

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Hydroxonium triaquabis(biuret- κ^2 O,O')-dichloridolanthanum(III) dichloride dihydrate

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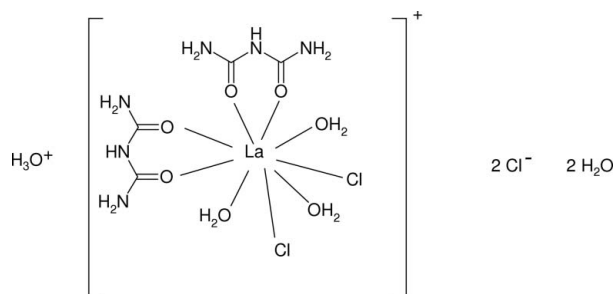
Received 10 April 2008; accepted 15 April 2008

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{N}-\text{C}) = 0.003$ Å; R factor = 0.017; wR factor = 0.042; data-to-parameter ratio = 31.3.

In the title compound, $(\text{H}_3\text{O})[\text{LaCl}_2(\text{C}_2\text{H}_5\text{N}_3\text{O}_2)_2(\text{H}_2\text{O})_3]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$, the La atom is bonded to seven O atoms (arising from two O,O' -bidentate biuret molecules and three water molecules) and two chloride ions in an irregular arrangement. A network of $\text{N}-\text{H} \cdots \text{O}$, $\text{N}-\text{H} \cdots \text{Cl}$, $\text{O}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{Cl}$ hydrogen bonds helps to establish the packing, leading to a three-dimensional network. The La atom, one Cl atom and four O atoms lie on a crystallographic mirror plane.

Related literature

For related structures, see: Carugo *et al.* (1992); Rogers *et al.* (1993); Su *et al.* (2006); Haddad (1987, 1988); Harrison (2008*a,b*). For related literature, see: Brese & O'Keeffe (1991).



Experimental

Crystal data

 $(\text{H}_3\text{O})[\text{LaCl}_2(\text{C}_2\text{H}_5\text{N}_3\text{O}_2)_2(\text{H}_2\text{O})_3] \cdot 2\text{H}_2\text{O}$
 $M_r = 596.00$

 Orthorhombic, $Cmc2_1$
 $a = 17.6252$ (7) Å

 $b = 6.8868$ (3) Å

 $c = 17.0447$ (7) Å

 $V = 2068.91$ (15) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 2.63$ mm⁻¹
 $T = 293$ (2) K

 $0.30 \times 0.23 \times 0.17$ mm

Data collection

 Bruker SMART1000 CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 1999)
 $T_{\min} = 0.486$, $T_{\max} = 0.636$

 11912 measured reflections
 3793 independent reflections
 3716 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.016$
 $wR(F^2) = 0.042$
 $S = 1.09$

3793 reflections

121 parameters

1 restraint

 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.70$ e Å⁻³
 $\Delta\rho_{\min} = -0.67$ e Å⁻³
 Absolute structure: Flack (1983),
 1805 Friedel pairs
 Flack parameter: 0.001 (9)

Table 1

Selected bond lengths (Å).

La1—O3	2.503 (2)	La1—O4	2.542 (2)
La1—O2	2.5313 (13)	La1—O5	2.562 (2)
La1—O1	2.5318 (14)	La1—Cl1	2.9606 (4)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
N1—H1 ⁱ ···Cl2 ⁱ	0.86	2.94	3.643 (3)	141
N1—H2 ⁱⁱ ···Cl1 ⁱⁱ	0.86	2.83	3.574 (3)	145
N2—H3 ⁱⁱ ···Cl1 ⁱⁱ	0.86	2.28	3.1399 (14)	173
N3—H4 ⁱⁱ ···O7 ⁱⁱ	0.86	2.12	2.927 (3)	156
N3—H5 ⁱⁱ ···Cl2	0.86	2.75	3.3835 (18)	132
O3—H6 ⁱ ···Cl2 ⁱ	0.85	2.28	3.1181 (16)	168
O4—H7 ⁱⁱⁱ ···Cl2 ⁱⁱⁱ	0.84	2.32	3.1396 (17)	164
O5—H8 ^{iv} ···Cl1 ^{iv}	0.75	2.44	3.1566 (17)	160
O6—H9 ⁱⁱⁱ ···O2 ⁱⁱⁱ	0.88	2.23	3.044 (3)	153
O6—H10 ^v ···O7	0.86	2.35	2.941 (3)	126
O6—H10 ^v ···O7 ^v	0.86	2.35	2.941 (3)	126
O7—H11 ^{vi} ···Cl2 ^{vi}	0.86	2.48	3.264 (3)	151
O7—H12 ^{vii} ···O1 ^{vii}	0.84	2.09	2.922 (2)	168

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

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2113).

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supporting information

Acta Cryst. (2008). E64, m681–m682 [doi:10.1107/S1600536808010349]

Hydroxonium triaquabis(biuret- κ^2O,O')dichloridolanthanum(III) dichloride dihydrate

William T. A. Harrison

S1. Comment

No complexes of lanthanum(III) with biuret (biur), $H_2N-CO-NH-CO-NH_2$ (or $C_2H_5N_3O_2$) have been structurally characterized. The structures of two samarium-biuret complexes, $Sm(biur)_4(NO_3)_3$ (Haddad, 1987) and $Sm(biur)_4(ClO_4)_3$ (Haddad, 1988) have been described. In both cases, an SmO_8 square antiprismatic coordination arises for the metal ion. Based on X-ray photographs, it was suggested that all the $Ln(biur)_4(NO_3)_3$ and $Ln(biur)_4(ClO_4)_3$ compounds are isostructural with their samarium prototypes. In this paper, we describe the synthesis and structure of the title compound, (I), in which three different ligands are bonded to the trivalent cation.

Compound (I) is an ionic salt containing a new $[La(biur)_2(H_2O)_5Cl_2]^+$ complex ion. The complete cation is generated by crystallographic mirror symmetry, with La and the three water O atoms lying on the reflecting plane. A hydroxonium cation (with its O6 atom with site symmetry *m*), an uncoordinated chloride ion (Cl2) and an uncoordinated water molecule (O7) complete the structure (Fig. 1) of (I).

The resulting LaO_7Cl_2 polyhedral geometry in (I) (Table 1) can only be described as irregular. The Brese & O'Keeffe (1991) bond-valence sum for La1 in (I) of 3.29 is significantly larger than the expected value of 3.00. A local LaO_7Cl_2 grouping has been seen in various other compounds, including $[LaCl_2(H_2O)(C_{12}H_{24}O_6)]^+.Cl^-$ (Rogers *et al.*, 1993) and $[La(H_2O)_4Cl(C_3H_7O_3)]^{2+}.2Cl^-.H_2O$ (Su *et al.*, 2006), but otherwise these phases have no similarity to (I).

The *O,O'*-bidentate coordination of the biuret molecule to the lanthanum ion in (I) results in a six-membered chelate ring that is non-planar. As noted previously (Carugo *et al.*, 1992), the biuret molecule can be regarded as two planar amide fragments linked by the NH bridge. Here, the dihedral angle between the N1/C1/O1/N2 and N2/C2/O2/N3 units is $5.06(10)^\circ$. The lanthanum cation deviates from the N1/C1/O1/N2 and N2/C2/O2/N3 mean planes by $0.894(4)\text{Å}$ and $0.606(4)\text{Å}$, respectively.

The component species in (I) are linked by a dense array of $N-H\cdots O$, $N-H\cdots Cl$, $O-H\cdots Cl$ and $O-H\cdots O$ hydrogen bonds (Table 2) resulting in a three-dimensional network. Of note are the [001] chains resulting from the $O-H\cdots O$ hydrogen bonds involving the complex cation, H_3O_6 and H_2O_7 (Fig. 2).

The structure of (I) is different to those of the recently reported (Harrison, 2008*a,b*) $M(biur)_2(H_2O)_4.Cl_3$ ($M = Gd, Y$) phases, perhaps because the larger La^{3+} cation can accommodate nine atoms in its coordination sphere.

S2. Experimental

0.1 *M* Aqueous solutions of $LaCl_3$ (10 ml) and biuret (10 ml) were mixed and a small quantity of dilute hydrochloric acid was added, to result in a colourless solution. Colourless blocks of (I) grew over several days as the water slowly evaporated.

S3. Refinement

The N-bound hydrogen atoms were geometrically placed ($N-H = 0.88 \text{ \AA}$) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(N)$. The water and hydroxonium H atoms were located in difference maps and refined as riding in their as-found relative positions with $U_{iso}(H) = 1.2U_{eq}(O)$. Although a plausible hydrogen bonding scheme results, some of the peaks were barely above the noise level of the data, and thus the positions of the O-bonded H atoms should be regarded as less certain.

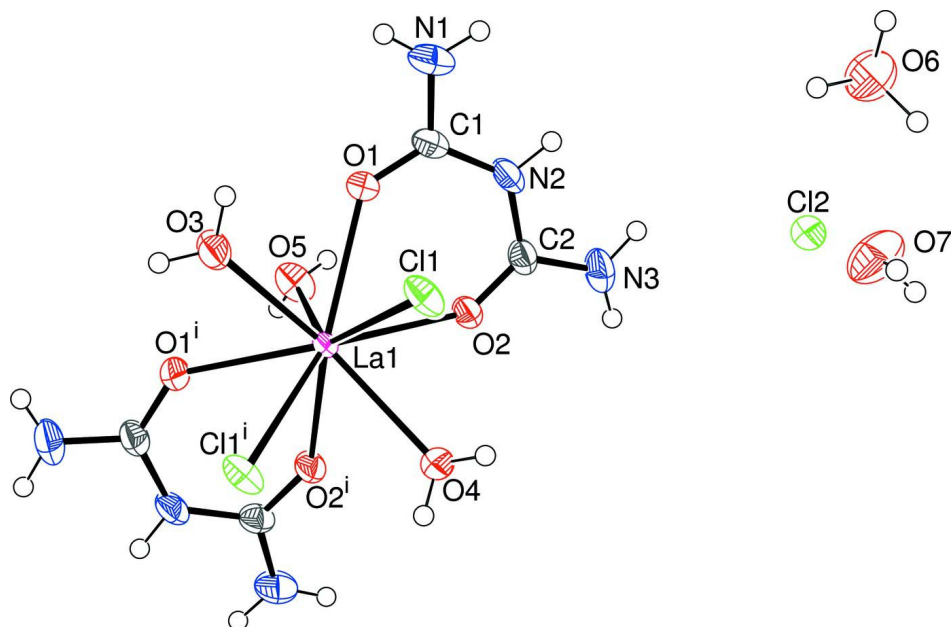


Figure 1

View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). Symmetry code: (i) $-x, y, z$.

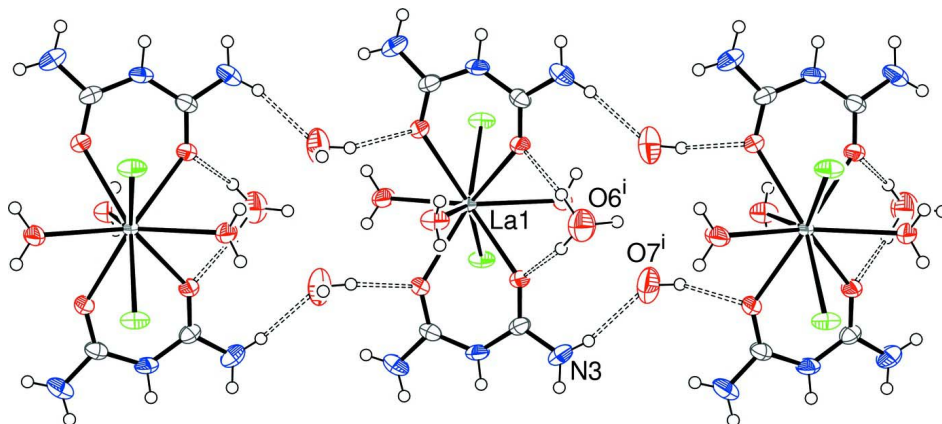


Figure 2

Fragment of the packing for (I) displaying the hydrogen bonds (shows as double dashed lines) leading to chains arising from the complex cation, the hydroxonium ion and the uncoordinated water molecule. Symmetry code: (i) $1/2 - x, 1/2 + y, z$.

Hydroxonium triaquabis(biuret- κ^2O,O')dichloridolanthanum(III) dichloride dihydrate

Crystal data

(H₃O)[LaCl₂(C₂H₅N₃O₂)₂(H₂O)₃]Cl₂·2H₂O
 $M_r = 596.00$
 Orthorhombic, $Cmc2_1$
 Hall symbol: C 2c -2
 $a = 17.6252$ (7) Å
 $b = 6.8868$ (3) Å
 $c = 17.0447$ (7) Å
 $V = 2068.91$ (15) Å³
 $Z = 4$

$F(000) = 1176$
 $D_x = 1.913$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 4771 reflections
 $\theta = 2.3$ – 32.5°
 $\mu = 2.63$ mm⁻¹
 $T = 293$ K
 Block, colourless
 $0.30 \times 0.23 \times 0.17$ mm

Data collection

Bruker SMART1000 CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 1999)
 $T_{\min} = 0.486$, $T_{\max} = 0.636$

11912 measured reflections
 3793 independent reflections
 3716 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\max} = 32.5^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -26 \rightarrow 26$
 $k = -10 \rightarrow 9$
 $l = -24 \rightarrow 25$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.016$
 $wR(F^2) = 0.042$
 $S = 1.09$
 3793 reflections
 121 parameters
 1 restraint
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0242P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.70$ e Å⁻³
 $\Delta\rho_{\min} = -0.67$ e Å⁻³
 Absolute structure: Flack (1983), 1805 Friedel
 pairs
 Absolute structure parameter: 0.001 (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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
La1	0.0000	0.234447 (14)	0.341713 (14)	0.01947 (3)
Cl1	0.10789 (2)	-0.09170 (6)	0.32281 (3)	0.03634 (12)
C1	0.18755 (10)	0.3315 (3)	0.40149 (12)	0.0293 (4)

C2	0.17044 (10)	0.4278 (3)	0.26382 (12)	0.0290 (3)
N1	0.24102 (12)	0.2964 (3)	0.45518 (14)	0.0459 (5)
H1	0.2283	0.2650	0.5022	0.055*
H2	0.2882	0.3052	0.4427	0.055*
N2	0.21433 (7)	0.3826 (2)	0.32868 (11)	0.0344 (4)
H3	0.2628	0.3870	0.3229	0.041*
N3	0.20884 (11)	0.4877 (3)	0.20138 (13)	0.0445 (4)
H4	0.1851	0.5181	0.1591	0.053*
H5	0.2575	0.4963	0.2032	0.053*
O1	0.11900 (8)	0.3202 (3)	0.41736 (8)	0.0333 (3)
O2	0.10047 (7)	0.41314 (19)	0.26393 (8)	0.0298 (3)
O3	0.0000	0.0881 (4)	0.47616 (14)	0.0488 (7)
H6	0.0406	0.0729	0.5026	0.059*
O4	0.0000	0.1452 (4)	0.19698 (13)	0.0384 (5)
H7	0.0377	0.0935	0.1750	0.046*
O5	0.0000	0.5850 (3)	0.39200 (13)	0.0388 (5)
H8	-0.0323	0.6527	0.3843	0.047*
Cl2	0.36579 (3)	0.51610 (15)	0.08766 (5)	0.04627 (14)
O6	0.5000	0.1924 (5)	0.17243 (18)	0.0617 (7)
H9	0.4686	0.1472	0.2081	0.074*
H10	0.5000	0.1363	0.1273	0.074*
O7	0.39583 (14)	-0.0187 (4)	0.06811 (12)	0.0695 (6)
H11	0.3960	-0.1404	0.0560	0.083*
H12	0.3976	0.0476	0.0265	0.083*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
La1	0.01375 (4)	0.02565 (5)	0.01902 (5)	0.000	0.000	0.00102 (8)
Cl1	0.01972 (15)	0.03484 (19)	0.0545 (3)	0.00363 (13)	0.00053 (17)	-0.00126 (18)
C1	0.0219 (8)	0.0309 (9)	0.0351 (9)	-0.0001 (6)	-0.0063 (7)	-0.0041 (7)
C2	0.0240 (7)	0.0303 (8)	0.0325 (9)	-0.0042 (6)	0.0059 (7)	-0.0006 (7)
N1	0.0305 (9)	0.0593 (11)	0.0479 (11)	0.0024 (8)	-0.0163 (9)	0.0009 (10)
N2	0.0164 (5)	0.0455 (7)	0.0412 (11)	-0.0021 (5)	0.0022 (6)	0.0039 (7)
N3	0.0322 (9)	0.0584 (11)	0.0430 (10)	-0.0067 (8)	0.0141 (8)	0.0071 (9)
O1	0.0228 (6)	0.0510 (8)	0.0260 (6)	-0.0028 (6)	-0.0025 (5)	-0.0012 (6)
O2	0.0211 (6)	0.0395 (7)	0.0288 (6)	-0.0032 (5)	0.0006 (5)	0.0062 (6)
O3	0.0248 (10)	0.086 (2)	0.0352 (13)	0.000	0.000	0.0282 (12)
O4	0.0265 (10)	0.0609 (15)	0.0279 (10)	0.000	0.000	-0.0113 (10)
O5	0.0301 (10)	0.0299 (9)	0.0564 (14)	0.000	0.000	-0.0004 (9)
Cl2	0.03018 (19)	0.0726 (4)	0.0360 (2)	0.0037 (3)	-0.0008 (3)	-0.0124 (2)
O6	0.076 (2)	0.0640 (16)	0.0453 (16)	0.000	0.000	0.0068 (14)
O7	0.0972 (16)	0.0730 (12)	0.0384 (10)	0.0050 (13)	-0.0051 (10)	0.0115 (10)

Geometric parameters (Å, °)

La1—O3	2.503 (2)	C2—N2	1.385 (3)
La1—O2	2.5313 (13)	N1—H1	0.8600

La1—O2 ⁱ	2.5313 (12)	N1—H2	0.8600
La1—O1	2.5318 (14)	N2—H3	0.8600
La1—O1 ⁱ	2.5318 (14)	N3—H4	0.8600
La1—O4	2.542 (2)	N3—H5	0.8600
La1—O5	2.562 (2)	O3—H6	0.8522
La1—Cl1 ⁱ	2.9606 (4)	O4—H7	0.8418
La1—Cl1	2.9606 (4)	O5—H8	0.7477
C1—O1	1.241 (2)	O6—H9	0.8786
C1—N1	1.336 (3)	O6—H10	0.8615
C1—N2	1.374 (3)	O7—H11	0.8631
C2—O2	1.237 (2)	O7—H12	0.8446
C2—N3	1.327 (3)		
O3—La1—O2	132.46 (4)	O2—La1—Cl1	82.10 (3)
O3—La1—O2 ⁱ	132.46 (4)	O2 ⁱ —La1—Cl1	139.57 (4)
O2—La1—O2 ⁱ	88.78 (6)	O1—La1—Cl1	72.56 (4)
O3—La1—O1	68.14 (5)	O1 ⁱ —La1—Cl1	139.86 (4)
O2—La1—O1	64.78 (4)	O4—La1—Cl1	73.20 (5)
O2 ⁱ —La1—O1	137.12 (5)	O5—La1—Cl1	138.709 (15)
O3—La1—O1 ⁱ	68.14 (5)	Cl1 ⁱ —La1—Cl1	79.930 (17)
O2—La1—O1 ⁱ	137.12 (5)	O1—C1—N1	121.8 (2)
O2 ⁱ —La1—O1 ⁱ	64.78 (4)	O1—C1—N2	123.21 (16)
O1—La1—O1 ⁱ	111.87 (7)	N1—C1—N2	115.01 (18)
O3—La1—O4	142.27 (9)	O2—C2—N3	122.33 (19)
O2—La1—O4	67.01 (5)	O2—C2—N2	122.51 (17)
O2 ⁱ —La1—O4	67.01 (5)	N3—C2—N2	115.16 (17)
O1—La1—O4	123.41 (3)	C1—N1—H1	120.0
O1 ⁱ —La1—O4	123.41 (3)	C1—N1—H2	120.0
O3—La1—O5	94.19 (9)	H1—N1—H2	120.0
O2—La1—O5	73.56 (5)	C1—N2—C2	125.93 (14)
O2 ⁱ —La1—O5	73.56 (5)	C1—N2—H3	117.0
O1—La1—O5	67.03 (4)	C2—N2—H3	117.0
O1 ⁱ —La1—O5	67.03 (4)	C2—N3—H4	120.0
O4—La1—O5	123.54 (8)	C2—N3—H5	120.0
O3—La1—Cl1 ⁱ	78.13 (5)	H4—N3—H5	120.0
O2—La1—Cl1 ⁱ	139.57 (4)	C1—O1—La1	135.26 (12)
O2 ⁱ —La1—Cl1 ⁱ	82.10 (3)	C2—O2—La1	137.53 (12)
O1—La1—Cl1 ⁱ	139.86 (4)	La1—O3—H6	122.3
O1 ⁱ —La1—Cl1 ⁱ	72.56 (4)	La1—O4—H7	122.3
O4—La1—Cl1 ⁱ	73.20 (5)	La1—O5—H8	121.9
O5—La1—Cl1 ⁱ	138.709 (15)	H9—O6—H10	117.3
O3—La1—Cl1	78.13 (5)	H11—O7—H12	108.9
O1—C1—N2—C2	-0.1 (3)	Cl1 ⁱ —La1—O1—C1	103.6 (2)
N1—C1—N2—C2	-179.32 (19)	Cl1—La1—O1—C1	54.6 (2)
O2—C2—N2—C1	-5.1 (3)	N3—C2—O2—La1	159.25 (15)
N3—C2—N2—C1	174.93 (19)	N2—C2—O2—La1	-20.7 (3)
N1—C1—O1—La1	-149.81 (17)	O3—La1—O2—C2	21.4 (2)

N2—C1—O1—La1	31.0 (3)	O2 ⁱ —La1—O2—C2	175.02 (16)
O3—La1—O1—C1	138.5 (2)	O1—La1—O2—C2	29.91 (18)
O2—La1—O1—C1	-34.7 (2)	O1 ⁱ —La1—O2—C2	125.52 (18)
O2 ⁱ —La1—O1—C1	-91.9 (2)	O4—La1—O2—C2	-119.5 (2)
O1 ⁱ —La1—O1—C1	-167.88 (17)	O5—La1—O2—C2	101.81 (19)
O4—La1—O1—C1	-0.6 (2)	Cl1 ⁱ —La1—O2—C2	-108.69 (18)
O5—La1—O1—C1	-116.7 (2)	Cl1—La1—O2—C2	-44.48 (18)

Symmetry code: (i) $-x, y, z$.

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...Cl2 ⁱⁱ	0.86	2.94	3.643 (3)	141
N1—H2...Cl1 ⁱⁱⁱ	0.86	2.83	3.574 (3)	145
N2—H3...Cl1 ⁱⁱⁱ	0.86	2.28	3.1399 (14)	173
N3—H4...O7 ⁱⁱⁱ	0.86	2.12	2.927 (3)	156
N3—H5...Cl2	0.86	2.75	3.3835 (18)	132
O3—H6...Cl2 ⁱⁱ	0.85	2.28	3.1181 (16)	168
O4—H7...Cl2 ^{iv}	0.84	2.32	3.1396 (17)	164
O5—H8...Cl1 ^v	0.75	2.44	3.1566 (17)	160
O6—H9...O2 ^{iv}	0.88	2.23	3.044 (3)	153
O6—H10...O7	0.86	2.35	2.941 (3)	126
O6—H10...O7 ^{vi}	0.86	2.35	2.941 (3)	126
O7—H11...Cl2 ^{vii}	0.86	2.48	3.264 (3)	151
O7—H12...O1 ^{viii}	0.84	2.09	2.922 (2)	168

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