(Supporting Information)

# Helicity induction and memory of a lipophilic Brønsted acid-type poly(phenylacetylene) in non-polar solvents

Yuki Nishikawa, [‡,a] Daisuke Hirose,[*,‡,b] Shota Sona,[a] and Katsuhiro Maeda[*,b,c]

[a]Graduate School of Frontier Science Initiative, Kanazawa University, Kakuma-machi, Kanazawa, 920-1192, Japan.

[b]Graduate School of Natural Science and Technology, Kanazawa University, Kakuma-machi, Kanazawa, 920-1192, Japan.

[c]WPI Nano Life Science Institute (WPI-NanoLSI), Kanazawa University, Kakuma-machi, Kanazawa, 920-1192, Japan.

*To whom correspondence should be addressed. E-mail: dhirose@se.kanazawa-u.ac.jp, maeda@se.kanazawa-u.ac.jp

‡These two authors contributed equally to this work.

## Experimental Section

### 1. Materials

$[Rh(nbd)Cl]_2$, PEPPSI™-IPr catalyst, tetrabutylammonium fluoride (TBAF), and 1.0 M hydrogen chloride solution in diethyl ether were purchased from Aldrich (St. Louis, MO, USA). Citric acid, dimethylaminopyridine (DMAP), (trimethylsilyl)acetylene (TMSA), 4-iodobenzenesulfonyl chloride, bis(triphenylphosphine)palladium(II) dichloride ($PdCl_2(PPh_3)_2$), tetramethylammonium hydroxide ($Me_4NOH$, 10% in methanol), (*S*)-(–)-, (*R*)-(–)-, and *rac*-1-phenylethylamine ((*S*)-, (*R*)-, and *rac*-**2a**), (1*S*,2*R*)-(–)-1-amino-2-indanol ((1*S*,2*R*)-**2c**), (1*S*,2*S*)-(–)-1,2-diphenylethylenediamine ((1*S*,2*S*)-**2d**), and 1,4-dinitrobenzene were obtained from Tokyo Chemical Industry (TCI, Tokyo, Japan). Potassium carbonate, (*S*)-(–)-1-phenylethanol ((*S*)-**2e**), and *n*-dodecylbenzenesulfonic acid (DBSA) were obtained from Kanto Kagaku (Tokyo, Japan). Bromine, 25% ammonia aq., copper(I) iodide (CuI), triethylamine, and phosphoryl chloride were purchased from FUJIFILM Wako Pure Chemical (Osaka, Japan). 1,2-Dimethoxyethane (DME) was purchased from Kishida Chemical Co., Ltd. (Osaka, Japan). (4-(Octyloxy)phenyl)boronic acid, L-valine ((*S*)-**2b**), and benzylamine (BA) were obtained from BLD pharmatech (Shanghai, China), Peptide Institute, Inc. (Osaka, Japan), and Nacalai Tesque (Kyoto, Japan), respectively. Anhydrous tetrahydrofuran (THF), chloroform ($CHCl_3$), dichloromethane, toluene, benzene, chlorobenzene, dimethyl sulfoxide (DMSO), methanol, 1-butanol, *n*-hexane, and acetonitrile were purchased from Kanto Kagaku and FUJIFILM Wako Pure Chemical. These solvents were stored under nitrogen. 5,5'-Dibromo-2,2'-dihydroxybiphenyl[1] and 4-ethynylbenzenesulfonamide[2, 3] were prepared according to the previous reports. Poly((4-carboxyphenyl)acetylene) (PCPA) was prepared by polymerization of 4-ethynylbenzoic acid according to the reported method[4]. The number-average molar mass ($M_n$) and molar-mass dispersity ($M_w/M_n$) were $3.7 \times 10^4$ and 3.1, respectively, determined by size exclusion chromatography (SEC) with polystyrene standards using $CHCl_3$ as the eluent as its methyl ester.

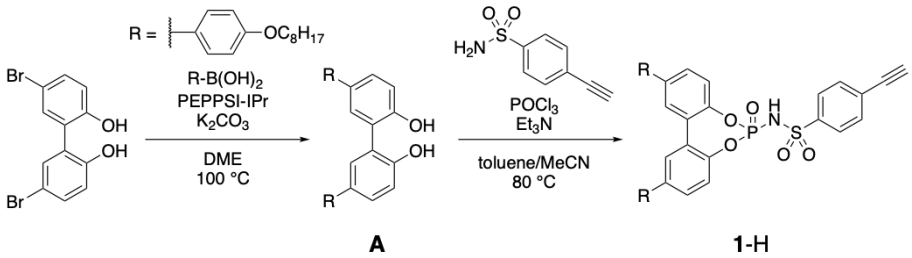
### 2. Instruments

NMR spectra were taken on a JNM-ECZ 500 and -ECA 500 (JEOL, Tokyo, Japan) (500 MHz for $^1H$, 125 MHz for $^{13}C$, and 202 MHz for $^{31}P$) spectrometer in $CDCl_3$ or acetonitrile-$d_3$ using TMS (for $CDCl_3$, $^1H$ and $^{13}C$) or a solvent residual peak (for acetonitrile-$d_3$, $^1H$ and $^{13}C$) as the internal standards. $H_3PO_4$ (for acetonitrile-$d_3$) was used as the external standard for $^{31}P$ NMR measurements. IR and Raman spectra were recorded with a JASCO (Hachioji, Japan) Fourier Transform IR-4700 spectrophotometer and Thermo Fisher Scientific DXR2 Raman confocal microscope, respectively.

Absorption and CD spectra were measured in a 1.0-mm quartz cell (GL Sciences, Tokyo, Japan) on a JASCO V-750 spectrophotometer and a JASCO J-1500 spectropolarimeter, respectively. The temperature was controlled with a JASCO ETCS-761 (for absorption spectral measurements) and a JASCO PTC-510 apparatus (for CD spectral measurements). SEC measurements were performed with a JASCO PU-4180 liquid chromatograph equipped with a UV-vis (JASCO MD-4010) detector at 40 °C. The temperature was controlled with a JASCO CO-4060 column oven. A Shodex (Tokyo, Japan) KF-805L GPC column was used for the SEC measurements using THF as the eluent at flow rate of 1.0 mL·min$^{-1}$. The molecular weight calibration curves were obtained with polystyrene standards (Tosoh). Recycling preparative SEC was conducted on a JAI (Tokyo, Japan) LaboACE LC-7080 equipped with JAIGEL-1HR and JAIGEL-2HR columns using $CHCl_3$ as the eluent at flow rate of 10 mL·min$^{-1}$. The melting points were measured on a M-565 melting point apparatus (BUCHI Labortechnik, Flawil, Switzerland) and were uncorrected. High-resolution mass spectra were measured on a JEOL JMS-700. Elemental analyses were performed by the Research Institute for Instrumental Analysis of Advanced Science Research Center, Kanazawa University, Kanazawa, Japan.

## 3. Synthesis

A phenylacetylene monomer bearing a sulfonylphosphoramidic acid residue (**1**-H) was synthesized according to Scheme S1.




**Scheme S1**. Synthesis of **1**-H.

Synthesis of **A**

In a pressure tube, a mixture of 5,5'-dibromo-2,2'-dihydroxybiphenyl[1] (1.26 g, 3.67 mmol, 1.0 eq.), (4-(octyloxy)phenyl)boronic acid (2.75 g, 11.0 mmol, 3.0 eq.), potassium carbonate (3.04 g, 22.0 mmol, 6.0 eq.), and PEPPSI$^{TM}$-IPr catalyst (74.8 mg, 0.110 mmol, 0.03 eq.) in DME (18.4 mL) was stirred for 16 h at 100 °C under $N_2$ atmosphere. After evaporation, 100 mL of 17.4 wt% citric acid

(21.1 g, 110 mmol) aq. was added, and the solution was extracted with ethyl acetate three times. The combined organic layer was washed with brine and dried by $Na_2SO_4$. After filtration, the solvent was removed under reduced pressure. The crude product was purified by silica gel chromatography (*n*-hexane/ethyl acetate = 3/1, v/v) and recycling preparative SEC ($CHCl_3$) to afford **A** as a white solid (799 mg, 1.34 mmol, 37%). IR (KBr, $cm^{-1}$): $\upsilon$ 1245. $^1H$ NMR (500 MHz, $CDCl_3$, r.t.): $\delta$ 7.54–7.48 (m, 8H), 7.10 (d, $J$ = 8.0 Hz, 2H), 6.95 (d, $J$ = 8.6 Hz, 4H), 5.52 (s, 2H), 3.98 (t, $J$ = 6.6 Hz, 4H), 1.80 (tt, $J$ = 7.0, 7.0 Hz, 4H), 1.46 (tt, $J$ = 7.0, 7.0 Hz, 4H), 1.38–1.25 (m, 16H), 0.89 (t, $J$ = 6.9 Hz, 6H). $^{13}C$ NMR (125 MHz, $CDCl_3$, r.t.): $\delta$ 158.5, 151.9, 134.6, 132.6, 129.5, 128.2, 127.7, 123.9, 117.1, 114.8, 68.1, 31.8, 29.4, 29.3, 29.2, 26.0, 22.7, 14.1. HRMS (FAB+) (m/z): calcd for $C_{40}H_{50}O_4$ ($[M+H]^+$): 595.3782, found: 595.3797. M.p.: 139.1‑140.7 °C

Synthesis of **1**-H

In a pressure tube, a mixture of **A** (297 mg, 0.500 mmol, 1.0 eq.), DMAP (61.1 mg, 0.500 mmol, 1.0 eq.), and triethylamine (485 μL, 3.50 mmol, 7.0 eq.) in dry toluene (2.5 mL) was kept at 0 °C under $N_2$ atmosphere, and then $POCl_3$ (54.8 μL, 0.600 mmol, 1.2 eq.) was added. After the solution was stirred at room temperature for 1 h, dry acetonitrile (5.0 mL) and 4-ethynylbenzenesulfonamide[2, 3] (109 mg, 0.600 mmol, 1.2 eq.) were added and the mixture was stirred for 17 h at 80 °C. After evaporation, 1N HCl aq. was added, and the solution was extracted with ethyl acetate four times. The combined organic layer was washed with 1N HCl aq. and brine, and dried by $Na_2SO_4$. After filtration, the solvent was removed by evaporation. The crude product was purified by preparative TLC (dichloromethane/methanol = 10/1, v/v) to afford **1**-H as a white solid (175 mg, 0.214 mmol, 43%). IR (KBr, $cm^{-1}$): $\upsilon$ 3445, 1174. $^1H$ NMR (500 MHz, acetonitrile-$d_3$, r.t.): $\delta$ 7.89 (d, $J$ = 8.6 Hz, 2H), 7.84 (d, $J$ = 2.3 Hz, 2H), 7.64–7.55 (m, 8H), 7.08 (dd, $J$ = 8.6, 1.1 Hz, 2H), 6.98 (d, $J$ = 6.9 Hz, 4H), 4.01 (t, $J$ = 6.6 Hz, 4H), 3.50 (s, 1H), 1.76 (tt, $J$ = 7.0, 7.0 Hz, 4H), 1.45 (tt, $J$ = 7.0, 7.0 Hz, 4H), 1.40–1.25 (m, 16H), 0.89 (t, $J$ = 6.9 Hz, 6H). $^{13}C$ NMR (125 MHz, acetonitrile-$d_3$, r.t.): $\delta$ 159.9, 149.1, 149.1, 139.0, 133.1, 132.9, 130.5, 129.2, 128.64, 128.56, 126.9, 124.8, 123.0, 115.7, 83.4, 80.5, 68.9, 32.6, 30.05, 29.99, 29.95, 26.7, 23.4, 14.4. $^{31}P$ NMR (202 MHz, acetonitrile-$d_3$, r.t.): $\delta$ 5.50. HRMS (FAB–) (m/z): calcd for $C_{48}H_{54}NO_7PS$ ($[M–H]^–$): 818.3286, found: 818.3295. M.p.: 188.4–189.6 °C

**4. Polymerization**

Polymerization of **1**-H was carried out according to Figure 1 in a dry glass ampule under a dry nitrogen atmosphere using $[Rh(nbd)Cl]_2$ as a catalyst in a similar way as reported previously.[5]

A mixture of **1**-H (33.5 mg, 40.9 μmol, 1.0 eq.) and $Me_4NOH$ (10% in methanol) (133 μL, 123 μmol, 3.0 eq.) in THF (200 μL) was stirred at room temperature for 30 min. After evaporating the solvent, the residue was dissolved in water, which was extracted with dichloromethane. The combined organic layer was washed with water and the solvent was removed in vacuo to afford the $Me_4N$ salt as a pale yellow solid (31.9 mg, 35.7 μmol, 87%), which was employed for the polymerization without further purification. A mixture of the $Me_4N$ salt of **1**-H (31.4 mg, 35.2 μmol, 1.0 eq.) and triethylamine (14.6 μL, 106 μmol, 3.0 eq.) in dry THF (105 μL) and dry methanol (20 μL) was placed in a vial tube under $N_2$ atmosphere. To this was added a solution of $[Rh(nbd)Cl]_2$ in THF at 30 °C, and the reaction mixture was mixed with a vibrator (IKA VORTEX GENIUS3, Japan). The concentration of the monomer and the rhodium catalyst were 0.28 and 0.005 M, respectively. After 5 h, DBSA (230 mg, 704 μmol) in dichloromethane (200 μL) was added to dissolve the precipitate, and the solution was poured into a large amount of methanol. The precipitated polymer was collected by centrifugation and further purified by reprecipitation from dichloromethane to methanol. The precipitated polymer was collected by centrifugation and dried in vacuo at room temperature to afford poly-**1**-H (27.9 mg, 34.0 μmol, 97%) as a yellow solid. The number-average molar mass ($M_n$) and molar-mass dispersity ($M_w/M_n$) of poly-**1**-H were determined to be $7.0 \times 10^4$ and 3.7, respectively, by SEC using THF as the eluent. IR (KBr, $cm^{-1}$): $\upsilon$ 3444, 1244. $^1H$ NMR (500 MHz, $CDCl_3$, r.t.): $\delta$ 8.65–5.82 (br, aromatic), 4.28–3.46 (br, OC$H_2$), 1.97–0.46 (br, aliphatic). Elemental analysis: Calcd. for $(C_{48}H_{54}NO_7PS \cdot 1.5H_2O)_n$: C, 68.07; H, 6.78; N, 1.65. Found: C, 67.87; H, 6.57; N, 1.81.

## 5. Helicity Induction in Poly-1-H with Chiral Guests

A general experimental procedure is described below. The concentration of poly-**1**-H was calculated based on the monomer units and was 1.0 mM (monomer units) unless otherwise stated. Stock solutions of poly-**1**-H (2.0 mM) and chiral guests (1.0 mM) in $CHCl_3$ were prepared in vessels equipped with a screwcap. Typically, a 150 μL (0.3 μmol) aliquot of the stock solution of poly-**1**-H was transferred to a 2-mL vessel with a screwcap. After the removal of the solvent by evaporation, the residual was dried in vacuo thoroughly. To this was added 300 μL of the stock solution of chiral guests ([**2**]/[poly-**1**-H] = 1.0). After the solution was immediately mixed with a vibrator, the absorption and CD spectra were taken at 25 °C in a 1-mm quartz cell at the appropriate time intervals until no further increase in the CD intensity was observed.

**6. CD Titrations** (Figure 2C)

Stock solutions of poly-**1**-H (2.0 mM) and (*S*)-**2a** (0.1, 0.2, 0.4, 0.5, 0.6, 0.8, 1.0, 1.5, and 3.0 mM) in $CHCl_3$ were prepared in vessels equipped with a screwcap. A 150 μL (0.3 μmol) aliquot of the stock solution of poly-**1**-H was transferred to a 2-mL vessel with a screwcap. After the removal of the solvent by evaporation, the residual was dried in vacuo thoroughly. To each vessel was added 300 μL of the stock solution of (*S*)-**2a**; the molar ratios of (*S*)-**2a** to poly-**1**-H were 0.1, 0.2, 0.4, 0.5, 0.6, 0.8, 1.0, 1.5, and 3.0. After the solutions were immediately mixed with a vibrator, the absorption and CD spectra were taken at 25 °C in a 1-mm quartz cell at the appropriate time intervals until no further increase in the CD intensity was observed.

**7. Nonlinear Effects** (Figure 2D)

The nonlinear effects between ICD intensities of poly-**1**-H and percent ee of **2a** in the complexation with poly-**1**-H were investigated in $CHCl_3$. Stock solutions of poly-**1**-H (2.0 mM), (*S*)-**2a** (10 mM), and *rac*-**2a** (10 mM) in $CHCl_3$ were prepared. Aliquots of the stock solutions of (*S*)- and *rac*-**2a** were placed into four 2-mL vessels so that the percent ee of the mixtures (*S*-rich) was 20, 40, 60, and 80, respectively. A 150 μL (0.3 μmol) aliquot of the stock solution of poly-**1**-H was transferred to a 2-mL vessel with a screwcap. After the removal of the solvent by evaporation, the residual was dried in vacuo thoroughly. To each vessel of poly-**1**-H (0.3 μmol) was added 300 μL of each stock solution of **2a** (*S*-rich) with different ee values ([**2a**]/[poly-**1**-H] = 10) to keep the polymer concentrations at 1.0 mM. After the solutions were immediately mixed with a vibrator, the absorption and CD spectra were taken at 25 °C in a 1-mm quartz cell at the appropriate time intervals until no further increase in the CD intensity was observed.

**8. Dynamic Helicity Memory and Its Stability**

After helicity induction in poly-**1**-H (0.3 μmol) with (*S*)-**2a** ([(*S*)-**2a**]/[poly-**1**-H] = 1) in $CHCl_3$ (300 μL, 1 mM) at 25 °C for 24 h in a 2-mL vessel with a screwcap, $CHCl_3$ was evaporated at 25 °C. The residual was dissolved in toluene (150 μL) to give the *h*-poly-**1**–(*S*)-**2a** complex in toluene (2.0 mM). To this was added a solution of benzylamine (BA) in toluene (150 μL, 100 mM) ([BA]/[(*S*)-**2a**]/[poly-**1**-H] = 50/1/1) to convert the *h*-poly-**1**–(*S*)-**2a** to the *h*-poly-**1**–BA complex in situ. The absorption and CD spectra of the resulting *h*-poly-**1**–BA complex in toluene (1.0 mM) were then recorded at 25 °C (Figure 3A, (ii)). Time-dependent CD intensity changes ($\Delta\varepsilon_{2nd}$) were measured at –10 °C and 25 °C to investigate the stability of the dynamic helicity memory of *h*-poly-**1**–BA in

toluene (Figure 3B, blue and red circle). As for the stability of the dynamic helicity memory of *h*-poly-**1**–BA in $CHCl_3$ (Figure 3B, green circle), after helicity induction in poly-**1**-H with (*S*)-**2a** in $CHCl_3$ (300 μL, 1 mM) at 25 °C for 24 h, $CHCl_3$ was evaporated at 25 °C. The residual was dissolved in $CHCl_3$ (150 μL) and 150 μL of a solution of benzylamine (BA) (100 mM) in $CHCl_3$ was added ([BA]/[poly-**1**-H] = 50), and then time-dependent CD intensity changes ($\Delta\varepsilon_{2nd}$) were measured at 25 °C.

## 9. Static Helicity Memory, Isolation of *h*-Poly-1-H, and Its Stability

After helicity induction in poly-**1**-H (0.3 μmol) with (*S*)-**2a** ([(*S*)-**2a**]/[poly-**1**-H] = 1) in $CHCl_3$ (300 μL, 1 mM) at 25 °C for 24 h in a 2-mL vessel with a screwcap, $CHCl_3$ was evaporated at 25 °C. The residual was dissolved in toluene (255 μL) to give the *h*-poly-**1**–(*S*)-**2a** complex in toluene (1.2 mM). The solution was cooled to –10 °C and then 45 μL of a solution of *p*-dodecylbenzene sulfonic acid (DBSA) (10 mM) in toluene ([DBSA]/[poly-**1**-H] = 1.5) was added at –10 °C to convert the *h*-poly-**1**–(*S*)-**2a** complex to the acid-type *h*-poly-**1**-H and the (*S*)-**2a**·DBSA salt in situ. The absorption and CD spectra of the resulting *h*-poly-**1**-H in toluene (1.0 mM) were then recorded at –10 °C (Figure 4A, (ii)). To this was added a solution of BA (15 μL, 1.0 M) ([BA]/[*h*-poly-**1**-H] = 50) in toluene at –10 °C to estimate the memory efficiency. By comparing the ICD intensity of the second Cotton ($\Delta\varepsilon_{2nd}$) just after the addition of BA to *h*-poly-**1**-H with that of the original *h*-poly-**1**–(*S*)-**2a** in toluene, the memory efficiency was estimated to be about 93% (Figure 4A, (iii)).

To isolate the *h*-poly-**1**-H, the mixture was poured into a large amount of methanol at –78 °C. The precipitated *h*-poly-**1**-H was collected by centrifugation, washed with methanol, and dried in vacuo at room temperature. The obtained *h*-poly-**1**-H was dissolved in toluene or $CHCl_3$ at –10 °C (1.0 mM), and the absorption and CD spectra were then recorded at –10 °C (Figure S9, (iii)). Time-dependent CD intensity changes ($\Delta\varepsilon_{2nd}$) were measured at –10 °C and 25 °C to investigate the stability of the helicity memory of *h*-poly-**1**-H in toluene and $CHCl_3$ (Figure 4B). Based on the $\Delta\varepsilon_{2nd}$ value just after adding a solution of BA (15 μL, 1.0 M) ([BA]/[*h*-poly-**1**-H] = 50) to the isolated *h*-poly-**1**-H in toluene at –10 °C, the memory efficiency after isolation was estimated to be about 45% (Figure S9, (iv)).

## 10. Molecular Modeling of the *h*-Poly-1-H

The molecular modeling and molecular mechanic (MM) calculations of a possible structure for the *h*-poly-**1**-H were conducted with the Compass III force field as implemented in the Materials Studio 2022 Modeling software (Version 22; Dassault Systèmes BIOVIA, San Diego, CA, USA)

operated using a PC running under Windows 10 (Figure S3). The units of *h*-poly-**1**-H was fully energy minimized by the Forcite Geometry Optimization using the Smart algorithm with the 0.001 kcal/mol·Å convergence criterion.

## 11. Supporting Data

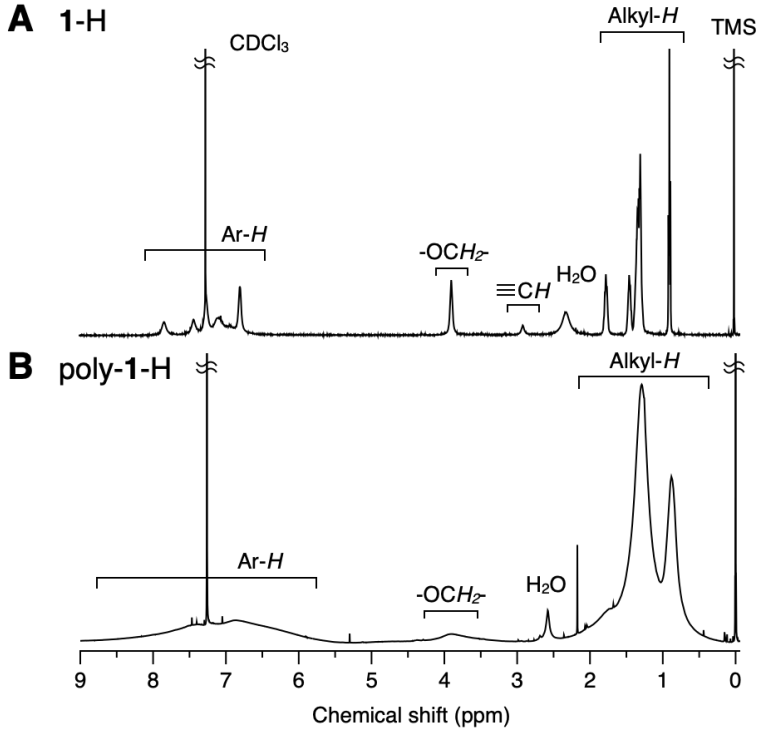



**Figure S1.** $^{1}$H NMR spectra of **1**-H (A) and poly-**1**-H (B) in $CDCl_3$ at 25 °C.

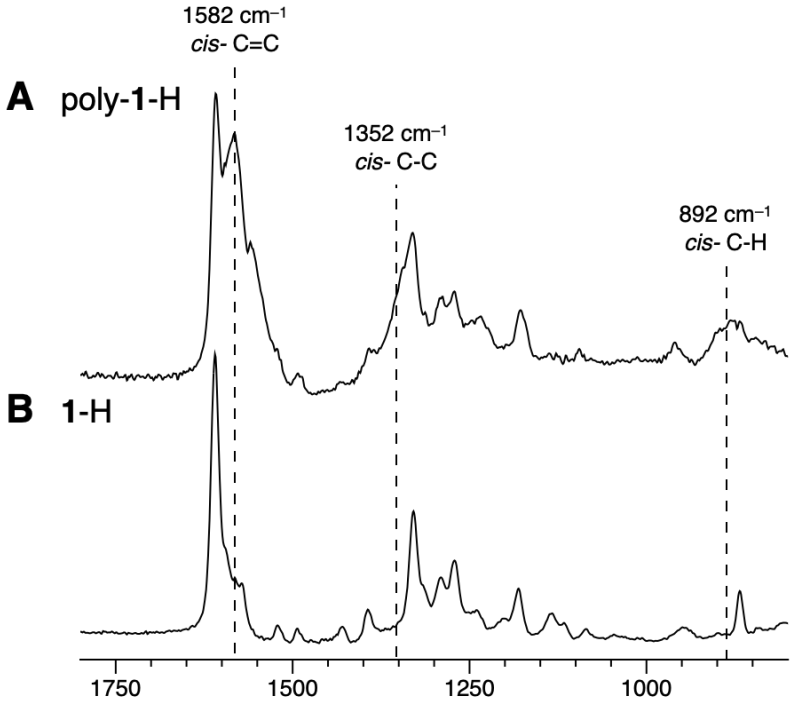



**Figure S2.** Laser Raman spectra of poly-**1**-H (A) and **1**-H (B) in the film state at room temperature (*ca.* 25°C).

**Table S1.** Solubility of Poly-**1**-H and PCPA[a]

| polymer | solubility | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | $H_2O$ (78.4)[b] | DMSO (46.5)[b] | MeOH (32.7)[b] | 1-butanol (17.5)[b] | THF (7.58)[b] | $CHCl_3$ (4.89)[b] | toluene (2.38)[b] | *n*-hexane (1.88)[b] |
| poly-**1**-H | × | × | × | × | ○ | ○ | ○ | × |
| PCPA-H | × | ○ | ○ | ○ | × | × | × | × |

[a]○：soluble, ×：insoluble, 1.0 mM, at room temperature (*ca.* 25°C). [b]Relative permittivity at 25 °C cited from ref 6.

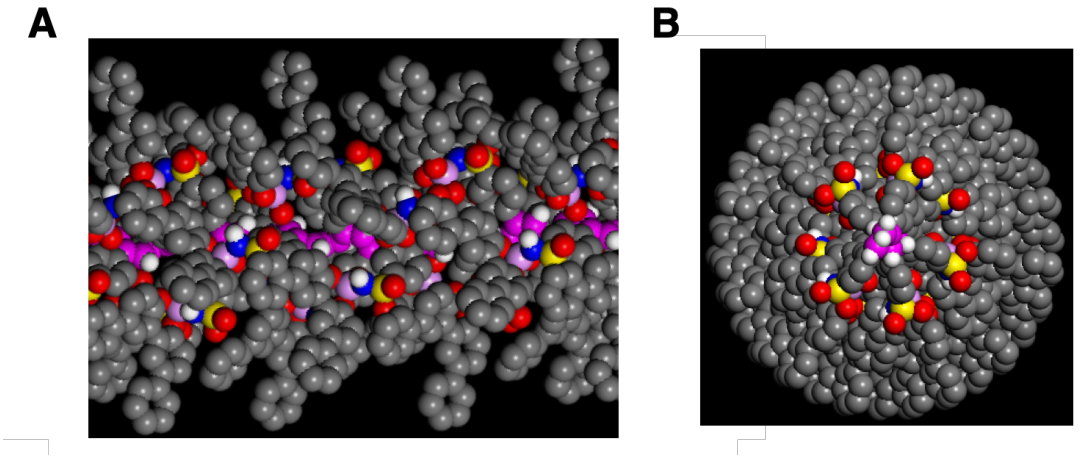
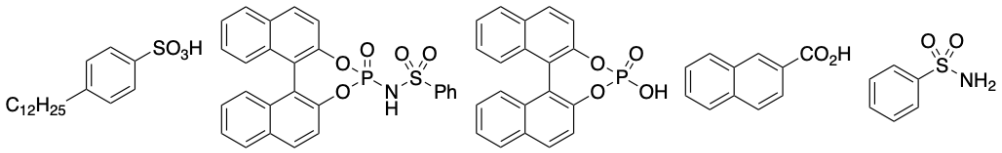



**Figure S3.** The side view (A) and top view (B) of optimized space-filling models of poly-**1**-H. Octyl ester groups of the pendants were omitted to simplify the images.

**Table S2.** Calculated p$K_a$ Values of Brønsted Acids DBSA and **3a**–**3d**

| compounds | p$K_{a(H_2O)}$[a] | p$K_{a(DMSO)}$[b] |
|---|---|---|
| DBSA | –0.45±0.50 | – |
| **3a** | 0.95±0.20 | 3.76 |
| **3b** | 1.14±0.20 | 3.15 |
| **3c** | 4.23±0.30 | ~11 |
| **3d** | 10.08±0.10 | 16.2 |

[a]Calculated using Advanced Chemistry Development (ACD/Labs) Software V11.02 (© 1994-2023 ACD/Labs) shown in SciFinder[n]. [b]Calculated by SMD/M06-2X/6-311++G (2df,2p)//B3LYP/6-31+G-(d) in DMSO in ref 7.




DBSA **3a** **3b** **3c** **3d**

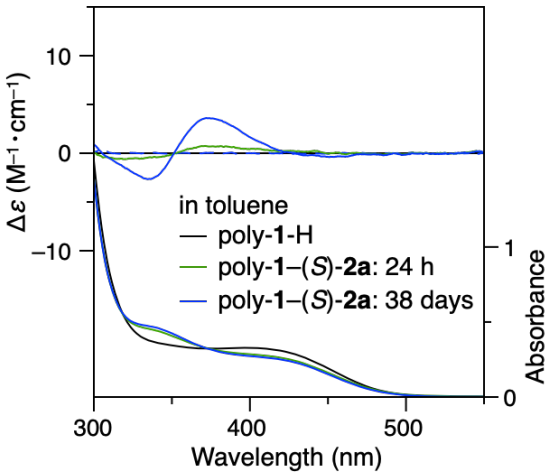



**Figure S4.** CD and absorption spectra of poly-**1**-H as prepared measured in toluene at 25 °C (black) and poly-**1**-H with (*S*)-**2a** ([(*S*)-**2a**]/[poly-**1**-H] = 1) measured in toluene at 25 °C after standing at 25 °C in toluene for 24 h (green) and 38 days (blue). [poly-**1**-H] = 1.0 mM.

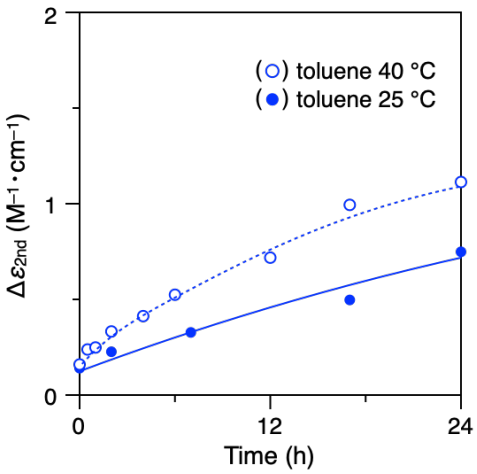



**Figure S5.** Time-dependent ICD intensity change ($\Delta\varepsilon_{2nd}$) of poly-**1**-H (1 mM) with (*S*)-**2a** ([(*S*)-**2a**]/[poly-**1**-H] = 1) measured in toluene at 25 °C after standing at 25 °C (closed circle) and 40 °C (open circle).

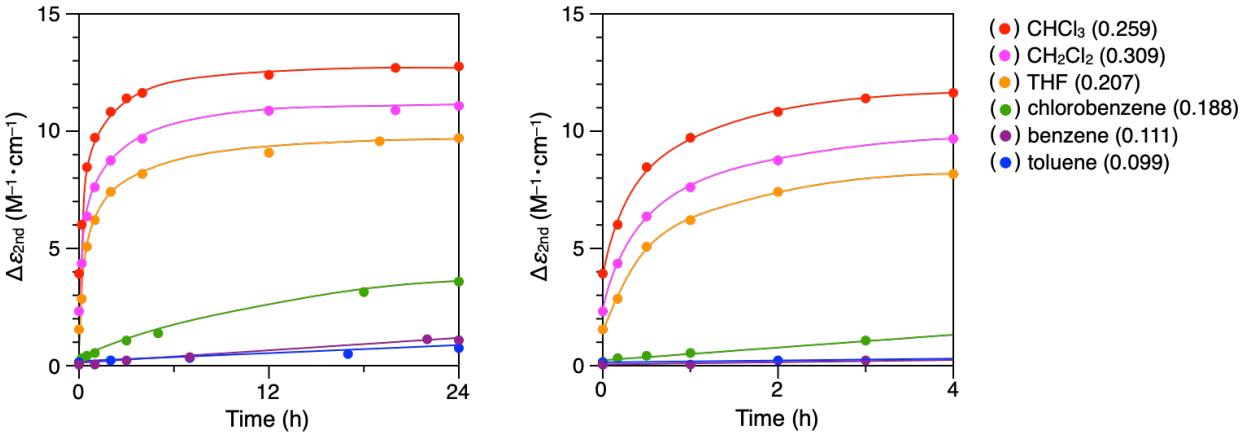



**Figure S6.** Time-dependent ICD intensity changes ($\Delta\varepsilon_{2nd}$) of poly-**1**-H (1 mM) with (*S*)-**2a** ([(*S*)-**2a**]/[poly-**1**-H] = 1) measured in various solvents at 25 °C after standing at 25 °C. The expanded details of the initial region (0–4 h) are shown in the right side. The values for relative polarity of the solvents, which are cited from ref 6, are shown in parentheses.

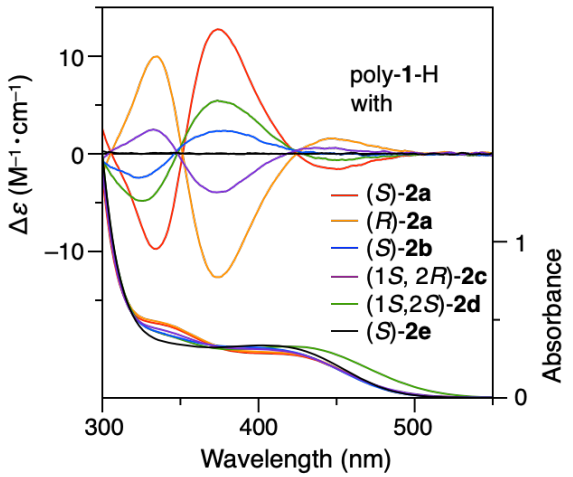



**Figure S7.** CD and absorption spectra of poly-**1**-H with chiral guests (**2a**–**2e**) ([**2**]/[poly-**1**-H] = 1) in $CHCl_3$ at 25 °C after standing at 25 °C until no further increase in the CD intensity was observed (24–31 h). [poly-**1**-H] = 1.0 mM.

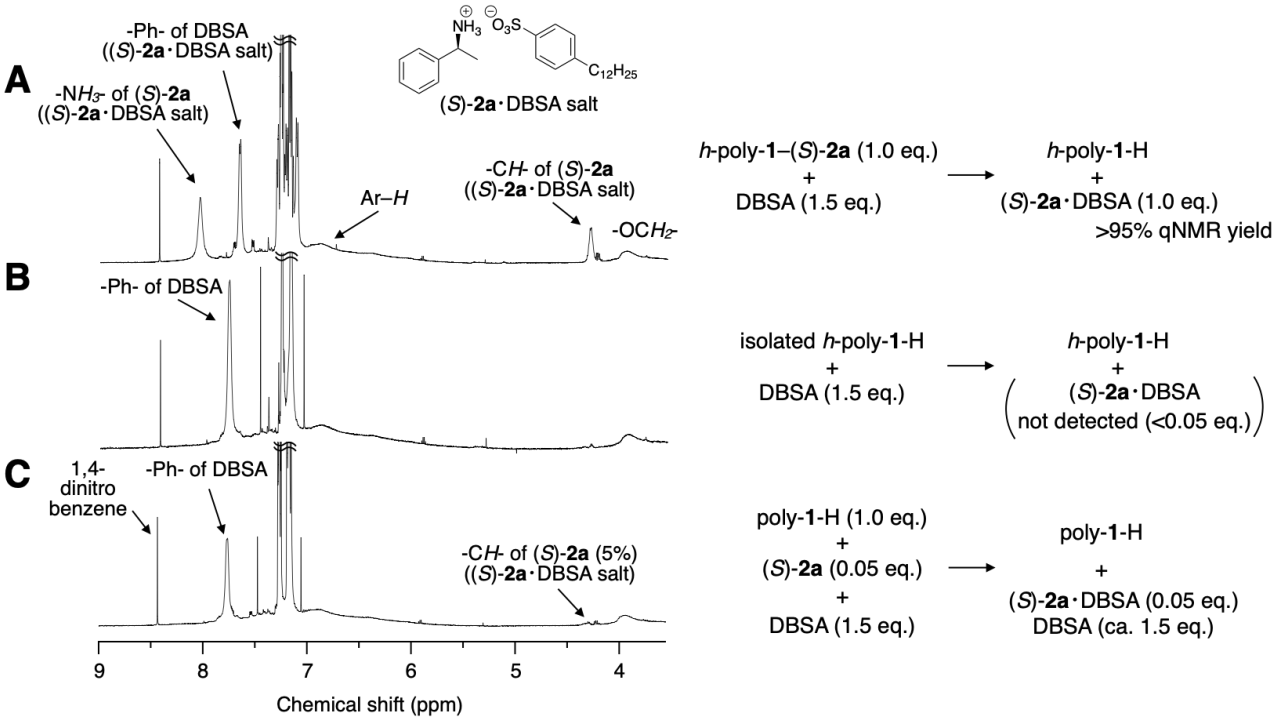



**Figure S8.** Quantitative $^1$H NMR spectra of *h*-poly-**1**–(*S*)-**2a** with DBSA ([(*S*)-**2a**]/[DBSA]/[poly-**1**-H] = 1/1.5/1 (A), the isolated *h*-poly-**1**-H with DBSA ([DBSA]/[poly-**1**-H] = 1.5) (B), and poly-**1**-H with (*S*)-**2a** and DBSA ([(*S*)-**2a**]/[DBSA]/[poly-**1**-H] = 0.05/1.5/1) (C) in $CDCl_3$ at 25 °C.

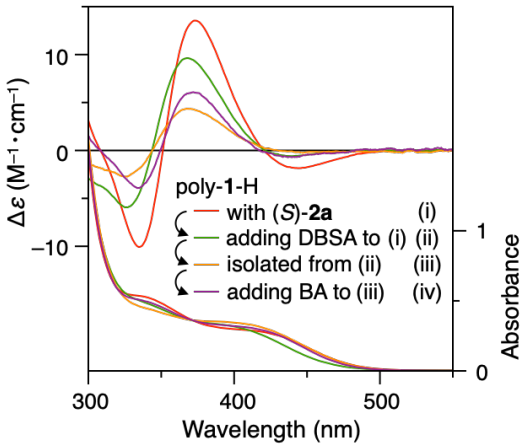



**Figure S9.** CD and absorption spectra of poly-**1**-H with (*S*)-**2a** ([**2a**]/[poly-**1**-H] = 1) measured in toluene at –10 °C (i) after helicity induction in $CHCl_3$ at 25 °C for 24 h（*h*-poly-**1**–(*S*)-**2a**）, (ii) after addition of DBSA ([DBSA]/[**2a**]/[poly-**1**-H] = 1.5/1/1) measured at –10 °C (*h*-poly-**1**-H), (iii) the isolated *h*-poly-**1**-H measured in toluene at –10 °C, and (iv) after further addition of BA ([BA]/[*h*-poly-**1**-H] = 50/1) measured at –10 °C. [poly-**1**-H] = 1.0 mM.

## NMR Data

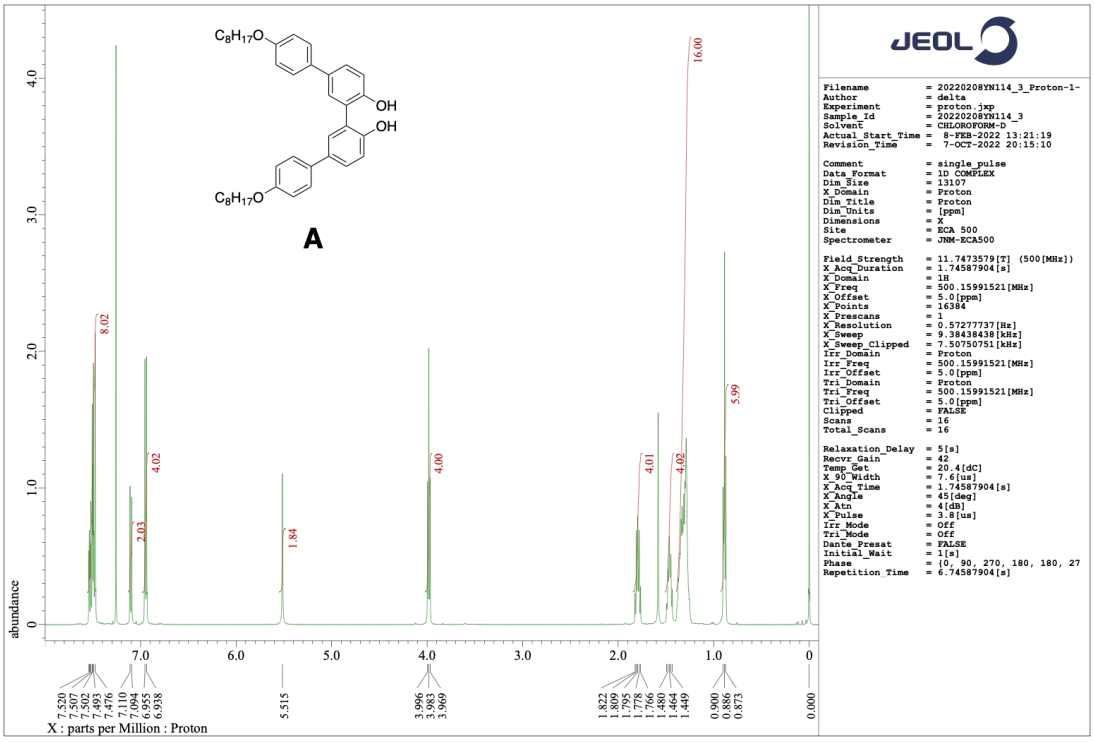



**Figure S10.** $^{1}$H NMR spectrum of **A** in $CDCl_3$ at rt.

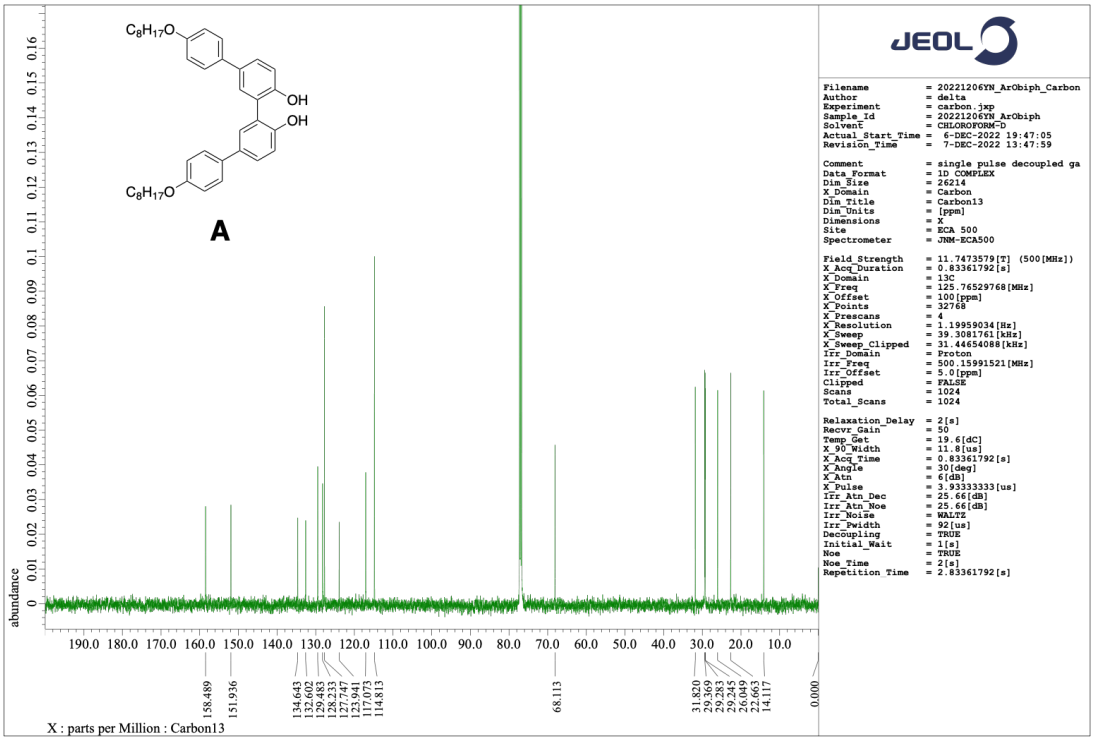



**Figure S11.** $^{13}$C NMR spectrum of **A** in $CDCl_3$ at rt.

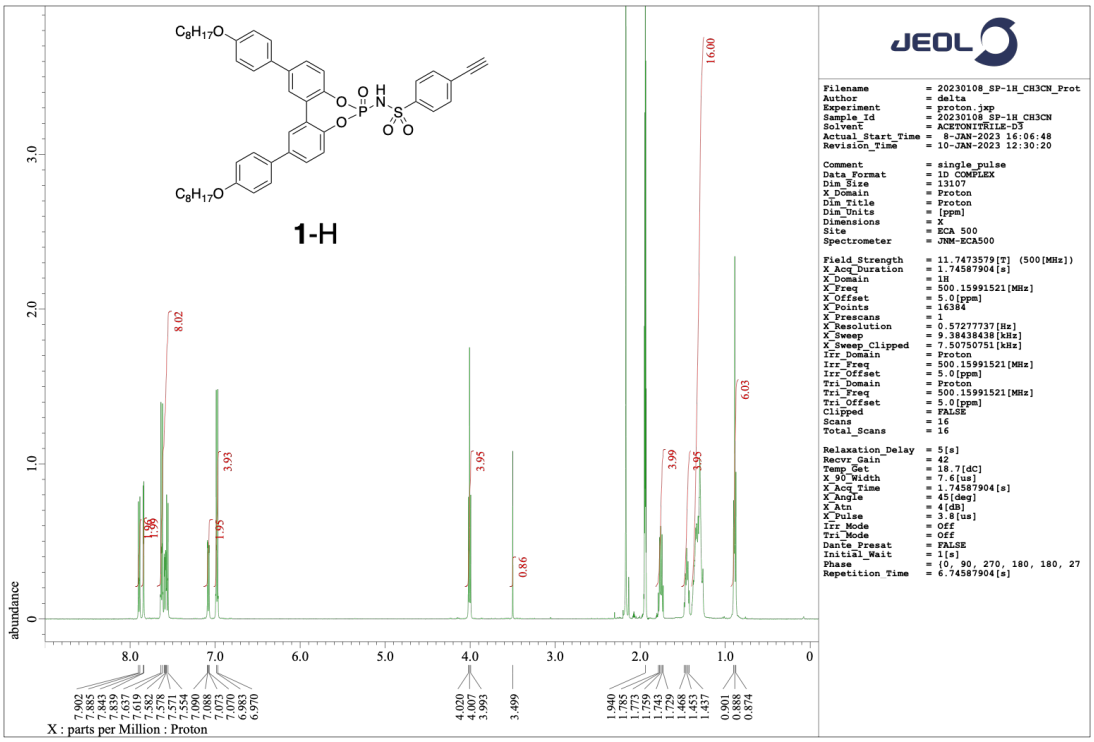



**Figure S12.** $^{1}$H NMR spectrum of **1**-H in acetonitrile-$d_3$ at rt.

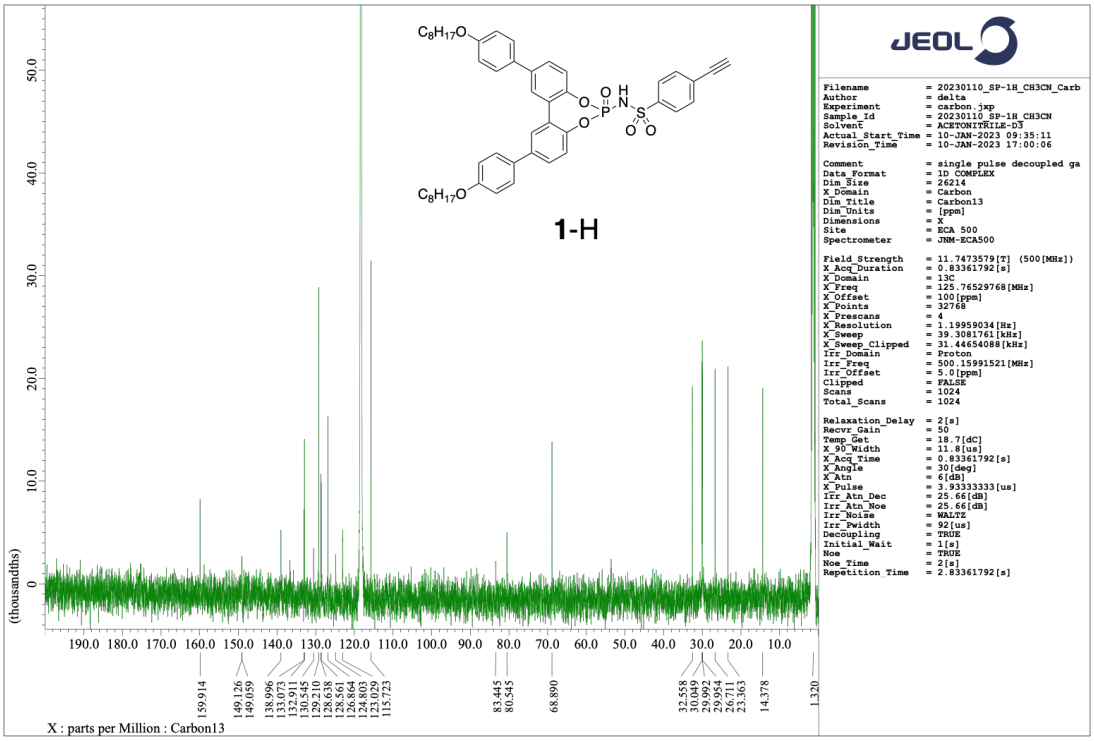



**Figure S13.** $^{13}$C NMR spectrum of **1**-H in acetonitrile-$d_3$ at rt.

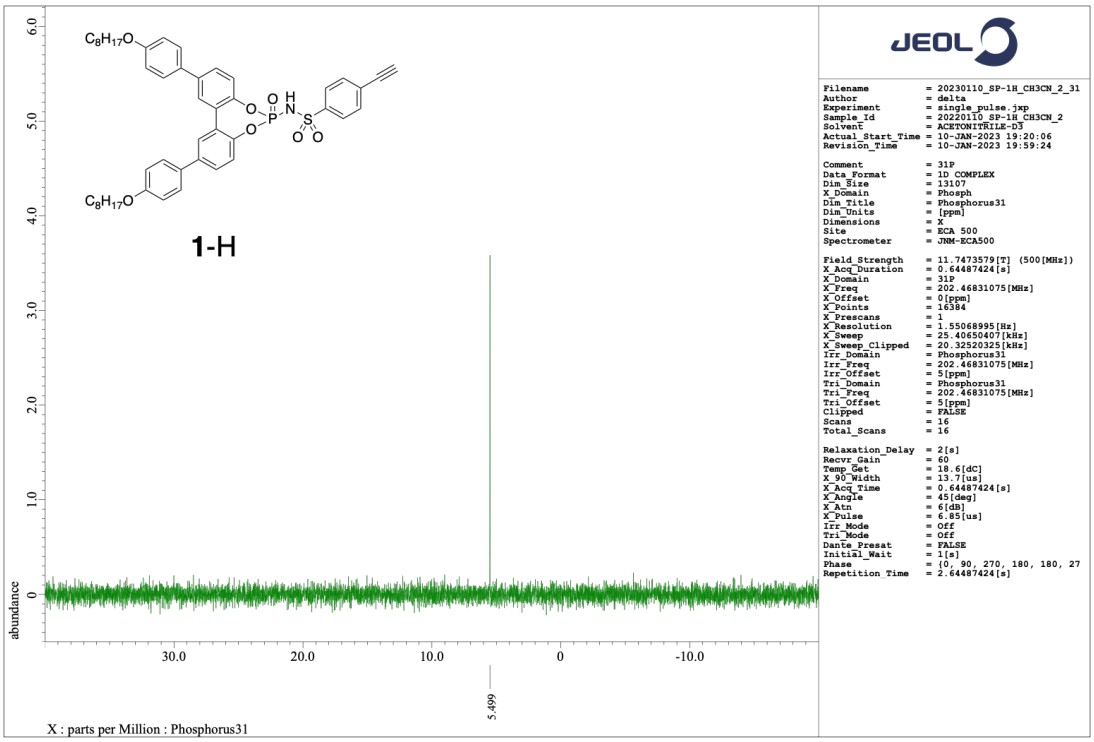



**Figure S14.** $^{31}$P NMR spectrum of **1**-H in acetonitrile-$d_3$ at rt.

**Reference**


1. J.-M. Wang, E.-F. He, H.-L. Wang, W.-L. Hou, J. Xu, L. Yu, L.-L. Zhao, Z.-L. Zhang and H.-Q. Zhang, *Chin. Chem. Lett.*, 2017, **28**, 383-387.
2. H. Mei and D. D. DesMarteau, *J. Fluor. Chem.*, 2014, **160**, 12-15.
3. S. Roscales, N. Bechmann, D. H. Weiss, M. Köckerling, J. Pietzsch and T. Kniess, *MedChemComm*, 2018, **9**, 534-544.
4. M. A. Saito, K. Maeda, H. Onouchi and E. Yashima, *Macromolecules*, 2000, **33**, 4616-4618.
5. H. Onouchi, K. Maeda and E. Yashima, *J. Am. Chem. Soc.*, 2001, **123**, 7441-7442.
6. C. Reichardt, in *Solvents and Solvent Effects in Organic Chemistry*, Wiley-VCH, 2010, DOI: https://doi.org/10.1002/9783527632220.app1, pp. 549-586.
7. C. Yang, X.-S. Xue, X. Li and J.-P. Cheng, *J. Org. Chem.*, 2014, **79**, 4340-4351.