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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 Disorder in main residue
 R factor = 0.020
 wR factor = 0.044
 Data-to-parameter ratio = 32.5

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

1,4-Diazoniabicyclo[2.2.2]octane tetraqua- tetrachlorolanthanate(III) chloride

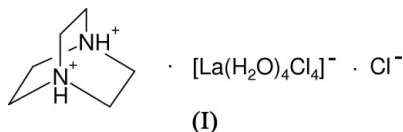
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The title compound, $(\text{C}_6\text{H}_{14}\text{N}_2)[\text{LaCl}_4(\text{H}_2\text{O})_4]\text{Cl}$, is built up from organic cations, $[\text{La}(\text{H}_2\text{O})_4\text{Cl}_4]^-$ complex anions and uncoordinated chloride ions. The previously unseen rare-earth-containing complex ion is irregular in shape. A network of $\text{O}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds helps to establish the structure. Prominent among these are two well defined trifurcated $\text{N}-\text{H}\cdots(\text{Cl},\text{Cl},\text{Cl})$ interactions. The La atom, one Cl atom, two N atoms and two C atoms possess site symmetry m .

Comment

The title compound, (I), which contains a new La/Cl/ H_2O complex ion, complements known adducts of lanthanum chloride with O-donor ligands such as the polymeric $[\text{LaCl}_3(\text{C}_7\text{H}_8\text{O}_2)_2]_n$ ($\text{C}_7\text{H}_8\text{O}_2$ is 2,6-dimethyl-4-pyrone; Bisi Castellani & Coda, 1985) and the dimeric $[\text{La}_2(\text{H}_2\text{O})_6\text{Cl}_8(\text{C}_6\text{H}_8\text{N})_2]$ ($\text{C}_6\text{H}_8\text{N}$ is 4-picoline; Mackenstedt & Urland, 1993). Although various materials containing $[\text{LaCl}_6]^{3-}$ (Matsumoto *et al.*, 2002), $[\text{La}(\text{H}_2\text{O})_8]^{3+}$ (Hardie *et al.*, 2001) and $[\text{La}(\text{H}_2\text{O})_9]^{3+}$ complex ions (Harrowfield *et al.*, 1983) have been described, the only monomeric La/Cl/ H_2O complex reported in version 5.27 of the Cambridge Structural Database (Allen, 2002) contains $[\text{La}(\text{H}_2\text{O})_6\text{Cl}_2]^+$ ions (Urland, 1985), although no atomic coordinates are available.



The $[\text{La}(\text{H}_2\text{O})_4\text{Cl}_4]^-$ species in (I) (Fig. 1) is generated from the unique atoms by mirror symmetry, with La1 and Cl1 lying on the reflecting plane. Refinements placing Cl2 on the plane led to a highly anisotropic displacement ellipsoid for this atom, and the final refinement cycles placed it just off the mirror plane, disordered over two symmetry-related positions [$\text{Cl}2\cdots\text{Cl}2^i = 0.530(4)\text{ \AA}$; symmetry code: (i) $x, \frac{1}{2} - y, z$]. The resulting LaO_4Cl_4 polyhedron is irregular in shape, although the mean La–O and La–Cl bond lengths of 2.547(2) and 2.8960(9) Å, respectively, are normal. The geometrical parameters for the 1,4-diazoniabicyclo[2.2.2]octane cation are also unexceptional (Bremner & Harrison, 2003).

As well as electrostatic forces, the component species in (I) are held together by a network of $\text{O}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds (Table 2). The most distinctive of these are two trifurcated $\text{N}-\text{H}\cdots(\text{Cl},\text{Cl},\text{Cl})$ interactions, one from each NH group of the $\text{C}_6\text{H}_{14}\text{N}_2^{2+}$ cation. This results (Fig. 2) in (001) sheets in which [100] columns of cations and anions

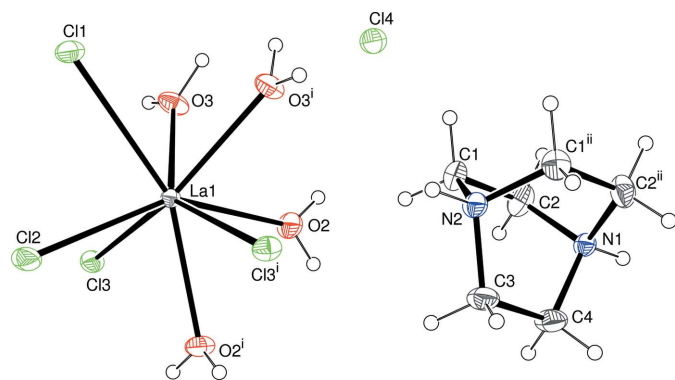


Figure 1
View of (I), showing 30% probability displacement ellipsoids (arbitrary spheres for the H atoms). Only one orientation of the disordered atom Cl2 is shown. [Symmetry codes: (i) $x, \frac{1}{2} - y, z$; (ii) $x, \frac{3}{2} - y, z$]

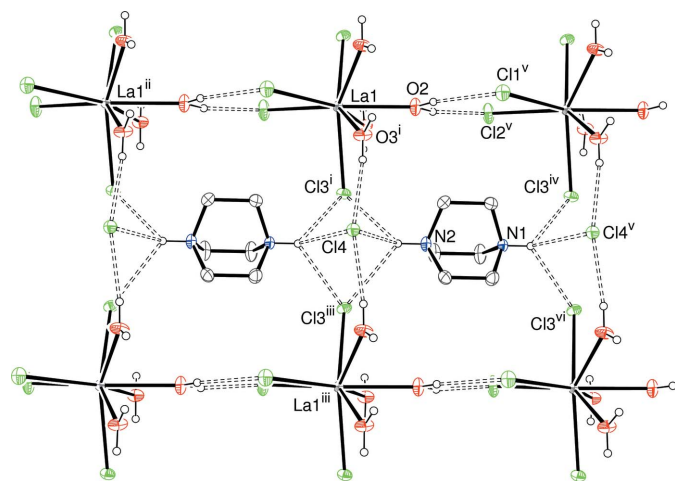


Figure 2
View down [001] of part of a $(C_6H_{14}N_2) \cdot [La(H_2O)_4Cl_4] \cdot Cl$ layer in (I), with hydrogen bonds shown as dashed lines. C-bound H atoms have been omitted for clarity and only one orientation of the disordered atom Cl2 is shown. [Symmetry codes: (i) $x, \frac{1}{2} - y, z$; (ii) $x + 1, y, z$; (iii) $x, y + 1, z$; (iv) $x - 1, \frac{1}{2} - y, z$; (v) $x - 1, y, z$; (vi) $x - 1, y + 1, z$]

alternate with respect to the [010] direction. Every trio of acceptor chloride ions (two Cl3 atoms bound to different La atoms and one free Cl4 species) accepts a trifurcated hydrogen bond from both N1/H6 and N2/H7. The average bond angles at H6 and H7 are 108 and 107°, respectively.

The [100] columns of $[La(H_2O)_4Cl_4]^-$ anions interact by way of two hydrogen bonds from the O2 water molecule to two chloride ion acceptors. Crystal symmetry results in all the anions in one (001) layer pointing in the same direction. The uncoordinated chloride ion participates in an O—H...Cl...H—O bridge between adjacent anion columns (Fig. 2). Still further O—H...Cl hydrogen bonds help to consolidate the inter-layer packing, to result in a dense three-dimensional network (Fig. 3).

Experimental

0.1 M LaCl₃, 1 M HCl and solid 1,4-diazabicyclo[2.2.2]octane (C₆H₁₂N₂) were mixed in a Petri dish in a 1:1:1 molar ratio, resulting

in a clear solution. Small block-like crystals of (I) appeared as the water slowly evaporated over a few days.

Crystal data

$(C_6H_{14}N_2)[LaCl_4(H_2O)_4]Cl$
 $M_r = 502.42$
 Orthorhombic, $Pnma$
 $a = 7.5005(3) \text{ \AA}$
 $b = 9.1759(4) \text{ \AA}$
 $c = 25.0388(10) \text{ \AA}$
 $V = 1723.27(12) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.937 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 3.26 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Block, colourless
 $0.35 \times 0.21 \times 0.16 \text{ mm}$

Data collection

Bruker SMART 1000 CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1999)
 $T_{min} = 0.395, T_{max} = 0.624$

18229 measured reflections
 3279 independent reflections
 2971 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.020$
 $\theta_{max} = 32.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.044$
 $S = 1.14$
 3279 reflections
 101 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0199P)^2 + 0.3724P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.58 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.43 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.00056 (9)

Table 1

Selected bond lengths (Å).

La1—O2	2.5240 (18)	La1—Cl2	2.8310 (8)
La1—O3	2.5492 (11)	La1—Cl3	2.8524 (4)
La1—O1	2.5650 (16)	La1—Cl1	3.0482 (6)

Table 2

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...Cl3 ⁱ	0.83	2.49	3.2429 (14)	151
O2—H2...Cl2 ⁱⁱ	0.84	2.28	3.1035 (19)	166
O2—H2...Cl2 ⁱⁱⁱ	0.84	2.28	3.1035 (19)	166
O2—H3...Cl1 ⁱⁱ	0.82	2.41	3.1834 (19)	159
O3—H4...Cl4 ^{iv}	0.83	2.33	3.1542 (12)	173
O3—H5...Cl1 ^v	0.93	2.26	3.1765 (12)	173
N1—H6...Cl3 ⁱⁱⁱ	0.91	2.67	3.2930 (15)	127
N1—H6...Cl3 ^{vi}	0.91	2.67	3.2930 (15)	127
N1—H6...Cl4 ⁱⁱ	0.91	2.71	3.3086 (19)	125
N2—H7...Cl4	0.91	2.54	3.208 (2)	131
N2—H7...Cl3 ^{vii}	0.91	2.80	3.3962 (17)	125
N2—H7...Cl3 ^{viii}	0.91	2.80	3.3962 (17)	125

Symmetry codes: (i) $-x + 1, -y, -z + 1$; (ii) $x - 1, y, z$; (iii) $x - 1, -y + \frac{1}{2}, z$; (iv) $x, y - 1, z$; (v) $x - \frac{1}{2}, y, -z + \frac{3}{2}$; (vi) $x - 1, y + 1, z$; (vii) $x, y + 1, z$; (viii) $x, -y + \frac{1}{2}, z$.

The O-bound H atoms were located in difference maps and refined as riding in their as-found relative locations. The C- and N-bound H atoms were placed in idealized locations (C—H = 0.97 and N—H = 0.91 Å) and refined as riding. The constraint $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$ was applied in all cases.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT-Plus (Bruker, 1999); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics:

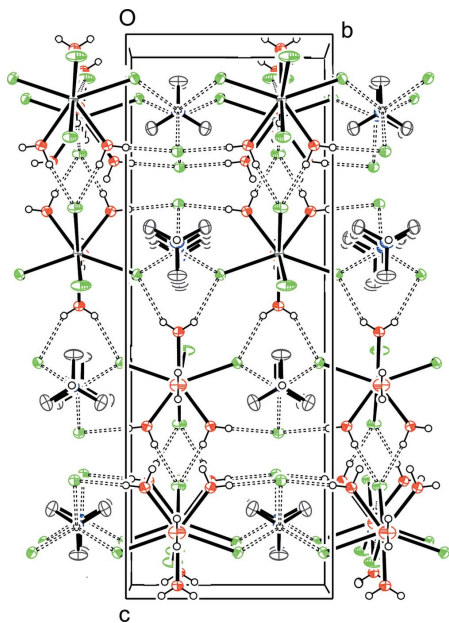


Figure 3
The packing in (I), viewed down [100]. Drawing conventions as in Fig. 2.

ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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supporting information

Acta Cryst. (2006). E62, m1255–m1257 [https://doi.org/10.1107/S1600536806016898]

1,4-Diazoniabicyclo[2.2.2]octane tetraquatetrachlorolanthanate(III) chloride

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Crystal data

(C₆H₁₄N₂)[LaCl₄(H₂O)₄]Cl

M_r = 502.42

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

a = 7.5005 (3) Å

b = 9.1759 (4) Å

c = 25.0388 (10) Å

V = 1723.27 (12) Å³

Z = 4

F(000) = 984

D_x = 1.937 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 5263 reflections

θ = 2.4–32.5°

μ = 3.26 mm⁻¹

T = 293 K

Block, colourless

0.35 × 0.21 × 0.16 mm

Data collection

Bruker SMART 1000 CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 1999)

T_{min} = 0.395, *T_{max}* = 0.624

18229 measured reflections

3279 independent reflections

2971 reflections with *I* > 2σ(*I*)

R_{int} = 0.020

θ_{max} = 32.5°, θ_{min} = 2.4°

h = -11→6

k = -13→13

l = -37→37

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.020

wR(*F*²) = 0.044

S = 1.14

3279 reflections

101 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: difmap (O-H) and geom
(others)

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0199*P*)² + 0.3724*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.58 e Å⁻³

Δρ_{min} = -0.43 e Å⁻³

Extinction correction: SHELXL97,

*F_c** = *kF_c*[1 + 0.001 × *F_c*²λ³/sin(2θ)]^{-1/4}

Extinction coefficient: 0.00056 (9)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
La1	0.590349 (16)	0.2500	0.617775 (4)	0.01947 (4)	
O1	0.4503 (3)	0.2500	0.52432 (6)	0.0339 (4)	
H1	0.4507	0.1732	0.5065	0.041*	
O2	0.2541 (2)	0.2500	0.62157 (7)	0.0391 (4)	
H2	0.1698	0.2500	0.5993	0.047*	
H3	0.1848	0.2500	0.6468	0.047*	
O3	0.50337 (19)	0.09026 (12)	0.69689 (4)	0.0369 (3)	
H4	0.5053	0.0001	0.7008	0.044*	
H5	0.4731	0.1284	0.7299	0.044*	
Cl1	0.90617 (9)	0.2500	0.69439 (2)	0.03609 (13)	
Cl2	0.90460 (11)	0.2211 (2)	0.55605 (3)	0.0453 (6)	0.50
Cl3	0.55658 (6)	-0.04244 (4)	0.580485 (16)	0.03060 (8)	
C1	0.1477 (2)	0.61758 (19)	0.65549 (8)	0.0352 (4)	
H1A	0.1859	0.6177	0.6925	0.042*	
H1B	0.1944	0.5306	0.6385	0.042*	
C2	-0.0537 (2)	0.6177 (2)	0.65261 (9)	0.0390 (4)	
H2A	-0.0946	0.5310	0.6342	0.047*	
H2B	-0.1032	0.6169	0.6884	0.047*	
C3	0.1595 (3)	0.7500	0.57038 (10)	0.0414 (6)	
H3A	0.2059	0.6643	0.5525	0.050*	
C4	-0.0427 (4)	0.7500	0.56802 (10)	0.0430 (7)	
H4A	-0.0842	0.8357	0.5490	0.052*	
N1	-0.1154 (2)	0.7500	0.62359 (7)	0.0251 (4)	
H6	-0.2367	0.7500	0.6222	0.030*	
N2	0.2166 (3)	0.7500	0.62779 (7)	0.0291 (4)	
H7	0.3378	0.7500	0.6293	0.035*	
Cl4	0.54768 (8)	0.7500	0.70889 (2)	0.02892 (11)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
La1	0.01944 (6)	0.02101 (5)	0.01796 (6)	0.000	0.00004 (4)	0.000
O1	0.0509 (11)	0.0277 (7)	0.0230 (7)	0.000	-0.0084 (7)	0.000
O2	0.0214 (8)	0.0584 (11)	0.0373 (9)	0.000	-0.0009 (7)	0.000
O3	0.0609 (9)	0.0255 (5)	0.0241 (5)	-0.0054 (6)	0.0066 (6)	-0.0002 (4)
Cl1	0.0366 (3)	0.0475 (3)	0.0242 (2)	0.000	-0.0057 (2)	0.000
Cl2	0.0253 (3)	0.0869 (18)	0.0236 (3)	-0.0029 (5)	0.0034 (3)	-0.0010 (4)
Cl3	0.0393 (2)	0.02462 (15)	0.02786 (17)	0.00411 (14)	-0.00325 (15)	-0.00412 (13)
C1	0.0304 (8)	0.0320 (8)	0.0433 (10)	0.0059 (7)	-0.0021 (7)	0.0040 (7)
C2	0.0310 (9)	0.0329 (8)	0.0532 (11)	-0.0023 (7)	-0.0025 (8)	0.0125 (8)

C3	0.0277 (12)	0.0739 (19)	0.0226 (11)	0.000	0.0010 (9)	0.000
C4	0.0271 (12)	0.077 (2)	0.0248 (11)	0.000	-0.0022 (9)	0.000
N1	0.0182 (8)	0.0301 (9)	0.0270 (9)	0.000	-0.0015 (7)	0.000
N2	0.0188 (8)	0.0396 (10)	0.0290 (9)	0.000	-0.0003 (7)	0.000
Cl4	0.0298 (3)	0.0300 (2)	0.0269 (2)	0.000	-0.0018 (2)	0.000

Geometric parameters (Å, °)

La1—O2	2.5240 (18)	C1—C2	1.512 (2)
La1—O3 ⁱ	2.5492 (11)	C1—H1A	0.9700
La1—O3	2.5492 (11)	C1—H1B	0.9700
La1—O1	2.5650 (16)	C2—N1	1.489 (2)
La1—Cl2	2.8310 (8)	C2—H2A	0.9700
La1—Cl2 ⁱ	2.8310 (8)	C2—H2B	0.9700
La1—Cl3	2.8524 (4)	C3—N2	1.500 (3)
La1—Cl3 ⁱ	2.8524 (4)	C3—C4	1.518 (4)
La1—Cl1	3.0482 (6)	C3—H3A	0.9700
O1—H1	0.8335	C4—N1	1.494 (3)
O2—H2	0.8433	C4—H4A	0.9700
O2—H3	0.8172	N1—C2 ⁱⁱ	1.489 (2)
O3—H4	0.8330	N1—H6	0.9100
O3—H5	0.9262	N2—C1 ⁱⁱ	1.491 (2)
Cl2—Cl2 ⁱ	0.530 (4)	N2—H7	0.9100
C1—N2	1.491 (2)		
O2—La1—O3 ⁱ	73.44 (5)	La1—O2—H2	136.4
O2—La1—O3	73.44 (5)	La1—O2—H3	131.6
O3 ⁱ —La1—O3	70.20 (5)	H2—O2—H3	92.0
O2—La1—O1	67.98 (6)	La1—O3—H4	131.2
O3 ⁱ —La1—O1	127.16 (4)	La1—O3—H5	122.6
O3—La1—O1	127.16 (4)	H4—O3—H5	105.9
O2—La1—Cl2	148.49 (4)	Cl2 ⁱ —Cl2—La1	84.63 (4)
O3 ⁱ —La1—Cl2	133.72 (4)	N2—C1—C2	108.86 (14)
O3—La1—Cl2	125.70 (5)	N2—C1—H1A	109.9
O1—La1—Cl2	80.97 (5)	C2—C1—H1A	109.9
O2—La1—Cl2 ⁱ	148.49 (4)	N2—C1—H1B	109.9
O3 ⁱ —La1—Cl2 ⁱ	125.70 (5)	C2—C1—H1B	109.9
O3—La1—Cl2 ⁱ	133.72 (4)	H1A—C1—H1B	108.3
O1—La1—Cl2 ⁱ	80.97 (5)	N1—C2—C1	109.54 (15)
Cl2—La1—Cl2 ⁱ	10.73 (8)	N1—C2—H2A	109.8
O2—La1—Cl3	85.617 (16)	C1—C2—H2A	109.8
O3 ⁱ —La1—Cl3	140.58 (3)	N1—C2—H2B	109.8
O3—La1—Cl3	71.99 (3)	C1—C2—H2B	109.8
O1—La1—Cl3	70.429 (9)	H2A—C2—H2B	108.2
Cl2—La1—Cl3	78.89 (4)	N2—C3—C4	108.8 (2)
Cl2 ⁱ —La1—Cl3	89.04 (4)	N2—C3—H3A	109.9
O2—La1—Cl3 ⁱ	85.617 (16)	C4—C3—H3A	109.9
O3 ⁱ —La1—Cl3 ⁱ	71.99 (3)	N1—C4—C3	109.2 (2)

O3—La1—Cl3 ⁱ	140.58 (3)	N1—C4—H4A	109.8
O1—La1—Cl3 ⁱ	70.429 (9)	C3—C4—H4A	109.8
Cl2—La1—Cl3 ⁱ	89.04 (4)	C2 ⁱⁱ —N1—C2	109.3 (2)
Cl2 ⁱ —La1—Cl3 ⁱ	78.89 (4)	C2 ⁱⁱ —N1—C4	109.94 (13)
Cl3—La1—Cl3 ⁱ	140.349 (16)	C2—N1—C4	109.94 (13)
O2—La1—Cl1	138.84 (4)	C2 ⁱⁱ —N1—H6	109.2
O3 ⁱ —La1—Cl1	73.13 (3)	C2—N1—H6	109.2
O3—La1—Cl1	73.13 (3)	C4—N1—H6	109.2
O1—La1—Cl1	153.18 (4)	C1 ⁱⁱ —N2—C1	109.12 (19)
Cl2—La1—Cl1	72.33 (2)	C1 ⁱⁱ —N2—C3	110.29 (13)
Cl2 ⁱ —La1—Cl1	72.33 (2)	C1—N2—C3	110.29 (13)
Cl3—La1—Cl1	105.963 (9)	C1 ⁱⁱ —N2—H7	109.0
Cl3 ⁱ —La1—Cl1	105.963 (9)	C1—N2—H7	109.0
La1—O1—H1	119.0	C3—N2—H7	109.0

Symmetry codes: (i) $x, -y+1/2, z$; (ii) $x, -y+3/2, z$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots Cl3 ⁱⁱⁱ	0.83	2.49	3.2429 (14)	151
O2—H2 \cdots Cl2 ^{iv}	0.84	2.28	3.1035 (19)	166
O2—H2 \cdots Cl2 ^v	0.84	2.28	3.1035 (19)	166
O2—H3 \cdots Cl1 ^{iv}	0.82	2.41	3.1834 (19)	159
O3—H4 \cdots Cl4 ^{vi}	0.83	2.33	3.1542 (12)	173
O3—H5 \cdots Cl1 ^{vii}	0.93	2.26	3.1765 (12)	173
N1—H6 \cdots Cl3 ^v	0.91	2.67	3.2930 (15)	127
N1—H6 \cdots Cl3 ^{viii}	0.91	2.67	3.2930 (15)	127
N1—H6 \cdots Cl4 ^{iv}	0.91	2.71	3.3086 (19)	125
N2—H7 \cdots Cl4	0.91	2.54	3.208 (2)	131
N2—H7 \cdots Cl3 ^{ix}	0.91	2.80	3.3962 (17)	125
N2—H7 \cdots Cl3 ⁱ	0.91	2.80	3.3962 (17)	125

Symmetry codes: (i) $x, -y+1/2, z$; (iii) $-x+1, -y, -z+1$; (iv) $x-1, y, z$; (v) $x-1, -y+1/2, z$; (vi) $x, y-1, z$; (vii) $x-1/2, y, -z+3/2$; (viii) $x-1, y+1, z$; (ix) $x, y+1, z$.