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A facile synthesis of ferrocene grafted *N*-methyl-spiropyrrolidines through 1,3-dipolar cycloaddition of azomethine ylides

A. R. Sureshbabu [a], R. Raghunathan [a,*], B. K. Satiskumar [b]

[a] Department of Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India
[b] Department of Physics, Presidency College, Chennai 600 005, India

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ABSTRACT

One-pot synthesis of novel ferrocene grafted *N*-methyl-spiropyrrolidines has been accomplished in good yields through a facile 1,3-dipolar cycloaddition reaction of various azomethine ylide derived from ninhydrin and sarcosine with various ferrocene derivatives as dipolarophile. The regiochemical and stereochemical outcome of the cycloaddition reaction is ascertained by X-ray crystallographic studies of one of the cycloadducts.

Ferrocene, an organometallic compound, was discovered in the early 1950s[1] and since then, there has been enormous growth in ferrocene chemistry.[2] Ferrocene is employed as a component for redox-active chemical sensors for voltametric detection of cations[3] as well as anions,[4] metal-containing signaling probes for the detection of estrogen receptors,[5] dinucleotides,[6] and DNA hybridization events[7] thus opening the way to DNA and gene sensors.[8] Ferrocene materials are used as asymmetric catalysts,[9] liquid crystals,[10] conductive,[11] magnetic,[12] and optical devices[13] and as electron transfer devices.[14] Ferrocene derivatives offer advantages over other organometallics due to their synthetic versatility and thermal and photochemical stability. Ferrocene-substituted organic molecules hold great potential due to their biological activity. Ferrocene derivatives have been used for the treatment of malaria and cancer.[15–18] Many ferrocene-based heterocycles are known to exhibit anti-bacterial and anti-fungal properties.[19–21] Hence, there has been renewed interest in the synthesis of ferrocene-based heterocycles.

The intermolecular [3+2]-cycloaddition reaction of azomethine ylides with olefinic and acetylenic dipolarophiles has resulted in a number of novel heterocyclic scaffolds which are particularly useful for the creation of diverse chemical libraries of drug-like molecules for biological screening.[22,23] Pyrrolidines are the central skeleton for numerous alkaloids and constitute classes of compounds with significant biological activity.[24,25] Spiroheterocycles are an important class of naturally occurring substances characterized by highly pronounced biological properties.[26] Recently, we reported on the bioactivity of several spiropyrrolidines.[27]

As part of ongoing program on the synthesis of complex novel spiropyrrolidines/pyrrolizidines,[28,29] herein we report an expeditious and facile protocol for the synthesis of novel ferrocene grafted *N*-methyl-spiroindanedione-pyrrolidines. The reaction of various ferrocene derivatives[30] **1a–e**, **5a–b**, **7**, **9a–b,** and **11** with the azomethine ylide generated from ninhydrin **2** and sarcosine **3** afforded a series of novel monospiroindanedionepyrrolidines **4a–e**, **6a–b**, **8**, **10a–b,** and **11**.[31] The structures of the ferrocenyl monospiroheterocycles were confirmed through spectral and elemental analysis.[32] The reactions were carried out under two different conditions and the results are given in Table 1.

Scheme 1 depicts the mechanism for the generation of azomethine ylide from ninhydrin **2** and sarcosine **3**. Schemes 2–6 depict the one-pot, three component reactions involving ninhydrin, sarcosine, and various ferrocene derivatives for the synthesis of novel ferrocenyl monospiroindanedionepyrrolidines.

The ferrocene-derived dipolarophiles namely 1-ferrocenyl-3-phenyl-prop-2-ene-1-one derivatives **1a–e** underwent smooth reaction with non-stabilized azomethine ylide generated from ninhydrin **2** and sarcosine **3** in refluxing acetonitrile affording the spiroindanedionepyrrolidines **4a–e** in good yield (Scheme 2). The formations of the cycloadducts **4a–e** were confirmed by spectral and elemental analysis. Thus, the IR spectrum of 1-*N*-methyl-spiro-[2.2′]-indane-1′,3′-dione-3-phenyl-4-ferrocenoyl-pyrrolidine **4a**, exhibited peaks at 1665 and 1740 cm^{-1} due to ferrocenyl and

* Corresponding author. Tel.: +91 044 22202811.
E-mail address: ragharaghunathan@yahoo.com (R. Raghunathan).

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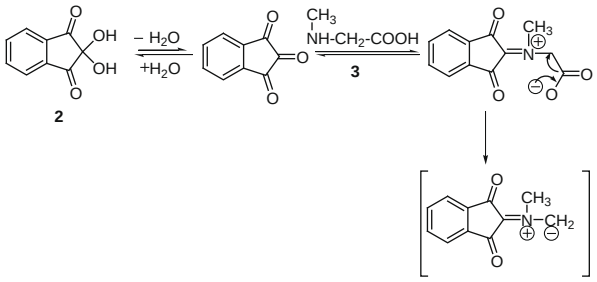
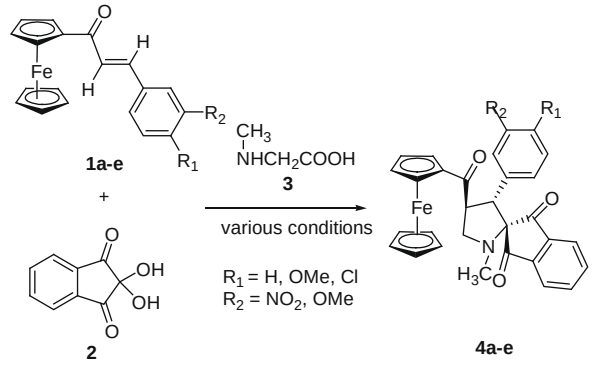
Table 1
1,3-Dipolar cycloaddition reaction of ferrocene-derived dipolarophiles **1a–b**/**5a–b**/**7**/**9a–b**/**11** with ninhydrin **2** and sarcosine **3**

Product	R_1	R_2	X	Method A		Method B		Melting point (°C)
				T (h)	*Y* (%)	*T* (h)	*Y* (%)	
4a	H	—	—	4.2	75	—	—	162–164
4b	OMe	—	—	5.0	70	11	40	183–185
4c	Cl	—	—	4.0	76	10	48	176–178
4d	—	NO_2	—	5.0	88	10	60	184–186
4e	OMe	OMe	—	5.0	60	12	40	141–143
6a	—	—	O	3.4	86	—	—	161–165
6b	—	—	S	4.8	68	12	55	120–122
8	—	—	—	3.5	80	—	—	207–209
10a	H	—	—	4.3	70	10	52	181–183
10b	OMe	—	—	5.2	66	—	—	207–209
12	—	—	—	5.0	70	—	—	193–195

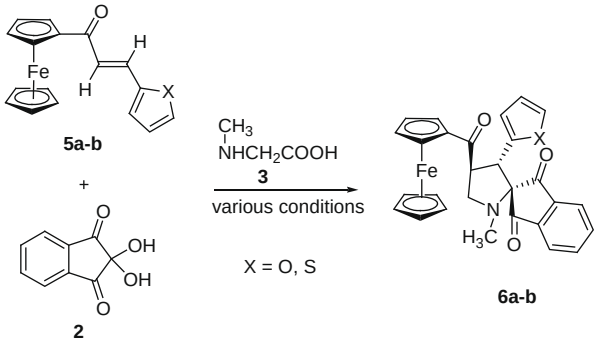
T (h) = time in hour, *Y* (%) = yield in percentage.
Method A: acetonitrile/reflux, Method B: toluene/reflux.



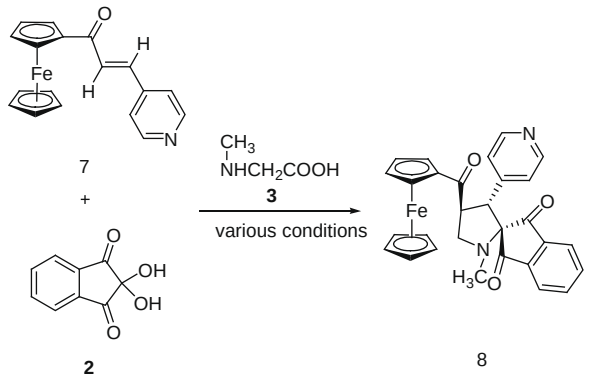
Scheme 1.



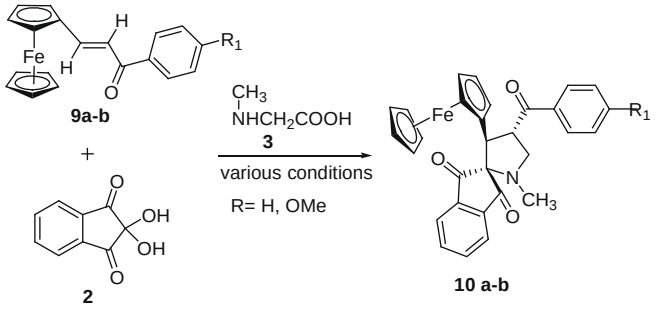
Scheme 2.



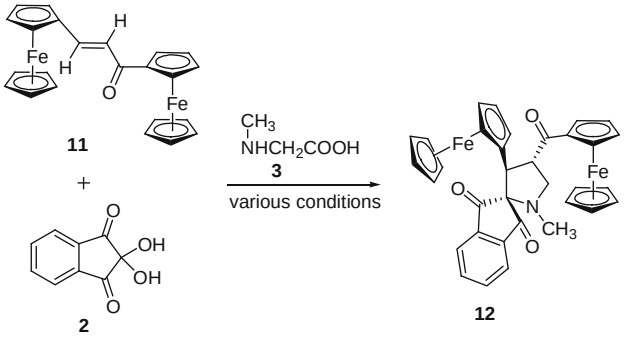
Scheme 3.



Scheme 4.



Scheme 5.



Scheme 6.

indanedione ring carbonyls. The ^{1}H NMR spectrum of **4a** exhibited peaks at δ 2.34 (s, 3H), 3.44 (t, 1H, J = 7.0 Hz), 3.74 (t, 1H, J = 8.4 Hz), 3.96 (s, 5H), 4.44–4.46 (m, 1H), 4.52 (d, 1H, J = 10.3 Hz), 4.48 (s, 2H), 4.76 (s, 2H), 6.54–7.86 (m, 9H). The –NCH_3 proton exhibited a singlet at δ 2.34. The –NCH_2 protons of the pyrrolidine ring appeared as triplets at δ 3.44 and δ 3.74. The pyrrolidine ring proton attached to the ferrocenoyl moiety appeared as a multiplet in the region δ 4.44–4.46 whereas the pyrrolidine ring proton attached to the aryl unit appeared as a doublet at δ 4.52 with the coupling constant value J = 10.3 Hz. The ferrocenyl protons exhibited singlets at δ 3.96, δ 4.48, and δ 4.76. The aromatic protons exhibited multiplets in the region δ 6.54–7.86. The off-resonance decoupled ^{13}C NMR spectra of **4a** exhibited peaks for the –NCH_3 carbon and the spiro carbon at δ 35.72 and δ 77.96 ppm. The indanedione ring carbonyls resonated at δ 202.23 and δ 202.56 ppm. The carbonyl group attached to the ferrocene and the pyrrolidine moiety resonated at δ 202.17 ppm. These observed chemical shift values confirmed the proposed

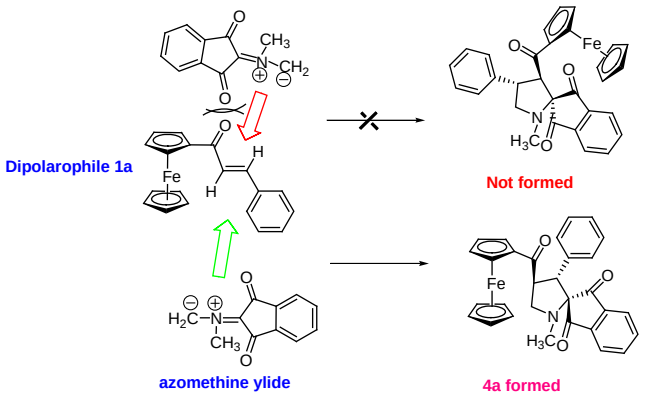


Figure 1. Mode of approach of the azomethine ylide to the ferrocene-derived dipolarophile **1a**.

structure. The formation of the product was confirmed by mass spectral and elemental analysis. The mass spectrum of **4a** showed a peak at *m/z* 502 (M^+) and gave satisfactory elemental analysis. The reactions were found to be highly regioselective leading to the formation of only one product **4a** and the formation of the other possible regioisomer was not observed (Fig. 1). This may be due to the unfavorable dipole–dipole repulsion between the carbonyl groups of indanedione and the dipolarophile. Similarly the formations of the **4b–e** were confirmed by spectral and elemental analysis. The dipolarophile with an electron-withdrawing group in the phenyl ring was found to be more reactive to the azomethine ylide generated from ninhydrin and sarcosine than the dipolarophile with an electron-donating group in the phenyl ring and the formations of the cycloadducts were observed in all the cases with moderate to good yield irrespective of the nature of the substituent present in the phenyl ring (Table 1).

In the next step a facile synthesis of *N*-methyl C-2-spiropyrrolidine heterocycle attached to the ferrocene moiety at C-3 and diverse heterocyclic entity such as furan, thiophene, and pyridyl unit at C-4 positions (**6a–b** and **8)** was accomplished easily by the reaction of ferrocene-derived dipolarophiles, 1-ferrocenyl-3-furyl-prop-2-ene-1one **5a**/1-ferrocenyl-3-thienyl-prop-2-ene-1-one **5b,** and 1-ferrocenyl-3-pyridyl-prop-2-ene-1-one **7** with ninhydrin **2** and sarcosine **3** in refluxing acetonitrile, in good yield. The formation of the cycloadducts was evidenced by spectral and elemental analysis (Schemes 3 and 4). The regio- and stereochemical outcome of the cycloaddition reaction was unambiguously ascertained by single-crystal X-ray analysis of the cycloadduct **6a** (Fig. 2).[33] The reactivity of the ferrocene bearing dipolarophile was observed to be in the order **5a** > **7** > **5b** as evident from the reaction time depicted in Table 1. Refluxing 1-phenyl-3-ferrocenyl-prop-2-ene-1-one derivatives **9a–b** with ninhydrin **2** and sarcosine **3** in acetonitrile afforded the ferrocenyl spiro-indanedione-pyrrolidines **10a–b** in good yield as evidenced by spectral and elemental analysis (Scheme 5).

It is interesting to note that the cycloadduct **12** has two ferrocene moieties on the pyrrolidine platform and was synthesized easily by trapping the azomethine ylide generated from ninhydrin **2** and sarcosine **3** with the unusual dipolarophile **11** (Scheme 6). Thus, the IR spectrum of the ferrocene grafted *N*-methyl spiropyrrolidine **12** showed peaks at 1670 and 1740 cm^{-1} due to the ferrocenoyl and indanedione carbonyl groups. The ^1H NMR spectrum of **12** exhibited a singlet at δ 2.36 due to the $-NCH_3$ protons of the pyrrolidine moiety. The pyrrolidine ring proton attached directly to the ferrocene moiety appeared as doublet at δ 4.08 (J = 10.4 Hz). The $-NCH_2$ protons of the pyrrolidine ring appeared as multiplets in the region δ *3.34–3.36* and δ *3.80–3.82* whereas the pyrrolidine ring proton attached to the ferrocenoyl moiety appeared as multiplet in the region δ *4.37–4.45.* The protons of the ferrocene moiety appeared as singlets at δ 3.72, δ 3.86, δ 3.98, δ 4.22, δ 4.26, δ 4.33, δ *4.48,* and δ 4.70. The aromatic protons exhibited multiplets in the region δ 7.52–8.16. The off-resonance proton decoupled ^{13}C spectrum of **12** exhibited peaks at δ 35.86 and δ 47.62 due to the pyrrolidine $-NCH_3$ and NCH_2 carbons. The spiro carbon resonated at δ 77.91. The indanedione and the ferrocenyl carbonyl carbons resonated at δ 201.14, δ *201.46,* and δ *203.38,* respectively. The structure of the product **12** was further confirmed through mass spectroscopy, which showed a molecular ion peak at 606 (M^+). The cycloadduct **12** gave satisfactory elemental analysis. The chemical yields of the cycloadducts are depicted in Table 1. From Table 1, it is evident that the rate of the reactions and the yields of the products are good in acetonitrile (60–88%).

In conclusion, we have synthesized successfully a series of hitherto unknown novel ferrocenyl-spiro-indanedione-*N*-methyl-pyrrolidines by employing various unusual ferrocene derivatives as efficient 2π-component in 1,3-dipolar cycloaddition reactions of azomethine ylides. The results demonstrate that ferrocene-derived dipolarophiles can further be exploited for the synthesis of a variety of complex heterocycles through cycloaddition reactions. These kinds of ferrocene grafted spiro heterocycles could serve as novel and potential candidates for biological screening. Solid-phase as well as microwave induced synthesis of such ferrocene bearing

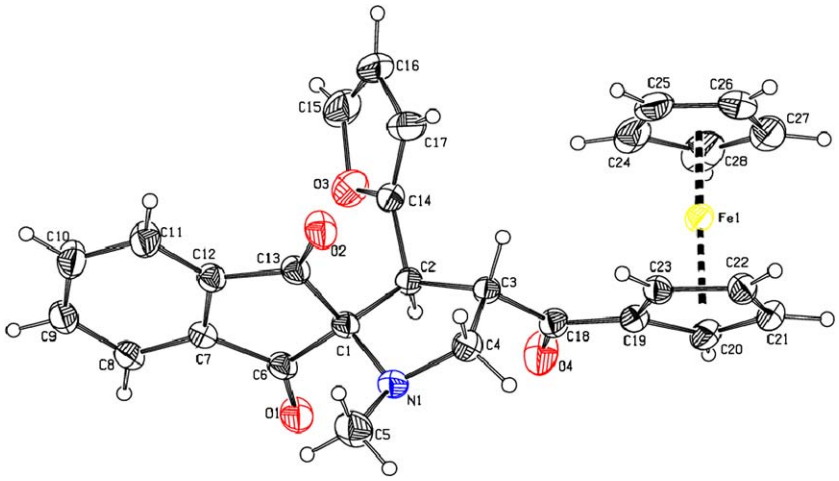


Figure 2. ORTEP diagram of **6a**.

spiropyrrolidines through 1,3-dipolar cycloaddition reaction is under investigation. It is also envisaged that complex ferrocene grafted dispiropyrrolidines could also be synthesized through 1,3-dipolar cycloaddition of azomethine ylides by suitably modifying the dipolarophile. Our further efforts in these directions are in progress.

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31. *Representative procedure for the synthesis of spiropyrrolidines derivatives* ***4a***: *Method A*: A solution of the ferrocene-derived dipolarophile **1a** (1 mmol), ninhydrin **2** (1 mmol), and sarcosine **3** (1 mmol) was refluxed in 10 mL acetonitrile until the completion of the reaction as evidenced by TLC. The solvent was removed under reduced pressure and the crude product was subjected to column chromatography using petroleum ether: ethyl acetate (4:1) as eluent. The product **4a** was then recrystallized from methanol.*Method B*: A solution of the ferrocene-derived dipolarophile **1a** (1 mmol), ninhydrin **2** (1 mmol), and sarcosine **3** (1 mmol) was refluxed in 10 mL toluene using a Dean-stark apparatus. After the completion of the reaction as evidenced by TLC, the solvent was removed under reduced pressure and the crude product was subjected to column chromatography using petroleum ether:ethyl acetate (4:1) as eluent. The product **4a** was then recrystallized from methanol.
32. *Physical properties and spectroscopic data 1-N-methyl-spiro-[2.2′]-indane-1′,3′-dione-3-phenyl-4-ferrocenoyl-pyrrolidine* ***4a***: IR (KBr): 1665, 1740 cm^{-1}; ^1H NMR ($CDCl_3$/300 MHz): δ 2.34 (s, 3H), 3.44 (t, 1H, J = 7.0 Hz), 3.74 (t, 1H, J = 8.4 Hz), 3.96 (s, 5H), 4.44–4.46 (m, 1H), 4.52 (d, 1H, J = 10.3 Hz), 4.48 (s, 2H), 4.76 (s, 2H), 6.54–7.86 (m, 9H); ^{13}C NMR ($CDCl_3$/100 MHz): δ 35.72, 46.49, 62.34, 66.50, 69.22, 72.47, 72.68, 77.96, 122.79, 122.97, 127.31, 128.84, 135.45, 136.74, 140.79, 141.82, 142.38, 200.23, 200.56, 202.17 ppm; EIMS *m/z*: 502 (M^+); CHN Anal. Calcd for $C_{30}H_{25}NO_3Fe$: C, 71.70; H, 5.01; N, 2.78. Found: C, 71.53; H, 5.16; N, 2.94.*1-N-Methyl-spiro-[2.2′]-indane-1′,3′-dione-3-furyl-4-ferrocenoyl-pyrrolidine* ***6a***: IR (KBr): 1670, 1740 cm^{-1}; ^1H NMR ($CDCl_3$/300 MHz): δ 2.34 (s, 3H), 3.46 (t, 1H, J = 7.5 Hz), 3.76 (t, 1H, J = 9.4 Hz)), 4.02 (s, 5H), 4.45–4.47 (m, 1H), 4.48 (s, 2H), 4.63 (d, J = 10.5 Hz), 4.78 (s, 1H), 4.88 (s, 1H), 5.99 (s, 2H), 7.04 (s, 1H), 7.79–7.95 (m, 4H); ^{13}C NMR (75 MHz): δ 36.17, 48.92, 49.68, 57.78, 69.32, 69.68, 69.74, 72.69, 78.49, 108.77, 110.34, 122.39, 123.01, 135.82, 136.10, 140.86, 141.65, 142.11, 149.27, 200.61, 201.10, 202.69 ppm; EIMS *m/z*: 493 (M^+); CHN Anal. Calcd for $C_{28}H_{23}NO_4Fe$: C, 68.16; H, 4.69; N, 2.83. Found: C, 68.35; H, 4.84; N, 2.67.*1-N-Methyl-spiro-[2.2′]-indane-1′,3′-dione-3-ferrocenyl-4-(p-methoxybenzoyl-pyrrolidine* ***10b***: IR (KBr): 1668, 1736 cm^{-1}; ^1H NMR ($CDCl_3$/300 MHz): δ 2.33 (s, 3H), 3.32–3.35 (m, 1H), 3.68 (s, 3H), 3.77 (s, 5H), 3.85–3.87 (m, 1H), 3.90 (s, 1H), 4.04 (d, 1H, J = 10.3 Hz), 4.19 (s, 1H),4.31 (s, 1H), 4.34–4.40 (m, 1H), 4.34 (s, 1H), 6.57–8.16 (m, 8H).; ^{13}C NMR ($CDCl_3$/75 MHz): δ 36.30, 48.11, 55.46, 63.40, 66.31, 68.57, 69.22, 72.54, 72.61, 76.58, 77.89, 113.20, 122.59, 122.75, 129.32, 129.65, 130.93, 136.50, 140.58, 141.99, 200.99, 201.28, 203.63 ppm; EIMS *m/z*: 533 (M^+); CHN Anal. Calcd for $C_{31}H_{27}NO_4Fe$: C, 69.80; H, 5.10; N, 2.62. Found: C, 69.93; H, 5.29; N, 2.78.*1-N-Methyl-spiro-[2.2′]-indane-1′,3′-dione-3-ferrocenyl-4-feroocenoyl-pyrrolidine* ***12***: IR (KBr): 1670, 1740 cm^{-1}; ^1H NMR (DMSO-d_6/300 MHz): δ 2.36 (s, 3H), 3.34–3.36 (m, 1H), 3.72 (s, 5H), 3.80–3.82 (m, 1H), 3.86 (s, 1H), 3.98 (s, 5H), 4.08 (d, 1H, J = 10.4 Hz), 4.22 (s, 1H), 4.26 (s, 1H), 4.33 (s, 1H), 4.37–4.45 (m, 1H), 4.48 (s, 2H), 4.70 (s, 2H), 7.52–8.16 (m, 4H).; ^{13}C NMR (DMSO-d_6/75 MHz): δ 35.86, 47.62, 62.33, 65.27, 69.02, 69.34, 72.36, 72.53, 72.64, 72.71, 76.49, 77.91, 129.60, 134.21, 136.33, 140.67, 142.02, 201.14, 201.46, 203.38 ppm; EIMS *m/z*: 606 (M^+); CHN Anal. Calcd for $C_{34}H_{29}NO_3Fe_2$: C, 67.35; H, 3.66; N, 2.31. Found: C, 67.19; H, 3.80; N, 2.47.
33. The crystal structure has been deposited at the Cambridge Crystallographic Data center CCDC number: 666153, molecular formula: $C_{28}H_{23}Fe_1N_1O_4$, unit cell parameters: *a* 10.2621 (6), *b* 10.9085, *c* 20.9435 (12), β 100.2100 (10), space group *P*21/*n*. Data acquisition: The Cambridge Crystallographic Data Center;deposit@ccdc.cam.ac.uk, http://www.ccdc.cam.ac.uk/deposit.