Synthesis, structure and characterization of some Schiff bases bearing phenylferrocene

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Some novel Schiff bases bearing phenylferrocene were synthesized by condensation reaction of 4-ferrocenylaniline with different aromatic aldehydes. The compounds prepared were characterized by spectroscopic methods (IR, UV-visible, 1 H and 13 C NMR) and elemental analysis. The single crystal analysis of compound F1 [monoclinic, space group, P2₁/c (no. 14), a = 19.858(2), b = 7.416(2), c = 12.095(5) Å, $\beta = 106.257(14)^\circ$] indicates a *trans* imine bond with a bond length of 1.270(2) Å, typical of a carbon-nitrogen double bond. Copyright © 2007 John Wiley & Sons, Ltd.

KEYWORDS: ferrocene; Schiff bases; phenylferrocene

INTRODUCTION

Since their discovery in 18641 Schiff bases have been the most thoroughly studied compounds in organic chemistry.² Extensive work has been carried out on the characterization of mono- and di-subsubstituted derivatives of ferrocene, as a typical organometallic species, and many publications have been devoted to organometallic derivatives of Schiff bases in ferrocenyl series.^{3–8} Ferrocene-containing ligands are of widespread interest in coordination chemistry as well.9 Ferrocene-based Schiff bases have been employed in various fields, such as biosensors, 10,11 asymmetric catalysis,12 polymer science as redox active polymers and dendrimers,¹³ and nonlinear optics.¹⁴ Their redox and electrical properties have resulted in this wide range of applications.¹⁵ In addition Schiff bases can also exhibit biological activity including antifungal,16 antiviral17 and anticancer activities.¹⁸ In this manuscript, we report the preparation and characterization (IR, ¹H and ¹³C NMR, UV-visible and elemental analyses) of some Schiff bases bearing phenyl ferrocene. The biological studies of these compounds are in progress.

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RESULTS AND DISCUSSION

Schiff bases bearing phenyl ferrocene (F1-F7) were synthesized using a reported method¹⁹ by condensing 4ferrocenylaniline with corresponding aldehydes (Scheme 1). The compound F1 was crystallized out and single crystal structure determination was performed (Fig. 1). The elemental analyses of all the products are in good agreement with the calculated values. The compounds F2 and F5 were reported earlier. 20,21 The I.R spectra of these products show all the characteristic peaks. A broad absorption band at 1591–1624 cm⁻¹ is assigned to $\nu_{C}=N$. The bands around $3100 \,\mathrm{cm}^{-1}$ can be attributed to aromatic ν_{CH} . In all these spectra the absence of ν_{N-H} bands at 3500-3300 cm $^{-1}$ and $v_{\rm C}$ bands at 1720–1660 cm⁻¹ reflects the formation of the products. A sharp band around 1000 and 1010 cm⁻¹ due to ferrocene is observed in the spectra of all compounds. An Fc-Cp stretching vibration is also seen around 480 cm⁻¹.

UV–visible studies were carried out in dichloromethane. All the synthesized Schiff bases bearing phenyl ferrocene are stable crystalline materials. They are all colored as normal for ferrocene-containing compounds, since these have highly intense absorptions in the 371–333 nm range assigned to the $n-\pi^*$ transition of azomethine group. Bands at higher energies 293–280 nm are associated with benzene $\pi-\pi^*$ transitions.²²

All the characteristic signals are observed in the ¹H NMR spectra of the synthesized Schiff bases. The incorporation of ferrocene is indicated by the signals observed due to ferrocene. The unsubstituted cylopentadienyl ring of



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Scheme 1. The synthesis of F1-F7.

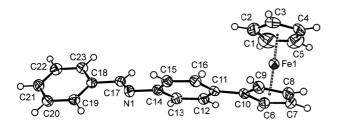


Figure 1. An ORTEP drawing of C₂₃H₁₉FeN (F1).

ferrocene gives a singlet in the range of 4.032–4.087 ppm for five hydrogens and the substituted one gives two triplets, at 4.344–4.379 ppm for two hydrogens and at 4.678–4.809 ppm for the other two hydrogens in agreement with the literature values. All the compounds show a sharp singlet around 7.046–8.706 ppm for azomethine proton in accordance with literature.²³

All signals in ¹³C NMR spectra of these Schiff bases can be assigned. Ferrocene gives three signals, one downfield signal of high intensity at about 69.19 ppm for the unsbstituted cyclopentadienyl ring with five carbons and another two at 68.80 and 66.40 ppm for the substituted cyclopentadienyl ring which are upfield. All these compounds show a downfield signal at about 159.22 ppm due to the presence of CH=N carbon.

The F1 crystallizes in a centrosymmetric space group $(P2_1/c, no. 14)$. An ORTEP view of the structure with numbering Scheme is shown in Fig. 1.

The molecule is composed of a ferrocene unit monosubstituted with a pendant benzylidene–phenyl–amine fragment. The planes defined by the C_5H_4 ring (C6–C10) and the phenyl group C11–C15 are non-coplanar and are twisted at an interplanar angle of 5.80(11)°. This most likely avoids unfavorable steric interactions between Hatoms bonded to C6, C9 and C12, C16. The ferrocene portion of trans-(η^5 – C_5H_5) $Fe(\eta^5$ – C_5H_4) – N=CH – C_6H_5 shows two virtually eclipsed cyclopentadienyl groups. The

rings C1–C5 and C6–C10 are nearly parallel with the least square planes at an angle of $2.46(14)^\circ$; the iron atom lies at 1.6477(12) and 1.6272(11) Å, respectively, from these planes. The C10–C11 bond connecting the ferrocene fragment and benzylidene–phenyl–amine fragment is essentially single [1.475(2) Å] and corresponds to a typical single bond between sp^2 carbons. This observation suggests negligible delocalization of electron density across this bond and into the phenyl group.

The C17–N1 imine bond is *trans* with a bond length of 1.270(2) Å, typical of carbon–nitrogen double bonds. The C14–N1 and C17–C18 distances 1.414(2) and 1.477(2) Å, respectively, indicate pure single bonds.

The observations noted above correlate well with a previous report by Coe *et al.* for the related 4-ferrocenyl-2'-methyl-4'-nitroazobenzene,²⁴ which shows a *trans* arrangement along the N=N bond, but contrasts with the structure of 1-ferrocenyl-2-(4-nitrophenyl)ethylene, showing a *cis* configuration for the C=C bond.²⁵

X-ray crystallography

Experimental crystalographic data for $C_{23}H_{19}FeN$ (F1) M=365.24, monoclinic, space group, $P2_1/c$ (no. 14), a=19.858(2), b=7.416(2), c=12.095(5) Å, $\beta=106.257(14)^\circ$, V=1710.0(9) Å³, Z=4, $D_c=1.419$ g cm⁻³, $\mu=8.86$ cm⁻¹, F(000)=760, λ (Mo $K_{\alpha})=0.71073$ Å, T=173(2) K, 6634 reflections collected $(\pm h, \pm k, \pm l)$, $[(\sin\theta)/\lambda]=0.648$ Å⁻¹, 3873 independent ($R_{int}=0.019$) and 3211 observed [$I>2\sigma(I)$], 226 refined parameters, R=0.0307, $wR_2=0.0801$, goodness of fit, S=1.019. Crystallographic experimental data is given in Tables 1 and 2.

EXPERIMENTAL

All the chemicals and reagents used for the synthesis of these Schiff bases were of highest purity



Table 1. Crystal data and structure refinement for C₂₃H₁₉FeN

Identification code	f1	
Empirical formula	$C_{23}H_{19}FeN$	
Formula weight	365.24	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 19.858(2) Å	$\alpha = 90^{\circ}$
	b = 7.416(2) Å	$\beta = 106.257(14)^{\circ}.$
	c = 12.095(5) Å	$\gamma = 90^{\circ}$.
Volume	$1710.0(9) \text{ Å}^3$	
Z	4	
Density (calculated)	$1.419 \ \text{mg} \ \text{m}^{-3}$	
Absorption coefficient	0.886 mm^{-1}	
F(000)	760	
Crystal size	$0.10 \times 0.07 \times 0.04 \text{ mm}^3$	
Theta range for data collection	4.04-27.43°.	
Index ranges	$-25 \le h \le 25, -9 \le k \le 8, -15 \le l \le 15$	
Reflections collected	6634	
Independent reflections	3873 [R(int) = 0.0190]	
Completeness to theta = 27.43°	99.3%	
Absorption correction	Multi-scan method	
Max. and min. transmission	0.9654 and 0.9167	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	3873/0/226	
Goodness-of-fit on F^2	1.019	
Final R indices $[I > 2\sigma(I)]$	$R1 = 0.0307, wR_2 = 0.0741$	
R indices (all data)	$R1 = 0.0416, wR_2 = 0.0801$	
Largest difference peak and hole	$0.275 \text{ and } -0.296 \text{ e Å}^{-3}$	

or purified by the standard methods of purification. Ferrocene, 4-nitroaniline, hydrochloric acid, hexadecyltrimethyl ammonium bromide, hydrazine monohydrate, 2-hydroxybenzaldehyde, 3-hydroxybenzaldehyde, 4-hydroxybenzaldehyde, 4-chlorobenzaldehyde, 4-methoxybenzaldehyde and 4-nitrobenzaldehyde were obtained from Fluka (Switzerland). Absolute ethanol, toluene, hexane, diethyl ether, acetone, chloroform, dichloromethane, ethyl acetate and petroleum ether were purchased from Merk (Germany). The syntheses of all the Schiff bases were conducted under inert atmosphere created by using vacuum line and dry argon gas.

Melting point determination

Melting point temperature of the compounds was determined using Mel-Temp, Mitamura Riken Kogyo Inc., Tokyo, Japan.

IR spectroscopy

The solid-state Fourrier transform infrared spectra (KBr pallets, $4000-400~{\rm cm}^{-1}$) were recorded on a Bio-Rad Excalibur FTIR, Model 3000 MX.

NMR spectroscopy

¹H NMR and ¹³C NMR spectral analyses were performed in CDCl₃ and recorded on a Bruker 300 MHz. Tetramethylsilane was used as an internal reference.

UV–*Visible spectroscopy*

UV-visible spectra of the compounds were recorded on 1601-Schimadzu in dichloromethane.

Elemental analysis

The elemental analyses were obtained from CHNS-932 LECO.

SYNTHESIS OF SCHIFF BASES BEARING PHENYLFERROCENE

General procedure

In a two-neck flask equipped with condenser and magnetic stirrer, (prebaked on vacuum to exclude any moisture) the corresponding aldehyde in 15–20 ml of absolute ethanol was refluxed for 15–20 min and then added a solution of 4-ferrocenylaniline in equimolar ratio in the same solvent.



Table 2. Selected bond lengths (Å) and angles (deg) for $C_{23}H_{19}FeN$

20 10	
Fe(1)-C(1)	2.028(2)
Fe(1)–C(6)	2.0390(19)
N(1)-C(17)	1.270(2)
N(1)-C(14)	1.414(2)
C(1)-C(2)	1.400(4)
C(1)-C(5)	1.422(4)
C(1)-H(1)	0.9500
C(2)-C(3)	1.393(3)
C(3)-C(4)	1.399(3)
C(4)-C(5)	1.412(4)
C(6)-C(7)	1.420(2)
C(10)-C(11)	1.475(2)
C(11)-C(12)	1.397(2)
C(12)-H(12)	0.9601
C(17)-C(18)	1.477(2)
C(17)-H(17)	0.9600
C(18)-C(23)	1.395(2)
C(19)-H(19)	0.9600
C(1)-Fe(1)-C(5)	41.01(11)
C(1)-Fe(1)-C(6)	106.80(10)
C(1)-Fe(1)-C(9)	150.46(10)
C(1)-Fe(1)-C(3)	67.83(11)
C(1)-Fe(1)-C(10)	116.08(10)
C(1)-Fe(1)-C(8)	166.78(10)
C(17)-N(1)-C(14)	119.89(14)
C(2)-C(1)-C(5)	107.4(2)
C(2)-C(1)-Fe(1)	70.00(12)

The reaction mixture was refluxed for 5–6 h under nitrogen atmosphere. After cooling the product was collected by filtration, washed with cold absolute ethanol, recrystallized from absolute ethanol and characterized by spectroscopic methods and elemental analyses.

Synthesis of *N*-(benzylidene)-4-ferrocenylaniline (F1)

N-(benzylidene)-4-ferrocenylaniline was synthesized by treating 4-ferrocenylaniline (0.5 g, 1.805 mmol) with benzaldehyde (0.18 ml, 1.805 mmol) in absolute ethanol as a solvent using the above procedure. Yield: 0.38g (76%). Anal. calcd For C₂₃H₁₉NFe: C, 75.62; H, 5.21; N, 3.83. Found: C, 74.73; H, 5.40; N, 3.73%. UV-visible [$\lambda_{\rm max}$ (CH₂Cl₂), nm]: 333, 290, 256, 237. IR (KBr, $\nu_{\rm max}$, cm⁻¹): 1519, 1624, 1167, 1104, 1031, 485. ¹H NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 7.046(s, 1H, CH=N), 7.49-7.55 (m, 5H, C₆H₅), 7.223 (d, 2H, C₆H₄, J = 8.7), 7.948 (d, 2H, C₆H₄, J = 8.6), 4.06 (s, 5H, C₅H₅), 4.355 (t, 2H, C₅H₄, J = 1.8), 4.688 (t, 2H, C₅H₄, J = 1.8). ¹³C NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 129.15 (C₆H₄), 126.60 (C₆H₄), 128.35 (C₆H₄), 137.37 (C₆H₄), 159.22 (C=NH), 69.19 (C₅H₅-Cp), 68.88 (C₅H₅-Cp), 66.45 (C₅H₄-Cp).

Synthesis of *N*-(2-hydroxybenzylidene)-4-ferrocenylaniline (F2)

N-(2-hydroxybenzylidene)-4-ferrocenylaniline was synthesized by treating 4-ferrocenylaniline (0.5 g, 1.805 mmol) with 2-hydroxybenzaldehyde (0.2 ml, 1.805 mmol) in absolute ethanol as a solvent using the above procedure. Yield: 0.375g (75%). Anal. calcd for C₂₃H₁₉NOFe: C, 72.44; H, 4.99; N, 3.67. Found: C,71.98; H, 5.16; N, 3.63%. UV-visible [λ_{max} , (CH₂Cl₂), nm]: 354, 284, 233, 228. IR (KBr, ν_{max} , cm⁻¹): 3445, 3087, 1620, 1108,1028, 496. ¹H NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 13.407 (s, 1H, Ar-OH), 8.706 (s, 1H, CH=N), 7.44 (d, 1H, C₆H₄, J = 7.8), 7.40 (d, 1H, C₆H₄, J = 7.8), 6.970–7.550 (m, 4H, C₆H₄), 4.087 (s, 5H, C₅H₅), 4.375 (t, 2H,C₅H₄, J = 1.8), 4.693 (t, 2H, C₅H₄, J = 1.8). ¹³C NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 126.75 (C₆H₄), 121.39 (C₆H₄), 145.95 (C₆H₄), 138.58 (C₆H₄), 161.21 (C=NH), 69.24 (C₅H₅-Cp), 69.08 (C₅H₅-Cp), 66.46 (C₅H₄-Cp).

Synthesis of *N*-(4-chlorobenzylidene)-4-ferrocenylaniline (F3)

N-(4-chlorobenzylidene)-4-ferrocenylaniline was synthesized by treating 4-ferrocenylaniline (0.5 g, 1.805 mmol) with 4-chlorobenzaldehyde (0.25 g, 1.805 mmol) in absolute ethanol as a solvent using the above procedure. Yield: 0.41g (82%). Anal. calcd for $C_{23}H_{18}NCIFe$: C, 69.08; H, 4.505; N, 3.504. Found: C, 68.48; H, 4.51; N, 3.60%. UV–visible [λ_{max} , (CH₂Cl₂), nm]: 343, 286. IR (KBr, ν_{max} , cm⁻¹): 3099, 1624, 1108, 1018, 489. ¹H NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 8.516 (s, 1H, CH=N), 7.44 (d, 2H, C_6H_4 , J=8.4), 7.206 (d, 2H, C_6H_4 , J=8.7), 7.881 (d, 2H, C_6H_4 , J=8.7), 4.053 (s, 5H, C_5H_5), 4.357 (t, 2H, C_5H_4 , J=1.8), 4.685 (t, 2H, C_5H_4 , J=1.8). ¹³C NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 126.70 (C₆H₄), 129.29 (C₆H₄), 149.37 (C₆H₄), 137.41 (C₆H₄), 157.84 (C=NH), 69.25 (C_5H_5 -Cp), 68.95 (C_5H_5 -Cp), 66.50 (C_5H_4 -Cp). IR (KBr, cm⁻¹): 3099, 1624, 1108, 1018, 489.

Synthesis of *N*-(4-methoxybenzylidene)-4-ferrocenylaniline (F4)

N-(4-chlorobenzylidene)-4-ferrocenylaniline was synthesized by treating 4-ferrocenylaniline (0.5 g, 1.805 mmol) with 4chlorobenzaldehyde (0.25 g, 1.805 mmol) in absolute ethanol as a solvent using the above procedure. Yield: 0.42 g (84%). Anal. calcd for C₂₄H₂₁NOFe: C, 72.91; H, 5.31; N, 3.54. Found: C, 71.56; H, 5.465; N, 3.65%. UV-visible [λ_{max} , (CH₂Cl₂), nm]: 341, 293, 244, 228. IR (KBr, v_{max} , cm⁻¹): 3092, 1601,1518, 2968, 2859, 1104, 1026. ¹H NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 8.479 (s, 1H, CH=N), 7.19 (d, 2H, C_6H_4 , J = 8.7), 7.517 (d, 2H, C_6H_4 , J = 8.7), 7.016 (d, 2H, C_6H_4 , J = 8.7), 7.894 (d, 2H, C_6H_4 , J = 8.7), 4.077 (s, 5H, C_5H_5), 4.344 (t, 2H, C_5H_4 , J = 1.8), 4.678 (t, $2H_1C_5H_4$, J = 1.8). ¹³C NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 132.35 (C₆H₄), 126.84 (C₆H₄), 149.92 (C₆H₄), 136.94 (C_6H_4) , 162.24 (C=NH), 69.20 (C_5H_5 -Cp), 69.10 (C_5H_5 -Cp), 66.37 (C₅H₄-Cp). IR (KBr, cm⁻¹): 3092, 1601,1518, 2968, 2859, 1104, 1026.



Synthesis of *N*-(4-hydroxybenzylidene)-4-ferrocenylaniline (F5)

N-(4-hydroxybenzylidene)-4-ferrocenylaniline was synthesized by treating 4-ferrocenylaniline (0.5 g, 1.805 mmol) with 4-hydroxybenzaldehyde (0.22 g, 1.805 mmol) in absolute ethanol as a solvent using the above general procedure. Yield: 0.45 g (90%). Anal. calcd for C₂₃H₁₉NOFe: C, 72.44; H, 4.99; N, 3.67. Found: C, 71.90; H, 5.10; N, 3.90%. UV-visible [λ_{max}, (CH₂Cl₂), nm]: 337, 291, 238, 214. IR (KBr, ν_{max} , cm⁻¹): 3094, 1591, 1520, 1103, 490. ¹H NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 9.069 (s, 1H, Ar-OH), 8.540 (s, 1H, CH=N), 7.194 (d, 2H, C₆H₄, J = 8.4), 7.589 (d, 2H, C₆H₄, J = 8.4), 6.98 (d, 2H, C₆H₄, J = 8.7), 7.859 (d, 2H, C₆H₄, J = 8.7), 4.052 (s, 5H, C₅H₅), 4.345 (t, 2H,C₅H₄, J = 1.8), 4.76 4.678 (t, 2H,C₅H₄, J = 1.8). ¹³C NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 126.72 (C₆H₄), 128.61 (C₆H₄), 126.61 (C₆H₄), 134.74 (C₆H₄), 160.73 (C=NH), 69.38 (C₅H₅-Cp), 68.82 (C₅H₅-Cp), 66.17 (C₅H₄-Cp).

Synthesis of *N*-(4-nitrobenzylidene)-4-ferrocenylaniline (F6)

N-(4-nitrobenzylidene)-4-ferrocenylaniline was synthesized by treating 4-ferrocenylaniline (0.5 g, 1.805 mmol) with 4-hydroxybenzaldehyde (0.27 g, 1.805 mmol) in absolute ethanol as a solvent using the above general procedure. Yield: 0.425 g (85%). Anal. calcd for C₂₃H₁₈N₂O₂Fe: C, 67.32; H, 4.39. Found: C, 66.48; H, 4.401%. UV-visible [λ_{max} (CH₂Cl₂), nm]: 371, 284, 251. IR (KBr, v_{max} , cm⁻¹): 3102, 1599, 1300, 1516, 1105, 501. UV-visible (λ_{max}, nm): 371, 284, 251. ¹H NMR (300 MHz, CDCl₃, Me₄Si, δ , ppm): 8.65 (s, 1H, CH=N), 7.55 (d, 2H, C_6H_4 , J = 8.4), 8.354 (d, 2H, C_6H_4 , J = 8.7), 7.268 (d, 2H, C_6H_4 , J = 8.1), 8.109 (d, 2H, C_6H_4 , J = 9.0), 4.081 (s, 5H, C_5H_5), 4.379 (t, 2H, C_5H_4 , J = 1.8), 4.70 (t, 2H, C_5H_4 , J = 1.8). ¹³C NMR (300 MHz, CDCl₃, Me₄Si, δ , ppm): 129.29 (C_6H_4) , 124.06 (C_6H_4) , 126.81 (C_6H_4) , 142.20 (C_6H_4) , 159.23 (C=NH), 69.71 (C_5H_5-Cp) , 69.28 (C_5H_5-Cp) , 66.50 (C_5H_4-Cp) Cp).

Synthesis of *N*-(3-hydroxybenzylidene)-4-ferrocenylaniline (F7)

N-(3-hydroxybenzylidene)-4-ferrocenylaniline was synthesized by treating 4-ferrocenylaniline (0.5 g, 1.805 mmol) with 3-hydroxybenzaldehyde (0.22 g, 1.805 mmol) in absolute ethanol as a solvent using the above general procedure. Yield: 0.36 g (72%). Anal. calcd for C₂₃H₁₉NOFe: C, 72.44; H, 4.99. Found: C, 71.15; H, 5.23%. IR (KBr, $\nu_{\rm max}$, cm⁻¹): 3387, 2966, 1624, 1584, 1104,1032, 490. ¹H NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 9.719 (s, 1H, Ar-OH), 8.600 (s, 1H, CH=N), 7.320–7.390 (m, 3H, C₆H₄), 7.225 (d, 2H, C₆H₄, J = 8.1), 6.937 (d, 2H, C₆H₄, J = 8.4), 7.576 (d, 2H, C₆H₄, J = 8.4), 4.032 (s, 5H, C₅H₅), 4.354 (t, 2H,C₅H₄, J = 1.8), 4.809 (t, 2H,C₅H₄, J = 1.8). ¹³C NMR (300 MHz, CDCl₃, Me₄Si, δ, ppm): 130.31 (C₆H₄),

120.69 (C_6H_4), 114.61 (C_6H_4), 149.35 (C_6H_4), 160.00 (C=NH), 69.84 (C_5H_5 -Cp), 69.41 (C_5H_5 -Cp), 66.65 (C_5H_4 -Cp).

CONCLUSION

The Schiff bases bearing phenyl ferrocene have been successfully synthesized and characterized. First the ferrocene was reacted with 4-nitroaniline to get 4-nitrophenyl ferrocene, which was then reduced by Pd-C/hydrazine monohydrate to get 4-ferrocenylaniline. This was then condensed with corresponding aromatic aldehydes to get different Schiff bases bearing phenyl ferrocene in quantitative yield. All these compounds are stable and soluble in most organic solvents. Study of the biological activities of these compounds is in progress and will be reported later.

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