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Nonlinear optical limitation of laser radiation power in the ultraviolet and visible ranges by bis-phthalocyanines of the clamshell type

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UV and visible laser radiation poses a high risk to the eyes. Interaction of a beam with a high energy fluence with eye tissues causes damages, including irreversible damage to the cornea or lens. The visible laser radiation destroys a retina. Dangerous level of radiation is achieved in nanosecond pulses, which makes it difficult to use active protective equipment. In a time shorter than the pulse duration, the attenuation of laser radiation is ensured by the use of nonlinear optical materials, for which clamshell-type bis-phthalocyanines were studied & metal complexes **1a, b** (**a** — Zn, **b** — Mg) and the original ligand **2**. The experiments were carried out in UV and visible range of the spectrum at wavelengths 355, 405 and 532 nm. Using the correlation method for assessing the efficiency of optical limitation (CORRELATO algorithm), it was shown that metal complexes have priority over the ligand, while the maximum efficiency was found for zinc complex **1a** in the UV range.

Keywords: optical limiting, phthalocyanines, *Z*-scan, nonlinear absorbers.

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Introduction

The relevance of modern fundamental scientific research in nonlinear optics is justified by the active development of technologies and engineering solutions using high-intensity laser radiation (laser lidars [1,2], altimeters-rangefinders [3], target designators [4], location systems [5], industrial and medical units [6–8]). The spectral range of laser devices extends from 350 to 1100 nm, while ultraviolet (UV) radiation poses a danger to the organs of vision in that it does not activate the protective blink reflex [9]. As a result of such exposure, irreversible damage to the cornea or lens of the eye may occur [10]. Radiation in the visible range is characterized by lower photon energy, but such radiation is additionally focused on the retina of the eye and reaches high energy flux densities [11]. To attenuate laser radiation in a time shorter than the duration of a nanosecond pulse, various nonlinear optical materials are being developed [12,13]. To create passive protection, it is required for the material to darken when the energy fluence reaches a dangerous level.

The vast majority of laser devices operate in the visible and near-IR regions of the spectrum, which is explained both by the use of reliable solid-state lasers with the generation of additional harmonics [14,15] and photodetecting equipment for recording radiation [16], and also features of the propagation of radiation in the atmosphere [17]. The high powers of modern laser units require the creation of materials and devices to protect photodetector matrices and organs of vision from damage when exposed to high-intensity laser radiation in the spectral range from 350 to 1100 nm. In connection with this, an important task is to evaluate nonlinear optical properties specifically in the UV range, since due to the high energy of photons, the effect of saturated absorption [18–20] is often observed, and to create a limiter of laser radiation, the effect of inverse saturation is necessary absorption [21].

1. Materials and research techniques

To carry out the research, zinc (**1a**) and magnesium (**1b**) bis-phthalocyaninates were synthesized, the macrocycles of which are linked by a cyclotriphosphazene spacer (Fig. 1),

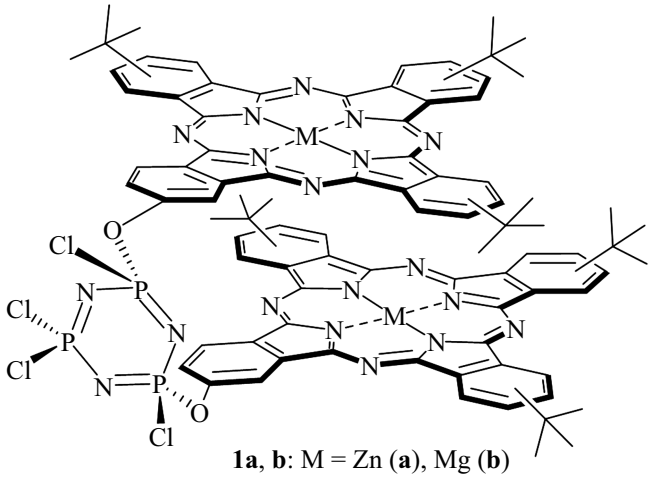

Figure 1. Structural formulas of bimacrocyclic dyes for optical limiters.

as well as the precursor — ligand **2**. In order to specify the influence of the metal (or its absence) on the nonlinear optical (NLO) properties of dyes in optical limiting.

An experimental study of the NLO properties of dyes **1,2** was carried out using the Z scanning method with an open aperture [9,12,21] and control of the pulse duration after passing through the optical medium, by analogy with the previously used technique [9]. The efficiency of dyes in optical limiting was assessed by the method of correlation analysis, which includes two coordinates — aggregation (σ_1) and NLO response (σ_2) from the point of view of quantum mechanics. The corresponding analytical expressions are presented below:

1.1. Materials

The synthesis of metal complexes **1a,b** was carried out by analogy with the precursor — ligand **2** according to the method [22], starting from the corresponding 2-hydroxyphthalocyanines [23,24]. Sodium hydride (0.3 mmol) was added to solutions of zinc and magnesium complexes of 2-hydroxyphthalocyanine (0.1 mmol) in absolute benzene (5 ml). After brief stirring, hexachlorocyclotriphosphazene (0.25 mmol) was added to the reaction mixtures. The target complexes **1a,b** after isolation on Bio-Beads SX-1 (BIORAD®) were obtained in yields of approximately 25%.

1.2. Open-aperture Z-scan method

The prepared samples were examined using the Z scanning method [25–27] with an open aperture. For studies in the UV and visible wavelength ranges, LS2147 (Lotis Tii, Belarus) and LS2145-OPO (Lotis Tii, Belarus) lasers were used as radiation sources. The diagram of the experiment is shown in Fig. 2. The total pulse energy 500 μJ (532 nm) was chosen so that on the Z scanning curve with an open aperture, areas with transmittance equal to linear far from the waist were visible. For the same reason, a pulse with energy 300 μJ was used at laser radiation wavelengths of 355 and 405 nm. It is the energy fluence that hits the sample that changes with this research method. The energy pulse increases due to the focusing of laser radiation, and the sample moves along the direction of beam propagation using a motorized slider 8MT50-200BS1 (Standa, Lithuania), and the values of the transmitted total pulse energy are recorded at each fixed position of the sample relative to the focus of the lens using a PD10 energy detector (Ophir, Israel). The system, which combines a DSOS604A oscilloscope (Keysight Technologies, USA) and two OD-08A photodetectors (Avesta, Russian Federation), allowed to control the pulse duration before and after interaction with the studied samples. For selective setting the total pulse energy, a polarization motorized attenuator OAGP-10-S (Avesta, Russian Federation) was used.

2. Theory

The solution to the radiative transfer equation (RTE) [21] for the general dependence of the absorption coefficient μ on intensity I is written by the expression

$$\int_{I_0(\rho,\varphi,t)}^{I(\rho,\varphi,t)} \frac{1}{I\mu(I)}\,dI = -d, \tag{1}$$

where I_0 — incident intensity, ρ and φ — polar coordinate system, t — time and d — material thickness. Meanwhile, during the experiment, the values of the total pulse energy U are determined, from which the values of the normalized transmittance T_{norm} are calculated. Intensity I in the general case is determined by the total pulse energy by the formula

$$I = UA(\rho)B(\varphi)C(t), \tag{2}$$

where $A(\rho)$ — radial pulse profile; ρ — distance from the laser beam axis; $B(\varphi)$ — angular dependence of the pulse profile; φ — angular coordinate and $C(t)$ — pulse shape in time t.

Often, to process the results of experiments on the interaction of laser radiation with a nonlinear medium, which consists of approximating the relationship between normalized transmittance and intensity, the postulate is used that laser pulses have a Gaussian shape, and this makes it possible to implement many theories [21,28,29]. However, it is not always possible to obtain a solution in elementary functions. Meanwhile, it was noted that the nonlinear optical response weakly depends on the pulse shape and is determined mainly by the energy parameters of the laser. Therefore, in calculations it is advisable to use expressions for the rectangular pulse shape, for which an analytical

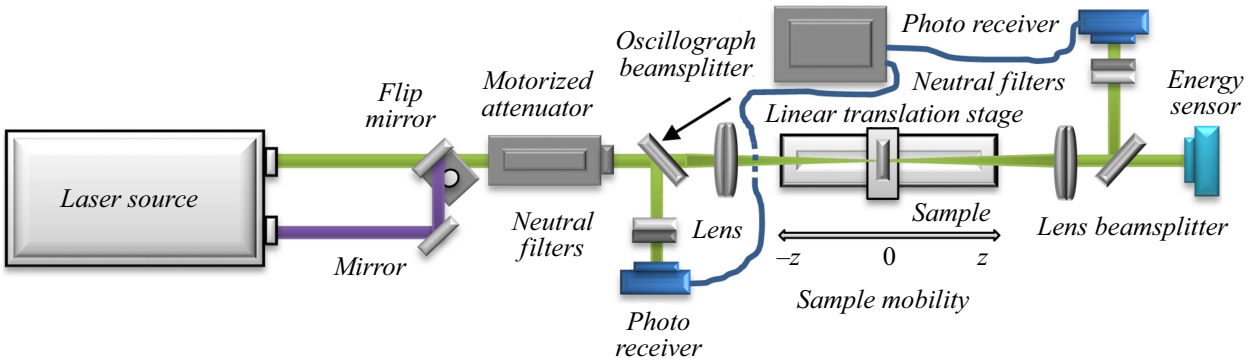

Figure 2. Diagram of the unit for identifying media with nonlinear optical properties.

solution can almost always be obtained:

$$\begin{cases} A(\rho) = \dfrac{1}{w^2\pi}, & 0 \le \rho \le w, \\ B(\varphi) = 1, & 0 \le \varphi \le 2\pi, \\ C(t) = \dfrac{1}{\tau}, & 0 \le t \le \tau. \end{cases} \quad (3)$$

The values of nonlinear transmittance (the ratio of the total energies of transmitted and incident pulses) in the experiment are normalized to linear transmittance (determined by the linear absorption coefficient α) [30,31]. After including the threshold intensity I_{thr} in the expression for the absorption coefficient $\mu(I)$ as follows:

$$\mu(I) = \alpha + \beta(I - I_{\text{thr}})\eta(I - I_{\text{thr}}), \quad (4)$$

and substituting this equation into (1), taking into account the general expression for the intensity (2) and the nature of the pulse intensity distribution (3), we obtain a formula for determining the normalized transmission from the RTE solution [21] in the following form:

$$T_{\text{norm}} = \exp\left(-\beta\left(\frac{U_0}{w^2\pi\tau} - I_{\text{thr}}\right)d\right), \quad (5)$$

where β — nonlinear absorption coefficient, I_{thr} — threshold intensity, U_0 — total energy of the incident pulse, w — beam radius, τ — pulse duration and η — Heaviside step function, which has a value of 1 at an intensity exceeding the threshold value, and 0 — at a lower intensity.

In the UV range, there can be a saturated absorption effect [32–34], which is characterized by a decrease in linear transmittance. In this case, the dependence of the absorption coefficient is given by the saturation intensity I_s [18,35,36] and the nonlinear absorption coefficient according to the formula [37–39]:

$$\mu(I) = \frac{\alpha J_s}{I_s + I} + \beta I. \quad (6)$$

From the solution of the RTE we obtain the value of the normalized transmittance in the case of the dependence of the absorption coefficient (6) for a rectangular pulse shape (3) taking into account (2) in the following form:

$$T_{\text{norm}} = \exp\left(-\left(\frac{\beta U_0}{w^2\pi\tau} - \frac{\alpha U_0}{I_s w^2\pi\tau + U_0}\right)d\right). \quad (7)$$

Figure 3 shows the dependence obtained using the Z-scanning method with an open aperture at a wavelength of 355 nm for the dye **2**. The saturated absorption effect is characterized by an increase in normalized transmittance. At each point, 30 measurements were made. Figure 3, *a* shows data on 10 points and approximation by equation (7). Figure 3, *b* shows data for standard measurements and indicates the upper and lower limits of the confidence interval. A statistical study of errors was carried out in 30 computational experiments using the t Student's t-test with a significance level of 0.01. For metal-free phthalocyanine **2** the linear absorption coefficient (α) was $2.68 \pm 0.02\,\text{cm}^{-1}$, the nonlinear absorption coefficient $\beta = 10 \pm 1$ cm/GW and the saturation intensity $I_s = 0.06 \pm 0.01\,\text{GW/cm}^2$.

Figure 4 shows Z scanning data with an open aperture for bis-phthalocyanine **1a** at a wavelength of 405 nm, showing that in the case of the zinc complex **1a** absorption saturation is not achieved. Figure 4, *a* shows the results of 10 measurements at each point and approximation by equation (5). In accordance with this expression (5), obtained for the threshold dependence of the absorption coefficient (4), the following values were determined: $\beta = 97$ cm/GW and the threshold energy fluence $F_{\text{thr}} = 0.02\,\text{J/cm}^2$ for the measured dependence of the normalized transmittance on the sample displacement, indicating the upper and lower limits of the confidence interval (Fig. 4, *b*).

To compare the efficiency of dyes in optical limiting, we used previously created correlation models based on the calculation of the σ_1 and σ_2 [12,40] descriptors:

$$\sigma_1 = \lg(\alpha^{-2}\beta^{-1}), \quad (8)$$

$$\sigma_2 = \frac{k_A\beta}{\text{DR} \cdot F_{\text{thr}}}, \quad (9)$$

where k_A — attenuation coefficient, F_{thr} — threshold energy fluence, and DR — dynamic range.

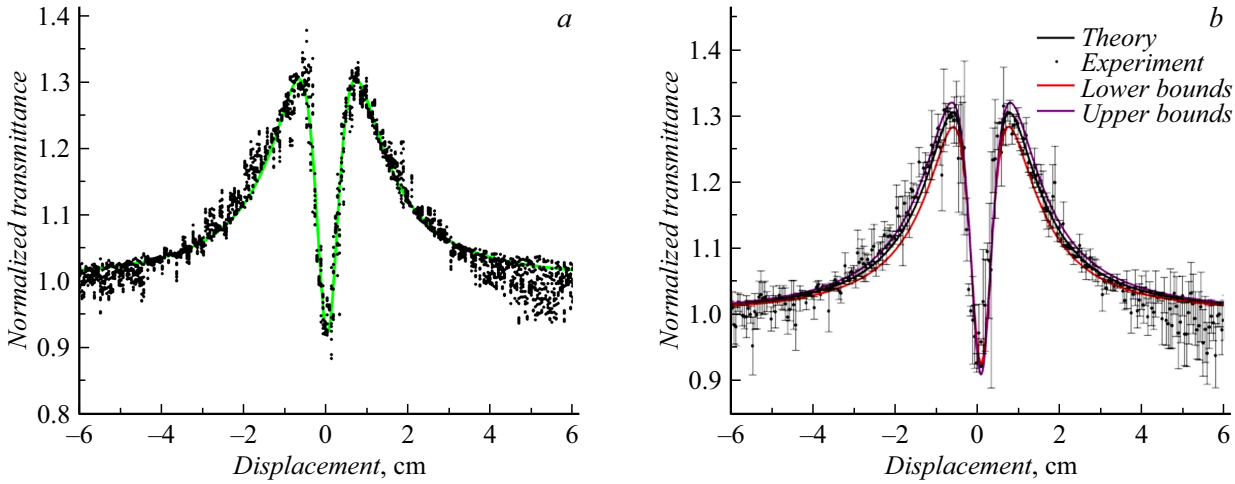

Figure 3. *Z*-scan data with an open aperture for metal-free phthalocyanine **2**: *a* — data from 10 measurements are given; *b* — theoretical curve and its upper and lower limits of the confidence interval.

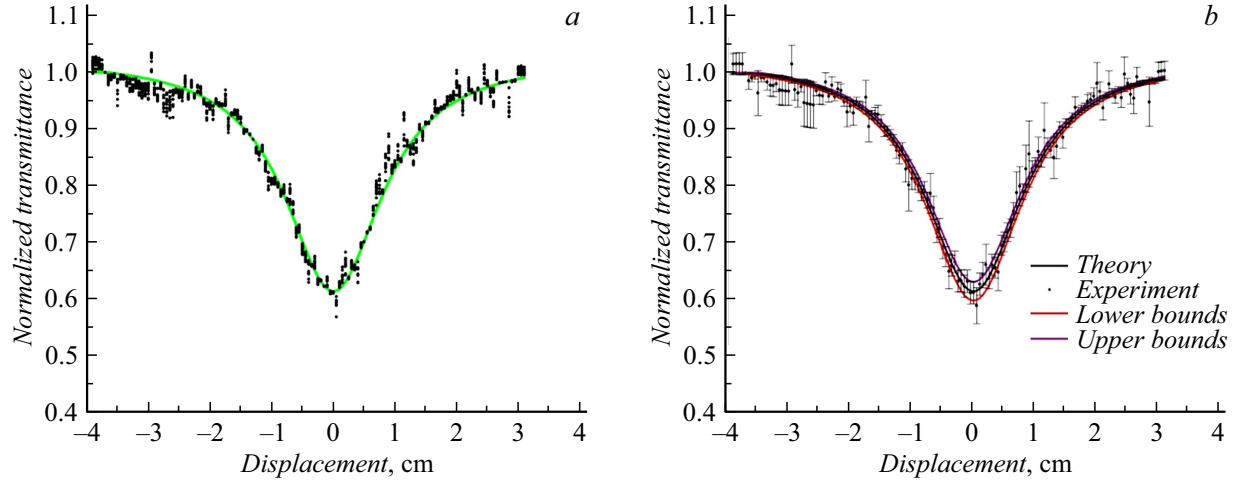

Figure 4. *Z*-scan data with an open aperture for phthalocyanine **1a**: *a* — data from 10 measurements are given; *b* — theoretical curve and its upper and lower limits of the confidence interval.

This approach was required because the dye concentrations differed in the experiments, and the parameters included in these expressions consistently influence each other. In the previous studies, we found that the more the σ_1 value is shifted to the negative region, the less aggregation affects the optical confinement. In turn, the larger the σ_2 value, the higher the nonlinear optical response of the dye from the point of view of quantum mechanics. The combination of these two coordinates allowed us in this work to move on to analytical expressions that determine the boundary conditions for sorting dyes according to the principle „bad−good “ in relation to aggregation and their NLO characteristics for the creation of optical limiters.

3. Results

For phthalocyanines **1,2**, studies were carried out at wavelengths of 532 and 405 nm with pulse durations of 10 and 7 ns, respectively. A change in the pulse duration was detected when the sample was displaced relative to the lens focus.

3.1. Measurements of the normalized transmission using the *Z*-scan method with an open aperture

In the visible range, the effect is observed only from the process of reverse saturation of absorption; the corresponding data are shown in Fig. 5 for phthalocyanines **1a** (Fig. 5, *a*) and **1b** (Fig. 5, *b*) at wavelengths of 405 and 532 nm. For phthalocyanine **2** in the UV range at a wavelength of 355 nm, both the effect of saturated absorption and reverse saturation of absorption were detected (Fig. 5, *c*). Formula (6) is used to describe such interaction. At a wavelength of 532 nm for phthalocyanine **2** there is an effect of reverse saturation of absorption. It was found that as the lens

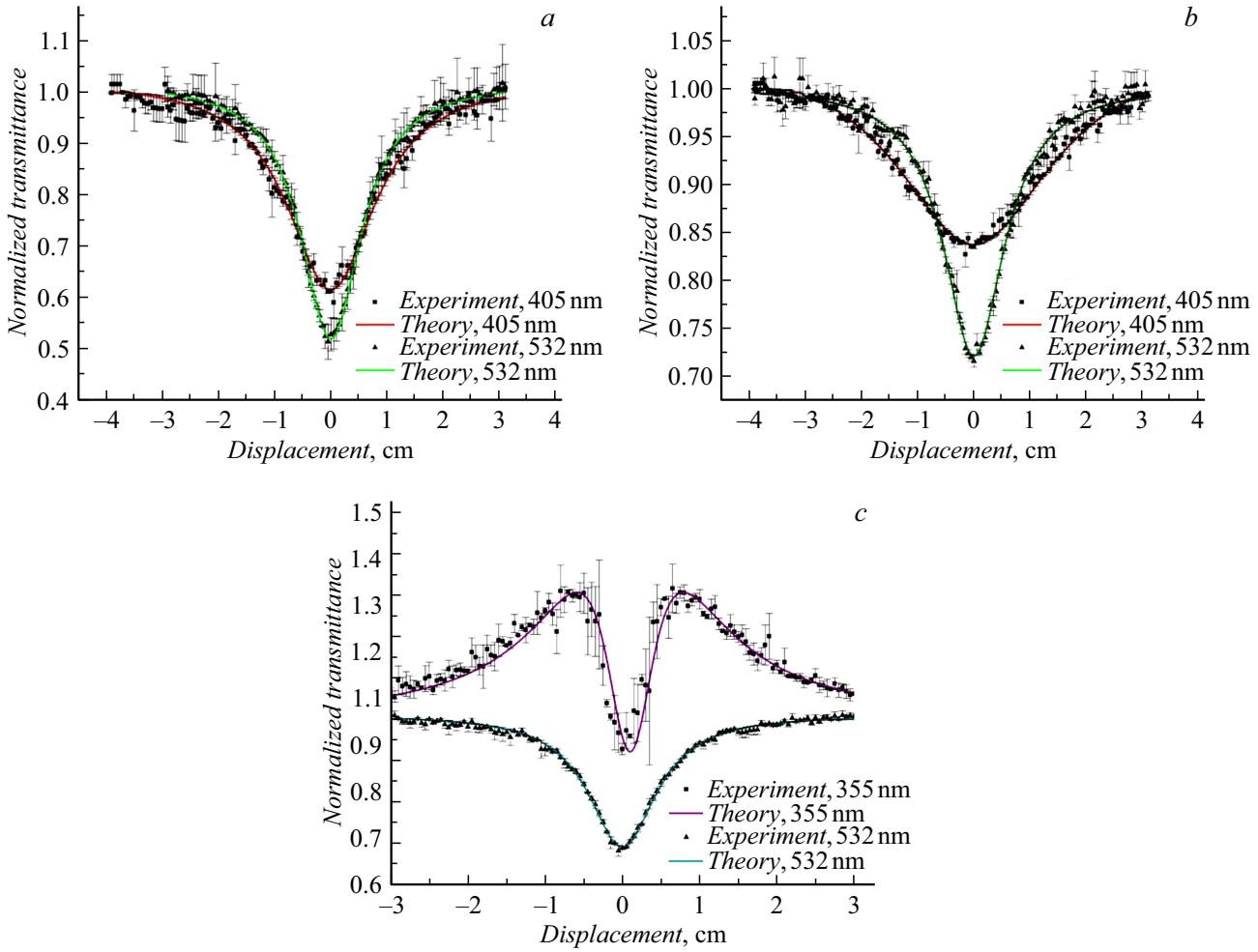

Figure 5. *Z* open-diaphragm scan data for dyes: **1a** (*a*), **1b** (*b*), **2** (*c*).

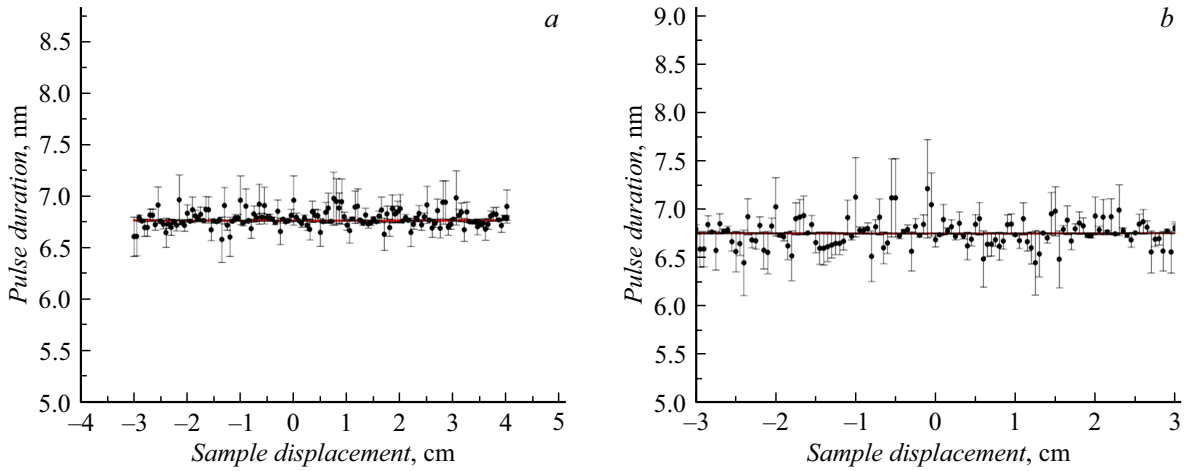

Figure 6. Changing the pulse duration at a wavelength of 405 nm for dyes:**1a** (*a*), **1b** (*b*).

approaches the focus, the pulse duration decreases, as shown in Figs. 6 and 7 for wavelengths 405 and 532 nm, respectively. Figures 6, *a* and 7, *a* show the results for phthalocyanine **1a**, and Fig. 6, *b* and 7, *b* data for phthalocyanine **1b**.

As the sample approaches the lens focus and nonlinear absorption increases, a change in the width of the transmitted pulse is detected. After such interaction, the pulse duration decreases by approximately 20%. The effect of changing the pulse duration is associated with reverse

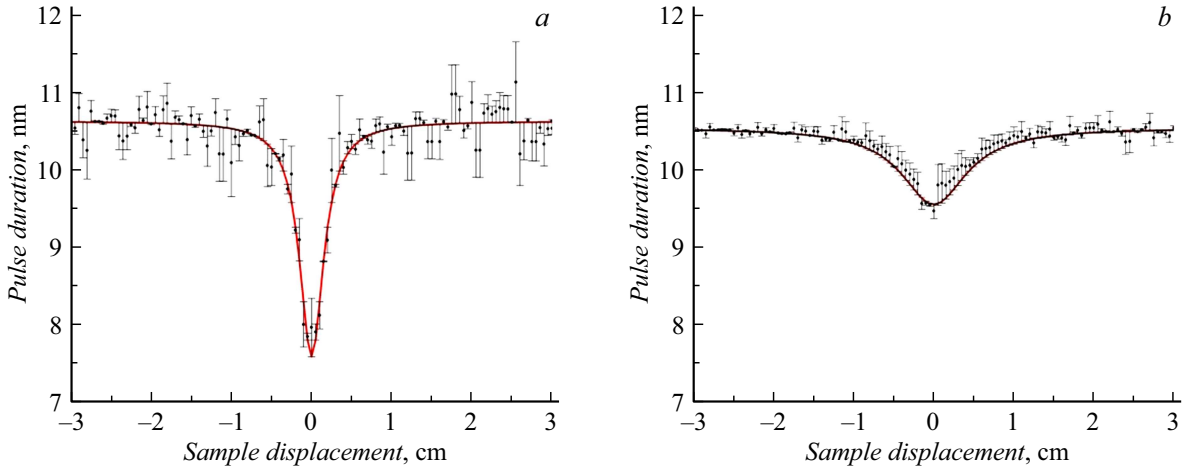

Figure 7. Changing the pulse duration at a wavelength of 532 nm for dyes:**1a** (*a*), **1b** (*b*).

saturation of absorption [41]. At first, the interaction is characterized by linear absorption of the laser pulse in the medium, and this energy fluence is not enough to cause nonlinear absorption. When approaching the focus, a nonlinear interaction is observed that occurs during interaction with the leading edge of the beam, which is accompanied by saturation of the medium, as a result of which, with a sufficient nanosecond pulse duration exceeding 1 ns, the trailing edge of the beam is weakened more strongly. The trailing edge of the beam is already passing through a saturated medium and thus experiences nonlinear absorption. Near the focus, the fluence becomes high enough that the leading edge of the pulse experiences nonlinear absorption, resulting in a sharp drop in the peak fluence. This eventually begins to influence the pulse duration and in the case of pulses with a duration of 10 ns at a wavelength of 532 nm, a noticeable decrease in intensity to 7.5 ns. This effect has a lesser effect for pulse durations less than 7 ns at a wavelength of 405 nm, as a result of which no decrease in duration was recorded.

3.2. Findings

The obtained measurement data for bis-phthalocyanines **1,2** are grouped in a table. The thickness of the cuvette in all cases was 3 mm. The required attenuation of laser radiation by bis-phthalocyanines was obtained at wavelengths of 405 and 532 nm with pulse durations of 7 and 10 ns, respectively.

It is shown that the relationship between the σ_2 and σ_1 descriptors is nonlinear, and this allows for an 2*D*-functional analysis of the effectiveness of optical limiting in relation to aggregation and NLO response (Fig. 8, see the table). The label „VIS“ indicates studies in the visible wavelength range, and the label „UV“ is used to indicate the UV wavelength range. As follows from Fig 8, *a*, the dyes are grouped into conventional zones. Based on the boundary conditions for enhancing the efficiency of optical limiting: $\sigma_1 \to -\infty$, $\sigma_2 \to +\infty$ [12,40], it can be argued that zinc bis-phthalocyanine **1a** has maximum efficiency in optical limiting in the UV region. This fact is very important, since it clearly demonstrates the participation of the *d* shells of the complexing metal in the effect of reverse saturation of absorption, which is confirmed by the moderate result for the magnesium complex **1b**, in which the 3*d* level lies above the occupied 2*s* orbitals and is also free. The obtained result can also be justified by the well-known fact of the presence of metal $\leftrightarrow$ ligand electronic transitions, which manifest themselves precisely in the UV region [42]. The value of the descriptor σ_2 for the ligand **2** in the UV region tends to zero. This means a decrease in the effectiveness of limiting the properties of the dye in relation to NLO. Since this weakening occurs clearly faster than the negative effect of aggregation on limiting (descriptor σ_1), then the parametric function „bends“. From this we conclude that the NLO characteristics of the dyes take precedence over aggregation. From Fig. 8, *a* it also follows that dyes **1,2** in the visible region somewhat disappoint expectations, occupying a region above the conventional „golden“ middle ground. Figure 8, *b* shows the result of the CORRELATO [40] algorithm, which allowed not only to arrange all the dyes in a line, but also to formulate specific boundary conditions efficiency of optical limiting. Filtering conditions are the following expressions: $\theta_1 = \lg(\sigma_2) > 0$ and $\theta_2 = \lg(\sigma_1^2\sigma_2^2) > 0$. In this case, we are dealing with higher limiting performance, and it is higher the further the signals are shifted diagonally upward. Otherwise, the absorbers will be ineffective, and this may not satisfy the design requirements. Let us note once again that the optical limiting parameters are: β, F_{th}, k_{A} and DR are interconnected not only with each other, but also with the concentration of the dye (parameter α). Therefore, it is not allowed to operate with their absolute values. The method for assessing the efficiency of dyes in optical limiting, which we use here, allows to evaluate the contribution of the listed parameters in the aggregate and will further serve

Data for assessing the efficiency of optical limitation for bis-phthalocyanines **1,2**

Sample	λ, nm	α, cm^{-1}	β, $cm \cdot GW^{-1}$	F_{th}, J/cm^2	k_A	DR	σ_1, a.u.	σ_2, a.u.
2a	355 ± 1	2.7 ± 0.1	10 ± 1	1.00 ± 0.01	1.1 ± 0.1	43 ± 4	-1.86	0.25
1a	405 ± 1	3.2 ± 0.1	97 ± 5	0.02 ± 0.01	2.3 ± 0.1	880 ± 70	-3.00	12.64
1b	405 ± 1	2.3 ± 0.1	49 ± 4	0.21 ± 0.02	1.3 ± 0.1	84 ± 7	-2.41	3.61
2	532 ± 1	1.6 ± 0.1	23 ± 3	0.04 ± 0.01	1.5 ± 0.1	960 ± 80	-1.77	0.90
1a	532 ± 1	1.3 ± 0.1	56 ± 4	0.05 ± 0.01	2.0 ± 0.1	680 ± 50	-1.98	3.30
1b	532 ± 1	0.8 ± 0.1	24 ± 3	0.04 ± 0.01	1.4 ± 0.1	850 ± 70	-1.21	0.98

Note. [a] $F_s = 2.12\,J/cm^2$ for phthalocyanine **2**.

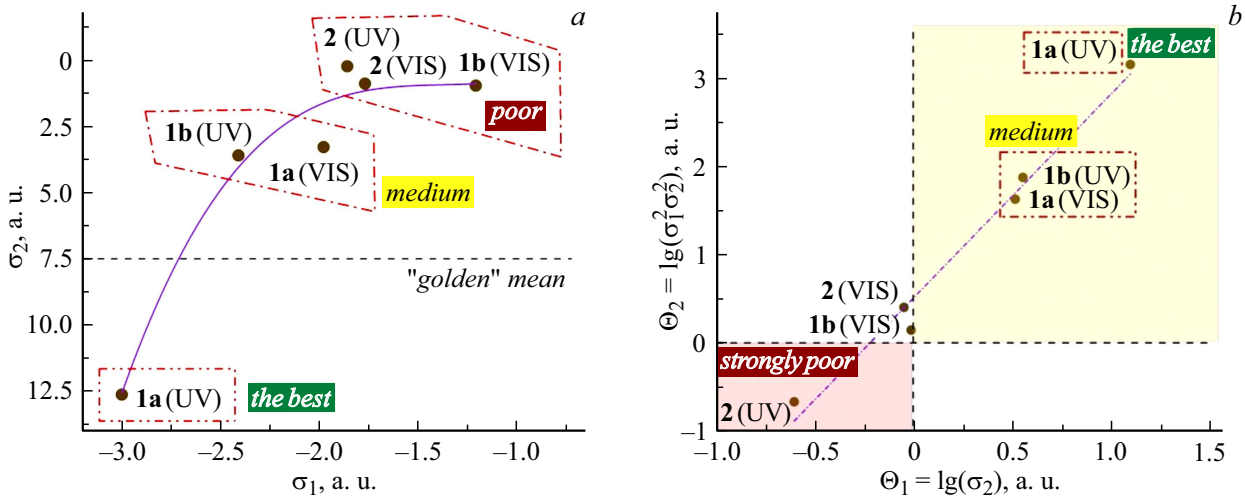

Figure 8. Functional (*a*) and band parametric (*b*) analysis of the efficiency of optical limiting based on the aggregation of dyes and their NLO response. Substitution of values into formulas (7), (8) was carried out in the dimensions indicated in the table. The dotted line shows the „golden“ mean: efficiency increases as you move from the top to the bottom of the graph.

as a generalized characteristic of NLO materials for optical limiters.

Conclusion

Using equations for functional and band parametric analysis of the efficiency of optical limiting based on dye aggregation and NLO response from the point of view of quantum mechanics, which we derived earlier in the works [12,40], it is shown that in terms of creating optical limiters for In the UV range, zinc bis-phthalocyanine **1a**, the macrocycles of which are linked by a cyclotriphosphazene spacer, should have maximum efficiency (Fig. 1). The nonlinear relationship of the descriptors responsible for the efficiency of limitation demonstrates the fact that the NLO properties of materials take precedence over aggregation. Based on the relationship between the descriptors σ_1 and σ_2, equations (8) and (9), respectively, strict boundary conditions for sorting dyes with respect to their efficiency in optical limiting are obtained. It is also shown that the synthesized dyes will work better in the UV range when comparing their total efficiency with the visible region of the spectrum.

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Conflict of interest

The authors declare that they have no conflict of interest.

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