83	2.7313 (14)	173
O6—H6···O4^{v}	0.90	1.97	2.8519 (15)	167
O7—H7···O2^{vi}	0.86	1.91	2.7699 (14)	173
O8—H8···O3^{vii}	0.85	1.94	2.7779 (14)	172
O9—H9···O4^{vi}	0.85	1.91	2.7559 (14)	171

Symmetry codes: (v) −*x*+1/2, *y*−1/2, −*z*+1; (vi) −*x*+1/2, −*y*+1/2, −*z*; (vii) −*x*, *y*, −*z*.