Supporting Information

High-Temperature Biferroicity in a Chiral 3D Hybrid Rare-Earth-Based Double Perovskite

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Supplementary Index

Experimental section

General Measurements.

Powder X-ray Diffraction (PXRD) patterns were collected on a Rigaku D/MAX 2000 PC diffractometer, utilizing Cu Kα radiation (λ = 0.15406 nm) with a scanning range of 5°–50° and a rate of 5° min^{-1}. Thermogravimetric (TG) analysis was conducted under a nitrogen atmosphere using a NETZSCH STA2500 instrument at a heating rate of 10 K min^{-1}; approximately 10 mg of powdered compound 1 was loaded into an aluminum crucible for this purpose. Differential scanning calorimetry (DSC) experiments were performed in a nitrogen environment with a DSC 214 Polyma apparatus, maintaining a heating/cooling rate of 10 K min^{-1}. Dielectric measurements on compound 1 were executed using a Tonghui TH2828A impedance analyzer. The polycrystalline samples were pressed into pellets and sputtered with silver electrodes. The temperature-dependent complex dielectric permittivity ($\varepsilon r = \varepsilon' - i\varepsilon''$) was recorded across the frequency range of 500 Hz to 1000 kHz at temperatures between 260 K and 385 K. Ferroelastic domains were observed under an Olympus BX51TRF optical polarizing microscope, with temperature control maintained to within 0.2 K via an INSTEC HCC602 stage. Second harmonic generation (SHG) signals were measured using a pulsed Nd:YAG laser source (1064 nm, 5 ns pulses, 1.6 MW peak power, 10 Hz repetition rate). Variable-temperature ferroelectric P–E hysteresis loops were obtained on thin crystals (0.65 mm × 0.10 mm) using a Radiant Precision Premier II system. Ferroelectric switching behavior was characterized via resonance-enhanced piezoresponse force microscopy (PFM) using an MFP-3D system (Asylum Research) with conductive Pt/Ir coated silicon probes (EFM-50, Nanoworld). Ultraviolet-visible (UV-Vis) absorption spectra were acquired on a Shimadzu UV-3600i Plus spectrometer (Japan) from 200 to 800 nm. Both steady-state emission spectra and time-resolved fluorescence lifetimes were determined using a FluoroLog 3-TCSPC spectrofluorometer (Horiba Jobin Yvon Inc).

X-ray diffraction experiments.

Variable-temperature X-ray diffraction experiments were performed utilizing a Rigaku Synergy diffractometer equipped with Mo-*Kα* radiation (λ = 0.71073 Å). The

processes of data collection, cell refinement, and data reduction were accomplished with the CrysAlisPro (v1.171.41.112a) XtaLAB Synergy-R online platform. Crystal structures were determined via direct methods and refined via full-matrix least-squares on F^2 with the OLEX2 software suite. Anisotropic refinement was applied to all non-hydrogen atoms, whereas hydrogen atom positions were set based on geometric calculations. Complete crystallographic details can be found in **Tables S1,S3**. Deposition of the crystal structure files (CIF) has been made with the Cambridge Crystal Data Center (**CCDC: 2445289–2445291**). Access to this material is provided free of charge by the Cambridge Crystallographic Data Centre.

Note

Note S1.

$$\Delta S_1 = \int_{T_1}^{T_2} \frac{Q}{T} dT \approx \frac{\Delta H}{T_c} = \frac{6.757\ J \cdot g^{-1} \times 837.82\ g \cdot mol^{-1}}{370\ K} = \frac{11322}{37}$$

$$15.3\ J \cdot mol^{-1} \cdot K^{-1}$$

$$\Delta S_1 = R \ln N$$

$$N_1 = \exp\left(\frac{\Delta S}{R}\right) = \exp\left(\frac{15.3\ J \cdot mol^{-1} \cdot K^{-1}}{8.314\ J \cdot mol^{-1} \cdot K^{-1}}\right) = 6.300$$

$$\Delta S_2 = \int_{T_1}^{T_2} \frac{Q}{T} dT \approx \frac{\Delta H}{T_c} = \frac{2.264\ J \cdot g^{-1} \times 837.82\ g \cdot mol^{-1}}{410.00\ K} = \frac{3793.6}{41}$$

$$4.626\ J \cdot mol^{-1} \cdot K^{-1}$$

$$\Delta S_1 = R \ln N$$

$$N_2 = \exp\left(\frac{\Delta S}{R}\right) = \exp\left(\frac{4.626\ J \cdot mol^{-1} \cdot K^{-1}}{8.314\ J \cdot mol^{-1} \cdot K^{-1}}\right) = 1.744$$

Supplemental Figures

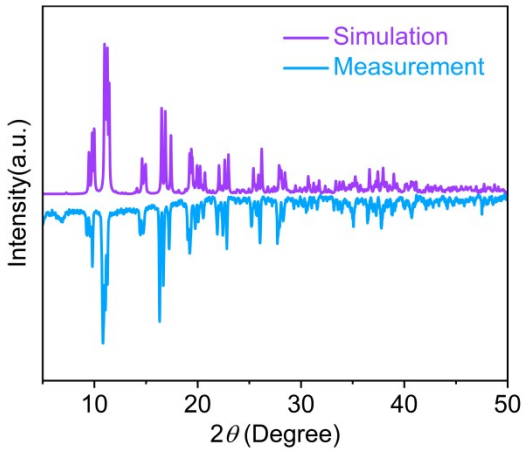

Figure S1. PXRD patterns of **1** were measured at room temperature.

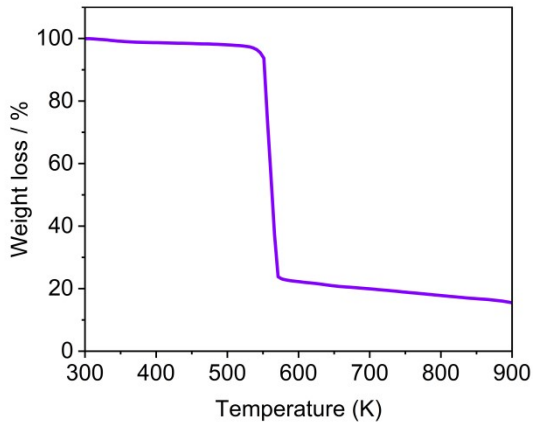

Figure S2. The thermogravimetric (TG) curves of **1** were recorded under a nitrogen atmosphere.

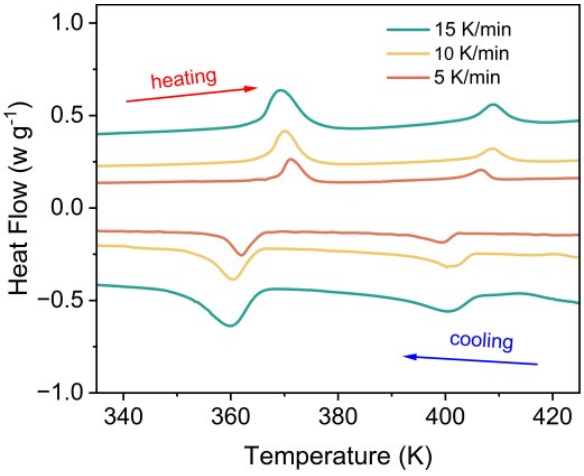

Figure S3. DSC curves of **1** measured a cooling-heating cycle with different scanning rates.

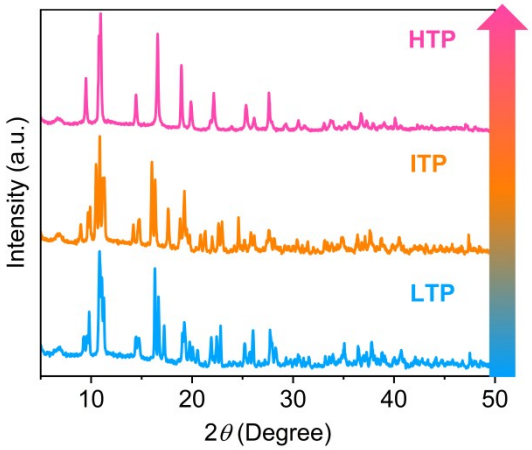

Figure S4. Measurement of PXRD patterns of LTP, ITP, and HTP of **1**.

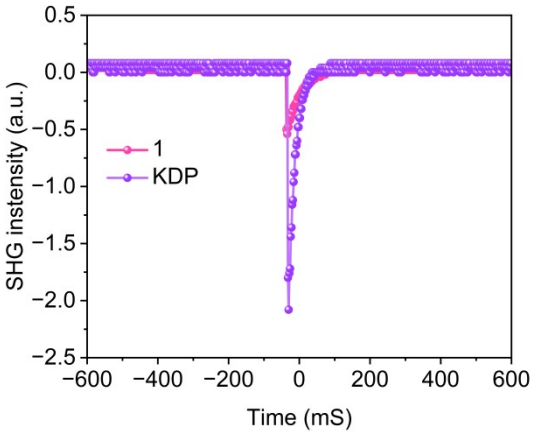

Figure S5. The SHG intensity of **1** is compared with that of the standard reference material KDP at room temperature.

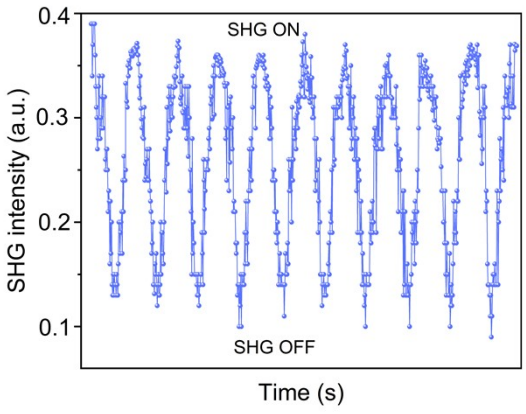

Figure S6. Plot of SHG intensity obtained for switching experiment of **1** with dT/dt = 30 K min^{-1} between 330 and 440 K.

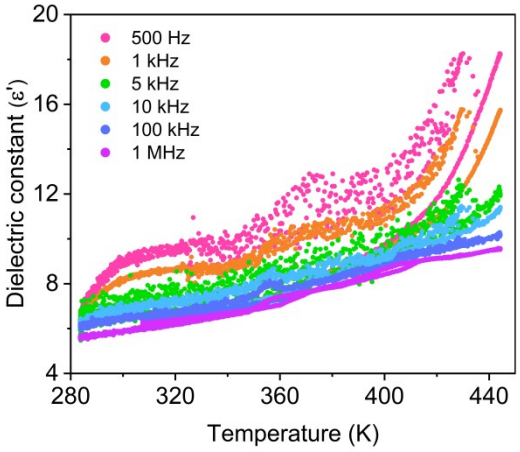

Figure. S7 Switchable *T*-*ε′* curves of **1** measured at 500 Hz to 1 MHz.

Supplemental Tables

Table S1 The elemental analysis for **1**.

$(RM3HQ)_4K_2[Nd(NO_3)_6]_2$	Anal. (%)	Found (%)
C	22.88	22.84
H	3.84	3.86
N	13.34	13.38

The description of "The element analysis for **1**. Anal. (%) calc.: C 22.88, H 3.84, N 13.34. Found (%): C 22.84, H 3.86, N 13.38."

Table S2. Crystal data and refinement detail of **1** at 293 K, 390 K, and 428 K.

T / K	**293**	**390**	**428**
Formula weight	837.82	809.77	779.56
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1$	$P2_1$	$P6_322$
a / Å	10.1821(2)	9.97856(6)	10.6105(5)
b / Å	15.9260(3)	16.0494(6)	10.6105(5)
c / Å	18.6690(4)	10.7872(7)	16.2217(11)
α / °	90	90	90
β / °	91.366(2)	116.318(7)	90
γ / °	90	90	120
V / Å^3	3026.51(10)	1548.49(17)	1581.61(19)
Z	4	2	2
λ/Å	0.71073	0.71073	0.71073
D_{calc} / g·cm^{-3}	1.839	1.737	1.637
μ / mm^{-1}	1.951	1.903	1.859
F(000)	1684	810	754
2θ range / °	2.18−30.89	4.21−62.05	2.22−31.00
Reflns collected	32364	35254	12056
Independent reflns (R_{int})	14093 (0.0361)	8099 (0.0472)	2610 (0.0374)
No. of parameters	833	435	173
R_1[a], wR_2[b] [I>2σ(I)]	0.0362, 0.0859	0.0742, 0.1984	0.0540, 0.1299
R_1, wR_2 [all data]	0.0424, 0.0891	0.1085, 0.2234	0.1069, 0.1615
GOF	1.024	0.963	0.956
$\Delta\rho$[c] / e·Å^{-3}	0.96, −0.946	1.48, −0.87	0.39, −0.307
CCDC	2445289	2445290	2445291

[a]$R_1=\Sigma||F_o|-|F_c||/|F_o|$; [b]$wR_2 = [\Sigma w(F_o^2-F_c^2)^2]/\Sigma w(F_o^2)^2]^{1/2}$; [c]maximum and minimum residual electron density.

Table S3. The T_c based on organic, inorganic and other hybrid perovskite ferroelectrics and reported cases of this work.

Compound	T_c / Ref.

	K	
Hybrid rare-earth double ferroelectrics		
$(R$M3HQ$)_2$RbLa$(NO_3)_6$	278	49
$(R$M3HQ$)_2$RbPr$(NO_3)_6$	280	41
$(R$M3HQ$)_2$RbEu$(NO_3)_6$	285	50
$(R$3HQ$)_4$KCe$(NO3)_8$	323	39
$(R$3HQ$)_4$KNd$(NO3)_8$	317	6
$(R$3HQ$)_4$RbCe$(NO3)_8$	285	52
$(HQ)_4$KEu$(NO3)_8$	291	53
$(S3HQ)_4$EuRb$(NO_3)_8$	94	54
$[(R)$-CM3HQ$]_2$CsEu$(NO_3)_6$	344	55
$[R$-3-HDMP$]_2$RbSm$(NO_3)_6$	251	56
$(R$M3HQ$)_4K_2[Nd(NO_3)_6]_2$	410	This work
Hybrid double ferroelectrics		
(3-Hydroxypyrrolidinium$)_2$RbBiBr$_6$	432	51
$(R$3HP$)_2$RbBiBr$_6$	485	15
$[(CH_3)_3NOH]_2[KCo(CN)_6]$	416	10
$[(CH_3)_3NOH]_2[KFe(CN)_6]$	406	10
$(R$-3P$)_2$KBiCl$_6$	392	51
(chloropropylammonium$)_4$AgBiBr$_8$	305	57
(3,3-difluorocyclobutanaminium$)_4$AgInBr$_8$	230	58
$[CH_2ClN(CH_3)_3]_2NaRuCl_6$	320	59
$(FCH_2CH_2NH_3)2[KFe(CN)_6]$	390	60

Table S4. Selected bond lengths [Å] and angles [°] at 293 K, 390 K, and 428 K.

293 K			
Nd(01)−O(005)	2.647(5)	O(005)−Nd(01)−O(006)	115.64(15)
Nd(01)−O(006)	2.697(5)	O(007)−Nd(01)−O(005)	177.23(17)
Nd(01)−O(007)	2.590(5)	O(007)−Nd(01)−O(006)	63.19(16)
Nd(01)−O(008)	2.570(5)	O(007)−Nd(01)−O(009)	69.89(16)
Nd(01)−O(009)	2.592(5)	O(007)−Nd(01)−O(00A)	116.40(16)
Nd(01)−O(00A)	2.605(4)	O(007)−Nd(01)−O(00C)	71.46(16)
Nd(01)−O(00C)	2.615(5)	O(007)−Nd(01)−O(00F)	131.89(16)
Nd(01)−O(00F)	2.638(4)	O(007)−Nd(01)−O(00G)	65.06(17)
Nd(01)−O(00G)	2.633(5)	O(007)−Nd(01)−O(00I)	113.27(16)
Nd(01)−O(00I)	2.603(5)	O(007)−Nd(01)−O(00U)	113.41(17)
Nd(01)−O(00P)	2.565(5)	O(008)−Nd(01)−O(005)	67.08(15)

Nd(01)−O(00U)	2.591(5)	O(008)−Nd(01)−O(006)	69.99(16)
Nd(02)−O(00E)	2.607(6)	O(008)−Nd(01)−O(007)	110.21(16)
Nd(02)−O(00J)	2.611(5)	O(008)−Nd(01)−O(009)	110.74(15)
Nd(02)−O(00Q)	2.614(5)	O(008)−Nd(01)−O(00A)	127.91(15)
Nd(02)−O(00R)	2.606(6)	O(008)−Nd(01)−O(00C)	175.07(16)
Nd(02)−O(00T)	2.582(6)	O(008)−Nd(01)−O(00F)	64.74(15)
Nd(02)−O(00V)	2.610(5)	O(008)−Nd(01)−O(00G)	49.27(16)
Nd(02)−O(012)	2.606(5)	O(008)−Nd(01)−O(00I)	69.80(15)
Nd(02)−O(014)	2.618(5)	O(008)−Nd(01)−O(00U)	114.47(16)
Nd(02)−O(016)	2.648(6)	O(009)−Nd(01)−O(005)	111.37(15)
Nd(02)−O(018)	2.588(5)	O(009)−Nd(01)−O(006)	47.83(15)
Nd(02)−O(01D)	2.648(5)	O(009)−Nd(01)−O(00A)	67.55(15)
Nd(02)−O(01E)	2.571(6)	(01E)−Nd(02)−O(01D)	175.97(18)
K(003)−O(00B)	2.925(5)	O(010)−K(003)−O(00B)	75.90(18)
K(003)−O(00L)	2.824(5)	N(00Z)−O(009)−Nd(01)	99.4(4)
K(003)−O(010)	2.780(6)	N(00Y)−O(00A)−Nd(01)	98.5(3)
K(003)−O(016)#1	3.024(6)	C(01C)−O(00B)−K(003)	116.4(4)
K(003)−O(018)#1	3.087(7)	N(00Y)−O(00C)−Nd(01)	97.0(3)
K(003)−O(01A)#2	2.830(6)	C(00X)−O(00D)−K(004)	119.6(4)
K(003)−O(01D)#1	3.026(6)	N(01L)−O(00E)−Nd(02)	98.2(5)
K(003)−O(01U)	2.820(6)	N(00W)−O(00F)−Nd(01)	97.6(3)
K(004)−O(006)#3	2.966(6)	O(010)−K(003)−O(00B)	75.90(18)
K(004)−O(007)#3	3.080(6)	N(00Z)−O(009)−Nd(01)	99.4(4)
K(004)−O(00D)	2.849(6)	N(00Y)−O(00A)−Nd(01)	98.5(3)
K(004)−O(00G)#3	3.018(6)	C(01C)−O(00B)−K(003)	116.4(4)
K(004)−O(00H)#2	2.992(6)	N(00Y)−O(00C)−Nd(01)	97.0(3)
K(004)−O(00O)	2.813(6)	C(00X)−O(00D)−K(004)	119.6(4)
K(004)−O(019)#4	2.919(6)	N(01L)−O(00E)−Nd(02)	98.2(5)
K(004)−O(026)	2.812(8)	N(00W)−O(00F)−Nd(01)	97.6(3)

Symmetry codes: [#1]: −*x*, 1−*y*, 1−*z*; [#2]: 1−*x*, 1−*y*, 2−*z*; [#3]: −0.5+*x*, 0.5−*y*, −0.5+*z*; [#4]: −0.5+*x*, 0.5−*y*, 0.5+*z*; [#5]: 0.5+*x*, 0.5−*y*, 0.5+*z*; [#6]: 0.5+*x*, 0.5−*y*, −0.5+*z*.

390 K

Nd(1)−O(1)	2.609(16)	O(22)#1−Nd(1)−O(1)	113.0(6)
Nd(1)−O(4)	2.562(16)	O(22)#1−Nd(1)−O(6)	112.1(6)
Nd(1)−O(6)	2.571(18)	O(22)#1−Nd(1)−O(12)	111.1(6)
Nd(1)−O(12)	2.603(16)	O(22)#1−Nd(1)−O(16)#1	48.7(6)
Nd(1)−O(16)#1	2.633(15)	O(22)#1−Nd(1)−O(2)	66.9(6)
Nd(1)−O(22)#1	2.571(18)	O(22)#1−Nd(1)−O(19)	42.4(9)
Nd(1)−O(2)	2.604(15)	O(22)#1−Nd(1)−O(11)#2	178.9(7)
Nd(1)−O(9)	2.54(3)	O(2)−Nd(1)−O(1)	110.6(5)
Nd(1)−O(19)	2.61(3)	O(2)−Nd(1)−O(12)	67.2(5)
Nd(1)−O(5)	2.54(3)	O(2)−Nd(1)−O(16)#1	69.3(6)
Nd(1)−O(21)#2	2.56(2)	O(2)−Nd(1)−O(19)	81.3(9)
Nd(1)−O(11)#2	2.604(18)	O(2)−Nd(1)−O(11)#2	112.0(6)
K(1)−O(1)#3	3.15(2)	O(9)−Nd(1)−O(1)	89.8(9)
K(1)−O(8)#3	3.11(3)	O(9)−Nd(1)−O(12)	137.4(10)
K(1)−O(16)	2.960(17)	O(9)−Nd(1)−O(2)	147.7(10)
K(1)−O(18)	2.889(14)	O(9)−Nd(1)−O(19)	68.9(12)
K(1)−O(20)	2.861(16)	O(9)−Nd(1)−O(5)	70.4(11)
K(1)−O(3)#4	2.838(15)	O(9)−Nd(1)−O(11)#2	98.8(11)
K(1)−O(13)	2.92(2)	O(5)−Nd(1)−O(1)	145.9(10)
K(1)−H(13)	2.1498	O(5)−Nd(1)−O(12)	144.1(8)
K(1)−O(17)#3	3.16(4)	O(5)−Nd(1)−O(2)	100.1(9)
K(1)−O(23)	2.99(4)	O(5)−Nd(1)−O(19)	49.8(11)
O(1)−Nd(1)−O(12)	47.6(5)	O(5)−Nd(1)−O(11)#2	88.3(9)
O(1)−Nd(1)−O(16)#1	67.2(6)	O(21)#2−Nd(1)−O(1)	109.6(8)
O(1)−Nd(1)−O(19)	147.5(8)	O(22)#1−Nd(1)−O(1)	113.0(6)
O(1)−Nd(1)−O(11)#2	67.1(7)	O(22)#1−Nd(1)−O(6)	112.1(6)
O(4)−Nd(1)−O(1)	176.7(6)	O(22)#1−Nd(1)−O(12)	111.1(6)

O(4)−Nd(1)−O(6)	114.1(7)	O(22)#1−Nd(1)−O(16)#1	48.7(6)
O(4)−Nd(1)−O(12)	129.1(6)	O(22)#1−Nd(1)−O(2)	66.9(6)
O(4)−Nd(1)−O(16)#1	112.8(7)	O(22)#1−Nd(1)−O(19)	42.4(9)
O(4)−Nd(1)−O(22)#1	68.1(6)	O(22)#1−Nd(1)−O(11)#2	178.9(7)
O(4)−Nd(1)−O(2)	66.7(6)	O(2)−Nd(1)−O(1)	110.6(5)
O(4)−Nd(1)−O(11)#2	111.7(7)	O(2)−Nd(1)−O(12)	67.2(5)
O(6)−Nd(1)−O(1)	68.6(6)	O(2)−Nd(1)−O(16)#1	69.3(6)
O(6)−Nd(1)−O(12)	112.5(6)	O(2)−Nd(1)−O(19)	81.3(9)
O(6)−Nd(1)−O(16)#1	109.2(6)	O(2)−Nd(1)−O(11)#2	112.0(6)
O(6)−Nd(1)−O(2)	178.5(6)	O(9)−Nd(1)−O(1)	89.8(9)
O(6)−Nd(1)−O(11)#2	69.0(7)	O(9)−Nd(1)−O(12)	137.4(10)
O(12)−Nd(1)−O(16)#1	68.3(6)	O(9)−Nd(1)−O(2)	147.7(10)
O(12)−Nd(1)−O(19)	146.7(10)	O(9)−Nd(1)−O(19)	68.9(12)

Symmetry codes: #1: 2−*x*, 2−*y*, 1−*z*; #2: 2−*x*, +*y*, 1−*z*; #3: +*x*, 2−*y*, +*z*; #4: +*x*, 1−*y*, +*z*; #5: 1−*x*, +*y*, −*z*; #6: 1−*x*, 1−*y*, −*z*; #7: 1−*x*, +*y*, 1−*z*; #8: 1−*x*, −*y*, 1−*z*; #9: +*x*, −*y*, +*z*; #10: 1−*x*, 1−*y*, 1−*z*.

428 K

Nd(1)−O(15)#1	2.59(2)	O(15)#1−Nd(1)−O(15)#2	104.1(9)
Nd(1)−O(15)#2	2.59(2)	O(15)#1−Nd(1)−O(15)#3	104.1(9)
Nd(1)−O(15)#3	2.59(2)	O(15)#2−Nd(1)−O(15)#3	104.1(9)
Nd(1)−O(8)#1	2.592(16)	O(15)#1−Nd(1)−O(8)#1	34.1(8)
Nd(1)−O(8)#2	2.592(16)	O(15)#2−Nd(1)−O(8)#1	76.9(8)
Nd(1)−O(8)#3	2.592(16)	O(15)#3−Nd(1)−O(8)#1	131.0(9)
Nd(1)−O(1)#4	2.604(16)	O(15)#1−Nd(1)−O(8)#2	131.0(9)
Nd(1)−O(1)	2.604(16)	O(15)#2−Nd(1)−O(8)#2	34.1(8)
Nd(1)−O(1)#5	2.604(16)	O(15)#3−Nd(1)−O(8)#2	76.9(8)
Nd(1)−O(7)	2.60(2)	O(8)#1−Nd(1)−O(8)#2	110.2(5)
Nd(1)−O(7)#5	2.60(2)	O(15)#1−Nd(1)−O(8)#3	76.9(8)
Nd(1)−O(7)#4	2.60(2)	O(15)#2−Nd(1)−O(8)#3	131.0(9)
Nd(1)−O(2)#4	2.611(13)	O(15)#3−Nd(1)−O(8)#3	34.1(8)

Nd(1)−O(2)#5	2.611(13)	O(8)#1−Nd(1)−O(8)#3	110.2(5)
Nd(1)−O(2)	2.611(13)	O(8)#2−Nd(1)−O(8)#3	110.2(5)
Nd(1)−O(9)	2.61(3)	O(15)#1−Nd(1)−O(1)#4	135.1(8)
Nd(1)−O(9)#4	2.61(3)	O(15)#2−Nd(1)−O(1)#4	119.7(10)
Nd(1)−O(9)#5	2.61(3)	O(15)#3−Nd(1)−O(1)#4	76.3(9)
K(2)−O(4)#4	2.860(19)	O(8)#1−Nd(1)−O(1)#4	146.5(6)
K(2)−O(4)	2.860(19)	O(8)#2−Nd(1)−O(1)#4	93.4(6)
K(2)−O(4)#5	2.860(19)	O(8)#3−Nd(1)−O(1)#4	81.7(7)
K(2)−O(6)#4	2.99(8)	O(15)#1−Nd(1)−O(1)	119.7(10)
K(2)−O(6)#5	2.99(8)	O(15)#2−Nd(1)−O(1)	76.3(9)
K(2)−O(6)	2.99(8)	O(15)#3−Nd(1)−O(1)	135.1(8)
K(2)−O(2)	3.087(15)	O(8)#1−Nd(1)−O(1)	93.4(6)
K(2)−O(2)#4	3.087(16)	O(8)#2−Nd(1)−O(1)	81.7(7)
K(2)−O(2)#5	3.087(15)	O(8)#3−Nd(1)−O(1)	146.5(6)
K(2)−O(1)#4	3.106(18)	O(1)#4−Nd(1)−O(1)	66.0(7)
K(2)−O(1)#5	3.106(18)	O(15)#1−Nd(1)−O(1)#5	76.3(9)
K(2)−O(1)	3.106(18)	O(15)#2−Nd(1)−O(1)#5	135.1(8)
O(1)−N(2)	1.209(18)	O(15)#3−Nd(1)−O(1)#5	119.7(10)
O(2)−N(2)	1.209(17)	O(8)#1−Nd(1)−O(1)#5	81.7(7)
N(3)−O(15)	1.09(3)	O(8)#2−Nd(1)−O(1)#5	146.5(6)
N(3)−O(4)	1.20(3)	O(8)#3−Nd(1)−O(1)#5	93.4(6)
N(3)−O(8)	1.26(3)	O(1)#4−Nd(1)−O(1)#5	66.0(7)
O(8)−O(15)	1.52(3)	O(1)−Nd(1)−O(1)#5	66.0(7)
O(8)−Nd(1)#6	2.592(16)	O(15)#1−Nd(1)−O(7)	100.6(8)
O(15)−Nd(1)#6	2.59(2)	O(15)#2−Nd(1)−O(7)	49.6(7)

Symmetry codes: [#1]: 2−*x*, 2−*y*, 1−*z*; [#2]: 2−*x*, +*y*, 1−*z*; [#3]: +*x*, 2−*y*, +*z*; [#4]: +*x*, 1−*y*, +*z*; [#5]: 1−*x*, +*y*, −*z*; [#6]: 1−*x*, 1−*y*, −*z*; [#7]: 1−*x*, +*y*, 1−*z*; [#8]: 1−*x*, −*y*, 1−*z*; [#9]: +*x*, −*y*, +*z*; [#10]: 1−*x*, 1−*y*, 1−*z*.