

## Spectroscopic Properties of $\text{KPb}_2\text{Cl}_5$ and $\text{RbPb}_2\text{Br}_5$ Doped with $\text{Er}^{3+}$ and $\text{Yb}^{3+}$

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**Abstract**—Alkali halide crystals doped with  $\text{Er}^{3+}$  exhibit several properties that make them promising for practical use in optical fibers and converters of the emission spectrum. In this work, we studied the luminescence spectra of crystals as a function of the excitation radiation wavelength and the temperature dependences of the luminescence intensity in the up-conversion mechanism of excitation of the most intense lines (530, 550, and 650 nm) and upon excitation at 980 nm. In  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$ ,  $\text{RbPb}_2\text{Br}_5:\text{Er}^{3+}$ , and  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+} + \text{Yb}^{3+}$  crystals, the luminescence kinetics were measured upon excitation at 313 and 365 nm, and the theoretical estimates of lifetimes were made using the Judd–Ofelt theory. The results can be used in the development of laser-based systems and for estimating the degree of approximation of calculations by the Judd–Ofelt method for these crystals.

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### 1. INTRODUCTION

Alkali lead halide crystals,  $(\text{K-Rb})\text{Pb}_2(\text{Cl-Br})_5$ , that are promising as laser media were investigated. These crystals are technologically advanced, have high chemical resistance and a vast transparency region, have a somewhat loose structure that enables rare earth ions (REIs) of type  $\text{Dy}^{3+}$ ,  $\text{Tb}^{3+}$ ,  $\text{Yb}^{3+}$ ,  $\text{Er}^{3+}$ , or  $\text{Nd}^{3+}$  to be included in it. They can effectively serve as converters of light emission from one wavelength range to another because of the narrowness of their phonon spectrum ( $\sim 200 \text{ cm}^{-1}$  for  $\text{KPb}_2\text{Cl}_5$  and  $144 \text{ cm}^{-1}$  for  $\text{RbPb}_2\text{Br}_5$ ). The crystals can be grown large enough and have a high optical quality for use in solid-state lasers. Optical transitions in  $\text{KPb}_2\text{Cl}_5:\text{RE}^{3+}$  crystals are characterized by high oscillator strengths and a large number of radiative transitions [1].

Up-conversion pumping can be used to obtain laser radiation from high excited REI states when pumped by a laser diode (LD). Up conversion of the excitation energy during LD pumping is possible both as a result of stepwise excitation (successive absorption of the pump energy from the ground and excited states) and in the nonradiative interaction of the excited states of the activator ions. These effects yield laser radiation in the visible region [2] and significantly increase the efficiency of laser radiation in the IR region [3]. From this point of view, crystals with

narrow phonon spectra, in which the loss for nonradiative relaxation is minimal, can be considered as the most effective with appropriate selection of the emission spectrum of laser diodes.

### 2. EXPERIMENTAL

$\text{KPb}_2\text{Cl}_5$  crystals were grown by the Stockbarger method in the Institute of Geology and Mineralogy of the Siberian Branch of the Russian Academy of Sciences (Novosibirsk). The initial potassium and lead chlorides (high-purity grade) were subjected to multiple purifications by zone melting in the presence of a chlorine agent.  $\text{KPb}_2\text{Cl}_5$  crystals were grown in evacuated quartz ampoules. The growth unit was a vertical two-zone furnace operated with a temperature gradient of approximately  $50^\circ\text{C}/\text{cm}$ . The crystal growth rate was 2–4 mm/week. The size of the crystals obtained was 15 mm in diameter and 30 mm in length [4].

The photoluminescence (PL) and photoluminescence excitation spectra were measured using a DDS-400W deuterium lamp and a DMR-4 double prism monochromator; a GaAs laser diode (980 nm, 100 mW) was used for the excitation of anti-Stokes luminescence (ASL). The photoluminescence was recorded using an MDR-23 monochromator and an R6358-10 photomultiplier (Hamamatsu). The photo-

luminescence excitation spectra were normalized to an equal number of photons incident on a sample using Lumogen Yellow, which has a constant quantum yield in the spectral range under study. The photoluminescence decay kinetics were measured using a pulsed xenon lamp (FWHM = 1  $\mu$ s, frequency 5 Hz). X-ray luminescence (XRL) spectra were measured using a URS-55A apparatus (Cu, 30 kV, 10 mA), an MDR-23 monochromator, and an FEU-106 photomultiplier as a detector.

The application of the Judd–Opelt theory for calculating the luminescence times in  $\text{KPb}_2\text{Cl}_5$  crystals doped with  $\text{Er}^{3+}$  ion was considered, and calculation corrections were introduced for  $\text{RbPb}_2\text{Br}_5$  crystals (the crystals have a structure similar to  $\text{KPb}_2\text{Cl}_5$ , which simplifies the necessary corrections). The calculation of lifetimes for crystals doped simultaneously with  $\text{Er}^{3+}$  and  $\text{Yb}^{3+}$  is complicated, because it requires the application of the method of model quantum-mechanical calculation, namely, the determination of energy transfer between ions, which was not taken into account in the method used. A more detailed description of the method can be found in [5]. The oscillator strengths of the magnetic dipole interaction, which are necessary for applying the method, are taken from [6].

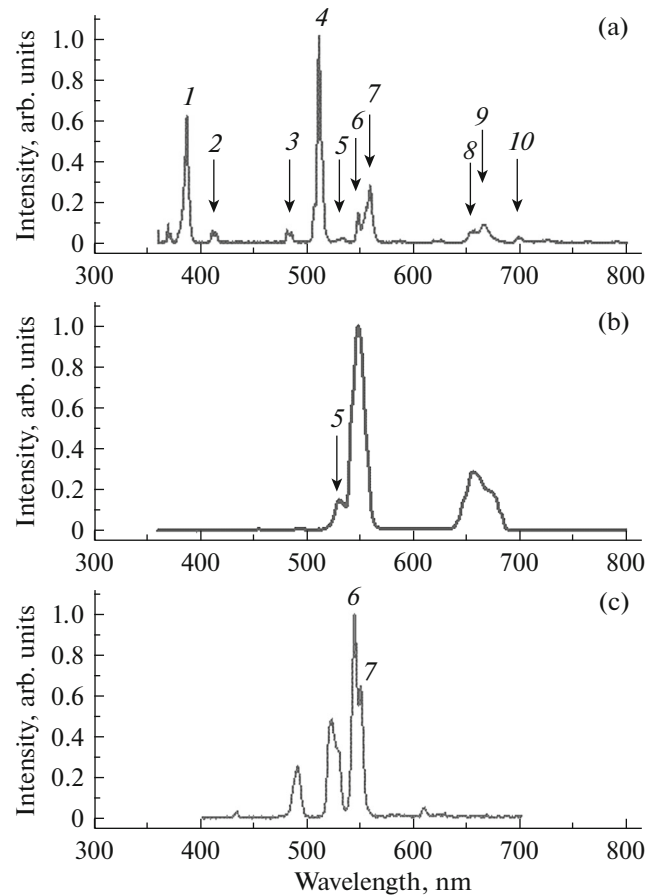
### 3. RESULTS AND DISCUSSION

The luminescence spectra of crystals (Fig. 1) were studied under excitation at a wavelength of 313 nm (excitation of the exciton states near the band gap) and 365 nm (transition  ${}^4I_{15/2} \rightarrow {}^2G_{11/2}$ ) and upon anti-Stokes excitation at 980 nm (transitions  ${}^4I_{15/2} \rightarrow {}^4I_{11/2}$ ,  ${}^4I_{11/2} \rightarrow {}^4F_{3/2}$ ).

Excitation at 313 nm brings the  $\text{Er}^{3+}$  ion into the  ${}^2G_{7/2}$  state, and therefore, radiative relaxation to the  $f$  levels becomes possible and nonradiative transitions to the underlying  ${}^4G_{9/2}$ ,  ${}^4G_{11/2}$ ,  ${}^2H(G)_{9/2}$  levels may occur. It is seen in the corresponding luminescence spectrum that emission occurs only from the  ${}^2H(G)_{9/2}$  level.

A significant change in the shape of the spectrum is observed upon excitation at 365 nm, which can be fully described in terms of multiphonon relaxation. The most intensive transitions are  ${}^4G_{11/2} \rightarrow {}^4I_{15/2}$  and  ${}^4G_{11/2} \rightarrow {}^4I_{13/2}$ ; excitation brings the ion to the  ${}^2H(G)_{9/2}$  level. Nonradiative transitions are largely manifested because the density of levels near the  ${}^2H(G)_{9/2}$  level is relatively high. These transitions virtually completely extinguish the transitions observed upon excitation at 313 nm.

The excitation by a GaAs diode (980 nm) leads to the anti-Stokes process, that is,  ${}^4I_{15/2} \rightarrow {}^4I_{11/2}$  and  ${}^4I_{11/2} \rightarrow {}^4F_{3/2}$ . Multiphonon nonradiative transitions may occur due to the relative proximity of the  ${}^4F_{3/2}$  level with the  ${}^2H(G)_{9/2}$  and  ${}^4F_{5/2}$  levels. Note that the

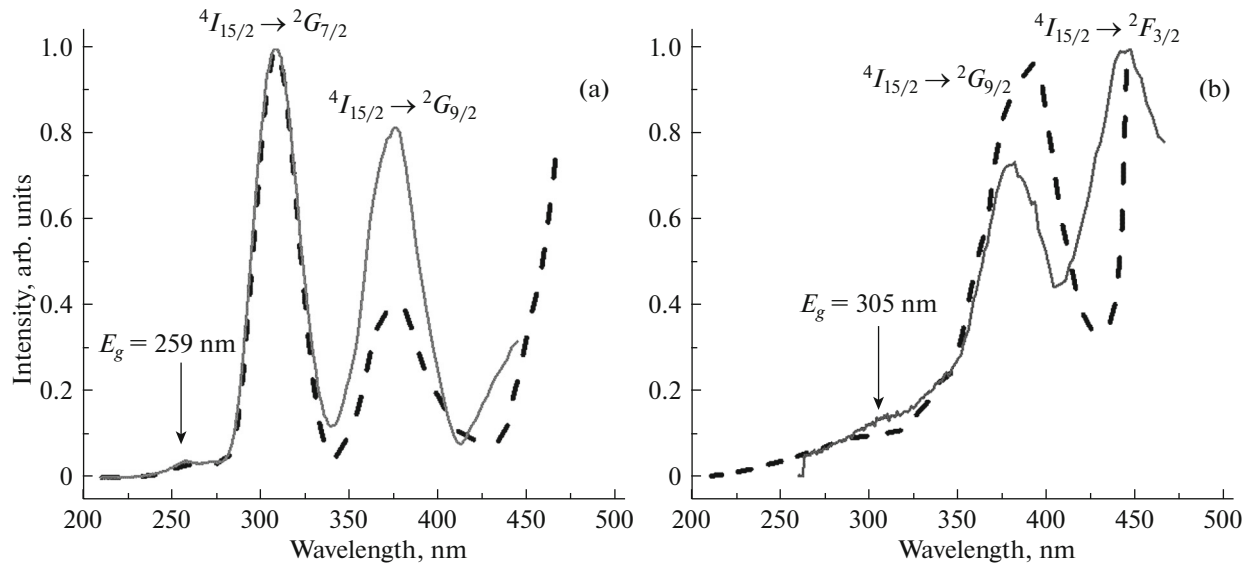


**Fig. 1.** Photoluminescence spectrum of  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  at  $\lambda_{\text{exc}} =$  (a) 313, (b) 365, and (c) 980 nm. The following transitions are indicated: (1)  ${}^4G_{11/2} \rightarrow {}^4I_{15/2}$ , (2)  ${}^2H(G)_{9/2} \rightarrow {}^4I_{15/2}$ , (3)  ${}^4F_{7/2} \rightarrow {}^4I_{15/2}$ , (4)  ${}^4G_{11/2} \rightarrow {}^4I_{13/2}$ , (5)  ${}^2H_{11/2} \rightarrow {}^4I_{15/2}$ , (6)  ${}^4S_{3/2} \rightarrow {}^4I_{15/2}$ , (7)  ${}^2H(G)_{9/2} \rightarrow {}^4I_{13/2}$ , (8)  ${}^4F_{3/2} \rightarrow {}^4I_{13/2}$ , (9)  ${}^4F_{9/2} \rightarrow {}^4I_{15/2}$ , and (10)  ${}^2H(G)_{9/2} \rightarrow {}^4I_{11/2}$ .

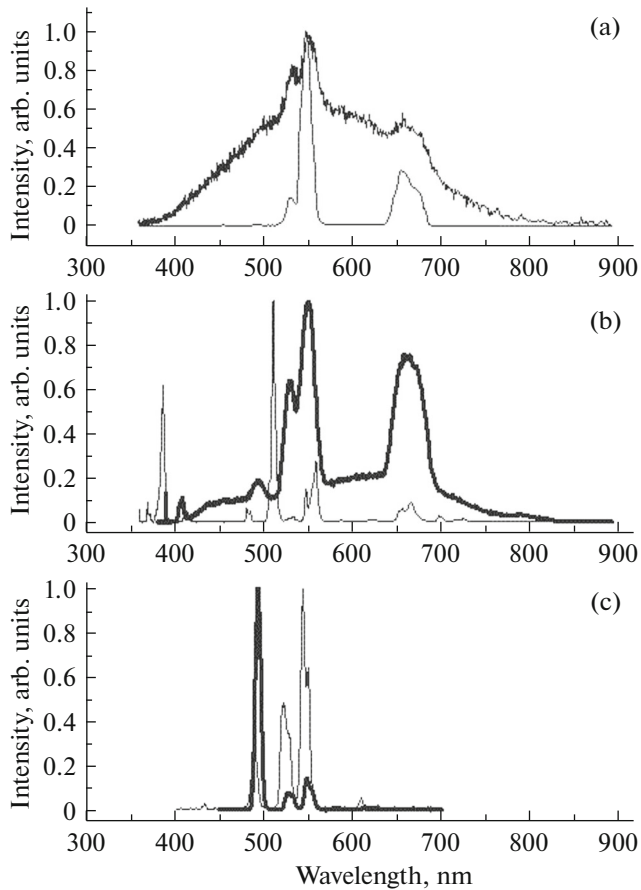
inverse transition of  ${}^4F_{3/2} \rightarrow {}^4I_{15/2}$ , which should be observed because of a significant excitation of the  ${}^4F_{3/2}$  level, proceeds to a minimal degree (see the peak at 430 nm). This can be explained by the substantial lifetime of the  $\text{Er}^{3+}$  ion in this state; therefore, radiative transitions are much less likely.

The excitation wavelengths were selected using the measured excitation spectra of the most significant lines at 550 and 650 nm (Fig. 2).

A comparison of the  $\text{KPb}_2\text{Cl}_5$  and  $\text{RbPb}_2\text{Br}_5$  spectra (Fig. 3) leads to the conclusion that the luminescence from the  ${}^4S_{3/2}$  and  ${}^2H(G)_{9/2}$  levels begins to compete with the luminescence caused by the transition of  ${}^2H_{11/2} \rightarrow {}^4I_{15/2}$ . This effect can be explained by the difference in the phonon energies in the matrices.



**Fig. 2.** Luminescence excitation spectra of (a)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  and (b)  $\text{RbPb}_2\text{Br}_5:\text{Er}^{3+}$  crystals; excitation of luminescence at a wavelength of (solid curve) 650 and (dashed curve) 550 nm.

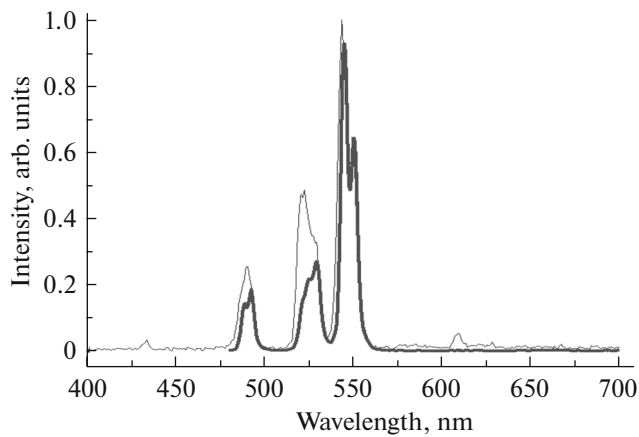


**Fig. 3.** Luminescence spectra of (thin curve)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  and (bold curve)  $\text{RbPb}_2\text{Br}_5:\text{Er}^{3+}$  at (a) 313, (b) 365, and (c) 980 nm.

Upon excitation at 313 nm, the luminescence of the self-trapped exciton is observed in  $\text{RbPb}_2\text{Br}_5$  crystals. The effect is associated with a smaller band gap in the  $\text{RbPb}_2\text{Br}_5$  matrix compared with the band gap of the  $\text{KPb}_2\text{Cl}_5$  matrix, caused by the replacement of the chlorine ion with the bromine ion. Since the levels of the self-trapped exciton are fixed relative to the bottom of the conduction band, this energy achieved at 313 nm is sufficient to excite an exciton in a  $\text{RbPb}_2\text{Br}_5$  crystal. The exciton glow actually extinguishes the glow of  $\text{Er}^{3+}$ .

A residual exciton emission is observed upon excitation at 365 nm. A relatively broad band at 650–700 nm is due to the  ${}^4F_{3/2} \rightarrow {}^4I_{13/2}$ ,  ${}^4F_{7/2} \rightarrow {}^4I_{13/2}$ , and  ${}^4F_{9/2} \rightarrow {}^4I_{15/2}$  transitions. The  ${}^4F_{7/2}$  and  ${}^2H_{11/2}$  levels are located close to each other; the  ${}^2H_{11/2}$  level is populated by a multiphonon mechanism, and therefore, luminescence by transition  ${}^2H_{11/2} \rightarrow {}^4I_{15/2}$  is observed (line 490 nm). In the case of  $\text{KPb}_2\text{Cl}_5$ , this transition is much less likely, which is explained by a smaller population of the  ${}^4F_{7/2}$  level.

Upon excitation at 980 nm, the intensity of the  ${}^4F_{7/2} \rightarrow {}^4I_{15/2}$  transition increases significantly, which is probably due to lower phonon energies in the  $\text{RbPb}_2\text{Br}_5$  matrix ( $133 \text{ cm}^{-1}$ ) compared to  $\text{KPb}_2\text{Cl}_5$  ( $200 \text{ cm}^{-1}$ ). Consequently, the  ${}^4F_{3/2} \rightarrow {}^4F_{5/2}$  transition and the subsequent relaxation of  ${}^4F_{5/2} \rightarrow {}^4F_{7/2}$  are less intense; the corresponding intensities of the spectral lines at 525 and 550 nm in the photoluminescence spectrum are lower for  $\text{RbPb}_2\text{Br}_5$  than those for  $\text{KPb}_2\text{Cl}_5$ .



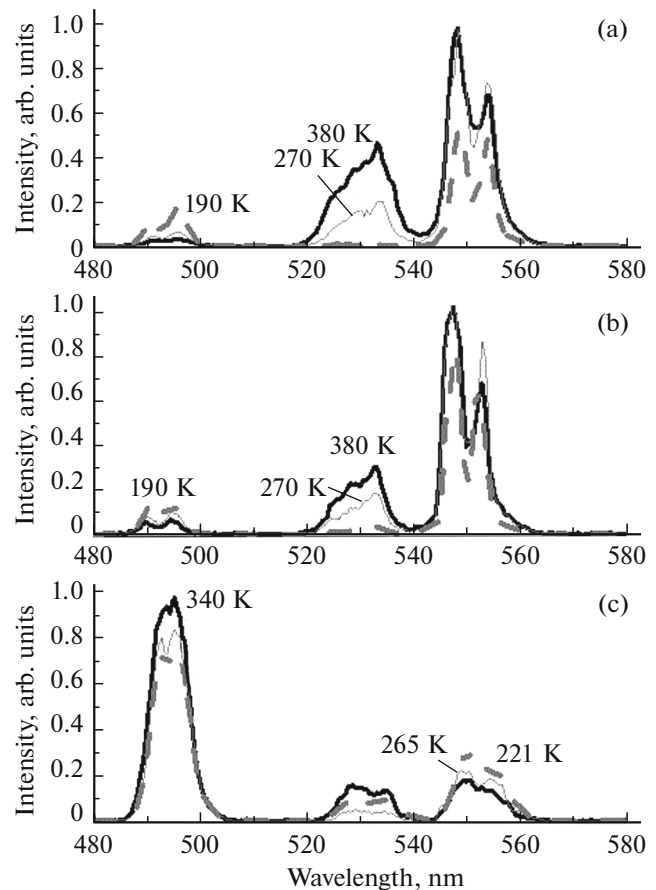
**Fig. 4.** Luminescence spectrum upon excitation of (thin curve)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  and (bold curve)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+} + \text{Yb}^{3+}$ .

The additional doping of the  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  matrix with  $\text{Yb}^{3+}$  ions significantly increases the quantum yield upon excitation at 980 nm and affects it only slightly at the other excitation energies. The integrated quantum yield upon excitation at 980 nm by a laser diode increases by approximately five to ten times; at different excitation wavelengths, the integrated quantum yield does not actually change. The luminescence spectrum of a  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+} + \text{Yb}^{3+}$  crystal (1 : 3) upon excitation at 980 nm by a laser diode actually coincides in shape with the luminescence spectrum of  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  under the same excitation conditions (Fig. 4). This is due to the absence of levels above  ${}^4F_{5/2}$  in the  $\text{Yb}^{3+}$  ion in the band gap in this matrix; the allowed energy transitions in the  $\text{Yb}^{3+}$  ion are represented only by the  ${}^4F_{7/2} \rightarrow {}^4F_{5/2}$  transition (near 980 nm).  $\text{Er}^{3+}$  ions also have an allowed  ${}^4I_{15/2} \rightarrow {}^4I_{11/2}$  transition, corresponding to an energy of 980 nm. Thus, if both types of ions are present in the matrix, nonradiative energy transfer from  $\text{Yb}^{3+}$  to  $\text{Er}^{3+}$  may occur. Since  $\text{Yb}^{3+}$  ions can absorb only phonons with an energy of 980 nm, their effect is only observed upon excitation at 980 nm.

We also note a smoother spectrum with additional doping with  $\text{Yb}^{3+}$ ; the smoothness is due to the significant amplification of the signal when  $\text{Yb}^{3+}$  is activated, so that the signal-to-noise ratio in the transmission path increases significantly (the integrated quantum yield increases approximately five to ten times).

The temperature dependences were measured for crystals in the temperature range of 90–420 K upon excitation by a laser diode at 980 nm. The results are shown in Fig. 5.

As the temperature increases, the luminescence line at 490 nm in  $\text{KPb}_2\text{Cl}_5$  is quenched, and a corresponding rise is observed for the line at 530 nm, which

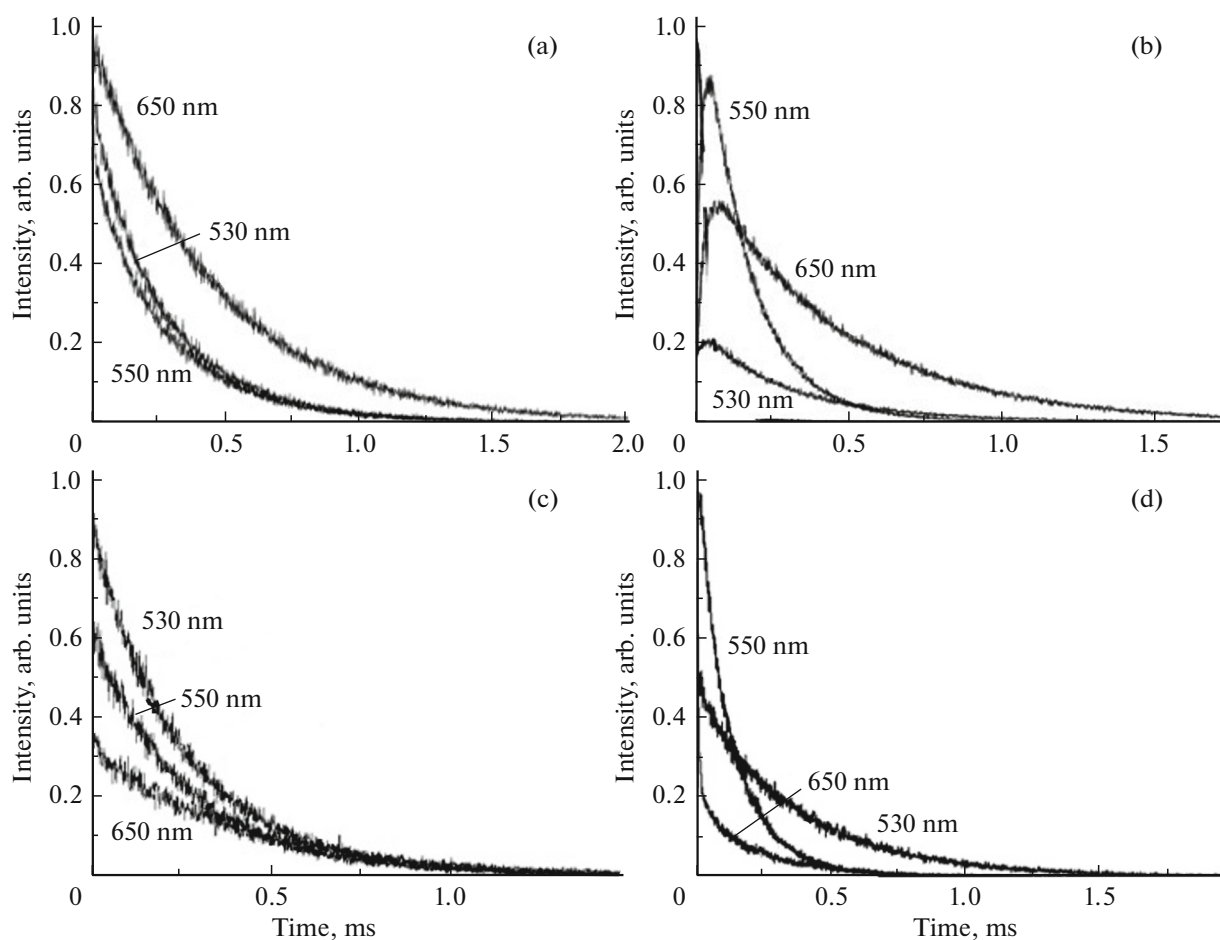


**Fig. 5.** Luminescence spectra in  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  and  $\text{RbPb}_2\text{Br}_5:\text{Er}^{3+}$  crystals at different temperatures: (a)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$ , (b)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+} + \text{Yb}^{3+}$ , and (c)  $\text{RbPb}_2\text{Br}_5:\text{Er}^{3+}$ .

is associated with the population of the overlying  ${}^4F_{5/2}$  level; in this case, a smaller number of photons is released upon transition from  ${}^4F_{7/2}$  to the ground state.

Note the pattern found for the line at 550 nm in  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  ( ${}^4S_{3/2} \rightarrow {}^4I_{15/2}$ ): as the temperature rises from 90 to 190 K, the line flares up. Further heating affects the intensity of the line only slightly; in fact, the line stabilizes in the temperature range of 190–400 K.

When the  $\text{RbPb}_2\text{Br}_5$  matrix is doped with  $\text{Er}^{3+}$  ions, the lines at 490 and 530 nm become less intense at low temperatures. In this case, the population of the  ${}^4F_{7/2}$  level is less likely because the phonon energy in  $\text{KPb}_2\text{Cl}_5$  is higher than that in  $\text{RbPb}_2\text{Br}_5$  (200 and  $144 \text{ cm}^{-1}$ , respectively). The quantum yield of  $\text{Er}^{3+}$  luminescence in the  $\text{KPb}_2\text{Cl}_5$  matrix is higher than that in the  $\text{RbPb}_2\text{Br}_5$  matrix by approximately five to ten times.



**Fig. 6.** Time dependences of the luminescence intensity upon excitation at (a, c) 313 and (b, d) 365 nm in (a, b)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  and (c, d)  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+} + \text{Yb}^{3+}$  crystal.

The time dependences of the luminescence spectra on temperature were measured for the lines 530, 550, and 650 nm (Fig. 6).

The spectra presented show that in the case of the excitation of  $\text{Er}^{3+}$  ions in the  $\text{KPb}_2\text{Cl}_5$  matrix (the  ${}^4I_{15/2} \rightarrow {}^4G_{11/2}$  transition), the luminescence undergoes a burning stage with a typical burning time of approximately 0.5 ms. This is explained by the transfer of energy between the levels through intermediate channels (presumably, through the  ${}^4F_{7/2}$  level).

The corresponding luminescence times were found from the kinetics obtained; the typical value of the error in the calculations was on average 2%. The experimental luminescence times were compared with those calculated by the Judd–Ofelt method (Table 1); in the case of  $\text{RbPb}_2\text{Br}_5$ , the phonon energy was renormalized.

As can be seen from the data of Table 1, the luminescence times in a crystal additionally activated with  $\text{Yb}^{3+}$  ions are in all cases smaller than the corresponding luminescence times in a crystal without a sensi-

tizer. This effect is in accordance with the idea of the existence of an additional channel for the transfer of energy between ligand ions, the effectiveness of which can be found using the equation

$$\eta = 1 - \frac{\tau_{D-A}}{\tau_D}, \quad (1)$$

where  $\tau_{D-A}$  is the luminescence time in a crystal doped with  $\text{Er}^{3+}$  and  $\text{Yb}^{3+}$ , and  $\tau_D$  is the luminescence time in a crystal doped with only  $\text{Er}^{3+}$ .

In accordance with the above, the experimental energy transfer efficiency upon excitation at 365 nm is 12% for a luminescence wavelength of 550 nm and 24% for a luminescence wavelength of 650 nm.

The theoretical luminescence times for  $\text{KPb}_2\text{Cl}_5:\text{Er}^{3+}$  crystals are taken from [7]; for  $\text{RbPb}_2\text{Br}_5:\text{Er}^{3+}$  crystals, the recalculation to phonon energy (200  $\text{cm}^{-1}$  for  $\text{KPb}_2\text{Cl}_5$  and 133  $\text{cm}^{-1}$  for  $\text{RbPb}_2\text{Br}_5$ ) was performed in accordance with the equations from [5].

**Table 1.** Luminescence times of the centers, found experimentally, depending on the wavelength of excitation and luminescence

Crystal	Excitation wavelength, nm	Luminescence line, nm	Luminescence time (experiment), ms	Luminescence time (theory), ms
KPb <sub>2</sub> Cl <sub>5</sub> :Er <sup>3+</sup>	313	530	0.28	0.04
		550	0.28	0.43
		650	0.45	0.32
	365	508	0.10	0.04
		530	0.32	0.01
		550	0.16	0.43
650		0.45	0.32	
RbPb <sub>2</sub> Br <sub>5</sub> :Er <sup>3+</sup>	313	530	0.18	0.03
		550	0.18	0.40
		650	0.02	0.30
	365	530	0.16	0.03
		550	0.17	0.40
		650	0.24	0.30

To recalculate the theoretical values of the luminescence times from the Er<sup>3+</sup> levels in RbPb<sub>2</sub>Br<sub>5</sub> crystals, we used the following phenomenological parameters  $B$  and  $\alpha$ :  $B = 4 \times 10^9 \text{ s}^{-1}$ ,  $\alpha = 1.2 \times 10^{-2} \text{ cm}^{-1}$  [7].

The recalculation of the luminescence times for KPb<sub>2</sub>Cl<sub>5</sub>:Er<sup>3+</sup> + Yb<sup>3+</sup> is complex and has not been done in this work, since it requires taking into account energy transfer between ions by different mechanisms.

The calculation presented proves that the results given by the theory predict relatively correctly the behavior of luminescent centers for the excitation relaxation from the <sup>4</sup>S<sub>3/2</sub> and <sup>4</sup>F<sub>9/2</sub> levels and give incorrect predictions for the excitation relaxation from the <sup>2</sup>H<sub>11/2</sub> level. This pattern is preserved when recalculated to the phonon energy, which was done in this work.

#### 4. CONCLUSIONS

The luminescence spectra of crystals were measured at different excitations (zone–zone transitions, transitions between the energy states of doping Er<sup>3+</sup> and Yb<sup>3+</sup> ions) with full identification of all observed transitions in the impurity center. Based on the experimental data, we demonstrated that KPb<sub>2</sub>Cl<sub>5</sub>:Er<sup>3+</sup> crystals could be used as efficient energy converters from one range to another. On the other hand, RbPb<sub>2</sub>Br<sub>5</sub>:Er<sup>3+</sup> crystals do not exhibit high conversion efficiency due to the exciton luminescence, and their use as converters is not recommended.

The anti-Stokes luminescence spectra of doping ions were measured at various temperatures (excitation by a laser diode at 980 nm), and the temperature dependences of the spectra were found. It is shown

that in the KPb<sub>2</sub>Cl<sub>5</sub> matrix, the populations of the <sup>4</sup>F<sub>7/2</sub> and <sup>2</sup>H<sub>11/2</sub> levels are correlated. In KPb<sub>2</sub>Cl<sub>5</sub>:Er<sup>3+</sup>, the effect of stabilization of the luminescence intensity was observed at 550 nm in the temperature range of 190–420 K. No such effect was found for codoping with Er<sup>3+</sup> and Yb<sup>3+</sup>.

The luminescence kinetics were measured for the most intense bands upon excitation at 313 and 365 nm; the corresponding luminescence times of center are obtained. In a KPb<sub>2</sub>Cl<sub>5</sub>:Er<sup>3+</sup> crystal, the luminescence build-up was observed upon excitation at 365 nm, which is evidence of the population of the level through intermediate states (presumably, through <sup>4</sup>F<sub>7/2</sub>). In crystals additionally activated with Yb<sup>3+</sup>, a decrease in the luminescence time was detected, which is because of the presence of an additional channel of energy transfer from the sensitizer to the activator.

The Judd–Ofelt method and the theory of energy transfer in the form of multiphonon nonradiative relaxation are considered with respect to the lifetimes of the centers. The results calculated by the Judd–Ofelt method are similar to the experimental data on the order of magnitude for luminescence from the <sup>4</sup>S<sub>3/2</sub> and <sup>4</sup>F<sub>9/2</sub> levels. For luminescence from the <sup>4</sup>F<sub>7/2</sub> level, the difference between the theory and experiment is significant, which may be due to a nonproper selection of the model parameters for the calculation. The values calculated for RbPb<sub>2</sub>Br<sub>5</sub> correlate with the calculations for KPb<sub>2</sub>Cl<sub>5</sub>; the correction takes into account the difference in the phonon energy of the lattice: 200 cm<sup>-1</sup> for KPb<sub>2</sub>Cl<sub>5</sub> and 133 cm<sup>-1</sup> for RbPb<sub>2</sub>Br<sub>5</sub>.

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