

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

catena-Poly[[penta- μ -benzoato- μ -chlorido-dioxanedineodymium(III) dioxane 2.5-solvate]

Jeffrey A. Bertke, Allen G. Oliver and Kenneth W. Henderson*

251 Nieuwland Science Hall, Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, IN 46556-5670, USA

Correspondence e-mail: khenders@nd.edu

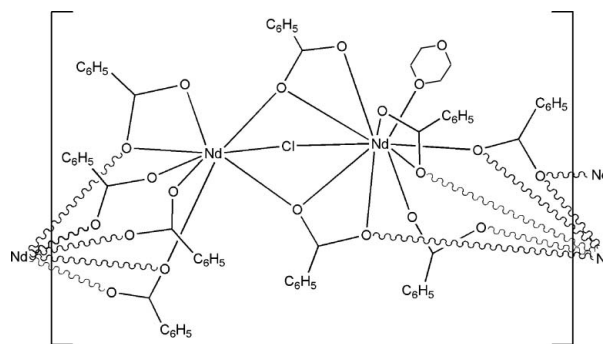
Received 4 April 2012; accepted 20 April 2012

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.015$ Å; some non-H atoms missing; disorder in main residue; R factor = 0.052; wR factor = 0.119; data-to-parameter ratio = 19.0.

The asymmetric unit of the title compound, $[\text{Nd}_2(\text{C}_6\text{H}_5\text{COO})_5\text{Cl}(\text{C}_4\text{H}_8\text{O}_2)] \cdot 2.5\text{C}_4\text{H}_8\text{O}_2$, consists of two Nd^{III} ions bridged by one Cl^- ion, five benzoate ions and one coordinating 1,4-dioxane molecule. One Nd^{III} ion is nine-coordinate, with a very distorted monocapped square-antiprismatic geometry. It is coordinated by two chelating carboxylate groups, three monodentate carboxylate groups, one chloride ion and one dioxane molecule. A second independent Nd^{III} ion is eight-coordinate in a distorted square-antiprismatic geometry by one chelating carboxylate group, five monodentate carboxylate groups and one chloride ion. The chains of the extended structure are parallel to the crystallographic b axis. There is a small amount of void space which is filled with five disordered dioxane solvent molecules per unit cell. The intensity contribution of the disordered solvent molecules was removed by applying the *SQUEEZE* procedure in *PLATON* [Spek (2009). *Acta Cryst.* **D65**, 148–155].

Related literature

For recent research on ditopic-linked secondary building units, see: Morris *et al.* (2008). For 2-D neodymium adducts, see: Nayak *et al.* (2010). For the synthesis of the neodymium precursor, see: Andersen *et al.* (1978). For *SQUEEZE* analysis of the data, see: Spek (2009).



Experimental

Crystal data

$[\text{Nd}_2(\text{C}_6\text{H}_5\text{O}_2)_5\text{Cl}(\text{C}_4\text{H}_8\text{O}_2)] \cdot 2.5\text{C}_4\text{H}_8\text{O}_2$

$M_r = 1237.84$

Triclinic, $P\bar{1}$

$a = 12.7631$ (5) Å

$b = 13.6077$ (6) Å

$c = 14.2614$ (7) Å

$\alpha = 102.785$ (3)°

$\beta = 96.943$ (3)°

$\gamma = 104.316$ (3)°

$V = 2299.68$ (18) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 2.37$ mm⁻¹

$T = 100$ K

$0.32 \times 0.18 \times 0.16$ mm

Data collection

Bruker APEXII CCD diffractometer

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008)

$T_{\text{min}} = 0.606$, $T_{\text{max}} = 0.685$

27066 measured reflections

8104 independent reflections

4870 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.087$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.119$

$S = 0.90$

8104 reflections

427 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 1.45$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.94$ e Å⁻³

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008; 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* and *pubCIF* (Westrip, 2010).

We gratefully acknowledge support of the Bayer Corp. and the Center for Sustainable Energy at Notre Dame (cSEND).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2541).

References

- Andersen, R. A., Templeton, D. H. & Zalkin, A. (1978). *Inorg. Chem.* **17**, 1962–1965.
- Bruker (2008). *APEX2* and *SAINT*. Bruker–Nonius AXS Inc., Madison, Wisconsin, USA.
- Morris, J. J., MacDougall, D. J., Noll, B. C. & Henderson, K. W. (2008). *Dalton Trans.* pp. 3429–3437.
- Nayak, S., Kostakis, G. E., Anson, C. E. & Powell, A. K. (2010). *CrystEngComm*, pp. 3008–3011.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2012). E68, m690 [doi:10.1107/S1600536812017746]

catena-Poly[[penta- μ -benzoato- μ -chlorido-dioxanedineodymium(III)] dioxane 2.5-solvate]

Jeffrey A. Bertke, Allen G. Oliver and Kenneth W. Henderson

S1. Comment

We are interested in building three-dimensional materials using pre-assembled molecular aggregates as secondary building units (SBUs) linked through ditopic organic molecules (Morris *et al.*, 2008). The title compound was synthesized with this goal in mind. However, instead of forming neodymium-benzoate SBUs which could be linked through 1,4-dioxane, a 1-D neodymium-benzoate polymer was crystallized. The closely related compound $[\text{Nd}_2(\text{C}_6\text{H}_5\text{COO})_6(\text{CH}_3\text{OH})_4]$ was previously reported as a 2-D material from PXRD data (Nayak *et al.*, 2010).

The asymmetric unit consists of two Nd^{3+} metal centers bridged by one Cl^- ion, five benzoate ions and one coordinated 1,4-dioxane molecule (Figure 1). The presence of chloride can be explained as carry-over from the synthesis of $\text{Nd}(\text{O}^i\text{Pr})_3$, which is synthesized from NdCl_3 and NaO^iPr (Andersen *et al.*, 1978). The extended crystal structure is a 1-D polymeric chain composed of $\text{Nd}/\text{Cl}/\text{carboxylate}$ interactions. The dioxane solvent molecule acts as a terminal donor. The 1-D chains extend along the crystallographic *b*-axis (Figure 2). Small channels also run along the crystallographic *b*-axis and are filled with disordered solvent molecules. The solvent could not be reliably modeled and was omitted through use of the SQUEEZE routine within *PLATON* (Spek, 2009; see refinement procedures for details).

Nd1 adopts a 9-coordinate distorted mono-capped square anti-prismatic geometry. It is coordinated by two chelating carboxylate groups, three monodentate carboxylate groups, one bridging chloride ion and one terminally coordinating dioxane molecule. Nd2 is 8-coordinate in a distorted square anti-prismatic geometry. It is coordinated by one chelating carboxylate group, five monodentate carboxylate groups and the bridging chloride ion.

S2. Experimental

Benzoic acid was purchased from Aldrich and used without further purification. $\text{Nd}(\text{O}^i\text{Pr})_3$ was purchased from Strem and used without further purification. 1,4-dioxane was distilled onto molecular sieves from potassium benzophenone.

Benzoic acid (0.366 g, 3 mmol) was dissolved in 1,4-dioxane (20 ml). Solid $\text{Nd}(\text{O}^i\text{Pr})_3$ (0.321 g, 1 mmol) was added and the solution was refluxed overnight. The solution was allowed to cool to room temperature and then filtered. The filtrate was concentrated and single crystals were grown upon cooling of this saturated solution to room temperature in a hot water bath over the course of several days. Crystalline yield 0.069 g (11.14%) calculated based on Nd.

S3. Refinement

Hydrogen atoms were included in idealized geometries riding on the atom to which they are bonded. Aromatic C–H distances were constrained to 0.95 Å and methylene C–H distances constrained to 0.99 Å. All hydrogen thermal parameters were set to $1.2 \times U_{\text{eq}}$ of the carbon to which they are bonded.

Disordered solvent could not be reliably modeled and was thus omitted from the model through the use of SQUEEZE (Spek, 2009). The void space analysis yielded a volume of 540 Å³ with an electron count of 224, located at a center of

symmetry. This corresponds well to five molecules of dioxane per unit cell. The atom count has been corrected to reflect this solvent inclusion.

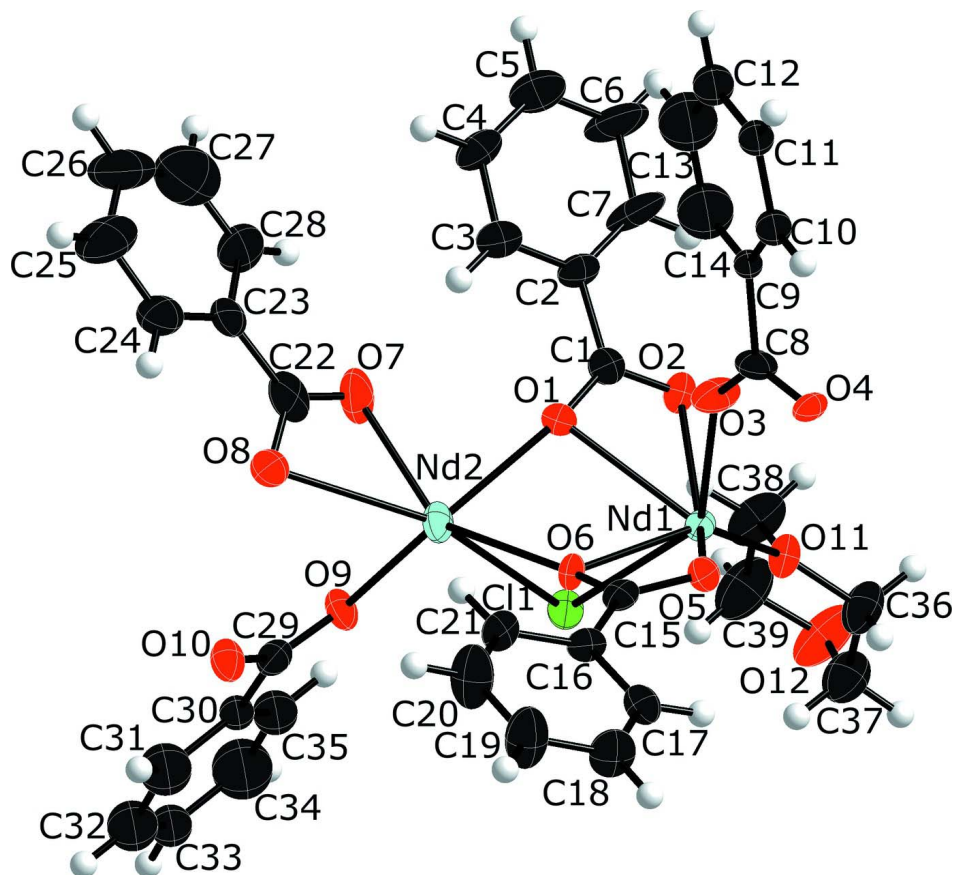
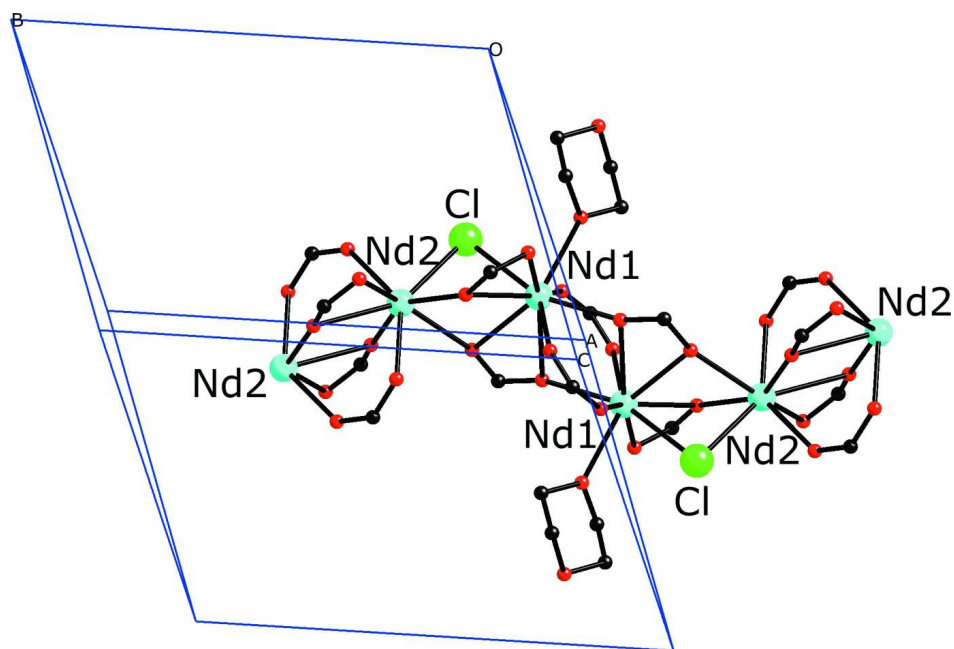


Figure 1

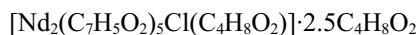
Labelling scheme for I. Thermal ellipsoids represented at 50% probability. H atoms depicted as spheres of a fixed radius.

**Figure 2**

Extended structure, with the one-dimensional chain extending along the *b*-axis. All phenyl rings of the benzoate ligands and all hydrogen atoms have been removed for clarity.

catena-Poly[[penta- μ -benzoato- μ -chlorido-dioxanedineodymium(III)] dioxane 2.5-solvate]

Crystal data



$M_r = 1237.84$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 12.7631\ (5)\ \text{\AA}$

$b = 13.6077\ (6)\ \text{\AA}$

$c = 14.2614\ (7)\ \text{\AA}$

$\alpha = 102.785\ (3)^\circ$

$\beta = 96.943\ (3)^\circ$

$\gamma = 104.316\ (3)^\circ$

$V = 2299.68\ (18)\ \text{\AA}^3$

$Z = 2$

$F(000) = 1240$

$D_x = 1.788\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2312 reflections

$\theta = 2.4\text{--}19.0^\circ$

$\mu = 2.37\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Needle, blue

$0.32 \times 0.18 \times 0.16\ \text{mm}$

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $8.33\ \text{pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2008)

$T_{\min} = 0.606$, $T_{\max} = 0.685$

27066 measured reflections

8104 independent reflections

4870 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.087$

$\theta_{\max} = 25.1^\circ$, $\theta_{\min} = 1.5^\circ$

$h = -15 \rightarrow 15$

$k = -16 \rightarrow 16$

$l = -13 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.119$
 $S = 0.90$
 8104 reflections
 427 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0572P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.45 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.94 \text{ e } \text{Å}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Nd1	0.42843 (3)	0.05580 (3)	0.39858 (3)	0.02441 (14)	
Nd2	0.46041 (4)	0.35430 (3)	0.41815 (4)	0.03265 (16)	
Cl1	0.27807 (15)	0.17311 (16)	0.36816 (16)	0.0329 (5)	
O1	0.5172 (4)	0.2130 (4)	0.3257 (4)	0.0310 (14)	
O2	0.4585 (4)	0.0526 (4)	0.2303 (4)	0.0341 (14)	
O3	0.6189 (4)	0.0707 (5)	0.3970 (5)	0.0456 (17)	
O4	0.7013 (4)	0.0001 (4)	0.5011 (4)	0.0316 (14)	
O5	0.4679 (4)	-0.1024 (4)	0.4236 (4)	0.0300 (13)	
O6	0.5077 (4)	-0.2327 (4)	0.4716 (4)	0.0291 (13)	
O7	0.5231 (7)	0.4308 (5)	0.2902 (5)	0.063 (2)	
O8	0.5524 (4)	0.5571 (4)	0.4251 (4)	0.0368 (15)	
O9	0.3103 (4)	0.4307 (4)	0.3939 (4)	0.0358 (15)	
O10	0.3508 (4)	0.5843 (4)	0.5070 (4)	0.0377 (15)	
O11	0.2669 (4)	-0.0860 (4)	0.2839 (4)	0.0374 (15)	
O12	0.0494 (6)	-0.1857 (6)	0.1846 (5)	0.074 (2)	
C1	0.5171 (7)	0.1464 (7)	0.2475 (6)	0.032 (2)	
C2	0.5931 (7)	0.1778 (7)	0.1806 (6)	0.034 (2)	
C3	0.6589 (7)	0.2799 (7)	0.1979 (7)	0.041 (2)	
H3	0.6534	0.3332	0.2512	0.049*	
C4	0.7321 (7)	0.3036 (8)	0.1375 (7)	0.044 (2)	
H4	0.7758	0.3739	0.1480	0.052*	
C5	0.7424 (9)	0.2266 (9)	0.0625 (8)	0.061 (3)	
H5	0.7949	0.2431	0.0224	0.074*	
C6	0.6765 (11)	0.1250 (10)	0.0450 (9)	0.087 (4)	
H6	0.6828	0.0715	-0.0077	0.104*	

C7	0.6023 (9)	0.1019 (8)	0.1038 (7)	0.062 (3)	
H7	0.5563	0.0321	0.0913	0.074*	
C8	0.7002 (7)	0.0470 (7)	0.4349 (7)	0.036 (2)	
C15	0.4761 (6)	-0.1963 (6)	0.4033 (6)	0.0265 (19)	
C16	0.4459 (6)	-0.2587 (7)	0.2995 (6)	0.029 (2)	
C17	0.4655 (7)	-0.2070 (7)	0.2282 (6)	0.036 (2)	
H17	0.4979	-0.1331	0.2458	0.043*	
C18	0.4375 (8)	-0.2634 (8)	0.1285 (7)	0.049 (3)	
H18	0.4518	-0.2286	0.0786	0.059*	
C19	0.3889 (10)	-0.3704 (8)	0.1055 (8)	0.066 (3)	
H19	0.3696	-0.4097	0.0389	0.079*	
C20	0.3682 (9)	-0.4208 (8)	0.1774 (7)	0.063 (3)	
H20	0.3328	-0.4941	0.1598	0.075*	
C21	0.3980 (6)	-0.3665 (6)	0.2748 (6)	0.033 (2)	
H21	0.3859	-0.4023	0.3243	0.039*	
C22	0.5731 (9)	0.5236 (9)	0.3398 (8)	0.052 (3)	
C23	0.6533 (9)	0.5968 (8)	0.3005 (8)	0.050 (3)	
C24	0.7152 (8)	0.6935 (8)	0.3578 (8)	0.053 (3)	
H24	0.7077	0.7161	0.4240	0.063*	
C25	0.7892 (9)	0.7580 (11)	0.3175 (11)	0.080 (4)	
H25	0.8355	0.8241	0.3564	0.096*	
C26	0.7940 (11)	0.7236 (13)	0.2186 (12)	0.091 (5)	
H26	0.8449	0.7665	0.1905	0.110*	
C27	0.7268 (14)	0.6292 (14)	0.1617 (11)	0.099 (5)	
H27	0.7296	0.6084	0.0941	0.119*	
C28	0.6564 (11)	0.5655 (10)	0.2008 (9)	0.076 (4)	
H28	0.6096	0.5000	0.1612	0.092*	
C29	0.2858 (7)	0.5090 (7)	0.4387 (7)	0.034 (2)	
C36	0.2039 (7)	-0.1803 (7)	0.3002 (8)	0.056 (3)	
H36A	0.2273	-0.1824	0.3683	0.067*	
H36B	0.2163	-0.2415	0.2555	0.067*	
C37	0.0851 (8)	-0.1856 (8)	0.2826 (8)	0.060 (3)	
H37A	0.0415	-0.2503	0.2961	0.072*	
H37B	0.0729	-0.1245	0.3277	0.072*	
C38	0.2271 (8)	-0.0800 (8)	0.1877 (7)	0.060 (3)	
H38A	0.2405	-0.1372	0.1386	0.072*	
H38B	0.2679	-0.0122	0.1778	0.072*	
C39	0.1106 (9)	-0.0887 (10)	0.1734 (9)	0.078 (4)	
H39A	0.0973	-0.0306	0.2216	0.093*	
H39B	0.0858	-0.0825	0.1071	0.093*	
C9	0.8010 (9)	0.0640 (11)	0.3862 (10)	0.021 (4)*	0.50
C10	0.9017 (11)	0.0620 (12)	0.4340 (9)	0.032 (9)*	0.50
H10	0.9062	0.0414	0.4935	0.038*	0.50
C11	0.9959 (8)	0.0900 (11)	0.3948 (10)	0.031 (6)*	0.50
H11	1.0647	0.0886	0.4275	0.037*	0.50
C12	0.9893 (9)	0.1201 (10)	0.3077 (10)	0.041 (5)*	0.50
H12	1.0537	0.1392	0.2810	0.049*	0.50
C13	0.8886 (11)	0.1221 (12)	0.2599 (8)	0.084 (8)*	0.50

H13	0.8842	0.1426	0.2004	0.101*	0.50
C14	0.7945 (9)	0.0941 (12)	0.2991 (10)	0.076 (7)*	0.50
H14	0.7257	0.0955	0.2664	0.092*	0.50
C9A	0.8092 (8)	0.0957 (9)	0.4084 (8)	0.019 (4)*	0.50
C10A	0.9047 (11)	0.0748 (11)	0.4459 (9)	0.040 (10)*	0.50
H10A	0.9030	0.0344	0.4925	0.048*	0.50
C11A	1.0026 (8)	0.1131 (11)	0.4154 (10)	0.032 (6)*	0.50
H11A	1.0678	0.0988	0.4411	0.039*	0.50
C12A	1.0050 (7)	0.1723 (9)	0.3474 (9)	0.037 (4)*	0.50
H12A	1.0720	0.1984	0.3265	0.044*	0.50
C13A	0.9096 (8)	0.1931 (8)	0.3098 (8)	0.042 (5)*	0.50
H13A	0.9113	0.2336	0.2633	0.051*	0.50
C14A	0.8117 (7)	0.1548 (8)	0.3403 (8)	0.022 (3)*	0.50
H14A	0.7465	0.1691	0.3147	0.026*	0.50
C30	0.1778 (10)	0.5269 (13)	0.4109 (13)	0.029 (7)*	0.50
C31	0.1526 (13)	0.6120 (13)	0.4677 (12)	0.066 (10)*	0.50
H31	0.2060	0.6599	0.5210	0.079*	0.50
C32	0.0493 (14)	0.6268 (12)	0.4467 (12)	0.062 (8)*	0.50
H32	0.0321	0.6850	0.4856	0.074*	0.50
C33	-0.0288 (10)	0.5566 (13)	0.3688 (11)	0.043 (5)*	0.50
H33	-0.0994	0.5668	0.3544	0.052*	0.50
C34	-0.0036 (11)	0.4716 (11)	0.3119 (10)	0.089 (8)*	0.50
H34	-0.0570	0.4236	0.2587	0.106*	0.50
C35	0.0997 (12)	0.4567 (10)	0.3329 (10)	0.048 (5)*	0.50
H35	0.1169	0.3986	0.2941	0.058*	0.50
C30A	0.1673 (10)	0.5126 (12)	0.4180 (12)	0.031 (7)*	0.50
C31A	0.1370 (11)	0.6039 (10)	0.4499 (13)	0.032 (6)*	0.50
H31A	0.1903	0.6658	0.4894	0.039*	0.50
C32A	0.0287 (12)	0.6048 (11)	0.4242 (13)	0.035 (5)*	0.50
H32A	0.0080	0.6673	0.4460	0.043*	0.50
C33A	-0.0493 (9)	0.5143 (14)	0.3665 (12)	0.079 (9)*	0.50
H33A	-0.1233	0.5149	0.3489	0.095*	0.50
C34A	-0.0190 (12)	0.4229 (11)	0.3346 (11)	0.098 (9)*	0.50
H34A	-0.0723	0.3611	0.2951	0.117*	0.50
C35A	0.0892 (13)	0.4221 (10)	0.3603 (11)	0.055 (6)*	0.50
H35A	0.1099	0.3596	0.3385	0.066*	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Nd1	0.0195 (2)	0.0248 (3)	0.0326 (3)	0.00797 (19)	0.0064 (2)	0.0122 (2)
Nd2	0.0263 (3)	0.0245 (3)	0.0492 (4)	0.0104 (2)	0.0034 (2)	0.0121 (2)
Cl1	0.0200 (10)	0.0315 (11)	0.0472 (14)	0.0080 (8)	0.0016 (9)	0.0118 (10)
O1	0.032 (3)	0.030 (3)	0.033 (4)	0.010 (3)	0.011 (3)	0.008 (3)
O2	0.034 (3)	0.027 (3)	0.039 (4)	0.003 (3)	0.004 (3)	0.011 (3)
O3	0.020 (3)	0.069 (4)	0.064 (4)	0.019 (3)	0.015 (3)	0.040 (4)
O4	0.025 (3)	0.042 (3)	0.044 (4)	0.015 (3)	0.017 (3)	0.031 (3)
O5	0.031 (3)	0.027 (3)	0.036 (4)	0.012 (2)	0.008 (3)	0.011 (3)

O6	0.030 (3)	0.028 (3)	0.037 (4)	0.012 (2)	0.007 (3)	0.018 (3)
O7	0.118 (7)	0.041 (4)	0.048 (5)	0.040 (4)	0.026 (4)	0.021 (4)
O8	0.033 (3)	0.040 (4)	0.044 (4)	0.016 (3)	0.009 (3)	0.015 (3)
O9	0.028 (3)	0.027 (3)	0.051 (4)	0.013 (3)	0.004 (3)	0.005 (3)
O10	0.027 (3)	0.029 (3)	0.055 (4)	0.009 (3)	0.002 (3)	0.009 (3)
O11	0.038 (3)	0.028 (3)	0.044 (4)	0.003 (3)	0.004 (3)	0.014 (3)
O12	0.048 (4)	0.090 (6)	0.059 (5)	-0.025 (4)	-0.019 (4)	0.034 (4)
C1	0.027 (5)	0.038 (5)	0.036 (6)	0.017 (4)	0.003 (4)	0.017 (5)
C2	0.032 (5)	0.048 (6)	0.028 (5)	0.013 (4)	0.008 (4)	0.022 (5)
C3	0.034 (5)	0.052 (6)	0.039 (6)	0.009 (4)	0.008 (4)	0.017 (5)
C4	0.031 (5)	0.048 (6)	0.049 (7)	-0.002 (4)	0.004 (5)	0.024 (5)
C5	0.067 (8)	0.079 (8)	0.043 (7)	0.016 (6)	0.021 (6)	0.027 (6)
C6	0.112 (11)	0.074 (9)	0.062 (8)	-0.009 (8)	0.059 (8)	0.010 (7)
C7	0.072 (8)	0.057 (7)	0.042 (7)	-0.017 (6)	0.027 (6)	0.012 (6)
C8	0.023 (5)	0.044 (5)	0.044 (6)	0.012 (4)	0.014 (4)	0.010 (5)
C15	0.012 (4)	0.032 (5)	0.034 (5)	0.002 (3)	0.002 (4)	0.011 (4)
C16	0.023 (4)	0.043 (5)	0.027 (5)	0.009 (4)	0.001 (4)	0.022 (4)
C17	0.034 (5)	0.035 (5)	0.039 (6)	0.015 (4)	0.005 (4)	0.007 (5)
C18	0.064 (7)	0.055 (7)	0.029 (6)	0.016 (5)	0.003 (5)	0.016 (5)
C19	0.101 (9)	0.046 (7)	0.045 (7)	0.016 (6)	0.000 (6)	0.012 (6)
C20	0.090 (9)	0.042 (6)	0.043 (7)	0.014 (6)	-0.013 (6)	0.003 (6)
C21	0.034 (5)	0.038 (5)	0.036 (6)	0.016 (4)	0.001 (4)	0.024 (4)
C22	0.076 (8)	0.059 (7)	0.044 (7)	0.050 (6)	0.010 (6)	0.029 (6)
C23	0.068 (7)	0.047 (6)	0.062 (8)	0.041 (6)	0.036 (6)	0.031 (6)
C24	0.046 (6)	0.065 (7)	0.063 (7)	0.027 (6)	0.023 (6)	0.029 (6)
C25	0.039 (7)	0.100 (10)	0.115 (12)	0.012 (6)	0.025 (7)	0.058 (9)
C26	0.071 (9)	0.129 (14)	0.108 (13)	0.031 (9)	0.054 (9)	0.076 (11)
C27	0.119 (14)	0.124 (14)	0.081 (11)	0.062 (12)	0.031 (10)	0.042 (11)
C28	0.127 (12)	0.064 (8)	0.063 (9)	0.040 (8)	0.053 (8)	0.036 (7)
C29	0.027 (5)	0.036 (5)	0.049 (6)	0.013 (4)	0.010 (4)	0.023 (5)
C36	0.043 (6)	0.041 (6)	0.081 (8)	0.002 (5)	0.002 (5)	0.026 (6)
C37	0.050 (7)	0.054 (7)	0.073 (8)	0.004 (5)	0.005 (6)	0.024 (6)
C38	0.058 (7)	0.069 (7)	0.045 (7)	-0.002 (6)	-0.010 (5)	0.031 (6)
C39	0.056 (7)	0.083 (9)	0.085 (9)	-0.003 (6)	-0.008 (6)	0.040 (7)

Geometric parameters (Å, °)

Nd1—O3	2.392 (5)	C22—C23	1.495 (14)
Nd1—O4 ⁱ	2.409 (5)	C23—C24	1.370 (13)
Nd1—O5	2.418 (5)	C23—C28	1.399 (14)
Nd1—O2	2.469 (5)	C24—C25	1.392 (14)
Nd1—O11	2.543 (5)	C24—H24	0.9500
Nd1—O6 ⁱ	2.565 (5)	C25—C26	1.398 (17)
Nd1—O5 ⁱ	2.578 (5)	C25—H25	0.9500
Nd1—O1	2.647 (5)	C26—C27	1.368 (18)
Nd1—C11	2.835 (2)	C26—H26	0.9500
Nd1—C1	2.918 (9)	C27—C28	1.349 (17)
Nd1—C15 ⁱ	2.958 (8)	C27—H27	0.9500

Nd1—Nd2	3.9235 (6)	C28—H28	0.9500
Nd2—O8 ⁱⁱ	2.339 (6)	C29—C30	1.482 (13)
Nd2—O10 ⁱⁱⁱ	2.373 (5)	C29—C30A	1.521 (13)
Nd2—O1	2.400 (5)	C36—C37	1.488 (13)
Nd2—O7	2.415 (7)	C36—H36A	0.9900
Nd2—O9	2.420 (5)	C36—H36B	0.9900
Nd2—O6 ⁱ	2.589 (5)	C37—H37A	0.9900
Nd2—O8	2.693 (6)	C37—H37B	0.9900
Nd2—C11	2.826 (2)	C38—C39	1.447 (14)
Nd2—C22	2.918 (11)	C38—H38A	0.9900
Nd2—Nd2 ⁱⁱ	3.9428 (9)	C38—H38B	0.9900
O1—C1	1.272 (9)	C39—H39A	0.9900
O2—C1	1.262 (9)	C39—H39B	0.9900
O3—C8	1.258 (10)	C9—C10	1.3900
O4—C8	1.251 (10)	C9—C14	1.3900
O4—Nd1 ⁱ	2.409 (5)	C10—C11	1.3900
O5—C15	1.280 (9)	C10—H10	0.9500
O5—Nd1 ⁱ	2.578 (5)	C11—C12	1.3900
O6—C15	1.254 (9)	C11—H11	0.9500
O6—Nd1 ⁱ	2.565 (5)	C12—C13	1.3900
O6—Nd2 ⁱ	2.589 (5)	C12—H12	0.9500
O7—C22	1.265 (12)	C13—C14	1.3900
O8—C22	1.284 (11)	C13—H13	0.9500
O8—Nd2 ⁱⁱ	2.339 (6)	C14—H14	0.9500
O9—C29	1.246 (9)	C9A—C10A	1.3900
O10—C29	1.276 (10)	C9A—C14A	1.3900
O10—Nd2 ⁱⁱ	2.373 (5)	C10A—C11A	1.3900
O11—C36	1.419 (10)	C10A—H10A	0.9500
O11—C38	1.431 (10)	C11A—C12A	1.3900
O12—C39	1.411 (12)	C11A—H11A	0.9500
O12—C37	1.417 (11)	C12A—C13A	1.3900
C1—C2	1.495 (11)	C12A—H12A	0.9500
C2—C7	1.368 (12)	C13A—C14A	1.3900
C2—C3	1.384 (12)	C13A—H13A	0.9500
C3—C4	1.374 (12)	C14A—H14A	0.9500
C3—H3	0.9500	C30—C31	1.3900
C4—C5	1.365 (13)	C30—C35	1.3900
C4—H4	0.9500	C31—C32	1.3900
C5—C6	1.379 (15)	C31—H31	0.9500
C5—H5	0.9500	C32—C33	1.3900
C6—C7	1.366 (13)	C32—H32	0.9500
C6—H6	0.9500	C33—C34	1.3900
C7—H7	0.9500	C33—H33	0.9500
C8—C9A	1.517 (12)	C34—C35	1.3900
C8—C9	1.528 (12)	C34—H34	0.9500
C15—C16	1.486 (11)	C35—H35	0.9500
C15—Nd1 ⁱ	2.958 (8)	C30A—C31A	1.3900
C16—C17	1.373 (11)	C30A—C35A	1.3900

C16—C21	1.387 (11)	C31A—C32A	1.3900
C17—C18	1.413 (12)	C31A—H31A	0.9500
C17—H17	0.9500	C32A—C33A	1.3900
C18—C19	1.380 (13)	C32A—H32A	0.9500
C18—H18	0.9500	C33A—C34A	1.3900
C19—C20	1.370 (14)	C33A—H33A	0.9500
C19—H19	0.9500	C34A—C35A	1.3900
C20—C21	1.378 (12)	C34A—H34A	0.9500
C20—H20	0.9500	C35A—H35A	0.9500
C21—H21	0.9500		
O3—Nd1—O4 ⁱ	136.30 (19)	C7—C6—C5	119.5 (11)
O3—Nd1—O5	70.64 (19)	C7—C6—H6	120.2
O4 ⁱ —Nd1—O5	77.57 (18)	C5—C6—H6	120.2
O3—Nd1—O2	72.27 (19)	C6—C7—C2	120.9 (10)
O4 ⁱ —Nd1—O2	145.82 (18)	C6—C7—H7	119.5
O5—Nd1—O2	103.26 (18)	C2—C7—H7	119.5
O3—Nd1—O11	125.9 (2)	O4—C8—O3	125.8 (8)
O4 ⁱ —Nd1—O11	73.60 (18)	O4—C8—C9A	118.0 (8)
O5—Nd1—O11	78.40 (17)	O3—C8—C9A	115.4 (9)
O2—Nd1—O11	73.18 (18)	O4—C8—C9	117.3 (8)
O3—Nd1—O6 ⁱ	86.92 (19)	O3—C8—C9	116.4 (9)
O4 ⁱ —Nd1—O6 ⁱ	86.44 (18)	C9A—C8—C9	17.2 (7)
O5—Nd1—O6 ⁱ	123.61 (17)	O6—C15—O5	119.0 (7)
O2—Nd1—O6 ⁱ	118.35 (17)	O6—C15—C16	122.6 (7)
O11—Nd1—O6 ⁱ	146.72 (17)	O5—C15—C16	118.4 (7)
O3—Nd1—O5 ⁱ	71.19 (19)	O6—C15—Nd1 ⁱ	59.7 (4)
O4 ⁱ —Nd1—O5 ⁱ	71.51 (17)	O5—C15—Nd1 ⁱ	60.4 (4)
O5—Nd1—O5 ⁱ	73.42 (19)	C16—C15—Nd1 ⁱ	170.4 (5)
O2—Nd1—O5 ⁱ	142.16 (17)	C17—C16—C21	120.7 (8)
O11—Nd1—O5 ⁱ	138.86 (17)	C17—C16—C15	118.4 (8)
O6 ⁱ —Nd1—O5 ⁱ	50.26 (16)	C21—C16—C15	121.0 (7)
O3—Nd1—O1	69.33 (18)	C16—C17—C18	120.1 (8)
O4 ⁱ —Nd1—O1	144.00 (17)	C16—C17—H17	120.0
O5—Nd1—O1	137.46 (17)	C18—C17—H17	120.0
O2—Nd1—O1	50.61 (17)	C19—C18—C17	118.3 (9)
O11—Nd1—O1	114.92 (18)	C19—C18—H18	120.8
O6 ⁱ —Nd1—O1	67.75 (17)	C17—C18—H18	120.8
O5 ⁱ —Nd1—O1	106.11 (17)	C20—C19—C18	120.9 (10)
O3—Nd1—C11	138.05 (15)	C20—C19—H19	119.5
O4 ⁱ —Nd1—C11	79.01 (13)	C18—C19—H19	119.5
O5—Nd1—C11	151.08 (13)	C19—C20—C21	120.9 (10)
O2—Nd1—C11	86.96 (13)	C19—C20—H20	119.5
O11—Nd1—C11	78.90 (13)	C21—C20—H20	119.5
O6 ⁱ —Nd1—C11	71.20 (12)	C20—C21—C16	119.0 (8)
O5 ⁱ —Nd1—C11	114.46 (12)	C20—C21—H21	120.5
O1—Nd1—C11	69.34 (12)	C16—C21—H21	120.5
O3—Nd1—C1	64.6 (2)	O7—C22—O8	119.3 (10)

O4 ⁱ —Nd1—C1	158.9 (2)	O7—C22—C23	121.7 (10)
O5—Nd1—C1	119.2 (2)	O8—C22—C23	118.9 (10)
O2—Nd1—C1	25.4 (2)	O7—C22—Nd2	54.6 (5)
O11—Nd1—C1	96.2 (2)	O8—C22—Nd2	67.1 (5)
O6 ⁱ —Nd1—C1	93.2 (2)	C23—C22—Nd2	165.5 (6)
O5 ⁱ —Nd1—C1	123.6 (2)	C24—C23—C28	121.4 (10)
O1—Nd1—C1	25.83 (19)	C24—C23—C22	121.4 (10)
Cl1—Nd1—C1	80.90 (16)	C28—C23—C22	117.0 (11)
O3—Nd1—C15 ⁱ	80.8 (2)	C23—C24—C25	118.9 (11)
O4 ⁱ —Nd1—C15 ⁱ	75.3 (2)	C23—C24—H24	120.5
O5—Nd1—C15 ⁱ	99.0 (2)	C25—C24—H24	120.5
O2—Nd1—C15 ⁱ	136.5 (2)	C24—C25—C26	118.6 (13)
O11—Nd1—C15 ⁱ	148.63 (19)	C24—C25—H25	120.7
O6 ⁱ —Nd1—C15 ⁱ	24.98 (18)	C26—C25—H25	120.7
O5 ⁱ —Nd1—C15 ⁱ	25.58 (19)	C27—C26—C25	121.2 (13)
O1—Nd1—C15 ⁱ	88.4 (2)	C27—C26—H26	119.4
Cl1—Nd1—C15 ⁱ	91.23 (16)	C25—C26—H26	119.4
C1—Nd1—C15 ⁱ	111.7 (2)	C28—C27—C26	120.4 (14)
O3—Nd1—Nd2	94.43 (15)	C28—C27—H27	119.8
O4 ⁱ —Nd1—Nd2	107.76 (13)	C26—C27—H27	119.8
O5—Nd1—Nd2	160.61 (12)	C27—C28—C23	119.3 (13)
O2—Nd1—Nd2	82.92 (12)	C27—C28—H28	120.4
O11—Nd1—Nd2	120.94 (13)	C23—C28—H28	120.4
O6 ⁱ —Nd1—Nd2	40.66 (12)	O9—C29—O10	125.1 (7)
O5 ⁱ —Nd1—Nd2	90.33 (12)	O9—C29—C30	121.7 (10)
O1—Nd1—Nd2	36.76 (12)	O10—C29—C30	113.1 (9)
Cl1—Nd1—Nd2	46.03 (4)	O9—C29—C30A	118.6 (9)
C1—Nd1—Nd2	61.10 (17)	O10—C29—C30A	116.1 (9)
C15 ⁱ —Nd1—Nd2	65.57 (16)	C30—C29—C30A	9.7 (12)
O8 ⁱⁱ —Nd2—O10 ⁱⁱ	79.77 (19)	O11—C36—C37	109.7 (8)
O8 ⁱⁱ —Nd2—O1	144.98 (19)	O11—C36—H36A	109.7
O10 ⁱⁱ —Nd2—O1	82.75 (18)	C37—C36—H36A	109.7
O8 ⁱⁱ —Nd2—O7	127.4 (2)	O11—C36—H36B	109.7
O10 ⁱⁱ —Nd2—O7	84.2 (2)	C37—C36—H36B	109.7
O1—Nd2—O7	80.1 (2)	H36A—C36—H36B	108.2
O8 ⁱⁱ —Nd2—O9	74.66 (19)	O12—C37—C36	110.5 (9)
O10 ⁱⁱ —Nd2—O9	136.64 (18)	O12—C37—H37A	109.6
O1—Nd2—O9	135.61 (19)	C36—C37—H37A	109.6
O7—Nd2—O9	84.3 (2)	O12—C37—H37B	109.6
O8 ⁱⁱ —Nd2—O6 ⁱ	74.84 (18)	C36—C37—H37B	109.6
O10 ⁱⁱ —Nd2—O6 ⁱ	73.25 (17)	H37A—C37—H37B	108.1
O1—Nd2—O6 ⁱ	71.14 (17)	O11—C38—C39	111.2 (9)
O7—Nd2—O6 ⁱ	145.2 (2)	O11—C38—H38A	109.4
O9—Nd2—O6 ⁱ	130.15 (18)	C39—C38—H38A	109.4
O8 ⁱⁱ —Nd2—O8	77.1 (2)	O11—C38—H38B	109.4
O10 ⁱⁱ —Nd2—O8	66.96 (17)	C39—C38—H38B	109.4
O1—Nd2—O8	122.75 (18)	H38A—C38—H38B	108.0
O7—Nd2—O8	50.7 (2)	O12—C39—C38	111.0 (10)

O9—Nd2—O8	73.49 (17)	O12—C39—H39A	109.4
O6 ⁱ —Nd2—O8	134.43 (17)	C38—C39—H39A	109.4
O8 ⁱⁱ —Nd2—C11	103.71 (14)	O12—C39—H39B	109.4
O10 ⁱⁱ —Nd2—C11	141.60 (14)	C38—C39—H39B	109.4
O1—Nd2—C11	72.87 (13)	H39A—C39—H39B	108.0
O7—Nd2—C11	119.06 (19)	C10—C9—C14	120.0
O9—Nd2—C11	79.17 (13)	C10—C9—C8	120.3 (9)
O6 ⁱ —Nd2—C11	71.02 (12)	C14—C9—C8	119.3 (9)
O8—Nd2—C11	151.44 (12)	C11—C10—C9	120.0
O8 ⁱⁱ —Nd2—C22	103.1 (3)	C11—C10—H10	120.0
O10 ⁱⁱ —Nd2—C22	70.4 (2)	C9—C10—H10	120.0
O1—Nd2—C22	99.2 (2)	C10—C11—C12	120.0
O7—Nd2—C22	25.3 (2)	C10—C11—H11	120.0
O9—Nd2—C22	82.0 (2)	C12—C11—H11	120.0
O6 ⁱ —Nd2—C22	143.3 (2)	C13—C12—C11	120.0
O8—Nd2—C22	26.1 (2)	C13—C12—H12	120.0
C11—Nd2—C22	141.6 (2)	C11—C12—H12	120.0
O8 ⁱⁱ —Nd2—Nd1	111.13 (14)	C14—C13—C12	120.0
O10 ⁱⁱ —Nd2—Nd1	96.31 (13)	C14—C13—H13	120.0
O1—Nd2—Nd1	41.32 (13)	C12—C13—H13	120.0
O7—Nd2—Nd1	120.26 (16)	C13—C14—C9	120.0
O9—Nd2—Nd1	125.35 (13)	C13—C14—H14	120.0
O6 ⁱ —Nd2—Nd1	40.19 (12)	C9—C14—H14	120.0
O8—Nd2—Nd1	160.40 (12)	C10A—C9A—C14A	120.0
C11—Nd2—Nd1	46.23 (4)	C10A—C9A—C8	120.4 (8)
C22—Nd2—Nd1	140.4 (2)	C14A—C9A—C8	119.4 (8)
O8 ⁱⁱ —Nd2—Nd2 ⁱⁱ	41.74 (14)	C11A—C10A—C9A	120.0
O10 ⁱⁱ —Nd2—Nd2 ⁱⁱ	68.11 (13)	C11A—C10A—H10A	120.0
O1—Nd2—Nd2 ⁱⁱ	148.79 (13)	C9A—C10A—H10A	120.0
O7—Nd2—Nd2 ⁱⁱ	85.83 (16)	C10A—C11A—C12A	120.0
O9—Nd2—Nd2 ⁱⁱ	69.45 (13)	C10A—C11A—H11A	120.0
O6 ⁱ —Nd2—Nd2 ⁱⁱ	108.84 (12)	C12A—C11A—H11A	120.0
O8—Nd2—Nd2 ⁱⁱ	35.32 (13)	C13A—C12A—C11A	120.0
C11—Nd2—Nd2 ⁱⁱ	137.78 (5)	C13A—C12A—H12A	120.0
C22—Nd2—Nd2 ⁱⁱ	61.4 (2)	C11A—C12A—H12A	120.0
Nd1—Nd2—Nd2 ⁱⁱ	149.02 (2)	C12A—C13A—C14A	120.0
Nd2—C11—Nd1	87.74 (5)	C12A—C13A—H13A	120.0
C1—O1—Nd2	153.5 (5)	C14A—C13A—H13A	120.0
C1—O1—Nd1	89.1 (5)	C13A—C14A—C9A	120.0
Nd2—O1—Nd1	101.92 (19)	C13A—C14A—H14A	120.0
C1—O2—Nd1	97.6 (5)	C9A—C14A—H14A	120.0
C8—O3—Nd1	143.1 (6)	C31—C30—C35	120.0
C8—O4—Nd1 ⁱ	134.2 (5)	C31—C30—C29	118.9 (11)
C15—O5—Nd1	159.1 (5)	C35—C30—C29	121.0 (11)
C15—O5—Nd1 ⁱ	94.0 (5)	C32—C31—C30	120.0
Nd1—O5—Nd1 ⁱ	106.58 (19)	C32—C31—H31	120.0
C15—O6—Nd1 ⁱ	95.3 (5)	C30—C31—H31	120.0
C15—O6—Nd2 ⁱ	164.7 (5)	C31—C32—C33	120.0

Nd1 ⁱ —O6—Nd2 ⁱ	99.15 (18)	C31—C32—H32	120.0
C22—O7—Nd2	100.2 (6)	C33—C32—H32	120.0
C22—O8—Nd2 ⁱⁱ	169.6 (7)	C32—C33—C34	120.0
C22—O8—Nd2	86.8 (6)	C32—C33—H33	120.0
Nd2 ⁱⁱ —O8—Nd2	102.9 (2)	C34—C33—H33	120.0
C29—O9—Nd2	135.2 (5)	C33—C34—C35	120.0
C29—O10—Nd2 ⁱⁱ	139.3 (5)	C33—C34—H34	120.0
C36—O11—C38	109.3 (7)	C35—C34—H34	120.0
C36—O11—Nd1	128.6 (5)	C34—C35—C30	120.0
C38—O11—Nd1	122.1 (5)	C34—C35—H35	120.0
C39—O12—C37	105.8 (8)	C30—C35—H35	120.0
O2—C1—O1	119.9 (8)	C31A—C30A—C35A	120.0
O2—C1—C2	120.4 (8)	C31A—C30A—C29	122.2 (10)
O1—C1—C2	119.5 (8)	C35A—C30A—C29	117.7 (10)
O2—C1—Nd1	57.0 (4)	C32A—C31A—C30A	120.0
O1—C1—Nd1	65.1 (4)	C32A—C31A—H31A	120.0
C2—C1—Nd1	160.5 (5)	C30A—C31A—H31A	120.0
C7—C2—C3	119.5 (8)	C31A—C32A—C33A	120.0
C7—C2—C1	118.6 (8)	C31A—C32A—H32A	120.0
C3—C2—C1	121.8 (8)	C33A—C32A—H32A	120.0
C4—C3—C2	119.6 (9)	C34A—C33A—C32A	120.0
C4—C3—H3	120.2	C34A—C33A—H33A	120.0
C2—C3—H3	120.2	C32A—C33A—H33A	120.0
C5—C4—C3	120.4 (9)	C33A—C34A—C35A	120.0
C5—C4—H4	119.8	C33A—C34A—H34A	120.0
C3—C4—H4	119.8	C35A—C34A—H34A	120.0
C4—C5—C6	120.0 (10)	C34A—C35A—C30A	120.0
C4—C5—H5	120.0	C34A—C35A—H35A	120.0
C6—C5—H5	120.0	C30A—C35A—H35A	120.0

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