



Hexa-Substituted Hybrid C₆₀ Derivatives with Norbornadiene Fragments: Synthesis and Structure [†]

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Abstract: New hybrid fullerene C₆₀ derivatives with five norbornadiene fragments, at a distance from the fullerene core of several methylene groups, were synthesized for the first time. Subsequent photoirradiation of these derivatives leads to only partial (up to 20%) opening of double bonds in norbornadiene fragments to form quadricyclane substituents. It is assumed that the resulting fullerene C₆₀ derivatives can serve as a basis to create high-energy materials.

Keywords: fullerene C₆₀; norbornadiene; photoirradiation; quadricyclane

1. Introduction

Fullerenes and their derivatives attract great attention due to their unique properties that allow them to be used as effective antioxidants [1], solar energy converters [2], semiconductor materials [3], additives for motor oils [4], and modern medicinal products [5]. Such a wide applied significance of fullerene C₆₀ derivatives is due to various methods of their functionalization, among which the most popular are currently the Prato [6] and Bingel–Hirsch reactions [7]. These approaches make it possible to selectively prepare not only mono-, bis-, and tris- but also hexa-adducts of fullerene C₆₀ [8–11] with specified symmetry types, in which addends are located almost throughout the entire fullerene sphere. Besides these studies, there are works devoted to the synthesis of various highly symmetric fullerene C₆₀ polyadducts, when a chlorofullerene C₆₀Cl₆ was used as a starting compound. Indeed, this method makes it possible to obtain various alkoxyfullerenes [12], aminofullerenes [13], sulfides [14], and arylated [15] C₆₀ derivatives, which have particular interest and application in medicine [16,17].

We reported recently [18–21] the synthesis of energy-rich methanofullerenes through the reaction of fullerene C₆₀ with mono- and bis-quadricyclane esters of malonic acid under Prato reaction conditions [22]. It was shown that some distance between a quadricyclane fragment and a fullerene core is necessary to maintain the metastable structure of a hybrid fullerene–quadricyclane molecule. At the same time, it seemed interesting and relevant to study the photochemical isomerization of norbornadiene fragments into quadricyclane ones in the C₆₀ cycloadducts synthesized.

Considering these facts, we performed a covalent binding of fullerene C₆₀ with norbornadienes and quadricyclanes in this work, where chlorofullerene C₆₀Cl₆ was used as a starting compound. It was assumed that the selective synthesis of C₆₀ adducts containing five addends, according to the method described in the literature [12], will increase the solubility of new hybrid molecules due to a higher number of quadricyclanes attached covalently to a C₆₀ carbon core. The photochemical isomerization of norbornadiene-containing penta-derivatives of fullerene C₆₀ into quadricyclane derivatives was also studied.



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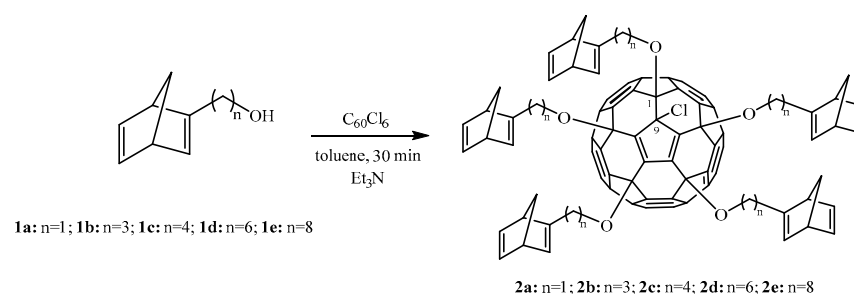


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2. Results and Discussion

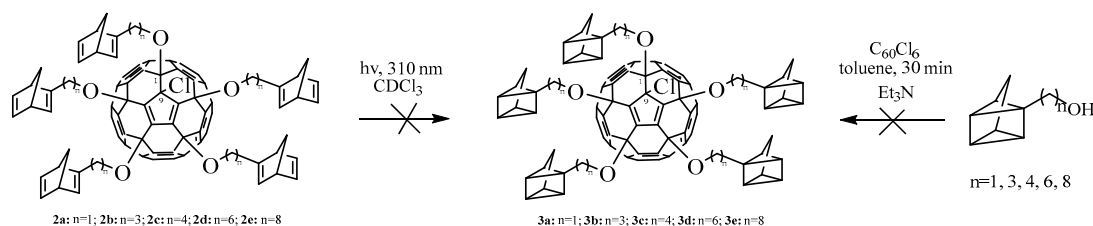
The interaction between hexachlorofullerene and norbornadiene alcohols **1a–1e** [23,24] at room temperature for 30 min led to the formation of hexa-substituted adducts **2a–2e**, in which the fullerene core was bound to a polycycle fragment through an ether bond. Fullerene polyadducts **2a–2e** were isolated from the reaction mass using preparative HPLC with a chromatographic purity of ~99.9%.

The structure of compounds **2a–2e** was elucidated with one-dimensional (^1H and ^{13}C) and two-dimensional (^1H – ^1H COSY, ^1H – ^{13}C HSQC, ^1H – ^{13}C HMBC) NMR techniques and MALDI–TOF mass spectrometry. High-frequency signals (δ 158.72, 144.39, 142.30, and 133.30 ppm), characteristic of sp^2 hybridized carbon atoms of a norbornadiene fragment, and low-frequency ones (δ 73.53, 55.66 and 50.20 ppm), typical of sp^3 carbon atoms of this fragment, for example, are observed in the ^{13}C NMR spectrum of compound **2e**, in which the norbornadiene fragment is removed from a fullerene core to the greatest extent compared to those in the derivatives **2a–2d**. The ^{13}C NMR signals of seven methylene groups appear in the low-frequency region at δ 31.73, 30.57, 29.77, 29.63, 27.44, 26.67, and 26.59 ppm, and the signal of a methylene group bound to an oxygen atom is at δ 68.34 ppm. The sp^3 hybridized carbon atom of a fullerene sphere bound to a chlorine one resonates at δ 71.47 ppm in the ^{13}C NMR spectrum, and fullerene carbon atoms in the sp^3 hybridization bound to oxygen atoms give signals at δ 79.39 and 81.66 ppm, which is in agreement with previous published data [12,25]. Fullerene carbon atoms in the sp^2 hybridization give 47 signals at δ 138–154 ppm, of which seven signals have a double relative intensity, and the rest have a single one. This indicates that the molecule of this derivative is assigned to a C_1 symmetry point group, probably due to the asymmetric arrangement of a norbornadiene fragment at the C-1 fullerene carbon atom relative to the conventional symmetry plane passing through the C-1 and C-9 carbon atoms of a fullerene sphere (see Scheme 1 for numbering of carbon atoms) [26]. A molecular ion peak in the MALDI–TOF mass spectrum recorded in the negative ion mode at m/z 1851.8505 indicates that five oxooctylnorbornadiene fragments and one chlorine atom are attached to a fullerene sphere, which confirms the proposed structure of derivative **2e**. Similarly, the molecules of the compounds **2a–2d** are assigned to the C_1 symmetry group, regardless of the length of an alkyl fragment that connects an oxygen atom and a norbornadiene fragment.



Scheme 1. Preparation of hexa-adducts of fullerene C_{60} .

We reported previously [18] that it is impossible to perform the photoisomerization of norbornadiene fragments into quadricyclane ones in hybrid molecules, but it is known [11] that such compounds are capable of converting into quadricyclane derivatives under certain conditions. We converted the hybrid molecules **2a–2e** with norbornadiene fragments into those with quadricyclane ones **3a–3e** via photochemical isomerization according to the method described earlier [11] (Scheme 2). As a result, it was found that the norbornadiene fragments are only partially isomerized into quadricyclane ones with a yield of no more than 20%. It was impossible to isolate and to determine the composition of new hybrid molecules because of the formation of a complex mixture of stereoisomers consisting of norbornadiene and quadricyclane fragments bound to one fullerene molecule. We assumed that quadricyclane fullerene C_{60} derivatives can be synthesized through the reaction between chlorofullerene C_{60}Cl_6 and the corresponding quadricyclanes in this regard.



Scheme 2. Synthesis of quadricyclane fullerene C₆₀ derivatives.

The reaction between quadricyclane alcohols and hexachlorofullerene also led to the formation of a difficult-to-identify reaction mass of the compounds **3a–3e** under previously developed conditions [12], which is probably because of the partial opening of strained C–C bonds in quadricyclane fragments.

3. Materials and Methods

All reactions were performed in an argon atmosphere and in an anhydrous solvent. The solvents and reagents were dried or purified according to procedures described in the literature. Commercially available fullerene C₆₀ (purity of 99.5%, Sigma-Aldrich, 9402 Alberene Drive, Houston, TX 77074, USA) was used.

The reaction products were analyzed on a Shimadzu SPD-20A HPLC chromatograph (1900 SE 4th Avenue, Canby, OR 97013, USA) equipped with a UV detector at 313 or 340 nm. The mixtures were separated on a Cosmosil Buckyprep Waters preparative column (250 × 10 mm) at a temperature of ~20 °C. Toluene was used as an eluent, and the flow rate was 3.0 mL min^{−1}. The ¹H, ¹³C, and two-dimensional NMR spectra were acquired on a Bruker Avance III HD 500 NMR spectrometer with frequencies of 500 and 125 MHz, respectively. A mixture of CDCl₃ and CS₂ (1:5) was used as a solvent. The ¹H and ¹³C NMR chemical shifts (δ) are given in ppm relative to internal standard SiMe₄. Mass spectra were recorded on a MALDI-TOF/TOF mass spectrometer (Bruker Daltonik GmbH, Germany), operating in linear (TOF) and reflective (TOF/TOF) modes of positive and negative ions. S₈ and DCTB (trans-2-[3-(4-tert-butylphenyl)-2-methyl-2-propenylidene]malononitrile) were used as a matrix. The solutions of the samples in toluene were used to apply them to a metal target for mass spectrometric measurements. The photoisomerization of norbornadiene derivatives into quadricyclane derivatives was performed on a HAMAMATSU LC 8 irradiator at 310 nm.

Compound 2a

Brown powder. ¹H NMR, δ: 1.60 (m, 1H), 2.06 (m, 1H), 2.19 (m, 1H), 2.21 (m, 1H), 3.59 (m, 2H), 6.12 (m, 1H), 6.77 (m, 1H), 6.78 (m, 1H). ¹³C NMR, δ: 25.84, 27.31, 29.41, 29.49, 29.56, 29.83, 31.62, 32.92, 50.11, 50.28, 51.57, 51.99, 53.59, 63.14, 66.63, 66.81, 67.04, 67.26, 67.69, 68.05, 69.10, 69.65, 70.06, 70.29, 73.50, 73.73, 76.83, 77.08, 77.34, 133.21, 137.21, 137.33, 137.45, 137.62, 137.81, 138.18, 142.36, 142.51, 142.61, 143.31, 143.40, 143.55, 143.83, 144.39, 147.28, 147.70, 148.08, 148.28, 148.46, 148.93, 149.14, 149.48, 149.57, 154.68, 154.85, 154.99, 155.14, 158.92. MALDI-TOF, [M][−] calcd. for C₁₀₀H₄₅ClO₅ 1361.3024, found 1361.3020.

Compound 2b

Brown powder. ¹H NMR, δ: 1.62 (m, 2H), 1.71 (m, 1H), 1.99 (m, 2H), 2.07 (m, 1H), 3.02 (m, 2H), 3.52 (m, 2H), 6.13 (m, 1H), 6.76 (m, 2H). ¹³C NMR, δ: 22.96, 26.49, 28.15, 29.47, 29.93, 30.43, 30.62, 31.56, 32.10, 42.00, 50.34, 50.58, 50.87, 53.71, 60.47, 65.95, 67.73, 67.99, 68.16, 68.33, 68.58, 68.80, 73.61, 133.81, 134.00, 134.17, 134.44, 136.12, 136.32, 136.39, 136.48, 142.20, 142.26, 142.32, 143.84, 143.92, 143.99, 146.94, 147.00, 147.10, 147.16, 147.32, 147.50, 147.76, 147.86, 147.93, 148.07, 148.14, 148.42, 148.55, 148.92, 149.06, 149.21, 157.74. MALDI-TOF, [M][−] calcd. for C₁₁₀H₆₅ClO₅ 1501.4657, found 1501.4662.

Compound 2c

Brown powder. ¹H NMR, δ: 1.29 (m, 2H), 1.56 (m, 2H), 1.78 (m, 1H), 1.99 (m, 2H), 2.03 (m, 1H), 3.29 (m, 1H), 3.32 (m, 1H), 3.51 (m, 2H), 6.18 (m, 1H), 6.76 (m, 2H). ¹³C NMR, δ:

24.12, 29.96, 30.10, 31.48, 50.29, 53.65, 67.83, 68.06, 68.45, 73.59, 133.78, 133.90, 134.00, 142.26, 143.86, 146.96, 147.08, 147.23, 147.36, 147.55, 147.77, 147.89, 148.18, 148.31, 148.41, 148.53, 149.02, 149.16, 149.46, 149.56, 158.07, 158.35. MALDI-TOF, $[M]^-$ calcd. for $C_{115}H_{75}ClO_5$ 1571.5337, found 1571.5331.

Compound 2d

Brown powder. 1H NMR, δ : 1.30 (m, 2H), 1.33 (m, 2H), 1.39 (m, 2H), 1.47 (m, 2H), 1.81 (m, 1H), 1.98 (m, 2H), 2.06 (m, 1H), 3.28 (m, 2H), 3.48 (m, 2H), 6.12 (m, 1H), 6.74 (m, 2H). ^{13}C NMR, δ : 26.48, 27.46, 28.67, 29.56, 30.03, 30.54, 31.77, 33.43, 50.31, 53.72, 67.77, 67.93, 68.12, 68.32, 73.60, 133.52, 142.25, 143.85, 144.40, 145.13, 145.30, 145.50, 145.63, 147.13, 147.30, 147.53, 147.71, 147.81, 148.08, 148.21, 148.41, 148.71, 148.88, 149.06, 149.21, 149.38, 149.51, 158.47. MALDI-TOF, $[M]^-$ calcd. for $C_{125}H_{95}ClO_5$ 1711.6943, found 1711.6939.

Compound 2e

Brown powder. 1H NMR, δ : 1.34 (m, 10H), 1.43 (m, 2H), 1.82 (m, 1H), 1.97 (m, 2H), 2.00 (m, 1H), 2.21 (m, 2H), 3.49 (m, 2H), 6.12 (m, 1H), 6.75 (m, 2H). ^{13}C NMR, δ : 26.60, 26.69, 27.45, 29.64, 29.72, 29.78, 30.58, 31.74, 50.20, 53.66, 68.34, 68.71, 73.54, 133.30, 142.30, 143.83, 144.05, 144.26, 144.39, 144.69, 144.81, 145.14, 145.29, 145.47, 145.63, 145.93, 146.93, 147.14, 147.35, 147.56, 147.68, 147.96, 148.11, 148.33, 149.14, 149.32, 149.44, 158.71. MALDI-TOF, $[M]^-$ calcd. for $C_{135}H_{115}ClO_5$ 1851.8515, found 1851.8505.

4. Conclusions

In summary, we synthesized hybrid molecules containing five norbornadiene or quadricyclane fragments bound covalently to a fullerene core through one, three, four, six, and eight oxymethylene units for the first time. The stereoselectivity of this reaction was studied thoroughly with spectral methods. It was shown for the first time that quadricyclane alcohols, reacting with $C_{60}Cl_6$, are converted under selected conditions into the corresponding norbornadiene derivatives.

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Conflicts of Interest: The authors declare no conflict of interest.

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