

RbZnFe(PO<sub>4</sub>)<sub>2</sub>: synthesis and crystal structure

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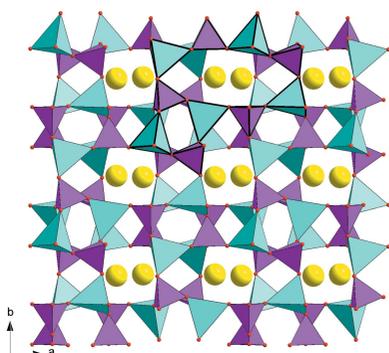
A new iron phosphate, rubidium zinc iron(III) phosphate, RbZnFe(PO<sub>4</sub>)<sub>2</sub>, has been synthesized as single crystals by the flux method. This compound is isostructural to the previously reported KCoAl(PO<sub>4</sub>)<sub>2</sub> [Chen *et al.* (1997). *Acta Cryst. C* **53**,1754–1756]. Its structure consists of a three-dimensional framework built up from corner-sharing PO<sub>4</sub> and (Zn,Fe)O<sub>4</sub> tetrahedra. This mode of linkage forms channels parallel to the [100], [010] and [001] directions in which the Rb<sup>+</sup> ions are located.

## 1. Chemical context

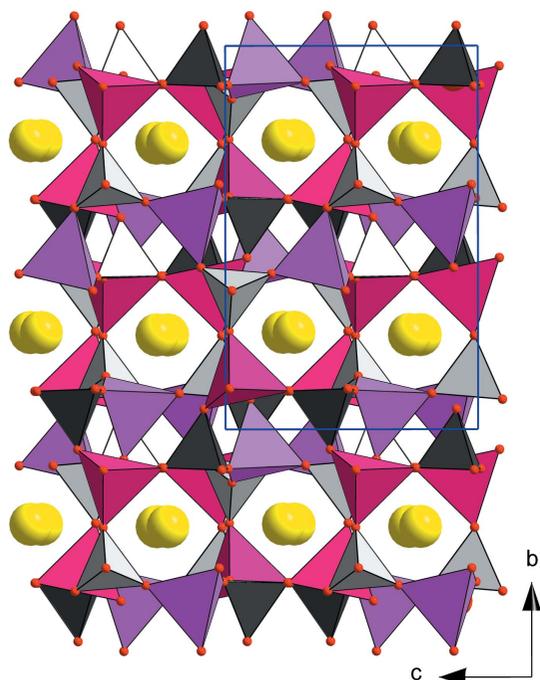
Phosphates with open-framework structures, similar to other porous materials such as zeolites, are interesting because of their wide industrial and environmental applications ranging from catalysis to ion-exchange and separation (Gier & Stucky, 1991; Maspoch *et al.*, 2007). Among them, iron phosphates (Redrup & Weller, 2009; Lajmi *et al.*, 2009) are particularly attractive because of their rich crystal chemistry (Moore, 1970; Gleitzer, 1991) and they present interesting and variable physical properties (Elbouaanani *et al.*, 2002; Riou-Cavellec *et al.*, 1999). Among the variety of iron orthophosphates synthesized and characterized over the past three decades, only two rubidium-containing compounds have been reported, namely Rb<sub>9</sub>Fe<sub>7</sub>(PO<sub>4</sub>)<sub>10</sub> (Hidouri *et al.*, 2010) and RbCuFe(PO<sub>4</sub>)<sub>2</sub> (Badri *et al.*, 2013). In this paper, we report the structure of a new rubidium iron orthophosphate, RbZnFe(PO<sub>4</sub>)<sub>2</sub>, synthesized during our investigation of the Rb<sub>3</sub>PO<sub>4</sub>–Zn<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>–FePO<sub>4</sub> quasi-system. This compound is isostructural to KCoAl(PO<sub>4</sub>)<sub>2</sub> (Chen *et al.*, 1997) and KZnFe(PO<sub>4</sub>)<sub>2</sub> (Badri *et al.*, 2014).

## 2. Structural commentary

The structure is made up of a three-dimensional assemblage of MO<sub>4</sub> ( $M = 0.5\text{Zn} + 0.5\text{Fe}$ ) and PO<sub>4</sub> tetrahedra through corner-sharing. This framework delimits crossing channels along the [100] and [001] directions, in which the Rb<sup>+</sup> ions are located (Figs. 1 and 2). A projection of the structure along [001] direction reveals that each MO<sub>4</sub> tetrahedron is linked to four PO<sub>4</sub> tetrahedra by sharing corners. In addition, it shows the presence of two kinds of rings through corner-sharing of MO<sub>4</sub> and PO<sub>4</sub> tetrahedra (Fig. 2). The first presents an elliptical form and comprises four MO<sub>4</sub> and four PO<sub>4</sub> tetrahedra, the second consists of two MO<sub>4</sub> and two PO<sub>4</sub> tetrahedra and has a quasi-rectangular form. From an examination of the interatomic distances (cation–oxygen), the M(1) and M(2) sites exhibit similar regular tetrahedral environments, as seen in the cation–oxygen distances which vary from 1.877 (5) to



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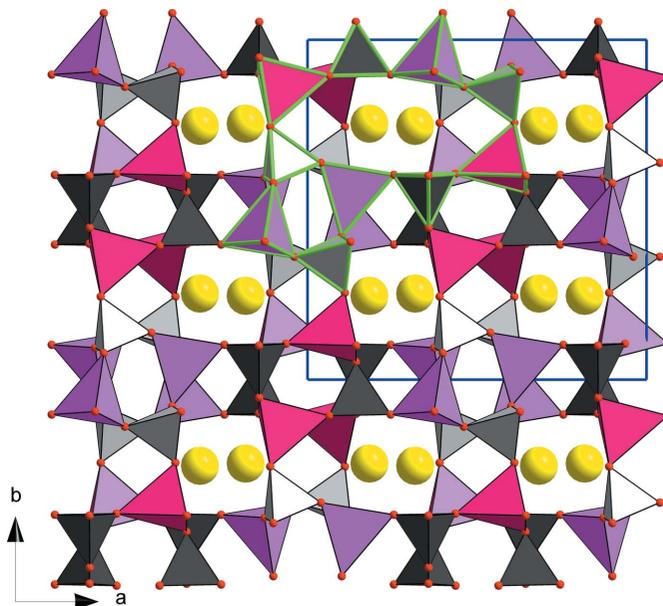
**Figure 1**  
A view of the crystal structure of  $\text{RbZnFe}(\text{PO}_4)_2$  along  $[100]$ . Colour key:  $M(1)\text{O}_4$  tetrahedra are purple,  $M(2)\text{O}_4$  tetrahedra are red,  $P(1)\text{O}_4$  tetrahedra are dark grey,  $P(2)\text{O}_4$  tetrahedra are light grey and  $\text{Rb}^+$  cations are yellow spheres.

1.900 (5) Å for  $M(1)$  and from 1.860 (6) to 1.919 (5) Å for  $M(2)$ . The average distances of 1.885 (2) and 1.888 (2) Å are between the values of 1.926 (2) Å observed for tetrahedrally coordinated  $\text{Zn}^{2+}$  ions in the zinc phosphate  $\text{RbZnPO}_4$  (Elammari & Elouadi, 1991) and 1.865 Å reported for the  $\text{Fe}^{3+}$  ions with the same coordination in the iron phosphate in  $\text{FePO}_4$  (Long *et al.*, 1983). The P–O distances within the  $\text{PO}_4$  tetrahedra are between 1.514 (5) and 1.535 (5) Å and with mean distances of 1.523 (9) Å for P(1) and 1.520 (3) Å for P(2), consistent with the value of 1.537 Å calculated by Baur (1974) for orthophosphate groups.

The  $\text{Rb}^+$  ions occupy a single site at the intersection of the crossing tunnels. Their environment was determined assuming all cation–oxygen distances to be shorter than the shortest distance between  $\text{Rb}^+$  and its nearest cation. This environment (Fig. 3) then consists of ten O atoms with  $\text{Rb}–\text{O}$  distances ranging from 2.925 (6) to 3.298 (7) Å.

### 3. Synthesis and crystallization

Single crystals of  $\text{RbZnFe}(\text{PO}_4)_2$  were grown in a flux of rubidium dimolybdate  $\text{Rb}_2\text{Mo}_2\text{O}_7$ , in an atomic ratio P:Mo = 4:1. Appropriate amounts of  $\text{Rb}_2\text{CO}_3$ ,  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ,  $(\text{NH}_4)_2\text{HPO}_4$  and  $\text{MoO}_3$  were used. All of the chemicals were analytically pure from commercial sources and used without further purification. The reagents were weighted in the atomic ratio P:Mo = 2:1 and dissolved in nitric acid and then dried for 24 h at 353 K. The dry residue was gradually heated to 873 K in a platinum crucible to remove the decomposition products. In a second step, the mixture was

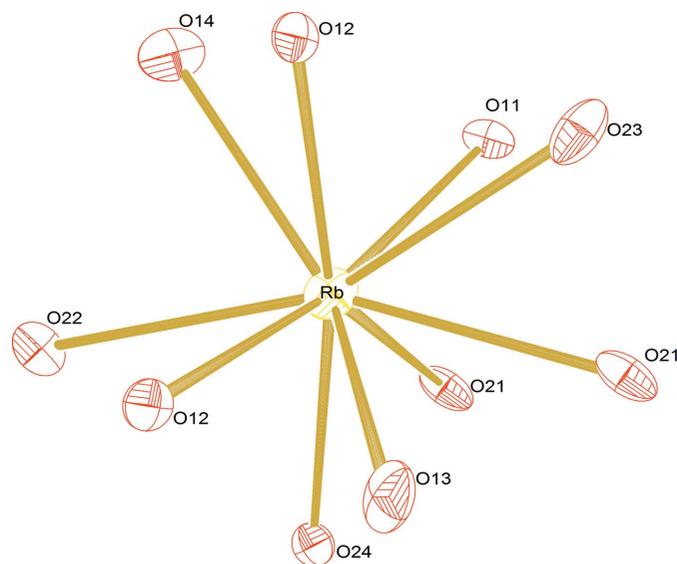


**Figure 2**  
A view of the crystal structure of  $\text{RbZnFe}(\text{PO}_4)_2$  along  $[001]$ , showing the elliptical and quasi-rectangular forms of corner-sharing  $\text{MO}_4$  and  $\text{PO}_4$  tetrahedra (edge with green colour). The colour key is as in Fig. 1.

ground, melted for 1 h at 1173 K and subsequently cooled at a rate of  $10 \text{ K h}^{-1}$  to 773 K, after which the furnace was turned off. The crystals obtained by washing the final product with warm water in order to dissolve the flux are essentially comprised of beige hexagonally shaped crystals of  $\text{RbZnFe}(\text{PO}_4)_2$ .

### 4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The application of direct methods



**Figure 3**  
The environment of the  $\text{Rb}$  cations, showing displacement ellipsoids drawn at the 50% probability level. **Authors: Define symmetry operators (in the Figure) and codes (in the caption)**

**Table 1**  
Experimental details.

Crystal data	
Chemical formula	RbZnFe(PO <sub>4</sub> ) <sub>2</sub>
<i>M<sub>r</sub></i>	396.63
Crystal system, space group	Monoclinic, <i>C2/c</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	13.601 (4), 13.304 (5), 8.978 (9)
$\beta$ (°)	100.76 (5)
<i>V</i> (Å <sup>3</sup> )	1596.0 (18)
<i>Z</i>	8
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	11.29
Crystal size (mm)	0.43 × 0.25 × 0.18
Data collection	
Diffractometer	Enraf–Nonius TurboCAD-4
Absorption correction	Part of the refinement model ( $\Delta F$ ) (Walker & Stuart 1983)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.054, 0.070
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	1409, 1409, 1227
<i>R<sub>int</sub></i>	0.089
( $\sin \theta/\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.594
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.036, 0.110, 1.05
No. of reflections	1409
No. of parameters	118
	$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 31.2735P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.85, -0.76

Computer programs: *CAD-4 EXPRESS* (Enraf–Nonius, 1994), *XCAD4* (Harms & Wocadlo, 1995), *SIR92* (Altomare *et al.*, 1993), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 1999) and *WinGX* (Farrugia, 2012).

revealed the Rb atoms and located two sites, labelled *M*(1) and *M*(2), statistically occupied by the Fe<sup>3+</sup> and Zn<sup>2+</sup> ions. This distribution was supported by the *M*(1)–O and *M*(2)–O distances which are between the classical pure Zn–O and Fe–O values. Succeeding difference Fourier syntheses led to the positions of all the remaining atoms.

Despite several synthesis attempts, all the obtained crystals of RbZnFe(PO<sub>4</sub>)<sub>2</sub> were of poor quality, resulting in the large

discrepancies found in a number of reflections; hence in this study the refinement was performed using a filter of the reflections by  $[\sin(\theta)/\lambda]$ . The four reflections ( $\bar{6}85$ ,  $\bar{9}34$ ,  $\bar{8}85$  and  $\bar{3}75$ ) were omitted as the difference between the observed and calculated structure factors was larger than 10 $\sigma$ .

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## supporting information

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## Computing details

Cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

## Rubidium zinc Iron(III) phosphate

## Crystal data

RbZnFe(PO<sub>4</sub>)<sub>2</sub>

$M_r = 396.63$

Monoclinic, *C2/c*

$a = 13.601$  (4) Å

$b = 13.304$  (5) Å

$c = 8.978$  (9) Å

$\beta = 100.76$  (5)°

$V = 1596.0$  (18) Å<sup>3</sup>

$Z = 8$

$F(000) = 1496$

$D_x = 3.301$  Mg m<sup>-3</sup>

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

Cell parameters from 25 reflections

$\theta = 8.1$ – $11.1$ °

$\mu = 11.29$  mm<sup>-1</sup>

$T = 293$  K

Prism, brown

$0.43 \times 0.25 \times 0.18$  mm

## Data collection

Enraf–Nonius TurboCAD-4  
diffractometer

Radiation source: fine-focus sealed tube  
non-profiled  $\omega/2\tau$  scans

Absorption correction: part of the refinement  
model ( $\Delta F$ )  
(Walker & Stuart 1983)

$T_{\min} = 0.054$ ,  $T_{\max} = 0.070$

1409 measured reflections

1409 independent reflections

1227 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.089$

$\theta_{\max} = 25.0$ °,  $\theta_{\min} = 2.2$ °

$h = -16$ → $15$

$k = 0$ → $15$

$l = 0$ → $10$

2 standard reflections every 120 min

intensity decay: 1%

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.110$

$S = 1.05$

1409 reflections

118 parameters

0 restraints

$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 31.2735P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.85$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.76$  e Å<sup>-3</sup>

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Rb	0.18260 (6)	0.24668 (6)	0.22827 (9)	0.0316 (3)	
Zn1	0.87122 (6)	0.55912 (6)	0.11383 (9)	0.0169 (3)	0.5
Fe1	0.87122 (6)	0.55912 (6)	0.11383 (9)	0.0169 (3)	0.5
Zn2	0.92406 (6)	0.12098 (6)	-0.05652 (9)	0.0166 (3)	0.5
Fe2	0.92406 (6)	0.12098 (6)	-0.05652 (9)	0.0166 (3)	0.5
P1	0.14761 (12)	0.06205 (13)	-0.08572 (19)	0.0166 (4)	
O11	0.1420 (4)	-0.0526 (4)	-0.0852 (6)	0.0295 (12)	
O12	0.2450 (3)	0.1026 (4)	0.0096 (6)	0.0243 (11)	
O13	0.3570 (5)	0.3996 (5)	0.2456 (6)	0.0397 (15)	
O14	0.0638 (4)	0.1055 (5)	-0.0151 (7)	0.0385 (14)	
P2	0.92645 (12)	0.36174 (12)	-0.03358 (18)	0.0144 (4)	
O21	0.8903 (5)	0.2550 (4)	-0.0146 (7)	0.0323 (13)	
O22	0.0389 (4)	0.3613 (4)	-0.0253 (6)	0.0269 (11)	
O23	0.3731 (4)	0.0942 (5)	0.3168 (6)	0.0367 (14)	
O24	0.8990 (4)	0.4217 (4)	0.0972 (6)	0.0252 (11)	

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Rb	0.0383 (5)	0.0321 (5)	0.0259 (4)	0.0006 (3)	0.0097 (3)	-0.0044 (3)
Zn1	0.0197 (4)	0.0151 (5)	0.0149 (4)	0.0019 (3)	0.0006 (3)	-0.0023 (3)
Fe1	0.0197 (4)	0.0151 (5)	0.0149 (4)	0.0019 (3)	0.0006 (3)	-0.0023 (3)
Zn2	0.0194 (5)	0.0149 (5)	0.0149 (4)	-0.0013 (3)	0.0021 (3)	-0.0026 (3)
Fe2	0.0194 (5)	0.0149 (5)	0.0149 (4)	-0.0013 (3)	0.0021 (3)	-0.0026 (3)
P1	0.0191 (8)	0.0138 (8)	0.0156 (8)	-0.0026 (7)	-0.0004 (6)	0.0035 (6)
O11	0.043 (3)	0.014 (3)	0.033 (3)	-0.003 (2)	0.013 (2)	0.003 (2)
O12	0.019 (2)	0.023 (3)	0.028 (3)	-0.0014 (19)	-0.003 (2)	-0.003 (2)
O13	0.059 (4)	0.043 (3)	0.015 (3)	-0.011 (3)	0.002 (3)	0.011 (2)
O14	0.021 (3)	0.042 (3)	0.052 (4)	0.000 (2)	0.007 (3)	-0.017 (3)
P2	0.0211 (9)	0.0095 (8)	0.0127 (8)	-0.0020 (6)	0.0031 (6)	0.0001 (6)
O21	0.053 (4)	0.016 (3)	0.034 (3)	-0.010 (2)	0.024 (3)	-0.010 (2)
O22	0.023 (3)	0.023 (3)	0.037 (3)	0.000 (2)	0.010 (2)	0.006 (2)
O23	0.037 (3)	0.056 (4)	0.016 (3)	-0.006 (3)	0.004 (2)	-0.011 (2)
O24	0.040 (3)	0.014 (2)	0.023 (3)	0.005 (2)	0.009 (2)	-0.0051 (19)

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

Rb—O21 <sup>i</sup>	2.925 (6)	Zn1—O22 <sup>vi</sup>	1.900 (5)
Rb—O12	2.979 (5)	Zn2—O13 <sup>vii</sup>	1.860 (6)

Rb—O14	3.098 (6)	Zn2—O14 <sup>viii</sup>	1.878 (5)
Rb—O13	3.107 (6)	Zn2—O21	1.897 (5)
Rb—O22	3.109 (5)	Zn2—O11 <sup>ix</sup>	1.919 (5)
Rb—O24 <sup>i</sup>	3.123 (5)	P1—O13 <sup>iii</sup>	1.514 (5)
Rb—O11 <sup>ii</sup>	3.181 (5)	P1—O14	1.519 (6)
Rb—O12 <sup>iii</sup>	3.215 (6)	P1—O11	1.527 (5)
Rb—O23	3.269 (6)	P1—O12	1.535 (5)
Rb—O21 <sup>iv</sup>	3.298 (7)	P2—O22 <sup>viii</sup>	1.517 (5)
Zn1—O23 <sup>v</sup>	1.877 (5)	P2—O23 <sup>vii</sup>	1.520 (5)
Zn1—O24	1.879 (5)	P2—O24	1.523 (5)
Zn1—O12 <sup>v</sup>	1.886 (5)	P2—O21	1.522 (5)
O21 <sup>i</sup> —Rb—O12	142.06 (14)	O12 <sup>iii</sup> —Rb—O23	102.79 (14)
O21 <sup>i</sup> —Rb—O14	115.16 (17)	O21 <sup>i</sup> —Rb—O21 <sup>iv</sup>	76.81 (19)
O12—Rb—O14	47.17 (13)	O12—Rb—O21 <sup>iv</sup>	98.31 (14)
O21 <sup>i</sup> —Rb—O13	108.19 (16)	O14—Rb—O21 <sup>iv</sup>	139.07 (14)
O12—Rb—O13	98.37 (15)	O13—Rb—O21 <sup>iv</sup>	54.77 (14)
O14—Rb—O13	136.49 (16)	O22—Rb—O21 <sup>iv</sup>	148.78 (13)
O21 <sup>i</sup> —Rb—O22	110.80 (16)	O24 <sup>i</sup> —Rb—O21 <sup>iv</sup>	89.55 (14)
O12—Rb—O22	92.90 (15)	O11 <sup>ii</sup> —Rb—O21 <sup>iv</sup>	80.58 (14)
O14—Rb—O22	66.86 (16)	O12 <sup>iii</sup> —Rb—O21 <sup>iv</sup>	98.09 (13)
O13—Rb—O22	94.89 (14)	O23—Rb—O21 <sup>iv</sup>	44.69 (13)
O21 <sup>i</sup> —Rb—O24 <sup>i</sup>	47.19 (13)	O23 <sup>v</sup> —Zn1—O24	110.7 (3)
O12—Rb—O24 <sup>i</sup>	169.12 (13)	O23 <sup>v</sup> —Zn1—O12 <sup>v</sup>	104.6 (2)
O14—Rb—O24 <sup>i</sup>	128.20 (14)	O24—Zn1—O12 <sup>v</sup>	115.9 (2)
O13—Rb—O24 <sup>i</sup>	79.99 (16)	O23 <sup>v</sup> —Zn1—O22 <sup>vi</sup>	112.0 (3)
O22—Rb—O24 <sup>i</sup>	76.59 (15)	O24—Zn1—O22 <sup>vi</sup>	110.8 (2)
O21 <sup>i</sup> —Rb—O11 <sup>ii</sup>	56.47 (13)	O12 <sup>v</sup> —Zn1—O22 <sup>vi</sup>	102.6 (2)
O12—Rb—O11 <sup>ii</sup>	85.60 (14)	O13 <sup>vii</sup> —Zn2—O14 <sup>viii</sup>	118.1 (3)
O14—Rb—O11 <sup>ii</sup>	76.11 (17)	O13 <sup>vii</sup> —Zn2—O21	103.5 (3)
O13—Rb—O11 <sup>ii</sup>	135.33 (15)	O14 <sup>viii</sup> —Zn2—O21	109.7 (3)
O22—Rb—O11 <sup>ii</sup>	129.51 (14)	O13 <sup>vii</sup> —Zn2—O11 <sup>ix</sup>	110.9 (3)
O24 <sup>i</sup> —Rb—O11 <sup>ii</sup>	103.19 (13)	O14 <sup>viii</sup> —Zn2—O11 <sup>ix</sup>	113.5 (3)
O21 <sup>i</sup> —Rb—O12 <sup>iii</sup>	139.13 (14)	O21—Zn2—O11 <sup>ix</sup>	98.8 (2)
O12—Rb—O12 <sup>iii</sup>	78.67 (16)	O13 <sup>iii</sup> —P1—O14	111.5 (4)
O14—Rb—O12 <sup>iii</sup>	95.38 (16)	O13 <sup>iii</sup> —P1—O11	110.3 (3)
O13—Rb—O12 <sup>iii</sup>	45.51 (13)	O14—P1—O11	109.7 (3)
O22—Rb—O12 <sup>iii</sup>	55.68 (13)	O13 <sup>iii</sup> —P1—O12	106.8 (3)
O24 <sup>i</sup> —Rb—O12 <sup>iii</sup>	92.85 (14)	O14—P1—O12	105.7 (3)
O11 <sup>ii</sup> —Rb—O12 <sup>iii</sup>	163.87 (13)	O11—P1—O12	112.8 (3)
O21 <sup>i</sup> —Rb—O23	101.14 (15)	O22 <sup>viii</sup> —P2—O23 <sup>vii</sup>	110.8 (3)
O12—Rb—O23	56.67 (14)	O22 <sup>viii</sup> —P2—O24	110.8 (3)
O14—Rb—O23	94.63 (15)	O23 <sup>vii</sup> —P2—O24	109.5 (3)
O13—Rb—O23	80.27 (17)	O22 <sup>viii</sup> —P2—O21	109.5 (3)
O22—Rb—O23	147.51 (14)	O23 <sup>vii</sup> —P2—O21	110.3 (3)

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O24 <sup>i</sup> —Rb—O23	132.85 (14)	O24—P2—O21	105.7 (3)
O11 <sup>ii</sup> —Rb—O23	64.93 (15)		

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