

11 parameters	Atomic scattering factors
$w = 1/[\sigma^2(F_o^2) + (0.04F_o^2)^2]$	from <i>International Tables</i>
$(\Delta/\sigma)_{\max} = 0.017$	for <i>Crystallography</i> (1992,
$\Delta\rho_{\max} = 3.75 \text{ e } \text{Å}^{-3}$	Vol. C, Tables 4.2.6.8 and
$\Delta\rho_{\min} = -4.15 \text{ e } \text{Å}^{-3}$	6.1.1.4)

Table 1. *Positional parameters, site occupancies and equivalent isotropic displacement parameters (Å²)*

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j.$$

	Occupancy	x	y	z	U_{eq}
Cs1	1.00	0	0	0	0.0176 (4)
Ag1	0.320 (8)	0	1/2	1/4	0.0151 (4)
Mn1	0.590 (5)	0	1/2	1/4	0.0151 (4)
Te1	1.00	0	0	0.35612 (4)	0.0154 (4)

Table 2. *Selected bond distances (Å) and angles (°) (M = Ag, Mn)*

Cs1—Te1 ⁱ	3.892 (2)	M—Te1 ⁱⁱ	2.794 (1)
Te1 ⁱⁱⁱ —M—Te1 ⁱⁱⁱⁱ	110.23 (2)	Te1 ^v —M—Te1 ^{vi}	107.96 (4)

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

Initial cell parameters and symmetry information for $\text{CsAg}_{0.64}\text{Mn}_{1.18}\text{Te}_2$ were obtained from precession photographs taken at room temperature. Intensity data were processed (Waters & Ibers, 1977) and corrected for absorption (de Meulenaer & Tompa, 1965) on an IBM RS/6000 series computer. The initial Cs, Ag, Mn, and Te positional parameters were obtained from the structure of the isostructural compound CsLiMnS_2 (Schmitz & Bronger, 1987). The structure was refined with the program *SHELXL93* (Sheldrick, 1993). The Ag^+ and Mn^{2+} cations are disordered unequally over one site. The stoichiometry of these metals was determined by constraining the total charge of the Ag/Mn site to be +3 (to maintain charge balance) while refining the occupancies of the Ag^+ and Mn^{2+} cations. The refinement included anisotropic displacement parameters and an extinction parameter. The anisotropic displacement parameters of the Ag^+ and Mn^{2+} cations were constrained to be equal. The final difference electron density map shows no feature with a height greater than 1.3% that of a Cs atom.

$\text{CsAg}_{0.64}\text{Mn}_{1.18}\text{Te}_2$ exhibits the ThCr_2Si_2 structure type (Klepp & Boller, 1978) and comprises layers of edge-sharing *M*-centered tetrahedra (*M* = Ag/Mn) separated by Cs^+ cations.

Data collection: local program. Cell refinement: local program. Data reduction: local program. Molecular graphics: *SHELXTLPC* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXTLPC*.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1142). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Klepp, K. & Boller, H. (1978). *Monatsh. Chem.* **109**, 1049–1078.
 Meulenaer, J. de & Tompa, H. (1965). *Acta Cryst.* **19**, 1014–1018.
 Schmitz, D. & Bronger, W. (1987). *Z. Anorg. Allg. Chem.* **553**, 248–260.
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1994). *SHELXTLPC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Waters, J. M. & Ibers, J. A. (1977). *Inorg. Chem.* **16**, 3273–3277.

Acta Cryst. (1996). **C52**, 2385–2387

A New Europium Borosilicate, $\text{Eu}_3\text{BSi}_2\text{O}_{10}$

LISHENG CHI, HUAYANG CHEN, SHUIQUAN DENG, HONGHUI ZHUANG AND JINSHUN HUANG*

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fujian, Fuzhou 350002, People's Republic of China

(Received 13 February 1996; accepted 18 April 1996)

Abstract

A single crystal of a new europium borosilicate, boron trieuropium disilicon decaoxide, was obtained by high-temperature solid-state reaction and chemical vapour transport methods. The structure is composed of layers of isolated BSiO_6^{5-} and $\text{Si}_2\text{O}_4^{4-}$ anions which alternate along the *c* axis. The layers are linked through three crystallographically independent Eu^{3+} ions to form a three-dimensional network structure.

Comment

Studies of rare earth borosilicates in the last ten years have shown these compounds to be promising phosphor and laser materials. The compounds LnBMO_5 (*M* = Si, Ge) with the stillwellite structure contain a distorted chain of tetrahedral BO_4 groups (Voronkov & Pyatenko, 1967; Ruimont & Tarte, 1988; Jsaryuk *et al.*, 1991); Eu^{3+} in these compounds shows relatively high luminescent efficiency (Antic-Fidancev, 1994). $\text{Nd}_3\text{BGe}_{1.08}\text{Si}_{0.92}\text{O}_{10}$ contains the planar BO_3 group (Serhan *et al.*, 1994) and the Ge and Si atoms are distributed statistically over the tetrahedral sites. The strong B—O bond in this compound influences the crystal field around the rare earth sites, which may be helpful in raising the fluorescence lifetime of the $^4F_{3/2}$ emitting state of Nd^{3+} . We have recently investigated the system $\text{Ln}_2\text{O}_3\text{—B}_2\text{O}_3\text{—SiO}_2$, and we report here the synthesis and crystal structure of $\text{Eu}_3\text{BSi}_2\text{O}_{10}$.

Fig. 1 shows the unit cell of $\text{Eu}_3\text{BSi}_2\text{O}_{10}$. It is apparent from this view that the structure consists of isolated BSiO_6^{5-} and $\text{Si}_2\text{O}_4^{4-}$ anion layers alternating along the *c* axis. The BSiO_6^{5-} anions consist of a BO_3 triangle and an SiO_4^{4-} tetrahedron linked by corner

sharing through an O4c atom. The B—O bond lengths range from 1.34 (1) to 1.43 (1) Å, with a mean value of 1.38 Å; the bond angles range from 114 (1) to 127 (1)°. The Si1O₄⁴⁻ and Si2O₄⁴⁻ tetrahedra are very regular (Liebau, 1985), with Si1—O distances from 1.60 (1) to 1.648 (9) Å (mean value 1.627 Å) and Si2—O distances from 1.612 (9) to 1.649 (8) Å (mean value 1.629 Å), the bond angles ranging from 101.6 (5) to 115.1 (5)° for O—Si1—O and from 101.6 (5) to 119.9 (5)° for O—Si2—O.

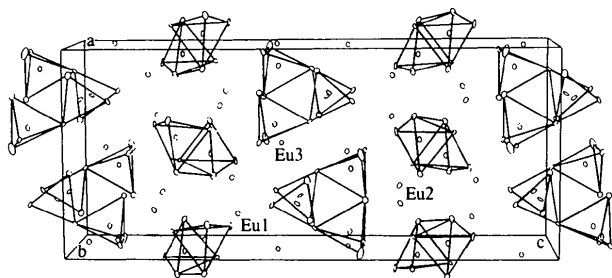


Fig. 1. The arrangement of the BSi1O₆⁵⁻ and Si2O₄⁴⁻ anion layers along *c*. Displacement ellipsoids are plotted at the 50% probability level.

Three crystallographically independent rare earth ions, Eu1³⁺, Eu2³⁺ and Eu3³⁺, are located at interlayer sites and connect the BSi1O₆⁵⁻ and Si2O₄⁴⁻ anion layers. Eu1³⁺ and Eu2³⁺ are coordinated by nine O atoms in tricapped trigonal prismatic geometry, the bond distances varying from 2.298 (8) to 2.69 (1) Å (mean value 2.531 Å) for Eu1³⁺ and from 2.292 (9) to 2.94 (1) Å (mean value 2.505 Å) for Eu2³⁺. Eu3³⁺ is surrounded by eight O atoms in a dodecahedral geometry, the bond distances varying from 2.301 (9) to 2.583 (9) Å (mean value 2.428 Å). The coordination polyhedra of Eu1³⁺, Eu2³⁺ and Eu3³⁺ share edges with each other to form a three-dimensional network structure (Fig. 2).

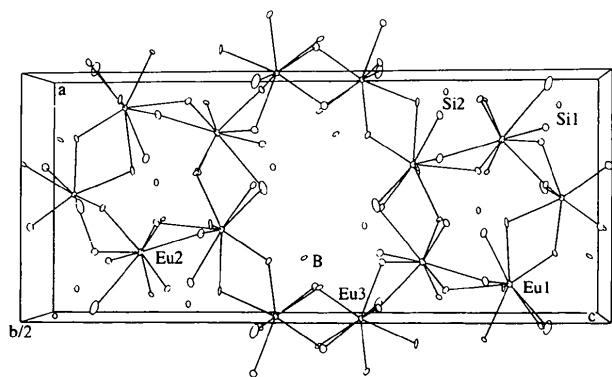


Fig. 2. A representation of the connection of the Eu—O polyhedra in half the unit cell. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

The single crystal was obtained by solid-state reaction and chemical vapour transport methods. The starting materials, Eu₂O₃ (> 99.9%), B₂O₃ (> 99.999%) and SiO₂ (AR), were mixed in the ratio 3:1:4, sealed in an evacuated quartz tube and heated at 1073 K for 10 h, then at 1273 K for 10 d. After cooling at a rate of 2 K h⁻¹ to 1073 K, cooling was continued to room temperature in air. Colourless needle-shaped single crystals were obtained.

Crystal data

Eu₃BSi₂O₁₀
M_r = 682.86
 Orthorhombic
Pbca
a = 9.673 (1) Å
b = 6.992 (3) Å
c = 22.867 (3) Å
V = 1546.6 (8) Å³
Z = 8
D_x = 5.87 Mg m⁻³
D_m not measured

Mo *K*α radiation
λ = 0.71073 Å
 Cell parameters from 25 reflections
θ = 14–17°
μ = 24.44 mm⁻¹
T = 293 K
 Needle
 0.40 × 0.05 × 0.01 mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω/*2θ* scans
 Absorption correction: *ψ* scans (North, Phillips & Mathews, 1968)
T_{min} = 0.358, *T_{max}* = 0.997
 5373 measured reflections
 5373 independent reflections

3286 observed reflections [*I* > 3.0σ(*I*)]
θ_{max} = 40.0°
h = 0 → 17
k = 0 → 12
l = -41 → 0
 3 standard reflections
 frequency: 166 min
 intensity decay: none

Refinement

Refinement on *F*
R = 0.0527
wR = 0.0608
S = 0.61
 3286 reflections
 146 parameters
w = 1/[σ(*F*)² + (0.020*F*)² + 1.0]

(*Δ*/σ)_{max} = 0.0004
Δρ_{max} = 6.94 e Å⁻³
Δρ_{min} = -1.56 e Å⁻³
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Eu1	0.13469 (6)	0.17862 (8)	0.33592 (2)	0.00635 (8)
Eu2	0.73177 (5)	-0.09239 (8)	0.31809 (2)	0.00595 (8)
Eu3	0.49044 (5)	0.13630 (8)	0.42790 (2)	0.00501 (7)
Si1	-0.1225 (3)	0.1423 (4)	0.4222 (1)	0.0052 (5)
Si2	0.4394 (3)	0.1863 (5)	0.2789 (1)	0.0057 (5)
B	0.244 (1)	0.339 (2)	0.4699 (5)	0.007 (2)
O1	0.4023 (8)	0.133 (1)	0.2105 (3)	0.006 (1)
O2	-0.2742 (9)	0.147 (2)	0.3903 (4)	0.012 (2)
O3	0.1076 (9)	0.206 (1)	0.2221 (4)	0.009 (1)
O4	0.3640 (9)	0.337 (2)	0.5063 (4)	0.012 (2)
O5	0.1259 (9)	0.419 (1)	0.4912 (4)	0.010 (1)
O6	-0.0473 (9)	0.326 (1)	0.3930 (4)	0.009 (1)
O7	0.3444 (9)	0.375 (1)	0.2894 (4)	0.010 (1)

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)

Acta Cryst. (1996). **C52**, 2387–2389

KGaAs₂O₇

Table 2. Selected geometric parameters (Å)

Eu1—O1 ⁱ	2.507 (8)	Eu3—O4	2.583 (9)
Eu1—O3	2.622 (9)	Eu3—O5 ⁱⁱⁱ	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 ⁱⁱⁱⁱ	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 ⁱⁱ	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 than 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: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *GCIF*, local program.

The work was supported by the National Natural Science Foundation of China.

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.

References

- Antic-Fidancev, E. (1994). *J. Phys. Condens. Matter*, **6**, 6857–6864.
- Enraf–Nonius (1977). *CAD-4 Operations Manual*. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Jsaryuk, V. I., Dzhurinskii, B. F., Zolin, V. F., Lysanova, G. V., Komova, M. G. & Savchenko, V. D. (1991). *Izv. Akad. Nauk. SSSR Neorg. Mater.* **27**, 68. (In Russian.)
- Liebau, F. (1985). *Structural Chemistry of Silicates, Structure, Bonding and Classification*. Berlin/New York: Springer-Verlag.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Ruimont, A. & Tarte, P. (1988). *J. Solid State Chem.* **75**, 244–250.
- Serhan, K., Taibi, M., Aride, J., Boukhari, A., Darriet, J. & Le Flem, G. (1994). *J. Solid State Chem.* **110**, 384–388.
- Voronkov, A. A. & Pyatenko, Y. A. (1967). *Sov. Phys. Crystallogr.* **12**, 214–221.

KUAN-JIUH LIN AND KWANG-HWA LIU

Institute of Chemistry, Academia Sinica, Taipei, Taiwan.

E-mail: kjlin@chem.sinica.edu.tw

(Received 20 September 1995; accepted 19 April 1996)

Abstract

Potassium gallium diarsenate(4–), KGaAs₂O₇, has been synthesized from a flux and structurally characterized by single-crystal X-ray diffraction. It is isostructural with KAlAs₂O₇ [Boughzala & Jouini (1995). *Acta Cryst.* **C51**, 179–181] and RbAlAs₂O₇ [Boughzala, Driss & Jouini (1993). *Acta Cryst.* **C49**, 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, GaAsO₄ (Goiffon, Jumas, Maurin & Philippot, 1986) and Ga₆(AsO₄)₆(C₂H₇N)₄(HF)₂(H₂O) (Chen, Li, Yang & Xu, 1989). In this paper, we describe the synthesis and crystal structure of a potassium gallium arsenate, KGaAs₂O₇.

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 KAlAs₂O₇ (Boughzala & Jouini, 1995). The framework is composed of GaO₆ octahedra joined through As₂O₇ diarsenate groups by corner sharing. The atom labeling of the asymmetric unit is shown in Fig. 2.

All of the GaO₆ octahedra are regular [Ga—O 1.936 (4)–2.002 (4) Å, O—Ga—O 87.4 (1)–93.0 (2)°]. The AsO₄ tetrahedra of the As₂O₇ 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 KAlAs₂O₇.

The two crystallographically distinct K1 and K2 cations in KGaAs₂O₇ lie on inversion centers. The coordination numbers of the K⁺ ions were determined on