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Radiophotoluminescence of silver-doped lithium triborate glass

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Please publish the article "Radiophotoluminescence of silver-doped lithium triborate glass" in the journal Materials Science and Engineering: B. These results are of considerable interest, given that for LiB_3O_5 the absorption coefficient Z_{eff} = 7.39 is close to Z_{eff} = 7.42 of human body tissues, LiB_3O_5:Ag glass is very promising in the development of a new effective cheap dosimetric material for γ-dosimetry in medical practice. Studies conducted at the Oncology Regional Medical and Diagnostic Center have demonstrated the possibility of its use for dosimetry in the range of 1-3 Gray. This dose range corresponds to the range of single radiation doses during the gradual accumulation of the full dose of several tens of Gray (for example, 50-60 Gy) prescribed by an oncologist for a specific cancer tumor. Due to the presence of lithium isotopes Li(6) and boron B(10), LiB_3O_5:Ag glass is highly sensitive to neutrons, which makes it possible to manufacture individual neutron dosimeters for use in a new field of radiation therapy - neutron therapy.

- It was found that LiB_3O_5:Ag glass is promising for γ-dosimetry in medical practice

- It was found high sensitivity of LiB_3O_5:Ag glass for dosimetry in the range 1 – 3 Gy

- Ability to manufactur e neutron dosimeters for the neutron therapy

Radiophotoluminescence of silver-doped lithium triborate glass

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Abstract

The paper presents the results of studies of the radiophotoluminescence (RPL) of LiB_3O_5:Ag glass after irradiation with γ-rays in the dose range of 1-3 Gray on the remote γ-therapeutic apparatus "TERAGAM" Co(60) at the Lviv Oncology Regional Medical and Diagnostic Center. A clear dependence of the intensity of the RPL (λ_{max} = 300 nm) on the dose value when excited by light λ_{exc} = 220 nm was found. The mechanism of RPL in γ-irradiated LiB_3O_5:Ag glass is proposed as a consequence of radiation annihilation with the emission of relaxed exciton-like electronic excitations with the participation of impurity defects (Ag^0) in the glass structure.

Keywords: radiophotoluminescence, LiB_3O_5 glass, γ-irradiation, lithium isotopes Li(6), boron isotopes B(10), dosimeter.

1. Introduction.

In recent decades, intensive research has been conducted on optically stimulated luminescence (OSL), or in other words, radiophotoluminescence (RFL), the name depends on the excitation spectrum, because of their prospects for application in ionizing radiation dosimetry [1], especially in those areas related to medical practice [2, 3]. In particular, this applies to radiation therapy of patients with cancer [4] using almost all types of radiation, which stimulates relevant research. For example, here are some recent publications on the results of studies of OSL caused by the following types of radiation: gamma rays [5], beta radiation [6], protons (proton therapy) [7], neutrons (neutron therapy) [8], and ions [9].

The main component of OSL - RFL dosimeters is the working element, in which their own luminescence centers are excited or stable centers of luminescence are formed during radiation exposure. This means that the efficiency of working elements of such dosimeters is mainly determined by the material from which they are made [10, 11]. Therefore, at present, intensive searches and studies of various materials are ongoing, especially materials promising for the manufacture of 2D (or even 3D) dosimeters [12-14].

In our opinion, borate compounds can be a promising material for OSL-RPL dosimetry. In particular, there are reports on the study of OSL in Ag-doped $Li_2B_4O_7$ single crystals

[15,16] and in $Li_2B_4O_7$ nanoscale crystals doped Cu, Ag, and co-doped Cu, Ag [17]. And the OSL of Ce^{3+} and Li^+ co-doped magnesium borate glass ceramics MgB_4O_7:Ce,Li has already been proposed for use in gamma and neutron dosimetry [18, 19]. However, the growth of borate single crystals is a complex and time-consuming process, which increases their cost [20, 21]. An additional problem of borate single crystals is their resistance to doping, which severely limits the ability to control the concentration of luminescent impurities. Therefore, it is worth paying attention to borate glasses as a material for OSL-RPL dosimeters. As it turned out, borate glasses of different compositions with different impurities exhibit quite intense photoluminescence (PL). For example, intense FL was observed in tetraborate glasses doped with one impurity: $Li_2B_4O_7$:Eu [22], $Li_2B_4O_7$:Er [23], $Li_2B_4O_7$:Tm [24], $Li_2B_4O_7$:Sm [25], $Li_2B_4O_7$:Sm and $Li_2B_4O_7$:Yb [26], $Li_2B_4O_7$:Tb and $Li_2B_4O_7$:Dy [27], $Li_2B_4O_7$:Cu [28], as well as doped with two impurities: $Li_2B_4O_7$:Er,Ag [29], $Li_2B_4O_7$:Eu,Ag [30], $Li_2B_4O_7$:Sm,Ag [31], $Li_2B_4O_7$:Tm,Ag [32], and $Li_2B_4O_7$:Gd,Ag [33]. Moreover, it was found that co-doped Ag increases the intensity of some emission bands of rare earth impurities [23,24,26,27]. The authors did not ignore doped borate glasses of other compositions, in particular: LiB_3O_5:Eu [34], $LiCaBO_3$:Nd, CaB_4O_7:Nd, $Li_2B_4O_7$:Nd [35], CaB_4O_7:Tb [36]. However, the authors are not aware of any reports on the study of RFL in lithium triborate (LiB_3O_5) glasses. At the same time, LiB_3O_5 may be very interesting for X-ray dosimetry in medical practice. After all, the value of a tissue-equivalent absorption coefficient Z_{eff} is very important for dosimetric material in radiotherapy [37]. And for LiB_3O_5 compound, Z_{eff} = 7.39, which is the closest to Z_{eff} = 7.42 of human body tissue and allows obtaining more accurate dose values without introducing additional coefficients [38]. The advantage of LiB_3O_5 glass

is also the low cost of the starting reagents Li_2CO_3 and H_3BO_3 for the synthesis of the compound, an increased tendency to glass transition due to the high content of boron oxide B_2O_3 in its composition, a low melting point T_{melt} = 1107 K and, accordingly, lower glass boiling temperatures, as well as the absence of concentration restrictions during doping. In addition, LiB_3O_5 glass allows the formation of crystallized glass, i.e. pyroceramics, by special annealing. It has been found that Ag-doped LiB_3O_5 pyroceramics are promising for thermoluminescence (TL) dosimetry, i.e., for the manufacture of individual dosimeters [39].

Increased sensitivity to neutrons due to the presence of lithium isotopes Li(6) and boron B(10) with high neutron capture cross-section of interaction with thermal neutrons σ (945 and 3840 barn, respectively) makes LiB_3O_5 glass promising for the manufacture of neutron dosimeters, similar to what was proposed for Ag-doped $Li_2B_4O_7$ single crystal [16]. If boric acid (H_3BO_3) enriched with B(10) and B(11) isotopes is used to make LiB_3O_5 glass, it allows to obtain glasses of the corresponding isotopic composition $Li^{10}B_3O_5$ and $Li^{11}B_3O_5$. The simultaneous use of two glasses with different isotopic compositions as a working body makes it possible to determine the neutron component of the dose in the presence of other types of radiation by the difference in their luminescence intensity measurements. This option has been demonstrated for solar neutron detection using $Li_2{}^{10}B_4O_7$ and $Li_2{}^{11}B_4O_7$ single crystals on the ISS [40]. Thus, LiB_3O_5 glass may prove to be a promising material for the manufacture of X-ray dosimeters for various types of radiation used in medical practice.

Therefore, the aim of this study was to investigate, with the participation of practicing radiation therapy specialists, the RPL of Ag-doped LiB_3O_5 glass after irradiation with

γ-quanta in the dose range of 1-3 Gy, since this is the range of a single dose, and for the set the radiation dose prescribed by a doctor for a specific cancer tumor in several tens of Gy (e.g., 50-60 Gy), the exposure is divided into the appropriate number of stages [41].

2. Material and methods

The solid-state reaction method was used for the synthesis of LiB_3O_5 compound. High purity lithium carbonate Li_2CO_3 and boric acid H_3BO_3 were used as the starting materials. The reagents were mixed in a certain proportion according to the phase diagram of borate compounds [42]. The mixture was loaded into a ceramic crucible and heated slowly in a resistive furnace to a temperature of 700°C (973 K) to ensure the successful completion of the reaction

$$Li_2CO_3 + 6\ H_3BO_3 \rightarrow 2\ LiB_3O_5 + CO_2 + 9\ H_2O.$$

As a result, LiB_3O_5 powder with T_{melt} = 1107 K was obtained. The doping was carried out by adding $AgNO_3$ to this powder at the rate of 0.1 mol%. The mixtures of the respective compositions were thoroughly ground and mixed in an agate mortar. The glass was prepared by fusing in a platinum crucible (Pt) in an air atmosphere at a temperature of 1170 K. To obtain a homogeneous distribution of components, the melt was kept for at least 1 hour. After that, the melt was poured onto a metal substrate at room temperature. The resulting glass block was used to cut out plates of ~ 6×7×1.5 mm in size, and their surfaces were ground and polished.

Absorption spectra were recorded on Perkin Elmer Lambda 25 and Ava Spec - 2048-USB2 spectrophotometers. A CM 2203 spectrofluorometer was used to study the spectroscopic properties and optically stimulated luminescence. The use of two double monochromators ensures minimal noise and high measurement accuracy. A 150 W pulsed xenon lamp is used as an excitation source. The device is controlled by a personal computer. The spectral range of measurements in the spectrofluorimeter mode is from 220 to 820 nm with a minimum spectral scanning step of the excitation and registration monochromators of 0.1 nm.

The samples were irradiated with γ-rays under real conditions of radiation therapy using a remote γ-therapy apparatus "TERAGAM" Co(60) at the Lviv Regional Oncology Treatment and Diagnostic Center.

3. Results and discussion

Fig. 1 shows the RPL spectra of LiB_3O_5:Ag glass samples under excitation with 220 nm light: unirradiated - curve 1, γ-irradiated with a dose of 1.5 Gy - curve 2 and a dose of 3.0 Gy - curve 3. As can be seen from the figure, the luminescence curves are in the region of 300 nm, have a rather pronounced Gaussian shape and their intensity clearly depends on the γ-irradiation dose. It should be noted that there is a slight shift in the maximum luminescence intensity with increasing γ-dose: λ_{max} = 299.2 nm for the unirradiated sample, λ_{max} = 296.5 for the γ-irradiated sample with a dose of 1.5 Gy and λ_{max} = 294.6 nm with a dose of 3.0 Gy.

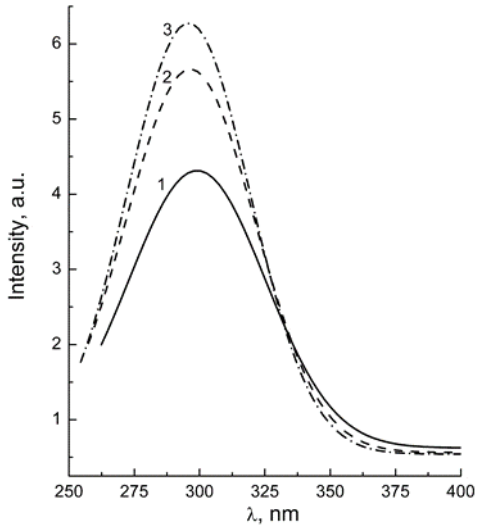

Fig.1. RPL spectra of LiB_3O_5:Ag glass under excitation by 220 nm light: 1 - unirradiated; 2 - γ-irradiation dose of 1.5 Gray; 3 - gamma-irradiation dose of 3.0 Gray.

In order to understand the mechanism of RPL in LiB_3O_5:Ag glass, it is first necessary to determine the location of the Ag impurity in the glass structure. To do this, it is first worth recalling the crystal structure of LiB_3O_5 and its luminescent properties in the single crystal version. Crystalline LiB_3O_5 has an orthorhombic crystal structure with the Pna21 space group and lattice parameters a = 8.46 Å, b = 5.13 Å, and c = 7.38 Å [43]. Its crystal structure is a framework of boron-oxygen complexes $(B_3O_7)^{5-}$. These complexes consist of three boron atoms, which are all in non-equivalent positions, and seven oxygen atoms in five non-equivalent positions. In particular, two of the three non-equivalent boron atoms have a flat three-coordinated BO_3 bond structure, and the third boron atom has a tetrahedral four-coordinated BO_4 bond structure. Lithium ions Li^+ are located in open voids between $(B_3O_7)^{5-}$ complexes in a distorted oxygen tetrahedron, with all Li^+ positions equivalent. Later refinements showed that the LiB_3O_5 unit cell consists of 36 atoms, which are divided into four groups of nine atoms

and are best described as spirals of fully bonded $(B_3O_7)^{5-}$ groups separated by Li^+ cations [44].

The study of the absorption, excitation, and photoluminescence spectra of pure oriented single crystals of LiB_3O_5 in a wide spectral range of 1.2-10.5 eV has shown that the broadband luminescence of LiB_3O_5 single crystals in the region of 3.5-4.5 eV (354-275 nm) is effectively excited by photons with energy above 7.5 eV (165 nm), which is at the edge of the fundamental absorption. This luminescence occurs due to radiation annihilation of relaxed exciton-like electronic excitations that can be formed either directly or as a result of recombination involving the main lattice defects [45].

Since there is no information on luminescence studies of doped LiB_3O_5:Ag single crystals, it is also worth paying attention to the results of studies of $Li_2B_4O_7$:Ag single crystals similar in composition [15, 46-49]. The authors of these works convincingly proved that in a single crystal of $Li_2B_4O_7$:Ag, an Ag^+ impurity can be found in two positions: as a substitution impurity at Li^+ positions and in the interstices. Completely similar results were obtained by the authors of [50] for LiB_3O_5:Cu single crystals, where it was proved that a copper impurity in the form of a Cu^+ ion can be located in two positions in the crystal lattice - to replace Li^+ , or to be located in the interstices.

To understand what position the Ag impurity occupies in the structure of LiB_3O_5:Ag glass, it is necessary to consider in more detail what structural changes occur during the formation of LiB_3O_5 glass, in addition to disturbances of the long-range order. It is known that the triborate oxygen complex in the $(B_3O_7)^{5-}$ crystal consists of $2(BO_3)^{3-}$ + $(BO_4)^{4-}$, and since the average B-O bonding distance in BO_3 is 0.137 nm and in BO_4 is 0.147 nm, the average B-O bonding distance in the entire complex is 140 nm. At the

same time, an X-ray structural investigation showed that the average B-O bond distance in glass is 0.144 nm [51]. This may indicate that the ratio of concentrations of triangular and tetraborate complexes in LiB_3O_5 glass slightly changes in favor of the latter. The situation with the Li - O bonds is somewhat different. After all, the Li^+ ion is located in a single crystal of LiB_3O_5 in a distorted oxygen tetrahedron with an average Li - O distance of 0.204 nm, while in glass it increases to 0.250 nm [51]. Thus, it can be unequivocally concluded that LiB_3O_5 glass not only lacks the long-range order, but also significantly distorts the short-range order compared to the single crystal. Thus, given the difference in the ionic radii of Ag^+ (1.13 nm) and B^{3+} (0.21 nm), it can be confidently stated that Ag^+ cannot occupy the places of B^{3+} in BO_3 and BO_4 elemental complexes of LiB_3O_5:Ag glass, but only can occupy the places reserved for Li^+, because there are no positions in the glass that can be interpreted as interstices. Moreover, in contrast to the crystalline state, in LiB_3O_5 glass, we should not mention the distorted oxygen tetrahedron around Li^+, but rather talk about the coordination numbers of its oxygen environment. And, as the experience of studying glasses of various lithium borates has shown, the coordination numbers of the oxygen environment of Li^+ in them can vary from 4 to 6, as shown, for example, in [28, 51].

Accordingly, the absorption spectrum of LiB_3O_5 glass (Fig. 2) differs significantly from that of a single crystal because it does not have a clearly defined absorption edge. This is typical for glassy samples, since the glass structure lacks translational symmetry. Therefore, to describe the electronic structure of such disordered media as glass, universal characteristics of electronic states, namely the distribution of electron energy density, are used. In this case, the long-wavelength shift of the absorption edge

of glass compared to single crystals can be explained by the blurring of the electronic density of states. Moreover, the energy band model can still be applied to glass, but taking into account the fact that direct interband transitions are prohibited, and only indirect transitions via phonons and excitons can occur [52].

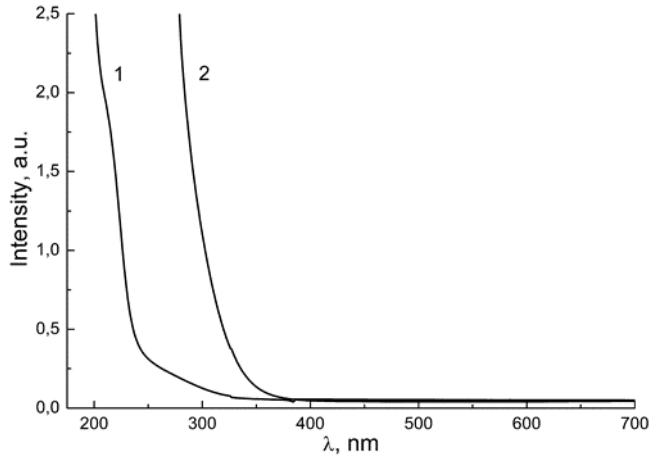

Fig. 2. Absorption spectra of LiB_3O_5 glass: 1 - doped with 0.1 mol.% Ag; 2 - pure.

The luminescence of LiB_3O_5:Ag glass in the region of 3.54 - 4.9 eV (350 - 250 nm) is effectively excited by photons with energy of 5.64 - 5.16 eV (220 - 240 nm), as shown by the luminescence excitation spectrum in the 300 nm region (Fig. 3). As can be seen from Figure 3, this excitation spectrum has a fairly wide range, which is explained precisely by the structural features of the glass and, accordingly, by the inhomogeneities in the coordination numbers of the oxygen environment of Ag. The very mechanism of luminescent emission of LiB_3O_5:Ag glass is most likely similar to the process proposed in [45] for LiB_3O_5 single crystals, i.e., it occurs due to radiation annihilation of relaxed exciton-like electronic excitations (i.e., the so-called self-trapped excitons) that can be formed with the participation of defects in the glass structure. Such defects in the structure of LiB_3O_5:Ag glass can be impurity Ag^0 atoms formed due to electron capture from a neighboring defective boron oxide complex.

Indeed, due to the much higher work of yield (ionization potential) of Ag^0 atoms (7.57 eV) compared to Li^0 (5.39 eV), Ag^+ ions in the structure of LiB_3O_5 glass can capture free electrons quite actively to form a neutral Ag^0 atom. This property of Ag^+ ions is actively used for the formation of silver nanoparticles in glasses, in particular borate glasses [53].

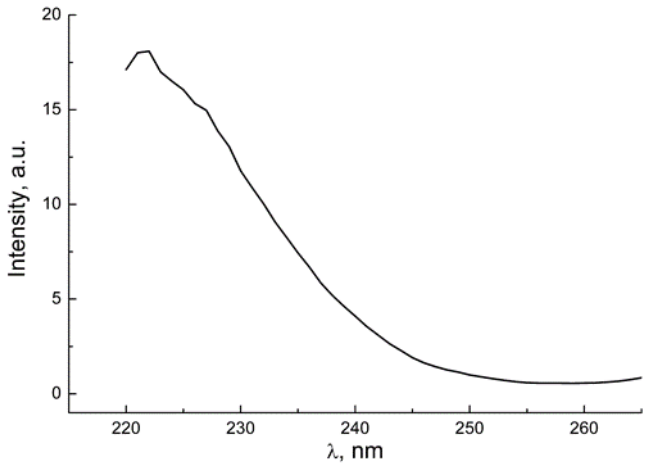

Fig. 3. The excitation spectrum of the LiB_3O_5:Ag glass. Registration of radiation at 310 nm.

Let us now consider the process of luminescent emission in LiB_3O_5:Ag glass not irradiated by γ-radiation. When LiB_3O_5:Ag samples are irradiated with light at a wavelength of 220 nm (5.64 eV), Ag^0 atoms transition to the excited state Ag* with the formation of a small-radius polaron upon interaction with a hole on a neighboring $(BO_4)^{4-}$ complex, followed by the formation of an excited state self-trapped exitons [45]. The subsequent radiative annihilation of this self-trapped exitons leads to luminescent emission in the region of 250 - 350 nm (4.9 - 3.54 eV) with a maximum at about 300 nm (4.13 eV).

Under γ-irradiation of LiB_3O_5:Ag glass samples, a large number of electron-hole pairs ($e^- + h^+$) are formed in its structure, some of which are rapidly annihilated, and some electrons are captured by Ag^+ ions to form additional neutral Ag^0 atoms, i.e., the process $Ag^+ + e^- \rightarrow Ag^0$. Thus, under γ-irradiation, the concentration of neutral Ag^0 atoms in LiB_3O_5:Ag glass increases. The holes, respectively, migrate through the boron oxide complexes and are captured by defective boron oxide complexes, which contain an extra complex $(BO_4)^{5-}$, defective in relation to the triborate complex, located near the Ag^0 impurity, i.e., the process occurs: $(BO_4)^{5-} + h^+ \rightarrow (BO_4)^{4-}$. Due to this, the subsequent excitation of the γ-irradiated LiB_3O_5:Ag glass sample with 220 nm light leads to an increase in the intensity of the RPL radiation, which increases in proportion to the received dose of γ-irradiation, as shown in Fig. 1. Thus, LiB_3O_5:Ag glass may be promising for use in dosimetry in medical practice during radiation therapy of patients with cancer.

4. Conclusion.

The conducted RPL studies of silver-doped lithium triborate glass LiB_3O_5:Ag after γ-ray irradiation with the TERAGAM Co(60) remote γ-therapy apparatus at the Lviv Regional Cancer Treatment and Diagnostic Center demonstrated the possibility of its use for dosimetry in the range of 1 - 3 Gray. This dose range corresponds to the range of single radiation doses during the gradual accumulation of the full dose of several tens of Gray (e.g., 50-60 Gy) prescribed by an oncologist for a specific cancer tumor. Considering that LiB_3O_5 has an absorption coefficient of Z_{eff} = 7.39, which is the closest to Z_{eff} = 7.42 of human body tissue, LiB_3O_5:Ag glass can be very promising for γ-dosimetry in medical practice. The increased sensitivity of LiB_3O_5:Ag glass to

neutrons due to the presence of lithium isotopes Li(6) and boron B(10) allows the manufacture of individual neutron dosimeters for applications in the new field of radiation therapy - neutron therapy.

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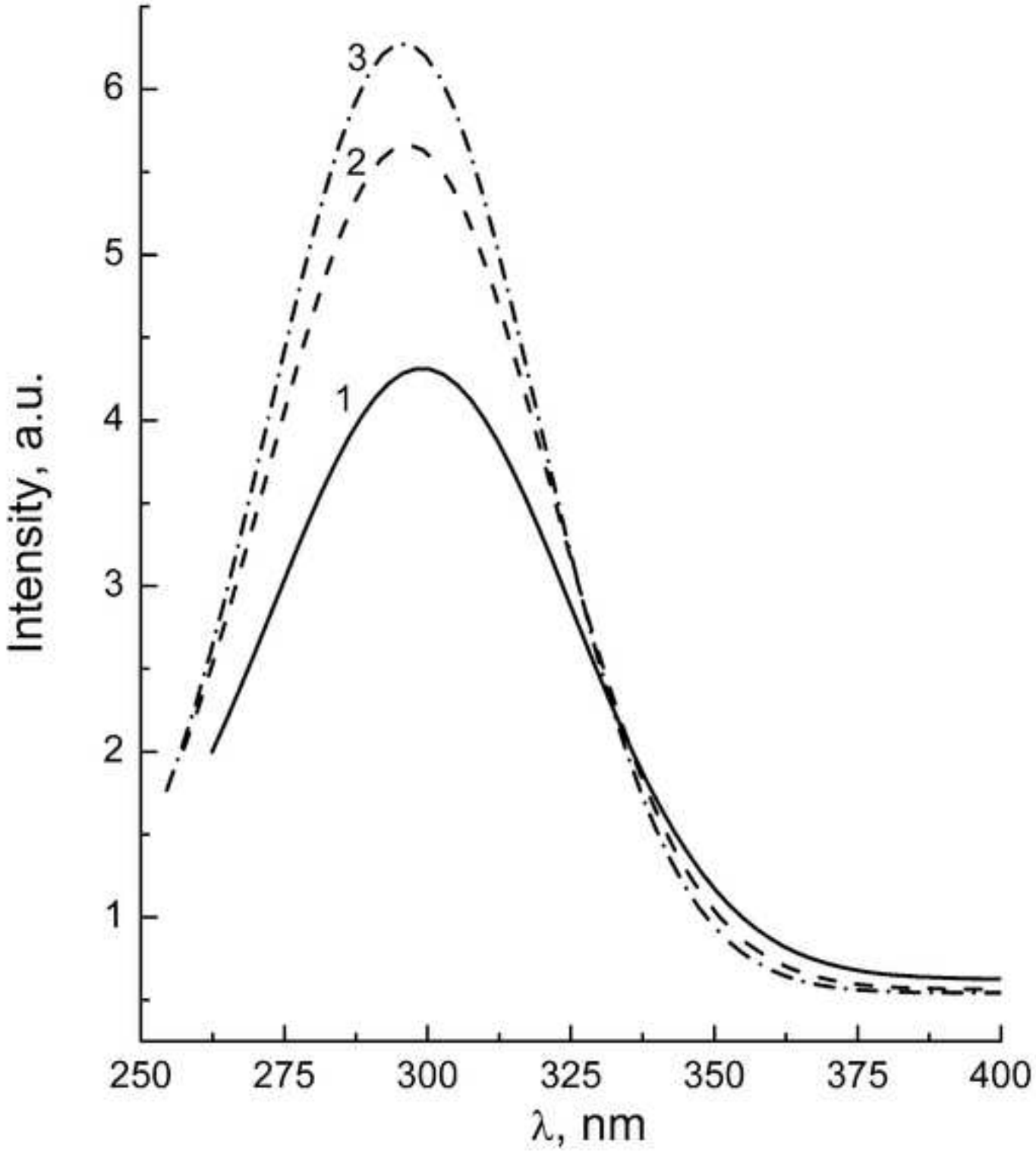

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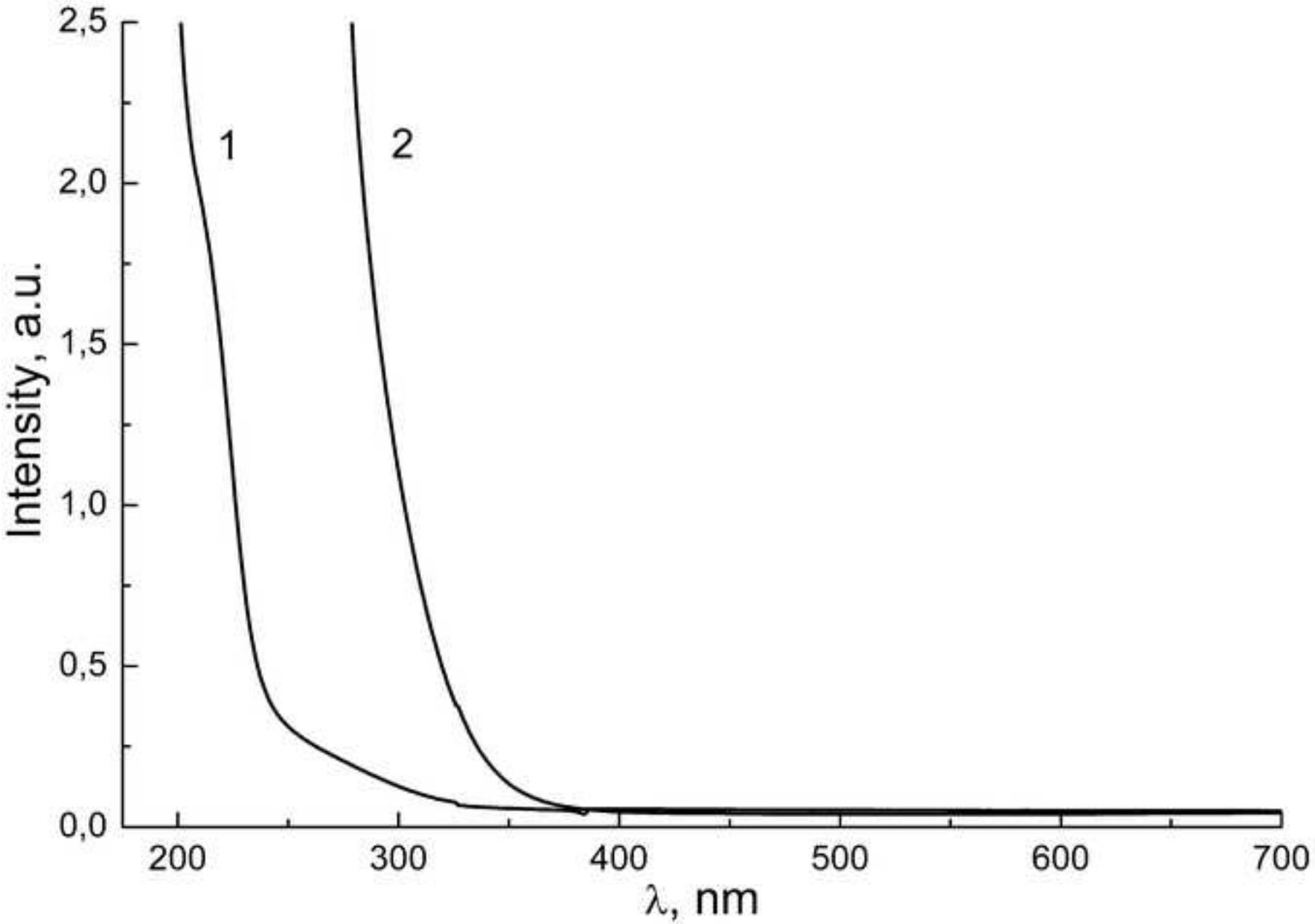

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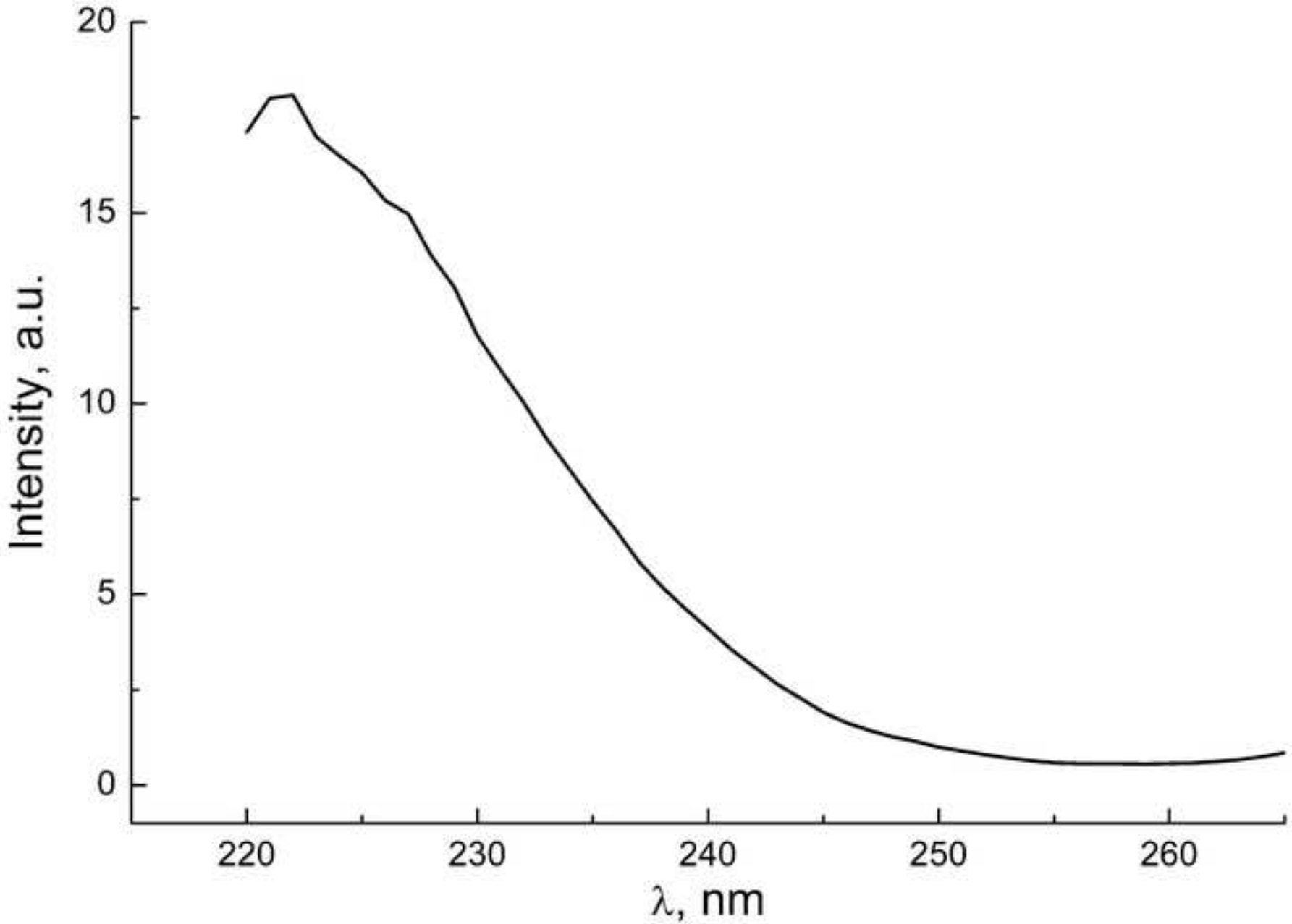

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☒The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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