Electronic Supplementary Information

Effect of main and side chain chlorination on the photovoltaic properties of benzodithiophene-*alt*-benzotriazole polymers

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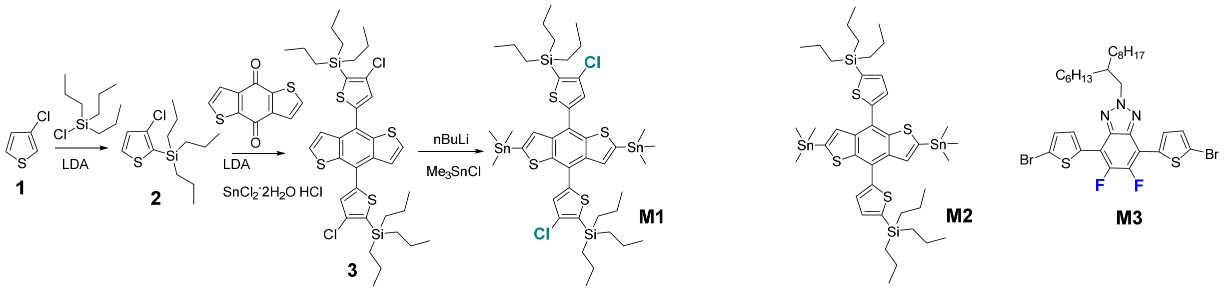
Experimental Section

Materials. The related chemical reagents and catalysts were purchased from Sigma Aldrich and TCI Chemical Co., monomer **M2**, **M3** and the acceptor IT4F were purchased from Solarmer Materials Inc. (Beijing, China). **M1**, **M4** and polymers were synthesized according to Schemes with the same procedure reported in the literature.[1–6]

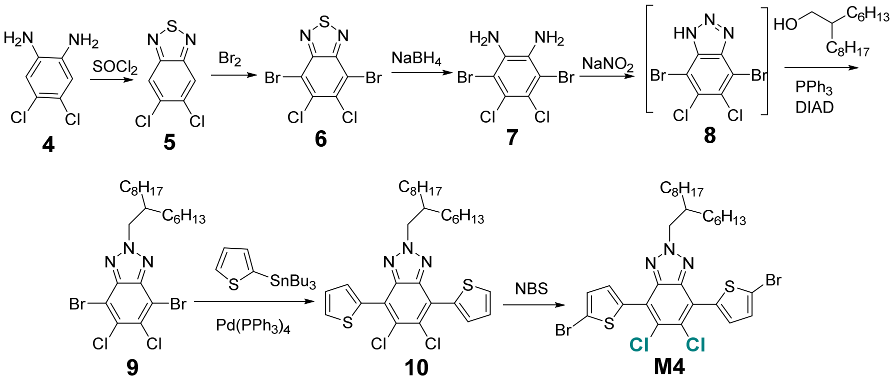


Scheme S1. Synthetic route to the monomer **M1**, and structures of **M2** and **M3**.

Synthesis of compound 2. Under argon protection, starting compound **1** (5.9 g, 50 mmol) and dry THF (100 mL) were mixed at −78 °C, then LDA (50 mmol, 25 mL, 2 M in THF) was slowly added and the mixture was stirred at −78 °C for 1 hour. Then chlorotripropylsilane (10.6 g, 55 mmol) was added and the mixture was stirred overnight at room temperature. The mixture was poured into water and extracted by diethyl ether and washed with water and brine. Removal of the solvent by rotary evaporation under reduced pressure, afforded a colorless liquid that was used without further purification.

Synthesis of compound 3. Under argon protection, to a solution of compound **2** (11.0 g, 40 mmol) in THF (60 mL) at −78 °C was slowly added LDA (20 mL, 2.0 M in THF), the mixture was kept at −78 °C for 2 hours. Then, benzo[1,2-*b*:4,5-*b*']dithiophene-4,8-dione (2.2 g, 10 mmol) was added quickly, and the mixture stirred at room temperature for 30 min. Then heated at 50 °C for 2 hours. After cooling down to room temperature, $SnCl_2.2H_2O$ (18 g, 80 mmol) in 10% HCl (80 mL) was added, and the mixture was stirred at 50 °C for 2 hours. The mixture was extracted by diethyl ether and washed by water and brine. The crude product was purified by column chromatography using petroleum ether as eluent to obtain pure compound **3** as a light yellow solid (5.52 g, yield 75%). ^1H NMR (400 MHz, $CDCl_3$) δ(ppm): 7.61-7.60 (d, 2H, J=4 Hz), 7.51-7.49 (d, 2H, J=8 Hz), 7.40 (m, 2H), 1.54-1.44 (m, 12H), 1.04-0.95 (m, 30H) (Fig. S9). MALDI-TOF MS: Calcd. for $C_{36}H_{48}Cl_2S_4Si_2$ m/z = 734.15 (1st peak) and 736.15 ((100% peak). Found m/z = 734.17 (1st peak) and 736.17 (100% peak) (Fig. S10).

Synthesis of M1. Under argon protection, to a solution of compound **3** (1.47 g, 2 mmol) in THF (30 mL) at −78 °C was added n-BuLi (2 mL, 2.5M in hexane) dropwise. After the addition, the mixture was kept at −78 °C for 40 min; chlorotrimethylstannane (6 mL, 1 M in THF) was added. The resulting mixture was stirred for 2 hours at room temperature. Then, it was poured into water and extracted with diethyl ether, washed by water and brine and after drying over MgSO4, the solvent was removed and the residue was recrystallized with methanol to afford a yellow solid (1.40 g, yield 67%). ^1H NMR (400 MHz, $CDCl_3$) δ(ppm): 7.69-7.60 (m, 2H), 7.43 (s, 2H), 1.49 (m, 12H), 1.07-0.95 (m, 30H), 0.51-0.33 (m, 18H) (Fig. S11). MALDI-TOF MS: Calcd. for $C_{42}H_{64}Cl_2S_4Si_2Sn_2$ m/z = 1054.08 (1st peak) and 1060.08 (100% peak). Found m/z = 1054.08 (1st peak), 1060.09 (100% peak) (Fig. S12).



Scheme S2. Synthetic route to monomer **M4**.

Synthesis of compound 5. To a solution of compound **4** (3.54 g, 20 mmol) in $CHCl_3$ (200 mL) and triethylamine (8.2 mL, 80 mmol), thionyl chloride(5.2 g, 40 mmol) was slowly added and the mixture was heated to reflux for overnight. The mixture was then cooled to room temperature and extracted with CH_2Cl_2, the organic layers were combined and dried over $MgSO_4$. The solvent was evaporated, and the product was obtained as white needle-like crystals (3.61 g, yield 88%).

Synthesis of compound 6. A mixture of compound **5** (4.08 g, 20 mmol), Br_2 (12.8 g, 80 mmol) and fuming sulfuric acid (100 mL) was stirred at 60 °C overnight. After cooling to room temperature, the mixture was poured into ice water (200 mL), then extracted with $CHCl_3$ and the organic layer was further washed by water, 1M NaOH and saturated $NaHCO_3$, and then dried over anhydrous $MgSO_4$. The solvent was removed via rotary evaporation, and the white solid obtained was further dried under high vacuum and used without further purification.

Synthesis of compound 7. To a stirred solution of compound **6** (3.62 g, 10 mmol) in ethanol (400 mL) at 0 °C was added portion-wise sodium tetrahydroborate (8.0 g, 210.5 mmol). After being stirred at 0 °C for 1 hour, the reaction mixture was slowly warmed to room temperature and stirred overnight. Then the reaction mixture was poured into cold water (200 mL), extracted with $CHCl_3$, and the organic layer was further washed by water and brine. The solvent was removed via rotary evaporation, and the white solid obtained was further dried under high vacuum and used without further purification.

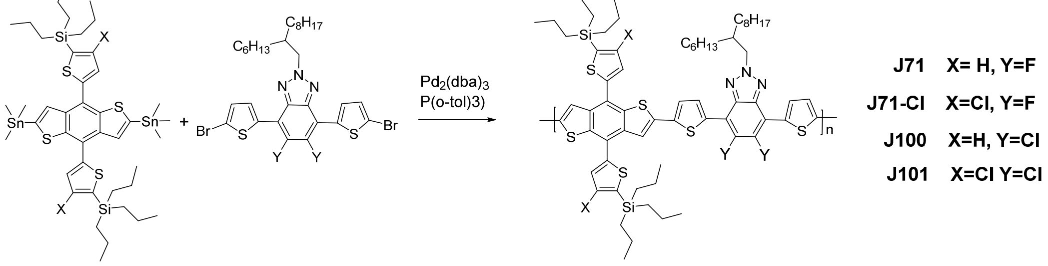
Synthesis of compound 8. Compound **7** (3.3 g, 10 mmol) was dissolved in 100 mL of AcOH at room temperature. A solution of $NaNO_2$ (2.76 g, 40 mmol) in H_2O (20 mL) was added slowly. The mixture was stirred at room temperature for 1 hour and precipitated into ice water. The resulting brown solid was filtered and washed by water, and further dried under high vacuum and used without further purification.

Synthesis of compound 9. Compound **8** (2.06 g, 6 mmol), 2-hexyldecan-1-ol (2.9 g, 12 mmol), and PPh_3 (3.15 g, 12 mmol) were dissolved in anhydrous THF (50 mL). Diisopropyl azodicarboxylate (DIAD) (2.43 g, 12 mmol) was added at 0 °C. After 30 min., the temperature was raised to room temperature and the mixture was stirred for 6 hours. The reaction mixture was then poured into water and extracted with ethyl acetate. The organic phase was washed with brine, dried with anhydrous $MgSO_4$, and then the solvent was removed under vacuum. The crude product was purified by column chromatography (silica gel, heptane/dichloromethane, 3:1, as eluent) to obtain **9** as a colorless oil (1.85 g, yield 54%). ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 4.66-4.64 (d, 2H, J=8 Hz), 2.34-2.25 (m, 1H), 1.34-1.18 (m, 24H), 0.85 (m, 6H) (Fig. S13). ^{13}C NMR (400 MHz, $CDCl_3$) δ(ppm): 142.31, 132.22, 111.43, 61.23, 39.07, 31.86, 31.67, 31.17, 31.14, 29.74, 29.42, 29.24, 26.02, 25.98, 22.67, 22.60, 14.12, 14.07 (Fig. S14).

Synthesis of compound 10. Compound **9** (2.75 g, 5 mmol) and tributyl(4-(2-ethylhexyl)thiophene-2-yl)stannane (6.1 g, 12.5 mmol) were dissolved in anhydrous toluene (30 mL). The solution was purged with argon for 15 min. and $Pd(PPh_3)_4$ (150 mg) was added at room temperature under argon protection. The mixture was heated up to 120 °C for 2 days in the sealed Schlenk tube. The solvent was evaporated under a vacuum, and the crude product was purified by column chromatography (silica gel, heptane/dichloromethane, 10:1, as eluent) to give compound **10** as a yellow solid (1.96 g. yield 68%). ^{1}H NMR (400 MHz, $CDCl_3$,) δ(ppm): 7.94-7.93 (dd, 2H, J_1=3.6 Hz, J_2=1.2 Hz), 7.57-7.56 (dd, 2H, J_1=5.2 Hz, J_2=1.2 Hz), 7.24-7.22 (t, 2H, J=4 Hz), 4.68 (d, 2H), 2.34-2.25 (m, 1H), 1.34-1.27 (m, 24H), 0.92 (m, 6H) (Fig. S15). ^{13}C NMR (400 MHz, $CDCl_3$) δ(ppm): 142.26, 135.09, 131.29, 129.47, 127.84, 126.70, 123.62, 60.28, 39.06, 31.88, 31.77, 31.38, 29.88, 29.55, 29.29, 28.28, 26.78, 26.17, 26.13, 22.66, 17.29, 14.10, 13.60 (Fig. S16). MALDI-TOF MS: Calcd. for $C_{30}H_{39}Cl_2N_3S_2$ m/z = 731.02 (1st peak) and 733.02 (100% peak). Found m/z = 731.01 (1st peak), 733.02 (100% peak) (Fig. S17).

Synthesis of compound M4. Compound **10** (1.15 g, 2 mmol) was dissolved in $CHCl_3$ (30 mL), then *N*-bromosuccinimide (0.79 g, 4.4 mmol) in DMF (10 mL) was added at 0 °C and stirred for 15 min. The mixture was then stirred at room temperature overnight and then quenched with water (200 mL). The solution was extracted with chloroform and washed by water and saturated

$Na_2S_2O_3$. The solvent was removed under reduced pressure, and the residue was further purified by silica column chromatography using dichloromethane and heptane ether (1:10) as eluent to afford **M4** as a yellow solid (1.32 g, yield 90%).%). ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 7.81-7.80 (d, 2H, *J*=4 Hz), 7.18-7.17 (d, 2H, *J*=4 Hz), 4.65-4.63 (d, 2H, *J*=8 Hz), 2.26-2.20 (m, 1H), 1.35-1.24 (m, 24H), 0.89-0.85 (m, 6H) (Fig. S18). ^{13}C NMR (400 MHz, $CDCl_3$) δ(ppm): 141.76, 136.54, 132.02, 129.65, 129.20, 122.79, 115.84, 60.27, 39.11, 31.89, 31.78, 31.38, 29.89, 29.56, 29.31, 26.18, 26.13, 22.68, 22.65, 14.12, 14.09 (Fig. S19). MALDI-TOF MS: Calcd. for $C_{30}H_{37}Br_2Cl_2N_3S_2$ *m/z* = 731.02 (1st peak) and 733.02 (100% peak). Found *m/z* = 731.01 (1st peak), 733.02 (100% peak). (Fig. S20).



Scheme S3. Synthetic route to polymers.

Synthesis of polymers, general procedure. BDT monomer (0.20 mmol) and BTz monomer (0.20 mmol) and dry toluene (10 mL) were added to a 50 mL Schlenk tube. The reaction container was purged with argon for 20 min., and then $Pd_2(dba)_3$ (2.4 mg) and $P(o\text{-}tol)_3$ (1.1 mg) were added. After another flushing with argon for 20 min., the reaction mixture was heated to reflux for overnight, then the mixture was cooled down to room temperature and poured into MeOH (100 mL) and filtered. The polymer was collected by filtration through a Soxhlet extractor and then subjected to Soxhlet extractions with methanol, hexane, and chloroform. The chloroform fraction was concentrated and precipitated with methanol, affording the polymers as purple-black solids.

J71: M_n = 28.2 kDa; PDI=2.16. ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 8.51-7.74 (br, 2H), 7.86-7.31 (br, 6H), 7.14-6.80 (br, 2H), 4.70 (br, 2H), 2.21 (br, 1H), 1.63-0.84 (br, 72H).

J71-Cl: M_n = 18.9 kDa; PDI=1.94. ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 8.17-8.16 (br, 2H), 7.52-7.33(br, 4H), 7.23-7.00 (br, 2H), 4.75 (br, 2H), 2.29 (br, 1H), 1.61-0.82 (br, 72H).

J100: M_n = 35.5 kDa; PDI=2.71. ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 8.53-7.89 (br, 2H), 7.87-7.33 (br, 6H), 7.05-6.70 (br, 2H), 4.66 (br, 2H), 2.28 (br, 1H), 1.61-0.85 (br, 72H).

J101: M_n = 24.6 kDa; PDI=2.31. ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 8.49-7.91 (br, 2H), 7.76-7.37 (br, 4H), 7.09-6.81 (br, 2H), 4.68 (br, 2H), 2.28 (br, 1H), 1.52-0.82 (br, 72H).

Material characterizations. ^{1}H NMR spectra were measured on a Bruker DMX–400 spectrometer with *d*–chloroform as the solvent and tetramethyl silane as the internal reference. Matrix assisted laser desorption ionization time of flight (MALDI-TOF) mass spectrometry was performed on a Bruker Autoflex Speed spectrometer. Polymer molecular-weight distributions were estimated by GPC at 140 °C on a PL-GPC 120 system using a PL-GEL 10 μm MIXED-C column with *o*-DCB as the eluent and calibrated by polystyrene internal standards. Samples were first dissolved in *o*-DCB at 140 °C in a concentration of 0.1 mg mL^{-1}. UV-visible spectroscopy was recorded on a Perkin Elmer Lambda 1050 UV/vis/near IR spectrophotometer at room temperature. All solution UV-vis experiments were performed in diluted chloroform (CF) solutions. Films were prepared by spin coating CF solutions on glass substrates. Square-wave voltammetry (SWV) studies were performed with a scan rate of 0.1 V s^{-1} under an inert atmosphere with 1 M tetrabutylammonium hexafluorophosphate in acetonitrile as the electrolyte. All potentials were reported versus the ferrocene/ferrocenium redox couple (Fc/Fc^+). The morphologies of films were characterized by a Dimension 3100 AFM with tapping mode.

Device fabrication and characterization. OSCs devices were fabricated and characterized in a N_2-filled glovebox. Pre-structured ITO coated glass substrates were cleaned in acetone and isopropyl alcohol for 10 min each. After drying, the substrates were coated via spin-coating with 40 nm PEDOT:PSS (Heraeus, Clevios P VP.Al 4083). The active layer was spin-coated in a N_2-filled glovebox. The methanol solution of PDINO at a concentration of 1.0 mg mL^{-1} was deposited atop the active layer. Finally, top Al electrode was deposited in vacuum onto the cathode buffer layer at a pressure of ≈5.0×10^{-7} Pa.

Mobility measurement. The hole and electron mobilities were calculated by using the space-charge-limited current (SCLC) method. $J \cong (9/8)\varepsilon\varepsilon_0\mu_0 V^2 \exp(0.89\sqrt{V/E_0 L})/L^3$ (1), where ε is the dielectric constant of the polymers, ε_0 is the permittivity of the vacuum, μ_0 is the zero-field mobility, E_0 is the electric field, J is the current density, and L is the thickness of the film.

GIWAXS Characterization. Grazing-incidence wide-angle X-ray scattering (GIWAXS) measurements were conducted at Advanced Light Source (ALS), Lawrence Berkeley National Laboratory, Berkeley, CA at the beamline 7.3.3. Data was acquired at the critical angle (0.13°) of the film with a hard X-ray energy of 10 keV. X-ray irradiation time was 10 s, dependent on the saturation level of the detector.

R-SoXS Characterization. R-SoXS measurements were performed in transmission geometry with linearly polarized photons under high vacuum (1×10^{-7} Torr) at beamline 11.0.1.2 at the Advanced Light Source (ALS). The scattering 2D images were collected in a vacuum cooled 2D charge-coupled device (CCD) detector (Princeton Instruments, PI-MTE, 2048×2048 pixels). The soft X-ray energies were tuned between 270 and 285 eV in the R-SoXS experiments. Using a custom Nika analysis package, 2D scattering patterns were reduced to 1D scattering profiles and normalized for the X-ray flux. Since distance travelled through the samples by the X-rays affects the scattering intensity, it was normalized for absorption and film thickness. Samples for R-SoXS measurements were first prepared on a polystyrene sulfonate (PSS) modified Si substrate and floating in water to a 1.5 × 1.5 mm, 100 nm thick Si_3N_4 membrane supported by a 5 × 5 mm, 200 um thick Si frame.

Additional Figures and Tables

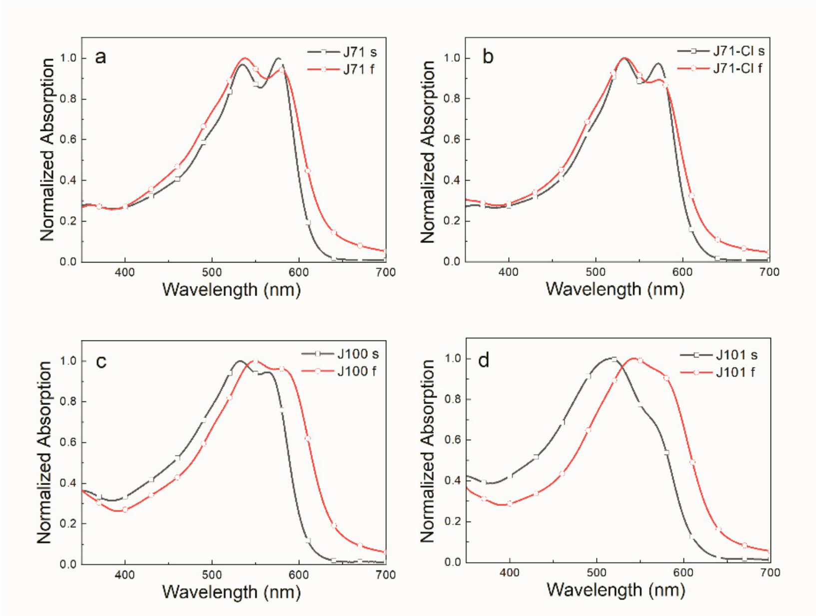


Fig. S1. Normalized absorption spectra of the four polymers in dilute solution and in thin film.

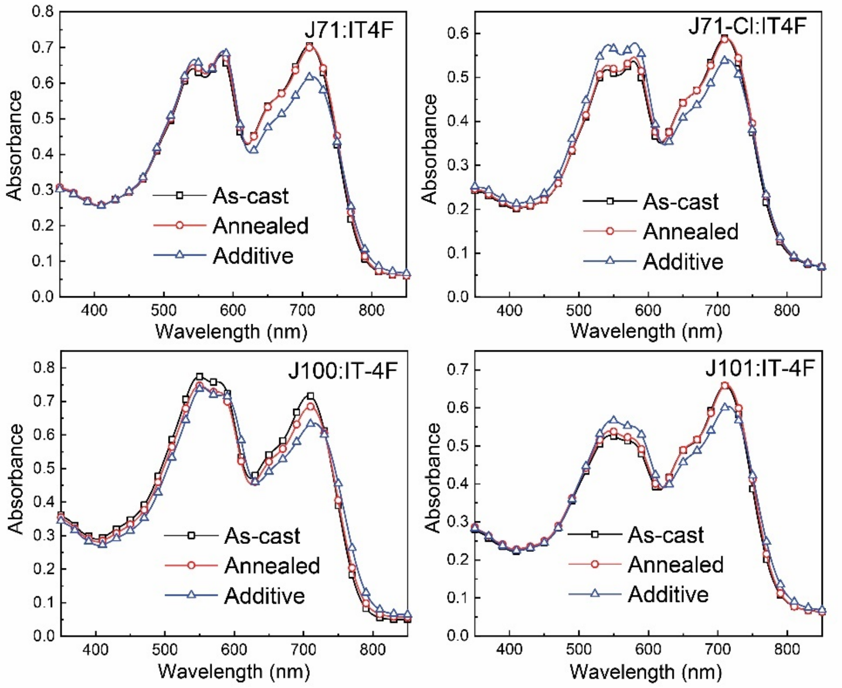


Fig. S2. Absorption spectra of the four blend films without and with post-treatment.

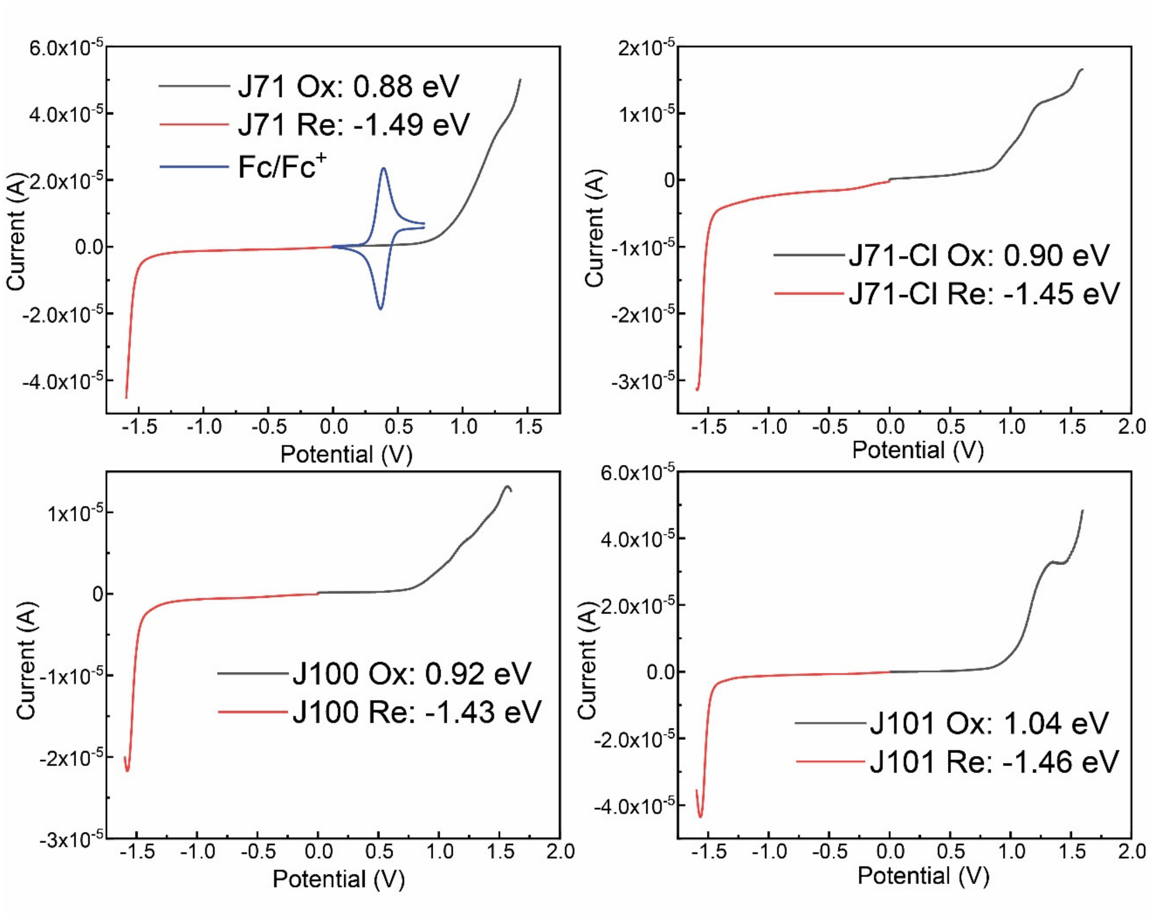


Fig. S3. Square-wave voltammograms of J71, J71-Cl, J100, and J101. Potentials are vs. Ag/AgCl.

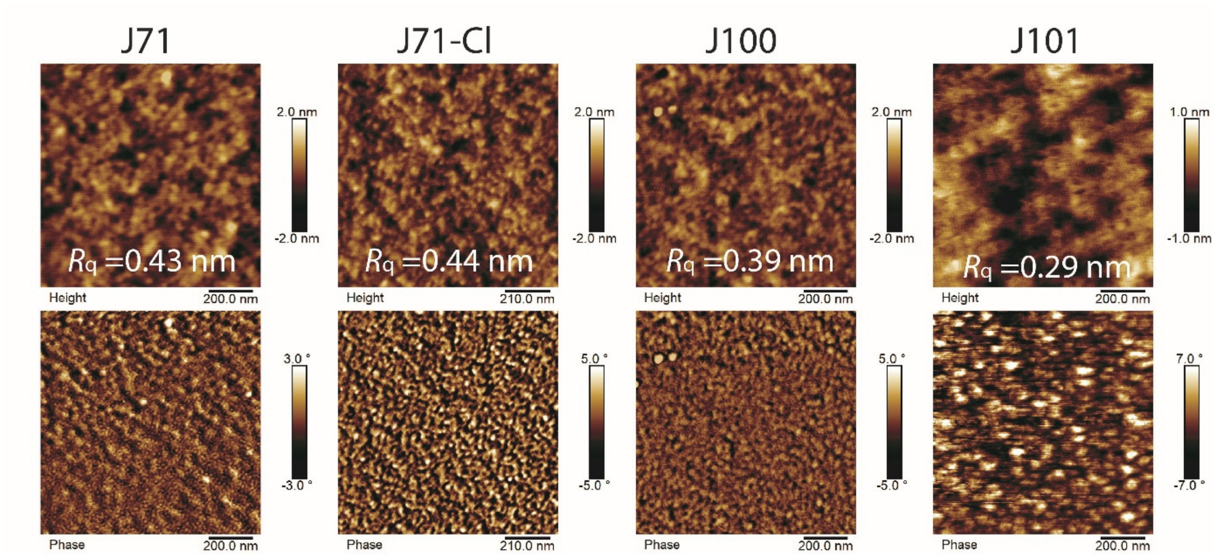


Fig. S4. AFM images of J71, J71-Cl, J100, and J101.

Note S1. Note on the signal intensities of OOP and IP signals in GIWAXS

A 2D powder of a conjugated polymer has an isotropic distribution of the π-π and the lamellar stacking directions, *i.e.* in- and out-of-plane orientations (and every orientation in between) are equally probable. The same holds for any in-plane angle of the polymer backbone. In the in-plane scattering geometry, X-rays record only those molecules with angles aligned that fulfill diffraction conditions. In the out-of-plane direction, all backbone orientations are recorded. The correction factor is sin(χ), where χ is the azimuthal angle, defined such that $\chi = 0°$ is the out-of-plane (q_z) axis, while $\chi = 90°$ is the in-plane (q_{xy}) axis. The lamellar and π-π scattering will look exactly the same in the raw data due to the random 2D powder. Because the orientation distribution is constant, the actual data of a 2D powder will give a signal intensity proportional to 1/sin(χ) signal in the raw WAXS data, i.e. high at $\chi = 0°$ and lower at $\chi = 90°$. Hence, the 1/sin(χ) term causes *apparent* (100) *and* (010) peaks in the out-of-plane direction and lower intensity in the in-plane direction, even though the orientation probability is constant. This fact can result in an *apparent* simultaneous "edge-on" and "face-on" orientation if simply the raw out-of-plane intensities are used for interpretation, which makes no sense and is a misinterpretation because the sin(χ) correction and overall symmetry is neglected. For further information we refer readers to the website: http://gisaxs.com/index.php/Example:P3HT_orientation_analysis

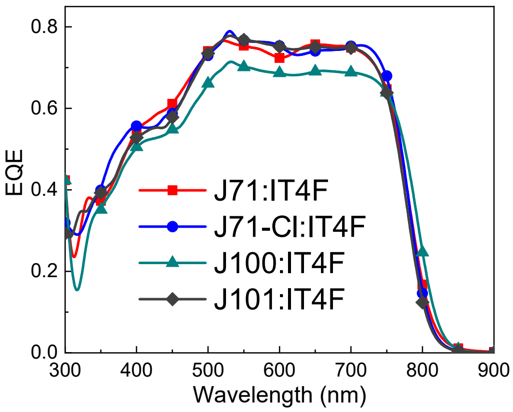


Fig. S5. EQE curves for the optimized OSCs based on J71, J71-Cl, J100 and J101.

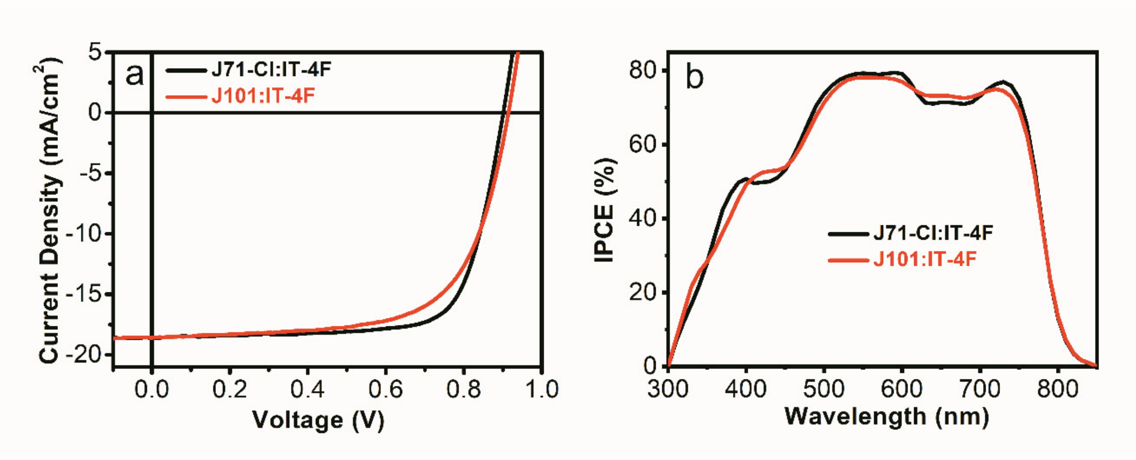


Fig. S6. (a) $J-V$ characteristics of PSCs based on J71-Cl, J101 as donor, and IT4F as acceptor with PNDIT-F3N as cathode interlayer and Ag as the cathode (b) Corresponding EQE spectra.

Table S1. Photovoltaic parameters of the PSCs with PNDIT-F3N as cathode interlayer and Ag as the cathode.

Blend	J_{sc} (mA cm^{-2})	V_{oc} (V)	FF (%)	PCE (%)	J_{sc}^{EQE} (mA cm^{-2})	PCE^{EQE} (%)
J71-Cl:IT4F	18.59	0.90	74.0	12.4	18.21	12.1
J101:IT4F	18.60	0.92	66.0	11.3	18.07	11.0

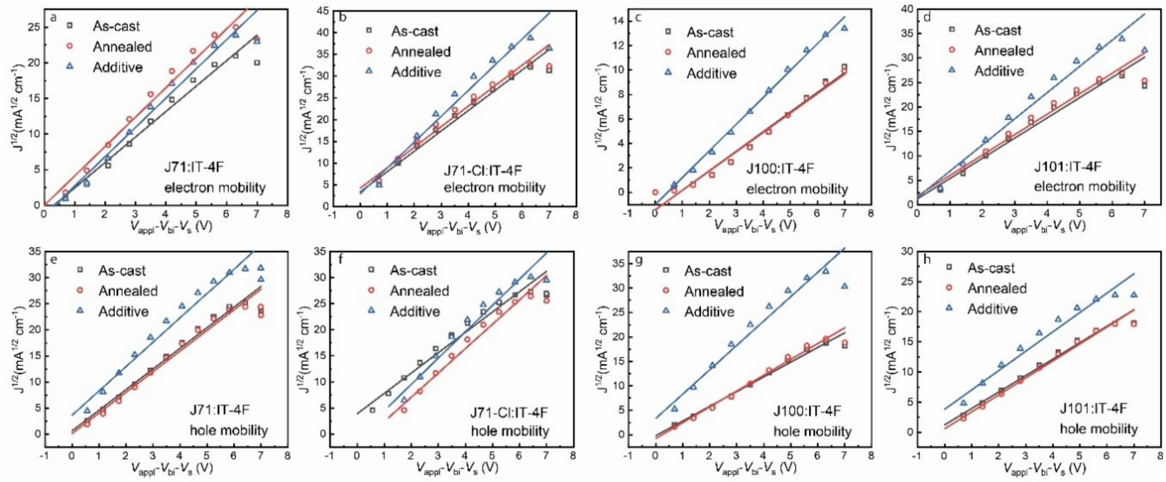


Fig. S7. $J^{1/2}$-V characteristics of the devices. Solid lines are the fitting lines of the data.

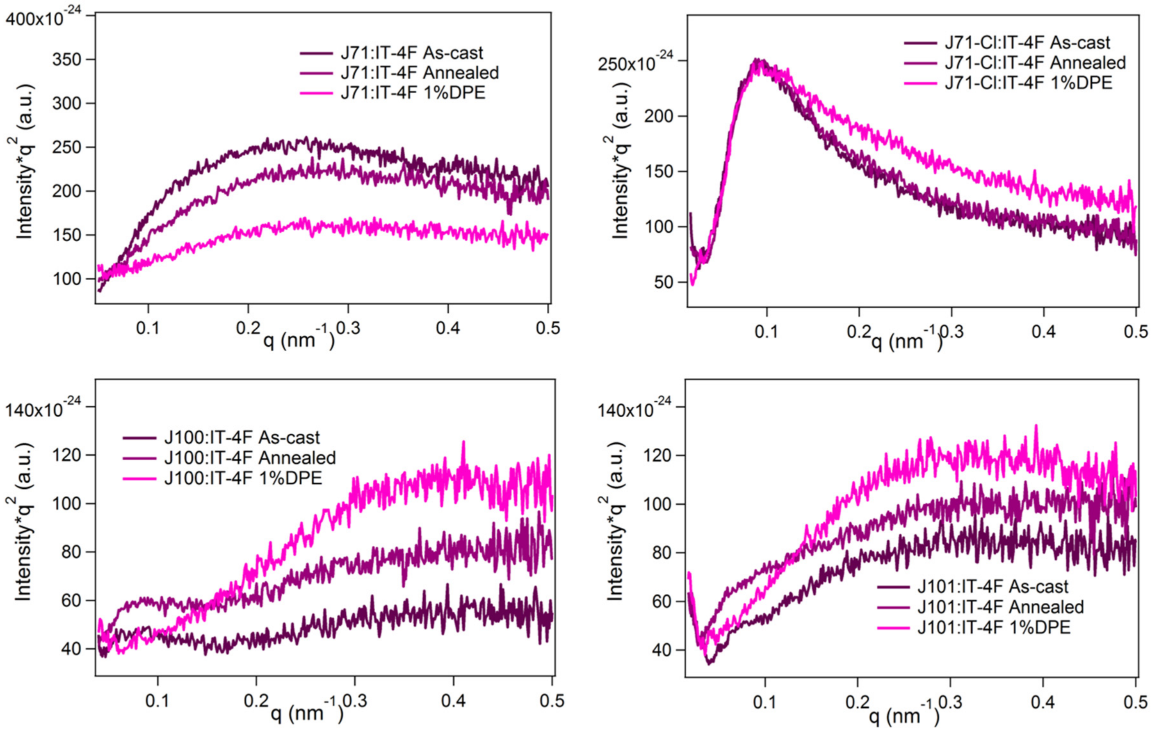


Fig. S8. R-SoXS 1-D profiles at 284.3 eV of the four series bend films.

Table S2. R-SoXS parameters of blend films without and with post-treatment

Blend	Processing	Normalized total ISI	CSI of high *q* peak	FF
J71:IT4F	as cast	0.93	0.93	0.50
	annealed	1.00	1.00	0.52
	additive	0.80	0.80	0.50
J71-Cl:IT4F	as cast	0.39	0.24	0.57
	annealed	0.39	0.27	0.60
	additive	0.44	0.33	0.69
J100:IT4F	as cast	0.22	0.21	0.46
	annealed	0.21	0.21	0.52
	additive	0.41	0.41	0.57
J101:IT4F	as cast	0.21	0.21	0.46
	annealed	0.20	0.20	0.48
	additive	0.26	0.26	0.67

Table S3. The GIWAXS data of the blend films in the out-of-plane direction

Blend	Processing	π-π stacking distance (OOP)		π-π stacking coherence (OOP)		Integrated Intensity (π-π peak)	*g* Parameter
		q (Å^{-1})	*d* (Å)	Δ*q* (Å^{-1})	CCL (Å)		
J71:IT4F	as cast	1.72	3.65	0.28	22	7.09×10^{-7}	16.2
	annealed	1.74	3.61	0.28	23	1.01×10^{-6}	15.9
	additive	1.72	3.65	0.27	23	5.75×10^{-8}	15.9
J71-Cl:IT4F	as cast	1.70	3.70	0.28	22	8.44×10^{-7}	16.2
	annealed	1.70	3.70	0.32	20	8.89×10^{-7}	17.3
	additive	1.71	3.67	0.23	28	1.12×10^{-6}	14.6
J100:IT4F	as cast	1.70	3.70	0.224	28	1.27×10^{-6}	14.5
	annealed	1.71	3.67	0.296	21	4.07×10^{-6}	16.6
	additive	1.71	3.67	0.271	23	1.76×10^{-6}	15.9
J101:IT4F	as cast	1.68	3.74	0.3	21	1.13×10^{-6}	16.9
	annealed	1.69	3.72	0.29	22	9.02×10^{-7}	16.5
	additive	1.69	3.72	0.268	23	1.75×10^{-6}	15.9

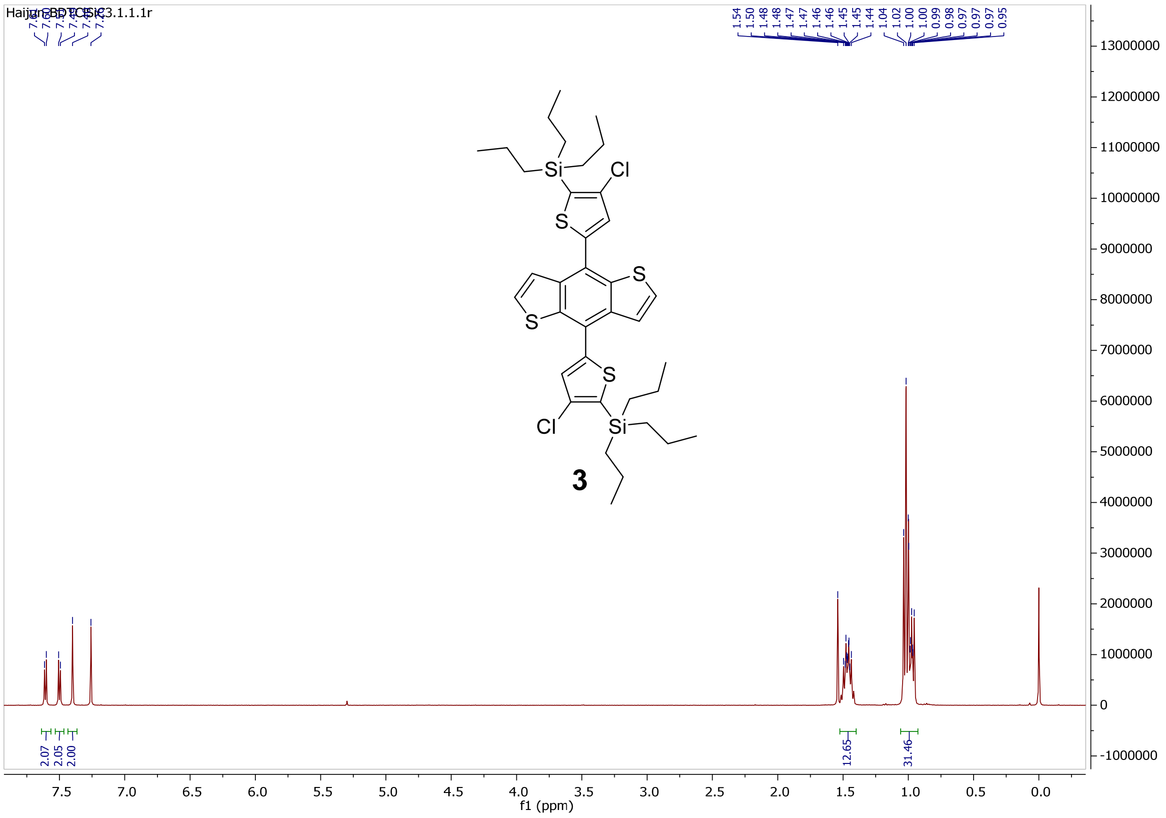


Fig. S9. ^{1}H NMR of compound **3**.

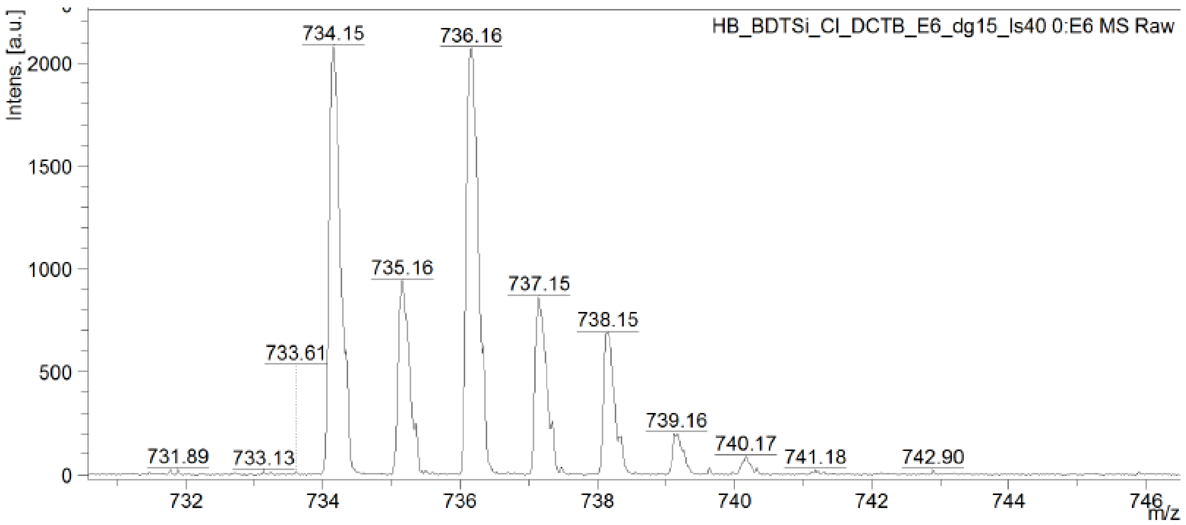


Fig. S10. The MALDI-MS of compound **3**. Calcd. for $C_{36}H_{48}Cl_2S_4Si_2$ *m/z* = 734.15 (1st peak) and 736.15 (100% peak). Found *m/z* = 734.15 (1st peak) and 736.16 (100% peak).

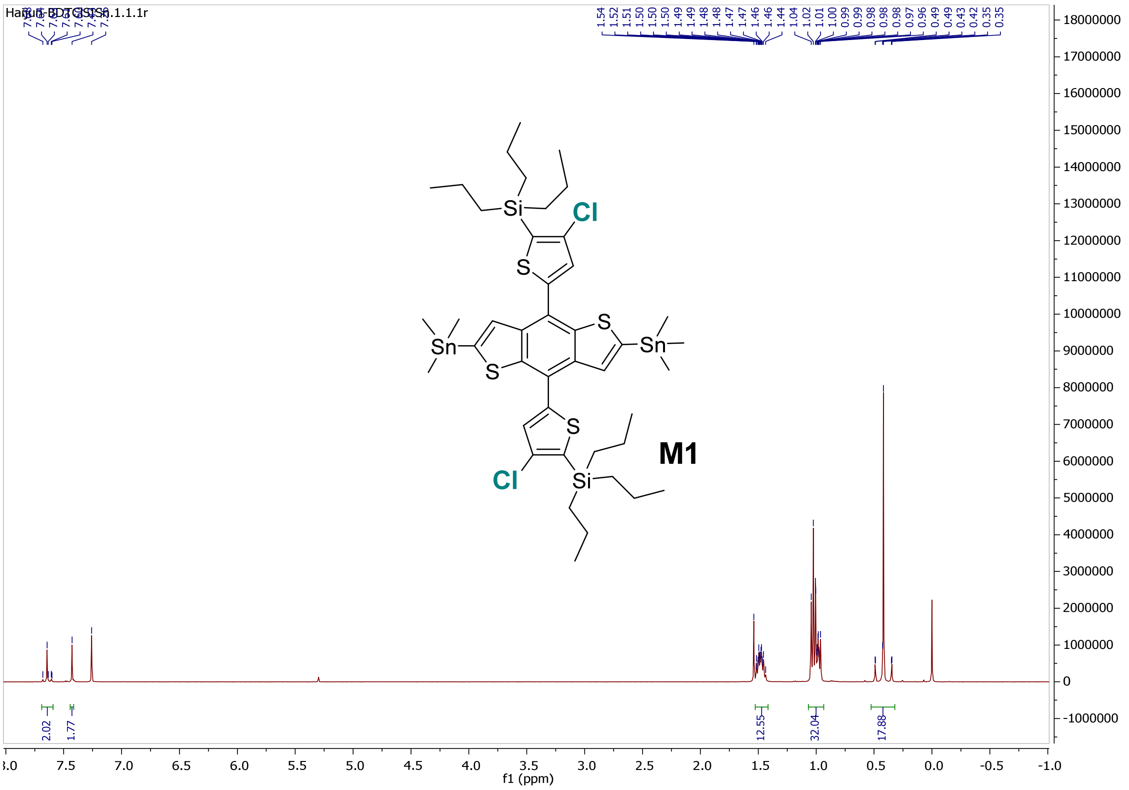


Fig. S11. ^{1}H NMR of monomer **M1**.

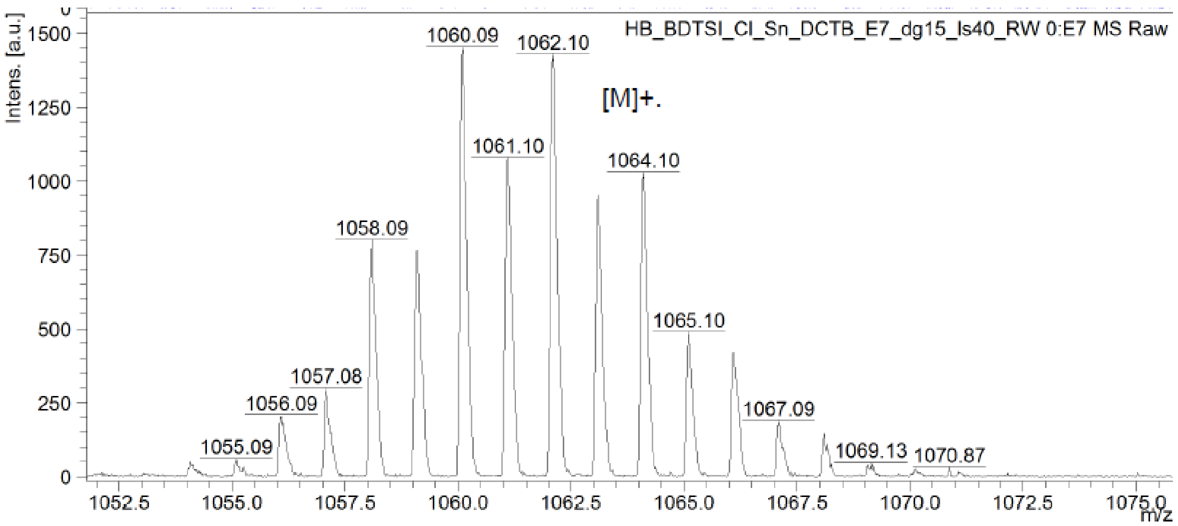


Fig. S12. The MALDI-MS of monomer **M1**. Calcd. for $C_{42}H_{64}Cl_2S_4Si_2Sn_2$ *m/z* = 1054.08 (1st peak) and 1060.08 (100% peak). Found *m/z* = 1054.08 (1st peak), 1060.09 (100% peak).

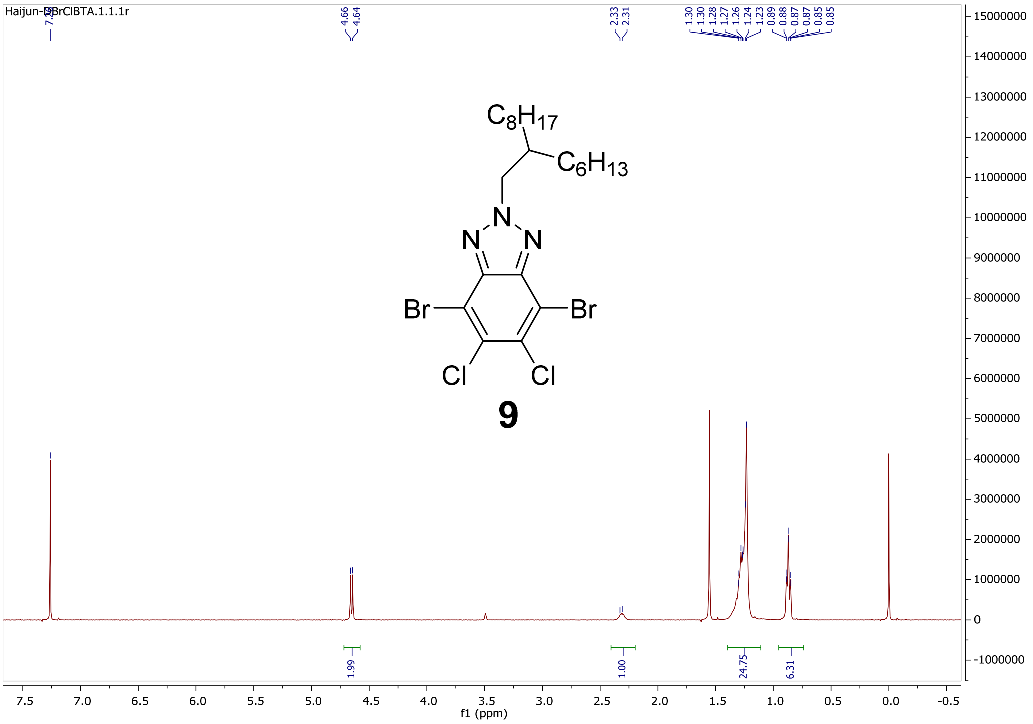


Fig. S13. ^{1}H NMR of compound **9**.

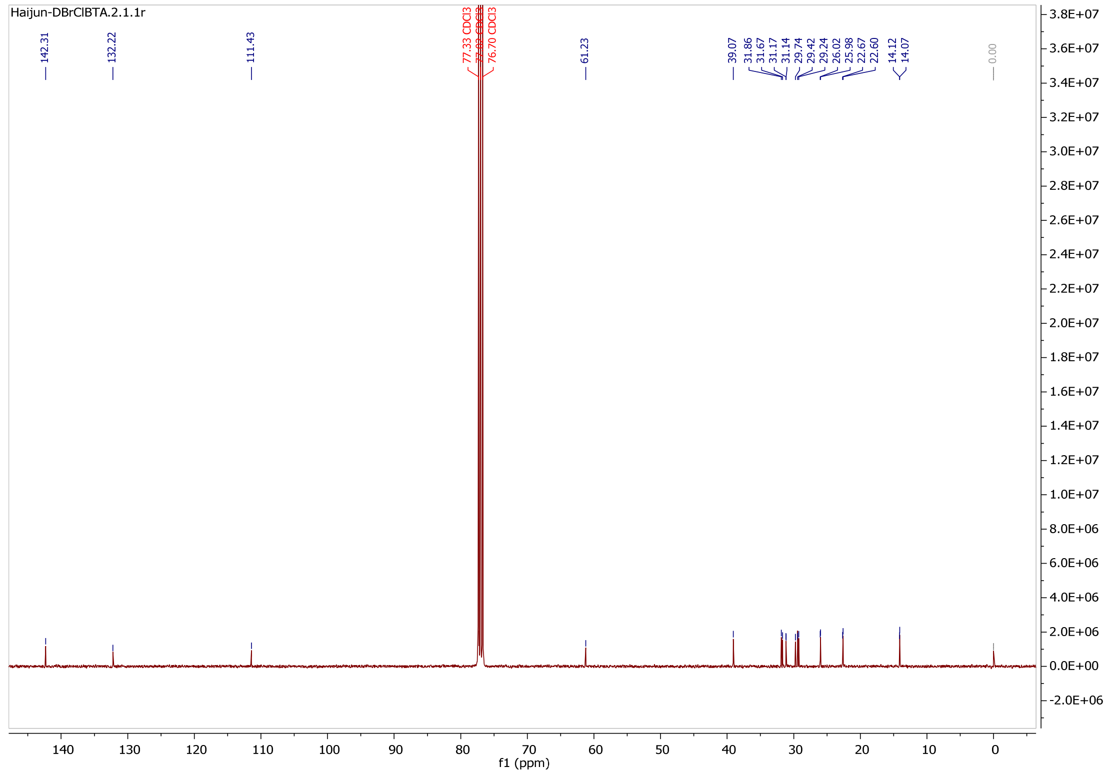


Fig. S14. ^{13}C NMR of compound **9**.

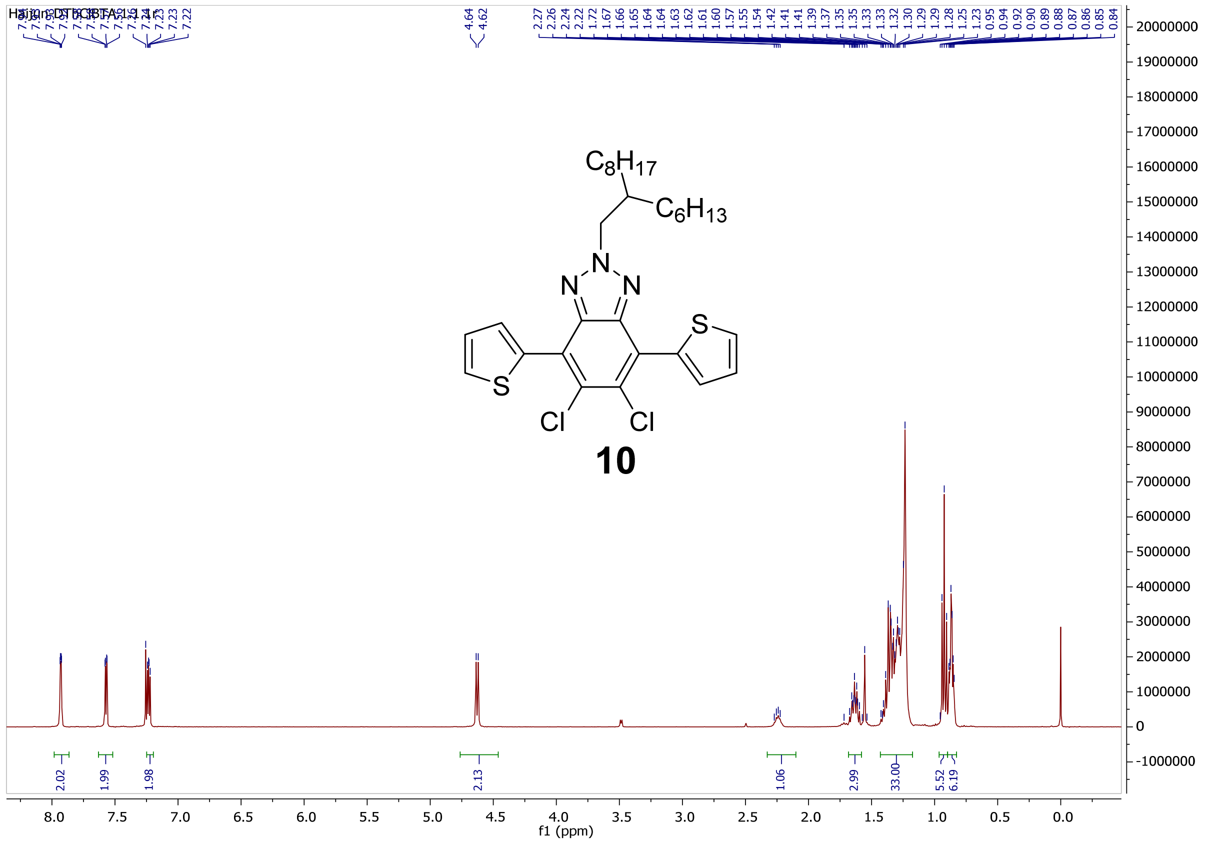


Fig. S15. ^{1}H NMR of compound **10**.

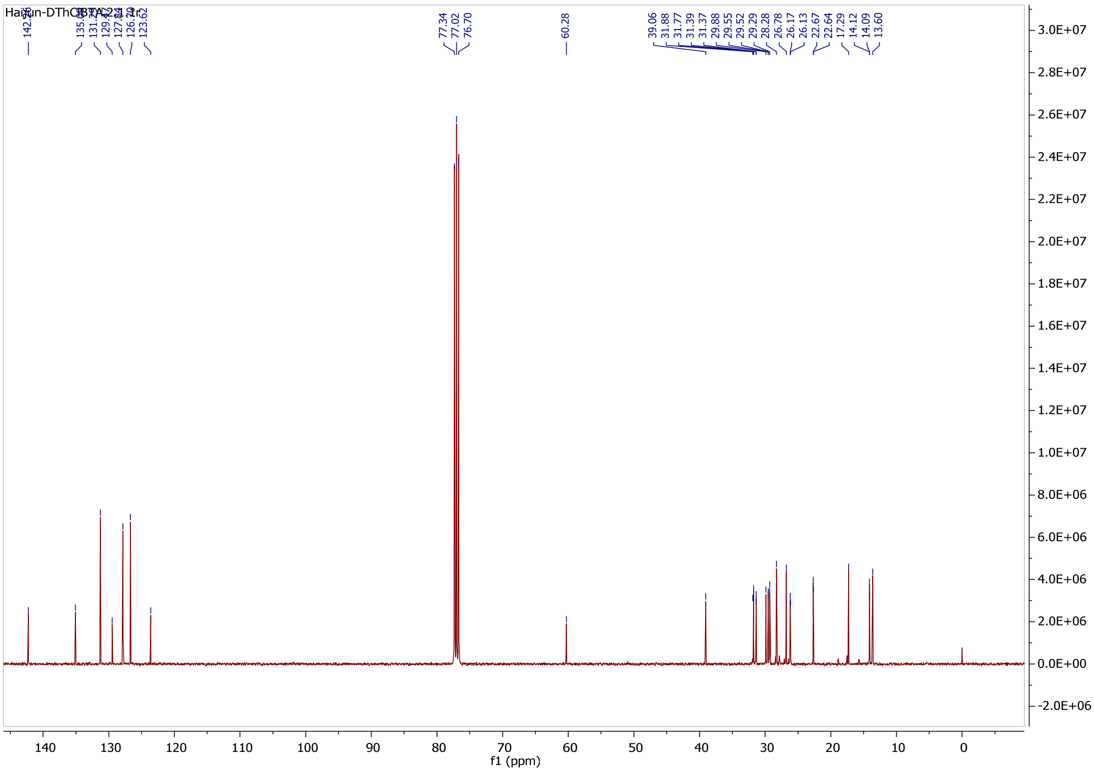


Fig. S16. ^{13}C NMR of compound **10**.

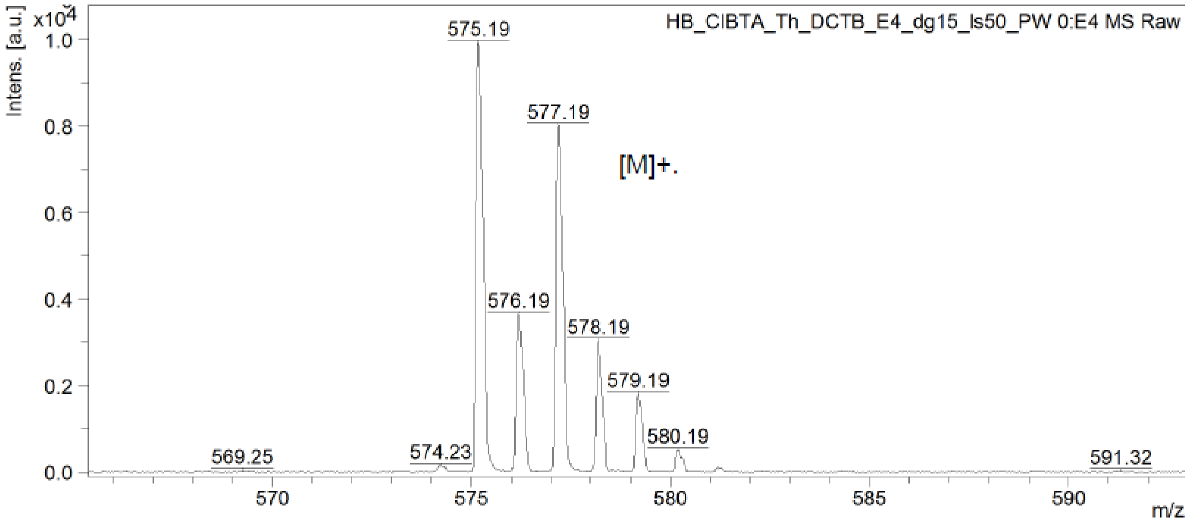


Fig. S17. The MALDI-MS of compound **10**. Calcd. for $C_{30}H_{39}Cl_2N_3S_2$ *m*/*z* = 575.20 (1st and 100% peak); Found *m*/*z* = 575.19. (1st and 100% peak).

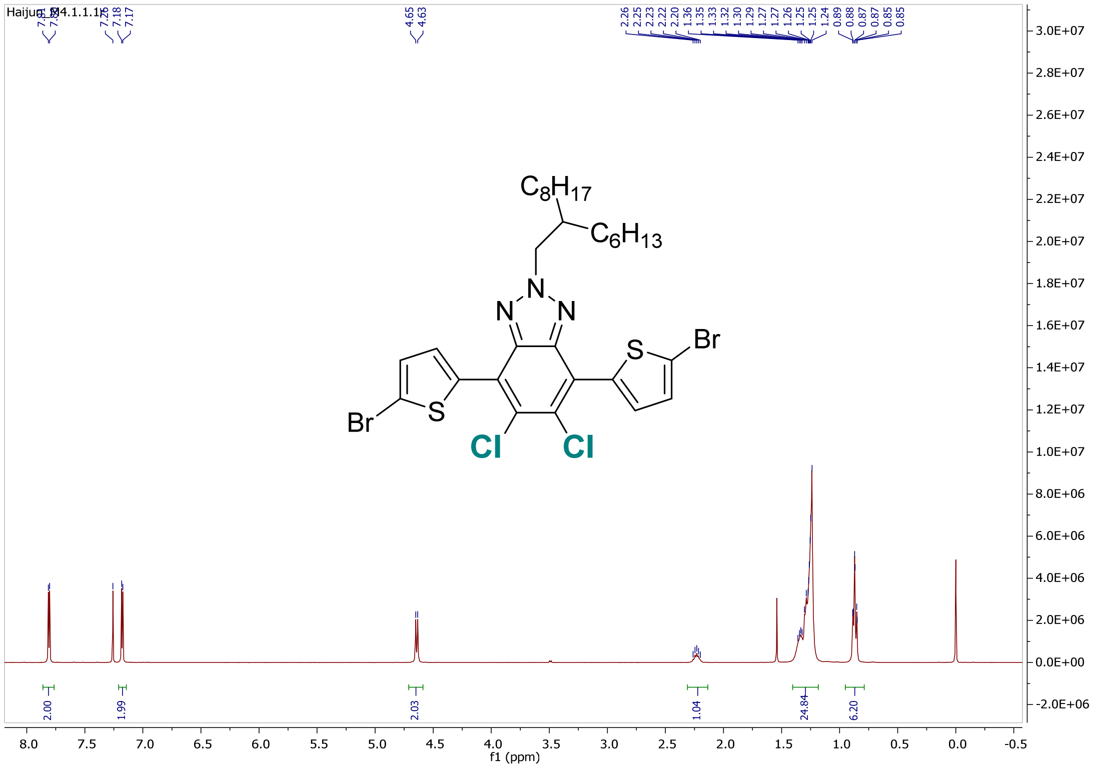


Fig. S18. ^{1}H NMR of monomer **M4**.

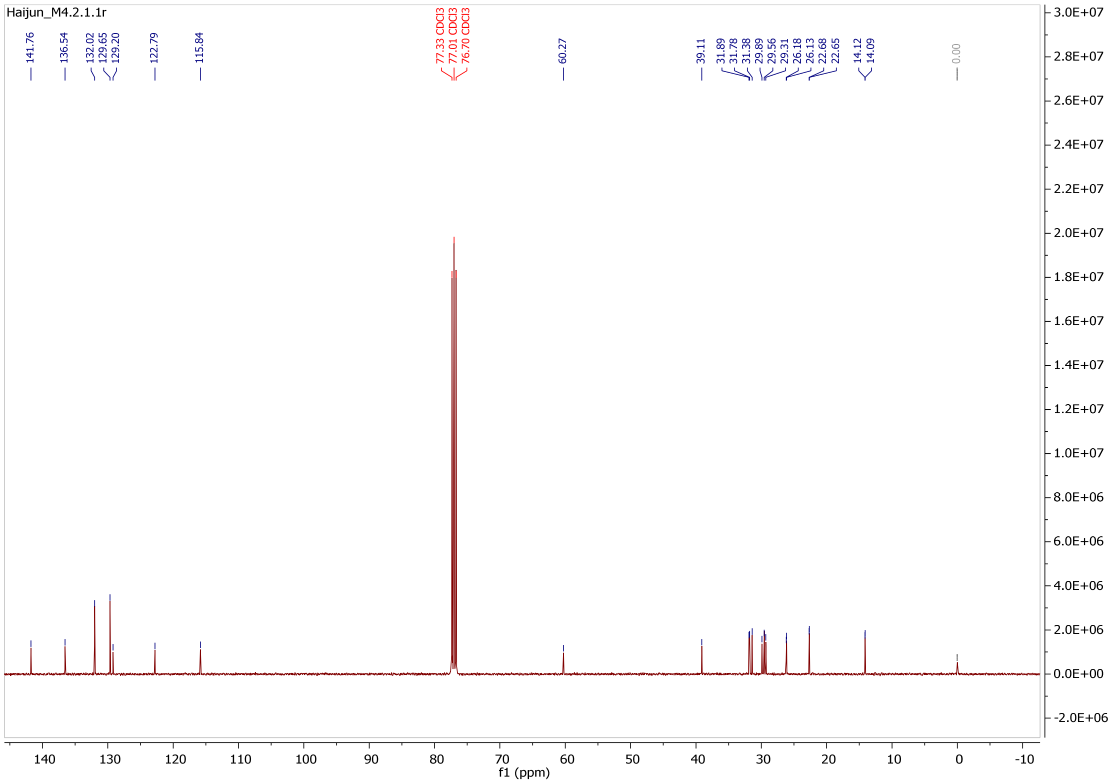


Fig. S19. ^{13}C NMR of monomer **M4**.

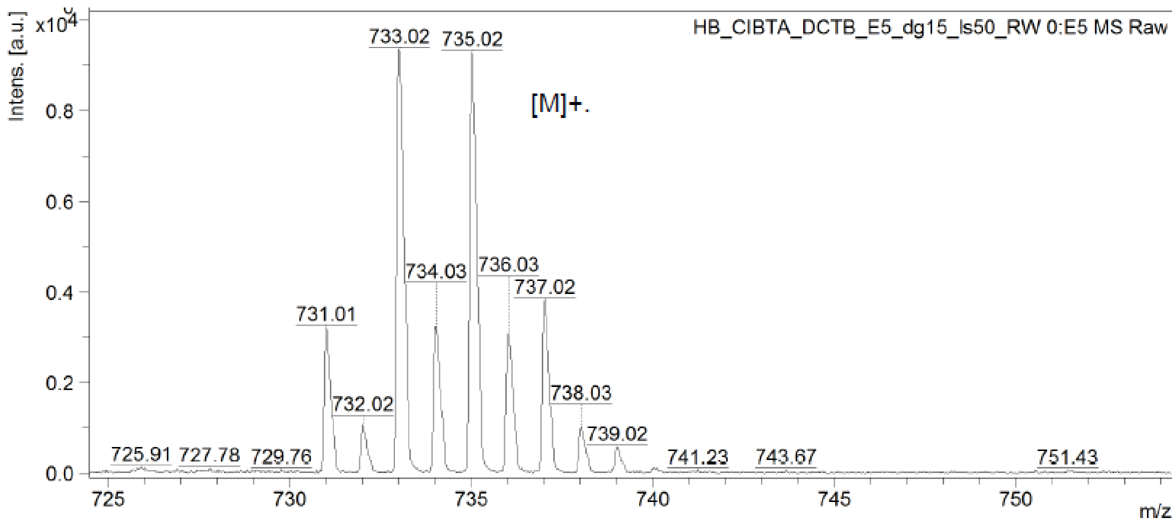


Fig. S20. The MALDI-MS of monomer **M4**. Calcd. for $C_{30}H_{37}Br_2Cl_2N_3S_2$ *m/z* = 731.02 (1st peak) and 733.02 (100% peak). Found *m/z* = 731.01 (1st peak), 733.02 (100% peak).

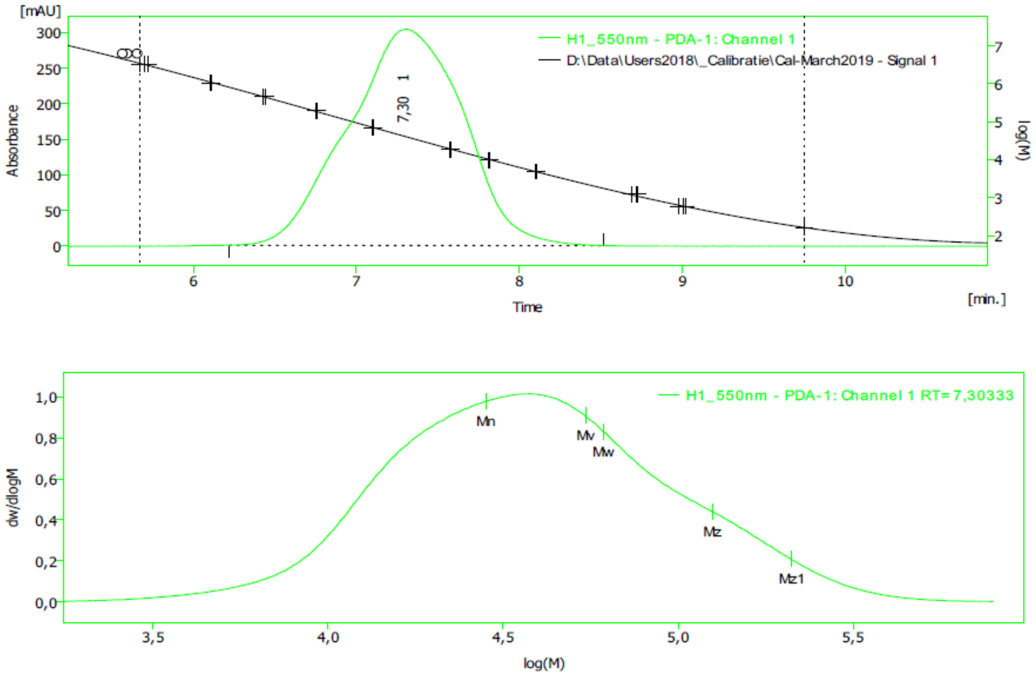


Fig. S21. The GPC trace of J71.

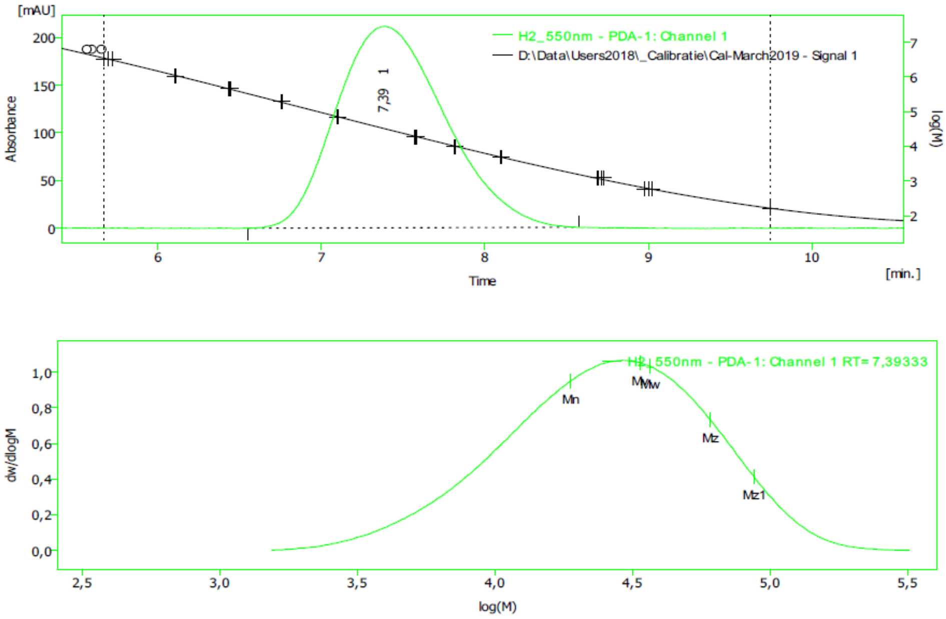


Fig. S22. The GPC trace of J71-Cl.

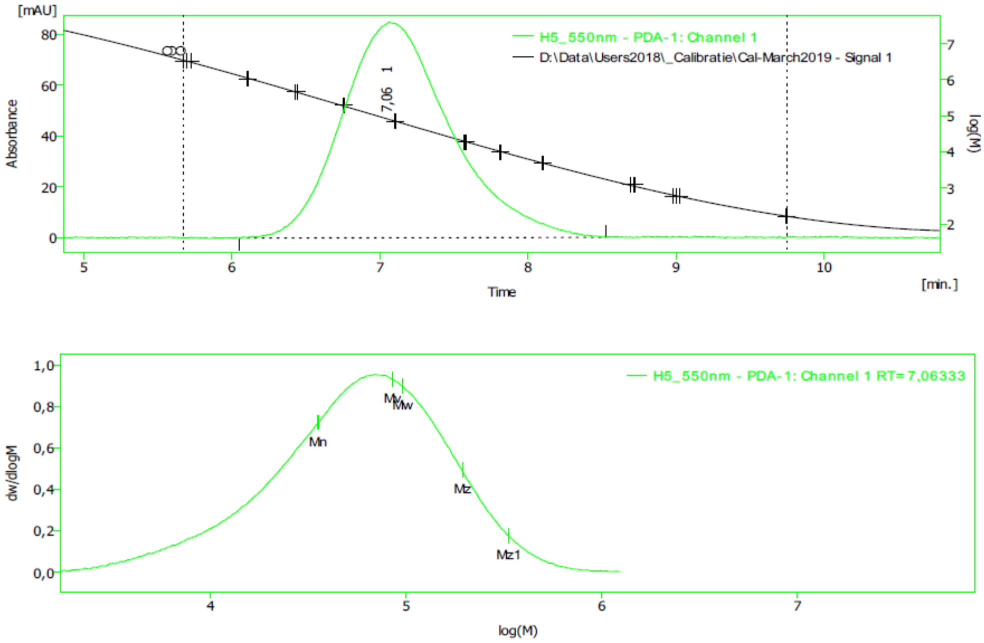


Fig. S23. The GPC trace of J100.

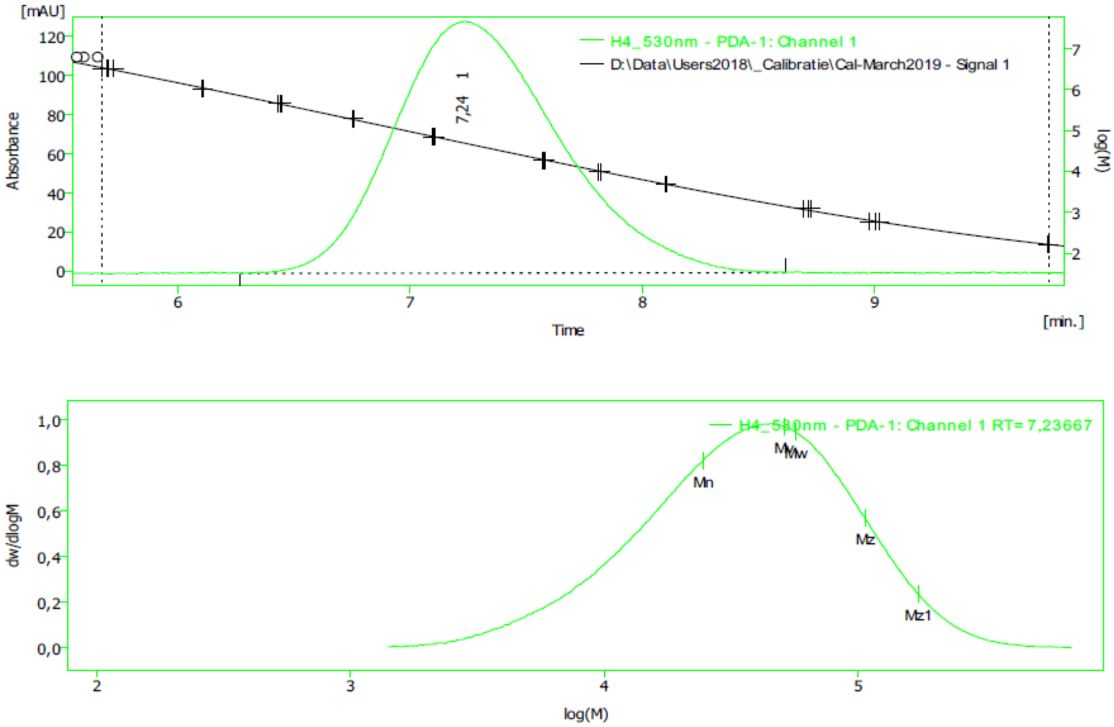


Fig. S24. The GPC trace of J101.

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