

Chemistry of vinylidene complexes

XII¹. Transmetalation of the μ -vinylidene ligand in the reaction of $\text{Cp}(\text{CO})_2\text{MnPt}(\mu\text{-C}=\text{CHPh})(\text{dppp})$ with $\text{Fe}_2(\text{CO})_9$. Formation of new PtFe , PtFe_2 and PtFe_3 complexes

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Abstract

The reaction between $[\text{Fe}_2(\text{CO})_9]$ and $[\text{Cp}(\text{CO})_2\text{MnPt}(\mu\text{-C}=\text{CHPh})(\text{dppp})]$ (1), where dppp is $\text{Ph}_2\text{P}(\text{CH}_2)_3\text{PPh}_2$, proceeds stepwise, initially through transmetalation of the bridging vinylidene ligand, i.e. with replacement of the $[\text{Mn}(\text{CO})_2\text{Cp}]$ fragment by the $[\text{Fe}(\text{CO})_4]$ group, to produce the novel binuclear μ -vinylidene complex $[(\text{dppp})\text{PtFe}(\mu\text{-C}=\text{CHPh})(\text{CO})_4]$ (2), which reacts further with $[\text{Fe}_2(\text{CO})_9]$ to yield the tetranuclear cluster $[(\text{dppp})\text{PtFe}_3(\mu_4\text{-C}=\text{CHPh})(\text{CO})_9]$ (3) and the trinuclear complex $[(\text{dppp})\text{PtFe}_2(\text{CO})_8]$ (4). The IR and ¹H, ¹³C and ³¹P NMR data for the new complexes are reported and discussed.

Keywords: Vinylidene complexes; Manganese carbonyl derivatives; Iron carbonyl derivatives; Platinum complexes; Synthesis; Tetranuclear clusters

Introduction

Recently [1,2], we have proposed an isolobal relationship between methylenecyclopropanes $\text{CH}_2\text{CH}_2\text{C}=\text{CHR}$ and transition metal binuclear vinylidene bridged complexes of the type $[\text{L}_n\text{M}-\text{M}'\text{L}'_m-\text{C}=\text{CHR}]$. It may be expected, therefore, that the exocyclic $\text{C}=\text{C}$ double bond of the latter, which is similar to that of the former, should η^2 -coordinate an additional metal-containing $[\text{M}''\text{L}''_n]$ unit. Subsequent rearrangement and complementary metal–metal bond formation may result in a new trimetal $\text{MM}'\text{M}''$ vinylidene complex.

This working hypothesis was corroborated by synthesis of a series of trimetal μ_3 -vinylidene clusters $[\text{CpMnFePt}(\mu_3\text{-C}=\text{CHPh})(\text{CO})_6\text{L}]$ and $[\text{CpMnFePt}(\mu_3\text{-C}=\text{CHPh})(\text{CO})_5\text{L}_2]$ by means of reactions between

$[\text{Fe}_2(\text{CO})_9]$ and the μ -vinylidene complexes $[\text{Cp}(\text{CO})_2\text{MnPt}(\mu\text{-C}=\text{CHPh})\text{L}_2]$ containing monodentate phosphine or phosphite ligands L such as PPh_3 , $\text{P}(\text{OEt})_3$, $\text{P}(\text{O}^i\text{Pr})_3$ or $\text{P}(\text{OPh})_3$ [1,3].

A quite different result has been obtained [4,5] in the reaction between $[\text{Fe}_2(\text{CO})_9]$ and $[\text{Cp}(\text{CO})_2\text{MnPt}(\mu\text{-C}=\text{CHPh})(\text{dppe})]$, where the Pt atom is chelated by the diphosphine ligand $\text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2$. In this case elimination of the $[\text{Mn}(\text{CO})_2\text{Cp}]$ fragment took place to give the phenylvinylidene-containing product of the new type, namely the cluster $[(\text{dppe})\text{PtFe}_3(\mu_4\text{-C}=\text{CHPh})(\text{CO})_9]$ (5), which was isolated in 91% yield as dark green crystals. A similar reaction was noted earlier by Stone and coworkers [6,7]. The reaction between $[(\text{Cp}(\text{CO})\text{Fe})_2(\mu\text{-C}=\text{CH}_2)(\mu\text{-CO})]$ and $[\text{Co}_2(\text{CO})_8]$ proceeded with elimination of one of the two $[\text{Fe}(\text{CO})\text{Cp}]$ groups to give the tetranuclear μ_4 -vinylidene cluster $[\text{CpFeCo}_3(\mu_4\text{-C}=\text{CH}_2)(\mu\text{-CO})_2(\text{CO})_7]$ (6).

¹ For Part XI, see Ref. [1].

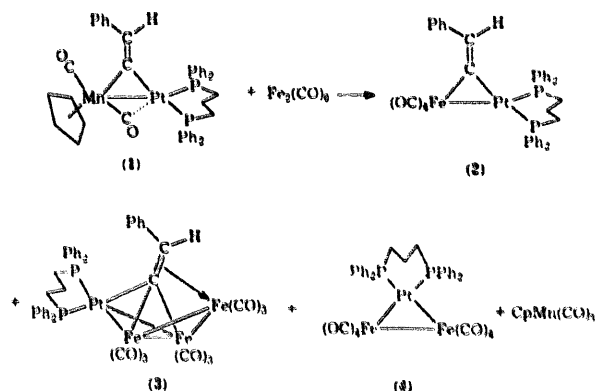
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The mechanism of these complicated reactions has not been determined. It may be possible that the transformation of the methylenedimetalacyclopropane systems into tetranuclear μ_4 -vinylidene clusters consists of several consecutive stages: (i) metalation of the bridging vinylidene ligand resulting in the formation of the trinuclear complex of μ, η^2 -type, (ii) elimination of one of the two initial metal-containing fragments to give the new binuclear μ -vinylidene complex (transmetalation of the vinylidene ligand), and (iii) following new metalation of the μ -vinylidene ligand. However, no intermediate species which would confirm this mechanism have been isolated.

In order to elucidate a pathway for the formation of heterometallic clusters from binuclear μ -vinylidene complexes, we have extended our investigations of the chemistry of the MnPt vinylidene series. This paper describes in detail the results of a study of the reaction between the recently synthesized complex $[\text{Cp}(\text{CO})_2\text{MnPt}(\mu\text{-C}=\text{CHPh})(\text{dppp})]$ (**1**) [8] and $[\text{Fe}_2(\text{CO})_9]$.

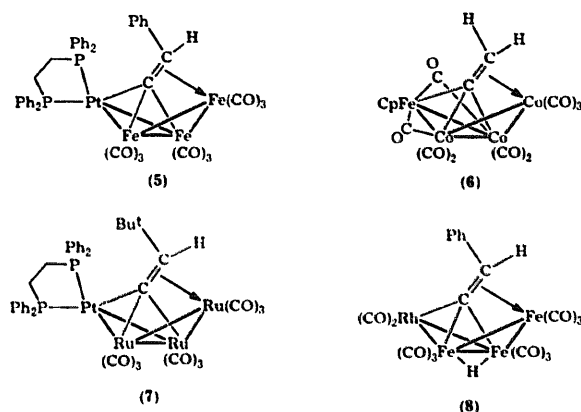
2. Results and discussion

The reaction of $[\text{Cp}(\text{CO})_2\text{MnPt}(\mu\text{-C}=\text{CHPh})(\text{dppp})]$ (**1**) with $[\text{Fe}_2(\text{CO})_9]$ was carried out in a benzene solution at 20°C for 4 h and resulted in a series of new compounds: the binuclear μ -vinylidene complex $[(\text{dppp})\text{PtFe}(\mu\text{-C}=\text{CHPh})(\text{CO})_3]$ (**2**) (yield 13%), the tetranuclear μ -vinylidene cluster $[(\text{dppp})\text{PtFe}_3(\mu_4\text{-C}=\text{CHPh})(\text{CO})_9]$ (**3**) (46%) and the trinuclear cluster $[(\text{dppp})\text{PtFe}_2(\text{CO})_6]$ (**4**) (2%), which does not contain the vinylidene ligand. The new products were characterized by IR and NMR spectroscopy and microanalyses. The eliminated $[\text{Cp}(\text{CO})_2\text{Mn}]$ fragment was isolated as $[\text{CpMn}(\text{CO})_3]$ in 66% yield after chromatography of a reaction solution on an alumina column. No signs of the formation of any trimetal MnFePt μ_3 -vinylidene clusters were seen.



The major product of the reaction is the stable tetranuclear PtFe_3 cluster **3**, which was isolated as a

black–green crystalline substance, poorly soluble in most organic solvents. The principal spectral characteristics of **3** are similar to those found for the known clusters containing the $\mu_4\text{-C}=\text{CHR}$ ligands bridging the “butterfly” type MM'_3 core: **5** [5], **6** [7], **7** [9] and **8** [10], the structures of which have been determined by X-ray studies.



Thus, the IR spectrum of **3** in the $\nu(\text{CO})$ region ($\nu(\text{CO})$ (CH_2Cl_2): 2049s, 1993sh, 1986s, 1930m, br cm^{-1}) is almost identical to that of its dppe analogue **5** (for **5** $\nu(\text{CO})$ (CH_2Cl_2): 2047s, 1994sh, 1987s, 1929m, br cm^{-1} [5]). The resonances of the vinylidene proton $=\text{CHPh}$ were found in the ^1H NMR spectra as triplet signals at low field: δ 6.18 [$J(\text{HP})$ 3.7; $J(\text{HPt})$ 6.2 Hz] for **3** and δ 6.34 [$J(\text{HP})$ 3.4; $J(\text{HPt})$ 5.4 Hz] for **5**.

The presence in the ^{13}C NMR spectra of the signals of C_α at δ 300–320 and C_β at δ 60–100 is typical for the known clusters, which have the $\mu_4\text{-C}_\alpha=\text{C}_\beta\text{HR}$ ligands [2,5–11]. The spectrum of **3** exhibits two vinylidene carbon resonances in the appropriate regions: for C_α at δ 316.03 [$J(\text{CP})$ 36 Hz] and C_β at δ 88.25 [$J(\text{CPt})$ 57 Hz], whereas in the spectrum of **5** only the signal of the C_β carbon at δ 90.74 [$J(\text{CP})$ 12 Hz] was found.

The ^{31}P NMR spectrum of **3** contains two resonances with $J(\text{PPt})$ values close to each other: δ 4.24 [$J(\text{PPt})$ 3520 Hz] and 6.73 [$J(\text{PPt})$ 3450 Hz], which confirms the coordination of both phosphorus atoms with the Pt atom.

The electrochemical behavior of cluster **3** has been studied by means of polarography using the Kalousek switching technique and cyclic voltamperometry [12]. The reduction of **3** in an MeCN solution on a Hg electrode proceeds with successive addition of four electrons at $E_{1/2}$ – 1.33, – 1.55, – 2.63 and – 2.80 V; the first two stages are reversible. On a Pt electrode **3** shows an irreversible one-electron oxidation wave at $E_{1/2} = 0.23$ V.

The minor product from the reaction of **1** with $[\text{Fe}_2(\text{CO})_9]$ is the bright red PtFe_2 cluster **4**, IR spectral

characteristics of which [$\nu(\text{CO})$ (CH_2Cl_2): 2049s, 2003vs, 1967s and 1890m,br cm^{-1}] bear a strong resemblance to those of its crystallographically characterized analogue [(dppe)PtFe₂(CO)₈] (cf. $\nu(\text{CO})$: 2051s, 2002vs, 1968s, 1953m and 1909w cm^{-1}) [5,13] and other related compounds of the type [L₂PtFe₂(CO)₈] with monodentate ligands L (L = Ph₃P, Et₃P, Ph₃As, Ph₃Sb, (RO)₃P, etc.) at the Pt atom [13–15], and differ from those of the PtFe₂ triangle species with bridging diphosphine ligands [16].

Undoubtedly, cluster 4 is a side-product of the reaction between MnPt complex 1 and [Fe₂(CO)₉], and formation of 4 may result from a joining of two [Fe(CO)₄] units with the carbene-like [Pt(dpmp)] moiety arising in a reaction mixture from the complex 2.

Most unexpected was the isolation of the binuclear μ -vinylidene complex [(dpmp)PtFe(μ -C=CHPh)(CO)₄] (2). Only a few complexes containing unsaturated carbenes (vinylidene, allenylidene) linked to one or two [Fe(CO)₄] groups are known, and examples of heterodinuclear vinylidene compounds of the type [(CO)₄FeM(μ -C=CR₂)L_n] are quite rare [2,11].

Complex 2 was isolated as lemon yellow crystals, very soluble in organic solvents. In contrast with the very stable MnPt compound 1 [8], solutions of 2 decompose rapidly even under argon. The structure proposed for 2 is based on analytical and spectroscopic data, since crystals of this unstable compound were not available for an X-ray diffraction study.

The IR spectrum of 2 shows three carbonyl stretching bands with different intensities in the region 2040–1960 cm^{-1} . These $\nu(\text{CO})$ bands are shifted significantly to low frequency compared with those found at 2120–1980 cm^{-1} for the μ -vinylidene complexes [(CO)₄Fe]₂(μ -C=CF₂) [17] and [(CO)₄FeMn(μ -C=CHCOOH)(CO)₂Cp] [18], as well as for the allenylidene compounds [(CO)₄Fe=C=C=C'Bu₂], [(CO)₄Fe]₂(μ -C=C=C=C'Bu₂) [19] and [(CO)₄FeMn(μ -C=C=C=Ph₂)(CO)₂Cp] [18]. Apparently, it is caused by a greater electron-donating capacity of the Pt(dpmp) group and, in addition, the C=CHPh ligand exerts a lesser electron-withdrawing influence on the CO groups than C=CF₂ or C=CHCOOH. It is noteworthy that the $\nu(\text{CO})$ region for 2 is similar to that of phosphine derivatives of iron carbonyl, for example 2040–1940 cm^{-1} for [(CO)₄FePR₃] (R = Ph, Fe) [20].

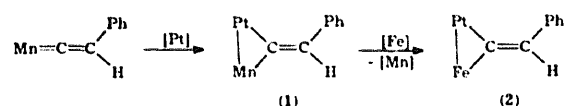
The ³¹P NMR spectrum of 2 reveals two broadened [$J(\text{PP}) \leq 3$ Hz] signals at δ 7.65 and 15.68 with very different values of spin–spin coupling constants $J(\text{PPt})$ (2390 and 4010 Hz respectively) due to the non-equivalent phosphorus nuclei of dpmp coordinated to the Pt atom in a bidentate manner. This spectrum resembles that of the parent complex 1: δ 10.7 [$J(\text{PPt})$ 2365 Hz] and 18.2 [$J(\text{PPt})$ 4030 Hz] [8] but differs significantly from the spectrum of the cluster 3.

The ¹H NMR spectrum of 2 shows resonances of the

CH₂ and C₆H₅ protons, but the signal of the vinylidene proton is masked by those of the phenyl groups.

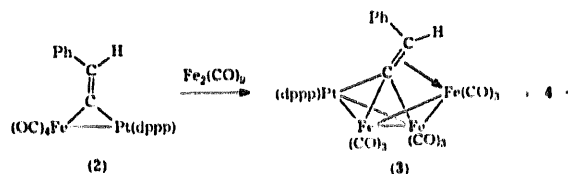
Formally, the complex 2 results from substitution of the [Mn(CO)₂Cp] fragment in the molecule of [Cp(CO)₂MnPt(μ -C=CHPh)(dpmp)] (1) by the [Fe(CO)₄] group, in other words it is a product of transmetalation of the bridging vinylidene ligand. Earlier [21,22] transmetalation reactions were used for synthesis of the μ_3 -vinylidene CoFeM (M = Mo, W, Ni) complexes by means of exchange of one of the two cobalt fragments in Co₂Fe precursors for M-containing groups.

In contrast, since a source of the C=CHPh ligand for the parent MnPt complex 1 was the mononuclear compound [Cp(CO)₂Mn=C=CHPh] [8], the sequence of reactions:



resulting in 2 may be considered as a transfer of the vinylidene ligand from the Mn atom to the binuclear PtFe system. Other examples of transfer of the vinylidene ligand from one metal atom (Mn) to another (Re), to a triangle system (Os₃) and to a tetranuclear cluster core (PtFe₃), which we have established, have been summarized in Ref. [2].

Yields of the products 2 and 3 depend on the duration of the reaction between 1 and [Fe₂(CO)₉]. Thus, an increase of the reaction time from 4 to 6 h results in a decrease in yield of the complex 2 from 13 to 5%, and at the same time the yield of the cluster 3 grows from 46 to 72%. This observation allowed us to suggest that the complex 2 might be a precursor to the tetranuclear PtFe₃ cluster 3. The direct reaction between 2 and [Fe₂(CO)₉] (benzene, 20°C, 4 h) gave cluster 3 in 50% yield, the PtFe₂ triangle complex 4 (3%) and several unidentified species.

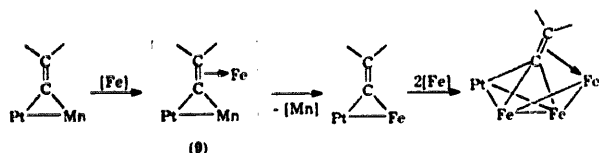


The low yield of 3 in this reaction may result from the instability of the initial compound 2, a significant proportion of which may decompose in the reaction solution before completion for the addition of new iron carbonyl moieties.

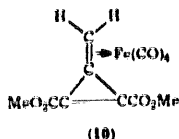
Formation of the PtFe₂ cluster 4 in the last reaction shows that a partial cleavage of both Pt–Fe and Pt–C_u

bonds in the molecule **2** occurs, and the eliminated [Pt(dppp)] unit then combines with two [Fe(CO)₄] units present in the reaction solution.

The isolation of the binuclear PtFe complex **2** sheds some light on the mechanism of formation of the tetranuclear clusters [L₂PtFe₃(μ₄-C=CHPh)(CO)₉] with bidentate ligands L₂ = dppp (**3**) or dppe (**5**) from the appropriate complexes [Cp(CO)₂MnPt(μ-C=CHPh)L₂] and [Fe₂(CO)₉]. Most likely, in the first step coordination of the [Fe(CO)₄] moiety to the exocyclic C=C double bond involved in the methylenedimetallacyclopentane system occurs and results in the unstable trimetal intermediate **9**, where the three-membered MnPtC_α ring remains intact.



The structure of **9** may be similar to that of the iron tetracarbonyl complex **10** with η²-coordinated methylenecyclopropane, which has been established by X-ray studies [23,24].



The stabilization of **9** may result from migration of the [Fe(CO)₄] group into the dimetallacycle, accompanied by displacement of the [Mn(CO)₂Cp] fragment to give [L₂PtFe(μ-C=CHPh)(CO)₄], which undergoes further metalation by [Fe₂(CO)₉] to yield the PtFe₃ cluster. Apparently, the complex [(dppe)PtFe(μ-C=CHPh)(CO)₄] is more reactive towards [Fe₂(CO)₉] than its dppp analogue **2**, and therefore we have failed to isolate it from the reaction between [Cp(CO)₂MnPt(μ-C=CHPh)(dppe)] and [Fe₂(CO)₉] [**5**].

3. Conclusions

New heterometallic compounds [(dppp)PtFe(μ-C=CHPh)(CO)₄] (**2**), [(dppp)PtFe₃(μ₄-C=CHPh)(CO)₉] (**3**) and [(dppp)PtFe₂(CO)₈] (**4**) have been isolated from reaction of [Cp(CO)₂MnPt(μ-C=CHPh)(dppp)] (**1**) and [Fe₂(CO)₉]. Transmetalation of the vinylidene ligand, which occurs in the initial stage of the reaction, gives the binuclear PtFe complex **2**, which undergoes metalation with [Fe₂(CO)₉] to give the tetranuclear PtFe₃ cluster **3**.

4. Experimental details

All operations were carried out under argon. Pure solvents saturated with argon were used. The course of reactions was followed by means of TLC on Silufol plates. Chromatographic separation of products was performed on columns packed with neutral alumina (Brockman activity II).

Infrared spectra were recorded on a Specord IR-75 instrument. The ¹H NMR spectra were recorded on Tesla BS-567 A (100 MHz) and Bruker WP-200 SY (200 MHz) spectrometers. The ¹³C{¹H} and ³¹P{¹H} NMR spectra were registered on a Bruker WP-200 SY (50.31 and 81.01 MHz respectively) instrument. A polarographic study was carried out on a PA3 polarographic analyzer with a three-electrode cell on mercury dropping and platinum stationary electrodes in an MeCN solution, 0.1 M Et₄NBF₄, c = 1 × 10⁻³ M, Ag/0.1 M AgNO₃.

The synthesis of [Cp(CO)₂MnPt(μ-C=CHPh)(dppp)] (**1**) has been described in Ref. [8].

4.1. Reaction of [Cp(CO)₂MnPt(μ-C=CHPh)(dppp)] (**1**) with [Fe₂(CO)₉]

A mixture of the complex **1** (444 mg, 0.5 mmol) and [Fe₂(CO)₉] (920 mg, 2.5 mmol) in 40 ml of benzene was stirred at 20°C for 6 h, until no complex **1** remained (TLC). Then the reaction mixture was filtered through a ca. 1 cm alumina pad, and the filtrate was concentrated under reduced pressure to a volume of ca. 3 ml and cooled to -18°C. The residual black-green crystals of **3** (210 mg) were separated from the mother liquor, the solution was evaporated in vacuo. The residue was solved in 5 ml of a hexane/benzene (1:1) mixture and chromatographed on an alumina column. Five colored fractions were collected successively. A yellow band eluted with a hexane/benzene (2:1) mixture gave, after removal of solvents, 33 mg (42%) of [CpMn(CO)₃]. A red band (hexane/benzene 2:1) gave red crystalline [(dppp)PtFe₂(CO)₈] (**4**) (8 mg, 3%). IR: ν(CO) (CH₂Cl₂) 2049s, 2003vs, 1967s and 1890m, br cm⁻¹. A light brown band (hexane/benzene 1:1) contained an unidentified brown product (10 mg), IR: ν(CO) (CH₂Cl₂) 2040w, 2023s, 1984vs, 1967s and 1923w cm⁻¹.

A yellow band eluted with hexane/benzene (2:3) gave, after evaporation and recrystallization of the residue from hexane/benzene, lemon yellow crystals of [(dppp)PtFe(μ-C=CHPh)(CO)₄] (**2**) (25 mg, 5%). Anal. Found: C, 54.88; H, 4.03; P, 6.65; Fe, 5.63; Pt, 21.27. C₄₂H₃₂O₄P₂FePt Calc.: C, 55.20; H, 4.05; P, 7.02; Fe, 6.10; Pt, 21.37%. IR: ν(CO) (CH₂Cl₂) 2040s, 2013m and 1958vs cm⁻¹. ¹H NMR: δ (CD₂Cl₂) 1.18 (2H, m, CH₂), 2.70 (4H, m, PCH₂), 6.90–7.80 (26H, m, br, 5Ph

and =CH). ^{31}P NMR: δ (CH_2Cl_2) 7.65 (d, $J(\text{PPt})$ 2390 Hz) and 15.68 (d, $J(\text{PPt})$ 4010 Hz).

A dark green band eluted with benzene gave 34 mg of black–green crystals of $[(\text{dppp})\text{PtFe}_3(\mu_4\text{-C=CHPh})(\text{CO})_9]$ (**3**) identical with those residual from the reaction mixture (see above). Total yield of **3** 244 mg (72%). Anal. Found: C, 47.87; H, 3.59. $\text{C}_{44}\text{H}_{32}\text{O}_9\text{P}_2\text{Fe}_3\text{Pt}$ Calc.: C, 48.30; H, 3.00%. IR: $\nu(\text{CO})$ (CH_2Cl_2) 2049s, 1993sh, 1986s and 1930m, br cm^{-1} . ^1H NMR: δ (CDCl_3) 1.60 (2H, m, CH_2); 2.82 (4H, m, PCH_2); 6.18 [1H, t, $J(\text{HP})$ 3.7 Hz, $J(\text{HPt})$ 6.2 Hz, =CH]; 6.73–7.71 (25H, m, 5Ph). ^{13}C NMR: δ (tetrahydrofuran) 19.10 (s, CH_2); 25.74 (m, PCH_2); 88.25 [t, $J(\text{CPt})$ 57.1 Hz, C_β]; 127.10–136.43 (Ph); 212.31, 215.42 and 215.79 (CO); 316.03 [t, $J(\text{CP})$ 35.8 Hz, C_α]. ^{31}P NMR: δ (CH_2Cl_2) 4.24 [d, $J(\text{PP})$ 56.7 Hz, $J(\text{PPt})$ 3520 Hz]; 6.73 [d, $J(\text{PP})$ 56.7 Hz, $J(\text{PPt})$ 3450 Hz].

4.2. Reaction of $[(\text{dppp})\text{PtFe}(\mu\text{-C=CHPh})(\text{CO})_4]$ (**2**) with $[\text{Fe}_2(\text{CO})_9]$

A mixture of complex **2** (85 mg, 0.09 mmol) and $[\text{Fe}_2(\text{CO})_9]$ (117 mg, 0.3 mmol) in 40 ml of benzene was stirred at 20°C for 4 h. The reaction mixture was filtered through a ca. 1 cm alumina pad, concentrated under reduced pressure to a volume of around 5 ml and chromatographed on an alumina column. Three zones were eluted successively with hexane/benzene (2:3) and 34 mg of a mixture of $[(\text{dppp})\text{PtFe}_2(\text{CO})_8]$ (**4**), with an unidentified brown product, then traces of the initial complex **2** and finally the black–green cluster $[(\text{dppp})\text{PtFe}_3(\mu_4\text{-C=CHPh})(\text{CO})_9]$ (**3**) (50 mg, 50%) isolated.

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