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Poly[tris(μ_3-5-aminoisophthalato)diaquadicerium(III)]

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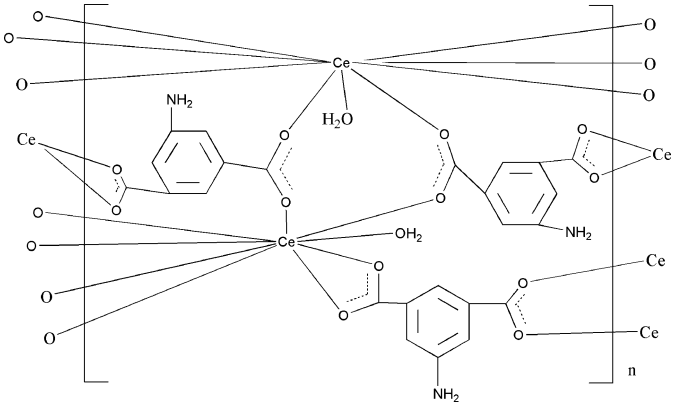
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.019; wR factor = 0.045; data-to-parameter ratio = 11.4.

In the title complex, $[Ce_2(C_8H_5NO_4)_3(H_2O)_2]_n$, each Ce ion is in nine-coordinated environment. Eight O atoms from six ligands participate in coordination, in addition to one O atom from a water molecule. Both carboxylate groups from the ligands chelate the Ce atoms, forming two four-membered rings. The 5-aminoisophthalate ligands also bridge the Ce centers, forming a two-dimensional network, and O—H···O and N—H···O hydrogen bonds complete the structure.

Related literature

For general background, see: Rzaczynska & Belsky (1994); Daiguebonne *et al.* (2000); Wu *et al.* (2002*a*,*b*); Liao *et al.* (2004).



Experimental

Crystal data

$[Ce_2(C_8H_5NO_4)_3(H_2O)_2]$
M_r = 853.66
Orthorhombic, *Pbcn*
a = 12.2360 (7) Å
b = 8.0600 (5) Å
c = 25.6700 (15) Å
V = 2531.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.63 mm^{-1}
T = 298 (2) K
0.21 × 0.20 × 0.19 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.516, T_{max} = 0.545
(expected range = 0.474–0.501)
11818 measured reflections
2233 independent reflections
2001 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.045
S = 1.04
2233 reflections
196 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O5^{i}	0.86	2.63	3.436 (3)	157
N1—H1D···O1^{ii}	0.86	2.39	3.154 (3)	148
O1W—H1A···O3^{i}	0.85	2.07	2.898 (3)	165

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2095).

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supporting information

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Poly[tris(μ_3-5-aminoisophthalato)diaquadicerium(III)]

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S1. Comment

5-aminoisophthalic acid forms covalent bonds with metal ions, especially with transition metals through nitrogen atom of amino group as well as oxygen atoms of carboxylic groups (Wu *et al.*, 2002a; Wu *et al.*, 2002b; Liao *et al.*, 2004) and with lanthanide ions as strong Pearsons acids through oxygen atoms of carboxylic groups (Rzaczynska *et al.*, 1994). Carboxylic groups of acid have a great ability to form infinite connection with metal ions and remarkable versatility in adopting different modes of bonding-from unidendate, chelating and bridging, sometimes in more than one way in the same compound (Daiguebonne *et al.*, 2000). In this paper, we present a title complex, $(C_{24}H_{19}Ce_2N_3O_{14})_n$, (I), synthesized by a condensation reaction of 5-aminoisophthalic acid with cerous nitrate under the condition of high pressure.

The molecular structure of the title complex, (I), is shown in Fig.1. The ligands construct a floor-like layer by chelating and bridging metal ions. The carboxy groups link layers in $\eta^1,^3$ mode, thus resulting in one-dimension metal-channels along *b*-axis, and the water molecules coordinating with metal ions are pending in these channels.

S2. Experimental

5-aminoisophthalic acid (0.3 mmol, 54.6 mg) and sodium hydroxide (0.3 mmol,12.5 mg) dissolved in 20 ml water, heated to boiled and then stop heating. Cerous nitrate hexahydrate (0.3 mmol,130.3 mg) dissolved in 5 ml water was mixed with the above solution, stirring for half an hour. Then transfer them into a 50 ml teflon reactor, under autogenous pressure at 160°C for 3 days and then cooled to room temperature, after which large brown block-shaped crystals of the title complex suitable for X-ray diffraction analysis were obtained.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model, with C—H 0.93 (aromatic) 0.93, N—H 0.86 (amino), O—H 0.85 Å (water), withU_{iso}(H) =1.2U_{eq}(C).

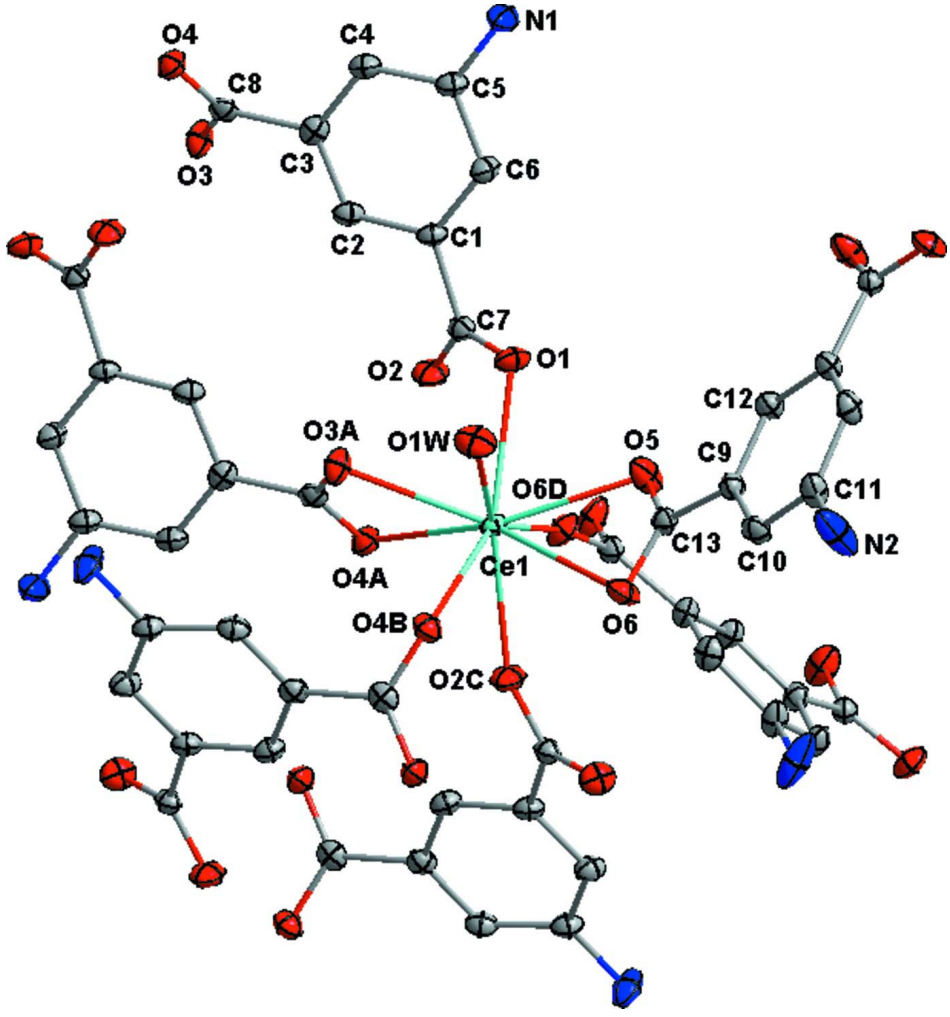

Figure 1

The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

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Crystal data

$[Ce_2(C_8H_5NO_4)_3(H_2O)_2]$
M_r = 853.66
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
a = 12.2360 (7) Å
b = 8.0600 (5) Å
c = 25.6700 (15) Å
V = 2531.6 (3) Å^3
Z = 4
$F(000)$ = 1648
D_x = 2.240 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71069 Å
Cell parameters from 3043 reflections
θ = 2.3–28.4°
μ = 3.63 mm^{-1}
T = 298 K
Block, brown
0.21 × 0.20 × 0.19 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.516, T_{max} = 0.545

11818 measured reflections
2233 independent reflections
2001 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = -14 \rightarrow 13$
$k = -9 \rightarrow 9$
$l = -30 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.045$
$S = 1.04$
2233 reflections
196 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 2.5416P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ce1	0.694451 (12)	0.038178 (18)	0.094984 (6)	0.01318 (7)
H1A	0.4903	−0.1228	0.0487	0.016*
H1B	0.4671	−0.1140	0.0998	0.016*
N1	0.1463 (2)	0.4699 (3)	0.15158 (11)	0.0302 (7)
H1C	0.1531	0.4128	0.1797	0.036*
H1D	0.1042	0.5534	0.1571	0.036*
N2	0.5000	−0.7310 (4)	0.2500	0.0418 (11)
H2A	0.5462	−0.7843	0.2311	0.050*
O1	0.53914 (15)	0.2297 (2)	0.12641 (8)	0.0227 (5)
O2	0.62001 (17)	0.4491 (3)	0.09148 (8)	0.0250 (5)
O3	0.38087 (15)	0.8402 (2)	−0.01169 (8)	0.0220 (5)
O4	0.77549 (15)	0.2778 (2)	0.03332 (8)	0.0204 (4)
O5	0.63242 (18)	−0.0423 (2)	0.18383 (9)	0.0282 (5)
O6	0.70692 (16)	−0.2648 (2)	0.15024 (8)	0.0232 (5)
O1W	0.51934 (16)	−0.1043 (3)	0.07824 (9)	0.0280 (5)
C1	0.4286 (2)	0.4564 (3)	0.09890 (11)	0.0159 (6)
C2	0.4177 (2)	0.5614 (3)	0.05642 (12)	0.0177 (6)
H2	0.4786	0.5885	0.0365	0.021*
C3	0.3163 (2)	0.6262 (3)	0.04351 (11)	0.0174 (6)
C4	0.2262 (2)	0.5922 (4)	0.07468 (12)	0.0179 (6)
H4	0.1576	0.6316	0.0652	0.021*

C5	0.2384 (2)	0.4998 (3)	0.11991 (12)	0.0189 (6)
C6	0.3393 (2)	0.4281 (3)	0.13138 (12)	0.0182 (6)
H6	0.3469	0.3613	0.1607	0.022*
C7	0.5365 (2)	0.3728 (3)	0.10682 (11)	0.0164 (6)
C8	0.3065 (2)	0.7357 (3)	−0.00284 (12)	0.0173 (6)
C9	0.5727 (2)	−0.2982 (3)	0.21853 (11)	0.0171 (6)
C10	0.5748 (2)	−0.4714 (3)	0.21935 (12)	0.0205 (6)
H10	0.6261	−0.5283	0.1995	0.025*
C11	0.5000	−0.5599 (5)	0.2500	0.0212 (9)
C12	0.5000	−0.2132 (5)	0.2500	0.0176 (9)
H12	0.5000	−0.0978	0.2500	0.021*
C13	0.6425 (2)	−0.1977 (3)	0.18270 (11)	0.0165 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ce1	0.01069 (10)	0.01242 (10)	0.01642 (12)	−0.00009 (6)	−0.00036 (6)	0.00135 (6)
N1	0.0194 (14)	0.0396 (16)	0.0314 (18)	0.0059 (12)	0.0102 (12)	0.0138 (13)
N2	0.070 (3)	0.0152 (19)	0.040 (3)	0.000	0.030 (2)	0.000
O1	0.0200 (10)	0.0169 (10)	0.0311 (13)	0.0053 (8)	0.0002 (9)	0.0026 (9)
O2	0.0132 (11)	0.0306 (12)	0.0314 (14)	−0.0012 (9)	0.0007 (9)	0.0030 (10)
O3	0.0243 (11)	0.0195 (11)	0.0224 (12)	−0.0062 (9)	−0.0041 (9)	0.0065 (9)
O4	0.0179 (10)	0.0227 (11)	0.0205 (11)	−0.0003 (8)	−0.0036 (9)	0.0016 (9)
O5	0.0395 (13)	0.0170 (11)	0.0280 (13)	0.0005 (9)	0.0133 (11)	0.0036 (9)
O6	0.0235 (11)	0.0225 (9)	0.0236 (12)	0.0025 (9)	0.0101 (9)	0.0048 (8)
O1W	0.0187 (11)	0.0323 (12)	0.0329 (13)	−0.0066 (10)	0.0010 (10)	−0.0076 (11)
C1	0.0136 (14)	0.0137 (14)	0.0204 (16)	−0.0002 (11)	−0.0011 (11)	−0.0010 (12)
C2	0.0151 (14)	0.0154 (14)	0.0226 (16)	−0.0004 (11)	0.0026 (13)	−0.0014 (12)
C3	0.0194 (15)	0.0126 (14)	0.0201 (16)	−0.0010 (11)	−0.0020 (12)	−0.0016 (12)
C4	0.0135 (13)	0.0178 (14)	0.0223 (16)	0.0017 (12)	−0.0013 (12)	−0.0024 (13)
C5	0.0140 (14)	0.0195 (14)	0.0233 (17)	0.0003 (12)	0.0011 (13)	0.0001 (13)
C6	0.0155 (14)	0.0168 (14)	0.0225 (17)	0.0002 (11)	−0.0007 (13)	0.0032 (12)
C7	0.0132 (14)	0.0191 (15)	0.0168 (15)	−0.0005 (12)	−0.0013 (12)	−0.0024 (13)
C8	0.0186 (14)	0.0156 (14)	0.0176 (16)	0.0047 (12)	−0.0009 (12)	−0.0033 (12)
C9	0.0185 (14)	0.0173 (14)	0.0155 (15)	−0.0003 (11)	0.0017 (12)	0.0005 (12)
C10	0.0255 (16)	0.0184 (14)	0.0176 (16)	0.0040 (12)	0.0050 (14)	−0.0020 (13)
C11	0.032 (2)	0.015 (2)	0.016 (2)	0.000	0.0031 (19)	0.000
C12	0.021 (2)	0.016 (2)	0.016 (2)	0.000	0.0012 (17)	0.000
C13	0.0162 (14)	0.0189 (14)	0.0144 (15)	−0.0002 (12)	0.0001 (12)	−0.0004 (12)

Geometric parameters (Å, º)

Ce1—O2^{i}	2.383 (2)	O6—Ce1^{i}	2.448 (2)
Ce1—O6^{ii}	2.448 (2)	O1W—H1A	0.8500
Ce1—O1W	2.469 (2)	O1W—H1B	0.8500
Ce1—O5	2.490 (2)	C1—C2	1.387 (4)
Ce1—O3^{iii}	2.5263 (19)	C1—C6	1.393 (4)
Ce1—O1	2.5779 (19)	C1—C7	1.496 (4)

Ce1—O4^{i}	2.654 (2)	C2—C3	1.387 (4)
Ce1—O4	2.6870 (19)	C2—H2	0.9300
Ce1—O6	2.828 (2)	C3—C4	1.389 (4)
Ce1—C8^{iii}	2.986 (3)	C3—C8	1.486 (4)
N1—C5	1.410 (4)	C4—C5	1.387 (4)
N1—H1C	0.8600	C4—H4	0.9300
N1—H1D	0.8599	C5—C6	1.395 (4)
N2—C11	1.379 (5)	C6—H6	0.9300
N2—H2A	0.8600	C8—O4^{iii}	1.276 (3)
O1—C7	1.258 (3)	C8—Ce1^{iii}	2.986 (3)
O2—C7	1.256 (3)	C9—C12	1.384 (3)
O2—Ce1^{ii}	2.383 (2)	C9—C10	1.396 (4)
O3—C8	1.261 (3)	C9—C13	1.494 (4)
O3—Ce1^{iii}	2.5263 (19)	C10—C11	1.402 (3)
O4—C8^{iii}	1.276 (3)	C10—H10	0.9300
O4—Ce1^{ii}	2.654 (2)	C11—C10^{iv}	1.402 (3)
O5—C13	1.259 (3)	C12—C9^{iv}	1.384 (3)
O6—C13	1.268 (3)	C12—H12	0.9300

O2^{i}—Ce1—O6^{ii}	75.39 (7)	C8^{iii}—O4—Ce1^{ii}	122.81 (17)
O2^{i}—Ce1—O1W	132.85 (7)	C8^{iii}—O4—Ce1	90.56 (16)
O6^{ii}—Ce1—O1W	146.17 (7)	Ce1^{ii}—O4—Ce1	105.55 (7)
O2^{i}—Ce1—O5	104.28 (7)	C13—O5—Ce1	102.02 (18)
O6^{ii}—Ce1—O5	77.79 (7)	C13—O6—Ce1^{i}	164.58 (18)
O1W—Ce1—O5	76.92 (7)	C13—O6—Ce1	85.86 (16)
O2^{i}—Ce1—O3^{iii}	115.63 (7)	Ce1^{i}—O6—Ce1	107.23 (7)
O6^{ii}—Ce1—O3^{iii}	114.74 (6)	Ce1—O1W—H1A	126.9
O1W—Ce1—O3^{iii}	73.53 (7)	Ce1—O1W—H1B	125.6
O5—Ce1—O3^{iii}	139.93 (7)	H1A—O1W—H1B	104.5
O2^{i}—Ce1—O1	153.47 (7)	C2—C1—C6	119.7 (3)
O6^{ii}—Ce1—O1	78.08 (6)	C2—C1—C7	117.8 (3)
O1W—Ce1—O1	72.14 (7)	C6—C1—C7	122.5 (2)
O5—Ce1—O1	69.18 (7)	C3—C2—C1	120.2 (3)
O3^{iii}—Ce1—O1	76.33 (7)	C3—C2—H2	119.9
O2^{i}—Ce1—O4^{i}	66.87 (7)	C1—C2—H2	119.9
O6^{ii}—Ce1—O4^{i}	142.22 (6)	C2—C3—C4	119.9 (3)
O1W—Ce1—O4^{i}	69.43 (6)	C2—C3—C8	119.1 (2)
O5—Ce1—O4^{i}	112.50 (6)	C4—C3—C8	121.0 (2)
O3^{iii}—Ce1—O4^{i}	81.52 (6)	C5—C4—C3	120.2 (3)
O1—Ce1—O4^{i}	139.66 (6)	C5—C4—H4	119.9
O2^{i}—Ce1—O4	80.95 (6)	C3—C4—H4	119.9
O6^{ii}—Ce1—O4	72.12 (7)	C4—C5—C6	119.6 (3)
O1W—Ce1—O4	123.51 (7)	C4—C5—N1	119.3 (3)
O5—Ce1—O4	147.09 (6)	C6—C5—N1	121.0 (3)
O3^{iii}—Ce1—O4	49.99 (6)	C1—C6—C5	120.0 (3)
O1—Ce1—O4	91.49 (6)	C1—C6—H6	120.0
O4^{i}—Ce1—O4	99.54 (6)	C5—C6—H6	120.0
O2^{i}—Ce1—O6	72.98 (6)	O2—C7—O1	123.6 (3)

O6^{ii}—Ce1—O6	104.06 (7)	O2—C7—C1	117.1 (2)
O1W—Ce1—O6	74.48 (7)	O1—C7—C1	119.3 (2)
O5—Ce1—O6	48.09 (6)	O3—C8—O4^{iii}	120.9 (3)
O3^{iii}—Ce1—O6	141.19 (6)	O3—C8—C3	118.9 (2)
O1—Ce1—O6	113.57 (6)	O4^{iii}—C8—C3	120.2 (2)
O4^{i}—Ce1—O6	66.99 (6)	O3—C8—Ce1^{iii}	56.79 (15)
O4—Ce1—O6	153.67 (5)	O4^{iii}—C8—Ce1^{iii}	64.13 (15)
O2^{i}—Ce1—C8^{iii}	99.07 (7)	C3—C8—Ce1^{iii}	175.47 (19)
O6^{ii}—Ce1—C8^{iii}	93.72 (7)	C12—C9—C10	119.9 (3)
O1W—Ce1—C8^{iii}	98.21 (7)	C12—C9—C13	117.3 (3)
O5—Ce1—C8^{iii}	152.07 (7)	C10—C9—C13	122.8 (3)
O3^{iii}—Ce1—C8^{iii}	24.68 (7)	C9—C10—C11	120.4 (3)
O1—Ce1—C8^{iii}	83.09 (7)	C9—C10—H10	119.8
O4^{i}—Ce1—C8^{iii}	90.61 (7)	C11—C10—H10	119.8
O4—Ce1—C8^{iii}	25.31 (6)	N2—C11—C10^{iv}	120.60 (18)
O6—Ce1—C8^{iii}	157.60 (7)	N2—C11—C10	120.60 (18)
C5—N1—H1C	119.9	C10^{iv}—C11—C10	118.8 (4)
C5—N1—H1D	116.1	C9^{iv}—C12—C9	120.6 (4)
H1C—N1—H1D	109.7	C9^{iv}—C12—H12	119.7
C11—N2—H2A	120.0	C9—C12—H12	119.7
C7—O1—Ce1	116.27 (17)	O5—C13—O6	120.0 (3)
C7—O2—Ce1^{ii}	155.62 (19)	O5—C13—C9	118.0 (2)
C8—O3—Ce1^{iii}	98.53 (17)	O6—C13—C9	121.9 (2)

Symmetry codes: (i) −*x*+3/2, *y*−1/2, *z*; (ii) −*x*+3/2, *y*+1/2, *z*; (iii) −*x*+1, −*y*+1, −*z*; (iv) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O5^{v}	0.86	2.63	3.436 (3)	157
N1—H1*D*···O1^{vi}	0.86	2.39	3.154 (3)	148
O1*W*—H1*A*···O3^{v}	0.85	2.07	2.898 (3)	165

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