A SPECIFIC π-LIGAND TRANSFER REACTION *via* TRIPLE-DECKER SANDWICH COMPLEX INTERMEDIATES

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Dedicated to Dr Karel Mach on the occasion of his 60th birthday.

Photochemically generated cationic cyclopentadienyl iron fragments $[FeCp]^+$ allow a specific stacking reaction of $[(t\text{-}Bu_3C_3P_2)Fe(t\text{-}Bu_2C_2P_3)]$. This results in the transient formation of an unstable cationic Fe–Fe triple-decker sandwich complex which decomposes by deliberating the neutral sandwich complex $[CpFe(t\text{-}Bu_2C_2P_3)]$, and is stacked again by a second $[FeCp]^+$-unit, yielding the moderately stable cationic triple-decker sandwich complex $[CpFe(t\text{-}Bu_2C_2P_3)FeCp]^+$ with a central $\mu\text{-}\eta^5\text{:}\eta^5$-triphospholyl ligand. Air exposure or storage of solutions at room temperature leads to a destacking and the neutral triphosphaferrocene derivative $[CpFe(t\text{-}Bu_2C_2P_3)]$ is formed again almost quantitatively in respect to the pentaphospha-ferrocene educt.

Key words: Iron compounds; Triple-Decker sandwich complexes; Triphospholyl complexes.

Pentaphospha-ferrocene derivatives are accessible by three principal routes: (i) co-thermolysis of $[Cp^*Fe(CO)_2]_2$, ($Cp^* = C_5Me_5$) and P_4 yields $[Cp^*Fe(cyclo\text{-}P_5)]$ (**1**), where the P-atoms form a coordinated P_5-ring[1], (ii) reactive Fe(0)-complexes transform phosphaalkynes into the five-membered ring ligands of $[(R_3C_3P_2)Fe(R_2C_2P_3)]$, R = *t*-Bu (**2a**), adamantyl (**2b**) (refs[2,3]), (iii) $FeCl_2$ reacts with the mixed anion lithium salt $Li^+(t\text{-}Bu_2C_2P_3)^-/(t\text{-}Bu_3C_3P_2)^-$ which generates **2a** in small amounts besides hexaphospha-ferrocene[4]. The cyclization reactions of phosphaalkynes at Fe(0) grant much better yields than the alternative routes. This is especially true, as the preparation of the lithium salt is based on *t*-BuC≡P (**3**) as well[5]. For developing the chemistry of oligophospha-metallocenes systematically, it would be profitable to use the easy to make and very stable pentaphosphaferrocene **2a** as an educt. For this pupose we want to

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develop its chemistry. It is our main goal, to work out specific and high-yield π-ligand transfer reactions of **2a**.

For cleaving the π-bond between a formal anionic five-membered ring ligand and a transition metal, the reductive degradation of metallocenes developed by Jonas[6] is one of the most versatile reactions. First attempts to apply this reaction on **2a**, however, failed fully, in spite of an easier reversible electrochemical reduction **2a** → [**2a**]$^-$ (E^0 = –1.53 V) in relation to ferrocene[7]. A second alternative is an attack on the π-ligands by electron-poor metal ligand fragments, which do not interfere with the lone pairs of the phosphorus atoms, but interact with all π-electrons of one ligand in a stacking reaction. This results in the transformation of a sandwich into a triple-decker sandwich complex. The creation of a μ-η^5:η^5 bonding for the attacked ligand significantly influences its bonding to the iron atom of **2**. In an optimal case, this bond is weakened to an extend that a slight additional chemical stress on the triple-decker results in its breaking and the ligand transfer to the incoming metal atom is completed.

A π-ligand transfer reaction *via* an observable triple-decker intermediate has been reported for the first time by Herberich *et al.*[8]. A boratabenzene is transferred from a Cp*Ru-fragment to Cp*Rh *via* a heteronuclear RuRh triple-decker with a central μ-η^6:η^6-borata-benzene ligand. [Cp*Fe(cyclo-P_5)] can be stacked successfully by suitable transition metal complexes, too[9]. The resulting triple-deckers always contain a μ-η^5:η^5-cyclo-P_5 ligand. Nevertheless the Cp* may function as the middle deck in triple-decker sandwich complexes as well, if only carbacyclic ligands are available[10]. [Cp*Ru(*t*-$Bu_2C_2P_3$)] and related P_2As-species have been stacked recently by cationic [Cp*Ru]$^+$-units, yielding the stable salt [Cp*Ru(μ-η^5:η^5-*t*-$Bu_2C_2P_3$)RuCp*]$^+$[PF_6]$^-$ and its P_2As-analogue[11]. In the same paper Nixon *et al.* report about the failure to stack [Ru(*t*-$Bu_3R_3C_3P_2$)]-units by [Cp*Ru]$^+$ or to prepare corresponding Fe(μ-η^5:η^5-*t*-$Bu_2C_2P_3$)Fe triple-deckers by reacting [Cp*Fe(*t*-$Bu_2C_2P_3$)] with [(C_5H_5)Fe(C_6H_6)]$^+$ [PF_6]$^-$ (**4**) or the corresponding Cp*-salt. Especially UV irradiation of the reaction mixtures caused a decomposition of the educts and a reason for this finding was not found. Reactions of [(C_5R_5)Fe(*t*-$Bu_2C_2P_3$)] with [Cp*Ru(MeCN)$_3$]$^+$[PF_6]$^-$ did not result in the formation of FeRu-triple-deckers, but [Cp*Ru(μ-η^5:η^5-*t*-$Bu_2C_2P_3$)RuCp*]$^+$[PF_6]$^-$ is formed again and the FeRu-species is proposed as an intermediate only. This reaction represents the first successful π-ligand transfer reaction of the triphospholyl ring *t*-$Bu_2C_2P_3$ in 30% yield in maximum.

EXPERIMENTAL

All reactions were carried out under a dry oxygen free nitrogen atmosphere. Solvents were purified by conventional methods, distilled and stored under nitrogen. NMR spectra were recorded close to room temperature on JEOL Lambda 400, FT-JNM-EX 270 and FT-JNM-GX 270 spectrometers, using solvent signals as internal standards, ^{31}P(109.37) MHz δ *vs* 85% H_3PO_4 as external standard. Mass spectra were recorded on a Varian MAT 212 spectrometer. Microanalyses were performed at the Analytical Department of the Institute, using Carlo Erba Elemental Analysers Mod. 1106 and Mod. 1108.

Electrochemical measurements have been carried out with a three-electrode potentiostat PAR M-173, M-174 fitted with a M-178 amplifier and function generator M-175. This equipment was used in combination with a Houston Instruments Series 2000 X–Y-recorder. A Methrom electrochemical cell 6.1415.110 was used with a self-constructed Pt wire working electrode, Pt auxiliary electrode PAR K 0030, and SCE reference electrode PAR K 77. Calibration of measurement is done with ferrocene. Solvent: dimethoxyethane, conducting electrolyte: purified $[n\text{-}Bu_4N]^+[PF_6]^-$.

Crystal structure determination of [(η^5-cyclopentadienyl)(η^5-2,5-di-*tert*-butyl-1,3,4-triphosphacyclopentadienyl)iron] **6**: The data collection was obtained on an automatic fourcircle diffractometer (Siemens P3) with graphite-monochromated MoKα radiation. The crystal structure was solved by direct methods (SHELXS 86) and refined by SHELXL 93. The positional parameters of all hydrogen atoms have been determined from difference Fourier maps and refined anisotropically. Suitable crystals were obtained from light petroleum ether at –30 °C.

The compound crystallizes in monoclinic space group $P2_1/c$ (No. 14), a = 1 370.0(7), b = 963.1(5), c = 1 273.7(10), α = 90°, β = 93.26(5)°, χ = 90°, Z = 4, V = 1.678(2) nm^3, ρ = 1.394 g/cm^3, $R1$ = 0.0328, $wR2$ = 0.0905, 264 refined parameters*.

$[(t\text{-}Bu_3C_3P_2)Fe(t\text{-}Bu_2C_2P_3)]$ **2a** was prepared as reported[2,12] (5 mole *t*-BuC≡P per mole Fe(0) complex, 30% isolated yield of pure material). The new reaction of Binger and Glaser, however, grants even better yields[3].

[Bis(η^5-cyclopentadienyl)(η^5-2,5-di-*tert*-butyl-1,3,4-triphosphacyclopentadienyl)iron]-hexafluorophosphate (**5**)

$[CpFe(C_6H_6)]PF_6$ (1 823 mg, 5.29 mmol) and $[(t\text{-}Bu_3C_3P_2)Fe(t\text{-}Bu_2C_2P_3)]$ (1 376 mg, 2.47 mmol) were dissolved in 100 ml dichloromethane. The green solution was irradiated for 10 h in a photolysis glass apparatus by visible light (125 W mercury lamp), stirred and the temperature was hold constant by cooling with tap water. After removing the solvent *in vacuo* the green residue was dissolved in dichloromethane and the same volume of light petroleum ether was added. Storing the solution for 12 h at –30 °C yields 1.152 mg (1.86 mmol, 75.3%) of green crystals of $[CpFe(t\text{-}Bu_2C_2P_3)FeCp]PF_6$. For $C_{20}H_{28}F_6Fe_2P_4$ (618) calculated: 38.87% C, 4.57% H; found: 39.20% C, 4.99% H. ^{1}H NMR (CD_2Cl_2)(δ/Hz): 1.62 s, 8 H ($C(CH_3)_3$); 4.45 s, 10 H (Cp). $^{13}C\{^1H\}$ NMR (CD_2Cl_2)(δ/Hz): 74.28 s (Cp); 38.81 m ($C(\mathbf{C}H_3)_3$); 36.07 m ($\mathbf{C}(CH_3)_3$), due to a limited solubility the ring carbon signals of the heterocycle are not observable. $^{31}P\{^1H\}$ NMR (CD_2Cl_2)(δ/Hz): AB_2-system; 17.45 t (P_A); 4.6 d (P_B); $^2J(PP)$ = 37.4; –143.62 sept. ($\mathbf{P}F_6$). FD MS (70 eV), *m/z* (%): 473 ((M-$[PF_6]^-$), 100), correct isotopic pattern.

The same product can be obtained by reacting $[CpFe(t\text{-}Bu_2C_2P_3)]$ (**6**; 250 mg, 0.71 mmol) with $[CpFe(C_6H_6)]PF_6$ (260 mg, 0.76 mmol) under the same photochemical conditions in 100 ml dichloromethane for only 15 min. Removing of the solvent *in vacuo* and recrystallization from dichloromethane–heptane (1 : 1) yields 352 mg (0.57 mmol, 80.2%) of pure $[CpFe(t\text{-}Bu_2C_2P_3)FeCp]PF_6$.

[(η^5-Cyclopentadienyl)(η^5-2,5-di-*tert*-butyl-1,3,4-triphosphacyclopentadienyl)iron] (**6**)

$[CpFe(C_6H_6)]PF_6$ (344 mg, 1.0 mmol) and **2a** (184 mg, 0.33 mmol) are reacted in 100 ml dichloromethane photolytically for 12 h as described above. While stirring the mixture without irradiation for

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* Further details of the crystal structure investigations of **6** may be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein–Leopoldshafen (Germany), on quoting the depository number CSD-406024.

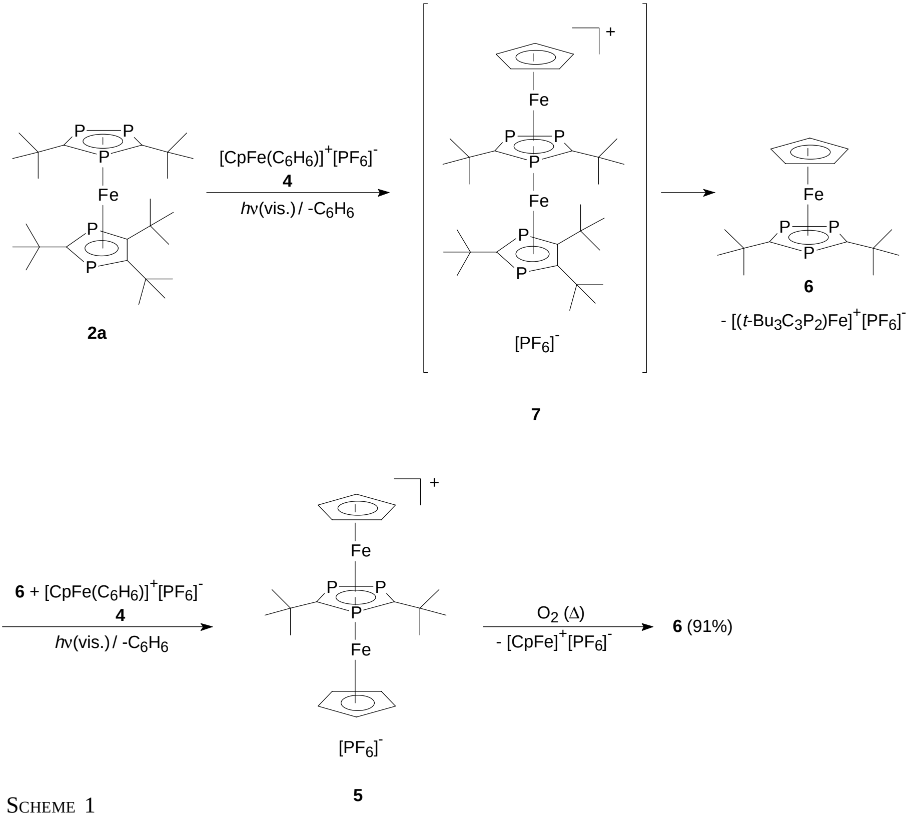
additional 12 h, the colour changes from green to orange. The solvent was removed *in vacuo*, the brown residue was dissolved in light petroleum ether and subjected to column chromatography on SiO_2/5% H_2O. Light petroleum ether as eluat leads to two fractions: the first one yields 17 mg (0.03 mmol) of the green educt $[(t\text{-}Bu_3C_3P_2)Fe(t\text{-}Bu_2C_2P_3)]$ and the second one yields 104 mg (0.3 mmol, 91%) of red $[CpFe(t\text{-}Bu_2C_2P_3)]$. For $C_{15}H_{23}FeP_3$ (352) calculated : 51.17% C, 6.58% H; found: 51.14% C, 7.03% H. ^1H NMR (C_6D_6)(δ/Hz): 1.40 s, 18 H ($C(CH_3)_3$); 4.35 s, 5 H (Cp). $^{13}C\{^1H\}$ NMR(C_6D_6) (δ/Hz): ABCX-system; 144.68 dt, J(C-P) = 299.6; $J(C\text{-}P_2)$ = 207.2 (C-ring); 73.48 s (Cp); 38.57 dt, J(C-P) = 65.6; $J(C\text{-}P_2)$ = 29.7 (**C**$(CH_3)_3$); 36.37 dt, J(C-P) = 32.8; $J(C\text{-}P_2)$ 17.98 ($C(\mathbf{C}H_3)_3$). $^{31}P\{^1H\}$ NMR ($CDCl_3$) (δ/Hz): AB_2-system; 37.84 t (P_A); 37.31 d (P_B); J_{PP} = 35.7. FD MS (70 eV), *m/z* (%) 352 (M^+, 100), correct isotopic pattern.

RESULTS

We performed our stacking experiments of **2a** with **4** which was introduced by Rybinskaya *et al.*[10] in their photochemical route to Cp*-bridged triple-decker sandwich complex cations. To avoid photochemical degradation of products or starting materials, we used visible light from a mercury lamp as a photon source. The reactor was cooled by tap water, which grants a reaction temperature slightly below room temperature. No significant colour change is observed through the course of photolysis: deep green **2a** is transformed into the green salt **5**, which is of moderate stability. Storage of solutions of **5** at room temperature for more than ten hours, warmig up or access of air to the solutions results in a colour change from deep green to orange and a brown precipitate is formed. Red $[CpFe(t\text{-}Bu_2C_2P_3)]$ **6** was isolated from filtered solutions after column chromatography in almost quantitative yield (91%) in respect to the amounts of **2a** used. It was observed for the first time spectroscopically by Bartsch and Nixon as one component of an oily, inseparable reaction mixture, but they have been able to use it as a σ-ligand in connection with a $W(CO)_5$-fragment[13].

To reach quantitative ligand transfer of the triphospholyl ligand $t\text{-}Bu_2C_2P_3$ from **2a** to the [CpFe]-fragment, a minimum of two moles of **4** have to be reacted with one mole of **2a** (Scheme 1). A little higher excess is even better. The same is true for the formation of the intermediate salt **5**. Isolation of pure **5** in good yield is possible, too, if its solutions are handled consequently in the cold. Crystallization by adding light petroleum ether to the filtered reaction mixtures in dichloromethane allows the isolation of analytically and spectroscopically pure material, which is stable as a pure substance at room temperature. To our surprise, the characterization of **5** qualified it as the dark green FeFe-triple-decker salt $[CpFe(\mu\text{-}\eta^5{:}\eta^5\text{-}t\text{-}Bu_2C_2P_3)FeCp]^+[PF6]^-$.

Thus, as proposed, a FeFe-triple-decker sandwich complex with a bridging triphosphacyclopentadienyl ligand is accessible by this reaction, but not the target product $[(t\text{-}Bu_3C_3P_2)Fe(t\text{-}Bu_2C_2P_3)FeCp]^+[PF_6]^-$ (**7**). If we regard **7** as an intermediate of the formation of **5**, lower stability of **7** has to be stated. Its decomposition under the conditions of the reactions must deliberate **6** and leave a cationic $[(t\text{-}Bu_3C_3P_2)Fe]^+$-unit. Up to now, we do not know what happens to this fragment, experiments to reveal its fate are underway. As we did not obtain significant amounts of **6** in fresh photolysis reaction mixtures of **2a** and **4**, **6** seems to react more rapid with **4** than **2a** to form **5**, and its concentration always remains small. An independent photolysis experiment of pure **6** with **4** is in agreement with this assumption. It results in the much more rapid formation of **5**.



SCHEME 1

Spectroscopic and Structural Characterization

The spectroscopic data of the neutral sandwich complex **6** do not agree fully with those, which have been reported earlier[13]. As experimental details are not reported there, the reason for the differences is not clear but the essentials are the same: The phosphorus atoms form an AB_2 spin system and all ^1H and ^{13}C NMR signals of the ligand atoms are in full agreement with a sandwich complex structure. The same is true for triple-decker sandwich salt **5**. Again the P atoms of the heterocycle form an AB_2 spin system in $^{31}P\{^1H\}$ NMR. The coupling constant $^2J(P\text{-}P) = 37.4$ Hz is not only very close to that of the neutral sandwich complex **6**, but is nearly identical with that of isostructural $[Cp^*Ru(\mu\text{-}\eta^5{:}\eta^5\text{-}t\text{-}Bu_2C_2P_3)RuCp^*]^+[PF_6]^-$ (ref.[11]). In both cases the P signals are shifted to higher field, if compared with their neutral mononuclear counterparts. The effect is slightly smaller for the FeFe-compound **5** (*ca* 30 ppm *vs* 60 ppm). Such a high-field shift is characteristic for the NMR signals of μ-η^5:η^5-P-heterocycles, but its value is variable. In the case of the μ-η^5:η^5-cyclo-P_5 ligand bridging two iron atoms the high-field shift reaches more than 160 ppm, for example (ref.[9]).

The terminal Cp ligands of **5** are completely equivalent in their NMR-spectroscopic properties. This proves the existence of an element of symmetry (C_2-axis or horizontal mirror plane) in the central part of the cation, which transforms one Cp ligand into the other, at least under the condition of rapid ring rotation. Therefore alternative complex structures of **5** are rather unlikely. A hypothetical σ-coordination of a second CpFe-fragment to sandwich complex **6** for example would require inequivalent Cp ligands and an ABC spin system for the triphosphacyclopentadienyl ring. The σ-donating properties of η^5-coordinated t-$Bu_2C_2P_3$ are well established: In all cases one of the two neighbouring P atoms functions as a two-electron donor ligand, never the single P atom which is situated between two bulky *t*-Bu substituents[13,14].

The molecular structure of complexes **6** reveals the details of this mixed ligand triphospha-ferrocene derivative (Fig. 1, Table I).

TABLE I
Selected bond lengths (pm) and angles (°) of compound **6**

Bond	Lengths	Bond	Angles
P1–C1	177.0(2)	C2–P2–C1	97.17(10)
P2–C2	176.4(2)	P2–C1–P1	121.37(12)
P3–C2	175.5(2)	P3–C2–P2	122.26(12)
P1–P3	206.08(13)	C1–P1–P3	99.70(8)
P2–C1	176.5(2)	C2–P3–P1	99.43(9)
Fe1–P1	231.9(2)	C32–C31–C35	107.8(3)
Fe1–P2	230.21(10)	C32–C33–C34	108.8(3)
Fe1–P3	231.3(2)	C31–C32–C33	109.0(3)
Fe1–C1	213.1(2)	C33–C34–C35	107.5(3)
Fe1–C2	213.1(2)	C34–C35–C31	107.0(3)
Fe1–C31	206.5(3)		
Fe1–C32	206.9(3)		
Fe1–C33	206.2(3)		
Fe1–C34	205.8(3)		
Fe1–C35	205.0(3)		

As there are no unusual short intermolecular distances in the crystals, packing effects of the neutral molecules do not contribute much to the details of the structures. The two cyclic ligands are almost parallel to each another and all relevant metal to ligand atom distances are as well in the proposed ranges, as the intra ring distances within homo- and heterocycle. The data can be compared with those of binuclear [CpFe(μ-η^5:σ^1-*t*-$Bu_2C_2P_3$)$W(CO)_5$] which contains **6** as the sandwich complex part[13]. All differences between both can be related to the presence of a bulky $W(CO)_5$-fragment in the binuclear species being bonded through one of the two neighbouring P atoms.

DISCUSSION

The data give clear evidence of a new and promising preparative route for the specific transfer of a triphospholyl ligand from a readily available organometallic precursor to another transition metal. Not a trace of the spectating diphospholyl ligand is transferred at the same time. The almost quantitative yield of analytically pure isolated material is by no means common in this field of chemistry, thus mechanistic considerations may help to widen the applicability of the reaction.

As **4**, is supposed to loose coordinated benzene without formation of stable intermediates, the extremely electron-poor iron atom of $[CpFe]^+$ has to attack the π-ligands of **2a** without a real chance of forming a stable σ-complex. At this stage chemoselectivity is established. Possibly because of its sterical hindrance by three *t*-Bu groups, the *t*-$Bu_3C_3P_2$ ligand is not attacked at all. A corresponding observation was made by Nixon, as $[Cp^*Ru]^+$-generating complexes did not react with (*t*-$Bu_3C_3P_2$)M complexes (M = Fe, Ru) (ref.[11]). We thus believe in the formation of triple-decker salt **7** as the primary product. But what may be the reason of its obvious unstability under the reaction conditions while closely related **5** is an isolable compound?

Cyclic voltammetry of **2a** at platinum electrodes proves a high resistance towards oxidation of this pentaphospha-ferrocene derivative. Its oxidation at E_{ox} = +1.33 V is completely irreversible, even at low temperatures and all attempts to characterize a

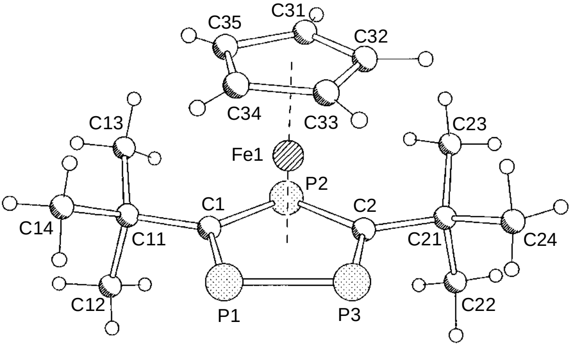


FIG. 1
Molecular structure of [CpFe(*t*-$Bu_2C_2P_3$)] **6**

transient radical cation [**2a**]$^+$ by EPR spectroscopy failed so far. In contrast to this, ferrocene is oxidized reversible at E^0 = +0.43 V (ref.[7]). Diphospha-ferrocene derivative $[(C_4Me_4P)_2Fe]$ mediates between both. Its oxidation potential E^0 = +0.53 V (ref.[15]) is a little more positive than that of ferrocene, but the electron transfer is only partly reversible. $[(C_4Me_4P)_2Fe]^+$ is a short living intermediate. As a consequence we can state an increasing resistance of ferrocene analogues towards oxidation with the number of phosphorus atoms they contain. Parallel to that, the stability of cations decreases dramatically with the introduction of the heteroelements.

If we apply these experimental findings to an asymmetric cationic oligophospha-triple-decker sandwich complex like $[(t\text{-}Bu_3C_3P_2)Fe(t\text{-}Bu_2C_2P_3)FeCp]^+$, the side, containing more P atoms is destabilized. Splitting off the $[(t\text{-}Bu_3C_3P_2)Fe]^+$-unit releases **6**, which is stacked by a second $[CpFe]^+$-fragment to yield symmetric **5**. Thus **6** plays the role of an intermediate as well as the end product of the reaction sequence.

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