# New triple-decker complexes prepared by the stacking reactions of cationic metallofragments with sandwich compounds

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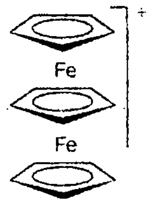
The results of our recent studies devoted to the synthesis of cationic triple-decker complexes are summarized. The stacking reactions of cationic metallofragments with sandwich compounds can be used as a general method for the synthesis of these complexes. This method was used for the preparation of 30- and 34-electron cationic triple-decker complexes containing cyclopentadienyl and pentaphospholyl ligands in the bridging position and carbocycles $C_nH_n$ ($n$ = 4—7) and carboranes as terminal ligands.

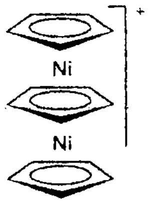
**Key words:** sandwich compounds, triple-decker complexes.

The first sandwich compound, *viz.*, ferrocene, was discovered in 1951—1952.[1—4] This discovery gave impetus to the extensive development of the chemistry of organometallic transition-metal compounds.

Triple-decker complexes belong to a special type of sandwich compounds. Their molecules consist of three parallel cyclic π-ligands between which two transition metal atoms are located. This class of compounds is typified by cations **1** and **2**, which were detected for the first time in 1964 in mass spectrometric studies of ferrocene and nickelocene.[5] However, these data[5] have never received proper attention until the first triple-decker cation **2** was synthesized in 1972 by H. Werner and A. Salzer[6,7] by the reaction of nickelocene with the electrophilic reagents $H^+$, $Ph_3C^+$, or $C_7H_7^+$. It was demonstrated[8] that under the action of $HBF_4$, nickelocene is protonated at one cyclopentadienyl ring to form the $[(\eta\text{-}C_5H_5)Ni(\eta\text{-}C_5H_6)]^+$ cation. The latter eliminates the diene ligand to generate the 14-electron $[(\eta\text{-}C_5H_5)Ni]^+$ species. The reaction of this species with 20-electron nickelocene affords 34-electron cation **2**. An analogous reaction proceeds also under the action of other electrophilic reagents.



**1**



**2**

In 1976, R. Hoffmann and coworkers[9] described the results of extended Hückel molecular orbital calculations for triple-decker complexes. According to these calculations, systems containing 30 and 34 valence electrons should possess enhanced stability. However, attempts to synthesize 30-electron triple-decker complexes with cyclopentadienyl ligands were long unsuccessful.[10,11]

We have developed a general method for the synthesis of cationic triple-decker complexes based on the addition of specially generated cationic metallofragments to sandwich compounds in weakly coordinating polar solvents. In 1987, we described the synthesis of the first representatives of 30-electron triple-decker complexes of the iron-group metals, *viz.*, cations **3**, containing exclusively cyclopentadienyl ligands by the addition of the coordinatively unsaturated 12-electron $[(\eta\text{-}C_5R_5)M']^+$ fragments (M′ = Fe, R = H; M′ = Ru, R = H or Me) generated *in situ* to 18-electron decamethylmetallocenes $(\eta\text{-}C_5Me_5)_2M$ (M = Fe, Ru, or Os) (Scheme 1).[12,13] These reactions came to be known as stacking reactions.

It should be noted that iron- and ruthenium-containing coordinatively unsaturated cationic species were generated under substantially different conditions. The $[(\eta\text{-}C_5H_5)Fe]^+$ species was formed by visible-light irradiation of the $[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_6H_6)]PF_6$ complex in $CH_2Cl_2$ at 20 °C. The ruthenium-containing cations $[(\eta\text{-}C_5R_5)Ru]^+$ (R = H or Me) were generated by heating a solution of $[(\eta\text{-}C_5R_5)Ru(MeCN)_3]PF_6$ in $MeNO_2$. Cations **3** contain at least two pentamethylcyclopentadienyl rings (central and one terminal) and different iron-group metals in the following combinations: $Fe_2$, FeRu, FeOs, $Ru_2$, or RuOs.[12,13] All the cationic triple-decker complexes synthesized by us and described in the present review were isolated as salts with the $PF_6^-$ anion or, more rarely, with the $BF_4^-$ anion (the anions are omitted in the schemes).

In 1993, G. Herberich *et al.*[14] described the synthesis of the related $[(\eta\text{-}C_5Me_5)Ru(\mu\text{-}\eta\text{:}\eta\text{-}C_5H_5)M(\eta\text{-}$

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya*, No. 9, pp. 1636—1642, September, 1999.

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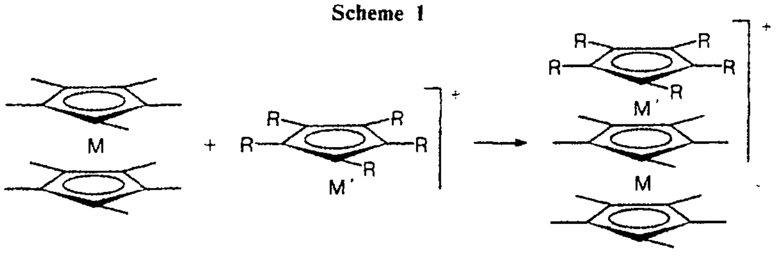
**Scheme 1**



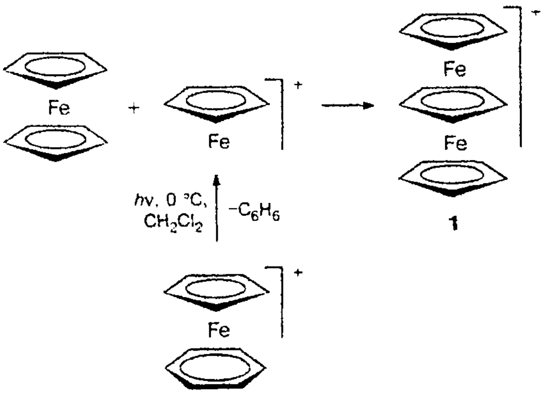
3

M = M' = Fe, R = H;
M = M' = Ru, R = H, Me;
M = Ru, M' = Fe, R = H;
M = Os, M' = Fe, R = H;
M = Os, M' = Ru, R = H.

$C_5Me_5)]CF_3SO_3$ complexes (M = Fe or Ru) in which two pentamethylcyclopentadienyl ligands occupy terminal positions.

The stability of the above-mentioned 30-electron cationic triple-decker complexes is determined to a large extent by the presence of the electron-donating methyl substituents. At the same time, the synthesis of the parent cation **1**, which, as mentioned above, was detected in the mass spectrum of ferrocene, seemed to be quite possible. Recently,[15] we succeeded in preparing cationic complex **1** by the stacking reaction of ferrocene with the cationic $[(\eta\text{-}C_5H_5)Fe]^+$ species generated by visible-light irradiation of the $[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_6H_6)]PF_6$ complex in $CH_2Cl_2$ at 0 °C (Scheme 2).

**Scheme 2**



When the temperature was increased to 20 °C, the same starting compounds gave the ferrocenium cation $[(\eta\text{-}C_5H_5)_2Fe]^+$, while decamethyl derivative **3** (M = M' = Fe; R = H) can be obtained at 20 °C due to its higher stability compared to complex **1**.

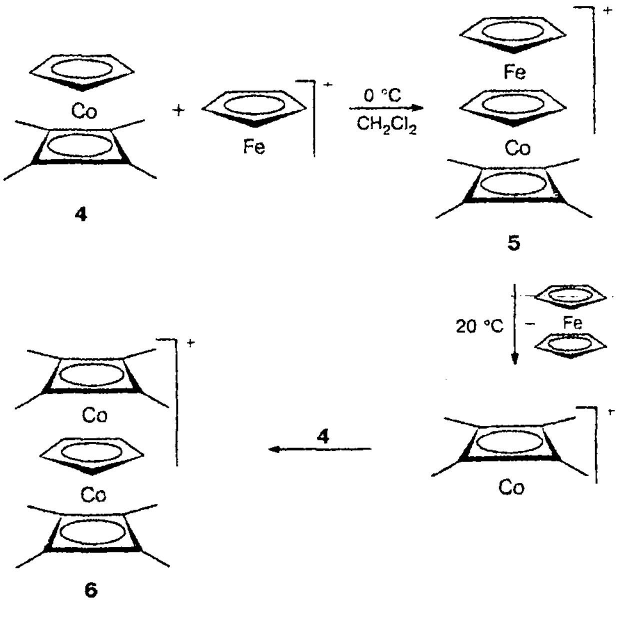
We extended stacking reactions to the synthesis of cobalt-containing triple-decker complexes. Thus the reaction of the $[(\eta\text{-}C_5H_5)Fe]^+$ species with sandwich cobalt compound **4** at 0 °C yielded thermally labile 30-electron iron-cobalt triple-decker complex **5** [15] (Scheme 3).

Complex 5 is formed as a result of the regioselective attack of the cationic $[(\eta\text{-}C_5H_5)Fe]^+$ species on the five-membered ring of compound **4**. If the reaction is performed at higher temperature (20 °C), complex **5** decomposes and eliminates a stable ferrocene molecule to generate the 12-electron cationic $[(\eta\text{-}C_4Me_4)Co]^+$ species. The stacking reaction of the latter with the starting compound **4**, which is present in the reaction mixture, affords the new dicobalt cationic triple-decker complex **6**. To prepare complex **6** in pure form, the starting compounds **4** and $[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_6H_6)]PF_6$ should be taken in a molar ratio of $\geq 2 : 1$.[15]

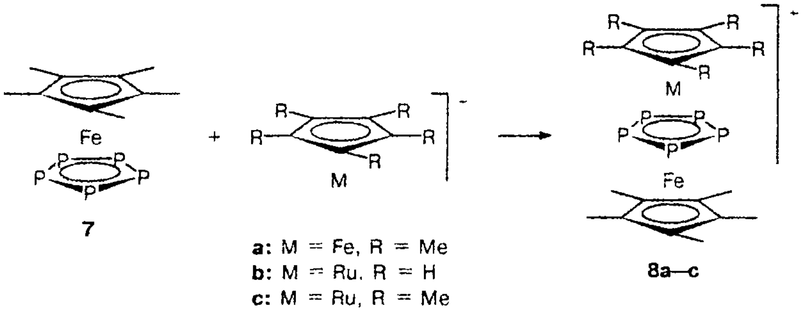
The formation of triple-decker compound **6** from labile complex **5** occurs through formal exchange of ferrocene for compound **4**. This reaction is the first example of the synthesis of cationic triple-decker complexes as a result of exchange of sandwich molecules, which play an unusual (for these compounds) role of π-ligands.

The stacking reactions were also used for the preparation of 30-electron triple-decker complexes containing the pentaphospholyl ligand instead of the cyclopenta-

**Scheme 3**



**Scheme 4**



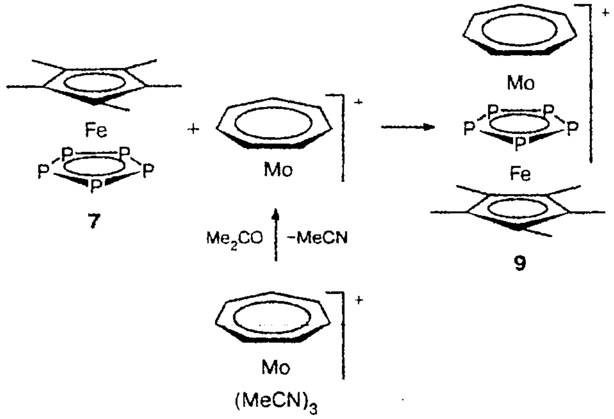
dienyl ring in the bridging position between two transition metal atoms. These reactions were carried out with pentamethylpentaphosphaferrocene $(\eta\text{-C}_5\text{Me}_5)\text{Fe}(\eta\text{-P}_5)$ (**7**) as a metallocene. The latter was synthesized by O. Scherer *et al.*[16,17] by the reaction of $[(\eta\text{-C}_5\text{Me}_5)\text{Fe(CO)}_2]_2$ with yellow phosphorus. The stacking reactions of compound **7** with the 12-electron fragments $(\text{CO})_3\text{M}$ (M = Cr or Mo) and $[(\eta\text{-C}_5\text{H}_5)\text{Fe}]^+$ were used by these authors for preparing triple-decker complexes with a central pentaphospholyl ligand, *viz.*, $(\text{CO})_3\text{M}(\mu\text{-}\eta{:}\eta\text{-P}_5)\text{Fe}(\eta\text{-C}_5\text{Me}_5)$ (M = Cr or Mo)[18] and $[(\eta\text{-C}_5\text{H}_5)\text{Fe}(\mu\text{-}\eta{:}\eta\text{-P}_5)\text{Fe}(\eta\text{-C}_5\text{Me}_5)]^+$.[19] The latter compound was prepared according to a procedure developed by us[12,13] for the synthesis of iron-containing triple-decker complexes **3** (M = Fe, Ru, or Os; M' = Fe; R = H).

We succeeded in extending substantially the range of triple-decker complexes with a central pentaphospholyl ligand using other 12-electron fragments. Thus the stacking reactions of the $[(\eta\text{-C}_5\text{R}_5)\text{M}]^+$ species (M = Fe, R = Me; M = Ru, R = H or Me) with compound **7** yielded triple-decker complexes **8a—c** [20] (Scheme 4). The synthesis of symmetrical diiron cationic complex **8a** required the $[(\eta\text{-C}_5\text{Me}_5)\text{Fe}]^+$ species, which was generated from the acetonitrile complex $[(\eta\text{-C}_5\text{Me}_5)\text{Fe(MeCN)}_3]\text{BF}_4$. The reaction was carried out in $\text{CH}_2\text{Cl}_2$ or acetone at 20 °C.

An analogous reaction with the ruthenium compound $[(\eta\text{-C}_5\text{H}_5)\text{Ru(MeCN)}_3]\text{PF}_6$ under mild conditions (20 °C, $\text{CH}_2\text{Cl}_2$) afforded a complex mixture of products, as was demonstrated by $^{31}$P NMR spectroscopy. Apparently, the formation of iron-ruthenium triple-decker complex **8b** (M = Ru; R = H) was accompanied by side reactions due to coordination of the ruthenium atom at the lone electron pairs of the phosphorus atoms of the starting compound **7**. Examples of analogous interactions were reported for the complexes $(\eta\text{-C}_5\text{Me}_5)\text{Fe}(\eta\text{-P}_5)[\text{Cr(CO)}_5]_2$ and $(\eta\text{-C}_5\text{Me}_5)\text{Fe}(\eta\text{-P}_5)[\text{Mn(CO)}_2(\eta\text{-C}_5\text{H}_5)]_n$ ($n$ = 1—4).[19] We succeeded in performing the selective synthesis of triple-decker complex **8b** under more drastic conditions upon heating to 100 °C in nitromethane. Apparently, products which are formed as a result of coordination of the ruthenium atom at the lone electron pairs of the phosphorus atoms are unstable under the reaction conditions and are converted into more stable triple-decker complex **8b**. The reaction of the pentamethylated $[(\eta\text{-C}_5\text{Me}_5)\text{Ru}]^+$ species with compound **7** yielded triple-decker complex **8c** as the only product.[20] In this case, the $[(\eta\text{-C}_5\text{Me}_5)\text{Ru}]^+$ fragment was generated from $[(\eta\text{-C}_5\text{Me}_5)\text{RuCl}_2]_2$ by refluxing in ethanol or by the reaction with a Zn dust and $\text{TlBF}_4$ in acetone at room temperature. Analogous conditions have been used previously in the synthesis of the cationic arene ruthenium complexes $[(\eta\text{-C}_5\text{Me}_5)\text{Ru}(\eta\text{-arene})]^+$.[21—23]

With the aim of synthesizing an iron-molybdenum triple-decker complex with a central pentaphospholyl ligand, we studied the reaction of compound **7** with the tris(acetonitrile) molybdenum complex $[(\eta\text{-C}_7\text{H}_7)\text{Mo(MeCN)}_3]\text{BF}_4$ as a source of the 12-electron $[(\eta\text{-C}_7\text{H}_7)\text{Mo}]^+$ species. It appeared that this reaction proceeded readily in acetone at room temperature to form unusual cationic triple-decker complex **9** containing the cycloheptatrienyl ligand at the molybdenum atom[24] (Scheme 5).

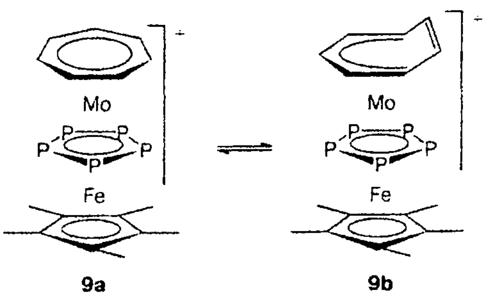
**Scheme 5**



It was found that the $^1$H NMR spectra of compound **9** in $\text{CD}_2\text{Cl}_2$ are temperature-dependent, the signal of the protons of the $\text{C}_7\text{H}_7$ ring being narrowed as the temperature decreases. Apparently, this is due to the fluxional behavior of this ligand. It should be noted that the fluxional behavior in solution is characteristic of $\eta^1$-,

$\eta^3$-, and $\eta^5$-cycloheptatrienyl complexes.[25,26] In the case under consideration, this behavior is attributable to the equilibrium process, which occurs in solution and is accompanied by a change in the coordination mode $\eta^7 \rightleftharpoons \eta^5$ of the cycloheptatrienyl ligand (Scheme 6).

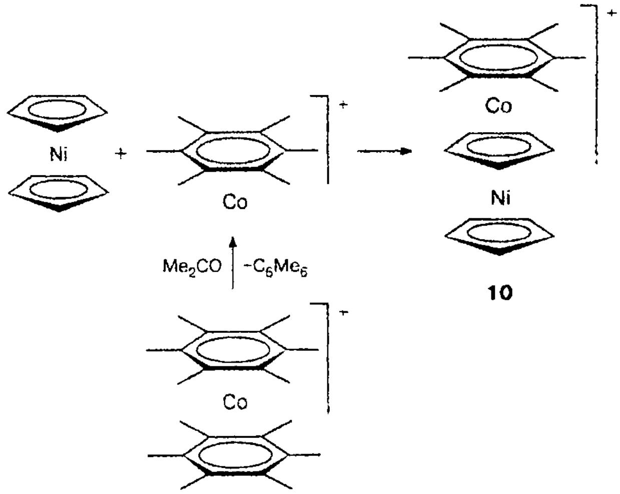
**Scheme 6**



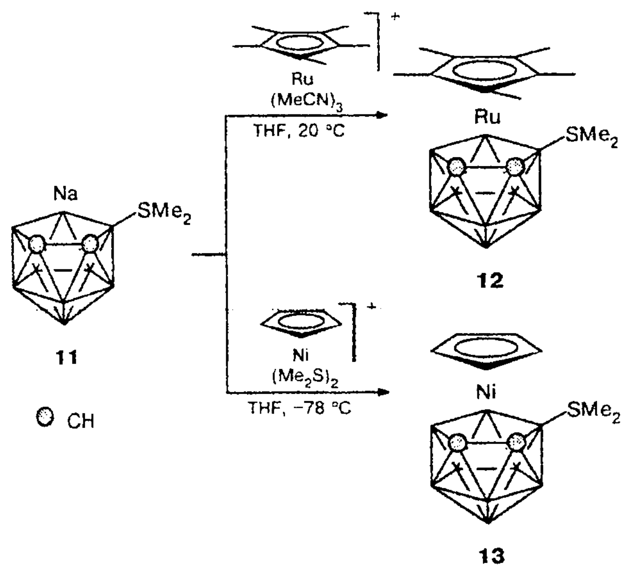
This process involves a change in the number of valence electrons in the triple-decker complex. Form **9a** with the $\eta^7$-$C_7H_7$ ligand has 30 valence electrons, which corresponds to the 18-electron environment of the iron and molybdenum atoms. In the case of form **9b** with the $\eta^5$-$C_7H_7$ ligand, the number of valence electrons in the triple-decker complex decreases to 28. The iron atom retains the 18-electron environment, while the molybdenum atom accepts two less electrons from the $C_7H_7$ ligand, which should lead to the 16-electron configuration of the molybdenum atom. However, in our opinion it is more probable that the molybdenum atom retains its 18-electron environment due to formation of the donor-acceptor Fe→Mo bond through the lone electron pair of the Fe atom. This explanation agrees well with the fact that metal—metal bonds can be formed through large rings (for example, *cyclo*-$P_5$ or *cyclo*-$P_6$), which was confirmed by X-ray diffraction study of a number of compounds, in particular, ($\eta$-$C_5Me_5$)Cr($\mu$-$\eta$:$\eta$-$P_5$)Cr($\eta$-$C_5Me_5$)[27] and ($\eta$-$C_5Me_5$)Mo($\mu$-$\eta$:$\eta$-$P_6$)Mo($\eta$-$C_5Me_5$),[28] and by the results of calculations by the extended Hückel method.[29,30] Apparently, 30-electron form **9a** with the $\eta^7$-$C_7H_7$ ligand is more favorable than 28-electron form **9b** with the $\eta^5$-$C_7H_7$ ligand, due to which the equilibrium is substantially shifted toward the left. This is evidenced by the presence of signals of $\eta^7$-$C_7H_7$ as well as by the absence of signals of $\eta^5$-$C_7H_7$ in the $^1H$ and $^{13}C\{^1H\}$ NMR spectra of compound **9** at 178 K. This compound is the first example of triple-decker complexes with a fluxional cycloheptatrienyl ligand.

As mentioned above, the first triple-decker complex synthesized was 34-electron complex **2**. However, all cationic triple-decker complexes with a central cyclopentadienyl ligand prepared more recently have 30 valence electrons. Hence, it was of great interest to study the possibility of the use of stacking reactions for preparing 34-electron analogs. These compounds would be expected to be prepared by the reactions of 14-electron species, which are isolobal to the $[(\eta$-$C_5H_5)Ni]^+$ fragment, with 20-electron nickelocene. We used the cobalt-containing cationic fragment $[(\eta$-$C_6Me_6)Co]^+$ as a 14-electron species. This fragment was generated *in situ* from the 20-electron bis(hexamethylbenzene) cobalt complex $[(\eta$-$C_6Me_6)_2Co]PF_6$ in acetone. The reaction of this fragment with nickelocene afforded cationic complex **10** [31] (Scheme 7). Complex **10** is the first example of unsymmetrical 34-electron triple-decker complexes with a central cyclopentadienyl ligand.

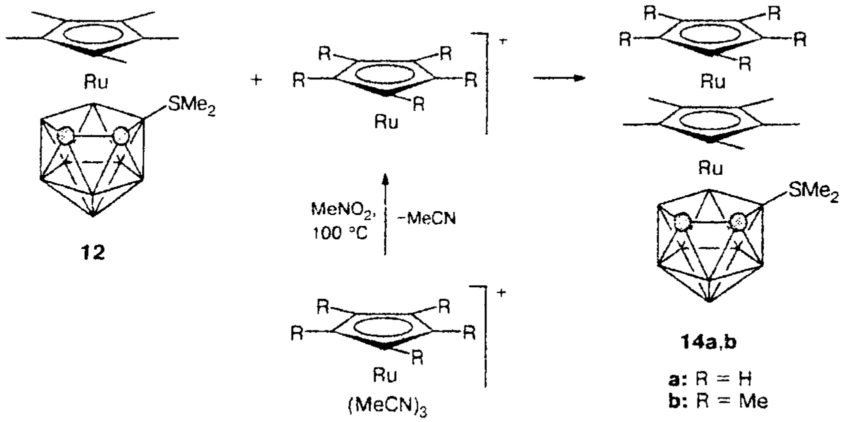
**Scheme 7**



**Scheme 8**



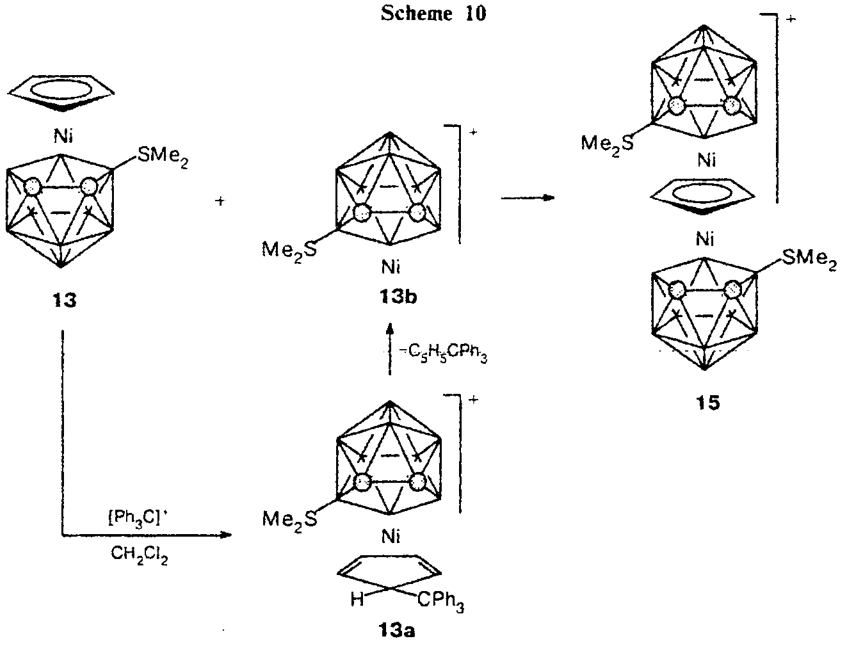
Scheme 9



As part of continuing studies, we demonstrated that not only carbocycles but also carboranes can serve as terminal ligands in cationic triple-decker complexes. We synthesized metallacarborane triple-decker complexes by the stacking reactions using cyclopentadienyl-metallacarboranes (instead of metallocenes) as well as cationic metallacarborane species. The charge-compensated anion $[9\text{-}Me_2S\text{-}7,8\text{-}C_2B_9H_{10}]^-$ was chosen as a carborane ligand. This monoanion,[32–35] like the $[C_2B_9H_{11}]^{2-}$ dianion, is capable of forming π-complexes with transition metals. However, unlike the above-mentioned dianion, the monoanion under consideration resembles to a greater extent the $[C_5H_5]^-$ anion because they carry equal charges. The reaction of the sodium salt $Na[9\text{-}Me_2S\text{-}7,8\text{-}C_2B_9H_{10}]$ (**11**) with the ruthenium complex $[(\eta\text{-}C_5Me_5)Ru(MeCN)_3]PF_6$ (or with $[(\eta\text{-}C_5Me_5)RuCl]_4$) yielded ruthenacarborane **12**[36] (Scheme 8). An analogous reaction of salt **11** with the nickel compound $[(\eta\text{-}C_5H_5)Ni(Me_2S)_2]BF_4$ afforded 20-electron nickelacarborane **13**. Metallacarboranes **12** and **13** can be considered as carborane analogs of ruthenocene and nickelocene, respectively, in which one cyclopentadienyl anion is replaced by the isolobal $[9\text{-}Me_2S\text{-}7,8\text{-}C_2B_9H_{10}]^-$ anion.

With the aim of preparing 30-electron cationic ruthenacarborane triple-decker complexes, we used ruthenacarborane **12** in the stacking reactions with the 12-electron $[(\eta\text{-}C_5R_5)Ru]^+$ species (R = H or Me) generated from the tris(acetonitrile) complexes $[(\eta\text{-}C_5R_5)Ru(MeCN)_3]PF_6$. The reactions were carried

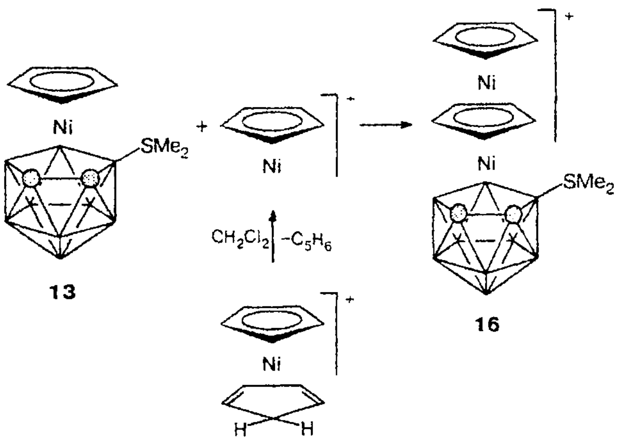
Scheme 10



out upon refluxing in $MeNO_2$, *i.e.*, under the conditions which we have used previously in the synthesis of triple-decker ruthenium complexes **3** (M = M′ = Ru; R = H or Me) with cyclopentadienyl ligands. As a result, unsymmetrical ruthenacarborane triple-decker complexes **14a,b** with a central pentamethylcyclopentadienyl ligand were obtained[36] (Scheme 9).

The use of 20-electron nickelacarborane **13** as the starting compound allowed us to synthesize 34-electron cationic nickelacarborane triple-decker complexes containing one or two carborane ligands. Thus the reaction of compound **13** with $[Ph_3C]PF_6$ taken in a molar ratio of 2 : 1 yielded symmetrical 34-electron bis(carborane) triple-decker cationic complex **15** (Scheme 10). By analogy with the synthesis of triple-decker complex **2** by the reaction of $[Ph_3C]X$ (X = $BF_4$ or $PF_6$) with nickelocene,[6] we believe that the reaction proceeds through intermediate formation of trityl-substituted cyclopentadiene complex **13a**. The latter serves as a source of nickelacarborane fragment **13b**. The subsequent stacking reaction of this fragment with compound **13** gives triple-decker complex **15**.[36] This symmetrical triple-decker complex can be considered as a carborane analog of the classical triple-decker nickel compound **2** containing two carborane anions $[9\text{-}Me_2S\text{-}7,8\text{-}C_2B_9H_{10}]^-$ instead of two terminal anions $[C_5H_5]^-$.

**Scheme 11**



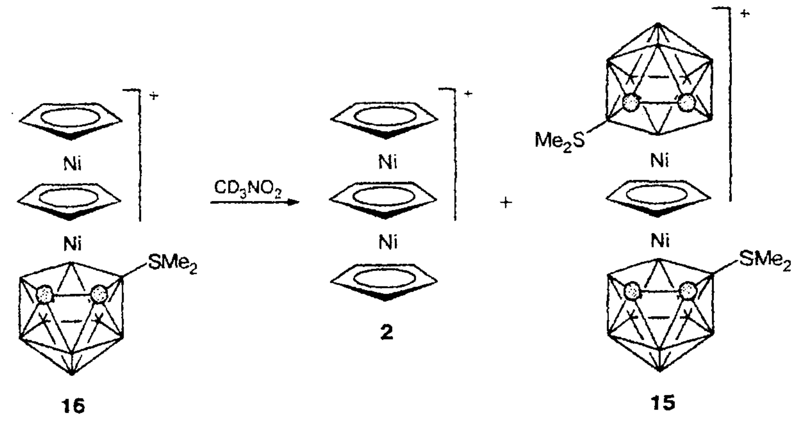
The reaction of compound **13** with the 14-electron $[(\eta\text{-}C_5H_5)Ni]^+$ fragment generated *in situ* from the corresponding cyclopentadienyl complex $[(\eta\text{-}C_5H_5)Ni(\eta\text{-}C_5H_6)]BF_4$ afforded unsymmetrical 34-electron triple-decker compound **16** [36] (Scheme 11). The latter is intermediate between classical triple-decker complex **2** and symmetrical bis(carborane) complex **15**. Complex **16** contains simultaneously the $[C_5H_5]^-$ and $[9\text{-}Me_2S\text{-}7,8\text{-}C_2B_9H_{10}]^-$ anions as terminal ligands.

An interesting feature of unsymmetrical nickelacarborane triple-decker complex **16** is its ability to undergo symmetrization in a nitromethane solution to form symmetrical compounds **2** and **15** (Scheme 12), which is evidenced by the data of $^1H$ NMR spectroscopy. In the case of unsymmetrical ruthenacarborane triple-decker complexes **14a,b**, symmetrization does not occur even in boiling nitromethane (it is these conditions that were used for their preparation). Apparently, this is due to the fact that compounds **14a,b** are substantially more stable than nickel analog **16**.

To confirm the structures of the triple-decker complexes synthesized, we used $^1H$ and $^{13}C$ NMR spectroscopy as the major method. In some cases, we used also $^{11}B$ and $^{31}P$ NMR spectroscopy. It should be noted that the NMR spectra of these compounds are sufficiently informative and, in particular, allow one to readily distinguish signals of the central and terminal ligands. The validity of identification of the cationic triple-decker complexes based on the NMR spectra was additionally confirmed by the X-ray diffraction data for some compounds of this type with central $C_5H_5$,[14,37] $C_5Me_5$,[13,38] and *cyclo*-$P_5$ [19] ligands.

The results surveyed in the present review demonstrate that the stacking reactions of cationic metallofragments with sandwich compounds of different types can serve as an efficient method for the synthesis of

**Scheme 12**



cationic triple-decker complexes containing the cyclopentadienyl and pentaphospholyl ligands in the bridging position.

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*Received March 15, 1999*