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INFLUENCE OF KI IMPURITY ON SPECTRAL-KINETIC PROPERTIES OF POLY (9,9-DI-N-OCTYL FlUORENYL-2,7-DIYL) FILMS

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The spectral-fluorescent properties of the semiconductor films of poly (9,9 – di -n-octylfluorenyl-2,7 - diyl) (PFO) doped with KI impurity have been investigated. The addition of the KI salt leads to a decrease in the degree of ordering of the PFO films. The complex nature of the dependence of the photoelectronic processes in the polymer on the impurity concentration KI is determined from an analysis of the values of the vibronic splitting, the Huang-Riesz factor, the concentration dependence of the intensity and lifetime of the PFO fluorescence. The addition of KI in the polymer leads to an increase in the concentration of excited triplet states in films. Analysis of the spectral-kinetic data of annihilation delayed fluorescence and phosphorescence indicates an increase in the disorder of polymer films with the addition of the KI salt.

Keywords: poly (9,9 – di -n-octylfluorenyl- 2,7 - diyl), KI salt, optical spectrum, fluorescence kinetics

Introduction

Interest in composite materials based on semiconductor polymers has grown with the development of organic electronics and photovoltaics. This is due to the possibility of regulating the optical and electrical properties of polymer composites (PC) [1-3]. Nanoparticles of metals [4, 5], dyes [3], organic compounds organic compounds with a donor or acceptor properties relatively to the polymer [6] often act as impurities for polymers. A chemical compounds, leading to the appearance of the effect of an external heavy atom [7] use as an external impurity. This effect is due to the enhancement of the inter combination transitions from the electron singlet state to the triplet state under the action of the spin-orbit interaction [8].

Attention to the effect of an external heavy atom is associated with the possibility of increasing the concentration of triplet excited states in semiconductor polymers. This can be used to increase the efficiency of polymer solar cells [9, 10]. Also, this effect can be used in other applied problems, for example, obtaining the electro-phosphorescence of organic films [11-13].

Adding an external heavy atom to a semiconductor polymer can not only change the speed and efficiency of the various photophysical reactions, but also change the probability of formation of free charge carriers in the polymer. Character external impact of heavy atoms on photoelectric processes in semiconductor polymers remains researched insufficiently. The results of the investigation of the influence of an external heavy atom on the spectral-luminescent properties of films of poly (9,9-di-n-octylfluorenyl-2,7-diyl) (PFO) doped with an impurity of KI are given in this paper.

1. Experimental part

PFO films doped with an inorganic impurity of KI salt are used in the work. The polymer was used of Sigma-Aldrich and with a molecular weight $M_w \geq 20000$. The concentration of the impurity in the film varied in the range from 0.1 to 1% by weight of the polymer. The films are made by centrifugation. Thermal annealing of films in an inert atmosphere (Ar_2) was performed to increase the degree of ordering of the films.

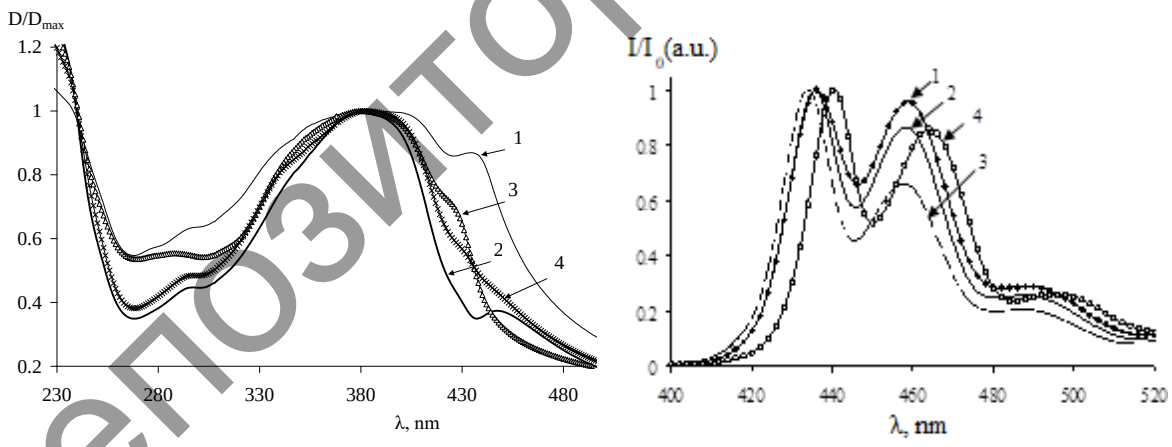
Spectrophotometer Agilent Cary 300 was used for registration of the absorption spectra of the films. The fluorescence spectra were measured on a Cary Eclipse spectro-fluorimeter of Agilent company. Kinetics of fast luminescense of films was measured using a pulsed spectro-fluorometer with picosecond resolution and registration with time-correlated photon counting mode (Becker & Hickl). Excitation of fluorescence was performed pulsed semiconductor laser with a wavelength λgen = 488 nm with full width at half maximum of pulse τ = 80 ps.

The kinetics of delay fluorescence in the micro- and millisecond time range was measured in a setup with registration in the photon counting mode [14-15]. The photoexcitation of the samples was performed by the third harmonic of the neodymium laser LCS-DTL-374QT. The recording part of the setup includes a photomultiplier with electronic unlocking H7421, a discriminator C8744 and an electronic pulse counting board M8784 (Hamamatsu Photonics).

2. Results and discussion

The absorption and fluorescence spectra of polymer composites (PC) PFO with KI additives are measured. In the absorption spectrum of a pure PFO film, a band with a maximum at 380 nm with an additional peak at 440 nm is observed (Figure 1, curve 1). The addition of KI to the polymer results in a decrease and a peak shift at 435 nm. It is known that the absorption spectrum of a highly ordered phase (β phase) PFO film has a pronounced vibronic structure with band maxima at 435 nm and 400 nm [17, 18]. The absorption spectrum of a disordered polymer film has a broad band with a maximum at 380 nm. The presence of a peak at 435 nm in the samples under study indicates the presence of an ordered phase in the polymer film. Addition of the KI impurity in the PFO leads to an increase in the disorder of the PC.

Using the technique given in [18], the fraction of the crystalline phase in the samples was estimated. The results are shown in Table 1. Also, the increase in the disorder of PFO films with KI addition is indicated by an increase in the absorption intensity in the region of 285 nm [18].



a) b)

Fig.1. The normalized absorption spectra (a) and fluorescence (b) of PFO polymer films with admixture of KI: 1- PFO; 2- PFO KI 0.1%; 3- PFO KI 0.5%; 4- PFO KI 1%.

From the absorption spectra of polymer films, the energy of the band gap (E_g) of the PFO polymer was determined upon addition of an inorganic impurity. The results are shown in Table 1. These results show that the main changes in the width of the band gap for unannealed polymer films occur when a minimum impurity concentration KI is added. Further growth of the impurity concentration does not change the width of the forbidden band. Thermal annealing in an inert atmosphere leads to a significant change in the width of the band gap of the PFO polymer.

The greatest decrease is observed for a film without the addition of an impurity and with a low impurity concentration (0.05%). For composite PFO films, there are three peaks in the fluorescence spectrum with maxima at 440, 460, and 490 nm (Figure 1, b). The luminescent data show that the glow of the samples under study is due to radiation from the crystalline phase of the PFO polymer [18, 19]. As shown in [19], in the polymer PFO film in the presence of ordered and disordered phases a significant singlet-singlet energy transfer from disordered polymer chains to ordered ones is observed. Therefore, in the presence of an ordered phase of more than 7% from the total value of the polymer is observed fluorescence only from β phase [17]. Spectral data on the fluorescence of the samples (Figure 1, b) correspond to the values of the fraction of β phase in films obtained from the absorption spectra (Table 1).

Table 1. Spectral and kinetic data of PC fluorescence PFO-KI

Sample	The proportion of the crystalline phase in the film (%)	*Eg* (eV)	Vibronic splitting (eV), ΔE	S-factor	τ, ps
PFO	0.23	2.555	0.146	0.95	510
PFO-KI 0.1%	0.10	2.555	0.143	0.86	500
PFO-KI 0.5%	0.15	2.740	0.147	0.66	496
PFO-KI 1%	0.15	2.695	0.141	0.86	413

Addition of an inorganic impurity results in shifts of the fluorescence spectra in the polymer first to the short-wave region of the spectrum, Figure 3, b curve 3, and then, with increasing impurity concentration KI, to the long-wave part of the spectrum (Figure 1, b curve 4). A shortwave shift in the fluorescence maximum is associated with a decrease in the degree of ordering of the film (Table 1). In this case, the long-wavelength shift of the fluorescence maximum of the PFO–1% KI film is not related to the degree of ordering of the films.

The value of the vibronic splitting $\Delta E = E_{0-0} - E_{0-1}$ and the Huang-Riesz factor were calculated for the fluorescence spectra from the formula [21-22]:

$$I_{0\to n} = \frac{e^{-S} S^n}{n!} \qquad (1)$$

where S is the Huang-Riesz factor.

As shown in a number of papers [20, 21], the growth of the disorder of the PC should lead to an increase in the Huang-Riesz factor (S). In our case, the growth of the disorder of the PC does not lead to an increase in the value of S. In this case, there is no change in the magnitude of the vibronic splitting ΔE observed in other works [22]. Thus, the addition of an impurity KI leads to complex changes in the fluorescence properties of a PC based on a PFO polymer with an admixture of KI.

The addition of the KI salt also leads to a decrease in the intensity of the fluorescence (Figure 2). Significant quenching of the fluorescence is observed at a low concentration of KI-0.1%. A further increase in the KI concentration leads to a decrease in the fluorescence intensity of the PC. Also in Figure 2, the change in the fluorescence lifetime of a PC from the KI impurity concentration in the nanosecond time range is reflected. The fluorescence lifetime (τ) decreases with increasing KI concentration. Graphs of the dependence of the intensity and lifetime of the PC fluorescence on the impurity concentration have a nonlinear dependence (Figure 2). This indicates the complex nature of the effect of KI impurity on the fluorescent properties of a PFO-based PC. When the samples are cooled to the boiling point of liquid nitrogen, two bands with maxima at 440 and 580 nm are observed in the delayed fluorescence spectrum (Figure 2, b).

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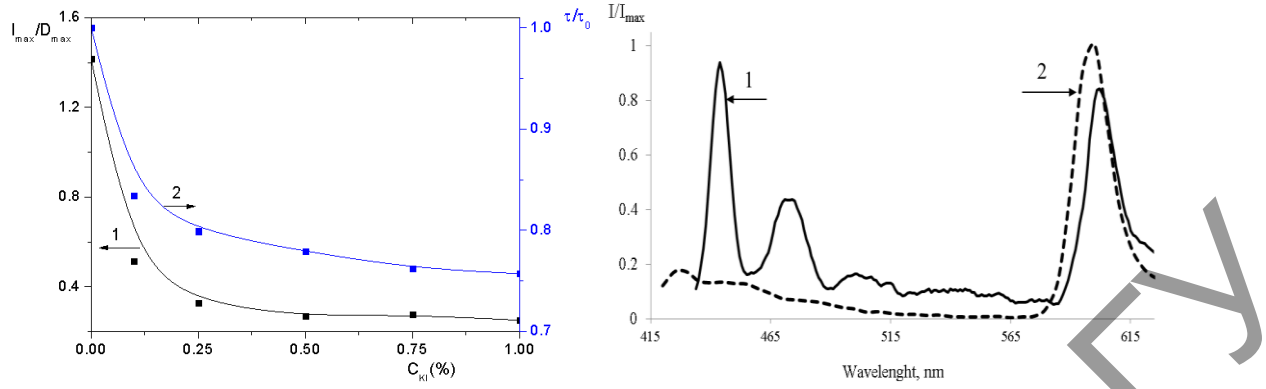
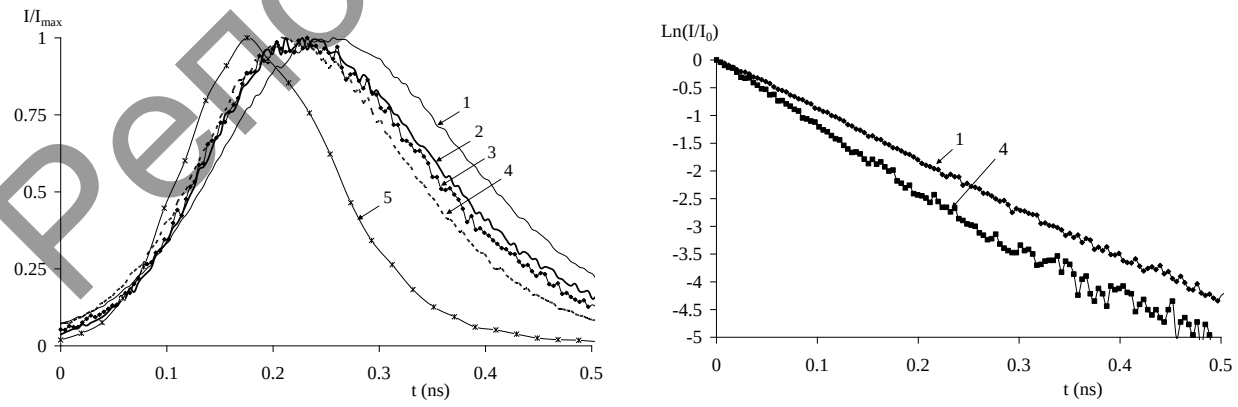


a) b)

Fig.2. Quenching of the intensity of the stationary fluorescence (1) and the lifetime (2) of the fluorescence (τ) by the impurity KI (a) and the long-term fluorescence spectra (b) of the PFO (1) and PFO KI films 1% (2).

The spectra of delayed fluorescence of a PC with a maximum at 440 nm completely coincide with the spectra of stationary fluorescence, shown in Fig. 3, b. As shown in the data of [23], the delayed fluorescence at 440 nm is the annihilation delayed fluorescence (ADF) of the PFO. A delayed fluorescence at 580 nm can be attributed to the phosphorescence of polymer [24]. Measurement of the temperature dependence of the delayed fluorescence of PFO showed that with increasing temperature, the fluorescence intensity at 440 nm and 580 nm decreases. The glow with a maximum of 580 nm is phosphorescence. It is interesting to compare the intensities of ADF and phosphorescence in PFO and PFO-KI films. The comparison shows that the phosphorescence intensity in the PFO-KI film is significantly increased in comparison with the fluorescence in the PFO film.

Thus, the addition of KI to the polymer leads to a significant increase in the concentration of excited triplet states in the PC. From the absorption spectra of the films, it follows that the addition of KI to the polymer leads to an increase in the disorder of the films. Delayed fluorescence spectra also indicate an increase in the disorder of polymer films, as seen from the shift in the short-wave side of both the spectra of the ADF and the phosphorescence spectra of the PFO-KI sample. The kinetics of PC fluorescence in the nanosecond time range was studied (Figure 3).



a) b)

Fig.3. Dependence of the fluorescence kinetics of PFO (a) and logarithmic fluorescence curves PFO (b) as a function of the impurity concentration KI:
1 - PFO; 2 – PFO - KI 0,1%; 3 – PFO-KI 0,5%; 4 – PFO-KI 1%; 5 – laser beam profile 375 nm.

The fluorescence kinetics was measured at a wavelength of 440 nm. Comparison of the form of the PC fluorescence kinetics (Figure 3, a) with the laser radiation profile (BDL-375-SMC, Becker and Hickle) shows that the curves coincide at the stage of growth of the fluorescence intensity and at a time interval of 0.2 ns from the maximum of the fluorescence intensity.

This indicates that at this time interval the shape of the PFO fluorescence curves is formed by the profile of the laser pulse. In this case, the addition of an impurity KI results in a shift of the recorded fluorescence decay curve toward short times (Figure 3, a). This may indicate the acceleration of the photographic processes occurring in the polymer film when KI is added in the time range below the time range allowed by the experimental equipment. For a longer time range, there is a monoexponential attenuation of the PFO fluorescence (Figure 3, b). The fluorescence lifetime (τ) was determined using SPCImage 3.9.4 software [24] and is shown in Table 1. The value of τ is in the range 0.4 – 0.5 ns and agrees well with the data obtained in other studies [18, 25-26]. An increase in the KI concentration in the film leads to a slight decrease in τ (Table 1). From the kinetic data obtained, it can be seen that the addition of the KI salt leads to an acceleration of the photoprocesses both at the stage of increasing the fluorescence intensity (Figure 3, a) and for longer signal acquisition times (Figure 3, b).

The fluorescence kinetics of PFO-KI films was studied in the micro- and millisecond time range. The form of the fluorescence kinetics is shown in Figure 4. The general form of the kinetic curve has an exponential form of damping. On the long-term part of the kinetic curves, having a shape close to exponential, the lifetime of the fluorescence (τ) was determined. Addition of the KI impurity to the polymer film results in a slight drop in the lifetime of both the ADF and the phosphorescence of the PFO (Table 2).

Table 2. The delayed luminescence parameters of PFO and PFO-KI films 1%

Polymer	PFO	PFO–KI
τ_{DF}, ms	1.3	1.25
τ_{PHOS}, ms	2.6	2.4

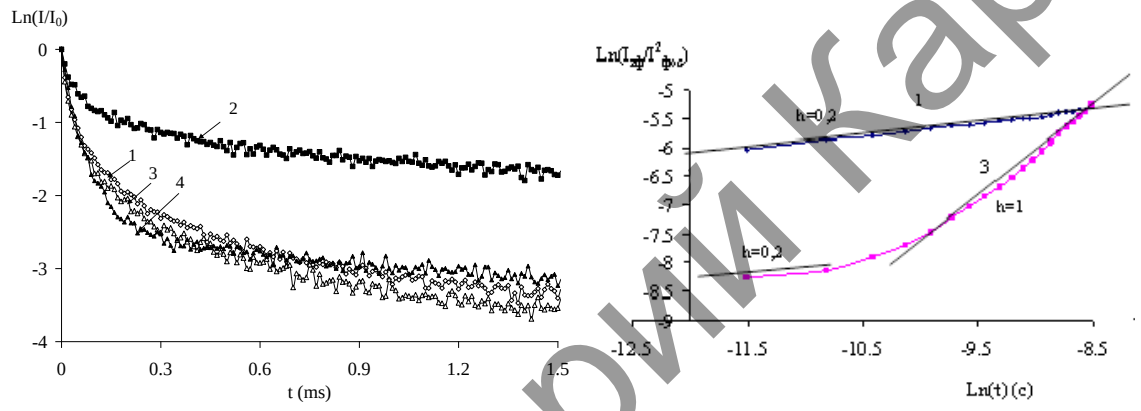
As can be seen from the spectral data, the impurity KI leads to an increase in the disorder of the PFO films (Fig. 1 and 2). The degree of influence of the disorder of the films on the character of migration of triplet excitations can be estimated using the percolation model developed in [27, 28]. In the percolation model, an important parameter is the parameter h, which characterizes the degree of local inhomogeneity of the medium. The lower limit h=0 expresses the motion in a homogeneous medium. The upper limit h=1 characterizes the motion in locally inhomogeneous clusters. To determine the parameter, a plot is plotted for the dependence of $\ln(I_{DF}/I^2_{PHOS})$ on ln(t), where I_{DF} is the delayed fluorescence intensity of the sample, and I_{PHOS} is the phosphorescence intensity. The tilt angle determines the parameter h.

An analysis of the data obtained within the percolation model showed that for a time interval of up to 200 μs, a linear dependence with the index h = 0.2 is observed for the PFO film. It shows on the walk of a triplet exciton in a practically homogeneous medium. For the PFO-KI film, the behavior of the $\ln(I_{DF}/I^2_{PHOS})$ curve versus ln(t) can be described using two linear dependences with h=0.2 and h = 1 (Fig. 4). It shows that composite film has two structurally different phases. At the initial instants of time after photoexcitation the dominant contribution in the intensity of the ADF is provided by rapidly migrating excitons in the ordered phase. The kinetics of the ADF is determined by the annihilation of triplets in the disordered phase at times greater than 30 μs.

Thus, from the data on the nature of the migration of electronic excited states in the microsecond time range, it can be seen that the addition of an impurity of KI to the polymer leads to an increase in the disorder of the film. This disorder has a large effect on the detection times above

50 μs after laser photoexcitation. The kinetic data are in good agreement with the spectral data on delayed fluorescence and phosphorescence (Fig. 4, b).

Thus, analysis of ADF kinetic shows that the addition of an impurity of KI to the polymer leads to an increase in the disorder of the film. The initial kinetics of the ADF is determined by pair annihilation of triplet excitons in the ordered phase. The annihilation in the disordered phase becomes dominant over time more than 30 μs. For increase the generation time of the electron-hole pairs in the nanosecond time range, the PFO-KI sample was cooled 1% to a temperature of 100 K. An analysis of the kinetics of fluorescence generation and quenching showed that at a low temperature, the fluorescence generation intensity maximum shifted toward longer times from the end of the action of the laser pulse (50 ps). This can be explained on the basis of Onsager's formula. With decreasing temperature, the Onsager radius will increase (r_{Ons} are the characteristic distances between the electron hole and the hole). The growth of r_{Ons} leads to an increase in the time after which recombination fluorescence occurs.



a) b)

Fig.4. The delayed fluorescence kinetics (a) and description of the kinetics of fluorescence decay (b) within the percolation model: a) kinetics of fluorescence (1, 3) and phosphorescence (2, 4) of PFO (1, 2) and PFO + 1% KI (3, 4); b) 1 - PFO; 3 - PFO + 1% KI.

The analysis of the fluorescence damping curves of PFO and PFO-KI films of 1% at temperatures of 300 K and 100 K showed that they can be described within the framework of the empirical equation of E. Becquerel describing recombination fluorescence [29]. The comparison showed that in the interval from 1.5 ns to the attenuation of fluorescence (3-3.5 ns), the fluorescence kinetics is well described by a dependence of the form (2):

$$\frac{1}{\sqrt{I / I_0}} = 1 + k_{effect} t \quad , \qquad (2)$$

where k_{effect} · is the effective rate constant for the fluorescence decay of the film.

The k_{effect} · constant, determined for the PFO-KI film, does not change its value with a temperature change and is 17 * 10^{-9} (s^{-1}). While the value k_{effect} · for the PFO film was 54 * 10^{-9} (s^{-1}). Thus, the decay rate of the recombination fluorescence in the PFO film is higher compared to the PFO-KI film. This can be attributed both to an increase in the concentration of defects in the PFO-KI film and to a change in the main type of defects in the PFO polymer upon the addition of a heavy atom. It is not ruled out that the increase in the concentration of triplet excited states in the PFO-KI film has a significant influence on the recombination fluorescence. The time-resolved fluorescence spectra of PFO and PFO-KI films were measured at 1% in the nanosecond time range.

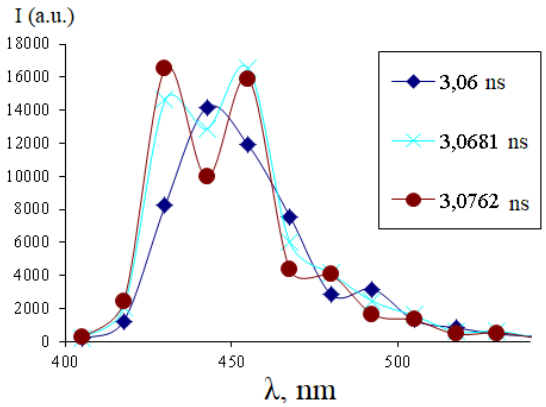


Fig.5. Time-resolved fluorescence spectra of PFO.

At the growth stage of the kinetics of the fluorescence of the films (Figure 5), the spectrum recorded for the ordered polymer phase with a maximum at 440 nm is observed. With increasing registration time, the shape of the fluorescence spectrum changes and a fluorescence from the amorphous phase of the film is observed on the decaying part of the kinetic curve. The main stages of spectrum transformation are shown in Figure 5. Comparison of the time-resolved fluorescence spectra of PFO and PFO-KI films 1% did not show any fundamental differences.

Conclusions

Spectral-luminescent properties of semiconductor films of PFO are investigated. The degree of ordering of the PC films is determined from the absorption spectra. Addition of the KI salt results in a decrease in the degree of ordering of the PFO films.

The magnitude of the vibronic splitting (ΔE) and the Huang-Riesz factor (S) were calculated for fluorescence spectra. The increase in the disorder of the PC does not lead to an increase in the value of S and does not lead to a change in the magnitude of the vibronic splitting ΔE.

The fluorescence kinetics of PFO-KI films was studied in the micro- and millisecond time range. Addition of KI to the polymer leads to an increase in the disorder of the film, as can be seen from the data on the nature of the migration of electronic excited states in the microsecond time range. This disorder has a large effect on the detection times above 50 μs after laser photoexcitation.

The decay rate of the recombination fluorescence in the PFO film is higher compared to the PFO-KI film. This can be attributed both to an increase in the concentration of defects in the PFO-KI film, and to a change in the main type of defects in the PFO polymer upon the addition of a heavy atom.

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