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A simple synthesis of ferrocenyl bis-amides by a Ugi four-component reaction

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ABSTRACT

An efficient and simple synthesis of ferrocenyl bis-amides by the Ugi four-component reaction of ferrocenecarboxaldehyde, carboxylic acids, isocyanides and amines in methanol at room temperature is reported.

1. Introduction

Multi-component reactions have recently become one of the favored methods to prepare pharmacologically relevant compounds [1,2]. The Ugi four-component reaction (U-4CR) is the known isocyanide-based multi-component reactions useful for generation of molecular diversity [3]. In U-4CR (Fig. 1) an α-amido carboxamide is formed from the reaction of an isocyanide, a carboxylic acid, and an imine, which is normally formed in situ from an aldehyde or ketone and an amine. Due to the great diversity of products which can be obtained by this reaction, the U-4CR is an important tool in combinatorial chemistry [4].

Because ferrocene derivatives are characterized by their ability to make metal-centred redox systems to generate oxidized or reduced form of different properties they have been widely employed in various fields such as: molecular recognition as biosensors [5–9], in asymmetric catalysis [10], in polymer science as redox active polymers and dendrimers [11], in nonlinear optics [12], in synthesis of complex photochemical systems [13] and in pharmacology [14]. Successful attempts of the synthesis of amino acids bearing ferrocene moiety have been also performed [15–20]. Ferrocenyl amino acids found their application in food chemistry as a possible substitute for phenylalanine in the commercial sweetener aspartame [20].

The reactions using ferrocenecarboxaldehyde as starting material have recently attracted the interest of the synthetic community because the formation of different ferrocene derivatives can be expected depending on the specific conditions and structure of the building blocks [21–29].

Considering the above reports, the development of new and simple synthetic methods for the efficient preparation of the new ferrocene derivatives will be a beneficial and interesting challenge. In this paper, we report an efficient synthesis of ferrocenyl bis-amides by a Ugi four-component reaction using ferrocenecarboxaldehyde.

2. Results and discussion

We found that a mixture of ferrocenecarboxaldehyde **1**, isocyanides **2a–d,** carboxylic acids **3a–c** and amines **4a–d** in the absence of any catalyst at room temperature in methanol for 24 h afforded ferrocenyl bis-amides **5a–j** in good yields (Scheme 1). The results are summarized in Table 1.

To the best of our knowledge, this new Ugi four-component strategy provides the first example of an efficient synthesis of ferrocenyl bis-amide derivatives. This method, based on catalyst-free reaction in methanol, is the most simple and convenient and would be applicable for the synthesis of different types of ferrocenyl bis-amides.

Compounds **5** are stable solids whose structures were established by IR, ^{1}H and ^{13}C-NMR spectroscopy and elemental analysis. The elucidation of the structure of **5** using IR, ^{1}H and ^{13}C-NMR spectroscopic data is discussed with **5a** as an example. The IR

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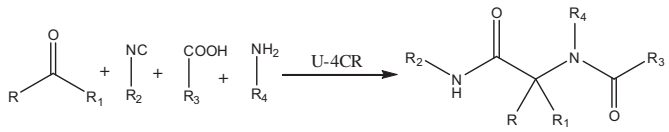


Fig. 1. General description of U-4CR.

spectrum of **5a** exhibited absorption bands due to carbonyl groups of amides at 1672 and 1624 cm^{-1} and the NH absorption of amide group was observed at 3323 cm^{-1}. The ^{1}H-NMR spectrum of **5a** consisted of multiplet signals for the cyclohexyl rings (δ_H 1.17–1.88 ppm) and the NH–CH resonance (δ_H 3.69) and a sharp singlet for the methine group (δ_H 6.04 ppm). A broad resonance (δ_H 8.23 ppm) was observed for the NH group and a multiplet signal for the ferrocenyl hydrogens (δ_H 3.93–4.17 ppm). The aromatic hydrogens exhibited two broad resonances in the aromatic region of the spectrum. In the ^{13}C-NMR spectrum, the signals at $\delta_=$ 67.7, 68.1, 69.2, 69.9 and 82.2 ppm was attributed to the carbon atoms of the ferrocenyl system. The carbonyl groups of amides were visible at $\delta = 167.9$ and 169.7 ppm and the methine carbon was observed at $\delta = 60.2$ ppm. The signal at $\delta = 48.5$ ppm was attributed to the methine carbon atom of the cyclohexyl group.

Compound **5** apparently results from the formation of ferrocenyl imine **6** (formed in situ by reaction of amine **4** and the ferrocenecarboxaldehyde **1**). Subsequent reaction of the imine **6** with the isocyanides **2** and the carboxylic acids **3** gives intermediate **7**, which rearranges via an acyl transfer into the bis-amides **5** (Scheme 2).

Ferrocene derivatives containing heterocyclic systems have attracted special attention in recent years [30–32]. Due to the importance of ferrocenyl heterocyclic compounds, we used 2-amino pyridine **8a** and 2-amino-pyrimidine **8b** as heterocyclic amines in the reaction. This made it possible to synthesize new ferrocenyl bis-amides containing pyridine and pyrimidine moiety **9a,b** (Scheme 3).

When the indole-3-carboxylic acid **10** was selected as a heterocyclic carboxylic acid (Scheme 4), the ferrocenyl bis-amide containing indole moiety **11** was obtained in 68% yield.

3. Conclusion

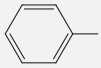
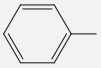
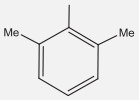
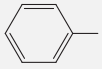
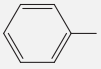
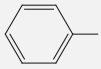
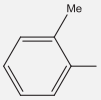
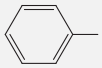
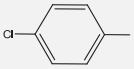
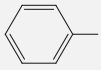
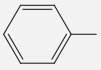
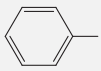
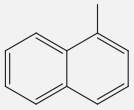
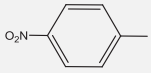
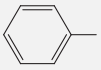
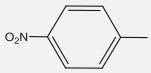
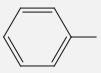
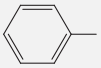
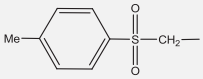
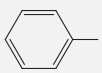
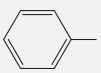
In conclusion, we have described an efficient Ugi four-component reaction for the synthesis of ferrocenyl bis-amide derivatives under mild and neutral reaction conditions. This method has the advantages of inexpensive reagents, simple operation and simple experimental work up procedures.

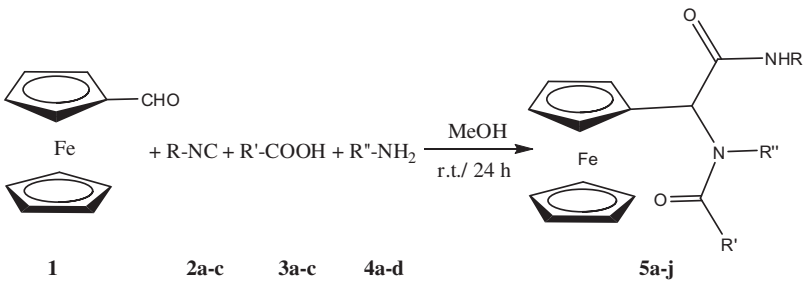
4. Experimental

4.1. General

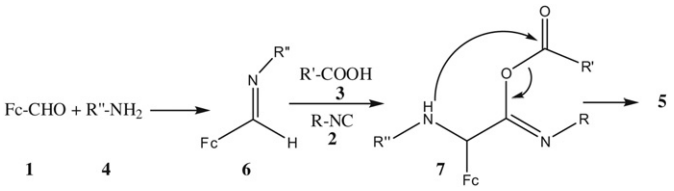
Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded on

Table 1
Synthesis of ferrocenyl bis-amides **5**.

Products **5**	R	R′	R″	Yield (%)
a				70
b				60
c				75
d				62
e				66
f				74
g				73
h				72
i		CH_3		51
j				45



Scheme 1. Synthesis of ferrocenyl bis-amides **5**.



Scheme 2. Mechanism of the reaction.

a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C-NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz, respectively. Elemental analyses were performed using a Heracus CHN-O-Rapid analyzer. Chemicals were purchased from Fluka or Merck and used as received.

4.2. General procedure for the preparation of ferrocenyl bis-amides 5, 8 and 10

A mixture of ferrocenecarboxaldehyde (1 mmol), carboxylic acid (1 mmol), isocyanide (1 mmol) and amine (1 mmol) in methanol (3 mL) was stirred for 24 h (The progress of reaction was monitored by TLC.). After completion of reaction, the reaction mixture was filtered and the precipitate washed with ether (5 mL) to afford the pure product.

4.3. Spectral data for selected compounds

4.3.1. Compound **5a**

Yellow powder (70%); mp 189–191 °C; IR (KBr) ν_{max} 3323 (NH), 2931, 1672 (CO), 1624 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.17–1.88 (m, 5 CH_2 of cyclohexyl, 10H), 3.69 (bs, CH–N of cyclohexyl, 1H), 3.93–4.17 (m, CH_{fer}, 9H), 6.04 (s, CH, 1H), 6.92 (bs, H–Ar, 5H), 7.10 (bs, H–Ar, 5H), 8.23 (bs, NH, 1H); ^{13}C-NMR (DMSO-d_6, 75.47 MHz): δ 25.2, 25.7, 32.8, 32.9, 48.5, 60.2, 67.7, 68.1, 69.2, 69.9, 82.2, 127.0, 127.8, 127.9, 128.2, 129.3, 131.2, 137.2, 140.5, 167.9, 169.7. Anal. Calcd for $C_{31}H_{32}FeN_2O_2$: C, 71.54; H, 6.20; N, 5.38%. Found: C, 71.41; H, 6.11; N, 5.29%.

4.3.2. Compound **5b**

White powder (60%); mp 120–122 °C; IR (KBr) ν_{max} 3300 (NH), 2918, 1689 (CO), 1622 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 2.38 (bs, $2CH_3$, 6H), 4.17 (bs, CH_{fer}, 9H), 6.62 (s, CH, 1H), 7.12 (bs, H–Ar, 13H), 8.56 (bs, NH, 1H). Anal. Calcd for $C_{33}H_{30}FeN_2O_2$: C, 73.07; H, 5.57; N, 5.16%. Found: C, 72.94; H, 5.50; N, 5.27%.

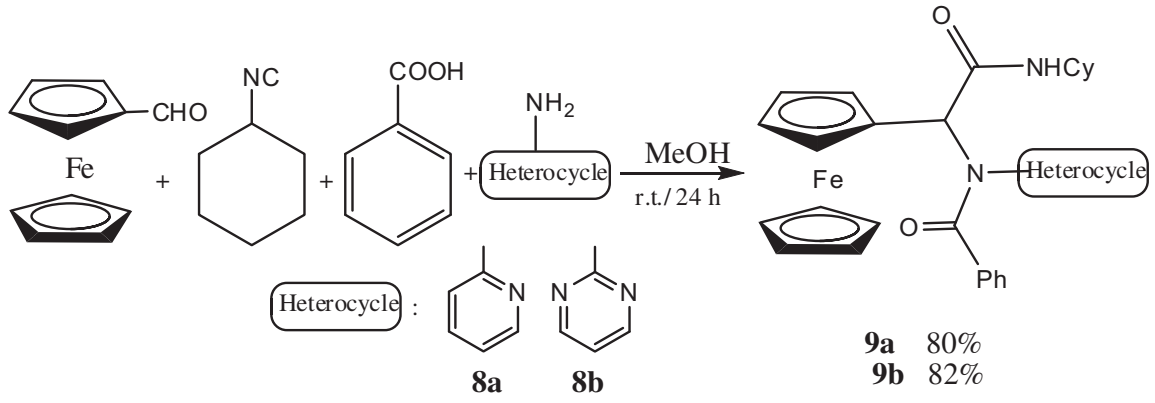
Due to very low solubility of the products **5b** and **5c**, we cannot report the ^{13}C-NMR data for these products.

4.3.3. Compound **5c**

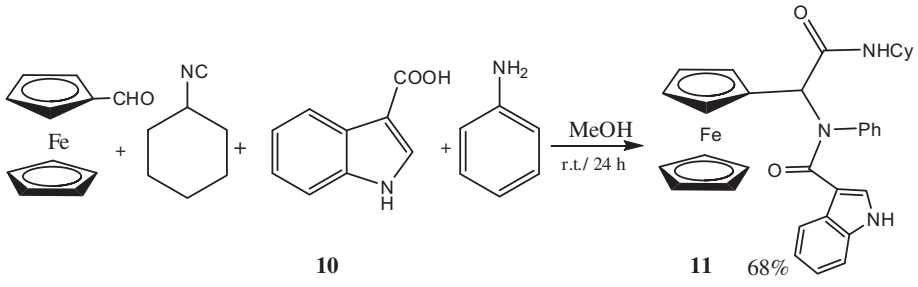
Cream powder (75%); mp 185–186 °C; IR (KBr) ν_{max} 3320 (NH), 2936, 1674 (CO), 1631 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.19–1.91 (m, 5 CH_2 of cyclohexyl, 10H), 1.67 (s, CH_3, 3H), 3.73 (bs, CH–N of cyclohexyl, 1H), 3.89–4.32 (m, CH_{fer}, 9H), 5.90 (s, CH, 1H), 6.68–6.99 (bs, H–Ar, 4H), 7.07–7.15 (m, H–Ar, 5H), 8.32 (bs, NH, 1H). Anal. Calcd for $C_{32}H_{34}FeN_2O_2$: C, 71.91; H, 6.41; N, 5.24%. Found: C, 71.83; H, 6.34; N, 5.18%.

4.3.4. Compound **5d**

Yellow powder (62%); mp 118–120 °C; IR (KBr) ν_{max} 3309 (NH), 2919, 1688 (CO), 1629 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.06–1.90 (m, 5 CH_2 of cyclohexyl, 10H), 3.70 (bs, CH–N of cyclohexyl, 1H), 4.05–4.20 (m, CH_{fer}, 9H), 6.04 (s, CH, 1H), 6.98 (bs, H–Ar, 4H), 7.11–717 (m, H–Ar, 5H), 8.33 (d, J = 8.4 Hz, NH, 1H); ^{13}C-NMR (DMSO-d_6, 75.47 MHz): δ 25.2, 25.7, 32.8, 32.9, 48.6, 60.1, 65.3, 67.8, 68.3, 69.3, 69.9, 82.0, 127.8, 128.1, 128.2, 129.6, 131.4, 133.0, 137.1, 139.6, 167.9, 169.5. Anal. Calcd for $C_{31}H_{31}ClFeN_2O_2$: C, 67.10; H, 5.63; N, 5.05%. Found: C, 67.19; H, 5.57; N, 5.13%.



Scheme 3. Synthesis of heterocyclic ferrocenyl bis-amides **9**.



Scheme 4. Synthesis of ferrocenyl bis-amide containing indole moiety **11**.

*4.3.5. Compound **5e***

Yellow powder (66%); mp 110–111 °C; IR (KBr) ν_{max} 3319 (NH), 2921, 1691 (CO), 1632 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.40 (s, $3CH_3$, 9H), 3.96–4.19 (m, CH_{fer}, 9H), 6.05 (s, CH, 1H), 6.93 (bs, H–Ar, 5H), 7.11–714 (m, H–Ar, 5H), 7.86 (bs, NH, 1H); ^{13}C-NMR (DMSO-d_6, 75.47 MHz): δ 28.9, 50.9, 60.7, 67.7, 68.0, 69.2, 69.8, 70.0, 82.5, 127.0, 127.9, 128.0, 128.3, 129.3, 131.1, 137.3, 140.6, 168.4, 169.7. Anal. Calcd for $C_{29}H_{30}FeN_2O_2$: C, 70.45; H, 6.12; N, 5.67%. Found: C, 70.33; H, 6.20; N, 5.74%.

Due to very low solubility of the products **5f** and **5g**, we cannot report the ^{13}C-NMR data for these products.

*4.3.6. Compound **5f***

Cream powder (74%); mp 115–117 °C; IR (KBr) ν_{max} 3323 (NH), 2919, 1699 (CO), 1636 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.08–1.93 (m, 5 CH_2 of cyclohexyl, 10H), 3.62 (bs, CH–N of cyclohexyl, 1H), 3.96–4.25 (m, CH_{fer}, 9H), 6.04 (s, CH, 1H), 6.73–7.89 (m, H–Ar, 12H), 8.16 (d, J = 8.6 Hz, NH, 1H). Anal. Calcd for $C_{35}H_{34}FeN_2O_2$: C, 73.69; H, 6.01; N, 4.91%. Found: C, 73.79; H, 5.93; N, 4.80%.

*4.3.7. Compound **5g***

Brown powder (73%); mp 146–148 °C; IR (KBr) ν_{max} 3334 (NH), 2931, 1699 (CO), 1638 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.30–1.90 (m, 5 CH_2 of cyclohexyl, 10H), 3.71 (bs, CH–N of cyclohexyl, 1H), 3.94–4.18 (m, CH_{fer}, 9H), 6.03 (s, CH, 1H), 6.95 (bs, H–Ar, 5H), 7.36 (bs, H–Ar, 2H), 7.96 (bs, H–Ar, 2H), 8.28 (bs, NH, 1H). Anal. Calcd for $C_{31}H_{31}FeN_3O_4$: C, 65.85; H, 5.53; N, 7.43%. Found: C, 65.77; H, 5.47; N, 7.52%.

*4.3.8. Compound **5h***

Orange powder (72%); mp 146–147 °C; IR (KBr) ν_{max} 3331 (NH), 2921, 1698 (CO), 1639 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.41 (s, $3CH_3$, 9H), 3.96–4.21 (m, CH_{fer}, 9H), 6.03 (s, CH, 1H), 6.94 (bs, H–Ar, 5H), 7.34 (d, J = 8.1 Hz, H–Ar, 2H), 7.99 (d, J = 8.1 Hz, H–Ar, 2H), 8.25 (bs, NH, 1H); ^{13}C-NMR (DMSO-d_6, 75.47 MHz): δ 28.9, 51.0, 60.8, 67.8, 68.1, 69.2, 69.7, 70.1, 82.1, 123.3, 127.6, 128.1, 129.3, 131.2, 139.6, 143.7, 147.4, 168.1, 168.3. Anal. Calcd for $C_{29}H_{29}FeN_3O_4$: C, 64.57; H, 5.42; N, 7.79%. Found: C, 64.47; H, 5.49; N, 7.68%.

*4.3.9. Compound **5i***

Cream powder (51%); mp 213–215 °C; IR (KBr) ν_{max} 3320 (NH), 2934, 1676 (CO), 1632 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.16–1.85 (m, 5 CH_2 of cyclohexyl, 10H), 1.59 (s, CH_3, 3H), 3.62 (bs, CH–N of cyclohexyl, 1H), 3.88–4.13 (m, CH_{fer}, 9H), 5.82 (s, CH, 1H), 7.02–7.14 (m, H–Ar, 5H), 8.17 (d, J = 6.9 Hz, NH, 1H). ^{13}C-NMR (DMSO-d_6, 75.47 MHz): δ 23.5, 25.2, 25.7, 32.7, 32.8, 48.4, 60.4, 67.7, 67.9, 69.0, 69.8, 82.5, 127.8, 128.4, 130.9, 139.8, 168.0, 169.2. Anal. Calcd for $C_{26}H_{30}FeN_2O_2$: C, 68.13; H, 6.60; N, 6.11%. Found: C, 68.23; H, 6.65; N, 6.02%.

*4.3.10. Compound **5j***

Yellow powder (45%); mp 205 °C (decomposed); IR (KBr) ν_{max} 3323 (NH), 2930, 1671 (CO), 1631 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): 2.26 (s, CH_3, 3H), 3.87–4.13 (m, CH_{fer}, 9H), 4.72 and 5.03 (ABSystem, J = 9.1 Hz, CH_2), 6.17 (s, CH, 1H), 6.65 (bs, H–Ar, 2H), 6.92 (bs, H–Ar, 3H), 7.12 (bs, H–Ar, 5H), 7.38 (bs, H–Ar, 2H), 7.84 (bs, H–Ar, 2H), 9.37 (bs, NH, 1H). Anal. Calcd for $C_{33}H_{30}FeN_2O_2S$: C, 65.35; H, 4.99; N, 4.62%. Found: C, 65.24; H, 4.91; N, 4.69%.

Due to very low solubility of the product **5j**, we cannot report the ^{13}C-NMR data for this product.

*4.3.11. Compound **9a***

Orange powder (80%); mp 195 °C (decomposed); IR (KBr) ν_{max} 3333 (NH), 2931, 1679 (CO), 1641 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.15–1.74 (m, 5 CH_2 of cyclohexyl, 10H), 3.07 (bs, CH–N of cyclohexyl, 1H), 4.01 (bs, CH_{fer}, 5H), 4.31–4.96 (m, H_{fer}, 4H), 4.96 (s, CH, 1H), 6.81 (bs, H–Ar, 1H), 7.09 (bs, H–Ar, 1H), 740–7.95 (m, H–Ar, 7H), 8.22 (bs, NH, 1H); ^{13}C-NMR (DMSO-d_6, 75.47 MHz): δ 25.2, 25.9, 34.1, 49.5, 56.8, 67.0, 68.4, 69.3, 80.1, 116.4, 123.4, 124.6, 128.9, 129.7, 131.3, 133.2, 135.6, 140.9, 165.5, 167.8. Anal. Calcd for $C_{30}H_{31}FeN_3O_2$: C, 69.10; H, 5.99; N, 8.06%. Found: C, 68.99; H, 5.91; N, 8.13%.

*4.3.12. Compound **9b***

Light brown powder (82%); mp 230 °C (decomposed); IR (KBr) ν_{max} 3336 (NH), 2930, 1676 (CO), 1647 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.13–1.77 (m, 5 CH_2 of cyclohexyl, 10H), 3.08 (bs, CH–N of cyclohexyl, 1H), 4.04 (bs, CH_{fer}, 5H), 4.30–4.97 (m, H_{fer}, 4H), 4.99 (s, CH, 1H), 6.80 (bs, H–Ar, 1H), 7.07 (bs, H–Ar, 1H), 742–7.97 (m, H–Ar, 6H), 8.25 (bs, NH, 1H). Anal. Calcd for $C_{29}H_{30}FeN_4O_2$: C, 66.67; H, 5.79; N, 10.72%. Found: C, 66.58; H, 5.71; N, 10.81%.

Due to very low solubility of the product **9b,** we cannot report the ^{13}C-NMR data for this product.

*4.3.13. Compound **11***

Cream powder (68%); mp 165–166 °C; IR (KBr) ν_{max} 3342 (NH), 3241 (NH), 2929, 1654 (CO), 1639 (CO) cm^{-1}; ^{1}H-NMR (DMSO-d_6, 300 MHz): δ 1.08–1.84 (m, 5 CH_2 of cyclohexyl, 10H), 3.63 (bs, CH–N of cyclohexyl, 1H), 3.89–4.13 (m, CH_{fer}, 9H), 5.89 (s, CH, 1H), 6.88–7.29 (m, H–Ar and CH, 10H), 8.09 (bs, NH, 1H), 10.77 (bs, NH, 1H); ^{13}C-NMR (DMSO-d_6, 75.47 MHz): δ 25.2, 25.7, 31.9, 32.7, 48.3, 59.3, 67.8, 67.9, 69.1, 69.9, 82.3, 108.8, 111.6, 118.5, 119.0, 121.2, 123.8, 127.5, 127.9, 128.3, 131.0, 136.4, 140.2, 168.1, 170.3. Anal. Calcd for $C_{32}H_{32}FeN_4O_2$: C, 68.58; H, 5.75; N, 10.00%. Found: C, 68.45; H, 5.65; N, 9.89%.

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