Crystal structure of ammonium [monozinco-monoboro-diphosphate], $NH_4[ZnBP_2O_8]$

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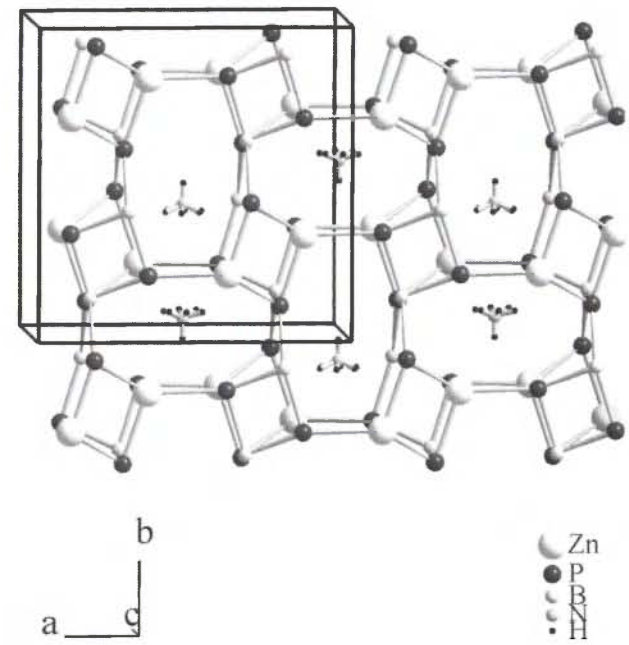

Abstract

$BH_4NO_8P_2Zn$, monoclinic, $C12/c1$ (No. 15), a = 12.848(3) Å, b = 12.896(3) Å, c = 8.519(1) Å, β = 91.024(3)°, V = 1411.2 Å^3, Z = 8, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.073, T = 293 K.

Source of material

$NH_4[ZnBP_2O_8]$ was prepared by solvothermal reaction (with ethylene glycol) of 0.700 g ZnO, 0.298 g B_2O_3, 2.264 g $(NH_4)_2HPO_4$ and 5 ml 85 % H_3PO_4 (molar ratio: 2 : 1 : 4, gel, pH = 1.5) in a teflon autoclave at 438 K.

Experimental details

Refinement with restraints: N—H distances to 0.86 Å, H–N–H angle to approximately the tetrahedral angle and U_{iso}(H) = 1.2×U_{iso}(N).

Discussion

The crystal structure of $NH_4[ZnBP_2O_8]$ (anhydrous borophosphate, B:P = 0.5 [1]) contains an anionic framework $[ZnBP_2O_8]^-$, which is formed by alternating zincate, borate and phosphate tetrahedra sharing common corners. The ammonium compound is an isotype of $K[ZnBP_2O_8]$ [2]. The protons H1, H2, H4, H6, H7 and H8 of the ammonium cations are disordered over two different positions. Bond lenghts and angles within the anionic partial structure are consistent with related borophosphates (see [1, 2] and refs. herein). A second polymorph of $NH_4[ZnBP_2O_8]$ with a gismondine type framework is also known [2].

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.05 × 0.125 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	39.51 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7 CCD, Φ-scan 360°, 60°-ω-scan at χ = 90°, 0.5° steps with 40 s exposure time per step, detector distance: 35 mm, 2θ offset: 10°
$2\theta_{max}$:	59.68°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8548, 1649
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1580
$N(param)_{refined}$:	135
Programs:	SHELXS-97 [3], SHELXS-97 [4], DIAMOND [5]

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

Atom	Site		x	y	z	U_{iso}
N(1)	4*e*		0	0.0346(3)	1/4	0.0200(9)
H(1)	8*f*	0.5	0.052(5)	0.057(2)	0.21(1)	0.024
H(2)	8*f*	0.5	–0.01(1)	0.056(2)	0.344(4)	0.024
H(3)	4*e*		0	–0.032(2)	1/4	0.024
H(4)	8*f*	0.5	–0.056(6)	0.054(2)	0.20(1)	0.024
N(2)	4*e*		0	0.3981(3)	1/4	0.025(1)
H(5)	4*e*		0	0.463(2)	1/4	0.03
H(6)	8*f*	0.5	–0.050(7)	0.373(2)	0.19(1)	0.03
H(7)	8*f*	0.5	0.058(6)	0.374(2)	0.21(2)	0.03
H(8)	8*f*	0.5	–0.01(1)	0.372(2)	0.343(4)	0.03

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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}
Zn(1)	8*f*	0.12436(3)	0.19147(3)	0.56717(4)	0.0121(2)	0.0130(2)	0.0114(2)	–0.0007(2)	–0.0003(1)	–0.0007(1)
B(1)	8*f*	0.3072(3)	0.1082(3)	0.0743(4)	0.012(2)	0.010(2)	0.010(2)	0.003(1)	0.000(1)	–0.001(1)
P(1)	8*f*	0.30153(6)	0.08082(6)	0.40616(9)	0.0111(4)	0.0096(4)	0.0086(4)	0.0006(3)	–0.0006(3)	0.0010(3)
P(2)	8*f*	0.37061(6)	0.31392(6)	0.06278(9)	0.0088(4)	0.0092(3)	0.0100(4)	–0.0003(3)	–0.0016(3)	0.0010(3)
O(1)	8*f*	0.1384(2)	0.3348(2)	0.5069(3)	0.023(1)	0.013(1)	0.020(1)	–0.0034(9)	–0.007(1)	0.0016(9)
O(2)	8*f*	0.1597(2)	0.4752(2)	0.0308(2)	0.017(1)	0.012(1)	0.016(1)	–0.0023(9)	0.0034(9)	–0.0050(9)
O(3)	8*f*	0.1856(2)	0.0941(2)	0.4205(3)	0.013(1)	0.020(1)	0.014(1)	0.0019(9)	–0.0009(9)	–0.0039(9)
O(4)	8*f*	0.1963(2)	0.1300(2)	0.0655(2)	0.012(1)	0.019(1)	0.012(1)	0.0029(9)	–0.0008(9)	0.0025(9)
O(5)	8*f*	0.3281(2)	0.3362(2)	0.2218(3)	0.022(1)	0.023(1)	0.009(1)	0.005(1)	0.0001(9)	–0.0013(9)
O(6)	8*f*	0.3359(2)	0.0767(2)	0.2342(3)	0.016(1)	0.031(1)	0.009(1)	0.008(1)	0.0017(9)	0.005(1)
O(7)	8*f*	0.3708(2)	0.1964(2)	0.0245(3)	0.017(1)	0.011(1)	0.022(1)	–0.0012(9)	0.007(1)	–0.0009(9)
O(8)	8*f*	0.4802(2)	0.3528(2)	0.0392(3)	0.008(1)	0.017(1)	0.026(1)	–0.0020(9)	–0.0037(9)	0.003(1)

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