Synthesis and crystal structure of a novel ternary oxoborate, PbBiBO$_4$

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Abstract

A novel ternary borate oxide, lead bismuth boron tetraoxide, PbBiBO$_4$, has been prepared by solid-state reaction at temperature below 800 °C. The single-crystal X-ray structural analysis showed that PbBiBO$_4$ crystallizes in the monoclinic space group P2$_1$/n with $a = 7.473(1)$ Å, $b = 7.517(1)$ Å, $c = 7.609(1)$ Å, $\beta = 91.48(1)$, $Z = 4$. It represents a new structure type in which distorted BiO$_6^{9-}$ octahedra are connected to each other in corner- and edge-sharing manner to form two-dimensional $_\infty^2$[BiO$_4$]$^{5-}$ layers that are bridged by B atoms of BO$_3$ triangles giving rise to a three-dimensional $_\infty^3$[BiBO$_4$]$^{2-}$ framework, with channels parallel to the [0 1 0] direction accommodating the pyramidally coordinated Pb^{2+} cations.
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Keywords: PbBiBO$_4$; Borate; Synthesis; Crystal structure

1. Introduction

Borates have attracted considerable interest because they have important practical applications in second harmonic generation (SHG). For example, β-BaB$_2$O$_4$, LiB$_3$O$_5$, CsB$_3$O$_5$, and YCa$_4$(BO$_3$)$_3$O are all well-known nonlinear optical (NLO) crystals [1]. The binary Bi$_2$O$_3$–B$_2$O$_3$ phase diagram has been investigated by Levin and McDaniel [2], and at least five compounds have been proposed and structurally characterized including Bi$_{24}$B$_2$O$_{39}$ [3], Bi$_4$B$_2$O$_9$ [4], Bi$_3$B$_5$O$_{12}$ [5], BiB$_3$O$_6$ [6], and Bi$_2$B$_8$O$_{15}$ [7]. Among them, Bi$_4$B$_2$O$_9$ was reported to have high double-refraction [8], Bi$_3$B$_5$O$_{12}$ displays stimulated Raman scattering and luminescence properties [9,10], and BiB$_3$O$_6$ is the most extensively studied because it was found to have potential value as a NLO material, displaying extraordinarily large SHG effect [11–13]. In contrast, the complex borates incorporating bismuth together with other metal elements are relatively less explored. Only four ternary (quaternary) compounds, i.e., Cu$_5$Bi$_2$B$_4$O$_{12}$ [14], BaBiBO$_4$ [15], Bi$_2$Zn B$_2$O$_7$, and CaBiGaB$_2$O$_7$ [16] have been recently characterized in detail. In the ternary system of PbO–Bi$_2$O$_3$–B$_2$O$_3$, a

phase "Pb$_4$Bi$_3$B$_7$O$_{19}$" was previously proposed by Shuster et al., who predicted this compound to crystallize in the orthorhombic non-centrosymmetric space group P222$_1$ with lattice constants $a = 15.76(7)$ Å, $b = 7.47(3)$ Å, $c = 10.61(5)$ Å based on the powder X-ray data, but the crystal structure has not been solved [17]. In the course of our systematic investigation of novel borate NLO materials, we have failed in preparing Pb$_4$Bi$_3$B$_7$O$_{19}$ phase, but obtained single crystals of a novel ternary borate, PbBiBO$_4$ instead. Our X-ray structural analysis has established that the newly prepared PbBiBO$_4$ crystallizes in a new unique structure type never observed for any of the known borates, and the cell dimensions are also different from those of "Pb$_4$Bi$_3$B$_7$O$_{19}$" reported previously. Herein we describe its synthesis and crystal structure.

2. Experimental

2.1. Sample preparation and general characterization

The title compound was synthesized by employing a conventional solid-state reaction method. All reagents were of analytical grade. A powder mixture of 5.292 g PbO, 4.144 g Bi$_2$O$_3$, and 2.566 g H$_3$BO$_3$ (the PbO/Bi$_2$O$_3$/B$_2$O$_3$ molar ratio = 8:3:7) was transferred to a Pt crucible. The

sample was gradually heated to 800 °C, where it was kept for two days, then cooled down to 570 °C at a rate of 5 °C/h, and further to 523 °C at 0.5 °C/h, followed by cooling to room temperature at a rate of 20 °C/h. The colorless, prismatic crystals of $PbBiBO_4$ with dimensions up to $0.4 \times 0.5 \times 1.0\, mm^3$ were embedded in a lead borate matrix. Several small crystals could be isolated mechanically from the reaction product for the further characterization by single-crystal X-ray diffraction measurements. Energy-dispersive X-ray analyses in a scanning electron microscope have revealed the presence of heavy elements of lead and bismuth with an approximate atomic ratio of 1:1 in the crystals. Subsequently, direct reaction of a stoichiometric mixture of PbO, Bi_2O_3, and B_2O_3 at 450 °C for two weeks with an intermediate grinding yielded a single-phase polycrystalline sample of $PbBiBO_4$ that was confirmed by powder X-ray analysis using the monochromatized Cu Kα radiation of a Bruker D8 ADVANCE diffractometer. The compound appears to be relatively stable in air and water, but soluble in hot diluted HNO_3 solution.

2.2. Structure determination

Single-crystal X-ray intensity data were collected at room temperature (298 K) on an automated Rigaku AFC7R four-circle diffractometer using monochromatized Mo Kα radiation. Cell dimensions were obtained from a least-square refinement with 25 automatically-centered reflections in the range $40.24° \leqslant 2\theta \leqslant 44.88°$. Three standard reflections were re-measured after every 150 reflections. No decay was observed except the statistic fluctuation in a range of $\pm 1.27\%$. The raw intensity data were corrected for Lorentz and polarization effects, and for absorption by empirical method based on ψ-scan data.

The crystal structure was solved by a direct method and refined in SHELX-97 system [18] by full-matrix least-squares methods on F_o^2. After introduction of anisotropic displacement parameters for all atoms the refinement of 64 parameters with 1499 observed reflections $[I \geqslant 2\sigma(I)]$ resulted in the residuals of $R1/wR2 = 0.0459/0.0944$. The final difference electron density maps were featureless, with the highest electron density of $3.97\, e\, Å^{-3}$ at a position that is very close to the heavy atomic site. Details of crystal parameters, data collection and structure refinements are given in Table 1 and the atomic coordinates and the equivalent isotropic displacement parameters are summarized in Table 2. Listings of the anisotropic displacement parameters and the structure factors are available from the authors.

3. Results and discussion

$PbBiBO_4$ represents a new structure type (Pearson symbol $mP28$) and the crystal structure has three-dimensional (3D) character, which can be considered as being built of two-dimensional (2D) sheets in the following way (see Figs. 1 and 2): within the (1 0 1) plane of the

monoclinic unit cell, two *inversion-center* related BiO_6^{9-} octahedra are linked together by sharing one edge (O1…O1) to form a $Bi_2O_{10}^{14-}$ bioctahedral dimer. Each $Bi_2O_{10}^{14-}$ dimer is connected to four others in a centered arrangement *via* common O4 atoms to generate a corrugated 2D $_{\infty}^{2}[BiO_4]^{5-}$ layer. Applying the *inversion-center* operations to this layer produces the neighboring equivalent layers along the [1 0 1] direction (see Fig. 1). These layers are further bridged by B atoms of BO_3 groups, resulting in the formation of a 3D $_{\infty}^{3}[BiBO_4]^{2-}$ framework. Because of the existence of 2_1 screw axes, two opposite orientations are found for the BO_3 groups which bridge octahedra both within a single layer and between adjacent layers, thus affording eight-edge channels that extend in the [0 1 0] direction. Two rows of Pb^{2+} cations reside in the channels to balance charge and also interact with the framework *via* Pb–O bonds to strengthen the structure.

Each Pb^{2+} ion is strongly bonded to three O atoms at distances of 2.306(7)–2.329(7) Å and also weakly bonded to three more oxygens at distances of 2.793(8)–3.002(10) Å (Table 3). Taking all these six bonds into account, the concept of bond valence [19] allows us to calculate a valence sum equal to 2.10 for Pb which proves that the long bonds indeed participate in the metal coordination scheme. The PbO_6 polyhedron is extremely distorted with all of six oxygen neighbors located approximately on the same side of the Pb atom, leaving room for the $6s^2$ lone pair of Pb^{2+} in the opposite direction (Fig. 3). This configuration is very common for Pb^{2+}, as found in $Pb_4Te_6M_{10}O_{41}$ (M = Nb^{5+} or Ta^{5+}) [20].

There is one crystallographically independent bismuth atom, which adopts a strongly distorted octahedral configuration, with the *trans* octahedral angles being 145.1(3)–170.4(2)° and the *cis* angles in the range 74.8(3)–135.29(8)° (Table 3), deviating significantly from the ideal values of 180° and 90°, respectively. The bismuth atom is displaced out of the center of a idealized BiO_6

Table 1
Crystallographic data for $PbBiBO_4$

Formula	$PbBiBO_4$
Formula weight	490.98
Space group	$P2_1/n$ (*No.14*)
a (Å)	7.473(1)
b (Å)	7.517(1)
c (Å)	7.609(1)
β	91.48(1)°
V (Å^3), Z	427.3(1), 4
d_{calc} (g/cm^3)	7.632
λMo Kα (Å)	0.71073
μ (mm^{-1})	80.355
$2\theta_{max}$	69.92°
Unique reflection	1871
Observed $[I \geqslant 2\sigma(I)]$	1499
No. of variables	64
GOF on F_o^2	1.037
$R1/wR2$ $[I \geqslant 2\sigma(I)]$	0.0459/0.0944
$R1/wR2$ (all data)	0.0607/0.0999

Table 2

Atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$) for PbBiBO$_4$

Atoms	X	Y	Z	U$_{eq}$
Pb	0.18062(5)	0.39025(5)	0.68163(4)	0.01561(10)
Bi	0.33771(5)	0.50009(4)	1.15935(4)	0.01179(10)
B	0.0150(16)	0.2429(15)	1.0119(13)	0.0122(18)
O1	0.1016(12)	0.1043(8)	0.6022(10)	0.0167(14)
O2	0.4755(11)	0.2971(11)	0.6886(10)	0.0222(17)
O3	0.1873(11)	0.2873(11)	0.9705(9)	0.0231(17)
O4	−0.1190(11)	0.2360(14)	0.8884(11)	0.0279(19)

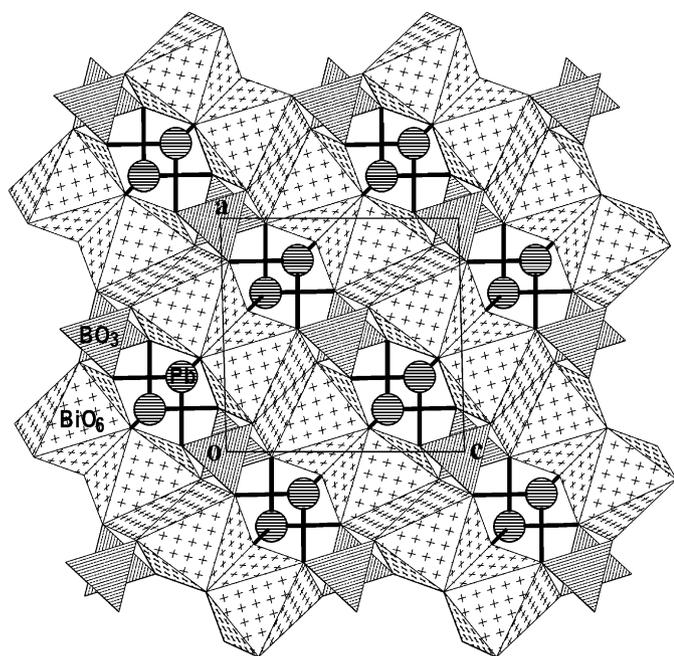*Note*: U$_{eq}$ is defined as one third of the trace of the orthogonalized U tensor.

Fig. 1. The crystal structure of PbBiBO$_4$ projected along the [0 1 0] direction, where the circles filled by parallel lines are Pb atoms, the octahedra filled by crosses are BiO$_6$ units, and the triangles filled by parallel dashed lines are BO$_3$ units. Only three short Pb-O bonds (2.306(7)–2.329(7) $\mathring{A}$) are drawn with solid lines for clarity.

Fig. 2. View of the $^2_\infty$[BiO$_4$]$^{5-}$ layer along the [1 0 1] direction, where the circles filled by crosses are Bi atoms and the open circles are O atoms.

octahedron approximately along a C_2-axis of the octahedron by about 0.318 $\mathring{A}$, as a consequence, the Bi–O bond lengths are divided into three sets, with two short distances (Bi–O1 = 2.177(8), Bi–O1 = 2.197(7) $\mathring{A}$) corresponding to bonds to a pair of equatorial oxygen atoms that form the common edge of a Bi$_2$O$_{10}^{14-}$ dimer, two intermediate distances (Bi–O2 = 2.351(8), Bi–O3 = 2.409(8) $\mathring{A}$) associated with bonds to two axial oxygen atoms, and two longer separations (Bi–O4 = 2.503(8), Bi–O4 = 2.590(9) $\mathring{A}$) involved in the interactions with the two remaining equatorial oxygen atoms. BiO$_6$ octahedra distort in this way so as to place their 6s^2 lone pairs in the centers of Bi$_6$O$_6$ 12-membered rings (Fig. 2). Bond valence analysis produced a reasonable value of 3.07 for Bi in this environment. Note that due to the presence of the lone electron pair, irregular coordination geometries with four to seven oxygen nearest-neighbours are preferred by Bi^{3+},
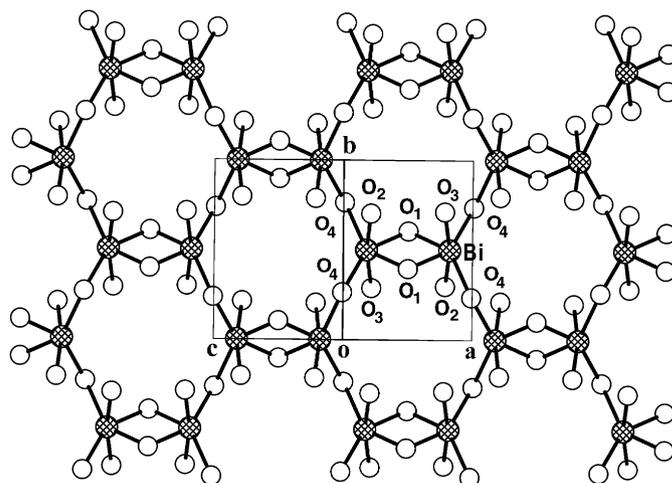
as found in a number of compounds, e. g., BiB$_3$O$_6$ [6], BaBiBO$_4$ [15], Bi$_2$ZnB$_2$O$_7$ [16], and Bi$_3$B$_5$O$_{12}$ [5]; the chains of edge-sharing BiO$_6$ octahedra are rather rare, but have been observed in Bi$_2$CuO$_4$ [21] where the Bi…Bi separations across the shared edge are 3.507(1) $\mathring{A}$, slightly larger than those observed in PbBiBO$_4$ (3.474(1) $\mathring{A}$); while the Bi$_2$O$_{10}^{14-}$ dimer composed of two edge-sharing BiO$_6$ octahedra, to the best of our knowledge, is present here for the first time.

In contrast to the strongly asymmetric coordination environments of Pb^{2+} and Bi^{3+}, the coordination geometry around B is relatively regular. The boron atom occupies one crystallographically distinct site and is bonded to three oxygen atoms to constitute a BO$_3$ triangle which has its plane perpendicular to the b-axis. The O–B–O bond angles cover the range between 118.7(9) and 122.0(8)$^\circ$ with a sum equal to 360°, indicating that the triangular coordination around the B atom is ideally planar. The B–O distances vary from 1.357(14) to 1.416(12) $\mathring{A}$ with an average of 1.382 $\mathring{A}$, which is comparable to those reported in other compounds having BO$_3$ groups e. g. Li$_3$In(BO$_3$)$_2$ (1.371–1.372 $\mathring{A}$) [22] and SrBe$_2$(BO$_3$)$_2$ (1.372–1.374 $\mathring{A}$) [23].

Table 3
Selected bond lengths (Å) and angles (°) for PbBiBO$_4$

| Pb-O1	2.306(7)	Bi -O2	2.351(8)
Pb-O2	2.311(8)	Bi -O3	2.409(8)
Pb-O3	2.329(7)	Bi -O4	2.503(8)
Pb-O1	2.793(8)	Bi -O4	2.590(9)
Pb-O4	2.880(10)	B-O4	1.357(14)
Pb-O4	3.002(10)	B-O3	1.374(13)
Bi-O1	2.177(8)	B-O2	1.416(12)
Bi-O1	2.197(7)		
O1- Bi -O1	74.8(3)	O3- Bi -O4	89.7(3)
O1- Bi -O2	78.6(3)	O1- Bi -O4	145.1(3)
O1- Bi -O2	94.6(3)	O1- Bi -O4	75.2(3)
O1- Bi -O3	93.0(3)	O2- Bi -O4	86.4(3)
O1- Bi -O3	78.5(3)	O3- Bi -O4	98.2(3)
O2- Bi -O3	170.4(2)	O4- Bi -O4	135.29(8)
O1- Bi -O4	77.4(3)	O4-B-O3	122.0(8)
O1- Bi -O4	149.1(3)	O4-B-O2	118.7(9)
O2- Bi -O4	93.0(3)	O3-B-O2	119.3(9)
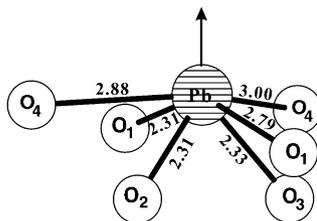
Fig. 3. Coordination geometry about the Pb atom, where Pb-O distances are shown in the unit of Å, and the direction of the electron lone pair is indicated by an arrow.

A bond valence sum of 2.92 can be computed for B, in good agreement with its expected formal valence.

In this structure, four unique oxygen atoms can be classified into two groups depending on their cationic bonding: O2, O3 and O4 atoms are connected to Pb, Bi, and B atoms; while O1 are bonded to Pb and Bi atoms only. Thus the compound is attributed to oxoborates with a formula PbBi(BO$_3$)O. A similar structural formula can also be used to label other borates with similar stoichiometries, such as PbM^{III}BO$_4$ (M^{III} = Ga, Al, Cr, Mn, Fe) [24–26], M^{II}GaBO$_4$ (M^{II} = Ca, Sr) [27–29], and BaBiBO$_4$ [15]. However, their crystal structures are entirely different. PbM^{III}BO$_4$ (M^{III} = Ga, Al, Cr, Mn, Fe) contain one-dimensional single chains of edge-sharing M^{III}O$_6$ octahedra that are arranged in a parallel manner and bridged by BO$_3$ triangles to form a three-dimensional network, with channels in the network hosting Pb^{2+} cations [24–26]. The compounds within the family of M^{II}GaBO$_4$ (M^{II} = Ca, Sr) have one-dimensional structures consisting of infinite chains of corner-sharing GaO$_4$ tetrahedra. These chains are strengthened by BO$_3$ triangles which share corners with adjacent GaO$_4$ tetrahedra, and M^{II+} cations are located around the chains to hold them together through coordination with oxygen atoms [27–29]. The recently

characterized BaBiBO$_4$ contains 1D chains of edge-sharing BO$_3$ triangles and BiO$_5$ groups interleaved with Ba^{2+} cations. The unique asymmetric five-fold coordination of the Bi^{3+} cations enables this compound to crystallize in a novel structure-type with the non-centrosymmetric space group $Pna2_1$ [15]. In PbBiBO$_4$ described here, due to the stereochemical activity of the electron lone pairs, Bi^{3+} are found in a strongly distorted octahedral geometry and Pb^{2+} in a very irregular six-coordinate environment. This is different from the situation in other known M^{II}M^{III}BO$_4$ compounds. It is the variation in the coordination environments around metal cations that is mainly responsible for the structural differences between PbBiBO$_4$ and other borates of similar stoichiometry.

The infrared spectrum of PbBiBO$_4$ was recorded from 4000 to 400 cm^{-1} on a Perkin Elmer 1730 FT-IR spectrometer from KBr pellets. Three sets of bands characteristic of the planar triangular BO$_3$ group have been observed. They are the out-of-plane bending modes ($v2$) occuring at 746.3 and 713.5 cm^{-1}, the antisymmetric stretch ($v3$) at 1220.7 and 1189.9 cm^{-1}, and the in-plane mode ($v4$) at 605.5, 582.4, and 528.4 cm^{-1}. These values correspond well to those reported for the borates containing the planar BO$_3$ groups [30].

It is clear from Fig. 1 that two rows of Pb^{2+} cations in the channels have their stereochemically active nonbonded electron pairs pointing in two opposite directions, which yields a structure without polarity. To confirm this, SHG measurements were performed on the powder sample of PbBiBO$_4$ using a modified Kurtz-NLO system with a 1064 nm light source [31]. No second-harmonic signal at 532 nm was observed which further supports the description of this new structure-type in the centrosymmetric P2$_1$/n space group.

Fig. 4 shows the observed X-ray powder diffraction pattern of PbBiBO$_4$, together with that calculated from the
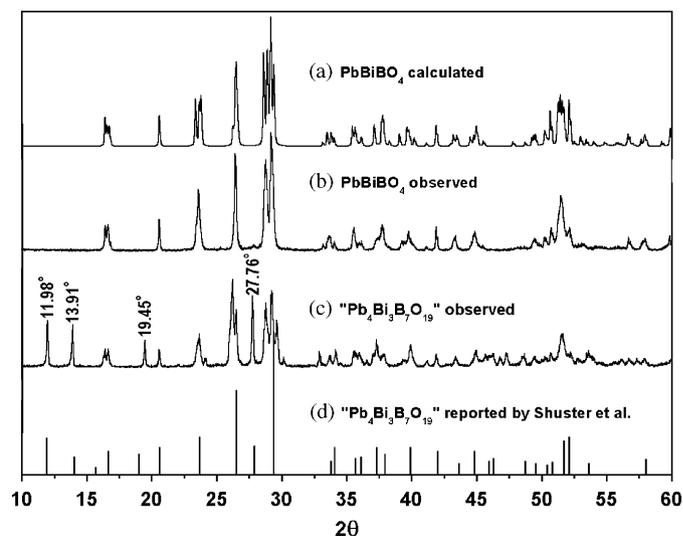

Fig. 4. The calculated (a) and observed (b) X-ray diffraction profiles of PbBiBO$_4$, as compared to those of "Pb$_4$Bi$_3$B$_7$O$_{19}$" prepared in this work (c) and reported by Shuster et al. (d).

single crystal data for comparison. It is clear that the observed XRD pattern is in good agreement with the theoretical one, further confirming our structural model. Note that the $PbBiBO_4$ powder used for X-ray measurements was obtained by heating a stoichiometric mixture of PbO, Bi_2O_3, and B_2O_3 at a low temperature of 450 °C. The crystalline quality of the sample is relatively poor, as indicated by broad and overlapping diffraction profiles. Thus the number of the observed X-ray powder diffraction peaks is less than that of the simulated ones.

As mentioned above, in the ternary system of PbO–Bi_2O_3–B_2O_3, only one phase, "$Pb_4Bi_3B_7O_{19}$", has been proposed on the basis of powder X-ray results [17]. However, no further crystallographic details have been given in the literature up to now. Our attempts to prepare this phase through direct reaction of a stoichiometric mixture of PbO, Bi_2O_3, and B_2O_3 at 500 °C for two weeks have led to a powder XRD pattern very similar to that reported by Shuster et al. [17] (Fig. 4). The pattern is also very close to that of the newly prepared $PbBiBO_4$, except some diffraction lines such as those at about $2\theta = 11.98°$, 13.91°, 19.45°, and 27.76°, which are absent in the pattern of $PbBiBO_4$. These additional lines do not completely match any known compounds in the Pb–Bi–B–O system. Therefore, the previously reported hypothetical compound, "$Pb_4Bi_3B_7O_{19}$", is probably a mixture of $PbBiBO_4$ and an unknown phase rather than an independent phase.

4. Conclusions

A new lead bismuth borate with the composition $PbBiBO_4$ has been synthesized and the crystal structure has been studied. It consists of an original three-dimensional network formed by triangles BO_3, distorted octahedra BiO_6 and irregular polyhedra PbO_6. This structure differs from those of the other known $M^{II}M^{III}BO_4$ compounds due to the presence of $6s^2$ lone pairs on both Pb^{2+} and Bi^{3+} cations. Further works indicated that "$Pb_4Bi_3B_7O_{19}$" reported previously does not exist, but it may be a mixture of $PbBiBO_4$ and an unknown phase.

Auxiliary Material: Further details of the crystal structure investigation may be obtained from the Fachinformationzentrum Karlsruhe, D-76344 Eggenstein- Leopoldshafen, Germany (Fax: (+49)7247-808-666; Email:crysdata@ fiz-karlsruhe.de) on quoting depository numbers CSD 416639.

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