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# Crystal structure of a new tripotassium hexanickel iron hexaphosphate

**Said Ouaatta,* Abderrazzak Assani, Mohamed Saadi and Lahcen El Ammari**

Laboratoire de Chimie Appliquée des Matŕiaux, Centre des Sciences des Matériaux, Faculty of Sciences, Mohammed V University in Rabat, Avenue Ibn Batouta, BP 1014, Rabat, Morocco. *Correspondence e-mail: saidouaatta87@gmail.com

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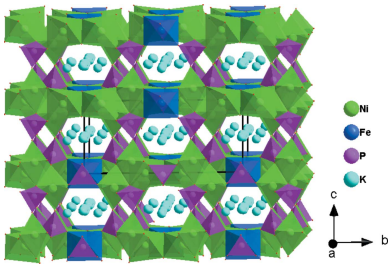
A new potassium-nickel iron phosphate, $K_3Ni_6Fe(PO_4)_6$, has been synthesized by solid-state reaction and structurally characterized by single-crystal X-ray diffraction and qualitative energy dispersive X-ray spectroscopy (EDS) analysis. The structure is built up by $[FeO_6]$, $[PO_4]$, and $[NiO_6]$ coordination polyhedra, which are linked to each other by edge and corner sharing to form zigzag layers parallel to the *ab* plane. These layers are interconnected by $[PO_4]$ tetrahedra and $[NiO_6]$ octahedra *via* common corners, leading to a three-dimensional framework delimiting large channels running along the [100] direction in which the $K^+$ cations are localized.

## 1. Chemical context

Iron-based phosphates are widely studied materials today. They present a promising field for various applications such as electronics (Saw *et al.*, 2014), ferroelectrics (Lazoryak *et al.*, 2004), magnetic materials (Hatert *et al.*, 2004; Essehli *et al.*, 2015) and catalytic processes (Moffat, 1978). The introduction of alkali metals into these phosphates materials can be of great interest to improve the ion-conduction properties for applications in rechargeable alkaline batteries (La Parola *et al.*, 2018; Orikasa *et al.*, 2016). The present work is part of our activity devoted particularly to the investigation of new materials based on phosphates belonging to the $A_2O$–$MO$–$Fe_2O_3$–$P_2O_5$ ($A$ = an alkali metal; $M$ = divalent cation) quaternary system, which could have interesting ionic conductivity or magnetic proprieties. We report herein on the synthesis and structural characterization by single crystal X-ray diffraction of a new potassium nickel iron phosphate with formula $K_3Ni_6Fe(PO_4)_6$.

## 2. Structural commentary

The asymmetric unit of the title compound, $K_3Ni_6Fe(PO_4)_6$, consists of two $[NiO_6]$ octahedra, one $[FeO_6]$ octahedron, two $[PO_4]$ tetrahedra, and three K atoms, as shown in Fig. 1. One $Ni^{2+}$, $Fe^{3+}$, $P^{5+}$, two $K^+$ cations and two of the seven oxygen atoms lie on special positions. The Ni2 atom occupies Wyckoff position 4*g* (2), the Fe atom is localized on the 2*a* (2/*m*) Wyckoff position, P2, K1, K3, O6 and O7 are positioned on 4*i* (*m*) sites. The octahedral coordination sphere of the nickel(II) cation is more distorted than that of the iron(III) atom, with average <Ni—O> distances of 2.066 and 2.119 Å for Ni1 and Ni2, respectively. The mean <P—O> distance in the two $PO_4$ tetrahedra is equal to 1.547 Å for P1 and 1.543 Å for P2.



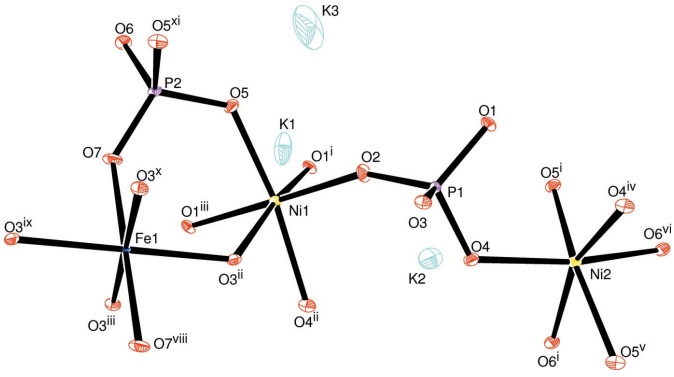



**Figure 1**
Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x+1, y, -z+1$; (iii) $x, y, z-1$; (iv) $-x+1, y, -z+2$; (v) $x+\frac{1}{2}, -y+\frac{3}{2}, z+1$; (vi) $x+\frac{1}{2}, y+\frac{1}{2}, z+1$; (vii) $-x+1, -y+1, -z$; (viii) $x, -y+1, z-1$; (ix) $-x+1, -y+1, -z+1$; (x) $x, -y+1, z$; (xi) $x, y, z+1$; (xii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z$; (xiii) $-x, -y+1, -z$; (xiv) $x, -y+2, z$.

The Fe atoms are coordinated octahedrally with an average <Fe—O> distance of 2.038 Å. The structure of the title compound is built up from two types of nickel sites and one iron site, each with an octahedral coordination environment, [Ni1$O_6$], [Ni2$O_6$] and [Fe$O_6$], besides two independent phosphor tetrahedra [P1$O_4$] and [P2$O_4$]. Edge-sharing [Ni2$O_6$] octahedra build up a dimeric [$Ni2_2O_{10}$] unit. Two [P2$O_6$] octahedra are connected to the [$Ni2_2O_{10}$] dimer by sharing edges to form an [$Ni(2)_2P(2)_2O_{12}$] unit, which alternates with an [Fe$O_6$] octahedron to establish an infinite chain along the [100] direction (Fig. 2). In addition, the association between the [P1$O_4$] tetrahedra and the [Ni1$O_6$] octahedra by means of edge-sharing allows the formation of a zigzag chain running parallel to the [100] direction. Each of the P1$O_4$ tetrahedra and Ni1$O_6$ octahedra, both belonging to the same layer, share vertices with Ni1$O_6$ and P1$O_4$, respectively, of the adjacent one (Fig. 3). The two types of chain linkages lead to the formation of layers parallel to the *ab* plane (Fig. 4). One vertex of an Ni1$O_6$ octahedron belonging to one layer is shared with a P1$O_4$ vertex of the neighbouring layer. This configuration leads to a three-dimensional centrosymmetric framework, delimiting hexagonal tunnels along the [100] direction, in which the $K^+$ cations are located (Fig. 5). The potassium cations are distributed over three independent crystallographic positions with partial occupancies

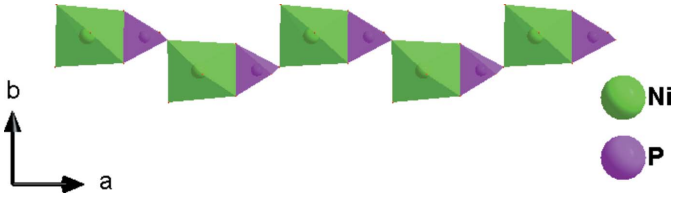



**Figure 3**
Corner- and edge-sharing [P1$O_4$] tetrahedra and [Ni1$O_6$] octahedra forming a zigzag shape chain running parallel to [100]

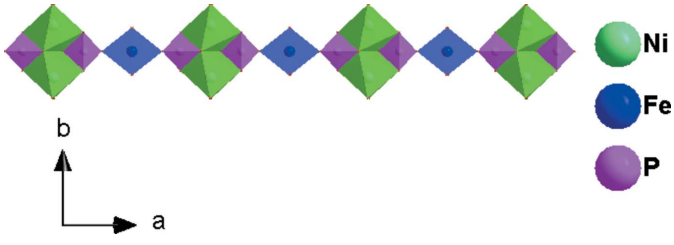



**Figure 2**
A chain formed by sharing edges and corners of [$Ni2_2O_{10}$] dimers, [P2$O_4$] tetrahedra and [Fe$O_6$] octahedra along the [100] direction

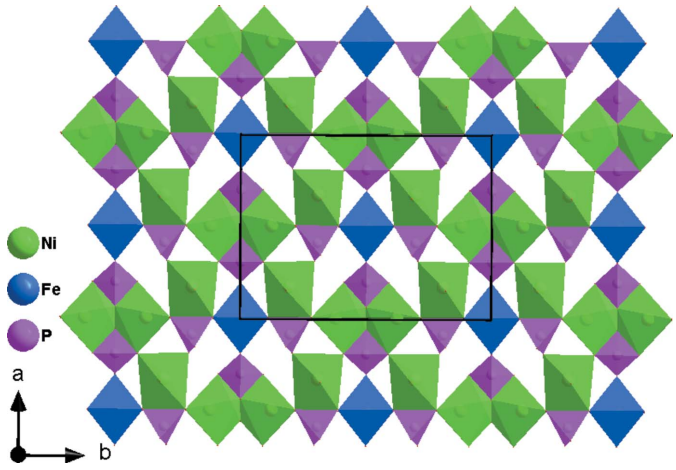



**Figure 4**
View along the *c* axis of corner- and edge-sharing [$PO_4$] tetrahedra and [$NiO_6$] octahedra forming a layer parallel to the *ab* plane.

## 3. Database survey

The investigated compound is a new member of the $\beta$-xenophyllite family that includes $Na_4Ni_7(PO_4)_6$ (Moring & Kostiner, 1986), $Na_4Co_7(PO_4)_6$ (Kobashi *et al.*, 1998),

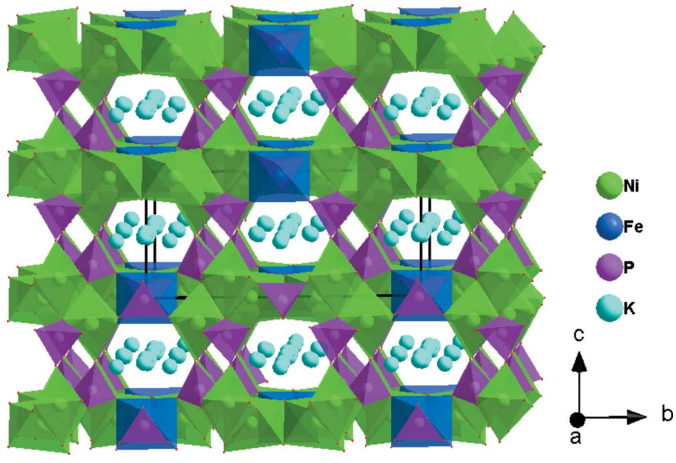



**Figure 5**
Polyhedral representation of the crystal structure of $K_3Ni_6Fe(PO_4)_6$ showing large tunnels running along the [100] direction that contain the $K^+$ cations.

**Table 1**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $K_3Ni_6Fe(PO_4)_6$ |
| $M_r$ | 1095.23 |
| Crystal system, space group | Monoclinic, *C*2/*m* |
| Temperature (K) | 296 |
| *a*, *b*, *c* (Å) | 10.6853 (4), 14.1009 (5), 6.5481 (2) |
| $\beta$ (°) | 103.842 (1) |
| $V$ (Å$^3$) | 957.97 (6) |
| $Z$ | 2 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 7.79 |
| Crystal size (mm) | 0.36 × 0.27 × 0.20 |
| | |
| Data collection | |
| Diffractometer | Bruker D8 VENTURE Super DUO |
| Absorption correction | Multi-scan (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.638, 0.746 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 13870, 1981, 1891 |
| $R_{int}$ | 0.021 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.781 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.024, 0.062, 1.07 |
| No. of reflections | 1981 |
| No. of parameters | 112 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 2.34, −1.16 |

Computer programs: *APEX3* and *SAINT-Plus* (Bruker, 2016), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006) and *publCIF* (Westrip, 2010).

$K_4Ni_7(AsO_4)_6$ (Ben Smail *et al.*, 1999), $Na_4Co_{5.63}Al_{0.91}(AsO_4)_6$ (Marzouki *et al.*, 2010), $Na_4Li_{0.62}Co_{5.67}Al_{0.71}(AsO_4)_6$ (Marzouki *et al.*, 2013), $Ag_4Co_7(AsO_4)_6$ (Marzouki *et al.*, 2014) and $Na_4Co_7(AsO_4)_6$ (Ben Smida *et al.*, 2016). The phosphates of these compounds crystallize in the non-centrosymmetric *Cm* space group while the arsenates adopt the *C*2/*m* space group.

## 4. Synthesis and crystallization

Single crystals of $K_3Ni_6Fe(PO_4)_6$ were prepared by solid-state reaction in air. A mixture of $K_2CO_3$, $Ni(NO_3)_2{\cdot}6H_2O$, $Fe(NO_3)_3{\cdot}9H_2O$ and $H_3PO_4$ (85 wt.%) reagents with a K:Ni:Fe:P molar ratio of 2:2:1:3 was dissolved in 50 mL of distilled water. The resulting solution was stirred without heating for 24 h and was subsequently evaporated to dryness at 343 K. The obtained dry residue was progressively heated in a platinum crucible up to 673 K in order to eliminate volatile products. In a second step, the powder was homogenized in an agate mortar and then progressively heated to 1303 K. Kept at this temperature for 2 h, the reaction mixture then underwent slow cooling at a rate of 5 Kh$^{-1}$ to 1103 K and then to room temperature with the furnace inertia. After washing with distilled water, the obtained crystals were brown with block-type shape. A qualitative EDX analysis (energy dispersive X-ray spectroscopy) detected the presence of the expected chemical elements corresponding to K, Ni, Fe, P and O atoms (see Fig. 6).

## 5. Refinement

Crystal data, data collection and structure refinement details of $K_3Ni_6Fe(PO_4)_6$ are summarized in Table 1. The highest peak and the deepest hole in the final Fourier map are at 0.71 and 0.59 Å, respectively, from atom K2.

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# supporting information

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## Crystal structure of a new tripotassium hexanickel iron hexaphosphate

**Said Ouaatta, Abderrazzak Assani, Mohamed Saadi and Lahcen El Ammari**

### Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT-Plus* (Bruker, 2016); data reduction: *SAINT-Plus* (Bruker, 2016); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

### Tripotassium hexanickel iron hexaphosphate

*Crystal data*

$K_3Ni_6Fe(PO_4)_6$
$M_r$ = 1095.23
Monoclinic, *C*2/*m*
*a* = 10.6853 (4) Å
*b* = 14.1009 (5) Å
*c* = 6.5481 (2) Å
*β* = 103.842 (1)°
*V* = 957.97 (6) Å$^3$
*Z* = 2
*F*(000) = 1066
$D_x$ = 3.797 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1981 reflections
*θ* = 2.4–33.7°
*μ* = 7.79 mm$^{-1}$
*T* = 296 K
Block, brown
0.36 × 0.27 × 0.20 mm

*Data collection*

Bruker D8 VENTURE Super DUO diffractometer
Radiation source: INCOATEC I*μ*S micro-focus source
HELIOS mirror optics monochromator
Detector resolution: 10.4167 pixels mm$^{-1}$
*φ* and *ω* scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.638, $T_{max}$ = 0.746
13870 measured reflections
1981 independent reflections
1891 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021
$\theta_{max}$ = 33.7°, $\theta_{min}$ = 2.4°
*h* = −14→16
*k* = −22→22
*l* = −10→10

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.062
*S* = 1.07
1981 reflections
112 parameters
0 restraints
$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 6.1957P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.16 e Å$^{-3}$

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Ni1 | 0.33034 (2) | 0.69342 (2) | 0.18429 (4) | 0.00599 (6) | |
| Ni2 | 0.500000 | 0.87799 (2) | 1.000000 | 0.00683 (7) | |
| Fe1 | 0.500000 | 0.500000 | 0.000000 | 0.00327 (9) | |
| K1 | 0.40983 (18) | 0.500000 | 0.4730 (2) | 0.0200 (3) | 0.472 |
| K2 | 0.38224 (13) | 0.90311 (11) | 0.48618 (19) | 0.0242 (3) | 0.417 |
| K3 | 0.1849 (7) | 0.500000 | 0.4893 (8) | 0.0560 (19) | 0.192 |
| P1 | 0.41063 (4) | 0.69522 (3) | 0.72442 (7) | 0.00485 (8) | |
| P2 | 0.19569 (6) | 0.500000 | −0.01061 (10) | 0.00557 (11) | |
| O1 | 0.31025 (13) | 0.70594 (10) | 0.8587 (2) | 0.0072 (2) | |
| O2 | 0.34525 (14) | 0.68172 (11) | 0.4959 (2) | 0.0102 (2) | |
| O3 | 0.50488 (13) | 0.60977 (10) | 0.7970 (2) | 0.0069 (2) | |
| O4 | 0.50525 (13) | 0.78185 (10) | 0.7692 (2) | 0.0082 (2) | |
| O5 | 0.19430 (13) | 0.58983 (10) | 0.1265 (2) | 0.0085 (2) | |
| O6 | 0.06136 (18) | 0.500000 | −0.1681 (3) | 0.0087 (3) | |
| O7 | 0.30728 (19) | 0.500000 | −0.1148 (3) | 0.0103 (3) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.00441 (10) | 0.00697 (10) | 0.00638 (10) | 0.00038 (7) | 0.00088 (7) | −0.00015 (7) |
| Ni2 | 0.00519 (13) | 0.00602 (14) | 0.00944 (14) | 0.000 | 0.00207 (10) | 0.000 |
| Fe1 | 0.00218 (18) | 0.00293 (18) | 0.00478 (19) | 0.000 | 0.00099 (15) | 0.000 |
| K1 | 0.0413 (9) | 0.0080 (5) | 0.0103 (5) | 0.000 | 0.0055 (5) | 0.000 |
| K2 | 0.0259 (6) | 0.0332 (7) | 0.0122 (4) | −0.0107 (5) | 0.0018 (4) | 0.0082 (4) |
| K3 | 0.056 (4) | 0.100 (6) | 0.0100 (17) | 0.000 | 0.005 (2) | 0.000 |
| P1 | 0.00395 (17) | 0.00570 (18) | 0.00483 (18) | 0.00067 (13) | 0.00093 (14) | −0.00035 (13) |
| P2 | 0.0033 (2) | 0.0049 (2) | 0.0082 (3) | 0.000 | 0.00071 (19) | 0.000 |
| O1 | 0.0055 (5) | 0.0095 (5) | 0.0072 (5) | 0.0022 (4) | 0.0028 (4) | 0.0009 (4) |
| O2 | 0.0091 (6) | 0.0162 (6) | 0.0048 (5) | 0.0011 (5) | 0.0003 (4) | 0.0002 (4) |
| O3 | 0.0049 (5) | 0.0070 (5) | 0.0089 (5) | 0.0014 (4) | 0.0018 (4) | 0.0012 (4) |
| O4 | 0.0069 (5) | 0.0072 (5) | 0.0110 (6) | −0.0012 (4) | 0.0031 (4) | −0.0022 (4) |
| O5 | 0.0071 (5) | 0.0067 (5) | 0.0120 (6) | −0.0012 (4) | 0.0027 (4) | −0.0031 (4) |
| O6 | 0.0045 (7) | 0.0124 (8) | 0.0081 (8) | 0.000 | −0.0005 (6) | 0.000 |
| O7 | 0.0056 (7) | 0.0088 (8) | 0.0181 (9) | 0.000 | 0.0060 (7) | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ni1—O2 | 2.0153 (14) | K1—O7$^{xi}$ | 3.146 (3) |
| Ni1—O5 | 2.0314 (14) | K2—O4 | 2.631 (2) |

| | | | |
|---|---|---|---|
| Ni1—O1$^{i}$ | 2.0366 (13) | K2—O6$^{xii}$ | 2.677 (2) |
| Ni1—O3$^{ii}$ | 2.0984 (14) | K2—O2$^{i}$ | 2.737 (2) |
| Ni1—O1$^{iii}$ | 2.0988 (14) | K2—O5$^{i}$ | 2.8470 (19) |
| Ni1—O4$^{ii}$ | 2.1161 (14) | K2—O4$^{ii}$ | 2.851 (2) |
| Ni2—O4 | 2.0411 (14) | K2—O6$^{vi}$ | 2.929 (2) |
| Ni2—O4$^{iv}$ | 2.0411 (14) | K2—O1$^{i}$ | 3.0776 (19) |
| Ni2—O5$^{v}$ | 2.0928 (14) | K2—O7$^{xii}$ | 3.086 (2) |
| Ni2—O5$^{i}$ | 2.0928 (14) | K2—O2 | 3.149 (2) |
| Ni2—O6$^{i}$ | 2.2241 (13) | K3—O7$^{xi}$ | 2.609 (6) |
| Ni2—O6$^{vi}$ | 2.2241 (13) | K3—O5 | 2.715 (5) |
| Fe1—O7 | 2.016 (2) | K3—O5$^{x}$ | 2.715 (5) |
| Fe1—O7$^{vii}$ | 2.016 (2) | K3—O6$^{xi}$ | 2.862 (6) |
| Fe1—O3$^{viii}$ | 2.0490 (13) | K3—O6$^{xiii}$ | 2.949 (7) |
| Fe1—O3$^{ii}$ | 2.0490 (13) | K3—O2 | 3.077 (4) |
| Fe1—O3$^{ix}$ | 2.0490 (13) | K3—O2$^{x}$ | 3.077 (4) |
| Fe1—O3$^{iii}$ | 2.0490 (13) | P1—O2 | 1.5042 (15) |
| K1—O3 | 2.6263 (18) | P1—O1 | 1.5482 (14) |
| K1—O3$^{x}$ | 2.6264 (18) | P1—O4 | 1.5679 (14) |
| K1—O2 | 2.6673 (16) | P1—O3 | 1.5694 (14) |
| K1—O2$^{x}$ | 2.6673 (16) | P2—O7 | 1.509 (2) |
| K1—O3$^{ix}$ | 2.6691 (19) | P2—O6 | 1.554 (2) |
| K1—O3$^{ii}$ | 2.6691 (19) | P2—O5$^{x}$ | 1.5546 (14) |
| K1—O5 | 3.091 (2) | P2—O5 | 1.5546 (14) |
| K1—O5$^{x}$ | 3.091 (2) | | |

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| O2—Ni1—O5 | 90.55 (6) | O3$^{ix}$—K1—O5 | 93.66 (6) |
| O2—Ni1—O1$^{i}$ | 94.23 (6) | O3$^{ii}$—K1—O5 | 65.70 (5) |
| O5—Ni1—O1$^{i}$ | 90.23 (6) | O3—K1—O5$^{x}$ | 155.60 (8) |
| O2—Ni1—O3$^{ii}$ | 91.89 (6) | O3$^{x}$—K1—O5$^{x}$ | 115.32 (5) |
| O5—Ni1—O3$^{ii}$ | 99.19 (5) | O2—K1—O5$^{x}$ | 106.12 (6) |
| O1$^{i}$—Ni1—O3$^{ii}$ | 168.72 (5) | O2$^{x}$—K1—O5$^{x}$ | 59.36 (5) |
| O2—Ni1—O1$^{iii}$ | 178.70 (6) | O3$^{ix}$—K1—O5$^{x}$ | 65.70 (5) |
| O5—Ni1—O1$^{iii}$ | 88.65 (6) | O3$^{ii}$—K1—O5$^{x}$ | 93.66 (6) |
| O1$^{i}$—Ni1—O1$^{iii}$ | 84.74 (6) | O5—K1—O5$^{x}$ | 48.38 (6) |
| O3$^{ii}$—Ni1—O1$^{iii}$ | 89.25 (5) | O3—K1—O7$^{xi}$ | 56.89 (5) |
| O2—Ni1—O4$^{ii}$ | 92.33 (6) | O3$^{x}$—K1—O7$^{xi}$ | 56.89 (5) |
| O5—Ni1—O4$^{ii}$ | 169.40 (5) | O2—K1—O7$^{xi}$ | 78.69 (5) |
| O1$^{i}$—Ni1—O4$^{ii}$ | 99.72 (5) | O2$^{x}$—K1—O7$^{xi}$ | 78.69 (5) |
| O3$^{ii}$—Ni1—O4$^{ii}$ | 70.53 (5) | O3$^{ix}$—K1—O7$^{xi}$ | 144.33 (3) |
| O1$^{iii}$—Ni1—O4$^{ii}$ | 88.64 (5) | O3$^{ii}$—K1—O7$^{xi}$ | 144.33 (3) |
| O4—Ni2—O4$^{iv}$ | 96.75 (8) | O5—K1—O7$^{xi}$ | 106.17 (7) |
| O4—Ni2—O5$^{v}$ | 103.69 (6) | O5$^{x}$—K1—O7$^{xi}$ | 106.17 (7) |
| O4$^{iv}$—Ni2—O5$^{v}$ | 92.95 (5) | O4—K2—O6$^{xii}$ | 135.41 (8) |
| O4—Ni2—O5$^{i}$ | 92.95 (5) | O4—K2—O2$^{i}$ | 89.14 (6) |
| O4$^{iv}$—Ni2—O5$^{i}$ | 103.69 (6) | O6$^{xii}$—K2—O2$^{i}$ | 128.60 (7) |
| O5$^{v}$—Ni2—O5$^{i}$ | 154.96 (8) | O4—K2—O5$^{i}$ | 66.22 (5) |
| O4—Ni2—O6$^{i}$ | 160.71 (6) | O6$^{xii}$—K2—O5$^{i}$ | 147.28 (7) |
| O4$^{iv}$—Ni2—O6$^{i}$ | 94.81 (6) | O2$^{i}$—K2—O5$^{i}$ | 61.95 (5) |

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| O5$^{v}$—Ni2—O6$^{i}$ | 91.06 (6) | O4—K2—O4$^{ii}$ | 79.26 (7) |
| O5$^{i}$—Ni2—O6$^{i}$ | 69.28 (6) | O6$^{xii}$—K2—O4$^{ii}$ | 69.19 (5) |
| O4—Ni2—O6$^{vi}$ | 94.80 (6) | K2$^{xiv}$—K2—O4$^{ii}$ | 126.86 (4) |
| O4$^{iv}$—Ni2—O6$^{vi}$ | 160.71 (6) | O2$^{i}$—K2—O4$^{ii}$ | 105.45 (6) |
| O5$^{v}$—Ni2—O6$^{vi}$ | 69.28 (6) | O5$^{i}$—K2—O4$^{ii}$ | 142.65 (7) |
| O5$^{i}$—Ni2—O6$^{vi}$ | 91.06 (6) | O4—K2—O6$^{vi}$ | 68.58 (5) |
| O6$^{i}$—Ni2—O6$^{vi}$ | 78.66 (8) | O6$^{xii}$—K2—O6$^{vi}$ | 97.82 (7) |
| O7—Fe1—O7$^{vii}$ | 180.0 | O2$^{i}$—K2—O6$^{vi}$ | 126.41 (7) |
| O7—Fe1—O3$^{viii}$ | 86.58 (5) | O5$^{i}$—K2—O6$^{vi}$ | 64.47 (5) |
| O7$^{vii}$—Fe1—O3$^{viii}$ | 93.42 (5) | O4$^{ii}$—K2—O6$^{vi}$ | 116.35 (6) |
| O7—Fe1—O3$^{ii}$ | 93.42 (5) | O4—K2—O1$^{i}$ | 109.01 (6) |
| O7$^{vii}$—Fe1—O3$^{ii}$ | 86.58 (5) | O6$^{xii}$—K2—O1$^{i}$ | 85.38 (5) |
| O3$^{viii}$—Fe1—O3$^{ii}$ | 180.00 (8) | O2$^{i}$—K2—O1$^{i}$ | 50.84 (4) |
| O7—Fe1—O3$^{ix}$ | 93.42 (5) | O5$^{i}$—K2—O1$^{i}$ | 112.77 (6) |
| O7$^{vii}$—Fe1—O3$^{ix}$ | 86.58 (5) | O4$^{ii}$—K2—O1$^{i}$ | 64.63 (5) |
| O3$^{viii}$—Fe1—O3$^{ix}$ | 81.87 (8) | O6$^{vi}$—K2—O1$^{i}$ | 176.79 (6) |
| O3$^{ii}$—Fe1—O3$^{ix}$ | 98.13 (8) | O4—K2—O7$^{xii}$ | 165.00 (7) |
| O7—Fe1—O3$^{iii}$ | 86.58 (5) | O6$^{xii}$—K2—O7$^{xii}$ | 52.30 (6) |
| O7$^{vii}$—Fe1—O3$^{iii}$ | 93.42 (5) | O2$^{i}$—K2—O7$^{xii}$ | 78.76 (5) |
| O3$^{viii}$—Fe1—O3$^{iii}$ | 98.13 (8) | O5$^{i}$—K2—O7$^{xii}$ | 114.35 (7) |
| O3$^{ii}$—Fe1—O3$^{iii}$ | 81.87 (8) | O4$^{ii}$—K2—O7$^{xii}$ | 95.32 (5) |
| O3$^{ix}$—Fe1—O3$^{iii}$ | 180.00 (5) | O6$^{vi}$—K2—O7$^{xii}$ | 125.92 (6) |
| O2—P1—O1 | 110.92 (8) | O1$^{i}$—K2—O7$^{xii}$ | 56.34 (4) |
| O2—P1—O4 | 114.19 (8) | O4—K2—O2 | 52.08 (5) |
| O1—P1—O4 | 108.73 (8) | O6$^{xii}$—K2—O2 | 125.05 (6) |
| O2—P1—O3 | 108.41 (8) | O2$^{i}$—K2—O2 | 56.55 (6) |
| O1—P1—O3 | 112.61 (8) | O5$^{i}$—K2—O2 | 87.27 (5) |
| O4—P1—O3 | 101.72 (8) | O4$^{ii}$—K2—O2 | 59.33 (5) |
| O7—P2—O6 | 113.84 (12) | O6$^{vi}$—K2—O2 | 120.57 (5) |
| O7—P2—O5$^{x}$ | 112.26 (7) | O1$^{i}$—K2—O2 | 56.94 (4) |
| O6—P2—O5$^{x}$ | 104.37 (7) | O7$^{xii}$—K2—O2 | 113.14 (5) |
| O7—P2—O5 | 112.26 (7) | O7$^{xi}$—K3—O5 | 139.0 (2) |
| O6—P2—O5 | 104.37 (7) | O7$^{xi}$—K3—O5$^{x}$ | 139.0 (2) |
| O5$^{x}$—P2—O5 | 109.14 (11) | O5—K3—O5$^{x}$ | 55.61 (11) |
| O3—K1—O3$^{x}$ | 72.22 (7) | O7$^{xi}$—K3—O6$^{xi}$ | 55.72 (11) |
| O3—K1—O2 | 56.19 (4) | O5—K3—O6$^{xi}$ | 143.7 (2) |
| O3$^{x}$—K1—O2 | 125.17 (6) | O5$^{x}$—K3—O6$^{xi}$ | 143.7 (2) |
| O3—K1—O2$^{x}$ | 125.17 (6) | O7$^{xi}$—K3—O6$^{xiii}$ | 149.1 (3) |
| O3$^{x}$—K1—O2$^{x}$ | 56.19 (4) | O5—K3—O6$^{xiii}$ | 65.77 (12) |
| O2—K1—O2$^{x}$ | 147.77 (10) | O5$^{x}$—K3—O6$^{xiii}$ | 65.77 (12) |
| O3—K1—O3$^{ix}$ | 138.56 (8) | O6$^{xi}$—K3—O6$^{xiii}$ | 93.4 (2) |
| O3$^{x}$—K1—O3$^{ix}$ | 93.79 (6) | O7$^{xi}$—K3—O2 | 80.84 (14) |
| O2—K1—O3$^{ix}$ | 136.75 (7) | O5—K3—O2 | 59.12 (9) |
| O2$^{x}$—K1—O3$^{ix}$ | 67.30 (4) | O5$^{x}$—K3—O2 | 105.29 (19) |
| O3—K1—O3$^{ii}$ | 93.79 (6) | O6$^{xi}$—K3—O2 | 110.40 (11) |
| O3$^{x}$—K1—O3$^{ii}$ | 138.56 (8) | O6$^{xiii}$—K3—O2 | 114.13 (11) |
| O2—K1—O3$^{ii}$ | 67.30 (4) | O7$^{xi}$—K3—O2$^{x}$ | 80.84 (14) |
| O2$^{x}$—K1—O3$^{ii}$ | 136.75 (7) | O5—K3—O2$^{x}$ | 105.29 (19) |

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| O3$^{ix}$—K1—O3$^{ii}$ | 70.89 (7) | O5$^{x}$—K3—O2$^{x}$ | 59.12 (9) |
| O3—K1—O5 | 115.32 (5) | O6$^{xi}$—K3—O2$^{x}$ | 110.40 (11) |
| O3$^{x}$—K1—O5 | 155.60 (8) | O6$^{xiii}$—K3—O2$^{x}$ | 114.13 (11) |
| O2—K1—O5 | 59.36 (5) | O2—K3—O2$^{x}$ | 112.8 (2) |
| O2$^{x}$—K1—O5 | 106.12 (6) | | |

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+1, *y*, −*z*+1; (iii) *x*, *y*, *z*−1; (iv) −*x*+1, *y*, −*z*+2; (v) *x*+1/2, −*y*+3/2, *z*+1; (vi) *x*+1/2, *y*+1/2, *z*+1; (vii) −*x*+1, −*y*+1, −*z*; (viii) *x*, −*y*+1, *z*−1; (ix) −*x*+1, −*y*+1, −*z*+1; (x) *x*, −*y*+1, *z*; (xi) *x*, *y*, *z*+1; (xii) −*x*+1/2, −*y*+3/2, −*z*; (xiii) −*x*, −*y*+1, −*z*; (xiv) *x*, −*y*+2, *z*.