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Crystal Structures of Room- and Low-Temperature (170 K) Molecular Complexes between Fullerene and 2,2',6,6'-Tetraphenyldipyranylidene, $TPDP(C_{60})_2(CS_2)_4$

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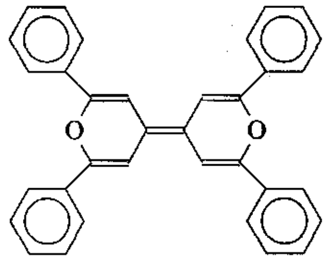
Abstract—The single-crystal X-ray structure of $TPDP(C_{60})_2(CS_2)_4$ is determined at room temperature (293 K; $a = 13.199$, $b = 15.592$, and $c = 22.199$ Å; $\alpha = 102.87°$; and $V = 4453.8$ Å^3) and at low temperature (170 K; $a = 13.050$, $b = 15.510$, and $c = 22.010$ Å; $\alpha = 102.87°$; $V = 4343.0$ Å^3; sp. gp. $I2/m$; and $Z = 2$). A characteristic feature of the structure is the alternation of the layers consisting of C_{60} and CS_2 molecules with the donor layers along the c direction.

INTRODUCTION

Although fullerenes have been intensively studied since their discovery in 1985 [1], there still remain many unsolved problems relating to the properties of fullerene-based materials. Initially, most attention was concentrated on the properties of isolated fullerene molecules (C_{60}, C_{70}, etc.), but the discovery of superconductivity in the M_3C_{60} salts (where M is an alkali metal) [2] and ferromagnetism in TDAE · C_{60} [3] [where TDAE is tetrakis(dimethylamino)ethylene] aroused considerable interest in the preparation of new materials based on C_{60} and investigation of their solid-state properties. In particular, a number of charge-transfer complexes with organic and organometallic donors [4–7] and radical ion salts with the C_{60}^- monoanion, such as $[(C_6H_5)_4P^+]_2C_{60}^-X^-$ (X = I or Cl) [8, 9] or $[Ni(C_5Me_5)_2]$ + $C_{60}^-CS_2$ [10], and also with the C_{60}^{2-} dianion, $(PPN^+)_2C_{60}^{2-}$ [where PPN^+ is the bis(triphenylphosphine)iminium cation] [11], were obtained as single crystals.

However, there are only a few works focusing on both the complete structural characterization of the C_{60} molecular complexes and their single-crystal properties. The main reason for this is the problem of molecular disorder. This specific property is inherent in most materials based on C_{60} and its analogues because of their high molecular symmetry and weak intermolecular interactions.

Recently, we investigated single crystals of the C_{60} and C_{70} molecular complexes with an organic Te-containing donor—9.9'-*trans*-bis(telluraxanthenyl) [12, 13]. In this paper, we report the results of a single-crystal X-ray structure investigation of a new C_{60} molecular complex with 2,2',6,6'-tetraphenyldipyranylidene (TPDP) as a donor—$TPDP(C_{60})_2(CS_2)_4$.



TPDP

The preparation of crystals of this complex was described in [14, 15].[1]

EXPERIMENTAL

The intensities were measured on an Enraf–Nonius CAD-4 automated diffractometer (MoK_α radiation, graphite monochromator, ω scan mode). Two experimental-data sets were collected at 293 and 170 K (with a liquid nitrogen cooling device) up to $(\sin\theta/\lambda)_{max} = 0.481$ and 0.594, respectively. The sets contained 1709 and 2706 unique reflections with $I \geq 4\sigma(I)$.

Main crystal data for the structure of $TPDP(C_{60})_2(CS_2)_4$ are presented in Table 1. We failed to solve the room-temperature structure. The solution of the structure was complicated by the ambiguous choice of the space group—Im, $I2$, or $I2/m$. The model of the structure was obtained from the low-temperature

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[1] The initial donor TPDP was obtained at the Mendeleev University of Chemical Technology by T.A. Chibisova *et al.*

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Table 1. Crystal data for TPDP $(C_{60})_2(CS_2)_4$ at low (170 K) and room temperatures [$C_{158}H_{24}O_2S_8$, $F(000) = 2232$, $M = 2210.05$, sp. gr. $I2/m$; and $Z = 2$]

Parameter	170 K	293 K
a, Å	13.050(3)	13.199(3)
b, Å	15.510(4)	15.592(4)
c, Å	22.010(6)	22.199(6)
α, deg	102.87(2)	102.87(2)
V, Å^3	4343.0	4453.8
d_{calcd}, g/cm^3	1.69	1.65
μ, cm^{-1}	28.1	27.4

data by the direct method with the AREN program [16]. The positions of hydrogen atoms were calculated geometrically. The least-squares refinement of the structure was performed in the anisotropic–isotropic (for H atoms) approximation with the SHELXL93 program [17] to $R = 0.049$ (170 K) and $R = 0.062$ (293 K). A total of 397 parameters was refined. No fundamental differences in the structure of the complex are observed when the temperature decreases from 293 to 170 K. The final coordinates and equivalent thermal parameters of non-hydrogen atoms are listed in Table 2.

Figure 1 shows the projection of the TPDP$(C_{60})_2(CS_2)_4$ structure onto the *bc* plane. The C_{60} and CS_2 molecules are situated in the *m* symmetry planes, and the TPDP molecules occupy the $2/m$ positions. A characteristic feature of the structure is the alternation of the layers formed by C_{60} and CS_2 molecules with the layers of donor TPDP molecules along the *c* direction. The short C···C and S···C contacts between the acceptor and donor layers are given in Table 3.

Note that six of ten C_{60}···TPDP intermolecular contacts occur between the phenyl ring of TPDP and the C(8), C(14), C(15), and C(27) atoms of a six-membered ring in C_{60}, and the remaining four contacts are formed with the C(18), C(19), and C(20) atoms of a five-membered ring in C_{60}. The small values of the corresponding dihedral angles (~25° and 19°) possibly indicate the π–π-character of the interactions between the phenyl rings in TPDP and the π-system of fullerene. The directions of the interactions between C_{60} and TPDP are schematically represented by arrows in Fig. 1.

The projection of an acceptor layer along the *c* direction is shown in Fig. 2. Molecules of C_{60} and CS_2 are in staggered order. Each cavity between the

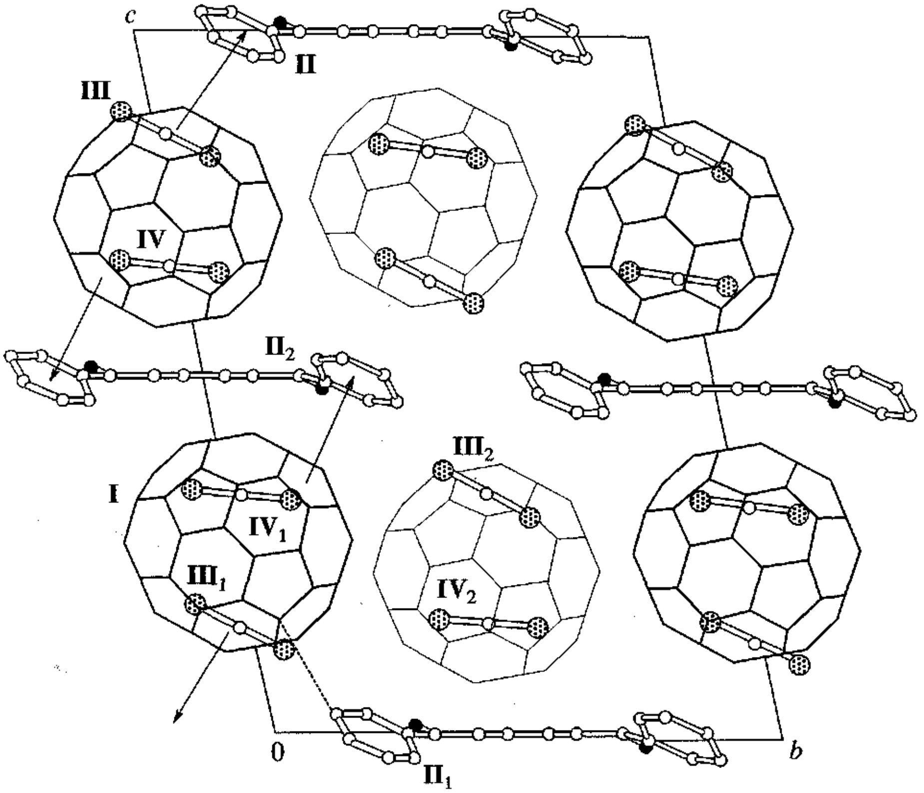

Fig. 1. The projection of the structure onto the *bc* plane. Designations of molecules and symmetry operations are as follows: C_{60} **I** (x, y, z); TPDP **II** (x, y, z), **II**$_1$ $(x, y, z-1)$, and **II**$_2$ $(1/2-x, 1/2-y, 3/2-z)$; CS_2 **III** and **IV** $(0, y, z)$, **III**$_1$ and **IV**$_1$ $(0, -y, 1-z)$, and **III**$_2$ and **IV**$_2$ $(1/2, y+1/2, z-1/2)$.

Table 2. Atomic coordinates and isotropic equivalent thermal parameters U_{eq} (Å^2) at 170 K (the first row) and 293 K (the second row)

Atom	x/a	y/b	z/c	U_{eq}	Atom	x/a	y/b	z/c	U_{eq}
		C_{60}			C(25)	0.4099(3)	−0.1786(2)	0.3220(2)	0.042(1)
C(1)	0.2341(3)	−0.0183(3)	0.2890(2)	0.049(1)		0.4103(8)	−0.1767(5)	0.3221(4)	0.104(3)
	0.2386(7)	−0.0190(8)	0.2877(9)	0.134(5)	C(26)	0.4442(3)	−0.2206(2)	0.2595(2)	0.034(1)
C(2)	0.2682(3)	0.0224(3)	0.3512(2)	0.052(1)		0.4455(6)	−0.2187(4)	0.2601(4)	0.079(2)
	0.2724(9)	0.0224(9)	0.3500(8)	0.133(4)	C(27)	0.4450(3)	−0.0647(2)	0.1062(2)	0.045(1)
C(3)	0.3235(4)	−0.0431(3)	0.3764(2)	0.053(1)		0.4450(4)	−0.0667(8)	0.1092(3)	0.119(4)
	0.3257(11)	−0.0438(9)	0.3753(6)	0.136(5)	C(28)	0.4096(4)	0.0275(3)	0.1202(2)	0.045(1)
C(4)	0.3238(3)	−0.1253(2)	0.3288(2)	0.047(1)		0.4112(9)	0.0250(7)	0.1224(4)	0.107(3)
	0.3268(9)	−0.1252(7)	0.3285(6)	0.122(4)	C(29)	0.5000(0)	0.0830(4)	0.1281(2)	0.044(1)
C(5)	0.2690(3)	−0.1108(2)	0.2754(2)	0.045(1)		0.5000(0)	0.0806(9)	0.1295(6)	0.122(5)
	0.2735(7)	−0.1107(7)	0.2745(7)	0.121(4)	C(30)	0.5000(0)	0.1616(3)	0.1726(3)	0.043(1)
C(6)	0.2348(3)	0.0281(3)	0.2430(2)	0.047(1)		0.5000(0)	0.1572(8)	0.1727(5)	0.090(4)
	0.2401(7)	0.0277(8)	0.2442(8)	0.138(5)	C(31)	0.4098(3)	0.1878(2)	0.2110(2)	0.041(1)
C(7)	0.2691(3)	−0.0128(3)	0.1815(2)	0.048(1)		0.4111(7)	0.1850(5)	0.2118(4)	0.090(3)
	0.2738(8)	−0.0150(8)	0.1821(6)	0.116(3)	C(32)	0.4447(3)	0.2303(2)	0.2734(2)	0.052(1)
C(8)	0.3248(3)	0.0521(3)	0.1563(2)	0.047(1)		0.4451(6)	0.2274(4)	0.2739(4)	0.078(2)
	0.3277(9)	0.0517(8)	0.1588(5)	0.116(4)			TPDP		
C(9)	0.3241(3)	0.1347(2)	0.2032(2)	0.044(1)	C(33)	0.5000(0)	0.4553(3)	0.9996(3)	0.029(1)
	0.3281(9)	0.1336(6)	0.2052(6)	0.123(4)		0.5000(0)	0.4560(5)	0.9999(4)	0.046(2)
C(10)	0.2686(3)	0.1203(2)	0.2570(2)	0.047(1)	C(34)	0.4071(3)	0.4037(2)	0.9993(1)	0.031(1)
	0.2739(7)	0.1199(8)	0.2594(8)	0.123(4)		0.4078(5)	0.4046(3)	0.9990(2)	0.047(1)
C(11)	0.3008(3)	−0.1500(2)	0.2166(2)	0.042(1)	C(35)	0.4094(3)	0.3187(2)	1.0030(1)	0.027(1)
	0.3053(7)	−0.1489(6)	0.2169(7)	0.113(3)		0.4104(4)	0.3199(4)	1.0020(2)	0.042(1)
C(12)	0.3917(3)	−0.2064(2)	0.2078(2)	0.036(1)	C(36)	0.3205(3)	0.2604(2)	1.0001(1)	0.028(1)
	0.3934(7)	−0.2056(4)	0.2100(4)	0.091(3)		0.3228(4)	0.2603(3)	0.9994(2)	0.040(1)
C(13)	0.4473(3)	−0.1918(2)	0.1546(2)	0.038(1)	C(37)	0.2329(3)	0.2730(2)	0.9656(1)	0.031(1)
	0.4482(6)	−0.1915(4)	0.1573(4)	0.091(3)		0.2352(5)	0.2732(4)	0.9669(3)	0.051(2)
C(14)	0.3916(3)	−0.1264(2)	0.1298(2)	0.043(1)	C(38)	0.1516(3)	0.2164(2)	0.9600(2)	0.034(1)
	0.3940(10)	−0.1256(6)	0.1320(4)	0.113(3)		0.1542(5)	0.2184(4)	0.9619(3)	0.054(2)
C(15)	0.3009(3)	−0.0999(2)	0.1676(2)	0.042(1)	C(39)	0.1539(3)	0.1455(2)	0.9897(2)	0.038(1)
	0.3050(8)	−0.0978(7)	0.1686(6)	0.112(3)		0.1585(5)	0.1481(4)	0.9911(3)	0.063(2)
C(16)	0.3012(3)	0.1597(2)	0.3159(2)	0.044(1)	C(40)	0.2368(3)	0.1319(2)	1.0241(2)	0.037(1)
	0.3050(7)	0.1578(6)	0.3172(8)	0.123(4)		0.2419(5)	0.1334(4)	1.0234(4)	0.061(2)
C(17)	0.3914(3)	0.2161(2)	0.3250(2)	0.039(1)	C(41)	0.3210(3)	0.1889(2)	1.0293(2)	0.035(1)
	0.3936(8)	0.2142(5)	0.3233(5)	0.102(3)		0.3246(5)	0.1900(4)	1.0278(3)	0.056(2)
C(18)	0.4468(3)	0.2012(2)	0.3782(2)	0.045(1)	O	0.5000(0)	0.2747(2)	1.0093(2)	0.031(1)
	0.4478(7)	0.1996(5)	0.3762(5)	0.108(4)		0.5000(0)	0.2761(3)	1.0077(3)	0.048(1)
C(19)	0.3905(4)	0.1359(3)	0.4031(2)	0.052(1)			CS_2		
	0.3933(13)	0.1347(7)	0.4021(5)	0.145(5)	C(42)	0.0000(0)	0.0165(3)	0.8517(2)	0.032(1)
C(20)	0.3011(3)	0.1095(3)	0.3649(2)	0.050(1)		0.0000(0)	0.0176(6)	0.8542(4)	0.059(2)
	0.3045(10)	0.1053(8)	0.3645(7)	0.132(4)	S(1)	0.0000(0)	0.0950(1)	0.8200(1)	0.041(0)
C(21)	0.4451(4)	0.0737(3)	0.4271(2)	0.054(1)		0.0000(0)	0.0944(2)	0.8231(1)	0.080(1)
	0.4445(4)	0.0744(9)	0.4253(3)	0.146(6)	S(2)	0.0000(0)	−0.0625(1)	0.8833(1)	0.066(0)
C(22)	0.4092(4)	−0.0186(3)	0.4136(2)	0.055(1)		0.0000(0)	−0.0611(2)	0.8852(2)	0.135(1)
	0.4091(12)	−0.0175(8)	0.4122(5)	0.126(4)	C(43)	0.0000(0)	−0.0240(3)	0.6589(2)	0.032(1)
C(23)	0.5000(0)	−0.0739(4)	0.4054(2)	0.054(2)		0.0000(0)	−0.0271(6)	0.6603(4)	0.054(2)
	0.5000(0)	−0.0722(14)	0.4057(7)	0.149(7)	S(3)	0.0000(0)	0.0729(1)	0.6518(1)	0.038(0)
C(24)	0.5000(0)	−0.1526(4)	0.3604(2)	0.045(1)		0.0000(0)	0.0698(2)	0.6532(1)	0.073(1)
	0.5000(0)	−0.1494(10)	0.3614(6)	0.117(6)	S(4)	0.0000(0)	−0.1201(1)	0.6671(1)	0.043(0)
						0.0000(0)	−0.1214(2)	0.6673(2)	0.084(1)

Table 3. Short intermolecular C···C ($r \leq 3.60$ Å), S···C ($r \leq 3.65$ Å), and S···S ($r \leq 3.70$ Å) contacts at 170 K (the first row) and 293 K (the second row)

Contact	r, Å	Molecules*	Contact	r, Å	Molecules*
C_{60}···TPDP					
C(8)···C(40)	3.595(6) –	**I–II**$_1$	C(14)···C(36)	3.272(4) 3.328(9)	**I–II**$_3$
C(18)···C(39)	3.570(4) –	**I–II**$_2$	C(14)···C(37)	3.427(4) 3.501(11)	**I–II**$_3$
C(19)···C(37)	3.335(5) 3.397(13)	**I–II**$_2$	C(14)···C(41)	3.541(5) 3.585(10)	**I–II**$_3$
C(19)···C(38)	3.399(4) 3.417(11)	**I–II**$_2$	C(15)···C(38)	3.563(5) –	**I–II**$_3$
C(20)···C(36)	3.569(5) –	**I–II**$_2$	C(27)···C(41)	3.559(4) 3.586(9)	**I–II**$_3$
C_{60}···CS_2					
C(1)···S(3)	3.499(4) 3.568(12)	**I–IV**$_1$	C(26)···S(3)	3.609(4) –	**I**$_3$–**IV**$_3$
C(1)···C(43)	3.278(4) 3.376(10)	**I–IV**$_1$	C(30)···S(4)	3.414(5) 3.482(12)	**I–IV**$_2$
C(2)···C(43)	3.508(4) 3.604(12)	**I–IV**$_1$	C(31)···S(4)	3.532(4) 3.577(9)	**I–IV**$_2$
C(7)···C(42)	3.585(4) –	**I–III**$_1$	C(32)···S(2)	3.639(3) –	**I–III**$_2$
C(26)···S(1)	3.489(4) 3.574(8)	**I–III**$_3$			
TPDP···CS_2					
C(33)···S(3)	3.263(5) 3.331(8)	**II**$_2$–**IV**	C(33')···C(43)	3.447(7) 3.513(12)	**II**$_2$–**IV**
C(33')···S(3)	3.438(4) 3.470(7)	**II**$_2$–**IV**	C(34)···S(3)	3.632(3) –	**II**$_2$–**IV**
CS_2···CS_2					
S(1)···S(3)	3.642(2) –	**III–IV**	S(1)···C(43)	3.615(4) –	**III–IV**

* Symmetry operations for C_{60}, TPDP, and CS_2 molecules are also given in the caption to Fig. 1, for **II**$_3$ ($x, -y, 1-z$), and for **III**$_3$ and **IV**$_3$ ($1/2, y-1/2, z-1/2$).

fullerene molecules contains two CS_2 molecules. The in-layer distances between the centers of gravity of the C_{60} molecules are 10.152 and 10.170 Å at 170 K and 10.203 and 10.285 Å at 293 K. These distances are longer than those observed in the cubic structure of C_{60} at 110 K (9.936 Å) [18] and 293 K (10.041 Å) [19]. The shortest interlayer distances between the centers of the C_{60} molecules are 10.292 and 11.720 Å at 170 K and 10.329 and 11.871 Å at 293 K. Short contacts between the fullerene molecules are absent: all the intermolecular C···C distances exceed 3.60 Å (the sum of the van der Waals radii of carbon atoms). Several short contacts between the solvent CS_2 and C_{60} molecules within the acceptor layer are given in Table 3 and shown by dotted lines in Fig. 2.

Because of considerable atomic thermal vibrations at room temperature, the bond lengths are estimated with low accuracy, which makes their detailed discussion impossible. For this reason, only the low-temperature data are used in analysis of interatomic distances in the fullerene and donor molecules. Note that, with a decrease in temperature, the atomic thermal parameters (Table 2) decrease by approximately half for the donor and solvent molecules and by approximately two thirds for the fullerene molecule.

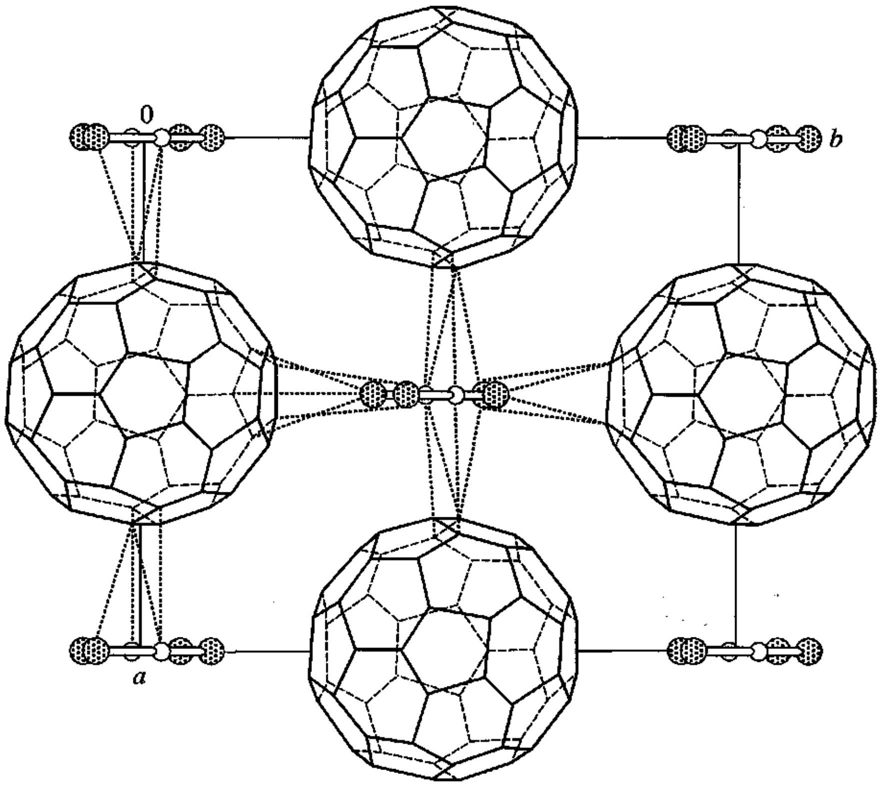

Fig. 2. The projection of the acceptor layer along the *c* direction.

The C–C bonds in the six-membered rings (6 : 6) of the fullerene molecule vary from 1.366(6) to 1.394(6) Å, and, in the five-membered rings (6 : 5), they fall in the range 1.432(6)–1.472(6) Å. Table 4 presents the mean bond lengths in the six-membered and five-membered rings of fullerene in the studied molecular complex $TPDP(C_{60})_2(CS_2)_4$ and the corresponding values for free fullerene and a number of molecular complexes. Note that all the crystal structure data [4, 10, 11, 18] given in this table were obtained at low temperatures, and their accuracies allow for bond-length analysis. It can be seen that, in going from the neutral molecule C_{60} [18] to the monoanion C_{60}^{-} [10] and the dianion C_{60}^{2-} [11], the bond lengths in the six-membered rings (6 : 6) tend to increase, and in the five-membered rings (6 : 5), to decrease. The bond lengths in $TPDP(C_{60})_2(CS_2)_4$ are close to the corresponding values for the charge-transfer complex $[Fe(C_5H_5)_2]C_{60}$ [9].

It is of interest to elucidate geometric changes in the donor TPDP molecule upon its incorporation into a molecular complex. Figure 3 shows the TPDP molecule with atomic numbering, and Table 5 lists the bond lengths and angles in TPDP. Examination of the bond lengths in TPDP demonstrates that their distribution is closely similar to that in the neutral $TPDP^0$ molecule [20]. For example, the length of the central C=C' bond [1.384(9) Å] coincides with that observed in free $TPDP^0$ [1.385(8) Å], whereas it is well known that this bond is most sensitive to an increase in the charge on the molecule. Indeed, for the sulfur analogue of TPDP—tetraphenyldithiapyranylidene [21]—an increase in charge from 0 to +1 in its polyiodides is accompanied by the lengthening of the central bond from 1.389 to 1.451 Å [22]. However, the molecular conformation of TPDP in the complex differs essentially from the conformation of $TPDP^0$. The pyranylidene ring *A* exhibits a boat conformation with the C(33) and O atoms displaced by 0.053 and 0.093 Å from the plane through the remaining four carbon atoms. For $TPDP^0$, the corresponding values are 0.021 and 0.045 Å, respectively. The angle of folding along

Table 4. Bond lengths in the six-membered (6 : 6) and five-membered (6 : 5) rings of fullerene and its molecular complexes

Complex	6 : 6	6 : 5	References
C_{60}	1.355(9)	1.467(2)	[18]
$[Fe(C_5H_5)_2]C_{60}$	1.387(6)	1.450(6)	[4]
$[Ni(C_5Me_5)_2^+](C_{60}^-)CS_2$	1.389(3)	1.449(3)	[10]
$[PPN^+]_2(C_{60}^{2-})$	1.399(2)	1.446(2)	[11]
$TPDP(C_{60})_2(CS_2)_4$	1.381(6)	1.451(6)	

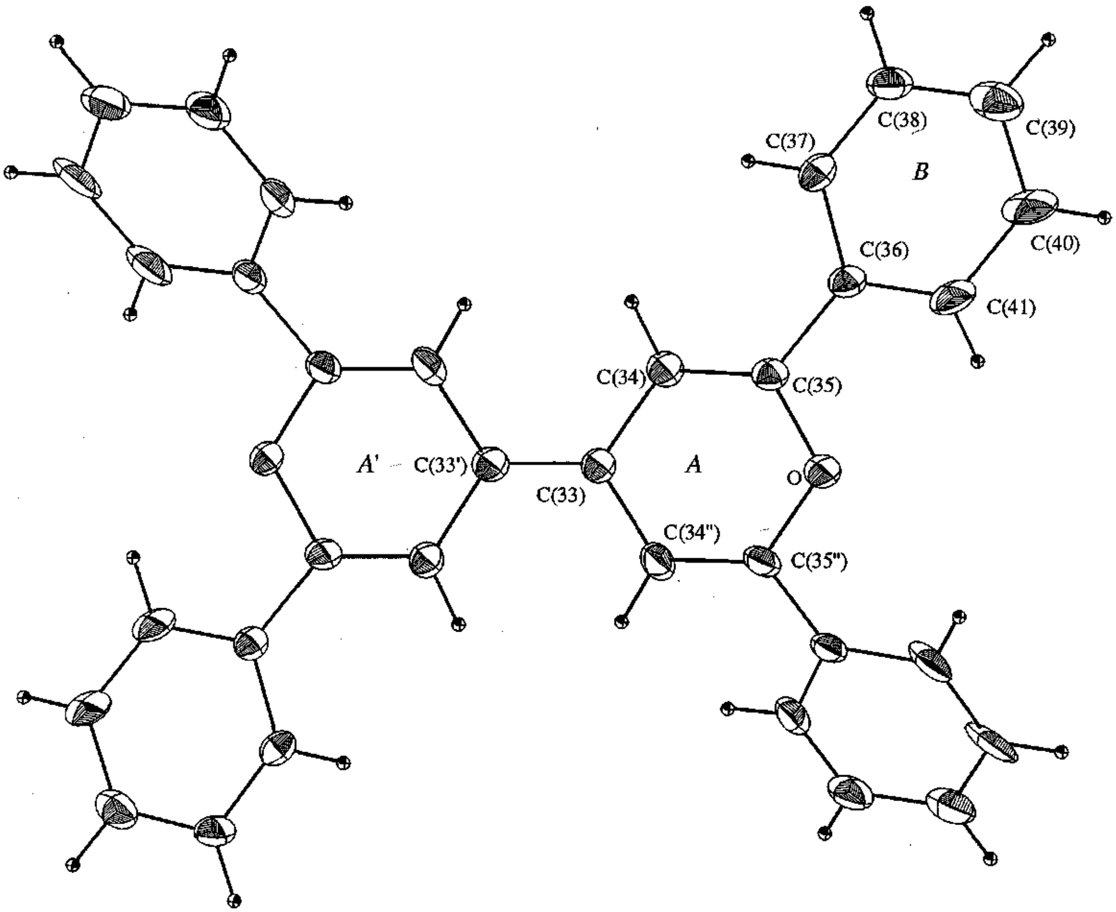

Fig. 3. Molecular structure of TPDP with thermal ellipsoids of thermal and atomic labeling.

the O–C(33) line is 7° (3° in TPDP^0). The planes of the pyranylidene rings *A* and *A*' are parallel and are separated by 0.20 Å (0.02 Å in TPDP^0). The rotation of the phenyl rings *B* with respect to the pyranylidene ring (32.9°) is essentially larger than the corresponding angles in TPDP^0 (10.2° and 7.6°). An increase in the phenyl ring rotation relative to the thiapyranylidene heterocycle was also observed for the sulfur analogue of TPDP, when going from the neutral to charged molecule (8.2° and 12.5° ⟶ 26.5°) [23].

Table 5. Bond lengths *d* (Å) and angles ω (deg) in the TPDP molecule at 170 K

Bond	*d*	Angle	ω
C(33)–C(33')	1.384(9)	C(33')C(33)C(34)	123.3(2)
C(33)–C(34)	1.451(4)	C(34")C(33)C(34)	113.3(4)
C(34)–C(35)	1.340(4)	C(33)C(34)C(35)	121.9(3)
C(35)–C(36)	1.464(5)	C(34)C(35)O	122.6(3)
C(36)–C(37)	1.410(5)	C(35)OC(35")	116.9(3)
C(37)–C(38)	1.365(5)	C(34)C(35)C(36)	125.9(3)
C(38)–C(39)	1.401(5)	OC(35)C(36)	111.5(3)
C(39)–C(40)	1.362(5)	C(35)C(36)C(37)	120.5(3)
C(40)–C(41)	1.399(5)	C(35)C(36)C(41)	121.4(3)
C(36)–C(41)	1.400(4)	C(41)C(36)C(37)	118.1(3)
C(35)–O	1.387(4)	C(36)C(37)C(38)	121.0(3)
		C(37)C(38)C(39)	119.8(3)
		C(38)C(39)C(40)	120.7(3)
		C(39)C(40)C(41)	119.8(3)
		C(40)C(41)C(36)	120.6(3)

These facts are indirect evidence for the charge transfer in the molecular complex TPDP$(C_{60})_2(CS_2)_4$. The conductivity of the TPDP$(C_{60})_2(CS_2)_4$ single crystals [10^{-7} $(\Omega\ \mathrm{cm})^{-1}$] is characteristic of the complexes with a weak charge transfer. Moreover, it is shown [24] that the complex can be doped with iodine after the solvent molecules were removed by heating. This increases the conductivity of crystals by an order of magnitude. Examination of our structural data provides an explanation for removing the solvent and introducing the I_2 molecules into the lattice. Table 3 indicates the increase of all the intermolecular $CS_2 \cdots C_{60}$ distances at room temperature; consequently, heating a sample to higher temperatures results in further weakening of intramolecular interactions and enables the escape of solvent molecules from the crystal. In two symmetry-independent CS_2 molecules, the interatomic C–S distances and the angle are 1.533(5) Å, 1.538(5) Å, and 179.8(4)°; and 1.546(5) Å, 1.541(5) Å, and

179.1(4)°; that is, the mean length of the CS_2 molecule is 3.09 Å. The I–I interatomic distance in the iodine molecule is 2.72 Å [25]. Taking into account different van der Waals radii (1.85 Å for S and 2.10 Å for I), we conclude that the volumes of the CS_2 and I_2 molecules are very close—~83 and 85 $Å^3$, respectively.

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