**Supporting Information**

**to**

# Stereo- and Regioselective Cyclopolymerization of Chiral 1,7-Octadiynes

by

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## model compounds

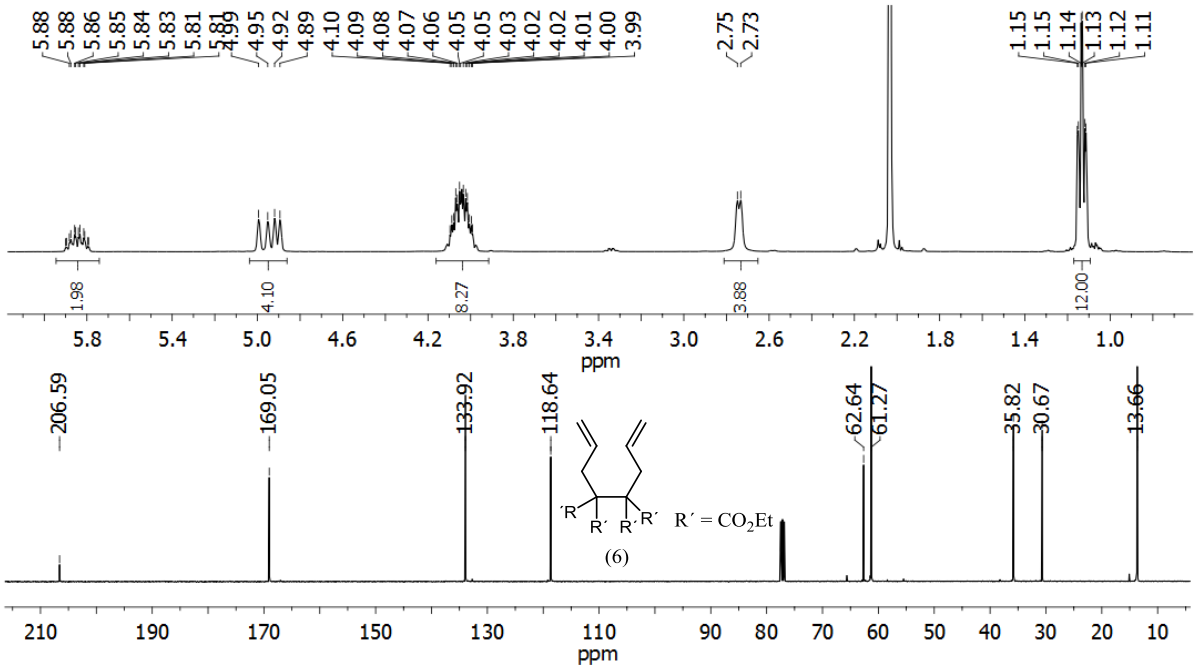



**Figure S1.** $^{1}H$ and $^{13}C$ NMR spectra of diethyl 1,7-octadien-4,4,5,5-tetracarboxylate (**6**), ($CDCl_3$).

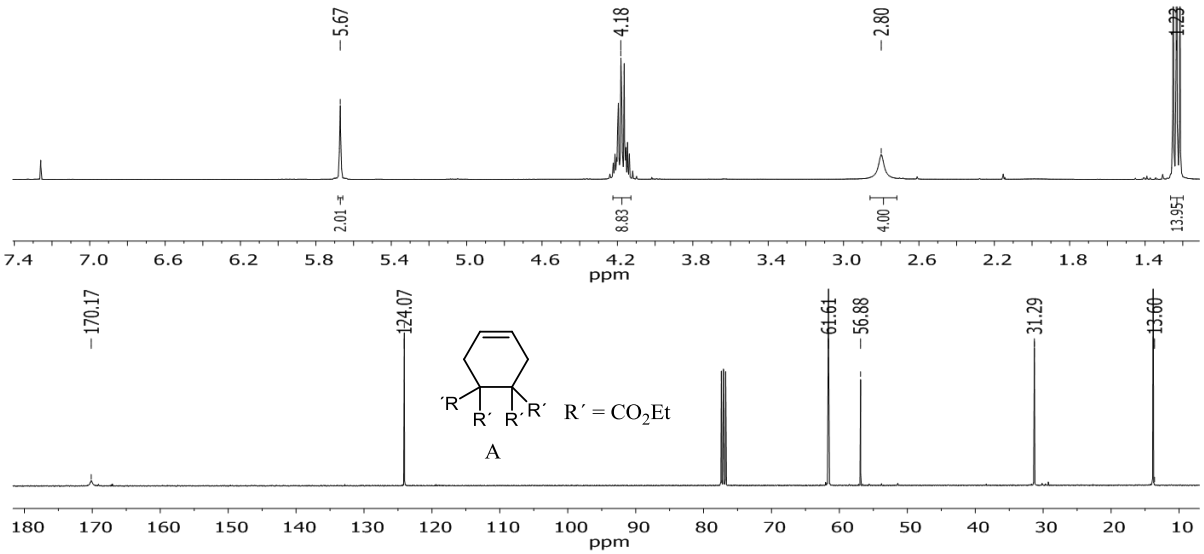



**Figure S2**. $^{1}H$ and $^{13}C$ NMR spectra of diethyl 1,2-cyclohexene-4,4,5,5-tetracarboxylate (**A**), ($CDCl_3$).

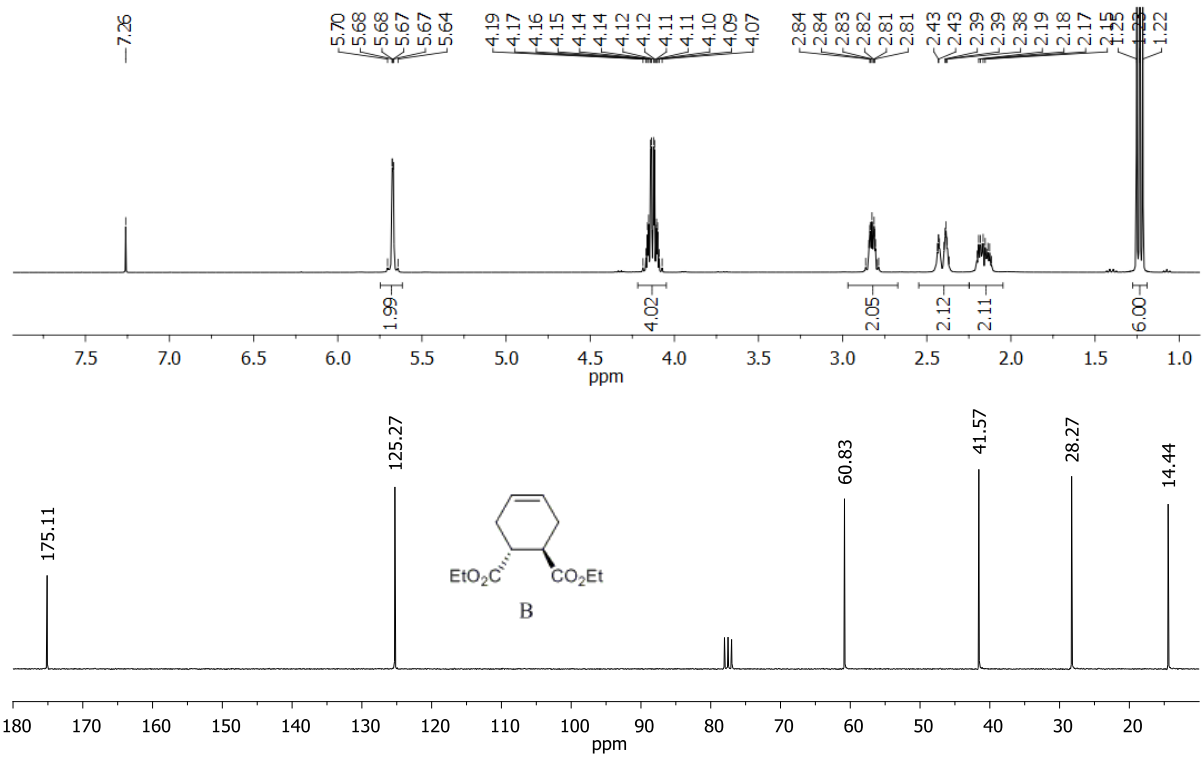



**Figure S3.** $^{1}H$ and $^{13}C$ NMR spectra of diethyl (*1R,2R*)-4-cyclohexen-1,2-dicarboxylate (**B**), ($CDCl_3$).

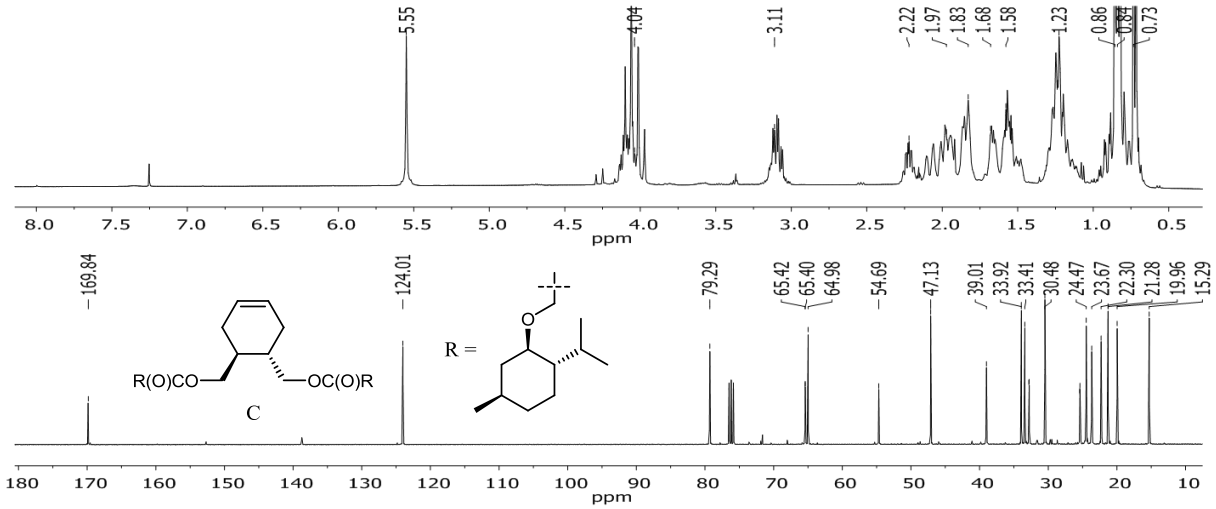
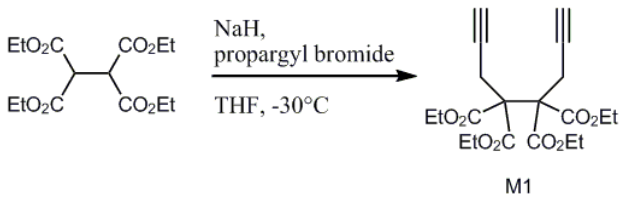



**Figure S4.** $^{1}H$ and $^{13}C$ NMR spectra of (*1R,2R*)-cyclohex-4-ene-1,2-dimethyl dimenthylate (**C**), ($CDCl_3$).

## Synthesis and NMR-spectra of monomers and polymers




**Scheme S1.** Synthesis of tetraethyl-1,7-octadiyne-4,4,5,5-tetracarboxylate (**M1**).

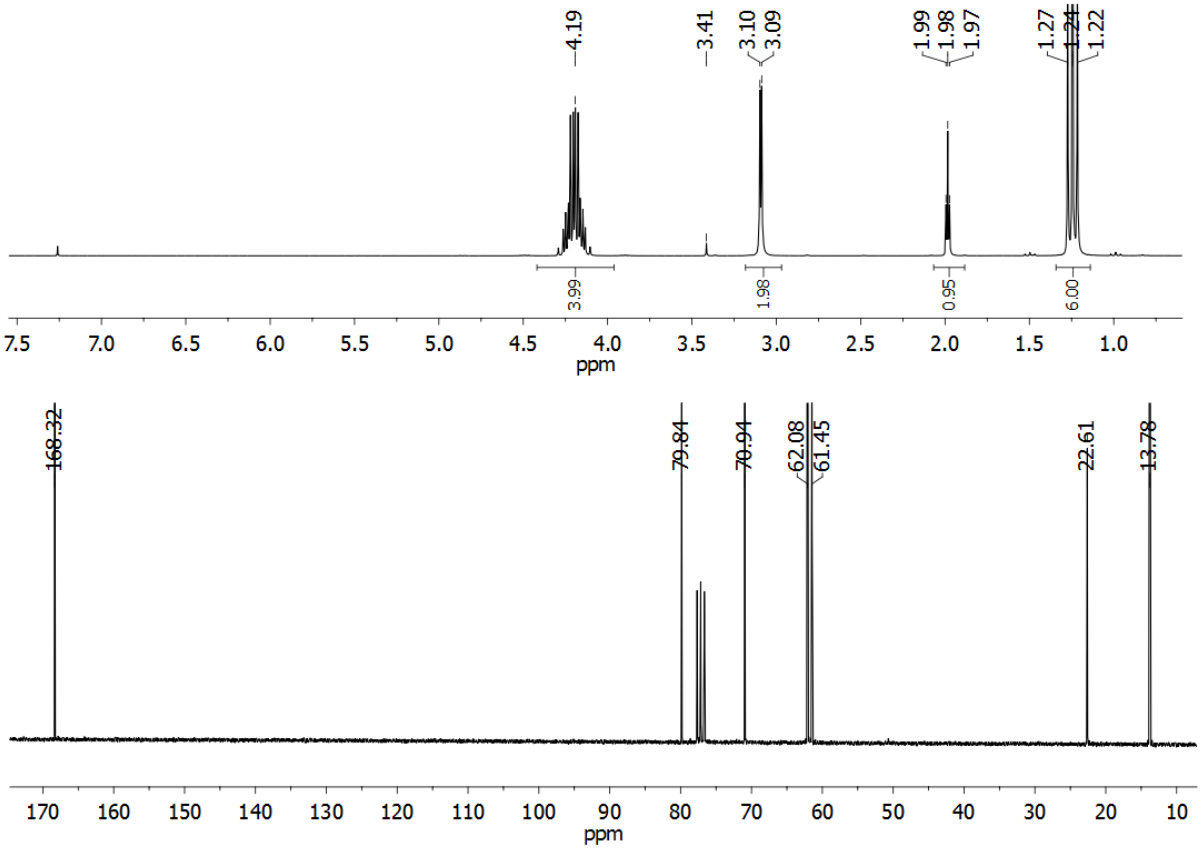



**Figure S5.** $^{1}H$ and $^{13}C$ NMR spectra of monomer **M1**, ($CDCl_3$).

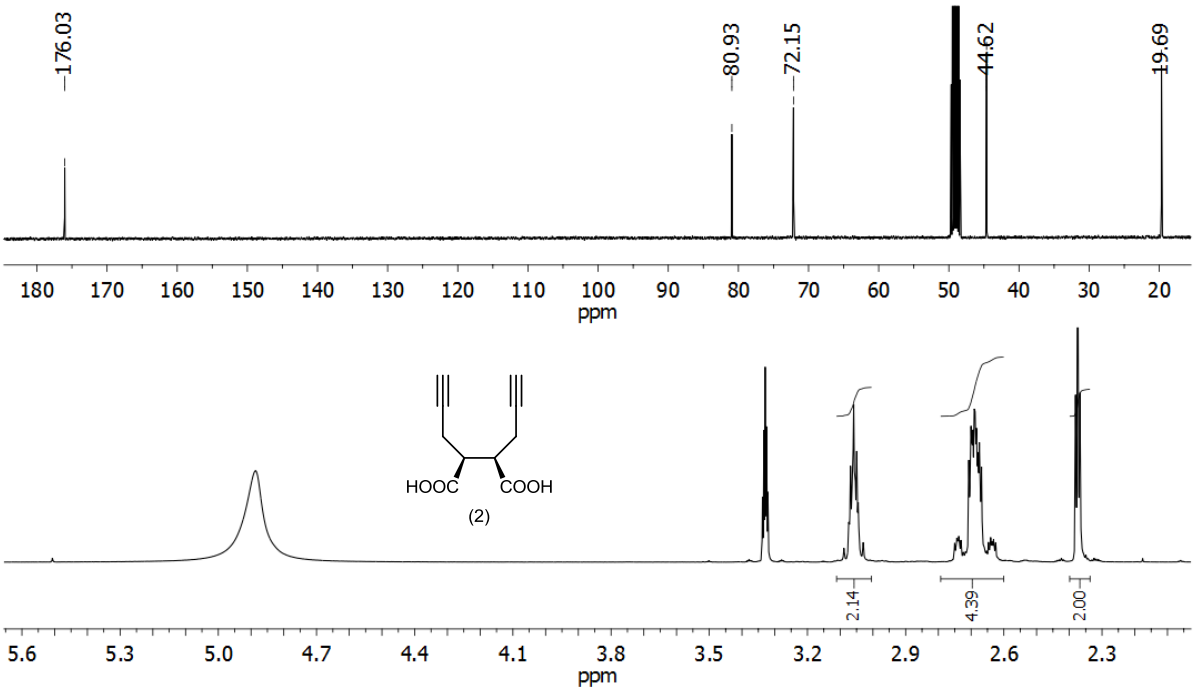



**Figure S6.** $^{1}$H and $^{13}$C NMR spectra of *meso*-1,7-octadiyne-4,5-dicarboxylic acid (***meso*-2**), (MeOD).

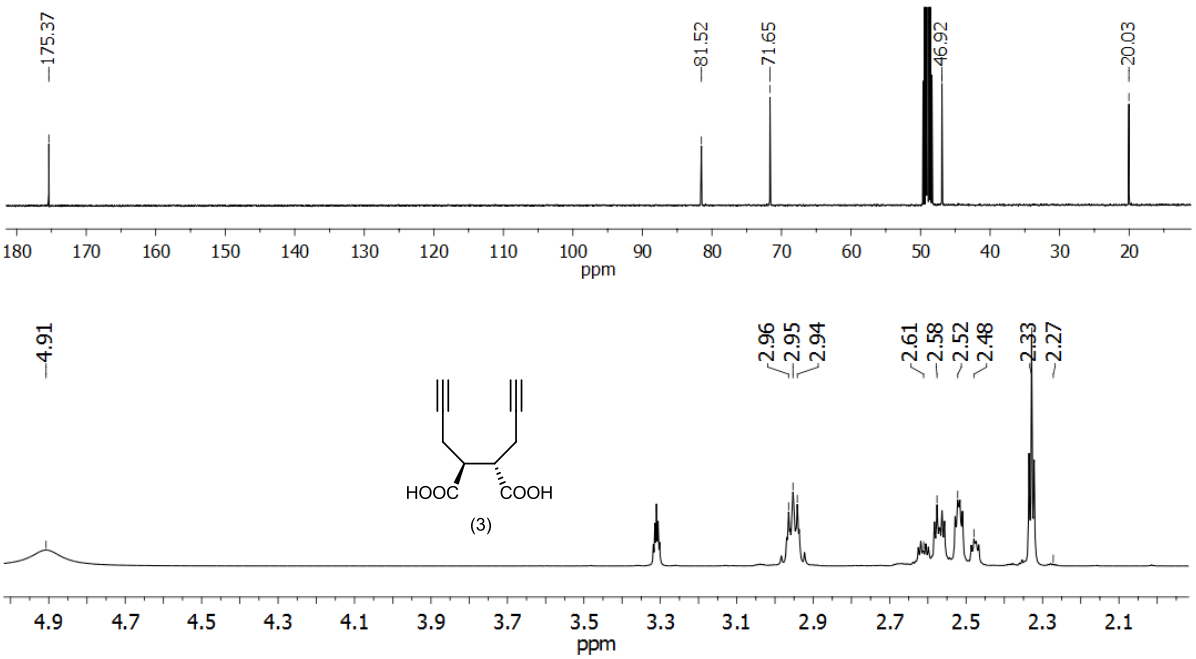



**Figure S7.** $^{1}$H and $^{13}$C NMR spectra of (*R*,*R*),(*S*,*S*)- 1,7-octadiyne-4,5-dicarboxylic acid (***rac*-3**), (MeOD). Structure ***rac*-(3)** represents one of the possible stereoisomers.

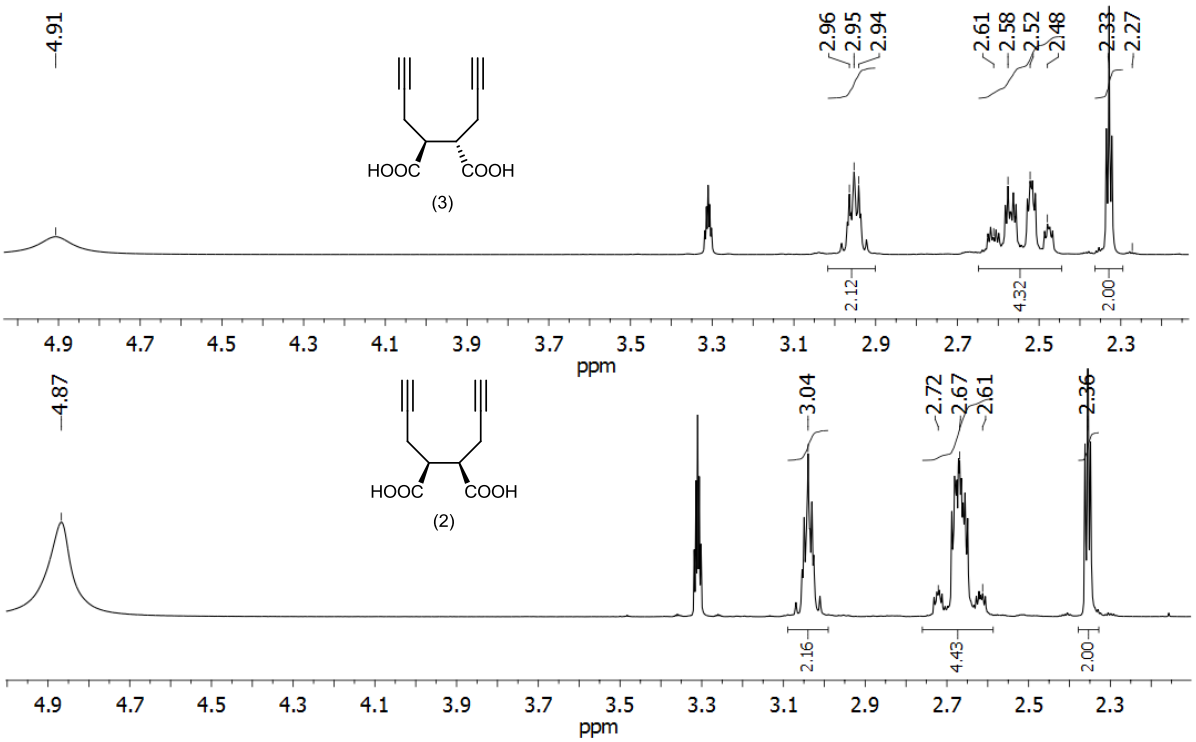



**Figure S8.** $^{1}$H NMR-spectra of ***rac*-3** (top) und ***meso*-2** (bottom), (MeOD). Structure *of* ***rac*-(3)** represents one of the possible stereoisomers.

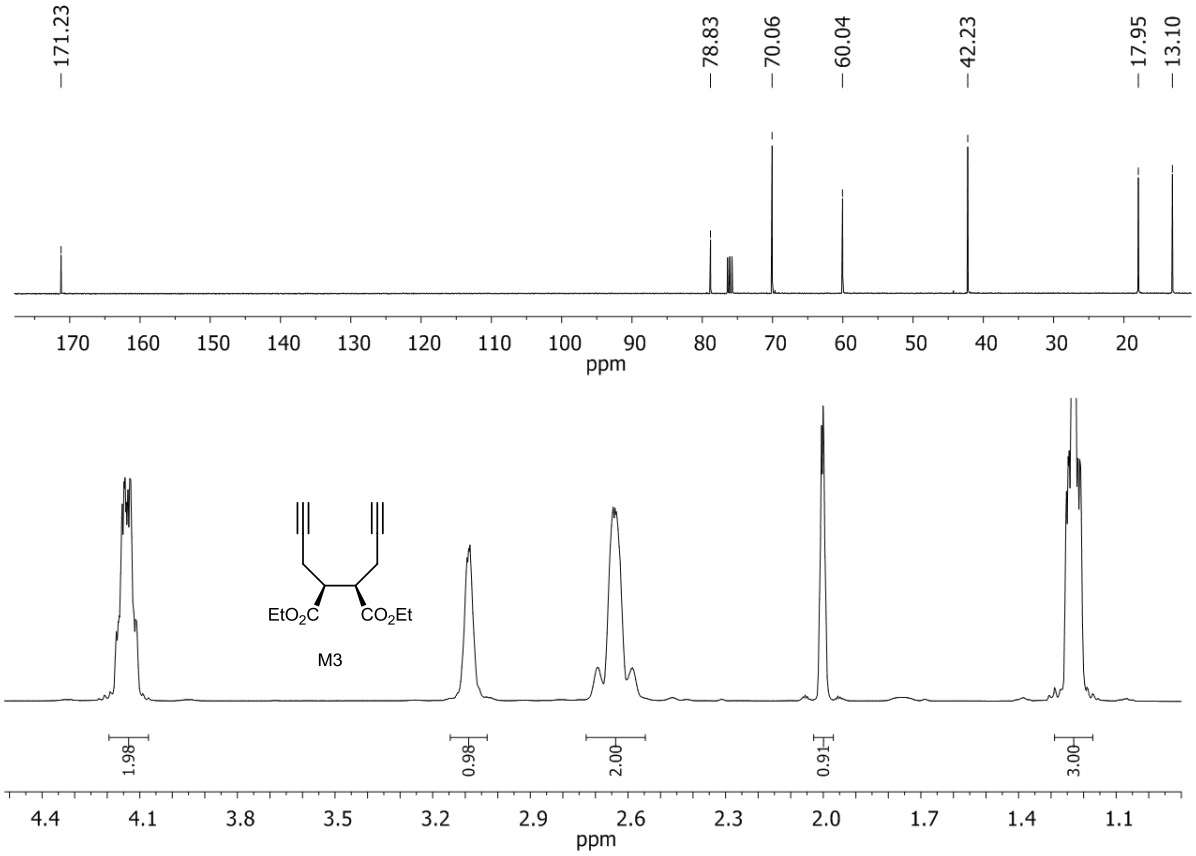



**Figure S9.** $^{1}$H and $^{13}$C NMR spectra of monomer diethyl *meso*-1,7-octadiyne-4,5-dicarboxylate (***meso*-M3**), ($CDCl_3$).

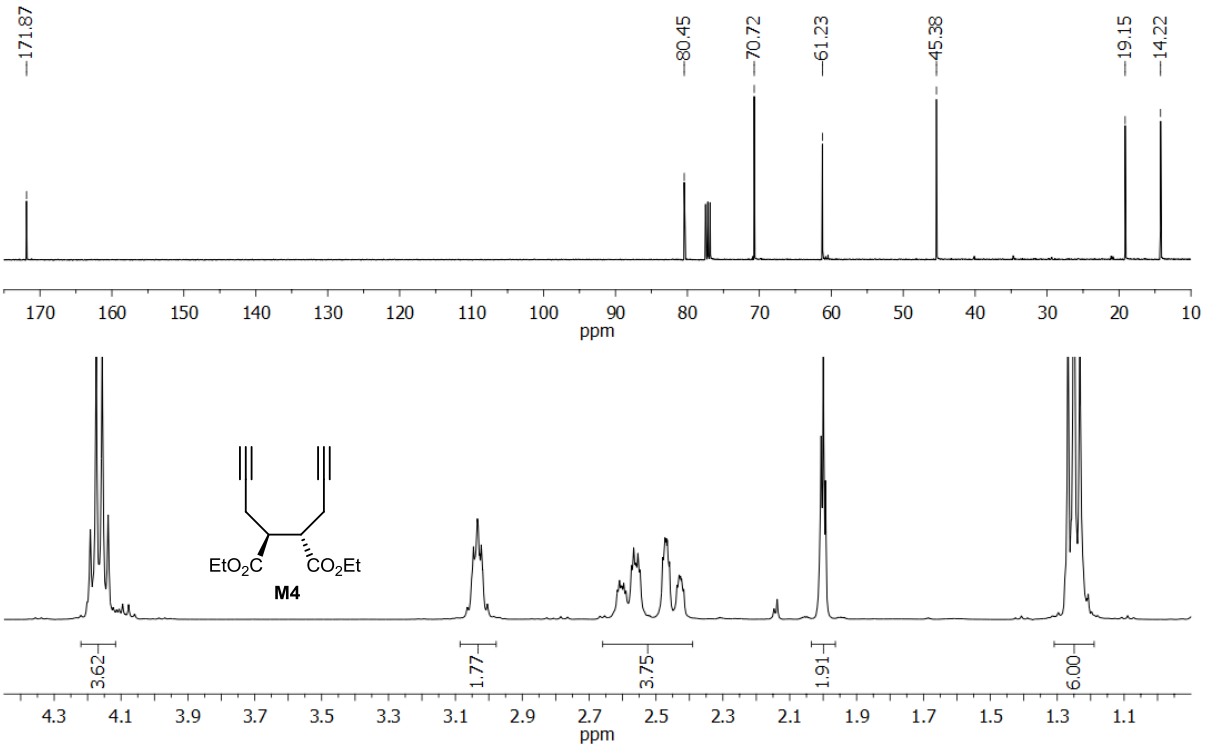



**Figure S10.** $^{1}$H and $^{13}$C NMR spectra of monomer diethyl (*R*,*R*),(*S*,*S*)-1,7-octadiyne-4,5-dicarboxylate (***rac*-M4**), ($CDCl_3$). Structure ***rac*-M4** represents one of the stereoisomers.

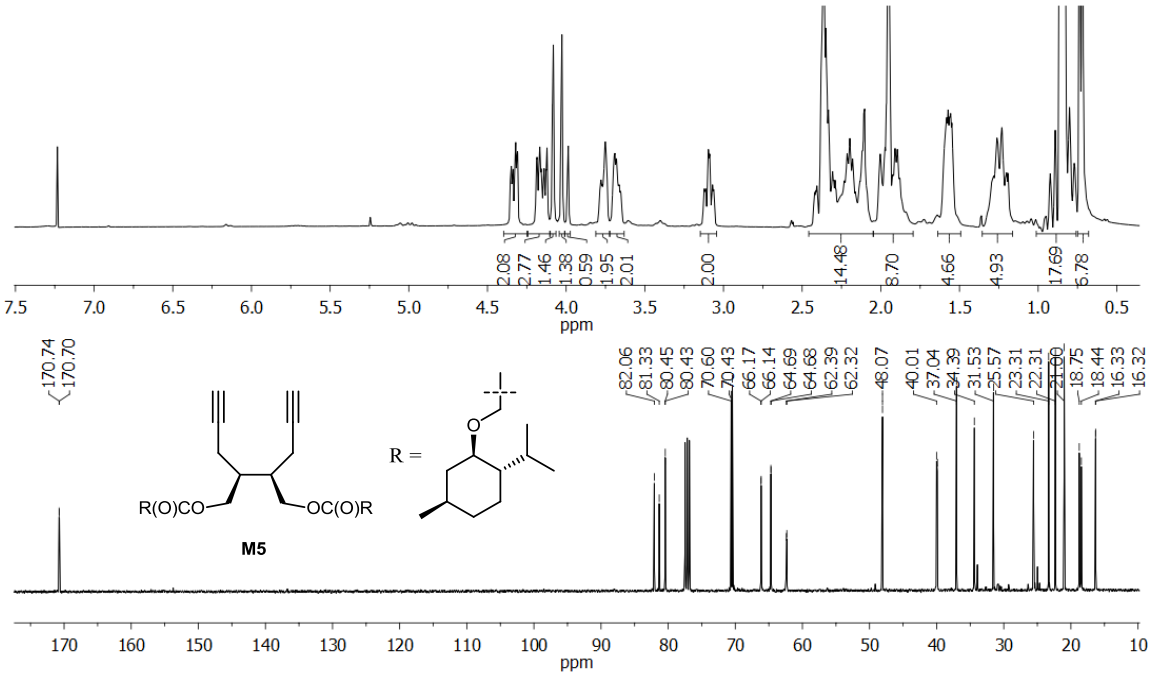



**Figure S11.** $^{1}$H and $^{13}$C NMR spectra of monomer *meso*-1,7-octadiyne-4,5-dimethyl dimenthylate (***meso*-M5**), ($CDCl_3$). Structure ***meso*-M5** represents one of the stereoisomers.

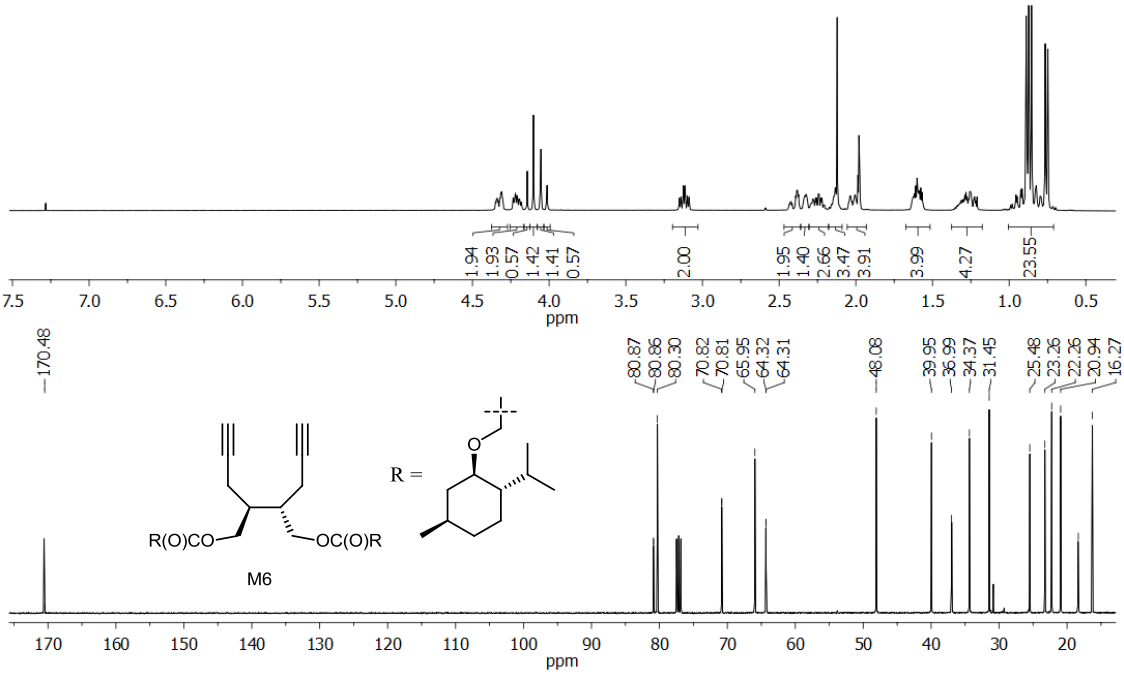



**Figure S12.** $^{1}$H and $^{13}$C NMR spectra of monomer (*R*,*R*),(*S*,*S*)-1,7-octadiyne-4,5-dimethyl dimenthylate (***rac*-M6**), ($CDCl_3$). Structure ***rac*-*M*6** represents one of the stereoisomers.

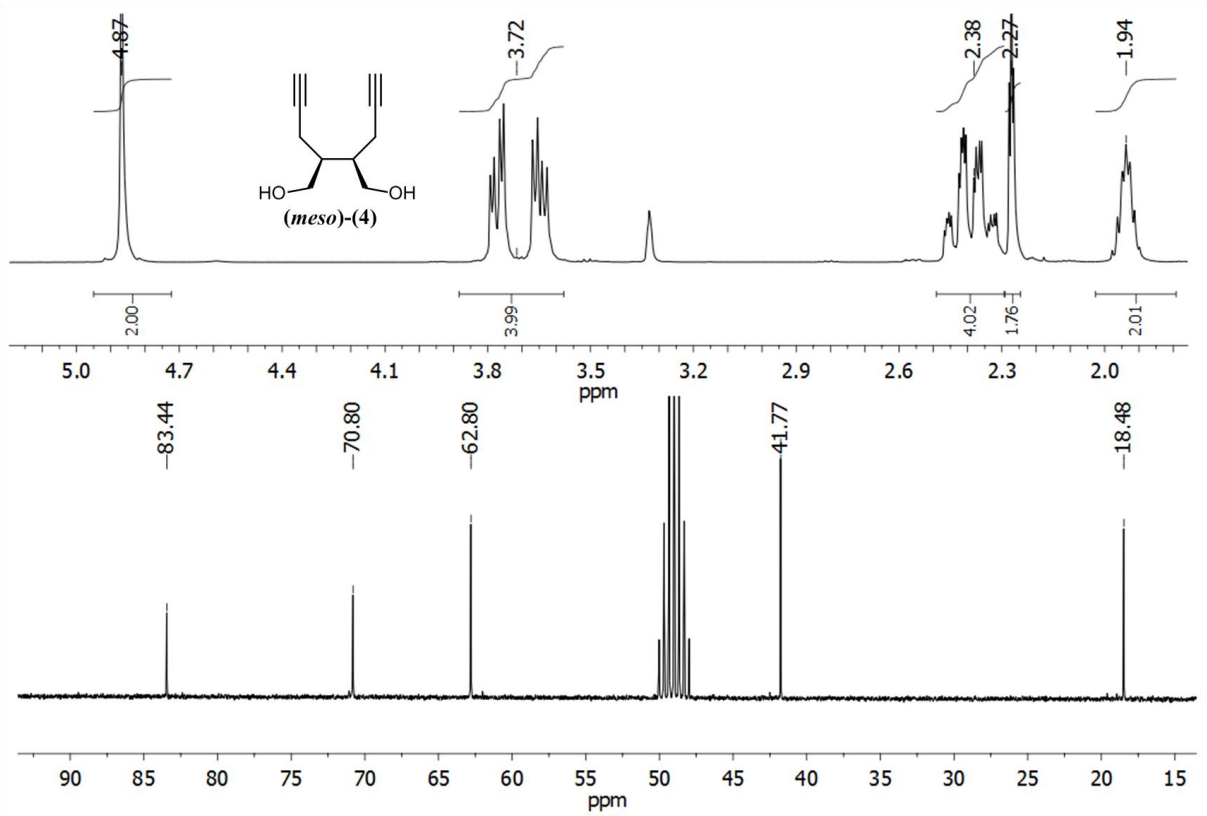



**Figure S13.** $^{1}$H and $^{13}$C NMR spectra of *meso*-1,7-octadiyne-4,5-dimethanol (***meso*-4**) ($CDCl_3$). Structure ***meso*-4** represents one of the stereoisomers.

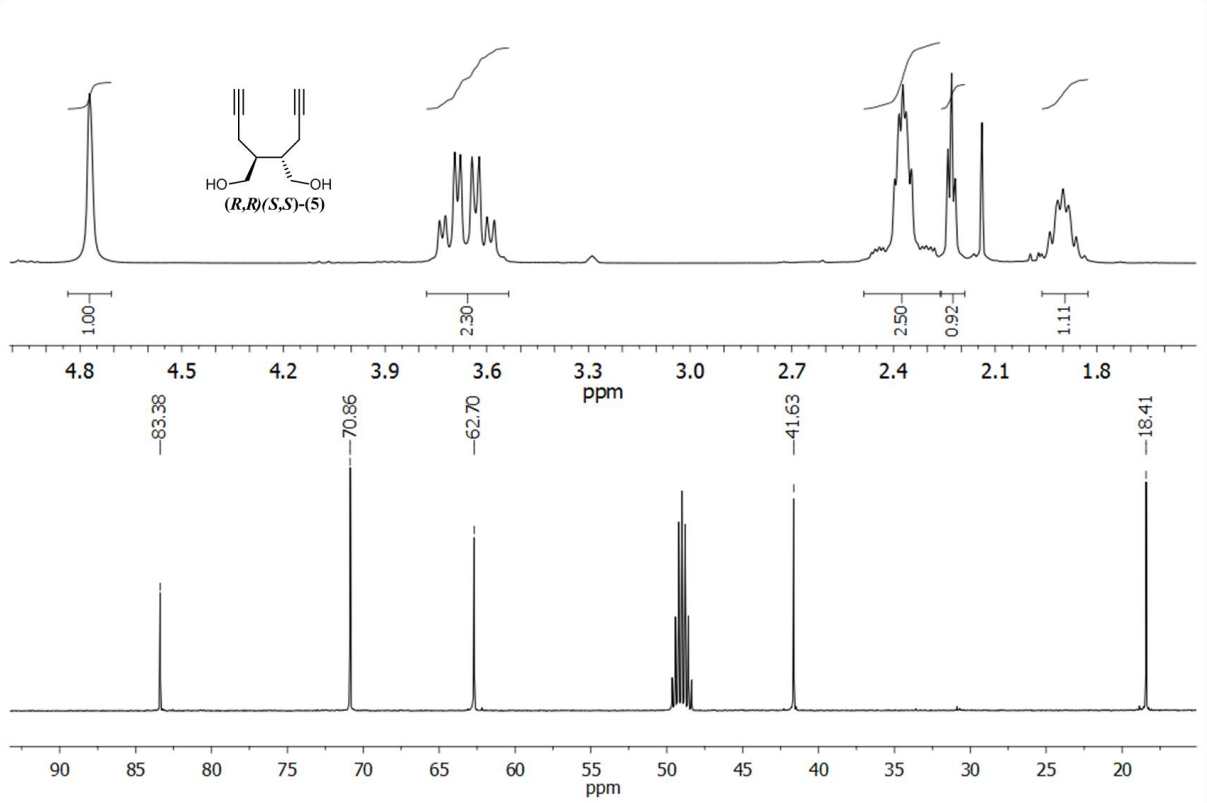



**Figure S14.** $^{1}$H and $^{13}$C NMR spectra of *(R,R),(S,S)*-1,7-octadiyne-4,5-dimethanol (***rac*-5**), ($CDCl_3$). Structure ***rac*-5** represents one of the stereoisomers.

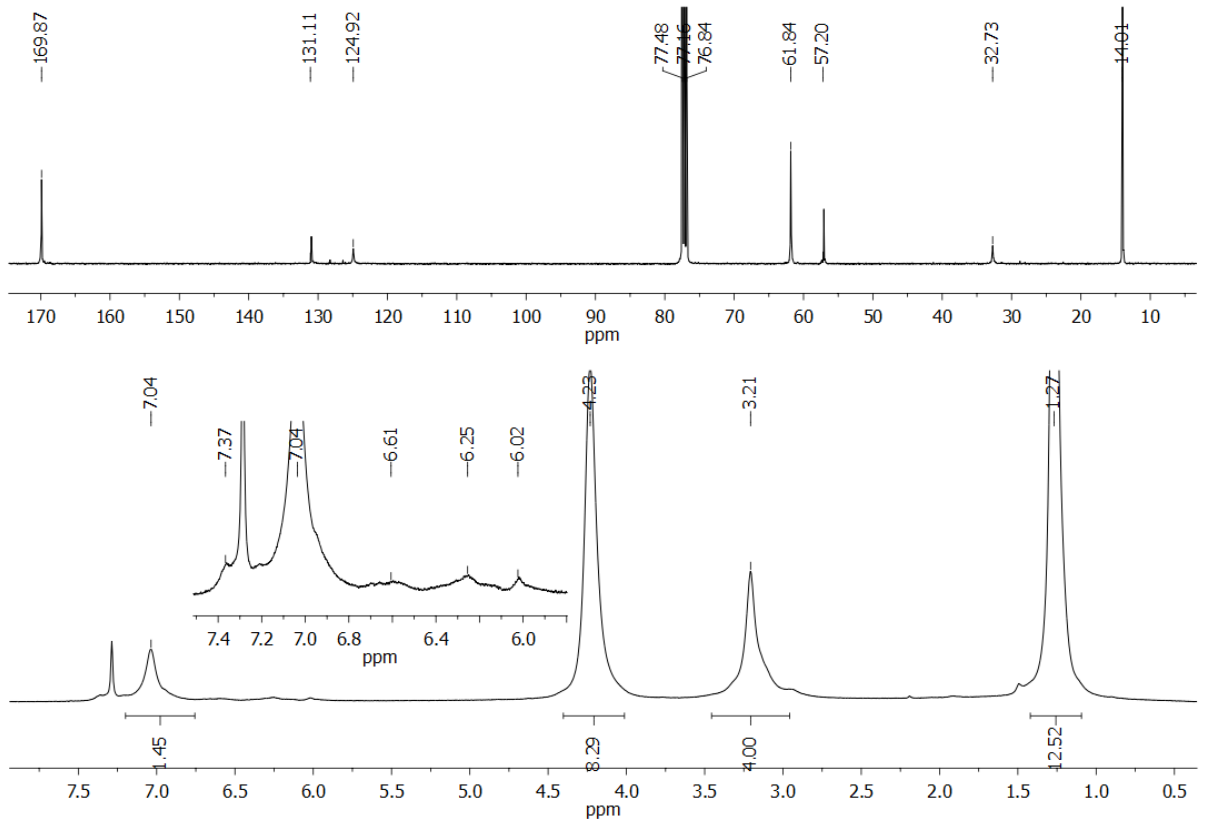



**Figure S15.** $^{1}$H and $^{13}$C NMR spectra of **poly-M1** prepared by the action of **I1**·**quinuclidine**, ($CDCl_3$).

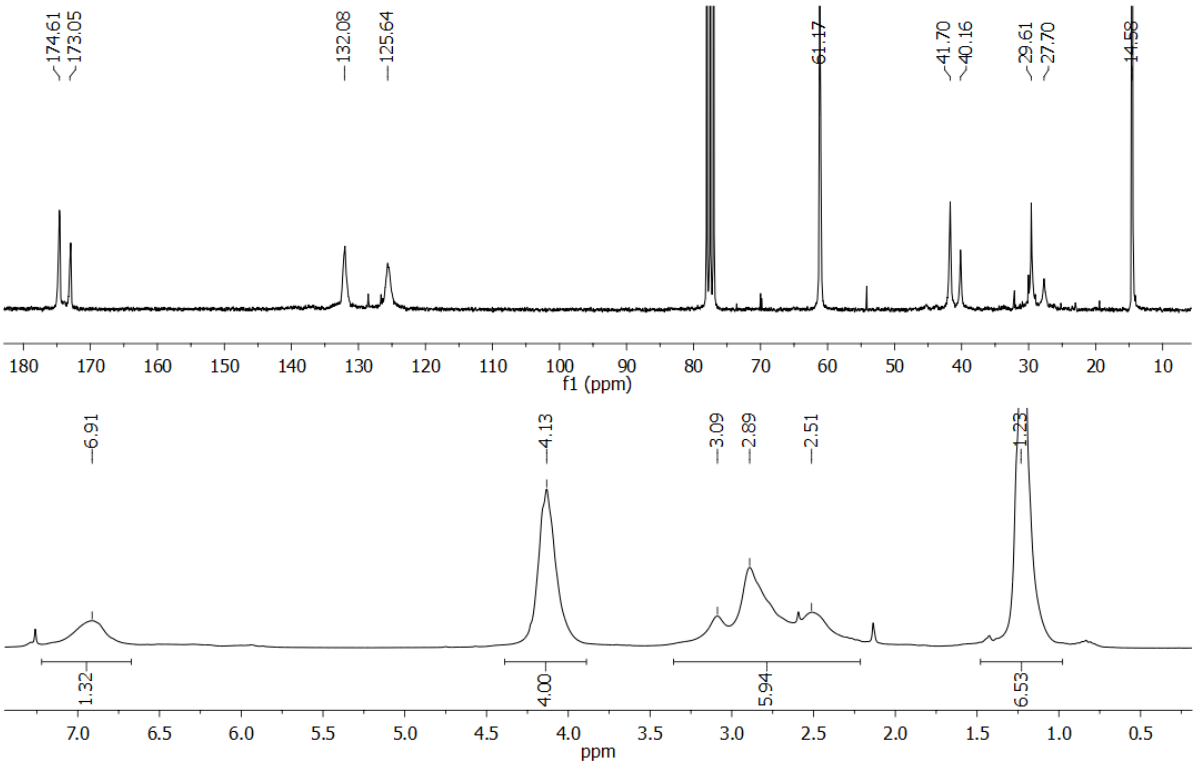



**Figure S16.** $^{1}$H and $^{13}$C NMR spectra of **poly-M2** prepared by the action of **I1**·**quinuclidine**, ($CDCl_3$).

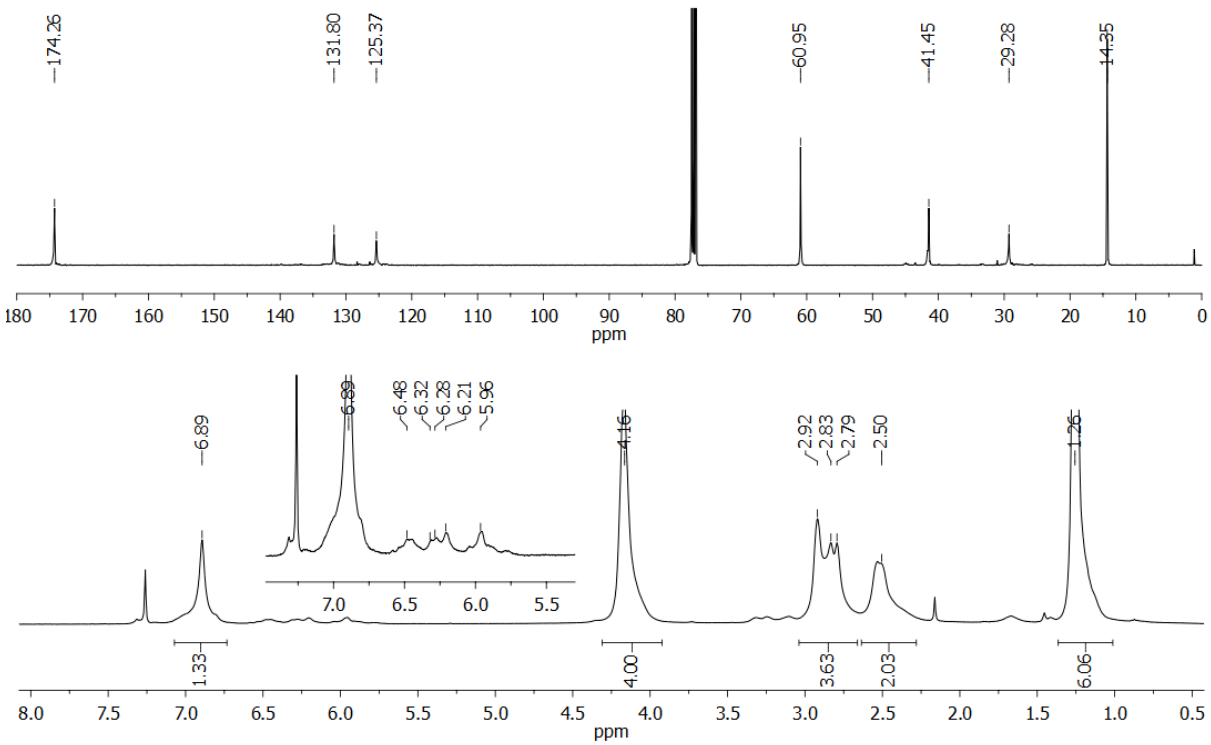



**Figure S17.** $^{1}$H- and $^{13}$C-NMR spectra of **poly-(meso-M3)** prepared by the action of **I1**·**quinuclidine**, ($CDCl_3$).

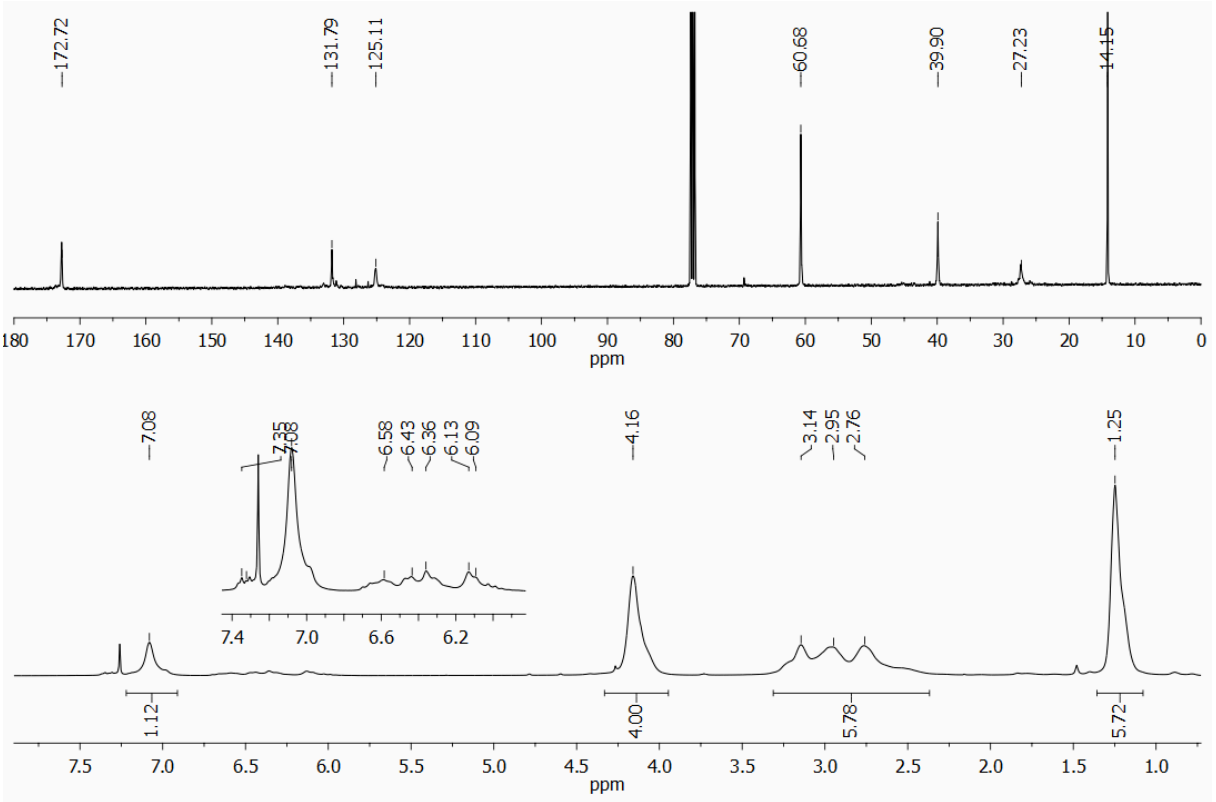



**Figure S18.** $^{1}H$ and $^{13}C$ NMR spectra of **poly-(*rac*-M4)** prepared by the action of **I1**·**quinuclidine**, ($CDCl_3$).

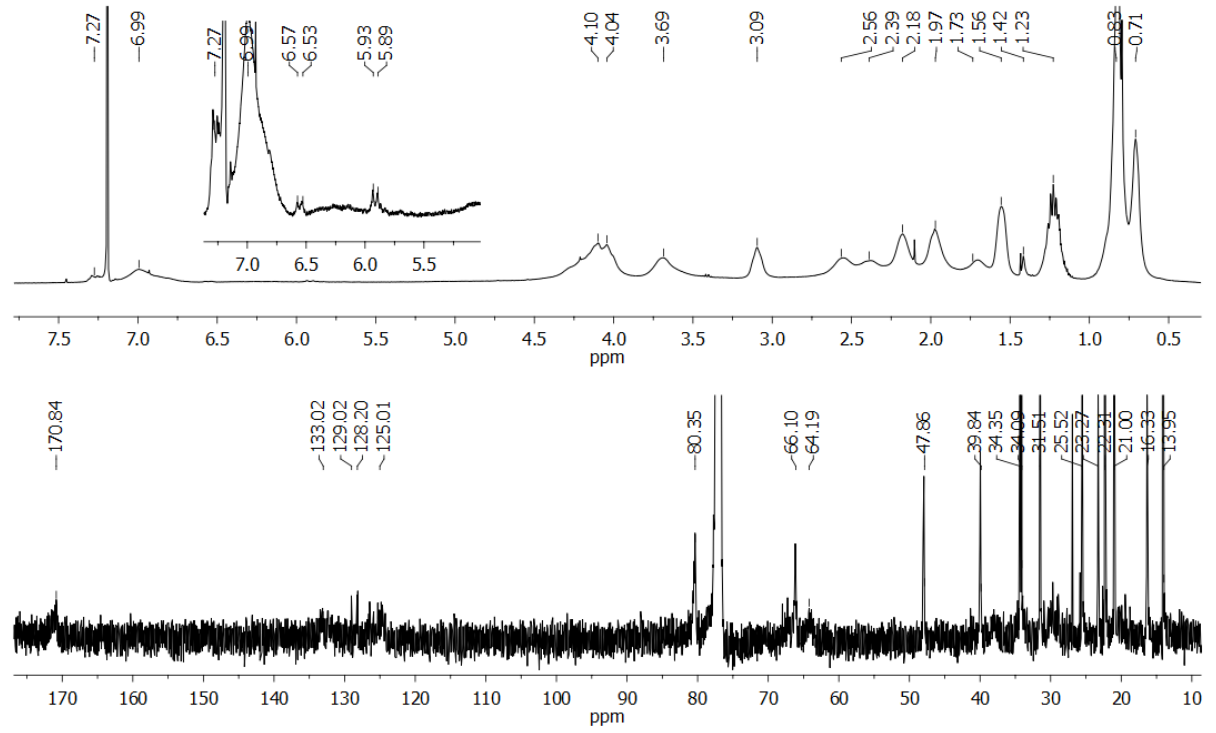



**Figure S19.** $^{1}H$ and $^{13}C$ NMR spectra of **poly-(*meso*-M5)** prepared by the action of **I1**·**quinuclidine**, ($CDCl_3$).

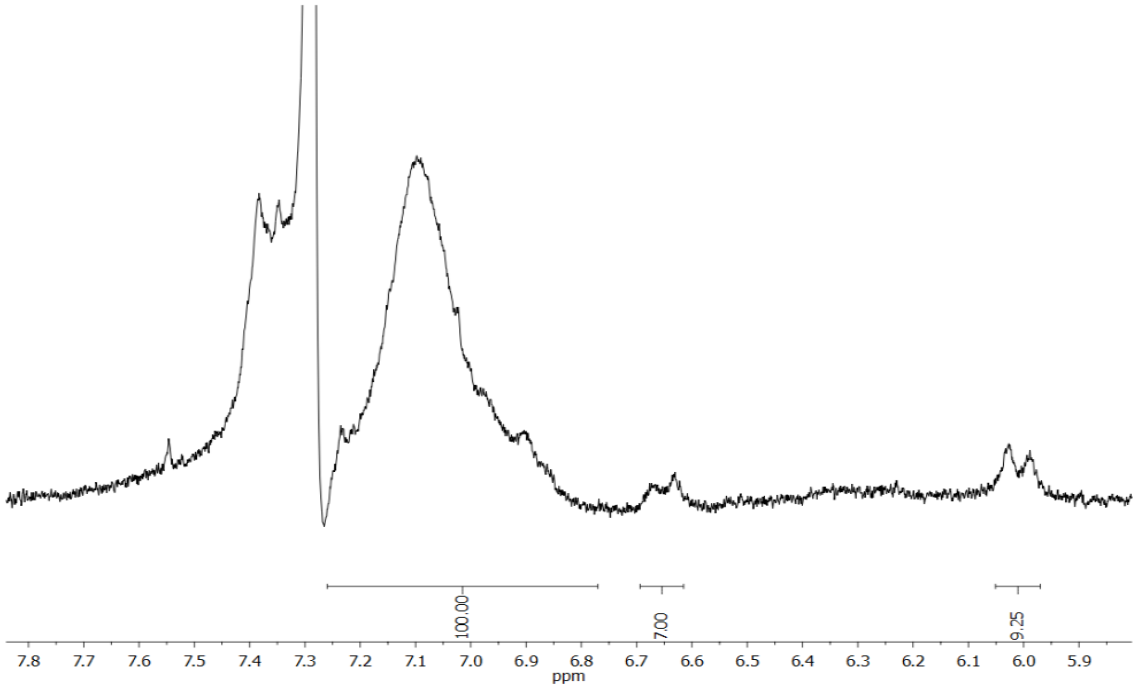



**Figure S20.** Part of the $^{1}$H NMR spectrum of **poly-(*meso*-M5)** prepared by the action of **I1·quinuclidine**, ($CDCl_3$).

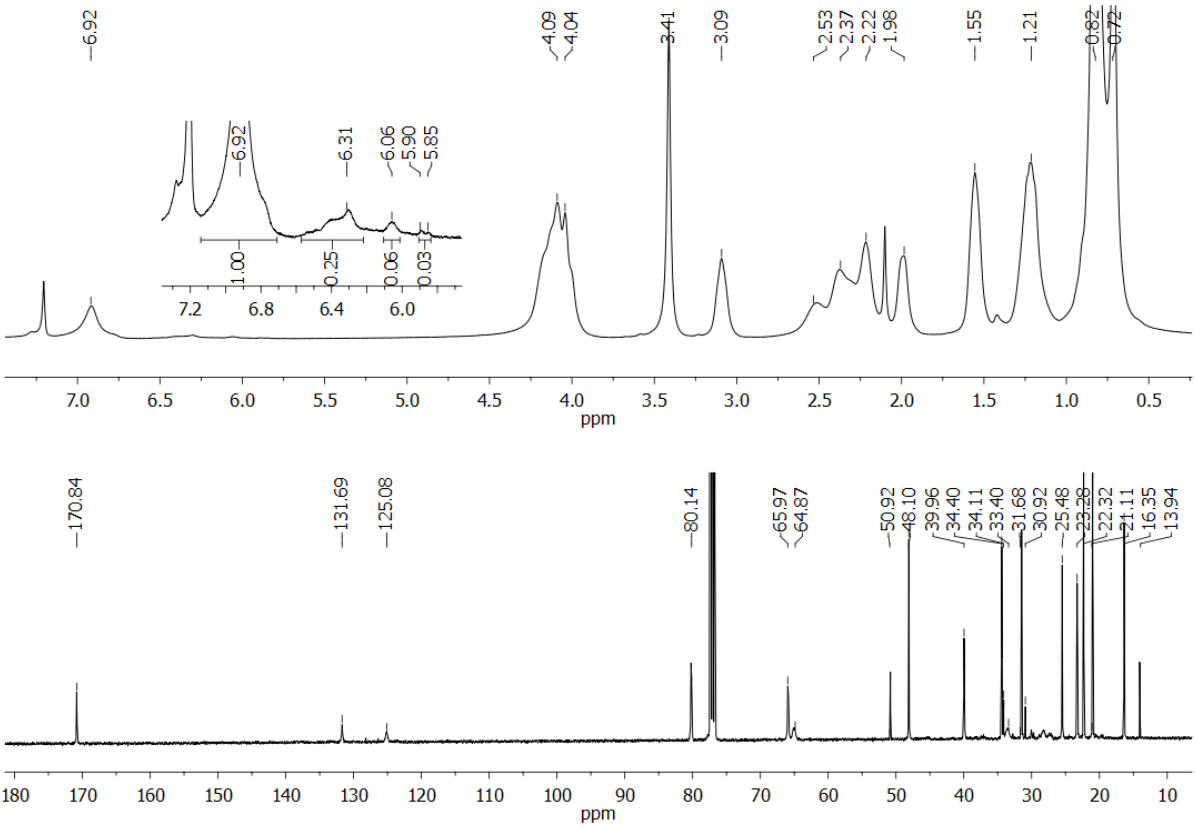



**Figure S21.** $^{1}$H and $^{13}$C NMR spectra of **poly-(*rac*-M6)** prepared by the action of **I1·quinuclidine**, ($CDCl_3$).

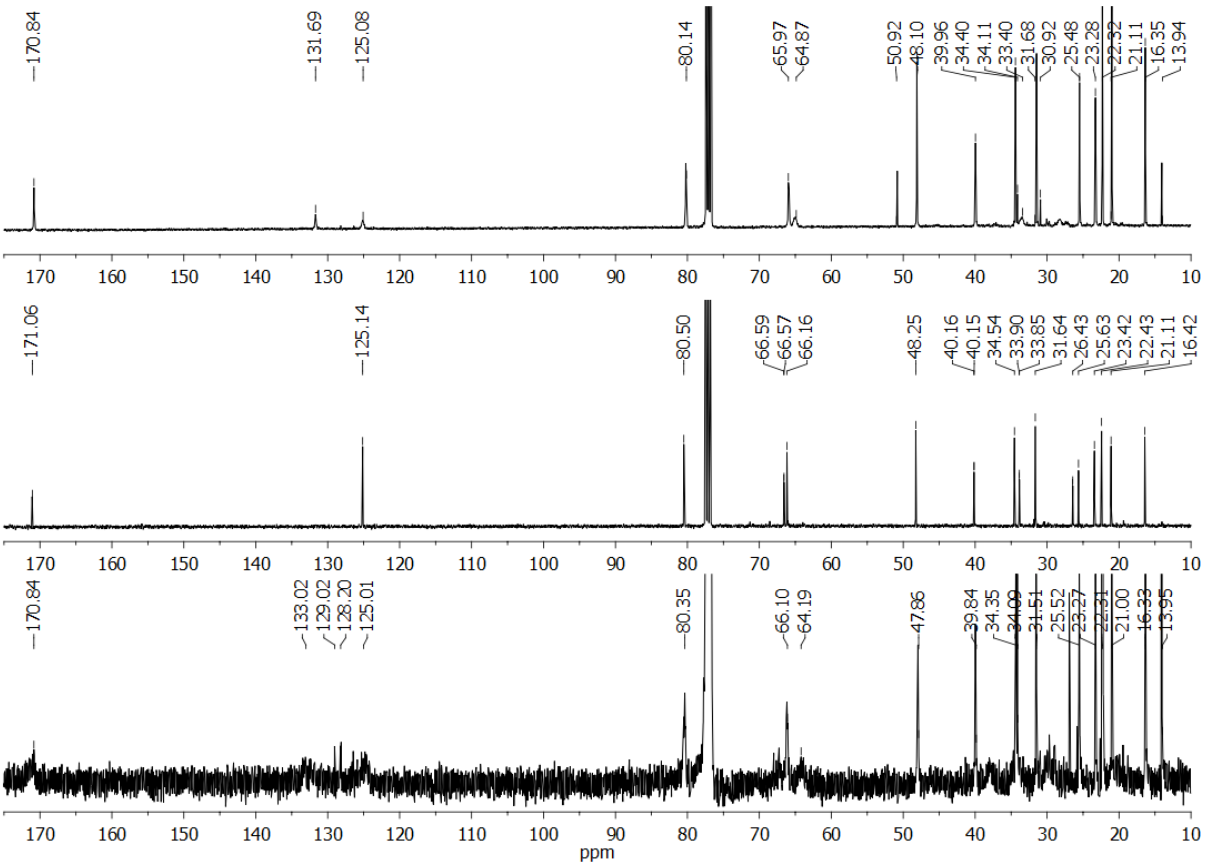



**Figure S22.** $^{13}C$ NMR spectra of model compound (**C**) (middle), **poly-(*meso*-M5)** (bottom) and **poly-(*rac*-M6)** (top) prepared by the action of **I1·quinuclidine**, ($CDCl_3$).

## GC-MS analysis of monomers M2, M3 and M4

The ratio of the diastereomers ***meso*-M3** and ***rac*-M4** was determined by GC-MS analysis using a chiral ß-dextrin column. This analysis also demonstrated the purity of each fraction (Figure 12). The diastereomeric mixture contained 40.5% of the *trans*-monomer ***rac*-M4** ($t_R$=10.5 min) and 59.5% of the *cis*-configured monomer ***meso*-M3** ($t_R$=10.7 min, Fig. 10). Chromatograms of ***meso*-M3** and ***rac*-M4** are shown in Figure16-18**.** Monomer ***meso*-M3** was contaminated with a small amount of ***rac*-M4** (<3%). ***rac*-M4** was pure.

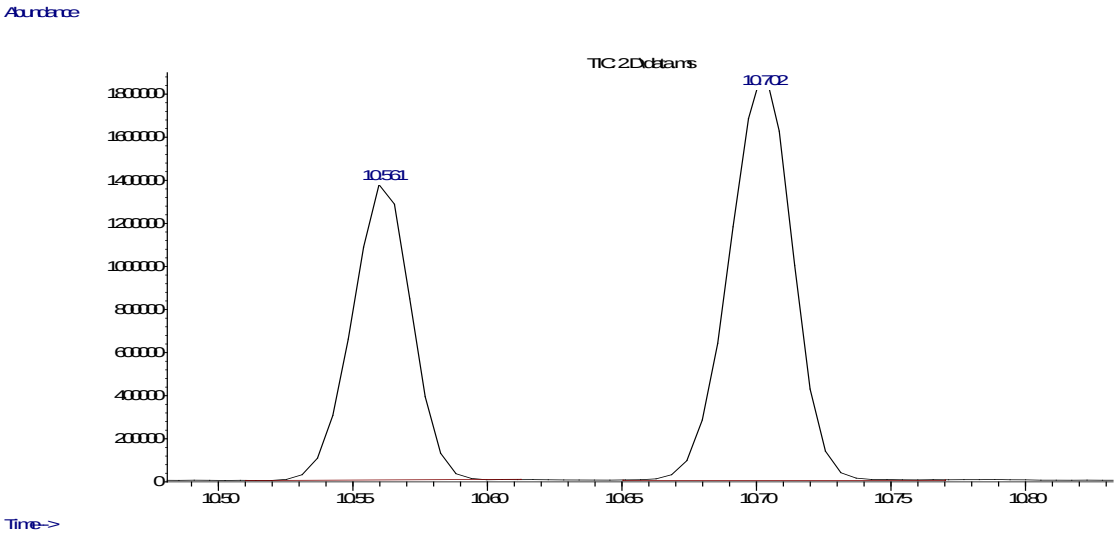



**Figure S23.** GC-MS of monomer **M2** and values of peak integration.

| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height | corr. area | corr. % max. | % of total |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 10.561 | 1297 | 1306 | 1315 | M | 1383734 | 21286748 | 68.22% | 40.554% |
| 2 | 10.702 | 1322 | 1331 | 1343 | M | 1927614 | 31202621 | 100.00% | 59.446% |

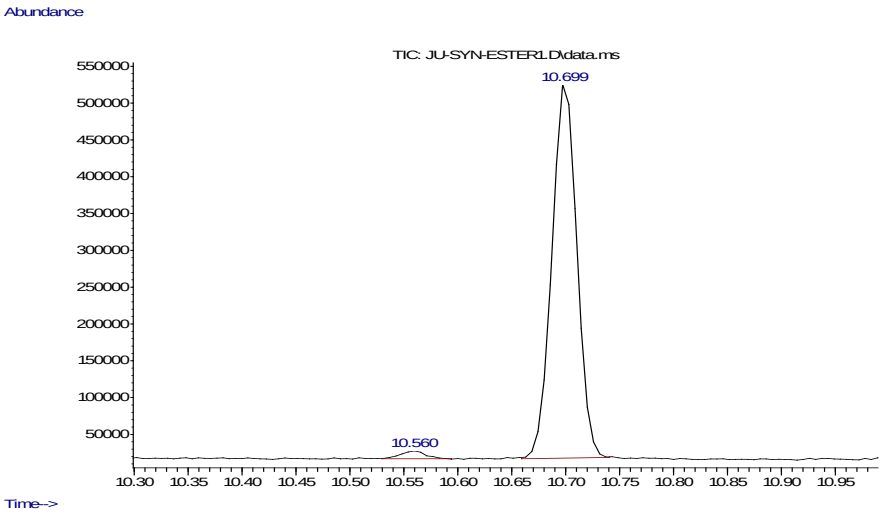



**Figure S24**. GC-MS of monomer ***meso*-M3** and values of peak integration.

| peak # | R.T. min | first scan | max scan | last scan | PK TY | peak height | corr. area | corr. % max. | % of total |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 10.560 | 1301 | 1306 | 1312 | M6 | 10694 | 171377 | 2.09% | 2.047% |
| 2 | 10.699 | 1323 | 1330 | 1338 | M | 513724 | 8199410 | 100.00% | 97.953% |

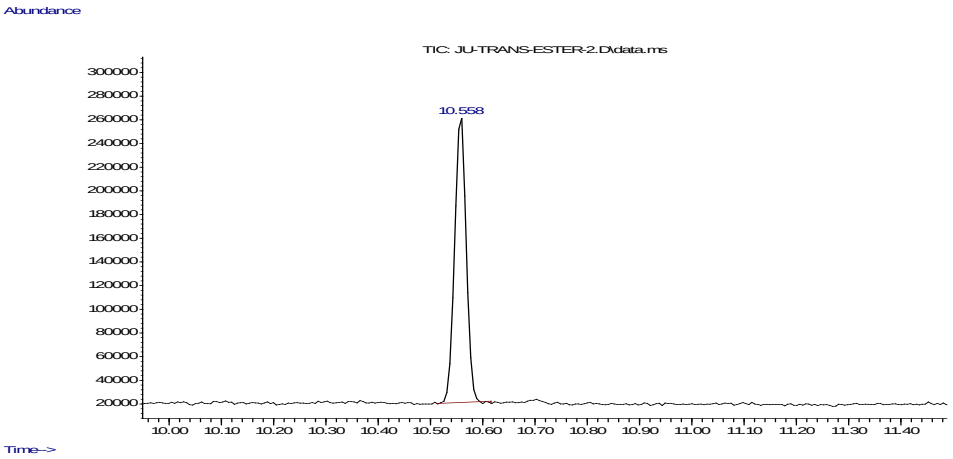



**Figure S25**. GC-MS of monomer ***rac*-M4**.

## DLS-measurements

Dynamic Light Scattering (DLS) was conducted on a Zetasizer Nano-ZS by Malvern Instruments GmbH with the model ZEN3600. A glass cuvette was used to analyze 1 mg/mL polymer solutions at 20°C in Chloroform. The equilibration time between runs was 3 min, and 3 runs were made with an average of 15 measurements each and a 1 s delay between each measurement.

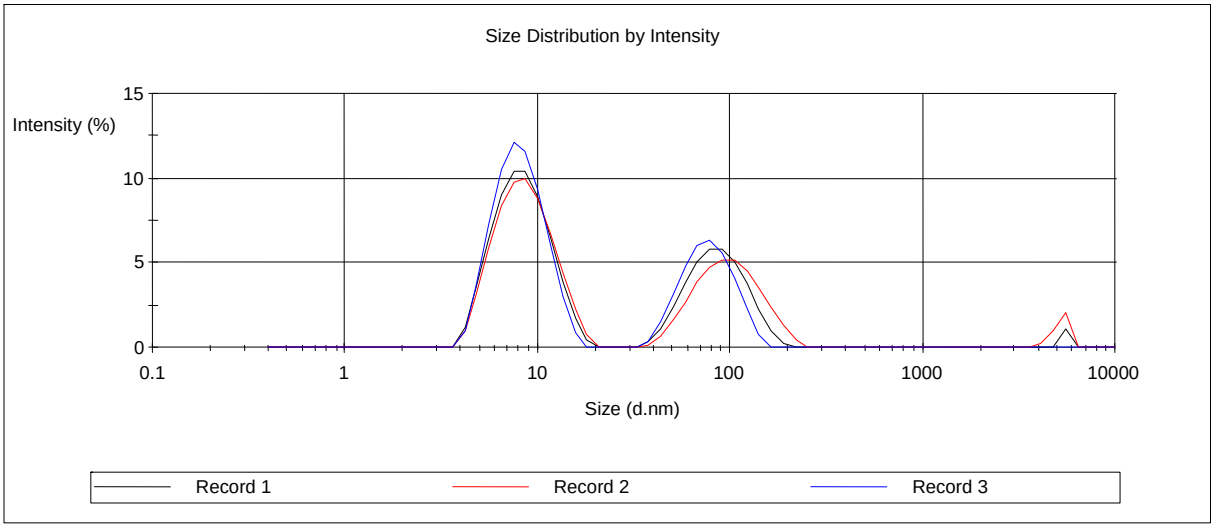



**Figure S26**. DLS spectra of monomer **poly-M1-I1·quinuclidine**.

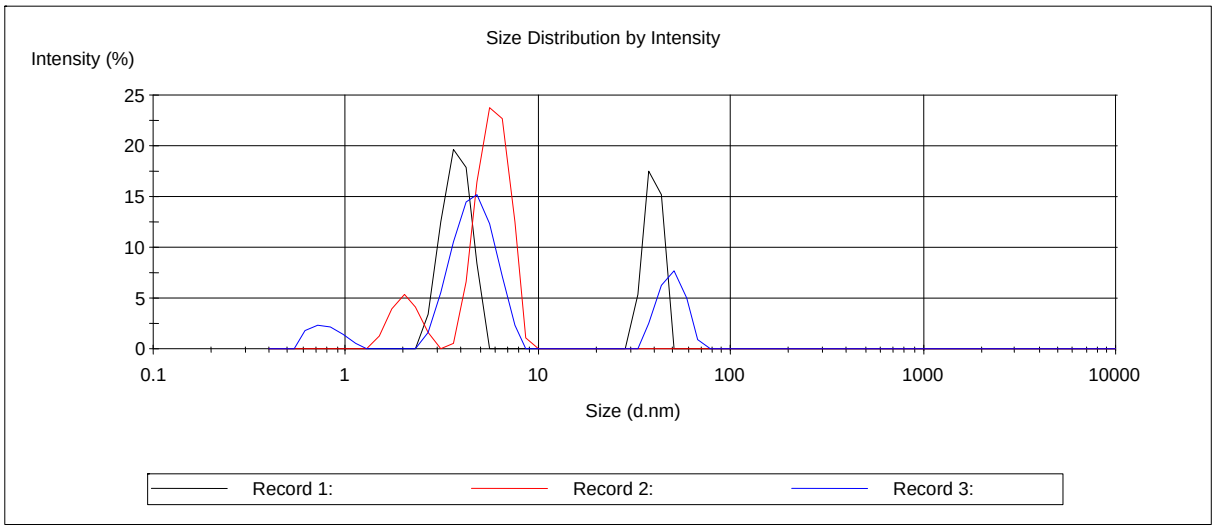



**Figure S27**. DLS spectra of monomer **poly-M2-I1·quinuclidine**.

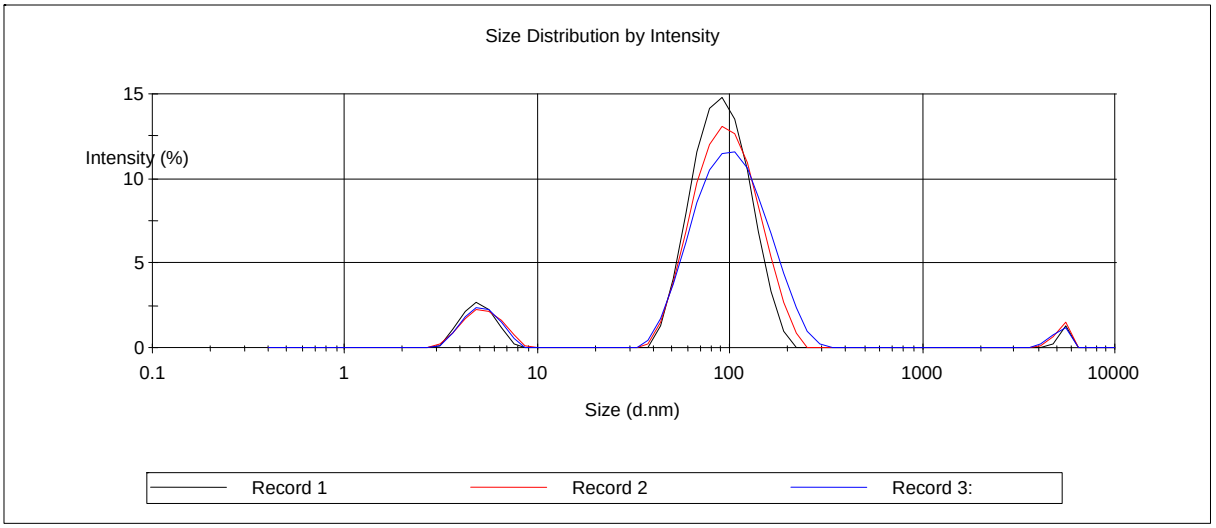



**Figure S28**. DLS spectra of monomer **poly-(*meso*-M5)-I1˙quinuclidine**.

## MALDI-ToF mass analysis

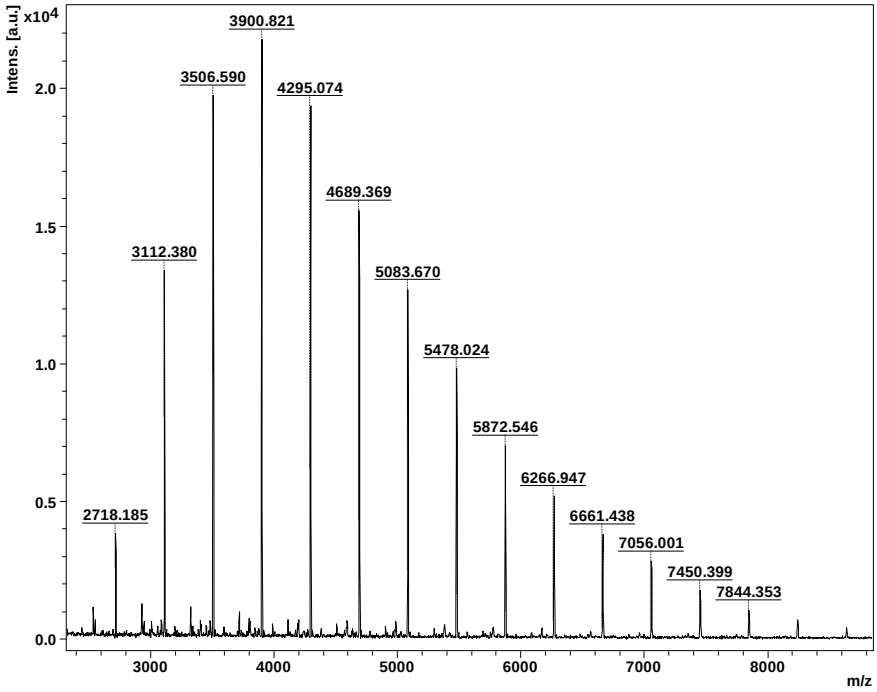



**Figure S29.** MALDI-ToF mass spectrum of oligo-**M1** prepared by the action of **I1˙quinuclidine**.

MALDI-TOF result of **oligo-M1**

| Ser. | rep. unit<br>Mw | resid.<br>PDI | end 1<br>DP | end 2<br>%int | cation | Mn |
|---|---|---|---|---|---|---|
| 1 | 394.163<br>4562.17 | 2.65726<br>1.04509 | 196.018<br>11.0750 | 132.094<br>97.7 | 22.9898 | 4365.34 |

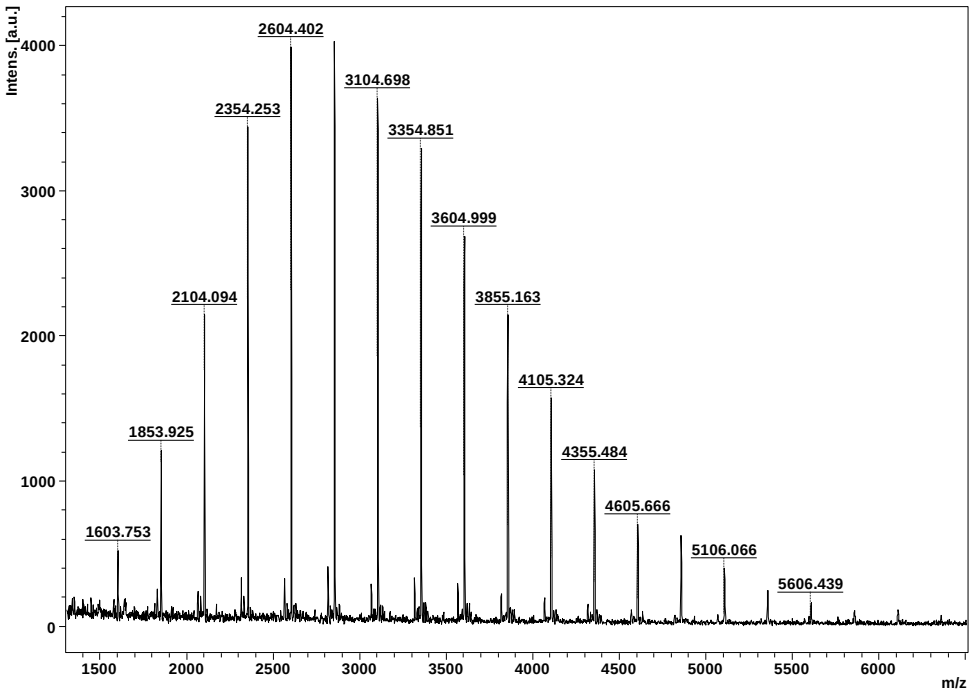



**Figure S30.** MALDI-ToF mass spectrum of oligo-**M2** prepared by the action of **I1**·**quinuclidine**.

MALDI-TOF result of **oligo-M2**

| Ser. | rep. unit<br>$M_w$ | resid.<br>PDI | end 1<br>DP | end 2<br>%int | cation | $M_n$ |
|---|---|---|---|---|---|---|
| 1 | 250.121<br>3312.60 | 2.37397<br>1.06970 | 196.018<br>12.3810 | 132.094<br>91.8 | 22.9898 | 3096.75 |

## IR- spectra

All IR-spectra were measured on a Nicolet 6700 spectrometer with MCTA-detector. Polymer spectra were measured as a film using a smart orbit unit.

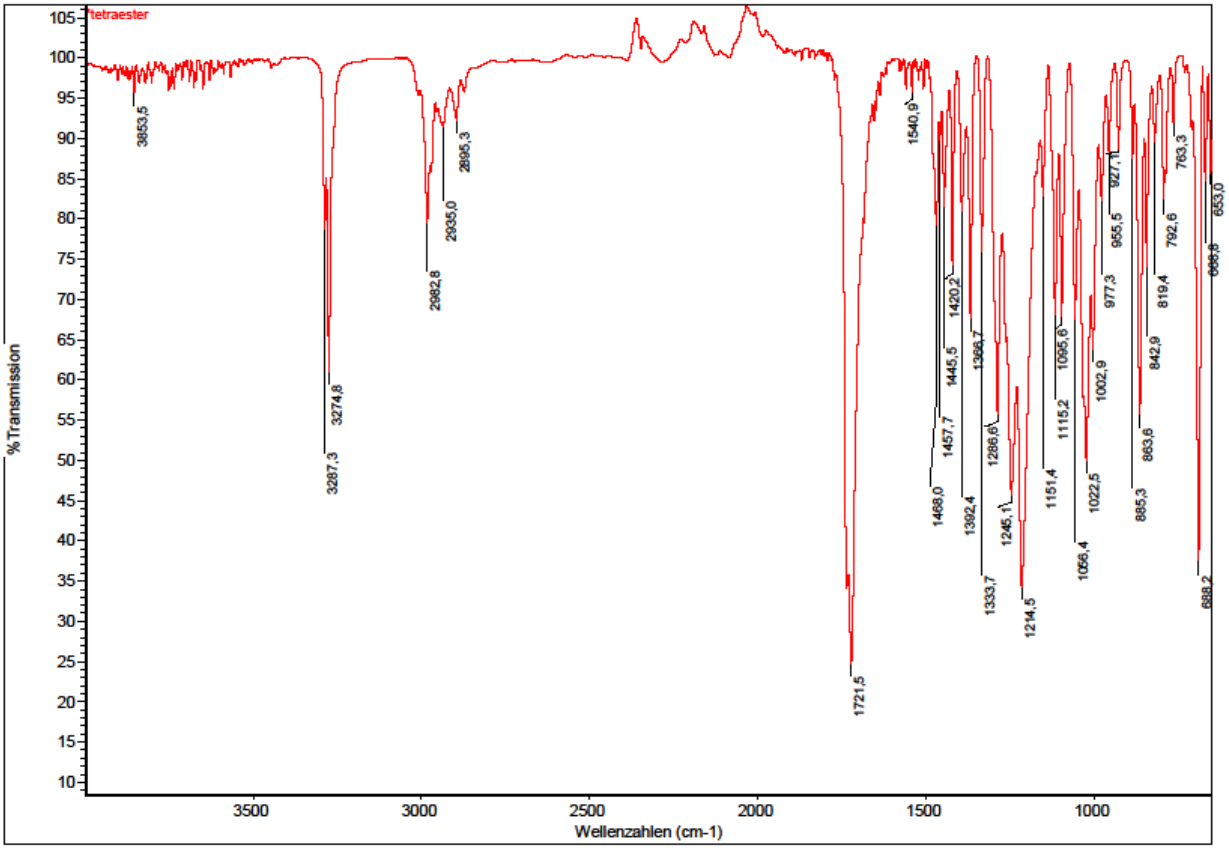



**Figure S31.** FT-IR spectrum of **M1**.

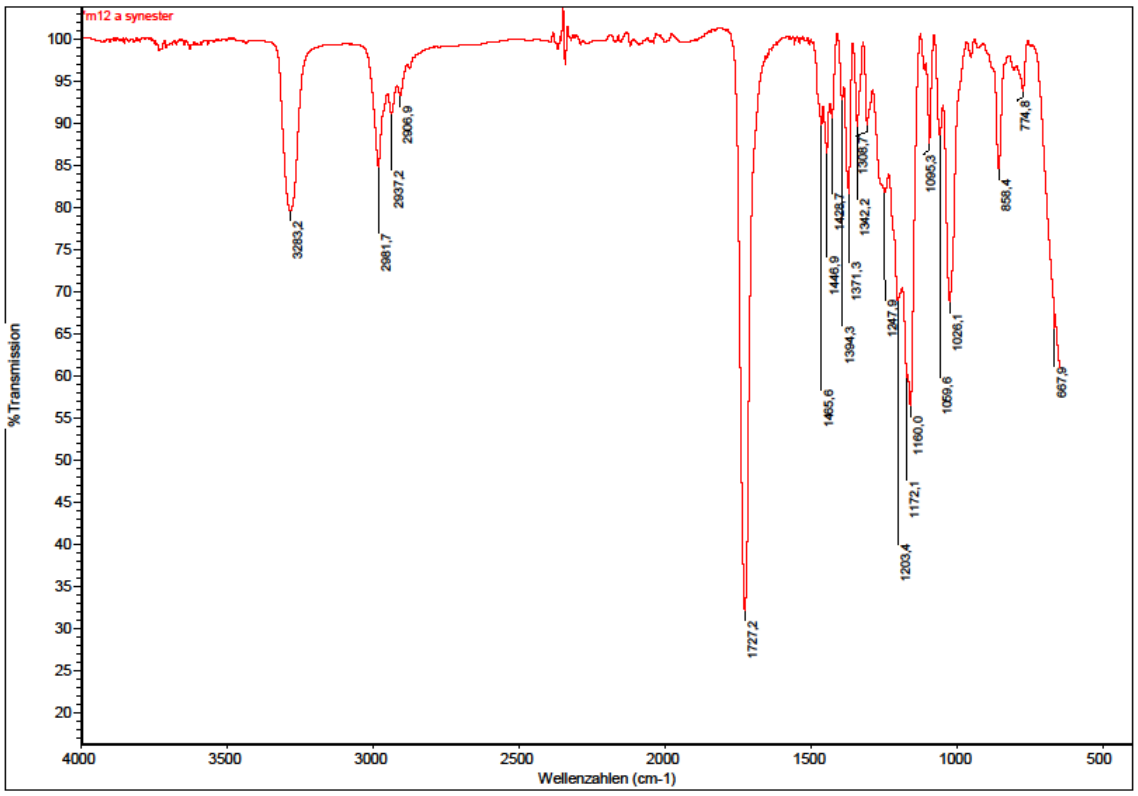



**Figure S32.** FT-IR spectrum of ***meso*-M3**.

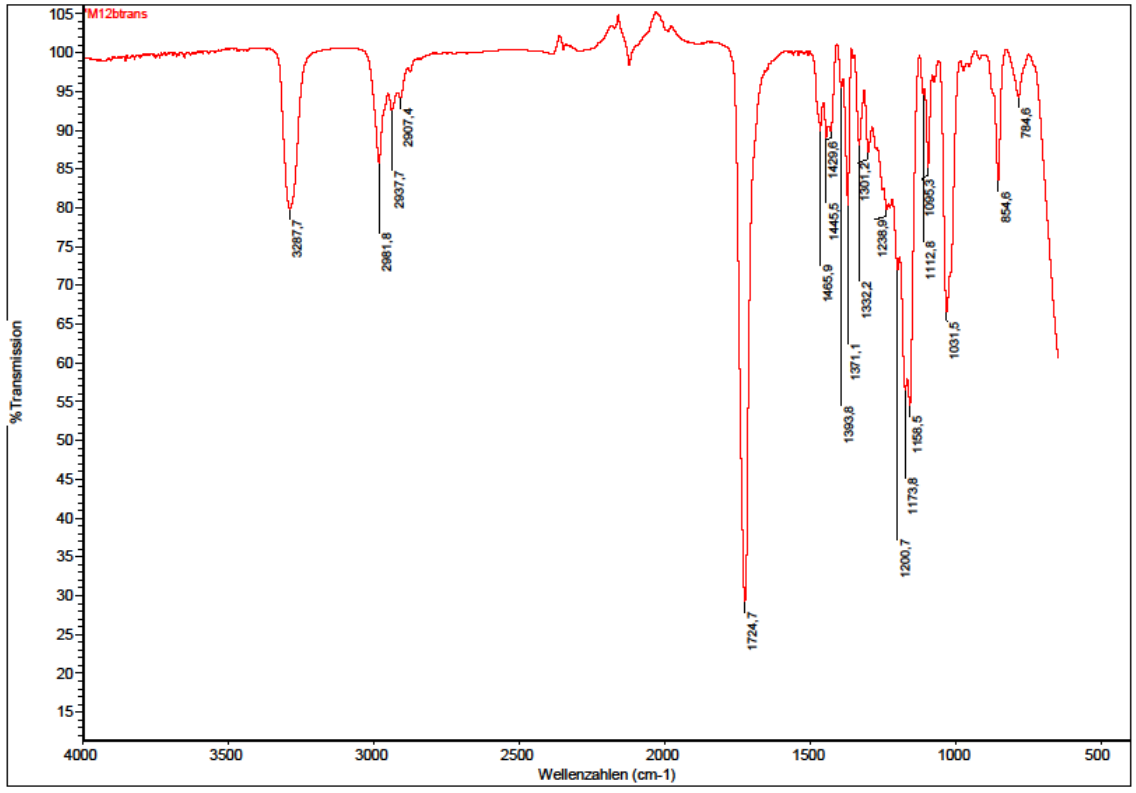



**Figure S33.** FT-IR spectrum of ***rac*-M4**.

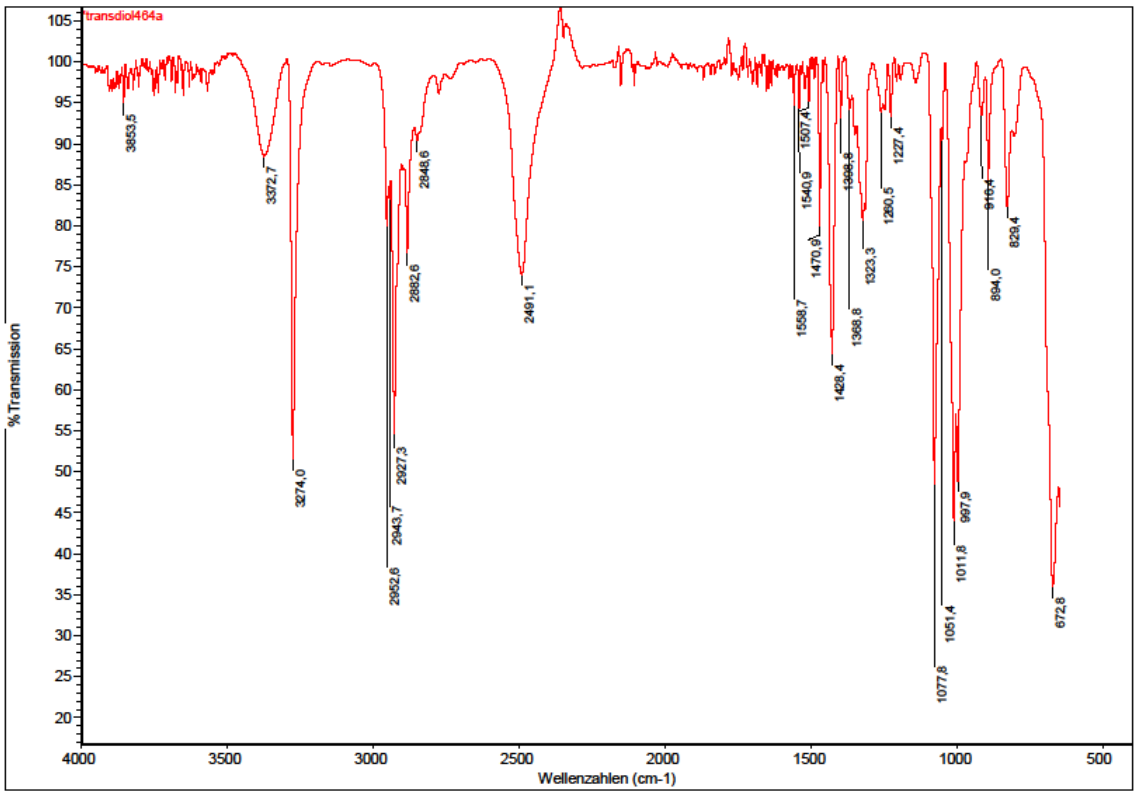



**Figure S34.** FT-IR spectrum of ***meso*-M5.**

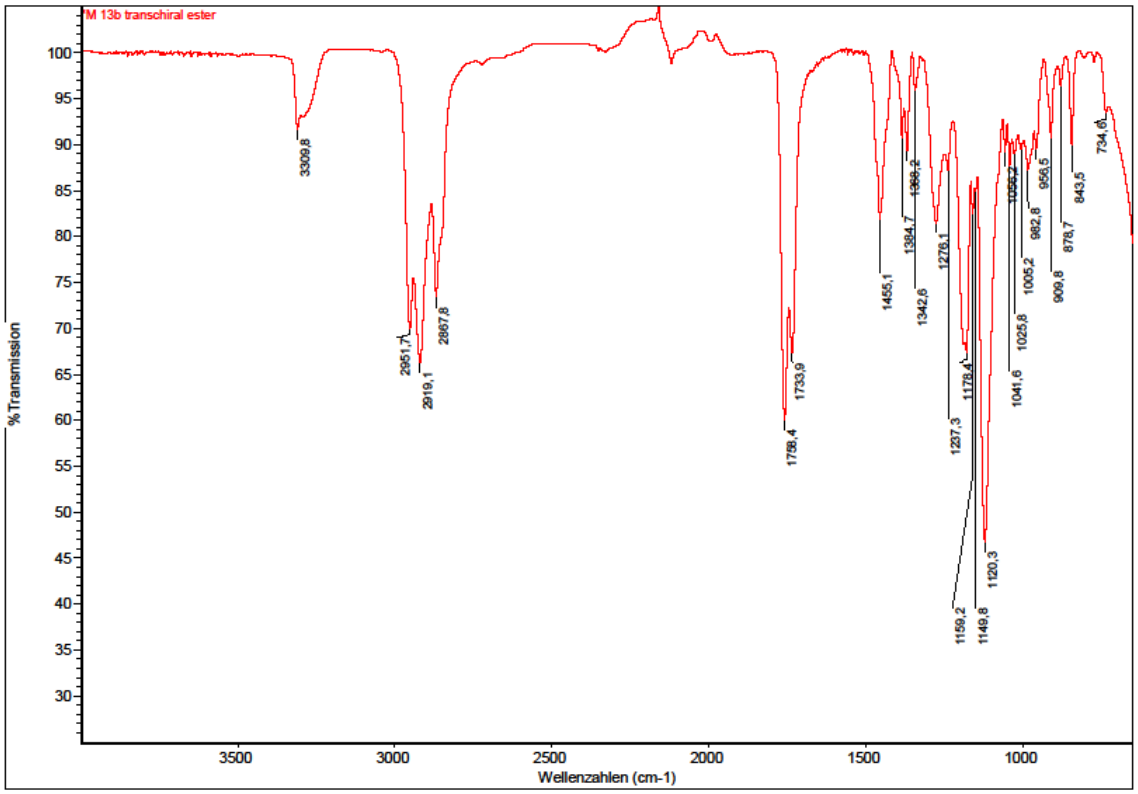



**Figure S35.** FT-IR spectrum of ***rac*-M6**.

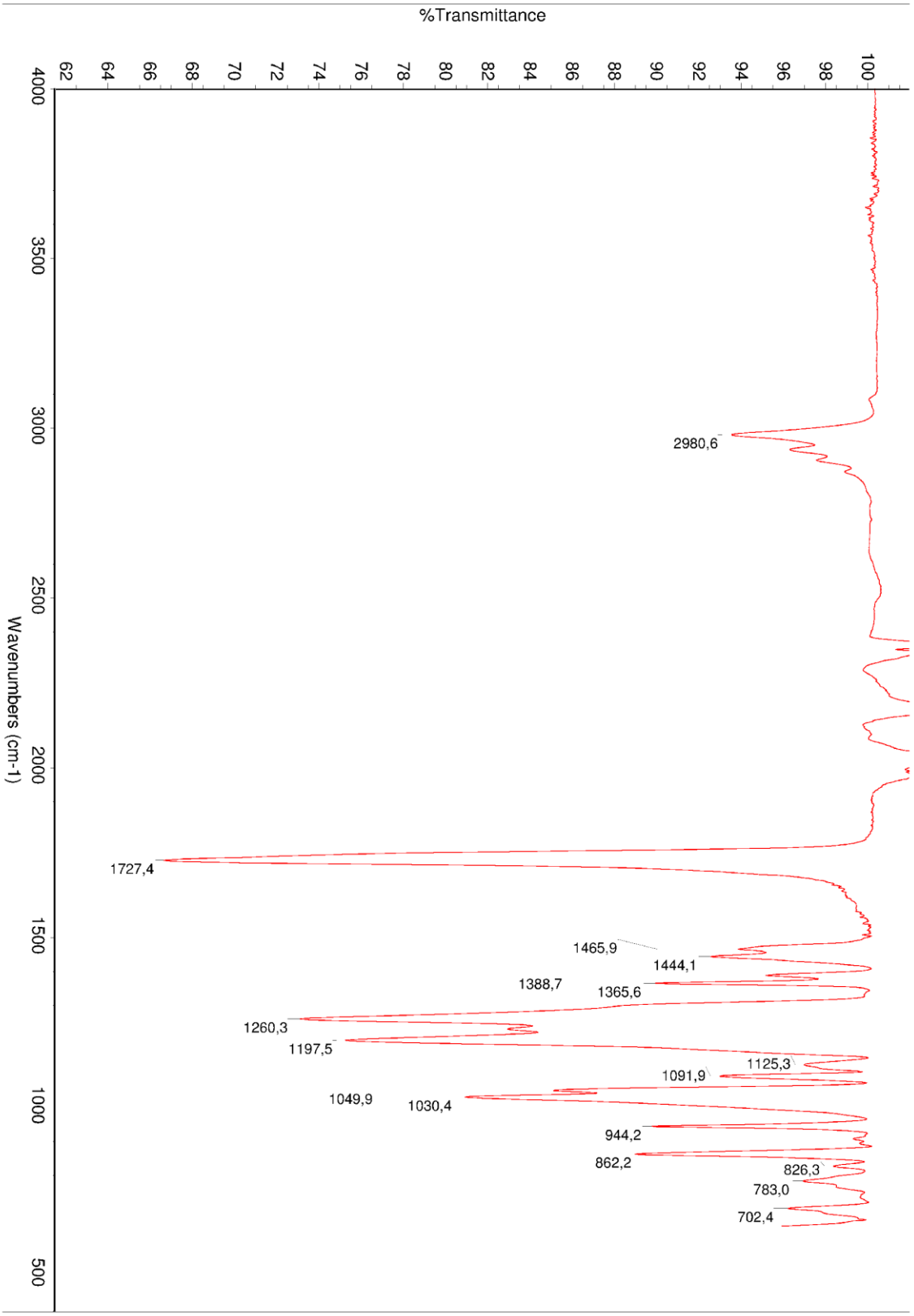



**Figure S36**. FT-IR spectrum of **poly-M1-I1˙quinuclidine**.

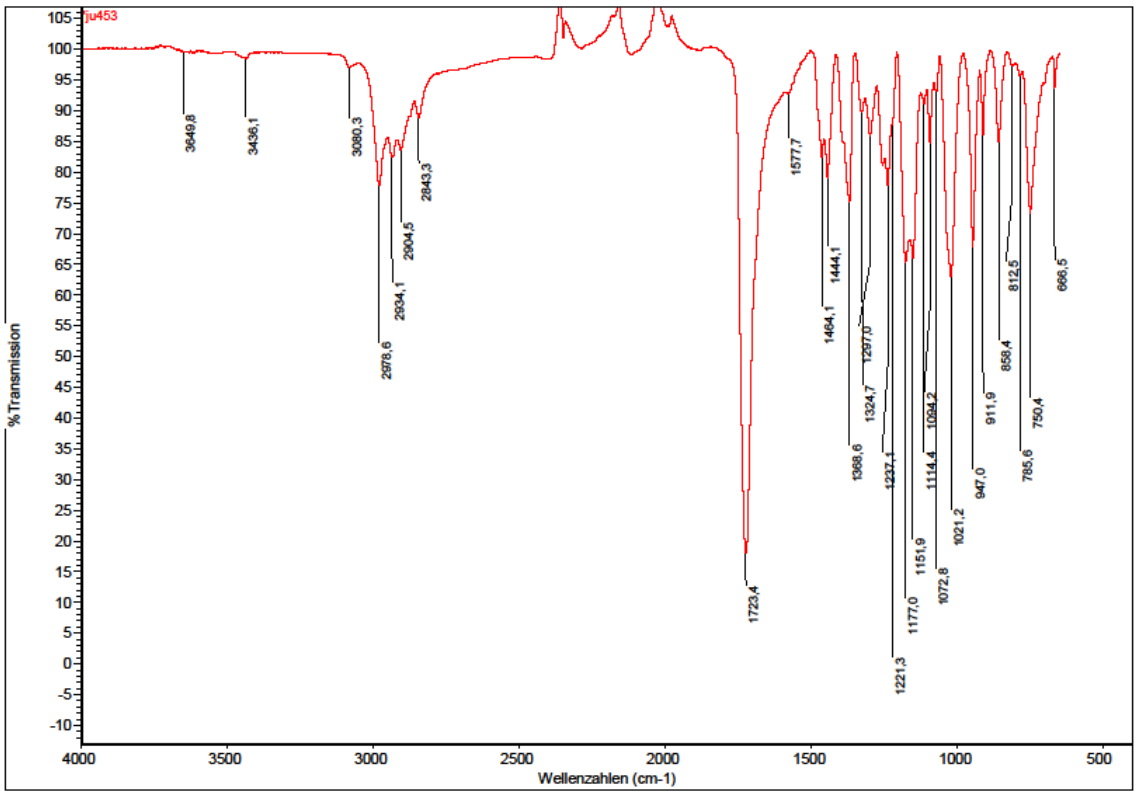



**Figure S37.** FT-IR spectrum of **poly-(*meso*-M3)-I1quinuclidine**.

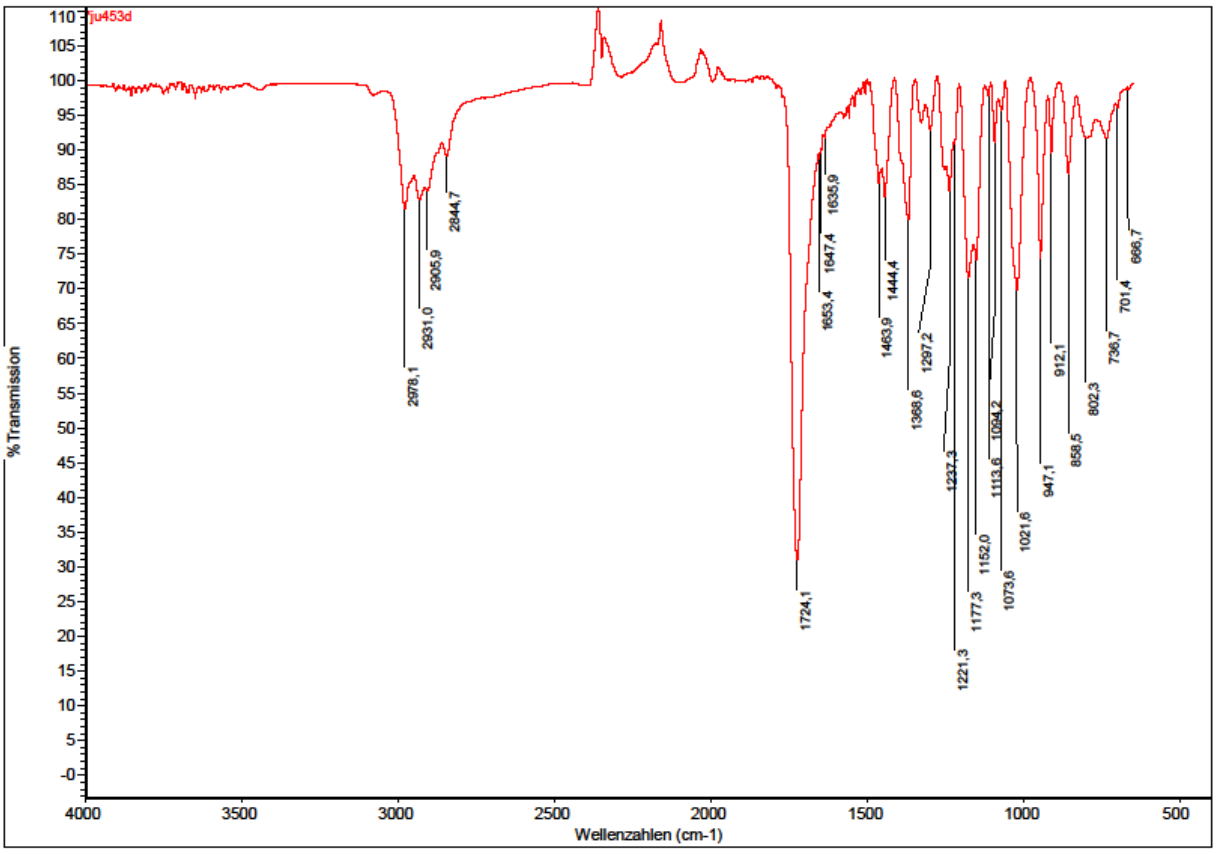



**Figure S38.** FT-IR spectrum of **poly-(*meso*-M3)-I1quinuclidine** after boiling in benzene, 60°C, 2h.

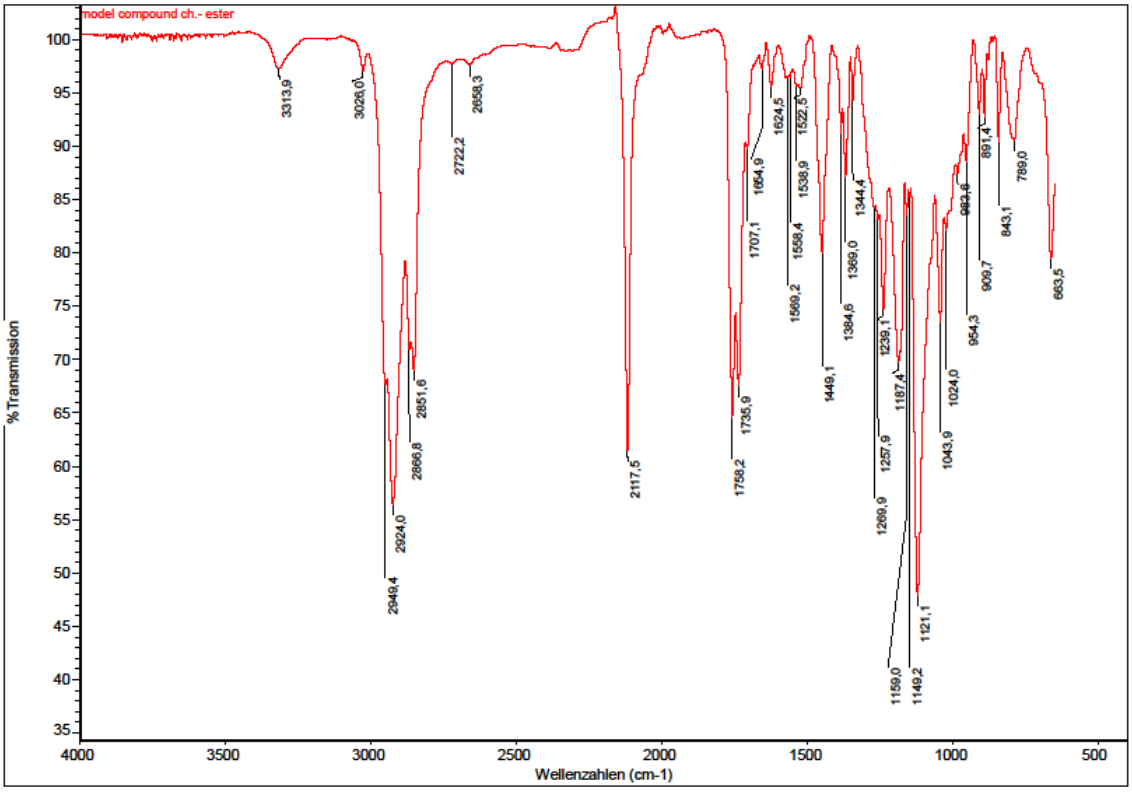



**Figure S39**. FT-IR spectrum of model compound **(C)**.

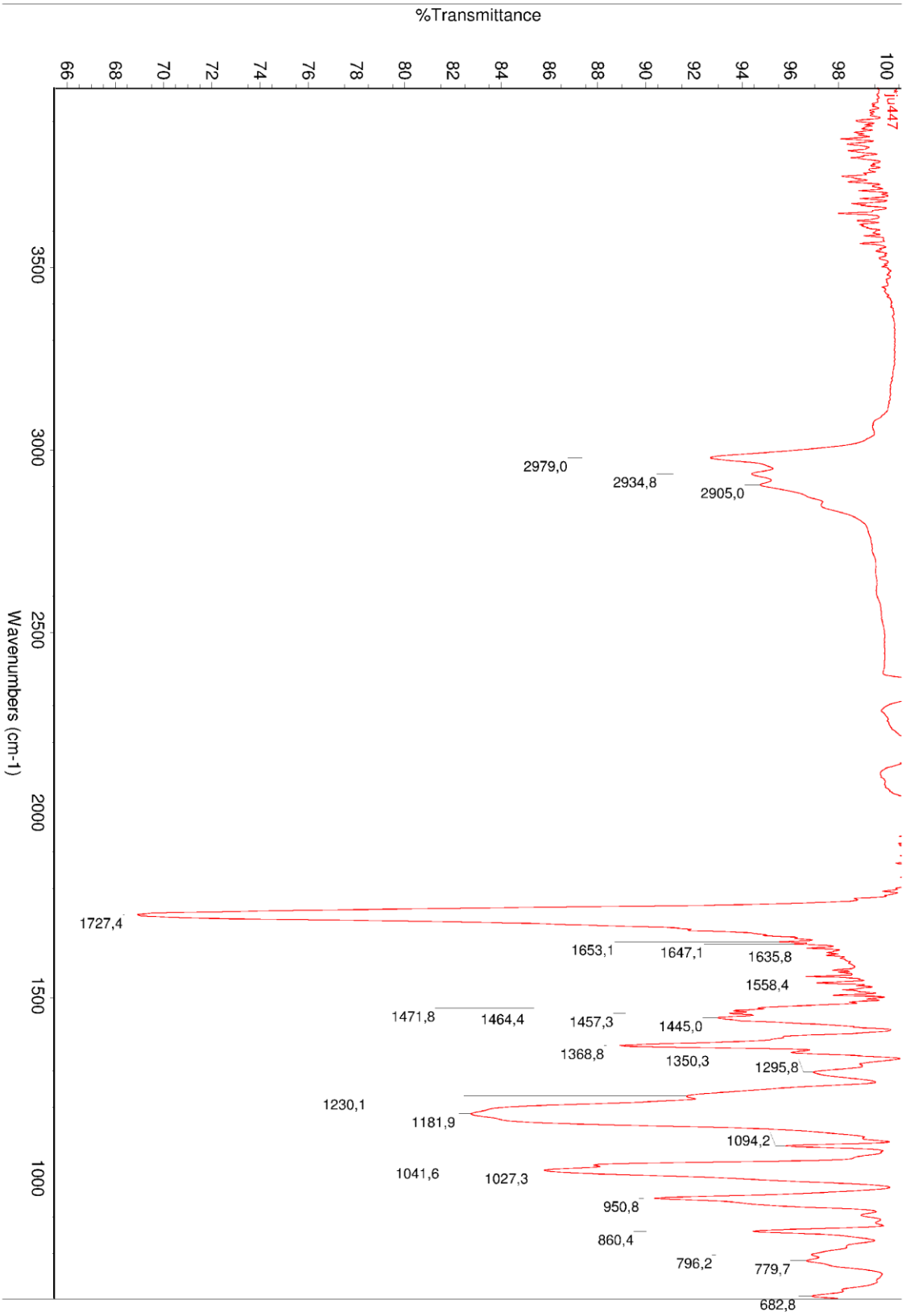



**Figure S40.** FT-IR spectrum of **poly-(*rac*-M4)-I1˙quinuclidine**.

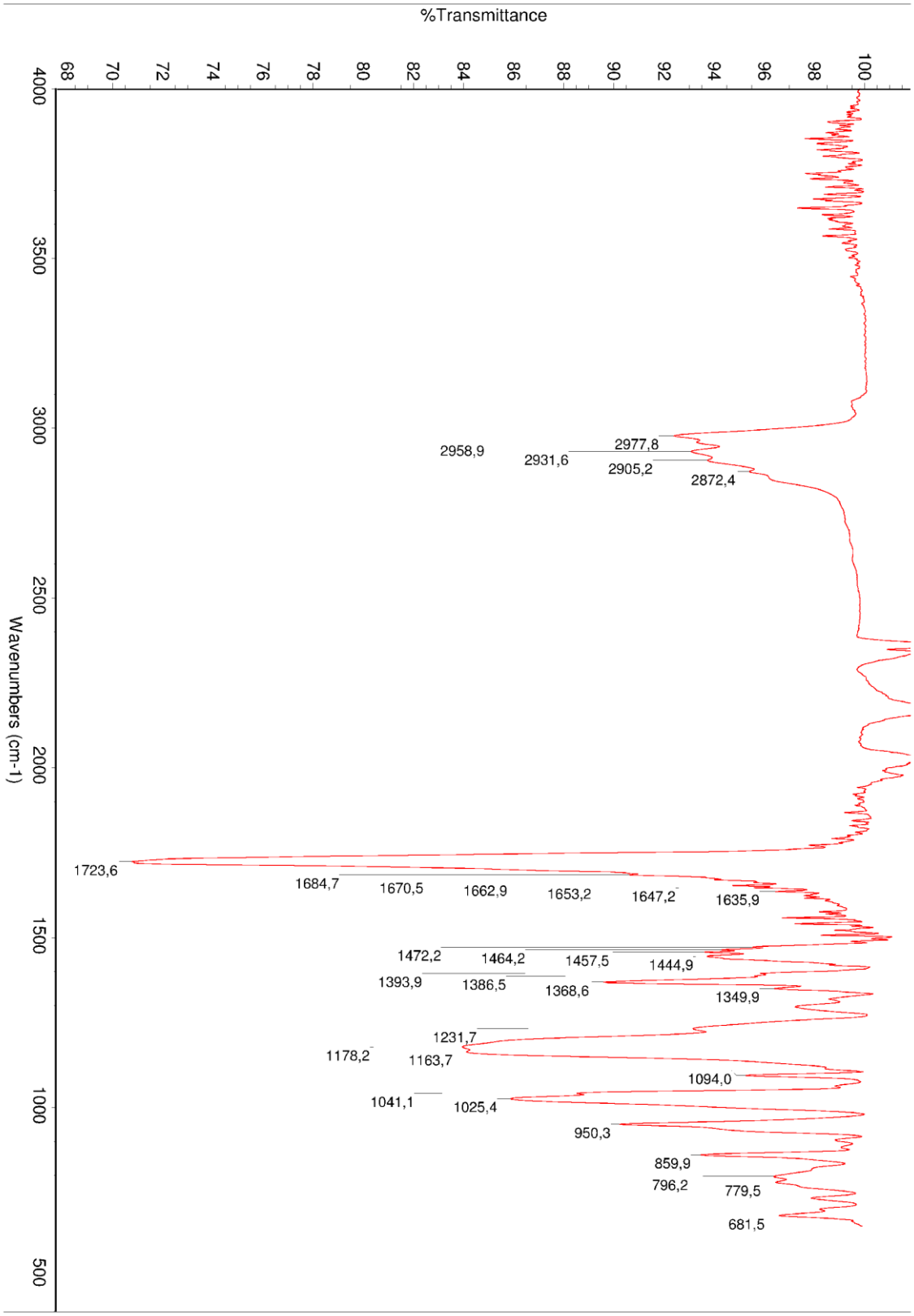



**Figure S41**. FT-IR spectrum of **poly-(*rac*-M4)-I1·quinuclidine** after boiling in benzene, 60°C, 2h.

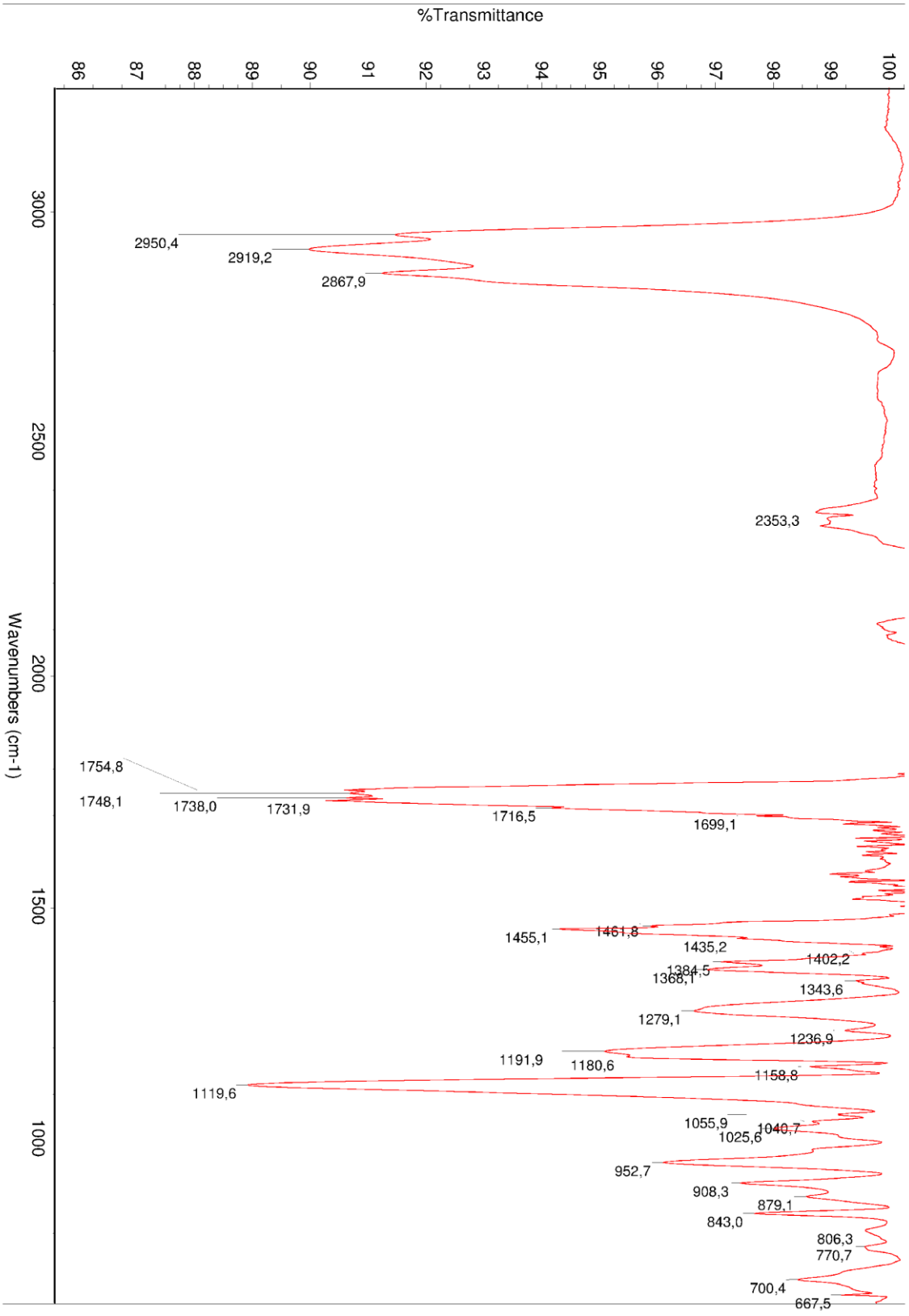



**Figure S42**. FT-IR spectrum of **poly-(*meso*-M5)-I1˙quinuclidine**.

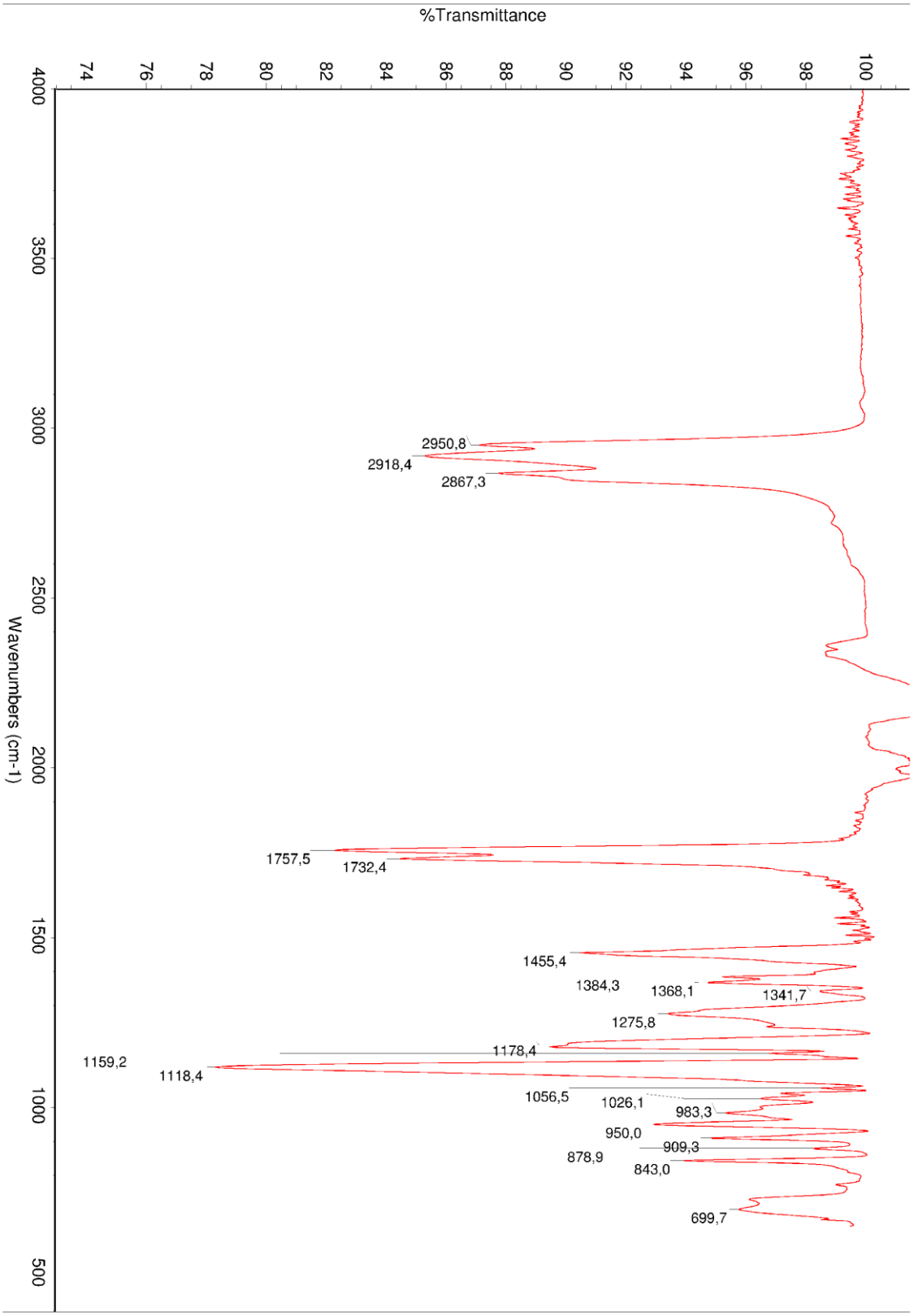



**Figure S43**. FT-IR spectrum of **poly-(*rac*-M6)-I1˙quinuclidine**.

## AB-block copolymers

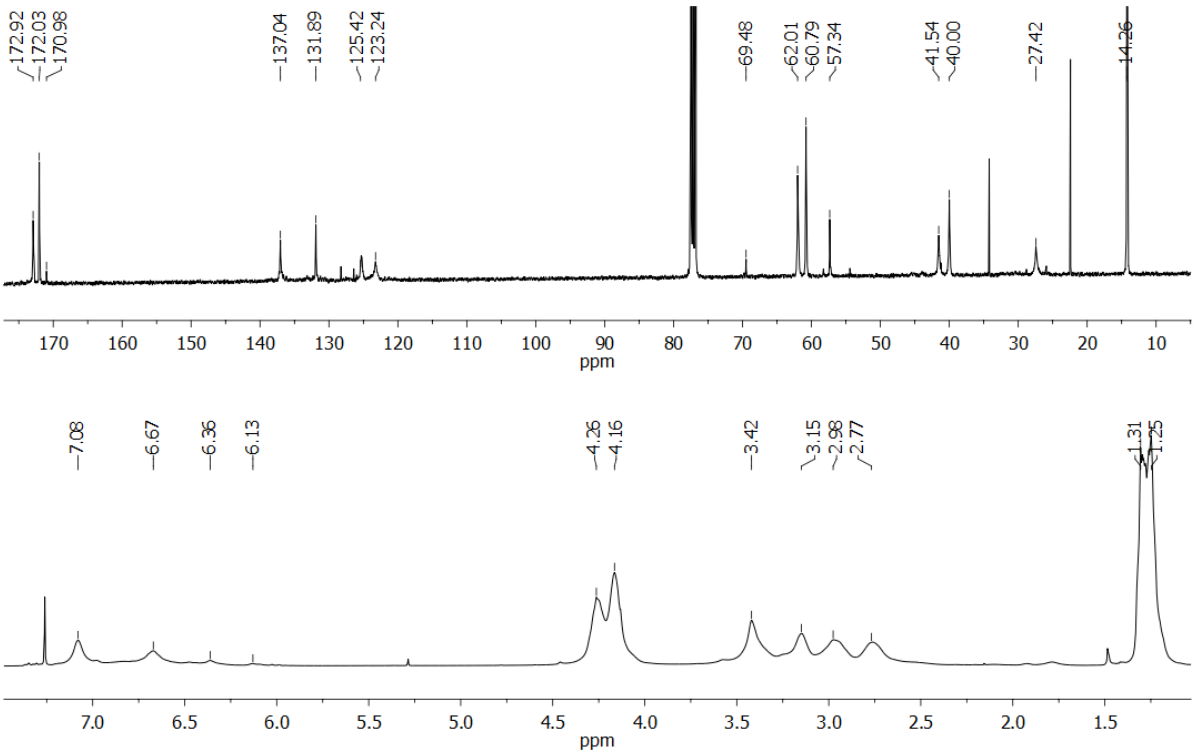
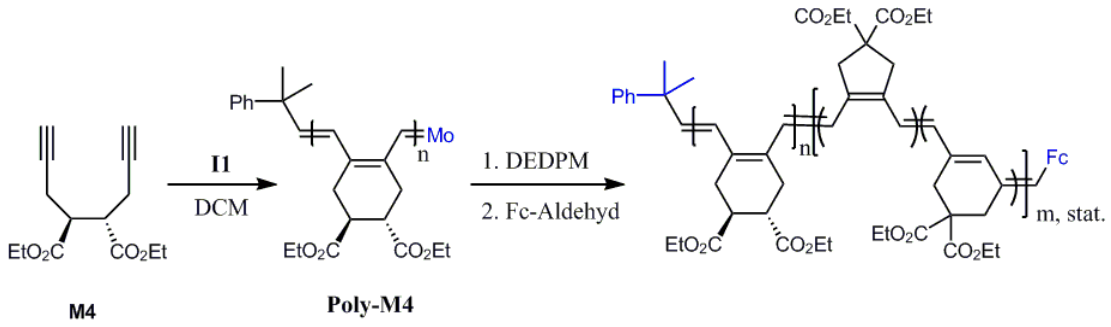
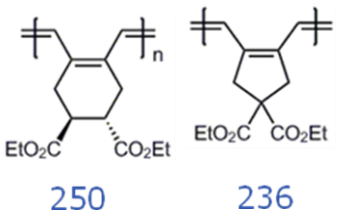



**Figure 44.** $^{1}$H - and $^{13}$C-NMR -spectra of **poly[(*rac*-M4)$_{50}$-*b*-(DEDPM)$_{50}$]** prepared by the action of **I1·quinuclidine**; $CDCl_3$.




**Scheme S2.** Synthesis of **poly[(*rac*-M4)$_{50}$-*b*-(DEDPM)$_{50}$]**.

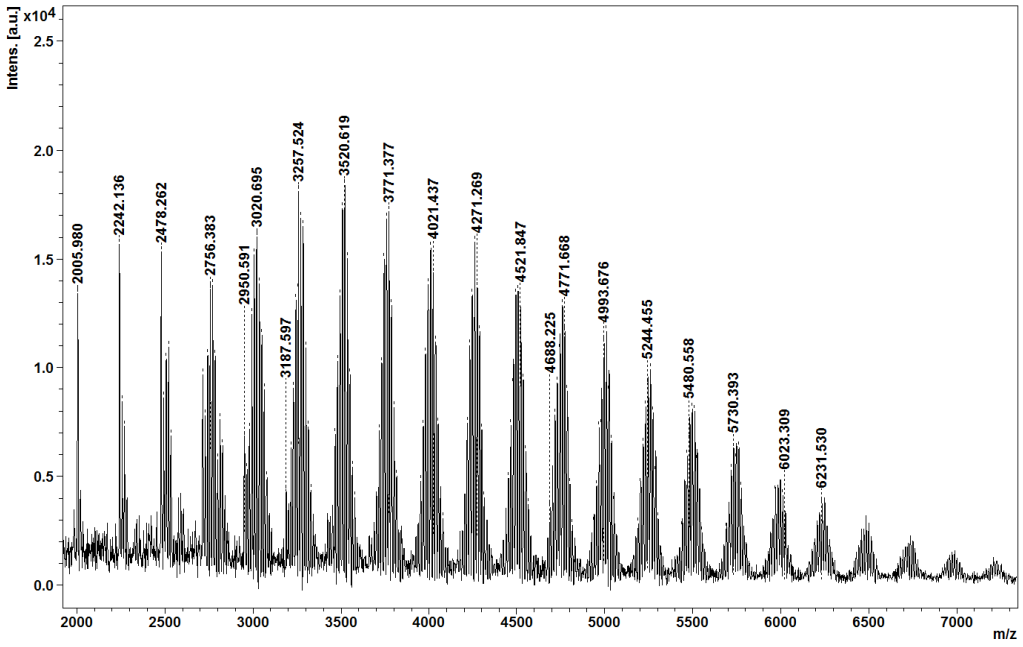



**Figure S45.** MALDI-ToF mass spectrum of **oligomeric-[(*rac*-M4)$_{50}$-*b*-(DEDPM)$_{50}$]** block copolymer prepared by the action of **I1·quinuclidine**.

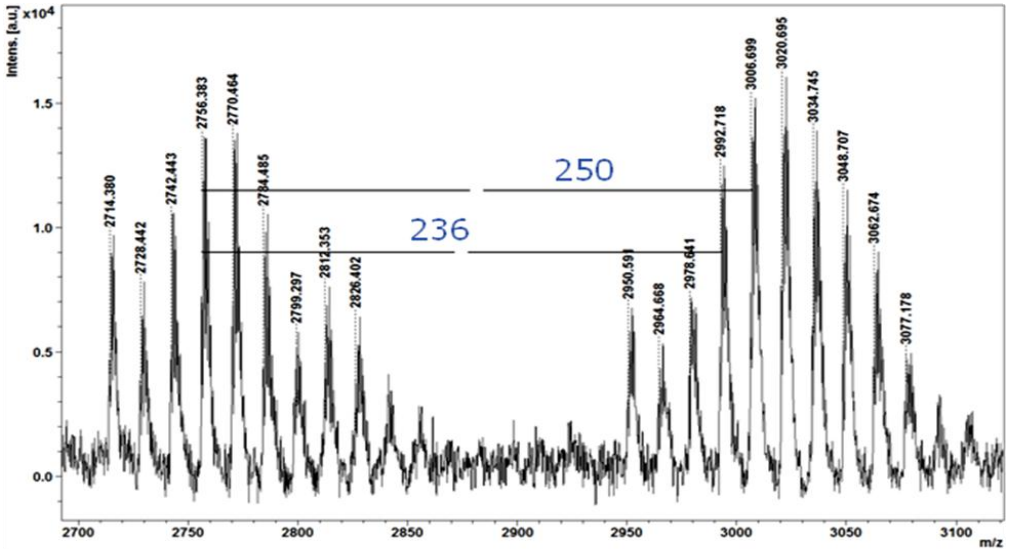



**Figure S46.** Part of the MALDI-ToF mass spectrum of **oligomeric-[(*rac*-M4)$_{50}$-*b*-(DEDPM)$_{50}$]** block copolymer prepared by the action of **I1·quinuclidine,** both repeat units are visible.

Positive ion MALDI-TOF (matrix-assisted laser desorption ionization time-of-flight) measurements were performed on Bruker Autoflex III with a smart ion beam laser (337 nm). Measurements were carried out in reflector mode. Samples were prepared from THF solution

by mixing matrix dithranol (10 mg/ml), polymer (5 mg/ml), and sodium trifluoromethansulfonate (17 mg/ml) in a ratio of 25:5.1.

## UV-VIS spectra

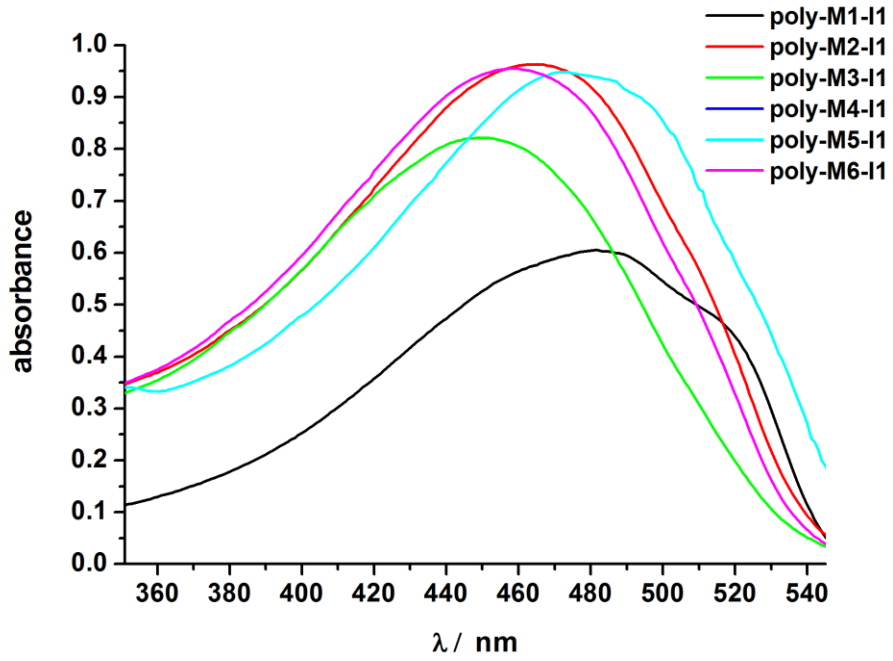



**Figure S47**. UV-Vis spectra of **poly-M1-M6** prepared by the action of **I1·quinuclidine**.

## Kinetic studies

Kinetic studies of monomer **M2,** consisting of both diastereomers **(*R*,*S*)-M2)** and **(*R*,*R*)-M2)**. The monomer consumption was identified by GC-MS using a chiral ß-dextrin-based column.

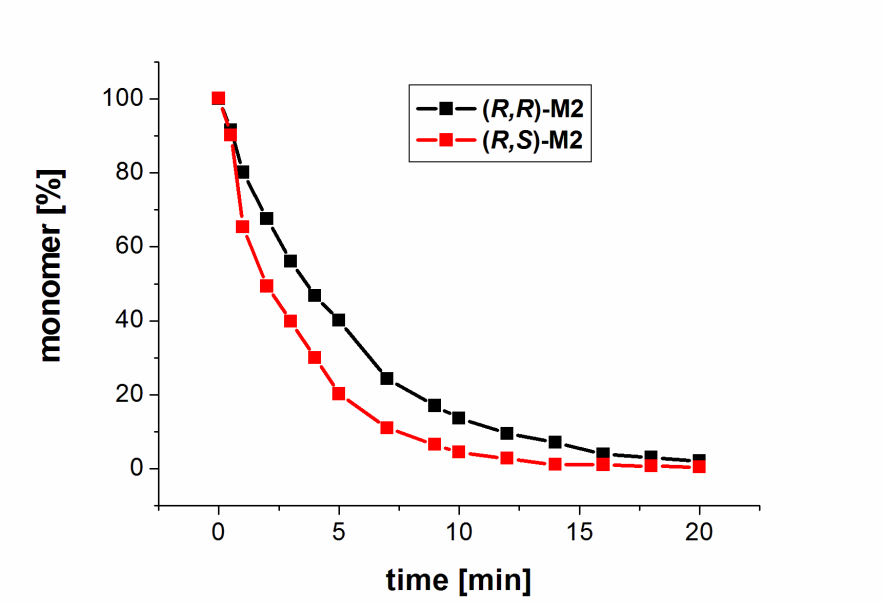



**Figure S48.** plot of the kinetic studies of **M2** by the action of **I1** quinuclidine.

## X-ray structures and X-ray data

Data were collected on a Bruker Kappa Apex 2 duo diffractometer at 100 K. The structure was solved using direct methods with refinement by full matrix least-squares of $F^2$, with the program system SHELXL 97 in connection with a multi-scan absorption correction.[1] All non-hydrogen atoms were refined anisotropically.

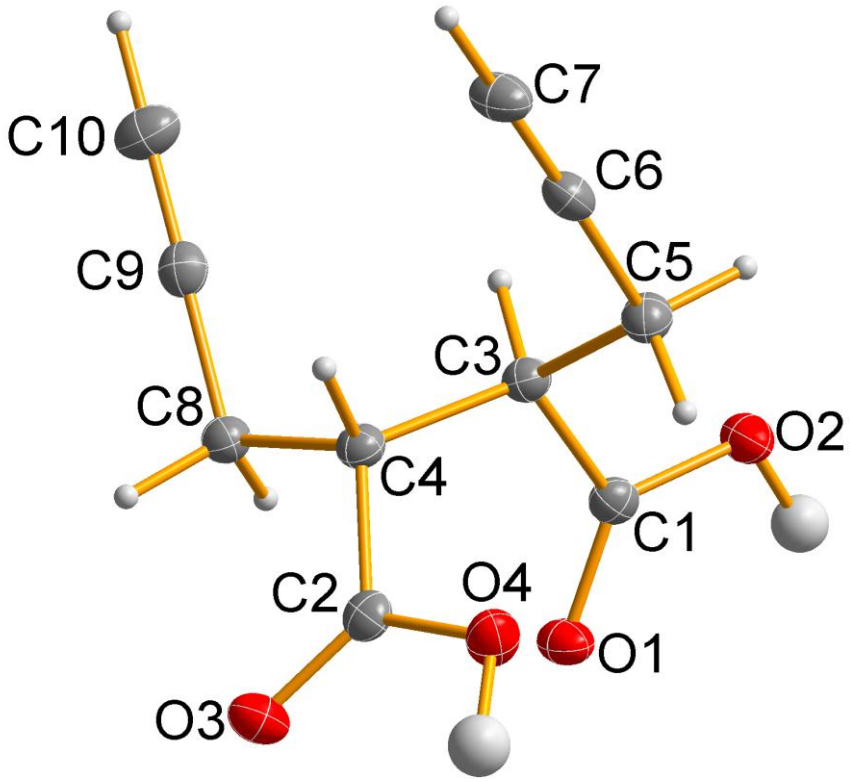



**Figure 49.** X-ray structure of (*R,R),(S,S*)- 1,7-octadiyne-4,5-dicarboxylic acid (***rac*-3**).

**Table S1**. Crystal data of (*R,R),(S,S*)- 1,7-octadiyne-4,5-dicarboxylic acid (***rac*-3**).

| | |
|---|---|
| Empirical Formula | $C_{10}H_{10}O_4$ |
| Molecular weight | 194.18 |
| Crystal system | triclinic |
| Space Group | P-1 |
| Unit cell dimensions | a = 6.8784(5) A alpha = 78.119(3) deg.<br>b = 8.2785(6) A beta = 87.930(3) deg.<br>c = 8.5427(6) A gamma = 88.399(3) deg. |
| Volume | 475.61(6) $Å^3$ |

| | |
|---|---|
| Z | 2 |
| Crystal size | 0.41 x 0.25 x 0.16 mm |
| Theta range for data collection | 5.29 to 66.62° |
| Limiting indices | -7<=h<=8, -9<=k<=9, -9<=l<=9 |
| | |
| Number of Reflections | 7314 |
| Data / restraints / parameters | 1583 / 0 / 136 |
| Goodness-of-fit on $F^2$ | 1.073 |
| Final R indices [I > 2σ (I)] | R1 = 0.0304, wR2 = 0.0758 |
| R indices (all data) | R1 = 0.0584, wR2 = 0.0765 |
| Largest diff. peak and hole | 0.209 and -0.164 e.$A^{-3}$ |

**Table S2**. Bond lengths [Å] and angles [°] for ***rac*-3**.

| | |
|---|---|
| O(1)-C(1) | 1.2229(15) |
| C(1)-O(2) | 1.3166(16) |
| C(1)-C(3) | 1.5130(17) |
| O(2)-H(2A) | 1.00(2) |
| C(2)-O(3) | 1.2263(16) |
| C(2)-O(4) | 1.3123(16) |
| C(2)-C(4) | 1.5128(18) |
| C(3)-C(4) | 1.5364(18) |
| C(3)-C(5) | 1.5520(17) |
| C(3)-H(3) | 1.0000 |
| O(4)-H(4A) | 1.02(2) |
| C(4)-C(8) | 1.5392(17) |
| C(4)-H(4) | 1.0000 |
| C(5)-C(6) | 1.4638(18) |
| C(5)-H(5A) | 0.9900 |
| C(5)-H(5B) | 0.9900 |
| C(6)-C(7) | 1.1884(19) |
| C(7)-H(7) | 0.9500 |
| C(8)-C(9) | 1.4682(18) |
| C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 |
| C(9)-C(10) | 1.1880(19) |
| C(10)-H(10) | 0.9500 |

| | |
|---|---|
| O(1)-C(1)-O(2) | 123.77(11) |
| O(1)-C(1)-C(3) | 123.33(11) |
| O(2)-C(1)-C(3) | 112.90(11) |
| C(1)-O(2)-H(2A) | 109.4(12) |
| O(3)-C(2)-O(4) | 123.86(12) |
| O(3)-C(2)-C(4) | 122.04(11) |
| O(4)-C(2)-C(4) | 113.97(11) |
| C(1)-C(3)-C(4) | 112.54(10) |
| C(1)-C(3)-C(5) | 109.98(10) |
| C(4)-C(3)-C(5) | 115.28(10) |
| C(1)-C(3)-H(3) | 106.1 |
| C(4)-C(3)-H(3) | 106.1 |
| C(5)-C(3)-H(3) | 106.1 |
| C(2)-O(4)-H(4A) | 110.0(13) |
| C(2)-C(4)-C(3) | 115.16(10) |
| C(2)-C(4)-C(8) | 110.50(10) |
| C(3)-C(4)-C(8) | 111.77(10) |
| C(2)-C(4)-H(4) | 106.3 |
| C(3)-C(4)-H(4) | 106.3 |
| C(8)-C(4)-H(4) | 106.3 |
| C(6)-C(5)-C(3) | 111.60(10) |
| C(6)-C(5)-H(5A) | 109.3 |
| C(3)-C(5)-H(5A) | 109.3 |
| C(6)-C(5)-H(5B) | 109.3 |
| C(3)-C(5)-H(5B) | 109.3 |
| H(5A)-C(5)-H(5B) | 108.0 |
| C(7)-C(6)-C(5) | 178.09(14) |
| C(6)-C(7)-H(7) | 180.0 |
| C(9)-C(8)-C(4) | 112.22(11) |
| C(9)-C(8)-H(8A) | 109.2 |
| C(4)-C(8)-H(8A) | 109.2 |
| C(9)-C(8)-H(8B) | 109.2 |
| C(4)-C(8)-H(8B) | 109.2 |
| H(8A)-C(8)-H(8B) | 107.9 |
| C(10)-C(9)-C(8) | 179.30(14) |
| C(9)-C(10)-H(10) | 180.0 |

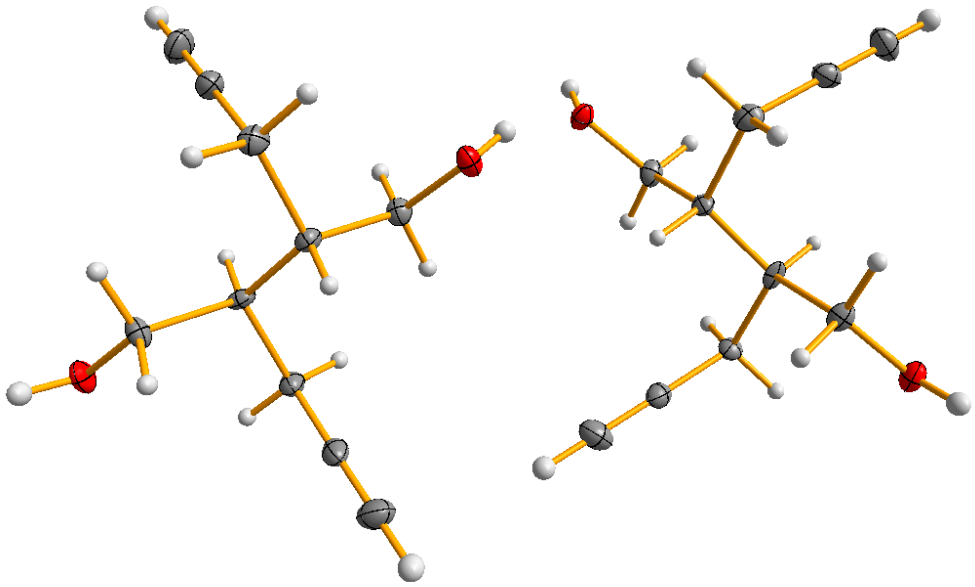


**Figure S50.** X-ray structure of (*R,R),(S,S*)-1,7-octadiyne-4,5-dimethanol (***rac*-5**).

**Table S3**. Crystal data of (*R,R),(S,S*)-1,7-octadiyne-4,5-dimethanol (***rac*-5**).

| | |
|---|---|
| Empirical Formula | $C_{10}H_{14}O_2$ |
| Molecular weight | 166.21 |
| Crystal system | monoclinic |
| Space Group | Cc |
| Unit cell dimensions | a = 14.8047(19) A alpha = 90 deg.<br>b = 24.843(3) A beta = 101.707(6)<br>c = 5.0851(7) A gamma = 90 deg. |
| Volume | 1831.4(4)Å$^3$ |
| Z | 8 |
| Crystal size | 0.72 x 0.38 x 0.31 mm |
| Theta range for data collection | 1.63 to 30.67° |
| Limiting indicies | -20<=h<=21, -35<=k<=35, -7<=l<=3 |
| Number of Reflections | 10773 |
| Data / restraints / parameters | 3955 / 2 / 221 |
| Goodness-of-fit on $F^2$ | 1.033 |
| Final R indices [I > 2σ (I)] | R1 = 0.0416, wR2 = 0.1120 |
| R indices (all data) | R1 = 0.0707, wR2 = 0.1171 |
| Largest diff. peak and hole | 0.225 and -0.328 e.A$^{-3}$ |

**Table S4.** Bond lengths [Å] and angles [°] of (*R,R),(S,S*)-1,7-octadiyne-4,5-dimethanol **(*rac*-5).**

| | |
|---|---|
| O(1A)-C(3A) | 1.432(3) |
| O(1A)-H(1A) | 0.8400 |
| C(1A)-C(3A) | 1.510(4) |
| C(1A)-C(5A) | 1.543(4) |
| C(1A)-C(2A) | 1.545(3) |
| C(1A)-H(1A1) | 1.0000 |
| O(2A)-C(4A) | 1.429(4) |
| O(2A)-H(2A) | 0.8400 |
| C(2A)-C(4A) | 1.532(4) |
| C(2A)-C(8A) | 1.539(4) |
| C(2A)-H(2A1) | 1.0000 |
| C(3A)-H(3A1) | 0.9900 |
| C(3A)-H(3A2) | 0.9900 |
| C(4A)-H(4A1) | 0.9900 |
| C(4A)-H(4A2) | 0.9900 |
| C(5A)-C(6A) | 1.475(4) |
| C(5A)-H(5A1) | 0.9900 |
| C(5A)-H(5A2) | 0.9900 |
| C(6A)-C(7A) | 1.183(5) |
| C(7A)-H(7A) | 0.9500 |
| C(8A)-C(9A) | 1.457(5) |
| C(8A)-H(8A1) | 0.9900 |
| C(8A)-H(8A2) | 0.9900 |
| C(9A)-C(10A) | 1.193(5) |
| C(10A)-H(10A) | 0.9500 |
| O(1B)-C(3B) | 1.433(4) |
| O(1B)-H(1B) | 0.8400 |
| C(1B)-C(3B) | 1.511(5) |
| C(1B)-C(2B) | 1.536(4) |
| C(1B)-C(5B) | 1.546(5) |
| C(1B)-H(1B1) | 1.0000 |
| O(2B)-C(4B) | 1.432(4) |
| O(2B)-H(2B) | 0.8400 |
| C(2B)-C(4B) | 1.519(5) |
| C(2B)-C(8B) | 1.555(5) |
| C(2B)-H(2B1) | 1.0000 |
| C(3B)-H(3B1) | 0.9900 |
| C(3B)-H(3B2) | 0.9900 |
| C(4B)-H(4B1) | 0.9900 |
| C(4B)-H(4B2) | 0.9900 |
| C(5B)-C(6B) | 1.476(5) |
| C(5B)-H(5B1) | 0.9900 |
| C(5B)-H(5B2) | 0.9900 |
| C(6B)-C(7B) | 1.184(5) |
| C(7B)-H(7B) | 0.9500 |
| C(8B)-C(9B) | 1.455(5) |
| C(8B)-H(8B1) | 0.9900 |
| C(8B)-H(8B2) | 0.9900 |

| | |
|---|---|
| C(9B)-C(10B) | 1.196(6) |
| C(10B)-H(10B) | 0.9500 |
| C(3A)-O(1A)-H(1A) | 109.5 |
| C(3A)-C(1A)-C(5A) | 109.7(2) |
| C(3A)-C(1A)-C(2A) | 111.1(2) |
| C(5A)-C(1A)-C(2A) | 113.3(2) |
| C(3A)-C(1A)-H(1A1) | 107.5 |
| C(5A)-C(1A)-H(1A1) | 107.5 |
| C(2A)-C(1A)-H(1A1) | 107.5 |
| C(4A)-O(2A)-H(2A) | 109.5 |
| C(4A)-C(2A)-C(8A) | 109.6(2) |
| C(4A)-C(2A)-C(1A) | 110.6(2) |
| C(8A)-C(2A)-C(1A) | 113.4(2) |
| C(4A)-C(2A)-H(2A1) | 107.7 |
| C(8A)-C(2A)-H(2A1) | 107.7 |
| C(1A)-C(2A)-H(2A1) | 107.7 |
| O(1A)-C(3A)-C(1A) | 109.0(2) |
| O(1A)-C(3A)-H(3A1) | 109.9 |
| C(1A)-C(3A)-H(3A1) | 109.9 |
| O(1A)-C(3A)-H(3A2) | 109.9 |
| C(1A)-C(3A)-H(3A2) | 109.9 |
| H(3A1)-C(3A)-H(3A2) | 108.3 |
| O(2A)-C(4A)-C(2A) | 108.7(2) |
| O(2A)-C(4A)-H(4A1) | 109.9 |
| C(2A)-C(4A)-H(4A1) | 109.9 |
| O(2A)-C(4A)-H(4A2) | 109.9 |
| C(2A)-C(4A)-H(4A2) | 109.9 |
| H(4A1)-C(4A)-H(4A2) | 108.3 |
| C(6A)-C(5A)-C(1A) | 113.0(2) |
| C(6A)-C(5A)-H(5A1) | 109.0 |
| C(1A)-C(5A)-H(5A1) | 109.0 |
| C(6A)-C(5A)-H(5A2) | 109.0 |
| C(1A)-C(5A)-H(5A2) | 109.0 |
| H(5A1)-C(5A)-H(5A2) | 107.8 |
| C(7A)-C(6A)-C(5A) | 178.7(4) |
| C(6A)-C(7A)-H(7A) | 180.0 |
| C(9A)-C(8A)-C(2A) | 113.4(3) |
| C(9A)-C(8A)-H(8A1) | 108.9 |
| C(2A)-C(8A)-H(8A1) | 108.9 |
| C(9A)-C(8A)-H(8A2) | 108.9 |
| C(2A)-C(8A)-H(8A2) | 108.9 |
| H(8A1)-C(8A)-H(8A2) | 107.7 |
| C(10A)-C(9A)-C(8A) | 179.5(4) |
| C(9A)-C(10A)-H(10A) | 180.0 |
| C(3B)-O(1B)-H(1B) | 109.5 |
| C(3B)-C(1B)-C(2B) | 111.9(2) |
| C(3B)-C(1B)-C(5B) | 109.2(3) |
| C(2B)-C(1B)-C(5B) | 112.7(2) |
| C(3B)-C(1B)-H(1B1) | 107.6 |
| C(2B)-C(1B)-H(1B1) | 107.6 |
| C(5B)-C(1B)-H(1B1) | 107.6 |

| | |
|---|---|
| C(4B)-O(2B)-H(2B) | 109.5 |
| C(4B)-C(2B)-C(1B) | 111.3(2) |
| C(4B)-C(2B)-C(8B) | 109.3(3) |
| C(1B)-C(2B)-C(8B) | 112.3(2) |
| C(4B)-C(2B)-H(2B1) | 108.0 |
| C(1B)-C(2B)-H(2B1) | 108.0 |
| C(8B)-C(2B)-H(2B1) | 108.0 |
| O(1B)-C(3B)-C(1B) | 109.3(3) |
| O(1B)-C(3B)-H(3B1) | 109.8 |
| C(1B)-C(3B)-H(3B1) | 109.8 |
| O(1B)-C(3B)-H(3B2) | 109.8 |
| C(1B)-C(3B)-H(3B2) | 109.8 |
| H(3B1)-C(3B)-H(3B2) | 108.3 |
| O(2B)-C(4B)-C(2B) | 108.8(3) |
| O(2B)-C(4B)-H(4B1) | 109.9 |
| C(2B)-C(4B)-H(4B1) | 109.9 |
| O(2B)-C(4B)-H(4B2) | 109.9 |
| C(2B)-C(4B)-H(4B2) | 109.9 |
| H(4B1)-C(4B)-H(4B2) | 108.3 |
| C(6B)-C(5B)-C(1B) | 112.9(3) |
| C(6B)-C(5B)-H(5B1) | 109.0 |
| C(1B)-C(5B)-H(5B1) | 109.0 |
| C(6B)-C(5B)-H(5B2) | 109.0 |
| C(1B)-C(5B)-H(5B2) | 109.0 |
| H(5B1)-C(5B)-H(5B2) | 107.8 |
| C(7B)-C(6B)-C(5B) | 178.8(4) |
| C(6B)-C(7B)-H(7B) | 180.0 |
| C(9B)-C(8B)-C(2B) | 113.5(3) |
| C(9B)-C(8B)-H(8B1) | 108.9 |
| C(2B)-C(8B)-H(8B1) | 108.9 |
| C(9B)-C(8B)-H(8B2) | 108.9 |
| C(2B)-C(8B)-H(8B2) | 108.9 |
| H(8B1)-C(8B)-H(8B2) | 107.7 |
| C(10B)-C(9B)-C(8B) | 179.3(4) |

## References

[1] R. Blessing, *Acta Crystallographica Section A* **1995**, *51*, 33.