

Energies of charge transfer for C₆₀ and C₇₀ complexes in solutions and in the solid state

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Abstract

Complexes of the fullerenes C₆₀ and C₇₀ with various donors have been studied in solution and in the solid state by electronic absorption spectroscopy. Charge transfer (CT) bands are found in the 620–1300 nm range. It is shown that in solution the CT energies ($h\nu_{CT}$) for C₆₀ and C₇₀ complexes are similar and almost independent of solvent polarity. The dependence of the $h\nu_{CT}$ energies on donor ionization energy were found for C₆₀ complexes with substituted tetrathiafulvalenes in the solid state and in toluene. In the solid state the $h\nu_{CT}$ energies are ~ 0.15 – 0.20 eV lower than those in the solution. The linear dependence of $h\nu_{CT}$ on donor ionization energy for the C₆₀ complexes lie ~ 0.6 – 0.7 eV higher than those for the TCNE ones. © 1999 Elsevier Science B.V. All rights reserved.

1. Introduction

Recently many donor–acceptor complexes involving fullerenes have been prepared [1–13]. Electronic absorption spectra of some of these show charge transfer (CT) bands in the solid state [2–11] or in solution [14–17], so that these compounds may be considered as charge transfer complexes (CTC).

Charge transfer in C₆₀-conducting polymer composites and fullerene-based compounds is currently of great interest since these materials can be utilised in xerography, energy phototransducers and molecular switchers [18]. Because of this, the derivation of general dependences of the CT energy in fullerene compounds on different factors is important.

Charge transfer in fullerene compounds has been studied mainly in solution [14–17]. The $h\nu_{CT}$ energies for C₆₀ complexes with substituted anilines [14], substituted naphthalenes [15], aromatic hydrocarbons [16], and tertiary amines [17] have been estimated and linear dependencies of $h\nu_{CT}$ energies on donor ionization energy were observed in the 7.2–8.2 eV ionization energy range.

There is much less data for charge transfer in C₆₀ compounds in the solid state. The $h\nu_{CT}$ energies have been determined by electronic spectroscopy for several C₆₀ complexes [2–5], and the dependence of the $h\nu_{CT}$ energies on the redox potentials of the donors considered [3].

This Letter reports studies of C₆₀ and C₇₀ complexes with donors of the tetrathiafulvalene family and some other families in solution and in the solid state by electronic spectroscopy. The study of a large

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number of complexes with a series of identical donors allows the dependence of $h\nu_{CT}$ energies on the ionization energy of the tetrathiafulvalenes to be derived and the influence of solvent polarity on the $h\nu_{CT}$ energies, and the changes in $h\nu_{CT}$ energies on going from fullerene complexes in solution to those in the solid state and from C_{60} complexes to C_{70} ones to be established. A comparison of the derived dependences with those for TCNE complexes has also been carried out.

2. Experimental

Fullerene C_{60} of 99.9% purity and C_{70} of 98% purity were used. Toluene was distilled over Na/benzophenone under argon and benzonitrile (Aldrich, HPLC grade) was distilled in vacuum under

argon. These solvents do not absorb in the range under study. Donor compounds and their vertical ionization potentials (I_p) are presented in Table 1.

The C_{60} and C_{70} complexes in the solid state were prepared as crystals, by a slow evaporation of a solution containing the donor and the fullerene under argon. CS_2 , benzene, toluene, and pyridine (Py) were used as solvents [6–11]. The composition of the complexes was determined by elemental and thermogravimetric analyses and the complexes were characterised by IR, electronic absorption, ESR- and X-ray photoelectron spectroscopies [6–11].

The ESR signals of C_{60} or C_{70} were not detected in the complexes studied [6–9,11] and the shifts of the $F_{lu}(4) C_{60}$ mode at 1429 cm^{-1} in their IR spectra do not exceed 2 cm^{-1} [10]. Thus these are complexes with a neutral ground state.

Electronic absorption spectra were measured with a Perkin–Elmer Lambda 19 UV–VIS–NIR spectro-

Table 1

Donor compounds and their ionisation potentials, the position of the maxima of CTBs in the absorption spectra (λ , nm) of the C_{60} and C_{70} complexes in the solid state and the corresponding $h\nu_{CT}$ energies

Donor ^a	I_p (eV)	Complex	N	λ (nm)	$h\nu_{CT}$ (eV)	Reference
TPDP	5.80 [19]	$D(C_{60})_2(CS_2)_4$	1	1240	0.98	[8]
OMTTF	6.3 [20]	$D \cdot C_{60} \cdot C_6H_6$	2	1040	1.20	[3,10]
		$D \cdot C_{60} \cdot C_5H_5N$	3	980	1.26	[11]
		$D \cdot C_{60}$	4	974	1.27	[12]
TMPDA	6.75 [21]	$(D)_2 \cdot C_{60} \cdot C_6H_6$	—	940	1.32	[10]
DMDP-TTF	—	$(D)_2 \cdot C_{70} \cdot C_6H_6$	—	1000	1.24	[11]
		$D \cdot C_{60} \cdot C_6H_6$	—	935	1.32	[9,10]
		$(D)_2 C_{60} \cdot CS_2$	—	900	1.35	[7,10]
EDY-BEDT-DT	—	$D \cdot C_{60} \cdot C_6H_6$	—	920	1.38	[7,10]
EDT-TTF	—	$D \cdot C_{60} \cdot C_6H_6$	—	920	1.38	[7,10]
		$D \cdot C_{60} \cdot C_6H_6$	—	895	1.38	[10]
		$(D)_2 C_{60}(CS_2)_3$	—	900	1.39	[7,9,10]
BEDO-TTF	6.46 [22]	$(D)_2 C_{60}(C_5H_5N)_2$	6	820	1.51	[11]
DP-TTF	—	$(D)_2 C_{60}$	7	790	1.57	[2]
		$D \cdot C_{70} \cdot CS_2$	—	860	1.38	[11]
		$D \cdot C_{60} \cdot C_6H_6$	8	735	1.69	[6]
TMDTDM-TTF	—	$D \cdot C_{60} \cdot C_5H_5N$	9	750	1.64	[6]
		$D \cdot C_{60} \cdot C_7H_8$	—	725	1.71	[11]
		$D \cdot C_{60} \cdot CS_2$	—	620	2.00	[9]
BEDT-TTF	6.70 [22]					
DBTTF	6.81 [23]					
OOT-TTF	—					
BTX	—					

TPDP-3,3',5,5'-3,3',5,5'-tetraphenyldipyranilidene; OMTTF-octamethylenetetrathiafulvalene (TTF); TMPDA-*N,N,N',N'*-tetramethyl-*p*-phenylenediamine; DMDP-TTF-*trans*-4,4'-dimethyl-5,5'-diphenyl-TTF; EDT-BEDT-DT-2, 2'-ethanedithiilidene-bis(4,5-ethylene-1,3-dithiol); EDT-TTF-ethylenedithio-TTF; BEDO-TTF-bis(ethylenedioxy)-TTF; DP-TTF-*trans*-4,4'-diphenyl-TTF; TMDTDM-TTF-tetramethylenedithio-4,5-dimethyl-TTF; BEDT-TTF-bis(ethylenedithio)-TTF; DBTTF-dibenzo-TTF; OOT-TTF-bis(oxydimethylenedithio)-TTF, BTX-*trans*-9,9'-bis(telluraxanthenyl).

photometer in 220–3000 nm range. The solid samples were prepared in KBr pellets in a ratio of 1:2000. Charge transfer bands (CTB) were observed in the 620–1300 nm range where the fullerenes and donors exhibit no absorptions. To determine more precisely the position of the maximum of these bands, we subtracted a spectrum of the fullerenes normalised with respect to the absorption at 500 or 600 nm from that of a complex. The intensity of CTBs constitutes 0.03–0.20 units of optical density and exceeds the noise level in this spectral range (0.01).

The spectra in toluene and benzonitrile were measured in the 400–1600 nm range at 23°C in a 0.5 cm thick hermetic quartz cell (1 ml volume). The spectra of the fullerene solutions (0.5 mg/ml) and the spectra of the same solution containing the donor at different concentrations were measured. CTBs were identified by a subtraction of the spectrum of the starting fullerene solution from that of the same solution containing the donor at a concentration of ~ 7–8 mg/ml.

The complexes in solution were essentially found to have a 1:1 composition from the optical density at the maximum of CTBs which depends linearly on the donor concentration.

3. Results and discussion

3.1. The observation of charge transfer bands

To estimate the $h\nu_{CT}$ energies in fullerene complexes in solution we used tetrathiafulvalenes with known ionization potentials DBTTF, TMTTF, and

Table 2

The $h\nu_{CT}$ energies for C_{60} and C_{70} complexes in toluene and benzonitrile

Donor	I_p (eV)	$h\nu_{CT}$ (eV)			
		Toluene		Benzonitrile	
		C_{60}	C_{70}	C_{60}	C_{70}
OMTTF	6.3 [20]	1.44	1.43	1.43	1.43
TMTTF ^a	6.39 [24]	1.48	1.44	1.47	–
DBTTF	6.81 [23]	1.85	1.82	1.85	–

^aTMTTF – tetramethyltetrathiafulvalene.

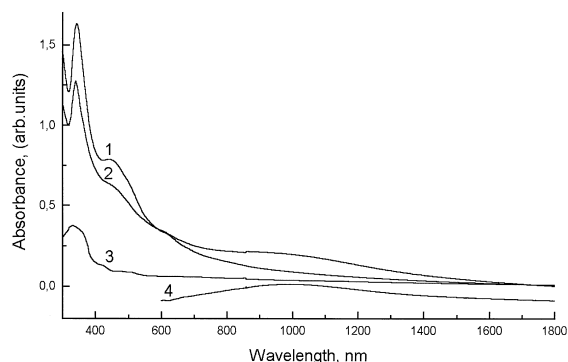


Fig. 1. Absorption spectra of C_{60} (1), OMTTF· C_{60} ·Py (2), and OMTTF (3) in KBr pellets and a CTB (4).

OMTTF (Table 2). The absorption spectra of C_{60} in toluene in the presence of OMTTF show an increase in the absorption intensity at 800–1000 nm as the OMTTF concentration increases. This absorption is absent in the spectra of the initial solutions of OMTTF and C_{60} . The appearance of new absorption bands was also observed in the presence of DBTTF and TMTTF in a toluene solution of C_{60} . The new bands can be attributed to CTBs since their position depends on the ionization potential of the donors and their intensity depends on the donor concentration.

To estimate the effect of solvent polarity on the $h\nu_{CT}$ energy we measured analogous absorption spectra of C_{60} and C_{70} complexes with the same donors in benzonitrile which is more polar than toluene. These data are also presented in Table 2. It is seen that the $h\nu_{CT}$ energies are the same within experimental accuracy for C_{60} and C_{70} complexes in toluene and benzonitrile. Therefore, the $h\nu_{CT}$ energies are almost independent of solvent polarity for fullerene complexes in contrast to other π -type acceptors [25].

To obtain the $h\nu_{CT}$ energies for fullerene complexes in the solid state we measured their electronic absorption spectra in KBr pellets (Table 1). Fig. 1 shows the absorption spectrum of the OMTTF· C_{60} ·Py complex (2), which contains a new broad absorption band with the maximum at ~ 1000 nm (4). This band is absent in the spectra of OMTTF (3) and C_{60} (1) and can be attributed to a CTB.

The CTBs identified from the spectra of various C_{60} and C_{70} complexes in the solid state are shown in Fig. 2. These bands are wide, nonstructural, asym-

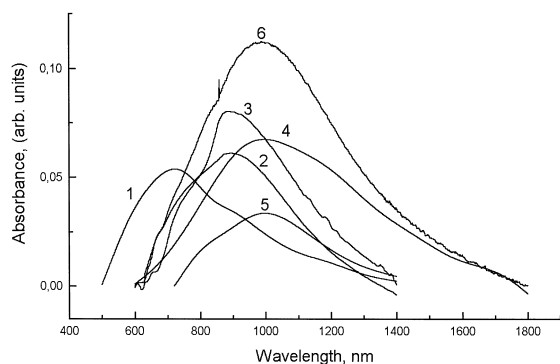
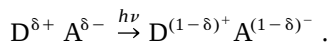


Fig. 2. CTBs identified in the absorption spectra of C_{60} and C_{70} complexes in the 400–1800 nm range: OOT-TTF· C_{60} · C_7H_8 (1), DP-TTF· C_{60} · C_6H_6 (2), BEDT-TTF· C_{70} · CS_2 (3), TMPDA· C_{60} (4), (DMDP-TTF) $_2$ C_{70} · C_6H_6 (5), and OMTTF· C_{60} ·Py (6).

metric, and their positions depend on the ionization potentials of the donors which is characteristic of CTBs. The $h\nu_{CT}$ energies corresponding to the maximum of CTBs for the C_{60} and C_{70} complexes are presented in Table 1.

3.2. Dependences of $h\nu_{CT}$ energies on ionization potential of the donors

For complexes with a neutral ground state, a CTB corresponds to a transfer of an electron from a donor to an acceptor molecule with the absorption of a quantum:



For neutral complexes formed by one acceptor with a series of donors, the dependence of or ionization potential $h\nu_{CT}$ in solution is usually close to linear and is described by the following expression:

$$h\nu_{CT} = \alpha \cdot (I_p - E_A) - E_C \quad (1)$$

where α is a constant, E_A and I_p are the vertical electron affinity and ionization potential, respectively, and E_C is the energy of electrostatic interaction in the $D^+-C_{60}^-$ pair in the excited state.

The $h\nu_{CT}$ – I_p dependences obtained in this work for C_{60} complexes with tetrathiafulvalenes in solution and in the solid state are presented in Fig. 3. In both cases the $h\nu_{CT}$ energies show a practically linear dependence on the I_p of the tetrathiafulvalenes. The $h\nu_{CT}$ – I_p dependence for C_{60} complexes with OMTTF, BEDT-TTF, BEDO-TTF, and

DBTTF in the solid state may be approximated by the following expression:

$$h\nu_{CT} = 0.82 \cdot I_p - 3.93 \text{ eV} \quad (R 0.985, SD 0.04) \quad (2)$$

The $h\nu_{CT}$ energies for C_{60} complexes with donors of other classes, namely, TPDP and TMPDA do not fit with expression (2). This is obviously associated with the different E_C values for C_{60} complexes with donors of various types due to the difference in the crystalline packing and average distances between the donor and C_{60} molecules in the crystal.

The decrease in $h\nu_{CT}$ energies (Fig. 3) by ~ 0.15 – 0.20 eV was observed on going from the solution to the solid state for C_{60} complexes with DBTTF and OMTTF. A similar decrease of the $h\nu_{CT}$ energies (~ 0.2 eV) was observed for TCNE complexes [26]. This decrease is obviously associated with the closer distances between the donor and acceptor molecules in the crystals compared with in solution and the additional stabilisation of the excited ionic state in the crystal due to the resonance stabilisation energy.

The $h\nu_{CT}$ energies for C_{60} and C_{70} complexes with the same donors (DBTTF, TMTTF, and OMTTF) are similar in solution (Table 2). At the same time the $h\nu_{CT}$ energies for C_{70} complexes in the solid state lie ~ 0.08 – 0.19 eV lower than those for C_{60} complexes with the same donors (BEDT-TTF and DMDP-TTF) (Table 1).

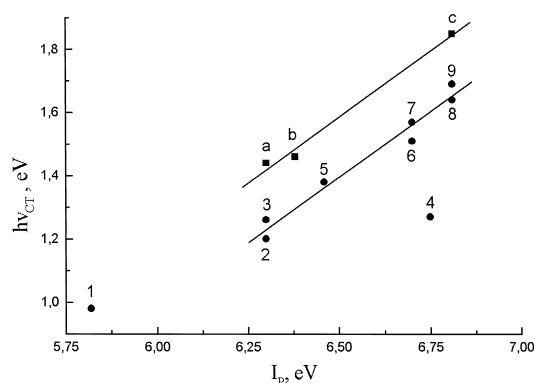


Fig. 3. Dependence of $h\nu_{CT}$ energies on the I_p of donors: (a,b,c) complexes of C_{60} with OMTTF (a), TMTTF (b), and DBTTF (c) in toluene. (1–9) Complexes of C_{60} in the solid state. The numeration is according to Table 1.

The combination of the $h\nu_{CT}-I_p$ dependence obtained by us for C_{60} complexes with tetrathiafulvalenes in toluene with those obtained previously for C_{60} complexes with substituted anilines and naphthalenes in toluene [14,15] allows one to derive a general $h\nu_{CT}-I_p$ dependence (Fig. 4) for C_{60} complexes with different donors covering a wide range of I_p (6.3–8.2 eV). The resulting relationship is linear and is described by the expression:

$$h\nu_{CT} = 0.90 \cdot I_p - 4.28 \text{ eV} \quad (R 0.995, SD 0.06). \quad (3)$$

The linearity of this relationship enables the estimation of unknown I_p s of other donors from the $h\nu_{CT}$ energy of the C_{60} complex in toluene.

The $h\nu_{CT}-I_p$ dependences for C_{60} complexes in solution and in the solid state (Fig. 5) lines 1 and 2, respectively) were compared with the similar dependencies for TCNE complexes [26] with aromatic hydrocarbons (Fig. 5, lines 3 and 4, respectively). For TCNE complexes in dichloromethane, this dependence is approximated by the following expression [26]:

$$h\nu_{CT} = 0.87 \cdot I_p - 4.86 \text{ eV}. \quad (4)$$

It is seen that both in solution and in the solid state the $h\nu_{CT}-I_p$ dependencies lie $\sim 0.6\text{--}0.7$ eV higher for C_{60} complexes than for TCNE ones.

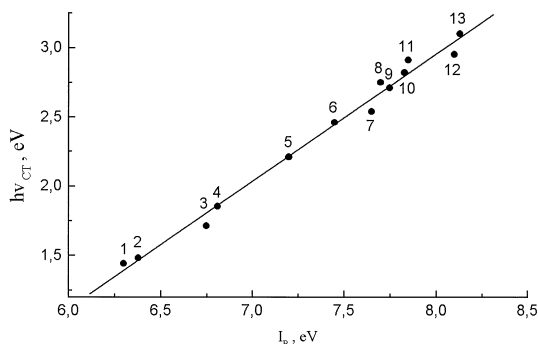


Fig. 4. Dependence of $h\nu_{CT}$ energies on the I_p of tetrathiafulvalenes (obtained in this work), tertiary amines [17], substituted anilines [14], and naphthalenes [15] for complexes of C_{60} in toluene: OMTTF (1), TMTTF (2), TMPDA [17] (3), DBTTF (4), *N,N*-diethylaniline [14] (5), *N,N*-dimethylaniline [14] (6), *N*-methylaniline [14] (7), 1-methoxynaphthalene [15] (8), 2,6-dimethylaniline [14] (9), *ortho*-toluidine [14] (10), 1-methylnaphthalene [15] (11), aniline [14] (12), and 1-chloronaphthalene [15] (13).

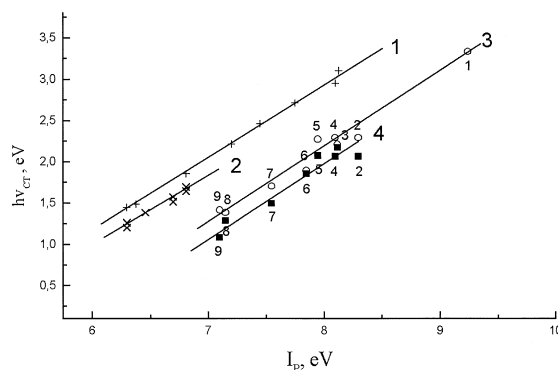


Fig. 5. Linear dependences of $h\nu_{CT}$ energies on the I_p of donors for C_{60} complexes in toluene (1) and in the solid state (2); for TCNE complexes in dichloromethane (3) and in the solid state (4) [26]: benzene (1), pentamethylbenzene (2), phenanthrene (3), naphthalene (4), hexamethylbenzene (5), acenaphthene (6), pyrene (7), perylene (8), and anthanthrene (9).

The values of the $h\nu_{CT}$ energies in the complexes in solution have been used for the calculation of vertical E_A of the acceptors according to Eq. (1). This is possible as the E_C values for the most of the organic π -acceptors are similar ($E_C = 2.94 \pm 0.11$ eV) [27]. The E_A values for more than 150 acceptors have been obtained by this method. For example, the value of E_A of TCNE calculated from the $h\nu_{CT}$ energies is equal to 2.77 ± 0.11 eV [27]. We used this method for the estimation of the vertical E_A of fullerenes.

The difference in $h\nu_{CT}$ energies for C_{60} and TCNE complexes derived from Eqs. (3) and (4), respectively, is:

$$h\nu_{CT}(C_{60}) - h\nu_{CT}(TCNE) = 0.03 \cdot I_p + 0.58 \text{ eV}. \quad (5)$$

Eq. (1) yields:

$$\begin{aligned} h\nu_{CT}(C_{60}) - h\nu_{CT}(TCNE) &= [\alpha(I_p - E_A(C_{60})) - E_C(D-C_{60})] \\ &\quad - [\alpha_1(I_p - E_A(TCNE)) - E_C(D-TCNE)]. \end{aligned} \quad (6)$$

The combination of Eqs. (5) and (6) yields:

$$\begin{aligned} [0.87 \cdot E_A(TCNE) - 0.90 \cdot E_A(C_{60})] \\ - [E_C(D-TCNE) - E_C(D-C_{60})] = 0.58 \text{ eV}. \end{aligned} \quad (7)$$

Assuming that $E_C(D-TCNE) = E_C(D-C_{60})$ in Eq. (7) is valid and that $E_A = 2.77 \pm 0.11$ eV for TCNE, the vertical E_A for C_{60} is derived to be equal to 2.03 ± 0.11 eV. Fullerene C_{70} has the same E_A value as a result of the similar values for the $h\nu_{CT}$ energies for C_{60} and C_{70} complexes with the same donors in solution. The E_A values obtained for fullerenes in solution are close to adiabatic E_A of C_{60} (2.1–2.2 eV) [3].

However, the vertical E_A values of fullerenes estimated from the $h\nu_{CT}$ energies in solution are ~ 0.65 eV lower than those in gas phase (2.67 and 2.68 eV for C_{60} and C_{70} , respectively) [28,29]. We assume that this is a result of equating the energies of electrostatic interaction ($E_C(D-A)$) for TCNE and C_{60} complexes in Eq. (7). The energy of electrostatic interaction in the excited state of the complexes depends on the average distances between charges in the D^+-A^- pairs. In the complexes with planar acceptors (TCNE), the values of $E_C(D-A)$ are close due to the fact that the distances between planar donor and acceptor molecules in the D^+-A^- pairs differ only insignificantly. In contrast to planar acceptors the charge of C_{60}^- radical anion in the excited state is delocalized on a sphere with a 3.55 Å radius. This results in a significant increase in the average distances between charges in the $D^+-C_{60}^-$ pairs and consequently in a decrease of the energy of electrostatic interaction in fullerene complexes as compared to the complexes with planar acceptors.

4. Conclusion

The $h\nu_{CT}-I_p$ dependences in C_{60} and C_{70} complexes with tetrathiafulvalenes in solutions and in the solid state have been obtained. It is shown that the $h\nu_{CT}$ energies for C_{60} and C_{70} complexes are close, almost independent of the solvent polarity and lie ~ 0.15 – 0.20 eV higher than those in the solid state.

The $h\nu_{CT}-I_p$ dependences for C_{60} complexes with tetrathiafulvalenes lie ~ 0.6 – 0.7 eV higher than for TCNE ones both in solution and in the solid state. The vertical E_A of the fullerenes estimated from the $h\nu_{CT}$ energies in solution was found to be 0.65 eV lower than those in gas phase. This is obviously due to the decrease in the energy of electrostatic interaction in fullerene complexes as compared to the complexes with planar π -acceptors.

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