Electron-transfer induced change of direction of interannular haptotropic isomerization of fluorenyltricarbonylchromium anions

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It has been shown by cyclic voltammetry in a THF medium in the temperature range from −70 °C to +20 °C that one-electron electrochemical reduction of $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ (**1**) to the corresponding 19-electron anion radical ($\mathbf{1}^-$) is accompanied by splitting off of a H atom to form the 18-electron carbon-centered anion $(\eta^6\text{-}C_{13}H_9)Cr(CO)_3^-$ ($^-\mathbf{2}$), which at room temperature undergoes intramolecular haptotropic isomerization to the metal-centered $(\eta^5\text{-}C_{13}H_9)Cr(CO)_3^-$ ($^-\mathbf{3}$) anion. The reversible one-electron reduction of $\mathbf{3}^-$ to the corresponding 19-electron radical dianion $\mathbf{3}^{2\cdot-}$ induces $\eta^5 \to \eta^6$ interannular isomerization. In contrast to the equilibrium shift to the η^5-isomer in 18-electron complexes **2** and **3**, in their 19-electron analogs the equilibrium is shifted to the η^6-isomer.

Key words: fluorenyltricarbonylchromium anion, haptotropic rearrangement, electron-transfer induced reactions, cyclic voltammetry.

Electron-transfer induced reactions (ETIR) are promising for the development of new methods for synthesizing and modifying organometallic complexes. This is due to the significantly higher reactivity of the 17- and 19-electron complexes that are generated by electron transfer in various reactions in comparison with their 18-electron precursors.[1—3] Interannular haptotropic isomerization (IHI) reactions are one type of ETIR that occur in a number of sandwich and semisandwich complexes of transition metals with polycyclic aromatic ligands. For example, electron transfer is known to be able to induce the substitution of π-ligands in sandwich complexes, change the haptobinding type in sandwich and semisandwich complexes,[5,6] *etc.* In addition to the synthetic aspect of the haptotropic isomerization reaction, the haptobinding changes are also of interest due to their part as key stages in many catalytic processes involving arenes,[7] in particular, the processes of their hydrogenation,[8] ligand substitution reactions and isomerization of organometallic complexes.[10] Therefore, widening the range of organometallic complexes taking part in these reactions, and studying their mechanisms as well as the ways to accelerate them are of undoubted interest.

We have previously shown[11] that the transfer of an electron to dibenzoferrocene (**4**) induces IHI to yield η^6-fluorenylcyclopentadienyliron (**5**). At the stage of the electrogenerated 19-electron anion radicals, this reaction takes place under markedly milder conditions and with a higher rate than at the stage of the 18-electron analogs of **4** and **5** (see Ref. 12). In principle, these reactions may be induced both by the action of chemical redox agents and electrochemically. In our opinion, the latter is preferable since the electron transfer occurs "in its pure aspect", and this process is finely controlled by the potential of the electron, which permits one to define the conditions of its occurrence.

In the present study the IHI reactions are extended to the anion $(\eta^5\text{-fluorenyl})Cr(CO)_3^-$ ($^-\mathbf{3}$). As has been previously shown,[14] the 18-electron complex $(\eta^6\text{-fluorenyl})Cr(CO)_3^-$ ($^-\mathbf{2}$) is isomerized at equilibrium to $\mathbf{3}^-$ with the η^5-isomer predominating. At the stage of the formation of the electrogenerated 19-electron complexes of the substances under investigation the equilibrium is displaced in the opposite direction.

Results and Discussion

In the cyclic voltammograms of the $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ complex (**1**) there is an irreversible one-electron cathodic peak (A_1) controlled by diffusion (Fig. 1, *a*) over the temperature range examined. This peak is characterized by comparing its height with the

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Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 1, pp. 48—53, January, 1994.

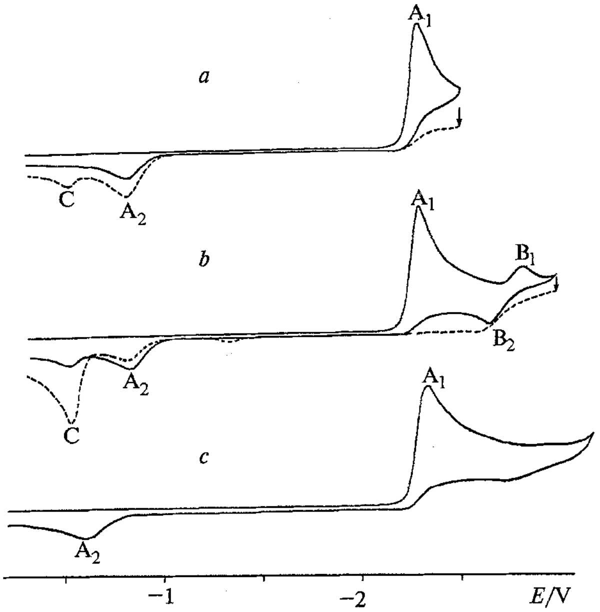


Fig. 1. Cyclic voltammograms of the $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ complex (**1**; $1 \cdot 10^{-3}$ mol L^{-1}) in a THF/0.05 mol L^{-1} Bu_4NPF_6 medium on a Pt electrode (v = 0.2 V s^{-1}) at 20 °C (*a*, *b*) and −30 °C (*c*). The dashed lines show voltammograms recorded after potentiostatic exposure of the electrode at the potentials indicated by the arrows. Peak heights at various temperatures are normalized to the height of a ferrocene oxidation peak under otherwise equal conditions.

Table 1. The potentials of peaks in cyclic voltammograms of complexes **1**, **2**$^-$, **3**$^-$, and **4** on a Pt electrode in THF/0.1 *M* Bu_4NPF_6 at v = 0.2 V s^{-1} and at 20 °C

Complex	Peak	E_p/V (sce)
1	A_1	−2.26
	A_2	−0.82
	B_1	−2.73 (−2.68)*
	C	−0.48
2$^-$	B_1	−2.69
	C	−0.48
3$^-$	D_1	−2.88
	D_2	−0.37 (−0.40)*
4	A_1	−2.26 (−2.23)*
	A_2	−0.86

* The values of $E^0 = (E_p{}^a - E_p{}^c)/2$ are shown in brackets, where $E_p{}^a$, $E_p{}^c$ are the potentials of the anodic and cathodic peaks, respectively.

height of a one-electron ferrocene oxidation peak under the same conditions and the quick transformation of the initially generated 19-electron anion radical $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3{}^{-}$ into the product oxidized at the peak A_2 potentials in the anodic branch of the voltammograms. In addition to peak A_1 in the cathodic branch of the $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ voltammogram, over the more negative potential range the quasi-reversible pair of peaks B_1/B_2, which are smaller in height, is registered (Fig. 1, *b*). Changing the potential scanning direction results in the appearance of the additional peak C in the anodic branch of the voltammogram (Fig. 1, *b*). Peak C is also observed when the electrode is kept at potentials between the potentials of peaks A_1 and B_1. The potentials of peaks B_1 and C coincide with the reduction and oxidation potentials, respectively, for the $(\eta^6\text{-}C_{13}H_9)Cr(CO)_3{}^{-}$ anion ($^-$**2**, Table 1) formed during deprotonation **1** with excess Bu^tONBu_4 in solution (see below). The temperature depression leads to a decrease in the heights of the B_1/B_2 and C peaks and at $T < -60$ °C only the irreversible peaks A_1 and A_2 are observed in the voltammograms of complex **1** (Fig. 1, *c*).

Preparative electroreduction of complex **1** at 20 °C and at the peak A_1 potentials is accompanied by the consumption of 1 F mol^{-1} and the appearance of peaks B_1 and C. The product oxidized at the potentials of anodic peak A_2 is not formed during the electrolysis, thus, the product of the preparative electroreduction of **1** is only the η^6-fluorenyl complex **2**$^-$. At electrolysis time (τ) > 5 min in the voltammograms of the solution of **1** the irreversible cathodic peak D_1 and the reversible anodic peak D_2 arise. Their potentials coincide with the potentials of the corresponding peaks of the reduction and oxidation of $(\eta^5\text{-}C_{13}H_9)Cr(CO)_3{}^{-}$ ($^-$**3**) measured independently (see below). In time the analogous peaks also emerge in the voltammograms of the solution of **2**$^-$ formed during deprotonation of **1** by excess Bu^tONBu_4. The appearance of peaks corresponding to the redox reactions of η^5-fluorenyl complex **3**$^-$ in voltammograms of a solution of **2**$^-$ at room temperature indicates that $\eta^6 \rightleftharpoons \eta^5$ interannular haptotropic isomerization occurs (see Refs. 15, 16).

Unlike those of complex **1**, the voltammograms of $(\eta^6\text{-}9,9\text{-}Me_2C_{13}H_8)Cr(CO)_3$ complex (**6**) contain only the one-electron reduction peak A_1 and anodic irreversible peak A_2 (Fig. 2, *a*). As the temperature is reduced,

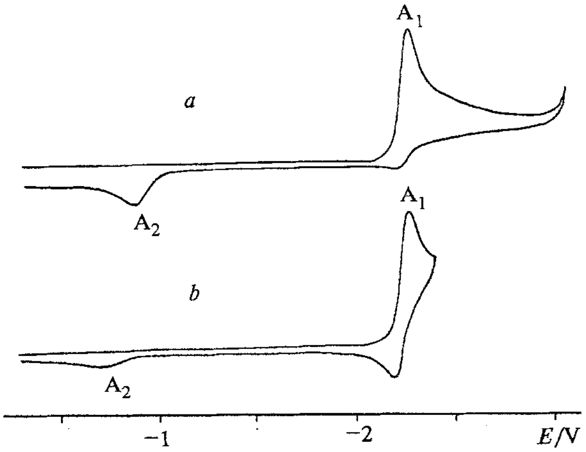
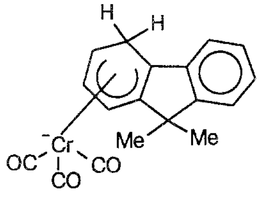


Fig. 2. Cyclic voltammograms of the $(\eta^6\text{-}9,9\text{-}Me_2C_{13}H_8)Cr(CO)_3$ complex (**6**; $1 \cdot 10^{-3}$ mol L^{-1}) at 20 °C (*a*) and −30 °C (*b*). Conditions are given in Fig. 1.

peak A_2 disappears and peak A_1 becomes reversible, which gives evidence for the stability of the electrogenerated 19-electron anion radical $\mathbf{6}^{\cdot -}$ at this temperature in the time scale of cyclic voltammetry (Fig. 2, *b*).

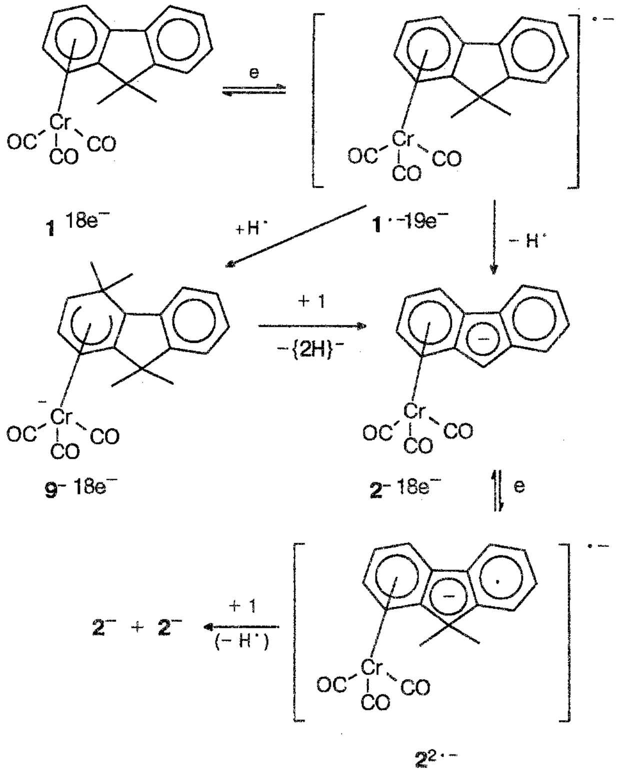
Reduction of complex **6** with an equimolar amount of sodium naphthalide in THF at −70 °C results in the appearance of the absorption band (AB) characteristic of carbonyl groups in the IR-spectrum of the reaction mixture: 1734, 1805 (band E) and 1880 cm^{-1} (band A). Analogous AB are registered in the IR-spectra of $(\eta^5\text{-}C_6H_7)Cr(CO)_3^-\ {}^+K$ (1740, 1790 and 1890 cm^{-1}) (see Ref. 17) as well as $(\eta^5\text{-}C_{10}H_9)Cr(CO)_3^-\ Na^+$ (1745, 1800, 1895 cm^{-1}) (see Ref. 18). According to the MS-GLC data the product obtained by reduction of **6** followed by oxidation with $CuBr_2$ is protonated under the action of trifluoroacetic acid at −70 °C to form 3,4-dihydro-9,9-dimethylfluorene (**7**). The formation of analogous diene type substances is characteristic of the protonation reactions of η^5-cyclohexadienyl tricarbonylchromium complexes.[19] The evidence presented indicates that the product of **6** reduction oxidized at the peak A_2 potentials is the (1-3,4a,9a-η^5-3,4-dihydro-9,9-dimethylfluorene-3-yl)tricarbonylchromium anion ($\mathbf{8}^-$).



$\mathbf{8}^-$

After the reduction of **1** with sodium naphthalide at −70 °C the same AB characteristic of carbonyl groups are observed in the IR-spectra of the reaction mixture as after the reduction of **6**. This testifies that the transformation of the $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3^-$ anion radical generated by the reduction of **1** produces the (1-3,4a,9a-η^5-3,4-dihydro-9,9-dimethylfluorene-3-yl)tricarbonylchromium anion $[(\eta^5\text{-}C_{13}H_{11})Cr(CO)_3]^-$ ($\mathbf{9}^-$). Oxidation of this anion corresponds to peak A_2 in the voltammograms. The absence of complex $\mathbf{9}^-$ in the products of the preparative electroreduction of **1** is apparently related to the small life-time of $\mathbf{9}^-$, which is chemically converted into $\mathbf{2}^-$. On the other hand, as mentioned above, when the potential scanning is reversed following the potentials of peak A_1 as well as at reduced temperatures the anodic peak C of the oxidation of complex $\mathbf{2}^-$ is absent in voltammograms of **1**, which also points to the consecutive formation of $\mathbf{2}^-$ *via* intermediate $\mathbf{9}^-$.

Scheme 1



In accordance with Scheme 1, complex **1** is converted into the 19-electron radical anion $\mathbf{1}^{\cdot -}$ by one-electron reduction, and $\mathbf{1}^{\cdot -}$ is then subjected to interannular haptotropic $\eta^6 \rightarrow \eta^5$ isomerization followed by the addition of a H atom and formation of complex $\mathbf{9}^-$. The latter is an unstable compound and undergoes subsequent chemical transformations resulting in complex $\mathbf{2}^-$. However, the data obtained don't exclude the possibility of the parallel generation of $\mathbf{2}^-$ from radical anion $\mathbf{1}^{\cdot -}$ as a result of splitting off a H atom at position 9 of the fluorene ligand. To confirm the proposed mechanism, a detailed investigation of the kinetics is necessary.

During electrode exposure in the range of potentials more negative than the peak B_1 potential followed by anodic scanning the height of peak C in the anodic branch of the voltammograms significantly increases, while at potentials more positive than that of peak B_1 the height of peak C does not increase. Taking into consideration the highly basic properties of the radical dianion $\mathbf{2}^{2\cdot -}$ electrogenerated at the peak B_1 potentials it can be assumed that this radical dianion deprotonates the initial complex **1** in the layer near the electrode to produce two anions $\mathbf{2}^-$ (see Scheme 1). Therefore, one-electron reduction of $\mathbf{2}^-$ and consequent interaction of $\mathbf{2}^{2\cdot -}$ with **1** results in the formation of two molecules of $\mathbf{2}^-$, that is, at peak B_1 potentials the formation of $\mathbf{2}^-$ is an autocatalytic reaction with the participation of an electrogenerated base. Under the conditions of cyclic voltammetry the small height of catalytic peak B_1 seems to be caused by the low stationary concentration of complex $\mathbf{2}^-$ and/or substrate **1** in the layer near the electrode, which is reduced to form $\mathbf{9}^-$ in the diffusion conditions at these potentials.

In principle, radical anion $\mathbf{1}^{\cdot -}$ may be an alternate product (by protonation of atom C(9) of the fluorenyl ligand) of electrogenerated radical dianion $\mathbf{2}^{2\cdot -}$ protonation. However, as discussed earlier, the chemical transformations of $1^{\cdot -}$ result in the generation of cyclohexadienyl complex 9^-, which is stable in the time scale of cyclic voltammetry. Hence, the existence of this channel should lead to a significant increase in the height of anodic peak A_2, but this is not observed in the experiment described above.

According to Scheme 1, the η^5-cyclohexadienyl complex 9^- is a product of the transformation of the electrogenerated anion radical $1^{\cdot -}$. It is evident from the literature[19] that complex 9^- is able to eliminate a hydride ion to regenerate the initial complex **1**. In its turn, the hydride ion can interact with the initial complex **1** to form 2^-. The small height of peak B_1 corresponding to the reduction of 2^- and the peak of the one-electron reduction of **1** testify to the low rates of these reactions in the time scale of cyclic voltammetry. As the temperature is reduced, the stability of 9^- increases, which is indicated by the disappearance of peak B_1 at $T < 60$ °C. The reduction of complex 2^- formed in the layer near the electrode at peak B_1 potentials is an autocatalytic reaction with the participation of an electrogenerated base leading to the accumulation of 2^- in the layer near the electrode.

The evidence obtained contradicts the conclusions made in publications,[20,21] according to which in the layer near the electrode the electrogenerated 19-electron anion radical $1^{\cdot -}$ undergoes the elimination of a H atom to yield 2^-. These authors erroneously attribute peak A_2 to 2^- oxidation, peak B_1 to reduction of the η^5-isomer of 3^-, and peak C to oxidation of the product of the quasi-reversible reduction of 3^-. The peaks mentioned* correspond to the electrode reactions of complexes 9^- (peak A_2) and 2^- (peaks B_1 and C). Moreover, in accordance with the experimental data for deprotonation of **1** in a solution with excess Bu^tONBu_4, the formation of complex 3^- in electrochemically detectable amounts during the $\eta^6 \rightarrow \eta^5$-isomerization of complex 2^- at room temperature is achieved at $\tau > 5$ min, which significantly exceeds the time scale of cyclic voltammetry. At peak D_1 potentials more negative than the peak B_1 potential (see Table 1), the irreversible two-electron reduction of complex 3^- occurs.

It is known[16] that at room temperature 18-electron η^6-fluorenyl complex $2^{\cdot -}$ undergoes reversible interannular haptotropic isomerization to produce the η^5-ISOMER of 3^- in the ratio of $2^-:3^- = 1:7$. For isoelectron fluorenyl iron complexes $(\eta^6\text{-}C_{13}H_9)Fe(\eta^5\text{-}C_5H_5)$ (**5**) and $(\eta^5\text{-}C_{13}H_9)Fe(\eta^5\text{-}C_5H_5)$ (**4**) the analogous reaction occurs only at $T > 80$ °C,[12] and the equilibrium $\eta^6 \rightleftharpoons \eta^5$ is strongly shifted toward the formation of η^5-isomer **4**. We have previously shown[11] that the one-electron reduction of dibenzoferrocene **4** to the 19-electron radical anion $4^{\cdot -}$ induces hapto-isomerization at temperatures as low as −80 °C. In contrast to 18-electron complexes, the $\eta^6 \rightleftharpoons \eta^5$ equilibrium between the 19-electron anion radicals is completely displaced toward the η^6-isomer $5^{\cdot -}$. Thus, in the transition from the 18-electron to the 19-electron state the reactivity of fluorenyl iron complexes in hapto-isomerisation changes significantly. Therefore, it is of interest to observe the effect of the transition from the 18-electron to the 19-electron state on the reactivity of fluorenylchromiumtricarbonyl complexes 2^- and 3^- in interannular haptotropic rearrangement.

As discussed above, the reduction of 3^- proceeds irreversibly and is accompanied by subsequent chemical conversions resulting probably in the destruction of the initial complex structure. Nevertheless, in the anodic branch of the voltammogram, peak C is observed, and its potential coincides with the potential of the oxidation of 2^- oxidation peak. The availability of this peak points to the occurrence of interannular $\eta^6 \rightleftharpoons \eta^5$ isomerization at the stage of 19-electron radical dianions, though the small height indicates the insignificant contribution of this process to the reactions undergone by electrogenerated radical dianion $3^{2\cdot -}$.

When a 10^{-3} mol L^{-1} THF solution of **1** is treated with excess Bu^tONBu_4 at room temperature the cathodic peak A_1 disappears completely, and only peaks B_1 and C are observed in the voltammograms (see Fig. 3, *a*) at the beginning ($\tau < 5$ min). Peak B_1 corre-

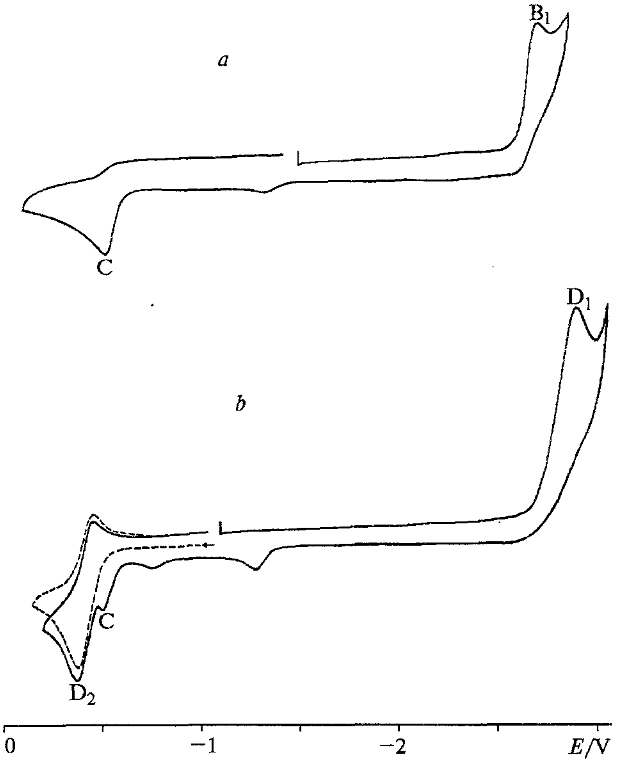


Fig. 3. *a*, Cyclic voltammogram of the $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ complex (**1**; $1 \cdot 10^{-3}$ mol L^{-1}) in the presence of excess Bu^tONBu_4 at 20 °C; *b*, cyclic voltammogram of the $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3^-$ anion ($^-$**3**; $1 \cdot 10^{-3}$ mol L^{-1}) at 20 °C. Conditions are given in Fig. 1.

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* The peak designations previously used[21] are retained in the present work.

sponds to the irreversible character of the process, which seems to be related to protonation of radical dianion $\mathbf{2}^{2\cdot-}$ that is generated at these potentials in the layer near the electrode by the tert-butanol formed in the deprotonation of **1** in the solution. In time the heights of the B_1 and C peaks decrease, and the reversible anodic peak D_2 and the irreversible cathodic peak D_1 appear in the voltammograms. Their potentials coincide with that of the peaks of the oxidation and reduction of $\mathbf{3}^-$, respectively. After 70 min the height of peak C is no more than 10 % of its initial height, and essentially only peaks D_1 and D_2 are registered on the voltammograms. Such behavior corresponds to the previously observed reaction of interannular haptotropic isomerization of 18-electron complexes $\mathbf{2}^- \rightleftharpoons \mathbf{3}^-$ (see Ref. 16). In the voltammograms of complex $\mathbf{2}^-$ the absence of peaks D_1 and D_2 at the beginning of experiment demonstrates that the reduction of $\mathbf{2}^-$ is not accompanied by $\eta^6 \to \eta^5$ isomerization, at least for the time of the voltammetric measurements.

Thus, the transfer of an electron to the η^5-isomer of $\mathbf{3}^-$ induces the $\eta^6 \to \eta^5$ isomerization reaction here as well as in the case of fluorenyl complexes of iron **4** and **5**. In the case of the 19-electron complexes of iron as well as chromium the direction of the interannular haptotropic isomerization reaction is the opposite of that of the 18-electron analogs. Unfortunately, the presence of the dominant contribution of the radical dianion $\mathbf{3}^{2\cdot-}$ disappearance channel does not permit one to quantitatively compare the kinetic and thermodynamic parameters of the hapto-isomerization reactions $\mathbf{5}^{\cdot-} \rightleftharpoons \mathbf{4}^{\cdot-}$ and $\mathbf{3}^{2\cdot-} \rightleftharpoons \mathbf{2}^{2\cdot-}$.

Experimental

The electrochemical measurements were carried out in a medium of absolute THF purified by the ketyl method with distillation of the purified solvent directly into a cell previously evacuated and then filled with dry argon according to the procedure described earlier.[22] The electrolyte was a 0.05 *M* solution of Bu_4NPF_6 in THF. To remove the traces of moisture the weighted sample of Bu_4NPF_6 was preliminary melted in a vacuum. Cryogenic electrochemical measurements were performed with cooling of the electrochemical cell by an ethanol—liquid nitrogen mixture in a Dewar vessel. Controlled potential electrolysis was carried out in a diaphragm cell on a base mercury cathode. The electrochemical measurements were performed using blocks of the electrochemical system PAR 370.

MS-GLC analysis was carried out on a Kratos DB 1073 mass-spectrometer (70 eV) (ion-source temperature was 200 °C) equipped with a DS-5 (J & W) chromatography column (0.32 mm×60 mm×0.1 μ) (temperature program: 50 °C(2′) — 4 °C/min, heating up to 250 °C, throw off 1:10). IR spectra were recorded on a Specord 75IR instrument.

Complexes $\mathbf{2}^-$ and $\mathbf{3}^-$ were obtained using standard procedures.[15] Complex $\mathbf{3}^-$ was also generated in an electrochemical cell by two-electron reduction of $[\eta^5\text{-}C_{13}H_9)Cr(CO)_3]_2Hg$ obtained as described earlier.[15] All experiments were performed in a purified argon atmosphere using absolute solvent media.

Reduction of η^6-9,9-dimethylfluorentricarbonylchromium (6) with sodium naphthalide followed by protonation with trifluoroacetic acid. Sodium naphthalide (0.056 g, 0.37 mmole) was added to a solution of **6** (0.12 g, 0.37 mmole) in THF (40 mL) at −70 °C. After 15 min CF_3COOH (0.5 mL, 6.5 mmoles) was introduced into the reaction mixture and the resulting dark red solution changed to light orange when heated to 20 °C. Then $CuBr_2.H_2O$ (0.1 g, 0.41 mmole) was added to the solution, the reaction mixture was poured into diethyl ether, and the ether solution was carefully washed with water and dried over $MgSO_4$. After removal of the ether the residue was dissolved in hexane and passed through silicagel (50×150 μ, Chemapol). After removal of the hexane an oily light liquid was obtained. According to MS-GLC data the liquid contained a mixture of naphthalene, 9,9-dimethylfluorene, and 9,9-dimethyl- 3,4-dihydrofluorene (**7**) in an amount of no more than 10 %.

Acknowledgements. The authors are very thankful to S. V. Vitt for performing the MS-GLC analysis.

This work was carried out with financial support from the Russian Foundation for Basic Research (No. 93-03-5209).

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Received October 1, 1993