

## Dipotassium hexaaquanickel(II) bis[hexafluoridozirconate(IV)]

Abdelghani Oudahmane, Noura Mnaouer, Malika El-Ghozzi and Daniel Avignant\*

Université Blaise Pascal, Laboratoire des Matériaux Inorganiques, UMR CNRS 6002, 24 Avenue des Landais, 63177 Aubière, France

Correspondence e-mail: daniel.avignant@univ-bpclermont.fr

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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{Ni}-\text{O}) = 0.001$  Å;  $R$  factor = 0.023;  $wR$  factor = 0.052; data-to-parameter ratio = 33.9.

Single crystals of the title compound,  $\text{K}_2[\text{Ni}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2$ , were grown by slow evaporation of a 40% aqueous HF solution in which a stoichiometric mixture of  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{ZrF}_4$  and  $\text{KCl}$  was dissolved. The monoclinic structure is isotypic with its  $\text{K}_2\text{Cu}$ ,  $\text{K}_2\text{Zn}$ ,  $\text{Cs}_2\text{Zn}$  and  $\text{Cs}_2\text{Cu}$  analogues. The structure is built up from isolated, slightly elongated octahedral  $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$  complex cations (symmetry  $\bar{1}$ ) and dimeric  $[\text{Zr}_2\text{F}_{12}]^{4-}$  complex anions (symmetry  $\bar{1}$ ) that are also isolated from each other. The  $[\text{Zr}_2\text{F}_{12}]^{4-}$  anion results from the association of two distorted pentagonal-bipyramidal  $[\text{ZrF}_7]$  coordination polyhedra by sharing an equatorial edge passing through an inversion center of the unit cell. Both isolated  $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$  and  $[\text{Zr}_2\text{F}_{12}]^{4-}$  complex ions are situated in planes parallel to (010). They are connected by the eight-coordinated  $\text{K}^+$  ions into a three-dimensional structure. An intricate  $\text{O}-\text{H} \cdots \text{F}$  hydrogen-bonding network consolidates the structure.

### Related literature

For isotypic structures, see: Fischer & Weiss (1973); Bukvetskii *et al.* (1993); Hitchman *et al.* (2002). For a review on the stereochemistry of zirconium and hafnium fluoro complexes, see: Davidovich (1998). For background to distortion indices, see: Momma & Izumi (2008).

### Experimental

#### Crystal data

$\text{K}_2[\text{Ni}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2$   
 $M_r = 655.45$   
 Monoclinic,  $P2_1/n$   
 $a = 6.6090$  (1) Å  
 $b = 10.0398$  (1) Å  
 $c = 11.7843$  (1) Å  
 $\beta = 95.897$  (1)°

$V = 777.79$  (2) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 3.20$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.28 \times 0.14 \times 0.09$  mm

#### Data collection

Bruker APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2008)  
 $T_{\min} = 0.613$ ,  $T_{\max} = 0.748$

17267 measured reflections  
 4436 independent reflections  
 3858 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$   
 $wR(F^2) = 0.052$   
 $S = 1.06$   
 4436 reflections

131 parameters  
 All H-atom parameters refined  
 $\Delta\rho_{\max} = 0.63$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.57$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H11} \cdots \text{F1}^i$	0.79 (3)	2.94 (2)	3.3300 (14)	112.8 (19)
$\text{O1}-\text{H11} \cdots \text{F2}^{ii}$	0.79 (3)	1.92 (3)	2.6877 (14)	164 (2)
$\text{O1}-\text{H12} \cdots \text{F4}^{iii}$	0.82 (3)	1.82 (3)	2.6375 (13)	177 (2)
$\text{O1}-\text{H12} \cdots \text{F5}^{iv}$	0.82 (3)	2.61 (2)	3.0526 (13)	116 (2)
$\text{O2}-\text{H21} \cdots \text{F1}^i$	0.78 (2)	2.59 (2)	3.0048 (13)	115 (2)
$\text{O2}-\text{H21} \cdots \text{F2}^{ii}$	0.78 (2)	1.95 (2)	2.7227 (13)	167 (2)
$\text{O2}-\text{H22} \cdots \text{F4}^v$	0.82 (2)	1.92 (2)	2.7362 (13)	173 (2)
$\text{O2}-\text{H22} \cdots \text{F6}^{vi}$	0.82 (2)	2.76 (2)	3.1924 (14)	114.9 (17)
$\text{O3}-\text{H31} \cdots \text{F1}^{vii}$	0.80 (3)	2.86 (3)	3.2552 (16)	113.3 (19)
$\text{O3}-\text{H31} \cdots \text{F5}^{viii}$	0.80 (3)	1.92 (3)	2.7019 (14)	168 (2)
$\text{O3}-\text{H32} \cdots \text{F3}^{iv}$	0.80 (3)	3.00 (2)	3.4527 (16)	119 (2)
$\text{O3}-\text{H32} \cdots \text{F6}^{ix}$	0.80 (3)	1.97 (3)	2.7427 (15)	163 (2)

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

**Table 2**

Comparison of the geometrical characteristics of the coordination polyhedra in isotypic  $M^I_2[M^{II}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2$  structures determined from single-crystal data ( $\text{\AA}$ ,  $^\circ$ ,  $\text{\AA}^3$ ).

	$\text{K}_2[\text{Ni}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2^a$	$\text{K}_2[\text{Cu}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2^b$	$\text{K}_2[\text{Zn}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2^c$	$\text{Cs}_2[\text{Zn}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2^d$
Space group	$P2_1/n$	$P2_1/c$	$P2_1/c$	$P2_1/n$
$a$	6.6090 (1)	6.631 (6)	6.631 (1)	6.970 (1)
$b$	10.0398 (1)	9.981 (10)	10.071 (1)	10.515 (2)
$c$	11.7843 (1)	12.921 (12)	12.952 (1)	11.803 (2)
$\beta$	95.897 (1)	114.20 (15)	114.96 (2)	93.56 (3)
$V$	777.786 (16)	780.01 (1)	784.16 (2)	863.4 (3)
Distances $M^{II}-\text{O}$	Ni—O1 = 2.0548 (10) (2 $\times$ ) Ni—O3 = 2.0570 (11) (2 $\times$ ) Ni—O2 = 2.0781 (9) (2 $\times$ )	Cu—O1 = 1.966 (4) (2 $\times$ ) Cu—O2 = 2.025 (6) (2 $\times$ ) Cu—O3 = 2.327 (5) (2 $\times$ )	Zn—O1 = 2.0856 (2) (2 $\times$ ) Zn—O2 = 2.0940 (1) (2 $\times$ ) Zn—O3 = 2.1185 (2) (2 $\times$ )	Zn—O3 = 2.096 (6) (2 $\times$ ) Zn—O1 = 2.099 (5) (2 $\times$ ) Zn—O2 = 2.105 (5) (2 $\times$ )
Average $M^{II}-\text{O}$ bond length	2.063	2.106	2.099	2.100
Polyhedral volume	11.684	12.335	12.318	12.341
Distortion index (bond length)	0.00483	0.06089	0.00607	0.00156
Quadratic elongation	1.0013	1.0124	1.0011	1.0006
Distances Zr—F ( $\text{\AA}$ )	Zr—F6 = 1.9718 (9) Zr—F5 = 2.0006 (8) Zr—F3 = 2.0293 (9) Zr—F2 = 2.0554 (8) Zr—F1 = 2.0708 (8) Zr—F4 = 2.1468 (9) Zr—F4 = 2.1614 (8)	Zr—F3 = 1.968 (5) Zr—F1 = 2.004 (5) Zr—F5 = 2.029 (4) Zr—F6 = 2.059 (4) Zr—F2 = 2.063 (4) Zr—F4 = 2.156 (5) Zr—F4 = 2.160 (4)	Zr—F3 = 1.9727 (3) Zr—F1 = 2.0018 (3) Zr—F5 = 2.0277 (3) Zr—F6 = 2.0570 (3) Zr—F2 = 2.0668 (4) Zr—F4 = 2.1501 (4) Zr—F4 = 2.1628 (4)	Zr—F3 = 1.962 (5) Zr—F6 = 1.977 (5) Zr—F5 = 2.037 (5) Zr—F4 = 2.067 (4) Zr—F2 = 2.069 (4) Zr—F1 = 2.156 (4) Zr—F1 = 2.180 (4)
Average Zr—F bond length	2.062	2.063	2.063	2.064
Polyhedral volume	13.669	13.674	13.675	13.692
Distortion index (bond length)	0.02662	0.02650	0.02654	0.02985
Distances K—F/O	K—F5 = 2.6496 (10) K—F3 = 2.7366 (9) K—F6 = 2.7603 (11) K—F2 = 2.7658 (9) K—F1 = 2.7895 (9) K—O2 = 2.8927 (10) K—O1 = 3.1012 (10) K—F6 = 3.1684 (12)	K—F1 = 2.668 (5) K—F6 = 2.750 (6) K—F3 = 2.756 (5) K—F5 = 2.767 (5) K—F2 = 2.799 (5) K—O3 = 2.942 (5) K—O1 = 2.980 (5) K—F3 = 3.1307 (7)	K—F1 = 2.6506 (3) K—F2 = 2.7395 (4) K—F5 = 2.7633 (2) K—F6 = 2.7739 (2) K—F3 = 2.8094 (2) K—O1 = 2.8968 (3) K—O2 = 3.0873 (5) K—F3 = 3.1707 (7)	Cs—F6 = 2.911 (5) Cs—F4 = 3.046 (4) Cs—F2 = 3.057 (4) Cs—F3 = 3.065 (5) Cs—F3 = 3.102 (5) Cs—O2 = 3.218 (5) Cs—O1 = 3.236 (5) Cs—F5 = 3.228 (6)
Average $M^I-\text{F/O}$ bond length	2.858	2.849	2.861	3.118
Polyhedral volume	38.458	38.158	38.569	48.130
Distortion index (bond length)	0.05146	0.04429	0.04984	0.02627

Notes: (a) this work; (b) Fischer & Weiss (1973); (c) Bukvetskii *et al.* (1993); (d) Hitchman *et al.* (2002).

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2439).

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**supplementary materials**

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## Dipotassium hexaaquanickel(II) bis[hexafluoridozirconate(IV)]

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### Comment

The existence of the title compound  $K_2[Ni(H_2O)_6][ZrF_6]_2$  as member of the large family of zirconium fluoro complexes with general formula  $M^I_2[M^{II}(H_2O)_6][ZrF_6]_2$  where  $M^I = K, Rb, Cs$  or  $NH_4$  and  $M^{II} = Co, Ni, Cu$  or  $Zn$ , has already been mentioned by Davidovich (1998). The monoclinic structure of the title compound is isotypic with those of  $K_2[Cu(H_2O)_6][ZrF_6]_2$  (Fischer & Weiss, 1973),  $K_2[Zn(H_2O)_6][ZrF_6]_2$  (Bukvetskii *et al.*, 1993) and  $Cs_2[Zn(H_2O)_6][ZrF_6]_2$  (Hitchman *et al.*, 2002). In the title structure the  $Ni^{2+}$  cation (site symmetry  $\bar{1}$ ) is coordinated by six water molecules with two Ni—O distances slightly longer (2.0781 (9) Å) than the four others (2x 2.0548 (10) Å and 2x 2.0570 (11) Å). The  $Zr^{4+}$  cation is 7-coordinated by the fluoride ions but rather than being isolated anions, the fluoridozirconate(IV) ions form centrosymmetric  $[Zr_2F_{12}]^{4-}$  dimers. Thus the structure is built up from isolated and slightly elongated octahedral  $[Ni(H_2O)_6]^{2+}$  complex cations and dimeric  $[Zr_2F_{12}]^{4-}$  complex anions, also isolated from each other. The  $[ZrF_7]$  coordination polyhedron is a distorted pentagonal bipyramid (symmetry 1) and the centrosymmetric  $[Zr_2F_{12}]^{4-}$  complex anion results from the association of two pentagonal bipyramids by sharing an equatorial edge F1—F1 passing through an inversion center of the unit cell corresponding to either the 2 *b* or 2 *c* Wyckoff positions. Both isolated  $[Ni(H_2O)_6]^{2+}$  and  $[Zr_2F_{12}]^{4-}$  complex ions lying in planes parallel to (010) are connected by the 8-coordinated  $K^+$  ions (Fig. 1) and an intricate O—H...F hydrogen bonds network (Fig. 2 and Table 1) to form the three-dimensional structure.

A careful examination of the geometry of the  $[Zr_2F_{12}]^{4-}$  complex anion in isotypic structures, refined from single-crystal data, shows this anion being quasi unvarying for all the members (Table 2). The distortion index (bond length) (Momma & Izumi, 2008) is the same for all the K compounds (0.0265) and is only very slightly higher (0.02985) for the Cs analogue. It is also worth noting that the higher the index of distortion of the  $[M^{II}(H_2O)_6]$  cationic polyhedron, the lower the index of distortion of the counter cation  $K^+$  (0.04429). This observation is obvious because water molecules are only shared between  $K^+$  and  $M^{2+}$  ions.

### Experimental

Single crystals of the title compound were obtained by reacting a mixture of  $NiCl_2 \cdot 6H_2O$ ,  $ZrF_4$  and  $KCl$  in the molar ratio 1:2:2 with a 40% aqueous HF boiling solution in a platinum crucible. Then the solution was poured out into a PTFE beaker and slowly evaporated to dryness using a sand bath. Green single-crystals of the title compound were extracted from the dry residue.

## Refinement

The highest residual peak in the final difference Fourier map was located 0.60 Å from the Zr atom and the deepest hole was located 0.86 Å from the same atom. H atom parameter were refined freely.

## Figures

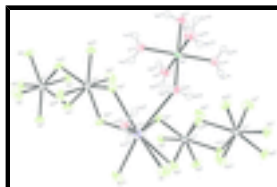


Fig. 1. View of the polyhedral linkage in  $\text{K}_2[\text{Ni}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2$ . Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i)  $x, y + 1, z$ ; (ii)  $x - 1, y + 1, z$ ; (iii)  $-x, -y + 1, -z + 1$ ; (iv)  $-x, -y, -z$ ; (v)  $-x, -y - 1, -z$ ; (vi)  $-x - 1/2, y + 1/2, -z + 1/2$ ; (vii)  $-x + 1/2, y + 1/2, -z + 1/2$ ; (viii)  $x + 1/2, -y + 3/2, z - 1/2$ ; (ix)  $x + 1, y + 1, z$ ; (x)  $-x + 1/2, y - 1/2, -z + 1/2$ ; (xi)  $x, y - 1, z$ ; (xii)  $x - 1, y - 1, z$ ; (xiii)  $x + 1, y - 1, z$ ; (xiv)  $x - 1, y, z$ ; (xv)  $x - 1/2, -y + 3/2, z + 1/2$ ; (xvi)  $-x - 1/2, y - 1/2, -z + 1/2$ .

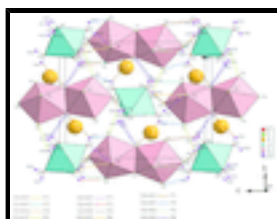


Fig. 2. Projection of the crystal structure of  $\text{K}_2[\text{Ni}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2$  along  $[100]$  showing the hydrogen-bonding interactions.

## Dipotassium hexaaquanickel bis[hexafluoridozirconate(IV)]

### Crystal data

$\text{K}_2[\text{Ni}(\text{H}_2\text{O})_6][\text{ZrF}_6]_2$

$M_r = 655.45$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 6.6090$  (1) Å

$b = 10.0398$  (1) Å

$c = 11.7843$  (1) Å

$\beta = 95.897$  (1)°

$V = 777.79$  (2) Å<sup>3</sup>

$Z = 2$

$F(000) = 628$

$D_x = 2.799$  Mg m<sup>-3</sup>

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

Cell parameters from 7940 reflections

$\theta = 3.7\text{--}38.7^\circ$

$\mu = 3.20$  mm<sup>-1</sup>

$T = 296$  K

Block, green

$0.28 \times 0.14 \times 0.09$  mm

### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

Detector resolution: 8.3333 pixels mm<sup>-1</sup>

$\omega$  and  $\phi$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2008)

$T_{\min} = 0.613$ ,  $T_{\max} = 0.748$

17267 measured reflections

4436 independent reflections

3858 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 38.8^\circ$ ,  $\theta_{\min} = 4.2^\circ$

$h = -11 \rightarrow 11$

$k = -17 \rightarrow 12$

$l = -18 \rightarrow 20$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.023$	All H-atom parameters refined
$wR(F^2) = 0.052$	$w = 1/[\sigma^2(F_o^2) + (0.0182P)^2 + 0.3179P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
4436 reflections	$(\Delta/\sigma)_{\max} = 0.032$
131 parameters	$\Delta\rho_{\max} = 0.63 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$
0 constraints	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0068 (4)

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Zr	-0.462200 (17)	0.984514 (11)	0.350424 (9)	0.01376 (3)
Ni	0.0000	0.0000	0.0000	0.01508 (4)
K	0.47534 (5)	0.71630 (3)	0.08423 (3)	0.02839 (7)
O1	0.21314 (15)	-0.11683 (10)	-0.06747 (9)	0.02154 (17)
O2	-0.23852 (15)	-0.12500 (10)	-0.05901 (9)	0.02039 (17)
O3	-0.04437 (19)	-0.88871 (12)	-0.14701 (9)	0.0293 (2)
F1	0.53736 (16)	0.11314 (9)	0.49592 (7)	0.0300 (2)
F2	-0.53154 (14)	0.78950 (8)	0.31006 (7)	0.02440 (16)
F3	-0.17901 (13)	0.93040 (11)	0.39692 (9)	0.0346 (2)
F4	-0.46635 (13)	0.97177 (8)	0.17481 (7)	0.02228 (15)
F5	-0.35001 (14)	0.16232 (8)	0.30407 (8)	0.02609 (17)
F6	-0.75507 (12)	0.03609 (11)	0.31945 (8)	0.02759 (18)
H11	0.159 (4)	-0.169 (3)	-0.111 (2)	0.044 (6)*
H12	0.289 (4)	-0.072 (3)	-0.103 (2)	0.044 (6)*
H21	-0.196 (3)	-0.178 (2)	-0.099 (2)	0.041 (6)*
H22	-0.327 (3)	-0.084 (2)	-0.0981 (19)	0.038 (6)*

## supplementary materials

H31	0.028 (4)	-0.829 (3)	-0.163 (2)	0.046 (7)*
H32	-0.086 (4)	-0.925 (3)	-0.205 (2)	0.048 (7)*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Zr	0.01662 (5)	0.01302 (5)	0.01144 (5)	-0.00068 (3)	0.00044 (3)	0.00045 (3)
Ni	0.01666 (9)	0.01442 (9)	0.01395 (9)	-0.00119 (6)	0.00064 (7)	-0.00028 (6)
K	0.03371 (15)	0.02741 (14)	0.02299 (14)	-0.00580 (11)	-0.00224 (11)	0.00113 (10)
O1	0.0229 (4)	0.0194 (4)	0.0230 (5)	-0.0021 (3)	0.0058 (4)	-0.0031 (3)
O2	0.0207 (4)	0.0186 (4)	0.0213 (4)	-0.0003 (3)	-0.0007 (3)	-0.0024 (3)
O3	0.0407 (6)	0.0279 (5)	0.0175 (5)	-0.0127 (4)	-0.0053 (4)	0.0056 (4)
F1	0.0579 (6)	0.0175 (4)	0.0158 (4)	-0.0105 (4)	0.0091 (4)	-0.0021 (3)
F2	0.0394 (5)	0.0162 (3)	0.0173 (4)	-0.0029 (3)	0.0014 (3)	-0.0013 (3)
F3	0.0216 (4)	0.0442 (6)	0.0365 (5)	0.0060 (4)	-0.0048 (3)	0.0052 (4)
F4	0.0280 (4)	0.0239 (4)	0.0152 (3)	-0.0041 (3)	0.0037 (3)	-0.0007 (3)
F5	0.0367 (4)	0.0181 (4)	0.0247 (4)	-0.0067 (3)	0.0091 (3)	0.0002 (3)
F6	0.0193 (4)	0.0350 (5)	0.0280 (5)	0.0055 (3)	0.0004 (3)	-0.0062 (3)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Zr—F3	1.9717 (9)	K—F3 <sup>xi</sup>	3.1685 (12)
Zr—F6 <sup>i</sup>	2.0006 (8)	O1—K <sup>xiii</sup>	2.8929 (11)
Zr—F5 <sup>i</sup>	2.0293 (8)	O1—H11	0.79 (3)
Zr—F2	2.0554 (8)	O1—H12	0.82 (3)
Zr—F4	2.0708 (8)	O2—K <sup>xiii</sup>	3.1015 (11)
Zr—F1 <sup>ii</sup>	2.1467 (8)	O2—H21	0.78 (2)
Zr—F1 <sup>iii</sup>	2.1614 (8)	O2—H22	0.82 (2)
Ni—O1 <sup>iv</sup>	2.0548 (10)	O3—Ni <sup>xii</sup>	2.0570 (11)
Ni—O1	2.0548 (10)	O3—H31	0.80 (3)
Ni—O3 <sup>v</sup>	2.0570 (11)	O3—H32	0.80 (3)
Ni—O3 <sup>i</sup>	2.0570 (11)	F1—Zr <sup>xiv</sup>	2.1467 (8)
Ni—O2 <sup>iv</sup>	2.0781 (9)	F1—Zr <sup>iii</sup>	2.1614 (8)
Ni—O2	2.0781 (9)	F2—K <sup>xv</sup>	2.7658 (9)
K—F6 <sup>vi</sup>	2.6497 (9)	F3—K <sup>xvi</sup>	2.7603 (10)
K—F5 <sup>vii</sup>	2.7365 (10)	F3—K <sup>vii</sup>	3.1685 (12)
K—F3 <sup>viii</sup>	2.7603 (10)	F4—K <sup>xv</sup>	2.7896 (9)
K—F2 <sup>ix</sup>	2.7658 (9)	F5—Zr <sup>xii</sup>	2.0293 (8)
K—F4 <sup>ix</sup>	2.7896 (9)	F5—K <sup>xi</sup>	2.7365 (10)
K—O1 <sup>i</sup>	2.8929 (11)	F6—Zr <sup>xii</sup>	2.0006 (8)
K—O2 <sup>x</sup>	3.1015 (11)	F6—K <sup>xvii</sup>	2.6497 (9)
F3—Zr—F6 <sup>i</sup>	174.24 (4)	F5 <sup>vii</sup> —K—F2 <sup>ix</sup>	72.14 (3)
F3—Zr—F5 <sup>i</sup>	87.40 (4)	F3 <sup>viii</sup> —K—F2 <sup>ix</sup>	150.45 (3)
F6 <sup>i</sup> —Zr—F5 <sup>i</sup>	95.54 (4)	F6 <sup>vi</sup> —K—F4 <sup>ix</sup>	121.62 (3)

F3—Zr—F2	89.11 (4)	F5 <sup>vii</sup> —K—F4 <sup>ix</sup>	85.15 (3)
F6 <sup>i</sup> —Zr—F2	90.90 (4)	F3 <sup>viii</sup> —K—F4 <sup>ix</sup>	145.26 (3)
F5 <sup>i</sup> —Zr—F2	148.71 (3)	F2 <sup>ix</sup> —K—F4 <sup>ix</sup>	53.33 (2)
F3—Zr—F4	100.10 (4)	F6 <sup>vi</sup> —K—O1 <sup>i</sup>	109.85 (3)
F6 <sup>i</sup> —Zr—F4	85.44 (4)	F5 <sup>vii</sup> —K—O1 <sup>i</sup>	150.10 (3)
F5 <sup>i</sup> —Zr—F4	75.70 (3)	F3 <sup>viii</sup> —K—O1 <sup>i</sup>	70.50 (3)
F2—Zr—F4	74.34 (3)	F2 <sup>ix</sup> —K—O1 <sup>i</sup>	111.88 (3)
F3—Zr—F1 <sup>ii</sup>	91.31 (4)	F4 <sup>ix</sup> —K—O1 <sup>i</sup>	75.79 (3)
F6 <sup>i</sup> —Zr—F1 <sup>ii</sup>	84.80 (4)	F6 <sup>vi</sup> —K—O2 <sup>x</sup>	167.54 (3)
F5 <sup>i</sup> —Zr—F1 <sup>ii</sup>	73.52 (3)	F5 <sup>vii</sup> —K—O2 <sup>x</sup>	77.96 (3)
F2—Zr—F1 <sup>ii</sup>	137.67 (3)	F3 <sup>viii</sup> —K—O2 <sup>x</sup>	92.00 (3)
F4—Zr—F1 <sup>ii</sup>	146.55 (3)	F2 <sup>ix</sup> —K—O2 <sup>x</sup>	117.28 (3)
F3—Zr—F1 <sup>iii</sup>	86.30 (4)	F4 <sup>ix</sup> —K—O2 <sup>x</sup>	70.61 (3)
F6 <sup>i</sup> —Zr—F1 <sup>iii</sup>	88.22 (4)	O1 <sup>i</sup> —K—O2 <sup>x</sup>	74.11 (3)
F5 <sup>i</sup> —Zr—F1 <sup>iii</sup>	138.27 (3)	F6 <sup>vi</sup> —K—F3 <sup>xi</sup>	66.39 (3)
F2—Zr—F1 <sup>iii</sup>	72.35 (3)	F5 <sup>vii</sup> —K—F3 <sup>xi</sup>	55.25 (2)
F4—Zr—F1 <sup>iii</sup>	145.96 (3)	F3 <sup>viii</sup> —K—F3 <sup>xi</sup>	72.07 (3)
F1 <sup>ii</sup> —Zr—F1 <sup>iii</sup>	65.45 (4)	F2 <sup>ix</sup> —K—F3 <sup>xi</sup>	102.83 (3)
F3—Zr—Zr <sup>xviii</sup>	88.57 (3)	F4 <sup>ix</sup> —K—F3 <sup>xi</sup>	139.92 (3)
F6 <sup>i</sup> —Zr—Zr <sup>xviii</sup>	85.86 (3)	O1 <sup>i</sup> —K—F3 <sup>xi</sup>	142.29 (3)
F5 <sup>i</sup> —Zr—Zr <sup>xviii</sup>	106.09 (3)	O2 <sup>x</sup> —K—F3 <sup>xi</sup>	103.01 (3)
F2—Zr—Zr <sup>xviii</sup>	104.90 (2)	F6 <sup>vi</sup> —K—Zr <sup>xi</sup>	89.24 (2)
F4—Zr—Zr <sup>xviii</sup>	171.25 (2)	F5 <sup>vii</sup> —K—Zr <sup>xi</sup>	28.097 (18)
F1 <sup>ii</sup> —Zr—Zr <sup>xviii</sup>	32.85 (2)	F3 <sup>viii</sup> —K—Zr <sup>xi</sup>	93.88 (2)
F1 <sup>iii</sup> —Zr—Zr <sup>xviii</sup>	32.60 (2)	F2 <sup>ix</sup> —K—Zr <sup>xi</sup>	93.56 (2)
F3—Zr—K <sup>vii</sup>	51.84 (3)	F4 <sup>ix</sup> —K—Zr <sup>xi</sup>	112.72 (2)
F6 <sup>i</sup> —Zr—K <sup>vii</sup>	129.16 (3)	O1 <sup>i</sup> —K—Zr <sup>xi</sup>	151.51 (2)
F5 <sup>i</sup> —Zr—K <sup>vii</sup>	39.43 (3)	O2 <sup>x</sup> —K—Zr <sup>xi</sup>	83.09 (2)
F2—Zr—K <sup>vii</sup>	139.29 (3)	F3 <sup>xi</sup> —K—Zr <sup>xi</sup>	29.294 (16)
F4—Zr—K <sup>vii</sup>	99.03 (2)	F6 <sup>vi</sup> —K—Zr <sup>ix</sup>	97.85 (2)
F1 <sup>ii</sup> —Zr—K <sup>vii</sup>	63.98 (3)	F5 <sup>vii</sup> —K—Zr <sup>ix</sup>	75.428 (19)
F1 <sup>iii</sup> —Zr—K <sup>vii</sup>	110.72 (3)	F3 <sup>viii</sup> —K—Zr <sup>ix</sup>	163.11 (2)
Zr <sup>xviii</sup> —Zr—K <sup>vii</sup>	87.200 (6)	F2 <sup>ix</sup> —K—Zr <sup>ix</sup>	26.431 (17)
F3—Zr—K <sup>xv</sup>	92.84 (3)	F4 <sup>ix</sup> —K—Zr <sup>ix</sup>	27.023 (17)
F6 <sup>i</sup> —Zr—K <sup>xv</sup>	90.62 (3)	O1 <sup>i</sup> —K—Zr <sup>ix</sup>	95.67 (2)
F5 <sup>i</sup> —Zr—K <sup>xv</sup>	112.37 (3)	O2 <sup>x</sup> —K—Zr <sup>ix</sup>	93.42 (2)
F2—Zr—K <sup>xv</sup>	36.80 (2)	F3 <sup>xi</sup> —K—Zr <sup>ix</sup>	121.99 (2)
F4—Zr—K <sup>xv</sup>	37.74 (2)	Zr <sup>xi</sup> —K—Zr <sup>ix</sup>	102.640 (7)
F1 <sup>ii</sup> —Zr—K <sup>xv</sup>	172.94 (3)	F6 <sup>vi</sup> —K—Zr <sup>xvii</sup>	20.660 (19)
F1 <sup>iii</sup> —Zr—K <sup>xv</sup>	109.12 (2)	F5 <sup>vii</sup> —K—Zr <sup>xvii</sup>	120.27 (2)
Zr <sup>xviii</sup> —Zr—K <sup>xv</sup>	141.547 (7)	F3 <sup>viii</sup> —K—Zr <sup>xvii</sup>	67.44 (2)



## supplementary materials

K <sup>vii</sup> —Zr—K <sup>xv</sup>	123.004 (4)	F2 <sup>ix</sup> —K—Zr <sup>xvii</sup>	83.04 (2)
F3—Zr—K <sup>vi</sup>	148.57 (3)	F4 <sup>ix</sup> —K—Zr <sup>xvii</sup>	121.05 (2)
F6 <sup>i</sup> —Zr—K <sup>vi</sup>	27.86 (3)	O1 <sup>i</sup> —K—Zr <sup>xvii</sup>	89.46 (2)
F5 <sup>i</sup> —Zr—K <sup>vi</sup>	83.54 (3)	O2 <sup>x</sup> —K—Zr <sup>xvii</sup>	157.34 (2)
F2—Zr—K <sup>vi</sup>	113.62 (3)	F3 <sup>xi</sup> —K—Zr <sup>xvii</sup>	80.348 (18)
F4—Zr—K <sup>vi</sup>	106.63 (3)	Zr <sup>xi</sup> —K—Zr <sup>xvii</sup>	106.804 (8)
F1 <sup>ii</sup> —Zr—K <sup>vi</sup>	57.25 (3)	Zr <sup>ix</sup> —K—Zr <sup>xvii</sup>	103.843 (7)
F1 <sup>iii</sup> —Zr—K <sup>vi</sup>	80.80 (3)	F6 <sup>vi</sup> —K—Zr <sup>viii</sup>	65.81 (2)
Zr <sup>xviii</sup> —Zr—K <sup>vi</sup>	65.435 (5)	F5 <sup>vii</sup> —K—Zr <sup>viii</sup>	137.16 (2)
K <sup>vii</sup> —Zr—K <sup>vi</sup>	106.804 (8)	F3 <sup>viii</sup> —K—Zr <sup>viii</sup>	20.89 (2)
K <sup>xv</sup> —Zr—K <sup>vi</sup>	118.448 (4)	F2 <sup>ix</sup> —K—Zr <sup>viii</sup>	131.81 (2)
F3—Zr—K <sup>xvi</sup>	29.95 (3)	F4 <sup>ix</sup> —K—Zr <sup>viii</sup>	137.38 (2)
F6 <sup>i</sup> —Zr—K <sup>xvi</sup>	144.59 (3)	O1 <sup>i</sup> —K—Zr <sup>viii</sup>	63.49 (2)
F5 <sup>i</sup> —Zr—K <sup>xvi</sup>	110.23 (3)	O2 <sup>x</sup> —K—Zr <sup>viii</sup>	107.49 (2)
F2—Zr—K <sup>xvi</sup>	79.65 (3)	F3 <sup>xi</sup> —K—Zr <sup>viii</sup>	82.684 (18)
F4—Zr—K <sup>xvi</sup>	123.50 (3)	Zr <sup>xi</sup> —K—Zr <sup>viii</sup>	109.132 (8)
F1 <sup>ii</sup> —Zr—K <sup>xvi</sup>	79.97 (3)	Zr <sup>ix</sup> —K—Zr <sup>viii</sup>	143.549 (10)
F1 <sup>iii</sup> —Zr—K <sup>xvi</sup>	56.37 (3)	Zr <sup>xvii</sup> —K—Zr <sup>viii</sup>	50.252 (5)
Zr <sup>xviii</sup> —Zr—K <sup>xvi</sup>	64.313 (5)	Ni—O1—K <sup>xii</sup>	118.84 (4)
K <sup>vii</sup> —Zr—K <sup>xvi</sup>	70.868 (8)	Ni—O1—H11	110.3 (17)
K <sup>xv</sup> —Zr—K <sup>xvi</sup>	100.955 (4)	K <sup>xii</sup> —O1—H11	102.4 (18)
K <sup>vi</sup> —Zr—K <sup>xvi</sup>	129.748 (5)	Ni—O1—H12	111.2 (17)
O1 <sup>iv</sup> —Ni—O1	180.00 (7)	K <sup>xii</sup> —O1—H12	106.2 (17)
O1 <sup>iv</sup> —Ni—O3 <sup>v</sup>	91.62 (5)	H11—O1—H12	107 (2)
O1—Ni—O3 <sup>v</sup>	88.38 (5)	Ni—O2—K <sup>xiii</sup>	127.74 (4)
O1 <sup>iv</sup> —Ni—O3 <sup>i</sup>	88.38 (5)	Ni—O2—H21	107.6 (17)
O1—Ni—O3 <sup>i</sup>	91.62 (5)	K <sup>xiii</sup> —O2—H21	105.2 (17)
O3 <sup>v</sup> —Ni—O3 <sup>i</sup>	180.0	Ni—O2—H22	111.1 (16)
O1 <sup>iv</sup> —Ni—O2 <sup>iv</sup>	93.00 (4)	K <sup>xiii</sup> —O2—H22	97.1 (15)
O1—Ni—O2 <sup>iv</sup>	87.00 (4)	H21—O2—H22	106 (2)
O3 <sup>v</sup> —Ni—O2 <sup>iv</sup>	90.50 (4)	Ni <sup>xii</sup> —O3—H31	124.4 (17)
O3 <sup>i</sup> —Ni—O2 <sup>iv</sup>	89.50 (4)	Ni <sup>xii</sup> —O3—H32	118.5 (19)
O1 <sup>iv</sup> —Ni—O2	87.00 (4)	H31—O3—H32	108 (2)
O1—Ni—O2	93.00 (4)	Zr <sup>xiv</sup> —F1—Zr <sup>iii</sup>	114.55 (4)
O3 <sup>v</sup> —Ni—O2	89.50 (4)	Zr—F2—K <sup>xv</sup>	116.77 (3)
O3 <sup>i</sup> —Ni—O2	90.50 (4)	Zr—F3—K <sup>xvi</sup>	129.16 (5)
O2 <sup>iv</sup> —Ni—O2	180.00 (5)	Zr—F3—K <sup>vii</sup>	98.87 (4)
F6 <sup>vi</sup> —K—F5 <sup>vii</sup>	99.70 (3)	K <sup>xvi</sup> —F3—K <sup>vii</sup>	107.93 (3)
F6 <sup>vi</sup> —K—F3 <sup>viii</sup>	78.70 (3)	Zr—F4—K <sup>xv</sup>	115.24 (3)
F5 <sup>vii</sup> —K—F3 <sup>viii</sup>	121.39 (3)	Zr <sup>xii</sup> —F5—K <sup>xi</sup>	112.48 (4)
F6 <sup>vi</sup> —K—F2 <sup>ix</sup>	72.85 (3)	Zr <sup>xii</sup> —F6—K <sup>xvii</sup>	131.48 (4)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H11 $\cdots$ F1 <sup>xi</sup>	0.79 (3)	2.94 (2)	3.3300 (14)	112.8 (19)
O1—H11 $\cdots$ F2 <sup>xix</sup>	0.79 (3)	1.92 (3)	2.6877 (14)	164 (2)
O1—H12 $\cdots$ F4 <sup>xx</sup>	0.82 (3)	1.82 (3)	2.6375 (13)	177 (2)
O1—H12 $\cdots$ F5 <sup>iv</sup>	0.82 (3)	2.61 (2)	3.0526 (13)	116 (2)
O2—H21 $\cdots$ F1 <sup>xi</sup>	0.78 (2)	2.59 (2)	3.0048 (13)	115 (2)
O2—H21 $\cdots$ F2 <sup>xix</sup>	0.78 (2)	1.95 (2)	2.7227 (13)	167 (2)
O2—H22 $\cdots$ F4 <sup>xxi</sup>	0.82 (2)	1.92 (2)	2.7362 (13)	173 (2)
O2—H22 $\cdots$ F6 <sup>xxii</sup>	0.82 (2)	2.76 (2)	3.1924 (14)	114.9 (17)
O3—H31 $\cdots$ F1 <sup>xxiii</sup>	0.80 (3)	2.86 (3)	3.2552 (16)	113.3 (19)
O3—H31 $\cdots$ F5 <sup>xxiv</sup>	0.80 (3)	1.92 (3)	2.7019 (14)	168 (2)
O3—H32 $\cdots$ F3 <sup>iv</sup>	0.80 (3)	3.00 (2)	3.4527 (16)	119 (2)
O3—H32 $\cdots$ F6 <sup>xxv</sup>	0.80 (3)	1.97 (3)	2.7427 (15)	163 (2)

Symmetry codes: (xi)  $-x+1/2, y-1/2, -z+1/2$ ; (xix)  $x+1/2, -y+1/2, z-1/2$ ; (xx)  $-x, -y+1, -z$ ; (iv)  $-x, -y, -z$ ; (xxi)  $-x-1, -y+1, -z$ ; (xxii)  $-x-1, -y, -z$ ; (xxiii)  $x-1/2, -y-1/2, z-1/2$ ; (xxiv)  $x+1/2, -y-1/2, z-1/2$ ; (xxv)  $-x-1, -y-1, -z$ .

Table 2

Comparison of the geometrical characteristics of the coordination polyhedra in isotypic  $M^I_2[M^{II}(H_2O)_6][ZrF_6]_2$  structures determined from single-crystal data ( $\text{\AA}, ^\circ, \text{\AA}^3$ )

	$K_2[Ni(H_2O)_6][ZrF_6]_2^a$	$K_2[Cu(H_2O)_6][ZrF_6]_2^b$	$K_2[Zn(H_2O)_6][ZrF_6]_2^c$	$Cs_2[Zn(H_2O)_6][ZrF_6]_2^d$
Space group	$P2_1/n$	$P2_1/c$	$P2_1/c$	$P2_1/n$
$a$	6.6090 (1)	6.631 (6)	6.631 (1)	6.970 (1)
$b$	10.0398 (1)	9.981 (10)	10.071 (1)	10.515 (2)
$c$	11.7843 (1)	12.921 (12)	12.952 (1)	11.803 (2)
$\beta$	95.897 (1)	114.20 (15)	114.96 (2)	93.56 (3)
$V$	777.786 (16)	780.01 (1)	784.16 (2)	863.4 (3)
Distances $M^{II}-O$	Ni—O1 = 2.0548 (10) (2 $\times$ )	Cu—O1 = 1.966 (4) (2 $\times$ )	Zn—O1 = 2.0856 (2) (2 $\times$ )	Zn—O3 = 2.096 (6) (2 $\times$ )
	Ni—O3 = 2.0570 (11) (2 $\times$ )	Cu—O2 = 2.025 (6) (2 $\times$ )	Zn—O2 = 2.0940 (1) (2 $\times$ )	Zn—O1 = 2.099 (5) (2 $\times$ )
	Ni—O2 = 2.0781 (9) (2 $\times$ )	Cu—O3 = 2.327 (5) (2 $\times$ )	Zn—O3 = 2.1185 (2) (2 $\times$ )	Zn—O2 = 2.105 (5) (2 $\times$ )
Average $M^{II}-O$ bond length	2.063	2.106	2.099	2.100
Polyhedral volume	11.684	12.335	12.318	12.341
Distortion index (bond length)	0.00483	0.06089	0.00607	0.00156
Quadratic elongation	1.0013	1.0124	1.0011	1.0006
	Zr—F6 = 1.9718 (9)	Zr—F3 = 1.968 (5)	Zr—F3 = 1.9727 (3)	Zr—F3 = 1.962 (5)
	Zr—F5 = 2.0006 (8)	Zr—F1 = 2.004 (5)	Zr—F1 = 2.0018 (3)	Zr—F6 = 1.977 (5)
	Zr—F3 = 2.0293 (9)	Zr—F5 = 2.029 (4)	Zr—F5 = 2.0277 (3)	Zr—F5 = 2.037 (5)

## supplementary materials

Distances Zr—F (Å)	Zr—F2 = 2.0554 (8)	Zr—F6 = 2.059 (4)	Zr—F6 = 2.0570 (3)	Zr—F4 = 2.067 (4)
	Zr—F1 = 2.0708 (8)	Zr—F2 = 2.063 (4)	Zr—F2 = 2.0668 (4)	Zr—F2 = 2.069 (4)
	Zr—F4 = 2.1468 (9)	Zr—F4 = 2.156 (5)	Zr—F4 = 2.1501 (4)	Zr—F1 = 2.156 (4)
	Zr—F4 = 2.1614 (8)	Zr—F4 = 2.160 (4)	Zr—F4 = 2.1628 (4)	Zr—F1 = 2.180 (4)
Average Zr—F bond length	2.062	2.063	2.063	2.064
Polyhedral volume	13.669	13.674	13.675	13.692
Distortion index (bond length)	0.02662	0.02650	0.02654	0.02985
Distances K—F/O	K—F5 = 2.6496 (10)	K—F1 = 2.668 (5)	K—F1 = 2.6506 (3)	Cs—F6 = 2.911 (5)
	K—F3 = 2.7366 (9)	K—F6 = 2.750 (6)	K—F2 = 2.7395 (4)	Cs—F4 = 3.046 (4)
	K—F6 = 2.7603 (11)	K—F3 = 2.756 (5)	K—F5 = 2.7633 (2)	Cs—F2 = 3.057 (4)
	K—F2 = 2.7658 (9)	K—F5 = 2.767 (5)	K—F6 = 2.7739 (2)	Cs—F3 = 3.065 (5)
	K—F1 = 2.7895 (9)	K—F2 = 2.799 (5)	K—F3 = 2.8094 (2)	Cs—F3 = 3.102 (5)
	K—O2 = 2.8927 (10)	K—O3 = 2.942 (5)	K—O1 = 2.8968 (3)	Cs—O2 = 3.218 (5)
	K—O1 = 3.1012 (10)	K—O1 = 2.980 (5)	K—O2 = 3.0873 (5)	Cs—O1 = 3.236 (5)
K—F6 = 3.1684 (12)	K—F3 = 3.1307 (7)	K—F3 = 3.1707 (7)	Cs—F5 = 3.228 (6)	
Average $M^I$ —F/O bond length	2.858	2.849	2.861	3.118
Polyhedral volume	38.458	38.158	38.569	48.130
Distortion index (bond length)	0.05146	0.04429	0.04984	0.02627

Notes: (a) this work; (b) Fischer & Weiss (1973); (c) Bukvetskii *et al.* (1993); (d) Hitchman *et al.* (2002).

Fig. 1

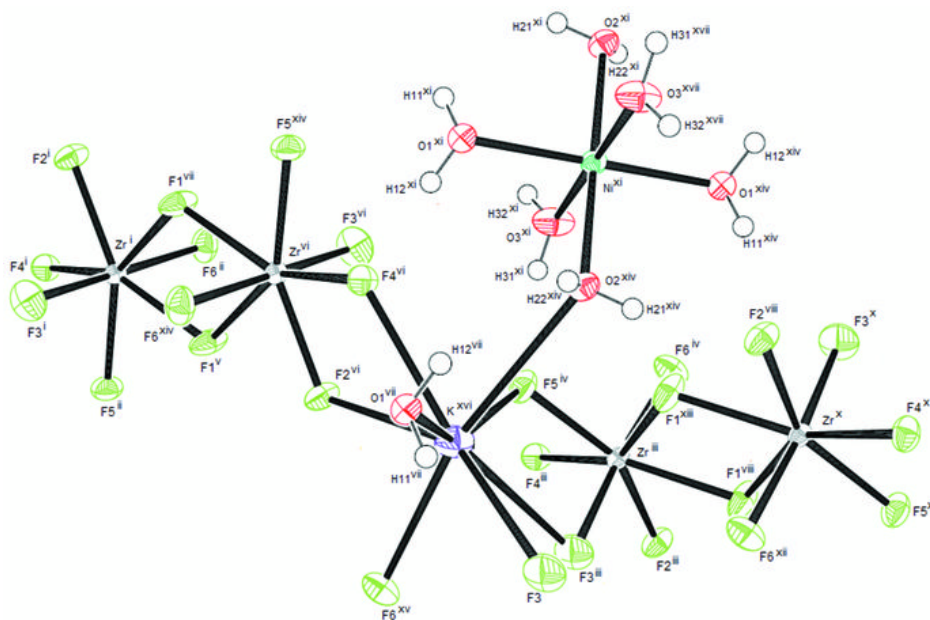


Fig. 2

