Crystal structure of ammonium indium (monophosphate-hydrogenmonoborate-monophosphate), $(\text{NH}_4)\text{In}[\text{BP}_2\text{O}_8(\text{OH})]$

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Abstract

$\text{BH}_5\text{InNO}_9\text{P}_2$, triclinic, $P\bar{1}$ (No. 2), $a = 5.2980(4)$ Å, $b = 8.4880(4)$ Å, $c = 8.3901(5)$ Å, $\alpha = 93.077(5)^\circ$, $\beta = 93.331(6)^\circ$, $\gamma = 80.634(5)^\circ$, $V = 371.3$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.038$, $wR_{\text{ref}}(F^2) = 0.091$, $T = 293$ K.

Source of material

$(\text{NH}_4)\text{In}[\text{BP}_2\text{O}_8(\text{OH})]$ was synthesized under mild hydrothermal conditions. The reactions were carried out with mixtures of In metal (0.057g) dissolved in 1ml of HCl (18%), $(\text{NH}_4)_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ (2.088g) and H_3BO_3 (0.310g) (molar ratio of In:B:P = 1:10:12) in aqueous solution. The mixtures were sealed in glass tubes (after adding 1ml H_2O to achieve a degree of filling of 30%) with subsequent heating at 403 K for 14 days. The starting materials were all of analytical grade.

Discussion

With the increasing interest in microporous materials the syntheses of compounds like borophosphates with open framework structures have drawn much attention during the past few years and show a rich crystal chemistry [1]. Systems including a p-block metal have not been widely explored up to now besides two compounds with Al and Ga [2,3] reported only recently and two phases with In in the present Issue [4,5]. The structure of the

title compound is isotypic to the K-In and K-Fe analogues [5,6] and is characterized by isolated $\text{InO}_5(\text{OH})$ octahedra sharing common O-corners with phosphate and common $\text{O}(\text{OH})$ -corner with hydrogenborate groups from the trinuclear oligomeric units $[\text{B}_2\text{P}_4\text{O}_{16}(\text{OH})_2]^{8-}$. The condensation of the borophosphate oligomers with In-coordination octahedra via common corners results in an overall three-dimensional framework which contains elliptical 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 borate-groups). Ammonium ions are distributed within the open channels. The In—O and In—OH bond distances range from 2.104 Å to 2.171 Å. Both the oligomeric borophosphate and the coordination octahedron of In are similar to its K-In-analogue [5] and the Na-In compound [4] by considering the bond distances In—O, P—O and B—O and bond angles.

Table 1. Data collection and handling.

Crystal:	colorless prism, size $0.1 \times 0.2 \times 0.3$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	36.48 cm^{-1}
Diffraction, scan mode:	Enraf Nonius CAD4, $\omega/2\theta$
$2\theta_{\text{max}}$:	67.9°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	6584, 3022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2743
$N(\text{param})_{\text{refined}}$:	128
Program:	SHELXL-97 [7]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	2i	0.4017	0.4683	1.3493	0.05
H(2)	2i	0.8349	0.1606	1.2094	0.05
H(3)	2i	0.8236	0.0224	1.1231	0.05
H(4)	2i	0.6678	0.1431	1.0773	0.05
H(5)	2i	0.6338	0.062	1.2057	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
In	2i	0.72294(4)	0.20588(3)	0.68316(3)	0.0093(1)	0.0096(1)	0.0100(1)	−0.00154(7)	0.00029(7)	−0.00047(6)
P(1)	2i	0.2180(2)	0.1890(1)	1.4297(1)	0.0092(3)	0.0089(3)	0.0093(3)	−0.0014(3)	0.0004(2)	0.0004(2)
P(2)	2i	0.1954(2)	0.3102(1)	0.9154(1)	0.0082(3)	0.0101(3)	0.0088(3)	0.0004(3)	0.0001(2)	−0.0001(2)
O(1)	2i	0.0419(5)	0.3223(3)	1.3348(3)	0.011(1)	0.013(1)	0.017(1)	−0.0016(8)	−0.0005(8)	0.0037(8)
O(2)	2i	0.1304(5)	0.4684(3)	0.8259(3)	0.013(1)	0.013(1)	0.019(1)	0.0000(9)	−0.0038(9)	0.0050(9)
O(3)	2i	−0.0258(5)	0.2150(3)	0.8961(3)	0.011(1)	0.016(1)	0.012(1)	−0.0033(8)	0.0015(8)	0.0015(8)
O(4)	2i	0.0723(5)	0.1542(3)	1.5693(3)	0.015(1)	0.017(1)	0.012(1)	−0.0028(9)	0.0042(8)	0.0000(8)
O(5)	2i	0.4684(5)	0.2512(3)	0.4811(3)	0.013(1)	0.020(1)	0.016(1)	−0.0066(9)	−0.0050(9)	0.0031(9)
O(6)	2i	0.2532(5)	0.3505(3)	1.0948(3)	0.015(1)	0.021(1)	0.009(1)	0.0023(9)	−0.0001(8)	−0.0014(9)
O(7)	2i	0.2757(6)	0.0433(3)	1.3132(3)	0.022(1)	0.011(1)	0.015(1)	−0.0026(9)	0.0073(9)	−0.0024(8)
O(8)	2i	0.2811(5)	0.5388(3)	1.3115(3)	0.018(1)	0.009(1)	0.019(1)	−0.0009(9)	−0.007(1)	−0.0003(9)
O(9)	2i	0.4431(5)	0.2164(3)	0.8581(3)	0.010(1)	0.014(1)	0.013(1)	0.0024(8)	0.0023(8)	−0.0008(8)
N	2i	0.7193(6)	0.1148(4)	1.1607(4)	0.015(1)	0.022(2)	0.020(1)	−0.004(1)	0.001(1)	0.004(1)
B	2i	0.1120(7)	0.4369(4)	1.2278(4)	0.010(1)	0.008(1)	0.013(1)	−0.001(1)	0.000(1)	0.000(1)

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