Electronic Supplementary Information (ESI)

Molecular design driving tetraporphyrin self-assembly on graphite: a joint STM, electrochemical and computational study

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1. Synthesis of P1 and P2

1.1 General methods.

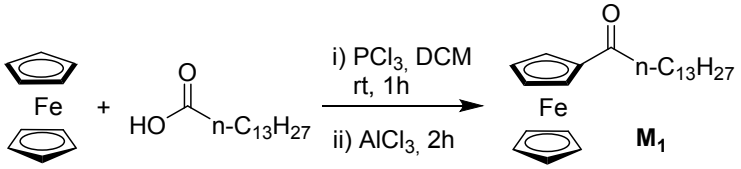
^{1}H NMR spectra were recorded on Varian Mercury 400 and Inova 600 spectrometers. Chemical shifts are reported in ppm from TMS with the solvent resonance as the internal standard (deuterochloroform: δ = 7.27 ppm, d_6-DMSO: δ = 2.50 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = duplet, t = triplet, q = quartet, dd = double duplet, dt = double triplet, pd = pseudo duplet, pt = pseudo triplet, m = multiplet), coupling constants (Hz). ^{13}C NMR spectra were recorded on Varian MR400 spectrometers and Inova 600 spectrometers. Chemical shifts are reported in ppm from TMS with the solvent as the internal standard (deuterochloroform: δ= 77.0 ppm, d_6-DMSO: δ = 39.51 ppm). If rotamers are present, the splitted signals are labeled as A (major rotamer) and B (minor rotamer). GC-MS spectra were taken by EI ionization at 70 eV on a Hewlett-Packard 5971 with GC injection. LC-electrospray ionization mass spectra (ESI-MS) were obtained with Agilent Technologies MSD1100 single-quadrupole mass spectrometer. They are reported as: *m/z* (rel. intense). Electrospray Ion Trap Mass Spectrometry were obtained with Agilent Technologies LC/MSD 1100 ion trap mass spectrometer injecting a solution in chloroform/methanol of the pure compound. Chromatographic purification was done with 240-400 mesh silica gel. Purification on preparative thin layer chromatography was done on Merck TLC silica gel 60 F_{254}.

1.2 Materials

If not otherwise stated, all reactions were carried under anhydrous conditions using standard Schlenk apparatus. Anhydrous solvents were supplied by Aldrich in Sureseal® bottles and were used as received avoiding further purification.

Reagents were purchased from Aldrich and used without further purification unless otherwise stated.

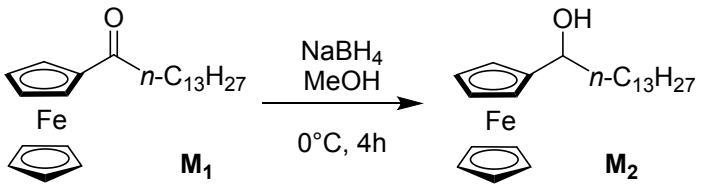
*1.3 Synthesis of **P1***



1-ferrocenyl-tetradecan-1-one (M1):[1] In a 250 mL balloon under nitrogen atmosphere, myristic acid (548 mg, 2.4 mmol and ferrocene (372 mg, 2.0 mmol) were dissolved in

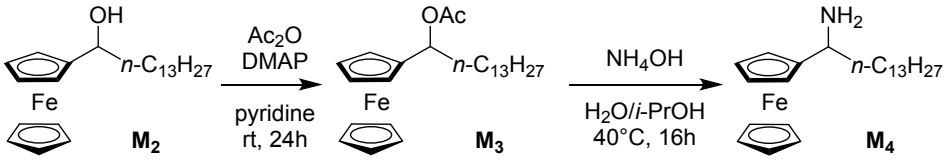
dichloromethane (40 mL). Phosphorous trichloride (200 µL, 2.28 mmol) was added at room temperature. After one hour of stirring aluminium trichloride (399 mg, 3.0 mmol) was added to the reaction mixture that was stirred for 2 hours. Then it was cooled to 0°C and 60 mL of aqueous NaOH 1M were slowly added until pH = 14. The solution turned from violet to orange. After 10 min. of stirring the organic phase was separated and the aqueous one was extracted with diethyl ether (2 x 20 mL). The collected organic phases were filtrated on a Celite® pad, dried over Na_2SO_4 and evaporated under reduced pressure to afford pure 1-ferrocenyl-tetradecan-1-one **M1** as an orange oil (79% yield, 628 mg, 1.58 mmol).

^{1}H NMR (400 MHz, $CDCl_3$, 25°C): δ = 0.89 (t, J = 7.7 Hz, 3H), 1.20-1.41 (m, 20H), 1.71 (p, J = 7.4 Hz, 2H), 2.70 (t, J = 7.6 Hz, 2H), 4.20 (s, 5H), 4.50 (t, J = 1.9 Hz, 2H), 4.79 (t, J = 1.9 Hz, 2H); ^{13}CNMR (100 MHz, $CDCl_3$, 25°C): δ = 14.1, 22.6, 24.6, 29.3, 29.4 (2C), 29.5, 29.6 (2C), 29.7 (2C), 31.9, 39.7, 69.3 (2C), 69.7 (5C), 72.0 (2C), 79.1, 204.6. Exact mass calculated for $C_{24}H_{36}FeO$: 396,2; ESI-MS: m/z = 396.2 $[M]^+$, 397.2 $[M+H]^+$.



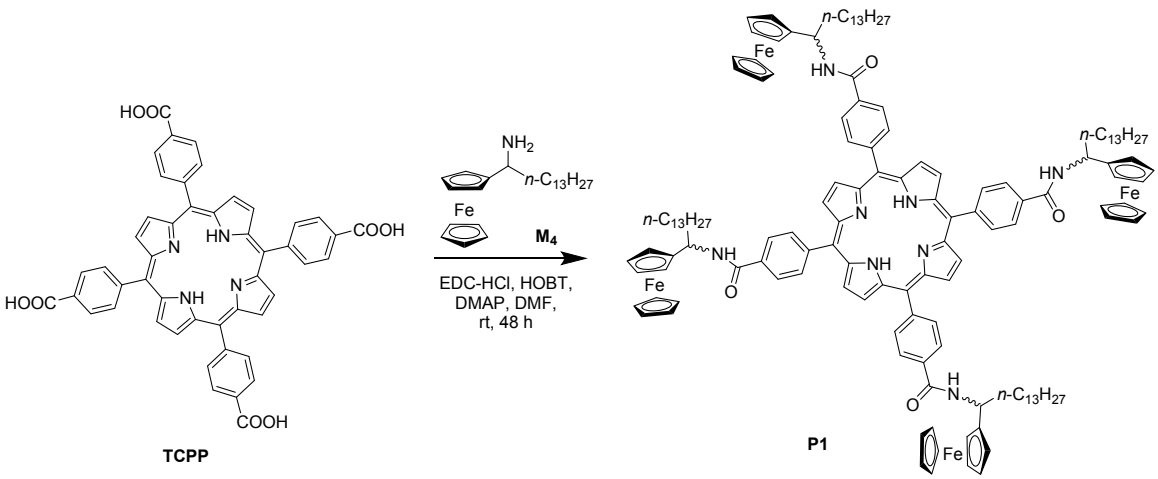
1-ferrocenyl-1-tetradecanol (M2): In a 50 mL flask, 1-ferrocenyl-tetradecan-1-one **M1** (198 mg, 0.5 mmol) was dissolved in methanol (10 mL) and cooled to 0°C. Then $NaBH_4$ (76 mg, 2 mmol) was added. After 4 hours complete conversion was achieved (monitored by TLC) and the reaction was quenched by addition of water and ice. Then methanol was evaporated under reduced pressure and the aqueous phase was extracted with diethyl ether (3 x 10 mL). The collected organic phases were dried over Na_2SO_4 and evaporated under reduced pressure to afford pure 1-ferrocenyl-1-tetradecanol **M2** as an orange oil (94% yield, 187 mg, 0.47 mmol).

^{1}H NMR (400 MHz, $CDCl_3$, 25°C): δ = 0.89 (t, J = 7.1 Hz, 3H), 1.16-1.34 (m, 21H), 1.34-1.47 (m, 1H), 1.54-1.74 (m, 2H), 1.95 (d, J = 2.5 Hz, 1H), 4.14-4.19 (m, 3H), 4.21 (s, 5H), 4.23-4.26 (m, 1H), 4.28-4.34 (m, 1H); ^{13}CNMR (100 MHz, $CDCl_3$, 25°C): δ = 14.1, 22.7, 26.0, 29.3, 29.5, 29.6 (5C), 29.70, 31.9, 38.2, 65.1, 67.3, 67.7, 69.9, 68.2 (5C), 69.6, 94.7. Exact mass calculated for $C_{24}H_{38}FeO$: 398,2; ESI-MS: m/z = 398.2 $[M]^+$, 399.2 $[M+H]^+$.



1-ferrocenyl-1-tetradecyl amine M4: In a 50 mL Schlenk tube under nitrogen atmosphere, 1-ferrocenyl-1-tetradecanol **M2** (374 mg, 0.94 mmol), acetic anhydride (1.8 mL, 18.8 mmol), 4-dimethylaminopyridine (11 mg, 0.94 mmol) and pyridine (3.0 mL) were added. The mixture was stirred for 24 hours at rt, until complete conversion to 1-ferrocenyl-1-tetradecyl acetate **M3** was observed by TLC. Pyridine and excess of acetic anhydride were evaporated under reduced pressure and the residue was dissolved in isopropanol (8 mL). Ammonium hydroxide (30% V/V in water, 2.0 mL, 25 mmol) was added and the mixture was heated at 40°C for 16 hours until complete conversion to **M4** was observed by TLC. The residue was evaporated under reduced pressure and directly purified by column chromatography (gradient elution from 95:5 to 0:100 cyclohexane:ethyl acetate) to afford pure 1-ferrocenyl-1-tetradecyl amine **M4** (67 % yield, 250 mg, 0.63 mmol) as an orange oil.

^{1}H NMR (400 MHz, $CDCl_3$, 25°C): δ = 0.89 (t, J = 6.9 Hz, 3H), 1.21-1.35 (m, 21H), 1.35-1.44 (m, 1H), 1.45-1.66 (m, 2H), 1.70-1.99 (m, 2H), 3.62 (t, J = 6.5 Hz, 1H), 4.10-4.14 (m, 3H), 4.17 (s, 5H), 4.19-4.22 (m, 1H); ^{13}CNMR (100 MHz, $CDCl_3$, 25°C): δ = 14.0, 22.5, 26.4, 29.2, 29.41, 29.4 (6C), 31.7, 39.2, 50.4, 64.8, 66.6, 66.9, 69.0, 68.0 (5C), 95.6. Exact mass calculated for $C_{24}H_{39}FeN$: 397,2; ESI-MS: m/z = 397.2 $[M]^+$, 398.2 $[M+H]^+$.



P1: In a 10 mL round bottomed flask under nitrogen atmosphere, TCPP (79, 0.1 mmol) was dissolved in DMF (3.5 mL). *N*-(3-Dimethylaminopropyl)-N′-ethylcarbodiimide (EDC) hydrochloride (114 mg, 0.6 mmol) and hydroxybenzonitrile (HOBT) (81mg, 0.6 mmol) were added. The solution was shielded by light and stirred for 1h. Then 1-ferrocenyl-1-tetradecyl

amine **M4** (175 mg, 0.44 mmol) and DMAP (54mg, 0.44 mmol) were added. After 48 hours the reaction was diluted with DCM (20 mL) and washed 5 times with water (10 mL). The organic phase was concentrated and purified by column chromatography (60:40 DCM:cyclohexane, then 90:10 DCM:MeOH) to afford pure **P1** (71 % yield, 166 mg, 0.71 mmol) as a dark purple solid (m.p. not observed up to 320°C).

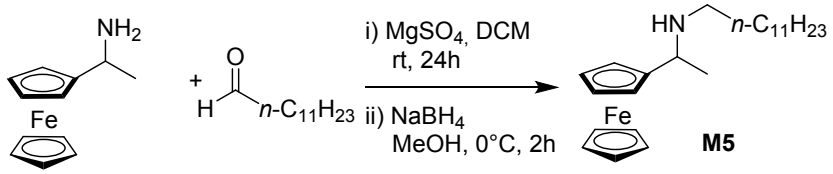
In $CDCl_3$ the ^1H NMR signals resulted broad and not resolved. Addition of d_6-DMSO allowed to record a higher quality spectra.

^1H NMR (600 MHz, $CDCl_3$:d_6-DMSO 4:1, 25°C, TMS as internal reference: 0.00 ppm): δ = -2.92 (s, 2H), 0.78 (t, J = 7.3 Hz, 12H), 1.13-1.34 (m, 72H), 1.34-1.46 (m, 8H), 1.46-1.58 (m, 8H), 1.80-1.90 (m, 4H), 1.91-1.99 (m, 4H), 4.08-4.18 (m, 8H), 4.18-4.31 (m, 24H), 4.41-4.52 (m, 4H), 5.06-5.20 (m, 4H), 7.86-7.94 (m, 4H), 8.21 (d, J = 7.3 Hz, 8H), 8.25 (d, J = 7.3 Hz, 8H), 8.79 (s, 8H).

Recording ^{13}C NMR in $CDCl_3$ or in other deuterated solvents or mixtures did not allow to resolve the signals corresponding to the ferrocene moiety that resulted a broad signal. Only recording the spectra in d_6-DMSO allowed to resolve the ferrocene signals, but unfortunately the solubility of **P1** in DMSO is very low (less than 1 mg for 500 μL) so that the porphyrin core signals could not be detected.

^{13}CNMR (100 MHz, $CDCl_3$, 25°C): δ = 14.1 (4C), 22.7 (4C), 26.5 (4C), 29.3 (4C), 29.6 (4C), 29.7 (20C), 29.8 (4C), 31.9 (4C), 36.2 (4C), 48.4 (4C), 68-71 (m, 40C), 119.3 (8C), 125,4 (8C), 130-132 (m, 8C), 134.4 (4C), 134.7 (12C), 145.5 (4C), 166.2 (4C). Exact mass calculated for $C_{144}H_{179}Fe_4N_8O_4^+$: 2308,1. Electrospray ion trap mass spectrometry: $[M+H]^+$: 2308.1

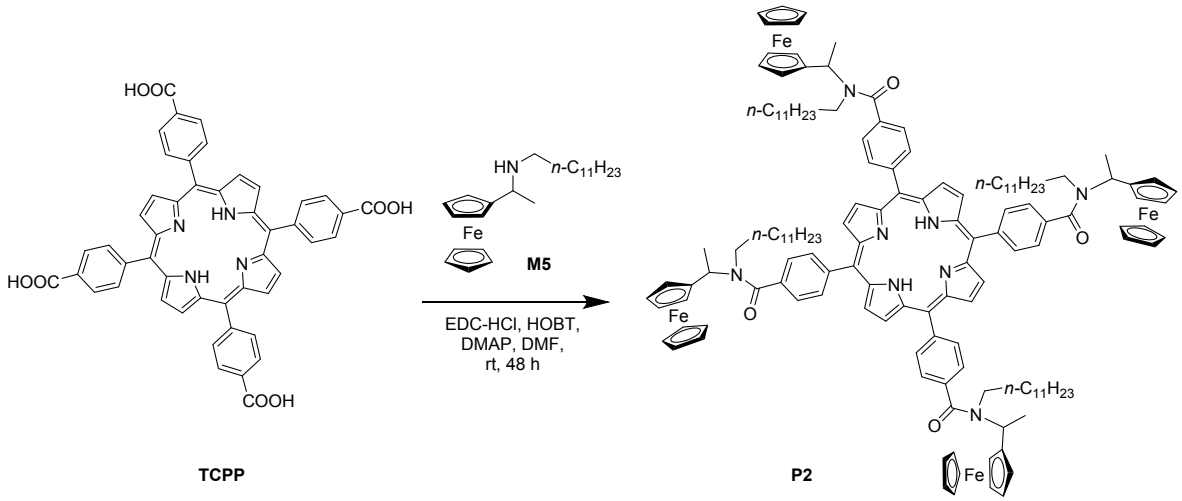
1.4 Synhtesis of ***P2***



***N*-dodecyl-1-ferrocenyl-1-ethyl amine M5**: In a 25 mL round bottomed flask under nitrogen atmosphere, 1-ferrocenyl-1-ethyl [2] (135 mg, 0.59 mmol) was dissolved in dichloromethane (4 mL). Dodecyl aldehyde (131 μL, 0.59 mmol) and $MgSO_4$ (600 mg were subsequently added and the mixture was stirred for 24 hours at room temperature. Then it was filtered over a small Celite® pad directly into a 25 mL balloon under nitrogen atmosphere. The pad was washed with methanol (5 mL) and the filtrate was cooled to 0°C. $NaBH_4$ (45 mg, 1.18 mmol)

was added and the mixture was stirred for 2h. The solvent was evaporated under reduced pressure and water (10 mL) and ethyl acetate (10 mL) were added, the organic phase was separated and the aqueous one was extracted with ethyl acetate (2x10 mL). The collected organic layers were dried over Na_2SO_4 and evaporated under reduced pressure. The crude was purified by column chromatography (70:29:1 cyclohexane:ethyl acetate:30% aq. NH_4OH) to afford pure 1-ferrocenyl-1-tetradecyl amine **M5** (54 % yield, 126 mg, 0.32 mmol) as an orange oil.

^{1}H NMR (400 MHz, $CDCl_3$, 25°C): δ = 0.78 (t, J = 6.3 Hz, 3H), 1.20-1.37 (m, 18H), 1.38 (d, J = 6.2 Hz, 3H), 1.41-1.55 (m, 2H), 1.55-1.72 (m, 1H), 2.52-2.68 (m, 2H), 3.52 (q, J = 6.2 Hz, 1H), 4.10-4.12 (m, 2H), 4.14 (s, 5H), 4.18 (s, 1H), (s, 1H); APT (attached proton test) (100 MHz, $CDCl_3$, 25°C): δ = 14.1, 21.6, 22.6, 27.4, 29.3, 29.5 (5C), 30.2, 31.9, 47.7, 52.4, 65.8, 67.0, 67.1, 67.2, 68.3 (5C), 93.7. Exact mass calculated for $C_{24}H_{39}FeN$: 397,2; ESI-MS: *m/z* = 397.2 $[M]^+$, 398.2 $[M+H]^+$.



TCPP P2

P2: In a 10 mL round bottomed flask under nitrogen atmosphere, TCPP (47, 0.06 mmol) was dissolved in DMF (2 mL). EDC hydrochloride (64 mg, 0.33 mmol) and HOBT (45 mg, 0.33 mmol) were added. The solution was shielded by light and stirred for 1h. Then *N*-dodecyl-1-ferrocenyl-1-ethyl amine **M5** (100 mg, 0.25 mmol) and DMAP (30 mg, 0.25 mmol) were added. After 48 hours the reaction was diluted with DCM (15 mL) and washed 5 times with water (8 mL). The organic phase was concentrated and purified by column chromatography (45:45:10 cyclohexane:EtOAc:DCM) to afford pure **P2** (63 % yield, 87 mg, 0.38 mmol) as a dark purple sticky solid.

^{1}H NMR (400 MHz, $CDCl_3$, 25°C, two rotamers A/B 1.8/1): δ = -2.84-(-2.75) (m, $2H_{A+B}$),

0.64-0.73 (m, 12H_B), 0.83-0.94 (m, 12H_A), 0.99-1.20 (m, 16H_{A+B}), 1.21-1.36 (m, 56H_{A+B}), 1.54-1.66 (m, 8H_{A+B}), 1.66-1.76 (m, 12H_{A+B}), 3.08-3.29 (m, 4H_A + 8H_B), 3.35-3.47 (m, 4H_A), 4.05-4.15 (m, 20H_A), 4.18-4.46 (m, 16H_A + 32H_B), 4.48-4.58 (m, 4H_B), 5.39-5.49 (m, 4H_A), 5.91-6.05 (m, 4H_B), 7.68-7.77 (m, 8H_B), 7.83-7.93 (d, J = 5.1 Hz, 8H_A), 8.17-8.25 (m, 8H_B), 8.28-8.38 (d, J = 5.1 Hz, 8H_A), 8.77-8.85 (m, 8H_B), 8.85-8.95 (m, 8H_A).

^{13}CNMR (100 MHz, $CDCl_3$, 25°C, two rotamers A/B 1.8/1): δ = 13.9 (4C_B), 14.1 (4C_A), 17.8 (4C_B), 18.5 (4C_A), 22.5 (4C_B), 22.7 (4C_A), 27.0 (4C_B), 27.5 (4C_A), 28.8 (4C_B), 29.0 (4C_A), 29.2 (4C_B), 29.3 (8C_A), 29.6 (8C_{A+B}), 29.7 (8C_{A+B}), 30.9 (4C_B), 31.7 (4C_B), 31.9 (4C_A), 42.1 (4C_A), 45.1 (4C_B), 49.0 (4C_B), 54.7 (4C_A), 66.6 (4C_B), 66.8 (4C_A), 67.6 (4C_{A+B}), 68.5 (4C_A), 68.7 (4C_{A+B}), 69.0 (20C_{A+B}), 69.3 (4C_B), 88.0 (4C_A), 88.5 (4C_B), 119.4 (8C_{A+B}), 124.8 (8C_B), 124.9 (8C_A), 131.7 (12C_{A+B}), 134.3 (8C_B), 134.5 (8C_A), 137.1 (4C_{A+B}), 142.4 (4C_B), 142.9 (4C_A), 171.1 (4C_A), 171.3 (4C_B). Exact mass calculated for $C_{144}H_{179}Fe_4N_8O_4^+$: 2308,1. Electrospray ion trap mass spectrometry: $[M+H]^+$: 2308.1.

2. Scanning Tunneling Microscopy

2.1 Experimental section

Scanning Tunneling Microscopy (STM) measurements were performed using a Veeco scanning Tunneling microscope (multimode Nanoscope III, Veeco) at the interface between a highly oriented pyrolitic graphite (HOPG) substrate and a supernatant solution, thereby mapping a maximum area of 1μm × 1μm. Solution of molecules were applied to the basal plane of the surface. For STM measurements, the substrates were glued to a magnetic disk and an electric contact was made with silver paint (Aldrich Chemicals). The STM tips were mechanically cut from a Pt/Ir wire (90/10, diameter 0.25 mm). The raw STM data were processed through the application of background flattening and the drift was corrected using the underlying graphite lattice as a reference. The lattice was visualizedby lowering the bias voltage to 20 mV and raising the current up to 65 pA. STM imaging was carried out in constant height mode without turning off the feedback loop, to avoid tip crashes. All of the molecular models were minimized with MMFF and processed with QuteMol visualization software (http://qutemol.sourceforge.net).

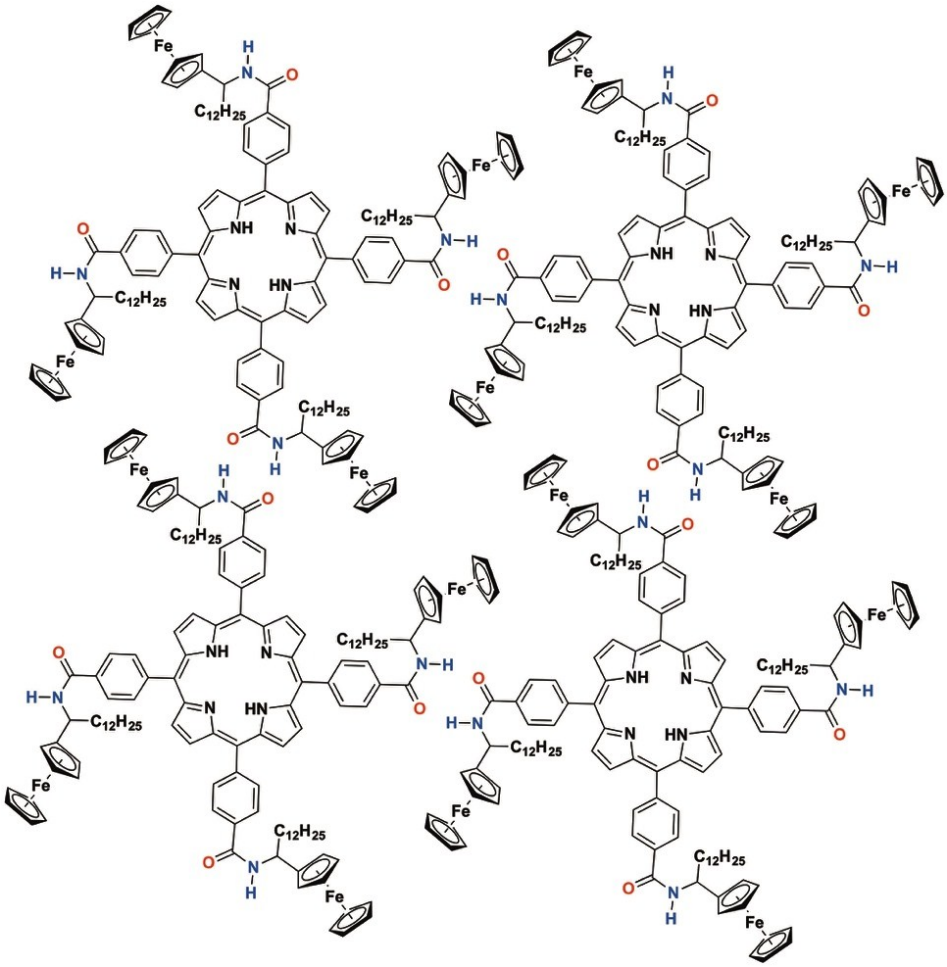


Figure S1. Model showing the formation of H-bonds between assembled **P1** compound. H-bonding donor and acceptor sites are indicated with red and blue respectively.

3. Cyclic voltammetry

3.1 Electrochemical study in solution

All chemicals used were reagent grade. Tetrabutylammoniumhexafluorophosphate (TBAH, Fluka) was used as supporting electrolyte as received. Dichlorometane (DCM, Sigma-Aldrich) was treated according to a procedure previuosly described [1]. The voltammetric experiments in ultra dry condition were reported elsewhere [2]. The working electrode was platinum disk ultramicroelectrodes (Ø =125 μm) sealed in glass. The counter electrode was a platinum spiral, and the quasi-reference electrode was a silver spiral. Potentials were measured with decamethylferrocene standard and were to Ag/AgCl(sat).

In Figure S3 are reported the cyclic voltammetry of **P1** and **P2** in DCM.

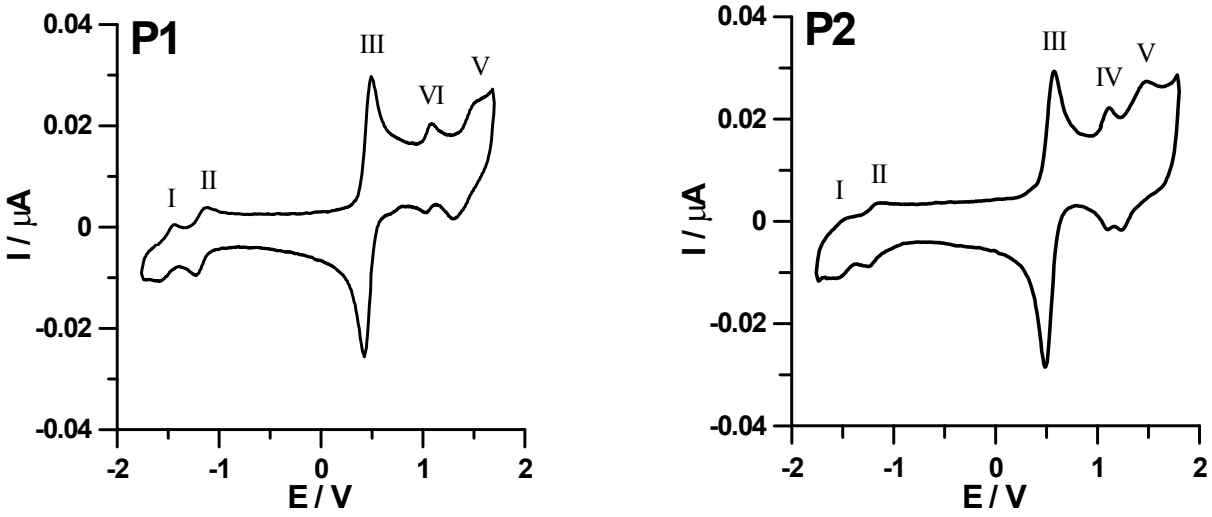


Figure S2. CV curves of **P1** and **P2** (0.2 mM), scan rate: 1 V s^{-1}, T = 25 °C; All CVs were performed under ultra-dry conditions.

Table S1. Redox potentials (V) of **P1** and **P2** in solution, referred to Ag/AgCl(sat).

	P1		**P2**	
	P-based	**Fc-based**	**P-based**	**Fc-based**
I	-1,51		-1.52	
II	-1,18		-1,22	
III		0,46		0.53
IV	1,06		1.09	
V	1,41		1.33	

In Table S1 are reported the five redox couple of porphyrins **P1** and **P2**, where I, II, IV and V are monoelectronic redox process associated to the porphyrin rings [3], while III is the redox process of the four ferrocenes.

4. DFT calculations

4.1 Mechanical stability: models to study the intra-cell interactions

In order to investigate the energetic contribution of each of the intra-cell interactions we carry out calculation using single and dimer models as displayed in Figs. S1, S2. The single molecules have been optimized within the calculated unit cell and then placed in a bigger box in order to calculate its total energy. In order to construct the dimer models, we have used the four-nearest neighbors given by the periodic boundary conditions of the single

molecule unit cell. This lay-out gives us the opportunity to asses at the same time the strength of the hydrogen bonding interactions and the repulsive electrostatic interaction due to the close distances of the inter-cell ferrocene molecules in the case of **P1** complex. The same procedure stated above has been used for the **P2** complex with the aim to investigate the two possible intra-cell interactions, namely, pure vdW interaction along the ***a*** axis and a combination of vdW interaction with a repulsion coming from the ferrocene -ferocene and *π–π stacking* of the nearest neighbors organic-cores.

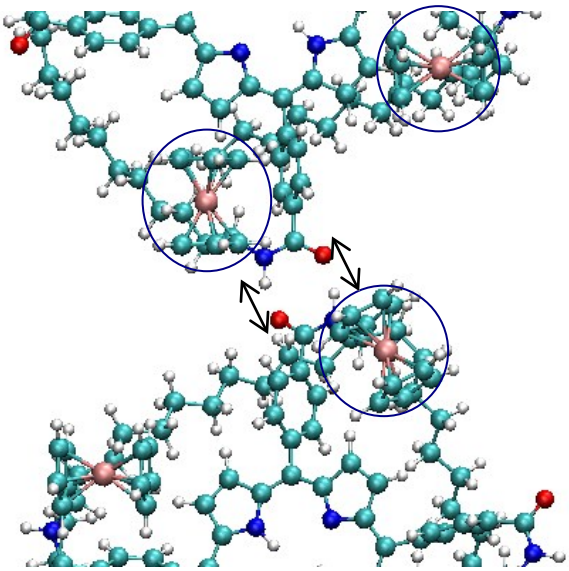


Figure S3. Theoretical model used to assess the intra- and inter cell interactions in **P1**. Intra and inter- cell electrostatic repulsion between the ferrocenes (encircled) takes place. The black arrows show the inter-cell hydrogen bonding.

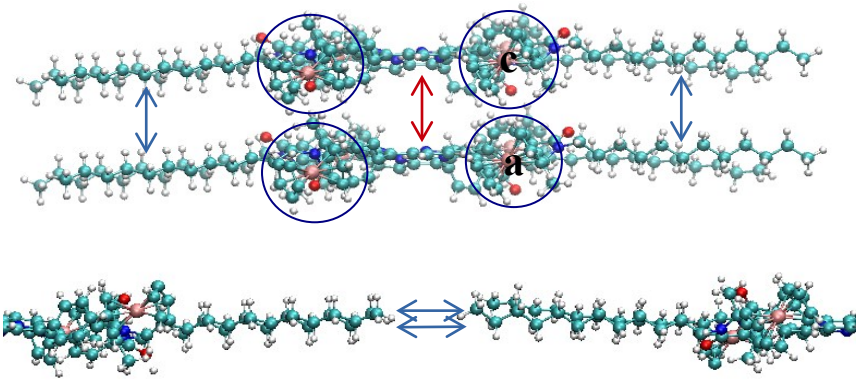


Figure S4. Theoretical model used to asses intra- and inter cell interactions in the **P2** molecular complex. Upper panel: Electrostatic repulsion between the upper 4 ferrocenes (encircled). The red arrow indicates also the repulsive nature of the organic core due to π–π stacking. Blue arrows indicate the vdW interaction experienced by the alkyl-chains along the

same axis. Lower panel: vdW interactions along the **b** lattice vector between the inter-cell alkyl chains.

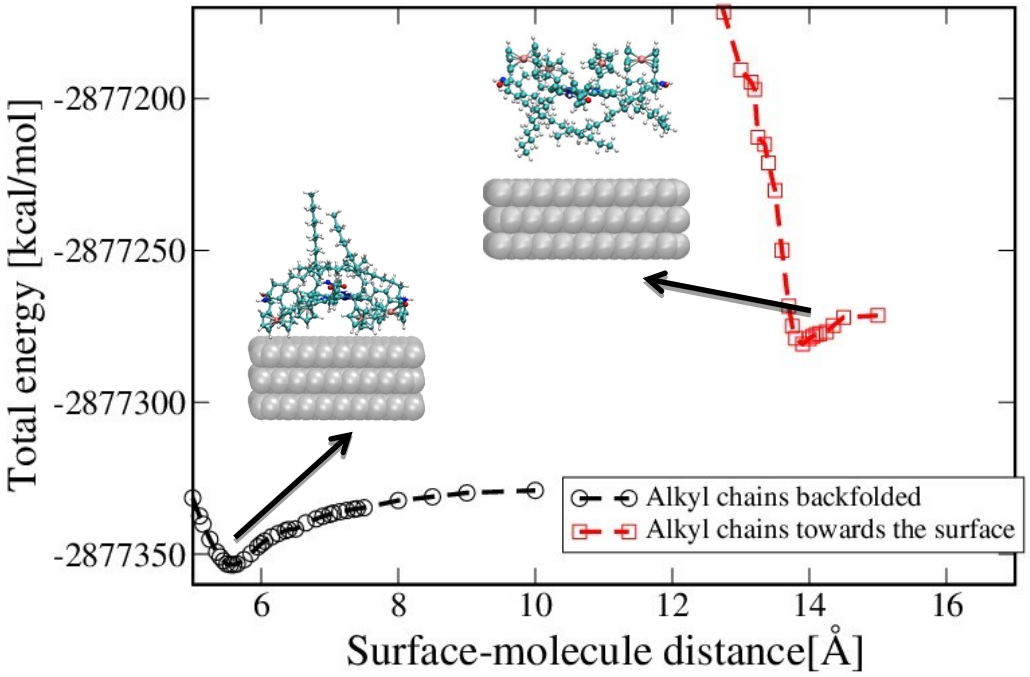


Figure S5. Computed total energy curve for the **P1** molecule with two possible configurations of the long alkyl chains: backfolded and facing the HOPG substrate. The relevant distance between the molecule and the HOPG surface was chosen to be the distance of the normal vector centered at the plane created by the four iron atoms with a carbon atom of the surface**.**

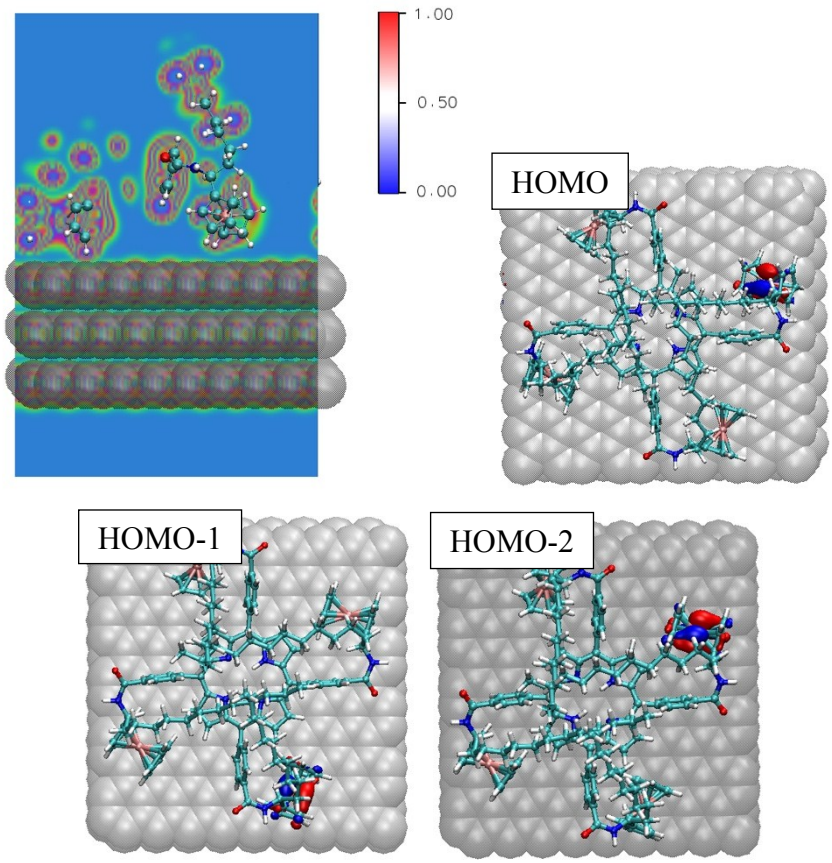
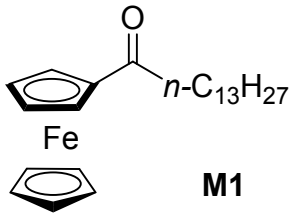
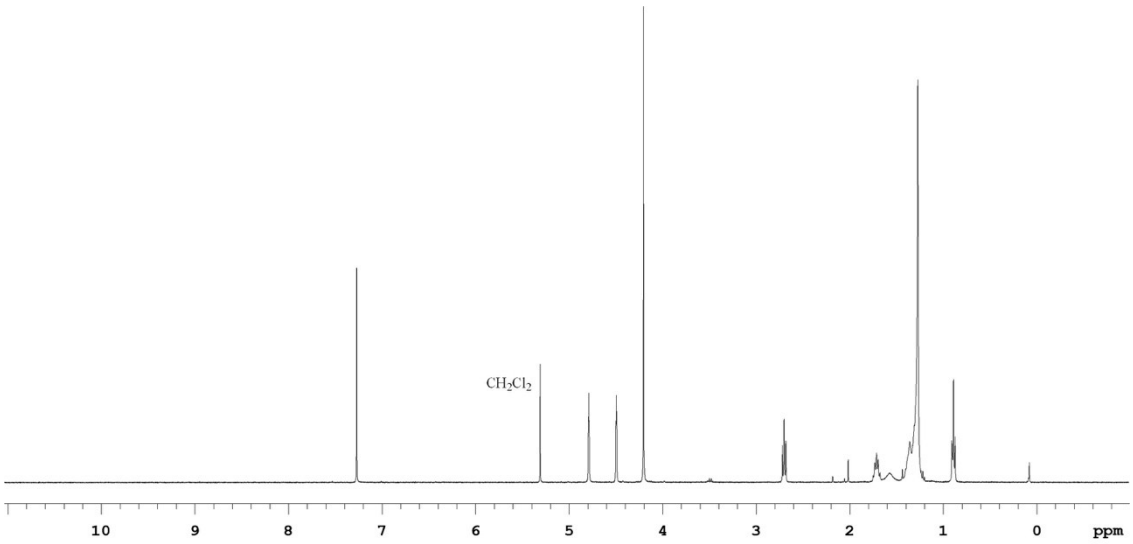


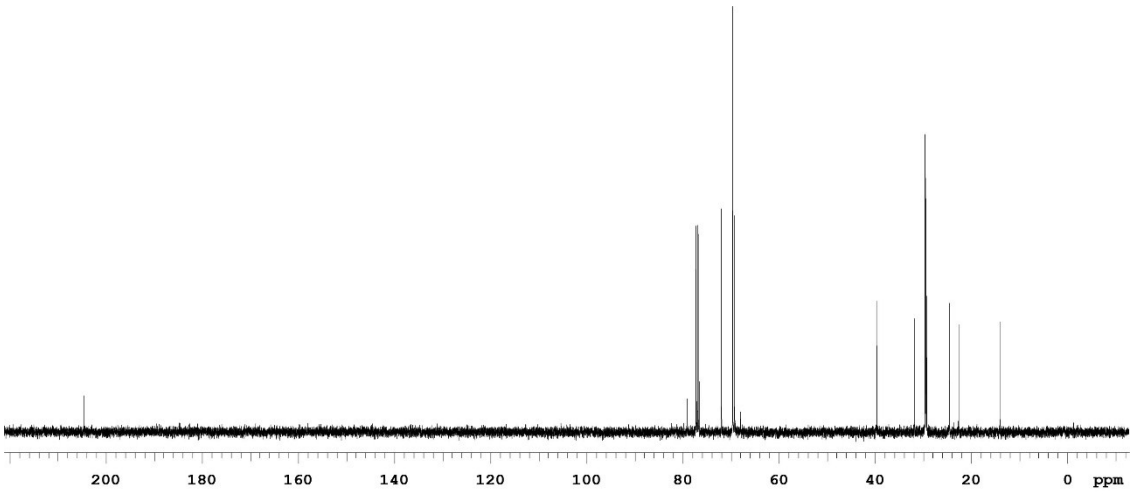
Figure S6. Upper panel: Electron localization function (ELF) for the **P1** molecule on HOPG. Lower panel: Frontier occupied molecular orbitals (HOMO, HOMO-1, HOMO-2).

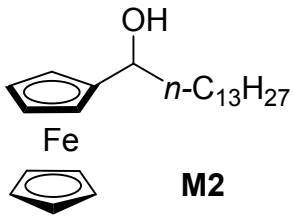
5. Copies of NMR spectra



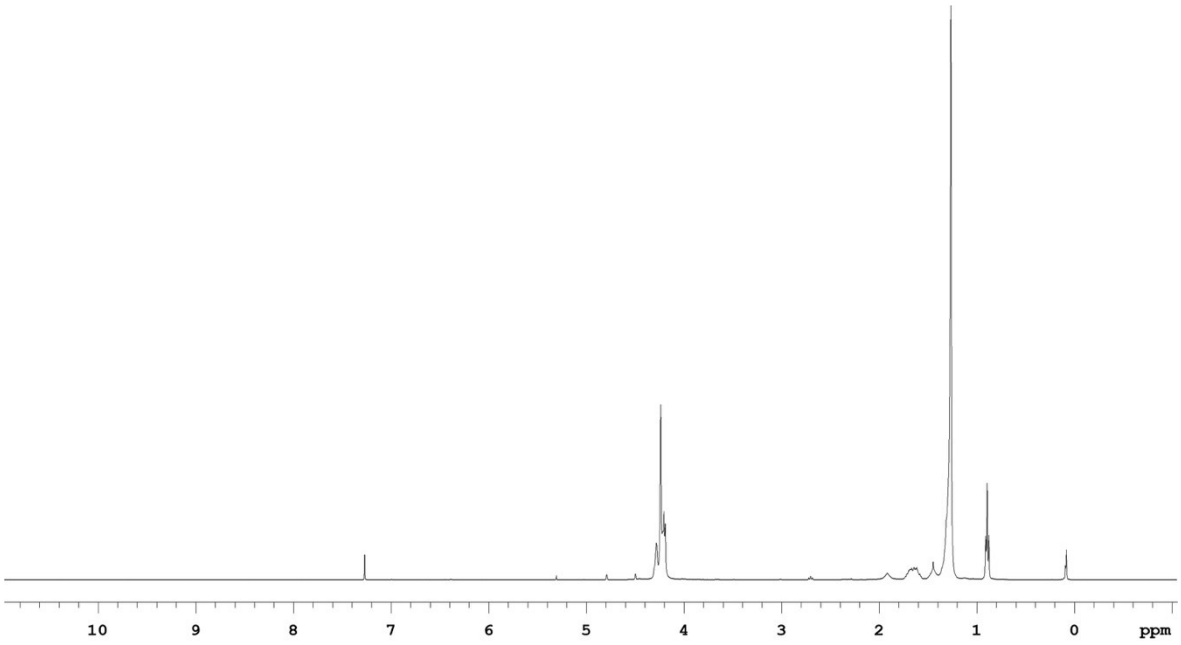
M1






OH
n-C13H27
Fe

M2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7
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5
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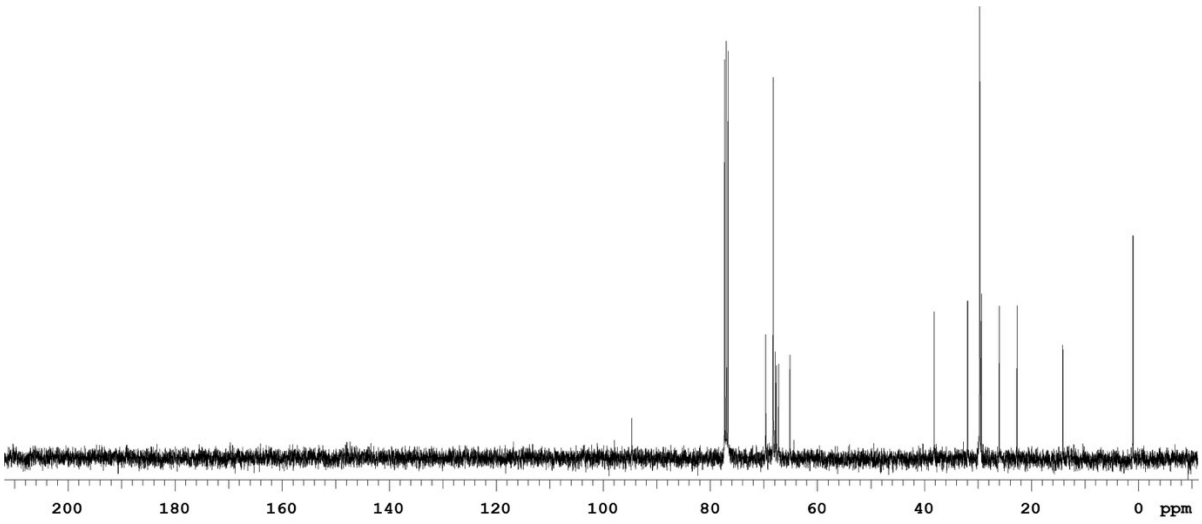
ppm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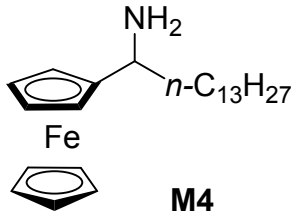
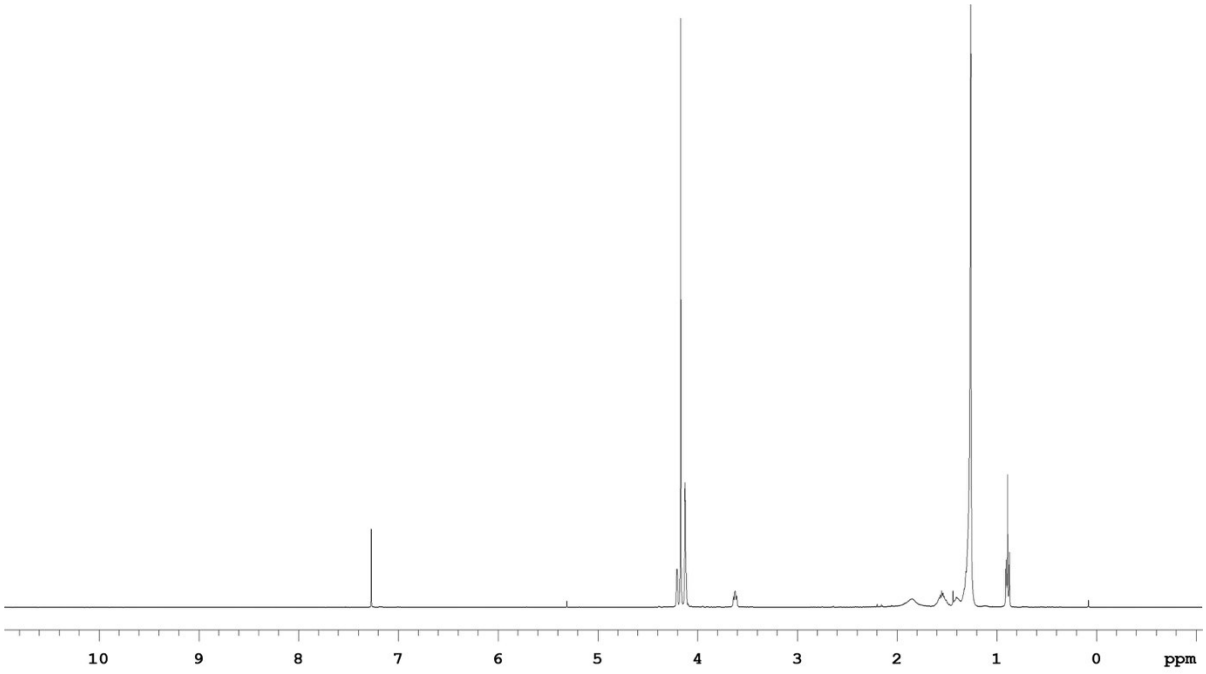

NH2
n-C13H27
Fe

M4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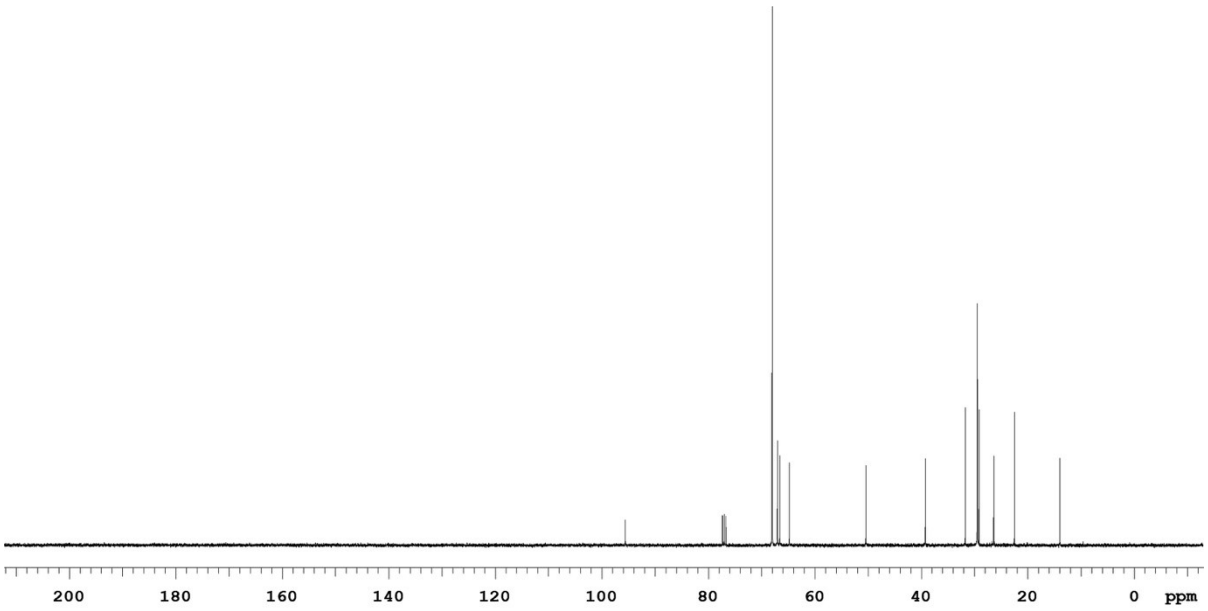
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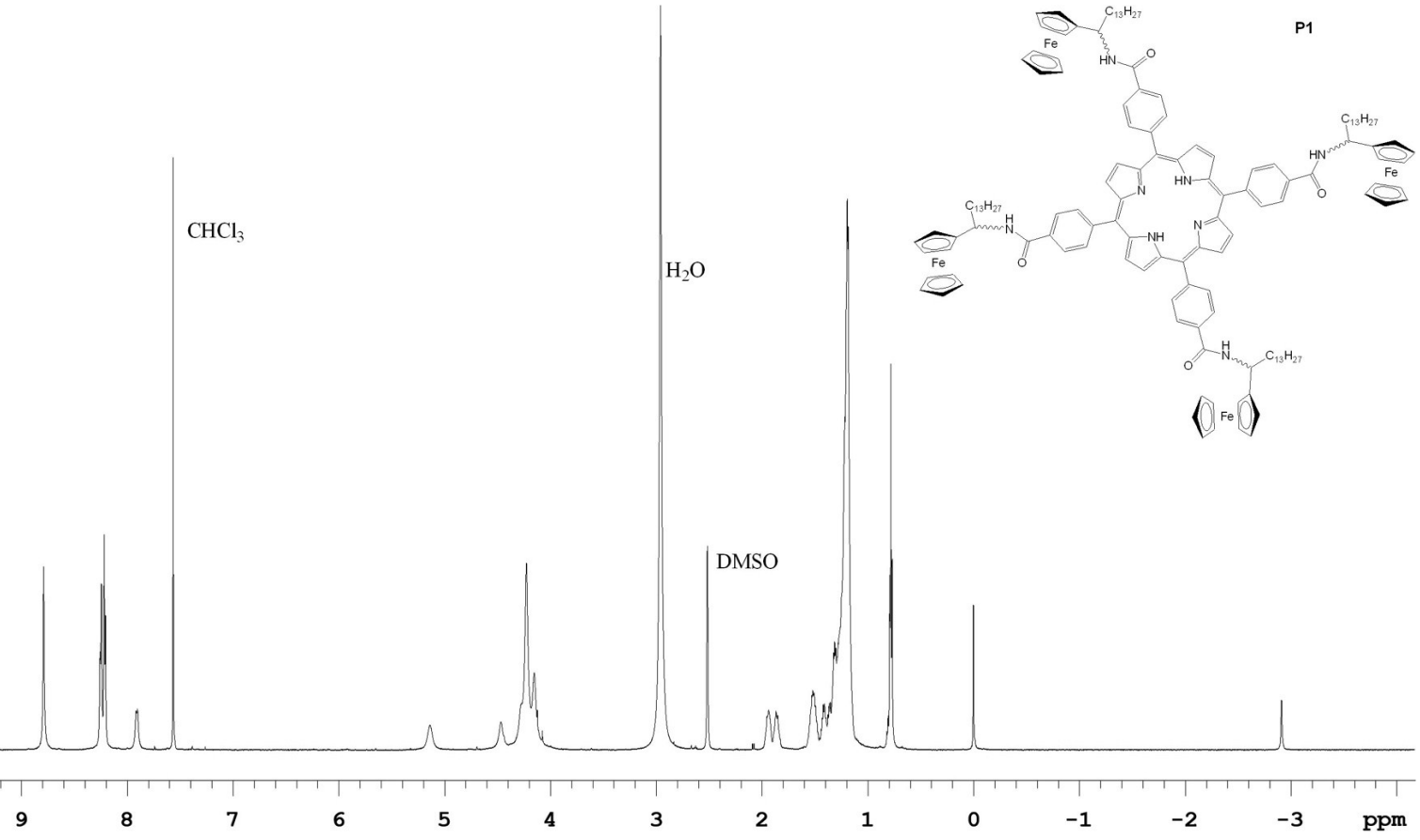
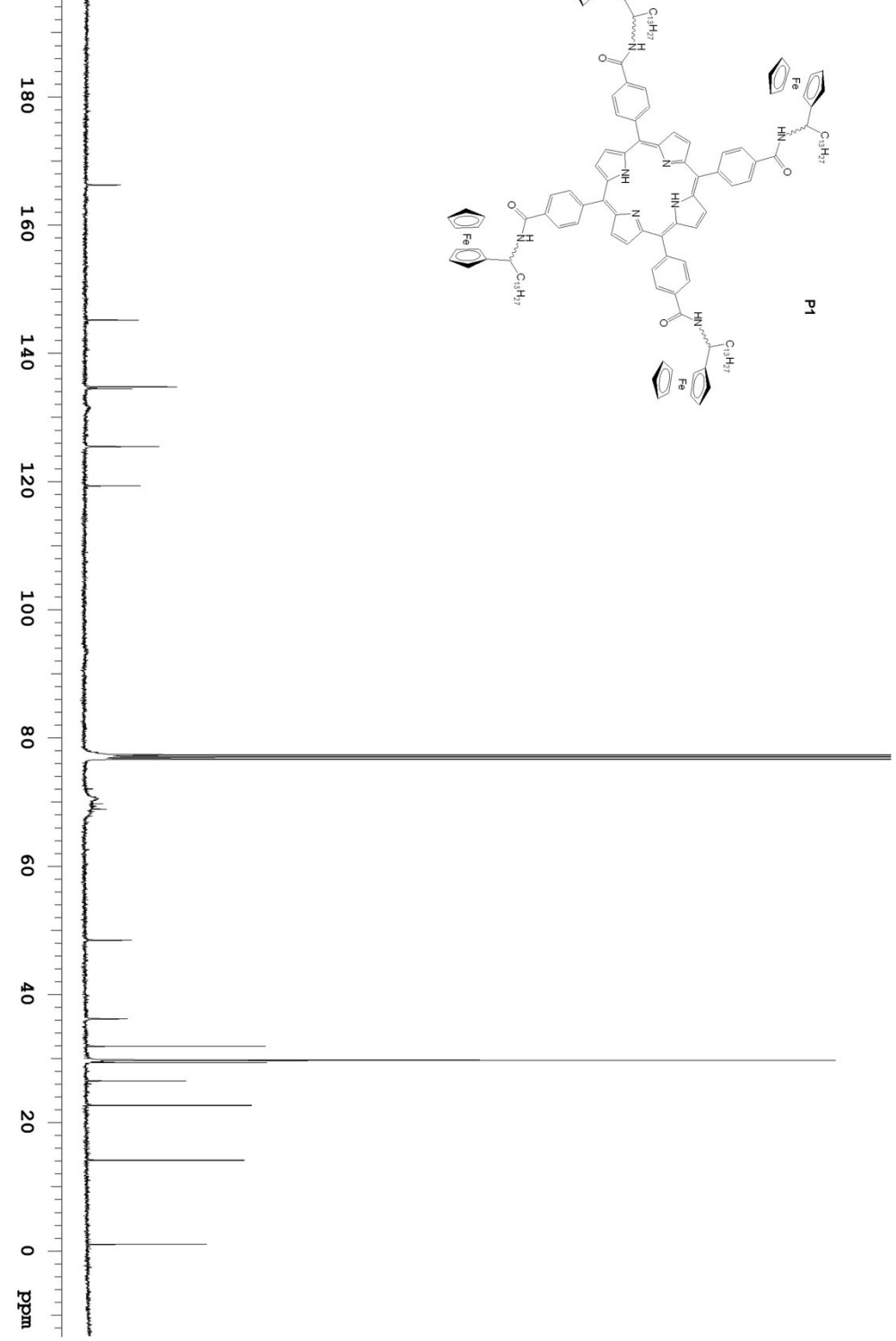

CHCl3
H2O
DMSO
P1
Fe
HN
O
C13H27
N
NH
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm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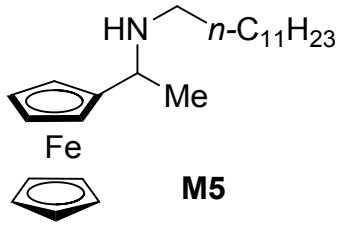
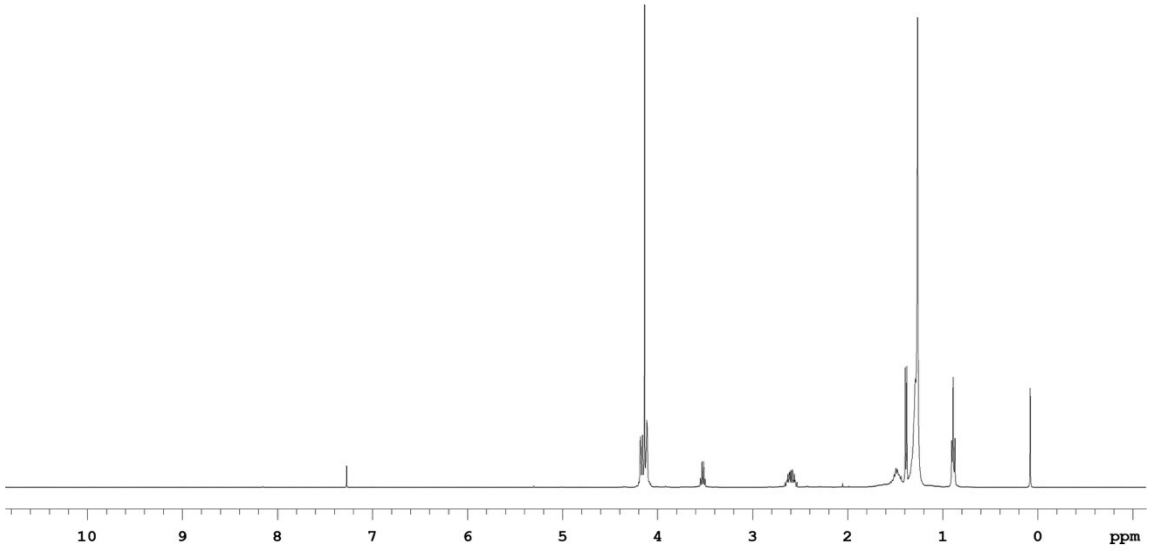
ppm
P1


HN
n-C11H23
Me
Fe

M5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4
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2
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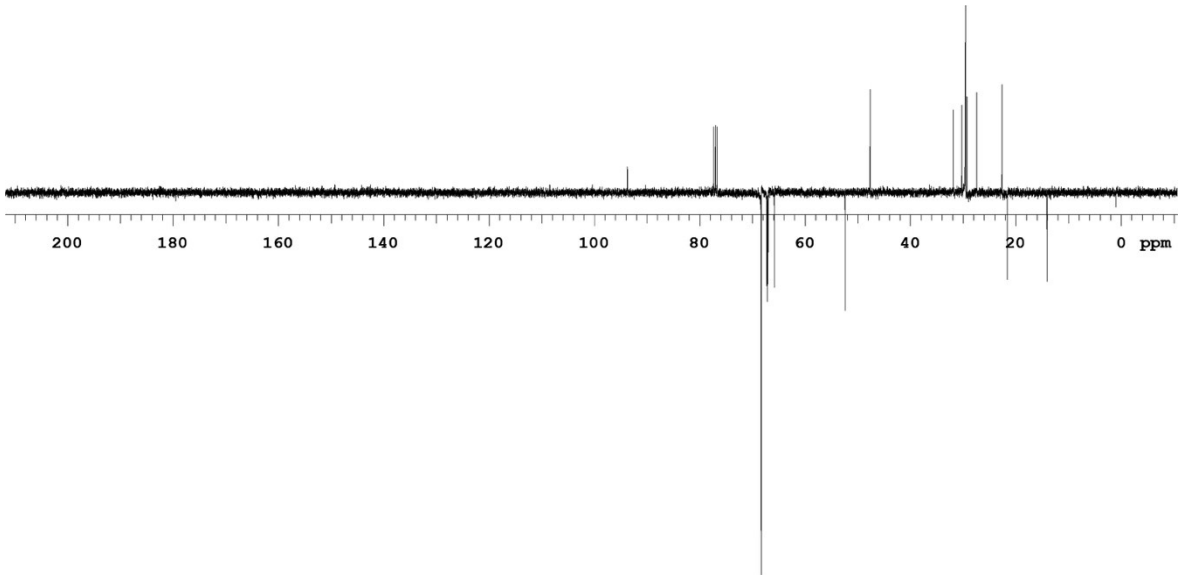
0
ppm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60
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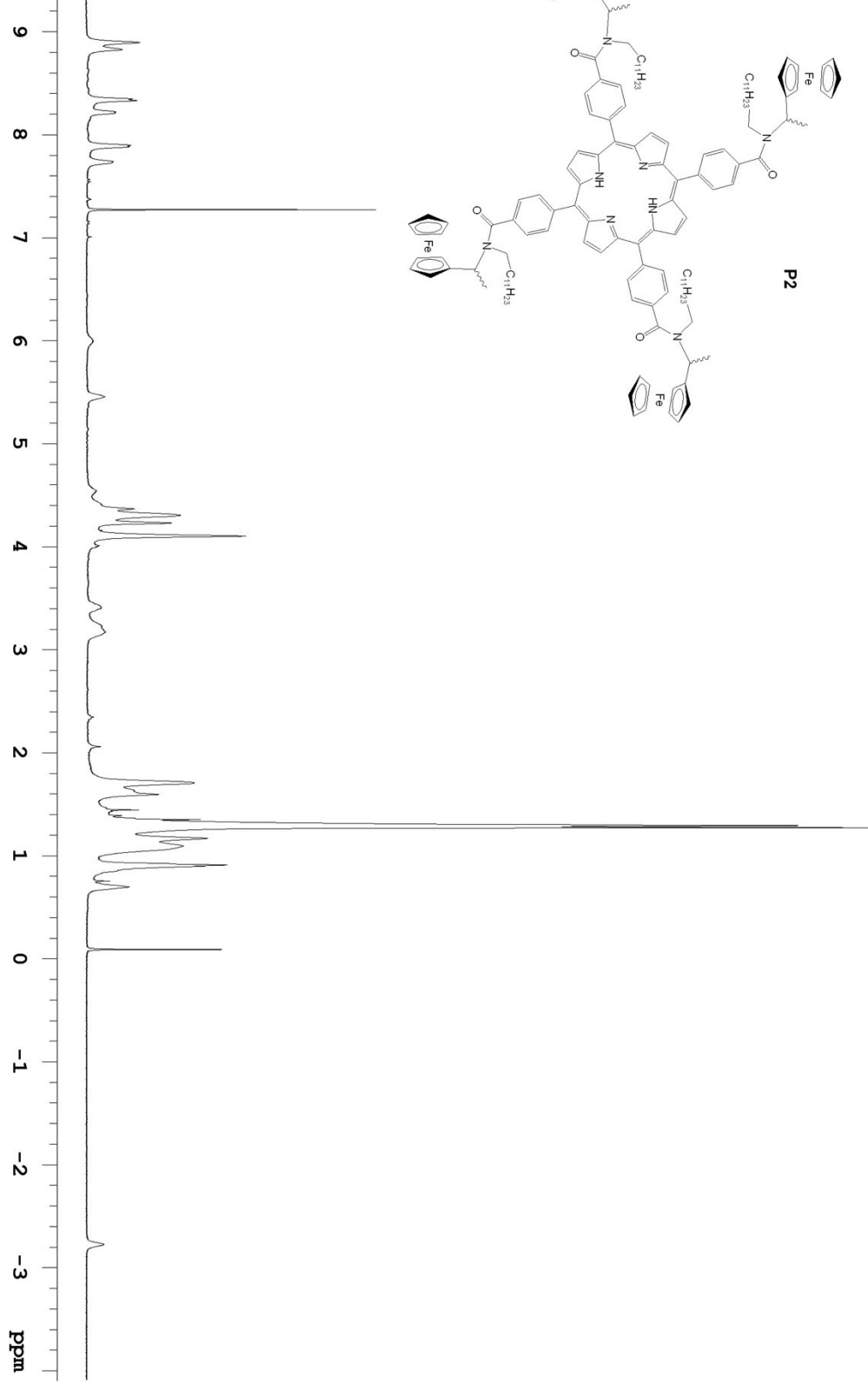
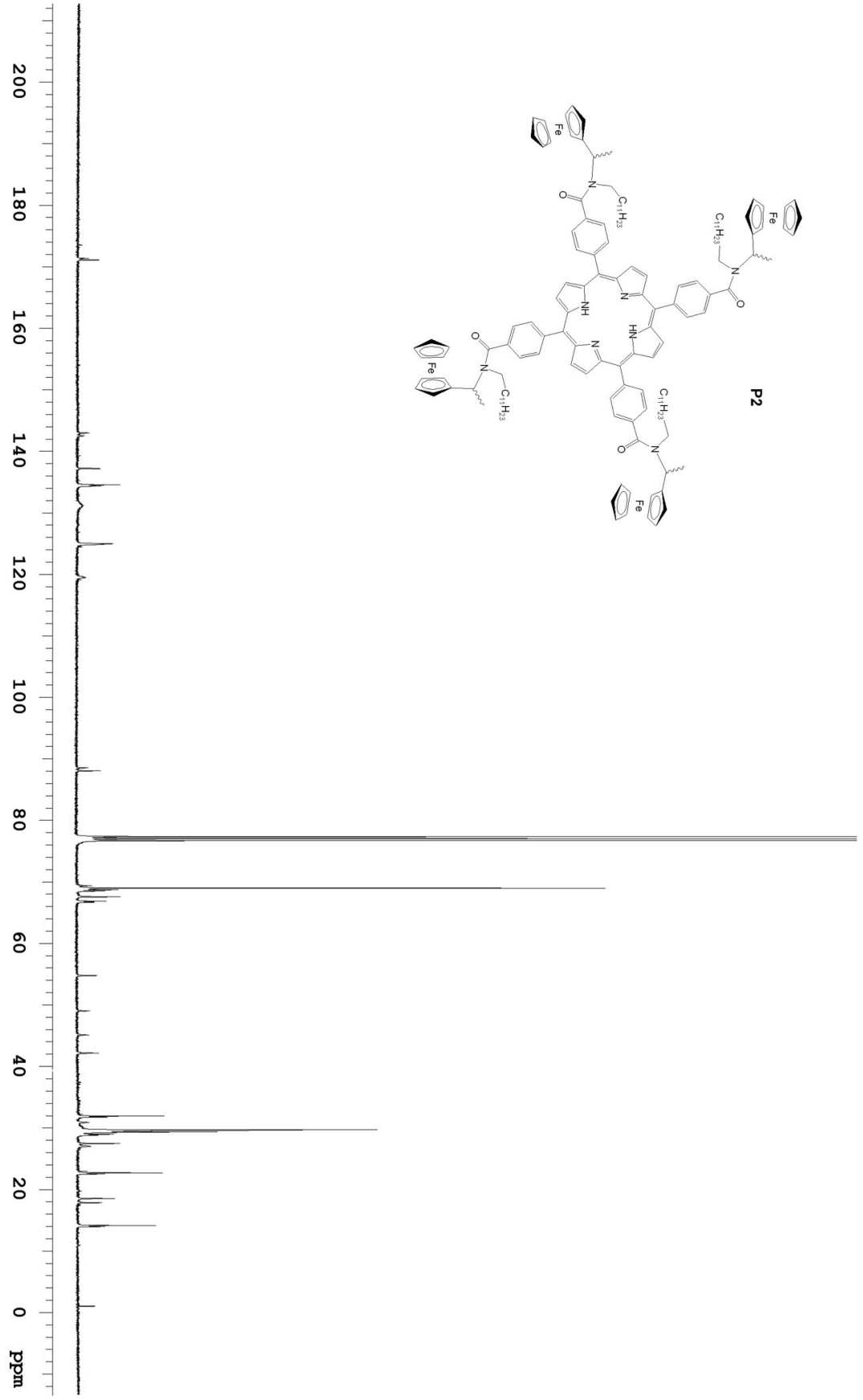
20
0 ppm


9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
P2
Fe
$C_{11}H_{23}$
NH
HN
N
O


200
180
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140
120
100
80
60
40
20
0
ppm
Fe
Fe
Fe
Fe
NH
HN
N
N
N
N
O
O
O
O
$C_{11}H_{23}$
$C_{11}H_{23}$
$C_{11}H_{23}$
$C_{11}H_{23}$
P2

6. References

1. Vukićević, M. D.; Ratković, Z. R.; Teodorović, A. V.; Stojanović, G. S.; Vukićević, R. D. *Tetrahedron* **2002**, *58*, 9001-9006.

2. Iurlo M.; Mengozzi L.; Rapino S.; Marcaccio M.; Perone R.C.; Masiero S.; Cozzi P.; Paolucci F. *Organometallics*, **2014**, *33*, 4986−4993

3. Sooambar C.; Troiani V.; Bruno C.; Marcaccio M.; Paolucci F.; Listorti A.; Belbakra A.; Armaroli N.; Magistrato A.; De Zorzi R.; Geremia S.; Bonifazi D. *Org. Biomol. Chem.* **2009**, *7*, 2402-2413.