

Supporting Information for

**Reversible helix-random coil transition of
poly(*m*-phenylenediethynylene) by rotaxane switch**

Sakiko Suzuki, Fumitaka Ishiwari, Kazuko Nakazono, and Toshikazu Takata*

Department of Organic and Polymeric Materials

Tokyo Institute of Technology

2-12-1 (H-126), Ookayama, Meguro-ku, Tokyo 152-8552, Japan.

ttakata@polymer.titech.ac.jp

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1. Materials and Methods

Commercially available materials and solvents including triethylamine (Wako Pure Chemical Industries, Ltd.), pyridine (Wako Pure Chemical Industries, Ltd.), *N,N'*-diisopropylcarbodiimide (Wako Pure Chemical Industries, Ltd.), NaBH(AcO)₃ (TCI), paraformaldehyde (Nakarai Tesque, Ltd.), trifluoroacetic acid (TCI), 1,8-diazabicyclo[5.4.0]undec-7-ene (Aldrich), *N*-methylpyrrolidone (NMP, Wako Pure Chemical Industries, Ltd.), and CuCl (Aldrich) were used without further purification. SiO₂ column chromatography was performed using Wakogel C-400HG (Wako Pure Chemical Industries Ltd.). Al₂O₃ column chromatography was performed using Merck Aluminum Oxide 90 standardized. *sec*-Ammonium salt **1**^[1], (*R*)-binaphthobenzo-26-crown-8-ether (**R**)-**2**^[2] and 3,5-diethynylbenzoic acid^[3] were prepared according to the literature.

¹H (400 MHz) and ¹³C (100 MHz) NMR spectra were recorded on a JEOL AL-400 spectrometer using CDCl₃ as the solvent and tetramethylsilane as the internal standard. Samples were purified by repeated preparative gel permeation chromatography (GPC) on a JAI Co., Ltd. LC-9204 system (JAIGEL-1H-40) with CHCl₃ as the eluent. IR spectra were recorded on a JASCO FT/IR-230 spectrometer. Melting points were measured with a Stuart Scientific SMP3 (Bibby Scientific). UV-vis spectra were taken on a JASCO V-550 UV-vis spectrophotometer. CD spectra were taken on a JASCO J-820 spectropolarimeter. Specific optical rotations were measured on a JASCO DIP-1000 digital polarimeter in a 10 cm cuvette. Molecular weight and its distribution were measured by gel permeation chromatography (GPC) on a JASCO HSS-1500 System equipped with consecutive linear polystyrene gel columns (TOSO TSK gel G2000HXL and GMHXL) at 30 °C, eluted with 0.3% LiCl at a flow rate of 0.85 mL/min calibrated using polystyrene standards. High-resolution mass spectra (HR-MS) data were taken by the National University Corporation, Tokyo Institute of Technology, Center for Advanced Materials Analysis, on request.

2. Experiments

Synthesis of rotaxane (**R**)-**3**: (Scheme 1)

To a solution of 3,5-diethynylbenzoic acid (357 mg, 2.10 mmol), (*R*)-binaphthobenzo-26-crown-8-ether (**R**)-**2** (1.30 g, 2.10 mmol), and *sec*-ammonium salt **1** (1.01 g, 1.76 mmol) in CHCl_3 (10 mL) was added PBU_3 (1 drops) and *N,N'*-diisopropylcarbodiimide (1.00 mL, 5.60 mmol) at room temperature, and stirred for 24 h. The reaction solution was poured into *n*-hexane (300 mL) and the precipitates were corrected by decantation and purified by SiO_2 column chromatography (CHCl_3 / EtOAc = 1 / 1) and recycle preparative GPC (CHCl_3) to give (**R**)-**3** (1.51 g, 1.20 mmol, 68%) as a colorless foam.

$[\alpha]_{\text{D}}^{25}$: +148° (c = 0.15, THF), m.p. 127.1 – 128.9 °C. ^1H NMR (400 MHz, CDCl_3 , 298 K): δ 8.14 (s, 2H), 7.94 (d, 1H, J = 8.9 Hz), 7.89 (d, 1H, J = 8.3 Hz), 7.84 (d, 1H, J = 8.3 Hz), 7.78 (s, 1H), 7.78 (d, 1H, J = 8.9 Hz), 7.43 (s, 1H), 7.40 (dd, 1H, J = 8.3, 8.3 Hz), 7.36 (dd, 2H, J = 8.3, 8.3 Hz), 7.35 (d, 1H, J = 8.9 Hz), 7.26 (s, 2H), 7.26 (dd, 1H, J = 8.3, 8.3 Hz), 7.26 (dd, 1H, J = 8.3, 8.3 Hz), 7.21 (d, 1H, J = 8.3 Hz), 7.20 (d, 1H, J = 8.3 Hz), 7.03 (d, 1H, J = 8.0 Hz), 7.01 (d, 1H, J = 8.9 Hz), 6.94 (d, 1H, J = 8.0 Hz), 6.86 (dd, 1H, J = 8.0, 8.0 Hz), 6.18 (dd, 1H, J = 8.0, 8.0 Hz), 6.73 (d, 1H, J = 8.0 Hz), 6.56 (d, 1H, J = 8.0 Hz), 5.23 (s, 2H), 4.36 – 4.34 (m, 4H), 4.17 – 4.02 (m, 3H), 3.92 – 3.88 (m, 1H), 3.75 – 3.73 (m, 3H), 3.62 – 3.59 (m, 3H), 3.45 – 3.37 (m, 4H), 3.31 – 3.15 (m, 8H), 3.18 (s, 2H), 3.10 – 3.05 (m, 1H), 2.96 – 2.91 (m, 1H), 1.24 (s, 18H), 1.12 (m, 21H) ppm. ^{13}C NMR (100 MHz, CDCl_3 , 298 K) δ 164.42, 154.40, 154.26, 151.50, 146.40, 146.34, 139.40, 136.63, 133.81, 133.56, 133.07, 130.92, 130.65, 130.02, 129.87, 129.81, 129.67, 128.10, 127.99, 126.56, 126.51, 125.21, 124.88, 124.47, 124.18, 123.72, 123.49, 123.12, 121.99, 121.58, 121.49, 120.64, 117.40, 116.72, 111.97, 111.84, 81.34, 79.36, 71.06, 70.68, 70.60, 70.32, 70.18, 69.28, 69.07, 67.67, 66.26, 52.80, 52.02, 34.77, 31.22 ppm. IR (KBr) ν 3277, 3145, 3065, 2954, 2872,

2110, 1726, 1591, 1506, 1457, 1355, 1306, 1250, 1211, 1111, 948, 845, 749, 671, 558 cm^{-1} . ESI-TOF

HR-MS Calcd for $\text{C}_{72}\text{H}_{78}\text{NO}_{10}$ $[\text{M}-\text{HPF}_6]^+$: $m/z = 1116.5620$ Found: $m/z = 1116.5621$.

Synthesis of (**R**)-**4_N**: (Scheme 1)

A solution of rotaxane (**R**)-**3** (1.11 g, 0.880 mmol), paraformaldehyde (531 mg, 17.7 mmol), $\text{NaBH}(\text{AcO})_3$ (888 mg, 4.40 mmol), and triethylamine (2.53 mL, 17.7 mol) in NMP (5 mL) was stirred for 24 h at room temperature under an Ar atmosphere. The reaction mixture was poured into water (500 mL), and the precipitates were collected by filtration. The products were dissolved in EtOAc; washed with H_2O , sat. NaHCO_3 aq. and brine, dried over MgSO_4 , and concentrated *in vacuo*. The residue was purified by Al_2O_3 column chromatography (EtOAc) to give rotaxane (**R**)-**4_N** (824 mg, 0.730 mmol, 83%) as a colorless foam.

$[\alpha]_{\text{D}}^{25}$: $+173^\circ$ ($c = 0.15$, THF). m.p. = $71.2\text{--}76.8^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3 , 298 K) δ 8.13 (t, 2H, $J = 1.5$ Hz), 7.91 (d, 1H, $J = 9.0$ Hz), 7.87 (d, 1H, $J = 8.2$ Hz), 7.71 (d, 1H, $J = 8.2$ Hz), 7.71 (d, 1H, $J = 8.2$ Hz), 7.53 (d, 1H, $J = 1.5$ Hz), 7.33–7.13 (m, 13H), 6.93 (d, 1H, 1H, $J = 7.8$ Hz), 6.79–6.73 (m, 2H), 6.67 (dd, 1H, $J = 6.9, 2.2$ Hz), 6.62 (dd, 1H, $J = 6.9, 2.2$ Hz), 5.43–5.32 (m, 2H), 4.33–4.28 (m, 1H), 4.02–3.96 (m, 3H), 3.87–3.81 (m, 2H), 3.79–3.72 (m, 2H), 3.66–3.62 (m, 1H), 3.58–3.48 (m, 3H), 3.42–3.38 (m, 4H), 3.34–3.08 (m, 10H), 2.89–2.77 (m, 2H), 2.84 (s, 2H), 2.14 (s, 3H), 1.33 (s, 18H) ppm. ^{13}C NMR (100 MHz, CDCl_3 , 298 K) δ 164.8, 153.8, 153.6, 151.8, 151.5, 147.7, 147.5, 147.4, 147.3, 139.7, 136.9, 136.5, 133.6, 133.5, 133.4, 132.95, 132.91, 132.6, 132.2, 132.1, 130.7, 130.6, 130.4, 130.3, 129.9, 129.8, 129.4, 129.1, 128.1, 128.0, 127.8, 127.5, 126.7, 126.5, 126.0, 125.2, 125.1, 125.0, 124.7, 124.6, 124.5, 124.4, 123.9, 123.7, 123.1, 123.0, 121.4, 121.3, 121.2, 121.1, 121.0, 120.5, 120.1, 119.0, 115.5, 115.2, 115.1, 114.4, 112.1, 112.0, 111.8, 111.6, 104.6, 93.5, 81.6, 79.3, 79.2, 71.4, 71.3, 71.2, 71.1, 71.0, 70.9,

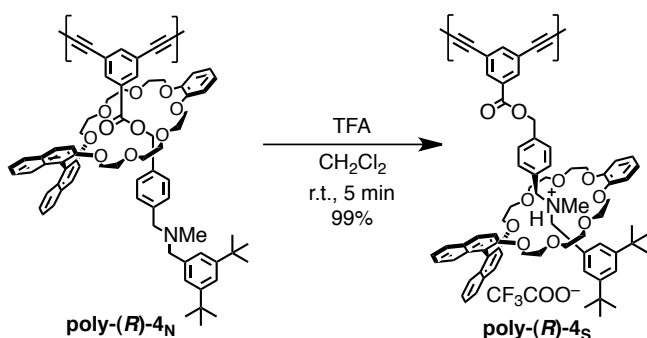
70.7, 70.6, 70.5, 70.4, 70.2, 70.1, 69.5, 68.9, 68.0, 67.9, 67.8, 67.6, 66.5, 66.3, 61.1, 60.9, 60.7, 60.4, 53.5, 39.3, 39.2, 34.8, 34.7, 31.4, 31.3, 18.7, 11.2 ppm. IR (KBr) ν 3431, 3234, 3061, 2952, 2872, 2780, 2109, 1718, 1622, 1591, 1506, 1453, 1362, 1307, 1252, 1218, 1126, 1071, 983, 898, 805, 770, 745 cm^{-1} .
ESI-TOF HR-MS Calcd for $\text{C}_{73}\text{H}_{80}\text{NO}_{10}$ $[\text{M}+\text{H}]^+$: $m/z = 1130.5777$, Found: $m/z = 1130.5786$.

Synthesis of **poly-(R)-4_N**: Polymerization of **(R)-4_N**

To a solution of **(R)-4_N** (100 mg, 88.5 μmol) and CuCl (2.63 mg, 26.6 mmol) in pyridine (500 μL) was stirred for 1 h under O_2 bubbling. To the reaction mixture CHCl_3 and sat. NH_4Cl aq. were added, and the mixture was stirred for 12 h at room temperature. The organic layer was washed with H_2O and brine, dried over MgSO_4 , and concentrated *in vacuo*. The residue was dissolved in CH_2Cl_2 (1 mL) and poured into *n*-hexane (100 mL) to remove pyridine, the precipitates were collected by filtration to give **poly-(R)-4_N** (84 mg, 74.3 mmol, 84%) as a white solid.

$[\alpha]_{\text{D}}^{25}$: $+223^\circ$ ($c = 0.10$, THF). ^1H NMR (400 MHz, CDCl_3 , 293 K) δ 8.18 (s, 2H), 7.91 (d, 1H, $J = 9.0$ Hz), 7.87 (d, 1H, $J = 8.2$ Hz), 7.70 (d, 1H, $J = 8.2$ Hz), 7.69 (d, 1H, $J = 8.2$ Hz), 7.53 (s, 1H), 7.33–7.13 (m, 13H) 6.95 (d, 1H, $J = 7.8$ Hz), 6.95 (d, 1H, $J = 7.8$ Hz), 6.81–6.74 (m, 2H), 6.70 (dd, 1H, $J = 6.9, 2.2$ Hz), 6.65 (dd, 1H, $J = 6.9, 2.2$ Hz), 5.43 (d, 1H, $J = 12.9$ Hz), 5.38 (d, 1H, $J = 12.9$ Hz), 4.35–4.29 (m, 1H), 4.05–3.74 (m, 7H), 3.69–3.09 (m, 20H), 2.93–2.88 (m, 1H), 2.84–2.78 (m, 1H), 2.84 (s, 2H), 2.15 (s, 3H), 1.32 (s, 18H) ppm. IR (KBr) ν 3061, 2952, 2872, 2785, 2359, 2341, 2211, 1723, 1591, 1506, 1456, 1362, 1323, 1251, 1217, 1126, 1068, 943, 805, 745 cm^{-1} . $M_n = 29000$, $M_w = 51000$, PDI = 1.75 (GPC, DMF with 0.3% LiCl, PSt standards)

Scheme S1

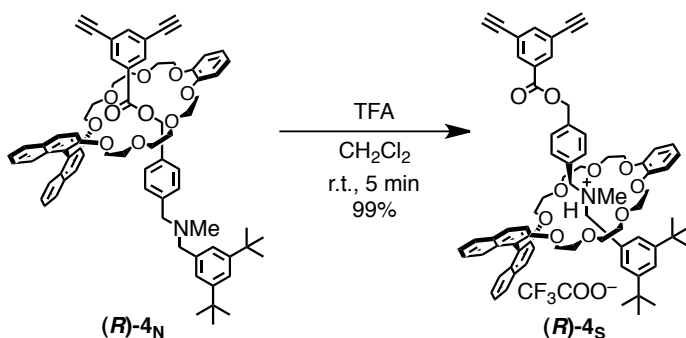


Synthesis of poly-(R)-4_s (Scheme S1)

To the solution of **poly-(R)-4_N** (100 mg, 88.5 μmol) in CH₂Cl₂ (5 mL) was added trifluoroacetic acid (101 mg, 0.880 mmol), and the reaction mixture was stirred for 5 min at room temperature. Then CH₂Cl₂ and trifluoroacetic acid were removed *in vacuo*, and the residue was dissolved in CH₂Cl₂ (1 mL) and poured into *n*-hexane (100 mL) and the precipitates were collected by filtration to give **poly-(R)-4_s** (110 mg, 88.4 μmol, 99%).

[α]_D²⁵: +185° (*c* = 0.10, THF). ¹H NMR (400 MHz, CDCl₃, 298 K) δ 8.23–8.17 (m, 2H), 7.94–7.70 (m, 5H), 7.45 (s, 1H), 7.38–6.78 (m, 17H), 6.71–6.64 (m, 1H), 5.25–5.14 (m, 2H), 4.74–3.15 (m, 29H), 2.63–2.46 (m, 3H), 1.24–1.23 (m, 18H) ppm. IR (KBr) ν 3066, 2946, 2924, 2871, 2222, 1729, 1689, 1623, 1591, 1506, 1457, 1430, 1355, 1250, 1200, 1128, 949, 805, 748, 719, 588 cm⁻¹.

Scheme S2



Synthesis of (R)-4_S (Scheme S2)

To the solution of **(R)-4_N** (100 mg, 88.4 μmol) in CH_2Cl_2 (5 mL) was added trifluoroacetic acid (101 mg, 0.880 mmol), and the reaction mixture was stirred for 5 min at room temperature. Then CH_2Cl_2 and trifluoroacetic acid were removed *in vacuo*, and the residue was purified by SiO_2 column chromatography (CHCl_3) to give rotaxane **(R)-4_S** (110 mg, 88.5 μmol , 99%) as a colorless foam.

$[\alpha]_{\text{D}}^{25}$: +153° ($c = 0.15$, THF). m.p. = 75.6–79.8 °C. ^1H NMR (400 MHz, CDCl_3 , 298 K) δ 8.17 (s, 1.2H), 8.11 (s, 0.8H), 7.92 (d, 0.6H, $J = 9.0$ Hz), 7.86–7.76 (m, 3.4H) 7.71–7.67 (m, 1H), 7.46 (s, 0.6H), 7.42 (s, 0.4H), 7.38–7.03 (m, 12H), 6.97–6.83 (m, 5H), 6.69–6.66 (m, 1H), 5.18 (d, 0.4H, $J = 12.9$ Hz), 5.13 (d, 1.6H, $J = 12.9$ Hz), 4.74–4.71 (m, 0.6H), 4.64–4.44 (m, 2H), 4.35–4.30 (m, 0.4H), 4.24–3.11 (m, 27H), 2.65 (d, 1.2H, $J = 4.5$ Hz), 2.53 (d, 1.8H, $J = 4.5$ Hz), 2.84–2.78 (m, 1H), 2.84 (s, 2H), 2.15 (s, 3H), 1.24–1.23 (m, 18H) ppm. ^{13}C NMR (100 MHz, CDCl_3 , 298 K) δ 164.5, 164.4, 160.3, 160.0, 153.7, 153.6, 151.9, 151.6, 147.6, 147.4, 147.2, 139.6, 139.5, 136.9, 136.6, 133.6, 133.5, 133.4, 133.2, 133.1, 131.9, 130.7, 130.6, 130.3, 130.2, 130.1, 130.0, 129.9, 129.8, 129.7, 129.6, 129.4, 129.2, 129.1, 128.2, 128.1, 128.0, 127.8, 127.5, 126.7, 126.6, 126.5, 125.7, 125.2, 125.1, 125.0, 124.9, 124.7, 124.4, 124.3, 124.0,

123.8, 123.3, 123.2, 121.5, 121.4, 121.3, 121.2, 121.0, 120.7, 120.1, 119.2, 115.6, 115.2, 114.6, 112.2, 111.8, 111.6, 81.4, 79.4, 79.3, 71.4, 71.3, 71.1, 71.0, 70.9, 70.8, 70.7, 70.6, 70.5, 70.4, 70.3, 70.2, 70.1, 69.5, 69.0, 68.1, 68.0, 67.8, 67.6, 66.5, 66.3, 61.1, 60.9, 60.7, 60.6, 39.3, 39.2, 34.8, 34.7, 31.3, 31.3, 29.7 ppm. IR (KBr) ν 3283, 3066, 2955, 2875, 2110, 1780, 1729, 1684, 1622, 1591, 1506, 1456, 1436, 1363, 1355, 1307, 1250, 1206, 1132, 1087, 1068, 985, 950, 900, 880, 868, 807, 749, 708 cm^{-1} . ESI-TOF HR-MS Calcd for $\text{C}_{73}\text{H}_{80}\text{NO}_{10}$ $[\text{M}-\text{CF}_3\text{COO}]^+$: $m/z = 1130.5777$, Found: $m/z = 1130.5780$.

3. Spectra

3-1. ^1H and ^{13}C NMR spectra, HH COSY correlations

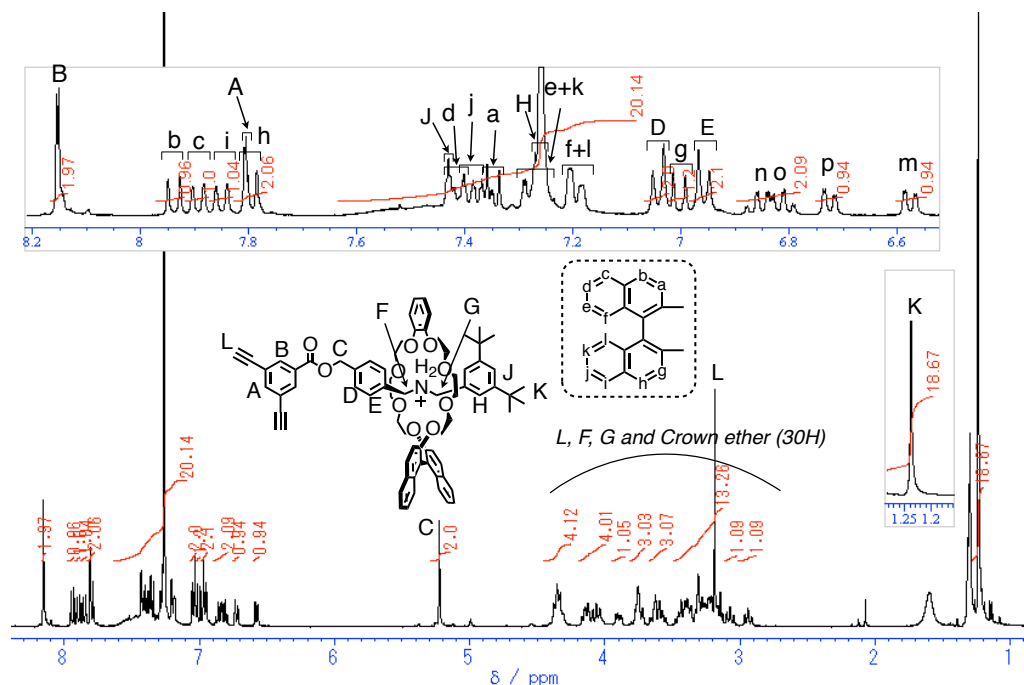


Figure S1. ^1H NMR spectrum of (R) -3 (400 MHz, CDCl_3 , 298 K)

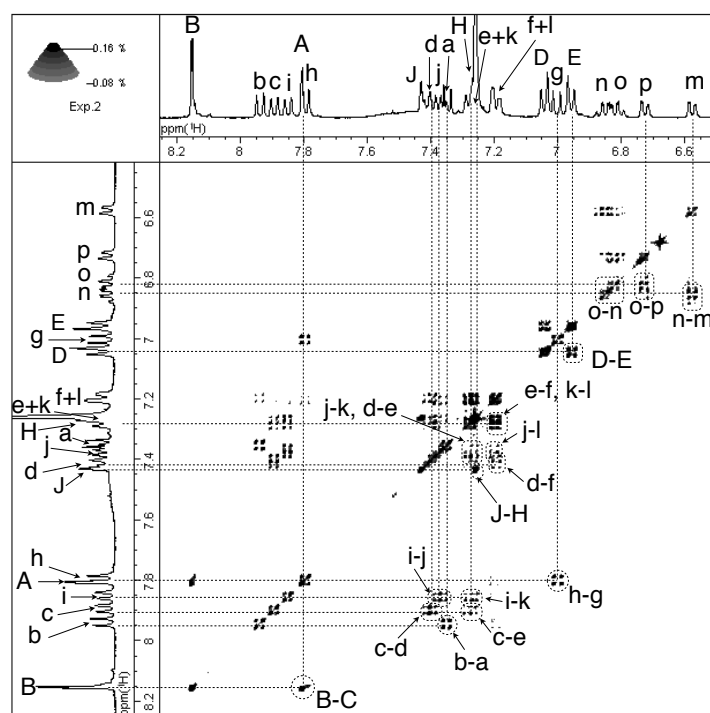


Figure S2. HH COSY correlations of (**R**)-**3** (400 MHz, CDCl₃, 298 K)

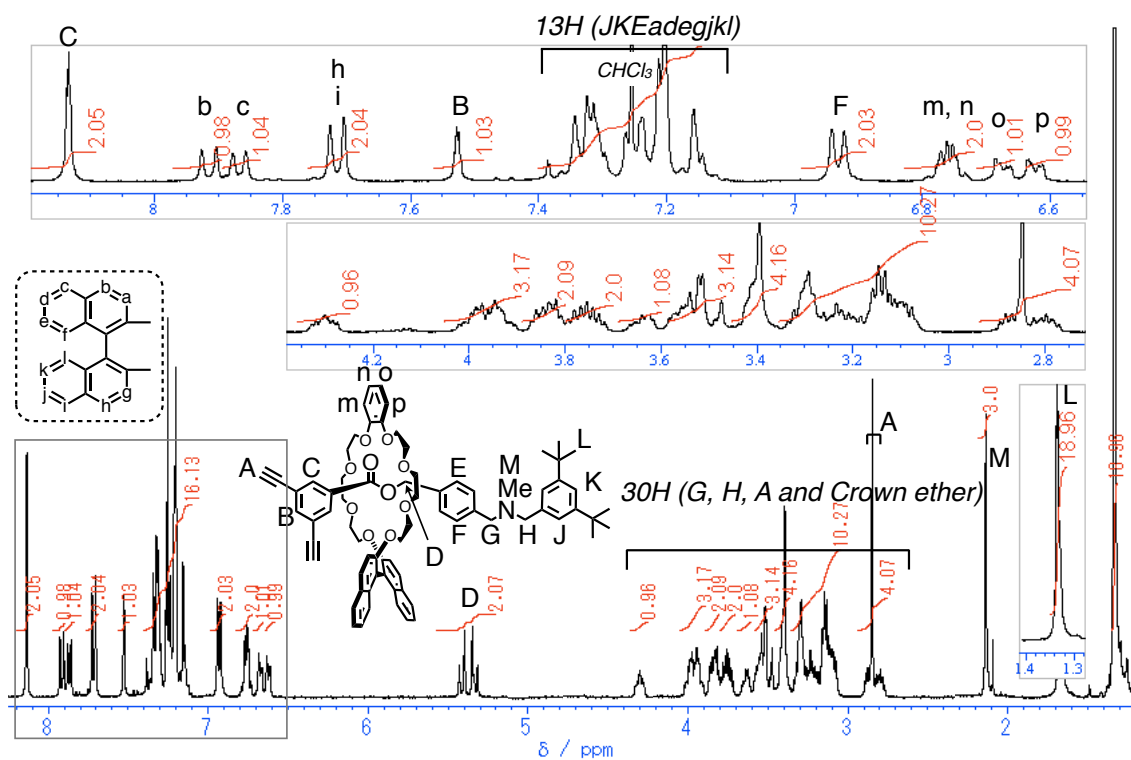


Figure S3. ¹H NMR spectrum of (**R**)-**4_N** (400 MHz, CDCl₃, 298 K)

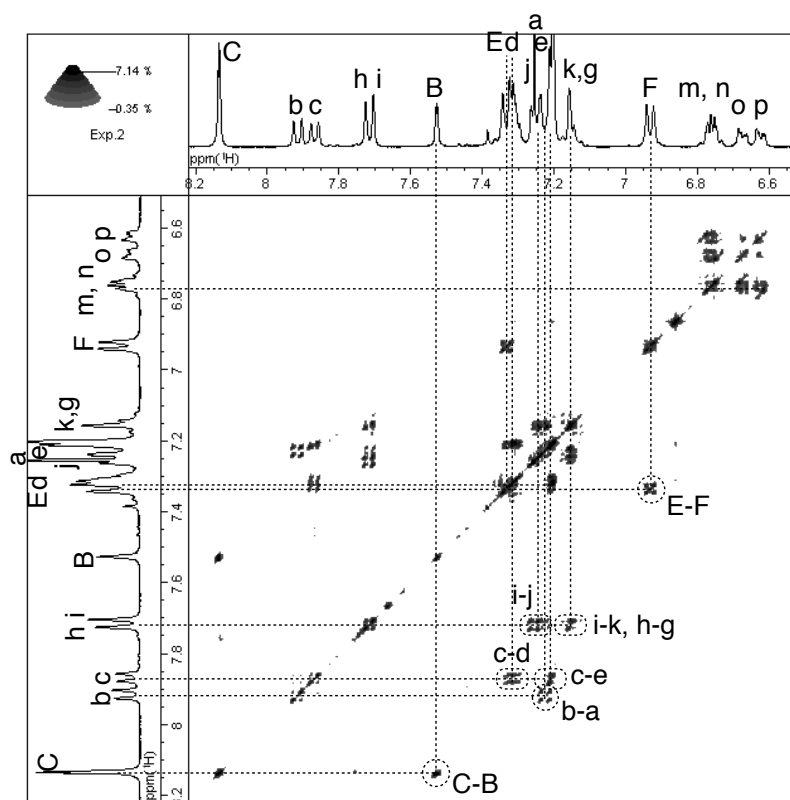


Figure S4. ^1H COSY correlations of (*R*)-**4_N** (400 MHz, CDCl_3 , 298 K)

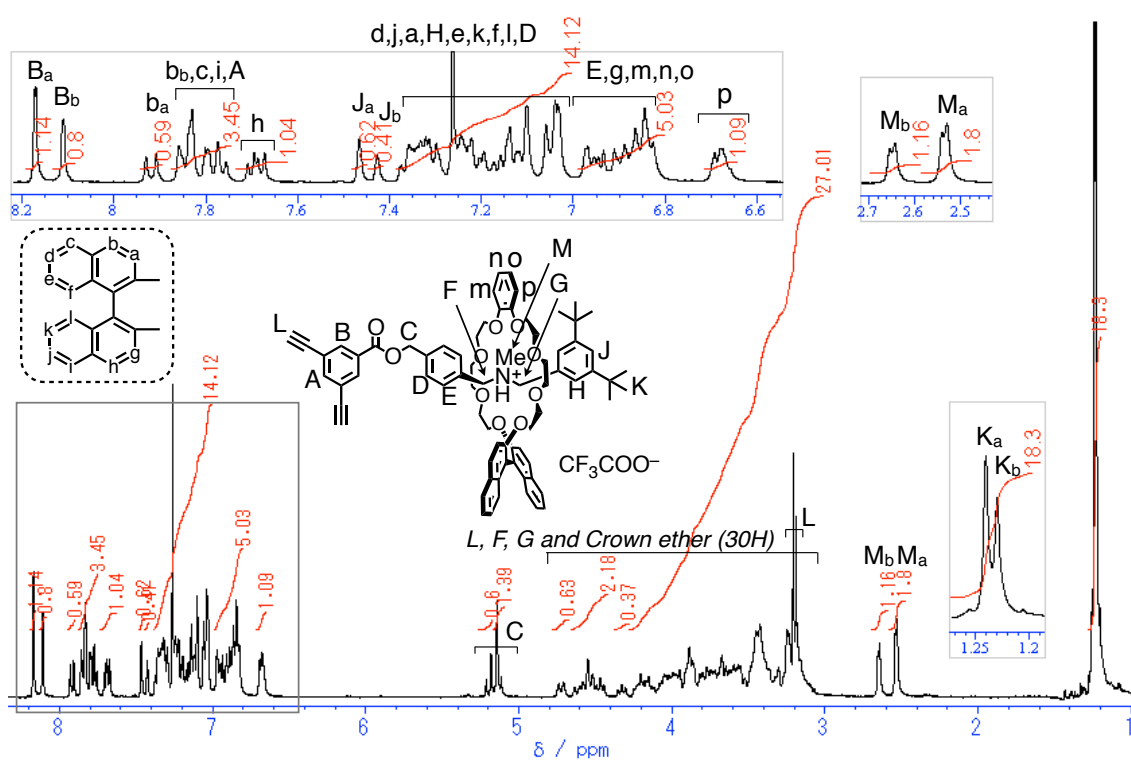


Figure S5. ^1H NMR spectrum of (*R*)-**4_S** (400 MHz, CDCl_3 , 298 K)

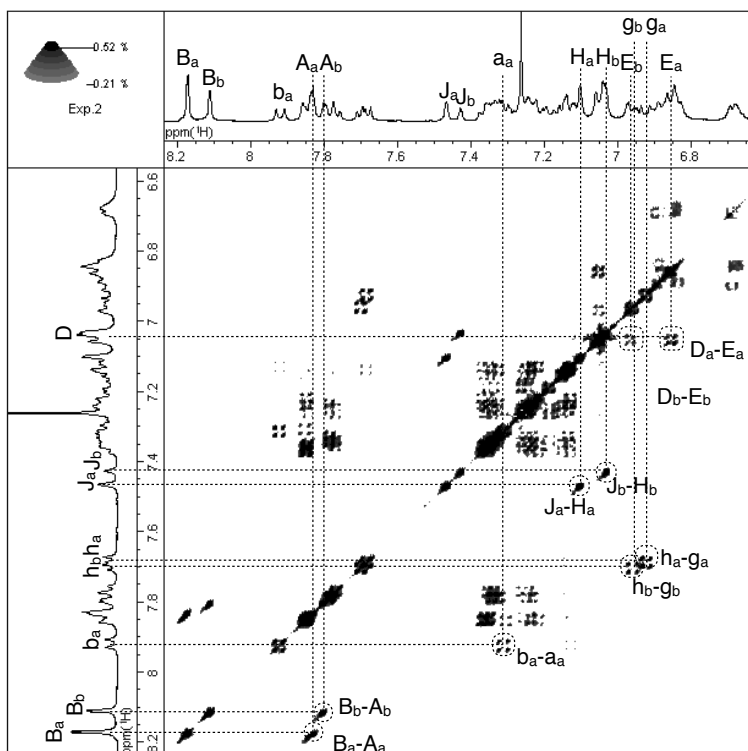


Figure S6. HH COSY correlations of **(R)-4_s** (400 MHz, CDCl₃, 298 K)

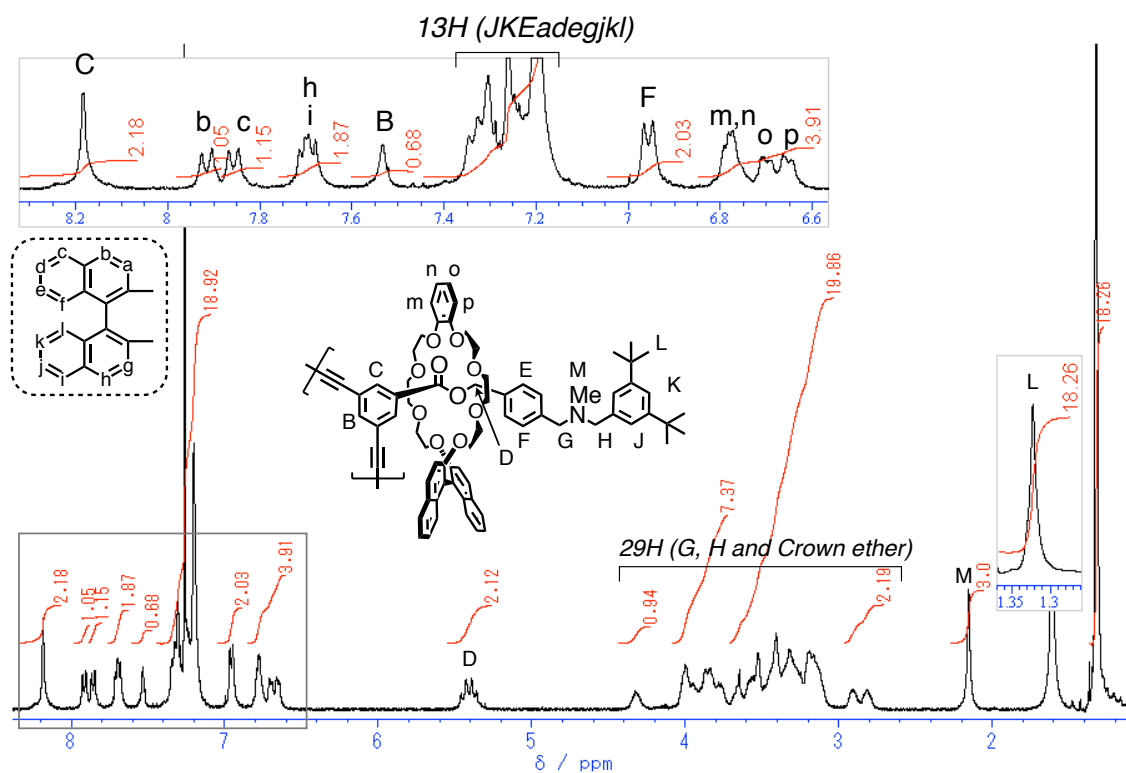


Figure S7. ¹H NMR spectrum of **poly-(R)-4_N** (400 MHz, CDCl₃, 298 K)

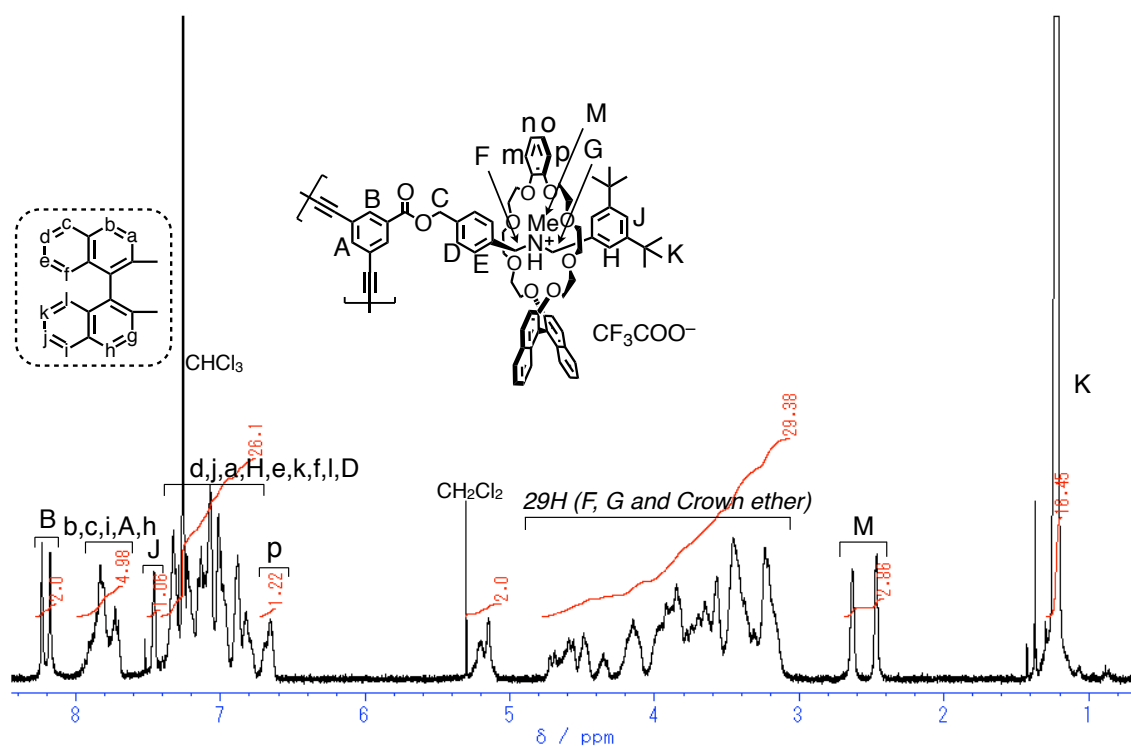


Figure S8. ^1H NMR spectrum of poly-(*R*)-4_s (400 MHz, CDCl_3 , 298 K)

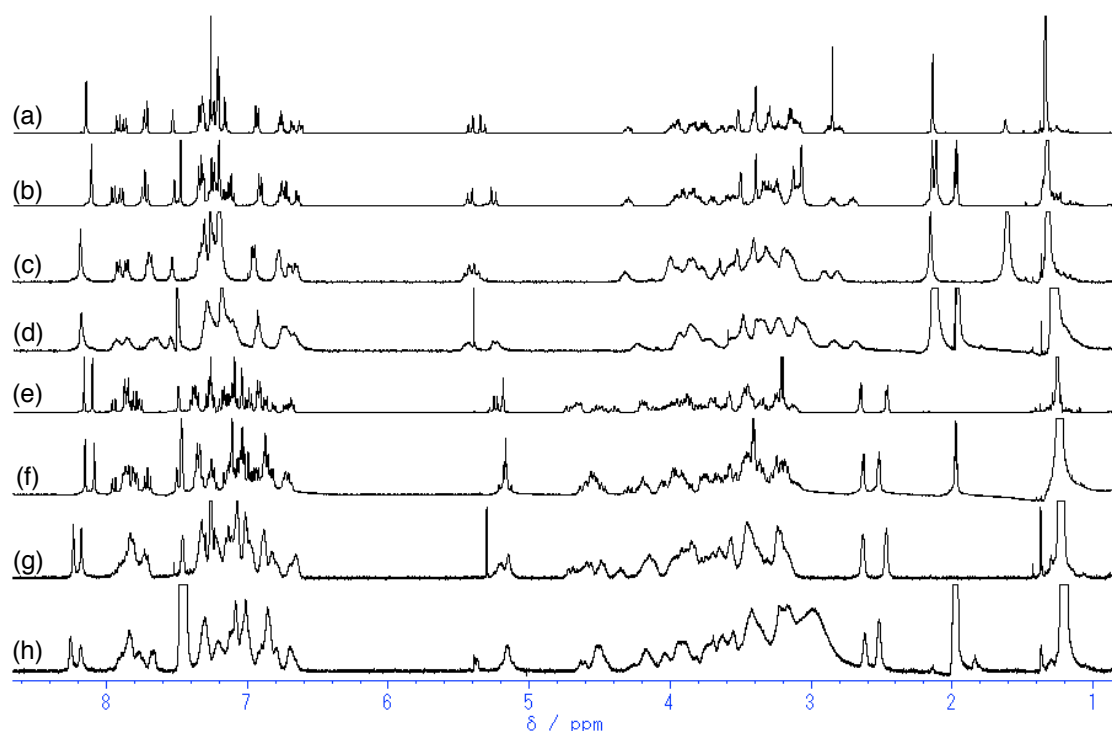


Figure S9. ¹H NMR spectra of (a) **(R)-4_N** in CDCl₃, (b) **(R)-4_N** in CDCl₃/CD₃CN = 1/1, (c) **poly-(R)-4_N** in CDCl₃, (d) **poly-(R)-4_N** in CDCl₃/CD₃CN = 1/1, (e) **(R)-4_S** in CDCl₃, (f) **(R)-4_S** in CDCl₃/CD₃CN = 1/1, (g) **poly-(R)-4_S** in CDCl₃, (h) **poly-(R)-4_S** in CDCl₃/CD₃CN = 1/1 (400 MHz, 298 K)

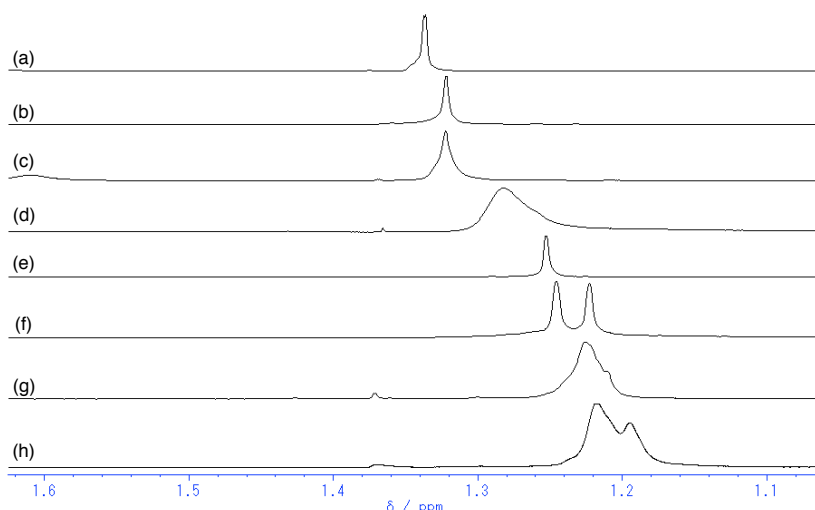


Figure S10. Partial ¹H NMR spectra of (a) **(R)-4_N** in CDCl₃, (b) **(R)-4_N** in CDCl₃/CD₃CN = 1/1, (c) **poly-(R)-4_N** in CDCl₃, (d) **poly-(R)-4_N** in CDCl₃/CD₃CN = 1/1, (e) **(R)-4_S** in CDCl₃, (f) **(R)-4_S** in CDCl₃/CD₃CN = 1/1, (g) **poly-(R)-4_S** in CDCl₃, (h) **poly-(R)-4_S** in CDCl₃/CD₃CN = 1/1 (400 MHz, 298 K)

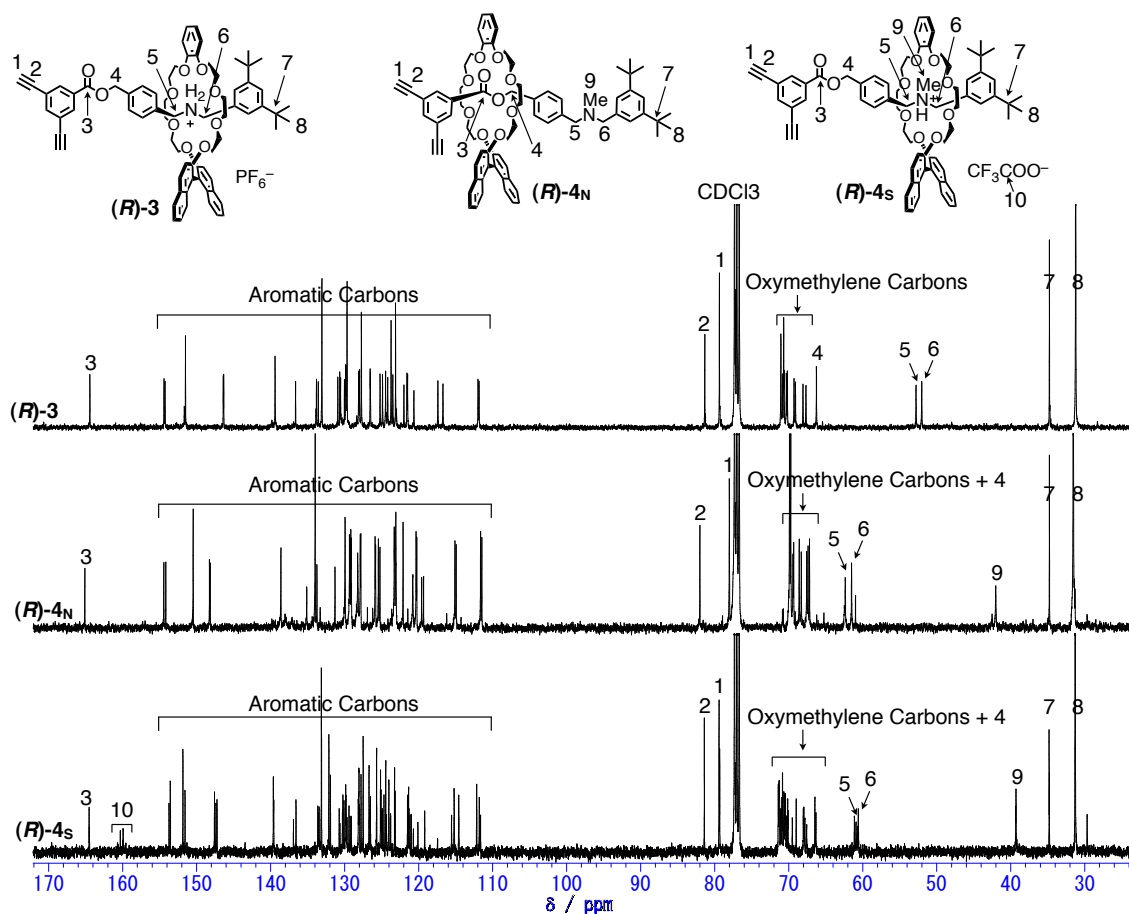


Figure S11. ^{13}C NMR spectra of (R) -3, (R) -4_N and (R) -4_S (100 MHz, CDCl_3 , 298 K)

3-2. High-resolution mass spectra

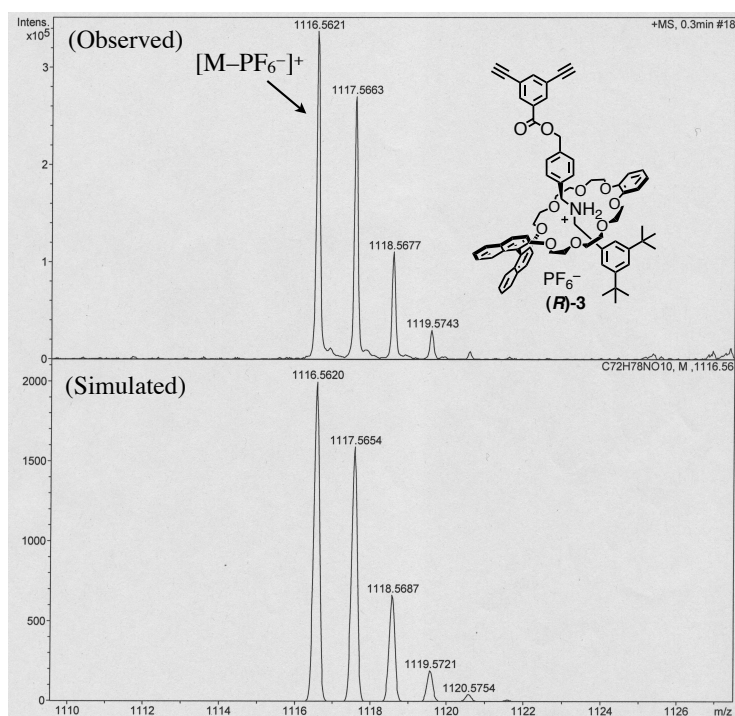


Figure S12. HR-ESI-TOF-MS spectrum of **(R)-3**

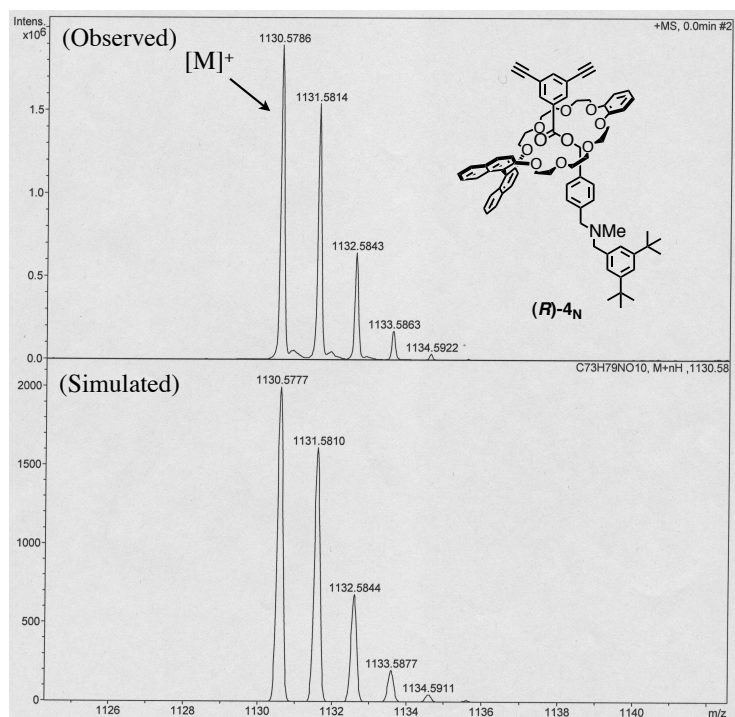


Figure S13. HR-ESI-TOF-MS spectrum of **(R)-4_N**

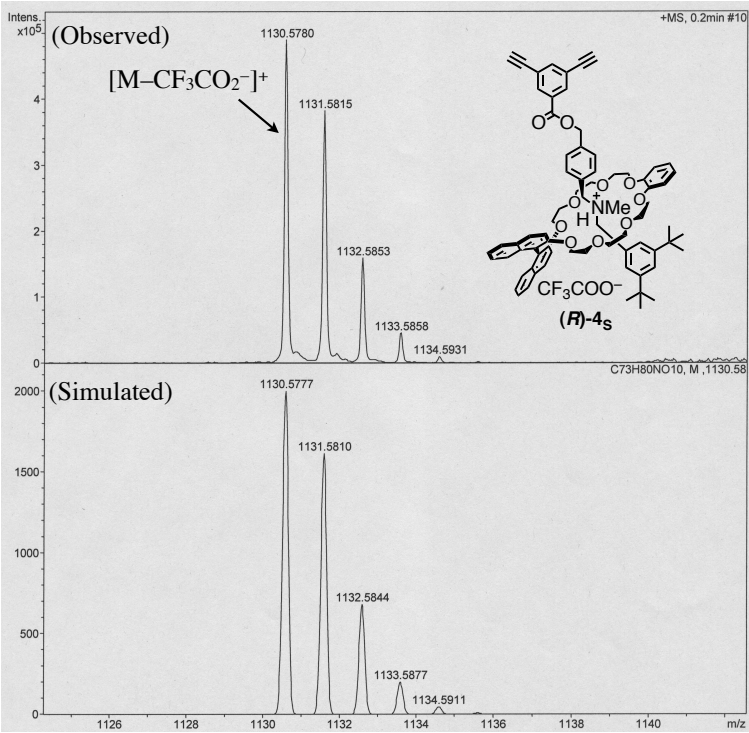


Figure S14. HR-ESI-TOF-MS spectrum of **(R)-4_s**

3-3. FT-IR spectra

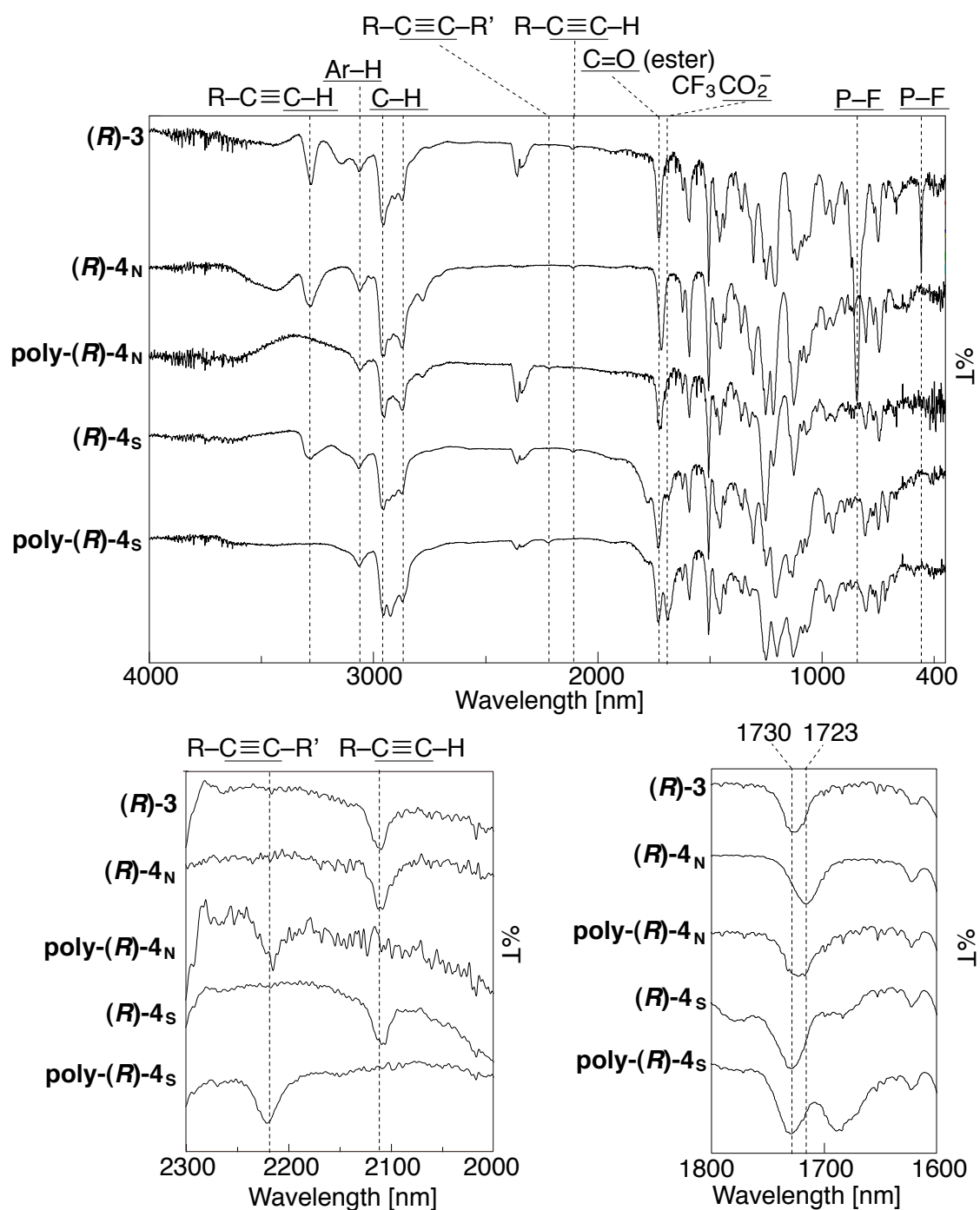


Figure S15. FT-IR spectra of (R)-3, (R)-4_N, (R)-4_S, poly-(R)-4_N and poly-(R)-4_S (KBr)

3-4. GPC charts

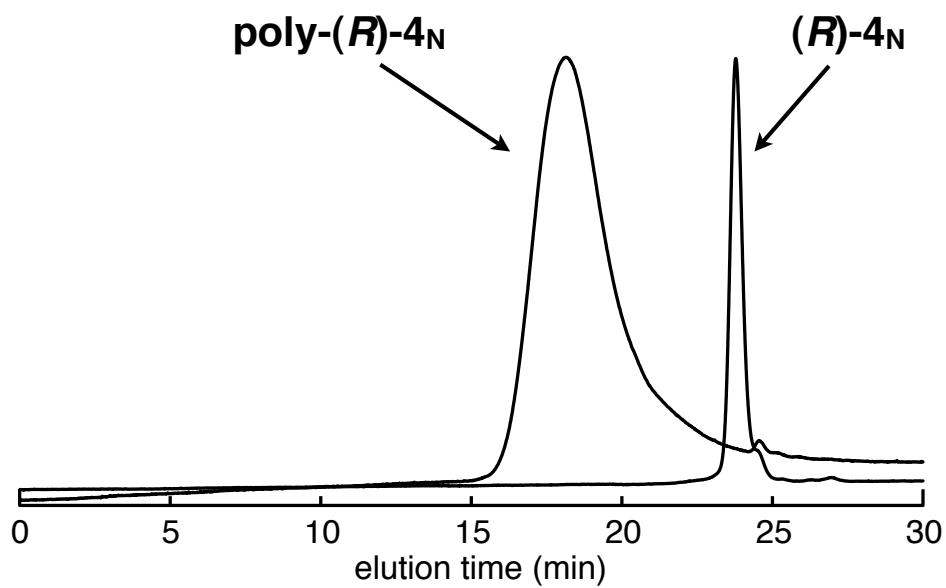


Figure S16. GPC charts of **poly-(R)-4_N** ($M_n = 29000$, $M_w = 51000$, $PDI = 1.75$) and **(R)-4_N** (DMF with 0.3% LiCl, PSt standards)

3-4. CD and UV-vis spectra

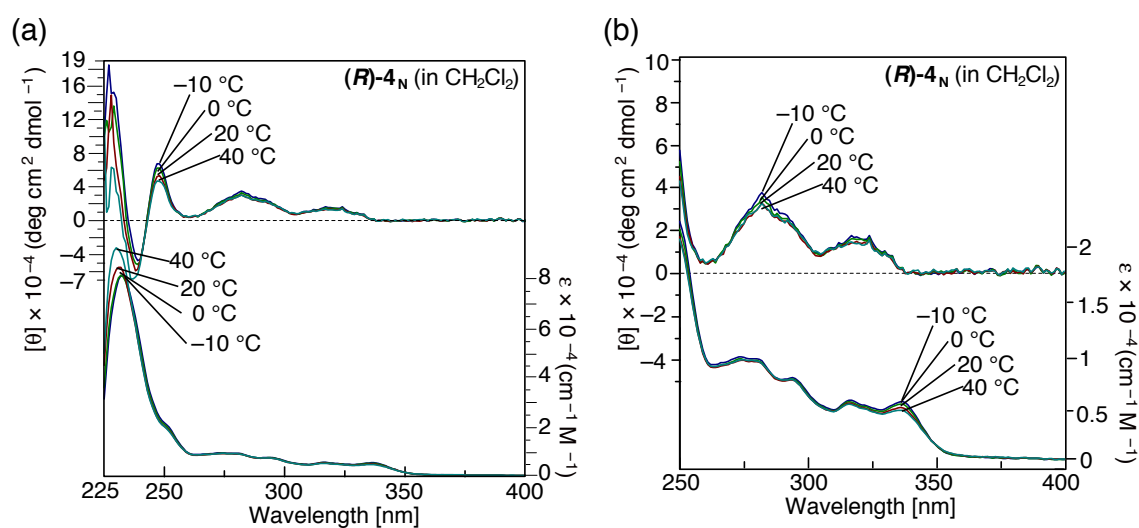


Figure S17. (a) CD and UV-vis spectra of **(R)-4_N** and (b) expanded CD and UV-vis spectra (CH_2Cl_2 , 0.10 mM, 263–313 K)

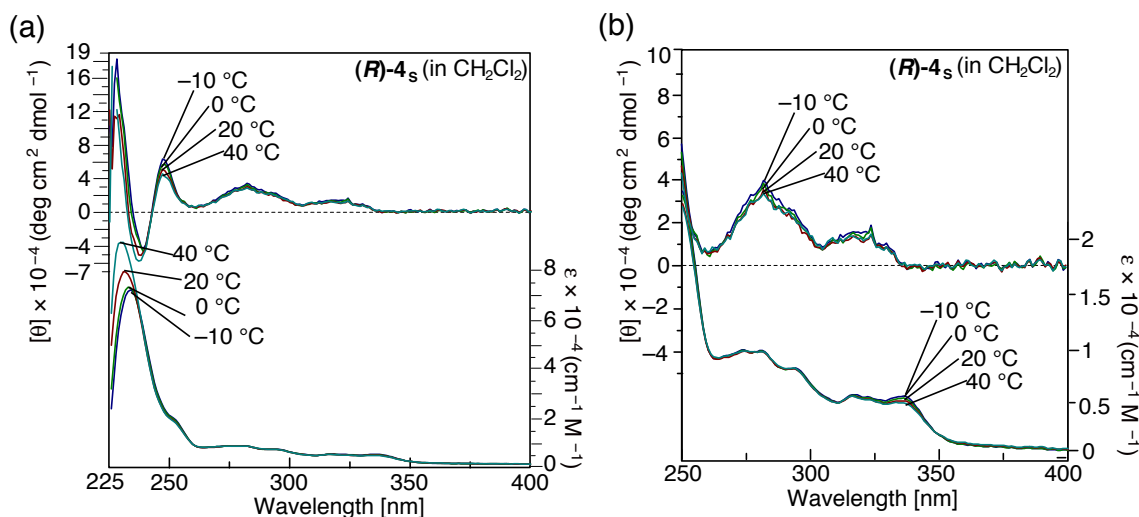


Figure S18. (a) CD and UV-vis spectra of **(R)-4_S** and (b) expanded CD and UV-vis spectra (CH_2Cl_2 , 0.10 mM, 263–313 K)

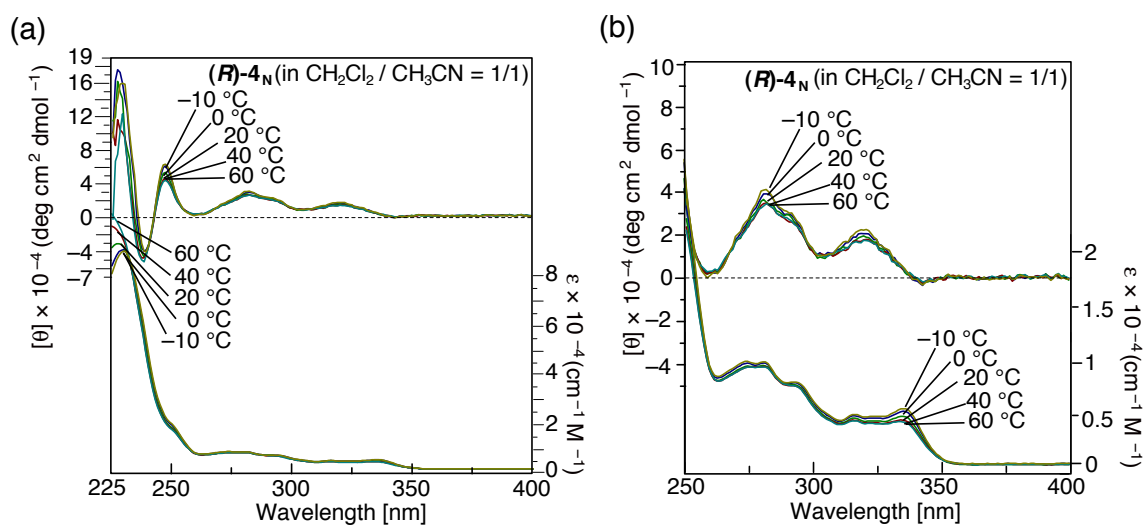


Figure S19. (a) CD and UV-vis spectra of **(R)-4_N** and (b) expanded CD and UV-vis spectra ($\text{CH}_2\text{Cl}_2/\text{CH}_3\text{CN} = 1/1$, 0.10 mM, 263–333 K)

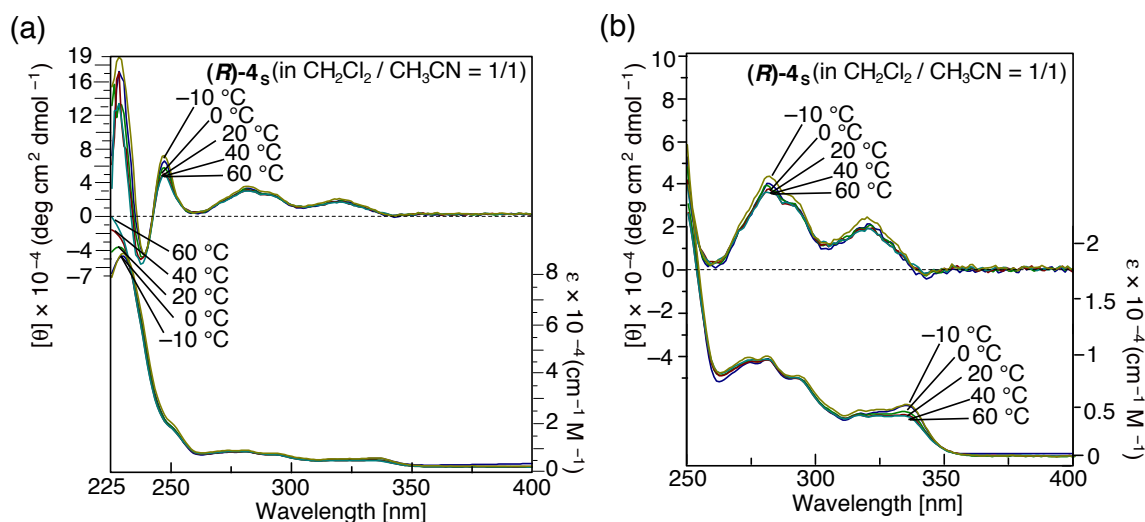


Figure S20. (a) CD and UV-vis spectra of **(R)-4_S** and (b) expanded CD and UV-vis spectra ($\text{CH}_2\text{Cl}_2/\text{CH}_3\text{CN} = 1/1$, 0.10 mM, 263–333 K)

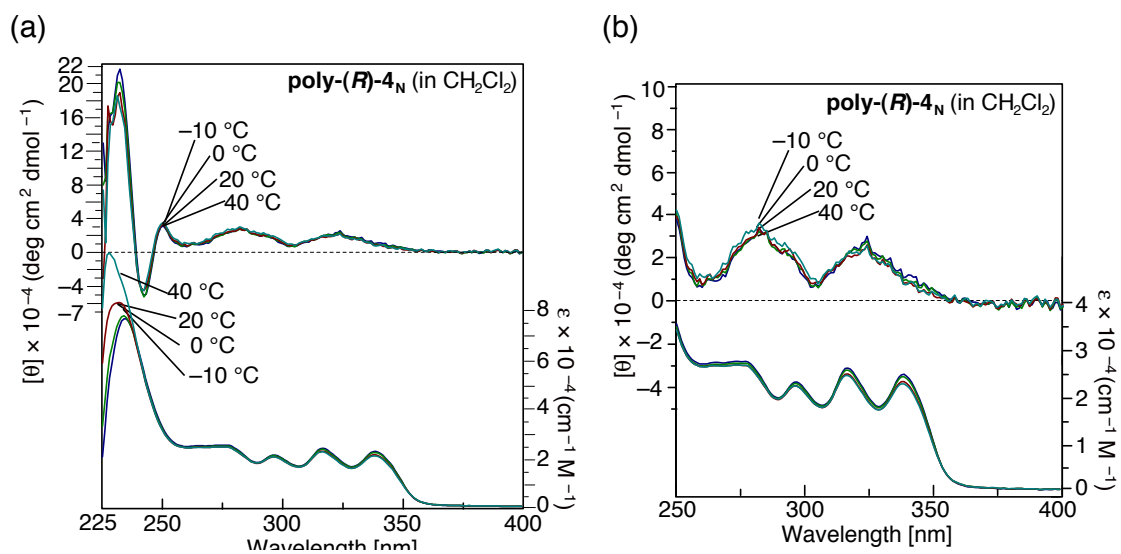


Figure S21. (a) CD and UV-vis spectra of **poly-(R)-4_N** and (b) expanded CD and UV-vis spectra (CH₂Cl₂, 0.10 mM, 263–313 K)

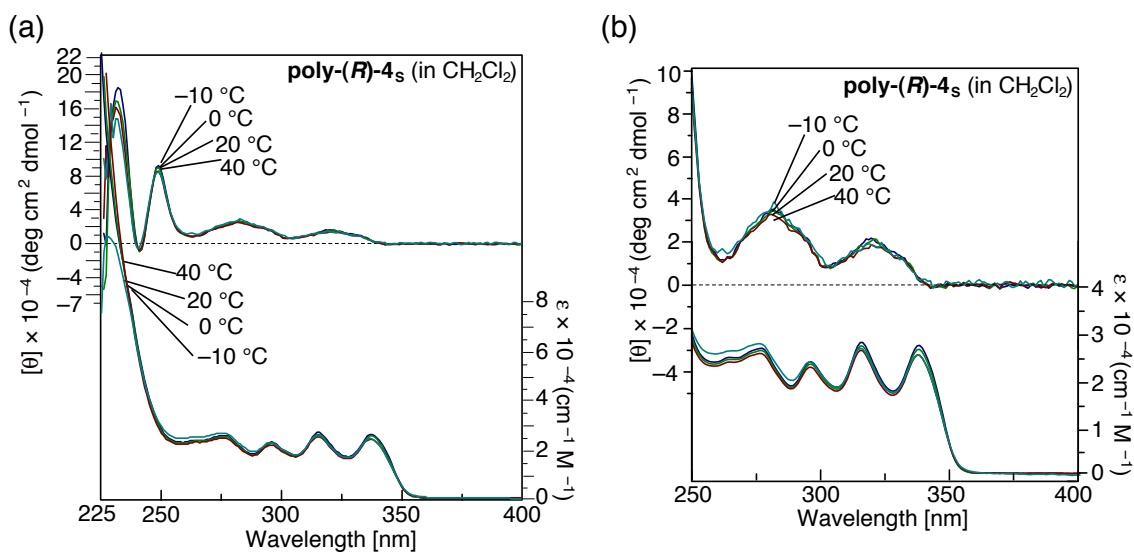


Figure S22. (a) CD and UV-vis spectra of **poly-(R)-4_S** and (b) expanded CD and UV-vis spectra (CH₂Cl₂, 0.10 mM, 263–313 K)

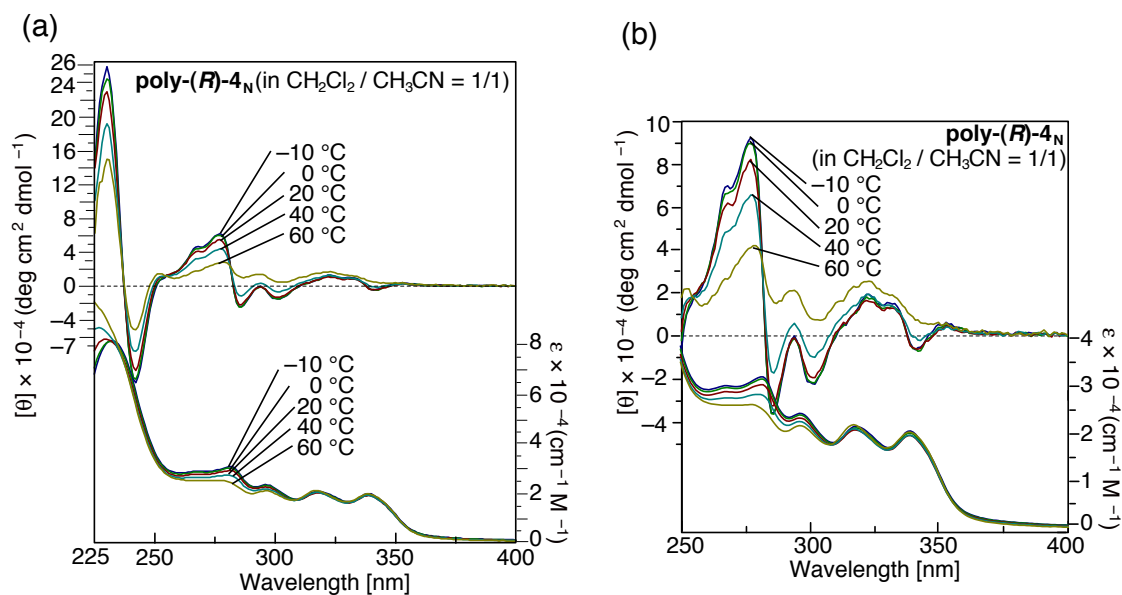


Figure S23. (a) CD and UV-vis spectra of **poly-(R)-4_N** and (b) expanded CD and UV-vis spectra (CH₂Cl₂/CH₃CN = 1/1, 0.10 mM, 263–333 K)

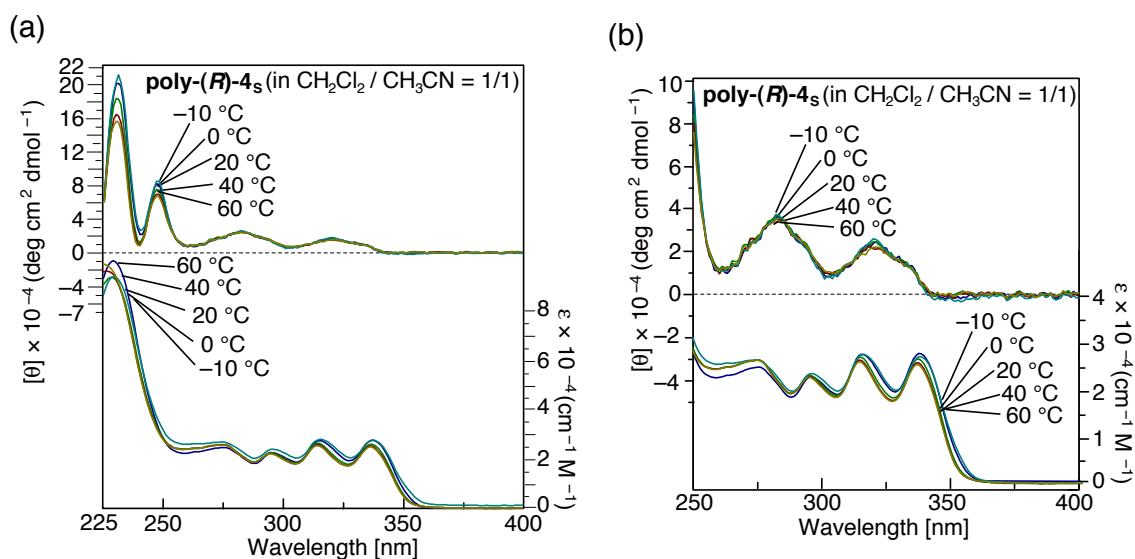


Figure S24. (a) CD and UV-vis spectra of **poly-(R)-4_S** and (b) expanded CD and UV-vis spectra (CH₂Cl₂/CH₃CN = 1/1, 0.10 mM, 263–333 K)

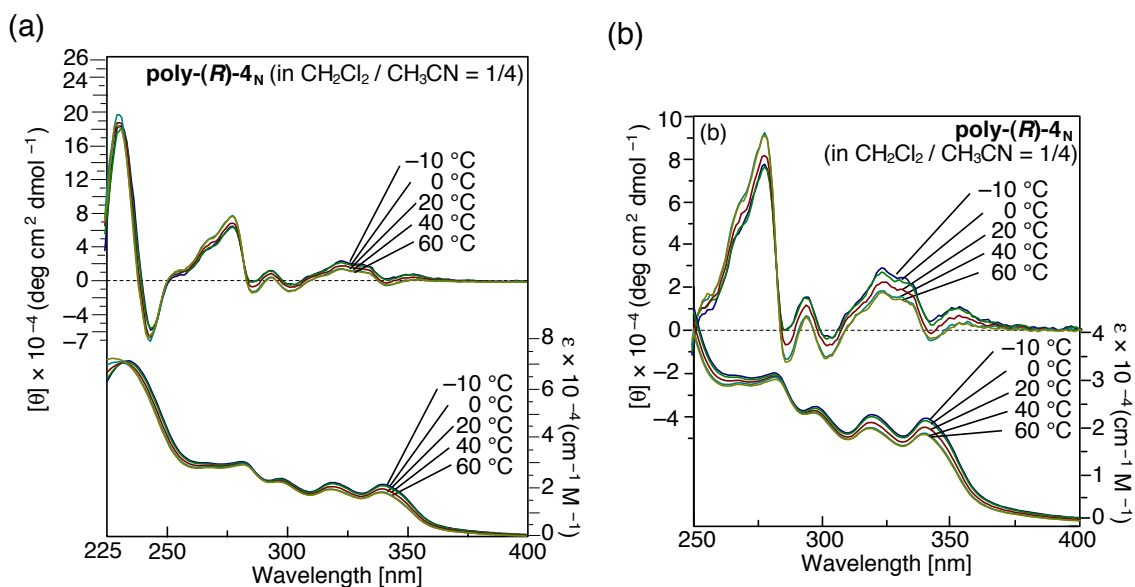


Figure S25. (a) CD and UV-vis spectra of **poly-(R)-4_N** and (b) expanded CD and UV-vis spectra ($\text{CH}_2\text{Cl}_2/\text{CH}_3\text{CN} = 1/4$, 0.10 mM, 263–333 K)

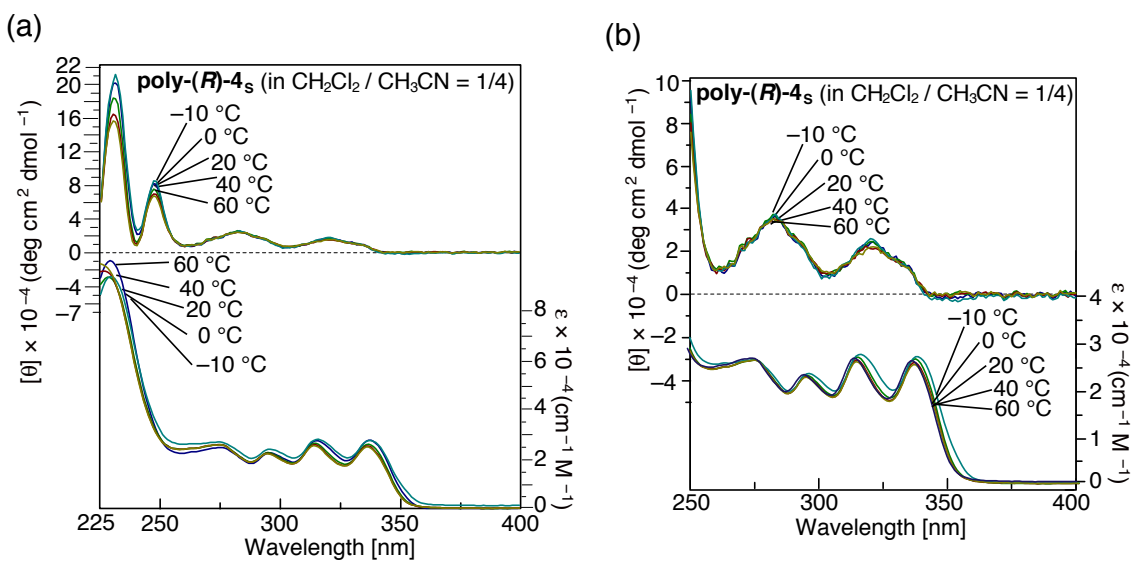


Figure S26. (a) CD and UV-vis spectra of **poly-(R)-4_S** and (b) expanded CD and UV-vis spectra ($\text{CH}_2\text{Cl}_2/\text{CH}_3\text{CN} = 1/4$, 0.10 mM, 263–333 K)

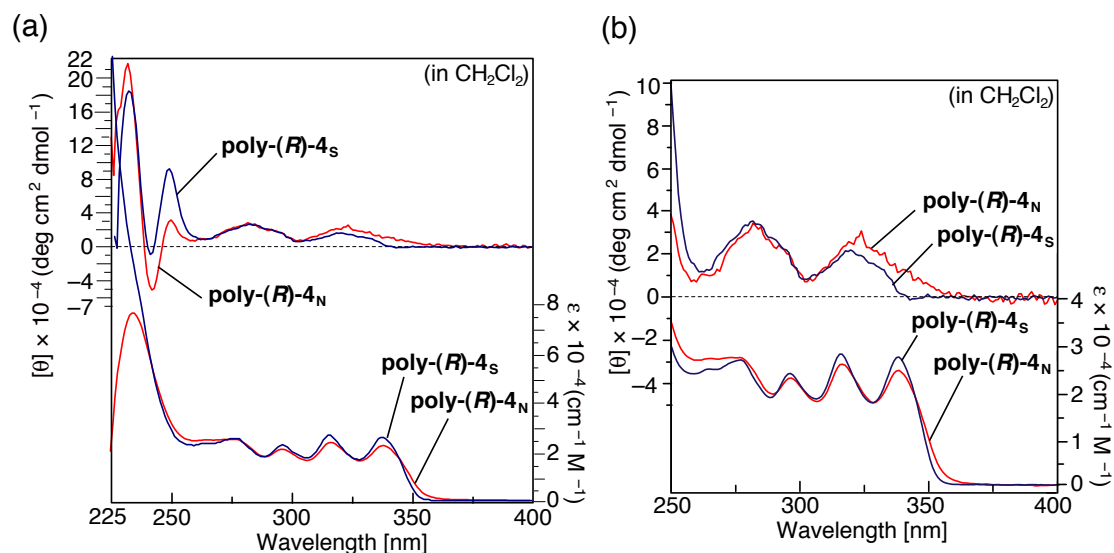


Figure S27. (a) CD and UV-vis spectra of **poly-(R)-4_s** and **poly-(R)-4_N** (b) expanded CD and UV-vis spectra (CH₂Cl₂, 0.10 mM, 293 K)

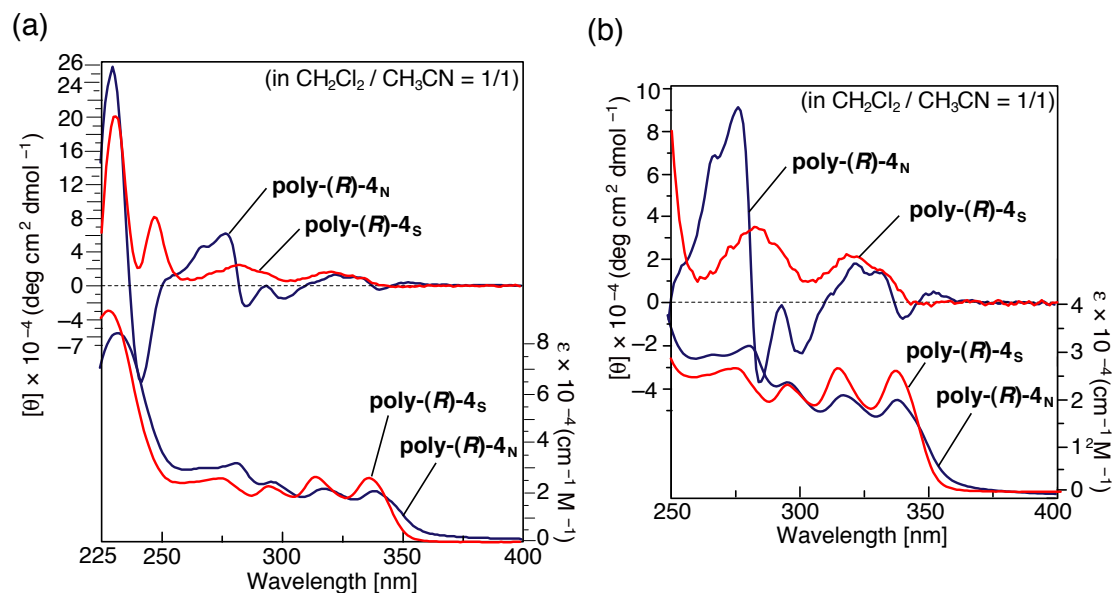


Figure S28. (a) CD and UV-vis spectra of **poly-(R)-4_s** and **poly-(R)-4_N** (b) expanded CD and UV-vis spectra (CH₂Cl₂/CH₃CN = 1/1, 0.10 mM, 293 K)

4. References

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