# Electronic Supporting Information (ESI)

## Void and filled supramolecular nanoprisms - Notable differences between seemingly identical construction principles

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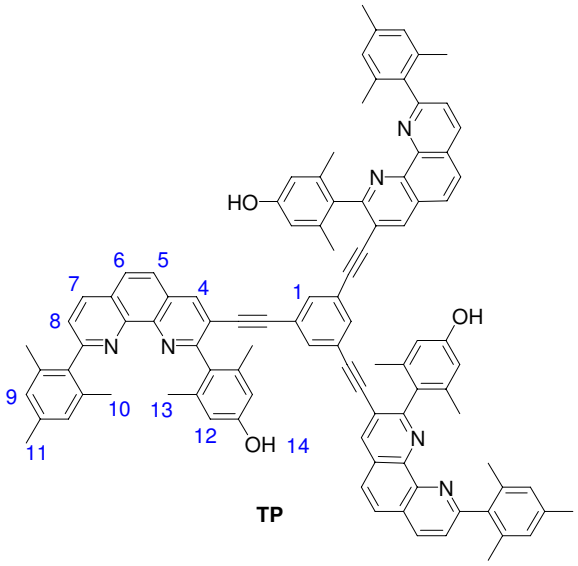
# 1. Experimental Section

### *1.1 General*

All reagents were commercially available and used without further purification. Purification and drying of the used solvents was performed according to standard methods. Thin-layer chromatography was performed using thin-layer chromatography plates (Silica Gel 60 $F_{254}$, Merck). Silica Gel 60 was used for column chromatography. Confirmation of the structures of all products was obtained by $^{1}$H-NMR and $^{13}$C-NMR spectroscopy (Bruker AC200 and Avance AC 400 spectrometer, using the deuterated solvent as the lock and residual solvent as the internal reference). The numbering of the carbon atoms of the molecular formulae shown in the experimental section is used only for the NMR assignments and is not in accordance with the IUPAC nomenclature rules. Melting points were taken using a melting point apparatus of Dr. Tottoli (Büchi) and are uncorrected. Electrospray mass spectra (ESI-MS) were recorded using a ThermoQuest LCQ Deca. The purity of all compounds was checked by thin-layer chromatography on $SiO_2$ (silica gel 60 F254, Merck). Infrared spectra were recorded on a Perkin Elmer 1750 FT-IR spectrometer with the software of IRDM 1700. UV/Vis spectra were recorded on a J&M Tidas II/Rev. 1 UV/visible Spectrometer. The degree of lithiation can readily be monitored by GC, while the progress of substitution at phenanthroline is best controlled by ESI-MS.

## *1.2 Synthesis*

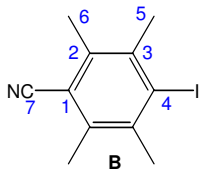
### 1.2.1 Synthesis of trisphenanthroline **TP**




**TP**

3-Ethynyl-2-(4-hydroxy-2,6-dimethylphenyl)-9-mesityl-[1,10]-phenanthroline [1] (500 mg, 1.13 mmol), 1,3,5-triiodobenzene (171 mg, 376 µmol) and $Pd[(PPh_3)_4]$ (69.0 mg, 60.0 µmol) were dissolved in dry DMF (30 mL) and dry triethylamine (30 mL). After deoxygenating the reaction mixture with nitrogen gas for 15 min., it was heated to 60 °C for 4 hours. Then water (200 mL) was slowly added and the resulting precipitate was collected by filtration. After the collected solid had been washed with 100 mL of water, 30 mL of acetone and 100 mL of dichloromethane, the crude product was purified by recrystallisation from DMF to obtain product as yellow solid (480 mg, 343 µmol, 91%).
MP: >300 °C; IR (KBr): $\tilde{\nu}$ = 3432 (w), 3413 (w), 2918 (w), 2358 (w), 1656 (s), 1615 (s), 1588 (s), 1542 (w), 1508 (w), 1459 (s), 1390 (m), 1317 (s), 1158 (s), 1030 (w), 889 (m), 850 (s), 776 (w), 639 (w); $^1$H-NMR (DMF-$d_7$, 400 MHz): δ = 2.03 (s, 18H, 10-H), 2.04 (s, 18H, 13-H), 2.31 (s, 9H, 11-H), 6.74 (s, 6H, 12-H), 6.98 (s, 6H, 9-H), 7.09 (s, 3H, 1-H), 7.77 (d, *J* = 8.1 Hz, 3H, 8-H), 8.14 (d, *J* = 8.9 Hz, 3H, 6-H), 8.19 (d, *J* = 8.9 Hz, 3H, 5-H), 8.65 (d, *J* = 8.1 Hz, 3H, 7-H), 8.90 (s, 3H, 4-H), 9.57 (s, 3H, 14-H); $^{13}$C-NMR (DMF-$d_7$, 100 MHz): δ = 20.2, 20.4, 21.0, 89.6, 92.7, 114.7, 119.7, 124.5, 125.8, 126.6, 127.6, 128.1, 128.7, 128.9, 132.0, 134.4, 136.1, 137.3, 137.8, 137.9, 139.0, 140.3, 145.7, 146.4, 158.2, 160.9, 162.1; ESI-MS Calcd for $[C_{99}H_{78}N_6O_3+H]^+$: m/z = 1399.6, Found: m/z = 1400.0, ESI-MS Calcd for $[C_{99}H_{78}N_6O_3+2H]^{2+}$: *m/z* = 700.3, Found: *m/z* = 700.5, ESI-MS Calcd for $[C_{99}H_{78}N_6O_3+3H]^{3+}$: *m/z* = 467.2, Found: *m/z* = 467.4; Anal Calcd for $C_{99}H_{78}N_6O_3$•3DMF: C, 80.12; H, 6.16; N, 7.79; Found: C, 79.78; H, 6.07; N, 7.64.

1.2.2 Synthesis of 1-cyano-4-iododurene (**B**)

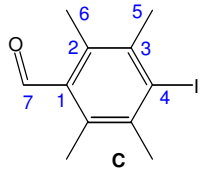



**B**

To a solution of 4.48 g (50.0 mmol) of cuprous cyanide in 100 mL of DMF was added 19.3 g (50.0 mmol) of 1,4-diiododurene and the resultant reaction mixture was heated at reflux (150-160 °C) for 12 h. Subsequently, the reaction mixture was cooled to room temperature and a solution of $FeCl_3$ (2.00 g in 1 mL of concentrated HCl and 4 mL of water) was added. The mixture was heated at 70-80 °C for 20 min and then extracted with $CH_2Cl_2$. The combined extracts were washed with saturated NaCl solution, dried over $MgSO_4$ and the solvents removed in vacuum. The residue was subjected to column chromatography (10% EtOAc/hexane, $R_f = 0.6$) to obtain 11.3 g (39.6 mmol, 79%) of 1-cyano-4-iododurene as a white solid

MP: 179-180 °C; IR (KBr): $\tilde{\nu}$ = 2949 (w), 2216 (s), 1545 (m), 1443 (m), 1386 (m), 1258 (m), 1001 (w), 908 (m); $^1$H-NMR ($CDCl_3$, 400 MHz): $\delta$ = 2.51 (s, 6H, 5-H), 2.56 (s, 6H, 6-H); $^{13}$C-NMR ($CDCl_3$, 100 MHz): $\delta$ = 20.6, 27.6, 114.6, 117.7, 118.2, 137.4, 139.1.

1.2.3 Synthesis of 4-iodo-2,3,5,6-tetramethylbenzaldehyde (**C**)



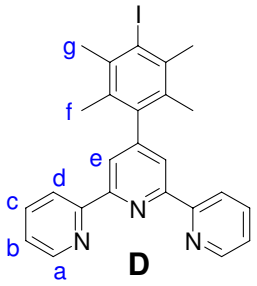

**C**

1-Cyano-4-iododurene (5.70 g, 20.0 mmol) was dissolved in 50 mL of dichloromethane at 0 °C under $N_2$ in a 250 mL round bottom flask, then 22.0 mL of DIBAL (22.0 mmol, 1 M solution in toluene) was added slowly. The reaction mixture was allowed to attain room temperature and stirred overnight. The reaction mixture was quenched with diluted HCl, and the contents were heated to reflux for 30 min. Then the reaction mixture was extracted with $CH_2Cl_2$ (2x100 mL), and the combined organic layer was dried over $MgSO_4$. The crude product was purified by column chromatography ($SiO_2$, hexane:ethyl

acetate, 100 : 10, $R_f$ = 0.5) to obtain product as a colourless powder in 4.49 g (15.6 mmol, 71%).

MP: 165-167 °C; IR (KBr): $\tilde{\nu}$ = 2923 (w), 2858 (w), 2742 (w), 1691 (s), 1536 (m), 1442 (w), 1382 (m), 1276 (w), 1063 (w), 918 (m), 672 (w); $^1$H-NMR (400 MHz, $CDCl_3$): δ = 2.44 (s, 6H, 5-H), 2.54 (d, $J$ = 4.7 Hz, 6H, 6-H), 10.6 (d, $J$ = 4.7 Hz, 1H, 7-H); $^{13}$C-NMR (100 MHz, $CDCl_3$): δ = 17.6, 27.6, 117.3, 134.3, 135.5, 139.0, 196.6; Anal Calcd for $C_{11}H_{13}IO\bullet0.25H_2O$: C, 45.15; H, 4.65; Found: C, 45.19; H, 4.27.

1.2.4 Synthesis of 4-(4-iododuryl)-2,2',6',2''-terpyridine (**D**)[2]



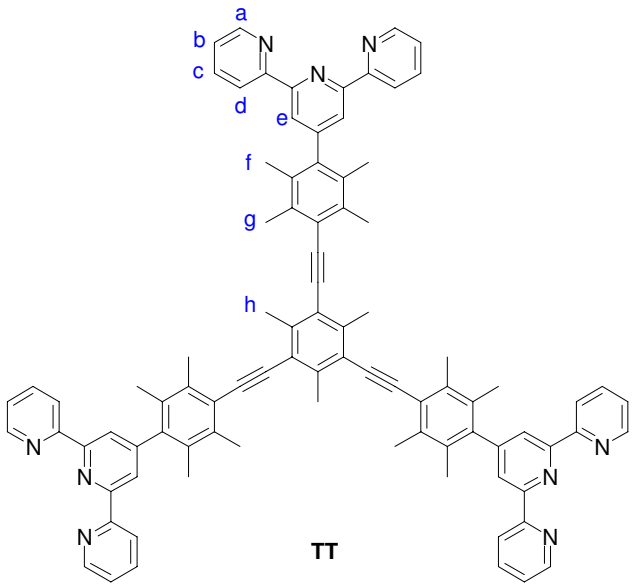

**D**

Compound **C** (4.00 g, 13.9 mmol) was dissolved in a solution of NaOH (10.0 g, 250 mmol) in MeOH (500 mL). After stirring for 5 min at room temperature, 2-acetyl-pyridine (6.06 g, 5.61 ml, 50.0 mmol) was added and the mixture was stirred for 1 h. The volume of reaction mixture was reduced to about 150 mL, then water (200 mL) was added and the mixture was extracted with $CH_2Cl_2$ (3×100 mL). The organic layers were dried over $MgSO_4$ and concentrated in vacuum. The solid residue and ammonium acetate (30.0 g, 390 mmol) were dissolved in EtOH (200 ml) and heated to reflux overnight. The solution was then cooled, reduced in volume, and water (100 mL) was added. The mixture was extracted with $CH_2Cl_2$ (3×100 ml), and the organic layers were dried over $MgSO_4$ and concentrated in vacuum. Purification by column chromatography ($Al_2O_3$, $CH_2Cl_2$, $R_f$ = 0.9) gave 2.90 g of 4-(4-iododuryl)-2,2',6',2''-terpyridine (5.90 mmol, 42%) as a yellow solid.

MP: 240-242 °C; IR (KBr): $\tilde{\nu}$ = 3048 (w), 2923 (w), 2920 (w), 2360 (w), 1605 (w), 1584 (s), 1566 (m), 1541 (m), 1465 (s), 1391 (s), 1264 (w), 1214 (w), 1116 (w), 989 (w), 902 (m), 788 (s), 731 (s), 659 (m), 622 (w); $^1$H-NMR ($CDCl_3$, 400 MHz): δ = 2.08 (s, 6H, f-H), 2.56 (s, 6H, g-H), 7.31-7.34 (m, 2H, b-H), 7.87 (td, $J$ = 7.8 and 1.6 Hz, 2H, c-H), 8.29 (s, 2H, e-H), 8.66-8.68 (m, 4H, a-, d-H); $^{13}$C-NMR ($CDCl_3$, 100 MHz): δ = 20.1, 27.8, 111.6, 121.4, 121.9, 124.0, 131.9, 137.0, 137.7, 140.2, 149.3, 152.6, 155.8, 156.2; ESI-

MS Calcd for $[C_{25}H_{22}IN_3+H]^+$: *m/z* 492.1, Found: *m/z* 492.0; Anal Calcd for $C_{25}H_{22}IN_3 \bullet 0.5H_2O$: C, 60.01; H, 4.63; N, 8.40; Found: C, 59.94; H, 4.46; N, 8.17.

### 1.2.5 Synthesis of tris-terpyridine **TT**



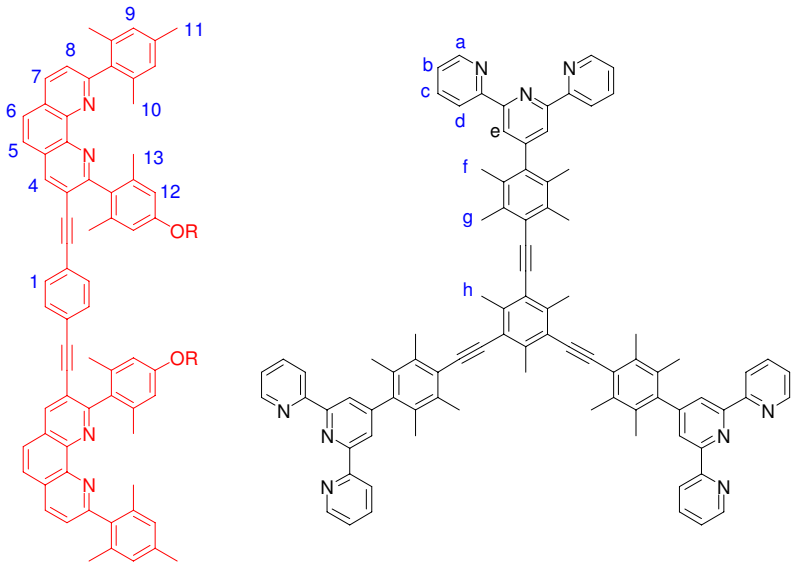

**TT**

Compound **D** (2.00g, 4.07 mmol), 1,3,5-triethynyl-2,4,6-trimethylbenzene [3] (260 mg, 1.35 mmol) and $Pd[(PPh_3)_4]$ (230 mg, 200 µmol) were dissolved in dry DMF (50 mL) and dry triethylamine (50 mL). After deoxygenating the reaction mixture with nitrogen gas for 15 min., it was heated to 60 °C for 4 hours. Then water (200 mL) was added, and the mixture was extracted twice with dichloromethane (2x100 mL). The organic layers were combined and dried over $MgSO_4$. After removal of the solvents, the residue was purified first by column chromatography on aluminium oxide ($CH_2Cl_2$, $R_f = 0.5$) and second by recrystallization from chloroform to furnish a light yellow solid (1.52 g, 1.18 mmol, 87%).

MP: >300 ºC; IR (KBr): $\tilde{\nu}$ = 2920 (w), 2360 (w), 2343 (w), 1604 (w), 1584 (s), 1566 (s), 1543 (m), 1467 (s), 1388 (m), 1263 (w), 1131 (w), 990 (w), 854 (w), 793 (s), 741 (m), 654 (s), 621 (w); $^1$H-NMR ($CDCl_3$, 400 MHz): δ = 2.08 (s, 18H, f-H), 2.67 (s, 18H, g-H), 3.00 (s, 9H, h-H), 7.34-7.38 (m, 6H, b-H), 7.91 (td, *J* = 7.8 and 1.6 Hz, 6H, c-H), 8.36 (s, 6H, e-H), 8.71-8.76 (m, 12H, a-, d-H); $^{13}$C-NMR ($CDCl_3$, 100 MHz): δ = 18.3, 19.0, 21.3, 95.1, 96.8, 121.4, 122.0, 122.4, 123.7, 123.9, 131.3, 136.2, 137.0, 140.2, 141.7, 149.3, 153.0, 155.8, 156.3; ESI-MS Calcd for $[C_{90}H_{75}N_9+H]^+$: *m/z* 1282.6, Found: *m/z*

1283.0; ESI-MS Calcd for $[C_{90}H_{75}N_9+2H]^{2+}$: *m/z* 641.8, Found: *m/z* 1642.0; Anal Calcd for $C_{90}H_{75}N_9 \bullet 0.5CHCl_3$: C, 80.98; H, 5.67; N, 9.39; Found: C, 80.91; H, 5.67; N, 9.02.

1.2.6 Synthesis of prism **P3**

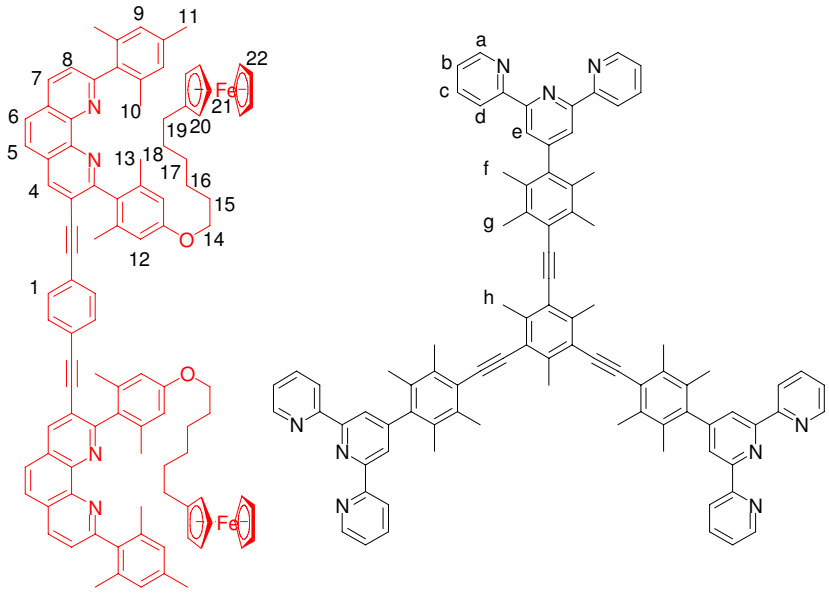



The linear bis-phenanthroline **BP1** (3.20 mg, 3.34 µmol) and $Zn(CF_3SO_3)_2$ (2.43 mg, 6.68 µmol) were dissolved slowly in a mixture of dichloromethane/methanol (8.0 mL / 2.0 mL), then the mixture was heated at 40 °C for 5 minutes before a solution of tris-terpyridine **TT** (2.86 mg, 2.23 µmol) in dichlorometane (10 mL) was added. After 10 ml of acetonitrile had been added to this mixture, the mixture was stirred for 30 minutes at room temperature. The solvents were removed. The residue was analysed by $^1$H-NMR, $^{13}$C-NMR, ESI-MS, and UV/Vis without further purification.

MP: >300 ºC; IR (KBr): $\tilde{\nu}$ = 2919 (w), 2361 (w), 1614 (s), 1586 (s), 1507 (w), 1458 (m), 1395 (w), 1257 (s), 1260 (s), 1032 (s), 908 (w), 850 (m), 794 (w), 640 (m); $^1$H-NMR ($CD_3CN$, 400 MHz): δ = 1.06 (s, 36H, 10-H), 1.26 (s, 54H, 11-, 13-H), 2.18 (s, 36H, g-H), 2.22 (s, 18H, $f_1$-H), 2.25 (s, 18H, $f_2$-H), 2.99 (s, 18H, h-H), 6.08 (s, 12H, 9-H), 6.28 (s, 12H, 12-H), 6.54 (s, 12H, 1-H), 7.50 (t, *J* = 6.2 Hz, 12H, b-H), 7.84 (d, *J* = 6.2 Hz, 12H, a-H), 8.15 (d, *J* = 8.4 Hz, 6H, 8-H), 8.26 (t, *J* = 7.8 Hz, 12H, c-H), 8.40 (s, 12H, e-H), 8.50 (d, *J* = 9.1 Hz, 6H, 6-H), 8.53 (d, *J* = 7.8 Hz, 12H, d-H), 8.58 (d, *J* = 9.1 Hz, 6H, 5-H), 9.09 (s, 6H, 4-H), 9.15 (d, *J* = 8.4 Hz, 6H, 7-H); $^{13}$C-NMR ($CD_3CN$, 100 MHz): δ = 18.6, 18.7, 18.8, 19.4, 19.5, 19.6, 20.8, 22.2, 88.5, 97.3, 97.6, 99.0, 113.9, 120.1, 122.4, 122.9, 123.3, 124.1, 124.2, 124.8, 125.6, 128.1, 128.2, 128.8, 129.0, 129.3, 130.2, 130.4, 131.6, 132.2, 132.4, 135.5, 135.8, 137.0, 137.4, 138.2, 138.7, 140.3, 140.4, 142.1,

142.3, 143.2, 143.4, 147.0, 148.1, 150.1, 158.5, 160.4, 161.8, 164.3; ESI-MS Calcd for $[C_{384}H_{312}N_{30}O_6Zn_6+8OTf]^{4+}$ $[M-4OTf]^{4+}$: *m/z* 1756.9, Found: *m/z* 1756.8, ESI-MS Calcd for $[C_{384}H_{312}N_{30}O_6Zn_6+7OTf]^{5+}$ $[M-5OTf]^{5+}$: *m/z* 1375.7, Found: *m/z* 1375.6, ESI-MS Calcd for $[C_{384}H_{312}N_{30}O_6Zn_6+6OTf]^{6+}$ $[M-6OTf]^{6+}$: *m/z* 1121.6, Found: *m/z* 1121.5, ESI-MS Calcd for $[C_{384}H_{312}N_{30}O_6Zn_6+5OTf]^{7+}$ $[M-7OTf]^{7+}$: *m/z* 940.1, Found: *m/z* 939.8, ESI-MS Calcd for $[C_{384}H_{312}N_{30}O_6Zn_6+4OTf]^{8+}$ $[M-8OTf]^{8+}$: *m/z* 803.9, Found: *m/z* 803.7, ESI-MS Calcd for $[C_{384}H_{312}N_{30}O_6Zn_6+3OTf]^{9+}$ $[M-9OTf]^{9+}$: *m/z* 698.0, Found: *m/z* 698.0, ESI-MS Calcd for $[C_{384}H_{312}N_{30}O_6Zn_6+2OTf]^{10+}$ $[M-10OTf]^{10+}$: *m/z* 613.3, Found: *m/z* 613.3; Anal Calcd for $C_{396}H_{312}F_{36}N_{30}O_{42}S_{12}Zn_6\bullet 8CH_2Cl_2$: C, 58.44; H, 3.98; N, 5.06; S, 4.63; Found: C, 58.20; H, 3.89; N, 5.29; S, 5.01.

1.2.7 Synthesis of prism **P4**




The linear bis-phenanthroline **BP2** (5.10 mg, 3.34 µmol) and $Zn(CF_3SO_3)_2$ (2.43 mg, 6.68 µmol) were dissolved slowly in a mixture of dichloromethane/methanol (8.0 mL / 2.0mL). Then the mixture was heated at 40 °C for 5 minutes before a solution of tris-terpyridine **TT** (2.86 mg, 2.23 µmol) in dichlorometane (10 mL) was added. After 10 ml of acetonitrile had been added to this mixture, the mixture was stirred at room temperature for 30 minutes. The solvents were removed, and the residue was analysed without further purification by $^1$H-NMR, $^{13}$C-NMR, ESI-MS and UV/Vis.

MP: >300 ºC; IR (KBr): $\tilde{\nu}$ = 2927 (w), 2361 (w), 1603 (m), 1576 (w), 1260 (s), 1161 (m), 1031 (s), 854 (w), 796 (w), 638 (m); $^1$H-NMR ($CD_3CN$, 400 MHz): δ = 1.17 (s, 36H, 10-H), 1.26(s, 54H, 11-, 13-H), 1.38 (m, 24H, 16-, 17-H), 1.52 (m, 12H, 18-H), 2.15 (s, 36H, g-H), 2.18 (s, 18H, $f_1$-H), 2.20 (s, 18H, $f_2$-H), 2.28 (m, 12H, 19-H), 2.79 (s, 18H, h-

H), 3.11 (m, 12H, 15-H), 3.59 (m, 12H, 14-H), 4.00 (m, 54H, 20-, 21-, 22-H), 5.96 (s, 12H, 9-H), 6.31 (s, 12H, 12-H), 6.88 (s, 12H, 1-H), 7.51 (t, $J$ = 6.2 Hz, 12H, b-H), 7.80 (d, $J$ = 6.2 Hz, 12H, a-H), 8.18 (d, $J$ = 8.4 Hz, 6H, 8-H), 8.25 (t, $J$ = 8.0 Hz, 12H, c-H), 8.26 (s, 12H, e-H), 8.43 (d, $J$ = 8.0 Hz, 6H, d-H), 8.52 (d, $J$ = 9.1 Hz, 6H, 6-H), 8.58 (d, $J$ = 9.1 Hz, 6H, 5-H), 9.16 (d, $J$ = 8.4 Hz, 6H, 7-H), 9.18 (s, 6H, 4-H); $^{13}$C-NMR ($CD_3CN$, 100 MHz): δ = 19.0, 19.3, 19.4, 19.6, 19.8, 19.9, 21.1, 22.3, 26.7, 26.9, 30.0, 30.4, 31.5, 68.9, 69.2 (Fc), 69.6 (Fc), 70.8 (Fc), 87.6, 96.8, 97.8, 97.9, 113.4, 120.5, 123.3, 123.4, 123.7, 124.3, 124.4, 125.2, 125.9, 128.6, 129.2, 129.7, 130.0, 130.4, 130.8, 132.2, 132.4, 132.8, 135.8, 136.2, 137.7, 137.8, 138.3, 138.6, 140.8, 140.9, 142.4, 143.3, 143.6, 143.7, 144.0, 147.2, 148.3, 150.4, 160.3 160.7, 162.1, 163.6; ESI-MS Calcd for $[C_{480}H_{432}Fe_6N_{30}O_6Zn_6+7OTf]^{5+}$ $[M-5OTf]^{5+}$: *m/z* 1697.5, Found: *m/z* 1697.8, ESI-MS Calcd for $[C_{480}H_{432}Fe_6N_{30}O_6Zn_6+6OTf]^{6+}$ $[M-6OTf]^{6+}$: *m/z* 1389.8, Found: *m/z* 1390.0, ESI-MS Calcd for $[C_{480}H_{432}Fe_6N_{30}O_6Zn_6+5OTf]^{7+}$ $[M-7OTf]^{7+}$: *m/z* 1169.9, Found: *m/z* 1170.0, ESI-MS Calcd for $[C_{480}H_{432}Fe_6N_{30}O_6Zn_6+4OTf]^{8+}$ $[M-8OTf]^{8+}$: *m/z* 1005.0, Found: *m/z* 1004.7, ESI-MS Calcd for $[C_{480}H_{432}Fe_6N_{30}O_6Zn_6+3OTf]^{9+}$ $[M-9OTf]^{9+}$: *m/z* 876.8, Found: *m/z* 876.6, ESI-MS Calcd for $[C_{480}H_{432}Fe_6N_{30}O_6Zn_6+2OTf]^{10+}$ $[M-10OTf]^{10+}$: *m/z* 774.2, Found: *m/z* 774.2; Anal Calcd for $C_{492}H_{432}F_{36}Fe_6N_{30}O_{42}S_{12}Zn_6\bullet 6CH_2Cl_2$: C, 61.39; H, 4.59; N, 4.31; S, 3.95; Found: C, 61.03; H, 4.27; N, 4.00; S, 4.33.

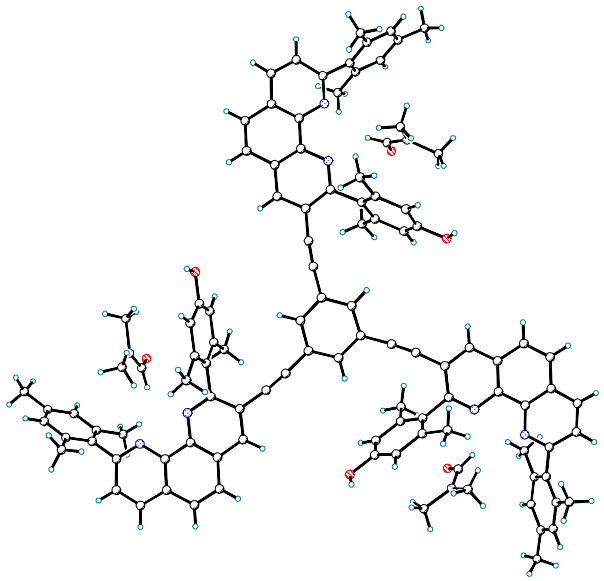


**Figure S1.** X-ray structure of ligand **TP**. Three DMF solvate molecules cocrystallised with **TP** and are bound by hydrogen bonds.

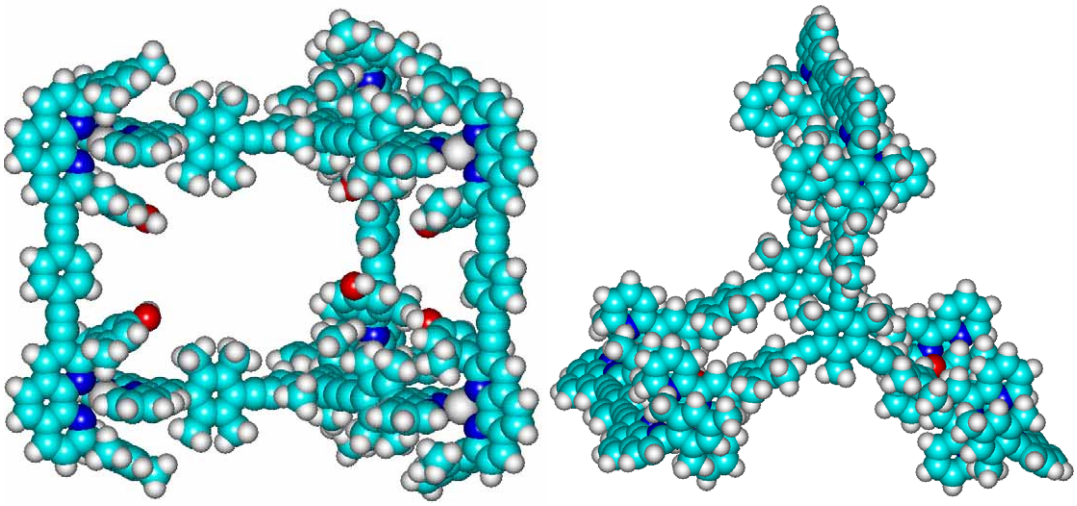


**Figure S2.** HyperChem® structrue of prism **P3**. The atoms are colour coded for clarity: carbon: cyan; nitrogen: blue; oxygen: red; zinc: *hidden*. Left: side view; right: top view.

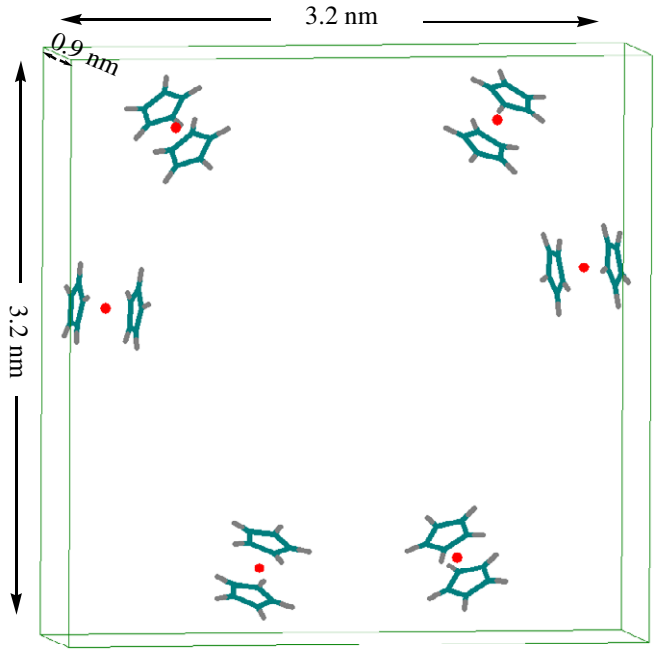



**Figure S3.** Six ferrocene units represent those in **P4**. They fill a periodic box of 9216 $Å^3$ as calculated by Hyperchem®. The average distance between every two iron atoms are 2.08 nm.

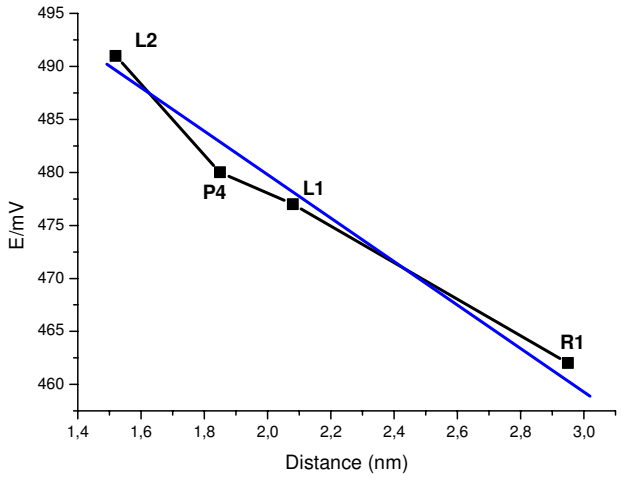



**Figure S4.** A plot of ferrocene redox potentials *vs*. the average distance between ferrocene units of ferrocene containing supramolecular complexes. The supramolecular complexes **R1**, **L1** and **L2** were reported earlier.[1]

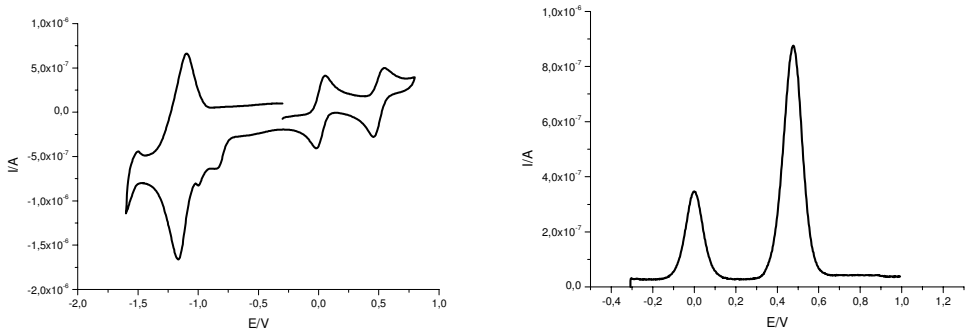



**Figure S5.** CV (left: scan rate = 100 mV $s^{-1}$) and DPV (right: step potential 2 mV) of prism **P4** recorded in acetonitrile (0.10 M $^{n}Bu_4NPF_6$) using DMFc (decamethyl-ferrocene) as internal standard ($E_{1/2}$ = 0). Peaks at −0.8 V, −0.9 V and −1.2 V are due to the reduction of the phenanthroline and terpyridine units, while the peak at 0.5 V corresponds to the redox potential of the ferrocene units.

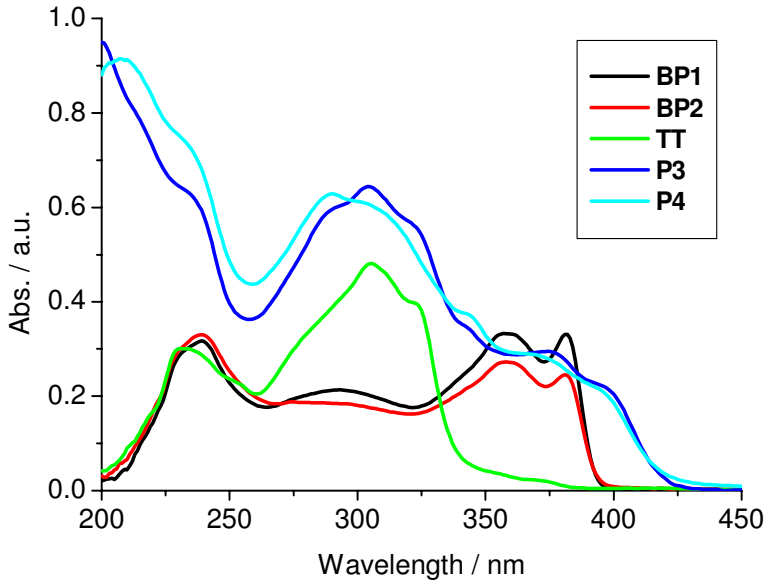



**Figure S6.** UV-Vis absorption spectra of various ligands (3.0 x $10^{-6}$ M in DCM) and complexes (1.0 x $10^{-6}$ M in acetonitrile). **BP1** and **BP2** display broad absorption peaks at 357 and 382 nm that can be assigned to the excitation of the phenylethynyl (p-e) units.[4] The p-e absorptions in **P3** and **P4** are red-shifted by about 20 nm, due to the presence of the phen-Zn(II)-terpy coordination centres at the two termini of **BP1** and **BP2**.

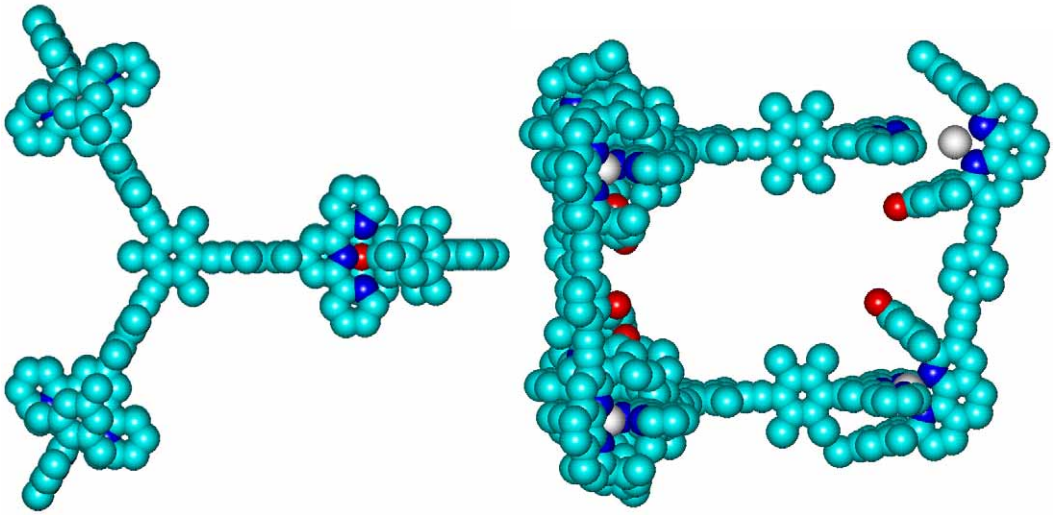


**Figure S7.** Illustration of the assumed final step of the prism assembly of **P3**. The atoms are colour coded for clarity: carbon: cyan; nitrogen: blue; oxygen: red; zinc: white. Left: top view; right: side view.

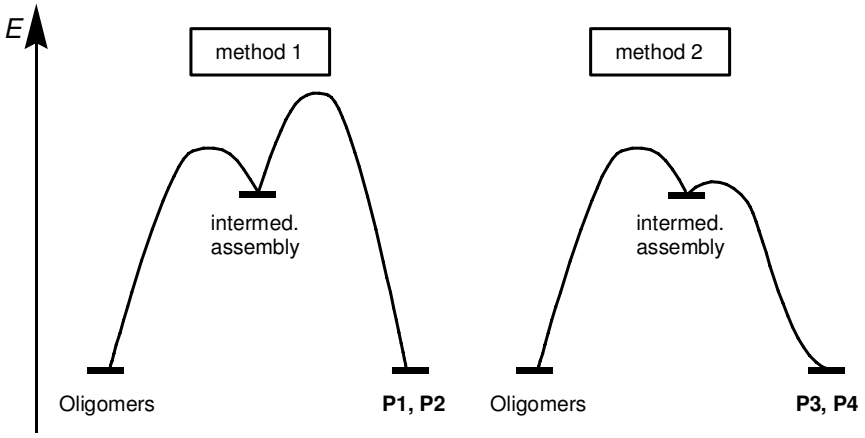



**Figure S8.** Graphical illustration of the final step of the prism assembly along route 1 (left) and 2 (right). Oligomers = mononuclear and oligomeric zinc(II) bisterpyridine complexes.

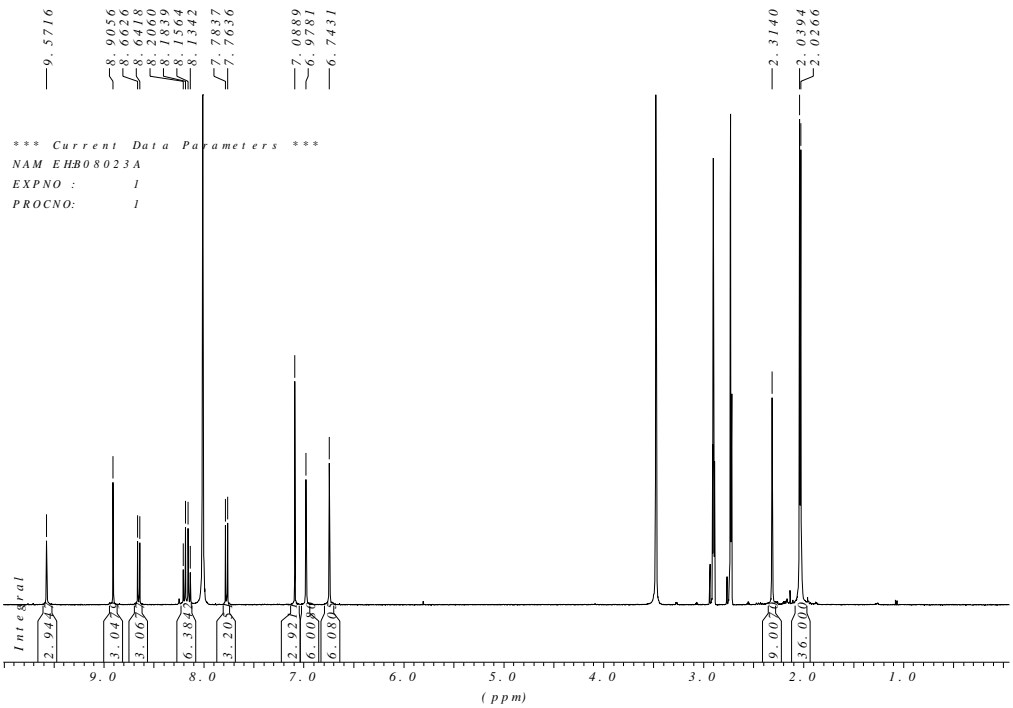



**Figure S9.** $^{1}$H-NMR spectrum of ligand **TP**.

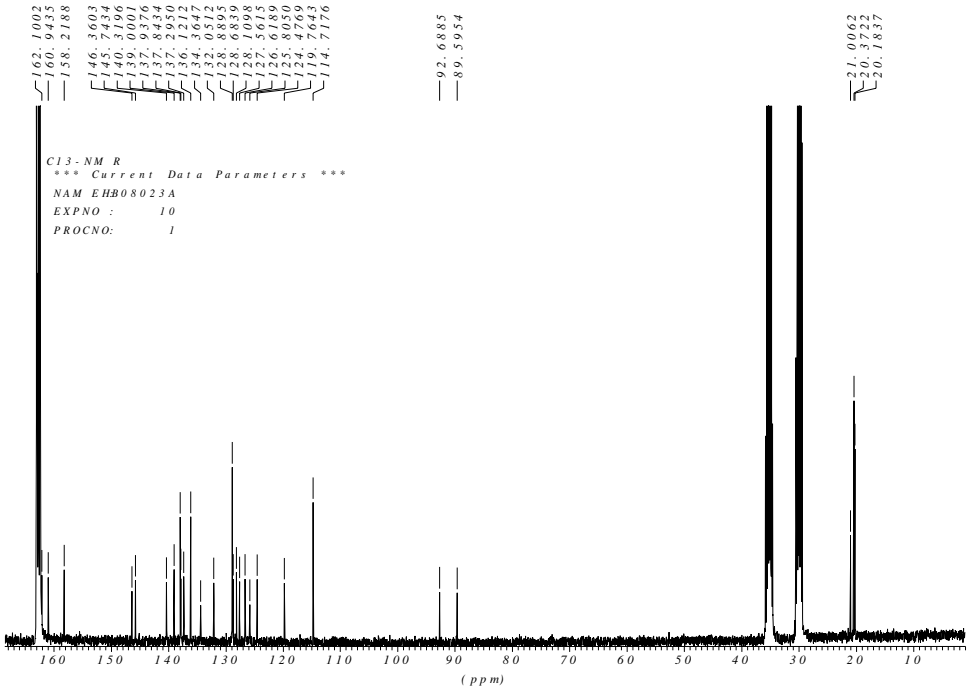



**Figure S10.** $^{13}$C-NMR spectrum of ligand **TP**.

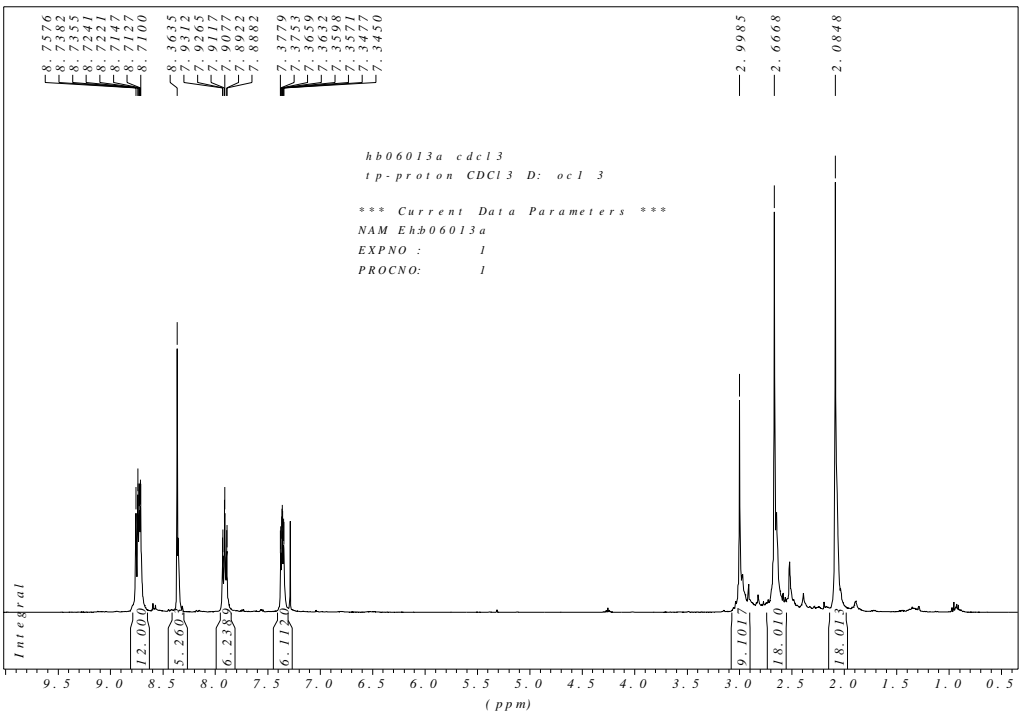



**Figure S11.** $^{1}$H-NMR spectrum of ligand **TT**.

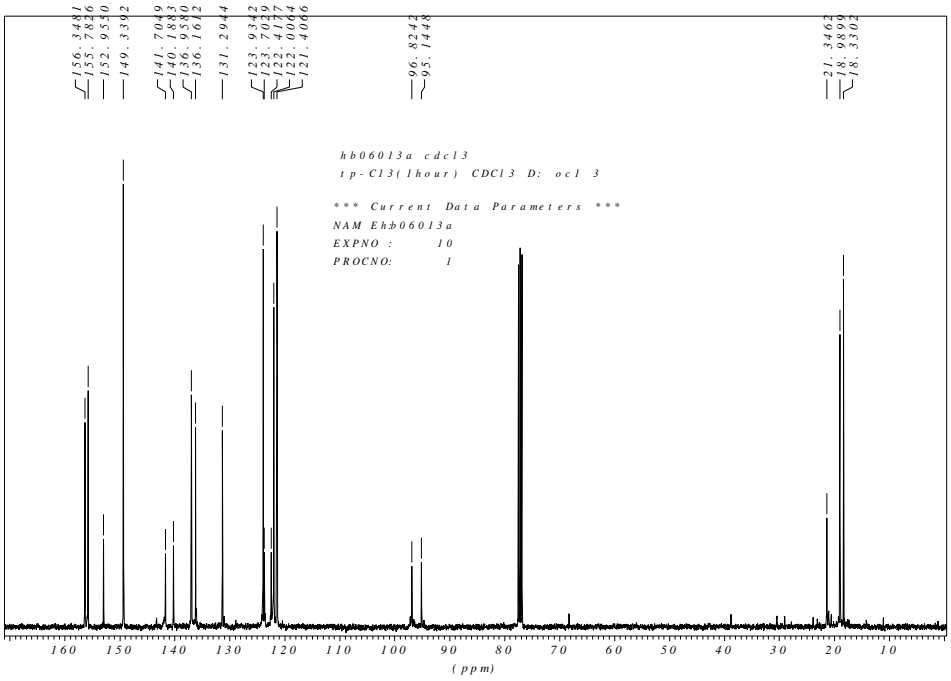



**Figure S12.** $^{13}$C-NMR spectrum of ligand **TT**.

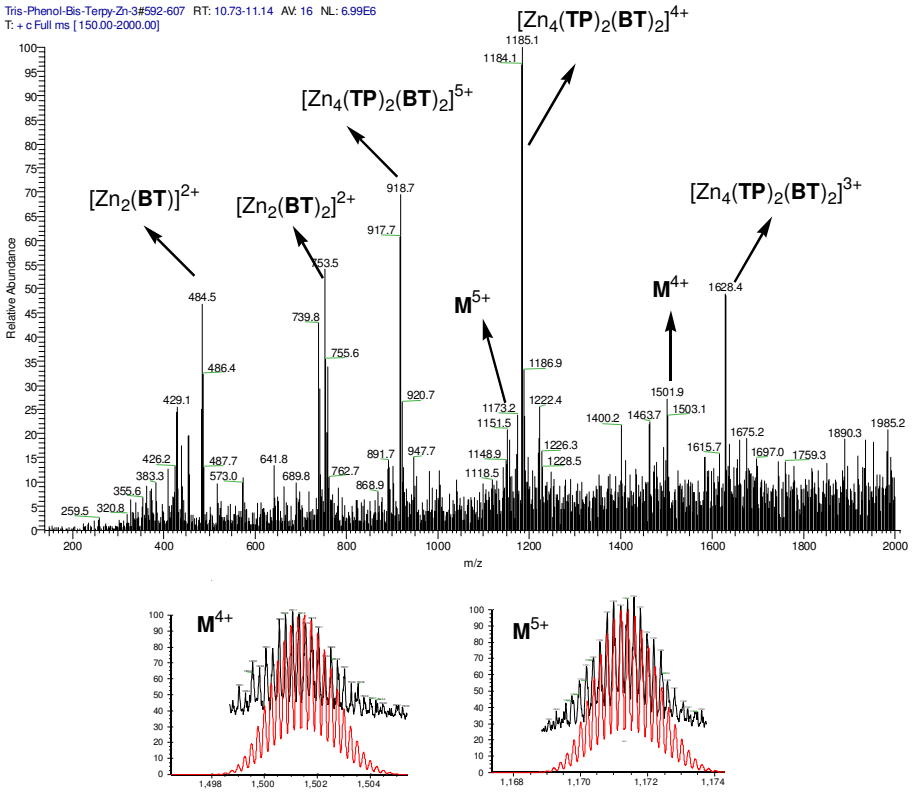



**Figure S13.** Top: ESI-MS spectrum of prism **P1**. Bottom: Theoretical (red) and experimental (black) isotopic distributions of nanoprism **P1**. For clarity purposes, the counter anion count was omitted in the labeling.

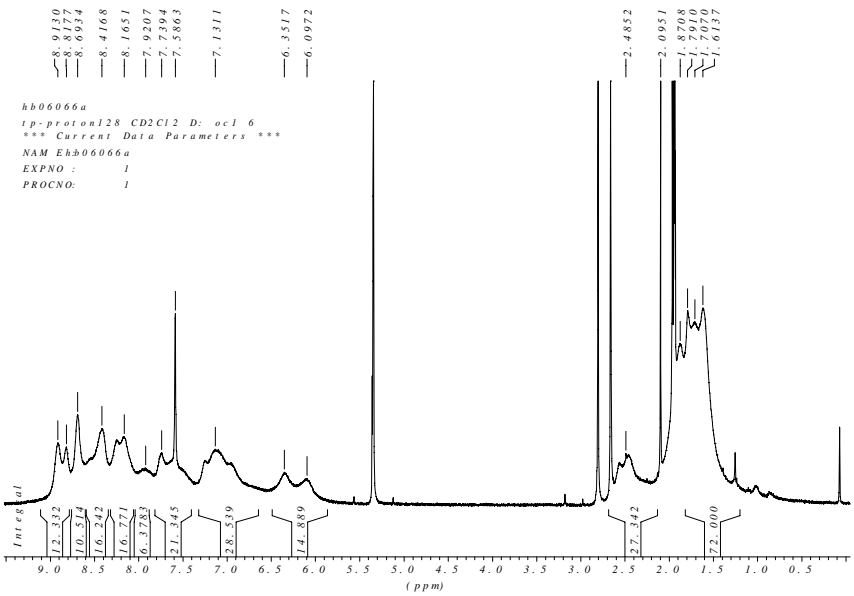



**Figure S14.** $^{1}$H-NMR spectrum of prism **P1** that is broadened due to rapid equilibration with oligomeric terpyridine complexes.

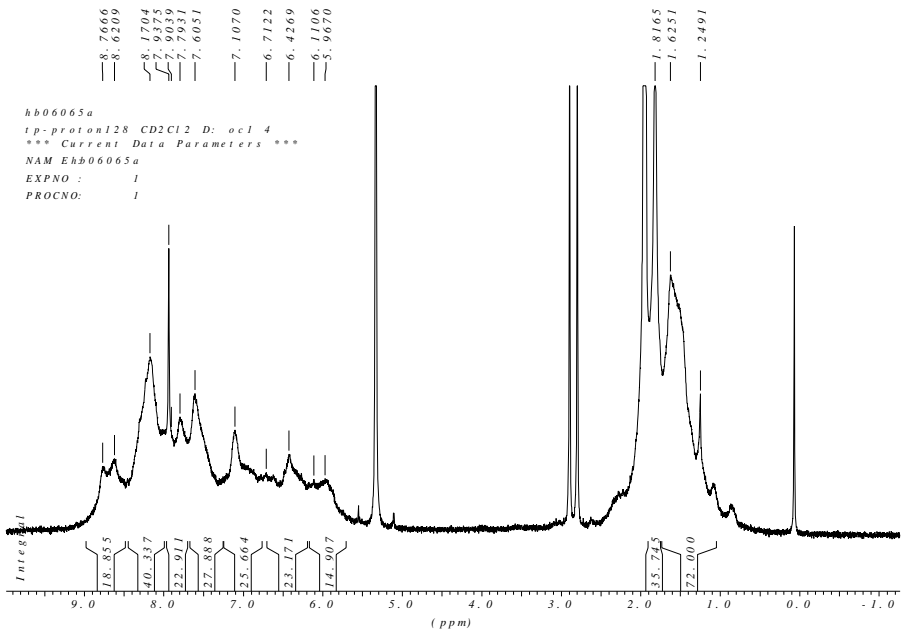



**Figure S15.** $^{1}$H-NMR spectrum of prism **P2** that is broadened due to rapid equilibration with oligomeric terpyridine complexes..

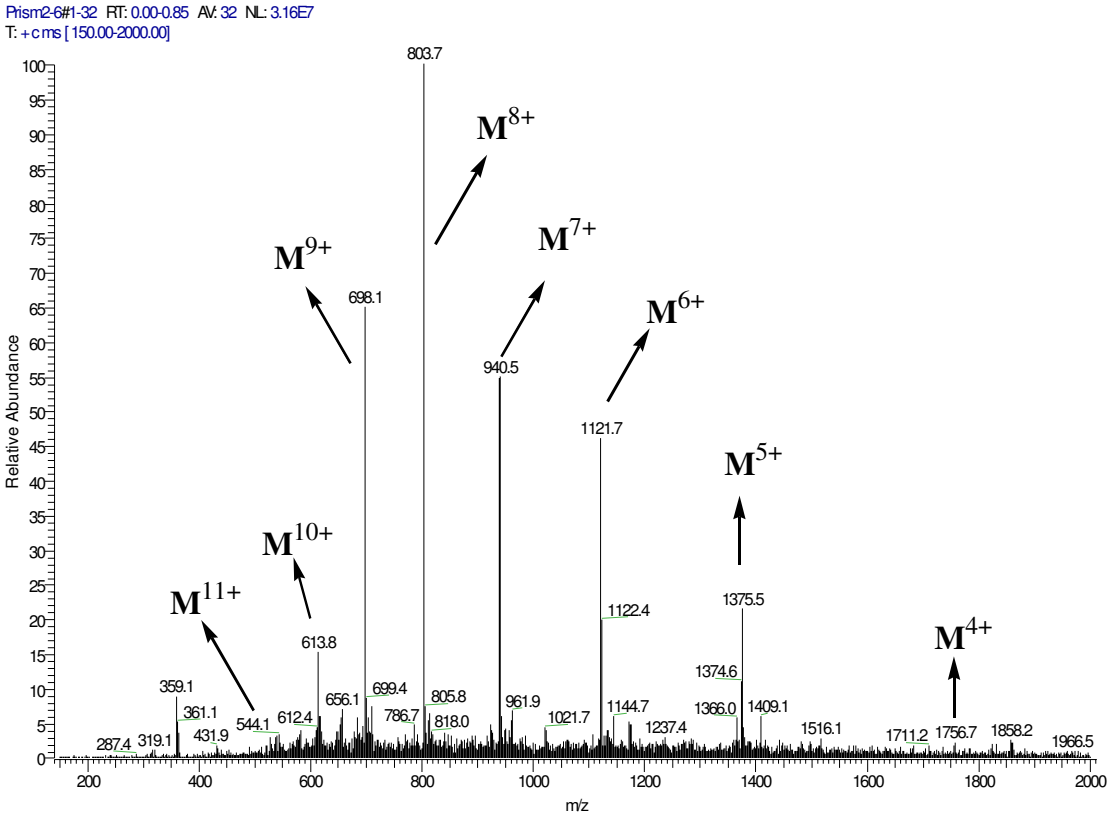



**Figure S16.** ESI-MS spectrum of prism **P3**. For clarity purposes, the counter anion count was omitted in the labeling.

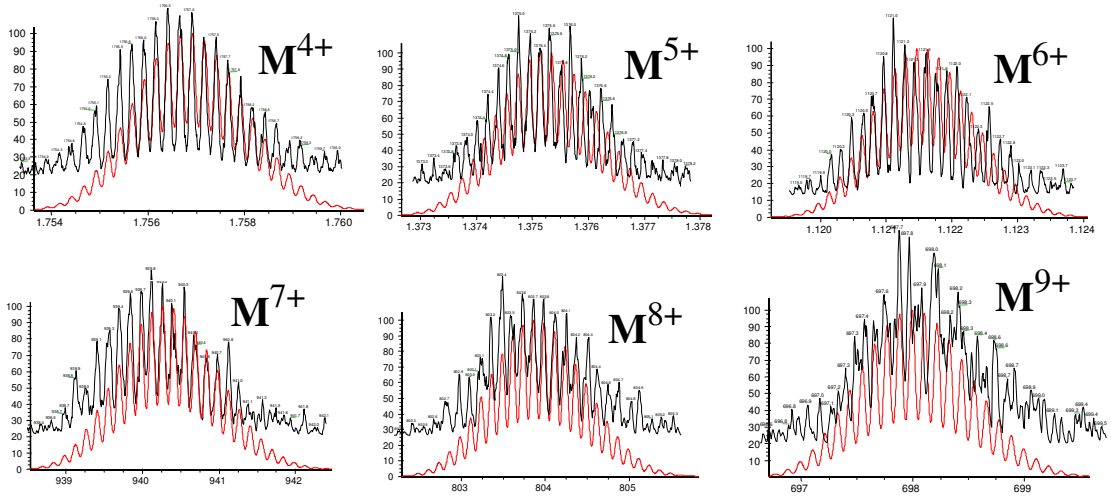



**Figure S17.** Theoretical (red) and experimental (black) isotopic distributions of nanoprism **P3**. For clarity purposes, the counter anion count was omitted in the labeling.

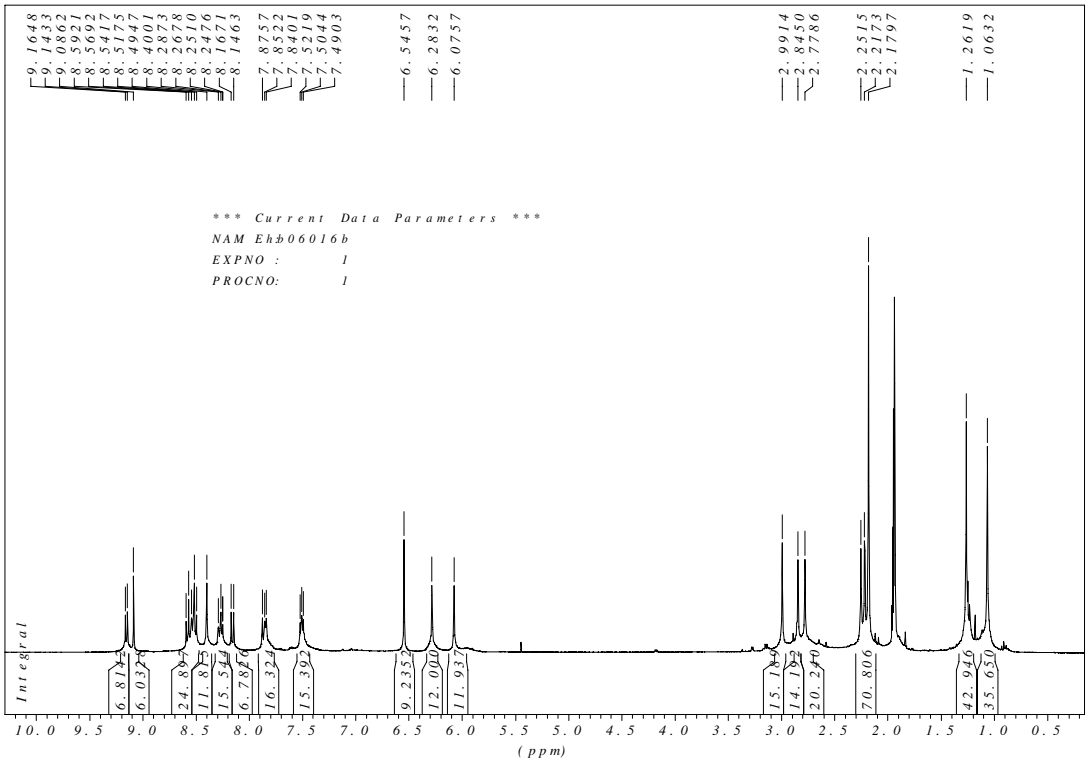



**Figure S18.** $^{1}$H-NMR spectrum of nanoprism **P3**.

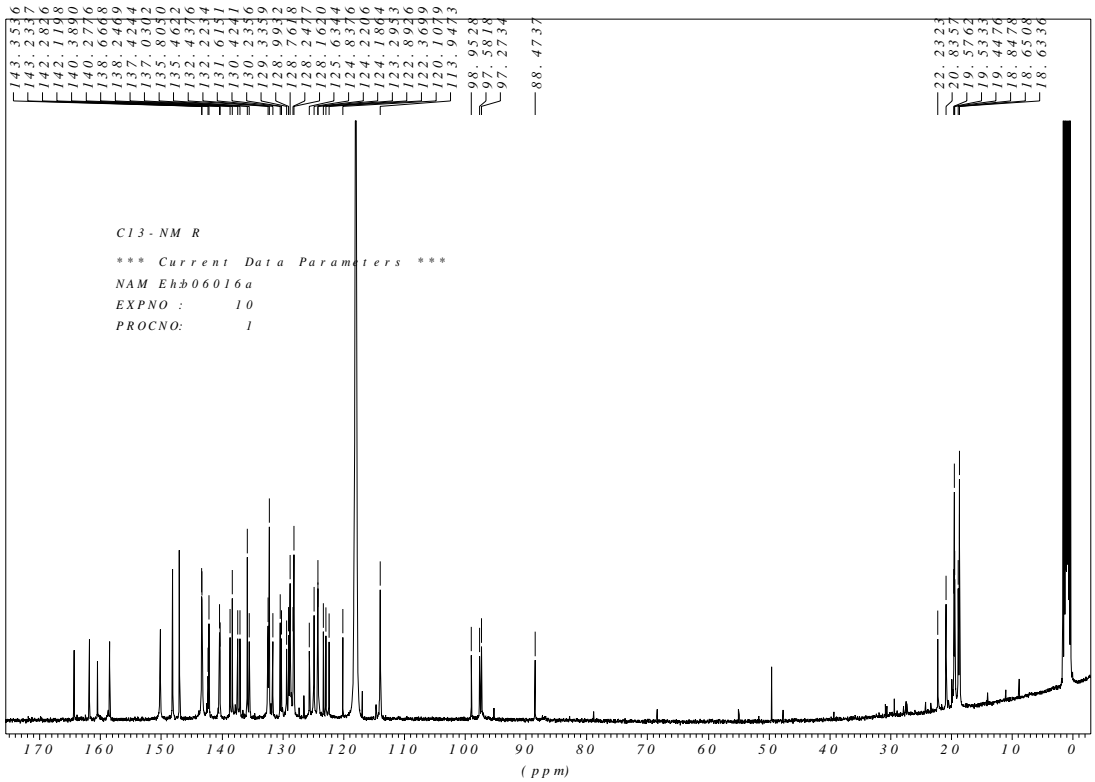



**Figure S19.** $^{13}$C-NMR spectrum of prism **P3**.

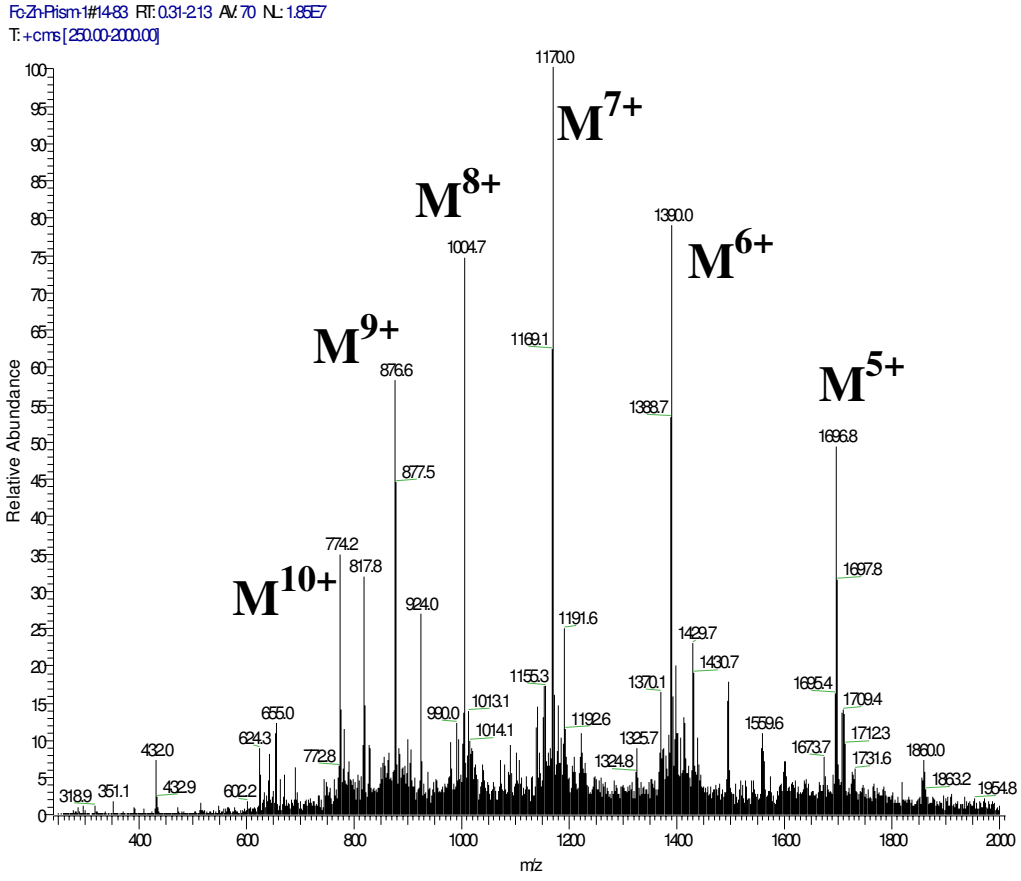



**Figure S20.** ESI-MS spectrum of nanoprism **P4**. For clarity purposes, the counter anion count was omitted in the labeling.

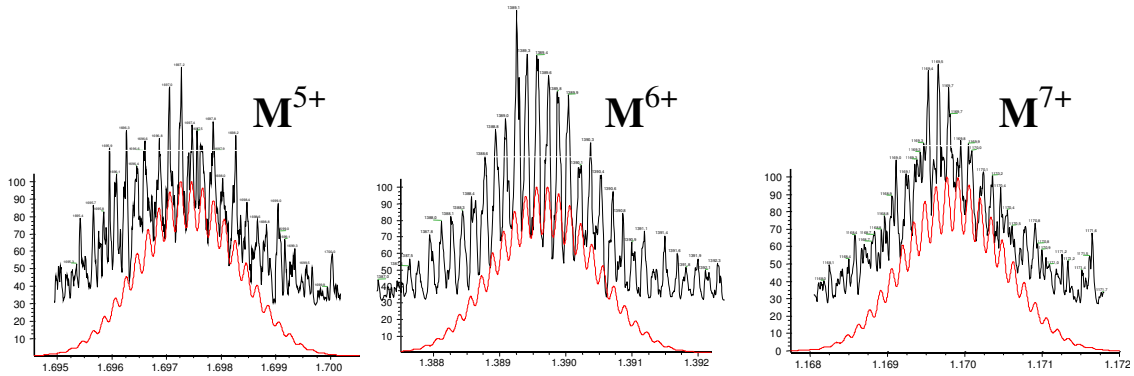



**Figure S21.** Theoretical (red) and experimental (black) isotopic distribution of prism **P4**. For clarity purposes, the counter anion count was omitted in the labeling.

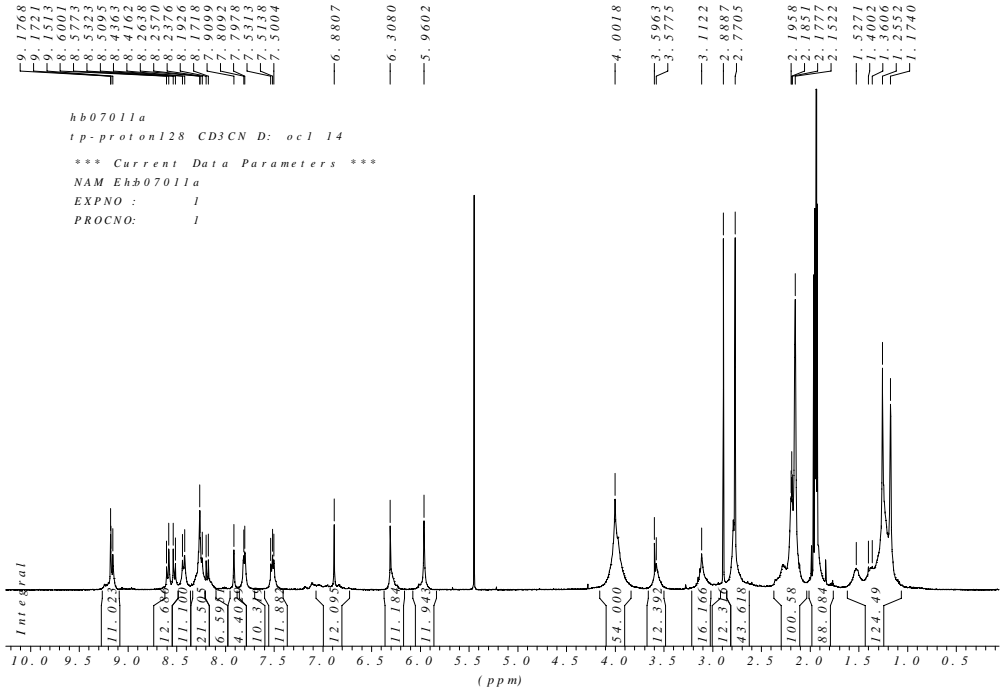



**Figure S22.** $^{1}$H-NMR spectrum of nanoprism **P4**.

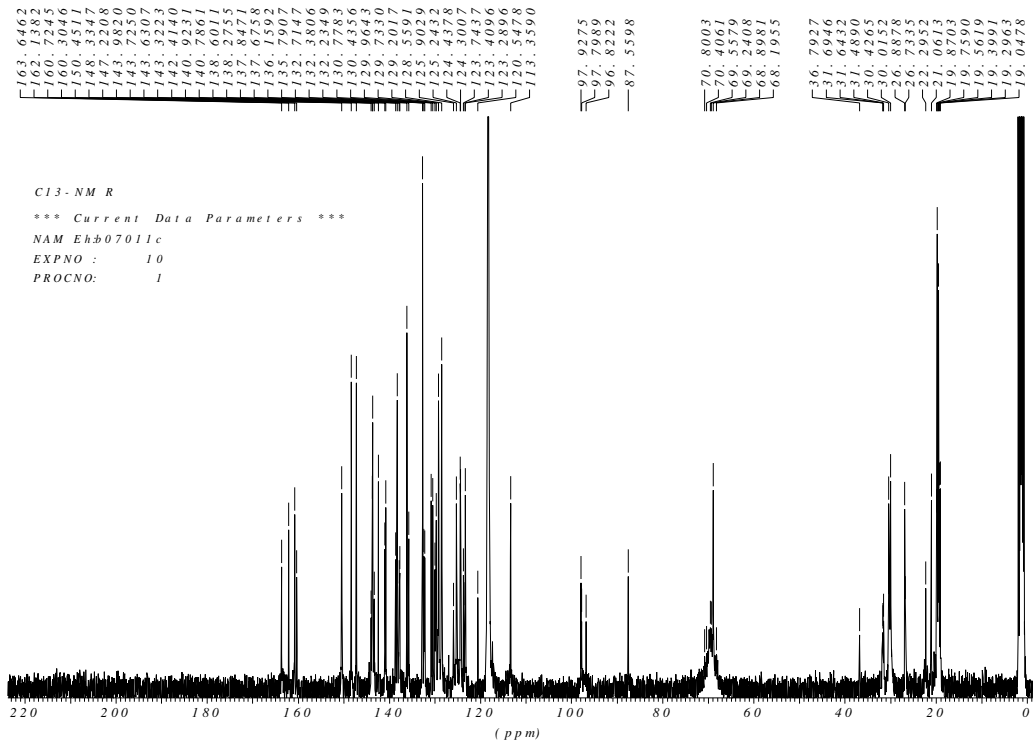



**Figure S23.** $^{13}$C-NMR spectrum of nanoprism **P4**.

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2 E. C. Constable, E. Figgemeier, C. E. Housecroft, J. Olsson and Y. C. Zimmermann, *Dalton Transactions,* 2004, 1918-1927.

3 N. Ohshiro, F. Takei, K. Onitsuka and S. Takahashi, *Chem. Lett.,* 1996, 871-872.

4 (a) H. S. Joshi, R. Jamshidi and Y. Tor, *Angew. Chem., Int. Ed. Engl.,* 1999, **38**, 2721-2725. (b) E. Birckner, U. W. Grummt, A. H. Goeller, T. Pautzsch, D. A. M. Egbe, M. Al-Higari and E. Klemm, *J. Phys. Chem. A,* 2001, **105**, 10307-10315.