

Red glass absorption filters luminescence in ir spectrum region

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Using red and infrared (IR) filters in various studies in the near IR region, in particular, studying photosensitized generation of singlet oxygen and photosensitizers triplet states, in many cases filters luminescence is not taken into account. The paper shows how standard absorbing glass filters affect the luminescence signal in the near IR region.

Keywords: filter luminescence, time-resolved spectroscopy, luminescence spectra.

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Introduction

Absorbing filters are often used in the study of luminescence parameters of various objects, including in the study of IR luminescence of polyatomic and, in particular, biological molecules, to suppress the excitation radiation or to cut off radiation in the second diffraction order of the monochromator grating. One class of filters used in scientific studies are absorption filters based on colored optical glass, in which spectral selection of the incident radiation occurs due to selective absorption by absorption additives. As such additives, metal oxides (such as Mn, Co, Fe, Cu, Cr, Mo, Ti, Ni), rare-earth metals, etc., as well as colloidal dyes (such as Se, Cd, Au, Ag, etc.) can be used [1]. For such filters, transmission spectra are standardized, and sometimes absorption spectra are specified, however, indications of their own luminescence are either absent or general in nature, mentioning only that such luminescence is possible [2]. The presence of luminescence of glass filters in the visible range has been known for quite a long time [3–8]. Luminescence of some filters was studied in works, in [9,10] which luminescence spectra of the glass filter OC12 in the range 530–1040 nm [9] were obtained and studied and the filter KC19 in the range 680–880 nm and the intrinsic luminescence of the KC19 filter in the 660–1000 nm region was recorded. However, literature lacks publications devoted to systematic investigations of the intrinsic luminescence parameters of glass absorbing filters, especially in the near-IR spectral region.

The absence of this information leads to the fact that when working with filters, the possible occurrence of luminescence is often not taken into account, which can cause distortion of experimental results due to detection of parasitic emission signals of the filter together with signals from the studied sample, while the magnitude of the parasitic signal can significantly exceed the useful signal from the sample. Red and IR glass filters are used, for example, in the study of phosphorescence signals of photosensitizers and singlet oxygen in the IR spectral

region with the aim of cutting off the fluorescence of the photosensitizer in the second diffraction order on the monochromator grating [11–17].

Besides absorbing glass filters, interference filters are actively used for studying IR luminescence parameters, which represent a substrate onto which thin layers with different refractive indices are deposited and a blocking absorbing filter to block additional interference peaks. In particular, interference filters with a transmission peak around 1270 nm are actively used for detecting singlet oxygen luminescence [18–20].

In the present work, a study was conducted of the spectra and luminescence lifetimes of blocking red and IR absorbing glass filters from an optical glass [21] catalog, as well as a narrowband interference filter with a transmission band in the luminescence region of singlet oxygen.

Materials and Methods

The work studied glass absorbing filters KC11, KC17, KC18, KC19, IKC3, and IKC7, [21], which contain elements Cd, Se, S in different proportions [1]. The short-wavelength edge of the transmission band of the studied filters is presented in Table 1. [21]. Also studied was a narrowband interference filter (bk-1270-10-B, Interferenzoptik Elektronik GmbH) with a transmission band 1270 ± 3 nm, frequently used for detection of singlet oxygen luminescence. The spectral resolution of the studied signals was performed using the MDR12 monochromator (LOMO). For signal registration in the visible range, a diffraction grating with 1200 lines/mm was used, and in the IR range — 600 lines/mm with the same input and output slit parameters. The monochromator slit width was, when possible, set to values close to those usually used for sample measurements: around 0.3 mm for the visible region, and 1–2 mm for the IR region. Excitation laser radiation was directed onto the monochromator input slit, and the studied filters were installed directly in front of the input slit. For measurement of signals in the visible spectral

Table 1. Lower edge of transmission bands of filters λ_{trans}^0 , luminescence maxima under excitation by laser radiation at wavelengths 405, 510 and 660 nm

Filter	λ_{trans}^0 , nm	λ_{max}^{405} , nm	λ_{max}^{510} , nm	λ_{max}^{660} , nm
KC11	630	< 950	965	—
KC17	690	< 950	995	—
KC18	710	990	1015	1094
KC19	720	1024	1055	1136
IKC3	880	1018	1050	1176
IKC7	950	1026	1050	1166

range, a silicon photodiode SPD-100 (Ioffe Institute, Russia) with a sensitivity band of 400–1100 nm was used, in the IR region, detection was performed using a PMT H10330B-45 (Hamamatsu) with spectral sensitivity in the range 950–1400 nm. As luminescence excitation sources, laboratory-made pulsed semiconductor lasers at wavelengths 405 nm (beam cross-sectional area $S = 2 \text{ mm}^2$), 510 nm ($S = 5 \text{ mm}^2$) and 660 nm ($S = 5 \text{ mm}^2$) were used. Laser power was selected so that the luminescence signal was sufficiently intense for a good signal-to-noise ratio, but did not cause detector overload. During spectra acquisition, lasers operated in pulse mode with 100 ns pulse duration and 100 kHz pulse repetition rate. To obtain time-dependent characteristics of luminescence signals, a multichannel time-correlated single photon counting module of own design with 10 ns resolution was used, which allowed changing the laser pulse repetition frequency [22]. Depending on the characteristic luminescence decay time, laser pulse repetition rates of 100 or 40 kHz were used in experiments, with pulse duration of 15 ns.

Experimental results and their interpretation

Figure 1 shows the luminescence spectra of colored glasses KC11, KC17, KC18, KC19 in their transmission band under excitation by laser radiation at wavelengths 405 nm (a) and 510 nm (b) (laser power 10 W, monochromator slit width 0.3 mm). For colored glasses IKC, luminescence in the visible region was not observed. Under excitation with a 660 nm laser, visible luminescence was not observed in any of the colored glasses used. As seen from Fig. 1, luminescence spectra under excitation at different wavelengths have different characteristics. When excited at 405 nm, luminescence in the red range was more intense or comparable to the luminescence in the IR range, whereas at 510 nm excitation, the luminescence signal was more intense in the IR region. It is presumably due to luminescence at different excitation wavelengths originating from different excited states of the luminophore molecule. It should be noted that the spectra obtained in Fig. 1 are valid for glasses with thicknesses of 3 and 5 mm. In addition, we

observed luminescence of the glasses in the red range under 405 nm excitation by laser pulses of various durations, from picosecond to continuous wave. Thus, it can be concluded that luminescence of filters should be expected for all filters with similar composition, regardless of the frequency and temporal parameters of the excitation radiation.

Figure 2 shows luminescence spectra of colored glasses KC and IKC in their transmission band in the IR range under excitation at 405 nm. For KC glasses, the excitation power was $800 \mu\text{W}$ with a monochromator slit width of 0.3 mm, while for IKC-type glasses, 10 mW excitation power was used with a monochromator slit width of 1 mm. The decrease in excitation laser power by a factor of 10 compared to studies in the visible range was due to the higher sensitivity of the PMT compared to the silicon photodiode. As seen in Fig. 2, intense luminescence was observed for filters KC11, KC17, and KC18, while for the KC19 filter luminescence was more than 5 times weaker at the same excitation power. For IKC filters, to achieve a signal comparable to KC filters, it was necessary to increase the excitation power to 10 mW and widen the monochromator slit to 1 mm.

Figure 3 shows luminescence spectra of colored glasses KC11, KC17, KC18, KC19, IKC3, and IKC7 in the IR spectral region under excitation at a wavelength of 510 nm. Here, the registration of luminescence spectra of KC filters was carried out under laser excitation with a power of 1.2 mW and slit widths of 0.5 mm, while for IKC filters the excitation power was increased to 9.5 mW and the slit width to 1.5 mm. It is evident that the character of the luminescence spectrum under excitation at 510 nm is approximately the same as at 405 nm, but there is a shift of the luminescence maximum toward the red region of the spectrum. This is likely related to the decrease in the energy of the excited energy levels as the energy of the excitation photons decreases.

Figure 4 shows IR luminescence spectra of filters KC18, KC19, IKC3, and IKC7 under excitation with laser radiation at a wavelength of 660 nm and power of $500 \mu\text{W}$ with a slit width of 0.3 mm. As can be seen, for this excitation wavelength there is a shift of the luminescence maximum toward the red spectral region compared with the position of the luminescence maximum obtained under excitation at 405 and 510 nm. The positions of the luminescence maxima of these filters are presented in Table 1.

Thus, it can be concluded that for studying IR luminescence of samples, IKC-type filters are preferable due to the lower intensity of luminescence. However, even IKC-type filters can contribute significantly to the total detected luminescence signal, especially for weak signals from the studied sample. Therefore, to minimize parasitic luminescence signals of filters, instead of placing the filter before the monochromator entrance slit, the authors of this article usually place the absorbing filter after the monochromator exit slit, directly in front of the photodetector input window.

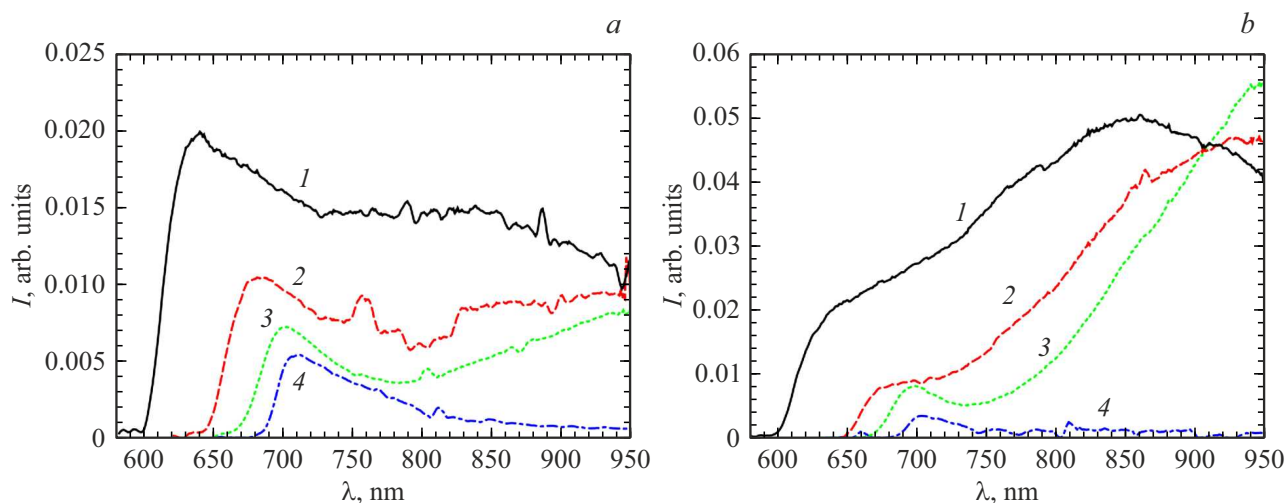


Figure 1. (a) Luminescence spectra of red colored glasses KC in their transmission band under excitation by laser radiation at wavelengths (a) and 510 nm (b). Filter designations in figures a and b: 1 — KC11, 2 — KC17, 3 — KC18, 4 — KC19.

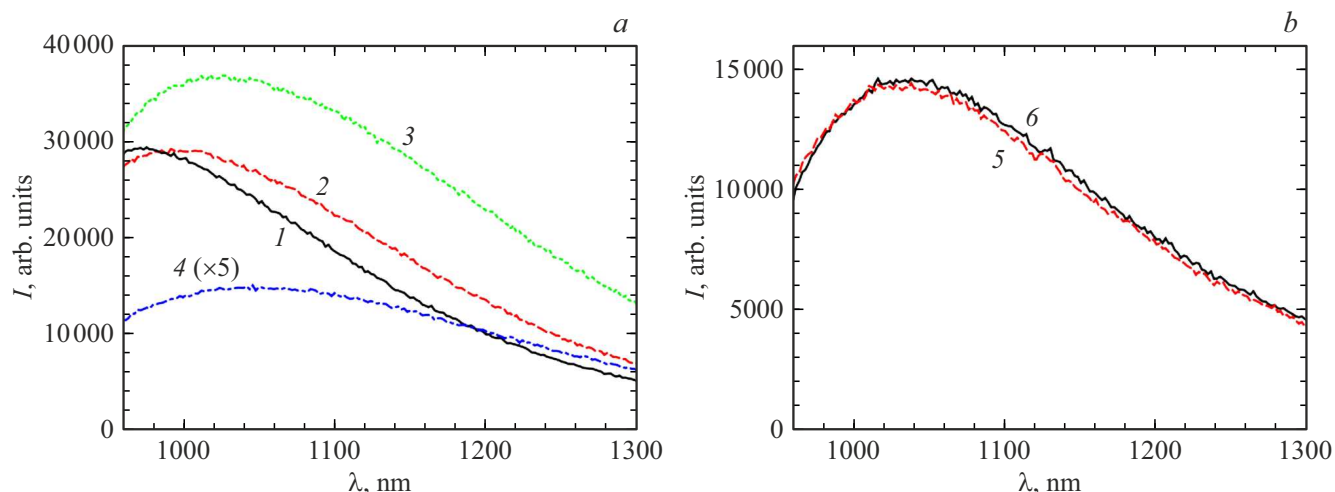


Figure 2. (a) IR luminescence spectra of KC-type filters under excitation at 405 nm: KC11 (1), KC17(2), KC18(3), KC19(4). For the KC19 filter, the luminescence signal on the graph is amplified 5 times relative to the actual signal. (b) IR luminescence spectra of filters IKC3 (5) and IKC7 (6) under excitation at 405 nm.

Table 2. Luminescence lifetimes of filters under laser excitation at wavelengths 405, 510, and 660 nm

Filter	$\tau_1^{405}, \mu s$	$\tau_2^{405}, \mu s$	$\tau_1^{510}, \mu s$	$\tau_2^{510}, \mu s$	$\tau_1^{660}, \mu s$	$\tau_2^{660}, \mu s$
KC11	0.3 ± 0.1	2.0 ± 0.2	0.2 ± 0.1	1.7 ± 0.2	—	—
KC17	0.5 ± 0.1	2.9 ± 0.2	0.3 ± 0.1	2.3 ± 0.2	—	—
KC18	0.7 ± 0.1	3.5 ± 0.2	0.4 ± 0.1	3.1 ± 0.2	1.2 ± 0.1	8.3 ± 0.3
KC19	0.6 ± 0.1	3.2 ± 0.2	0.5 ± 0.1	3.5 ± 0.2	0.3 ± 0.1	6.4 ± 0.2
IKC3	0.3 ± 0.1	1.9 ± 0.2	~ 0.2	—	~ 0.1	—
IKC7	0.2 ± 0.1	1.7 ± 0.2	~ 0.1	—	~ 0.1	—

To determine the nature of the luminescence of the studied filters, time-resolved experiments were carried out. Decay curves of luminescence were obtained for all studied filters under laser excitation at wavelengths 405, 510, and 660 nm (except for filters KC11 and KC17, which do not

absorb at 660 nm). Figure 5 shows typical time-resolved luminescence decay curves for filters under excitation at 405 nm. For all filters, registration of luminescence decay signals was conducted at wavelengths 1000 and 1100 nm. Approximation of the obtained signals by a two-

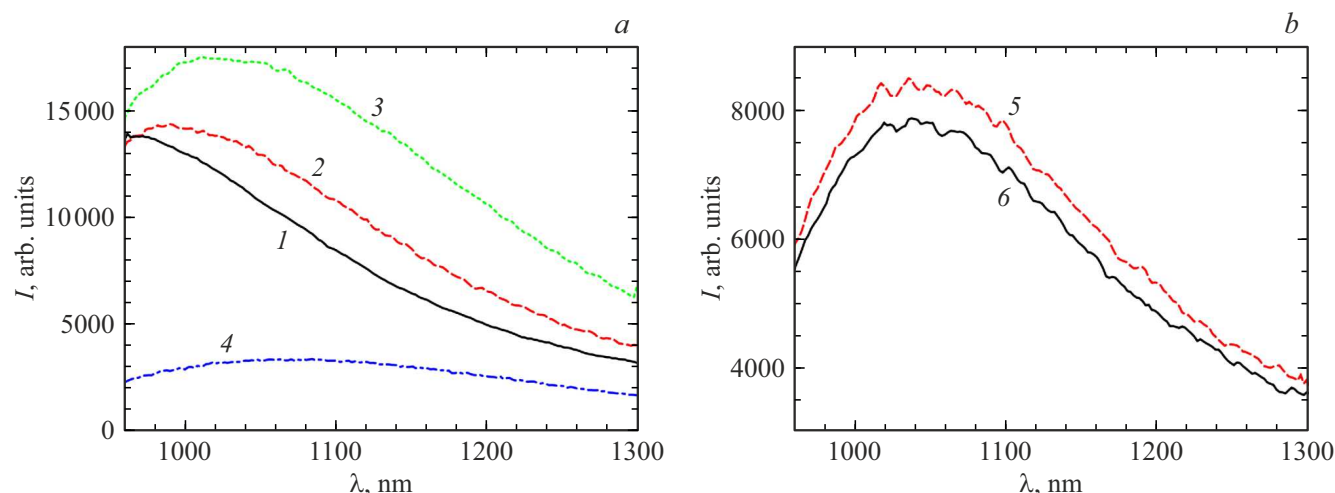


Figure 3. (a) IR luminescence spectra of KC-type filters under excitation at a wavelength of 510 nm: KC11 (1), KC17 (2), KC18 (3), KC19 (4). (b) IR luminescence spectra of filters IKC3 (5) and IKC7 (6) under excitation at a wavelength of 510 nm.

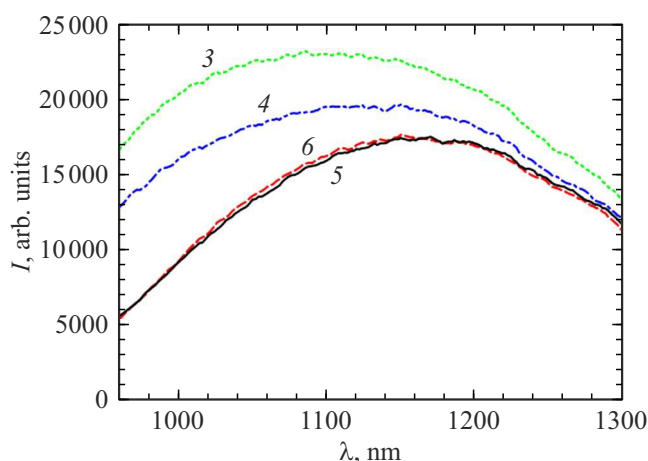


Figure 4. IR luminescence spectra of filters KC18 (3), KC19 (4), IKC3 (5), IKC7 (6) under laser excitation at wavelength 660 nm.

exponential function showed that the temporal parameters of the signals were practically identical when registered at these two wavelengths. Table 2 presents the obtained luminescence lifetimes of the filters. For all types of filters, one of the obtained luminescence decay times was about several hundred nanoseconds, and the other about a few microseconds. It is evident that luminescence decay times strongly differ depending on the excitation wavelength. For KC filters, an increase in the second lifetime is observed with increasing excitation wavelength, whereas for IKC filters, on the contrary, an increase in excitation wavelength leads to rapid decay of the luminescence signal, with the decay time in our experiment approximately 100 ns.

Thus, when studying long-lived phosphorescence signals with lifetimes $1\ \mu\text{s}$ and longer, IKC-type filters practically do not distort the phosphorescence signal from the sample if excitation occurs at wavelengths 510 and 660 nm, while

KC filters exhibit lifetimes on the order of microseconds for all studied excitation wavelengths.

In addition to colored glasses, experiments were conducted with the interference glasses filter (bk-1270-10-B, Interferenzoptik Elektronik GmbH) with a transmission band $1270 \pm 3\ \text{nm}$ intended for detecting phosphorescence signals of singlet oxygen. Figure 6 shows luminescence spectra of this filter under laser excitation at wavelengths 405 and 660 nm. As seen in Fig. 6, an intense luminescence band of this filter was observed near 1270 nm at both excitation wavelengths, and therefore could introduce significant error when detecting singlet oxygen phosphorescence signals. A solution to this problem may be to place a silicon plate before the interference filter, which effectively blocks excitation laser radiation with wavelengths shorter than 1100 nm. In this case, the observed luminescence signal is

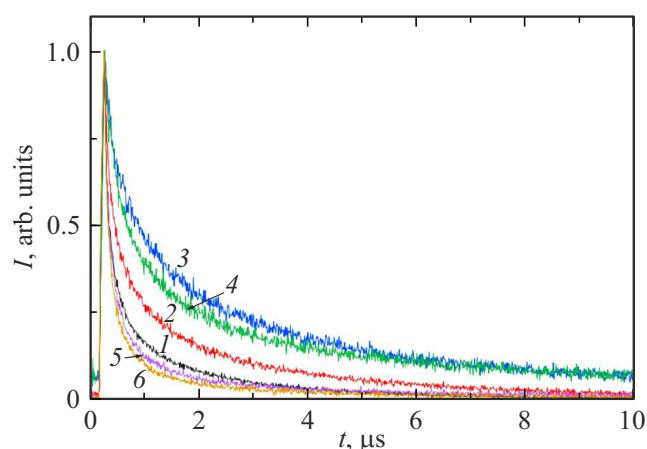


Figure 5. Typical time-resolved decay curves of phosphorescence of filters KC11 (1), KC17 (2), KC18 (3), KC19 (4), IKC3 (5) and IKC7 (6) under laser excitation at 405 nm and detection at 1100 nm, normalized to the signal maximum.

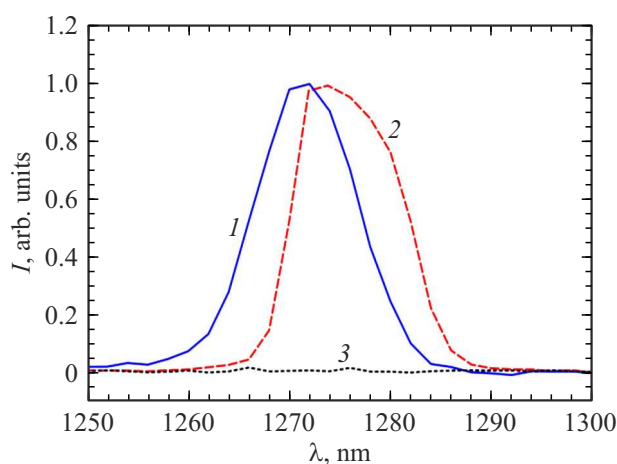


Figure 6. IR luminescence spectrum of interference filter bk-1270-10-B (Interferenzoptik Elektronik GmbH) with a transmission band 1270 ± 3 nm under excitation at wavelengths 405 (1) and 660 nm (2). Curve 3 corresponds to the signal obtained when a silicon filter is placed before the interference filter.

at the PMT noise level, as shown in Fig. 6 (curve 3). Since the exact construction of the studied filter is unknown, no definitive conclusion can be made about the cause of the phosphorescence shown in Fig. 6 (curves 1 and 2), but it can be assumed that this effect is due to the presence of a blocking absorbing filter in the interference filter assembly.

During the experiments, we also showed that the input window of the PMT H10330B-45, which is equipped with an optical filter, can also phosphoresce when visible light enters it. In particular, when laser radiation with a wavelength of 405 nm hits the PMT input window, phosphorescence in the IR range with a characteristic lifetime $\tau = 10.3 \pm 0.2 \mu\text{s}$ is observed. Tests for luminescence of the PMT input window with lasers at 510 and 660 nm were not performed due to the possibility of PMT „crosstalk“. However, in other experiments, sensitivity of this PMT to wavelengths in the range 640–700 nm was observed.

Conclusions

Thus, it was shown that when using any absorption filters, preliminary measurements of possible luminescence of these filters in their transmission band are necessary. If absorbing filters are used in a setup with a monochromator, it is necessary, if possible, to place the absorbing filter after the monochromator output slit directly before the detector input window. If the filter must be installed close to the sample, preference should be given to IKC-type filters. When using an interference filter to isolate singlet oxygen phosphorescence signals near 1270 nm, it is advisable to place an additional absorption filter a silicon plate blocking radiation with wavelengths shorter than 1100 nm before it. When detecting signals with PMT H10330B-45 (Hamamatsu), the

possibility of phosphorescence of the PMT input window under visible radiation should be taken into account.

Conflict of interest

The authors declare that they have no conflict of interest.

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