ELSEVIER

Synthetic Metals 131 (2002) 87–92

SYNTHETIC METALS

www.elsevier.com/locate/synmet

Synthesis and crystal structures of new C_{60} complexes with asymmetric tetrathiafulvalenes

Dmitri V. Konarev[a,b,*], Ivan S. Neretin[c], Yury L. Slovokhotov[c], Alexey L. Litvinov[b], Akihiro Otsuka[a,d], Rimma N. Lyubovskaya[b], Gunzi Saito[a,1]

[a] *Division of Chemistry, Graduate School of Science, Kyoto University, Sakyo-ku, Kyoto 606-8502, Japan*
[b] *Institute of Problems of Chemical Physics RAS, Chernogolovka 142432, Moscow Region, Russia*
[c] *Institute of Organoelement Compounds RAS (INEOS), 28 Vavilov Street, 119991 Moscow, Russia*
[d] *Research Center for Low Temperature and Materials Sciences, Kyoto University, Sakyo-ku, Kyoto 606-8502, Japan*

Received 21 May 2002; received in revised form 14 June 2002; accepted 19 July 2002

Abstract

New charge transfer complexes of fullerene C_{60} with two types of tetrathiafulvalenes: bis(4,5-dihydronaphtho[1,2-*d*])-tetrathiafulvalene (BDHN-TTF$(C_{60})_2C_7H_8$, **1**) and three bis(alkylthio)ethylenedithio-tetrathiafulvalenes (C_nTET-TTF$(C_{60})_x$, where $x = 2$, $n = 2$ (**2**); $x = 1.5$, $n = 4$ (**3**); $x = 3$, $n = 7$ (**4**)) were obtained. Compounds **1** and **2** were studied by single crystal X-ray diffraction. In both crystal structures, the three-dimensional (3D) packing of fullerene molecules formed by waved dense hexagonal layers was revealed. The donor molecules arranged in the fullerene framework attain bent conformation to fit to the spherical fullerene surface. Intermolecular contacts have typical van der Waals lengths. According to optical data, the complexes obtained have a neutral ground state with the charge transfer bands at 11×10^3 cm^{-1}.

Keywords: Fullerenes; Synthesis; Crystal structure; Tetrathiafulvalenes

1. Introduction

Fullerene derivatives are of great interest due to their intriguing physical and structural properties [1,2]. Although fullerene molecular complexes usually have neutral ground state [3,4], their further modification by photoinduced electron transfer [5], doping by alkali metals in gaseous phase [6], or with charge carriers using the field-induced transistor (FET) technology [7] may result in unusual magnetic and superconductive properties. Variation of the fullerene packing and related properties is possible as a result of the use of different donor molecules.

C_{60} and C_{70} fullerenes form molecular complexes with various tetrathiafulvalene (TTF) derivatives. The packing of fullerene molecules in these complexes ranges from a three-dimensional (3D) framework in (TMTSeF)$C_{60}(CS_2)_2$ [8] through planar layers in (TMTSeF)$C_{60}(C_6H_6)_{0.5}$ [9], (TMDTDM-TTF)$_2C_{60}(CS_2)_3$ [10], BET-TTF$(C_{60})C_7H_8$ [11], BEDT-TTF$(I_3)C_{60}$ [12], and 1D chains in OMTTF$(C_{60})C_6H_6$ [3], (BEDT-TTF)$_2C_{60}$ [13], *twin*-BEDT-TTF$(C_{60})CS_2$ [14] and (C_1TET-TTF)$_2C_{60}$ [15] to an isolated arrangement in DBTTF$(C_{60})C_6H_6$ [16] and BDMT-TTeF$(C_{60})CS_2$ [17]. TTF derivatives have already attracted attention as donor molecules for ion-radical salts possessing superconductivity and other interesting properties [18], and the above class of compounds is also promising from this viewpoint.

In this work, we present new complexes of C_{60} with two types of tetrathiafulvalenes: bis(4,5-dihydronaphtho[1,2-*d*])-tetrathiafulvalene (BDHN-TTF) and bis(alkylthio)ethylenedithio-tetrathiafulvalenes (C_nTET-TTF, where $n = 2$, 4, and 7) (Fig. 1). The IR- and UV-Vis–NIR spectra of the complexes are presented and the crystal structures of BDHN-TTF$(C_{60})_2C_7H_8$ (**1**) and C_2TET-TTF$(C_{60})_2$ (**2**) are described.

TMTSeF, tetramethyltetraselenafulvalene; TMDTDM-TTF, tetramethylenedithiodimethyl-tetrathiafulvalene; BET-TTF, bis(ethylenethio)-tetrathiafulvalene; BEDT-TTF, bis(ethylenedithio)-tetrathiafulvalene; OMTTF, octamethylenetetrathiafulvalene; *twin*-BEDT-TTF, bis(ethylenedithiotetrathiafulvaleno)[*b*,*h*]-1,4,7,10-tetrathiacyclododeca-2,8-diene; DBTTF, dibenzotetrathiafulvalene; C_1TET-TTF, bis(methylthio)ethylenedithio-tetrathiafulvalene; BDMT-TTeF, bis(dimethylthieno)-tetratellurafulvalene; TTM-TTF, tetrakis(methylthio)-tetrathiafulvalene

[*] Corresponding author. +7-2517-1852; fax: +7-96-515-3588.
E-mail address: konarev@icp.ac.ru (D.V. Konarev).

[1] Co-corresponding author.

0379-6779/02/$ – see front matter © 2002 Elsevier Science B.V. All rights reserved.
PII: S0379-6779(02)00156-X

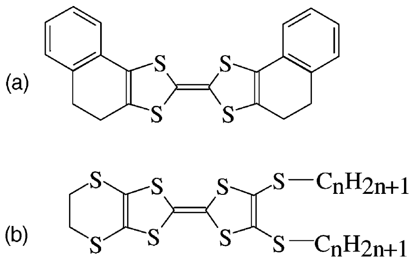


Fig. 1. The molecular structure of the donors: (a) BDHN-TTF; (b) C_nTET-TTF, where $n = 2$, 4, and 7.

2. Results and discussion

The molecular complexes BDHN-TTF$(C_{60})_2C_7H_8$ (**1**), C_2TET-TTF$(C_{60})_2$ (**2**), C_4TET-TTF$(C_{60})_{1.5}$ (**3**), and C_7TET-TTF$(C_{60})_3$ (**4**) were obtained by evaporation of toluene or benzene solutions containing C_{60} and corresponding TTF derivatives.

2.1. IR- and UV-Vis–NIR spectra

The IR spectra (KBr pellets) of **1–4** show that the frequencies and the ratio of intensities of the absorption bands attributed to C_{60} (527, 577, 1182, and 1429 cm^{-1}) remain unchanged with respect to parent C_{60} (± 1 cm^{-1}). Other bands (see Section 3) are attributed to the donor molecules. The shift of these bands up to 8 cm^{-1} relative to the parent donors may be associated with the changes in initial geometry of the donors due to crystal packing forces.

The UV-Vis–NIR spectrum of **2** is presented in Fig. 2. The UV-Vis–NIR spectra of the other complexes are similar. The main absorption bands of C_{60} at 37.5×10^3 and 29.1×10^3 cm^{-1} do not shift relative to initial fullerene. The band at 22.2×10^3 cm^{-1} may probably be attributed to intermolecular charge transfer excitations between the neighboring C_{60} molecules [19]. This band is observed in the spectrum of pure C_{60}, but is absent in the spectrum of dissolved C_{60} [20] or in the spectra of solid complexes with the isolated C_{60} molecules [4]. Relatively high intensity of this band in the spectra of **1–4** (Fig. 2) may reflect close contacts of fullerene molecules in the crystals.

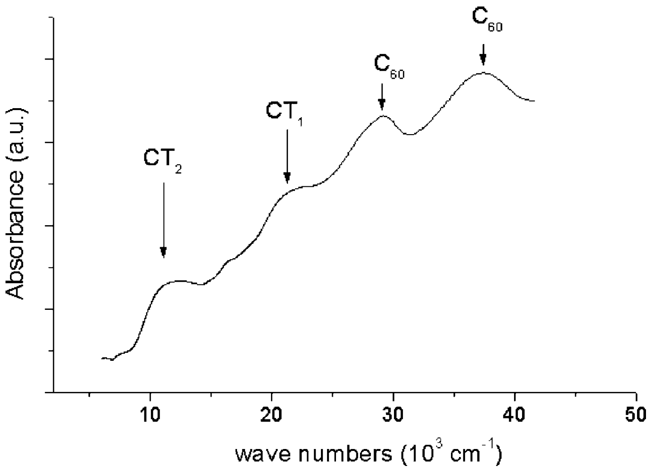


Fig. 2. The UV-Vis–NIR spectrum of C_2TET-TTF$(C_{60})_2$ (**2**) in the 5×10^3 to 42×10^3 cm^{-1} range.

A relatively weak band in the NIR range with the maximum at 11×10^3 cm^{-1} (Fig. 2, CT_2) may be ascribed to charge transfer from the C_2TET-TTF molecule to C_{60}. This band has the close position for **1–4** (± 100 cm^{-1}). Thus, the compounds obtained may be attributed to charge transfer complexes with a neutral ground state according to the optical spectroscopy data.

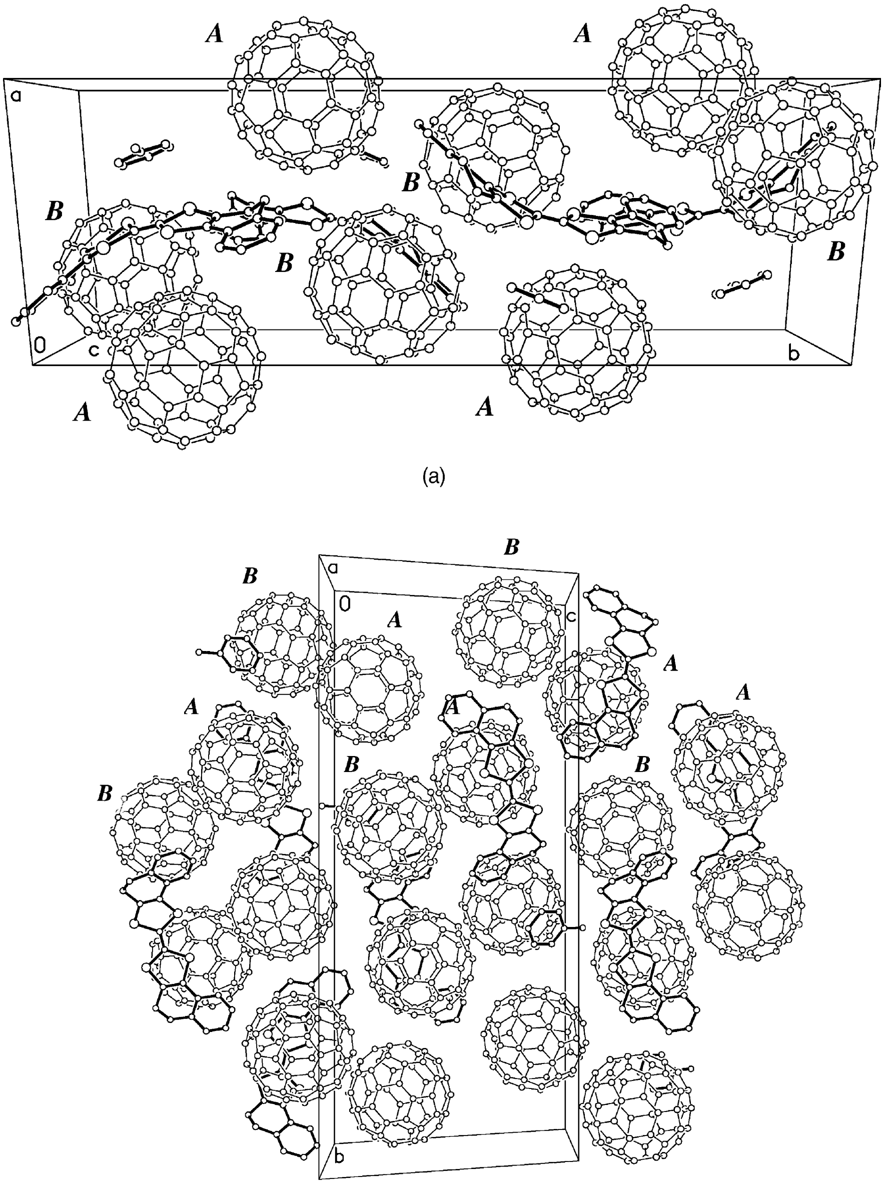
2.2. Crystal structures

In the monoclinic crystal structure of **1**, two crystallographically independent C_{60} molecules (***A*** and ***B*** in Fig. 3a and b), one BDHN-TTF molecule and one toluene (C_7H_8) molecule are located in general positions (Fig. 3a). Both fullerene molecules show no rotational disorder.

The fullerene packing may be described as a 3D framework composed of slightly puckered hexagonal layers parallel to the *bc*-plane (Fig. 3b). Each C_{60} molecule of the ***A***-type has seven neighbors, six of them within the same layer and one from the adjacent layer. Molecule ***B*** also has seven neighbors, however, five of them are within the same layer and two are from the adjacent one. Center-to-center distances lie in the range of 9.92–10.12 Å.

The BDHN-TTF molecule attains "boat" conformation in **1** (Fig. 3a and b). The dihedral angles between the central S_4C_2 fragment and the outer S_2C_2 planes are 15.6 and 20.9°. It is known that the TTF molecule and its alkyl- or benzo-substituted derivatives with no bulky substituents tend to be planar. Among more than 300 structures of the TTF derivatives in the Cambridge Structural Database, planar conformation obviously dominates. The average value of the dihedral angle is 7.9°. However, large and non-planar substituents may cause the TTF moiety to be bent. For example, bis- and tetrakis(alkylchalcogen)-substituted TTF derivatives have a tendency to form non-planar structures in the neutral state (φ for C_1TET-TTF in the 5.5–17.5° range [21] and φ for a non-planar phase of TTM-TTF are 19.3 and 23.7° [22]), while a planar one is in the charged state [22,23]. Bent conformation is typical for TTFs in the complexes with C_{60} (see Table 1) except for a few cases, e.g. neutral OMTTF$(C_{60})C_6H_6$ [3] and the three-component ionic complex BEDT-TTF$^{\bullet+}(I_3^-)C_{60}$ with the planar BEDT-TTF$^{\bullet+}$ radical cation [12]. This is caused by the tendency of the TTF molecules to fit to the spherical shape of C_{60} one due to packing forces.

Van der Waals contacts in BDHN-TTF(C_{60}) are 3.20–3.40 Å (C···C contacts) and 3.46–3.60 Å (S···C contacts), i.e. equal to or slightly shorter than the sum of van der Waals radii (3.42 Å for C···C and 3.57 Å for C···S) [24]. The C_7H_8 molecules are located in the cavities (Fig. 3a), their shortest C···C contacts with fullerene are 3.64 Å.



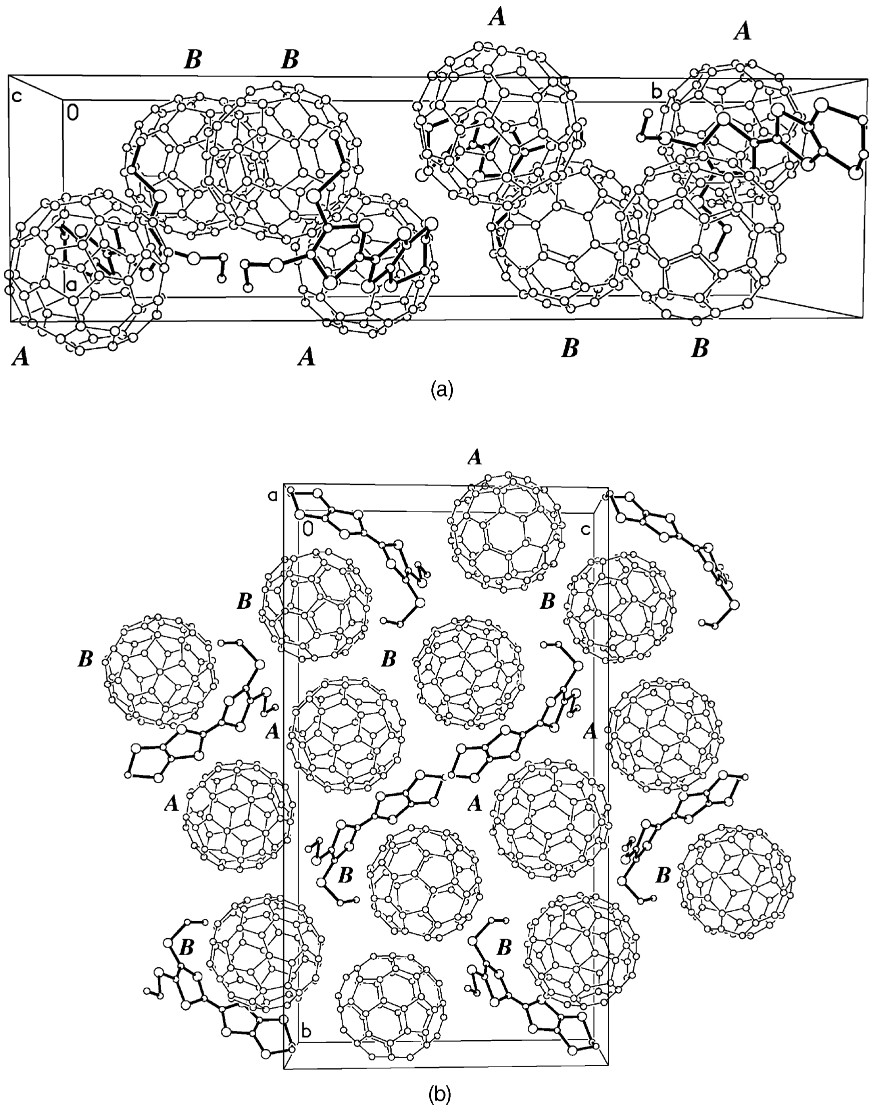
(a)

(b)

Fig. 3. Crystal structure of BDHN-TTF$(C_{60})_2C_7H_8$: (a) the projection along the *c*-axis; (b) the projection along the *a*-axis showing the puckered hexagonal layers of the C_{60} molecules.

Table 1
Dihedral angles φ in the TTF moiety (see Fig. 5 for the notations) and fullerene packing motif in the complex

Complex	Dihedral angles φ_1 and φ_2	Fullerene packing motif	Reference
BDHN-TTF$(C_{60})_2C_7H_8$ (**1**)	15.6, 20.9	3D framework	This work
C_2TET-TTF$(C_{60})_2$ (**2**)	13.9, 27.8	3D framework	This work
(TMTSeF)$C_{60}(CS_2)_2$	24.4, 23.4	3D framework	[8]
(TMTSeF)$C_{60}(C_6H_6)_{0.5}$	25.8, 28.3	Hexagonal layers	[9]
(TMDTDM-TTF)$_2C_{60}(CS_2)_3$	24.0, 24.9	Square layers	[10]
BEDT-TTF$(I_3)C_{60}$	6.9	Hexagonal layers	[12]
(BEDT-TTF)$_2C_{60}$	23.3, 33.6	1D double chains	[13]
OMTTF$(C_{60})C_6H_6$	10.6	1D chains in 2D layer	[3]
twin-BEDT-TTF$(C_{60})CS_2$	5.1, 11.1, 11.3, 20.1	1D double chains	[14]
DBTTF$(C_{60})C_6H_6$	25.3	Isolated	[16]



(a)

(b)

Fig. 4. Crystal structure of C_2TET-TTF$(C_{60})_2$: (a) the projection along the *a*-axis; (b) the projection along the *c*-axis. Only the major orientation of the disordered "***B***" C_{60} molecule is shown.

Monoclinic complex **2** is solvent-free. Two crystallographycally independent C_{60} and one C_2TET-TTF molecule occupy general positions in **2** (Fig. 4a). One independent C_{60} molecule is ordered (***A***), whereas the other one (***B***) is rotationally disordered (Fig. 4a and b). A complicated disorder pattern could not be resolved as a superposition of two orientations and results in a relatively high value of *R*-factor for the crystal structure of **2**. The C_2TET-TTF molecule is bent over the surface of the disordered "***B***" C_{60} molecule (Fig. 4b).

The fullerene molecular packing in **2** may be described as a 3D framework (Fig. 4a). The C_{60} molecule has seven (***A***) or six (***B***) neighbors with center-to-center distances for both C_{60} molecules in the range of 9.83–10.19 Å. The donor molecules are arranged in the 3D fullerene framework (Fig. 4b) and also have "boat" conformation (the dihedral angles are 13.9 and 27.8°).

Possibly such type of the 3D packing of fullerene molecules allows the formation of the C_{60} complexes with TTF with long alkyl chains (up to C_7H_{15}). It is interesting that C_1TET-TTF also forms a complex with fullerene [15]. However, this complex has a different composition: $(C_1\text{TET-TTF})_2C_{60}$ and its structure may be described as 1D double chains of the C_{60} molecules similar to those in (BEDT-TTF)$_2C_{60}$. Each pair of the C_{60} molecules in the complex is capped by two pairs of the C_1TET-TTF ones. Thus, the step from methyl to ethyl substituents in C_nTET-TTF drastically affects the crystal structure of the complex with C_{60}. Probably C_2TET-TTF is too long to arrange four molecules on the surface of the pair of C_{60}, thus, it is forced to pack in a different structural motif.

Geometry of the donor molecules in **1** and **2** is presented in Table 2. The TTM-TTF molecule is also included. The lengths of the central double C=C bond (bond 1 in Fig. 5)

Table 2
Averaged bond lengths in the TTF moiety (see Fig. 5 for the notations)

Bond number	**1**	**2**	Neutral TTM-TTF [22]	(TTM-TTF$^{\bullet +}$)IBr_2^- [25]
1	1.317 (6)	1.356 (11)	1.348 (5)	1.392 (13)
2	1.758 (2)	1.744 (4)	1.751 (4)	1.728 (10)
3	1.745 (2)	1.760 (4)	1.767 (4)	1.739 (10)
4	1.343 (4)	1.337 (8)	1.340 (4)	1.378 (14)

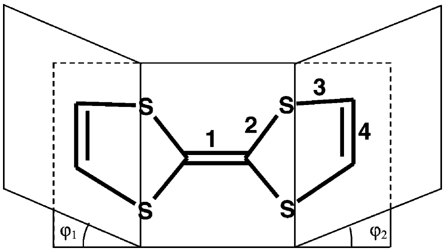


Fig. 5. Notation of the bonds and the dihedral angles in the TTF moieties for Tables 1 and 2.

and the C–S bonds (bonds 2 and 3 in Fig. 5), which depend on charge on the TTF molecule are closer rather to neutral TTM-TTF [22] than to the radical cation in ionic (TTM-TTF$^{\bullet +}$)IBr_2^- [25]. This demonstrates the absence of charge transfer in the ground state of **1** and **2** that is in a good agreement with the spectral data.

Thus, new complexes of C_{60} with two types of the asymmetric tetrathiafulvalenes: bis(4,5-dihydronaphtho[1,2-*d*])-tetrathiafulvalene (BDHN-TTF) and bis(alkylthio)ethylenedithio-tetrathiafulvalenes (C_nTET-TTF, $n = 2, 4$, and 7) were obtained. According to the optical data, all these complexes have a neutral ground state. The molecular packing in **1** and **2** involves different types of 3D frameworks of fullerene spheres with the cavities occupied by TTFs. The 3D fullerene packing makes these complexes interesting as potential precursors for the further modification (i.e. doping with alkali metals) to obtain ionic species.

3. Experimental

3.1. Materials

C_{60} of 99.0% purity (MTR Ltd.), BDHN-TTF (Aldrich) and C_nTET-TTF ($n = 2$, 4, and 7) [21] were used for syntheses of **1**–**4**. Toluene (C_7H_8) and benzene (C_6H_6) were distilled over Na/benzophenone in argon atmosphere.

3.2. Synthesis

The crystals of BDHN-TTF$(C_{60})_2C_7H_8$ (**1**) were obtained by slow evaporation of the toluene solution of BDHN-TTF and C_{60} at a 2:1 molar ratio. The crystals of C_nTET-TTF$(C_{60})_x$ (where $x = 2$, $n = 2$ (**2**); $x = 1.5$, $n = 4$ (**3**); $x = 3$, $n = 7$ (**4**)) were obtained by the slow evaporation of benzene solution containing C_nTET-TTF and C_{60} at a 1:1 molar ratio. The solvent was decanted from the crystals and they were washed with acetone giving black parallelograms (**1**) or needle like crystals (**2**–**4**) with 50–70% yield. The composition of the complexes was determined from the X-ray diffraction data (**1** and **2**) and by the elemental analysis (**1**–**4**).

BDHN-TTF$(C_{60})_2C_7H_8$ (**1**). Calcd: C, 92.18; H, 1.23; S, 6.59. Found: C, 91.86; H, 1.14; S, 6.84. The IR spectrum: (C_{60}) 527, 577, 1182, 1428; (BDHN-TTF) 430, 601, 640, 717, 751, 778, 888, 931, 977, 1039, 1121, 1267, 1449, 1485, 1561, 1598 cm^{-1}.

C_2TET-TTF$(C_{60})_2$ (**2**). Calcd: C, 85.44; H, 0.59; S, 13.43. Found: C, 85.46; H, 0.75; S, 13.79. The IR spectrum: (C_{60}) 526, 576, 1182, 1427; (C_2TET-TTF) 727, 779, 884, 913, 1215, 1254, 1283, 1371, and 1405 cm^{-1}.

C_4TET-TTF$(C_{60})_{1.5}$ (**3**). Calcd: C, 82.08; H, 1.41; S, 16.51. Found: C, 82.14; H, 1.47; S, 15.89. The IR spectrum: (C_{60}) 526, 577, 1182, 1428; (C_4TET-TTF) 460, 725, 774, 803, 878, 909, 1215, 1259, 1376, 1405, and 1459 cm^{-1}.

C_7TET-TTF$(C_{60})_3$ (**4**). Calcd: C, 88.95; H, 1.60; S, 9.56. Found: C, 89.33; H, 1.25; S, 9.42. The IR spectrum: (C_{60}) 526, 577, 1182, 1428; (C_7TET-TTF) 670, 722, 774, 890, 910, 1374, 1405, and 1458 cm^{-1}.

The UV-Vis–NIR spectra were measured on a Shimadzu-3100 spectrometer in the 240–2600 nm range. The FT-IR spectra were measured in KBr pellets with a Perkin-Elmer 1000 Series spectrometer (400–7800 cm^{-1}).

The X-ray diffraction data were collected on a Bruker SMART diffractometer with an area detector in the Centre for X-ray Structural Study, INEOS. Both structures were solved with direct methods and refined with the full-matrix least squares on F^2 using the SHELXTL-97 program package [26]. Disordered C_{60} moieties in **2** (molecules ***B***) were refined as a superposition of two orientations with a restrained bond length. Occupancy ratio is 58:42. Hydrogen atoms were placed in geometrically calculated positions and refined within the ‘riding atom’ model. All non-hydrogen atoms except those in the disordered moieties were refined in anisotropic approximation.

The crystal data for BDHN-TTF$(C_{60})_2C_7H_8$ (**1**) are: $C_{149}H_{24}S_4$, $M = 1941.92$, monoclinic, space group $P2_1/c$, $a = 13.647$ (3), $b = 36.37$ (1), $c = 16.953$ (4) Å, $\beta = 113.221$ (7)°, $V = 7732.6$ (2) Å^3, $Z = 4$, $D_c = 1.668$ g/cm^3, Mo Kα radiation (a graphite monochromator, $\lambda = 0.71073$ Å), $\mu = 0.20$ mm^{-1}, $T = 110$ K; 22565 unique reflections with $2\theta_{max} = 60°$ were collected, 8686 of them observed ($F > 4\sigma$). Finally, $R_1 = 0.0989$, $R_w = 0.2886$ (1378 parameters), and GOF = 0.875 were attained.

The crystal data for C_2TET-TTF$(C_{60})_2$ (**2**) are: $C_{132}H_{41}S_8$, $M = 1855.91$, monoclinic, space group $P2_1/c$, $a = 10.027$ (5), $b = 35.24$ (1), $c = 19.691$ (7) Å, $\beta = 93.92$ (3)°, $V = 6941.2$ (2) Å^3, $Z = 4$, $D_c = 1.776$ g/cm^3, Mo Kα radiation (a graphite monochromator, $\lambda = 0.71073$ Å), $\mu = 0.33$ mm^{-1}, $T = 110$ K; 15944 unique reflections with $2\theta_{max} = 55°$ were collected, 7306 of them observed ($F > 4\sigma$). Finally, $R_1 = 0.136$, $R_w = 0.397$ (1202 parameters), and GOF = 1.031 were attained.

Acknowledgements

The authors are grateful to Prof. M. Antipin for the access to the single crystal X-ray diffraction facility. The work was supported by the COE Research on Elemental Science No. 12CE2005, JSPS and by RFBR (Grant Nos. 00-03-32577a, 01-03-06345, 02-03-33225 and 01-03-33009).

References

[1] C.A. Reed, R.D. Bolskar, Chem. Rev. 100 (2000) 1075.

[2] D.V. Konarev, R.N. Lyubovskaya, Russ. Chem. Rev. 68 (1999) 19.

[3] G. Saito, T. Teramoto, A. Otsuka, Y. Sugita, T. Ban, M. Kusunoki, K. Sakaguchi, Synth. Met. 64 (1994) 359.

[4] D.V. Konarev, R.N. Lyubovskaya, N.V. Drichko, E.I. Yudanova, Yu.M. Shul'ga, A.L. Litvinov, V.N. Semkin, B.P. Tarasov, J. Mater. Chem. 10 (2000) 803.

[5] D.V. Konarev, G. Zerza, M. Sharber, N.S. Sariciftci, R.N. Lyubovskaya, Synth. Met. 121 (2001) 1127.

[6] A. Otsuka, G. Saito, S. Hirate, S. Pac, T. Ishida, A.A. Zakhidov, K. Yakushi, in: Proceedings of the Materials Research Society Symposium, vol. 488, Materials Research Society, 1998, p. 495.

[7] J.H. Schön, Ch. Kloc, B. Batlogg, Science 293 (2001) 2432.

[8] S.V. Konovalikhin, O.A. D'yachenko, G.V. Shilov, N.G. Spitsina, K.V. Van, E.B. Yagubskii, Izv. Akad. Nauk Ser. Khim. (1997) 1480.

[9] S.V. Konovalikhin, O.A. D'yachenko, G.V. Shilov, N.G. Spitsina, E.B. Yagubskii, Fullerene Sci. Technol. 6 (1998) 563.

[10] D.V. Konarev, E.F. Valeev, Yu.L. Slovokhotov, Yu.M. Shul'ga, O.S. Roschupkina, R.N. Lyubovskaya, Synth. Met. 88 (1997) 85.

[11] J. Llacay, J. Tarres, J. Veciana, C. Rovira, Synth. Met. 70 (1995) 1453.

[12] D.V. Konarev, A.Yu. Kovalevsky, P. Coppens, R.N. Lyubovskaya, J. Chem. Soc., Chem. Commun. (2000) 2357.

[13] A. Izuoka, T. Tachikawa, T. Sugawara, Y. Suzuki, M. Konno, Y. Saito, H. Shinohara, J. Chem. Soc., Chem. Commun. (1992) 1472.

[14] A. Izuoka, T. Tachikawa, T. Sugawara, Y. Saito, H. Shinohara, Chem. Lett. (1992) 1049.

[15] Y. Li, Y. Gao, F. Bai, Y. Mo, B. Zhang, H. Han, D. Zhu, Synth. Met. 70 (1995) 1459.

[16] D.V. Konarev, Y.V. Zubavichus, Yu.L. Slovokhotov, Yu.M. Shul'ga, V.N. Semkin, N.V. Drichko, R.N. Lyubovskaya, Synth. Met. 92 (1998) 1.

[17] P. Wang, W.-J. Lee, I. Scherbakova, M.P. Cava, R.M. Metzger, Synth. Met. 64 (1994) 319.

[18] T. Ishiguro, K. Yamaji, G. Saito, Organic Superconductors, 2nd ed., Springer, Berlin, 1998.

[19] M. Ichida, A. Nakamura, H. Shinohara, Y. Saito, Chem. Phys. Lett. 289 (1998) 579.

[20] M.S. Dresselhaus, G. Dresselhaus, P.C. Eclund, J. Mater. Res. 8 (1993) 2054.

[21] A. Otsuka, G. Saito, K. Ohfuchi, M. Konno, Phos. Sulfur Silicon 67 (1992) 333.

[22] C. Katayama, M. Honda, H. Kumagai, J. Tanaka, G. Saito, H. Inokuchi, Bull. Chem. Soc. Jpn. 58 (1985) 2272.

[23] A. Otsuka, G. Saito, T. Sugano, M. Kinoshita, Thin Solid Films 179 (1989) 259.

[24] A. Bondi, J. Phys. Chem. 70 (1966) 3006.

[25] K. Honda, M. Goto, M. Kurahashi, H. Anzai, M. Tokumoto, T. Ishiguro, Bull. Chem. Soc. Jpn. 61 (1988) 588.

[26] SHELXTL, An integrated system for solving, refining and displaying crystal structures from diffraction data, Version 5.10, Bruker Analytical X-ray Systems, Madison, WI, USA, 1997.