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Crystal structure of $K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot 0.5H_2O$, an open-framework iron phosphite with mixed-valent Fe^{II}/Fe^{III} ions

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Single crystals of the title compound, potassium hexaphosphitopentaferrate(II,III) hemihydrate, $K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot 0.5H_2O$, were grown under mild hydrothermal conditions. The crystal structure is isotypic with $Li_{1.43}[Fe^{II}_{4.43}Fe^{III}_{0.57}(HPO_3)_6]\cdot 1.5H_2O$ and $(NH_4)_2[Fe^{II}_5(HPO_3)_6]$ and exhibits a $[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]^{0.75-}$ open framework with disordered K^+ (occupancy 3/4) as counter-cations. The anionic framework is based on (001) sheets of two $[FeO_6]$ octahedra (one with point group symmetry 3.. and one with point group symmetry .2.) linked along [001] through $[HPO_3]^{2-}$ oxoanions. Each sheet is constructed from 12-membered rings of edge-sharing $[FeO_6]$ octahedra, giving rise to channels with a radius of ca 3.1 Å where the K^+ cations and likewise disordered water molecules (occupancy 1/4) are located. O···O contacts between the water molecule and framework O atoms of 2.864 (5) Å indicate hydrogen-bonding interactions of medium strength. The infrared spectrum of the compound shows vibrational bands typical for phosphite and water groups. The Mössbauer spectrum is in accordance with the presence of Fe^{II} and Fe^{III} ions.

1. Chemical context

Open-framework materials have been a major research topic in materials science during the last decades because of their potential applications (Barrer, 1982; Wilson *et al.*, 1982; Davis, 2002, Adams & Pendlebury, 2011). Many efforts have been made to obtain porous materials using different oxoanions in combination with metals (Yu & Xu, 2010). The use of structure-directing agents or templates, not only organic but also inorganic, has also been extended in order to achieve this purpose. In this context, a new porous mixed-valent Fe^{II}/Fe^{III} phosphitoferrate with lithium cations and an open-framework structure, $Li_{1.43}[Fe^{II}_{4.43}Fe^{III}_{0.57}(HPO_3)_6]\cdot 1.5H_2O$, has been reported (Chung *et al.*, 2011). This structure presents channels of ca 5.5 Å diameter along the [001] direction in which water molecules and lithium ions are located. The same type of framework but with Fe^{II} cations and with ammonium counteranions was reported recently for $(NH_4)_2[Fe^{II}_5(HPO_3)_6]$ (Berrocal *et al.*, 2014).

Here we report on the synthesis and crystal structure of isotypic $K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot 0.5H_2O$ resulting from the replacement of lithium/ammonium by potassium. The iron cations in this compound are again in a mixed valence oxidation state of +II and +III.
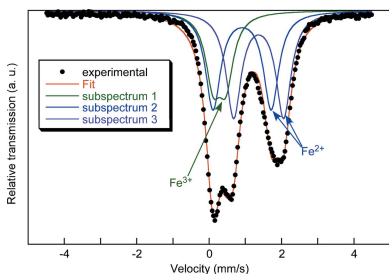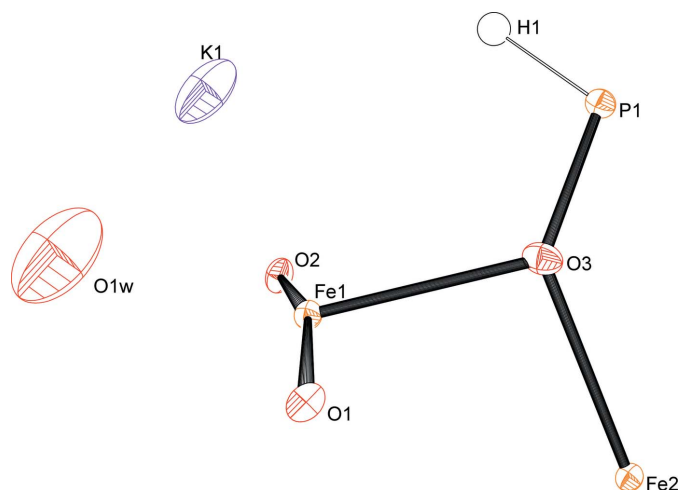
Figure 1
Asymmetric unit of $K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot0.5H_2O$ with displacement parameters drawn at the 50% probability level.

Figure 2
Mössbauer spectrum of the title compound showing the presence of Fe^{II} and Fe^{III}. The fit was made with the *NORMOS* program (Brand *et al.*, 1983).

2. Structural commentary

The asymmetric unit of $K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot0.5H_2O$ (Fig. 1) contains two Fe sites on special positions (6*f* and 4*d*) with site symmetries of .2. and .3., respectively, three O sites, one P site and one H site. In addition, disordered sites associated with a water molecule (O1*W*) and the potassium counter-cation are present. The crystal structure is made up of two types of [FeO$_6$] octahedra linked *via* edge-sharing into sheets parallel to (001). These sheets consist of 12-membered rings formed by six [Fe1O$_6$] octahedra and six [Fe2O$_6$] octahedra. In one of the FeO$_6$ octahedra (Fe1), the Fe—O bond lengths range from 2.046 (2) to 2.179 (2) Å while in the [Fe2O$_6$] octahedron, a more uniform bond-length distribution from 2.134 (2) to 2.143 (2) is observed. In order to assign the content of Fe^{II} and Fe^{III} on these sites, a Mössbauer spectrum was recorded (Fig. 2). Three different components were observed, two doublets, corresponding to Fe^{II} cations, and a third doublet, corresponding to Fe^{III} cations. The determined Fe^{II}/Fe^{III} ratio is 3.1, in good agreement with the formula. According to bond-valence calculations (Brown, 2002), a clear assignment of which of the two iron sites carries the Fe^{III} cations cannot be made. The calculated bond-valence sum for site Fe1 assuming Fe^{II} is 2.213 valence units (v.u.), while assuming Fe^{III} gives 2.367. Corresponding values for the Fe2 site are 2.014 v.u. assuming Fe^{II} and 2.155 assuming Fe^{III}. The O—Fe—O bond angles of the two [FeO$_6$] octahedra are in the range between 78.10 (8) and 102.63 (7)° for *cis*- and between 175.77 (11) and 163.23 (8)° for the *trans*-angles.

The (001) iron oxide sheets are linked through phosphite groups whereby six anions share the innermost oxygen atoms of each ring (Fig. 3), forming 12-membered channels extending along [001]. The channels have a radius of about 3.1 Å. The P—O bond lengths of the anion range from 1.529 (2) to 1.541 (2) Å and are comparable with those of the two isotypic structures. The P—H distance in the title compound is 1.29 (4) Å, and the O—P—O bond angles range from 110.24 (11) to 114.32 (11)°.
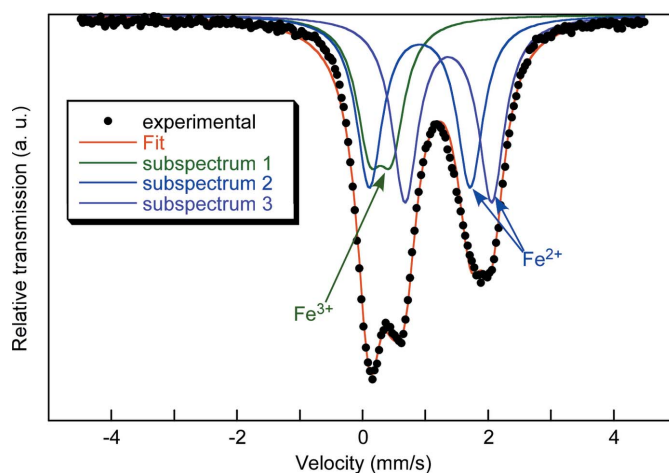
The disordered potassium cations and water molecules are located on special positions in the twelve-membered channels of the framework with site symmetries of 32. and 3.., respectively. The occupancy factors are 0.75 for potassium and 0.25 for the water molecule. Although the hydrogen atoms of the water molecule could not be located, the O$\cdots$O distance of 2.864 (5) Å between the water O1*W* atom and the O1 atom of the framework indicates possible hydrogen-bonding interactions of medium strength. Because the O1*W* site is located on a threefold rotation axis, three hydrogen bonds with the inorganic skeleton with an angle of 113.42 (5)° are possible.

3. Synthesis and characterization

$K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot0.5H_2O$ was synthesized under mild hydrothermal conditions and autogeneous pressure
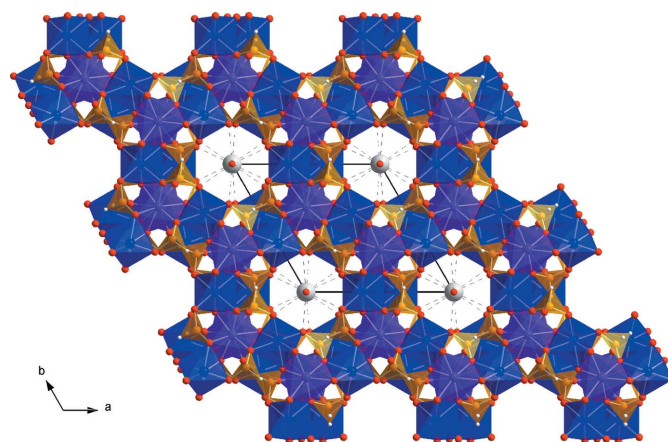
Figure 3
Crystal structure of $K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot0.5H_2O$ in polyhedral representation, in a projection along [001]. Colour code: Fe1O$_6$ octahedra are blue, Fe2O$_6$ octahedra are magenta, HPO$_3$ tetrahedra are orange, O atoms are red and K$^+$ ions are grey. Hydrogen-bonding interactions between O1 from the framework and O1*W* are shown with dashed lines.

Table 1
Experimental details.

Crystal data	
Chemical formula	$K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot 0.5H_2O$
M_r	797.45
Crystal system, space group	Trigonal, $P\bar{3}c1$
Temperature (K)	100
a, c (Å)	10.1567 (5), 9.2774 (6)
V (Å^3)	828.82 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	5.14
Crystal size (mm)	$0.29 \times 0.05 \times 0.04$
Data collection	
Diffractometer	Agilent SuperNova
Absorption correction	Analytical (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.423, 0.845
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5132, 647, 618
R_{int}	0.026
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.664
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.026, 0.061, 1.19
No. of reflections	647
No. of parameters	54
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.8, −0.50

Computer programs: *CrysAlis PRO* (Agilent, 2014), *OLEX2* (Dolomanov, 2009), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2001) and *WinGX* (Farrugia, 2012).

(10–20 bar at 343 K). The reaction mixture was prepared from 30 ml water, 2 ml of hypophosphorous acid, 0.17 mmol of KOH and 0.37 mmol of $FeCl_3\cdot 6H_2O$. The mixture had a pH value of $\simeq$ 3.0. The reaction mixture was sealed in a polytetrafluoroethylene (PTFE)-lined steel pressure vessel, which was maintained at 343 K for five days. This procedure allowed the formation of single crystals of the title compound with a dark green to black colour.

The IR spectrum (see supporting information for this submission) shows typical bands corresponding to the stretching and deformation mode of the water molecules at 3235 and 2410 cm^{-1}, respectively. The spectrum also shows the stretching and deformation modes of the P—H bond at 1750 cm^{-1}. The bands corresponding to the symmetric (ν_s) and antisymmetric (ν_{as}) stretching vibrational modes of the (PO_3) groups appear at 930 and 1151 cm^{-1}, whereas the symmetric (δ_s) and antisymmetric (δ_{as}) deformation modes of this group are centred at 450 and 590 cm^{-1} (Nakamoto, 1997; Chung *et al.*, 2011).

Thermogravimetric analysis of the title compound (see supporting information for this submission) shows a first mass-loss process of 1.05% between room temperature and 498 K. This mass loss corresponds to the removal of water (theoretical value: 1.13%). Between 498 K and 673 K, another mass loss of 0.45% takes place which could not be assigned to a chemical reaction. This second process is followed by a third

continuous process associated with a considerable gain of mass due to the oxidation of the compound.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms of the water molecule were not modelled. The hydrogen atom of the phosphite group was located in a difference density map and was refined without any constraint. Potassium and water oxygen sites are located in the channels. The occupancy factors of both atoms were initially set taking into account the previous character-ization (themogravimetric measurement, Mössbauer spectrum fit). Some trials to refine the occupancy factors of these atoms were made. However, the results were very similar to those initially set, with a slight increase of reliability factors. Therefore, for the final model the occupancy factors were fixed at 0.75 for the K1 and at 0.25 for the O1*W* site.

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supporting information

Crystal structure of $K_{0.75}[Fe^{II}_{3.75}Fe^{III}_{1.25}(HPO_3)_6]\cdot0.5H_2O$, an open-framework iron phosphite with mixed-valent Fe^{II}/Fe^{III} ions

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *OLEX2* (Dolomanov, 2009); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Potassium hexaphosphitopentaferrate(II,III) hemihydrate

Crystal data

$K_{0.75}[FeII3.75FeIII_{1.25}(HPO_3)_6]\cdot0.5H_2O$	$D_x = 3.195$ Mg m^{-3}
$M_r = 797.45$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Trigonal, $P\bar{3}c1$	Cell parameters from 3002 reflections
Hall symbol: -P 3 2"c	$\theta = 2.3–28.0°$
$a = 10.1567\,(5)$ Å	$\mu = 5.14$ mm^{-1}
$c = 9.2774\,(6)$ Å	$T = 100$ K
$V = 828.82\,(8)$ Å^3	Prism, black
$Z = 2$	$0.29 \times 0.05 \times 0.04$ mm
$F(000) = 779$	

Data collection

Agilent SuperNova diffractometer	$T_{min} = 0.423$, $T_{max} = 0.845$
Radiation source: Nova (Mo) X-ray micro-source	5132 measured reflections
Multilayer optics monochromator	647 independent reflections
Detector resolution: 16.2439 pixels mm^{-1}	618 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.026$
Absorption correction: analytical	$\theta_{max} = 28.2°$, $\theta_{min} = 2.3°$
(*CrysAlis PRO*; Agilent, 2014)	$h = -13\rightarrow12$
	$k = -9\rightarrow13$
	$l = -12\rightarrow10$

Refinement

Refinement on F^2	0 restraints
Least-squares matrix: full	Primary atom site location: iterative
$R[F^2 > 2\sigma(F^2)] = 0.026$	Secondary atom site location: difference Fourier map
$wR(F^2) = 0.061$	
$S = 1.19$	Hydrogen site location: difference Fourier map
647 reflections	All H-atom parameters refined
54 parameters	

$w = 1/[\sigma^2(F_o^2) + (0.0254P)^2 + 2.2546P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.015$

$\Delta\rho_{max} = 0.8$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	$U_{iso}*/U_{eq}$	Occ. (<1)
Fe1	0.62108 (5)	0	0.25	0.00709 (16)	
Fe2	0.6667	0.3333	0.33159 (7)	0.00659 (18)	
P1	0.70307 (8)	0.11196 (8)	0.58780 (7)	0.00748 (18)	
O3	0.6916 (2)	0.1569 (2)	0.4316 (2)	0.0110 (4)	
O2	0.3954 (2)	−0.1473 (2)	0.3120 (2)	0.0095 (4)	
O1	0.8210 (2)	0.1342 (2)	0.1437 (2)	0.0124 (4)	
K1	1	0	0.25	0.0247 (5)	0.75
O1W	1	0	0.063 (2)	0.033 (4)	0.25
H1	0.645 (4)	−0.033 (4)	0.592 (4)	0.011 (9)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0070 (2)	0.0067 (3)	0.0075 (3)	0.00336 (14)	−0.00016 (10)	−0.00031 (19)
Fe2	0.0062 (2)	0.0062 (2)	0.0074 (3)	0.00310 (11)	0	0
P1	0.0072 (3)	0.0087 (3)	0.0068 (3)	0.0042 (3)	0.0006 (2)	0.0003 (2)
O3	0.0112 (10)	0.0147 (10)	0.0083 (9)	0.0075 (8)	0.0018 (7)	0.0024 (8)
O2	0.0091 (9)	0.0079 (9)	0.0113 (9)	0.0041 (8)	0.0023 (7)	−0.0004 (7)
O1	0.0153 (10)	0.0095 (10)	0.0140 (10)	0.0073 (9)	−0.0003 (8)	0.0002 (8)
K1	0.0151 (7)	0.0151 (7)	0.0439 (15)	0.0075 (3)	0	0
O1W	0.018 (5)	0.018 (5)	0.062 (12)	0.009 (2)	0	0

Geometric parameters (Å, º)

Fe1—O1	2.046 (2)	P1—H1	1.29 (4)
Fe1—O1^i	2.046 (2)	O2—P1^{vii}	1.534 (2)
Fe1—O2	2.096 (2)	O2—Fe2^{viii}	2.134 (2)
Fe1—O2^i	2.096 (2)	O1—P1^{ix}	1.529 (2)
Fe1—O3^i	2.179 (2)	O1—K1	2.935 (2)
Fe1—O3	2.179 (2)	K1—O1^x	2.935 (2)
Fe1—K1	3.8486 (6)	K1—O1^{xi}	2.935 (2)
Fe2—O2^i	2.134 (2)	K1—O1^{xii}	2.935 (2)

Fe2—O2ⁱⁱ	2.134 (2)	K1—O1ˣⁱⁱⁱ	2.935 (2)
Fe2—O2ⁱⁱⁱ	2.134 (2)	K1—O1ⁱ	2.935 (2)
Fe2—O3	2.143 (2)	K1—Fe1ˣⁱⁱ	3.8486 (6)
Fe2—O3ⁱᵛ	2.143 (2)	K1—Fe1ˣ	3.8486 (6)
Fe2—O3ᵛ	2.143 (2)	K1—K1ˣⁱᵛ	4.6387 (3)
P1—O1ᵛⁱ	1.529 (2)	K1—K1ˣᵛ	4.6387 (3)
P1—O2ᵛⁱⁱ	1.534 (2)	O1W—O1Wˣᵛ	1.17 (4)
P1—O3	1.541 (2)		
O1—Fe1—O1ⁱ	97.50 (12)	O1ˣⁱ—K1—O1ˣⁱⁱ	63.23 (8)
O1—Fe1—O2	167.07 (8)	O1ˣ—K1—O1ˣⁱⁱⁱ	63.23 (8)
O1ⁱ—Fe1—O2	89.81 (8)	O1ˣⁱ—K1—O1ˣⁱⁱⁱ	109.30 (4)
O1—Fe1—O2ⁱ	89.81 (8)	O1ˣⁱⁱ—K1—O1ˣⁱⁱⁱ	78.54 (8)
O1ⁱ—Fe1—O2ⁱ	167.07 (8)	O1ˣ—K1—O1	109.30 (4)
O2—Fe1—O2ⁱ	85.10 (11)	O1ˣⁱ—K1—O1	78.54 (8)
O1—Fe1—O3ⁱ	90.97 (8)	O1ˣⁱⁱ—K1—O1	109.30 (4)
O1ⁱ—Fe1—O3ⁱ	91.82 (8)	O1ˣⁱⁱⁱ—K1—O1	171.12 (8)
O2—Fe1—O3ⁱ	78.11 (8)	O1ˣ—K1—O1ⁱ	78.54 (8)
O2ⁱ—Fe1—O3ⁱ	98.73 (7)	O1ˣⁱ—K1—O1ⁱ	109.30 (4)
O1—Fe1—O3	91.82 (8)	O1ˣⁱⁱ—K1—O1ⁱ	171.12 (8)
O1ⁱ—Fe1—O3	90.97 (8)	O1ˣⁱⁱⁱ—K1—O1ⁱ	109.30 (4)
O2—Fe1—O3	98.73 (7)	O1—K1—O1ⁱ	63.23 (8)
O2ⁱ—Fe1—O3	78.11 (8)	O1ˣ—K1—Fe1ˣⁱⁱ	140.73 (4)
O3ⁱ—Fe1—O3	175.77 (11)	O1ˣⁱ—K1—Fe1ˣⁱⁱ	31.62 (4)
O1—Fe1—K1	48.75 (6)	O1ˣⁱⁱ—K1—Fe1ˣⁱⁱ	31.62 (4)
O1ⁱ—Fe1—K1	48.75 (6)	O1ˣⁱⁱⁱ—K1—Fe1ˣⁱⁱ	94.44 (4)
O2—Fe1—K1	137.45 (6)	O1—K1—Fe1ˣⁱⁱ	94.44 (4)
O2ⁱ—Fe1—K1	137.45 (6)	O1ⁱ—K1—Fe1ˣⁱⁱ	140.73 (4)
O3ⁱ—Fe1—K1	92.11 (5)	O1ˣ—K1—Fe1ˣ	31.62 (4)
O3—Fe1—K1	92.11 (5)	O1ˣⁱ—K1—Fe1ˣ	140.73 (4)
O2ⁱ—Fe2—O2ⁱⁱ	85.13 (8)	O1ˣⁱⁱ—K1—Fe1ˣ	94.44 (4)
O2ⁱ—Fe2—O2ⁱⁱⁱ	85.13 (8)	O1ˣⁱⁱⁱ—K1—Fe1ˣ	31.62 (4)
O2ⁱⁱ—Fe2—O2ⁱⁱⁱ	85.13 (8)	O1—K1—Fe1ˣ	140.73 (4)
O2ⁱ—Fe2—O3	78.10 (8)	O1ⁱ—K1—Fe1ˣ	94.44 (4)
O2ⁱⁱ—Fe2—O3	93.44 (7)	Fe1ˣⁱⁱ—K1—Fe1ˣ	120
O2ⁱⁱⁱ—Fe2—O3	163.22 (8)	O1ˣ—K1—Fe1	94.44 (4)
O2ⁱ—Fe2—O3ⁱᵛ	163.23 (8)	O1ˣⁱ—K1—Fe1	94.44 (4)
O2ⁱⁱ—Fe2—O3ⁱᵛ	78.10 (8)	O1ˣⁱⁱ—K1—Fe1	140.73 (4)
O2ⁱⁱⁱ—Fe2—O3ⁱᵛ	93.44 (7)	O1ˣⁱⁱⁱ—K1—Fe1	140.73 (4)
O3—Fe2—O3ⁱᵛ	102.63 (7)	O1—K1—Fe1	31.62 (4)
O2ⁱ—Fe2—O3ᵛ	93.44 (7)	O1ⁱ—K1—Fe1	31.62 (4)
O2ⁱⁱ—Fe2—O3ᵛ	163.22 (8)	Fe1ˣⁱⁱ—K1—Fe1	120
O2ⁱⁱⁱ—Fe2—O3ᵛ	78.10 (8)	Fe1ˣ—K1—Fe1	120
O3—Fe2—O3ᵛ	102.63 (7)	O1ˣ—K1—K1ˣⁱᵛ	109.64 (4)
O3ⁱᵛ—Fe2—O3ᵛ	102.63 (7)	O1ˣⁱ—K1—K1ˣⁱᵛ	70.36 (4)
O1ᵛⁱ—P1—O2ᵛⁱⁱ	112.13 (11)	O1ˣⁱⁱ—K1—K1ˣⁱᵛ	109.64 (4)
O1ᵛⁱ—P1—O3	114.32 (11)	O1ˣⁱⁱⁱ—K1—K1ˣⁱᵛ	70.36 (4)
O2ᵛⁱⁱ—P1—O3	110.24 (11)	O1—K1—K1ˣⁱᵛ	109.64 (4)

O1^{vi}—P1—H1	105.9 (16)	O1^{i}—K1—K1^{xiv}	70.36 (4)
O2^{vii}—P1—H1	105.6 (16)	Fe1^{xii}—K1—K1^{xiv}	90
O3—P1—H1	108.1 (16)	Fe1^{x}—K1—K1^{xiv}	90
P1—O3—Fe2	135.47 (12)	Fe1—K1—K1^{xiv}	90
P1—O3—Fe1	123.82 (12)	O1^{x}—K1—K1^{xv}	70.36 (4)
Fe2—O3—Fe1	98.26 (8)	O1^{xi}—K1—K1^{xv}	109.64 (4)
P1^{vii}—O2—Fe1	127.45 (12)	O1^{xii}—K1—K1^{xv}	70.36 (4)
P1^{vii}—O2—Fe2^{viii}	130.31 (12)	O1^{xiii}—K1—K1^{xv}	109.64 (4)
Fe1—O2—Fe2^{viii}	101.19 (8)	O1—K1—K1^{xv}	70.36 (4)
P1^{ix}—O1—Fe1	129.84 (13)	O1^{i}—K1—K1^{xv}	109.64 (4)
P1^{ix}—O1—K1	124.86 (11)	Fe1^{xii}—K1—K1^{xv}	90
Fe1—O1—K1	99.63 (7)	Fe1^{x}—K1—K1^{xv}	90
O1^{x}—K1—O1^{xi}	171.12 (8)	Fe1—K1—K1^{xv}	90
O1^{x}—K1—O1^{xii}	109.30 (4)	K1^{xiv}—K1—K1^{xv}	180

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