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# Photoinduced singlet and triplet energy transfer in fullerene–oligothiophene–fullerene triads

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## Abstract

Photophysical properties of fullerene–oligothiophene–fullerene (C<sub>60</sub>–*n*T–C<sub>60</sub>) triads with *n* = 3, 6, or 9 thiophene units have been investigated using photoinduced absorption (PIA) and (time-resolved) fluorescence spectroscopy in toluene and compared to mixtures of oligothiophenes (*n*T) with *N*-methylfulleropyrrolidine (MP-C<sub>60</sub>). Photoexcitation of the oligothiophene moiety of the C<sub>60</sub>–*n*T–C<sub>60</sub> triads in toluene results in a very fast singlet-energy transfer reaction to the fullerene moiety with rates varying between 10<sup>12</sup> and 10<sup>13</sup> s<sup>−1</sup>. In the mixtures, an *inter*-molecular triplet-energy transfer reaction occurs. The preference for *intra*- and *inter*-molecular energy instead of electron transfer reactions in these triads in toluene is in full agreement with predictions that can be made for the change in free energy upon charge separation. © 2001 Elsevier Science B.V. All rights reserved.

**Keywords:** Polythiophene and fullerene derivatives; Photoinduced absorption spectroscopy; Photoluminescence; Energy and electron transfer

## 1. Introduction

Composite films consisting of conjugated polymers and fullerene derivatives are being considered for solid state polymer photovoltaic cells [1,2]. The underlying photophysical process is a very fast (<1 ps) [3] forward photoinduced electron transfer reaction from the conjugated polymer as a donor to the fullerene as an electron acceptor, while the recombination of photogenerated electrons and holes is slow and extends into the millisecond time domain. Although the attention has mainly been focused on electron transfer reactions, the possibility of an energy transfer reaction has received less attention.

Discrimination between photoinduced electron and energy transfer in solution is governed by the change in free energy change for charge separation ( $\Delta G_{cs}$ ) given by the Weller equation, which includes terms for charge separation and solvation of the radicals ions in addition to the energies for oxidation, reduction, and excitation [4]:

$$\Delta G_{cs} = e(E_{ox}^D - E_{red}^A) - E_{00} - \frac{e^2}{4\pi\epsilon_0\epsilon_s R_{cc}} - \frac{e^2}{8\pi\epsilon_0} \left( \frac{1}{r^+} + \frac{1}{r^-} \right) \left( \frac{1}{\epsilon_{ref}} - \frac{1}{\epsilon_s} \right) \quad (1)$$

In this equation,  $E_{ox}^D$  and  $E_{red}^A$  are the oxidation and reduction potentials of the donor and acceptor in a solvent with relative permittivity  $\epsilon_{ref}$ ;  $E_{00}$  the energy of the excited state from which electron transfer occurs;  $R_{cc}$  the center-to-center distance of the positive and negative charges in the charge separated state;  $r^+$  and  $r^-$  are the radii of the positive and negative ions;  $\epsilon_s$  is the relative permittivity of the solvent; and  $-e$  and  $\epsilon_0$  are the electron charge and the vacuum permittivity, respectively. The Weller equation shows that in an apolar medium such as a toluene solution, energy transfer might be favored over electron transfer as a result of decreased screening of the photogenerated charges.

Various reports exist on the synthesis and photophysical properties of molecular dyads and triads involving covalent bonds of organic donors to fullerenes (for recent reviews see [5–7]). Oligothiophenes covalently bound to fullerenes, however, have received limited attention [8,9]. Here, we describe the photoexcitations formed in three novel molecular triads of conjugated oligothiophenes covalently linked to fullerenes, i.e. C<sub>60</sub>–*n*T–C<sub>60</sub> (*n* = 3, 6, and 9) (Fig. 1), in toluene solution using (time-resolved) fluorescence and photoinduced absorption (PIA) spectroscopy and compare the results to those of mixtures of the individual *n*T oligothiophenes with *N*-methylfulleropyrrolidine (MP-C<sub>60</sub>). We demonstrate that *intra*-molecular singlet-energy transfer reactions occur upon photoexcitation of the C<sub>60</sub>–*n*T–C<sub>60</sub>

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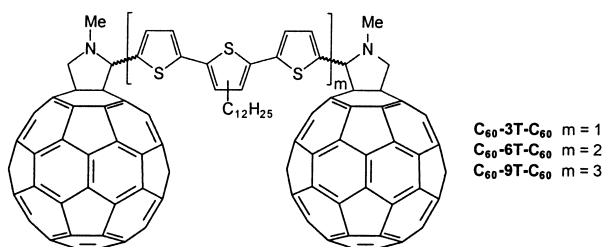


Fig. 1.  $C_{60}$ - $n$ T- $C_{60}$  fullerene-oligothiophene-fullerene triads.

triads on a very short time scale. The energy transfer can also take place *inter*-molecularly, but then proceeds from the triplet state. Although *inter*-molecular energy transfer from the oligothiophene excited singlet state is energetically possible in  $n$ T/MP- $C_{60}$  mixtures, it is less likely to occur because such transfer is limited by diffusion and the sub-nanosecond lifetime of the singlet-excited state. The discrimination between the energy and electron transfer processes strongly depends on the permittivity of the medium and we show that the energy transfer is consistent with the values of  $\Delta G_{cs}$ , obtained from the Weller equation.

## 2. Results and discussion

The absorption spectra of the  $C_{60}$ - $n$ T- $C_{60}$  triads closely correspond to a superposition of the individual spectra of  $n$ T and MP- $C_{60}$ . Hence, electron transfer between donor and acceptor does not occur in the ground state. At 600 nm, the absorption of the  $n$ T oligomers is negligible and, therefore, this wavelength can be used to selectively excite the fullerene moiety. For  $C_{60}$ -6T- $C_{60}$  and  $C_{60}$ -9T- $C_{60}$ , light of 430–470 nm will result in almost selective excitation of the oligothiophene moiety, but selective excitation of the terthiophene unit in  $C_{60}$ -3T- $C_{60}$  is not possible.

### 2.1. Photoexcitation of $n$ T and MP- $C_{60}$

Photoexcitation of the  $n$ T oligothiophenes in toluene solution results in a transient singlet excited state  $n$ T( $S_1$ ) which decays radiatively (Table 1) and non-radiatively to the ground state and via intersystem crossing to the  $n$ T( $T_1$ ) triplet state [10,11]. In solution, the quantum yields for fluorescence are high. The singlet excited state lifetime  $\tau(nT)$  of the  $n$ T oligomers determined in toluene solution decreases with conjugation length (Table 1). The triplet state

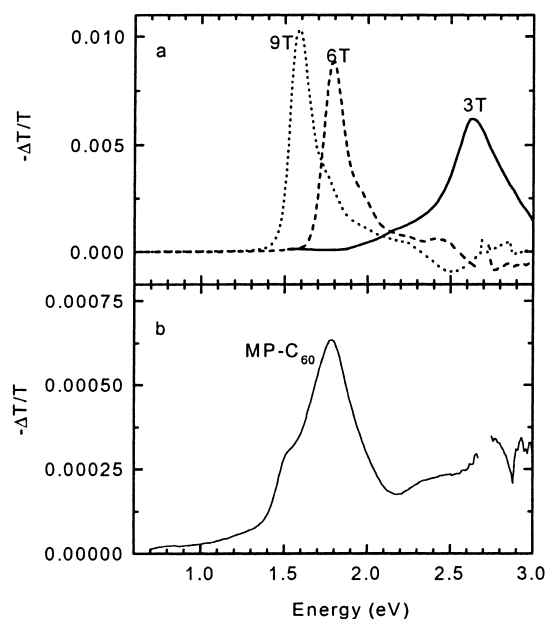


Fig. 2. (a) PIA spectra of oligothiophenes  $n$ T ( $n = 3, 6, 9$ ) in toluene solution ( $2.3 \times 10^{-4}$  M) at 295 K. Recorded with 50 mW excitation at 351.1 and 363.8 nm for 3T and 457.9 nm for 6T and 9T. (b) PIA spectra of MP- $C_{60}$  in toluene ( $4.6 \times 10^{-4}$  M) at 295 K, recorded with excitation at 457.9 nm.

absorption spectra of 3T, 6T, and 9T have been recorded using PIA spectroscopy (Fig. 2) in toluene. The spectra invariably exhibit a  $T_n \leftarrow T_1$  transition, which shifts to lower energy with increasing chain length. The lifetime of the  $n$ T( $T_1$ ) triplet state decreases with chain length from  $\sim 400$   $\mu$ s for 3T to about 100  $\mu$ s for 9T (Table 1). The triplet state energies of  $\alpha$ -oligothiophenes have recently been determined [12,13]. For 3T the triplet energy is 1.92–1.93 eV. From the linear relation of the triplet energy as a function of the reciprocal  $n$  [13], it is possible to estimate the triplet energy levels for 6T ( $\sim 1.64$  eV) and 9T ( $\sim 1.55$  eV).

Photoexcitation of MP- $C_{60}$  in toluene results in a weak fluorescence at 1.74 eV and a long-lived triplet state. The fluorescence quantum yield of MP- $C_{60}$  is  $6 \times 10^{-4}$  and the triplet state is formed with a quantum yield near unity [14,15]. The lifetime of the MP- $C_{60}$ ( $S_1$ ) state determined from time-resolved fluorescence is 1.45 ns (Table 1), close to the value of 1.28 ns previously reported [14,15]. The PIA spectrum of the MP- $C_{60}$ ( $T_1$ ) state exhibits a peak at 1.78 eV with a characteristic shoulder at 1.54 eV (Fig. 2). Identical PIA spectra have been obtained when exciting at 363.8,

Table 1  
Spectroscopic data of  $n$ T oligomers and MP- $C_{60}$  in toluene

	$S_1 \leftarrow S_0$ (eV)	$S_1 \rightarrow S_0$ (eV)	$\tau(S_1)$ (ns)	$T_n \leftarrow T_1$ (eV)	$\tau(T_1)$ ( $\mu$ s)
3T	3.56	3.00	0.81	2.63	410
6T	2.92	2.42	0.78	1.78	145
9T	2.74	2.26	0.65	1.59	93
MP- $C_{60}$	1.76	1.74	1.45	1.78	240

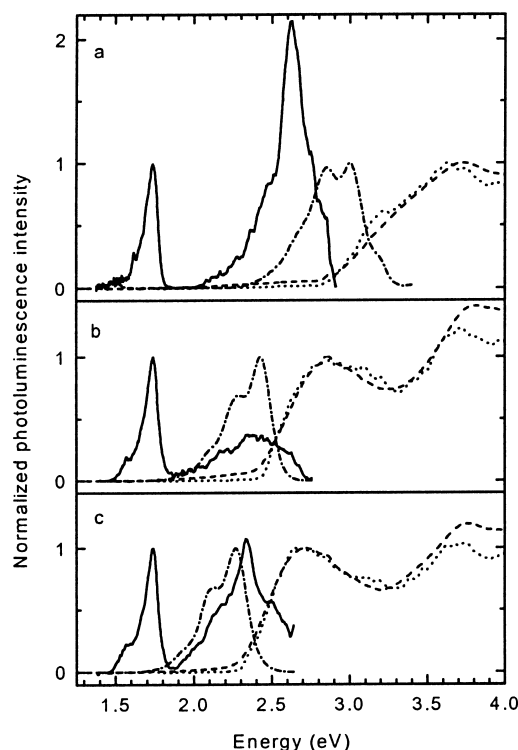


Fig. 3. Normalized fluorescence (solid lines), absorption (dashed lines), and excitation (dotted lines) spectra of the 715 nm (1.73 eV) emission of  $C_{60}$ - $nT$ - $C_{60}$  in toluene solution for (a)  $n = 3$ ; (b)  $n = 6$ ; (c)  $n = 9$ . Residual Raman bands are clearly present at 2.62 eV for  $n = 3$ . For comparison, the normalized fluorescence spectra of the  $nT$  oligomers are included (dashed-dotted lines). The concentration was adjusted in each experiment to give an optical density of 0.1 at the excitation wavelength.

457.9, or 600 nm. The lifetime of the  $MP-C_{60}(T_1)$  state in solution at 295 K is about 200  $\mu s$  (Table 1). The triplet energy level of  $MP-C_{60}$  has been determined from phosphorescence to be 1.50 eV [14,15].

## 2.2. Photoexcitation of $C_{60}$ - $nT$ - $C_{60}$ and $nT/MP-C_{60}$ mixtures

In toluene solutions, the fluorescence of the oligothiophene moieties of the  $C_{60}$ - $nT$ - $C_{60}$  triads is strongly quenched with respect to the emission of the unsubstituted  $nT$  oligomers (Fig. 3). Apart from some residual oligothiophene emission, the fluorescence spectra show a character-

istic band at 1.73 eV, which results from emission from the  $MP-C_{60}(S_1)$  state. The excitation spectra of the fullerene emission coincide with the absorption spectra of the  $C_{60}$ - $nT$ - $C_{60}$  triads. The fullerene fluorescence quantum yield of the  $C_{60}$ - $nT$ - $C_{60}$  triads in toluene is nearly identical to that of  $MP-C_{60}$ , irrespective of the excitation wavelength. The emission spectra of the  $C_{60}$ - $nT$ - $C_{60}$  triads shown in Fig. 3 were corrected for Raman scattering of toluene, but residual Raman bands are clearly present at 2.62 eV for  $C_{60}$ -3T- $C_{60}$  where an excitation wavelength of 350 nm is used. The quenching factors ( $\phi(nT)/\phi$ ) are given in Table 2. The fluorescence quenching of the oligothiophenes and the simultaneous emission of the  $MP-C_{60}$  moiety are clear characteristics of an efficient *intra*-molecular energy transfer reaction of the  $nT(S_1)$  state to the  $MP-C_{60}(S_1)$  state. The quenching and the fluorescence lifetimes of the pristine  $nT$  oligomers (Table 1) can be used to estimate the rate constants for the energy transfer reaction ( $k_{ET}$ ) via the relation:

$$k_{ET} = \frac{[\phi(nT)/\phi] - 1}{\tau(nT)} \quad (2)$$

The resulting values collected in Table 2 show that the energy transfer process is extremely fast and occurs within about 1 ps for  $C_{60}$ -3T- $C_{60}$  and within about 100 fs for  $C_{60}$ -6T- $C_{60}$  and  $C_{60}$ -9T- $C_{60}$ .

The energy transfer reaction from the  $nT(S_1)$  states which populates the  $MP-C_{60}(S_1)$  state, also affects the PIA spectra of the  $C_{60}$ - $nT$ - $C_{60}$  triads in comparison with parent  $nT$  oligomers. The PIA spectra of  $C_{60}$ -3T- $C_{60}$  and  $C_{60}$ -6T- $C_{60}$  are identical to that of the  $MP-C_{60}(T_1)$  state (Fig. 4a). For both triads the lifetime of the  $MP-C_{60}(T_1)$  state is about 250  $\mu s$ . This result demonstrates that the *intra*-molecular singlet-energy transfer to the fullerene moiety, as inferred from fluorescence quenching, is followed by efficient intersystem crossing to the  $MP-C_{60}(T_1)$  state. In contrast, the PIA spectrum of  $C_{60}$ -9T- $C_{60}$  is significantly different and actually corresponds to a superposition of the  $T_n \leftarrow T_1$  spectra of 9T( $T_1$ ) and  $MP-C_{60}(T_1)$  (Fig. 4a). The concurrent absence of a signal at  $\sim 0.70$  eV shows that the PIA band at  $\sim 1.60$  eV is not associated with  $9T^{\bullet+}$ . We conclude that the triplet energy of 9T is very close to the triplet energy of  $MP-C_{60}$  (1.50 eV). Based on the quenching of the 9T fluorescence in  $C_{60}$ -9T- $C_{60}$  we propose that the 9T( $T_1$ ) state in  $C_{60}$ -9T- $C_{60}$  is formed after initial photoexcitation of 9T via a singlet-energy transfer to  $MP-C_{60}$ , subsequent intersystem

Table 2

Fluorescence quenching ( $\phi(nT)/\phi$ ), rate constant for energy transfer ( $k_{ET}$ ), oxidation potential ( $E_{ox}(nT)$ ) vs. SCE, center-to-center distance ( $R_{cc}$ ), radius of positive ion ( $r^+(nT)$ ), and free energy change for *intra*-molecular ( $\Delta G_{cs}$ ) and *inter*-molecular ( $\Delta G_{cs}^\infty$ ) electron transfer in the  $C_{60}$ - $nT$ - $C_{60}$  triads and  $nT/MP-C_{60}$  mixture relative to the energy of the  $nT(S_1)$ ,  $MP-C_{60}(S_1)$  and  $MP-C_{60}(T_1)$  states

	$\phi(nT)/\phi$	$k_{ET}$ ( $s^{-1}$ )	$E_{ox}(nT)$ (V)	$R_{cc}$ ( $\text{\AA}$ )	$r^+(nT)$ ( $\text{\AA}$ )	$nT(S_1)$	$MP-C_{60}(S_1)$	$MP-C_{60}(T_1)$	
						$\Delta G_{cs}$ (eV)	$\Delta G_{cs}$ (eV)	$\Delta G_{cs}$ (eV)	$\Delta G_{cs}^\infty$ (eV)
3T	700	$8.6 \times 10^{11}$	1.07	10.0	4.03	-0.93	0.28	0.54	1.17
6T	9600	$1.2 \times 10^{13}$	0.84	15.4	5.08	-0.45	0.18	0.44	0.83
9T	5900	$9.1 \times 10^{12}$	0.66	21.1	5.80	-0.39	0.07	0.33	0.61

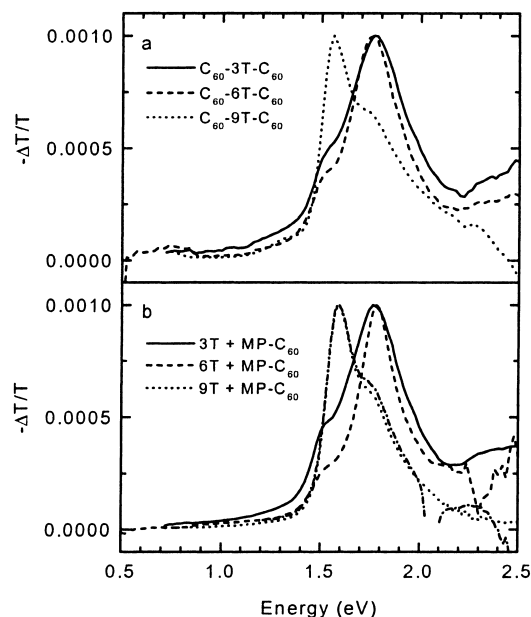


Fig. 4. (a) PIA spectra of  $C_{60}$ - $nT$ - $C_{60}$  ( $n = 3, 6, 9$ ) in toluene ( $2.3 \times 10^{-4}$  M) at 295 K recorded with excitation at 351.1 and 363.8 nm for  $C_{60}$ -3T- $C_{60}$ , and at 457.9 nm for  $C_{60}$ -6T- $C_{60}$  and  $C_{60}$ -9T- $C_{60}$ . (b) PIA spectra of mixtures of  $nT$  ( $2.3 \times 10^{-4}$  M) and MP- $C_{60}$  ( $4.6 \times 10^{-4}$  M) in toluene at 295 K. The dashed-dotted line in graph (b) is for the 9T/MP- $C_{60}$  mixture with excitation at 600 nm.

crossing to the MP- $C_{60}(T_1)$  state, and finally a triplet energy transfer to 9T. Consistent with this model, the same PIA spectrum has been recorded when the fullerene moiety of  $C_{60}$ -9T- $C_{60}$  is selectively excited at 600 nm in toluene solution.

After photoexcitation of the  $nT$  oligomers in toluene solution containing MP- $C_{60}$  (a 1:2 molar proportion is used to mimic the ratio present in the triads), the  $nT(S_1)$  state decays radiatively and non-radiatively to  $nT(S_0)$  and via intersystem crossing to  $nT(T_1)$ . The PIA spectra of the mixtures of 3T or 6T with MP- $C_{60}$  give evidence for *inter*-molecular triplet energy transfer from the  $nT(T_1)$  state to the lower lying MP- $C_{60}(T_1)$  state (Fig. 4b). For 6T, the oligothiophene moiety can be nearly selectively excited at 458 nm and the sole observation of the MP- $C_{60}(T_1)$  spectrum, and hence quenching of the  $nT(T_1)$  transitions, demonstrates the effectiveness of the *inter*-molecular triplet energy transfer reaction. Similar to  $C_{60}$ -9T- $C_{60}$ , we observe only a partial quenching of the 9T( $T_1$ ) state by MP- $C_{60}$  (Fig. 4b). Fig. 4b shows that the same PIA spectra are obtained in mixtures of 9T and MP- $C_{60}$ , regardless whether 9T (at 458 nm) or MP- $C_{60}$  (at 600 nm) is selectively excited. Hence, in both cases the same long-lived photoexcitations are formed eventually.

### 2.3. Energetic considerations for electron transfer

To describe the change in free energy for photoinduced electron transfer reactions in the  $C_{60}$ - $nT$ - $C_{60}$  triads and

$nT$ /MP- $C_{60}$  mixtures using Eq. (1) we measured the oxidation potentials of the  $nT$  oligomers (Table 2) and the first reduction potential of MP- $C_{60}$  ( $E_{\text{red}}(\text{MP-}C_{60}) = -0.67$  V) in dichloromethane ( $\epsilon_{\text{ref}} = 8.93$ ) versus SCE. The *intra*-molecular  $R_{\text{cc}}$  distances have been determined by molecular modeling (Table 2). For *inter*-molecular charge transfer  $R_{\text{cc}}$  was set to infinity. The value for  $r^- = 5.6$  Å for  $C_{60}$  has been calculated by Verhoeven et al. from the density of  $C_{60}$  [14,15]. We estimated the values for  $r^+$  for the oligothiophenes in a similar approach using the X-ray crystallographic data of 3T and 6T [16,17], and extrapolating to 9T (Table 2). With these approximations we come to the values listed in Table 2 for the change in free energy for *inter*- and *intra*-molecular photoinduced charge transfer in toluene. The energies of the neutral and charge-separated states (Tables 1 and 2) are schematically shown in Fig. 5 and reveal a number of interesting effects. First of all, both the MP- $C_{60} S_1$  and  $T_1$  states are energetically situated below a charge-separated state and, hence, *intra*-molecular photoinduced electron transfer from the MP- $C_{60} S_1$  or  $T_1$  state is not expected to occur ( $\Delta G_{\text{cs}} > 0$ ). In contrast, electron transfer from the  $nT(S_1)$  state is energetically possible ( $\Delta G_{\text{cs}} < 0$ ). If a charge-separated state is formed in toluene from the  $nT(S_1)$  state, it must, however, recombine efficiently to yield the lower lying MP- $C_{60}(S_1)$  state because the fullerene fluorescence is not quenched for  $C_{60}$ - $nT$ - $C_{60}$ .

The predictions based on the Weller equation (Fig. 5) are in full agreement with the experimental results, in particular the observed fullerene fluorescence (Fig. 3) and the long-lived triplet states (Fig. 4). According to Eq. (1), both *intra*-

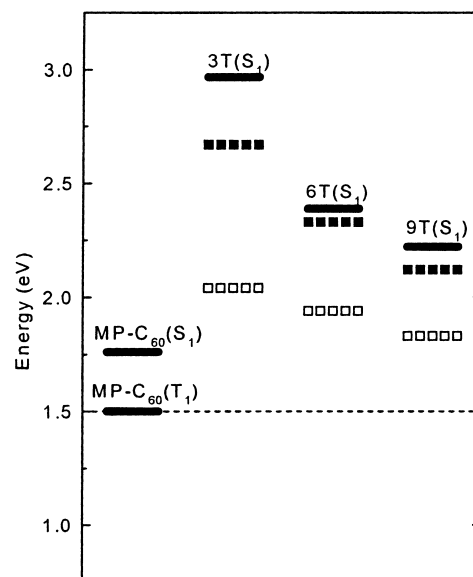


Fig. 5. Excited state energy levels. The levels of the charge separated states for (a) *intra*-molecular charge transfer in  $C_{60}$ - $nT$ - $C_{60}$  triads (open squares) and (b) *inter*-molecular charge transfer in  $nT$ /MP- $C_{60}$  mixtures (solid squares) were determined using Eq. (1).

and *inter*-molecular electron transfer to the fullerene moiety become energetically favored for 6T and 9T in the more polar solvents *o*-dichlorobenzene and benzonitrile, consistent with recent experiments [18].

### 3. Conclusions

Photoexcitation of the oligothiophene moieties of C<sub>60</sub>–*n*T–C<sub>60</sub> triads in toluene solution results in an energy transfer reaction to give a singlet-excited fullerene state which undergoes a near-quantitative intersystem crossing to the MP-C<sub>60</sub>(T<sub>1</sub>) triplet state. From the strong quenching of the *n*T fluorescence we estimate that the energy transfer occurs within ~100 fs for C<sub>60</sub>–6T–C<sub>60</sub> and C<sub>60</sub>–9T–C<sub>60</sub>. Energy transfer may also occur *inter*-molecularly: in toluene triplet energy transfer takes place between *n*T and MP-C<sub>60</sub>. The experimentally observed energy transfer reactions are in full agreement with the energetic predictions based on the Weller equation.

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