

# Optical properties of alkali and alkaline earth tetraborate glasses prepared in the alumina crucible

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*Received March 23, 2011*

The optical absorption spectra and refractive index have been recorded in the wavelength range 200–400 nm and 400–700 nm, respectively, for the glasses of the composition  $\text{Li}_2\text{B}_4\text{O}_7$ ,  $\text{LiCaBO}_3$ ,  $\text{CaB}_4\text{O}_7$ ,  $\text{SrB}_4\text{O}_7$  prepared using the normal alloying technique in  $\text{Al}_2\text{O}_3$  crucible. The density, the values of optical band gap for indirect transitions, refractive index, the average molar refraction and electronic polarizability, the average electronic polarizability of the oxide ion and the optical basicity, have been determined.

Получены спектры поглощения и дисперсия показателей преломления в диапазоне 200–400 нм и 400–700 нм, соответственно, стекол с химической формулой  $\text{Li}_2\text{B}_4\text{O}_7$ ,  $\text{LiCaBO}_3$ ,  $\text{CaB}_4\text{O}_7$ ,  $\text{SrB}_4\text{O}_7$ , изготовленных стандартным методом сплавления в  $\text{Al}_2\text{O}_3$  тигле. Определены плотность, оптическая ширина запрещенной зоны для непрямых переходов, показатели преломления, средняя молярная рефракция и электронная поляризованность, средняя электронная поляризованность кислородного иона и оптическая щелочность.

## 1. Introduction

Borate compounds offer a separate wide class of perspective materials in the form of single crystals, polycrystals or glasses. The tetraborates of alkali and alkaline earth elements take an important place among them. Many of the compounds are perspective for nonlinear optics, laser engineering, thermoluminescent dosimetry and radiation detection. Pure and doped by different elements single crystals and powders: lithium, potassium, cesium, strontium, and calcium tetraborates ( $\text{Li}_2\text{B}_4\text{O}_7$ ,  $\text{LiCaBO}_3$ ,  $\text{CaB}_4\text{O}_7$ ,  $\text{SrB}_4\text{O}_7$ ) have already found their practical applications [1–6]. However, borates single crystals growth is technologically difficult and durable process that leads to high value

of the final product. Moreover, in connection with a low growing velocity and a large viscosity of melts serious problems arise with doping.

Since boron is one of the elements, which promotes vitrification of oxides, boric anhydride  $\text{B}_2\text{O}_3$  plays the fundamental role in the modern glass technology, because both stable oxygen coordinations of boron atoms — triangles  $\text{BO}_3$  and tetrahedron  $\text{BO}_4$  — show a tendency to polycondensation [7]. Borate glasses are typical glass systems characterized by the rather large glass-forming region. The structure of borate glasses is particularly interesting because of the so-called boron anomaly and variety of skeleton modification and owing to influ-

ence of nonbridging oxygen or M–O bonds on the optical properties of the glasses [8]. In addition, doping of the borate glasses is not so critical, as for single crystals [9–11]. Thus from the technological point of view, borate glass can be more attractive in comparison with their single crystal analogues. Because the glasses can be prepared in the air atmosphere using comparatively inexpensive ceramic crucibles made from the high-temperature oxides. The crucibles from  $\text{Al}_2\text{O}_3$  ceramics are well applicable by their parameters for preparation of the borate glasses. But oxides at high temperatures can interact one with another, in other words, the borate melts particularly can be dirtied by  $\text{Al}_2\text{O}_3$ . Such uncontrollable doping by the aluminum oxide can influence on the physical properties of the borate glasses, but information about such investigations is absent. Therefore, the given article is devoted to investigation of optical properties of glasses from stoichiometric composition  $\text{Li}_2\text{B}_4\text{O}_7$ ,  $\text{LiCaBO}_3$ ,  $\text{CaB}_4\text{O}_7$  and  $\text{SrB}_4\text{O}_7$  obtained by melting in the corundum crucibles.

## 2. Experimental procedure

The glasses have been prepared using the normal melt quenching technique. Reagent-grade  $\text{Li}_2\text{CO}_3$ ,  $\text{CaCO}_3$ ,  $\text{SrCO}_3$  and boric acid  $\text{H}_3\text{BO}_3$  were used as raw materials. Six grams of mixture, which corresponded to the chemical formula of appropriate glass, were stirred and grinded to obtain a fine powder. The mixture was melted in a  $\text{Al}_2\text{O}_3$  ceramic crucible under ordinary atmospheric conditions at temperature 1270 K for  $\text{Li}_2\text{B}_4\text{O}_7$  and  $\text{LiCaBO}_3$  glasses and at 1400 K for  $\text{MB}_4\text{O}_7$  ( $M = \text{Ca}, \text{Sr}$ ) glasses and melts were homogenized during 2 h. Time and temperature of homogenization have been determined experimentally with the purpose to obtain the glasses of the optical quality. The glasses have been formed by the melt casting to metal form that being at room temperature. After being annealed at 680–730 K for 3 h, the glasses samples were then grinded and optically polished to have the dimensions  $10 \times 7 \times 1 \text{ mm}^3$  for the optical measurements. To check the non-crystallinity of the glass samples, X-ray measurements were performed. The results showed that XRD patterns of the glasses did not contain any discrete or sharp peaks, but broad humps were characteristic for amorphous materials. Content of  $\text{Al}_2\text{O}_3$  in the glasses was determined by means of energodispersive X-ray microanalyzer on

the scanning microscope PEMMA-102-02. Accuracy of  $\text{Al}_2\text{O}_3$  content in glasses was calculated from the results of three melting processes for each composition. The glass transmittance at room temperature was determined by experimental plant, assembled on the base of MDR-23 monochromator and photomultiplier PhEM-100 in the wavelength range of 200–400 nm. A quartz halogen-filled lamp KHM-200 was used as a source of ultraviolet radiation. A personal computer controlled experiment, accumulated and processed the information. The refractive index of glass was measured in the wavelength range of 400–700 nm by refractometric method. The mixture of kerosene and  $\alpha$ -bromonaphthalene was used as an immersion liquid. The quantity  $\Delta n = \pm 0.0005$  was absolute error of measurements of the refractive index.

## 3. Results and discussion

Preparing of glass by melting of corresponding mixture in  $\text{Al}_2\text{O}_3$  crucibles leads to uncontrollable doping of glass owing to particular dissolving of the crucible material  $\text{Al}_2\text{O}_3$  in corresponding oxide melts. This is caused by interaction between oxides at high temperatures, and it is impossible in practice to avoid it. As it is seen from Table 1, concentration of  $\text{Al}_2\text{O}_3$  in glass depends significantly on its chemical composition, and it is much higher in the lithium glasses in comparison with the alkali-earth ones. If to compare the boiling temperatures for alkali (1270 K) and alkali-earth (1400 K) borates, it becomes clear that temperature is not so much critical for interaction between the borate melts and  $\text{Al}_2\text{O}_3$ . Therefore, in our case, one can state that we deal with glasses of chemical compositions  $x\text{Li}_2\text{B}_4\text{O}_7 \times (1-x)\text{Al}_2\text{O}_3$ ,  $y\text{LiCaBO}_3 \times (1-y)\text{Al}_2\text{O}_3$ ,  $z\text{MB}_4\text{O}_7 : (1-z)\text{Al}_2\text{O}_3$  ( $M = \text{Ca}, \text{Sr}$ ) (Table 1).

Table 1. Concentration  $\text{Al}_2\text{O}_3$ , density and optical band gap energy  $E_g^{opt}$  for borate glasses at 293 K

Glass composition	$\text{Al}_2\text{O}_3$ , mol. %	Density, $\text{g/cm}^3$	Optical band gap energy, $E_g^{opt}$ (eV)
$\text{Li}_2\text{B}_4\text{O}_7$	$8.7 \pm 0.5$	2.22	3.88
$\text{LiCaBO}_3$	$8.6 \pm 0.5$	2.47	3.46
$\text{CaB}_4\text{O}_7$	$2.6 \pm 0.3$	2.48	3.61
$\text{SrB}_4\text{O}_7$	$2.9 \pm 0.3$	2.88	3.58

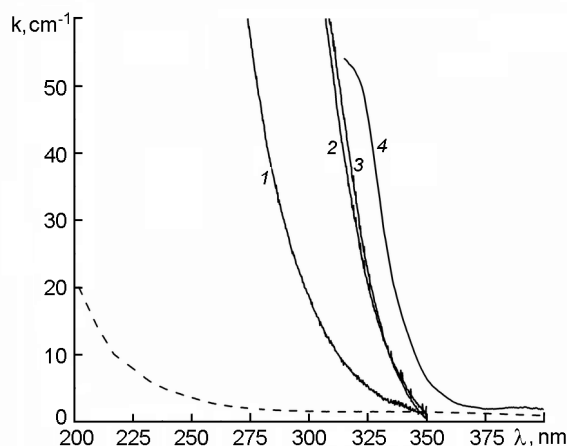


Fig. 1. Optical absorption spectra in the region of the short wavelength edge for borate glasses at 293 K: 1 –  $91.3\text{Li}_2\text{B}_4\text{O}_7\cdot 8.7\text{Al}_2\text{O}_3$ ; 2 –  $97.4\text{CaB}_4\text{O}_7\cdot 2.6\text{Al}_2\text{O}_3$ ; 3 –  $97.1\text{SrB}_4\text{O}_7\cdot 2.9\text{Al}_2\text{O}_3$ ; 4 –  $91.4\text{LiCaBO}_3\cdot 8.6\text{Al}_2\text{O}_3$ ; dotted curve is the absorption spectra of  $\text{Li}_2\text{B}_4\text{O}_7$  glass prepared by the melt quenching technique in carbon crucible.

The optical absorption coefficient  $k$  of the glass samples  $\text{Li}_2\text{B}_4\text{O}_7\text{:Al}_2\text{O}_3$ ,  $\text{LiCaBO}_3\text{:Al}_2\text{O}_3$ ,  $\text{CaB}_4\text{O}_7\text{:Al}_2\text{O}_3$  and  $\text{SrB}_4\text{O}_7\text{:Al}_2\text{O}_3$  is evaluated from the optical transmittance ( $T$ ) and reflectance ( $r$ ) using the relation

$$k = \frac{1}{d} \ln \frac{(1-r)^2}{T}, \quad (1)$$

where  $d$  is the thickness of the sample. Obtained dependency  $k(\lambda)$  for all investigated chemical compositions of borate glasses are presented in Fig. 1. As it is evident from Fig. 1, absorption edge of glasses is much essentially dependent on their chemical composition (the curves 1–4). In Fig. 1 there is also given the absorption spectrum for  $\text{Li}_2\text{B}_4\text{O}_7$  glass of 1 mm thick prepared by melting in graphite crucible in vacuum (dashed line), which differs significantly from the absorption spectrum for  $91.3\text{Li}_2\text{B}_4\text{O}_7\cdot 8.7\text{Al}_2\text{O}_3$  glass, in other words, prepared by melting of  $\text{Li}_2\text{B}_4\text{O}_7$  in crucible from  $\text{Al}_2\text{O}_3$  (curve 1).

Because of the absence of long-range order in glasses it is impossible to use band theory directly owing to the lack of translational symmetry. Thus for such disordered mediums as glasses it is worthy to apply such a universal characteristic of electron states as density of electron states distribution in energy. Long-wavelength shift of absorption edge of glasses in comparison with

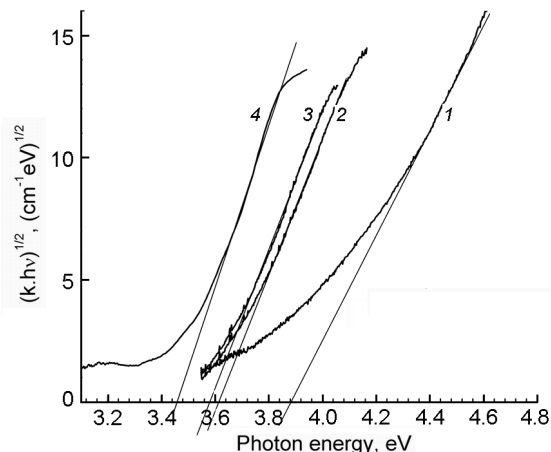


Fig. 2. Absorption edge spectra plot on a graph of  $(k \cdot hv)^{1/2}$  versus  $hv$  for borate glasses: 1 –  $91.3\text{Li}_2\text{B}_4\text{O}_7\cdot 8.7\text{Al}_2\text{O}_3$ ; 2 –  $97.4\text{CaB}_4\text{O}_7\cdot 2.6\text{Al}_2\text{O}_3$ ; 3 –  $97.1\text{SrB}_4\text{O}_7\cdot 2.9\text{Al}_2\text{O}_3$ ; 4 –  $91.4\text{LiCaBO}_3\cdot 8.6\text{Al}_2\text{O}_3$ .

the appropriate single crystals can be attributed to the smearing of the edge density of electron states distribution. But the concept of the band theory can be used even in the case of glasses, however, taking into consideration that direct band-to-band transitions are impossible here, but only indirect transitions with participation of phonons and excitons can be realized. Such indirect optical band energy transitions are considered in detail enough in some articles, in particular [12, 13]. In [14] for amorphous materials, indirect transitions are valid according to the relation, which for single phonon indirect band-to-band transitions looks as

$$\alpha(h\nu) \sim (h\nu - E_g^{opt} \pm h\nu_{ph})^\gamma, \quad (2)$$

where  $E_g^{opt}$  is a value of indirect optical band gap energy,  $h\nu_{ph}$  is the phonon energy,  $\gamma = 2$  for indirect permitted transitions and  $\gamma = 3$  for indirect forbidden transitions.  $E_g$  can be obtained from Eq. (2) by extrapolating of the absorption coefficient to zero absorption in the  $(k \cdot hv)^{1/2}$  coordinates versus  $hv$  plot, that is  $E_g^{opt} = h\nu$  at  $(k \cdot hv)^{1/2} = 0$  [15]. In Fig. 2 absorption spectra of all glasses (as graphs of  $(k \cdot hv)^{1/2}$  versus  $hv$ ) are presented. The values of absorption edge of borate glasses, determined from Fig. 2, are presented in Table 1. The comparison with absorption edges of corresponding single crystals (135–180 nm) demonstrates that for all investigated glasses the essential shift of the absorption edges into long-wave spectral region takes place. Absorption edge of

$\text{Li}_2\text{B}_4\text{O}_7$  glass, prepared by melting in graphite crucible, is also no exclusion. Therefore, on example of  $91.3\text{Li}_2\text{B}_4\text{O}_7\cdot 8.7\text{Al}_2\text{O}_3$  glasses and undoped  $\text{Li}_2\text{B}_4\text{O}_7$  one can state that presence of  $\text{Al}_2\text{O}_3$  in the borate glass shifts significantly the absorption edge into long-wave spectral region. But on example of  $91.3\text{Li}_2\text{B}_4\text{O}_7\cdot 8.7\text{Al}_2\text{O}_3$ , and  $91.4\text{LiCaBO}_3\cdot 8.6\text{Al}_2\text{O}_3$  glasses one can make a conclusion that concentration of  $\text{Al}_2\text{O}_3$  in glass is not definitive.

Differences in behavior of absorption spectra of  $91.3\text{Li}_2\text{B}_4\text{O}_7\cdot 8.7\text{Al}_2\text{O}_3$ ,  $97.4\text{CaB}_4\text{O}_7\cdot 2.6\text{Al}_2\text{O}_3$ ,  $97.1\text{SrB}_4\text{O}_7\cdot 2.9\text{Al}_2\text{O}_3$  and  $91.4\text{LiCaBO}_3\cdot 8.6\text{Al}_2\text{O}_3$  glasses are caused, most probably, by difference between the boron-oxygen complexes, which are the main elements of structure, and from which the frames of our glasses are formed. For example,  $\text{Li}_2\text{B}_4\text{O}_7$  compound is based on  $(\text{B}_4\text{O}_9)^{6-}$  boron-oxygen complexes which consist of elementary complexes which are based on two  $\text{BO}_4$  and two  $\text{BO}_3$  [16],  $\text{LiCaBO}_3$  compound is based on elementary  $(\text{BO}_3)^{3-}$  complexes [17],  $\text{CaB}_4\text{O}_7$  and  $\text{SrB}_4\text{O}_7$  compounds are based on elementary  $(\text{BO}_4)^{4-}$  complexes [18, 19]. As absorption edge in the anhydrous borate single crystals is formed by anion boron-oxygen complexes [20], then it is logically to assume, that the main role in forming the density of electron states distribution as a function of energy (that is displayed in optical absorption spectra of glasses) is played by the same boron-oxygen complexes, which are the main structural elements of borate compounds. Since the boron-oxygen complexes of  $\text{Li}_2\text{B}_4\text{O}_7$ ,  $\text{CaB}_4\text{O}_7$  and  $\text{SrB}_4\text{O}_7$  compounds form frame structures of their lattices [16, 18, 19], the structure of such glasses must be like a disfigured boron-oxygen frame of corresponding crystal. In  $\text{LiCaBO}_3$  glass the elementary boron-oxygen complexes do not form a frame, in result of which they are located more chaotically in the structure of this glass.

Revealed influence of  $\text{Al}_2\text{O}_3$  on the absorption spectra of the borate glasses occurs, most probably, because of additional deformation of structure and increasing of imperfection of glass. For example, in publication [21] it was ascertained that presence of aluminum oxide in the borate glass leads to increasing of number of  $\text{BO}_3$  polyhedrons, whereas amount of boron with tetrahedral coordination decreases. Therefore, disorder in corresponding borate glasses with  $\text{Al}_2\text{O}_3$  grows, and their imperfection increases. All this leads to complication of

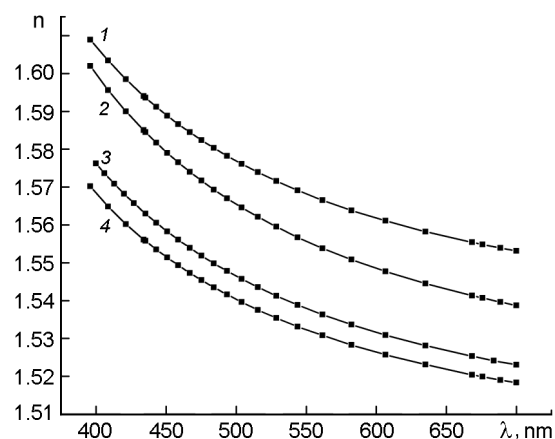


Fig. 3. Dispersion of refractive index borate glasses: 1 –  $97.1\text{SrB}_4\text{O}_7\cdot 2.9\text{Al}_2\text{O}_3$ ; 2 –  $97.4\text{CaB}_4\text{O}_7\cdot 2.6\text{Al}_2\text{O}_3$ ; 3 –  $91.4\text{LiCaBO}_3\cdot 8.6\text{Al}_2\text{O}_3$ ; 4 –  $91.3\text{Li}_2\text{B}_4\text{O}_7\cdot 8.7\text{Al}_2\text{O}_3$ .

phonon spectrum of glasses and electron-phonon interaction, and, adequately, to shift of the absorption edge.

In Fig. 3 experimental dispersion spectra  $n(\lambda)$  for all glasses in the range 400–700 nm, measured at 293 K are presented. All dispersion curves in Fig. 3 measured at room temperature are well approximated by single-oscillator Sellmeier formula:

$$n^2 = 1 + \frac{A\lambda^2\lambda_0^2}{\lambda^2 - \lambda_0^2}, \quad (3)$$

where  $\lambda_0$  — wavelength of the maximum of effective absorption band; fitting parameter  $A = Ne^2f/pmc^2$  — is related with the oscillator force  $f$ . Other symbols denote  $e$  and  $m$  — the charge and the mass of an electron, respectively,  $N$  — the number of particles in the unit of volume,  $c$  — the speed of light in vacuum.

Using mathematical analysis of experimental results for each glass compound we have calculated the parameters  $A$  and  $\lambda_0$  from Sellmeier formula (3) for dispersion curves, described in the first approximation by single oscillator model. The results are presented in Table 2. According to Lorentz-Lorenz equation, the density of the material affects the refractive index in direct proportion. The data comparison from Table 1 and 2 reveals, that this rule is observed for all investigated compositions of glasses.

It is known that optical non-linearity is caused by the electronic polarization of materials [22]. Therefore, the average electronic polarizability of ions is considered to be one of the most important properties of

Table 2. Refractive index  $n$ , Sellmeier relation  $A$  and  $\lambda_0$ , refraction  $R_m$ , electron polarizability  $\alpha_m$  and  $\alpha_{O^{2-}}$  and optical basicity  $B$  for borate glasses at 293 K and  $\lambda = 632.8$  nm

Glass composition	$n$	$A \cdot 10^{-5}, \text{nm}^{-2}$	$\lambda_0, \text{nm}$	$R_m, \text{cm}^3$	$\alpha_m \cdot 10^{-24}, \text{cm}^3$	$\alpha_{O^{2-}} \cdot 10^{-24}, \text{cm}^3$	$B$
$\text{Li}_2\text{B}_4\text{O}_7$	1.524	5.2145	154.38	23.314	9.25	1.312	0.397
$\text{LiCaBO}_3$	1.528	5.0035	158.20	12.314	4.89	1.441	0.511
$\text{CaB}_4\text{O}_7$	1.544	4.6818	166.04	24.967	9.91	1.337	0.421
$\text{SrB}_4\text{O}_7$	1.557	5.5131	156.01	27.,244	10.77	1.394	0.473

glass, which is closely related to their applicability in the field of optics and electronics. The molar electronic polarizability of the material is proportional to the molar refraction through the Clausius-Mosotti equation:

$$\alpha_m = (3/4\pi N_0)R_m, \quad (4)$$

where  $N_0$  is the Avogadro's number and  $R_m = [(n^2 - 1)/(n^2 + 2)]M/\rho$  — the average molar refraction,  $M$  — the molar mass,  $\rho$  — the density.

Calculated on the basis of the values of the refractive indices by these formulas values of  $\alpha_m$  and  $R_m$  for all glasses compositions are presented in Table 2. In addition there are given the values of electronic polarizability of oxide ion calculated using the equation [23]:

$$\alpha_{O^{2-}} = [(R_m/2.52 - \sum \alpha_{cat}]/N_{O^{2-}}, \quad (5)$$

where  $\sum \alpha_{cat} = p\alpha_{(\text{Li}^+, \text{K}^+, \text{Cs}^+, \text{Ca}^{2+}, \text{Sr}^{2+})} + q\alpha_{\text{B}_3}$ ,  $N_{O^{2-}}$  — the number of oxide ions in the chemical formula of glass.

As it is evident from Table 1 and 2, for considered glasses there is no observed correlation between electronic polarizability of oxide ion and the refractive index. In the last column of Table 2, there are presented the values of optical basicity  $B$  in the wavelength 632.8 nm calculated by the following formula [24].

$$B = 1.67(1 - 1/\alpha_{O^{2-}}). \quad (6)$$

According to this equation, an increase in the polarizability increases the optical basicity. The optical basicity proposed by Duffy et al. [25] was used as a measurement of acid-base properties of the oxide glasses because an intrinsic relationship exists between the electronic polarizability of the oxide ions and the optical basicity of the oxide medium.

#### 4. Conclusions

The optical absorption spectra and refractive index in the wavelength range 200–400 nm and 200–700 nm respectively have been investigated for the glasses with compositions  $\text{Li}_2\text{B}_4\text{O}_7$ ,  $\text{LiCaBO}_3$ ,  $\text{CaB}_4\text{O}_7$  and  $\text{SrB}_4\text{O}_7$  prepared using the normal alloying technique in  $\text{Al}_2\text{O}_3$  crucible. The density, the values of optical band gap, the average molar refraction and electronic polarizability, the average electronic polarizability of the oxide ion and the optical basicity have been determined. Optical absorption edge of all investigated glasses is shifted significantly into long-wave spectral region that is caused, most probably, by influence of  $\text{Al}_2\text{O}_3$  on their structure and imperfection. Refractive indices for all compositions of the investigated glasses on the fixed wavelength, are less, then for corresponding single crystals and there is no correlation between electronic polarizability of oxide ion and the refractive index.

*Acknowledgements.* This work was supported by the Ministry of Education and Science of Ukraine, Project 0109U001063.

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## Оптичні властивості лужних і лужноземельних тетраборатних стекел, одержаних у тиглях з оксиду алюмінію

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Отримано спектри поглинання і дисперсію показників заломлення у діапазоні 200–400 нм і 400–700 нм, відповідно, стекел з хімічною формулою  $\text{Li}_2\text{B}_4\text{O}_7$ ,  $\text{LiCaBO}_3$ ,  $\text{CaB}_4\text{O}_7$ ,  $\text{SrB}_4\text{O}_7$ , одержаних стандартним методом сплавлення в  $\text{Al}_2\text{O}_3$  тиглі. Визначено густину, оптичну ширину забороненої зони для непрямих переходів, показники заломлення, середню молярну рефракцію і електронну поляризованість, середню електронну поляризованість кисневого іона і оптичну лужність. Проведено аналіз і обговорення результатів.