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Synthesis and Investigation of New Organophosphorus Dyes of Green and Red Luminescence

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Abstract–On the basis of amino derivatives of *N*-phenylnaphthalimide, naphthoylenebenzimidazole, benzanthrone, and phenalenone new phosphorus-containing luminophores containing phosphonomethyl and phosphazo groups in their structures were synthesized. Luminiscence spectrum and excitation characteristics of compounds obtained were studied. Some of the synthesized substances were proposed for application as effective laser dyes for green and red spectral regions with a wide range of retuning.

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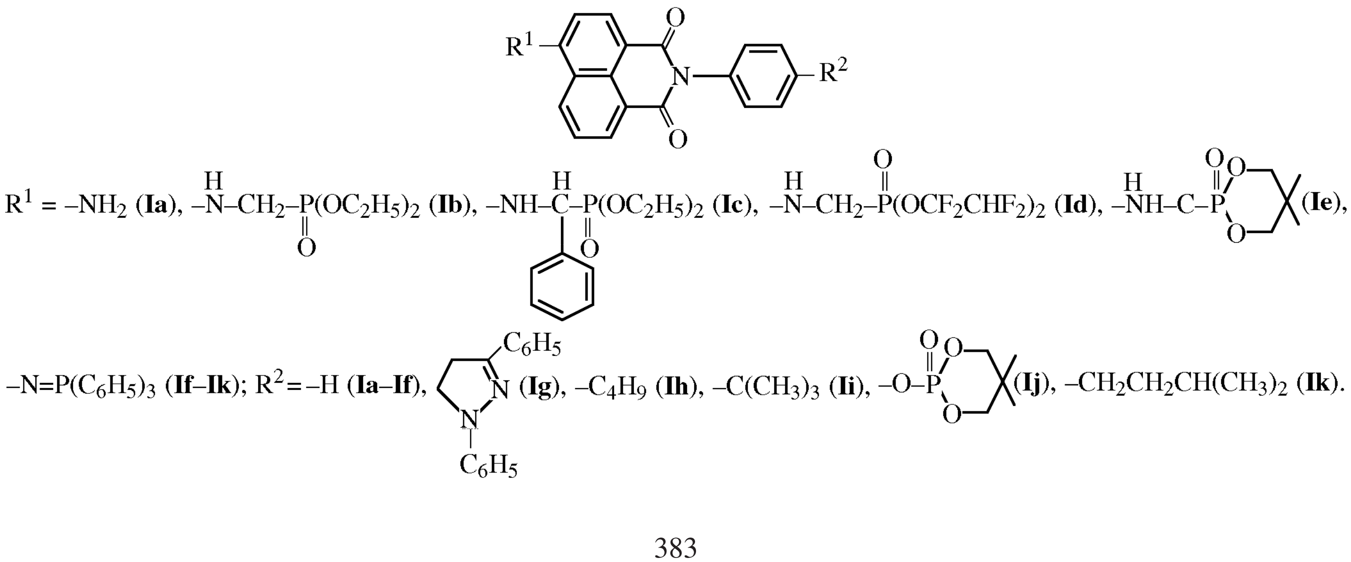
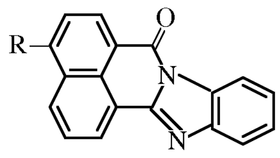
In continuation of previous studies in the field of the chemistry of phosphorus-containing organic dyes [1–5. we undertook a synthesis and study of spectral luminescence properties of new luminophores luminescing in green and red regions of spectrum and having characteristics required for practical application. The studied compounds possess a common structural feature: most of them are derivatives of *peri*-indenones or their aza-analogs.

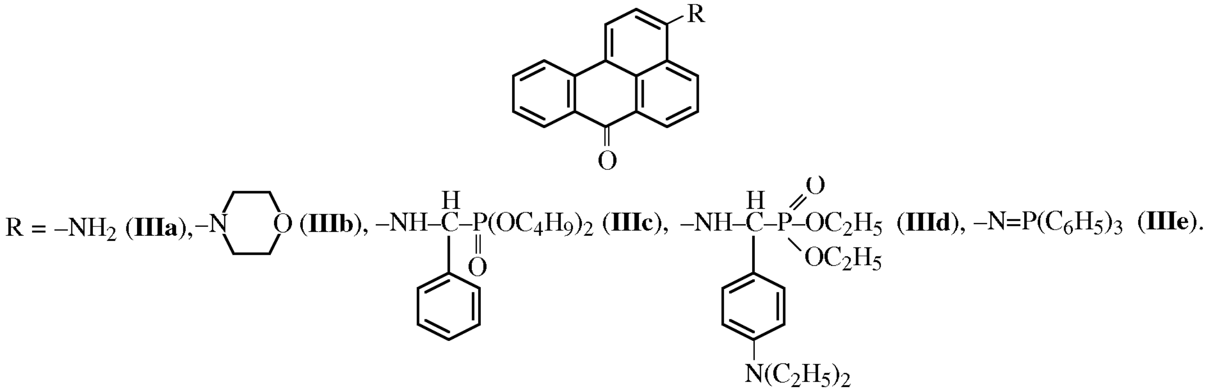
Some syntheses of phosphorylated *peri*naphthindenones were reported [6–11] (mainly, derivatives of phenalenone and benzanthrone), but the published investigations were not systematic. These publications practically lacked the data on spectral luminescence characteristics of compounds obtained. We regard as interesting patent publications on the synthesis of phosphorus-containing dyes undertaken in connection with the research of new fluorescent substrates for phosphatases in biochemical investigations [10] and development of effective antiviral preparations with diminished toxicity [11].

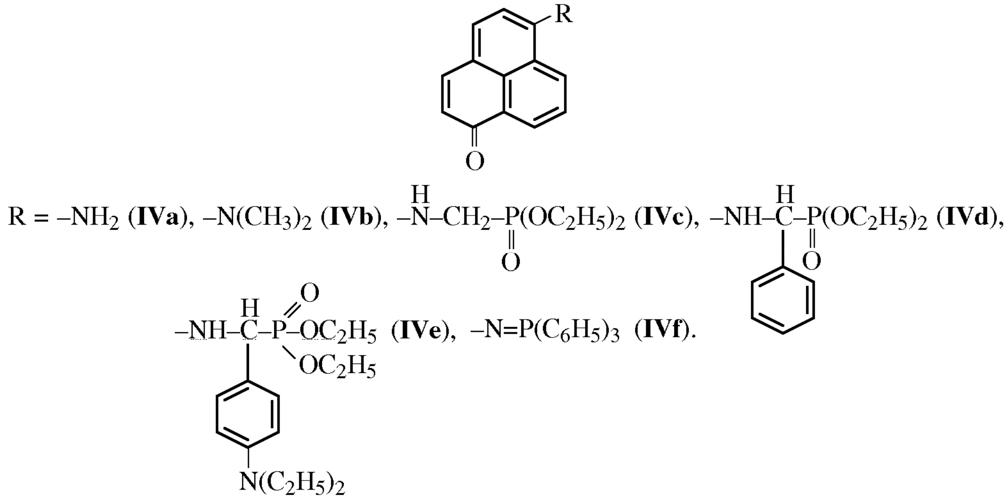
Here we describe a group of novel phosphorus-containing dyes. Structural formulas of the synthesized compounds are shown on the scheme below.



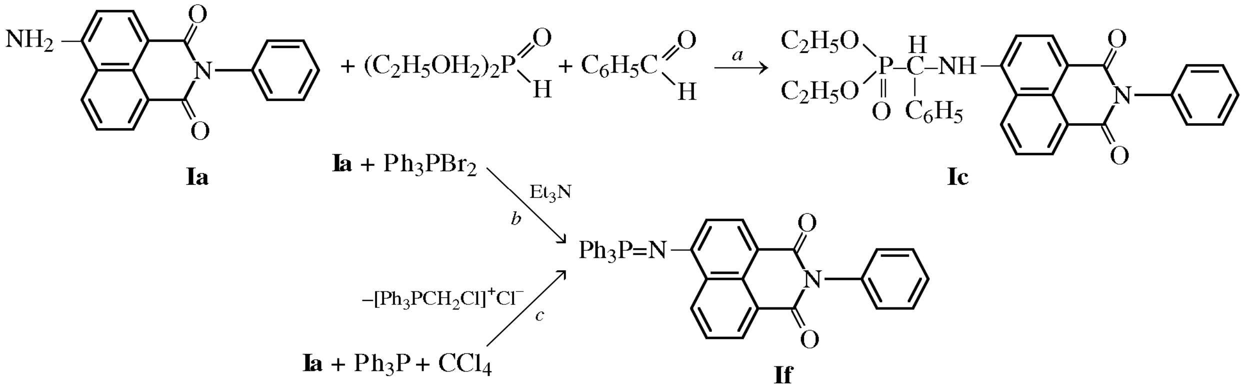


R = –NH$_2$ (**IIa**), –N=P(C$_6$H$_5$)$_3$ (**IIb**).





We used as basic compounds amino derivatives of *N*-phenylnaphthalimide (**I**), naphthaylenebenzimidazole (**II**), benzanthrone (**III**) and phenalenone (**IV**). For the phosphorylation we used the following reactions: (*a*) Kabacnik – Filds, (*b*) Kirsanov, in Horner and Edinger modification [12], and (*c*) Appel [13]. Various modes of phosphorylation are shown with amine **Ia** as an example.



Ia **Ic** **If**

Other compounds have been obtained along the same schemes under commonly applied conditions that have been adjusted for each group and are described in Experimental.

The physicochemical characteristics of the obtained dyes are compiled in Table 1. This table contains also the properties of several model benzanthrone and phenalenone amino derivatives that have been prepared from the corresponding bromo derivatives via nucleophilic substitution by excess of a secondary amine under the conditions close to those described in [14].

In the ^{31}P NMR spectra of the synthesized phoshonomethylated derivatives single signals existed in the region typical for dialkyl(α-aminoalkyl) phosphonates (21 to 23 ppm) [1]; signals of phosphazo compounds were registered in a region characteristic of triphenylphosphazoarenes (7 to 10 ppm) [15].

Certain features of the synthesis of each group compounds should be mentioned. While the Kabachnik–Filds reaction proceeds successfully with a sufficiently high yield for the dyes of all types at heating in dioxane or *o*-xylene, or with dialkylphosphite excess, the synthesis of compounds containing phosphazo group along the scheme either (*b*), or (*c*) was successful only at boiling solutions in chlorinated benzenes, probably due to lower basicity of amino groups in the initial luminophores. In the most cases the best results were obtained with 1,2,4-trichlorobenzene as solvent, satisfactory yields were achieved with 1,2-dichlorobenzene, 4-amino-*N*-phenylnaphthalimide (**Ia**) and 3-aminobenzanthrone (**IIIa**) were obtained with comparable yields using chlorobenzene. Good yields of dyes were registered when hexachloroethane was used instead of carbon tetrachloride.

While Horner–Ediger reaction has been used for preparation of various phosphazo compounds for relatively long period [16-18], the Appel reaction is less known despite its interesting recent applications [19-20]. Synthesis of dyes along the Appel scheme is smooth enough at short (few minutes) boiling of the reaction mixture in a preliminary heated oil bath till the disappearence of the initial amino compound (TLC monitoring). In all cases the ^{31}P NMR spectra revealed the formation of triphenylchloromethylphosphonium chloride (δ_P = –22.1 ppm), the side product in all systems containing three components: $Ph_3P–CCl_4$–nucleophile. The mechanism of its formation was considered in detail in [13, 20]. Note that the Appel reaction applied to the synthesis of the luminophores studied by us proceeded selectively and with higher yield compared with the use of dibromophosphorane, leading to formation of dyes containing phosphazo groups.

Purification of compounds to analytical purity was carried out using column chromatography on silica gel (eluent chloroform); the yields listed in Table 1 were obtained after chromatographic purification.

Reaction progress and purity of compounds obtained were monitored by TLC in several azeotropic eluting systems differing by polarity that we had earlier suggested [21] for separation and identification of organophosphorus compounds of various types. Note that in all the cases the synthesized dyes were featured by high chromatographic mobility and solubility in organic liquid and polymeric media as compared with the corresponding parent luminophores.

Various phosphorus-containing fragments introduced into molecules of dyes are differently manifested in electronic spectra of the synthesized compounds. In the spectra of dyes with aminophosphonate group the absorption and luminescence bands suffer a small blue sfuft as compared with the parent compounds induced apparently by the effect of phosphorylmethyl groups, moderate acceptors [22]. The $\lambda_{\text{absorb}}^{\text{max}}$ values of compounds **Ia, IIIa** and **IVa** in ethanol are 435, 526 and 534 nm respectively (cf. with the data in Table 1). However, intensity of luminiscence in all the cases was higher than in the parent compounds: the fluorescence quantum yield φ for **IIIa, IIIc**, and **IIId** equals 0.66 (565 nm), 0.90 (555 nm), and 0.92 (560 nm) respectively ($\lambda_{\text{absorb}}^{\text{max}}$ in parentheses). Increase in φ is likely to result from steric effects of bulky phosphorylmethyl groups restricting the rotation of amino groups and thus reducing the probability of nonradiation deactivation of the electronic excitation. The presence in the molecules of dyes of triphenylphosphazo groups, in all likelihood the strongest donor substituents, changes considerably electronic spectra of compounds: the absorption and luminescence bands undergo a strong red shift, and the Stocks shift increases significantly, up to 140 nm. For all the synthesized iminophosphoranes an intensity of luminescence considerably increased as compared with the parent luminophores, apparently due both to the steric effect of bulky triphenylphosphazene groups and the mobility of effective partial charges along the phosphazo bond because of the involvement of the nitrogen lone pair in its formation. Thus, we measured quantum yields of fluorescence for some phosphazo

Table 1. Physicochemical and spectral characteristics of synthesized organophosphorus dyes (numbers correspond to the scheme)

Comp. no.	mp, °C	Formula	Yield %	Elemental analysis								λ_{absorb}^{max}, nm			^{31}P NMR spectrum, δ_P ppm
				found, %				calculated, %				($\varepsilon \times 10^{-4}$)			
				C	H	N	P	C	H	N	P	toluene	ethanol	propylene carbonate	
Ib	242–243	$C_{23}H_{23}N_2O_5P$	72.4	63.21	5.35	6.22	6.84	63.01	5.29	6.39	7.06	411	450	424.5	23.06
Ic	258–259	$C_{23}H_{27}N_2O_5P$	76.3	67.25	5.11	5.27	5.73	67.70	5.29	5.44	6.02	410	418	414	21.45
Id	124–125	$C_{25}H_{19}N_2O_5P$	69.5	48.95	3.05	4.35	4.89	49.19	3.14	4.58	5.07	360	380	384	
Ie	162–163	$C_{24}H_{23}N_2O_5P$	74.8	63.78	5.02	6.11	6.53	64.00	5.15	6.22	6.88	376	432	381	
If	280–281	$C_{36}H_{25}N_2O_2P$	48.7	79.60	4.74	4.92	4.81	79.82	4.59	5.10	5.65	430 (1.84)	450 (2.02)	445 (2.64)	9.08
Ig	169–170	$C_{51}H_{37}N_2O_2P$	45.5	79.67	4.82	7.25	3.91	79.67	4.85	7.28	4.03	435 (2.46)	450 (1.80)	447 (2.24)	
Ih	262–263	$C_{40}H_{33}N_2O_2P$	64.2	78.65	5.51	4.51	5.03	79.45	5.50	4.63	5.13	432	451	442	10.33
Ii	295–296	$C_{40}H_{33}N_2O_2P$	76.2	78.89	5.45	4.56	5.01	79.45	5.50	4.63	5.13	430 (2.70)	455 (3.60)	445 (2.44)	
Ij	235–236	$C_{41}H_{34}N_2O_6P_2$	37.2	70.41	4.97	3.27	8.36	69.09	4.81	3.93	8.70	430 (2.56)	450 (2.20)	445 (2.40)	
Ik	258–259	$C_{41}H_{35}N_2O_2P$	48.2	79.10	5.41	3.10	4.95	79.59	5.70	3.20	5.00	431	450.5	448.5	
IIb	258–259	$C_{36}H_{24}N_3OP$	34.6	79.16	4.25	7.52	5.06	79.25	4.43	7.70	5.68	485 (2.07)	495 (2.44)	493 (2.12)	10.57
IIIb	215–217	$C_{21}H_{17}NO_2$	85	79.75	5.52	4.12	–	79.98	5.43	4.44	–	432	445 (3.82)	444	–
IIIc	141–142	$C_{32}H_{37}NO_4P$	38	73.40	6.42	2.52	5.73	73.22	6.55	2.60	5.86	469	497	495	21.30
IIId	161–162	$C_{32}H_{35}N_2O_4P$	31.5	70.63	6.40	5.25	5.83	70.83	6.50	5.16	5.71	468	496	482	27.32
IIIf	261–262	$C_{35}H_{24}NOP$	60	83.08	4.66	3.20	5.67	83.15	4.79	2.77	6.13	495	528	539	7.85
IVb	108–109	$C_{15}H_{13}NO$	68	80.17	5.36	6.12	–	80.69	5.87	6.27	–	463	497	502	–
IVc	87–88	$C_{18}H_{20}NO_4P$	69	61.80	5.78	3.95	8.70	62.60	5.84	4.06	8.97	473	527	524	21.43
IVd	142–143	$C_{24}H_{24}NO_4P$	58.9	68.11	5.68	3.25	7.10	68.40	5.74	3.32	7.35	468.5	500	497	20.71
IVe	164–165	$C_{24}H_{33}N_2O_4P$	28.9	64.15	7.47	6.02	6.50	64.85	7.48	6.30	6.97	470.3	498.5	496	20.16
IVf	149–150	$C_{31}H_{22}NOP$	58	81.16	4.72	2.97	6.52	81.74	4.87	3.08	6.80	495 (0.93)	555 (1.06)	513	

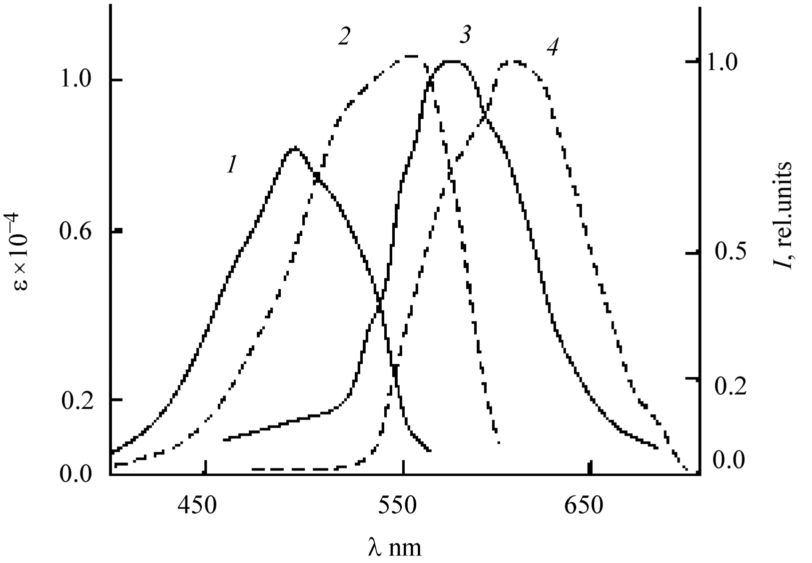


Fig. 1. Adsorption (*1*, *2*) and fluorescence spectra (*3*, *4*) of 3-phosphazophenalenone (**IVe**): (*1*, *3*) in toluene, (*2*, *4*) in ethanol.

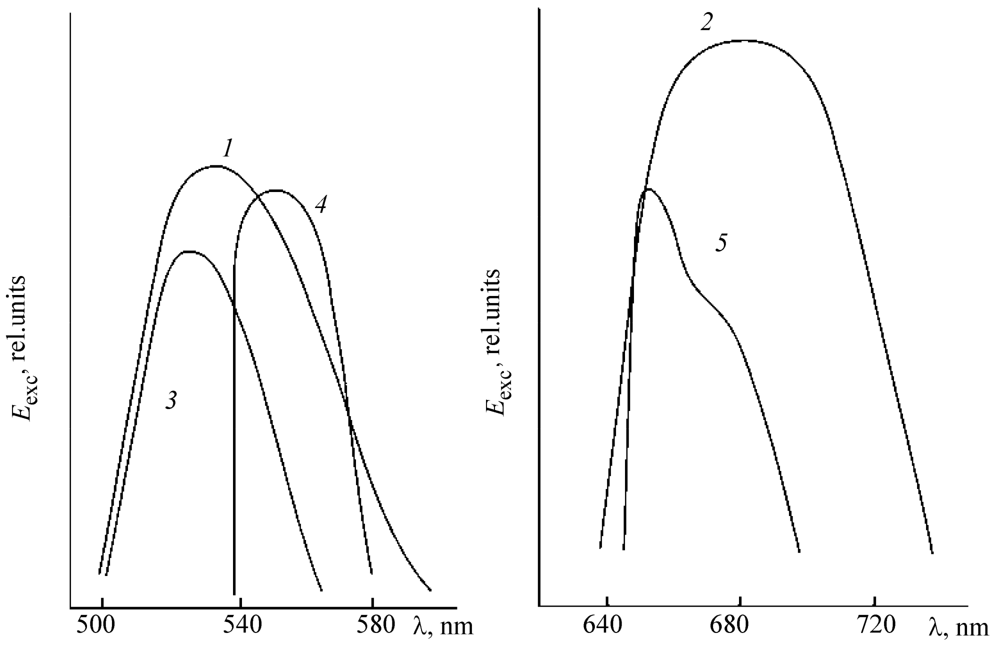


Fig. 2. Dependence of excitation energy of dye solutions in ethanol on the wavelength: (*1*) dye **Ic**; (*2*) dye **IIIe**; (*3*) coumarin 7; (*4*) uranine; (*5*) cresyl violet.

compounds (in parentheses are given the values of λ_{absorb}^{max} and solvents): 0.94 (541 nm, propylene carbonate), 0.75 (500 nm, toluene) (**If**); 0.78 (500 nm, toluene), 0.88 (530 nm, propylene carbonate) (**Ii**); 0.92 (570 nm, toluene), 0.82 (600 nm, propylene carbonate) (**IVf**).

Figure 2 shows absorption and fluorescence spectra of dye **IVf** in ethanol and toluene at 25°C ($c = 0.5 \times 10^{-4}$ mol l^{-1}).

For some of dyes with strong luminescence we studied the possibility of their application as com-

Table 2. Excitation properties of ethanol solutions of some of the synthesized compounds as compared with the known laser dyes for green and red spectral regions at the pumping with illumination of 3d harmonic of neodimium laser

Dye	λ_{exc}, nm	E_{pump}, mJ	Differential efficiency, %	E_{exc}, mJ (E_{pump}, 20 mJ)	Retuning region, nm
Coumarin 7	524	0.25±0.12	11.6	1.24	500–565
Uranine	557	0.25±0.06	13.6	2.58	540–580
Ia	542	1.2±0.6	11.2	1.61	
Ib	542	2.3±0.3	12.7	1.63	
Ic	530	0.2±0.02	22.0	3.9	500–600
Id	542	0.80±0.15	9.2	1.64	495–605
Cresyl violet	676	0.2±0.1	9.3	1.53	645–700
IIIa	679	4.8±0,5	10.4	0.89	
IIIc	640	4.0±0,6	22.0	1.70	640–740
IIIe	670	0.30±0.15	15.5	2.26	
IIIe[a]	668	0.13±0.05	28.0		

[a] Pumping by second harmonic of neodymium laser on phosphate glass (527 nm).

ponents of liquid media for dye lasers. Under identical conditions we measured principal excitation characteristics: the excitation wave length in a broadband resonator, pumping energy, energy of excitation at various pumping energies, and we determined differential performance near the threshold. For comparison of the studied compounds with the existing laser dyes we measured under the same conditions the energy of excitation for ethanol solutions of several widely used substances regarded as the most effective [23]. For the most effective compounds we measured retuning curves, namely, the dependence of excitation energy on the wave length at the use of selective resonator. The results are shown in Fig. 3. It is seen that the regions of excitation wave lengths of dyes **Ic** and **IIIc** considerably exceed the areas of retuning of other dyes in the related spectral range. These compounds also are no worse in efficiency than the reference substances.

Table 2 comprises the excitation characteristics of the studied dyes. As seen, among the naphthalimides, compound **Ib** shows excitation activity on the level of the reference compounds in the green region of spectrum, and **Ic** is notably better than both coumarine 7 and uranin. The efficiency of excitation for this compound is above 20% in a wide range of pumping energy and falls insignificantly in the region of extreme pumping. Optimization of the dye concentration and parameters of resonator, and use of pumping with more appropriate spectrum probably may increase its excitation efficiency. It is of great value that the excitation wave length of this compound (530 nm) falls into the most "unfavorable" spectral region. It can also be noted that the naphthalylimides studied by us can be easily synthesized from the known parent dye **Ia** providing an opportunity of their preparation in large amounts at low cost.

Thus, the studied compounds by their fundamental characteristics are better than the dyes widely applied in green and red spectral regions and thus they are promising for extensive application as active laser media.

Among the studied benzanthrones with excitation in the red spectral region a special attention should be given to dye possessing triphenylphosphazenyl substituent $N=P(C_6H_5)_3$.This compound by excita-

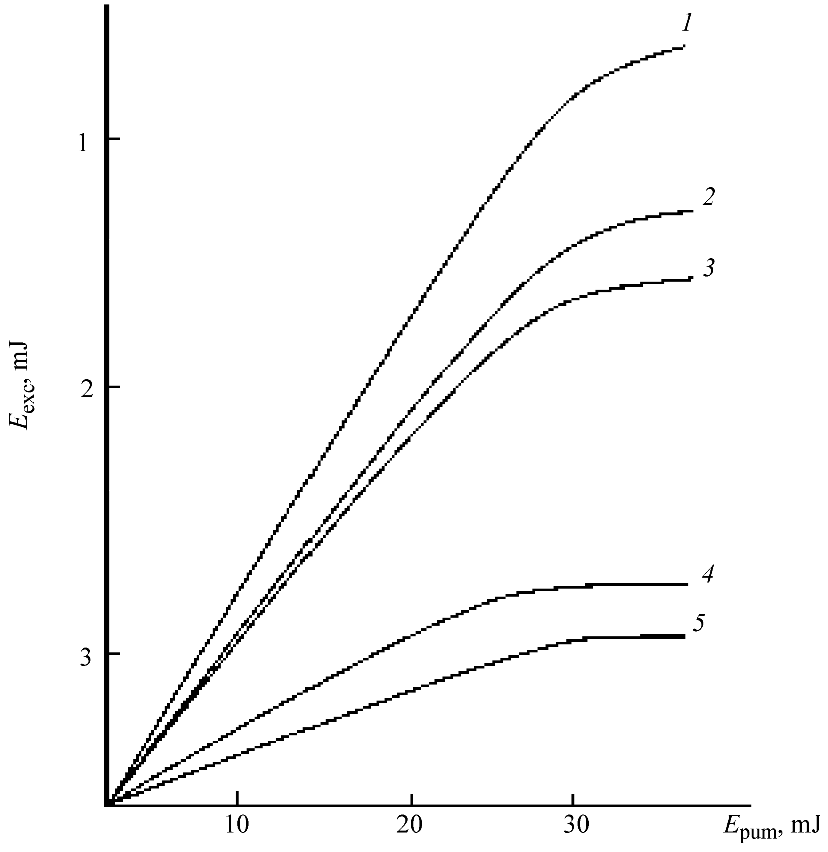


Fig. 3. Dependence of excitation energy of dye solutions in ethanol on the pumping energy: (*1*) dye **Ic**; (*2*) uranine; (*3*) dye **IIIe**; (*4*) cresyl violet; (*5*) coumarin 7.

tion energy and differential efficiency is better than cresyl violet commonly used шт this region (λ_{exc}= 670 nm): at equal pumping the excitation energy of **IIIe** is 1.5 times higher (see Fig. 3).

An important characteristics of the dyes is their photostability, therefore we carried out comparative measurement of the rate of photo-damage of the best dyes at illumination with mercury lamp. The results of measuring фку listed below.

Dye	Photodecomposition period, h
Ia	2.3
Ic	4.9
If	5.2
IIIa	4.8
IIIc	7.6
IIIe	10.7
Coumarin 7	5.1
Uranine	14
Cresyl violet	0.2

It was showт that photostability of the phosphorus-containing dyes is higher as compared with the parent compounds. Dye **Ic**, the most effective in the green region, by its photostability is no worse than coumarin, althouth it is inferior to uranin 7. In the red region dye **IIIe** is much more stable than cresyl violet.

Thus, we synthesized new phosphorus-containing organic dyes based on the available luminophores; their spectroluminiscence and excitation characteristics were measured. It is shown that the synthesized compounds are effective luminescent substances. Some of these newly synthesized compounds by their excitation characteristics exceed the laser dyes commonly used in the green and red spectral regions, and can be recommended as the components of active laser media.

EXPERIMENTAL

The ^{31}P NMR spectra were taken from solutions in chloroform on a Bruker WP-80SY instrument, operating frequency 32.4 MMHz, reference compound 85% H_3PO_4. The UV spectra were measured on a Specord UV-VIS spectrophotometer, the luminescence spectra were registered on an installation assembled on the basis of MDR-3 monochromator. Excitation characteristics were measured at pumping with third harmonics of Nd-laser (351 nm), transverse pumping. In some cases we used pumping with second harmonics of Nd-laser. The excitation threshold and differential efficiency were measured using a short nonselective resonator 3 cm in length. For the most effective dyes retuning curves were measured, and the range of wavelength retuning was determined. The retuning was carried out using selective prisms placed inside the resonator. The rate of photo-damage of the ethanol solutions of dyes was measured at illumination with mercury lamp DRT-220 at the distance of 10 cm. The solutions were placed into the rectangular cells of fused glass 1 cm thick, initial optical density in the maximum of adsorption spectrum was taken at equal to 0.3. The illumination was interrupted intermittently, and optical density of solutions was measured. As the period of photo-damage was taken the time of 2.71-fold decrease in optical density at the maximum of adsorption.

Thin layer chromatography was carried out with Silufol UV-366 plates from Cavalier (Czech republic) by ascending method in the following systems: chloroform–ethanol (10:1) (**I**), chloroform– acetone (331:169) (**II**), benzene–dioxane (88:12) (**III**) under conditions like those described in [21].

All syntheses were carried out under nitrogen atmosphere, with anhydrous solvents. The parent dyes **Ia, IIIa, IVa** were synthesized along the known procedures [24], compound **IIa** by the method in [25].

***N*-Phenyl-4-[α-(diethoxyphosphoryl)benzylamino]naphthalimide (Ic).** A mixture of 1 g of 4-amino-*N*-phenylnaphthalimide and 0.37 g of freshly distilled benzaldehyde in 20 ml of diethylphosphite was heated for 2 h at 140°C. The reaction mixture was cooled to room temperature and treated with 50 ml of 10% aqueous sodium hydroxide for removing phosphite excess. The precipitate separated was filtered off and dried. Yield of **Ic** 3.1 g (86.3%). Analitically pure sample was obtained after chromatography on a column with silica gel L 100/160 (eluent chloroform), R_f 0.72 (**I**), 0.39 (**II**), 0.64 (**III**). Compounds **Ib, IIIc** and **IVc-IVe** were prepared similarly. Compounds **Id** and **Ie** were synthesized with equimolar amount of the corresponding phosphite in *o*-xylene at 3 h boiling.

3-(Triphenylphosphazo)benzanthrone (IIIe). *a*. A mixture of 0.61 g of **IIIa**, 0.66 g of triphenylphosphine and 0.38 g of carbon tetrachloride in 30 ml of *o*-dichlorobenzene was refluxed for 10 min in a preheated oil bath to disappearance of the parent amine (TLC monitoring). The mixture was filtered and after chromatography of the filtrate on a column with L 40/100 silica gel (eluent chloroform) 0.48 g of **IIIe** was obtained as dark-cherry crystals. R_f 0.48 (I), 0.72 (II), 0.44 (III). Phosphazo compounds **Ie-Ik, IIb** and **IVf** were obtained similarly.

b. To a solution of 1.11 g of triphenyldibromophosphorane [12] in 30 ml of chlorobenzene was added at stirring 0.61 g of **IIIa** and 0.5 g of triethylamine, the mixture was refluxed for 7 h, filtered, and the filtrate was passed through a column with silica gel L 40/100 (eluent chloroform). Yield 59%; the product was identical to that obtained by method *a* by its adsorpton spectra, R_f, and mp of a mixed sample.

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