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Some ferrocenyl Schiff bases with nonlinear optical properties

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Eleven new ferrocenyl Schiff bases containing a benzene ring and C═N in a conjugating chain have been synthesized by facile methods and characterized by ^{1}H NMR, elemental analysis and fast atom bombardment mass spectrometry. Cyclic voltammetry was used to determine their electrochemical properties and electron-withdrawing effects. It is shown that these pull–push ferrocenyl compounds have nonlinear optical responses according to their electronic absorption spectra and powder second harmonic generation measurements. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: ferrocenyl Schiff base; NLO; electronic absorption spectra; cyclic voltammetry; powder SHG

INTRODUCTION

Materials with nonlinear optical (NLO) responses have a promising future in the optical field.[1–3] They can be used in the fields of telecommunications, lasers, processing optical information and optical transferring, *etc*. A molecule containing a pushing moiety and a pulling moiety in a conjugating system may have large second-order optical nonlinearities.[4] Green *et al*.[5] first reported a large second-order optical nonlinearity of organometallic compounds containing a ferrocenyl group. Actually, the ferrocenyl group is an excellent π-donor in conjugating chains. From then on, research on ferrocenyl NLO materials has became popular.[6–8] Experimental and theoretical studies on organic chromophores indicate that the presence of hetero-aromatics in the conjugating chains is greatly beneficial to the observation of large NLO properties.[9] Thomas *et al*.[10] has reported new ferrocenyl derivatives based on donor–acceptor complexes with large NLO responses containing thiophene and furan in the conjugating chain. Furthermore, the larger a conjugating system, the larger the NLO responses.[11] Bella *et al*.[2] described a series of Schiff base complexes having NLO properties. Recently, Pal *et al*.[12] have studied ferrocenyl Schiff bases for NLO responses. In the present work, we prepared some of ferrocenyl derivatives with C═N, C═C and a benzene ring in a conjugating chain and monitored their NLO responses. In order to examine their π-donor–acceptor interactions, solvatochromic and electrochemical studies have also been performed.

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EXPERIMENTAL

General

All the reagents were dried by standard methods. Aromatic aldehydes were obtained according to the literature methods.[13,14] The UV spectra were recorded on a Shimadzu UV-260 spectrometer. IR spectra were obtained using KBr pellets on a Nicolet 179SX FT-IR spectrometer. ^{1}H NMR spectra were obtained using an FT-400 spectrometer using $CDCl_3$ as solvent and tetramethylsilane as an internal standard. Mass spectra were obtained on a ZAB-HS mass spectrometer by fast atom bombardment (FAB, MASPEC II data base). Elemental analysis was measured on a Carlo Erba 1106 instrument. Cyclic voltammetric experiments were performed on a CH-2 electrochemical analyzer equipped with a three-electrode assembly with 0.1 M *n*-Bu_4NClO_4 as the supporting electrolyte and CH_2Cl_2 as solvent. The working electrode was a platinum disk. The reference electrode was a KCl saturated

calomel. The solutions were saturated and blanketed with nitrogen before the first scan. The scale of potentials was calibrated *via* the FcH/FcH^+ couples. The melting points of all products were monitored by an uncorrected thermometer. Powder second-harmonic generation (SHG) efficiencies were determined by using the 1097 nm (SH at 953.5 nm) output of a Q-switched Nd : YAG laser. The powder SHG measurements were made using the modified Kurtz method.[15] This is a nonlinear process in which two photons of frequency ν are corrected to a single photon of frequency 2ν. This requires a strong electric field and a noncentrosymmetric medium. The SHG process is one of the most efficient ways to shorten the wavelength of light. A dual-beam system (with a urea sample as a reference beam) was used to normalize the SHG signals for laser shot-to-shot fluctuations. Pulse energies used were in the range of $100–500 \times 10^{-6}$ J with spot sizes of 2–3 mm. The diffusely backscattered SH signals were collected and isolated by using filters and a monochromator and detected with photomultiplier tubes (Hamanmatsu R406) whose outputs were amplified and integrated by using 10 ns gate widths. Samples were ground, unsized microcrystalline powders; the particles were griddled by a 200 mesh sieve.

Synthesis of 3a–3f

To a solution of the amine (0.005 mol) in absolute ethanol (10 ml) was added several drops of acetic acid. Over a period of 10 min the aromatic aldehyde (0.005 mol) in 10 ml of absolute ethanol was added dropwise at room temperature. The precipitate was filtered and washed with 2×5 ml ethanol and recrystallized from ethanol.

Synthesis of 3g–3k[16]

To a solution of the amine (0.005 mol) in absolute ethanol (10 ml) was added several drops of acetic acid. Over a period of 10 min the aromatic aldehyde (0.005 mol) in 10 ml of absolute ethanol was added dropwise at room temperature. Then the solution was refluxed 1–8 h (monitored by thin-layer chromatography). The product was isolated by column chromatography in silica using petroleum ether/ether (5 : 1) as eluant.

RESULTS AND DISCUSSION

All of these ferrocenyl Schiff bases are prepared by the condensation between a ferrocenyl amine and an aldehyde or an amine and a ferrocenyl aldehyde, as shown in Scheme 1.

The condensation reaction between the ferrocenyl amine and aldehyde for preparing compounds **3a–f** was easy to carryout at room temperature, but the reaction between the amine and ferrocenyl aldehyde for preparing **3h–k** was difficult and it must be performed under reflux. Compound **3g** was also obtained under reflux condition due to *o*-nitro hindrance. All products were characterized by ^{1}H NMR, FAB mass spectrometry (MS), IR and elemental analyses and these data are shown in Tables 1 and 2.

Table 1. ^{1}H NMR data (δ, ppm) for compounds **3a–3k**

Compound	C_5H_5	C_5H_4	C_6H_4 or C_6H_5	CH=CH	FcC_6H_4	CH=N
3a	4.05 (s, 5H)	4.38 (t, 2H), 4.69 (s, 2H)	7.15 (m, 4H)		7.51 (m, 4H)	8.71 (d, 1H)
3b	4.06 (s, 5H)	4.33 (s, 2H), 4.67 (s, 2H)	7.17 (t, 1H), 7.19 (t, 2H), 7.58 (d, 2H)	7.36 (d, 1H, $J = 16.0$ Hz), 7.40 (q, 1H, $J = 16.0$ Hz)	7.40 (d, 2H), 7.49 (d, 2H)	8.36 (d, 1H)
3c	4.06 (s, 5H)	4.35 (s, 2H), 4.68 (s, 2H)	7.67 (t, 1H), 8.27 (d, 1H), 8.32 (d, 1H), 8.76 (s, 1H)		7.22 (d, 2H), 7.53 (d, 2H)	8.62 (s, 1H)
3d	4.05 (s, 5H)	4.53 (s, 2H), 4.66 (s, 2H)	7.47 (d, 2H), 8.86 (d, 2H)		7.20 (d, 2H), 7.52 (d, 2H)	8.50 (s, 1H)
3e	4.05 (s, 5H)	4.36 (s, 2H), 4.66 (s, 2H)	7.70 (d, 2H), 8.27 (d, 2H)	7.21 (q, 1H, $J = 18.0$ Hz), 7.24 (d, 1H, $J = 18.0$ Hz)	7.26 (d, 2H), 7.51 (d, 2H)	8.43 (d, 1H)
3f	4.06 (s, 5H)	4.36 (s, 2H), 4.68 (s, 2H)	8.10 (d, 2H), 8.33 (d, 2H)		7.24 (d, 2H) 7.53 (d, 2H)	8.63 (s, 1H)
3g	4.06 (s, 5H)	4.35 (s, 2H), 4.67 (s, 2H)	7.54 (t, 1H), 7.67 (t, 1H), 7.71 (d, 1H), 8.04 (d, 1H)	7.13 (q, 1H, $J = 17.9$ Hz), 7.60 (d, 1H, $J = 18.0$ Hz)	7.19 (d, 2H), 7.51 (d, 2H)	8.42 (d, 1H)
3h	4.06 (s, 5H)	4.43 (t, 2H), 4.74 (t, 2H)	7.89 (m, 4H)		7.57 (m, 4H)	8.48 (s, 1H)
3i	4.06 (s, 5H)	4.42 (t, 2H), 4.74 (t, 2H)	7.22 (d, 1H), 7.83 (d, 2H)		7.39 (d, 2H), 7.59 (d, 2H)	8.41 (d, 1H)
3j	4.05 (s, 5H)	4.40 (t, 2H), 4.73 (t, 2H)	7.13 (d, 2H), 7.79 (d, 2H)		7.53 (d, 4H)	8.40 (s, 1H)
3k	4.06 (s, 5H)	4.41 (s, 2H), 4.72 (s, 2H)	7.84 (d, 4H)		7.18 (d, 2H), 7.61 (d, 2H)	8.44 (d, 1H)

Table 2. The yields, melting points, elemental analyses, IR spectra and mass spectra of **3a–3k**

Compound	Yield (%)	M. p. (°C)	Found (Calc.) (%) C	H	N	IR (cm^{-1})	Mass spectra m/z (%)
3a	94	176–178	72.16 (72.44)	5.44 (5.43)	3.65 (3.67)	3082.4m, 3041.3w, 1618.3s, 1282.5s, 843.9, 818.4s	381 (M^+, 50), 277 (52), 185 (100)
3b	97	172–174	76.68 (76.72)	5.67 (5.41)	3.53 (3.58)	3091m, 1618.0vs, 1518.1vs, 844.7s, 815.44m	390.8 (M^+, 100), 277 (40), 185 (80)
3c	96.5	134–136	67.18 (67.32)	4.71 (4.43)	6.93 (6.83)	3079m, 1635.0m, 849.5s, 816.3m	410 (M^+, 53), 364 (36), 185 (87), 180 (50)
3d	98	202–204	68.87 (69.10)	4.57 (4.54)	3.53 (3.51)	3087w, 1620.0m, 846.6s, 818.0m	399 (M^+, 36), 401 (M + 2, 18), 364 (45), 185 (78), 179 (24)
3e	98.5	219–221	68.59 (68.82)	4.63 (4.62)	6.40 (6.42)	3079w, 1597.9s, 1512.8vs, 856.2s, 820.0s	436 (M^+, 18), 290 (100), 205 (34), 185 (83)
3f	99	244–246	67.32 (67.32)	4.43 (4.43)	6.83 (6.83)	3079s, 1589.7s, 858.6s, 848.8s	410 (M^+, 27), 364 (18), 185 (65), 180 (30)
3g	98	198–200	68.94 (68.82)	4.39 (4.62)	6.53 (6.42)	3099w, 1604.9s, 1524.7vs, 846.23s, 822.35m	436 (M^+, 75), 290 (83), 205 (40), 185 (77)
3h	94	173–174	66.71 (67.32)	4.55 (4.43)	7.01 (6.83)	3088.9w, 2921.3m, 1598.3s, 1524.5vs, 834.4s, 819.0s	410.0 (M^+, 65), 364 (78), 185 (36), 179 (45)
3i	80	190–192	68.92 (69.10)	4.44 (4.54)	3.68 (3.51)	3087.4w, 2868.9m, 1604.8vs, 1561.6vs, 836.5s	399 (M^+, 66), 401 (M + 2, 31), 364 (23), 185 (47), 179 (24)
3j	81	162–164	62.35 (62.30)	4.10 (4.43)	3.16 (6.83)	2852.2m, 1599.0vs, 1562.1vs, 833.3vs, 819.0s	443 (M^+, 39), 445 (M + 2, 41), 363 (30), 185 (47), 179 (25)
3k	75	212–214	67.48 (67.32)	4.52 (4.43)	6.88 (6.83)	3089w, 2963s, 1616s, 1595.2vs, 1219.2s, 835.3m, 815m	410 (M^+, 52), 364 (38), 185 (90), 179 (28)

$$R^1NH_2 \quad (\mathbf{1}) \quad + \quad R^2CHO \quad (\mathbf{2}) \quad \xrightarrow{\text{EtOH, HOAc}} \quad R^1N{=}CHR^2 \quad (\mathbf{3})$$

R^1 = *p*-Fc-$C_6H_4^-$; R^2 = *o*-$HOC_6H_4^-$ (**3a**),
$C_6H_5CH{=}CH$ (**3b**), *m*-$O_2NC_6H_4$ (**3c**),
p-Cl-$C_6H_4^-$ (**3d**), *p*-$O_2NC_6H_4CH{=}CH$ (**3e**),
p-$O_2NC_6H_4^-$ (**3f**), *o*-$O_2NC_6H_4CH{=}CH$ (**3g**)

R^2 = *p*-Fc-$C_6H_4^-$; R^1 = *m*-$O_2NC_6H_4$ (**3h**),
p-Cl-$C_6H_4^-$ (**3i**), *p*-$BrC_6H_4^-$ (**3j**), *p*-$O_2NC_6H_4^-$ (**3k**)

Scheme 1.

^{1}H NMR spectra

The ^{1}H NMR spectra of products **3b**, **3g** and **3e** indicated the presence of *cis* and *trans* isomers according to the literature,[17] because we found four groups of signals and their coupling constants are 16–18 H_3 (important product) and 9–11 H_3 (trace) respectively. No attempt was made to separate the isomers, but we converted the isomeric mixtures to the *trans* isomer according to a previously reported method.[9]

There are three separate signals for the ferrocenyl group. The chemical shift of the five protons of the unsubstituted cyclopentadienyl ring is observed at 4.05–4.06 ppm as a sharp singlet. The signals of protons of the *mono*-substituted cyclopentadienyl ring appear at 4.33–4.36 (2H) and 4.66–4.68 ppm (2H) as triplet corresponding to the spectrum of an A_2B_2 pattern. One of the vinyl protons appears as a doublet at 7.1–7.6 ppm. Their coupling constants (14–18 Hz) indicate the expected *trans* isomer. The proton signal in CH=N appears at 8.36–8.63 ppm. The signals of both vinyl protons, CH=N and the protons of the *mono*-substituted cyclopentadienyl ring are considerably downfield due to the presence of nitrogen, the conjugating system and a π-acceptor. The important IR absorptions of products at about 1600 cm^{-1} are assigned to ν(C=N). The ferrocenyl perpendicular C–H bend of cyclopentadienyl ring appears at about 820 cm^{-1}.

Electronic absorption spectra

Designed electronic absorption spectra were used in determining the NLO responses according to the literature.[9] The electronic absorption data for these compounds are listed in Table 3. There are two important reasons for this dot.

Firstly, it is necessary to know the transparency regions. Secondly, it indicates high molecular potential bulk NLO properties. There are two pre-eminent absorption bands in the transparent and the UV region. This is typical of metal-to-ligand charge transfer (MLCT) excitation for the transparent region and *d*–*d* character for the UV region. The stronger the acceptor withdraws, the closer the absorption is to longer wave. So, the compounds containing NO_2 possess an MLCT absorption at lower energy than those of the others. Furthermore, the larger the conjugating system is, then the stronger the effect is. For example, the data listed in Table 3 are consistent with this theory. The hydroxyl group is a II donor, and hence the electronic absorption of **3a** is closer to the UV region than that of the other compounds.

The solvatochromism properties[9] are listed in Table 4. It was observed that all the products showed a large red shift in dimethyl sulfoxide. This result is attributed to the high polarity of the solvent. However, both absorption and solvatochromism data indicate that the position of nitrogen, whether it is close to the ferrocenyl group or not, does not affect the electronic absorption spectrum.

Electrochemistry

All compounds showed a ferrocenium–ferrocene redox wave with a half-wave potential of 0.47–0.53 V. The potential values, as shown in Table 5, are affected significantly by the nature of the substituents. The –Ph–CH=N–R or –Ph–N=CH–R substituents behave as electron-withdrawing groups, so the corresponding substituted ferrocenes are less readily oxidized than ferrocene. The hydroxyl group displays –I and +R effects, but the predominance of the electron-accepting inductive character means that the

Table 3. Electronic absorption data[a] and powder SHG efficiencies for compounds **3a**–**3k**

	3a	**3b**	**3c**	**3d**	**3e**	**3f**	**3g**	**3h**	**3i**	**3j**	**3k**
λ_{max} (nm)	359	361	359	367	359	352	377	354	354	354	352
	458	461	458	465	481	454	485	470	462	465	467
$10^{-3}\epsilon$ (l mol^{-1} cm^{-1})	17.56	16.44	17.56	13.00	10.00	15.92	18.32	14.40	13.11	12.44	10.66
	2.72	2.92	2.72	3.24	2.44	2.64	4.48	2.56	2.34	2.86	2.72
SHG value[b]	13	13	60	25	95	75	190	95	30	20	110

[a] Data measured for dichloromethane solution of concentration 5×10^{-5} mol l^{-1}.
[b] SH intensity measured with use of 1907 nm fundamental radiation. All the values reported are relative to use of urea as reference (particle size <200 mesh).

Table 4. Solvatochromic data (nm) for compounds **3a**–**3k**

Compound	Et_2O	MeCN	Me_2CO	THF	MeOH	CH_2Cl_2	DMF	DMSO
3a	319	324	337	346	351	359	360	401
	417	425	429	434	440	458	459	495
3b	330	338	349	359	360	361	366	488
	429	439	443	454	458	461	473	536
3c	320	329	344	351	355	359	358	406
	422	426	438	449	457	458	451	529
3d	333	347	355	363	366	367	372	434
	423	429	443	449	463	465	449	538
3e	309	337	341	347	359	360	353	443
	437	454	466	470	476	481	477	599
3f	317	332	337	340	350	352	358	426
	431	438	447	448	455	454	461	537
3g	358	362	364	366	373	377	382	427
	448	462	474	475	485	485	491	595
3h	317	321	327	333	345	354	359	422
	429	433	436	442	459	470	473	533
3i	310	318	323	329	340	354	361	411
	418	427	431	440	459	462	465	529
3j	313	321	324	327	342	354	359	413
	421	427	431	439	461	465	469	532
3k	327	329	334	341	347	352	355	424
	439	441	449	452	463	467	477	543

Table 5. Electrochemical data for the oxidation of Schiff base measured at 25 °C in 0.1 M [NBu_4] ClO_4–CH_2Cl_2 solution[a]

Compound	$E_{p,a}$ (V)	$E_{p,c}$ (V)	ΔE_p (V)	$E_{1/2}$ (V)	$I_{p,a}$ (μA)	$I_{p,c}$ (μA)	$I_{p,a}/I_{p,c}$
3a	0.590	0.410	0.180	0.500	1.240	1.330	0.933
3b	0.540	0.410	0.130	0.475	0.728	0.761	0.956
3c	0.560	0.420	0.140	0.490	0.765	0.799	0.957
3d	0.590	0.390	0.200	0.490	1.440	1.590	0.900
3e	0.580	0.390	0.190	0.485	1.290	1.280	1.000
3f	0.570	0.420	0.150	0.495	0.943	1.010	0.933
3g	0.550	0.410	0.140	0.480	0.873	0.917	0.952
3h	0.580	0.490	0.090	0.535	0.275	0.283	0.971
3i	0.560	0.470	0.090	0.515	1.060	1.110	0.955
3j	0.560	0.480	0.080	0.520	1.110	1.130	0.982
3k	0.540	0.470	0.080	0.515	1.180	1.140	0.991
FcH	0.479	0.388	0.088	0.434	—	—	1.073

[a] $E_{p,a}$ and $E_{p,c}$ are the anodic and cathodic peak potentials, $I_{p,a}$ and $I_{p,c}$ are the anodic and cathodic peak currents, ΔE_p is the peak-to-peak potential splitting $E_{p,a}-E_{p,c}$, and $E_{1/2}$ is the half-wave potential for the successive one-electron oxidation of these compounds. Scan rate 0.05 V s^{-1}.

oxidation potential of the corresponding complex **3a** is greater than that of ferrocene. The inductive effects of the strongly electron-withdrawing $-NO_2$ and $-X$ groups lead to the ferrocenyl derivatives (**3d**–**3k**) having relatively high oxidation potentials and the electronic effect increases in the order $-Cl > -Br > -NO_2$ (**3h**, **3i**, **3j**, **3k**). The position of nitrogen in the conjugation chain is not the key factor affecting the potentials of these compounds.

Powder SHG measurement

In order to determine the effect of chain length and aromatic substituent patterns on the second-order nonlinear susceptibilities of these compounds, we examined their powder SHG efficiencies. The SHG values indeed varied as a function of chain length and substitution pattern (Table 3). The SHG signals varied from 13 to 190 times that of the urea reference. Strong electron-withdrawing groups such as $-NO_2$ (**3c**, **3e**, **3f**, **3g**, **3h**, **3k**) result in large SH efficiencies with values at least 60 times as large as that of the urea reference. The largest SHG value was found for compound **3g**, which has an $-NO_2$ substituent and a long chain. The large SH efficiencies imply large components of the χ, $\chi^{(2)}$ susceptibility, which could lead to large electrooptic coefficients, where $\chi^{(n)}$ is the nth-order NLO susceptibility in the equation $P = P_0 + \chi^{(1)}E + \chi^{(2)}E + \chi^{(3)}E + \ldots$, i.e. the macroscopic quantities of interest are defined.[15,18] For large SH efficiency[8] it is necessary that a compound contains a good electron acceptor and a long chain. In contrast, for a compound whose benzene ring has a donor group (as in compound **3a**) or is without any substituent (as in compound **3b**) the SH efficiencies are only as large as 13 times that of the urea sample.

Briefly, a series of pull–push ferrocenyl compounds containing a C═N double bond, a C≡C and a benzene ring have been synthesized successfully and their spectra and electrochemistry studied. All the results indicate that compounds with strong electron-withdrawing groups generate NLO responses. The greater the π-donor acceptor interaction, the stronger is the NLO response. Compounds with *o*-, *m*-, *p*-nitro groups have excellent NLO responses, especially that with *o*-nitro.

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