O8	0.390 (1)	0.037 (1)	0.3283 (3)	0.010 (1)
O9	0.2603 (9)	0.255 (1)	0.4180 (4)	0.011 (1)
O10	−0.037 (1)	−0.040 (1)	0.4001 (4)	0.016 (2)

Table 2. *Selected geometric parameters (Å)*

Eu1—O1^{i}	2.507 (8)	Eu3—O4	2.583 (9)
Eu1—O3	2.622 (9)	Eu3—O5^{vii}	2.301 (9)
Eu1—O6	2.423 (9)	Eu3—O5^{ii}	2.379 (9)
Eu1—O7	2.670 (9)	Eu3—O6^{ii}	2.375 (9)
Eu1—O7^{ii}	2.384 (9)	Eu3—O8	2.572 (8)
Eu1—O8	2.667 (9)	Eu3—O9	2.385 (9)
Eu1—O8^{iii}	2.521 (9)	Eu3—O10^{iii}	2.39 (1)
Eu1—O9	2.298 (8)	Si1—O2	1.639 (9)
Eu1—O10	2.69 (1)	Si1—O4^{viii}	1.648 (9)
Eu2—O1^{iv}	2.409 (8)	Si1—O6	1.620 (9)
Eu2—O1^{v}	2.372 (8)	Si1—O10	1.60 (1)
Eu2—O2^{vi}	2.35 (1)	Si2—O1	1.649 (8)
Eu2—O2^{ii}	2.49 (1)	Si2—O3^{v}	1.633 (9)
Eu2—O3^{iv}	2.292 (9)	Si2—O7	1.62 (1)
Eu2—O3^{v}	2.575 (9)	Si2—O8	1.612 (9)
Eu2—O6^{ii}	2.539 (8)	B—O4	1.43 (1)
Eu2—O7^{iv}	2.576 (9)	B—O5	1.36 (1)
Eu2—O10^{vi}	2.94 (1)	B—O9	1.34 (1)
Eu3—O2^{vi}	2.435 (9)		

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

The five highest peaks in the residual electron density are all less that 0.75 Å from a rare earth ion.

Data collection: CAD-4 software (Enraf–Nonius, 1977). Cell refinement: CAD-4 software. Data reduction: *MolEN* (Lorentz–polarization) (Fair, 1990). Program(s) used to solve structure: *MolEN* (Patterson). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *GCIF*, local program.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: DU1155). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$KGaAs_2O_7$

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Abstract

Potassium gallium diarsenate(4−), $KGaAs_2O_7$, has been synthesized from a flux and structurally characterized by single-crystal X-ray diffraction. It is isostructural with $KAlAs_2O_7$ [Boughzala & Jouini (1995). *Acta Cryst.* C**51**, 179–181] and $RbAlAs_2O_7$ [Boughzala, Driss & Jouini (1993). *Acta Cryst.* C**49**, 425–427].

Comment

Aluminophosphates have been extensively studied because they are known to be useful materials for catalysis and adsorption (Wilson, Lok, Messing, Cannan & Flanigen, 1982). The replacement of aluminium by gallium has yielded both novel structure types and phases that are analogous to known aluminophosphates and aluminosilicates. In contrast, few galloarsenates have been reported. There are only two structurally characterized galloarsenates in the literature, namely, $GaAsO_4$ (Goiffon, Jumas, Maurin & Philippot, 1986) and $Ga_6(AsO_4)_6(C_2H_7N)_4(HF)_2(H_2O)$ (Chen, Li, Yang & Xu, 1989). In this paper, we describe the synthesis and crystal structure of a potassium gallium arsenate, $KGaAs_2O_7$.

The structure contains intersecting tunnels with the K^+ cations located at the tunnel intersections. A polyhedral view of the structure along the *c* axis is shown in Fig. 1. The shortest O···O distance across the window of the tunnel along the *c* axis is 5.3 Å, which is only slightly larger than the corresponding distance in $KAlAs_2O_7$ (Boughzala & Jouini, 1995). The framework is composed of GaO_6 octahedra joined through As_2O_7 diarsenate groups by corner sharing. The atom labeling of the asymmetric unit is shown in Fig. 2.

All of the GaO_6 octahedra are regular [Ga—O 1.936 (4)–2.002 (4) Å, O—Ga—O 87.4 (1)–93.0 (2)°]. The AsO_4 tetrahedra of the As_2O_7 group are in a staggered configuration. The mean bridging and terminal As—O bond lengths are 1.760 (4) and 1.664 (4) Å, respectively, and the As—O—As angle is 119.1 (2)°. These values are comparable with 1.764 (4), 1.660 Å and 118.3 (2)° for $KAlAs_2O_7$.

The two crystallographically distinct K1 and K2 cations in $KGaAs_2O_7$ lie on inversion centers. The coordination numbers of the K^+ ions were determined on

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the basis of the maximum gap in the K—O distance ranked in increasing order. The maximum K—O distance agrees with the 3.35 Å cut-off of Donnay & Allmann (1970). Atoms K1 and K2 are therefore ten- and eight-coordinated, respectively. The K1—O distances, 2.841 (4)–3.206 (4) Å, are similar to those in $KAlAs_2O_7$. The longest K2—O bond distance in $KGaAs_2O_7$ is 3.346 (4) Å, which is a little longer than the corresponding distance in the Al analogue [3.201 (5) Å]. Bond valence sums for K1 and K2 are 1.16 and 0.96, respectively. The relatively high bond valence sum for K1 indicates that it is more tightly bound, and therefore its displacement parameters are smaller than those of K2 [U_{eq}(K2) is twice as large as U_{eq}(K1)].

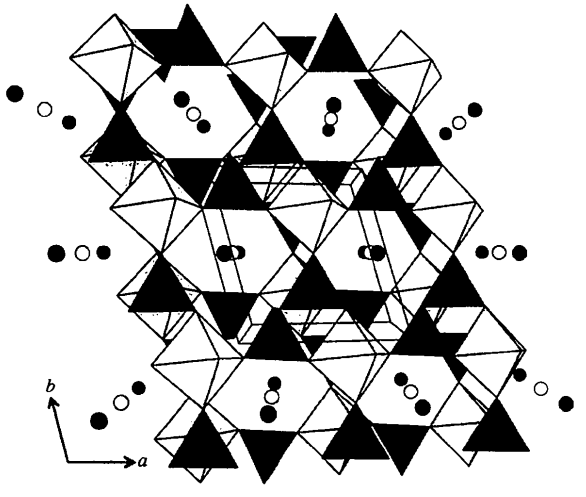

Fig. 1. A polyhedral representation of the $KGaAs_2O_7$ structure viewed along the c axis. In this representation, the corners of the octahedra and tetrahedra are O atoms and the Ga and As atoms are at the center of each octahedron and tetrahedron, respectively. Black circles are K1 atoms; open circles are K2 atoms.

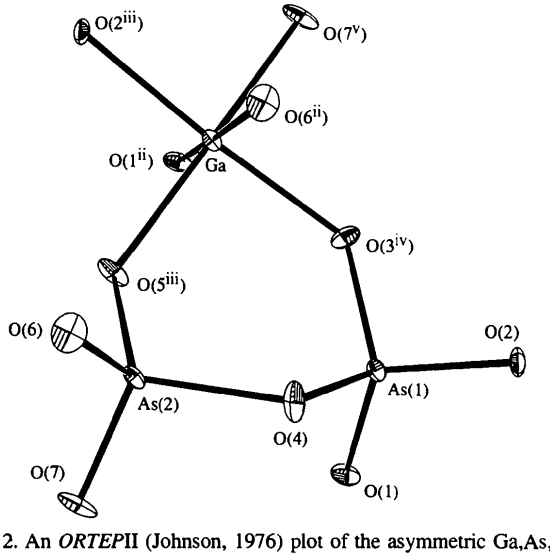

Fig. 2. An *ORTEP*II (Johnson, 1976) plot of the asymmetric Ga,As,O unit with 50% probability displacement ellipsoids. Symmetry codes: (ii) $-x$, $1-y$, $1-z$; (iii) $1-x$, $1-y$, $1-z$; (iv) x, $y-1$, z; (v) $-x$, $-y$, $1-z$.

Experimental

KH_2AsO_4 (s) and $(NH_4)H_2AsO_4$ (s) were prepared from a solution of arsenic acid with potassium hydroxide and ammonium hydroxide, respectively. 0.6735 g KH_2AsO_4, 0.4433 g $(NH_4)H_2AsO_4$ and 0.1847 g Ga_2O_3 were thoroughly mixed in a 15 ml platinum crucible. The mixture was heated at a rate of 100 K h^{-1} to 773 K, maintained at 773 K for 6 h, heated to 973 K, maintained at 973 K for 10 h, cooled at 5 K h^{-1} to 773 K and quenched to room temperature by removing the crucible from the furnace. The flux was dissolved with hot water and the solid product was obtained by suction filtration. The product contained colorless chunk-shaped crystals of $KGaAs_2O_7$ and a small amount of $GaAsO_4$, as indicated by X-ray analysis using a Rigaku powder diffractometer with filtered Cu radiation. The intensity of the strongest reflection of the impurity phase $GaAsO_4$ (2θ = 25.94°) was about a tenth that of the strongest reflection of the major phase and all other observed reflections in the diffraction pattern corresponded very well with those calculated from the single-crystal data.

Crystal data

$KGaAs_2O_7$	Mo $K\alpha$ radiation
M_r = 370.65	λ = 0.71069 Å
Triclinic	Cell parameters from 25 reflections
$P\bar{1}$	θ = 10.30–18.20°
a = 6.271 (1) Å	μ = 16.03 mm^{-1}
b = 6.376 (1) Å	T = 293 K
c = 8.169 (1) Å	Chunk
α = 96.45 (1)°	0.30 × 0.20 × 0.13 mm
β = 103.86 (1)°	Colorless
γ = 103.87 (1)°	
V = 302.84 (8) $Å^3$	
Z = 2	
D_x = 4.065 Mg m^{-3}	
D_m not measured	

Data collection

Nonius CAD-4 diffractometer	1571 observed reflections [$I_{net} > 2.5\sigma(I_{net})$]
$\omega/2\theta$ scans	R_{int} = 0.038
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)	θ_{max} = 29.89°
T_{min} = 0.451, T_{max} = 0.998	$h = -8 \rightarrow 8$
2068 measured reflections	$k = 0 \rightarrow 8$
1752 independent reflections	$l = -11 \rightarrow 11$
	3 standard reflections frequency: 60 min intensity decay: 2.5%

Refinement

Refinement on F	Extinction correction: Larson (1970)
R = 0.033	

$wR = 0.041$
$S = 2.43$
1571 reflections
104 parameters
$w = 1/[\sigma^2(F) + 0.0001F^2]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.73$ e Å^{-3}
$\Delta\rho_{min} = -1.13$ e Å^{-3}

Extinction coefficient: 25936 (709)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
K1	0	1/2	1/2	0.036 (1)
K2	0	1/2	0	0.071 (2)
Ga	0.37526 (9)	0.19236 (8)	0.72415 (6)	0.0122 (3)
As1	0.17389 (8)	0.96533 (8)	0.30932 (6)	0.0121 (2)
As2	−0.48212 (8)	0.25386 (7)	0.16579 (6)	0.0119 (3)
O1	−0.0908 (6)	0.8039 (6)	0.2183 (4)	0.016 (2)
O2	0.3361 (6)	0.7913 (6)	0.3308 (5)	0.017 (2)
O3	0.2068 (6)	1.1509 (6)	0.4786 (4)	0.015 (2)
O4	0.2337 (6)	1.1146 (6)	0.1486 (4)	0.017 (2)
O5	0.4469 (7)	0.7384 (6)	0.0285 (4)	0.018 (2)
O6	−0.4286 (7)	0.4979 (6)	0.2852 (5)	0.019 (2)
O7	−0.3102 (7)	0.1238 (6)	0.2775 (5)	0.017 (2)

Table 2. *Selected geometric parameters (Å, °)*

K1—O1	3.206 (4)	Ga—O5^{iii}	2.002 (4)
K1—O2	3.146 (4)	Ga—O6^{ii}	1.936 (4)
K1—O3^{i}	2.841 (4)	Ga—O7^{iv}	1.956 (4)
K1—O6	2.832 (4)	As1—O1	1.669 (3)
K1—O7	2.816 (4)	As1—O2	1.672 (4)
K2—O1	2.738 (4)	As1—O3	1.654 (3)
K2—O2	3.076 (4)	As1—O4	1.765 (3)
K2—O5	2.790 (4)	As2—O4^{v}	1.754 (4)
K2—O6	3.346 (4)	As2—O5^{vi}	1.658 (3)
Ga—O1^{ii}	1.955 (4)	As2—O6	1.654 (4)
Ga—O2^{iii}	1.949 (4)	As2—O7	1.676 (4)
Ga—O3^{i}	1.985 (3)		
O1^{ii}—Ga—O2^{iii}	176.4 (2)	O6^{ii}—Ga—O7^{iv}	177.2 (2)
O1^{ii}—Ga—O3^{i}	88.4 (2)	O1—As1—O2	104.3 (2)
O1^{ii}—Ga—O5^{iii}	92.0 (2)	O1—As1—O3	116.3 (2)
O1^{ii}—Ga—O6^{ii}	88.7 (2)	O1—As1—O4	103.4 (2)
O1^{ii}—Ga—O7^{iv}	90.6 (2)	O2—As1—O3	117.8 (2)
O2^{iii}—Ga—O3^{i}	92.0 (2)	O2—As1—O4	108.1 (2)
O2^{iii}—Ga—O5^{iii}	87.4 (2)	O3—As1—O4	105.9 (2)
O2^{iii}—Ga—O6^{ii}	87.7 (2)	O4^{v}—As2—O5^{vi}	109.6 (2)
O2^{iii}—Ga—O7^{iv}	93.0 (2)	O4^{v}—As2—O6	106.6 (2)
O3^{i}—Ga—O5^{iii}	175.1 (2)	O4^{v}—As2—O7	108.5 (2)
O3^{i}—Ga—O6^{ii}	84.3 (2)	O5^{vi}—As2—O6	114.5 (2)
O3^{i}—Ga—O7^{iv}	93.0 (2)	O5^{vi}—As2—O7	111.3 (2)
O5^{iii}—Ga—O6^{ii}	90.9 (2)	O6—As2—O7	106.1 (2)
O5^{iii}—Ga—O7^{iv}	91.9 (2)	As1—O4—As2^{vii}	119.1 (2)

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

Data collection: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Cell refinement: *NRCVAX*. Data reduction: *NRCVAX*. Program(s) used to solve structure: *NRCVAX*. Program(s) used to refine structure: *NRCVAX*. Molecular graphics: *NRCVAX* and *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: KH1086). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$LaMo_8O_{14}$

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Abstract

The crystal structure of lanthanum octamolybdate contains an equal mixture of *cis*-edge-sharing and *trans* bi-face-capped octahedral Mo_8 clusters. These clusters and the O atoms, the arrangement of which derives from close packing with the layer sequence *ABAC*..., form sheets parallel to the (*bc*) plane of the orthorhombic unit cell. The Mo—Mo distances range from 2.5877 (4) to 2.7780 (5) Å and from 2.5961 (5) to 2.8866 (7) Å in the *trans* and *cis*-edge-sharing isomeric clusters, respectively. The shortest Mo—Mo distance between the Mo_8 clusters within the same layer is 3.0807 (4) Å and that between clusters in adjacent layers is 3.6380 (5) Å. The Mo—O distances lie between 2.026 (3) and 2.120 (3) Å in the *trans* isomer and between 1.938 (3) and 2.169 (3) Å in the *cis*-edge-sharing

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