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ELECTROREDUCTION OF (BENZOPHENONE)TRICARBONYLCHROMIUM AND (FLUORENONE)TRICARBONYLCHROMIUM IN THE PRESENCE OF ELECTROPHILES

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Summary

(Benzophenone)$Cr(CO)_3$ (**2**) and (fluorenone)$Cr(CO)_3$ (**3**) are reduced at a mercury pool cathode in aprotic media, in the presence of an electrophile which is more difficult to reduce. The results are compared with those which are obtained with the corresponding parent ketone. In the presence of 1,3-dibromopropane, a complexed olefinic alcohol **7a** is the major isolated compound when **2** is the substrate, whereas a complexed spiro ether **8b** is obtained with **3** as substrate. The electroreduction of **3** in acetonitrile in the presence of an equivalent amount of 4-chlorobutyryl chloride gives mainly the diacylated complex **10a**. The unexpected formation of a nitrile complex **12a** is also observed, which indicates an interaction between an intermediate species and acetonitrile. Complex **12a** is obtained in low yield by the electroreduction of **3** in the presence of 3-bromopropionitrile. Under these conditions the major compound is (fluorenol)$Cr(CO)_3$.

Introduction

The modifications to the chemistry of arenes which are caused by coordination to $Cr(CO)_3$ moieties explains the importance of the complexes (η^6-arene)$Cr(CO)_3$ (**1**) in chromium π-arene chemistry [1,2]. From the electrochemical point of view, the electron-withdrawing effect of the $Cr(CO)_3$ group facilitates the electroreduction of the arene ligand. For the coordinated ketones **2** and **3**, the standard redox potentials of the first electron uptake are -1.450 and -1.100 V, respectively (in DMF, relative to a SCE) whereas they are -1.776 and -1.278 V for the parent ketones [3].

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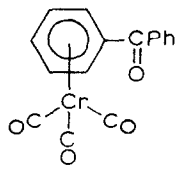

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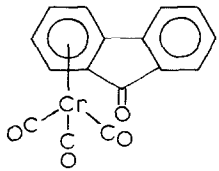

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Electroreduction or oxidation of complexes of type **1** leads, through electron exchanges, to complexes in which the arene ligand may be different from the molecule obtained when the parent compound is the substrate [4].

The electroreduction of complexes of type **1** in the presence of electrophiles which are more difficult to reduce has not so far been investigated. Considering the arene part of such reduced complexes, it can be anticipated that their nature and/or their yield may be different from the nature and/or the yield of the species obtained when the parent compound is the substrate. We have reduced the complexed ketones **2** and **3** in aprotic media in the presence of compounds **4–6** as electrophiles. The results are presented below and compared with those which were obtained with the parent ketones.

$Br(CH_2)_3Br$ (4)

$ClC(=O)(CH_2)_3Cl$ (5)

$BrCH_2CH_2CN$ (6)

Results and discussion

The electroreduction of **2** and **3** in the presence of **4** as a dielectrophile was performed in *N*,*N*-dimethylformamide (DMF). The experimental conditions are summarized in Table 1 which also indicates the nature of the isolated compounds and their yield. The results obtained previously with the parent ketones [5] are also presented in Table 1.

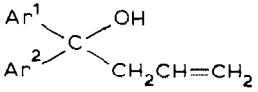
TABLE 1

ELECTROREDUCTION IN DMF OF **2**, **3** AND THE PARENT KETONES IN THE PRESENCE OF **4**

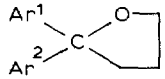
Ketone	**4** (equiv)	*n*	Applied potential (V vs. SCE)	Compounds isolated (% yield [a])		
				From addition to the carbonyl group	From substitution in the nucleus	Other compounds
2	1	1.8	−1.40	**7a** (36) **7b** (15)		**2** (26)
Benzophenone [b]	1	2.2	−1.72	**7b** (8) **8a** (10)	**9** (8)	Benzophenone (26) Benzhydrol (2)
3	2	1.8	−1.10	**8b** (64 [c])		**3** (18 [c]) Fluorenone (18 [c])
Fluorenone [b]	1	1.9	−1.40	**7c** (14) **8c** (41)	2 unidentified compounds (5)	

[a] After purification by column chromatography if not specified. [b] See ref. 5. [c] From NMR data.

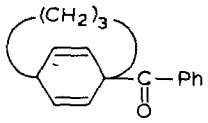
After total passage of the Faradaic current, the electroreduction of **2** gave the complexed olefinic alcohol **7a** as the main compound, together with its parent molecule **7b** and some substrate. **7a** which was very light-sensitive in solution, decomposed to give **7b**.



7 { **a**, $Ar^1 = C_6H_5$, $Ar^2 = (C_6H_5)Cr(CO)_3$
b, $Ar^1 = Ar^2 = C_6H_5$
c, Ar^1, $Ar^2 = C_6H_4-C_6H_4$



8 { **a**, $Ar^1 = Ar^2 = C_6H_5$
b, Ar^1, $Ar^2 = C_6H_4-(C_6H_4)Cr(CO)_3$
c, Ar^1, $Ar^2 = C_6H_4-C_6H_4$



(9)

Less selectivity in the product distribution was observed when benzophenone was reduced in the presence of dibromopropane [5]. Three reduced alkylated compounds were isolated in similar amounts. The alcohol **7b** and the cyclic ether **8a** resulted from attack at the carbonyl group whereas formation of the bridged ketone **9** involves attack at the nucleus. Some substrate was again isolated, together with a trace of benzhydrol.

In the case of **3**, the coordinated spiro ether **8b** was the only reduced moiety obtained. For fluorenone, the formation of the spiro ether **8c** competed with the formation of an olefinic alcohol.

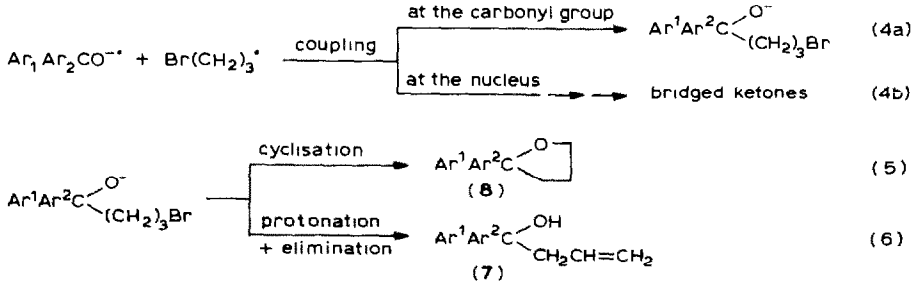
In each case, several reaction pathways are involved in the electroreduction process. Among these, reactions 1–6 were previously used to account for the formation of the compounds isolated from benzophenone and fluorenone [5]. All of these equations, except 4b, are compatible with the results observed with **2** and **3** as the substrates.

$$Ar^1Ar^2CO \overset{+e}{\rightleftharpoons} Ar^1Ar^2CO^{-\cdot} \tag{1}$$

$$Ar^1Ar^2CO^{-\cdot} + Br(CH_2)_3Br \rightleftharpoons Ar^1Ar^2CO + [Br(CH_2)_3Br]^{-\cdot} \tag{2}$$

(**4**)

$$[Br(CH_2)_3Br]^{-\cdot} \rightarrow Br(CH_2)_3^{\cdot} + Br^- \tag{3}$$



The direct reduction of **4** occurs at −2 V [6] but its indirect reduction can take place through an homogeneous electron transfer (eq. 2) between **4** and the cathodically-generated radical anion of the substrate (eq. 1). This exchange occurs because formation of the unstable species $\mathbf{4}^{-\cdot}$ (eq. 2) is followed by its fast decomposition (eq. 3). The bromopropyl radical thus generated can either couple according to eqs. 4a or 4b (the case with the parent ketones) or escape the coupling reaction and give, for instance, bromopropane. If the coupling reaction occurs according to eq. 4a, an alcoholate anion is generated, whose cyclisation (eq. 5) competes with its protonation (eq. 6).

For complexes **2** and **3**, the decrease of the aromaticity of the arene ligand because of the presence of the $Cr(CO)_3$ group [1] reduces the ease of nuclear couplings according to eq. 4b. No bridged ketones were isolated. The electron withdrawing properties of the $Cr(CO)_3$ group also decreases the basicity of the intermediate alcoholate anion generated according to eq. 4a. Therefore, the formation of **7** is less favoured with the complexes **2** and **3** than with the parent ketones, and so high yields of cyclic ethers of type **8** can be expected in the case of **2** and **3**. For complex **3**, the coordinated spiro ether **8b** was indeed isolated in high yield. Surprisingly, in the case of **2**, only the complexed olefinic alcohol **7a** was obtained. It can be suggested that steric hindrance by the $Cr(CO)_3$ group prevented the cyclisation reaction 5 and so the formation of **7a** was kinetically preferred.

In the four electrolyses summarized in Table 1, some bromopropyl radicals escaped the coupling reactions 4a and/or 4b and so less than one equivalent of **4** (two in the case of **3**) was effectively available for the coupling reactions. After depletion of the Faradaic current (at the end of the electrolysis), some substrate radical anions were left and these were reoxidised to the substrate by oxygen during treatment of the catholyte. In the case of **3**, the formation of the parent ketone in 18% yield would be related to the instability in DMF of the electrogenerated intermediate radical anion $\mathbf{3}^{-\cdot}$.

The electroreduction of **3** in the presence of an equivalent amount of 4-chlorobutyryl chloride **5** was performed in acetonitrile since acid chlorides are not stable in DMF [7,8]. The results showed much similarity with the results previously obtained for fluorenone [8] (Table 2). In both cases, the reduced form obtained in the highest yield was a diacylated derivative. Complex **10a** was very light-sensitive in solution,

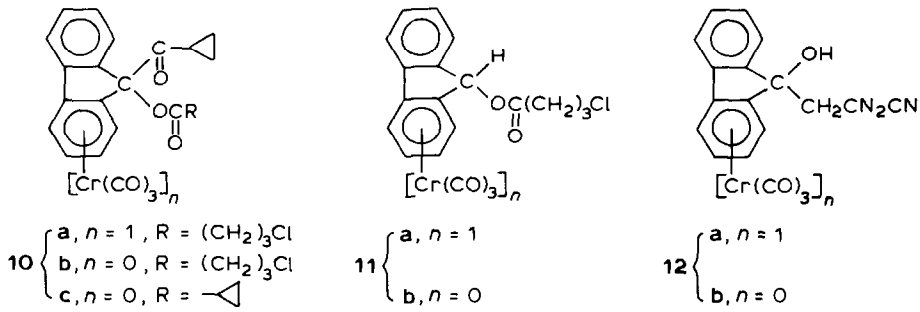
TABLE 2

ELECTROREDUCTION OF 3 AND FLUORENONE IN ACETONITRILE, IN THE PRESENCE OF ONE EQUIVALENT OF **5**

Ketone	*n*	Applied potential (V vs. SCE)	Compounds isolated (% yield [a])		
			From addition at the carbonyl group	Nitrile derivatives	Other compounds
3	1.8	−1.1	**10a** (21.5) **10b** (19) **11a** (7.5)	**12a** (13.5)	**3** (26)
Fluorenone [b]	1.7	−1.35	**10b** (17) **10c** (8) **11b** (3.5)	**12b** (5)	Fluorenone (15) Fluorenol (11)

[a] After purification by colum chromatography. [b] See ref. 8.

its decomposition giving the parent molecule **10b**. In both cases, the unexpected formation of a nitrile derivative was observed, showing that some reaction had occurred between the acetonitrile and an intermediate species. For fluorenone, a reaction sequence to the nitrile **12b** was tentatively suggested [9].

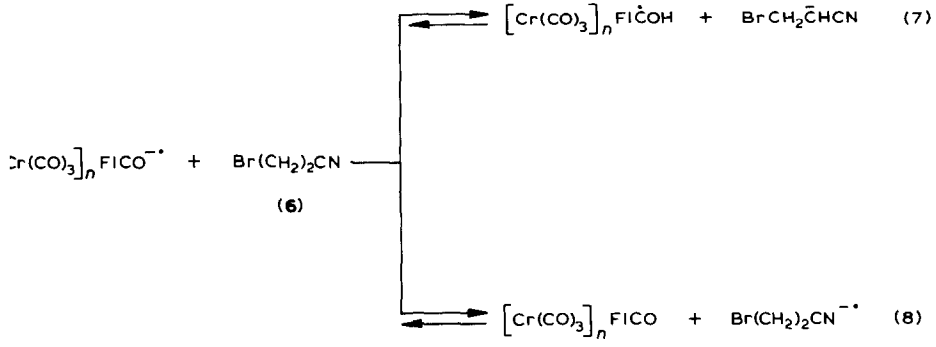


10 a, n = 1, R = $(CH_2)_3Cl$; b, n = 0, R = $(CH_2)_3Cl$; c, n = 0, R = cyclopropyl

11 a, n = 1; b, n = 0

12 a, n = 1; b, n = 0

The nitriles **12a** and **12b** were obtained in low yields by the electroreduction of **3** and of fluorenone in DMF, in the presence of **6** (Table 3). Because of the acidic nature of the electrophile **6**, protonation of the intermediate radical anion of the substrate occurred (eq. 7), and this competed with the formation of the nitrile derivatives by eqs. 8–11.



$$[Br(CH_2)_2CN]^{-\cdot} \rightarrow {}^{\cdot}CH_2CH_2CN + Br^- \quad (9)$$

TABLE 3

ELECTROREDUCTION IN DMF OF 3 AND OF FLUORENONE IN THE PRESENCE OF ONE EQUIVALENT OF **6**

Ketone	n	Applied potential (V vs. SCE)	Compounds isolated (% yield [a])	
			From addition at the carbonyl group	Other compounds
3	1.7	−1.1	**12a** (9)	(Fluorenol)$Cr(CO)_3$ (55)
Fluorenone	1.2	−1.4	**12b** (11)	Fluorenol (1)
				Bisfluorenol (58)

[a] After purification by column chromatography.

$$[Cr(CO)_3]_nFlCO^{-\cdot} + {}^{\cdot}CH_2CH_2CN \rightarrow [Cr(CO)_3]_nFlC\begin{matrix}O^-\\ \\ CH_2CH_2CN\end{matrix} \quad (10)$$

$$[Cr(CO)_3]_nFlC\begin{matrix}O^-\\ \\ CH_2CH_2CN\end{matrix} \xrightarrow{+6} [Cr(CO)_3]_nFlC\begin{matrix}OH\\ \\ CH_2CH_2CN\end{matrix} \quad (11)$$

12a,b ($n = 1, 0$)

$n = 0$ or 1; $Fl = C_6H_4{-}C_6H_4$

As a result of the electron-withdrawing properties of the $Cr(CO)_3$ group in complex **3**, both of the equilibria 7 and 8 are shifted more to the left for this substrate than for the parent ketone. In the case of **3**, both the proton exchange (eq. 7) and the homogeneous electron exchange (eq. 8) are also made more difficult because a decrease in the basicity of the radical anion $\mathbf{3}^{-\cdot}$ together with an increase of its oxidation potential occur. It is therefore not possible to predict whether the competition between 7 and 8 will be in favour of **12a** at the expense of (fluorenol)$Cr(CO)_3$. The experimental results in Table 3 show that the relative yields of nitriles and alcohols were not much changed by coordinating the fluorenone to a $Cr(CO)_3$ group. Only the nature of the main isolated alcohol was different. Probably for steric reasons, the complexed bisfluorenol could not be generated and so a further reduction of the radical generated in reaction 7 occurred to give (fluorenol)$Cr(CO)_3$.

Experimental

Complex **2** was prepared according to ref. 10. The electrosynthesis of **3** in 46% yield from (fluorene)$Cr(CO)_3$ [11] and electrogenerated superoxide anion is described elsewhere [12]. Solvents (DMF and acetonitrile) of analytical grade were carefully dried on neutral alumina.

An Amel-552 potentiostat and a Tacussel IG 5-N integrator were used. The three compartments of a H-type cell were filled with solvent containing 0.1 *M* Bu_4NPF_6. The cathode was a mercury pool, the anode a Pt grid and the reference electrode a saturated calomel electrode (SCE). The catholyte (60 ml) was deaerated with argon prior to the introduction of **2** or **3** (1 mmol if not specified) and of an electrophile (1 equivalent if not specified). The electrolyses were carried out under an argon atmosphere at constant potentials. Their values and the number of electrons consumed (n) are indicated in Tables 1–3. The electrolyses were stopped when the Faradaic current became negligible. The catholyte was diluted with water and the electrolysis products were extracted with diethyl ether. They were purified by column chromatography. Their yields are indicated in Tables 1–3.

^1H NMR spectra were recorded on a Perkin–Elmer R-24 spectrometer, IR spectra on a Perkin–Elmer 577 spectrophotometer and mass spectra on a Finnigan 3003 spectrometer (direct introduction, EI, 70 eV). Microanalyses were worked out by Service Central d'Analyse CNRS, Lyon.

Electroreduction of 2 (2 mmol) in the presence of 4 in DMF

The crude product (0.798 g) was subjected to column chromatography using 4/6 diethyl ether/hexane mixture as eluant. The compounds were isolated in the following order: **7b** [13], **7a**, **2**.

(1,1-Diphenylbut-3-en-1-ol)tricarbonylchromium (7a). Bright yellow needles, m.p. 107°C (from Et_2O/hexane). ^1H NMR ($CDCl_3$, TMS), δ 2.57 (s, 1 H, OH), 2.5–3.4 (m, 2 H, allylic H), 5.0–5.9 (mf, 8 H, vinylic and complexed aromatic H), 7.2–7.7 ppm (mf, 5 H, aromatic H); MS *m/e* 361 (9), 360 (M^+, 27), 277 (25), 276 (M – 3CO, 100), 259 (22), 258 (276 – H_2O, 76), 257 (18), 256 (23); IR (KBr), ν(max) cm^{-1} 3540 (OH), 3100, 3090, 1960 and 1875 (C≡O), 1635 (C=C), 1408, 1300, 1000, 987 and 928 (δ C=CH). Anal. Found: C, 63.25; H, 4.33; Cr, 14.49. $C_{19}H_{16}CrO_4$ calcd.: C, 63.33; H, 4.47; Cr, 14.43%.

Electroreduction of 3 in the presence of 4 (2 equiv) in DMF

The crude product (0.314 g) was separated by column chromatography with a 4/6 diethyl ether/hexane mixture as eluant. The compounds were isolated in the following order: fluorenone, **8b**, **3**.

[Spiro(tetrahydrofuran-2 : 9'-fluorene)]tricarbonylchromium, 8b. Bright yellow powder, m.p. 180°C (from Et_2O/hexane). ^1H NMR ($CDCl_3$, TMS), δ ppm 2.36 (mf, 4 H, CH_2), 4.34 (mf, 2 H, OCH_2), 5.15 and 5.45 (two t, *J* 6 Hz, 1 and 1 H, complexed aromatic H-2 and H-3), 5.75 (d, *J* 6 Hz, 2 H, complexed aromatic H-1 and H-2), 7.37 (mf, 4 H, aromatic H); MS *m/e* 359 (13), 358 (29, M^+), 357 (13), 303 (11), 302 (25, $M - C_3H_4O$), 301 (16), 275 (17), 274 (66, M – 3CO), 273 (20), 272 (10), 247 (28), 246 (100, 302 – 3CO), 245 (77), 244 (36), 243 (11); IR (KBr), ν cm^{-1} 3080, 2990, 2870, 1960 and 1875 (C≡O), 1420, 1045. Anal. Found: C, 63.44; H, 3.83; Cr, 14.40. $C_{19}H_{14}CrO_4$ calcd.: C, 63.69; H, 3.93; Cr, 14.51%.

Electroreduction of 3 in the presence of 5 in acetonitrile

The crude product (0.415 g) was separated by column chromatography using as eluant a 5/5 diethyl ether/hexane mixture. The compounds were isolated in the following order: **10b** [8], **11a**, **10a**, **12a**, **3**.

[9-(4'-chlorobutyroyloxy)fluorene]tricarbonylchromium (11a). Bright yellow powder, m.p. 129°C (from Et_2O/hexane). ^1H NMR ($CDCl_3$, TMS), δ ppm 2.22 (q, *J* 7 Hz, 2 H, CH_2–CH_2–CH_2), 2.74 (t, *J* 7 Hz, 2 H, $CH_2CH_2CH_2CO_2$), 3.66 (t, *J* 7 Hz, 2 H, $CH_2CH_2CH_2Cl$), 5.13 and 5.57 (two t, *J* 6 Hz, 1 and 1 H, complexed aromatic H-2 and H-3), 5.74 and 5.99 (two d, *J* 6 Hz, 1 and 1 H, complexed aromatic H-1 and H-4), 6.73 (s, 1 H, H-9), 7.44 (mf, 4 H, aromatic H); MS *m/e* 424 (3, M^+), 422 (9, M^+), 340 (10), 338 (27, M – 3CO), 302 (13, 340 and 338 – HCl), 254 (26, 340 – $(CH_2)_3CO_2$), 253 (23), 252, (87, 338 – $(CH_2)_3CO_2$), 217 (10, 254 and 252 – Cl), 165 (100, 254 and 252 – CrCl and/or 217 – Cr, $FlCH^+$); IR (KBr), ν cm^{-1} 1955 and 1880 (C≡O), 1732 (O–C=O), 1410, 1190, 1140.

[9-(4'-chlorobutyroyloxy)-9-(cyclopropylcarbonyl)fluorene]tricarbonylchromium (10a). Orange powder, m.p. 122°C (from Et_2O/hexane). ^1H NMR ($CDCl_3$, TMS), δ ppm 0.5–1.4 (mf, 5 H, cyclopropyl H), 2.19 (~ q, *J* 7 Hz, 2 H, $CH_2CH_2CH_2$), 2.75 (~ t, *J* 7 Hz, 2 H, $COCH_2$), 3.67 (~ t, *J* 7 Hz, 2 H, CH_2Cl), 5.14 (mf, 1 H, complexed aromatic H), 5.71 (mf, 2 H, complexed aromatic H), 6.40 (d, *J* 7 Hz, 1 H, complexed aromatic H), 7.51 (mf, 4 H, aromatic H); MS *m/e* 492 (M^+, 4), 490 (M^+, 14), 409 (8), 408 (41), 407 (32), 406 (82, M – 3CO), 405 (17), 370 (32, 408 and 406 – HCl), 323 (11), 322 (36), 321 (32), 320 (100, 406 – $(CH_2)_3CO_2$), 319 (15), 285 (19), 284 (24, 370 – $(CH_2)_3CO_2$), 279 (17), 254 (17), 253 (20), 252 (36), 251 (30), 231 (28), 216 (26), 215 (45), 205 (32), 203 (22), 189 (31), 165 (51, $FlCH^+$), 164 (24), 163 (16); IR (KBr), ν cm^{-1} 1978, 1960, 1927 and 1862 (C≡O), 1735 (O-C=O), 1700

(C=O), 1425, 1375, 1140. Anal. Found: C, 58.82; H, 3.76; Cl, 6.53. $C_{24}H_{19}O_6CrCl$ calcd.: C, 58.72; H, 3.90; Cl, 7.22%.

[9-(Cyanoethyl)-9-hydroxyfluorene]tricarbonylchromium (12a). Bright yellow powder, m.p. 118°C (from Et_2O/hexane). ^1H NMR ($CDCl_3$, TMS), 2.02 (s, 1 H, OH exchangeable with D_2O), 2.29 and 2.39 (mf, 4 H, CH_2CH_2CN), 5.20 and 5.64 (two t, $J \simeq 6$ Hz, 1 and 1 H, complexed aromatic H-1 and H-4), 5.78 and 5.97 (two d, $J \simeq 6$ Hz, 1 and 1 H, complexed aromatic H-2 and H-3), 7.46 ($\simeq$ s, 4 H, aromatic H); MS *m/e* 371 (22, M^+), 288 (19), 287 (89, $M - 3CO$), 286 (12), 248 (30), 247 (100, $287 - CH_2CN$), 246 (39), 245 (14); IR (KBr), ν cm^{-1} 3460 (OH), 2250 (C≡N), 1965, 1902 and 1860 (C≡O), 1420, 1075.

Electroreduction of 3 (0.5 mmol) in the presence of 6 in DMF

The crude product (0.166 g) was separated by column chromatography using as eluant a 5/5 methylene chloride/hexane mixture. The compounds were isolated in the following order: (fluorenol)$Cr(CO)_3$ [14], **12a**.

(Fluorenol)tricarbonylchromium. Orange powder, m.p. 142°C (from Et_2O/hexane) (lit. [14] an oil). ^1H NMR ($CDCl_3$, TMS), δ ppm 1.62 and 1.74 (two s, 1 H, two diastereoisomeric OH, exchangeable with D_2O), 5.20 and 5.76 (two t, J 6 Hz, 1 and 1 H, complexed aromatic H-2 and H-3), 5.73 and 6.05 (two d, J 6 Hz, 1 and 1 H, complexed aromatic H-4 and H-1, respectively), 5.58 and 5.62 (two s, 1 H, two distereoisomeric H-9), 7.44 (mf, 4 H, aromatic H); MS *m/e* 318 (13, M^+), 262 (17, $M - 2CO$), 235 (18), 234 (78, $M - 3CO$), 233 (36), 232 (10), 166 (21), 165 (100, $FlCH^+$), 164 (53), 163 (21); IR (KBr), ν cm^{-1} 3530 and 3430 (OH), 3075, 1965, 1945 and 1870 (C≡O), 1370, 1180, 1025. Anal. Found: C, 60.28; H, 3.29; Cr, 16.52. $C_{16}H_{10}CrO_4$ calcd.: C, 60.38; H, 3.17; Cr, 16.34%.

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