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# Dipotassium dialuminium cyclooctaphosphate

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Key indicators: single-crystal X-ray study; T = 296 K; mean  $\sigma$ (P–O) = 0.002 Å; R factor = 0.042; wR factor = 0.124; data-to-parameter ratio = 32.7.

Single crystals of the title compound, K<sub>2</sub>Al<sub>2</sub>P<sub>8</sub>O<sub>24</sub>, were obtained by solid-state reaction. The monoclinic structure is isotypic with that of the Ga<sup>III</sup> analogue and is built of eightmembered phosphate ring anions  $P_8O_{24}^{8-}$  (2/m symmetry) isolated from each other and further linked by isolated AlO<sub>6</sub> octahedra ( $\overline{1}$  symmetry) by sharing corners. Each AlO<sub>6</sub> octahedron is linked to four  $P_8O_{24}^{8-}$  rings in such a way that two rings are linked through bidentate diphosphate groups attached in the cis positions on two opposite parallel edges of the octahedron. The two other rings are linked via cornersharing to the two remaining corners in the *trans* positions of the AlO<sub>6</sub> octahedron. Each  $P_8O_{24}^{8-}$  ring anion is linked to eight AlO<sub>6</sub> octahedra. More accurately, each ring anion is linked to four AlO<sub>6</sub> octahedra through bidentate diphosphate groups attached in the cis positions to the AlO<sub>6</sub> octahedron and to the four remaining octahedra by sharing corners. This threedimensional linkage delimits channels running parallel to [001] in which the ten-coordinated  $K^+$  cations (2 symmetry) are distributed over two columns. These columns alternate with empty octagonally-shaped channels expanding through the  $P_8O_{24}^{8-}$  ring anions.

#### **Related literature**

The synthesis and an approximate unit cell with a slightly smaller  $\beta$  angle were reported for the title compound more than a quarter of a century ago (Grunze *et al.*, 1983). The crystal structures of isotypic compounds determined from single-crystal data have been reported for K<sub>2</sub>Ga<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Palkina *et al.*, 1979) and K<sub>2</sub>Mn<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Murashova & Chudinova, 1999). The isostructural potassium-containing cyclo-octaphosphates K<sub>2</sub>V<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Lavrov *et al.*, 1981), K<sub>2</sub>Fe<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Grunze *et al.*, 1983) and K<sub>2</sub>Cr<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Grunze & Chudinova, 1988) were reported without detailed structure analyses. For a review of the crystal chemistry of cyclooctaphosphates, see:

V = 1011.4 (2) Å<sup>3</sup>

Mo  $K\alpha$  radiation

 $0.30 \times 0.10 \times 0.08 \text{ mm}$ 

9643 measured reflections

2844 independent reflections

2252 reflections with  $I > 2\sigma(I)$ 

 $\mu = 1.31 \text{ mm}^{-1}$ 

T = 296 K

 $R_{\rm int}=0.047$ 

87 parameters

 $\Delta \rho_{\rm max} = 1.40$  e Å<sup>-3</sup>

 $\Delta \rho_{\rm min} = -1.04 \text{ e } \text{\AA}^{-3}$ 

Z = 2

Durif (1995, 2005). For potential applications of aluminophosphates, see: Cheetham *et al.* (1999); Hartmann & Kevan (1999). For background to distortion indices, see: Momma & Izumi (2008).

#### **Experimental**

Crystal data  $K_2Al_2P_8O_{24}$   $M_r = 763.92$ Monoclinic, C2/m a = 16.598 (2) Å b = 12.2150 (17) Å c = 5.0705 (7) Å  $\beta = 100.315$  (4)°

#### Data collection

Bruker APEXII CCD diffractometer Absorption correction: multi-scan (SADABS; Bruker, 2008) T<sub>min</sub> = 0.562, T<sub>max</sub> = 0.748

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$  $wR(F^2) = 0.124$ S = 1.072844 reflections

## Table 1

Selected bond lengths (Å).

| P1-O1 <sup>i</sup> | 1.4861 (12) | P2-O5 | 1.4622 (14) |
|--------------------|-------------|-------|-------------|
| P1-O3              | 1.4956 (12) | P2-O2 | 1.4983 (12) |
| P1-O4              | 1.5701 (13) | P2-O6 | 1.5957 (10) |
| P1-O7              | 1.5794 (8)  | P2-O4 | 1.6070 (13) |

Symmetry code: (i)  $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$ .

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999), CaRine (Boudias & Monceau, 1998) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2355).

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# supporting information

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### S1. Comment

The title compound, K<sub>2</sub>Al<sub>2</sub>P<sub>8</sub>O<sub>24</sub>, belongs to porous crystalline open-framework materials that gain growing interest for their potential applications as molecular sieves or catalysts (Cheetham *et al.* 1999; Hartmann & Kevan, 1999). The title cyclooctaphosphate is isotypic with K<sub>2</sub>Ga<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Palkina *et al.*, 1979) and K<sub>2</sub>Mn<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Murashova & Chudinova, 1999). This structural family also includes K<sub>2</sub>V<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Lavrov *et al.*, 1981), K<sub>2</sub>Fe<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Grunze *et al.*, 1983) and K<sub>2</sub>Cr<sub>2</sub>P<sub>8</sub>O<sub>24</sub> (Grunze & Chudinova, 1988), the structures of which have not yet been refined from X-ray diffraction data.

The crystal structure of  $K_2M_2P_8O_{24}$  is built up of 8-membered phosphate ring anions  $P_8O_{24}^{8-}$  (symmetry 2/m) (Fig. 1), further linked by isolated  $M^{IIO_6}$  octahedra to form the three-dimensional skeleton. Each  $MO_6$  octahedron is linked to four ring anions  $P_8O_{24}^{8}$ . Two ring anions are linked by corner-sharing in *trans* positions whereas the two others are connected in a bidentate fashion in cis positions on two opposite edges of the equatorial plane of the octahedron. This threedimensional framework of  $K_2Al_2P_8O_{24}$  delimits two kinds of channels expanding along the [001] direction (Fig. 2). The first channel is octagonally shaped since passing through the ring anions and is empty despite a size of  $\approx 5.2$  Å in diameter. The second channel, cross shaped, accommodates the  $K^+$  ions in a [6 + 4] coordination. The  $K^+$  ions are located over two columns shifted of about c/2 with respect to each other along the c axis. Thus they form two distinct K—K pairs with common square faces involving only O(5) oxygen atoms. In the first pair, corresponding to the shortest K—K distance (3.634 (9) Å) (Fig. 3), the four O(5) atoms delimit a pseudo-square face with O—O separations of 2.995 (2) and 3.064 (6) Å and O(5)—O(5)—O(5) angles of 90°. This pair corresponds to the two shortest K—O distances, viz. 2.7559 (15) and 2.8612 (15) Å. In the second pair with a K-K distance of 4.365 (7) Å, the four O(5) atoms at the vertices of the pseudo-square face (O—O separation 2.995 (2) and 3.139 (7) Å with O(5)—O(5)—O(5) angles of 90°), the respective K—O bonds lengths are 2.8612 (15) and 3.2790 (17) Å. The polyhedra surrounding three potassium cations engaged in two successive pairs (one long and one short) form a cluster within they share a common O(5)—O(5)edge with a contact distance of 2.995 (2) Å. This potassium-oxygen polyhedra packing also prevails in the Ga and Mn cyclooctaphosphate analogues but the K—O distances spread over larger ranges, viz. from 2.754 (28) Å to 3.359 (28) Å and from 2.738 (2) to 3.506 (2) Å, respectively. Thus the respective coordinations of the potassium cations can be regarded as being [6+4] and [8+2] for Ga and Mn cyclooctaphosphate.

A careful examination of the geometry of the  $M^{III}O_6$  octahedra in this structural type shows that the distortion index (bond length) (Momma & Izumi, 2008) increases from Al to Mn (0.0117 for Al, 0.0202 for Ga and 0.0574 for Mn). The AlO<sub>6</sub> octahedron is only very slightly distorted with two shorter Al—O distances of 1.8523 (12) Å and four others very close to 1.90 Å (1.9013 (12) (× 2) and 1.9021 (11) (× 2) Å). The significant larger distortion of the MnO<sub>6</sub> octahedron is probably due to the Jahn-Teller effect associated with the  $d^4$  electronic configuration of Mn<sup>III</sup> (Murashova & Chudinova, 1999). Besides the structural family to which the title compound belongs, only another sodium and silver- containing cyclooctaphosphate,  $Ag_9NaP_8O_{24}(NO_3)_2.4H_2O$ , exhibits a ring anion with internal 2/m symmetry among the presently known cyclooctaphosphates (Durif, 1995, 2005). However, despite the common internal symmetry, the shape of the 8-membered ring anion present in this structure is very different from that of the title compound as shown in Fig. 4.

## **S2. Experimental**

Single-crystals of the title compound were obtained by solid state reaction, from the reagents  $K_2CO_3$ ,  $Al_2O_3$  and  $(NH_4)H_2PO_4$  in the molar ratio K / P / Al = 57 / 34 / 9. The mixture has progressively been heated up to 873 K over a period of 12 h. Then the temperature was slowly decreased down to 723 K at the rate of 5 K h<sup>-1</sup> and maintained at this value for 12 h. Then a new cooling step down to 573 K at the rate of 5 K h<sup>-1</sup> was carried out before the furnace was switched off. Single-crystals of  $K_2Al_2P_8O_{24}$  were extracted from the batch by washing with hot water in order to remove the excess of  $P_2O_5$ . A translucent colorless needle of the title compound was used for the structure refinement.

### **S3. Refinement**

The highest residual peak in the final difference Fourier map was located 0.74 Å from atom P2 and the deepest hole was located 0.69 Å from atom P1.



## Figure 1

*ORTEP*-3 view (Farrugia, 1997) of the centrosymmetric  $(P_8O_{24})^{8-}$  ring anion. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) -*x*, -*y* + 1, -*z* + 1; (iv) -*x*, *y*, -*z*; (v) -*x*, -*y* + 1, -*z*; (vi) *x* - 1/2, -*y* + 1/2, *z*; (ix) -*x* + 1/2, -*y* + 1/2, -*z* + 1; (xi) *x*, -*y*, *z*.





Projections of the 8-membered ring anions  $P_8O_{24}^{8-}$  along [010] (left) and [001] (right).



# Figure 3

Partial view showing the packing of the K—O polyhedra along the *c* axis in the ball and stick (left) and polyhedral (right) representation.



# Figure 4

Comparison of the 8-membered ring anions  $P_8O_{24}^{8-}$  (both with internal 2/m symmetry) in  $K_2Al_2P_8O_{24}$  (top) and Ag<sub>9</sub>NaP<sub>8</sub>O<sub>24</sub>(NO<sub>3</sub>)<sub>2</sub>.4H<sub>2</sub>O (bottom).

# Dipotassium dialuminium cyclooctaphosphate

| Crystal data   |   |
|--|---|
| $K_{2}Al_{2}P_{8}O_{24}$ $M_{r} = 763.92$ Monoclinic, C2/m<br>Hall symbol: -C 2y<br>a = 16.598 (2) Å<br>b = 12.2150 (17) Å<br>c = 5.0705 (7) Å<br>$\beta = 100.315$ (4)°<br>V = 1011.4 (2) Å <sup>3</sup><br>Z = 2   | F(000) = 752<br>$D_x = 2.508 \text{ Mg m}^{-3}$<br>Mo K\alpha radiation, \lambda = 0.71073 \mathcal{A}<br>Cell parameters from 3017 reflections<br>$\theta = 3.3-37.7^{\circ}$<br>$\mu = 1.31 \text{ mm}^{-1}$<br>T = 296  K<br>Needle, colourless<br>$0.30 \times 0.10 \times 0.08 \text{ mm}$ |
| Data collection  |   |
| Bruker APEXII CCD<br>diffractometer<br>Radiation source: fine-focus sealed tube<br>Graphite monochromator<br>Detector resolution: 8.3333 pixels mm <sup>-1</sup><br>$\omega$ and $\varphi$ scans<br>Absorption correction: multi-scan<br>( <i>SADABS</i> ; Bruker, 2008)<br>$T_{min} = 0.562, T_{max} = 0.748$ | 9643 measured reflections<br>2844 independent reflections<br>2252 reflections with $I > 2\sigma(I)$<br>$R_{int} = 0.047$<br>$\theta_{max} = 38.7^{\circ}, \theta_{min} = 4.1^{\circ}$<br>$h = -29 \rightarrow 28$<br>$k = -18 \rightarrow 20$<br>$l = -8 \rightarrow 8$                         |

Refinement

| Refinement on $F^2$             | Primary atom site location: structure-invariant  |
|---------------------------------|--|
| Least-squares matrix: full      | direct methods   |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | Secondary atom site location: difference Fourier   |
| $wR(F^2) = 0.124$               | map  |
| S = 1.07                        | $w = 1/[\sigma^2(F_o^2) + (0.0738P)^2 + 0.0398P]$  |
| 2844 reflections                | where $P = (F_o^2 + 2F_c^2)/3$   |
| 87 parameters                   | $(\Delta/\sigma)_{max} < 0.001$  |
| 0 restraints                    | $\Delta\rho_{max} = 1.40$ e Å <sup>-3</sup>  |
| 0 restraints                    | $\Delta  ho_{ m max} = 1.40 \ { m e} \ { m \AA}^{-3} \ \Delta  ho_{ m min} = -1.04 \ { m e} \ { m \AA}^{-3}$ |

### 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.

**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  $F^2 > \sigma(F^2)$  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  $(\hat{A}^2)$ 

|    | x            | У            | Ζ            | $U_{ m iso}$ */ $U_{ m eq}$ |  |
|----|--------------|--------------|--------------|-----------------------------|--|
| K  | -0.09371 (4) | 0.5000       | 0.25194 (13) | 0.02290 (13)                |  |
| Al | 0.2500       | 0.2500       | 0.0000       | 0.00771 (13)                |  |
| P1 | 0.19023 (2)  | 0.12098 (3)  | 0.46962 (8)  | 0.00720 (9)                 |  |
| P2 | 0.07782 (2)  | 0.27631 (3)  | 0.16477 (8)  | 0.00835 (9)                 |  |
| 01 | 0.28060 (8)  | 0.36965 (10) | 0.2366 (2)   | 0.0118 (2)                  |  |
| O2 | 0.14059 (7)  | 0.28394 (11) | -0.0132 (2)  | 0.0132 (2)                  |  |
| O3 | 0.24968 (7)  | 0.15349 (10) | 0.2942 (2)   | 0.0105 (2)                  |  |
| O4 | 0.10711 (7)  | 0.18408 (11) | 0.3873 (2)   | 0.0122 (2)                  |  |
| O5 | 0.05469 (9)  | 0.37739 (12) | 0.2866 (3)   | 0.0184 (3)                  |  |
| 06 | 0.0000       | 0.21471 (15) | 0.0000       | 0.0140 (3)                  |  |
| 07 | 0.16028 (11) | 0.0000       | 0.3993 (3)   | 0.0125 (3)                  |  |

Atomic displacement parameters  $(Å^2)$ 

|            | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|------------|--------------|--------------|--------------|--------------|--------------|--------------|
| Κ          | 0.0299 (3)   | 0.0147 (3)   | 0.0240 (3)   | 0.000        | 0.0046 (2)   | 0.000        |
| Al         | 0.0085 (3)   | 0.0083 (3)   | 0.0064 (3)   | 0.0001 (2)   | 0.0016 (2)   | 0.0006 (2)   |
| P1         | 0.00956 (16) | 0.00518 (17) | 0.00672 (15) | 0.00032 (12) | 0.00110 (11) | 0.00033 (11) |
| P2         | 0.00850 (17) | 0.00632 (18) | 0.01023 (16) | 0.00094 (12) | 0.00164 (12) | 0.00031 (12) |
| 01         | 0.0186 (5)   | 0.0084 (5)   | 0.0078 (4)   | -0.0009 (4)  | 0.0006 (4)   | -0.0006 (3)  |
| 02         | 0.0095 (5)   | 0.0177 (6)   | 0.0130 (5)   | 0.0020 (4)   | 0.0033 (4)   | 0.0049 (4)   |
| 03         | 0.0104 (5)   | 0.0111 (5)   | 0.0105 (5)   | 0.0011 (4)   | 0.0037 (3)   | 0.0033 (4)   |
| 04         | 0.0108 (5)   | 0.0125 (6)   | 0.0138 (5)   | 0.0033 (4)   | 0.0034 (4)   | 0.0051 (4)   |
| 05         | 0.0247 (6)   | 0.0105 (6)   | 0.0198 (6)   | 0.0053 (5)   | 0.0030 (5)   | -0.0045 (5)  |
| 06         | 0.0096 (7)   | 0.0092 (8)   | 0.0217 (8)   | 0.000        | -0.0011 (6)  | 0.000        |
| <b>O</b> 7 | 0.0177 (7)   | 0.0051 (7)   | 0.0129 (7)   | 0.000        | -0.0021 (5)  | 0.000        |

Geometric parameters (Å, °)

| K                                      | 2.7559 (15) | Al—O3                                     | 1.9021 (11) |
|--|-------------|---|-------------|
| K—O5 <sup>ii</sup>                     | 2.7559 (15) | P1—O1 <sup>ix</sup>                       | 1.4861 (12) |
| K—05                                   | 2.8612 (15) | P1—O3                                     | 1.4956 (12) |
| K—O5 <sup>iii</sup>                    | 2.8612 (15) | P1—O4                                     | 1.5701 (13) |
| K—O2 <sup>iv</sup>                     | 2.9493 (14) | P1—O7                                     | 1.5794 (8)  |
| $K - O2^v$                             | 2.9493 (13) | P2—O5                                     | 1.4622 (14) |
| K—O3 <sup>vi</sup>                     | 3.2431 (13) | P2—O2                                     | 1.4983 (12) |
| K—O3 <sup>vii</sup>                    | 3.2431 (13) | P2—O6                                     | 1.5957 (10) |
| K—O5 <sup>iv</sup>                     | 3.2790 (17) | P2—O4                                     | 1.6070 (13) |
| K—O5 <sup>v</sup>                      | 3.2790 (17) | P2—K <sup>v</sup>                         | 3.4928 (7)  |
| K—P2 <sup>iv</sup>                     | 3.4927 (7)  | O1—P1 <sup>ix</sup>                       | 1.4861 (12) |
| $K - P2^{v}$                           | 3.4927 (7)  | O2—K <sup>v</sup>                         | 2.9494 (13) |
| Al—O2 <sup>viii</sup>                  | 1.8523 (12) | O3—K <sup>x</sup>                         | 3.2431 (13) |
| Al—O2                                  | 1.8523 (12) | O5—K <sup>i</sup>                         | 2.7559 (15) |
| Al—O1 <sup>viii</sup>                  | 1.9013 (12) | O5—K <sup>v</sup>                         | 3.2790 (17) |
| Al—01                                  | 1.9013 (12) | O6—P2 <sup>iv</sup>                       | 1.5957 (10) |
| Al—O3 <sup>viii</sup>                  | 1.9021 (11) | O7—P1 <sup>xi</sup>                       | 1.5793 (8)  |
| O5 <sup>i</sup> —K—O5 <sup>ii</sup>    | 65.84 (6)   | O5—K—P2 <sup>v</sup>                      | 106.81 (4)  |
| O5 <sup>i</sup> —K—O5                  | 99.41 (4)   | $O5^{iii}$ —K—P2 <sup>v</sup>             | 58.01 (3)   |
| O5 <sup>ii</sup> —K—O5                 | 66.08 (6)   | $O2^{iv}$ —K— $P2^{v}$                    | 119.65 (3)  |
| O5 <sup>i</sup> —K—O5 <sup>iii</sup>   | 66.08 (6)   | $O2^{v}$ —K— $P2^{v}$                     | 25.13 (2)   |
| O5 <sup>ii</sup> —K—O5 <sup>iii</sup>  | 99.41 (4)   | $O3^{vi}$ —K—P2 <sup>v</sup>              | 129.83 (3)  |
| 05—K—05 <sup>iii</sup>                 | 63.13 (6)   | $O3^{vii}$ —K— $P2^{v}$                   | 74.69 (2)   |
| $O5^{i}$ —K— $O2^{iv}$                 | 147.08 (5)  | $O5^{iv}$ —K—P2 <sup>v</sup>              | 78.81 (3)   |
| $O5^{ii}$ —K— $O2^{iv}$                | 82.35 (4)   | O5 <sup>v</sup> —K—P2 <sup>v</sup>        | 24.68 (2)   |
| $O5-K-O2^{iv}$                         | 73.61 (4)   | $P2^{iv}$ —K— $P2^{v}$                    | 102.94 (2)  |
| $O5^{iii}$ —K— $O2^{iv}$               | 130.89 (4)  | O2 <sup>viii</sup> —Al—O2                 | 180.0       |
| $O5^{i}$ —K— $O2^{v}$                  | 82.35 (4)   | O2 <sup>viii</sup> —Al—O1 <sup>viii</sup> | 90.00 (6)   |
| $O5^{ii}$ —K— $O2^{v}$                 | 147.08 (5)  | O2—A1—O1 <sup>viii</sup>                  | 89.99 (6)   |
| $O5-K-O2^{v}$                          | 130.89 (4)  | O2 <sup>viii</sup> —Al—O1                 | 89.99 (6)   |
| $O5^{iii}$ —K— $O2^{v}$                | 73.61 (4)   | O2—Al—O1                                  | 90.01 (6)   |
| $O2^{iv}$ —K— $O2^{v}$                 | 126.97 (6)  | O1 <sup>viii</sup> —Al—O1                 | 180.00 (5)  |
| O5 <sup>i</sup> —K—O3 <sup>vi</sup>    | 109.11 (4)  | O2 <sup>viii</sup> —Al—O3 <sup>viii</sup> | 91.53 (5)   |
| $O5^{ii}$ —K— $O3^{vi}$                | 72.48 (4)   | O2—A1—O3 <sup>viii</sup>                  | 88.47 (5)   |
| 05—K—O3 <sup>vi</sup>                  | 112.63 (4)  | O1 <sup>viii</sup> —Al—O3 <sup>viii</sup> | 91.12 (5)   |
| 05 <sup>iii</sup> —K—O3 <sup>vi</sup>  | 171.88 (4)  | O1—Al—O3 <sup>viii</sup>                  | 88.88 (5)   |
| O2 <sup>iv</sup> —K—O3 <sup>vi</sup>   | 49.77 (3)   | O2 <sup>viii</sup> —Al—O3                 | 88.47 (5)   |
| O2 <sup>v</sup> —K—O3 <sup>vi</sup>    | 112.88 (4)  | O2—Al—O3                                  | 91.52 (5)   |
| O5 <sup>i</sup> —K—O3 <sup>vii</sup>   | 72.48 (4)   | O1 <sup>viii</sup> —Al—O3                 | 88.88 (5)   |
| 05 <sup>ii</sup> —K—O3 <sup>vii</sup>  | 109.11 (4)  | O1—Al—O3                                  | 91.12 (5)   |
| 05—K—O3 <sup>vii</sup>                 | 171.88 (4)  | O3 <sup>viii</sup> —Al—O3                 | 180.0       |
| O5 <sup>iii</sup> —K—O3 <sup>vii</sup> | 112.63 (4)  | O1 <sup>ix</sup> —P1—O3                   | 116.38 (7)  |
| O2 <sup>iv</sup> —K—O3 <sup>vii</sup>  | 112.88 (4)  | O1 <sup>ix</sup> —P1—O4                   | 109.98 (7)  |
| O2 <sup>v</sup> —K—O3 <sup>vii</sup>   | 49.77 (3)   | O3—P1—O4                                  | 110.70 (7)  |
| O3 <sup>vi</sup> —K—O3 <sup>vii</sup>  | 70.64 (5)   | O1 <sup>ix</sup> —P1—O7                   | 109.34 (8)  |

| $O5^{i}$ —K— $O5^{iv}$                | 154.67 (6) | O3—P1—O7                          | 109.20 (8)  |
|---------------------------------------|------------|-----------------------------------|-------------|
| O5 <sup>ii</sup> —K—O5 <sup>iv</sup>  | 114.04 (5) | O4—P1—O7                          | 99.98 (8)   |
| $O5-K-O5^{iv}$                        | 61.04 (5)  | O5—P2—O2                          | 117.73 (9)  |
| $O5^{iii}$ —K— $O5^{iv}$              | 89.66 (4)  | O5—P2—O6                          | 111.73 (8)  |
| $O2^{iv}$ —K— $O5^{iv}$               | 47.65 (3)  | O2—P2—O6                          | 107.39 (6)  |
| O2 <sup>v</sup> —K—O5 <sup>iv</sup>   | 98.27 (4)  | O5—P2—O4                          | 111.39 (8)  |
| $O3^{vi}$ —K— $O5^{iv}$               | 94.08 (3)  | O2—P2—O4                          | 108.11 (7)  |
| O3 <sup>vii</sup> —K—O5 <sup>iv</sup> | 126.82 (4) | O6—P2—O4                          | 98.73 (7)   |
| O5 <sup>i</sup> —K—O5 <sup>v</sup>    | 114.04 (5) | O5—P2—K <sup>v</sup>              | 69.45 (6)   |
| O5 <sup>ii</sup> —K—O5 <sup>v</sup>   | 154.67 (6) | O2—P2—K <sup>v</sup>              | 56.71 (5)   |
| O5—K—O5 <sup>v</sup>                  | 89.66 (4)  | O6—P2—K <sup>v</sup>              | 101.18 (5)  |
| $O5^{iii}$ —K— $O5^{v}$               | 61.04 (5)  | O4—P2—K <sup>v</sup>              | 157.99 (5)  |
| $O2^{iv}$ —K— $O5^{v}$                | 98.27 (4)  | P1 <sup>ix</sup> —O1—A1           | 133.95 (8)  |
| O2 <sup>v</sup> —K—O5 <sup>v</sup>    | 47.65 (3)  | P2—O2—Al                          | 138.36 (8)  |
| $O3^{vi}$ —K— $O5^{v}$                | 126.82 (4) | P2—O2—K <sup>v</sup>              | 98.17 (6)   |
| $O3^{vii}$ —K— $O5^{v}$               | 94.08 (3)  | Al—O2—K <sup>v</sup>              | 113.58 (5)  |
| $O5^{iv}$ —K— $O5^{v}$                | 54.36 (5)  | P1—O3—Al                          | 136.54 (8)  |
| $O5^{i}$ — $K$ — $P2^{iv}$            | 155.10 (4) | P1—O3—K <sup>x</sup>              | 120.51 (6)  |
| O5 <sup>ii</sup> —K—P2 <sup>iv</sup>  | 93.25 (3)  | Al—O3—K <sup>x</sup>              | 101.05 (4)  |
| O5—K—P2 <sup>iv</sup>                 | 58.01 (3)  | P1—O4—P2                          | 132.20 (8)  |
| $O5^{iii}$ — $K$ — $P2^{iv}$          | 106.81 (4) | P2K <sup>i</sup>                  | 141.08 (8)  |
| $O2^{iv}$ —K— $P2^{iv}$               | 25.13 (2)  | Р2—О5—К                           | 134.77 (8)  |
| $O2^{v}$ —K— $P2^{iv}$                | 119.65 (3) | K <sup>i</sup> —O5—K              | 80.59 (4)   |
| $O3^{vi}$ —K— $P2^{iv}$               | 74.69 (2)  | P2—O5—K <sup>v</sup>              | 85.87 (6)   |
| $O3^{vii}$ —K— $P2^{iv}$              | 129.83 (3) | K <sup>i</sup> —O5—K <sup>v</sup> | 114.04 (5)  |
| $O5^{iv}$ —K— $P2^{iv}$               | 24.68 (2)  | K—O5—K <sup>v</sup>               | 90.34 (4)   |
| $O5^{v}$ —K— $P2^{iv}$                | 78.81 (3)  | P2—O6—P2 <sup>iv</sup>            | 123.73 (12) |
| $O5^{i}$ — $K$ — $P2^{v}$             | 93.25 (3)  | P1 <sup>xi</sup> —O7—P1           | 138.66 (12) |
| $O5^{ii}$ —K— $P2^{v}$                | 155.10 (4) |                                   |             |
|                                       |            |                                   |             |

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