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Crystal structures of $Ca(ClO_4)_2{\cdot}4H_2O$ and $Ca(ClO_4)_2{\cdot}6H_2O$

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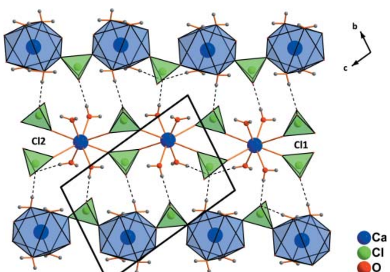
The title compounds, calcium perchlorate tetrahydrate and calcium perchlorate hexahydrate, were crystallized at low temperatures according to the solid–liquid phase diagram. The structure of the tetrahydrate consists of one Ca^{2+} cation eightfold coordinated in a square-antiprismatic fashion by four water molecules and four O atoms of four perchlorate tetrahedra, forming chains parallel to $[01\bar{1}]$ by sharing corners of the ClO_4 tetrahedra. The structure of the hexahydrate contains two different Ca^{2+} cations, each coordinated by six water molecules and two O atoms of two perchlorate tetrahedra, forming $[Ca(H_2O)_6(ClO_4)]_2$ dimers by sharing two ClO_4 tetrahedra. The dimers are arranged in sheets parallel (001) and alternate with layers of non-coordinating ClO_4 tetrahedra. O—H⋯O hydrogen bonds between the water molecules as donor and ClO_4 tetrahedra and water molecules as acceptor groups lead to the formation of a three-dimensional network in the two structures. $Ca(ClO_4)_2{\cdot}6H_2O$ was refined as a two-component inversion twin, with an approximate twin component ratio of 1:1 in each of the two structures.



1. Chemical context

Since the detection of perchlorates on Mars during the Phoenix Mission (Chevrier *et al.*, 2009), interest in these salts, and especially their hydrates, has risen considerably (Kim *et al.*, 2013; Quinn *et al.*, 2013; Kerr, 2013; Davila *et al.*, 2013; Schuttlefield *et al.*, 2011; Navarro-González *et al.*, 2010; Marion *et al.*, 2010). To gain more knowledge about the behavior of salts and salt hydrates, it is essential to determine the corresponding phase diagrams. For calcium perchlorate, this was performed by several authors (Marion *et al.*, 2010; Pestova *et al.*, 2005; Dobrynina, 1984; Lilich & Djurinskii, 1956; Nicholson & Felsing, 1950; Willard & Smith, 1923) for different concentration areas with different results. The stable salt hydrate phase at room temperature in this system is calcium perchlorate tetrahydrate. At lower temperatures, a higher hydrated phase, *i.e.* the hexahydrate, occurs as the stable phase.

2. Structural commentary

The Ca^{2+} cation in $Ca(ClO_4){\cdot}4H_2O$ is coordinated by four water molecules (O1, O2, O7, O8) and four O atoms from two pairs of symmetry-related perchlorate tetrahedra as shown in Fig. 1*a*. The resulting coordination polyhedron is a distorted square anti-prism (Fig. 1*b*). The Ca—O bond lengths involving the water molecules range from 2.3284 (17) to 2.4153 (16) Å and are considerably shorter than the Ca—O bond lengths involving the perchlorate O atoms [2.5417 (16) to 2.5695 (17) Å].

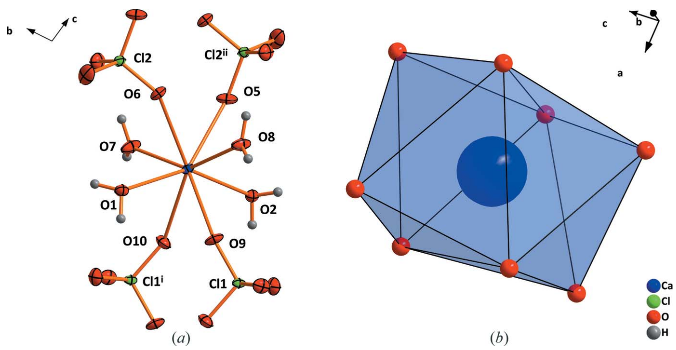


Figure 1
(*a*) The principle building block in the structure of $Ca(ClO_4)_2·4H_2O$ and (*b*) the square anti-prismatic coordination of Ca^{2+}. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $1 - x, 1 - y, 2 - z$.]

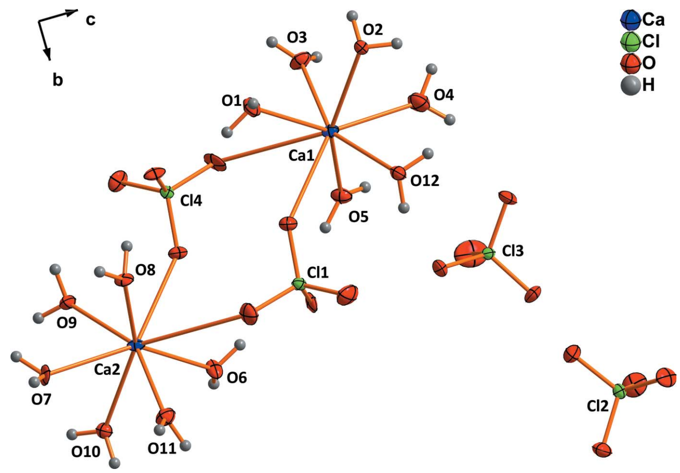


Figure 2
The principle building blocks in the structure of $Ca(ClO_4)_2·6H_2O$. Displacement ellipsoids are drawn at the 50% probability level.

The two different Ca^{2+} cations in $Ca(ClO_4)·6H_2O$ are each coordinated by six water molecules and two perchlorate tetrahedra (Fig. 2). Again, the bond lengths between the cations and water molecules [2.319 (6)–2.500 (6) Å] are shorter than those to the perchlorate groups. For the latter, one of the two distances for each of the Ca^{2+} cations is by 0.5 Å markedly longer than the other (∼3.07 *versus* ∼2.53 Å). Nevertheless, according to the bond-valence model (Brown, 2002), the longer bond contributes *ca.* 0.05 valence units to the overall bond-valence sum and hence should not be neglected. If this longer bond is considered to be relevant, again a square anti-prismatic coordination polyhedron is realised for both Ca^{2+} cations, however with a much greater distortion. Two perchlorate tetrahedra in the hexahydrate are shared between two Ca^{2+} ions, leading to the formation of $[Ca(H_2O)_6(ClO_4)]_2$ dimers oriented in layers parallel to (001).

3. Supramolecular features

The perchlorate tetrahedra in the structure of $Ca(ClO_4)·4H_2O$ are shared between two adjacent Ca^{2+} ions, forming chains extending parallel to $[01\bar{1}]$ (Fig. 3) whereby each Ca^{2+} ion is connected along the chain on one side with a pair of Cl1 perchlorate tetrahedra, and on the opposite side with a pair of Cl2 perchlorate tetrahedra. The chains are arranged in sheets parallel to $(0\bar{1}1)$ and are linked by O—H···O hydrogen bonds into a three-dimensional network with similar O···O distances between the water molecules and the perchlorate tetrahedra (Table 1).

In addition to the two coordinating perchlorate tetrahedra in $Ca(ClO_4)·6H_2O$, two 'free' perchlorate tetrahedra are present in the crystal structure. These 'free' ClO_4 tetrahedra are arranged in sheets and alternate with the $[Ca(H_2O)_6(ClO_4)]_2$ sheets along [001] (Fig. 4). The 'free' perchlorate tetrahedra are connected to the dimers *via* O—H···O hydrogen bonds, as shown in Fig. 4. The dimers are additionally connected through further O—H···O hydrogen bonds (Table 2) into a three-dimensional network (Fig. 5).

Table 1
Hydrogen-bond geometry (Å, °) for $Ca(ClO_4)_2·4H_2O$.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O11^{i}	0.82 (1)	2.11 (2)	2.888 (2)	158 (3)
O1—H1*A*···O3^{ii}	0.82 (1)	2.13 (1)	2.947 (2)	174 (3)
O2—H2*A*···O11^{iii}	0.82 (1)	2.17 (2)	2.947 (2)	159 (3)
O2—H2*B*···O4^{iv}	0.82 (1)	2.02 (1)	2.830 (2)	172 (3)
O7—H7*B*···O4	0.81 (1)	2.22 (2)	2.924 (2)	146 (3)
O7—H7*A*···O1^{iii}	0.82 (1)	2.06 (1)	2.870 (2)	172 (3)
O8—H8*A*···O4^{v}	0.82 (1)	2.33 (3)	2.986 (2)	137 (4)
O8—H8*B*···O2^{vi}	0.82 (1)	2.14 (1)	2.950 (2)	169 (5)

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

4. Database survey

For crystal structures of other $M(ClO_4)_2·4H_2O$ phases, see: Robertson & Bish (2010; M = Mg); Hennings *et al.* (2014; Sr);

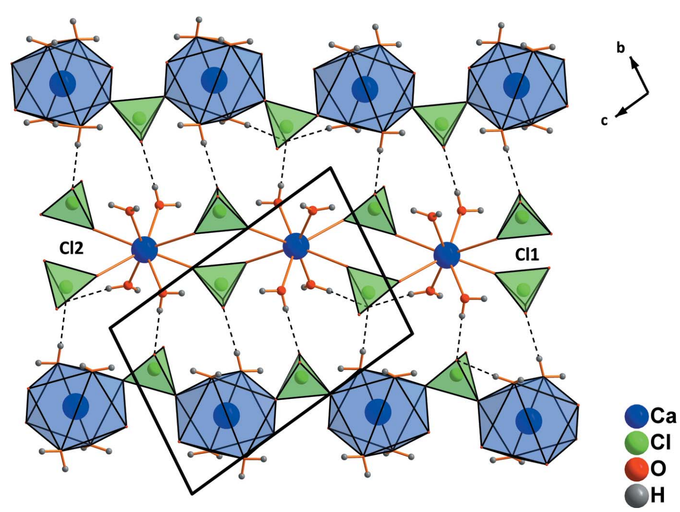


Figure 3
Formation of sheets and interconnection of chains *via* hydrogen bonds in $Ca(ClO_4)_2·4H_2O$. Only the strongest hydrogen bonds are shown, represented by dashed lines.

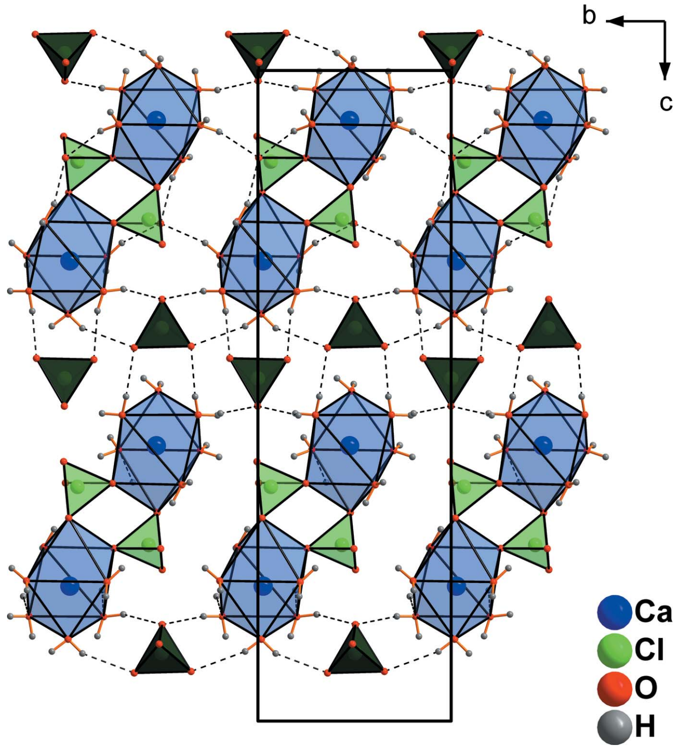


Figure 4
Formation of perchlorate-bridged dimers in $Ca(ClO_4)_2{\cdot}6H_2O$ and location of 'free' perchlorate tetrahedra in the gaps between the dimers (highlighted in dark green). Only the strongest hydrogen bonds are shown, represented by dashed lines.

Table 2
Hydrogen-bond geometry (Å, °) for $Ca(ClO_4)_2{\cdot}6H_2O$.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1A⋯O15	0.84 (2)	2.07 (3)	2.887 (10)	164 (8)
O1—H1B⋯O5^{i}	0.84 (2)	2.25 (5)	2.915 (7)	136 (6)
O1—H1B⋯O16^{i}	0.84 (2)	2.44 (5)	3.132 (10)	140 (6)
O2—H2A⋯O23^{ii}	0.84 (2)	2.03 (2)	2.856 (9)	169 (7)
O2—H2B⋯O26^{iii}	0.84 (2)	2.14 (3)	2.932 (8)	155 (6)
O3—H3A⋯O12^{iv}	0.84 (2)	2.07 (2)	2.899 (8)	168 (8)
O3—H3B⋯O19^{iii}	0.84 (2)	2.15 (3)	2.934 (8)	156 (7)
O4—H4A⋯O27	0.84 (2)	2.28 (3)	3.074 (11)	158 (8)
O4—H4B⋯O28^{iii}	0.84 (2)	2.36 (3)	3.177 (10)	163 (8)
O5—H5A⋯O2^{iv}	0.84 (2)	1.98 (3)	2.783 (8)	159 (7)
O5—H5B⋯O19	0.84 (2)	2.20 (5)	2.903 (9)	142 (6)
O6—H6A⋯O8^{v}	0.84 (2)	2.18 (4)	2.925 (7)	149 (7)
O6—H6B⋯O19	0.84 (2)	2.08 (3)	2.891 (10)	162 (8)
O7—H7A⋯O23^{vi}	0.84 (2)	2.29 (4)	3.042 (9)	149 (6)
O7—H7B⋯O24^{vii}	0.84 (2)	2.50 (5)	3.199 (9)	141 (6)
O7—H7B⋯O27^{viii}	0.84 (2)	2.57 (5)	3.242 (11)	138 (6)
O8—H8A⋯O10^{ix}	0.84 (2)	2.08 (4)	2.805 (8)	145 (6)
O8—H8B⋯O15	0.84 (2)	2.07 (3)	2.879 (9)	162 (7)
O9—H9A⋯O27^{x}	0.84 (2)	2.06 (3)	2.865 (10)	161 (7)
O9—H9B⋯O21^{vi}	0.84 (2)	2.23 (5)	2.962 (10)	145 (7)
O10—H10A⋯O21^{vii}	0.84 (2)	2.12 (3)	2.930 (9)	163 (7)
O10—H10B⋯O28^{x}	0.84 (2)	2.10 (3)	2.902 (10)	162 (7)
O11—H11A⋯O9^{ix}	0.84 (2)	2.14 (4)	2.893 (9)	150 (7)
O11—H11B⋯O15^{xi}	0.84 (2)	2.11 (3)	2.915 (9)	161 (7)
O12—H12A⋯O26	0.84 (2)	2.35 (5)	2.995 (9)	135 (6)
O12—H12A⋯O20	0.84 (2)	2.40 (4)	3.102 (9)	142 (6)
O12—H12B⋯O24^{ii}	0.84 (2)	2.03 (2)	2.861 (9)	171 (7)

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

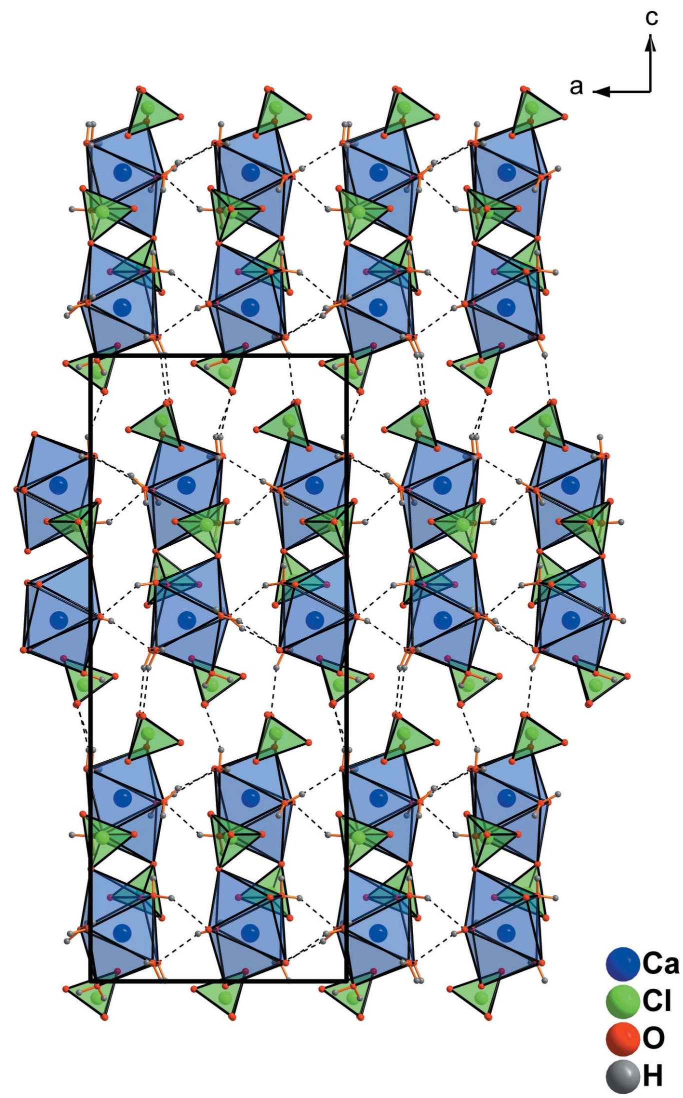


Figure 5
Formation of layers parallel to (001) in $Ca(ClO_4)_2{\cdot}6H_2O$. Only the strongest hydrogen bonds are shown, represented by dashed lines.

Solovyov (2012; Mg); Johansson (1966; Hg). For crystal structures of other $M(ClO_4)_2{\cdot}6H_2O$ phases, see: Ghosh *et al.* (1997; M = Ni, Zn); Ghosh & Ray (1981; Fe); Johansson *et al.* (1978; Hg); Mani & Ramaseshan (1961; Cu); Johansson & Sandström (1987; Cd); Gallucci & Gerkin (1989; Cu); West (1935; Mg).

5. Synthesis and crystallization

$Ca(ClO_4)_2{\cdot}4H_2O$ was crystallized from an aqueous solution of 62.96 wt% $Ca(ClO_4)_2$ at 273 K after one day and $Ca(ClO_4)_2{\cdot}6H_2O$ from an aqueous solution of 57.55 wt% $Ca(ClO_4)_2$ at 238 K after one week. For the preparation of these aqueous solutions, $Ca(ClO_4)_2{\cdot}4H_2O$ (Acros Organics, p.A.) was used. The Ca^{2+} content was analysed *via* complexometric titration with EDTA. The crystals remain stable in the saturated aqueous solution over at least four weeks.

Table 3
Experimental details.

	$Ca(ClO_4)_2·4H_2O$	$Ca(ClO_4)_2·6H_2O$
Crystal data		
M_r	311.04	347.08
Crystal system, space group	Triclinic, $P\overline{1}$	Orthorhombic, $Pca2_1$
Temperature (K)	200	180
a, b, c (Å)	5.4886 (11), 7.8518 (15), 11.574 (2)	10.9603 (4), 7.9667 (7), 26.7735 (18)
α, β, γ (°)	99.663 (16), 90.366 (16), 90.244 (16)	90, 90, 90
V (Å^3)	491.71 (17)	2337.8 (3)
Z	2	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	1.24	1.06
Crystal size (mm)	0.04 × 0.03 × 0.02	0.38 × 0.31 × 0.08
Data collection		
Diffractometer	Stoe IPDS2	Stoe IPDS2
Absorption correction	Integration Coppens (1970)	Integration (Coppens, 1970)
T_{min}, T_{max}	0.644, 0.789	0.684, 0.923
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	2659, 2636, 2529	15755, 5326, 4919
R_{int}	0.074	0.062
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.686	0.650
Refinement		
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.031, 0.089, 1.20	0.042, 0.113, 1.09
No. of reflections	2636	5326
No. of parameters	168	380
No. of restraints	12	37
H-atom treatment	All H-atom parameters refined	Only H-atom coordinates refined
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.36, −0.75	0.41, −0.67
Absolute structure	–	Refined as an inversion twin
Absolute structure parameter	–	0.45 (9)

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 2009), *SHELXS97* and *SHELXL2012* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 2006) and *publCIF* (Westrip, 2010).

The samples were stored in a freezer or a cryostat at low temperatures. The crystals were separated and embedded in perfluorinated ether for X-ray analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms of each structure were placed in the positions indicated by difference Fourier maps. For $Ca(ClO_4)_2·4H_2O$, distance restraints were applied for all water molecules, with O—H and H—H distance restraints of 0.82 (1) and 1.32 (1) Å, respectively. For $Ca(ClO_4)_2·6H_2O$, U_{iso} values were set at $1.2U_{eq}$(O) using a riding-model approximation. Distance restraints were applied for that structure for all water molecules, with O—H and H—H distance restraints of 0.84 (2) and 1.4 (2) Å, respectively. $Ca(ClO_4)_2·6H_2O$ was refined as a two-component inversion twin, with an approximate twin component ratio of 1:1.

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supporting information

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Crystal structures of $Ca(ClO_4)_2·4H_2O$ and $Ca(ClO_4)_2·6H_2O$

Erik Hennings, Horst Schmidt and Wolfgang Voigt

Computing details

For both compounds, data collection: *X-AREA* (Stoe & Cie, 2009); cell refinement: *X-AREA*(Stoe & Cie, 2009); data reduction: *X-RED* (Stoe & Cie, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2012* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(CaClO4_4H2O_200K) Calcium perchlorate tetrahydrate

Crystal data

$Ca(ClO_4)_2·4H_2O$
M_r = 311.04
Triclinic, $P\overline{1}$
a = 5.4886 (11) Å
b = 7.8518 (15) Å
c = 11.574 (2) Å
α = 99.663 (16)°
β = 90.366 (16)°
γ = 90.244 (16)°
V = 491.71 (17) Å^3
Z = 2
F(000) = 316
D_x = 2.101 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 26892 reflections
θ = 1.8–29.6°
μ = 1.24 mm^{-1}
T = 200 K
Plate, colourless
0.04 × 0.03 × 0.02 mm

Data collection

Stoe IPDS-2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration Coppens (1970)
T_{min} = 0.644, T_{max} = 0.789
2659 measured reflections
2636 independent reflections
2529 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 29.2°, θ_{min} = 1.8°
h = −6→7
k = −10→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.089
S = 1.20
2636 reflections
168 parameters
12 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.6952P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Ca1	0.51082 (7)	0.23667 (5)	0.75582 (3)	0.01027 (10)
Cl1	0.24098 (7)	−0.16433 (5)	0.55712 (4)	0.00941 (11)
Cl2	0.32358 (8)	0.67561 (5)	0.92062 (4)	0.01142 (11)
O2	0.7504 (3)	−0.01296 (18)	0.76729 (13)	0.0139 (3)
O1	0.2602 (3)	0.38606 (19)	0.63310 (12)	0.0138 (3)
O3	0.4297 (3)	−0.2742 (2)	0.58913 (14)	0.0186 (3)
O4	0.5616 (3)	0.7400 (2)	0.89554 (15)	0.0212 (3)
O10	0.2625 (3)	−0.1487 (2)	0.43554 (13)	0.0178 (3)
O11	0.0048 (3)	−0.2367 (2)	0.57611 (13)	0.0169 (3)
O9	0.2594 (3)	0.00467 (19)	0.62752 (14)	0.0182 (3)
O5	0.2746 (3)	0.7265 (2)	1.04351 (13)	0.0205 (3)
O8	0.2156 (3)	0.1319 (2)	0.86975 (14)	0.0203 (3)
O7	0.7938 (3)	0.4545 (2)	0.74134 (15)	0.0209 (3)
O6	0.3224 (3)	0.48966 (18)	0.89187 (14)	0.0177 (3)
O12	0.1399 (3)	0.7434 (2)	0.85344 (17)	0.0290 (4)
H2B	0.683 (6)	−0.078 (3)	0.806 (2)	0.033 (9)*
H1A	0.317 (6)	0.477 (2)	0.618 (2)	0.025 (8)*
H2A	0.788 (6)	−0.072 (3)	0.7045 (15)	0.031 (9)*
H7A	0.931 (3)	0.444 (4)	0.714 (3)	0.033 (9)*
H7B	0.792 (6)	0.541 (3)	0.791 (2)	0.040 (10)*
H8B	0.087 (5)	0.083 (6)	0.848 (3)	0.077 (16)*
H1B	0.219 (6)	0.328 (3)	0.5705 (15)	0.026 (8)*
H8A	0.196 (7)	0.179 (5)	0.9380 (15)	0.058 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ca1	0.00953 (17)	0.00980 (17)	0.01102 (17)	−0.00055 (12)	−0.00070 (12)	0.00046 (12)
Cl1	0.00864 (19)	0.01026 (19)	0.00859 (19)	−0.00147 (14)	−0.00038 (13)	−0.00050 (14)
Cl2	0.0136 (2)	0.00949 (19)	0.0102 (2)	0.00073 (14)	−0.00324 (15)	−0.00092 (14)
O2	0.0161 (7)	0.0134 (6)	0.0121 (6)	0.0015 (5)	0.0011 (5)	0.0018 (5)
O1	0.0149 (6)	0.0131 (6)	0.0129 (6)	−0.0014 (5)	−0.0024 (5)	0.0006 (5)
O3	0.0151 (7)	0.0190 (7)	0.0226 (8)	0.0042 (6)	−0.0026 (6)	0.0062 (6)
O4	0.0218 (8)	0.0199 (7)	0.0220 (8)	−0.0058 (6)	0.0042 (6)	0.0039 (6)
O10	0.0210 (7)	0.0236 (7)	0.0094 (6)	0.0030 (6)	0.0031 (5)	0.0040 (5)
O11	0.0119 (6)	0.0209 (7)	0.0172 (7)	−0.0079 (5)	0.0007 (5)	0.0013 (6)
O9	0.0177 (7)	0.0133 (7)	0.0201 (7)	−0.0032 (5)	−0.0029 (5)	−0.0072 (5)
O5	0.0185 (7)	0.0267 (8)	0.0128 (7)	0.0025 (6)	0.0015 (5)	−0.0071 (6)
O8	0.0193 (7)	0.0199 (7)	0.0188 (7)	−0.0092 (6)	0.0044 (6)	−0.0055 (6)

O7	0.0168 (7)	0.0175 (7)	0.0251 (8)	−0.0064 (6)	0.0063 (6)	−0.0062 (6)
O6	0.0226 (7)	0.0085 (6)	0.0203 (7)	0.0012 (5)	0.0008 (6)	−0.0029 (5)
O12	0.0314 (9)	0.0213 (8)	0.0347 (10)	0.0029 (7)	−0.0216 (8)	0.0065 (7)

Geometric parameters (Å, º)

Ca1—O8	2.3284 (17)	Cl1—O10	1.4385 (15)
Ca1—O7	2.3329 (17)	Cl1—O9	1.4387 (15)
Ca1—O2	2.3866 (15)	Cl1—O11	1.4461 (15)
Ca1—O1	2.4153 (16)	Cl2—O12	1.4274 (16)
Ca1—O10^{i}	2.5417 (16)	Cl2—O5	1.4388 (15)
Ca1—O9	2.5439 (16)	Cl2—O6	1.4420 (15)
Ca1—O6	2.5463 (16)	Cl2—O4	1.4473 (17)
Ca1—O5^{ii}	2.5695 (17)	O10—Ca1^{i}	2.5417 (16)
Cl1—O3	1.4365 (15)	O5—Ca1^{ii}	2.5695 (17)
O8—Ca1—O7	146.78 (6)	O7—Ca1—O5^{ii}	78.03 (6)
O8—Ca1—O2	89.06 (6)	O2—Ca1—O5^{ii}	70.60 (5)
O7—Ca1—O2	104.79 (6)	O1—Ca1—O5^{ii}	142.78 (5)
O8—Ca1—O1	100.92 (6)	O10^{i}—Ca1—O5^{ii}	122.42 (5)
O7—Ca1—O1	84.26 (6)	O9—Ca1—O5^{ii}	136.94 (6)
O2—Ca1—O1	146.29 (5)	O6—Ca1—O5^{ii}	70.75 (5)
O8—Ca1—O10^{i}	140.33 (6)	O3—Cl1—O10	110.02 (9)
O7—Ca1—O10^{i}	72.75 (6)	O3—Cl1—O9	110.18 (10)
O2—Ca1—O10^{i}	70.60 (5)	O10—Cl1—O9	109.06 (10)
O1—Ca1—O10^{i}	81.78 (5)	O3—Cl1—O11	109.83 (9)
O8—Ca1—O9	70.75 (6)	O10—Cl1—O11	109.13 (9)
O7—Ca1—O9	140.80 (6)	O9—Cl1—O11	108.59 (9)
O2—Ca1—O9	79.34 (5)	O12—Cl2—O5	109.54 (11)
O1—Ca1—O9	73.95 (5)	O12—Cl2—O6	109.31 (10)
O10^{i}—Ca1—O9	72.18 (5)	O5—Cl2—O6	109.33 (10)
O8—Ca1—O6	71.00 (6)	O12—Cl2—O4	110.57 (11)
O7—Ca1—O6	79.25 (6)	O5—Cl2—O4	108.97 (10)
O2—Ca1—O6	139.24 (5)	O6—Cl2—O4	109.09 (10)
O1—Ca1—O6	73.94 (5)	Cl1—O10—Ca1^{i}	147.22 (10)
O10^{i}—Ca1—O6	144.46 (5)	Cl1—O9—Ca1	150.03 (10)
O9—Ca1—O6	123.19 (5)	Cl2—O5—Ca1^{ii}	140.90 (10)
O8—Ca1—O5^{ii}	78.49 (6)	Cl2—O6—Ca1	142.92 (10)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O11^{iii}	0.82 (1)	2.11 (2)	2.888 (2)	158 (3)
O1—H1*A*···O3^{iv}	0.82 (1)	2.13 (1)	2.947 (2)	174 (3)
O2—H2*A*···O11^{v}	0.82 (1)	2.17 (2)	2.947 (2)	159 (3)
O2—H2*B*···O4^{vi}	0.82 (1)	2.02 (1)	2.830 (2)	172 (3)
O7—H7*B*···O4	0.81 (1)	2.22 (2)	2.924 (2)	146 (3)

O7—H7*A*···O1^{v}	0.82 (1)	2.06 (1)	2.870 (2)	172 (3)
O8—H8*A*···O4^{ii}	0.82 (1)	2.33 (3)	2.986 (2)	137 (4)
O8—H8*B*···O2^{vii}	0.82 (1)	2.14 (1)	2.950 (2)	169 (5)

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

(CaClO4_6H2O_180K) Calcium perchlorate hexahydrate

Crystal data

$Ca(ClO_4)_2·6H_2O$
M_r = 347.08
Orthorhombic, $Pca2_1$
a = 10.9603 (4) Å
b = 7.9667 (7) Å
c = 26.7735 (18) Å
V = 2337.8 (3) Å^3
Z = 8
F(000) = 1424
D_x = 1.972 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 17254 reflections
θ = 2.9–29.6°
μ = 1.06 mm^{-1}
T = 180 K
Plate, colourless
0.38 × 0.31 × 0.08 mm

Data collection

Stoe IPDS-2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (Coppens, 1970)
T_{min} = 0.684, T_{max} = 0.923
15755 measured reflections
5326 independent reflections
4919 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 27.5°, θ_{min} = 1.5°
h = −15→15
k = −11→9
l = −37→37

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
S = 1.09
5326 reflections
380 parameters
37 restraints
Hydrogen site location: difference Fourier map
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2 + 2.3411P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.45 (9)

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. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ca1	0.87471 (15)	0.02736 (19)	0.29261 (6)	0.0110 (3)
Ca2	0.87640 (16)	0.47462 (18)	0.07672 (6)	0.0112 (3)
Cl3	0.7770 (2)	0.50239 (14)	0.39582 (8)	0.0102 (5)

Cl4	0.79753 (11)	0.06649 (15)	0.13985 (8)	0.0110 (2)
Cl1	0.95348 (11)	0.43473 (15)	0.22917 (8)	0.0109 (2)
Cl2	0.0260 (2)	0.99991 (14)	0.47324 (8)	0.0128 (5)
O5	0.7128 (5)	0.2189 (8)	0.2859 (2)	0.0169 (11)
H5B	0.714 (6)	0.295 (6)	0.264 (2)	0.020*
H5A	0.660 (5)	0.209 (10)	0.3081 (19)	0.020*
O3	0.7416 (5)	−0.1972 (7)	0.2841 (2)	0.0185 (12)
H3A	0.672 (3)	−0.190 (10)	0.296 (3)	0.022*
H3B	0.752 (7)	−0.274 (6)	0.263 (2)	0.022*
O7	0.9726 (7)	0.4844 (6)	−0.0075 (3)	0.0162 (14)
H7B	0.995 (7)	0.557 (7)	−0.028 (2)	0.019*
H7A	0.989 (6)	0.406 (6)	−0.027 (2)	0.019*
O8	1.0380 (5)	0.2796 (7)	0.0823 (2)	0.0132 (10)
H8A	1.094 (4)	0.245 (8)	0.064 (2)	0.016*
H8B	1.013 (6)	0.186 (4)	0.093 (2)	0.016*
O6	0.7539 (4)	0.6064 (6)	0.13676 (16)	0.0190 (8)
H6B	0.761 (7)	0.565 (10)	0.1655 (14)	0.023*
H6A	0.684 (3)	0.648 (9)	0.133 (3)	0.023*
O1	0.9967 (4)	−0.1049 (6)	0.23219 (17)	0.0186 (8)
H1A	0.987 (7)	−0.060 (10)	0.2041 (15)	0.022*
H1B	1.0726 (17)	−0.118 (9)	0.232 (3)	0.022*
O2	0.9964 (5)	−0.1849 (7)	0.3393 (2)	0.0119 (11)
H2A	1.007 (7)	−0.184 (9)	0.3702 (7)	0.014*
H2B	0.967 (6)	−0.281 (4)	0.335 (2)	0.014*
O4	0.7803 (9)	0.0171 (8)	0.3739 (3)	0.0262 (18)
H4B	0.793 (8)	−0.068 (6)	0.392 (3)	0.031*
H4A	0.795 (7)	0.093 (7)	0.395 (2)	0.031*
O15	0.9234 (8)	0.0045 (5)	0.1339 (4)	0.0152 (16)
O14	0.7230 (8)	−0.0046 (5)	0.1019 (3)	0.0194 (18)
O19	0.8302 (8)	0.4971 (5)	0.2349 (3)	0.0142 (15)
O20	1.0316 (9)	0.5052 (6)	0.2678 (4)	0.027 (2)
O26	0.8567 (9)	0.5044 (5)	0.3525 (3)	0.0215 (19)
O12	1.0148 (5)	0.2008 (7)	0.3398 (2)	0.0129 (11)
H12B	1.034 (7)	0.189 (9)	0.3699 (10)	0.016*
H12A	1.006 (6)	0.303 (3)	0.334 (2)	0.016*
O16	0.7540 (9)	0.0215 (7)	0.1877 (3)	0.0234 (15)
O21	0.1020 (10)	0.9989 (6)	0.5168 (3)	0.026 (2)
O28	0.7947 (7)	0.6558 (9)	0.4237 (3)	0.0231 (15)
O27	0.8080 (7)	0.3589 (9)	0.4260 (3)	0.0260 (16)
O23	0.0566 (6)	0.8560 (8)	0.4423 (3)	0.0183 (13)
O24	0.0519 (7)	1.1496 (9)	0.4444 (3)	0.0212 (14)
O18	0.9975 (9)	0.4783 (7)	0.1797 (3)	0.0223 (14)
O13	0.7991 (3)	0.2473 (5)	0.13424 (18)	0.0150 (8)
O17	0.9520 (4)	0.2544 (5)	0.23441 (19)	0.0158 (8)
O9	0.7381 (6)	0.2976 (8)	0.0297 (2)	0.0171 (12)
H9A	0.721 (7)	0.338 (8)	0.0016 (13)	0.021*
H9B	0.753 (7)	0.195 (3)	0.025 (3)	0.021*
O22	−0.0976 (9)	0.9933 (7)	0.4886 (5)	0.034 (2)

O25	0.6508 (8)	0.4900 (7)	0.3824 (5)	0.034 (2)
O10	0.7580 (5)	0.6907 (7)	0.0295 (2)	0.0139 (11)
H10B	0.746 (7)	0.658 (8)	0.0002 (11)	0.017*
H10A	0.783 (6)	0.789 (4)	0.025 (2)	0.017*
O11	1.0097 (5)	0.7020 (8)	0.0838 (3)	0.0192 (12)
H11A	1.079 (3)	0.736 (9)	0.075 (3)	0.023*
H11B	0.987 (7)	0.799 (4)	0.092 (3)	0.023*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ca1	0.0083 (6)	0.0090 (5)	0.0157 (7)	0.0010 (5)	0.0018 (4)	−0.0005 (7)
Ca2	0.0095 (6)	0.0084 (5)	0.0155 (7)	0.0010 (5)	0.0021 (4)	0.0016 (7)
Cl3	0.0096 (11)	0.0097 (11)	0.0113 (11)	−0.0005 (4)	0.0016 (10)	0.0004 (4)
Cl4	0.0127 (5)	0.0095 (6)	0.0108 (5)	−0.0007 (4)	0.0020 (4)	0.0012 (5)
Cl1	0.0124 (6)	0.0080 (6)	0.0122 (5)	−0.0018 (4)	0.0019 (4)	0.0008 (5)
Cl2	0.0180 (13)	0.0098 (11)	0.0105 (11)	−0.0001 (4)	0.0016 (11)	0.0002 (4)
O5	0.015 (2)	0.015 (2)	0.020 (2)	0.0012 (19)	0.0022 (19)	0.0054 (19)
O3	0.019 (3)	0.013 (2)	0.024 (2)	−0.007 (2)	0.007 (2)	−0.0047 (18)
O7	0.022 (3)	0.019 (3)	0.008 (3)	0.0018 (18)	0.009 (2)	−0.0048 (16)
O8	0.011 (2)	0.0077 (19)	0.020 (2)	0.0033 (17)	0.0061 (18)	−0.0006 (17)
O6	0.0201 (19)	0.024 (2)	0.0130 (16)	0.0123 (17)	0.0008 (17)	0.0029 (18)
O1	0.0196 (19)	0.021 (2)	0.0151 (17)	0.0079 (17)	0.0003 (17)	0.0028 (18)
O2	0.017 (2)	0.009 (2)	0.009 (2)	−0.0002 (19)	−0.0009 (17)	0.0010 (17)
O4	0.033 (4)	0.023 (3)	0.023 (4)	0.000 (2)	0.008 (3)	0.006 (2)
O15	0.014 (4)	0.010 (3)	0.022 (4)	0.0049 (13)	−0.001 (3)	−0.0002 (14)
O14	0.019 (4)	0.019 (4)	0.021 (4)	−0.0067 (15)	0.000 (3)	−0.0045 (15)
O19	0.013 (4)	0.019 (3)	0.011 (3)	0.0049 (14)	0.007 (3)	0.0011 (13)
O20	0.027 (5)	0.023 (4)	0.033 (5)	−0.0106 (18)	−0.016 (4)	−0.0050 (18)
O26	0.034 (5)	0.011 (3)	0.020 (4)	−0.0004 (16)	0.014 (4)	0.0002 (14)
O12	0.016 (2)	0.011 (2)	0.013 (2)	0.0011 (19)	−0.0010 (17)	0.0021 (18)
O16	0.027 (3)	0.022 (2)	0.021 (3)	0.001 (2)	0.015 (2)	0.011 (2)
O21	0.035 (5)	0.029 (4)	0.013 (4)	0.0009 (18)	−0.010 (4)	0.0018 (15)
O28	0.037 (4)	0.011 (3)	0.021 (3)	−0.004 (2)	0.002 (3)	−0.005 (2)
O27	0.042 (4)	0.017 (3)	0.019 (3)	0.008 (3)	0.003 (3)	0.008 (3)
O23	0.027 (3)	0.014 (3)	0.015 (3)	0.002 (2)	0.001 (2)	−0.006 (2)
O24	0.034 (3)	0.012 (3)	0.017 (3)	−0.003 (2)	−0.006 (2)	0.008 (2)
O18	0.025 (3)	0.027 (2)	0.015 (3)	0.001 (2)	0.012 (2)	0.002 (2)
O13	0.0195 (19)	0.0081 (18)	0.017 (2)	0.0011 (14)	0.0007 (15)	0.0020 (15)
O17	0.0197 (19)	0.0074 (17)	0.020 (2)	−0.0009 (15)	−0.0007 (15)	0.0014 (16)
O9	0.025 (3)	0.011 (2)	0.016 (2)	−0.003 (2)	−0.003 (2)	0.0006 (19)
O22	0.024 (5)	0.032 (4)	0.044 (6)	0.0013 (19)	0.013 (4)	0.000 (2)
O25	0.010 (4)	0.038 (4)	0.054 (6)	−0.0011 (19)	−0.014 (4)	0.004 (2)
O10	0.012 (2)	0.014 (2)	0.016 (2)	0.0008 (19)	0.0021 (18)	0.0005 (18)
O11	0.016 (3)	0.015 (2)	0.027 (2)	−0.002 (2)	0.000 (2)	−0.0030 (19)

Geometric parameters (Å, º)

Ca1—O3	2.319 (6)	Cl3—O25	1.432 (9)
Ca1—O5	2.347 (6)	Cl3—O27	1.440 (7)
Ca1—O1	2.349 (5)	Cl3—O28	1.446 (7)
Ca1—O4	2.412 (9)	Cl3—O26	1.452 (9)
Ca1—O12	2.421 (6)	Cl4—O16	1.414 (8)
Ca1—O2	2.490 (6)	Cl4—O14	1.421 (8)
Ca1—O17	2.533 (5)	Cl4—O13	1.449 (4)
Ca1—O16	3.104 (9)	Cl4—O15	1.474 (8)
Ca2—O11	2.335 (6)	Cl1—O17	1.444 (4)
Ca2—O6	2.343 (5)	Cl1—O19	1.448 (8)
Ca2—O8	2.360 (5)	Cl1—O18	1.451 (8)
Ca2—O9	2.423 (6)	Cl1—O20	1.455 (9)
Ca2—O7	2.491 (7)	Cl2—O22	1.416 (10)
Ca2—O10	2.500 (6)	Cl2—O21	1.433 (10)
Ca2—O13	2.523 (4)	Cl2—O24	1.449 (7)
Ca2—O18	3.061 (9)	Cl2—O23	1.453 (7)
O3—Ca1—O5	91.1 (3)	O7—Ca2—O10	74.9 (2)
O3—Ca1—O1	86.81 (19)	O11—Ca2—O13	135.8 (2)
O5—Ca1—O1	132.0 (2)	O6—Ca2—O13	73.17 (15)
O3—Ca1—O4	78.0 (2)	O8—Ca2—O13	75.00 (17)
O5—Ca1—O4	76.5 (3)	O9—Ca2—O13	71.91 (17)
O1—Ca1—O4	148.3 (2)	O7—Ca2—O13	135.81 (17)
O3—Ca1—O12	152.9 (2)	O10—Ca2—O13	128.95 (17)
O5—Ca1—O12	98.5 (2)	O11—Ca2—O18	69.4 (2)
O1—Ca1—O12	104.72 (19)	O6—Ca2—O18	68.03 (19)
O4—Ca1—O12	79.7 (3)	O8—Ca2—O18	67.9 (2)
O3—Ca1—O2	82.1 (2)	O9—Ca2—O18	138.2 (2)
O5—Ca1—O2	152.3 (2)	O7—Ca2—O18	129.2 (3)
O1—Ca1—O2	74.68 (18)	O10—Ca2—O18	132.42 (19)
O4—Ca1—O2	75.8 (2)	O13—Ca2—O18	66.58 (17)
O12—Ca1—O2	77.7 (2)	O25—Cl3—O27	108.3 (5)
O3—Ca1—O17	134.5 (2)	O25—Cl3—O28	108.5 (5)
O5—Ca1—O17	75.01 (18)	O27—Cl3—O28	110.4 (5)
O1—Ca1—O17	72.92 (15)	O25—Cl3—O26	112.4 (7)
O4—Ca1—O17	136.29 (19)	O27—Cl3—O26	108.3 (4)
O12—Ca1—O17	72.61 (17)	O28—Cl3—O26	108.8 (4)
O2—Ca1—O17	127.91 (18)	O16—Cl4—O14	110.7 (5)
O3—Ca1—O16	68.4 (2)	O16—Cl4—O13	110.5 (3)
O5—Ca1—O16	67.5 (2)	O14—Cl4—O13	109.2 (3)
O1—Ca1—O16	67.2 (2)	O16—Cl4—O15	109.2 (5)
O4—Ca1—O16	129.3 (3)	O14—Cl4—O15	109.1 (5)
O12—Ca1—O16	138.63 (19)	O13—Cl4—O15	108.1 (3)
O2—Ca1—O16	132.21 (19)	O17—Cl1—O19	108.7 (3)
O17—Ca1—O16	66.22 (16)	O17—Cl1—O18	109.3 (3)
O11—Ca2—O6	87.4 (2)	O19—Cl1—O18	109.0 (5)

O11—Ca2—O8	92.1 (3)	O17—Cl1—O20	108.8 (3)
O6—Ca2—O8	133.0 (2)	O19—Cl1—O20	110.0 (5)
O11—Ca2—O9	152.3 (2)	O18—Cl1—O20	111.1 (5)
O6—Ca2—O9	105.0 (2)	O22—Cl2—O21	108.6 (7)
O8—Ca2—O9	96.8 (2)	O22—Cl2—O24	111.9 (4)
O11—Ca2—O7	77.6 (2)	O21—Cl2—O24	108.9 (4)
O6—Ca2—O7	148.20 (18)	O22—Cl2—O23	110.9 (4)
O8—Ca2—O7	76.1 (2)	O21—Cl2—O23	108.9 (4)
O9—Ca2—O7	79.2 (2)	O24—Cl2—O23	107.5 (5)
O11—Ca2—O10	80.3 (2)	Cl4—O16—Ca1	132.3 (5)
O6—Ca2—O10	75.00 (17)	Cl1—O18—Ca2	132.5 (5)
O8—Ca2—O10	151.0 (2)	Cl4—O13—Ca2	141.3 (3)
O9—Ca2—O10	79.2 (3)	Cl1—O17—Ca1	140.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O15	0.84 (2)	2.07 (3)	2.887 (10)	164 (8)
O1—H1*B*···O5^{i}	0.84 (2)	2.25 (5)	2.915 (7)	136 (6)
O1—H1*B*···O16^{i}	0.84 (2)	2.44 (5)	3.132 (10)	140 (6)
O2—H2*A*···O23^{ii}	0.84 (2)	2.03 (2)	2.856 (9)	169 (7)
O2—H2*B*···O26^{iii}	0.84 (2)	2.14 (3)	2.932 (8)	155 (6)
O3—H3*A*···O12^{iv}	0.84 (2)	2.07 (2)	2.899 (8)	168 (8)
O3—H3*B*···O19^{iii}	0.84 (2)	2.15 (3)	2.934 (8)	156 (7)
O4—H4*A*···O27	0.84 (2)	2.28 (3)	3.074 (11)	158 (8)
O4—H4*B*···O28^{iii}	0.84 (2)	2.36 (3)	3.177 (10)	163 (8)
O5—H5*A*···O2^{iv}	0.84 (2)	1.98 (3)	2.783 (8)	159 (7)
O5—H5*B*···O19	0.84 (2)	2.20 (5)	2.903 (9)	142 (6)
O6—H6*A*···O8^{v}	0.84 (2)	2.18 (4)	2.925 (7)	149 (7)
O6—H6*B*···O19	0.84 (2)	2.08 (3)	2.891 (10)	162 (8)
O7—H7*A*···O23^{vi}	0.84 (2)	2.29 (4)	3.042 (9)	149 (6)
O7—H7*B*···O24^{vii}	0.84 (2)	2.50 (5)	3.199 (9)	141 (6)
O7—H7*B*···O27^{viii}	0.84 (2)	2.57 (5)	3.242 (11)	138 (6)
O8—H8*A*···O10^{ix}	0.84 (2)	2.08 (4)	2.805 (8)	145 (6)
O8—H8*B*···O15	0.84 (2)	2.07 (3)	2.879 (9)	162 (7)
O9—H9*A*···O27^{x}	0.84 (2)	2.06 (3)	2.865 (10)	161 (7)
O9—H9*B*···O21^{vi}	0.84 (2)	2.23 (5)	2.962 (10)	145 (7)
O10—H10*A*···O21^{vii}	0.84 (2)	2.12 (3)	2.930 (9)	163 (7)
O10—H10*B*···O28^{x}	0.84 (2)	2.10 (3)	2.902 (10)	162 (7)
O11—H11*A*···O9^{ix}	0.84 (2)	2.14 (4)	2.893 (9)	150 (7)
O11—H11*B*···O15^{xi}	0.84 (2)	2.11 (3)	2.915 (9)	161 (7)
O12—H12*A*···O26	0.84 (2)	2.35 (5)	2.995 (9)	135 (6)
O12—H12*A*···O20	0.84 (2)	2.40 (4)	3.102 (9)	142 (6)
O12—H12*B*···O24^{ii}	0.84 (2)	2.03 (2)	2.861 (9)	171 (7)

Symmetry codes: (i) *x*+1/2, −*y*, *z*; (ii) *x*+1, *y*−1, *z*; (iii) *x*, *y*−1, *z*; (iv) *x*−1/2, −*y*, *z*; (v) *x*−1/2, −*y*+1, *z*; (vi) −*x*+1, −*y*+1, *z*−1/2; (vii) −*x*+1, −*y*+2, *z*−1/2; (viii) −*x*+2, −*y*+1, *z*−1/2; (ix) *x*+1/2, −*y*+1, *z*; (x) −*x*+3/2, *y*, *z*−1/2; (xi) *x*, *y*+1, *z*.