# Dynamics of photoexcited donor-acceptor complexes between $C_{60}$ and *N*,*N*-diethylaniline. Polarization picosecond spectroscopy study

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The kinetics of the formation and decay of photoexcited radical ion pairs of donor-acceptor charge-transfer complexes between $C_{60}$ and *N*,*N*-diethylaniline (DEA) in chlorobenzene was studied by picosecond laser-induced diffraction gratings. It was established that the anisotropy of polarization of the diffraction signal decreases as the concentration of DEA increases. The radical ion states of the photoexcited $C_{60}^{-}$...$DEA^{+}$ complex have zero anisotropy. This effect is likely due to the isotropic intracomplex transfer of an electron from the local excited state to the radical ion state. The rate constant of quenching of the singlet excited $C_{60}$ by *N*,*N*-diethylaniline ($1.4 \cdot 10^{10}$ L $mol^{-1}$ $s^{-1}$) and the lifetimes of the solvent-separated $C_{60}^{-}$...$DEA^{+}$ and tight [$C_{60}^{-}$...$DEA^{+}$] (95±7 and 31±4 ps, respectively) radical ion pairs were measured.

**Key words:** fullerenes, charge-transfer complexes; electron transfer; picosecond laser photolysis, polarization picosecond laser-induced gratings.

In previous experiments with picosecond induced diffraction gratings, it has been shown that optical anisotropy of the diffraction signal is observed for solutions of fullerenes $C_{60}$ and $C_{70}$ in organic solvents.[1—8] The kinetics of the decay of the anisotropic response is caused mainly by the orientational rotational motion of the fullerene molecules. For several typical organic solvents such as toluene, dichlorobenzene, xylene, nitrobenzene, decalin, and others, the times of the orientational rotational relaxation of fullerenes $C_{60}$ and $C_{70}$ are anomalously low compared to those predicted by the Debye—Stokes—Einstein theory. This was explained in terms of the theory of rough spheres, which adequately describes orientational motion for boundary conditions close to free slipping conditions, *i.e.*, the absence of a chemical bond between the fullerene and solvent molecules.[1—8] In this work, *N*,*N*-diethylaniline (DEA) or its mixture with dichlorobenzene were chosen as solvents. Similarly to other tertiary aromatic amines, DEA forms a donor-acceptor complex with $C_{60}$. The kinetics of relaxation of radical ion pairs (RIP) formed during photoexcitation of the complex has been studied previously.[9—11] The purpose of this work is to establish the character of the optical anisotropy during excitation of the charge-transfer complex (CTC) between $C_{60}$ and DEA by a polarized light pulse.

## Experimental

The picosecond induced diffraction gratings method was used. A schematic diagram is presented in Fig. 1. The setup and method have been described previously.[1—8] The method of ordinary gratings was used: two exciting pulses with intensities of $I_{exc,1}$ and $I_{exc,2}$ have parallel polarization, $\mathbf{E}_{exc,1} \parallel \mathbf{E}_{exc,2}$. According to the interference light field, a periodic pattern of optical density and refraction index appears in the medium. A third pulse delayed in time is diffracted by the periodic structure in the medium. The detected intensity of the diffrac-

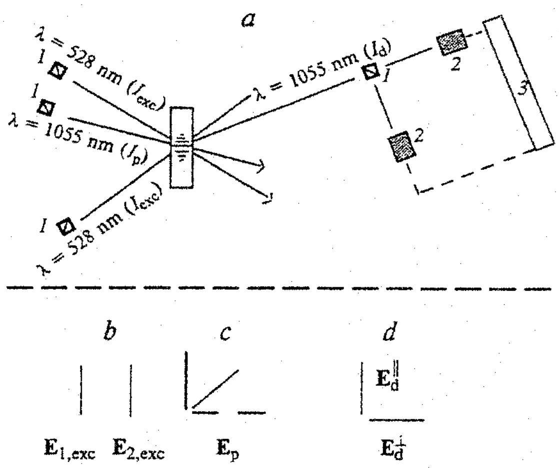

**Fig. 1.** Schematic optical diagram of the experiment (*a*): *1*, Glan polarizers; *2*, photodiodes; *3*, analog-to-digital converter combined with a computer; $I_{exc}$, exciting pulse; $I_p$, probing pulse; $I_d$, diffraction pulse. The mutual orientation of polarization of exciting (*b*), probing (*c*), and diffraction pulses (*d*) is shown.

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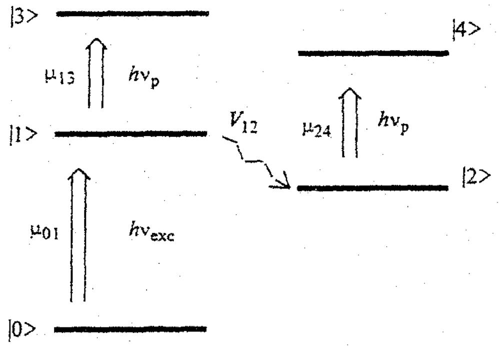

**Fig. 2.** Scheme of levels and resonance transitions determining the diffraction signal.

tion signal is $I_d \sim I_{exc,1} \cdot I_{exc,2} \cdot I_p$ ($I_p$ is the intensity of the probing pulse). In the general case, the efficiency of the diffraction signal ($\eta$) is determined by the amplitude $(\Delta D)^2$ and phase $(\Delta n)^2$ components:

$$\eta = I_d/I_p = a(\Delta D)^2 + b(\Delta n)^2, \qquad (1)$$

where $\Delta D$ and $\Delta n$ are changes in the absorption coefficients and the refraction index along the grating, respectively, *i.e.*, the difference between these values in the maxima and minima of the interference pattern. Experiments[3—8] with $C_{60}$ have shown that the contribution of the phase component is negligible in excitation with light at $\lambda_{exc}$ = 528 nm and probing at 1055 nm, because the signal is determined by resonance optical transitions. The general scheme of the resonance optical dipole transitions participating in the formation of the diffraction signal is presented in Fig. 2. The intensities of the two components of the diffraction signal with parallel ($I_d^{\parallel}$) and perpendicular ($I_d^{\perp}$) polarization relative to the polarization of the exciting light are determined by the population of the states and the mutual orientation of the dipoles of optical transitions $\mu_{01}$, $\mu_{13}$, and $\mu_{24}$. The optical anisotropy, $R(t)$, depends on the mutual orientation of the dipoles of the molecular optical transitions:[12]

$$R(t) = (2/5)\langle P_2[\mu_{ab}(t=0)\cdot\mu_{\alpha\beta}(t)]\rangle, \qquad (2)$$

where $P_2$ is the Legendre polynomial. The $R(t)$ value can be experimentally determined from the intensities of the $I_d^{\parallel}$ and $I_d^{\perp}$ components of the diffraction signal:

$$R(t)=\left[\sqrt{I_d^{\parallel}(t)}-\sqrt{I_d^{\perp}(t)}\right]\Big/\left[\sqrt{I_d^{\parallel}(t)}+2\sqrt{I_d^{\perp}(t)}\right]. \qquad (3)$$

Thus, this method allows one, in principle, to measure the kinetics of relaxation of excited states and the correlation times of dephasing of optically polarized states.

The pulse duration was 6 ps, and the wavelengths of the exciting and probing pulses were 528 and 1055 nm, respectively. Polarization of exciting pulses was vertical, and the angle of beam convergence was 0.02 rad, which corresponds to a pattern with a period of 50 μm. The size of the grating area was 250—300 μm. The energy of the exciting pulse was 5 μJ, and that of the probing pulse was ten times lower. The polarization of the probing pulse was directed at an angle of 45° to the polarization of the exciting pulse. The $I_d^{\parallel}$ and $I_d^{\perp}$ components were separated from the diffraction signal by a Glan polarizer. Both signals were simultaneously detected by photodiodes. Signals were detected and the delay path length was established automatically using a computer.

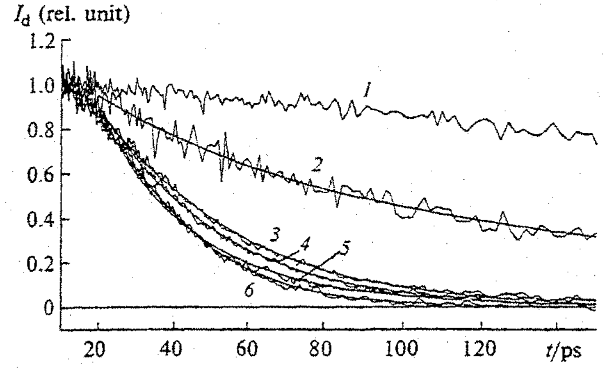

**Fig. 3.** Dependence of the intensity of the diffraction signal ($I_d$) on the delay time at different concentrations of DEA in the $C_{60}$—DEA—PhCl system: [DEA] (vol.%) = 0 (*1*), 2 (*2*), 8 (*3*), 15 (*4*), 30 (*5*), and 100 (*6*).

Solutions of $C_{60}$ with a concentration of $5 \cdot 10^{-3}$ mol $L^{-1}$ were used. Experiments were carried out in a quartz cell 0.4 mm thick at 23±1 °C.

## Results and Discussion

The kinetics of the diffraction signal $I_d$ at different concentrations of DEA is presented in Fig. 3. When delay times are short (0—20 ps), the $I_d^{\perp}(t)$ and $I_d^{\parallel}(t)$ components of the diffraction signal differ, which indicates that an anisotropy signal exists in this time interval.

The kinetics of the decay of anisotropy $R(t)$ at different concentrations of DEA is shown in Fig. 4. An increase in the concentration of DEA mainly results in a decrease in the initial value of the anisotropy signal $R_{t=0}$. The characteristic decay time of $R(t)$ (within experimental error) is independent of the concentration of DEA and almost coincides with the characteristic time of the anisotropy decay of $C_{60}$ in neat chlorobenzene.

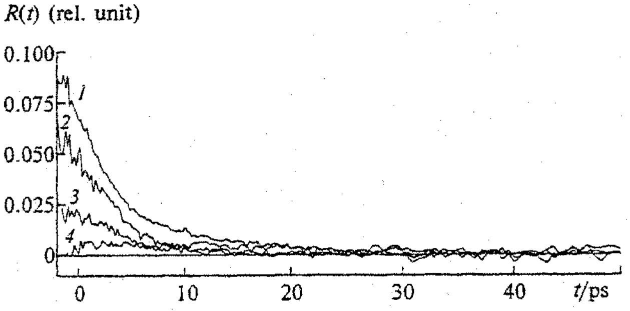

**Fig. 4.** Dependence of the anisotropy signal ($R(t)$) on the delay time at different concentrations of DEA in the $C_{60}$—DEA—PhCl system: [DEA] (vol.%) = 0 (*1*), 8 (*2*), 15 (*3*), and 30 (*4*).

The concentrations of free $C_{60}$ and the $[C_{60}...DEA]$ complex are determined by the equilibrium:

$$C_{60} + DEA \overset{K}{\rightleftharpoons} C_{60}...DEA, \qquad (4)$$

$K = 0.29$ L mol$^{-1}$ (see Ref. 10).

The action of the laser pulse results in the formation of excited $C_{60}$ molecules (for fullerene molecules unbound in a complex) and photoexcited CTC. A previous study of the spectra of the excited complexes[10] showed that the photoexcited complex is a tight RIP. The formation of the laser-induced gratings occurs due to the excitation of both unbound $C_{60}$ molecules and CTC.

$$C_{60} + h\nu \rightarrow C_{60}(S_1), \qquad (5)$$

$$C_{60}...DEA + h\nu \rightarrow C_{60}^{-}...DEA^{+}. \qquad (6)$$

The absorption spectra of singlet-excited $C_{60}(S_1)$ and $C_{60}^{-}...DEA^{+}$ RIP partially overlap, since the absorption band of the singlet-excited $C_{60}(S_1)$ has a maximum near 940—980 nm,[13—15] and a maximum in the absorption spectrum of the $C_{60}^{-}$ radical anion lies at 1070 nm.[16] Therefore, the efficiency of diffraction at $\lambda = 1055$ nm is determined by the sum of the contributions of absorption from $C_{60}(S_1)$ $(\Delta D_{C_{60}})$ and $C_{60}^{-}$ $(\Delta D_{CT})$.

$$\eta \sim (\Delta D_{C_{60}} + \Delta D_{CT})^2 \qquad (7)$$

Relaxation of the signal of the laser-induced grating are caused by both the relaxation of excited states:

$$C_{60}^{-}...DEA^{+} \rightarrow C_{60} + DEA\ (\tau_c), \qquad (8)$$

$$C_{60}(S_1) \rightarrow C_{60}\ (\tau^*), \qquad (9)$$

$$C_{60}(S_1) + DEA \xrightarrow{k_q} C_{60}^{-}...DEA^{+}, \qquad (10)$$

($\tau_c$ is the relaxation time of RIP; $\tau^*$ is the lifetime of $C_{60}(S_1)$; $k_q$ is the quenching rate constant of excited $C_{60}(S_1)$) and processes related to the relaxation of optically induced polarization in the system. The anisotropy of the diffraction signal ($R$) reaches zero when time delays >20 ps. (Relaxation of polarization will be considered in detail below.)

Let us consider the decay kinetics of the diffraction signal $I_d$ at delay times >20 ps when anisotropy of the diffraction signal is almost absent. The $I_d$ signal gives information on the concentration of excited states in the system and is determined by the relaxation kinetics of singlet-excited $C_{60}(S_1)$ and RIP. The experiments were carried out with the same thickness of the cell and the same intensities of the probing pulse; therefore, the total diffraction signal can be expressed through the concentrations of the excited $C_{60}(S_1)$ and $[C_{60}^{-}...DEA^{+}]$ CTC:

$$I_d \sim (\Delta\varepsilon^{*}_{C_{60}} \cdot [C_{60}(S_1)] + \Delta\varepsilon_{CT} \cdot [C_{60}^{-}...DEA^{+}])^2, \qquad (11)$$

where $\Delta\varepsilon^{*}_{C_{60}}$ and $\Delta\varepsilon_{CT}$ are the differences between the extinction coefficients in the ground and excited states of the $C_{60}$ molecule and complex at $\lambda = 1055$ nm, respectively.

Solving the system of differential equations for reactions (5), (6), (8)—(10) and inserting the determined expressions for concentrations into Eq. (11), we obtain

$$I_d(t) \sim [A\exp(-k_c t) - B\exp(-k_{\mathrm{eff}} t)]^2, \qquad (12)$$

where $k_c = 1/\tau_c$; $k_{\mathrm{eff}} = k_q[DEA] + 1/\tau^*$ ($\tau_c$ is the lifetime of RIP); $A$ and $B$ are coefficients depending on the initial concentrations $[C_{60}(S_1)]_0$ and $[C_{60}^{-}...DEA]_0$ after the exciting pulse and on the $\tau_c$, $\tau^*$, and $k_q$ values. The lifetime of $C_{60}(S_1)$ $(\tau^*)$ is known and is equal to 1200±100 ps.[13—15] Equation (12) predicts biexponential decay kinetics $I_d(t)$. In fact, the experimental time dependences of the diffraction signals can be described best of all by the difference between two exponents. This can be seen from Fig. 3, which presents the theoretical curves obtained after the fitting procedure of Eq. (12) to experimental points along with the experimental curves.

The decay of the diffraction signal becomes biexponential at the initial delay times when delayed decay of the diffraction signal is observed, which is probably related to an additional accumulation of RIP due to quenching of excited $C_{60}(S_1)$ (see reaction (10)). The determined value of the quenching rate constant of excited $C_{60}(S_1)$, $k_q = (1.4\pm0.6)\cdot10^{10}$ L mol$^{-1}$ s$^{-1}$, is independent of the concentration of DEA in the solution. The $k_q$ value almost coincides with the diffusion-controlled quenching constant ($1.6\cdot10^{10}$ L mol$^{-1}$ s$^{-1}$) calculated for solutions of $C_{60}$ and DEA in chlorobenzene.

The treatment of the experimental curves at different concentrations of DEA in a solution using Eq. (12) gave the values of relaxation times $\tau_c$. The determined relaxation times of RIP differ noticeably at low and high concentrations of DEA in the solution. When the concentration of DEA was high (from 8 to 100%), the relaxation time of RIP was $\tau_{c,1} = 31\pm4$ ps and depended slightly on the content of DEA in the solution. When the concentration of DEA was low (0.5 and 2%), the relaxation time of RIP was $\tau_{c,2} = 95\pm7$ ps. This indicates that the RIP formed in the solution have different natures. When the concentration of DEA is low, $C_{60}$ exists predominantly in the bound form. The quenching of singlet-excited $C_{60}$ results in the appearance of RIP with a long lifetime. When the concentration of DEA is high, the signal from RIP formed in the excitation of the complex predominates. The lifetime of these pairs is short. In the first case, RIP can be considered to be solvent-separated RIP, and in the second case, to be tight pairs. The stronger electron-electron interaction in tight pairs causes a higher rate constant of relaxation of this state.[17] The processes of the formation and recombination of solvate-separated and tight RIP can be presented as follows:

$$C_{60}...DEA + h\nu \rightarrow C_{60}^{-}/DEA^{+}, \qquad (13)$$

$$C_{60}(S_1) + DEA \rightarrow C_{60}^{-}//DEA^{+}, \qquad (14)$$

$$C_{60}^{-}/DEA^{+} \rightarrow C_{60}...DEA\ (\tau_{c,1}), \quad (15)$$

$$C_{60}^{-}//DEA^{+} \rightarrow C_{60}...DEA\ (\tau_{c,2}). \quad (16)$$

**Anisotropy of the diffraction signal.** Analysis of the observed anisotropy $R(t)$ makes it possible to estimate the value of anisotropy of the polarized state of tight RIP ($r_{CT}$). As can be seen in Fig. 4, when the content of DEA increases, a monotonic decrease in the amplitude of the $R(t)$ signal is observed, but the characteristic decay time of $R(t)$ remains unchanged. This indicates that the anisotropy of the diffraction signal is caused mainly by unbound $C_{60}$, and the anisotropy of the radical ion pair $r_{CT}$ is either insignificant or equal to zero. These qualitative observations are confirmed by the more detailed analysis of the anisotropy signal at different concentrations of DEA. For example, the following equations can be written for the vertical $(I_d^{\parallel})_{r=0}$ and horizontal $(I_d^{\perp})_{r=0}$ components:[18]

$$(I_d^{\parallel})_{r=0} = C\cdot\{(\Delta\varepsilon^{*}_{C_{60}}\cdot[C_{60}(S_1)]_0\cdot G^{\parallel}_{C_{60}}) + (\Delta\varepsilon_{CT}\cdot[C_{60}^{-}...DEA^{+}]_0\cdot G^{\parallel}_{CT})\}^2, \quad (17a)$$

$$(I_d^{\perp})_{r=0} = C\cdot\{(\Delta\varepsilon^{*}_{C_{60}}\cdot[C_{60}(S_1)]_0\cdot G^{\perp}_{C_{60}}) + (\Delta\varepsilon_{CT}\cdot[C_{60}^{-}...DEA^{+}]_0\cdot G^{\perp}_{CT})\}^2, \quad (17b)$$

where the factors $G^{\parallel}_{C_{60}} = 1 + 2r_{C_{60}}$, $G^{\parallel}_{CT} = 1 + 2r_{CT}$ and $G^{\perp}_{C_{60}} = 1 - r_{C_{60}}$, $G^{\perp}_{CT} = 1 - r_{CT}$ are determined by the values of the initial anisotropies $r_{C_{60}}$ and $r_{CT}$ for $C_{60}(S_1)$ and the $[C_{60}^{-}...DEA^{+}]$ complex in chlorobenzene, respectively; $C$ is a coefficient that depends on the intensities of the exciting and probing pulses.

The concentrations of excited $C_{60}(S_1)$ and $[C_{60}^{-}...DEA^{+}]$ are related to the intensity of the probing pulse by the correlations:

$$[C_{60}(S_1)]_0 = \frac{k[C_{60}]\sigma_{C_{60}}I_{exc}}{\sigma_{C_{60}}I_{exc} + \tau^{-1}_{C_{60}}}, \quad (18)$$

$$[C_{60}^{-}...DEA]_0 = \frac{k[C_{60}...DEA]\sigma_{CT}I_{exc}}{\sigma_{CT}I_{exc} + \tau^{-1}_{CT}}, \quad (19)$$

where $k$ is a coefficient that depends on the duration of the exciting pulse; $\sigma_{C_{60}}$ and $\sigma_{CT}$ are the absorption cross sections for $C_{60}$ and the $[C_{60}...DEA]$ complex at 528 nm, respectively. Since the diffraction signal is proportional to the square of $I_{exc}$, the conditions are far from saturation of the optical transition, and the concentrations of excited states are proportional to the concentrations of free $C_{60}$ and the complex:

$$[C_{60}(S_1)]_0 \sim [C_{60}]\sigma_{C_{60}} \quad (20)$$

$$[C_{60}^{-}...DEA^{+}]_0 \sim [C_{60}...DEA]\sigma_{CT} \quad (21)$$

The last two equations make it possible to express the anisotropy $R_{r=0}$ through the ratio of concentrations of free $C_{60}$ and CTC and through the ratio of extinction coefficients for different transitions in $C_{60}$ and in the complex:

$$I_d^{\parallel} \sim \left(G^{\parallel}_{C_{60}} + EG^{\parallel}_{CT}\cdot\frac{[C_{60}...DEA]}{[C_{60}]}\right)^2, \quad (22a)$$

$$I_d^{\perp} \sim \left(G^{\perp}_{C_{60}} + EG^{\perp}_{CT}\cdot\frac{[C_{60}...DEA]}{[C_{60}]}\right)^2, \quad (22b)$$

$$R_{r=0} = \left(\sqrt{I_d^{\parallel}} - \sqrt{I_d^{\perp}}\right)\Big/\left(\sqrt{I_d^{\parallel}} + 2\sqrt{I_d^{\perp}}\right), \quad (22c)$$

where $E = (\sigma_{CT}\Delta\varepsilon_{CT})/(\sigma_{C_{60}}\Delta\varepsilon^{*}_{C_{60}})$. The concentrations of $C_{60}$, the complex, and DEA are related by the correlation $[C_{60}...DEA] = K\cdot[C_{60}]\cdot[DEA]$. The $E$ value can be estimated from the dependence of the intensity of diffraction $I_d$ on the amine concentration at delay times when the diffraction anisotropy becomes zero, the condition $I_d^{\parallel} \approx I_d^{\perp}$ is fulfilled, and the increase in $I_d$ due to the decay of excited states is still insignificant. In this case, the diffraction signal depends only on the $E$ coefficient and the concentration of DEA:

$$I_d \sim \left(\frac{1 + EK\cdot[DEA]}{1 + K\cdot[DEA]}\right)^2. \quad (23)$$

The best fitting of the experimental points and the curve corresponding to expression (23) is achieved at $E = 5.5\pm0.5$. Thus, the initial anisotropy $r_{CT}$ can be determined at different concentrations of DEA from the observed anisotropy $R_{r=0}$ using correlations (22). The $r_{C_{60}}$ value of free $C_{60}$ in chlorobenzene was used for the solution of the equations. The determined value $r_{CT} = 0\pm0.01$ is independent of the concentration of DEA in the solution.

The fact that the anisotropy in the RIP state is zero confirms the even distribution of RIP relative to the direction of the vector of polarization of the exciting light. In the general case, when molecules are excited by polarized light, photoselection occurs due to the predominant excitation of those molecules whose transition momentum coincides with the vector of polarization of the exciting light. When these molecules are probed by the polarized light, the diffraction signals $I_d^{\parallel}$ and $I_d^{\perp}$ differ to give an anisotropy value not equal to zero. The nonzero vector of the transition momentum $\mu$ must correspond to the excitation process (see reaction (6)); therefore, irradiation with polarized light will result in the predominant excitation of those complexes whose maximum component of $\mu$ is directed the same way as the vector of polarization of the exciting light $\mathbf{E}_{exc}$. Thus, direct photoexcitation with polarized light to the RIP state (see reaction (6)) suggests the existence of photoselection for RIP and nonzero anisotropy. It is noteworthy that the times of orientational rotational relaxation of fullerenes in solutions are 7—12 ps, and the limiting time of orientational rotational relaxation of

$C_{60}$ under the conditions of free motion is about 3 ps. Therefore, the fast depolarization of the charge-transfer state is not related to the rotational dynamics of fullerenes.

The zero value of $r_{CT}$ can be due to the existence of a local state, into which the complex is transformed immediately after excitation. Since the absorption spectra of $C_{60}(S_1)$ and $C_{60}^-...DEA^+$ overlap, it is easy to suggest that this local state is in the form of a $C_{60}(S_1)...DEA$ complex at the first moment the excitation is localized on the fullerene molecule. Then a charge-transfer state is formed due to nonradiative transition related to electron transfer in the complex:

$$C_{60}(S_1)...DEA \xrightarrow{k_{et}} C_{60}^-...DEA^+. \qquad (24)$$

Therefore, the polarization is equal to zero for $C_{60}^-...DEA^+$ if the electron-electron interaction ($V$) of $C_{60}(S_1)...DEA$ with $C_{60}^-...DEA^+$ has an isotropic character. According to modern concepts, in the general case, the $V$ value is a tensor.[19] For this particular case, it can be expected that the high symmetry of the $C_{60}$ molecule causes the electron transfer in the complex to be isotropic and the loss of polarization in the charge-transfer state. Analysis of the time dependence of the diffraction signal using Eq. (12) assumes the existence of a nonzero concentration of RIP already at the initial delay times, immediately after the excitation pulse. Therefore, electron transfer *via* reaction (24) must be faster than the resolution time of the experiment, *i.e.*, $(k_{et})^{-1} \le 1$ ps.

Thus, the results obtained allow us to draw the following conclusions.

1. In experiments with picosecond laser-induced gratings, the kinetics of the diffraction signal $I_d$ during excitation with light at $\lambda_{exc}$ = 528 nm and probing at the wavelength of 1055 nm in the $C_{60}$—DEA—PhCl system are determined by the singlet-excited states $C_{60}(S_1)$ and RIP $C_{60}^-...DEA^+$ and by the dynamics of quenching of the excited $C_{60}(S_1)$ by diethylaniline.

2. The determined relaxation times of RIP differ for low and high concentrations of DEA in the solution. The dependence of the relaxation time of RIP on the content of DEA is explained by the fact that tight and solvent-separated RIP with different relaxation times exist in the solution.

3. Anisotropy of the diffraction signal $I_d$ is caused by unbound $C_{60}$. The anisotropy of the state of the tight RIP within the experimental error is equal to zero, which is related to the fast isotropic transfer of an electron from the local excited state in the complex to form the radical ion state.

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