Crystal structure of potassium gallium (monophosphate-hydrogenmonoborate-monophosphate), $\text{KGa}[\text{BP}_2\text{O}_7(\text{OH})_3]$

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Abstract

$\text{BGaH}_3\text{KO}_{10}\text{P}_2$, monoclinic, $C12/c1$ (No. 15), $a = 10.863(1) \text{ \AA}$, $b = 8.1623(7) \text{ \AA}$, $c = 9.305(1) \text{ \AA}$, $\beta = 116.587(4)^\circ$, $V = 737.8 \text{ \AA}^3$, $Z = 4$, $R_g(F) = 0.020$, $wR_{\text{ref}}(F^2) = 0.053$, $T = 295 \text{ K}$.

Source of material

$\text{KGa}[\text{BP}_2\text{O}_7(\text{OH})_3]$ was synthesized under mild hydrothermal conditions. The reactions were carried out with mixtures of Ga metal (0.139 g) dissolved in 1 ml of HCl (18%) with KCl (0.447 g), H_3BO_3 (0.371 g) and $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$ (0.817 g), (molar ratio of Ga:P:B = 2:6:10) in aqueous solution. The mixtures were sealed in glass tubes (after adding 1 ml H_2O to achieve a degree of filling of 30%) and subsequently heated at 408 K for 14 days. The starting materials are all of analytical-grade purity.

Discussion

Open framework borophosphates have become of interests in solid state studies for several years [1]. Our extensive studies on gallium and indium systems have lead to a number of new compounds [2,3]. The structure of the title compound is isotypic to

that of the aluminium [4], iron [5], gallium [2] and indium [3] analogues with sodium cations. The structure contains unbranched triple tetrahedral anions $[\text{BP}_2\text{O}_7(\text{OH})_3]^{4-}$, which are built up by two hydrogenphosphate and one dihydroborate tetrahedra sharing common corners. The condensation of the borophosphate anions with Ga-coordination octahedra $\text{GaO}_4(\text{OH})_2$ via common corners results in an overall three-dimensional framework which contains elliptical channels running along the [001] direction. The cross section of the channels is defined by eight-membered octahedral/tetrahedral rings (four Ga coordination octahedra, two phosphate- and two borate-groups). Potassium ions are distributed within the open channels. The Ga—O and Ga—OH bond distances range from 1.939 Å to 2.007 Å. The P—O and B—O bond distances in the oligomeric borophosphate groups correspond to respective values in the isotypic compounds [2–5].

Table 1. Data collection and handling.

Crystal:	colorless prism, size $0.05 \times 0.05 \times 0.20 \text{ mm}$
Wavelength:	Mo K_α radiation ($0.71073 \text{ \AA}$)
μ :	47.73 cm^{-1}
Diffractionmeter, scan mode:	Rigaku AFC7-CCD, 450 images, $\Delta\phi = 0.6^\circ$, $60-\omega$ scan, $\Delta\omega = 0.6^\circ$, $\chi = 90^\circ$
$2\theta_{\text{max}}$:	64.32°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3353, 1131
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1066
$N(\text{param})_{\text{refined}}$:	76
Programs:	SHELXL-97 [6], DIAMOND [7]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	x	y	z	U_{iso}
H(1)	8f	0.484(4)	0.333(4)	0.078(4)	0.03(1)
H(2)	4d	$-1/4$	$1/4$	0	0.05

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ga(1)	4c	1/4	1/4	0	0.0086(1)	0.0070(1)	0.0066(1)	−0.00004(9)	0.0036(1)	−0.00041(8)
K(1)	4e	0	0.36975(7)	1/4	0.0121(3)	0.0121(3)	0.0190(3)	0	0.0078(2)	0
P(1)	8f	0.23646(5)	0.56514(5)	0.18773(5)	0.0070(2)	0.0070(2)	0.0072(2)	−0.0001(1)	0.0037(2)	−0.0007(1)
B(1)	4e	1/2	0.4553(3)	1/4	0.008(1)	0.008(1)	0.009(1)	0	0.0030(9)	0
O(1)	8f	0.4370(1)	0.3542(2)	0.1044(2)	0.0084(6)	0.0142(6)	0.0107(5)	−0.0018(5)	0.0051(5)	−0.0041(5)
O(2)	8f	0.1795(1)	0.4007(2)	0.1064(2)	0.0104(6)	0.0095(5)	0.0104(5)	−0.0019(4)	0.0059(5)	−0.0039(4)
O(3)	8f	0.1037(1)	0.0644(2)	0.2409(2)	0.0071(6)	0.0107(6)	0.0173(6)	0.0002(4)	0.0054(5)	0.0034(5)
O(4)	8f	−0.1809(2)	0.2933(2)	−0.0672(2)	0.0167(7)	0.0127(6)	0.0127(6)	0.0022(5)	0.0061(5)	0.0046(5)
O(5)	8f	0.3042(1)	0.0931(2)	0.1780(2)	0.0130(6)	0.0113(6)	0.0097(5)	0.0023(5)	0.0077(5)	0.0035(4)

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