Spectroscopic and Laser Studies of Mixed Rhodamine 6G and 10-Phenyl-9-acetoxyanthracene Solutions

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The spectroscopic and laser parameters of a mixed donor (10-phenyl-9-acetoxyanthracene derivations)–acceptor (rhodamine 6G) solutions have been determined. The studies performed show that rhodamine 6G is responsible for the lasting of the mixed solution. The 10-phenyl-9-acetoxyanthracene added to the R6G lasing solution decreases the spectral range of lasing and the gain value, $G(\lambda)$, of the solution. These changes in the lasing parameters are caused by electron energy transfer processes of different types. The results of fluorescence studies of the mixed donor–acceptor solutions point out that the long-range dipole–dipole interaction mechanism of the static quenching accompanied by the formation of nonemitting complexes in the dye mixture is responsible for the noted lasing and fluorescence changes.

INTRODUCTION

Electronic energy transfer in a dye mixture is established as an additional effective mechanism to extend the wavelength of lasing and tunability in an energy transfer dye laser (ETDL) [1–3]. For such dye lasers an enhancement or decrease in the laser energy output is noticed [4–6]. An ETDL with wide tunability has been achieved using two-, three-, and four-component dye mixtures [5,7–9]. The most frequently used donor–acceptor pairs in the ETDL are: cresyl violet–rhodamine 6G, rhodamine B–DODC, coumarin–acriflavine, different coumarin dyes with some aromatic compounds [5–11].

Transfer of the electronic excitation energy from the donor molecule (D) to an acceptor molecule (A) generally occurs in one of the following processes: (a) radiative transfer, (b) energy resonance transfer, (c) collisional energy transfer, or (d) molecular complexation [12–15]. The energy transfer mechanism appearing in a given dye mixture depends on the fluorescence characteristics of the donor and acceptor and, in many cases, is influenced by the method of excitation. In the liquid media of ETDLs pumped by nitrogen lasers the dominating mechanisms are radiative transfer (for dilute solutions) and resonance transfer (for concentrated solutions) [16–18].

During the last few years the lasing potentials of 10-phenyl-9-acetoxyanthracene derivatives have been investigated in our laboratory [19–22]. Many of them show lasing in benzene, dioxin, and toluene solvents. It was shown that the lasing gain, $G(\lambda,t)$, threshold, and lasing spectral region depend mainly on the number and kind of functional groups substituted in the anthracene ring. Additionally, the lasing spectral region has been determined with the position of the $T_1 \rightarrow T_n$ spectrum.

In this paper we report the results of electronic energy transfer studies from some of the 10-phenyl-9-acetoxyanthracene derivatives (donor molecule D) to rhodamine 6G (R6G; acceptor molecule A) in ethanol. R6G

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excited by a nitrogen laser ($\lambda_{ex} = 337.1$ nm) shows lasing in ethanol solutions, whereas 10-phenyl-9-acetoxyanthracenes do not. Both dyes are very well soluble in ethanol. The chemical structures of the molecules under study are shown in Scheme I.

EXPERIMENTAL

Laser-grade R6G and spectrograde ethanol were used without further purification. 10-Phenyl-9-acetoxyanthracene derivatives were synthesized and purified by Gronowska *et al.* [23]. The absorption spectra of the dyes and their mixtures were recorded using a Carl Zeiss Jena M-40 spectrophotometer. The fluorescence spectra of the dyes and their mixtures were recorded on a Perkin Elmer Type 402 spectrofluorometer using front-surface excitation geometry. Necessary corrections were made for the grating monochromator and photomultiplier tube (IP28) sensitivities.

Lasing of the R6G solution and its mixtures with 10-phenyl-9-acetoxyanthracene derivatives were studied using a pulsed nitrogen laser (Lasertechnik Berlin Type MSG800SM; peak power, 1 MW; pulse width, 1 ns; repetition rate, 20–30 pps) with a transverse dye laser oscillator cavity. The dye laser output was detected by a grating monochromator (Zeiss Jena Type SPM2; grating, 650 slits/mm) and by an EMI9684QB photomultiplier and then recorded with a digital oscilloscope (HP Type 54510B) connected to a personal computer (PC) used for storage and further computation. The laser gain spectra were determined using the method of Shank *et al.* [24]

and the recording setup described in Refs. 22 and 25. The dye cell (20 × 20 mm) was transversely pumped by a pulsed beam from a N_2 laser operated at 337.1 nm with 25-Hz repetition. The pumping beam had a rectangular cross section and was focused by a cylindrical lens so that its size on the dye cell was 2 × 20 mm.

The amplified spontaneous emission (ASE) of the dye solution was directed into a grating monochromator. The aperture, as an additional horizontal slit, was set in front of the entrance slit of the SPM-2. The output signal from the photomultiplier was integrated and transformed into a digital signal. These signals were averaged over 50 laser shots and stored in the PC. The intensity spectrum of the ASE light was measured for the full length (l) and for the one-half of the full length ($l/2$) of the sample cell. According to Shank *et al.* [24] the gain value, $G(\lambda)$, of the ASE can be calculated using the formula:

$$G(\lambda) = \frac{2}{l} \ln\left(\frac{I_l(\lambda)}{I_{l/2}(\lambda)} - 1\right) \qquad (1)$$

where $I_l(\lambda)$ and $I_{l/2}(\lambda)$ are the ASE intensities measured when the full length and half-length of the cell are pumped, respectively.

The fluorescence decay times of the donor (τ_D) and acceptor (τ_A) molecules were measured with a pulsed fluorometer (Edinburg Analytical Instruments FLA-900). The samples were excited by a monochromator with pulsed light from a hydrogen lamp at $\lambda_{ex} = 364$ nm. The fluorescence decay curves were determined using standard single-photon counting equipment. The experimental points of the decay curves were fitted by an itera-
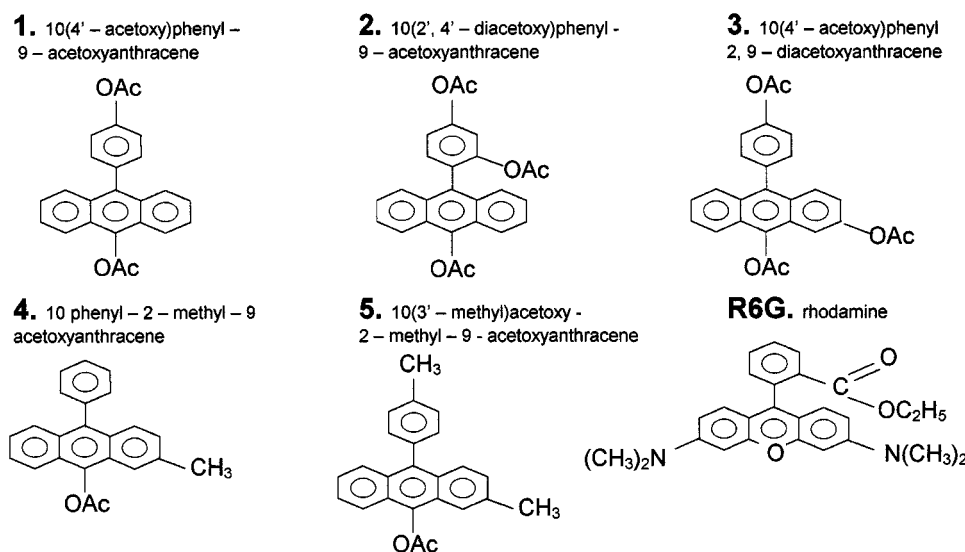
Scheme 1. Chemical structures of 10-phenyl-9-acetoxyanthracene derivatives.

tive deconvolution technique (Marquardt algorithm) to a single- or double-exponential model function.

The fluorescence quantum yields, ϕ_F, were measured using the experimental setup described by Gryczyński *et al.* [26]. The apparatus was calibrated using a solution of 9,10-diphenylanthracene ($\phi_F = 0.84$ [27]) and quinine bisulfate in 0.1 *M* sulfuric acid ($\phi_F = 0.51$ [28]) as references.

The ϕ_F values were calculated using the relation [29]:

$$\phi_F = \phi_F^s \frac{\displaystyle\int_0^\infty I_F(\tilde{v})d\tilde{v}}{\displaystyle\int_0^\infty I_F^s(\tilde{v})d\tilde{v}} \frac{1 - 10^{-A_s}}{1 - 10^{-A}} \frac{n^2}{n_s^2} \qquad (2)$$

where ϕ_F^s, is the absolute quantum yield of the standard substances, $A_s = \epsilon_s(\tilde{v}) \cdot c_s \cdot l$ and $A = \epsilon(\tilde{v}) \cdot c \cdot l$ are the absorbancies of the standard and studied sample, respectively, and n_s and n are the refractive indices of their solutions. The standard deviation of ϕ_F is ± 0.05.

RESULTS AND DISCUSSION

One-Compound Solutions

Figure 1 shows the absorption and emission spectra of an exemplary donor molecule, i.e., 10-(4′-acetoxy)phenyl-9-acetoxyanthracene, and the acceptor molecule rho-
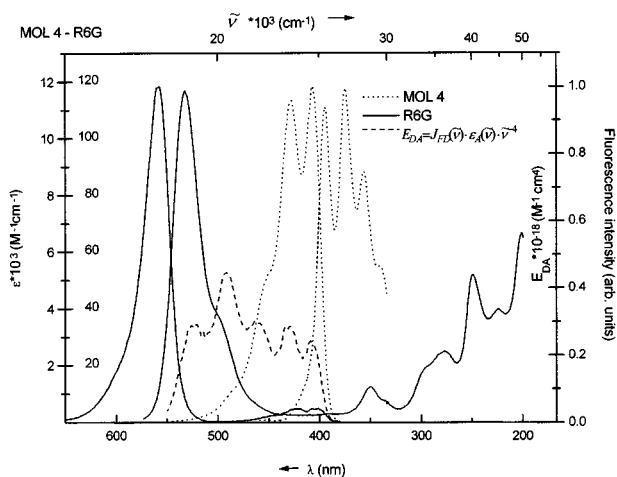

Fig. 1. Absorption and fluorescence spectra of 10-(4′-acetoxy)phenyl-9-acetoxyanthracene (**1**) (········) and rhodamine 6G (——) in ethanol, $c = 10^{-5}$ *M*. The dashed line gives the overlap spectrum of the **1**–R6G pair.

damine 6G in ethanol at equal concentrations, $c = 10^{-5}$ *M*. The overlap spectrum of this pair of D–A molecules, shown in Fig. 1 by a dashed line, suggests that energy transfer from **1** to R6G can take place. Similar overlap spectra were obtained for all other molecular D–A(R6G) pairs under study. The values of the overlap integrals were obtained using the following formula [15]:

$$G_{DA} = \int_0^\infty J_{FD}(\tilde{v}) \cdot \epsilon_A(\tilde{v}) \cdot \tilde{v}^{-4}d\tilde{v} \qquad (3)$$

In Eq. (3) $\tilde{v}$ is the wavenumber, $J_{FD}(\tilde{v})$ is the spectral distribution of the normalized donor fluorescence intensity $\int_0^\infty J_{FD}(\tilde{v})d\tilde{v} = 1$, and $\epsilon_A(\tilde{v})$ is the decimal molar extinction coefficient of the acceptor. The calculated G_{DA} values are listed in Table I. The G_{DA} data obtained for the different D–A pairs increase with an increasing number of functional groups (–OAc) or (–Me) substituted in the parent molecule of the donor. Within each group of donor molecules, (**1, 2**, and **3**) and (**4 and 5**), the G_{DA} value increases on adding a successive functional group to the donor 10-phenyl-9-acetoxyanthracene, by about $0.40 \cdot 10^{-14}$ dm³ cm³ M^{-1}. This constitutes an increase of about 25%. These noted changes of the G_{DA} values result from the shift and spectral half-width as well as from the transition probability changes of the absorption and emission spectra caused by the substituents in the parent molecule (the additive substitution effect) [21–23].

Figure 2A shows an exemplary fluorescence decay curve of molecule **4** obtained by excitation of the ethanol solution at the maximum of its longwave absorption band. The fluorescence decay data obtained for the one-compound solutions were fitted to a single-exponential function. The accuracy of the fits, determined by $\chi^2 \cong 1$, for all molecules under study and the D–A pairs was similar. The experimental uncertainty of the τ determination was ± 0.05 ns. Also, the fluorescence decay curves of the donor molecules quenched with R6G are single-exponential (see Fig. 2B).

Fluorescence Quenching of 10-(4′-Acetoxy)phenyl-9-acetoxyanthracenes by Rhodamine 6G

In our earlier work [24] it was shown that the quantum yield of the fluorescence of several 10-phenyl-9-acetoxyanthracene derivatives decreases as the temperature of the solvent increases. This observation was explained by population changes of the S_1 state caused by nonradiative $S_1 \rightarrow T_2$ transitions. This supposition has been confirmed by quantum mechanic calculations [22].

Table I. Experimental Values of the Energy Transfer Parameters for the Donor—Acceptor Pairs Investigated

Molecule	$G_{DA} = \int_0^{\infty} J_{FD}(\tilde{v}) \cdot \epsilon_A(\tilde{v}) \cdot \tilde{v}^{-4}\, d\tilde{v}$ $(10^{-14}\ M^{-1}\ cm^3)$	$K_{SV} = tg\alpha$ $(10^5\ M^{-1})$	τ_D (ns)	k_{ET} (S–V) $(10^{12}\ M^{-1}\ s^{-1})^a$	R_0 (nm)^b	ϕ_F^D
1	1.620	0.1744	7.38	2.36	13.3	0.88
2	1.879	0.1203	7.15	1.68	13.6	0.89
3	2.062	0.1417	8.26	1.72	13.8	0.88
4	1.840	0.1458	8.49	1.72	13.5	0.87
5	2.042	0.1637	7.92	2.07	13.7	0.83

^{a} Data determined from the Stern–Volmer plots.
^{b} Calculated using Eq. (6).

Figure 3 shows the emission spectrum of 10-(4′-acetoxy)phenyl-9-acetoxyanthracene (**1**) obtained in the presence of R6G at various concentrations. As shown, the total fluorescence intensity of the donor molecules decreases with increasing concentrations of the acceptor, whereas the shape of the spectrum remains unchanged. The same dependence is noted for all other D–A pairs under study.

Since the fluorescence spectra of the donor molecules in the presence of R6G were obtained under continuous and constant illumination, the fluorescence intensity changes caused by the quencher (acceptor molecules) can be described by the Stern–Volmer equation [15]:

$$\phi_{D0}/\phi_D = 1 + K_{SV}\,[A] = 1 + k_{ET} \cdot \tau_D[A] \qquad (4)$$

where ϕ_{D0} and ϕ_D are the fluorescence quantum yields of the donor in solution without and with an acceptor molecule, respectively, k_{ET} is the quenching rate constant (or energy transfer constant), $[A]$ is the concentration of acceptor molecules, and τ_D is the fluorescence decay time of the donor. Figure 4 shows Stern–Volmer plots of the D–A pairs under study versus the R6G concentration.
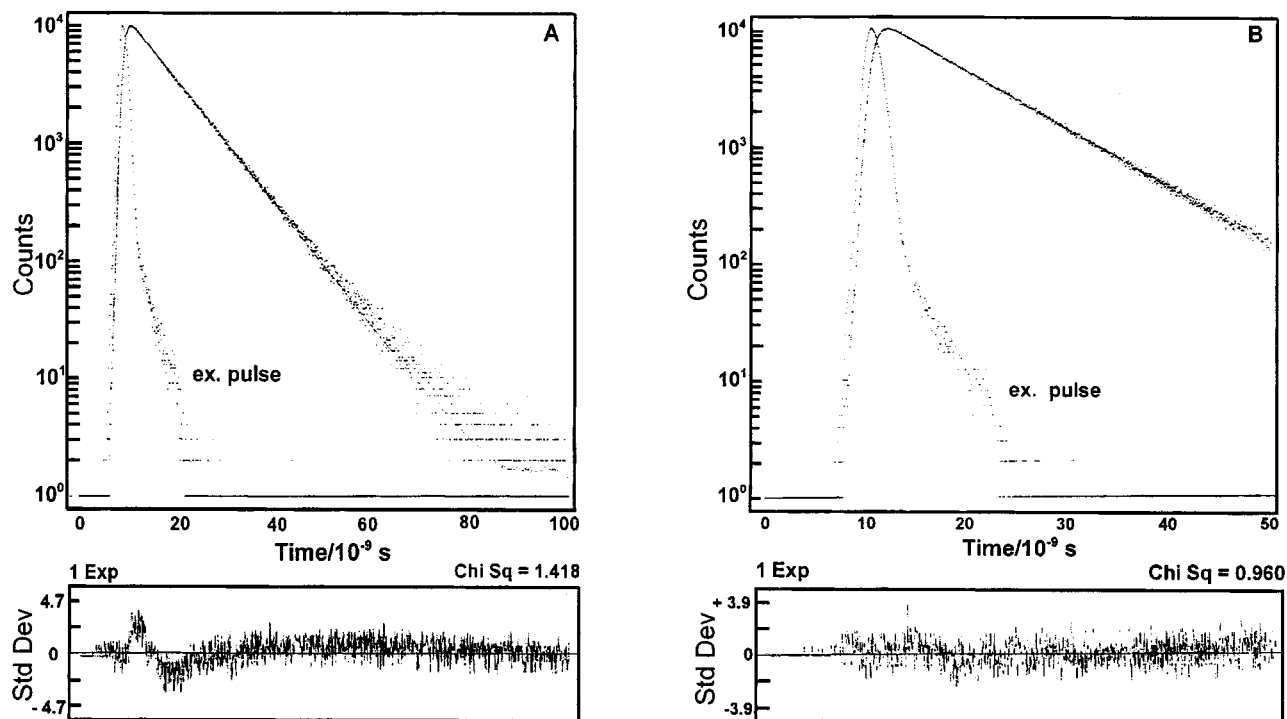

Fig. 2. Fluorescence decay curves of 10-phenyl-2-methyl-9-acetoxyanthracene (**4**) in pure ethanol (A) and in a mixture of **4** with rhodamine 6G, $c_{R6G} = 2 \cdot 10^{-4}\ M$ and $c_4 = 10^{-4}\ M$ (B). $\lambda_{ex} = 364$ nm, $\lambda_F = 425$ nm.
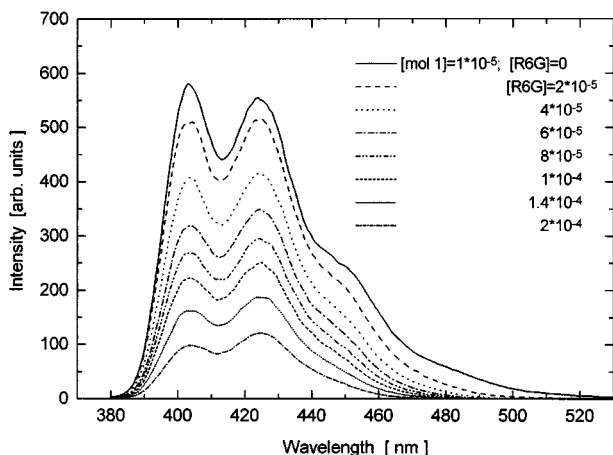
Fig. 3. The fluorescence spectrum of 10-(2′-acetoxy)phenyl-9-acetoxy-anthracene (D) quenched by rhodamine 6G (A). The concentration of D molecules, $c_D = 10^{-5}$ M, whereas c_A changes from $2 \cdot 10^{-5}$ to $2 \cdot 10^{-4}$ M. $\lambda_{ex} = 364$ nm.

Knowing the slope values of the Stern–Volmer curves and the $K_{SV} = tg\alpha_{SV}$ and τ_D values determined from separate measurements, the k_{ET} values were determined. Table I assembles all the experimentally obtained data. Analyzing them it follows that the K_{SV} and τ_D of the donor molecules depend on the number of substituted

functional groups in the parent molecule, whereas the fluorescence quantum yield of the donor molecules, ϕ_F^D, is constant in the error limit of its determination.

Taking into account the value of the overlap integral (see Table I), we can assume that the decrease in the fluorescence intensity of the donor molecules in the mixed solutions is caused by resonance energy transfer D → A. According to Förster's theory [15], if the energy transfer between the D–A pair is caused by dipole–dipole interaction, the energy transfer constant is given by [15]:

$$k_{ET} = 9.0 \times 10^{-25} \chi^2 \phi_D \cdot G_{DA}/R^6 \tau_D \cdot n^4 \qquad (5)$$

where χ^2 is a dimensionless orientation factor which is equal to 3/2 for randomly distributed transition moments of molecules involved in the energy transfer, R is the distance between D and A, and n is the refractive index of the solvent. Using the k_{ET} value determined using the Stern–Volmer formula and Eq. (5), the critical energy transfer distance, R_0, at which the energy transfer rate, k_{ET}, is equal to the fluorescence decay rate of the donor, $k_{ET} = (1/\tau_D^0)(R_0/R)^2$, can be calculated using the formula

$$R_0^6 = 9.0 \times 10^{-25} \chi^2 \phi_D G_{DA} n^{-4} \qquad (6)$$

These R_0 values (see Table I, column 6) correspond to the case where the D–A distance is constant, i.e., as in the solid phase or for bichromophoric D–A molecules.
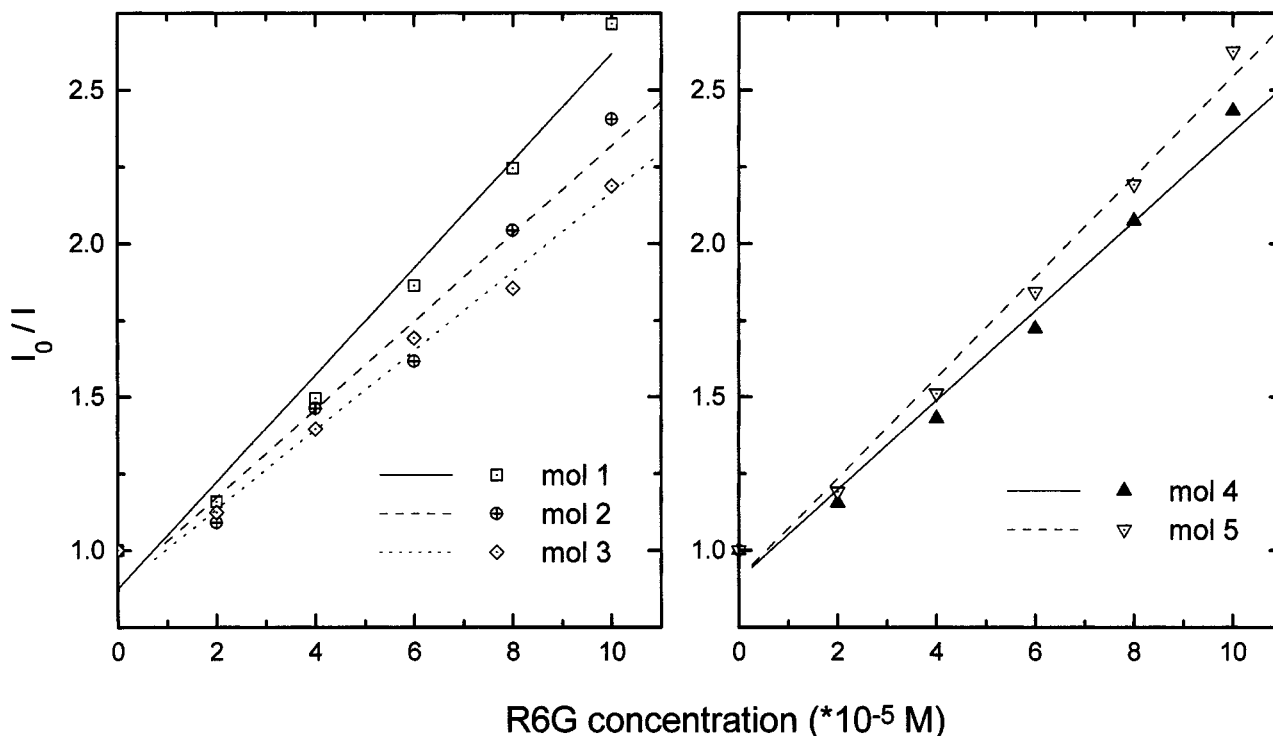

Fig. 4. Stern–Volmer dependence of the quenching fluorescence of 10-phenyl-9-acetoxyanthracene derivatives with rhodamine 6G in methanol.

For D–A pairs in solution, as in our case, the R_0 values have to be calculated using a modified relation [31]:

$$k_{ET} = \frac{R_0^3}{(7.35 \cdot 10^{-8})^3 \, \tau_D} \quad (7)$$

Making use of the experimentally determined k_{ET} data and Eq. (7), the respective R_0 values were calculated. They are also presented in Table II (column 5).

Figure 5 shows the ratio of quantum yield ϕ_{D+A}/ϕ_D and fluorescence decay time τ_D^0/τ_D dependence versus the quencher concentration, according to Förster's theory. As shown, the two curves do not overlap. The ϕ_{D+A}/ϕ_D curve behaves as predicted by theory, whereas the τ_{D/τ_D}^0 curve shows a very weak dependence on the acceptor concentration. This behavior is noted for all D–A pairs under study. This allows us to conclude that in the mixed solutions a static quenching process takes place which is accompanied by the formation of an excited nonemitting (DA) complex.

The ϕ_{D+A}/ϕ_D dependence on the quencher concentration (see Fig. 5) allows us to determine the concentration of the acceptor required to decrease the fluorescence of the donor by one-half. The determined values of $[A]_{1/2}$ are listed in Table I. These $[A]_{1/2}$ data can be used to determine the critical energy transfer distance, R_0, using the dependence [14]

$$[A]_{1/2} = \frac{3}{4\pi N R_0^3} \quad (8)$$

Equation (8) gives an independent way to determine the R_0 value at which the excitation energy transfer from the donor to the acceptor equals 50%. In Eq. (8) N is Avogadro's number. The R_0 values calculated from Eq.
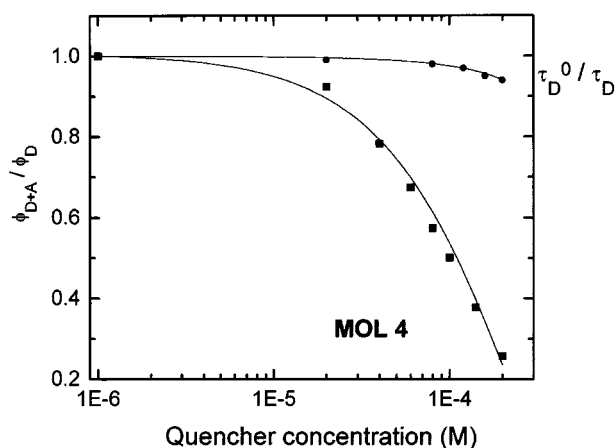
(8) are listed in Table II (column 3). Using them, the respective k_{ET} constants were calculated. They are also listed in Table II.

Analyzing the data presented in both tables, the following conclusions can be drawn.

1. The theoretically calculated R_0 values obtained using formula (8) are larger than those determined based on the Stern–Volmer plots and Eq. (6). The R_0 data show a weak dependence on the number of substituents of the donors. The changes in R_0 values are small and within the error limit.
2. The k_{ET} data show an irregular dependence on the number and kind of functional groups in the D molecule. They are two orders larger then the corresponding values supposing that the collision mechanism is involved in the quenching process: $k_{diff} = 0.5 \cdot 10^{10} \, M^{-1} \, s^{-1}$ [32].
3. The R_0 values calculated using Eq. (6) are about one order of magnitude larger than the radii of the D–A collision complex, $R_{diff} = R_A + R_D \cong 9.5 \cdot 10^{-10}$ m. The noted difference points out that the collision energy transfer mechanism does not play an important role.

The noted differences between the R_0 and the R_{diff} values as well as the good coincidence between the respective k_{ET}^{S-V} and k_{ET}^F data indicate that in the studied solution mixtures, the long-range dipole–dipole interaction is responsible for the fluorescence quenching of the donor molecules.

Laser Characteristics

These studies concern the experimental determination of the gain spectrum, $G(\lambda)$, and tuning range of the spontaneous amplified emission (ASE) of dye mixtures. The measurements were performed maintaining a con-

Fig. 5. The relative quantum yield ϕ_{D+A}/ϕ_D and τ_D^0/τ_D ratio of R6G (A) versus the concentration of quencher c_A. The donor molecule is 2,10-(2′,4′-acetoxy)phenyl-9-acetoxyanthracene.

Table II. Calculated Data of the Energy Transfer Parameters of the D–A Pairs Investigated

Molecule	$[A]_{1/2}$ $(10^{-5} \, M)$	$R_{0[A]_{1/2}}$ $(nm)^a$	k_{ET}^F $(10^{12} \, M^{-1} \, s^{-1})^b$	R_0 $(10^{-10} \, m)$
1	8.9	16.4	1.52	1.18
2	8.0	17.0	1.75	1.70
3	7.8	17.1	1.55	1.40
4	1.1	15.2	1.05	1.42
5	9.6	16.1	1.32	1.17

^{a} Calculated using Eq. (8).
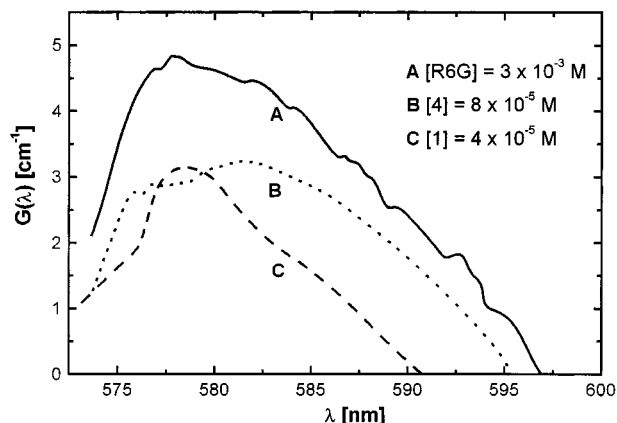^{b} Calculated using Eq. (7) and R_0 values determined using Eq. (8).

Fig. 6. The gain spectrum of R6G in mixed solutions of donor molecules: (A) solution of R6G $c_A = 3 \cdot 10^{-3}$ M; (B) R6G, $c_A = 3 \cdot 10^{-3}$ M, and 10-phenyl-2-methyl-9-acetoxyanthracene, $c_D = 8 \cdot 10^{-5}$ M; (C) R6G, $c_A = 3 \cdot 10^{-3}$ M, and 10-(4′-acetoxy)phenyl-9-acetoxyanthracene, $c_D = 4 \cdot 10^{-5}$ M.

stant concentration of the acceptor (R6G) and varying the concentration of the donor.

Figure 6 shows the effect of donor concentration changes on the gain spectrum of the dye mixture. This spectrum gives at the tuning range of ASE radiation. As follows from comparison of the gain spectrum of an R6G ethanol solution (solid line) and dye solution mixtures of the 1–R6G pair (dotted and dashed lines), the two active laser media possess different spectral ranges and gain values. For all other derivatives of 10-phenyl-9-axetoxy-anthracene added to the lasing solution, a decrease in the laser emission of R6G was observed (also at low donor concentrations, $\sim 10^{-5}$ M). Also, the lasing spectral range of R6G is narrower (see Fig. 6). The intensity of the laser emission decreases with increasing donor concentration; ASE is not observed at a donor concentration $c > 2 \cdot 10^{-4}$ M. The decrease in the lasing spectral range of R6G from 560–597 to 565–588 nm caused by the donor molecules can be attributed to the excited-state absorption, $S_1 \rightarrow S_n$, of the donor molecules. In the region 550–650 nm with $\lambda_{max} \cong 590$ nm the 10-phenyl-9-acetoxy-yanthracene derivatives possess a strong $S_1(B_{2u}) \rightarrow S_k$ ($^1A_{1g}$) absorption band [22].

CONCLUSIONS

From our studies on energy transfer from 10-phenyl-9-acetoxyanthracene derivatives (donors) to R6G (acceptor) in ethanol solutions, the following conclusions can be drawn:

1. R6G is an efficient quencher for the fluorescence of the donor molecules **1–5**. The quenching proc-

ess is well described by the Stern–Volmer formula and Förster theory.

2. Calculated and experimentally determined values of the Förster critical radius, R_0, and rate constants, k_{ET}, of the D–A pairs are larger than those for which the collision mechanism would be involved (i.e., $R_0 \gtrsim R_D + R_A$, and $k_{ET} \cong 2 \cdot 10^{12}$ dm^3 M^{-1} s^{-1} $\gtrsim k_{Diff} \cong 0.5 \cdot 10^{10}$ M^{-1} s^{-1}. This clearly indicates that in this case resonance energy transfer is responsible for the observed quenching of the donor fluorescence. The weak dependence of the τ_D^0/τ_D ratio on the acceptor concentration allows us to conclude that the static quenching is accompanied by the formation of an excited non emitting (DA)* complex.

3. 10-Phenyl-9-acetoxyanthracene derivatives in mixed solutions with R6G quench the lasing ability of R6G; i.e., they decrease the intensity of laser emission and narrow the lasing spectral range.

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