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ORGANOMETALLIC SYNTHONS

THE WITTIG REACTION OF TRICARBONYL[(3,4,5,6-η)-1,3,5-CYCLOHEPTATRIENE-1-CARBOXALDEHYDE]IRON

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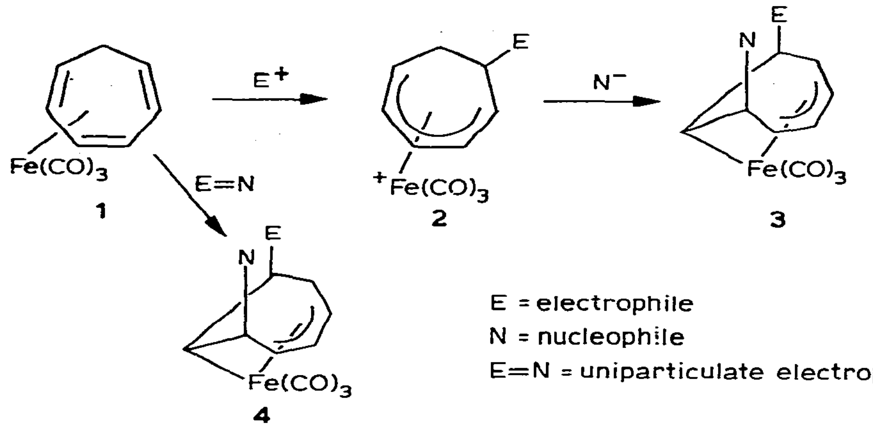
Summary

The utility of the Wittig reaction in synthesis of organometallic compounds is exemplified by its application to the preparation of a series of tetraene—$Fe(CO)_3$ complexes from aldehyde 9 and a variety of triphenylphosphoranes. Reaction of 9 with triphenylmethylene phosphorane afforded unexpectedly a 1/1 isomeric mixture of 8-methylheptafulveneiron tricarbonyl (12), probably via a (1,9) hydrogen shift of intermediate 11. Triphenylbenzylidene phosphorane condensed with 9 to give the *cis* and *trans* isomers of cycloheptatrienyl—styrene complex 14. The triphenylphosphoranes of carbomethoxymethylene, carboethoxymethylene and cyanomethylene react with 9 yielding the appropriate *trans* condensation products exclusively. Tetracyanoethylene (TCNE) and *N*-phenyltriazolinedione (NPTD) readily react periselectively with the unrearranged Wittig reaction products at the free diene moiety, affording the corresponding 4 + 2 cycloadducts. Heptafulvene complex 12, upon reaction with TCNE, gave the 8 + 2 cycloadduct 26.

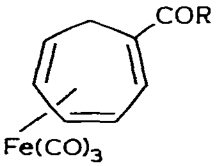
Introduction

The reactions of organic ligands in transition metal organometallic complexes has become a focal point of attention for an increasing number of synthetic organic chemists. Recently, the reactions of the free double bond in cycloheptatriene complexes (1) toward electrophilic attack have been widely explored (for a general review see ref. 1). It has been recognized that because of the large number of new synthetic possibilities opened by electrophilic substitution [2], addition [3] and cycloaddition [4—6] reactions, an interrelationship can be established between the tricarbonyliron complexes of cyclic η^4-hexatriene (1), η^5-pentadienylium (2) and σ,π-allylic (3 and 4) systems.



E = electrophile
N = nucleophile
E=N = uniparticulate electrophile

While the chemistry of a free double bond in organic complexes has received much attention, other functional groups have not been as widely examined. Among the functional groups which appear to be promising, the carbonyl function seems the most fruitful since it offers a site for both electrophilic and nucleophilic attack. Nucleophilic reactions of Grignard reagents and hydride ion with acylcycloheptatriene complexes (5) have been previously described by Lewis [7], while the Wittig reaction has been employed only for synthesis of open chain polyeneiron tricarbonyl complexes [8] (6 and 7) and in preparation of mono- and 1,2-dialkenylcyclobutadiene complexes (8) [9,10]. In this paper we report on the Wittig reaction of aldehyde complex 9 with a series of triphenylphosphoranes (10a—e) and describe the properties and cycloaddition reactions of the product tetraene complexes.



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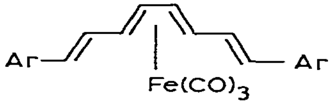

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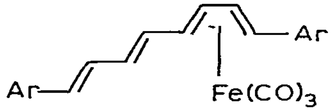

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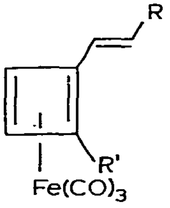

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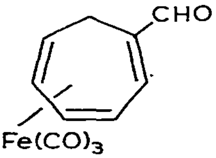


9

$Ph_3P{=}CHR$

10, a. R = H; b. R = Ph; c. R = COOMe; d. R = COOEt; e. R = CN

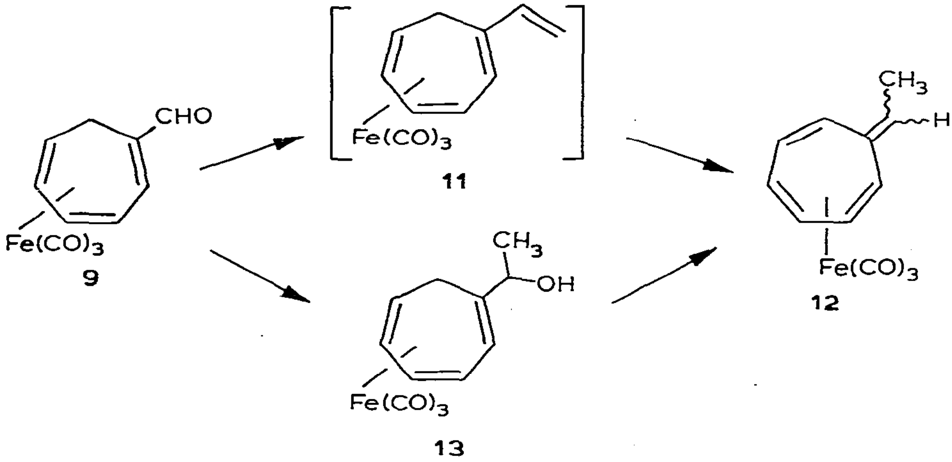
Results and discussion

The Wittig reaction

Treatment of aldehyde 9 [2] with methylenetriphenyl phosphorane (10a) in

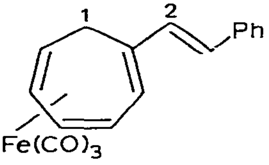
THF solution at 0°C led, after chromatography, to the isolation of 8-methylheptafulvene—$Fe(CO)_3$ complex **12**. The ^1H NMR spectrum of **12** (in $CDCl_3$) closely resembled that of **12** previously obtained by dehydration of carbinol **13** [7], revealing a 1/1 isomeric mixture, as indicated by a pair of vinylic methyl doublets ($J = 7$ Hz) at δ 1.56 and 1.68, each of relative intensity 1.5.

Formation of **12** may be rationalized by assuming, in an analogy with other condensations in this series (vide infra), the initial production of tetraene complex **11**, which subsequently undergoes a formal (1,9) hydrogen sigmatropic shift. An analogous hydrogen abstraction from the methylene carbon is observed in the dehydration of **13** under acid conditions [7].

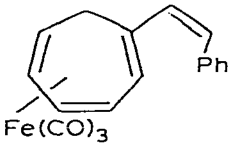


When **9** was treated with benzylidenetriphenyl phosphorane (**10b**) as above, it was converted into a mixture of isomers, **14c** and **14t**. The isomers were separated by column chromatography affording the *cis* isomer **14c** as an orange oil and the crystalline *trans* isomer **14t**, in 1/3 ratio. The ^1H NMR spectra (Table 1) of the two isomers, together with other spectroscopic data, are consistent with the structural assignment. In particular, we note a distinct difference in position and coupling constants of the olefinic protons H(8) and H(9) of the two isomers. These comprise an AB quartet centered at δ 5.92 (J_{89} = 12 Hz) for *cis*-**14c** and at δ 6.44 (J_{89} = 16 Hz) for *trans*-**14t** [11].

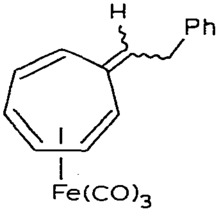
Unlike **11**, both **14c** and **14t** are stable molecules which do not rearrange to the corresponding heptafulvene **15**. This stability may be attributed to the phenyl-extended conjugation of the tetraene system, which would be lost upon rearrangement to the heptafulvene.



14t



14c



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The reaction of **9** with carbomethoxymethylene phosphorane (**10c**) [12] in

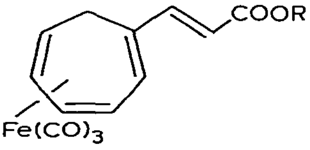
TABLE 1

^{1}H NMR DATA FOR THE WITTIG CONDENSATION PRODUCTS [a]

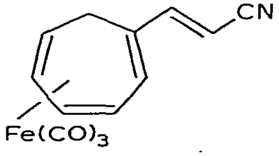
Compound	2H(1)	H(2)	H(3), H(4)	H(5)	H(6)	H(8)	H(9)	Functional group
14*t*	2.60	3.49	5.35	3.19	6.02	6.44 [b]		7.3 (5H, m)
14c	2.24	3.20	5.21	3.04	5.92	6.04 [b]		7.2 (5H, m)
17	2.45	3.45	5.38	3.12	6.32	7.06	5.63	3.71 (3H, s)
18	2.43	3.45	5.38	3.12	6.32	7.04	5.64	1.26 (3H, t, J = 7 Hz) 4.16 (2H, q)
19	2.36	3.44	5.40	3.09	6.28	6.72	5.01	
Multiplicity	ABX [c]	m	m	ddd	d	d	d	
Coupling constants	J_{11} = 20 Hz J_{12} = 5 Hz		J_{35} = 3 Hz J_{45} = 6 Hz	J_{56} = 8 Hz		J_{89} (*trans*) = 16 Hz J_{89} (*cis*) = 12 Hz		

[a] 100 MHz, $CDCl_3$, δ. [b] Center of an AB quartet. [c] Broad AB quartet furtherly splitted by H(2) at the lower field pair.

CH_2Cl_2 solution at room temperature took place very slowly, giving exclusively the stable *trans*-methyl ester **17**, as an orange crystalline compound, in 50% yield. The 16 Hz coupling constant between the exocyclic olefinic protons (H(8), H(9)) in the ^1H NMR spectrum of **17** established the *trans* configuration (Table 1). The *trans*-ethyl ester **18** was similarly obtained, as an uncrystallizable orange oil, by reaction of **9** with carboethoxymethylene phosphorane (**10d**) [12].



17, R = Me
18, R = Et



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Condensation of **9** with cyanomethylenetriphenyl phosphorane (**10e**) [13] required somewhat higher temperatures (40—50° C, benzene solution) and gave a moderate yield (20%) of the *trans*-nitrile **19** as the only isolated product. Higher reaction temperature led to extensive decomposition. The ^1H NMR spectrum (Table 1) again indicated the *trans* configuration. We also note that electron-withdrawing substituents prevent the rearrangement of the tetraene systems to the corresponding heptafulvene complexes, in analogy to phenyl conjugation observed in **15**.

Another clear confirmation for the presence of a linearly conjugated tetraenic system comes from the ^{13}C NMR spectra (Table 2). Carbons of the cycloheptatrienyl—$Fe(CO)_3$ moiety give resonances at positions similar to that of parent complex **1** and aldehyde **9**. In addition, the positions of C(8) and C(9) olefinic carbons strongly depend on the electron-withdrawing capabilities of the functional group attached to C(9).

The reactivity of aldehyde **9** with the various phosphoranes (**10a—e**) decreases in the order Ph > COOR > CN. It reflects the reduction in nucleophilicity of the phosphoranes due to increase of delocalization of the electron density on the ylid carbanion [14]. Indeed, aldehyde **9** failed to react under similar conditions with the more stable phosphoranes **20** and **21**.

$Ph_3P{=}CHCHO$ $\quad$ $Ph_3P{=}CHCH{=}CHCOOEt$

20 $\quad$ **21**

Cycloaddition reactions

The Wittig reaction of **9** extended the cyclohetatriene complex system by another double bond, introducing a free diene into the organic ligand now vulnerable to 4 + 2 cycloaddition (Diels—Alder) reaction with dienophiles. These reactions should a priori compete with the 1,3-cycloaddition usually observed in cyclic triene complexes [4,6,15]. It was previously shown [16] that the 4 + 2 cycloaddition predominates over the 1,3-σ,π-allylic cycloaddition in dicycloheptatrienyl complex **22**. We hoped to observe reversed periselectivity in the tetraenic complexes (**14,17—19**), since the diene system is flanked by deactivating groups such as phenyl (in **14**) and electron-withdrawing groups

TABLE 2

^{13}C NMR DATA FOR DERIVATIVES OF CYCLOHEPTATRIENE COMPLEXES [a]

Compound	C(1)	C(2), C(5)		C(3), C(4)		C(6)	C(7)	C(8)	C(9)	Ligand CO	Functional group
1 [b]	30.70	55.92	60.26	88.28	93.39	128.41	125.43			211.32	
9	26.85	53.85	60.42	89.23	94.61	154.0	136.90	192.33		209.72	
14t [c]	29.48	56.92	59.19	88.18	92.82	132.74	134.27	124.38	131.64	210.75	127.0, 126.15 128.65, 137.81 (aromatic)
17	29.05	55.48	48.95	88.63	93.69	141.41	132.26	147.29	113.09	210.14	51.37 (methyl)
18	29.04	55.53	59.01	88.55	93.67	141.23	132.31	147.02	113.51	210.19	14.34 (methyl) 60.12 (methylene)
19	28.31	54.84	58.82	88.67	90.63	142.69	131.64	152.40	94.05	209.84	119.06 (CN)

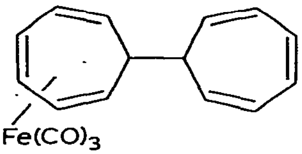
[a] $CDCl_3$, ppm relative to TMS. [b] Cf. ref. 20. [c] Assignment of vinylic and aromatic carbons unconfirmed.

TABLE 3

^{1}H NMR DATA FOR TCNE ADDUCTS (23) [a]

Adduct 23	2H(1)	H(2), H(5)	H(3), H(4)	H(6)	H(8)	H(9)	Functional group
a, R = Ph	2.92	3.17	5.53	3.55	5.47	4.19	7.46 (5H, m)
b, R = COOMe	2.88	3.08	5.45	3.50	5.66	3.93 [b]	3.93 (3H, s)
c, R = COOEt	2.89	3.10	5.45	3.51	5.67	3.94	1.42 (3H, t, J = 7 Hz) 4.42 (2H, q)
Multiplicity	m	m	m	bs [c]	bs	bs	

[a] 100 MHz, $CDCl_3$, δ. [b] Covered by the methyl signal. [c] Broad singlet.



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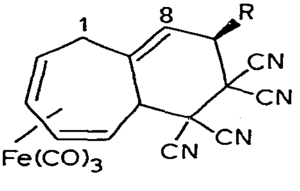
(17—19). To this end we chose the two powerful dienophiles, tetracyanoethylene (TCNE) and *N*-phenyltriazolinedione (NPTD), which are known to undergo 1,3- as well as 1,4-cycloadditions. Both react rapidly with the tetraene complexes to give regiospecifically the 4 + 2 series of adducts 23 and 24 respectively. No other products could be isolated. All but adduct 23d are stable crystalline compounds, best characterized by ^1H NMR spectroscopy (Tables 3 and 4). 23d was relatively stable in its crystalline form but decomposed readily in solution.



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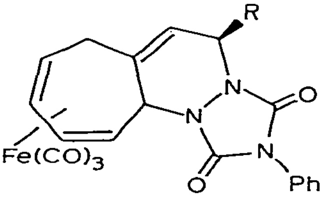

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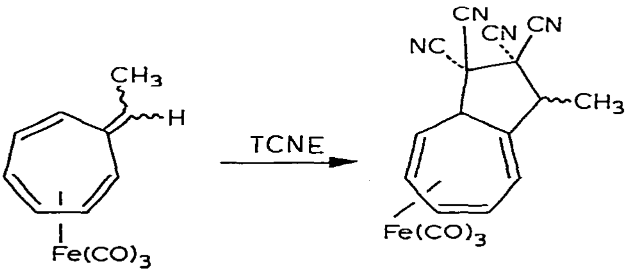
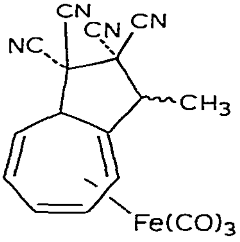
a. R = Ph
b. R = COOMe
c. R = COOEt
d. R = CN

The heptafulvene complex 12 also reacted readily with TCNE in CH_2Cl_2 solution affording a mixture of the two isomeric 8 + 2 adducts 26, which could not be separated. The ^1H NMR spectrum of 26 is characterized by a pair of methyl doublets ($J = 7$ Hz) at δ 1.37 and 1.47 (ca. 1/3 ratio) and by a series of four multiplets at δ 2.80—3.50 (3 H, two allyls and one outer complex), 3.70 (1 H, outer complex), 5.66 (2 H, inner complex) and 5.94 (1 H, vinyl).



12 **26**



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In principle, the formation of another 8 + 2 adduct 27, might be expected. However, isolation of only 26 indicates a highly stereospecific translocation of the $Fe(CO)_3$ moiety during cycloaddition. A recent communication by McArdle [17] reported similarly the stereospecific 8 + 2 cycloaddition of 8-arylheptafulvene complexes with TCNE. Finally, we note that while $Fe(CO)_3$ complexation did not alter the periselectivity of the cycloaddition as compared to noncomplexed heptafulvenes [18], the two step electrophilic addition (e.g. of HCN) to the parent heptafulvene complex took another course, affording a σ,π-allyl—$Fe(CO)_3$ complex exclusively [19].

TABLE 4

^{1}H NMR DATA FOR NPTD ADDUCTS (24)[a]

Adduct 24	2H(1)	H(2)	H(3), H(4)	H(5)	H(6)	H(8)	H(9)	N—Ph	Functional group
a, R = Ph	2.82	3.04	5.37	3.55	4.90	5.52	5.22	7.40	(10H, m)
b, R = COOMe	2.81	3.01	5.36	3.83[b]	4.84	5.57	4.87	7.45	3.83 (3H, s)
c, R = COOEt	2.80	3.03	5.31	3.80	4.80	5.55	4.87	7.42	1.30 (3H, t, J = 7 Hz) 4.27 (2H, q)
d, R = CN	2.82	3.08	5.42	3.55	4.80	5.42[c]	5.00	7.50	
Multiplicity	m	m	m	bd	bs	bd	m	m	
Coupling				J_{45} = 7 Hz		J_{89} = 6 Hz			

[a] 60 or 100 MHz, $CDCl_3$, δ. [b] Covered by methyl singlet. [c] Together with H(3), H(4) multiplet.

Experimental

General

^1H and ^{13}C NMR spectra were recorded on Varian Model HA-100 spectrometer and Varian Model CFT-20 instruments, with TMS as internal standard. IR spectra were recorded on a Perkin—Elmer Model-257 spectrometer. Mass spectra were determined with a Hitachi—Perkin—Elmer Model RMU-6 spectrometer at an ionizing energy of 70 eV. Analyses were performed at the Analytical Laboratories, Engelskirchen, G.F.R. All reactions were conducted under nitrogen.

Wittig reactions

8-Methylheptafulveneiron tricarbonyl (12) [7]. To a THF solution (150 ml) of triphenylmethylene phosphorane, prepared from 2.5 g (7 mmol) of triphenylmethylphosphonium bromide and 3.7 ml (6 mmol) 20% BuLi in hexane, at 0° C, was added a solution of 1.3 g (5 mmol) of 9 in 20 ml of dry THF. After magnetic stirring for 2 h the mixture was quenched with water (500 ml) and extracted with diethyl ether (3 X 200 ml). The combined ethereal solution was washed with water and dried over $MgSO_4$. Removal of the solvent under vacuum gave a brown oil which was chromatographed on silica gel. Elution with hexane afforded 247 mg (19%) of a yellow oil whose spectroscopic data match those of 12 prepared by Lewis et al. [7]. ^1H NMR ($CDCl_3$) δ 1.56 ($1\frac{1}{2}$ H, d, $J = 7$ Hz), 1.67 ($1\frac{1}{2}$ H, d, $J = 7$ Hz), 3.02 (1 H, m), 3.71 ($\frac{1}{2}$ H, d, $J = 8$ Hz), 3.90 ($\frac{1}{2}$ H, d, $J = 8$ Hz), 5.40 (4 H, m), 5.74 (1 H, d, $J = 5$ Hz).

cis- and trans-Tricarbonyl[2-(3,4,5,6-η)-1,3,5-cycloheptatrien-1-yl]styrene]-iron (14c and 14t). To a THF solution (200 ml) of triphenylbenzylidene phosphorane, prepared from 3 g (7 mmol) of benzyltriphenyl phosphonium bromide and 3.7 ml (6 mmol) 20% BuLi in hexane, at 0° C, was added 1.3 g (5 mmol) of 9 in dry THF (30 ml). After magnetic stirring for 4 h the mixture was quenched with water (600 ml) and extracted with diethyl ether (4 X 100 ml). The combined ethereal solution was washed with H_2O and dried over $MgSO_4$. Removal of the solvent under vacuum gave a brown oil which was chromatogrpahed on silica gel. Elution with hexane afforded first 0.13 g (8%) of the *cis* isomer 14c as a yellow oil. IR (neat) 2045, 1990, 1970 cm^{-1}. For ^1H and ^{13}C NMR, see Tables 1 and 2.

The crystalline *trans* isomer 14t, eluted second, was recrystallized from hexane to yield 0.40 g (24%) of orange-yellow crystals, m.p. 143° C; m/e 306 (M — CO), 278, 250, 194; IR (Nujol) 2050, 1990, 1975 cm^{-1}. Anal.: Found: C, 64.56; H, 4.10. $C_{18}H_{14}FeO_3$ calcd.: C, 64.67, H, 4.19%. For ^1H and ^{13}C NMR spectra see Tables 1 and 2.

Ethyl ester, 18. Ethyl ester was obtained as above from 1.3 g (5 mmol) of 9 and 2.1 g (6 mmol) of carboethoxymethylenetriphenyl phosphorane gave, after chromatography, 0.6 g (37%) of a yellow oil which could not be crystallized; m/e 330 (M), 302, 274, 244; IR (neat) 2045, 1990, 1950, 1690 cm^{-1}. For ^1H and ^{13}C NMR spectra see Tables 1 and 2.

Tricarbonyl[3-trans-[(3,4,5,6-η)-1,3,5-cycloheptatrien-1-yl]-2-propenitrile]-iron (19). A benzene solution (50 ml) of 1.3 g (5 mmol) of 9 and 1.8 g (6 mmol) of cyanomethylenetriphenyl phosphorane was kept at 40—50° C for

1 month. Removal of solvent under reduced pressure gave a brown oil which was chromatographed on silica gel. Elution with hexane gave 0.31 mg (21%) of orange crystals, m.p. 106°C (from hexane); *m/e* 255 (M — CO), 277, 199, 177, 143; IR (Nujol) 2204, 2025, 1990, 1960 cm^{-1}. Anal.: Found: C, 55.02, H, 3.13; N, 4.92. $C_{13}H_9FeNO_3$ calcd.: C, 55.12; H, 3.18; N, 4.95%. For ^1H and ^{13}C NMR spectra see Tables 1 and 2.

Reactions with TCNE

General procedure. Adducts were obtained by mixing equimolar amounts of freshly sublimed TCNE and the corresponding tetraene complex in CH_2Cl_2 and leaving the solution for 1 h at room temperature. Removal of solvent under reduced pressure was followed by recrystallization from CH_2Cl_2/hexane.

Adduct 26 was obtained as a mixture of isomers in 50% yield, m.p. 139°C (dec.); *m/e* 386 (M), 358, 330, 302; IR (Nujol) 2025, 1990, 1985 cm^{-1}. Anal.: Found: C, 56.07; H, 2.47; N, 14.5. $C_{18}H_{10}FeN_4O_3$ calcd.: C, 55.9; H, 2.59; N, 14.5%. ^1H NMR ($CDCl_3$), δ: 1.37(d), 1.47(d) (3 H, $J = 7$ Hz), 2.80—3.50 (3 H, m), 3.70 (1 H, m), 5.66 (2 H, m), 5.94 (1 H, m).

Adduct 23a was obtained in 70% yield, m.p. 167°C (dec.); *m/e* 306 (M — TCNE — CO), 278, 250; IR (Nujol) 2025, 1992, 1970 cm^{-1}. Anal.: Found: C, 62.21; H, 3.14; N, 12.27. $C_{24}H_{14}FeN_4O_3$ calcd.: C, 62.33; H, 3.03; N, 12.12%. For ^1H NMR spectrum see Table 3.

Adduct 23b was obtained in 58% yield, m.p. 146°C (dec.); *m/e* 444 (M), 416, 388, 360; IR (Nujol) 2032, 1995, 1965 cm^{-1}. Anal.: Found: C, 54.24; H, 2.80; N, 12.71. $C_{20}H_{12}FeN_4O_5$ calcd.: C, 54.05; H, 2.70; N, 12.61%. For ^1H NMR spectrum see Table 3.

Adduct 23c was obtained in 49% yield, m.p. 150°C (dec.); Anal.: Found: C, 54.94; H, 2.99; N, 12.35. $C_{21}H_{14}FeN_4O_5$ calcd.: C, 55.02; H, 3.05; N, 12.22%. For ^1H NMR spectrum see Table 3.

Adduct 23d was obtained in 44% yield, m.p. 138°C (dec.); ^1H NMR (acetone-d_6), δ 3.0—3.4 (4 H, m), 3.85 (1 H, bs), 4.4 (1 H, bs), 5.5—6.0 (3 H, m). Anal.: Found: C, 55.31; H, 2.10; N, 17.17. $C_{19}H_9FeN_5O_3$ calcd.: C, 55.47; H, 2.19; N, 17.03%. This compound decomposed rapidly in acetone solution.

Reactions with NPTD

General procedure. A solution of 1 mmol of tetraene complex in 10 ml CH_2Cl_2 was treated with a solution of 1 mmol of freshly sublimed NPTD in CH_2Cl_2, at room temperature. The discharge of the color of NPTD was very rapid. The solution was treated with activated charcoal, filtered and concentrated under reduced pressure. The residue was recrystallized from CH_2Cl_2/hexane.

Adduct 24a was obtained in 26% yield, m.p. 205°C (dec.); IR (Nujol), 2040, 1980, 1953, 1760, 1704 cm^{-1}. Anal.: Found: C, 61.13; H, 3.88; N, 8.28. $C_{26}H_{19}FeN_3O_5$ calcd.: C, 61.30; H, 3.74; N, 8.26%. For ^1H NMR spectrum see Table 4.

Adduct 24b was obtained in 20% yield, m.p. 181°C (dec.); IR (Nujol), 2040, 1980, 1960, 1760, 1700 cm^{-1}. Anal.: Found: C, 53.61; H, 3.57; N, 8.38. $C_{22}H_{17}FeN_3O_7$ calcd.: C, 53.77; H, 3.46; N, 8.55%. For ^1H NMR spectrum see Table 4.

Adduct 24c was obtained in 16% yield, m.p. 145°C (dec.); IR (Nujol), 2040, 1980, 1965, 1710 cm^{-1}; *m/e* 505 (*M*). For ^1H NMR spectrum see Table 4.

Adduct 24d was obtained in 27% yield, m.p. 152°C (dec.); IR (Nujol) 2040, 1990, 1950, 1760, 1715 cm^{-1}. Anal.: Found: C, 54.73; H, 3.17; N, 12.38. $C_{29}H_{14}FeN_4O_5$ calcd.: C, 55.02; H, 3.06; N, 12.23%. For ^1H NMR spectrum see Table 4.

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