Dipotassium trisodium triphosphate, $K_2Na_3P_3O_{10}$

Meryem Moutataouia,[a] Mohammed Lamire,[a]* Mohamed Saadi[b] and Lahcen El Ammari[b]

[a]Laboratoire de Physico-Chimie des Matériaux Inorganiques, Faculté des Sciences Aïn Chock, Casablanca, Morocco, and [b]Laboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Batouta, BP 1014, Rabat, Morocco

Correspondence e-mail: m_lamire@yahoo.fr

The structure of the title compound, $K_2Na_3P_3O_{10}$, is characterized by open chains of three PO_4 tetrahedra linked by single oxygen bridges. The P_3O_{10} 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 isotypic with that of the high-temperature form of $Na_5P_3O_{10}$ 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

$K_2Na_3P_3O_{10}$	b = 5.6332 (2) Å
M_r = 400.08	c = 18.6577 (8) Å
Monoclinic, $C2/c$	β = 96.199 (3)°
a = 9.8866 (4) Å	V = 1033.03 (7) Å^3

Z = 4	T = 296 K
Mo $K\alpha$ radiation	0.19 × 0.14 × 0.10 mm
μ = 1.55 mm^{-1}	

Data collection

Bruker APEXII CCD detector diffractometer	12463 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)	2830 independent reflections
T_{min} = 0.511, T_{max} = 0.638	2136 reflections with $I > 2\sigma(I)$
	R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032	85 parameters
$wR(F^2)$ = 0.080	$\Delta\rho_{max}$ = 0.63 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.97 e Å^{-3}
2830 reflections	

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).

References

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Corbridge, D. E. C. (1960). *Acta Cryst.* **13**, 263–269.
Cruickshank, D. W. J. (1964). *Acta Cryst.* **17**, 674–675.
Davies, D. R. & Corbridge, D. E. C. (1958). *Acta Cryst.* **11**, 315–319.
Dymon, J. J. & King, A. J. (1951). *Acta Cryst.* **4**, 378–379.
Dyroff, D. R. (1965). PhD thesis, California Institute of Technology, Pasadena, California, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (1999). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wiench, D. M., Jansen, M. & Hoppe, R. (1982). *Z. Anorg. Allg. Chem.* **488**, 80–86.

supplementary materials

Dipotassium trisodium triphosphate, K₂Na₃P₃O₁₀

Meryem Moutataouia, Mohammed Lamire, Mohamed Saadi and Lahcen El Ammari

Comment

The present triphosphate was obtained by chance, during the preparation of a mixed pyrophosphate. A bibliographic study of alkali triphosphates like $M_5P_3O_{10}$ 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 $Na2O_6$ octahedra, $Na1O_6$ and $K1O_8$ 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 $Na1^+$ 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).

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 shuts 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.

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.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); 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).
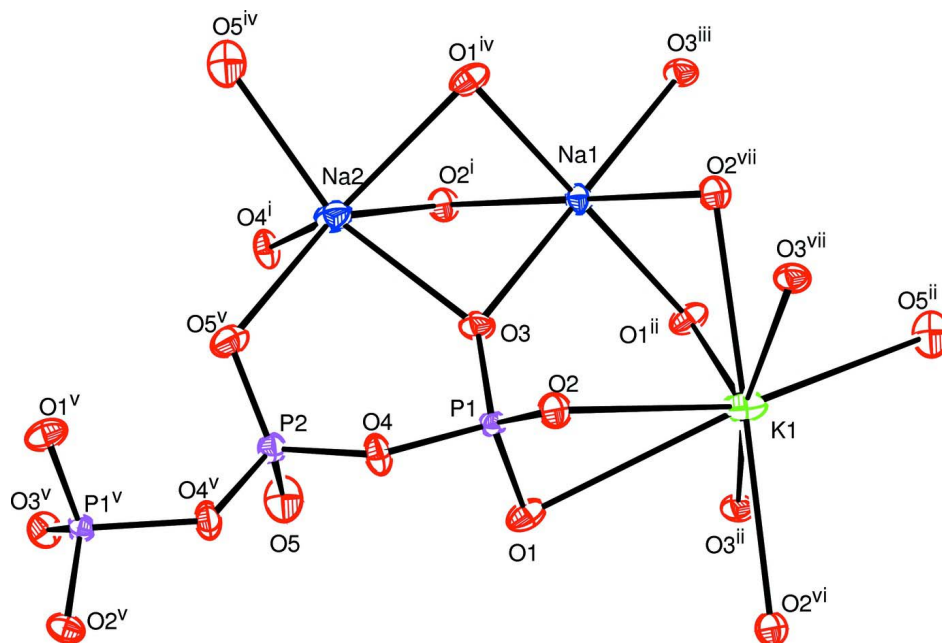
Figure 1

Plot of $K_2Na_3P_3O_{10}$ 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.
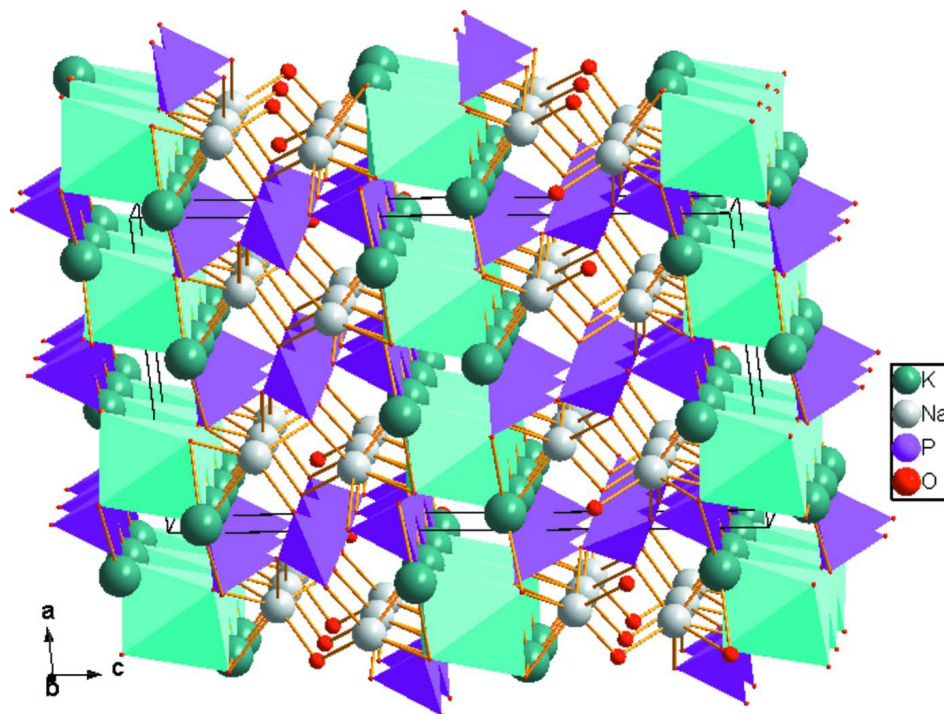
Figure 2

Projection view of the $K_2Na_3P_3O_{10}$ framework structure showing tunnel running along b direction where the Na1 atoms are located.

Dipotassium trisodium triphosphate

Crystal data

$K_2Na_3P_3O_{10}$	$F(000) = 784$
$M_r = 400.08$	$D_x = 2.572$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -c 2yc	Cell parameters from 2830 reflections
$a = 9.8866$ (4) Å	$\theta = 4.2–38.3°$
$b = 5.6332$ (2) Å	$\mu = 1.55$ mm^{-1}
$c = 18.6577$ (8) Å	$T = 296$ K
$\beta = 96.199$ (3)°	Block, colourless
$V = 1033.03$ (7) Å^3	$0.19 \times 0.14 \times 0.10$ mm
$Z = 4$	

Data collection

Bruker APEXII CCD detector diffractometer	12463 measured reflections
	2830 independent reflections
Radiation source: fine-focus sealed tube	2136 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.032$
ω and φ scans	$\theta_{max} = 38.3°$, $\theta_{min} = 4.2°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)	$h = -17 \rightarrow 17$
	$k = -8 \rightarrow 9$
$T_{min} = 0.511$, $T_{max} = 0.638$	$l = -32 \rightarrow 32$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.7541P]$
$wR(F^2) = 0.080$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.001$
2830 reflections	$\Delta\rho_{max} = 0.63$ e Å^{-3}
85 parameters	$\Delta\rho_{min} = -0.97$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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 (Å^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, º)

K1—O2ⁱ	2.7960 (11)	P2—O4	1.5961 (11)
K1—O1ⁱⁱ	2.8051 (11)	P2—O4ⁱᵛ	1.5961 (11)
K1—O3ⁱⁱ	2.8291 (11)	Na1—O5ᵛ	2.4172 (14)
K1—O1ⁱⁱⁱ	2.8443 (11)	Na1—O3	2.4278 (12)
K1—O3ⁱ	2.8987 (11)	Na1—O5ⁱᵛ	2.4655 (13)
K1—O2ⁱⁱⁱ	2.9106 (11)	Na1—O2ᵛⁱ	2.4753 (12)
K1—O2	3.0740 (11)	Na1—O1ᵛ	2.5864 (13)
K1—O5ⁱⁱ	3.1272 (12)	Na1—O4ᵛⁱ	2.8527 (13)
P1—O3	1.5080 (10)	Na2—O2ᵛⁱ	2.3599 (10)
P1—O2	1.5086 (10)	Na2—O2ⁱ	2.3599 (10)
P1—O1	1.5092 (11)	Na2—O1ⁱⁱ	2.3850 (10)
P1—O4	1.6506 (10)	Na2—O1ᵛ	2.3850 (10)
P2—O5	1.4951 (11)	Na2—O3	2.3871 (10)
P2—O5ⁱᵛ	1.4951 (11)	Na2—O3ᵛⁱⁱ	2.3871 (10)
O2ⁱ—K1—O1ⁱⁱ	68.95 (3)	O3—P1—O4	105.94 (6)
O2ⁱ—K1—O3ⁱⁱ	122.11 (3)	O2—P1—O4	101.64 (6)
O1ⁱⁱ—K1—O3ⁱⁱ	53.46 (3)	O1—P1—O4	105.69 (6)
O2ⁱ—K1—O1ⁱⁱⁱ	119.70 (3)	O5—P2—O5ⁱᵛ	117.54 (10)
O1ⁱⁱ—K1—O1ⁱⁱⁱ	171.32 (4)	O5—P2—O4	110.04 (6)
O3ⁱⁱ—K1—O1ⁱⁱⁱ	117.99 (3)	O5ⁱᵛ—P2—O4	110.66 (6)
O2ⁱ—K1—O3ⁱ	52.85 (3)	O5—P2—O4ⁱᵛ	110.66 (6)
O1ⁱⁱ—K1—O3ⁱ	121.17 (3)	O5ⁱᵛ—P2—O4ⁱᵛ	110.04 (6)
O3ⁱⁱ—K1—O3ⁱ	166.73 (4)	O4—P2—O4ⁱᵛ	95.75 (8)
O1ⁱⁱⁱ—K1—O3ⁱ	67.49 (3)	O5ᵛ—Na1—O3	156.77 (5)
O2ⁱ—K1—O2ⁱⁱⁱ	171.11 (4)	O5ᵛ—Na1—O5ⁱᵛ	103.17 (3)
O1ⁱⁱ—K1—O2ⁱⁱⁱ	119.38 (3)	O3—Na1—O5ⁱᵛ	83.79 (4)
O3ⁱⁱ—K1—O2ⁱⁱⁱ	66.53 (3)	O5ᵛ—Na1—O2ᵛⁱ	105.62 (4)
O1ⁱⁱⁱ—K1—O2ⁱⁱⁱ	51.95 (3)	O3—Na1—O2ᵛⁱ	79.92 (4)
O3ⁱ—K1—O2ⁱⁱⁱ	119.27 (3)	O5ⁱᵛ—Na1—O2ᵛⁱ	141.23 (5)
O2ⁱ—K1—O2	81.62 (3)	O5ᵛ—Na1—O1ᵛ	80.29 (4)
O1ⁱⁱ—K1—O2	109.04 (3)	O3—Na1—O1ᵛ	78.96 (4)
O3ⁱⁱ—K1—O2	119.82 (3)	O5ⁱᵛ—Na1—O1ᵛ	133.13 (5)
O1ⁱⁱⁱ—K1—O2	73.13 (3)	O2ᵛⁱ—Na1—O1ᵛ	77.52 (4)
O3ⁱ—K1—O2	72.92 (3)	O5ᵛ—Na1—O4ᵛⁱ	86.22 (4)
O2ⁱⁱⁱ—K1—O2	92.17 (3)	O3—Na1—O4ᵛⁱ	114.11 (4)
O2ⁱ—K1—O5ⁱⁱ	82.10 (3)	O5ⁱᵛ—Na1—O4ᵛⁱ	103.10 (4)
O1ⁱⁱ—K1—O5ⁱⁱ	65.69 (3)	O2ᵛⁱ—Na1—O4ᵛⁱ	54.20 (3)
O3ⁱⁱ—K1—O5ⁱⁱ	70.56 (3)	O1ᵛ—Na1—O4ᵛⁱ	123.75 (4)
O1ⁱⁱⁱ—K1—O5ⁱⁱ	114.55 (3)	O2ᵛⁱ—Na2—O2ⁱ	180.0
O3ⁱ—K1—O5ⁱⁱ	96.19 (3)	O2ᵛⁱ—Na2—O1ⁱⁱ	96.16 (4)
O2ⁱⁱⁱ—K1—O5ⁱⁱ	103.88 (3)	O2ⁱ—Na2—O1ⁱⁱ	83.85 (4)
O2—K1—O5ⁱⁱ	163.68 (3)	O2ᵛⁱ—Na2—O1ᵛ	83.85 (4)
O2ⁱ—K1—O1	108.73 (3)	O2ⁱ—Na2—O1ᵛ	96.15 (4)
O1ⁱⁱ—K1—O1	83.10 (3)	O1ⁱⁱ—Na2—O1ᵛ	180.000 (1)
O3ⁱⁱ—K1—O1	72.27 (3)	O2ᵛⁱ—Na2—O3	83.11 (3)
O1ⁱⁱⁱ—K1—O1	92.86 (3)	O2ⁱ—Na2—O3	96.89 (3)
O3ⁱ—K1—O1	120.50 (3)	O1ⁱⁱ—Na2—O3	96.09 (4)

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