

# Chemistry–A European Journal

Supporting Information

## **Consequences of Amide Connectivity in the Supramolecular Polymerization of Porphyrins: Spectroscopic Observations Rationalized by Theoretical Modelling**

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## 1. Materials and methods:

### 1.1 Materials

Unless stated otherwise, reagents and solvents were obtained from commercial sources and used without further purification. All solvents were of AR grade or of spectrophotometric grade, when used in spectroscopy experiments. Reactions were conducted under argon atmosphere and in dried glassware unless noted otherwise. Flash column chromatography was done on a Biotage Isolera One system with an ultraviolet detector.

### 1.2 Characterization

MALDI-TOF mass spectra were measured using a PerSeptive Biosystem Voyager-DE PRO spectrometer using either  $\alpha$ -cyano-4-hydroxycinnamic acid (CHCA) or 2-[(2E)-3-(4-tert-butylphenyl)-2-methylprop-2-enylidene]malononitrile (DCTB) as matrices. IR spectra were obtained on a Perkin-Elmer spectrum two FT-IR spectrometer. Atomic force microscopy (AFM) experiments were performed on an Asylum MFP3 system with Olympus AC240 tips in tapping mode. Image analysis for AFM was done with Gwyddion software version 2.50. Mica sheets used in the experiment were of V1-grade and freshly cleaved before dropcasting the solutions.

### 1.3 Spectroscopy

UV/Vis and circular dichroism (CD) spectra were recorded on a Jasco J-815 spectropolarimeter with a Jasco PFD-425S/15 Peltier. The measurement parameters for sensitivity, scanning rates and ranges were chosen appropriately for each experiment. Cuvette cells with an optical path length of either 1 mm or 1 cm were used for all experiments.

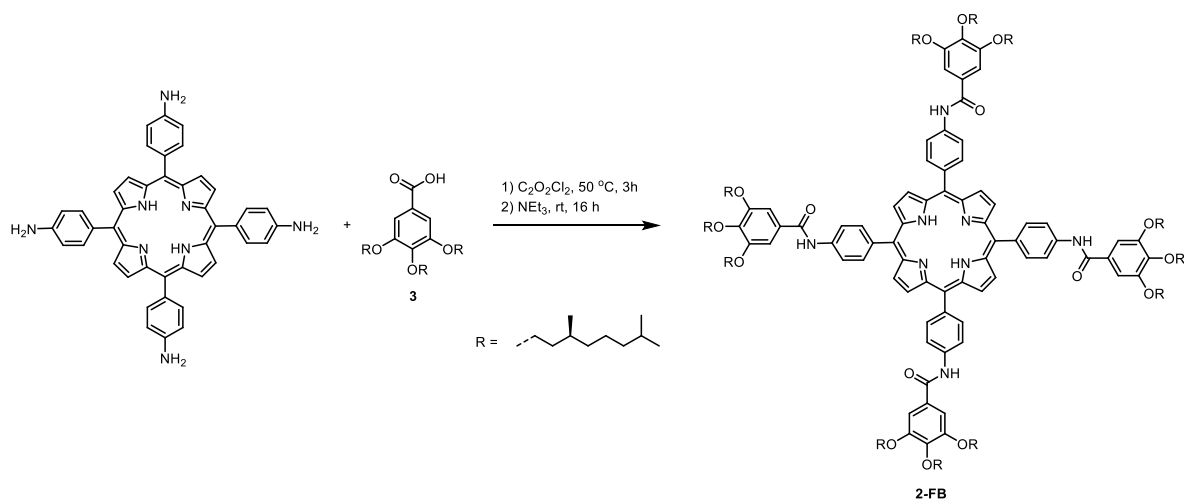
### 1.4 Sample preparation

Stock solutions (100  $\mu$ M) were prepared by weighing the compound into a vial and adding the appropriate amount of solvent. One milliliter of a polar solvent (dichloromethane or chloroform) was added to break up aggregates and re-evaporated. Then methylcyclohexane (MCH) was added, heated with a heat gun and sonicated until complete dissolution. Samples for measurements were prepared by diluting the stock solution to the respective concentration. The molar dichroism  $\Delta\epsilon$  was calculated as  $\Delta\epsilon = \text{CD effect} / (c \cdot l)$ , with the concentration  $c$  ( $\text{mol} \cdot \text{L}^{-1}$ ) and the optical path length  $l$  (cm).

## 2. Synthesis

The synthesis of (*S*)-3,7-dimethyloctyl functionalized acid **3**, free base ligand **1-FB** and zinc porphyrin **1-Zn** has been published previously.<sup>1</sup> The synthesis of **2-FB** was adapted from literature.<sup>2</sup> Tetra-4-aminophenyl porphyrin was purchased from PorphyrChem and used without further purification.

### 2.1 Synthesis of *N*-centered ligand **2-FB**

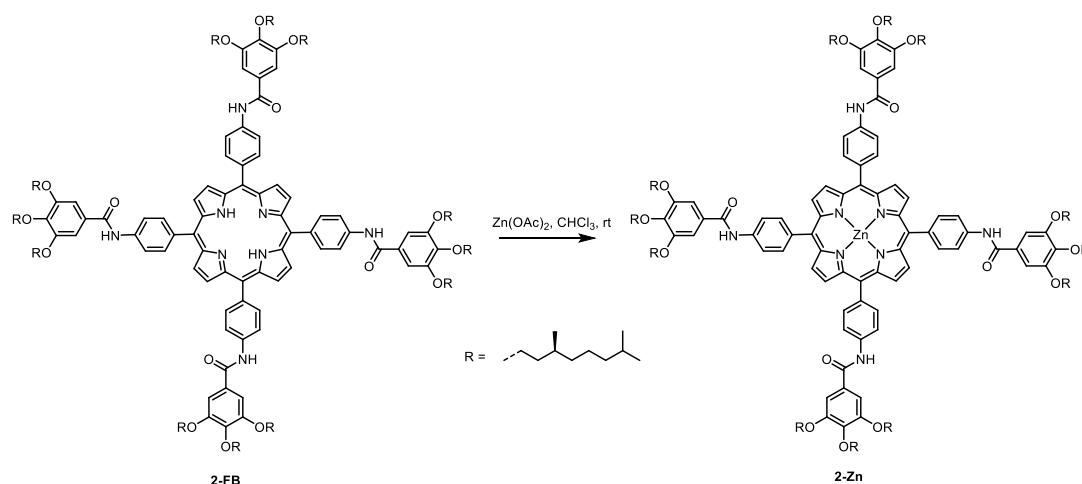


**Scheme S1:** Synthesis of *N*-centered free base porphyrin ligand **2-FB**.

Triple side chain functionalized acid **3** (650 mg, 1.1 mmol) was converted to the acid chloride by addition of oxalyl chloride (850 mg, 6.67 mmol) and refluxing for three hours. Due to the instability of the acid chloride it was immediately used for the next step without isolation. Tetra-4-aminophenyl porphyrin (150 mg, 0.22 mmol) was suspended together with the freshly prepared acid chloride in 100 mL dry chloroform with 1.5 mL triethylamine ( $\text{NEt}_3$ ) and stirred overnight with protection from light. The dark red reaction mixture was extracted with 2 x 50 mL of water and 1 x 50 mL of 0.5 M hydrochloric acid solution. The combined organic phases were dried over  $\text{MgSO}_4$ , filtered and the solvent removed in vacuo. The product was purified by column chromatography (Biotage®, gradient of EtOAc/ $\text{CHCl}_3$ /heptane in the ratio of 2:4:4). The product was precipitated from 5% chloroform in MeOH and isolated as a dark red powder (456 mg, yield: 69%).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.92 (s, 8H), 8.24 (d,  $J = 8.3$  Hz, 9H), 8.09 – 8.02 (m, 12H), 7.24 (d,  $J = 8.9$  Hz, 8H), 4.15 (m, 24H), 1.91 (m, 9H), 1.81 – 1.58 (m, 24H), 1.46 – 1.08 (m, 38H), 0.99 (m, 37H), 0.89 (d,  $J = 6.6$  Hz, 87H), -2.74 (s, 2H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  171.24, 166.19, 161.18, 153.59, 141.91, 138.45, 137.96, 135.35, 130.16, 119.73, 118.54, 106.09, 72.04, 68.05, 39.54, 39.45, 37.69, 37.54, 36.58, 30.05, 29.86, 28.17, 24.93, 24.90, 22.89, 22.80, 22.78, 19.79, 19.76. MALDI-TOF MS:  $[\text{M}^+]$  calcd. for  $\text{C}_{192}\text{H}_{290}\text{N}_8\text{O}_{16}$ : 2964.21, found 2966.27. IR ( $\text{cm}^{-1}$ ): 3316, 2953, 2925, 2869, 1677, 1648, 1582, 1517, 1492, 1468, 1426, 1383, 1365, 1330, 1234, 1205, 1111, 998, 966, 851, 800, 754, 734.

## 2.2 Synthesis of N-centered porphyrin monomer 2-Zn

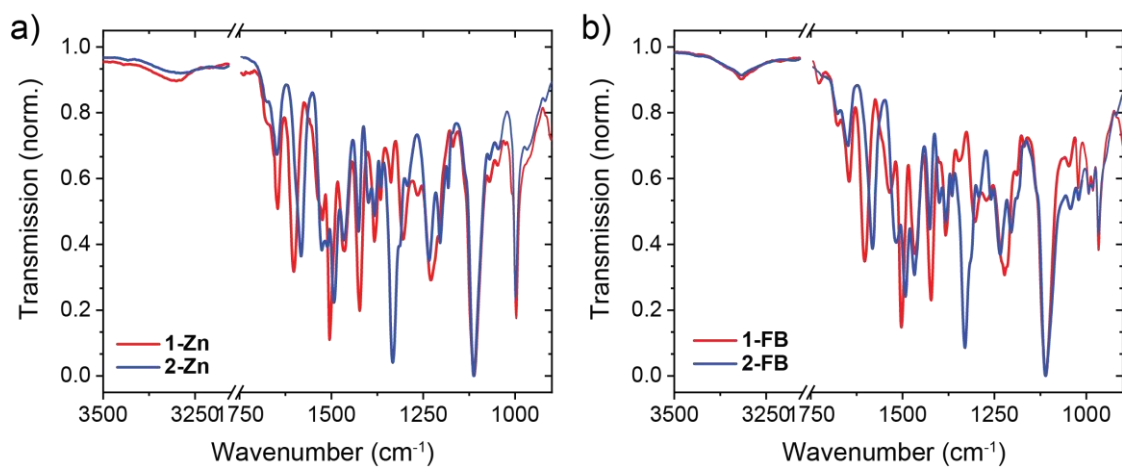


**Scheme S2:** Synthesis of **2-Zn** by zinc insertion into **2-FB**.

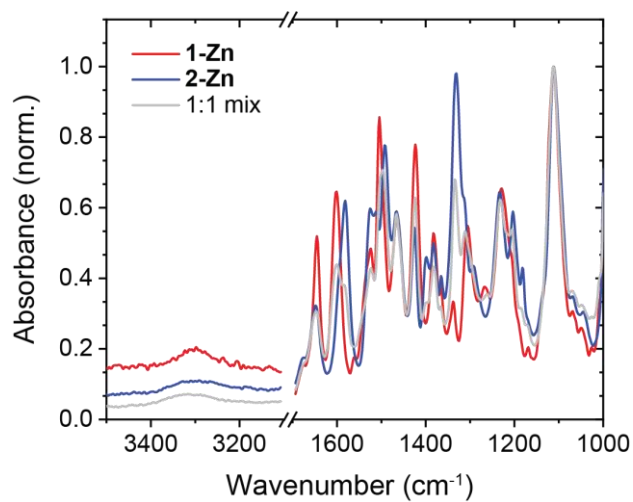
The ligand **2-FB** (250 mg, 0.85 mmol) was dissolved in 100 mL DCM and stirred together with  $\text{Zn(OAc)}_2$  (170 mg, 1.26 mmol) overnight at room temperature with protection from light. The resulting purple reaction mixture was extracted with 2 x 50 mL of water, dried over  $\text{MgSO}_4$ , filtered and the solvent removed in vacuo. After purification by column chromatography (Biotage®, EtOAc/ $\text{CHCl}_3$ /Heptane in the ratio of 2:4:4) and precipitation from 5%  $\text{CHCl}_3$  in MeOH the product was obtained as a purple, metallic shimmering powder (214 mg, yield: 84%).

$^1\text{H}$  NMR (399 MHz,  $\text{CDCl}_3$ )  $\delta$  9.01 (s, 8H), 8.29 – 8.15 (m, 12H), 8.01 (d,  $J$  = 8.3 Hz, 8H), 7.17 (d,  $J$  = 15.1 Hz, 8H), 4.16 – 4.03 (m, 31H), 2.00 – 1.83 (m, 9H), 1.73 (ddq,  $J$  = 33.1, 14.2, 7.7 Hz, 13H), 1.56 (tdd,  $J$  = 13.0, 9.5, 7.0 Hz, 16H), 1.45 – 1.09 (m, 118H), 0.99 (t,  $J$  = 7.0 Hz, 31H), 0.90 (dd,  $J$  = 6.7, 3.3 Hz, 92H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.21, 153.52, 153.49, 150.41, 141.82, 139.27, 137.69, 135.21, 132.12, 130.11, 120.56, 118.40, 106.08, 73.77, 72.00, 69.67, 67.98, 39.54, 39.49, 39.45, 37.69, 37.54, 37.25, 36.58, 32.91, 32.11, 32.08, 30.54, 30.19, 30.04, 29.93, 29.92, 29.87, 29.86, 29.83, 29.78, 29.61, 29.57, 29.53, 29.39, 28.16, 28.13, 27.24, 26.29, 24.93, 24.90, 22.87, 22.84, 22.79, 22.77, 19.88, 19.77, 19.75, 14.27, 14.21. MALDI-TOF MS:  $[\text{M}^+]$  calcd. for  $\text{C}_{192}\text{H}_{288}\text{N}_8\text{O}_{16}\text{Zn}$ : 3026.13, found 3029.11. IR ( $\text{cm}^{-1}$ ): 3299, 2958, 2925, 2868, 1648, 1582, 1526, 1493, 1467, 1426, 1383, 1365, 1333, 1234, 1204, 1182, 1113, 999, 854, 812, 797, 753, 676, 571.

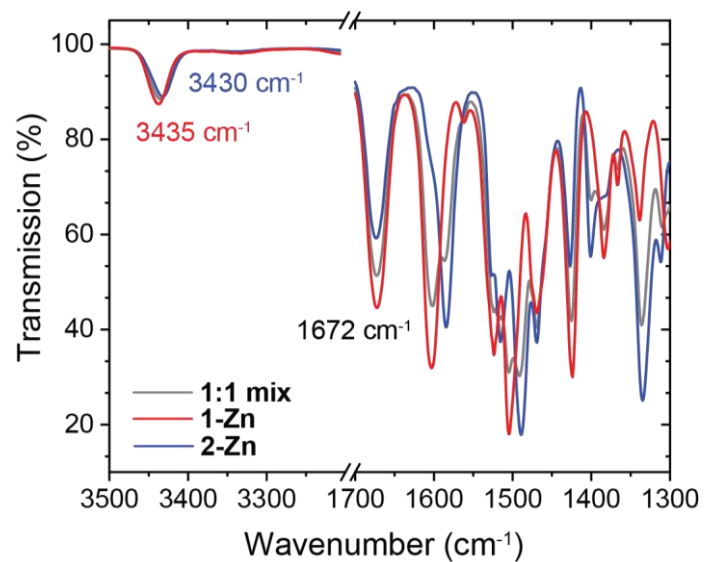
### 3. Fourier-Transform Infrared Spectroscopy



**Figure S1:** FT-IR spectra of zinc centered (a) and free base (b) ligands **1** and **2**.

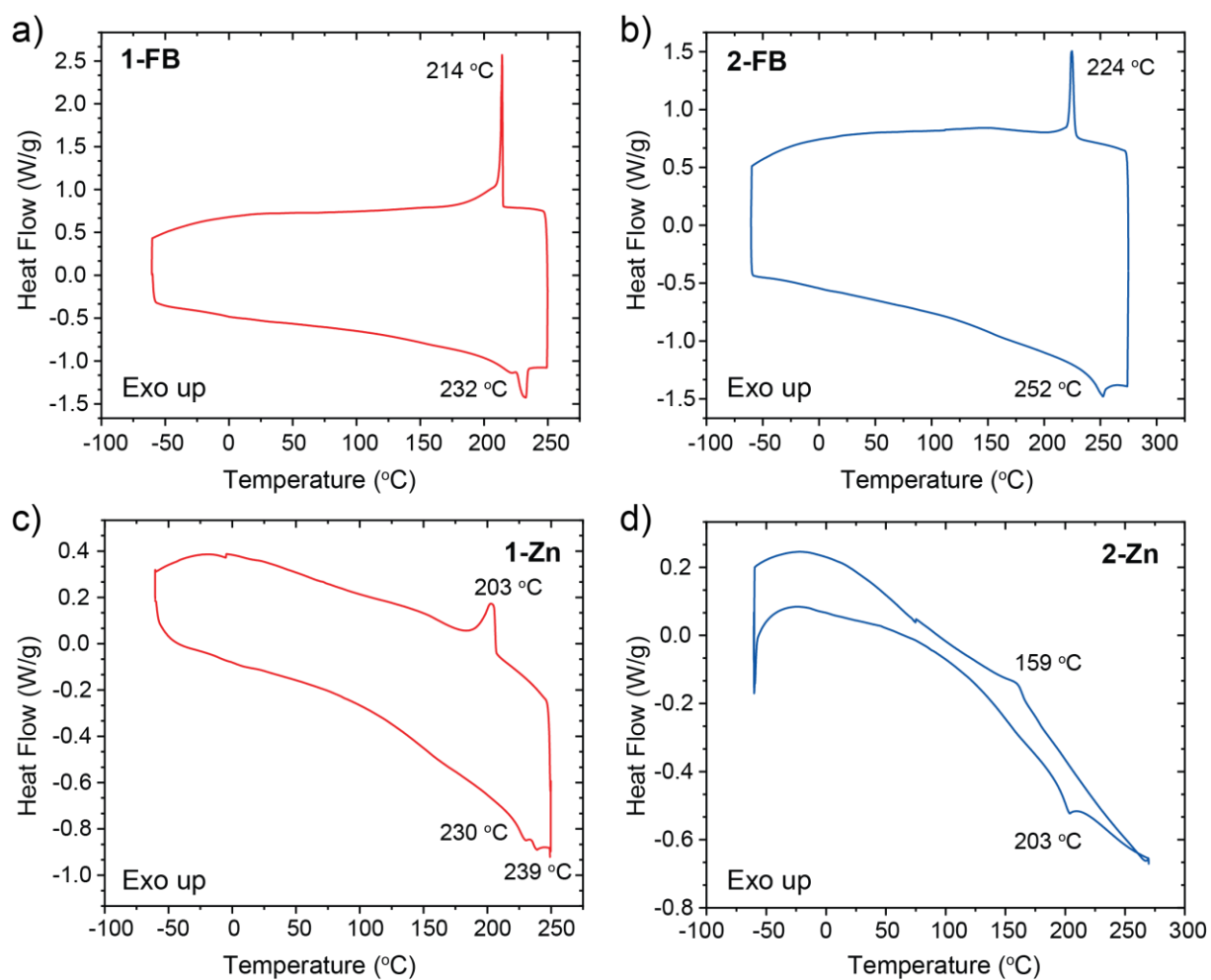


**Figure S2:** FT-IR spectra of **1**- and **2**-Zn and a 1:1 mixture. The mixture was prepared by dissolving both monomers together in a good solvent (chloroform) and letting the solvent evaporate slowly.



**Figure S3:** FT-IR spectra of 2 mM solutions of **1-** and **2-Zn** and a 1:1 mixture of both in chloroform. Both porphyrins exhibit a NH stretch of similar intensity at around 3430-3435 cm<sup>-1</sup>, indicative of non-hydrogen bonded NH groups.

#### 4. Dynamic Scanning Calorimetry (DSC)

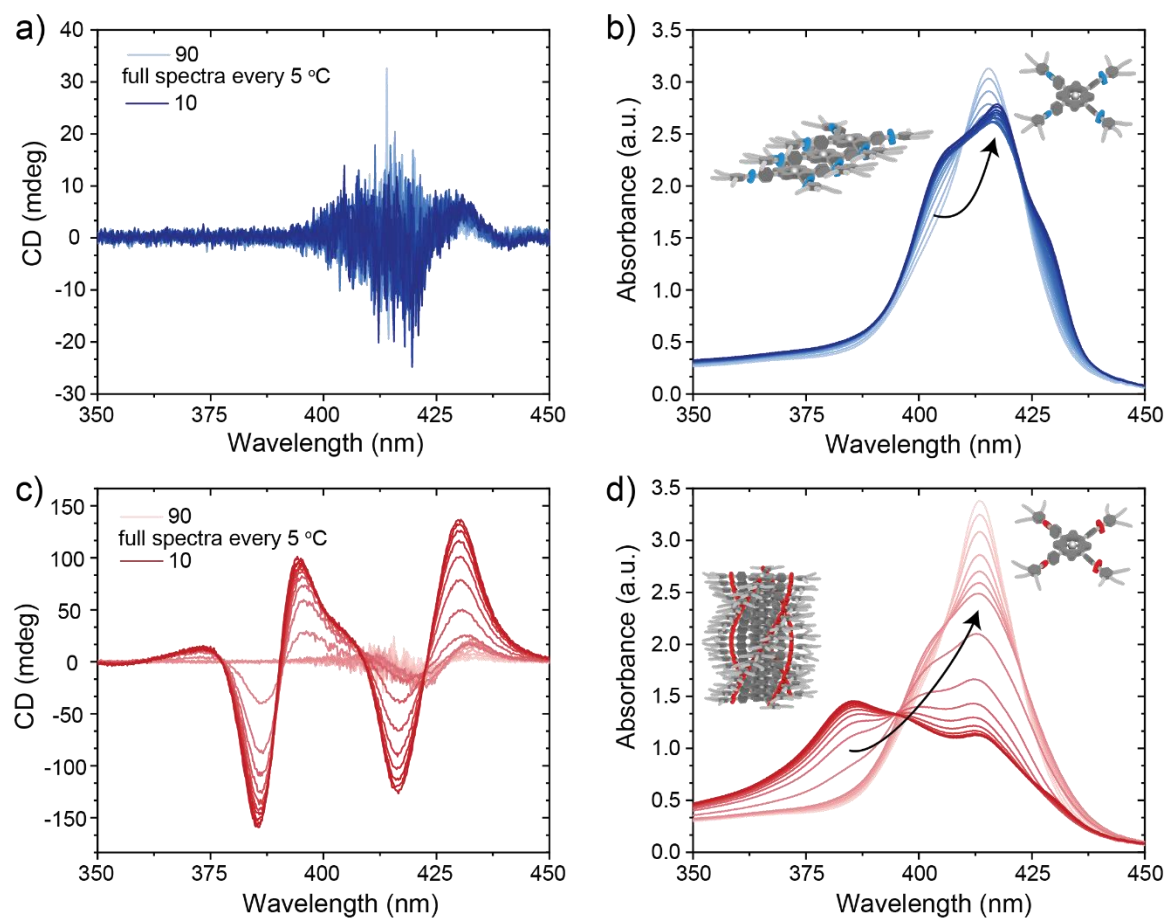


**Figure S4:** DSC graphs of free base (a, b) and zinc centered (c, d) ligands **1** and **2** with transition temperatures indicated in the plots. Data taken from the second heating/cooling run with a ramping rate of 10 °C/min.



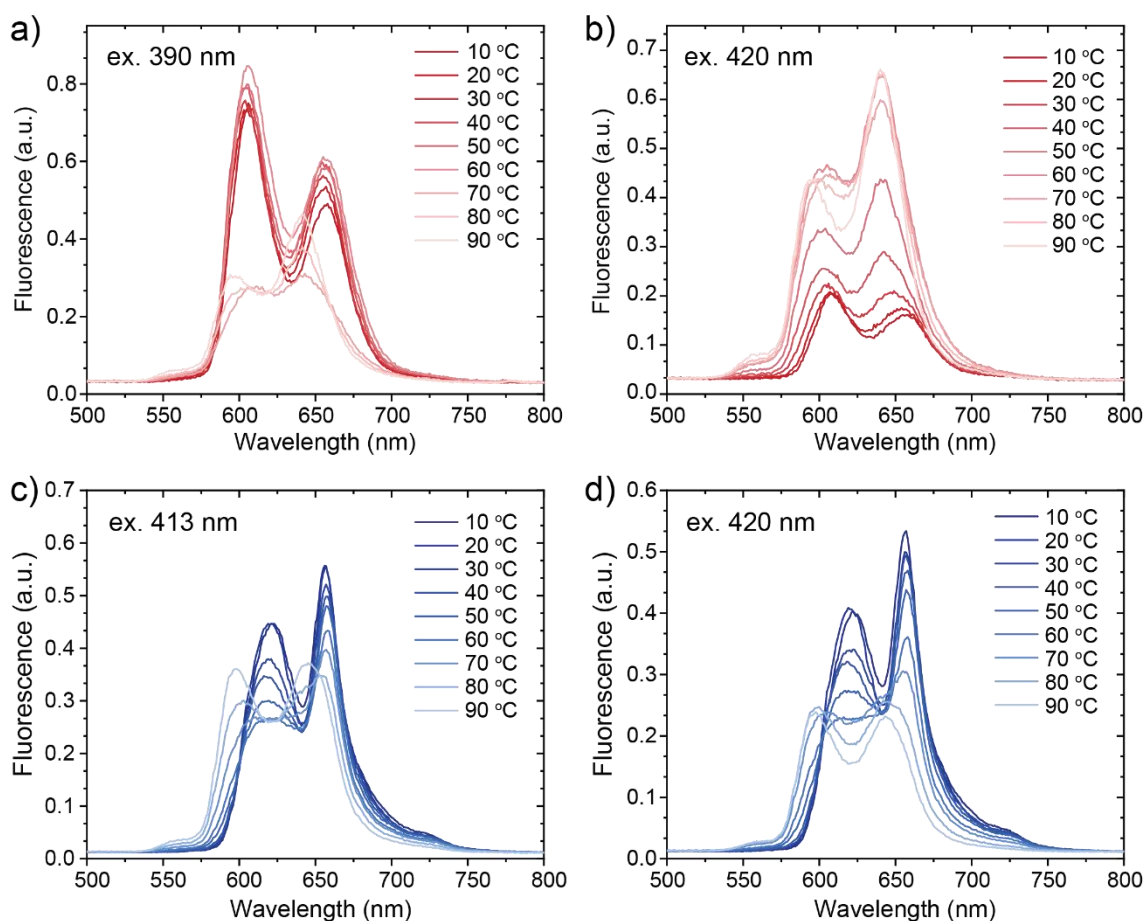
## 5. Assembly of 1-Zn and 2-Zn in apolar solvents

### 5.1 Assembly of free base ligands in apolar solvents

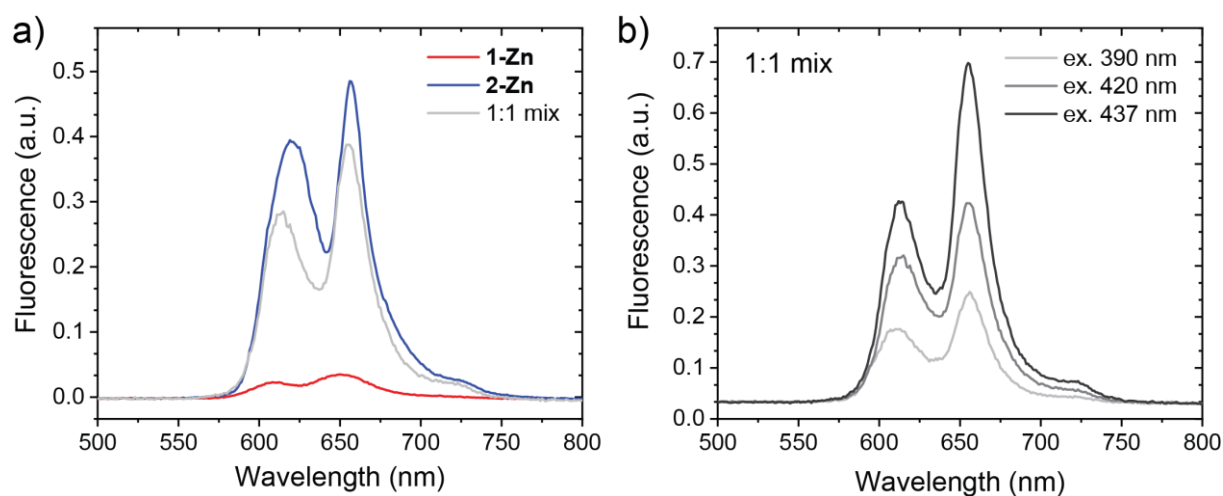


**Figure S5:** Full CD (a, c) and absorbance (b, d) spectra of free base monomers **1-FB** and **2-FB** ( $c = 50 \mu\text{M}$  in MCH) measured in intervals of  $5 \text{ }^\circ\text{C}$  from  $90\text{-}20 \text{ }^\circ\text{C}$  with a cooling rate of  $1 \text{ }^\circ\text{C}/\text{min}$ .

## 5.2 Emission spectra of 1-Zn and 2-Zn

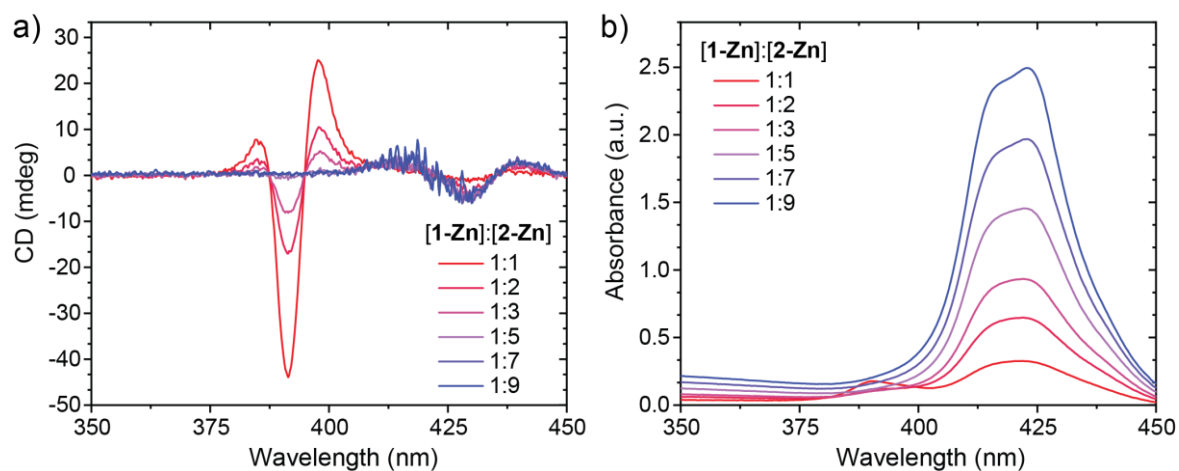


**Figure S6:** Emission spectra of **1-Zn** (a, b) and **2-Zn** (c, d) with respective excitation wavelengths noted in the plots ( $c = 10 \mu\text{M}$ ,  $l = 10 \text{ mm}$ ). For both monomers the biggest absorbance peak at 20 and 90 °C were excited as emission properties are dependent on aggregation states. Detector sensitivity was set to 800 mV for **1-Zn** and to 700 mV for the more emissive **2-Zn**.

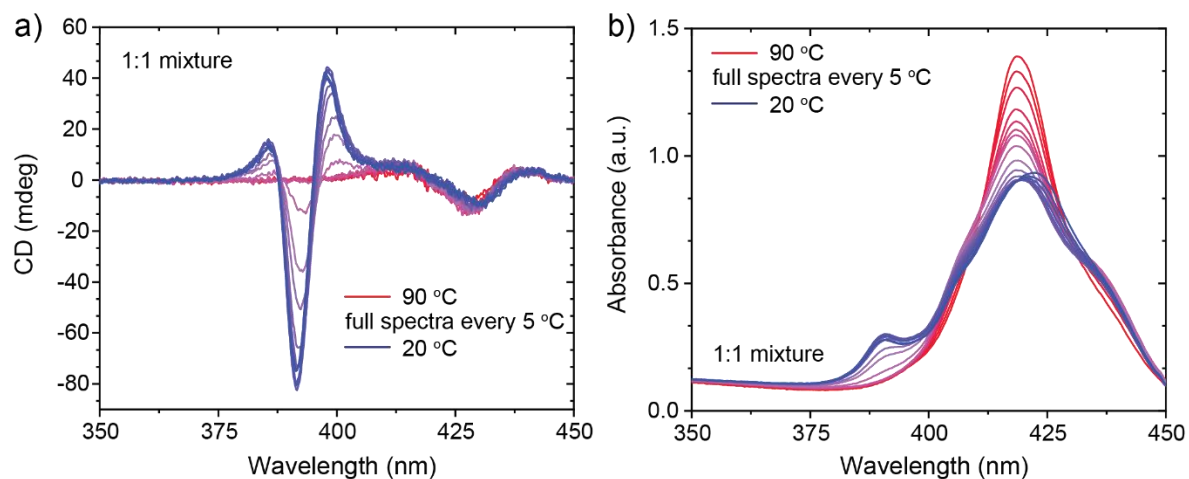


**Figure S7:** Emission spectra of a 1:1 mixture of **1-** and **2-Zn** ( $c = 10 \mu\text{M}$  of each). a) Comparison of emission of the pure polymers and a 1:1 mixture thereof (ex. 420 nm). b) Fluorescence intensity of the 1:1 mix plotted for excitation wavelength of 390 (H-aggregate), 420 (J-aggregate) and 437 nm (new co-aggregate). Detector sensitivity was set to 700 mV in all cases.

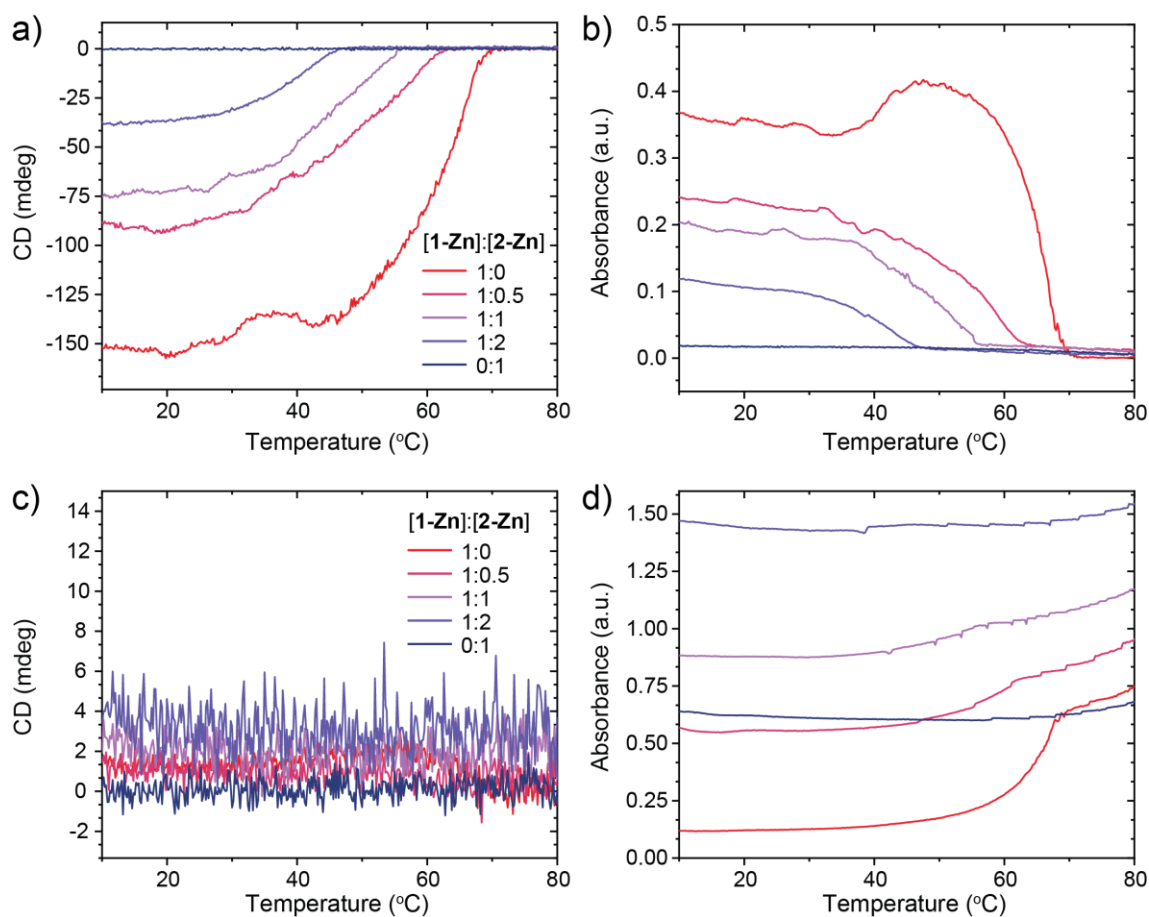
### 5.3 Copolymerization of 1-Zn and 2-Zn



**Figure S8:** Spectral changes with increasing molar ratios of 2-Zn. Concentration of 1-Zn was kept constant at  $c = 10 \mu\text{M}$  in MCH.



**Figure S9:** Full CD (a) and absorbance (b) spectra of a 1:1 mixture of 1-Zn and 2-Zn ( $c = 50 \mu\text{M}$  in MCH) measured in intervals of 5 °C from 90-20 °C with a cooling rate of 1 °C/min.

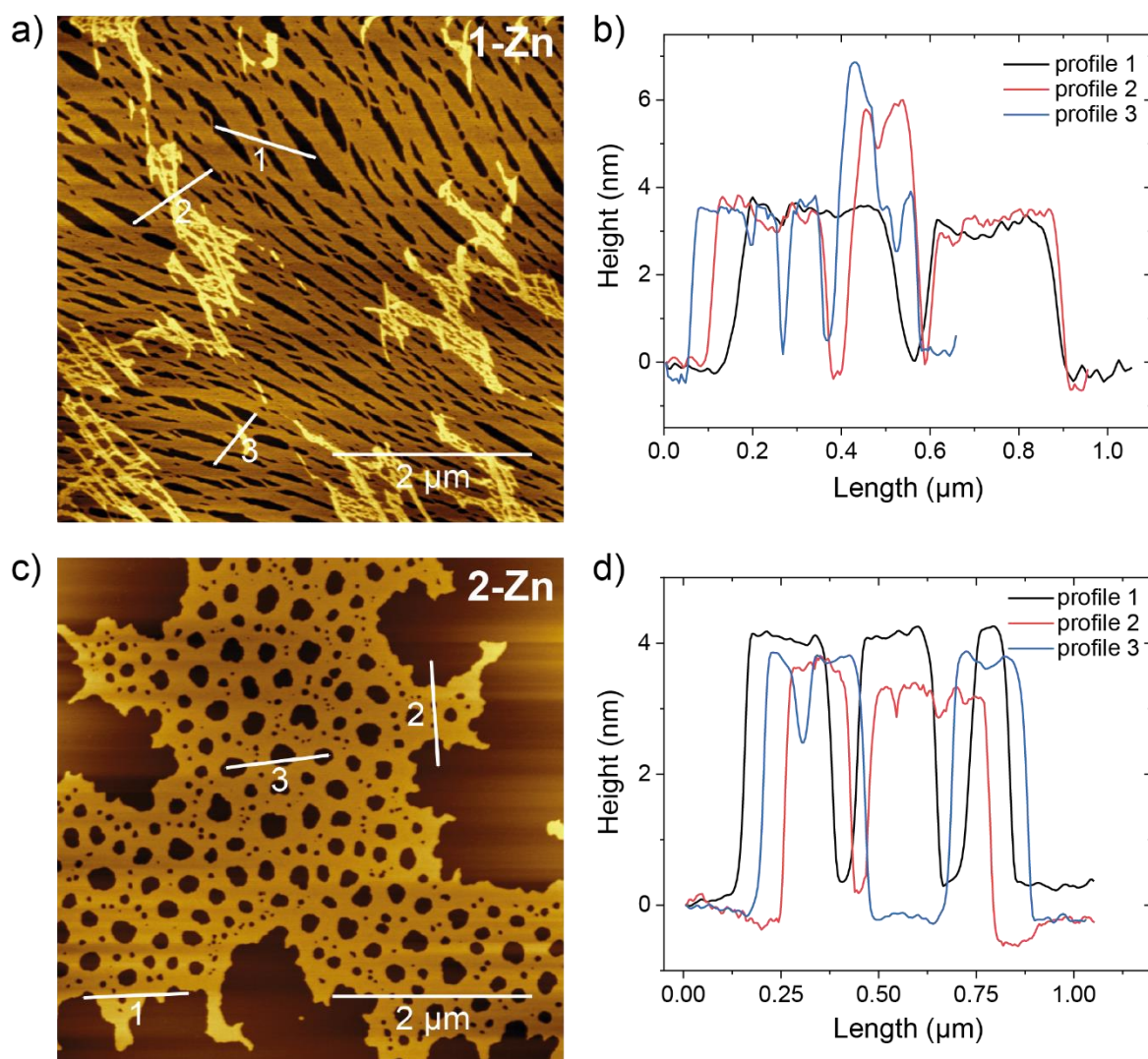


**Figure S10:** Melting curves of **1-Zn** and **2-Zn** and their mixtures followed by CD and UV at 390 nm (H-aggregate; a, b) and at 420 nm (J-aggregate plus monomer; c, d). The concentration of **1-Zn** and **2-Zn** was set to  $c = 25 \mu\text{M}$  in MCH, for mixtures in the respective ratios the concentration of **1-Zn** was kept constant at  $c_{1\text{-zn}} = 25 \mu\text{M}$  and aliquots of **2-Zn** were added to exclude dilution effects. Cooling rate was  $1 \text{ }^\circ\text{C}/\text{min}$ .

**Table S1:** Temperatures of elongation ( $T_e$ ) for homo- and copolymerization mixtures of **1-Zn** and **2-Zn**. Temperatures were taken from CD melting curves followed at 390 nm with a cooling rate of  $1 \text{ }^\circ\text{C}/\text{min}$  ( $c_{1\text{-zn}} = 25 \mu\text{M}$  for pure samples and mixtures,  $c_{2\text{-zn}} = 25 \mu\text{M}$  for pure sample).

sample	$T_e$ [ $^\circ\text{C}$ ]
<b>1-Zn</b> (1:0)	69
1:0.5	62
1:1	55
1:2	46
<b>2-Zn</b> (0:1)	-

## 6. Atomic force microscopy



**Figure S11:** AFM images of **1-Zn** and **2-Zn** dropcasted from  $c = 20 \mu\text{M}$  solutions in MCH on mica. For **1-Zn** (a) bundled supramolecular fibers were found while for **2-Zn** (c) only disordered deposits were observed. Height profiles of b) **1-Zn** and d) **2-Zn** shows structures of 3-4 nm thickness in both cases.

## 7. Density functional theory calculations of monomers

All DFT calculations were performed using the Vienna Ab Initio Simulation Package (VASP).<sup>3–6</sup> The Perdew–Burke–Ernzerhof (PBE) exchange-correlation functional<sup>7,8</sup> was used in conjunction with the projector augmented wave approach.<sup>9,10</sup> All structures were optimized to their local minima using the conjugate gradient algorithm as implemented in VASP. Optimization and other electronic settings are given below.

### Settings

Parameter	INCAR setting	value
Cutoff energy	ENCUT	500
Precision	PREC	High
Smearing type	ISMEAR	0 (Gaussian smearing)
Smearing width	SIGMA	0.0005
Electronic convergence threshold	EDIFF	1E-5
Ionic relaxation threshold	EDIFFG	1E-4

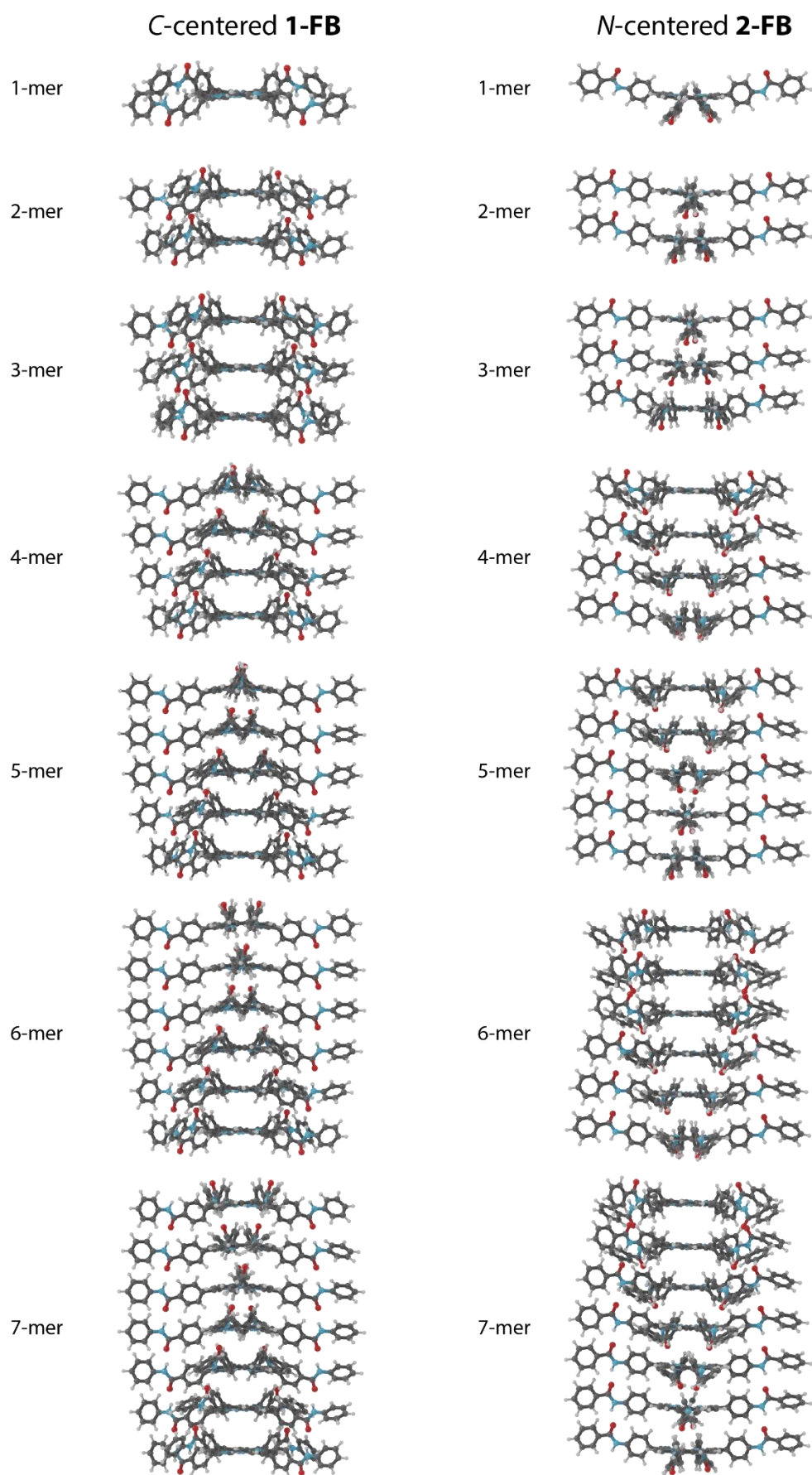
### K-point grid

For all calculations, only the  $\Gamma$ -point was used.

### Geometries

Below, the geometries of the optimized structures are given. These geometries are represented as POSCAR/CONTCAR files. Further information with regard to the structure of these files is provided in the link below: <http://cms.mpi.univie.ac.at/vasp/guide/node59.html>.





**Figure S12:** Images of optimized structures of mono- up to heptamers of C- and N-centered ligands.

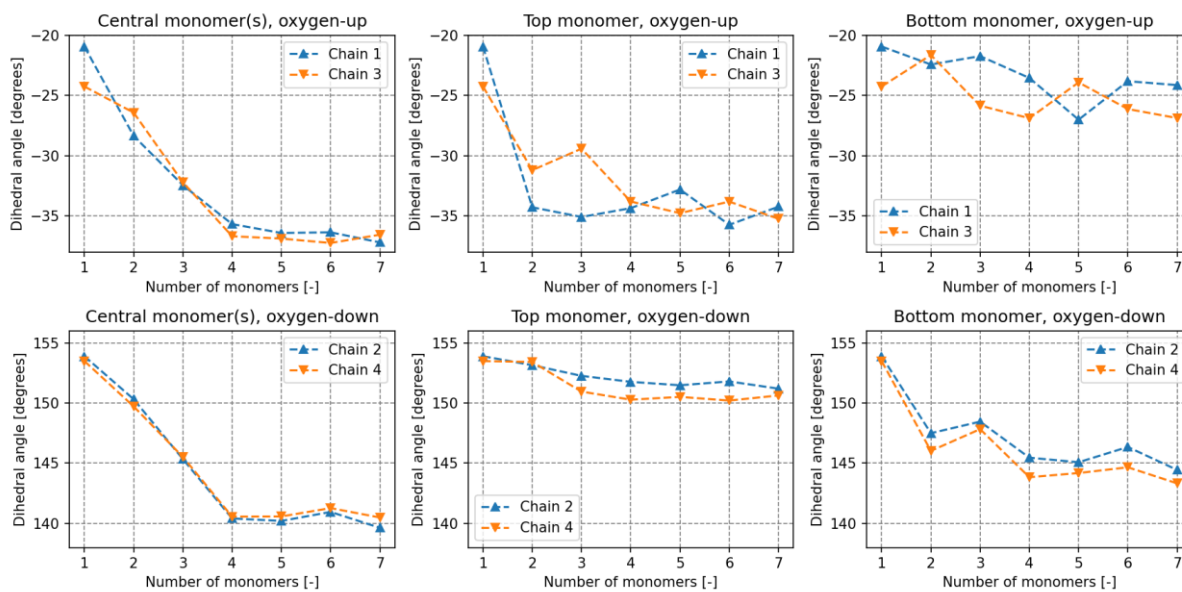


Figure S13: Dihedral angles of **1-FB** monomers in the stacks oriented either oxygen up or down.

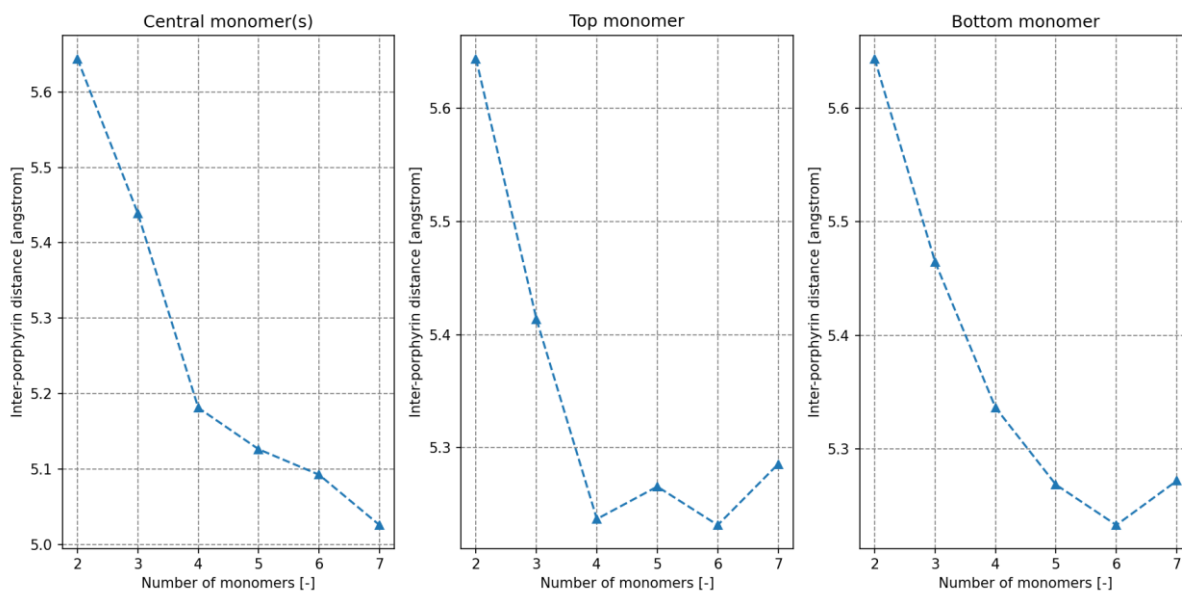


Figure S14: Distance between central, top or bottom monomers in the **1-FB** stack.



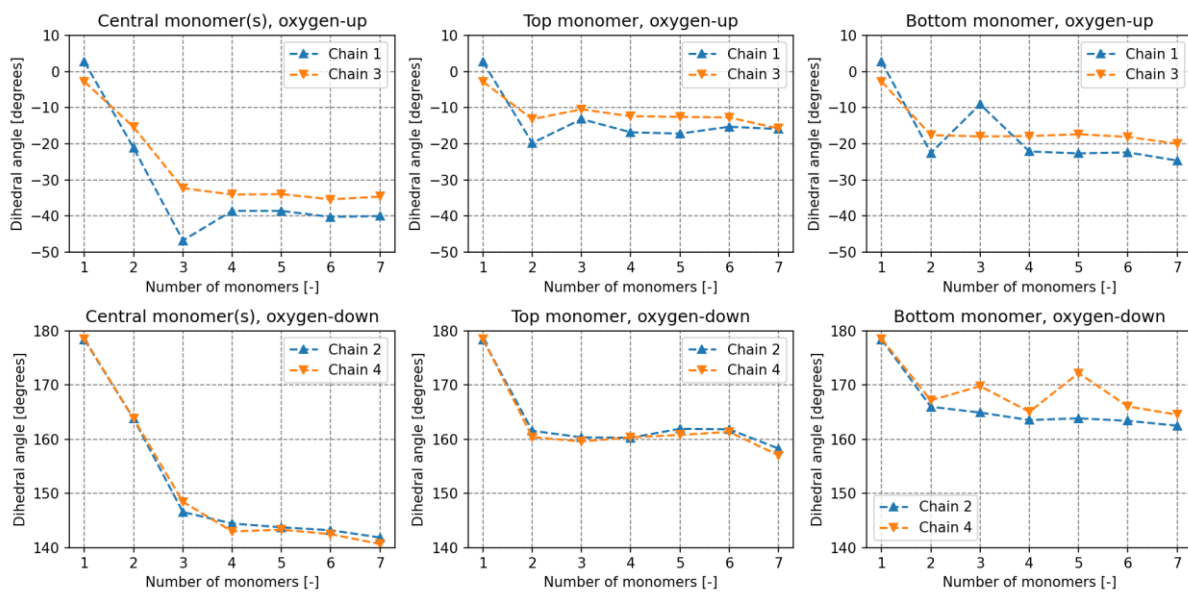


Figure S15: Dihedral angles of 2-FB monomers in the stacks oriented either oxygen up or down.

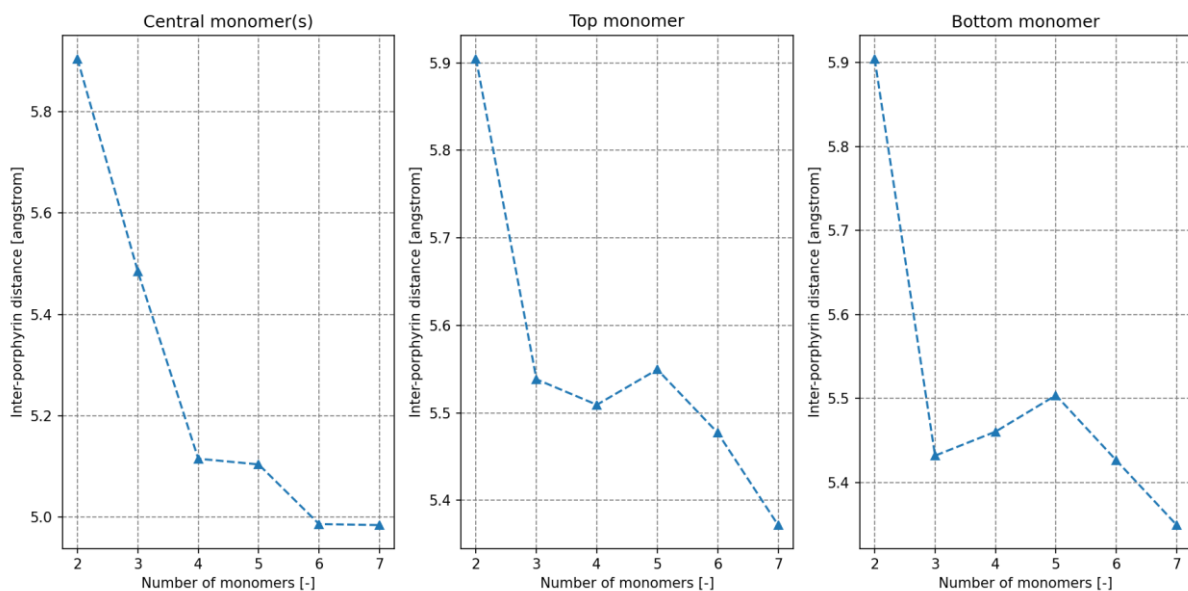


Figure S16: Distance between central, top or bottom monomers in the 2-FB stack.

## 8. Spectra of Compounds:

### 8.1 $^1\text{H}$ NMR spectra:

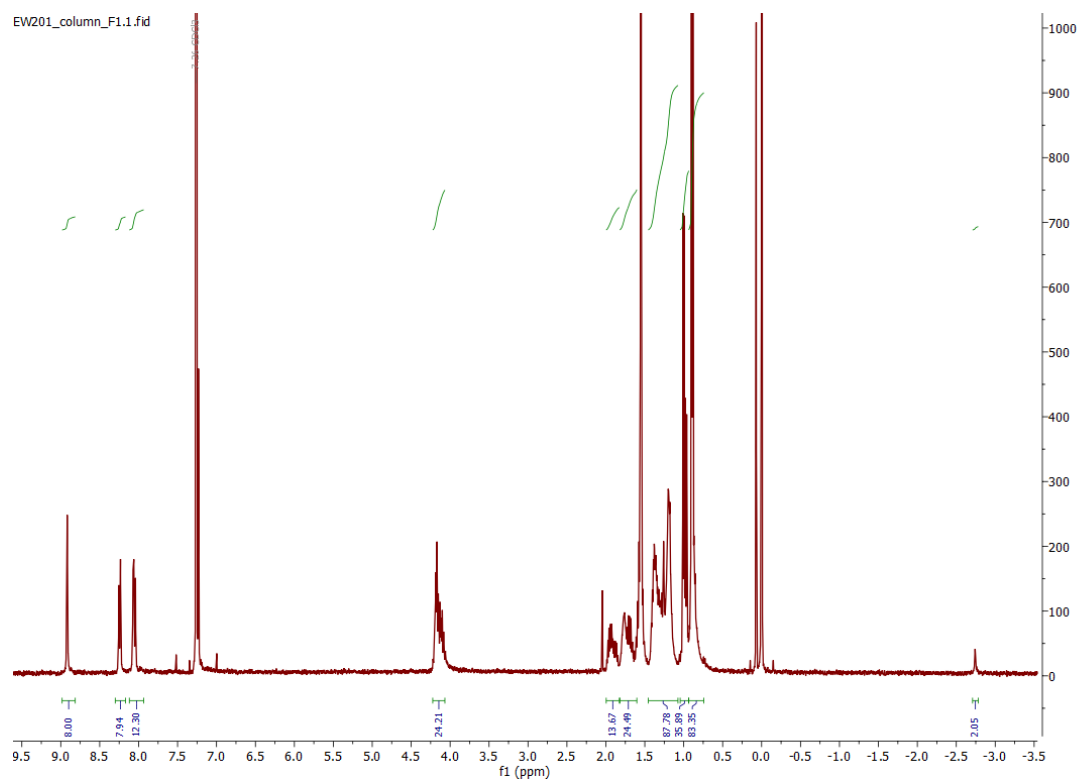


Figure S17:  $^1\text{H}$  NMR spectrum of 2-FB in chloroform- $d$ .

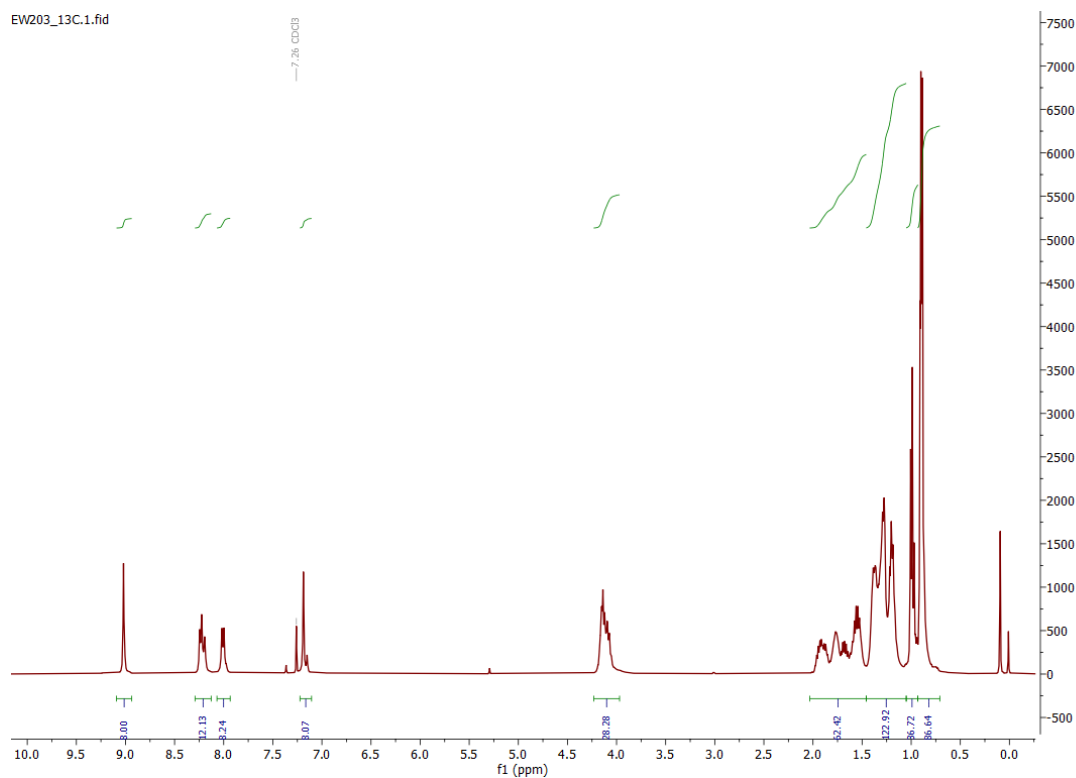


Figure S18:  $^1\text{H}$  NMR spectrum of 2-Zn in chloroform- $d$ .

## 8.2 $^{13}\text{C}$ and 2D NMR spectra:

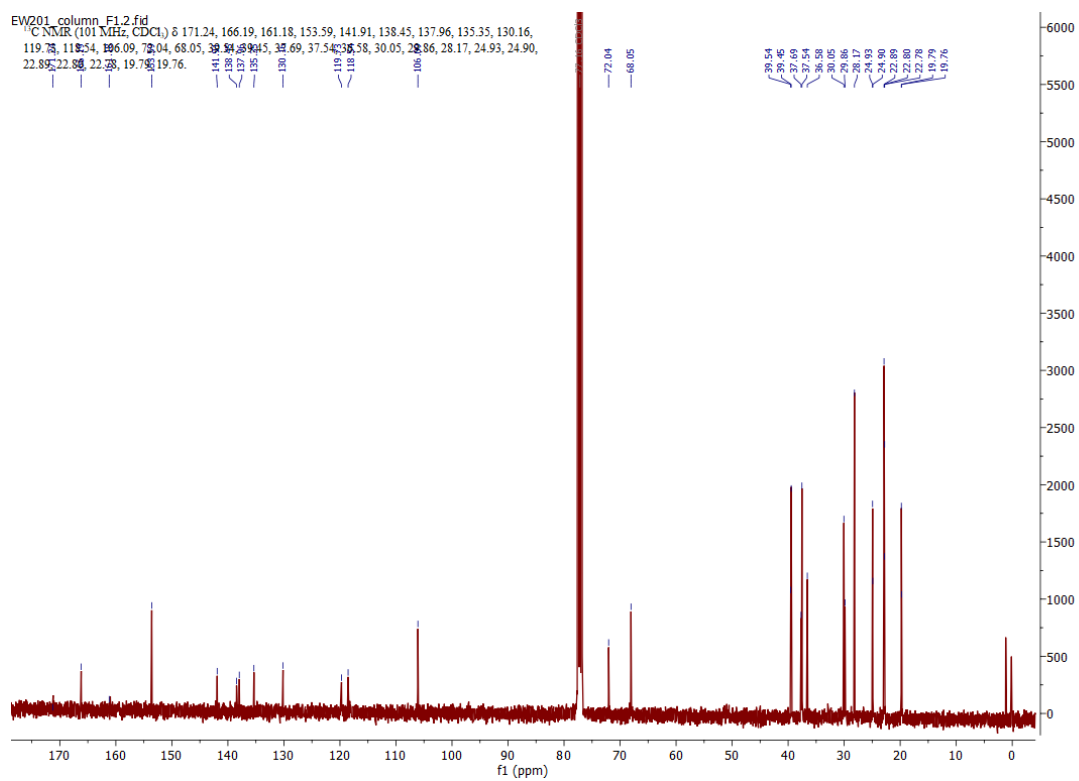


Figure S19:  $^{13}\text{C}$  NMR spectrum of **2-FB** in chloroform-*d*.

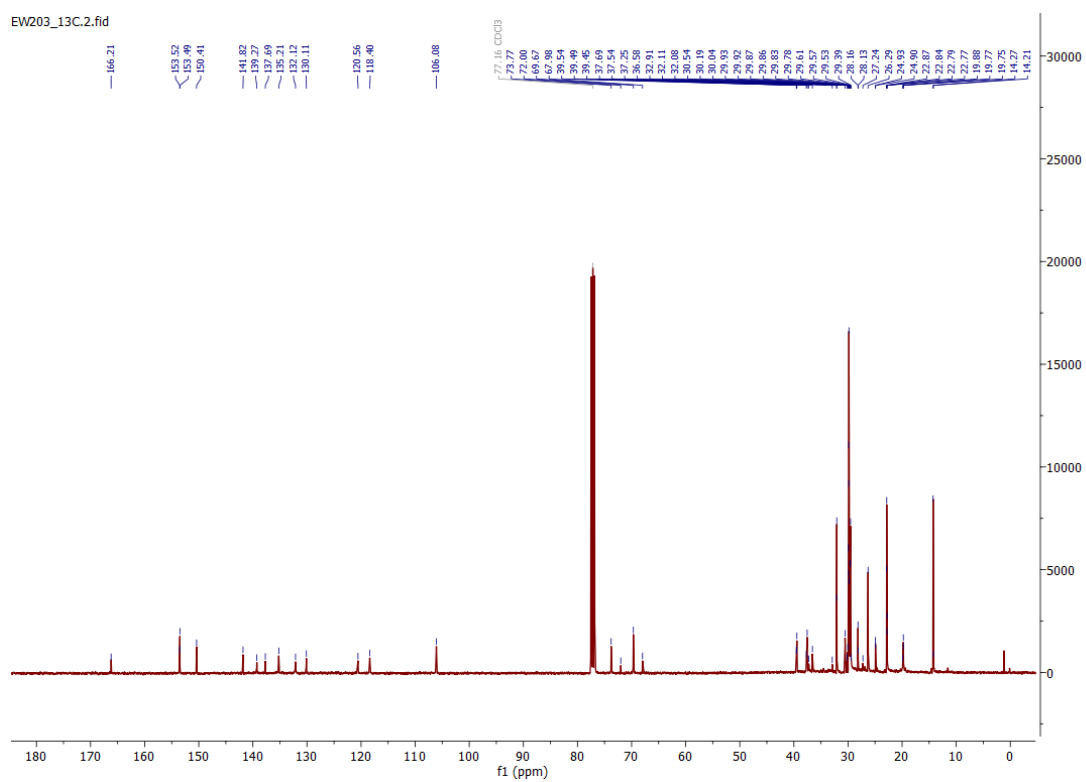


Figure S20:  $^{13}\text{C}$  NMR spectrum of **2-Zn** in chloroform-*d*.

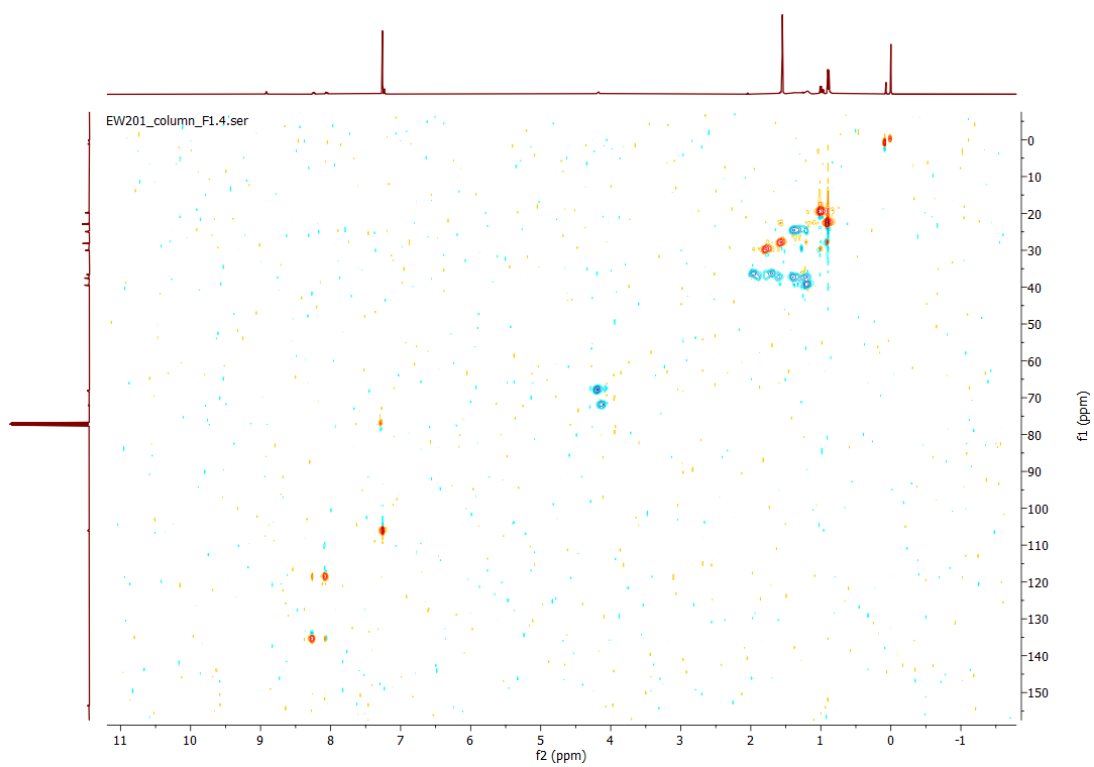


Figure S21: COSY NMR of 2-FB in chloroform-*d*.

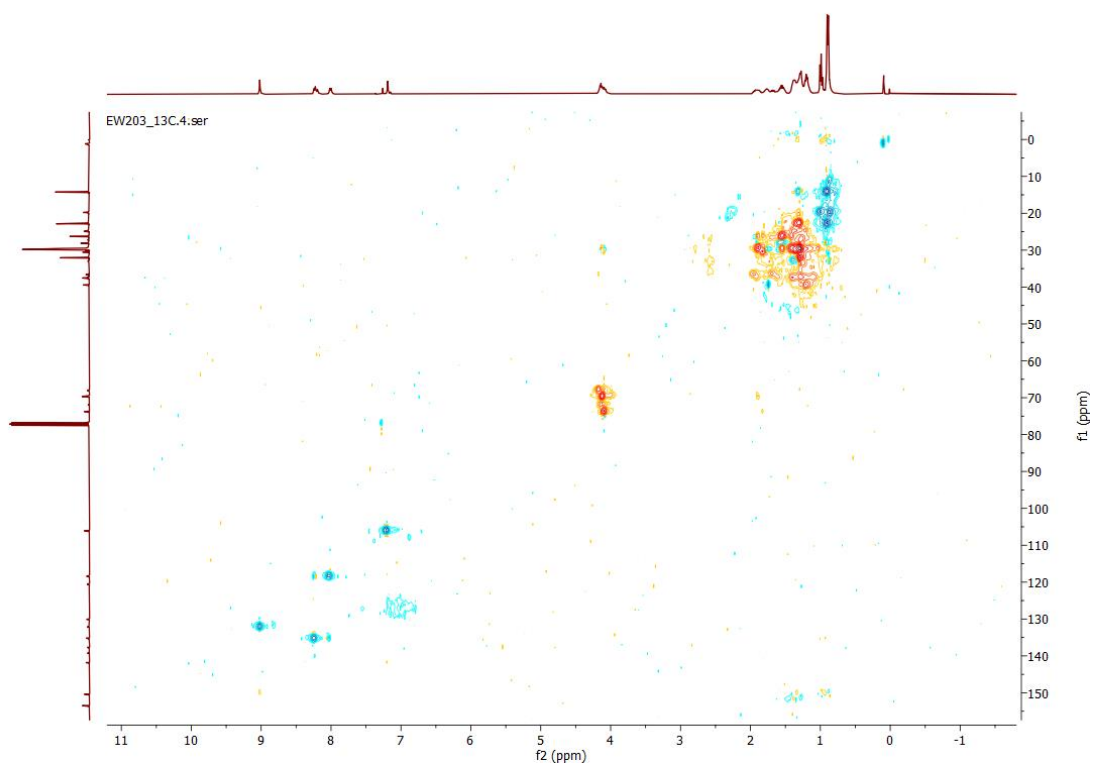


Figure S22: COSY NMR of 2-Zn in chloroform-*d*.

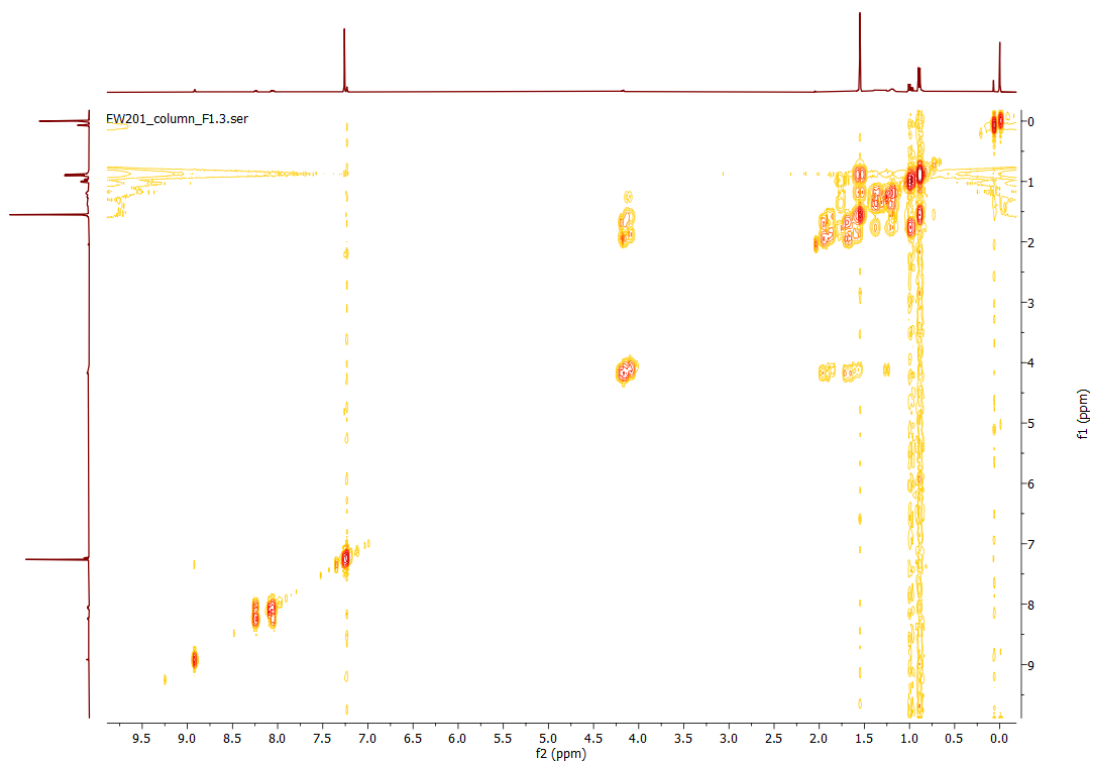


Figure S23: HSQC NMR of 2-FB in chloroform-*d*.

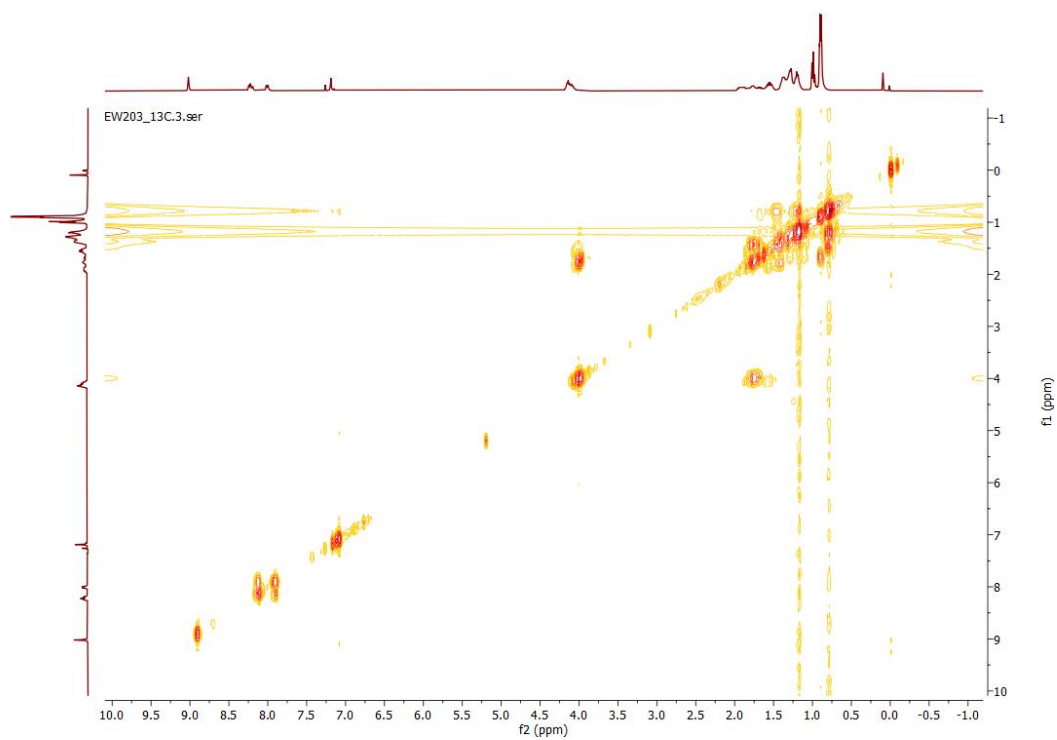


Figure S24: HSQC NMR of 2-Zn in chloroform-*d*.

### 8.3 MALDI-TOF:

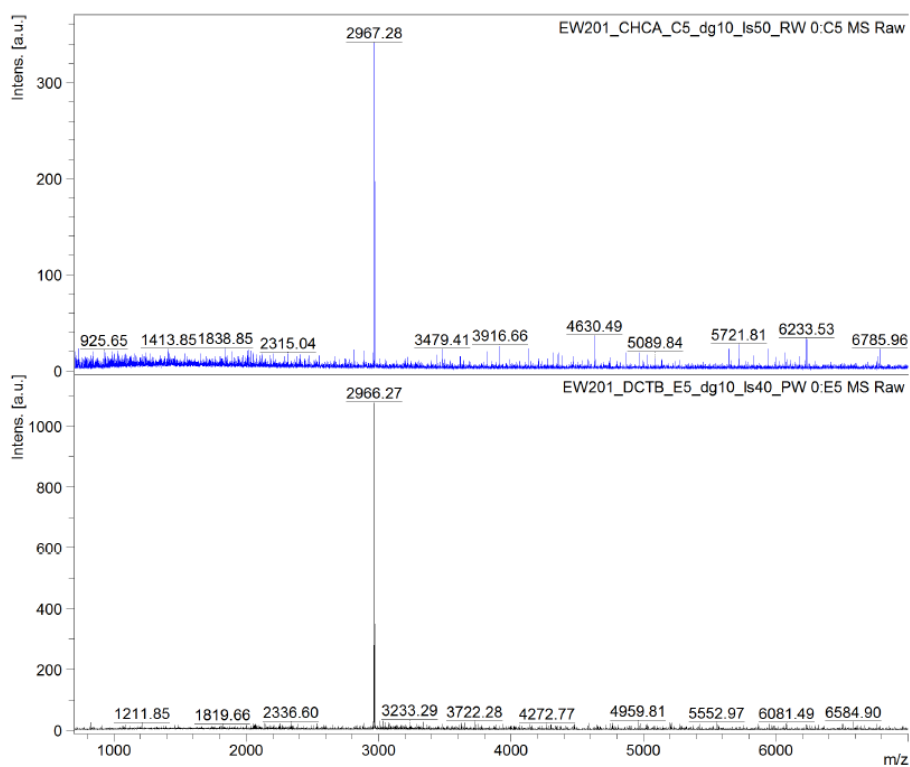


Figure S25: MALDI-TOF spectrogram of 2-FB.

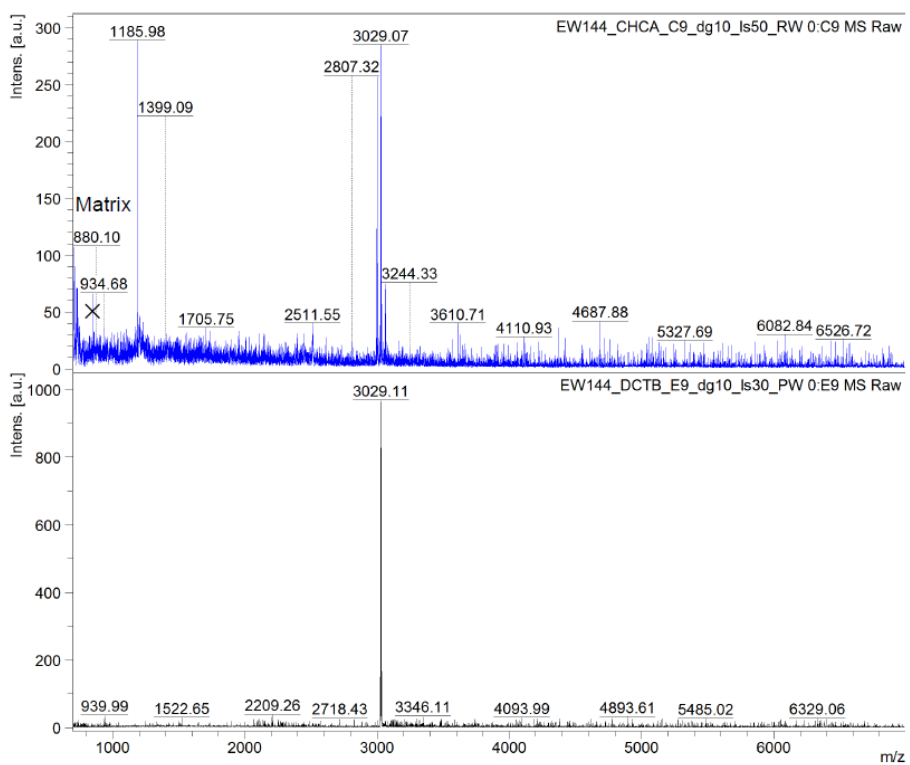


Figure S26: MALDI-TOF spectrogram of 2-Zn.

## 9. References

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- (10) Kresse, G.; Joubert, D. *Phys. Rev. B* **1999**, *59*, 1758.

## 10. Appendix: DFT coordinates

C-CENTERED MONOMER

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