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C. R. Chimie 9 (2006) 247-253

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# Photoinduced electron-transfer and magnetic-field effects on the decay rates of photogenerated biradicals in a phenothiazine- $C_{60}$ linked compound with six methylene groups: temperature dependence

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Received 31 August 2004; accepted after revision 4 February 2005

Available online 15 November 2005

## Abstract

Photoinduced electron-transfer reactions and magnetic-field effects (MFEs) on the decay of photogenerated biradical in a phenothiazine- $C_{60}$  linked compound with six methylene groups were examined in benzonitrile at various temperatures (283–343 K). The decay rate constants of the biradicals were dramatically decreased in the lower magnetic fields (0–0.2 T) and increased in the higher magnetic fields (0.2–1.0 T). These reverse phenomena in the MFEs were more clearly observed with the increase of temperature and are explained by the contribution of spin-lattice relaxation mechanism due to anisotropic Zeeman interaction. The temperature dependence on the MFEs supports the above mechanism. *To cite this article: S. Moribe et al., C. R. Chimie 9 (2006)*.

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### Résumé

Effets de transfert d'électrons photogénérés et de champ magnétique sur les vitesses de décroissance des biradicaux photogénérés dans un composé composite phénothiazinc- $C_{60}$  avec six groupes méthyle : dépendance en température. Les réactions de transfert des électrons photogénérés et les effets de champ magnétique (MFEs) sur la décroissance du biradical photogénéré dans un composé de phénothiazinc- $C_{60}$  comprenant six groupements méthyle ont été étudiés dans le benzonitrile à différentes températures (283–343 K). Les constantes de vitesse de décroissance des biradicaux sont considérablement diminuées dans les champs magnétiques faibles (0–0,2 T) et augmentées dans les champs magnétiques plus élevés (0,2–1,0 T). Ces phénomènes inverses des effets de champ magnétique sont plus clairement observés lorsque la température augmente. Ils sont expliqués par la contribution d'un mécanisme de relaxation spin–réseau dû à l'interaction Zeeman anisotrope. La dépendance en température des effets de champ magnétique est en accord avec le mécanisme proposé ci-dessus. *Pour citer cet article : S. Moribe et al., C. R. Chimie 9 (2006)*.

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Keywords: Phenothiazine-C<sub>60</sub>; Photoinduced electron transfer reaction; Magnetic-field effect; Photogenerated biradical

Mots-clés : Phenothiazine-C60 ; Réaction de transfert de charge photoinduite ; Effet de charp magnétique ; Biradical photogénéré

#### 1. Introduction

Photoinduced electron-transfer reactions in donor (D)-C<sub>60</sub> linked systems have been extensively investigated [1]. Though there is a number of papers for photoinduced electron-transfer reactions in D-C<sub>60</sub> linked compounds, most of them focused on intramolecular electron-transfer reactions of singlet excited state of C<sub>60</sub> ( ${}^{1}C_{60}^{*}$ ) [2] in spite of the very high quantum yield of the triplet excited state of C<sub>60</sub> ( ${}^{3}C_{60}^{*}$ ).

Magnetic-field effects (MFEs) on the reaction kinetics or yields of photochemical reactions in the condensed phase have been studied [3–6]. Previously, we have found that the decay rate of photogenerated biradicals in D-acceptor linked systems was remarkably reduced in the presence of magnetic fields up to 1 T [7–13]. In addition, the reverse phenomena in the MFEs, i.e. that the decay rate of photogenerated biradical steeply decreases in the lower region of magnetic fields and then increases in the higher region of magnetic fields, have been reported. The reverse phenomena have been observed only in high magnetic fields above 1 T [14–20].

Recently, we have reported the photoinduced electron-transfer reactions and the MFEs on the decay rates of biradical in D-C60 linked compounds with long methylene groups using zinc-tetraphenylporphyrin (ZnP) or phenothiazine (Ph) as D ( $ZnP(8)C_{60}$  or  $Ph(n)C_{60}$  [9–12]. In both systems, the reverse phenomena in the MFEs have been observed for the first time below 1 T. In the ZnP-C<sub>60</sub> system, the decay rate of the biradical dramatically decreased in lower magnetic fields (0-0.1 T) and gradually increased and finally became almost constant in higher magnetic fields (0.1-1.2 T) [12]. In the Ph– $C_{60}$  systems with methylene groups, similar phenomena in the MFEs at 288 K were observed, but smaller than that of the ZnP-C<sub>60</sub> system [10,11]. These unique phenomena may be explained by the contribution of the anisotropic Zeeman interaction to the spin-lattice relaxation (SLR) of the biradical

Temperature dependences on the lifetimes of triplet biradicals in the presence of 0.6 T have been examined for xanthone-xanthene and benzophenone-diphenylamine bifuntional chain molecules by Tanimoto et al. [21]. These results revealed that the lifetimes of the triplet biradicals were controlled by the spin-relaxation process. In the meantime, the temperature dependences for the triplet biradicals derived from 2-phenylcycloalkanones have been reported by Turro et al. [22]. In that case, the results indicated that the rate-determining step for biradical decay changed from intersystem crossing (ISC) process to chain dynamics process on going from room temperature to lower temperature. The temperature dependence on the lifetime of triplet biradical in xanthone-xanthene bifuntional chain molecules has also been reported by Tanimoto et al. [23]. The results indicated that the unusual temperature dependence on the lifetime of triplet biradical was ascribed to the difference in the temperature dependence on the chain motion and the recombination reaction. Therefore, the measurement of temperature dependence on the MFEs and of the biradical decay is very useful for verifying the mechanism of the MFEs and the decay dynamics of the biradicals. However, temperature dependence on the MFEs in  $Ph(n)C_{60}$  or  $ZnP(8)C_{60}$ has not been reported yet.

In the present paper, we have examined the temperature dependence on the dynamics of the biradical of a Ph–C<sub>60</sub> linked compound with six methylene group, **Ph(6)C<sub>60</sub>** in benzonitrile, in the absence and presence of magnetic field (up to 1 T) and we found the novel MFEs on the decay profiles. The results revealed that the novel MFEs were attributed to the contribution of the anisotropic Zeeman interaction to the SLR of the biradical.

### 2. Experimental

A phenothiazine- $C_{60}$  linked compound with flexible six methylene group (**Ph(6)** $C_{60}$ ) and a reference  $C_{60}$  compound ( $C_{60}$ ref) as shown in Scheme 1 were synthesised according to the previous papers [9,10]. Their structure and purity were confirmed by <sup>1</sup>H-NMR and MALDI-TOF MS spectra, and elemental analysis.



Scheme 1.

10-Methyl phenothiazine (**MePh**) (Wako, extra Pure) as another reference compound was purchased commercially. Benzonitrile (Wako, guaranteed reagent) was used as received. Steady-state absorption, transient absorption spectra, cyclic voltammetry (CV), differential pulse voltammetry (DPV) and MFEs were measured according to the previous papers [9–12]. The temperature of the sample solutions was controlled by circulating the temperature-controlled water through the water jacket equipped with the quartz cell.

#### 3. Results and discussion

# 3.1. Absorption spectra and electrochemical measurements

Absorption spectra of  $Ph(6)C_{60}$  and  $C_{60}ref$  were measured in benzene. The Ph moiety has no absorption band above 400 nm. Two spectra were essentially same in the 400–800 nm region. Similar results were also observed in benzonitrile. These results are in good agreement with those in  $Ph(n)C_{60}$  (n = 8-12) as reported previously in [9–11]. Thus, it is suggested that there are no appreciable electronic interactions between the C<sub>60</sub> and the Ph moieties in  $Ph(6)C_{60}$  in the two solvents.

Redox potentials of **Ph(6)**C<sub>60</sub>, **C**<sub>60</sub>**ref**, and **MePh** by CV and/or DPV in benzonitrile were measured. Two redox couples were observed for **Ph(6)**C<sub>60</sub> ( $E_{1/2}$ (Ph<sup>•+</sup>/Ph) = +0.26 V and  $E_{1/2}$ (C<sub>60</sub>/C<sub>60</sub><sup>•-</sup>) = -1.00 V versus Fc<sup>+</sup>/Fc). These values are roughly the sum of **MePh** ( $E_{1/2}$ (Ph<sup>•+</sup>/Ph) = +0.30 V versus Fc<sup>+</sup>/Fc) and **C**<sub>60</sub>**ref** ( $E_{1/2}$ (C<sub>60</sub>/C<sub>60</sub><sup>•-</sup>) = -0.98 V versus Fc<sup>+</sup>/Fc). These results are in good agreement with the absorption spectra, showing no appreciable electronic interaction between the C<sub>60</sub> and the Ph moieties in the ground state. Table 1

Gibbs free energy changes ( $\Delta G_{CS}(S)$  and  $\Delta G_{CS}(T)$ ) for CS from Ph to  ${}^{1}C_{60}^{*}(S)$  and  ${}^{3}C_{60}^{*}(T)$  and Gibbs free energy change ( $\Delta G_{CR}$ ) for CR in **Ph(6)C**<sub>60</sub>

	$\Delta G_{CS}(S)$	$\Delta G_{CS}(T)$	$\Delta G_{CR}$	
Benzonitrile	-0.50	-0.24	-1.26	
Benzene	0.28	0.54	-2.04	

Thermodynamic data for the present intramolecular electron-transfer reactions in Ph(6)C<sub>60</sub> were evaluated as reported previously [9-12]. In the case of benzonitrile, accurate driving forces  $\Delta G (\Delta G_{CR}, \Delta G_{CS}(S))$  and  $\Delta G_{CS}(T)$ ) for the intramolecular charge recombination (CR) process from  $C_{60}^{-}$  to Ph<sup>•+</sup> and for the intramolecular charge separation (CS) processes from Ph to  ${}^{1}C_{60}^{*}(S)$  and  ${}^{3}C_{60}^{*}(T)$  were calculated using the redox potentials of C<sub>60</sub> and Ph moieties as described above. A centre-to-centre distance ( $R_c$ ) between Ph and C<sub>60</sub> moieties for  $Ph(6)C_{60}$  was estimated to be 14.0 Å by assuming the conformation based on MM2 analysis. The Coulombic term in  $Ph(6)C_{60}$  can be negligible because of the relatively large  $R_c$  and the high dielectric constant ( $\epsilon = 25.2$ ) of benzonitrile. The evaluated Gibbs free energy changes are summarised in Table 1.

In the case of benzene, the free energy changes,  $\Delta G$ ( $\Delta G_{CR}$ ,  $\Delta G_{CS}(S)$  and  $\Delta G_{CS}(T)$ ), were calculated from the Rehm-Weller and Born equations [9–11] by using the redox potentials in benzonitrile for convenience (Table 1). However, it should be noted that the Born equation was tentatively used due to the overestimation of the non-polarity of benzene.

#### 3.2. Transient absorption spectra

Transient absorption spectra in benzene and benzonitrile were measured to elucidate the photoinduced electron-transfer reactions in **Ph(6)C<sub>60</sub>** by nanosecond laser-flash photolysis. The transient absorption spectra of **Ph(6)C<sub>60</sub>** by laser excitation at 532 nm in both solvents are shown in Fig. 1. In benzene, the broad absorption band around 700 nm due to  $T_1-T_n$  absorption of the C<sub>60</sub> moiety was observed (Fig. 1a). No appreciable absorption bands due to Ph<sup>•+</sup> were observed. Therefore, the results indicate that photoinduced electrontransfer reaction dose not occur in benzene. On the other hand, the  $T_1-T_n$  absorption band of the C<sub>60</sub> moiety disappeared and the absorption band due to the Ph<sup>•+</sup> ( $\lambda_{max}$ , 520 nm) [7–11] was clearly observed in benzonitrile, as shown in Fig. 1b. The results indicate that intramo-



Fig. 1. Transient absorption spectra of  $Ph(6)C_{60}$ ( $1.0 \times 10^{-4}$  mol dm<sup>-3</sup>) (**a**) at 1.0 µs in benzene and (**b**) at 0.06 µs in benzonitrile after laser excitation at 288 K.

lecular electron transfer from Ph to  ${}^{1}C_{60}^{*}$  or  ${}^{3}C_{60}^{*}$  occurs, and then the biradical is generated in benzonitrile. Table 1 strongly indicates that the intramolecular electron transfer from Ph to  ${}^{1}C_{60}^{*}$  or  ${}^{3}C_{60}^{*}$  in **Ph(6)C**<sub>60</sub> is thermodynamically favourable in benzonitrile, but unfavourable in benzene. This is in fair consistency with the solvent effect in the transient absorption spectra (Fig. 1). These results in **Ph(6)C**<sub>60</sub> were in good agreement with those in **Ph(n)C**<sub>60</sub> (n = 8-12) [9–11].

# 3.3. Temperature dependence of photoinduced electron-transfer reactions and the decay of photogenerated biradical

Transient absorption spectra of **Ph(6)C**<sub>60</sub> were measured in benzonitrile at 283, 303, 323, and 343 K, respectively. The absorption band due to the Ph<sup>+</sup> at 520 nm was observed at all temperatures as in Fig. 1b.



Fig. 2. Temperature dependence on decay profiles of transient absorption at 520 nm of  $Ph(6)C_{60}$  in benzonitrile at 0, 0.1, and 1.0 T: (a) 283, (b) 343 K.

The decay profiles of transient absorption at 520 nm were shown at 283 K (**a**) and 343 K (**b**) in Fig. 2. The decay curves can be expressed by the following equation:

$$A(t) = A_0 \exp\left(-k_d t\right) + C \tag{1}$$

where A(t) is transient absorption at t (s),  $A_0$  and C are time-independent constants, and  $k_d$  (s<sup>-1</sup>) is the decay rate constant for the biradical. These parameters were calculated by the non-linear least-square method at various magnetic fields and temperatures. The  $k_d$ -values for **Ph(6)C\_{60}** at zero magnetic field were almost identical at all temperatures (Fig. 3). The results indicate that the decay for the biradical in **Ph(6)C\_{60}** at zero magnetic field is independent of temperature. In general,  $k_d$  for the triplet biradical at zero magnetic field is controlled by isotropic hyperfine (hf)-induced ISC ( $k_{isc2}$ ), the back electron transfer from the singlet biradical ( $k_{bet}$ ), and the spin-orbit (SO)-induced ISC



Fig. 3. Temperature dependence of the MFEs on the decay rate constants ( $k_d$ ) for the biradical as evaluated from the transient absorption at 520 nm upon laser excitation of **Ph(6)C<sub>60</sub>** in benzonitrile 283 ( $\odot$ ), 303 ( $\diamond$ ), 323 ( $\triangle$ ), and 343 ( $\square$ ) K; (a) in  $0 \le H \le 0.1$  T and (b) in  $0 \le H \le 1$  T.

processes ( $k_{SOC}$ ), as shown in Scheme 2. On the basis of these observations, the  $k_d$ -value for the triplet biradical in **Ph(6)C**<sub>60</sub> at zero magnetic field is mainly governed by the isotropic hf-induced ISC process, since the  $k_{bet}$  and  $k_{SOC}$  are dependent on temperature, but  $k_{isc2}$  is independent on temperature. This is consistent with the long-lived charge-separated state, in spite of being around the top region in the Marcus theory in **Ph(n)C**<sub>60</sub> (n = 10, 12) [9,10]. The  $k_d$ -value for **Ph(6)C**<sub>60</sub> in zero magnetic field can be estimated to be  $7.5 \times 10^6 \text{ s}^{-1}$  at 288 K. The  $k_d$ -value for **Ph(6)C**<sub>60</sub> is slightly smaller than those for **Ph(n)C**<sub>60</sub> (n = 8, 10, 12) [9–11]. The effect of spacer chain length on the  $k_d$ -



values is very interesting and is probably caused by the variation of triplet–singlet energy separation. This is related to the exchange interaction in the biradical, with the interradical distance as pointed out in the case of donor (Ph or ZnP)-viologen compounds [8,24,25].

# 3.4. MFEs on the decay of biradical at various temperatures

The MFEs on the photoinduced electron-transfer in Ph(6)C<sub>60</sub> were examined in benzonitrile at various temperatures (283-343 K) in Figs. 2 and 3. At lower temperature (283 K), the decay of transient absorption at 520 nm was suppressed in the presence of a low magnetic field (0.1 T), but became slightly faster at 1.0 T as compared with that at 0.1 T (Fig. 2a). With the increase of the magnetic field, the  $k_{d}$ -value decreased steeply at lower magnetic fields (< 0.2 T), and then recovered slightly in the 0.2 T  $\leq H < 0.6$  T region and finally became almost constant in the higher magnetic fields  $(0.6 \text{ T} \le H < 1.0 \text{ T})$ , as shown in Fig. 3 at 283 K. As a result, the reverse phenomenon in the MFEs occurring at 0.2 T was clearly observed. Similar reverse phenomena were also observed in Ph(8)C<sub>60</sub> and Ph(12)C<sub>60</sub> at 288 K in a previous paper [11]. However, these reverse phenomena had not been discussed in detail, because the magnitudes of the reverse phenomena were very small.

In contrast, the reverse phenomena in the MFEs appeared more clearly with increasing temperature in Fig. 3. At 303 K, the magnetic-field dependence of the  $k_d$ -value was similar as that at 283 K and the minimum of  $k_d$ -value (at 0.2 T) was larger than that at 283 K. On the other hand, at 323 and 343 K, the minima of  $k_d$ -values were observed at 0.1 T and showed a lower

field shift as compared with those at 283 and 303 K. In addition, the  $k_d$ -value decreased steeply at lower magnetic fields (< 0.1 T), and then recovered gradually (0.1 T  $\leq H < 1$  T). At 323 and 343 K, the  $k_d$ -values above 0.8 T were larger than that in the absence of magnetic field (0 T) as shown in Figs. 2b and 3, in a similar manner as in the case of **ZnP(8)C<sub>60</sub>** at 288 K [12].

The magnitudes of the MFEs were evaluated by the ratios ( $\Delta_{\text{low}} = k_{\text{d}}(0 \text{ T})/k_{\text{d}}(0.2 \text{ T})$  and  $\Delta_{\text{high}} = k_{\text{d}}(1.0 \text{ T})/k_{\text{d}}(0.2 \text{ T})$  $k_{\rm d}(0.2 \,{\rm T})$ ) in the case of the lower temperatures (283 and 303 K) or  $(\varDelta_{low} = k_d(0 \text{ T})/k_d(0.1 \text{ T}) \text{ and } \varDelta_{high} =$  $k_{\rm d}(1.0 \text{ T})/k_{\rm d}(0.1 \text{ T}))$  in the case of the higher temperatures (323 and 343 K), respectively. The  $\Delta_{low}$ -values at 283, 303, 323 and 343 K were estimated to be 1.84, 1.53, 1.36 and 1.30, respectively. Thus, the  $\Delta_{low}$ -value was reduced by 1.4 times on going from 283 to 343 K. On the other hand, the  $\Delta_{high}$ -values at 283, 303, 323 and 343 K were estimated to be 1.20, 1.21, 1.48 and 1.43, respectively. Thus, the  $\varDelta_{high}$ -value was enhanced by 1.2 times on going from 283 to 343 K. On the basis of these observations, it is concluded that with increasing temperature the magnitudes of MFEs in lower magnetic fields were reduced, while those in higher magnetic fields were enhanced. As the result of the temperature dependence, the reverse phenomena in the MFEs were more clearly observed at high temperatures. In the Ph-viologen systems, similar reverse phenomena of the MFEs have been reported in the higher magnetic fields (> 1 T), but not in the lower magnetic fields (< 1 T) [16]. From the comparison with Ph– $C_{60}$  and Ph-viologen linked compounds, the C<sub>60</sub> moiety in  $Ph(6)C_{60}$  is most likely responsible for the present unusual phenomenon on the MFE [9–12].

The temperature dependences in the MFEs in the lower magnetic fields were clearly observed. Thus the MFEs (< 0.2 T) cannot be explained only by the isotropic hyperfine coupling (hfc) mechanism, since the hf interactions are independent on temperature. In the region of lower magnetic fields (< 0.2 T), MFEs on the  $k_d$  for the triplet biradical can be explained by hfc mechanism and SLR mechanism due to the anisotropic hf and the dipole–dipole (dd) interactions [5,6]. The consideration is in fair agreement with that from a semitheoretical value ( $B_{1/2}$ ) [3,9,11,12].

In general, the MFEs for the biradical in higher magnetic fields can be explained by SLR mechanism due to anisotropic Zeeman interaction or  $\Delta g$  mechanism [3–6].  $\Delta g$  mechanism is dominated by the difference of g-values between the donor and acceptor, and is independent of temperature. On the other hand, SLR mechanism due to anisotropic Zeeman interaction is dominated by the g anisotropy, and is dependent on temperature. Thus, the temperature dependence revealed that the MFEs in the higher magnetic fields are ascribed by SLR mechanism due to anisotropic Zeeman interaction.

The MFEs on the decay of the radical pair between a  $C_{60}$  cluster anion and a pyrene cation have been observed in a micellar system [20]. In addition, MFEs, at 77 K on the decay of biradical have been also reported in a carotenoid-porphyrin-C<sub>60</sub> triad system, but no MFEs were observed at 298 K [26]. Those MFEs are different from the novel MFEs, as described in the donor-C<sub>60</sub> linked compounds including the present case  $(\mathbf{Ph}(\mathbf{6})\mathbf{C}_{\mathbf{60}})$ . The temperature effect strongly supports that the novel MFEs in higher magnetic fields are ascribed to properties of C60 moiety and probably interpreted by the SLR mechanism due to anisotropic Zeeman interaction in donor-C<sub>60</sub> linked systems. The present study provides useful informations for regulating the biradical lifetime in the donor- $C_{60}$  systems in novel molecular spin systems. Further investigations on the Ph-C<sub>60</sub> systems with different chain length and quantitative analysis of temperature dependence on the MFEs are in progress.

### Acknowledgements

The authors are grateful to Mr. H. Horiuchi for the preparation of quartz cells for transient absorption spectral experiments. The authors also thank The Centre of Advanced Instrumental Analysis, Kyushu University, for <sup>1</sup>H-NMR measurements. The present study was financially supported by the grants of the Inamori Foundation and the society for the support of Kyushu University, and by the Grant-in-Aids for Scientific Research: Priority Area of 'Innovative Utilisation of Strong Magnetic Fields' (Area 767, No. 15085203) and 'Molecular Nano Dynamics' (Area 432, No. 17034051), Scientific Research (C) (No. 17550131), and 21st Century COE Program 'Function Innovation of Molecular Informatics' from MEXT of the Japan.

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