The reactivity of cyclopentadienyl dicarbonyl complexes of iron, η^5-$C_5H_5(CO)_2Fe$-η^1-R (R = alkyl or aryl). Synthesis of novel derivatives substituted in the cyclopentadienyl ring

T. Yu. Orlova★ *and V. N. Setkina*

A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences,
28 ul. Vavilova, 117813 Moscow, Russian Federation.
Fax: +7 (095) 135 5085

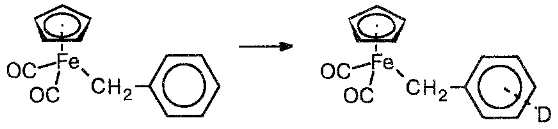
The results of the study of the reactivity of the cyclopentadienyl ligand in $Cp(CO)_2Fe$-η^1-R (R = Alk or Ar) iron complexes have been summarized. The methods for preparation of mono-, bi-, and trinuclear homo- and heterometallic complexes containing a $Cp(CO)_2Fe$ moiety are presented.

Key words: cyclopentadienyl ligand, metallation, polynuclear complexes, synthesis.

Cyclopentadienyl carbonyl complexes of iron, $Cp(CO)_2Fe$-η^1-R (R = Alk, Ar), were synthesized by Piper and Wilkinson more than 35 years ago.[1] These complexes simulate intermediate compounds in metallocomplex catalysis and are among the organometallic systems that are being studied the most intensively.[2] The attention of most researchers has been concentrated on investigating the nature and properties of the iron—carbon σ-bond. However, data on the reactivity of the cyclopentadienyl ligands in these complexes were missing from the literature until the beginning of the 1980 s. At the same time, it is evident that investigation of the chemistry of the cyclopentadienyl ligand in the complexes under consideration is important both for the development of the theoretical concepts of organometallic chemistry and from the practical standpoint. For example, incorporation of these complexes into polymeric chains *via* the cyclopentadienyl ligand is known to impart special conducting and catalytic properties to the polymers.[3]

The isotope exchange of hydrogen is a model reaction for studying the reactivity of compounds in acidic and basic media. In the beginning of the 1960 s, this reaction was used to estimate the reactivity of a variety of complexes of Group VI—VIII transition metals.[4] It was shown, in particular, that ferrocene and its homologs readily undergo hydrogen exchange with acids enriched in deuterium.[5,6] The study of the behavior of $Cp(CO)_2Fe$-η^1-R under the conditions of hydrogen isotope exchange carried out by us showed that in an acidic medium (CF_3COOD/CH_2Cl_2), *i.e.*, under conditions simulating electrophilic substitution reactions, the iron—carbon σ-bond usually cleaves. The $Cp(CO)_2Fe$-η^1-CH_2Ph complex containing an η^1-benzyl ligand, which is relatively stable in an acidic medium, is an exception. It turned out that the exchange involves only hydrogen atoms of the phenyl ring, while the cyclopentadienyl ring remains intact[7] (Scheme 1).

Scheme 1



Reagents and conditions: CH_3COOD/CF_3COOD, CH_2Cl_2, 20 °C

Study of the rates of the hydrogen exchange showed that the $Cp(CO)_2Fe$-η^1-CH_2 group as a substituent in the phenyl ring possesses strong electron-donating properties comparable with those of a methoxy group.

Isoelectronic complexes of tungsten and molybdenum, $Cp(CO)_3M$-η^1-CH_2Ph (M = W or Mo), behave in a similar way.[8] The strong electron-donating character of the $Cp(CO)_nM$-η^1-CH_2 moiety has also been identified in a number of papers on the basis of the determination of the deuterium content in the deuterotoluene formed during cleavage of the Fe—CH_2Ph σ-bond by deuterotrifluoroacetic acid,[9] measurement of the acidic dissociation constant of $Cp(CO)_nM$-η^1-CH_2COOH,[10] pK_a of the conjugated acids of the corresponding pyridine bases, $Cp(CO)_nM$-η^1-$CH_2C_5H_5N$,[11] and on the basis of variations of the ^{19}F chemical shifts of $Cp(CO)_nM$-η^1-$CH_2C_6H_4F$ *p*-substituted derivatives with

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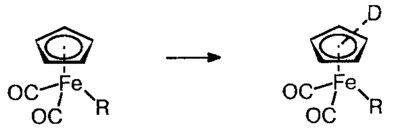
respect to fluorobenzene.[12] From all of these data, it follows that under the conditions of electrophilic substitution, the phenyl ring should react first, and subsequent investigations have confirmed this inference.

By using the optically active $Cp(CO)_2Fe$-η^1-$^*CH(CH_3)Ph$ complex to investigate the mechanism of acidic exchange in the phenyl ring of $Cp(CO)_2Fe$-η^1-CH_2Ph and also by measuring the kinetic isotope effect, we have shown that the reaction occurs without cleavage of the Fe—CH_2Ph σ-bond, as electrophilic aromatic substitution with the peculiarity that the intermediate carbenium ion is stabilized by the conjugation of the Fe—CH_2 σ-bond with the π-electron system of the benzene ring.[13,14]

Acylation of $Cp(CO)_nM$-η^1-CH_2Ph (M = Fe, $n = 2$; M = Mo or W, $n = 3$) according to Friedel—Crafts also involves the phenyl ring only.[15,16]

Although the complexes under study are unstable in acids, they turned out to be rather stable in a basic medium, under the conditions of protophilic substitution, and only the hydrogen atoms of the cyclopentadienyl ligand can undergo exchange reactions.[17] Measurement of the rate of hydrogen exchange showed that the acidity of the C—H bonds of the cyclopentadienyl ligands in these complexes is relatively high (pK_a 29—30) and is comparable to the C—H acidity of the methylene hydrogen atoms in Ph_2CH_2 or the methine hydrogen atom in Ph_3CH (Scheme 2).

Scheme 2



Reagents and conditions: EtONa/EtOD, C_6D_6, 100 °C

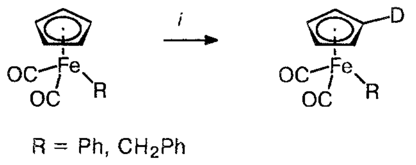
Thus, the study of the reactivity of the $Cp(CO)_2Fe$-η^1-R complexes with the aid of hydrogen exchange showed that protophilic exchange reactions are typical of the cyclopentadienyl ligands in these systems.

Reasoning from this conclusion, we have studied the behavior of the complexes in question under conditions of metallation and found that the reaction with *n*-butyllithium occurs under mild conditions (THF, —78 °C), involves the Cp ring only, and after treatment with D_2O affords monodeutero derivatives in high yields[18] (Scheme 3).

On the basis of this result, we used the metallation reaction discovered for introducing various substituents, including those with functional groups, into the cyclopentadienyl ligand,[18—21] and have prepared by this method the previously unknown $XC_5H_4(CO)_2FeR$ derivatives (Scheme 4).

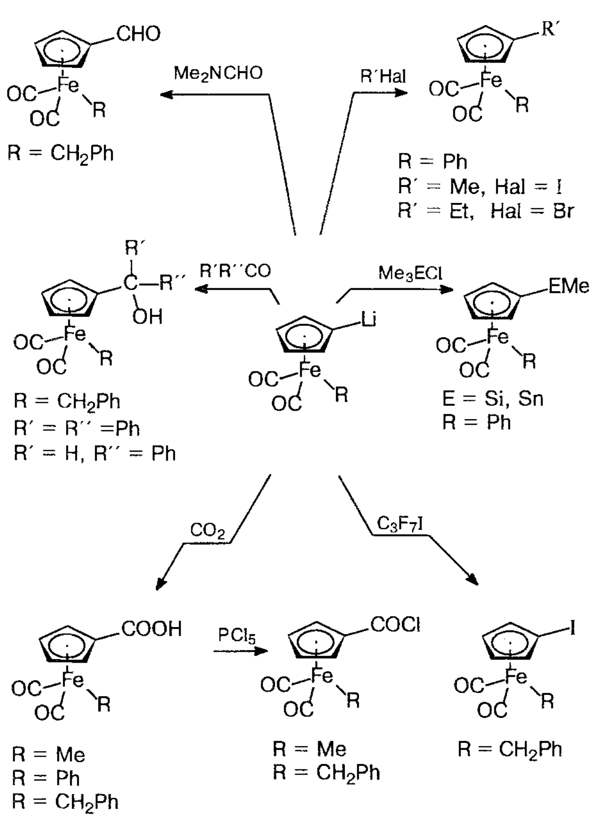
Stille *et al.* managed to introduce a PPh_2 group into the cyclopentadienyl ring by metallation of $Cp(CO)_nMMe$ (M= Fe, $n = 2$; M = W, $n = 3$) with *sec*-bytullithium followed by treatment with Ph_2PCl.[22]

Scheme 3



i. 1. *n*-BuLi; 2. D_2O

Scheme 4



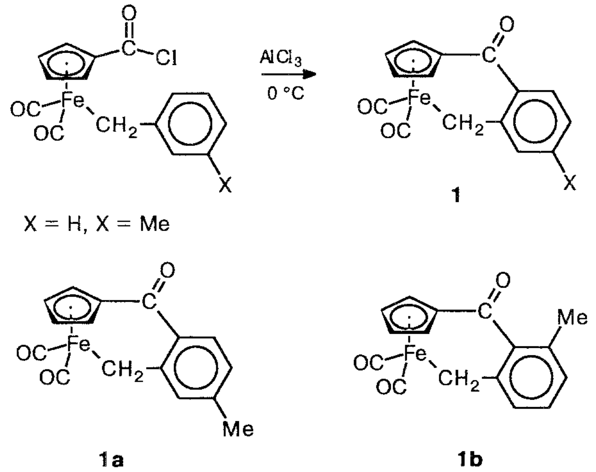
The metallation of the cyclopentadienyl ring in the $Cp(CO)_2Fe$-η^1-R complexes under study occurs with a high yield; thus, it is a convenient preparative-scale method for synthesizing novel derivatives with substituents in the cyclopentadienyl ring.

Attempts to metallate the Cp ligand in complexes in which one of the CO groups is replaced by the electron-donating PPh_3 ligand, $Cp(CO)(PPh_3)Fe$-η^1-R, were unsuccessful, even though the conditions of the reaction were varied.[23]

The range of complexes of this type may be extended by further transformations of the functional substituents in the cyclopentadienyl ligand. In fact, in the series of $XC_5H_4(CO)_3MMe$ (M = Cr, Mo, or W) isoelectronic complexes, conversion of aldehydes or ketones into olefins and carbinols by Wittig and Grignard reactions, respectively, hydrolysis of esters in a KOH/MeOH mixture to give acids, and conversion of acids into aroyl chlorides under the action of PCl_5 have been reported.[24]

By employing the above-noted activation of the phenyl ring with respect to electrophilic substitution by the $Cp(CO)_2Fe$-η^1-CH_2 group, we managed to carry out intramolecular acylation of the phenyl group in the $C_5H_4COCl(CO)_2Fe$-η^1-CH_2Ph complex according to the Friedel—Crafts reaction and obtained the metallacyclic ketone **1**.[25] In the case of the complex with the η^1-*m*-methylbenzyl ligand, the reaction carried out under the same conditions gave two isomeric metallacyclic ketones with the less sterically hindered ketone **1a** slightly predominating (Scheme 5).

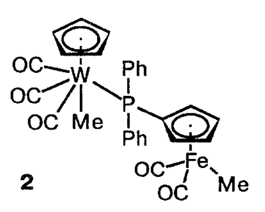
Scheme 5



The metallation of the cyclopentadienyl ligand in the complexes under consideration has been the key step in the preparation of homo- and heterometallic bi- and trinuclear complexes with bridging cyclopentadienyl ligands. The interest in these complexes is due to the fact that they can be used as model compounds for investigating metal—metal interaction, which determines unusual conducting and catalytic properties of polynuclear systems.[26]

The bond of the bridging ligand with metals may vary in nature: coordination, σ-, or π-bond. The geometry of these complexes also varies.

Stille *et al.* used metallation of the cyclopentadienyl ligand for preparing heterometallic binuclear complex **2** with the bridging η^1,η^5-$PPh_2C_5H_4$ ligand bound with the metal atoms by coordination and π-bonds.[22]



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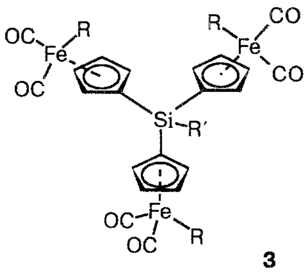
Wright *et al.* prepared the propeller-shaped trinuclear complexes **3** by treating the lithium derivatives, η^5-$C_5H_4LiM(CO)_nR$ (M = Fe, Mn, or W; R = Me, Et, or CH_2Ph), with trichlorosilanes $R'SiCl_3$ (R = Me, $(CH_2)_3Cl$, or *m,p*-$(CH_2)_2C_6H_4CH_2Cl$).

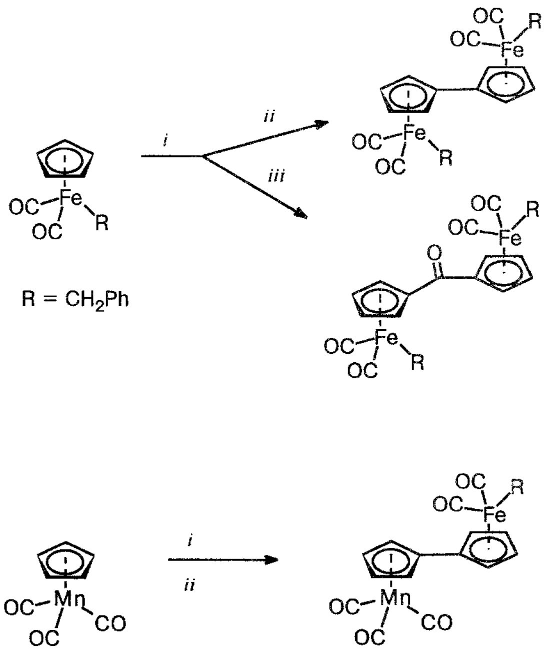


3

We used metallocomplexes containing a halogen atom in the Cp ring or at the metal atom as electrophilic reagents and thus prepared bi-, tri-, and tetranuclear systems with bridging cyclopentadienyl ligands bound with transition metals by π,π- or σ,π-bonds, respectively.

For example, by the reactions of lithium derivatives, η^5-$C_5H_4Li(CO)_2Fe$-η^1-CH_2Ph or η^5-$C_5H_4LiMn(CO)_3$, with η^5-$C_5H_4I(CO)_2Fe$-η^1-CH_2Ph or η^5-$C_5H_4COCl(CO)_2Fe$-η^1-CH_2Ph, homo- and heterometallic binuclear complexes in which the two cyclopentadienyl rings are bound directly or through a C=O group have been prepared (Scheme 6).[27]

Scheme 6

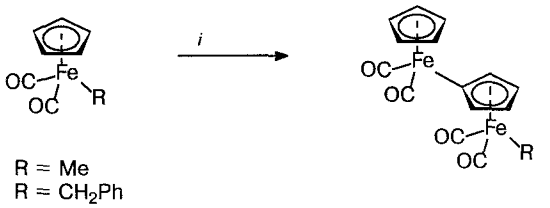


Reagents and conditions: *i*. *n*-BuLi, *ii*. $IC_5H_4(CO)_2FeR$, *iii*. $ClCOC_5H_4(CO)_2FeR$

Analogous homo- and heterometallic binuclear complexes have also been prepared by other authors starting from dicyclopentadienylmethanethallium or monometallic derivatives of difulvene.[28,29]

We prepared the "ladder-type" binuclear complexes with a bridging η^1,η^5-cyclopentadienyl ligand by the metallation of $Cp(CO)_2Fe$-η^1-R followed by treatment with $Cp(CO)_2FeI$ as an electrophile[30,31] (Scheme 7).

Scheme 7

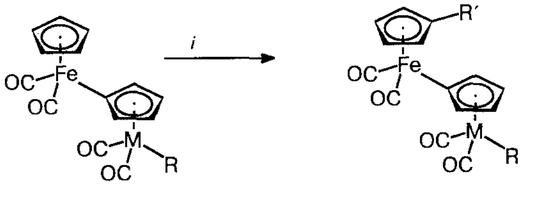


Reagents and conditions: *i*. Bu^{n}Li, $Cp(CO)_2FeI$

Binuclear heterometallic complexes, $Cp(CO)_nM$-η^1,η^5-$C_5H_4Mn(CO)_3$ (M = Fe, *n* = 2, M = Mo, *n* = 3), similar in structure have been described by Nesmeyanov *et al.*[32,33]

Further metallation of "ladder-type" complexes occurs exclusively into the unsubstituted cyclopentadienyl ring and, after reactions with various electrophilic reagents, affords complexes **5a—c**[20,21,34] (Scheme 8).

Scheme 8



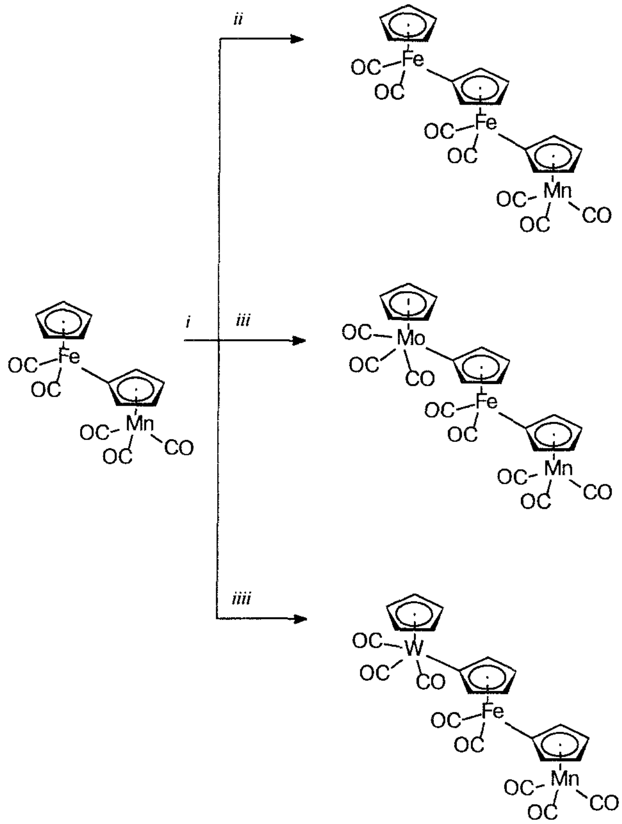
M = Fe, R = CH$_2$Ph
M = Mn, R = CO

5a: R′ = SiMe$_3$
5b: R′ = COOH
5c: R′ = I

Reagents and conditions: *i*. Bu^{n}Li; Me_3SiCl or CO_2 or C_3F_7I

Treatment of the lithium derivative of the $Cp(CO)_2Fe$-η^1,η^5-$C_5H_4Mn(CO)_3$ binuclear complex with the electrophiles $Cp(CO)_nMHal$ (M = Fe, *n* = 2, Hal = I, M = Mo or W, *n* = 3, Hal = Cl) affords "ladder-type" trinuclear heterometallic complexes with Fe—Fe—Mn, Mo—Fe—Mn, or W—Fe—Mn metal sequences[34,35] (Scheme 9).

Scheme 9



Reagents and conditions: *i*. *n*-BuLi, *ii*. $Cp(CO)_2FeI$, *iii*. $Cp(CO)_3MoCl$, *iiii*. $Cp(CO)_3WCl$

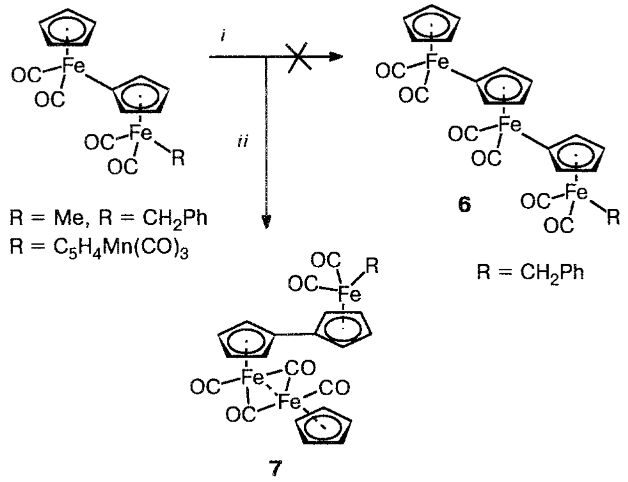
"Ladder-type" homometallic binuclear complexes containing only Fe atoms are less resistant to oxidation and heat than their heterometallic analogs (M = Fe and Mn), therefore, the homometallic trinuclear complexes **6** could not be prepared using $Cp(CO)_2FeI$. Complexes **7** were isolated as the reaction products in all cases (R = Me, CH$_2$Ph, or $C_5H_4Mn(CO)_3$) (Scheme 10).[34,36]

When $Cp(CO)_2FeI$ was replaced by $Cp(CO)_2FeCl$ we obtained complex **6**.[36] It is likely that in the case of $Cp(CO)_2FeI$, complex **6** is also formed, however, the reaction is more intricate and this complex cannot be isolated.

One may assume that complexes **7** are formed as a result of migration of the η^1-bonded ligand in $C_5H_4(CO)_2FeR$ from the Fe atom to the C atom of the lithiated cyclopentadienyl ring and the subsequent interaction of the resulting metal-centered anion with $Cp(CO)_2FeI$ (Scheme 11).

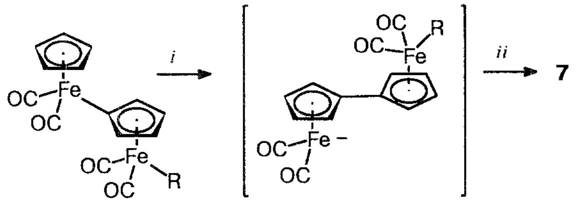
This migration is general and is observed quite often. For example, in an attempt to prepare the symmetrical

Scheme 10



Reagents and conditions: *i.* Bu^nLi, *ii.* $Cp(CO)_2FeI$

Scheme 11



Reagents and conditions: *i.* Bu^nLi, *ii.* $Cp(CO)_2FeI$

tetranuclear complex **8**, we obtained[27] complex **9** (Scheme 12).

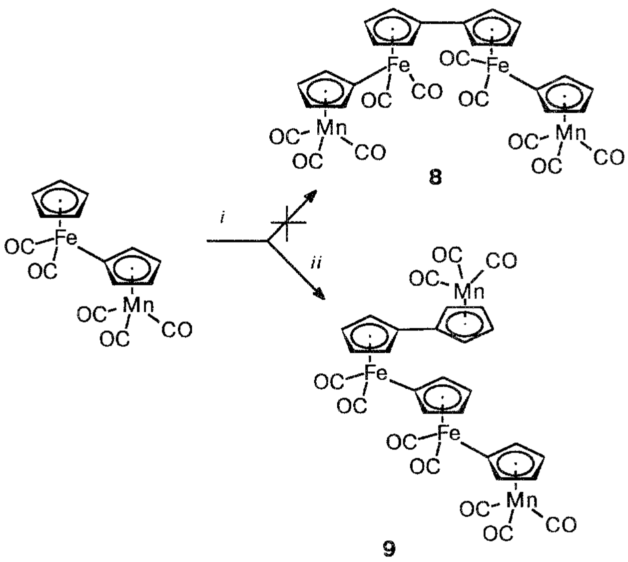
Complex **8** was isolated by us as a product of the reaction of $C_5H_4Li(CO)_2Fe\text{-}\eta^1,\eta^5\text{-}C_5H_4Mn(CO)_3$ with $Cp(CO)_nMCl$ (M = Mo or W), in addition to the "ladder-type" complexes.[35]

The metallation of the $Cp(CO)_2Fe\text{-}\eta^1,\eta^6\text{-}C_6H_5Cr(CO)_3$ binuclear complex[37] followed by the reaction with $PhCH_2Cl$ yielded complex **10** (Scheme 13).[38]

When the latter was treated with I_2, the $Cr(CO)_3$ group was removed and complex **11** formed (Scheme 14).[38] Thus, the migration discovered can be used as a method for introducing a phenyl group into the cyclopentadienyl ring.

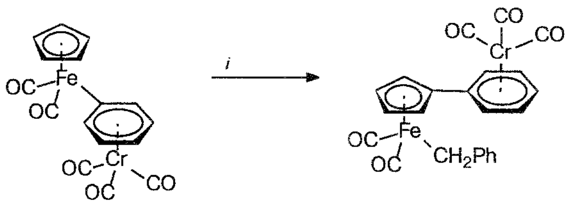
Migration of η^1-bonded ligands from a transition metal atom to the cyclopentadienyl ring during metallation has been observed in a number of works.[39–54]

Scheme 12



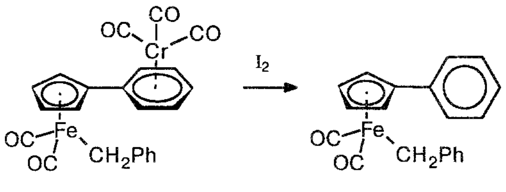
Reagents and conditions: *i.* Bu^nLi, *ii.* $IC_5H_4(CO)_2FeR$ ($R = C_5H_4Mn(CO)_3$)

Scheme 13



Reagents and conditions: *i.* 1. Bu^nLi, 2. $PhCH_2Cl$

Scheme 14



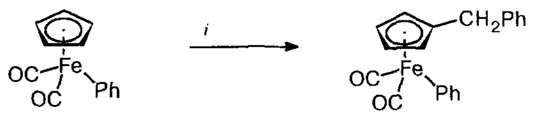
In particular, the migrations of silyl groups, SiR_3 (R = Me, Ph; $R_3 = Me_2Ph$, Me_2Pr^n, or Me_2NMe_2), polysilyl fragments, $(SiMe_2)_nSiMe_3$, $SiMe(SiMe_3)_2$, and $CH_2SiMe_2SiMe_3$ (migration with rearrangement),[39–46]

ER_3 groups (E = Sn, Ge, Pb; R = Me or Ph),[47–49] the phosphoryl group, P(=O)RR′,[50] and acyl groups, RC(=O) (R = H, Me, CH_2Ph, or Ph)[51–54] have been reported.

Barryhill *et al.*[41,55] have studied the mechanism of the migration of SiR_3 groups. It was found that the reaction is intramolecular and occurs with retention of configurations at the metal atom and at the migrating silyl group. One may suggest that the migration of a coordinated aromatic ligand (cyclopentadienyl or phenyl) from the Fe atom to the cyclopentadienyl ring observed by us also occurs according to an intramolecular nucleophilic substitution mechanism, which is made possible by the clear cut electron-withdrawing character of the metal carbonyl fragment.

Nevertheless, in some cases, no migration has been observed. For example, we showed that there is no migration when the $Cp(CO)_2Fe$-η^1-C_6H_5 complex incorporates the η^1-phenyl ligand (Scheme 15).[38]

Scheme 15



Reagents and conditions: *i.* 1. Bu^nLi, 2. $PhCH_2Cl$

As follows from the literature data, the degree of migration of an η^1-bonded ligand from a transition metal atom to the cyclopentadienyl ring during metallation depends on a number of factors and is largely determined by the basicity of the metallating reagent,* the nature of the metal atom, and also depends on the character of the η^1-bonded ligand.

Thus, study of the reactivity of cyclopentadienyl carbonyl complexes of iron, $Cp(CO)_2Fe$-η^1-R (R = Alk, Ar), showed that electrophilic substitution in the cyclopentadienyl ring, which is common in the ferrocene series, is not typical of these compounds. This fact probably hampered the development of the chemistry of the cyclopentadienyl ligands in these complexes for a long period. The discovery of the ability of cyclopentadienyl ligands to undergo protophilic substitution reactions, specifically, metallation, "revived" the cyclopentadienyl ligand in the systems under consideration and made it possible to prepare functionally substituted derivatives, such as aldehydes, alcohols, acids, etc. The presence of both cyclopentadienyl and η^1-bonded ligands in these complexes made it possible to prepare metallacyclic systems.

The synthesis of bi- and polynuclear homo- and heterometallic complexes with bridging cyclopentadienyl ligands including the unique "ladder-type" complexes, also became possible. These compounds are valuable for investigating the metal—metal interactions that give rise to unusual conducting properties of these systems. The migration of an η^1-bonded aromatic ligand, η^5- or η^6-coordinated with the metal carbonyl group, from the Fe atom to the cyclopentadienyl ring, which is observed in some cases in the metallation of "ladder-type" complexes, is an interesting example of the formation of a C—C bond in the coordination sphere of a metal. A thorough study of these processes and their application would enrich the practice of fine organometallic synthesis.

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* In our work, we used Bu^nLi as the metallating reagent. Other authors have also employed Bu^sLi, Bu^tLi, Bu^nLi with TMEDA added, Pr^i_2Li, and $LiN(SiMe_3)_2$.

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