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A STUDY OF THE METALATION OF 1,4-DISUBSTITUTED 2-BUTYNES BY SODIUM η^5-CYCLOPENTADIENYLDICARBONYLFERRATE AND HYDRIDO-η^5-CYCLOPENTADIENYLDICARBONYLIRON

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Summary

The metathesis between Na[η^5-$C_5H_5(CO)_2Fe$] (NaFp) and 1,4-dichloro-2-butyne or 2-butyne-1,4-diol bis(benzene)sulfonate afforded primarily $FpCH_2C\equiv CCH_2Fp$ (I) and small amounts of $FpC(=CH_2)CH=CH_2$ (VIII) and $FpCH_2C\equiv CCH_3$ (IX). 1,4-Dibromo-2-butyne gave only small amounts of VIII and IX and none of the binuclear complex I. The yields of VIII and IX increased dramatically when t-butanol was present in the reaction mixture. In the presence of methanol, ethanol, or i-propanol, η^3-*E*-[$CH_2CHCMe(CO_2R)$]-η^5-$C_5H_5(CO)Fe$ (R = Me, Et, i-Pr) was isolated in addition to VIII and IX. Complex VIII also was prepared by the reductive demetalation of $Fp[CH_2=CHC(Fp)=CH_2]^+PF_6^-$ which was obtained by protonation of I.

Introduction

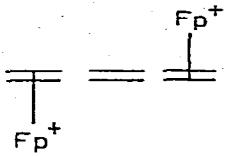
Although the binuclear butatriene complex I was first prepared twelve years ago, the chemistry of this substance has received little attention. In light of our recent discovery that novel η^2-cyclobutadienoid complexes [2,3] (IV and V) can be prepared by the oxidation of neutral binuclear complexes we began an investigation of the chemistry of I with the aim of preparing the unknown η^2-butatriene complexes II and III which would be of structural and synthetic interest. Herein we report the initial results of our studies that pertain to the metalation and reductive metalation of 1,4-disubstituted 2-butynes by Na[η^5-C_5H_5-$(CO)_2Fe$] (NaFp).

Results

The reactions between 1,4-dichloro-2-butyne (VI) or 2-butyne-1,4-diol bis-(benzene)sulfonate (VII) and NaFp at —78°C afforded primarily 1,4-bis(η^5-cy-

$FpCH_2C{\equiv}CCH_2Fp$

(I)



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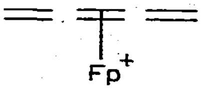

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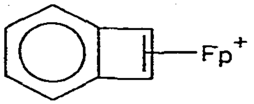

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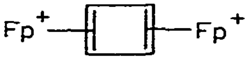
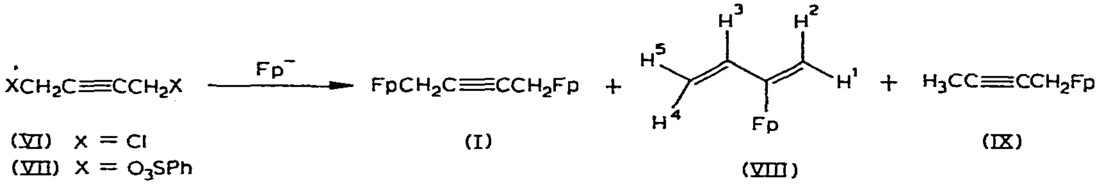


(V)

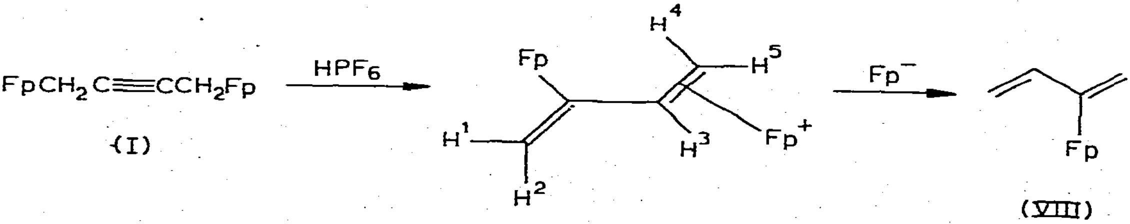
($Fp \equiv \eta^5$-$C_5H_5(CO)_2Fe$)

clopentadienyldicarbonyliron)-2-butyne (I) in 66 and 52% yields, respectively. In addition to I a 9% yield of the previously unreported 2-(η^5-cyclopentadienyldicarbonyliron)-1,3-butadiene (VIII) was obtained as a petroleum ether-soluble, deep orange oil. This compound was stable on activity III neutral alumina and could be short path distilled (10^{-3} mmHg) without extensive decomposition. Occasionally VIII was contaminated with variable small amounts of 1-(η^5-cyclopentadienyldicarbonyliron)-2-butyne (IX). These mononuclear complexes are readily separated by column chromatography on alumina.

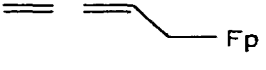


When treated with NaFp, 1,4-dibromo-2-butyne (X) afforded VIII and IX in 8% total yield but no detectable amounts of I were formed.

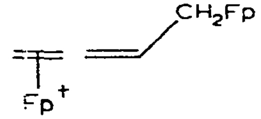
Compound VIII also was prepared by the reductive demetalation of XI which was prepared by protonation of I with hexafluorophosphoric acid in diethyl ether. The addition of butatriene to a tetrahydrofuran solution of FpH gave VIII and IX in 5% total yield.



The formulation of VIII as a 1,3-butadienyl rather than a 1,2-butadienyl complex XII is based primarily on its PMR spectrum. In particular, the part of the spectrum attributed to the butadiene ligand is typical of a 2-substituted 1,3-butadiene. Thus, the narrow multiplets observed at τ 4.22 and 4.80 were assigned to H^1 and H^2 although a definite assignment cannot be made. The resonance assigned to H^3, which shows small coupling between H^1, H^2 and H^3, is a doublet of doublets, $J_{3,4}$ 15 and $J_{3,5}$ 9.0 Hz. The resonance of H^5 is also a doublet of doublets ($J_{4,5}$ 2.5 Hz) centered at 5.08. The H^4 multiplet resonance, which is obscured by the cyclopentadienyl resonance (τ 5.24) when the spectrum is obtained on a carbon disulfide solution, is observable when benzene-d_6 is used as the solvent. In this solvent the cyclopentadienyl resonance is shifted to τ 5.80 whereas the butadienyl resonances are shifted downfield. The absence of a doublet resonance at ca. τ 8.0 also excluded XII as a possible structure.



(XII)



(XIII)

The product resulting from the protonation of I recently was reported to be the binuclear 1,2-butadienyl complex XIII on the basis of an IR spectrum only [4]. The PMR spectrum of this substance clearly supports the 1,3-butadienyl structure XI.

Thus the absence of a doublet resonance due to the methylene protons of XIII which would have been observed at ca. τ 8.0, and the observation of a pair of broad doublets ($J_{3,4}$ 14, $J_{3,5}$ 9 Hz) centered at τ 6.80 and 6.01, respectively, are consonant with structure XI. The H^1 and H^2 resonances are observed as broad singlets at τ 3.79 and 4.20. The H^3 resonance is a multiplet centered at τ 3.70. The PMR spectra of the butadienyl groups of VIII and XI are quite similar.

The primary mononuclear product of the reaction between VI, VII or X and NaFp in the presence of methanol was η^3-*E*-(1-carbomethoxy-1-methylpropenyl)-η^5-cyclopentadienylcarbonyliron (XIV) (Scheme 1). The assigned structure of XIV is supported by its spectral properties. Thus, the IR spectrum of XIV exhibits both a terminal carbonyl absorption at 1947 cm^{-1} and an acyl carbonyl absorption at 1692 cm^{-1}. In the PMR spectrum of XIV, the high field signal (τ 9.39) of the methyl protons is considerably higher than the analogous resonance (τ 8.92) of the η^3-butenyl complex XVIII * [5] despite the presence of the electron-withdrawing carbomethoxy group in XIV. This suggests that the methyl group is in the *anti* position and is shielded by the iron group.

The *anti* and *syn* protons, H^4 and H^5, are observed as a pair of multiplets ($J_{3,4}$ 12, $J_{3,5}$ 7.5, $J_{4,5}$ 2.4 Hz) centered at τ 6.84 and 8.76, respectively. The *geminal* coupling constant (2.4 Hz) is characteristic of the *exo* conformer XIV rather than of the *endo* conformer XVIII [6]. The multiplet resonance of the central proton is centered at τ 4.61.

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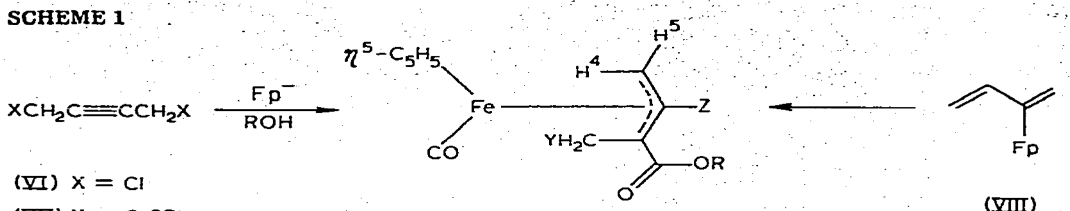
* The stereochemistry of XVIII has not been established but is probably the *exo*-*E* isomer.

SCHEME 1

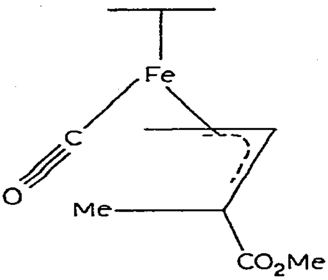


(VI) X = Cl
(VII) X = O_3SPh
(X) X = Br

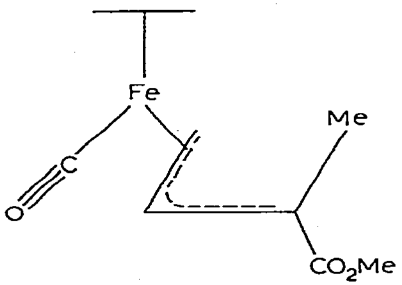
(VIII)

(XIV) Z = H^3, Y = H, R = Me
(XIV-*d*) Z = Y = D, R = Me
(XV) Z = H^3, Y = H, R = Et
(XVI) Z = H^3, Y = H, R = i-Pr

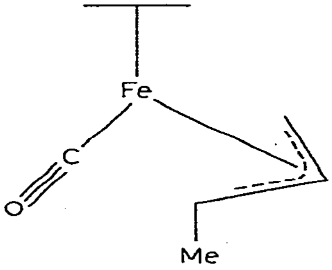
When methanol-OD was substituted for methanol, XIV-*d* was isolated. Incorporation of deuterium in the central position and methyl group was evidenced



(XIV)



(XVII)



(XVIII)

by the absence of a multiplet resonance at τ 4.61, the observation of the H^4 and H^5 resonances as broad singlets and methyl resonance as a broad two-proton singlet.

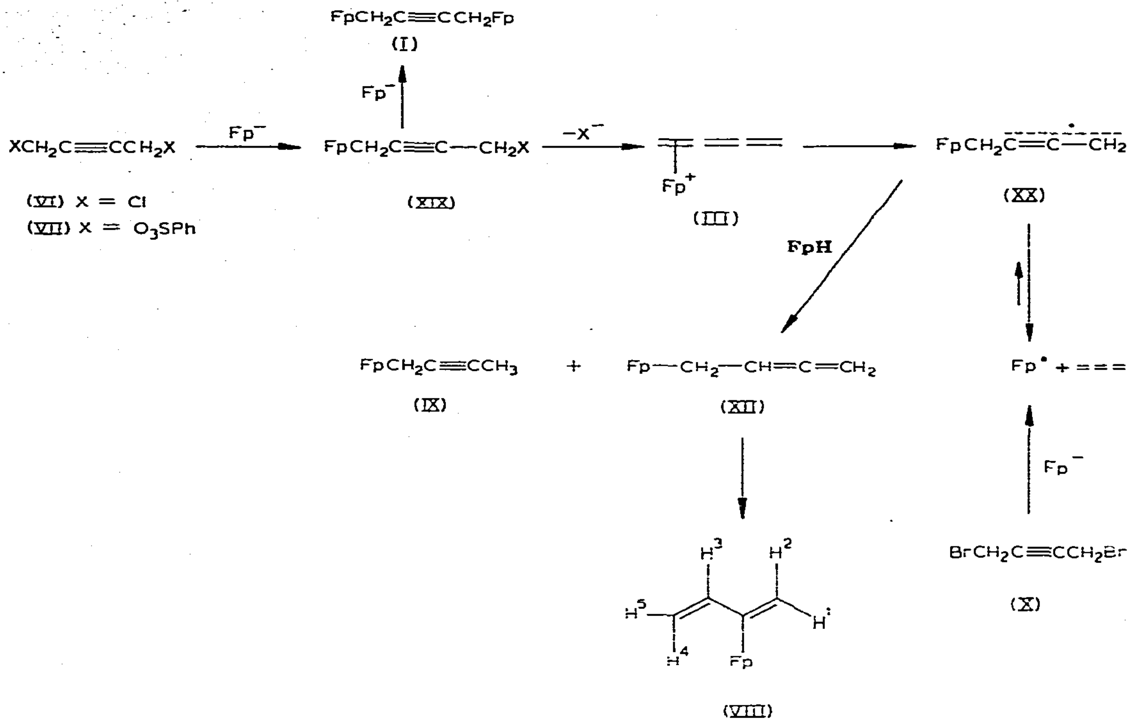
Although XIV was isolated in yields up to 40%, the carboethoxy complex XV was isolated in only 3% yield. Complex XV was characterized by its IR and PMR spectral properties which were in all respects comparable to those of XIV. Only a trace of the carboisopropoxy complex XVI was obtained. Although the yield of VIII and IX was increased to 31% when a large excess of t-butanol was added to the reaction mixture prior to the addition of VII, there was no evidence for a t-butoxy derivative analogous to XIV.

Both VIII and IX were converted to XIV by treatment with a mixture of NaFp and methanol or in the case of VIII by sodium methoxide in methanol. The binuclear complex I was recovered unchanged when treated with NaFp/methanol or methoxide/methanol mixtures.

Discussion

By analogy to the reductive metalation of 1,2-dhalobenzocyclobutenes [7], it is probable that the formation of the mononuclear complexes VIII and IX, from the butyne substrates VI, VII and X involves the interaction between the radical butatriene complex XX and FpH. Since VI and VII afford the binuclear complex I whereas VIII does not, it is likely that XX is formed via at least two

SCHEME 2



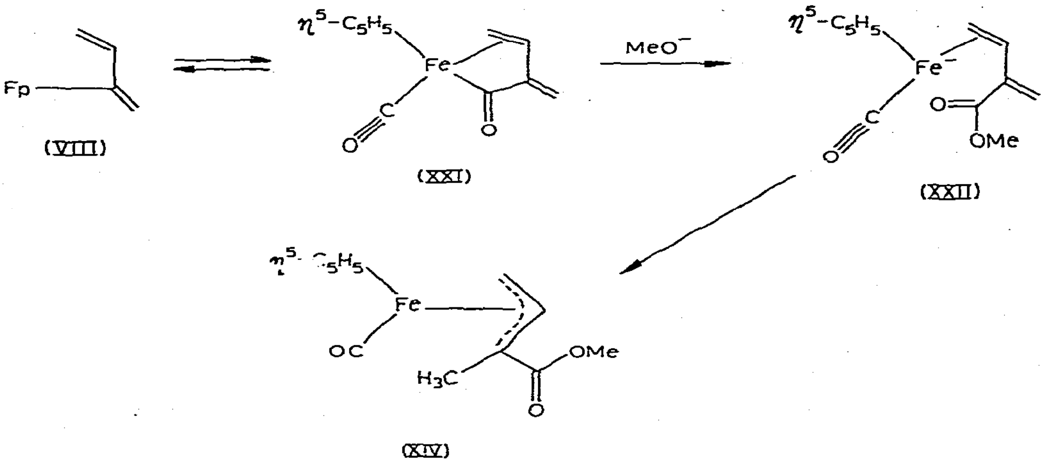
different reaction pathways. Thus VI and VII can be converted to XX as outlined in Scheme 2. The interception of XIX by Fp$^-$ would give I. Heterolytic dissociation of XX would generate the butatriene complex III which would be reduced to XX by Fp$^-$. In view of the affinity of Fp$^-$ for bromide [7,8], the direct debromination of X to butatriene is likely. Addition of Fp· to butatriene results in the formation of XX. Hydrogen abstraction from FpH by XX generates IX which is isolated occasionally and the 1,2-butadienyl complex XII which undergoes a 1,3-shift of the iron group to form VIII.

In support of these hypotheses, is the formation of VIII and IX when butatriene is bubbled through a tetrahydrofuran solution of FpH, and the dramatic increase in the yield of VIII and IX when VII is metallated in the presence of a high concentration of FpH.

1,3-Shifts of the Fp group are known [9] and usually afford the isomer in which the Fp group is attached to the least substituted carbon [10]. The proposed isomerization of XII to VIII can be reconciled with previous observations when it is realized that the iron—vinyl σ-bond is shorter and presumably stronger than the iron—alkyl σ-bond [11]. The attainment of the stronger iron—carbon σ-bond would at least partially supply the driving force for the rearrangement of XII to VIII. A similar argument concerning σ-bond energies can be advanced to account for the isolation of the cationic 1,3-butadienyl complex XI rather than the 1,2-butadienyl complex XIII when I is protonated. Furthermore the Fp$^+$—alkene bond strength of XI may be stronger than that of XIII since the first ionization potential of 1,3-butadiene is significantly smaller than that of allene [12].

The formation of the η^3-propenyl complexes was quite unexpected. η^1-Propargyl complexes are converted generally to η^3-(2-carboalkoxypropenyl) [13] complexes by treatment with alcohols although there is at least one example of the formation of a 1-carboalkoxypropenyl complex [14]. η^1-Propargyliron complexes have not been reported to enter into this type of reaction. The conversion of VIII to XIV is best envisioned as proceeding via the acyl complex XXI (Scheme 3) *. Addition of methoxide to the acyl carbonyl cleaves the iron—acyl bond and generates the anionic complex XXII, the protonation of which affords the η^3-propenyl complex XIV. From the data in hand the point at which *syn—anti* isomerization occurs to give the *E* isomers is not known.

SCHEME 3



The preference for the *E* (XIV) rather than *Z* isomer is probably due to steric factors. In the *Z* isomer, the carboalkoxy group must rotate out of the plane defined by the η^3-propenyl ligand in order to minimize the steric interaction between this group and the iron and the coordinated carbon monoxide. Hence, delocalization and the attendant stabilization is sterically inhibited. On the other hand in the *E* isomer (XIV) the carboalkoxy group is free to become coplanar with the η^3-propenyl group.

Experimental

General procedures

In general all reactions were carried out under a nitrogen atmosphere. Iron analyses were performed according to standard procedures.

Physical measurement

Infrared spectra were recorded on a Perkin—Elmer Model 180 spectrophotometer. PMR spectra were obtained on a Jeol C-60 High Resolution spectrometer using tetramethylsilane as an internal standard. Melting points are uncorrected.

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* Alkyliron complexes, when dissolved in coordinating solvents, have been shown to be in equilibrium with acyl complexes [15]. η^1-4-(1-Butenyl)-η^5-cyclopentadienyl dicarbonyliron rearranges to a stable complex analogous to XXI [16].

Materials

1,4-Dichloro-2-butyne (Aldrich), 2-butyne-1,4-diol (GAF), nitromethane-d_3 (Aldrich), chromatographic grade neutral alumina (Alfa—Ventron) were used as purchased. 1,4-Dibromo-2-butyne (Columbia) was distilled before use. 2-Butyne-1,4-diol bis(benzene)sulfonate and butatriene were prepared according to published procedures [17]. Tetrahydrofuran was routinely distilled from lithium aluminum hydride. All other solvents were reagent grade and were used without further purification.

Reaction between NaFp and 1,4-dichloro-2-butyne, 2-butyn-1,4-diol bis(benzene)sulfonate, or 1,4-dibromo-2-butyne

1,4-Dichloro-2-butyne, 1.23 g (10 mmol), was added to a solution of NaFp (25 mmol) in 45 ml of tetrahydrofuran at −78°C. After the mixture had been warmed to 24°C and 100 ml of petroleum ether (30—60°C) added to it, the resulting mixture was filtered through 4 cm of Celite. The residue resulting from the rotary evaporation of the filtrate was extracted with petroleum ether. The petroleum ether insoluble material (3.87 g) was identified via PMR spectroscopy as a 1 : 2 mixture of Fp_2 and I. (A 66% yield of I based on the amount of 1,4-dichloro-2-butyne used.)

The petroleum ether extract was rotary evaporated and the resulting residue was re-extracted with petroleum ether. Rotary evaporation and subsequent short path distillation (~70°C/10^{-3} mmHg) afforded 2-(η^5-cyclopentadienyldicarbonyliron)-1,3-butadiene (VIII) as a deep orange oil. PMR (CS_2): τ 3.50 (m, 1, H^3, $J_{3,4}$ 15.0, $J_{3,5}$ 9.0 Hz), 4.22 (m, 1, H^1 or H^2), 4.80 (m, 1, H^1 or H^2), 5.08 (m, 1, H^5, $J_{4,5}$ 2.4 Hz), 5.24 (s, 5, C_5H_5), 5.24 (m, 1, H^4); IR (neat): 2003, 1949 (C≡O) cm^{-1}; mass spectrum: *P* at *m/e* 230. (Found: Fe, 24.01. $C_{11}H_{10}O_2Fe$ calcd.: Fe, 24.28%.)

In similar experiments 2-butyne-1,4-diol bis(benzene)sulfonate gave a 52% yield of I and an 8% yield of VIII. 1,4-Dibromo-2-butyne gave a 9% yield of VIII only.

Reaction between NaFp and 1,4-dichloro-2-butyne, 2-butyn-1,4-diol bis(benzene)sulfonate or 1,4-dibromo-2-butyne

(a) In the presence of t-butanol. 1,4-Dichloro-2-butyne, 3.7 g (30 mmol) was added to a solution of NaFp (75 mmol) in 140 ml of tetrahydrofuran and t-butanol (29 g, 0.375 mol) at −78°C. After the mixture had been warmed to 24°C, 100 ml of petroleum ether was added to it. The resulting mixture was filtered through 4 cm of Celite. The residue resulting from the rotary evaporation of the filtrate was extracted with petroleum ether. The residue, 11.4 g, was identified by PMR spectroscopy as a 3 : 4 mixture of Fp_2 and I (57% yield). The extract was percolated through 4 cm of alumina, eluting with petroleum ether. Rotary evaporation of the orange elute gave 0.72 g (10%) of a dark red oil which was identified by PMR spectroscopy as VIII

In a similar experiment 2-butyne-1,4-diol bis(benzene)sulfonate gave, in addition to Fp_2 and I, a 30% yield of VIII. 1,4-Dibromo-2-butyne gave a 1 : 1 mixture of VIII and IX in a combined yield of 31%.

(b) In the presence of methanol. 1,4-Dichloro-2-butyne, 1.23 g (10 mmol) was added to a solution of NaFp (25 mmol) in 45 ml of tetrahydrofuran and

methanol (12 g, 0.375 mol) at −78°C. Petroleum ether, 100 ml, was added to the mixture after it had been warmed to 24°C. This mixture was filtered through 4 cm of Celite. The residue resulting from the rotary evaporation of the filtrate was extracted with petroleum ether. The petroleum ether insoluble residue (3.94 g) was identified by PMR spectroscopy as a 5 : 3 mixture of Fp_2 and I (43% yield). The residue (0.41 g) resulting from the rotary evaporation of the petroleum ether extract was shown by PMR spectroscopy to be a mixture of VIII and the η^3-propenyl complex XIV. Chromatography of this mixture on neutral activity III alumina (2 × 30 cm) eluting with 1 : 4 benzene/petroleum ether afforded 0.27 g (10%) of XIV. A complete characterization of XIV is given later. The use of methanol-OD in this experiment afforded XIV-*d*: PMR (CS_2): τ 5.63 (br, s, 5, Cp), 6.33 (s, 3, Me), 6.86 (br s, 1, H^5), 8.73 (br s, 1, H^4), 9.38 (br s, 2, CH_2D).

In similar experiments 2-butyn-1,4-diol bis(benzene)sulfonate gave a 9% and 44% yield of VIII and XIV.

Protonation of 1,4-bis(η^5-cyclopentadienyldicarbonyliron)-2-butyne (I) by hexafluorophosphoric acid

A solution of 2 ml of 65% aqueous hexafluorophosphoric acid in 20 ml of diethyl ether was added to a solution of I, 0.50 g (0.23 mmol) in 100 ml of diethyl ether. The precipitate, which formed immediately, was collected by filtration and washed with diethyl ether. The brown solid was dissolved in methylene chloride and reprecipitated by the addition of diethyl ether to yield 0.54 g (80%) of η^2-3,4-[2-(η^5-cyclopentadienyldicarbonyliron)-1,3-butadiene]-η^5-cyclopentadienyldicarbonyliron hexafluorophosphate (XI): PMR (CD_3NO_2): τ 3.70 (m, 1, H^3, $J_{3,4}$ 15, $J_{3,5}$ 8 Hz), 3.79 (s, 1, H^1), 4.21 (s, 1, H^2), 4.33 (s, 5, C_5H_5), 4.87 (s, 5, C_5H_5), 6.01 (d, 1, H^4), 6.80 (d, 1, H^5); IR (Nujol): 2065, 2037, 2011, 1971 ($C\equiv O$) cm^{-1}. (Found: Fe, 20.03. $C_{18}H_{15}O_4Fe_2P$ calcd.: Fe, 20.29%.

Reductive demetalation of XI

Complex XI, 0.35 g (0.63 mmol), was added to a rapidly stirred mixture of 0.25 g of Fp_2 in 10 ml of tetrahydrofuran and 200 g of a 0.25% sodium amalgam. After 0.5 h, the tetrahydrofuran component of the mixture was rotary evaporated and the residue dissolved in petroleum ether and chromatographed on neutral activity III alumina (2 × 30 cm), eluting with a 1 : 1 benzene/petroleum ether mixture. The residue resulting from the rotary evaporation of the yellow eluate gave 0.10 g (69%) of a dark orange oil that was identified as VIII by PMR spectroscopy.

Reaction between VIII and methanol and in the presence of NaFp or sodium methoxide

A solution of NaFp (1.1 mmol) in 2 ml of tetrahydrofuran was added to a solution of VIII, 0.26 g (1.1 mmol) in 1 ml of methanol. After this mixture had been stirred for 10 h, it was chromatographed on neutral, activity III alumina (2 × 30 cm). Elution with 1 : 1 benzene/petroleum ether afforded a mixture of Fp_2 and XIV. Extraction of this material with petroleum ether gave, after rotary evaporation of the extract and crystallization of residue from petroleum ether, η^3-2,3,4-[*E*-2-carbomethoxybutenyl]-η^5-cyclopentadienylcarbonyliron, 0.13 g (45%) as a dark red solid: M.p. 58—60°C PMR (CS_2) τ 4.62 (m, 1, H^3, $J_{3,4}$ 12,

$J_{3,5}$ 7.5 Hz), 5.63 (s, 5, C_5H_5), 6.32 (s, 3, OCH_3), 6.84 (m, 1, H^5, $J_{4,5}$ 2.4 Hz), 8.76 (m, 1, H^4), 9.39 (s, 3, CH_3); IR (hexachlorobutadiene): 1956 (C≡O), 1696 (C=O) cm^{-1}; mass spectrum: *P*-28 at *m/e* 234. (Found: Fe, 21.35. $C_{12}H_{14}O_3Fe$ calcd.: Fe, 21.37%.)

In a similar reaction that utilized sodium methoxide rather than NaFp, an 81% yield of XIV was obtained.

Reaction between NaFp and IX in the presence of methanol

Complex IX was added to a solution prepared by mixing 5 ml of methanol with a 5 ml solution of 0.8 *M* NaFp in tetrahydrofuran. After 18 h, a PMR spectrum of the residue resulting from the rotary evaporation of the solvents showed the presence of Fp_2 and XIV. This material was not further purified.

Reaction between I and sodium methoxide in methanol

Complex I, 0.406 g (1 mmol) and 10 ml of tetrahydrofuran were added to 5 ml of a 0.25 *M* solution of sodium methoxide in methanol. After the mixture had been stirred for 12 h, it was chromatographed on neutral activity III alumina (2 × 32 cm) without further workup. Elution with 1 : 1 benzene/petroleum ether and subsequent rotary evaporation of the yellow eluate led to the recovery of I, 0.383 g (90%).

Reaction between NaFp and 1,4-dibromo-2-butyne in the presence of ethanol or isopropanol

1,4-Dibromo-2-butyne, 2.12 g (10 mmol), was added to a solution of NaFp (25 mmol) in 45 ml of tetrahydrofuran and 22.5 (0.375 mol) of ethanol at —78°C. The solution was warmed to 24°C and 100 ml of petroleum ether was added. This mixture was filtered through Celite. The residue resulting from the rotary evaporation of the filtrate was chromatographed on neutral activity III alumina (4 × 35 cm) eluting with 1 : 4 benzene/petroleum ether. Rotary evaporation of the yellow eluates gave VIII, 0.07 g (3%), IX, 0.07 g (3%) and 0.07 g (3%) of η^3-2,3,4-(*E*-2-carboethoxybutenyl)-η^5-cyclopentadienylcarbonyliron (XV). PMR (CS_2): τ 4.70 (m, 1, H^3, $J_{3,4}$ 12, $J_{3,5}$ 7.5 Hz), 5.68 (s, 5, Cp), 5.90 (q, 2, CH_2, *J* 7.5 Hz), 6.90 (m, 1, H^5, $J_{4,5}$ 2.2 Hz), 8.75 (m, 5, H^4 and CH_3), 9.41 (s, 3, CH_3); IR (Nujol): 1951 (C≡O), 1686 (C=O) cm^{-1}.

A similar procedure which used isopropanol in place of ethanol afforded VIII (15%), IX (5%) and mixed in with the Fp_2 a small amount of XVI as evidenced by an absorption at 1688 cm^{-1} in the IR spectrum of this substance.

Reaction between butatriene and FpH

Butatriene was prepared from 1,4-dibromo-2-butyne, 8.5 g, (40 mmol) using acetonitrile as the solvent according to the procedure of Schubert, Liddicoet and Lanka [17] and was trapped at —196°C. In a stream of nitrogen, the butatriene was swept into a solution of hydrido-η^5-cyclopentadienyldicarbonyliron (20 mmol) in 35 ml of tetrahydrofuran. The residue resulting from the rotary evaporation of the reaction mixture was extracted with petroleum ether. The petroleum ether extract was percolated through neutral activity III alumina. Rotary evaporation of the yellow eluate afforded 1.2 g (13%) of a brown oil that was identified by PMR spectroscopy as a mixture of VIII and IX.

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