

## LUMINESCENCE AND SCINTILLATION PROPERTIES OF Cs<sub>3</sub>ZnCl<sub>5</sub> AND Cs<sub>3</sub>ZnCl<sub>5</sub>(Eu) SINGLE CRYSTALS

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This paper reports the results on obtaining and investigation of functional parameters of Cs<sub>3</sub>ZnCl<sub>5</sub> crystals grown using Bridgman method from the melts of 3CsCl-2ZnCl<sub>2</sub> and 3CsCl-1.995ZnCl<sub>2</sub>-0.005 EuCl<sub>2</sub> compositions. The study of photoluminescence spectra obtained at 237 nm excitation shows the presence of the following emission bands: one at 325 nm caused by the defects or impurities, slightly pronounced band at ca. 450 nm caused by the presence of Eu<sup>2+</sup> ions and a series of bands in 590–700 nm range due to the presence of Eu<sup>3+</sup> in the crystals. The presence of said Eu ions is confirmed by the luminescent studies with the use of excitation at wavelengths proper to Eu<sup>2+</sup> (340 nm) and Eu<sup>3+</sup> (465 nm). X-ray luminescence spectra include the bands with maxima of 235 nm and 285 nm which are caused by the core-valence luminescence, the band with the maximum at 320 nm caused by the defects and impurities and two bands with the maxima at 400 and 520 nm which nature is not clear (probably, it can be connected with the presence of Eu<sup>3+</sup> in the samples). The study of light yield performed at the light collection time of 2 μs showed that for all the samples its value is ca. 6% vs. BGO crystal (Bi<sub>4</sub>Ge<sub>3</sub>O<sub>12</sub>) and the form of the pulse-height spectra for the crystals grown from both melts practically coincide. This leads to the conclusion that the transfer of excitation from the matrix to Eu<sup>2+</sup> ions is absent and, according to the photoluminescence studies it can be assumed that Eu<sup>2+</sup> exists in the said crystals as inclusions of CsCl:Eu<sup>2+</sup> solid solution.

**Keywords:** cesium chloride, zinc chloride, europium chloride, luminescence, scintillation, light yield.

### Introduction

The recent progress of material science of halide scintillation materials is connected with the development of rare-earth activated (Eu<sup>2+</sup>, Ce<sup>3+</sup>) simple and complex compounds and their solid solutions. Some of the recently discovered materials are already established trademarks (LaCl<sub>3</sub>:Ce<sup>3+</sup> – BriLanCe<sup>TM</sup>350 [1, 2] and LaBr<sub>3</sub>:Ce<sup>3+</sup> – BriLanCe<sup>TM</sup>380 [2, 3]) and others (SrI<sub>2</sub>:Eu<sup>2+</sup> [4] and Cs<sub>2</sub>LiYCl<sub>6</sub>:Ce<sup>3+</sup> (CLYC) [5]) are efficiently developed and also are commercially available [6,7]. The said materials are solid solutions of halides formed by perfectly isomorphic cations, e.g., LaCl<sub>3</sub>:Ce<sup>3+</sup> means La<sub>1-x</sub>Ce<sub>x</sub>Cl<sub>3</sub> and so on.

The search for new activated halide luminescent materials is performed among solid solutions formed by restrictedly isomorphic cations, e.g., for Eu<sup>2+</sup>-activated materials compounds formed by Ca<sup>2+</sup> [8] and Mg<sup>2+</sup> [9] have been investigated.

This work presents an attempt to obtain Eu<sup>2+</sup>-activated material on the basis of Cs<sub>3</sub>ZnCl<sub>5</sub> compound formed by Zn<sup>2+</sup> cation restrictedly isomorphic to Eu<sup>2+</sup>. According to [10] the difference of the

electronegativities of Eu and Zn is 0.4 that is the limiting value for the perfect isomorphism (The Goldschmidt rule), as for the difference of the ionic radii [11] it considerably exceeds 15%.

It should be noted that the said non-activated material is also referred to scintillators for detection of X-ray irradiation [12].

The results of our study are presented below.

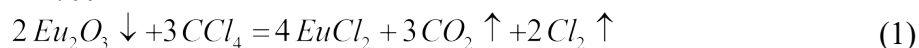
### Experimental part

**Reagents.** Extra pure cesium chloride was used for the crystal growth of crystals on the basis of Cs<sub>3</sub>ZnCl<sub>5</sub>.

Zinc chloride was obtained by the dissolution of metallic zinc of reagent grade in extra pure hydrochloric acid according to well-known routine [13]. Obtained ZnCl<sub>2</sub> was grinded in glow box and kept there.

Charge for the growth of Cs<sub>3</sub>ZnCl<sub>5</sub> was prepared by mixing of CsCl with ZnCl<sub>2</sub> in glow box in the mass ratio of 3,71:1.

The charge for the preparation of the growth melt of Cs<sub>3</sub>ZnCl<sub>5</sub> melt with addition of 0.5 mol. % of Eu<sup>2+</sup> with respect to Zn<sup>2+</sup> was prepared in such a manner. Primarily solid solution of CsCl-EuCl<sub>2</sub> composition was prepared by the dissolution of 0.0882 g of Eu<sub>2</sub>O<sub>3</sub> in 50 g of molten CsCl by the carbohalogenation process at 700 °C:



The primarily formed suspension of Eu<sub>2</sub>O<sub>3</sub> disappeared after 1h treatment the melt became transparent and further it was treated for 1 h, cooled, grinded and kept in glow box. The temperature of the carbohalogenation provided practically complete reduction of EuCl<sub>3</sub> into EuCl<sub>2</sub>.

For the preparation of Cs<sub>3</sub>ZnCl<sub>5</sub>:0.5 mol. % of Eu<sup>2+</sup> charge 19.72 g of CsCl:Eu<sup>2+</sup> powder was mixed in the glow box with 5.282 g of ZnCl<sub>2</sub>.

**The growth procedure.** The charge was placed in quartz ampoule of 12 mm diameter and 500 mm height. Before the growth the ampoule with the charge was kept under vacuum (1 Pa) for 24 h at 700 °C.

The growth of the crystals was performed by Bridgman method in two-zone furnace, the difference of temperature between the zones was 80 °C, temperature gradient was ca. 40 °C/cm, the rate of the dropping of the ampoule was 3.2 mm/h. After the finishing of the ampoule broaching the furnace was cooled to room temperature during 72 h.

After the cooling, ampoule was placed in the glow box where it was broken down and the ingot was removed. The transparent part of the ingot was cut, polished and kept in polyethylene batch in the glow box. The samples presented cylinders of 6÷10 mm diameter and 1 mm height.

The obtained samples were practically non-hygroscopic, therefore, they were not packed into containers at all the investigations.

**Investigation of the obtained samples.** The phase composition of the parts of the obtained ingot was determined using X-ray diffractometer DRON with Cu-K $\alpha$  radiation.

The study of luminescent properties of the obtained samples was performed using a combined fluorescent lifetime and steady-state spectrometer FLS 920 (Edinburgh Instruments) equipped with a xenon Xe 450 W and hydrogen filled nF 900 nanosecond flashlamp for time correlated single photon counting measurements. Photoluminescence excitation (PLE) spectra were corrected on the incident photon flux. Photoluminescence emission (PL) spectra were corrected for the spectral sensitivity of the detection system.

The spectra of X-ray excited luminescence were obtained under X-ray (Cu, 40 kV, 40  $\mu$ A) excitation in transmission mode and were not corrected for the spectral sensitivity of the detection system.

Scintillation decay time profiles of Cs<sub>3</sub>ZnCl<sub>5</sub>-based samples were obtained as follows. The samples were placed on the photocathode of Hamamatsu R6231 PMT. Scintillations were excited with 662 keV gammas from <sup>137</sup>Cs source. Signal from PMT anode was fed to the input of Rigol DS6064 oscilloscope. Decay curves were calculated by averaging of several hundreds of recorded pulses with amplitudes approximately corresponding to the full absorption peak.

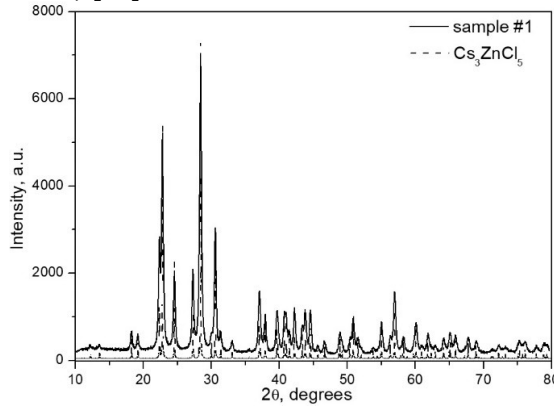
The light yield and the energy resolution of the obtained samples were determined by the method of comparison [14]. The measurements were recorded using a pulse processing chain consisting of an

R1307 SU 0192 PMT (Hamamatsu, Japan), a charge-sensitive preamplifier BUS 2-95, a custom shaping amplifier and a multichannel analyzer AMA-03F. Relative light yield was determined by comparing the peak position (abscissa) of the grown crystals and that of a BGO ( $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ ) crystal (10,000 photons/MeV and energy resolution of 10 %). All measurements were done under the same conditions using the shaping time of 8  $\mu\text{s}$ . The error of the light yield and energy resolution measurements was less than 5 %.

### Results and discussion

As it was mentioned in the previous section all the ingots consisted of lower opaque part and upper transparent one. Taking into account that  $\text{Eu}^{2+}$  is slightly isomorphic to  $\text{Zn}^{2+}$  we performed examination of both parts of the ingot. The X-ray diffraction patterns of the said samples coincide.

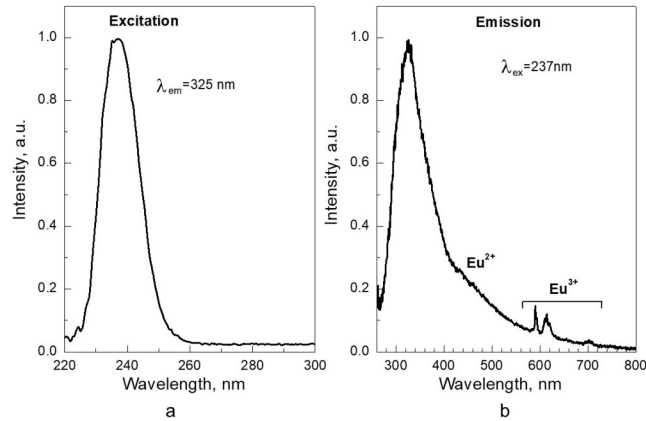
The X-ray diffraction pattern of the samples grown from  $\text{Cs}_3\text{ZnCl}_5$  melt containing  $\text{EuCl}_2$  is presented in Fig. 1. It is seen that the ingots consist of  $\text{Cs}_3\text{ZnCl}_5$  (tetragonal unit cell,  $a=0.926$  nm,  $c=1.450$  nm, space group I4/mcm) [15].



**Figure 1.** The X-ray diffraction pattern of the sample grown from  $\text{Cs}_3\text{ZnCl}_5$  (Eu) melt (thick line) and the database data for  $\text{Cs}_3\text{ZnCl}_5$  (thin line)

It means that there was no selective accumulation in the opaque part of  $\text{CsCl}$  or its compounds with  $\text{EuCl}_2$  of  $\text{CsEuCl}_3$  composition (tetragonal unit cell,  $a=0.5588(4)$  nm,  $c=0.5619(8)$  nm, space group  $P4$  mm) [16]. It may be concluded that  $\text{Eu}^{2+}$  is distributed throughout all the ingot.

The excitation and photoluminescence spectra of the transparent part of the sample are presented in Fig. 2.



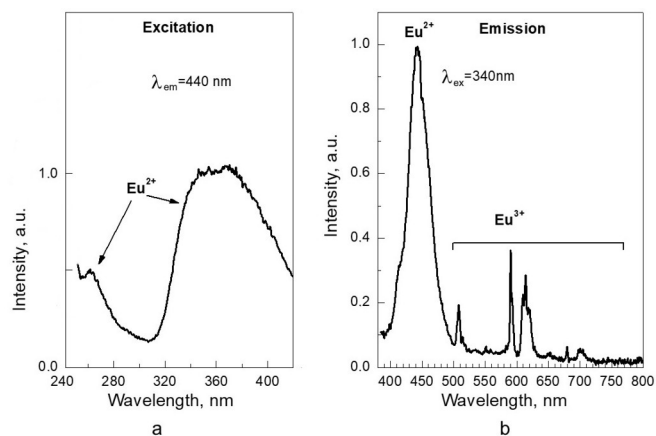
**Figure 2.** Excitation (a,  $\lambda_{em}=325$  nm) and emission (b,  $\lambda_{ex}=237$  nm) spectra of sample grown from  $\text{Cs}_3\text{ZnCl}_5$  (Eu) melt

It can be seen that the main luminescence band at excitation wavelength of 237 nm is placed at 310 nm. Authors [12] found that similar band with the emission band 290 nm was obtained under excitation at 70÷90 nm and it was ascribed to Auger-free luminescence (AFL). In [17] the same authors studied luminescence of crystals of  $\text{Cs}(\text{Ca}_{1-x}\text{Mg}_x)\text{Cl}_3$  composition and found that at the excitation at 240 nm the luminescence for all the studied materials is observed in 350÷390 nm range. Taking into account the excitation wavelength (240 nm) the authors assumed that this luminescence is

caused by defects or impurity sites. It is very probable that the emission band at 310 nm is also due to impurities or defects. The decay profile for the luminescence excited at 237 nm is monoexponential and the decay constant is estimated as 260 ns.

The beside of the above-discussed band in the emission spectra there is also the band proper to  $\text{Eu}^{2+}$  ( $\sim 430$  nm, slightly pronounced) and  $\text{Eu}^{3+}$  (three bands in 590–700 nm region).

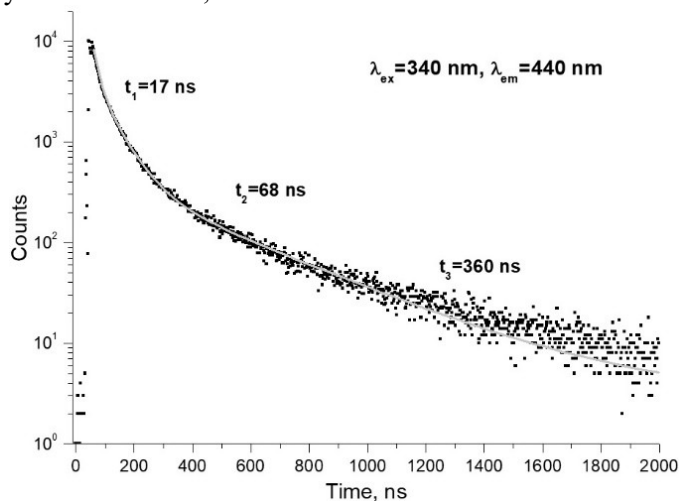
To check whether the 430 nm band is caused by the presence of  $\text{Eu}^{2+}$  we recorded the photoluminescence spectra using excitation at 340 nm proper for  $\text{Eu}^{2+}$ . These spectra are shown in Fig. 3.



**Figure 3.** Excitation (a,  $\lambda_{em}=440$  nm) and emission (b,  $\lambda_{ex}=340$  nm) spectra of sample grown from  $\text{Cs}_3\text{ZnCl}_5(\text{Eu})$  melt

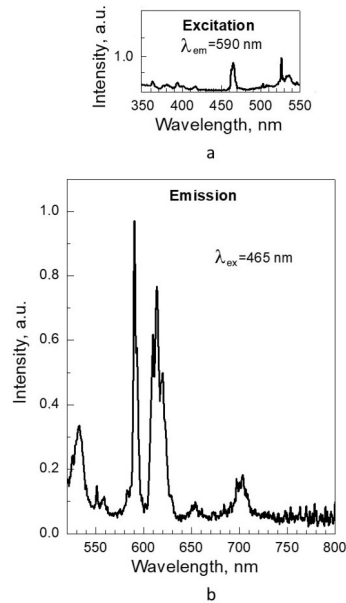
The excitation spectrum includes bands at 260 nm and wide band at 330–400 nm that is proper to excitation of  $\text{Eu}^{2+}$  ions and the emission band at 442 nm. We can assume that the 442 nm band can be ascribed to the formation of  $\text{CsCl}:\text{Eu}^{2+}$  solid solution since the same wavelength was obtained by the authors of [18].

As for the luminescence decay curve, it is presented in Fig. 4 and can be described by three components with decay constants of 17, 68 and 360 ns.



**Figure 4.** The decay curve ( $\lambda_{ex}=340$  nm,  $\lambda_{em}=440$  nm) for the sample grown from  $\text{Cs}_3\text{ZnCl}_5(\text{Eu})$  melt

It is interesting that authors [18] estimated the decay constant of longer component as 360 ns. As for the presence in the sample of  $\text{Eu}^{3+}$ , it is easily confirmed by the data of Fig. 5.

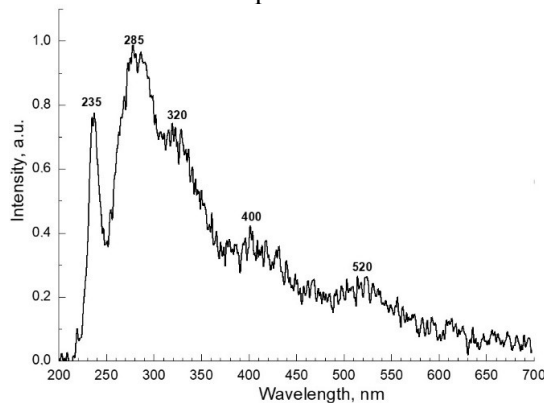


**Figure 5.** Excitation (a,  $\lambda_{em}=590$  nm) and emission (b,  $\lambda_{ex}=465$  nm) spectra of sample grown from  $Cs_3ZnCl_5$  (Eu) melt

The emission spectrum includes bands at 590, 612 and 700 nm which are referred to luminescence of  $Eu^{3+}$ . So, at least the traces of  $EuCl_3$  are present in the sample. This is proper to other chloride materials doped with  $Eu^{2+}$  since the decomposition temperature of  $EuCl_3$  to  $EuCl_2$  is close to 700 °C and even long-term keeping of the growth melt under vacuum does not provide the complete decomposition of  $EuCl_3$  to  $EuCl_2$ .

Now let us consider results connected with the scintillation properties of the obtained crystals. The X-ray luminescence spectrum is presented in Fig. 6.

According to [12] the bands with maxima of 235 nm and 285 nm can be referred to core-valence luminescence, the band with the maximum at 320 nm according to the description of Fig. 1 is caused by the defects and impurities. As for the band with the maximum at 520 nm its nature is unknown, it may be caused by the presence of  $Eu^{3+}$  in the samples.

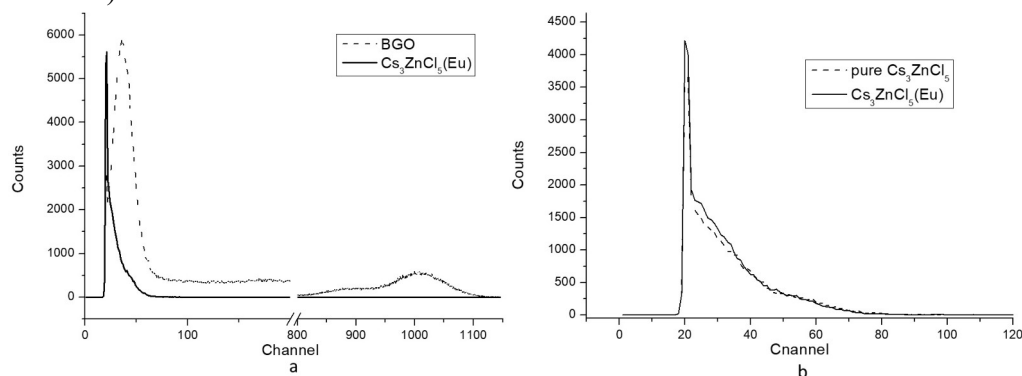


**Figure 6.** X-ray luminescence spectrum of sample grown from  $Cs_3ZnCl_5$  (Eu) melt (reflectance mode, silver anode,  $I=40$   $\mu$ A,  $U=40$  kV)

As for the light yield and energy resolution the light collection time of 2  $\mu$ s was chosen going from the luminescence decay constants, the pulse-height spectra of the obtained crystals are presented in Fig. 7.

From Fig. 7a it follows that the light yield of  $Cs_3ZnCl_5(Eu)$  is equal to 5.8 % against BGO, i.e., *ca.* 580 photons per MeV. As for undoped  $Cs_3ZnCl_5$  its light yield achieves 6 % against BGO (600 photons MeV), that agrees with the data of [12]. Due to complex structure of the photopeaks of the obtained crystals (Fig. 7b) any estimations of the energy resolution would be incorrect.

So, the light yield of Cs<sub>3</sub>ZnCl<sub>5</sub> is not dependent on the presence or absence of Eu<sup>2+</sup> ions in the sample and the pulse-height spectra are identical (Fig.7b). It means that the transfer of the excitation from matrix to Eu<sup>2+</sup> ion is negligible or absent due to restricted isomorphism of Eu<sup>2+</sup> and Zn<sup>2+</sup> ions, which is confirmed by the data of Fig. 6. The most possible form of Eu<sup>2+</sup> existence in the grown crystals considered in this paper may be CsCl:Eu<sup>2+</sup> inclusions or traces of CsEuCl<sub>3</sub> (see Fig. 3 and description for it).



**Figure 7.** Pulse height spectra of: 1 – Cs<sub>3</sub>ZnCl<sub>5</sub>:(Eu) sample vs. BGO (Bi<sub>4</sub>Ge<sub>3</sub>O<sub>12</sub>, standard), 2 – a comparison of the spectra for undoped Cs<sub>3</sub>ZnCl<sub>5</sub> and Cs<sub>3</sub>ZnCl<sub>5</sub>:(Eu)

### Conclusions

In this study crystals on the basis of Cs<sub>3</sub>ZnCl<sub>5</sub> were grown by Bridgman method from the melts of 3CsCl-2ZnCl<sub>2</sub> and 3CsCl-1.995ZnCl<sub>2</sub>-0.005 EuCl<sub>2</sub> compositions.

The luminescence spectra of the Cs<sub>3</sub>ZnCl<sub>5</sub> samples obtained at  $\lambda_{\text{ex}}=237$  nm include bands caused by defects or admixtures (325 nm), Eu<sup>2+</sup> ions (450 nm) and Eu<sup>3+</sup> ions (in the range of 590–700 nm). The presence of both Eu ions was confirmed by luminescence investigations at excitation at wavelengths proper for the corresponding ions. The X-ray luminescence spectra did not include the bands proper for Eu ions.

The study of light yield performed at the light collection time of 2  $\mu$ s showed that the light yield of the crystals grown from both melts is ca. 6% vs. BGO (Bi<sub>4</sub>Ge<sub>3</sub>O<sub>12</sub>) and the shapes of the pulse-height spectra are practically the same that confirms the negligible excitation transfer from the matrix to Eu<sup>2+</sup> ions. Taking into account the results of photoluminescence studies it can be assumed that Eu<sup>2+</sup> ions exist in the grown Cs<sub>3</sub>ZnCl<sub>5</sub> crystals as inclusions of CsCl:Eu<sup>2+</sup> solid solution.

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Received 11.09.2023

Accepted 17.11.2023

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Наведено результати робіт з отримання і дослідження функціональних властивостей кристалів  $\text{Cs}_3\text{ZnCl}_5$ , вирощених з розплавів складів  $3\text{CsCl}-2\text{ZnCl}_2$  і  $3\text{CsCl}-1.995\text{ZnCl}_2-0.005\text{EuCl}_2$ . У спектрах фотолюмінесценції кристалів, одержаних при  $\lambda_{\text{em}}=237$  нм, спостерігається смуга з максимумом при 325 нм, яка може бути обумовлена наявністю дефектів і домішок, слабка смуга при ~450 нм, що відноситься до люмінесценції  $\text{Eu}^{2+}$ , а також серія смуг в інтервалі 590-700 нм, які відносяться до люмінесценції  $\text{Eu}^{3+}$ . Присутність іонів європію Eu було доведено дослідженнями люмінесценції при збудженні на довжинах хвиль притаманних для  $\text{Eu}^{2+}$  (340 нм) і  $\text{Eu}^{3+}$  (465 нм). Спектри рентгенолюмінесценції містять смуги з максимумами при 235 нм і 285 нм, що відповідають остовно-валентній люмінесценції, при 320 нм (можливо, дефекти і домішки) і дві смуги з максимумами при 400 і 520 нм, природа яких не з'ясована (можливо, вони пов'язані з присутністю іонів  $\text{Eu}^{3+}$  у зразках). Дослідження світлового виходу одержаних кристалів, проведені при часі світлозбирання 2 мкс, показали, що кристали обох складів мають світловихід приблизно 6% відносно кристалів BGO ( $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ ), а форма їх амплітудних спектрів практично однакова, що свідчить про незначний перенос збудження від матриці до іонів  $\text{Eu}^{2+}$ . Виходячи з результатів дослідження фотолюмінесценції, можна припустити, що  $\text{Eu}^{2+}$  існує у вирощених кристалах у вигляді включень твердого розчину  $\text{CsCl}:\text{Eu}^{2+}$ .

**Ключові слова:** хлорид цезію, хлорид цинку, хлорид європію, люмінесценція, сцинтиляційні властивості, світловий вихід.

Надіслано до редакції 11.09.2023

Прийнято до друку 17.11.2023

Kharkiv University Bulletin. Chemical Series. Issue 41 (64), 2023