Crystal structure of rubidium gallium *catena*-[monohydrogen-monoborate-bis(monophosphate)] $RbGa[BP_2O_8(OH)]$, *from a twinned crystal*

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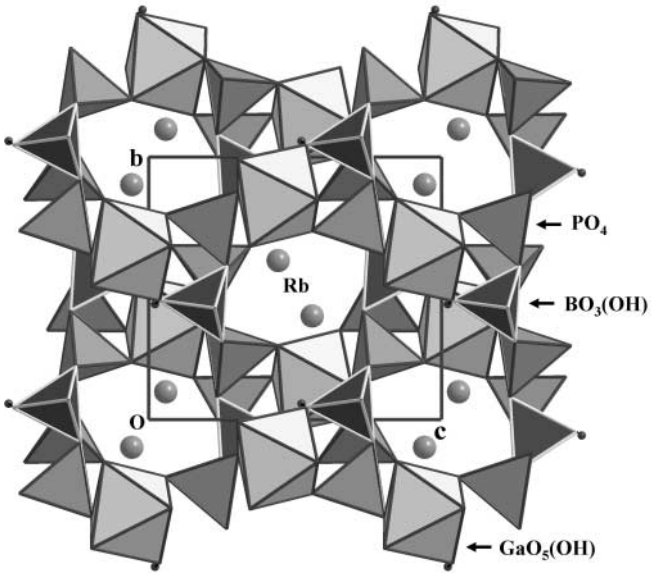

Abstract

$BGaHO_9P_2Rb$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.3207(8)$ Å, $b = 8.3686(7)$ Å, $c = 9.5371(9)$ Å, $\beta = 102.527(3)°$, $V = 726.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.050$, $wR_{ref}(F^2) = 0.147$, $T = 295$ K.

Source of material

The compound $RbGa[BP_2O_8(OH)]$ was synthesized under mild hydrothermal conditions. Reactions were carried out with mixtures of RbOH (1.538 g), $GaCl_3$ (0.35 g gallium metal dissolved in 2 ml 37% HCl), H_3BO_3 (0.309 g) and 3 ml 85% H_3PO_4 with molar ratio of Rb : Ga : B : P = 3 : 1 : 1 : 8. The mixture was filled in a 20 ml teflon-autoclave with a degree of filling of 50%. The autoclave was placed in an oven with subsequent heating at 413 K for 7 days. All starting materials were of analytical grade purity. The composition was confirmed by chemical analysis (ICP-OES) to Rb : Ga : B : P : H = 0.95 : 1 : 0.98 : 2.01 : 1.03.

Experimental details

Crystals from many different syntheses are always colorless and transparent, and have a form of regular prism. However, concave angles at certain edges of the prismatic crystals clearly indicate twinning. The twin characteristics are corroborated by oscillation photographs about the crystallographic axes of the monoclinic unit cell. Additional spots on the photograph about the *a* axis and a mirror plane normal to the *c* axis are caused by a second domain. The crystal used for CCD data collection and structure refinement was analysed applying two different orientation matrices but with volume contributions of 73.6 % (domain I) and 26.4 % (domain II), respectively. The twin components are related through a two-fold rotation around [001]. The data set contained a total of 6937 observed reflections, among which 3477 belong to domain I, 2557 to domain II, and 903 with contributions from both domains.

Discussion

In our systematic investigations on Ga-containing borophosphates, mild hydrothermal synthetic method has proved to be efficient in preparing new compounds with different structure types, such as $NaGa[BP_2O_7(OH)_3]$, $KGa[BP_2O_7(OH)_3]$ and $(NH_4)Ga[BP_2O_8(OH)]$ [1-3]. The title compound was also synthesized by a similar method.

The crystal structure of the title compound is isotypic to $CsFe[BP_2O_8(OH)]$ [4]. It is characterized by isolated $GaO_5(OH)$ octahedra sharing common O-atoms with five phosphate tetrahedra and a common OH-unit with a hydrogenborate group to form a three-dimensional framework structure. The anionic partial structure contains open-branched vierer-single chains $[BP_2O_8(OH)]^{4-}$, which are formed by alternating hydrogenborate and phosphate tetrahedra sharing common O-atoms. Rubidium cations are distributed within the open elliptical channels running along the *a* axis. Rubidium is coordinated by ten oxygen atoms with distances ranging from 2.939 Å to 3.253 Å. The Ga—O bond distances in the octahedron range from 1.915 Å to 1.970 Å, and the Ga—OH bond distance is 2.100 Å. The P—O bond distances range from 1.510 Å to 1.574 Å, and those of B—O range from 1.462 Å to 1.481 Å. Bond lengths and angles of hydrogenborate and phosphate tetrahedra within the anionic chains are in the same ranges as observed in other borophosphates [1-4].

Table 1. Data collection and handling.

Crystal:	colorless transparent prism, size 0.04 × 0.04 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	109.17 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7-CCD, 300 images, $\Delta\varphi = 0.8°$, 60°-ω scan, $\Delta\omega = 0.8°$, $\chi = 90°$
$2\theta_{max}$:	60.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5863, 5865
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5066
$N(param)_{refined}$:	129
Programs:	SHELXS-97 [7], SHELXL-97 [8], DIAMOND [9])

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.1028	0.0606	0.4785	0.05

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rb(1)	4*e*	0.30911(6)	0.39442(5)	0.44197(5)	0.0262(3)	0.0194(2)	0.0184(3)	–0.0046(2)	0.0040(2)	–0.0052(2)
P(1)	4*e*	0.4287(1)	0.9356(1)	0.2938(1)	0.0061(4)	0.0077(4)	0.0054(5)	–0.0007(3)	0.0011(4)	–0.0005(3)
P(2)	4*e*	0.0814(1)	0.7380(1)	0.2818(1)	0.0054(4)	0.0065(4)	0.0069(5)	–0.0010(3)	0.0003(3)	–0.0006(3)
Ga(1)	4*e*	0.29430(5)	0.84612(5)	0.56912(5)	0.0071(2)	0.0074(2)	0.0052(2)	–0.0005(1)	0.0010(2)	0.0003(1)
B(1)	4*e*	0.1659(5)	0.0566(5)	0.3042(5)	0.007(2)	0.007(2)	0.009(2)	–0.000(1)	–0.001(2)	0.000(1)
O(1)	4*e*	0.3133(3)	0.0756(3)	0.2745(3)	0.009(1)	0.007(1)	0.013(2)	0.0016(9)	0.002(1)	0.001(1)
O(2)	4*e*	0.9141(3)	0.7017(3)	0.2509(3)	0.006(1)	0.008(1)	0.016(2)	–0.0024(9)	0.001(1)	–0.003(1)
O(3)	4*e*	0.1431(3)	0.7188(3)	0.4409(3)	0.016(2)	0.013(1)	0.004(1)	–0.004(1)	–0.001(1)	0.001(1)
O(4)	4*e*	0.4209(3)	0.9901(3)	0.6949(3)	0.007(1)	0.013(1)	0.011(2)	–0.004(1)	0.002(1)	–0.002(1)
O(5)	4*e*	0.0888(3)	0.9151(3)	0.2334(3)	0.011(1)	0.009(1)	0.011(2)	–0.003(1)	–0.001(1)	0.001(1)
O(6)	4*e*	0.3966(3)	0.6596(3)	0.6543(3)	0.010(1)	0.012(1)	0.008(1)	0.003(1)	0.003(1)	0.004(1)
O(7)	4*e*	0.1547(3)	0.6275(3)	0.1930(4)	0.012(1)	0.011(1)	0.015(2)	0.001(1)	0.009(1)	–0.002(1)
O(8)	4*e*	0.4153(3)	0.8375(3)	0.4236(3)	0.010(1)	0.014(1)	0.009(2)	0.003(1)	0.006(1)	0.001(1)
O(9)	4*e*	0.1861(3)	0.0461(3)	0.4623(3)	0.013(1)	0.014(1)	0.007(2)	0.003(1)	0.003(1)	–0.000(1)

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