



## Original articles

Research article

<https://doi.org/10.17308/kcmf.2024.26/11943>**Synthesis and characterization of lead and cadmium hexaborates doped with Cr<sup>3+</sup>**T. N. Khamaganova *Baikal Institute of Nature Management Siberian Branch of the Russian Academy of Sciences,  
6 Sakhyanovoy str., Ulan-Ude 670047, Buryatia, Russian Federation***Abstract**

Borates doped with transition metals (Mn, Cu, Cr) exhibit a significant and long-lasting luminescence at room temperature, high power, and other outstanding characteristics. Therefore, the purpose of the study was to establish the possibility of the formation of borate materials containing chromium and the determination of their structure and thermal properties.

New phases of variable composition were synthesized in the  $\text{PbCd}_{2-x}\text{B}_6\text{O}_{12} \cdot x\text{Cr}^{3+}$  system by heterovalent substitution of  $\text{Cd}^{2+}$  ions with  $\text{Cr}^{3+}$  ions using solid-phase reactions at 640 °C. The phases were isolated in the concentration range  $0 \leq x \leq 7.0$  mol % and characterized by X-ray phase analysis (XRD), differential scanning calorimetry (DSC) and IR spectroscopy. According to XRD and IR spectra, the resulting borates crystallize in a monoclinic cell and are assigned to one structural type (space group  $P2_1/n$ ,  $Z = 4$ ).

The crystallographic characteristics of the new phases have been determined. The crystal lattice parameters and their volumes decrease monotonically, indicating the formation of a continuous series of substitutional solid solutions in the studied concentration range. According to the DSC results, the sample  $\text{PbCd}_{2-x}\text{B}_6\text{O}_{12} \cdot 0.03\text{Cr}^{3+}$  melts incongruently at 729 °C.

**Keywords:** Polycrystals of lead and cadmium borates,  $\text{Cr}^{3+}$  ions, Solid-state reaction method, Solid solutions, X-ray phase analysis, DSC, IR spectroscopy

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## 1. Introduction

One of the effective ways to improve the functional properties of many classes of inorganic compounds is the substitution of cations in their crystal structures. By the substitution of cations within the structural type, it is possible to carry out not only the directed synthesis of the desired crystalline phase, but also to set and regulate the necessary physicochemical properties [1–5].

In the excitation spectrum at 300 nm near the absorption edge of PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub> hexaborate two broad emission bands were detected at 510 nm and 617 nm [6]. According to the authors of [6], the emission bands are an integral property of the undoped compound and are consistent with the emission bands in CdB<sub>4</sub>O<sub>7</sub> and Cd<sub>2</sub>B<sub>6</sub>O<sub>11</sub>.

The obtained temperature dependences of the thermoluminescence intensity of the obtained new series of PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>:xM borates on the composition of active ions (M = Mn<sup>2+</sup>, Eu<sup>3+</sup>, Cu<sup>2+</sup>) were promising [7–9]. Thermal fluorescence curves for samples of solid solutions of the PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub>:Cu<sup>2+</sup> ( $x = 0.01; 0.03; 0.05; 0.06; 0.08$ ) system upon excitation with ultraviolet light for 5 min are shown in Fig. 1. The results of thermoluminescent sensitivity measurements were normalized by the signal from the standard, which was LiF:Mg,Ti lithium fluoride (TLD-100). The thermoluminescence intensities of the studied series of borates were not inferior to the

intensity of the first maximum of the industrial phosphor (TLD-100) [9].

The data obtained allow us to consider materials based on lead and cadmium hexaborate as promising phosphors. Chromium ions Cr<sup>3+</sup> are among the well-known activators successfully used to create luminescent materials for modern lighting technology [10–14]. The investigation of the properties of the described above hexaborates with active chromium ions Cr<sup>3+</sup> for the assessment of the efficiency of thermoluminescent properties, which are expected to be studied in the future, is of interest.

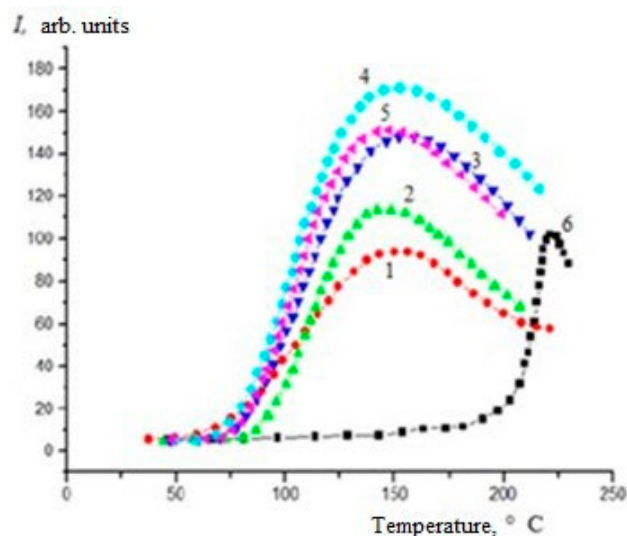
The purpose of this study was to obtain polycrystalline samples of phases based on double lead and cadmium borate PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub>, doped with chromium ions Cr<sup>3+</sup>, studying them using X-ray diffraction, IR spectroscopy and differential scanning calorimetry.

## 2. Experimental

Synthesis of powder preparations PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>:xCr<sup>3+</sup> was carried out using the method of solid-phase reactions, varying the activator content from 1 to 7 mol %.

The starting reagents were chemically pure boric acid H<sub>3</sub>BO<sub>3</sub> and metal oxides PbO, CdO, Cr<sub>2</sub>O<sub>3</sub> (OOO Krasny Khimik, Russia). Metal oxides were pre-calcined at 500 °C for 5–6 h. Stoichiometric amounts of the starting reagents were annealed in the range from 400 to 640 °C for 150 h with repeated intermediate homogenization. The synthesis temperature was successively increased by 50–100 °C. Before each increase in temperature, the samples were homogenized and their phase composition was determined using X-ray powder diffraction analysis (XRD). Identification of doped phases was carried out by comparison with the X-ray diffraction pattern of individual PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub> borate, the structure of which was determined for a single crystal [6].

X-ray diffraction data of polycrystals of the synthesized borates were obtained using D8 ADVANCE Bruker AXS powder autodiffractometer with a Vantec-1 detector (CuK<sub>α</sub>-radiation). The imaging of the samples was performed at room temperature in the diffraction angle range of 10–60° with a scanning step of 0.02°. The experimental intensities were processed and the unit cell parameters of the obtained phases were



**Fig. 1.** Thermal luminescence curves of PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>:xCu<sup>2+</sup> samples with  $x = 0.03$  (1), 0.05 (2), 0.07 (3), 0.06 (4), 0.08 (5); LiF – (6) upon excitation with UV for 5 min

refined using the TOPAS 4.2 software package [15]. Diffraction patterns of doped samples and pure lead and cadmium hexaborate are shown in Fig. 2. Monophasicity of polycrystals of pure  $\text{PbCd}_2\text{B}_6\text{O}_{12}$  and activated  $\text{PbCd}_{2-x}\text{B}_6\text{O}_{12} : 0.03 \text{ Cr}^{3+}$  was confirmed by thermal analysis.

Thermoanalytical studies were carried out by differential scanning calorimetry (DSC) using a Netzsch STA 449c F1 JUPITER synchronous thermal analyzer. A sample weighing 19.7 mg was placed in a Pt crucible with a lid and heated in an argon atmosphere in the range of 25–800 °C. The sample heating rate was 10 °C/min.

Infrared absorption spectra of the synthesized compounds were recorded using ALPHA IR Fourier spectrometer (BRUKER) in tablets with KBr in the range of 400–4000  $\text{cm}^{-1}$ . Interpretation of the spectra and assignment of absorption bands were performed based on data from [16–20].

### 3. Results and discussion

The crystal structure of  $\text{PbCd}_2\text{B}_6\text{O}_{12}$  is a three-dimensional framework formed by boron-oxygen layers  $[(\text{B}_6\text{O}_{12})^{6-}]_n$ , extending parallel to the plane  $ab$  [6]. Between them are one-dimensional tunnels of 8-membered rings filled with  $\text{Cd}(2)\text{O}_6$ -octahedra chains. Chains of  $\text{Cd}(2)\text{O}_6$ -octahedra, connected by common edges, form new two-

dimensional  $[\text{Cd}_2\text{B}_6\text{O}_{12}]^{4-}$  layers also parallel to the  $ab$  plane. Two-dimensional  $[\text{Cd}_2\text{B}_6\text{O}_{12}]^{4-}$  layers, connecting by bridging dimers of edge-linked  $\text{Cd}(1)\text{O}_7$ -polyhedra, form a three-dimensional  $[\text{Cd}_2\text{B}_6\text{O}_{12}]^{2-}$  anion network.  $\text{Pb}^{2+}$  ions with CN = 7 are located in the voids of the three-dimensional frame. The boron atoms in the structure are coordinated by three and four oxygen atoms.

XRD of samples activated with chromium ions showed the absence of impurity phases. The X-ray diffraction patterns of the borates of the studied system were indexed using the method of structural analogy using single-crystal data of  $\text{PbCd}_2\text{B}_6\text{O}_{12}$  [6]. Crystallographic characteristics based on the results of indexing X-ray diffraction patterns are given in Table 1. All doped borates crystallize in the monoclinic system, sp. gr.  $P2_1/n$ .

In the structure of monoclinic  $\text{PbCd}_2\text{B}_6\text{O}_{12}$  cadmium atoms exhibited double coordination with coordination number (CN) = 6 and 7. Radius of  $\text{Cr}^{3+}$  ion for CN = 6 according to [21] was 0.615 Å, which was slightly less than the radius of the  $\text{Cd}^{2+}$  ion, which for this coordination has a value of 0.65 Å, and for CN = 7 is 0.745 Å. The insignificant content of the dopant and their small size compared to the radius of the substituent ions did not lead to a restructuring of the structure, as evidenced by the lattice parameters of the solid solutions. It can be seen that the resulting phases crystallize

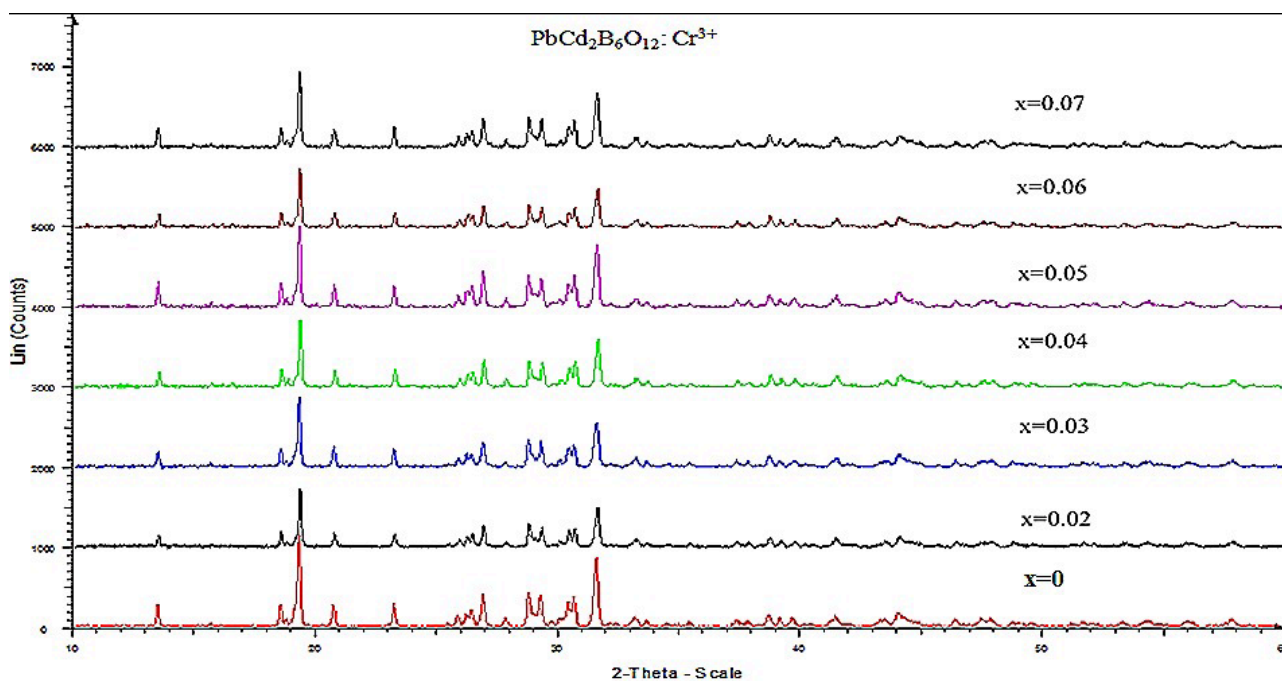


Fig. 2. X-ray diffraction patterns of the  $\text{PbCd}_{2-x}\text{B}_6\text{O}_{12} \cdot x\text{Cr}^{3+}$  system

**Table 1.** Crystallographic and thermal characteristics of phases PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>: xCr<sup>3+</sup> (sp, gr. P2<sub>1</sub>/n, Z = 4)

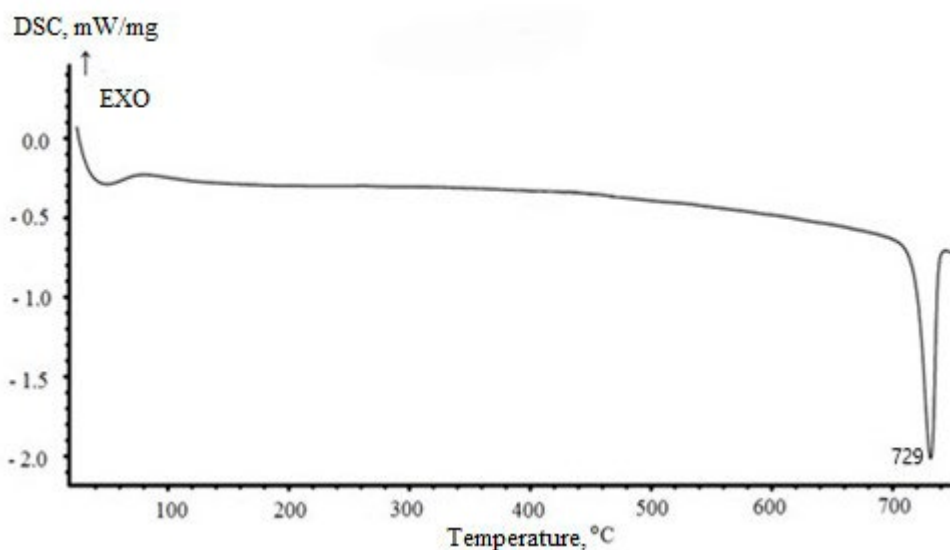
Phase	<i>a</i> , Å	<i>b</i> , Å	<i>c</i> , Å	β, deg.	<i>V</i> , Å <sup>3</sup>	<i>T</i> <sub>melt</sub> , °C
PbCd <sub>2</sub> B <sub>6</sub> O <sub>12</sub> [6]	6.5570(3)	6.9924(4)	19.2094(10)	90.285(4)	880.72(8)	731
PbCd <sub>2</sub> B <sub>6</sub> O <sub>12</sub>	6.5618(3)	6.9868(4)	19.2081(8)	90.250(3)	880.61(7)	734
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.02 Cr <sup>3+</sup>	6.5605(5)	6.9885(9)	19.213(2)	90.253(6)	880.9(2)	
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.03 Cr <sup>3+</sup>	6.5598(6)	6.9861(7)	19.219(1)	90.246(5)	880.8(1)	729
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.04 Cr <sup>3+</sup>	6.5573(6)	6.9855(7)	19.204(2)	90.248(6)	879.7(1)	
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.05 Cr <sup>3+</sup>	6.5567(7)	6.9852(6)	19.217(1)	90.244(6)	880.0(1)	
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.06 Cr <sup>3+</sup>	6.5546(6)	6.9793(7)	19.215(6)	90.228(6)	878.9(1)	
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.07 Cr <sup>3+</sup>	6.5508(5)	6.9801(8)	19.207(2)	90.224(5)	878.3(1)	

in the same structural type as the original matrix, forming a continuous series of PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>: xCr<sup>3+</sup> substitutional solid solutions in the considered concentration range 0 ≤ x ≤ 7.0 mol. %.

In Fig. 3 the heating curve of PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>: 0.03Cr<sup>3+</sup> sample, containing one endothermic effect at 729 °C, corresponding to the melting process of the sample is shown. As the temperature increased, the mass of borate remained constant until it melted. A similar thermal effect was discovered for pure hexaborate PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub> [7].

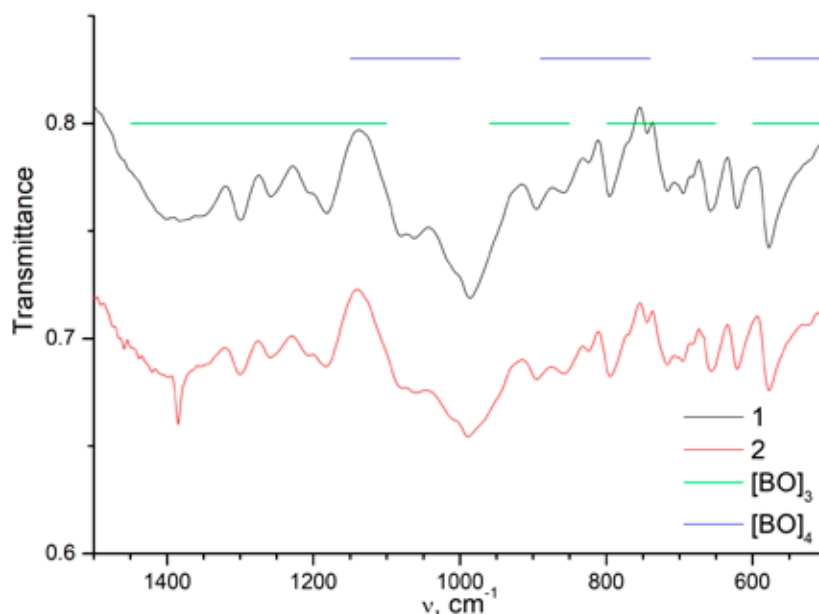
X-ray diffraction pattern of PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>: 0.03Cr<sup>3+</sup> powder melt contained the main reflections of phases identified as CdB<sub>4</sub>O<sub>7</sub>, PbO, pointing at incongruent melting pattern of borate. The XRD results were in agreement with our previously obtained data [7–9]. The melting temperatures of some activated lead and cadmium borates are shown in Table 2.

IR spectra of PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub> (1) and PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>: 0.03 Cr<sup>3+</sup> (2) samples, presented in Fig. 4, contained a large number of bands in the region of 600–1400 cm<sup>-1</sup> and show significant similarities. The complex structure of the boron-oxygen framework of the studied borates did not allow a strict interpretation of the spectra. The similarity in the shape and location of the absorption bands indicated the same type of coordination of boron atoms in both phases and confirmed the XRD results on the similarity of their crystal structures. The bands in the absorption spectra were due to the presence of PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub> BO<sub>3</sub>-triangles and BO<sub>4</sub>-tetrahedra in the structure, vibrations of which are usually observed in the spectral range 400–2000 cm<sup>-1</sup>. Spectrum (2) included intense bands at 1384, 1299, 1183, 989, 795 cm<sup>-1</sup>. Bands with lower intensity were observed at 1260, 1063, 895, 656,

**Fig. 3.** Heating curve of PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>:0.03Cr<sup>3+</sup> sample

**Таблица 2.** Термические характеристики фаз PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>: xCr<sup>3+</sup> (пр. гр. P2<sub>1</sub>/n, Z = 4)

Polycrystals	$T_{\text{melt}}, ^\circ\text{C}$	Melting pattern	Reference
PbCd <sub>2</sub> B <sub>6</sub> O <sub>12</sub>	731	incongruent	[6]
PbCd <sub>2</sub> B <sub>6</sub> O <sub>12</sub>	734	incongruent	[7]
Pb <sub>1-x</sub> Cd <sub>2</sub> B <sub>6</sub> O <sub>12</sub> : 0.03Eu <sup>3+</sup>	732	incongruent	[8]
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.05Cu <sup>2+</sup>	728	incongruent	[9]
PbCd <sub>2-x</sub> B <sub>6</sub> O <sub>12</sub> : 0.03Cr <sup>3+</sup>	729	incongruent	present study

**Fig. 4.** IR spectra of samples PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub> (1) and PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>:0.03Cr<sup>3+</sup> (2)

620, 577, 414 cm<sup>-1</sup>. Interatomic distances B–O in triangles varied within 1.334(9)–1.396(9) Å and were significantly shorter than in tetrahedrons (1.430(9)–1.538(8) Å [6]. Consequently, the high-frequency bands of the spectrum at 1384 cm<sup>-1</sup> and 1299 cm<sup>-1</sup> were caused by antisymmetric stretching vibrations ( $\nu_3$ ) BO<sub>3</sub> groups. Peaks in the lower frequency region (<1200 cm<sup>-1</sup>) were associated with symmetrical stretching vibrations of ( $\nu_1$ )BO<sub>3</sub> and ( $\nu_3$ )BO<sub>4</sub> groups. Absorption bands in the region of 577–795 cm<sup>-1</sup> were due to various deformation vibrations ( $\nu_2$ ) and ( $\nu_4$ ) of corrugated layer [(B<sub>6</sub>O<sub>12</sub>)<sup>6-</sup>]. Observed absorption peaks at 414 cm<sup>-1</sup> can be classified as deformation ( $\nu_2$ ) vibration of bonds in boron-oxygen tetrahedra.

#### 4. Conclusions

Phases of variable composition were obtained by heterovalent substitution of cadmium ions with chromium ions in the double borate PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub> using solid-phase reactions. Substitutional

solid solutions of PbCd<sub>2-x</sub>B<sub>6</sub>O<sub>12</sub>: xCr<sup>3+</sup> system were revealed in the concentration range 0 ≤ x ≤ 7.0 mol %. According to the XRD results, the parameters and volumes of monoclinic cells monotonically decreased with increasing activator content. Changes in the cell parameters of the new phases were consistent with the ionic radii of the activator and the substituted matrix ions. The incongruent melting temperature of PbCd<sub>2</sub>B<sub>6</sub>O<sub>12</sub>: 0.03Cr<sup>3+</sup> was 729 °C.

#### Conflict of interests

The author declares that they has no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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