Hydrogen Bonded Aryl Amide Macrocycles: Synthesis, Single Crystal Structures and Stacking Interactions with Fullerenes and Coronene

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Supporting Information

General methods. All reactions were carried out under a dry nitrogen atmosphere. All solvents were dried before use following standard procedures. Unless otherwise indicated, all starting materials were obtained from commercial suppliers and were used without further purification. Analytical thin-layer chromatography (TLC) was performed on 0.2 mm silica coated on glass plates with FB254B indicator. The ^{1}H NMR spectra were recorded on 500, or 300 MHz spectrometers in the indicated solvents. Chemical shifts are expressed in parts per million (δ) using residual solvent protons as internal standard (chloroform: δ 7.26 ppm; DMSO: δ 2.50 ppm). The ^{13}C NMR spectra were recorded on 300 MHz spectrometer in the indicated solvents. Chemical shifts are expressed in parts per million using $CDCl_3$ as internal standard (chloroform: δ 77.0 ppm). The ^{19}F NMR spectra were recorded on 300 MHz spectrometer in the indicated solvents. Chemical shifts are expressed in parts per million using $CFCl_3$ as external standard ($CFCl_3$: δ 0 ppm). Elemental analysis was carried out at the SIOC analytical center.

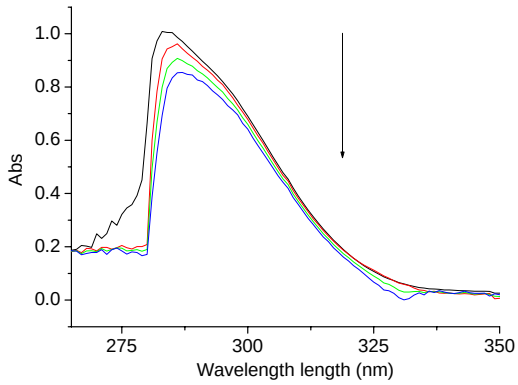


Figure 1S. Absorption spectral changes of **1** (2×10^{-5} M) with the addition of C_{60} (0 – 4.0×10^{-5} M) in the mixture of chloroform and toluene (v/v = 1 : 1) at 25 °C. The absorbance of C_{60} has been subtracted for all the spectra.

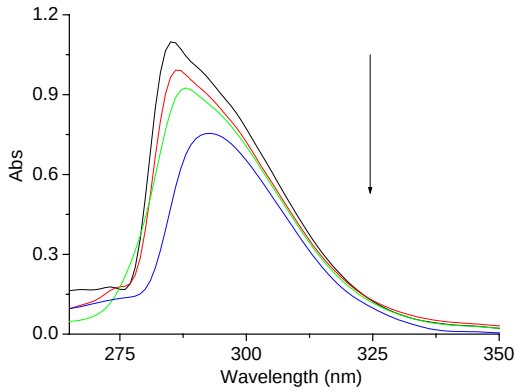


Figure 2S. Absorption spectral changes of **1** (2×10^{-5} M) with the addition of C_{70} (0 - 4×10^{-5} M) in the mixture of chloroform and toluene (v/v = 1 : 1) at 25 °C. The absorbance of C_{60} has been subtracted for all the spectra.

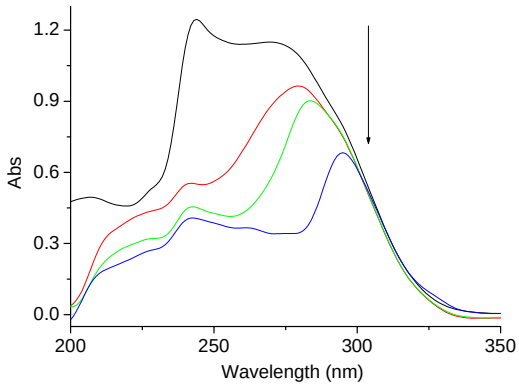


Figure 3S. Absorption spectral changes of **3** (2×10^{-5} M) with the addition of C_{60} (0 - 3×10^{-5} M) in chloroform at 25 °C. The absorbance of C_{60} has been subtracted for all the spectra.

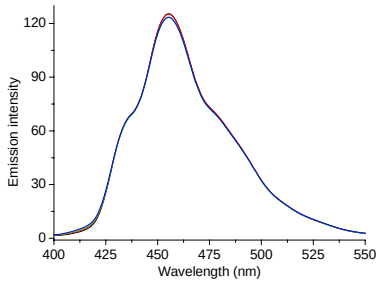


Figure 4S. Fluorescent spectra of coronene (5×10^{-5} M) with the addition of **1** (0 to 2×10^{-4} M) in chloroform at 25 °C (excitation wavelength = 340 nm).

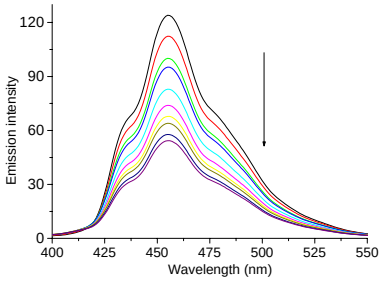


Figure 5S. Fluorescent spectra of coronene (5×10^{-5} M) with the addition of **2c** (0 to 4.5×10^{-4} M) in chloroform at 25 °C (excitation wavelength = 340 nm).

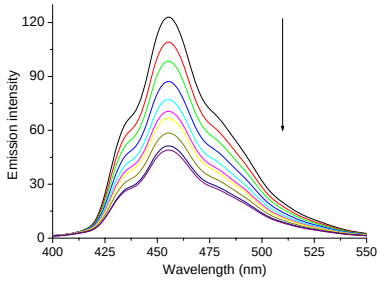


Figure 6S. Fluorescent spectra of coronene (5×10^{-5} M) with the addition of **3** (0 to 4.5×10^{-4} M) in chloroform at 25 °C (excitation wavelength = 340 nm).

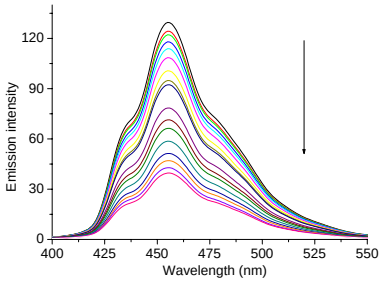


Figure 7S. Fluorescent spectra of coronene ((5×10^{-5} M) with the addition of **4** (0 to 1.2×10^{-4} M) in chloroform at 25 °C (excitation wavelength = 340 nm).

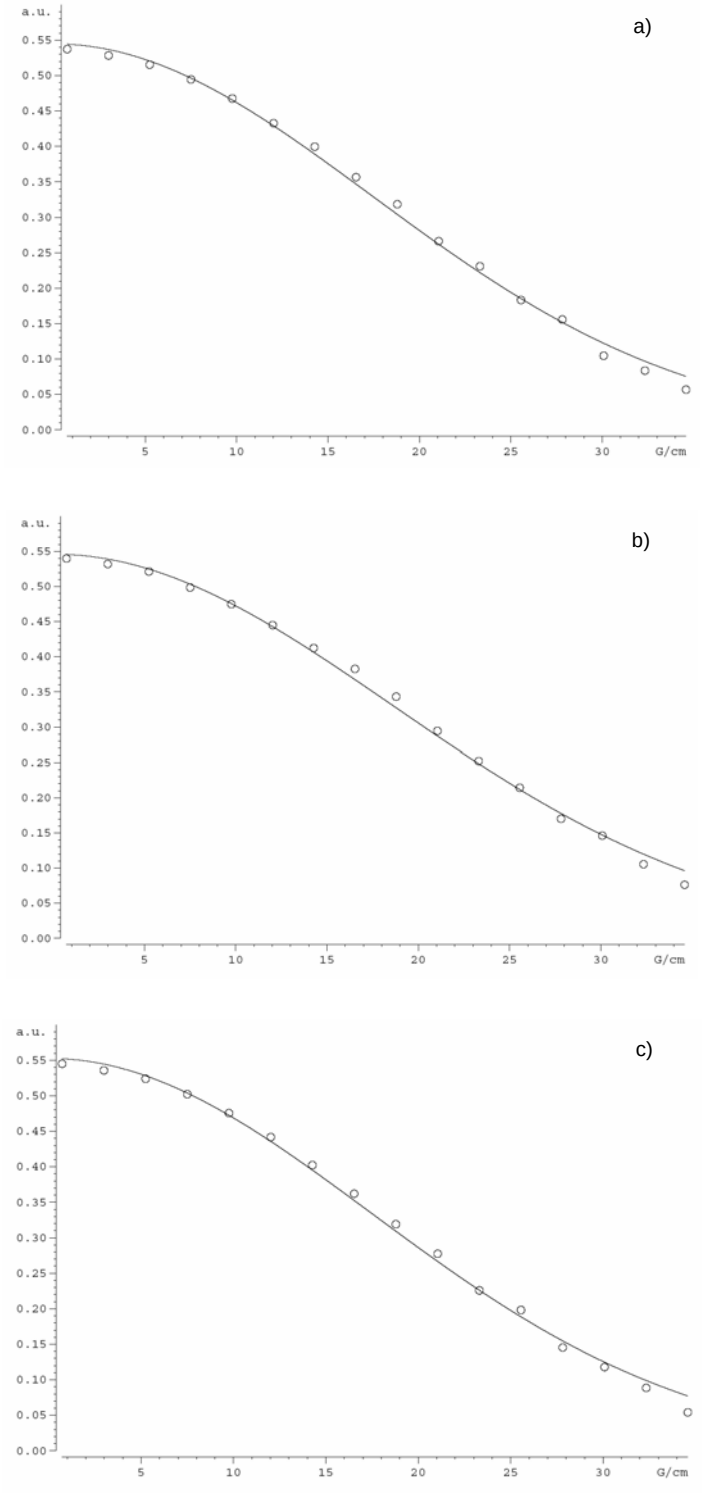
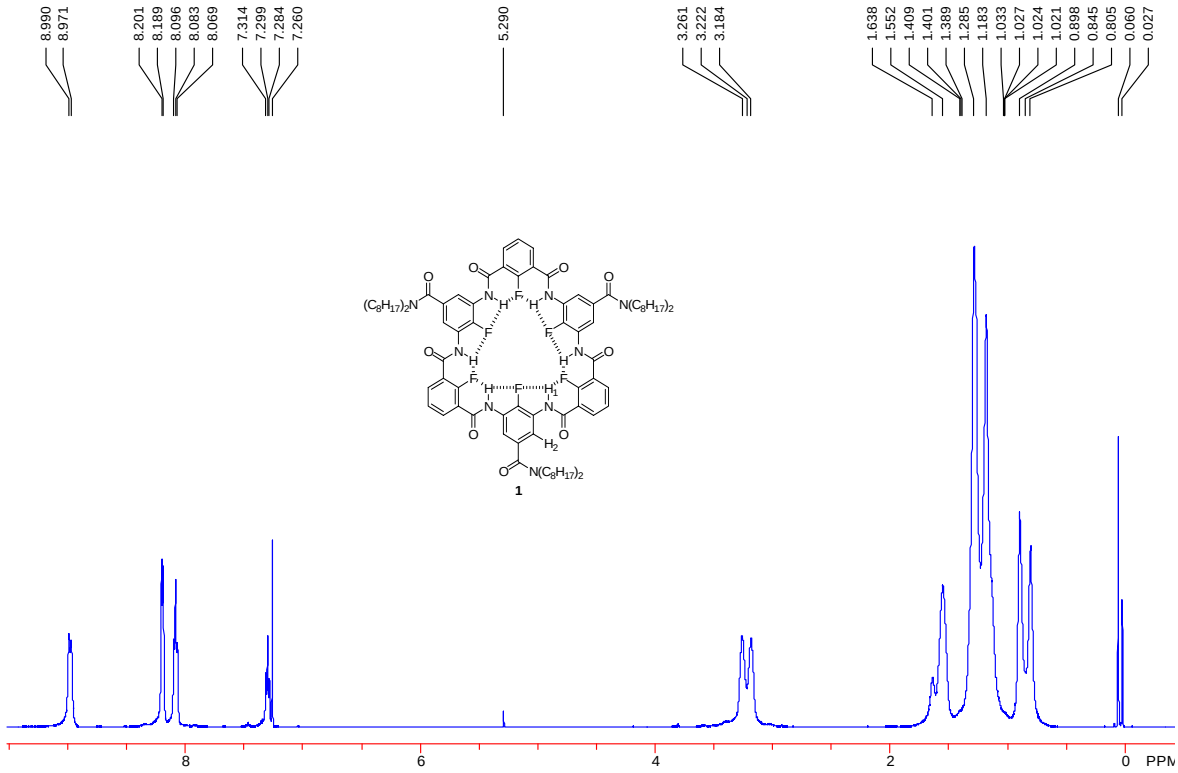


Figure 8S. Simulated diffusion decay curves by varying the gradient strength from 2 to 95% in 16 steps for the diffusion constant of the amide proton of (a) **1** (5 mM), (b) **1** + C_{60} (1:1, 5 mM) and (c) **1** + C_{70} (1:1, 5 mM) in $CDCl_3$-CS_2 (v/v 1:1).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3.222
3.184
1.638
1.552
1.409
1.401
1.389
1.285
1.183
1.033
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6
4
2
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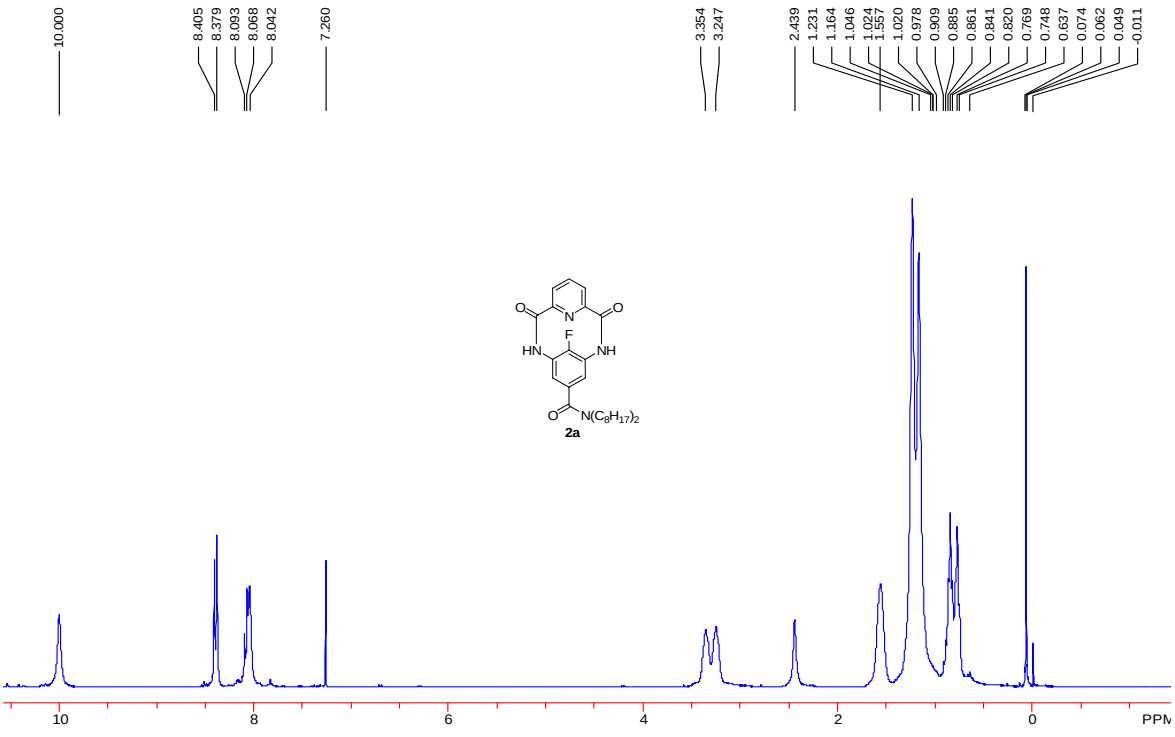
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1.024
1.557
1.020
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0.909
0.885
0.861
0.841
0.820
0.769
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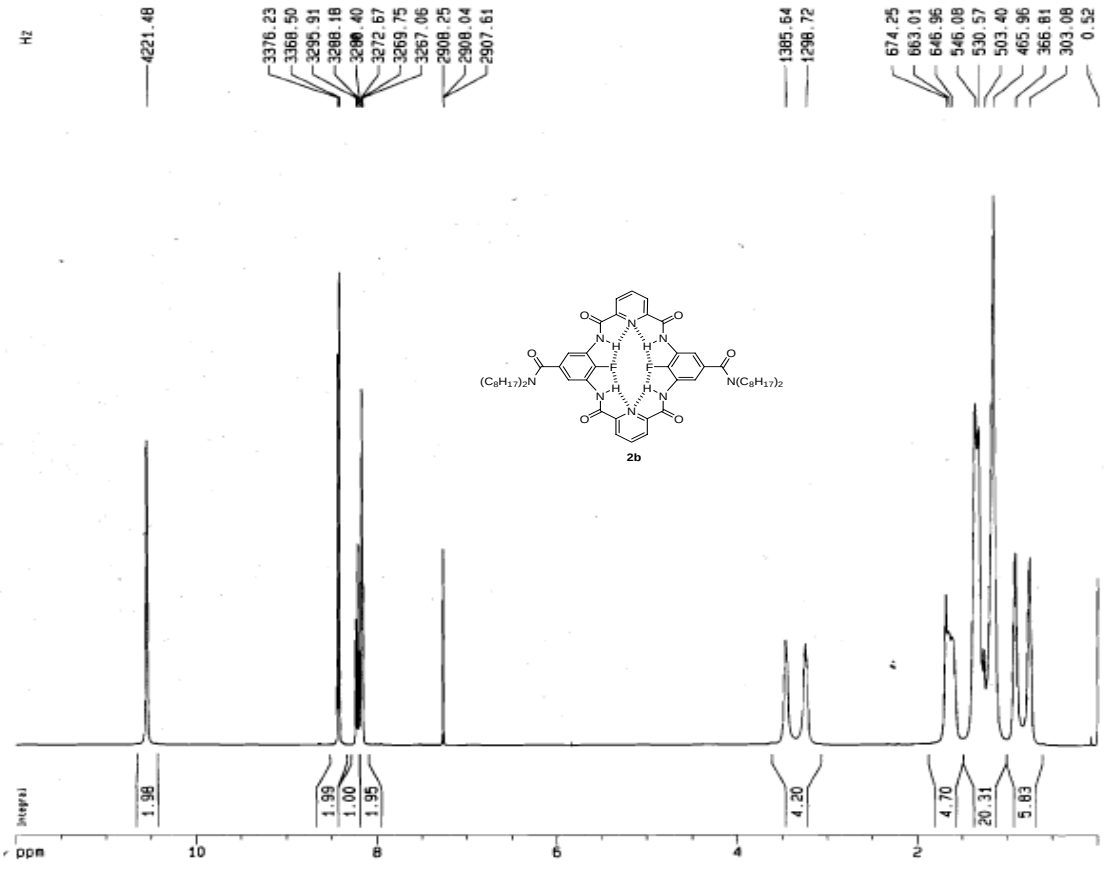
2a
10
8
6
4
2
0
PPM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2907.61
1385.64
1298.72
674.25
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0.52
Integral
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1.99
1.00
1.95
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8
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4
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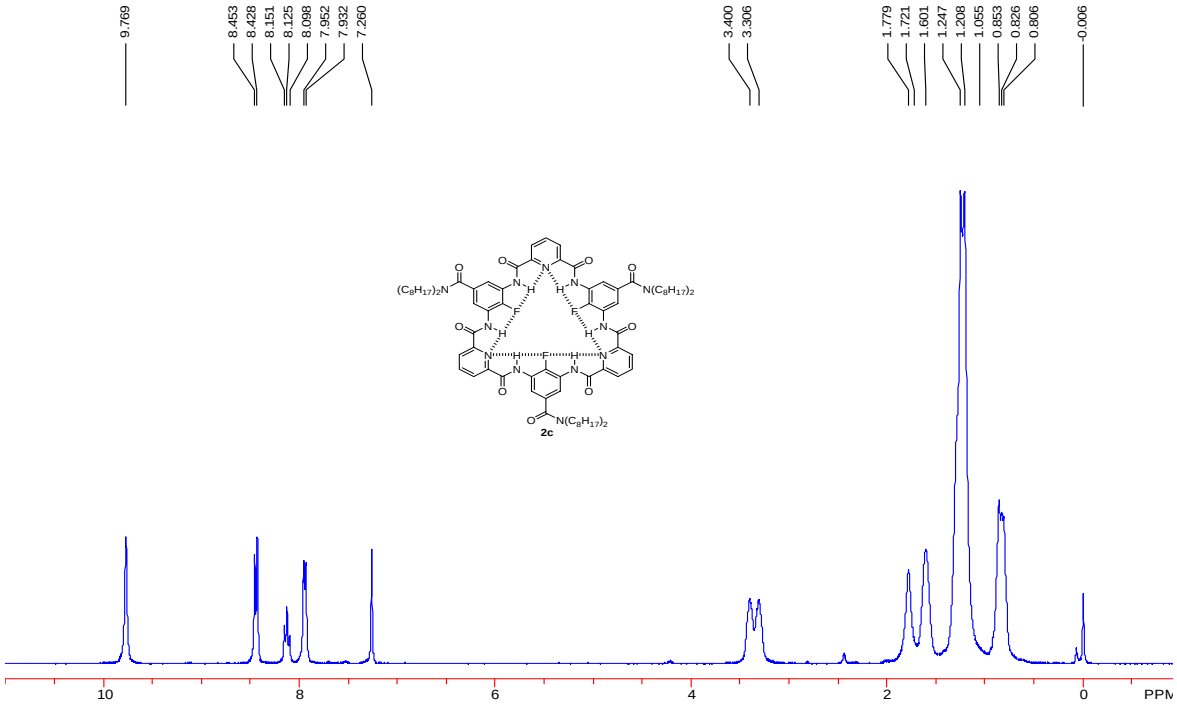
N(C8H17)2
2b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1.055
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0.826
0.806
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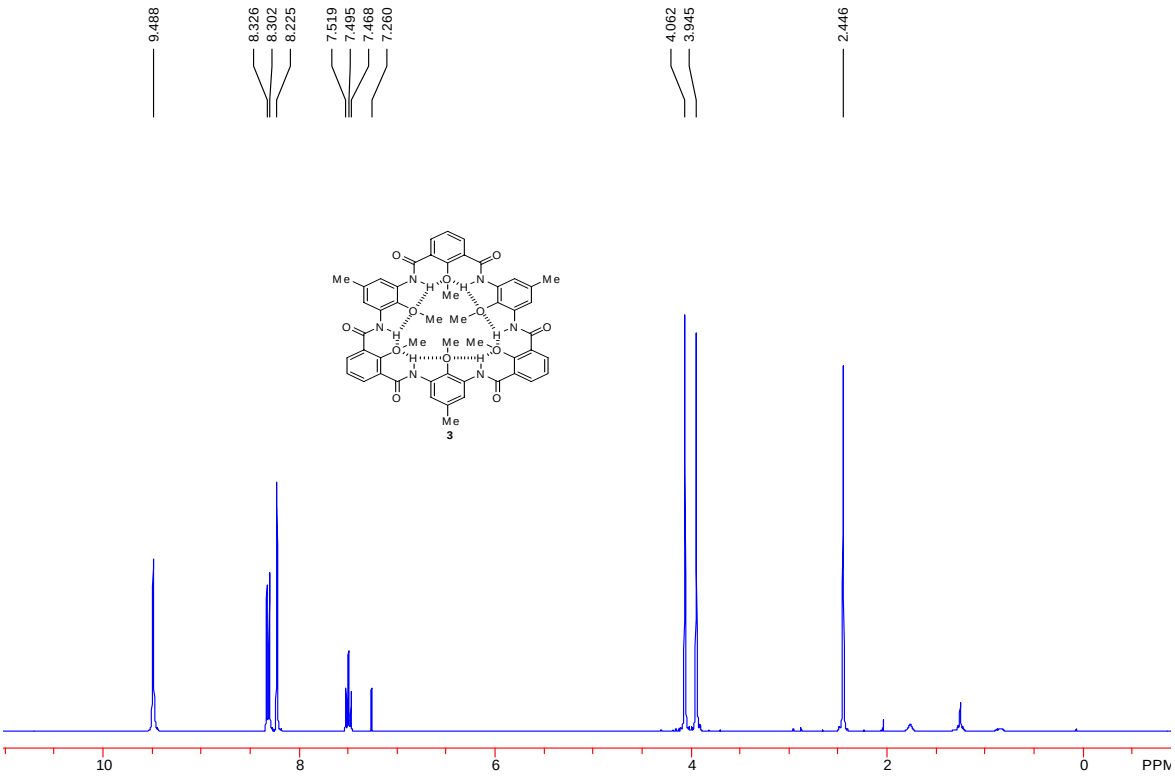
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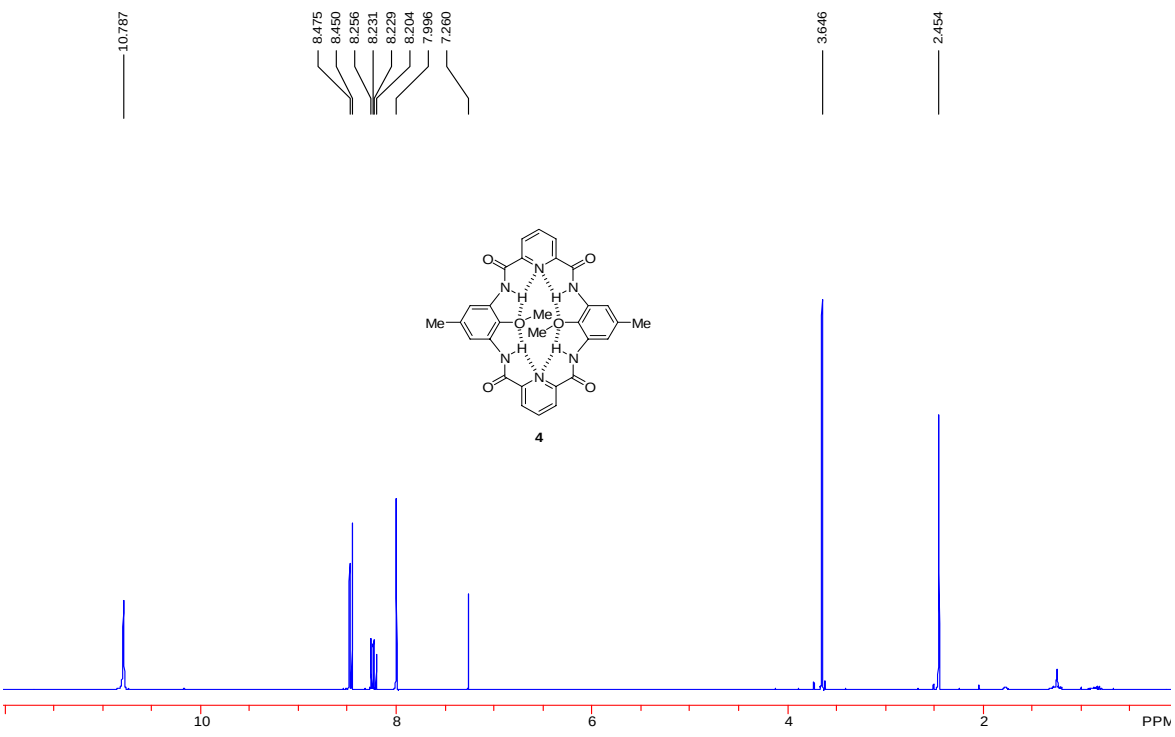
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4
2
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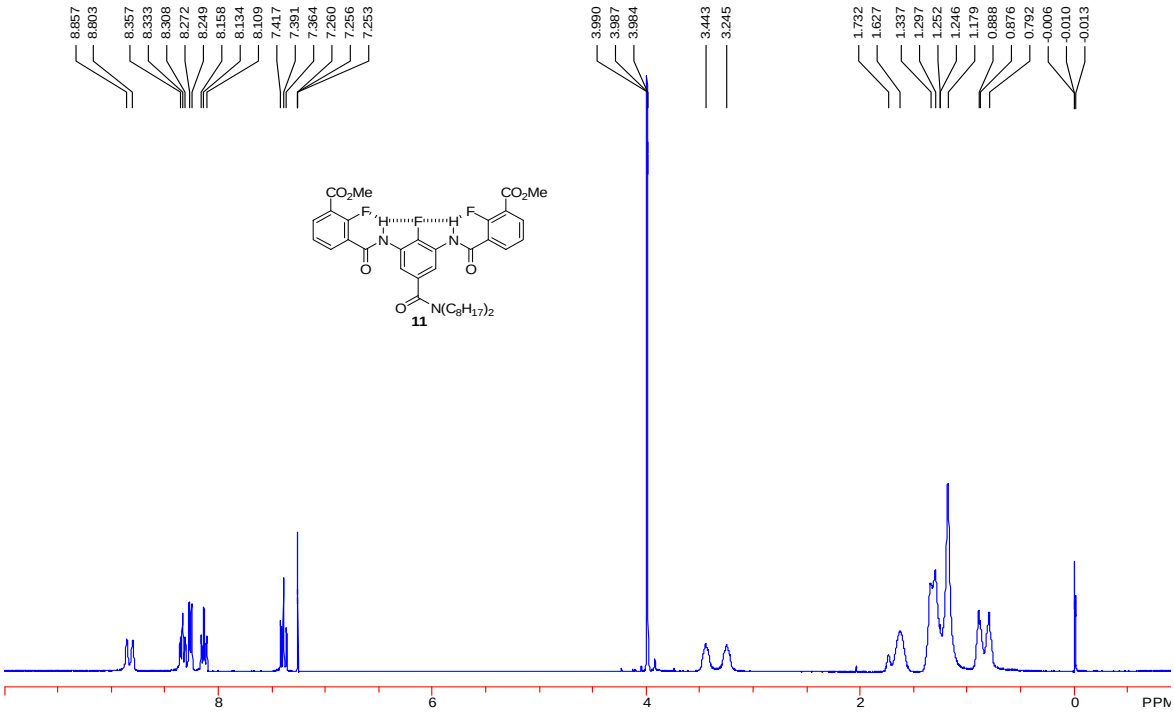
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1.627
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CO2Me
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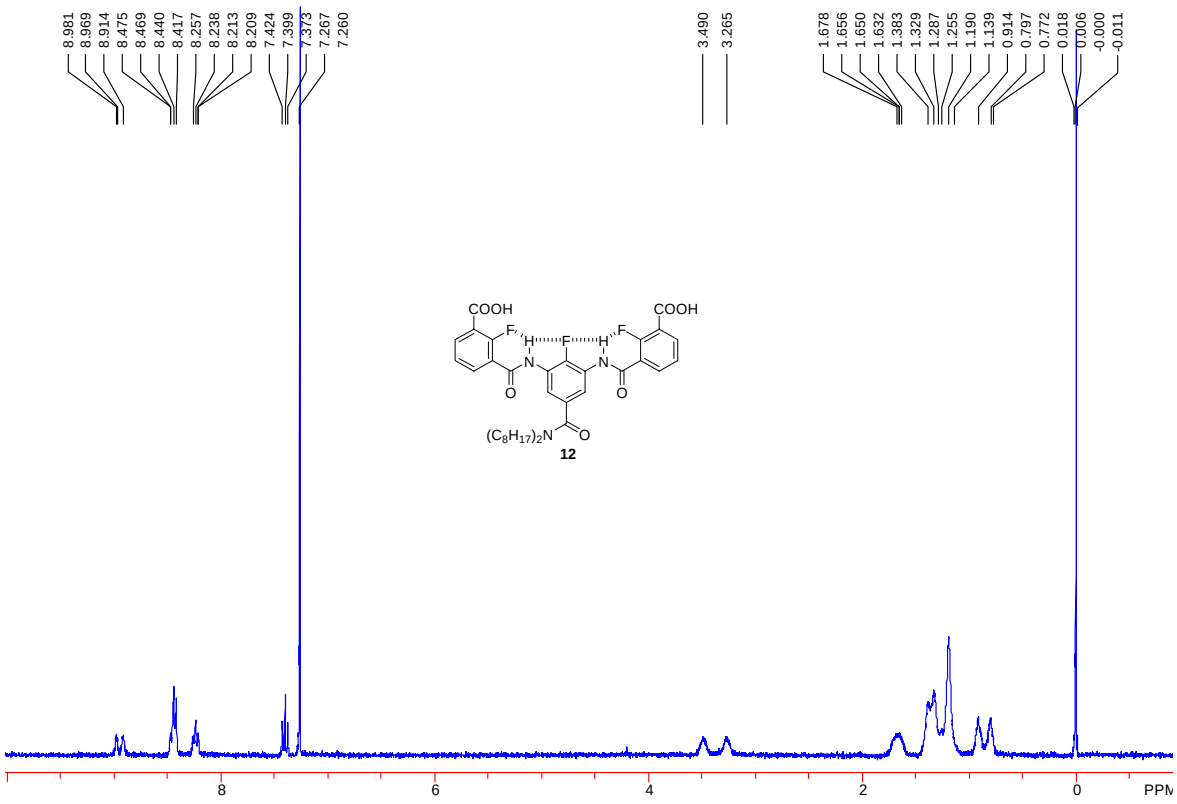
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1.656
1.650
1.632
1.383
1.329
1.287
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1.139
0.914
0.797
0.772
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COOH
COOH
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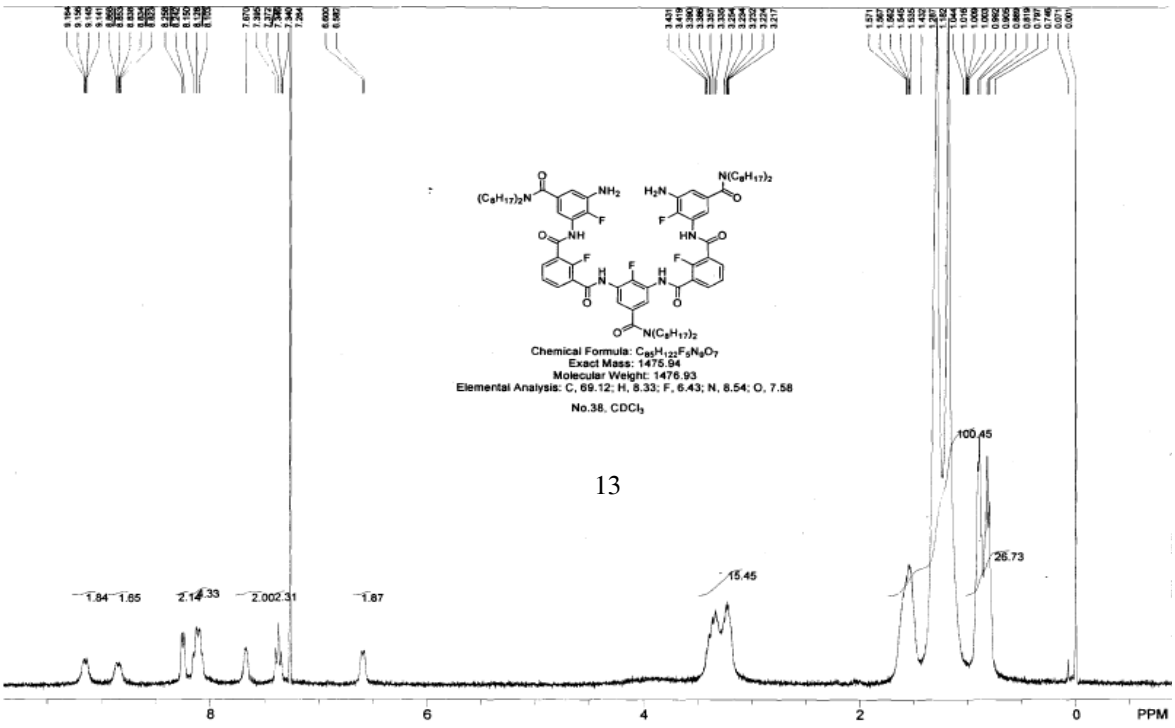
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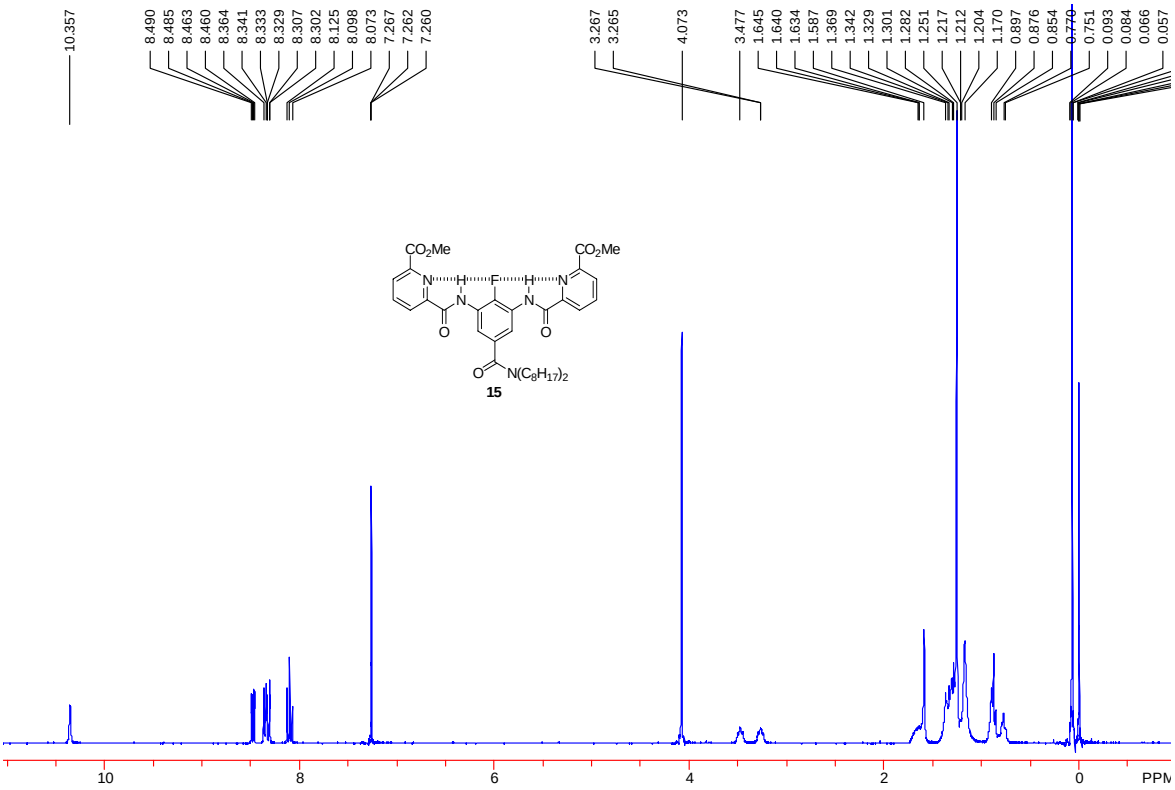
PPM

13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CO2Me
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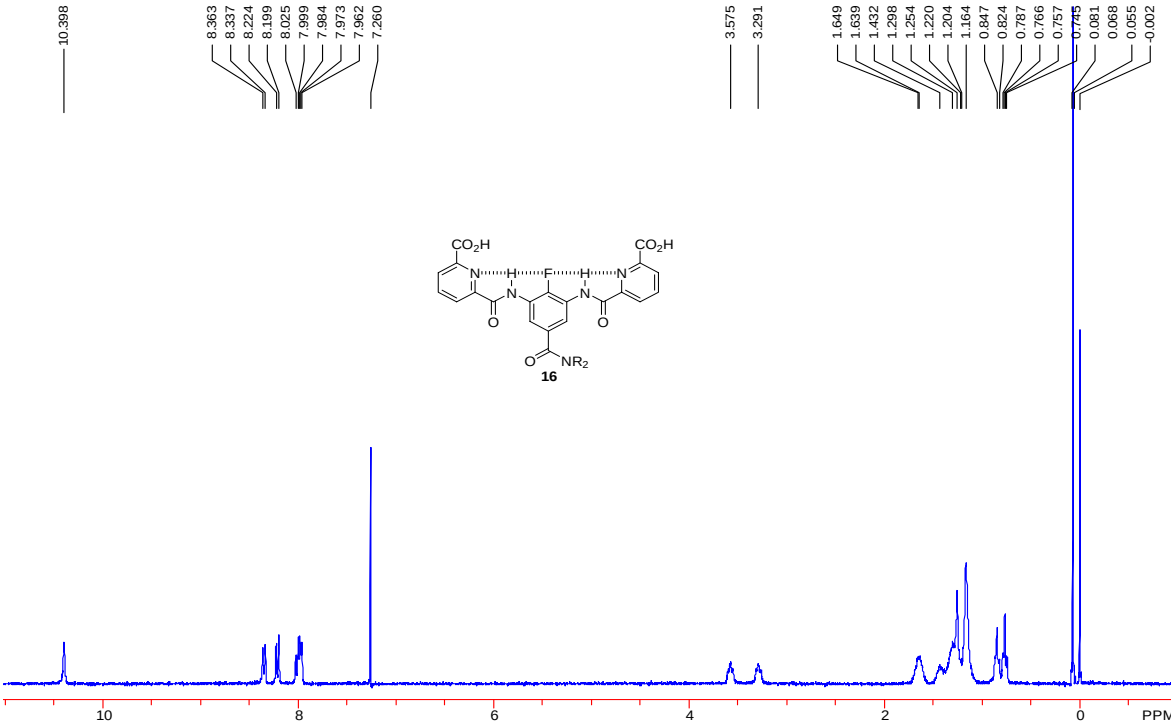
PPM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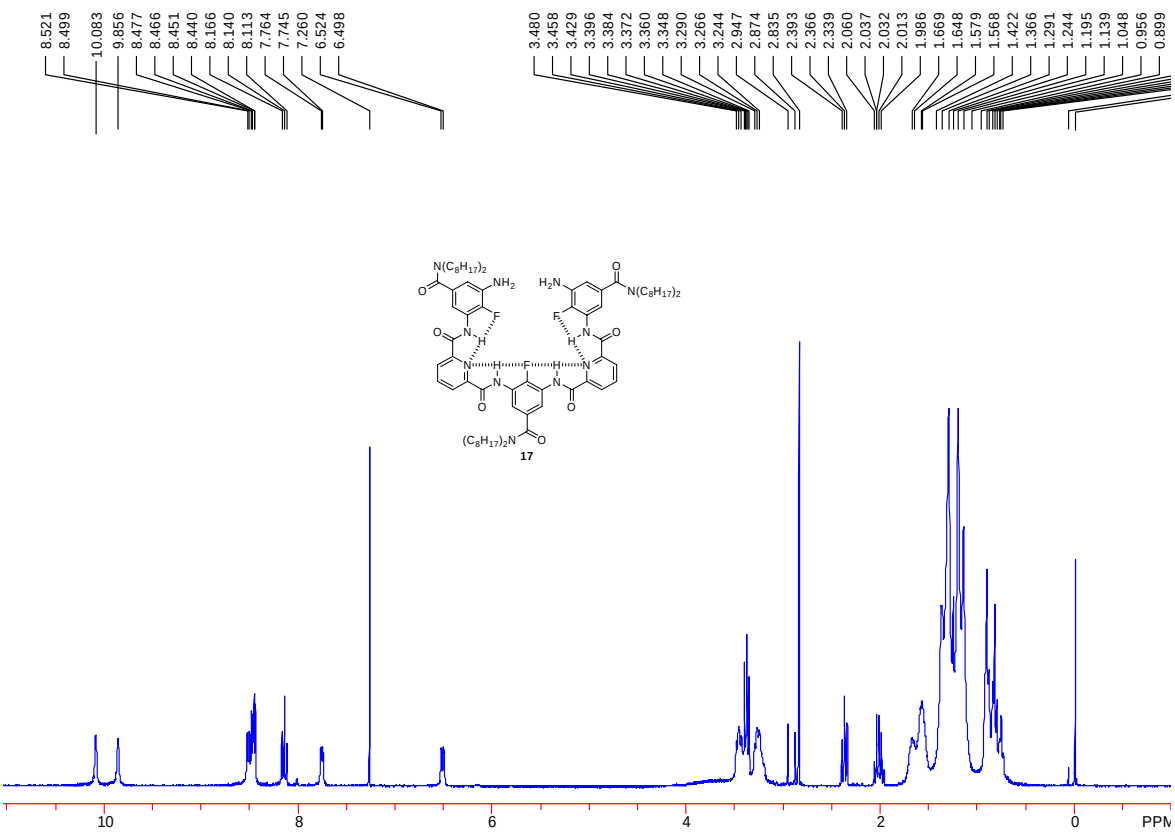
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