Charge transfer in complexes of C_{60} and C_{70} in solutions and solid state

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Electronic absorption spectra of complexes of C_{60} and C_{70} fullerenes with donors, tetrathiafulvalene and pyranylidene derivatives, were studied in solutions and in the solid state. Charge transfer bands were found in the 680—1300 nm range. The charge transfer energies ($h\nu_{ct}$) for the C_{60} and C_{70} complexes in solutions are close and almost independent of the solvent polarity. For the C_{60} complexes in the solid state, the dependence of $h\nu_{ct}$ on the ionization potential (IP) of donors was found to be $h\nu_{ct} = 0.82IP - 3.93$ eV. In the C_{60} complexes in the solid state, the $h\nu_{ct}$ values are 0.15—0.20 eV lower than those in the solution. The linear dependences of $h\nu_{ct}$ on IP of donors for the C_{60} complexes lie 0.6—0.7 eV higher than those in the complexes with tetracyanoethylene (TCNE). This is associated with lower values of the electron affinity of C_{60} and the energy of the electrostatic interaction in the fullerene complexes as compared to those of the TCNE complexes.

Key words: C_{60} and C_{70} fullerenes; charge transfer complexes; electronic absorption spectra; ionization potentials; energy of electrostatic interaction.

Donor-acceptor complexes of fullerenes, in which fullerenes act as π-acceptors,[1,2] can exhibit ferromagnetic and superconducting properties.[3—5]

To the present time, a considerable number of these complexes with fullerenes have been obtained.[6—16] Complex formation of fullerenes with different donors in solutions is under active study.[17—20] Optical absorption spectra of the majority of fullerene complexes contain charge transfer bands; hence, these compounds can be considered as charge transfer complexes (CTC).

The acceptor ability of fullerenes has been studied by different methods.[1,2] The vertical electron affinity (EA) estimated by the method of photodetachment of an electron from $C_{60}{}^{\cdot -}$ and $C_{70}{}^{\cdot -}$ radical anions in the gas phase is equal[2] to 2.67 and 2.68 eV for C_{60} and C_{70}, respectively. The adiabatic EA of C_{60} in a solution calculated from the values of the charge transfer energy ($h\nu_{ct}$) and redox potentials of C_{60} is 2.16 and 2.1—2.2 eV,[6] respectively. These values are substantially lower than the vertical EA of C_{60} in the gas phase.

For π-complexes formed by the same acceptor with a series of related donors in a solution, the dependence of the charge transfer energy on the ionization potential (IP) of donors is close to linear. These dependences are known for many planar organic π-acceptors.[21—23] For C_{60} complexes, these studies have been performed for substituted anilines,[18] aromatic hydrocarbons,[19] and substituted naphthalenes[20] in toluene solutions. According to the published data,[18,20] an almost linear dependence of $h\nu_{ct}$ on IP of donors is observed in the IP range from 7.2 to 8.2 eV.

For some C_{60} complexes in the solid state, the $h\nu_{ct}$ values have been determined[6—16] by optical spectroscopy, and the dependence of $h\nu_{ct}$ on the redox potential of donors has been considered.[6]

Charge transfer processes in polymer—C_{60} composites[24] and complexes of fullerenes with organic donors[16—20] are presently under intense study; therefore, it is especially urgent to reveal general dependences of changing the charge transfer energy in fullerene complexes.

In this work, we studied the C_{60} and C_{70} complexes with a wide series of donors, tetrathiafulvalene and pyranylidene derivatives, in solutions and in the solid state by optical spectroscopy. The dependences of the charge transfer energy on IP of donors were obtained. The influence of the solvent polarity on the $h\nu_{ct}$ value was considered. The change in the $h\nu_{ct}$ value on going from the fullerene complexes in a solution to their solid state was studied. The dependences obtained were compared to those for the tetracyanoethylene (TCNE) complexes.

Experimental

C_{60} and C_{70} fullerenes (99.9% and 98% purity, respectively) were used. Toluene was distilled over Na/benzophe-

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Table 1. Ionization potentials (*IP*) of donors (D), positions of maxima of charge transfer bands in the absorption spectrum (λ), and the corresponding $h\nu_{ct}$ values of C_{60} and C_{70} complexes in the solid state

Donor	*IP* /eV	Complex	λ /nm	$h\nu_{ct}$ /eV	Refs.
TPDP	—	$D(C_{60})_2(CS_2)_4$	1240	0.98	15, 25
OMTTF	6.3 [26]	$D \cdot C_{60} \cdot C_6H_6$ (1)	1040	1.20	7
		$D \cdot C_{60} \cdot C_5H_5N$ (2)	980	1.26	27
DMDPh-TTF	—	$D_2 \cdot C_{60} \cdot C_6H_6$	940	1.32	28
		$D_2 \cdot C_{70} \cdot C_6H_6$	1000	1.24	28
EDY-BEDT-DT	—	$D \cdot C_{60} \cdot C_6H_6$	935	1.32	16, 28
EDT-TTF	—	$D_2C_{60} \cdot CS_2$	900	1.35	13, 28
		$D \cdot C_{60} \cdot C_6H_6$	920	1.38	13, 28
BEDO-TTF	6.46 [29]	$D \cdot C_{60} \cdot C_6H_6$ (3)	900	1.38	7, 28
DPhTTF	—	$D \cdot C_{60} \cdot C_6H_6$	895	1.38	28
TMDTDM-TTF	—	$D_2C_{60}(CS_2)_3$	900	1.39	6, 13
BEDT-TTF	6.70 [29]	$D_2C_{60}(C_5H_5N)_2$ (4)	820	1.51	28
		$D \cdot C_{70} \cdot CS_2$ (5)	860	1.38	28
DBTTF	6.81 [30]	$D \cdot C_{60} \cdot C_6H_6$ (6)	735	1.69	11
		$D \cdot C_{60} \cdot C_5H_5N$ (7)	750	1.64	11

Note. TPDP is 3,3′,5,5′-tetraphenyldipyranylidene; OMTTF is octamethylenetetrathiafulvalene; DMDPh-TTF is *trans*-4,4′-dimethyl-5,5′-diphenyltetrathiafulvalene; EDY-BEDT-DT is 2,2′-ethanediylidene-bis(4,5-ethylenedithio-1,3-dithiol); EDT-TTF is ethylenedithiotetrathiafulvalene; BEDO-TTF is bis(ethylenedioxo)tetrathiafulvalene; DPhTTF is *trans*-4,4′-diphenyltetrathiafulvalene; TMDTDM-TTF is tetramethylenedithio-4,5-dimethyltetrathiafulvalene; BEDT-TTF is bis(ethylenedithio)tetrathiafulvalene; and DBTTF is dibenzotetrathiafulvalene.

none under argon, and benzonitrile (Aldrich, HPLC grade) was distilled in a vacuum in an argon flow. These solvents do not absorb in the 400—1200 nm region studied.

The donor compounds and their vertical *IP* are presented in Table 1.

The values of vertical *IP* of tetramethyltetrathiafulvalene (TMTTF), BEDO-TTF, BEDT-TTF, and DBTTF have been determined by photoelectron spectroscopy in the gas phase,[29—31] and that of OMTTF has been determined by spectroscopy of CTC.[26]

The C_{60} and C_{70} complexes were obtained in the crystalline form by slow evaporation of solutions of a donor and fullerene in the corresponding solvent (CS_2, benzene, toluene, or pyridine) under an argon atmosphere. The compounds synthesized were characterized by the data of elemental and thermogravimetric analyses and the methods of optical, X-ray photoelectron, and IR spectroscopy.[16,27,28,32]

The crystal structures of the complexes $OMTTF \cdot C_{60} \cdot C_6H_6$,[6] $TPDP(C_{60})_2(CS_2)_4$,[25] $DBTTF \cdot C_{60} \cdot C_6H_6$,[11] and $(TMDTDM\text{-}TTF)_2C_{60}(CS_2)_3$[13] have been studied previously.

Absorption spectra were recorded on a Perkin—Elmer Lambda 19 UV-VIS-NIR spectrophotometer within the 220—3000 nm wavelength region. Crystalline samples were prepared as pellets with KBr (1 : 2000). Charge transfer bands (CTB) were observed in the visible and near-IR regions at λ = 750—1300 nm. These regions contain no absorption bands related to intramolecular electron transitions of fullerenes and donors. To determine more exactly maxima of CTB, the spectrum of the starting fullerene normalized to the absorption at 600 nm was subtracted from the spectrum of the corresponding complex.

Absorption spectra of the starting solutions and solutions of the fullerene complexes in toluene and benzonitrile were recorded in the 400—1600 nm region at 23 °C. Solutions of C_{60} and C_{70} fullerenes with a concentration of 0.5 mg mL^{-1} were placed in a sealed quartz cell (thickness 0.5 cm, volume 1 cm^3). After the spectrum of the starting solution of fullerene was recorded, a donor was added to the solution, and the spectra of fullerene solutions with different concentrations of the donor were recorded. CTB were isolated by subtraction of the spectra of the starting solutions of fullerenes from the spectra of solutions containing the donor (concentration 6—8 mg mL^{-1}). No absorption of donors was observed in the region of CTB (680—1000 nm).

Results and Discussion

Charge transfer bands of the C_{60} and C_{70} complexes in solutions

Optical spectra of the fullerene complexes were studied in toluene and benzonitrile solutions. Tetrathiafulvalenes with known *IP* were chosen as donors: DBTTF, BEDT-TTF, TMTTF, and OMTTF. Because of a low solubility of BEDT-TTF (<2 mg mL^{-1}) in toluene and benzonitrile, we failed to determine the position of CTB for the BEDT-TTF complexes with C_{60} and C_{70}.

After OMTTF was added to a solution of C_{60} in toluene, the intensity of absorption in the 800—900 nm region increases proportionally to the concentration of OMTTF (Fig. 1). This absorption is absent in the spectra of the starting OMTTF and C_{60} (see Fig. 1, spectra *1* and *2*). Similar situation is observed for DBTTF and TMTTF. Since the positions of maxima of new absorption bands depends on *IP* of the corresponding donors, and their intensity is proportional to the concentration of the donor, the appearance of the new bands is evidently related to the formation of CTC.

The CTB isolated in the optical absorption spectra of the C_{60} complexes with DBTTF, TMTTF, and OMTTF

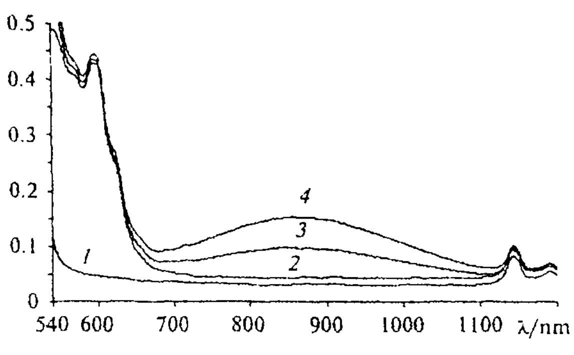

Fig. 1. Spectra of solutions of C_{60} and OMTTF in toluene in the 540—1200 nm region: donor OMTTF (3.5 mg mL^{-1}) (*1*), starting solution of C_{60} (0.5 mg mL^{-1}) (*2*), solutions of C_{60} and OMTTF with the concentration of OMTTF equal to 3.5 (*3*) and 7 mg mL^{-1} (*4*).

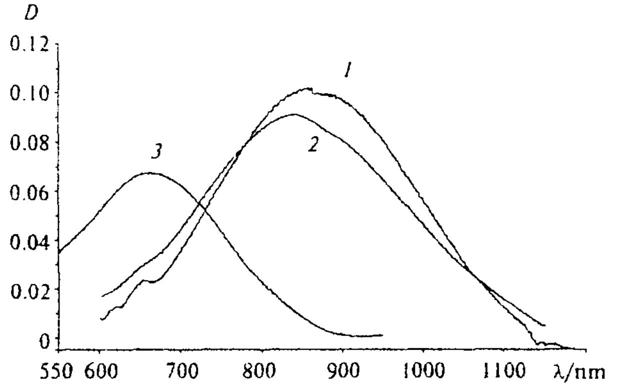

Fig. 2. CTB of C_{60} complexes with TTF derivatives in toluene: *1*, OMTTF (7 mg mL^{-1}); *2*, TMTTF (8 mg mL^{-1}); and *3*, DBTTF (8 mg mL^{-1}).

Table 2. Charge transfer energy corresponding to the maximum of CTB ($h\nu_{ct}$) for C_{60} and C_{70} complexes with different donors in toluene and benzonitrile

Donor	*IP* /eV	$h\nu_{ct}$/eV			
		Toluene		Benzonitrile	
		C_{60}	C_{70}	C_{60}	C_{70}
OMTTF	6.30 [26]	1.44	1.43	1.43	1.43
TMTTF	6.39 [31]	1.48	1.44	1.47	—
DBTTF	6.81 [30]	1.85	1.82	1.85	—

in toluene are presented in Fig. 2. The charge transfer energies corresponding to the maximum of CTB ($h\nu_{ct}$) of the C_{60} and C_{70} complexes in toluene and benzonitrile solutions are presented in Table 2. It is seen that the $h\nu_{ct}$ values of the complexes of C_{60} and C_{70} fullerenes with the same donors in solutions are close.

It has previously been shown[21] that a polar solvent can stabilize the excited ionic state of complexes of the π—π-type, which results in a decrease in $h\nu_{ct}$ on going from the nonpolar to polar solvent. To estimate the influence of the solvent polarity on the charge transfer energy, we performed similar measurements of the optical absorption spectra of the C_{60} and C_{70} complexes in solutions of more polar benzonitrile (see Table 2). It is seen that the $h\nu_{ct}$ values (within the experimental accuracy of measurements) remain unchanged on going from toluene to benzonitrile.

Thus, in the fullerene complexes, unlike planar acceptors of the π-type, the charge transfer energy is almost independent of the solvent polarity.

Charge transfer bands of the C_{60} and C_{70} complexes in the solid state

The absorption spectra of the starting OMTTF (spectrum *1*), C_{60} (spectrum *2*), and their complex OMTTF · C_{60} · C_6H_6 (spectrum *3*) in the solid state are presented in Fig. 3. It is seen that the spectrum of the complex contains a new broad absorption band with a maximum at ~1000 nm. This band is absent in the spectra of the starting OMTTF and C_{60} and can be assigned to CTB.

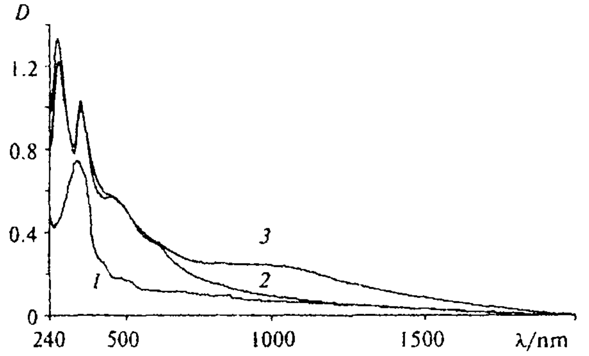

Fig. 3. Absorption spectra of OMTTF (*1*), C_{60} (*2*), and the OMTTF · C_{60} · C_6H_6 complex (*3*) in KBr pellets.

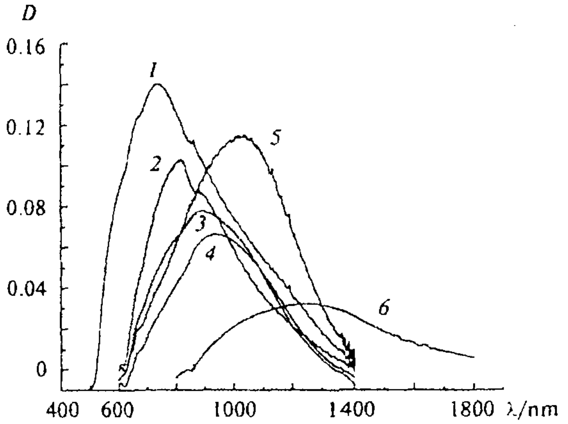

Fig. 4. CTB of C_{60} complexes in the solid state in the 500—1600 nm region: DBTTF · C_{60} · C_6H_6 (*1*); $(BEDT\text{-}TTF)_2C_{60}(Py)_2$ (*2*); $(EDT\text{-}TTF)_2C_{60}$ · CS_2 (*3*); $(DMDPh\text{-}TTF)_2C_{60}$ · C_6H_6 (*4*); OMTTF · C_{60} · C_6H_6 (*5*); and $TPDP(C_{60})_2(CS_2)_4$ (*6*).

The charge transfer bands isolated in the absorption spectra of several C_{60} complexes in the solid state are presented in Fig. 4. They are broad, structureless, and asymmetrical, and their position depends on *IP* of donors, which is typical of CTB.

The absorption spectra of the complexes of C_{70} fullerene (BEDT-TTF · C_{70} · CS_2, $(DMDPh\text{-}TTF)_2$ · C_{70} · C_6H_6) also contain low-intense CTB. However, no charge transfer bands were observed in the DBTTF · C_{70} · C_6H_6 and OMTTF · C_{70} · C_6H_6 complexes.

The charge transfer energies corresponding to the maximum of CTB ($h\nu_{ct}$) for the C_{60} and C_{70} complexes in the solid state are presented in Table 1.

Specific features of charge transfer in fullerene complexes

In the IR spectra of the C_{60} compounds studied, the shift of the absorption band of C_{60} at 1429 cm^{-1}, which is most sensitive to the charge transfer to the fullerene molecule, relative to that of individual C_{60} does not exceed[28] 2 cm^{-1}. The spectra of these complexes exhibit no ESR signals corresponding to $C_{60}^{\cdot -}$ or $C_{70}^{\cdot -}$. Thus, these compounds are neutral complexes.

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

$$D^{\delta+}A^{\delta-} \rightarrow D^{(1-\delta)+}A^{(1-\delta)-}.$$

The linear part of the dependence of the charge transfer energy on *IP* of the donor for neutral complexes of one acceptor with a series of π-donors is described by the expression[21,22]

$$h\nu_{ct} = \alpha IP - C_1, \qquad (1)$$

where α is constant, *IP* is the vertical ionization potential of the donor, and C_1 is the characteristics of the acceptor known as the apparent electron affinity.[22]

The C_1 value in a solution is mainly determined by the vertical electron affinity (*EA*) of the acceptor and the energy of the electrostatic interaction of the donor-acceptor pair in the excited state (E_s). Therefore, Eq. (1) for solutions can also be presented in another form[33]:

$$h\nu_{ct} = \alpha(IP - EA) - E_s. \qquad (2)$$

The dependence of $h\nu_{ct}$ on *IP* of the C_{60} complexes with tetrathiafulvalenes in toluene is presented in Fig. 5 (straight line *a*). It is seen that $h\nu_{ct}$ decreases as *IP* of the donor decreases.

The dependence of $h\nu_{ct}$ on *IP* for the C_{60} complexes with tetrathiafulvalenes obtained in this work is close to that published for the C_{60} complexes with substituted anilines and naphthalenes.[18,20] Based on these data, we can plot the general dependence of $h\nu_{ct}$ on *IP* of different donors (within a wide range of the *IP* values from 6.3 to 8.2 eV) for the C_{60} complexes in toluene (Fig. 6). It is almost linear and described by the expression

$$h\nu_{ct} = 0.90IP - 4.28 \text{ eV}, \qquad (3)$$

which makes it possible to use it for the estimation of *IP* of donors from the charge transfer energy of the C_{60} complexes in toluene.

For the C_{60} complexes in the solid state with the OMTTF, BEDT-TTF, BEDO-TTF, and DBTTF donors (whose *IP* values are known), we plotted the dependence of $h\nu_{ct}$ on *IP* of the donors (see Fig. 5, straight line *b*), which can be approximated by the expression

$$h\nu_{ct} = 0.82IP - 3.93 \text{ eV}. \qquad (4)$$

Linear correlations between *IP* and $h\nu_{ct}$ for CTC of one acceptor with a series of related donors are usually observed in solutions. The linearity of this correlation can be distorted in the solid state of CTC. This can be related to a change in the E_s value, which is strongly affected by the type of crystalline packing and the average distance between the donor and acceptor molecules in the crystal structure of CTC. In this case, the linearity of the correlation obtained for CTC of C_{60} in the solid state is evidently related to a similar coordination in the complexes of substituted tetrathiafulvalenes and C_{60}.

The $h\nu_{ct}$ values of the C_{70} complexes (see Table 1 and Fig. 5, complex 5) in the solid state lie ~0.08 eV lower than those of the C_{60} complexes with the same

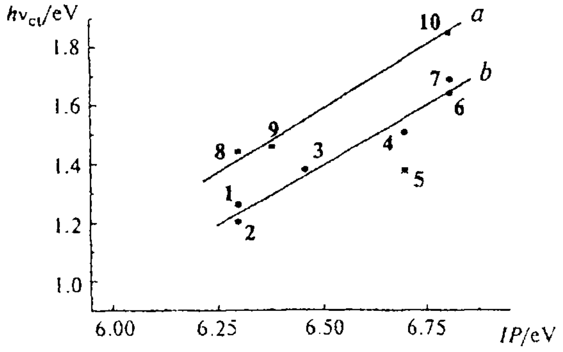

Fig. 5. Dependence of $h\nu_{ct}$ on *IP* of donors for C_{60} and C_{70} complexes with donors with known ionization potentials: *a*, complexes of C_{60} with OMTTF (**8**), TMTTF (**9**), and DBTTF (**10**) in toluene; *b*, complexes **1**—**7** of fullerenes C_{60} (**1**—**4**, **6**, **7**) and C_{70} (**5**) in the solid state. For numeration of the complexes, see Table 1.

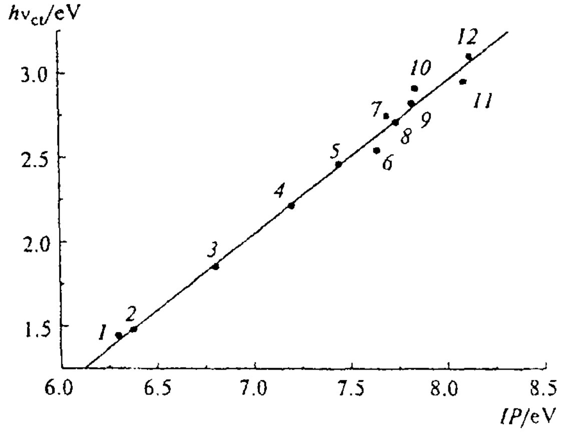

Fig. 6. Dependences of $h\nu_{ct}$ on *IP* of tetrathiafulvalenes, substituted anilines, and naphthalenes in C_{60} complexes in toluene: OMTTF (*1*); TMTTF (*2*); DBTTF (*3*); *N,N*-diethylaniline[18] (*4*); *N,N*-dimethylaniline[18] (*5*); *N*-methylaniline[18] (*6*); 1-methoxynaphthalene[20] (*7*); 2,6-dimethylaniline[18] (*8*); *ortho*-toluidine[18] (*9*); 1-methylnaphthalene[20] (*10*); aniline[18] (*11*); and 1-chloronaphthalene[20] (*12*).

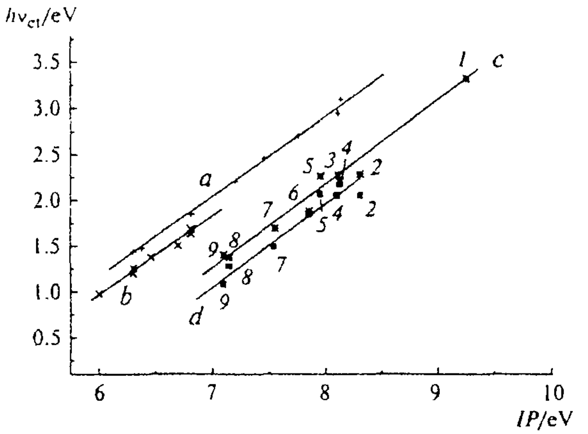

Fig. 7. Linear dependences of $h\nu_{ct}$ on *IP* of donors for C_{60} complexes in toluene solutions (*a*) and in the solid state (*b*); for TCNE complexes in dichloromethane solutions (*c*) and in the solid state (*d*)[22]: benzene (*1*); pentamethylbenzene (*2*); phenanthrene (*3*); naphthalene (*4*); hexamethylbenzene (*5*); acenaphthene (*6*); pyrene (*7*); perylene (*8*); and anthanthrene (*9*).

donors (BEDT-TTF and DMDPh-TTF), whereas the C_{60} and C_{70} complexes with the same donors in solutions have close $h\nu_{ct}$ values.

On going from the solution to the solid state, for the C_{60} complexes with DBTTF and OMTTF, $h\nu_{ct}$ decreases by 0.15—0.20 eV. A similar decrease in $h\nu_{ct}$ (~0.2 eV) has been observed previously[22] for the TCNE complexes with aromatic hydrocarbons.

The dependences of $h\nu_{ct}$ on *IP* of the donors obtained above for the C_{60} complexes in solution (*a*) and the solid state (*b*) are compared with similar dependences[22] for the TCNE complexes with aromatic hydrocarbons with the neutral ground state in solution (*c*) and the solid state (*d*) (see Fig. 7). For the TCNE complexes in dichloromethane, this dependence is described[22] by the expression

$$h\nu_{ct} = 0.87IP - 4.86 \text{ eV}. \qquad (5)$$

It is seen in Fig. 7 that in both solution and solid state, the linear dependences of $h\nu_{ct}$ on *IP* of the donors for the C_{60} complexes lie 0.6—0.7 eV higher. Since these dependences for the TCNE and C_{60} complexes in solution have close slope angles (α is 0.87 and 0.90), the $h\nu_{ct}$ values of these complexes can be compared directly.

The decrease in $h\nu_{ct}$ (0.6—0.7 eV) observed on going from the TCNE complexes to the C_{60} complexes, according to Eq. (2), is mainly related to the difference between the vertical *EA* of these acceptors and the energy of the electrostatic interaction in the complexes.

The vertical *EA* of C_{60} is[2] ~2.67 eV; *EA* of TCNE (according to the charge transfer energy in complexes with π-donors, which corresponds to the vertical value) is equal[34] to 2.77 eV. Thus, the *EA* of C_{60} is ~0.1 eV lower than that in the case of TCNE.

Taking into account that the decrease in $h\nu_{ct}$ of the C_{60} complexes relative to that for similar TCNE complexes is substantially higher than the difference in their *EA*, it can be assumed that the energy of the electrostatic interaction in the C_{60} complexes should also be lower (by 0.5—0.6 eV) than that in the TCNE complexes.

The decrease in the energy of the electrostatic interaction in the C_{60} complexes as compared to the planar π-acceptor TCNE is related, probably, to the spherical shape of this molecule. The charge delocalization in the excited state of the complex over the sphere of the fullerene radical anion with a radius of 3.55 Å results in an increase in the average distance between charges in the $D^{\cdot+}$—$C_{60}{}^{\cdot-}$ ion pair and a decrease in E_s.

Thus, the dependences of the charge transfer energy on *IP* of donors in the C_{60} complexes with tetrathiafulvalenes in toluene and in the solid state were obtained. The $h\nu_{ct}$ values for CTC of C_{60} and C_{70} in solutions are close, independent of the solvent polarity, and lie by ~0.15—0.20 eV higher in energy than those in the solid state.

The comparison of the linear dependences of $h\nu_{ct}$ on *IP* of donors for the C_{60} complexes and TCNE complexes indicates a decrease in the energy of the electrostatic interaction in the first complexes by 0.5—0.6 eV.

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