Crystal structure of rubidium indium (monophosphate-hydrogenmonoborate-monophosphate), RbIn[$BP_2O_8(OH)$]

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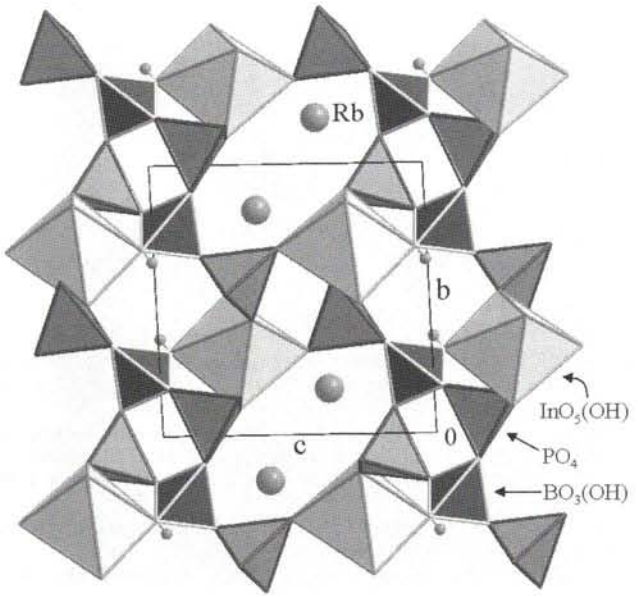

Abstract

$BHInO_9P_2Rb$, triclinic, $P\bar{1}$ (No. 2), $a = 5.3157(5)$ Å, $b = 8.3209(8)$ Å, $c = 8.4840(7)$ Å, $\alpha = 87.35(1)°$, $\beta = 80.23(1)°$, $\gamma = 86.78(1)°$, $V = 369.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.042$, $wR_{ref}(F^2) = 0.086$, $T = 295$ K.

Source of material

RbIn[$BP_2O_8(OH)$] was synthesized under mild hydrothermal conditions. The reactions were carried out with mixtures of $InCl_3$ (1.1058 g), H_3BO_3 (0.9277 g), RbOH (6.15 g) and 1.5ml H_3PO_4 (85%) with molar ratio Rb : In : B : P = 1 : 3 : 4. 1.0 ml HCl (37%) was added to adjust the pH value as 1.5. The mixture was filled in 10 ml teflon autoclaves (filling degree 60%) and heated at 443 K for 10 days under autogenous pressure. The chemical composition was confirmed by chemical analysis. IR spectroscopic investigations show the presence of OH groups.

Discussion

Although systematic investigations in borophosphates have only been carried out in recent years, a large number of new borophosphates has already been reported [1]. In our extensive studies on gallium and indium systems, several new compounds have been characterized recently [2–4].

The structure of the title compound is isotypic to that of the K–Fe [5], NH_4–In [3] and K–In [4] analogues. The anionic partial structure contains open branched 4-membered rings $[BP_2O_8(OH)]^{4-}$, which are formed by alternating hydrogenborate and phosphate tetrahedra sharing common corners with two additionally branching phosphate tetrahedra. The condensation of the borophosphate anions with In-coordination octahedra via common corners results in an overall three-dimensional framework which contains channels running along the *a* axis. The cross section of the channels is defined by eight-membered octahedral/tetrahedral rings (two In coordination octahedra, four phosphate- and two hydrogenborate-groups). Rubidium cations reside within the open channels. The In—O and In—OH bond distances range from 2.110 Å to 2.172 Å. Both, the borophosphate anions and the coordination octahedra are similar to its K–In– and NH_4–In analogues [3–4] by considering the bond lengths *d*(In—O), *d*(P—O) and *d*(B—O) as wel as the bond angles.

Table 1. Data collection and handling.

Crystal:	colorless platelet, size 0.014 × 0.04 × 0.07 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	102.17 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7-CCD, 550 images, $\Delta\phi = 0.6°$, $60-\omega$ scan, $\Delta\omega = 0.6°$, $\chi = 90°$
$2\theta_{max}$:	60.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4003, 1689
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1431
$N(param)_{refined}$:	131
Programs:	SHELXL-97 [6], DIAMOND[7]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	0.38(2)	0.64(2)	0.01(2)	0.05

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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}
In(1)	2*i*	0.2766(1)	0.68836(6)	0.29742(6)	0.0079(3)	0.0103(3)	0.0092(3)	–0.0006(2)	–0.0013(2)	–0.0007(2)
Rb(1)	2*i*	0.7225(1)	0.83045(9)	0.61678(9)	0.0125(4)	0.0184(4)	0.0198(4)	–0.0004(3)	–0.0026(3)	–0.0018(3)
P(1)	2*i*	0.2168(4)	0.5676(2)	0.6893(2)	0.008(1)	0.0080(8)	0.0080(9)	–0.0007(6)	–0.0002(7)	0.0000(7)
P(2)	2*i*	0.1940(3)	0.0821(2)	0.8074(2)	0.006(1)	0.0094(8)	0.0065(8)	–0.0004(6)	0.0009(7)	0.0004(7)
B(1)	2*i*	0.115(2)	0.770(1)	0.939(1)	0.008(4)	0.016(4)	0.009(4)	–0.002(3)	0.002(3)	0.004(3)
O(1)	2*i*	0.265(1)	0.6880(6)	0.5478(6)	0.013(3)	0.014(3)	0.007(2)	–0.004(2)	–0.002(2)	0.002(2)
O(2)	2*i*	0.528(1)	0.4829(6)	0.2560(6)	0.011(3)	0.012(2)	0.014(3)	0.005(2)	–0.004(2)	–0.006(2)
O(3)	2*i*	0.5608(9)	0.8604(6)	0.2877(6)	0.007(3)	0.010(2)	0.018(3)	–0.005(2)	0.001(2)	–0.001(2)
O(4)	2*i*	0.0275(9)	0.9027(6)	0.2839(6)	0.010(3)	0.010(2)	0.015(3)	0.001(2)	–0.004(2)	0.001(2)
O(5)	2*i*	0.074(1)	0.4256(6)	0.6526(6)	0.010(3)	0.012(2)	0.011(3)	–0.003(2)	0.000(2)	0.000(2)
O(6)	2*i*	0.282(1)	0.6887(7)	0.0422(6)	0.018(3)	0.017(3)	0.005(2)	0.008(2)	–0.002(2)	0.000(2)
O(7)	2*i*	0.8703(9)	0.8256(6)	0.0346(6)	0.008(3)	0.017(3)	0.007(2)	–0.001(2)	0.001(2)	–0.003(2)
O(8)	2*i*	0.0436(9)	0.6583(6)	0.8282(6)	0.011(3)	0.012(2)	0.009(2)	–0.001(2)	–0.001(2)	–0.002(2)
O(9)	2*i*	0.253(1)	0.9023(6)	0.8506(6)	0.010(3)	0.012(3)	0.012(3)	0.003(2)	0.002(2)	–0.001(2)

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