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K₂LaCl₅Christian M. Schurz,^a Thomas Schleid^a and Gerd Meyer^{b*}

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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{La}-\text{Cl}) = 0.003$ Å; R factor = 0.059; wR factor = 0.142; data-to-parameter ratio = 37.5.

The ternary title compound, dipotassium lanthanum pentachloride, K_2LaCl_5 , is isotypic with Y_2HfS_5 and various ternary rare-earth metal(III) halides with the general formula A_2MX_5 ($A = \text{NH}_4, \text{In}^I, \text{Na}-\text{Cs}$; $M = \text{La}-\text{Dy}$; $X = \text{Cl}-\text{I}$). The La^{3+} cations and three of the four symmetry-independent chloride anions are located on a crystallographic mirror plane. The La^{3+} cations are surrounded by seven chloride anions, each in the shape of a monocapped trigonal prism, whereas the coordination spheres of the K^+ cations exhibit one more cap. Three of the four independent chloride anions reside in a fivefold cationic coordination, leading to distorted square pyramids. The fourth chloride anion has only four cationic neighbours, forming no specific polyhedron.

Related literature

For the U_3Ch_5 -type structure ($\text{Ch} = \text{S}$ and Se) and its relationship to Y_2HfS_5 , see: Moseley *et al.* (1972); Potel *et al.* (1972); Jeitschko & Donohue (1975). For the low-temperature phase of Yb_5Sb_3 , see: Brunton & Steinfink (1971). For the series of the ternary rare-earth metal(III) halides with $A = \text{NH}_4, \text{In}^I, \text{Na}-\text{Cs}$; $M = \text{La}-\text{Dy}$; $X = \text{Cl}-\text{I}$, see: Meyer & Hüttel (1983); Meyer *et al.* (1985); Wickleder & Meyer (1995).

Experimental

Crystal data

K_2LaCl_5	$V = 905.35$ (10) Å ³
$M_r = 394.36$	$Z = 4$
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation
$a = 12.7402$ (8) Å	$\mu = 7.02$ mm ⁻¹
$b = 8.8635$ (6) Å	$T = 293$ K
$c = 8.0174$ (5) Å	$0.33 \times 0.28 \times 0.24$ mm

Data collection

Stoe IPDS-I diffractometer	12421 measured reflections
Absorption correction: numerical (<i>X-SHAPE</i> ; Stoe & Cie, 1999)	1650 independent reflections
$T_{\min} = 0.106, T_{\max} = 0.185$	872 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.139$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$	44 parameters
$wR(F^2) = 0.142$	$\Delta\rho_{\max} = 1.58$ e Å ⁻³
$S = 0.90$	$\Delta\rho_{\min} = -2.64$ e Å ⁻³
1650 reflections	

Table 1

Selected bond lengths (Å).

K—Cl ⁱ	3.160 (3)	La—Cl ^{3v}	2.812 (3)
K—Cl ²	3.177 (3)	La—Cl ¹¹	2.833 (3)
K—Cl ¹ⁱⁱ	3.206 (3)	La—Cl ^{2vi}	2.845 (3)
K—Cl ²ⁱⁱⁱ	3.234 (3)	La—Cl ⁴	2.858 (2)
K—Cl ^{3iv}	3.272 (4)	La—Cl ^{4vii}	2.858 (2)
K—Cl ⁴	3.304 (3)	La—Cl ^{4viii}	2.895 (2)
K—Cl ⁴ⁱⁱⁱ	3.327 (3)	La—Cl ^{4ix}	2.895 (2)
K—Cl ³	3.351 (4)		

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

Data collection: *DIF4* (Stoe & Cie, 1992); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5401).

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supporting information

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K₂LaCl₅

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S1. Comment

The ternary rare-earth metal(III) halide K₂LaCl₅ (Fig. 1) belongs to the A_2MX_5 series ($A = \text{NH}_4, \text{In}, \text{Na} - \text{Cs}$; $M = \text{La} - \text{Dy}$; $X = \text{Cl} - \text{I}$) (Meyer & Hüttl, 1983; Meyer *et al.*, 1985; Wickleder & Meyer 1995). It can be described as ordered structural variety of U_3Ch_5 ($\text{Ch} = \text{S}$ and Se) or the low-temperature phase of Yb_5Sb_3 , respectively, as anti-isotypical arrangement. While the K^+ cations have eight contacts to Cl^- anions (Fig. 2), the La^{3+} cations are surrounded by only seven of them. In both cases distorted mono- or bicapped trigonal prisms $[\text{LaCl}_7]^{4+}$ or $[\text{KCl}_8]^{7-}$ originate. For the lanthanum bearing ones they are linked *via* common edges and form chains, which run along $[010]$ (Fig. 3). Together with the chloride anions (Cl1^-), (Cl2^-) and (Cl3^-), La^{3+} occupies the $4c$ position and shows the site symmetry m , while the (Cl4^-) anion and the K^+ cation are located at the $8d$ position with the site symmetry 1.

S2. Experimental

Colourless, transparent, brick-shaped single crystals of K₂LaCl₅ were obtained as by-product from the reaction of potassium azide (KN₃), lanthanum (La), the corresponding sesquioxide (La₂O₃) and trichloride (LaCl₃) in the presence of KCl as flux with the purpose to synthesize K₂La₄ONCl₉. The mixture was transferred into a torch-sealed, evacuated, fused silica vessel, heated at 1123 K for seven days, followed by cooling to room temperature within 24 h.

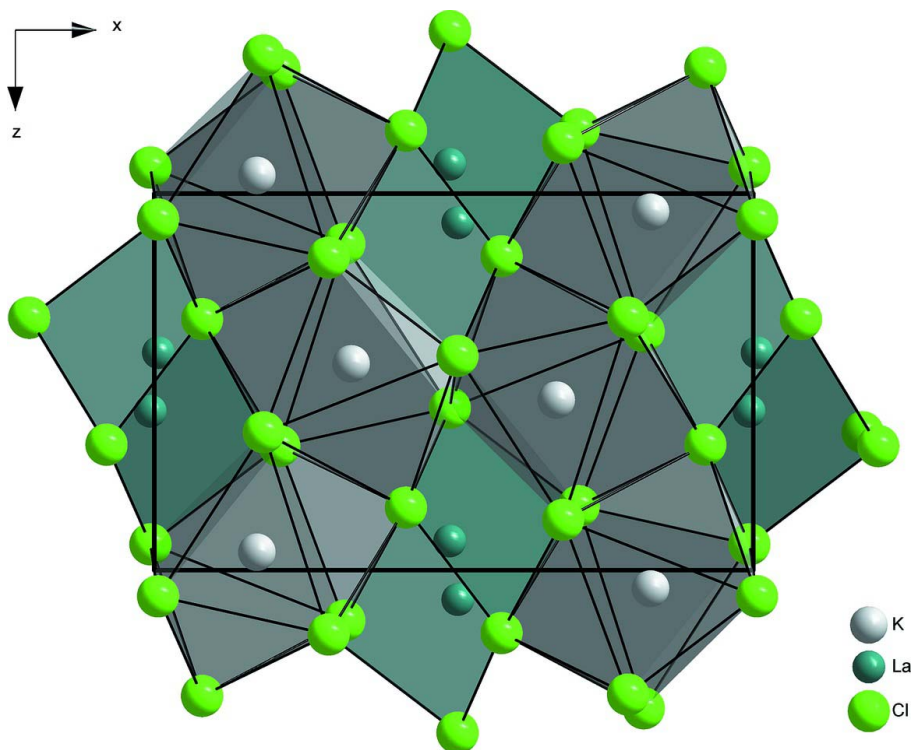


Figure 1
Crystal structure of K_2LaCl_5 as viewed along $[010]$.

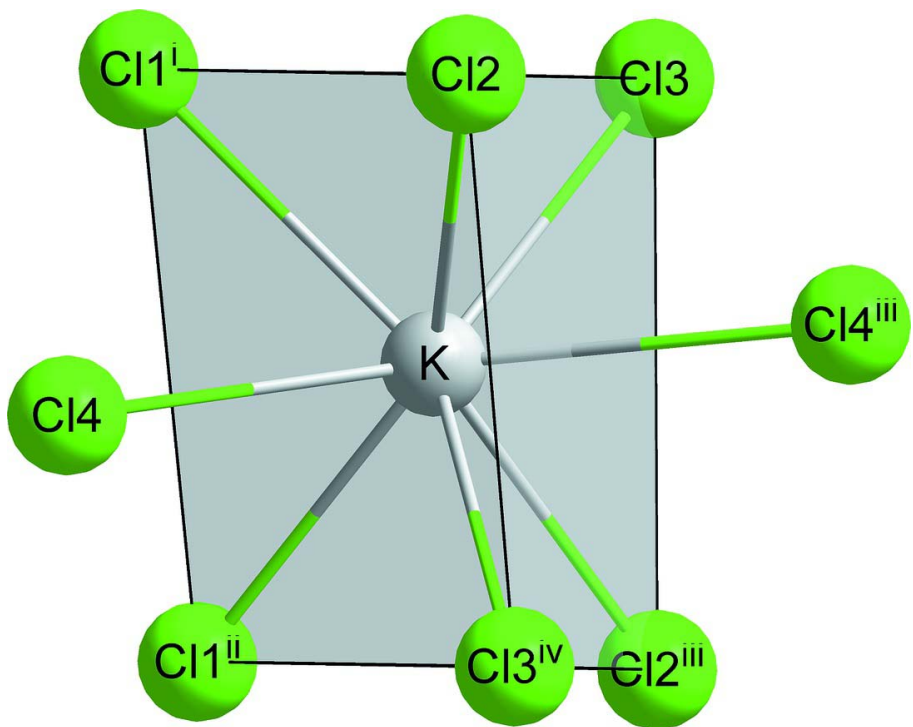


Figure 2
Coordination sphere of the K^+ cations with the shape of a bicapped trigonal prism. [Symmetry codes: (i) $-x+1/2, -y+1, z-1/2$; (ii) $x+1/2, y, -z+3/2$; (iii) $-x+3/2, -y+1, z+1/2$; (iv) $-x+3/2, -y+1, z-1/2$.]

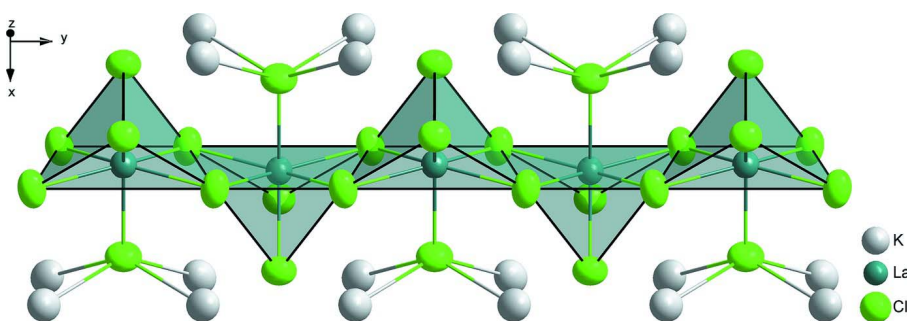


Figure 3

View at the chain formed by edge-sharing monocapped trigonal prisms $[\text{LaCl}_7]^{4-}$ with its contacts to the K^+ cations. Displacement ellipsoids are drawn at 90% probability level.

dipotassium lanthanum pentachloride

Crystal data

K_2LaCl_5

$M_r = 394.36$

Orthorhombic, $Pnma$

Hall symbol: $-P\ 2ac\ 2n$

$a = 12.7402\ (8)\ \text{\AA}$

$b = 8.8635\ (6)\ \text{\AA}$

$c = 8.0174\ (5)\ \text{\AA}$

$V = 905.35\ (10)\ \text{\AA}^3$

$Z = 4$

$F(000) = 720$

$D_x = 2.893\ \text{Mg m}^{-3}$

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

$\theta = 3.4\text{--}33.0^\circ$

$\mu = 7.02\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Bricks, colourless

$0.33 \times 0.28 \times 0.24\ \text{mm}$

Data collection

Stoe IPDS-I

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

imaging plate detector system scans

Absorption correction: numerical

($X\text{-SHAPE}$; Stoe & Cie, 1999)

$T_{\min} = 0.106$, $T_{\max} = 0.185$

12421 measured reflections

1650 independent reflections

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

$R_{\text{int}} = 0.139$

$\theta_{\max} = 33.0^\circ$, $\theta_{\min} = 3.4^\circ$

$h = -19 \rightarrow 19$

$k = -11 \rightarrow 11$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.059$

$wR(F^2) = 0.142$

$S = 0.90$

1650 reflections

44 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2]$

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

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 1.58\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -2.64\ \text{e \AA}^{-3}$

Extinction correction: $SHELXL97$ (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0094 (12)

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	$U_{\text{iso}}^*/U_{\text{eq}}$
K	0.67125 (15)	0.4946 (3)	0.5481 (3)	0.0379 (5)
La	0.50680 (5)	0.2500	0.07776 (8)	0.0248 (2)
Cl1	-0.0065 (2)	0.7500	0.9311 (4)	0.0310 (6)
Cl2	0.7911 (2)	0.2500	0.3299 (4)	0.0333 (7)
Cl3	0.6828 (2)	0.2500	0.8662 (4)	0.0374 (8)
Cl4	0.57990 (17)	0.5441 (3)	0.1663 (3)	0.0342 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K	0.0381 (10)	0.0363 (14)	0.0393 (11)	0.0014 (8)	-0.0014 (7)	-0.0070 (9)
La	0.0286 (3)	0.0234 (4)	0.0222 (3)	0.000	0.0022 (3)	0.000
Cl1	0.0347 (13)	0.0354 (17)	0.0228 (11)	0.000	0.0016 (12)	0.000
Cl2	0.0282 (13)	0.039 (2)	0.0322 (15)	0.000	0.0006 (11)	0.000
Cl3	0.0368 (15)	0.045 (2)	0.0308 (15)	0.000	0.0088 (12)	0.000
Cl4	0.0460 (12)	0.0267 (13)	0.0301 (10)	-0.0030 (9)	-0.0102 (8)	0.0025 (8)

Geometric parameters (\AA , $^\circ$)

K—Cl1 ⁱ	3.160 (3)	La—Cl4 ^x	2.895 (2)
K—Cl2	3.177 (3)	La—K ^{vi}	4.389 (2)
K—Cl1 ⁱⁱ	3.206 (3)	La—K ^{xi}	4.389 (2)
K—Cl2 ⁱⁱⁱ	3.234 (3)	Cl1—La ^{xii}	2.833 (3)
K—Cl3 ^{iv}	3.272 (4)	Cl1—K ^{xii}	3.160 (3)
K—Cl4	3.304 (3)	Cl1—K ^{xiii}	3.160 (3)
K—Cl4 ⁱⁱⁱ	3.327 (3)	Cl1—K ^{xiv}	3.206 (3)
K—Cl3	3.351 (4)	Cl1—K ^{xv}	3.206 (3)
K—K ^v	4.336 (5)	Cl2—La ^{xvi}	2.845 (3)
K—La ^{vi}	4.389 (2)	Cl2—K ^v	3.177 (3)
K—K ^{vi}	4.432 (4)	Cl2—K ^{xvii}	3.234 (3)
K—K ⁱⁱⁱ	4.4838 (18)	Cl2—K ^{iv}	3.234 (3)
La—Cl3 ^{vii}	2.812 (3)	Cl3—La ^{xviii}	2.812 (3)
La—Cl1 ⁱ	2.833 (3)	Cl3—K ⁱⁱⁱ	3.272 (4)
La—Cl2 ^{viii}	2.845 (3)	Cl3—K ^{xix}	3.272 (3)
La—Cl4	2.858 (2)	Cl3—K ^v	3.351 (4)
La—Cl4 ^v	2.858 (2)	Cl4—La ^x	2.895 (2)

La—C14 ^{ix}	2.895 (2)	C14—K ^{iv}	3.327 (3)
C11 ⁱ —K—C12	71.80 (8)	C13 ^{vii} —La—C14	83.68 (6)
C11 ⁱ —K—C11 ⁱⁱ	91.76 (5)	C11 ⁱ —La—C14	75.64 (5)
C12—K—C11 ⁱⁱ	148.21 (10)	C12 ^{viii} —La—C14	104.48 (5)
C11 ⁱ —K—C12 ⁱⁱⁱ	141.81 (10)	C13 ^{vii} —La—C14 ^v	83.68 (6)
C12—K—C12 ⁱⁱⁱ	142.29 (7)	C11 ⁱ —La—C14 ^v	75.64 (5)
C11 ⁱⁱ —K—C12 ⁱⁱⁱ	64.80 (8)	C12 ^{viii} —La—C14 ^v	104.48 (5)
C11 ⁱ —K—C13 ^{iv}	136.03 (10)	C14—La—C14 ^v	131.62 (9)
C12—K—C13 ^{iv}	87.34 (7)	C13 ^{vii} —La—C14 ^{ix}	84.06 (7)
C11 ⁱⁱ —K—C13 ^{iv}	86.34 (8)	C11 ⁱ —La—C14 ^{ix}	132.50 (6)
C12 ⁱⁱⁱ —K—C13 ^{iv}	75.11 (8)	C12 ^{viii} —La—C14 ^{ix}	78.89 (7)
C11 ⁱ —K—C14	65.30 (7)	C14—La—C14 ^{ix}	150.15 (6)
C12—K—C14	75.52 (8)	C14 ^v —La—C14 ^{ix}	73.58 (7)
C11 ⁱⁱ —K—C14	72.88 (8)	C13 ^{vii} —La—C14 ^x	84.06 (7)
C12 ⁱⁱⁱ —K—C14	127.34 (10)	C11 ⁱ —La—C14 ^x	132.50 (6)
C13 ^{iv} —K—C14	72.27 (8)	C12 ^{viii} —La—C14 ^x	78.89 (7)
C11 ⁱ —K—C14 ⁱⁱⁱ	130.30 (10)	C14—La—C14 ^x	73.58 (7)
C12—K—C14 ⁱⁱⁱ	68.18 (8)	C14 ^v —La—C14 ^x	150.15 (6)
C11 ⁱⁱ —K—C14 ⁱⁱⁱ	136.98 (9)	C14 ^{ix} —La—C14 ^x	78.15 (9)
C12 ⁱⁱⁱ —K—C14 ⁱⁱⁱ	74.46 (8)	C13 ^{vii} —La—K ^{vi}	145.53 (4)
C13 ^{iv} —K—C14 ⁱⁱⁱ	69.93 (8)	C11 ⁱ —La—K ^{vi}	46.84 (5)
C14—K—C14 ⁱⁱⁱ	127.83 (9)	C12 ^{viii} —La—K ^{vi}	47.41 (5)
C11 ⁱ —K—C13	79.12 (8)	C14—La—K ^{vi}	61.85 (6)
C12—K—C13	87.50 (8)	C14 ^v —La—K ^{vi}	117.97 (6)
C11 ⁱⁱ —K—C13	116.64 (9)	C14 ^{ix} —La—K ^{vi}	126.15 (5)
C12 ⁱⁱⁱ —K—C13	85.10 (7)	C14 ^x —La—K ^{vi}	86.55 (6)
C13 ^{iv} —K—C13	139.63 (8)	C13 ^{vii} —La—K ^{xi}	145.53 (4)
C14—K—C13	143.76 (10)	C11 ⁱ —La—K ^{xi}	46.84 (5)
C14 ⁱⁱⁱ —K—C13	71.00 (8)	C12 ^{viii} —La—K ^{xi}	47.41 (5)
C11 ⁱ —K—K ^v	46.68 (5)	C14—La—K ^{xi}	117.97 (6)
C12—K—K ^v	46.96 (6)	C14 ^v —La—K ^{xi}	61.85 (6)
C11 ⁱⁱ —K—K ^v	134.91 (5)	C14 ^{ix} —La—K ^{xi}	86.55 (6)
C12 ⁱⁱⁱ —K—K ^v	134.42 (6)	C14 ^x —La—K ^{xi}	126.15 (6)
C13 ^{iv} —K—K ^v	133.77 (6)	K ^{vi} —La—K ^{xi}	62.09 (7)
C14—K—K ^v	97.63 (6)	La ^{xii} —C11—K ^{xii}	107.21 (8)
C14 ⁱⁱⁱ —K—K ^v	84.07 (6)	La ^{xii} —C11—K ^{xiii}	107.21 (8)
C13—K—K ^v	49.68 (5)	K ^{xii} —C11—K ^{xiii}	86.64 (11)
C11 ⁱ —K—La ^{vi}	102.32 (7)	La ^{xii} —C11—K ^{xiv}	93.04 (7)
C12—K—La ^{vi}	167.59 (9)	K ^{xii} —C11—K ^{xiv}	159.73 (10)
C11 ⁱⁱ —K—La ^{vi}	40.13 (5)	K ^{xiii} —C11—K ^{xiv}	88.24 (5)
C12 ⁱⁱⁱ —K—La ^{vi}	40.37 (6)	La ^{xii} —C11—K ^{xv}	93.04 (7)
C13 ^{iv} —K—La ^{vi}	103.97 (7)	K ^{xii} —C11—K ^{xv}	88.24 (5)
C14—K—La ^{vi}	112.49 (7)	K ^{xiii} —C11—K ^{xv}	159.73 (10)
C14 ⁱⁱⁱ —K—La ^{vi}	110.55 (6)	K ^{xiv} —C11—K ^{xv}	89.82 (11)
C13—K—La ^{vi}	80.57 (6)	La ^{xvi} —C12—K ^v	108.72 (9)
K ^v —K—La ^{vi}	121.04 (3)	La ^{xvi} —C12—K	108.72 (9)
C11 ⁱ —K—K ^{vi}	46.30 (6)	K ^v —C12—K	86.07 (11)

Cl2—K—K ^{vi}	113.09 (10)	La ^{xvi} —Cl2—K ^{xvii}	92.22 (8)
Cl1 ⁱⁱ —K—K ^{vi}	45.45 (6)	K ^v —Cl2—K ^{xvii}	88.75 (3)
Cl2 ⁱⁱⁱ —K—K ^{vi}	104.62 (9)	K—Cl2—K ^{xvii}	159.00 (11)
Cl3 ^{iv} —K—K ^{vi}	117.84 (10)	La ^{xvi} —Cl2—K ^{iv}	92.22 (8)
Cl4—K—K ^{vi}	59.28 (6)	K ^v —Cl2—K ^{iv}	159.00 (11)
Cl4 ⁱⁱⁱ —K—K ^{vi}	171.90 (11)	K—Cl2—K ^{iv}	88.75 (3)
Cl3—K—K ^{vi}	100.93 (9)	K ^{xvii} —Cl2—K ^{iv}	88.84 (11)
K ^v —K—K ^{vi}	91.23 (7)	La ^{xviii} —Cl3—K ⁱⁱⁱ	100.62 (8)
La ^{vi} —K—K ^{vi}	66.36 (5)	La ^{xviii} —Cl3—K ^{xix}	100.62 (8)
Cl1 ⁱ —K—K ⁱⁱⁱ	125.25 (10)	K ⁱⁱⁱ —Cl3—K ^{xix}	87.54 (11)
Cl2—K—K ⁱⁱⁱ	106.95 (10)	La ^{xviii} —Cl3—K	115.09 (9)
Cl1 ⁱⁱ —K—K ⁱⁱⁱ	104.75 (8)	K ⁱⁱⁱ —Cl3—K	85.21 (4)
Cl2 ⁱⁱⁱ —K—K ⁱⁱⁱ	45.10 (6)	K ^{xix} —Cl3—K	144.27 (10)
Cl3 ^{iv} —K—K ⁱⁱⁱ	97.44 (9)	La ^{xviii} —Cl3—K ^v	115.10 (9)
Cl4—K—K ⁱⁱⁱ	169.44 (11)	K ⁱⁱⁱ —Cl3—K ^v	144.27 (10)
Cl4 ⁱⁱⁱ —K—K ⁱⁱⁱ	47.24 (6)	K ^{xix} —Cl3—K ^v	85.21 (4)
Cl3—K—K ⁱⁱⁱ	46.65 (6)	K—Cl3—K ^v	80.64 (10)
K ^v —K—K ⁱⁱⁱ	91.22 (6)	La—Cl4—La ^x	106.42 (7)
La ^{vi} —K—K ⁱⁱⁱ	67.00 (4)	La—Cl4—K	102.93 (8)
K ^{vi} —K—K ⁱⁱⁱ	126.56 (8)	La ^x —Cl4—K	147.62 (10)
Cl3 ^{vii} —La—Cl1 ⁱ	127.18 (9)	La—Cl4—K ^{iv}	98.37 (8)
Cl3 ^{viii} —La—Cl2 ^{viii}	157.97 (10)	La ^x —Cl4—K ^{iv}	103.65 (8)
Cl1 ⁱ —La—Cl2 ^{viii}	74.85 (9)	K—Cl4—K ^{iv}	85.10 (6)

Symmetry codes: (i) $-x+1/2, -y+1, z-1/2$; (ii) $x+1/2, y, -z+3/2$; (iii) $-x+3/2, -y+1, z+1/2$; (iv) $-x+3/2, -y+1, z-1/2$; (v) $x, -y+1/2, z$; (vi) $-x+1, -y+1, -z+1$; (vii) $x, y, z-1$; (viii) $x-1/2, y, -z+1/2$; (ix) $-x+1, y-1/2, -z$; (x) $-x+1, -y+1, -z$; (xi) $-x+1, y-1/2, -z+1$; (xii) $-x+1/2, -y+1, z+1/2$; (xiii) $-x+1/2, y+1/2, z+1/2$; (xiv) $x-1/2, -y+3/2, -z+3/2$; (xv) $x-1/2, y, -z+3/2$; (xvi) $x+1/2, y, -z+1/2$; (xvii) $-x+3/2, y-1/2, z-1/2$; (xviii) $x, y, z+1$; (xix) $-x+3/2, y-1/2, z+1/2$.