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ENHANCED REACTIVITY OF η^1-ALLYL LIGANDS IN TRIALKYLPHOSPHITE SUBSTITUTED $CpFe(CO)_2(\eta^1$-ALLYL) COMPLEXES

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Summary

$CpFe(CO)_2(\eta^1$-allyl) Ia and $CpFe(CO)_2(\eta^1$-methallyl) Ib undergo photochemically initiated carbonyl substitution by bicyclic phosphite III to give IIa and IIb respectively. These latter complexes are more readily protonated than $CpFe(CO)_2(\eta^1$-allyl) complexes and the resulting cationic olefin complexes V and VI are more stable to olefin ligand displacement than similar complexes of the parent system. Both IIa and IIb exhibit high thermal stability, in contrast to Ia or Ib. $CpFe(CO)_2(\eta^1$-2-butenyl) and $CpFe(CO)_2(\eta^1$-cyclopentyl) fail to undergo photochemically initiated CO substitution in the presence of phosphite III, but give instead the dinuclear complex $Cp_2Fe_2(CO)_3(L)$ (L = III). The complex $CpFe(CO)_2(\eta^1$-cyclopentenyl) (VIII) can, however, be prepared indirectly from IIb → VI → VII → VIII. Complex IIa is 900 times more reactive than Ia cycloaddition reaction with β,β-dicyanostyrene.

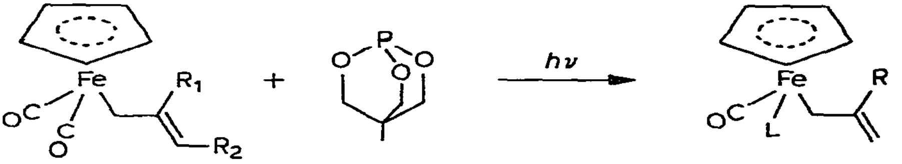
Introduction

The replacement of CO by phosphine or phosphite ligands in metal complexes has been achieved in a number of dinuclear metal carbonyl [1] and hydrido carbonyl [2] complexes by a photochemically initiated radical chain reaction. These reactions have more recently been shown to be an effective means for the replacement of CO by $P(OR)_3$ (R = Me, Ph) in $CpFe(CO)_2(\eta^1$-allyl) (I) [3,4]. Furthermore, it is well known that in general such ligand substitution results in increased electron density at the metal and remaining carbonyl ligands [5]. Consequently, the reactivity of η^1-allyl ligands in phosphite substituted complexes toward electrophiles should be increased since in these reactions charge is transferred from the metal to the electrophile. The present paper reports the preparation and reactions of two new phosphite substitution products of I which show a greatly enhanced degree of reactivity toward electrophiles.

Results and discussion

We chose to examine the reaction of the bicyclic phosphite III with I since it is among the strongest σ-donor phosphites [6] and its rigid cage structure must render the product complex resistant to decomposition through an intramolecular Arbuzov reaction [4]. Moreover, it may be expected to exhibit relatively low steric demand.

Brief irradiation of petroleum ether solutions of Ia and III in the presence of a trace of $[CpFe(CO)_2]$ gave the phosphite complex IIa as a yellow solid, dec. 127°C (90%). This substance exhibits high thermal stability in solution compared with the closely related triphenyl phosphite and trimethyl phosphite substitution complexes IIc and IId. These undergo extensive decomposition on heating at 40°C in CS_2 or $ClCH_2CH_2Cl$ solutions for several hours, while IIa suffers only slight decomposition after heating in $ClCH_2CH_2Cl$ solutions at 60°C for three days.



(I)

(a. R_1 = H, R_2 = H
b. R_1 = Me, R_2 = H
c. R_1 = H, R_2 = Me)

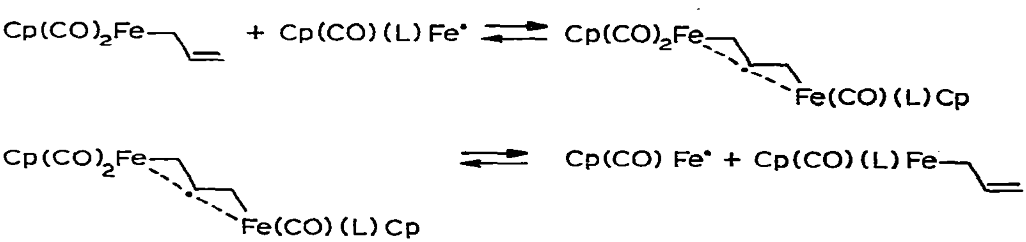
(III)

(II)

(a. L = III, R = H
b. L = III, R = Me
c. L = $P(OPh)_3$, R = H
d. L = $P(OMe)_3$, R = H)

Similar treatment of Ib gave IIb, but reaction of Ic or of $CpFe(CO)_2$(3-cyclopentenyl) IV, with phosphite III gave only the dinuclear complex $Cp_2Fe_2(CO)_3L$ (L = III) *. The failure of these reactions to yield the expected substitution products may be due to steric retardation of allyl ligand transfer in the postulated radical chain reactions (eqs. 1—3), and is consistent with the view that such transfer takes place by an addition elimination or S_H2' pathway [3].

$Cp(CO)_2Fe^{\bullet} + L \longrightarrow Cp(CO)(L)Fe^{\bullet} + CO$



Complex IIa is considerably more basic than in the parent substance Ia.

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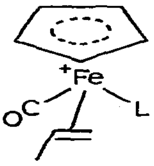
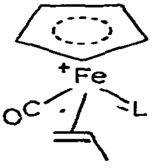
* Analogous phosphine and phosphite complexes have been prepared by direct substitution of carbonyl in $[CpFe(CO)_2]_2$ (ref. 7).

While the latter is not protonated by acetic acid, IIa is completely protonated in THF solution in the presence of two equivalents of acetic acid. A better measure of the comparative basicities of these two complexes was obtained by titration of each in 6 : 1 ethanol-water solutions (v/v) with hexafluorophosphoric acid. Under these conditions, their conjugate acids (V, L = CO, III) show apparent pK_as of 5.0 and 13.8 respectively. *

The NMR spectrum of the propene complex, obtained by protonation of IIa with aqueous hexafluorophosphoric acid, shows that it exists as a 2 : 1 mixture of two diastereomers (Va and Vb, L = III). The predominant isomer is assigned structure Va since in it steric interactions between the methyl group and the Cp ring as well as the phosphite ligand may be minimized in the normally preferred conformation of the olefin ligand [8].



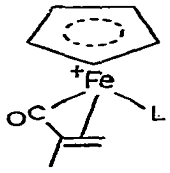
(Va)



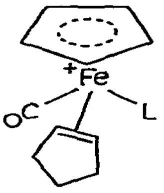
(Vb)

By contrast with the parent cationic complex (V, L = CO) and related substances, which are irreversibly deprotonated with Et_3N in CH_2Cl_2 [9] and undergo rapid olefin displacement on treatment with iodide, chloride or acetate ion, V (L = III) remains unchanged in the presence of these reagents. Deprotonation to regenerate IIa may, however, be effected with methoxide in methanol.

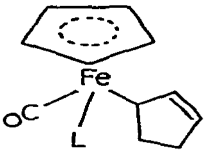
Protonation of IIb similarly gave the cation VI (L = III), which exhibits significantly greater thermal stability than the parent cation (L = CO). Nevertheless it does undergo slow exchange at 60° C in the presence of 10 equivalents of cyclopentene to give the cation VII (L = III). Such exchange reactions, coupled with deprotonation, should provide a useful route to complexes of structure II, which may otherwise be inaccessible through the radical chain reactions (eq. 1—3). Thus deprotonation of VII with methoxide gave the cyclopentenyl complex VIII.



(VI)



(VII)



(VIII)

Like the parent complex Ia, IIa reacts rapidly at —78° C with tetracyanoethylene to give the cycloadduct IX (L = III) in essentially quantitative yield.

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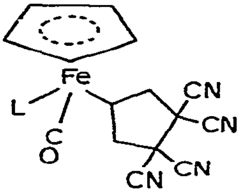
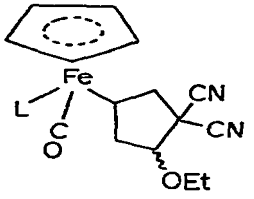
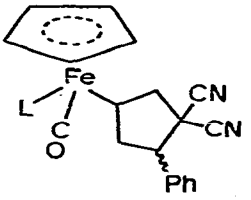
* Both IIa and IIb are strongly adsorbed even on activity III neutral alumina, and on elution of these columns with methanol the product isolated is the salt V or VI. The gegen ion has not been identified.

The greater reactivity of IIa compared with Ia is to be seen in the fact that it forms the adduct Xa with the milder electrophilic olefin, ethoxymethylenemalononitrile, in 85% yield after two hours of reaction at room temperature. By contrast, the parent complex Ia fails to yield any adduct on prolonged exposure to this olefin.


(IX)


(X)


(XI)

(a. L = III,
b. L = $P(OPh)_3$)

A more quantitative measure of the relative reactivities of Ia, IIa and IIc [3] may be derived from competition experiments involving pairs of these complexes with limiting quantities of electrophilic olefins, β,β-dicyanostyrene or ethoxymethylenemalononitrile. The results of these experiments are summarized in Table 1.

The great disparity in the reactivities of IIa and Ia precluded direct competition of these complexes, but the reactivity of the triphenyl phosphite complex IIc was found to lie between them and served as a suitable competing partner for each. Not surprisingly, the increasing reactivity of Ia, IIc and IIa in cycloaddition reactions with electrophilic olefins follows the order of basicity of these complexes, and no doubt reflects the increasing σ-donor power of CO, $P(OPh)_3$ and $P(OCH_2)_3CCH_3$ ligands. The high reactivity of IIa may make this reagent and its congeners useful components in cycloaddition reactions not accessible to those of the parent complex Ia. A further study of the chemistry of IIa and related analogs is in progress.

Experimental

All reactions and subsequent manipulations were carried out in a nitrogen atmosphere. Solvents were routinely dried by standard procedures, maintained under nitrogen over molecular sieves and degassed by passing through a stream

TABLE I

RELATIVE REACTIVITIES OF η^1-ALLYL COMPLEXES

Electrophile	Complex		
	Ia	IIc	IIa
$PhCH=C(CN)_2$	1	180	
$PhCH=C(CN)_2$		1	5
$EtOCH=C(CN)_2$		1	10

of nitrogen prior to use. Infrared spectra were recorded on a Perkin-Elmer spectrophotometer 457. ^{1}H NMR spectra were recorded on either a Varian A-60 spectrometer (NIH GM-13181), a Perkin-Elmer R-32 spectrometer (NSF GU 3852) or a Bruker WH-90 spectrometer (NSF GU 3852 GP 37156). Melting points were determined either in sealed capillaries or under nitrogen and are uncorrected. Elemental Analyses were determined by Galbraith Laboratories, Inc., Knoxville, Tennessee.

Preparation of CpFe(CO)[P(OPh)$_3$](η^1-C$_3$H$_5$) (IIc)

A solution of Ia (10 g, 45.8 mmol) triphenyl phosphite (15 g, 48.23 mmol) and [CpFe(CO)$_2$]$_2$ (1 g, 2.82 mmol) in 200 ml of petroleum ether was irradiated with a 275 watt Sylvania Sunlamp for 30 min. The reaction mixture was allowed to cool to room temperature and then filtered through celite. Solvent was removed in vacuo afforded a red oil, which was chromatographed on neutral alumina (Act. III, 40 × 4 cm). Elution with petroleum ether afforded 15 g of an orange oil, determined by its NMR spectrum to be a 4 to 1 mixture of IIa and triphenyl phosphite. Attempts to separate the triphenyl phosphite were unsuccessful. IR (neat) 1935 cm^{-1}; NMR (CS$_2$) δ 7.10 (m, 15, Ar), 6.00 (m, 1, CH=), 4.50 (m, 2, CH$_2$=), 3.85 (d, 5, J 1 Hz, Cp), 8.00 ppm (m, 2, CH$_2$).

Preparation of CpFe(CO)[P(OMe)$_3$](η^1-C$_3$H$_5$) (IId) [3]

The trimethyl phosphite substitution product IId was prepared as above for IIc: NMR (CS$_2$) δ 6.0 (m, 1, CH=), 4.44—4.7 (m + s, 7, CH$_2$=, Cp), 3.58 (d, 9, J(PH) 12 Hz, CH$_3$), 1.5—1.8 ppm (m, 2, CH$_2$).

Preparation of CpFe(CO)[P(OCH$_2$)$_3$CCH$_3$](η^1-allyl) (IIa)

A solution of Ia (8.77 g, 40.2 mmol), III (6 g, 40.2 mmol) and [CpFe(CO$_2$]$_2$ (0.1 g, 0.28 mmol) in 50 ml of petroleum ether was irradiated for 15 min with a Sylvania Sunlamp. The copious yellow solid which had separated was collected and recrystallized from methylene chloride by the addition of petroleum ether at —78°C to give 10 g of IIa (70%) as a yellow air stable solid, dec. 127°C: Ir (CH$_2$Cl$_2$) 1930 cm^{-1}; NMR (CDCl$_3$) δ 2.02 (m, 1, CH=), 4.78 (m, 1, CH$_2$=), 4.40 (d + m, 6, J 1.2 Hz, Cp and CH$_2$=), 4.18 (d, 6, J 5 Hz, CH$_2$O), 1.70 (m, 2, CH$_2$), 0.75 ppm (s, 3, CH$_3$).

Anal. Found: C, 49.03; H, 5.39. $C_{14}H_{19}FeO_4P$ Calcd.: C, 49.57; H, 5.60%.

Preparation of CpFe(CO)[P(OCH$_2$)$_3$CCH$_3$](η^1-C$_4$H$_7$) (IIb)

A solution of Ib (4 g, 17.24 mmol), III (2.5 g, 17.24 mmol) and [CpFe(CO)$_2$]$_2$ (0.1 g, 0.28 mmol) in 50 ml of petroleum ether was irradiated for 15 min. The yellow solid that formed was collected. Recrystallization from methylene chloride by addition of petroleum ether at —78°C gave 5.9 g of IIb (97%) as a yellow, air stable solid, dec. 91°C: IR (CH$_2$Cl$_2$) 1932 cm^{-1}; NMR (CDCl$_3$) δ 4.50 (m, 1, CH$_2$=), 4.40 (d + m, 6, J 1.2 Hz, Cp and CH$_2$=), 4.20 (d, 6, J 5 Hz, OCH$_2$), 2.10 (d, d, 1, J(HH) 9.0, J(PH) 8.5 Hz, CH$_2$), 1.80 (s, 3, CH$_3$), 1.35 (dd, 1, J(HH) 10.0, J(PH) 11.0 Hz, CH$_2$), 0.80 (s, 3, CH$_3$).

Anal. Found: C, 50.54; H, 6.16 $C_{15}H_{21}FeO_4P$ Calcd.: C, 51.04; H, 5.95%.

Protonation of IIa. Conversion to Va,b

A solution of 1 ml of 65% hexafluorophosphoric acid in 5 ml of diethyl ether was added to a solution of IIa in diethyl ether which had been previously cooled to −78°C. The solution was allowed to warm to room temperature and the solid that formed was collected and washed with diethyl ether. Recrystallization from methylene chloride by addition of ether afforded a 3 : 1 mixture of IVa and IVb (1.2 g, 84%) as a yellow, air stable solid, dec. 163°C: IR (CH_2Cl_2) 2000 cm^{-1}; NMR (CD_3NO_2) δ 5.25 (d, 5, J(PH) 1 Hz, Cp), 4.95 (m, 1, CH=, major isomer), 4.55 and 4.38 (two d, 6, J(PH) 7 Hz, OCH_2), 3.75 (m, CH_2=, minor isomer), 3.50 (d, J 9Hz, *cis*-CH_2=, major isomer), 2.85 (dd, 1, J(PH) 9, J(HH) 15 Hz, *trans*-CH_2=, major isomer, 1.85 and 1.65 (two d, 3, J(HH) 7 Hz, CH_3), 0.90 and 0.82 ppm (two s, 3, CH_3).

Anal. Found: C, 34.70; H, 4.22. $C_{14}H_{20}F_6FeO_4P_2$ Calcd.: C, 34.74; H, 4.13%.

Protonation of IIb. Conversion to VI

Protonation of 1 g of IIb was carried out as in the reaction of IIa and gave 1.18 g (84%) of VI as an orange solid, dec. 145°C: IR (CH_2Cl_2) 2005 cm^{-1}; NMR (CD_3NO_2) δ 5.28 (d, 5, J(PH) 1 Hz, Cp), 4.45 (d, 6, J(PH) 7 Hz, OCH_2), 3.75 (s, 1, CH_2=), 3.05 (d, 1, J 10 Hz, CH_2=), 2.00 (s, 3, CH_3), 1.65 (d, 3, J(PH) 3 Hz, CH_3), 0.85 (s, 3, CH_3).

Anal. Found: C, 36.08; H, 4.49. $C_{15}H_{22}F_6FeO_4P_2$ Calcd.: C, 36.18; H, 4.42%.

Determination of the pK_a's of Ia and IIIa

A solution of ethanol and water (6 : 1, v/v) containing 0.923 mmoles of IIIa was titrated with a 0.101 *N* tetrafluoroboric acid solution made up in the same solvent mixture and standardized with 0.100 *N* sodium hydroxide. The pH of the solution was determined after 0.5 ml increments of acid solution had been added and equilibrium had been reached (10 min). Measurements were carried out using a standard glass reference electrode (Fisher) and a Corning pH meter (model 7). The titration of a solution of Ia was carried out in the same manner. Both titration curves for Ia and IIa showed a sharp change in pH near the equivalent point, but for IIIa the change was more pronounced, as expected for a stronger base.

Attempted reaction of Ic with III. Formation of the dinuclear complex $Cp_2Fe_2(CO)_3(L)$ (L = III)

A solution of Ic (1.9 g, 8.19 mmol), III (1.2 g, 8.19 mmol) and $[CpFe(CO)_2]_2$ (1 g, 2.83 mmol) in 50 ml of petroleum ether was irradiated for 2 h. The solvent was removed in vacuo and the residue was dissolved in methylene chloride and chromatographed on neutral alumina (Act. III, 50 × 4 cm). Elution with petroleum ether afforded, as a first fraction, a yellow oil identified as unchanged Ic. Further elution with methylene chloride gave a purple solid. This was recrystallized from methylene chloride-petroleum ether to give 1.5 g of the dinuclear complex as a purple solid, dec. >260°C: IR (CH_2Cl_2) 1948, 1740 cm^{-1}; NMR ($CDCl_3$) δ 4.71 (s, 5, Cp), 4.61 (d, 5, J 1 Hz, Cp), 3.92 (d, 6, J 5 Hz, OCH_2), 0.52 ppm (s, 3, CH_3).

Anal. Found: C, 44.68; H, 4.01. $C_{18}H_{19}Fe_2O_6P$ Calcd.: C, 45.50; H, 4.00%.

Reaction of IIa with tetracyanoethylene. Preparation of IXa

Tetracyanoethylene (0.377 g, 2.95 mmol) was added to a solution of IIa (1.00 g, 2.95 mmol) in 30 ml of 1,2-dichloroethane at −20°C. The solution was allowed to warm to room temperature and solvent was removed in vacuo. The red oil remaining was dissolved in methylene chloride and passed through 4 cm of neutral alumina (Act. III). Addition of petroleum ether to the filtrate precipitated a yellow solid. Recrystallization of this material from methylene chloride-petroleum ether gave 1.3 g (95%) of IXa as a yellow solid, dec. 167°C: IR (CH_2Cl_2) 1945 cm^{-1}; NMR (CD_3NO_2) δ 4.75 (s, 5, Cp), 4.38 (d, 2, J(PH) 7 Hz, OCH_2), 3.30—2.29 (m, 5, CH, CH_2), 0.83 ppm (s, 3, CH_3).

Anal. Found: C, 51.57; H, 4.18. $C_{20}H_{19}FeN_4O_4P$ Calcd.: C, 51.55; H, 4.08%.

Reaction of IIa with ethoxymethylenemalononitrile. Preparation of Xa

Ethoxymethylenemalononitrile (0.36 g, 2.95 mmol) was added to a solution of IIa (1.00 g, 2.95 mmol) in 30 ml of methylene chloride at room temperature. After stirring for 2 h at room temperature, solvent was removed in vacuo. The red oil remaining was dissolved in methylene chloride and chromatographed on neutral alumina (Act. III, 40 × 4 cm). Elution with ether gave the purified product as a yellow oil which crystallized under high vacuum. Recrystallization from ether at −78°C gave the adduct Xa (1.10 g, 85%) as a yellow solid, dec. 49°C: IR (CH_2Cl_2) 1940 cm^{-1}; NMR ($CDCl_3$) δ 4.48 (d, 5, J(PH) 1 Hz, Cp), 4.20 (d, 6, J(PH) 7 Hz, OCH_2), 3.70 (m, 3, OCH_2CH_3, OCH), 2.70—1.90 (m, 5, CH_2, CHFp), 1.08 (t, 3, J 6 Hz, CH_3), 0.78 ppm (s, 3, CH_3).

Anal. Found: C, 52.30; H, 5.69. $C_{20}H_{25}FeN_2O_5P$ Calcd.: C, 52.23; H, 5.44%.

Reaction of IIc with ethoxymethylenemalononitrile. Preparation of Xb

Ethoxymethylenemalononitrile (0.36 g, 2.99 mmol) was added to a solution of IIc (1.5 g, 2.99 mmol) in 30 ml of methylene chloride at room temperature. After 24 h, solvent was removed in vacuo, leaving a red oil. This was dissolved in methylene chloride and chromatographed on neutral alumina (Act. III 40 × 4 cm). Elution with ether gave 1.5 g (80%) of Xb as a red oil: NMR (CS_2) δ 7.20 (m, 15, Ph), 5.95—6.00 (m, 5, Cp), 5.60 (m, 3, OCH_3 + CHOEt), 3.0—2.0 (m, 5, CH_2), 1.25 ppm (t, 3, J 6 Hz, CH_3).

Reaction of IIa with β,β-dicyanostyrene. Preparation of XIa

β,β-Dicyanostyrene (0.77 g, 2.29 mmol) was added to a solution of IIa (0.35 g, 2.29 mmol) in 30 ml of methylene chloride at room temperature. After stirring for 2 h, solvent was removed in vacuo. The residue was taken up in methylene chloride and passed through 6 cm of alumina (Act. III) with methylene chlorideether solution. Removal of solvent afforded 1 g (89%) of adduct XIa as a yellow air stable solid, dec. 88°C: IR 1935 cm^{-1}; NMR ($CDCl_3$) δ 7.40 (m, 5, Ph), 4.55 (s, 5, Cp), 4.20 (d, 6, J(PH) 7 Hz, CH_2O), 4.1—1.8 (m, 6, CH_2, CHFp, CHPh), 0.75 ppm (s, 3, CH_3).

Reaction of IIc with β,β-dicyanostyrene. Formation of adduct XIb

β,β-Dicyanostyrene (0.61 g, 3.90 mmol) was added to a solution of IIc (2 g, 3.90 mmol) in 30 ml of methylene chloride at room temperature. After stirring for 2 h, solvent was removed in vacuo, leaving a red oil. This was dissolved in methylene chloride and chromatographed on neutral alumina (Act. III, 60 × 4 cm). Elution with ether yielded 2.20 g (83%) of XIb as a red oil. Crystallization was effected by dissolving the oil in methylene chloride, adding an equal volume of petroleum ether and cooling to −78°C. The product was obtained as a partially solid material: IR (CH_2Cl_2) 1935 cm^{-1}; NMR ($CDCl_3$) δ 7.50 (m, 15, Ph), 4.27 and 4.24 (two s, 5, Cp), 4.0—2.0 ppm (m, 6, CHPh, CHFp, CH_2).

Competition reactions

The competing allyl complexes (3.90 mmol each) were dissolved in 30 ml of methylene chloride at room temperature and the electrophilic olefin (0.39 mmol) was added at once. The resulting solution was stirred at room temperature for 12 h. Solvent was then removed in vacuo and the residue was dissolved in a small volume of methylene chloride and chromatographed on neutral alumina (Act. III, 20 × 4 cm). Elution with petroleum ether-ether solutions gave the starting allyl complexes. Methylene chloride eluted the cycloadducts together. The ratio of these was determined from their NMR spectra using the ratio of cyclopentadienyl proton resonances.

For the competition reaction involving Ia and IIc with β,β-dicyanostyrene a 10 fold molar excess of Ia to IIc was used.

Exchange reaction of VI with cyclopentene. Formation of VII

A solution of VI (2.0 g, 45 mmol) and cyclopentene (3.0 g, 45.3 mmol) in 1,2-dichloroethane was refluxed for 30 min. After cooling the solution to room temperature, solvent was removed in vacuo, leaving a red oil. The crude product was taken up in methylene chloride and ether was then added to precipitate a yellow solid. Recrystallization of this material from methylene chloride-ether afforded VII as a yellow, air stable solid, dec. 148°C (1.0 g, 50%): IR 2005 cm^{-1}; NMR (CD_3NO_2) δ 5.55 (m, 2, CH=), 5.20 (d, 5, J(PH) 1 Hz, Cp), 4.55 (d, 6, J(PH) 7 Hz, OCH_2), 2.25—1.70 (m, 6, CH_2), 0.92 ppm (s, 3, CH_3).

Anal. Found: C, 36.99; H, 4.50. $C_{16}H_{22}FeO_4P_2F_6$ Calcd.: C, 37.51; H, 4.29%.

Deprotonation of VII by methoxide

The olefin salt VII (0.15 g, 0.33 mmol) was dissolved in 50 ml of absolute methanol at 0°C. Sodium metal (7.5 mg, 0.33 mmol) was added and the solution was stirred at 0°C for 10 min and then at room temperature for 45 min. The solution was filtered through 4 cm of celite and magnesium sulfate. Removal of solvent in vacuo left 60 mg of red oil, which was found, by IR an and NMR spectroscopy to be a mixture of VIII and an unidentified product. The IR spectrum of the mixture exhibited a band at 1980 cm^{-1} in addition to the absorption at 1925 cm^{-1}, assigned to VIII: NMR (CD_3NO_2) δ 5.30 (m, 2, CH=), 4.80 (s, 5, Cp), 4.60 (d, 6, J 7 Hz, CH_2O), 2.5—1.2 ppm (m, 6, CH, CH_2, CH_3).

Acknowledgement

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