

Supporting Information for

Efficient Synthesis and Photovoltaic Properties of Highly Rigid Perylene- embedded Benzothiazolyls

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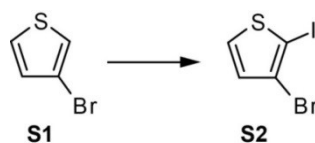
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1. General information

Chemicals and solvents were purchased from Aldrich or Across and used without further treatments. The manipulations involving air-sensitive reagents were performed in an atmosphere of dry argon. ^1H and ^{13}C -nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Ultra Shield Plus 400MHz instrument with CDCl_3 as the solvents and tetramethylsilane (TMS) as the internal standard. The matrix assisted laser desorption ionization time of flight mass spectroscopy (MALDI-TOF MS) measurements were carried out on a Shimadzu AXIMA-CFR mass spectrometer. Gas chromatography-mass spectrometry (GC-MS) analyses were conducted on a Shimadzu GCMS-QP2010. The number-average molecular weight (M_n) and weight-average molecular weight (M_w) of the polymer were measured by gel permeation chromatography (GPC) with Shim-pack GPC-80X columns, using polystyrenes as standard and tetrahydrofuran (THF) as eluent. Ultraviolet-visible (UV/Vis) absorption and fluorescence spectra were obtained by using Shimadzu UV-3600 UV/Vis-NIR spectrophotometer and a RF-5301PC spectrofluorophotometer, respectively. The thermogravimetric analyses (TGA) were conducted on a Shimadzu DTG-60H thermogravimetric analyses under a heating rate of $10^\circ\text{C}/\text{min}$ and a nitrogen flow rate of $50\text{ cm}^3/\text{min}$. Atomic force microscope (AFM) images were obtained using a Bruker Dimension Icon AFM equipped with Scanasyt-Air peak force AFM tips. Cyclic voltammetry (CV) measurements were performed at room temperature on a CHI660E system in a typical three-electrode cell with a working electrode (glass carbon), a reference electrode (Ag/Ag^+) referenced against ferrocene/ferrocenium (FOC), and a counter electrode (Pt wire) in an acetonitrile solution of 0.1M tetrabutylammonium hexafluorophosphate (Bu_4NPF_6) at a sweeping rate of 100 mV s^{-1} . Thus, the highest occupied molecular orbital (HOMO) energy levels (E_{HOMO} , in eV) can be identified from the CV curves according to: $E_{\text{HOMO}} = -(E_{\text{ox}} + 4.79)$; where E_{ox} is the onset oxidation voltage (in V) and the 4.79 eV is the reference energy level of FOC. The LUMO energy levels (E_{LUMO}) were deduced from the HOMO energy level and the optical bandgap ($^{\text{opt}}E_g$) by: $E_{\text{LUMO}} = E_{\text{HOMO}} + ^{\text{opt}}E_g$.

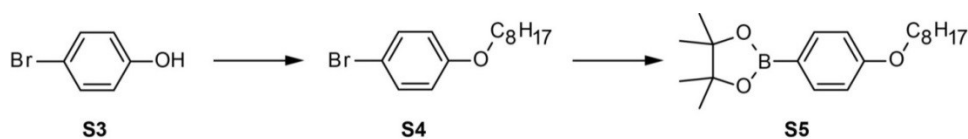
The density functional theory (DFT) calculations were performed on Gaussian 09 program. The widely used the Becke three-parameter hybrid functional combined with Becke-Lee-Yang-Parr correlation functional (B3LYP) using 6-31G(d) basis sets was employed to get a theoretical prediction of the single molecular geometries and the frontier orbital energy levels of the molecules at the ground states (S_0). The molecular electrostatic potential (ESP) was generated using Gaussview 5.0, where the red color describes negative electrostatic potential while the blue stands for positive electrostatic potential¹.

2. Synthesis and characterization



3-Bromo-2-iodothiophene (**S2**):

To a mixture of **S1** (3-bromothiophene) (14.00 g, 86.47 mmol), iodine (6.98 g, 27.50 mmol) and HIO_3 (2.80 g, 15.91 mmol) was added acetic acid (27.50 mL), water (10 mL), carbon tetrachloride (6.74 mL) and concentrated sulfuric acid (0.56 mL) in a three-necked bottle at room temperature. The reaction solution was stirred at 40°C until the iodine was consumed (3 h).² Then, sodium hydrogen carbonate aqueous solution was added to quench the reaction. The resulting mixture was extracted with dichloromethane, washed subsequently with sodium hydrogen carbonate aqueous solution and water, dried over magnesium sulfate (MgSO_4). After removing the solvent under vacuum, the crude product was purified by column chromatography on silica gel and eluted with petrol ether (PE) to provide compound **S2** (18.60 g, 74.8%) as colorless liquid. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.422 (d, 1H), 6.907 (d, 1H).



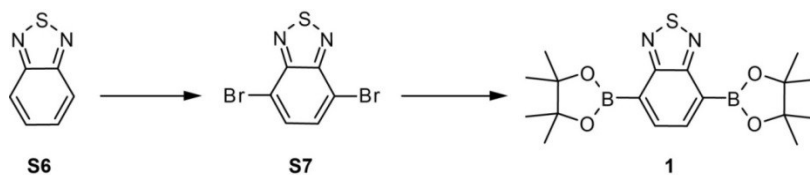
1-Bromo-4-(octyloxy)benzene (**S4**):

To a 55 mL ethyl alcohol solution of 0.50 g (21.74 mmol) sodium was added a 70 mL ethyl alcohol solution of *p*-bromophenol **S3** (10.00 g, 57.80 mmol). The resulting mixture was stirred at room temperature for 15 min, then 15.00 g (77.67 mmol) 1-bromine octane was added to the above formed sodium salt solution of **S3**. After refluxed for 5 h, the reaction solution was poured into water (70 mL) and extracted with dichloromethane (50 mL \times 3). The organic layers were collected, dried over MgSO_4 , and concentrated *in vacuo*. The resulting residue was purified on a silica gel column using petrol ether (PE) as eluent to afford compound **S4** (15.69 g, 95.6%) as colorless liquid.

4,4,5,5-Tetramethyl-2-(4-(octyloxy)phenyl)-1,3,2-dioxaborolane (**S5**):

1-Bromo-4-(octyloxy)benzene **S4** (15.65 g, 55.10 mmol) dissolved in 70 mL anhydrous THF under argon atmosphere was cooled to -78°C using dry ice/acetone solution. To the resulting solution, *n*-butyllithium in hexane (24.13 mL, 38.60 mmol, 1.6 M) was added dropwise with continuous stirring for 1 h

at -78°C . Then 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (12.36 mL, 60.57 mmol) was quickly added into. After stirred at -78°C for 0.5 h, the reaction mixture was allowed to warm to room temperature and further reacted overnight. Then, it was poured into water (50 mL), extracted with dichloromethane (50 mL \times 3), and dried over MgSO_4 , and concentrated *in vacuo*. The resulting residue was purified by column chromatography on a silica gel column using petrol ether (PE) as eluent to afford compound **S5** (14.69 g, 79.8%) as colorless liquid. ^1H NMR (400 MHz, CDCl_3 , ppm): δ (ppm) = 7.752 (d, 2H), 6.897 (d, 2H), 3.993 (t, 2H), 1.800 (t, 2H), 1.411 (d, 14H), 1.287 (d, 21H), 0.903 (t, 8H).

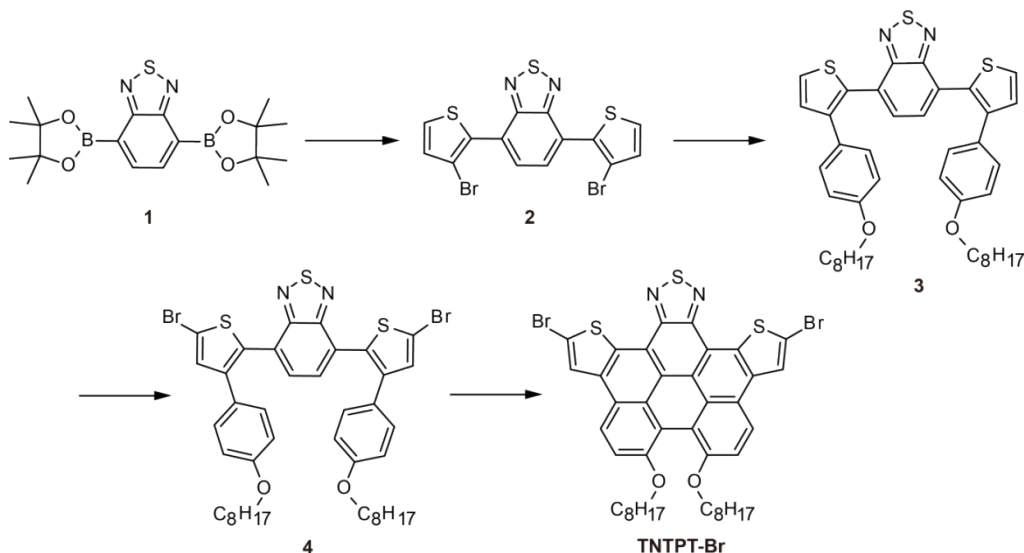


4,7-Dibromobenzo[*c*][1,2,5]thiadiazole (**S7**):

To 150 mL concentrated HBr (47%) solution of benzothiadiazole **S6** (10.00 g, 73.44 mmol), Br_2 (35.21 g, 220.32 mmol) dissolved in 100 mL of HBr (47%) was added dropwise very slowly at room temperature. The resulting solution was allowed to be refluxed for more than 6 h. After the mixture was cooled to room temperature, sufficient saturated aqueous solution of NaHSO_3 was added to remove completely any excess Br_2 . The precipitation of an orange solid was noted as the target product. After filtering, washing exhaustively with water and cold ether, and drying under vacuum for *ca.* 20 h, the dibrominated product **S7** (20.51 g) in 95.7% yield was obtained. ^1H NMR (400Hz, $\text{CDCl}_3/\text{DMSO-d}_6$ —two drops): δ (ppm) = 7.73 (s, 2H). ^{13}C NMR (100Hz, $\text{CDCl}_3/\text{DMSO-d}_6$ —two drops): δ (ppm) = 152.6, 132.1, 113.6.

4,7-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzo[*c*][1,2,5]thiadiazole (**1**):

The mixture of 4,7-dibromobenzo[*c*][1,2,5]thiadiazole **S7** (20.00 g, 68.53 mmol), bis(pinacolato)diboron (39.76 g, 156.57 mmol), 1,1'-bis(diphenylphosphino)-ferrocene dichloropalladium (II) ($\text{PdCl}_2(\text{dppf})$) (4.08 g, 5.00 mmol), and potassium acetate (40 g, 407.58 mmol) was dissolved in 1,4-dioxane (200 mL). The resulting solution was reacted at 85°C for 24 h under argon atmosphere. After cooling to room temperature, the reaction mixture was poured into water and extracted using ethyl acetate. The combined organic phase was washed with brine, dried over MgSO_4 , and concentrated *in vacuo*. The crude product was purified by column chromatography on silica gel (eluent: ester acetate (EA)/petrol ether (PE) = 1/5) to afford compound **1** (11.10 g, 41.7%) as a light yellow powder. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.129 (s, 2H), 1.436 (s, 4H). ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 156.980, 137.780, 84.453, 77.362, 77.044, 76.726, 24.914.



4,7-Bis(3-bromothiophen-2-yl)benzo[c][1,2,5]thiadiazole (**2**):

A mixture of **1** (10.00 g, 25.76 mmol), **S2** (16.44 g, 57.10 mmol), and tetrakis(triphenylphosphine)palladium(0) (Pd(PPh₃)₄) (1.00 g, 0.86 mmol) dissolved in toluene (110 mL) and 2 M aqueous solution of potassium carbonate (50 mL) was stirred at 90°C for 36 h under argon atmosphere. After cooling to room temperature, the mixture was poured into water and extracted with dichloromethane. The combined organic phase was dried over MgSO₄, and concentrated *in vacuo*. The residue was purified on a silica gel chromatography (eluent: dichloromethane (DCM)/petrol ether (PE): 1/6) to afford compound **2** (7.67 g, 64.8 %) as yellow powders. ¹H-NMR (400 MHz, CDCl₃): δ (ppm) = 8.096 (s, 2H), 7.539 (d, 2H), 7.200 (d, 2H). ¹³C NMR (100 MHz, CDCl₃): δ (ppm)=153.432, 132.884, 131.335, 129.908, 127.553, 125.996, 110.539, 77.330, 77.012, 6.695. MALDI-TOF m/z calcd for C₁₄H₆Br₂N₂S₃ [M+H]⁺: 458.21; found: 454.235.

4,7-Bis(3-(4-(octyloxy)phenyl)thiophen-2-yl)benzo[c][1,2,5]thiadiazole (**3**):

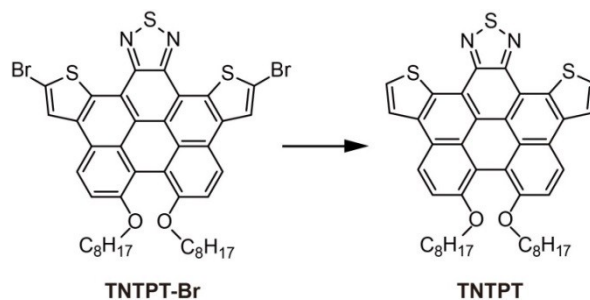
A mixture of **2** (2.00 g, 4.39 mmol), **S5** (3.63 g, 10.98 mmol), tetrakis(triphenylphosphine)palladium(0) (Pd(PPh₃)₄) (0.20 g, 0.17 mmol) dissolved in toluene (16 mL) and 2 M aqueous solution of potassium carbonate (8 mL) was stirred at 90°C for 36 h under argon atmosphere. After cooling to room temperature, the mixture was poured into water and then extracted with dichloromethane. The combined organic phase was dried over MgSO₄, and concentrated *in vacuo*. The residue was purified on a silica gel chromatography (eluent: dichloromethane (DCM)/petrol ether (PE) = 1/4) to afford compound **3** (2.01 g, 64.3 %) as red crystal. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.527 (d, 2H), 7.275 (d, 2H), 7.229 (d, 2H), 7.160 (d, 4H), 6.754 (d, 4H), 3.920 (t, 4H), 1.78 (m, 4H), 1.451 (t, 4H), 1.320 (t, 16H), 0.899 (t, 6H). ¹³C NMR (100 MHz, CDCl₃): δ (ppm)= 158.252, 153.957, 140.870, 131.965, 130.193, 129.971, 129.875, 128.829, 126.753, 126.459, 114.417, 77.354, 77.036, 76.719, 67.962, 31.837, 29.412, 29.320, 29.267, 26.085, 22.678, 14.126. MALDI-TOF m/z calcd for C₄₂H₄₈N₂O₂S₃ [M+H]⁺: 709.04; found: 708.749.

*4,7-Bis(5-bromo-3-(4-(octyloxy)phenyl)thiophen-2-yl)benzo[*c*][1,2,5]thiadiazole (4):*

A mixture of **3** (1.5 g, 2.12 mmol) and *N*-bromosuccinimide (NBS) (0.9 g, 5.07 mmol) dissolved in *N,N*-dimethyl formamide (DMF) (50 mL) was stirred at room temperature in dark for 24 h. After the removing of the solvent under reduced pressure, the residue was chromatographically purified on silica gel column eluting with dichloromethane (DCM)/petrol ether (PE) (1:3) to afford compound **4** as a red crystal (1.65 g, 90.2%). ¹H NMR (400 MHz, CDCl₃): δ (ppm)= 7.181 (d, 2H), 7.114 (d, 4H), 6.754 (d, 4H), 3.921 (t, 4H), 1.791 (m, 4H), 1.470 (m, 4H), 1.323 (m, 16H), 0.902 (t, 6H). ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 158.630, 153.568, 141.357, 133.104, 132.639, 129.838, 129.797, 127.697, 125.874, 114.559, 114.223, 77.361, 77.044, 76.726, 68.005, 31.838, 29.410, 29.266, 26.081, 22.681, 143.132, 14.128, 0.024. MALDI-TOF *m/z* calculated for C₄₂H₄₆Br₂N₂O₂S₃ [M+H]⁺: 866.83; found: 865.370.

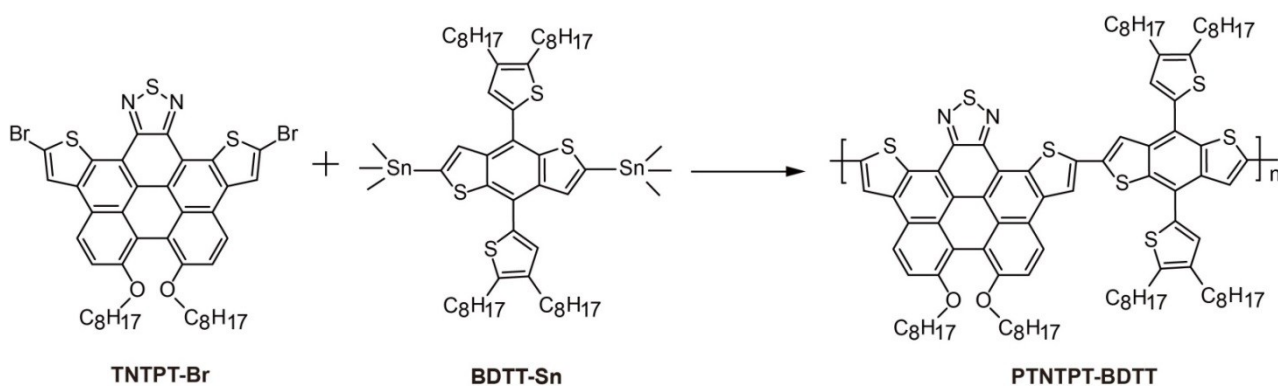
*2,11-Bibromo-6,7-bis(octyloxy)thieno[2'',3'':3',4']naphtho[2',1',8':3,4,5]thieno[3',2':9,10]pyreno[1,2-*c*][1,2,5]thiadiazole (TNTPT-Br):*

To a solution of **4** (1.50 g, 1.73 mmol) in 150 mL dry dichloromethane was added a nitromethane (CH₃NO₂) solution (5 mL) of ferric trichloride (FeCl₃) (3.37 g, 2.06 mmol) at 0°C under argon atmosphere. After stirring for 5 h, 30 mL of anhydride methanol was added to quench the reaction. The mixture was poured into water and then extracted with dichloromethane. The combined organic phase was dried over MgSO₄, and concentrated under vacuum. The residue was purified on a silica gel column (eluent: dichloromethane (DCM)/petrol ether (PE): 1/3) afforded **TNTPT-Br** (0.97 g, 65.4 %) as solid powder. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.980 (d, 2H), 7.597 (s, 2H), 7.435 (d, 2H), 4.222 (m, 4H), 1.913 (m, 4H), 1.507 (d, 4H), 1.426 (m, 17H), 0.933 (t, 6H). ¹³C NMR(100 MHz, CDCl₃): δ (ppm) = 155.104, 148.157, 135.038, 132.273, 125.838, 123.944, 122.997, 122.796, 121.403, 118.275, 115.761, 114.336, 114.288, 77.346, 77.028, 76.711, 69.019, 31.925, 29.953, 29.693, 29.428, 26.219, 22.755, 14.188, 0.014. MALDI-TOF *m/z* calcd for C₄₂H₄₀Br₂N₂O₂S₃ [M+H]⁺: 860.78; found: = 859.330.



*6,7-Bis(octyloxy)thieno[2'',3'':3',4']naphtho[2',1',8':3,4,5]thieno[3',2':9,10]pyreno[1,2-*c*][1,2,5]thiadiazole (TNTPT):*

TNTPT-Br (0.5 g, 0.58 mmol) was dissolved in 15 mL anhydrous THF under argon atmosphere and cooled to -78°C . *n*-Butyllithium in hexane (0.52 mL, 1.28 mmol, 2.5 M) was added dropwise with stirring for 1 h at -78°C . Then, methanol (1 mL) was added. The reaction mixture was allowed to warm to room temperature overnight. After poured into water (50 mL), extracted with dichloromethane (50 mL \times 3), dried over MgSO_4 , and concentrated *in vacuo*, the crude product was collected and purified on a silica gel column using dichloromethane (DCM)/petrol ether (PE): 1/4 as eluent to give the target compound of **TNTPT** (0.3 g, 73.7%) in red powder. ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.365 (d, 2H), 7.974 (d, 2H), 7.724 (d, 2H), 7.575 (d, 2H), 7.261 (s, 2H), 4.214 (m, 4H), 1.835 (t, 4H), 1.437 (s, 4H), 1.293 (s, 17H), 0.890 (t, 6H). ^{13}C NMR(100 MHz, CDCl_3): 155.396, 150.114, 135.887, 131.830, 129.492, 139.367, 126.967, 124.514, 123.241, 123.035, 121.403, 116.556, 114.889, 69.296, 114.288, 69.019, 31.837, 31.825, 29.846, 29.546, 29.528, 29.329, 29.313, 26.103, 22.682, 14.108. MALDI-TOF m/z calcd for $\text{C}_{42}\text{H}_{40}\text{N}_2\text{O}_2\text{S}_3$ $[\text{M}]^+$: 702.990; found: 702.451.



Poly(2-(4,8-bis(4,5-dioctylthiophen-2-yl)-6-methylbenzo[1,2-b:4,5-b']dithiophen-2-yl)-11-methyl-6,7-bis(octyloxy)thieno[2'',3'':3',4']naphtho[2',1',8':3,4,5]thieno[3',2':9,10]pyreno[1,2-c][1,2,5]thiadiazole) (**PTNTPT-BDTT**):

In a 50 mL reaction tube, **TNTPT-Br** (0.15 g, 0.17 mmol), (4,8-bis(4,5-dioctylthiophen-2-yl)benzo[1,2-b:4,5-b']dithiophene-2,6-diyl) bis(trimethylstannane) **BDTT** (0.20 g, 0.17 mmol) and tetrakis(triphenylphosphine) palladium(0) ($\text{Pd}(\text{PPh}_3)_4$) (0.01 g, 0.01 mmol) were dissolved in 5 mL dry toluene under argon atmosphere. After stirred at 110°C for 36 h, the mixture was cooled to room temperatures and dropped into methanol (80 mL). The precipitate was filtered out and washed with methanol, following further purification with sequential Soxhlet extraction using methanol and hexane to remove oligomers and catalyst residue. The target polymer of **PTNTPT-BDTT** was obtained as dark purple solid (170 mg, 66.6%). The molecular weight was measured using gel-permeation chromatography (GPC) in tetrahydrofuran against polystyrene standards.

3. Device fabrication and measurements:

The prototype bulky heterojunction (BHJ) solar cells of PTNTPT-BDTT were fabricated under a conventional device structure of ITO/PEDOT:PSS (40 nm)/polymer:PCBM (120 nm) /Ca (0.6 nm)/Al, where PEDOT:PSS is poly(3,4-ethylenedioxythiophene as hole injection and transport layer, PCBM is PC₆₁BM ([6,6]-phenyl-C₆₁-butyric acid methyl ester) or PC₇₁BM ([6,6]-phenyl-C₇₁-butyric acid methyl ester) as electron acceptor, and indium tin oxide (ITO) and Ca/Al act as anode and cathode materials respectively³. In a general procedure, patterned ITO-coated glass substrates were cleaned by sequential ultrasonic treatments using detergent, deionized water, isopropyl alcohol, and acetone, followed by UV-ozone treatment for 10 min. Then, a thin film of PEDOT: PSS (~40 nm) was spin-coated on the cleaned ITO substrate and dried at 150°C for 30 min. A blend of polymer and PC₇₁BM (10 mg/mL) was dissolved in chlorobenzene containing 0-3 vol% 1-chloronaphthalene (CN) or 1,8-diiodooctane (DIO), followed by filtering through a 0.45 μm poly(tetrafluoroethylene) (PTFE) filter, spin-coating at 1200 rpm for 40 s on the PEDOT: PSS coated ITO, and annealing at 110°C for 10 min. The Ca (0.6 nm, 0.2 Å/s) and Al (100 nm, 2 Å/s) layers were thermally evaporated on the active layer at a pressure of 1.0×10⁻⁶ mbar through a shadow mask (active area 4 mm²). The current density–voltage characteristics of the photovoltaic cells were measured using a Keithley 2400 digital source meter under a simulated AM 1.5G solar irradiation at 100 mW/cm² (Newport, Class AAA solar simulator, 94023A-U). The light intensity is calibrated by a certified Oriel Reference Cell (91150 V) and verified with a NREL calibrated Hamamatsu S1787-04 diode. The incident photon to current efficiency (IPCE) of the device was measured using a certified IPCE equipment (Zolix Instruments, Inc, SolarCellScan100).

The hole mobility of blend films used in OPVs was measured in a hole-only device (ITO/PEDOT/Blends/MoO₃/Al) by fitting the current density (J) at the applied electrical voltage (V) following a space charge limited current (SCLC) model in Mott-Gurney law (Equation 1)⁴.

$$J = \varepsilon_0 \varepsilon \mu \frac{9V^2}{8d^3} \quad (1)$$

where ε_0 is the permittivity of free space (8.854×10^{-12} F/m), ε is the dielectric constant of the film ($\varepsilon=3$), d is the thickness of the active layer (90 nm), and μ is the mobility. The hole mobility of the PTNTPT-BDTT:PC₇₁BM film was found to be 2.1×10^{-5} cm²V⁻¹S⁻¹ by fitting J and V in Figure S11 according to Equation 1.

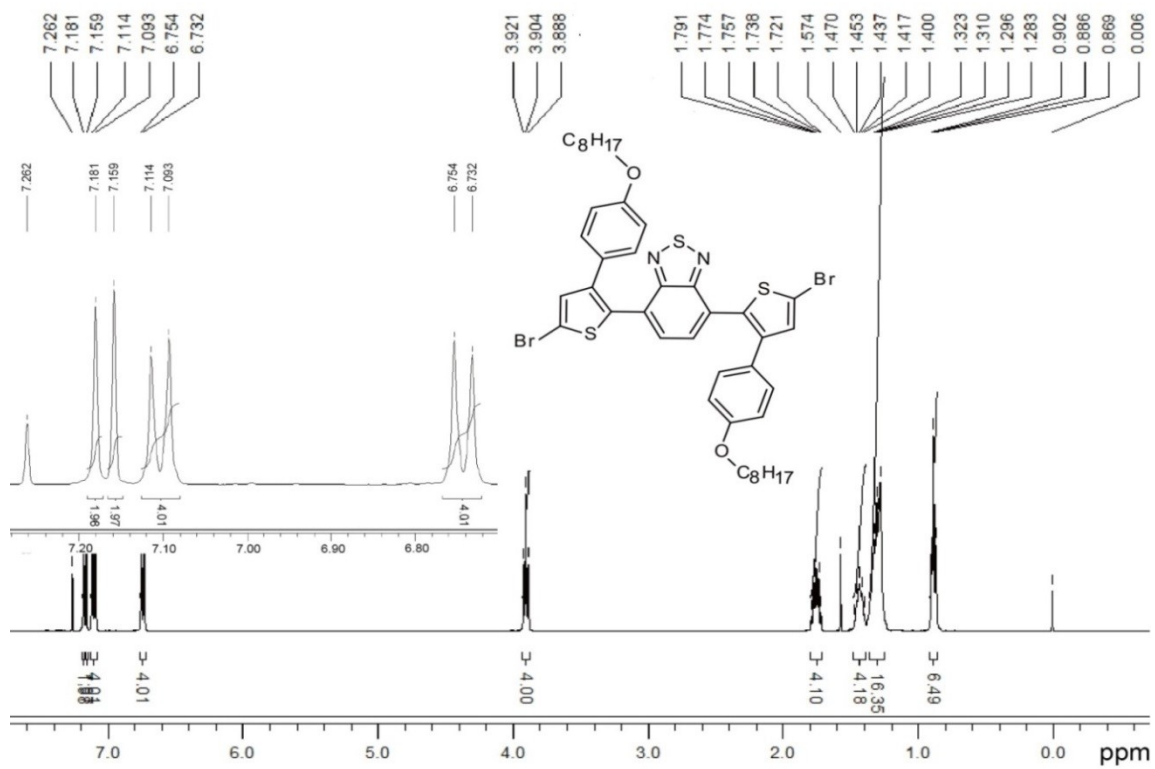


Figure S1. ^1H NMR spectrum of compound **4** in CDCl_3

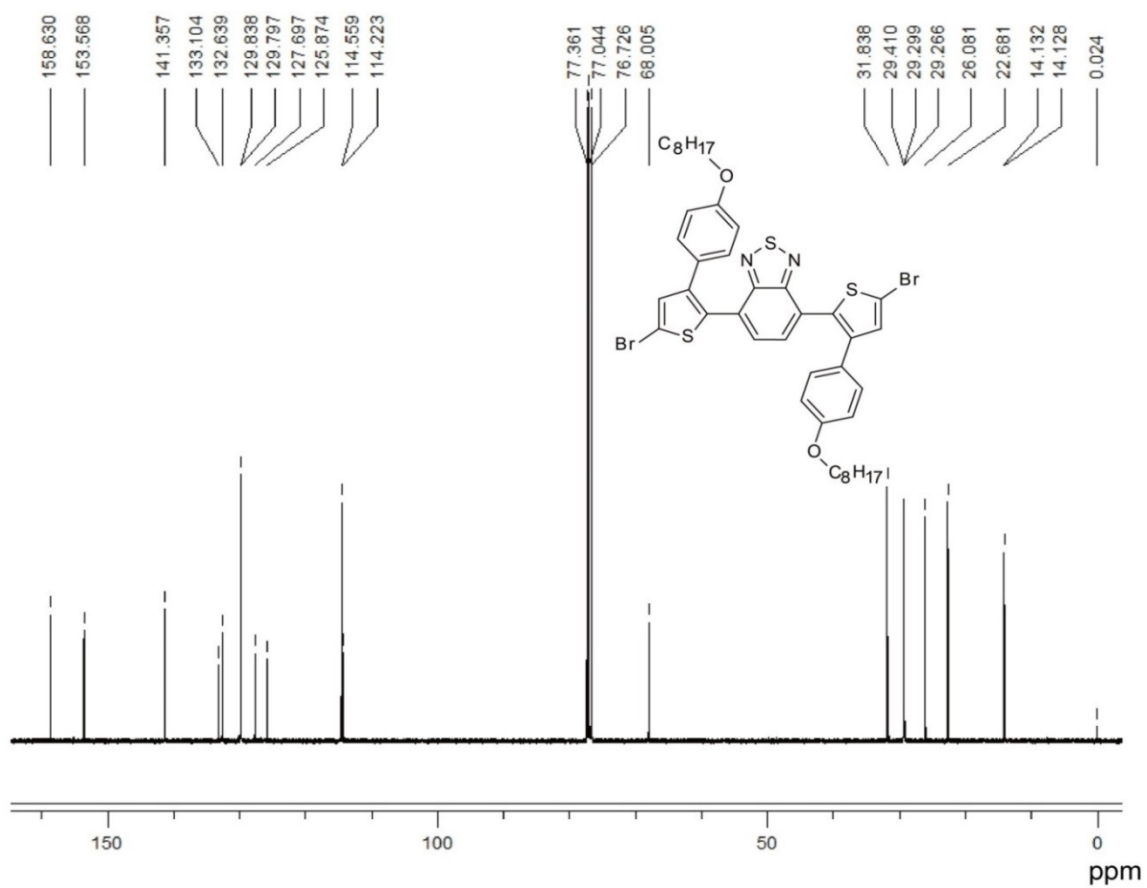


Figure S2. ^{13}C NMR spectrum of compound **4** in CDCl_3

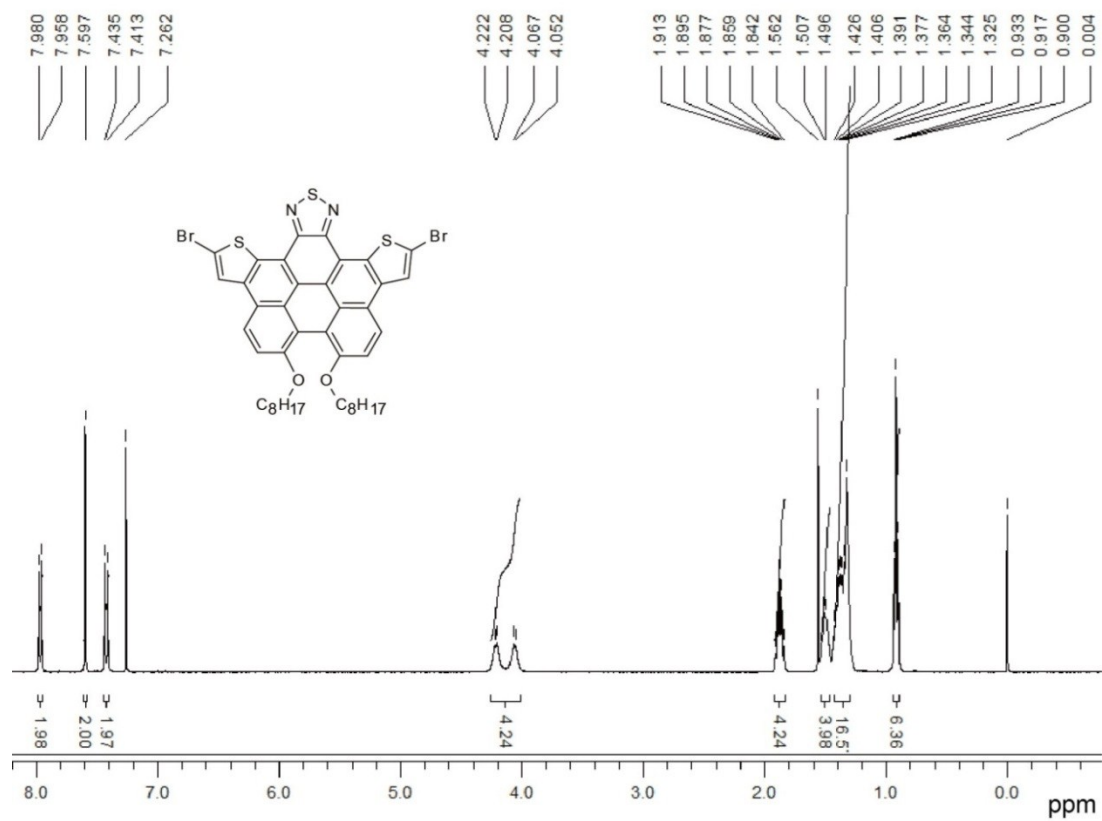


Figure S3. ¹H NMR spectrum of TNTPT-Br in CDCl₃

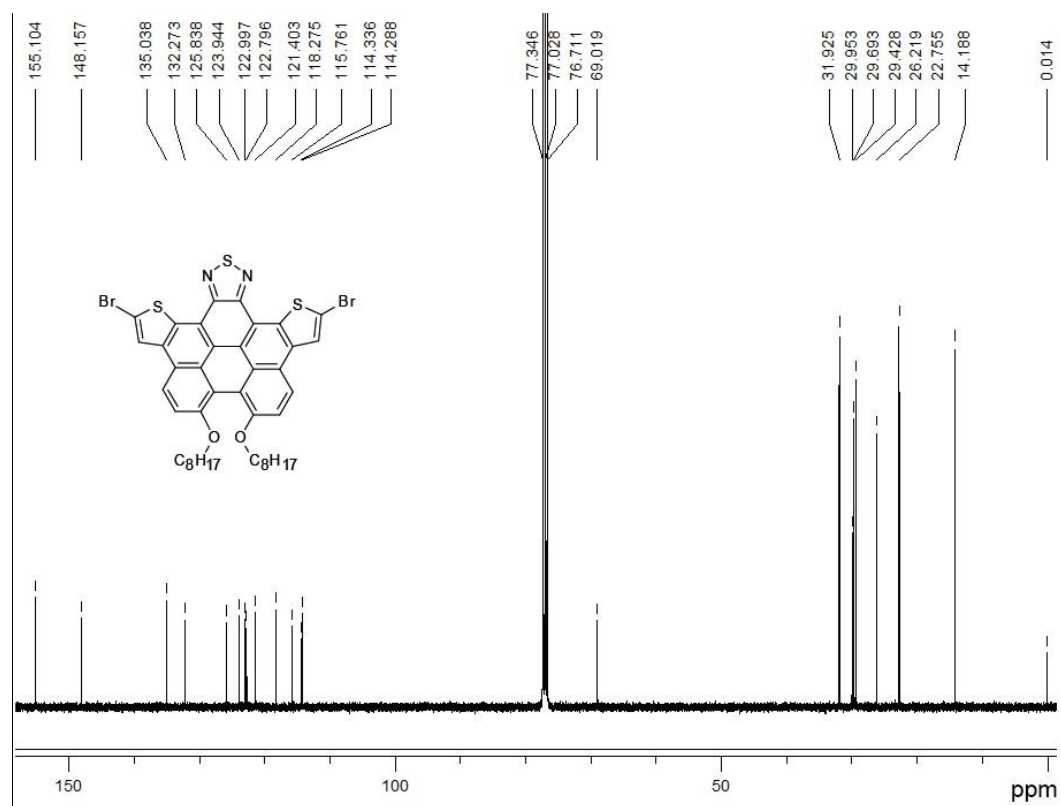


Figure S4. ¹³C NMR spectrum of TNTPT-Br in CDCl₃

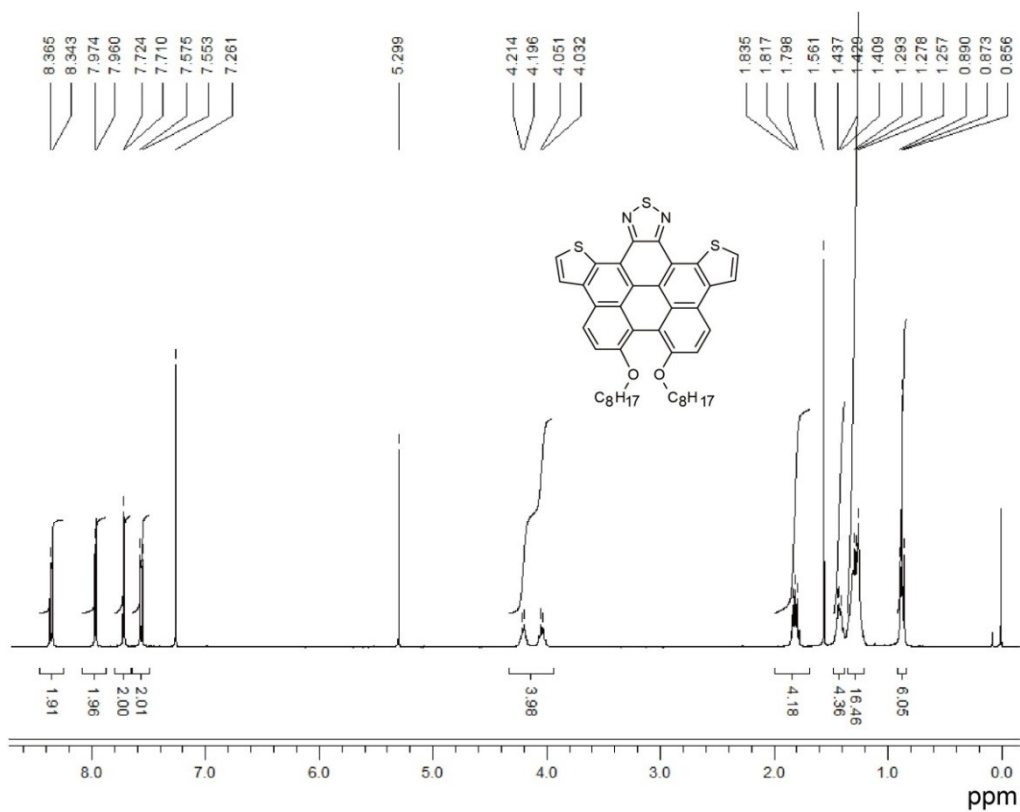


Figure S5. ¹H NMR spectrum of TNTPT in CDCl₃

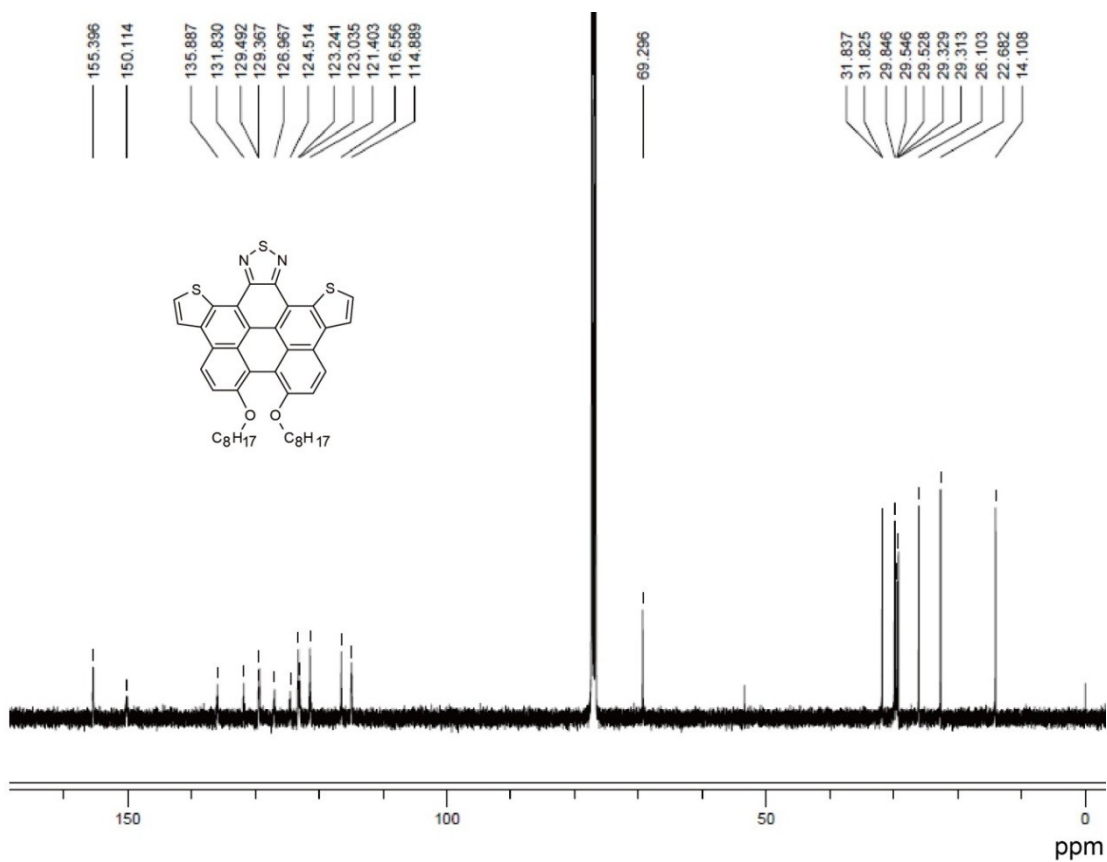


Figure S6. ¹³C NMR spectrum of TNTPT in CDCl₃

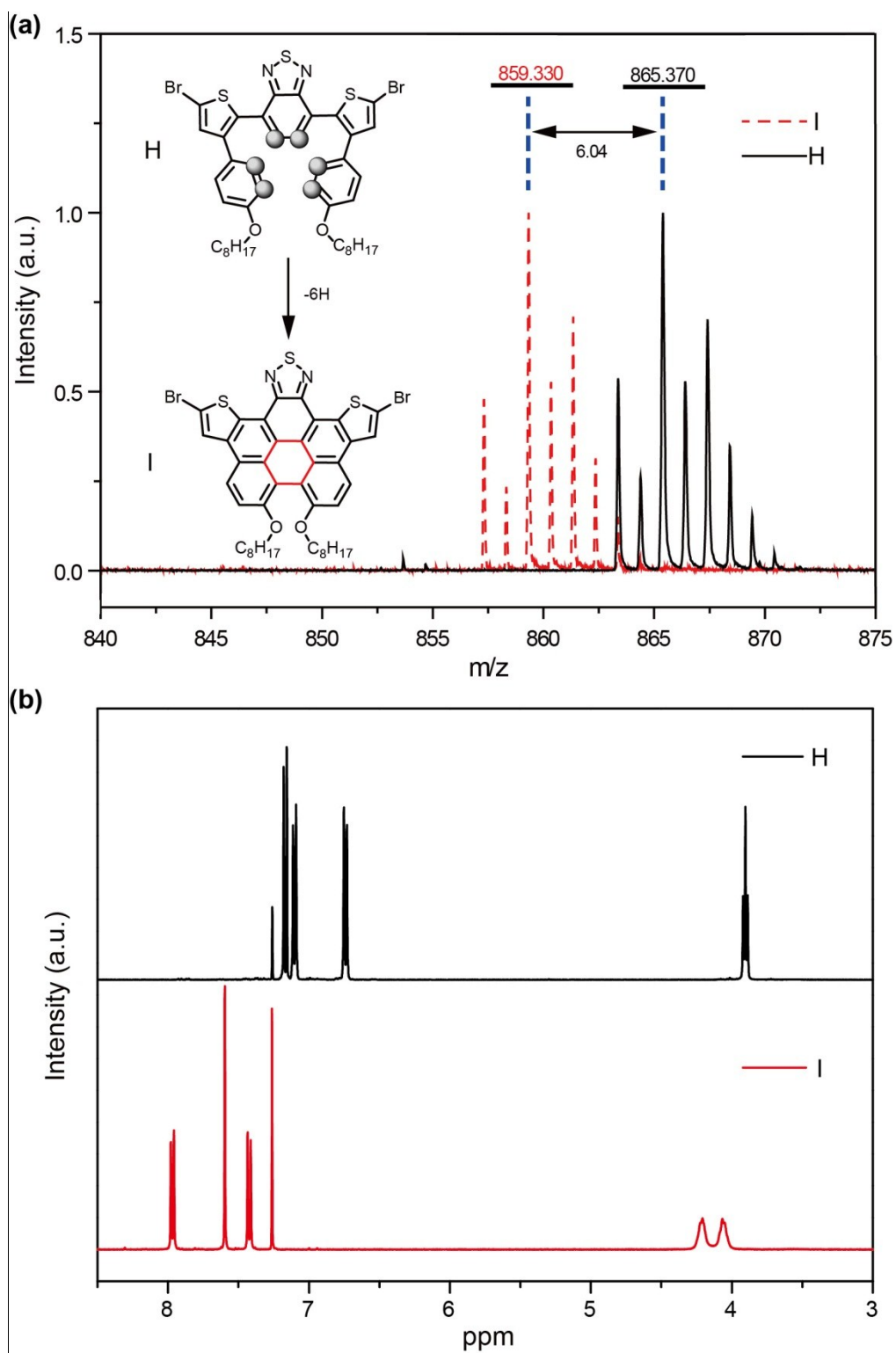


Figure S7. MALDI-TOF (a) and ^1H NMR (b) monitoring the formation of **TNTPT-Br** before (black, H) and after (red, I) the cyclization reaction

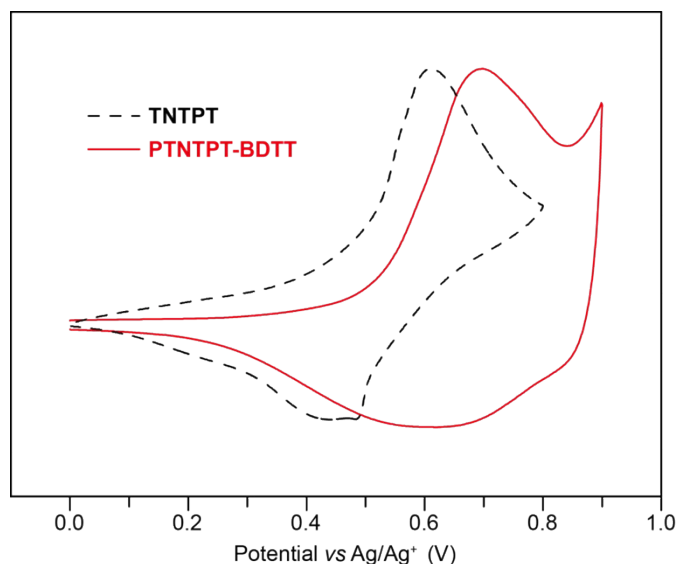


Figure S8. Cyclic voltammograms of TNTPT and PTNTPT-BDTT thin films at a scan rate of 100 mV/s.

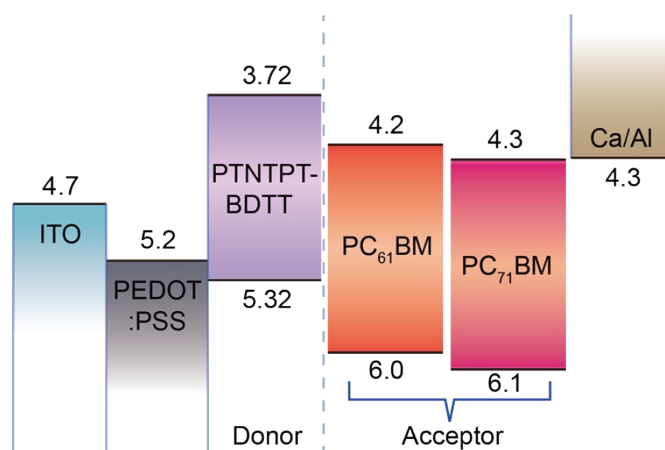


Figure S9. (a) The bulk heterojunction (BHJ) solar cell structure of ITO/PEDOT:PSS/Active Layer /Ca/Al. (b) Energy level diagrams of the materials in the BJJ organic solar cells (in eV).

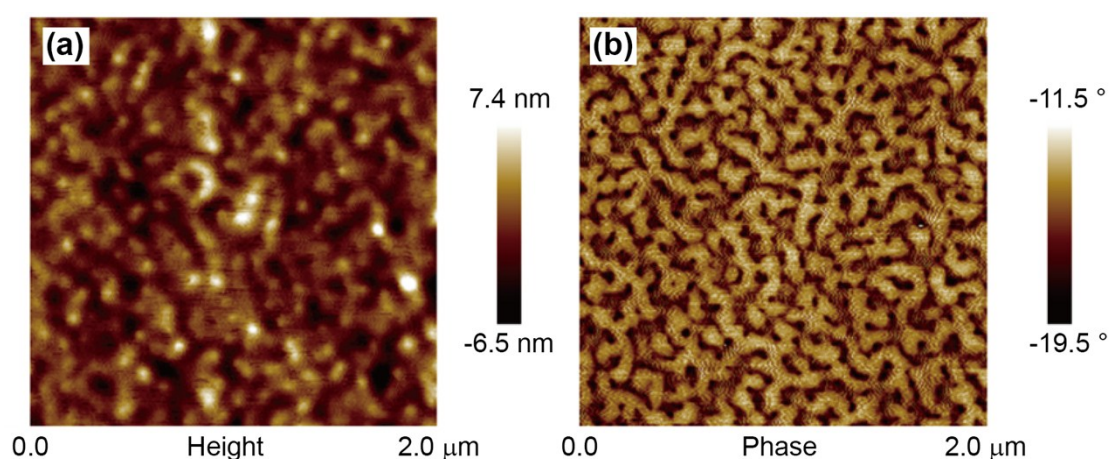


Figure S10. AFM height (a) and phase (b) images ($2 \times 2 \mu\text{m}^2$) of the PTNTPT-BDTT:PC₇₁BM blend film on ITO surface.

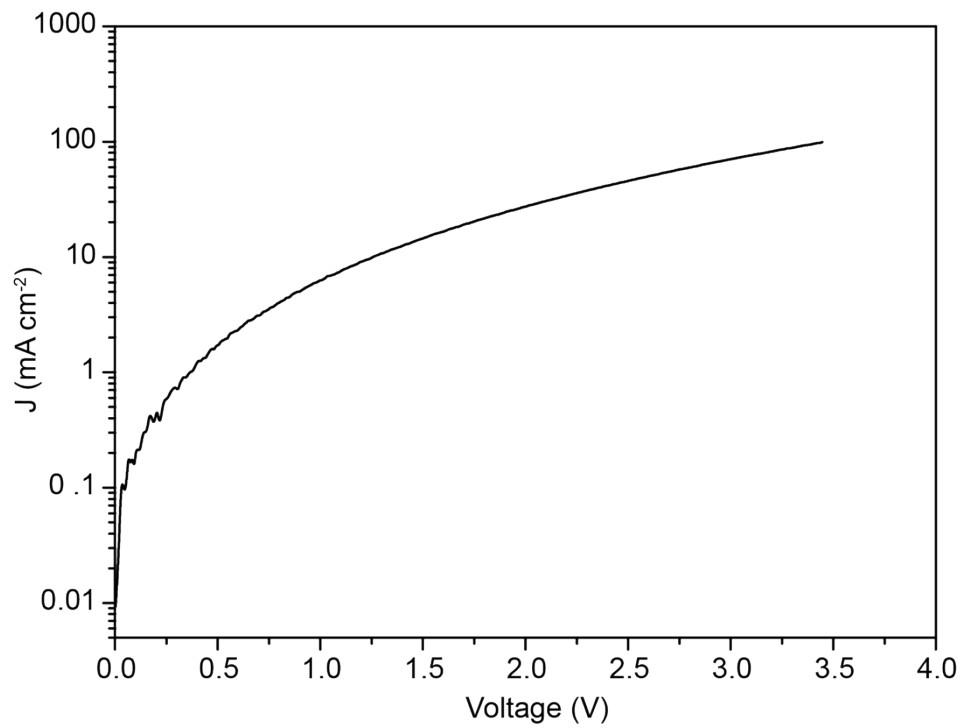
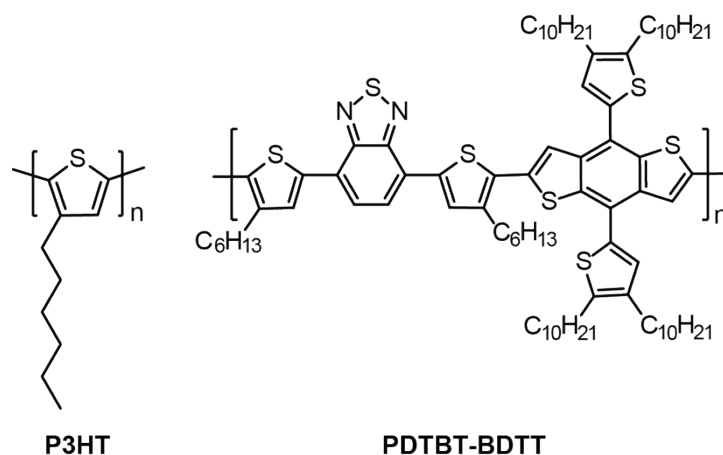


Figure S11. J - V characteristic of the PTNTPT-BDTP:PC₇₁BM-based hole-only device.



Scheme S1. Molecular structures of P3HT⁵ and PDTBT-BDTT⁴.

Table S1. Photovoltaic characteristics of the BHJ OSCs based on PTNTPT-BDTT with different weight ratio of PC₆₁BM under AM 1.5G illumination (100 mW/cm²)

PTNTPT-BDTT:PC ₆₁ BM (w/w)	Additive	J_{sc} (mA cm ⁻²)	V_{oc} (V)	FF (%)	R_s (Ω·cm ²)	R_{sh} (Ω·cm ²)	PCE (%)
1.0:0.5	-	4.76	0.73	29.4	90.91	205.17	1.02
1.0:1.0	-	6.75	0.76	35.8	54.75	221.88	1.84
1.0:1.5	-	6.32	0.75	38.5	54.97	294.12	1.80
1.0:4.0	-	3.40	0.75	40.0	68.73	436.68	1.02
1.0:1.0	CN (1 vol%)	8.07	0.77	34.6	38.62	163.93	2.15

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