Oxidation of isomeric η^6- and η^5-fluorenylchromiumtricarbonyl anions

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The oxidation of the carbon-centered $[(\eta^6\text{-}C_{13}H_9)Cr(CO)_3]^-$ anion (**1**$^-$) results in formation of (μ-η^6:η^6-9,9′-bifluorenyl)bis-chromiumtricarbonyl (**3**) due to coupling of the intermediate carbon-centered radical (**1**$^\bullet$). The oxidation of the metal-centered anion $[(\eta^5\text{-}C_{13}H_9)Cr(CO)_3]^-$ (**2**$^-$), which is isomeric to the **1**$^-$ anion, gives an equilibrium mixture of the chromium-centered radical $\{(\eta^5\text{-}C_{13}H_9)Cr(CO)_3\}^\bullet$ (**2**$^\bullet$) and its dimer $[(\eta^5\text{-}C_{13}H_9)Cr(CO)_3]_2$ (**6**). Radical **2**$^\bullet$ readily reacts with MeI and the solvent (THF); the resulting derivatives, $(\eta^5\text{-}C_{13}H_9)Cr(CO)_3R$ (R = Me (**10**); R = H (**7**)), undergo fast ricochet inter-ring $\eta^5 \rightarrow \eta^6$ rearrangements into $(\eta^6\text{-}9R\text{-}C_{13}H_9)Cr(CO)_3$ (R = CH_3 (**9**); R = H (**4**)).

Key words: transition metal complexes, free radicals, oxidation, dimerization.

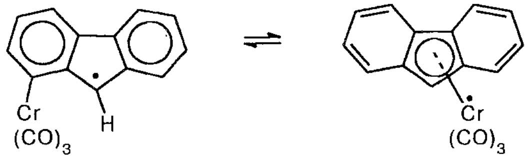
Owing to the growing use of odd-electron (17$\bar{e}$ and 19$\bar{e}$) complexes of transition metals in practical organometallic synthesis,[1,2,3] studies of similar reactions involving odd-electron compounds and their 18-electron precursors are of current interest since they provide valuable data on the changes in reactivity as the electronic configuration of the metal changes.

A typical example is a shift of equilibrium and an increase in the rate of inter-ring haptotropic rearrangements in isomeric η^6- and η^5-fluorenyl complexes of iron and chromium on transition from 18-electron to 19-electron compounds.[4,5,6]

However, it remains unclear whether such rearrangements can occur in the case of 17-electron complexes.

The aim of the present work is to determine the possibility of mutual transformations of a carbon-centered radical (**1**$^\bullet$) and a 17e chromium-centered radical (**2**$^\bullet$) (Scheme 1).

Scheme 1



1$^\bullet$ **2**$^\bullet$

In this work we studied the chemical and electrochemical oxidation of chromiumtricarbonyl(η^6-fluorenide) anion (**1**$^-$) and tricarbonyl(η^5-fluorenyl)chromate anion (**2**$^-$), respectively, for generating radicals **1**$^\bullet$ and **2**$^\bullet$.

An additional incentive for this study arose from the fact that, although 17-electron arene and cyclopentadienyl complexes of chromium subgroup metals, such as $(\eta^6\text{-Arene})_2Cr^+$,[7,8] $[(\eta^6\text{-Arene})M(CO)_3]^+$,[9–13] and $\{(\eta^5\text{-}C_5R_5)(CO)_2LCr\}^\bullet$ (R = H, Me; L = CO, PR_3, and $P(OR)_3$),[14–18] have been reported in the literature, similar complexes based on aromatic polycycles have not been studied.

Results and Discussion

As we have shown previously,[6] the η^6-fluorenylchromiumtricarbonyl anion (**1**$^-$) undergoes irreversible one-electron oxidation in THF at room temperature. The electrode process remains irreversible when the temperature is decreased to −80 °C. This suggests a high rate of subsequent transformations of radical species **1**$^\bullet$ generated by oxidation of anion **1**$^-$. Taking into account that the oxidation potential of anion **1**$^-$ is −0.48 V (*vs.* SCE), the reactivity of species **1**$^\bullet$ can be studied if it is generated by oxidizing anion **1**$^-$ with $AgBF_4$.

The preparative oxidation of **1**$^-$ with an equimolar amount of the $AgBF_4 \cdot 3C_4H_8O_2$ complex at −10 °C in THF followed by chromatographic separation gave (μ-η^6:η^6-9.9′-bifluorenyl)-*bis*-chromiumtricarbonyl (**3**, 30 %) and (η^6-fluorene)chromiumtricarbonyl (**4**, 10 %) (Scheme 2).

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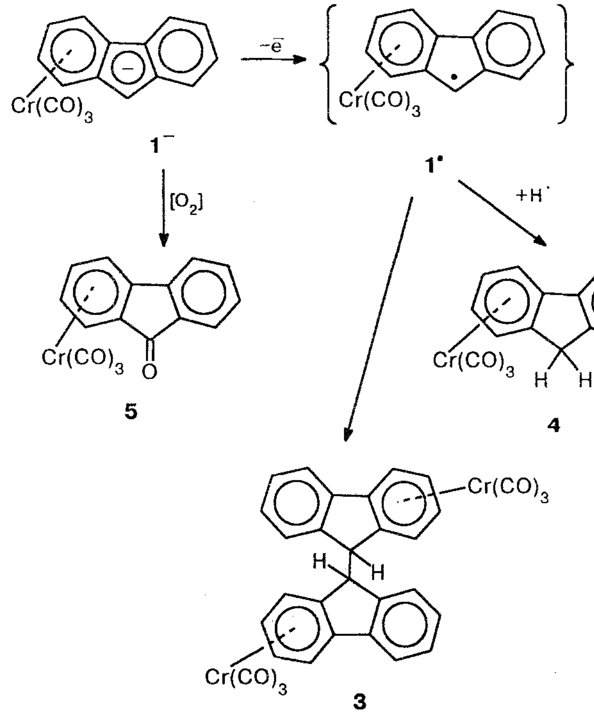
Scheme 2



The structure of binuclear complex **3**, which contains a bridging bifluorenyl ligand, was determined by IR, ^{1}H, and ^{13}C NMR spectroscopy and by FAB mass spectrometry. The FAB mass spectrum of compound **3** contains a molecular ion peak M^+ (*m/z* 602) and its fragmentation peaks.

The presence of two singlets of H(9) at δ 4.50; 4.56 (C_6D_6) and 4.78; 4.80 (CD_2Cl_2) in the ^{1}H NMR spectra suggests that complex **3** is a mixture of diastereomeric *meso- (RS, SR)* and racemic (*RR, SS*) forms. The formation of compound **3** as the main reaction product results from coupling of the C(9)-centered radical **1**$^\bullet$. This is consistent with the formation of η^6-fluorenone complex **5** when anion **1**$^-$ is oxidized with oxygen in the air (Scheme 2). Dimeric complex **3** is quite stable in the solid state but decomposes to give compound **4** in solutions exposed to light or during thin-layer chromatography. Thus, radical **1**$^\bullet$ undergoes rapid dimerization, and we did not succeed in observing a $\eta^6 \rightarrow \eta^5$ rearrangement for it.

Unlike the η^6-anionic complex **1**$^-$, the $[(\eta^5\text{-}C_{13}H_9)Cr(CO)_3]^-$ complex (**2**$^-$), which is isomeric to the former, undergoes reversible one-electron oxidation at room temperature in THF (E^0 = −0.40 V, SCE).[6] This agrees with the examples of reversible oxidation of the $[(\eta^5\text{-}C_5R_5)Cr(CO)_3]^-$ complexes (R = H, Me) reported in the literature[14] and suggests that the oxidation of **2**$^-$ results in radical species **2**$^\bullet$, which is stable on the time scale of cyclic voltammetry. We studied the properties of these species generated by chemical oxidation of complex **2**$^-$ by $AgBF_4$.

Table 1. Parameters of ESR spectra for related chromium-centered radicals

Radical	g_{xx}	g_{yy}	g_{zz}	Ref.
$\{(\eta^5\text{-}C_5H_5)Cr(CO)_3\}^\bullet$	2.035	1.997	2.134	15
	1.997	2.030	2.117	19
$\{(\eta^5\text{-}C_5Me_5)Cr(CO)_3\}^\bullet$	2.997	2.019	2.121	15
$\{(\eta^5\text{-}C_5Ph_5)Cr(CO)_3\}^\bullet$	2.139	2.023	1.995	20
$\{(\eta^5\text{-}C_{13}H_9)Cr(CO)_3\}^\bullet$	2.055	2.004	1.971	*

* This work.

The preparative oxidation of **2**$^-$ was carried out in THF and in 2,5-dimethyltetrahydrofuran by treatment with a $AgBF_4$—1,4-dioxane complex or $AgBF_4$ at −30 to −40 °C (the latter experiment was carried out similarly to the oxidation of anion **2**$^-$ with $[AgBF_4 \cdot 3C_4H_8O_2]$ described in Experimental). The combination of spectral data (ESR, IR) and the composition of the reaction products makes it possible to assume that an equilibrium between the metal-centered radical **2**$^\bullet$ and dimer **6** exists in the reaction solution (Scheme 3, *a*). The presence of radical **2**$^\bullet$ in the mixture was confirmed by ESR spectra of solid frozen solutions of the products of **2**$^-$ oxidation at 77 K in THF and 2,5-dimethyltetrahydrofuran. In both cases we detected identical signals (Fig. 1) of chromium-centered paramagnetic complex characterized by orthorhombic symmetry and three-axial anisotropy of the *g*-factor. The shape of the signal recorded, the *g*-factors observed, and their comparison with the literature data for related compounds (Table 1) suggest that the signal recorded probably corresponds to 17-electron η^5-fluorenylchromiumtricarbonyl radical **2**$^\bullet$ with the metal atom in the d^5 electronic configuration. Heating the samples to room temperature results in distortion of signal shape and its rapid complete disap-

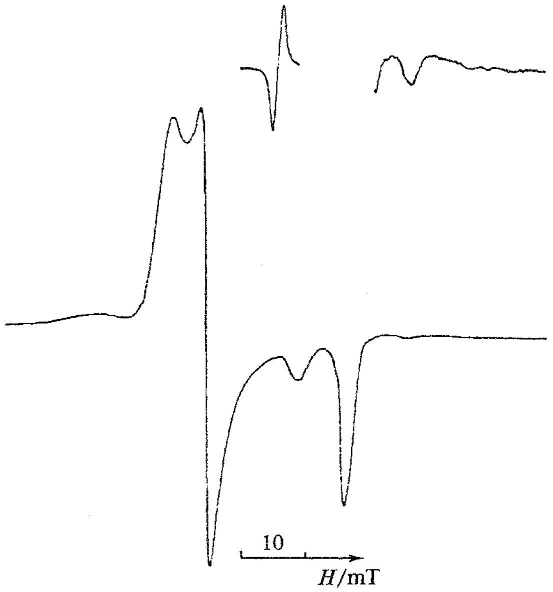
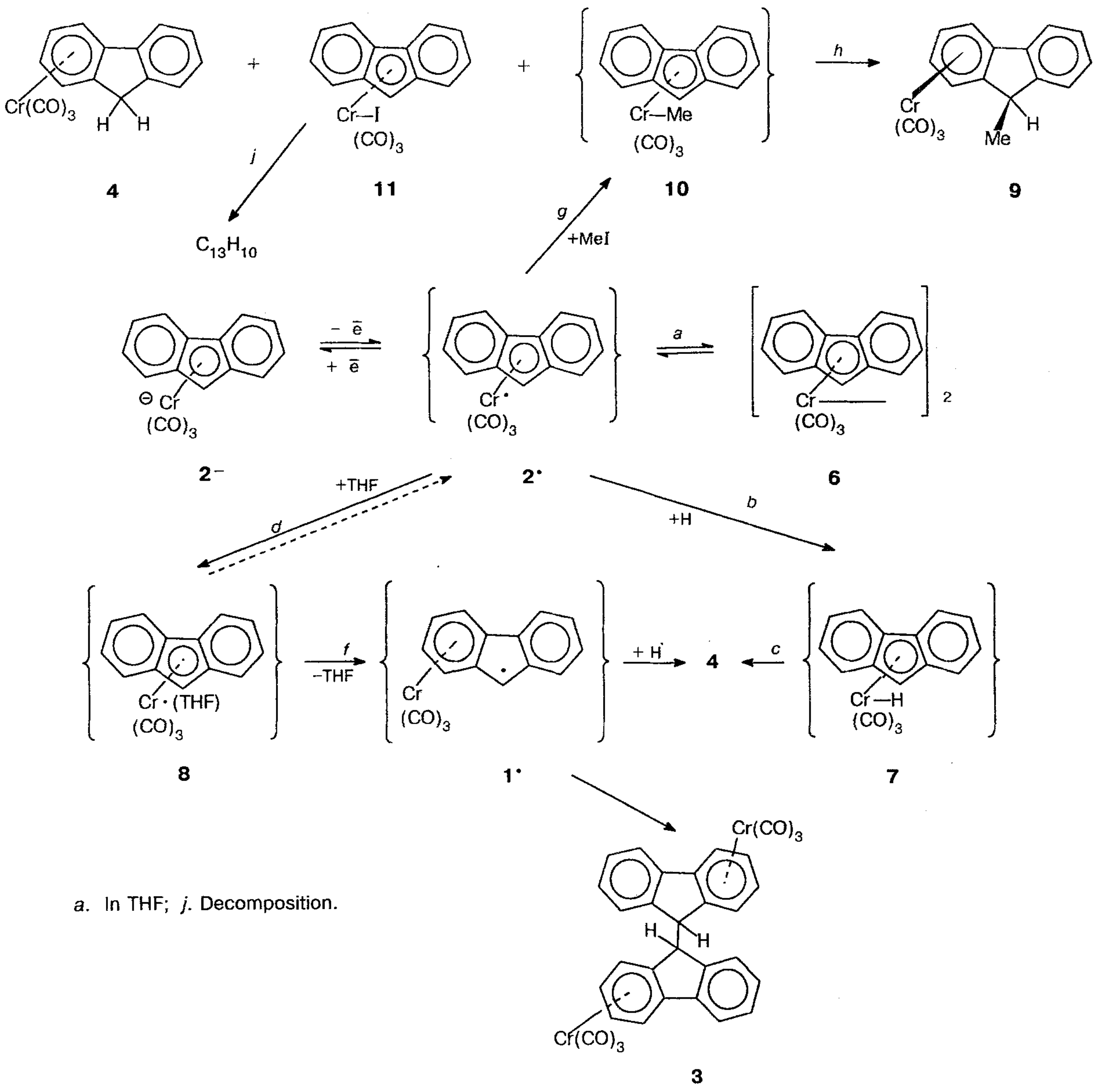


Fig. 1. ESR spectrum of the product of **2**$^-$ oxidation.

Scheme 3



a. In THF; *j.* Decomposition.

pearance. Immediately after addition of the oxidant to anion **2**$^-$, νCO bands at 1887 and 1927 cm^{-1}, which belong to dimer **6**, appear in the IR spectrum. The νCO frequencies for compound **6** agree with the literature data for structural analogs with a chromium—chromium bond, [(η^5-C$_5$Me$_5$)(CO)$_3$Cr]$_2$ 1876, 1902, and 1919 cm^{-1} (toluene)[16] and 1886, 1915 cm^{-1} (tetradecane).[21] The absence of νCO bands of radical **2**• is caused by its short life time and its low concentration in the solution.

The removal of radical **2**• from the reaction solution occurs, primarily, due to trapping of hydrogen from the solvent and ricochet inter-ring $\eta^5 \rightarrow \eta^6$ rearrangement of hydride (η^5-C$_{13}$H$_9$)(CO)$_3$CrH (**7**) into the η^6-form **4** (Scheme 3, *b*+*c*).[22]

Another possibility for the transformation of radical **2**• is the formation of a 19-electron adduct with a THF molecule, **8** (Scheme 3, *d*) accompanied by inter-ring rearrangement **8**→**1**• (Scheme 3, *f*) and trapping of a hydrogen atom from the solvent, **1**•→**4**. Thus, both types of transformation of radical **2**• should result in compound **4**, but pathway (*d*+*f*) also implies the formation of dimer **3**. In fact, we observed the formation of the latter in a low yield when anion **2**$^-$ was oxidized in THF, whereas complex **3** was not detected when the reaction occurred in 2,5-dimethyltetrahydrofuran. This difference originates from a lower coordinating ability of 2,5-dimethyltetrahydrofuran, which hampers the formation of adduct **8**. The negligible yield of complex **3** in THF suggests that the transformation of radical **2**• predominantly occurs through pathway (*b*+*c*), and only a minor amount reacts according to pathway (*d*+*f*).

Equilibria similar to those depicted in (Scheme 3, *a*), $2\{(\eta^5\text{-}C_5R_5)(CO)_2LCr\}^\bullet \rightarrow [(\eta^5\text{-}C_5R_5)(CO)_2LCr]_2$ (R = H, Me; L = CO, $P(OMe)_3$), have been studied in detail previously.[16] In agreement with the exothermic nature of dimerization, the concentration of monomeric radicals was reported to increase with increasing temperature. As noted above, in the case of equilibrium in Scheme 3, *a* the concentration of radicals decreases when the temperature increases, which is caused by their effective removal from the reaction bulk.

The action of methyl iodide on the oxidation product **2**$^\bullet$ confirms the proposed scheme of transformations of radical **2**$^\bullet$. In this case, the following compounds were isolated: the η^6-9-*endo*-methylfluorene (**9**) and η^6-fluorene (**4**) complexes, respectively, in a 3.5 : 1 ratio. It has previously been found that the reactions of the $\{(\eta^5\text{-}C_5R_5)Cr(CO)_3\}^\bullet$ 17-electron radicals with alkyl halides RX result in mixtures of the corresponding σ-alkyl complexes, $(\eta^5\text{-}C_5R_5)(CO)_3Cr{-}R$, and halide complexes, $(\eta^5\text{-}C_5R_5)(CO)_3CrX$.[16] Therefore, we could expect the formation of the following complexes: $(\eta^5\text{-}C_{13}H_9)(CO)_3CrMe$ (**10**) and $(\eta^5\text{-}C_{13}H_9)(CO)_3CrI$ (**11**).

The latter was not isolated because of its instability (the previous attempts at its synthesis failed[22]), while the σ-methyl complex **10** undergoes fast ricochet inter-ring rearrangement (Scheme 3, *h*) to give complex **9**.

Thus, we have shown in the present work that the oxidation of isomeric carbon-centered anion **1**$^-$ and metal-centered anion **2**$^-$ results in carbon-centered radical **1**$^\bullet$ and metal-centered radical **2**$^\bullet$, respectively. Radical **1**$^\bullet$ undergoes dimerization into complex **3** or traps H$^\bullet$ from the solvent to give complex **4**. We did not obtain any evidence of the $\mathbf{1}^\bullet \rightarrow \mathbf{2}^\bullet$ transformation (Scheme 1). Radical **2**$^\bullet$, which exists in equilibrium with dimer **6**, adds H$^\bullet$ from the environment and a methyl group from MeI, and the resulting derivatives $(\eta^5\text{-}C_{13}H_9)(CO)_3CrR$ rearrange into η^6-complexes such as $(\eta^6\text{-}9R\text{-}C_{13}H_9)Cr(CO)_3$, R = H (**4**), Me (**9**). The formation of dimer **3** in the oxidation of anion **2**$^-$ suggests that radical **1**$^\bullet$ appears during the reaction, and the inter-ring $\eta^5 \rightarrow \eta^6$-isomerization $\mathbf{2}^\bullet \rightarrow \mathbf{1}^\bullet$ probably occurs *via* complex **8**, a 19-electron adduct of **2**$^\bullet$ with THF.

Experimental

All reactions were carried out under dry purified argon. THF and 2,5-dimethyltetrahydrofuran were dried with benzophenoneketyl. ^{1}H and ^{13}C NMR spectra were obtained on Varian-VXR-400 and Bruker WP-200SY spectrometers. The mass spectrum was recorded on a KRATOS CONCEPT mass spectrometer by the FAB method with an energy of bombarding ions (Cs) of 8 keV and 2-nitrobenzyl alcohol as the matrix. ESR spectra were obtained on a Varian E 12A radio-frequency spectrometer. IR spectra were obtained on a Specord 75 IR spectrophotometer.

Electrochemical measurements were carried out according to the previously reported procedure.[6,23]

Oxidation of the $[(\eta^6\text{-}C_{13}H_9)Cr(CO)_3]^-K^+$ complex (1$^-$). The $[(\eta^6\text{-}C_{13}H_9)Cr(CO)_3]^-K^+$ complex (**1**$^-$) was obtained by the standard procedure[24] from $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ (**4**) (0.48 g, 1.35 mmol) and Bu^{t}OK (0.17 g, 1.52 mmol) in THF (40 mL) at −40 °C. The completeness of the transformation of **4** into **1**$^-$ was monitored by IR spectra in the νCO region. $[AgBF_4 \cdot 3C_4H_8O_2]$ (0.70 g, 1.52 mmol) was then added at −40 °C to the resulting solution of **1**$^-$. The mixture was stirred for 20 min, the temperature was adjusted to ~20 °C, and water (1 mL) was added.

The solvent was removed *in vacuo*, and the residue was treated with benzene and filtered. The benzene was concentrated, and the raw residue was reprecipitated from benzene with heptane. The resulting yellow crystalline compound was chromatographed on a column (250×30 mm) with SiO_2 (100/400 μm Chemapol). Elution of the first yellow band with benzene gave 0.040 g (10 %) of $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ (**4**); then the next less mobile band was eluted to give 0.130 g (30 %) of $[(\eta^6\text{-}C_{13}H_9)Cr(CO)_3]_2$ (**3**). M.p. 202—204 °C (dec.). Found (%): C, 63.97; H, 3.075; Cr, 17.21. $C_{32}H_{48}O_6Cr_2$. Calculated (%): C, 63.79; H, 2.99; Cr, 17.28. The ^{1}H and ^{13}C NMR spectral data are presented in Tables 2 and 3.

Oxidation of the $[(\eta^5\text{-}C_{13}H_9)Cr(CO)_3]^-Na^+$ complex (2$^-$). The $[(\eta^5\text{-}C_{13}H_9)Cr(CO)_3]^-Na^+$ complex (**2**$^-$) was obtained by the reduction of $[(\eta^5\text{-}C_{13}H_9)Cr(CO)_3]_2Hg$ [25] (0.13 g, 0.16 mmol) with excess sodium powder in THF (80 mL) with cooling by dry ice. After that, $[AgBF_4 \cdot 3C_4H_8O_2]$ (0.140 g, 10.30 mmol) was added with stirring and cooling to the resulting solution of anion **2**$^-$, and the mixture was stirred for 1 h with cooling by dry ice and then for 30 min at ~20 °C. The solvent was removed *in vacuo*, and the residue was treated with benzene and filtered. The benzene was concentrated, and the residue was reprecipitated from a benzene/heptane mixture. The resulting raw product was chromatographed on a column (100×25 mm) with SiO_2 (Porokvarts PKN-200, 50—150 μm). Elution with heptane gave fluorene (0.015 g, 28 %). The yellow band containing 21 % $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ (**4**) and the next weakly

Table 2. ^{1}H NMR spectra of complex **3**

A		B		C
δ	*J*/Hz	δ	*J*/Hz	δ
		C_6D_6 (TMS)		
6.455 (d, 1 H)	7.3	4.985 (d, 1 H)	6.3	4.505 (s, 1 H)
6.760 (t, 1 H)	7.3	4.930 (d, 1 H)	6.3	4.560 (s, 1 H)
6.920 (t, 1 H)	7.6	4.790 (d, 1 H)	6.0	
6.965 (d, 1 H)	7.6	4.360 (t, 1 H)	6.3	
7.040 (m, 4 H)		4.240 (m, 3 H)		
		3.980 (t, 1 H)	6.3	
		CD_2Cl_2 (TMS)		
7.50 (m, 1 H)		5.97 (d, 1 H)	~6	4.78 (s, 1 H)
7.20 (t, 1 H)	~7	5.90 (d, 1 H)	~6	4.81 (s, 1 H)
6.85 (d, 1 H)	~7	5.52 (d, 1 H)	~6	
		5.38 (t, 1 H)	~6	
		5.26 (m, 2 H)		
		5.00 (t, 1 H)	~6	
		4.58 (d, 1 H)	~6	

Note: A, the uncoordinated six-membered ring; B, the coordinated six-membered ring; C, signals of H in position 9 of the ligand.

Table 3. ^{13}C NMR spectra of complex **3** (C_6D_6; TMS, ppm)

A		B		D	E
I	II	I	II		
143.278	129.180	112.378	91.475(2C)	51.423	233.027
142.247	128.765	111.496	90.120		232.983
139.077	128.674	110.270	89.886		
138.749	128.479	109.906	89.363		
	124.632		89.217		
	124.480		84.965		
	120.119		84.906		
	120.053				

Note: A, the uncoordinated six-membered ring; B, the coordinated six-membered ring; C, signals of C_9 in the ligand; E, signals of the carbon atoms of CO groups; I, quaternary carbon atoms; II, tertiary carbon atoms.

yellow band containing traces of dimeric complex **3** (3 %) were eluted with benzene.

Reaction of $\{(\eta^5\text{-}C_{13}H_9)Cr(CO)_3\}^{\bullet}$ (2$^{\bullet}$) with MeI. $C_{13}H_9)Cr(CO)_3^-Na^+$ (**2**) was obtained as in the previous experiment from $[(\eta^5\text{-}C_{13}H_9)Cr(CO)_3]_2Hg$ (0.120 g, 0.28 mmol) in THF (70 mL). The oxidant was added, and after five minutes, excess methyl iodide (2.5 mL) was added with cooling (dry ice) to the reaction mixture. The mixture was stirred for 40 min with cooling and then for 30 min at ~20 °C. The solvent was removed *in vacuo*, and the residue was treated with benzene, filtered, and reprecipitated from benzene with heptane. The resulting raw product was chromatographed on a column (80×25 mm) with SiO_2 (Porokvarts PKN-200, 50—150 μm). Elution with heptane gave fluorene (0.025 g, 50 %); further elution with benzene gave 0.025 g (27 %) of a mixture of the complexes $(\eta^6\text{-}9\text{-}endo\text{-}MeC_{13}H_9)Cr(CO)_3$ (**9**) and $(\eta^6\text{-}C_{13}H_{10})Cr(CO)_3$ (**4**) in a 3.5 : 1 ratio (according to NMR spectral data).

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