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Synthesis of ferrocenyl-β-enamino ketones: A search of ferrocenylpyrido[2,3-*d*]pyrimidines using a $Ni(CN)_2$/NaOH/KCN system as catalytic precursor

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ABSTRACT

New ferrocenyl-β-enamino ketones (**1–6**), were obtained from 6-amino-1,3-dimethyluracil and several ferrocenyl-α-ketoalkynes *via* $MeNH^-$ anion in a monophasic aqueous system containing $Ni(CN)_2$/CO/NaOH/H_2O/KCN are described. A mechanism for obtaining of β-enamino ketones is suggested.

1. Introduction

β-Enamino ketones are versatile synthetic intermediates for the synthesis of several natural products [1], γ-aminoalcohols [2] and different heterocyclic compounds such as pyridones, pyrimidones, pyranones, isoquinolines [3], and present interesting pharmacological properties such as anticonvulsants and are pharmacophores for other several drugs [4]. These β-enamino ketones can be synthesized in variety of ways [3b] and the most common method includes the condensation of amines with β-dicarbonyl compounds under homogeneous or heterogeneous catalytic conditions [5]. Though variety of β-enamino ketones are reported in literature, ferrocenyl-β-enamino ketone, where the ferrocenyl group is linked to the nucleophilic side of the molecule, is unknown. On the other hand ferrocenyl group has been widely used in the design or redesign of drugs that can result favorable changes in their biological activities [6–9]. In view of the above it is worth while to prepare some new ferrocenyl-β-enamino ketones.

2. Results and discussion

New ferrocenyl-β-enamino ketones obtained in this work (Table 1) are characterized by various physicochemical techniques. In the IR spectra of all the synthesized ferrocenyl-β-enamino ketones, C=O and C=C vibrations are observed. A weak band at 3095 cm^{-1} can be attributed to the stretching vibration of the hydrogen bonded enamine N–H group. This band appears at a lower frequency as a consequence of N–H···O=C intramolecular interaction. A similar observation has been reported earlier [10]. In these compounds the N–H stretching vibration appears at lower frequency due to the presence of electron donor ferrocenyl moiety at the nucleophilic side of the compounds in comparison to the N–H vibration observed for similar ferrocenyl enaminones (3203 cm^{-1}) where the ferrocenyl moiety is at the electrophilic side [10]. IR spectrum of compound (**1**) in $CHCl_3$ shows C=O vibration at 1592 cm^{-1}, suggesting the presence of α,β unsaturated carbonyl system. In the mass spectra of all the compounds, molecular ion peaks are observed along with the loss of cyclopentadienyl fragment $[M\text{-}65]^+$ which corresponds to base peak in the fragmentation pattern. ^1H NMR spectra of all the compounds present similar chemical shifts pattern for ferrocenyl group. In all the compounds a singlet appearing at ~11.85 ppm and doublet appearing at ~3.13 ppm can be assigned to the aminic protons. The appearance of NH proton at higher chemical shift is due to the presence of intramolecular H-bonding. This observation also confirms the absence of the imine form in these compounds. The absence of two sets of signals in IR and NMR spectra for these compounds confirms the existence of only enamino configuration, contrary to the doubling of signals observed earlier in similar compounds [10]. In the ^{13}C NMR spectra of all these compounds a signal at 186–187 ppm can be assigned to the carbonyl group.

In the case of 1-*p*-methoxy-phenyl-3-methylamino-3-ferrocenyl-2-propenone (**1**), the molecular structure was unambiguously established by X-ray crystallography and exists as Z–s–Z configura-

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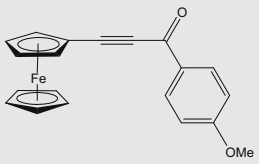
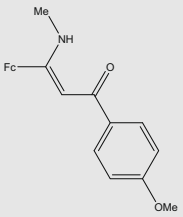
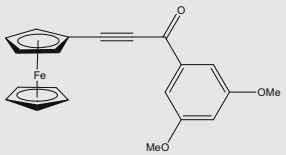
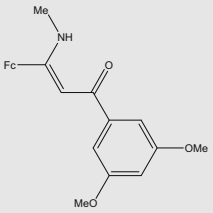
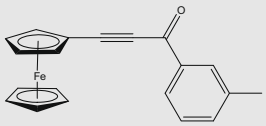
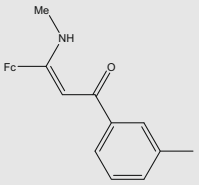
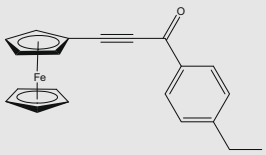
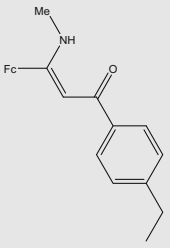
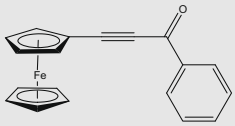
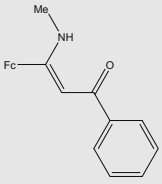
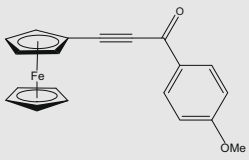
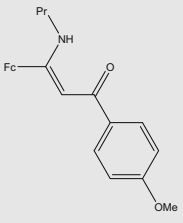
Table 1
Ferrocenyl β-enamino ketones.

Entry	Ferrocenyl-α-ketoalkynes	Uracil	Ferrocenyl-β-enamino ketones	Yield %
1		6-Amino-1,3-dimethyluracil		75
2		6-Amino-1,3-dimethyluracil		74
3		6-Amino-1,3-dimethyluracil		80
4		6-Amino-1,3-dimethyluracil		72
5		6-Amino-1,3-dimethyluracil		78
6		6-Amino-1,3-dipropyluracil		72

Reaction conditions: Time 15 min; temperature 55 °C; $Ni(CN)_2$/CO/5N NaOH/KCN; yielding 7-ferrocenyl-2,4-dioxopyrido[2,3-*d*]pyrimidines <5%.
No reaction without $(NiCN)_2$/CO/KCN for 24 h. at 55 °C. Fc = Ferrocenyl group.

tion as shown in Fig. 1. Crystal data and selected bond lengths and angles for **1** are given in Table 2 and 3, respectively. The compound is monomeric and crystallizes in the space group $P2_1$ with Flack parameter −0.041(18). This compound crystallizes with two independent molecules per asymmetric unit and presents an intramolecular N–H···O=C hydrogen bond. The C=C–C=O atoms are planar and the bond lengths indicate electron delocalization. C(3)–C(10) bond is a typical sp^2–sp^2 single bond, suggesting that the cyclopentadienyl ring is not involved in the conjugation.

Previously our group has reported that at room temperature in the presence of a nickel catalytic system *viz* $Ni(CN)_2$/CO/KCN/NaOH, reaction of ferrocenyl-α-ketoalkynes and 6-amino-1,3 dimethyluracil yield 7-ferrocenyl-2,4-dioxopyrido[2,3-*d*]pyrimidines [11]. But in this work, increasing the temperature from 25° at 55 °C, an anionic specie $MeNH^-$ is formed by the decomposition of 6-amino-1,3-dimethyluracil which is facilitated by the another anionic catalytic specie $[Ni(CN)_4]^{-4}$, obtaining the corresponding ferrocenyl-β-enamino ketones as is shown in Scheme 1. Ferrocenylpyrido[2,3-*d*]pyrimidines were also obtained but in very low yields (5%) at this temperature. But in the absence of $Ni(CN)_2$, CO and KCN catalytic system, no reaction occurred even after 24 h.

A plausible two steps pathway can be envisaged for the preparation of ferrocenyl-β-enamino ketones.

The first step involves the retrocondensation of 6-amino-1,3-dimethyluracil affording *N,N′*-dimethylurea and cyanoacetate ion [12] (Scheme 2). The second step involves a nucleophilic attack of $[Ni(CN)_4]^{-4}$, formed *in situ* in basic media, on the *N,N′*-dimethylurea, affording the methylamide $MeNH^-$ [13]. This anion after the

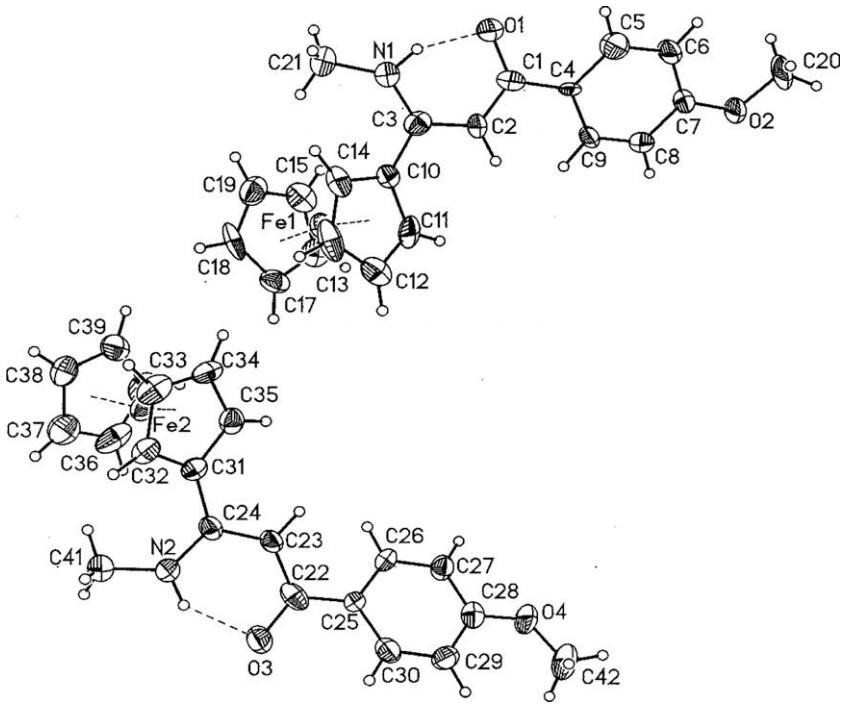
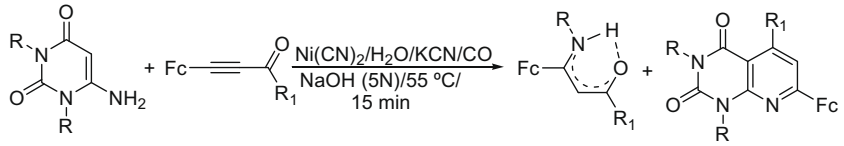


Fig. 1. ORTEP diagram of 1-*p*-methoxy-phenyl-3-methylamino-3-ferrocenyl-2-propenone (**1**).

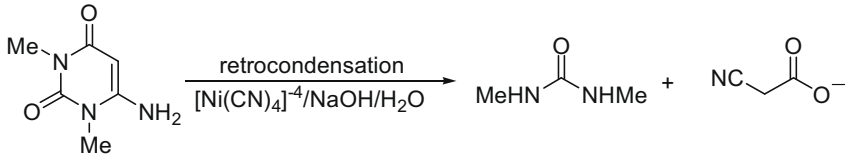
Table 2
Crystallographic data for compounds (**1**).

Parameter	Compound (**1**)	Parameter	Compound (**1**)
Empirical formula	$C_{21}H_{21}FeNO_2$	Z	4
Formula weight	375.24	D_{Calc} (Mg/m^3)	1.400
Crystal color	Red prism	λ (mm^{-1})	0.860
Crystal system	Monoclinic	2Θ (°)	1.81–25.35
Space group	$P2_1$	Reflections collected	14 810
Crystal size (mm)	0.34 × 0.13 × 0.03	Independent reflections	6486
a (Å)	11.4202(13)	R_{int}	0.0708
b (Å)	8.813(1)	R_1 [$I > 2\sigma(I)$]	0.0560
c (Å)	17.962(2)	wR_2	0.0801
α (°)	90	Goodness-of-fit (GOF)	0.808
β (°)	99.912(2)	Flacks parameter	−0.041(18)
γ (°)	90	Max/min $\Delta\rho$ (e Å^{-3})	0.577/−0.321
V (Å^3)	1780.9(3)		

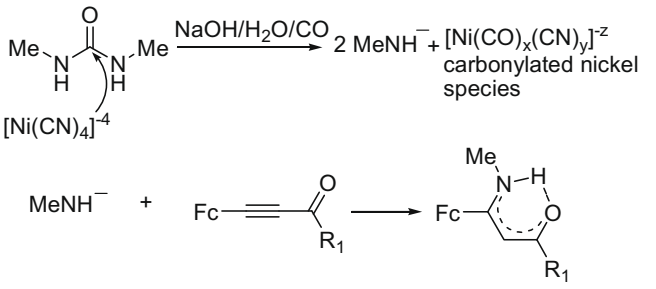


Fc= Ferrocenyl
1 R= Me, R_1= *p*-methoxyphenyl
2 R= Me, R_1= 3,5-dimethoxyphenyl
3 R= Me, R_1= *m*-tolyl
4 R= Me, R_1= *p*-ethylphenyl
5 R= Me, R_1= Phenyl
6 R= *n*-Pr, R_1= *p*-methoxyphenyl

Scheme 1.



Scheme 2.



Scheme 3.

Table 3
Selected bond length (Å) and selected bond angles (°) for the compounds (**1**).

Angles (°)		Bond length (Å)	
O(1)–C(1)–C(2)	121.4(6)	O(1)–C(1)	1.286(6)
N(1)–C(3)–C(2)	120.1(5)	C(1)–C(2)	1.404(7)
C(3)–C(2)–C(1)	125.8(6)	C(2)–C(3)	1.382(7)
O(3)–C(22)–C(23)	120.3(6)	C(3)–N(1)	1.322(7)
N(2)–C(24)–C(23)	118.5(5)	O(3)–C(22)	1.262(6)
C(24)–C(23)–C(22)	126.5(5)	C(22)–C(23)	1.442(7)
C(2)–C(3)–C(10)	118.7(5)	C(23)–C(24)	1.387(7)
C(23)–C(24)–C(31)	120.2(5)	C(24)–N(2)	1.343(6)

1,2-addition to ferrocenyl-α-ketoalkynes yields the ferrocenyl-β-enamino ketones (Scheme 3).

In order to confirm that alkylamide ion is the nucleophilic specie is formed in the second step, the reaction is carried out using 6-amino-1,3-dipropyluracil, instead of 6-amino-1,3-dimethyluracil with 1-*p*-methoxy-phenyl-3-ferrocenyl-propynone yielding 1-*p*-methoxy-phenyl-3-propylamino-3-ferrocenyl-2-propenone, (entry 6, Table 1).

In summary, we found a new one pot method to prepare ferrocenyl-β-enamino ketones from 6-amino-1,3-disustituted uracil derivatives and several ferrocenyl-α-ketoalkynes, these reactions can be performed in water with mild conditions in the presence of Ni catalyst.

3. Experimental

3.1. General procedure

A 5 N NaOH solution (25 mL) was saturated by slowly bubbling CO at room temperature for 30 min. To the solution was then added 2 mmol of $Ni(CN)_2{\cdot}4H_2O$ and stirred under a CO atmosphere until a pale yellow solution was obtained. Addition of 15 mmol of KCN resulted in a color change to orange. The 6-amino-1,3-disustituted uracil (5 mmol) and the ferrocenyl-α-ketoalkynes [6] (5 mmol) were added keeping the temperature at 55 °C, the mixture was stirred for 15 min. The evolution of the reaction was followed by TLC. The reaction products were quantified in a Hewlett Packard 5870 until the end of reaction. After the usual workup the products were purified by crystallization.

3.2. X-ray crystallography

The X-ray intensity data were measured at 298 K on a Bruker Smart APEX CCD-based X-ray diffractometer using a monochromatized Mo Kα radiation (Kα 0.71073 Å). The detector was placed at a distance of 4.837 cm from the crystal. Analysis of the data showed negligible decays during data collections. An analytical face indexed absorption correction was applied. Crystal structure was refined by full-matrix least squares. SMART software (data collection and data reduction) and SHELXTL were used for solution and refinement of the structure.

3.3. 1-p-Methoxyphenyl-3-methylamino-3-ferrocenyl-2-propenone (*1*)

The product was obtained as described in the general procedure as an orange solid (75%); Empirical formula: $C_{21}H_{21}NO_2Fe$; mp. 123 °C; IR ($CHCl_3$ solution, selected, cm^{-1}) 1326 (OMe), 1592 (C=O), 3095 (N–H); Mass spectrum EI: *m*/*z* (%) = 375 (66) $[M]^+$, 310 (100) $[M–cp]^+$; ^1H NMR (300 MHz, $CDCl_3$, δ in ppm) 3.13 (d, 3H, *J* = 5.5 Hz, NCH_3), 3.86 (s, 3H, OMe), 4.23 (s, 5H, cp-ring), 4.42 (t, 2H, *J* = 1.9 Hz, cp-ring), 4.61 (t, 2H, *J* = 1.9 Hz, ring-cp), 6.18 (s, 1H, C=C*H*), 6.94 (d, 2H, *J* = 8.5 Hz, 3,5-phenyl), 7.89 (d, 2H, *J* = 8.8 Hz, 2,6-phenyl), 11.80 (s, 1H, N*H*); ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm) 31.7 (CH_3N), 55.4 (*p*-OCH_3-phenyl), 69.7, 70.3, 70.4, 79.3 (*C*, cp-ring), 92.6 (C=*C*–CO), 113.5 (2*C*, 3,5-phenyl), 128.5 (1*C*, 1-phenyl), 133.8 (2*C*, 2,6-phenyl), 161.5 (1*C*, 4-phenyl), 166.9 (*C*=C), 185.8 (*C*=O).

3.4. 1-3,5-Dimethoxyphenyl-3-methylamino-3-ferrocenyl-2-propenone (*2*)

The product was obtained as described in the general procedure as an orange solid (74%); Empirical formula: $C_{22}H_{23}NO_3Fe$; mp. 140 °C; IR ($CHCl_3$ solution, selected, cm^{-1}) 1320 (OMe), 1548 (C=C), 1592 (C=O), 3092 (N–H); Mass spectrum EI: *m*/*z* (%) = 405 (54) $[M]^+$, 340 (100) $[M–cp]^+$; ^1H NMR (300 MHz, $CDCl_3$, δ in ppm) 3.15 (d, 3H, *J* = 5.5 Hz, NCH_3), 3.85 (s, 6H, OMe), 4.24 (s, 5H, cp-ring), 4.43 (t, 2H, *J* = 1.9 Hz, cp-ring), 4.61 (t, 2H, *J* = 1.9 Hz, ring-cp), 6.16 (s, 1H, C=C*H*), 6.55 (t, 1H, *J* = 2.3 Hz, 4-phenyl), 7.07 (d, 2H, *J* = 2.2 Hz, 2,6-phenyl), 11.87 (s, 1H, N*H*); ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm) 31.8 (CH_3N), 55.6 (3,5-OCH_3-phenyl), 69.9, 70.3, 70.4, 78.7 (*C*, cp-ring), 93.2 (C=*C*–CO), 102.7 (1*C*, 4-phenyl), 104.7 (2*C*, 2,6-phenyl), 143.5 (1*C*, 1-phenyl), 160.7 (2*C*, 3,5-phenyl), 167.8 (*C*=C), 185.9 (*C*=O).

3.5. 1-m-Methylphenyl-3-methylamino-3-ferrocenyl-2-propenone (*3*)

The product was obtained as described in the general procedure as an orange solid (80%); Empirical formula: $C_{21}H_{21}NOFe$; mp. 71 °C; IR ($CHCl_3$ solution, selected, cm^{-1}) 1322 (OMe), 1592 (C=O), 3044 (N–H); Mass spectrum EI: *m*/*z* (%) = 359 (56) $[M]^+$, 294 (100) $[M–cp]^+$; ^1H NMR (300 MHz, $CDCl_3$, δ in ppm) 2.41 (s, 3H, CH_3), 3.14 (d, 3H, *J* = 5.8 Hz, NCH_3), 4.23 (s, 5H cp-ring), 4.42 (t, 2H, *J* = 1.9 Hz, cp-ring), 4.61 (t, 2H, *J* = 1.9 Hz, ring-cp), 6.19 (s, 1H, C=C*H*), 7.24 (d, 2H, *J* = 6.6 Hz, 4-phenyl), 7.32 (t, 2H, *J* = 7.4 Hz, 5-phenyl), 7.70 (d, 1H, *J* = 7.6 Hz, 6-phenyl), 7.73 (s, 1H, 2-phenyl), 11.90 (s, 1H, N*H*); ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm) 21.7 (*m*-CH_3-Phenyl), 31.8 (CH_3N), 69.9, 70.3, 70.4, 78.9 (*C*, cp-ring), 93.2 (C=*C*–CO), 123.9 (1*C*, 6-phenyl), 127.6 (1*C*, 5-phenyl), 128.2 (1*C*, 2-phenyl), 131.1 (1*C*, 4-phenyl), 137.9 (1*C*, 1-phenyl), 141.2 (1*C*, 3-phenyl), 167.5 (*C*=C), 186.8 (*C*=O).

3.6. 1-p-Ethylphenyl-3-methylamino-3-ferrocenyl-2-propenone (*4*)

The product was obtained as described in the general procedure as an orange solid (72%); Empirical formula: $C_{22}H_{23}NOFe$; mp. 50 °C; IR ($CHCl_3$ solution, selected, cm^{-1}) 1324 (OMe), 1539 (C=C), 1592 (C=O); Mass spectrum EI: *m*/*z* (%) = 373 (45) $[M]^+$, 308 (100) $[M–cp]^+$; ^1H NMR (300 MHz, $CDCl_3$, δ in ppm) 1.26 (t, 3H, *J* = 7.5 Hz, CH_2CH_3), 2.70 (*c*, 2H, *J* = 15.1 Hz, CH_2CH_3), 3.13 (d, 3H, *J* = 5.5 Hz, NCH_3), 4.23 (s, 5H, ring-cp), 4.42 (t, 2H, *J* = 1.9 Hz, ring-cp), 4.61 (t, 2H, *J* = 1.9 Hz, ring-cp), 6.20 (s, 1H, C=C*H*), 7.26 (d, 2H, *J* = 8.0 Hz, 3,5-phenyl), 7.83 (d, 2H, *J* = 8.3 Hz, 2,6-phenyl), 11.86 (s, 1H, N*H*); ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm) 15.49

(CH_2CH_3), 28.84 (CH_3N), 31.79 (CH_2CH_3), 69.8, 70.3, 70.4, 79.3 (*C*, cp-ring), 92.9 (C=*C*–CO), 126.9 (2*C*, 3,5-phenyl), 127.8 (2*C*, 2,6-phenyl), 138.6 (1*C*, 1-phenyl), 146.9 (1*C*, 4-phenyl), 167.2 (*C*=C), 186.6 (*C*=O).

3.7. 1-Phenyl-3-methylamino-3-ferrocenyl-2-propenone (**5**)

The product was obtained as described in the general procedure as an orange solid (78%); Empirical formula: $C_{20}H_{19}NOFe$; mp. 53 °C; IR ($CHCl_3$ solution, selected, cm^{-1}) 1591 (C=C), 1545 (C=O), 3089 (N–H); Mass spectrum EI: *m*/*z* (%) = 345 (71) $[M]^+$, 280 (100) $[M-cp]^+$; ^1H NMR (300 MHz, $CDCl_3$, δ in ppm) 3.15 (d, 3H, *J* = 5.2 Hz, NCH_3), 4.24 (s, 5H, cp-ring), 4.43 (t, 2H, *J* = 1.9 Hz, cp-ring), 4.62 (t, 2H, *J* = 1.9 Hz, ring-cp), 6.20 (s, 1H, C=C*H*), 7.43 (t, 3H, *J* = 3.3 Hz, phenyl), 7.90 (dd, 2H, *J* = 6.6 Hz, phenyl), 11.90 (s, 1H, N*H*); ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm) 31.8 (CH_3N), 69.6, 70.1, 70.6, 78.8 (*C*, cp-ring), 92.9 (C=*C*–CO), 126.7, 126.9, 128.2, 128.4, 131.0, 141.2 (*C*-phenyl), 167.6 (*C*=C), 186.5 (*C*=O).

3.8. 1-p-Methoxy-phenyl-3-prophylamino-3-ferrocenyl-2-propenone (**6**)

The product was obtained as described in the general procedure as an orange solid (72%); Empirical formula: $C_{23}H_{25}NO_2Fe$; mp. 43 °C; IR ($CHCl_3$ solution, selected, cm^{-1}) 2926 (C=C), 1592(C=O); Mass spectrum EI: *m*/*z* (%) = 403 (48) $[M]^+$, 338 (100) $[M-cp]^+$; ^1H NMR (300 MHz, $CDCl_3$, δ in ppm) 1.00 (t, 3H, *J* = 11.1 Hz, $CH_2CH_2CH_3$), 1.66 (*sex*, 2H, *J* = 10.7 Hz, $CH_2CH_2CH_3$), 3.44 (*c*, 2H, *J* = 19.3 Hz, $NCH_2CH_2CH_3$), 3.86 (s, 3H, OMe), 4.23 (s, 5*H* cp-ring), 4.40 (t, 2*H*, *J* = 2.8 Hz, cp-ring), 4.58 (t, 2*H*, *J* = 2.7 Hz, ring-cp), 6.18 (s, 1H, C=C*H*), 6.94 (d, 2H, *J* = 8.8 Hz, 3,5-phenyl), 7.90 (d, 2H, *J* = 8.8 Hz, 2,6-phenyl), 11.85 (s, 1H, N*H*). ^{13}C NMR (75 MHz, $CDCl_3$, δ in ppm) 11.7 ($CH_3CH_2CH_2N$), 24.1 ($CH_3CH_2CH_2N$), 46.7 ($CH_3CH_2CH_2N$), 55.4 (*p*-OCH_3-phenyl), 69.6, 70.3, 70.4, 79.7 (*C*, cp-ring), 92.5 (C=*C*–CO), 113.5 (2*C*, 3,5-phenyl), 128.5 (*C*, 1-phenyl), 128.6 (2*C*, 2,6-phenyl), 161.4 (1*C*, 4-phenyl), 166.1 (*C*=C), 184.2 (*C*=O).

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.07.017.

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