

Poly[[diaquacerium(III)]- μ_5 -propane-1,2,3-tricarboxylato]Jennifer A. Armstrong and
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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.026

wR factor = 0.064

Data-to-parameter ratio = 14.1

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Crystals of polymeric cerium(III) 1,2,3-propanetricarboxylate dihydrate, $[\text{Ce}(\text{C}_6\text{H}_5\text{O}_6)(\text{H}_2\text{O})_2]_n$, were grown under hydrothermal conditions. The nine-coordinate Ce atoms occur as centrosymmetric pairs. Two bidentate and three monodentate carboxylate groups, each from a different anion, and two water molecules are coordinated to each cerium ion. Ce—O distances range from 2.390 (3) to 2.637 (2) Å. Each anion is joined to five cerium ions, forming a three-dimensional network.

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Comment

Metal salts of polycarboxylic acids can often be prepared only as powders by precipitation from aqueous solution. The very low solubilities of these powders frequently defeat attempts to grow single crystals from solution. For example, in spite of many attempts in this laboratory, only one metal salt of tetrahydrofuran tetracarboxylic acid has been obtained with crystals adequate for structure determination (Barnes & Paton, 1982). The exploitation of hydrothermal recrystallization by Yaghi *et al.* (1996) and Plater *et al.* (1997) offers a new route to these crystals. These workers have used the relatively rigid 1,3,5-benzenetricarboxylate anion in attempts to stabilize open-network structures. In the present work, we report the hydrothermal recrystallization and structure of a cerium(III) salt, (I), of the very flexible 1,3,5-propanetricarboxylate anion (tca).

Barnes & Paton (1988) reported the crystal structure of 1,2,3-propanetricarboxylic acid (H_3tca) at 298 K. A re-determination at 150 K, using better crystals, shows that no phase changes occur over this temperature range (Barnes, 2004).

Gupta & Powel (1963, 1964) prepared powdered samples of lanthanide–tca salts by precipitation from solutions at pH 4–5. They reported that, at room temperature, lanthanum formed $\text{La}(\text{tca})\cdot 5\text{H}_2\text{O}$, but all other lanthanides studied gave $\text{Ln}(\text{tca})\cdot 4\text{H}_2\text{O}$. From thermogravimetric analysis (TGA) measurements, they concluded that $\text{Ln}(\text{tca})\cdot n\text{H}_2\text{O}$ lost water by several different sequences to give anhydrous $\text{Ln}(\text{tca})$ between 423 and 473 K.

Solution studies by Barnes & Bristow (1969) showed the existence of LnHtca^+ and Lntca in the pH range 2–5. Almost quantitative precipitation occurs above pH 5.0.

In the present work, hydrothermal recrystallization of $\text{Ce}(\text{tca})\cdot 4\text{H}_2\text{O}$ powder gave crystals shown by thermogravimetry and structural analysis to be $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$, (I). Data were collected at 150 and 298 K using different crystals. There were no significant structural differences but the room-temperature crystal proved to be of much better quality; *R* is 0.028 compared with 0.055 for the low-temperature crystal.

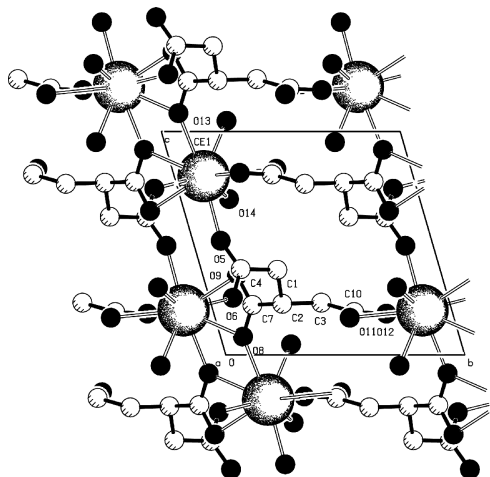


Figure 1
The structure of $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$, viewed down a .

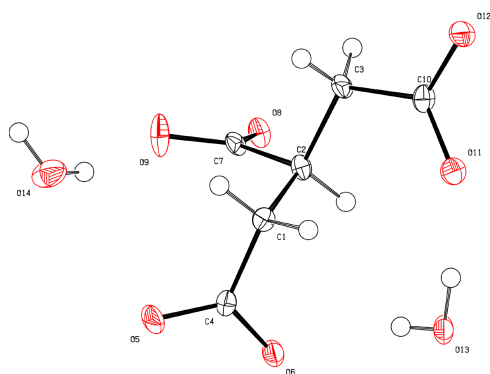


Figure 2
The tca anion and the water molecules, showing 50% probability displacement ellipsoids.

The low-temperature cell has $a = 6.840(6)$ Å, $b = 8.687(11)$ Å, $c = 8.819(14)$ Å, $\alpha = 99.72(17)^\circ$, $\beta = 110.25(7)^\circ$ and $\gamma = 110.25(7)^\circ$.

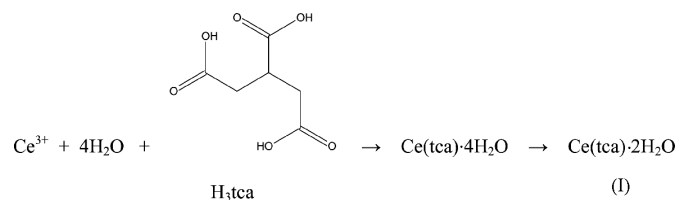


Fig. 1 shows that the Ce atoms occur as centrosymmetric pairs, bridged by the O8 atoms of two bidentate carboxylate groups. The nine-coordinate Ce^{3+} ion has an unusual 2–5–2 geometry, with *trans* bidentate carboxylate groups (C7/O8/O9 and C10/O11/O12) from different ligands. The angle between these carboxylate planes is $89.3(4)^\circ$. Atoms O5, O6 and O8 from three more ligands and two water molecules O13 and O14 make up a pentagonal median plane (r.m.s. deviation = 0.344 Å) at $88.4(3)^\circ$ to the plane Ce/O11/O12. (A more familiar but less precise visualization is a monocapped square antiprism with O9 as the cap.) Ce–O distances are in the ranges $2.390(3)$ – $2.489(2)$ Å (monodentate), $2.505(3)$ – $2.516(2)$ Å (water) and $2.544(2)$ – $2.637(2)$ Å (bidentate carboxylate groups).

The tca anion is shown in Fig. 2, with torsion angles in Table 1. Each anion is joined to five Ce atoms to form a three-dimensional network. This flexible anion allows the formation of a framework with no significant volumes of free space, unlike the open-network structures reported by Plater *et al.* (1997) for complexes of the rigid 1,3,5-benzenetricarboxylate anion.

The coordinated water molecules contribute to the network by inter-unit hydrogen bonding (Table 2).

X-ray powder diffraction showed that the powder sample of $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$ prepared at room temperature is identical to the observations of $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$ crystals. TGA studies on $\text{Ce}(\text{tca})\cdot 4\text{H}_2\text{O}$ confirmed the observation of Gupta & Powel (1963, 1964) that $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$ is not formed by dehydration of $\text{Ce}(\text{tca})\cdot 4\text{H}_2\text{O}$ at atmospheric pressure. In the present work, highly crystalline $\text{Ce}(\text{tca})\cdot 4\text{H}_2\text{O}$ was converted at 423 K into the monohydrate which had not been observed by Gupta & Powel. This $\text{Ce}(\text{tca})\cdot \text{H}_2\text{O}$ is of very low crystallinity. $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$ was thermally stable to 400 K, forming crystalline anhydrous $\text{Ce}(\text{tca})$ by 430 K.

Experimental

Two samples of hydrated $\text{Ce}(\text{tca})$ salts were prepared by mixing aqueous CeCl_3 and H_3tca solutions (0.01 mol) at room temperature and adjusting the pH to 5.5 with NaOH. The resulting fine precipitates were collected after stirring for 1 h. Analysis showed that one sample was $\text{Ce}(\text{tca})\cdot 4\text{H}_2\text{O}$ and the other $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$, in spite of no intentional differences in preparation.

Analytical data for $\text{Ce}(\text{tca})\cdot 4\text{H}_2\text{O}$: Ce found by edta titration: 37.92%; required: 38.14%. Thermogravimetry: weight loss to 423 K = 14.53%, required for $\text{Ce}(\text{tca})\cdot \text{H}_2\text{O}$ = 14.72%; weight loss to 1200 K = 51.68%, required for CeO_2 = 52.05%

Analytical data for $\text{Ce}(\text{tca})\cdot 2\text{H}_2\text{O}$: Ce found by edta titration 39.83%, required = 40.11%. Thermogravimetry: weight loss to 430 K = 10.42%, required for $\text{Ce}(\text{tca})$ = 10.31%, weight loss to 1200 K = 49.83%, required for CeO_2 = 49.58%.

Using a Parr model 4745 bomb with a PTFE liner, 0.5 g of $\text{Ce}(\text{tca})\cdot 4\text{H}_2\text{O}$ and 15 ml of water were heated to 423 K for 16 h, cooled at 7.5 K h^{-1} to 363 K and then allowed to cool naturally to room temperature. Colourless poorly shaped crystals were obtained.

Crystal data

$[\text{Ce}(\text{C}_6\text{H}_5\text{O}_6)(\text{H}_2\text{O})_2]$
 $M_r = 349.25$
 Triclinic, $P\bar{1}$
 $a = 6.9084(2)$ Å
 $b = 8.7738(4)$ Å
 $c = 8.8162(4)$ Å
 $\alpha = 99.552(2)^\circ$
 $\beta = 110.209(2)^\circ$
 $\gamma = 104.461(2)^\circ$
 $V = 466.51(3)$ Å³

$Z = 2$
 $D_x = 2.486 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 5965 reflections
 $\theta = 2.6$ – 27.5°
 $\mu = 4.90 \text{ mm}^{-1}$
 $T = 150(2)$ K
 Block, colourless
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SORTAV; Blessing, 1995)
 $T_{\min} = 0.353$, $T_{\max} = 0.613$
 5965 measured reflections

2091 independent reflections
 2046 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$
 $\theta_{\max} = 27.5^\circ$
 $h = -8 \rightarrow 8$
 $k = -11 \rightarrow 9$
 $l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.064$
 $S = 1.13$
 2091 reflections
 148 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 0.7556P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.018$
 $\Delta\rho_{\max} = 1.05 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -2.89 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Ce1—O5 ⁱ	2.390 (3)	C1—C2	1.539 (4)
Ce1—O6	2.489 (2)	C2—C7	1.517 (5)
Ce1—O14	2.505 (3)	C2—C3	1.537 (5)
Ce1—O13	2.518 (2)	C3—C10	1.513 (5)
Ce1—O12 ⁱⁱ	2.544 (2)	C4—O5	1.237 (4)
Ce1—O8 ⁱⁱⁱ	2.572 (2)	C4—O6	1.267 (4)
Ce1—O9 ^{iv}	2.611 (2)	C7—O9	1.251 (4)
Ce1—O8 ^{iv}	2.619 (2)	C7—O8	1.272 (4)
Ce1—O11 ⁱⁱ	2.637 (2)	C10—O12	1.257 (4)
C1—C4	1.510 (4)	C10—O11	1.270 (4)
O5—C4—C1—C2	−126.3 (3)	O5—C4—C1—C2	−126.3 (3)
C4—C1—C2—C3	−176.7 (3)	C4—C1—C2—C7	61.7 (4)
C1—C2—C3—C10	72.6 (4)	C1—C2—C7—O8	−151.6 (3)
C2—C3—C10—O11	−4.9 (5)		

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

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O13—H131 ^v ···O11 ^v	0.86 (5)	1.95 (5)	2.810 (3)	174 (5)
O13—H132 ^v ···O6 ^v	0.79 (5)	1.99 (3)	2.741 (4)	160 (5)
O14—H141 ⁱ ···O9 ⁱ	0.89 (5)	1.96 (5)	2.830 (4)	165 (5)
O14—H142 ^{vi} ···O12 ^{vi}	0.83 (5)	1.91 (5)	2.718 (4)	165 (5)

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

H atoms of the tricarballylate anion were placed in calculated positions with U_{iso} values set at 1.3 times the U_{eq} value of the parent atom and the C—H distance set at 0.97 \AA . H atoms of water were located in a difference synthesis and the coordinates were refined with U_{iso} values set at 1.3 times the U_{eq} value of the parent atom; the O—H distances were not constrained. The highest difference map peak was 0.83 \AA from Ce1 and the deepest hole was 0.86 \AA from Ce1.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL97*.

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Poly[[diaquacerium(III)]- μ_5 -propane-1,2,3-tricarboxylato]*Crystal data*

[Ce(C₆H₅O₆)(H₂O)₂]

$M_r = 349.25$

Triclinic, $P\bar{1}$

$a = 6.9084$ (2) Å

$b = 8.7738$ (4) Å

$c = 8.8162$ (4) Å

$\alpha = 99.552$ (2)°

$\beta = 110.209$ (2)°

$\gamma = 104.461$ (2)°

$V = 466.51$ (3) Å³

$Z = 2$

$F(000) = 334$

$D_x = 2.486$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5965 reflections

$\theta = 2.6$ – 27.5 °

$\mu = 4.90$ mm⁻¹

$T = 150$ K

Block, colourless

$0.30 \times 0.20 \times 0.10$ mm

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer

Radiation source: Enraf Nonius FR591 rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels/mm pixels mm⁻¹

π and ω scans

Absorption correction: multi-scan (SORTAV; Blessing, 1995)

$T_{\min} = 0.353$, $T_{\max} = 0.613$

5965 measured reflections

2091 independent reflections

2046 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.6$ °

$h = -8 \rightarrow 8$

$k = -11 \rightarrow 9$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.064$

$S = 1.13$

2091 reflections

148 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 0.7556P]$

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

$(\Delta/\sigma)_{\max} = 0.018$

$\Delta\rho_{\max} = 1.05$ e Å⁻³

$\Delta\rho_{\min} = -2.89$ 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.

H atoms of tricarallylate anion placed on calculated positions with $U(\text{iso})$ set to 1.3 times that of parent atom. H atoms of water located on a difference synthesis, coordinates refined with $U(\text{iso})$ set to 1.3 times that of parent atom. The O - H distances were not constrained.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ce1	0.02449 (2)	0.876953 (16)	0.199738 (17)	0.00936 (9)
C1	-0.2152 (5)	1.3184 (4)	0.3811 (4)	0.0125 (6)
H1A	-0.0956	1.4050	0.3820	0.016*
H1B	-0.2354	1.3532	0.4833	0.016*
C2	-0.4245 (5)	1.2984 (4)	0.2294 (4)	0.0142 (7)
H2	-0.4044	1.2626	0.1262	0.018*
C3	-0.4664 (6)	1.4628 (4)	0.2342 (5)	0.0161 (7)
H3A	-0.6153	1.4417	0.1558	0.021*
H3B	-0.4552	1.5095	0.3459	0.021*
C4	-0.1505 (5)	1.1664 (4)	0.3843 (4)	0.0135 (6)
O5	-0.1194 (5)	1.1165 (3)	0.5111 (3)	0.0265 (6)
O6	-0.1279 (4)	1.0985 (3)	0.2560 (3)	0.0203 (5)
C7	-0.6234 (5)	1.1717 (4)	0.2242 (4)	0.0130 (7)
O8	-0.7802 (3)	1.0940 (3)	0.0821 (3)	0.0155 (5)
O9	-0.6356 (4)	1.1430 (3)	0.3555 (3)	0.0250 (6)
C10	-0.3142 (6)	1.5888 (4)	0.1922 (4)	0.0153 (7)
O11	-0.1532 (4)	1.5636 (3)	0.1668 (3)	0.0217 (5)
O12	-0.3486 (4)	1.7220 (3)	0.1851 (3)	0.0221 (5)
O13	0.0587 (5)	0.7143 (3)	-0.0477 (3)	0.0239 (6)
H131	0.091 (8)	0.628 (6)	-0.077 (6)	0.031*
H132	0.107 (7)	0.764 (5)	-0.100 (5)	0.031*
O14	0.3613 (5)	0.7979 (4)	0.3020 (4)	0.0280 (6)
H141	0.434 (8)	0.827 (6)	0.414 (7)	0.036*
H142	0.436 (8)	0.777 (6)	0.250 (6)	0.036*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ce1	0.01162 (13)	0.00675 (12)	0.01070 (13)	0.00144 (8)	0.00547 (9)	0.00535 (8)
C1	0.0079 (13)	0.0101 (13)	0.0159 (15)	-0.0011 (11)	0.0039 (11)	0.0026 (11)
C2	0.0164 (15)	0.0114 (14)	0.0148 (16)	0.0034 (12)	0.0065 (13)	0.0056 (12)

C3	0.0164 (17)	0.0137 (15)	0.0232 (18)	0.0063 (13)	0.0102 (14)	0.0109 (14)
C4	0.0124 (14)	0.0155 (14)	0.0110 (14)	0.0019 (11)	0.0044 (11)	0.0042 (11)
O5	0.0443 (16)	0.0342 (14)	0.0174 (12)	0.0263 (13)	0.0173 (11)	0.0181 (11)
O6	0.0350 (14)	0.0186 (12)	0.0179 (11)	0.0151 (10)	0.0162 (10)	0.0098 (9)
C7	0.0123 (15)	0.0086 (14)	0.0179 (17)	0.0022 (12)	0.0053 (13)	0.0067 (12)
O8	0.0137 (10)	0.0128 (10)	0.0138 (11)	-0.0010 (8)	0.0018 (8)	0.0047 (8)
O9	0.0225 (12)	0.0269 (13)	0.0160 (12)	-0.0060 (10)	0.0044 (10)	0.0113 (10)
C10	0.0129 (15)	0.0131 (16)	0.0167 (16)	-0.0006 (12)	0.0056 (13)	0.0041 (13)
O11	0.0258 (13)	0.0118 (11)	0.0357 (14)	0.0059 (10)	0.0202 (11)	0.0113 (10)
O12	0.0226 (12)	0.0144 (11)	0.0394 (15)	0.0074 (9)	0.0199 (11)	0.0150 (10)
O13	0.0448 (16)	0.0201 (13)	0.0247 (13)	0.0185 (12)	0.0257 (12)	0.0140 (10)
O14	0.0253 (14)	0.0452 (17)	0.0171 (13)	0.0199 (12)	0.0079 (11)	0.0063 (12)

Geometric parameters (Å, °)

Ce1—O5 ⁱ	2.390 (3)	C2—C3	1.537 (5)
Ce1—O6	2.489 (2)	C2—H2	0.9800
Ce1—O14	2.505 (3)	C3—C10	1.513 (5)
Ce1—O13	2.518 (2)	C3—H3A	0.9700
Ce1—O12 ⁱⁱ	2.544 (2)	C3—H3B	0.9700
Ce1—O8 ⁱⁱⁱ	2.572 (2)	C4—O5	1.237 (4)
Ce1—O9 ^{iv}	2.611 (2)	C4—O6	1.267 (4)
Ce1—O8 ^{iv}	2.619 (2)	C7—O9	1.251 (4)
Ce1—O11 ⁱⁱ	2.637 (2)	C7—O8	1.272 (4)
Ce1—C10 ⁱⁱ	2.958 (4)	C10—O12	1.257 (4)
Ce1—C7 ^{iv}	2.994 (3)	C10—O11	1.270 (4)
C1—C4	1.510 (4)	O13—H131	0.86 (5)
C1—C2	1.539 (4)	O13—H132	0.79 (5)
C1—H1A	0.9700	O14—H141	0.89 (5)
C1—H1B	0.9700	O14—H142	0.83 (5)
C2—C7	1.517 (5)		
O5 ⁱ —Ce1—O6	86.43 (8)	O9 ^{iv} —Ce1—C10 ⁱⁱ	152.85 (9)
O5 ⁱ —Ce1—O14	71.53 (9)	O8 ^{iv} —Ce1—C10 ⁱⁱ	157.50 (9)
O6—Ce1—O14	142.83 (9)	O11 ⁱⁱ —Ce1—C10 ⁱⁱ	25.42 (9)
O5 ⁱ —Ce1—O13	136.27 (9)	O5 ⁱ —Ce1—C7 ^{iv}	101.71 (10)
O6—Ce1—O13	137.29 (8)	O6—Ce1—C7 ^{iv}	78.77 (9)
O14—Ce1—O13	70.71 (9)	O14—Ce1—C7 ^{iv}	77.01 (10)
O5 ⁱ —Ce1—O12 ⁱⁱ	80.17 (9)	O13—Ce1—C7 ^{iv}	90.53 (9)
O6—Ce1—O12 ⁱⁱ	76.77 (8)	O12 ⁱⁱ —Ce1—C7 ^{iv}	155.30 (8)
O14—Ce1—O12 ⁱⁱ	125.86 (10)	O8 ⁱⁱⁱ —Ce1—C7 ^{iv}	88.34 (8)
O13—Ce1—O12 ⁱⁱ	105.21 (9)	O9 ^{iv} —Ce1—C7 ^{iv}	24.59 (9)
O5 ⁱ —Ce1—O8 ⁱⁱⁱ	153.05 (8)	O8 ^{iv} —Ce1—C7 ^{iv}	25.08 (8)
O6—Ce1—O8 ⁱⁱⁱ	70.91 (7)	O11 ⁱⁱ —Ce1—C7 ^{iv}	154.72 (8)
O14—Ce1—O8 ⁱⁱⁱ	135.40 (8)	C10 ⁱⁱ —Ce1—C7 ^{iv}	177.42 (8)
O13—Ce1—O8 ⁱⁱⁱ	67.52 (8)	C4—C1—C2	115.1 (2)
O12 ⁱⁱ —Ce1—O8 ⁱⁱⁱ	80.56 (8)	C4—C1—H1A	108.5
O5 ⁱ —Ce1—O9 ^{iv}	77.23 (9)	C2—C1—H1A	108.5

O6—Ce1—O9 ^{iv}	75.00 (9)	C4—C1—H1B	108.5
O14—Ce1—O9 ^{iv}	71.21 (9)	C2—C1—H1B	108.5
O13—Ce1—O9 ^{iv}	109.96 (9)	H1A—C1—H1B	107.5
O12 ⁱⁱ —Ce1—O9 ^{iv}	144.63 (8)	C7—C2—C3	109.0 (3)
O8 ⁱⁱⁱ —Ce1—O9 ^{iv}	109.35 (7)	C7—C2—C1	112.0 (3)
O5 ⁱ —Ce1—O8 ^{iv}	126.58 (9)	C3—C2—C1	110.4 (2)
O6—Ce1—O8 ^{iv}	79.56 (8)	C7—C2—H2	108.4
O14—Ce1—O8 ^{iv}	89.83 (9)	C3—C2—H2	108.4
O13—Ce1—O8 ^{iv}	74.36 (8)	C1—C2—H2	108.4
O12 ⁱⁱ —Ce1—O8 ^{iv}	142.78 (8)	C10—C3—C2	115.6 (3)
O8 ⁱⁱⁱ —Ce1—O8 ^{iv}	64.65 (8)	C10—C3—H3A	108.4
O9 ^{iv} —Ce1—O8 ^{iv}	49.36 (7)	C2—C3—H3A	108.4
O5 ⁱ —Ce1—O11 ⁱⁱ	80.15 (9)	C10—C3—H3B	108.4
O6—Ce1—O11 ⁱⁱ	126.43 (8)	C2—C3—H3B	108.4
O14—Ce1—O11 ⁱⁱ	79.82 (9)	H3A—C3—H3B	107.4
O13—Ce1—O11 ⁱⁱ	72.25 (8)	O5—C4—O6	123.7 (3)
O12 ⁱⁱ —Ce1—O11 ⁱⁱ	49.96 (7)	O5—C4—C1	119.0 (3)
O8 ⁱⁱⁱ —Ce1—O11 ⁱⁱ	101.34 (8)	O6—C4—C1	117.3 (3)
O9 ^{iv} —Ce1—O11 ⁱⁱ	147.60 (8)	O9—C7—O8	119.9 (3)
O8 ^{iv} —Ce1—O11 ⁱⁱ	146.60 (7)	O9—C7—C2	121.6 (3)
O5 ⁱ —Ce1—C10 ⁱⁱ	75.71 (10)	O8—C7—C2	118.5 (3)
O6—Ce1—C10 ⁱⁱ	101.01 (9)	O12—C10—O11	120.1 (3)
O14—Ce1—C10 ⁱⁱ	101.94 (11)	O12—C10—C3	118.2 (3)
O13—Ce1—C10 ⁱⁱ	91.34 (9)	O11—C10—C3	121.7 (3)
O12 ⁱⁱ —Ce1—C10 ⁱⁱ	24.97 (9)	O12—C10—Ce1 ^v	58.71 (18)
O8 ⁱⁱⁱ —Ce1—C10 ⁱⁱ	94.03 (9)		
O5—C4—C1—C2	-126.3 (3)	O5—C4—C1—C2	-126.3 (3)
C4—C1—C2—C3	-176.7 (3)	C4—C1—C2—C7	61.7 (4)
C1—C2—C3—C10	72.6 (4)	C1—C2—C7—O8	-151.6 (3)
C2—C3—C10—O11	-4.9 (5)		

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

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>
O13—H131 \cdots O11 ^{vi}	0.86 (5)	1.95 (5)	2.810 (3)	174 (5)
O13—H132 \cdots O6 ^{vi}	0.79 (5)	1.99 (3)	2.741 (4)	160 (5)
O14—H141 \cdots O9 ⁱ	0.89 (5)	1.96 (5)	2.830 (4)	165 (5)
O14—H142 \cdots O12 ^{vii}	0.83 (5)	1.91 (5)	2.718 (4)	165 (5)

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