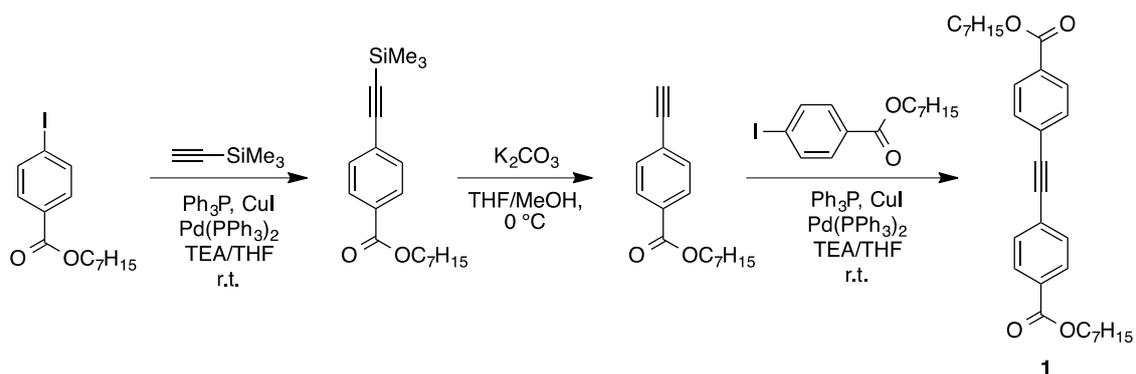


Supplementary Materials: Synthesis of Optically Active Poly(diphenylacetylene)s Using Polymer Reactions and an Evaluation of Their Chiral Recognition Abilities as Chiral Stationary Phases for HPLC

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Scheme S1. Synthesis of **1**.

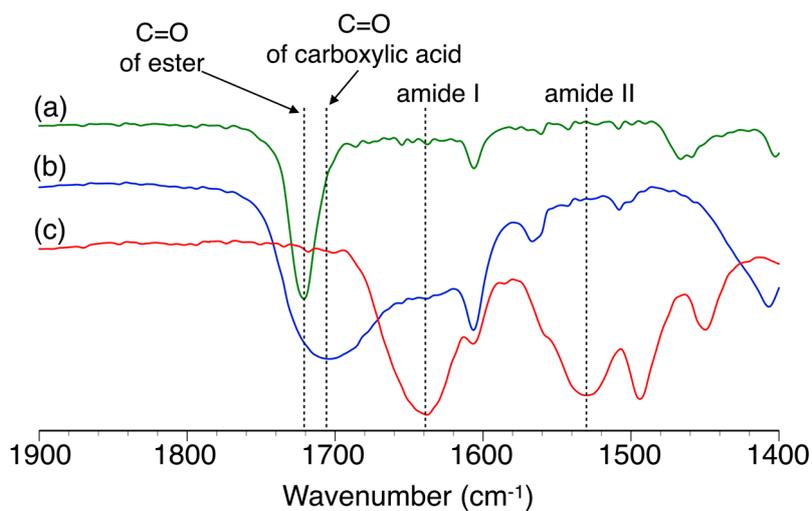


Figure S1. Infrared (IR) spectra of poly-1 (a); poly-1-H (b) and poly-2S (c) as KBr pellets.

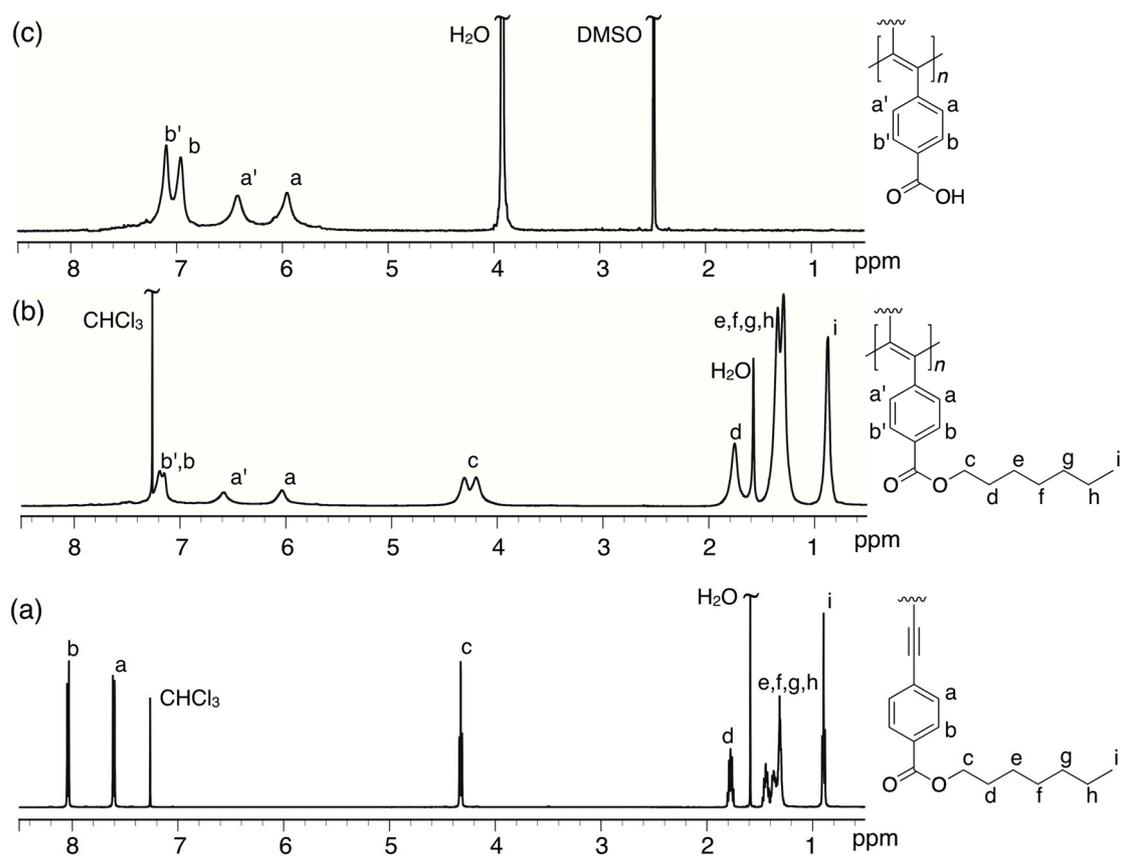


Figure S2. $^1\text{H-NMR}$ (500 MHz) spectra of **1** (a); poly-**1** (b) and poly-**1-H** (c) at r.t. These spectra were measured in CDCl_3 (a,b) and $\text{DMSO-}d_6/\text{D}_2\text{O}$ (9/1, v/v) (c).

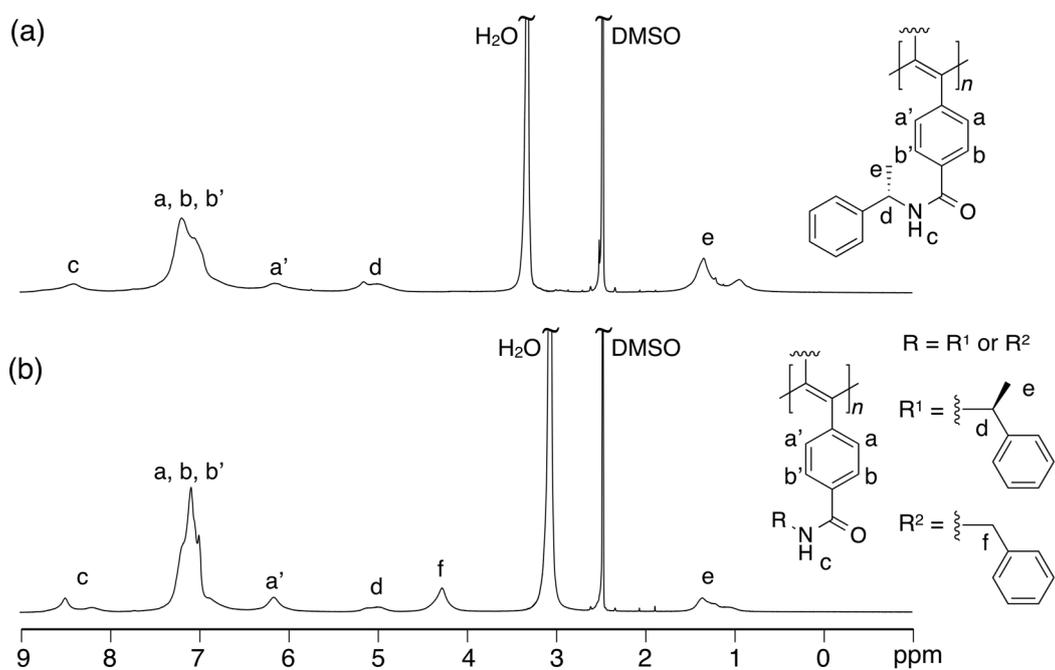


Figure S3. $^1\text{H-NMR}$ (500 MHz) spectra of poly-**2S** (a) and poly-**(2S_{0.36}-CO-30.64)** (b) in $\text{DMSO-}d_6$ at $25\text{ }^\circ\text{C}$.

Table S1. Synthesis of poly-(2S_x-co-3_{1-x})s by the polymer reaction of poly-1-H with (S)-2 followed by the reaction of the product with 3 using DMT-MM as a condensing reagent ^a.

Run	[DMT-MM]/[poly-1-H]	[(S)-2]/([(S)-2] + [3]) in Copolymer (x) ^b	Copolymer ^c
1	2.25	0.71	poly-(2S _{0.71} -co-3 _{0.29})
2	1.80	0.64	poly-(2S _{0.64} -co-3 _{0.36})
3	1.05	0.45	poly-(2S _{0.45} -co-3 _{0.55})
4	0.72	0.36	poly-(2S _{0.36} -co-3 _{0.64})
5	0.60	0.21	poly-(2S _{0.21} -co-3 _{0.79})
6	0.30	0.09	poly-(2S _{0.09} -co-3 _{0.91})
7	0.15	0.06	poly-(2S _{0.06} -co-3 _{0.94})

^a The reactions were carried out in DMSO at room temperature for 12 h. [(S)-2]/[DMT-MM] = 2, [poly-1-H] = 0.05 M (runs 1–3, 5–7) and 0.06 M (run 4). After the reaction with (S)-2, the resulting polymers were reacted with an excess of 3 in the presence of DMT-MM ([3]/[DMT-MM]/[poly-1-H] = 4/2.5/1) in DMSO at room temperature for 12 h; ^b Estimated by ¹H-NMR; ^c The subscript numbers represent the molar ratios of the corresponding units in the copolymers.

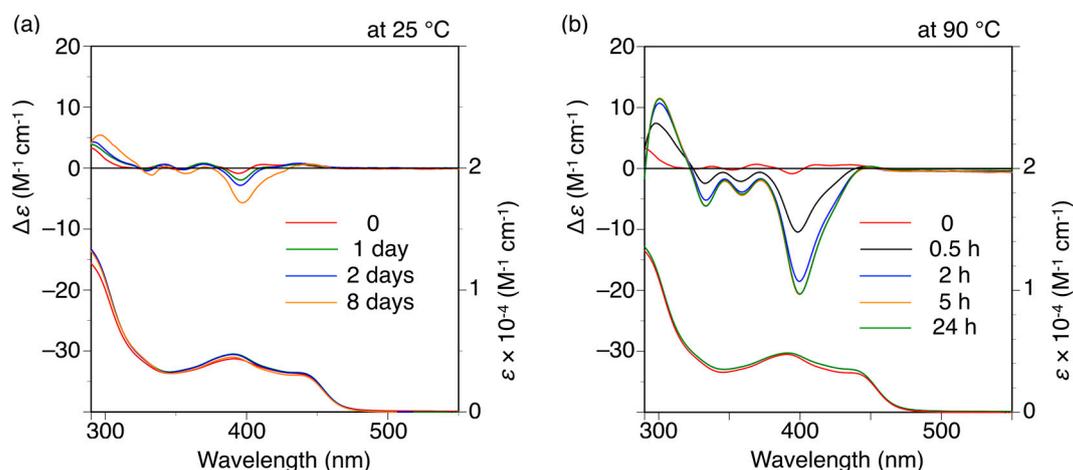


Figure S4. Time-dependent changes in the CD spectra of poly-2S at 25 °C (a) and 90 °C (b) in DMF.

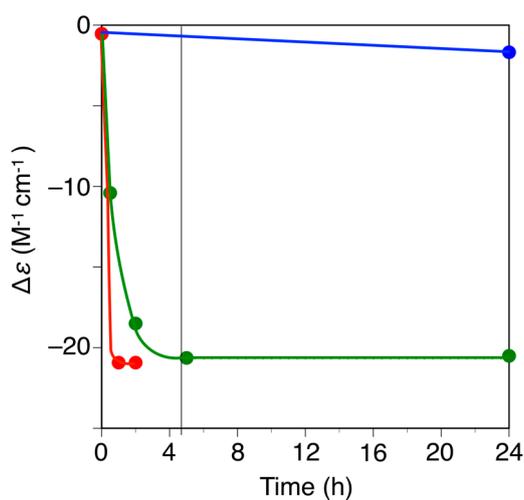


Figure S5. Plots of the CD intensity ($\Delta\epsilon_{1st}$) of poly-2S at 120 °C (red), 90 °C (green) and 25 °C (blue) in DMF with time.

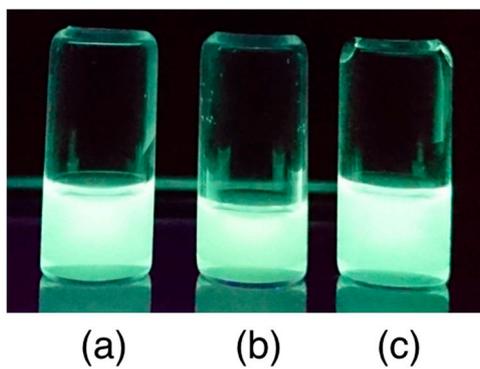


Figure S6. Photographs of *h*-poly-2S (a); *h*-poly-(2S_{0.36}-co-3_{0.64}) (b) and poly-(2S_{0.36}-co-3_{0.64}) (c) in DMF at 25 °C under irradiation of 365 nm UV light.