Structure of 7,9,12,15,18,20,39,24,45,57-$C_{60}(CF3)_{10}(1,2:3,4-O)_2$. The First Regiospecific Diepoxidation of a Fullerene Derivative

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Dedicated to Professor Boris Žemva on the occasion of his receiving the Zois Award for lifetime achievements.

Abstract

An unusual regiospecific diepoxidation of an isomer of $C_{60}(CF_3)_{10}$ was discovered to slowly occur in solution while exposed to air. This is the only known example of a perfluoroalkyl fullerene undergoing epoxidation under ambient conditions. This compound was characterized by a variety of analytical methods including ^{19}F NMR spectroscopy, APCI-MS, UV-vis spectroscopy, and X-ray crystallography. Numerous oxidation methods were explored including ozonation, photolysis, thermolysis, and the chemical oxidation by *m*-chloroperoxobenzoic acid, none of which has yielded the diepoxide. This indicated that the process is kinetically very slow, presumably due to steric crowding around the oxygen addition sites. These findings of spontaneous diepoxidation of the otherwise stable compound have far reaching implications as more fullerenes and fullerene derivatives are being used in organic electronics, and their instability toward oxidation in ambient conditions may alter the performance of the device.

Keywords: Fullerene, epoxide, X-ray structure, fulvene, diepoxidation, ^{19}F NMR

1. Introduction

Fullerenes and fullerene derivatives having epoxide groups directly attached to the cage are important intermediates in the development of functionalized fullerenes with physicochemical properties needed for a variety of practical applications.[1] Our laboratory has reported many dozens of well-characterized perfluoroalkylfullerenes (PFAFs, defined as fullerene$(R_F)_n$ derivatives with two or more perfluoroalkyl (R_F) groups),[2] many of which exhibit high thermal stabilities,[2–4] long radical-anion lifetimes in solution at 25 °C,[5–7] a wide range of reduction potentials,[6,8] and resistance to hydrolysis or attack by strong acids.[9] These properties make PFAFs and PFAF derivatives with additional substituents attractive molecular components for organic electronics.[10–11] Other substituents have been added to pre-formed PFAFs, including halogen and hydrogen atoms[12–13] and substituted carbenes (i.e.,

Bingel adducts).[10,14–15] However, only three monoepoxides of PFAFs, with four or six perfluoroalkyl (R_F) groups, are known. These are 6,12,15,18-$C_{60}(CF_3)_4(1,9-O)$,[16] 6,12,15,18-$C_{60}(C_2F_5)_4(1,9-O)$,[16] and $C_{70}(C_3F_7)_6(O)$.[17] (An IUPAC-numbered Schlegel diagram for C_{60} is shown in Supporting Information.) In this paper we report the structure of the first diepoxide of an isomer of $C_{60}(CF_3)_{10}$, which was formed by a very slow, adventitious, *but regiospecific* reaction of this PFAF with O_2 under ambient conditions.

2. Results and Discussion

2. 1. The Discovery of $C_{60}(CF_3)_{10}(O)_2$ (I)

The PFAF 1,3,7,10,14,17,23,28,31,40-$C_{60}(CF_3)_{10}$ (**II**) was the first PFAF to have its structure determined by X-ray diffraction.[18] Its addition pattern is shown in Figure 1, which also shows the remaining C=C bonds with di-

stances shorter than 140 pm (note that three times the standard error for these distances is 0.9 pm). Note that this addition pattern results in an *isolated* pentafulvene fragment composed of C atoms 2, 11, 12, 13, 29, and 30, the first of its kind in fullerene chemistry (a pentafulvene fragment that is *not* isolated from the rest of the fullerene π system was observed in the structure of 1,7,11,24-C_{60}(9-fluorenyl)$_4$ published in 1997[19]). An understanding of how the crystallographically-determined addition pattern is
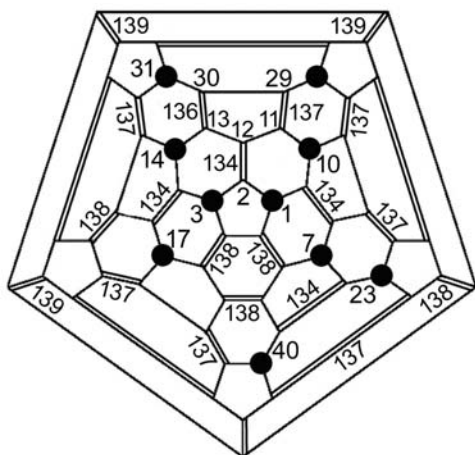

Fig. 1. Schlegel diagram of the isomer of $C_{60}(CF_3)_{10}$ used in this work, 1,3,7,10,14,17,23,-28,31,40-$C_{60}(CF_3)_{10}$ (**II**). The black circles indicate the fullerene C atoms to which the ten CF_3 groups are attached. The one and two-digit numbers are IUPAC locants. The three digit numbers are, in pm, the fullerene C=C bonds shorter than 140 pm, and are taken from the X-ray structure of this compound reported in reference [18] (note that three times the standard error for these bond distances is 0.9 pm).

re 2. Its ten quartets, quartets-of- quartets (apparent septets), or unresolved multiplets are due to through-space Fermi-contact $^{6,7}J_{FF}$ coupling of proximate CF_3 groups that share the same fullerene hexagon or pentagon.[18,20] The previously published spectrum of **II** was recorded in C_6D_6 solution,[18] and the two multiplets with -δ values < 62 were accidentally isochronous in that solvent (these multiplets arise from the CF_3 groups that share the same pentagon). Selective decoupling experiments performed in this work revealed the specific addition-pattern NMR assignments for these two multiplets; this is discussed in more detail in the Supporting Information (Figure S-1).

Significantly, single crystals of **II** suitable for X-ray diffraction were found to be air-stable under ambient laboratory light indefinitely, as are single crystals of all other PFAFs we have examined. However, prolonged exposure (many months) of oxygenated, tightly stoppered chloroform or toluene solutions of **II** to ambient light resulted in the formation of a new soluble species with a new set of multiplets, the spectrum of which is shown in Figure 2 (there was little or no loss of solvent to evaporation, and no precipitate formed, during this time). Assuming that the addition pattern of the CF_3 groups had not changed, the multiplets with the greatest chemical-shift changes belonged to the six CF_3 groups that surrounded the isolated fulvene fragment. Furthermore, the mass spectrum of a purified sample of the new compound indicated that its composition was $C_{60}(CF_3)_{10}(O)_2$ (**I**) (Figure S-2). The separation of **I** and **II** was accomplished using high-performance liquid chromatography, as shown on Figure S-4.

The structure of the new compound (**I**) was determined by single-crystal X-ray diffraction, and is shown in
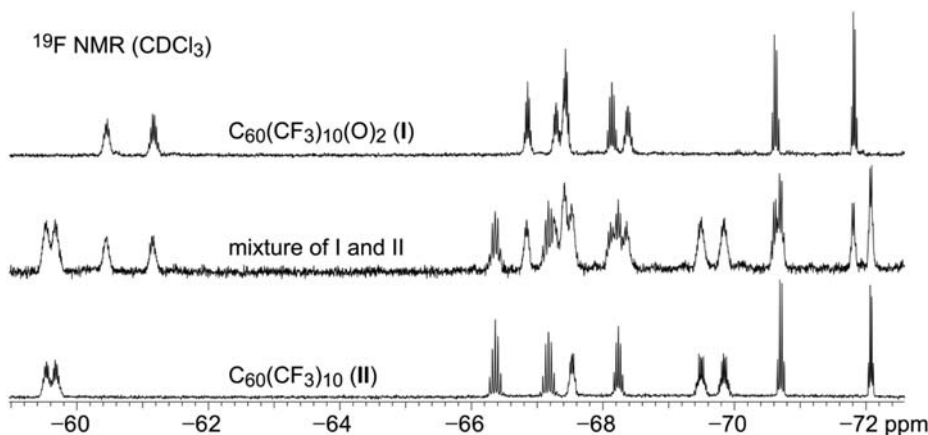

Fig. 2. The 376.5 MHz ^{19}F NMR spectra of (bottom) **II**, a sample of **II** exposed to air and ambient light for several months, which produced a mixture of **I** and **II** (middle), and a purified sample of **I** (top). The internal chemical shift standard was C_6F_6 (δ -164.9).

correlated with its 1D and 2D ^{19}F NMR spectra eventually led to the NMR spectroscopic determination of addition patterns of many other trifluoromethylfullerenes (TMFs) when X-ray structures were not available. The 1D ^{19}F NMR spectrum of **II** dissolved in $CDCl_3$ is shown in Figure 2 dissolved in $CDCl_3$ is shown in Figu-

Figure 3 (a drawing of the molecule showing all atoms as 50% probability ellipsoids is shown on Figure S-11). It is a PFAF diepoxide, and is only the third fullerene diepoxide to be structurally characterized (the first two are co-crystallized isomers of $C_{60}(O)_2(IrCl(CO)(PPh_3)_2)$[21]). In

the discussion that follows, the cage C atom locants for **I** are, for convenience, the same as the locants for **II**, although the IUPAC locants for **I** and **II** are different (using the same locants for **I** and **II**, the numbered formula for **I** is 1,3,7,10,14,17,23,28,31,40-$C_{60}(CF_3)_{10}(11,29:13,30-O)_2$, whereas the correct numbering for **I**, which will not be used below, is 7,9,12,15,18,20,39,24,45,57-$C_{60}(CF_3)_{10}$ (1,2:3,4-$O)_2$).
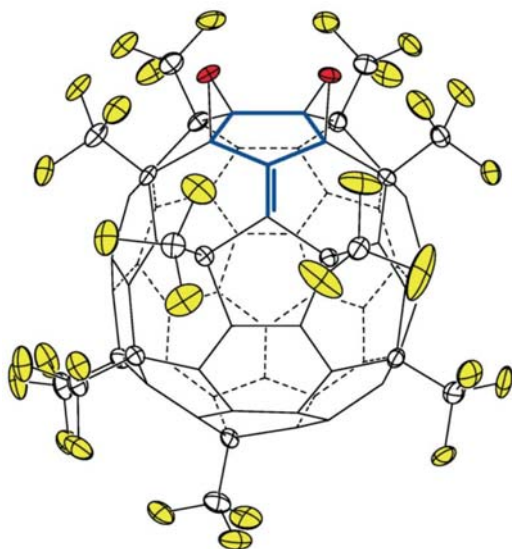

Fig. 3. The structure of $C_{60}(CF_3)_{10}(O)_2$ (**I**), with the remainder of the fulvene-like fragment in the structure of $C_{60}(CF_3)_{10}$ (**II**, see Fig. 1) highlighted in blue (50% probability ellipsoids for the O atoms, the CF_3 groups, and the cage C atoms to which the CF_3 groups are attached; the two molecules of $CDCl_3$ have been removed for clarity). The bond distance for the remaining C=C double bond in the former fulvene-like fragment is 133.5(8) pm.
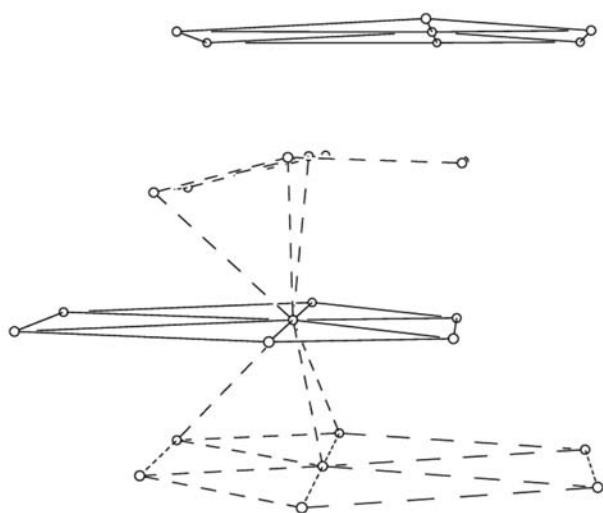

Fig. 4. The packing of the C_{60} centroids in the structure of $C_{60}(CF_3)_{10}(O)_2$ (**I**). Note that there are nearly-planar and significantly puckered pseudo-hexagonal arrays that are stacked in a complicated fashion in the third dimension.

The two epoxide O atoms in **I** have been added to the conjugated C11–C29 and C13–C30 double bonds of the pentafulvene fragment in **II**. These were C(sp^2)–C(sp^2) bonds in **II** (136.0(3) and 136.5(3) pm long, respectively[18]) and are C(sp^3)–C(sp^3) bonds in **I** (150.0(8) and 149.2(8) pm long, respectively). The four C–O distances in **I** range from 142.2(7) to 145.3(7) pm and are unexceptional. As expected, the C11–C12, C12–C13, and C29–C30 bonds are longer in **I** (148.9(8), 149.4(8), and 153.2(8) pm, respectively) than in **II** (1.465(3), 147.3(3), and 147.6(3) pm, respectively). The other cage C–C bonds in **I** and **II** are virtually the same. For example, the C2–C12 double bond is 133.5(8) pm in **I** and 134.4(3) in **II**.

If one ignores the $CDCl_3$ solvates, the molecules of **I** are arranged in both nearly-planar and significantly puckered pseudo-hexagonal arrays, as shown in Figure 4. Within the hexagonal arrays, the C_{60} centroid–centroid distances range from 980 to 1,810 pm. The corresponding centroid–centroid distances in the structure of **II**, which did not contain any molecules of solvent, are similar and range from 1,017 to 1,417 pm.[18] As can be seen in Figure 4, the stacking of the pseudo-hexagonal arrays in the third dimension is irregular and will not be discussed further.

2. 2. The Unique Nature of 1,3,7,10,14,17,23,28,31,40-$C_{60}(CF_3)_{10}$ (II)

PFAF **II** is unique in two, presumably interconnected, ways. It is the only PFAF with a pentafulvene fragment that is isolated from the rest of the fullerene π system, and it is the only PFAF that undergoes air oxidation in solution at 25 °C, albeit very slowly. No other PFAF we have studied forms even a monoepoxide, let alone a diepoxide, under such mild conditions, even after many months.

The two isostructural PFAF monoepoxides we reported in 2006, shown as a Schlegel diagram in Figure 5, were formed during the high-temperature perfluoroalkylation of C_{60} with either CF_3I or C_2F_5I, presumably due to the presence of adventitious O_2 or H_2O in the 430 °C reactor hot zone. Although the mechanism of formation of these two $C_{60}(R_F)_4O$ compounds is unknown, a reasonable intermediate would be the unknown PFAFs 6,9,15,18-$C_{60}(R_F)_4$ (R_F = CF_3, C_2F_5). This para-para-para addition pattern results in a pentafulvene fragment that is conjugated to the greater fullerene π system via the pentafulvene exocyclic C=C bond. Several groups have proposed that such an addition pattern should be especially prone to epoxidation at, or other types of additions to, this C=C bond.[19,22–23] The slow oxidation of **II** produces the diepoxide **I**, not the hypothetical monoepoxide 1,7,10,14,17,23,28,31,40-$C_{60}(CF_3)_{10}(2,12-O)$. Therefore, the reactivity of the isolated pentafulvene fragment in **II** is entirely different than the reactivity of the »open« pentafulvene fragments in other fullerene derivatives.
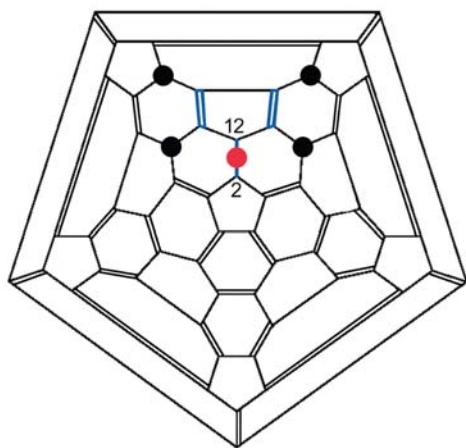
Fig. 5 Schlegel diagram for the PFAF monoepoxides $C_{60}(CF_3)_4O$ and $C_{60}(C_2F_5)_4O$, with the remainder of the fulvene-like fragment in the structure of $C_{60}(CF_3)_{10}$ (**II**, see Fig. 1) highlighted in blue. The black circles indicate the cage C atoms to which the R_F groups are attached. The red circle represents the epoxide O atom, which is bonded to C2 and C12. Note that the conjugated blue C=C double bonds are isolated from the remainder of the fullerene π system.

2. 3. Attempts to Intentionally Epoxidize 1,3,7,10,14,17,23,28,31,40-$C_{60}(CF_3)_{10}$ (II)

Fullerene epoxides were among the first synthesized fullerene derivatives, and a variety of methods have been developed for the addition of up to 12 epoxide O atoms to C_{60}.[21,24–25] Once the structure of **I** was determined, we attempted to prepare it from purified samples of **II** in a rational manner using a number of these methods.

Photolysis in the presence of O_2. Compound **II** was irradiated in the presence of dioxygen for various amounts of time (up to 24 hours) in solution. Several experiments were performed in non-polar solvents including benzene, toluene, dichloromethane (DCM) and heptane in order to determine if the solvent played a role in oxidation. Additionally, a more polar solvent (THF) was investigated as were several mixtures of polar and non-polar solvents (benzene: acetonitrile and heptane: acetonitrile). Furthermore, **II** was deposited as a film from DCM and irradiated for 12 hours. It was determined by ^{19}F NMR spectroscopy that **II** does not undergo oxidation under any of the above conditions. This result that **II** does not undergo rapid photooxidation reinforces earlier claims that PFAFs, in general, are robust and light- and air-stable molecules.

Thermal Reaction with O_2. Compound **II** was sublimed in the presence of dioxygen at 555 °C, the resulting ^{19}F NMR spectrum indicated that there was not oxidation or decomposition. Several additional experiments were conducted where oxygenated solutions of **II** in various solvents were heated to 80 °C until the solvent had fully evaporated. Upon subsequent analysis of the resulting ^{19}F NMR spectra, we concluded that no oxidation or decomposition occurred. Solutions of **II** were also heated to ref-

lux for 24 hours and again, no oxidation or decomposition was observed by ^{19}F NMR spectroscopy. It has been shown in the literature that oxidation of a derivatized fullerene occurred more readily in a more polar solvent.[16] Two solutions of pure **II** were prepared, one in 50:50 benzene: acetonitrile and the other in CS_2. These two solutions were heated for 24 hours and no decomposition or oxidation was observed in either one.

Ozonation. A mixture of O_2 and O_3 gas was bubbled through a room temperature toluene solution of **II** exposed to ambient light for 5 min. The subsequent analysis using APCI-MS, HPLC and ^{19}F NMR spectroscopy indicated that a complex mixture of oxides of **II** formed (Figures S-5 to S-7). This result is not unexpected as the literature indicates that complex mixtures typically result when C_{60} is treated with O_3.[25] The presence of **I** could not be reliably identified in the ^{19}F NMR spectrum of ozonated **II** indicating that it was not among the abundant products.

Reaction with *m*-chloroperoxobenzoic acid (*m*-CPBA). When a pure sample of **II** was dissolved in toluene and reacted with excess *m*CPBA for 12 hours, a complex mixture of oxides and toluene adducts of **II** were observed by HPLC, APCI-MS and ^{19}F NMR (Figures S-8 to S-10). Shorter reaction times (up to one hour) resulted in no reaction, indicating that even with a strong oxidizer, the oxidation reaction proceeds slowly.

3. Conclusions

In this work, an unusual regiospecific diepoxidation of **II** was discovered to slowly occur upon storage of organic solutions in open air and ambient light. In the course of few months, only half of the parent PFAF was converted into a single isomer of **I**, which was isolated and characterized by X-ray crystallography. This is the first time a PFAF diepoxide has been observed and structurally characterized. Furthermore, this is the only known PFAF to undergo oxidation in solution under ambient conditions. Numerous oxidation methods were employed to make **II** oxidize at a faster rate; including photolysis, ozonation, thermolysis, and the use of *m*CPBA as a chemical oxidant. All these attempts to prepare **I** selectively were unsuccessful indicating that diepoxidation of **II** is a kinetically very slow process. We propose that the reason for such low reaction rate is steric hindrance from the CF_3 groups that block the two fulvene endocyclic double bond addition sites. It is even more surprising that addition to the short exocyclic pentafulvene double bond or to one of the several less hindered double bonds on the cage of compound **II** does not occur at all. This observation adds credence to the hypothesis that the shorter double bonds in the fully isolated fulvene moiety are highly activated and may serve as a possible synthetic handle for further regiospecific functionalization of PFAFs that possess such a moiety on the fullerene core.

The observed slow epoxidation of an otherwise stable fullerene derivative by adventitious oxygen also has significance from the practical point of view. As more and more fullerene derivatives find practical applications as components in organic electronic devices, e.g., in active layers in OPVs, or in OFETs, their instability towards air (oxygen) may adversely affect device performance.

For example, a recent report indicated that 1,4-bis(dimethylphenylsilylmethyl)-[60]fullerene that was used in the OPV devices undergoes photo-oxygenation in solution as well as in the thin films at room temperature over the period of several days.[9] Furthermore, it was shown that as the percentage of the oxidized fullerene increases in the active layer of the OPV cell over time, the performance of the device deteriorates accordingly.[9] These data and results of our current study should warn researchers working on introducing new compounds into OPV devices on the necessity to rigorously investigate stability of these new materials towards oxidation. The fact that compound **II**, as well as other PFAFs, does not undergo any detectable oxidation in the solid state (in contrast to the organic derivative reported in ref. [9]) indicate that PFAFs belong to a potentially valuable class of air-stable organic acceptor materials for real-life applications.

4. Experimental

4. 1. Chemicals and Instrumentation

All solvents were A.C.S. grade or better and were used as received without further purification. The 3-chloroperoxybenzoic acid (mCPBA) was purchased from Aldrich and was 77% max with the remainder being 3-chlorobenzoic acid and water. Compound **II** was prepared as previously described in the literature.[18] $O_2{:}O_3$ gas mixture was prepared by passing technical grade oxygen through a corona arc discharge.

Thermal experiments were performed on either a hot plate or in a Lindberg model 55035 tube furnace equipped with an external thermocouple. Photolysis experiments were carried out in a photochemical cabinet equipped with a ventilation fan and a Hanovia #679A36 450 W Hg lamp placed in a water-cooled quartz immersion well. The operating temperature was approximately 30 °C throughout the entirety of the experiment.

HPLC analysis and purifications were carried out using a Shimadzu HPLC instrument (CBM-20A control module, SPD-20A UV-detector set to 300 nm, LC-6AD pump, manual injector valve) equipped with a semi-preparative 10 mm I.D. × 250 mm or a preparative 25 mm I.D. × 250 mm Cosmosil Buckyprep column (Nacalai Tesque, Inc.).

Fluorine-19 NMR spectra were recorded using a Varian 400 spectrometer operating at 376.5 MHz (C_6F_6 internal standard, δ -164.9).

Negative-ion APCI mass spectra were recorded using a Finnigan 2000 LCQ-DUO spectrometer. The samples were injected as ca. 50:50 v:v toluene:ACN solutions; the mobile phase was ACN.

4. 2. X-ray diffraction Data

Crystals of compound I (grown by slow evaporation from saturated chloroform-*d* solution) were recorded on a Bruker Kappa APEX II CCD diffractometer employing Mo Kα radiation (graphite monochromator). Selected details related to the crystallographic experiments are listed in the Table 1. Unit cell parameters were obtained from least-squares fits to the angular coordinates to all reflections, and intensities were integrated from a series of frames converting more than a hemisphere of reciprocal space. The structures were solved by using direct methods and refined (on *F2,* using all data) by a full-matrix, weighted least-squares process. All atoms were refined by using anisotropic atomic displacement parameters. Standard Bruker control and integration software (APEX II) was employed, and Bruker SHELXTL software was used for structure solution and refinement.

4. 3. Reactions of II With Oxidizing Agents

50 mL of toluene was added to a flask containing approximately 1 mg of **II** and excess mCPBA. This solution was heated to reflux for 12 hours and kept in the dark. After 12 hours the toluene was removed via vacuum. The m-CPBA was removed by washing with petroleum ether 5 times. Ozonolysis reactions were performed by bubbling a mixture of $O_3{:}O_2$ gas as previously described through a toluene solution of **II** while exposed to ambient light and air.

Table 1. Crystallographic data collection and structure refinement parameters for $C_{60}(CF_3)_{10}(O)_2 2CDCl_3$

molecular formula	$C_{70}F_{30}O_2 2(CDCl_3)$
formula weight	1683.45
crystal system	tetragonal
space group	$I4_1/a$
Z	16
color of crystals	yellow
unit cell dimensions (Å)	$a = 42.4640(19)$
	$b = 42.4640(19)$
	$c = 12.0696(6)$
data collection temperature, K	100(2)
final *R* indices, [$I > 2\sigma(I)$]	
	$R_1 = 0.0781$
$wR_2 = 0.2555$	
goodness-of-fit on F^2	1.270

6. Appendix A. Supplementary data

A Supporting Information document is available on the Acta Chim. Slovenica website. Crystallographic data (excluding structure factors) for the structure of **I** has been deposited at the Cambridge Crystallographic Center, CCDC No. 904348. Copies of the data can be obtained free of charge on http:www.ccdc.cam.ac.

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Povzetek

Nenavadna regiospecifična diepoksidacija izomera $C_{60}(CF_3)_{10}$ poteka počasi v raztopini, ki je izpostavljena zraku. To je do sedaj edini poznan primer perfluoroakil fulerena, pri katerem poteka epoksidacija pri sobni temperaturi. Spojino smo karakterizirali z različnimi analitskimi metodami: ^{19}NMR, APCI–MS, UV–VIS spektroskopijo in rentgensko strukturno analizo. Preizkušene so bile številne metode oksidacije, kot so: ozoniranje, fotoliza, termoliza in kemijska oksidacija z m-kloroperoksibenzojsko kislino. Pri nobeni izmed metod ni potekala diepoksidacija. Na podlagi teh ugotovitev lahko zaključimo, da je proces zelo počasen, najverjetneje zaradi steričnih ovir okoli kisika. Rezultati te raziskave, v kateri smo opisali spontano diepoksidacijo spojine, ki je sicer stabilna, pa bodo lahko imeli daljnosežne posledice. Vedno več fulerenov in njihovih derivatov se namreč uporablja v organski elektroniki in njihova nestabilnost napram oksidaciji pri običajnih zunanjih pogojih lahko spremeni delovanje naprav.