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Synthesis, structural investigation, and theoretical study of pentafluoroethyl derivatives of [60]fullerene

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Pentafluoroethyl derivatives of [60]fullerene $C_{60}(C_2F_5)_n$ (n = 6, 8, and 10) were synthesized by the reaction of C_{60} with C_2F_5I in glass ampoules at 380—440 °C. Isomers of composition $C_{60}(C_2F_5)_6$ (one isomer), $C_{60}(C_2F_5)_8$ (five isomers), and $C_{60}(C_2F_5)_{10}$ (two isomers) were isolated by chromatographic separation. Their molecular structures were established by X-ray diffraction. The relative stabilities of isomers were compared by density functional theory calculations.

Key words: fullerene C_{60}, pentafluoroethyl derivatives, X-ray diffraction analysis, density functional theory.

Perfluoroalkyl derivatives of fullerenes are thermally and electrochemically stable compounds. Due to the presence of attached electron-withdrawing groups, these compounds hold promise for the use in photovoltaics. A certain advantage of these compounds over fluorofullerenes is associated with high chemical stability, in particular, with resistance against hydrolysis.

Perfluoroalkyl derivatives of fullerenes were synthesized for the first time in 1993.[1] The reactions of fullerene with reagents, which readily release fluoroalkyl radicals on heating or irradiation (fluoroalkyl iodides or fluoroacyl peroxides), produced compounds containing up to 16 fluoroalkyl groups. Mixtures of trifluoromethyl (3—14 CF_3 groups), pentafluoroethyl (9—16 C_2F_5 groups), and perfluorohexyl (6—12 C_6F_{13} groups) derivatives of fullerene C_{60} were characterized by mass spectrometry. Subsequent studies have been focussed primarily on the synthesis and investigations of trifluoromethyl derivatives. Trifluoromethyl radicals were generated by thermal dissociation of either silver trifluoroacetate[2] or trifluoromethyl iodide.[3,4] As a rule, individual isomers are isolated by the HPLC separation of mixtures produced in the synthesis. The derivatives $C_{60}(CF_3)_n$ (n = 8—12 (see Refs 3—6) and 16—18 (see Ref. 7)) and $C_{70}(CF_3)_m$ (m = 6—18)[8—13] were isolated and characterized by X-ray diffraction.

Data on individual pentafluoroethyl derivatives are more scarce. The reaction of C_2F_5I vapor with [60]fullerene at 440 °C afforded a mixture of products, from which one isomer of $C_{60}(C_2F_5)_6$ and one isomer of $C_{60}(C_2F_5)_8$ were isolated and structurally characterized.[14] Oxide $C_{60}(C_2F_5)_4O$ was isolated from a mixture prepared analogously, and its molecular structure was established.[15]

In the present study, we performed the high-temperature ampoule synthesis of the pentafluoroethyl derivatives $C_{60}(C_2F_5)_n$ (n = 6 (**1**), 8 (**2**), and 10 (**3**)) with the use of C_2F_5I. The separation of the resulting mixtures allowed us to isolate eight new isomers containing from six to ten attached pentafluoroethyl groups. These isomers were studied by X-ray diffraction. The relative energy stabilities of the pentafluoroethyl derivatives were investigated by quantum chemical methods, and these results were compared with the data for trifluoromethyl analogs.

Experimental

Synthesis. The reaction of fullerene C_{60} with C_2F_5I was carried out in evacuated glass ampoules. A mixture of fullerene C_{60} (99.98%, TermUSA; 30—50 mg) and C_2F_5I (98%, Apollo; 0.5—0.6 mL) was placed in a two-section ampoule, which was evacuated and sealed. The section containing fullerene was heated to 380—440 °C, whereas the temperature of the compartment containing liquid C_2F_5I was maintained equal to 160—180 °C. After 40—70 h, the reactions were almost completed (the weight of the residue in the section with fullerene was 1—2% of the weight of starting fullerene). The reaction

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products, *viz.*, pentafluoroethyl derivatives and iodine, were sublimed into the colder compartment of the ampoule. Then the ampoule was opened, after which excess C_2F_5I was evaporated and iodine was removed by heating in air at 100 °C for several hours. The yield of the mixture of dark-brown pentafluoroethyl derivatives was ~90% based on used fullerene.

Separation and analysis. Mixtures of derivatives were separated by HPLC. The mixtures were dissolved in a minimum amount of hexane and chromatographed on a semipreparative column (10×250 mm, Cosmosil Buckyprep, Nakalai Tesque Inc.) with the use of hexane as the eluent (4.6 mL $\min^{-1}$); the detection was performed at the wavelength of 290 nm. The compositions of individual fractions were determined by negative-ion MALDI mass spectrometry on a Bruker AutoFlex spectrometer using DCTB (*trans*-2-[3-(4-*tert*-butylphenyl)-2-methyl-2-propenylidene]malononitrile, ≥99%, Fluka) as the matrix. The matrix-to-analyte ratio was 1000—4000. The resulting fractions were additionally chromatographed, and their UV-Vis absorption spectra were recorded.

X-ray diffraction study. For some fractions, large crystals were grown by slow evaporation of hexane solutions. In other cases, recrystallization from another solvent (primarily, from toluene) was required. Depending on the size and quality of the grown crystals, X-ray diffraction data were collected either on a laboratory diffractometer equipped with an area detector (IPDS, Stoe) or using synchrotron radiation and an area detector (CCD or Image-Plate, Bruker). No absorption corrections were applied. Crystallographic data and the X-ray data collection and refinement statistics are given in Table 1. The structures were determined using the SHELXD program package.[16] In the crystal structures of $C_{60}(C_2F_5)_8$ (**2B** and **2C**), there are two independent molecules per asymmetric unit, which substantially complicated both the structure determination and the least-squares refinement.[17] For most of the structures, the least-squares refinement was carried out with anisotropic displacement parameters for nonhydrogen atoms. In the structure of **1B**, some C and F atoms were refined isotropically because of a low ratio between the number of reflections and the number of parameters in the refinement. The atoms of the disordered solvent molecules were also refined isotropically. More complete structural data were deposited at the Cambridge Crystallographic Data Centre (CCDC, refcodes 646403—646410).

Theoretical calculations. To estimate the relative stabilities of individual isomers, their molecular geometry was optimized and the total energies were calculated by the density functional theory (DFT) method using the PRIRODA program[18] and the functional PBE.[19] The starting conformations of the isomers were chosen based on the X-ray diffraction data.

Results and Discussion

Experiments on the synthesis at different temperatures of the hot zone (fullerene) resulted in the formation of mixtures of $C_{60}(C_2F_5)_n$ with a rather narrow composition range. The reaction at 420—440 °C afforded a mixture of products **1** and **2**, whereas the reaction at lower temperature (380—390 °C) gave a mixture of compounds **1—3**. In both cases, compounds **2** with $n = 8$ were obtained as the major products. As in the case of the corresponding trifluoromethylation reactions, the ob-

Table 1. Crystallographic data and the X-ray data collection and refinement statistics for different isomers of compounds **1—3**

Parameter	**1B**	**2B**	**2C**[a]	**2D**	**2E**[b]	**2F**[c]	**3A**	**3B**
Crystal system	Orthorhombic	Monoclinic	Orthorhombic	Triclinic			Monoclinic	
Space group	$Pna2_1$	$P2_1$	$Pna2_1$	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$	$P2_1/n$	$P2_1/n$
a/Å	9.866(1)	10.2033(1)	21.730(3)	13.5059(3)	13.511(3)	13.712(1)	17.9057(4)	17.3406(1)
b/Å	22.702(3)	45.4772(4)	15.603(3)	14.5758(3)	14.415(3)	14.334(1)	16.7890(3)	16.4140(1)
c/Å	21.324(2)	12.0641(2)	35.367(5)	15.2514(5)	16.021(3)	15.936(2)	20.372(1)	21.0493(2)
α/deg	90	90	90	81.574(1)	90.64(2)	87.795(5)	90	90
β/deg	90	106.720(1)	90	86.090(1)	96.59(2)	75.770(5)	99.013(1)	92.7528(4)
γ/deg	90	90	90	64.556(1)	110.32(2)	61.440(6)	90	90
V/Å^3	4776.1(9)	5361.3(1)	11991(3)	2681.9(1)	2902(1)	2655.1(4)	6048.6(3)	5984.3(1)
Z	4	4	8	2	2	2	4	4
Diffractometer	MAR345	MAR345	IPDS-2	MAR345	IPDS-1	MAR345	MAR345	MAR225
T/K	100	100	220	100	140	100	100	100
Radiation (λ/Å)	Synchrotron (0.9100)	Synchrotron (0.9100)	Mo-Kα (0.71073)	Synchrotron (0.9100)	Mo-Kα (0.71073)	Synchrotron (0.9100)	Synchrotron (0.9100)	Synchrotron (0.9100)
N_r/N_p[d]	3896/534	16547/2144	22099/2217	7697/1050	14017/1106	7684/1046	11257/1172	10236/1172
R_1/wR_2	0.139/0.357	0.087/0.217	0.093/0.198	0.059/0.167	0.045/0.117	0.054/0.152	0.096/0.261	0.054/0.155

Note. Crystal dimensions/mm: 0.1×0.01×0.01 (**1B**, **2B**, **2D**, **2F**), 0.5×0.3×0.2 (**2C**), 0.5×0.16×0.12 (**2E**), 0.1×0.07×0.07 (**3A**), and 0.07×0.07×0.02 (**3B**).

[a] A solvate with one toluene molecule.

[b] A solvate of composition $C_{60}(C_2F_5)_8 \cdot 0.5C_7H_8$.

[c] The crystals were grown from a solution in hexane. The crystals grown from toluene are characterized by the similar unit cell parameters and contain another conformer of this isomer (see the text).

[d] The number of the used reflections/the number of parameters in the refinement.

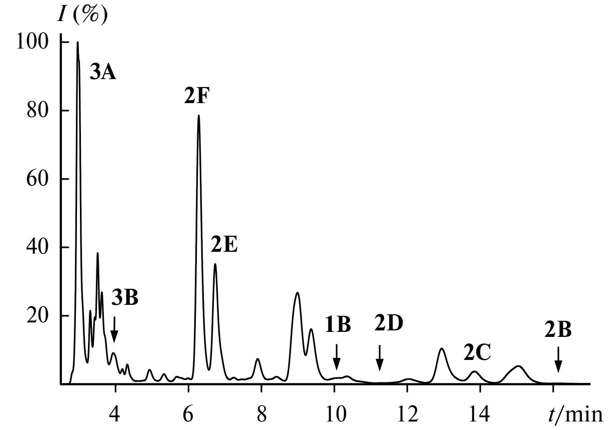

Fig. 1. Chromatogram of the mixture prepared by the reaction of C_{60} with C_2F_5I at 380 °C. The positions of selected chromatographic peaks for the mixture of isomers prepared at 440 °C are indicated by arrows.

served change in the composition of the sublimed product depending on the temperature of the hot zone is associated with higher volatility of higher fluoroalkyl derivatives of fullerenes compared to that of lower derivatives. According to the available data,[14] two isomers with $n = 6$ and 8 were isolated from a mixture of pentafluoroethyl derivatives of [60]fullerene prepared in a reagent flow at 440 °C, but the composition of the mixture was not reported.

The chromatogram of the product prepared in our experiments at 380—390 °C is presented in Fig. 1, which also gives the molecular compositions corresponding to the isolated fractions.* The chromatogram of the product prepared by the high-temperature synthesis (440 °C) contained no fractions with $n = 10$, whereas almost all other peaks with $n = 6$ and 8 were present and had similar retention times. Some isomeric fractions were found only in mixtures prepared by the high-temperature synthesis (indicated by arrows). The elution times of the isomers, whose molecular structures were determined by X-ray diffraction, were 2.96 (**3A**), 3.94 (**3B**), 6.28 (**2F**), 6.73 (**2E**), 10.10 (**1B**), 11.32 (**2D**), 13.84 (**2C**), and 16.11 min (**2B**).

In some cases, crystals suitable for X-ray diffraction were prepared by concentrating hexane fractions obtained after chromatographic separation. In some instances, crystals were grown by recrystallization from another solvent, whereas in some other cases amorphous films and aggregates were obtained upon evaporation. Depending on the size and quality of the crystals, the crystal and molecular structures were determined with different accuracy. This is reflected in both the R factors (R_1 and wR_2; see Table 1) and the standard deviations of the C—C and C—F bond lengths, which vary in the range from 0.003 Å (isomer **2E**) to 0.03—0.04 Å (isomer **1B**). Nevertheless, the addition patterns of the pentafluoroethyl groups at the fullerene cage were unambiguously determined for all structures. However, some attached groups can be slightly disordered, which is manifested, for example, in the observation of two positions of the terminal trifluoromethyl groups belonging to the same C_2F_5 group. In the further discussion, emphasis will be given to the addition patterns of the groups to the fullerene cage.

Examples of the molecular structures of $C_{60}(C_2F_5)_n$ with $n = 6$, 8, and 10 are given in Fig. 2. However, it is convenient to discuss the addition patterns with the use of planar Schlegel diagrams, which are presented in Figs 3—5 for all structurally characterized pentafluoroethyl derivatives of C_{60}. For each isomer, the calculated energy relative to the most stable pentafluoroethyl isomer of the

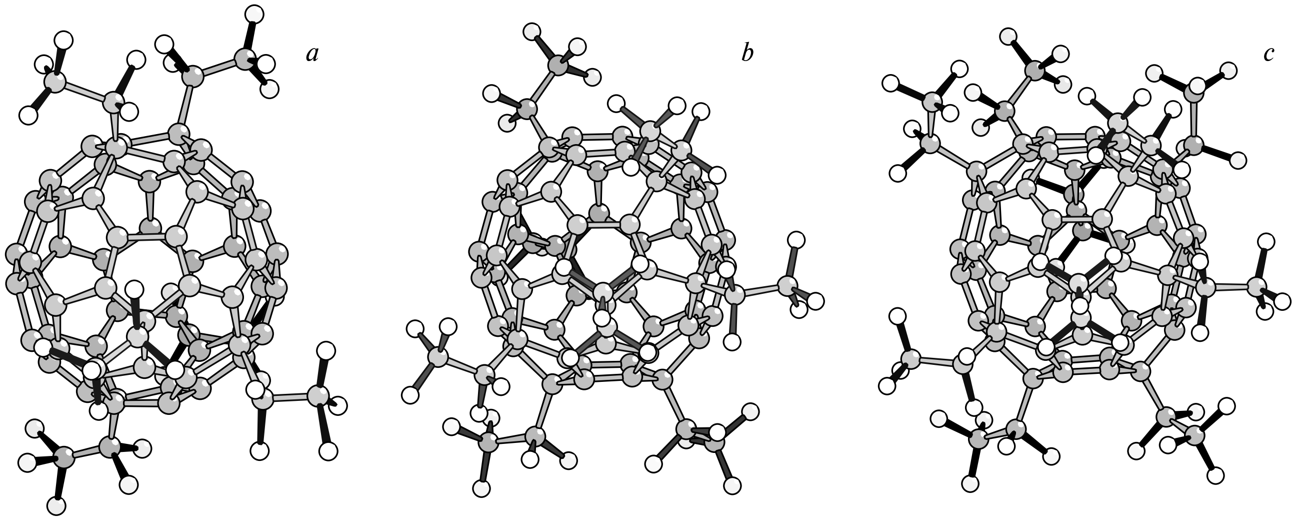

Fig. 2. Molecules **1B** (*a*), **2E** (*b*), and **3B** (*c*). The orientation of the molecules corresponds to that used in the Schlegel diagrams (Figs 3—5).

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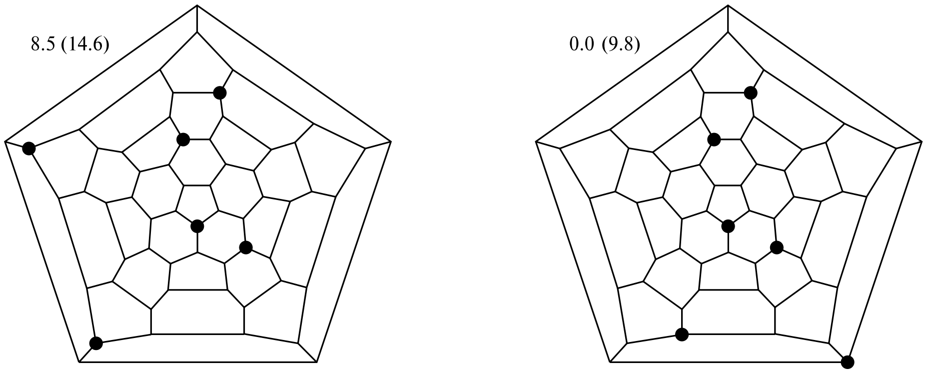
* Isomers of the $C_{60}(C_2F_5)_n$ compounds ($n = 6$ (**1**), 8 (**2**), and 10 (**3**)), whose structures were established by X-ray diffraction, are denoted by the letters **A—F**; the $C_{60}(C_2F_5)_n$ compounds characterized in the study[14] correspond to isomers **A**.



1A

1,7,16,36,46,49-$C_{60}(C_2F_5)_6$ [14]

1B

1,7,16,36,45,57-$C_{60}(C_2F_5)_6$

Fig. 3. Schlegel diagrams for molecules **1A** and **1B**. The calculated relative energies of the presented isomers and the corresponding isomers of $C_{60}(CF_3)_6$ (in parentheses; kJ mol^{-1}) as well as the notations according to the IUPAC nomenclature[20] are given.

same composition, the energy of the corresponding trifluoromethyl derivative (relative to the zero-energy trifluoromethyl derivative), and the notations according to the IUPAC nomenclature are given.[20]

Both structurally characterized isomers of $C_{60}(C_2F_5)_6$ (**1**), *viz.*, isomer **A** described earlier[14] and isomer **B** prepared in the present study, are characterized by the similar modes of attachment of six C_2F_5 groups to the fullerene cage as three isolated (non-adjacent) $C_6(C_2F_5)_2$ hexagons with the C_2F_5 groups in *para* positions in each hexagon (*p*,*p*,*p*) (see Fig. 3). Due to the similarity of the addition patterns, the energies of two isomers differ by only 8.5 kJ mol^{-1}. The corresponding trifluoromethyl derivatives, in which six CF_3 groups are arranged as in isomers **A** and **B**, the energy difference is also small (4.8 kJ mol^{-1}), whereas the p^3mp chain rather than three pairs of isolated hexagons is characteristic of the most stable arrangement. For the pentafluoroethyl derivatives, the transition of the patterns consisting of three isolated pairs to more stable structures is, apparently, caused by an increase in the bulkiness of the attached group (C_2F_5 instead of CF_3). However, to confirm this hypothesis, it is necessary to obtain experimental data for a larger number of $C_{60}(C_2F_5)_6$ isomers.

Six isomers of the $C_{60}(C_2F_5)_8$ compounds (**2**) were structurally characterized. The structures of five of these isomers (**B—F**) were established in the present study. A comparison of the Schlegel diagrams (see Fig. 4) shows the structural similarity of most of the isomers (**A—C**, **E**, and **F**), whose structural motif consists of six C_2F_5 groups in five adjacent hexagons $C_6(C_2F_5)_2$ and two C_2F_5 groups in the isolated hexagon in the *para* positions with respect to each other. In these structures, six C_2F_5 groups are arranged in the same mode (this pattern is additionally outlined for isomer **A** in Fig. 4) as the *para—para—para—meta—para* chain (p^3mp) of adjacent hexagons $C_6(C_2F_5)_2$. It should be noted that this addition pattern is observed in the energetically most stable isomer of $C_{60}(CF_3)_6$, whose structure has recently been determined* by X-ray crystallography. The structural motif of isomer **D** consists of four C_2F_5 groups in three adjacent hexagons $C_6(C_2F_5)_2$ and two pairs of C_2F_5 groups in isolated hexagons. The arrangement of the attached groups in this isomer is most similar to that observed in isomer **A**, because these isomers differ by the positions of only two groups. The crystallographic study of isomer **2B** revealed the statistical disorder of one of the C_2F_5 groups at the end of the p^3mp chain in both independent molecules. This is apparently due to the low energy barrier to the rotation of this group and the absence of steric hindrance, which appears in the case of the closer arrangement of these groups. In addition, the isolated hexagons $C_6(C_2F_5)_2$ of these two molecules differ by the rotation of one of the C_2F_5 groups with respect to the fullerene cage by ~120°, which is indicative of the presence of two conformers in the crystal structure. Two crystal structures were found for isomer **2F**. These structures correspond to conformers, which also differ by the position of one of the C_2F_5 groups belonging to the isolated hexagon $C_6(C_2F_5)_2$ with respect to the fullerene cage.

The relative energy stabilities of the isomers of $C_{60}(C_2F_5)_8$ (**2**) calculated in terms of the density functional theory decrease in the following series: **F** > **E** > **A** > **D** > **C** > **B**. The maximum difference in stability (between isomers **B** and **F**) is 21 kJ mol^{-1}. Nowadays, it is impossible to analyze the relationship between the addition pattern and the energy stability. However, it is clear that this analysis should account also for the arrangement of the pentafluoroethyl fragments with respect to the cage. It should be noted that the most stable

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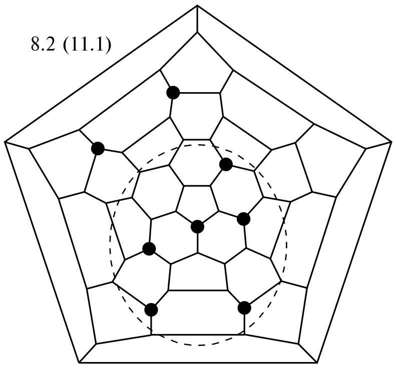
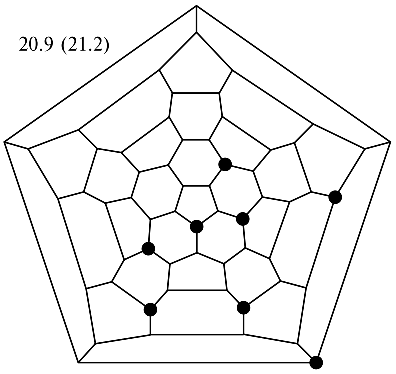
* E. I. Dorozhkin, A. A. Goryunkov, D. V. Ignat´eva, N. B. Tamm, S. M. Avdoshenko, I. N. Ioffe, L. N. Sidorov, and S. I. Troyanov, *Eur. J. Org. Chem.*, 2007, in press.



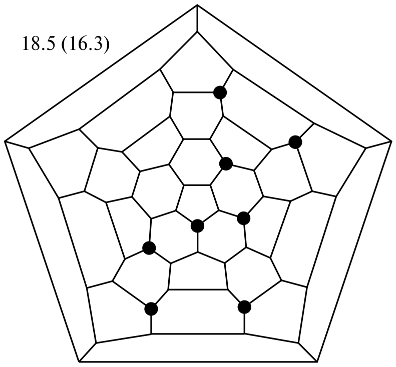
2A

1,6,11,18,24,27,32,35-$C_{60}(C_2F_5)_8$ [14]



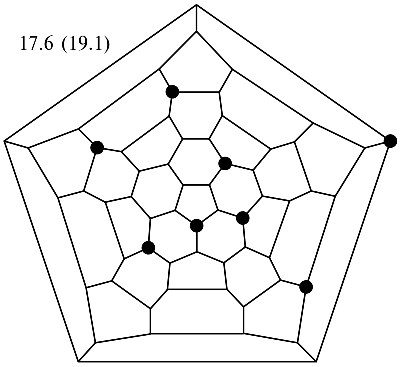
2B

1,6,11,18,24,27,41,55-$C_{60}(C_2F_5)_8$



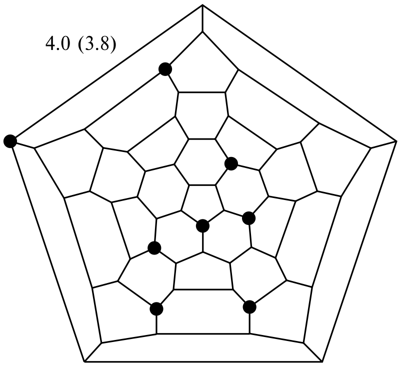
2C

1,6,11,18,24,27,36,39-$C_{60}(C_2F_5)_8$



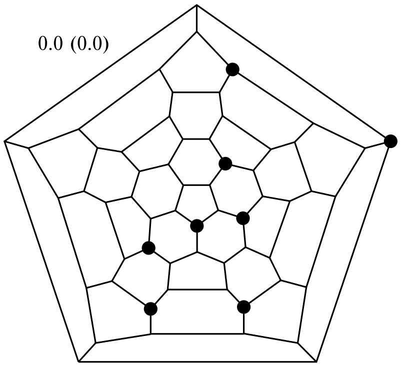
2D

1,6,11,18,32,35,42,56-$C_{60}(C_2F_5)_8$



2E

1,6,11,18,24,27,51,59-$C_{60}(C_2F_5)_8$



2F

1,6,11,18,24,27,53,56-$C_{60}(C_2F_5)_8$

Fig. 4. Schlegel diagrams for isomers **A—F** of the $C_{60}(C_2F_5)_8$ molecule (**2**). For each isomer, the calculated relative energy, the relative energy of the corresponding isomer of $C_{60}(CF_3)_8$ (in parentheses; kJ mol^{-1}), and the notation according to the IUPAC nomenclature[20] are given.

trifluoromethyl derivatives $C_{60}(CF_3)_8$ are characterized by the arrangement of the attached groups involving the p^3mp chain and the isolated hexagon.[5,6] However, the energy sequence for the trifluoromethyl derivatives $C_{60}(CF_3)_8$ having analogous addition patterns (**F** > **E** > **A** > **C** > **D** > **B**) is somewhat different from that

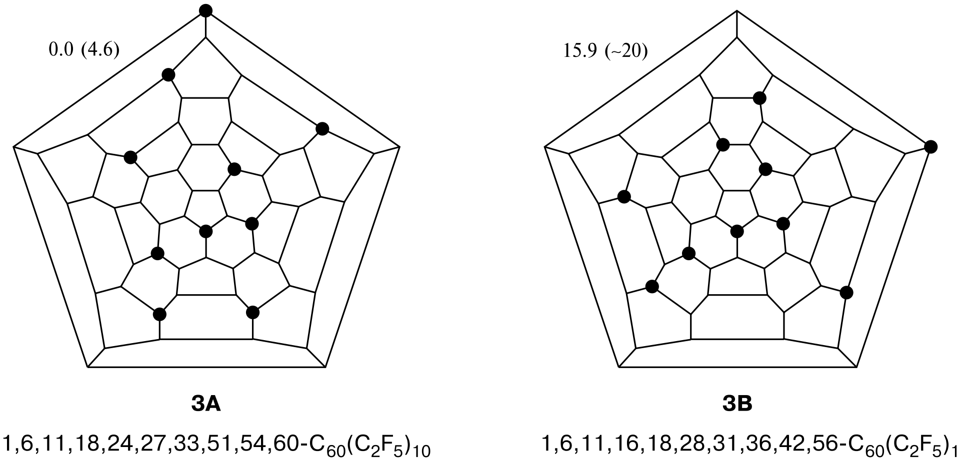


Fig. 5. Schlegel diagrams for molecules **3A** and **3B**. The calculated relative energies of the presented isomers and the corresponding isomers of $C_{60}(CF_3)_{10}$ (in parentheses; kJ mol^{-1}) as well as the notations according to the IUPAC nomenclature[20] are given.

given above. This is also evidence that it is important to take into account the difference in the attachment of pentafluoroethyl groups and trifluoromethyl groups. At least two structurally characterized isomers of $C_{60}(CF_3)_8$, although having the addition pattern 6 + 2 (p^3mp,p), slightly differ in the position of the isolated hexagon $C_6(C_2F_5)_2$.[5,6]

Of the two characterized isomers of the $C_{60}(C_2F_5)_{10}$ compound (**3**), isomer **A** contains 6 + 4 C_2F_5 groups, whereas isomer **B** has 8 + 2 C_2F_5 groups (see Fig. 5). These patterns can be represented as *para—meta* sequences of hexagons $C_6(C_2F_5)_2$: p^3mp,pmp (isomer **A**) and $(pm)^3p,p$ (isomer **B**). The fragment of six C_2F_5 groups in isomer **A** is characterized by the arrangement identical to that observed in the majority of the structures of $C_{60}(C_2F_5)_8$, whereas the arrangement of six C_2F_5 groups in the structure of isomer **B** shows similarity to the pattern observed in structure **2D** (see Fig. 4). Calculations of the energies demonstrated that isomer **A** is 15.9 kJ mol^{-1} more stable than isomer **B**. This difference in stability, apparently, accounts for a large predominance of isomer **A** in the mixture of isomers (see Fig. 1). Among three structurally characterized isomers of $C_{60}(CF_3)_{10}$,[3] the addition patterns with isolated hexagons $C_6(CF_3)_2$ have not been observed. Isomers **3A** and $C_{60}(CF_3)_{10}$-**B*** (with the chain $p^3(mp)^3$),[3b] which differ in the position of only two fluoroalkyl groups, are most similar in the mode of attachment.

On the whole, the diversity of compositions for the available pentafluoroethyl derivatives of C_{60} containing from six to ten attached groups is substantially smaller than that found for trifluoromethyl derivatives, for which compounds containing from two to eighteen groups are known.[1—7] At the same time, there are several isomers of the same composition with n = 10 among the available and characterized $C_{70}(C_2F_5)_n$ compounds.[21]

To summarize, we performed the ampoule synthesis of mixtures of isomers of composition $C_{60}(C_2F_5)_n$ (n = 6, 8, and 10). The fractions were isolated by chromatography, and the molecular structures of eight new isomers were determined by X-ray diffraction. The relative energy stabilities of isomers with different structures were determined by theoretical calculations. Further investigations of pentafluoroethyl derivatives of [60]fullerene will extend the range of known structures and allow the determination of their position among possible isomers in this system.

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* In the study,[3b] this isomer was denoted as $C_{60}(CF_3)_{10}$-II.

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