

Ferrocenyl substituted chlorostilbenes and butadienes

K. Senthil Kumar, K.C. Kumara Swamy *

School of Chemistry, University of Hyderabad, Hyderabad 500046, Andhra Pradesh, India

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Abstract

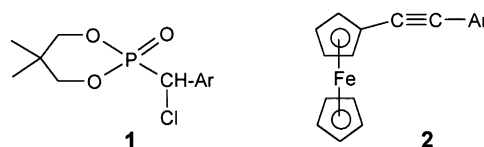
The readily accessible α -chlorophosphonates $(\text{OCH}_2\text{CMe}_2\text{CH}_2\text{O})\text{P}(\text{O})\text{CH}(\text{Cl})\text{-C}_6\text{H}_4\text{-4-R}$ [**1**: R = Me (**a**), OMe (**b**), Cl (**c**), H (**d**)] react with ferrocenecarboxaldehyde in the presence of NaH [Horner–Wadsworth–Emmons reaction] to give good yields of ferrocenyl substituted chlorostilbenes. The novel bis ferrocenyl butadiene $\text{C}_5\text{H}_5\text{FeC}_5\text{H}_4\text{-CH=CH-C}(\text{CN})\text{CHC}_5\text{H}_4\text{FeC}_5\text{H}_5$ (**9**) as well as the ferrocenyl 2-cyano-1,3-butadienes $4\text{-R-C}_6\text{H}_4\text{-CH=CH-C}(\text{CN})=\text{CHC}_5\text{H}_4\text{FeC}_5\text{H}_5$ [R = Me (**10a**), OMe (**10b**), Cl (**10c**)] have been obtained by using the new allylphosphonate $(\text{OCH}_2\text{CMe}_2\text{CH}_2\text{O})\text{P}(\text{O})\text{CH}_2\text{C}(\text{CN})=\text{CHC}_5\text{H}_4\text{FeC}_5\text{H}_5$ (**8**); the latter compound was prepared in good yields by the reaction of the Baylis–Hillman adduct, $\text{C}_5\text{H}_5\text{FeC}_5\text{H}_4\text{CH}(\text{OH})\text{C}(\text{CN})=\text{CH}_2$ (**7**), with the chlorophosphite $(\text{OCH}_2\text{CMe}_2\text{CH}_2\text{O})\text{PCl}$. The electrochemical behavior of the ferrocenyl compounds thus synthesized has been studied; two reversible one-electron processes are observed in the case of compound **9** suggesting a cooperative interaction between the two ferrocenyl residues. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Ferrocenyl substituted olefins; Horner–Wadsworth–Emmons reaction; Baylis–Hillman adducts

1. Introduction

A convenient route to the α -chlorophosphonates of the type $(\text{OCH}_2\text{CMe}_2\text{CH}_2\text{O})\text{P}(\text{O})\text{CH}(\text{Cl})\text{Ar}$ (**1**) that are useful precursors for the Horner–Wadsworth–Emmons (HWE) reaction has been reported earlier by us [1,2]. Based on these phosphonates, we also developed a simple route to ferrocenyl substituted acetylenes $\text{C}_5\text{H}_5\text{FeC}_5\text{H}_4\text{C}\equiv\text{CC}_6\text{H}_4\text{-4-Cl}$ (**2**) [3] formed by HCl elimination from the HWE products obtained by the reaction of **1** with ferrocenecarboxaldehyde. In this context, it was of interest to see (i) whether the precursor HWE products of the type $(\text{C}_5\text{H}_5\text{FeC}_5\text{H}_4)\text{CH}=\text{C}(\text{Cl})(\text{Ar})$ (**3**) are stable or not and (ii) whether products in which Ar = ferrocenyl could be obtained or not. It can be noted that although a few reports are available on the synthesis of ferrocenyl substituted olefins [4–7] a general synthetic strategy for this class of compounds has not been developed. In this connection we also considered the possibility of using phosphonates derived from the reaction of a chlorophosphite $(\text{RO})_2\text{PCl}$ with Baylis–Hillman adducts [8] of the type $(\text{C}_5\text{H}_5\text{FeC}_5\text{-}$

$\text{H}_4)\text{CH}(\text{OH})(\text{CN})=\text{CH}_2$. Thus, in this paper, we describe the synthesis of: (a) ferrocenyl substituted stilbenes; and (b) β -cyano substituted ferrocenyl butadienes.



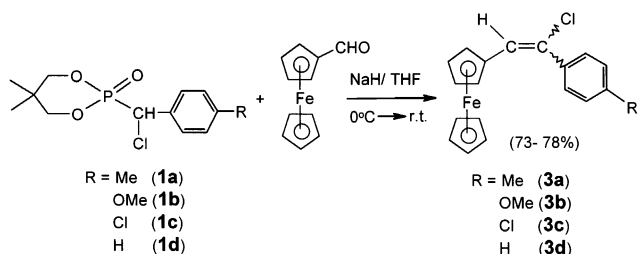
2. Results and discussion

Reaction of α -chlorophosphonates **1(a–d)** [1–3] with NaH followed by ferrocenecarboxaldehyde using THF as the solvent gave the chloro substituted olefins **3(a–d)** as a (*Z* + *E*) isomeric mixture of products in good yields (Scheme 1). The stoichiometry of NaH and the solvent are important here; larger quantities of NaH and the use of dimethylsulfoxide as a solvent lead to acetylenes as reported before [3].

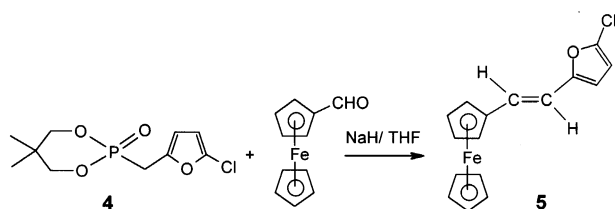
The 5-chlorofurfuryl phosphonate (**4**) [3] also reacts readily with ferrocenecarboxaldehyde to give the olefin **5**, but in this case, only the *E*-olefin is obtained (Scheme 2).

* Corresponding author. Fax: +91-40-301-2460.

E-mail address: kckssc@uohyd.ernet.in (K.C. Kumara Swamy).

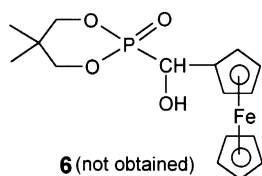


Scheme 1.



Scheme 2.

As an alternative, we wanted to prepare the α -hydroxyphosphonate (**6**) by treating $(\text{OCH}_2\text{CMe}_2\text{CH}_2\text{O})\text{P}(\text{O})\text{H}$ with ferrocenecarboxaldehyde (it was presumed that if **6** was obtained, it could be chlorinated at the α -position using SOCl_2). Unfortunately, this reaction did not proceed in the expected manner, and the only product that we could isolate was a phosphate ester [$\delta(\text{P})$: -12.8], which was not analyzed further.

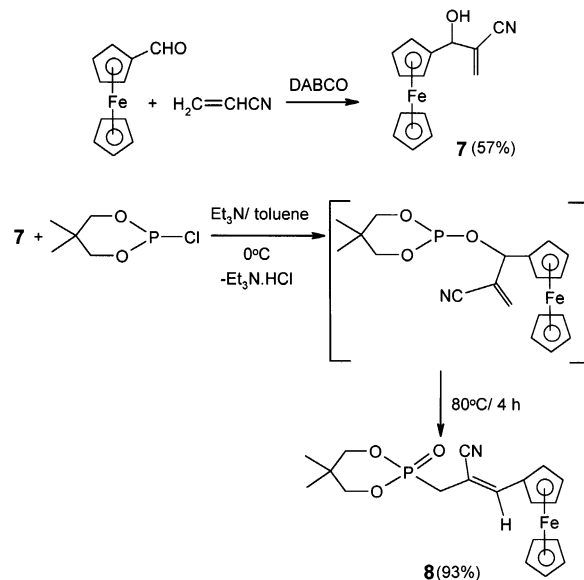


For the synthesis of ferrocenyl substituted butadienes we first prepared the precursor allyl alcohol **7** by the Baylis–Hillman methodology [8–11]. Treatment of **7** with the cyclic chlorophosphite $(\text{OCH}_2\text{CMe}_2\text{CH}_2\text{O})\text{PCl}$ [3] followed by thermal Arbuzov rearrangement gave the phosphonate **8** (Scheme 3). Treatment of **8** with one mole equivalent of ferrocenecarboxaldehyde in the presence of NaH readily formed the novel bis (ferrocenyl) substituted butadiene **9** as a single isomer in high yields (Scheme 4). Similar reactions with aromatic aldehydes formed the monoferrocenyl substituted butadienes **10(a–c)**. Two points are to be noted in this synthesis: (i) although other chlorophosphites [e.g. $(\text{EtO})_2\text{PCl}$] can also be utilized to prepare phosphonates similar to **8**, they are expensive or inconvenient to handle; (ii) variations that are possible include the use of other vinylic systems [e.g. $\text{CH}_2=\text{CH}(\text{COOMe})$] in place of acrylonitrile in the preparation of ferrocenyl substituted Baylis–Hillman adducts (cf. Scheme 3).

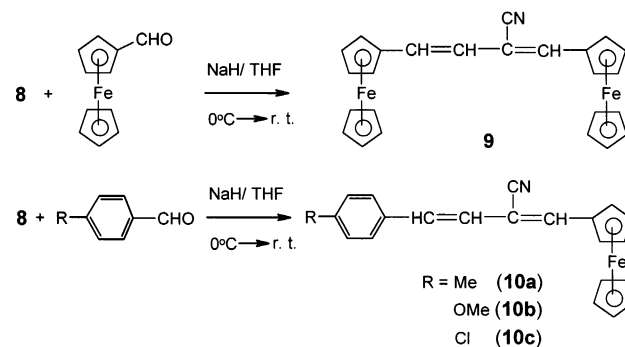
As ferrocene and its derivatives are redox active [12–14], we have recorded the cyclic voltammograms

for **3(a–d)**, **9**, **10(a–c)** as well as the acetylenes $(\text{C}_5\text{H}_5\text{FeC}_5\text{H}_4)\text{C}\equiv\text{C}-\text{C}_6\text{H}_4-4-\text{R}$ [$\text{R} = \text{Cl}, \text{Me}$] to compare the electrochemical behavior of these compounds with that of ferrocene. In all the cases except **9**, a reversible redox couple with $E_{1/2}$ in the range 0.42–0.54 V (Table 1) at a scan rate of 100 mV s^{-1} is observed. The equal positive and negative peak heights and peak-to-peak separation (ΔE_p) suggest the characteristic one-electron reversible process. For **9**, two reversible one-electron processes are observed (Fig. 1) with $E_{1/2}$ values 0.38 and 0.58 V suggesting a cooperative interaction [15–17] between the two ferrocenyl residues through the conjugated system.

In summary, new ferrocenyl substituted olefins that include the bis (ferrocenyl) butadiene **8** have been synthesized. Particularly, the use of phosphonates derived from the Baylis–Hillman methodology may be developed further to incorporate ferrocenyl residues in complex organic systems as these olefins have many reactive centers.



Scheme 3.



Scheme 4.

Table 1

Cyclic voltammetric data for compounds **3a–d**, **9**, **10a–c**, $C_5H_5FeC_5H_4C \equiv CC_6H_4-4-Cl$ and $C_5H_5FeC_5H_4C \equiv CC_6H_4-4-Me$ ^a

Compound	$E_{1/2}$ (ΔE_p)
3a	+0.44 (97)
3b	+0.42 (72)
3c	+0.44 (86)
3d	+0.47 (93)
9	+0.38 (73), +0.58 (59)
10a	+0.53 (76)
10b	+0.52 (63)
10c	+0.54 (83)
$C_5H_5FeC_5H_4C \equiv CC_6H_4-4-Cl$ ^b	+0.52 (76)
$C_5H_5FeC_5H_4C \equiv CC_6H_4-4-Me$ ^b	+0.49 (70)

^a $E_{1/2}$ in V (with respect to Ag/AgCl) and ΔE_p in mV; standardized with respect to the ferrocene–ferrocenium ion couple in acetonitrile, TBAP [$E_{1/2}(\Delta E_p) = 0.38$ (85)].

^b Prepared as outlined in Ref. [3].

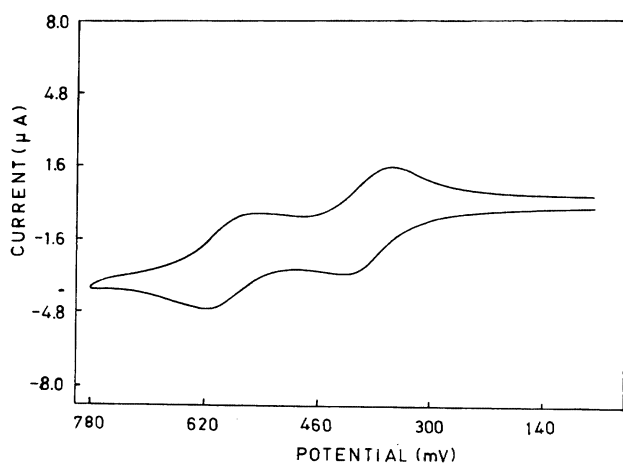


Fig. 1. Cyclic voltammogram for compound **9** showing the two reversible one-electron processes.

3. Experimental

Solvents were dried and distilled prior to use [18]. Chemicals were purchased from Aldrich or local sources. Phosphonates $(OCH_2CMe_2CH_2O)P(O)CH(Cl)-C_6H_4-4-R$ [**1**: R = Me (**a**), OMe (**b**), Cl (**c**), H (**d**)] and $(OCH_2CMe_2CH_2O)P(O)CH_2-5-Cl-C_4H_3O$ (**4**) were prepared by using the methods outlined in the literature [1–3]. The NMR spectra were recorded in $CDCl_3$ on a Bruker 200 MHz NMR spectrometer. IR spectra were recorded on a JASCO FTIR 5300 spectrophotometer. Elemental analyses were obtained for representative compounds and were carried out on a Perkin–Elmer 240C CHN analyzer. Mass spectra were recorded on a CEC-21-110B double focusing mass spectrometer. Cyclic voltammograms were recorded on a Cypress systems model CS-1090/CS-1087 electroanalytical system; these measurements were made under dry nitrogen in MeCN with 0.1 M $[Bu_4N][ClO_4]$ as the

supporting electrolyte using glassy carbon working electrode, Ag/AgCl reference electrode and platinum wire as auxiliary electrode with ferrocene–ferrocenium couple as the redox standard [13].

3.1. Synthesis of the chloro substituted olefins

$C_5H_5FeC_5H_4CH=C(Cl)-C_6H_4-4-R$ [**3**: R = Me (**a**), OMe (**b**), Cl (**c**), H (**d**)]

3.1.1. Typical procedure for **3a**

The phosphonate **1a** (0.5 g, 1.73 mmol) in THF (10 ml) was added to a stirred suspension of NaH (washed earlier with hexane) (0.16 g, 6.66 mmol) in THF (20 ml) at 0 °C. After 0.5 h, ferrocenecarboxaldehyde (0.37 g, 1.73 mmol) in THF (10 ml) was added dropwise during 15 min and the mixture was stirred for 24 h. After quenching with cold water (30 ml), the mixture was extracted with ether (3×20 ml). The ether layer was dried (Na_2SO_4) and the solvent was removed to obtain a semi-solid which was purified by column chromatography (silica gel, hexane) to obtain **3a** (mixture of isomers). Yield: 0.46 g (79%). M.p. (dec.): 100–102 °C. IR (cm^{-1} , major bands): ν 1508, 1456, 1406. ¹H-NMR: δ 2.41 (2 s merged, 3H, Ar- CH_3), 3.94, 4.13, 4.15, 4.21, 4.82 (m, together 9H, ferrocenyl-*H*), 6.62, 6.81 (2 s, ratio 2:1, 1H, (Cl)C=CH), 7.19–7.60 (m, 4H, Ar-*H*). ¹³C-NMR: δ 21.2, 21.4 (Ar- CH_3), 68.7, 69.2, 70.1 (ferrocenyl-*C*), 79.7, 80.4 (*C*(ferrocenyl)CH=CCl-), 123.9, 126.0, 126.6, 128.1, 129.0, 129.1, 135.5, 138.6, (olefinic *C* + Ar-*C*). MS: 338, 336 $[M]^+$ (^{35,37}Cl), 165, 152, 139.

Compounds **3b–d** and **5** were prepared similarly using the same molar quantities.

3b. Yield: 0.36 g (75%). M.p. (dec.): 68–70 °C. IR (cm^{-1} , major bands): ν 1599, 1508, 1458. ¹H-NMR: δ 3.85 (2 s merged, 3H, Ar- OCH_3), 3.93, 4.13, 4.15, 4.30, 4.75 (m, together 9H, ferrocenyl-*H*), 6.56, 6.70, (2 s, ratio 1:1, 1H, (Cl)C=CH), 6.80–7.65 (m, 4H, Ar-*H*). ¹³C-NMR: δ 55.3 (Ar- OCH_3), 68.7, 69.1, 69.9 (ferrocenyl-*C*), 79.7, 80.4, (*C*(ferrocenyl)CH=CCl-), 113.7, 123.0, 126.3, 127.4, 130.4, (olefinic *C* + Ar-*C*). MS: 352, 354 $[M]^+$ (^{35,37}Cl), 165, 152, 139.

3c. Yield: 0.45 g (78%). M.p. (dec.): 88–90 °C. IR (cm^{-1} , major bands): ν 1618, 1487, 1400. ¹H-NMR: δ 3.91, 4.13, 4.19, 4.36, 4.79 (m, together 9H, ferrocenyl-*H*), 6.63, 6.81 (2 s, ratio 1:2, 1H, (Cl)C=CH), 7.26–7.57 (m, 4H, Ar-*H*). ¹³C-NMR: δ 68.8, 69.0, 69.3, 69.5, 70.2 (ferrocenyl-*C*), 79.2, 80.4, (*C*(ferrocenyl)CH=CCl-), 125.4, 127.2, 127.6, 128.5, 128.6, 130.5, 133.8 (olefinic *C* + Ar-*C*). Anal. Found: C, 60.45; H, 3.68. Calc. for $C_{18}H_{14}Cl_2Fe$: C, 60.52; H, 3.92%.

3d. Yield: 0.43 g (73%, liquid). IR (cm^{-1}): ν 1625, 1490, 1448, 1402. ¹H-NMR: δ 3.89, 4.11, 4.20, 4.36, 4.81 (m, together 9H, ferrocenyl-*H*), 6.62, 6.83 (2 s ratio 1:1, 1H, (Cl)C=CH), 7.26–7.69 (m, 4H, Ar-*H*). ¹³C-NMR: δ 68.8, 69.2, 70.1 (ferrocenyl-*C*), 79.2, 80.1,

(C(ferrocenyl)CH=C(Cl)-), 124.8, 126.1, 126.9, 128.0, 128.4, 128.6, 129.1 (olefinic C + Ar-C).

5. Yield: 0.30 g (52%). IR (cm⁻¹): ν 1710, 1670, 1639, 1570, 1504, 1450. ¹H-NMR: 4.15, 4.30, 4.43 (3 s, 9H, ferrocenyl-H), 6.15 (s, 2H, furfuryl-H), 6.36, 6.76 (AB qrt, ³J(H-H) = 16 Hz, 2H, CH=CH). ¹³C-NMR: 66.9, 69.7 (ferrocenyl-C), 82.0 (C(ferrocenyl)CH=CH), 108.0, 126.5, 136.0 (olefinic C + furfuryl C).

3.1.2. Preparation of the Baylis–Hillman adduct C₅H₅FeC₅H₄CH(OH)C(CN)=CH₂ [7]

A mixture of ferrocenecarboxaldehyde (0.5 g, 2.3 mmol), acrylonitrile (0.12 g, 2.3 mmol) and 1,4-diazabicyclo[2.2.2]octane (DABCO, 0.05 g, 0.4 mmol) was allowed to stand at room temperature (r.t.) for 3 days, then taken up in Et₂O (100 ml), washed with 10% dilute HCl (50 ml). The ether phase was washed with water (50 ml) and dried (Na₂SO₄). The solvent was removed to obtain a solid which was purified by column chromatography (silica gel, hexane + EtOAc (99:1), yellow band after ferrocenecarboxaldehyde) to obtain a yellow solid. [There was also a second product (red) with a low R_f value]. Yield: 0.36 g (57%). M.p. (dec.): 78 °C. IR (cm⁻¹): 3466, 2226, 1734. ¹H-NMR: δ 2.44 (2 d, ²J(H-H) = 5.5 Hz, 1H, CH(OH)), 4.28 (s, 7H, ferrocenyl-H), 4.93 (m, 2H, ferrocenyl-H), 6.0 (AB qrt → 2 s, 2H, C=CH₂). ¹³C-NMR: δ 65.8, 67.4, 68.8, 70.2, (ferrocenyl-C), 90.1 (CH(OH)), 117.3 (C≡N), 125.9 (C(CN)=CH₂), 129.8 (=CH₂).

3.1.3. Preparation of 2-cyano-2-alkenylphosphonates (OCH₂CMe₂CH₂O)P(O)CH₂C(CN)=C(H)C₅H₄FeC₅H₅ [8]

To a stirred solution of the Baylis–Hillman adduct 7 (0.65 g, 2.45 mmol) and Et₃N (0.24 g, 2.45 mmol) in toluene (50 ml) (OCH₂CMe₂CH₂O)PCl [3] (0.41 g, 2.45 mmol) was added dropwise at 0 °C under N₂ and the mixture was stirred for 6 h. The precipitate was filtered off, washed with toluene (2 × 10 ml) and the washings were added to the filtrate. The solvent from the combined filtrate was removed and the residue was heated at 80 °C under N₂ for 4 h. The resultant product was purified by column chromatography (silica gel, hexane + EtOAc (1:1)) to obtain a dark-red solid. Yield: 0.85 g (86%). M.p. 148 °C. IR (cm⁻¹): 2208, 1618, 1473, 1400, 1265. ¹H-NMR: δ 1.07, 1.15 (2 s, 6H, 2 CH₃), 2.84 (d, ²J(P-H) = 19.9 Hz, 2H, P-CH₂), 3.88–4.31 (m, 4H, 2 OCH₂), 4.25 (s, 5H, C₅H₅FeC₅H₄), 4.46 (t, ³J(H-H) = 2.0 Hz, 2 H, C₅H₅FeC₅H₄), 4.83 (t, ³J(H-H) = 2.0 Hz, 2 H, C₅H₅FeC₅H₄), 7.04 (d, ⁴J(P-H) = 4.9 Hz, C(CN)=CH). ¹³C-NMR: δ 21.5, 21.6 (2 CH₃), 30.8 (d, ¹J(P-C) = 137.5 Hz, P-CH₂) 32.7 (CMe₂), 69.8, 71.3 (ferrocenyl-C), 75.7, 75.8 (d, ²J(P-C) = 6.0 Hz, -OCH₂), 94.6 (d, ³J(P-C) = 5.0 Hz, CH=C(CN)), 118.2 (C≡N), 149.6 (d, ³J(P-C) = 10.0 Hz, C(H)=C(CN)). ³¹P-NMR: δ 18.1. Anal. Found: C,

57.08; H, 5.45; N, 3.32. Calc. for C₁₉H₂₂FeNO₃P: C, 57.14; H, 5.51; N, 3.51%.

3.2. Synthesis of 2-cyano-1,3-butadienes containing ferrocene C₅H₅FeC₅H₄CH=C(CN)CH=CH-R [R=C₅H₅FeC₅H₄ (**9**), 4-Me-C₆H₄ (**10a**), 4-OMe-C₆H₄ (**10b**), 4-Cl-C₆H₄ (**10c**)]

3.2.1. Typical procedure for **10a**

Ferrocenyl allylphosphonate (**8**) (0.61 g, 1.53 mmol) in THF (10 ml) was added to a stirred suspension of NaH (0.15 g of 80% dispersion, 6.25 mmol) in THF (20 ml) at 0 °C. After 0.5 h, *p*-tolualdehyde (0.18 g, 1.49 mmol) in THF (10 ml) was added over a period of 5 min. The reaction mixture was stirred for 6 h, quenched with cold water and then extracted with Et₂O (3 × 20 ml). The ether layer was dried (Na₂SO₄) and the solvent was removed to obtain a solid which was then purified by column chromatography (silica gel, hexane) to obtain **10a** as a red solid (only one isomer). Yield: 0.5 g (93%). M.p. 132–134 °C. IR (cm⁻¹): 2214, 1583, 1512. ¹H-NMR: δ 2.35 (s, 3H, Ar-CH₃), 4.23, 4.51, 4.90 (m, together 9H, ferrocenyl-H), 6.69 (d, ³J(H-H) = 16.0 Hz, 1H, CH=CH), 6.92 (d, ³J(H-H) = 15.8 Hz, 1H, CH=CH), 6.94–7.37 (m, 4H, Ar-H). ¹³C-NMR: δ 21.4 (s, Ar-CH₃), 69.9, 71.5 (ferrocenyl-C), 107.2, 125.0, 126.5, 129.6, 130.7, 144.3 (olefinic C + CN + Ar-C). MS: 353 [M]⁺, 288, 212, 203, 190, 186, 121, 115, 107, 91.

Compounds **9** and **10b** and **10c** were obtained by using the same procedure with the same molar quantities.

9. Yield: 0.24 g (88%). M.p. 198–200 °C. IR (cm⁻¹): 2216, 1614, 1587. ¹H-NMR: δ 4.15, 4.22, 4.31, 4.41, 4.48, 4.86 (m, together 18H, 2C₅H₅FeC₅H₄), 6.33 (d, ³J(H-H) = 15.7 Hz, 1H, CH=CH), 6.72 (d, ³J(H-H) = 15.7 Hz, 1H, CH=CH-), 6.82 (s, 1H, CH=C(CN)). ¹³C-NMR: δ 67.0, 69.3, 69.5, 69.5, 69.6, 69.8, 71.2 (ferrocenyl-C), 82.1 (C(ferrocenyl)-CH=CH), 107.6, 117.7 (C≡N), 123.3, 129.7, 141.8 (olefinic-C). MS: 447 [M]⁺, 382, 325, 305, 300, 260, 237, 224, 205, 186, 178, 172, 121.

10b. Yield: 0.35 g (88%). M.p. 102–104 °C. IR (cm⁻¹): 2216, 1684, 1602, 1510. ¹H-NMR: δ 3.83 (s, 3H, Ar-OCH₃), 4.22 (s), 4.51 (m), 4.89 (m) [together 9H, all ferrocenyl-H], 6.61 (d, ³J(H-H) = 15.8 Hz, CH=CH), 6.86–7.42 (m, 6H, olefinic H + CH=C(CN) + Ar-H). ¹³C-NMR: δ 55.4 (-OCH₃), 69.8, 69.9, 70.9, 71.4 (ferrocenyl-C), 77.9 (C(ferrocenyl)-CH=CH), 107.3, 114.3, 117.7, 120.9, 123.9, 127.9, 128.3, 129.2, 130.1, 143.7, 159.7 (olefinic C + CN + Ar-C).

10c. Yield: 0.17 g (85%). M.p. 120–122 °C. IR: 2218, 1579, 1489 cm⁻¹. ¹H-NMR: δ 4.23 (s), 4.54 (m), 4.91 (m), [together 9H, ferrocenyl-H], 6.70 (d, ³J(H-H) = 15.8 Hz, CH=CH), 6.85–7.43 (m, 6H, olefinic-H +

$CH=C(CN)$, Ar- H). ^{13}C -NMR: 68.3, 69.9, 70.7, 71.7 (ferrocenyl- C), 106.6, 108.6, 119.0, 121.9, 126.6, 127.7, 128.0, 129.1, 130.8, 132.5, 134.2, 134.8, 141.8, 143.3, 145.6 (olefinic $C + C\equiv N + Ar-C$). Anal. Found: C, 67.35; H, 4.17; N, 3.56. Calc. for $C_{21}H_{16}FeNCl$: C, 67.48; H, 4.28; N, 3.75%.

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