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Synthesis of cationic π-complexes of transition metals of groups VI–VIII by exchange of the carbonyl ligands in strong protonic acids

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Abstract

The review of the works of the author and coworkers reveals that metal carbonyl complexes may be successfully used as starting compounds in the synthesis of cationic π-complexes of transition metals of groups VI–VIII. The carbonyl ligands may be substituted be allyl, diene, propargyl, ethylene and arene ligands with the oxidation state of the metals being either increased or retained depending on the electronic requirements of the metal. Numerous examples demonstrate efficiency of this one-step synthesis making use of the combined action of strong protonic acid and UV irradiation.

Introduction

Since the discovery of $Ni(CO)_4$ by L. Mond [1], one hundred years ago, the properties of metal carbonyl complexes have been studied extensively. Many important applications of these compounds have been found. As far as synthetic organometallic chemistry is concerned, their ability to exchange carbonyl groups for other ligands proved of fundamental importance. The present paper summarizes our results concerning a simple synthesis of cationic complexes of the transition metals by substitution of organic ligands for the carbonyl ligands in the π-complexes of transition metals of the groups VI–VIII in strong protonic acid medium. At the same time it is noteworthy that the method acquires more general importance. Our works [2,3] as well as the works of other authors [4] have revealed that in addition to carbonyl ligands other ligands are also capable of substitution by π-ligands in the presence of strong protonic acids. Cationic π-complexes of transition metals are of significant interest as reagents and catalysts in organic and organometallic synthesis [5–8]. Development of a simple synthetic route starting from easily accessible metal carbonyls therefore was of special interest.

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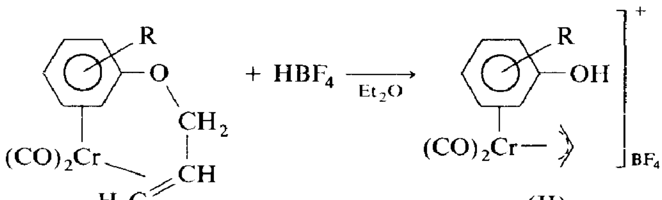
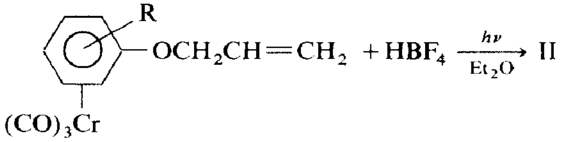
Synthesis of cationic allyl complexes

This work dates back to our study of the behaviour of chelate areneolefinechromiumdicarbonyl complexes (I) in ether in the presence of 48% HBF_4 [9]. Under the conditions mentioned above these compounds readily undergo transformation into the previously unknown cationic areneallyldicarbonylchromium complexes (II) in yields of 60–90%, while their precursor non-chelate complexes (III) undergo transformation into II in the presence of HBF_4 only under accompanying UV irradiation:

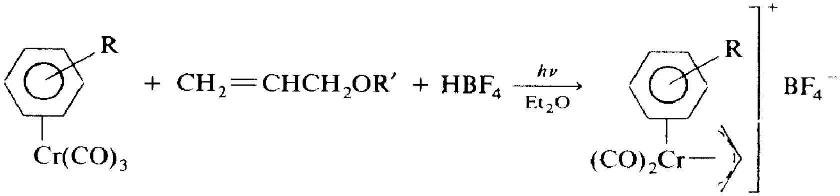


(II)



(III)

This example shows that protonic acid and UV irradiation can be successfully combined. We then made a successful attempt to obtain areneallyldicarbonylchromium complexes (IV, analogous to II), in one step from the mixture of arenetricarbonylchromium complexes and allyl alcohol or its derivatives in ether in the presence of HBF_4 under UV irradiation. Complexes IV were obtained with good yields [10].



(IV)

(R = H, alkyl; R′ = H, Me)

This reaction increases the metal oxidation state. Thus we suggested a first method for the oxidative substitution of the carbonyl ligand by the π-ligand under the action of protonic acid accompanied by UV irradiation. We have widely used this technique in our subsequent work. Already after our publication some new data appeared concerning synthesis of allyl complexes from the corresponding carbonyls in two steps [11,12]. For example, the unstable η^2-olefinic complexes were initialy obtained from allyl alcohol and were subsequently treated with acid in the presence of carbon monoxide [11]. This technique complicated the synthesis and lowered the yield. We have shown later that the two-step synthesis may be of use in some special cases which will be discussed below.

Strong protonic acids are frequently used for synthesis of cationic species in organic and inorganic chemistry and therefore our results did not seem surprising at all. However successive application of this method in case of metal carbonyls which tend to decompose after protonation [13], seemed rather unexpected. We have demonstrated that our method may be universally applied to the synthesis of cationic complexes with allyl, diene, propargyl, ethylene and arene ligands with protonic acids playing simultaneously roles of reagent and solvent.

Iron, molybdenum and tungsten carbonyls turned out to react with allyl alcohol in the presence of $HBF_4 \cdot Et_2O$ under UV irradiation with formation of the corresponding cationic allyl complexes [14].

$$M(CO)_n + CH_2{=}CHCH_2OH + HBF_4 \cdot Et_2O \xrightarrow[S]{h\nu} \left[M(CO)_{n-1}(\eta^3\text{-}C_3H_5)\right]^+ BF_4^-$$

$(M = Fe,\ n = 5,\ S = Et_2O;$
$M = Mo,\ n = 6,\ S = C_6H_6;$
$M = W,\ n = 6,\ S = Et_2O)$

The iron and tungsten complexes are readily formed in ether while the less basic solvent benzene is required for the formation of the less stable molybdenum complex. The corresponding chromium complexes could not be obtained even in benzene, which may be explained by lowering the basicity of the initial carbonyls [15] and the stability of the series W > Mo > Cr. Stability of the Mo and Cr containing cations may be improved if the CO ligands of the initial carbonyls are substituted by $P(OMe)_3$.

We have shown that the cationic allyl complex $[Fe(CO)_4(\eta^3\text{-}C_3H_5)]^+ BF_4^-$ may be obtained in 92% yield from $Fe_2(CO)_9$ without UV irradiation * [14].

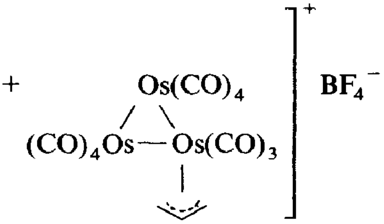
Mononuclear cationic allyl complexes have been also obtained from all the trinuclear carbonyls of the metals of the iron subgroup. Only in the case of $Os_3(CO)_{12}$ is the trinuclear allyl containing cation V formed together with the mononuclear complex. However the resulting mixture was difficult to separate [16].

$$M_3(CO)_{12} + CH_2{=}CHCH_2OH \xrightarrow[Et_2O]{HBF_4} \left[(CO)_4M(\eta^3\text{-}C_3H_5)\right]^+ BF_4^-$$

$(M = Fe, Ru)$

$$Os_3(CO)_{12} + CH_2{=}CHCH_2OH \xrightarrow[Et_2O]{HBF_4}$$

$$\left[(CO)_4Os(\eta^3\text{-}C_3H_5)\right]^+ BF_4^- +$$



(V)

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* It is noteworthy that $[(CO)_4Fe(\eta^3\text{-}C_3H_5)]^+ BF_4^-$ was obtained by Nicholas and coworkers [11] in a two-step synthesis.

In order to obtain exclusively the trinuclear cation V the two-step method was used [17] since the ease of metal–metal bond cleavage in the clusters limits application of the one-step technique. The structure of the cationic cluster V has been confirmed by means of X-ray structural analysis [17].

In order to obtain substituted allyl complexes by the one-step technique we have used the conjugated dienes. However under the conditions described for allyl alcohol, irradiation of the mixture of $Fe(CO)_5$ with butadiene in the presence of $HBF_4 \cdot Et_2O$, gives the neutral butadienetricarbonyliron complex instead of the cationic crotyl complex VI [18].

$$\mathrm{Fe(CO)_5 + C_4H_6 + HBF_4 \cdot Et_2O} \xrightarrow{h\nu}$$

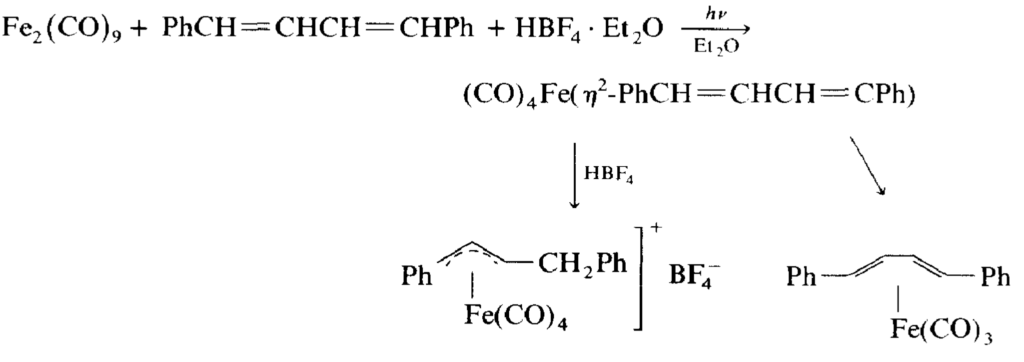
$$\begin{tikzcd}
& \mathrm{(CO)_4Fe(\eta^2\text{-}C_4H_6)} \arrow[dl] \arrow[dr, "\times\ \mathrm{HBF_4}"] & \\
\mathrm{(CO)_3Fe(\eta^4\text{-}C_4H_6)} & & \mathrm{[(CO)_4Fe(\eta^3\text{-}C_4H_7)]^+\ BF_4^-} \\
& & \mathrm{(VI)}
\end{tikzcd}$$

The η^2-$C_4H_6Fe(CO)_4$ formed on the first step of the reaction is likely to undergo transformation into an tricarbonyliron complex more quickly than protonation with formation of the cationic complex VI. Presumably the acidity of the medium is not enough for the protonation of the η^4-complex. Use of $Fe_2(CO)_9$ in this reaction without irradiation gives VI in 18% yield. According to the IR spectra the solution contains a certain amount of the neutral η^2-complex. Evaporation of the solvent results in increase of the yield of VI up to 78%. The analogous situation is observed in the case of reaction of $W(CO)_6$ with butadiene: the neutral η^2-butadiene complex VII is also formed on the first step and only after evaporation of the solvent the cationic complex VIII is obtained.

$$\mathrm{W(CO)_6 + C_4H_6 + HBF_4 \cdot Et_2O} \xrightarrow[\mathrm{Et_2O}]{}$$

$$\underset{\mathrm{(VII)}}{\mathrm{(CO)_5W(\eta^2\text{-}C_4H_6)}} \xrightarrow{\mathrm{HBF_4}} \underset{\mathrm{(VIII)}}{\mathrm{\left[(CO)_4W(\eta^3\text{-}C_4H_7)\right]^+\ BF_4^-}}$$

The aryl substituents naturally lower the yield of the cationic complex IX down to 2%. The η^4-diene complex X is the main reaction product.



(IX) (X)

When the reaction is carried out in benzene, the yield of IX is increased to 28%. The corresponding tungsten complex cannot be formed even under these conditions [18].

It is quite natural that the half-sandwich metal carbonyls form allyl or substituted allyl complexes more readily than the common metal carbonyls. Along with arenetricarbonylchromium complexes discussed above, arenetricarbonylmolybdenum complexes as well as cyclopentadienyltricarbonyl-manganese and -rhenium complexes were used in these reactions. All of these compounds form quite stable cationic complexes with allyl alcohols and dienes even in the presence of aqueous 48% HBF_4 [19,20].

$$\mathrm{LM(CO)_3 + CH_2{=}CHCH_2OH + HBF_4} \xrightarrow[\mathrm{Et_2O}]{h\nu} [\mathrm{LM(CO)_2(\eta^3\text{-allylR}''')}]^+\ \mathrm{BF_4}^-$$
$$(\text{or } \mathrm{CH_2{=}CHCR'{=}CH_2R''})$$

(M = Cr, Mo; L = arene; R = H, Me; M = Mn, Re; L = Cp; R′ = H, Me)

Moreover the cyclopentadienyldicarbonyl compounds of the cobalt subgroup $CpM(CO)_2$ (M = Co, Rh) do not require irradiation [21]. In the case of formation of cationic half-sandwich complexes with the substituted allyl ligand all three types of isomerism are observed; *syn*-, *anti*-isomerism, isomerism connected with structural isomerism of the substituted allyl ligand and conformational *endo*–*exo* isomerism. For a series of examples these types of isomerism were observed with NMR and IR spectroscopy [22]. Formation of a particular isomer to a certain extent depends on the nature of the initial complex and the diene used [22].

Synthesis of the cationic butadiene complexes

Reactions with the conjugated dienes illustrate the flexibility of the π-electronic system of the ligand, which readily meets the electronic requirements of the metal forming the complex. Under the conditions discussed above the dienes give cationic allyl complexes. However if under the same conditions binuclear carbonyl complexes $Co_2(CO)_8$ and $[CpMo(CO)_3]_2$ are used, the cationic η^4-diene complexes are formed [23]. Reaction of $Co_2(CO)_8$ with excess butadiene does not stop at the monobutadiene complex which has been obtained earlier by Pauson and coworkers [24] but results in a mixture of mono- and bis-butadiene compounds. The latter was hitherto unknown but was isolated as an individual complex by treatment of the solution of the reaction mixture in nitromethane by butadiene [23]:

$$\mathrm{Co_2(CO)_8 + C_4H_6 + HBF_4\cdot Et_2O} \xrightarrow[\mathrm{Et_2O}]{h\nu}$$
$$[(\eta^4\text{-}\mathrm{C_4H_6})\mathrm{Co(CO)_3}]^+\ \mathrm{BF_4}^- + [(\eta^4\text{-}\mathrm{C_4H_6})_2\mathrm{Co(CO)}]^+\ \mathrm{BF_4}^- \xrightarrow[\mathrm{CH_3NO_2}]{\mathrm{C_4H_6}}$$
$$[(\eta^4\text{-}\mathrm{C_4H_6})_2\mathrm{Co(CO)}]^+\ \mathrm{BF_4}^-$$

The complex $[CpMo(CO)_3]_2$ behaves in this reaction in an analogous way [25]:

$$[\mathrm{C_5H_5Mo(CO)_3}]_2 + \text{diene} + \mathrm{HBF_4\cdot Et_2O} \longrightarrow$$
$$[\mathrm{C_5H_5Mo(CO)_2}(\eta^4\text{-diene})]^+\ \mathrm{BF_4}^-$$

(diene = butadiene, isoprene)

It is noteworthy that complexes of this type were synthesized earlier using silver salts and $CpMo(CO)_3Hal$ [26].

Synthesis of the cationic complexes in strong protonic acid may be performed by exchange of other ligands as well. Thus by treatment of the neutral allyl complex in the presence of $HBF_4 \cdot Et_2O$ we succeeded in the synthesis of the diene complexes of molybdenum [27]. Application of 48% HBF_4 requires accompanying UV irradiation. The oxidation state of the metal is retained:

$$C_5H_5Mo(CO)_2(\eta^3\text{-}C_3H_5) + \text{diene} + HBF_4 \cdot Et_2O \longrightarrow [C_5H_5Mo(CO)_2(\eta^4\text{-diene})]^+ \; BF_4^-$$

(diene = butadiene, isoprene, cyclohexadiene, cyclooctadiene)

Cationic cobalt complexes with phosphate ligands were obtained similarly [27]:

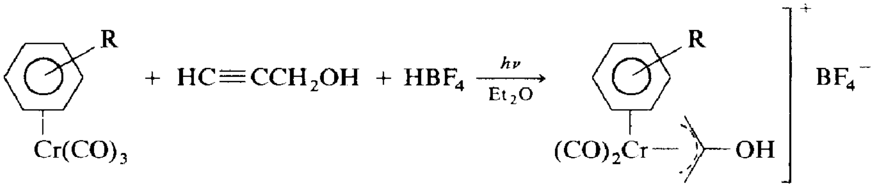
$$(\eta^3\text{-}C_4H_7)Co(CO)_2P(OMe)_3 + \text{diene} + HBF_4 \cdot Et_2O \longrightarrow [(\eta^4\text{-diene})Co(CO)_2P(OMe)_3]^+ \; BF_4^-$$

(diene = isoprene, cyclohexadiene)

It is noteworthy that the complex allyltricarbonylcobalt does not participate in this reaction.

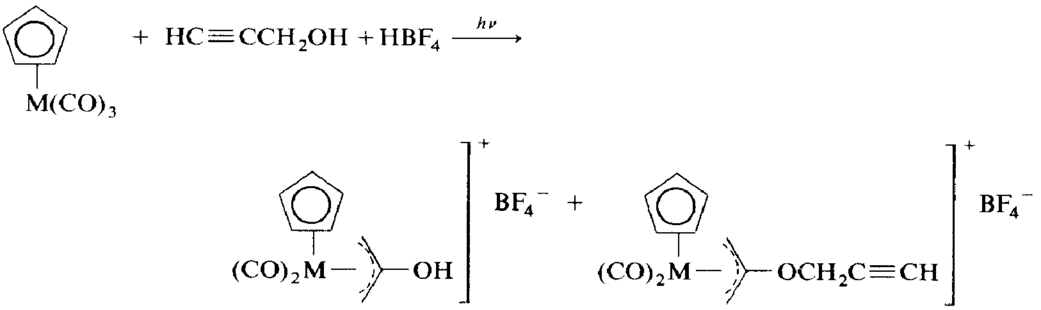
Synthesis of cationic complexes with the propargyl ligand and the products of their transformations

Reactions of propargyl alcohol with carbonyls gives completely unexpected results. UV irradiation of arenetricabonylchromium complexes with propargyl alcohol in the presence of HBF_4 results in arene-2-hydroxyallyldicarbonylchromium cations [9]:



($R_6 = H_6$; 1,3,5-Me_3, H_3; Me_6)

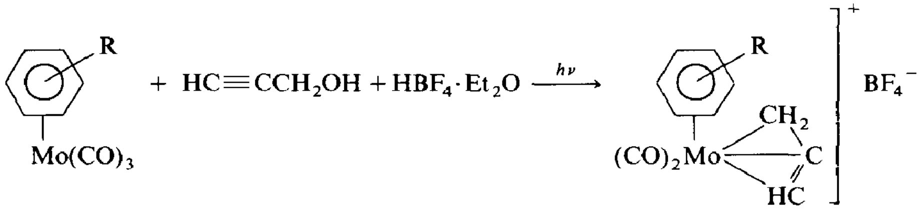
Analogous results have been obtained also with cyclopentadienyltricarbonylmanganese and -rhenium complexes where both 2-hydroxyallyl derivatives and complexes with 2-propargyloxyallyl ligands have also been isolated:



(M = Mn, Re)

It is noteworthy that the yields of the latter reaction are low, but can be improved considerably if a two-step method is applied, i.e. if a π-acetylene complex with propargyl alcohol $CpM(CO)_2(\pi\text{-}H\text{-}C{\equiv}CCH_2OH)$ is obtained in the first step which is then treated with HBF_4.

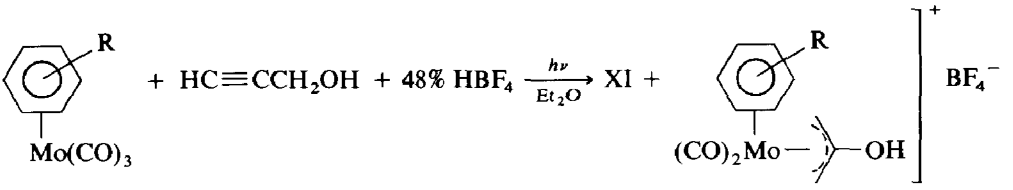
Use of arenetricarbonylmolybdenum complexes for reaction with propargyl alcohol in the presence of $HBF_4 \cdot Et_2O$ results in formation of stable cationic complexes (XI) with propargyl ligands in high yields (80–90%) [28].



(XI)

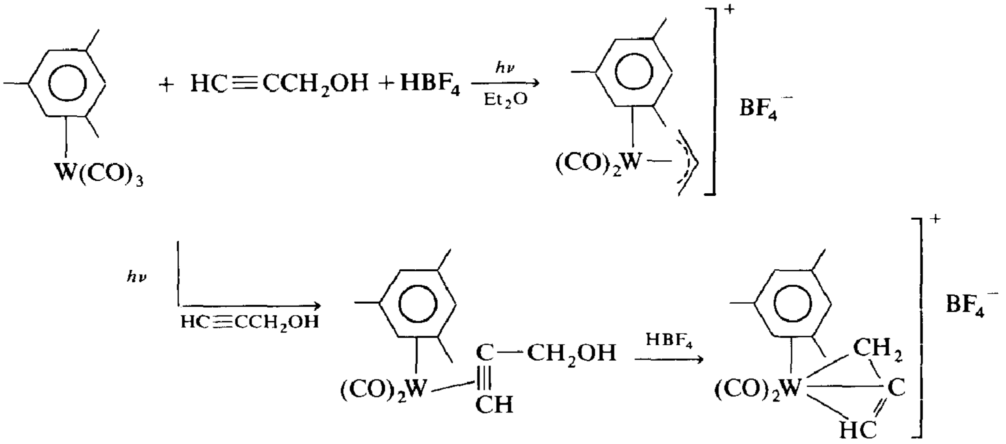
$(R_6 = 1,3,5\text{-}Me_3, H_3; Me_6)$

However in 48% HBF_4 complex XI has been obtained in a mixture with a cationic complex with the 2-hydroxyallyl ligand:



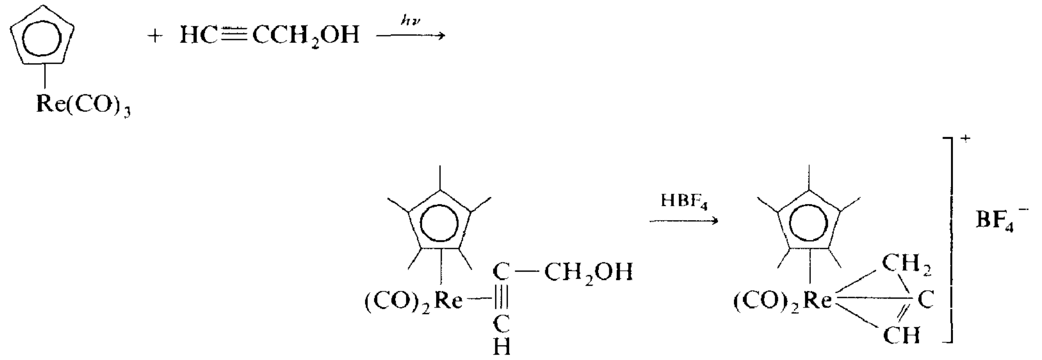
Mononuclear cationic complexes with propargyl ligands have been obtained for the first time. Complexes with hexamethylbenzene ligands are the most stable.

In the case of the tungsten compound the direct reaction has unexpectedly led to a cationic complex with an unsubstituted allyl ligand (33% yield), which has been earlier obtained using allyl alcohol [28]:

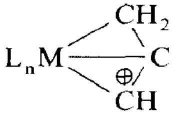


A propargyl complex has been synthesized in only two steps with isolation of the π-acetylene complex as an intermediate and its subsequent treatment with acid.

Pentamethylcyclopentadienylrhenium also gives a stable cationic complex with the propargyl ligand if the reaction is carried out in two steps:



These preliminary results [28] suggest that in all one step syntheses using propargyl alcohol, the products are containing propargyl ligands. Their thermodynamic and kinetic stability depends on the nature of the metal atom and the number of methyl groups in the aromatic ligands. The kinetically less stable chromium complexes readily add water to the propargyl ligand. The OH group attack occurs at the carbon atom * carrying a certain positive charge as a carbon atom of the three-membered metallocyclopropenylium ring which may be depicted as follows:



Manganese and rhenium complexes with non-methylated cyclopentadienyl ligands behave in the same way while the ring permethylated propargylrhenium derivatives are quite stable and may be isolated in a pure state. At the same time, in the one-step synthesis under irradiation in the presence of HBF_4 the starting tungsten compound probably forms alongside with the cationic complex hydride tungsten compounds which reduce the propargyl ligand in the cationic complex to allyl.

Our investigations of the structure and reactivity of the cationic propargyl complexes are in progress. The stabilization mechanism of the α-carbocationic centre in these compounds is likely to be analogous to that for α-carbocationic centres of metallocenes. Our review of permethylated metallocenylcarbocations of the iron subgroup reveals that the distance from the α-carbocationic centre to the metal atom decreases from the upper to the lower elements of the group i.e. stabilization of the carbocationic centre is enhanced [30]. The same observation is likely to be true for complex propargyl cations.

Synthesis of the cationic complexes with ethylene

Cationic complexes of ethylene were synthesized from the binuclear compounds $[CpFe(CO)_2]_2$, $Mn_2(CO)_{10}$ or $Re_2(CO)_{10}$. Although the yields are rather low (20–25%), reaction occurs at an ethylene pressure of 1 atm, using accesible metal carbonyls [25].

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* The preliminary results showed that in the presence of PPh_3 a mixture of three products is formed as a result of Ph_3P-attack on all the three carbon atoms of propargyl ligand. The first result was published in ref. 29.

$$[CpFe(CO)_2]_2 + C_2H_4 + HBF_4 \cdot Et_2O \longrightarrow [CpFe(CO)_2(\eta^2\text{-}C_2H_4)]^+ \; BF_4^-$$

$$M_2(CO)_{10} + C_2H_4 + HBF_4 \cdot Et_2O \longrightarrow [M(CO)_5(\eta^2\text{-}C_2H_4)]^+ \; BF_4^-$$

$$(M = Mn, Re)$$

The $[CpFe(CO)_2C_2H_4]^+$ cation has been synthesized in acidic medium using quinone or gaseous oxygen as oxidizing agents [31–33]. We have shown that supplementary oxidation in this case is unnecessary, as protonic acid successfully plays this role [25].

Synthesis of cationic arene complexes

Only few examples of interaction of metal carbonyls with arenes leading to cationic arene complexes with increased oxidation state of the metal atom are known. Vanadium complexes of this type have been synthesized in reactions of $V(CO)_6$ with arenes in heptane [34,35]. There are a few patents published, concerning synthesis of the cationic arene complexes of iron, ruthenium, osmium [36], cobalt and nickel [37] in the presence of $AlCl_3$.

We have reported that arene complexes may be obtained from some metal carbonyls using CF_3COOH as a protonic aicd. Application of this acid instead of HBF_4 is convenient as it acts not only as a protonating and oxidizing agent, but also as a solvent. The UV irradiation in these cases is unnecessary. Thus refluxing $Mn_2(CO)_{10}$ with mesitylene in CF_3COOH gives a cationic complex $[Mn(1,3,5\text{-}C_6H_3Me_3)(CO)_3]^+$ isolated as its tetrafluoroborate in 62% yield. The same complex as well as complexes with other arene ligands have been obtained using a mixed halogenocarbonylmanganese complex [38].

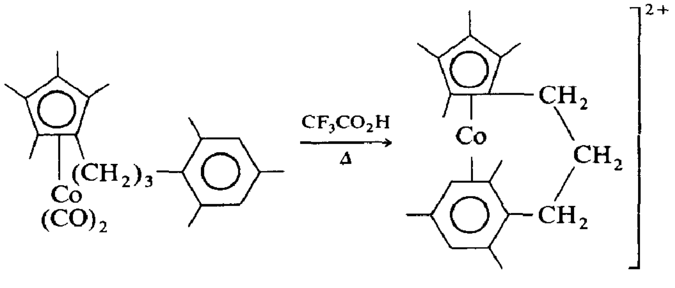
$$Mn(CO)_5X \xrightarrow{\text{arene}} [Mn(\eta\text{-arene})(CO)_3]^+ \; X^-$$

$$(X = Cl, Br;\ \text{arene} = C_6H_6;\ MeC_6H_5;\ 1,3,5\text{-}Me_3C_6H_3;$$

$$Me_6C_6;\ EtOC_6H_5;\ 2,4,6\text{-}Me_3C_6H_2(CH_2)_3COOH)$$

In the case of mesitylene and hexamethylbenzene the yields are 70–80% and the reaction proceeds much faster than in the case of $Mn_2(CO)_{10}$ as the starting material. Rhenium carbonyl halides were also successfully used for synthesis of $[Re(\eta\text{-Arene})(CO)_3]^+ \; X^-$. This method requires no treatment with water, which is extremely important in view of the high sensitivity of the rhenium cations towards water.

Application of $Me_5C_5Co(CO)_2$ with arenes in CF_3COOH proved convenient for synthesis of cationic complexes of cobalt of the $[Me_5C_5CoArene]^{2+} \; X_2^-$ type. A previously unknown dicationic complex has been synthesized by this method [39]:



However for the synthesis of the cationic arene complexes we have more frequently used exchange of halogen ligands in transition metal complexes in CF_3COOH [2,3]. Thus refluxing of $[(\eta\text{-}C_6H_6)RuCl_2]_2$ with arenes in CF_3COOH readily yields mixed bis-arene cations [40] which have been synthesized by other workers with by using silver salts [41,42]:

$$\begin{tikzcd}
{[(\eta\text{-}C_6H_6)RuCl_2]_2} \arrow[r, "\text{arene}", "CF_3COOH"'] \arrow[dr, "AgBF_4", "Me_2CO"'] & {[Ru(\eta\text{-}C_6H_6)(\eta\text{-arene})]^{2+}} \\
 & {[Ru(\eta\text{-}C_6H_6)(Me_2CO)_3]^{2+}} \arrow[u, "\text{arene, H}^+"']
\end{tikzcd}$$

Thus strong protonic acids proved to be highly efficient reagents and ideal media for synthesis of cationic π-complexes of transition metals by means of ligand exchange. This quite productive method has allowed us to synthesize a series of previously unknown complexes from readily available metal carbonyls and their derivatives.

Acknowledgment

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