

Poly[[diaquabis[μ -(2,4-dichlorophenoxy)-acetato]calcium(II)] monohydrate]

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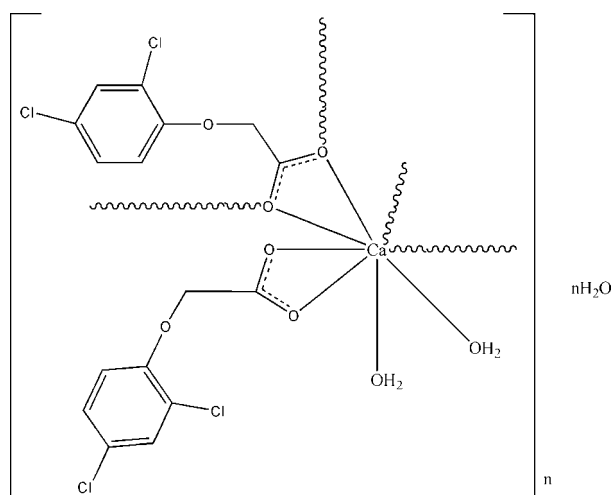
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.051; wR factor = 0.159; data-to-parameter ratio = 17.5.

In the title coordination polymer, $\{[\text{Ca}(\text{C}_8\text{H}_5\text{Cl}_2\text{O}_3)_2(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}\}_n$, the Ca^{II} atom is eight-coordinated by six O atoms from four different (2,4-dichlorophenoxy)acetate ligands and two water molecules, and displays a distorted square-antiprismatic coordination geometry. The compound forms an infinite zigzag chain through connection of the metal centers by (2,4-dichlorophenoxy)acetate ligands and hydrogen bonding of coordinated and interstitial water molecules. These chains are further hydrogen bonded with neighboring chains, forming a supramolecular network.

Related literature

For related literature, see: Song *et al.* (2006); Hao *et al.* (2006).



Experimental

Crystal data

$[\text{Ca}(\text{C}_8\text{H}_5\text{Cl}_2\text{O}_3)_2(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$
 $M_r = 534.17$
 Monoclinic, $P2_1/c$
 $a = 17.8354$ (7) Å
 $b = 6.8077$ (3) Å
 $c = 18.5276$ (8) Å
 $\beta = 101.297$ (3)°

$V = 2206.00$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.81$ mm⁻¹
 $T = 296$ (2) K
 $0.30 \times 0.26 \times 0.23$ mm

Data collection

Bruker APEXII area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.790$, $T_{\text{max}} = 0.840$

15522 measured reflections
 5049 independent reflections
 2962 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.158$
 $S = 1.00$
 5049 reflections
 289 parameters
 9 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.55$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.60$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O3W}-\text{H6W} \cdots \text{O2}^{\text{i}}$	0.848 (10)	2.176 (14)	3.013 (4)	169 (3)
$\text{O2W}-\text{H4W} \cdots \text{O2}^{\text{ii}}$	0.823 (10)	2.079 (16)	2.866 (3)	160 (4)
$\text{O2W}-\text{H3W} \cdots \text{O1}^{\text{iii}}$	0.822 (10)	2.205 (18)	2.986 (4)	159 (4)
$\text{O1W}-\text{H1W} \cdots \text{O3W}^{\text{iv}}$	0.823 (10)	1.927 (11)	2.745 (4)	173 (4)
$\text{O3W}-\text{H5W} \cdots \text{O1}$	0.850 (10)	1.986 (12)	2.830 (4)	172 (5)
$\text{O1W}-\text{H2W} \cdots \text{Cl4}$	0.818 (10)	2.88 (2)	3.530 (3)	138 (3)
$\text{O1W}-\text{H2W} \cdots \text{O6}$	0.818 (10)	2.20 (2)	2.938 (3)	150 (4)

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: XP in SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2102).

References

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 Hao, X.-M., Gu, C.-S., Song, W.-D., Ma, D.-Y. & Liu, Z.-Y. (2006). *Acta Cryst. E62*, m2618–m2620.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
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 Song, W.-D. & Xi, D.-L. (2006). *Acta Cryst. E62*, m2594–m2596.

supplementary materials

Acta Cryst. (2008). E64, m654 [doi:10.1107/S1600536808009379]

Poly[[diaquabis[μ -(2,4-dichlorophenoxy)acetato]calcium(II)] monohydrate]

W.-D. Song, X.-H. Huang, J.-B. Yan and D.-Y. Ma

Comment

In the structural investigation of 2,4-dichlorophenoxyacetate complexes, it has been found that the (2,4-dichlorophenoxy)acetate functions as a multidentate ligand [Song *et al.* (2006); Hao *et al.* (2006)], with versatile binding and coordination modes. In this paper, we report the crystal structure of the title compound, (I), a new Ca complex obtained by the reaction of (2,4-dichlorophenoxy)acetate and calcium chloride in an alkaline aqueous solution.

As illustrated in Figure 1, the Ca^{II} atom exists in a distorted square-antiprismatic environment, defined by six O atoms from four different 2,4-dichlorophenoxyacetate ligands and two water molecules. The 2,4-dichlorophenoxyacetate ligands link the calcium ions to form infinite zigzag like chains, which are further stabilized by hydrogen bonding of the coordinated and interstitial water molecules O2W and O3W to carboxylate oxygen atoms (Table 1, Fig. 2). O1W, *via* a hydrogen bond to the ether oxygen atom O6, also stabilizes the chains, but also forms another intermolecular hydrogen bond to a water molecule O3W that is part of a neighboring chain, thus forming a supramolecular network of H-bonded chains.

Experimental

A mixture of calcium chloride (1 mmol), 2,4-dichlorophenoxyacetate (1 mmol), NaOH (1.5 mmol) and H₂O (12 ml) was placed in a 23 ml Teflon reactor, which was heated to 433 K for three days and then cooled to room temperature at a rate of 10 K h⁻¹. The crystals obtained were washed with water and dried in air.

Refinement

Carbon-bound H atoms were placed in calculated positions and were treated as riding on the parent C atoms with C—H = 0.93–0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. Water H atoms were tentatively located in difference Fourier maps and were refined with distance restraints of O—H = 0.84 Å and H \cdots H = 1.39 Å, each within a standard deviation of 0.01 Å, and with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$

Figures

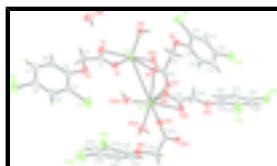


Fig. 1. The structure of (I), showing the atomic numbering scheme. Non-H atoms are shown as 30% probability displacement ellipsoids.

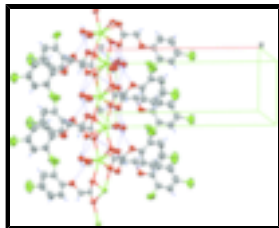


Fig. 2. A packing view of (I).

Poly[[diaquabis[μ -(2,4-dichlorophenoxy)acetato]calcium(II)] monohydrate]

Crystal data

[Ca(C₈H₅Cl₂O₃)₂(H₂O)₂] \cdot H₂O

$M_r = 534.17$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 17.8354$ (7) Å

$b = 6.8077$ (3) Å

$c = 18.5276$ (8) Å

$\beta = 101.297$ (3)°

$V = 2206.00$ (16) Å³

$Z = 4$

$F_{000} = 1088$

$D_x = 1.608$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5837 reflections

$\theta = 2.8$ – 27.9 °

$\mu = 0.81$ mm⁻¹

$T = 296$ (2) K

Block, colorless

$0.30 \times 0.26 \times 0.23$ mm

Data collection

Bruker APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.790$, $T_{\max} = 0.840$

15522 measured reflections

5049 independent reflections

2962 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\text{max}} = 27.5$ °

$\theta_{\text{min}} = 1.2$ °

$h = -22$ → 23

$k = -8$ → 8

$l = -23$ → 24

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.158$

$S = 1.00$

5049 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0794P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.55$ e Å⁻³

289 parameters

$$\Delta\rho_{\min} = -0.60 \text{ e } \text{\AA}^{-3}$$

9 restraints

Extinction correction: none

Primary atom site location: structure-invariant direct methods

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.10806 (18)	0.6762 (5)	0.41264 (16)	0.0380 (7)
C2	0.18125 (18)	0.6618 (5)	0.46951 (19)	0.0450 (8)
H2A	0.1707	0.6023	0.5140	0.054*
H2B	0.2175	0.5790	0.4510	0.054*
C3	0.28244 (17)	0.8609 (6)	0.53280 (18)	0.0448 (9)
C4	0.3216 (2)	1.0370 (6)	0.5327 (2)	0.0570 (10)
C5	0.3898 (2)	1.0689 (7)	0.5798 (3)	0.0700 (13)
H5	0.4156	1.1874	0.5791	0.084*
C6	0.4196 (2)	0.9231 (7)	0.6281 (2)	0.0621 (11)
C7	0.3831 (2)	0.7454 (7)	0.6279 (2)	0.0622 (11)
H7	0.4046	0.6463	0.6600	0.075*
C8	0.31519 (19)	0.7145 (6)	0.5804 (2)	0.0555 (10)
H8	0.2908	0.5937	0.5801	0.067*
C9	-0.24793 (17)	1.0577 (5)	0.26633 (18)	0.0408 (8)
C10	-0.2516 (2)	1.2253 (6)	0.2240 (2)	0.0529 (10)
H10	-0.2156	1.3243	0.2373	0.063*
C11	-0.3083 (2)	1.2475 (7)	0.1617 (2)	0.0632 (11)
H11	-0.3112	1.3622	0.1340	0.076*
C12	-0.3600 (2)	1.1003 (8)	0.1412 (2)	0.0655 (12)
C13	-0.3568 (2)	0.9291 (7)	0.1815 (2)	0.0638 (12)
H13	-0.3919	0.8289	0.1668	0.077*
C14	-0.30089 (18)	0.9088 (6)	0.2439 (2)	0.0481 (9)
C15	-0.13933 (18)	1.1724 (5)	0.35198 (17)	0.0400 (8)
H15A	-0.1135	1.1485	0.4023	0.048*
H15B	-0.1637	1.3001	0.3502	0.048*
C16	-0.08085 (15)	1.1739 (5)	0.30233 (15)	0.0290 (6)
Ca1	-0.02416 (3)	0.67481 (9)	0.29818 (3)	0.03103 (18)
Cl1	0.28379 (8)	1.21602 (17)	0.46954 (10)	0.1105 (6)
Cl2	0.50445 (6)	0.9670 (2)	0.69082 (8)	0.1050 (5)

supplementary materials

Cl3	-0.43126 (7)	1.1255 (3)	0.06300 (7)	0.1062 (5)
Cl4	-0.29530 (6)	0.69273 (16)	0.29402 (7)	0.0722 (3)
O1	0.07745 (13)	0.5164 (3)	0.39084 (12)	0.0501 (6)
O2	0.08134 (12)	0.8394 (3)	0.38986 (12)	0.0456 (6)
O3	0.21318 (13)	0.8512 (4)	0.48578 (14)	0.0531 (7)
O4	-0.06060 (11)	1.0138 (3)	0.27914 (11)	0.0369 (5)
O5	-0.05492 (12)	1.3347 (3)	0.28650 (12)	0.0387 (5)
O6	-0.19623 (11)	1.0256 (3)	0.33051 (12)	0.0427 (6)
O1W	-0.10329 (14)	0.6837 (4)	0.38884 (13)	0.0494 (6)
H2W	-0.1418 (13)	0.750 (5)	0.3754 (18)	0.074*
H1W	-0.0840 (18)	0.731 (5)	0.4291 (12)	0.074*
O2W	-0.12281 (14)	0.6700 (4)	0.18973 (12)	0.0480 (6)
H3W	-0.123 (2)	0.766 (3)	0.1629 (15)	0.072*
H4W	-0.122 (2)	0.577 (3)	0.1615 (14)	0.072*
O3W	0.0390 (2)	0.1918 (4)	0.47185 (15)	0.0702 (8)
H5W	0.047 (3)	0.286 (3)	0.444 (2)	0.105*
H6W	0.049 (3)	0.084 (3)	0.453 (2)	0.105*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0464 (17)	0.033 (2)	0.0330 (15)	-0.0011 (16)	0.0041 (13)	0.0020 (17)
C2	0.0487 (18)	0.035 (2)	0.0459 (17)	0.0030 (16)	-0.0045 (14)	-0.0007 (18)
C3	0.0376 (16)	0.045 (2)	0.0480 (18)	0.0036 (16)	-0.0011 (14)	-0.0061 (18)
C4	0.051 (2)	0.038 (2)	0.075 (2)	0.0067 (18)	-0.0046 (18)	-0.008 (2)
C5	0.047 (2)	0.053 (3)	0.103 (3)	-0.003 (2)	-0.005 (2)	-0.016 (3)
C6	0.0421 (19)	0.069 (3)	0.068 (2)	0.007 (2)	-0.0073 (17)	-0.020 (3)
C7	0.056 (2)	0.070 (3)	0.055 (2)	0.013 (2)	-0.0043 (18)	0.003 (2)
C8	0.0472 (19)	0.059 (3)	0.056 (2)	-0.0014 (19)	-0.0016 (16)	0.005 (2)
C9	0.0363 (15)	0.039 (2)	0.0495 (18)	0.0025 (15)	0.0130 (14)	-0.0018 (18)
C10	0.0451 (18)	0.045 (2)	0.067 (2)	0.0010 (18)	0.0072 (17)	0.009 (2)
C11	0.051 (2)	0.067 (3)	0.070 (3)	0.010 (2)	0.0087 (19)	0.017 (2)
C12	0.044 (2)	0.083 (3)	0.067 (2)	0.002 (2)	0.0049 (18)	0.000 (3)
C13	0.050 (2)	0.075 (3)	0.066 (2)	-0.015 (2)	0.0083 (18)	-0.014 (3)
C14	0.0439 (18)	0.045 (2)	0.058 (2)	-0.0077 (17)	0.0157 (16)	-0.007 (2)
C15	0.0465 (17)	0.0297 (19)	0.0453 (17)	-0.0031 (15)	0.0128 (14)	-0.0073 (17)
C16	0.0321 (13)	0.0212 (17)	0.0317 (14)	-0.0017 (13)	0.0012 (11)	-0.0023 (15)
Ca1	0.0361 (3)	0.0204 (3)	0.0360 (3)	-0.0013 (3)	0.0055 (2)	-0.0010 (3)
Cl1	0.0981 (9)	0.0400 (7)	0.1650 (15)	-0.0089 (7)	-0.0439 (10)	0.0245 (8)
Cl2	0.0585 (6)	0.1143 (12)	0.1209 (11)	0.0061 (7)	-0.0348 (6)	-0.0308 (10)
Cl3	0.0697 (7)	0.1599 (15)	0.0775 (8)	0.0059 (8)	-0.0140 (6)	0.0030 (9)
Cl4	0.0734 (6)	0.0479 (7)	0.0945 (8)	-0.0211 (5)	0.0145 (6)	0.0040 (6)
O1	0.0637 (14)	0.0287 (14)	0.0493 (13)	-0.0065 (12)	-0.0102 (11)	-0.0002 (12)
O2	0.0485 (12)	0.0296 (14)	0.0523 (13)	-0.0008 (11)	-0.0060 (10)	0.0041 (12)
O3	0.0491 (13)	0.0330 (14)	0.0660 (15)	0.0001 (11)	-0.0159 (11)	0.0012 (13)
O4	0.0465 (11)	0.0192 (12)	0.0479 (12)	0.0012 (10)	0.0163 (10)	-0.0007 (11)
O5	0.0455 (11)	0.0198 (12)	0.0528 (13)	-0.0056 (10)	0.0143 (10)	-0.0036 (11)
O6	0.0399 (11)	0.0360 (14)	0.0535 (13)	-0.0074 (11)	0.0121 (10)	0.0018 (12)

O1W	0.0600 (15)	0.0416 (16)	0.0490 (13)	0.0065 (13)	0.0161 (11)	0.0002 (13)
O2W	0.0533 (13)	0.0359 (15)	0.0495 (13)	0.0035 (13)	-0.0031 (11)	-0.0039 (12)
O3W	0.109 (2)	0.0463 (18)	0.0554 (16)	-0.0093 (19)	0.0159 (15)	-0.0003 (15)

Geometric parameters (Å, °)

C1—O1	1.248 (4)	C14—C14	1.732 (4)
C1—O2	1.250 (4)	C15—O6	1.424 (4)
C1—C2	1.512 (4)	C15—C16	1.520 (4)
C2—O3	1.417 (4)	C15—H15A	0.9700
C2—H2A	0.9700	C15—H15B	0.9700
C2—H2B	0.9700	C16—O5	1.246 (3)
C3—O3	1.367 (4)	C16—O4	1.251 (3)
C3—C8	1.382 (5)	C16—Ca1 ⁱ	2.888 (3)
C3—C4	1.387 (5)	Ca1—O5 ⁱⁱ	2.379 (2)
C4—C5	1.370 (5)	Ca1—O1W	2.397 (2)
C4—C11	1.732 (4)	Ca1—O2W	2.398 (2)
C5—C6	1.370 (6)	Ca1—O4	2.405 (2)
C5—H5	0.9300	Ca1—O1	2.485 (2)
C6—C7	1.373 (6)	Ca1—O4 ⁱⁱⁱ	2.527 (2)
C6—C12	1.745 (4)	Ca1—O2	2.536 (2)
C7—C8	1.367 (5)	Ca1—O5 ⁱⁱⁱ	2.550 (2)
C7—H7	0.9300	Ca1—C16 ⁱⁱⁱ	2.888 (3)
C8—H8	0.9300	Ca1—Ca1 ⁱⁱⁱ	4.0148 (6)
C9—O6	1.372 (4)	Ca1—Ca1 ⁱ	4.0148 (6)
C9—C10	1.379 (5)	O4—Ca1 ⁱ	2.527 (2)
C9—C14	1.392 (5)	O5—Ca1 ^{iv}	2.379 (2)
C10—C11	1.386 (5)	O5—Ca1 ⁱ	2.550 (2)
C10—H10	0.9300	O1W—H2W	0.818 (10)
C11—C12	1.364 (6)	O1W—H1W	0.823 (10)
C11—H11	0.9300	O2W—H3W	0.822 (10)
C12—C13	1.379 (6)	O2W—H4W	0.823 (10)
C12—C13	1.739 (4)	O3W—H5W	0.850 (10)
C13—C14	1.379 (5)	O3W—H6W	0.848 (10)
C13—H13	0.9300		
O1—C1—O2	123.5 (3)	O2W—Ca1—O1	152.75 (8)
O1—C1—C2	115.6 (3)	O4—Ca1—O1	131.10 (8)
O2—C1—C2	120.9 (3)	O5 ⁱⁱ —Ca1—O4 ⁱⁱⁱ	71.29 (7)
O3—C2—C1	110.2 (3)	O1W—Ca1—O4 ⁱⁱⁱ	155.21 (8)
O3—C2—H2A	109.6	O2W—Ca1—O4 ⁱⁱⁱ	86.63 (8)
C1—C2—H2A	109.6	O4—Ca1—O4 ⁱⁱⁱ	120.50 (6)
O3—C2—H2B	109.6	O1—Ca1—O4 ⁱⁱⁱ	76.53 (8)
C1—C2—H2B	109.6	O5 ⁱⁱ —Ca1—O2	128.21 (8)
H2A—C2—H2B	108.1	O1W—Ca1—O2	88.91 (8)
O3—C3—C8	126.0 (3)	O2W—Ca1—O2	153.37 (8)
O3—C3—C4	115.7 (3)	O4—Ca1—O2	79.51 (7)

supplementary materials

C8—C3—C4	118.3 (3)	O1—Ca1—O2	51.97 (7)
C5—C4—C3	121.4 (4)	O4 ⁱⁱⁱ —Ca1—O2	97.10 (7)
C5—C4—C11	119.9 (3)	O5 ⁱⁱ —Ca1—O5 ⁱⁱⁱ	120.33 (6)
C3—C4—C11	118.7 (3)	O1W—Ca1—O5 ⁱⁱⁱ	153.23 (8)
C4—C5—C6	118.9 (4)	O2W—Ca1—O5 ⁱⁱⁱ	83.90 (8)
C4—C5—H5	120.6	O4—Ca1—O5 ⁱⁱⁱ	70.50 (7)
C6—C5—H5	120.6	O1—Ca1—O5 ⁱⁱⁱ	101.18 (8)
C5—C6—C7	120.9 (3)	O4 ⁱⁱⁱ —Ca1—O5 ⁱⁱⁱ	51.11 (7)
C5—C6—C12	119.0 (4)	O2—Ca1—O5 ⁱⁱⁱ	78.24 (7)
C7—C6—C12	120.1 (3)	O5 ⁱⁱ —Ca1—C16 ⁱⁱⁱ	96.11 (8)
C8—C7—C6	119.8 (4)	O1W—Ca1—C16 ⁱⁱⁱ	175.59 (8)
C8—C7—H7	120.1	O2W—Ca1—C16 ⁱⁱⁱ	85.51 (8)
C6—C7—H7	120.1	O4—Ca1—C16 ⁱⁱⁱ	95.63 (8)
C7—C8—C3	120.7 (4)	O1—Ca1—C16 ⁱⁱⁱ	88.12 (8)
C7—C8—H8	119.7	O4 ⁱⁱⁱ —Ca1—C16 ⁱⁱⁱ	25.61 (7)
C3—C8—H8	119.7	O2—Ca1—C16 ⁱⁱⁱ	86.73 (8)
O6—C9—C10	125.0 (3)	O5 ⁱⁱⁱ —Ca1—C16 ⁱⁱⁱ	25.52 (7)
O6—C9—C14	116.4 (3)	O5 ⁱⁱ —Ca1—Ca1 ⁱⁱⁱ	36.91 (5)
C10—C9—C14	118.6 (3)	O1W—Ca1—Ca1 ⁱⁱⁱ	122.70 (6)
C9—C10—C11	120.6 (4)	O2W—Ca1—Ca1 ⁱⁱⁱ	78.57 (6)
C9—C10—H10	119.7	O4—Ca1—Ca1 ⁱⁱⁱ	145.47 (6)
C11—C10—H10	119.7	O1—Ca1—Ca1 ⁱⁱⁱ	75.42 (6)
C12—C11—C10	119.6 (4)	O4 ⁱⁱⁱ —Ca1—Ca1 ⁱⁱⁱ	34.50 (5)
C12—C11—H11	120.2	O2—Ca1—Ca1 ⁱⁱⁱ	118.48 (6)
C10—C11—H11	120.2	O5 ⁱⁱⁱ —Ca1—Ca1 ⁱⁱⁱ	84.01 (5)
C11—C12—C13	121.1 (4)	C16 ⁱⁱⁱ —Ca1—Ca1 ⁱⁱⁱ	59.25 (7)
C11—C12—C13	120.2 (4)	O5 ⁱⁱ —Ca1—Ca1 ⁱ	148.24 (6)
C13—C12—C13	118.7 (3)	O1W—Ca1—Ca1 ⁱ	119.82 (6)
C14—C13—C12	119.0 (4)	O2W—Ca1—Ca1 ⁱ	79.94 (6)
C14—C13—H13	120.5	O4—Ca1—Ca1 ⁱ	36.54 (5)
C12—C13—H13	120.5	O1—Ca1—Ca1 ⁱ	118.95 (6)
C13—C14—C9	121.0 (4)	O4 ⁱⁱⁱ —Ca1—Ca1 ⁱ	84.92 (5)
C13—C14—C14	119.4 (3)	O2—Ca1—Ca1 ⁱ	74.18 (5)
C9—C14—C14	119.6 (3)	O5 ⁱⁱⁱ —Ca1—Ca1 ⁱ	34.07 (5)
O6—C15—C16	111.8 (2)	C16 ⁱⁱⁱ —Ca1—Ca1 ⁱ	59.49 (7)
O6—C15—H15A	109.3	Ca1 ⁱⁱⁱ —Ca1—Ca1 ⁱ	115.95 (3)
C16—C15—H15A	109.3	C1—O1—Ca1	93.37 (19)
O6—C15—H15B	109.3	C1—O2—Ca1	90.98 (19)
C16—C15—H15B	109.3	C3—O3—C2	117.1 (3)
H15A—C15—H15B	107.9	C16—O4—Ca1	150.93 (19)
O5—C16—O4	122.7 (3)	C16—O4—Ca1 ⁱ	93.54 (17)

O5—C16—C15	118.6 (3)	Ca1—O4—Ca1 ⁱ	108.96 (8)
O4—C16—C15	118.7 (3)	C16—O5—Ca1 ^{iv}	157.43 (19)
O5—C16—Ca1 ⁱ	61.88 (15)	C16—O5—Ca1 ⁱ	92.60 (17)
O4—C16—Ca1 ⁱ	60.85 (14)	Ca1 ^{iv} —O5—Ca1 ⁱ	109.02 (8)
C15—C16—Ca1 ⁱ	177.18 (19)	C9—O6—C15	116.8 (3)
O5 ⁱⁱ —Ca1—O1W	86.07 (8)	Ca1—O1W—H2W	112 (3)
O5 ⁱⁱ —Ca1—O2W	78.00 (8)	Ca1—O1W—H1W	117 (3)
O1W—Ca1—O2W	98.71 (9)	H2W—O1W—H1W	103.9 (16)
O5 ⁱⁱ —Ca1—O4	150.41 (8)	Ca1—O2W—H3W	114 (3)
O1W—Ca1—O4	84.21 (8)	Ca1—O2W—H4W	116 (3)
O2W—Ca1—O4	75.96 (8)	H3W—O2W—H4W	103.2 (16)
O5 ⁱⁱ —Ca1—O1	76.38 (8)	H5W—O3W—H6W	109.8 (17)
O1W—Ca1—O1	88.67 (8)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3W—H6W \cdots O2 ⁱⁱ	0.848 (10)	2.176 (14)	3.013 (4)	169 (3)
O2W—H4W \cdots O2 ⁱⁱⁱ	0.823 (10)	2.079 (16)	2.866 (3)	160 (4)
O2W—H3W \cdots O1 ⁱ	0.822 (10)	2.205 (18)	2.986 (4)	159 (4)
O1W—H1W \cdots O3W ^v	0.823 (10)	1.927 (11)	2.745 (4)	173 (4)
O3W—H5W \cdots O1	0.850 (10)	1.986 (12)	2.830 (4)	172 (5)
O1W—H2W \cdots C14	0.818 (10)	2.88 (2)	3.530 (3)	138 (3)
O1W—H2W \cdots O6	0.818 (10)	2.20 (2)	2.938 (3)	150 (4)

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

Fig. 1

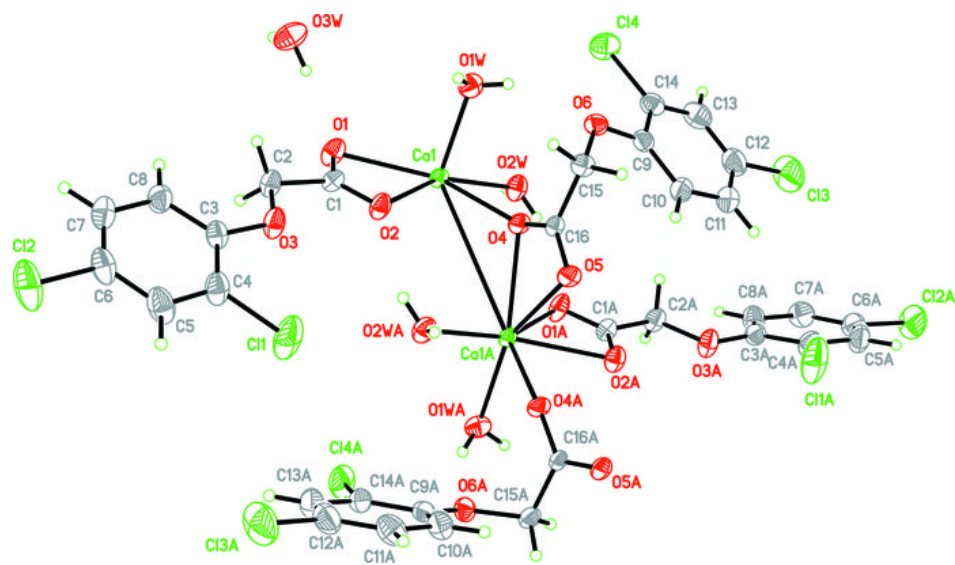


Fig. 2

