Electronic Supplementary Information (ESI)

CONTENTS

1) General

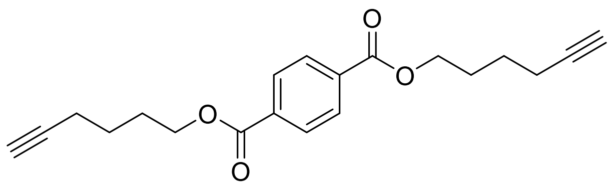
All reagents are commercially available and used without further purification unless otherwise noted. All polymerization reactions were carried out under nitrogen atmosphere. 200-300 mesh silica gel (Qingdao, China) was employed for chromatography, ^{1}H and ^{13}C NMR spectra were recorded at 400 MHz with Bruker ARX 400 spectrometer, 500 MHz with Bruker-500 MHz NMR (AVANCE III) or 600 MHz with Bruker-600 MHz NMR (AVANCE NEO). Chemical shifts were reported in ppm using tetramethylsilane as the internal standard. Size exclusion chromatography (SEC) for the analysis of number-average molecular weights M_n and M_w/M_n of the polymers was carried out with Waters 515 GPC instrument using THF as eluent at a flow rate of 1.0 mL/min at 35 °C. The GPC calibration curve was obtained with linear polystyrene standards. Thermal gravimetric analysis (TGA) was conducted on a Q600-SDT thermogravimetric analyzer at a heating rate of 10 °C/min under a nitrogen atmosphere of 100 mL/min. Differential scanning calorimetry (DSC) was performed on a Q100 differential scanning calorimeter at a heating rate of 10 °C/min under a nitrogen atmosphere of 50 mL/min. Data of the endothermic thermograms were recorded from the second scan and analyzed with a TA Universal Analysis software. HRMS data were obtained on Brucker Apex IV FTMS spectrometer. MALDI-TOF mass spectra were recorded on a Bruker BIFLEX-III spectrometer equipped with a 337 nm laser in the linear mode using *α*-cyano-4-hydroxycinnamic acid as a matrix. Enantioselectivities were measured with HPLC (Agilent Technologies 1200 series) with a Daicel chiral column, eluted with *n*-hexane and isopropanol. Optical rotations were measured with JH-P300 polarimeter (Shanghai Precious Instrument). IR spectra are performed neat on an FT-IR spectrophotometer and reported in wave numbers, cm^{-1}.

2) Preparation of the monomers

Synthesis and characterization of dialkyne monomers M1-M12

Monomer **M7** was commercially available and used without further purification. Monomers **M4**,[1] **M5**,[2] **M6**,[3] **M8**,[4] **M9**,[5] **M10**,[6] **M11**[7] were prepared by the methods reported in the previous literatures.

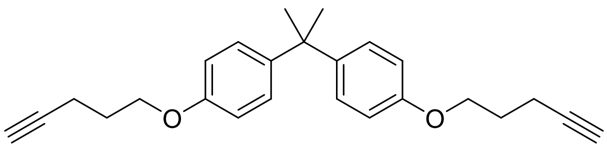
Di(hex-5-yn-1-yl)terephthalate (M1)



To a solution of terephthaloyl dichloride (2.03 g, 10 mmol, 1 equiv) and 5-hexyn-1-ol (2.16 g, 22 mmol, 2.2 equiv), in anhydrous DCM (20 mL) at 0 °C was added successively DMAP (0.244 g, 2 mmol, 0.2 equiv) and pyridine (4.8 mL, 60 mmol, 6 equiv). The reaction mixture was allowed to warm to room temperature and stirred for 1 h, and 10 mL of water was added to quench the reaction. The crude reaction mixture was extracted with dichloromethane (3×20 mL). The combined organic phases were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The resulting residue was purified by SiO_2 gel column chromatography (1:10 EtOAc/Petroleum ether) which afforded di(hex-5-yn-1-yl)terephthalate **(M1)** as a white solid (1.94 g, 59% yield), mp = 87-88 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 4H), 4.38 (t, *J* = 6.4 Hz, 4H), 2.30 (td, *J* = 6.9, 2.0 Hz, 4H), 1.99 (d, *J* = 2.0 Hz, 2H), 1.97 – 1.88 (m, 4H), 1.75 – 1.67 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 165.74, 134.03, 129.48, 83.72, 68.84, 64.86, 27.65, 24.97, 18.07. HRMS (ESI) calcd for $C_{20}H_{22}O_4Na$ $[M+Na]^+$ 349.1416, found 349.1409.

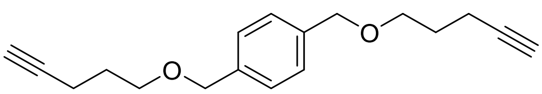
4,4'-(propane-2,2-diyl)bis((pent-4-yn-1-yloxy)benzene) (M2)



To a mixture of biphenyl-4,4’-diol (1.14 g, 5 mmol, 1 equiv) and K_2CO_3 (1.04 g, 7.5 mmol, 1.5 equiv), in anhydrous DMF (20 mL) was added 5-chloro-1-pentyne (1.538 g, 15 mmol, 3 equiv). The reaction mixture was stirred for 24 hours at 80 °C. After the reaction mixture was cooled to rt, 10 mL of water was added to quench the reaction. The crude reaction mixture was extracted with dichloromethane (3×20 mL). The combined organic phases were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The resulting residue was purified by SiO_2 gel column chromatography (1:10 EtOAc/petroleum ether) which afforded 4,4'-(propane-2,2-diyl)bis((pent-4-yn-1-yloxy) benzene) **(M2)** as a vigorous colorless liquid (1.15 g, 64% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (d, *J* = 8.3 Hz, 4H), 6.80 (d, *J* = 8.3 Hz, 4H), 4.03 (t, *J* = 5.9 Hz, 4H), 2.39 (td, *J* = 5.9, 2.2 Hz, 4H), 2.02 – 1.92 (m, 6H), 1.63 (s, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.65, 143.16, 127.69, 113.79, 83.58, 68.77, 65.96, 41.64, 31.03, 28.24, 15.18. HRMS (ESI) calcd for $C_{25}H_{28}O_2Na$ $[M+Na]^+$ 383.1987, found 383.1983.

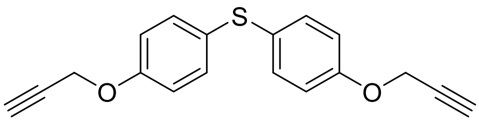
1,4-bis((pent-4-yn-1-yloxy)methyl)benzene (M3)



Under N_2 atmosphere, to a solution of 1,4-phenylenedimethanol (0.69 g, 5 mmol, 1 equiv) in anhydrous DMF (15 mL) at 0 °C was added NaH (60% dispersion in mineral oil, 1.2 g, 30 mmol, 6 equiv). The mixture was stirred for 2 hours at 0 °C. Then 5-chloro-1-pentyne (2.05 g, 20 mmol, 4 equiv) was added, and the reaction mixture was stirred overnight at rt 5 mL of water was added at 0 °C to quench the reaction. The crude reaction mixture was extracted with dichloromethane (3×20 mL). The combined organic phases were washed with water (2×80 mL) and dried over Na_2SO_4, filtered and concentrated *in vacuo*. The resulting residue was purified by SiO_2 gel column chromatography (1:40 EtOAc/petroleum ether) which afforded 1,4-bis((pent-4-yn-1-yloxy)methyl)benzene **(M3)** as a pale yellow liquid (0.46 g, 34% yield).

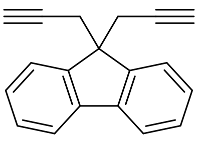
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (s, 4H), 4.51 (s, 4H), 3.56 (t, *J* = 6.1 Hz, 4H), 2.32 (td, *J* = 7.1, 1.9 Hz, 4H), 1.94 (t, *J* = 1.9 Hz, 2H), 1.83 (tt, *J* = 7.1, 6.1 Hz, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.78, 127.67, 83.94, 72.74, 68.60, 68.43, 28.63, 15.27. HRMS (ESI) calcd for $C_{18}H_{22}O_2Na$ $[M+Na]^+$ 293.1517, found 293.1512.

Bis(4-(prop-2-yn-1-yloxy)phenyl)sulfane (M4)[1]



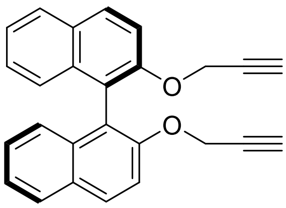
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.8 Hz, 4H), 6.91 (d, *J* = 8.8 Hz, 4H), 4.67 (d, *J* = 2.4 Hz, 4H), 2.52 (t, *J* = 2.4 Hz, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.88, 132.63, 128.29, 115.70, 78.26, 75.73, 55.86.

9,9-di(prop-2-yn-1-yl)-9*H*-fluorene (M5)[2]



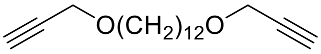
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.6 Hz, 2H), 7.73 (d, *J* = 7.5 Hz, 2H), 7.40 (td, *J* = 7.5, 1.2 Hz, 2H), 7.33 (td, *J* = 7.5, 1.2 Hz, 2H), 2.84 (d, *J* = 2.7 Hz, 4H), 2.02 (t, *J* = 2.7 Hz, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.40, 139.98, 128.05, 127.27, 123.79, 119.90, 80.94, 70.67, 49.94, 27.49.

(*R*)-2,2'-bis(prop-2-yn-1-yloxy)-1,1'-binaphthalene (M6)[3]



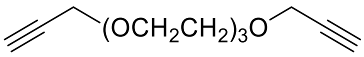
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 9.0 Hz, 2H), 7.87 (d, *J* = 8.1 Hz, 2H), 7.58 (d, *J* = 9.0 Hz, 2H), 7.34 (ddd, *J* = 8.1, 6.7, 1.3 Hz, 2H), 7.21 (ddd, *J* = 8.1, 6.6, 1.3 Hz, 2H), 7.13 (dd, *J* = 8.6, 1.2 Hz, 2H), 4.60 (t, *J* = 2.6 Hz, 4H), 2.38 (t, *J* = 2.4 Hz, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.14, 133.92, 129.75, 129.37, 127.89, 126.38, 125.57, 124.05, 120.59, 116.01, 79.28, 75.18, 57.24.

1,12-bis(prop-2-yn-1-yloxy)dodecane (M8)[4]



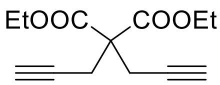
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13 (d, *J* = 2.0 Hz, 4H), 3.51 (t, *J* = 6.7 Hz, 4H), 2.42 (t, *J* = 2.0 Hz, 2H), 1.59 (p, *J* = 6.9 Hz, 4H), 1.39 – 1.23 (m, 16H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 80.05, 74.00, 70.30, 57.98, 29.54, 29.49, 29.41, 26.07.

4,7,10,13-tetraoxahexadeca-1,15-diyne (M9)[5]



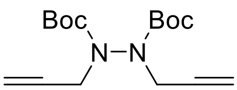
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.21 (d, *J* = 2.0 Hz, 4H), 3.75 – 3.62 (m, 12H), 2.43 (t, *J* = 2.0 Hz, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 79.65, 74.47, 70.59, 70.41, 69.10, 58.39.

Diethyl 2,2-di(prop-2-yn-1-yl)malonate (M10)[6]



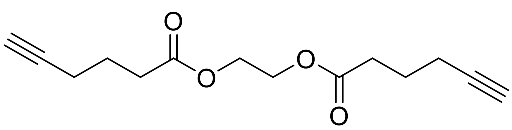
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.23 (q, *J* = 7.1 Hz, 4H), 3.00 (d, *J* = 2.7 Hz, 4H), 2.03 (t, *J* = 2.7 Hz, 2H), 1.27 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 168.58, 78.42, 71.64, 62.06, 56.23, 22.48, 13.99.

***Di*-tert-butyl 1,2-di(prop-2-yn-1-yl)hydrazine-1,2-dicarboxylate (M11)**[7]



^{1}H NMR (400 MHz, $CDCl_3$) δ 4.62 – 3.96 (m, 4H), 2.26 (t, *J* = 2.5 Hz, 2H), 1.68 – 1.19 (m, 18H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.95, 82.20, 81.91, 78.73, 78.18, 72.68, 72.38, 72.14, 41.54, 39.36, 28.13, 28.07.

Ethane-1,2-diyl bis(hex-5-ynoate) (M12)

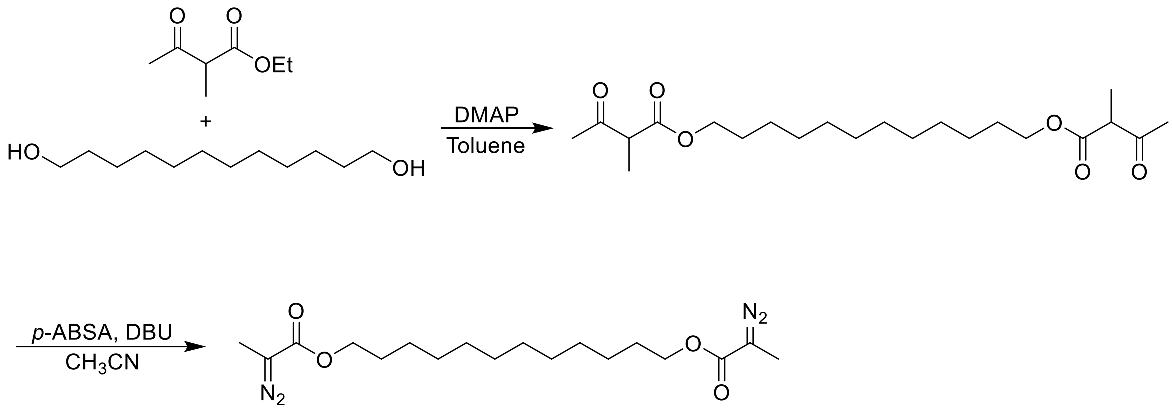


Under N_2 atmosphere, to a solution of hex-5-ynoic acid (1.23 g, 11 mmol, 2.2 equiv) in anhydrous DMF (20 mL) was added K_2CO_3 (1.52 g, 11 mmol, 2.2 equiv) and 1,2-dibromoethane (0.94 g, 5 mmol, 1 equiv). The reaction was stirred at rt for 60 h. 10 mL of water was added to quench the reaction. The crude reaction mixture was extracted with dichloromethane (3×20 mL). The combined organic phases were washed with water (2×20 mL) to get rid of DMF, and then dried over Na_2SO_4, filtered and concentrated *in vacuo*. The resulting residue was purified by SiO_2 gel column chromatography (1:10 EtOAc/Petroleum ether) which afforded ethane-1,2-diyl bis(hex-5-ynoate) **(M12)** as an orange liquid (0.93 g, 74% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.30 (s, 4H), 2.49 (t, *J* = 7.4 Hz, 4H), 2.28 (td, *J* = 6.9, 2.6 Hz, 4H), 1.98 (t, *J* = 2.7 Hz, 2H), 1.86 (p, *J* = 7.1 Hz, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.79, 83.14, 69.18, 62.08, 32.66, 23.48, 17.77. HRMS (ESI) calcd for $C_{14}H_{18}O_4Na$ $[M+Na]^+$ 273.1103, found 273.1096.

Synthesis and characterization of diazo monomers N1 & N2

Dodecane-1,12-diyl bis(2-diazopropanoate) (N1)



N1

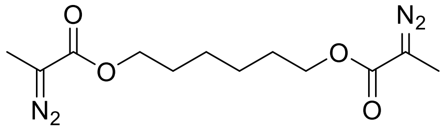
Dodecane-1,12-diol (6.07 g, 30 mmol, 1 equiv), DMAP (3.70 g, 30.3 mmol, 1.01 equiv), ethyl 2-methylacetoacetate (10.08 g, 70 mmol, 2.33 equiv) and toluene (27 mL) were added to a 100 mL round bottom flask. Distillation devices were set up and the reaction mixture was heated at 170-180 °C. The azeotrope of toluene and ethanol was distilled. Thin layer chromatography (TLC) was employed to detect the reaction progress until dodecane-1,12-diol was fully converted. The reaction mixture was allowed to cool to room temperature and filtered through flash column chromatography on SiO_2 gel (1:1 EtOAc/petroleum ether) to get rid of DMAP. Then the solvent was evaporated *in vacuo* and the crude product was purified by SiO_2 gel column chromatography (1:10 EtOAc/petroleum ether) which afforded dodecane-1,12-diyl bis(2-methyl-3-oxobutanoate) as a pale yellow liquid (10.60 g, 89%).

Under N_2 atmosphere, DBU (23.74 mL, 159 mmol, 6 equiv) was slowly added to a mixture of dodecane-1,12-diyl bis(2-methyl-3-oxobutanoate) (10.58 g, 26.5 mmol, 1 equiv), *p*-ABSA (19.10 g, 79.6 mmol, 3 equiv) and acetonitrile (100 mL) at 0 °C in a 500 mL Schlenk flask. The reaction mixture was allowed to warm to room temperature and stirred overnight. 60 mL of saturated NH_4Cl solution was added to quench the reaction. The crude reaction mixture was extracted with dichloromethane (3×60 mL). The combined organic phases were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The resulting residue was purified by SiO_2 gel column chromatography (1:40 EtOAc/Petroleum ether) which afforded dodecane-1,12-diyl bis(2-diazopropanoate) (**N1**) as a yellow solid (6.179 g, 64%), mp = 40-41 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 (t, *J* = 6.7 Hz, 4H), 1.96 (s, 6H), 1.62 (p, *J* = 6.8 Hz, 4H), 1.37 – 1.21 (m, 16H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 168.04, 64.93, 29.46, 29.43, 29.18, 28.80, 25.78, 8.39. HRMS (ESI): calcd for $C_{18}H_{30}N_4O_4Na$ $[M+Na]^+$ 389.2165, found 389.2156.

Monomer **N2** was prepared as the same procedure for the preparation of **N1**. Yield = 46% in two steps (84% × 55%), yellow liquid.

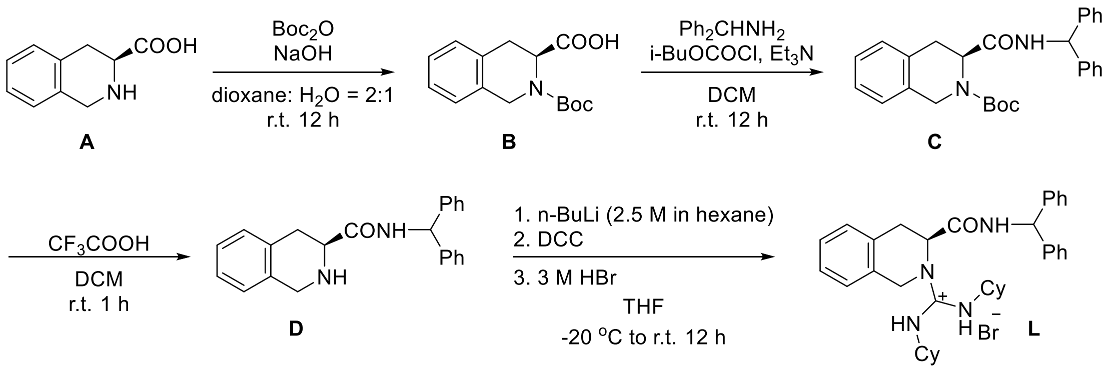
Hexane-1,6-diyl bis(2-diazopropanoate) (N2)



^{1}H NMR (400 MHz, $CDCl_3$): δ 4.16 (t, *J* = 6.5 Hz, 4H), 1.97 (s, 6H), 1.67-1.64 (m, 4H), 1.38 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 167.89, 64.62, 28.65, 25.43, 8.33. HRMS (ESI) calcd for $C_{12}H_{18}N_4O_4Na$ $[M+Na]^+$ 305.1226, found 305.1215.

3) Preparation of ligand L6

Synthesis of (*S*)-3-(benzhydrylcarbamoyl)-2-(bis(cyclohexylamino)methylene)-1,2,3,4-tetrahydroisoquinolin-2-ium bromide (L6)[8]



To a solution of (*S*)-1,2,3,4-tetrahydro-3-isoquinolinecarboxlic acid **A** (5.316 g, 30 mmol, 1 equiv) in water-dioxane (v/v = 1/2, 57 mL), NaOH (1 M, 30 mL, 1 equiv) followed by di-*tert*-butyl dicarbonate (7.857 g, 36 mmol, 1.2 equiv) were added at room temperature. The reaction mixture was stirred at room temperature overnight.

Dioxane was evaporated *in vacuo* and dissolved with EtOAc. The pH was adjusted to 2-4 by adding aqueous $KHSO_4$, and the product was extracted with EtOAc (3×30 mL). The solvent was evaporated to give **B** as a white soild. It was used directly in the next step without further purification.

To a solution of **B** in CH_2Cl_2 (60 mL) was added NEt_3 (4.5 mL, 33 mmol, 1.1 equiv), isobutyl carbonochloridate (4.504 g, 33 mmol, 1.1 equiv) at 0 °C under stirring. After 10 min, diphenylmethanamine (6.039 g, 33 mmol, 1.1 equiv) was added. The reaction was allowed to warm to room temperature overnight. The mixture was washed with 1 M $KHSO_4$ (50 mL) solution, saturated $NaHCO_3$ (50 mL) solution, dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The resulting residue was purified by SiO_2 gel column chromatography (1:2 EtOAc/petroleum ether) which afforded **C** as a white solid.

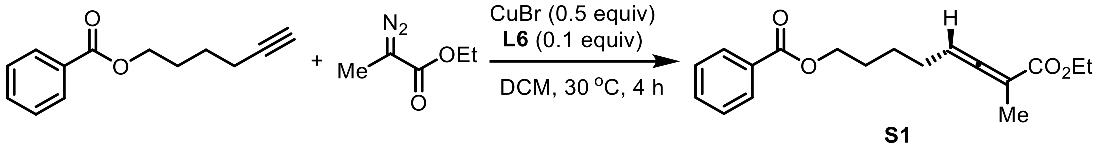
TFA (10 mL) was added to the CH_2Cl_2 (30 mL) solution of **C**, and the solution was stirred for 1 h. The pH value of the mixture was brought into the range of 10-12 by the addition of 2 M NaOH solution. The aqueous phase was extracted with CH_2Cl_2 (3×50 mL). The combined organic phases were dried over anhydrous Na_2SO_4, and concentrated *in vacuo*. The resulting residue was purified by SiO_2 gel column chromatography (1:10 methanol/dichloromethane) which afforded **D** as a white solid. The yield of total three steps is 80%.

n-BuLi (2.5 M in hexane, 4.1 mL, 10.25 mmol, 2.05 equiv) was injected into a solution of **D** (1.712 g, 5 mmol, 1 equiv) in dry THF (20 mL) dropwise over 10 min under nitrogen atmosphere at −20ºC with well stirring. After additional 10 min, a solution of *N*,*N*-dicyclohexylcarbodiimide (1.133 g, 5.5 mmol, 1.1 equiv) in 10 mL of THF was added dropwise within 10 min. The reaction was allowed to warm to room temperature and detected by TLC. After 12 hours, 2 mL of water was added to quench the reaction, and the mixture was evaporated under reduced pressure to get rid of THF, and the pH value of the mixture was brought into the range of 0–1 by the addition of 3 M HBr. The aqueous phase was extracted with CH_2Cl_2 (5×10 mL). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The resulting residue was purified through flash SiO_2 gel chromatography (1:8 methanol/EtOAc) which afforded the guanidinium salt as a pale yellow solid (83% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (d, *J* = 9.0 Hz, 1H), 7.49 (d, *J* = 7.6 Hz, 2H), 7.32 – 7.18 (m, 8H), 7.08 – 6.85 (m, 3H), 6.22 (d, *J* = 8.8 Hz, 1H), 5.63 (d, *J* = 6.4 Hz, 1H), 4.58 (q, *J* = 14.1 Hz, 2H), 3.44 – 3.20 (m, 3H), 3.08 (dd, *J* = 15.2, 9.0 Hz, 1H), 1.95 – 1.00 (m, 22H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.14, 169.97, 157.58, 141.76, 140.49, 133.62, 132.67, 128.58, 128.50, 128.14, 128.09, 127.61, 127.43, 127.30, 127.16, 126.93, 125.71, 60.37, 58.19, 57.42, 55.63, 49.99, 33.78, 32.98, 32.85, 25.22, 25.06, 24.63, 21.02, 14.16.

4) Model reaction

Model product **S1** was prepared by the method from previous literature.[8]



S1

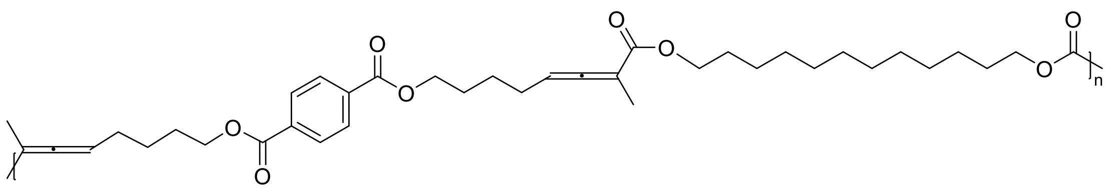
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.7 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 5.54 – 5.40 (m, 1H), 4.34 (t, *J* = 6.5 Hz, 2H), 4.24 – 4.07 (m, 2H), 2.19 (q, *J* = 7.0 Hz, 2H), 1.92 – 1.78 (m, 5H), 1.72 – 1.53 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 209.98, 167.80, 166.50, 132.78, 130.29, 129.42, 128.24, 95.85, 93.16, 64.62, 60.72, 27.81, 27.44, 25.13, 15.12, 14.18.

5) General procedures of polymerization

Ligand **L6** (12.59 mg, 0.02 mmol, 0.2 equiv), CuBr (14.35 mg, 0.1 mmol, 1 equiv), dialkynes **M** (0.1 mmol, 1 equiv) and bis-*α*-diazoester **N** (0.1 mmol, 1 equiv) were added successively to a flame-dried 10 mL Schlenk tube. The reaction tube was degassed three times with N_2 and dry dichloromethane (1 mL) was added by syringe. The reaction was stirred for 6 hours at 30 °C. Then the crude product was filtered through a filter membrane (0.22 μm) to remove deactivated catalyst. The solvent was evaporated *in vacuo*. The molecular weights (M_n) and the polymer dispersity indexes (M_w/M_n) were measured by gel permeation chromatography. The products were then purified by preparative recycling GPC and dried *in vacuo*.

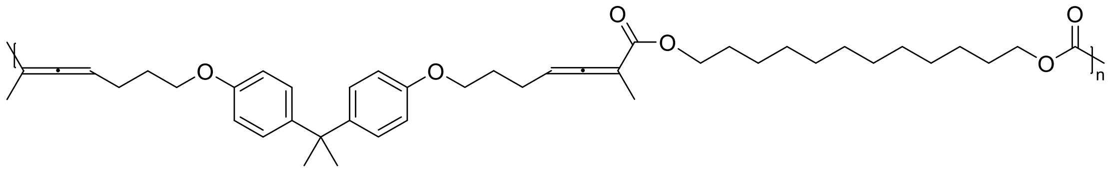
6) ^{1}H and ^{13}C NMR characterization data for the polymers

P1



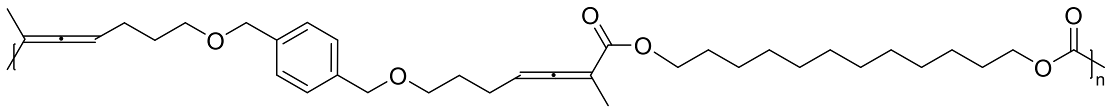
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (s, 4H), 5.46 – 5.42 (m, 2H), 4.33 (t, *J* = 6.5 Hz, 4H), 4.10 – 4.05 (m, 4H), 2.17 (q, *J* = 7.0 Hz, 4H), 1.86 – 1.81 (m, 10H), 1.63 – 1.57 (m, 8H), 1.29 – 1.22 (m, 16H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.03, 167.77, 165.65, 134.08, 129.41, 96.01, 93.08, 65.09, 64.91, 29.47, 29.15, 28.59, 27.84, 27.44, 25.82, 25.17, 15.12.

P2



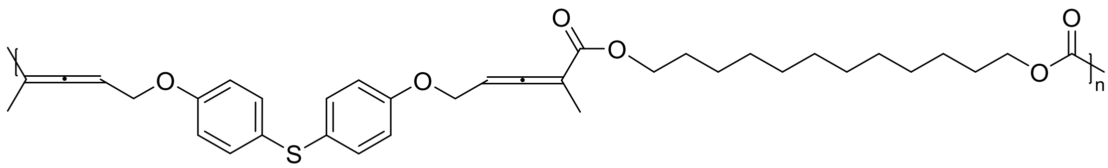
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.7 Hz, 4H), 6.77 (d, *J* = 8.7 Hz, 4H), 5.51 – 5.48 (m, 2H), 4.08 (t, *J* = 6.0 Hz, 4H), 3.98 (t, J = 6.2 Hz, 4H), 2.27 (q, *J* = 7.1 Hz, 4H), 1.94 – 1.88 (m, 4H), 1.84 (d, *J* = 2.8 Hz, 6H), 1.62 – 1.59 (m, 10H), 1.29 – 1.25 (m, 16H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.03, 167.83, 156.77, 143.06, 127.62, 113.79, 96.25, 93.00, 66.75, 64.94, 41.62, 31.04, 29.51, 29.20, 28.62, 28.43, 25.85, 24.44, 15.14.

P3



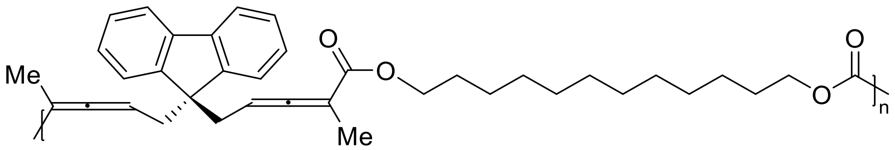
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (s, 4H), 5.48 – 5.45 (m, 2H), 4.48 (s, 4H), 4.09 (t, *J* = 6.7 Hz, 4H), 3.51 (t, *J* = 6.4 Hz, 4H), 2.19 (q, *J* = 7.2 Hz, 4H), 1.84 (d, *J* = 2.9 Hz, 6H), 1.77 – 1.74 (m, 4H), 1.67 – 1.57 (m, 4H), 1.41 – 1.17 (m, 16H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.01, 167.89, 137.84, 127.59, 96.04, 93.24, 72.69, 69.37, 64.90, 29.50, 29.20, 28.87, 28.63, 25.86, 24.58, 15.16.

P4



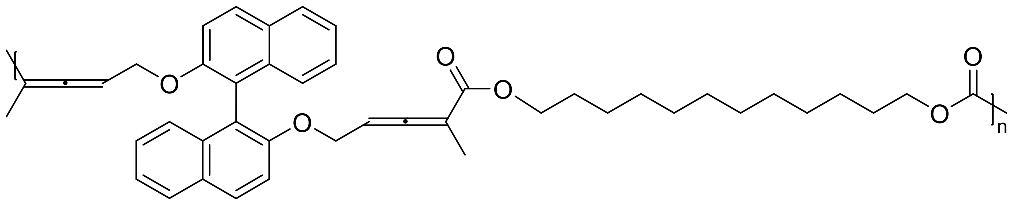
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.4 Hz, 0.7H), 7.17 (d, *J* = 8.4 Hz, 3.3H), 6.90 (d, *J* = 8.4 Hz, 0.7H), 6.80 – 6.75 (d, *J* = 8.4 Hz, 3.3H), 5.61 – 5.55 (m, 2H), 4.61 – 4.51 (m, 4H), 4.11 – 3.95 (m, 4H), 1.80 – 1.74 (m, 6H), 1.57 – 1.49 (m, 4H), 1.31 – 1.09 (m, 16H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.29, 166.97, 157.47, 132.58, 127.89, 115.79, 97.86, 90.62, 65.24, 64.79, 29.48, 29.45, 29.17, 28.55, 25.80, 14.74.

P5



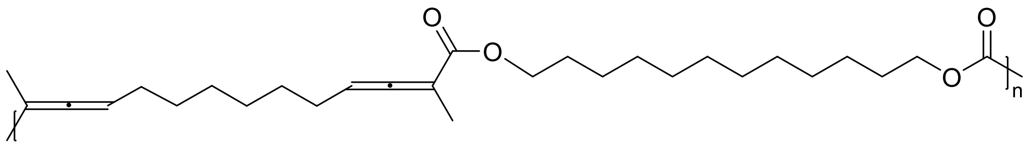
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.3 Hz, 2H), 7.41 (d, *J* = 7.3 Hz, 2H), 7.36 – 7.26 (m, 4H), 4.90 – 4.86 (m, 2H), 4.06 – 3.95 (m, 4H), 2.87 – 2.69 (m, 4H), 1.59 – 1.53 (m, 4H), 1.41 (d, *J* = 2.9 Hz, 6H), 1.32 – 1.21 (m, 16H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.89, 167.65, 148.19, 141.00, 127.53, 127.22, 123.30, 120.01, 95.14, 88.68, 64.88, 54.44, 37.63, 29.53, 29.22, 28.58, 25.84, 14.85.

P6



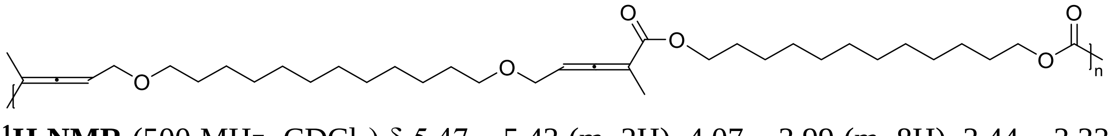
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.9 Hz, 2H), 7.85 (d, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 9.0 Hz, 2H), 7.34 – 7.30 (m, 2H), 7.23 – 7.17 (m, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 5.38 – 5.34 (m, 2H), 4.67 – 4.55 (m, 4H), 4.11 – 3.91 (m, 4H), 1.70 (d, *J* = 2.7 Hz, 6H), 1.53 – 1.45 (m, 4H), 1.39 – 1.15 (m, 16H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 210.02, 167.07, 153.30, 133.99, 129.48, 129.11, 127.79, 126.21, 125.42, 123.80, 120.81, 115.85, 97.30, 91.24, 65.96, 65.10, 29.49, 29.46, 29.14, 28.46, 25.73, 14.64.

P7



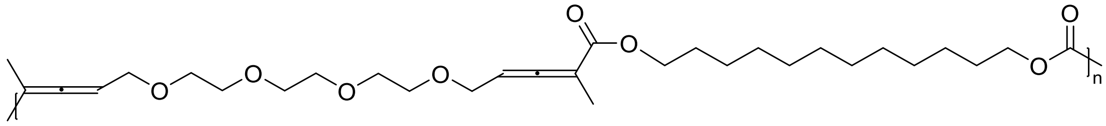
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.46 – 5.42 (m, 2H), 4.13 – 4.09 (m, 4H), 2.09 (q, *J* = 7.1 Hz, 4H), 1.85 (d, *J* = 2.9 Hz, 6H), 1.66 – 1.60 (m, 4H), 1.45 – 1.26 (m, 24H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 209.99, 168.01, 95.60, 93.60, 64.86, 29.53, 29.48, 29.21, 28.74, 28.69, 28.62, 27.88, 25.85, 15.20.

P8



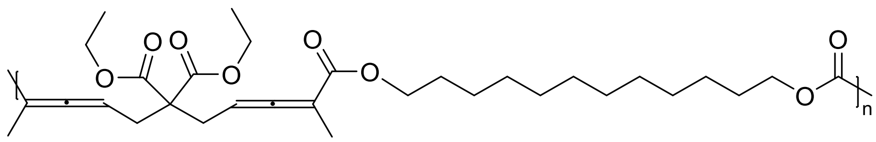
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.47 – 5.43 (m, 2H), 4.07 – 3.99 (m, 8H), 3.44 – 3.33 (m, 4H), 1.81 (d, *J* = 2.9 Hz, 6H), 1.59 – 1.46 (m, 8H), 1.30 – 1.15 (m, 32H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.21, 167.40, 96.50, 91.57, 70.05, 67.49, 65.10, 29.70, 29.59, 29.53, 29.52, 29.22, 28.62, 26.22, 25.85, 14.97.

P9



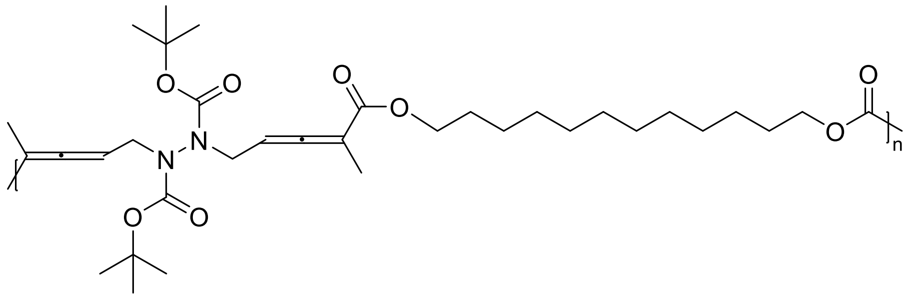
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.49 – 5.44 (m, 2H), 4.09 – 4.02 (m, 8H), 3.63 – 3.52 (m, 12H), 1.81 (d, *J* = 2.9 Hz, 6H), 1.58 – 1.52 (m, 4H), 1.30 – 1.16 (m, 16H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.32, 167.28, 96.60, 91.29, 70.56, 70.50, 69.01, 67.91, 65.13, 29.51, 29.50, 29.19, 28.59, 25.82, 14.94.

P10



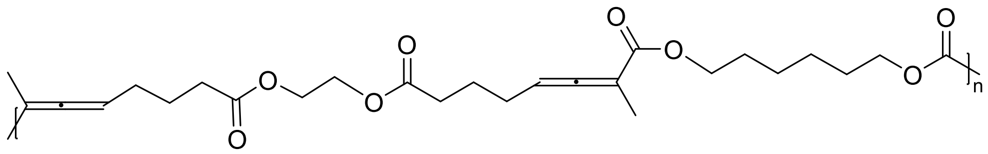
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.25 – 5.17 (m, 2H), 4.20 – 3.95 (m, 8H), 2.91 – 2.64 (m, 4H), 1.79 – 1.71 (m, 6H), 1.62 – 1.52 (m, 4H), 1.32 – 1.10 (m, 22H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 211.36, 169.80, 167.33, 95.78, 87.93, 65.15, 61.53, 57.39, 30.77, 29.56, 29.52, 29.25, 28.59, 25.85, 15.00, 14.02.

P11



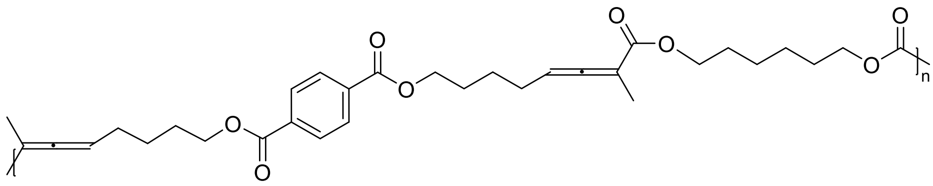
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.66 – 5.44 (m, 2H), 4.18 – 3.96 (m, 8H), 1.82 – 1.78 (m, 6H), 1.61 – 1.50 (m, 4H), 1.47 – 1.33 (m, 18H), 1.30 – 1.16 (m, 16H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 209.75, 166.19, 153.33, 95.94, 89.08, 80.43, 64.24, 47.23, 28.60, 28.56, 28.27, 27.65, 27.25, 24.85, 14.08.

P12



^{1}H NMR (400 MHz, $CDCl_3$) δ 5.47 – 5.41 (m, 2H), 4.27 (s, 4H), 4.11 (t, *J* = 6.8 Hz, 4H), 2.41 (t, *J* = 7.5 Hz, 4H), 2.15 (q, *J* = 7.1 Hz, 4H), 1.86 (d, *J* = 2.9 Hz, 6H), 1.82 – 1.75 (m, 4H), 1.67 – 1.64 (m, 4H), 1.41 – 1.36 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 210.06, 172.95, 167.71, 96.06, 92.72, 64.71, 62.00, 32.99, 28.44, 27.20, 25.44, 23.80, 15.12.

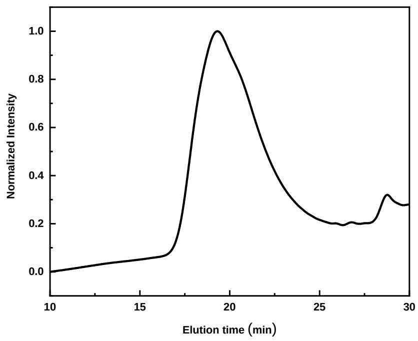
P13



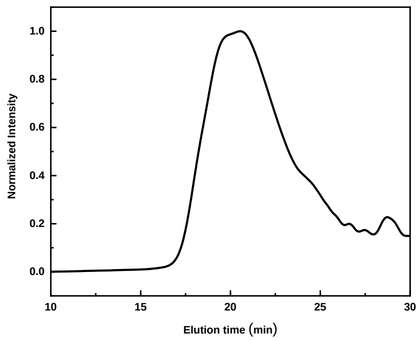
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 4H), 5.50 – 5.45 (m, 2H), 4.36 (t, *J* = 6.5 Hz, 4H), 4.10 (t, *J* = 6.7 Hz, 4H), 2.19 (q, *J* = 7.1 Hz, 4H), 1.91 – 1.81 (m, 10H), 1.69 – 1.58 (m, 8H), 1.41 – 1.34 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 210.06, 167.81, 165.71, 134.04, 129.44, 95.94, 93.17, 65.11, 64.70, 28.47, 27.82, 27.43, 25.45, 25.16, 15.19.

7) SEC traces of polymers

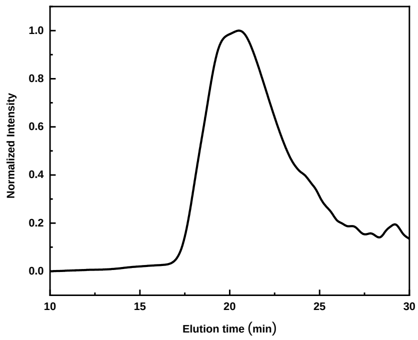
P1 M_n = 22.7 kDa, M_w/M_n = 1.95



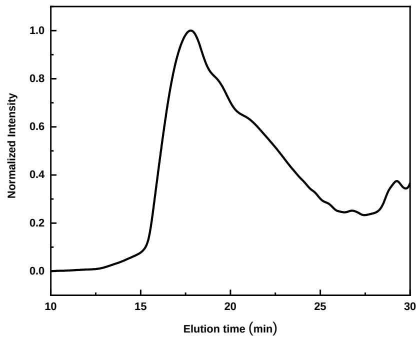
P2 M_n = 14.0 kDa, M_w/M_n = 2.24



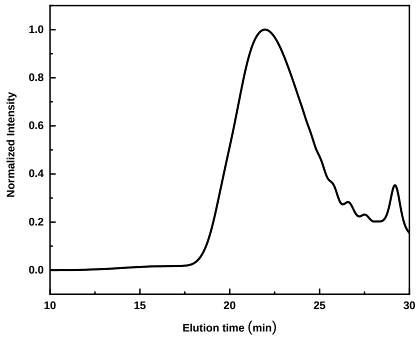
P3 M_n = 13.8 kDa, M_w/M_n = 2.17



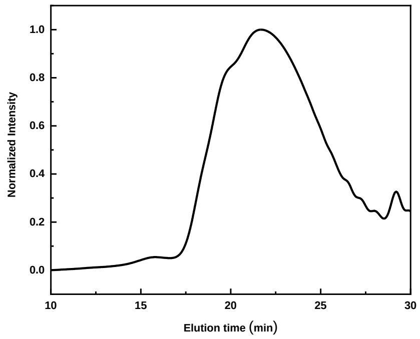
P4 M_n = 22.8 kDa, M_w/M_n = 4.57



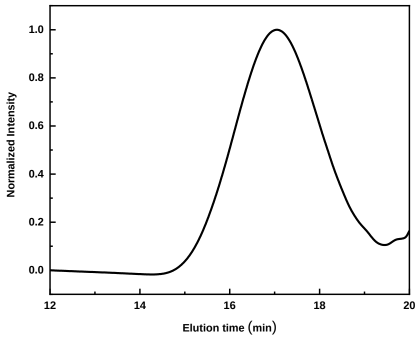
P5 M_n = 8.8 kDa, M_w/M_n = 1.64



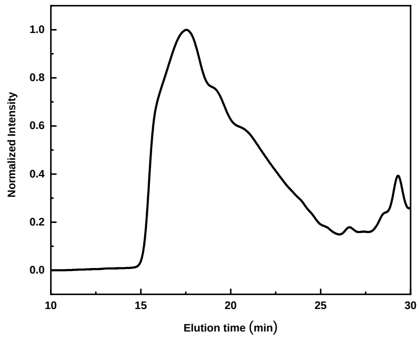
P6 M_n = 7.7 kDa, M_w/M_n = 2.63



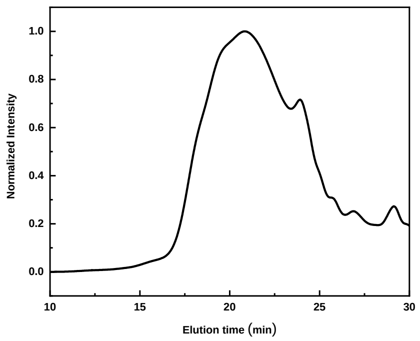
P7 M_n = 11.8 kDa, M_w/M_n = 1.93



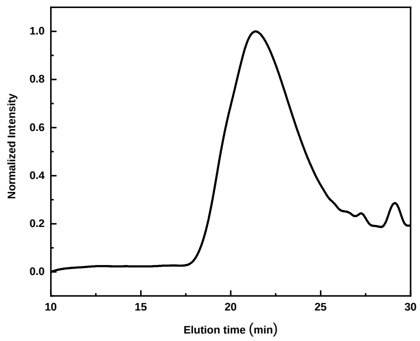
P8 M_n = 30.4 kDa, M_w/M_n = 4.17



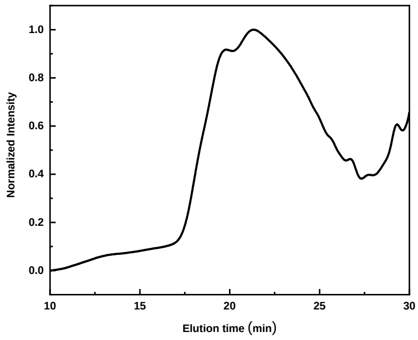
P9 M_n = 12.2 kDa, M_w/M_n = 2.58



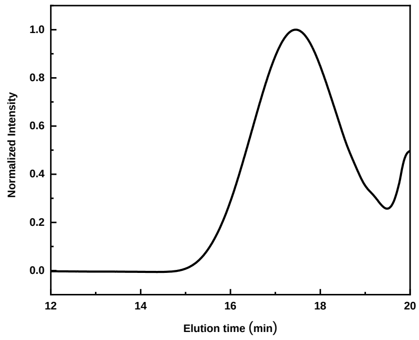
P10 M_n = 10.1 kDa, M_w/M_n = 1.74



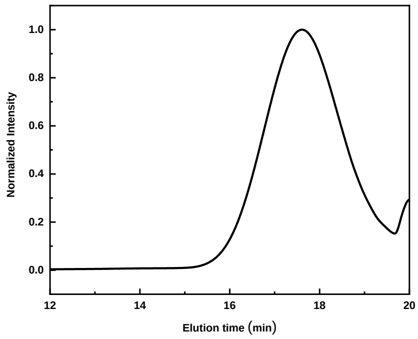
P11 M_n = 8.9 kDa, M_w/M_n = 2.62



P12 M_n = 8.8 kDa, M_w/M_n = 1.81

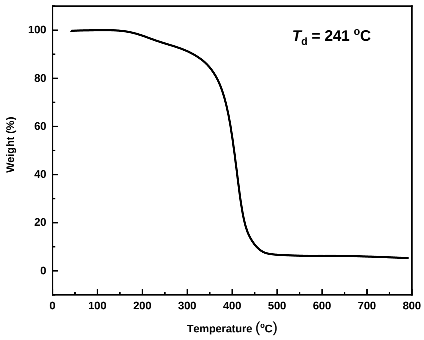


P13 M_n = 7.4 kDa, M_w/M_n = 1.69

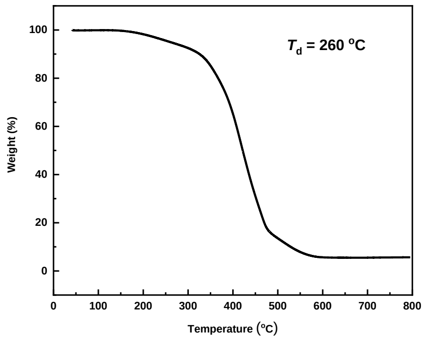


8) TGA data of polymers

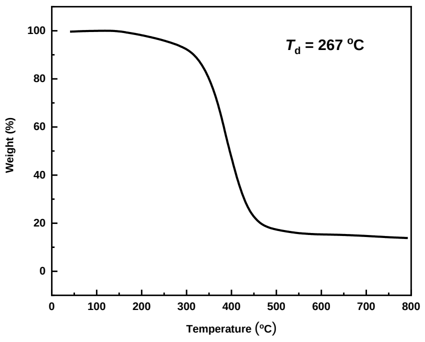
P1



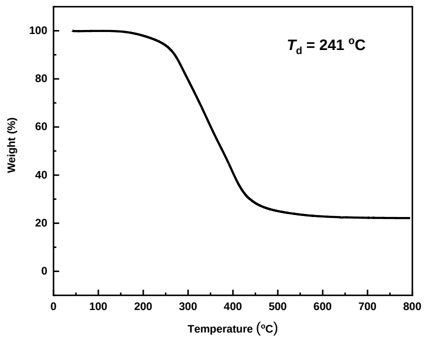
P2



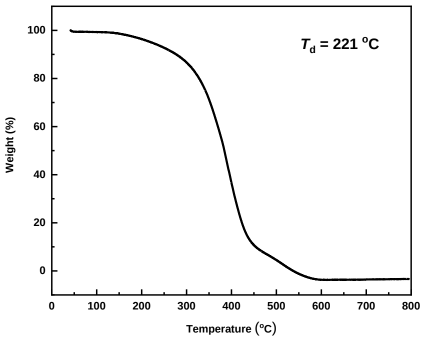
P3



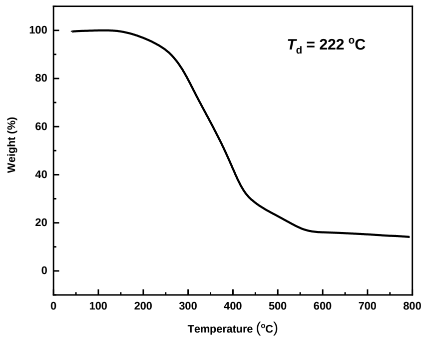
P4



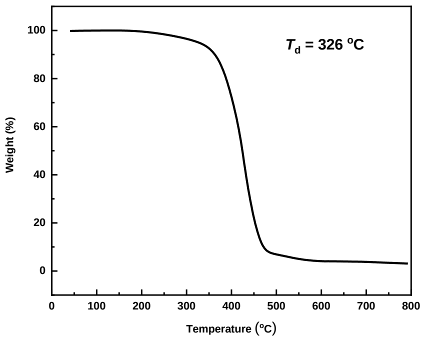
P5



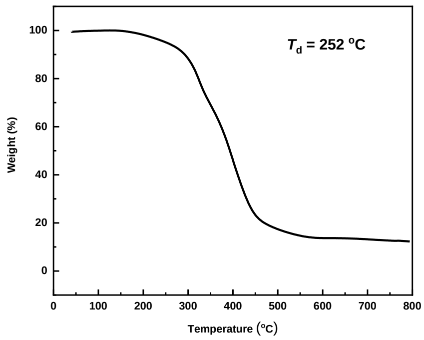
P6



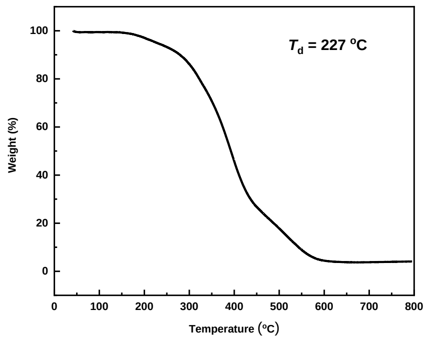
P7



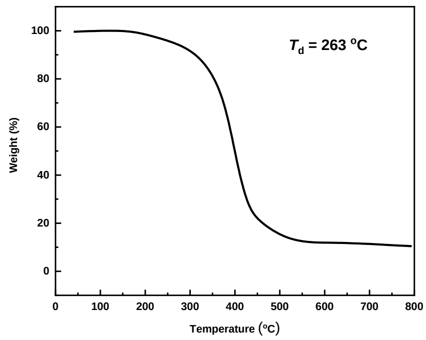
P8



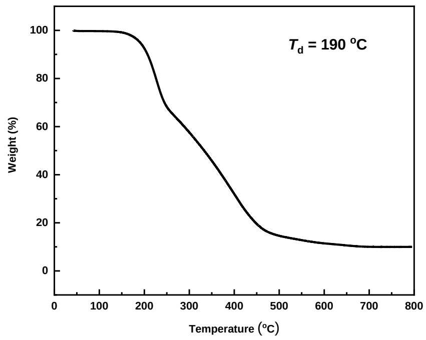
P9



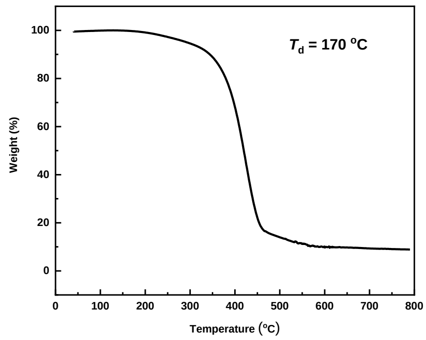
P10



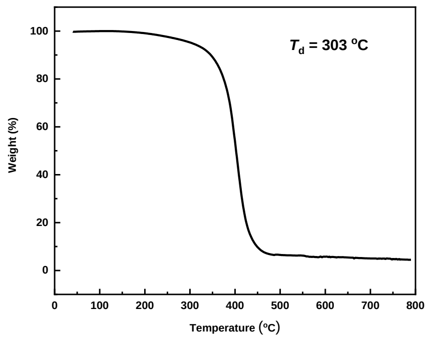
P11



P12

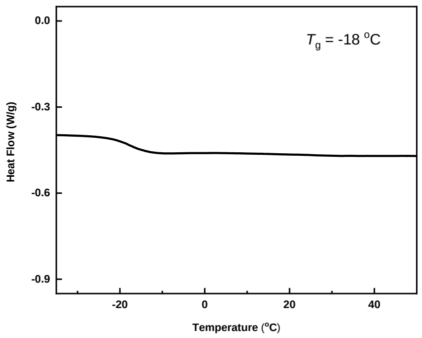


P13

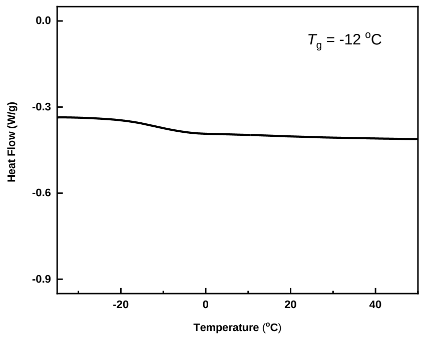


9) DSC data of polymers

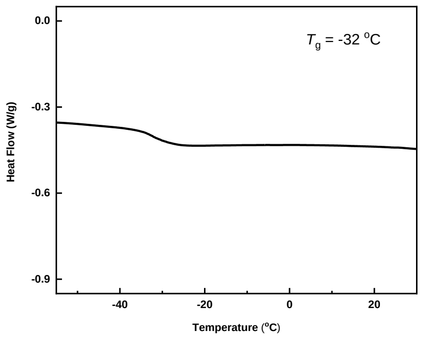
P1



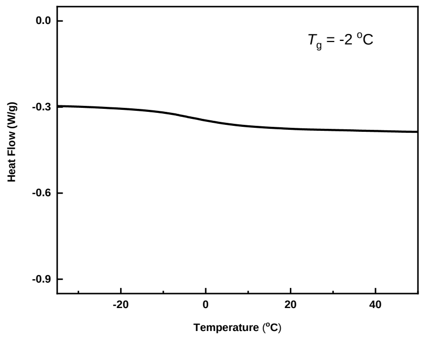
P2



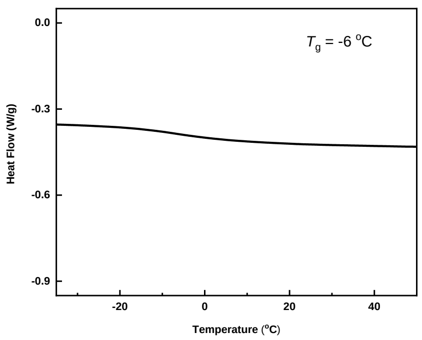
P3



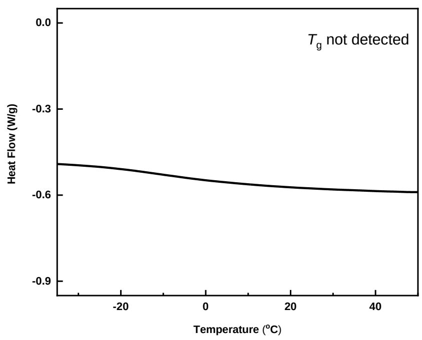
P4



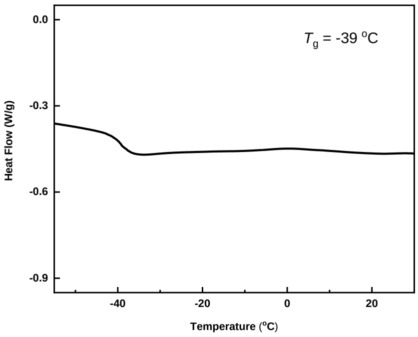
P5



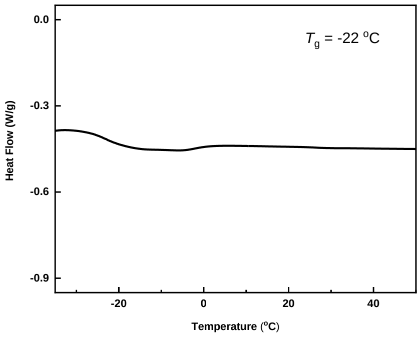
P6



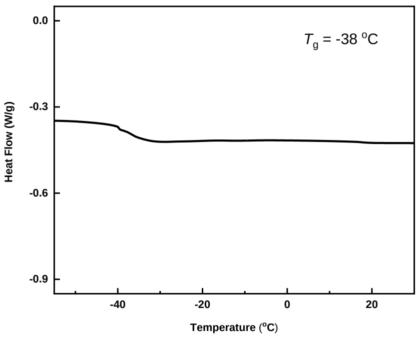
P7



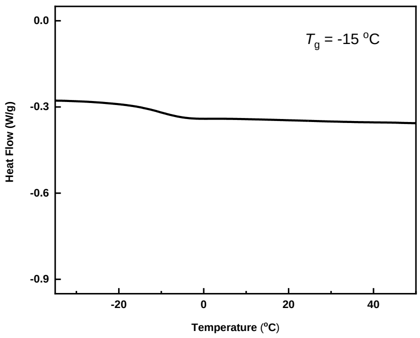
P8



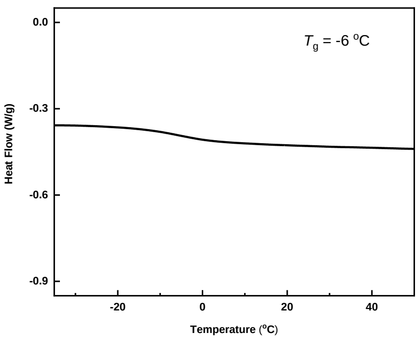
P9



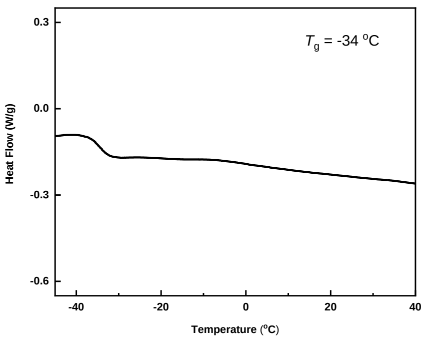
P10



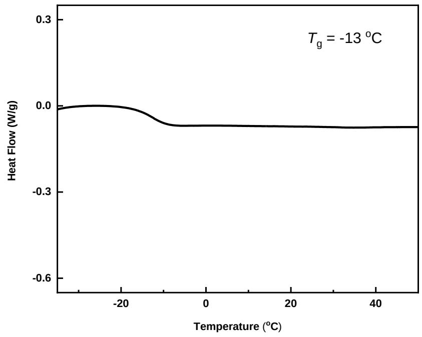
P11



P12



P13



10) MALDI-TOF-MS spectrum of polymer P1

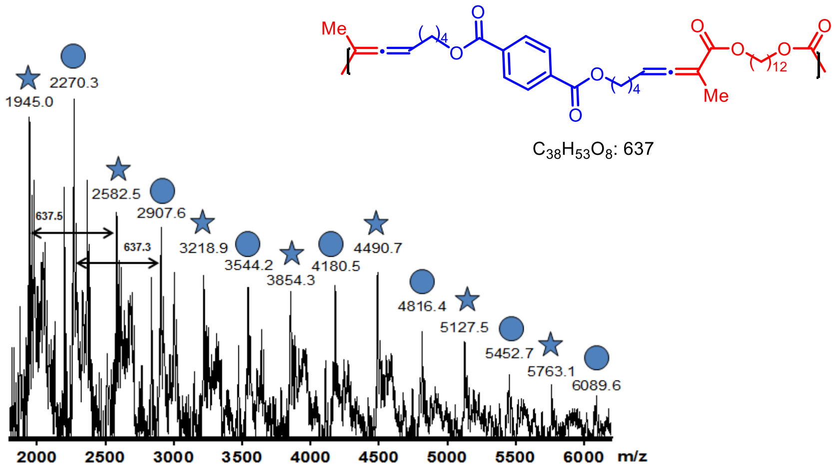
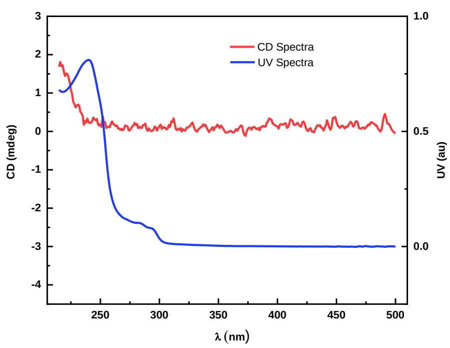


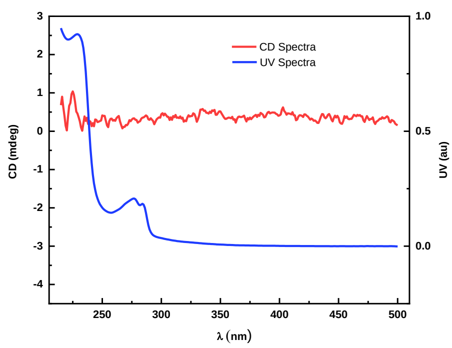
Figure S1. MALDI-TOF-MS spectrum of polymer **P1**

11) CD and UV spectrum

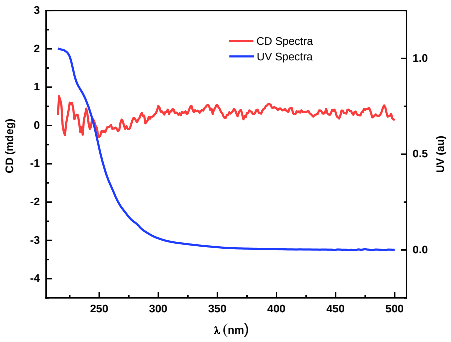
P1 (2.5 mM solution in THF)



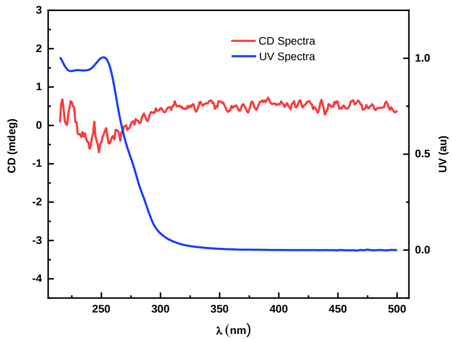
P2 (2.5 mM solution in THF)



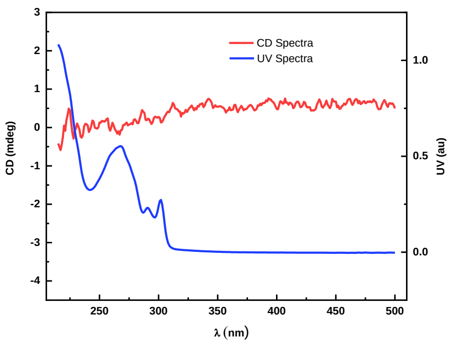
P3 (5.0 mM solution in THF)



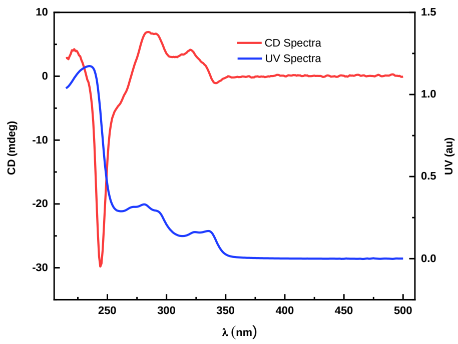
P4 (5.0 mM solution in THF)



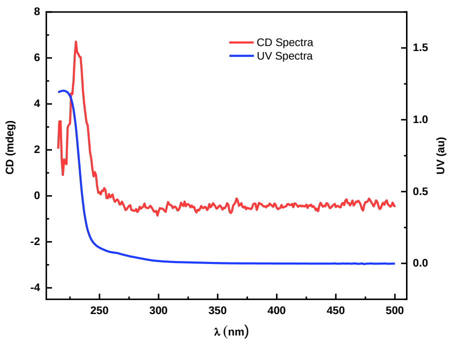
P5 (2.5 mM solution in THF)



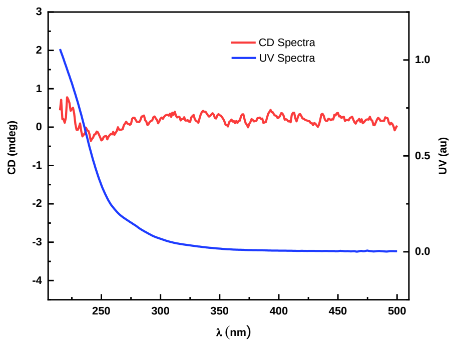
P6 (2.5 mM solution in THF)



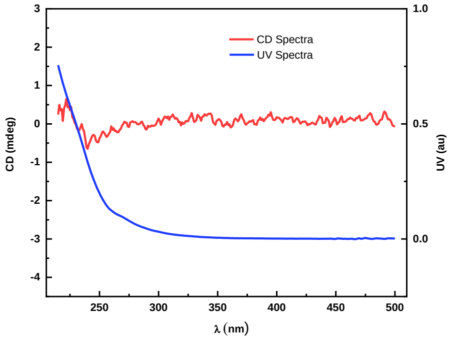
P7 (7.5 mM solution in THF)



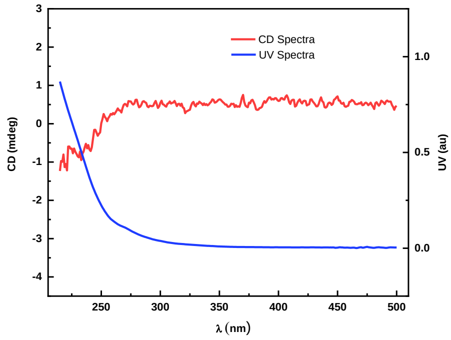
P8 (7.5 mM solution in THF)



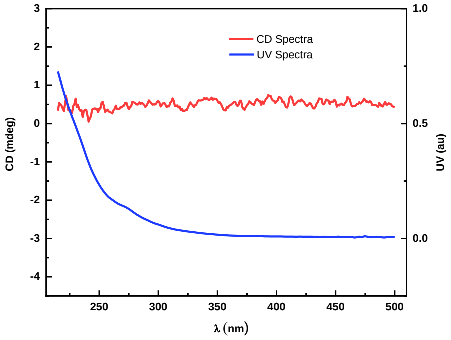
P9 (7.5 mM solution in THF)



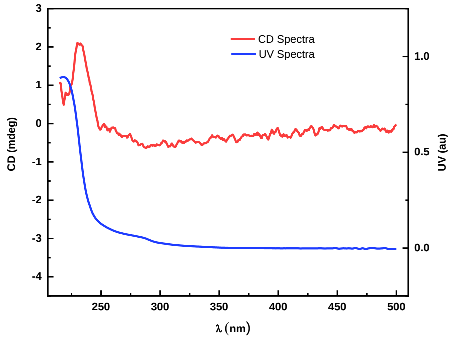
P10 (7.5 mM solution in THF)



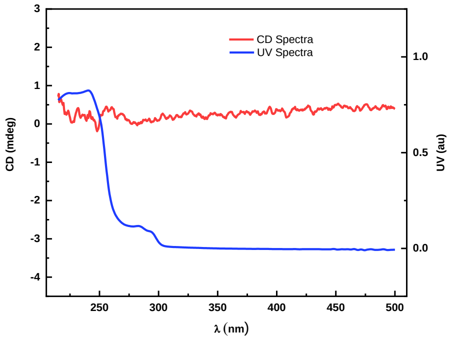
P11 (7.5 mM solution in THF)



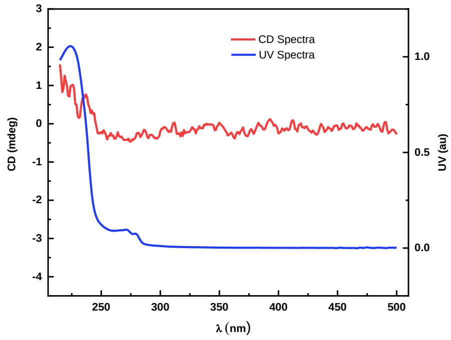
P12 (5.0 mM solution in THF)



P13 (5.0 mM solution in THF)



S1 (2.5 mM solution in THF)



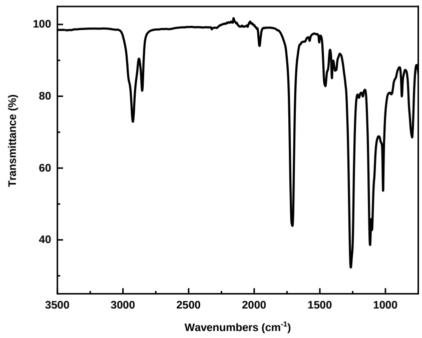
12) Optical rotation values

Product	*T*/°C	[α]$_D$/°	Product	*T*/°C	[α]$_D$/°
S1	28	+47.8	**P7**	30	+84.5
P1	30	+51.2	**P8**	29	+29.1
P2	29	+45.6	**P9**	30	+47.2
P3	28	+52.2	**P10**	29	+30.0
P4	31	+53.5	**P11**	30	+32.8
P5	28	+85.0	**P12**	19	+60.7
P6	30	+32.7	**P13**	20	+50.9

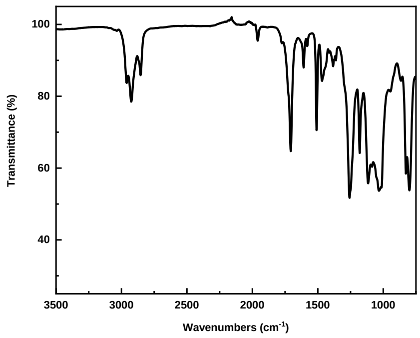
The samples were detected in 10 mg mL^{-1} $CHCl_3$ solution with a 10.0 cm sample tube.

13) IR spectrum of the polymers

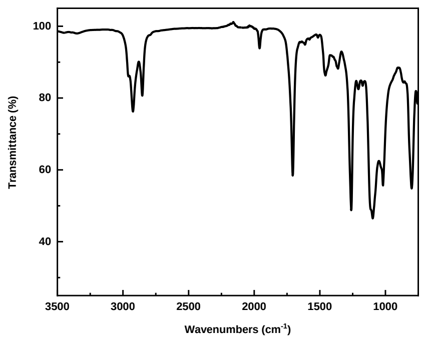
P1



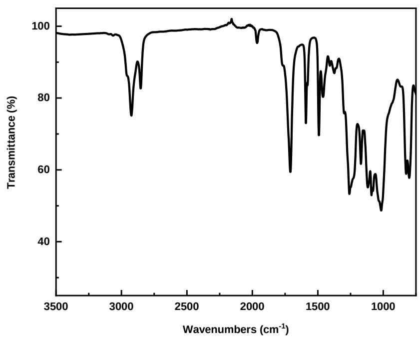
P2



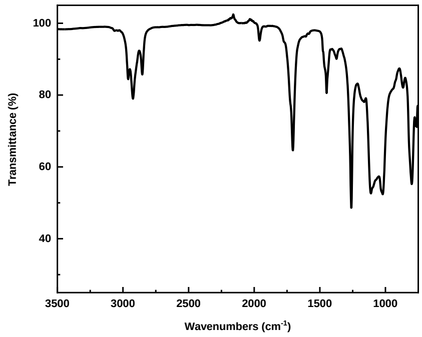
P3



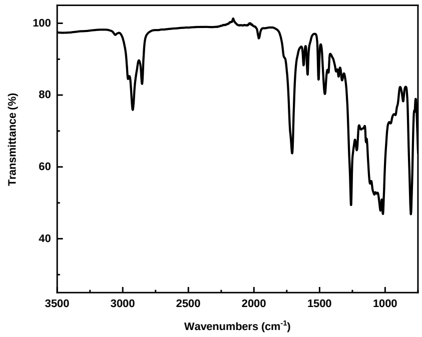
P4



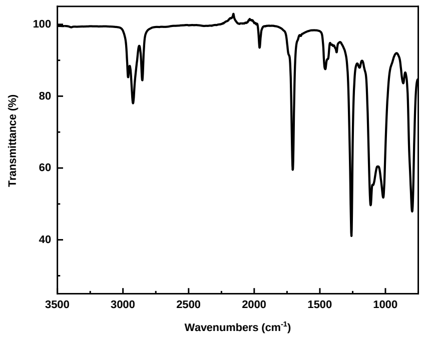
P5



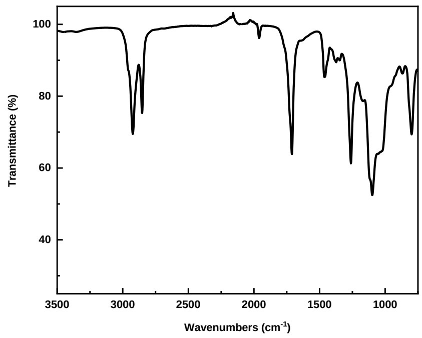
P6



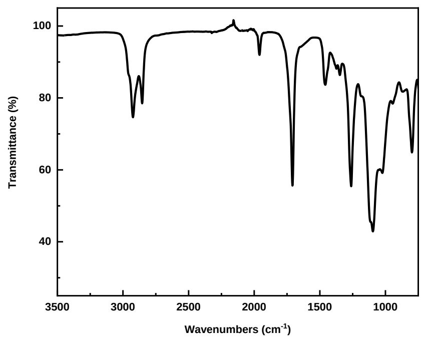
P7



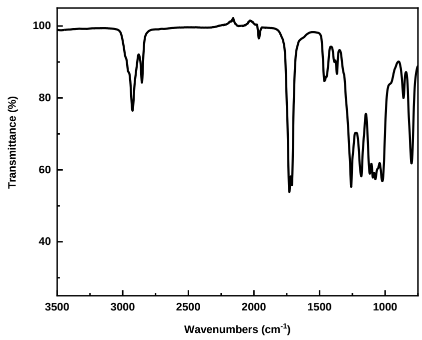
P8



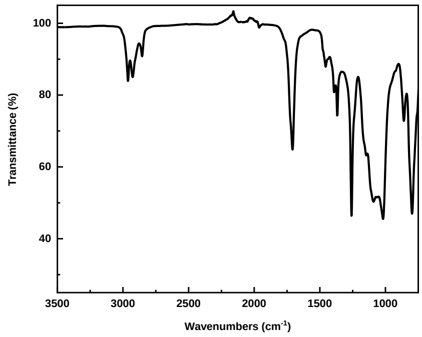
P9



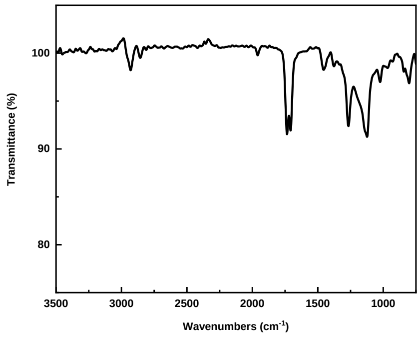
P10



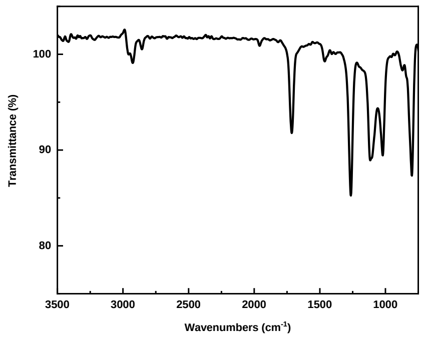
P11



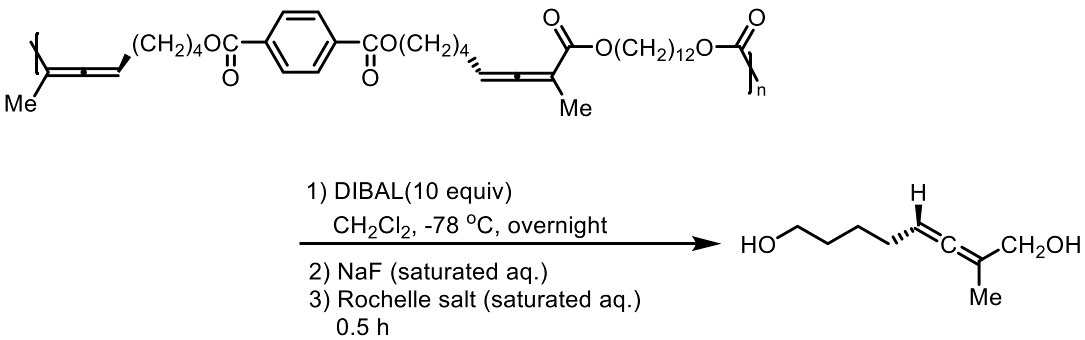
P12



P13



14) Degradation experiments for polymers P1, P12 and P13

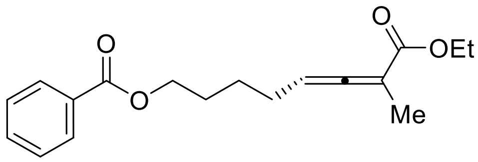


DIBAL-H (1 M in hexanes, 1.0 mL, 1.0 mmol) was added dropwise to an anhydrous solution of allene polymers in dichloromethane (2.5 mL) at -78ºC under nitrogen. The slightly yellow homogenous solution was stirred at -78ºC overnight. The reaction was quenched by the slow addition of saturated aqueous NaF (1.25 mL). The mixture was vigorously stirred while allowing it to warm to room temperature. The mixture was diluted with dichloromethane (1.25 mL) and saturated aqueous Rochelle salt (3.2 mL). The mixture was vigorously stirred for 0.5 hour at room temperature. The layers were separated, and the aqueous portion was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with Na_2SO_4, filtered and concentrated *in vacuo*. The resulting residue was purified by SiO_2 preparative TLC (1:1 EtOAc/petroleum ether) which afforded (*S*)-2-methylocta-2,3-diene-1,8-diol as a colorless liquid.

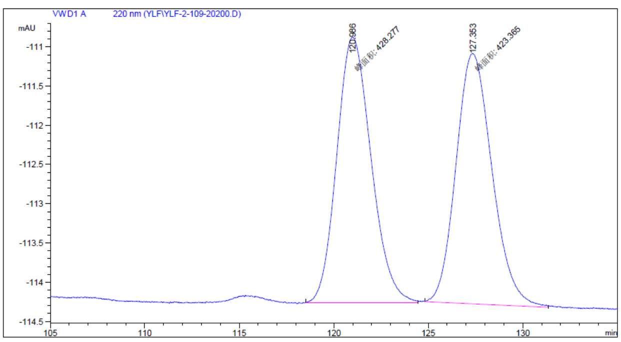
^{1}H NMR (400 MHz, CD_3OD) δ 5.16 – 5.08 (m, 1H), 3.96 (d, *J* = 2.2 Hz, 2H), 3.54 (t, *J* = 6.5 Hz, 2H), 2.01 (q, *J* = 7.0 Hz, 2H), 1.69 (d, *J* = 2.9 Hz, 3H), 1.61 – 1.52 (m, 2H), 1.51 – 1.42 (m, 2H); **^{13}C NMR** (101 MHz, CD_3OD) δ 203.38, 101.57, 92.75, 66.21, 63.66, 33.87, 30.66, 27.34, 16.91. HRMS (ESI) calcd for $C_9H_{16}O_2Na$ $[M+Na]^+$ 179.1048, found 179.1041.

Reagents:

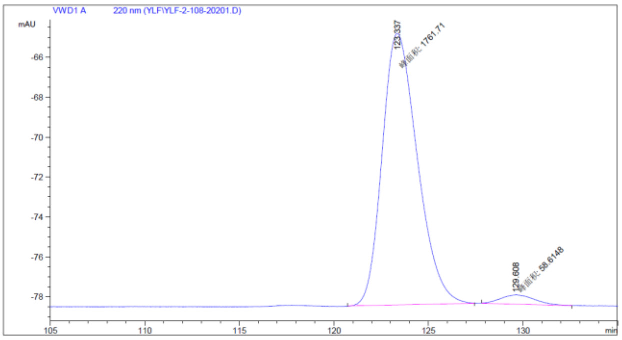
S1



52.3 mg (0.173 mmol) of **S1** and 0.86 mL of DIBAL-H solution was added. The reaction mixture was stirred for 4 hours. 20.2 mg of the diol product was afforded, yield = 75%, 94% *ee*. HPLC (Chiralcel OD-3, hexane/*i*PrOH, 98:2, 0.3 mL min^{-1}, 220 nm): t_R (*S*) = 123.3 min, t_R (*R*) = 129.6 min. $[\alpha]_D^{25}$ = +10.0 (*c* = 0.3 in CH_2Cl_2).

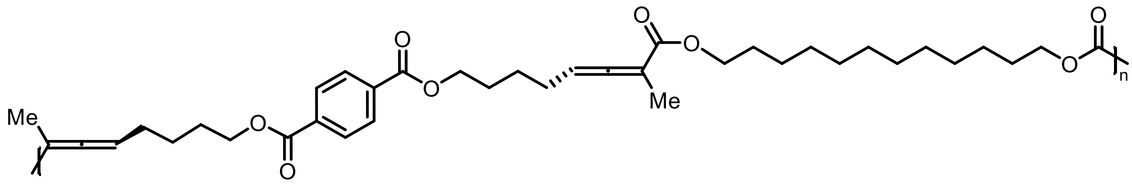


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	120.986	MM	2.1058	428.27731	3.38969	50.2884
2	127.353	MM	2.2103	423.36475	3.19234	49.7116

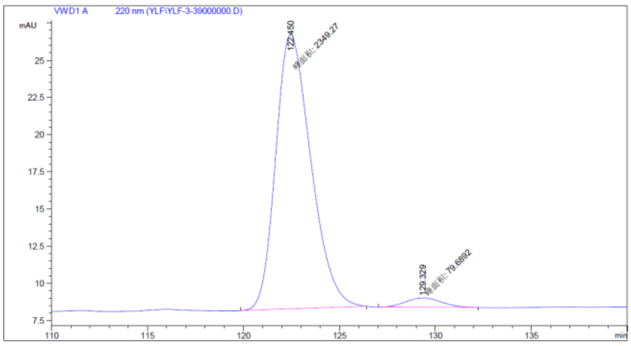


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	123.337	MM	2.1599	1761.70862	13.59396	96.7800
2	129.608	MM	2.0860	58.61483	4.68308e-1	3.2200

P1

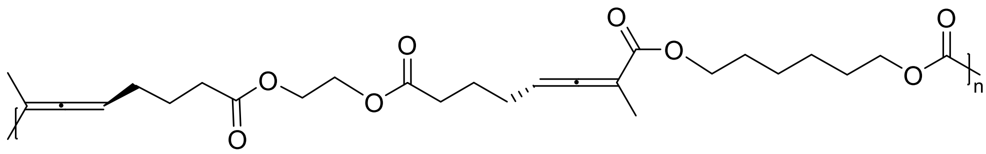


M_n = 14.2 kDa, M_w/M_n = 1.92. 59.5 mg (0.093 mmol) of **P1** was added. The reaction mixture was stirred overnight. 10.5 mg of the diol product was afforded, yield = 36%, 93% *ee*. HPLC (Chiralcel OD-3, hexane/*i*PrOH, 98:2, 0.3 mL min^{-1}, 220 nm): t_R (*S*) = 122.4 min, t_R (*R*) = 129.3 min. $[\alpha]_D^{26}$ = +10.3 (*c* = 0.3 in CH_2Cl_2).

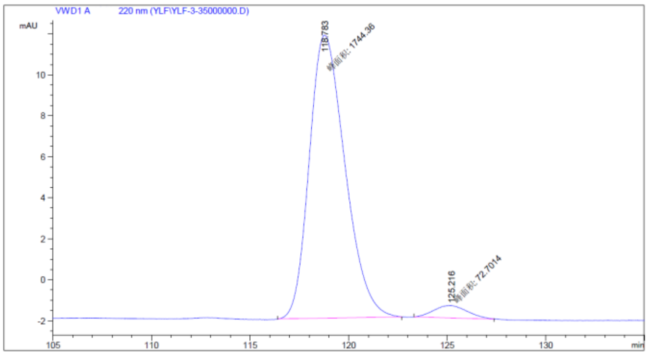


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	122.450	MM	2.1239	2349.27368	18.43504	96.7192
2	129.329	MM	2.1288	79.68921	6.23897e-1	3.2808

P12

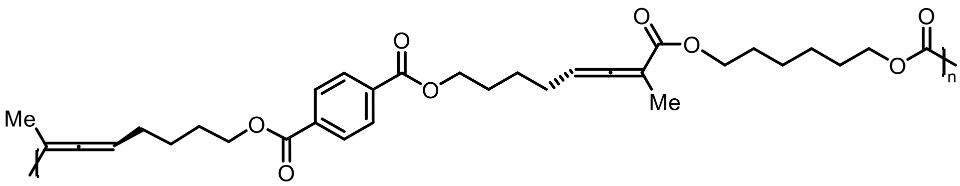


M_n = 7.6 kDa, M_w/M_n = 1.83. 38.6 mg (0.081 mmol) of **P12** was added. The reaction mixture was stirred overnight. 7.1 mg of the diol product was afforded, yield = 28%, 92% *ee*. HPLC (Chiralcel OD-3, hexane/*i*PrOH, 98:2, 0.3 mL min^{-1}, 220 nm): t_R (*S*) = 118.8 min, t_R (*R*) = 125.2 min. $[\alpha]_D^{26}$ = +10.3 (*c* = 0.3 in CH_2Cl_2).

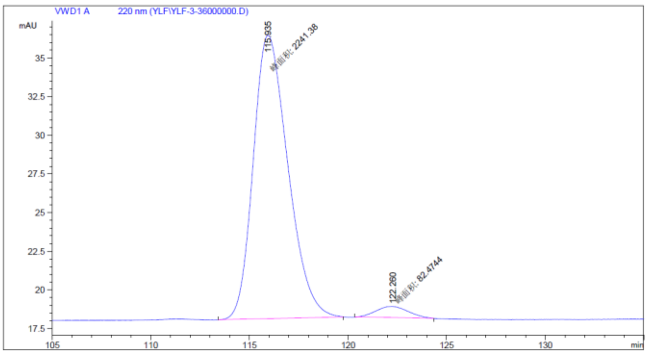


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	118.783	MM	2.1039	1744.36499	13.81883	95.9990
2	125.216	MM	1.9577	72.70139	6.18948e-1	4.0010

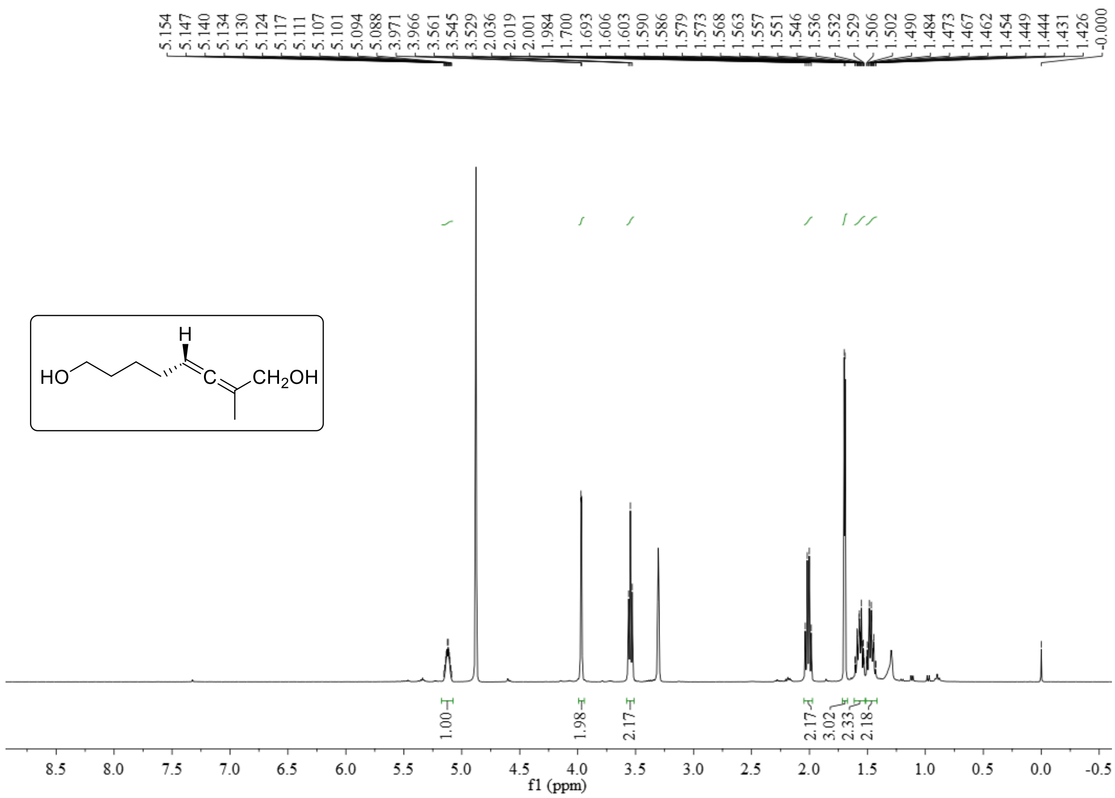
P13

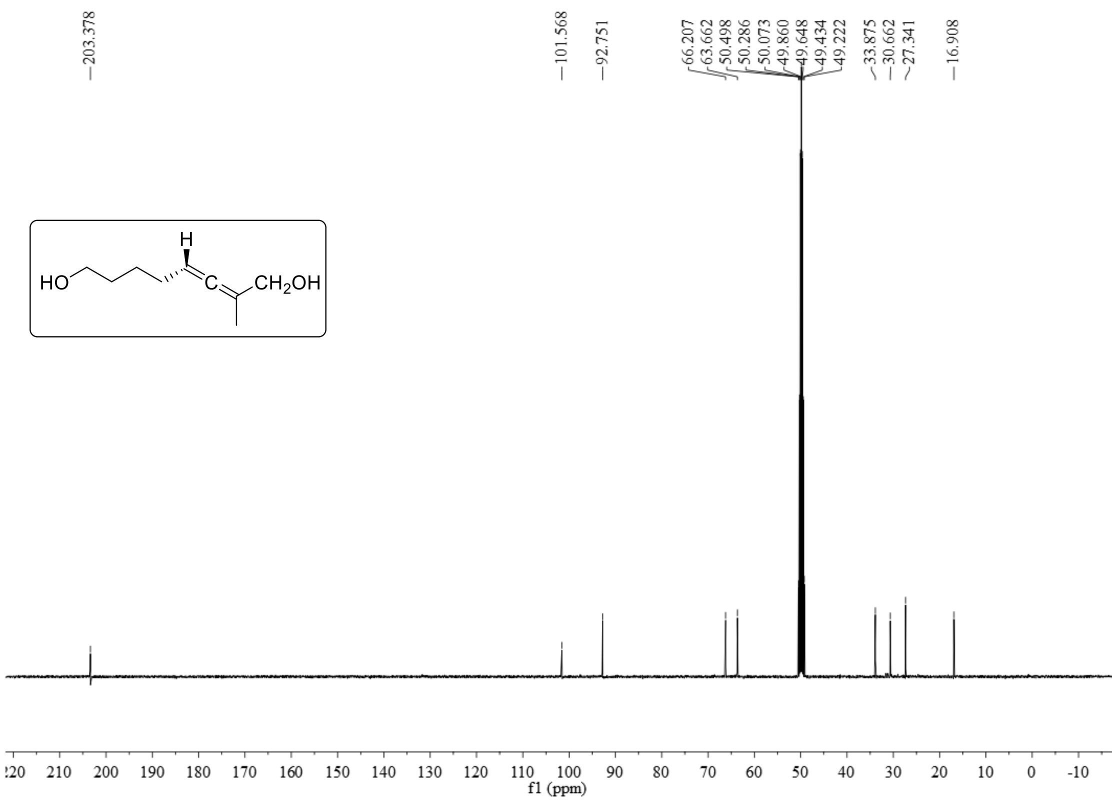


M_n = 6.7 kDa, M_w/M_n = 1.71. 50.3 mg (0.091 mmol) of **P13** was added. The reaction mixture was stirred overnight. 8.4 mg of the diol product was afforded, yield = 30%, 93% *ee*. HPLC (Chiralcel OD-3, hexane/*i*PrOH, 98:2, 0.3 mL min^{-1}, 220 nm): t_R (*S*) = 115.9 min, t_R (*R*) = 122.3 min. $[\alpha]_D^{26}$ = +10.3 (*c* = 0.3 in CH_2Cl_2).



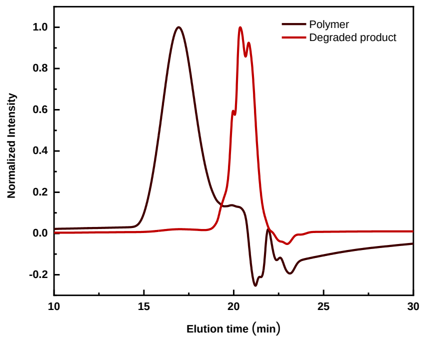
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	115.935	MM	2.0382	2241.38232	18.32791	96.4510
2	122.260	MM	1.8798	82.47440	7.31237e-1	3.5490


5.154
5.147
5.140
5.134
5.130
5.124
5.117
5.111
5.107
5.101
5.094
5.088
3.971
3.966
3.561
3.545
3.529
2.036
2.019
2.001
1.984
1.700
1.693
1.606
1.603
1.590
1.586
1.579
1.573
1.568
1.563
1.557
1.551
1.546
1.536
1.532
1.529
1.506
1.502
1.490
1.484
1.473
1.467
1.462
1.454
1.449
1.444
1.431
1.426
-0.000
H
HO
C
CH2OH
1.00
1.98
2.17
2.17
3.02
2.33
2.18
f1 (ppm)


203.378
101.568
92.751
66.207
63.662
50.498
50.286
50.073
49.860
49.648
49.434
49.222
33.875
30.662
27.341
16.908
H
HO
C
CH2OH
f1 (ppm)

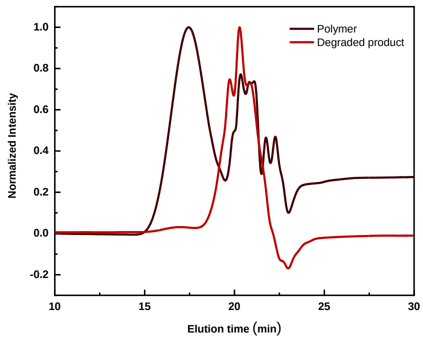
SEC traces of degradation products:

P1



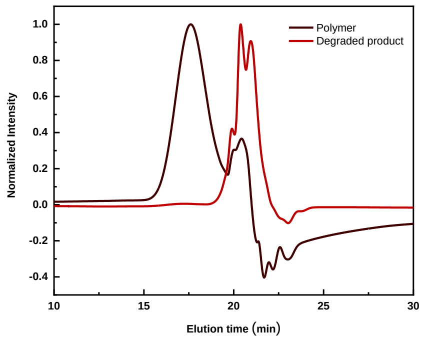
Polymer: M_n = 13.4 kDa, M_w/M_n = 1.88
Product: M_n = 1.5 kDa, M_w/M_n = 1.01

P12



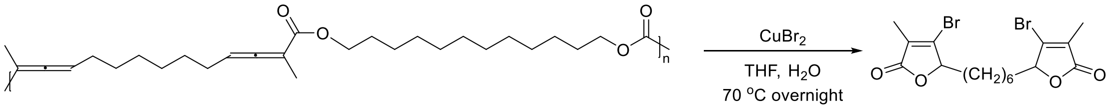
Polymer: M_n = 8.8 kDa, M_w/M_n = 1.81
Product: M_n = 1.8 kDa, M_w/M_n = 1.09

P13



Polymer: M_n = 7.4 kDa, M_w/M_n = 1.69
Product: M_n = 1.5 kDa, M_w/M_n = 1.01

15) Degradation experiments by $CuBr_2$ of polymer P7

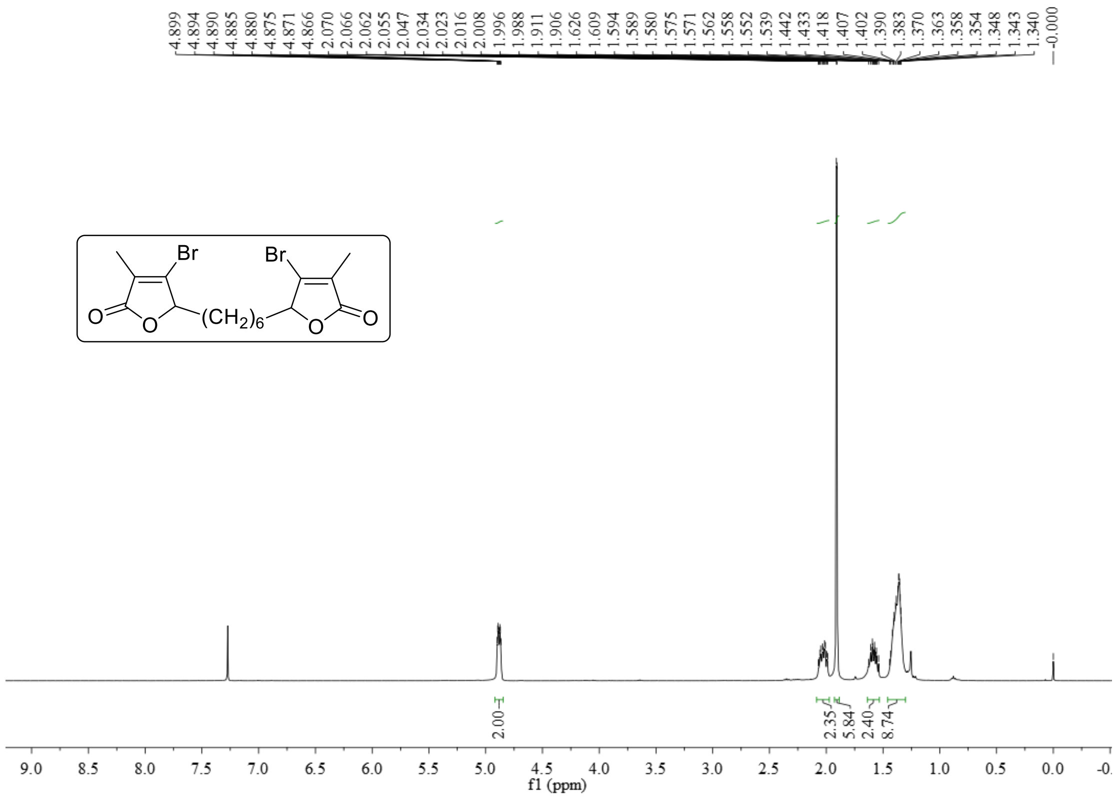


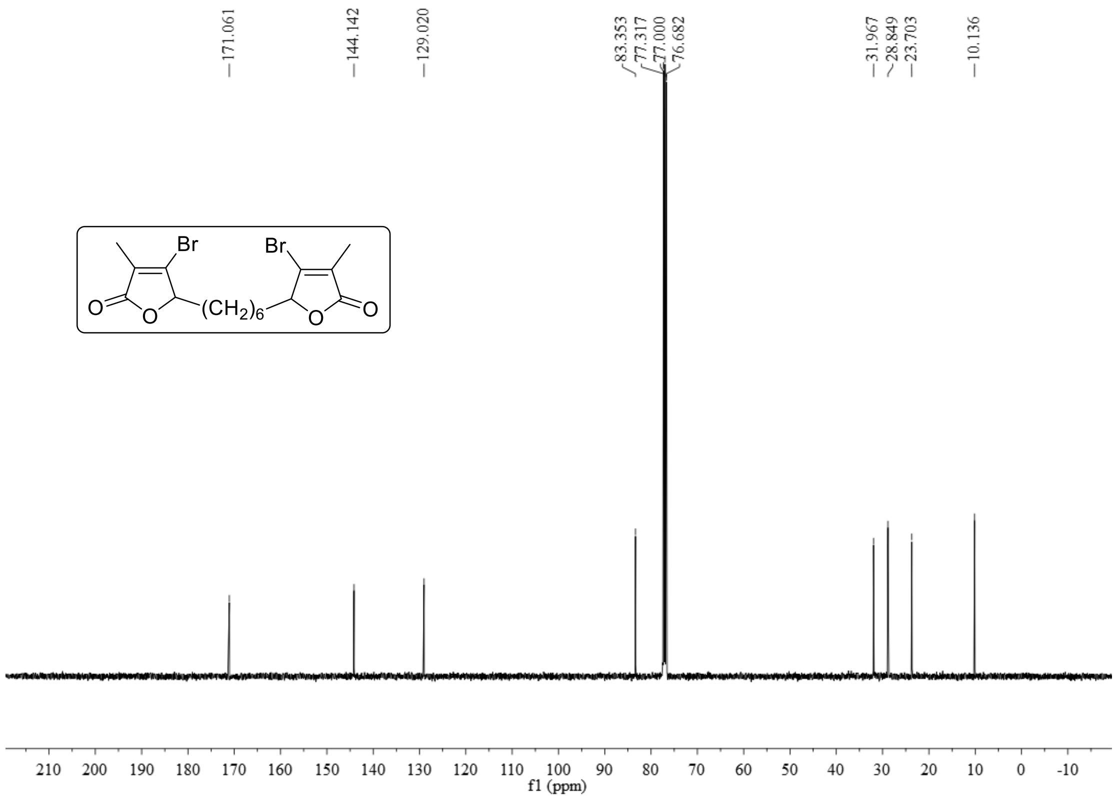
THF (1.0 mL) and water (0.65 mL) was added into the mixture of **P7** (M_n = 7.8 kDa, M_w/M_n = 1.75, 35.0 mg, 0.079 mmol, 1 eq) and $CuBr_2$ (141 mg, 0.630 mmol, 8 equiv). The reaction mixture was stirred for 11 h at 70 °C. After the reaction mixture was cooled to rt, 5 mL of water was added to quench the reaction. The crude reaction mixture was extracted with dichloromethane (3×10 mL). The combined organic phases were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The crude product was purified by SiO_2 flash column chromatography to get rid of insoluble salts. The filtrate was concentrated in *vacuo*. The resulting residue was purified by SiO_2 preparative TLC (1:2 EtOAc/petroleum ether) which afforded 5,5'-(hexane-1,6-diyl)-bis(4-bromo-3- methylfuran-2(5*H*)-one) as a white solid (19.9 mg, 58% yield), mp = 97-100 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.90 – 4.87 (m, 2H), 2.07 – 1.99 (m, 2H), 1.91 (d, *J* = 1.9 Hz, 6H), 1.63 – 1.54 (m, 2H), 1.46 – 1.30 (m, 8H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.06, 144.14, 129.02, 83.35, 31.97, 28.85, 23.70, 10.14. HRMS (ESI) calcd for $C_{16}H_{20}Br_2O_4Na$ $[M+Na]^+$ 456.9626, found 456.9623. $[\alpha]_D^{24}$ = -21.6 (*c* = 0.7 in $CHCl_3$).

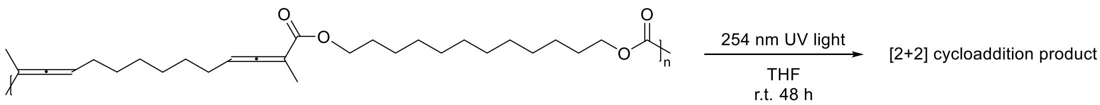
Optimization of the reaction conditions:

Entry	m_0/mg	n_0/mmol	M_n/kDa	PDI	time/h	*m*/mg	yield/%
1	40.7	0.092	9.0	2.21	8	18.5	46
2	35.0	0.079	7.8	1.75	11	19.9	58
3	37.6	0.085	9.9	1.78	15	20.9	57
4	42.0	0.094	10.4	1.89	18	22.8	56
5	36.2	0.081	7.5	1.65	21	16.5	46





16) Photochemical [2+2] crosslinking experiment of polymer P7



P7 (M_n = 11.2 kDa, M_w/M_n = 2.23, 20.7 mg, 0.047 mmol, 1 equiv) was dissolved in 1 mL of THF in a 10 mL quartz tube. The reaction mixture was irradiated under 254 nm UV light in room temperature under nitrogen atmosphere. After irradiation, insoluble gels could be observed, and then the solvent was evaporated under reduced pressure. The crosslinked product was dried *in vacuo* and tested to afford IR spectrum. NMR analysis of the crosslink product was precluded in view of its insolubility.

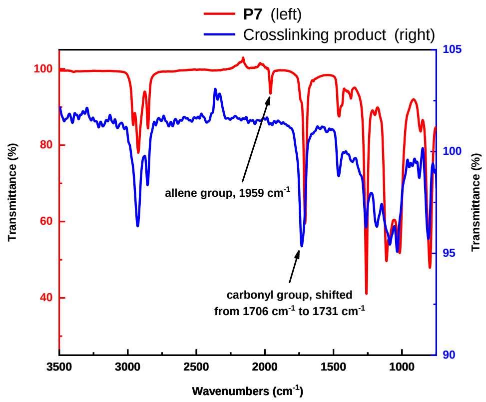


Figure S2. IR spectrum of crosslinked product from polymer **P7**

17) References

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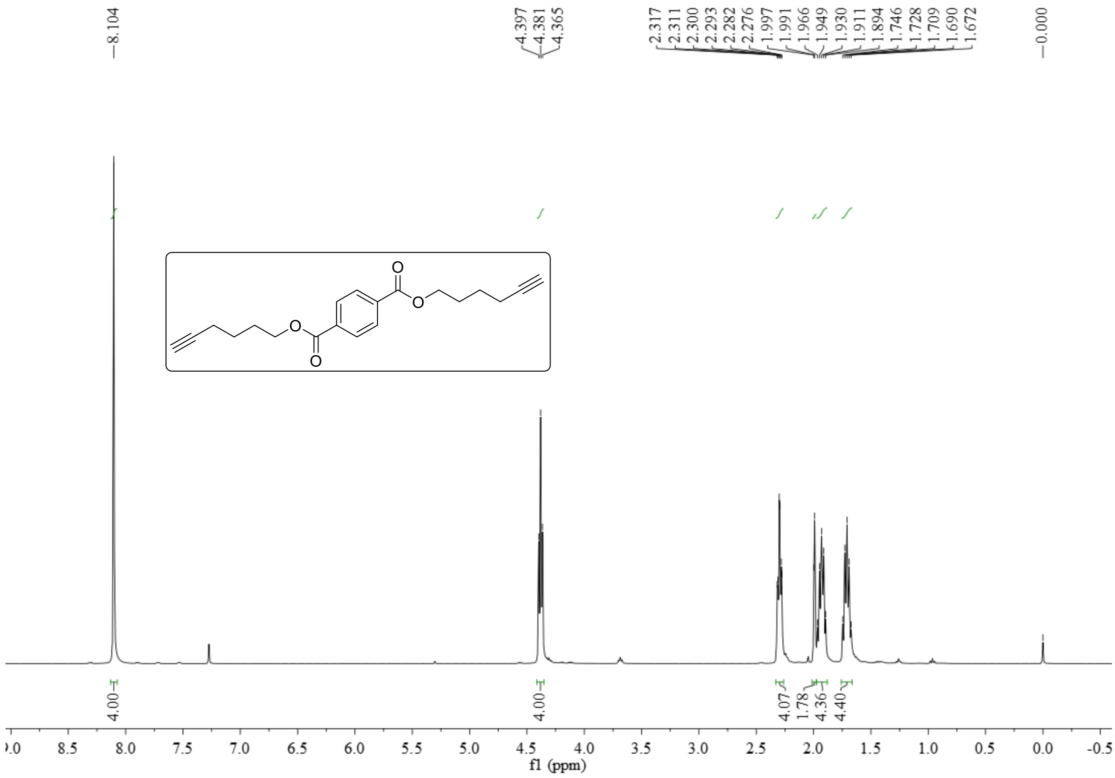
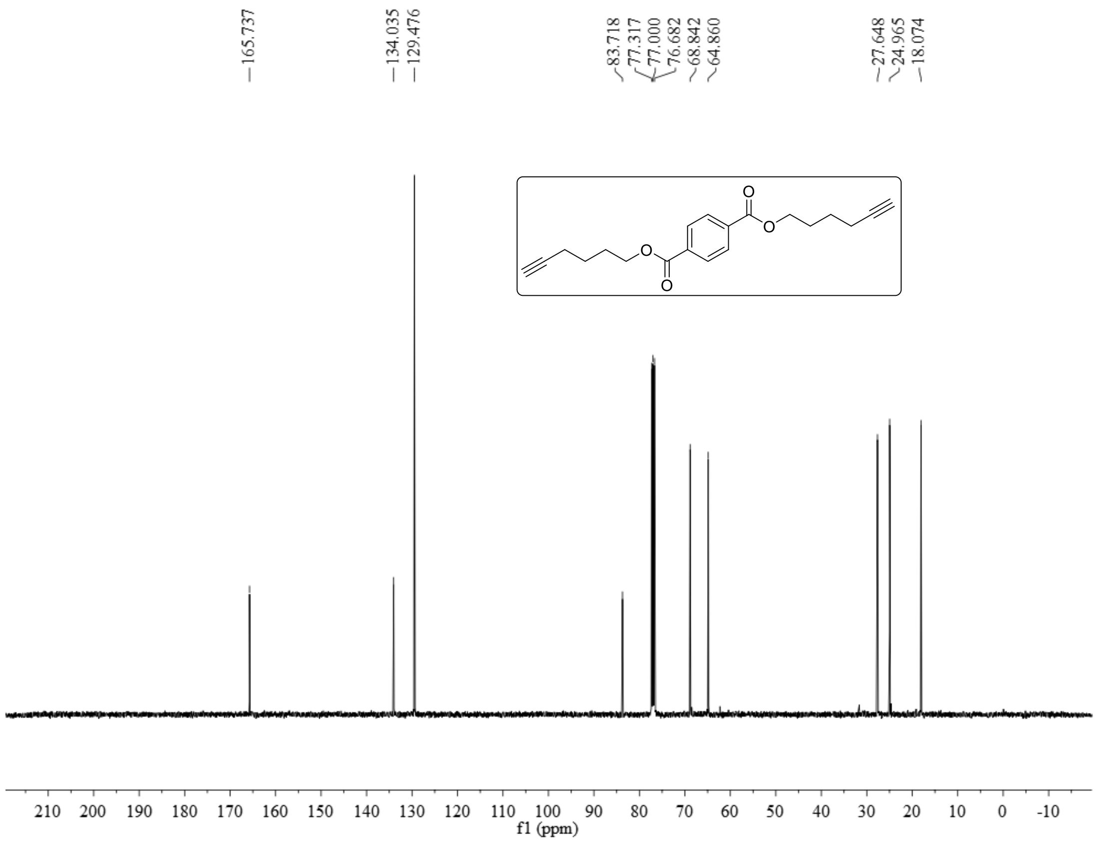
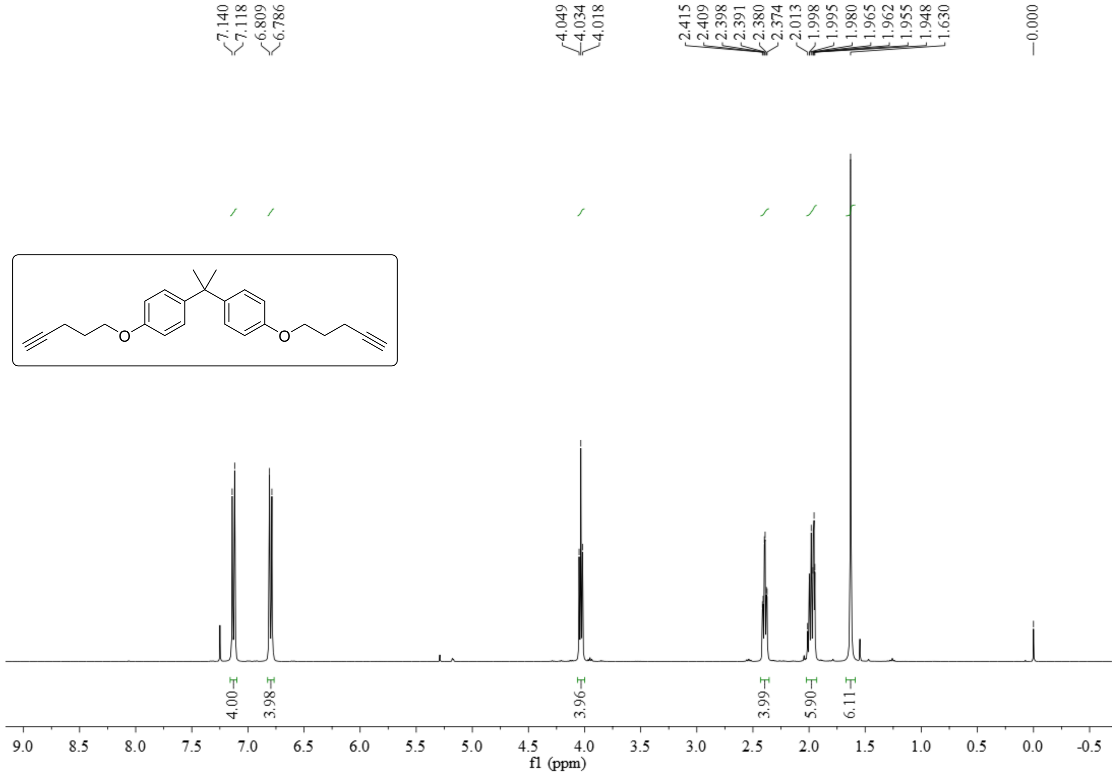
18) ^{1}H and ^{13}C NMR spectrum for the monomers and ligand

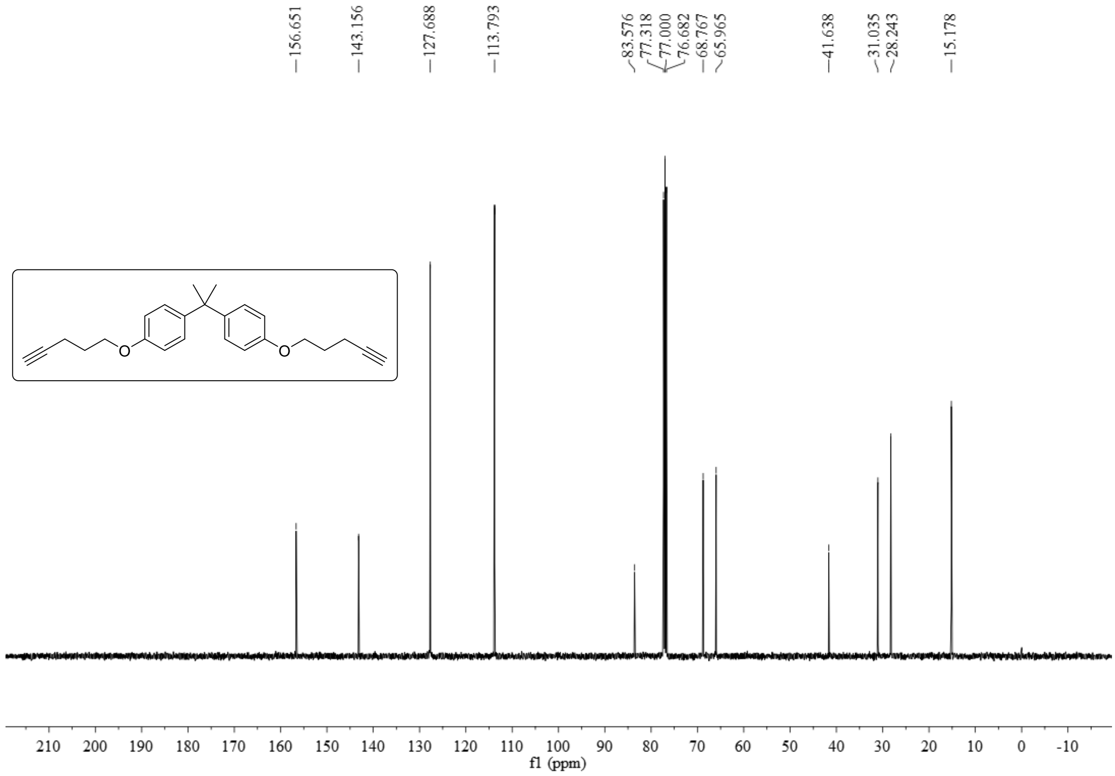
Di(hex-5-yn-1-yl)terephthalate (M1)



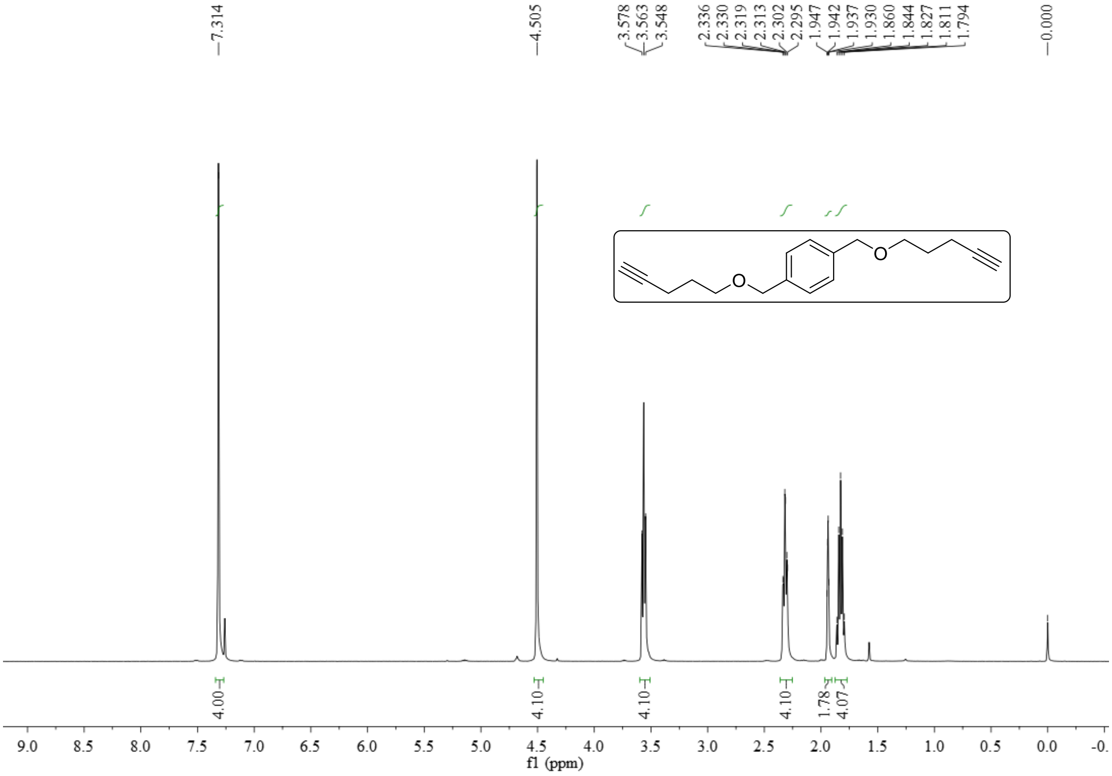


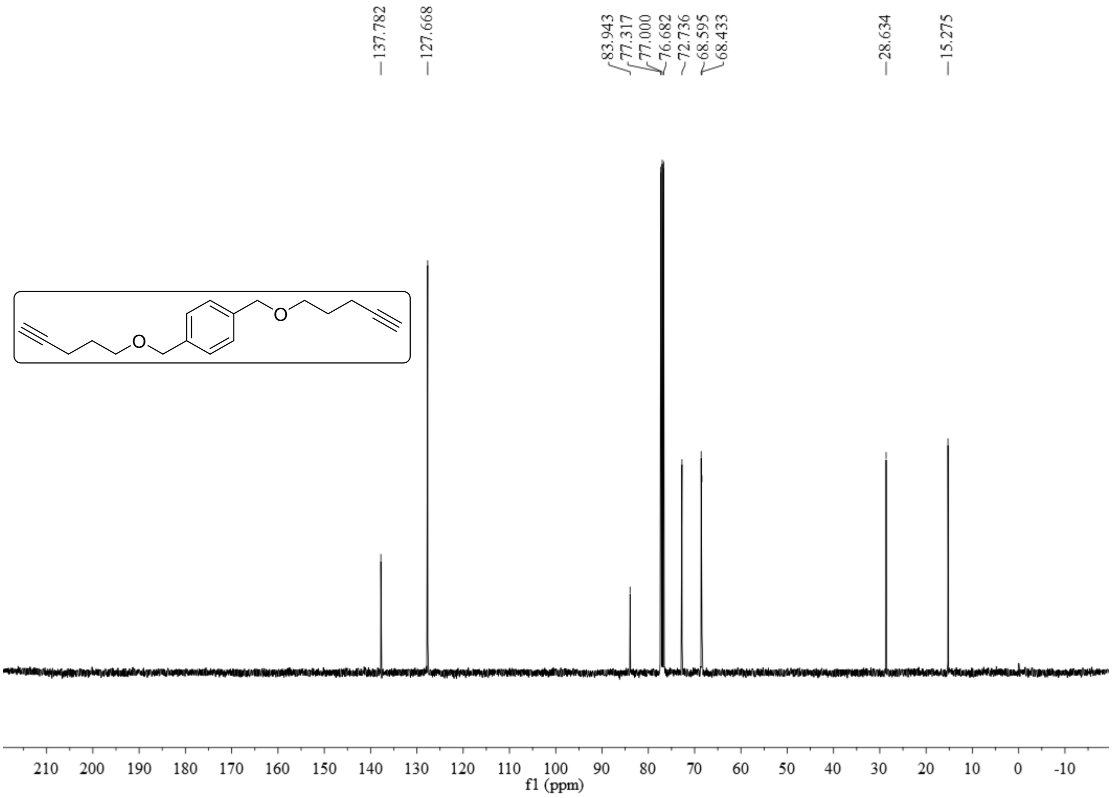
4,4'-(propane-2,2-diyl)bis((pent-4-yn-1-yloxy)benzene) (M2)



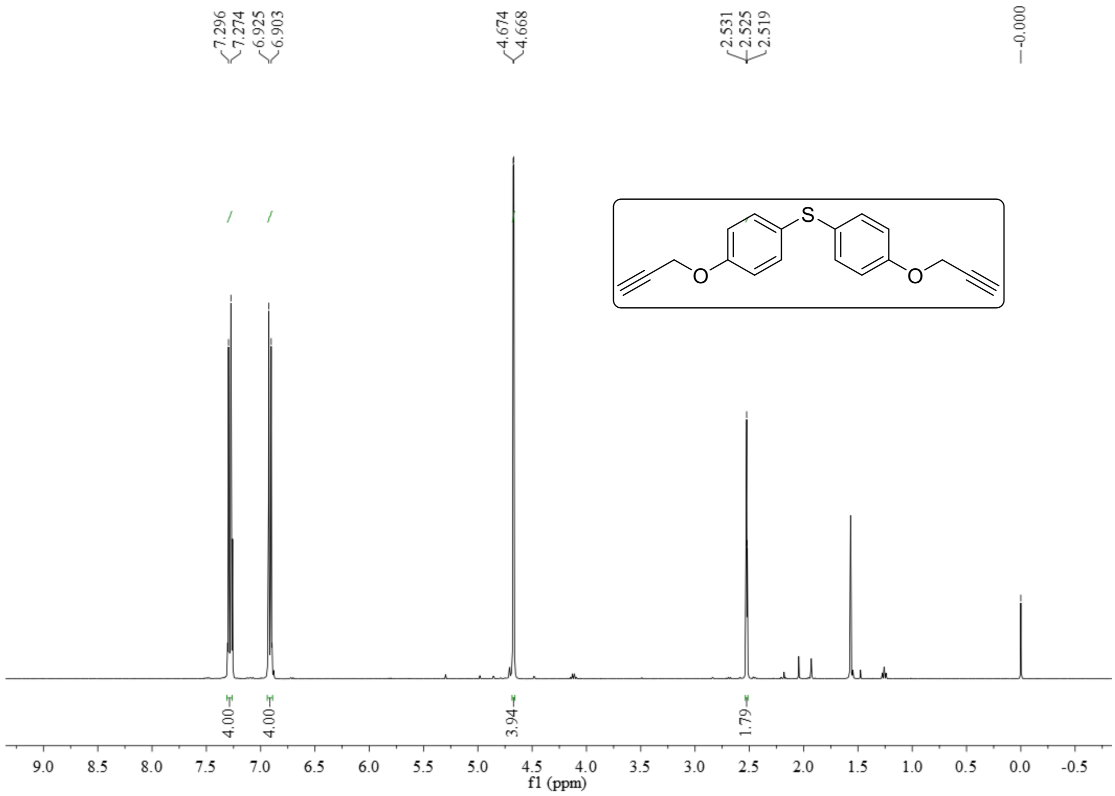


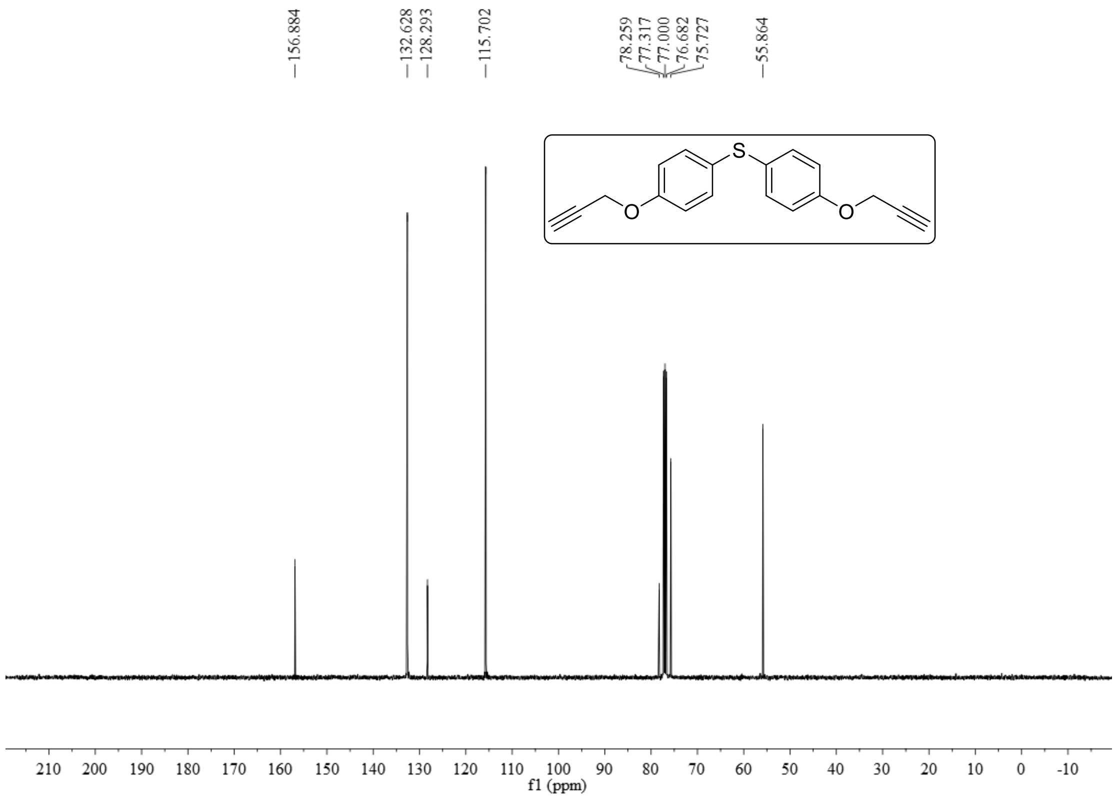
1,4-bis((pent-4-yn-1-yloxy)methyl)benzene (M3)



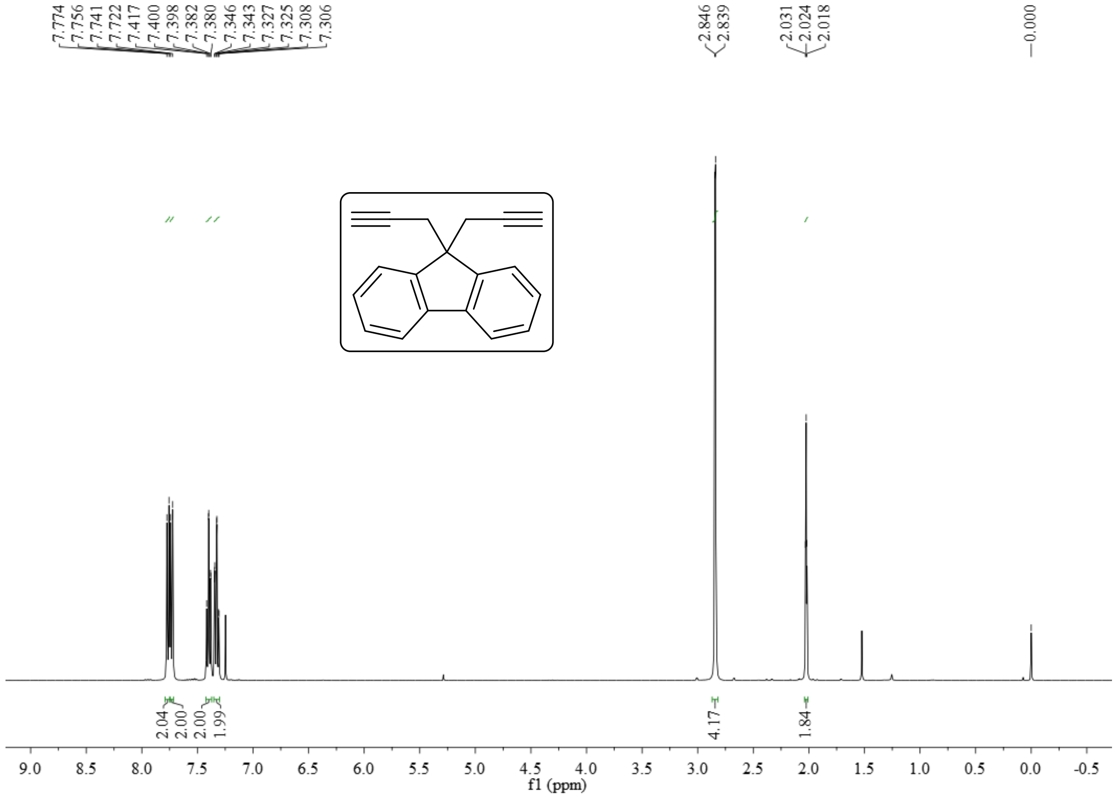


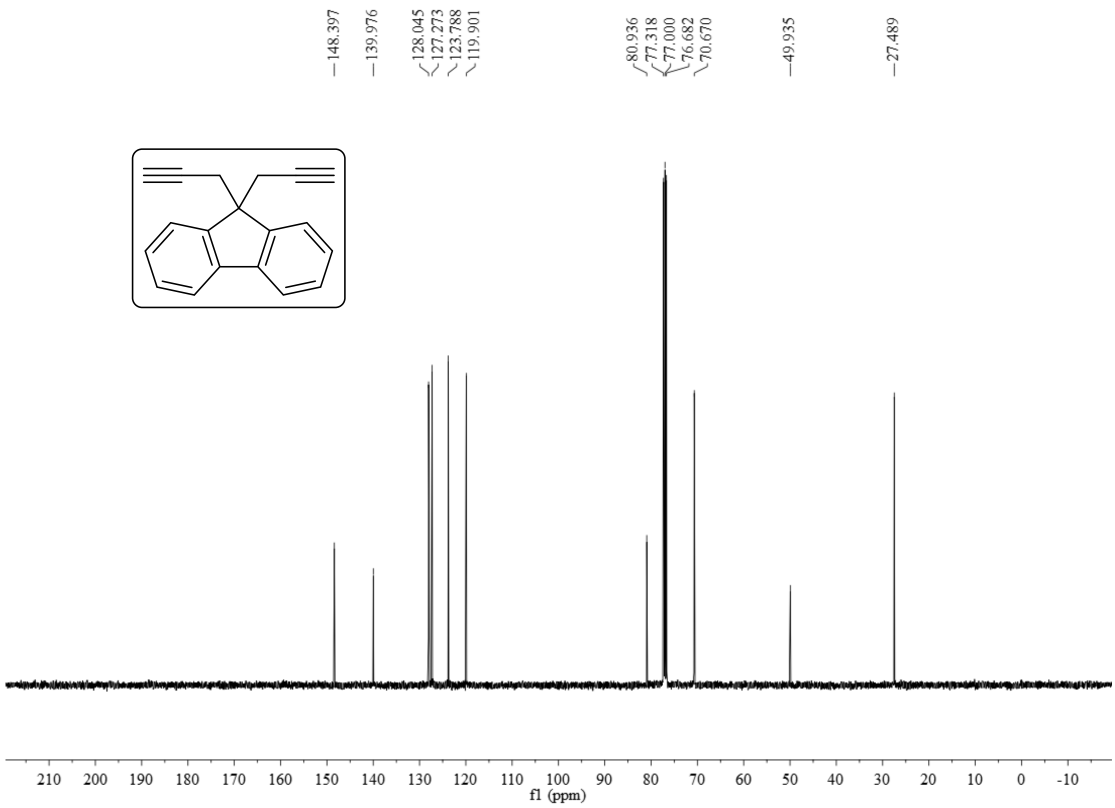
Bis(4-(prop-2-yn-1-yloxy)phenyl)sulfane (M4)



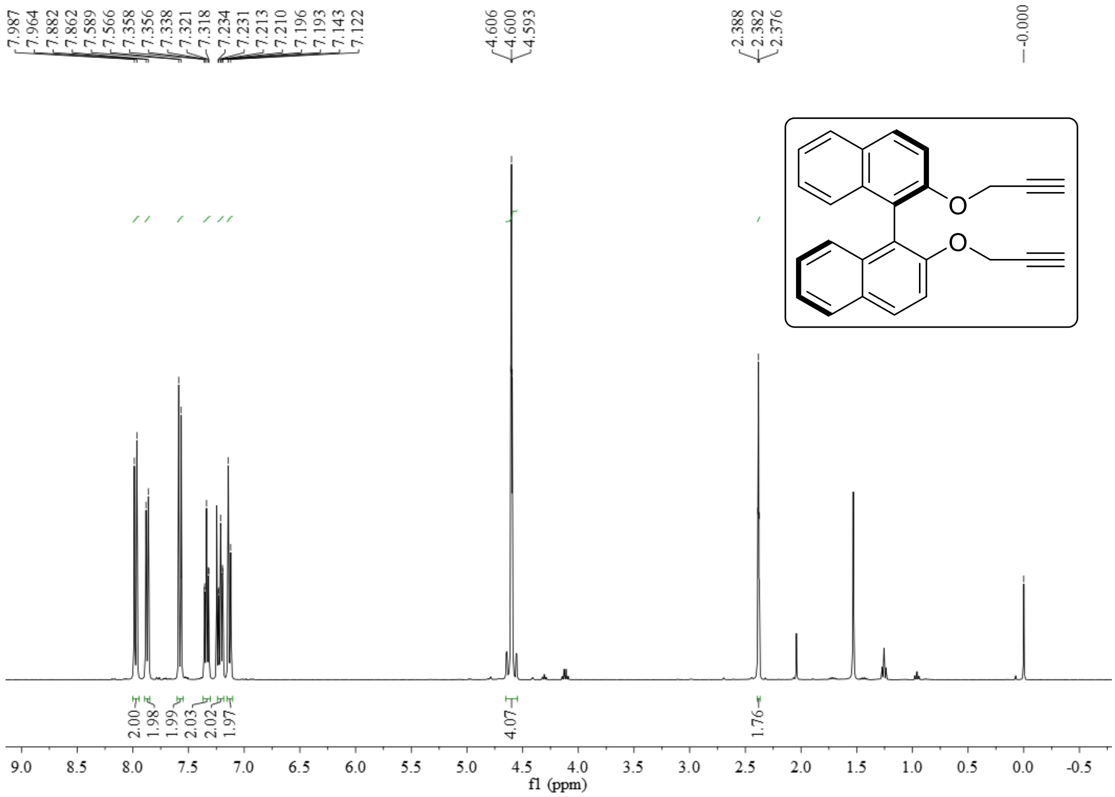


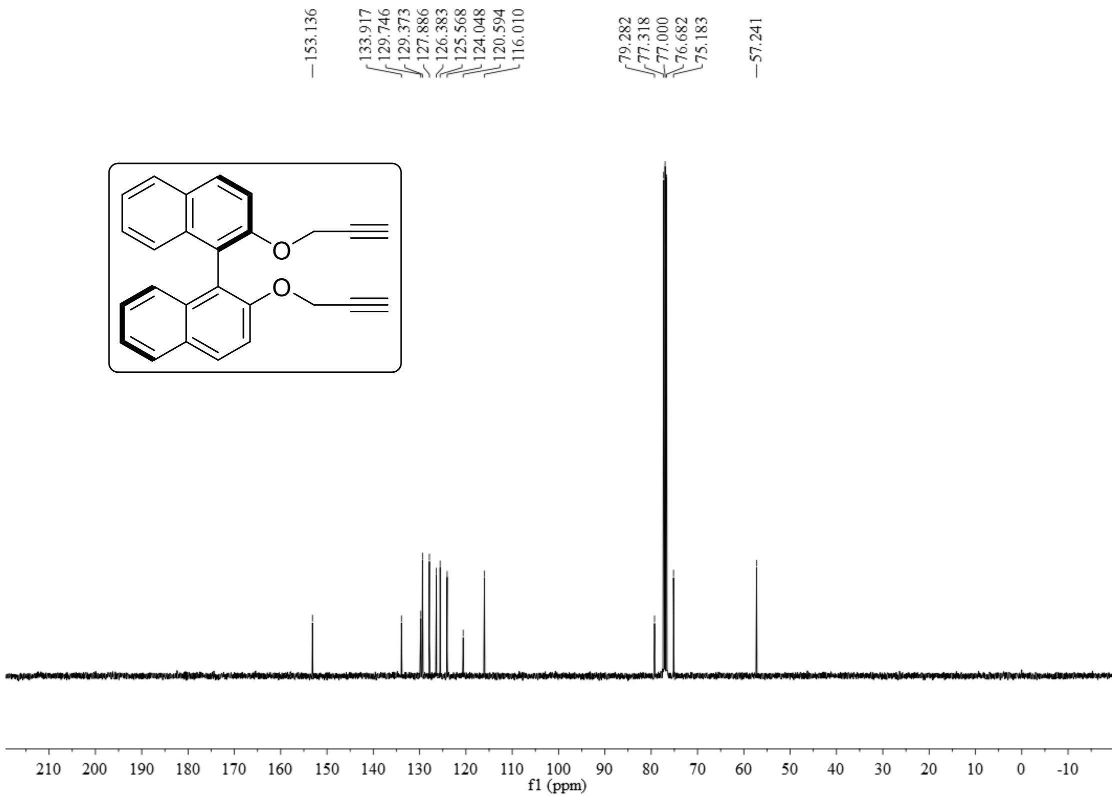
9,9-di(prop-2-yn-1-yl)-9*H*-fluorene (M5)



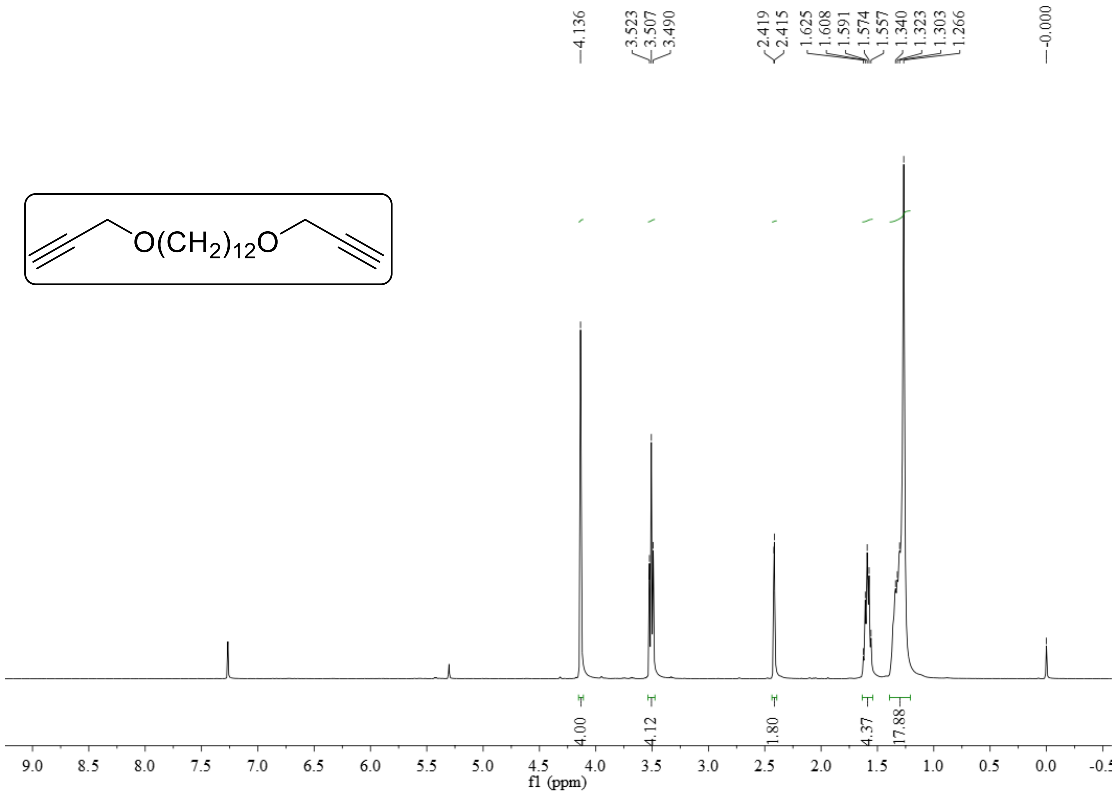


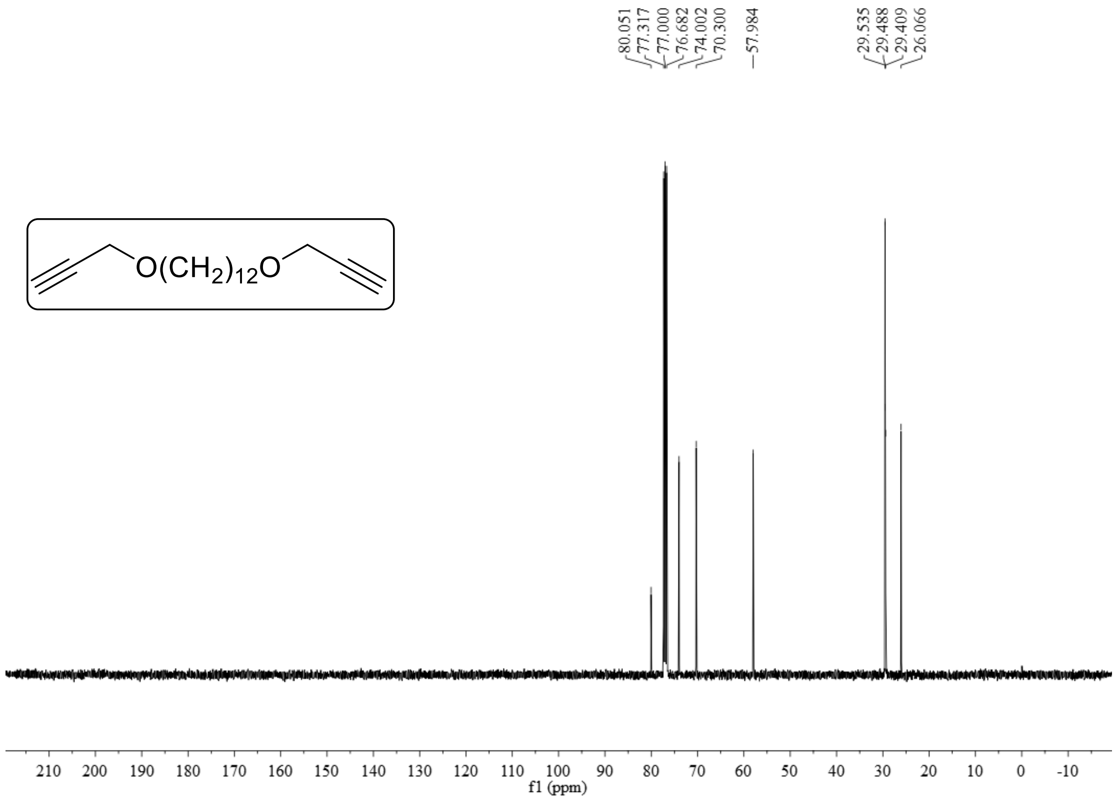
(*R*)-2,2'-bis(prop-2-yn-1-yloxy)-1,1'-binaphthalene (M6)



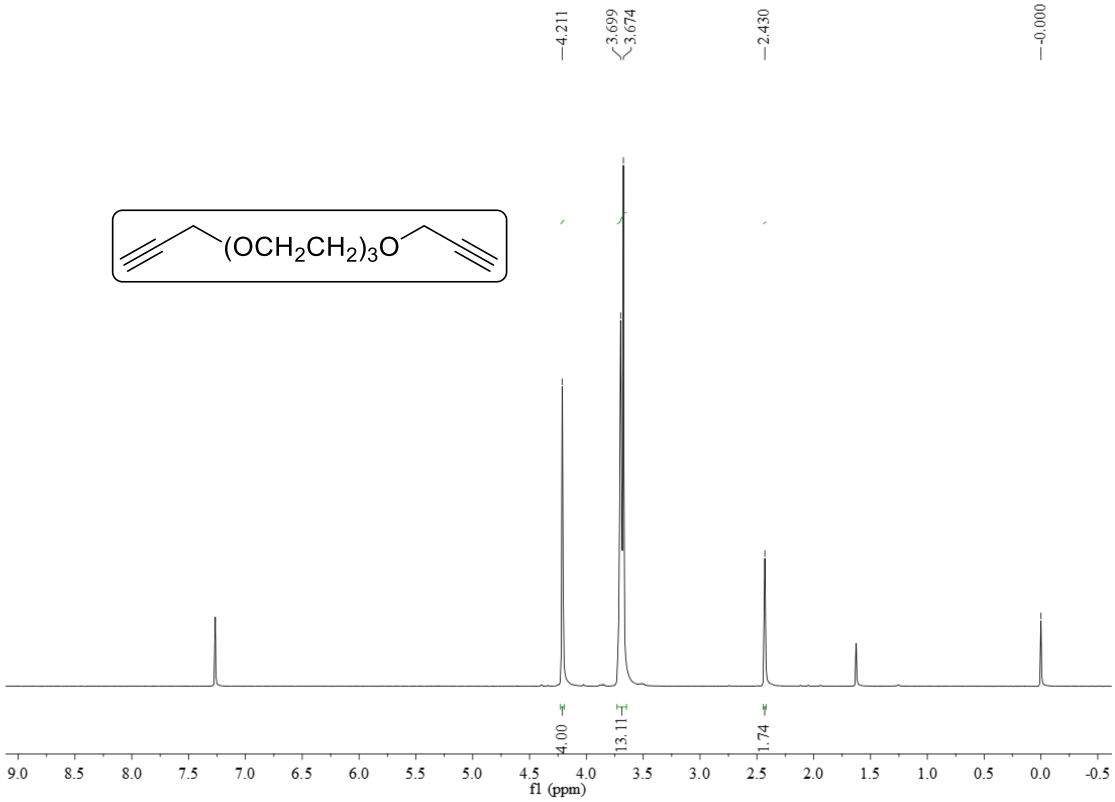


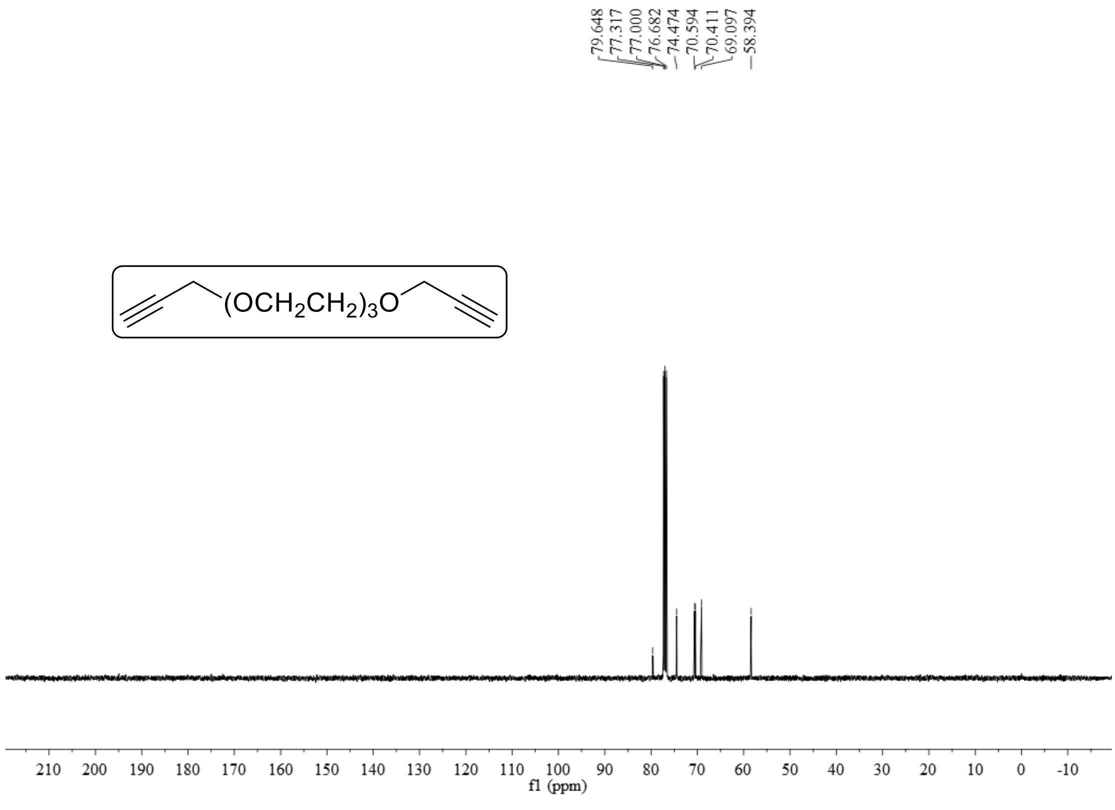
1,12-bis(prop-2-yn-1-yloxy)dodecane (M8)



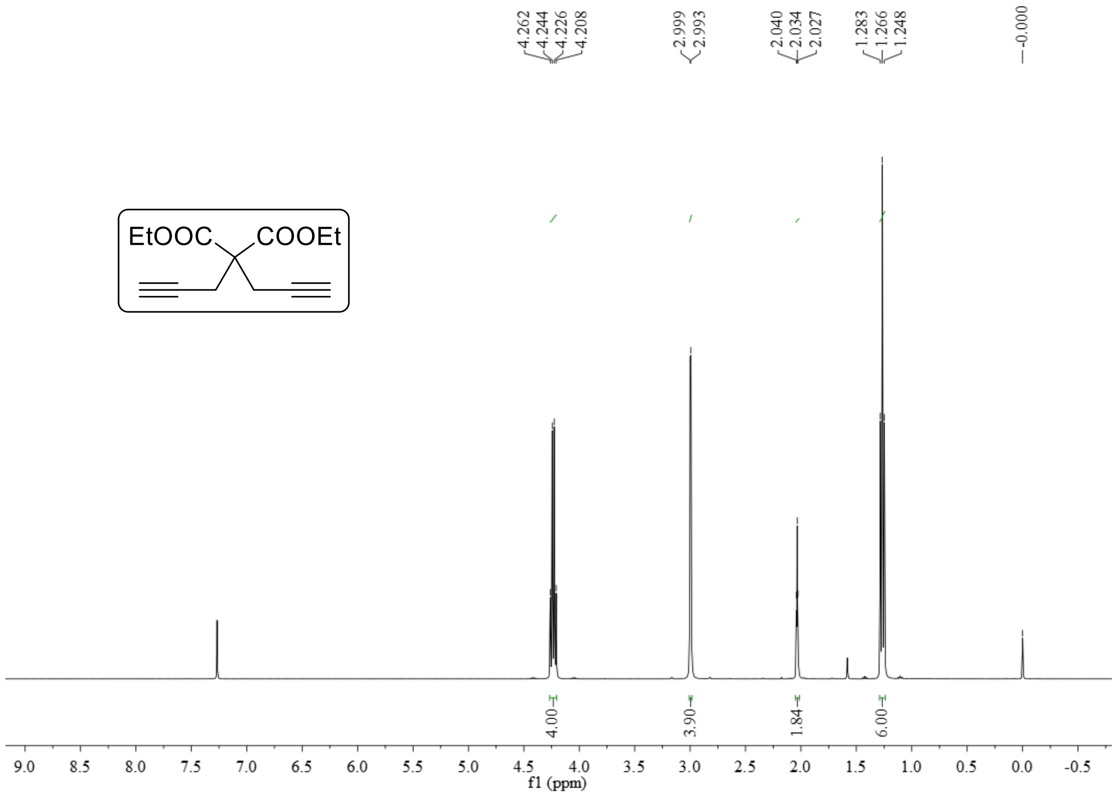


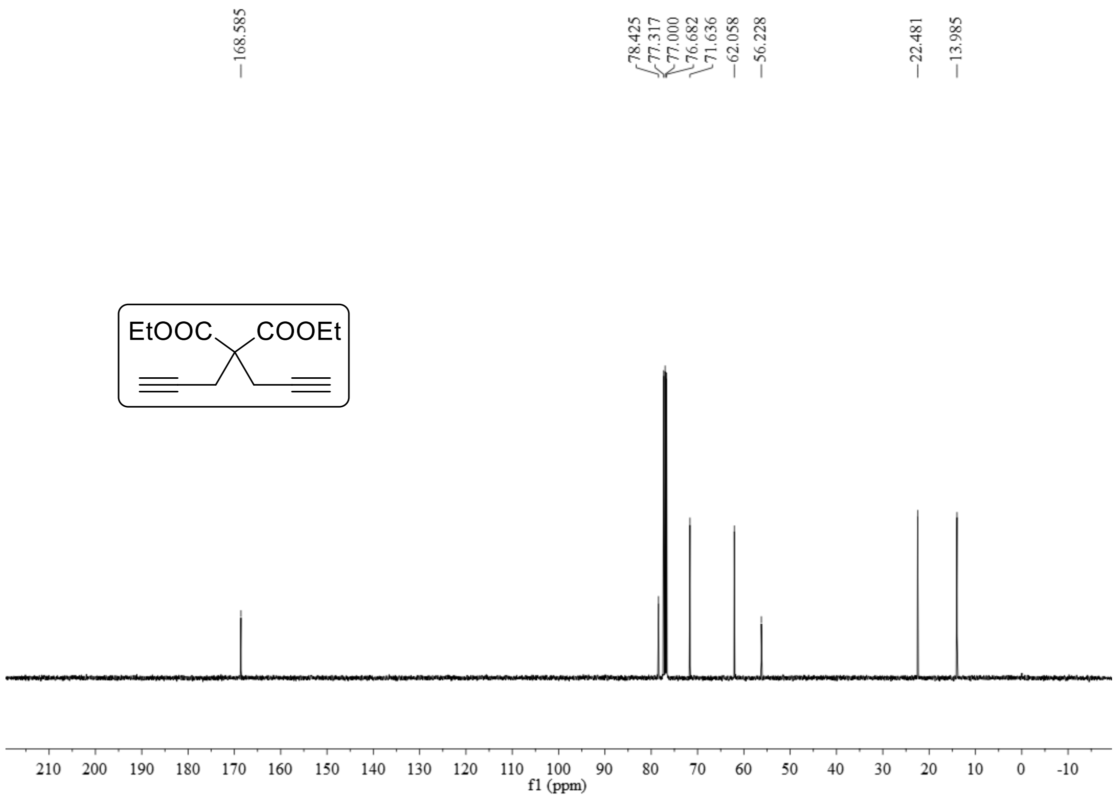
4,7,10,13-tetraoxahexadeca-1,15-diyne (M9)



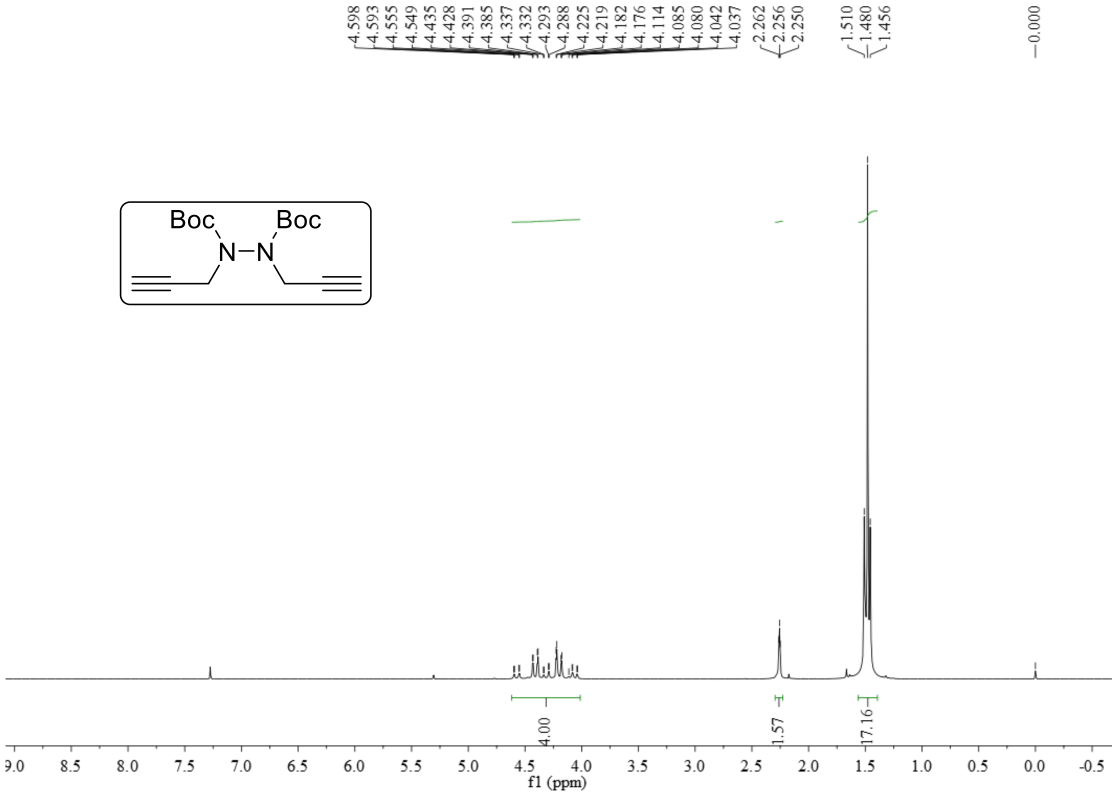


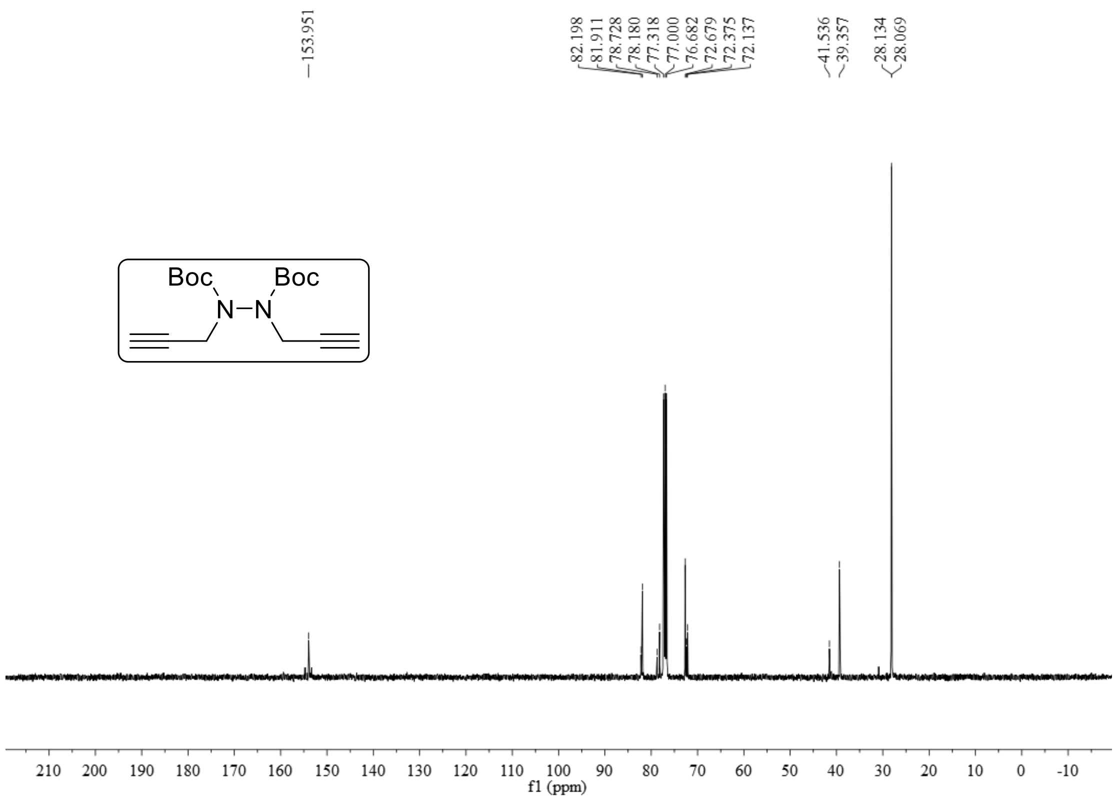
Diethyl 2,2-di(prop-2-yn-1-yl)malonate (M10)



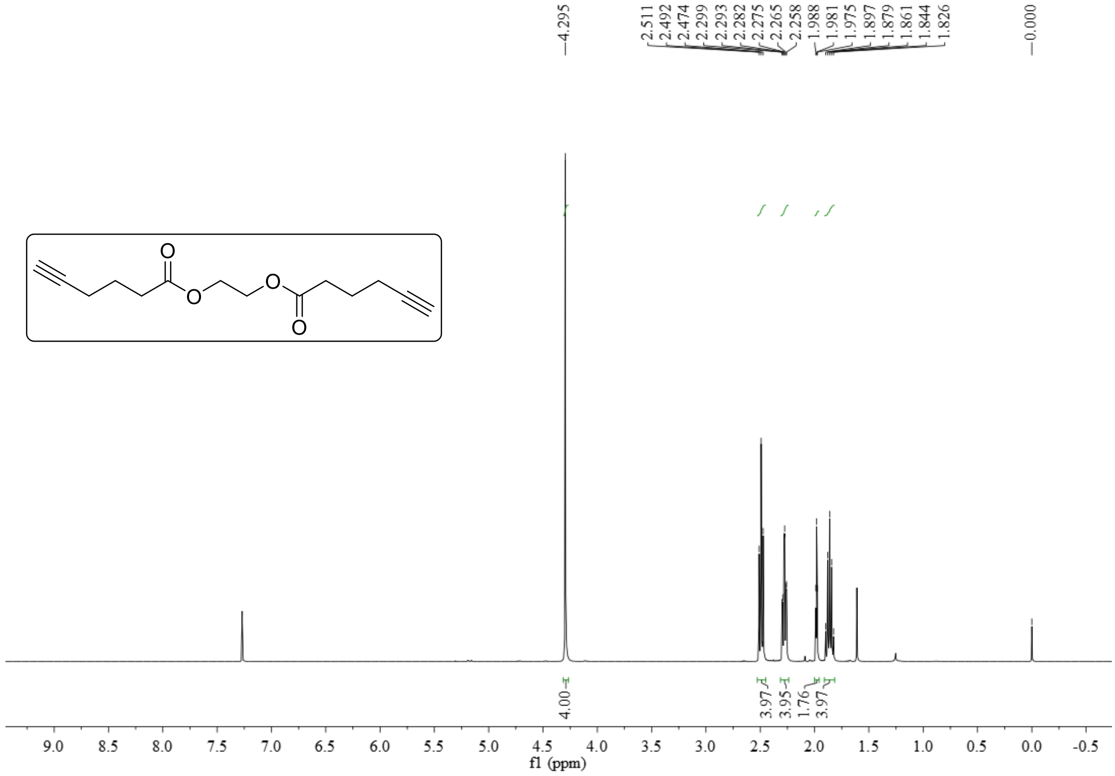


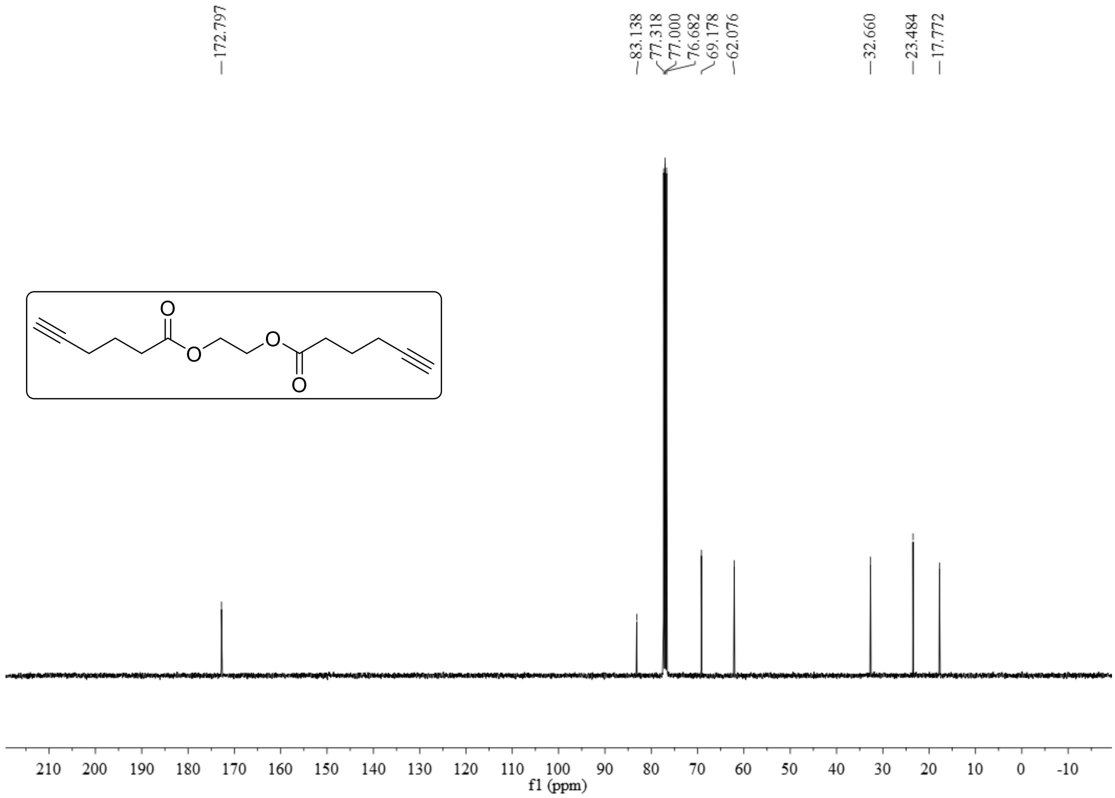
***Di*-tert-butyl 1,2-di(prop-2-yn-1-yl)hydrazine-1,2-dicarboxylate (M11)**



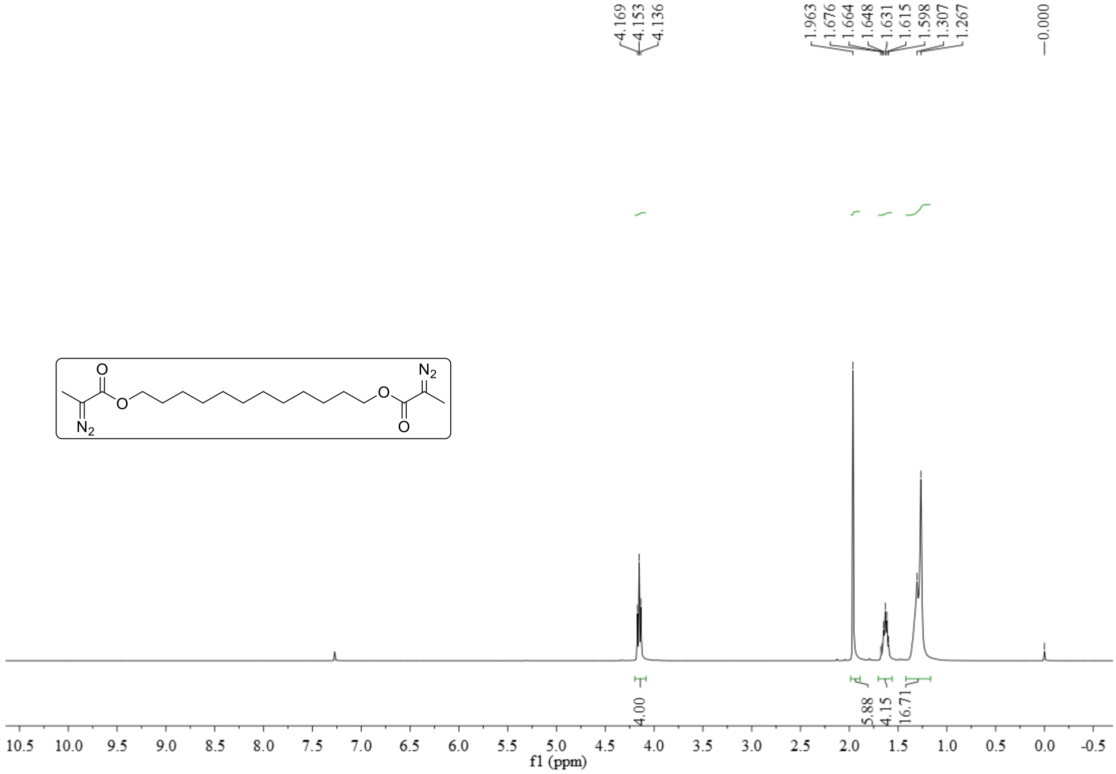


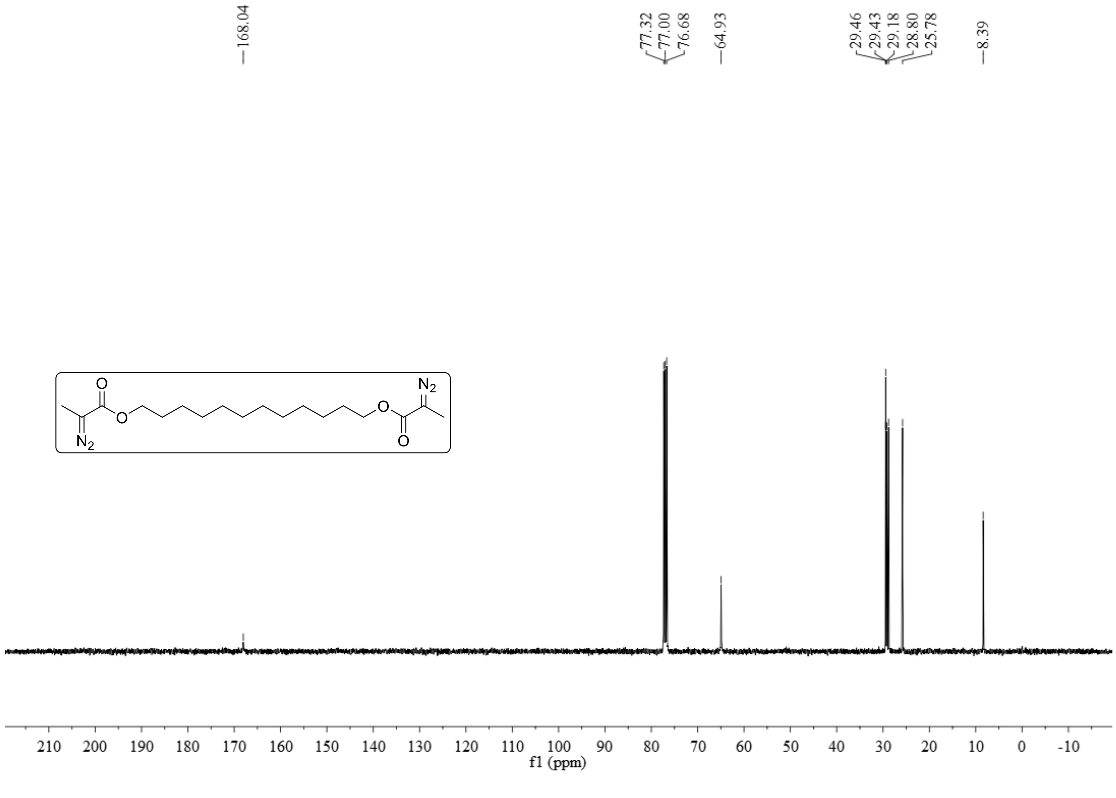
Ethane-1,2-diyl bis(hex-5-ynoate) (M12)



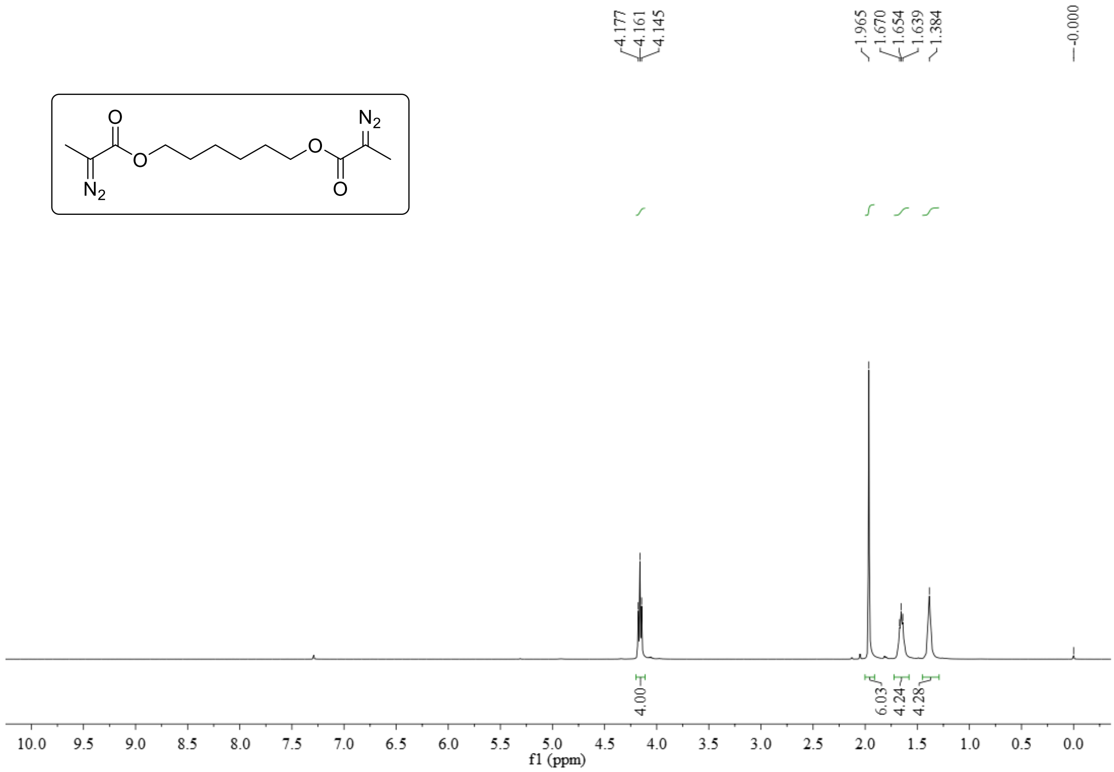


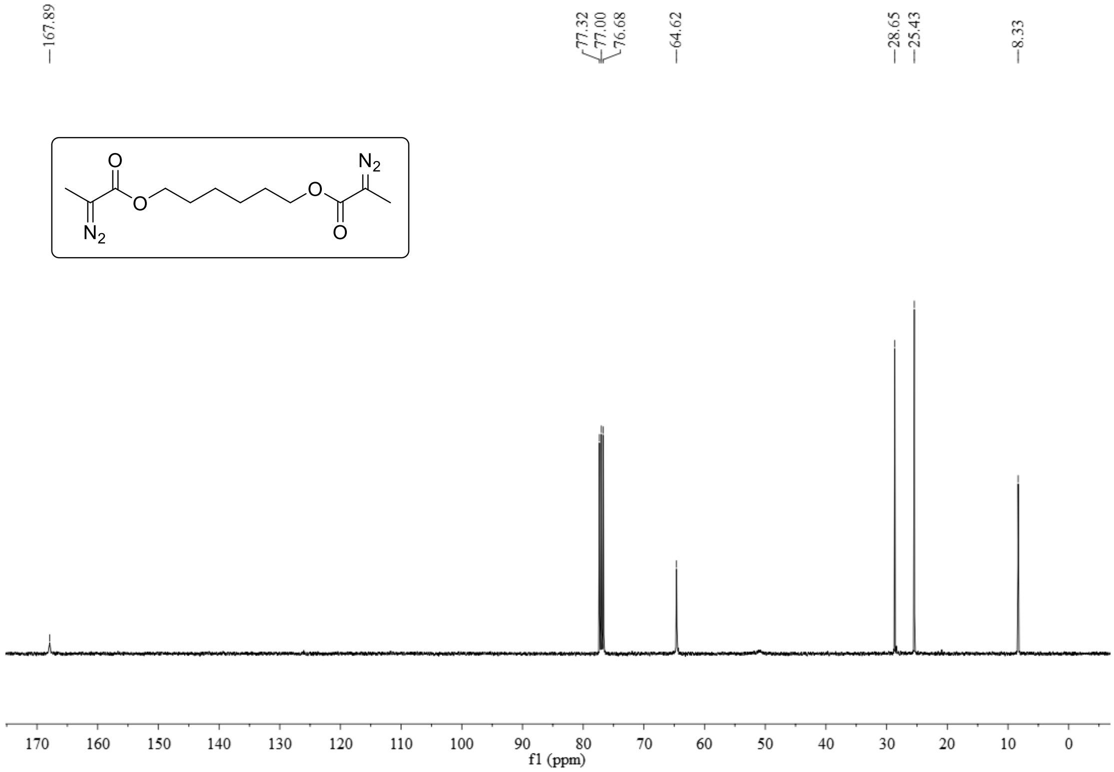
Dodecane-1,12-diyl bis(2-diazopropanoate) (N1)



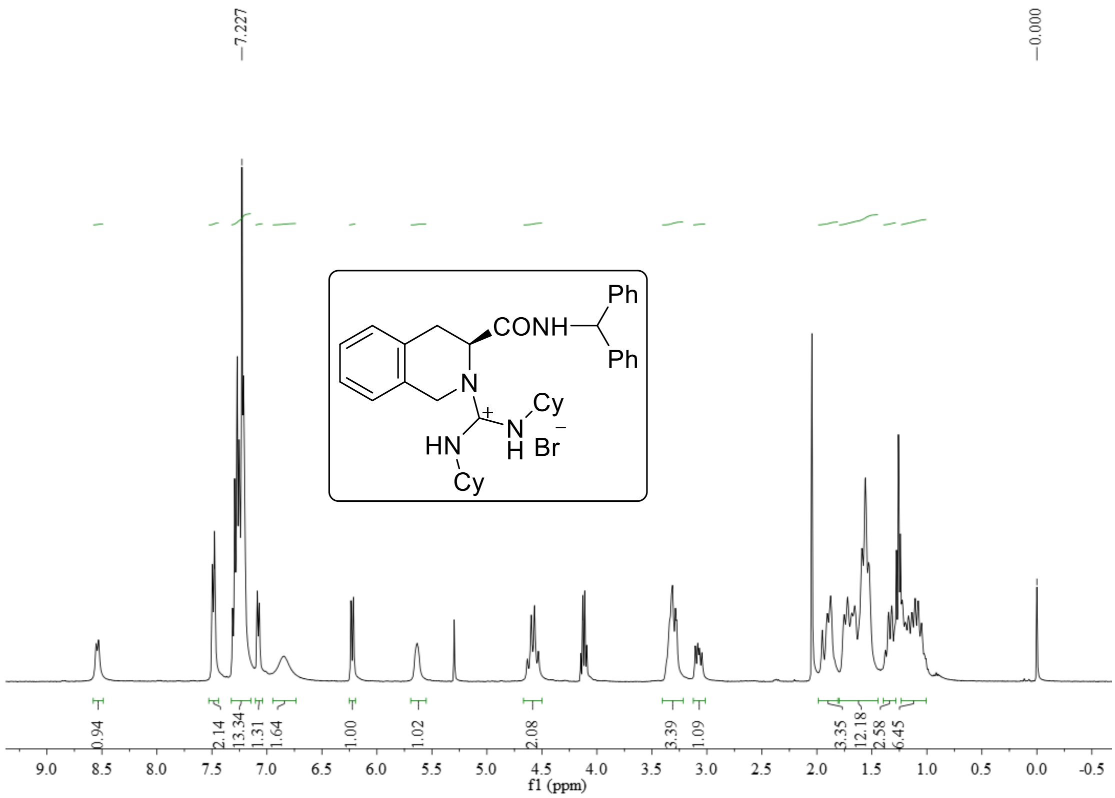


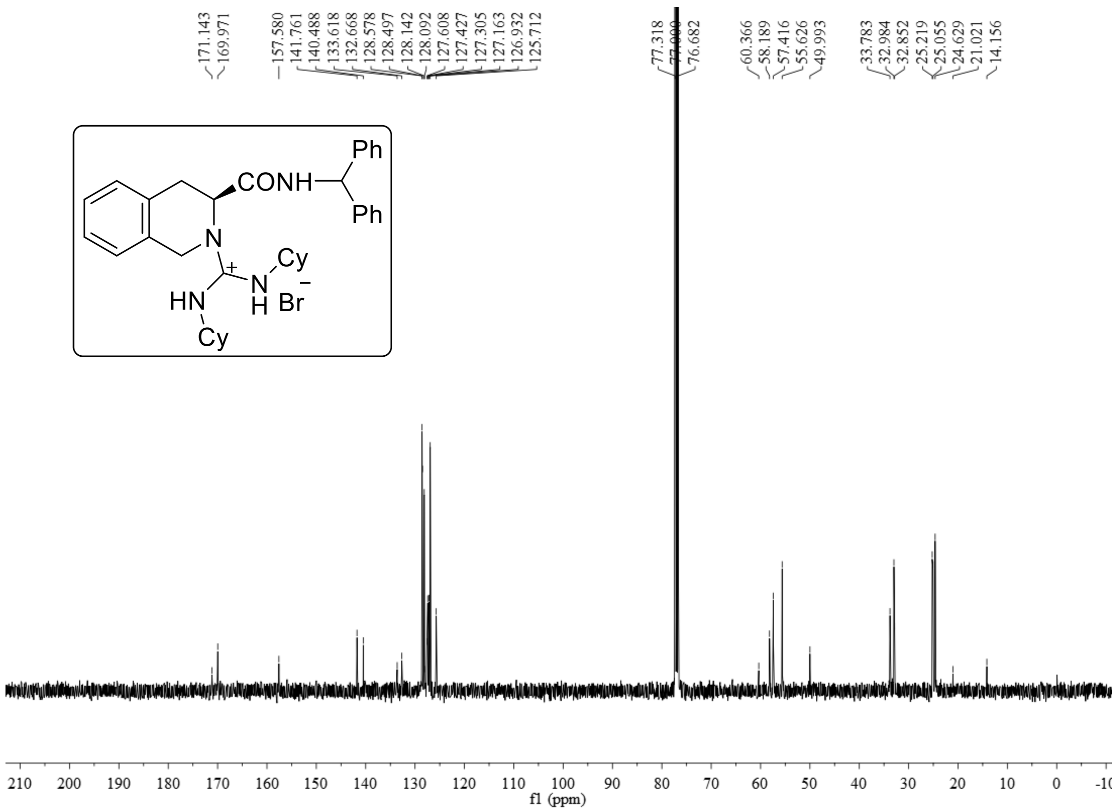
Hexane-1,6-diyl bis(2-diazopropanoate) (N2)





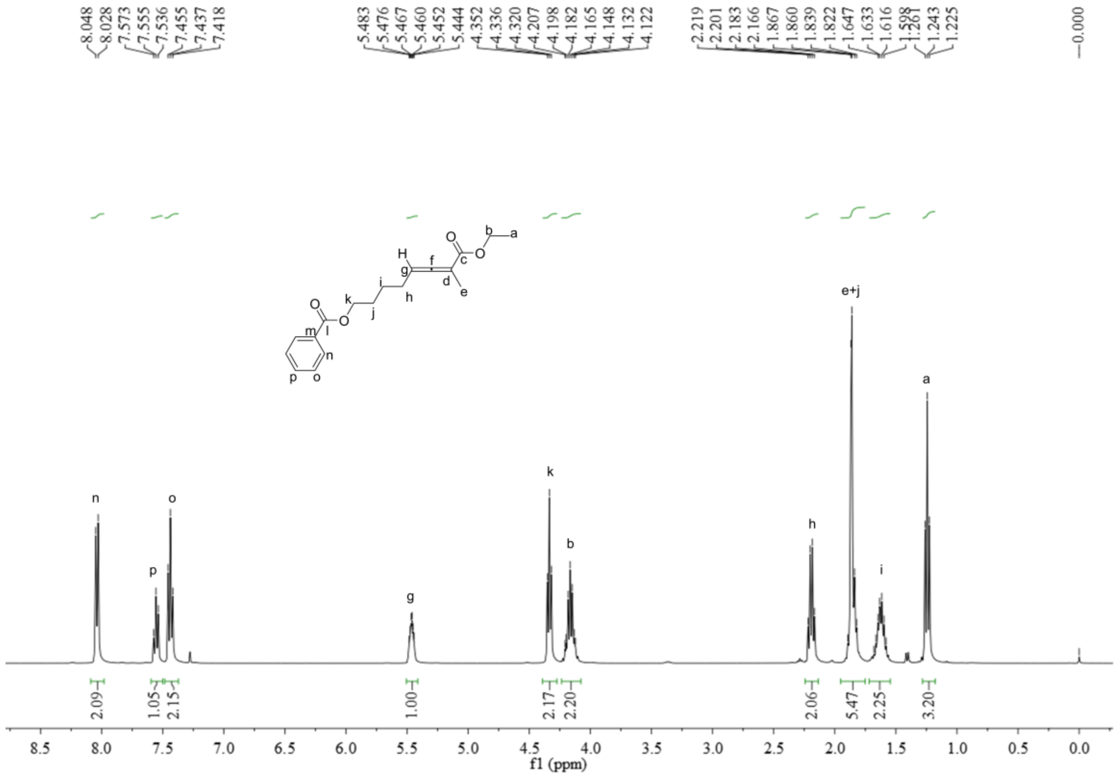
(*S*)-3-(benzhydrylcarbamoyl)-2-(bis(cyclohexylamino)methylene)-1,2,3,4-tetrahydroisoquinolin-2-ium bromide (L)

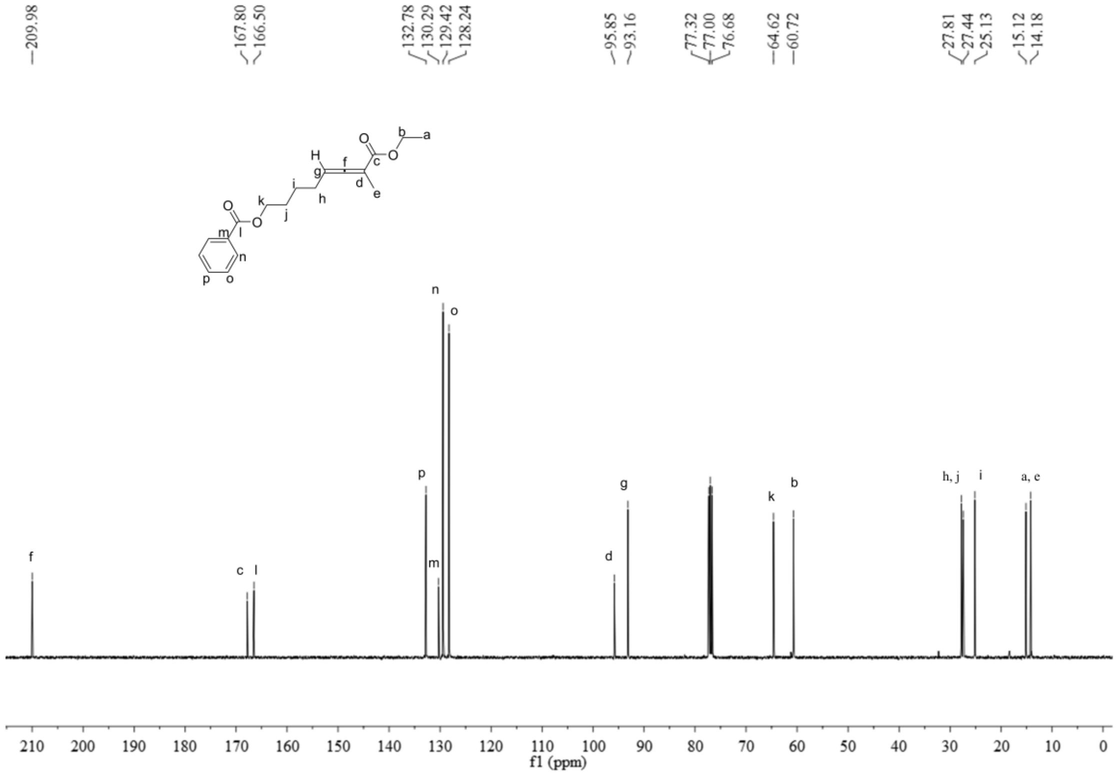




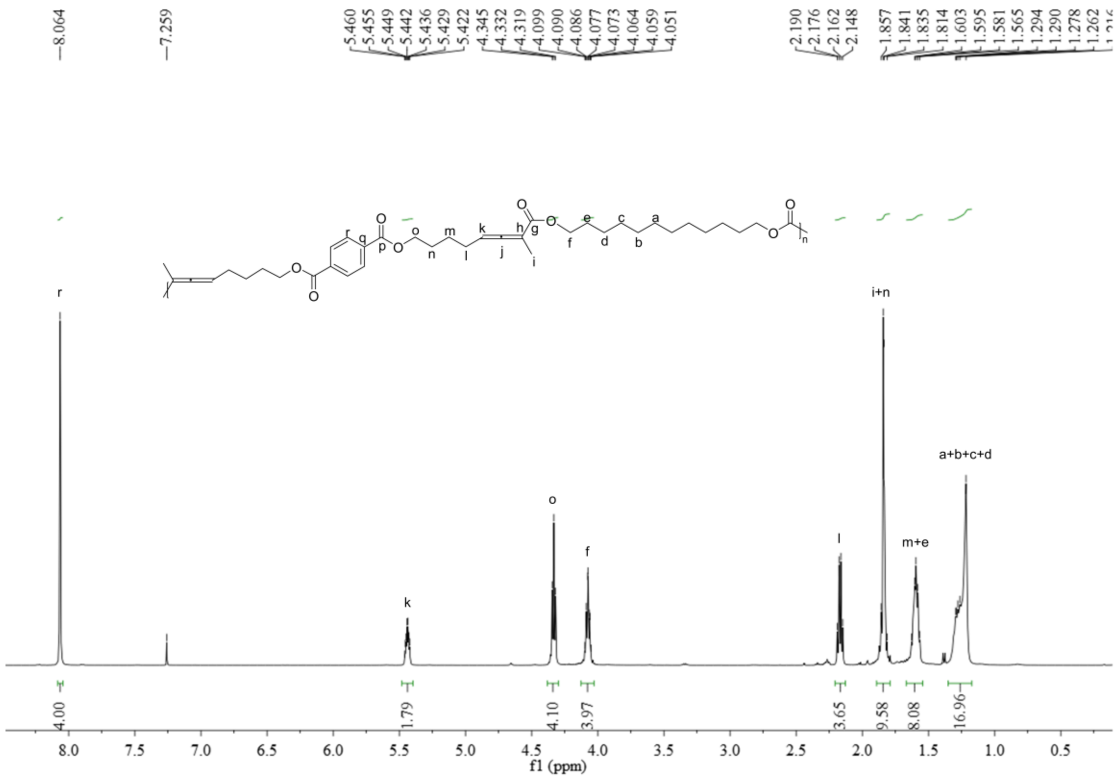
19) ^{1}H and ^{13}C NMR spectrum for S1 and polymers P1-P13

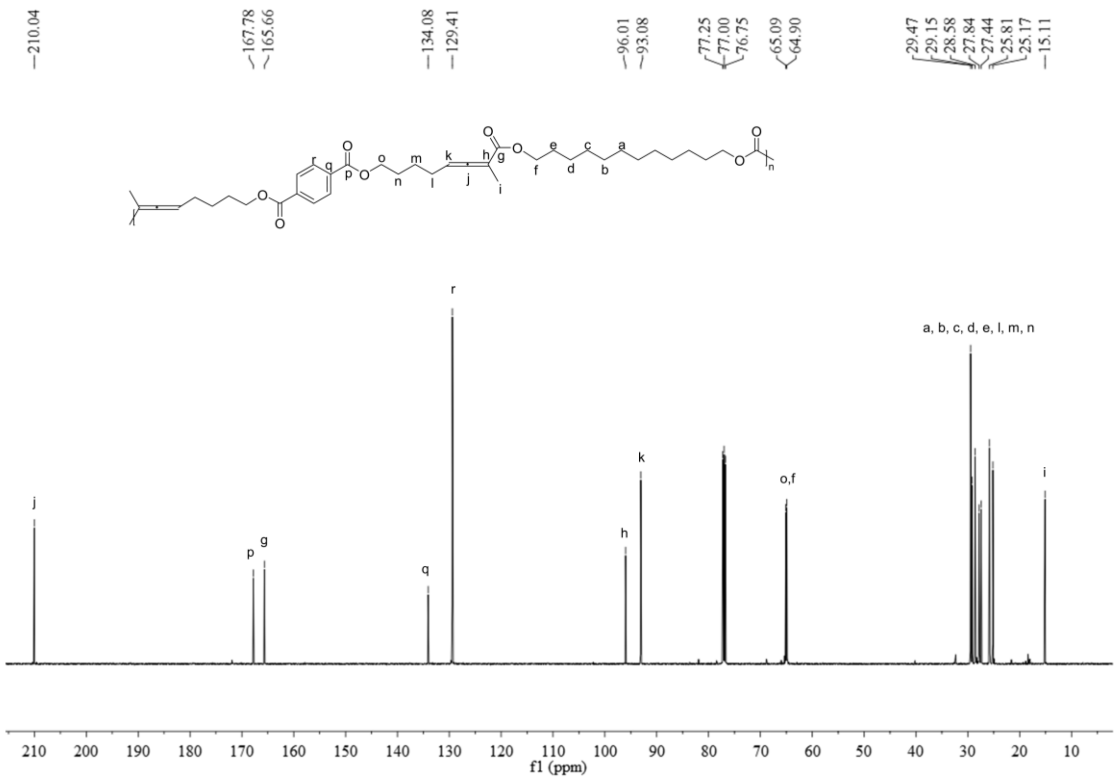
S1



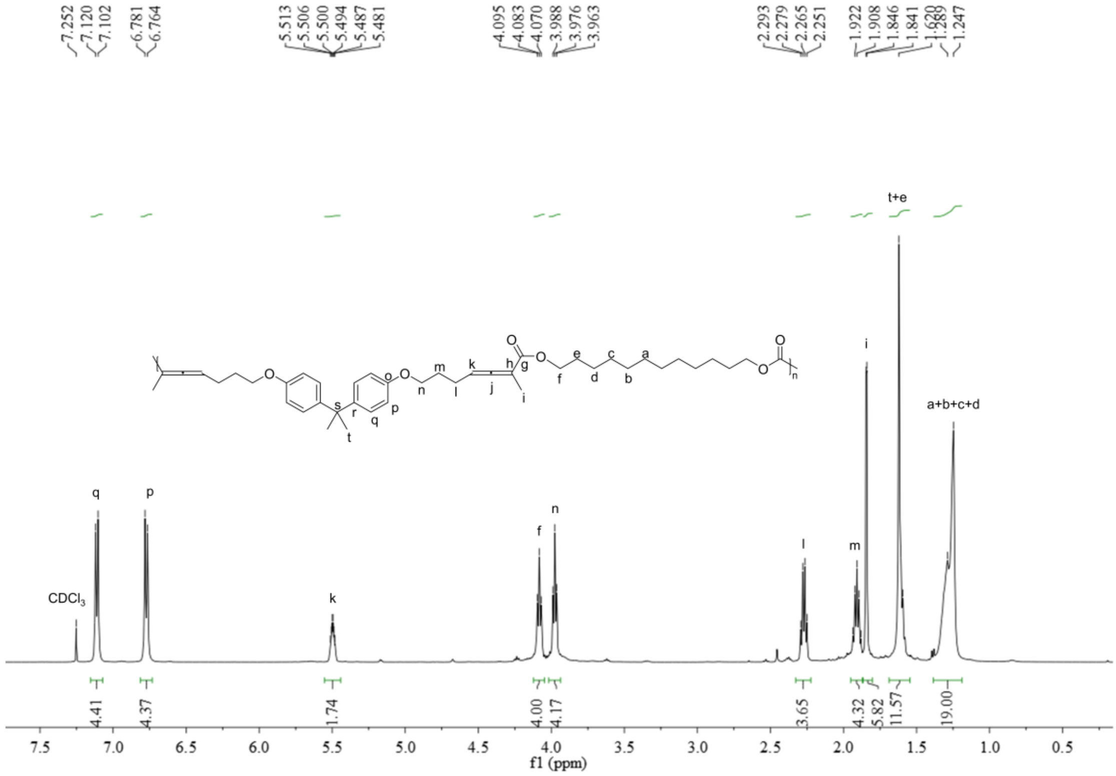


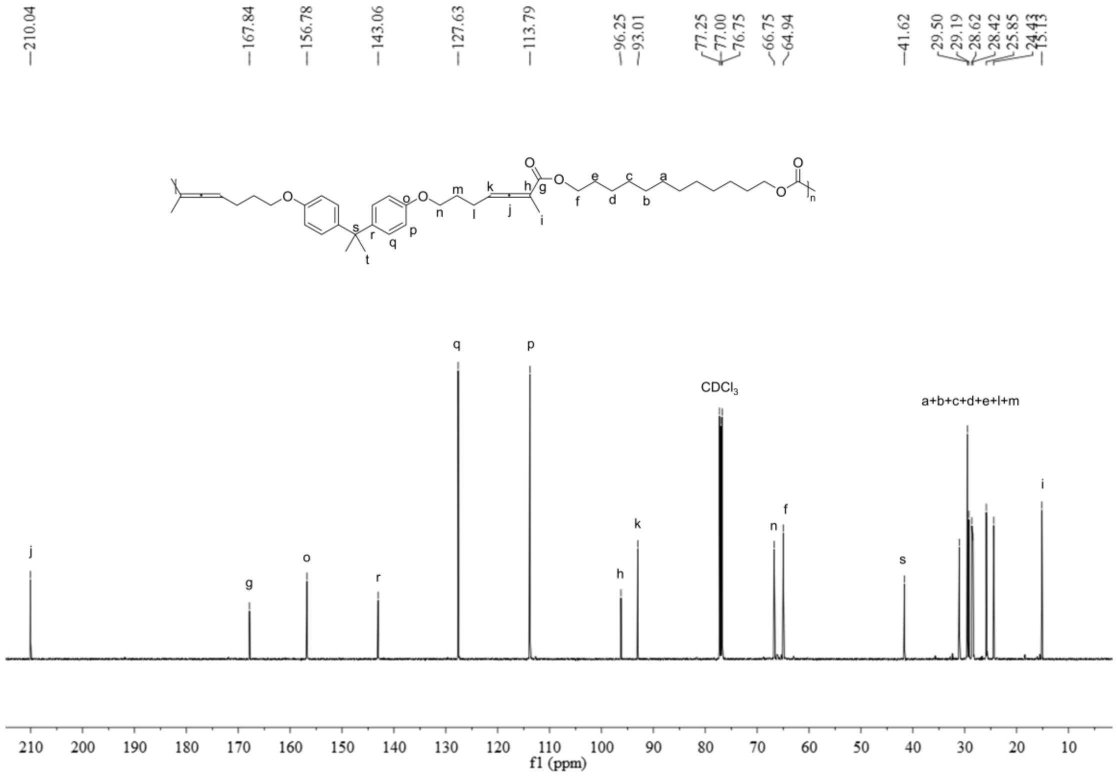
P1


8.064
7.259
5.460
5.455
5.449
5.442
5.436
5.429
5.422
4.345
4.332
4.319
4.099
4.090
4.086
4.077
4.073
4.064
4.059
4.051
2.190
2.176
2.162
2.148
1.857
1.841
1.835
1.814
1.603
1.595
1.581
1.565
1.294
1.290
1.278
1.262
r
i+n
a+b+c+d
o
f
k
l
m+e
4.00
1.79
4.10
3.97
3.65
9.58
8.08
16.96
f1 (ppm)

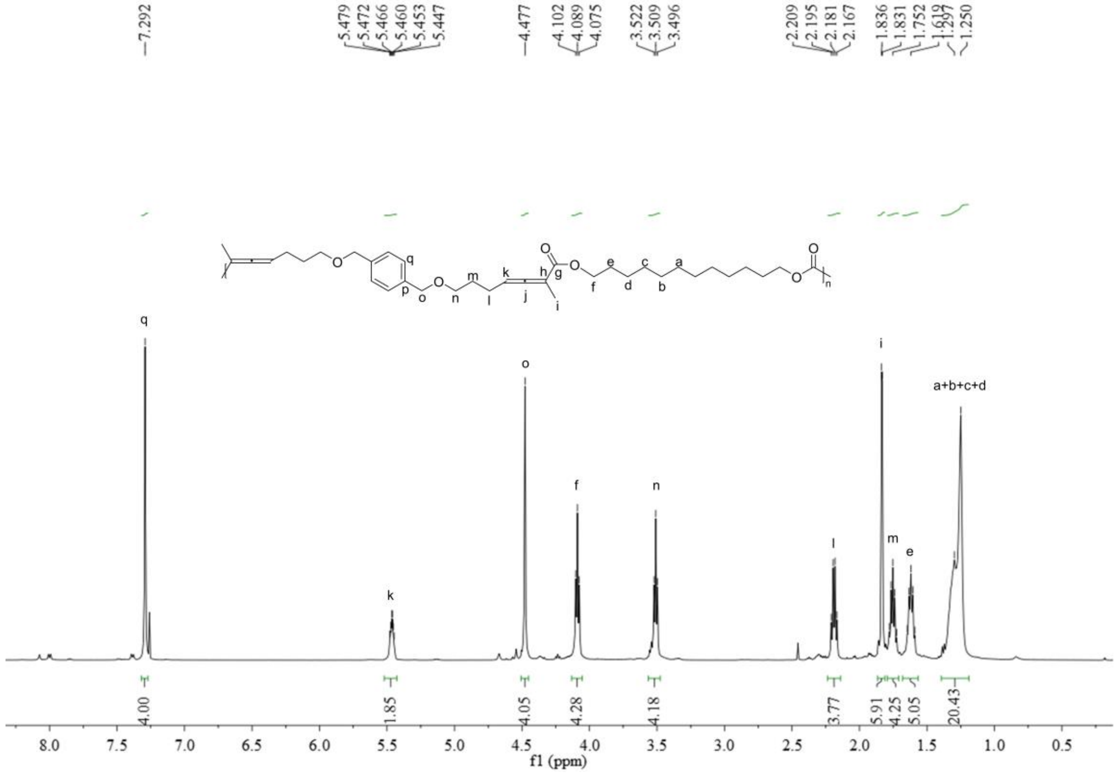

210.04
167.78
165.66
134.08
129.41
96.01
93.08
77.25
77.00
76.75
65.09
64.90
29.47
29.15
28.58
27.84
27.44
25.81
25.17
15.11
r
a, b, c, d, e, l, m, n
j
p
g
q
h
k
o,f
i
f1 (ppm)

P2

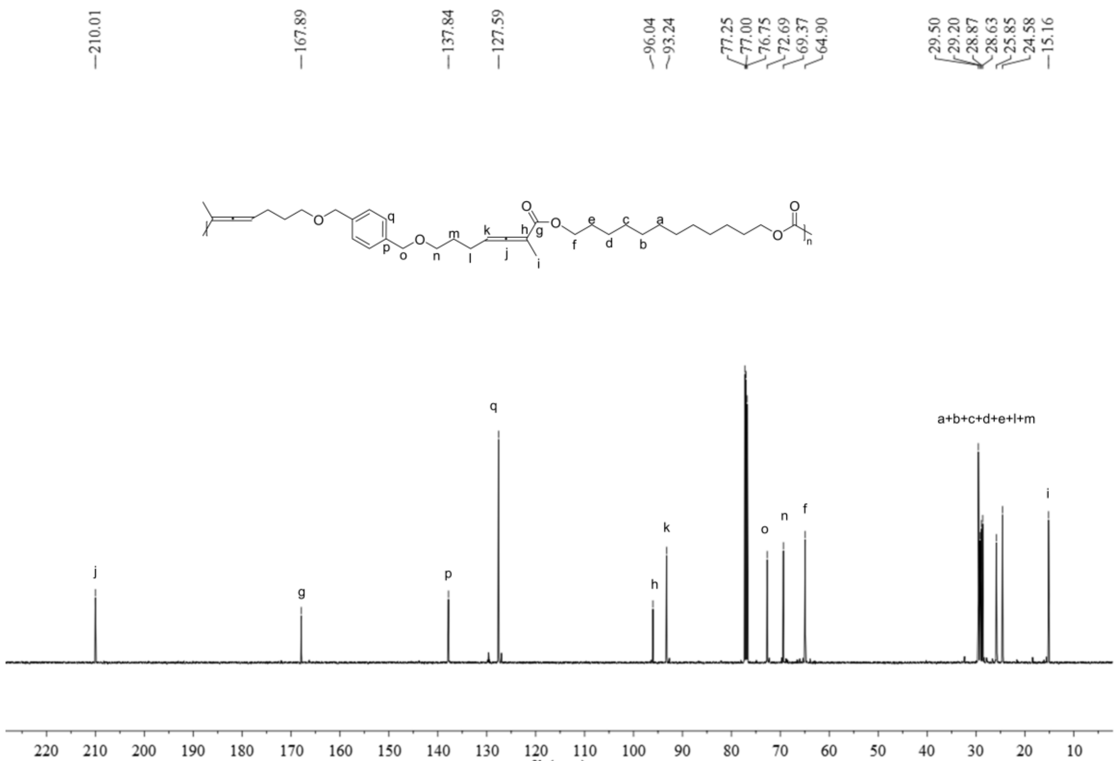

7.252
7.120
7.102
6.781
6.764
5.513
5.506
5.500
5.494
5.487
5.481
4.095
4.083
4.070
3.988
3.976
3.963
2.293
2.279
2.265
2.251
1.922
1.908
1.846
1.841
1.620
1.289
1.247
t+e
a+b+c+d
CDCl3
q
p
k
f
n
l
m
i
4.41
4.37
1.74
4.00
4.17
3.65
4.32
5.82
11.57
19.00
f1 (ppm)


210.04
167.84
156.78
143.06
127.63
113.79
96.25
93.01
77.25
77.00
76.75
66.75
64.94
41.62
29.50
29.19
28.62
28.42
25.85
24.43
15.13
q
p
CDCl3
a+b+c+d+e+l+m
j
g
o
r
h
k
n
f
s
i
f1 (ppm)

P3

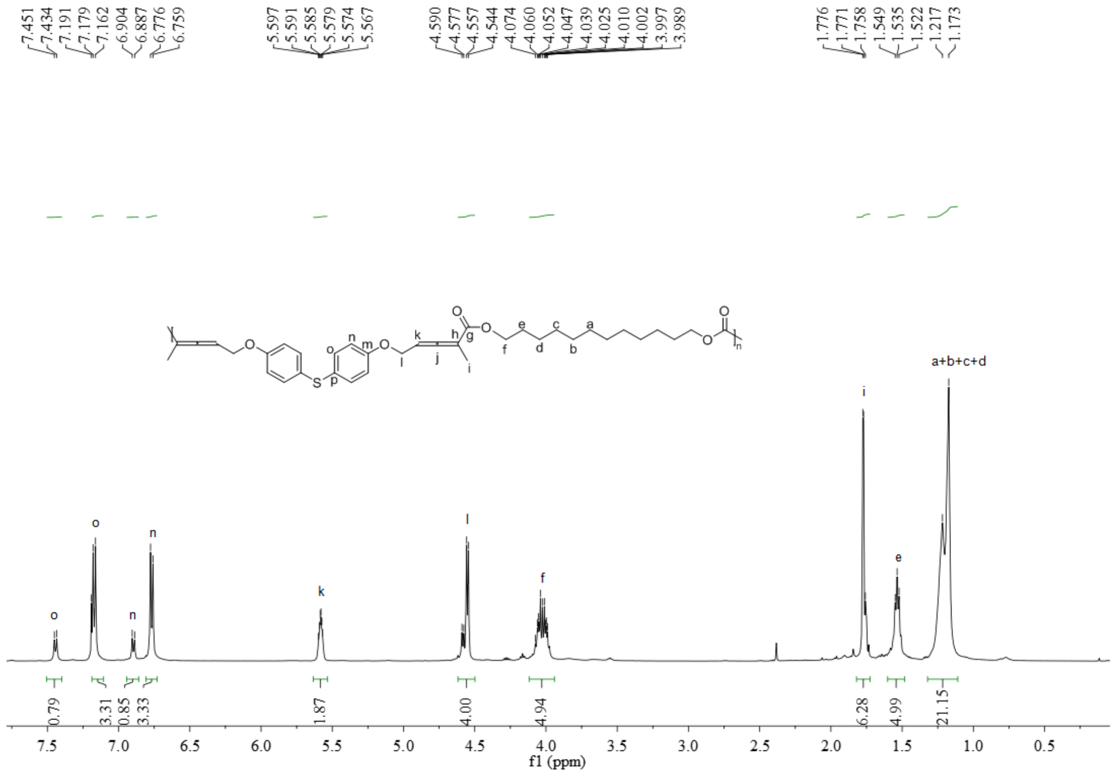


7.292
5.479
5.472
5.466
5.460
5.453
5.447
4.477
4.102
4.089
4.075
3.522
3.509
3.496
2.209
2.195
2.181
2.167
1.836
1.831
1.752
1.619
1.297
1.250
q
o
f
n
k
l
i
m
e
a+b+c+d
4.00
1.85
4.05
4.28
4.18
3.77
5.91
4.25
5.05
20.43
f1 (ppm)



210.01
167.89
137.84
127.59
96.04
93.24
77.25
77.00
76.75
72.69
69.37
64.90
29.50
29.20
28.87
28.63
25.85
24.58
15.16
j
g
p
q
h
k
o
n
f
a+b+c+d+e+l+m
i
f1 (ppm)

P4


7.451
7.434
7.191
7.179
7.162
6.904
6.887
6.776
6.759
5.597
5.591
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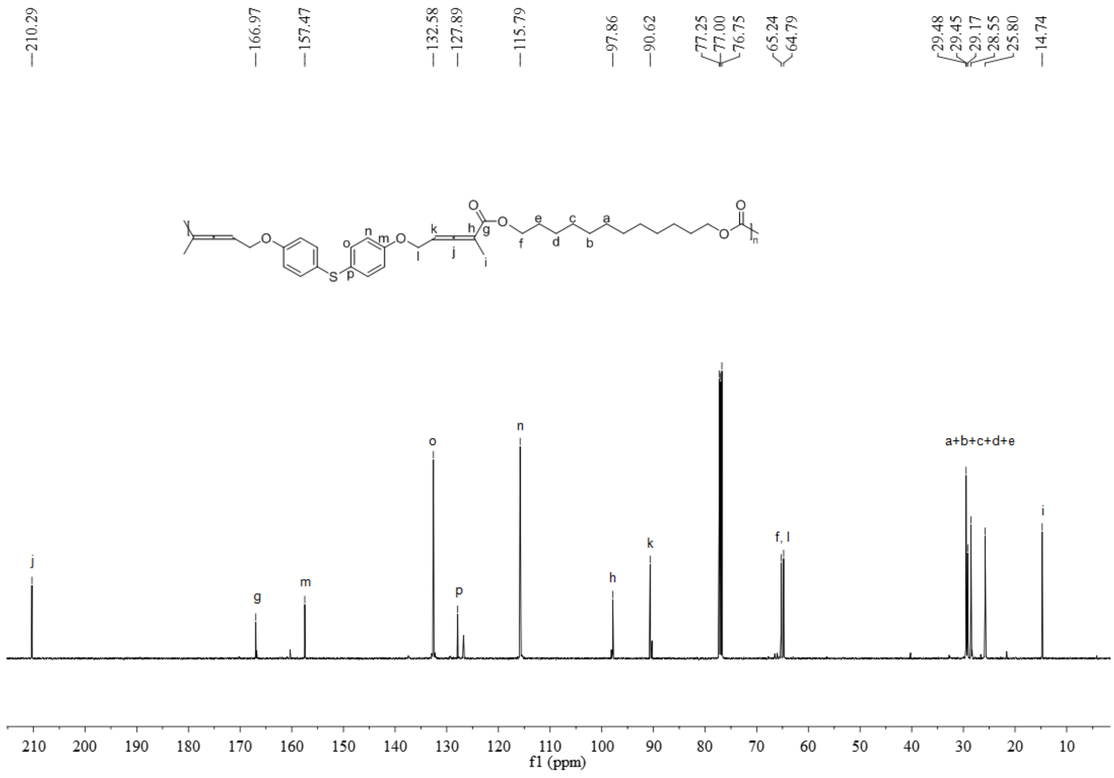
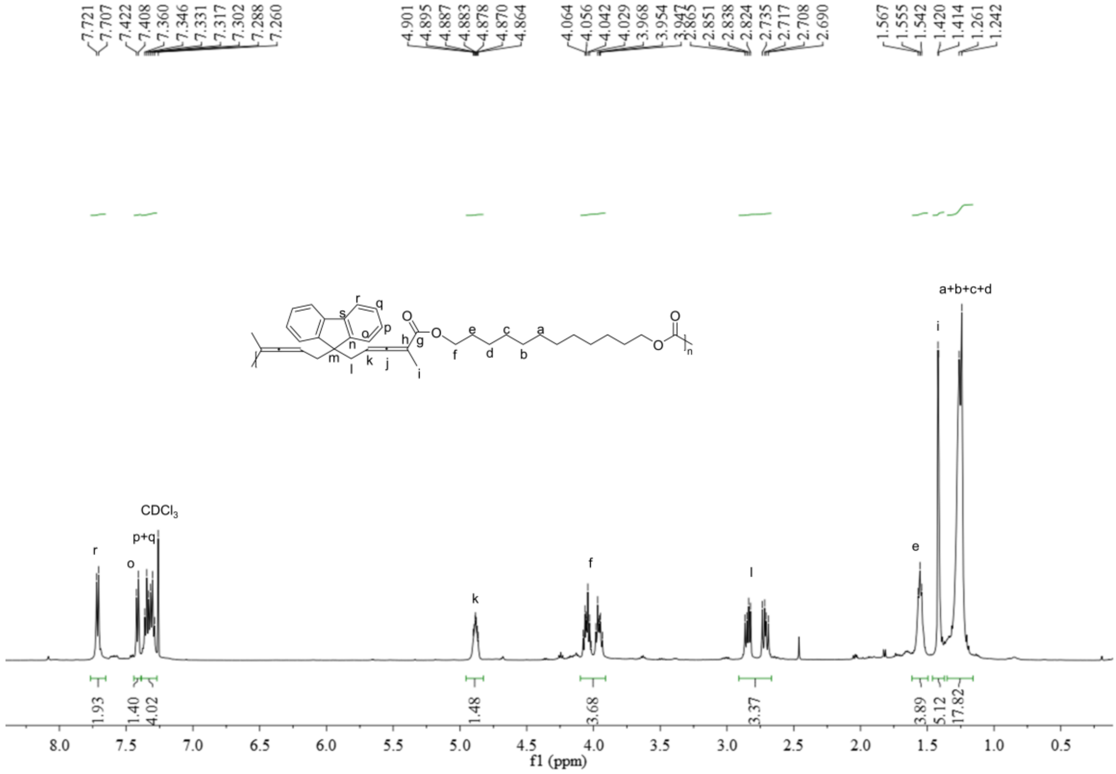
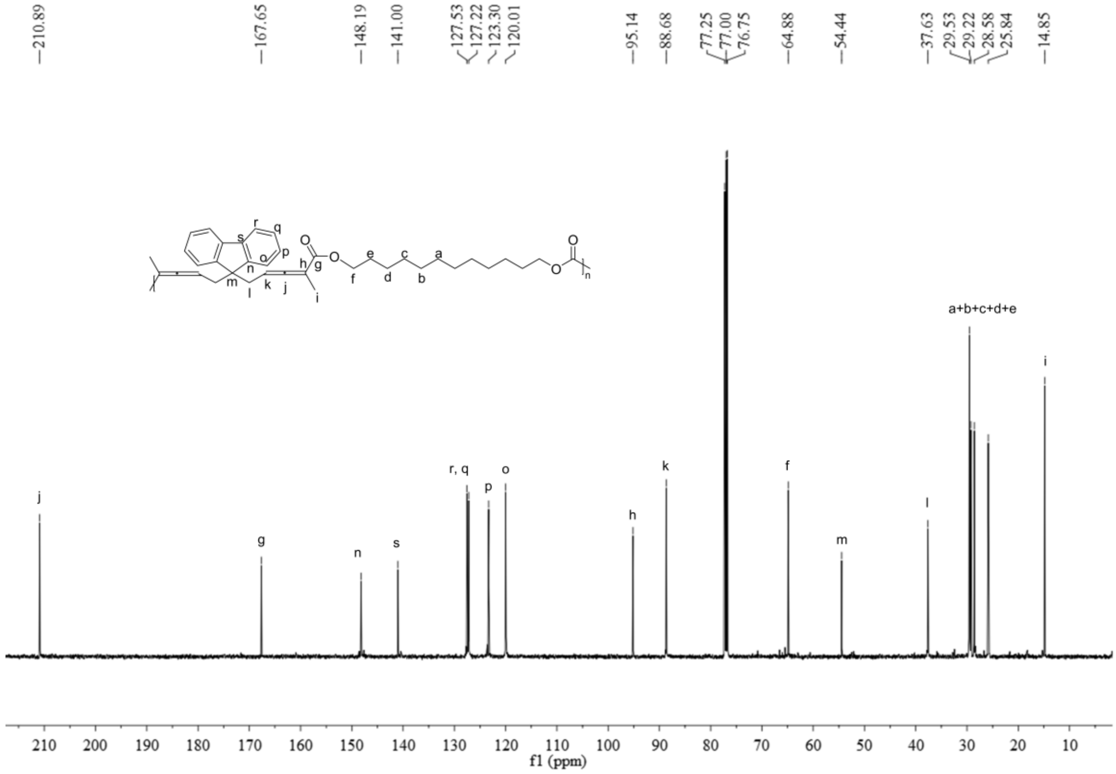
5.579
5.574
5.567
4.590
4.577
4.557
4.544
4.074
4.060
4.052
4.047
4.039
4.025
4.010
4.002
3.997
3.989
1.776
1.771
1.758
1.549
1.535
1.522
1.217
1.173
a+b+c+d
0.79
3.31
0.85
3.33
1.87
4.00
4.94
6.28
4.99
21.15
f1 (ppm)

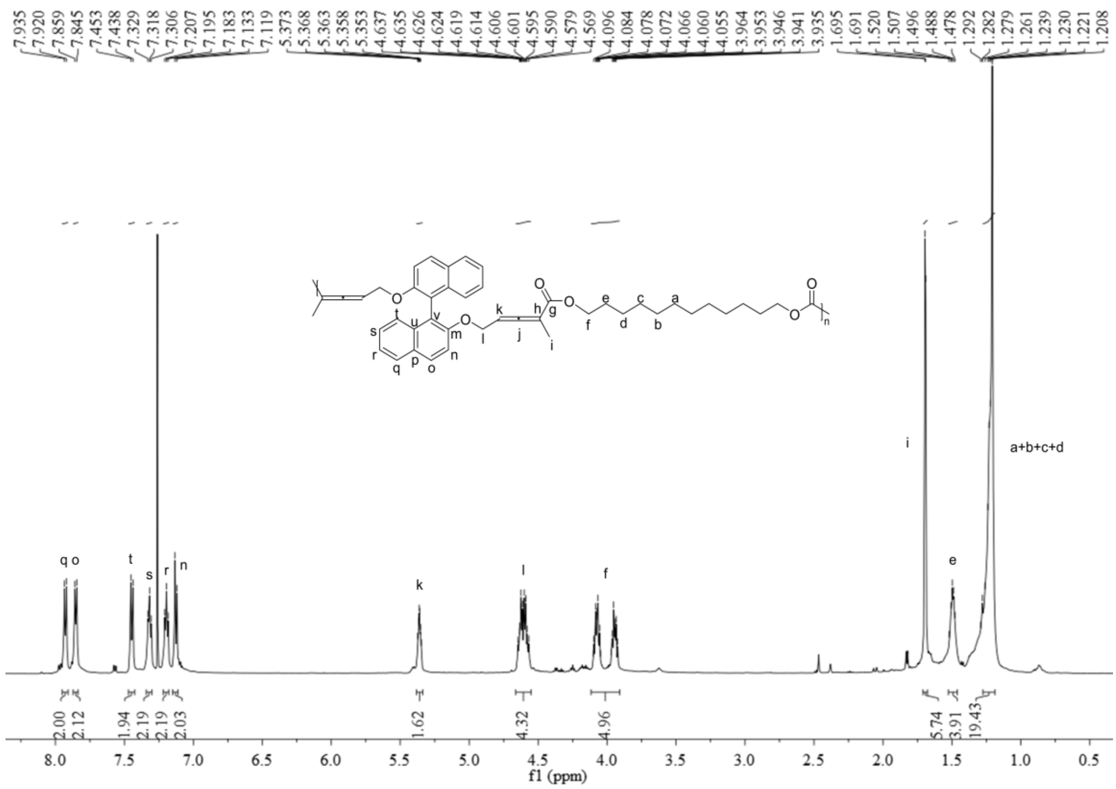

210.29
166.97
157.47
132.58
127.89
115.79
97.86
90.62
77.25
77.00
76.75
65.24
64.79
29.48
29.45
29.17
28.55
25.80
14.74
a+b+c+d+e
f, l
f1 (ppm)

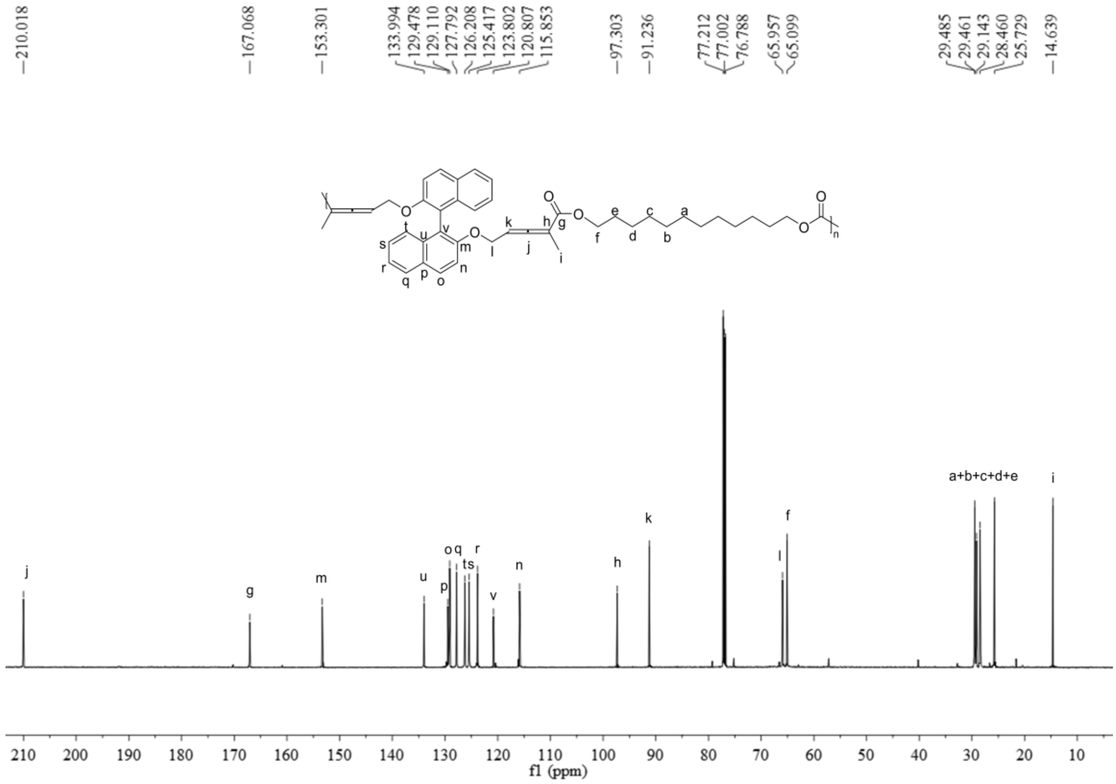
P5


7.721
7.707
7.422
7.408
7.360
7.346
7.331
7.317
7.302
7.288
7.260
4.901
4.895
4.887
4.883
4.878
4.870
4.864
4.064
4.056
4.042
4.029
3.968
3.954
3.947
2.865
2.851
2.838
2.824
2.735
2.717
2.708
2.690
1.567
1.555
1.542
1.420
1.414
1.261
1.242
a+b+c+d
i
CDCl3
p+q
r
o
k
f
l
e
1.93
1.40
4.02
1.48
3.68
3.37
3.89
5.12
17.82
f1 (ppm)

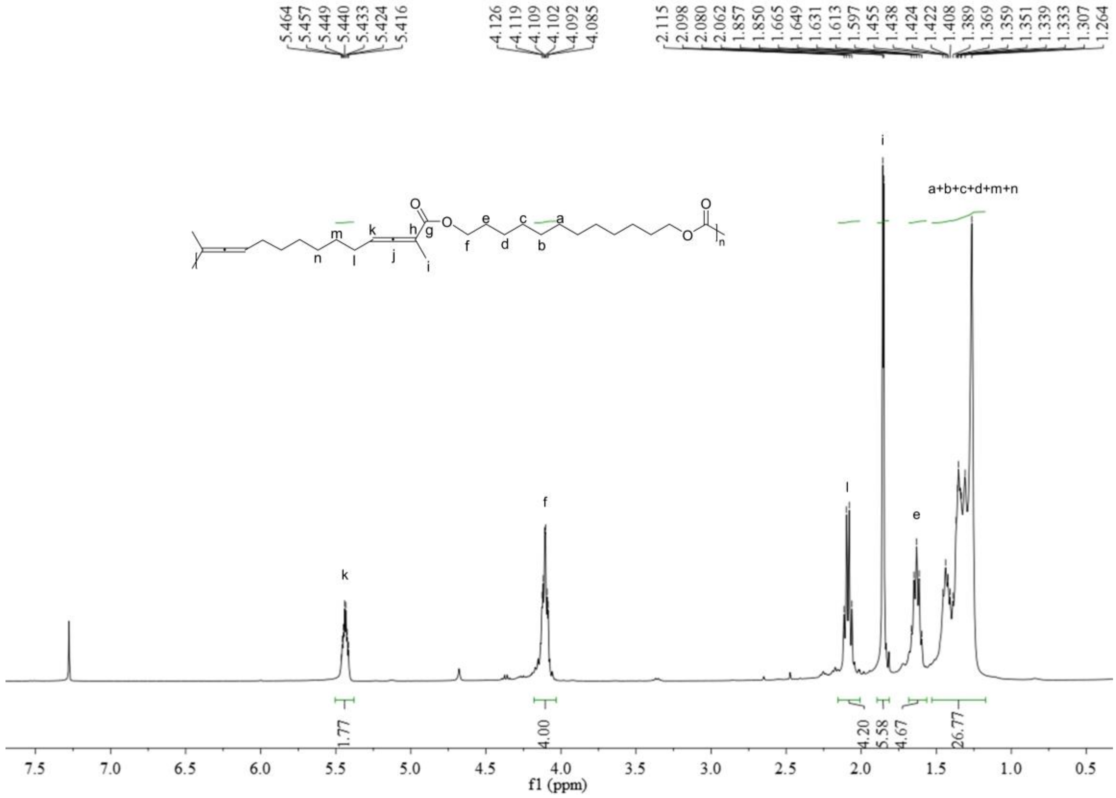

210.89
167.65
148.19
141.00
127.53
127.22
123.30
120.01
95.14
88.68
77.25
77.00
76.75
64.88
54.44
37.63
29.53
29.22
28.58
25.84
14.85
a+b+c+d+e
i
j
g
n
s
r, q
p
o
h
k
f
m
l
f1 (ppm)

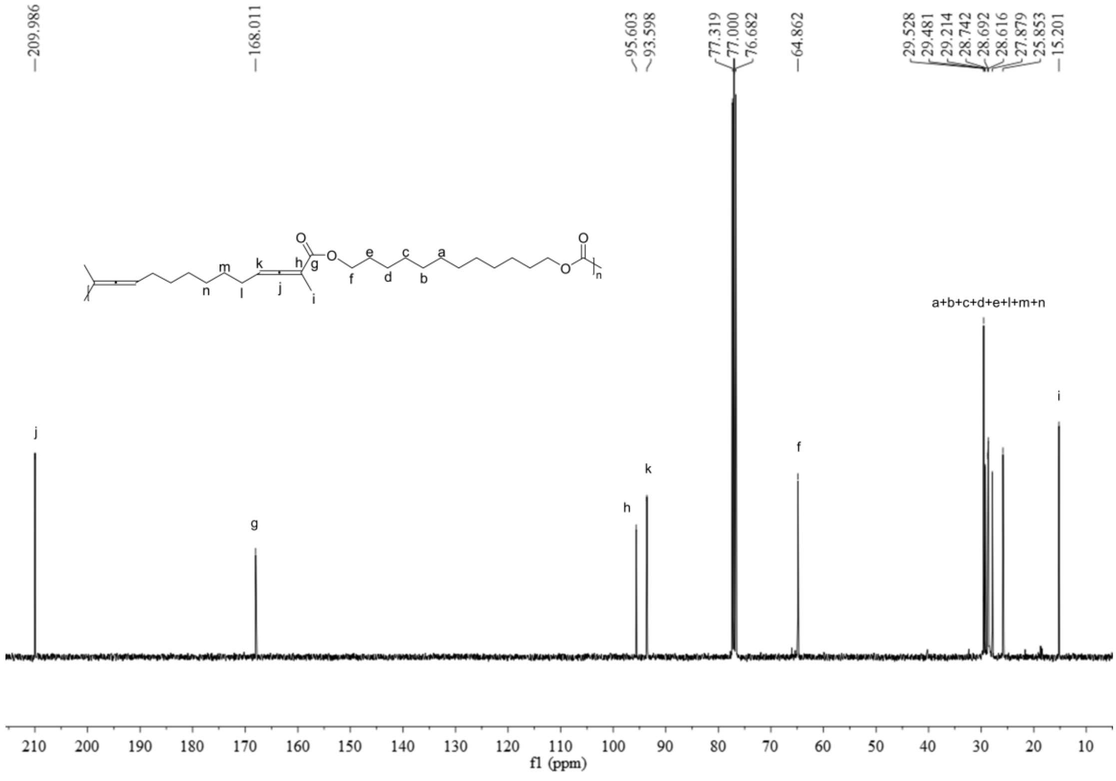
P6


q o
t
s
r
n
k
l
f
i
e
a+b+c+d
2.00
2.12
1.94
2.19
2.19
2.03
1.62
4.32
4.96
5.74
3.91
19.43
f1 (ppm)

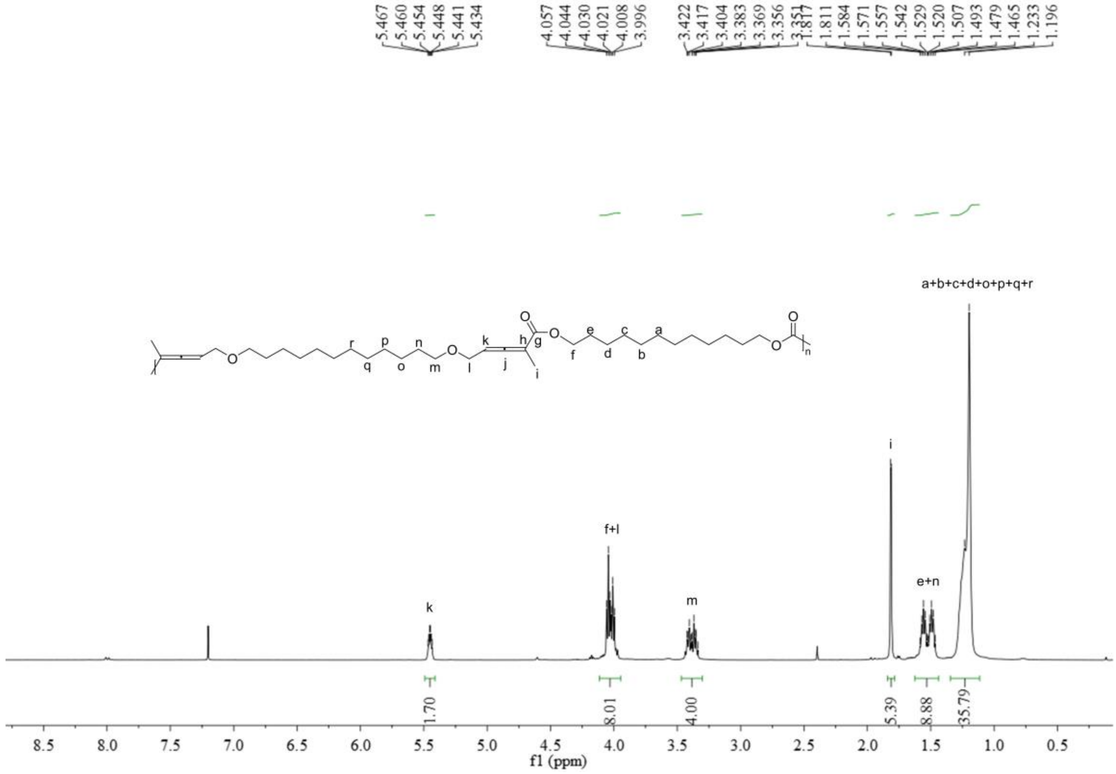

j
g
m
u
p
o q
t s
r
n
v
h
k
l
f
a+b+c+d+e
i
f1 (ppm)

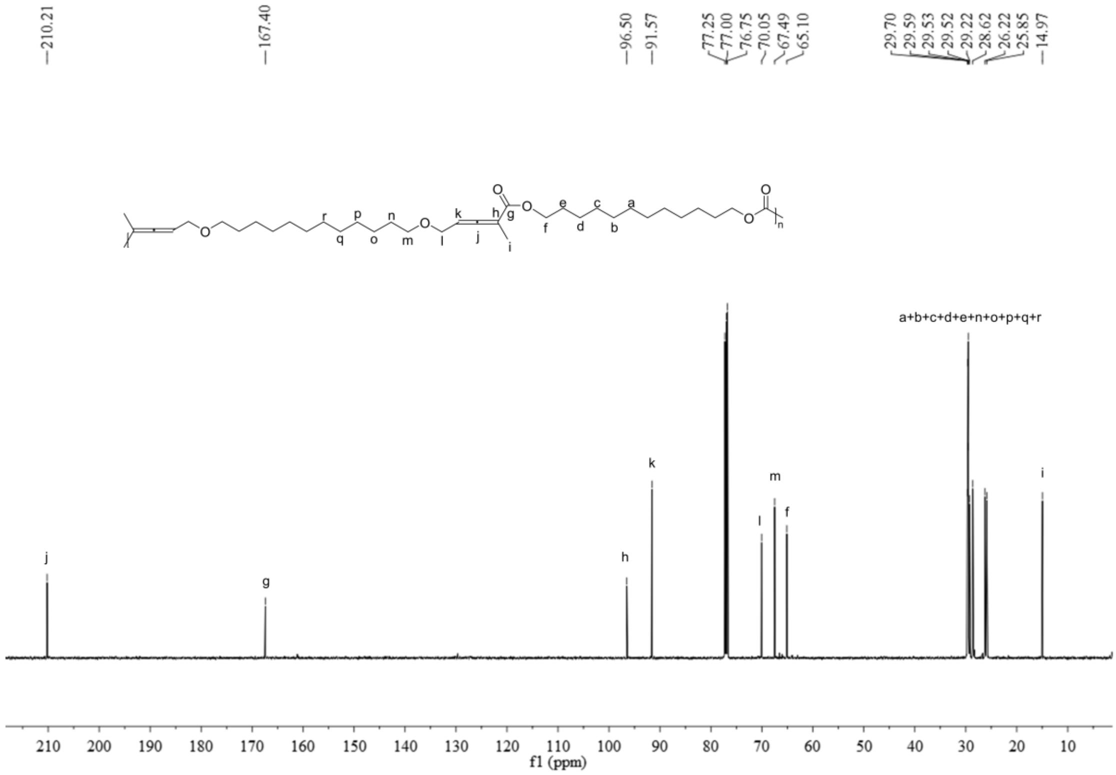
P7



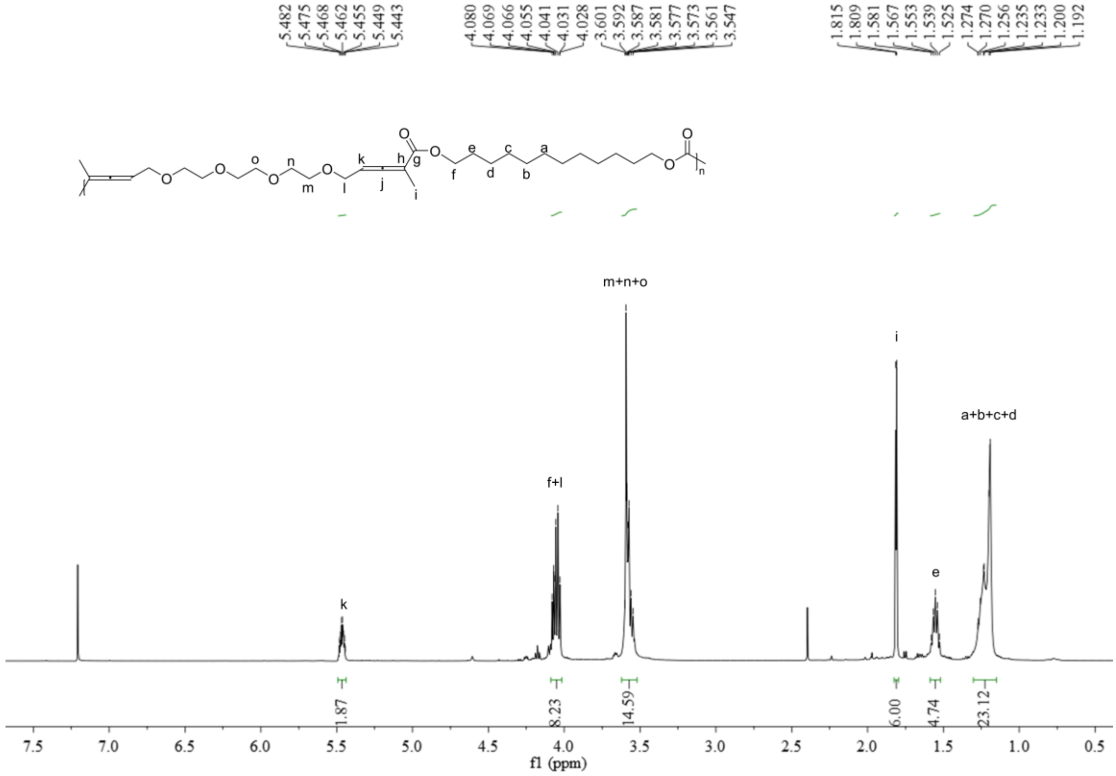


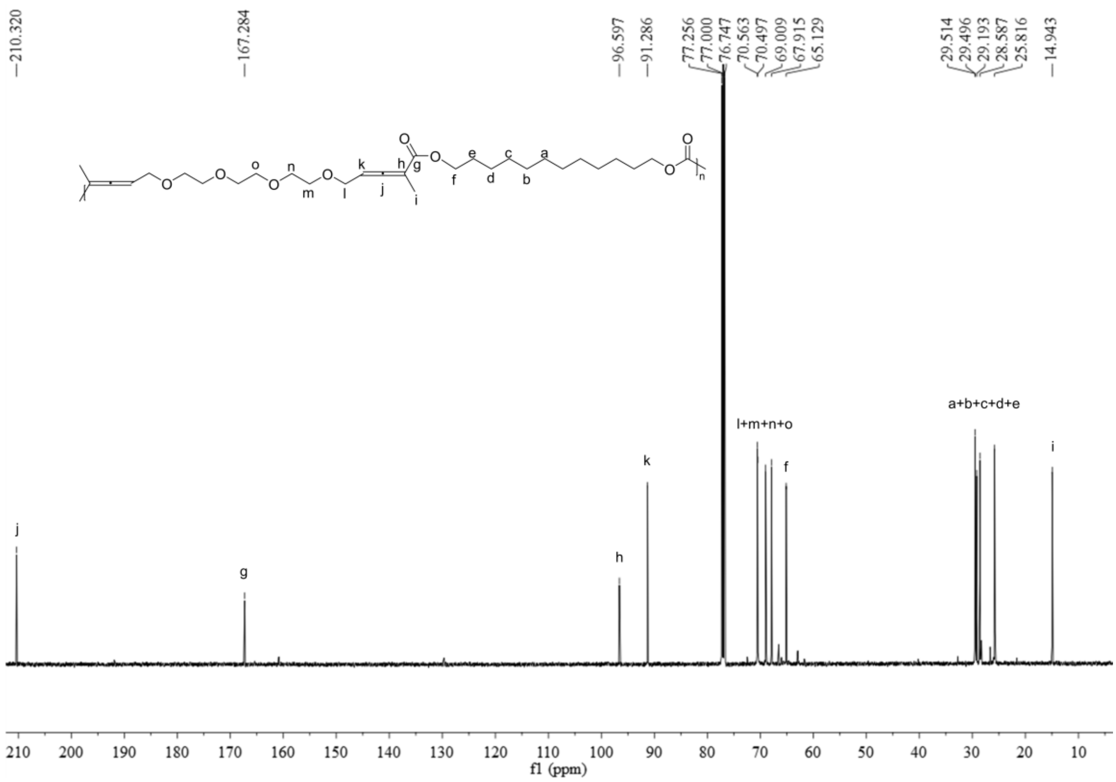
P8


5.467
5.460
5.454
5.448
5.441
5.434
4.057
4.044
4.030
4.021
4.008
3.996
3.422
3.417
3.404
3.383
3.369
3.356
3.351
1.817
1.811
1.584
1.571
1.557
1.542
1.529
1.520
1.507
1.493
1.479
1.465
1.233
1.196
a+b+c+d+o+p+q+r
i
f+l
e+n
k
m
1.70
8.01
4.00
5.39
8.88
35.79
f1 (ppm)

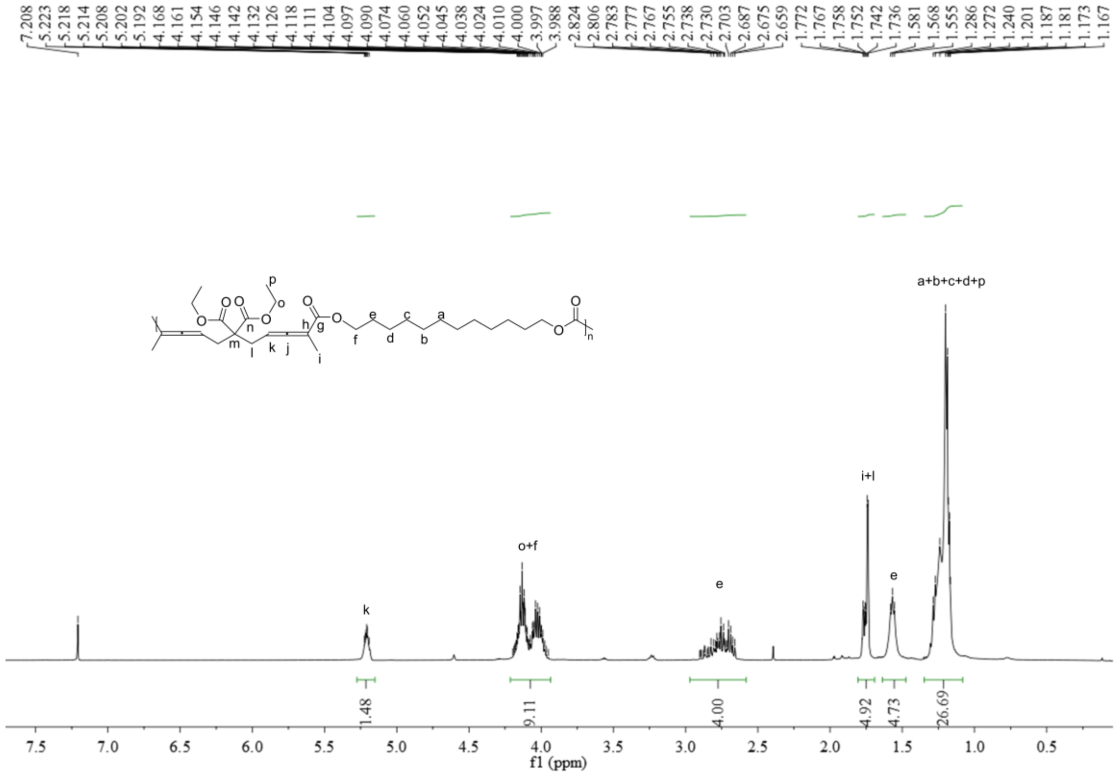

210.21
167.40
96.50
91.57
77.25
77.00
76.75
70.05
67.49
65.10
29.70
29.59
29.53
29.52
29.22
28.62
26.22
25.85
14.97
a+b+c+d+e+n+o+p+q+r
k
m
l
f
i
j
g
h
f1 (ppm)

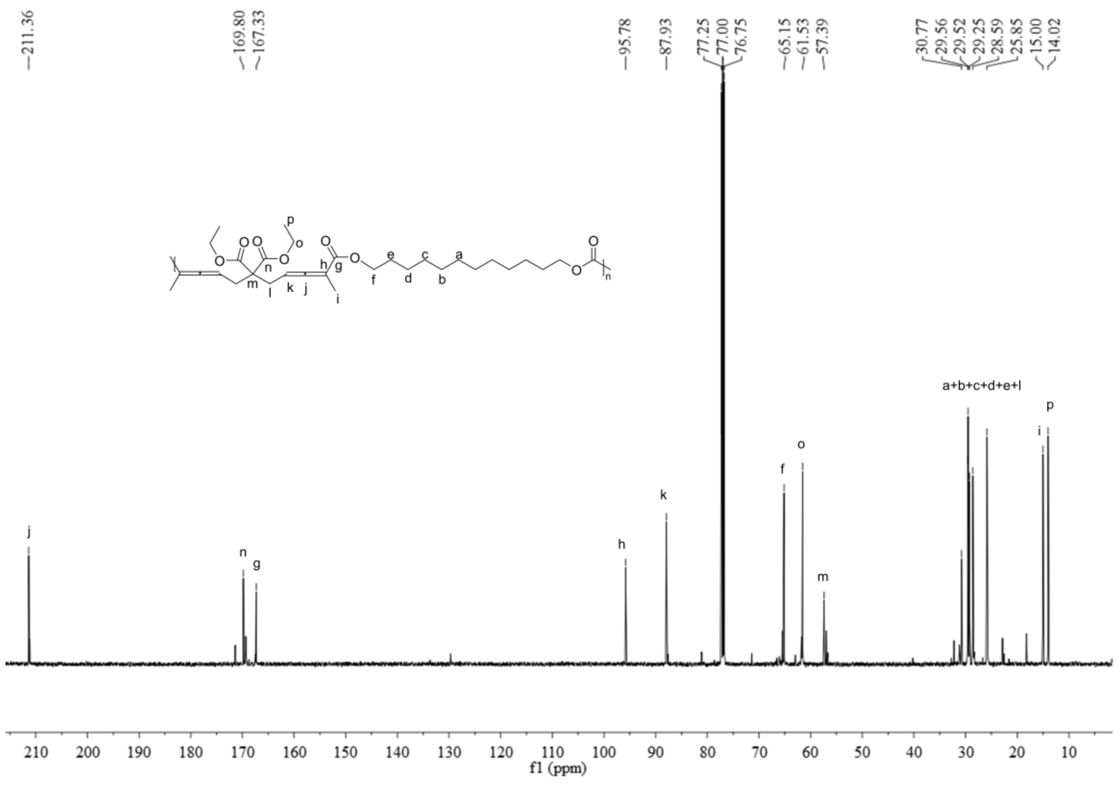
P9


m+n+o
f+l
k
i
e
a+b+c+d
1.87
8.23
14.59
6.00
4.74
23.12
f1 (ppm)

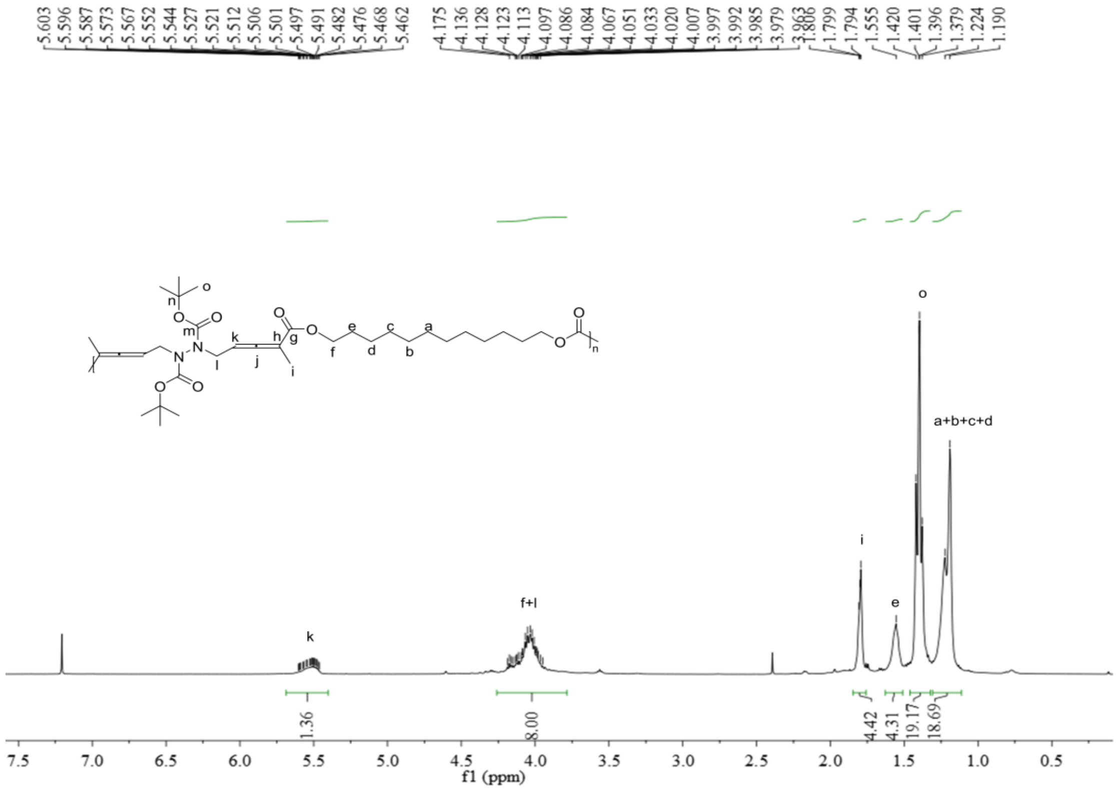

j
g
h
k
l+m+n+o
f
a+b+c+d+e
i
f1 (ppm)

P10

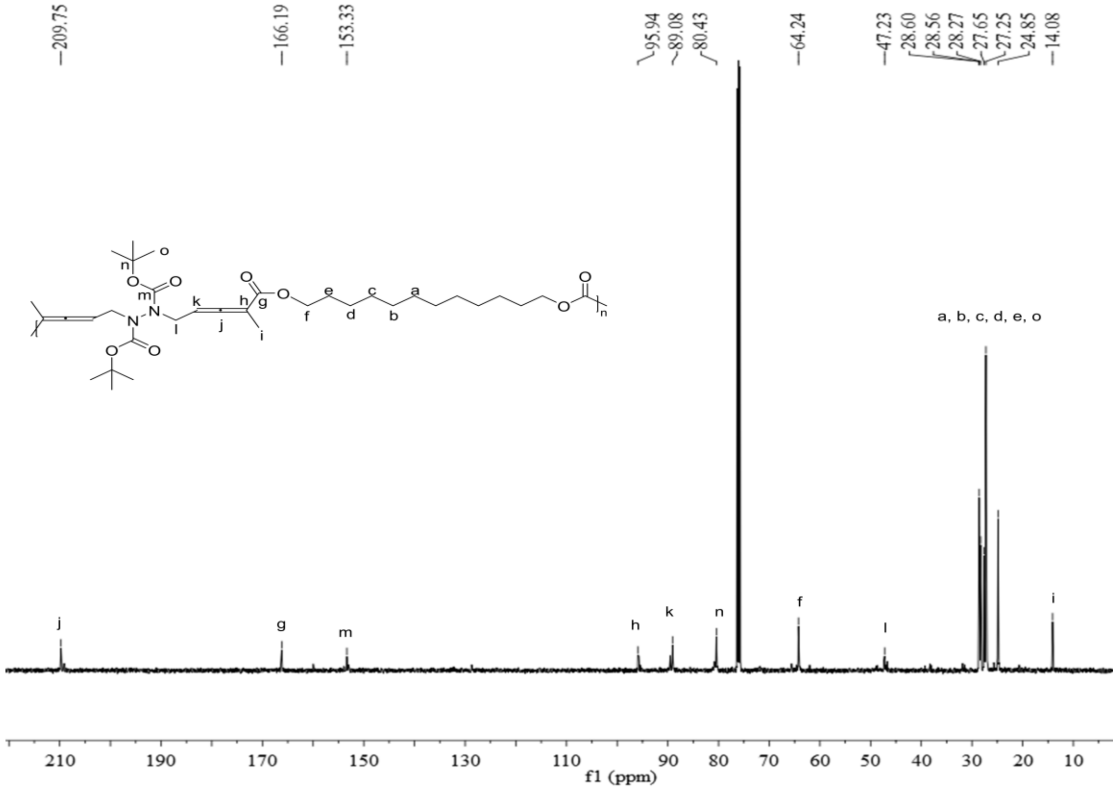

7.208 5.223 5.218 5.214 5.208 5.202 5.192 4.168 4.161 4.154 4.146 4.142 4.132 4.126 4.118 4.111 4.104 4.097 4.090 4.074 4.060 4.052 4.045 4.038 4.024 4.010 4.000 3.997 3.988 2.824 2.806 2.783 2.777 2.767 2.755 2.738 2.730 2.703 2.687 2.675 2.659 1.772 1.767 1.758 1.752 1.742 1.736 1.581 1.568 1.555 1.286 1.272 1.240 1.201 1.187 1.181 1.173 1.167
a+b+c+d+p
i+l
o+f
e
k
e
1.48
9.11
4.00
4.92
4.73
26.69
f1 (ppm)


211.36
169.80 167.33
95.78
87.93
77.25 77.00 76.75
65.15 61.53 57.39
30.77 29.56 29.52 29.25 28.59 25.85 15.00 14.02
a+b+c+d+e+l
p
i
o
f
k
m
h
j
n
g
f1 (ppm)

P11

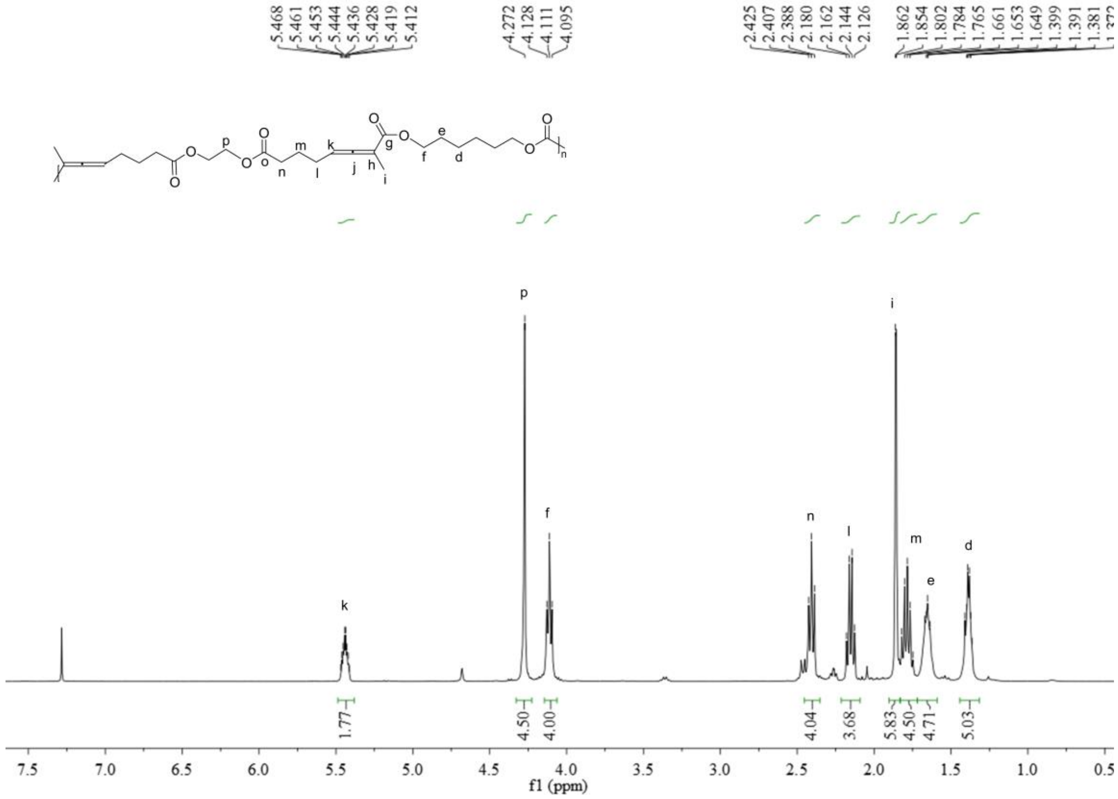


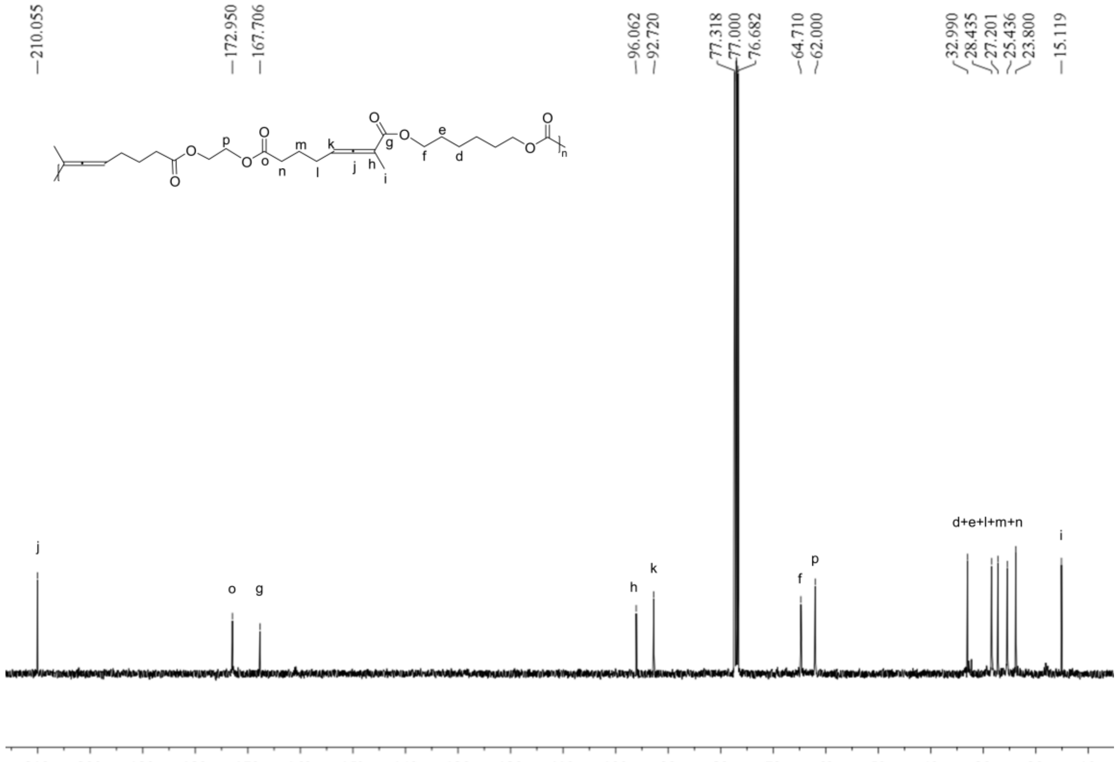
5.603
5.596
5.587
5.573
5.567
5.552
5.544
5.527
5.521
5.512
5.506
5.501
5.497
5.491
5.482
5.476
5.468
5.462
4.175
4.136
4.128
4.123
4.113
4.097
4.086
4.084
4.067
4.051
4.033
4.020
4.007
3.997
3.992
3.985
3.979
3.963
1.806
1.799
1.794
1.555
1.420
1.401
1.396
1.379
1.224
1.190
o
a+b+c+d
i
e
f+l
k
1.36
8.00
4.42
4.31
19.17
18.69
f1 (ppm)



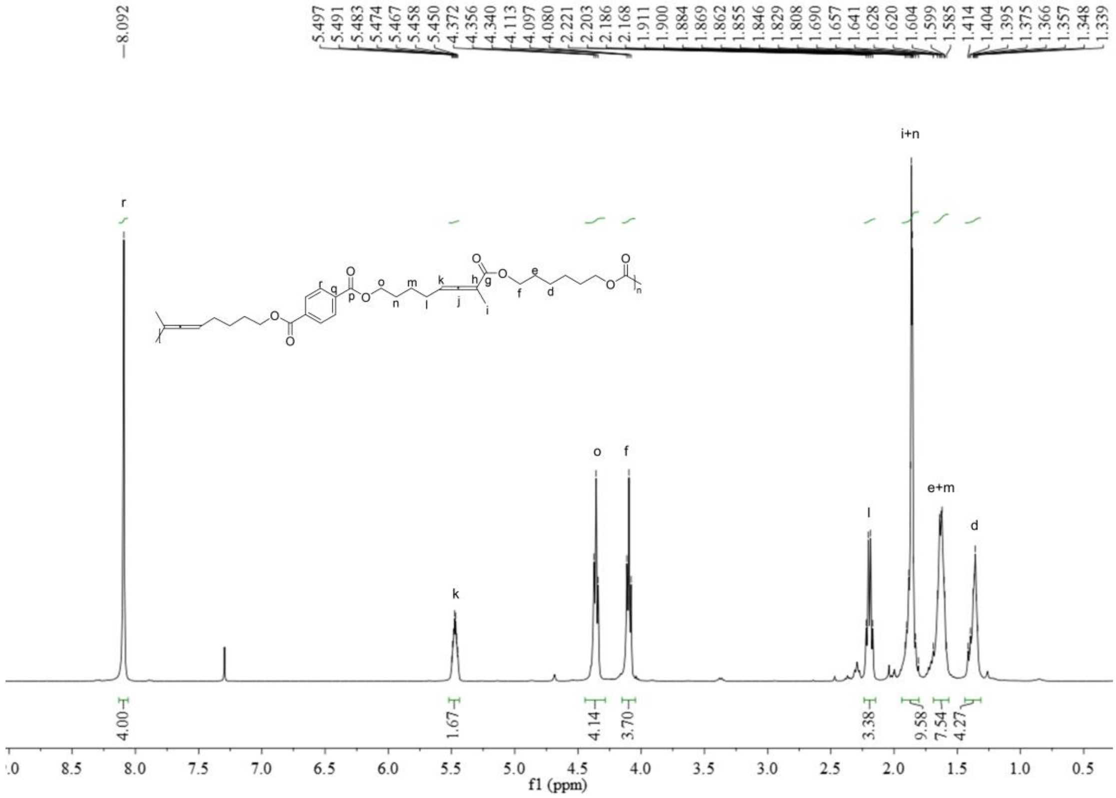
209.75
166.19
153.33
95.94
89.08
80.43
64.24
47.23
28.60
28.56
28.27
27.65
27.25
24.85
14.08
a, b, c, d, e, o
j
g
m
h
k
n
f
l
i
f1 (ppm)

P12

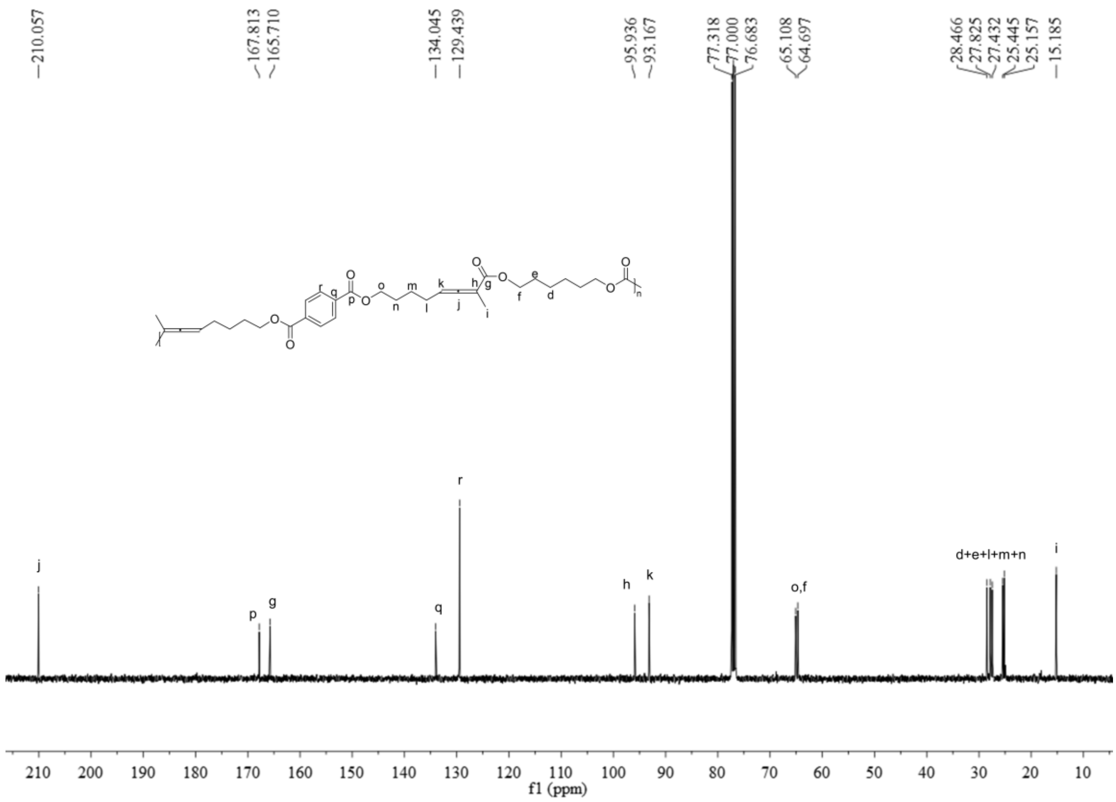

5.468
5.461
5.453
5.444
5.436
5.428
5.419
5.412
4.272
4.128
4.111
4.095
2.425
2.407
2.388
2.180
2.162
2.144
2.126
1.862
1.854
1.802
1.784
1.765
1.661
1.653
1.649
1.399
1.391
1.381
p
f
n
l
i
m
e
d
k
1.77
4.50
4.00
4.04
3.68
5.83
4.50
4.71
5.03
f1 (ppm)


210.055
172.950
167.706
96.062
92.720
77.318
77.000
76.682
64.710
62.000
32.990
28.435
27.201
25.436
23.800
15.119
j
o
g
h
k
f
p
d+e+l+m+n
i
f1 (ppm)

P13



8.092
5.497
5.491
5.483
5.474
5.467
5.458
5.450
4.372
4.356
4.340
4.113
4.097
4.080
2.221
2.203
2.186
2.168
1.911
1.900
1.884
1.869
1.862
1.855
1.846
1.829
1.808
1.690
1.657
1.641
1.628
1.620
1.604
1.599
1.585
1.414
1.404
1.395
1.375
1.366
1.357
1.348
1.339
r
k
o
f
l
i+n
e+m
d
4.00
1.67
4.14
3.70
3.38
9.58
7.54
4.27
f1 (ppm)



210.057
167.813
165.710
134.045
129.439
95.936
93.167
77.318
77.000
76.683
65.108
64.697
28.466
27.825
27.432
25.445
25.157
15.185
j
p
g
q
r
h
k
o,f
d+e+l+m+n
i
f1 (ppm)