

Langbeinite-related $K_2FeSn(PO_4)_3$ from single-crystal dataIgor V. Zatonvsky,^{a*} Michael M. Yatskin,^a Vyacheslav N. Baumer,^b Nikolay S. Slobodyanik^a and Oleg V. Shishkin^b^aDepartment of Inorganic Chemistry, Taras Shevchenko National University, 64 Volodymyrska Street, 01033 Kyiv, Ukraine, and ^bSTC Institute for Single Crystals, NAS of Ukraine, 60 Lenin Avenue, 61001 Kharkiv, Ukraine
Correspondence e-mail: zvigoy@yandex.ru

Received 22 October 2007; accepted 13 November 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(P-O) = 0.002$ Å; disorder in main residue; R factor = 0.021; wR factor = 0.045; data-to-parameter ratio = 22.8.

Crystals of dipotassium iron(III) tin(IV) tris(orthophosphate) were grown from a self-flux in the system $K_2O-P_2O_5-Fe_2O_3-SnO_2$. The title compound is isotypic with the mineral langbeinite, $K_2Mg_2(SO_4)_3$. Its three-dimensional $[M_2(PO_4)_3]_\infty$ framework is composed of $[MO_6]$ ($M = Sn, Fe$) octahedra and $[PO_4]$ tetrahedra interlinked *via* vertices. In comparison with the previous refinement from X-ray powder data [Aatiq, Haggouch, Bakri, Lakhdar & Saadouné, (2006). *Powder Diffraction*, **21**, 214–219], the present reinvestigation from single-crystal data allows a more precise determination of the distribution of the Fe and Sn atoms over two crystallographic positions and a revision of the P–O and M–O [$M = K, (Fe, Sn)$] bond lengths. For the two K^+ cations, two different polyhedra with coordination numbers of 9 and 12 are observed. The (Fe, Sn) and K atoms are located on threefold axes.

Related literature

For the previous powder study of the title compound, see Aatiq *et al.* (2006). The crystal structure of the mineral langbeinite was determined by Zemmann & Zemmann (1957). Other phosphates with langbeinite-type structure have been investigated by Norberg (2002), Ogorodnyk *et al.* (2006), Ogorodnyk, Zatonvsky & Slobodyanik (2007), Ogorodnyk, Zatonvsky, Baumer *et al.* (2007), and Orlova *et al.* (2003).

Experimental

Crystal data

$K_2FeSn(PO_4)_3$	$Z = 4$
$M_r = 537.65$	Mo $K\alpha$ radiation
Cubic, $P2_13$	$\mu = 5.47$ mm ⁻¹
$a = 9.91473$ (7) Å	$T = 293$ (2) K
$V = 974.64$ (1) Å ³	$0.08 \times 0.06 \times 0.05$ mm

Data collection

Oxford Diffraction XCalibur-3 diffractometer	5809 measured reflections
Absorption correction: multi-scan (Blessing; 1995)	1434 independent reflections
$T_{\min} = 0.669, T_{\max} = 0.772$	1338 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$	$\Delta\rho_{\max} = 0.50$ e Å ⁻³
$wR(F^2) = 0.045$	$\Delta\rho_{\min} = -0.57$ e Å ⁻³
$S = 1.1$	Absolute structure: Flack (1983), with 621 Friedel Pairs
1434 reflections	Flack parameter: -0.03 (2)
63 parameters	
3 restraints	

Table 1

Selected bond lengths (Å).

K1–O1 ⁱ	2.852 (2)	Fe1–O1 ^v	2.0195 (19)
K1–O2 ⁱⁱ	3.013 (2)	Fe2–O3 ⁱ	1.9830 (19)
K1–O4 ⁱⁱⁱ	3.117 (2)	Fe2–O4	2.010 (2)
K2–O3 ⁱⁱ	2.868 (2)	P1–O4	1.521 (2)
K2–O2 ⁱⁱⁱ	2.960 (2)	P1–O2	1.521 (2)
K2–O4 ⁱⁱⁱ	3.053 (3)	P1–O3	1.525 (2)
K2–O4 ⁱⁱⁱ	3.243 (2)	P1–O1	1.5306 (19)
Fe1–O2 ^{iv}	2.0032 (19)		

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the ICDD for financial support (grant No. 03-02).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2157).

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supplementary materials

Acta Cryst. (2007). E63, i199 [doi:10.1107/S1600536807058588]

Langbeinite-related $K_2FeSn(PO_4)_3$ from single-crystal data

I. V. Zatovsky, M. M. Yatskin, V. N. Baumer, N. S. Slobodyanik and O. V. Shishkin

Comment

Langbeinite-type frameworks $[M_2(PO_4)_3]_\infty$ can be composed from various types of tetravalent and bi- or trivalent metal pairs. For example, the structures of $K_2M_{0.5}Ti_{1.5}(PO_4)_3$ ($M = Ni, Co, Mn$) (Ogorodnyk *et al.*, 2006; 2007a), $K_2MTi(PO_4)_3$ ($M = Y, Yb, Er$) (Norberg, 2002), $K_2FeZr(PO_4)_3$ (Orlova *et al.*, 2003) and $K_2LuZr(PO_4)_3$ (Ogorodnyk *et al.*, 2007b) have been investigated in the past years. Among other langbeinite-related compounds, the structures of only two tin(IV)-containing phosphates, *viz.* $K_2MSn(PO_4)_3$ ($M = Fe, Yb$) refined from X-ray powder data, were previously reported (Aatiq *et al.*, 2006). Herein we report the single-crystal growth and re-investigation of $K_2FeSn(PO_4)_3$ (I), a structure that is isotypic with the mineral langbeinite, $K_2Mg_2(SO_4)_3$ (Zemann, & Zemann, 1957).

Sn and Fe atoms are statistically distributed over two octahedrally coordinated positions with an insignificant priority of the M1 site occupation by Sn and of the M2 site by Fe. The {Sn(Fe)1-Sn(Fe)2-K1—K2} sequence runs along [111] (Fig. 1). K1 atoms are coordinated by 9 O atoms, while K2 atoms are surrounded by 12 O atoms (Fig. 2), with K—O bond lengths ranging from 2.852 (2) to 3.243 (2) Å.

Comparing the results from the single-crystal study with the results of the previous powder study, the following points are notable. The distribution of two type of metals (Fe and Sn) over two octahedrally coordinated positions are close to those reported earlier, but the accuracy of single-crystal refinement is more precise. The values of corresponding Fe/Sn—O bond lengths insignificantly differs from one another in each type of octahedron in the present structure model, while the values found by Rietveld refinement differs a lot. Actually, the new results of the single-crystal refinement suggest a lower degree of distortion of the $[(Fe,Sn)O_6]$ polyhedra. Moreover, in contrast to the results of the powder refinement with each of the K^+ cations in 9-fold coordination, we assume that K1 is 9-coordinate, while K2 is 12-coordinate, which agrees with the most of earlier discussed structures of langbeinite-related phosphates.

Experimental

Crystals of (I) were obtained in the pseudo-quaternary system $K_2O—P_2O_5—SnO_2—Fe_2O_3$ using the high-temperature flux crystallization technique. A well ground mixture of 0.9 g SnO_2 (6.0 mmol), 1.44 g Fe_2O_3 (9.0 mmol), 6.63 g KPO_3 (56.2 mmol) and 1.03 g $K_4P_2O_7$ (3.1 mmol) was placed into a platinum crucible, and was heated up to 1373 K. The melt was exposed during 6 h at this temperature to dissolve the metal oxides, and then was cooled down 1013 K at a rate 25 K/h. At the final stage of crystallization the melt was sustained under isothermal conditions for 1 h to gain equilibrium, and was then poured out onto a copper sheet for quenching. Yellow-brownish crystals with mostly tetrahedral shape were recovered from the remaining flux by leaching out with deionized water.

Refinement

As suggested from the previous powder refinement (Aatiq *et al.*, 2006), the Fe and Sn atoms are statistically distributed. Their coordinates and displacement ellipsoids were constrained. Three additional restraints were applied, *viz.* two for full occupancies of the Fe/Sn sites and one for the total charge of iron and tin (charge sum = 7.00). The highest peak in the final Fourier map is located 0.87 Å from atom P1, and the deepest hole is 1.05 Å from atom Sn1.

Figures

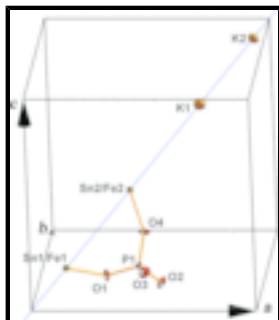


Fig. 1. A part of the crystal structure of (I), displayed with ellipsoids at the 70% probability level. The blue line shows the [111] direction.

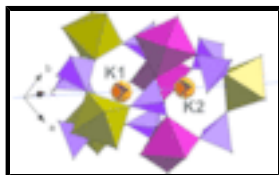


Fig. 2. Arrangement of [MO₆] octahedra and [PO₄] tetrahedra forming cavities around the K⁺ cations (70% probability displacement ellipsoids). [M1O₆] octahedra are light brown, [M2O₆] octahedra are dark red, and [PO₄] tetrahedra are purple.

dipotassium iron(III) tin(IV) tris(orthophosphate)

Crystal data

K₂FeSn(PO₄)₃

M_r = 537.65

Cubic, *P*2₁3

Hall symbol: P 2ac 2ab 3

a = 9.91473 (7) Å

b = 9.91473 (7) Å

c = 9.91473 (7) Å

α = 90°

β = 90°

γ = 90°

V = 974.636 (12) Å³

Z = 4

*F*₀₀₀ = 1020

D_x = 3.664 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 5809 reflections

θ = 2.9–35.0°

μ = 5.47 mm⁻¹

T = 293 (2) K

Tetrahedron, yellow brown

0.08 × 0.06 × 0.05 mm

Data collection

XCalibur-3
diffractometer

Monochromator: graphite

1338 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.035

$T = 293(2)$ K $\theta_{\max} = 35.0^\circ$
 φ and ω scans $\theta_{\min} = 2.9^\circ$
 Absorption correction: multi-scan (Blessing; 1995) $h = -13 \rightarrow 16$
 $T_{\min} = 0.669$, $T_{\max} = 0.772$ $k = -15 \rightarrow 15$
 5809 measured reflections $l = -15 \rightarrow 16$
 1434 independent reflections

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0194P)^2 + 0.5777P]$
 Least-squares matrix: full where $P = (F_o^2 + 2F_c^2)/3$
 $R[F^2 > 2\sigma(F^2)] = 0.021$ $(\Delta/\sigma)_{\max} < 0.001$
 $wR(F^2) = 0.045$ $\Delta\rho_{\max} = 0.50 \text{ e } \text{\AA}^{-3}$
 $S = 1.1$ $\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$
 1434 reflections Extinction correction: SHELXL,
 63 parameters $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 3 restraints Extinction coefficient: 0.0058 (5)
 Absolute structure: Flack (1983), 621 Friedel Pairs
 Flack parameter: -0.03 (2)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
K1	0.70742 (8)	0.70742 (8)	0.70742 (8)	0.0278 (3)	
K2	0.93262 (6)	0.93262 (6)	0.93262 (6)	0.0220 (2)	
Fe1	0.14686 (2)	0.14686 (2)	0.14686 (2)	0.00732 (8)	0.4464 (11)
Fe2	0.41421 (2)	0.41421 (2)	0.41421 (2)	0.00737 (8)	0.5538 (12)
Sn1	0.14686 (2)	0.14686 (2)	0.14686 (2)	0.00732 (8)	0.5536 (11)
Sn2	0.41421 (2)	0.41421 (2)	0.41421 (2)	0.00737 (8)	0.4462 (11)
P1	0.46113 (6)	0.22920 (6)	0.12592 (6)	0.00634 (10)	
O1	0.31483 (18)	0.2389 (2)	0.0776 (2)	0.0154 (4)	
O2	0.5516 (2)	0.2973 (2)	0.0223 (2)	0.0180 (4)	
O3	0.5006 (2)	0.08280 (19)	0.1516 (2)	0.0172 (3)	
O4	0.4825 (2)	0.3057 (2)	0.2572 (2)	0.0203 (4)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0278 (3)	0.0278 (3)	0.0278 (3)	-0.0001 (3)	-0.0001 (3)	-0.0001 (3)

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K2	0.0220 (2)	0.0220 (2)	0.0220 (2)	-0.0026 (2)	-0.0026 (2)	-0.0026 (2)
Fe1	0.00732 (8)	0.00732 (8)	0.00732 (8)	-0.00009 (7)	-0.00009 (7)	-0.00009 (7)
Fe2	0.00737 (8)	0.00737 (8)	0.00737 (8)	-0.00050 (7)	-0.00050 (7)	-0.00050 (7)
Sn1	0.00732 (8)	0.00732 (8)	0.00732 (8)	-0.00009 (7)	-0.00009 (7)	-0.00009 (7)
Sn2	0.00737 (8)	0.00737 (8)	0.00737 (8)	-0.00050 (7)	-0.00050 (7)	-0.00050 (7)
P1	0.0061 (2)	0.0066 (2)	0.0063 (2)	-0.00007 (18)	0.00116 (17)	-0.00006 (17)
O1	0.0068 (7)	0.0201 (9)	0.0194 (9)	-0.0031 (6)	-0.0024 (7)	0.0086 (8)
O2	0.0155 (9)	0.0211 (10)	0.0174 (9)	-0.0008 (7)	0.0091 (7)	0.0087 (8)
O3	0.0220 (9)	0.0078 (7)	0.0219 (9)	0.0063 (7)	0.0031 (8)	0.0032 (8)
O4	0.0240 (11)	0.0235 (11)	0.0135 (9)	-0.0030 (8)	0.0022 (8)	-0.0117 (8)

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

K1—O1 ⁱ	2.852 (2)	Fe1—O1 ^{xiv}	2.0195 (19)
K1—O1 ⁱⁱ	2.852 (2)	Fe1—K2 ^{xv}	3.6790 (12)
K1—O1 ⁱⁱⁱ	2.852 (2)	Fe1—K1 ^{xvi}	3.8453 (6)
K1—O2 ^{iv}	3.013 (2)	Fe1—K1 ^{xvii}	3.8453 (6)
K1—O2 ^v	3.013 (2)	Fe1—K1 ^{xviii}	3.8453 (6)
K1—O2 ^{vi}	3.013 (2)	Fe2—O3 ⁱ	1.9830 (19)
K1—O4 ^{iv}	3.117 (2)	Fe2—O3 ⁱⁱ	1.9830 (19)
K1—O4 ^v	3.117 (2)	Fe2—O3 ⁱⁱⁱ	1.9830 (19)
K1—O4 ^{vi}	3.117 (2)	Fe2—O4	2.010 (2)
K1—P1 ^{iv}	3.4419 (9)	Fe2—O4 ^{xiv}	2.010 (2)
K1—P1 ^v	3.4419 (9)	Fe2—O4 ^{xiii}	2.010 (2)
K1—P1 ^{vi}	3.4419 (9)	Fe2—K2 ^{xix}	3.7636 (4)
K2—O3 ^{iv}	2.868 (2)	Fe2—K2 ^{xx}	3.7636 (4)
K2—O3 ^{vi}	2.868 (2)	Fe2—K2 ^{xxi}	3.7636 (4)
K2—O3 ^v	2.868 (2)	P1—O4	1.521 (2)
K2—O2 ^{vii}	2.960 (2)	P1—O2	1.521 (2)
K2—O2 ^{viii}	2.960 (2)	P1—O3	1.525 (2)
K2—O2 ^{ix}	2.960 (2)	P1—O1	1.5306 (19)
K2—O4 ^{iv}	3.053 (3)	O1—K1 ^{xvi}	2.852 (2)
K2—O4 ^v	3.053 (3)	O2—Sn1 ^{xxii}	2.0032 (19)
K2—O4 ^{vi}	3.053 (3)	O2—Fe1 ^{xxii}	2.0032 (19)
K2—O4 ^{vii}	3.243 (2)	O2—K2 ^{xix}	2.960 (2)
K2—O4 ^{viii}	3.243 (2)	O2—K1 ^{xxi}	3.013 (2)
K2—O4 ^{ix}	3.243 (2)	O3—Sn2 ^{xvi}	1.9830 (19)
Fe1—O2 ^x	2.0032 (19)	O3—Fe2 ^{xvi}	1.9830 (19)
Fe1—O2 ^{xi}	2.0032 (19)	O3—K2 ^{xxi}	2.868 (2)
Fe1—O2 ^{xii}	2.0032 (19)	O4—K2 ^{xxi}	3.053 (3)
Fe1—O1 ^{xiii}	2.0195 (19)	O4—K1 ^{xxi}	3.117 (2)
Fe1—O1	2.0195 (19)	O4—K2 ^{xix}	3.243 (2)
O1 ⁱ —K1—O1 ⁱⁱ	92.31 (7)	O3 ^v —K2—O4 ^{viii}	86.13 (6)

O1 ⁱ —K1—O1 ⁱⁱⁱ	92.31 (7)	O2 ^{vii} —K2—O4 ^{viii}	88.43 (6)
O1 ⁱⁱ —K1—O1 ⁱⁱⁱ	92.31 (7)	O2 ^{viii} —K2—O4 ^{viii}	45.82 (5)
O1 ⁱ —K1—O2 ^{iv}	57.28 (5)	O2 ^{ix} —K2—O4 ^{viii}	100.56 (6)
O1 ⁱⁱ —K1—O2 ^{iv}	81.42 (6)	O4 ^{iv} —K2—O4 ^{viii}	137.22 (4)
O1 ⁱⁱⁱ —K1—O2 ^{iv}	148.35 (7)	O4 ^v —K2—O4 ^{viii}	54.34 (8)
O1 ⁱ —K1—O2 ^v	148.35 (7)	O4 ^{vi} —K2—O4 ^{viii}	104.276 (11)
O1 ⁱⁱ —K1—O2 ^v	57.28 (5)	O4 ^{vii} —K2—O4 ^{viii}	115.51 (3)
O1 ⁱⁱⁱ —K1—O2 ^v	81.42 (6)	O3 ^{iv} —K2—O4 ^{ix}	86.13 (6)
O2 ^{iv} —K1—O2 ^v	119.065 (15)	O3 ^{vi} —K2—O4 ^{ix}	156.82 (6)
O1 ⁱ —K1—O2 ^{vi}	81.42 (6)	O3 ^v —K2—O4 ^{ix}	56.20 (5)
O1 ⁱⁱ —K1—O2 ^{vi}	148.35 (7)	O2 ^{vii} —K2—O4 ^{ix}	100.56 (6)
O1 ⁱⁱⁱ —K1—O2 ^{vi}	57.28 (5)	O2 ^{viii} —K2—O4 ^{ix}	88.43 (6)
O2 ^{iv} —K1—O2 ^{vi}	119.065 (15)	O2 ^{ix} —K2—O4 ^{ix}	45.82 (5)
O2 ^v —K1—O2 ^{vi}	119.065 (15)	O4 ^{iv} —K2—O4 ^{ix}	54.34 (8)
O1 ⁱ —K1—O4 ^{iv}	103.75 (6)	O4 ^v —K2—O4 ^{ix}	104.276 (11)
O1 ⁱⁱ —K1—O4 ^{iv}	82.12 (6)	O4 ^{vi} —K2—O4 ^{ix}	137.22 (4)
O1 ⁱⁱⁱ —K1—O4 ^{iv}	163.15 (6)	O4 ^{vii} —K2—O4 ^{ix}	115.51 (3)
O2 ^{iv} —K1—O4 ^{iv}	46.64 (6)	O4 ^{viii} —K2—O4 ^{ix}	115.51 (3)
O2 ^v —K1—O4 ^{iv}	82.20 (6)	O2 ^x —Fe1—O2 ^{xi}	88.12 (9)
O2 ^{vi} —K1—O4 ^{iv}	129.53 (7)	O2 ^x —Fe1—O2 ^{xii}	88.12 (9)
O1 ⁱ —K1—O4 ^v	163.15 (6)	O2 ^{xi} —Fe1—O2 ^{xii}	88.12 (9)
O1 ⁱⁱ —K1—O4 ^v	103.75 (6)	O2 ^x —Fe1—O1 ^{xiii}	88.81 (8)
O1 ⁱⁱⁱ —K1—O4 ^v	82.12 (6)	O2 ^{xi} —Fe1—O1 ^{xiii}	176.13 (9)
O2 ^{iv} —K1—O4 ^v	129.53 (7)	O2 ^{xii} —Fe1—O1 ^{xiii}	89.43 (9)
O2 ^v —K1—O4 ^v	46.64 (6)	O2 ^x —Fe1—O1	89.43 (9)
O2 ^{vi} —K1—O4 ^v	82.20 (6)	O2 ^{xi} —Fe1—O1	88.81 (8)
O4 ^{iv} —K1—O4 ^v	83.78 (7)	O2 ^{xii} —Fe1—O1	176.13 (9)
O1 ⁱ —K1—O4 ^{vi}	82.12 (6)	O1 ^{xiii} —Fe1—O1	93.51 (9)
O1 ⁱⁱ —K1—O4 ^{vi}	163.15 (6)	O2 ^x —Fe1—O1 ^{xiv}	176.13 (9)
O1 ⁱⁱⁱ —K1—O4 ^{vi}	103.75 (6)	O2 ^{xi} —Fe1—O1 ^{xiv}	89.43 (9)
O2 ^{iv} —K1—O4 ^{vi}	82.20 (6)	O2 ^{xii} —Fe1—O1 ^{xiv}	88.81 (8)
O2 ^v —K1—O4 ^{vi}	129.53 (7)	O1 ^{xiii} —Fe1—O1 ^{xiv}	93.51 (9)
O2 ^{vi} —K1—O4 ^{vi}	46.64 (6)	O1—Fe1—O1 ^{xiv}	93.51 (9)
O4 ^{iv} —K1—O4 ^{vi}	83.78 (7)	O3 ⁱ —Fe2—O3 ⁱⁱ	93.35 (9)
O4 ^v —K1—O4 ^{vi}	83.78 (7)	O3 ⁱ —Fe2—O3 ⁱⁱⁱ	93.35 (9)
O3 ^{iv} —K2—O3 ^{vi}	100.64 (6)	O3 ⁱⁱ —Fe2—O3 ⁱⁱⁱ	93.35 (9)
O3 ^{iv} —K2—O3 ^v	100.64 (6)	O3 ⁱ —Fe2—O4	172.59 (9)
O3 ^{vi} —K2—O3 ^v	100.64 (6)	O3 ⁱⁱ —Fe2—O4	93.04 (9)
O3 ^{iv} —K2—O2 ^{vii}	95.91 (6)	O3 ⁱⁱⁱ —Fe2—O4	82.53 (9)
O3 ^{vi} —K2—O2 ^{vii}	100.74 (6)	O3 ⁱ —Fe2—O4 ^{xiv}	93.04 (9)
O3 ^v —K2—O2 ^{vii}	149.93 (6)	O3 ⁱⁱ —Fe2—O4 ^{xiv}	82.53 (9)

supplementary materials

O3 ^{iv} —K2—O2 ^{viii}	149.93 (6)	O3 ⁱⁱⁱ —Fe2—O4 ^{xiv}	172.59 (9)
O3 ^{vi} —K2—O2 ^{viii}	95.91 (6)	O4—Fe2—O4 ^{xiv}	91.52 (9)
O3 ^v —K2—O2 ^{viii}	100.74 (6)	O3 ⁱ —Fe2—O4 ^{xiii}	82.53 (9)
O2 ^{vii} —K2—O2 ^{viii}	56.14 (6)	O3 ⁱⁱ —Fe2—O4 ^{xiii}	172.59 (9)
O3 ^{iv} —K2—O2 ^{ix}	100.74 (6)	O3 ⁱⁱⁱ —Fe2—O4 ^{xiii}	93.04 (9)
O3 ^{vi} —K2—O2 ^{ix}	149.93 (6)	O4—Fe2—O4 ^{xiii}	91.52 (9)
O3 ^v —K2—O2 ^{ix}	95.91 (6)	O4 ^{xiv} —Fe2—O4 ^{xiii}	91.52 (9)
O2 ^{vii} —K2—O2 ^{ix}	56.14 (6)	O4—P1—O2	105.93 (13)
O2 ^{viii} —K2—O2 ^{ix}	56.14 (6)	O4—P1—O3	107.23 (13)
O3 ^{iv} —K2—O4 ^{iv}	48.81 (5)	O2—P1—O3	112.54 (12)
O3 ^{vi} —K2—O4 ^{iv}	114.59 (7)	O4—P1—O1	111.60 (13)
O3 ^v —K2—O4 ^{iv}	52.71 (6)	O2—P1—O1	108.63 (12)
O2 ^{vii} —K2—O4 ^{iv}	132.57 (6)	O3—P1—O1	110.83 (12)
O2 ^{viii} —K2—O4 ^{iv}	141.48 (6)	P1—O1—Fe1	130.29 (12)
O2 ^{ix} —K2—O4 ^{iv}	95.42 (6)	P1—O1—K1 ^{xvi}	112.12 (11)
O3 ^{iv} —K2—O4 ^v	114.59 (7)	Fe1—O1—K1 ^{xvi}	102.92 (7)
O3 ^{vi} —K2—O4 ^v	52.71 (6)	P1—O2—Sn1 ^{xxii}	165.01 (15)
O3 ^v —K2—O4 ^v	48.81 (5)	P1—O2—Fe1 ^{xxii}	165.01 (15)
O2 ^{vii} —K2—O4 ^v	141.48 (6)	P1—O2—K2 ^{xix}	93.70 (9)
O2 ^{viii} —K2—O4 ^v	95.42 (6)	Sn1 ^{xxii} —O2—K2 ^{xix}	93.67 (8)
O2 ^{ix} —K2—O4 ^v	132.57 (6)	Fe1 ^{xxii} —O2—K2 ^{xix}	93.67 (8)
O4 ^{iv} —K2—O4 ^v	85.96 (7)	P1—O2—K1 ^{xxi}	92.83 (10)
O3 ^{iv} —K2—O4 ^{vi}	52.71 (6)	Sn1 ^{xxii} —O2—K1 ^{xxi}	98.07 (8)
O3 ^{vi} —K2—O4 ^{vi}	48.81 (5)	Fe1 ^{xxii} —O2—K1 ^{xxi}	98.07 (8)
O3 ^v —K2—O4 ^{vi}	114.59 (7)	K2 ^{xix} —O2—K1 ^{xxi}	103.77 (7)
O2 ^{vii} —K2—O4 ^{vi}	95.42 (6)	P1—O3—Sn2 ^{xvi}	148.98 (14)
O2 ^{viii} —K2—O4 ^{vi}	132.57 (6)	P1—O3—Fe2 ^{xvi}	148.98 (14)
O2 ^{ix} —K2—O4 ^{vi}	141.48 (6)	P1—O3—K2 ^{xxi}	105.79 (11)
O4 ^{iv} —K2—O4 ^{vi}	85.96 (7)	Sn2 ^{xvi} —O3—K2 ^{xxi}	100.16 (8)
O4 ^v —K2—O4 ^{vi}	85.96 (7)	Fe2 ^{xvi} —O3—K2 ^{xxi}	100.16 (8)
O3 ^{iv} —K2—O4 ^{vii}	56.20 (5)	P1—O4—Fe2	151.99 (15)
O3 ^{vi} —K2—O4 ^{vii}	86.13 (6)	P1—O4—K2 ^{xxi}	98.04 (10)
O3 ^v —K2—O4 ^{vii}	156.82 (6)	Fe2—O4—K2 ^{xxi}	93.77 (8)
O2 ^{vii} —K2—O4 ^{vii}	45.82 (5)	P1—O4—K1 ^{xxi}	88.91 (9)
O2 ^{viii} —K2—O4 ^{vii}	100.56 (6)	Fe2—O4—K1 ^{xxi}	118.50 (9)
O2 ^{ix} —K2—O4 ^{vii}	88.43 (6)	K2 ^{xxi} —O4—K1 ^{xxi}	77.63 (6)
O4 ^{iv} —K2—O4 ^{vii}	104.276 (11)	P1—O4—K2 ^{xix}	83.19 (10)
O4 ^v —K2—O4 ^{vii}	137.22 (4)	Fe2—O4—K2 ^{xix}	88.28 (8)
O4 ^{vi} —K2—O4 ^{vii}	54.34 (8)	K2 ^{xxi} —O4—K2 ^{xix}	172.75 (8)
O3 ^{iv} —K2—O4 ^{viii}	156.82 (6)	K1 ^{xxi} —O4—K2 ^{xix}	95.28 (7)
O3 ^{vi} —K2—O4 ^{viii}	56.20 (5)		

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

Fig. 1

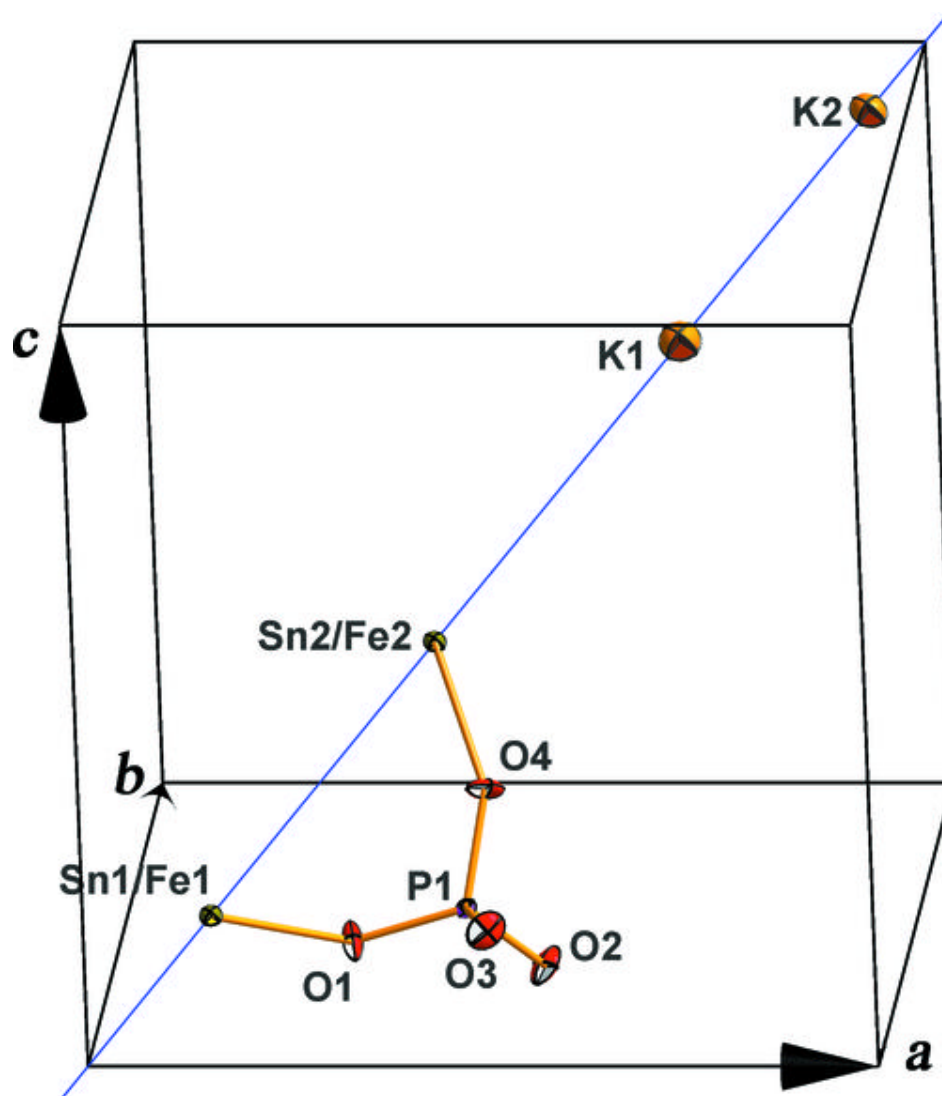


Fig. 2

