

## SUPPLEMENTAL INFORMATION

### **A new set of $\text{K}_3\text{Fe}_3(\text{PO}_4)_4 \cdot y\text{H}_2\text{O}$ ( $0 \leq y \leq 1$ ) layered phases obtained by topotactic reactions.**

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# S1

## Selected distances resulting from the Rietveld refinement of the XRD pattern of $K_3Fe_3(PO_4)_4 \cdot 0.75H_2O$ .

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Fe(1) – O(12) = 1.98(1) Å x 2	Fe(2) – O(11) = 2.21(1) Å
Fe(1) – O(14) = 2.22(1) Å x 2	Fe(2) – O(12) = 2.14(1) Å
Fe(1) – O(24) = 2.13(1) Å x 2	Fe(2) – O(13) = 1.93(1) Å
< Fe(1) – O > = 2.11 Å	Fe(2) – O(14) = 2.06(1) Å
$\Delta Fe(1) = 2.2 \cdot 10^{-3}$	Fe(2) – O(21) = 2.19(2) Å
	Fe(2) – O(22) = 1.73(1) Å
	< Fe(2) – O > = 2.04 Å
	$\Delta Fe(2) = 6.4 \cdot 10^{-3}$

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P(1) – O(11) = 1.36(1) Å	P(2) – O(21) = 1.42(1) Å
P(1) – O(12) = 1.65(2) Å	P(2) – O(22) = 1.70(1) Å
P(1) – O(13) = 1.55(1) Å	P(2) – O(23) = 1.36(1) Å
P(1) – O(14) = 1.39(1) Å	P(2) – O(24) = 1.38(1) Å
< P(1) – O > = 1.48 Å	< P(2) – O > = 1.46 Å
$\Delta P(1) = 6.4 \cdot 10^{-3}$	$\Delta P(2) = 8.8 \cdot 10^{-3}$

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K(1) – O(21) = 2.74(1) Å x 2	K(2) – O(11) = 2.77(1) Å
K(1) – O(22) = 2.78(1) Å x 2	K(2) – O(21) = 2.66(1) Å
K(1) – O(23) = 2.65(1) Å x 2	K(2) – O(22) = 2.69(1) Å
< K(1) – O > = 2.72 Å	K(2) – O(23) = 2.93(1) Å
	K(2) – O(24) = 2.63(1) Å
	K(2) – O(24) = 2.63(2) Å
	< K(2) – O > = 2.71 Å

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S2 : Refined structural parameters using the Rietveld method from the XRD pattern of the  $K_3Fe_3(PO_4)_4$  phase obtained at 600°C.

**$K_3Fe_3(PO_4)_4$**  Space group: C2/c

a = 20.9061(3) Å

b = 6.5346(4) Å

c = 10.7307(2) Å

$\beta$  = 90.938(1)°

Atom	Site	Wyckoff position			Occupancy
Fe(1)	4d	1/4	1/2	0.4882(3)	1
Fe(2)	8f	0.1624(6)	0.5076(4)	0.7548(2)	1
P(1)	8f	0.2055(1)	-0.0318(5)	0.2746(3)	1
O(11)	8f	0.1695(2)	0.792(5)	0.2922(3)	1
O(12)	8f	0.2523(2)	0.0534(4)	0.3607(3)	1
O(13)	8f	0.1594(2)	0.1781(5)	0.2318(3)	1
O(14)	8f	0.2588(2)	0.9768(4)	0.1407(3)	1
P(2)	8f	0.9045(1)	0.3312(4)	0.0047(3)	1
O(21)	8f	0.9021(2)	0.4983(5)	0.8716(3)	1
O(22)	8f	0.9004(2)	0.4945(5)	0.1141(3)	1
O(23)	8f	0.9626(8)	0.2262(3)	0.0042(5)	1
O(24)	8f	0.8445(10)	0.2171(3)	-0.0078(3)	1
K(1)	4e	0	0.3447	3/4	1
K(2)	8f	0.083(2)	0.1411(5)	0.0154(4)	1
H	8f	-0.006(2)	0.7805(8)	0.6911(3)	1
O	4e	0	0.7956(8)	3/4	

**Conditions of the run**

Temperature

Angular range

Step scan increment (2 $\theta$ )300 K

5° ≤ 2 $\theta$  ≤ 120°

0.016°

X-ray diffraction

**Profile parameters**

**Conventional Rietveld R-factors :**

R<sub>wp</sub> = 16.3%; R<sub>B</sub> = 12.8%