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New complexes of fullerenes C_{60} and C_{70} with organic donor DBTTF: synthesis, some properties and crystal structure of DBTTF·C_{60}·C_6H_6 (DBTTF = dibenzotetrathiafulvalene)

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Abstract

New complexes of fullerenes C_{60} and C_{70} with dibenzotetrathiafulvalene (DBTTF), DBTTF·C_{60}·C_6H_6, DBTTF·C_{60}·Py and DBTTF·C_{70}·C_6H_6, were obtained. The crystal structure of DBTTF·C_{60}·C_6H_6 was determined. The arrangement of fullerene molecules is approximately simple cubic packing where each C_{60} molecule is located in slightly distorted octahedral surrounding. DBTTF molecules have a concave conformation. X-ray photoelectron spectroscopy (XPS) and IR spectroscopy show only weak charge transfer in these compounds. A weak charge transfer band near 900 nm was found in the UV–Vis–NIR absorption spectrum of DBTTF·C_{60}·C_6H_6 single crystals. DBTTF molecules are coordinated on C_{60} ones by π–π and n–π interactions. It was shown that the steric discrepancy between DBTTF and C_{60} molecules does not provide favourable conditions for charge transfer in the DBTTF·C_{60}·C_6H_6 complex.

Keywords: Fullerenes and derivatives; Dibenzotetrathiafulvalene; Crystal structures

1. Introduction

Fullerenes form various charge transfer (CT) complexes [1–6] and ion-radical salts [7–9] with different physical properties [5,7]. Most CT complexes have a weak CT rate. This is associated with weak acceptor properties of fullerenes. Tetrathiafulvalene (TTF) derivatives are strong donors and are planar in the initial state. They change conformation and become concave at the C_{60} coordination [1,4,6].

CT defines basic properties of the complexes, especially their electrical, optical and magnetic ones. CT in such complexes has some peculiarities at the donor–fullerene interaction due to the spherical shape of the C_{60} molecules.

The variety of donor molecules gives the possibility for the preparation of CT complexes of C_{60} with different arrangements of fullerene molecules in crystals (one-, two- or three-dimensional packings of the molecules with different distances between them [1,4–6]). The electron state of C_{60} can be changed by alkali metal doping. The first reports on the alkali doping of C_{60} CT complexes with organic donors of the TTF family show the appearance of superconductivity in doped materials [10]. Thus, the preparation of new CT complexes and their doping can result in materials with various conducting and magnetic properties.

Here we report the synthesis of C_{60} and C_{70} complexes with organic donor, dibenzotetrathiafulvalene (DBTTF): DBTTF·C_{60}·C_6H_6, DBTTF·C_{60}·Py and DBTTF·C_{70}·C_6H_6. The crystal structure of DBTTF·C_{60}·C_6H_6 is determined. The UV–Vis–NIR spectrum of single crystals, and spectra of some complexes via IR spectroscopy and X-ray photoelectron spectroscopy (XPS) are studied. The possibilities for CT in DBTTF·C_{60}·C_6H_6 depending on the coordination of DBTTF and C_{60} molecules are discussed.

2. Experimental

DBTTF·C_{60}·C_6H_6 (**1**) and DBTTF·C_{70}·C_6H_6 (**3**) were obtained by the evaporation of benzene solution of C_{60}, C_{70} and DBTTF at 1:3.5 molar ratio and at 1:4 molar ratio, respectively, under Ar for 5 days. Quality single crystals of **1** are formed only at 3.5–4.0 DBTTF molar excess. The adduct C_{60}

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with a benzene is formed with a DBTTF molar excess of less than 3.5. Splices of crystals of **1** are formed when using a molar excess of DBTTF of more than 4.0. Single crystals were separated from donor ones by using a microscope and were washed by ether. They are formed as black parallelograms up to 1–2 mm size with 70% yield for **1** and 50% for **3**. *Anal.* Found for **1**: $C_{80}H_{14}S_4$; S, 10.97; H, 1.54; C, 86.81. Calc.: S, 11.55; H, 1.26; C, 87.19%. *Anal.* Found for **3**: $C_{90}H_{14}S_4$; S, 11.31; H, 1.42; C, 87.37. Calc.: S, 10.50; H, 1.14; C, 88.36%.

DBTTF·C_{60}·Py (**2**) was obtained by the evaporation of 50 ml of saturated pyridine solution containing 25 mg of C_{60} and 105 mg of DBTTF (1:10 molar ratio) under Ar. The formation of single crystals in a solution was observed after 5 days of the evaporation. The solution was decanted after 10 days of the evaporation before the crystallization of DBTTF crystals began. The single crystals were washed by ether. They were obtained with 70% yield as black parallelograms up to 1 mm size. *Anal.* Found: $C_{79}H_{13}S_4N$; S, 12.20; N, 1.32; H, 1.24; C, 87.64. Calc.: S, 11.52; N, 1.26; H, 1.17; C, 86.05%.

The single-crystal X-ray study was carried out in the X-ray Structural Centre of the Institute of Organoelement Compounds RAS (Moscow) with an automated four-circle Siemens P3 diffractometer. The measurements were performed at 153 K by using graphite-monochromatized Mo Kα radiation. The crystal data for the C_{60}·DBTTF·C_6H_6 complex are: $C_{82}H_{14}S_4$, F.W. = 1127.25, monoclinic space group *C2/c*, $a = 16.257(3)$, $b = 13.311(2)$, $c = 20.799(5)$ Å, $\beta = 103.02(2)°$, $V = 4385(2)$ Å^3, $Z = 4$; $D_c = 1.583$ g cm^{-3}. The intensities were measured in the $\theta/2\theta$ scanning mode up to $2\theta_{max} = 58°$, the total number of reflections was $N_{tot} = 4821$ with $N_{obs} = 3198$ ($F_o > 2\sigma$). The structure was solved by direct methods of SHELXS-86 and refined by using the least-squares methods of SHELXL-93 with anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were located on the difference Fourier map and refined isotropically. Finally, the values $R_f = 0.069$ and $R_w = 0.170$ (590 parameters) were achieved.

The IR spectra of C_{60}, C_{70}, DBTTF, **1**, **2** and **3** were measured in KBr pellets. XPS spectra were excited by Mg Kα radiation ($h\nu = 1253.6$ eV). The preparation of the samples for XPS investigation is described in detail in [11]. The spectra were calibrated to the N(1s) peak (285.0 eV). This value corresponds to the position of C(1s) peaks for solid C_{60} and C_{60} films deposited on different supports [12]. The reflection spectrum of DBTTF·C_{60}·C_6H_6 single crystals was measured on a two-beam spectroreflectometer in the 250–1100 nm range. The absorption spectrum was obtained by the Kramers–Kronig transformation of the reflection spectrum.

3. Results and discussion

All compounds of 1:1:1 composition were obtained by a slow evaporation of fullerene and DBTTF solutions. The complexes can be obtained only at a large molar excess of the donor.

The thermogravimetry of **1** revealed that benzene is released from the crystals at 405 K which is above the b.p. of benzene (353 K). A partial decomposition of DBTTF begins only from 705 K and is consistent with the decomposition temperature of the individual donor. The loss of mass after 1075 K is due to C_{60} sublimation.

The IR spectra of **1**, **2** and **3** show that the positions of absorption bands of C_{60} at 1430 (coincides with DBTTF), 1182, 557 and 527 cm^{-1}, and C_{70} at 1430 (coincides with DBTTF), 1133, 796, 642, 566, 535 and 451 cm^{-1} remain practically unchanged with respect to those of individual fullerenes. The absorption bands of the donor molecule are presented in Table 1. The frequency associated with DBTTF molecule bond at 1444 cm^{-1}, which is more sensitive to the changes of electron density on donor molecules, is downshifted by 2 cm^{-1}. This indicates only weak CT from DBTTF molecules to fullerenes in these complexes. The shifts of some other absorption bands by 1–6 cm^{-1} are obviously associated with a breakdown of the planarity of the donor molecule because of its coordination with C_{60}. The absorption bands in **1** and **3** at 674 (coincides with DBTTF) and 1478 cm^{-1} are attributed to benzene, and those in **2** at 601, 691, 701 and 1580 cm^{-1} are attributed to pyridine.

XPS data on the energies of N(1s) and S(2p) peaks of DBTTF and Py in the individual state and in fullerene complexes are presented in Table 2. The positive shift of the S(2p) peak by 0.3 eV was observed for complex **1**. This is

Table 1
Absorption bands of DBTTF in individual state and in fullerene complexes

Compound	Absorption bands[a] (cm^{-1})											
DBTTF	1444	1431[b]	1283	1260	1119 1123S	1043	1029	924 930S	775	742	675	669
1	1142	1430[b]	1285	1260	1113	1043	1029	927	776	740	674[c]	665
2	1442	1430[b]	1285	1260	1113	1043	1029	927	776	738	675	664
3	1442	1430[b]	1283	1260	1117	1043	1029	927	776	739	674[c]	

[a] S = shoulder.

[b] Coincides with C_{60}.

[c] Coincides with C_6H_6.

Table 2
Position of N(1s) and S(2p) peaks of DBTTF and Py in individual state and in fullerene complexes

Compound	N(1s) (±0.1 eV)	S(2p) (±0.1 eV)
Py [a]	399.1	
o-Chloranyl–Py [a]	402.4	
DBTTF		164.0
1		164.3
2	400.7	164.1
3		164.1

[a] Ref. [13].

obviously associated with small CT from DBTTF to C_{60}. The shift of the N(1s) peak in **2** evidences the change of the effective charge on nitrogen atoms of pyridine with respect to pure pyridine. This shift is similar to that in the donor–acceptor *ortho*-chloranyl–pyridine complex [13]. Thus, the pyridine molecule takes part in the donor–acceptor interaction with C_{60} as a donor also.

XPS spectra of solid C_{60}, 1 and pure DBTTF are presented in Fig. 1. A satellite peak is observed near the photoelectron C(1s) peak as a shoulder in all spectra. The results of the Gaussian simulation of the spectra are presented in Table 3. It is seen that see that the satellite peak in the spectrum of **1** is closer to that in the spectrum of C_{60} than in the spectrum of DBTTF.

All valence electron plasma oscillations are also observed in XPS spectra. The $\sigma+\pi$ plasmon energy ($\hbar\omega_p(\sigma+\pi)$), determined according to [14], is equal to 25.2 eV for **1**. The same measurements for solid C_{60} yield 26.1 eV. The calculations of $\hbar\omega_p(\sigma+\pi)$ in terms of the free electron model ($\omega_p=(4\pi ne^2/m)^{1/2}$, where n is valence electrons density, e and m are the charge and the mass of the electron, respectively, yield 21.2 eV for the complex and 21.4 eV for solid C_{60}. In the case of C_{60} the valence electron density was evaluated from the mass density of solid fullerene which is equal to 1.65 g cm^{-3} [15] ($n(C_{60})=0.331$ e Å^{-3}). In the case of the complex, $n=0.327$ e Å^{-3}. This value can be calculated from structural data presented in this work. The values of δ ($\delta=1-\hbar\omega_p(\text{theor})/\hbar\omega_p(\text{exp})$), which show a degree of deviation of calculated energies from experimental ones, are equal to 0.16 and 0.18 for the complex and solid C_{60}, respectively. According to [16], the $\sigma+\pi$ plasmon is more delocalized in the complex than in solid fullerene.

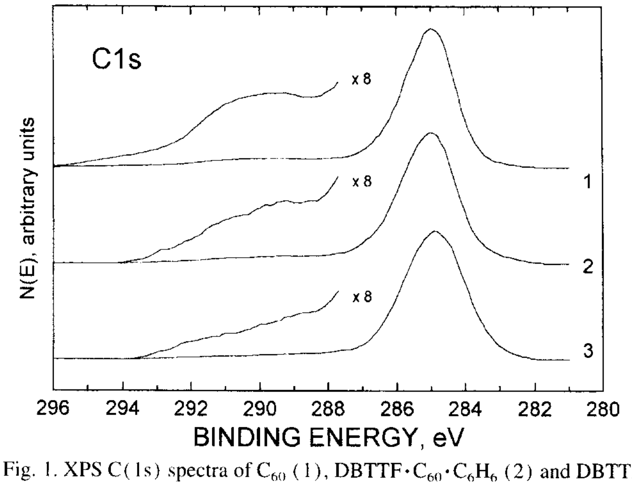

Fig. 1. XPS C(1s) spectra of C_{60} (1), DBTTF·C_{60}·C_6H_6 (2) and DBTTF (3) after the subtraction of a linear background and normalization to peak intensity.

Table 3
Gaussian simulation of C(1s) spectra

Compound	Peak	Area (%)	Width (eV)	Energy loss (eV)
C_{60}	C(1s)	76	1.48	
	satellite	24	6.01	3.14
DBTTF·C_{60}·C_6H_6	C(1s)	76	1.61	
	satellite	24	6.08	2.07
DBTTF	C(1s)	89	1.77	
	satellite	9	4.56	3.34

The crystal structure of C_{60}·DBTTF·C_6H_6 shows the composition of the complex to be 1:1:1. Donor and solvent molecules occupy special positions: DBTTF molecules are on a twofold axis and C_6H_6 molecules are in the inversion centre. The fullerene molecule also occupies a special position on a twofold axis.

The crystal packing of the structure is shown in Fig. 2. The arrangement of fullerene molecules can be considered as an approximately simple cubic close packing. Therefore, each C_{60} molecule is surrounded by six nearest C_{60} neighbours which form a distorted octahedron. The shortest distance between the centres of C_{60} molecules in the *c* direction is 10.4 Å. The analogous value in the *ab* plane is 10.5 Å. These distances are longer than those in individual fullerene (10.02 Å) [17], and C_{60} van der Waals radius (10.18 Å). Thus, the packing of C_{60} molecules is isolated.

DBTTF and benzene molecules occupy cavities in the spatial framework. DBTTF molecules are arranged in chains along the *c* direction. The shortest intermolecular C...C contact between carbon atoms of DBTTF benzene rings is about 3.30 Å (the van der Waals radii sum of sp^2 carbon atoms 3.35 Å [18]). But π–π interaction between the neighbouring DBTTF molecules is impossible due to strongly unparallel arrangements of benzene rings.

The efficient C_{60}–DBTTF packing is achieved due to a 'concave' (boat) conformation of the donor molecule. This conformation is typical for other fullerene donor–acceptor complexes with TTF derivatives [1,4,6]. Molecular geometry and the main structural parameters of the DBTTF molecule are shown in Fig. 3.

Fullerene molecules are statistically disordered between two equi-probable orientations generated by molecule rotation about one of its threefold axes by 60°. This rotation forms a statistical twofold axis which crosses two opposite pentagonal rings and coincides with the crystallographic twofold axis in the direction normal to the rotational threefold axis. This symmetry operation also transfers one hexagonal ring

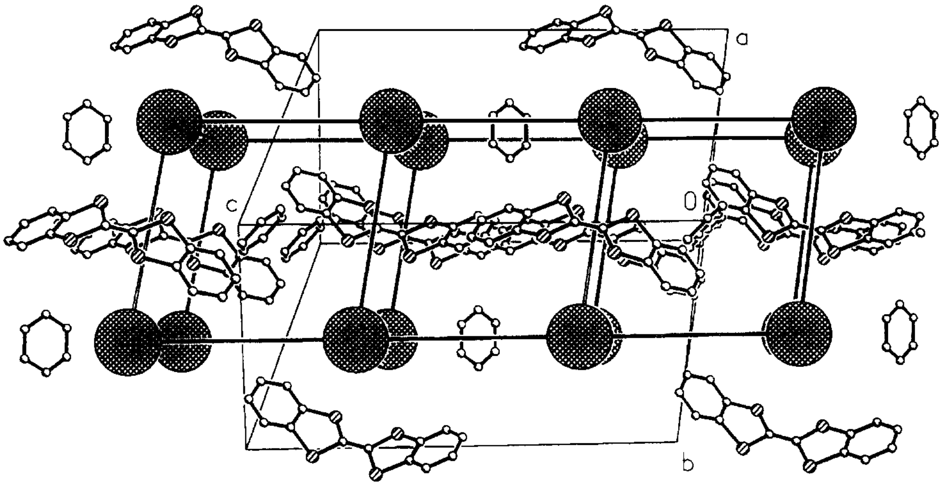

Fig. 2. Crystal packing of the donor–acceptor complex C_{60}·DBTTF·C_6H_6 (positions of fullerene molecules are marked by grey spheres, a unit cell is marked by fine lines).

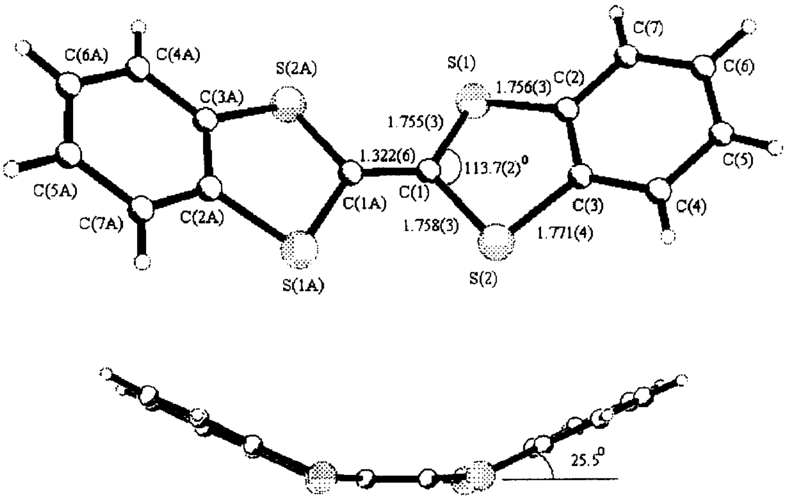

Fig. 3. Molecular geometry and main structural parameters of the DBTTF molecule in the C_{60}·DBTTF·C_6H_6 complex.

(of an asymmetric unit) into itself in such a way that six carbon atoms forming this hexagonal ring are processed as ordered at the structure refinement. The solution and refinement of the structure in the *C*/*c* space group lead to the same type of disorder.

Sulfur atoms of the DBTTF molecule form intermolecular contacts with fullerene ones and therefore their coordination should be discussed. Each DBTTF molecule has close van der Waals contacts with four fullerene molecules formed in two different ways (Fig. 4). The first way is that a sulfur atom S(1) and its symmetry equivalent S(1A) (see Fig. 3) are coordinated with an ordered hexagonal ring of the fullerene molecules; S...C distances are in the 3.47–3.72 Å range which is close to the sum of the van der Waals radii of S and C atoms (3.55 Å [18]). The plane of four sulfur atoms is virtually perpendicular to the plane of this hexagonal ring. This coordination is assumed to be realized through the n–π interaction of DBTTF sulfur atoms with the electron-poor C_{60} hexagonal ring. In the second mode, the plane of sulfur atoms coordinates pentagonal rings of fullerene molecules by the π–π interaction; the dihedral angle between these planes is 10.8° and is equal for both orientations of fullerene molecules. Benzene molecules are coordinated on pentagonal rings with 9.2° dihedral angles in one C_{60} orientation and on C_{60} electron-rich 6–6 bonds in the other orientation. The fact that the disorder of fullerene molecules does not change the coordination type with DBTTF sulfur atoms indicates these interactions to be principal for the crystal packing formation.

The reflection spectrum of DBTTF·C_{60}·C_6H_6 single crystals is presented in Fig. 5. The main absorption bands at 275 nm (4.60 eV) and 345 nm (3.60 eV) are associated with allowed C_{60} electron transitions. The position of these bands are slightly shifted with respect to those of C_{60} single crystals at 262 nm (4.45 eV) and 350 nm (3.54 eV), respectively [19]. The C_{60} absorption band at 450 nm (2.75 eV) [19] is practically not distinguished in the spectrum of **1**. The absorption bands of the DBTTF electron transition (below 220, 293, 360 nm in individual donor) are closed by C_{60} absorption bands in the spectrum of **1**. A very broad and weak band near 900 nm is associated with CT from DBTTF to fullerene. Surprising is the very weak intensity of this band. It is known that CT proceeds from the HOMO orbital of the donor to

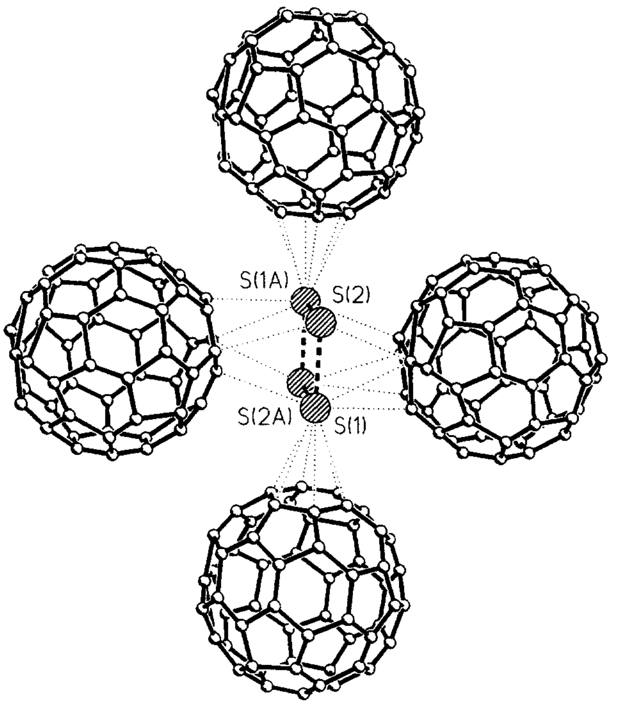

Fig. 4. Coordination of sulfur atoms on fullerene molecules. In the DBTTF molecule carbon atoms are omitted for simplicity.

LUMO orbitals of the acceptor at their overlapping. Thus, favourable conditions for CT are short distances (less than the sum of van der Waals radii of corresponding atoms) and the more or less parallel arrangement [20] of the HOMO b_{1u} π molecular orbitals of DBTTF delocalized over the whole conjugated molecule [21] and the t_{1u} π^* molecular orbitals of C_{60}.

The arrangement of the DBTTF molecule relative to the C_{60} surface is shown in Fig. 6. The DBTTF molecule has a boat conformation with three planar fragments, namely, the

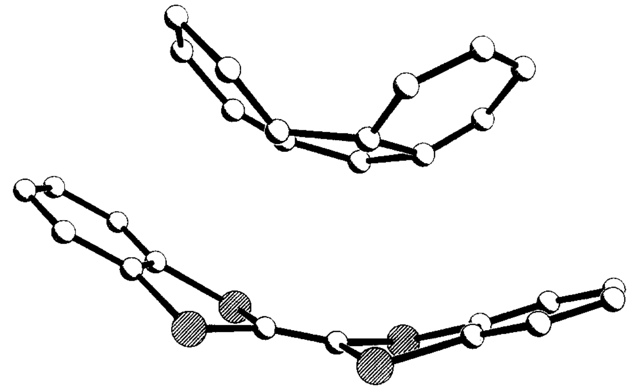

Fig. 6. Coordination of the DBTTF molecule on a fragment of the fullerene molecule.

tetrathiafulvalene core (S_4C_2) and two outer phenyl rings. They are coordinated on the pentagonal and two hexagonal rings of C_{60} with the corresponding angles 10.8, 19.0 and 28.8° for one C_{60} orientation, and 10.8, 28.8 and 19.0° for the other C_{60} orientation. In this case only the π-orbital partially localized on planar S_4C_2 fragments is slightly overlapped with the C_{60} π^* orbital of the pentagonal ring due to the dihedral angle between them corresponding to the parallel arrangement (10.8°) and the shortest S(DBTTF)...C (C_{60}) distance (3.505 Å) being a little less than the sum of the van der Waals radii of sulfur and carbon atoms (3.55 Å) [22]. The transfer of electron density from the π-orbital partially localized on phenyl rings of DBTTF to the C_{60} molecule is hindered by a large dihedral angle and a quite large C(DBTTF)...C(C_{60}) distance. It should be noted that the electron density transfer in the DBTTF molecule between phenyl rings and the S_4C_2 fragment is also hindered by a large dihedral angle (25.5°) between these planar parts. Thus, the CT from the HOMO orbital of DBTTF to the LUMO orbital

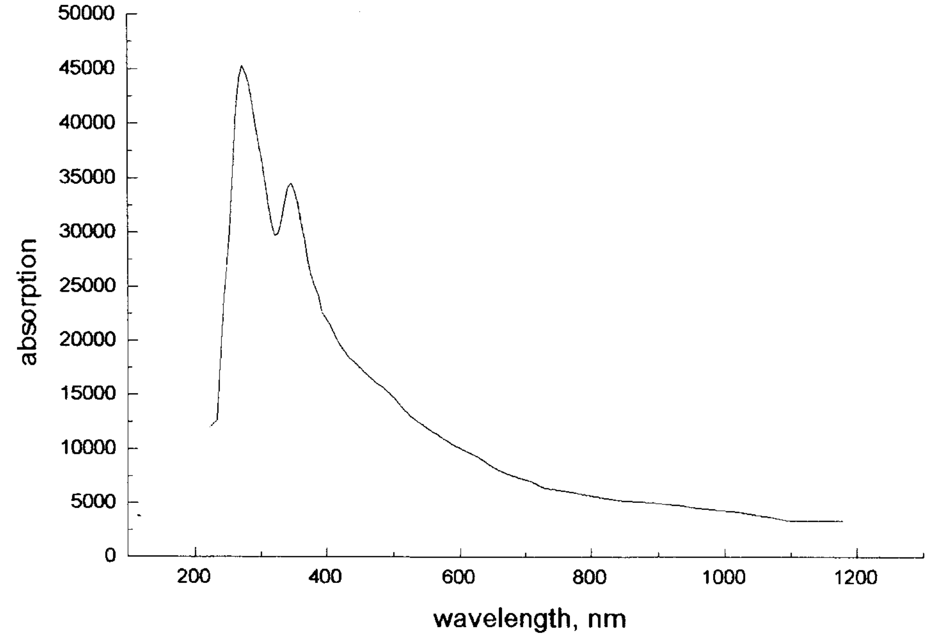

Fig. 5. Absorption spectrum of $C_{60}\cdot DBTTF\cdot C_6H_6$ single crystals in the 250–1100 nm range.

of C_{60} is hindered by a small overlapping of these orbitals, obviously due to the discrepancy between the DBTTF and C_{60} molecule shapes.

4. Conclusions

New complexes of fullerenes C_{60} and C_{70} with dibenzotetrathiafulvalene (DBTTF), DBTTF·C_{60}·C_6H_6, DBTTF·C_{60}·Py and DBTTF·C_{70}·C_6H_6, were obtained. The small shifts of S(2p) peaks in the XPS spectra of **1** together with the small shift of the DBTTF central C=C bond frequency (only 2 cm^{-1}) in the complexes show weak CT. It is attributed to weak donor properties of DBTTF (vertical ionization potential is 6.81 eV [21] and $E_{ox}(\mathbf{1}) = +0.62$ eV [22]). The consideration of DBTTF–C_{60} coordination shows a small HOMO–LUMO orbital overlapping. The π–π^* orbital overlapping is realized only through a coordination of the S_4C_2 fragment of DBTTF with the C_{60} pentagonal ring. It is obviously also one of the reasons for the small CT rate and for a weak intensity in the CT band in the Vis–NIR range. The DBTTF·C_{60}·C_6H_6 complex shows a unique arrangement of fullerene molecules which form approximately simple cubic packing with large distances between fullerene molecules. The electron state of these neutral complexes can be changed by doping with oxidant and reductant dopants. The doping of the DBTTF·C_{60}·C_6H_6 complex by iodine was carried out. Iodine substitutes benzene molecules, and DBTTF·C_{60}·$(C_6H_6)_xI_{3.5}$ and DBTTF·C_{60}·I_9 compounds were formed [23]. The donor molecule is oxidized in doping. The study of the doping of the DBTTF·C_{60}·C_6H_6 complex by alkali metals is in progress.

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