Structural features of the oxidonitridophosphates K$_3M^{III}$(PO$_3$)$_3$N (M^{III} = Al, Ga)

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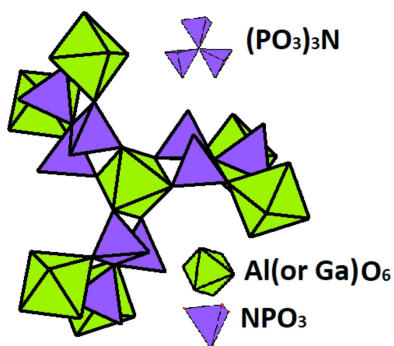
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Cubic crystals of tripotassium aluminium (or gallium) nitridotriphosphate, K$_3M^{III}$(PO$_3$)$_3$N (M^{III} = Al, Ga), were grown by application of the self-flux method. In their isostructural crystal structures, all metal cations and the N atom occupy special positions with site symmetry 3, while the P and O atoms are situated in general positions. The three-dimensional framework of these oxidonitridophosphates is built up from [M^{III}O$_6$] octahedra linked together via (PO$_3$)$_3$N groups. The latter are formed from three PO$_3$N tetrahedra sharing a common N atom. The coordination environments of the three potassium cations are represented by two types of polyhedra, viz. KO$_9$ for one and KO$_9$N for the other two cations. An unusual tetradentate type of coordination for the latter potassium cations by the (PO$_3$)$_3$N^{6-} anion is observed. These K$_3M^{III}$(PO$_3$)$_3$N (M^{III} = Al, Ga) compounds are isostructural with the Na$_3M^{III}$(PO$_3$)$_3$N (M^{III} = Al, V, Ti) compounds.

1. Chemical context

Oxidonitridophosphates with general compositions $M^I_3M^{III}$(PO$_3$)$_3$N and $M^I_2M^{II}_2$(PO$_3$)$_3$N (M^I = Li, Na, K; M^{III} = Al, Cr, Ga, V and Ti; M^{II} = Mg, Fe) have been prepared by solid-state synthesis (Feldmann, 1987a,b; Massiot et al., 1996; Conanec et al., 1996), high-temperature thermal ammonolysis (Kim & Kim, 2013; Kim et al., 2017; Zhang et al., 2017), flux-growth (Zatovsky et al., 2006) or solid–solid ion-exchange (Liu et al., 2018). In recent years, oxidonitridophosphates containing the (PO$_3$)$_3$N^{6-} anion have been intensively studied as promising cathode materials for Na-ion and Li-ion batteries. In particular, ionic conductivities and redox properties were investigated in detail for Na$_3$Ti(PO$_3$)$_3$N (Liu et al., 2014), Na$_3$V(PO$_3$)$_3$N (Reynaud et al., 2017; Kim et al., 2017; Zhang et al., 2017; Xiao et al., 2021; Wang et al., 2021), Li$_3$V(PO$_3$)$_3$N (Liu et al., 2018), Na$_2$Fe$_2$(PO$_3$)$_3$N and Li$_2$Fe$_2$(PO$_3$)$_3$N (Liu et al., 2013), Na$_2$Mg$_2$(PO$_3$)$_3$N (Cosby et al., 2020), and Li$_2$Mg$_2$(PO$_3$)$_3$N (Liu et al., 2017). In addition, rare-earth-doped Na$_3$Al(PO$_3$)$_3$N has been studied as a promising phosphor (Bang et al., 2013). However, the structural data for oxidonitridophosphates include only Na- or Li-containing compounds (Massiot et al., 1996; Zatovsky et al., 2006; Kim & Kim, 2013; Liu et al., 2013, 2017, 2018; Zhang et al., 2017).

In this communication, we report the flux-growth synthesis, structural characterization and FTIR spectra for the two
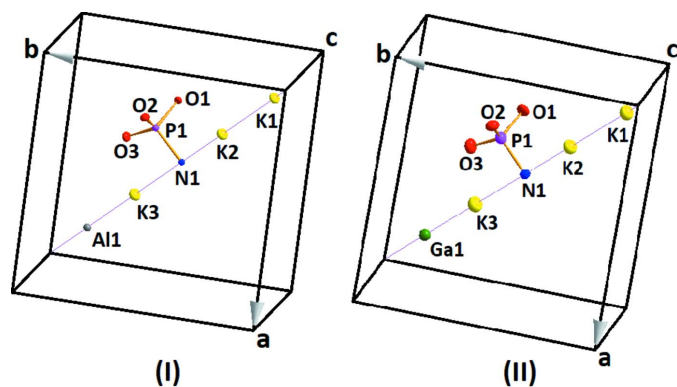

(PO$_3$)$_3$N

Al(or Ga)O$_6$

NPO$_3$

Figure 1
A view of the asymmetric units of $K_3Al(PO_3)_3N$ (I) and $K_3Ga(PO_3)_3N$ (II), with displacement ellipsoids drawn at the 50% probability level.

K-containing oxidonitridophosphates $K_3Al(PO_3)_3N$ (I) and $K_3Ga(PO_3)_3N$ (II).

2. Structural commentary

Compounds (I) and (II) (Fig. 1) are isotypic and crystallize in the cubic $Na_3Al(PO_3)_3N$ structure in space-group type $P2_13$. The K, Al (or Ga) and N atoms are localized on threefold rotation axes (Wyckoff position 4 *a*), and the P and all O atoms occupy general 12 *b* sites (Fig. 1). As shown in Fig. 2, the crystal structures of the $K_3M^{III}(PO_3)_3N$ (M^{III} = Al, Ga) title compounds consist of $[M^{III}O_6]$ octahedra and $(PO_3)_3N^{6-}$ anions, which are linked *via* vertices, forming a three-dimensional framework. The $(PO_3)_3N^{6-}$ anion is built up from three PO_3N tetrahedra sharing a common N vertex atom. The P—O bond lengths for both structures range between 1.473 (9) and 1.534 (3) Å (Tables 1 and 2), while the P—N bond lengths are 1.7084 (12) and 1.701 (4) Å for (I) and (II), respectively. The lengths of the P—O and P—N bonds are similar to those
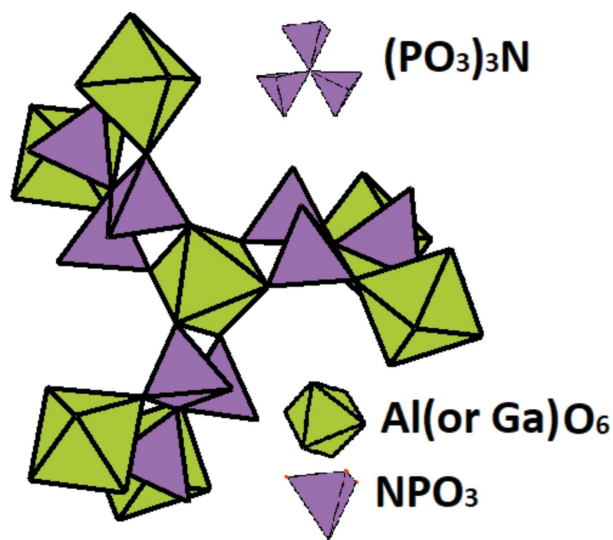

Figure 2
$[M^{III}O_6]$ octahedra and 'three-blade propeller'-type anions $(PO_3)_3N^{6-}$ as principle building units for formation of the three-dimensional framework of (I) and (II).

Table 1
Selected geometric parameters (Å, °) for (I).

K1—O3[i]	2.623 (3)	K3—N1	3.340 (6)
K1—O1[ii]	2.761 (3)	K3—O3[iv]	3.379 (3)
K1—O2[ii]	3.261 (3)	Al1—O1[v]	1.905 (3)
K2—O3[iii]	2.765 (3)	Al1—O2[iv]	1.909 (3)
K2—N1	2.904 (6)	P1—O3	1.493 (3)
K2—O1[iii]	2.967 (3)	P1—O1	1.530 (3)
K2—O2	3.263 (3)	P1—O2	1.534 (3)
K3—O3	2.679 (3)	P1—N1	1.7084 (12)
K3—O2[iv]	2.803 (3)		
O3—P1—O1	111.58 (17)	O1—P1—N1	105.82 (13)
O3—P1—O2	113.45 (16)	O2—P1—N1	105.20 (17)
O1—P1—O2	109.85 (16)	P1[vi]—N1—P1	118.53 (8)
O3—P1—N1	110.5 (2)		

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

Table 2
Selected geometric parameters (Å, °) for (II).

K1—O3[i]	2.642 (8)	K3—N1	3.310 (18)
K1—O1[ii]	2.807 (9)	K3—O3[v]	3.412 (8)
K1—O2[iii]	3.216 (8)	Ga1—O2[vi]	1.968 (9)
K2—O3[iv]	2.808 (9)	Ga1—O2[vii]	1.968 (9)
K2—O1[i]	2.925 (9)	P1—O3	1.473 (9)
K2—N1	2.977 (18)	P1—O2	1.516 (9)
K2—O2	3.287 (8)	P1—O1	1.524 (9)
K3—O3	2.665 (9)	P1—N1	1.701 (4)
K3—O2[v]	2.785 (8)		
O3—P1—O2	113.8 (5)	O2—P1—N1	107.0 (5)
O3—P1—O1	112.1 (5)	O1—P1—N1	106.8 (4)
O2—P1—O1	107.9 (5)	P1—N1—P1[viii]	118.8 (2)
O3—P1—N1	108.9 (7)		

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

found in the isotypic oxidonitridophosphates such as $Na_3Al(PO_3)_3N$ (Conanec *et al.*, 1994, 1996), $Na_3Ti(PO_3)_3N$ (Zatovsky *et al.*, 2006), or $Na_3V(PO_3)_3N$ (Kim & Kim, 2013). The octahedral coordination environments around M^{III} are slightly distorted, as indicated by the different M^{III}—O bond lengths (Tables 1 and 2). The $[M^{III}O_6]$ octahedra are slightly squeezed along the cubic cell diagonal. The average Al—O and Ga—O distances are 1.907 (3) and 1.963 (9) Å, respectively. These values are close to the sums of the ionic radii (Shannon, 1976) of Al^{3+} and O^{2-} (1.92 Å) and Ga^{3+} and O^{2-} (2.00 Å), respectively.

Fig. 3 shows the coordination environments of potassium cations for (I) and (II). K1 has nine O-atom neighbors with
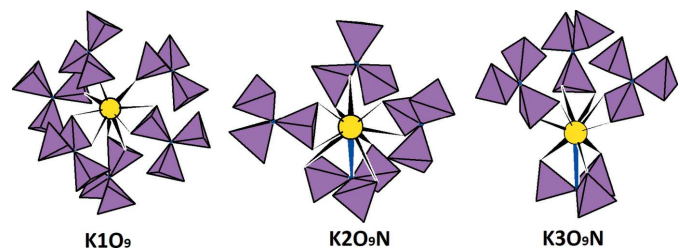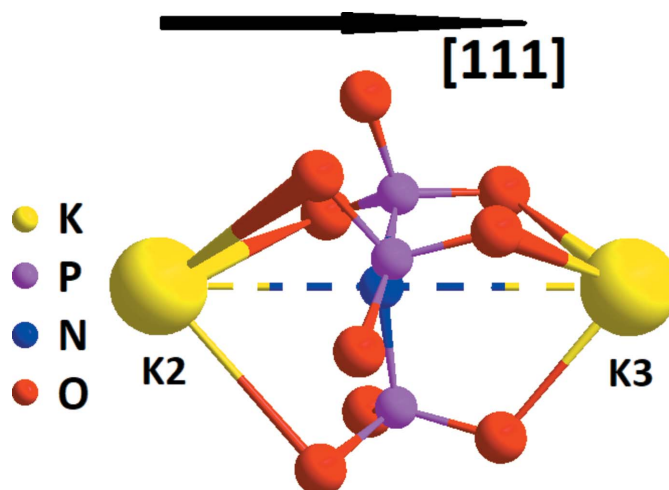

Figure 3
The coordination environment of potassium cations in (I) and (II).

Table 3
BVS results (v.u.) for (I) and (II).

Central Atom	(I)	(II)
Al1/Ga1	3.004	3.197
K1	1.486	1.400
K2	1.122	1.069
K3	1.315	1.360
P1	3 × 4.903	3 × 5.084
Σ	21.636	22.278

K—O distances ranging from 2.623 (3) to 3.261 (3) Å, which includes three mono- and three bidentately coordinating $(PO_3)_3N^{6-}$ anions. $K2O_9N$ and $K3O_9N$ polyhedra are formed as a result of one tetra- and three bidentately coordinating oxidonitridophosphate anions. In the latter case, the upper boundary for K—O distances is 3.412 (8) Å; K2—N distances are 2.904 (6) and 2.977 (18) Å and K3—N contacts are 3.340 (6) and 3.310 (18) Å for (I) and (II), respectively. The coordination environments around the alkali metal for K-containing oxidonitridophosphates ($K2O_9N$ and $K3O_9N$ polyhedra) differ from those of the Na-containing compounds ($Na2O_6N$ and $Na3O_6N$ polyhedra). In addition, the $(PO_3)_3N^{6-}$ anions coordinate the two potassium cations in a tetradentate manner (Fig. 4). As is shown schematically in Fig. 4 and in Table 3 for the isotypic $M^I_3M^{III}(PO_3)_3N$ compounds, the M^I, M^{III} and N atoms are arranged along the [111] direction in the sequence $-M1^I-M2^I-N-M3^I-M^{III}-M1^I-$ whereby the $M2^I-N-M3^I$ distances change in a different manner. In case of (I) and (II), the shape of the $(PO_3)_3N^{6-}$ anion is similar (the P—N distances are about 1.70 Å and the P—N—K3 angles are within 83-84°; Tables 1 and 2). For the Na-containing analogues, the P—N bond is slightly larger (1.71–1.74 Å) and the P—N—Na3 angles are smaller within a wider range from 75 to 78° (Conanec *et al.*, 1994, 1996; Zatovsky *et al.*, 2006; Kim & Kim, 2013).

To clarify some points regarding the structural changes we have calculated the bond-valence sums (BVS) for the K, P and Al atoms (I) and Ga atoms (II), respectively. Parameters were taken from Brown & Altermatt (1985) and for the K—N bond from Brese & O'Keeffe (1991). The BVS for positively charged atoms in (I) is 21.64 v.u. and 22.28 v.u. for (II) while the sum of the charges of nine O and one N atom is equal to 21 v.u. (Table 4). The higher values for the Ga-containing compound might be explained as follows. The BVS for Al in $[AlO_6]$ was found to be 3.00 v.u. while for Ga in $[GaO_6]$ it is 3.20 v.u. The remaining atoms also show a slight overbonding (Table 4). We suppose that the anionic part $(PO_3)_3N^{6-}$ is rigid enough and cannot be stretched to larger sizes relative to the

Figure 4
Coordination of K2 and K3 cations by the $(PO_3)_3N^{6-}$ anion for (I) and (II).

larger $[GaO_6]$ octahedron into a more expanded framework. This is the reason why shorter interatomic K—O and P—O distances are observed in the structure of (II) compared to that of (I) (Tables 1 and 2). As expected, the unit-cell parameters of (I) are smaller than for (II), in good agreement with the ionic radii of Al^{3+} and Ga^{3+} (Shannon, 1976). In other words, the rigid and almost flat 'three-blade propeller' anions combine with $[M^{III}O_6]$ octahedra to form the framework in which the cavities for the alkali cations become smaller as greater octahedra are involved. Moreover, the greater $[M^{III}O_6]$ octahedra strongly influence the 'three-blade propeller' anion, resulting in slightly shorter P—O and P—N bonds. On the other hand, the local environments of the K cations should also be mentioned. In the $K2O_9N$ polyhedron, the BVS for K2 is 1.12 v.u. in (I) and 1.07 in (II). The contribution to the K2—N1 bond to the valence sum is 0.12 v.u. for (I) and 0.10 for (II). These high values indicate a strong interaction between the two atoms. The cavities in which K1 and K2 are located become larger with longer or almost the same K⋯O and K⋯N contacts, while the cavities in which K3 is situated become smaller with shortened K⋯O contacts in (II) in comparison with (I). BVS calculations for the phosphate tetrahedra show similar results (Table 4).

It should also be noted that further theoretical calculations of the electronic structure can bring final clarity to the principles of bonding of alkali cations with $(PO_3)_3N^{6-}$ anions in $K_3M^{III}(PO_3)_3N$ compounds. This could be a way for the creation of new materials with desired properties based on K-containing oxidonitridophosphates.

Table 4
Distances (Å) between atoms for (I), (II) and isotypic $M^I_3M^{III}(PO_3)_3N$ compounds along [111].

Compound	Atomic distance between neighboring atoms	Reference
$Na_3Al(PO_3)_3N$	–Na1–3.438–Na2–2.875–N–3.197–Na3–3.068–Al–3.486–Na1–	Massiot *et al.* (1996)
$Na_3Ti(PO_3)_3N$	–Na1–3.448–Na2–3.078–N–3.188–Na3–3.100–Ti–3.638–Na1–	Zatovsky *et al.* (2006)
$Na_3V(PO_3)_3N$	–Na1–3.477–Na2–2.947–N–3.234–Na3–3.100–V–3.606–Na1–	Kim & Kim (2013)
$K_3Al(PO_3)_3N$	–K1–3.723–K2–2.904–N–3.340–K3–3.400–Al–3.429–K1–	This work
$K_3Ga(PO_3)_3N$	–K1–3.747–K2–2.978–N–3.310–K3–3.350–Ga–3.470–K1–	This work

3. Synthesis and crystallization

For the synthesis of (I) and (II), KH_2PO_4, $K_4P_2O_7 \cdot 10H_2O$, urea, Al_2O_3 or Ga_2O_3 (all analytically or extra pure grade) were used as initial reagents. The sequence of preparation procedure was as follows: (1) phosphates KPO_3 and $K_4P_2O_7$ were each prepared by calcining KH_2PO_4 and $K_4P_2O_7 \cdot 10H_2O$ at 873 K; (2) a mixture of 20.07 g of KPO_3, 13.21 g of $K_4P_2O_7$ and 30.03 g of urea (molar ratio K:P = 1:1.32, urea:P = 2:1) ground in an agate mortar, was heated to complete degassing at 623 K in a porcelain dish. The resulting solid was reground and heated to become a homogeneous liquid at 1023 K and then quenched by pouring the melt onto a copper sheet to form a glass. The glass was crushed using a mill, and a powder with a particle size of less than 125 μm was separated. According to chemical analysis, the prepared glass had the composition $K_{1.32}PO_{2.43}N_{0.50}$; (3) a mixture of 10 g of glass $(K_{1.32}PO_{2.43}N_{0.50})$ and 0.3 g of Al_2O_3 or 0.7 g of Ga_2O_3 powders were placed into porcelain crucibles and heated up to 1043 K and then cooled to 923 K at a rate of 25 K h^{-1}. After cooling to room temperature, colorless tetrahedral crystals of (I) or (II) were washed with deionized water. Elemental analysis indicated the presence of K, Al (or Ga), P and N in the atomic ratio 3:1:3:1.

The growing of well-shaped crystals of (I) and (II) suitable for single crystal X-ray diffraction analysis was one of main tasks during the present study. Hence, a similar way for the preparation of the potassium-containing phosphates to that for the previously reported sodium-containing compounds was applied, following the self-flux method for the preparation of crystalline nitrogen-containing phosphates (Zatovsky *et al.*, 2006). Thermal decomposition of urea is a multistage process

Figure 5
FTIR spectra of (I) and (II).

and leads to the formation of C_3N_4 (Wang *et al.*, 2017). The initial M^I–P–O–N (M^I = alkali metal) melt can be obtained by the reaction of urea with alkali metal phosphates, when a mixture of phosphates and C_3N_4 interact. The change of the phosphate:C_3N_4 ratio (or phosphate:urea) and the nature of the alkali metal affects the composition of the resulting melt. In our case, the molar ratio of K:P was chosen to be 1:1.32 because a mixture of KPO_3 and $K_4P_2O_7$ in this ratio has the lowest melting point close to 886 K, and the urea:P ratio was set to 2:1. As a result, a glass of composition $K_{1.32}PO_{2.43}N_{0.50}$ was obtained.

The solubilities of Al_2O_3 and Ga_2O_3 in the $K_{1.32}PO_{2.43}N_{0.50}$ self-fluxes differ significantly. Crystallization of compound (I) occurs as a result of the interaction of self-fluxes and 2–4%wt. Al_2O_3. The formation of a mixture of (I) and Al_2O_3 was observed when the initial amount of aluminum oxide was higher than 5%wt. The solubility of Ga_2O_3 in the self-flux is about 7%wt. at 1043 K, and subsequent cooling of the homogeneous melt led to the crystallization of compound (II). In all cases, the amount of nitrogen in the self-fluxes rapidly decreases above 1063 K. This process occurs due to the thermal instability of P—N bonds at high temperatures, and leads to a redox reaction with the release of nitrogen and phosphorus. The latter vaporizes from the phosphate melts and starts to burn, which can be observed by periodical sparks on the melt surface (Zatovsky *et al.*, 2000). As a result, K–M^{III}–P–O (M^{III} = Al, Ga) melts prone to vitrifying are formed.

The obtained compounds (I) and (II) were further characterized using FTIR spectroscopy; FTIR spectra were collected at room temperature on KBr discs using a Thermo NICOLET Nexus 470 spectrophotometer. As can be seen in Fig. 5, the spectra of (I) and (II) are similar with respect to intensities and band positions (the difference in the band positions does not exceed 27 cm^{-1}). The characteristic bands are in good agreement with the presence of the $N(PO_3)_3^{6-}$ anion with C_{3v} symmetry, which provides for a set of vibration modes: $6A1 + 5A2 + 11E$ ($3A1 + A2 + 4E$ belong to stretching vibrations, and $3A1 + 4A2 + 7E$ are due to deformation vibrations). As shown in Fig. 5, the following regions can be distinguished in the FTIR spectra: (1) the bands in the region between 980 and 1220 cm^{-1} are assigned to ν_{as} and $\nu_s(PO_3)$ stretching vibrations [four absorption bands in the frequency range between 1070 and 1220 cm^{-1} belong to $\nu_{as}(P-O)$, and two bands of between 980 and 1060 cm^{-1} correspond to $\nu_s(P-O)$]; (2) $\nu_{as}(P-N-P)$ and $\nu_s(P-N-P)$ bands can be observed in the regions between 920 and 950 cm^{-1} and around 920 cm^{-1}, respectively; (3) the range between 400 and 680 cm^{-1} includes absorption bands due to δ (P—O) and ν (Al—O) or ν (Ga—O) vibrations. In summary, in terms of the set of absorption bands, the FTIR spectra of the $N(PO_3)_3^{6-}$ anion resemble those of the $P_2O_7^{4-}$ anion.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. As a result of the shapes of the obtained crystals, their cell parameters and chemical compo-
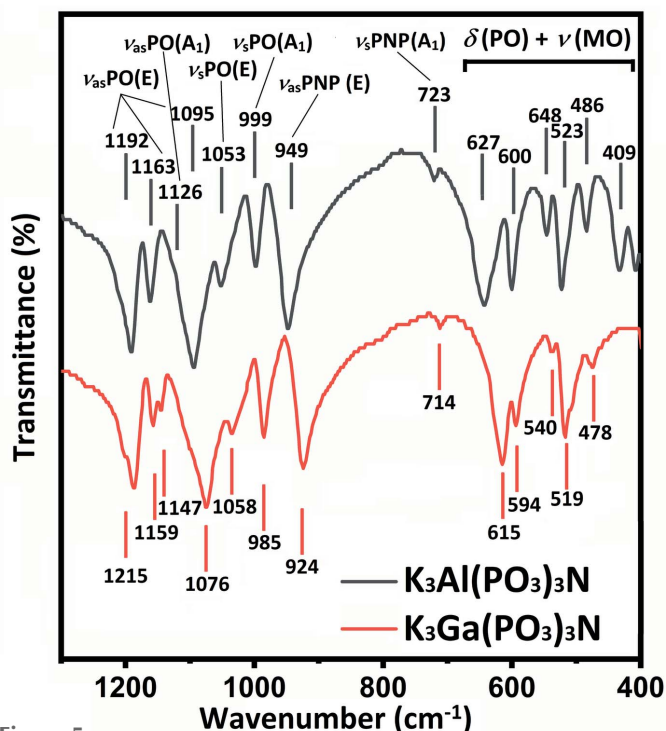
Table 5
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$K_3Al(NP_3O_9)$	$K_3Ga(NP_3O_9)$
M_r	395.2	437.94
Crystal system, space group	Cubic, $P2_13$	Cubic, $P2_13$
Temperature (K)	293	293
a (Å)	9.6970 (4)	9.7313 (9)
V (Å^3)	911.83 (11)	921.5 (3)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.16	4.90
Crystal size (mm)	$0.15 \times 0.12 \times 0.1$	$0.12 \times 0.07 \times 0.04$
Data collection		
Diffractometer	Oxford Diffraction Xcalibur-3	Oxford Diffraction Xcalibur-3
Absorption correction	Multi-scan (Blessing, 1995)	Multi-scan (Blessing, 1995)
T_{min}, T_{max}	0.844, 0.869	0.738, 0.804
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7172, 745, 667	4964, 753, 517
R_{int}	0.078	0.155
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.660	0.660
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.026, 0.052, 1.05	0.059, 0.102, 1
No. of reflections	745	753
No. of parameters	53	53
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.27	0.67, −0.60
Absolute structure	Flack x determined using 269 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)	Flack x determined using 153 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter	−0.02 (6)	0.03 (5)

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2006), *CrysAlis RED* (Oxford Diffraction, 2006), *SHELXS* (Sheldrick, 2008), *SHELXS2013/1* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2006), *WinGX* (Farrugia, 2012), *enCIFer* (Allen et al., 2004) and *publCIF* (Westrip, 2010).

sitions, we had expected that both structures should be isostructural with the previously reported $Na_3Al(PO_3)_3N$ and $Na_3Ti(PO_3)_3N$ structures. In fact, analysis of the single-crystal data showed that both compounds crystallize in the same space group type ($P2_13$) as the Na-containing oxidoni-tridophosphates. Originally, the crystal structures were solved by direct methods but we also performed refinements using the atomic coordinates of $Na_3Ti(PO_3)_3N$ as a starting model. The results were the same, confirming that both structures are isostructural with $Na_3Ti(PO_3)_3N$ (as well as with all previously reported cubic oxionitridophosphates with the same formula type).

References

Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.

Bang, S. Y., Khai, T. V., Oh, D. K., Maneeratanasarn, P., Choi, B. G., Ham, H. & Shim, K. B. (2013). J. Ceram. Process. Res. 14(S1), 74–76.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Brese, N. E. & O'Keeffe, M. (1991). *Acta Cryst.* B**47**, 192–197.

Brown, I. D. & Altermatt, D. (1985). *Acta Cryst.* B**41**, 244–247.

Conanec, R., Feldmann, W., Marchand, R. & Laurent, Y. (1996). *J. Solid State Chem.* **121**, 418–422.

Conanec, R., l'Haridon, P., Feldmann, W., Marchand, R. & Laurent, Y. (1994). *Eur. J. Solid State Inorg. Chem.* **31**, 13–24.

Cosby, M. R., Mattei, G. S., Wang, Y., Li, Z., Bechtold, N., Chapman, K. W. & Khalifah, K. W. (2020). *J. Phys. Chem. C*, **124**, 6522–6527.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Feldmann, W. (1987a). *Z. Chem.* **27**, 100–101.

Feldmann, W. (1987b). *Z. Chem.* **27**, 182–183.

Kim, J., Yoon, G., Lee, M. H., Kim, H., Lee, S. & Kang, K. (2017). *Chem. Mater.* **29**, 7826–7832.

Kim, M. & Kim, S.-J. (2013). *Acta Cryst.* E**69**, i34.

Liu, J., Chang, D., Whitfield, P., Janssen, Y., Yu, X., Zhou, Y., Bai, J., Ko, J., Nam, K.-W., Wu, L., Zhu, Y., Feygenson, M., Amatucci, G., Van der Ven, A., Yang, X.-Q. & Khalifah, P. (2014). *Chem. Mater.* **26**, 3295–3305.

Liu, J., Whitfield, P. S., Saccomanno, M. R., Bo, S.-H., Hu, E., Yu, X., Bai, J., Grey, C. P., Yang, X.-Q. & Khalifah, P. G. (2017). *J. Am. Chem. Soc.* **139**, 9192–9202.

Liu, J., Yin, L., Yang, X.-Q. & Khalifah, P. G. (2018). *Chem. Mater.* **30**, 4609–4616.

Liu, J., Yu, X., Hu, E., Nam, K.-W., Yang, X.-Q. & Khalifah, P. G. (2013). *Chem. Mater.* **25**, 3929–3931.

Massiot, D., Conanec, R., Feldmann, W., Marchand, R. & Laurent, Y. (1996). *Inorg. Chem.* **35**, 4957–4960.

Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Reynaud, M., Wizner, A., Katcho, N. A., Loaiza, L. C., Galceran, M., Carrasco, J., Rojo, T., Armand, M. & Casas-Cabanas, M. (2017). *Electrochem. Commun.* **84**, 14–18.

Shannon, R. D. (1976). *Acta Cryst.* A**32**, 751–767.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Wang, A., Wang, C., Fu, L., Wong-Ng, W. & Lan, Y. (2017). *Nano-Micro Lett.* **9**, 47.

research communications

Wang, S., Li, H., Zhang, W., Zheng, J., Li, S., Hu, J., Lia, Y. & Zhang, Z. (2021). *ACS Appl. Energy Mater.* In the press. https://doi.org/10.1021/acsaem.1c02042.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Xiao, J., Hua, W. & Chen, M. (2021). *Nanotechnology*, **32**, 425404.

Zatovsky, I. V., Slobodyanik, N. S., Stratiychuk, D. A., Domasevitch, K. V., Sieler, J. & Rusanov, E. B. (2000). *Z. Naturforsch. Teil B*, **55**, 291–298.

Zatovsky, I. V., Vorobjova, T. V., Domasevitch, K. V., Ogorodnyk, I. V. & Slobodyanik, N. S. (2006). *Acta Cryst.* E**62**, i32–i34.

Zhang, H., Buchholz, D. & Passerini, S. (2017). *Energies*, **10**, 889.

supporting information

Structural features of the oxidonitridophosphates K₃*M*ᴵᴵᴵ(PO₃)₃N (*M*ᴵᴵᴵ = Al, Ga)

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Computing details

For both structures, data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis CCD* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED* (Oxford Diffraction, 2006). Program(s) used to solve structure: *SHELXS* (Sheldrick, 2008) for (I); *SHELXS2013*/1 (Sheldrick, 2008) for (II). Program(s) used to refine structure: *SHELXL* (Sheldrick, 2015) for (I); *SHELXL2018*/3 (Sheldrick, 2015) for (II). For both structures, molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 2012), *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2010).

Tripotassium aluminium nitridotriphosphate (I)

Crystal data

K₃Al(NP₃O₉)	$D_x = 2.879$ Mg m⁻³
$M_r = 395.2$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cubic, $P2_13$	Cell parameters from 7172 reflections
Hall symbol: P 2ac 2ab 3	$\theta = 3.0$–28.0°
$a = 9.6970$ (4) Å	$\mu = 2.16$ mm⁻¹
$V = 911.83$ (11) Å³	$T = 293$ K
$Z = 4$	Tetrahedron, colorless
$F(000) = 776$	$0.15 \times 0.12 \times 0.1$ mm

Data collection

Oxford Diffraction Xcalibur-3 diffractometer	745 independent reflections
Graphite monochromator	667 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.078$
Absorption correction: multi-scan (Blessing, 1995)	$\theta_{max} = 28.0°$, $\theta_{min} = 3.0°$
$T_{min} = 0.844$, $T_{max} = 0.869$	$h = -12\rightarrow12$
7172 measured reflections	$k = -12\rightarrow10$
	$l = -12\rightarrow12$

Refinement

Refinement on F^2	$'w = 1/[\sigma^2(F_o^2) + (0.0211P)^2]$
Least-squares matrix: full	where $P = (F_o^2 + 2F_c^2)/3'$
$R[F^2 > 2\sigma(F^2)] = 0.026$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.052$	$\Delta\rho_{max} = 0.27$ e Å⁻³
$S = 1.05$	$\Delta\rho_{min} = -0.27$ e Å⁻³
745 reflections	Extinction correction: SHELXL2018/3
53 parameters	(Sheldrick, 2015)
0 restraints	Extinction coefficient: 0.0037 (14)

Absolute structure: Flack x determined using
269 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al.,
2013)

Absolute structure parameter: −0.02 (6)

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
K1	0.04655 (9)	0.04655 (9)	0.04655 (9)	0.0178 (4)
K2	0.26820 (10)	0.26820 (10)	0.26820 (10)	0.0195 (4)
K3	0.63996 (10)	0.63996 (10)	0.63996 (10)	0.0207 (4)
Al1	0.84238 (12)	0.84238 (12)	0.84238 (12)	0.0095 (5)
P1	0.32280 (11)	0.56862 (11)	0.46909 (11)	0.0088 (3)
N1	0.4411 (3)	0.4411 (3)	0.4411 (3)	0.0073 (11)
O1	0.2129 (3)	0.5055 (3)	0.5631 (3)	0.0107 (6)
O2	0.2606 (3)	0.5994 (3)	0.3269 (3)	0.0120 (6)
O3	0.3898 (3)	0.6909 (3)	0.5344 (3)	0.0161 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0178 (4)	0.0178 (4)	0.0178 (4)	−0.0037 (4)	−0.0037 (4)	−0.0037 (4)
K2	0.0195 (4)	0.0195 (4)	0.0195 (4)	−0.0034 (4)	−0.0034 (4)	−0.0034 (4)
K3	0.0207 (4)	0.0207 (4)	0.0207 (4)	−0.0040 (4)	−0.0040 (4)	−0.0040 (4)
Al1	0.0095 (5)	0.0095 (5)	0.0095 (5)	−0.0006 (5)	−0.0006 (5)	−0.0006 (5)
P1	0.0081 (5)	0.0087 (6)	0.0096 (5)	0.0003 (4)	0.0010 (4)	0.0003 (4)
N1	0.0073 (11)	0.0073 (11)	0.0073 (11)	0.0001 (13)	0.0001 (13)	0.0001 (13)
O1	0.0103 (15)	0.0108 (14)	0.0110 (14)	0.0004 (11)	0.0043 (12)	0.0021 (12)
O2	0.0116 (15)	0.0147 (15)	0.0099 (14)	0.0022 (12)	−0.0019 (12)	0.0007 (12)
O3	0.0163 (16)	0.0116 (16)	0.0204 (16)	−0.0023 (12)	−0.0019 (14)	−0.0037 (13)

Geometric parameters (Å, º)

K1—O3^i	2.623 (3)	K2—P1^{ii}	3.4199 (12)
K1—O3^{ii}	2.623 (3)	K3—O3^{viii}	2.679 (3)
K1—O3^{iii}	2.623 (3)	K3—O3^{ix}	2.679 (3)
K1—O1^{iv}	2.761 (3)	K3—O3	2.679 (3)
K1—O1^v	2.761 (3)	K3—O2^x	2.803 (3)
K1—O1^{vi}	2.761 (3)	K3—O2^{xi}	2.803 (3)
K1—O2^{iv}	3.261 (3)	K3—O2^{xii}	2.803 (3)
K1—O2^{vi}	3.261 (3)	K3—N1	3.340 (6)
K1—O2^v	3.261 (3)	K3—O3^x	3.379 (3)
K1—Al1^{vii}	3.429 (3)	K3—O3^{xi}	3.379 (3)

K1—P1^{iv}	3.5911 (13)	K3—O3^{xii}	3.379 (3)
K1—P1^{vi}	3.5911 (13)	K3—Al1	3.400 (3)
K2—O3^{iii}	2.765 (3)	K3—P1^{xii}	3.4997 (14)
K2—O3^{i}	2.765 (3)	Al1—O1^{xiii}	1.905 (3)
K2—O3^{ii}	2.765 (3)	Al1—O1^{xiv}	1.905 (3)
K2—N1	2.904 (6)	Al1—O1^{xv}	1.905 (3)
K2—O1^{iii}	2.967 (3)	Al1—O2^{x}	1.909 (3)
K2—O1^{i}	2.967 (3)	Al1—O2^{xi}	1.909 (3)
K2—O1^{ii}	2.967 (3)	Al1—O2^{xii}	1.909 (3)
K2—O2	3.263 (3)	P1—O3	1.493 (3)
K2—O2^{viii}	3.263 (3)	P1—O1	1.530 (3)
K2—O2^{ix}	3.263 (3)	P1—O2	1.534 (3)
K2—P1^{iii}	3.4199 (12)	P1—N1	1.7084 (12)
O3^{i}—K1—O3^{ii}	79.95 (10)	O2^{x}—K3—O3^{xi}	60.45 (8)
O3^{i}—K1—O3^{iii}	79.95 (10)	O2^{xi}—K3—O3^{xi}	47.20 (7)
O3^{ii}—K1—O3^{iii}	79.95 (10)	O2^{xii}—K3—O3^{xi}	99.58 (8)
O3^{i}—K1—O1^{iv}	111.53 (9)	N1—K3—O3^{xi}	113.91 (5)
O3^{ii}—K1—O1^{iv}	165.73 (9)	O3^{x}—K3—O3^{xi}	104.69 (6)
O3^{iii}—K1—O1^{iv}	109.72 (8)	O3^{viii}—K3—O3^{xii}	143.68 (7)
O3^{i}—K1—O1^{v}	109.72 (8)	O3^{ix}—K3—O3^{xii}	66.44 (10)
O3^{ii}—K1—O1^{v}	111.53 (9)	O3—K3—O3^{xii}	111.63 (2)
O3^{iii}—K1—O1^{v}	165.73 (9)	O2^{x}—K3—O3^{xii}	99.58 (8)
O1^{iv}—K1—O1^{v}	57.44 (9)	O2^{xi}—K3—O3^{xii}	60.45 (8)
O3^{i}—K1—O1^{vi}	165.73 (9)	O2^{xii}—K3—O3^{xii}	47.20 (7)
O3^{ii}—K1—O1^{vi}	109.72 (8)	N1—K3—O3^{xii}	113.91 (5)
O3^{iii}—K1—O1^{vi}	111.53 (9)	O3^{x}—K3—O3^{xii}	104.69 (6)
O1^{iv}—K1—O1^{vi}	57.44 (9)	O3^{xi}—K3—O3^{xii}	104.69 (6)
O1^{v}—K1—O1^{vi}	57.44 (9)	O3^{viii}—K3—Al1	129.57 (6)
O3^{i}—K1—O2^{iv}	63.69 (8)	O3^{ix}—K3—Al1	129.57 (6)
O3^{ii}—K1—O2^{iv}	143.60 (8)	O3—K3—Al1	129.57 (6)
O3^{iii}—K1—O2^{iv}	94.55 (8)	O2^{x}—K3—Al1	34.15 (6)
O1^{iv}—K1—O2^{iv}	48.34 (7)	O2^{xi}—K3—Al1	34.15 (6)
O1^{v}—K1—O2^{iv}	81.13 (8)	O2^{xii}—K3—Al1	34.15 (6)
O1^{vi}—K1—O2^{iv}	105.77 (8)	N1—K3—Al1	180.00 (9)
O3^{i}—K1—O2^{vi}	143.60 (8)	O3^{x}—K3—Al1	66.09 (5)
O3^{ii}—K1—O2^{vi}	94.55 (8)	O3^{xi}—K3—Al1	66.09 (5)
O3^{iii}—K1—O2^{vi}	63.69 (8)	O3^{xii}—K3—Al1	66.09 (5)
O1^{iv}—K1—O2^{vi}	81.13 (8)	O3^{viii}—K3—P1^{xii}	168.66 (7)
O1^{v}—K1—O2^{vi}	105.77 (8)	O3^{ix}—K3—P1^{xii}	86.65 (6)
O1^{vi}—K1—O2^{vi}	48.34 (7)	O3—K3—P1^{xii}	101.22 (6)
O2^{iv}—K1—O2^{vi}	115.34 (4)	O2^{x}—K3—P1^{xii}	82.96 (7)
O3^{i}—K1—O2^{v}	94.55 (8)	O2^{xi}—K3—P1^{xii}	64.96 (6)
O3^{ii}—K1—O2^{v}	63.69 (8)	O2^{xii}—K3—P1^{xii}	25.22 (6)
O3^{iii}—K1—O2^{v}	143.60 (8)	N1—K3—P1^{xii}	125.75 (3)
O1^{iv}—K1—O2^{v}	105.77 (8)	O3^{x}—K3—P1^{xii}	79.71 (6)
O1^{v}—K1—O2^{v}	48.34 (7)	O3^{xi}—K3—P1^{xii}	112.01 (6)
O1^{vi}—K1—O2^{v}	81.13 (8)	O3^{xii}—K3—P1^{xii}	24.99 (5)

Angle	Value	Angle	Value
O2^{iv}—K1—O2^{v}	115.34 (4)	Al1—K3—P1^{xii}	54.25 (3)
O2^{vi}—K1—O2^{v}	115.34 (4)	O1^{xiii}—Al1—O1^{xiv}	88.28 (14)
O3^{i}—K1—Al1^{vii}	132.11 (7)	O1^{xiii}—Al1—O1^{xv}	88.28 (14)
O3^{ii}—K1—Al1^{vii}	132.11 (7)	O1^{xiv}—Al1—O1^{xv}	88.28 (14)
O3^{iii}—K1—Al1^{vii}	132.11 (7)	O1^{xiii}—Al1—O2^{x}	92.92 (12)
O1^{iv}—K1—Al1^{vii}	33.70 (6)	O1^{xiv}—Al1—O2^{x}	175.81 (12)
O1^{v}—K1—Al1^{vii}	33.70 (6)	O1^{xv}—Al1—O2^{x}	87.74 (11)
O1^{vi}—K1—Al1^{vii}	33.70 (6)	O1^{xiii}—Al1—O2^{xi}	87.74 (11)
O2^{iv}—K1—Al1^{vii}	77.34 (5)	O1^{xiv}—Al1—O2^{xi}	92.92 (12)
O2^{vi}—K1—Al1^{vii}	77.34 (5)	O1^{xv}—Al1—O2^{xi}	175.81 (12)
O2^{v}—K1—Al1^{vii}	77.34 (5)	O2^{x}—Al1—O2^{xi}	91.14 (13)
O3^{i}—K1—P1^{iv}	88.98 (7)	O1^{xiii}—Al1—O2^{xii}	175.81 (12)
O3^{ii}—K1—P1^{iv}	168.77 (7)	O1^{xiv}—Al1—O2^{xii}	87.74 (11)
O3^{iii}—K1—P1^{iv}	100.08 (6)	O1^{xv}—Al1—O2^{xii}	92.92 (12)
O1^{iv}—K1—P1^{iv}	23.55 (6)	O2^{x}—Al1—O2^{xii}	91.14 (14)
O1^{v}—K1—P1^{iv}	70.37 (6)	O2^{xi}—Al1—O2^{xii}	91.14 (13)
O1^{vi}—K1—P1^{iv}	80.83 (7)	O1^{xiii}—Al1—K3	126.47 (10)
O2^{iv}—K1—P1^{iv}	25.29 (5)	O1^{xiv}—Al1—K3	126.47 (10)
O2^{vi}—K1—P1^{iv}	95.49 (6)	O1^{xv}—Al1—K3	126.47 (10)
O2^{v}—K1—P1^{iv}	115.87 (6)	O2^{x}—Al1—K3	55.54 (10)
Al1^{vii}—K1—P1^{iv}	55.54 (3)	O2^{xi}—Al1—K3	55.54 (10)
O3^{i}—K1—P1^{vi}	168.77 (7)	O2^{xii}—Al1—K3	55.54 (10)
O3^{ii}—K1—P1^{vi}	100.08 (6)	O1^{xiii}—Al1—K1^{xvi}	53.53 (10)
O3^{iii}—K1—P1^{vi}	88.98 (7)	O1^{xiv}—Al1—K1^{xvi}	53.53 (10)
O1^{iv}—K1—P1^{vi}	70.37 (6)	O1^{xv}—Al1—K1^{xvi}	53.53 (10)
O1^{v}—K1—P1^{vi}	80.83 (7)	O2^{x}—Al1—K1^{xvi}	124.46 (10)
O1^{vi}—K1—P1^{vi}	23.55 (6)	O2^{xi}—Al1—K1^{xvi}	124.46 (10)
O2^{iv}—K1—P1^{vi}	115.87 (6)	O2^{xii}—Al1—K1^{xvi}	124.46 (10)
O2^{vi}—K1—P1^{vi}	25.29 (5)	K3—Al1—K1^{xvi}	180.00 (10)
O2^{v}—K1—P1^{vi}	95.49 (6)	O1^{xiii}—Al1—K2^{xv}	45.00 (8)
Al1^{vii}—K1—P1^{vi}	55.54 (3)	O1^{xiv}—Al1—K2^{xv}	125.19 (12)
P1^{iv}—K1—P1^{vi}	91.14 (4)	O1^{xv}—Al1—K2^{xv}	67.60 (8)
O3^{iii}—K2—O3^{i}	75.10 (9)	O2^{x}—Al1—K2^{xv}	54.13 (8)
O3^{iii}—K2—O3^{ii}	75.10 (9)	O2^{xi}—Al1—K2^{xv}	108.53 (8)
O3^{i}—K2—O3^{ii}	75.10 (9)	O2^{xii}—Al1—K2^{xv}	139.06 (9)
O3^{iii}—K2—N1	135.27 (6)	K3—Al1—K2^{xv}	106.68 (3)
O3^{i}—K2—N1	135.27 (6)	K1^{xvi}—Al1—K2^{xv}	73.32 (3)
O3^{ii}—K2—N1	135.27 (6)	O1^{xiii}—Al1—K2^{xvii}	67.60 (8)
O3^{iii}—K2—O1^{iii}	51.57 (8)	O1^{xiv}—Al1—K2^{xvii}	45.00 (8)
O3^{i}—K2—O1^{iii}	123.82 (8)	O1^{xv}—Al1—K2^{xvii}	125.19 (12)
O3^{ii}—K2—O1^{iii}	103.08 (8)	O2^{x}—Al1—K2^{xvii}	139.06 (9)
N1—K2—O1^{iii}	85.66 (6)	O2^{xi}—Al1—K2^{xvii}	54.13 (8)
O3^{iii}—K2—O1^{i}	103.08 (8)	O2^{xii}—Al1—K2^{xvii}	108.53 (8)
O3^{i}—K2—O1^{i}	51.57 (8)	K3—Al1—K2^{xvii}	106.68 (3)
O3^{ii}—K2—O1^{i}	123.82 (8)	K1^{xvi}—Al1—K2^{xvii}	73.32 (3)
N1—K2—O1^{i}	85.66 (6)	K2^{xv}—Al1—K2^{xvii}	112.11 (3)
O1^{iii}—K2—O1^{i}	119.434 (16)	O1^{xiii}—Al1—K2^{xii}	125.19 (12)
O3^{iii}—K2—O1^{ii}	123.82 (8)	O1^{xiv}—Al1—K2^{xii}	67.60 (8)

O3ⁱ—K2—O1ⁱⁱ	103.08 (8)	O1^{xv}—Al1—K2^{xii}	45.00 (8)
O3ⁱⁱ—K2—O1ⁱⁱ	51.57 (8)	O2^x—Al1—K2^{xii}	108.53 (8)
N1—K2—O1ⁱⁱ	85.66 (6)	O2^{xi}—Al1—K2^{xii}	139.06 (9)
O1ⁱⁱⁱ—K2—O1ⁱⁱ	119.434 (16)	O2^{xii}—Al1—K2^{xii}	54.13 (8)
O1ⁱ—K2—O1ⁱⁱ	119.434 (16)	K3—Al1—K2^{xii}	106.68 (3)
O3ⁱⁱⁱ—K2—O2	120.12 (8)	K1^{xvi}—Al1—K2^{xii}	73.32 (3)
O3ⁱ—K2—O2	155.11 (7)	K2^{xv}—Al1—K2^{xii}	112.11 (3)
O3ⁱⁱ—K2—O2	89.38 (7)	K2^{xvii}—Al1—K2^{xii}	112.11 (3)
N1—K2—O2	49.01 (5)	O3—P1—O1	111.58 (17)
O1ⁱⁱⁱ—K2—O2	78.18 (7)	O3—P1—O2	113.45 (16)
O1ⁱ—K2—O2	131.74 (9)	O1—P1—O2	109.85 (16)
O1ⁱⁱ—K2—O2	52.43 (7)	O3—P1—N1	110.5 (2)
O3ⁱⁱⁱ—K2—O2^{viii}	89.38 (7)	O1—P1—N1	105.82 (13)
O3ⁱ—K2—O2^{viii}	120.12 (8)	O2—P1—N1	105.20 (17)
O3ⁱⁱ—K2—O2^{viii}	155.11 (7)	O3—P1—K2^{xviii}	52.04 (12)
N1—K2—O2^{viii}	49.01 (5)	O1—P1—K2^{xviii}	60.00 (11)
O1ⁱⁱⁱ—K2—O2^{viii}	52.43 (7)	O2—P1—K2^{xviii}	124.79 (11)
O1ⁱ—K2—O2^{viii}	78.18 (7)	N1—P1—K2^{xviii}	130.01 (13)
O1ⁱⁱ—K2—O2^{viii}	131.74 (9)	O3—P1—K3^{xix}	72.98 (11)
O2—K2—O2^{viii}	81.64 (8)	O1—P1—K3^{xix}	98.65 (11)
O3ⁱⁱⁱ—K2—O2^{ix}	155.11 (7)	O2—P1—K3^{xix}	51.11 (11)
O3ⁱ—K2—O2^{ix}	89.38 (7)	N1—P1—K3^{xix}	151.24 (7)
O3ⁱⁱ—K2—O2^{ix}	120.12 (8)	K2^{xviii}—P1—K3^{xix}	75.63 (3)
N1—K2—O2^{ix}	49.01 (5)	O3—P1—K2	161.98 (12)
O1ⁱⁱⁱ—K2—O2^{ix}	131.74 (9)	O1—P1—K2	83.96 (11)
O1ⁱ—K2—O2^{ix}	52.43 (7)	O2—P1—K2	66.87 (11)
O1ⁱⁱ—K2—O2^{ix}	78.18 (7)	N1—P1—K2	54.41 (18)
O2—K2—O2^{ix}	81.64 (8)	K2^{xviii}—P1—K2	143.90 (4)
O2^{viii}—K2—O2^{ix}	81.64 (8)	K3^{xix}—P1—K2	114.96 (3)
O3ⁱⁱⁱ—K2—P1ⁱⁱⁱ	25.20 (6)	O3—P1—K3	43.33 (11)
O3ⁱ—K2—P1ⁱⁱⁱ	99.59 (7)	O1—P1—K3	113.68 (11)
O3ⁱⁱ—K2—P1ⁱⁱⁱ	86.94 (7)	O2—P1—K3	136.05 (12)
N1—K2—P1ⁱⁱⁱ	111.81 (3)	N1—P1—K3	68.57 (19)
O1ⁱⁱⁱ—K2—P1ⁱⁱⁱ	26.52 (6)	K2^{xviii}—P1—K3	74.83 (3)
O1ⁱ—K2—P1ⁱⁱⁱ	115.21 (6)	K3^{xix}—P1—K3	114.91 (3)
O1ⁱⁱ—K2—P1ⁱⁱⁱ	123.59 (6)	K2—P1—K3	122.98 (3)
O2—K2—P1ⁱⁱⁱ	98.86 (5)	O3—P1—K1^{xx}	119.94 (11)
O2^{viii}—K2—P1ⁱⁱⁱ	71.74 (5)	O1—P1—K1^{xx}	46.13 (11)
O2^{ix}—K2—P1ⁱⁱⁱ	152.93 (6)	O2—P1—K1^{xx}	65.25 (11)
O3ⁱⁱⁱ—K2—P1ⁱⁱ	99.59 (7)	N1—P1—K1^{xx}	128.30 (17)
O3ⁱ—K2—P1ⁱⁱ	86.94 (7)	K2^{xviii}—P1—K1^{xx}	78.82 (3)
O3ⁱⁱ—K2—P1ⁱⁱ	25.20 (6)	K3^{xix}—P1—K1^{xx}	61.97 (3)
N1—K2—P1ⁱⁱ	111.81 (3)	K2—P1—K1^{xx}	77.22 (3)
O1ⁱⁱⁱ—K2—P1ⁱⁱ	115.21 (6)	K3—P1—K1^{xx}	153.19 (4)
O1ⁱ—K2—P1ⁱⁱ	123.59 (6)	P1^{viii}—N1—P1^{ix}	118.53 (8)
O1ⁱⁱ—K2—P1ⁱⁱ	26.52 (6)	P1^{viii}—N1—P1	118.53 (8)
O2—K2—P1ⁱⁱ	71.74 (5)	P1^{ix}—N1—P1	118.54 (8)
O2^{viii}—K2—P1ⁱⁱ	152.93 (6)	P1^{viii}—N1—K2	97.01 (18)

O2^{ix}—K2—P1^{ii}	98.86 (5)	P1^{ix}—N1—K2	97.01 (18)
P1^{iii}—K2—P1^{ii}	107.04 (3)	P1—N1—K2	97.01 (18)
O3^{viii}—K3—O3^{ix}	83.76 (9)	P1^{viii}—N1—K3	82.99 (18)
O3^{viii}—K3—O3	83.76 (9)	P1^{ix}—N1—K3	82.99 (18)
O3^{ix}—K3—O3	83.76 (9)	P1—N1—K3	82.99 (18)
O3^{viii}—K3—O2^{x}	104.80 (8)	K2—N1—K3	180.0 (3)
O3^{ix}—K3—O2^{x}	162.69 (9)	P1—O1—Al1^{xxi}	144.58 (18)
O3—K3—O2^{x}	111.80 (8)	P1—O1—K1^{xx}	110.32 (14)
O3^{viii}—K3—O2^{xi}	111.80 (8)	Al1^{xxi}—O1—K1^{xx}	92.77 (12)
O3^{ix}—K3—O2^{xi}	104.80 (8)	P1—O1—K2^{xviii}	93.47 (13)
O3—K3—O2^{xi}	162.69 (9)	Al1^{xxi}—O1—K2^{xviii}	108.00 (11)
O2^{x}—K3—O2^{xi}	58.18 (9)	K1^{xx}—O1—K2^{xviii}	101.99 (8)
O3^{viii}—K3—O2^{xii}	162.69 (9)	P1—O2—Al1^{xix}	131.82 (18)
O3^{ix}—K3—O2^{xii}	111.80 (8)	P1—O2—K3^{xix}	103.68 (13)
O3—K3—O2^{xii}	104.80 (8)	Al1^{xix}—O2—K3^{xix}	90.31 (11)
O2^{x}—K3—O2^{xii}	58.18 (9)	P1—O2—K1^{xx}	89.45 (12)
O2^{xi}—K3—O2^{xii}	58.18 (9)	Al1^{xix}—O2—K1^{xx}	138.56 (12)
O3^{viii}—K3—N1	50.43 (6)	K3^{xix}—O2—K1^{xx}	73.60 (7)
O3^{ix}—K3—N1	50.43 (6)	P1—O2—K2	87.50 (12)
O3—K3—N1	50.43 (6)	Al1^{xix}—O2—K2	97.57 (11)
O2^{x}—K3—N1	145.85 (6)	K3^{xix}—O2—K2	156.49 (11)
O2^{xi}—K3—N1	145.85 (6)	K1^{xx}—O2—K2	86.08 (7)
O2^{xii}—K3—N1	145.85 (6)	P1—O3—K1^{xviii}	153.19 (17)
O3^{viii}—K3—O3^{x}	111.63 (2)	P1—O3—K3	114.19 (14)
O3^{ix}—K3—O3^{x}	143.68 (7)	K1^{xviii}—O3—K3	87.05 (9)
O3—K3—O3^{x}	66.44 (10)	P1—O3—K2^{xviii}	102.76 (15)
O2^{x}—K3—O3^{x}	47.20 (7)	K1^{xviii}—O3—K2^{xviii}	87.38 (8)
O2^{xi}—K3—O3^{x}	99.58 (8)	K3—O3—K2^{xviii}	102.41 (10)
O2^{xii}—K3—O3^{x}	60.45 (8)	P1—O3—K3^{xix}	82.03 (12)
N1—K3—O3^{x}	113.91 (5)	K1^{xviii}—O3—K3^{xix}	73.74 (6)
O3^{viii}—K3—O3^{xi}	66.44 (10)	K3—O3—K3^{xix}	158.41 (11)
O3^{ix}—K3—O3^{xi}	111.63 (2)	K2^{xviii}—O3—K3^{xix}	86.76 (7)
O3—K3—O3^{xi}	143.68 (7)		

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

Tripotassium gallium nitridotriphosphate (II)

Crystal data

$K_3Ga(NP_3O_9)$	$D_x = 3.157$ Mg m^{-3}
$M_r = 437.94$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cubic, $P2_13$	Cell parameters from 4964 reflections
Hall symbol: P 2ac 2ab 3	$\theta = 3.0$–28.0°
$a = 9.7313$ (9) Å	$\mu = 4.90$ mm^{-1}
$V = 921.5$ (3) Å^3	$T = 293$ K
$Z = 4$	Irregular tetrahedron, colorless
$F(000) = 848$	0.12 × 0.07 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur-3 diffractometer	753 independent reflections
Graphite monochromator	517 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.155$
Absorption correction: multi-scan (Blessing, 1995)	$\theta_{max} = 28.0°$, $\theta_{min} = 3.0°$
$T_{min} = 0.738$, $T_{max} = 0.804$	$h = -12 \rightarrow 9$
4964 measured reflections	$k = -12 \rightarrow 9$
	$l = -10 \rightarrow 12$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
Least-squares matrix: full	$\Delta\rho_{max} = 0.67$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.059$	$\Delta\rho_{min} = -0.60$ e Å^{-3}
$wR(F^2) = 0.102$	Extinction correction: SHELXL2018/3 (Sheldrick 2015)
$S = 1$	Extinction coefficient: 0.0038 (15)
753 reflections	Absolute structure: Flack x determined using
53 parameters	153 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al.,
0 restraints	2013)
$'w = 1/[\sigma^2(F_o^2) + (0.0188P)^2]$ where $P = (F_o^2 + 2F_c^2)/3'$	Absolute structure parameter: 0.03 (5)

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
K1	0.0456 (3)	0.0456 (3)	0.0456 (3)	0.0326 (13)
K2	0.2679 (3)	0.2679 (3)	0.2679 (3)	0.0329 (13)
K3	0.6410 (3)	0.6410 (3)	0.6410 (3)	0.0358 (14)
Ga1	0.83974 (15)	0.83974 (15)	0.83974 (15)	0.0235 (7)
P1	0.3255 (4)	0.5696 (3)	0.4719 (3)	0.0221 (8)
N1	0.4446 (10)	0.4446 (10)	0.4446 (10)	0.019 (4)
O1	0.2172 (8)	0.5088 (9)	0.5672 (8)	0.024 (2)
O2	0.2578 (8)	0.5989 (8)	0.3347 (9)	0.026 (2)
O3	0.3941 (8)	0.6899 (8)	0.5330 (8)	0.028 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0326 (13)	0.0326 (13)	0.0326 (13)	−0.0026 (14)	−0.0026 (14)	−0.0026 (14)
K2	0.0329 (13)	0.0329 (13)	0.0329 (13)	−0.0034 (14)	−0.0034 (14)	−0.0034 (14)
K3	0.0358 (14)	0.0358 (14)	0.0358 (14)	−0.0040 (16)	−0.0040 (16)	−0.0040 (16)
Ga1	0.0235 (7)	0.0235 (7)	0.0235 (7)	0.0007 (7)	0.0007 (7)	0.0007 (7)
P1	0.0243 (18)	0.0217 (18)	0.0204 (17)	−0.0025 (15)	−0.0006 (15)	−0.0001 (14)
N1	0.019 (4)	0.019 (4)	0.019 (4)	0.003 (5)	0.003 (5)	0.003 (5)
O1	0.022 (5)	0.029 (5)	0.021 (5)	−0.002 (4)	0.007 (4)	0.010 (4)

O2	0.022 (4)	0.031 (5)	0.026 (5)	0.005 (4)	−0.001 (4)	−0.003 (4)
O3	0.039 (6)	0.023 (5)	0.023 (5)	−0.005 (4)	−0.002 (4)	−0.006 (4)

Geometric parameters (Å, º)

K1—O3^i	2.642 (8)	K2—P1^i	3.409 (4)
K1—O3^{ii}	2.642 (8)	K3—O3	2.665 (9)
K1—O3^{iii}	2.642 (8)	K3—O3^{viii}	2.665 (9)
K1—O1^{iv}	2.807 (9)	K3—O3^{ix}	2.665 (9)
K1—O1^v	2.807 (9)	K3—O2^x	2.785 (8)
K1—O1^{vi}	2.807 (9)	K3—O2^{xi}	2.785 (8)
K1—O2^v	3.216 (8)	K3—O2^{xii}	2.785 (8)
K1—O2^{vi}	3.216 (8)	K3—N1	3.310 (18)
K1—O2^{iv}	3.216 (8)	K3—Ga1	3.350 (5)
K1—Ga1^{vii}	3.470 (6)	K3—O3^x	3.412 (8)
K1—P1^{iv}	3.624 (5)	K3—O3^{xi}	3.412 (8)
K1—P1^{vi}	3.624 (5)	K3—O3^{xii}	3.412 (8)
K2—O3^{ii}	2.808 (9)	K3—P1^{xii}	3.516 (4)
K2—O3^i	2.808 (9)	Ga1—O1^{xiii}	1.958 (8)
K2—O3^{iii}	2.808 (9)	Ga1—O1^{xiv}	1.958 (8)
K2—O1^i	2.925 (9)	Ga1—O1^{xv}	1.958 (8)
K2—O1^{ii}	2.925 (9)	Ga1—O2^{xi}	1.968 (9)
K2—O1^{iii}	2.925 (9)	Ga1—O2^x	1.968 (9)
K2—N1	2.977 (18)	Ga1—O2^{xii}	1.968 (9)
K2—O2^{viii}	3.287 (8)	P1—O3	1.473 (9)
K2—O2^{ix}	3.287 (8)	P1—O2	1.516 (9)
K2—O2	3.287 (8)	P1—O1	1.524 (9)
K2—P1^{ii}	3.409 (4)	P1—N1	1.701 (4)
O3^i—K1—O3^{ii}	80.8 (3)	O2^x—K3—O3^x	46.3 (2)
O3^i—K1—O3^{iii}	80.8 (3)	O2^{xi}—K3—O3^x	101.1 (2)
O3^{ii}—K1—O3^{iii}	80.8 (3)	O2^{xii}—K3—O3^x	60.3 (2)
O3^i—K1—O1^{iv}	111.5 (2)	N1—K3—O3^x	114.13 (16)
O3^{ii}—K1—O1^{iv}	165.6 (3)	Ga1—K3—O3^x	65.87 (16)
O3^{iii}—K1—O1^{iv}	108.0 (3)	O3—K3—O3^{xi}	143.91 (19)
O3^i—K1—O1^v	108.0 (3)	O3^{viii}—K3—O3^{xi}	111.65 (7)
O3^{ii}—K1—O1^v	111.5 (2)	O3^{ix}—K3—O3^{xi}	67.3 (3)
O3^{iii}—K1—O1^v	165.6 (3)	O2^x—K3—O3^{xi}	60.3 (2)
O1^{iv}—K1—O1^v	58.5 (3)	O2^{xi}—K3—O3^{xi}	46.3 (2)
O3^i—K1—O1^{vi}	165.6 (3)	O2^{xii}—K3—O3^{xi}	101.1 (2)
O3^{ii}—K1—O1^{vi}	108.0 (3)	N1—K3—O3^{xi}	114.13 (16)
O3^{iii}—K1—O1^{vi}	111.5 (2)	Ga1—K3—O3^{xi}	65.87 (16)
O1^{iv}—K1—O1^{vi}	58.5 (3)	O3^x—K3—O3^{xi}	104.44 (18)
O1^v—K1—O1^{vi}	58.5 (3)	O3—K3—O3^{xii}	111.65 (7)
O3^i—K1—O2^v	93.7 (2)	O3^{viii}—K3—O3^{xii}	67.3 (3)
O3^{ii}—K1—O2^v	64.5 (2)	O3^{ix}—K3—O3^{xii}	143.91 (19)
O3^{iii}—K1—O2^v	145.2 (3)	O2^x—K3—O3^{xii}	101.1 (2)
O1^{iv}—K1—O2^v	106.0 (3)	O2^{xi}—K3—O3^{xii}	60.3 (2)

O1^{v}—K1—O2^{v}	47.6 (2)	O2^{xii}—K3—O3^{xii}	46.3 (2)
O1^{vi}—K1—O2^{v}	80.4 (2)	N1—K3—O3^{xii}	114.13 (16)
O3^{i}—K1—O2^{vi}	145.2 (3)	Ga1—K3—O3^{xii}	65.87 (16)
O3^{ii}—K1—O2^{vi}	93.7 (2)	O3^{x}—K3—O3^{xii}	104.44 (18)
O3^{iii}—K1—O2^{vi}	64.5 (2)	O3^{xi}—K3—O3^{xii}	104.44 (18)
O1^{iv}—K1—O2^{vi}	80.4 (2)	O3—K3—P1^{xii}	101.2 (2)
O1^{v}—K1—O2^{vi}	106.0 (3)	O3^{viii}—K3—P1^{xii}	86.72 (18)
O1^{vi}—K1—O2^{vi}	47.6 (2)	O3^{ix}—K3—P1^{xii}	168.3 (2)
O2^{v}—K1—O2^{vi}	114.96 (13)	O2^{x}—K3—P1^{xii}	85.1 (2)
O3^{i}—K1—O2^{iv}	64.5 (2)	O2^{xi}—K3—P1^{xii}	66.1 (2)
O3^{ii}—K1—O2^{iv}	145.2 (3)	O2^{xii}—K3—P1^{xii}	24.51 (18)
O3^{iii}—K1—O2^{iv}	93.7 (2)	N1—K3—P1^{xii}	125.23 (9)
O1^{iv}—K1—O2^{iv}	47.6 (2)	Ga1—K3—P1^{xii}	54.77 (9)
O1^{v}—K1—O2^{iv}	80.4 (2)	O3^{x}—K3—P1^{xii}	79.98 (17)
O1^{vi}—K1—O2^{iv}	106.0 (3)	O3^{xi}—K3—P1^{xii}	112.13 (19)
O2^{v}—K1—O2^{iv}	114.96 (13)	O3^{xii}—K3—P1^{xii}	24.49 (15)
O2^{vi}—K1—O2^{iv}	114.96 (13)	O1^{xiii}—Ga1—O1^{xiv}	88.9 (4)
O3^{i}—K1—Ga1^{vii}	131.6 (2)	O1^{xiii}—Ga1—O1^{xv}	88.9 (4)
O3^{ii}—K1—Ga1^{vii}	131.6 (2)	O1^{xiv}—Ga1—O1^{xv}	88.9 (4)
O3^{iii}—K1—Ga1^{vii}	131.6 (2)	O1^{xiii}—Ga1—O2^{xi}	91.7 (3)
O1^{iv}—K1—Ga1^{vii}	34.33 (17)	O1^{xiv}—Ga1—O2^{xi}	176.3 (3)
O1^{v}—K1—Ga1^{vii}	34.33 (17)	O1^{xv}—Ga1—O2^{xi}	87.5 (3)
O1^{vi}—K1—Ga1^{vii}	34.33 (17)	O1^{xiii}—Ga1—O2^{x}	176.3 (3)
O2^{v}—K1—Ga1^{vii}	76.82 (17)	O1^{xiv}—Ga1—O2^{x}	87.5 (3)
O2^{vi}—K1—Ga1^{vii}	76.82 (17)	O1^{xv}—Ga1—O2^{x}	91.7 (4)
O2^{iv}—K1—Ga1^{vii}	76.82 (17)	O2^{xi}—Ga1—O2^{x}	92.0 (3)
O3^{i}—K1—P1^{iv}	89.16 (19)	O1^{xiii}—Ga1—O2^{xii}	87.5 (3)
O3^{ii}—K1—P1^{iv}	169.8 (2)	O1^{xiv}—Ga1—O2^{xii}	91.7 (3)
O3^{iii}—K1—P1^{iv}	98.99 (18)	O1^{xv}—Ga1—O2^{xii}	176.3 (3)
O1^{iv}—K1—P1^{iv}	23.26 (16)	O2^{xi}—Ga1—O2^{xii}	92.0 (3)
O1^{v}—K1—P1^{iv}	70.29 (19)	O2^{x}—Ga1—O2^{xii}	92.0 (3)
O1^{vi}—K1—P1^{iv}	81.7 (2)	O1^{xiii}—Ga1—K3	126.1 (2)
O2^{v}—K1—P1^{iv}	115.3 (2)	O1^{xiv}—Ga1—K3	126.1 (2)
O2^{vi}—K1—P1^{iv}	95.39 (17)	O1^{xv}—Ga1—K3	126.1 (2)
O2^{iv}—K1—P1^{iv}	24.71 (16)	O2^{xi}—Ga1—K3	56.2 (2)
Ga1^{vii}—K1—P1^{iv}	55.56 (9)	O2^{x}—Ga1—K3	56.2 (2)
O3^{i}—K1—P1^{vi}	169.8 (2)	O2^{xii}—Ga1—K3	56.2 (2)
O3^{ii}—K1—P1^{vi}	98.99 (18)	O1^{xiii}—Ga1—K1^{xvi}	53.9 (2)
O3^{iii}—K1—P1^{vi}	89.16 (19)	O1^{xiv}—Ga1—K1^{xvi}	53.9 (2)
O1^{iv}—K1—P1^{vi}	70.29 (19)	O1^{xv}—Ga1—K1^{xvi}	53.9 (2)
O1^{v}—K1—P1^{vi}	81.7 (2)	O2^{xi}—Ga1—K1^{xvi}	123.8 (2)
O1^{vi}—K1—P1^{vi}	23.26 (17)	O2^{x}—Ga1—K1^{xvi}	123.8 (2)
O2^{v}—K1—P1^{vi}	95.39 (17)	O2^{xii}—Ga1—K1^{xvi}	123.8 (2)
O2^{vi}—K1—P1^{vi}	24.71 (16)	K3—Ga1—K1^{xvi}	180.00 (7)
O2^{iv}—K1—P1^{vi}	115.3 (2)	O1^{xiii}—Ga1—K2^{xiv}	124.6 (3)
Ga1^{vii}—K1—P1^{vi}	55.56 (9)	O1^{xiv}—Ga1—K2^{xiv}	68.5 (3)
P1^{iv}—K1—P1^{vi}	91.16 (13)	O1^{xv}—Ga1—K2^{xiv}	43.4 (3)
O3^{ii}—K2—O3^{i}	75.1 (3)	O2^{xi}—Ga1—K2^{xiv}	108.3 (2)

O3ⁱⁱ—K2—O3ⁱⁱⁱ	75.1 (3)	O2ˣ—Ga1—K2ˣⁱᵛ	54.3 (2)
O3ⁱ—K2—O3ⁱⁱⁱ	75.1 (3)	O2ˣⁱⁱ—Ga1—K2ˣⁱᵛ	140.0 (2)
O3ⁱⁱ—K2—O1ⁱ	123.7 (3)	K3—Ga1—K2ˣⁱᵛ	107.30 (4)
O3ⁱ—K2—O1ⁱ	51.4 (2)	K1ˣᵛⁱ—Ga1—K2ˣⁱᵛ	72.70 (4)
O3ⁱⁱⁱ—K2—O1ⁱ	102.9 (2)	O1ˣⁱⁱⁱ—Ga1—K2ˣᵛⁱⁱ	43.4 (3)
O3ⁱⁱ—K2—O1ⁱⁱ	51.4 (2)	O1ˣⁱᵛ—Ga1—K2ˣᵛⁱⁱ	124.6 (3)
O3ⁱ—K2—O1ⁱⁱ	102.9 (2)	O1ˣᵛ—Ga1—K2ˣᵛⁱⁱ	68.5 (3)
O3ⁱⁱⁱ—K2—O1ⁱⁱ	123.7 (3)	O2ˣⁱ—Ga1—K2ˣᵛⁱⁱ	54.3 (2)
O1ⁱ—K2—O1ⁱⁱ	119.49 (5)	O2ˣ—Ga1—K2ˣᵛⁱⁱ	140.0 (2)
O3ⁱⁱ—K2—O1ⁱⁱⁱ	102.9 (2)	O2ˣⁱⁱ—Ga1—K2ˣᵛⁱⁱ	108.3 (2)
O3ⁱ—K2—O1ⁱⁱⁱ	123.7 (3)	K3—Ga1—K2ˣᵛⁱⁱ	107.30 (4)
O3ⁱⁱⁱ—K2—O1ⁱⁱⁱ	51.4 (2)	K1ˣᵛⁱ—Ga1—K2ˣᵛⁱⁱ	72.70 (4)
O1ⁱ—K2—O1ⁱⁱⁱ	119.49 (5)	K2ˣⁱᵛ—Ga1—K2ˣᵛⁱⁱ	111.55 (3)
O1ⁱⁱ—K2—O1ⁱⁱⁱ	119.49 (5)	O1ˣⁱⁱⁱ—Ga1—K2ˣⁱⁱ	68.5 (3)
O3ⁱⁱ—K2—N1	135.25 (18)	O1ˣⁱᵛ—Ga1—K2ˣⁱⁱ	43.4 (3)
O3ⁱ—K2—N1	135.25 (18)	O1ˣᵛ—Ga1—K2ˣⁱⁱ	124.6 (3)
O3ⁱⁱⁱ—K2—N1	135.25 (18)	O2ˣⁱ—Ga1—K2ˣⁱⁱ	140.0 (2)
O1ⁱ—K2—N1	85.89 (18)	O2ˣ—Ga1—K2ˣⁱⁱ	108.3 (2)
O1ⁱⁱ—K2—N1	85.89 (18)	O2ˣⁱⁱ—Ga1—K2ˣⁱⁱ	54.3 (2)
O1ⁱⁱⁱ—K2—N1	85.89 (18)	K3—Ga1—K2ˣⁱⁱ	107.30 (4)
O3ⁱⁱ—K2—O2ᵛⁱⁱⁱ	90.1 (2)	K1ˣᵛⁱ—Ga1—K2ˣⁱⁱ	72.70 (4)
O3ⁱ—K2—O2ᵛⁱⁱⁱ	156.0 (2)	K2ˣⁱᵛ—Ga1—K2ˣⁱⁱ	111.55 (3)
O3ⁱⁱⁱ—K2—O2ᵛⁱⁱⁱ	119.8 (2)	K2ˣᵛⁱⁱ—Ga1—K2ˣⁱⁱ	111.55 (3)
O1ⁱ—K2—O2ᵛⁱⁱⁱ	131.8 (3)	O3—P1—O2	113.8 (5)
O1ⁱⁱ—K2—O2ᵛⁱⁱⁱ	53.5 (2)	O3—P1—O1	112.1 (5)
O1ⁱⁱⁱ—K2—O2ᵛⁱⁱⁱ	77.6 (2)	O2—P1—O1	107.9 (5)
N1—K2—O2ᵛⁱⁱⁱ	48.52 (15)	O3—P1—N1	108.9 (7)
O3ⁱⁱ—K2—O2ⁱˣ	119.8 (2)	O2—P1—N1	107.0 (5)
O3ⁱ—K2—O2ⁱˣ	90.1 (2)	O1—P1—N1	106.8 (4)
O3ⁱⁱⁱ—K2—O2ⁱˣ	156.0 (2)	O3—P1—K2ˣᵛⁱⁱⁱ	53.9 (4)
O1ⁱ—K2—O2ⁱˣ	53.5 (2)	O2—P1—K2ˣᵛⁱⁱⁱ	122.8 (3)
O1ⁱⁱ—K2—O2ⁱˣ	77.6 (2)	O1—P1—K2ˣᵛⁱⁱⁱ	58.8 (4)
O1ⁱⁱⁱ—K2—O2ⁱˣ	131.8 (3)	N1—P1—K2ˣᵛⁱⁱⁱ	130.2 (4)
N1—K2—O2ⁱˣ	48.52 (15)	O3—P1—K3ˣⁱˣ	73.8 (3)
O2ᵛⁱⁱⁱ—K2—O2ⁱˣ	80.9 (2)	O2—P1—K3ˣⁱˣ	49.6 (3)
O3ⁱⁱ—K2—O2	156.0 (2)	O1—P1—K3ˣⁱˣ	98.5 (3)
O3ⁱ—K2—O2	119.8 (2)	N1—P1—K3ˣⁱˣ	150.7 (2)
O3ⁱⁱⁱ—K2—O2	90.1 (2)	K2ˣᵛⁱⁱⁱ—P1—K3ˣⁱˣ	75.90 (10)
O1ⁱ—K2—O2	77.6 (2)	O3—P1—K3	42.8 (3)
O1ⁱⁱ—K2—O2	131.8 (3)	O2—P1—K3	138.4 (3)
O1ⁱⁱⁱ—K2—O2	53.5 (2)	O1—P1—K3	113.1 (3)
N1—K2—O2	48.52 (15)	N1—P1—K3	67.9 (6)
O2ᵛⁱⁱⁱ—K2—O2	80.9 (2)	K2ˣᵛⁱⁱⁱ—P1—K3	75.44 (8)
O2ⁱˣ—K2—O2	80.9 (2)	K3ˣⁱˣ—P1—K3	115.45 (9)
O3ⁱⁱ—K2—P1ⁱⁱ	25.10 (18)	O3—P1—K2	160.8 (4)
O3ⁱ—K2—P1ⁱⁱ	86.7 (2)	O2—P1—K2	66.3 (3)
O3ⁱⁱⁱ—K2—P1ⁱⁱ	99.6 (2)	O1—P1—K2	84.9 (4)
O1ⁱ—K2—P1ⁱⁱ	123.33 (17)	N1—P1—K2	55.6 (6)

O1ⁱⁱ—K2—P1ⁱⁱ	26.44 (17)	K2ˣᵛⁱⁱⁱ—P1—K2	143.62 (12)
O1ⁱⁱⁱ—K2—P1ⁱⁱ	115.25 (16)	K3ˣⁱˣ—P1—K2	113.74 (10)
N1—K2—P1ⁱⁱ	111.98 (9)	K3—P1—K2	123.46 (11)
O2ᵛⁱⁱⁱ—K2—P1ⁱⁱ	72.82 (16)	O3—P1—K1ˣˣ	121.5 (3)
O2ⁱˣ—K2—P1ⁱⁱ	98.30 (15)	O2—P1—K1ˣˣ	62.4 (3)
O2—K2—P1ⁱⁱ	153.45 (17)	O1—P1—K1ˣˣ	46.7 (3)
O3ⁱⁱ—K2—P1ⁱ	99.6 (2)	N1—P1—K1ˣˣ	128.7 (6)
O3ⁱ—K2—P1ⁱ	25.10 (18)	K2ˣᵛⁱⁱⁱ—P1—K1ˣˣ	78.68 (8)
O3ⁱⁱⁱ—K2—P1ⁱ	86.7 (2)	K3ˣⁱˣ—P1—K1ˣˣ	61.82 (9)
O1ⁱ—K2—P1ⁱ	26.44 (17)	K3—P1—K1ˣˣ	153.65 (11)
O1ⁱⁱ—K2—P1ⁱ	115.25 (16)	K2—P1—K1ˣˣ	76.43 (8)
O1ⁱⁱⁱ—K2—P1ⁱ	123.33 (17)	P1—N1—P1ᵛⁱⁱⁱ	118.8 (2)
N1—K2—P1ⁱ	111.98 (9)	P1—N1—P1ⁱˣ	118.8 (2)
O2ᵛⁱⁱⁱ—K2—P1ⁱ	153.45 (17)	P1ᵛⁱⁱⁱ—N1—P1ⁱˣ	118.8 (2)
O2ⁱˣ—K2—P1ⁱ	72.82 (16)	P1—N1—K2	96.3 (6)
O2—K2—P1ⁱ	98.30 (15)	P1ᵛⁱⁱⁱ—N1—K2	96.3 (6)
P1ⁱⁱ—K2—P1ⁱ	106.85 (10)	P1ⁱˣ—N1—K2	96.3 (6)
O3—K3—O3ᵛⁱⁱⁱ	82.9 (3)	P1—N1—K3	83.7 (6)
O3—K3—O3ⁱˣ	82.9 (3)	P1ᵛⁱⁱⁱ—N1—K3	83.7 (6)
O3ᵛⁱⁱⁱ—K3—O3ⁱˣ	82.9 (3)	P1ⁱˣ—N1—K3	83.7 (6)
O3—K3—O2ˣ	111.2 (2)	K2—N1—K3	180.0 (9)
O3ᵛⁱⁱⁱ—K3—O2ˣ	164.8 (3)	P1—O1—Ga1ˣˣⁱ	143.5 (6)
O3ⁱˣ—K3—O2ˣ	103.8 (2)	P1—O1—K1ˣˣ	110.0 (4)
O3—K3—O2ˣⁱ	164.8 (3)	Ga1ˣˣⁱ—O1—K1ˣˣ	91.7 (3)
O3ᵛⁱⁱⁱ—K3—O2ˣⁱ	103.8 (2)	P1—O1—K2ˣᵛⁱⁱⁱ	94.8 (4)
O3ⁱˣ—K3—O2ˣⁱ	111.2 (2)	Ga1ˣˣⁱ—O1—K2ˣᵛⁱⁱⁱ	109.2 (3)
O2ˣ—K3—O2ˣⁱ	61.1 (3)	K1ˣˣ—O1—K2ˣᵛⁱⁱⁱ	102.2 (3)
O3—K3—O2ˣⁱⁱ	103.8 (2)	P1—O2—Ga1ˣⁱˣ	129.8 (5)
O3ᵛⁱⁱⁱ—K3—O2ˣⁱⁱ	111.2 (2)	P1—O2—K3ˣⁱˣ	105.8 (4)
O3ⁱˣ—K3—O2ˣⁱⁱ	164.8 (3)	Ga1ˣⁱˣ—O2—K3ˣⁱˣ	87.9 (3)
O2ˣ—K3—O2ˣⁱⁱ	61.1 (3)	P1—O2—K1ˣˣ	92.9 (4)
O2ˣⁱ—K3—O2ˣⁱⁱ	61.1 (3)	Ga1ˣⁱˣ—O2—K1ˣˣ	137.2 (4)
O3—K3—N1	49.85 (19)	K3ˣⁱˣ—O2—K1ˣˣ	74.99 (18)
O3ᵛⁱⁱⁱ—K3—N1	49.85 (19)	P1—O2—K2	88.7 (3)
O3ⁱˣ—K3—N1	49.85 (19)	Ga1ˣⁱˣ—O2—K2	96.6 (3)
O2ˣ—K3—N1	144.05 (18)	K3ˣⁱˣ—O2—K2	156.9 (3)
O2ˣⁱ—K3—N1	144.05 (18)	K1ˣˣ—O2—K2	86.6 (2)
O2ˣⁱⁱ—K3—N1	144.05 (18)	P1—O3—K1ˣᵛⁱⁱⁱ	153.3 (5)
O3—K3—Ga1	130.15 (19)	P1—O3—K3	115.2 (4)
O3ᵛⁱⁱⁱ—K3—Ga1	130.15 (19)	K1ˣᵛⁱⁱⁱ—O3—K3	87.5 (3)
O3ⁱˣ—K3—Ga1	130.15 (19)	P1—O3—K2ˣᵛⁱⁱⁱ	101.0 (4)
O2ˣ—K3—Ga1	35.95 (18)	K1ˣᵛⁱⁱⁱ—O3—K2ˣᵛⁱⁱⁱ	86.8 (3)
O2ˣⁱ—K3—Ga1	35.95 (18)	K3—O3—K2ˣᵛⁱⁱⁱ	102.2 (3)
O2ˣⁱⁱ—K3—Ga1	35.95 (18)	P1—O3—K3ˣⁱˣ	81.7 (3)
N1—K3—Ga1	180.0 (3)	K1ˣᵛⁱⁱⁱ—O3—K3ˣⁱˣ	73.36 (19)
O3—K3—O3ˣ	67.3 (3)	K3—O3—K3ˣⁱˣ	158.9 (3)

O3^{viii}—K3—O3^{x}	143.91 (19)	K2^{xviii}—O3—K3^{xix}	85.9 (2)
O3^{ix}—K3—O3^{x}	111.65 (7)		

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