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Dipotassium trisodium triphosphate, K₂Na₃P₃O₁₀

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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{P-O}) = 0.001$ Å; R factor = 0.032; wR factor = 0.080; data-to-parameter ratio = 33.3.

The structure of the title compound, K₂Na₃P₃O₁₀, is characterized by open chains of three PO₄ tetrahedra linked by single oxygen bridges. The P₃O₁₀ groups have crystallographic twofold symmetry, with the central P atom being located on the twofold rotation axis. One of the sodium ions lies on a centre of inversion, whereas all the remaining atoms are in general positions. The structure is isotopic with that of the high-temperature form of Na₅P₃O₁₀ phase I.

Related literature

For compounds with related structures, see: Cruickshank (1964); Davies & Corbridge (1958); Dyroff (1965), Wiench *et al.* (1982); Dymon & King (1951); Corbridge (1960).

Experimental

Crystal data

K₂Na₃P₃O₁₀
 $M_r = 400.08$
 Monoclinic, $C2/c$
 $a = 9.8866$ (4) Å

$b = 5.6332$ (2) Å
 $c = 18.6577$ (8) Å
 $\beta = 96.199$ (3)°
 $V = 1033.03$ (7) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.55$ mm⁻¹

$T = 296$ K
 $0.19 \times 0.14 \times 0.10$ mm

Data collection

Bruker APEXII CCD detector
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1999)
 $T_{\min} = 0.511$, $T_{\max} = 0.638$

12463 measured reflections
 2830 independent reflections
 2136 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.080$
 $S = 1.08$
 2830 reflections

85 parameters
 $\Delta\rho_{\text{max}} = 0.63$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.97$ e Å⁻³

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and DIAMOND (Brandenburg, 2006); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5872).

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supporting information

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Dipotassium trisodium triphosphate, $K_2Na_3P_3O_{10}$

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S1. Comment

The present triphosphate was obtained by chance, during the preparation of a mixed pyrophosphate. A bibliographic study of alkali triphosphates like $M_3P_3O_{10}$ shows that there are few known structures. Thus, in the case of sodium triphosphate, the crystal structures of two anhydrous forms noted Phase I and II were determined by Cruickshank (1964) and Davies & Corbridge (1958) and that of the hexahydrate was performed by Dyroff (1965) and re-examined by Wiench *et al.* (1982).

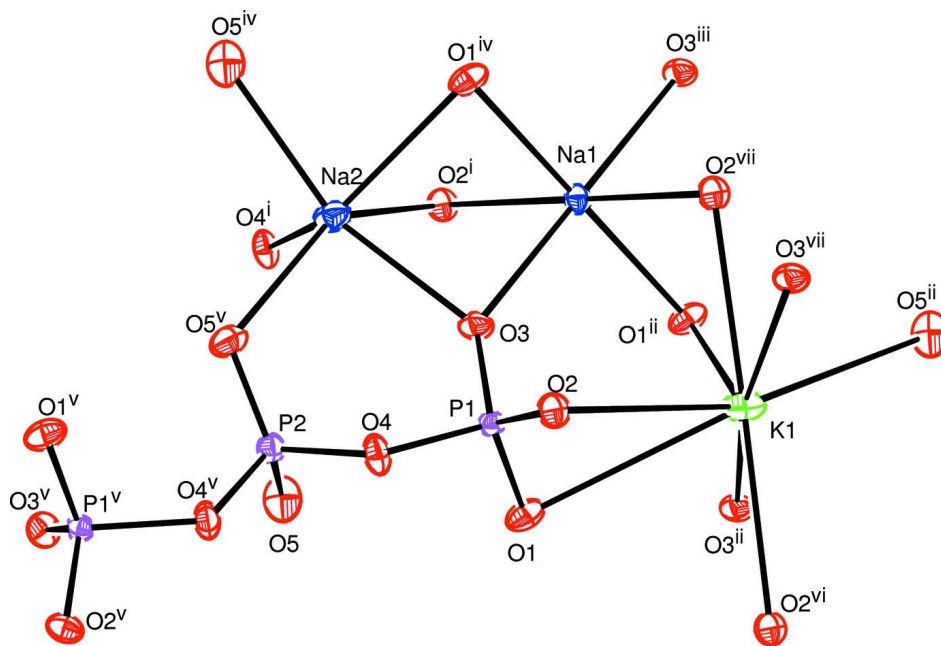
The structure of dipotassium trisodium triphosphate consists of open chains of three PO_4 tetrahedra linked by single oxygen bridges. The values of P–P (2.883 Å) distances and P—O—P (125.25°) angles are within the limits generally observed in condensed phosphate crystal chemistry. The internal symmetry of the P_3O_{10} groups has a twofold symmetry, with the central phosphorus P2 atom being located on a binary axis. Moreover, the Na2 sodium ion lies on the symmetry center whereas all the remaining atoms are in general positions of the $C2/c$ space group. The Na2 sodium atom located at Wyckoff position $4c$ (1/4, 3/4, 1/2) could be surrounded by a roughly octahedral arrangement of six oxygen atoms and the other sodium and potassium (Na1, K1) atoms are coordinated to six and eight oxygen atoms respectively. The Na_2O_6 octahedra, Na_1O_6 and K_1O_8 polyhedra are connected through the apices to triphosphate groups and form a three-dimensional host lattice (Fig.1). The resulting 3-D framework presents intersecting tunnels running along the [010] and [110] directions, where the six-coordinated Na_1^+ cations are located (Fig.2). The structure of this compound is isotype to that of the high-temperature form of $Na_5P_3O_{10}$ phase I (Dymon and King, 1951 and Corbridge, 1960).

S2. Experimental

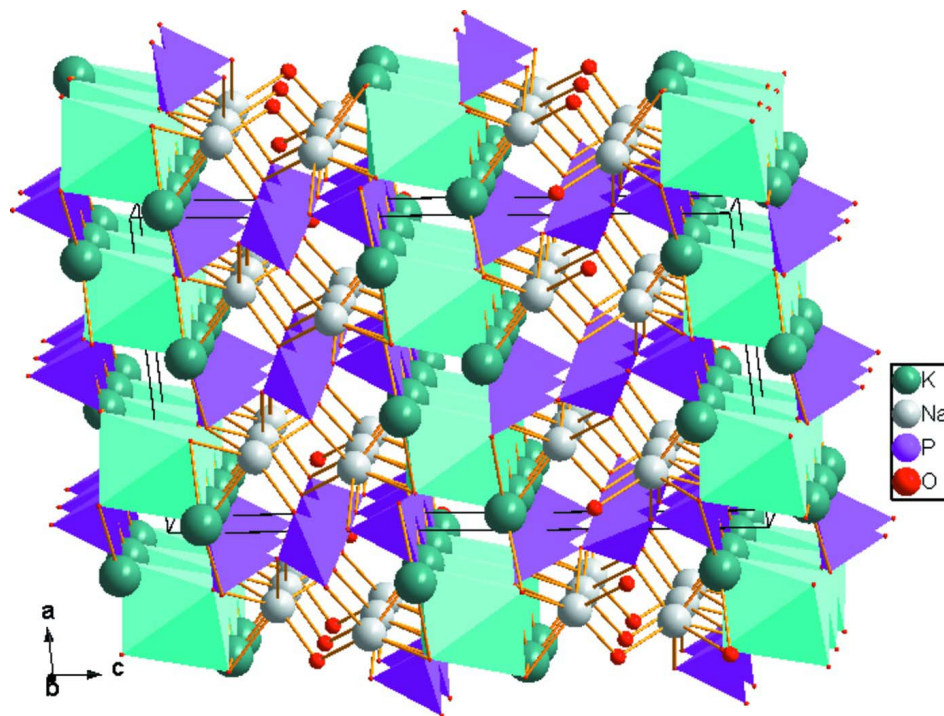
The present triphosphate is obtained by chance, during the preparation of a mixture of pyrophosphate. Indeed, the powder phase $NaKNiP_2O_7$ synthesized by wet process is introduced into a platinum crucible, and then gradually heated to a temperature above its melting point (1173 K) for 2 h, followed by slow cooling of the order of 6 K per hour up to 673 K. Then the furnace is shut down and the cooling is continued until room temperature. Small colourless single crystals of $K_2Na_3P_3O_{10}$ were isolated from the mixtures of phases.

S3. Refinement

The highest peak and the deepest hole in the final Fourier map are at 0.63 Å and 0.58 Å, respectively, from K1.


Figure 1

Plot of $\text{K}_2\text{Na}_3\text{P}_3\text{O}_{10}$ crystal structure showing polyhedra linkage. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) $-x + 1/2, -y + 1/2, -z + 1$; (ii) $-x, -y, -z + 1$; (iii) $-x, -y + 1, -z + 1$; (iv) $x, y + 1, z$; (v) $-x, y, -z + 1/2$; (vi) $x - 1/2, y + 1/2, z$; (vii) $-x + 1/2, y + 1/2, -z + 1/2$; (viii) $x + 1/2, y - 1/2, z$; (ix) $x, y - 1, z$; (x) $-x + 1/2, -y - 1/2, -z + 1$.

**Figure 2**

Projection view of the $\text{K}_2\text{Na}_3\text{P}_3\text{O}_{10}$ framework structure showing tunnel running along b direction where the Na1 atoms are located.

Dipotassium trisodium triphosphate

Crystal data

$\text{K}_2\text{Na}_3\text{P}_3\text{O}_{10}$

$M_r = 400.08$

Monoclinic, $C2/c$

Hall symbol: $-c\ 2yc$

$a = 9.8866\ (4)\ \text{\AA}$

$b = 5.6332\ (2)\ \text{\AA}$

$c = 18.6577\ (8)\ \text{\AA}$

$\beta = 96.199\ (3)^\circ$

$V = 1033.03\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 784$

$D_x = 2.572\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2830 reflections

$\theta = 4.2\text{--}38.3^\circ$

$\mu = 1.55\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colourless

$0.19 \times 0.14 \times 0.10\ \text{mm}$

Data collection

Bruker APEXII CCD detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1999)

$T_{\min} = 0.511$, $T_{\max} = 0.638$

12463 measured reflections

2830 independent reflections

2136 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 38.3^\circ$, $\theta_{\min} = 4.2^\circ$

$h = -17 \rightarrow 17$

$k = -8 \rightarrow 9$

$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.080$
 $S = 1.08$
 2830 reflections
 85 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 $w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.7541P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.63 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.97 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0011 (3)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	0.09445 (3)	0.27218 (6)	0.57736 (2)	0.02014 (8)
P1	0.06781 (3)	0.22944 (6)	0.398705 (18)	0.00976 (7)
P2	0.0000	0.09573 (9)	0.2500	0.01288 (10)
Na1	0.21820 (7)	-0.30829 (11)	0.32759 (3)	0.01917 (13)
Na2	0.2500	-0.2500	0.5000	0.01344 (15)
O1	-0.07276 (11)	0.22433 (19)	0.42318 (6)	0.0184 (2)
O2	0.15106 (10)	0.44080 (18)	0.42676 (5)	0.01569 (19)
O3	0.14234 (10)	-0.00428 (18)	0.40638 (6)	0.01571 (19)
O4	0.04508 (11)	0.28577 (18)	0.31142 (5)	0.01663 (19)
O5	-0.12023 (12)	-0.0419 (2)	0.26927 (6)	0.0231 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.01413 (13)	0.01460 (13)	0.03200 (18)	0.00043 (10)	0.00389 (11)	0.00128 (11)
P1	0.00897 (13)	0.00981 (14)	0.01046 (14)	0.00010 (10)	0.00091 (10)	-0.00060 (10)
P2	0.0142 (2)	0.0135 (2)	0.0108 (2)	0.000	0.00129 (15)	0.000
Na1	0.0199 (3)	0.0173 (3)	0.0212 (3)	0.0060 (2)	0.0062 (2)	0.0047 (2)
Na2	0.0132 (3)	0.0131 (4)	0.0139 (3)	-0.0003 (3)	0.0008 (3)	-0.0006 (3)
O1	0.0136 (4)	0.0187 (5)	0.0244 (5)	-0.0006 (4)	0.0090 (4)	-0.0020 (4)
O2	0.0151 (4)	0.0140 (4)	0.0174 (4)	-0.0028 (3)	-0.0007 (3)	-0.0041 (3)
O3	0.0162 (4)	0.0126 (4)	0.0180 (4)	0.0044 (3)	0.0003 (3)	0.0015 (3)
O4	0.0239 (5)	0.0159 (4)	0.0095 (4)	-0.0021 (4)	-0.0010 (3)	0.0012 (3)
O5	0.0238 (5)	0.0246 (5)	0.0216 (5)	-0.0107 (4)	0.0056 (4)	0.0005 (4)

Geometric parameters (Å, °)

K1—O2 ⁱ	2.7960 (11)	P2—O4	1.5961 (11)
K1—O1 ⁱⁱ	2.8051 (11)	P2—O4 ^{iv}	1.5961 (11)
K1—O3 ⁱⁱ	2.8291 (11)	Na1—O5 ^v	2.4172 (14)
K1—O1 ⁱⁱⁱ	2.8443 (11)	Na1—O3	2.4278 (12)
K1—O3 ⁱ	2.8987 (11)	Na1—O5 ^{iv}	2.4655 (13)
K1—O2 ⁱⁱⁱ	2.9106 (11)	Na1—O2 ^{vi}	2.4753 (12)
K1—O2	3.0740 (11)	Na1—O1 ^v	2.5864 (13)
K1—O5 ⁱⁱ	3.1272 (12)	Na1—O4 ^{vi}	2.8527 (13)
P1—O3	1.5080 (10)	Na2—O2 ^{vi}	2.3599 (10)
P1—O2	1.5086 (10)	Na2—O2 ⁱ	2.3599 (10)
P1—O1	1.5092 (11)	Na2—O1 ⁱⁱ	2.3850 (10)
P1—O4	1.6506 (10)	Na2—O1 ^v	2.3850 (10)
P2—O5	1.4951 (11)	Na2—O3	2.3871 (10)
P2—O5 ^{iv}	1.4951 (11)	Na2—O3 ^{vii}	2.3871 (10)
O2 ⁱ —K1—O1 ⁱⁱ	68.95 (3)	O3—P1—O4	105.94 (6)
O2 ⁱ —K1—O3 ⁱⁱ	122.11 (3)	O2—P1—O4	101.64 (6)
O1 ⁱⁱ —K1—O3 ⁱⁱ	53.46 (3)	O1—P1—O4	105.69 (6)
O2 ⁱ —K1—O1 ⁱⁱⁱ	119.70 (3)	O5—P2—O5 ^{iv}	117.54 (10)
O1 ⁱⁱ —K1—O1 ⁱⁱⁱ	171.32 (4)	O5—P2—O4	110.04 (6)
O3 ⁱⁱ —K1—O1 ⁱⁱⁱ	117.99 (3)	O5 ^{iv} —P2—O4	110.66 (6)
O2 ⁱ —K1—O3 ⁱ	52.85 (3)	O5—P2—O4 ^{iv}	110.66 (6)
O1 ⁱⁱ —K1—O3 ⁱ	121.17 (3)	O5 ^{iv} —P2—O4 ^{iv}	110.04 (6)
O3 ⁱⁱ —K1—O3 ⁱ	166.73 (4)	O4—P2—O4 ^{iv}	95.75 (8)
O1 ⁱⁱⁱ —K1—O3 ⁱ	67.49 (3)	O5 ^v —Na1—O3	156.77 (5)
O2 ⁱ —K1—O2 ⁱⁱⁱ	171.11 (4)	O5 ^v —Na1—O5 ^{iv}	103.17 (3)
O1 ⁱⁱ —K1—O2 ⁱⁱⁱ	119.38 (3)	O3—Na1—O5 ^{iv}	83.79 (4)
O3 ⁱⁱ —K1—O2 ⁱⁱⁱ	66.53 (3)	O5 ^v —Na1—O2 ^{vi}	105.62 (4)
O1 ⁱⁱⁱ —K1—O2 ⁱⁱⁱ	51.95 (3)	O3—Na1—O2 ^{vi}	79.92 (4)
O3 ⁱ —K1—O2 ⁱⁱⁱ	119.27 (3)	O5 ^{iv} —Na1—O2 ^{vi}	141.23 (5)
O2 ⁱ —K1—O2	81.62 (3)	O5 ^v —Na1—O1 ^v	80.29 (4)
O1 ⁱⁱ —K1—O2	109.04 (3)	O3—Na1—O1 ^v	78.96 (4)
O3 ⁱⁱ —K1—O2	119.82 (3)	O5 ^{iv} —Na1—O1 ^v	133.13 (5)
O1 ⁱⁱⁱ —K1—O2	73.13 (3)	O2 ^{vi} —Na1—O1 ^v	77.52 (4)
O3 ⁱ —K1—O2	72.92 (3)	O5 ^v —Na1—O4 ^{vi}	86.22 (4)
O2 ⁱⁱⁱ —K1—O2	92.17 (3)	O3—Na1—O4 ^{vi}	114.11 (4)
O2 ⁱ —K1—O5 ⁱⁱ	82.10 (3)	O5 ^{iv} —Na1—O4 ^{vi}	103.10 (4)
O1 ⁱⁱ —K1—O5 ⁱⁱ	65.69 (3)	O2 ^{vi} —Na1—O4 ^{vi}	54.20 (3)
O3 ⁱⁱ —K1—O5 ⁱⁱ	70.56 (3)	O1 ^v —Na1—O4 ^{vi}	123.75 (4)
O1 ⁱⁱⁱ —K1—O5 ⁱⁱ	114.55 (3)	O2 ^{vi} —Na2—O2 ⁱ	180.0
O3 ⁱ —K1—O5 ⁱⁱ	96.19 (3)	O2 ^{vi} —Na2—O1 ⁱⁱ	96.16 (4)
O2 ⁱⁱⁱ —K1—O5 ⁱⁱ	103.88 (3)	O2 ⁱ —Na2—O1 ⁱⁱ	83.85 (4)
O2—K1—O5 ⁱⁱ	163.68 (3)	O2 ^{vi} —Na2—O1 ^v	83.85 (4)
O2 ⁱ —K1—O1	108.73 (3)	O2 ⁱ —Na2—O1 ^v	96.15 (4)
O1 ⁱⁱ —K1—O1	83.10 (3)	O1 ⁱⁱ —Na2—O1 ^v	180.000 (1)
O3 ⁱⁱ —K1—O1	72.27 (3)	O2 ^{vi} —Na2—O3	83.11 (3)

O1 ⁱⁱⁱ —K1—O1	92.86 (3)	O2 ⁱ —Na2—O3	96.89 (3)
O3 ⁱ —K1—O1	120.50 (3)	O1 ⁱⁱ —Na2—O3	96.09 (4)
O2 ⁱⁱⁱ —K1—O1	70.83 (3)	O1 ^v —Na2—O3	83.91 (4)
O2—K1—O1	47.59 (3)	O2 ^{vi} —Na2—O3 ^{vii}	96.89 (3)
O5 ⁱⁱ —K1—O1	141.07 (3)	O2 ⁱ —Na2—O3 ^{vii}	83.11 (3)
O3—P1—O2	114.42 (6)	O1 ⁱⁱ —Na2—O3 ^{vii}	83.91 (4)
O3—P1—O1	114.27 (6)	O1 ^v —Na2—O3 ^{vii}	96.09 (4)
O2—P1—O1	113.31 (6)	O3—Na2—O3 ^{vii}	180.00 (4)

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