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Crystal structure of rubidium scandium (monophosphate-hydrogen-monoborate-monophosphate), $RbSc[BP_2O_8(OH)]$

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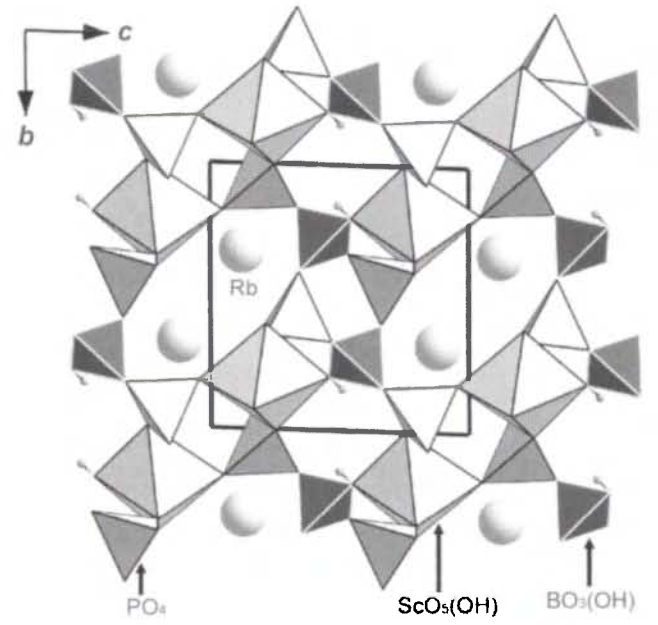

Abstract

BHO_9P_2RbSc, triclinic, $P\bar{1}$ (no. 2), a = 5.3296(2) Å, b = 8.3919(8) Å, c = 8.4319(5) Å, α = 87.27(1)°, β = 80.124(6)°, γ = 86.60(1)°, V = 370.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.024, $wR_{ref}(F^2)$ = 0.066, T = 293 K.

Source of material

Single crystals of $RbSc[BP_2O_8(OH)]$ used for the structure determination were isolated from a sample synthesized hydrothermally by using a mixture of 0.25 g Sc_2O_3 (99 %, ABCR), 1.009 g RbOH (99 %, Alfa Aesar), 0.6043 g H_3BO_3 (99.9 %, Alfa Aesar) and 2.8126 g phosphoric acid (85 %, Merck) in molar ratio Rb:Sc:B:P = 3:1:4:10. 1.0 ml HCl (37 %) was added to adjust the pH value to 1. The mixture was placed in a 10 ml Teflon-lined autoclave (filling degree 30 %), treated under autogenous pressure at 443 K for 10 days, cooled down to 333 K and kept there for 12 hours before cooling down to room temperature. The resulting product was filtered off, washed with water and dried in air. The chemical composition was confirmed by chemical analysis. IR spectra are consistent with the presence of B—OH groups.

Experimental details

The hydrogen atom was located in the Fourier difference map and O—H distance was fixed to 0.75 Å during the final refinement.

Discussion

Substantial efforts have been made during the past decades to synthesize novel open framework structures that might find potential applications as catalysts, ion-exchangers or molecular sieves [1]. Besides, a large number of borophosphates have been widely explored with various anionic partial structures [2-4]. During our systematic investigations in the systems M_2O–Sc_2O_3–B_2O_3–P_2O_5–H_2O (M = Li, Na, K, Rb, Cs) a new borophosphate, $RbSc[BP_2O_8(OH)]$, was synthesized.

The crystal structure of the title compound is isotypic to those of the K-Sc [5], K-Fe [6], NH_4-In [7], K-In [8] and Rb-In [9] analogues. The anionic partial structure comprises open branched 4-membered rings (a loop-branched hexamer) $[BP_2O_8(OH)]^{4-}$, 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 $ScO_5(OH)$ octahedra via common corners results in an overall three-dimensional framework which contains channels extending along [100]. The cross sections of the channels are defined by eight-membered rings consisting of two Sc coordination octahedra, four phosphate tetrahedra and two hydrogenborate-groups. Rubidium ions reside within the channels. The structure also contains six- and three-membered rings with different combinations of $ScO_5(OH)$ octahedra, PO_4 tetrahedra and $BO_3(OH)$ groups. The Sc—O and Sc—OH bond distances range from 2.05 Å to 2.15 Å. Bond lengths and angles of borate and phosphate tetrahedra within the anions are similar to those in related borophosphates [5-9].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.065 × 0.140 × 0.175 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	79.76 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC-7 & Mercury CCD, φ/ω
$2\theta_{max}$:	63.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6669, 2013
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1927
$N(param)_{refined}$:	131
Programs:	SHELXL-97 [10], DIAMOND [11]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.389(9)	0.147(6)	0.500(7)	0.08(2)

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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}
Rb(1)	2*i*	0.72182(5)	0.32589(3)	0.12116(3)	0.0104(1)	0.0162(2)	0.0174(1)	0.00006(9)	−0.00116(9)	−0.00075(9)
Sc(1)	2*i*	0.27970(8)	0.19187(5)	0.80025(5)	0.0053(2)	0.0063(2)	0.0065(2)	−0.0002(2)	−0.0008(1)	0.0005(1)
P(1)	2*i*	0.1920(1)	0.58201(7)	0.30461(7)	0.0047(2)	0.0053(3)	0.0065(3)	−0.0004(2)	0.0008(2)	0.0002(2)
P(2)	2*i*	0.2117(1)	0.06279(7)	0.19368(7)	0.0050(3)	0.0052(3)	0.0057(2)	−0.0004(2)	−0.0003(2)	−0.0002(2)
B(1)	2*i*	0.1165(5)	0.2739(3)	0.4387(3)	0.010(1)	0.006(1)	0.006(1)	0.0003(9)	0.0000(9)	−0.0004(8)
O(1)	2*i*	0.0283(3)	0.3981(2)	0.7891(2)	0.0067(7)	0.0083(8)	0.0121(8)	0.0003(6)	−0.0029(6)	−0.0007(6)
O(2)	2*i*	0.0458(3)	0.1585(2)	0.3325(2)	0.0088(8)	0.0098(9)	0.0098(8)	0.0008(6)	0.0013(6)	−0.0032(6)
O(3)	2*i*	0.0512(3)	0.9294(2)	0.1617(2)	0.0098(8)	0.0089(9)	0.0124(8)	−0.0039(6)	−0.0013(6)	−0.0011(6)
O(4)	2*i*	0.1238(3)	0.6658(2)	0.4675(2)	0.0079(8)	0.0132(9)	0.0084(8)	0.0017(7)	0.0005(6)	−0.0031(6)
O(5)	2*i*	0.2675(3)	0.1774(2)	0.0466(2)	0.0130(8)	0.0119(9)	0.0074(8)	−0.0041(7)	−0.0019(6)	0.0015(6)
O(6)	2*i*	0.2549(3)	0.4022(2)	0.3407(2)	0.0100(8)	0.0068(8)	0.0132(8)	0.0011(6)	0.0025(6)	0.0026(6)
O(7)	2*i*	0.2865(4)	0.1987(2)	0.5436(2)	0.0129(9)	0.015(1)	0.0074(8)	0.0057(7)	−0.0005(7)	0.0008(7)
O(8)	2*i*	0.4365(3)	0.6443(2)	0.2108(2)	0.0061(7)	0.0088(8)	0.0103(8)	−0.0016(6)	0.0017(6)	0.0005(6)
O(9)	2*i*	0.4621(3)	0.0036(2)	0.2474(2)	0.0078(8)	0.0102(9)	0.0150(8)	0.0024(6)	−0.0041(6)	−0.0015(6)

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