Molecular electrochemistry of hydrofullerenes $C_{70}H_{36-46}$

A. S. Lobach★ and V. V. Strelets

Institute of Problems of Chemical Physics, Russian Academy of Sciences,
142432 Chernogolovka, Moscow Region, Russian Federation.
Fax: +7 (096) 515 3588. E-mail: lobach@cat.icp.ac.ru, strelets@icp.ac.ru

Electrochemistry of a mixture of hydrofullerenes $C_{70}H_{36-46}$ composed of $C_{70}H_{36}$, $C_{70}H_{38}$, $C_{70}H_{44}$, and $C_{70}H_{46}$ (50, 20, 14, and 15%, respectively) was studied by cyclic voltammetry in THF and CH_2Cl_2 in the −43—−13 °C temperature range. Two cathodic peaks, namely, one-electron reversible ($E^\circ = -3.16$ V ($Fc^{0/+}$), Fc is ferrocene) and irreversible ($E_p^c = -3.37$ V ($Fc^{0/+}$)) were observed for this mixture in THF. The irreversible broad oxidation peak ($E_p^a = 1.22$ V ($Fc^{0/+}$)) was observed in CH_2Cl_2. The reversible reduction peak ($E^\circ = -3.16$ V) and irreversible oxidation peak ($E_p^a = 1.22$ V) were attributed to the most stable hydrofullerene $C_{70}H_{36}$. The irreversible reduction ($E_p^c = -3.37$ V) and oxidation ($E_p^a = 1.22$ V) peaks were attributed to hydrofullerenes $C_{70}H_{44-46}$ with a higher degree of hydrogenation. The values of an "electrochemical gap," which is an analog of the energy gap (HOMO—LUMO), are 4.38 and 4.59 V for $C_{70}H_{36}$ and $C_{70}H_{44-46}$, respectively, and indicate that these hydrofullerenes are sufficiently "hard" molecules with low reactivity in redox reactions.

Key words: hydrofullerene $C_{70}H_{36}$, $C_{70}H_{44-46}$, redox properties, cyclic voltammetry, electron affinity, electrochemical gap.

We have previously[1] studied the electrochemical behavior and determined several parameters (electron affinities, EA, "electrochemical gap," *G*, electrochemical electronegativity, χ, and others) for hydrofullerene $C_{60}H_{36}$. The obtained data indicate that this hydrofullerene exists predominantly as an isomer with *T* symmetry in which 12 double bonds form four isolated benzene rings in the vertices of an imaginary inscribed tetrahedron. Along with $C_{60}H_2$ [2,3] and $C_{70}H_2$,[3,4] this is the third electrochemically studied hydrofullerene. It seemed reasonable to extend the scope of electrochemically characterized hydrofullerenes because this provides a valuable information about the reactivity of electrogenerated radical ions, the energy of their frontier orbitals (HOMO, LUMO), *etc.* We chose hydrofullerene $C_{70}H_{36}$ as the object of the study. It was synthesized by several methods and identified by mass spectrometry but was not studied by physicochemical methods (see, *e.g.*, Ref. 5). Previously,[6,7] several isomeric structures for $C_{70}H_{36}$ were considered. According to theoretical calculations, this is a molecule, which is either completely "aromatic" or contains partially isolated double bonds. The study of the electrochemical parameters of $C_{70}H_{36}$ could give additional data on isomeric structures of fullerenes.

In this work, we present the results of cyclic voltammetric (CVA) study of the molecular electrochemistry of hydrofullerenes $C_{70}H_{36-46}$ in THF and CH_2Cl_2 in the −43—13 °C temperature interval. The obtained data were used for the calculation of the electrochemical and other parameters of hydrofullerenes $C_{70}H_{36-46}$.

Experimental

Hydrofullerenes $C_{70}H_{36-46}$ were synthesized by the hydrogenation of C_{70} with 9,10-dihydroanthracene in its melt at 350 °C. The reaction products were purified according to a previously described procedure.[8] According to mass spectral analysis data, a mixture of $C_{70}H_{36-46}$ contains the following hydrofullerenes (content in the mixture): $C_{70}H_{36}$ (50%), $C_{70}H_{38}$ (20%), $C_{70}H_{40}$ (0.5%), $C_{70}H_{42}$ (0.5%), $C_{70}H_{44}$ (14%), and $C_{70}H_{46}$ (16%). The composition of the mixture depends on the hydrogenation duration, and an increase in the reaction time enriches the mixture in hydrofullerene $C_{70}H_{36}$ and depletes it in hydrofullerenes with a higher content of hydrogen, which indicates the stability of $C_{70}H_{36}$ compared to other hydrofullerenes. Thus obtained $C_{70}H_{36-46}$ samples contaminated with 0.5—1 wt.% anthracene formed from 9,10-dihydroanthracene (according to spectrophotometric and fluorescence analyses).

The composition of a mixture of hydrofullerenes was determined by FD mass spectrometry on a Varian MAT-731 mass spectrometer with double focusing at a resolution of ~2000.

Hydrofullerenes $C_{70}H_{36-46}$ are poorly dissolved in solvents normally used for electrochemical measurements, although their solubility is higher than that of $C_{60}H_{36}$,[1] and their concentration in both CH_2Cl_2 and THF solutions did not exceed $2 \cdot 10^{-4}$ mol L^{-1}. Therefore, due to a higher solubility of anthracene, the concentrations of hydrofullerene and anthracene in tested solutions were comparable.

CVA measurements were carried out under dry argon in CH_2Cl_2 and THF preliminarily purified and distilled directly into an electrochemical cell filled with argon according to a previously described procedure.[9] Tetrahydrofuran (Aldrich) was purified by the "ketyl" method, and CH_2Cl_2 (Aldrich) was refluxed for 2—3 h above phosphorus pentaoxide followed by distillation. A 0.05 *M* solution of Bu_4NPF_6 was used as the supporting electrolyte. Tetrabutylammonium hexafluoro-

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phosphate (Aldrich) was dehydrated by melting *in vacuo*. Low-temperature CVA measurements were carried out with thermostatting of an electrochemical cell with an ethanol—liquid nitrogen mixture in a Dewar vessel.

All potentials are presented relatively to the $Fc^{0/+}$ redox pair. The reference electrode Ag/AgCl/4 *M* aqueous solution of LiCl was separated from the studied solution by a bridge filled with a solution of the supporting electrolyte. The potential of the $Fc^{0/+}$ redox transition $E^\circ = 0.44$ and 0.43 V, SCE in THF and CH_2Cl_2, respectively. The CVA curves were detected in the absence of Fc, which was added at the end of each experiment.

A disk glassy carbon electrode (diameter 3 mm, Tokai, Japan) sealed into glass and polished with a diamond paste (particle size ≤1 μm). Voltammetric measurements were carried out on a PAR 175 signal generator with ohmic loss compensation by the positive feedback method. Voltammetric curves were recorded on a RE0074 XY-recorder.

Results and Discussion

Unlike fullerene C_{60} hydrogenation, the hydrogenation of fullerene C_{70} with 9,10-dihydroanthracene produces a mixture of hydrofullerenes with different hydrogen concentrations.[8] The following hydrofullerenes are the main reaction products (content in the mixture) $C_{70}H_{36}$ (50%), $C_{70}H_{38}$ (20%), $C_{70}H_{44}$ (14%), and $C_{70}H_{46}$ (16%). In this work, we ignored traces of $C_{70}H_{40}$ (0.5%) and $C_{70}H_{42}$ (0.5%) due to their low content.

As in the case of $C_{60}H_{36}$,[1] difficulties in recording reliable CVA curves because of the poor solubility of $C_{70}H_{36-46}$ ($<2 \cdot 10^{-4}$ mol L^{-1}) were prevented by the use of a glassy carbon electrode with a sufficiently large surface area (0.07 cm^2) as the working electrode. The cathodic CVA curves of $C_{70}H_{36-46}$ in THF at 13 °C in the region of studied sweeps (v) of 0.02—0.5 V s^{-1} exhibit four diffusionally controlled ($I_p \cdot v^{-1/2}$ is const, where I_p is the peak current at the maximum)[10] peaks (*A*, *B*, *C*, and *D*, respectively,* Fig. 1, curve *1*)[1] with different heights and degrees of reversibilities. The potentials of these peaks $E_p{}^c$ and/or formal redox potentials ($E^\circ = (E_p{}^c + E_p{}^a)/2$, where $E_p{}^c$ and $E_p{}^a$ are the potentials of the cathodic and anodic peaks) for the corresponding redox pairs are presented in Table 1.

Similarly to the previously studied[1] hydrofullerene $C_{60}H_{36}$, the $C_{70}H_{36-46}$ samples, obtained by C_{70} hydrogenation with 9,10-dihydroanthracene,[8] even after the purification of target hydrofullerene by sublimation *in vacuo* contained traces (0.5—1.0 wt.%) of anthracene,[11,12] which is much better soluble than $C_{70}H_{36-46}$. During the preparation of a saturated solution of $C_{70}H_{36-46}$, anthracene contained in the sample (**1**) is completely transferred to the solution. Therefore, the reversible couple of peaks *A*/*A*′ (see Fig. 1, curve *2*) is referred to the $\mathbf{1}^{0/\bullet-}$ redox pair, which is confirmed by CVA of the authentic sample of **1** (see Table 1 and Fig. 2 in Ref. 1). Peak *C* also concerns the reduction of **1** ($\mathbf{1}^{\bullet-} + e \rightarrow \mathbf{1}^{2-}$) (see Table 1 and Fig. 2 in Ref. 1). It follows from this that the reduction of hydrofullerene $C_{70}H_{36-46}$ is characterized by the reversible couple of peaks *D*/*D*′ and irreversible cathodic peak *B*. At temperatures below −34 °C, the *C* and *D* peaks are transformed into one peak (see Fig. 1, curve *3*). Unlike $C_{60}H_{36-46}$,[1] the *D* peak for $C_{70}H_{36-46}$ is reversible already at ~20 °C, which indicates a higher stability of the $C_{70}H_{36}{}^{\bullet-}$ radical anion compared to $C_{60}H_{36}{}^{\bullet-}$.

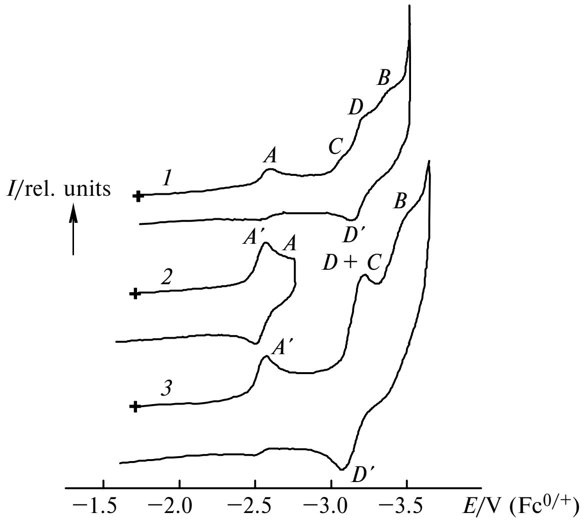

Fig. 1. Cyclic voltammograms for reduction of a saturated solution of hydrofullerenes $C_{70}H_{36-46}$ on a glassy carbon electrode ($\nu = 0.2$ V s^{-1}) in THF/0.05 *M* Bu_4NPF_6 at 13 (*1*) and −34 °C (*2* and *3*).

Table 1. Potentials of peaks on CVA curves of hydrofullerenes $C_{70}H_{36-46}$ and anthracene on a glassy carbon electrode ($\nu = 0.2$ V s^{-1}) relatively to the redox pair $Fc^{0/+}$

Compound	Solvent	*T*/°C	Peak	E° (E_p)*/V
$C_{70}H_{36-46}$	THF	+13	*A*/*A*′	−2.55
			C	(−3.08)
			D/*D*′	−3.18
			B	(−3.37)
$C_{70}H_{36-46}$	THF	−34	*A*/*A*′	−2.55
			D/*D*′	−3.16
			B	(−3.49)
$C_{70}H_{36-46}$	CH_2Cl_2	−31	*H*	(0.46)
			E	(0.88)
			F	(1.22)
			G	(1.57)
Anthracene	THF	+14	*A*/*A*′	−2.57
			C	(−3.13)
Anthracene	CH_2Cl_2	−47	*E*/*E*′	0.85
			G	(1.57)

* The potentials of irreversible peaks are indicated in parentheses.

The quasi-reversible cathodic peak *D* is one-electron, which follows from $\Delta E_p = E_p{}^a - E_p{}^c = 65$ mV for this peak at 13 °C. Note for comparison that for the authentic ferrocene redox pair $Fc^{0/+}$ $\Delta E_p = 60$ mV under identical conditions.

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* Henceforth the abbreviations of peaks are the same as those used by us previously[1] for $C_{60}H_{36}$.

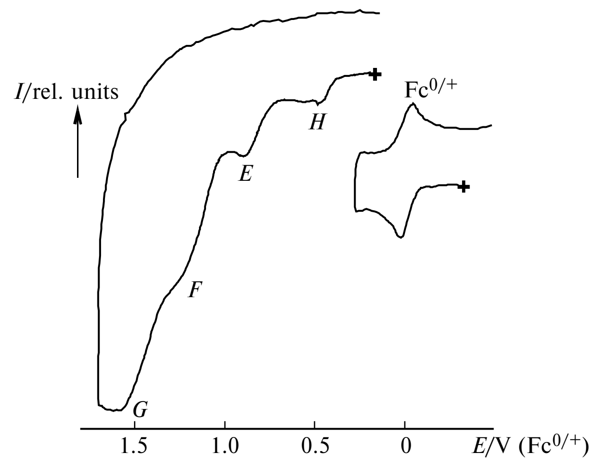

Fig. 2. Cyclic voltammograms for oxidation of a saturated solution of hydrofullerenes $C_{70}H_{36-46}$ on a glassy carbon electrode (v = 0.2 V s^{-1}) in CH_2Cl_2/0.05 *M* Bu_4NPF_6 at −31 °C.

The oxidation of $C_{70}H_{36-46}$ was studied in CH_2Cl_2 at −31 °C on a glassy carbon electrode. The CVA curve of hydrofullerene contains four diffusionally controlled ($I_p \cdot v^{-1/2}$ is const)[10] irreversible anodic peaks (Fig. 2, *H*, *E*, *F*, and *G*), whose potentials ($E_p{}^a$) are presented in Table 1. Figure 2 shows that the anodic peaks *E* and *G* are attributed to anthracene (*cf.*, Fig. 2 in this work and Fig. 3 in Ref. 1). The *E* peak should be attributed to the reversible $\mathbf{1}^{0/\cdot+}$ redox pair (peaks *E*/*E'*, Fig. 3, curve *3*),[1] and the *G* peak is referred to the irreversible redox transition $\mathbf{1}^{\cdot+/2+}$ (see Table 1 and Fig. 3 in Ref. 1). Probably, the electrochemically generated radical cation $\mathbf{1}^{\cdot+}$ reacts with the initial $C_{70}H_{36-46}$, which can be a reason for irreversibility of the *E* peak in the CVA of hydrofullerene. Therefore, the oxidation of hydrofullerenes $C_{70}H_{36-46}$ is characterized by the broad unresolved irreversible peak *F*. We cannot unambiguously say whether the irreversible peak *H* concerns hydrofullerene oxidation or not, and the nature of this peak remains unclear. Experimental data on fullerene[3,13] and hydrofullerene[1,2] oxidation show that the oxidation potentials differ insignificantly and range within 1.13—1.39 V (see Table 2). Note that $C_{70}H_{36-46}$ are oxidized at higher positive potentials than C_{70} (see Table 2 and Refs. 3 and 13). The oxidation potentials of $C_{70}H_2$ are unknown.

Table 2. Electrochemical parameters of fullerenes, hydrofullerenes, and benzenes (potentials are given relatively to the redox pair Fc$^{0/+}$)

Compound	Solvent	E^{ox}	E^{red} *	χ	*G*	Ref.
		V				
C_{60}	CH_2Cl_2	1.39	−1.04	0.17	2.43	3
C_{60}	THF	—	−0.86			3
C_{60}	Toluene/DMF	—	−0.93	—	—	4
C_{60}	TCE*	1.26	−1.06	0.10	2.32	12
C_{70}	TCE*	1.20	−1.02	0.09	2.22	12
C_{70}	THF	—	−0.85			3
C_{70}	Toluene/DMF	—	−0.91	—	—	4
$C_{60}H_2$	CH_2Cl_2	—	−1.11			2
$C_{60}H_2$	Benzonitrile	1.13	−1.04	0.05	2.17	2
$C_{60}H_2$	Toluene/DMF	—	−1.02	—	—	4
$C_{70}H_2$	Toluene/DMF	—	−1.04	—	—	4
$C_{60}H_{36}$	CH_2Cl_2	1.22	—			1
$C_{60}H_{36}$	THF		−3.18	−0.98	4.40	1
$C_{70}H_{36-38}$	CH_2Cl_2	1.22	—			**
$C_{70}H_{36-38}$	THF		−3.16	−0.97	4.38	**
$C_{70}H_{44-46}$	CH_2Cl_2	1.22	—			**
$C_{70}H_{44-46}$	THF		−3.37	−1.08	4.59	**
C_6H_6	THF		−3.81			13
C_6H_6	MeCN	1.86		−0.98	5.67	14

* In 1,1,2,2-tetrachloroethane. Reversible oxidation and reduction peaks.

** This work.

The difference between the reduction potentials of hydrofullerenes $C_{70}H_{36-46}$ is −0.21 V, and the addition of one H atom to the molecule shifts the potential by ~0.02 V to the cathodic region. If we assume a linear change in the potential values with changing the hydrogen content in hydrofullerene, the differences in the reduction potentials of $C_{70}H_{36}$ and $C_{70}H_{38}$, $C_{70}H_{44}$ and $C_{70}H_{46}$ lie within errors of the method and, hence, they are not pronounced in CVA at this stage of studies. The reversible reduction peak *D* (E° = −3.16 V, Fc$^{0/+}$) observed in the CVA of hydrofullerenes $C_{70}H_{36-46}$ should be assigned, in our opinion, to $C_{70}H_{36}$ because this hydrofullerene, similarly to $C_{60}H_{36}$, is most stable, which follows from our findings and theoretical calculation, according to which the $C_{70}H_{36}$ and $C_{60}H_{36}$ are most stable.[6] Probably, they have similar redox properties by analogy to the initial fullerenes,[13] hydrides $C_{70}H_2$ [4] and $C_{60}H_2$,[4] as well as on the basis of theoretical studies indicating the presence in these molecules of fragments with the aromatic character.[6,7] We assigned the second irreversible reduction peak at $E_p{}^c$ = −3.37 V (Fc$^{0/+}$) to hydrofullerenes with a higher degree of hydrogenation $C_{70}H_{44}$ and $C_{70}H_{46}$, which should be characterized by a higher degree of delocalization due to benzenoid fragments in the molecule. This conclusion is based on the fact that compounds containing benzenoid fragments are reduced at higher cathodic potentials (for benzene, *e.g.*, E° = −3.81 V, Fc$^{0/+}$ in THF)[14] than compounds containing double bonds.[15] The broad oxidation peak (see Fig. 2, peak *F*) is, most probably, a superposition of the oxidation peaks of all hydrofullerenes, which indicates the proximity of the oxidation potentials of these compounds and fullerenes (see, *e.g.*, Table 2).

The electron affinity (*EA*) of hydrofullerene $C_{70}H_{36}$ can be estimated from its reduction potential using the equation

$$E^\circ = EA - \Delta\Delta G + \text{const}, \qquad (1)$$

where E° and *EA* are the experimentally measured reduction potential of the molecule in the liquid phase and its electron affinity in the gas phase, respectively. The $\Delta\Delta G$ value is the difference in solvation energies of neutral molecules and their anions, which depends on the nature of the solvent and supporting electrolyte. The constant in Eq. (1) depends on the reference electrode used.

It can be assumed that, under similar experimental conditions (solvent, supporting electrolyte, reference electrode), the $\Delta\Delta G$ values for hydrofullerene $C_{70}H_{36}$ and benzene are approximately equal because satisfactory correlation obtained from Eq. (1) for fullerenes and their derivatives and for aromatic compounds are known.[16] The calculation of the electron affinity of hydrofullerene $C_{70}H_{36}$ by Eq. (1) gives $EA \sim -0.3$ eV. The corresponding value for a benzene molecule ($E^\circ = -3.81$ B ($Fc^{0/+}$)) is −1 eV. The negative value of the electron affinity of hydrofullerenes was confirmed by the study of hydrofullerene $C_{70}H_{36-46}$ by mass spectroscopy of negative ions. The spectra contained the negative ions $C_{70}H_x^-$ (x = 0—24) with the approximately equal intensities for x = 0—18, which decreased rapidly at x = 24, and these peaks were absent at $x > 24$.[17] This experimental fact indicates that hydrofullerenes $C_{70}H_{36-46}$ have a negative electron affinity, whereas $C_{70}H_x$ ($x < 24$) are characterized by positive EA values.

The reactivity of hydrofullerene $C_{70}H_{36}$ in redox reactions can be predicted using the value of the "intrinsic electrochemical gap" G and electrochemical electronegativity (χ), which are calculated from the experimental reduction and oxidation potentials of the molecules: $G = E^{ox} - E^{red}$ and $\chi = (E^{ox} + E^{red})/2$ (see Refs. 18 and 19). Table 2 contains G and χ for fullerenes C_{60} and C_{70}, hydrofullerenes C_xH_2 and C_xH_{36} (where x = 60 and 70, respectively), and benzene.

The oxidation potentials for hydrofullerenes are irreversible (see this work and Ref. 3). Since the redox potentials were measured in different solvents, they cannot be used, strictly speaking, for the calculation of G and χ. However, these calculations give tentative results for the comparable evaluation of these values.[18,19] The oxidation potential of $C_{70}H_2$ is unknown, therefore, we cannot calculate G for this hydrofullerene but can assume that it is close to that for $C_{60}H_2$. It follows from Table 2 that the G, value, which is an analog of the "energy gap" (HOMO—LUMO), for $C_{70}H_{36}$ and $C_{70}H_{44-46}$ is approximately twofold higher than that for C_{70} and hydrofullerene $C_{70}H_2$ and more close to G for benzene. Based on this, we can assume that $C_{70}H_{36-46}$ in redox reactions should act as sufficiently "hard" (according to Pearson) molecules with a low reactivity. The electrochemical electronegativity of $C_{70}H_{36}$ and $C_{70}H_{44-46}$ is substantially lower than that of C_{70} and close to χ of benzene. This additionally favors the conclusion that hydrofullerene $C_{70}H_{36-46}$ molecules contain benzenoid fragments similarly to $C_{60}H_{36}$. .

Note in conclusion that the measured reduction potentials of hydrofullerenes $C_{70}H_{36-46}$ indicate, most likely, the aromatic character of these molecules, *i.e.*, the molecules with aromatic fragments on the surface rather than the molecules containing isolated double bonds.

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