



High-resolution crystal structure of the double nitrate hydrate $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$

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This study introduces bis[hexakis(nitrato- κ^2O,O')lanthanum(III)] tris[hexa-aquanickel(II)] hexahydrate, $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$, with a structure refined in the hexagonal space group $R\bar{3}$. The salt comprises $[\text{La}(\text{NO}_3)_6]^{3-}$ icosahedra and $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ octahedra, thus forming an intricate network of interpenetrating honeycomb lattices arranged in layers. This arrangement is stabilized through strong hydrogen bonds. Two successive layers are connected *via* the second $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ octahedra, forming sheets which are stacked perpendicular to the *c* axis and held in the crystal by van der Waals forces. The synthesis of $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$ involves dissolving lanthanum(III) and nickel(II) oxides in nitric acid, followed by slow evaporation, yielding green hexagonal plate-like crystals.

1. Chemical context

Double nitrates, which contain two different metal cations and nitrate anions, have applications in various fields. They exhibit unique solubility properties, crystalline structures and special magnetic properties, and act as excellent precursors for the synthesis of mixed oxides. For example, double nitrates of Zn and Cu have been used to produce a mixed Cu and Zn oxide that exhibits high catalytic activity in reactions such as the water gas shift reaction to produce CO_2 and H_2 from CO and H_2O (Smith *et al.*, 2010), as well as selective CO_2 hydrogenation into methanol (Zhong *et al.*, 2020). Rare earth (RE) transition-metal (TM) double nitrates with the general formula $\text{RE}_2\text{TM}_3(\text{NO}_3)_{12} \cdot 24\text{H}_2\text{O}$ attracted much attention in the 1960s, in which the RE is a trivalent cation with an atomic number lower than that of Ho and the TM is a divalent cation, including Mg^{2+} , Mn^{2+} , Fe^{2+} , Co^{2+} , Ni^{2+} , Cu^{2+} and Zn^{2+} (Hellwege & Hellwege, 1953; Brochard & Hellwege, 1953; Buckmaster *et al.*, 1968). It should be noted that when RE has a +4 oxidation state, double nitrates are isomorphic with the triclinic $\text{MgTh}(\text{NO}_3)_6(\text{H}_2\text{O})_8$ salt (Šćavničar & Prodić, 1965). Cerium(III) magnesium and cerium(III) zinc double nitrates have been used extensively in nuclear orientation experiments because very low temperatures can be obtained by adiabatic demagnetization of the salt (Culvahouse, 1961). Their properties make them suitable magnetic thermometers (Thornley, 1963). Like ruby single crystals ($\text{Cr}^{3+}:\text{Al}_2\text{O}_3$), a Ce-doped lanthanum magnesium double nitrate is an ideal medium to study phonon avalanche, a delayed and sudden relaxation of paramagnetic ions by the emission of phonons (Mims & Taylor, 1969). Heat capacity and susceptibility measurements suggested that Mn, Ni, Co and Cu lanthanum double nitrates show antiferromagnetic transitions below 0.5 K (Mess *et al.*, 1967, 1968). The lack of high-quality crystalline structures of these salts limits the profound understanding of the magnetic

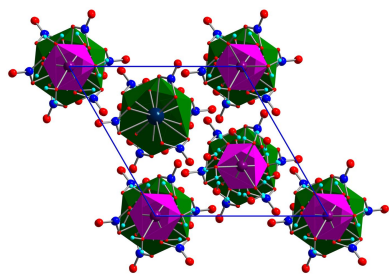


Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O8—H8A \cdots O9	0.85	2.00	2.8386 (15)	169
O10—H10A \cdots O9 ⁱ	0.85	2.03	2.8149 (13)	153
O10—H10B \cdots O5 ⁱⁱ	0.86	2.14	2.9626 (13)	161
O7—H7A \cdots O4 ⁱⁱⁱ	0.86	1.92	2.7656 (11)	168
O7—H7B \cdots O6 ^{iv}	0.86	2.13	2.9393 (11)	158

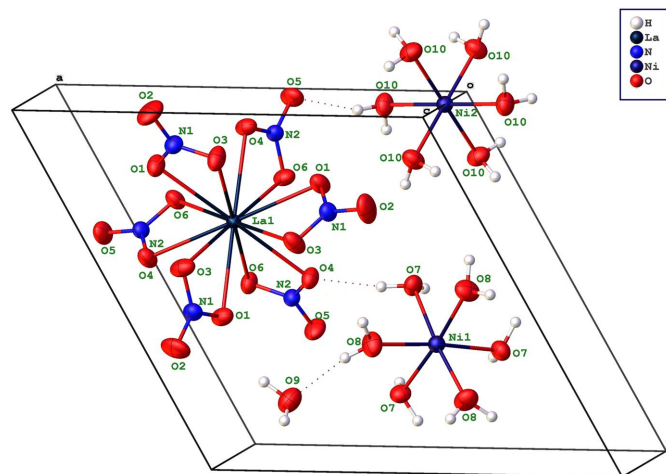
Symmetry codes: (i) $-x + y, -x + 1, z$; (ii) $x - y, x, -z + 1$; (iii) $-y, x - y, z$; (iv) $y, -x + y + 1, -z + 1$.

properties, as well as their theoretical investigation. We report herein on the growth of centimeter-large crystals of $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$ (Fig. 1) and the crystal structure determined by single-crystal X-ray diffraction.

2. Structural commentary

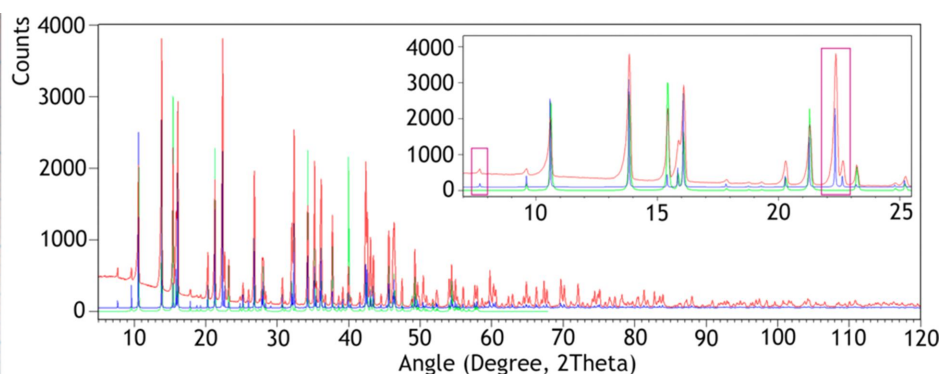
Similar to those found in the corresponding magnesium double salt, the title compound is made up of two types of ions, $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ and $[\text{La}(\text{NO}_3)_6]^{3-}$, which are linked together by hydrogen bonds with water molecules in the structure. The La atom on the threefold axis is coordinated by 12 O atoms from six nitrate groups to form a slightly distorted icosahedron. The La—O distances range from 2.6339 (8) to 2.7012 (8) Å, which are comparable to those found in $\text{La}_2\text{Mg}_3(\text{NO}_3)_{12} \cdot 24\text{H}_2\text{O}$ determined by neutron diffraction (Anderson *et al.*, 1977). As depicted in Fig. 2, the structure includes two crystallographically independent positions for Ni^{2+} . Three water H7a—O7—H7b molecules and three water H8a—O8—H8b molecules surround Ni1, resulting in a distorted $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ octahedron with C_3 symmetry. In contrast, the Ni2-containing $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ octahedron is highly symmetric, as Ni2 is situated in a site with $\bar{3}$ symmetry. The Ni—O bond lengths in both octahedra vary from 2.0471 (8) to 2.0531 (8) Å, similar to those found in $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$ (Breternitz *et al.*, 2015).

As illustrated in Fig. 3, each $[\text{La}(\text{NO}_3)_6]^{3-}$ icosahedron is surrounded by three Ni1-containing $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ clusters, and each Ni1-containing octahedron is surrounded by three


Figure 2

The molecular structure of $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$, with displacement ellipsoids for all non-H atoms drawn at the 50% probability level. H atoms are represented by small spheres of arbitrary radius. H-atom labels have been omitted for clarity. The colour scheme for the different elements can be found in the legend. The viewing direction is slightly tilted from the c axis, in order to prevent overlap between atoms. Hydrogen bonds are indicated with thin dotted lines.

icosahedra. These two interpenetrating honeycomb networks are arranged in a layer parallel to the ab plane. In this layer, the icosahedra are linked to the $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ clusters through strong O7—H7A \cdots O4, O9—H9A \cdots O1 and O10—H10A \cdots O9 hydrogen bonds. The six water H9A—O9—H9B molecules per unit cell do not participate in the coordination of either La or Ni. Two successive layers are separated by Ni2-containing $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ clusters, which bridge the layers between them *via* O10—H10A \cdots O9 hydrogen bonds. The complex hydrogen-bonding network between the clusters is shown in Fig. 2 and the actual data for the hydrogen bonds are given in Table 1. The network of bonded clusters form sheets that are stacked perpendicular to the c axis (Fig. 4). The sheets are held together by van der Waals forces in the $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$ structure.


Figure 1

Left: large green crystals of $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$ with a pseudo-hexagonal shape placed on a scale paper. The distance between two thick lines is 1 cm. Right: powder X-ray diffraction patterns of $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$. The PDF5 (ID = 00-049-1235) pattern available in the database is shown in green. The PXRD pattern calculated from the structure refined from single-crystal data and the experimental pattern measured on a Panalytical Empyrean diffractometer with $\text{Cu } K\alpha_1$ radiation ($\lambda = 1.540596$ Å) are shown in blue and red, respectively. In the inset, the high-intensity diffraction peaks absent in the PDF5 pattern are highlighted with purple rectangles.

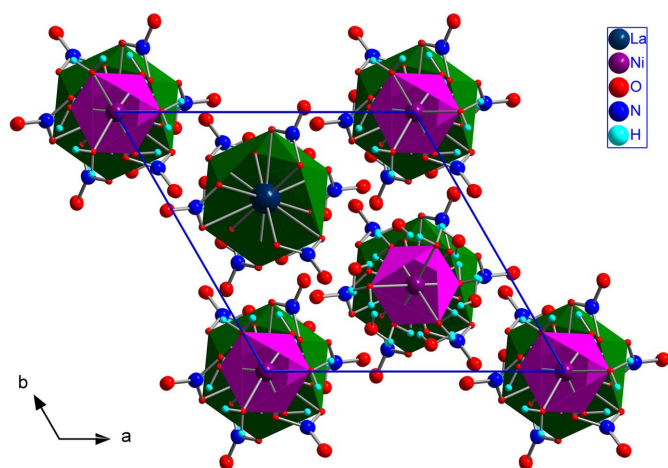


Figure 3
The $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$ structure represented along the c axis. The colour scheme for the different elements of the structure can be found in the figure inset. $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ octahedra and $[\text{La}(\text{NO}_3)_6]^{3-}$ icosahedra are plotted as pink and green front-opening polyhedron, respectively. The unit-cell edges are plotted with blue lines.

3. Database survey

No record of the same compound was found in the Crystallography Open Database (COD) or the Inorganic Crystal Structure Database (ICSD). It is listed only once in the Powder Diffraction File (PDF) 2024 version, entry 00-049-1235, without any atomic positions provided. The powder X-ray diffraction (PXRD) pattern available in this database

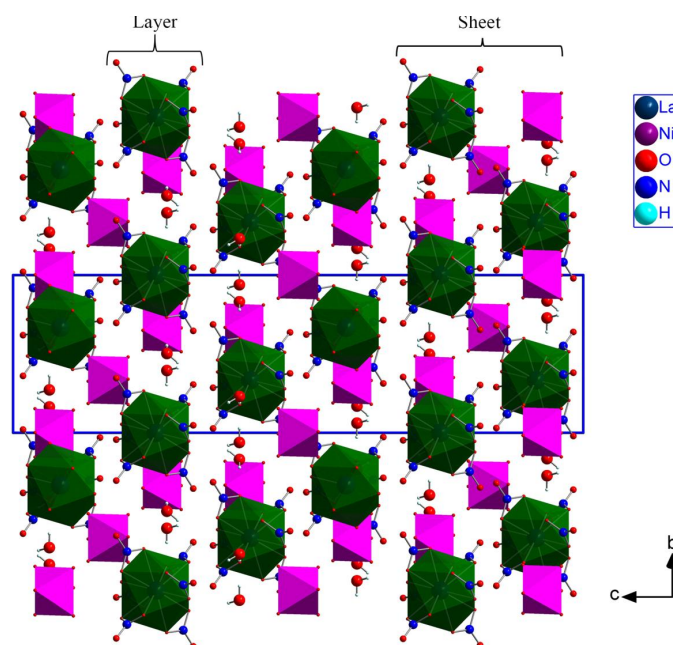


Figure 4
The $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$ structure represented along the a axis. The colour scheme for the different elements of the structure can be found in the inset. $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ octahedra and $[\text{La}(\text{NO}_3)_6]^{3-}$ icosahedra are plotted as pink and green polyhedra, respectively. The unit-cell edges are plotted with blue lines.

Table 2
Experimental details.

Crystal data	$[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$
Chemical formula	1630.45
M_r	Hexagonal, $R\bar{3}$
Crystal system, space group	297
Temperature (K)	11.0230 (1), 34.4826 (4)
a, c (Å)	3628.53 (8)
V (Å ³)	3
Z	Mo $K\alpha$
Radiation type	μ (mm ⁻¹)
μ (mm ⁻¹)	3.04
Crystal size (mm)	0.21 × 0.19 × 0.11
Data collection	XtaLAB Synergy-i HyPix3000
Diffractometer	Gaussian (<i>CrysAlis PRO</i> ; Rigaku OD, 2023)
Absorption correction	T_{\min}, T_{\max}
T_{\min}, T_{\max}	0.573, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	27264, 2833, 2663
R_{int}	0.023
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.746
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.013, 0.034, 1.06
No. of reflections	2833
No. of parameters	126
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.30, -0.38

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT2018* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b), *DIAMOND* (Brandenburg, 2006) and *publCIF* (Westrip, 2010).

deviates significantly from both the theoretical pattern simulated from the structure refined *via* single-crystal XRD data and the experimental pattern recorded with powder obtained by crushing a few $[\text{La}(\text{NO}_3)_6]_2[\text{Ni}(\text{H}_2\text{O})_6]_3 \cdot 6\text{H}_2\text{O}$ single crystals. Notably, peaks below 10° , as well as those in the $22\text{--}23^\circ$ region, are missing in the pattern found in PDF-00-49-1235 (Fig. 1).

4. Synthesis and crystallization

Lanthanum(III) oxide was dissolved in dilute HNO_3 with a concentration of 1 mol l^{-1} (1 M) and nickel(II) oxide in dilute HNO_3 with a concentration of 0.5 mol l^{-1} (0.5 M). In order to dissolve the nickel(II) oxide in the dilute HNO_3 , the solution was heated at 423 K over a period of 12 h until the nickel(II) oxide completely dissolved and a green transparent solution was obtained. Lanthanum(III) oxide solution (0.2 l) was first mixed with nickel(II) oxide solution (1.2 l) and then 1 mol of citric acid was added to the mixture under vigorous stirring until complete dissolution. The solution was transferred to a fume hood for slow evaporation. After 30 d, green hexagonal plate-shaped crystals formed with different sizes, the maximum dimension being 2 cm.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms on O atoms were first located in a difference Fourier map and then refined isotropically in riding mode, with $U_{\text{iso}}(\text{H})$ values of $1.5U_{\text{eq}}$ of the

parent O atoms. The O–H distance was refined against the residual peaks, without further constraint.

References

- Anderson, M. R., Jenkin, G. T. & White, J. W. (1977). *Acta Cryst.* **B33**, 3933–3936.
- Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Breternitz, J., Farrugia, L. J., Godula-Jopek, A., Saremi-Yarahmadi, S., Malka, I. E., Hoang, T. K. A. & Gregory, D. H. (2015). *J. Cryst. Growth*, **412**, 1–6.
- Brochard, J. & Hellwege, K. H. (1953). *Z. Phys.* **135**, 620–638.
- Buckmaster, H. A., Dering, J. C. & Fry, D. J. I. (1968). *J. Phys. C.: Solid State Phys.* **1**, 599–607.
- Culvahouse, J. W. (1961). *Phys. Rev.* **124**, 1413–1417.
- Hellwege, A. M. & Hellwege, K. H. (1953). *Z. Phys.* **135**, 615–619.
- Mess, K. W., Blöte, H. W. J. & Huiskamp, W. J. (1968). *Phys. Lett. A*, **27**, 353–354.
- Mess, K. W., Lagendijk, E. & Huiskamp, W. J. (1967). *Phys. Lett. A*, **25**, 329–331.
- Mims, W. B. & Taylor, D. R. (1969). *Phys. Rev. Lett.* **22**, 1430–1432.
- Rigaku OD (2023). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
- Ščavničar, S. & Prodić, B. (1965). *Acta Cryst.* **18**, 698–702.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Smith, B., Loganathan, M. & Shantha, M. (2010). *Int. J. Chem. Reactor Eng.* **8**. <https://doi.org/10.2202/1542>.
- Thornley, J. H. M. (1963). *Phys. Rev.* **132**, 1492–1493.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Zhong, J., Yang, X., Wu, Z., Liang, B., Huang, Y. & Zhang, Y. (2020). *Chem. Soc. Rev.* **49**, 1385–1413.

supporting information

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High-resolution crystal structure of the double nitrate hydrate

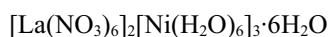


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Computing details

Bis[hexakis(nitrato- $\kappa^2\text{O},\text{O}'$)lanthanum(III)] tris[hexaaquanickel(II)] hexahydrate

Crystal data



$M_r = 1630.45$

Hexagonal, $R\bar{3}$

$a = 11.0230$ (1) Å

$c = 34.4826$ (4) Å

$V = 3628.53$ (8) Å³

$Z = 3$

$F(000) = 2430$

$D_x = 2.238$ Mg m⁻³

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

Cell parameters from 22661 reflections

$\theta = 2.2\text{--}36.2^\circ$

$\mu = 3.04$ mm⁻¹

$T = 297$ K

Plate, blue

0.21 × 0.19 × 0.11 mm

Data collection

XtaLAB Synergy-i HyPix3000
diffractometer

Radiation source: micro-focus sealed X-ray tube

ω scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2023)

$T_{\min} = 0.573$, $T_{\max} = 1.000$

27264 measured reflections

2833 independent reflections

2663 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 32.0^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -16 \rightarrow 16$

$k = -16 \rightarrow 16$

$l = -51 \rightarrow 51$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.013$

$wR(F^2) = 0.034$

$S = 1.06$

2833 reflections

126 parameters

0 restraints

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0161P)^2 + 2.1621P]$

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

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.30$ e Å⁻³

$\Delta\rho_{\min} = -0.38$ e Å⁻³

Extinction correction: SHELXL2018

(Sheldrick, 2015b),

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00015 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
La1	0.666667	0.333333	0.58402 (2)	0.01660 (3)
Ni1	0.333333	0.666667	0.59443 (2)	0.02146 (5)
O1	0.82133 (9)	0.59977 (8)	0.60265 (2)	0.03067 (17)
N1	0.88316 (10)	0.58535 (9)	0.63163 (3)	0.02614 (17)
Ni2	0.000000	0.000000	0.500000	0.02201 (6)
O2	0.97264 (11)	0.68693 (10)	0.64928 (3)	0.0487 (2)
N2	0.45188 (9)	0.08678 (9)	0.53286 (2)	0.02312 (16)
O3	0.84704 (10)	0.46142 (9)	0.64135 (2)	0.03567 (19)
O10	0.14730 (9)	0.15389 (9)	0.53499 (2)	0.03423 (18)
H10A	0.107682	0.185298	0.549969	0.051*
H10B	0.202599	0.224634	0.521158	0.051*
O9	0.76944 (11)	0.83079 (11)	0.60307 (3)	0.0422 (2)
H9A	0.807994	0.887099	0.621866	0.063*
H9B	0.807786	0.780452	0.602857	0.063*
O4	0.51001 (9)	0.06874 (8)	0.56231 (2)	0.02866 (16)
O5	0.36341 (10)	-0.01345 (9)	0.51426 (3)	0.03803 (19)
O6	0.49052 (9)	0.21141 (8)	0.52368 (2)	0.02951 (16)
O7	0.18944 (8)	0.68410 (8)	0.56094 (2)	0.02971 (16)
H7A	0.114671	0.604003	0.559052	0.045*
H7B	0.219721	0.704444	0.537602	0.045*
O8	0.48738 (10)	0.66808 (11)	0.62819 (3)	0.0386 (2)
H8A	0.572758	0.707366	0.621395	0.058*
H8B	0.478129	0.605850	0.644424	0.058*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
La1	0.01709 (4)	0.01709 (4)	0.01561 (5)	0.00855 (2)	0.000	0.000
Ni1	0.02300 (7)	0.02300 (7)	0.01838 (10)	0.01150 (3)	0.000	0.000
O1	0.0387 (4)	0.0262 (4)	0.0281 (4)	0.0169 (3)	-0.0104 (3)	-0.0022 (3)
N1	0.0273 (4)	0.0246 (4)	0.0244 (4)	0.0113 (3)	-0.0047 (3)	-0.0029 (3)
Ni2	0.02193 (9)	0.02193 (9)	0.02215 (14)	0.01097 (4)	0.000	0.000
O2	0.0486 (6)	0.0326 (5)	0.0447 (5)	0.0052 (4)	-0.0219 (4)	-0.0080 (4)
N2	0.0238 (4)	0.0218 (4)	0.0237 (4)	0.0114 (3)	-0.0024 (3)	-0.0033 (3)
O3	0.0502 (5)	0.0268 (4)	0.0307 (4)	0.0198 (4)	-0.0134 (4)	-0.0016 (3)
O10	0.0303 (4)	0.0312 (4)	0.0322 (4)	0.0086 (3)	-0.0002 (3)	-0.0060 (3)
O9	0.0550 (6)	0.0493 (6)	0.0348 (5)	0.0353 (5)	0.0018 (4)	0.0013 (4)
O4	0.0368 (4)	0.0233 (3)	0.0258 (4)	0.0149 (3)	-0.0086 (3)	-0.0018 (3)
O5	0.0370 (4)	0.0285 (4)	0.0420 (5)	0.0115 (4)	-0.0174 (4)	-0.0113 (3)

O6	0.0382 (4)	0.0229 (3)	0.0281 (4)	0.0158 (3)	-0.0059 (3)	0.0002 (3)
O7	0.0262 (4)	0.0313 (4)	0.0279 (4)	0.0116 (3)	-0.0052 (3)	-0.0006 (3)
O8	0.0364 (4)	0.0502 (5)	0.0326 (4)	0.0244 (4)	-0.0028 (3)	0.0101 (4)

Geometric parameters (Å, °)

La1—O1	2.6339 (8)	N1—O2	1.2217 (12)
La1—O1 ⁱ	2.6340 (8)	N1—O3	1.2621 (12)
La1—O1 ⁱⁱ	2.6340 (8)	Ni2—O10	2.0531 (8)
La1—O4	2.6481 (8)	Ni2—O10 ^v	2.0531 (8)
La1—O4 ⁱⁱ	2.6481 (8)	Ni2—O10 ^{vi}	2.0531 (8)
La1—O4 ⁱ	2.6481 (8)	Ni2—O10 ^{vii}	2.0531 (8)
La1—O3	2.6546 (8)	Ni2—O10 ^{viii}	2.0531 (8)
La1—O3 ⁱ	2.6547 (8)	Ni2—O10 ^{ix}	2.0531 (8)
La1—O3 ⁱⁱ	2.6547 (8)	N2—O5	1.2271 (11)
La1—O6 ⁱⁱ	2.7011 (8)	N2—O6	1.2585 (11)
La1—O6 ⁱ	2.7011 (8)	N2—O4	1.2684 (11)
La1—O6	2.7012 (8)	O10—H10A	0.8536
Ni1—O7 ⁱⁱⁱ	2.0471 (8)	O10—H10B	0.8554
Ni1—O7 ^{iv}	2.0471 (8)	O9—H9A	0.8499
Ni1—O7	2.0472 (8)	O9—H9B	0.8492
Ni1—O8	2.0526	O7—H7A	0.8576
Ni1—O8 ⁱⁱⁱ	2.0526	O7—H7B	0.8571
Ni1—O8 ^{iv}	2.0526	O8—H8A	0.8489
O1—N1	1.2638 (11)	O8—H8B	0.8510
O1—La1—O1 ⁱ	114.254 (14)	O4—La1—O6	47.44 (2)
O1—La1—O1 ⁱⁱ	114.253 (14)	O4 ⁱⁱ —La1—O6	111.22 (2)
O1 ⁱ —La1—O1 ⁱⁱ	114.252 (14)	O4 ⁱ —La1—O6	70.19 (3)
O1—La1—O4	177.58 (2)	O3—La1—O6	177.63 (2)
O1 ⁱ —La1—O4	67.33 (2)	O3 ⁱ —La1—O6	111.60 (3)
O1 ⁱⁱ —La1—O4	66.00 (2)	O3 ⁱⁱ —La1—O6	110.71 (3)
O1—La1—O4 ⁱⁱ	67.33 (2)	O6 ⁱⁱ —La1—O6	67.04 (3)
O1 ⁱ —La1—O4 ⁱⁱ	66.00 (2)	O6 ⁱ —La1—O6	67.04 (3)
O1 ⁱⁱ —La1—O4 ⁱⁱ	177.58 (2)	O7 ⁱⁱⁱ —Ni1—O7 ^{iv}	91.31 (3)
O4—La1—O4 ⁱⁱ	112.340 (15)	O7 ⁱⁱⁱ —Ni1—O7	91.31 (3)
O1—La1—O4 ⁱ	66.00 (2)	O7 ^{iv} —Ni1—O7	91.31 (3)
O1 ⁱ —La1—O4 ⁱ	177.58 (2)	O7 ⁱⁱⁱ —Ni1—O8	92.55 (4)
O1 ⁱⁱ —La1—O4 ⁱ	67.33 (2)	O7 ^{iv} —Ni1—O8	85.37 (4)
O4—La1—O4 ⁱ	112.341 (15)	O7—Ni1—O8	174.96 (4)
O4 ⁱⁱ —La1—O4 ⁱ	112.340 (15)	O7 ⁱⁱⁱ —Ni1—O8 ⁱⁱⁱ	174.96 (4)
O1—La1—O3	47.95 (2)	O7 ^{iv} —Ni1—O8 ⁱⁱⁱ	92.55 (4)
O1 ⁱ —La1—O3	72.20 (3)	O7—Ni1—O8 ⁱⁱⁱ	85.37 (3)
O1 ⁱⁱ —La1—O3	115.50 (3)	O8—Ni1—O8 ⁱⁱⁱ	91.0
O4—La1—O3	134.33 (2)	O7 ⁱⁱⁱ —Ni1—O8 ^{iv}	85.37 (4)
O4 ⁱⁱ —La1—O3	66.91 (3)	O7 ^{iv} —Ni1—O8 ^{iv}	174.96 (4)
O4 ⁱ —La1—O3	108.96 (3)	O7—Ni1—O8 ^{iv}	92.55 (4)
O1—La1—O3 ⁱ	115.51 (3)	O8—Ni1—O8 ^{iv}	91.0

O1 ⁱ —La1—O3 ⁱ	47.95 (2)	O8 ⁱⁱⁱ —Ni1—O8 ^{iv}	91.0
O1 ⁱⁱ —La1—O3 ⁱ	72.20 (3)	N1—O1—La1	98.15 (6)
O4—La1—O3 ⁱ	66.91 (3)	O2—N1—O3	122.21 (10)
O4 ⁱⁱ —La1—O3 ⁱ	108.96 (3)	O2—N1—O1	121.18 (10)
O4 ⁱ —La1—O3 ⁱ	134.33 (2)	O3—N1—O1	116.61 (9)
O3—La1—O3 ⁱ	70.62 (3)	O10—Ni2—O10 ^v	180.0
O1—La1—O3 ⁱⁱ	72.20 (3)	O10—Ni2—O10 ^{vi}	91.03 (3)
O1 ⁱ —La1—O3 ⁱⁱ	115.50 (3)	O10 ^v —Ni2—O10 ^{vi}	88.97 (3)
O1 ⁱⁱ —La1—O3 ⁱⁱ	47.95 (2)	O10—Ni2—O10 ^{vii}	88.97 (3)
O4—La1—O3 ⁱⁱ	108.96 (3)	O10 ^v —Ni2—O10 ^{vii}	91.03 (3)
O4 ⁱⁱ —La1—O3 ⁱⁱ	134.33 (2)	O10 ^{vi} —Ni2—O10 ^{vii}	91.03 (3)
O4 ⁱ —La1—O3 ⁱⁱ	66.91 (3)	O10—Ni2—O10 ^{viii}	91.03 (3)
O3—La1—O3 ⁱⁱ	70.62 (3)	O10 ^v —Ni2—O10 ^{viii}	88.97 (3)
O3 ⁱ —La1—O3 ⁱⁱ	70.62 (3)	O10 ^{vi} —Ni2—O10 ^{viii}	88.97 (3)
O1—La1—O6 ⁱⁱ	108.56 (3)	O10 ^{vii} —Ni2—O10 ^{viii}	180.00 (4)
O1 ⁱ —La1—O6 ⁱⁱ	66.37 (3)	O10—Ni2—O10 ^{ix}	88.97 (3)
O1 ⁱⁱ —La1—O6 ⁱⁱ	130.25 (2)	O10 ^v —Ni2—O10 ^{ix}	91.03 (3)
O4—La1—O6 ⁱⁱ	70.19 (3)	O10 ^{vi} —Ni2—O10 ^{ix}	180.00 (4)
O4 ⁱⁱ —La1—O6 ⁱⁱ	47.43 (2)	O10 ^{vii} —Ni2—O10 ^{ix}	88.97 (3)
O4 ⁱ —La1—O6 ⁱⁱ	111.22 (2)	O10 ^{viii} —Ni2—O10 ^{ix}	91.03 (3)
O3—La1—O6 ⁱⁱ	111.60 (3)	O5—N2—O6	122.29 (9)
O3 ⁱ —La1—O6 ⁱⁱ	110.71 (3)	O5—N2—O4	120.90 (9)
O3 ⁱⁱ —La1—O6 ⁱⁱ	177.63 (2)	O6—N2—O4	116.80 (8)
O1—La1—O6 ⁱ	66.37 (3)	N1—O3—La1	97.19 (6)
O1 ⁱ —La1—O6 ⁱ	130.25 (2)	Ni2—O10—H10A	109.7
O1 ⁱⁱ —La1—O6 ⁱ	108.56 (3)	Ni2—O10—H10B	109.5
O4—La1—O6 ⁱ	111.22 (2)	H10A—O10—H10B	104.1
O4 ⁱⁱ —La1—O6 ⁱ	70.19 (3)	H9A—O9—H9B	104.7
O4 ⁱ —La1—O6 ⁱ	47.43 (2)	N2—O4—La1	98.97 (6)
O3—La1—O6 ⁱ	110.71 (3)	N2—O6—La1	96.66 (6)
O3 ⁱ —La1—O6 ⁱ	177.63 (2)	Ni1—O7—H7A	109.7
O3 ⁱⁱ —La1—O6 ⁱ	111.60 (3)	Ni1—O7—H7B	109.6
O6 ⁱⁱ —La1—O6 ⁱ	67.04 (3)	H7A—O7—H7B	104.2
O1—La1—O6	130.25 (2)	Ni1—O8—H8A	123.3
O1 ⁱ —La1—O6	108.57 (3)	Ni1—O8—H8B	127.0
O1 ⁱⁱ —La1—O6	66.37 (3)	H8A—O8—H8B	104.4
La1—O1—N1—O2	177.19 (10)	O5—N2—O4—La1	−177.06 (9)
La1—O1—N1—O3	−3.22 (10)	O6—N2—O4—La1	3.67 (9)
O2—N1—O3—La1	−177.23 (10)	O5—N2—O6—La1	177.16 (9)
O1—N1—O3—La1	3.19 (10)	O4—N2—O6—La1	−3.58 (9)

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O8—H8A \cdots O9	0.85	2.00	2.8386 (15)	169

O10—H10A···O9 ^{iv}	0.85	2.03	2.8149 (13)	153
O10—H10B···O5 ^{vi}	0.86	2.14	2.9626 (13)	161
O7—H7A···O4 ^{vii}	0.86	1.92	2.7656 (11)	168
O7—H7B···O6 ^x	0.86	2.13	2.9393 (11)	158

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