



Synthesis, structure characterization and optical properties of a new tripotassium cadmium pentaborate, $K_3CdB_5O_{10}$

Hongwei Yu ^{a,b}, Shilie Pan ^{a,*}, Hongping Wu ^a, Jian Han ^a, Xiaoyu Dong ^{a,b}, Zhongxiang Zhou ^a

^a Xinjiang Key Laboratory of Electronic Information Materials and Devices, Xinjiang Technical Institute of Physics & Chemistry, Chinese Academy of Sciences, 40-1 South Beijing Road, Urumqi 830011, China

^b Graduate school of Chinese Academy of Sciences, Beijing 100049, China

ARTICLE INFO

Article history:

Received 5 March 2011

Received in revised form

25 April 2011

Accepted 2 May 2011

Available online 7 May 2011

Keywords:

$K_3CdB_5O_{10}$

Pentaborate

Synthesis

Crystal structure

Optical property

ABSTRACT

A new ternary borate oxide, $K_3CdB_5O_{10}$, has been synthesized by solid-state reaction at 580 °C. The compound crystallizes in the monoclinic space group $P2_1/n$ with $a=7.6707$ (7) Å, $b=19.1765$ (17) Å, $c=7.8784$ (6) Å, $\beta=115.6083$ (49)°, and $Z=4$. The crystal structure consists of a two-dimensional infinite $[CdB_5O_{10}]$ layer, which forms by connecting isolated double ring $[B_5O_{10}]$ groups and CdO_4 tetrahedra. K atoms filling in the interlayer and intralayer link the layers together and balance charge. The IR spectrum has been studied and confirmed the presence of both BO_3 and BO_4 groups, and the UV–vis–IR diffuse reflectance spectrum exhibits a band gap of about 3.4 eV. The DSC analysis proves that $K_3CdB_5O_{10}$ is a congruent melting compound.

© 2011 Elsevier Inc. All rights reserved.

1. Introduction

Owing to the large electronegative difference between boron and oxygen and the rich chemistry structure, a boron atom may adopt triangular or tetrahedral oxygen coordination; the BO_3 and BO_4 groups may be further linked via common oxygen atoms to form isolated rings and cages or polymerize into infinite chains, sheets and networks; borates are expected to be excellent non-linear optical materials [1–6]. In addition, it is also found that many borates can be used as phosphors with their promising luminescence and optical properties [7–10]. So borates have attracted material scientists' considerable interests.

It is reported that zinc-containing borates are potential catalysis and optical materials [11] and the systems of $A_2O-ZnO-B_2O_3$ (A =alkali-metal atoms) have been studied. A series of new borate crystals, for example $AZn_4(BO_3)_3$ ($A=K, Rb, Cs$) [12], $LiZnBO_3$ [13,14], $Na_3ZnB_5O_{10}$ [15] and $K_2NaZnB_5O_{10}$ [16] have been synthesized and characterized. Considering that the cadmium locates in the same main group with the zinc in periodic table and they have similar coordination configuration, we believe that cadmium-containing borates can also have potential value as catalysis and optical materials. Besides, it is well-known that the polar displacement of $d^{10} Cd^{2+}$ ion is an aid to the formation of noncentrosymmetric structure [17], which is responsible for the

second harmonic generation of materials. However, in the ternary system of $A_2O-CdO-B_2O_3$ (A =alkali-metal atoms), there are few compounds reported except $LiCdBO_3$ [18,19] and $KCdB_3O_6$ [20] because a more stable binary phase $Cd_2B_2O_5$ forms easily in the system [15]. It is promising to produce a series of new crystals with interesting optical properties by mixing alkali-metal atoms and cadmium atom.

After investigations, we have obtained a new phase, $K_3CdB_5O_{10}$ (KCBO). Herein we report its synthesis, crystal structure, spectrum properties and thermal behavior for the first time.

2. Experimental

2.1. Compound synthesis

Polycrystalline KCBO was prepared by solid-state reaction techniques. All reagents were of analytical grade. A stoichiometric ratio of KNO_3 (Tianjin Huadong Chemical Co., Ltd., 99.0%), CdO (Tianjin Bodi Chemical Co., Ltd., 99.0%) and H_3BO_3 (Tianjin Baishi Chemical Co., Ltd., 99.5%) was mixed thoroughly. The mixture was heated at 450 °C for 4 h and at 580 °C for 48 h. The material was ground between all heating procedures. A single-phase powder of KCBO was obtained when repeated heat treatment caused no further changes in the X-ray powder diffraction pattern.

X-ray powder diffraction analysis of KCBO was performed at room temperature in the angular range of $2\theta=10-70^\circ$ with a scan step width of 0.02° and a fixed counting time of 1 s/step using an

* Corresponding author. Fax: +86 991 3838957.

E-mail address: slpan@ms.xjb.ac.cn (S. Pan).

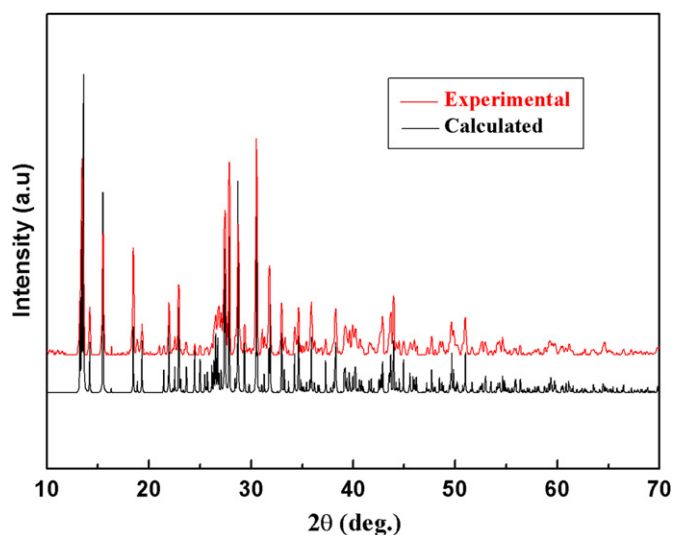


Fig. 1. Experimental and calculated XRD patterns of $K_3CdB_5O_{10}$. The red curve is its experimental pattern; the black one is its calculated one. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

automated Bruker D8 ADVANCE X-ray diffractometer equipped with a diffracted beam monochromator set for $CuK\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The experimental powder X-ray diffraction pattern of KCBO is in agreement with the calculated data based on the single-crystal data, suggesting that it is a pure phase (Fig. 1).

2.2. Crystal growth

Single crystals of KCBO were grown from a high temperature solution using $PbO-PbF_2$ as the flux system. The solution was prepared in a platinum crucible by melting a mixture of $KF \cdot 2H_2O$ (Tianjin Hengxing Chemical Reagent Co., Ltd., 99.0%), CdO (Tianjin Bodi Chemical Co., Ltd., 99.0%), H_3BO_3 (Tianjin Baishi Chemical Co., Ltd., 99.5%) and PbO (Tianjin Baishi Chemical Reagent Co., Ltd., 99.0%) at a molar ratio of 3:1:5:2. The Pt crucible, which was placed in the center of a vertical, programmable temperature furnace, was gradually heated to $750 \text{ }^\circ\text{C}$, held at this temperature for 20 h and then quickly cooled to the initial crystallization temperature ($680 \text{ }^\circ\text{C}$). Then, a platinum wire was promptly dipped into the solution. The temperature was decreased to $640 \text{ }^\circ\text{C}$ at a rate of $0.5 \text{ }^\circ\text{C/h}$. Then the platinum wire was pulled out of the solution, and allowed to cool to room temperature at a rate of $10 \text{ }^\circ\text{C/h}$. Thus a few colorless, transparent crystals were obtained for the structure determination.

2.3. X-ray crystallographic studies

A plate single crystal of KCBO with dimensions $0.03 \text{ mm} \times 0.09 \text{ mm} \times 0.15 \text{ mm}$ was selected for the structure determination. The crystal structure of KCBO was determined by single-crystal X-ray diffraction on an APEX II CCD diffractometer using monochromatic $MoK\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$) at $296(2) \text{ K}$ and integrated with the SAINT program [21]. All calculations were performed with programs from the SHELXTL crystallographic software package [22]. All atoms were refined using full matrix least-squares techniques with anisotropic thermal parameters; final least-squares refinement is on F_o^2 with the data having $F_o^2 \geq 2\sigma(F_o^2)$. The final difference Fourier synthesis map showed the maximum and minimum peaks at $0.824e \text{ \AA}^{-3}$ (1.25 \AA from $Cd(1)$) and $-0.928e \text{ \AA}^{-3}$ (0.82 \AA from $Cd(1)$), respectively. The structure was checked with PLATON [23]. Crystal data and

Table 1

Crystal data and structure refinement for $K_3CdB_5O_{10}$.

| Empirical formula | $K_3Cd B_5O_{10}$ |
|---|---|
| Formula weight | 443.75 |
| Temperature | 296(2) K |
| Wavelength | 0.71073 \AA |
| Crystal system | Monoclinic |
| Space group, Z | $P2_1/n$, 4 |
| Unit cell dimensions | $a = 7.6707(7) \text{ \AA}$ $b = 19.1765(17) \text{ \AA}$ $c = 7.8784(6) \text{ \AA}$ $\beta = 115.6083(49)^\circ$ |
| Volume | $1045.05(15) \text{ \AA}^3$ |
| Density (calculated) | 2.820 mg/m^3 |
| Absorption coefficient | $3.325/\text{mm}$ |
| $F(000)$ | 840 |
| Crystal size | $0.15 \text{ mm} \times 0.09 \text{ mm} \times 0.03 \text{ mm}$ |
| Theta range for data collection | $2.12\text{--}27.46^\circ$ |
| Limiting indices | $-7 \leq h \leq 9, -24 \leq k \leq 24, -10 \leq l \leq 10$ |
| Reflections collected/unique | 9178/2368 [$R(\text{int}) = 0.0371$] |
| Completeness to theta = 27.46 | 99.5% |
| Refinement method | Full-matrix least-squares on F^2 |
| Data/restraints/parameters | 2368/0/173 |
| Goodness-of-fit on F^2 | 1.107 |
| Final R indices [$F_o^2 > 2\sigma(F_o^2)$] ^a | $R_1 = 0.0344, WR_2 = 0.0834$ |
| R indices (all data) ^a | $R_1 = 0.0470, WR_2 = 0.0884$ |
| Extinction coefficient | 0.0006(5) |
| The largest diff. peak and hole | 0.824 and $-0.928e \text{ \AA}^{-3}$ |

^a $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$ and $WR_2 = [\sum W(F_o^2 - F_c^2)^2 / \sum W F_o^4]^{1/2}$ for $F_o^2 > 2\sigma(F_o^2)$ and $W^{-1} = \sigma^2(F_o^2) + (0.0461P)^2 + 0.31P$ where $P = (F_o^2 + 2F_c^2)/3$.

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $K_3CdB_5O_{10}$. U_{eq} is defined as the one-third of the trace of the orthogonalized U_{ij} tensor.

| Atom | x | y | z | U (eq.) | BVS |
|-------|-----------|---------|----------|---------|-------|
| Cd(1) | 5247(1) | 973(1) | 1521(1) | 20(1) | 1.972 |
| K(1) | 10,065(2) | 781(1) | 1411(1) | 25(1) | 1.127 |
| K(2) | 5458(2) | 655(1) | -2985(1) | 26(1) | 1.039 |
| K(3) | 2197(2) | 2526(1) | -544(1) | 27(1) | 1.222 |
| B(1) | 6914(7) | 2165(3) | 4(6) | 18(1) | 3.02 |
| B(2) | 1637(7) | 694(3) | -1865(6) | 18(1) | 3.009 |
| B(3) | 1319(7) | 1584(3) | -4368(6) | 19(1) | 3.006 |
| B(4) | 3030(7) | 1896(3) | 3656(6) | 21(1) | 3.098 |
| B(5) | 8600(7) | 742(3) | 5233(6) | 18(1) | 3.009 |
| O(1) | 4369(4) | 3718(2) | -375(4) | 21(1) | 2.013 |
| O(2) | 6764(4) | 1478(2) | 156(4) | 20(1) | 2.104 |
| O(3) | 6863(4) | 475(2) | 4232(4) | 23(1) | 1.977 |
| O(4) | 2297(4) | 1268(2) | -2457(4) | 19(1) | 1.988 |
| O(5) | 2454(4) | 1415(2) | -5416(4) | 20(1) | 2.229 |
| O(6) | 2695(4) | 353(2) | -273(4) | 26(1) | 1.871 |
| O(7) | 1158(4) | 2343(2) | -4218(4) | 19(1) | 2.149 |
| O(8) | 10,274(4) | -473(2) | 2940(4) | 25(1) | 2.119 |
| O(9) | 3705(5) | 1717(2) | 2427(4) | 32(1) | 2.108 |
| O(10) | -2085(5) | 2398(2) | -1005(5) | 32(1) | 1.979 |

structure refinement information are given in Table 1. The final refined atomic positions and isotropic thermal parameters are summarized in Table 2. Selected bond distances (\AA) and angles (deg.) for KCBO are listed in Table 3.

2.4. Infrared spectroscopy

Infrared spectrum was recorded on Shimadzu IRAffinity-1 Fourier transform infrared spectrometer in the $400\text{--}4000 \text{ cm}^{-1}$ range. The sample was mixed thoroughly with dried KBr (4.3 mg of the sample; 400 mg of KBr).

2.5. UV–vis-IR optical diffuse reflectance

Optical diffuse reflectance spectrum was measured at room temperature with Shimadzu SolidSpec-3700DUV spectrophotometer. Data were collected in the wavelength range 190–2500 nm.

2.6. Thermal analysis

The thermal analyses were carried out on a simultaneous NETZSCH STA 449C thermal analyzer instrument, with a heating

Table 3

Selected bond lengths (Å) and angles (deg.) for $K_3CdB_5O_{10}$.

| | | | |
|---------------------|------------|----------------------|----------|
| Cd(1)–O(2) | 2.128(3) | K(3)–O(1) | 2.798(3) |
| Cd(1)–O(9) | 2.162(3) | K(3)–O(9)#8 | 2.852(4) |
| Cd(1)–O(3) | 2.174(3) | K(3)–O(4) | 2.863(3) |
| Cd(1)–O(6) | 2.203(3) | K(3)–O(10) | 3.154(4) |
| K(1)–O(2) | 2.648(3) | K(3)–O(10)#2 | 3.378(3) |
| K(1)–O(8) | 2.664(3) | B(1)–O(2) | 1.331(6) |
| K(1)–O(5)#4 | 2.669(3) | B(1)–O(7)#2 | 1.382(6) |
| K(1)–O(6)#1 | 2.894(3) | B(1)–O(10)#5 | 1.396(6) |
| K(1)–O(1)#2 | 2.964(3) | B(2)–O(6) | 1.334(5) |
| K(1)–O(6)#5 | 2.969(4) | B(2)–O(4) | 1.374(6) |
| K(1)–O(9)#5 | 3.118(4) | B(2)–O(8)#1 | 1.405(6) |
| K(1)–O(8)#6 | 3.375(3) | B(3)–O(7) | 1.470(6) |
| K(2)–O(5) | 2.700(3) | B(3)–O(5) | 1.472(6) |
| K(2)–O(3)#1 | 2.705(3) | B(3)–O(1)#8 | 1.475(6) |
| K(2)–O(2) | 2.735(3) | B(3)–O(4) | 1.490(5) |
| K(2)–O(3)#7 | 2.851(3) | B(4)–O(9) | 1.325(6) |
| K(2)–O(4) | 2.882(3) | B(4)–O(5)#10 | 1.364(6) |
| K(2)–O(6)#1 | 3.031(3) | B(4)–O(10)#2 | 1.391(6) |
| K(2)–O(8)#6 | 3.277(4) | B(5)–O(3) | 1.323(6) |
| K(3)–O(9) | 2.624(3) | B(5)–O(1)#2 | 1.376(6) |
| K(3)–O(7) | 2.672(3) | B(5)–O(8)#11 | 1.417(5) |
| K(3)–O(7)#2 | 2.765(3) | | |
| O(2)–Cd(1)–O(9) | 111.29(13) | O(7)–B(3)–O(5) | 110.7(4) |
| O(2)–Cd(1)–O(3) | 119.51(11) | O(7)–B(3)–O(1)#8 | 109.4(4) |
| O(9)–Cd(1)–O(3) | 97.27(12) | O(5)–B(3)–O(1)#8 | 108.6(3) |
| O(2)–Cd(1)–O(6) | 116.74(11) | O(7)–B(3)–O(4) | 110.2(3) |
| O(9)–Cd(1)–O(6) | 96.12(13) | O(5)–B(3)–O(4) | 108.4(4) |
| O(3)–Cd(1)–O(6) | 111.38(12) | O(1)#8–B(3)–O(4) | 109.5(4) |
| O(2)–B(1)–O(7)#2 | 124.4(4) | O(9)–B(4)–O(5)#10 | 122.4(5) |
| O(2)–B(1)–O(10)#5 | 117.3(4) | O(9)–B(4)–O(10)#2 | 118.0(5) |
| O(7)#2–B(1)–O(10)#5 | 118.3(4) | O(5)#10–B(4)–O(10)#2 | 119.6(4) |
| O(6)–B(2)–O(4) | 123.3(4) | O(3)–B(5)–O(1)#2 | 123.8(4) |
| O(6)–B(2)–O(8)#1 | 118.1(4) | O(3)–B(5)–O(8)#11 | 118.6(4) |
| O(4)–B(2)–O(8)#1 | 118.6(4) | O(1)#2–B(5)–O(8)#11 | 117.5(4) |

Note: Symmetry transformations used to generate equivalent atoms:

#1 $-x+1, -y, -z$; #2 $2x+1/2, -y+1/2, z+1/2$; #3 $x-1, y, z$; #4 $x+1, y, z+1$; #5 $x+1, y, z$; #6 $-x+2, -y, -z$; #7 $x, y, z-1$; #8 $x-1/2, -y+1/2, z-1/2$; #9 $x-1, y, z-1$; #10 $x, y, z+1$; #11 $-x+2, -y, -z+1$.

rate of $10^\circ\text{C min}^{-1}$ in an atmosphere of flowing N_2 from 25 to 900°C .

3. Results and discussion

3.1. Crystal structure

KCBO crystallizes in space group $P2_1/n$ of the monoclinic system. In the asymmetric unit, there are three unique K atoms, one unique Cd atom, five unique B atoms and ten unique O atoms (Table 2). The crystal structure of KCBO consists of a two-dimensional infinite $[\text{CdB}_5\text{O}_{10}]$ layer (Fig. 2). Each $[\text{B}_5\text{O}_{10}]$ group connects with four different CdO_4 tetrahedra through its terminal O atoms and likewise each CdO_4 tetrahedron shares its four vertices with four neighboring $[\text{B}_5\text{O}_{10}]$ groups to form a two-dimensional (2D) infinite $[\text{CdB}_5\text{O}_{10}]$ layer proliferating in the (010) plane (Fig. 2b). The layers further are connected together by sharing O atoms with K(1) and K(2) cations located between these layers to generate a three-dimensional network with open channels along the [101] direction, where K(3) is situated (Fig. 3).

The four BO_3 -groups (Δ) and one BO_4 -tetrahedron (\square) are connected via common corners to form an isolated double ring $[\text{B}_5\text{O}_{10}]$, and the two hexagonal rings are almost perpendicular to each other. According to the modern descriptions of borate groups by Burns et al. [24], the unit is symbolized as $4\Delta 1\square$: $\langle 2\Delta\square \rangle - \langle 2\Delta\square \rangle$. In the unit, there are five crystallographically independent B atoms, of which B(3) is in tetrahedral coordination and the other B atoms in triangular coordination. For BO_3 groups, the B–O bond lengths range from 1.331(6) to 1.417(6) Å, bearing an average distance of 1.374 Å. In the BO_4 group, the B–O distances are in the range 1.470(6)–1.490(5) Å and in an average bond distance of 1.477 Å. These values are in agreement with other borate compounds reported previously [25–28]. In addition bond valence sum (BVS) [29,30] calculation using Brown's formula for B also gave reasonable values in the range 3.006–3.020 (Table 2).

It is well-known that the cadmium atoms form distorted CdO_4 tetrahedra in CdB_4O_7 [31] and CdO_6 octahedra in both $\text{Cd}_3(\text{BO}_3)_2$ and $\text{Cd}_2\text{B}_2\text{O}_5$ [32,33]. In the KCBO, the Cd atoms are coordinated to a tetrahedron of four O atoms. The Cd–O bond lengths range from 2.128(3) to 2.203(3) Å, consistent with that of 2.170(1)–2.306(1) Å in LiCdBO_3 [19]. A bond valence analysis produced a BVS value of 1.972 for the Cd atom, which supports the choice of 4-fold coordination to describe the Cd environment.

The three K atom types possess different coordination environments (Fig. 4). The K(1) and K(3) atoms assume two distinctive irregular polyhedra coordinated by eight O atoms, respectively,

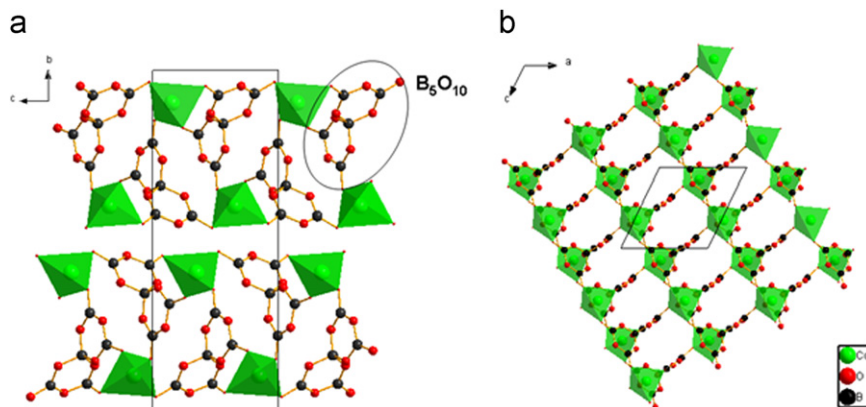


Fig. 2. Layer structure of $K_3CdB_5O_{10}$ with K atoms omitted for clarity (a) along a -axis and (b) a single $\text{CdB}_5\text{O}_{10}$ layer along the ac -plane.

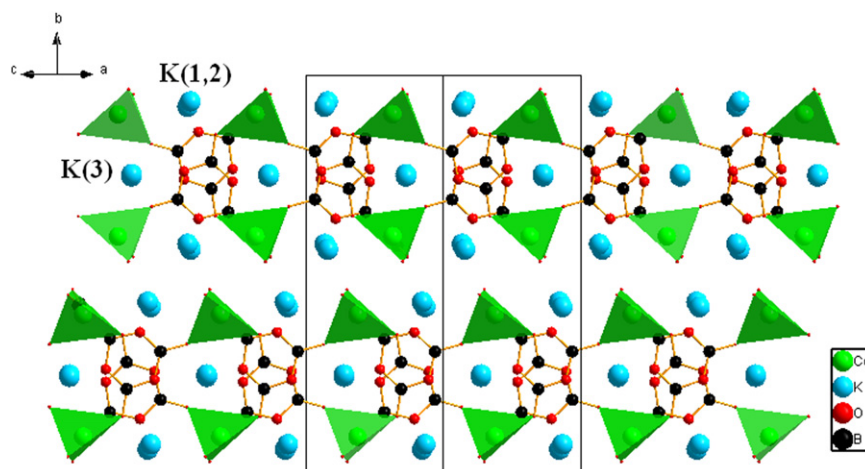


Fig. 3. Structures of $K_3CdB_5O_{10}$ with K(1) and K(2) atoms filled in the interlayer and K(3) atom filled in the intralayer, with all the K–O bonds omitted.

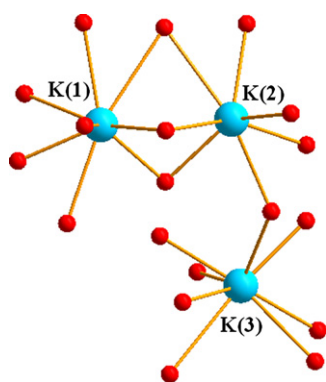


Fig. 4. Potassium-coordinated environments.

while the K(2) atoms are coordinated to a distorted pentagonal bipyramid of seven O atoms. The K–O bond lengths for three type K atoms are in a similar range. It ranges from 2.648(3) to 3.375(3) Å for K(1) atom, 2.700(3) to 3.277(4) Å for K(2) atom and 2.624(3) to 3.378(3) Å for K(3) atom. They are all closely comparable to other compounds reported previously [16,34,35]. BVS calculation for K(1) atom equals to 1.127, 1.039 for K(2) atom and 1.222 for K(3) atom, which further prove that the coordination of K atoms is very reasonable.

In the K_2O – CdO – B_2O_3 system, another compound $KCdB_3O_6$ has also been synthesized and characterized [20]. However, its crystal structure is obviously different from KCBO. In the crystal structure of $KCdB_3O_6$, the Cd atom is five-coordinated by O atoms in distorted trigonal bipyramidal geometry and the CdO_5 polyhedra connect each other by sharing O atoms to form a one-dimensional CdO_5 chain along the *c*-axis. And the basic B–O units of $KCdB_3O_6$ are the B_3O_6 groups, which are in the planes almost perpendicular to the CdO_5 chain. In addition, the CdO_5 chains and B_3O_6 units interlink each other forming a three-dimensional $[CdB_3O_6]$ framework structure. The differences exhibiting in the coordinated environment of Cd atoms, basic B–O units and crystal framework structure make the two compounds ($KCdB_3O_6$ and $K_3CdB_5O_{10}$) crystallize in different space groups ($C2/c$ and $P2_1/n$).

3.2. Infrared spectrum measurements

In order to further confirm the coordination surroundings of B–O in the KCBO structure, infrared spectrum was carried out and the spectrum from 400–4000 cm^{-1} is shown in Fig. 5. According to previous works [36–38], the peaks at 1417, 1246 and

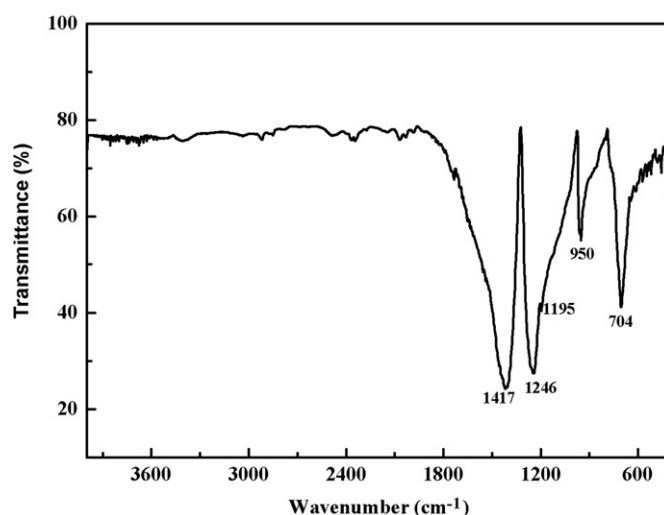


Fig. 5. The IR spectrum of $K_3CdB_5O_{10}$.

1195 cm^{-1} can be attributed to asymmetric stretching and symmetric stretching vibrations of BO_3 groups, respectively. 905 cm^{-1} and 704 cm^{-1} are likely the asymmetric and symmetric stretchings of B–O in BO_4 , respectively.

3.3. UV-vis-IR diffuse reflectance spectrum

The UV-vis-IR diffuse reflectance spectrum of KCBO in the region 190–2500 nm is shown in Fig. 6. It is clear that KCBO has no obvious absorption from 360 to 2500 nm and the absorption sharply increases below 360 nm. So we can estimate that the optical band gap of KCBO is 3.4 eV.

3.4. DSC analysis

The DSC curve is shown in Fig. 7: one endothermic peak can be observed on the DSC curve, which tentatively suggests that KCBO melts congruently. To verify that KCBO melts congruently, KCBO compound powder was put into a platinum crucible and heated to 900 °C, then slowly cooled to room temperature. Analysis of the powder XRD pattern of the solidified melt revealed that the solid product exhibited a diffraction pattern identical to that of the initial KCBO powder, further demonstrating that KCBO is a congruent melting compound (Fig. S1 in the Supporting Information). For the congruent melting compound, it is well-known that

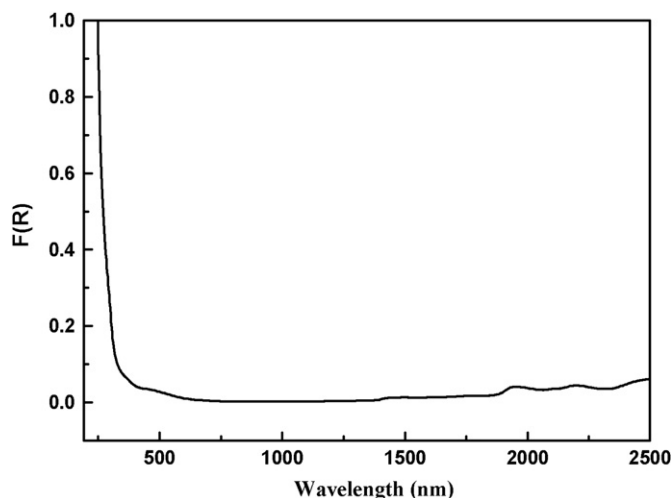


Fig. 6. UV-vis-IR diffuse reflectance spectrum of $K_3CdB_5O_{10}$.

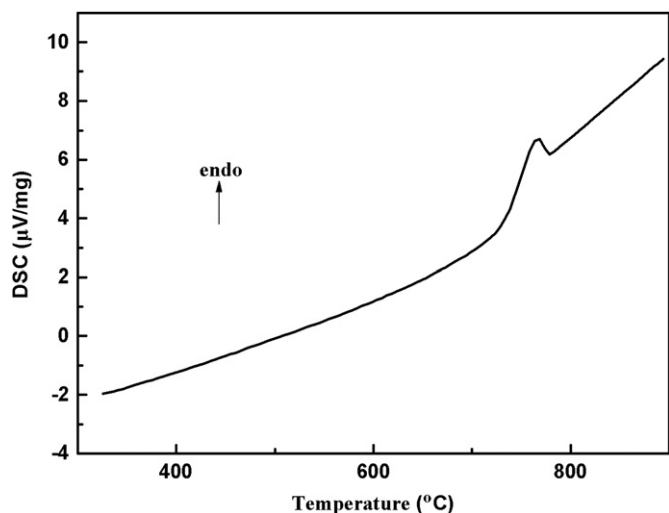


Fig. 7. DSC curve of $K_3CdB_5O_{10}$.

large single crystals could be grown from the melt using the Czochralski pulling method [39].

4. Conclusion

Totally we have been successful in preparation of a novel ternary borate with the composition $K_3CdB_5O_{10}$ and studied its synthesis, structure, optical properties and thermal behavior. Its structure exhibits a complicated three-dimensional network with a $[CdB_5O_{10}]$ layers composed of CdO_4 tetrahedra and $[B_5O_{10}]$ groups, which are interconnected through sharing terminal O atoms of $[B_5O_{10}]$ groups. DSC analysis exhibits KCBO is a congruent melting compound, which means that we can get the large single crystals from the melt using the Czochralski pulling method.

Acknowledgments

This work is supported by the Main Direction Program of Knowledge Innovation of Chinese Academy of Sciences (Grant no.

KJJCX2-EW-H03-03), the “National Natural Science Foundation of China” (Grant nos. 50802110, 21001114), the “One Hundred Talents Project Foundation Program” of Chinese Academy of Sciences, the “Western Light Joint Scholar Foundation” Program of Chinese Academy of Sciences, the “High Technology Research and Development Program” of Xinjiang Uygur Autonomous Region of China (Grant no. 200816120) and Scientific Research Program of Urumqi of China (Grant no. G09212001).

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jssc.2011.05.007.

References

- [1] P. Becker, *Adv. Mater.* 10 (1998) 979.
- [2] P.S. Halasyamani, K.R. Poeppelmeier, *Chem. Mater.* 10 (1998) 2753.
- [3] J.D. Grice, P.C. Burns, F.C. Hawthorne, *Can. Mineral.* 37 (1999) 731.
- [4] T. Sasaki, Y. Mori, M. Yoshimura, Y.K. Yap, T. Kamimura, *Mater. Sci. Eng. R* 30 (2000) 1.
- [5] S.L. Pan, Y.C. Wu, P.Z. Fu, G.C. Zhang, Z.H. Li, C.X. Du, C.T. Chen, *Chem. Mater.* 15 (2003) 2218.
- [6] C. Chen, Z. Lin, Z. Wang, *Appl. Phys. B—Lasers Opt.* 80 (2005) 1.
- [7] Z.W. Pei, Q. Su, Y. Zhang, *J. Alloys Compd.* 198 (1993) 51.
- [8] A. Diaz, D.A. Keszler, *Chem. Mater.* 9 (1997) 2071.
- [9] R. Sankar, *Solid State Sci.* 10 (2008) 1864.
- [10] C.J. Duan, W.F. Li, J.L. Yuan, J.T. Zhao, *J. Alloys Compd.* 458 (2008) 536.
- [11] R.W. Smith, D.A. Keszler, *J. Solid State Chem.* 100 (1992) 325.
- [12] R.W. Smith, J.L. Luce, D.A. Keszler, *Inorg. Chem.* 31 (1992) 4679.
- [13] K.S. Chang, *J. Korean Chem. Soc.* 45 (2001) 251.
- [14] O.S. Bondareva, M.A. Simonov, Y.K. EgorovTismenko, N.V. Belov, *Kristallografiya* 23 (1978) 487.
- [15] X.A. Chen, M. Li, X.A. Chang, H.G. Zang, W.Q. Xiao, *J. Solid State Chem.* 180 (2007) 1658.
- [16] X.A. Chen, C.Y. Yang, X.N. Chang, H.G. Zang, W.Q. Xiao, *J. Alloys Compd.* 492 (2010) 543.
- [17] W.L. Zhang, W.D. Cheng, H. Zhang, L. Geng, C.S. Lin, Z.Z. He, *J. Am. Chem. Soc.* 132 (2010) 1508.
- [18] E.V. Kazanskaia, P.A. Sandomirskii, M.A. Simonov, N.V. Belov, *Dok. Akad. Nauk* 238 (1978) 1340.
- [19] E.V. Sokolova, M.A. Simonov, N.V. Belov, *Kristallografiya* 25 (1980) 1285.
- [20] S.F. Jin, G.M. Chai, J. Liu, W.Y. Wang, X.L. Chen, *Acta Crystallogr. C* 65 (2009) i42.
- [21] SAINT, Version 7.60A, Bruker Analytical X-ray Instruments, Inc., Madison, WI, 2008.
- [22] G.M. Sheldrick, SHELXTL, Version 6.14, Bruker Analytical X-ray Instruments, Inc., Madison, WI, 2003.
- [23] A.L. Spek, *J. Appl. Crystallogr.* 36 (2003) 7.
- [24] P.C. Burns, J.D. Grice, F.C. Hawthorne, *Can. Mineral.* 33 (1995) 1131.
- [25] F. Li, X.L. Hou, S.L. Pan, X. Wang, *Chem. Mater.* 21 (2009) 2846.
- [26] S.L. Pan, J.P. Smit, M.R. Marvel, E.S. Stampler, J.M. Haag, J. Baek, P.S. Halasyamani, K.R. Poeppelmeier, *J. Solid State Chem.* 181 (2008) 2087.
- [27] Y.J. Wang, S.L. Pan, X.L. Tian, Z.X. Zhou, G. Liu, J.D. Wang, D.Z. Jia, *Inorg. Chem.* 48 (2009) 7800.
- [28] J.H. Zhang, C.L. Hu, X.A. Xu, F. Kong, J.G. Mao, *Inorg. Chem.* 50 (2011) 1973.
- [29] N.E. Brese, M. O'Keefe, *Acta Crystallogr. Sect. B: Struct. Sci.* 47 (1991) 192.
- [30] I.D. Brown, D. Altermatt, *Acta Crystallogr. Sect. B: Struct. Sci.* 41 (1985) 244.
- [31] M. Ihara, J. Krogh-Moe, *Acta Crystallogr.* 20 (1966) 132.
- [32] Y. Laureiro, M.L. Veiga, M.L. Lopez, S. Garcia-Martin, A. Jerez, C. Pico, *Powder Diffr.* 6 (1991) 28.
- [33] M. Weil, *Acta Crystallogr. E* 59 (2003) i95.
- [34] X.C. Luo, S.L. Pan, X.Y. Fan, J.D. Wang, G. Liu, *J. Cryst. Growth* 311 (2009) 3517.
- [35] Y. Wu, J.Y. Yao, J.X. Zhang, P.Z. Fu, Y.C. Wu, *Acta Crystallogr. E* 66 (2010) i45.
- [36] F. Kong, H.L. Jiang, T. Hu, J.G. Mao, *Inorg. Chem.* 47 (2008) 10611.
- [37] X.E. Chen, H.P. Xue, X.N. Chang, L. Zhang, Y.H. Zhao, J.L. Zuo, H.G. Zang, W.Q. Xiao, *J. Alloys Compd.* 425 (2006) 96.
- [38] G.J. Chen, Y.C. Wu, P.Z. Fua, *J. Solid State Chem.* 180 (2007) 2194.
- [39] G. Aka, F. Mougél, F. Auge, A. Kahn-Harari, D. Vivien, J.M. Benitez, F. Salin, D. Pelenc, F. Balembois, P. Georges, A. Brun, N. Le Nain, M. Jacquet, *J. Alloys Compd.* 401 (2000) 408.