Supporting Information

Quinoid-Controlled Bond-Length Alternation Enables High-Mobility Non-Fused π-Conjugated Polymers

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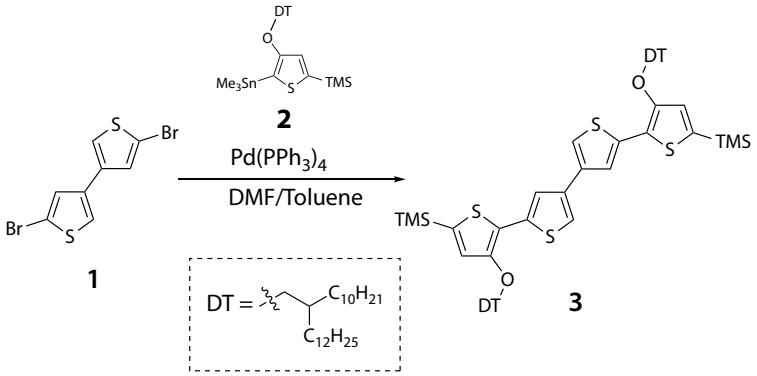
1. Materials and synthesis.

1-1. Materials

All chemicals and solvents were used as received unless otherwise indicated. Super dehydrated tetrahydrofuran (THF), *N*,*N*-dimethylformamide (DMF) and toluene was purchased from Wako Pure Chemical Industries Ltd. 5,5'-dibromo-3,3'-bithiophene (**1**) and (4-((2-decyltetradecyl)oxy)-5-(trimethylstannyl)thiophen-2-yl)trimethylsilane (**2**) were synthesized according to the reported procedure.[S1,S2] Nuclear magnetic resonance (NMR) spectra of all the compounds were taken on a Varian-500 or a Varian-400 spectrometer, using $CDCl_3$ calibrated with chloroform (CF) at 7.26 ppm for ^{1}H-NMR spectra and at 77.16 ppm for ^{13}C-NMR spectra, respectively. ^{1}H-NMR spectrum for the polymer was taken on a JNM-ECA500 spectrometer, using deuterated *o*-dichlorobenzene (DCB-d_4) at 130 ºC, calibrated with *o*-dichlorobenzene (DCB) at 7.15 and 6.87 ppm. High-resolution mass spectrum (HRMS) was performed using LTQ Orbitrap XL (Thermo Fisher Scientific, Inc.). Polymerization was carried out using a microwave reactor (Biotage Initiator). Molecular weights were determined by gel permeation chromatography (GPC) by calibrating with polystyrene standards using a TOSOH at 180 ℃ with 1,2,4-trichlorobenzene as a solvent, which was calibrated with polystyrene standards.

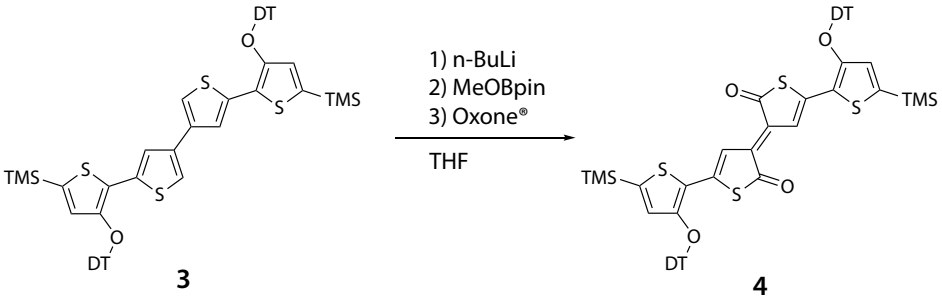
1-2. Synthesis and Characterization



(3,3‴-Bis((2-decyltetradecyl)oxy)-[2,2′:4′,3″:5″,2‴-quaterthiophene]-5,5‴-diyl)bis(trimethylsilane) (***3***)

A solution of **1** (1.30 g, 4.0 mmol), **2** (10.8 g, 16.0 mmol), and $Pd(PPh_3)_4$ (231 mg, 0.20 mmol) in anhydrous toluene/DMF (30 mL/30 mL) in a three neck round bottom flask was heated at 100 °C for 12 h. After cooling to room temperature, water was added, and the resulting mixture was extracted with CF. The organic layer was washed with water and brine, and dried over anhydrous magnesium sulfate. After removing the solvent by evaporation, the crude product was purified by silica-gel column chromatography using hexane (R_f = 0.35) as eluent to give **3** as a pale-yellow oil (2.01 g, 1.71 mmol, yield = 43%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, J = 1.5 Hz, 2H), 7.23 (d, J = 1.5 Hz, 2H), 6.94 (s, 2H), 4.03 (d, J = 5.2 Hz, 4H), 1.85–1.79 (m, 2H), 1.33–1.23 (m , 80H), 0.90–0.85 (m, 12H), 0.33 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 154.47, 136.94, 136.06, 123.12, 121.48, 120.02, 118.03, 74.31, 38.62, 32.10, 31.53, 30.21, 29.88, 29.84, 29.55, 27.10, 22.87, 14.29. HRMS (APCI): $C_{70}H_{122}S_4Si_2$ Calcd. for $[M+H]^+$: 1176.78607, Found: 1176.78613.

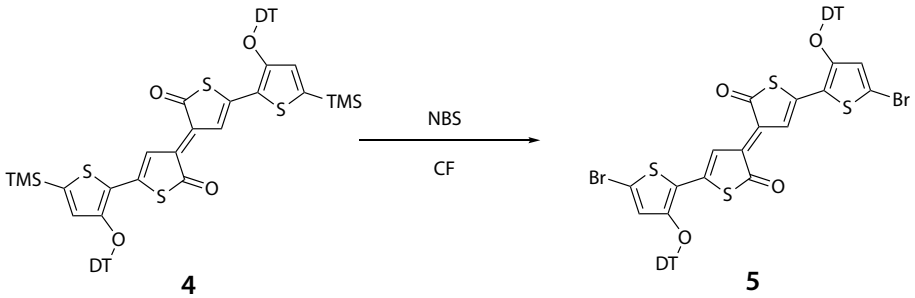


3 **4**

(E)-3,3'''-Bis((2-decyltetradecyl)oxy)-5,5'''-bis(trimethylsilyl)-2''H,5'H-[2,2':4',3'':5'',2'''-quaterthiophene]-2'',5'-dione (***4***)

n-Butyllithium (1.6 M in hexane) (6.48 mL, 10.4 mmol) was added dropwise to a solution of **3** (3.1 g, 2.6 mmol) in anhydrous THF (30 mL) at −78 °C. After stirring for 1 h and then warm up to 0 °C for 10 min, 2-methoxy 4,4,5,5-tertramethyl-1,3,2-dioxaborolane (2.96 mL, 18.2 mmol) was added in one portion at −78 °C. After stirring for 3 h while gradually raising the temperature to room temperature, toluene (20 mL) was poured into the mixture and filtered through the PTFE membrane filter. After evaporation of the solvent under reduced pressure, the residue was dissolved in dichloromethane (DCM) (20 mL)/acetone (30 mL)/H_2O (20 mL). Oxone® ($2KHSO_3 \cdot K_2SO_4 \cdot KHSO_4$) (7.8 g, 13.0 mmol) was added to a solution and stirred for 12 h. The mixture was cooled to 0 °C and quenched with saturated sodium sulfate aqueous solution. After evaporation of organic solvents under reduced pressure, the residue was extracted with CF, washed with water and brine, and dried over magnesium sulfate. After removing the solvent, the crude product was purified by silica gel column chromatography using hexane/DCM = 19/1 as eluent (R_f = 0.30) to give **4** as a deep blue oil (806 mg, 0.67 mmol, yield = 26%).

^{1}H NMR (500 MHz, $CDCl_3$): 8.22 (s, 2H), 6.92 (s, 2H), 4.05 (d, 4H), 1.89–1.83 (m, 2H), 1.26–1.24 (m, 80H), 0.88–0.86 (m, 12H), 0.34 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.51, 159.45, 144.79, 139.35, 132.88, 122.23, 120.80, 116.92, 74.56, 38.12, 31.77, 31.03, 29.81, 29.57, 29.54, 29.52, 29.50, 29.23, 26.68, 22.54, 13.97. HRMS (APCI) m/z calcd for $C_{70}H_{120}O_2S_4Si_2$ $[M+H]^+$: 1176.76025, Found: 1176.76160.

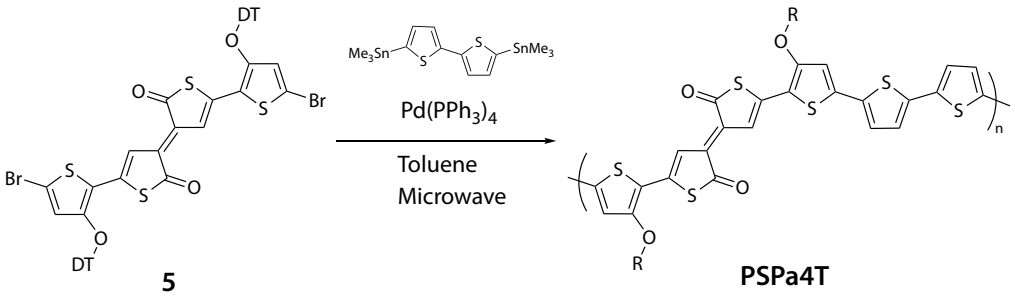


(E)-5,5'''-Dibromo-3,3'''-bis((2-decyltetradecyl)oxy)-2''H,5'H-[2,2':4',3'':5'',2'''-quaterthiophene]-2'',5'-dione (***5***)

To a solution of **4** (265 mg, 0.22 mmol) in 30 mL of CF, *N*-bromosuccinimide (NBS) (81.9 mg, 0.46 mmol) was added in 5 portions at 0 °C. The reaction mixture was gradually warmed to room temperature and stirred for 3 h. The reaction mixture was quenched by water and then extracted with CF three times. The organic layer was washed with water and dried over magnesium sulfate. After removing the solvent by evaporation, the crude product was purified by silica gel column chromatography using hexane/DCM = 9/1 as eluent (R_f = 0.40) and recrystallized from hexane/ethanol =1/3 to give **5** as a deep blue solid (163 mg, 0.13 mmol, yield = 60%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (s, 2H), 6.88 (s, 2H), 4.00 (d, 4H), 1.84–1.81 (m, 2H), 1.27–1.24 (m, 80H), 0.88–0.85 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.18, 157.48, 138.72, 133.02, 120.61, 118.07, 117.07, 75.44, 38.31, 32.09, 31.20, 30.08, 29.89, 29.83, 29.79, 29.55, 26.92, 22.86, 14.29. HRMS (APCI) m/z calcd for $C_{64}H_{102}Br_2O_4S_4$ $[M+H]^+$: 1191.50537. Found: 1191.50222.

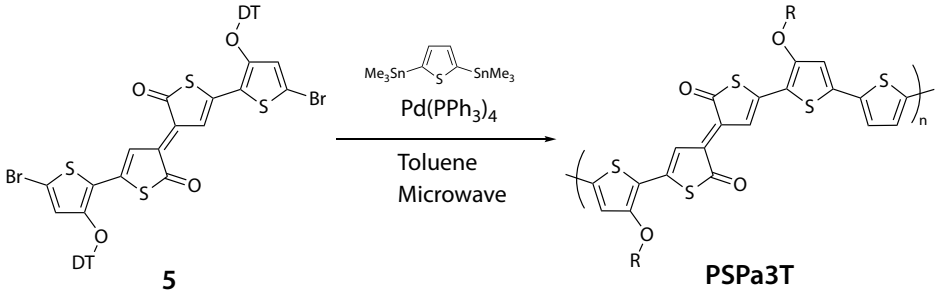
PSPa4T.



5 **PSPa4T**

5 (61.18 mg, 0.05 mmol), 5,5'-bis(trimethylstannyl)-2,2'-bithiophene (24.59 mg, 0.05 mmol), tetrakis(triphenylphosphine)palladium(0) (1.16 mg, 0.001 mmol) and 2 mL of toluene was added in a 2 mL reaction vessel. The vessel was purged with argon and sealed. The vessel was put into a microwave reactor and heated at 200 °C for 2 h. After cooling to room temperature, the reaction mixture poured into methanol/HCl solution (95 mL/5 mL) and then vigorously stirred for 2 h at room temperature. The precipitate was filtered and subjected to sequential Soxhlet extraction with methanol, hexane, dichloromethane, chloroform, and chlorobenzene to remove low molecular weight fraction. The residue was extracted with *o*-dichlorobenzene, which was then reprecipitated in methanol. The precipitate was filtered and dried in vacuo to obtain **PSPa4T** (53 mg, 0.043 mmol) in 86% yield as a dark brown solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.20–3.80 (m), 1.80–1.20 (m), 1.0–0.80 (m).

PSPa3T.

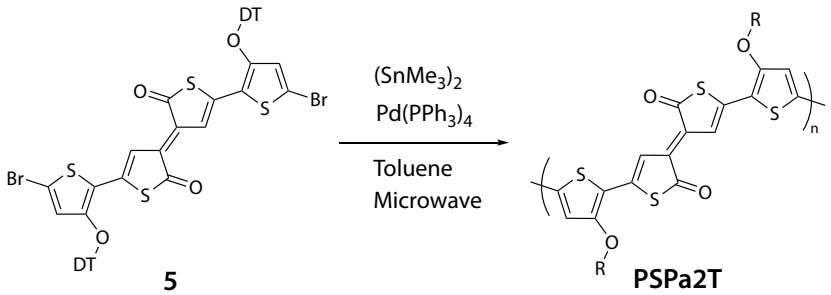


5 **PSPa3T**

5 (43.01 mg, 0.035 mmol), 2,5-bis(trimethylstannyl)thiophene (14.4 mg, 0.035 mmol), tetrakis(triphenylphosphine)palladium(0) (0.81 mg, 0.0007 mmol) and 2 mL of toluene was added in a 2 mL reaction vessel. The vessel was purged with argon and sealed. The vessel was put into a microwave reactor and heated at 140 °C for 2 h. After cooling to room temperature, the reaction mixture poured into methanol/HCl solution (95 mL/5 mL) and then vigorously stirred for 2 h at room

temperature. The precipitate was filtered and subjected to sequential Soxhlet extraction with methanol, hexane, and dichloromethane to remove low molecular weight fraction. The residue was extracted with chloroform, which was then reprecipitated in methanol. The precipitate was filtered and dried in vacuo to obtain **PSPa3T** (38 mg, 0.033 mmol) in 94% yield as a dark brown solid. δ 4.40–3.90 (m), 1.80–1.20 (m), 1.0–0.80 (m).

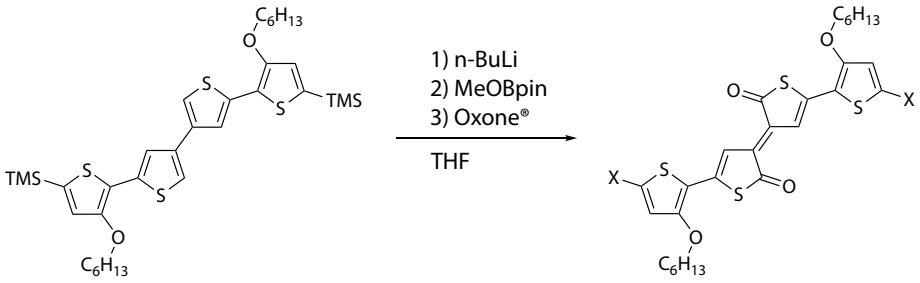
PSPa2T.



5 **PSPa2T**

5 (61.18 mg, 0.05 mmol), hexamethyldistannane (16.38 mg, 0.05 mmol), tetrakis(triphenylphosphine)palladium(0) (1.17 mg, 0.001 mmol) and 2 mL of toluene was added in a 2 mL reaction vessel. The vessel was purged with argon and sealed. The vessel was put into a microwave reactor and heated at 140 °C for 2 h. After cooling to room temperature, the reaction mixture poured into methanol/HCl solution (95 mL/5 mL) and then vigorously stirred for 2 h at room temperature. The precipitate was filtered and subjected to sequential Soxhlet extraction with methanol, hexane, and dichloromethane to remove low molecular weight fraction. The residue was extracted with chloroform, which was then reprecipitated in methanol. The precipitate was filtered and dried in vacuo to obtain **PSPa2T** (47 mg, 0.044 mmol) in 89% yield as a dark brown solid. δ 4.20–3.70 (m), 1.80–1.10 (m), 1.00–0.80 (m).

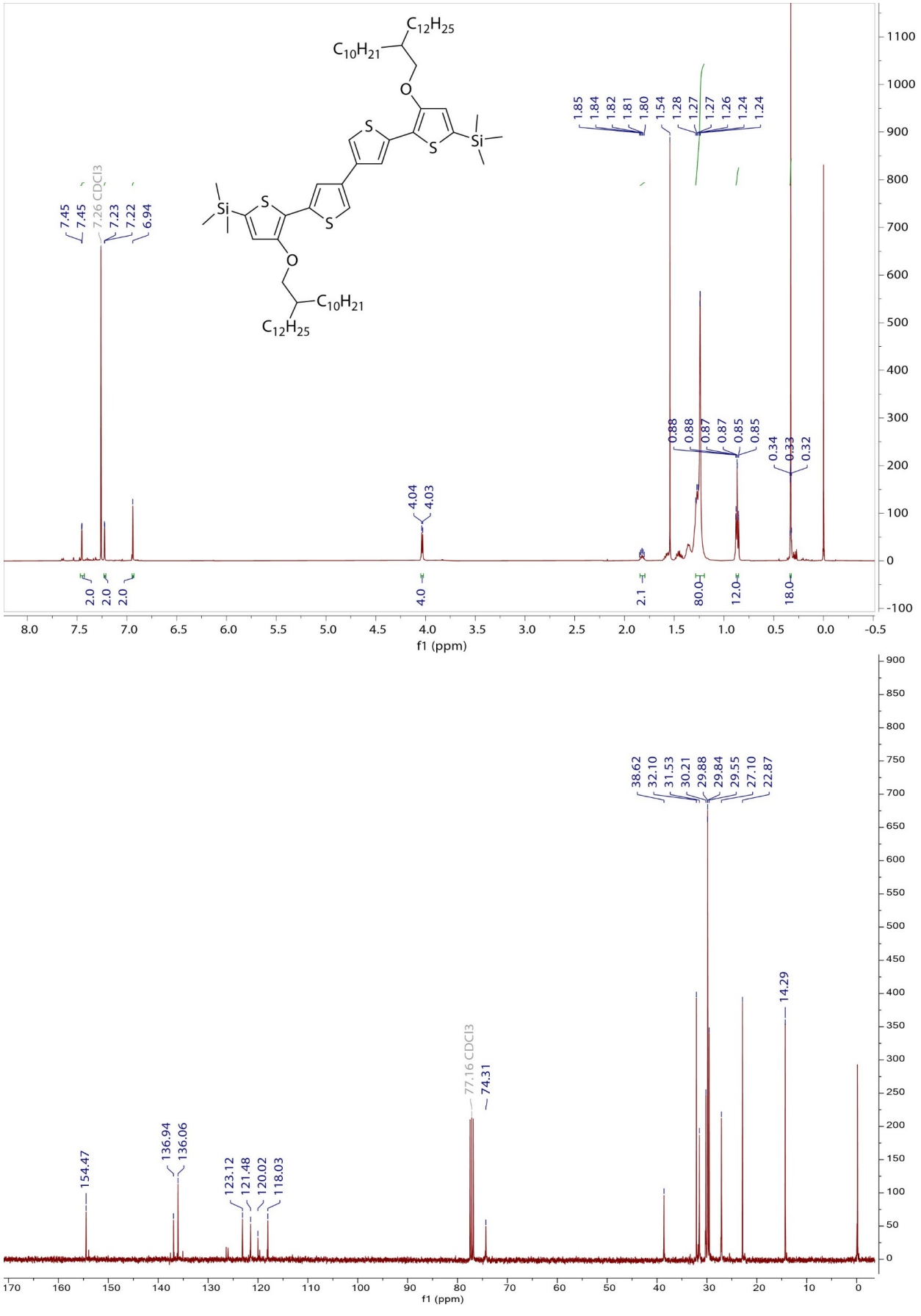
SPa2T



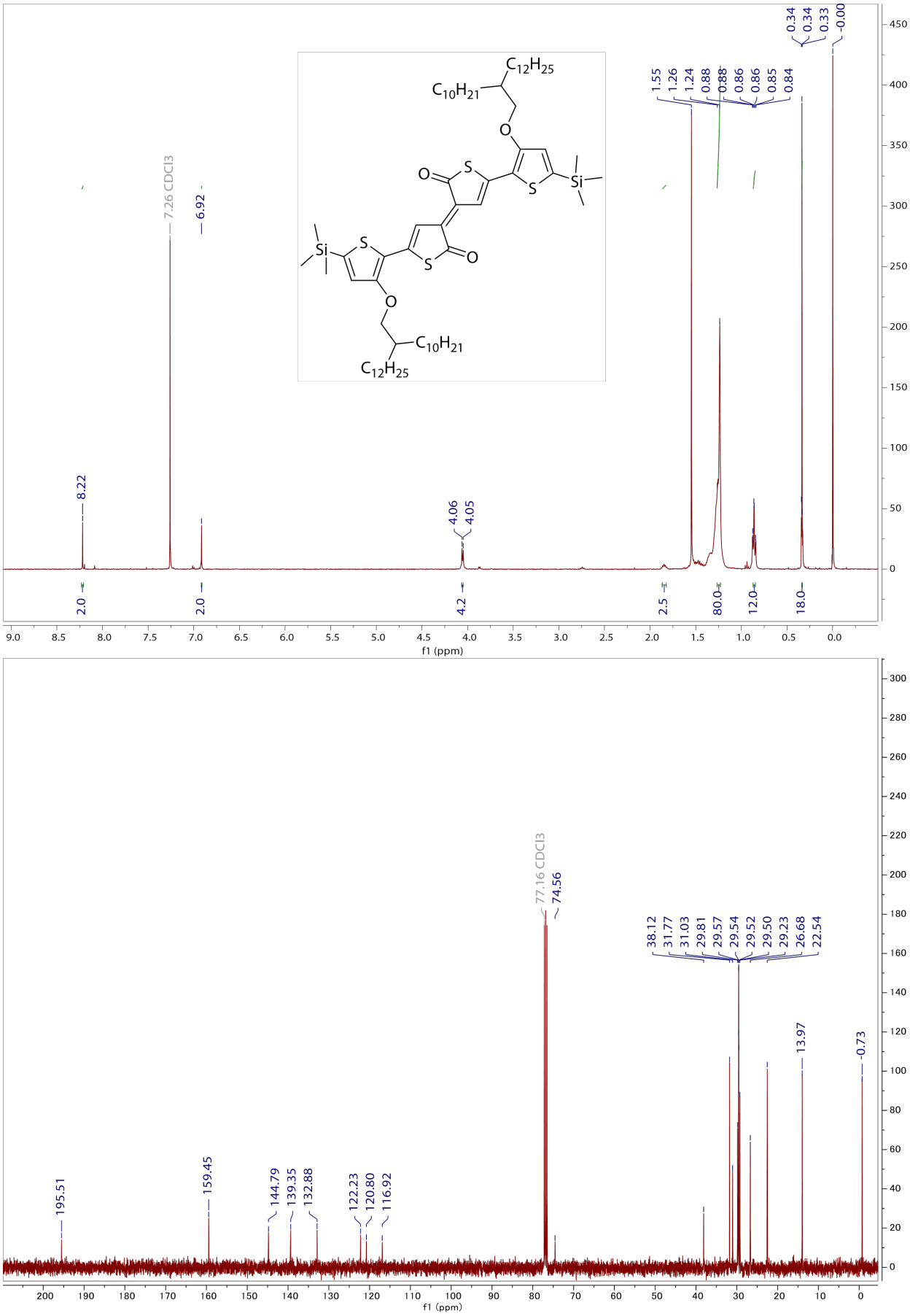
3-C$_6$ **4′** (X = TMS)
SP2T (X = H)

The title compound was synthesized by the same procedure as **4** using **3-C6** (205 mg, 0.30 mmol), *n*-BuLi (0.80 mL, 1.28 mmol), MeOBpin (0.36 mL, 2.24 mmol) in THF (20 mL) and Oxone (980 mg, 1.60 mmol) in DCM/Acetone/H_2O (10 mL/10 mL/10 mL). The crude product was purified by silica gel column chromatography using hot hexane/DCM = 5/1 as eluent and recrystallized from chloroform/ethyl acetate to obtain **SPa2T** as a deep metallic green solid (65 mg, 0.12 mmol, 39%). Note that the TMS-protected (**4′**) and deprotected products (**SPa2T**) were obtained at this time. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (s, 2H), 7.36 (d, *J* = 5.5 Hz, 2H), 6.85 (d, *J* = 5.6 Hz, 2H), 4.16 (t, 4H), 1.90–1.86 (m, 4H), 1.40–1.25 (m , 12H), 0.93–0.89 (m, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.66, 158.55, 139.83, 128.31, 117.01, 72.40, 31.45, 29.69, 29.47, 25.66, 22.56, 14.03. HRMS (APCI): $C_{28}H_{32}S_4O_4$ Calcd. for $[M+H]^+$: 561.12562, Found: 561.12579.

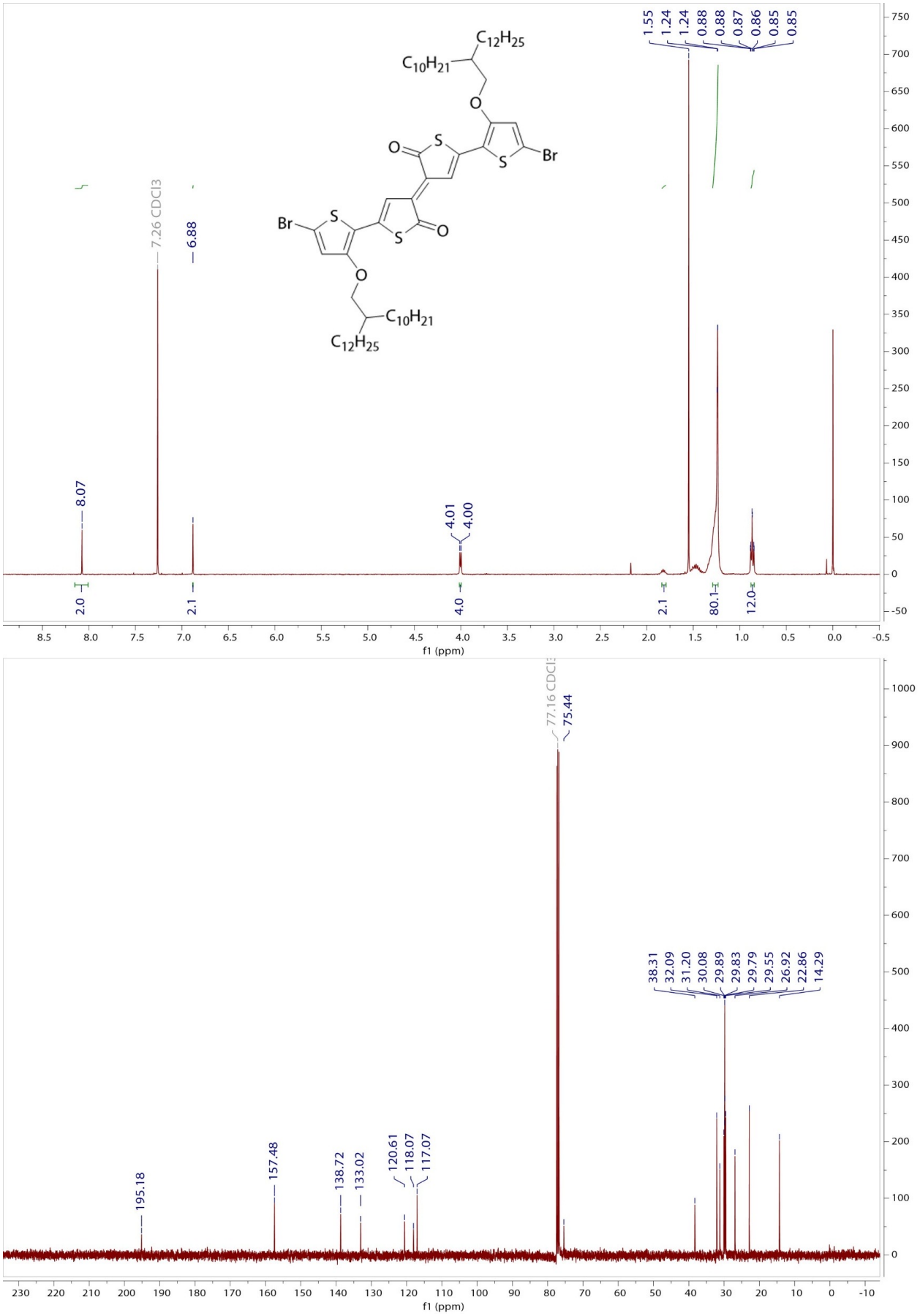
1-3. NMR charts.



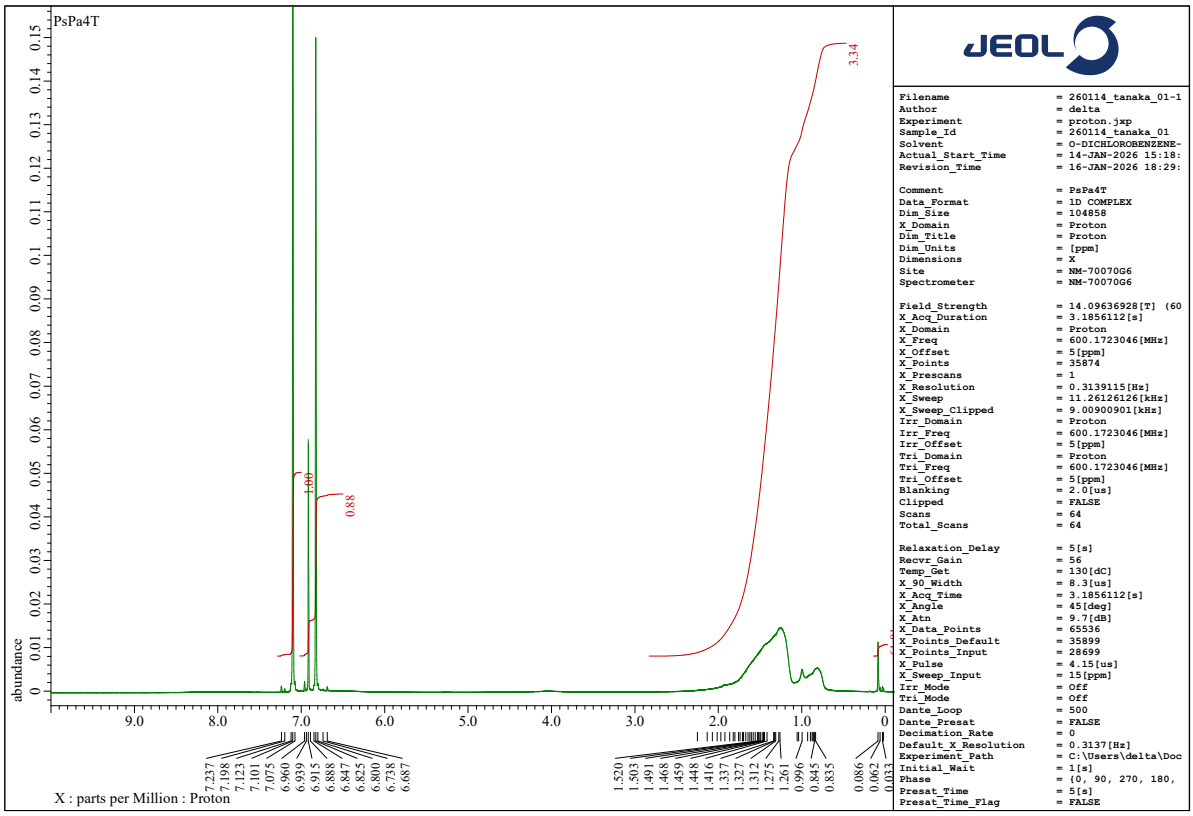
^{1}H-(upper) and ^{13}C-NMR spectra (lower) of **3**



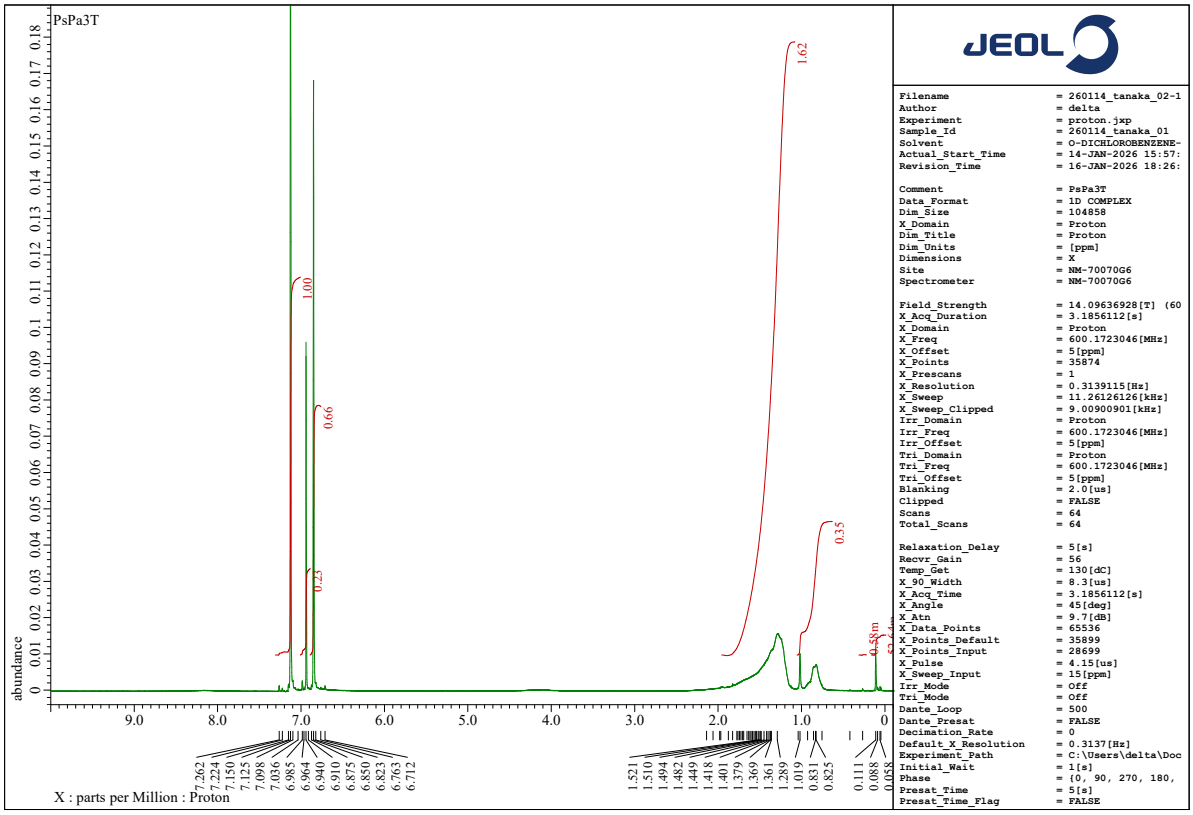
^{1}H-(upper) and ^{13}C-NMR spectra (lower) of **4**



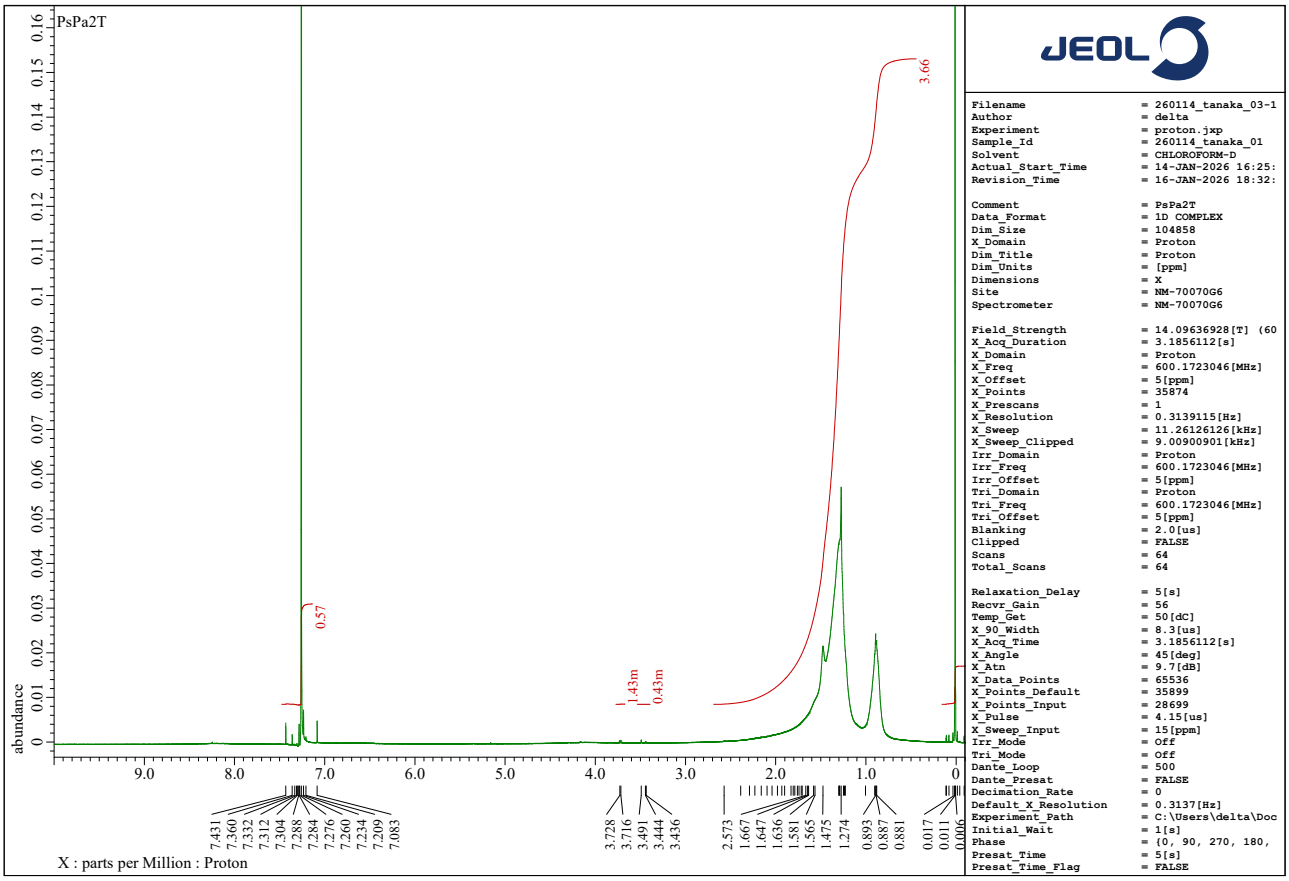
^{1}H-(upper) and ^{13}C-NMR spectra (lower) of **5.**



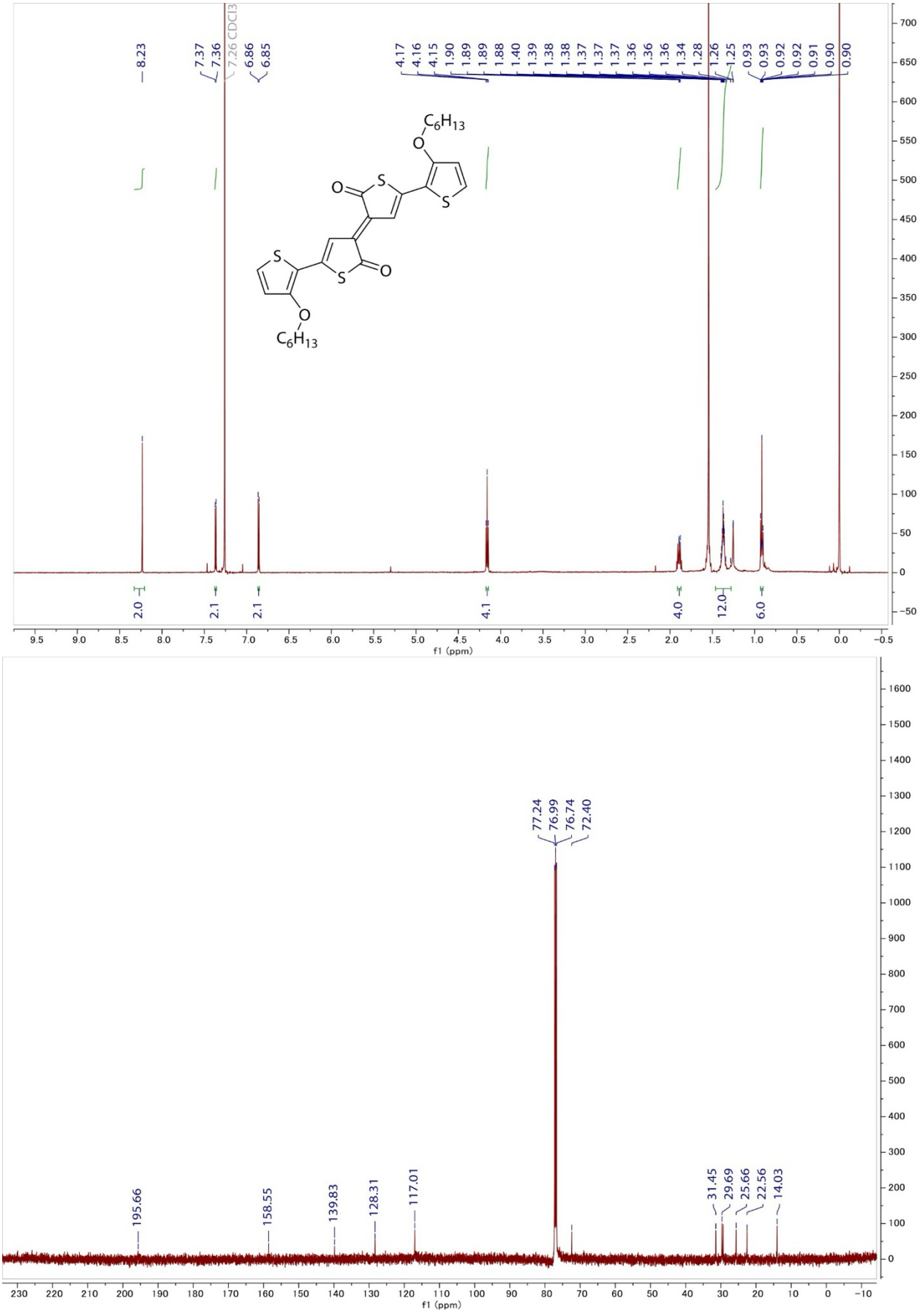
^{1}H-NMR spectra of **PSPa4T**



^{1}H-NMR spectra of **PSPa3T**



^{1}H-NMR spectra of **PSPa2T**



^{1}H-(upper) and ^{13}C-NMR spectra (lower) of **SPa2T**

2. Instrumentation.

Cyclic voltammograms were recorded on an ALS Electrochemical Analyzer Model 612D with the three electrodes system consisting of a platinum disc working electrode (φ = 3 mm), a platinum wire counter electrode, and an Ag/Ag^+ reference electrode in acetonitrile containing tetrabutylammonium hexafluorophosphate (0.1 M) at a scan rate of 100 mV s^{-1}. The thin films of the materials were cast from CB solution directly on the working electrode. All the potentials were calibrated with the half-wave potential of the ferrocene/ferrocenium redox couple measured under identical condition (Fc/Fc^+ = 0.16 V). HOMO and LUMO energy levels (E_{HOMO} and E_{LUMO}) were estimated by the following equations, E_{HOMO}: $-4.80 - E_{ox}^{onset}$, E_{LUMO}: $-4.80 - E_{red}^{onset}$, where E_{ox}^{onset} and E_{red}^{onset} are onset potentials for the oxidation and reduction peaks. Photoemission yield spectroscopy (PYS) in air was performed by a spectrometer, model AC-2S (Riken Keiki Co., Ltd). The thin film samples for the PYS measurements were prepared by spin-coating from DCB solution (for **PSPa4T**) and CF (for **PSPa3T**, and **PSPa2T**) on ITO/glass substrate. UV–vis absorption spectroscopy was performed with Shimadzu UV-3600 Plus spectrometer. As for the samples for the UV–vis measurements, the polymer solutions (5×10^{-6} M) were prepared using DCB as the solvent and the polymer thin films were prepared by spin-coating from the DCB or CF solution.

GIXD experiments were conducted at the SPring-8 on the beamline BL46XU. The sample was irradiated with the X-ray energy of 12.39 keV (λ = 1 Å) at a fixed incident angle on the order of 0.12 ° through a Huber diffractometer. The 2D GIXD patterns were recorded with a 2D image detector (Pilatus 300 K). Samples for the X-ray measurements were prepared in the same manner as that for solar cell fabrication. Tapping mode atomic force microscopy was carried out on an SPM-9700HT scanning probe microscope (Shimadzu Corp). Transmission electron microscopy (TEM) was conducted on JEM-2021 (JEOL).

3. Calculation of band structure and effective mass.[S3,S4]

We investigated the electronic properties of isolated polymers, **PSPa4T**, **PSPa3T**, and **PSPa2T**, based on density functional theory[S2] with a plane wave basis having cutoff energies of 50 Rydberg for plane

wave and of 500 Rydberg for charge density. The Brillouin zone integration is performed with a 4 × 4 × 4 k-point set. For **PSPa4T**, **PSPa3T**, and **PSPa2T,** first, the cell parameters as well as the internal degrees of freedom are fully relaxed using the van der Waals density functional (vdW-DF2).[S3,S4] The resulted conformation of both polymers were all-trans and nearly coplanar structure which was consistent with the DFT (B3LYP/6-31G(d,p)) calculated torsion potential in which such conformation locates at the minimum energy point. After obtaining the relaxed structures, we calculated the electronic band structures along the backbone of polymers. The hole and electron effective mass along the backbone is obtained by fitting to analytic expression $E(k) \approx E_0 - (\hbar^2/2m^*)(k - k_0)^2$, where the top of the HOMO and LUMO band with energy E_0 is located at $k = k_0$ on the Γ-X direction.

4. Single crystal X-ray analysis.

Experimantal: Single dark blue plate-shaped crystals of **SPa2T** were obtained by recrystallisation from chloroform/ethyl acetate. A suitable crystal 0.08 × 0.06 × 0.02 mm^3 was selected and mounted on a suitable support on an XtaLAB Synergy R, DW system, HyPix diffractometer. The crystal was kept at a steady T = 100.00(10) K during data collection. The structure was solved with the ShelXT 2018/2 (Sheldrick, 2018) structure solution program using the Intrinsic Phasing solution method and by using Olex2 (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of ShelXL 2018/3 (Sheldrick, 2015) using Least Squares minimization.

Crystal Data. $C_{28}H_{32}O_4S_4$, *M*r = 560.77, triclinic, P-1 (No. 2), a = 8.6729(7) Å, b = 9.1389(6) Å, c = 9.2507(6) Å, α = 69.637(6)°, β = 89.678(6)°, γ = 78.894(6)°, V = 673.02(9) $Å^3$, T = 100.00(10) K, Z = 1, Z' = 0.5, μ (Mo Kα) = 0.386, 4754 reflections measured, 2946 unique (Rint = 0.0511) which were used in all calculations. The final wR2 was 0.1418 (all data) and R1 was 0.0558 (I > 2(I)).

Compound	**SPa2T**
Formula	$C_{28}H_{32}O_4S_4$
Dcalc. / g cm^{-3}	1.384
μ / mm^{-1}	0.386
Formula	560.77
Colour	Dark blue
Shape	plate
Size / mm^3	0.08 × 0.06 × 0.02
T / K	100.00(10)

Crystal system	Triclinic
Space group	P-1
a/Å	8.6729(7)
b/Å	9.1389(6)
c/Å	9.2507(6)
a/°	69.637(6)
b/°	89.678(6)
g/°	78.894(6)
V/Å^3	673.02(9)
Z	1
Z'	0.5
Wavelength/Å	0.71073
Radiation type	Mo K_α
Q_{min}/°	2.354
Q_{max}/°	30.059
Measured Refl.	4754
Independent Refl.	2946
Reflections I >	2019
2(I)	
R_{int}	0.0511
Parameters	164
Restraints	0
Largest peak	0.806
Deepest hole	-0.355
GooF	1.006
wR2 (all data)	0.1418
wR2	0.1265
R1 (all data)	0.091
R1	0.0558

5. OFET fabrication and measurements.

Top-gate-bottom-contact devices were fabricated on an alkaline-free glass substrate patterned with Cr/Au source and drain electrode, deposited via photolithography. The glass substrates were ultrasonicated with isopropanol for 10 min, and rinsed in boiled isopropanol for 10 min, and then were subjected to UV-ozone treatment for 30 min. The cleaned substrates were treated by 1-octanethiol (OT) to modify the Au source and drain electrodes. For the OT treatment, the substrate was soaked in a solution of OT in toluene (10 mM) in a petri dish for 10 minutes at room temperature. The substrates were rinsed with water and hot isopropanol. Polymer thin films were spin-coated from hot (~100 ºC) 3 g L^{-1} 1,2-dichlorobenzene (for **PSPa4T**) and 4 g L^{-1} CF solutions (for **PSPa3T** and **PSPa2T**), and then were annealed at 200 ºC for 30 min. The PMMA dielectric layer (C_i = 3.6 nFcm^{-2}) was spin-coated onto the polymer layer from a 2-butanone solution (70 mg mL^{-1}) at 1500 rpm for 40 s and

subsequently dried at 80 ºC for 3 h.[S5] Finally, 100 nm Ag was evaporated on top as the gate electrode through a shadow mask.

Current–voltage characteristics were measured at room temperature under vacuum with a KEYSIGHT B2902A and a semiconductor parameter analysis software (SYSTEMHOUSE SUNRISE). Threshold voltages were estimated from the transfer plots by extrapolating the square root of the drain current to the horizontal axis. Hole and electron field-effect mobilities were extracted from the square root of the drain current in the saturation regime by using the following equation,

$$\mu = \frac{2L}{WC_i}\left(\frac{d\sqrt{|I_d|}}{dV_g}\right)^2$$

where, L (25 μm) and W (10000 μm) are channel length and width, respectively, and C_i is capacitance of the gate insulator. The average hole and electron mobilities and threshold voltages were obtained over more than five devices.

6. Supporting References.

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7. Supporting figures and tables.

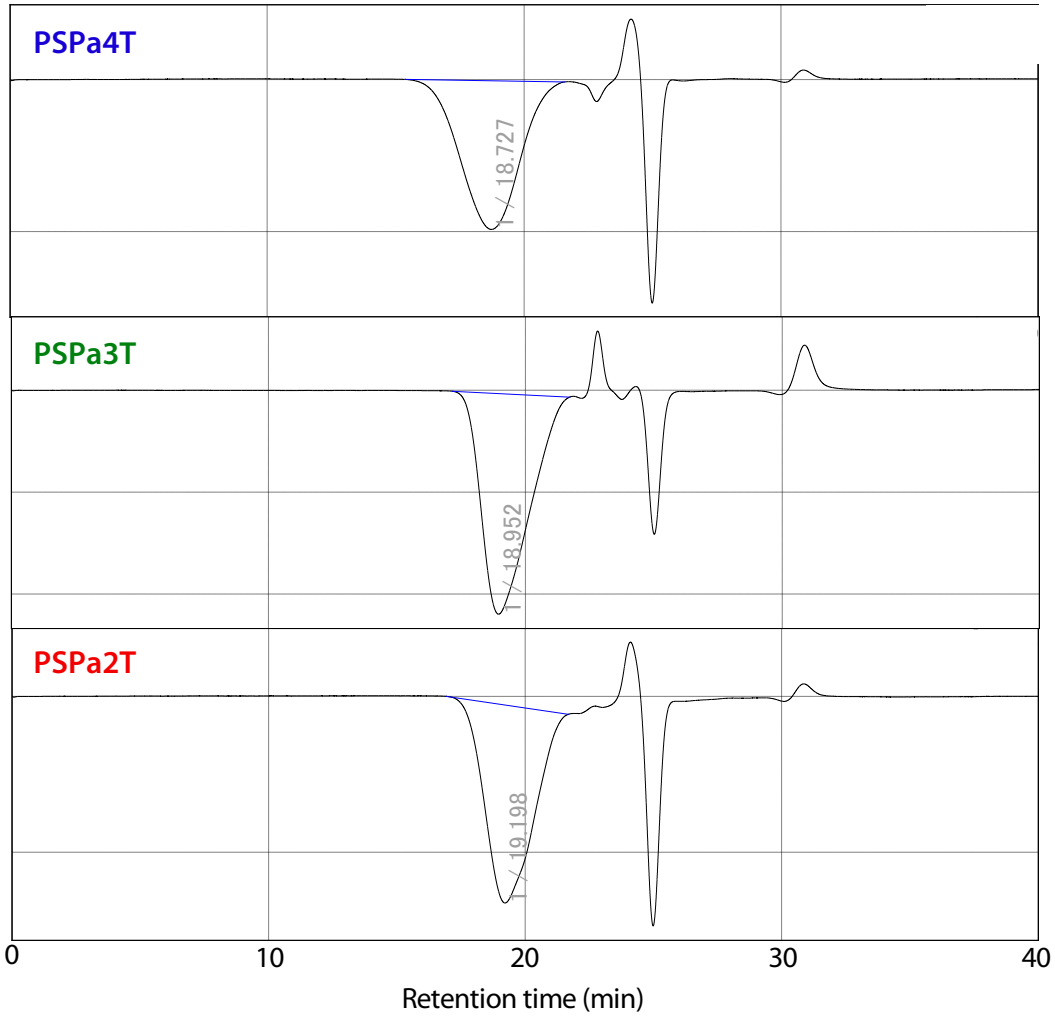

Fig. S1 GPC chart for the SP-based polymers.

Table S1. Polymerization results of SP-based polymers.

Polymer	M_n[a]	M_w[a]	$Đ$[b]
PSPa4T	23,100	53,100	2.3
PSPa3T	18,700	29,900	1.6
PSPa2T	12,800	24,300	1.9

[a]Determined by high-temperature GPC using polystyrene standard and TCB as the eluent at 180 ℃. [b]Dispersity index (M_w/M_n).

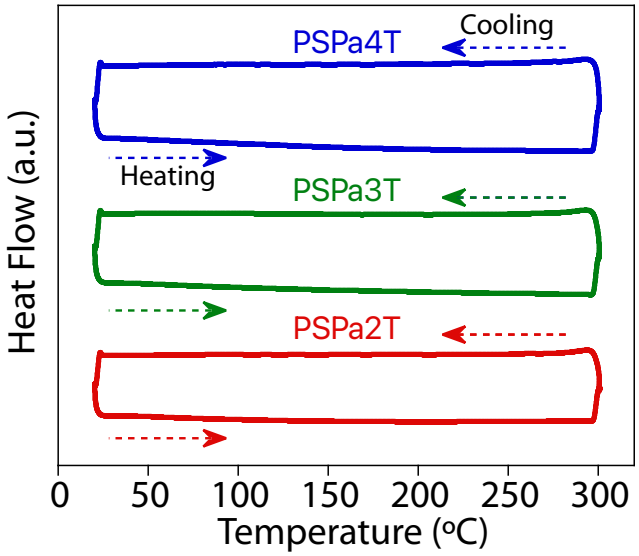

Fig S2 DSC curves of the SP-based polymers.

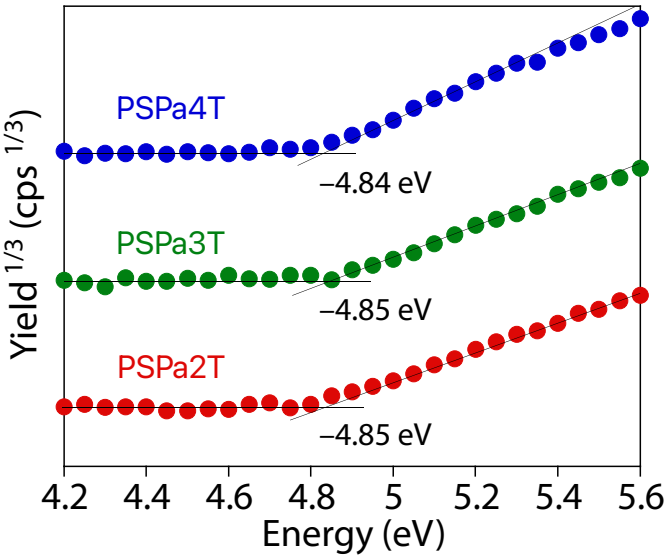

Fig. S3 PYS spectra of the SP-based polymers.

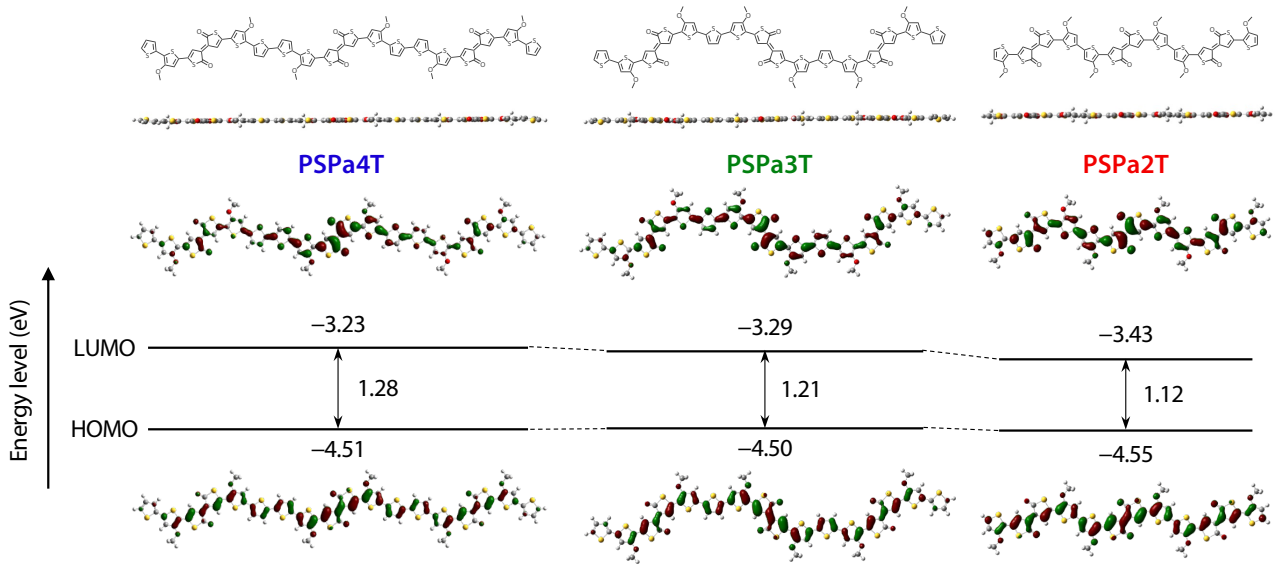

Fig. S4 Optimized molecular structure, side view, and calculated HOMO and LUMO geometries for the dimer models for **PSPa4T**, **PSPa3T**, and **PSPa2T**. Tahe long-branched DT groups were replaced by methyl groups to simplify the calculation. Calculations were carried out using the DFT method at the B3LYP/6-31G(d) level.

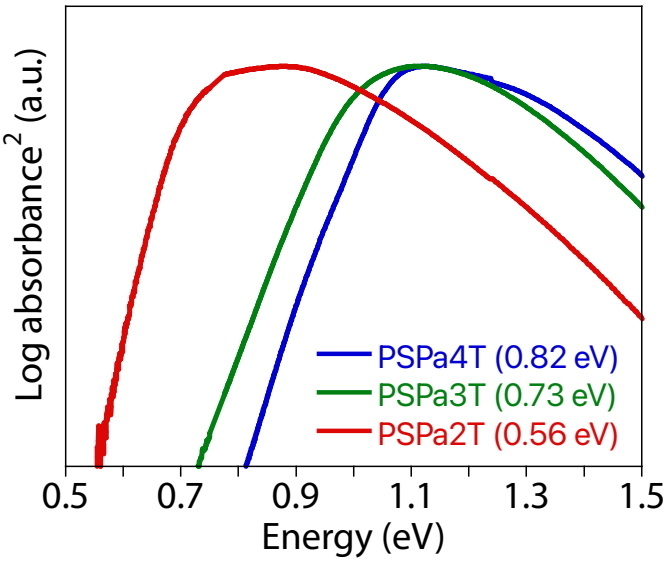

Fig. S5 Tauc plot of the polymers.

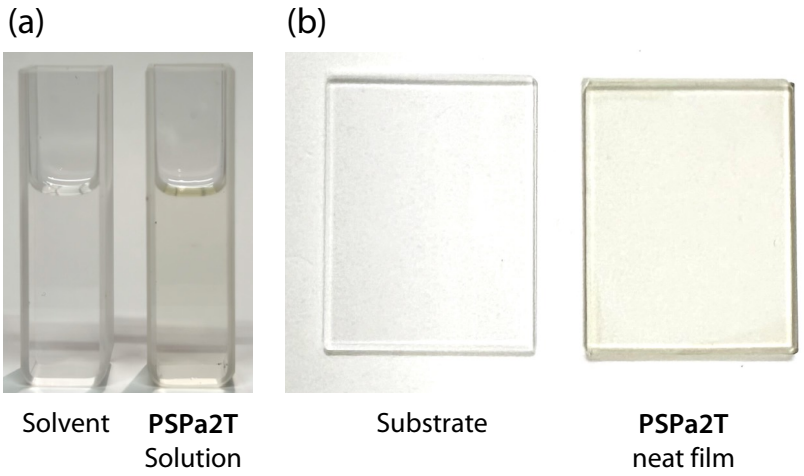

Fig. S6 Photos of (a) the polymer solution and (b) thin films in comparison with solvent and substrate, respectively.

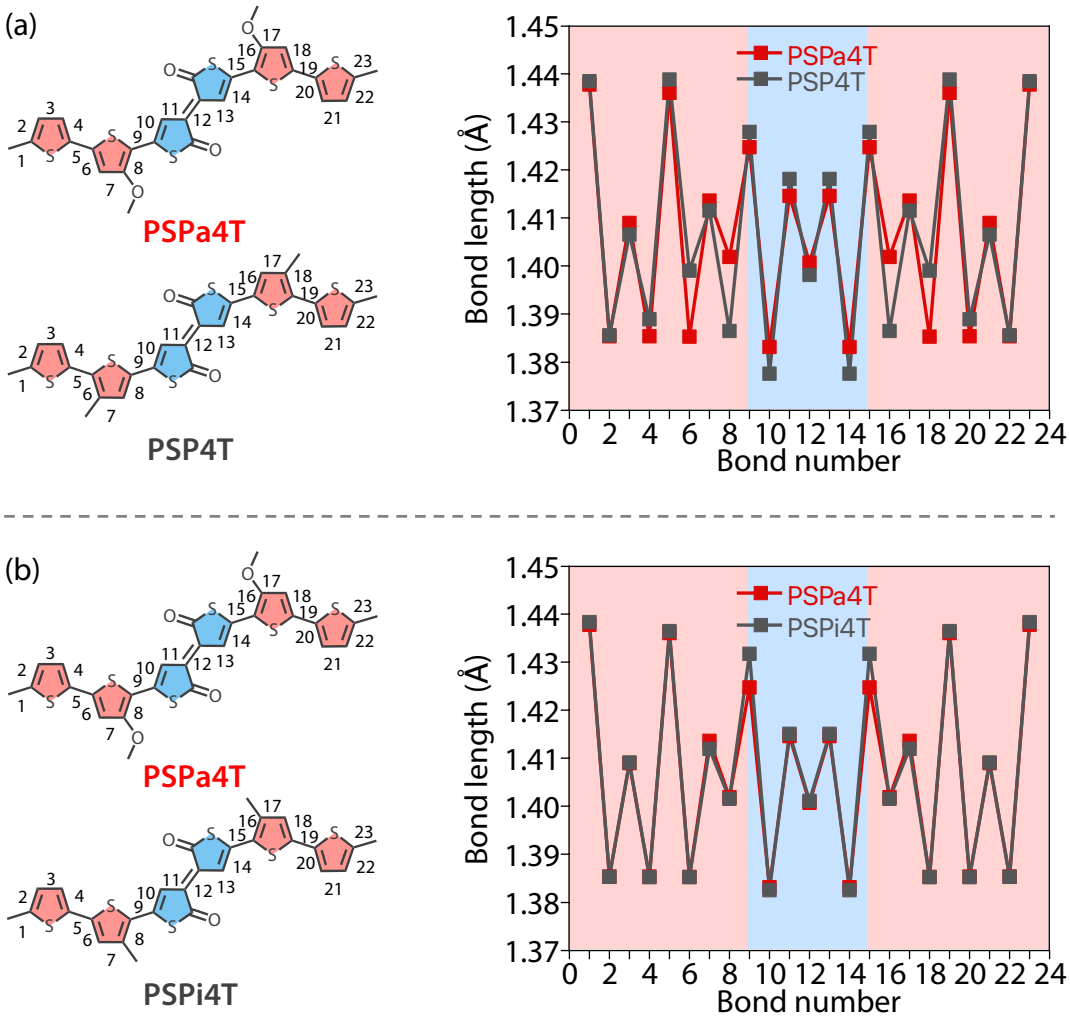

Fig. S7 (a,b) Plots of the lengths of carbon−carbon single bonds (bonds in even numbers) and double bonds (bonds in add numbers) at the respective bond number for (a) **PSP4T** and (b) **PSPi4T** in comparison with that for **PSPa4T**.

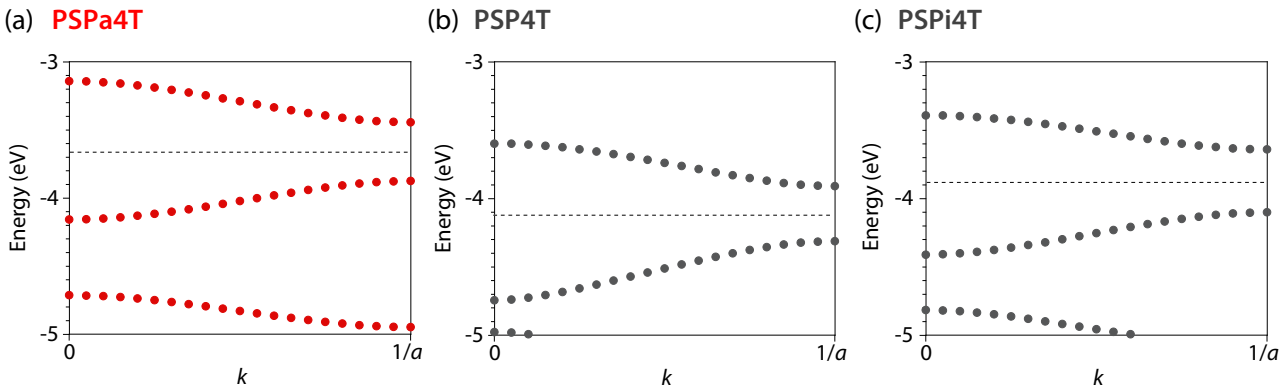

Fig. S8 Calculated band structure of (a) **PSPa4T**, (b) **PSP4T**, and (c) **PSPi4T**.

Table S2. Calculated effective mass of SP-based polymers

Polymer	m_h (m_0)[a]	m_e (m_0)[a]
PSPa4T	0.096	0.095
PSP4T	0.068	0.076
PSPi4T	0.099	0.120

[a]Effective masses for hole (m_h) and electron (m_e).