in its transparency. This process results in a decrease in the efficiency of small-diameter lamps and an increase in their heat losses (Fig. 1).

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FLUORESCENCE OF VAPORS OF COMPLEX MOLECULES IN THE ULTRAVIOLET REGION OF THE SPECTRUM

L. A. Barkova, V. V. Gruzinskii, P. I. Petrovich, and E. Yu. Shishkina

Intensely fluorescent, stable, polyatomic organic compounds having a high absorption coefficient at the maximum of the long-wave band ε_{max} (or a high probability of a radiative transition A) have not been found previously for the shortwave region of the spectrum. According to various sources p-terphenyl with $\lambda_{max}^{f1} = 340$ nm [1] has a quantum fluorescence yield of $\gamma = 0.2$ [2] and 1.0 [3]. In the vapor the shortest-wave generation of radiation has been obtained for quaterphenyl vapor having $\lambda_{max}^{gen} = 350$ nm [4]. The reasons for the absence of generation in para-terphenyl vapor are discussed in [5].

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In this investigation we studied the spectral properties of a new class of organic compound, viz., derivatives of monobenzoxazole. The dibenzoxazoles have been studied previously [6, 7].

Benzoxazole has a comparatively high value of $\varepsilon_{max} = 3600$ liter·mole⁻¹·cm⁻¹ (viz., the strongly fluorescent derivatives of phthalimide) but it scarcely fluoresces at all in the gas phase, owing to the appreciable value of the intercombination conversion constants k_{ST}. The excited electronic state $S_{1\pi\pi\star}^*$ is lower (the $\tilde{X}^1A' \rightarrow \tilde{A}^1A'$ ($\pi\pi^*$) transition with a 0-0 band at $\lambda = 274$ nm [8]).

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Extension of the chain conjugate bonds led to an intense fluorescence of the vapors of dibenzoxazoles [7]. According to experimental and theoretical data [7, 9] phenyl derivatives of benzoxazole also have a system of low electronic states $T_{\pi\pi\star}$, $S_{\pi\pi\star}^{\star}$, $T_{n\pi\star}$, $S_{n\pi}^{\star}$ (group 5 according to the system in [10]), caused by the more rapid decrease in the energy of the $S_{\pi\pi\star}^{\star}$ state with respect to $T_{n\pi\star}$ with the extension in the chain of conjugate bonds. This promotes their intense fluorescence. The above considerations and preliminary experiments [11] enable us to suggest that there is a large group of benzoxazole derivatives which may fluoresce in the gas phase



(1)

where R₁, R₂, R₃, and R₄ are H, Cl, F, cycloalkyl, alkenyl, alkoxyl, arylalkyl, alkaryl, aryl, NH₂ (or possibly the replacement of hydrogen by an alkyl or acyl group), OH, alkoxycarbonyl, heteryl, etc., and A is H, alkyl, cycloalkyl, alkenyl, aralkyl, arethenyl, arbutenyl, alkaryl, aryl, heteryl.

Substituted or unsubstituted phenyl, biphenyl, naphthyl, anthryl, etc. may be used as the aromatic radicals and the heterocyclic radicals may be furyl, thienyl, thiazolyl, oxazolyl, oxadiazolyl, imidazolyl, triazolyl, coumarinyl and other nitrogen-, oxygen-, and sulfur-containing heteryls.

For comparison we studied the vapors of compounds having different positions of the heterocyclic and phenyl rings:



2,5-diphenyloxazole (PPO),



1,4-di[2-(5-phenyloxazolyl)]benzene (POPOP),



1,4-di[benzoxazole-2'-y1]benzene (BoPBo),

2-phenylbenzoxazole (BoP), 2-phenyl-5-methylbenzoxazole (5-methyl-BoP), and 2-phenyl-5-tertbutylbenzoxazole (5-tert-butyl-BoP).

BoP, 5-methyl-BoP, and 5-tert-butyl-BoP were obtained under the preparation conditions given in [12] by condensing benzoic acid with 2-aminophenol, 3-amino-4-hydroxytoluene, and 2-amino-4-tert-butylphenol in the presence of boric acid and in an atmosphere of nitrogen. The benzoxazoles were purified by repeated recrystallization from ethanol and vacuum distillation.

Figure 1 shows the absorption and fluorescence spectral of the vapors and solutions of the phenyl derivatives of benzoxazole. Compared with the solutions in cyclohexane the maxima of the spectra of the vapors for BoP are displaced in the shortwave direction by 1300 and 1650 cm⁻¹, respectively, i.e., by approximately 1000 cm⁻¹ less than for BoPBo [6] (for comparison, the 0,0 band of the absorption spectrum of benzoxazole is displaced by 400 cm⁻¹). In the gas phase virtually total coincidence of the 0,0 bands is observed in the absorption and fluorescence spectra, while in solution the difference is 200 cm⁻¹. Common to the mono-



Fig. 1. Absorption spectra (1, 1') and fluorescence spectra (λ_B = 296.7 nm) (2, 2') of vapors and solutions of phenylbenzoxazoles: a) BoP, vapor at T = 360°K (1, 2), solution in cyclohexane (1', 2'); b) BoP, T = 430°K (1) and 565°K (1'), 5-methyl-BoP (2), 5-tert-butyl-BoP (2'), T = 373°K. The arrows indicate the pumping wavelength in nm.

benzoxazoles and dibenzoxazoles is only a slight diffuseness of the vibrational structure of the vapors compared with the solutions (Fig. 1a), this being large for, e.g., the arylderivatives of oxazole [6]. With an increase in temperature the absorption spectrum is diffused appreciably (Fig. 1a, spectrum 1 and Fig. 1b, spectra 1 and 1'). The presence of the CH₃ substituent in the 5 position of the benzoxazole ring leads to a slight shift in the spectra in the long-wave direction (Fig. 1b, spectrum 2) and the fluorescence intensity $W_{\nu,qu}$ corresponds approximately to that for BoP. Replacing CH₃ by C(CH₃)₃ does not lead to a marked shift of the spectra but causes an appreciable decrease in $W_{\nu,qu}$ of the vapors. For example, in spite of the low temperature $T_m = 357^{\circ}K$ the fluorescence spectra 2 (Fig. 1b) are obtained with an appreciably higher temperature of the lower branch of the cuvette containing a substance which gives a vapor pressure ($T_n = 380^{\circ}K$) than for the vapor of BoP ($T_n = 350^{\circ}K$), although ε is increased somewhat by λ_B owing to the shift in the absorption spectrum in the long-wave direction; in this case $W_{\nu,qu}$ is lower by a factor ≈ 5 for identical parameters of the recording system.

The low evaporation temperature of the phenylbenzoxazoles is noteworthy. This is particularly important for their use in a discharge, the intrinsic temperature of which is higher than the experimental temperature. For the compounds used previously, which evaporate at high temperature, the stability of the intramolecular bonds is close to the maximum for high excitation powers [1].

With an increase in vapor pressure p the optical density D increases rapidly for BoP (at a rate $\Delta D/\Delta T \sim 0.4 \text{ deg}^{-1}$), while in the region of the melting point ($T_m = 377.6-378.2^{\circ}$ K) it decreases sharply ($\Delta D/\Delta T \approx 0.007 \text{ deg}^{-1}$). For comparison, $T_m = 350-352^{\circ}$ K for BoPBo and and higher p values are achieved up to T_m with a monotonic increase in D. The absorption coefficient of the vapor of BoP measured for complete evaporation of a definite amount of



Fig. 2. Dependence of the fluorescence spectra of the vapors of BoP (a-c), POPOP (d, f), BoPBo (e) on the wavelength of the stimulating radiation, temperature, and vapor pressure: I) $\lambda_B = 334$ (1), 313 (2), 302 (3), 296.7 (4), and 289.3 (5), T = 380°K and $T_n = 362°K$ (a), T = 460°K and $T_n = 443°K$ (d); II) T = 385 (1), 470 (2), 480 (3), and 580°K (4), $\lambda_B = 302$ (b) and 313 nm (e), $T_n = 345$ (b), and 450°K (e); III) $T_n = 345$ (1) and 415°K (2), T = 435°K, $\lambda_B = 302$ nm (c); $T_n = 450$ (1) and 540°K (2), T = 605°K, $\lambda_B = 313$ nm (f).

Fig. 3. Variation in the fluorescence spectra of vapors of POPOP (a) and BoPBo (b) under the action of laser radiation. a) $\lambda_B = 334$ (1) and 337 nm (2-4, 4'); T = 430 (2), 460 (1, 3), and 510°K (4, 4'); p = 2.3 \cdot 10^{-4} (2), 4.4 $\cdot 10^{-3}$ (1,3), and 2 $\cdot 10^{-2}$ mm Hg (4,4'); b) $\lambda_B = 337$ nm; T = 470 (1) and 573°K (2, 3, 3'); p = 2.3 $\cdot 10^{-4}$ (2), 1.2 $\cdot 10^{-3}$ (1), and 10⁻² mm Hg (3, 3'); the intensity of the excitation radiation is lowered ten-fold (3', 4').

substance in the cuvette at the maximum of the long-wave band $\varepsilon_{max} = 27,000$ liters·mole⁻¹·cm⁻¹ and is close to $\varepsilon_{max} = 28,480$ liters·mole⁻¹ in cyclohexane, which indicates that the configuration of the molecule is preserved in the gas phase. The natural lifetime configuration of the molecule is preserved in the gas phase. The natural lifetime of the BoP molecules in the excited state, calculated from the absorption spectrum is $\tau_0 \approx 1.8$ nsec, and A = 5.5·10⁸ sec⁻¹.

The quantum yield of the vapors of the phenylbenzoxazoles is fairly high. Thus, for BoP $\gamma = 0.73$ (T = 380°K, $\lambda_B = 302$ nm) measured with respect to $\gamma = 0.78$ in cyclohexane [13]. From γ and τ_0 , $\tau \approx 1.3$ nsec. Estimated from these data, assuming nonradiative conversion only through the triplet state, we have $k_{\rm ST} = 2 \cdot 10^8 \ {\rm sec^{-1}}$.

The fluorescence spectra of the phenylbenzoxazoles depend on the reserve of vibrational energy communicated to the molecule by an optical (λ_B) or a thermal (T) path (Fig. 2a, b). An increase in T, like a decrease in λ_B , causes diffusion of the vibrational structure. The introduction of substituents leads to a shift in the spectra in the long-wave direction (Fig. 1).

The effects of λ_B , T, and p on the fluorescence spectra of BoP, BoPBo, and POPOP are compared in Fig. 2. With a change in p by more than two orders of magnitude the fluorescence spectra of BoPBo and POPOP are unchanged, except for reabsorption on the long-wave wing, over the limits $T_n = 420-485$ °K and 440-495°K, respectively. However, the spectra of BoP are unaltered over the range T = 345-385°K, while an increase in T_n to 415°K (Fig. 2c) causes (in the region $T_n > T_m$) a growth of the long-wave wing in the spectrum and a sharp decrease in the fluorescence intensity ($\lambda_{obs} = \lambda_{max}^{f1}$). In the region of λ_{max}^{f1} , ϵ is small for BoP (Fig. 1), while for POPOP (Fig. 2f) the spectrum is unchanged even with appreciable reabsorption of the long-wave wing. We observed a similar variation in the fluorescence spectrum with an increase in $T_n > T_m$ for the vapor of PPO, for which the formation of fluorescent excimers takes place in solutions at high concentrations with the interaction of excited and unexcited molecules [14] as well as a decrease in the total luminescence intensity [15]. In the absorption spectra of BoP and PPO additional absorption bands are not observed with a change in T_n within the limits 350-425 and 373-415°K, respectively. It can be supposed that excimers are also formed both in BoP and PPO vapors with an increase in p.

It has been found that the intensity of the long-wave wing of the fluorescence spectra of BoPBo and POPOP increases when the vapors are excited by laser radiation $\lambda_B = 337$ nm compared with the close $\lambda_B = 334$ nm of a mercury lamp [16]. The broadening of the spectrum depends on the power of the excitation radiation, temperature, and vapor pressure. An LGI-21 nitrogen laser created a radiation power density $U_B = 43$ kW/cm² (a single rotating mirror was also used to increase U_B). Such an excitation power enabled the fluorescence spectra to be measured for p an order of magnitude lower than the usual pressures and for low temperatures. The deformation of the spectral increases with an increase in U_B (Fig. 3a, curves 4, 4' and Fig. 3b, curves 3, 3'). For POPOP vapor the changes in the fluorescence spectrum are more substantial than for BoPBo vapor. In contrast to the first compound, they appear appreciable for the second only at T > 470°K. For laser excitation the fluorescence intensity of POPOP was decreased after 30 min by approximately 10%, while for BoPBo it remained constant for a given U_B .

The appreciable changes in the fluorescence spectra of the compounds POPOP and BoPBo on laser excitation may arise from their being superheated on multiple absorption, decomposition, changes in the configuration of the molecules in the excited state, the action of powerful radiation on the molecule, etc.; the probability of induced radiation B is only an order of magnitude lower than A for such an excitation power. In [17, 18] a cleavage of the intramolecular hydrogen bond in the 1-aminoanthraquinone molecule was observed even under the action of the radiation from a mercury lamp under steady-state conditions. Instead of the luminescence spectrum of 1-aminoanthraquinone, this led to the appearance of a spectrum displaced into the shortwave region and similar to the spectrum of anthraquinone. Here the excess of vibrational energy in the excited state above the equilibrium reserve necessary for the cleavage of the weak hydrogen by an optical means was $E^* = 6000$ cm⁻¹, which corresponds to the excess of temperature above the experimental temperature $\Delta T = 245$ °C for a value of the vibrational heat capacity $C_{vib} = 24.6 \text{ cm}^{-1} \cdot \text{deg}^{-1}$. On laser excitation these values are low for POPOP and equal to E* = 1150 cm⁻¹ and $\Delta T = 25^{\circ}$ C, which is appreciably lower than the highest we used in the excitation of vapors by the radiation from a mercury lamp: $\lambda_{\rm B}$ = 296.7 nm, E* = 5500 cm⁻¹, ΔT = 124°C (Fig. 2d, curve 4) or λ_B = 334 nm, T = 773°K, where such changes are not observed in the fluorescence spectra. After prolonged irradiation appreciable amounts of decomposition products are not detected in the cuvette and the fluorescence spectrum of solutions of the irradiated substance is identical to that of the unirradiated substance. The fluorescence intensity of repeatedly irradiated (after the dark period) POPOP vapor was restored completely. Excitation by radiation from a mercury lamp after irradiation led to the normal fluorescence spectrum. Furthermore, in a discharge, where as a result of the interaction between molecules and a wide spectrum of electrons of various energies an intense decomposition of the substance takes place, the fluorescence spectrum of the principal substance is observed. It should be noted that the position of the vibrational bands in the fluorescence spectrum coincides for the different methods of excitation, only their intensity being altered. The facts available at present indicate that the changes observed in the fluorescence spectra of POPOP and BoPBo may apparently be associated with the action of powerful excitation on the molecule. This is of undoubted theoretical and practical interest for a special investigation.

In contrast to condensed aromatic compounds [19], γ decreases appreciably with an increase in T for BoP. Figure 4 shows the dependence of the fluorescence intensity $W_{qu}^{rel}(T)$ of several compounds. The observation of the change W_{qu} was carried out in the band maxima which is scarcely shifted at all with a change in T (Fig. 2). Since in the Stokes region of excitation the absorption coefficient ε usually decreases only slightly with an increase in T [19], to a first approximation it may be considered that the $W_{qu}^{rel}(T)$ relationships pre-





sented in the Stokes region correspond to the $\gamma^{\text{rel}}(T)$ relationships in [19]. This is seen for POPOP vapor (curve 8 in Fig. 4 [20]). As was to be expected, with an increase in the total reserve of vibrational energy E* (a decrease in λ_B) in the excited state the rate of decrease in W^{rel}(T) increases. The introduction of stabilizing pentane vapor (p = 460 mm Hg) into the cuvette leads to a slight decrease in the drop in W_{f1}(T). Since for the compounds cited there is an equivalence between the optical and thermal methods of varying the vibrational energy, established from the change in the fluorescence spectra with λ_B and T, as for dibenzoxazoles [6] (in Fig. 2a and b the spectra 5 and 3 respectively are close for BoP) then an appreciable change in $\gamma(\lambda_B)$ can also be expected. The $\gamma(T)$ obtained over a small range for POPOP (Fig. 3, curves 7-7" [21]) are in good agreement with our relationships and the relative changes in $\gamma(\lambda_B)$ agree with the conclusion presented above. Taking into account the good agreement between the vibrational heat capacities, as determined from spectral data and by calculation [22], the 'C_{vib}(T) relationship have been calculated (Fig. 4b).

The data obtained indicate the potential possibility of generating radiation for compounds of formula (1) in the gas phase with optical and nonoptical pumping. In solutions the generation of these compounds is effective [11]. The absorption cross section at the maximum of the long-wave band for BoP $\sigma_{13} = 1.04 \cdot 10^{-16}$ cm², the cross section of induced emission is fairly high, $\sigma_{31} = 0.95 \cdot 10^{-16}$ cm². For a pumping power density P = 1.5 MW/cm², kabs = 10 cm⁻¹ an estimate of the amplication coefficient for BoP vapor by the relationship presented in [23] gives the values $k_{amp} \approx 1 \text{ cm}^{-1}$. According to the calculations in [9] the absorption cross section in the system of singlet excited states σ_{3n} is small in the generation region. For the pure vapors, pumping by radiation from well-known laser lines is limited by the strong $\gamma(T)$ relationship. For example, taking account of the vibrational heat capacity (Fig. 4c) and using $\lambda_{\rm R} = 266$ nm of the fourth harmonic of a neodymium laser, the superheating of molecules after one absorption-emission cycle is 330°C; for less powerful pumping from an excimer laser having λ = 281.8 nm, which lies approximately at the maximum of the absorption band (Fig. 1), the superheating is also high, 205°C. The most suitable is $\lambda_B = 308$ nm from an XeCl* excimer laser. This value of λ_B lies on the long-wave wing of the absorption band. Under such conditions, where λ_B occurred in the region of the 0,0 band, solutions of, e.g., PPO generated effectively [24]. In the case of λ_B = 308 nm the superheating temperature is already ~40°C, while according to Fig. 4a γ decreases to 0.7 γ_{max} . Therefore, it is also desirable to use stabilizing gases.

Phenylbenzoxazoles, like the other compounds studied, fluoresce intensely on excitation in an electric discharge, in which T is higher than T_{exp} [1], and for compounds having a high evaporation temperature the rate of thermal decomposition of the substance is increased. Owing to the low evaporation temperature and the high value of γ , they are promising for the search for generation from excitation by electron impact.

Thus a new class of organic compounds (phenylbenzoxazoles) has been found which fluoresce efficiently in the ultraviolet region of the spectrum and which may provide very shortwave generation radiation (λ_{max}^{f1} = 315 nm for BoP).

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