

Electronic Supplementary Information
Carbazole-functionalized Polyphenylene-decorated solid state emissive D-A-D molecules: Reduced Donor-Acceptor interaction and enhanced emission in solid state

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Experimental Section

General Information

All reagents were purchased from Aldrich and were used without further purification. CH₂Cl₂ was distilled from CaH₂. Mass spectra were recorded on Bruker MicroToff/QII. Differential scanning calorimetry (DSC), and thermal gravimetric analysis (TGA) was recorded on EXSTAR TG/DTA 6300. HPLC analyses were performed on Shimadzu LC-20AD using silica column. The TEM images were recorded from Transmission Electron Microscope (TEM) - JEOL 2100F. The dynamic light scattering (DLS) data were recorded with MALVERN Instruments (Nano-ZS). The X-ray powder diffraction (XRPD) measurements were recorded on a Rigaku miniflex X ray diffractometer with Cu K α radiation in the range of $\theta = 0^\circ$ to 100° . Elemental analysis was carried out on Elementar vario EL cube. The Time resolved fluorescence spectra were recorded with a HORIBA Time Resolved Fluorescence Spectrometer. UV-vis spectra were recorded on SHIMADZU UV-2450 spectrophotometer, with a quartz cuvette (path length, 1 cm). The cell holder was thermostatted at 25 °C. The fluorescence spectra were recorded with a SHIMADZU 5301 PC spectrofluorimeter. For fluorescence measurements, each time 3 mL respective solution of **1-3** (10 μ M) was filled in a quartz cuvette (path length, 1 cm) and excitation was provided at their respective absorption maxima. ¹H and ¹³C were recorded on JOEL-FT NMR-AL 300 MHz spectrophotometer and BRUKER-AVANCE-II FT-NMR-AL 500 MHz spectrophotometer using CDCl₃ as solvent and TMS as internal standard. Data is reported as follows: chemical shifts in ppm (δ), multiplicity (s = singlet, d = doublet, m = multiplet), coupling constant *J* (Hz), integration, and interpretation. Silica gel 60–120 mesh was used for column chromatography.

Quantum yield calculation

Fluorescence quantum yield was determined using optically matching solutions of diphenylanthracene ($\Phi_F = 0.9$ in cyclohexane) as standard and quantum yield was calculated using the equation:

$$\Phi_{Fs} = \Phi_{Fr} \frac{1-10^{-A_r L_r}}{1-10^{-A_s L_s}} \times \frac{N_s^2}{N_r^2} \times \frac{D_s}{D_r}$$

Φ_{Fs} and Φ_{Fr} are the radiative quantum yields of sample and the reference respectively, A_s and A_r are the absorbance of the sample and the reference respectively, D_s and D_r the respective areas of emission for sample and reference. L_s and L_r are the lengths of the absorption cells of

sample and reference respectively. N_s and N_r are the refractive indices of the sample and reference solutions.

Electrochemical studies

Electrochemical studies were carried out on CH Instruments CH1660D in DCM solution with 0.1M tetrabutylammonium perchlorate as electrolyte. Glassy carbon electrode was used as working electrode, Ag/AgNO₃ as reference electrode and platinum wire as counter electrode. Experiments were calibrated with standard ferrocenium redox system. The cell was purged with nitrogen prior to each scan and the scans were performed at the rate of 50mV/sec. at room temperature.

Experimental section

Compound **4**,¹ **5**² and **6**³ were synthesised according to previous reported procedures in literature while compound **7** and **11** were commercially available and used as such.

Synthesis

Synthesis of 2,5-bis(4-((9-hexyl-9H-carbazol-3-yl)ethynyl)phenyl)-1,3,4-oxadiazole (**8**)

In a flame dried two necked round bottomed flask, precursor **5** (0.62 g, 1.3 mmol), CuI (0.49 g, 0.26 mmol), and Pd(PPh₃)₄ catalyst (0.2 g, 0.13 mmol) was stirred in toluene (10 ml) and triphenylamine (5 ml) under nitrogen for 20 min. Alkyne **4** (0.83 g, 3.01 mmol) in toluene (5 ml) was added dropwise. The resulting mixture was stirred at 55 °C for 24 h. After cooling to room temperature, a saturated solution of NH₄Cl was added and the mixture was extracted with DCM twice. The organic layer was washed with brine and dried over Na₂SO₄. The solvent was evaporated to dryness and the crude was purified by column chromatography in hexane–Ethyl acetate (9: 1) to afford 0.45 g of a yellow solid in 45% yield.¹H NMR (400 MHz, CDCl₃) δ 8.33 (s, 2H), 8.12 (t, $J = 7.3$ Hz, 6H), 7.68 (dd, $J = 20.3, 8.3$ Hz, 6H), 7.50 (t, $J = 7.6$ Hz, 2H), 7.40 (dd, $J = 12.2, 8.4$ Hz, 4H), 7.28 (d, $J = 7.4$ Hz, 2H), 4.30 (t, $J = 7.2$ Hz, 4H), 1.93 – 1.79 (m, 4H), 1.41 – 1.25 (m, 12H), 0.87 (t, $J = 6.7$ Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 164.31, 140.78, 140.34, 131.91, 129.30, 127.58, 126.79, 126.20, 124.28, 122.87, 122.50, 122.35, 120.51, 119.44, 112.42, 108.98, 108.82, 94.33, 87.02, 43.21, 31.53,

¹ R. Grisorio, C. Piliago, P. Fini, P. Cosma, P. Mastrorilli, G. Gigli, G. P. Suranna and C. F. Nobile, J. Phys. Chem. C, **2008**, 112, 7005.

² Dong-Cheol Shin, Jun-Hwan Ahn, Yun-Hi Kim, Soon-Ki Kwon. Journal of Polymer Science: Part A: Polymer Chemistry, Vol. 38, 3086–3091 (**2000**).

³ C. R. Newmoyer, E. D. Amstutz, J. Am. Chem. Soc. **1947**, 69, 1920.

28.91, 26.93, 22.53, 14.00. HPLC analysis, 100% (silica column, $\lambda = 308$ nm, *i*-PrOH : hexane = 05 : 95, flow rate = 1.0 mL/min, $T_R = 3.456$ min.) MALDI-TOF MS m/z calcd. for $C_{54}H_{48}N_4O$ (M^+): 768.38. Found: 768.38.

Synthesis of 3,7-bis((9-hexyl-9H-carbazol-3-yl)ethynyl)dibenzo[b,d]thiophene 5,5-dioxide (9). The same procedure as described for **3** was followed. Precursor **6** (0.47 g, 1.25 mmol), CuI (43 mg, 0.22 mmol), and Pd(PPh₃)₂Cl₂ catalyst (93 mg, 0.13 mmol), Alkyne **4** (0.76 g, 2.76 mmol). The product was purified by column chromatography in hexane–Ethyl acetate (9: 1). Yield: 60% (0.57g). ¹H NMR (300 MHz, CDCl₃) δ 8.31 (s, 2H), 8.11 (d, $J = 7.5$ Hz, 2H), 7.99 (s, 2H), 7.80 – 7.70 (m, 4H), 7.64 (d, $J = 7.3$ Hz, 2H), 7.44 (ddd, $J = 40.6, 18.7, 11.7$ Hz, 8H), 4.29 (t, $J = 6.6$ Hz, 4H), 1.89 (dd, $J = 14.6, 9.4$ Hz, 4H), 1.32 (s, 12H), 0.87 (s, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 140.91, 140.60, 137.76, 137.04, 136.71, 129.43, 129.17, 127.16, 125.54, 124.94, 124.47, 123.01, 122.39, 121.53, 120.62, 119.61, 111.95, 109.07, 108.94, 95.53, 85.94, 43.18, 31.45, 28.83, 26.85, 22.43, 13.89. HPLC analysis, 100% (silica column, $\lambda = 297$ nm, *i*-PrOH : hexane = 05 : 95, flow rate = 1.0 mL/min, $T_R = 3.462$ min.) MALDI-TOF MS m/z calcd. for $C_{52}H_{46}N_2O_2S$ (M^+): 762.33. Found: 762.51.

Synthesis of 4,7-bis((9-hexyl-9H-carbazol-3-yl)ethynyl)benzo[c][1,2,5]thiadiazole (10)
The same procedure as described for **3** was followed. Precursor **7** (0.26 g, 0.88 mmol), CuI (33 mg, 0.17 mmol), and Pd(PPh₃)₂Cl₂ catalyst (61mg, 0.08 mmol), Alkyne **4** (0.584 g, 2.12 mmol). The product was purified by column chromatography in hexane–Ethyl acetate (9: 1). Yield: 42 % (0.195 g, orange red solid). ¹H NMR (300 MHz, CDCl₃) δ 8.45 (s, 2H), 8.13 (d, $J = 7.7$ Hz, 2H), 7.84 – 7.75 (m, 4H), 7.54 – 7.39 (m, 6H), 7.29 (d, $J = 7.4$ Hz, 2H), 4.31 (t, $J = 7.2$ Hz, 4H), 1.87 (dd, $J = 14.5, 7.2$ Hz, 4H), 1.39 – 1.21 (m, 12H), 0.87 (t, $J = 6.8$ Hz, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 154.67, 140.93, 140.65, 132.09, 129.69, 126.28, 124.71, 122.99, 122.54, 120.70, 119.57, 117.22, 112.48, 109.04, 108.95, 99.39, 84.10, 43.21, 31.46, 28.84, 26.86, 22.44, 13.88. HPLC analysis, 100% (silica column, $\lambda = 301$ nm, *i*-PrOH : hexane = 05 : 95, flow rate = 1.0 mL/min, $T_R = 3.267$ min.) MALDI-TOF MS m/z calcd. for $C_{46}H_{42}N_4S$ ($M+1$): 682.31. Found: 683.34.

Synthesis of 2,5-bis(6'-(9-hexyl-9H-carbazol-3-yl)-3',4',5'-triphenyl-[1,1':2',1''-terphenyl]-4-yl)-1,3,4-oxadiazole (1).

Compound **9** (0.16 g, 0.2 mmol) and compound **11** (0.16 g, 0.4 mmol) were refluxed in diphenyl ether for 48 hours under nitrogen atmosphere. Reaction mixture was cooled and methanol was added. Resulting crude solid product was purified by column chromatography

using hexane–Ethyl acetate (9: 1) as eluent to get 77 mg off white solid in 25 % yield. ¹H NMR (500 MHz, CDCl₃) δ 7.74 (d, *J* = 7.7 Hz, 2H), 7.53 (s, 2H), 7.44 (d, *J* = 7.5 Hz, 2H), 7.33 (dd, *J* = 18.1, 9.5 Hz, 4H), 7.23 (d, *J* = 7.5 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 2H), 6.95 (d, *J* = 8.1 Hz, 4H), 6.89 – 6.69 (m, 44H), 4.05 (t, *J* = 7.2 Hz, 4H), 1.67 (brs, 4H), 1.16 (s, 12H), 0.74 (brs, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 164.05, 144.96, 141.07, 140.81, 140.74, 140.64, 140.53, 140.48, 140.28, 140.15, 140.08, 139.77, 138.53, 132.05, 131.96, 131.47, 131.38, 131.29, 130.53, 129.14, 126.74, 126.58, 126.49, 125.43, 125.22, 125.01, 123.33, 122.93, 121.59, 120.34, 119.97, 118.27, 108.46, 106.97, 42.90, 31.38, 28.69, 26.77, 22.44, 13.90. HPLC analysis, 100% (silica column, λ = 290 nm, *i*-PrOH : hexane = 05 : 95, flow rate = 1.0 mL/min, *T_R* = 3.453 min.) MALDI-TOF MS *m/z* calcd. for C₁₁₀H₈₈N₄O (M+1): 1480.70. Found: 1481.68. Elemental analysis: Calculated for C₁₁₀H₈₈N₄O: C 89.15; H 5.99; N 3.78; Found: C 89.39%; H 6.17%; N 3.54%.

Synthesis of 3,7-bis(4'-(9-hexyl-9H-carbazol-3-yl)-5',6'-diphenyl-[1,1':2',1''-terphenyl]-3'-yl)dibenzo[b,d]thiophene 5,5-dioxide (2).

50mg of compound **9** (0.25 g, 0.32 mmol) and **11** (0.302 g, 0.78 mmol) were refluxed in diphenyl ether for 48 hours under nitrogen atmosphere. Reaction mixture was cooled and methanol was added. Resulting crude solid product was purified by column chromatography using hexane–Ethyl acetate (9: 1) as eluent to get 170 mg of creamish white solid in 39 % yield. ¹H NMR (300 MHz, CDCl₃) δ 7.76 (d, *J* = 7.3 Hz, 2H), 7.51 (t, *J* = 7.5 Hz, 2H), 7.34 (dd, *J* = 17.0, 9.5 Hz, 4H), 7.24 – 7.03 (m, 6H), 6.94 – 6.70 (m, 46H), 4.10 (brs, 4H), 1.71 (brs, 24), 1.22 (brs, 12H), 0.81 (brs, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 143.19, 141.32, 141.15, 140.86, 140.68, 140.50, 140.35, 140.18, 139.87, 139.83, 138.64, 138.49, 138.39, 136.72, 136.63, 136.27, 131.50, 131.31, 131.23, 131.14, 130.88, 130.73, 130.25, 129.29, 129.16, 127.99, 127.20, 127.13, 126.76, 126.64, 126.51, 125.70, 125.63, 125.25, 125.21, 125.08, 124.91, 124.75, 123.59, 123.10, 122.84, 121.91, 121.56, 120.46, 119.85, 119.42, 118.59, 118.19, 108.56, 108.25, 107.64, 106.95, 42.95, 31.44, 28.63, 26.78, 22.50, 14.00. HPLC analysis, 100% (silica column, λ = 291 nm, *i*-PrOH : hexane = 05 : 95, flow rate = 1.0 mL/min, *T_R* = 3.414 min.) MALDI-TOF MS *m/z* calcd. for C₁₀₈H₈₆N₅O₂S (M+1): 1474.64. Found: 1475.58. Elemental analysis: Calculated for C₁₀₈H₈₆N₅O₂S : C 87.89; H 5.87; N 1.90; S 2.17; Found: C 87.60%; H 5.98%; N 1.76%; S 2.05%.

Synthesis of 4,7-bis(4'-(9-hexyl-9H-carbazol-3-yl)-5',6'-diphenyl-[1,1':2',1''-terphenyl]-3'-yl)benzo[c][1,2,5]thiadiazole (3).

50mg of compound **10** (0.22 g, 0.322 mmol) and **11** (0.247g, 0.644 mmol) were refluxed in diphenyl ether for 48 hours under nitrogen atmosphere. Reaction mixture was cooled and methanol was added. Resulting crude solid product was purified by column chromatography using hexane–Ethyl acetate (9: 1) as eluent to get 166 mg of light green solid in 37 % yield. ¹H NMR (300 MHz, CDCl₃) δ 7.85 (dd, *J* = 18.6, 7.5 Hz, 2H), 7.77 – 7.48 (m, 6H), 7.41 – 7.31 (m, 4H), 7.21 – 7.02 (m, 4H), 6.94 – 6.53 (m, 40H), 4.14 (brs, 4H), 1.73 (brs, 4H), 1.24 (brs, 12H), 0.85 (brs, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 153.70, 141.77, 141.58, 140.91, 140.83, 140.68, 140.55, 140.29, 140.25, 139.98, 139.57, 138.58, 135.55, 132.80, 131.44, 131.34, 131.27, 131.15, 131.01, 130.84, 130.04, 129.94, 129.84, 128.48, 128.25, 126.47, 126.34, 126.20, 126.15, 125.60, 125.38, 125.21, 125.02, 124.91, 124.71, 124.59, 123.34, 122.48, 122.26, 121.62, 120.25, 118.37, 118.29, 108.43, 106.70, 106.39, 42.91, 31.56, 28.82, 26.85, 22.51, 14.00. HPLC analysis, 100% (silica column, λ = 298 nm, *i*-PrOH : hexane = 05 : 95, flow rate = 1.0 mL/min, *T_R* = 3.265 min.). MALDI-TOF MS *m/z* calcd. for C₁₀₂H₈₂N₄S (M+1): 1394.63. Found: 1395.64. Elemental analysis: Calculated for C₁₀₂H₈₂N₄S: C 87.77; H 5.92; N 4.01; S 2.30; Found: C 87.66%; H 5.81%; N 3.88%; S 2.19%.

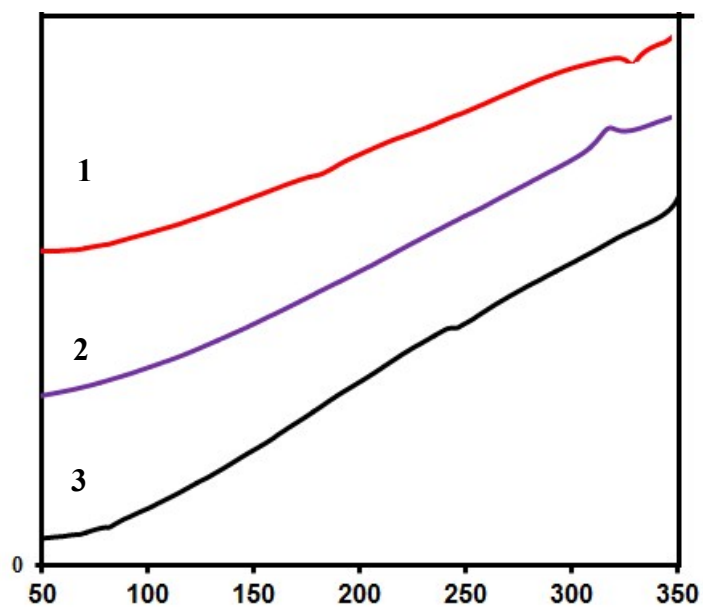


Figure S1. DSC measurements of derivative **1**, **2** and **3** recorded at $10^{\circ}\text{Cmin}^{-1}$

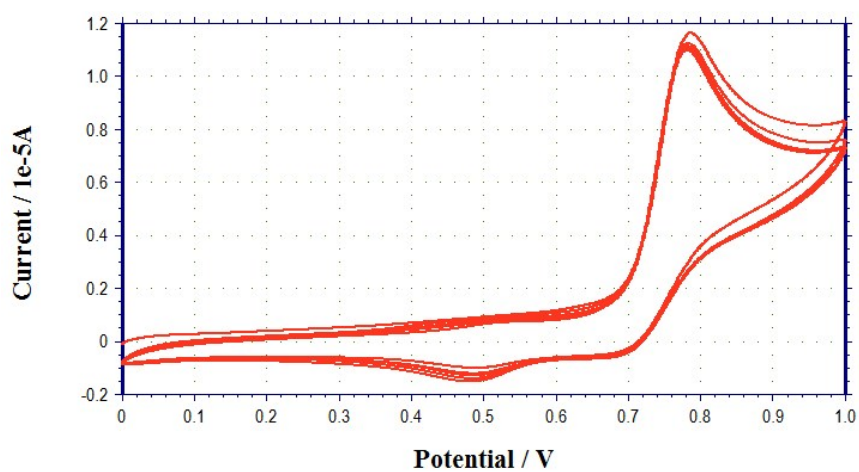


Figure S2. Repeated Cyclic voltammograms of **1** (5 cycles) at scan rate of 50 mV/s in DCM

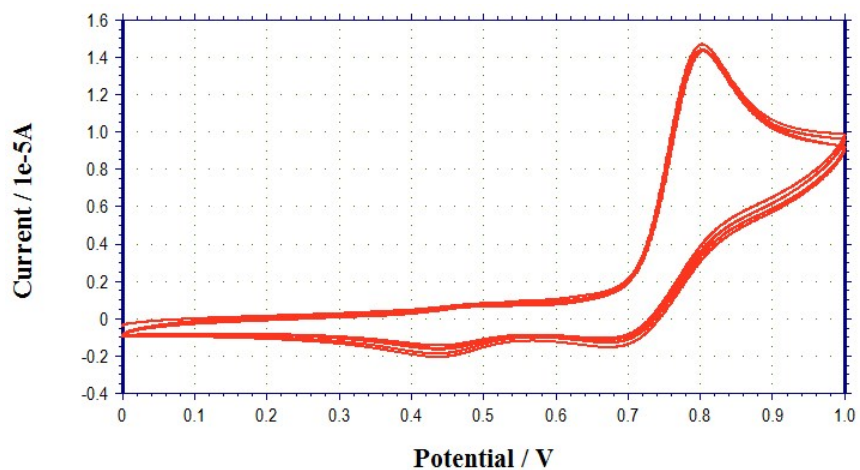


Figure S3. Repeated Cyclic voltammograms of **2** (5 cycles) at scan rate of 50 mV/s in DCM

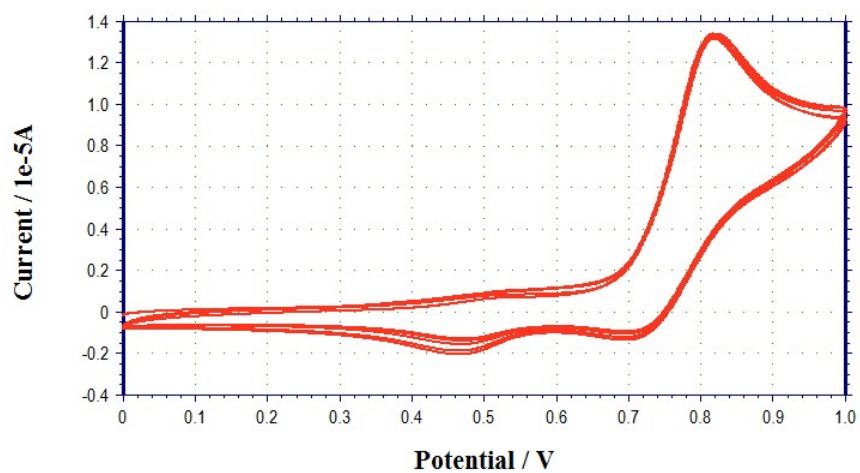


Figure S4. Repeated Cyclic voltammograms of **3** (5 cycles) at scan rate of 50 mV/s in DCM

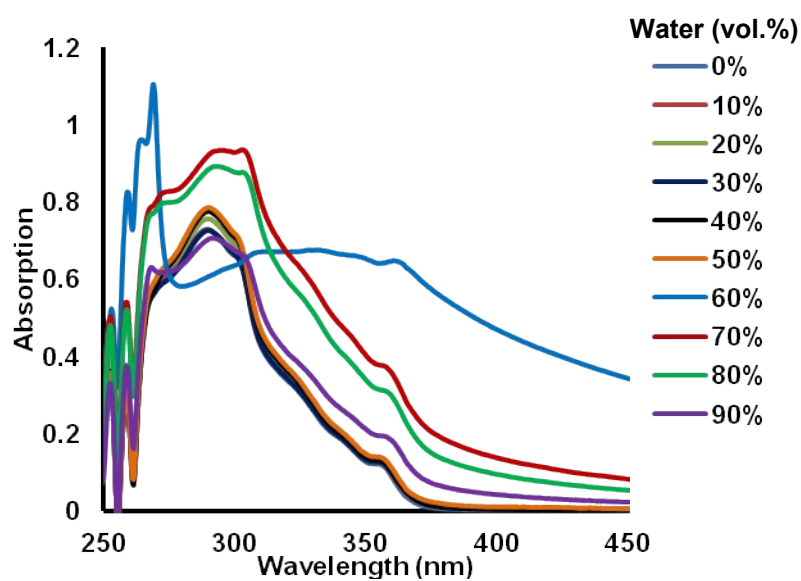


Figure S5. UV-visible spectrum of derivative **1** in different fractions of THF/Water [conc. - $10\mu\text{M}$, $\lambda_{\text{ex}} = 355\text{ nm}$]

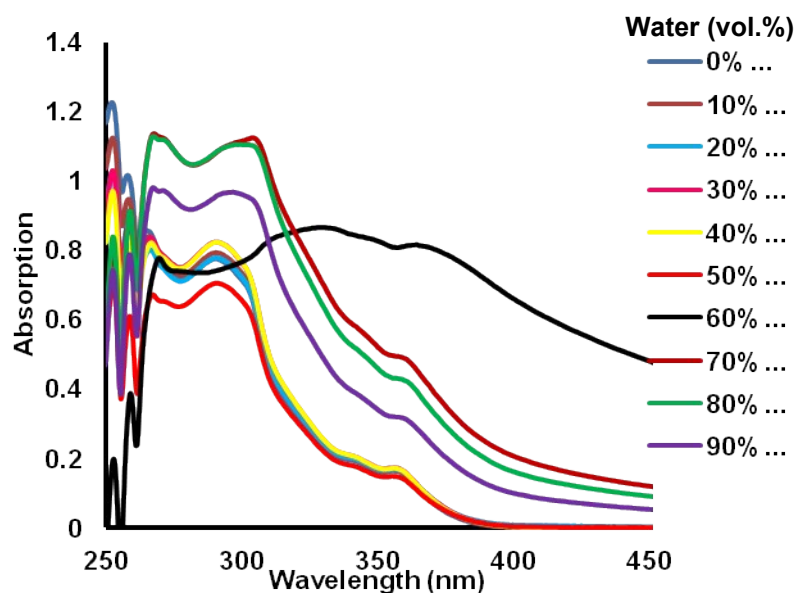


Figure S6. UV-visible spectrum of derivative **2** in different fractions of THF/Water [conc. - $10\mu\text{M}$, $\lambda_{\text{ex}} = 355\text{ nm}$]

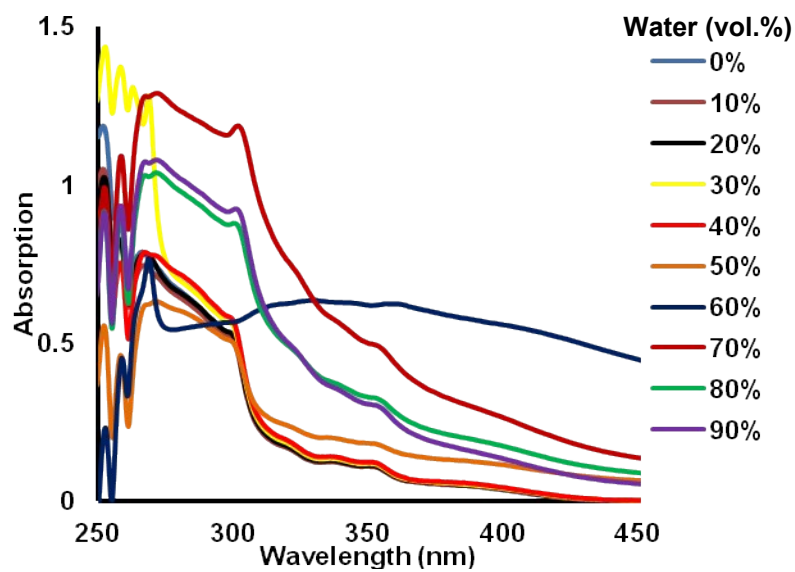


Figure S7. UV-visible spectrum of derivative **3** in different fractions of THF/Water [conc. - 10 μ M, λ_{ex} = 352 nm]

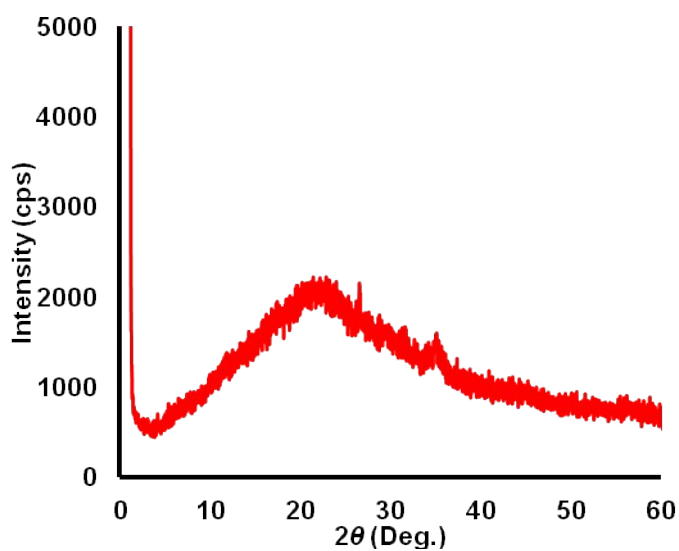
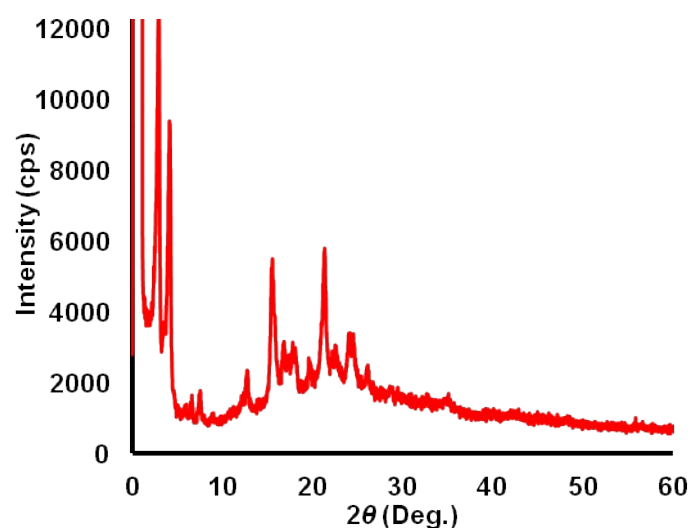


Figure S8. Powder XRD patterns of derivative **1**



FigureS 9. Powder XRD patterns of derivative 2

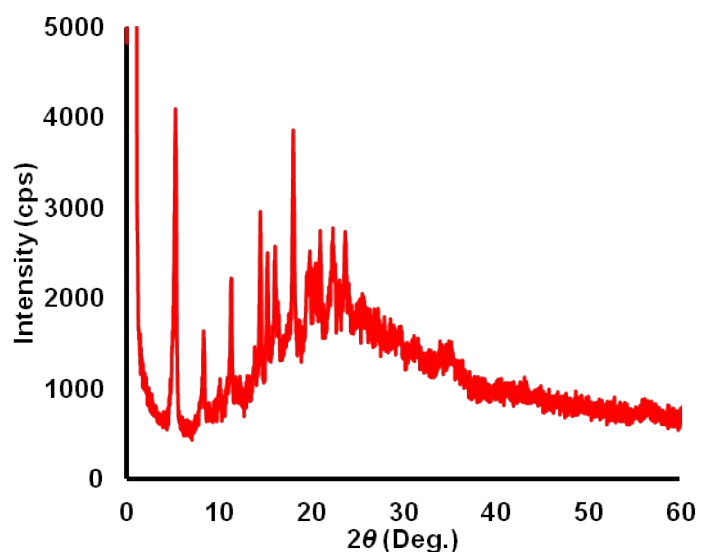


Figure S10. Powder XRD patterns of derivative 3

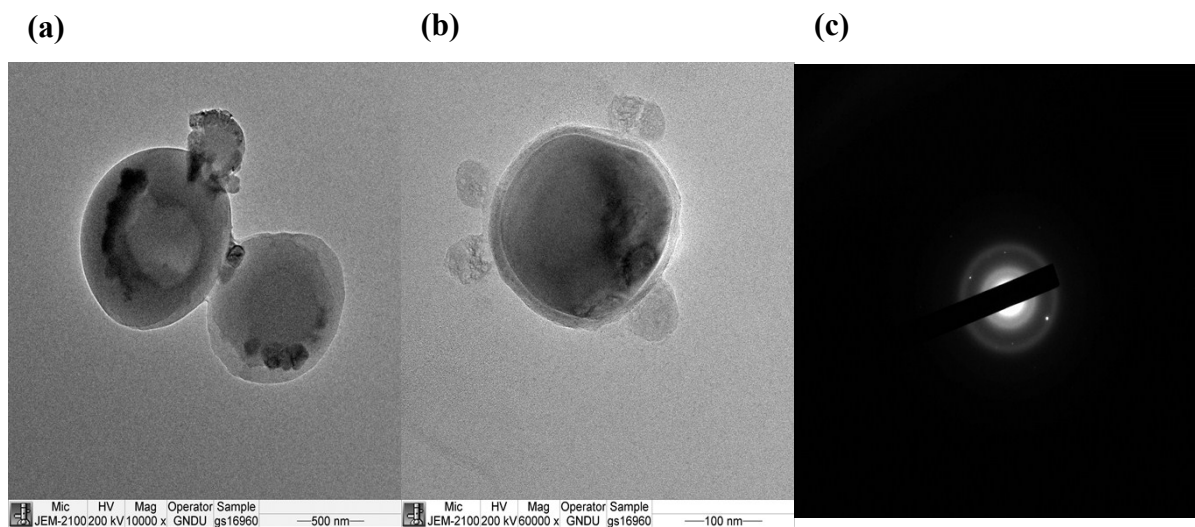


Figure S11. TEM images (a and b) and ED spectra (c) of derivative **1** in 60% water

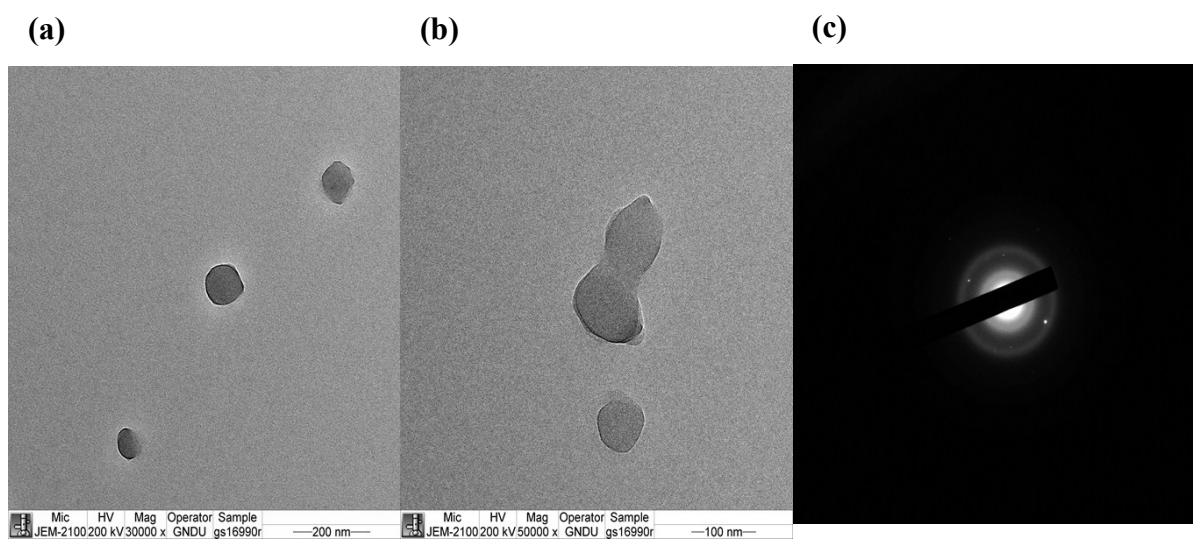


Figure S12. TEM images (a and b) and ED spectra (c) of derivative **1** in 90% water

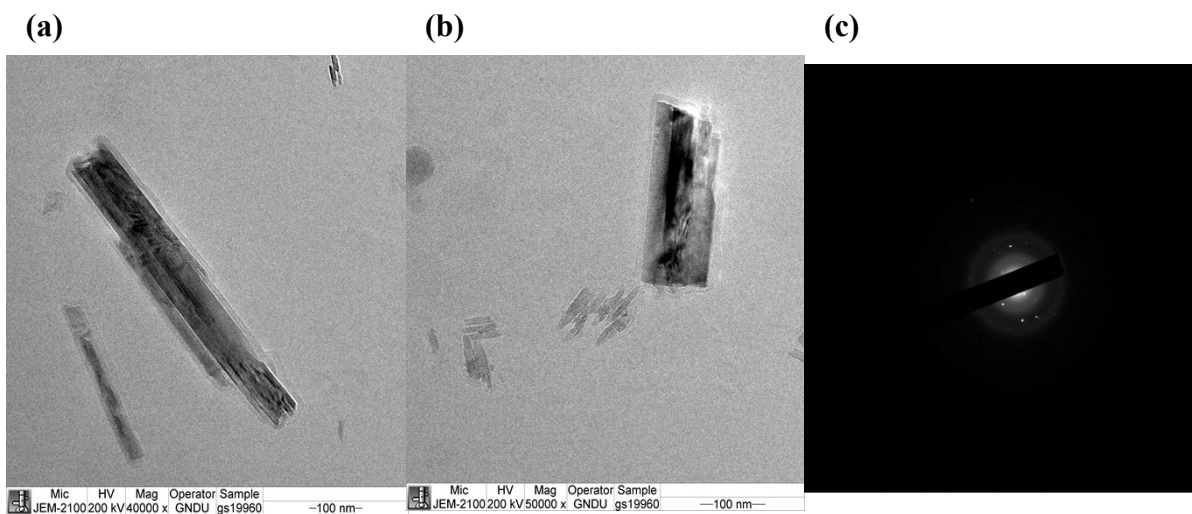


Figure S13. TEM images (a and b) and ED spectra (c) of derivative 2 in 60% water

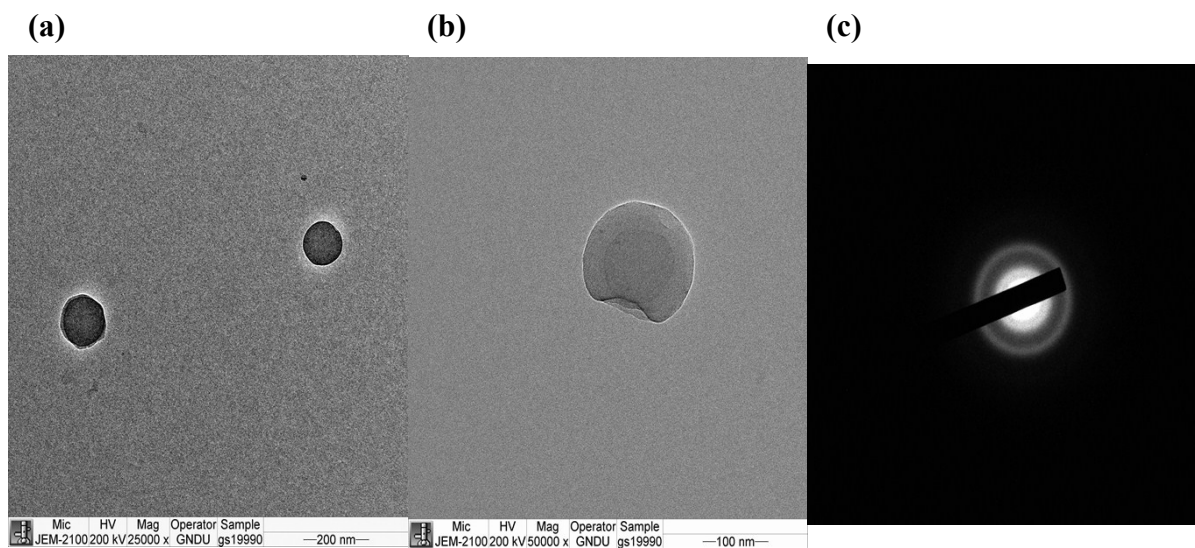


Figure S14. TEM images (a and) and ED spectra (c) of derivative 2 in 90% water

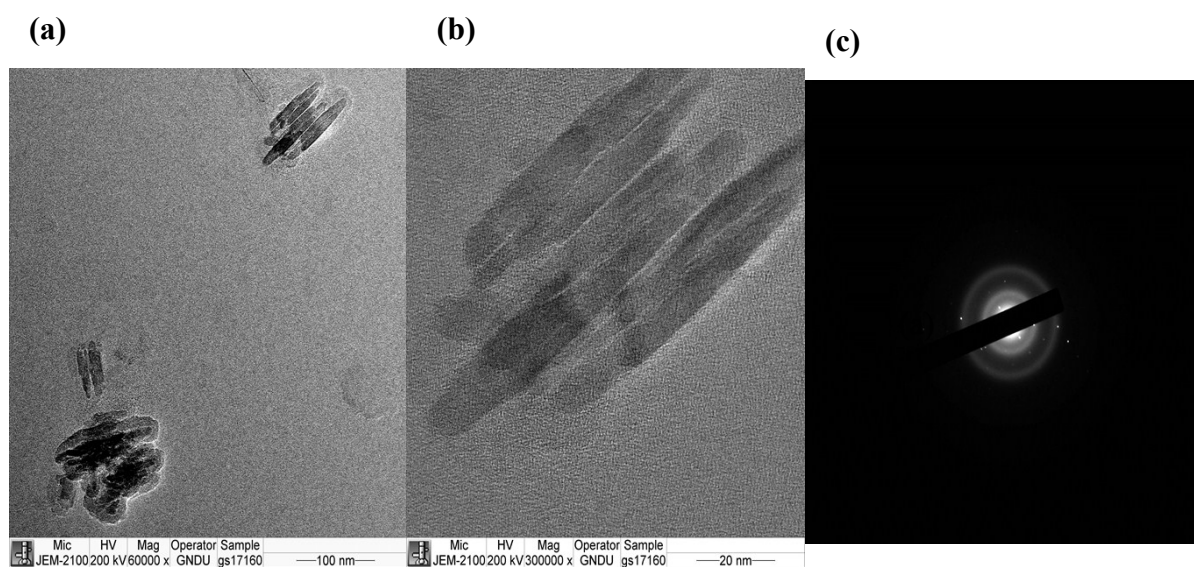


Figure S15. TEM images (a and b) and ED spectra (c) of derivative **3** in 60% water

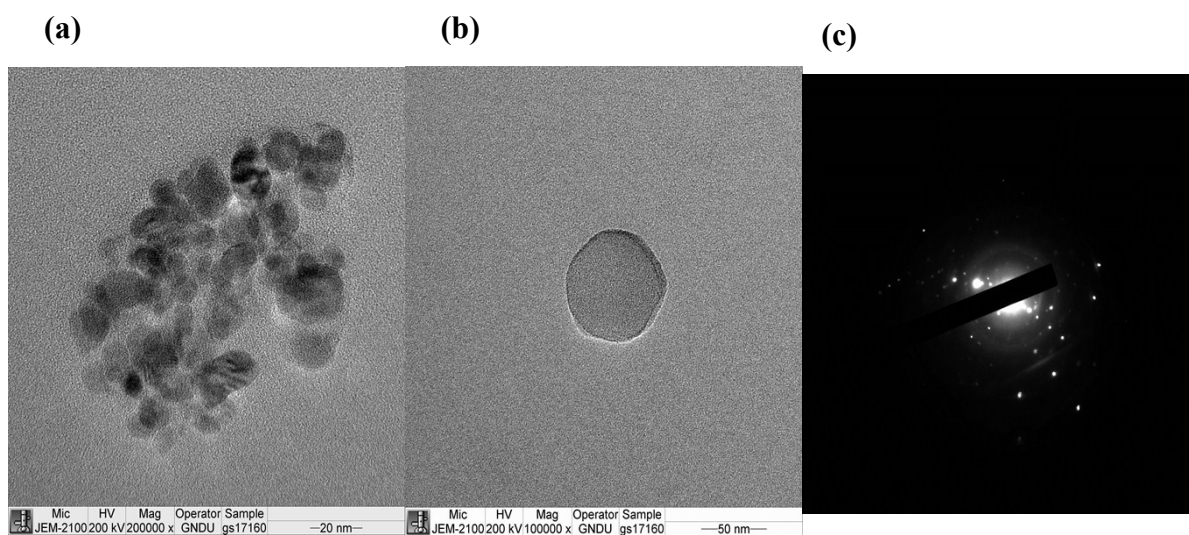


Figure S16. TEM images (a and b) and ED spectra (c) of derivative **3** in 90% water

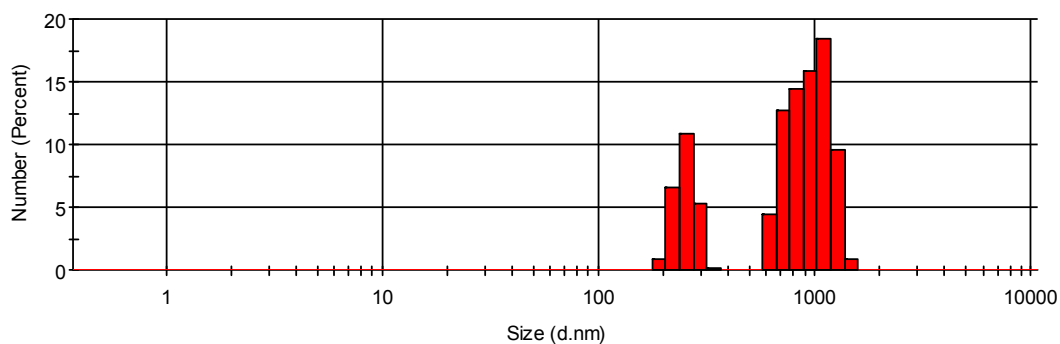


Figure S17. Differential Light Scattering (DLS) results of derivative **1** showing particle size diameter in 90% water.

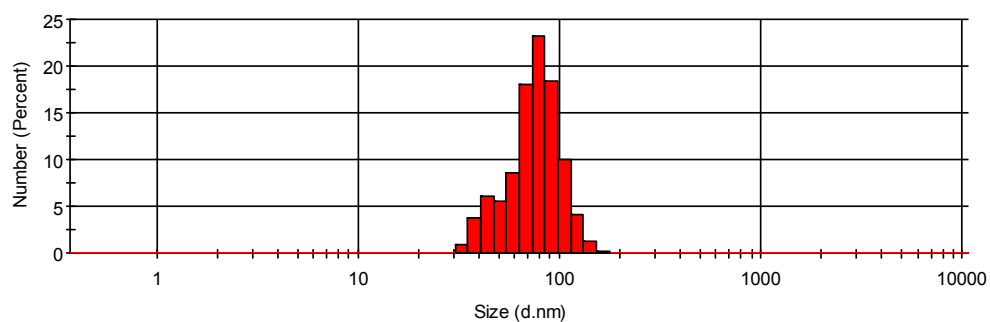


Figure S18. Differential Light Scattering (DLS) results of derivative **1** showing particle size diameter in 90% water.

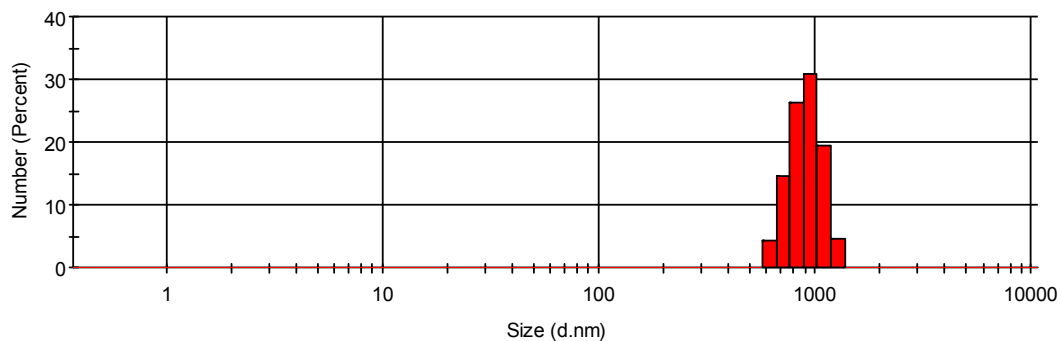


Figure S19. Differential Light Scattering (DLS) results of derivative **2** showing particle size diameter in 90% water.

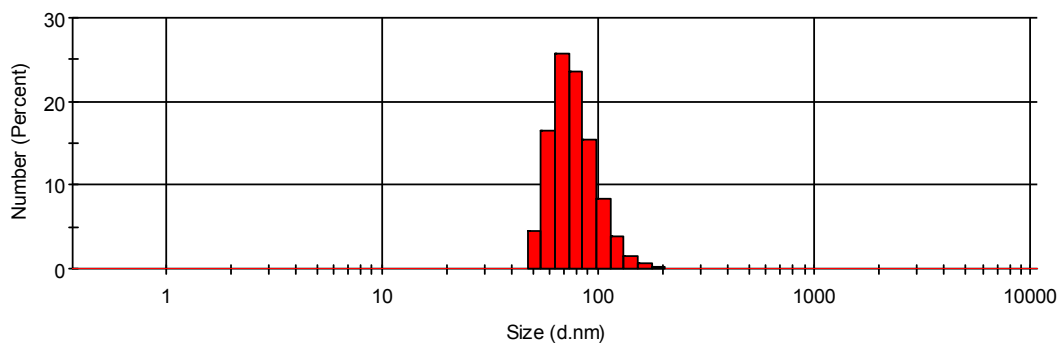


Figure S20. Differential Light Scattering (DLS) results of derivative **2** showing particle size diameter in 90% water.

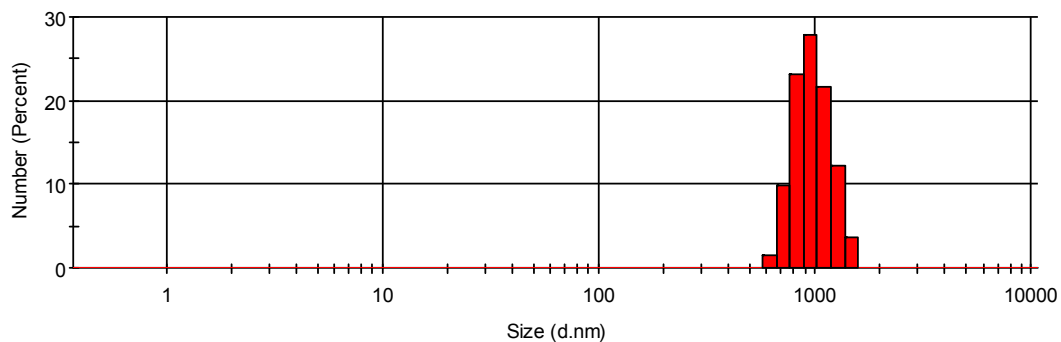


Figure S21. Differential Light Scattering (DLS) results of derivative **3** showing particle size diameter in 90% water.

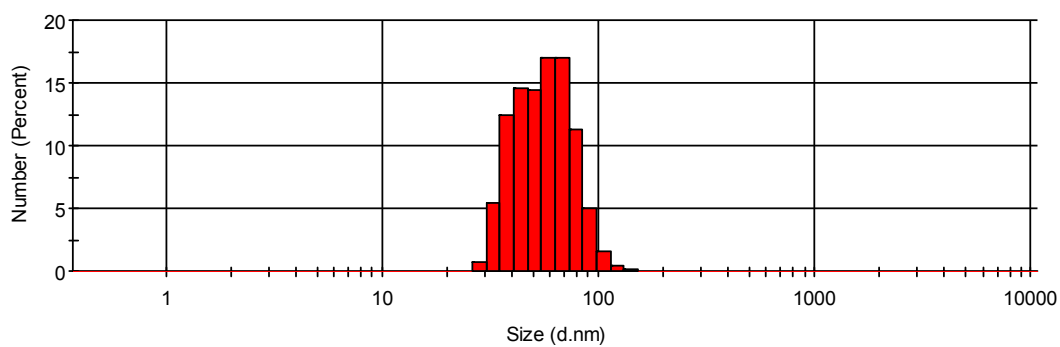


Figure S22. Differential Light Scattering (DLS) results of derivative **3** showing particle size diameter in 90% water.

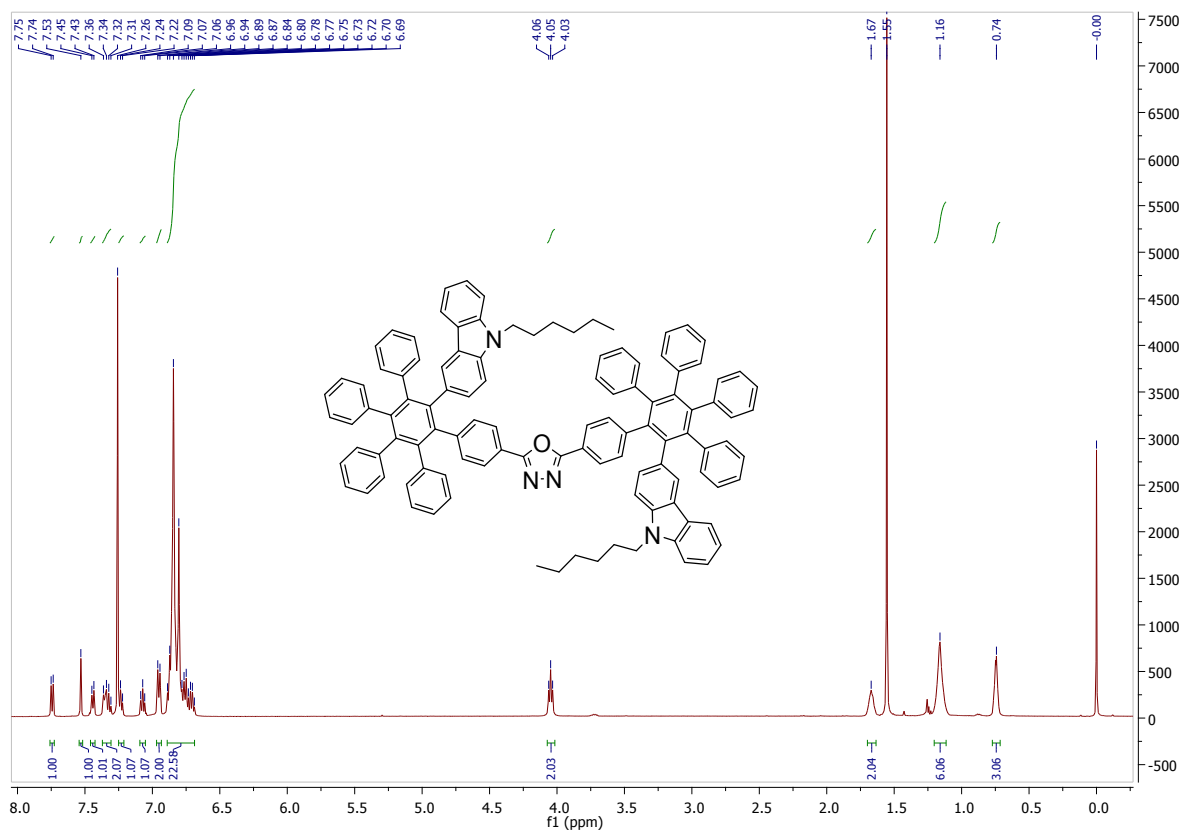


Figure S23. ¹H NMR of derivative 1 (CDCl₃, 300 MHz)

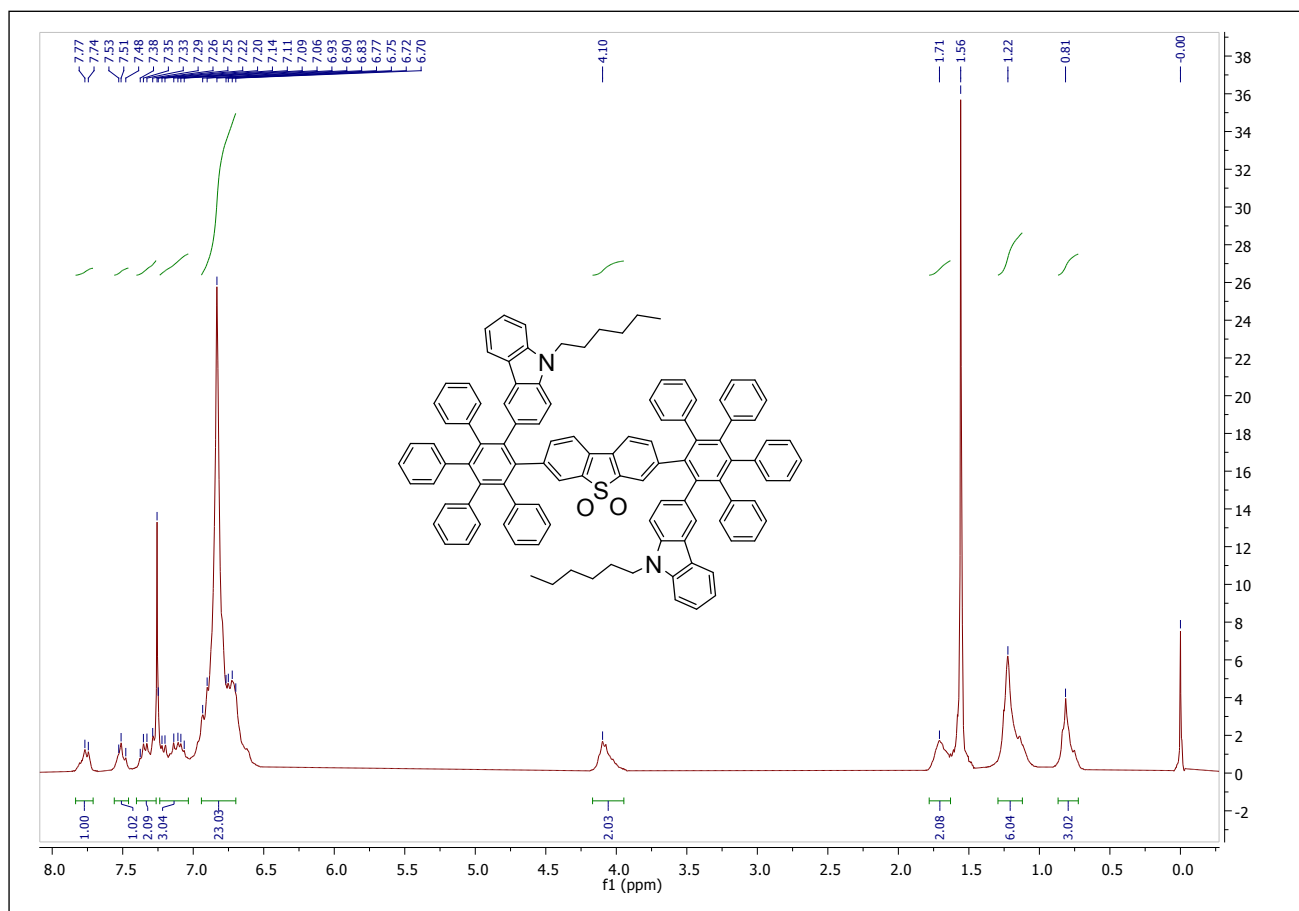


Figure S24. ^1H NMR of derivative 2 (CDCl₃, 300 MHz)

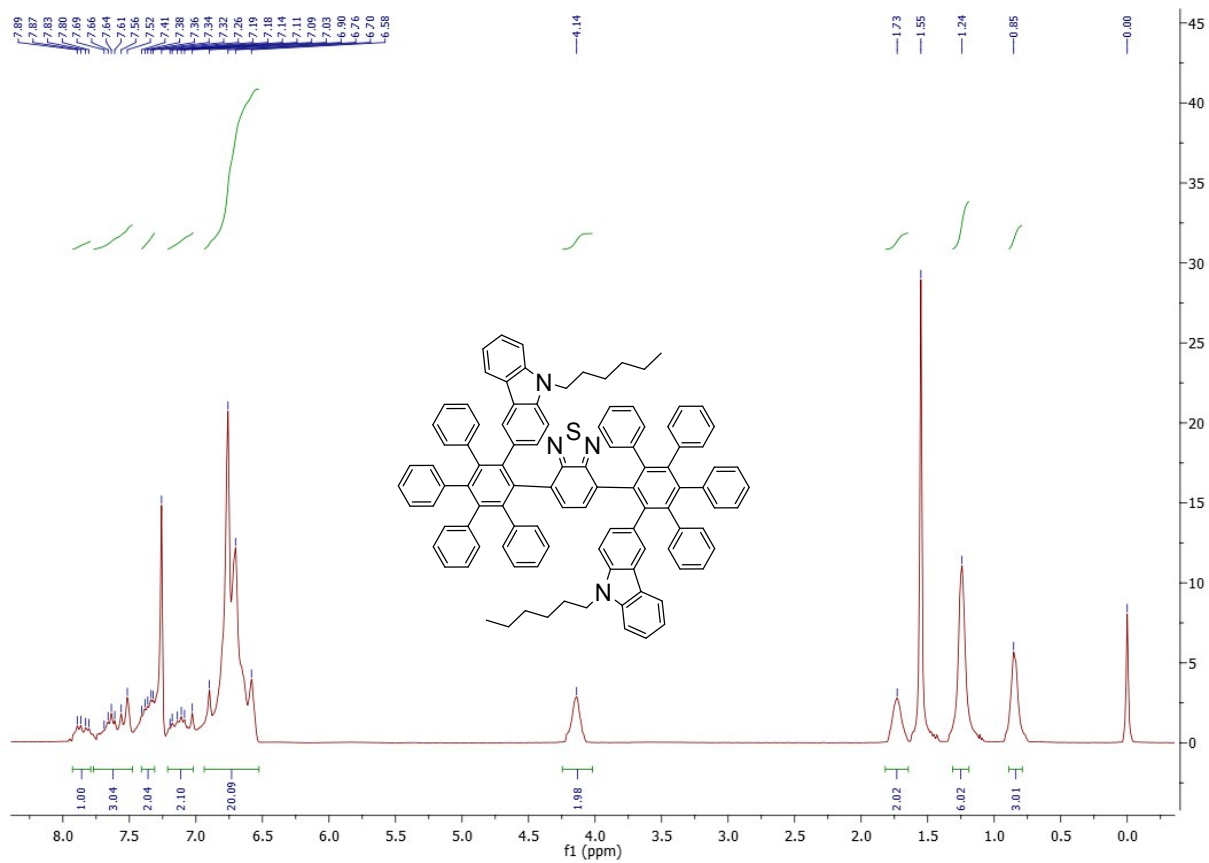


Figure S25. ^1H NMR of derivative **3** (CDCl_3 , 300 MHz)

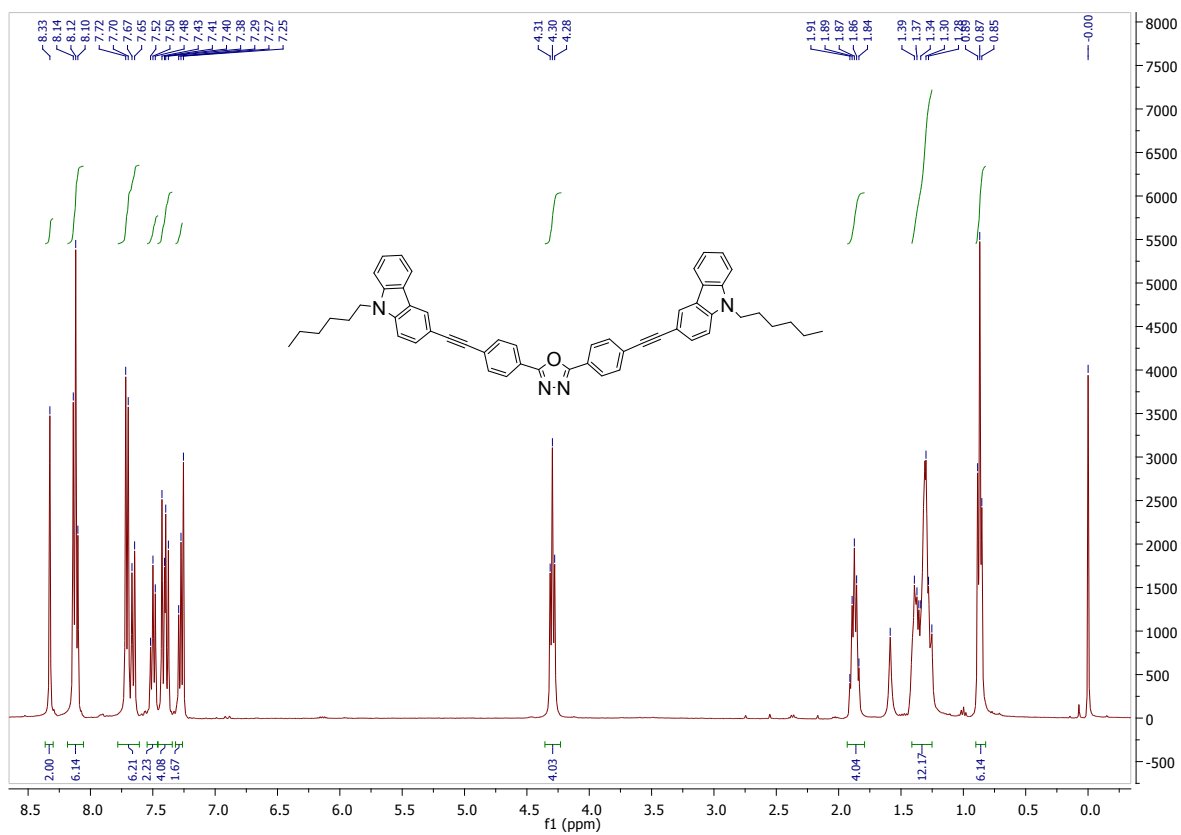


Figure S26. ¹H NMR of derivative 8 (CDCl₃, 400 MHz)

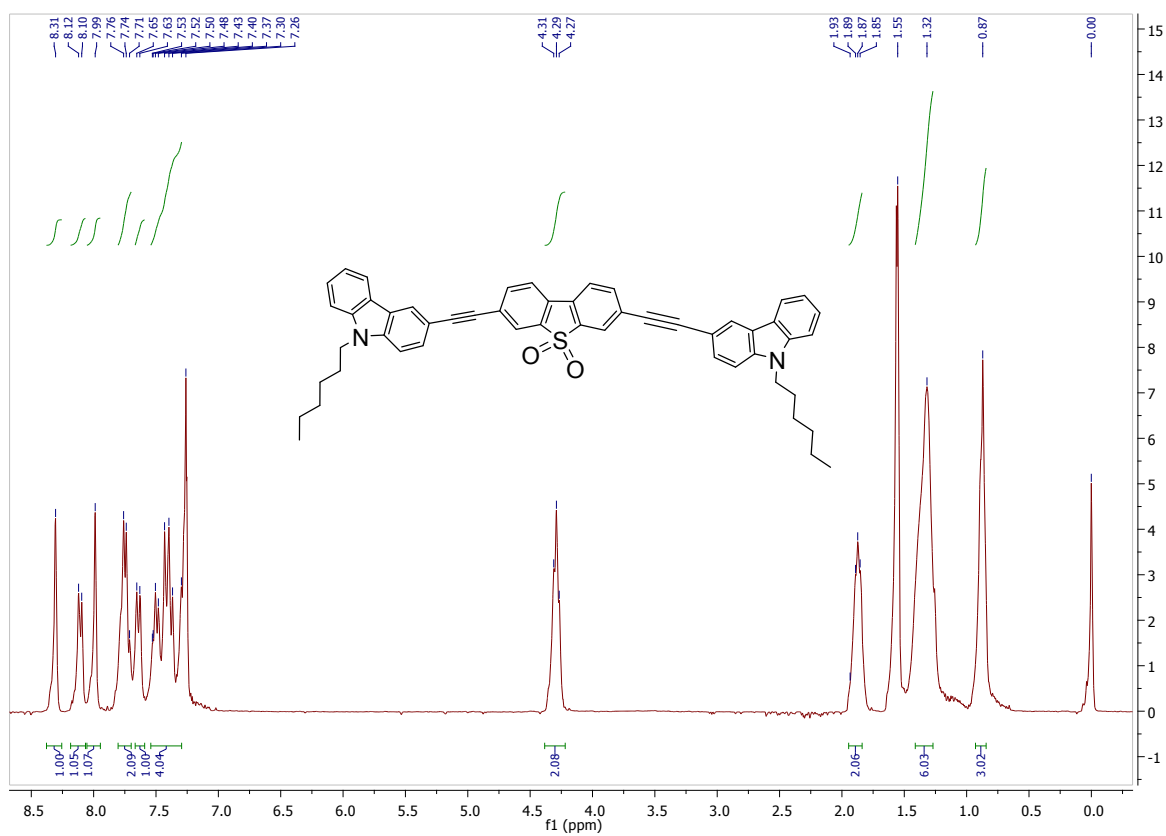


Figure S27. ^1H NMR of derivative 9 (CDCl_3 , 300 MHz)

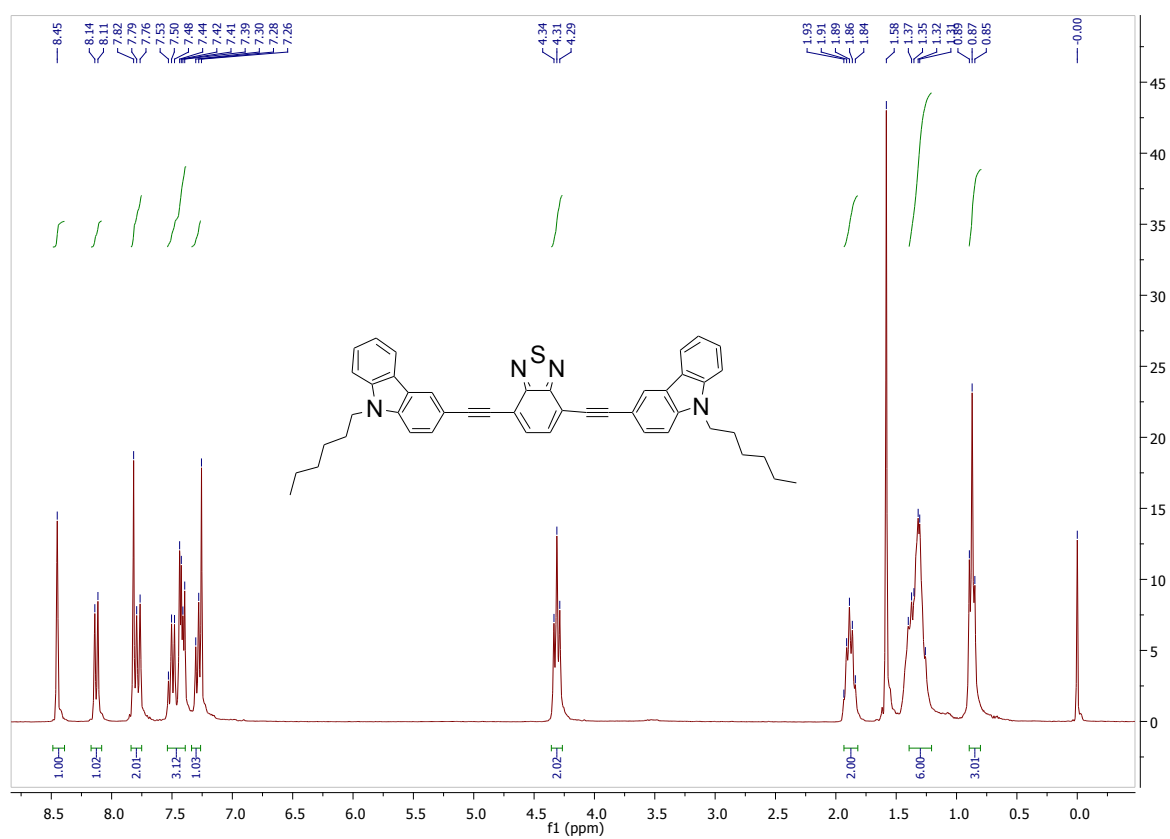


Figure S28. ¹H NMR of derivative 10(CDCl₃, 300 MHz)

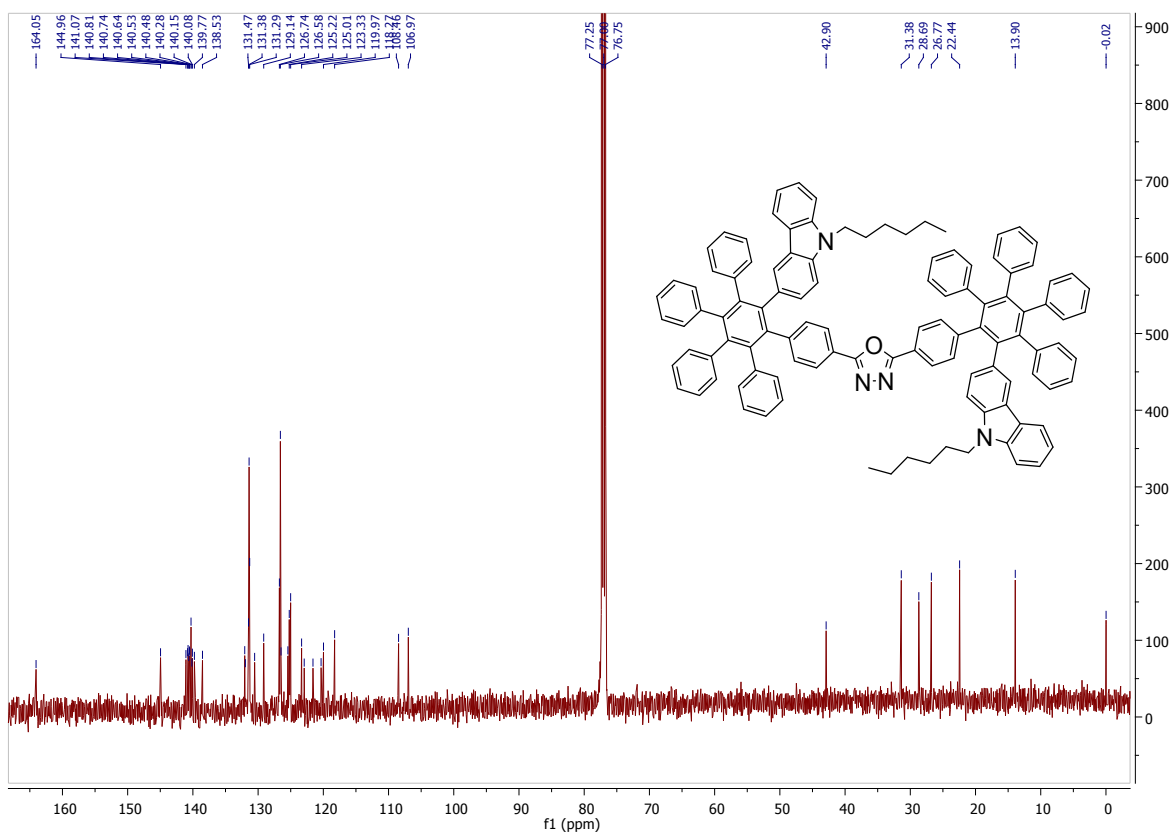


Figure S29. ^1H NMR of derivative 1 (CDCl_3 , 126 MHz)

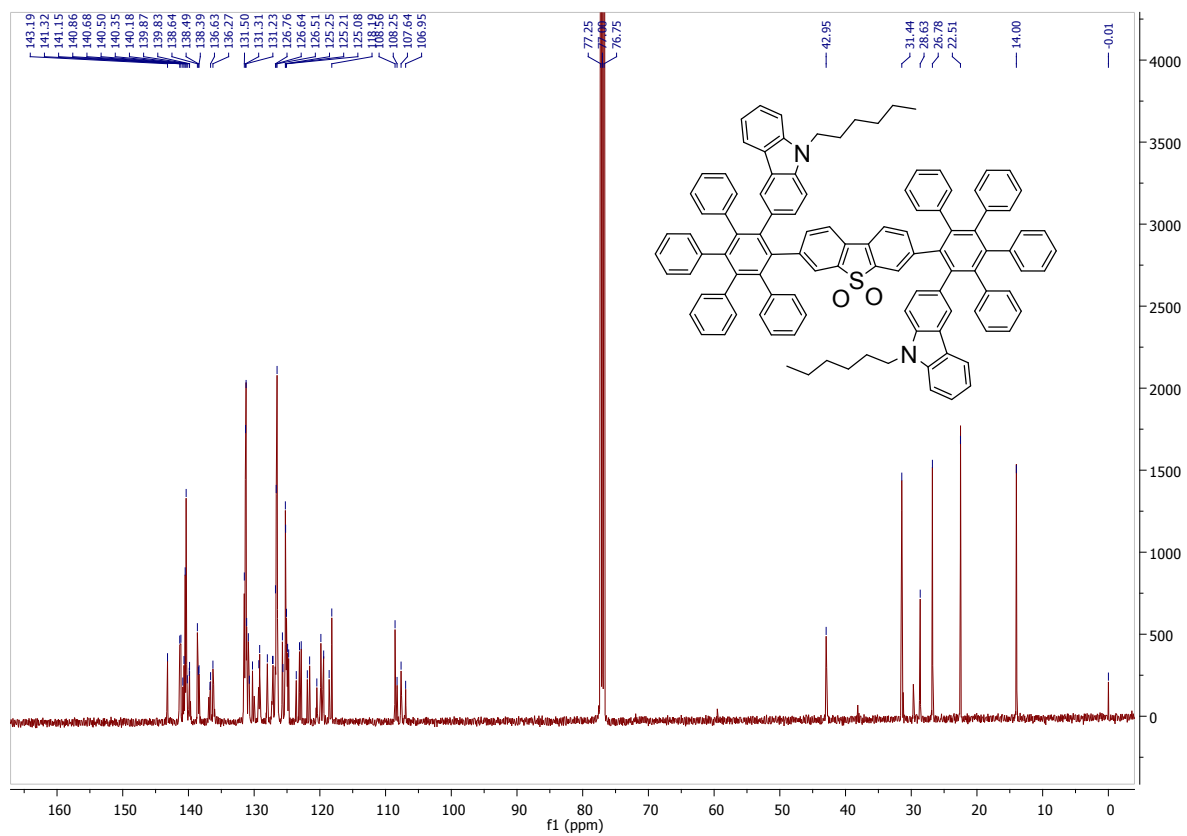


Figure S30. ^1H NMR of derivative **2** (CDCl_3 , 126 MHz)

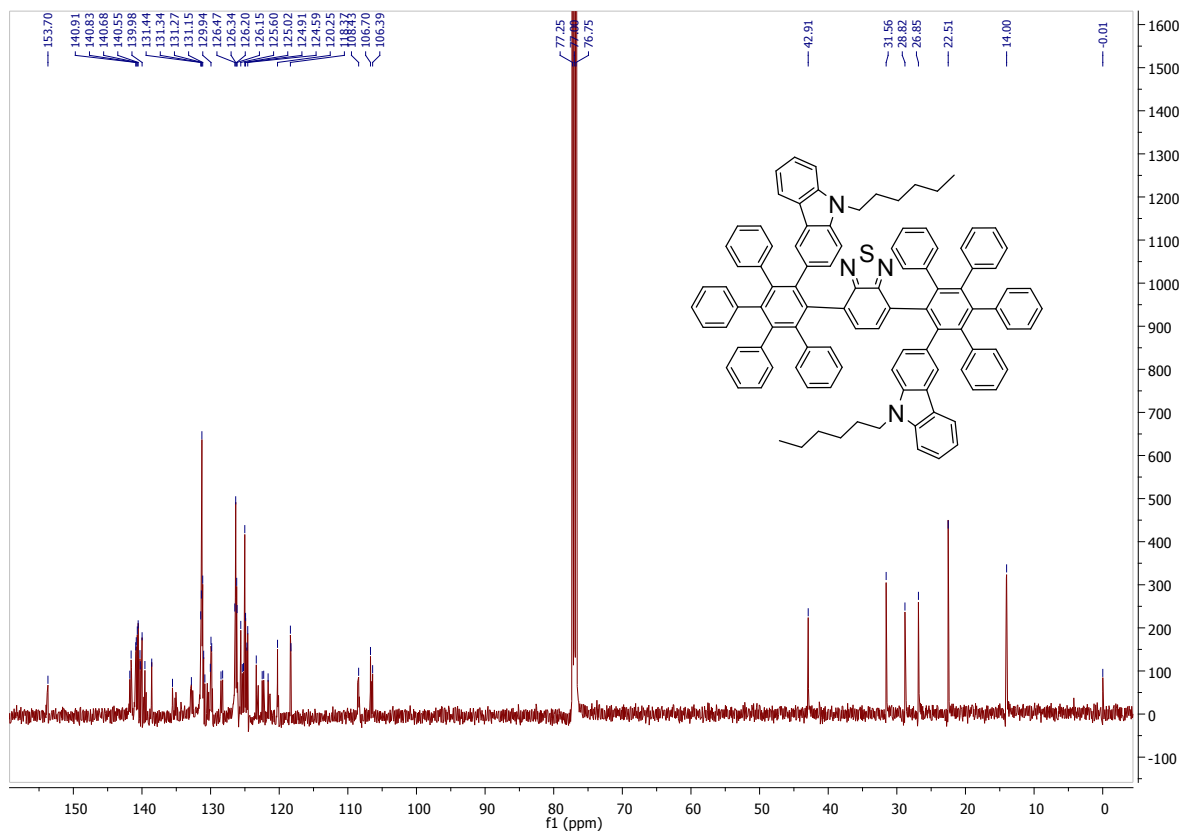


Figure S31. ^1H NMR of derivative 3 (CDCl_3 , 126 MHz)

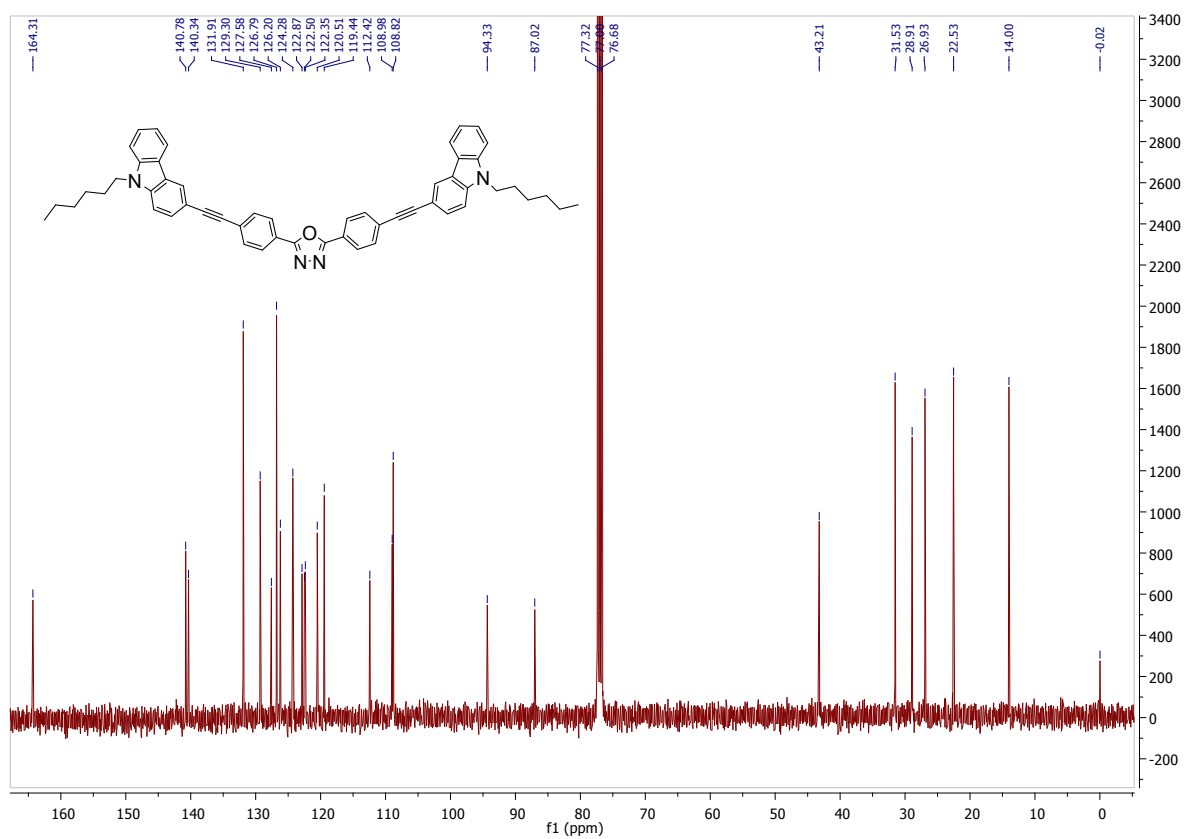


Figure S32. ^1H NMR of derivative **8** (CDCl_3 , 101 MHz)

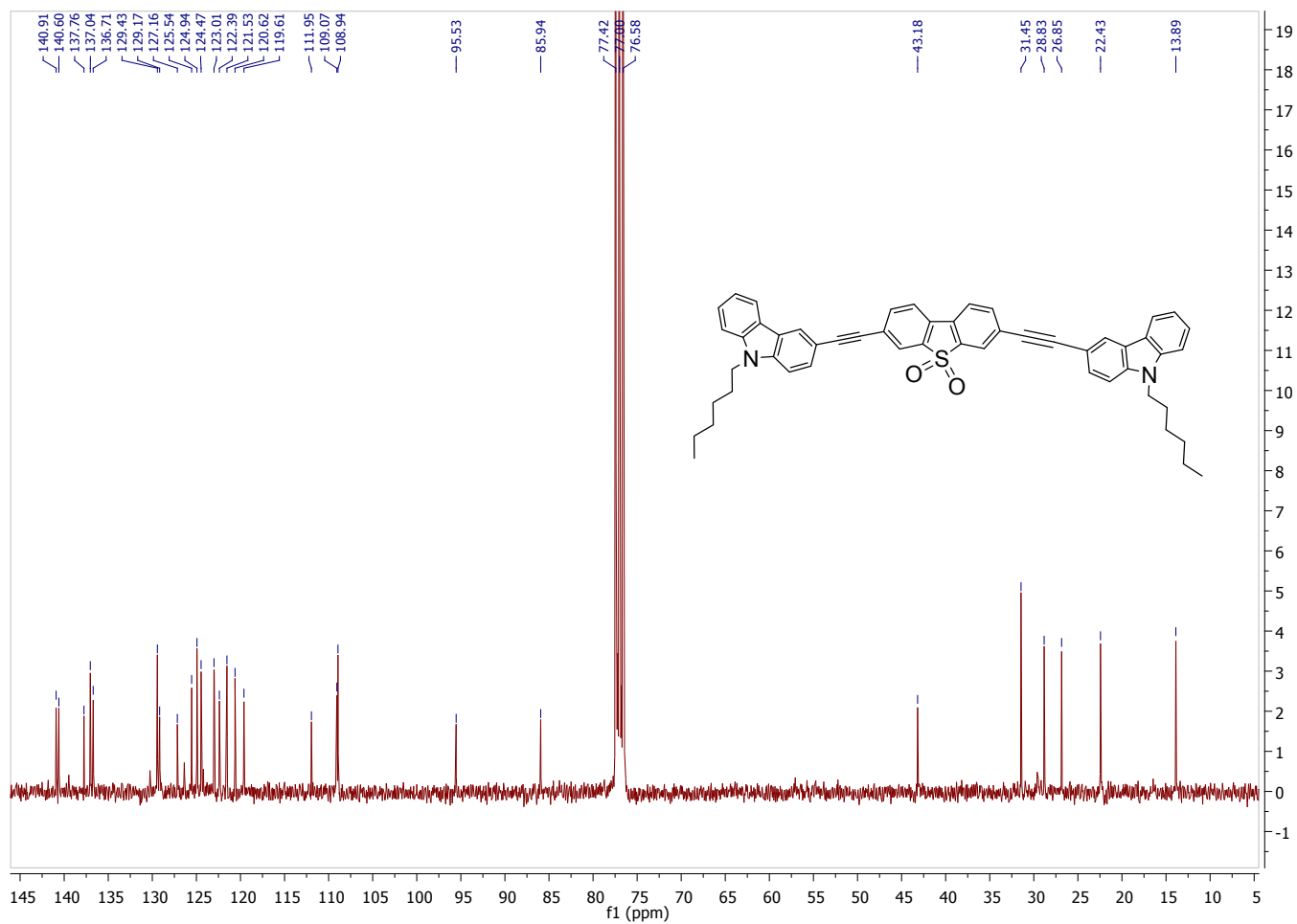


Figure S33. ^1H NMR of derivative 9(CDCl_3 , 75 MHz)

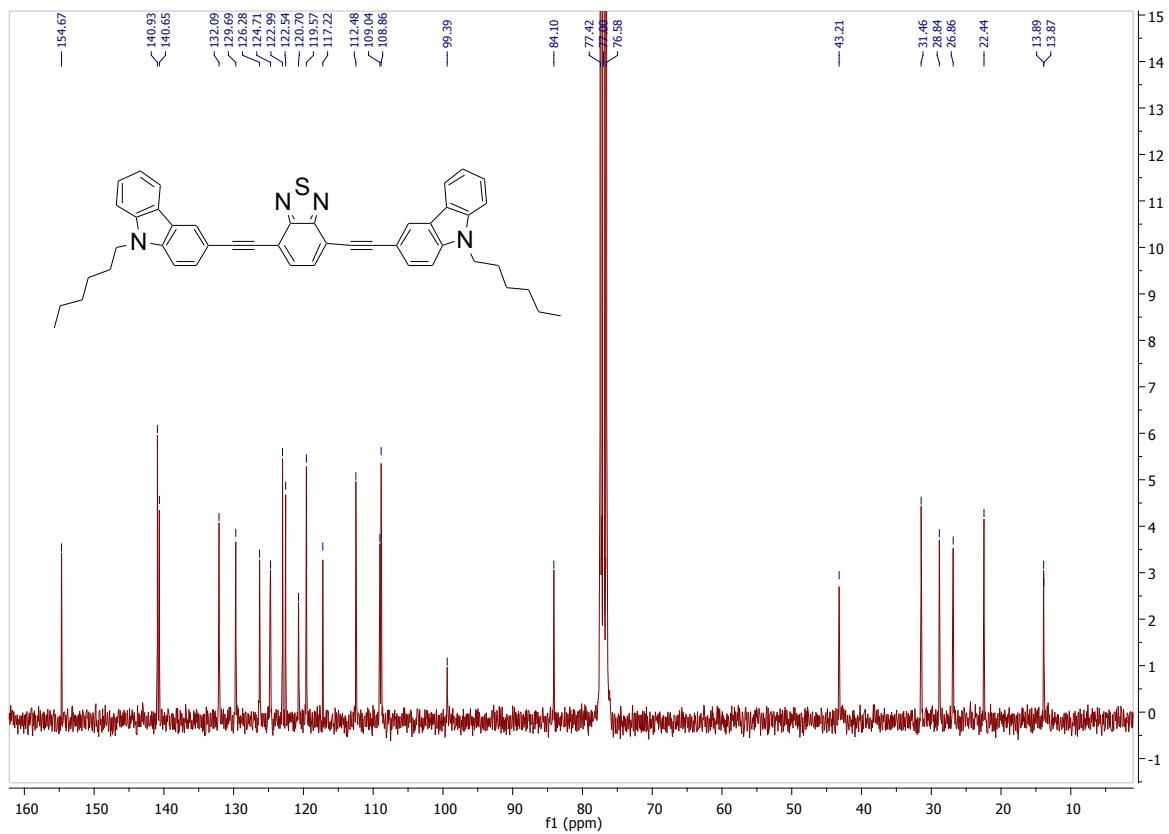


Figure S34. ¹H NMR of derivative **10** (CDCl₃, 75 MHz)

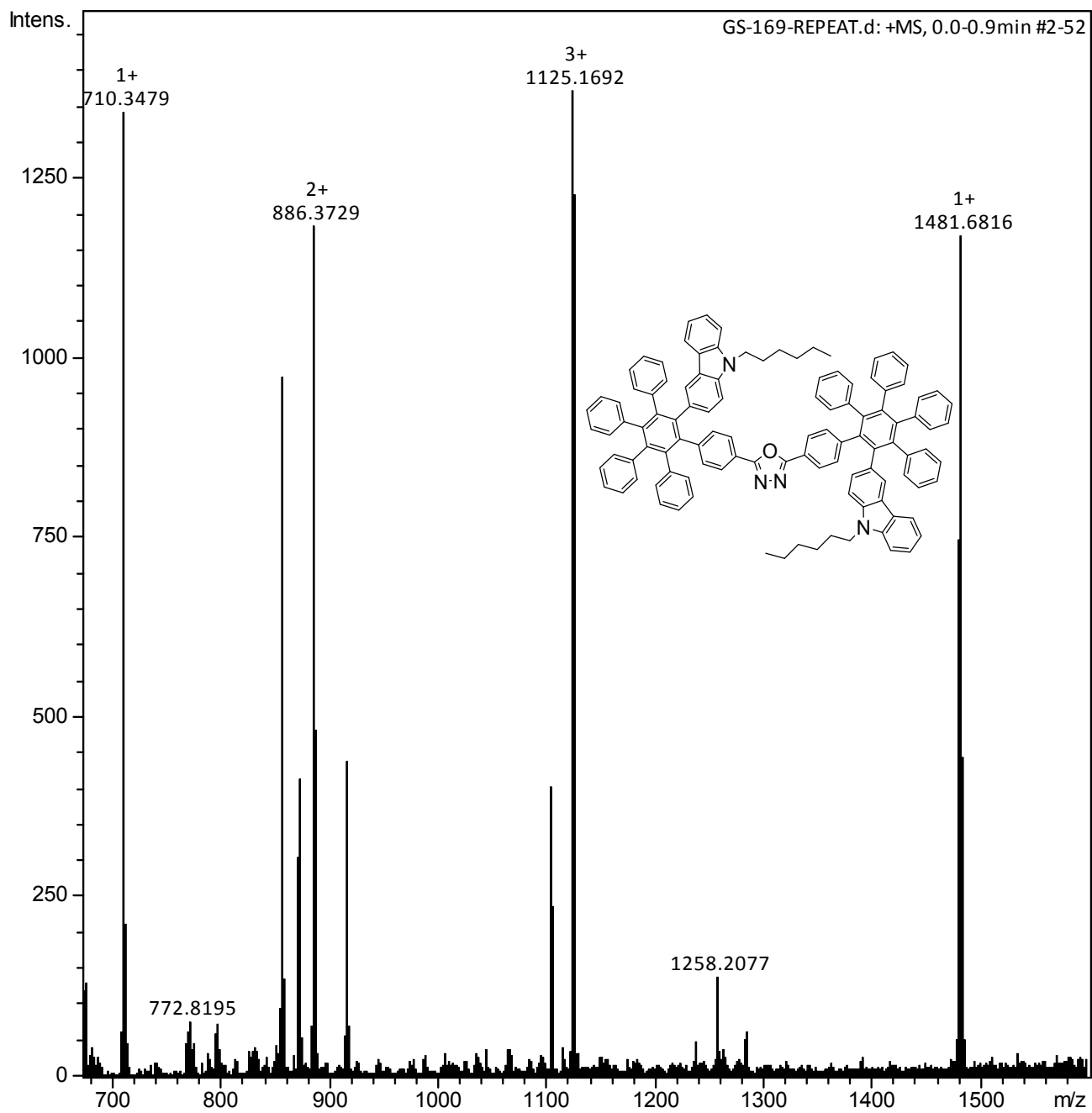


Figure S35. Mass spectrum of derivative 1

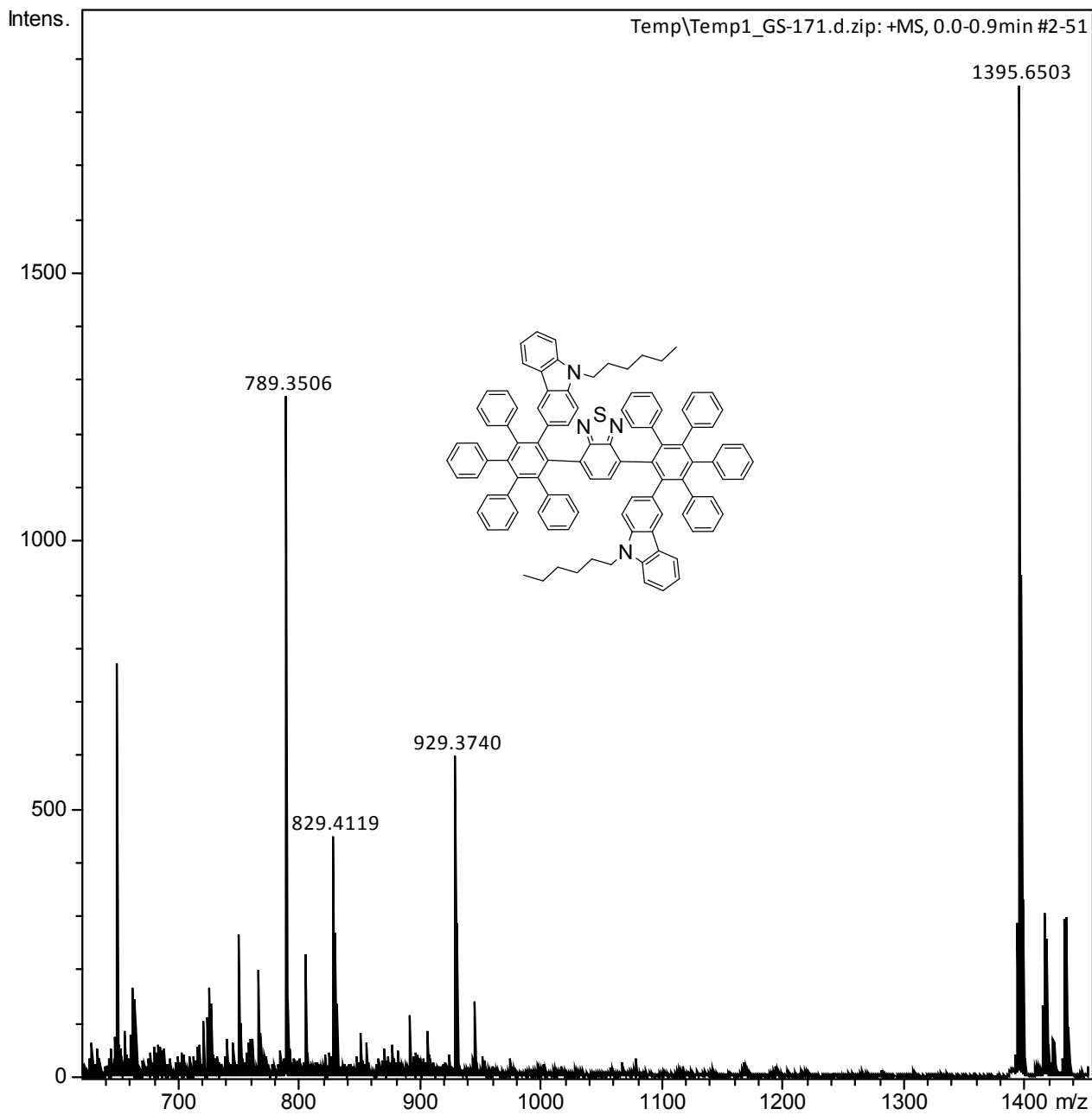


Figure S37. Mass spectrum of derivative **3**

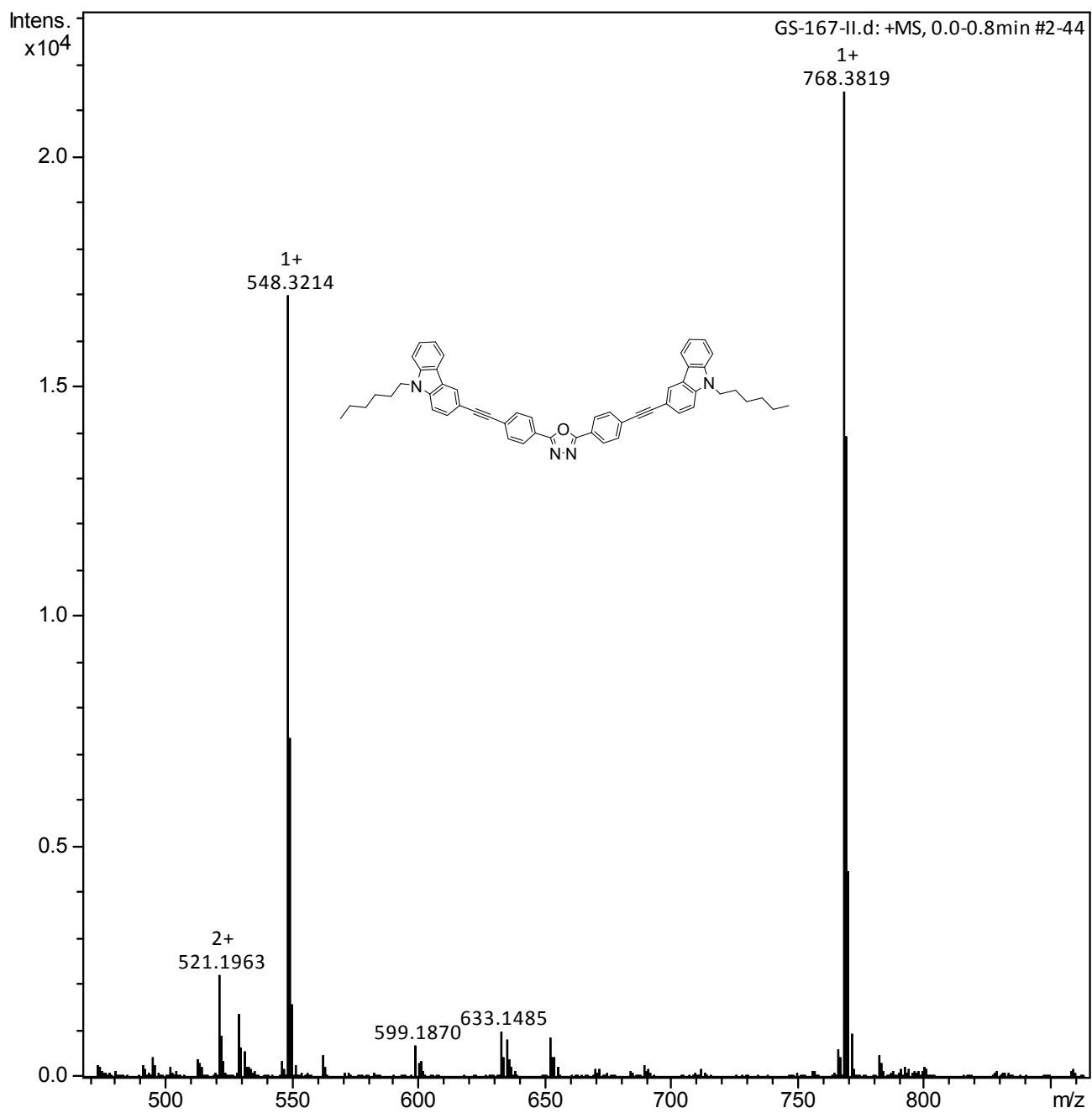
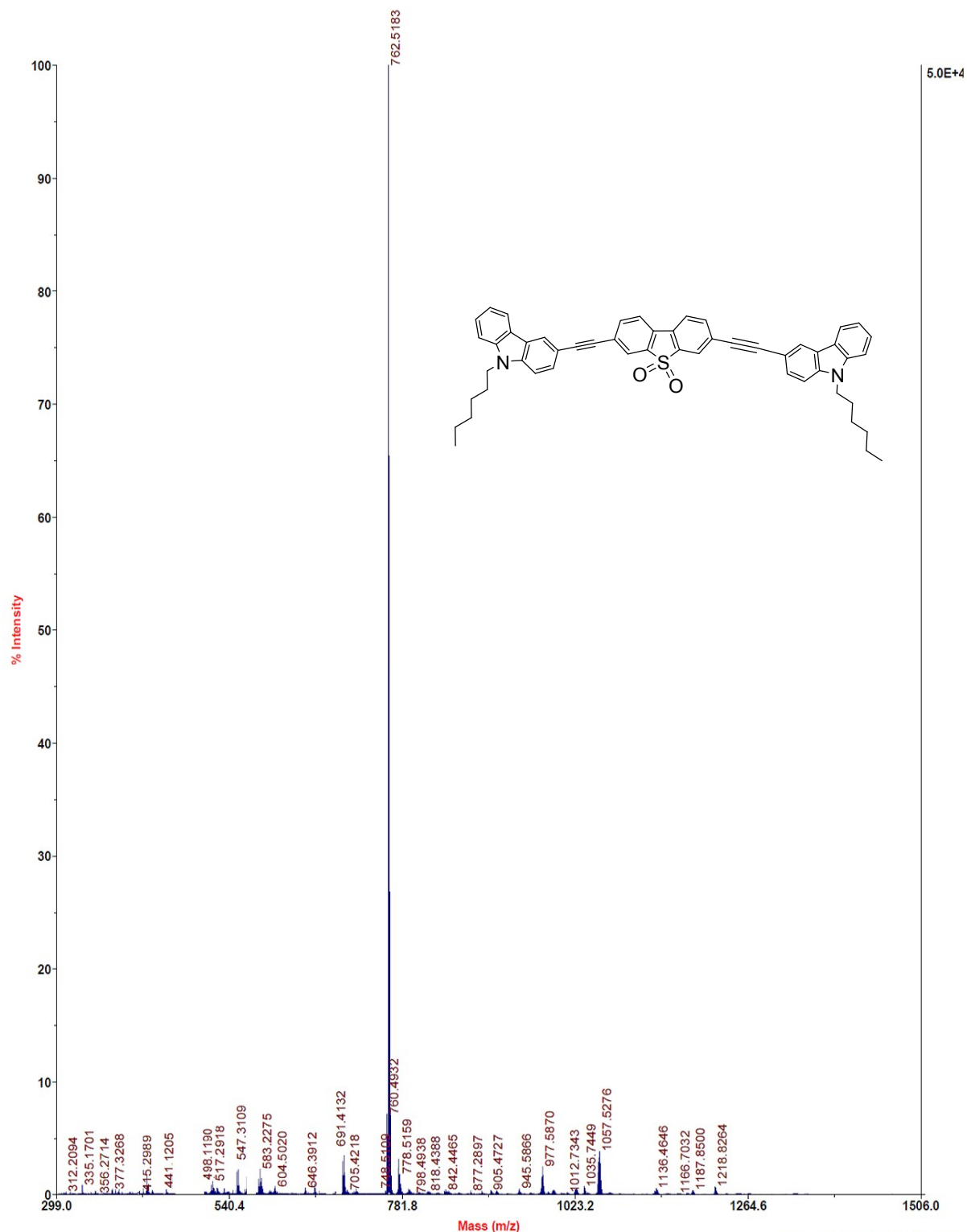


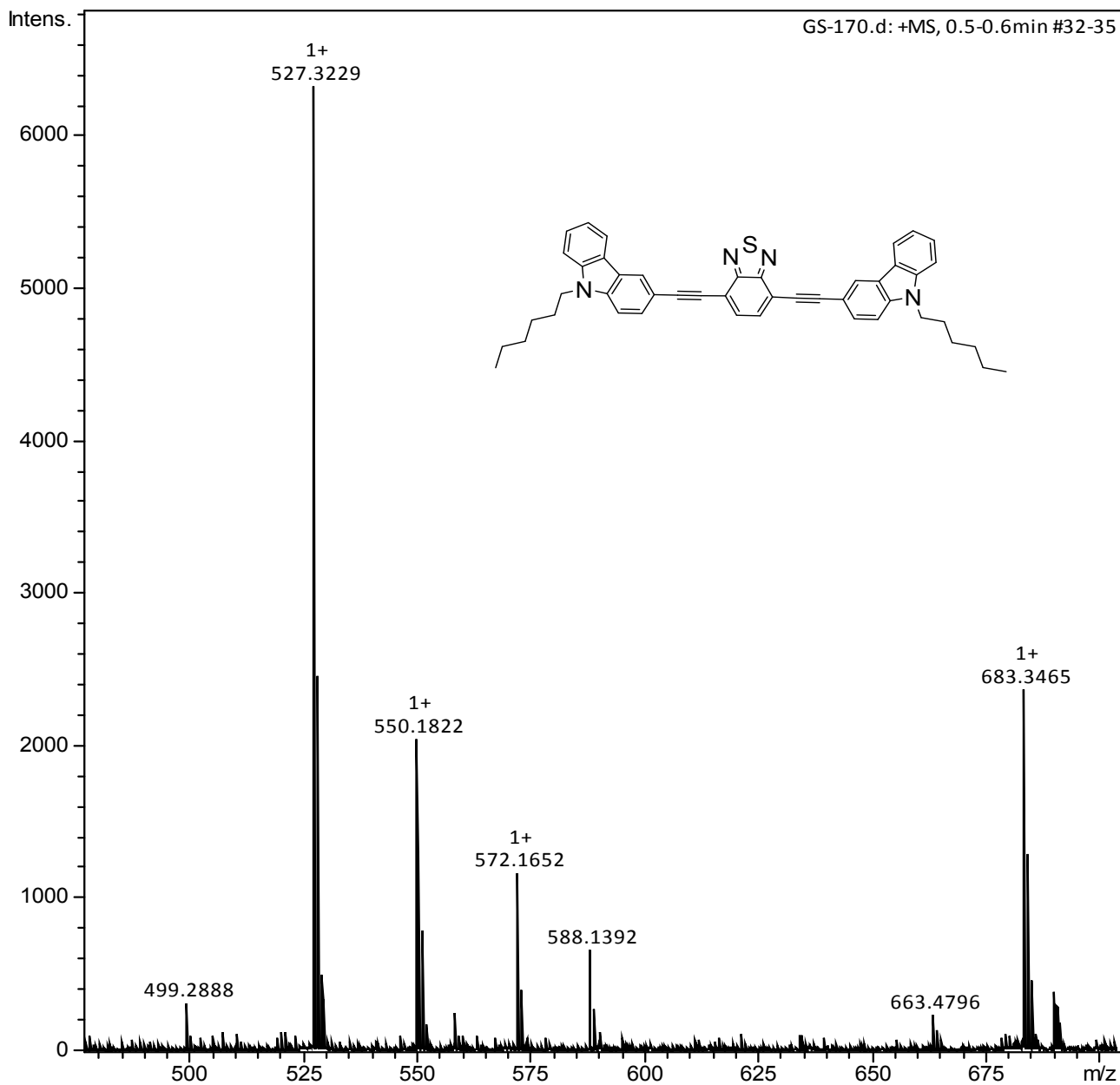
Figure S38. Mass spectrum of derivative **8**



Printed: 18:37, March 30, 2012

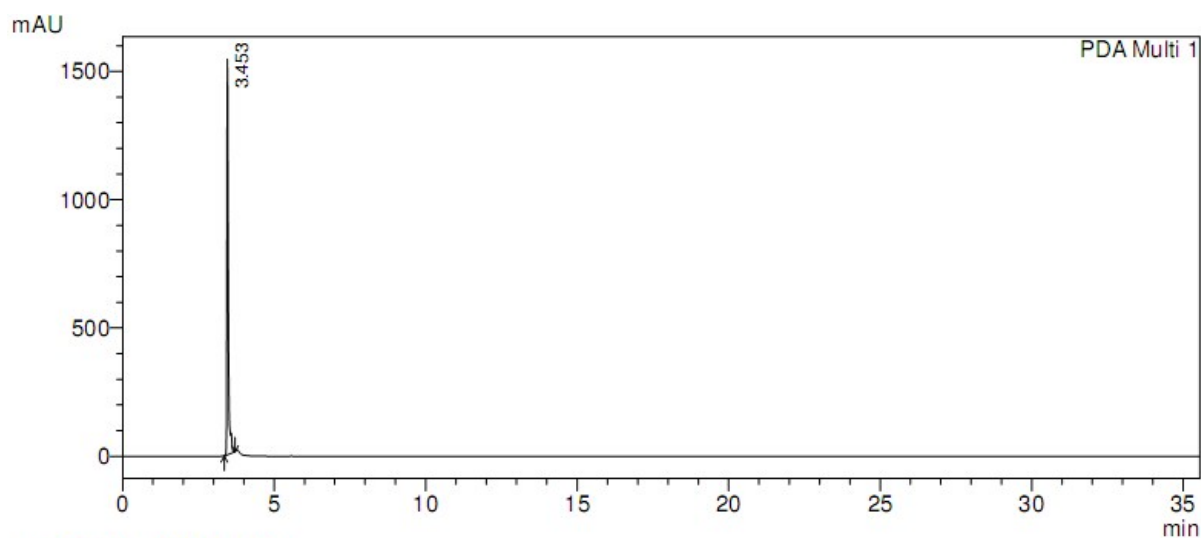
C:\Applied Biosystems MDS Analytical Technologies\TOFTOF Data\ExportT2D\AIRF-MASS\28032012\H12_MS.t2d

Figure S39. Mass spectrum of derivative 9



FigureS40. Mass spectrum of derivative 10

Figure S41. HPLC data of derivative 1



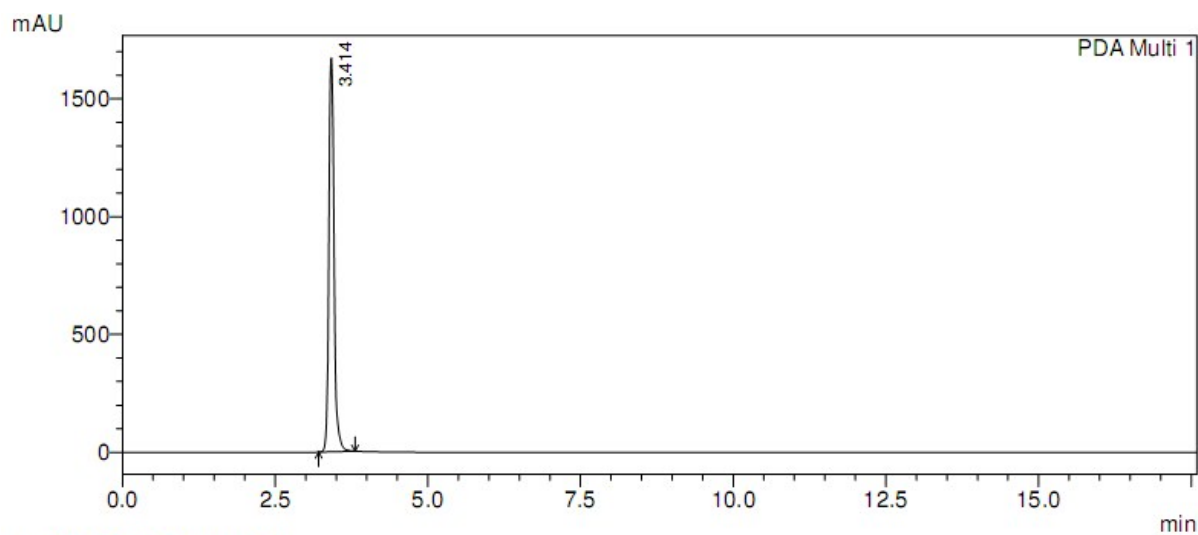
1 PDA Multi 1/290nm 4nm

PeakTable

PDA Ch1 290nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.453	5182746	1542309	100.000	100.000
Total		5182746	1542309	100.000	100.000

Figure S42. HPLC data of derivative 2



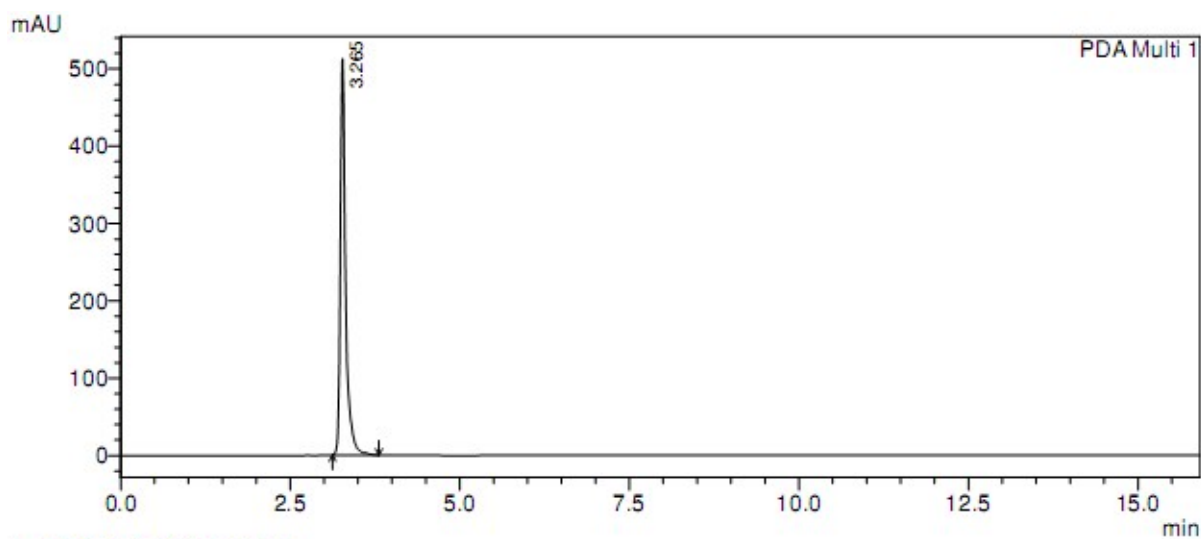
1 PDA Multi 1/291nm 4nm

Peak Table

PDA Ch1 291nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.414	9996355	1672070	100.000	100.000
Total		9996355	1672070	100.000	100.000

Figure S43. HPLC data of derivative **3**



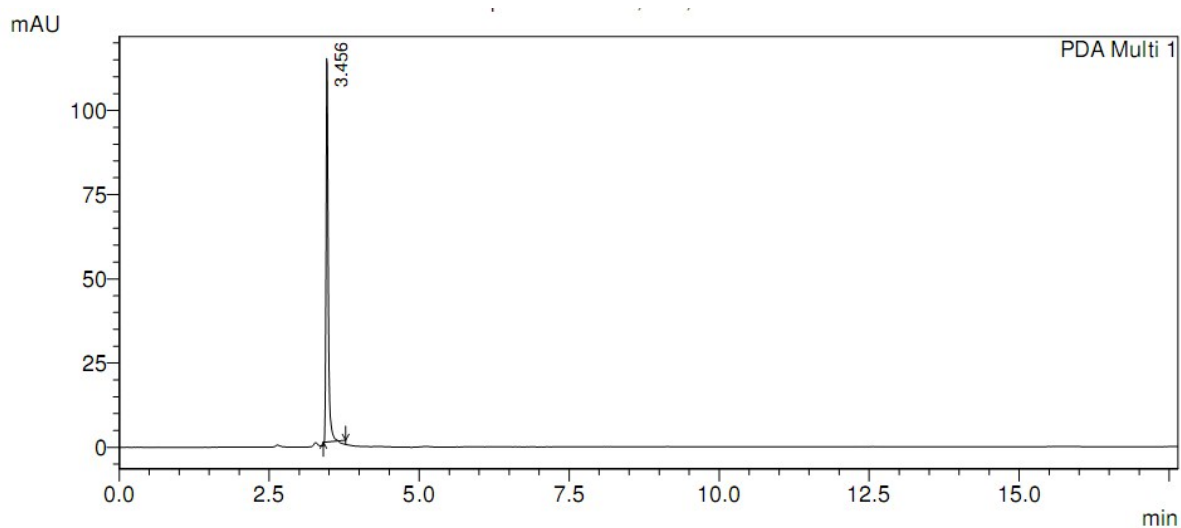
1 PDA Multi 1/298nm 4nm

PeakTable

PDA Ch1 298nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.265	2816841	512540	100.000	100.000
Total		2816841	512540	100.000	100.000

Figure S44. HPLC data of derivative **8**



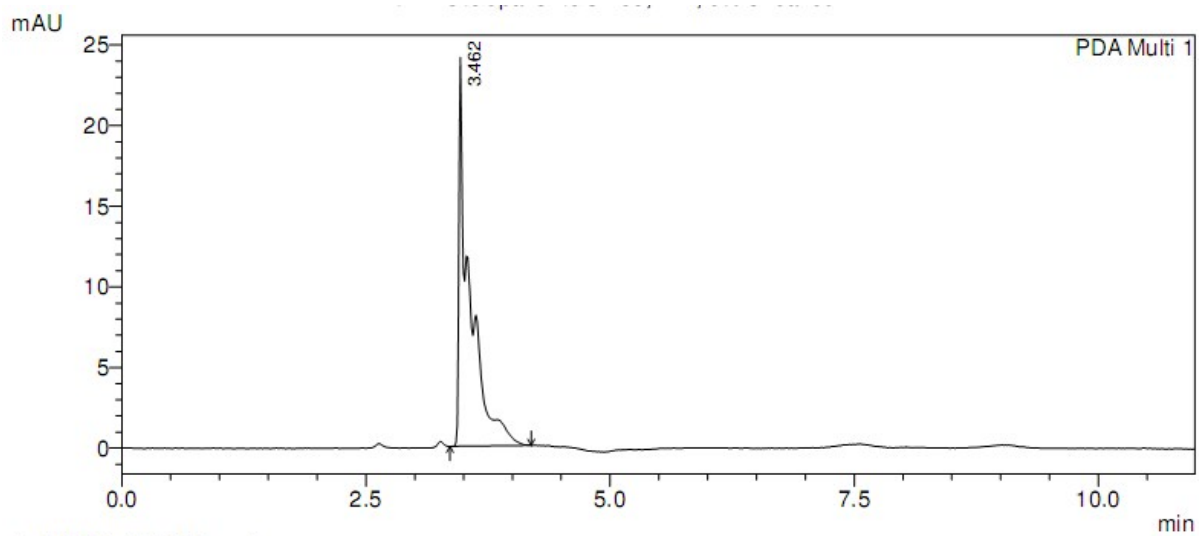
1 PDA Multi 1/308nm 4nm

PeakTable

PDA Ch1 308nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.456	291170	113852	100.000	100.000
Total		291170	113852	100.000	100.000

Figure S45. HPLC data of derivative 9

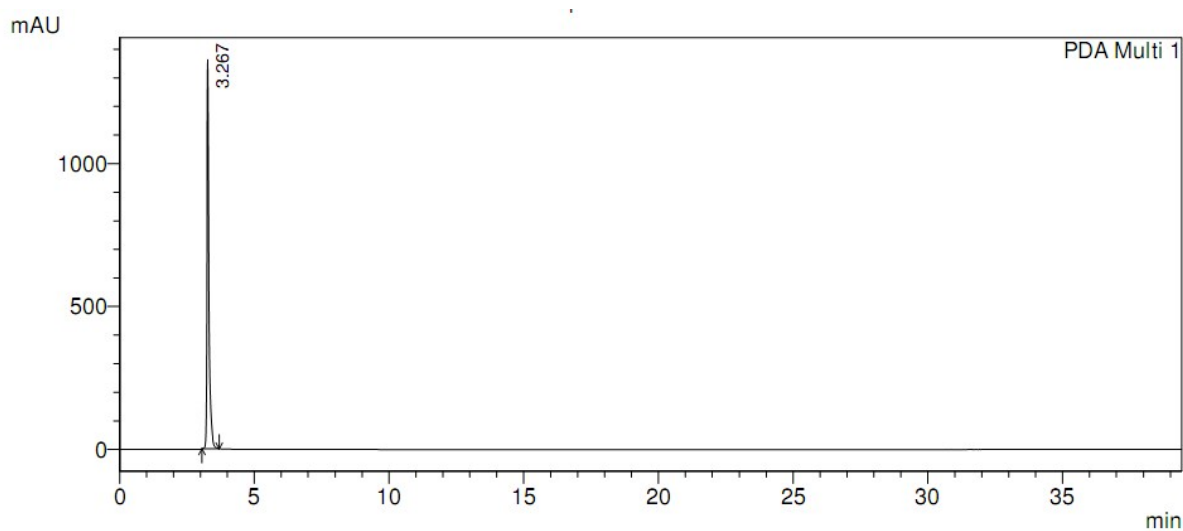


PeakTable

PDA Ch1 297nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.462	183212	24100	100.000	100.000
Total		183212	24100	100.000	100.000

Figure S46. HPLC data of derivative 10



PeakTable

PDA Ch1 301nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.267	7128071	1362178	100.000	100.000
Total		7128071	1362178	100.000	100.000