Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(4,4″-difluoro-1,1′:3′,1″-terphenyl-2′-carboxylato-κ*O*)tetrakis(methanol-κ*O*)-calcium methanol tetrasolvate

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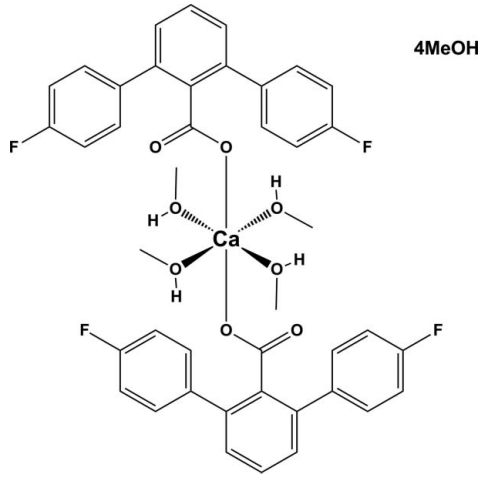
Received 21 November 2012; accepted 2 January 2013

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.165; data-to-parameter ratio = 19.0.

In the title compound, $[Ca(C_{19}H_{11}F_2O_2)_2(CH_3OH)_4]\cdot 4CH_3OH$, the Ca^{2+} ion is located on an inversion centre and is hexacoordinated by two O atoms of two 4,4″-difluoro-1,1′:3′,1″-terphenyl-2′-carboxylate ligands and four O atoms of four methanol ligands, forming a CaO_6 polyhedron with a slightly distorted octahedral coordination geometry. The Ca—O—C angle between the carboxylate group and the calcium ion is 171.8 (2)°. Two types of intermolecular hydrogen-bond interactions (C=O···H and O—H···O) between the carboxylate ligand, the methanol solvent molecules and the coordinating methanol ligands generate a two-dimensional network parallel to (001).

Related literature

For background to metal complexes with 4,4″-difluoro-1,1′:3′,1″-terphenyl-2′-carboxylate ligands, see: Kannan *et al.* (2011); Chavez *et al.* (2001). For mononuclear calcium complexes with carboxylate ligands, see: Perrin *et al.* (2009); Godino Salido *et al.* (2004); Huang *et al.* (2010). For their polymerization behavior, see: Jisha *et al.* (2010); Murugavel & Banerjee (2003); Yang *et al.* (2004).



Experimental

Crystal data

$[Ca(C_{19}H_{11}F_2O_2)_2(CH_4O)_4]\cdot 4CH_4O$
M_r = 914.97
Orthorhombic, *Pbca*
a = 15.4611 (19) Å
b = 14.2436 (18) Å
c = 20.886 (3) Å
V = 4599.5 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 200 K
0.32 × 0.23 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.521, T_{max} = 1.00
32627 measured reflections
5709 independent reflections
2624 reflections with $I > 2\sigma(I)$
R_{int} = 0.143

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.165
S = 0.98
5709 reflections
300 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ca1—O1	2.2693 (19)	Ca1—O3	2.346 (2)
Ca1—O4	2.325 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O6^{ii}	0.89 (4)	1.78 (4)	2.636 (3)	162 (4)
O4—H2···O5^{iii}	0.91 (4)	1.76 (4)	2.660 (3)	172 (4)
O6—H6*A*···O2^{iv}	0.84	1.96	2.804 (3)	177
O5—H5*A*···O2^{v}	0.84	1.92	2.755 (3)	170

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

This work was supported by the International Collaborative R&D Program of the Korea Institute of Energy Technology Evaluation and Planning (KETEP) grant funded by Korea Government Ministry of Knowledge Economy (20118520010020).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RU2047).

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supporting information

Acta Cryst. (2013). E**69**, m122–m123 [doi:10.1107/S1600536813000044]

Bis(4,4′′-difluoro-1,1′:3′,1′′-terphenyl-2′-carboxylato-κO)tetrakis(methanol-κO)calcium methanol tetrasolvate

Namseok Kim, Heeso Noh, Sungho Yoon and Chan Ryang Park

S1. Comment

Octahedral mononuclear transition metal complexes with ligated carboxylates are well known structure for basic of inorganic chemistry (Chavez *et al.*, 2001; Kannan *et al.*, 2011). Few mononuclear calcium complexes with carboxylate ligands (Perrin *et al.*, 2009; Godino Salido *et al.*, 2004; Huang *et al.*, 2010) are reported possible due to easy polymerization behavior (Murugavel *et al.*, 2003; Jisha *et al.*, 2010; Yang *et al.*, 2004). Here, we report the structure of an octahedrally coordinated Ca^{2+} complex which crystallizes in the orthorhombic space group *Pbca* with one half molecule in the asymmetric unit. The selected bond distances and angles of $[Ca(C_{19}H_{11}O_2F_2)_2(CH_4O)_4]$ are given in Table 1 with the structure of the molecule shown, in Fig 1, and its crystal packing involving strong intermolecular C=O···H, O—H···O interactions are detailed in Fig 2 and Table 2.

S2. Experimental

To a solution of sodium 4,4′′-difluoro-1,1′:3′,1′′-terphenyl-2′-carboxylate (0.200 g, 0.602 mmol) in 15 ml of methanol, $Ca(CF_3SO_3)_2$ (0.204 g, 0.602 mmol) was added at room temperature. After stirring for 30 min, colorless block type crystals were collected from slow evaporization. Yield = 51%, (0.281 g).

S3. Refinement

H atoms were placed at calculated positions and refined as riding with C–H(aromatic) = 0.95 Å, C–H(CH_3) = 0.98 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl groups. The O-bound H atoms of methanol were located in a difference Fourier map and refined isotropically.

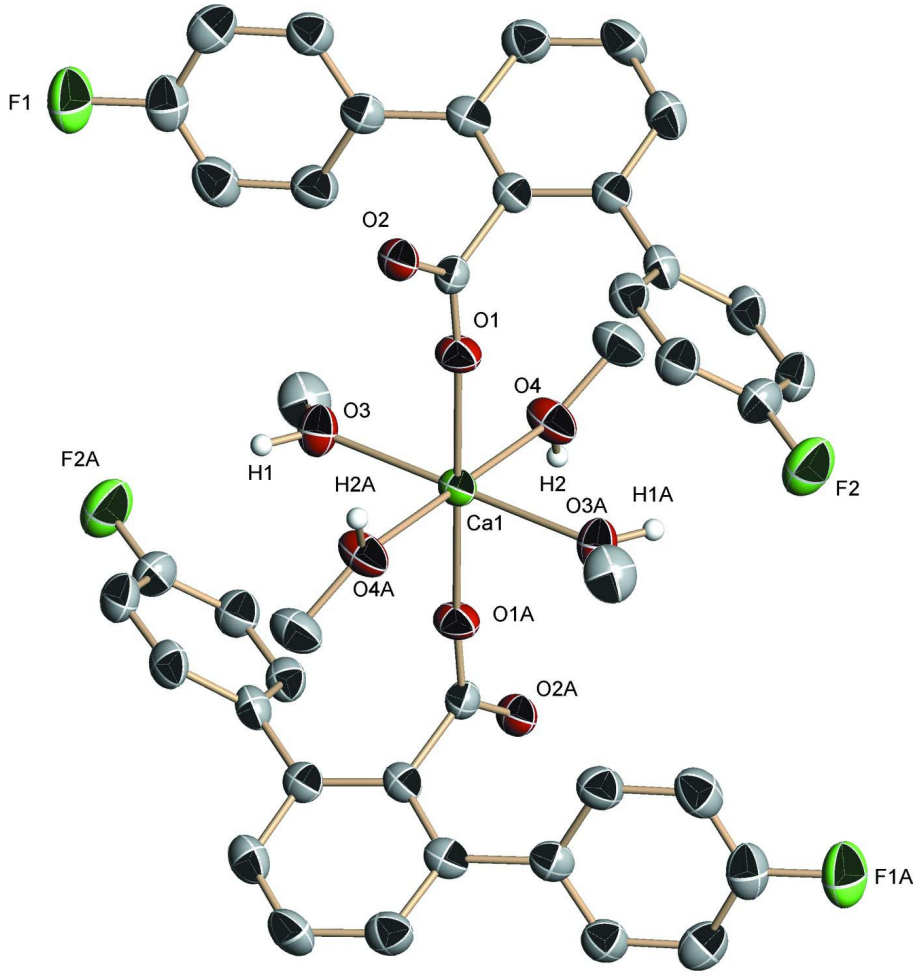

Figure 1

The molecular structure of the title compound, showing the atom-numbering and with displacement ellipsoids drawn at the 50% probability level.

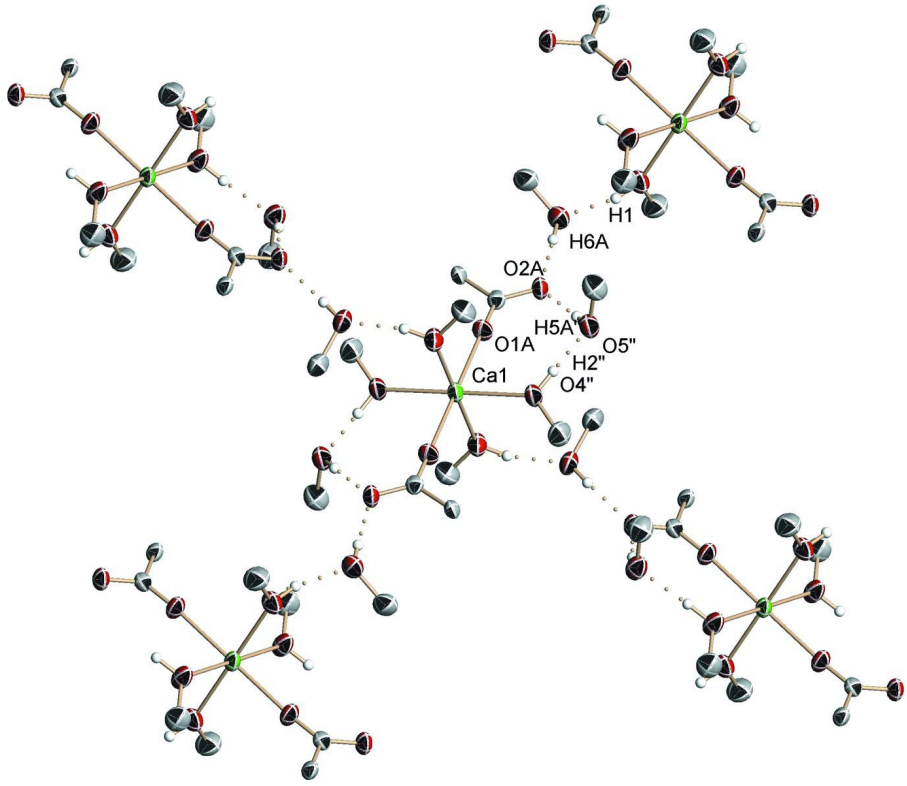

Figure 2

A crystal packing diagram of the title compound, showing the hydrogen bonds and with displacement ellipsoids drawn at the 50% probability level.

Bis(4,4′′-difluoro-1,1′:3′,1′′-terphenyl-2′-carboxylato- *κO*)tetrakis(methanol-*κO*)calcium methanol tetrasolvate

Crystal data

$[Ca(C_{19}H_{11}F_2O_2)_2(CH_4O)_4]\cdot 4CH_4O$
$M_r = 914.97$
Orthorhombic, *Pbca*
Hall symbol: -p 2ac 2ab
$a = 15.4611\ (19)$ Å
$b = 14.2436\ (18)$ Å
$c = 20.886\ (3)$ Å
$V = 4599.5\ (10)$ Å^3
$Z = 4$
$F(000) = 1928$
$D_x = 1.321$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2898 reflections
$\theta = 2.2–22.0°$
$\mu = 0.21$ mm^{-1}
$T = 200$ K
Block, colorless
0.32 × 0.23 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.521$, $T_{max} = 1.00$
32627 measured reflections
5709 independent reflections
2624 reflections with $I > 2\sigma(I)$
$R_{int} = 0.143$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.0°$
$h = -14\rightarrow20$
$k = -18\rightarrow18$
$l = -23\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.165$
$S = 0.98$
5709 reflections
300 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
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
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ca1	1.0000	0.0000	0.5000	0.0296 (2)
O2	0.79691 (12)	0.20766 (14)	0.46541 (10)	0.0359 (5)
O1	0.92097 (13)	0.12979 (14)	0.47660 (10)	0.0382 (6)
O3	0.94225 (15)	−0.07389 (16)	0.40917 (11)	0.0416 (6)
O4	1.12075 (14)	0.04631 (15)	0.44165 (11)	0.0436 (6)
F2	1.02858 (13)	0.35764 (16)	0.71251 (10)	0.0657 (6)
F1	0.68180 (14)	0.03347 (16)	0.22211 (11)	0.0698 (7)
C15	0.7834 (2)	0.2459 (2)	0.27894 (15)	0.0414 (8)
H15	0.7766	0.3120	0.2754	0.050*
C1	0.87736 (19)	0.1973 (2)	0.45564 (14)	0.0293 (7)
C8	0.99166 (19)	0.3377 (2)	0.51732 (15)	0.0349 (8)
C2	0.92369 (18)	0.27084 (19)	0.41636 (15)	0.0297 (7)
C6	0.9544 (2)	0.3437 (2)	0.31427 (17)	0.0439 (9)
H6	0.9460	0.3476	0.2693	0.053*
C14	0.8506 (2)	0.2098 (2)	0.31613 (15)	0.0353 (8)
C3	0.97877 (18)	0.3349 (2)	0.44720 (16)	0.0327 (8)
C7	0.91018 (19)	0.2756 (2)	0.35003 (15)	0.0344 (8)
C5	1.0100 (2)	0.4050 (2)	0.34434 (17)	0.0455 (9)
H5	1.0407	0.4501	0.3197	0.055*
C9	0.9219 (2)	0.3391 (2)	0.56012 (16)	0.0368 (8)
H9	0.8647	0.3351	0.5436	0.044*
C13	1.0747 (2)	0.3425 (2)	0.54382 (17)	0.0400 (8)
H13	1.1235	0.3414	0.5161	0.048*
C19	0.8596 (2)	0.1135 (2)	0.31971 (16)	0.0451 (9)

H19	0.9049	0.0876	0.3448	0.054*
C10	0.9335 (2)	0.3462 (2)	0.62520 (16)	0.0428 (8)
H10	0.8855	0.3479	0.6535	0.051*
C4	1.0216 (2)	0.4017 (2)	0.40898 (18)	0.0426 (9)
H4	1.0595	0.4455	0.4287	0.051*
C11	1.0168 (2)	0.3508 (2)	0.64801 (17)	0.0454 (9)
C12	1.0876 (2)	0.3486 (2)	0.60847 (17)	0.0439 (9)
H12	1.1444	0.3512	0.6256	0.053*
C17	0.7379 (2)	0.0923 (3)	0.25238 (17)	0.0479 (9)
C21	1.1492 (2)	0.1374 (2)	0.42276 (18)	0.0514 (10)
H21A	1.1019	0.1703	0.4011	0.077*
H21B	1.1984	0.1314	0.3934	0.077*
H21C	1.1669	0.1730	0.4607	0.077*
C20	0.9850 (2)	−0.1026 (3)	0.35239 (19)	0.0562 (10)
H20A	1.0103	−0.0477	0.3312	0.084*
H20B	0.9433	−0.1327	0.3236	0.084*
H20C	1.0309	−0.1474	0.3632	0.084*
C18	0.8036 (2)	0.0540 (2)	0.28735 (17)	0.0503 (10)
H18	0.8108	−0.0122	0.2895	0.060*
C16	0.7266 (2)	0.1872 (3)	0.24715 (16)	0.0472 (9)
H16	0.6808	0.2123	0.2222	0.057*
O5	0.24558 (14)	0.91787 (15)	0.43929 (12)	0.0451 (6)
H5A	0.2373	0.8828	0.4711	0.068*
O6	0.78040 (14)	0.86827 (15)	0.41295 (12)	0.0483 (6)
H6A	0.7576	0.8192	0.4275	0.073*
C23	0.7269 (2)	0.9463 (2)	0.42684 (19)	0.0504 (10)
H23A	0.7059	0.9417	0.4710	0.076*
H23B	0.6775	0.9468	0.3974	0.076*
H23C	0.7601	1.0044	0.4217	0.076*
C22	0.2469 (2)	0.8631 (3)	0.38278 (18)	0.0554 (10)
H22A	0.2503	0.9045	0.3454	0.083*
H22B	0.1940	0.8254	0.3803	0.083*
H22C	0.2974	0.8215	0.3835	0.083*
H2	1.163 (2)	0.003 (3)	0.4370 (19)	0.078 (13)*
H1	0.892 (3)	−0.104 (3)	0.4134 (19)	0.084 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ca1	0.0215 (4)	0.0302 (4)	0.0370 (5)	0.0015 (4)	0.0028 (4)	0.0044 (4)
O2	0.0218 (12)	0.0384 (12)	0.0474 (15)	0.0003 (9)	0.0029 (9)	0.0063 (11)
O1	0.0323 (13)	0.0324 (12)	0.0498 (15)	0.0075 (10)	−0.0001 (10)	0.0092 (11)
O3	0.0317 (14)	0.0506 (15)	0.0424 (15)	−0.0045 (12)	0.0031 (10)	−0.0080 (11)
O4	0.0311 (13)	0.0363 (13)	0.0635 (17)	0.0029 (11)	0.0164 (11)	0.0084 (12)
F2	0.0575 (14)	0.0938 (17)	0.0456 (15)	−0.0085 (12)	−0.0073 (10)	−0.0017 (12)
F1	0.0726 (16)	0.0703 (15)	0.0663 (16)	−0.0189 (12)	−0.0069 (12)	−0.0205 (12)
C15	0.049 (2)	0.0419 (19)	0.034 (2)	−0.0009 (17)	0.0049 (16)	0.0006 (16)
C1	0.0267 (18)	0.0323 (16)	0.0289 (18)	−0.0018 (14)	−0.0024 (13)	−0.0011 (14)

C8	0.0302 (18)	0.0283 (16)	0.046 (2)	−0.0047 (14)	0.0023 (15)	−0.0028 (14)
C2	0.0221 (16)	0.0286 (16)	0.039 (2)	0.0030 (13)	0.0053 (13)	0.0005 (14)
C6	0.046 (2)	0.048 (2)	0.038 (2)	−0.0018 (17)	0.0053 (16)	0.0051 (17)
C14	0.038 (2)	0.0380 (18)	0.0298 (19)	0.0003 (15)	0.0071 (14)	0.0016 (15)
C3	0.0244 (17)	0.0344 (17)	0.039 (2)	0.0024 (14)	0.0027 (14)	0.0016 (15)
C7	0.0326 (19)	0.0334 (17)	0.037 (2)	0.0036 (14)	0.0092 (14)	0.0017 (15)
C5	0.050 (2)	0.0404 (19)	0.046 (2)	−0.0122 (17)	0.0131 (17)	0.0055 (17)
C9	0.0301 (19)	0.0355 (18)	0.045 (2)	0.0006 (14)	0.0005 (15)	−0.0024 (15)
C13	0.0250 (18)	0.0425 (19)	0.052 (2)	−0.0040 (15)	0.0025 (15)	−0.0006 (17)
C19	0.055 (2)	0.0374 (19)	0.043 (2)	0.0013 (17)	−0.0025 (17)	0.0010 (16)
C10	0.036 (2)	0.047 (2)	0.045 (2)	0.0004 (15)	0.0014 (16)	−0.0009 (17)
C4	0.035 (2)	0.0391 (19)	0.053 (2)	−0.0065 (15)	0.0053 (16)	−0.0005 (17)
C11	0.050 (2)	0.049 (2)	0.038 (2)	−0.0034 (17)	−0.0063 (17)	−0.0002 (17)
C12	0.032 (2)	0.051 (2)	0.049 (2)	−0.0045 (16)	−0.0059 (16)	−0.0017 (18)
C17	0.049 (2)	0.056 (2)	0.039 (2)	−0.0108 (19)	0.0065 (17)	−0.015 (2)
C21	0.051 (2)	0.044 (2)	0.060 (3)	−0.0037 (18)	0.0096 (18)	0.0131 (18)
C20	0.053 (2)	0.053 (2)	0.062 (3)	0.0039 (19)	0.0097 (19)	−0.011 (2)
C18	0.065 (3)	0.037 (2)	0.049 (2)	−0.0060 (19)	0.0053 (19)	−0.0065 (18)
C16	0.047 (2)	0.059 (2)	0.036 (2)	0.0054 (18)	−0.0005 (17)	−0.0051 (18)
O5	0.0352 (13)	0.0449 (14)	0.0553 (17)	0.0028 (11)	0.0104 (12)	0.0055 (12)
O6	0.0331 (14)	0.0410 (13)	0.0710 (18)	−0.0088 (11)	0.0044 (11)	0.0040 (13)
C23	0.045 (2)	0.039 (2)	0.067 (3)	−0.0086 (17)	0.0080 (18)	0.0013 (18)
C22	0.054 (2)	0.060 (2)	0.052 (3)	0.006 (2)	0.0038 (18)	0.001 (2)

Geometric parameters (Å, º)

Ca1—O1^{i}	2.2692 (19)	C9—C10	1.375 (4)
Ca1—O1	2.2693 (19)	C9—H9	0.9500
Ca1—O4^{i}	2.325 (2)	C13—C12	1.368 (4)
Ca1—O4	2.325 (2)	C13—H13	0.9500
Ca1—O3	2.346 (2)	C19—C18	1.388 (4)
Ca1—O3^{i}	2.346 (2)	C19—H19	0.9500
O2—C1	1.269 (3)	C10—C11	1.375 (5)
O1—C1	1.253 (3)	C10—H10	0.9500
O3—C20	1.418 (4)	C4—H4	0.9500
O3—H1	0.89 (4)	C11—C12	1.371 (5)
O4—C21	1.425 (4)	C12—H12	0.9500
O4—H2	0.91 (4)	C17—C18	1.364 (5)
F2—C11	1.363 (4)	C17—C16	1.367 (5)
F1—C17	1.362 (4)	C21—H21A	0.9800
C15—C16	1.382 (5)	C21—H21B	0.9800
C15—C14	1.395 (4)	C21—H21C	0.9800
C15—H15	0.9500	C20—H20A	0.9800
C1—C2	1.511 (4)	C20—H20B	0.9800
C8—C13	1.400 (4)	C20—H20C	0.9800
C8—C9	1.401 (4)	C18—H18	0.9500
C8—C3	1.479 (4)	C16—H16	0.9500
C2—C7	1.403 (4)	O5—C22	1.415 (4)

C2—C3	1.404 (4)	O5—H5A	0.8400
C6—C5	1.377 (5)	O6—C23	1.416 (4)
C6—C7	1.403 (4)	O6—H6A	0.8400
C6—H6	0.9500	C23—H23A	0.9800
C14—C19	1.380 (4)	C23—H23B	0.9800
C14—C7	1.492 (4)	C23—H23C	0.9800
C3—C4	1.408 (4)	C22—H22A	0.9800
C5—C4	1.363 (5)	C22—H22B	0.9800
C5—H5	0.9500	C22—H22C	0.9800

O1^{i}—Ca1—O1	180.00 (8)	C12—C13—H13	119.1
O1^{i}—Ca1—O4^{i}	95.07 (8)	C8—C13—H13	119.1
O1—Ca1—O4^{i}	84.93 (8)	C14—C19—C18	121.2 (3)
O1^{i}—Ca1—O4	84.93 (8)	C14—C19—H19	119.4
O1—Ca1—O4	95.07 (8)	C18—C19—H19	119.4
O4^{i}—Ca1—O4	180.0	C9—C10—C11	117.9 (3)
O1^{i}—Ca1—O3	90.78 (8)	C9—C10—H10	121.0
O1—Ca1—O3	89.22 (8)	C11—C10—H10	121.0
O4^{i}—Ca1—O3	89.48 (8)	C5—C4—C3	121.5 (3)
O4—Ca1—O3	90.52 (8)	C5—C4—H4	119.2
O1^{i}—Ca1—O3^{i}	89.22 (8)	C3—C4—H4	119.2
O1—Ca1—O3^{i}	90.78 (8)	F2—C11—C12	119.4 (3)
O4^{i}—Ca1—O3^{i}	90.52 (8)	F2—C11—C10	118.1 (3)
O4—Ca1—O3^{i}	89.48 (8)	C12—C11—C10	122.5 (3)
O3—Ca1—O3^{i}	180.0	C13—C12—C11	118.7 (3)
C1—O1—Ca1	171.8 (2)	C13—C12—H12	120.6
C20—O3—Ca1	128.9 (2)	C11—C12—H12	120.6
C20—O3—H1	110 (3)	F1—C17—C18	118.4 (3)
Ca1—O3—H1	118 (3)	F1—C17—C16	119.3 (3)
C21—O4—Ca1	130.6 (2)	C18—C17—C16	122.2 (3)
C21—O4—H2	112 (2)	O4—C21—H21A	109.5
Ca1—O4—H2	116 (2)	O4—C21—H21B	109.5
C16—C15—C14	121.2 (3)	H21A—C21—H21B	109.5
C16—C15—H15	119.4	O4—C21—H21C	109.5
C14—C15—H15	119.4	H21A—C21—H21C	109.5
O1—C1—O2	124.1 (3)	H21B—C21—H21C	109.5
O1—C1—C2	117.8 (3)	O3—C20—H20A	109.5
O2—C1—C2	118.1 (3)	O3—C20—H20B	109.5
C13—C8—C9	117.0 (3)	H20A—C20—H20B	109.5
C13—C8—C3	121.1 (3)	O3—C20—H20C	109.5
C9—C8—C3	121.9 (3)	H20A—C20—H20C	109.5
C7—C2—C3	120.8 (3)	H20B—C20—H20C	109.5
C7—C2—C1	119.9 (3)	C17—C18—C19	118.7 (3)
C3—C2—C1	119.3 (3)	C17—C18—H18	120.6
C5—C6—C7	120.0 (3)	C19—C18—H18	120.6
C5—C6—H6	120.0	C17—C16—C15	118.6 (3)
C7—C6—H6	120.0	C17—C16—H16	120.7
C19—C14—C15	118.1 (3)	C15—C16—H16	120.7

C19—C14—C7	122.4 (3)	C22—O5—H5A	109.5
C15—C14—C7	119.5 (3)	C23—O6—H6A	109.5
C2—C3—C4	117.7 (3)	O6—C23—H23A	109.5
C2—C3—C8	123.6 (3)	O6—C23—H23B	109.5
C4—C3—C8	118.7 (3)	H23A—C23—H23B	109.5
C6—C7—C2	119.1 (3)	O6—C23—H23C	109.5
C6—C7—C14	118.9 (3)	H23A—C23—H23C	109.5
C2—C7—C14	122.0 (3)	H23B—C23—H23C	109.5
C4—C5—C6	120.8 (3)	O5—C22—H22A	109.5
C4—C5—H5	119.6	O5—C22—H22B	109.5
C6—C5—H5	119.6	H22A—C22—H22B	109.5
C10—C9—C8	122.1 (3)	O5—C22—H22C	109.5
C10—C9—H9	119.0	H22A—C22—H22C	109.5
C8—C9—H9	119.0	H22B—C22—H22C	109.5
C12—C13—C8	121.8 (3)		

Symmetry code: (i) $-x+2, -y, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O6^{ii}	0.89 (4)	1.78 (4)	2.636 (3)	162 (4)
O4—H2···O5^{iii}	0.91 (4)	1.76 (4)	2.660 (3)	172 (4)
O6—H6*A*···O2^{iv}	0.84	1.96	2.804 (3)	177
O5—H5*A*···O2^{v}	0.84	1.92	2.755 (3)	170

Symmetry codes: (ii) $x, y-1, z$; (iii) $x+1, y-1, z$; (iv) $-x+3/2, y+1/2, z$; (v) $-x+1, -y+1, -z+1$.