High-pressure synthesis and crystal structure of HP-Al$_2$B$_3$O$_7$(OH)

Abstract: Orthorhombic HP-Al$_2$B$_3$O$_7$(OH) was synthesized in a Walker-type multianvil apparatus under high-pressure/high-temperature conditions of 12.4 GPa and 1200 °C, respectively. Its structure is isotypic to that of Ga$_2$B$_3$O$_7$(OH) and has been determined via single-crystal X-ray diffraction at room temperature. HP-Al$_2$B$_3$O$_7$(OH) crystallizes in the space group Cmce (Z = 8) with the lattice parameters $a = 10.3124(4)$, $b = 7.3313(3)$, $c = 10.4801(5)$ Å, and $V = 792.33(6)$ Å$^3$. The compound has also been characterized by IR and Raman spectroscopy.

Keywords: aluminum borate; crystal structure; high-pressure synthesis; vibrational spectroscopy.

1 Introduction

Regarding the system Al–B–O–H, the literature research yields five compounds so far. Besides the well-known mineral Jeremejevite (Al$_6$(BO$_3$)$_5$(F,OH)$_3$) [1], our working group recently prepared the compound Al$_5$B$_{12}$O$_{25}$(OH) which represents a new member of the high-pressure structure type M$_5$B$_{12}$O$_{25}$(OH) (M = Al, Ga, In, Ga/In) [2, 3]. Jianhua Lin et al. who are working on hydrothermally synthesized zeolite-like borate structures, published the remaining three compounds in the system Al–B–O–H.

In detail, this research working group found three microporous aluminum borates, which they named PKU-1 [4], PKU-2 [5], and PKU-6 [6] (PKU stands for Peking University). PKU-1 with the sum formula HAl$_5$B$_{12}$O$_{25}$(OH)$_6$ was published in 2003. In this compound all Al atoms are octahedrally coordinated building up a porous structure with 10- and 18-membered ring windows. PKU-2 (Al$_5$B$_{12}$O$_{29}$(OH)$_{17}$nH$_2$O) is even more porous featuring 24-membered rings around channels of edge-sharing AlO$_6$ octahedra. PKU-6 (Al$_5$B$_{12}$O$_{29}$(OH) – designated as HAl$_5$B$_{12}$O$_{29}$ in reference [6]) was published in 2007 and shares the sum formula and point group with our title compound but shows a completely different structure. Infinite chains of square-pyramidally coordinated Al atoms are interlinked by three corner-sharing trigonal planar BO$_3$ groups. Thus, in the hydrothermally synthesized PKU-6, the Al atoms are pentacoordinated and the B atoms show a three-fold coordination. In our title compound HP-Al$_2$B$_3$O$_7$(OH), the coordination numbers of the aluminum and boron atoms are increased by one as one would expect for a high-pressure phase due to the pressure-coordination rule in solid state chemistry. However, the condensed structure of HP-Al$_2$B$_3$O$_7$(OH) is not unprecedented as it has already been described in 2015 for the gallium containing isotype Ga$_2$B$_3$O$_7$(OH) [7].

The present report contains a detailed description of the synthesis and crystal structure of our title compound as well as vibrational spectroscopic data of HP-Al$_2$B$_3$O$_7$(OH) single crystals.

2 Experimental section

2.1 Synthesis

The starting materials Al(NO$_3$)$_3$·9H$_2$O (>98%, Sigma-Aldrich) and H$_2$BO$_3$ (99.5% Carl Roth) were mixed in a stoichiometric ratio of 2:3, ground in an agate mortar and encapsulated in platinum foil (0.027 mm, 99.9%, Chem-PUR). The platinum capsule was then placed into a crucible with a lid, both made of a-BN (Henz Boron Nitride Products AG) and centered in a “14/8” assembly which includes, amongst others, a graphite furnace for resistance heating. Inside a uniaxially compressed Walker-type...
module, six steel wedges and eight tungsten carbide cubes surround the octahedral assembly to generate a quasi-hydrostatic pressure on the sample. A detailed description of the experimental setup is given in the literature [8–10]. HP-Al\(_2\)B\(_3\)O\(_7\)(OH) did also form as a byproduct in syntheses of volatile parts in aluminum nitrate nonahydrate. The required pressure and temperature were adjusted within 6 h and 10 min, respectively. The heating power was maintained for 10 min and then gradually lowered to \(T = 800 \, ^\circ\text{C}\) within 30 min. Finally, the sample was quenched to room temperature and then gradually lowered to a lower pressure of 12.0 GPa, or a lower pressure of 12.0 GPa. Compared to our starting mixture, the product quantity was obviously diminished, which we explain with the great amount of volatile parts in aluminum nitrate nonahydrate. HP-Al\(_2\)B\(_3\)O\(_7\)(OH) did also form as a byproduct in syntheses performed with Al\(_2\)O\(_3\) (99.9%, Alfa Aesar) instead of Al(NO\(_3\))\(_3\)·9H\(_2\)O, slightly varying ratios of Al to B, higher temperatures up to 1400 °C, or a lower pressure of 12.0 GPa.

2.2 Crystal structure determination by X-ray diffraction

The reaction product was analyzed with a STOE Stadi P powder diffractometer equipped with a Mythen 1 K detector (Dectris, Switzerland). The measurement was carried out with Ge(111)-monochromatized MoK\(_\alpha\) radiation (\(\lambda = 0.7093 \, \text{Å}\)) in transmission geometry across a 2\(\theta\) range of 2.0–50.0°. Figure 1 shows a comparison of the experimental powder pattern with a simulation derived from single-crystal structure data. HP-Al\(_2\)B\(_3\)O\(_7\)(OH) clearly constitutes the main phase, but nevertheless the experimental powder pattern shows additional reflections of at least one byproduct.

The colorless single crystals of HP-Al\(_2\)B\(_3\)O\(_7\)(OH) were measured with a Bruker D8 Quest diffractometer equipped with a Photon 100 CMOS detector. For structure solution and parameter refinement, the software tools SHELXS-2013/1 [11, 12] and SHELXL-2013/4 [13] implemented in the program WinGX-2018.1 [14] were used. According to the systematic reflection conditions, HP-Al\(_2\)B\(_3\)O\(_7\)(OH) was solved and refined in the space group \(\text{Cmce}\) (no. 64) and afterwards standardized with the routine STRUCTURE TIDY [15] implemented in PLATON [16] (version 170613). Except for the proton, all atoms could be refined anisotropically. Details of the data acquisition can be found in the synoptical Table 1. Table 2 contains the positional parameters and Wyckoff positions and Table 3 the displacement parameters of all atoms in HP-Al\(_2\)B\(_3\)O\(_7\)(OH).

![Figure 1](image-url)    

**Figure 1:** Experimental powder pattern (MoK\(_\alpha\)) of the optimized synthesis leading to HP-Al\(_2\)B\(_3\)O\(_7\)(OH) (top) compared to a simulation of the powder pattern of HP-Al\(_2\)B\(_3\)O\(_7\)(OH) based on single-crystal data (bottom). The asterisk-marked reflections in the experimental powder pattern refer to an unidentified byproduct.

### Table 1: Single-crystal data and structure refinement of HP-Al\(_2\)B\(_3\)O\(_7\)(OH).

| Parameter                  | Value                  |
|----------------------------|------------------------|
| Empirical formula          | Al\(_2\)B\(_3\)O\(_7\)(OH) |
| Molar mass, g mol\(^{-1}\) | 215.40                 |
| Crystal system             | orthorhombic           |
| Space group                | \(\text{Cmce}\) (no. 64) |
| Single-crystal diffractometer | Bruker D8 Quest Kappa |
| Radiation/wavelength \(\lambda\), pm | MoK\(_\alpha\)/71.07 |
| a, Å                       | 10.3124(4)             |
| b, Å                       | 7.3313(3)              |
| c, Å                       | 10.4801(5)             |
| \(V\), Å\(^3\)             | 792.33(6)              |
| Formula units per cell \(Z\) | 8                      |
| Calculated density, g cm\(^{-3}\) | 3.61                  |
| Crystal size, mm\(^3\)     | 0.030 × 0.050 × 0.090  |
| Temperature, K              | 183(2)                 |
| Absorption coefficient, mm\(^{-1}\) | 0.7                  |
| \(f(000)\), e               | 848                    |
| \(\theta\) range, deg      | 3.89–39.44             |
| Range in \(hkl\)           | ±18; ±13; ±18          |
| Refl. total/independent    | 8079/1236              |
| \(R\)\(_{int}\)/\(R\)\(_e\) | 0.0324/0.0226         |
| Refl. with \(I > 2 \sigma(I)\) | 1095                |
| Data/ref. parameters       | 1236/69                |
| Absorption correction      | multiscan              |
| Final \(R\)\(_1\)/\(wR\)\(_2\) (all data) | 0.0198/0.0469       |
| Final \(R\)\(_1\)/\(wR\)\(_2\) (all data) | 0.0250/0.0497       |
| Goodness-of-fit on \(F^2\) | 0.921                  |
| Largest diff. peak/hole, e Å\(^{-1}\) | 0.38/−0.34          |
Table 2: Wyckoff positions, atomic coordinates, isotropic $U_{iso}$ or equivalent isotropic displacement parameters $U_{eq}$ (Å$^2$) for HP-Al$_2$B$_3$O$_7$(OH). $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor (standard deviations in parentheses).

| Atom | Wyckoff position | $x$     | $y$     | $z$     | $U_{eq}$ ($U_{iso}$ for H4) |
|------|-----------------|---------|---------|---------|-----------------------------|
| Al1  | 8f              | 1/2     | 0.7885(3) | 0.8807(2) | 0.00190(6) |
| Al2  | 8d              | 0.85164(2) | 0           | 0          | 0.00208(6) |
| B1   | 16g             | 0.62480(6) | 1.14999(9) | 0.87176(6) | 0.00290(9) |
| B2   | 8e              | 3/4     | 0.8957(2)  | 3/4         | 0.0030(2)  |
| O1   | 16g             | 0.62447(4) | 1.24742(6) | 0.98924(4) | 0.00291(7) |
| O2   | 16g             | 0.63173(4) | 0.78036(6) | 0.73973(4) | 0.00280(7) |
| O3   | 16g             | 0.73359(4) | 1.01421(6) | 0.86569(4) | 0.00263(7) |
| O4   | 8f              | 1/2     | 0.53723(8) | 0.88783(6) | 0.00289(9) |
| O5   | 8f              | 1/2     | 1.04199(8) | 0.86111(6) | 0.00277(9) |
| H4   | 8f              | 1/2     | 0.468(3)   | 0.812(3)   | 0.065(8)   |

Table 3: Anisotropic displacement parameters $U_{ij}$ (Å$^2$) of HP-Al$_2$B$_3$O$_7$(OH) (standard deviations in parentheses).

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|------|----------|----------|----------|----------|----------|----------|
| Al1  | 0.0023(2) | 0.0016(2) | 0.0019(2) | 0        | 0        | 0.00032(7) |
| Al2  | 0.0023(2) | 0.0019(2) | 0.0020(2) | 0        | 0        | -0.00020(7) |
| B1   | 0.0029(2) | 0.0030(2) | 0.0029(2) | -0.0003(2) | -0.0002(2) | 0.0001(2) |
| B2   | 0.0032(3) | 0.0026(3) | 0.0031(3) | 0        | 0.0000(2) | 0        |
| O1   | 0.0035(2) | 0.0029(2) | 0.0024(2) | -0.0003(2) | 0.0002(2) | -0.0007(2) |
| O2   | 0.0027(2) | 0.0029(2) | 0.0028(2) | -0.0007(2) | 0.0003(2) | -0.0008(2) |
| O3   | 0.0027(2) | 0.0028(2) | 0.0024(2) | 0.0008(2) | -0.0002(2) | -0.0006(2) |
| O4   | 0.0035(2) | 0.0028(2) | 0.0024(2) | 0        | 0        | -0.0003(2) |
| O5   | 0.0020(2) | 0.0023(2) | 0.0040(2) | 0        | 0        | -0.0001(2) |

CSD-2027675 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

2.3 Vibrational spectroscopy

The transmission FT-IR spectrum of an HP-Al$_2$B$_3$O$_7$(OH) single crystal was measured in the spectral range of 600–4000 cm$^{-1}$ with a Vertex 70 FT-IR spectrometer (spectral resolution 4 cm$^{-1}$) equipped with a KBr beam splitter, a liquid nitrogen cooled MCT (Mercury Cadmium Telluride) detector and a Hyperion 3000 microscope (Bruker). 320 scans of the sample were acquired using a Globar (silicon carbide) rod as mid-IR source and a 15× IR objective as focus. During the measurement, the sample was positioned on a BaF$_2$ window. Atmospheric influences were corrected with the software OPUS 6.5 [17].

A single-crystal Raman spectroscopic analysis was performed using a HORIBA JOBIN-YVON LabRam-HR800 spectrometer with a focal length of 800 mm. The spectrum was recorded unpolarized at ambient conditions. The analyzed area was excited using a (green) Nd-YAG laser with an excitation wavelength of 532 nm. Scattered light was dispersed by an optical grating with 1800 grooves per mm resulting in a spectral resolution of 1.92 cm$^{-1}$, and collected with a cooled Andor$^\text{TM}$ CCD detector. An Olympus 50× objective, a confocal pinhole of 1000 µm and a slit of 100 µm was used for the measurement to maximize the intensity at a lateral resolution of ~5 µm$^2$. Before the analyses, the spectral position of the Raman mode of a Si standard wafer was measured against the position of the Rayleigh line, resulting in the expected Raman shift of 520.7 cm$^{-1}$. The spectrum was obtained in multi-window acquisition mode provided by the LABSPEC (version 5.93.20) [18] software package in the frequency range of 200–1500 cm$^{-1}$ and represents the average of two single measurements with an acquisition time of 250 s.
each. Background subtraction and the determination of band position were performed with the same software. The background was subtracted using an 8th order line-function. Band positions were obtained by fitting a Gaussian–Lorentzian function to the individual bands.

3 Results and discussion

3.1 Crystal structure

HP-Al₂B₃O₇(OH) crystallizes in the orthorhombic space group Cmce (no. 64) with eight formula units (Z = 8) and the lattice parameters \( a = 10.3124(4) \), \( b = 7.3313(3) \), \( c = 10.4801(5) \text{Å} \), and \( V = 792.33(6) \text{Å}^3 \). Details of the single-crystal data and structure refinement can be found in Table 1. Similar to the isotypic compound Ga₂B₃O₇(OH), HP-Al₂B₃O₇(OH) consists of two fundamental building blocks (BB1 and BB2). BB1 represents a cluster of four edge-sharing AlO₆ octahedra (Figure 2a), and BB2 is composed of 12 corner-sharing BO₄ tetrahedra forming a flexed unit of two sechser rings [19] that are interlinked by two vierer rings (Figure 2b). As depicted in Figure 3, each BB1 is encircled by two BB2s. The flexed BB2 units are interconnected forming endless zig-zag chains along the crystallographic \( b \) axis. Between these zig-zag chains, the BB1 clusters fill the space in alternating orientations in relation to \( c \) (visualized in Figure 4). All oxygen atoms belong to both building blocks except O₄, which connects to three Al atoms and the proton in HP-Al₂B₃O₇(OH). While the proton position was geometrically constructed in Ga₂B₃O₇(OH), it was possible to freely refine it in the aluminum compound. The proton is located in the middle of a BO₄ sechser ring at a distance of 0.94(3) Å to O₄ and forms hydrogen bonds to two O2 atoms and to O5. Figure 5 shows the hydrogen bonding situation in HP-Al₂B₃O₇(OH). As the proton needs space, it provokes the connecting O4 atom to a position farther away resulting in diminished O–Al–O angles of 76.53(3)° for O₄–Al₂–O₄ and 78.13(2)° for O₁–Al₂–O₄ instead of 90° required for a regular octahedron. All Al–O distances in the range between 1.8439(6) and 2.0086(5) Å and the average values of 1.90 and 1.92 Å are in accordance with data from the literature [20–22]. Likewise, the B–O distances (\( \bar{0} = 1.49 \text{Å} \) and tetrahedral angles (\( \bar{0} = 109.5^\circ \) are as expected [23]. All interatomic distances and angles are given in Tables 4–6. To support the crystal structure solution, the Madelung part of lattice energy (MAPLE value) [24, 25] of HP-Al₂B₃O₇(OH) was calculated and compared to the stoichiometric sum of the compounds \( \alpha \text{-Al}_2\text{O}_3 \), HP-B₂O₃, and \( \text{H}_3\text{BO}_3 \). The results showed only a minimal deviation of 0.15% (see Table 7). The bond valence sums (BVS) for HP-Al₂B₃O₇(OH) were calculated with the bond-length/bond-strength concept [26] and the charge distribution was derived by using the charge distribution in solids (CHARDI) concept [27]. The results are listed in Table 8. Both calculations led to reasonable values with only one noticeable BVS deviation for O₄ being the donor of the hydrogen bond.

3.2 Vibrational spectroscopy

For the isotypic compound Ga₂B₃O₇(OH) [7], the IR and Raman bands were assigned based on DFT calculations and therefore provide a good basis for the interpretation of the spectra of HP-Al₂B₃O₇(OH). The FT-IR spectrum of an HP-Al₂B₃O₇(OH) single crystal is shown in Figure 6. In the fingerprint region up to \( \sim 1200 \text{ cm}^{-1} \), at the lower wavenumbers presumably O–Al–O bending as well as Al–O stretching vibrations contribute to the spectral pattern, followed mainly by O–B–O bending and B–O stretching vibrations. As in Ga₂B₃O₇(OH), the O–H stretching vibrations give a distinct signal at about 3500 cm⁻¹. Unlike the Ga containing analog, the single

![Figure 2: Building blocks in HP-Al₂B₃O₇(OH). a) BB1: a cluster of four edge-sharing AlO₆ octahedra; b) BB2: an interlinked ring-formation of 12 corner-sharing BO₄ tetrahedra.](image-url)
Figure 3: Two BB2 building blocks consisting of BO₄ tetrahedra fully enclose one BB1 AlO₁₆ cluster.

Figure 4: Structural buildup of HP-Al₂B₃O₇(OH): Alternating AlO₁₆ clusters (BB1) are connected through endless chains of BO₄ tetrahedra along c. For reasons of clarity, the protons and the interlinking BO₄ tetrahedra forming the vicer rings in BB2 are omitted.

Figure 5: Hydrogen bonding situation in HP-Al₂B₃O₇(OH).

Table 4: Interatomic distances (Å) in HP-Al₂B₃O₇(OH) (standard deviations in parentheses).

|        | O4  | B1–  | O5  | B1–  | O1  | 1.4234(7) |
|--------|-----|------|-----|------|-----|------------|
| Al1–   | 1.8439(6) | 1.8696(6) | 1.8900(5) 2× | 2.0086(5) 2× | 1.5017(8) | 1.5112(7) |
| Ø Al1–O| 1.92 | Ø B1–O | 1.49 | 1.49 |
| Al2–   | 1.8726(5) 2× | 1.8715(5) 2× | 1.9486(4) 2× | 1.90 |
| Ø Al2–O| 1.90 | Ø B2–O | 1.49 | 1.49 |

Table 5: Interatomic angles (deg) in HP-Al₂B₃O₇(OH) (standard deviations in parentheses).

|        | O4–Al1–O1 | O4–Al2–O4 | O1–Al2–O4 | O1–Al1–O1 | O1–Al2–O1 | O1–Al1–O1 | O1–Al2–O1 | O1–Al1–O1 | O1–Al2–O1 | O1–Al1–O1 | O1–Al2–O1 |
|--------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|
| Ø Al1–O| 89.9       | 89.7       | 89.9       | 89.9       | 89.9       | 89.9       | 89.9       | 89.9       | 89.9       | 89.9       | 89.9       |
| Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      | Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      | Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      |
| Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      | Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      | Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      |
| Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      | Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      | Ø O–Al1–O₁₆₀ | 172.2      | Ø O–Al2–O₁₆₀ | 166.5      |
| Ø O–B1–O | 109.5      | Ø O–B1–O   | 109.5      | Ø O–B1–O   | 109.5      | Ø O–B1–O   | 109.5      | Ø O–B1–O   | 109.5      | Ø O–B1–O   | 109.5      |

Table 6: Hydrogen bonds (Å, deg) in HP-Al₂B₃O₇(OH) (standard deviations in parentheses).

|        | d(O–H) | d(H–A) | d(O–A) | \(\angle\) (O–H–A) |
|--------|--------|--------|--------|-----------------|
| O4–H₄–O₂ | 0.94(3) | 2.01(2) | 2.6793(7) | 127(2) |
| O4–H₄–O₅ | 0.94(3) | 1.90(3) | 2.6091(8) | 131(2) |

Table 7: Comparison of the calculated MAPLE value (Madelung Part of Lattice Energy) of HP-Al₂B₃O₇(OH) and the MAPLE value received from \(\alpha\)-Al₂O₃ [28], HP-B₂O₃ [29], and H₂BO₃ [30] according to the reaction equation: \(3 \text{Al}_2\text{O}_3 + 4 \text{B}_2\text{O}_3 + 3 \text{H}_2\text{BO}_3 \rightarrow 3 \text{HP-Al}_2\text{B}_3\text{O}_7\)(OH).

|                    | kJ mol\(^{-1}\) |
|--------------------|-----------------|
| Calculated MAPLE value for HP-Al₂B₃O₇(OH) | 161077 |
| Calculated MAPLE value based on \(\alpha\)-Al₂O₃, HP-B₂O₃, and H₂BO₃ | 161316 |
| Difference, %       | 0.15            |
The Raman spectrum is presented in Figure 7 and shows only the frequency range between 200 and 1500 cm$^{-1}$ due to strong luminescence at higher Raman shifts that becomes even more pronounced when exciting with a red (633 nm) laser, so that OH-related vibrations (expected between 3000 and 4000 cm$^{-1}$) are not displayed. The DFT calculations for the isotypic compound Ga$_2$B$_3$O$_7$(OH) also allow assignments for the aluminum analog. The Al$^{3+}$ = Ga$^{3+}$ exchange mainly results in higher vibrational frequencies and consequently increasing Raman shifts of Al-related bands if compared to the respective Ga-related counterparts. Bands in the 250–700 cm$^{-1}$ frequency range can be mainly assigned to vibrations of the AlO$_6$ units. The Al–O–H, Al–O–Al bending vibrations are expected to appear in a wide range between 250 and 1150 cm$^{-1}$, while the stretching vibrations for the BO$_4$ tetrahedra appear in the 800–1150 cm$^{-1}$ region.

4 Conclusion

The present report presents a detailed synthesis protocol and the determination and discussion of the crystal structure of the new aluminum borate HP-Al$_2$B$_3$O$_7$(OH), which is isotypic to Ga$_2$B$_3$O$_7$(OH). While the proton position in the gallium compound had to be calculated geometrically, it was possible to locate it in the electron density map and refine it via difference Fourier analysis of the aluminum analog. IR data additionally confirm the presence of the hydroxyl group. Attempts to synthesize the isotypic indium compound have not been successful yet.

Acknowledgements: We thank Ass.-Prof. Dr. Klaus Wurst for the recording of the single-crystal data, Freia Ruegenberg for the IR spectrometric data, and Univ.-Prof. Dr. Roland Stalder for granting us access to the single-crystal IR spectrometer.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

| Table 8: Charge distribution in HP-Al$_2$B$_3$O$_7$(OH), calculated with the bond-length/bond-strength (V) and CHARDI (Q) concept. |
| --- |
| V | Q |
| Al1 | 2.96 | 2.98 |
| Al2 | 3.10 | 2.94 |
| B1 | 2.93 | 3.01 |
| B2 | 2.90 | 3.09 |
| O1 | −1.94 | −2.04 |
| O2 | −1.84 | −1.99 |
| O3 | −1.97 | −2.11 |
| O4 | −2.34 | −1.90 |
| O5 | −1.97 | −2.04 |
| H4 | 1.01 | 0.98 |

![Figure 6: IR spectrum of an HP-Al$_2$B$_3$O$_7$(OH) single crystal in the range of 600–4000 cm$^{-1}$.](image6)

![Figure 7: Raman spectrum of an HP-Al$_2$B$_3$O$_7$(OH) single crystal in the range of 200–1500 cm$^{-1}$. The following band positions can be assigned to the HP-Al$_2$B$_3$O$_7$(OH) structure (cm$^{-1}$): 251, 266, 277, 303, 316, 321, 339, 350, 364, 412, 429, 438, 492, 501, 539, 582, 607, 616, 650, 674, 683, 702, 720, 749, 772, 788, 817, 833, 871, 899, 969, 1023, 1059, 1104, 1136, 1098.](image7)
Research funding: None declared.
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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