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#### Key indicators

Powder X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{O}-\text{B}) = 0.009$  Å  
 $R$  factor = 0.146  
 $wR$  factor = 0.069  
Data-to-parameter ratio = 11.0

For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## The layered gallium borophosphate $\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$ refined from X-ray powder data

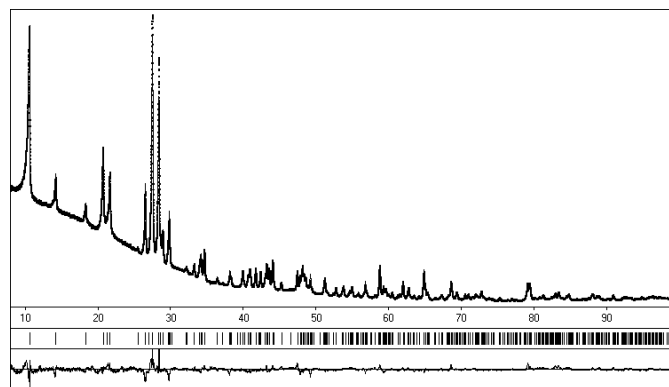
The crystal structure of  $\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$ , obtained by mild hydrothermal synthesis, has been refined from X-ray powder diffraction data using the Rietveld method. It is isotypic with the iron borophosphate  $\text{Fe}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  and has a two-dimensional layered structure.

#### Comment

In the past few years, borophosphates have attracted much attention from chemists and materials scientists as potential microporous materials. In our systematic investigations on gallium borophosphates, two alkali metal gallium borophosphates, *viz.*  $\text{NaGa}[\text{BP}_2\text{O}_7(\text{OH})_3]$  (Huang *et al.*, 2001) and  $\text{KGa}[\text{BP}_2\text{O}_7(\text{OH})_3]$  (Li *et al.*, 2002), and the ammonium gallium borophosphate  $(\text{NH}_4)\text{Ga}[\text{BP}_2\text{O}_8(\text{OH})]$  (Mi *et al.*, 2002) have been investigated. Gallium borophosphates without additional alkali metal or ammonium ions have not been reported up to now, although three compounds  $\{\text{Fe}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  (Boy *et al.*, 1998),  $\text{Al}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5(\text{H}_2\text{O})]$  (Kniep *et al.*, 2002) and  $\text{Cr}_2(\text{BP}_3\text{O}_{12})$  (Mi *et al.*, 2000) $\}$  were structurally characterized for the crystal-chemically related trivalent Fe, Al and Cr cations. We report here the first gallium borophosphate without an additional cation. The crystal structure of the title compound is isotypic with  $\text{Fe}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  and is closely related to  $\text{Al}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5(\text{H}_2\text{O})]$ .

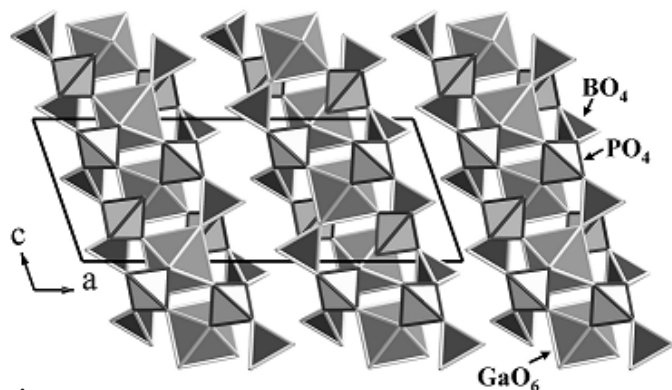
$\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  has a two-dimensional layered structure and the crystals clearly show micaceous aggregate thin plates under the microscope. Because of its micaceous character, it is difficult to find suitable crystals for single-crystal X-ray diffraction. The crystal structure was therefore refined from X-ray powder diffraction data (Fig. 1). The Ga–O bond

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**Figure 1**

A Rietveld refinement plot for  $\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$ , showing the observed and difference profiles. The reflection positions are shown above the difference profile.



**Figure 2**  
The crystal structure of  $\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  in polyhedral representation, viewed down the  $b$  axis.

distances range from 1.840 (3) to 2.035 (4) Å in the  $\text{GaO}_6$  octahedra, and those of P—O from 1.532 (4) to 1.594 (4) Å in the  $\text{PO}_4$  tetrahedra. Although the average B—O bond distance of the title compound (1.450 Å) is reasonable in comparison with its Fe and Al analogues (Fe 1.471 Å and Al 1.470 Å), the individual bond lengths show considerable scatter, caused by the limitation of the X-ray powder data refinement and the lower precision of the determined positions of the light atoms.

The crystal structure of  $\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  is characterized by sandwich-type ‘tetrahedra–octahedra–tetrahedra’ layers parallel to (100) (Fig. 2) which are connected by hydrogen bonds (Table 2). Atom O1 is part of the OH group attached to the phosphate tetrahedron, whereas atoms O5 and O6 belong to OH functions of the borate group. The  $\text{PO}_4$  and  $\text{BO}_4$  tetrahedra share one O-atom corner with each other, forming an infinite zigzag single chain of  $[-\text{BO}_4-\text{PO}_4-\text{BO}_4-\text{PO}_4-]$  units along the  $c$  axis. One set of  $\text{GaO}_6$  octahedra connect with two infinite single chains by sharing O-atom corners. These two chains are connected again through another two sets of  $\text{GaO}_6$  octahedra to two other chains shifted by a period of  $a/2$ . This construction leads to the puckered shape of the layer parallel to (100). Each  $\text{GaO}_6$  octahedron shares O-atom corners with four  $\text{PO}_4$  and two  $\text{BO}_4$  tetrahedra in the layer; each  $\text{PO}_4$  tetrahedron shares O-atom corners with two  $\text{GaO}_6$  octahedra and two  $\text{BO}_4$  tetrahedra, while each  $\text{BO}_4$  shares vertices with one  $\text{GaO}_6$  and two  $\text{PO}_4$  besides the terminal OH group.

## Experimental

$\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  was synthesized under mild hydrothermal conditions. The reaction was carried out with mixtures of  $\text{GaCl}_3$  (0.523 g gallium metal dissolved in 2.5 ml 37% HCl), 1.237 g  $\text{H}_3\text{BO}_3$  and 2.5 ml 85%  $\text{H}_3\text{PO}_4$  in a Ga:B:P molar ratio of 1.5:4:7. The container was about 50% full of solution. The autoclave was placed in an oven with subsequent heating at 443 K for 7 d. All starting materials were of analytical grade and used without further purification. The formula was confirmed by chemical analysis (ICP) with a Ga:B:P:H ratio of 1:1.88:1.97:5.36. TG/DTA analysis showed an endothermic peak at 636 K associated with about 12.1% weight loss. This is consistent with the theoretical value of 12.9% for loss of two and a half water mol-

ecules. It was confirmed that the elimination of all water molecules occurs at nearly the same temperature.

## Crystal data

$\text{Ga}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$   
 $M_r = 350.32$   
Monoclinic,  $C2/c$   
 $a = 17.6404$  (3) Å  
 $b = 6.70735$  (8) Å  
 $c = 6.99525$  (8) Å  
 $\beta = 109.157$  (1)°  
 $V = 781.85$  (2) Å<sup>3</sup>  
 $Z = 4$

$D_x = 2.976$  Mg m<sup>-3</sup>  
Cu  $K\alpha_1$  radiation  
 $\mu = 9.56$  mm<sup>-1</sup>  
 $T = 295$  K  
White  
Specimen shape: Plate  
25 × 25 × 1 mm  
Particle morphology: thin sheet

## Data collection

Stoe STADI-P diffractometer  
Debye–Scherrer scans  
Specimen mounting: packed powder pellet  
Specimen mounted in transmission mode  
Absorption correction: none  
396 measured reflections

397 independent reflections  
 $\theta_{\text{max}} = 49.5^\circ$   
 $h = 0 \rightarrow 17$   
 $k = 0 \rightarrow 6$   
 $l = -6 \rightarrow 6$   
 $2\theta_{\text{min}} = 3$ ,  $2\theta_{\text{max}} = 100^\circ$   
Increment in  $2\theta = 0.005^\circ$

## Refinement

Refinement on  $I_{\text{net}}$   
 $R_p = 0.146$   
 $R_{\text{wp}} = 0.069$   
 $R_{\text{exp}} = 0.025$   
397 reflections  
36 parameters

H-atom parameters not refined  
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
Preferred orientation was refined (CSD; Pecharsky *et al.*, 1987),  
 $I_{\text{corr}} = I_{\text{obs}}/[1 + (\tau^2 - 1)\sin^2\varphi]^{1/2}$   
axis [100],  $\tau = 1.869$  (1)

**Table 1**

Selected geometric parameters (Å, °).

Ga1—O2 <sup>i</sup>	1.840 (3)	P1—O2 <sup>vii</sup>	1.545 (4)
Ga1—O2 <sup>ii</sup>	1.840 (3)	P1—O3 <sup>viii</sup>	1.560 (5)
Ga1—O1	1.978 (4)	P1—O4 <sup>ix</sup>	1.593 (4)
Ga1—O1 <sup>iii</sup>	1.978 (4)	B1—O5 <sup>v</sup>	1.372 (10)
Ga1—O6 <sup>iv</sup>	2.035 (4)	B1—O3	1.386 (9)
Ga1—O6 <sup>v</sup>	2.035 (4)	B1—O4 <sup>v</sup>	1.482 (8)
P1—O1 <sup>vi</sup>	1.532 (4)	B1—O6 <sup>v</sup>	1.559 (9)
O1 <sup>vi</sup> —P1—O2 <sup>vii</sup>	116.2 (2)	O5 <sup>v</sup> —B1—O3	121.6 (6)
O1 <sup>vi</sup> —P1—O3 <sup>viii</sup>	109.8 (2)	O5 <sup>v</sup> —B1—O4 <sup>v</sup>	104.6 (6)
O1 <sup>vi</sup> —P1—O4 <sup>ix</sup>	104.2 (2)	O5 <sup>v</sup> —B1—O6 <sup>v</sup>	103.7 (5)
O2 <sup>vii</sup> —P1—O3 <sup>viii</sup>	109.5 (2)	O3—B1—O4 <sup>v</sup>	114.6 (6)
O2 <sup>vii</sup> —P1—O4 <sup>ix</sup>	109.0 (2)	O3—B1—O6 <sup>v</sup>	109.1 (6)
O3 <sup>viii</sup> —P1—O4 <sup>ix</sup>	107.8 (2)	O4 <sup>v</sup> —B1—O6 <sup>v</sup>	100.8 (5)

Symmetry codes: (i)  $1 - x, y, \frac{1}{2} - z$ ; (ii)  $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$ ; (iii)  $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$ ; (iv)  $1 - x, y, \frac{3}{2} - z$ ; (v)  $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$ ; (vi)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (vii)  $x, 1 - y, \frac{1}{2} + z$ ; (viii)  $1 - x, 1 - y, 1 - z$ ; (ix)  $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$ .

**Table 2**

Hydrogen-bonding  $D \cdots A$  distances (Å).

O1 <sup>vi</sup> ...O6 <sup>iv</sup>	2.783 (5)	O5 <sup>v</sup> ...O5 <sup>viii</sup>	2.447 (5)
O1 <sup>vi</sup> ...O6 <sup>v</sup>	2.892 (6)	O5 <sup>v</sup> ...O5 <sup>iv</sup>	2.704 (6)

Symmetry codes: (iv)  $1 - x, y, \frac{3}{2} - z$ ; (v)  $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$ ; (viii)  $1 - x, 1 - y, 1 - z$ .

The crystal structure was refined with the atomic coordinates of the isotopic iron compound  $\text{Fe}[\text{B}_2\text{P}_2\text{O}_7(\text{OH})_5]$  (Boy *et al.*, 1998) as starting parameters. All atoms were refined with isotropic displacement parameters.

Data collection: *WinXPOW* (Stoe & Cie, 1999); cell refinement: *CSD* (Akselrud *et al.*, 1989); data reduction: *CSD*; program(s) used to refine structure: *CSD*; molecular graphics: *DIAMOND* (Brandenburg, 1996–2001).

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