presence of weak reflections with $h + k + l = 2n + 1$ indicated a superstructure with a primitive unit cell.

In the first stage, the superstructure reflections were neglected and the average structure was refined in the space group *I*4/*mcm* using the structure of $PdGa_5$ as an initial model (Table 1*a*).

Because of their consecutive atomic numbers, aluminium and silicon are difficult to distinguish precisely. Nevertheless, the site labelled Al appeared to be occupied – at least predominantly – by aluminium. For the mixed site, the occupancies of Si and Al were chosen to fit the empirical formula $FeAl_{2.7}Si_{2.3}$.

In the second stage, the superstructure was investigated. As the observed reflection conditions (*hk*0: $h + k = 2n$; 00*l*: $l = 2n$; 0*kl*: $k = 2n$ and/or $l = 2n$) did not match any of the diffraction symbols, the crystal was considered to be twinned by pseudosymmetry. Among the subgroups of *I*4/*mcm*, the space group *Pbcn* was consistent with all the observed conditions. The two components of the twin could be related either by a (110) symmetry plane or by a [001] fourfold axis. The perfect 4/*mmm* symmetry observed led us to suppose that the crystal was polysynthetically twinned, *i.e.* constituted of numerous small domains of the two components *A* and *B* with virtually equal volumes V_A and V_B. Then the observed intensity of an *hkl* reflection is the half-sum of the *hkl* and *khl* reflection intensities of a similar untwinned crystal. The superstructure was refined using the average structure as an initial model. Only reflections unaffected by the twinning were used:

(*a*) the *hhl* reflections: 34 of the substructure ($l = 2n$) and 15 of the superstructure ($l = 2n + 1$);

(*b*) among the *h*0*l* (and 0*kl*) reflections, two sets satisfying the condition (i) *h* (or *k*) $= 2n + 1$ and $l = 2n$, and the condition (ii) *h* (or *k*) $= 2n$ and $l = 2n + 1$; the reflections of the two sets were considered as *h*0*l* and 0*kl*, respectively, and their intensities were multiplied by two.

The distribution of the Al and Si atoms on the two 8(*d*) sites corresponds to the lowest *R* factor for the stoichiometric formula $FeAl_3Si_2$ (Table 1*b*). No attempt was made to fit the empirical formula $FeAl_{2.7}Si_{2.3}$ by refining the Al(2) site as a mixed $(Al_{1-x}Si_x)(2)$ site.

All the programs used belong to the *SDP* system (B. A. Frenz & Associates, Inc., 1982). The absorption corrections were applied using the *ABSCOR* program (Coppens, Leiserowitz & Rabinovich, 1965).

We thank the Laboratoire Central de Recherche de Chedde of the Péchiney Electrométallurgie Society for crystal preparation.

Lists of structure factors, anisotropic displacement parameters, experimental details for the average structure and complete geometry have been deposited with the IUCr (Reference: DU1090). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 179–181

$KAlAs_2O_7$

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(*Received* 16 *May* 1994; *accepted* 12 *September* 1994)

Abstract

Potassium aluminium pyroarsenate, $KAlAs_2O_7$, is isostructural with $RbAlAs_2O_7$. Although these two structures are not isotypic with structures of the series $M^IM^{III}P_2O_7$ form I, they are closely related to them. Relevant discussion and references are given in a previous paper [Boughzala, Driss & Jouini (1993). *Acta Cryst.* C**49**, 425–427].

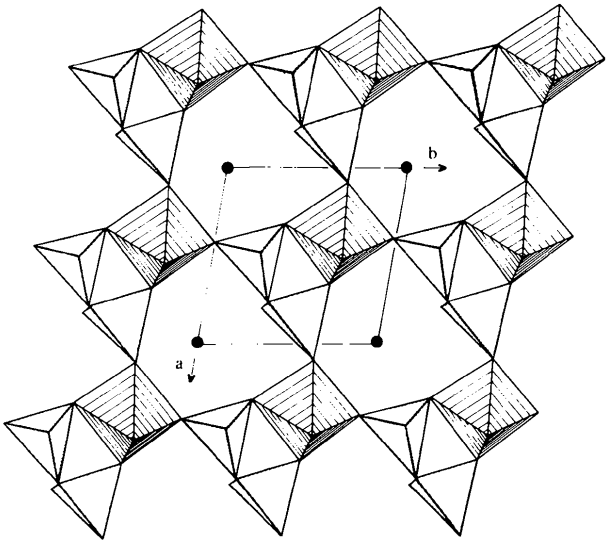

Fig. 1. Projection of half of the unit-cell contents of the title structure onto the (001) plane showing a layer of connected polyhedra.

Comment

The structure of $KAlAs_2O_7$ is built from $AlAs_2O_{11}$ units comprising one AlO_6 octahedron and one As_2O_7 diarsenate group in which two AsO_4 tetrahedra point in opposite directions. These units are connected by hetero-polyhedral linkages to form a three-dimensional framework having intersecting tunnels in which the K^+ ions are located.

Experimental

The compound was prepared according the method described by Boughzala & Jouini (1992).

Crystal data

$KAlAs_2O_7$
$M_r = 327.92$
Triclinic
$P\bar{1}$
$a = 6.192$ (4) Å
$b = 6.297$ (3) Å
$c = 8.1060$ (10) Å
$\alpha = 96.600$ (8)°
$\beta = 104.517$ (8)°
$\gamma = 102.864$ (7)°
$V = 293.4$ (2) Å^3
$Z = 2$
$D_x = 3.712$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 14$–16°
$\mu = 12.234$ mm^{-1}
$T = 293$ (2) K
Tablet
0.08 × 0.07 × 0.006 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans [θ scan width $(0.65 + 0.35\tan\theta)°$]
Absorption correction: spherical
$T_{\min} = 0.9986$, $T_{\max} = 1.0000$
1057 measured reflections
983 independent reflections

883 observed reflections [$I > 2\sigma(I)$]
$\theta_{\max} = 24.93°$
$h = -7 \rightarrow 7$
$k = -7 \rightarrow 7$
$l = 0 \rightarrow 9$
2 standard reflections frequency: 120 min intensity decay: 0.66%

Refinement

Refinement on F^2
$R(F) = 0.0331$
$wR(F^2) = 0.0902$
$S = 0.537$
983 reflections
104 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1264P)^2 + 4.1692P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.570$ e Å^{-3}
$\Delta\rho_{\min} = -1.871$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0191 (33)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
As1	0.48649 (9)	0.24567 (9)	0.33563 (7)	0.0069 (3)
As2	0.82650 (9)	0.53194 (9)	0.18966 (7)	0.0070 (3)
K1	0	0	0	0.0382 (7)
K2	0	0	1/2	0.0460 (8)
Al1	0.3752 (3)	0.6911 (3)	0.2262 (2)	0.0039 (4)
O1	0.4218 (7)	−0.0059 (7)	0.2210 (6)	0.0132 (9)
O2	0.4569 (7)	0.2484 (7)	0.5329 (5)	0.0121 (9)
O3	0.3179 (7)	0.3807 (7)	0.2200 (5)	0.0112 (9)
O4	0.7743 (7)	0.3726 (7)	0.3477 (5)	0.0112 (9)
O5	0.7847 (7)	0.3495 (7)	0.0129 (5)	0.0120 (9)
O6	0.6607 (7)	0.7086 (7)	0.1776 (6)	0.0109 (9)
O7	1.0944 (7)	0.6892 (7)	0.2787 (5)	0.0096 (9)

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

As1—O1	1.651 (4)	K1···O6^{iv}	3.203 (4)
As1—O2	1.653 (4)	K1···O6^{v}	3.203 (4)
As1—O3	1.667 (4)	K2···O7^{iv}	2.760 (4)
As1—O4	1.756 (4)	K2···O7^{vi}	2.760 (4)
As2—O5	1.656 (4)	K2···O2^{vii}	2.842 (4)
As2—O7	1.661 (4)	K2···O2	2.842 (4)
As2—O6	1.669 (4)	K2···O6^{iv}	3.007 (4)
As2—O4	1.764 (4)	K2···O6^{vi}	3.007 (4)
K1···O1	2.783 (4)	K2···O4^{viii}	3.201 (4)
K1···O1^{i}	2.783 (4)	K2···O4^{iii}	3.201 (4)
K1···O3^{i}	2.821 (4)	Al1—O1^{ix}	1.872 (5)
K1···O3	2.821 (4)	Al1—O6	1.888 (5)
K1···O5^{ii}	2.820 (4)	Al1—O7^{iii}	1.889 (4)
K1···O5^{iii}	2.820 (4)	Al1—O3	1.900 (4)
K1···O7^{iv}	3.184 (4)	Al1—O5^{v}	1.901 (4)
K1···O7^{v}	3.184 (4)	Al1—O2^{vi}	1.917 (4)
O1—As1—O2	113.6 (2)	O1^{ix}—Al1—O3	177.1 (2)
O1—As1—O3	106.2 (2)	O6—Al1—O3	92.3 (2)
O2—As1—O3	112.0 (2)	O7^{iii}—Al1—O3	90.4 (2)
O1—As1—O4	106.9 (2)	O1^{ix}—Al1—O5^{v}	85.2 (2)
O2—As1—O4	109.9 (2)	O6—Al1—O5^{v}	91.9 (2)
O3—As1—O4	108.0 (2)	O7^{iii}—Al1—O5^{v}	88.9 (2)
O5—As2—O7	116.3 (2)	O3—Al1—O5^{v}	92.0 (2)
O5—As2—O6	117.5 (2)	O1^{ix}—Al1—O2^{vi}	91.3 (2)
O7—As2—O6	104.9 (2)	O6—Al1—O2^{vi}	87.4 (2)
O5—As2—O4	105.3 (2)	O7^{iii}—Al1—O2^{vi}	91.7 (2)
O7—As2—O4	103.8 (2)	O3—Al1—O2^{vi}	91.6 (2)
O6—As2—O4	108.0 (2)	O5^{v}—Al1—O2^{vi}	176.4 (2)
O1^{ix}—Al1—O6	88.2 (2)	As1—O3—Al1	127.6 (2)
O1^{ix}—Al1—O7^{iii}	89.1 (2)	As1—O4—As2	118.3 (2)
O6—Al1—O7^{iii}	177.1 (2)	As2—O6—Al1	131.2 (3)

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

Data were corrected for Lorentz and polarization effects.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: NA1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 181–183

Lithium Berylloarsenate Hydrate, $LiBeAsO_4.H_2O$

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(*Received* 4 *August* 1994; *accepted* 1 *September* 1994)

Abstract

The full structure of lithium berylloarsenate hydrate, $LiBeAsO_4.H_2O$, as refined using X-ray powder data, is reported. This phase is briefly compared with other known Li–*A*-type materials.

Comment

Open-framework tetrahedrally connected berylloarsenates are part of the growing family of Group 2/12/15 (Be/Zn)(P/As) molecular sieves (Gier & Stucky, 1991; Harrison, Nenoff, Gier & Stucky, 1993). Berylloarsenate isostructures of zeolite rho (Parise *et al.*, 1992) and sodalite (Gier, Harrison & Stucky, 1991; Harrison, Gier & Stucky, 1994) have been described previously.

Lithium berylloarsenate hydrate is confirmed to be an aluminosilicate Li–*A*-type isostructure (Gier & Stucky, 1991). The Li–*A*-type framework topology has been described fully by Smith (1988) and consists of an open three-dimensional array built up from four, six and eight rings, enclosing one-dimensional channels, which propagate in the polar [001] direction. Like other known Li–*A*-type phases, $LiBeAsO_4.H_2O$ shows complete 1:1

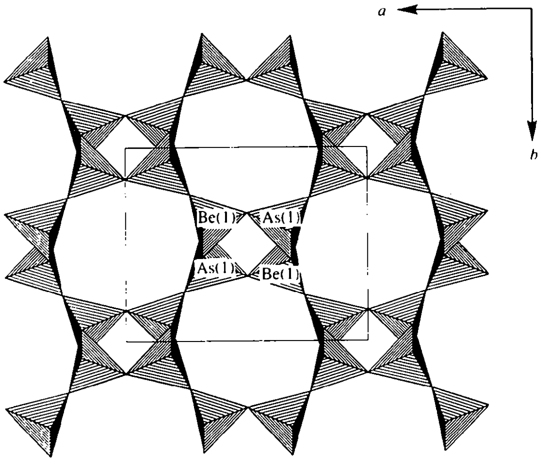

Fig. 1. Polyhedral view down [001] of the $LiBeAsO_4.H_2O$ framework structure, showing the ordered alternating BeO_4 and AsO_4 units.

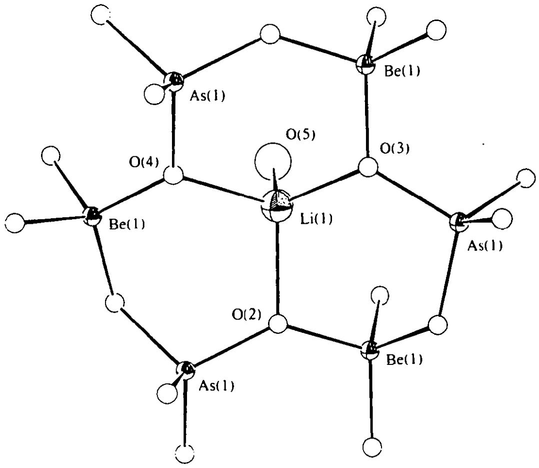

Fig. 2. View showing detail of the six-ring window environment in $LiBeAsO_4.H_2O$. The Li^+ cation makes three bonds to framework O atoms and a fourth tetrahedral vertex to the water molecule [atom O(5)], occupying the central region of the main [001] channel (arbitrary atomic radii).

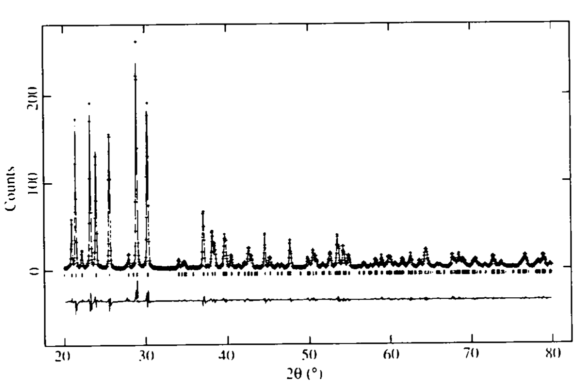

Fig. 3. Final observed (crosses), calculated (line) and difference profiles for the Rietveld refinement of the structure of $LiBeAsO_4.H_2O$. Allowed reflection positions are indicated by tick marks.

Acta Crystallographica Section C
ISSN 0108-2701 ©1995