Supporting Information

Topochemical Polymerization of Phenylacetylene Macrocycles: A New Strategy for the Preparation of Organic Nanorods

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1- General

Chemical reagents were purchased from Sigma-Aldrich Co. Canada, Alfa Aesar Co., TCI America Co. or Oakwood Products Inc. and were used as received. Solvents used for organic synthesis were obtained from Fisher Scientific (except THF from Sigma- Aldrich Co. Canada) and purified with a Solvent Purifier System (SPS) (Vacuum Atmosphere Co., Hawthorne, USA). Other solvents were obtained from Fisher Scientific and were used as received. Tetrahydrofuran (THF) and triethylamine (Et_3N) used for Sonogashira reactions were degassed 30 minutes prior to use. All anhydrous and air sensitive reactions were performed in oven-dried glassware under positive argon pressure. Analytical thin-layer chromatographies were performed with silica gel 60 F254, 0.25 mm pre-coated TLC plates (Silicycle, Québec, Canada). Compounds were visualized using 254 nm

and/or 365 nm UV wavelength and/or aqueous sulfuric acid solution of ammonium heptamolybdate tetrahydrate (10 g/100 mL H_2SO_4 + 900 mL H_2O). Flash column chromatographies were performed on 230-400 mesh silica gel R10030B (Silicycle, Québec, Canada). TEM copper grids pre-coated with amorphous carbon were purchased from Ted Pella, Inc. (Redding, USA).

Apparatus

Nuclear magnetic resonance (NMR) spectra were recorded on a Varian Inova AS400 spectrometer (Varian, Palo Alto, USA) at 400 MHz (^{1}H) and 100 MHz (^{13}C). Highresolution mass spectra (HRMS) were recorded with an Agilent 6210 Time-of-Flight (TOF) LC-MS apparatus equipped with an ESI or APPI ion source (Agilent Technologies, Toronto, Canada). MALDI-TOF measurements were performed on a Bruker Biflex IV equipped with nitrogen laser. FT-IR was recorded in ATR mode on Infrared spectrometer (Thermo-Nicolet Magne 850) equipped with Golden Gate. UV-visible absorption spectra were recorded on a Varian diode-array spectrophotometer (model Cary 500) using 3-mm path length quartz cells. Scanning electron microscopy (SEM) images were taken using a JEOL JSM-6360 LV. Transmission electron microscopy (TEM) images were taken using a JEOL-1230. High resolution transmission microscopy (HRTEM) images were taken using a JEOL 2100 equipped with a field emission gun. X-ray diffraction was recorded on Siemens X-rays Diffractometer (Model S3 D5000).

Gelation test

To test the gelation properties of PAM 2 in a given solvent, we proceeded as follow: in a vial, PAM 2 was dissolved in a solvent. After dissolution by sonication, the vial was sealed and heated until a clear solution was obtained. The clear solution was allowed to slowly cool down at room temperature. The stability of the gel was confirmed by tube inversion.

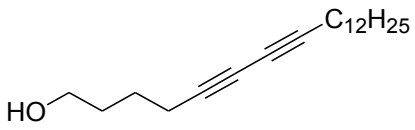
SEM imaging

Organogel obtained in ethyl acetate was deposited on a stainless steel substrate and allowed to dry for 3-4 days. Then, gold particles were sputtered on dried gel prior to imaging.

TEM imaging

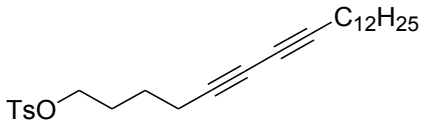
The SEC-purified PDA was dissolved in $CHCl_3$ and was sonicated for two hours. The resulting solution was directly deposited on a copper grid pre-coated with amorphous carbon and slowly evaporated prior to imaging.

2- Synthetic procedure



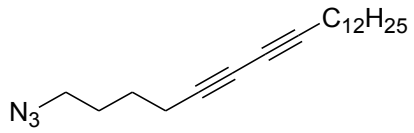
Compound 2. A 250 mL round bottom flask equipped with a magnetic stir bar was charged with KOH (4.60 g, 81.5 mmol) in H_2O (80 mL) at 0°C. Bromine (1.26 mL, 24.5 mmol) was added dropwise followed by 5-hexyn-1-ol (2.25 mL, 20.4 mmol). The mixture was stirred for 2 hours at 0°C in the dark and was then diluted with AcOEt, washed with brine (3x), dried with sodium sulfate and the solvent was removed under reduced pressure. The crude product was then charged without further purification in a 250 mL round bottom flask equipped with a magnetic stir bar with 30% aqueous *n*-$BuNH_2$ (92.6 mL) and CuCl (92 mg, 0.92 mmol). Small amount of hydroxylamine was added to the solution after appearance of a blue colour. Another 100 mL round bottom flask equipped with a magnetic stir bar was charged with 1-tetradecyne (5.70 mL, 23.2 mmol) and Et_2O (46 mL). The solution was added dropwise to the catalyst/bromoalkyne solution. At this step, hydroxylamine was continually added until a blue color appeared. A small amount of *n*-$PrNH_2$ was added until the reaction mixture become translucent and the mixture was stirred for 2 hours. The reaction mixture was then diluted with AcOEt, washed with 10% aqueous HCl (3x), dried with sodium sulfate and the solvent was removed under reduced pressure to afford compound 2 (6.62 g, 96% yield) as a colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz): 3.66 (t, J = 6.1 Hz, 2H), 2.31 (t, J = 6.3 Hz, 2H), 2.24

(t, J = 7.0 Hz, 2H), 2.18 (t, J = 7.0 Hz, 1H), 1.65 (m, 4H), 1.51 (m, 4H), 1.37 (m, 4H), 1.26 (m, 12H), 0.87 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 78.1, 77.7, 68.3, 65.9, 32.2, 31.9, 29.9 (3C), 29.7, 29.6, 29.3, 29.1, 28.7, 28.6, 24.8, 22.9, 19.4, 18.6, 14.4; HRMS (APPI-TOF) *m/z* calcd for $C_{20}H_{34}O[M+H]^+$: 291.2682, found 291.2695; FTIR (ATR): 3314w, 2920m, 2852m, 1466w, 1059w.

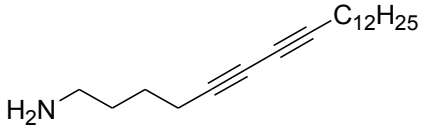


Icosa-5,7-diynyltosylate. A 250 mL round bottom flask equipped with a magnetic stir bar was charged with compound 2 (6.50 g, 23.4 mmol), CH_2Cl_2 (74 mL), tosyl chloride (6.39 g, 33.6 mmol), DMAP (273 mg, 2.24 mmol) and Et_3N (9.23 mL, 67.1 mmol). The reaction mixture was stirred for 2 hours, diluted with CH_2Cl_2, washed with water (3x), dried with sodium sulfate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using hexanes to 4% acetone/hexanes as eluents to afford compound 3 (5.5 g, 56% yield) as a colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz): 7.73 (d, J = 8.2 Hz, 2H), 7.31 (t, J = 8.0 Hz, 2H), 3.99 (t, J = 6.4 Hz, 2H), 2.40 (s, 3H), 2.18 (q, J = 6.4 Hz, 4H), 1.70 (m, 2H), 1.47 (m, 4H), 1.32 (m, 2H), 1.21 (m, 20H), 0.83 (t, J = 7.0Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 145.0, 133.2, 130.1, 128.0, 127.2, 78.1, 76.2, 70.1, 66.3, 65.3, 32.1, 29.9 (3C), 29.7, 29.6, 29.3, 29.1, 28.5, 28.0, 24.4, 22.9, 21.8, 19.4, 18.7, 14.4; HRMS (APPI-TOF) *m/z* calcd for

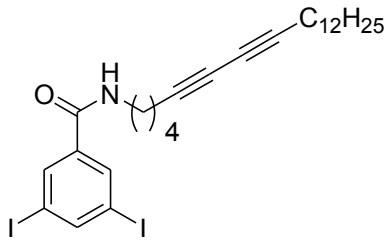
$C_{27}H_{40}O_3S[M+H]^+$: 446.2804, found 446.2816; FTIR (ATR): 3449b, 2924m, 2854m, 1176m.



Compound 3. A 25 mL round bottom flask equipped with a magnetic stir bar was charged with icosa-5,7-diynyltosylate (2.50 g, 5.60 mmol), DMF (5.6 mL) and sodium azide (914 mg, 14.1 mmol) under nitrogen. The reaction was heated to 70°C and was stirred for 3 hours. The reaction mixture was diluted with Et_2O, washed with water (3x), dried with sodium sulfate. The solvent was removed under reduced pressure to afford compound 3 (1.56 g, 88% yield) without further purification as a colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz): 3.31 (t, *J* = 6.6 Hz, 2H), 2.31 (t, *J* = 6.7 Hz, 2H), 2.25 (t, *J* = 6.7 Hz, 2H), 1.72 (m, 2H), 1.61 (m, 2H), 1.52 (m, 4H), 1.37 (m, 2H), 1.26 (m, 14H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 77.2, 76.7, 50.9, 31.9, 29.7 (3C), 29.5, 29.4, 29.1, 28.9, 28.3, 27.9, 22.4, 22.7, 19.2, 18.8, 14.1; HRMS (APPI-TOF) *m/z* calcd for $C_{20}H_{33}N_3[M+H]^+$: 316.2747, found 316.2759; FTIR (ATR): 3026w, 2924m, 2852w, 2096w, 1493m, 1452m.

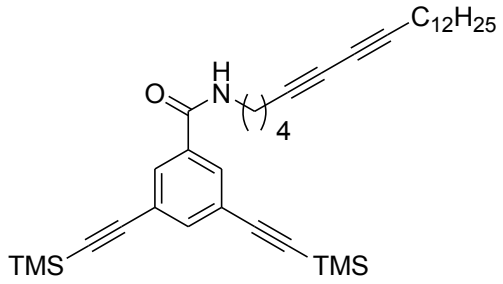


Compound 4. A 25 mL round bottom flask equipped with a magnetic bar was charged with compound 3 (2.00 g, 6.33 mmol), MeOH (8 mL) and tin chloride (3.50 g, 15.8 mmol) at 0°C. The reaction mixture was stirred for 4 hours at room temperature and was diluted with Et_2O. The organic layer was washed with 1M NaOH (3x), dried with sodium sulfate and the solvent was removed under pressure to afford compound 4 (1.43 g, 78% yield) without further purification as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz): 3.69 (s, 2H), 2.78 (t, *J* = 6.7 Hz, 2H), 2.29 (t, *J* = 6.7 Hz, 2H), 2.23 (t, *J* = 6.9 Hz, 2H), 1.60 (m, 4H), 1.50 (m, 2H), 1.36 (m, 2H), 1.25 (m, 14H), 0.87 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 78.1, 76.9, 65.9, 65.3, 41.2, 32.1, 31.5, 29.9 (3C), 29.7, 29.6, 29.3, 29.1, 28.6, 25.8, 22.9, 19.4, 19.2, 14.4; HRMS (APPI-TOF) *m/z* calcd for $C_{20}H_{35}N[M+H]^+$: 290.2482, found 290.9854; FTIR (ATR): 3750w, 2921m, 2851m, 2337w.



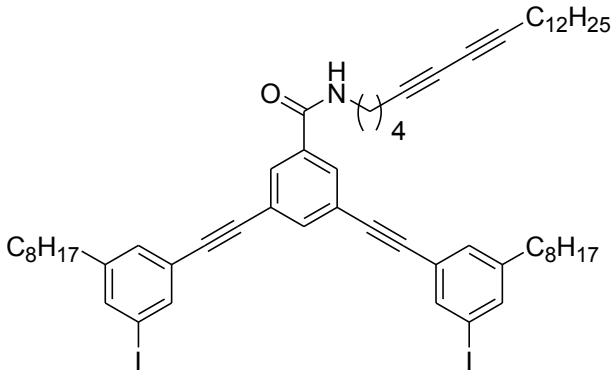
Compound 5. A 50 mL round bottom flask equipped with a magnetic stir bar was charged with 3,5-diiodobenzoïc acid (1.20 g, 3.21 mmol), CH_2Cl_2 (16 mL), EDC (923 mg, 4.81 mmol), DMAP (196 mg, 1.61 mmol) and compound 4 (1.39 g, 4.81 mmol). The

reaction mixture was stirred overnight and diluted with CH_2Cl_2. The organic layer was washed with water (3x), dried with sodium sulfate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using hexanes to 7% acetone/hexanes as eluents to afford compound **5** (1.73 g, 84% yield) as a white powder. m.p: 76-79°C; ^{1}H NMR ($CDCl_3$, 400 MHz): 8.11 (s, 1H), 7.99 (s, 2H), 6.5 (m, 1H), 3.43 (q, *J* = 6.0 Hz, 2H), 2.30 (t, *J* = 6.7 Hz, 2H), 2.23 (t, *J* = 6.8 Hz, 2H), 1.72 (m, 2H), 1.59 (m, 2H), 1.49 (m, 2H), 1.35 (m, 2H), 1.24 (m, 16H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 164.9, 147.9, 138.2, 135.5, 117.7, 95.1, 78.4, 76.8, 66.3, 65.3, 39.9, 32.2, 29.9 (3C), 29.7, 29.6, 29.4, 28.8, 28.6, 25.8, 22.9, 19.5, 19.2, 14.4; HRMS (APPI-TOF) *m/z* calcd for $C_{27}H_{37}I_2NO[M+H]^+$: 646.1037, found 646.1064; FTIR (ATR): 3281w, 2919m, 2850m, 1638m, 1536m.



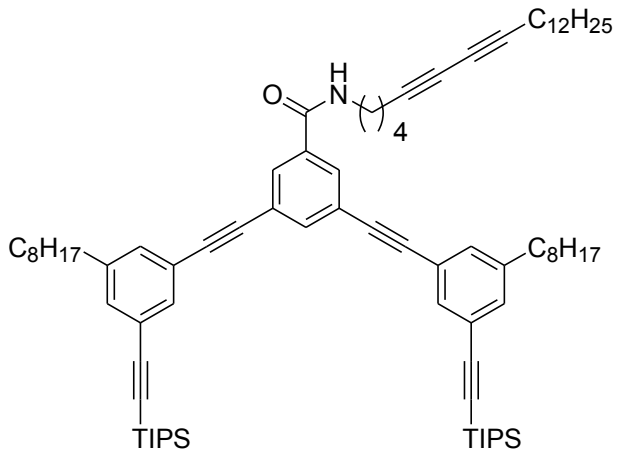
Compound 6. A 100 mL round bottom flask equipped with a magnetic stir bar was charged with **5** (1.73 g, 2.68 mmol), degassed THF (26 mL), degassed Et_3N (1.47 mL, 10.7 mmol), $PdCl_2(PPh_3)_2$ (75 mg, 0.11 mmol), CuI (20 mg, 0.11 mmol) and trimethylsilylacetylene (1.51 mL, 10.7 mmol) under argon atmosphere. The reaction mixture was stirred overnight at room temperature, diluted with CH_2Cl_2, washed with

NH_4Cl (3x) and dried with sodium sulfate. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography on silica gel using 10% acetone/hexanes as eluent to afford compound **6** (1.26 g, 80% yield) as a dark orange oil. ^1H NMR ($CDCl_3$, 400 MHz): 7.78 (s, 2H), 7.65 (s, 1H), 6.35 (m, 1H), 3.44 (q, *J* = 6.4 Hz, 2H), 2.31 (t, *J* = 6.6 Hz, 2H), 2.23 (t, *J* = 6.6 Hz, 2H), 1.72 (m, 2H), 1.59 (m, 2H), 1.51 (m, 2H), 1.36 (m, 2H), 1.25 (m, 16H), 0.88 (t, *J* = 7.0 Hz, 3H), 0.24 (s, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): 166.2, 137.8, 135.2, 130.3, 124.2, 103.3, 96.4, 78.2, 76.7, 66.2, 65.3, 39.4, 32.1, 29.9 (3C), 29.7, 29.6, 29.3, 29.1, 28.9, 28.6, 25.8, 22.9, 19.4, 19.1, 14.4, 0.04; HRMS (APPI-TOF) *m/z* calcd for $C_{37}H_{55}NOSi_2[M+H]^+$: 586.3895, found 586.3915; FTIR (ATR): 3317b, 2925m, 2854m, 1640m, 1250m.



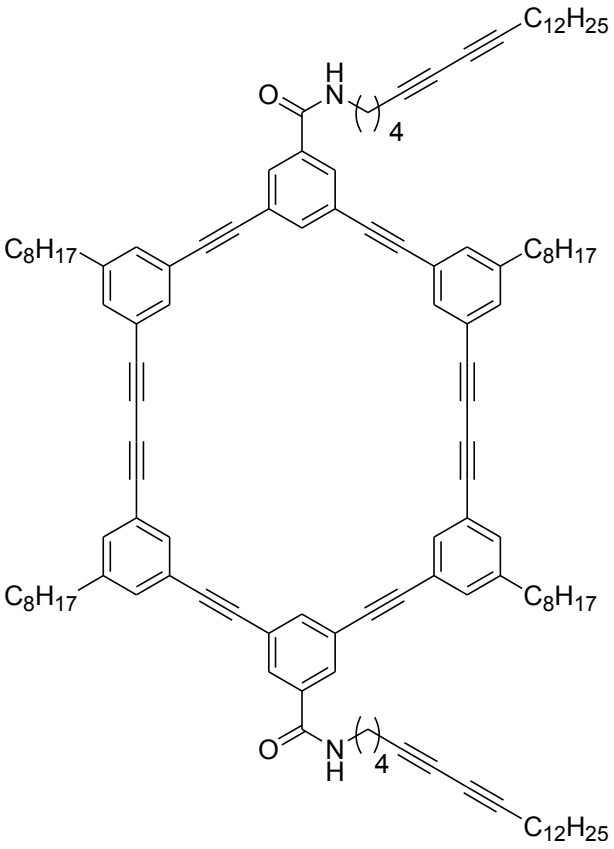
Compound 8. A 25 mL round bottom flask equipped with a magnetic stir bar was charged with compound **6** (1.20 g, 2.15 mmol), KOH (362 mg, 6.45 mmol), THF (5.3 mL) and MeOH (5.3 mL). The reaction mixture was stirred until complete disappearance of the starting product by TLC. After completion, the reaction mixture was diluted with CH_2Cl_2, washed with 10% aqueous HCl (2x), dried with sodium sulfate and the solvent

was removed under reduced pressure. The resulting product was charged without further purification in a 50 mL round bottom flask equipped with a magnetic stir bar with compound **7** (3.75 g, 8.49 mmol), degassed THF (17 mL), degassed DIPEA (2.37 mL, 13.6 mmol), $PdCl_2(PPh_3)_2$ (48 mg, 0.07 mmol) and CuI (13 mg, 0.07 mmol). The reaction mixture was stirred overnight and diluted with CH_2Cl_2. The organic layer was washed with saturated aqueous NH_4Cl (3x), dried with sodium sulfate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using hexanes to 10% acetone/hexanes as eluents to afford compound **8** (900 mg, 49% yield) as a dark orange oil. ^{1}H NMR ($CDCl_3$, 400 MHz): 7.84 (s, 2H), 7.73 (s, 1H), 7.69 (s, 2H), 7.52 (s, 2H), 7.29 (s, 2H), 6.28 (m, 1H), 3.48 (q, J = 6.4 Hz, 2H), 2.53 (t, J = 7.8 Hz, 4H), 2.33 (t, J = 6.8 Hz, 2H), 2.22 (t, J = 6.8 Hz, 2H), 1.75 (m, 2H), 1.60 (m, 6H), 1.49 (m, 2H), 1.26 (m, 38H), 0.88 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): 166.2, 145.5, 138.2, 137.7, 137.1, 135.6, 131.2, 130.0, 124.5, 124.2, 94.0, 89.8, 88.7, 78.4, 76.8, 66.3, 65.3, 39.9, 35.6, 32.2, 32.1, 31.4, 31.2, 28.9 (3C), 29.7, 29.6 (2C), 29.5, 29.4, 29.3, 29.1, 28.9, 28.6, 25.8, 22.9 (2C), 19.4, 19.2, 14.4; HRMS (APPI-TOF) *m/z* calcd for $C_{59}H_{77}I_2NO[M+H]^+$: 1070.4167, found 1070.4186; FTIR (ATR): 3312b, 2924m, 2853m, 1637w, 1593w, 1553w.



Compound 9. A 25 mL round bottom flask equipped with a magnetic stir bar was charged with compound 8 (1.10 g, 1.03 mmol), degassed THF (10 mL), degassed Et_3N (0.57 mL, 4.11 mmol), $PdCl_2(PPh_3)_2$ (36 mg, 0.05 mmol), CuI (10 mg, 0.05 mmol) and triisopropylsilylacetylene (0.92 mL, 4.11 mmol). The reaction mixture was stirred overnight, diluted with CH_2Cl_2, washed with NH_4Cl and dried with sodium sulfate. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography on silica gel using 5% acetone/hexanes as eluent to afford compound 9 (1.18 g, 97% yield) as an orange oil.^{1}H NMR ($CDCl_3$, 400 MHz): 7.86 (s, 2H), 7.77 (s, 1H), 7.48 (s, 2H), 7.29 (s, 2H), 7.27 (s, 2H), 6.33 (m, 1H), 3.48 (q, J = 6.4 Hz, 2H), 2.57 (t, J = 7.6 Hz, 4H), 2.33 (t, J = 6.7 Hz, 2H), 2.22 (t, J = 6.7 Hz, 2H), 1.74 (m, 2H), 1.62 (m, 6H), 1.49 (m, 2H), 1.27 (m, 38H), 1.14 (s, 42H), 0.88 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): 166.3, 143.6, 137.1, 135.6, 132.9, 132.5, 131.9, 129.8, 127.2, 124.4, 124.0, 122.8, 106.5, 91.2, 90.7, 88.0, 78.3, 76.8, 66.3, 65.3, 39.8, 35.8, 32.2, 32.1, 31.5, 29.9 (3C), 29.7 (2C), 29.6, 29.5 (2C), 29.4, 29.1, 28.9, 28.6, 25.9, 22.9 (2C), 19.4, 19.2, 18.9,

14.4, 11.6: HRMS (APPI-TOF) *m/z* calcd for $C_{81}H_{119}NOSi_2[M+H]^+$: 1178.8903, found 1178.8947; FTIR (ATR): 3317b, 2923m, 2855m, 1639m, 1590m, 1462m.



PAM 2. A 50 mL round bottom flask equipped with a magnetic stir bar was charged with compound **9** (700 mg, 0.59 mmol), THF (12 mL) and TBAF 1.0M solution in THF(1.78 mL, 1.78 mmol). The reaction mixture was stirred until complete disappearance of the starting product by TLC, diluted with CH_2Cl_2, washed with water (3x) and dried with sodium sulfate. The solvent was removed under reduced pressure. The resulting product was charged without further purification in a 50 mL round bottom flask equipped with a

magnetic stir bar with degassed pyridine (15 mL). Another round bottom flask equipped with a magnetic stir bar was charged with CuCl (3.65 g, 36.9 mmol), $CuCl_2$ (768 mg, 5.71 mmol) and degassed pyridine (74 mL) under N_2 atmosphere. The first solution was added dropwise to the catalyst solution over 4 days using a syringe pump and the reaction mixture was stirred for an additional 7 days. The reaction mixture was diluted with $CHCl_3$ and poured in water. The organic phase was extracted successively with water, 25% aqueous NH_4OH, water, 10% aqueous CH_3COOH, water, 10% aqueous NaOH and brine. The organic layer was dried with sodium sulfate and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using 20% hexanes/$CHCl_3$ to $CHCl_3$ as eluents to afford PAM 2 (390 mg, 49% yield) as a white amorphous powder. m.p: >150°C; ^{1}H NMR ($CDCl_3$, 400 MHz): 7.71 (s, 2H), 7.64 (s, 4H), 7.49 (s, 4H), 7.20 (s, 4H), 7.18 (s, 4H), 6.70 (s, 2H), 3.53 (q, J = 7.0 Hz, 4H), 2.53 (t, J = 7.5 Hz, 8H), 2.39 (t, J = 6.9 Hz, 4H), 2.24 (t, J = 6.9 Hz, 4H), 1.83 (m, 4H), 1.69 (m, 4H), 1.62 (m, 8H), 1.52 (m, 4H), 1.29 (m, 76H), 0.90 (m, 18H): ^{13}C NMR ($CDCl_3$, 100 MHz): 166.7, 143.2, 135.1, 133.4, 132.1, 129.6, 129.3, 127.0, 123.9, 122.9, 122.1, 90.1, 88.1, 80.9, 78.1, 76.6, 74.3, 66.1, 65.1, 41.9, 39.7, 35.5, 31.9, 31.1, 29.7 (3C), 29.5, 29.4, 29.3 (2C), 29.1, 28.9, 28.7, 28.4, 25.7, 22.7 (2C), 21.5, 19.2, 19.0, 17.7, 14.1, 12.3; MS (MALDI-TOF): *m/z* calcd for $C_{126}H_{154}N_2O_2[M+H]^+$: 1727.1, found 1732.7; FTIR (ATR): 3271w, 2919m, 2851m, 1638m, 1592m, 1015b, 802m.

3- ^{1}H and ^{13}C NMR spectra

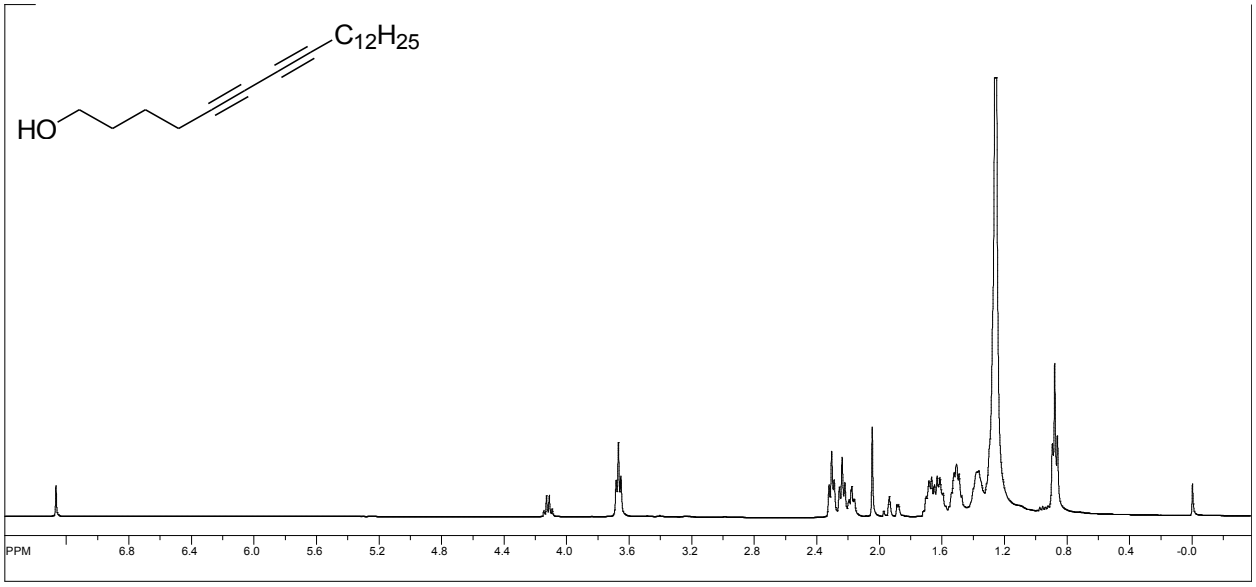


Figure S1. ^{1}H NMR of compound 2 in $CDCl_3$

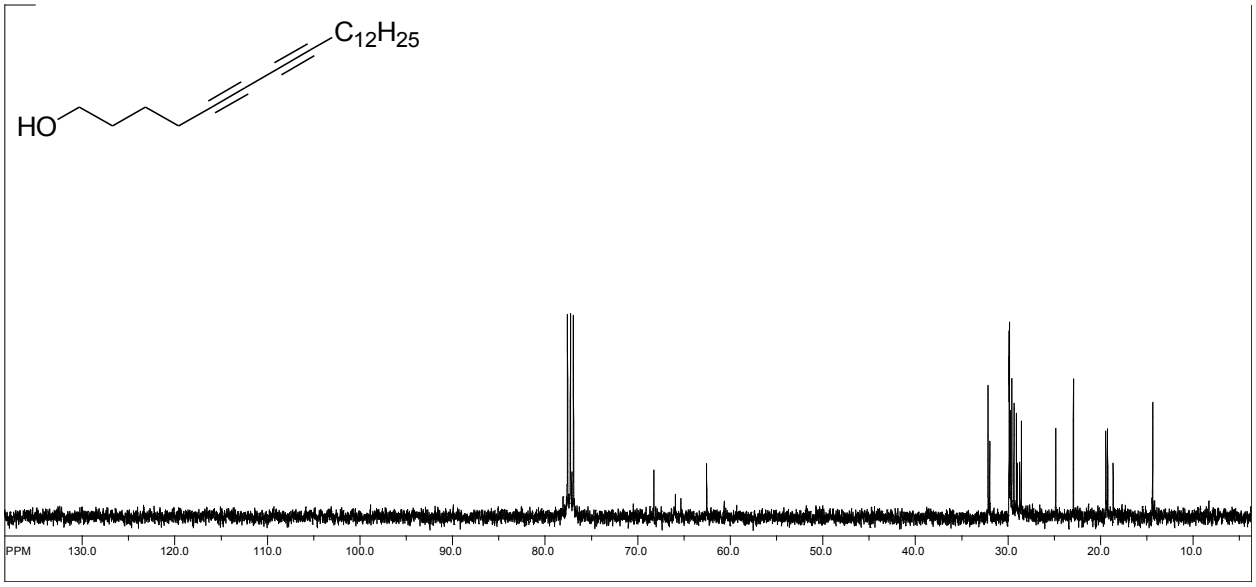


Figure S2. ^{13}C NMR of compound 2 in $CDCl_3$

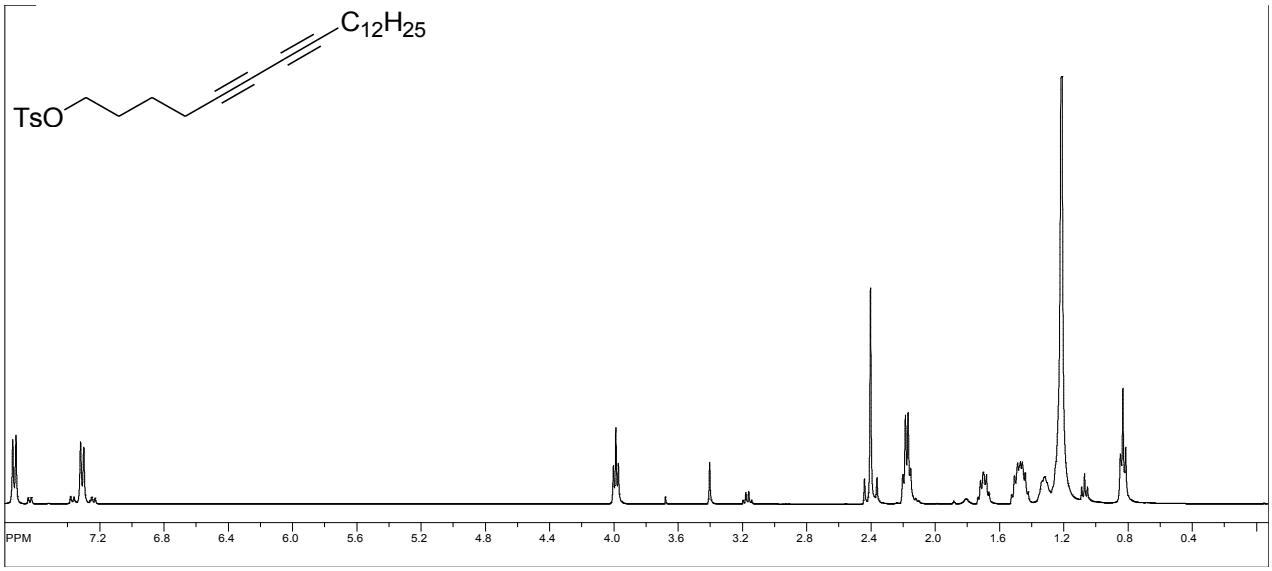


Figure S3. ^{1}H NMR of compound icosa-5,7-diynyltosylate in $CDCl_3$

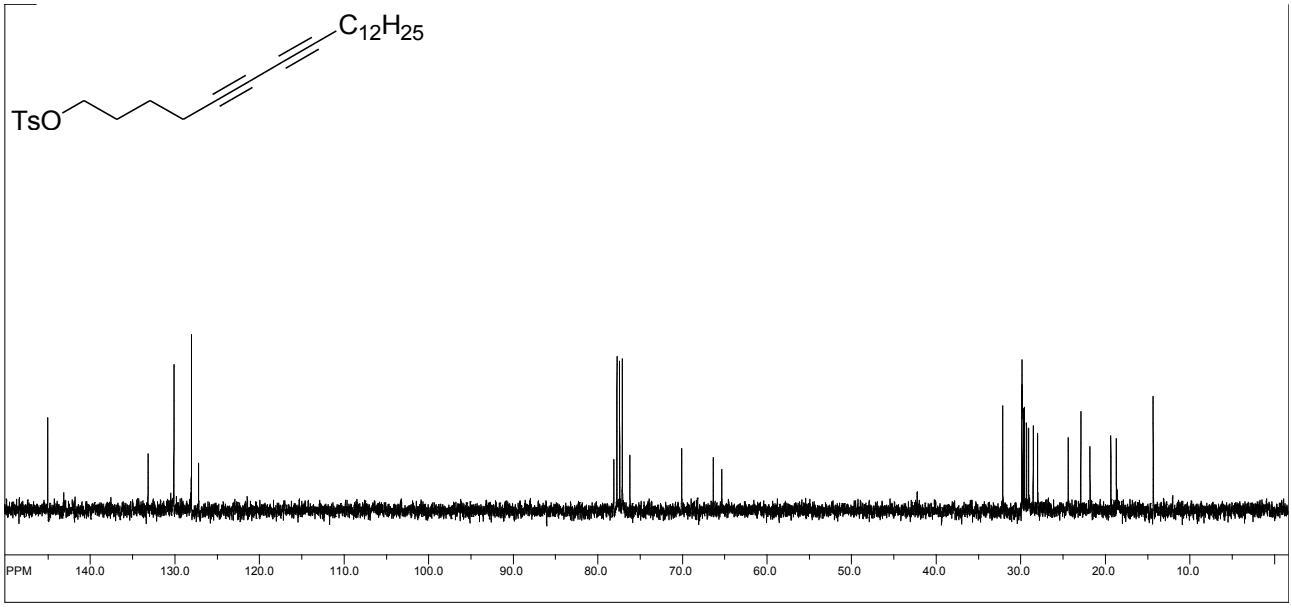


Figure S4. ^{13}C NMR of compound icosa-5,7-diynyltosylate in $CDCl_3$

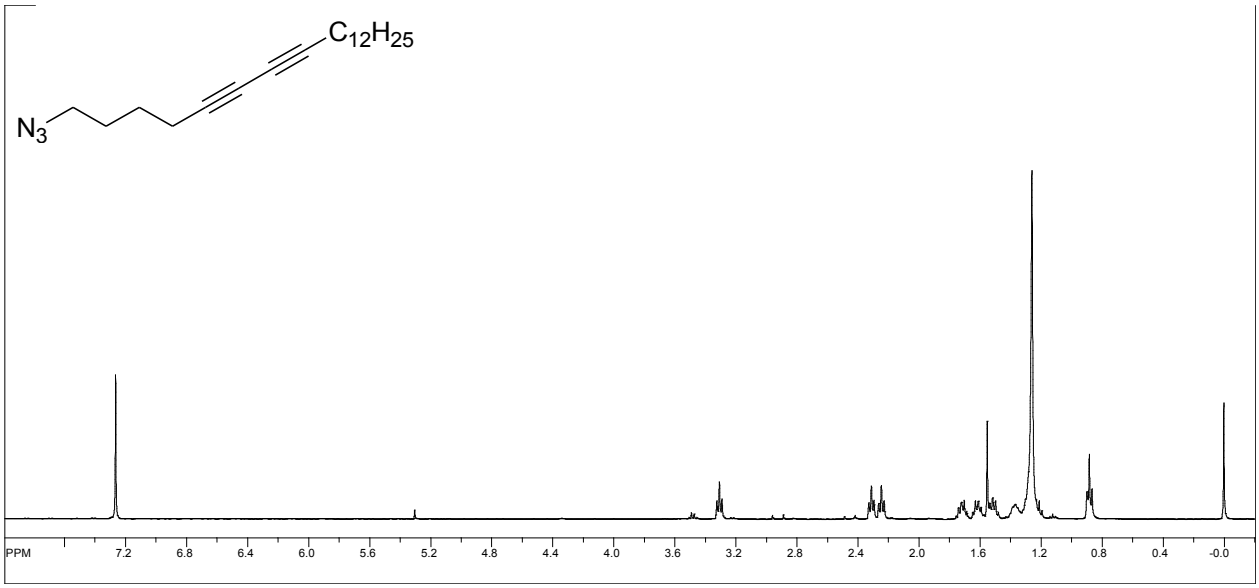


Figure S5. ^{1}H NMR of compound 3 in $CDCl_3$

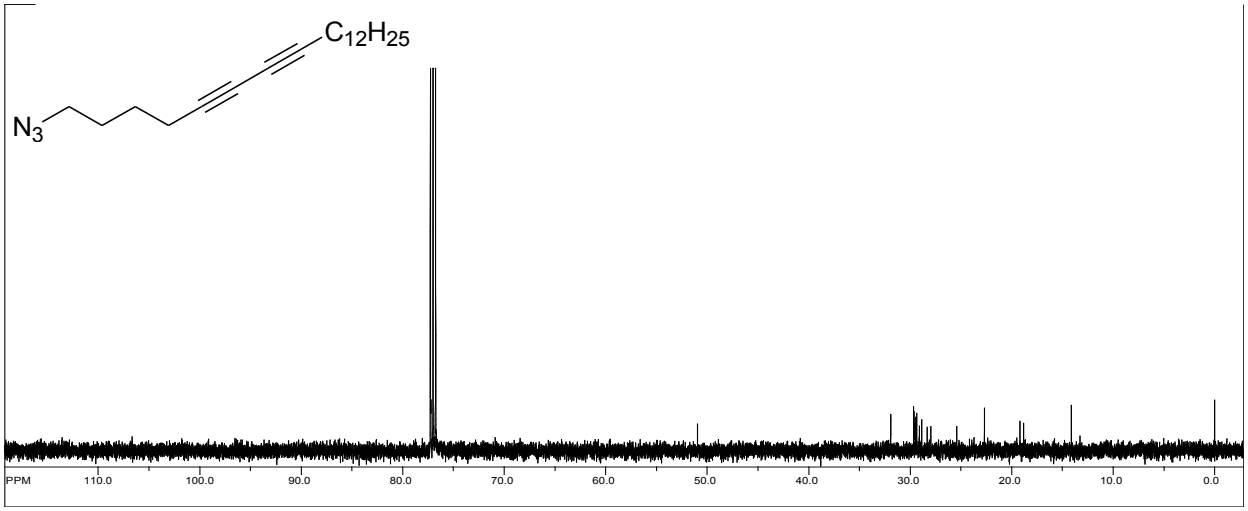


Figure S6. ^{13}C NMR of compound 3 in $CDCl_3$

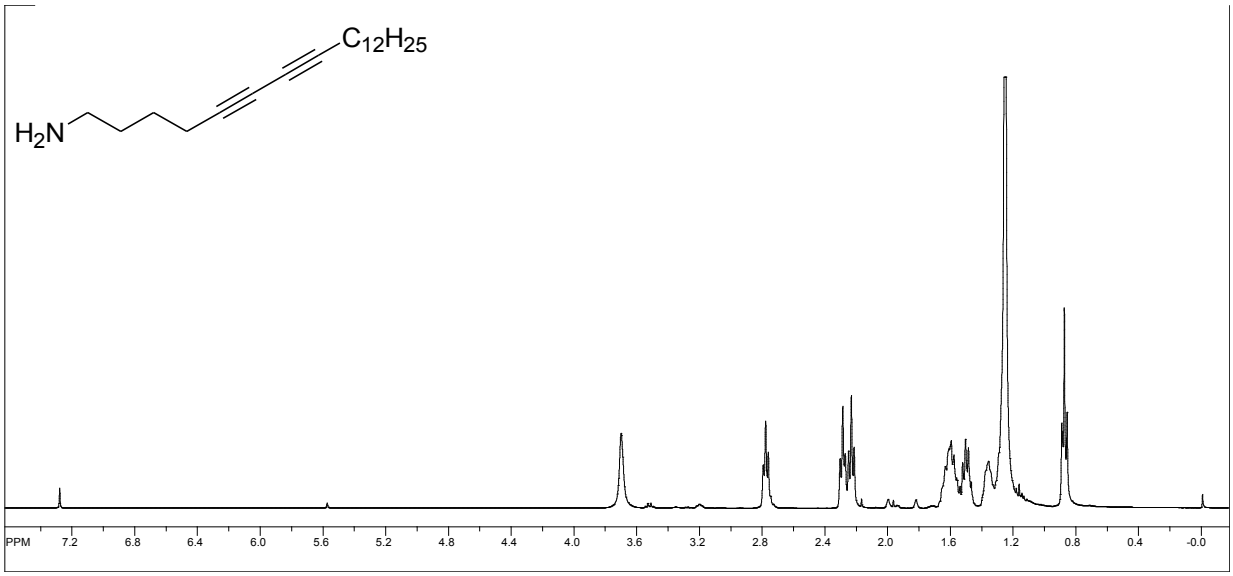


Figure S7. ^{1}H NMR of compound 4 in $CDCl_3$

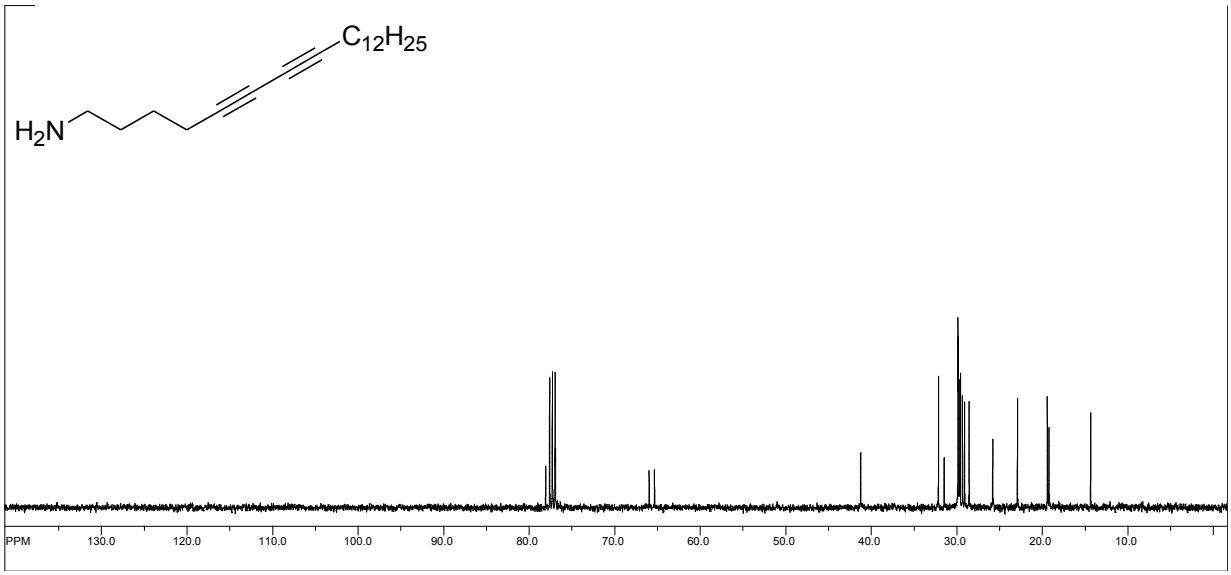


Figure S8. ^{13}C NMR of compound 4 in $CDCl_3$

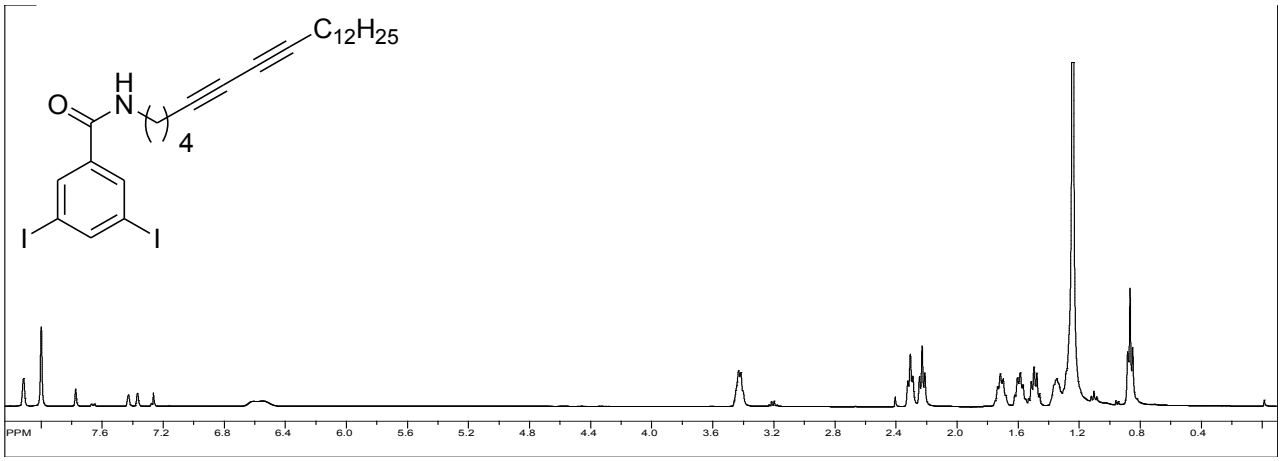


Figure S9. ^{1}H NMR of compound 5 in $CDCl_3$

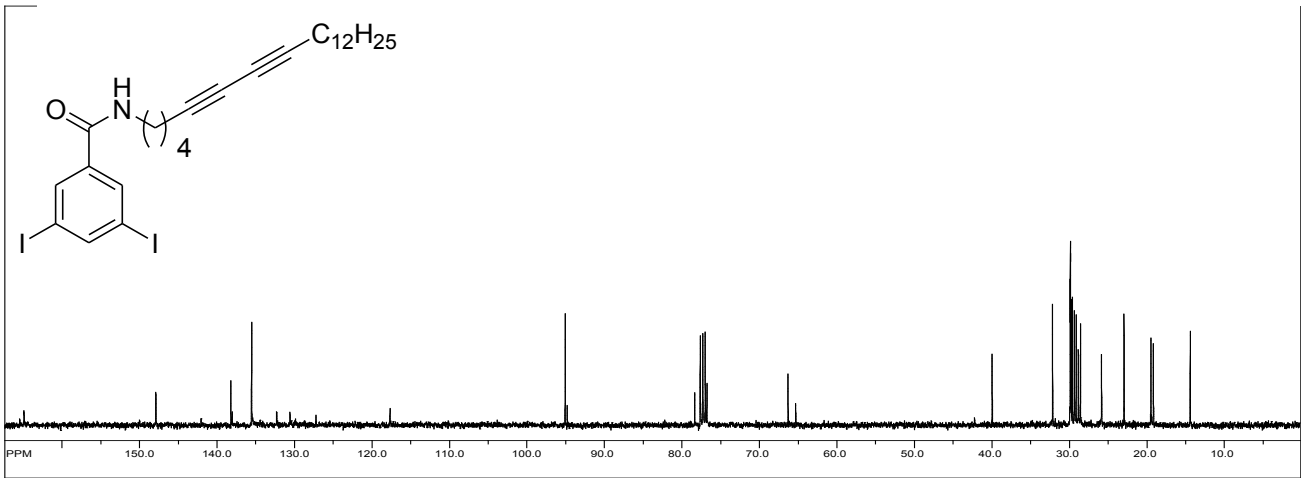


Figure S10. ^{13}C NMR of compound 5 in $CDCl_3$

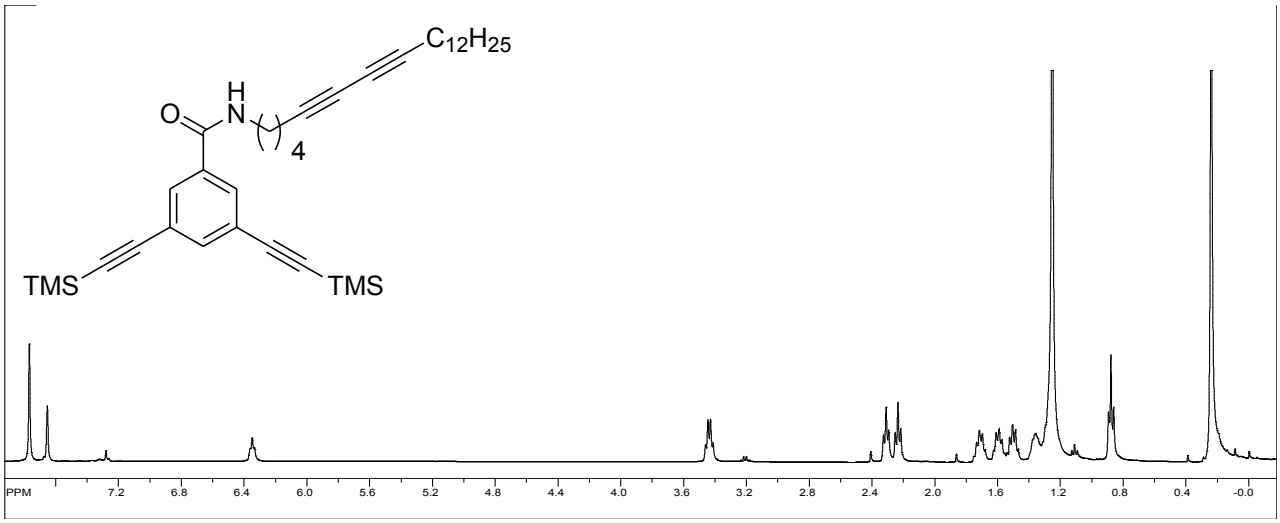


Figure S11. ^{1}H NMR of compound 6 in $CDCl_3$

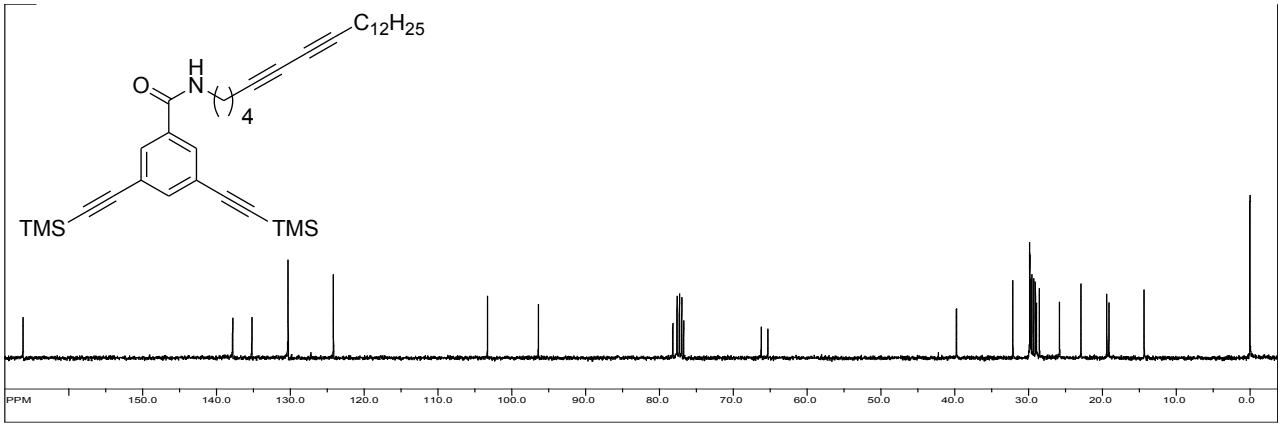


Figure S12. ^{13}C NMR of compound 6 in $CDCl_3$

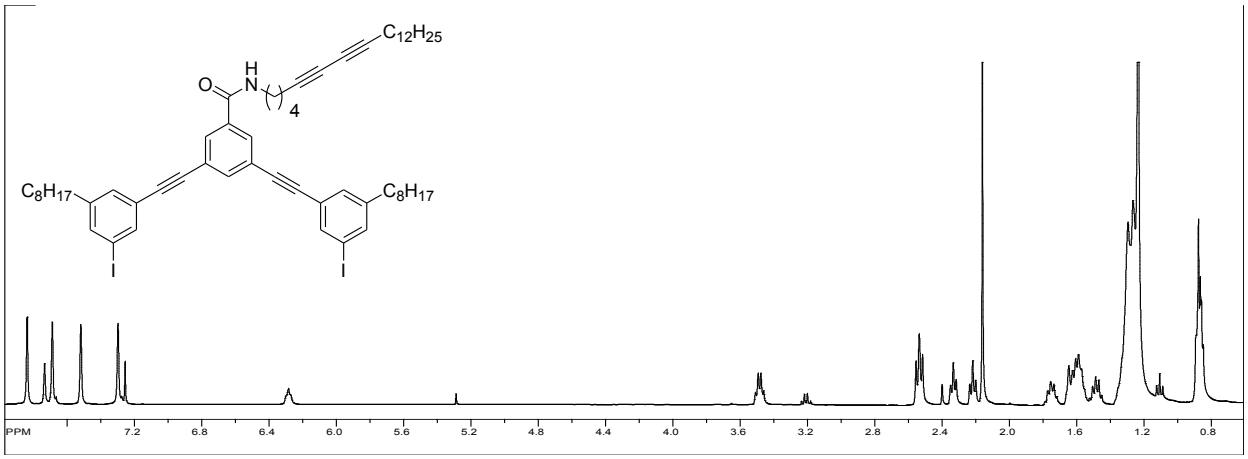


Figure S13. ^{1}H NMR of compound **8** in $CDCl_3$

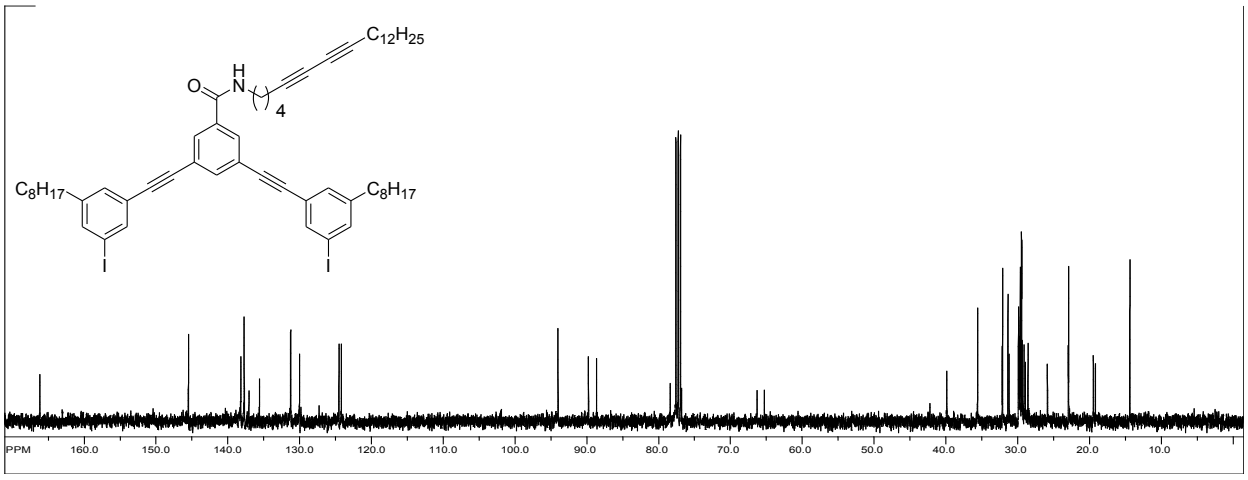


Figure S14. ^{13}C NMR of compound **8** in $CDCl_3$

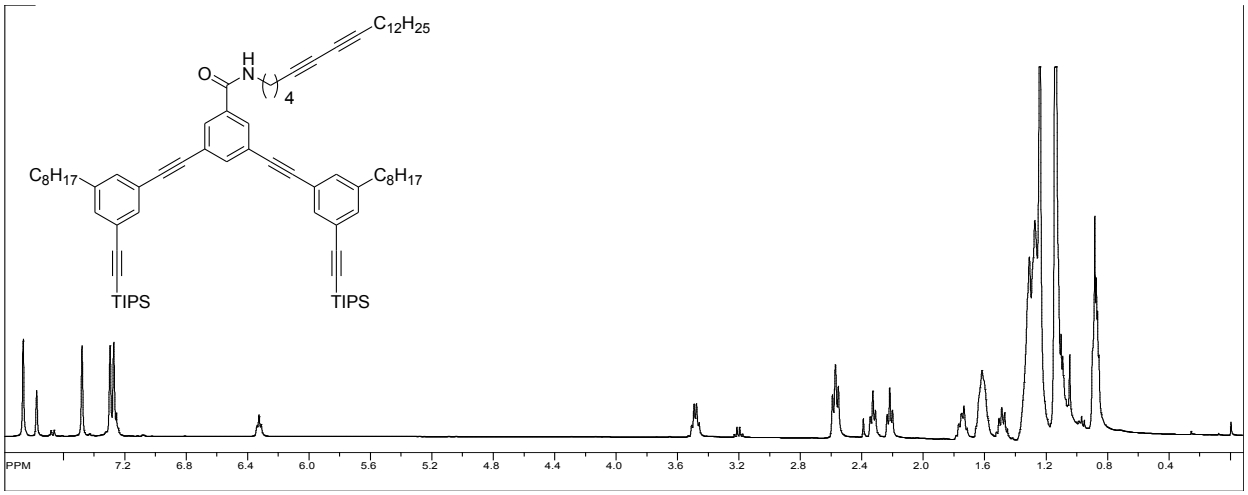


Figure S15. ^{1}H NMR of compound 9 in $CDCl_3$

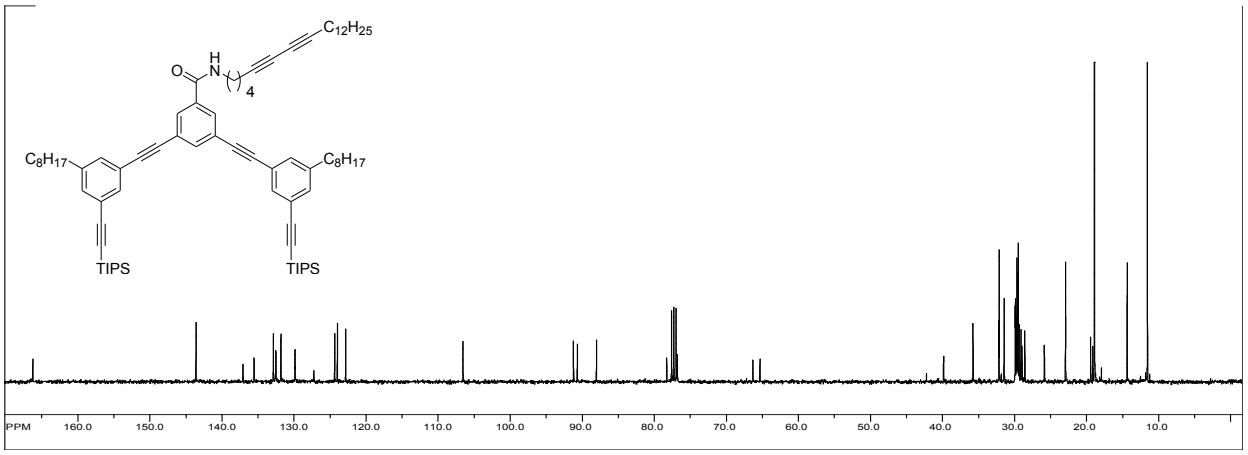


Figure S16. ^{13}C NMR of compound 9 in $CDCl_3$

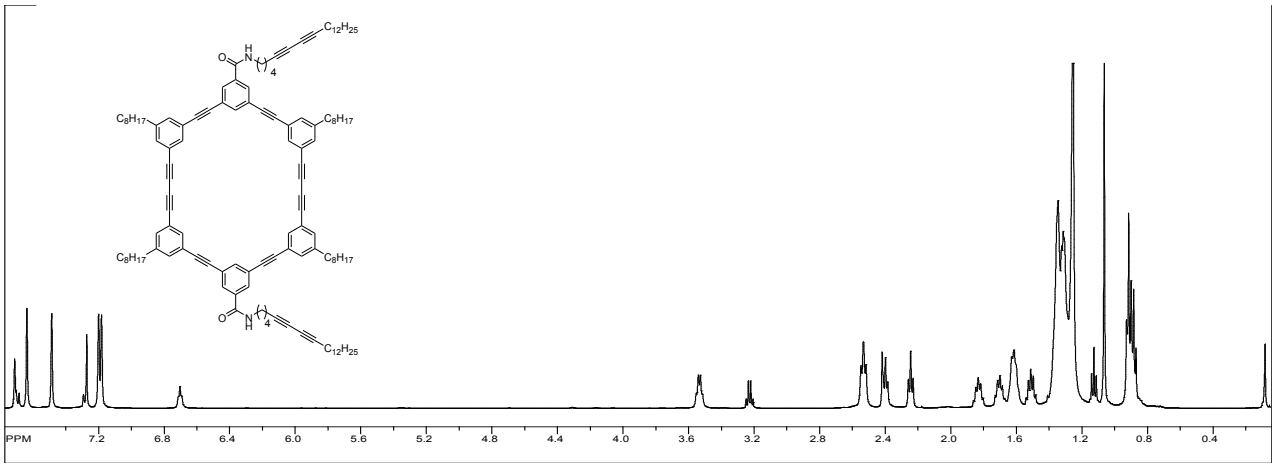


Figure S17. ^{1}H NMR of PAM 2 $CDCl_3$

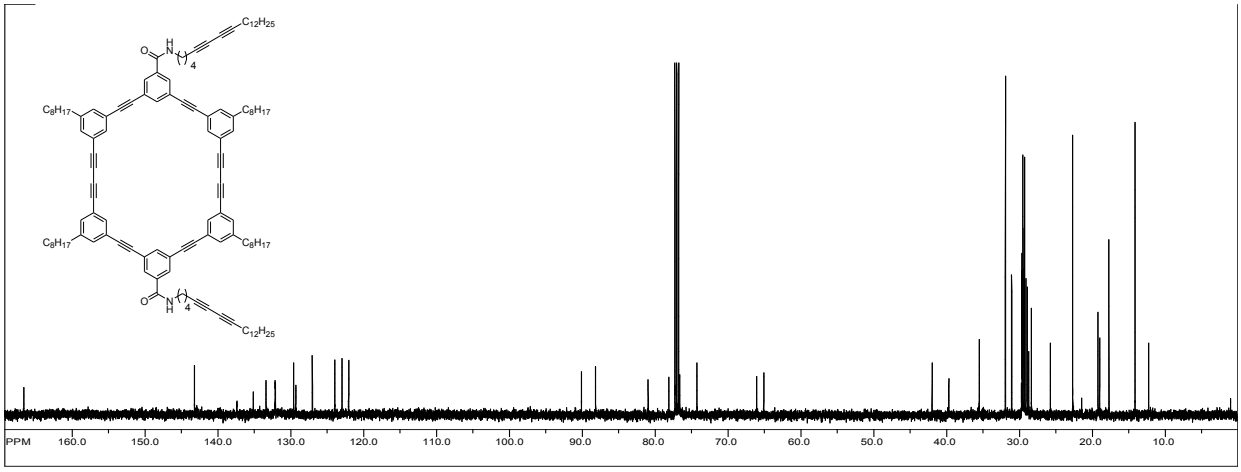


Figure S18. ^{13}C NMR of PAM 2 in $CDCl_3$

4- Gelation Properties

Table S1. Gelation properties of PAM 2 at 1.0 w/v %

Solvent	Observation
toluene	S
1,2-dichlorobenzene	G
benzene	G
pyridine	S
tetrahydrofuran	P
methanol	I
acetone	G
ethyl acetate	G
acetonitrile	P
cyclohexane	G
1,4-dioxane	P
hexanes	G
1,2-dichloroethane	PG
chloroform	S
ethanol	I
dichloromethane	S

S = Soluble

G = Gelation

P = Precipitate

I = Insoluble

PG = Partial gelation

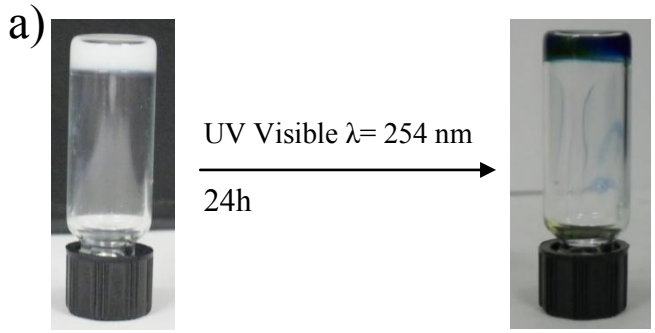


Figure S19. a) Organogel of PAM **2** in ethyl acetate (10 mg/mL) before and after the topochemical polymerization, b) Solution of PAM **2** organogel in $CHCl_3$ after the topochemical polymerization.

5- XRD analysis

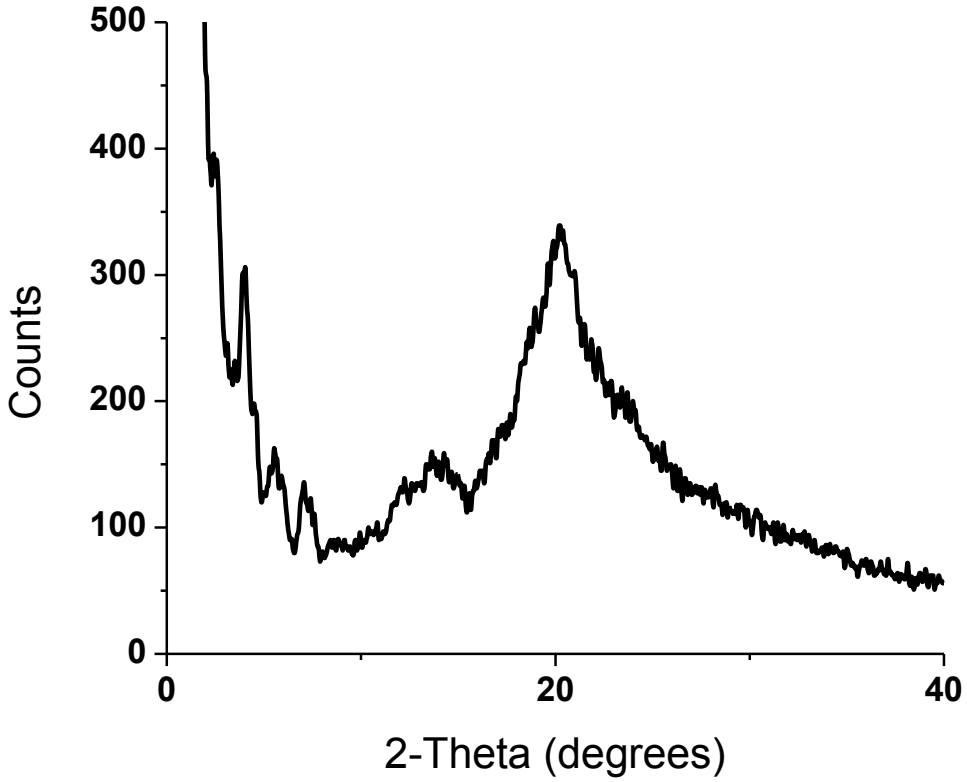


Figure S20. X-ray diffraction spectrum of PAM 2 gel in ethyl acetate (10 mg/mL)

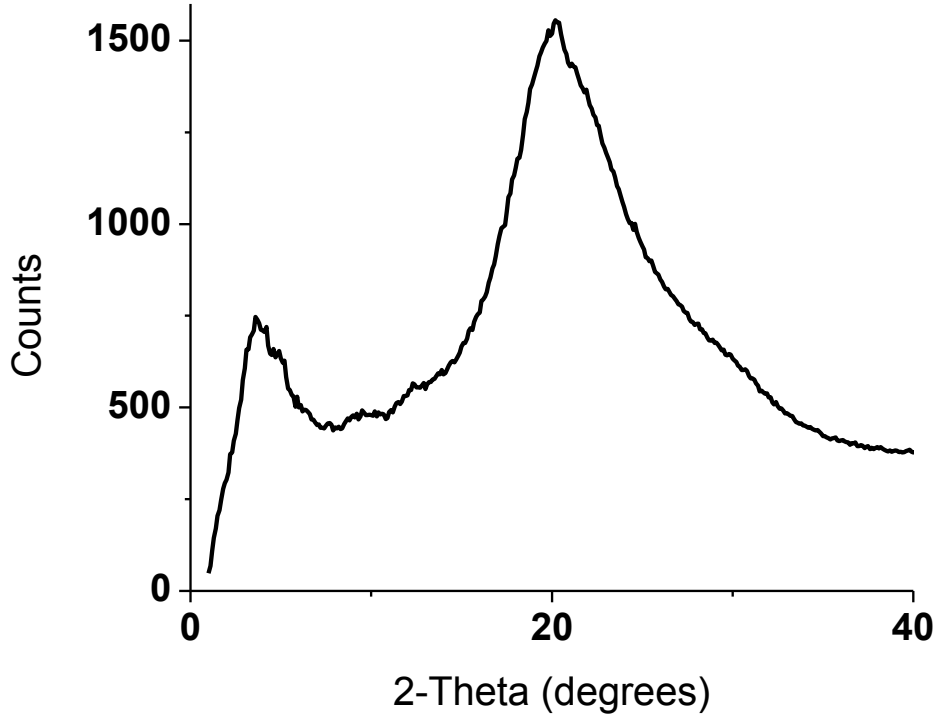


Figure S21. X-Ray diffraction spectrum of SEC-purified PDA

6- UV Visible spectroscopy

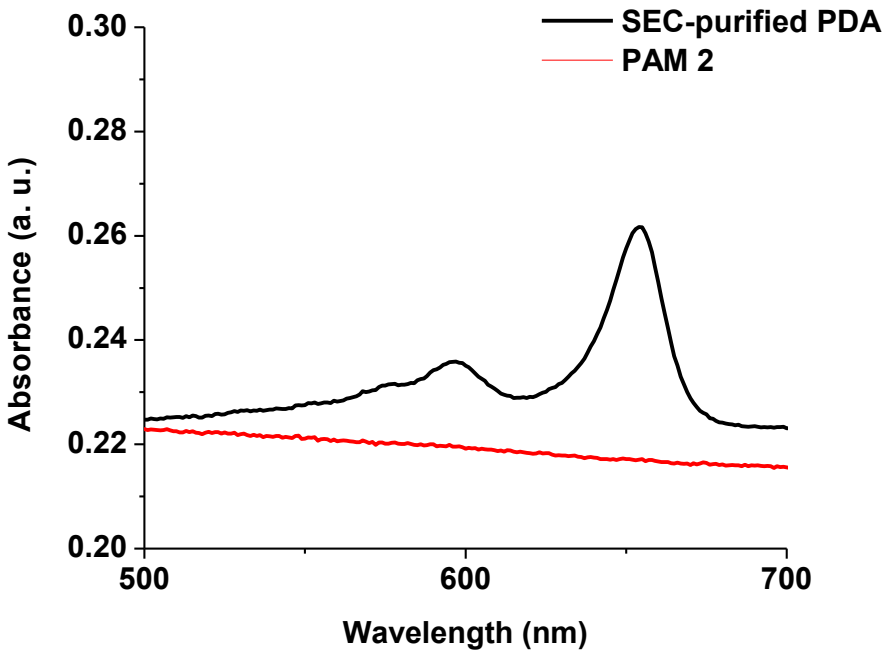


Figure S22. UV Visible spectrum of PAM 2 and SEC-purified PDA in $CHCl_3$

7- SEC chromatography

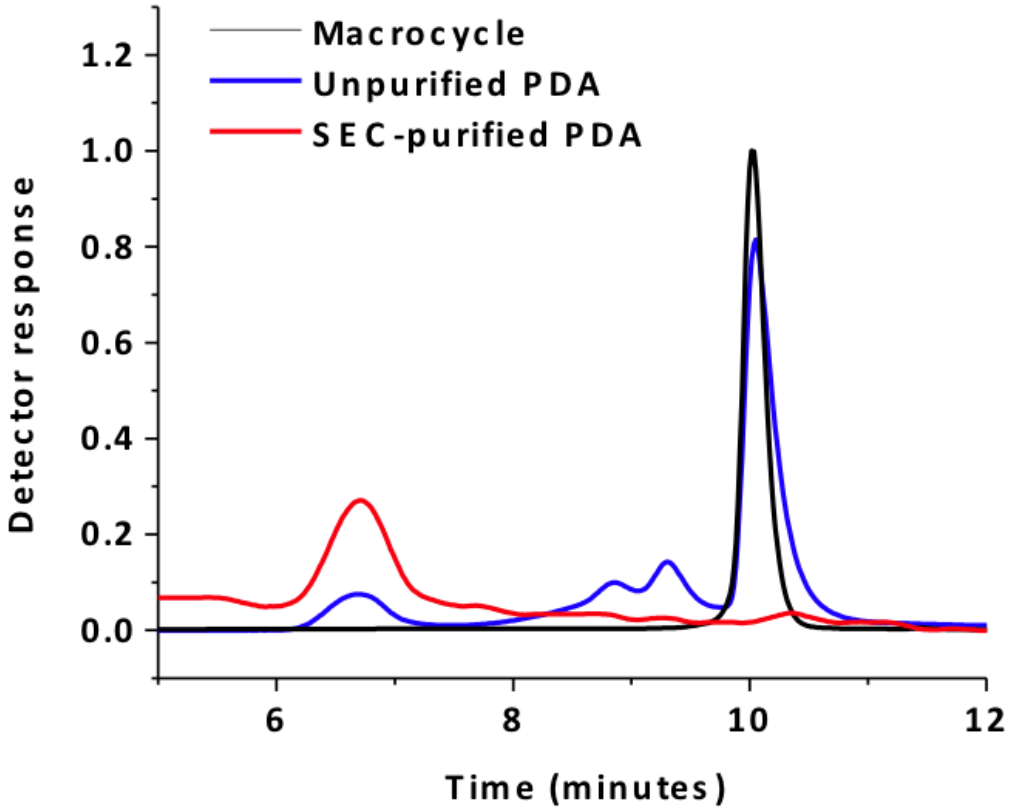


Figure S23. SEC chromatograms of PAM 2 before (black) and after (blue) UV irradiation. The red trace corresponds to the SEC-purified blue material.

9. Theoretical representation

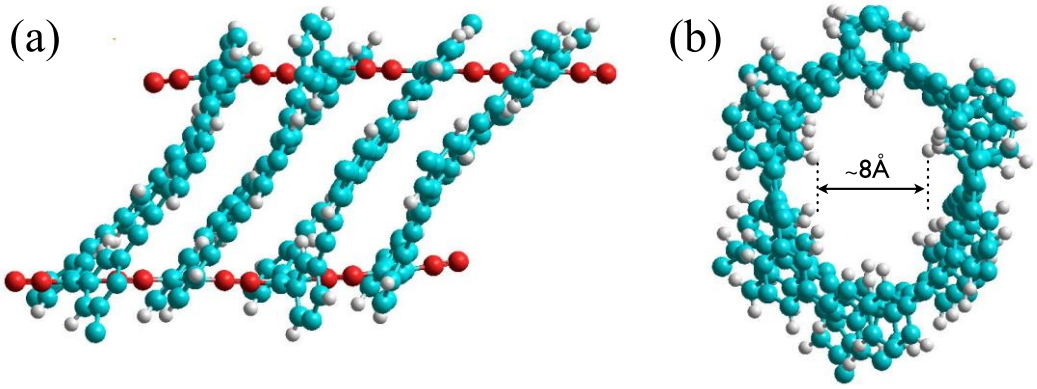


Figure S24. Models of side view (a) and front view (b) of the nanotube after the topochemical polymerizations. Red spheres on (a) are the alkyne carbon atoms of the PDA chains. Peripheral groups have been omitted for clarity.