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From nickelocene to novel organonickel compounds

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Contents

Abstract

Nickelocene reacts with organolithium or -magnesium compounds forming unstable 16 VE species {CpNiR}. These species reacts further yielding many new organonickel compounds. The type of R group has a decisive influence on the course of {CpNiR} transformations. If R possesses easily accessible α- or β-hydrogen atoms, then hydrogen elimination is the main reaction course and many cyclopentadienylnickel clusters are formed. The absence of α- and β-hydrogen favours a coupling reaction with R–R formation. Facile stabilisation of free radicals causes homolytic cleavage of the Ni–C bond with alkylation of cyclopentadienyl ring. Some kinds of R groups e.g. $R = CHPh_2$ facilitate formation of a carbene $:CPh_2$ which can react with nickelocene yielding alkylated nickelocene derivatives. {CpNiR} can also undergo intramolecular stabilisation as it occurs for $R = CPh_3$. In most cases the above reactions proceed simultaneously yielding a complex mixture of several compounds. On the other hand some reactions proceed quite selectively forming products in high yield so they can be applied in syntheses. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Nickel; Nickelocene; Nickel clusters; Cyclopentadienyl; Crystal structures

1. Introduction

Nickelocene is the only metallocene with 20 valence electrons. It is paramagnetic and easily oxidised to the nickelocinium ion (19 VE) of limited stability. The cyclopentadienyl groups, especially the first one, are fairly easily displaced from nickelocene. Thus phosphines and phosphites yield NiL_4 [$L = PR_3$, $P(OR_3)$] [1,2], nitrogen monoxide gives CpNi(NO) [3] and nickel tetracarbonyl gives the dimeric complex $[CpNi(CO)]_2$ [4]. The cyclopentadienyl group is also displaced by allyl magnesium chloride [5] and by an azobenzene molecule [6]. The reactivity of nickelocene, and especially the lability of the first cyclopentadienyl group probably arise from the electronic structure of the nickelocene and there is general agreement that the highest occupied MOs have mainly 3d character with the unpaired electrons occupying the antibonding e_{1g} orbitals.

The first step of the reaction of nickelocene with organolithium or -magnesium compounds MR ($M = Li$ or MgX) is the exchange of one cyclopentadienyl group for an alkyl, vinyl or acetylenic group R leading to the formation of unstable 16 VE species {CpNiR}. This coordinately and electronically unsaturated species ap-

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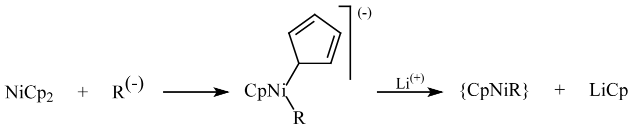
peared to be a precursor of many novel organonickel compounds and clusters and of an active catalyst in acetylene derivative polymerisation. Carrying out reactions of nickelocene with organolithium or -magnesium compounds in the presence of alkenes or alkynes substantially widen the range of new compounds formed and showed that {CpNiR} activated C–H bond in alkenes and alkynes.

2. Reactions of nickelocene with organolithium or -magnesium compounds

Nickelocene easily reacts with organolithium or -magnesium compounds according to Eq. (1):

$$NiCp_2 + \underset{\text{where } M=Li,\ MgX}{MR} \rightarrow \{CpNiR\} + MCp \qquad (1)$$

The exchange of a cyclopentadienyl group for R proceeds in the temperature range from −80 °C to room temperature. Tetrahydrofuran as a solvent distinctly favours the reaction. The mechanism of the exchange reaction has not been studied up to now. It can be assumed, that it proceeds via nucleophilic attack of an anion $R^{(-)}$ on nickel with the change of the bonding mode of one cyclopentadienyl group from η^5-CpNi to η^1-CpNi (Eq. (2)),

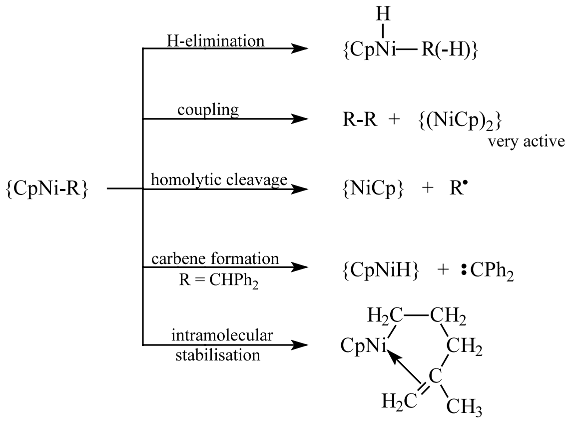


(2)

or via attack of $Li^{(+)}$ on cyclopentadienyl group with the formation of LiCp and $CpNi^{(+)}$ (Eq. (3)).

$$NiCp_2 + Li^{(+)} \rightarrow LiCp + CpNi^{(+)} \xrightarrow{R^{(-)}} \{CpNiR\} \qquad (3)$$

Further reactions lead to the formation of {CpNiR}. {CpNiR} are unstable species, but they can be isolated as complexes with alkenes $CpNiR(R_2C{=}CR_2)$ [7,8].



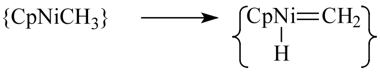
Scheme 1.

Several reactions of nickelocene with organolithium or -magnesium compounds, where R = alkyl, phenyl, vinyl, benzyl, alkynyl and their derivatives, have been studied (Eq. (1)). The intermediate {CpNiR}, formed in the first step of these reactions, undergoes further transformations owing to its coordinative and electronic unsaturation. The main and most important pathways of these transformations are: H-elimination, coupling of R ligands, homolytic cleavage of Ni–R bond, and carbene formation. These reactions are presented in Scheme 1.

The pathways of above reactions and therefore the resulting products depend mainly on the type of the ligand R. Conditions of the reaction (temperature, time stirring, solvent) have less influence. If R possesses easily accessible α- or β-hydrogen atoms, then hydrogen elimination is the main reaction pathway. Absence of α- and β-hydrogen favours a coupling reaction which for R = Ph or C≡CR proceeds in high yield. Facile stabilisation of free radicals causes simultaneous homolytic cleavage and H-elimination as exemplified by $R = C(CH_3)_3$. On the other hand, for $R = CHPh_2$ the α-H transfer to nickel atom facilitates formation of a carbene $:CPh_2$. Thus reactions of unstable {CpNiR} rarely proceed in one direction. Most frequently such reactions lead to a mixture of several compounds. Their separation and isolation as pure compounds frequently present considerable difficulties.

2.1. *H-elimination reactions*

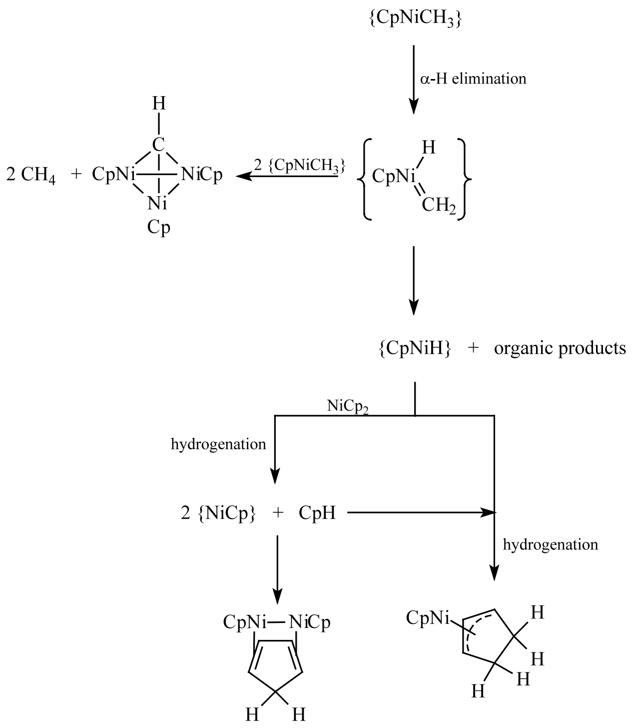
{CpNiR}, formed in the reaction (1), is unstable and easily eliminates hydrogen, which migrates to nickel atom (Eq. (4))



$$\{CpNiCH_3\} \longrightarrow \left\{\underset{H}{\underset{|}{CpNi}}{=}CH_2\right\} \qquad (4)$$

The nickel hydride formed in reaction (4) is unstable although it is 18 VE species. Nickel hydrides are stable in the presence of such ligands as phosphine or carbonyl, but in those cases hydrido ligands are bonded to two or more nickel atoms [9]. Nickel hydrides with σ-bonded organic ligands have not been obtained up to now. The nickel hydride formed in the reaction (4) is a strong hydrogenating agent and immediately reacts with other species present in the reaction mixture (Scheme 2) forming methylidyne trinickel clusters [10], dinickel $(NiCp)_2(\mu\text{-}C_5H_6)$ clusters [11], and mononickel $(NiCp)(\eta^3\text{-}C_5H_7)$ [11] complexes. All the above compounds were isolated and characterised by means of spectroscopic methods or by single crystal X-ray measurements.

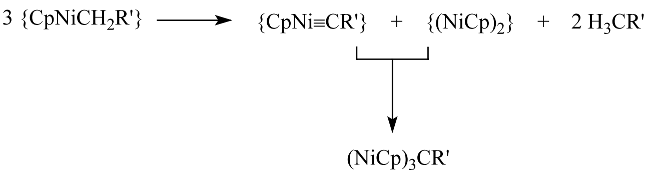
The methylidyne trinickel cluster $(CpNi)_3CH$ can react further with $\{CpNiCH_3\}$ [10] forming fully characterised ethylidyne trinickel cluster $(CpNi)_3C{-}CH_3$ [14]



Scheme 2.

as well as the higher clusters $(CpNi)_5CCH_3$, $(CpNi)_6C$, $(CpNi)_6C_2$, which have not been isolated in a pure form up to now.

{CpNiR} species, where $R = CH_2Ph$ [12], $CH_2C(CH_3)_3$ [13], $CH_2Si(CH_3)_3$ [13], react similarly to $R = CH_3$, eliminating both α-hydrogen atoms and forming tri(cyclopentadienylnickel) clusters (Eq. (5)).

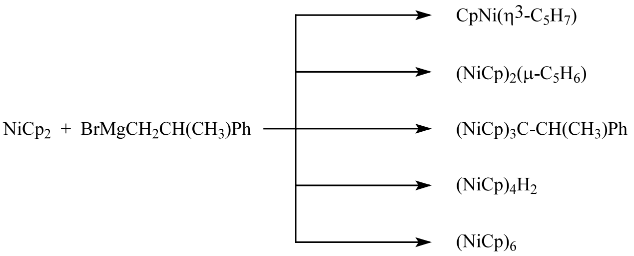


(5)

The reaction of nickelocene with methyllithium and other ligands R possessing two α-hydrogen atoms is the only described efficient method of synthesis of cyclopentadienyltrinickel clusters.

If R possesses β-hydrogen atoms, {CpNiR} undergoes mainly β-H elimination forming various tri-, tetra- and hexanickel clusters. Also mono- and dinickel compounds are formed in these reactions. Reactions of $NiCp_2$ with LiR or BrMgR, where $R = -CH_2CH_3$, $-(CH_2)_2CH_3$, $-(CH_2)_7CH_3$ and $-CH_2CH(CH_3)Ph$ have been studied [15]. The products of the reaction of nickelocene with $BrMgCH_2CH(CH_3)C_6H_5$, separated by column chromatography are presented in Scheme 3.

β-H elimination produces an alkene and active hydrogenating species {CpNiH} (Eq. (6)), which reacts further with nickelocene and other compounds present in the reaction mixture (Scheme 4).



Scheme 3.

$$NiCp_2 + LiR \rightarrow LiCp + \{CpNiR\} \xrightarrow{\beta\text{-H elim.}} \{CpNiH\} + H_2C{=}CHR' \qquad (6)$$

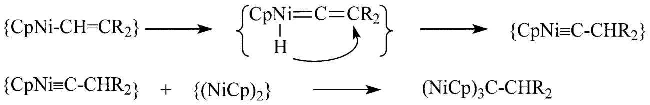
All the products shown below have been characterised by spectroscopic methods and X-ray measurements and the details are given in the cited references.

Vinyllithium or -magnesium compounds possessing an α-hydrogen also form cyclopentadienyltrinickel clusters in the reactions with nickelocene (Eq. (7)).

$$NiCp_2 + Li{-}CH{=}CR_2 \rightarrow \{CpNi{-}CH{=}CR_2\} + LiCp$$

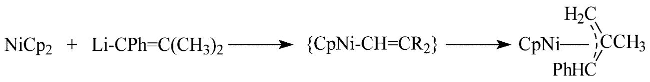
$$3\{CpNi{-}CH{=}CR_2\} \rightarrow (NiCp)_3C{-}CHR_2 + R_2C{=}CH{-}CH{=}CR_2 \qquad (7)$$

Cyclopentadienylnickelvinyl species undergoes α-H elimination followed by 1,3-hydrogen transfer to give carbyne-type unstable species, which react with $\{(NiCp)_2\}$ formed in a coupling reaction to give the trinickel cluster (Eq. (8)) [18].



(8)

Vinylnickel compounds without α- and β-hydrogen, but with γ-H, form π-allyl complexes (Eq. (9)) [19,20].



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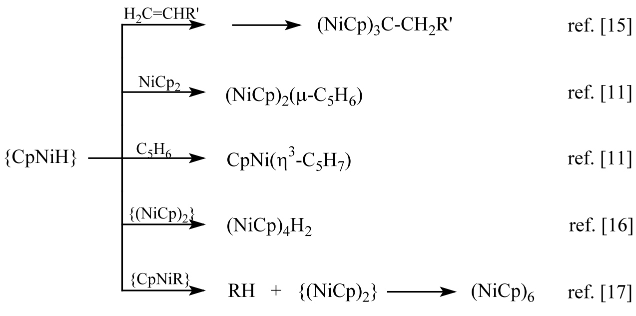


Scheme 4.

Thus reactions of nickelocene with organolithium or -magnesium compounds provide new and easy methods for syntheses of various cyclopentadienylnickel clusters and π-allylnickel compounds. There is no doubt that the main reaction pathway consists in facile α- or β-hydrogen elimination from the ligand R to nickel, followed by its transfer to other carbon atoms what results in the formation of a wide range of new organonickel compounds.

2.2. Coupling reactions

Unstable species {CpNiR} formed in reactions of nickelocene with organolithium or -magnesium compounds (Eq. (1)), besides H-elimination reactions, also undergo coupling reactions. Those reactions occur simultaneously and the ratio of products formed via these two pathways depends on the type of R group.

Such coupling reaction can be considered as reductive elimination, which may proceed via a concerted reaction with low energy pathways. It seems possible that homolytic cleavage generates short-lived radicals (to be distinguished from 'free' radicals) that react with an alkyl group (Eq. (10)).

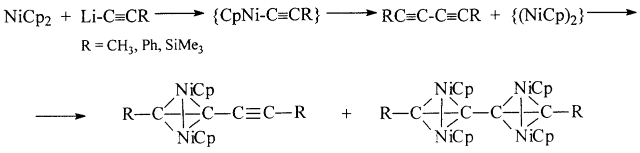
$$2\{CpNi–R\} \rightarrow \{(NiCp)_2\} + R–R \qquad (10)$$

For {CpNiR}, where $R = CH_3$, the main reaction is α-H elimination and methane formation. Coupling of methyl groups with ethane formation occurs at low yield and the molar ratio of CH_4 to C_2H_6 is 6:1 [21]. For R with β-hydrogen atoms (C_2H_5, C_3H_7 etc.) the molar ratio of alkane to alkene is 1:1 and no coupling products R–R are observed, what proves that coupling reaction is insignificant and β-H elimination is the dominant reaction [21].

For R in {CpNiR} without α- and β-hydrogens, coupling reactions predominate. Thus biphenyl is formed in high yield in the reaction of nickelocene with phenyllithium (Eq. (11)) [22].

$$NiCp_2 + LiPh \rightarrow LiCp + \{CpNiPh\} \rightarrow Ph–Ph + \{(NiCp)_2\} \qquad (11)$$

In the reaction of nickelocene with Li–C≡CR, where $R = CH_3$, Ph, $SiMe_3$, a nickel acetylide complex is formed which undergoes coupling to form di- and tetracyclopentadienylnickel complexes (Eq. (12)). The structure of these complexes has been determined by X-ray measurements. [23].



(12)

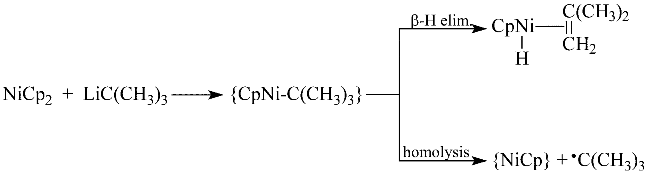
In the above reactions the R groups bonded to nickel do not posses α- and β-hydrogen atoms and therefore coupling is the main course of the reaction. The coupling of vinyl ligands in $\{CpNi–CH=C(CH_3)_2\}$ [19] and $\{CpNi–CPh=CR_2\}$ [11] proceeds in a similar way forming dienes and $\{(NiCp)_2\}$ (Eq. (13)).

$$2\{CpNi–CR=CR'_2\} \rightarrow R'_2C=CR–CR=CR'_2 + \{(NiCp)_2\} \qquad (13)$$

In that case α-, β- or γ-H elimination proceed together with the coupling reaction. The ratio between H-elimination and coupling products depends on the presence and position of hydrogen in the R ligand.

2.3. Homolytic cleavage

Homolytic cleavage of Ni–C bonds occurs when the resulting radical $^{\bullet}R$ is relatively stable and when H-elimination and coupling reactions are not dominant. The unstable species $\{CpNi–{}^tBu\}$ is formed in the reaction of nickelocene with *tert*-buthyllithium. This species undergoes β-H elimination as well as homolytic cleavage of the Ni–C bond (Eq. (14)) [24].

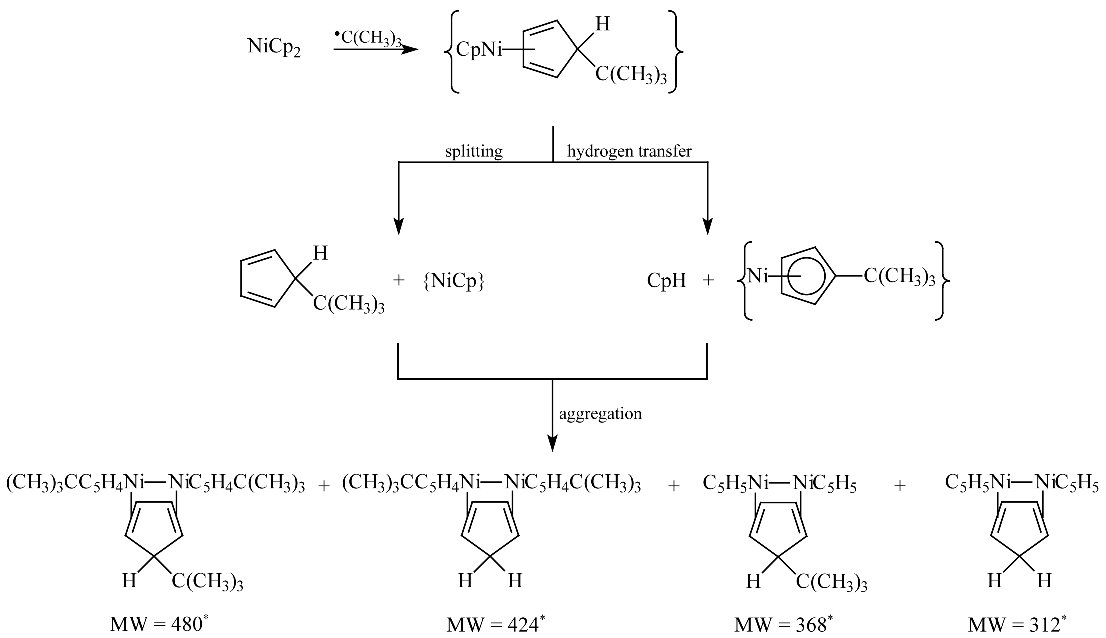


(14)

A free ^tBu radical attacks nickelocene forming unstable $\{CpNiC_5H_5C(CH_3)_3\}$, which undergoes splitting and hydrogen transfer to form cyclopentadiene. An aggregation of the formed active species gives a mixture of four di(cyclopentadienylnickel) complexes (Scheme 5).

Alkylation of the cyclopentadienyl ring of nickelocene leads to the formation of the dinickel complex $[NiC_5H_4C(CH_3)_3]_2[C_5H_5C(CH_3)_3]$ with alkylated rings.

This compound product was isolated and fully characterised by means of spectrometric methods and by X-ray measurements [24]. This was the first example of the alkylation of a cyclopentadienyl ring in nickelocene. Alkylation of cyclopentadienyl ring was postulated in the reaction of nickelocene with benzyllithium [12], in the reaction of (cyclopentadienyl)nitrosylnickel with phenyl- and *tert*-butyllithium [25] and in the reaction of nickelocene with ^tBuMgX in the presence of ethylene [26]. Unfortunately, none of the postulated products of alkylation was isolated and characterised. The other products formed in the reaction of nickelocene with ^tBuMgX in the presence of ethylene have been fully characterised by means of spectrometric methods and by X-ray analysis [26].



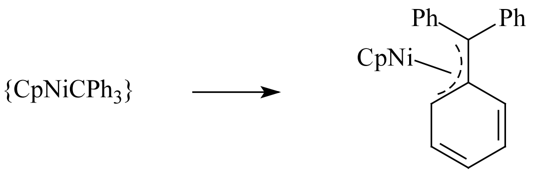
* molecular weight calculated for ^{58}Ni

Scheme 5.

The reaction of nickelocene with triphenylmethyllithium leads to the formation of unstable, 16-electron cyclopentadienyl(triphenylmethyl)nickel {CpNiCPh$_3$} (Eq. (15)).

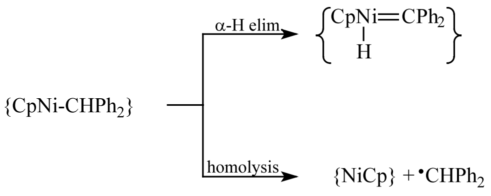
$$NiCp_2 + LiCPh_3 \rightarrow \{CpNiCPh_3\} + LiCp \qquad (15)$$

{CpNiCPh$_3$} does not cleave to form triphenylmethyl radical, but instead rearranges to form a stable π-allylnickel complex, the structure of which has been determined by single crystal X-ray measurements [27,28] (Eq. (16)).



(16)

For this reason, the reaction of free triphenylmethyl radical with nickelocene occurs in very low yield giving unidentified products.



Scheme 6.

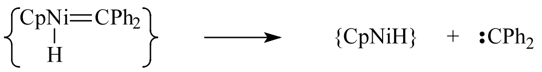
2.4. Carbene formation

Nickelocene reacts with diphenylmethyllithium to form unstable 16 VE species {CpNi–CHPh$_2$} [28] (Eq. (17)).

$$NiCp_2 + LiCHPh_2 \rightarrow \{CpNiCHPh_2\} + LiCp \qquad (17)$$

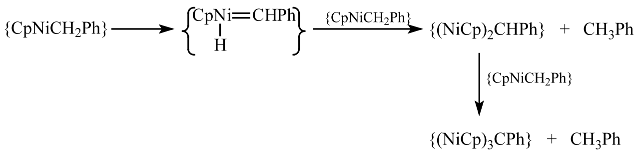
These species react in two ways, namely α-H elimination and homolytic cleavage of the Ni–C bond to form the free radical $^{\bullet}$CHPh$_2$ (Scheme 6).

The α-H elimination product undergoes further splitting to form unstable cyclopentadienylnickel hydride {CpNiH}, a strong hydrogenating agent, as well as the relatively stable carbene :CPh$_2$ (Eq. (18)).

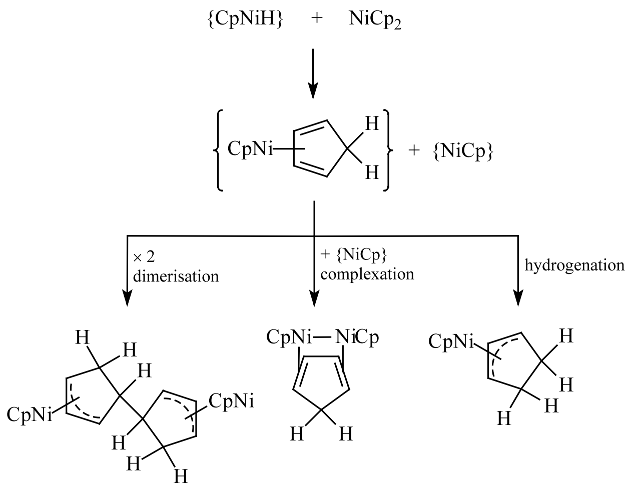


$$\left\{\begin{matrix}CpNi{=}CPh_2\\ | \\ H\end{matrix}\right\} \longrightarrow \{CpNiH\} + \mathbf{:}CPh_2 \qquad (18)$$

If {CpNi–R} possesses more than one α-hydrogen (R = CH$_3$, CH$_2$Ph), the species formed as the result of α-H elimination can undergo consecutive hydrogen elimination again leading to a low yield of the carbene. For this reason trinickel clusters are the main products of transformations of {CpNiR} species (Scheme 7) [28].



Scheme 7.

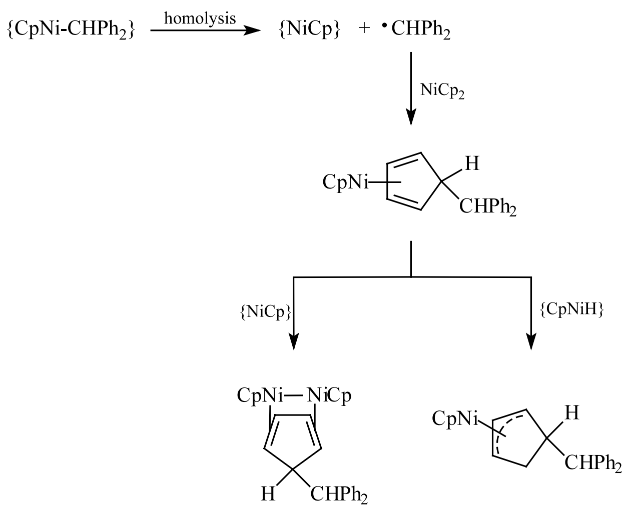


Scheme 8.

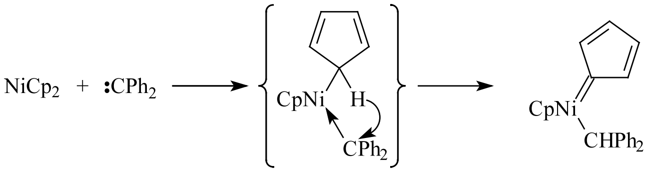
The hydride {CpNiH} (Eq. (18)) hydrogenates nickelocene forming several fully characterised products (Scheme 8).

The free radical $^{\bullet}CHPh_2$ formed by homolysis of $\{CpNi-CHPh_2\}$ (Scheme 6) gives similar types of products (Scheme 9). However, a dimeric compound was not formed owing to steric reasons.

A carbene $:CPh_2$ (Eq. (18) reacts with nickelocene in a different way. As it was shown previously [29–31] a stable carbene 1,3-dimesitylimidazol-2-ylidene attacks nickel atom of nickelocene forming the stable complex $(\eta^5\text{-Cp})Ni(carbene)(\eta^1\text{-Cp})$. Analogously, a carbene $:CPh_2$ reacts with nickelocene to form the unstable species $\{(\eta^5\text{-Cp})Ni(CPh_2)(\eta^1\text{-Cp})\}$, which undergoes hydrogen transfer to a more stable species $\{(\eta^5\text{-Cp})Ni(C_5H_4)(CHPh_2)\}$ (Eq. (19)) [32]. Cobalt analogues of such complexes are known [33–35].

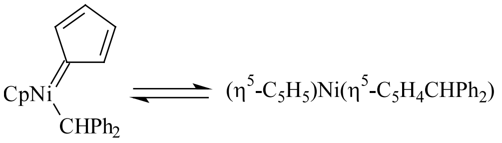


Scheme 9.



(19)

This 18-electron complex, being in equilibrium with alkylated nickelocene, undergoes further attack on a carbene $:CPh_2$, leading to intramolecular rearrangement to form a product in which one of the cyclopentadienyl rings is alkylated with $CHPh_2$ group (Eq. (20)).

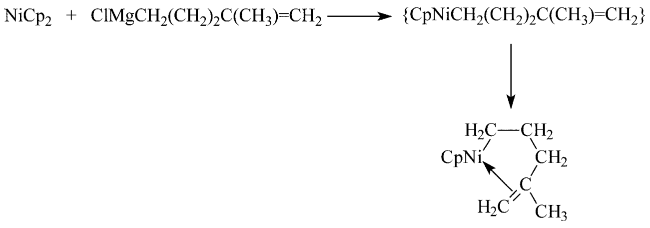


(20)

Further reactions of carbene with the products formed lead to higher alkylated complexes.

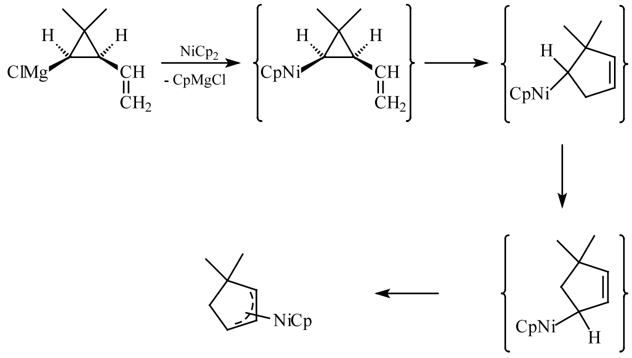
2.5. Intramolecular stabilisation

Some {CpNiR} species can undergo intramolecular transformation by chelation to achieve stable 18-electron form. Thus Lehmkuhl et al. [36] described the reaction of nickelocene with the organomagnesium compound $ClMg(CH_2)_3C(CH_3)=CH_2$. The 16-electron species formed in this reaction undergoes intramolecular transformation to form a stable 18-electron complex (Eq. (21)).



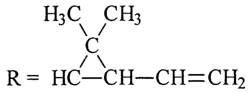
(21)

In this complex hydrogen elimination does not occur, despite the presence of both β- and α-hydrogen atoms.



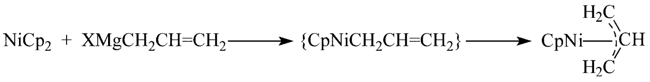
Scheme 10.

Reaction of nickelocene with the organomagnesium compound, where



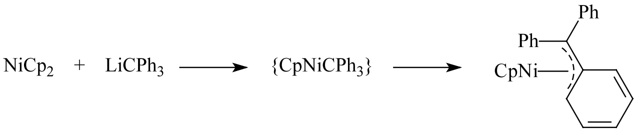
leads to the formation of an intermediate nickel compound which is stabilised by opening of the three-membered ring followed by new ring closure to the η^3-cyclopentenylnickel stable complex (Scheme 10) [37].

Nickelocene reacts with 2-alkenylmagnesium compounds forming cyclopentadienyl(alkenyl)nickel species, which undergoes intramolecular rearrangement to form π-allyl complexes (Eq. (22)) [38].



(22)

The cyclopentadienyl(triphenylmethyl)nickel species $\{CpNi–CPh_3\}$ is stabilised in a similar way (Eq. (23)) [28].



(23)

3. Conclusions

The unstable 16-electron species $\{CpNiR\}$ formed in the reaction of nickelocene with MR ($M = Li$; MgX) reacts further to form many organonickel compounds. For $R = CH_3$, CH_2CH_2R, $CH{=}CR_2$ hydrogen elimination is the main course of the reaction and further transformations lead to the formation of alkylidynenickel clusters. For $R = Ph$, $–C{\equiv}CR$, $–CPh{=}CR_2$ coupling is dominant owing to lack of easily accessible hydrogen for elimination. If R is a derivative of the phenylmethyl group ($\{CpNiCH_2Ph\}$, $\{CpNiCHPh_2\}$) then α-hydrogen elimination, homolytic cleavage of Ni–C bond with the formation of free radicals, and carbene $:CPh_2$ or $:CHPh$ formation proceed simultaneously. Carbenes can alkylate a cyclopentadienyl ring. For $R = –CPh_3$, $–(CH_2)_3CH{=}CH_2$ or $–CH_2CH{=}CH_2$, $\{CpNiR\}$ undergoes intramolecular rearrangement forming stable 18-electron complexes.

From this work $\{CpNiR\}$ can be seen to react in many directions. In these reactions nickel approaches a stable 18 valence electron configuration. The course of the reaction depends mainly on the type of the R group. For some R groups hydrogen elimination, homolytic cleavage of the Ni–C bond and carbene formation proceed at similar rate, what leads to the formation of complex product mixtures.

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