Crystal structure of caesium aluminum *catena*-[monohydrogen-monoborate-bis(monophosphate)], $CsAl[BP_2O_8(OH)]$

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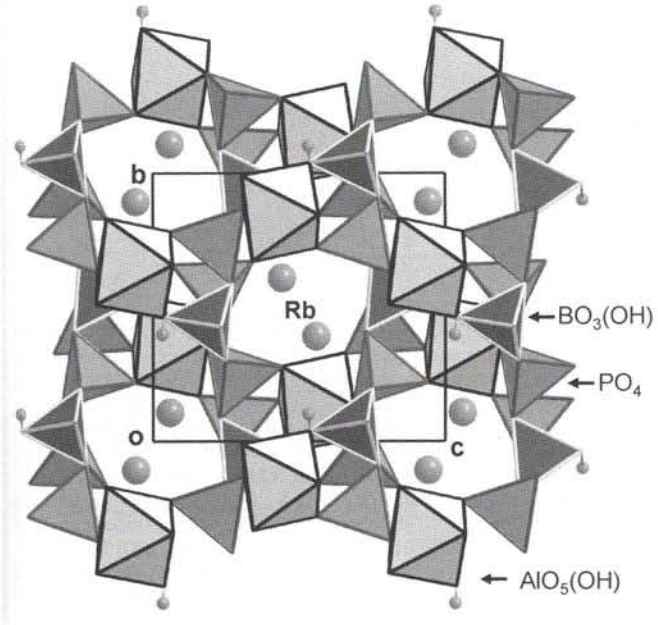

Abstract

$AlBCsHO_9P_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.208(2)$ Å, $b = 8.6957(9)$ Å, $c = 9.469(1)$ Å, $\beta = 104.189(6)°$, $V = 735.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.036$, $wR_{ref}(F^2) = 0.087$, $T = 295$ K.

Source of material

$CsAl[BP_2O_8(OH)]$ was synthesized under mild hydrothermal conditions. The reactions were carried out with mixture of CsOH (1.679 g), $Al(H_2PO_4)_3$ (3.179 g), H_3BO_3 (0.618 g) and 2 ml 85% H_3PO_4 with molar ratio of Cs : Al : B : P = 1 : 1 : 1 : 3.3. The mixture was filled in a teflon autoclave with about 20 ml in volume. The degree of filling was about 50%. The autoclave was placed in an oven with subsequent heating at 413 K for 7 days. The starting materials were all of analytical-grade purity.

Discussion

The variety of borophosphates in systems containing Ga and In [1-6], and the report on $NaAl[BP_2O_7(OH)_3]$ [7], intrigued our investigations in Al-containing borophosphates. The crystal structure of the title compound is isotypic to $CsFe[BP_2O_8(OH)]$, $(NH_4)Ga[BP_2O_8(OH)]$ and $RbAl[BP_2O_8(OH)]$ [8-10]. The crystal structure is characterized by isolated $AlO_5(OH)$ octahedra sharing common O corners with five phosphate tetrahedra and a common (OH) corner 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 corners. Caesium cations are distributed within the open elliptical channels running along the *a* axis. The Al—O and Al—OH bond distances range from 1.839Å to 2.031Å. Bond lengths and angles of hydrogenborate and phosphate tetrahedra within the anionic chains are in the same ranges as observed in other borophosphates [1-6].

Table 1. Data collection and handling.

Crystal:	colorless octahedra, size 0.07 × 0.07 × 0.07 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	56.21 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7-CCD, 300 images, $\Delta\phi = 0.8°$, $60-\omega$ scan, $\Delta\omega = 0.8°$, $\chi = 90°$
$2\theta_{max}$:	64.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6119, 2095
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1891
$N(param)_{refined}$:	128
Programs:	SHELXL-97 [11], DIAMOND [12]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.1093	0.0638	0.4915	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}
Cs(1)	4*e*	0.30445(4)	0.38395(4)	0.44598(4)	0.0199(2)	0.0161(2)	0.0157(2)	0.0001(1)	0.0034(1)	–0.0022(1)
P(1)	4*e*	0.4302(1)	0.9254(1)	0.3007(1)	0.0024(5)	0.0036(4)	0.0022(4)	–0.0003(3)	0.0004(4)	–0.0005(3)
P(2)	4*e*	0.9125(1)	0.2286(1)	0.2156(1)	0.0026(5)	0.0026(4)	0.0034(5)	0.0005(3)	0.0002(4)	0.0010(4)
Al(1)	4*e*	0.3003(2)	0.8469(2)	0.5734(1)	0.0034(6)	0.0036(5)	0.0034(6)	–0.0008(4)	0.0004(5)	0.0002(4)
B(1)	4*e*	0.1572(5)	0.0347(5)	0.3005(5)	0.006(2)	0.003(2)	0.007(2)	0.001(2)	0.003(2)	0.001(2)
O(1)	4*e*	0.3035(3)	0.0508(4)	0.2673(4)	0.003(1)	0.007(1)	0.009(1)	0.002(1)	0.002(1)	0.004(1)
O(2)	4*e*	0.0729(4)	0.8988(4)	0.2318(4)	0.009(2)	0.005(1)	0.007(1)	–0.002(1)	–0.000(1)	0.000(1)
O(3)	4*e*	0.4042(4)	0.8180(4)	0.1703(3)	0.007(2)	0.008(1)	0.005(1)	–0.003(1)	0.001(1)	–0.003(1)
O(4)	4*e*	0.0779(3)	0.1752(4)	0.2431(4)	0.004(2)	0.004(1)	0.011(2)	0.002(1)	0.002(1)	0.002(1)
O(5)	4*e*	0.8404(4)	0.2190(4)	0.0550(3)	0.007(2)	0.007(1)	0.005(1)	0.002(1)	–0.003(1)	0.001(1)
O(6)	4*e*	0.8350(4)	0.1325(4)	0.3081(4)	0.008(2)	0.008(1)	0.007(1)	–0.001(1)	0.005(1)	0.001(1)
O(7)	4*e*	0.5778(3)	0.0073(4)	0.3106(3)	0.004(1)	0.008(1)	0.006(1)	–0.004(1)	0.001(1)	–0.005(1)
O(8)	4*e*	0.4218(4)	0.8433(4)	0.4395(4)	0.008(2)	0.008(1)	0.007(1)	0.002(1)	0.005(1)	0.004(1)
O(9)	4*e*	0.1840(4)	0.0251(4)	0.4606(3)	0.010(2)	0.008(1)	0.004(1)	0.002(1)	0.003(1)	0.001(1)

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