

Supporting Information

High performance asymmetrical push-pull small molecules end-capped cyanophenyl with narrow band gap for solution-processed solar cells

Hang Gao,^a Yanqin Li,^a Lihui Wang,^a Changyan Ji,^a Yue Wang,^b Wenjing Tian,^b Xichuan Yang,^a and Lunxiang Yin^{*a}

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^a School of Chemistry, Dalian University of Technology, Dalian, China. Fax: 86-411-84986040;

Tel: 86-411-84986040; E-mail: lxyin@dlut.edu.cn

^b State Key Laboratory of Supramolecular Structure and Materials, College of Chemistry, Jilin University, Changchun, China.

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1. Measurement, Characterization and Calculation

¹H-NMR and ¹³C-NMR were recorded on a Bruker AVANCE II 400 MHz spectrometer using CDCl₃ as the solvent and TMS as the internal standard. The high-resolution mass spectra (HRMS) were collected using a MALDI Micro MX spectrometer. DFT calculations were accomplished by Gaussian 09 software at the Becke's three-parameter gradient-corrected functional (B3LYP) with a polarized 6-31G(d) basis¹. UV-vis spectral data were recorded on an Agilent Cary 5000 spectrophotometer at room temperature in chloroform solution and in thin film. Cyclic voltammetry (CV) was carried out on a CHI 610D electrochemical workstation from CH Instruments, Inc. The redox properties of two molecules were measured in Bu₄NBF₄/dichloromethane solutions under a nitrogen atmosphere. The glass-carbon electrode served as the working electrode, and an Ag/Ag⁺ electrode (Ag in 0.1 M AgNO₃ solution of MeCN) and platinum wire were chosen as the reference electrode and the count electrode, respectively. A ferrocene–ferrocenium (Fc/Fc⁺) couple was used as the internal standard. The electrochemical HOMO^{cv}, LUMO^{cv} and band gaps (E_g^{cv}) of the two materials were calculated from onset oxidation potentials and onset reduction potentials of CV curves according to the following equations¹, where the unit of potential is relative to Ag/Ag⁺, and $E_{1/2}^{\text{ferrocene}} = 0.05$ eV.

$$\text{HOMO/LUMO}^{\text{cv}} = - (E_{\text{Ox/Red}} - E_{1/2}^{\text{ferrocene}} + 4.8) \text{ eV}$$

$$E_g^{\text{cv}} = \text{HOMO}^{\text{cv}} - \text{LUMO}^{\text{cv}}$$

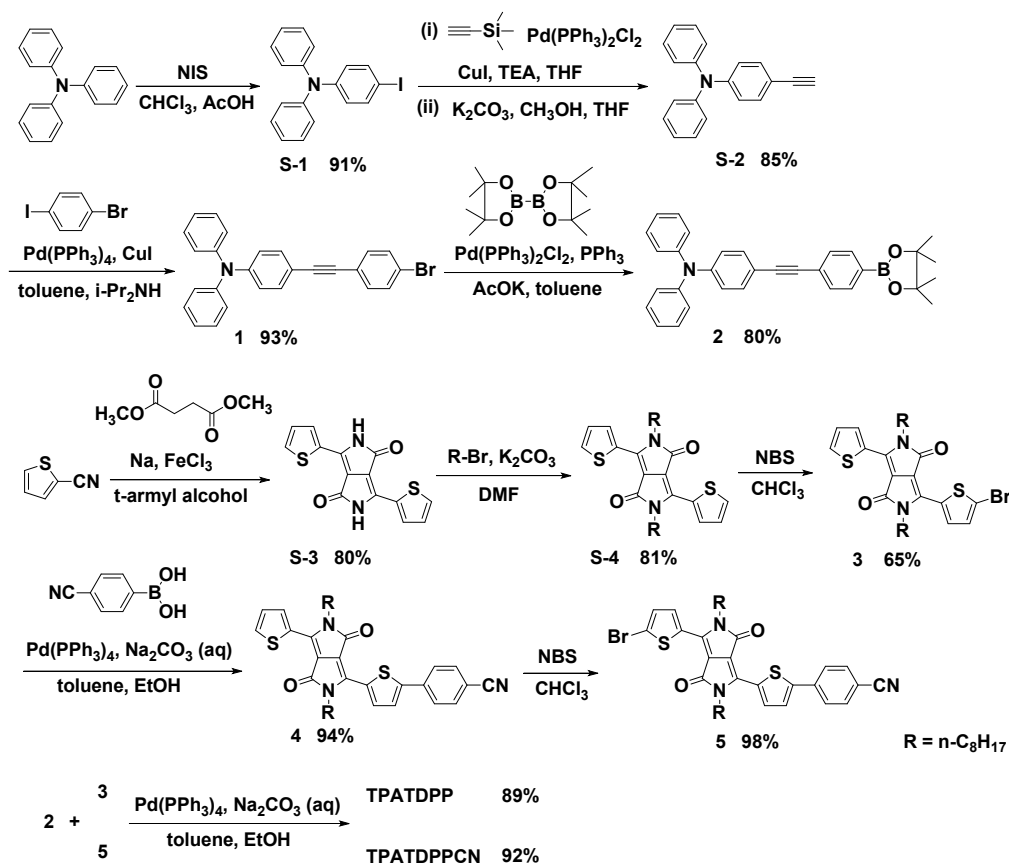
The current density-voltage (J - V) curves were recorded using a computer-controlled Keithley 2400 Source Measure Unit under AM 1.5 G illumination with an intensity of 100 mW cm⁻², whereas the J - V curves of hole-only devices with a structure of ITO/PEDOT:PSS/SMDs:PC₆₁BM/Au were measured with Keithley 2400 Source Measure Unit in the dark. The series resistance (R_s) and shunt resistance (R_{sh}) of devices were obtained from the slope of J - V curves at V_{oc} and J_{sc} , respectively. The monochromatic incident photon-to-electron conversion efficiency (IPCE) spectra were carried out using a SM-25 photoelectric conversion analyzer system. Atomic force microscopy (AFM) images of blend films based on SMDs:PC₆₁BM (1:2, w/w) were recorded on *Nanoscope IIIa Dimension 3100*. X-ray diffraction patterns (XRD) data of the two materials were collected on D/MAX-2400 X-ray Diffractometer.

2. Synthesis

2.1 Reagent and Materials

All reagents were obtained commercially and used as received unless specified. Tetrahydrofuran (THF) and toluene were distilled over Na/benzophenone under nitrogen prior to use. All reactions and manipulations were carried out under nitrogen atmosphere with a standard Schlenk technique.

2.2 Synthetic procedures



Scheme S1 Synthetic route for TPATDPP and TPATDPPCN.

4-Iodo-N,N-diphenylaniline (S-1)^{1d, 1e, 2} Triphenylamine (4.90 g, 20 mmol) and NIS (4.50 g, 20 mmol) were dissolved in 60 mL CHCl_3 and 60 mL glacial AcOH, then the mixture was stirred at room temperature without light for 12 h. The mixture was washed with water and aqueous $\text{Na}_2\text{S}_2\text{O}_3$, respectively. After the combined organic layers were dried over anhydrous Na_2SO_4 , the organic solvent was evaporated under reduced pressure to afford colorless oily liquid. The crude product was washed with little petroleum ether to give a white powder **S-1** (6.75 g, 91%). M.p.: 102-104 °C; $^1\text{H-NMR}$ (400 MHz, CDCl_3 , ppm): δ 7.50 (d, $J = 8.6$ Hz, 2H), 7.25 (t, $J = 8.0$ Hz, 4H), 7.08-7.02 (m, 6H), 6.82 (d, $J = 7.3$ Hz, 2H).

4-Ethynyl-N,N-diphenylaniline (S-2)^{1d, 1e, 2}

(i) A mixture of compound **S-1** (3.71 g, 10 mmol), trimethylsilylacetylene (1.55 mL, 1.08 g, 11 mmol), $\text{Pd(PPh}_3)_2\text{Cl}_2$ (176 mg, 0.25 mmol), CuI (96 mg, 0.5 mmol), 40 mL triethylamine and 40 mL THF was refluxed at 70 °C under N_2 atmosphere overnight. After being cooled to the room temperature, the mixture was poured into 300 mL water and the organic layer was separated. The aqueous layer was extracted with CH_2Cl_2 (3 \times 40 mL) and the combined organic layers were dried over anhydrous Na_2SO_4 . The organic solvent was evaporated under reduced pressure to provide a yellow oily liquid.

(ii) The above product was dissolved in 40 mL THF and 40 mL methanol, then K_2CO_3 (3.04 g, 22 mmol) was added to the solution. Then the mixture was stirred at room temperature for 2 h. After filtration, the organic solvent was evaporated under reduced pressure. The crude product was purified by silica column chromatography eluting with petroleum ether/ CH_2Cl_2 (30:1, v:v) to provide a white solid **S-2** (2.29 g, 85%). M.p.: 102-103 °C. $^1\text{H-NMR}$ (400 MHz, CDCl_3 , ppm): δ 7.33 (d, $J = 8.7$ Hz, 2H), 7.29-7.25 (m, 4H), 7.09 (d, $J = 7.8$ Hz, 4H), 7.07 (d, $J = 7.4$ Hz, 2H), 6.96 (d, $J = 8.7$ Hz, 2H), 3.01 (s, 1H).

N-{4-[2-(4-Bromophenyl)ethynyl]phenyl}-diphenylamine (1)^{1d, 1e, 2} A mixture of compound **S-2** (0.54 g, 2.0

mmol), 4-bromiodobenzene (0.59 g, 2.1 mmol), Pd(PPh₃)₄ (116 mg, 0.1 mmol), CuI (19 mg, 0.1 mmol), 5 mL diisopropylamine and 16 mL toluene was stirred at room temperature under N₂ atmosphere overnight. Then the reaction mixture was poured into 100 mL water and aqueous phase was extracted with CH₂Cl₂ (3 × 30 mL). The combined organic phase was washed with aqueous NH₄Cl, brine and dried over anhydrous Na₂SO₄. The organic solvent was removed under reduced pressure and the crude product was purified by silica column chromatography eluting with petroleum ether/CH₂Cl₂ (10:1, v:v) to provide a white powder **1** (789 mg, 93%). M.p.: 159-160 °C. ¹H-NMR (400 MHz, CDCl₃, ppm): δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.36 (d, *J* = 8.8 Hz, 4H), 7.29 (t, *J* = 8.0 Hz, 4H), 7.12 (d, *J* = 8.0 Hz, 4H), 7.08 (t, *J* = 7.2 Hz, 2H), 7.00 (d, *J* = 8.4 Hz, 2H).

N-{4-[2-[4-(4,4,5,5-tetramethyl-[1,3,2]dioxaborolan-2-yl)phenyl]ethynylphenyl]-diphenylamine (2)}^{1d, 1e} A mixture of compound **1** (0.85 g, 2.0 mmol), bis(pinacolato)-diborane (0.56 g, 2.2 mmol), Pd(PPh₃)₂Cl₂ (70 mg, 0.1 mmol), PPh₃ (53 mg, 0.2 mmol) and KOAc (0.78 g, 8.0 mmol) in 20 mL toluene was refluxed at 110 °C under N₂ atmosphere for 24 h. After being cooled to the room temperature, the mixture was poured into 100 mL water and the organic layer was separated. The aqueous layer was extracted with CH₂Cl₂ (3 × 20 mL) and the combined organic layers were dried over anhydrous Na₂SO₄. After the organic solvent was removed under reduced pressure, the crude product was purified by silica gel column chromatograph eluting with petroleum ether/ethyl acetate (20:1, v:v) to provide a white solid **2** (0.75 g, 80%). M.p.: 160-163 °C. ¹H-NMR (400 MHz, CDCl₃, ppm): δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.8 Hz, 2H), 7.27 (t, *J* = 8.0 Hz, 4H), 7.11 (d, *J* = 7.6 Hz, 4H), 7.06 (t, *J* = 7.2 Hz, 2H), 7.00 (d, *J* = 8.8 Hz, 2H), 1.35 (s, 12H).

3,6-Dithiophen-2-yl-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (S-3)^{1c, 1e, 3}

A mixture of sodium (2.20 g, 96 mmol), FeCl₃ (0.1 g) and 48 mL dry tert-amyl alcohol was stirring at 120 °C under N₂ atmosphere until the sodium disappeared completely. Then 2-carbonitrile thiophene (5.28 g, 48 mmol) was added to the mixture solution. A solution of succinic acid dimethyl ester (2.80 g, 19.2 mmol) in 20 mL dry tert-amyl alcohol was added dropwise. After stirring at 120 °C for 24 h, 20 mL AcOH was added and kept stirring for another 1 hour. After cooling to room temperature, the precipitate was filtered, then washed with hot water and hot methanol to afford a red solid compound **S-3** (4.61 g, 80 %).

3,6-Dithien-2-yl-2,5-dioctylpyrrolo[3,4-c]pyrrole-1,4-dione (S-4)^{1c, 1e} A mixture of compound **S-3** (1.5 g, 5 mmol), K₂CO₃ (2.07 g, 15 mmol) and 50 mL anhydrous DMF was stirring at 60 °C under N₂ for 1 hour. Then 1-bromooctane (2.9 g, 15 mmol) was added to the mixture solution and kept stirring for 18 h. After cooling to room temperature, the mixture was poured into 500 mL water. The suspension was obtained by suction filtration. The crude product was purified by silica gel column chromatograph eluting with CH₂Cl₂ to afford a purple-brown solid **S-4** (2.13 g, 81%). M.p.: 139-143 °C. ¹H-NMR (400 MHz, CDCl₃, ppm): δ 8.47 (d, *J* = 4.0 Hz, 2H), 7.24 (d, *J* = 4.0 Hz, 2H), 3.98 (t, *J* = 7.8 Hz, 4H), 1.75-1.67 (m, 4H), 1.44-1.20 (m, 20H), 0.88-0.84 (m, 6H).

3-(5-Bromo-thiophen-2-yl)-2,5-dioctyl-6-(thiophen-2-yl)-pyrrolo[3,4-c]pyrrole-1,4-dione (3)^{1c} Compound **S-4** (0.53 g, 1.0 mmol) was dissolved in 40 mL CHCl₃ and a solution of NBS (0.19 g, 1.05 mmol) in 30 mL CHCl₃ was added dropwise to the solution. Then the mixture was stirred at room temperature overnight. The solvent was removed under reduced pressure and the crude product was purified by silica gel column chromatograph eluting with petroleum ether/CHCl₃ (1:2, v:v) to afford a purple solid **3** (391 mg, 65%). M.p.: 152-154 °C. ¹H NMR (400 MHz, CDCl₃, ppm): δ 8.94 (d, *J* = 4.0 Hz, 1H), 8.67 (d, *J* = 4.4 Hz, 1H), 7.65 (d, *J* = 4.8 Hz, 1H), 7.28 (t, *J* = 4.4 Hz, 1H), 7.23 (d, *J* = 4.4 Hz, 1H), 4.08-3.97 (m, 4H), 1.77-1.70 (m, 4H), 1.45-1.27 (m, 20H), 0.89-0.86 (m, 6H).

4-{5-[2,5-dioctyl-3,6-dioxo-4-(thiophen-2-yl)-2,3,5,6-tetrahydropyrrolo[3,4-c]pyrrol-1-yl]thiophen-2-yl}benzonitrile (4) A mixture of compound **3** (0.60 g, 1 mmol), p-cyanobenzeneboronic acid (0.15 mg, 1 mmol), Pd(PPh₃)₄ (58 mg, 0.05 mmol), Na₂CO₃ (1.06 g, 10 mmol), 20 mL toluene, 5 mL H₂O and 2.5 mL EtOH was stirred at 110 °C under N₂ atmosphere for 12 h. After being cooled to the room temperature, the solution was poured into 200 mL water and extracted with CH₂Cl₂ (3 × 30 mL). The combined organic layers were dried over

anhydrous Na₂SO₄. The organic solvent was evaporated under reduced pressure and the crude product was purified by silica gel column eluting with petroleum ether/CH₂Cl₂ (2:1, v:v) to afford a red solid **4** (0.59 g, 94%). M.p.: 185-187 °C. ¹H-NMR (400 MHz, CDCl₃, ppm): δ 8.97 (d, *J* = 4.0 Hz, 1H), 8.90 (d, *J* = 4.0 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 5.2 Hz, 2H), 7.57 (d, *J* = 4.4 Hz, 1H), 7.31 (t, *J* = 4.4 Hz, 1H), 4.12-4.06 (m, 4H), 1.77-1.74 (m, 4H), 1.45-1.18 (m, 20H), 0.87-0.83 (m, 6H).

4-{5-[4-(5-bromothiophen-2-yl)-2,5-dioctyl-3,6-dioxo-2,3,5,6-tetrahydropyrrolo[3,4-c]pyrrol-1-yl]thiophen-2-yl}benzotrile (5**)** Compound **4** (0.63 g, 1 mmol) was dissolved in 40 mL CHCl₃ and a solution of NBS (0.18 g, 1 mmol) in 30 mL CHCl₃ was added dropwise to the solution. Then the mixture was stirred at room temperature overnight. The solvent was removed under reduced pressure and the crude product was purified by silica gel column chromatograph eluting with CH₂Cl₂ to provide a dark red solid **5** (0.69 g, 98%). M.p.: 205-207 °C. ¹H-NMR (400 MHz, CDCl₃, ppm) δ 8.92 (d, *J* = 4.4 Hz, 1H), 8.72 (d, *J* = 4.0 Hz, 1H), 7.77-7.69 (m, 4H), 7.55 (d, *J* = 4.0 Hz, 1H), 7.24 (d, *J* = 4.4 Hz, 1H), 4.10 (t, *J* = 7.6 Hz, 2H), 4.01(t, *J* = 7.8 Hz, 2H), 1.77-1.68 (m, 4H), 1.44-1.27 (m, 20H), 0.88-0.84 (m, 6H).

3-(5-(4-((4-(diphenylamino)phenyl)ethynyl)phenyl)thiophen-2-yl)-2,5-dioctyl-6-(thiophen-2-yl)pyrrolo[3,4-c]pyrrole-1,4-dione (TPATDPP) A mixture of compound **2** (0.21 g, 0.45 mmol), compound **3** (0.24 g, 0.4 mmol), Pd(PPh₃)₄ (46 mg, 0.04 mmol), Na₂CO₃ (0.42 g, 4.0 mmol), 10 mL toluene, 2 mL H₂O and 1 mL EtOH was stirred at 110 °C under N₂ atmosphere for 12 h. After being cooled to the room temperature, the solution was poured into 100 mL water and extracted with CH₂Cl₂ (3 × 20 mL). The combined organic layers were dried over anhydrous Na₂SO₄ and the organic solvent was removed under reduced pressure. The crude product was purified by silica gel column chromatograph eluting with petroleum ether/CH₂Cl₂ (1:1, v:v) to provide a dark red solid **TPATDPP** (0.31 g, 89%). M.p.: 214-216 °C. ¹H-NMR (400 MHz, CDCl₃, ppm): δ 8.98 (d, *J* = 4.4 Hz, 1H), 8.94 (d, *J* = 3.2 Hz, 1H), 7.64 (m, 3H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 4.4 Hz, 1H), 7.39 (d, *J* = 8.8 Hz, 2H), 7.31-7.26 (m, 5H), 7.13 (d, *J* = 4.2 Hz, 4H), 7.08 (t, *J* = 7.4Hz, 2H), 7.02 (d, *J* = 8.4 Hz, 2H), 4.12-4.05 (m, 4H), 1.78-1.74 (m, 4H), 1.45-1.27 (m, 20H), 0.89-0.85 (m, 6H). ¹³C-NMR (400MHz, CDCl₃, ppm): δ 161.35, 161.22, 149.06, 148.16, 147.13, 139.63, 136.75, 135.25, 132.60, 132.36, 132.08, 130.59, 129.85, 129.43, 129.15, 129.08, 128.61, 125.87, 125.08, 124.88, 124.12, 123.67, 122.14, 115.66, 107.97, 107.89, 91.88, 88.43, 42.29, 31.80, 29.99, 29.22, 26.92, 22.64, 14.11. MALDI-TOF HRMS: 868.3917 [M+H⁺] (calcd for C₅₆H₅₈N₃O₂S₂: 868.3970).

4-(5-{4-[5-(4-{4-(diphenylamino)phenyl}ethynyl)phenyl]thiophen-2-yl}-2,5-dioctyl-3,6-dioxo-2,3,5,6-tetrahydropyrrolo[3,4-c]pyrrol-1-yl}thiophen-2-yl)benzotrile (TPATDPPCN) A mixture of compound **2** (0.21 g, 0.45 mmol), compound **5** (0.28 g, 0.4 mmol), Pd(PPh₃)₄ (46 mg, 0.04 mmol), Na₂CO₃ (0.42 g, 4.0 mmol), 10 mL distilled toluene, 2 mL H₂O and 1 mL EtOH was stirred at 110 °C under N₂ atmosphere for 12 h. After being cooled to the room temperature, the solution was poured into 100 mL water and extracted with CH₂Cl₂ (3 × 20 mL). The combined organic layers were dried over anhydrous Na₂SO₄ and the organic solvent was removed under reduced pressure. The crude product was purified by silica gel column chromatograph eluting with petroleum ether/CH₂Cl₂ (1:2, v:v) to provide a dark red solid **TPATDPPCN** (356 mg, 92%). M.P.: 237-239 °C. ¹H-NMR (400 MHz, CDCl₃, ppm) : δ 9.02 (d, *J* = 4.4 Hz, 1H), 8.92 (d, *J* = 4.0 Hz, 1H), 7.75 (d, *J* = 8.8 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.55-7.53 (m, 3H), 7.49 (d, *J* = 4.0 Hz, 1H), 7.39-7.37 (m, 4H), 7.31-7.26 (m, 4H), 7.14-7.12 (m, 4H), 7.10-7.06 (m, 2H), 7.02-7.00 (m, 2H), 4.11-4.10 (m, 4H), 1.79-1.74 (m, 4H), 1.46-1.28 (m, 20H), 0.88-0.85 (m, 6H). ¹³C-NMR (400MHz, CDCl₃, ppm): δ161.37, 161.08, 149.65, 148.25, 147.15, 146.31, 140.24, 138.32, 137.35, 137.25, 136.09, 132.90, 132.63, 132.26, 132.12, 130.95, 129.45, 129.22, 128.95, 126.36, 125.91, 125.13, 124.97, 124.37, 123.72, 122.13, 118.49, 115.62, 111.82, 109.00, 108.11, 92.07, 88.39, 42.34, 31.81, 30.06, 30.02, 29.21, 26.94, 22.63, 14.08. MALDI-TOF HRMS: 968.4143 [M⁺] (calcd for C₆₃H₆₀N₄O₂S₂: 968.4158).

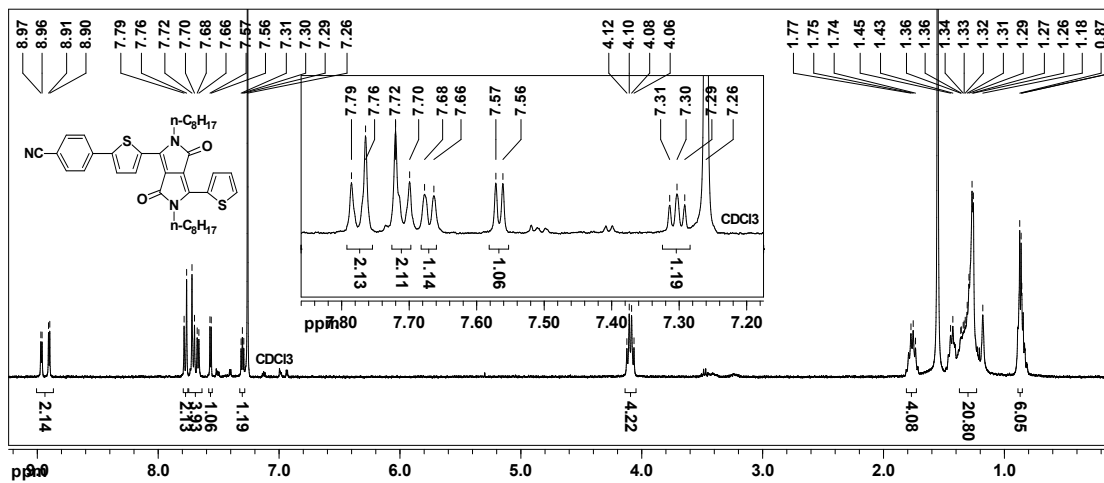
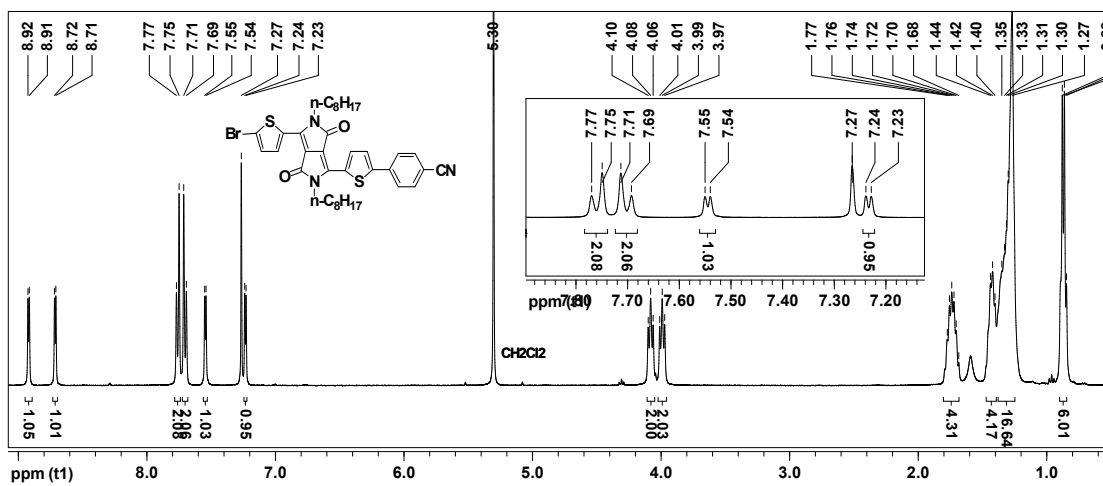


Fig. S4 ¹H-NMR spectrum of compound 4.



4. PV device fabrication and hole mobility (μ_h) measurement

4.1 PV device fabrication

Bulk heterojunction (BHJ) PV devices were fabricated by solution-processed spin-coating with a typical configuration of ITO/PEDOT:PSS/SMDs:PC₆₁BM (1:2, w/w)/Al. Firstly, the ITO glass was pre-cleaned in water, acetone, toluene and isopropyl alcohol for 10 min, respectively. Then a thin layer of poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS from Baytron P VP AI 4083) was spin-coated (4000 rpm, 60 s) onto the ITO glass. After being baked at 140 °C for 15 minutes, the substrate was transferred into a nitrogen-filled glove box. Subsequently, the active layer was spin-coated at 1500 rpm for 60 s with a blend solution of SMDs and PC₆₁BM (with the weight ratios of 1:2) on top of the ITO/PEDOT:PSS substrate. Finally, a 100 nm aluminium electrode was deposited by vacuum thermal evaporation (ca. 10⁻⁴ Pa) through a shadow mask, yielding six individual devices with 0.05 cm² nominal area.

4.2 Hole mobility (μ_h) measurement

In order to investigate the μ_h of the synthesized SMDs, J - V characteristics of the hole-only devices with a structure of ITO/PEDOT:PSS/SMDs:PC₆₁BM (1:2, w/w)/Au were fabricated and characterized in the dark. By fitting the J - V curves in a double logarithmic scale to a space-charge-limited currents (SCLC) model according to the Mott-Gurney law^{1d, 1e, 4}, the hole mobility can be derived:

$$J = (9/8) \varepsilon_0 \varepsilon_r \mu_h V^2 / L^3$$

where J is the current density, ε_0 is the permittivity of free space, ε_r is the relative permittivity and assumed as approximately 3.0, μ_h is the hole mobility, V is the effective voltage, and L is the thickness of the active layer.

5. UV-vis absorption spectra of TPATDPP and TPATDPPCN in CHCl₃ and the molar extinction coefficient

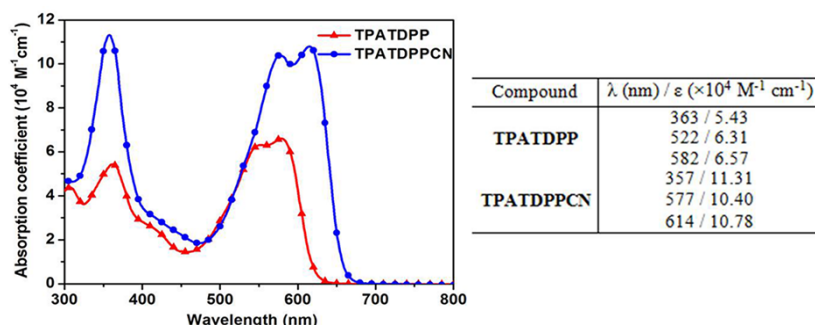


Fig. S10 UV-vis absorption spectra of TPATDPP and TPATDPPCN in CHCl₃ and the molar extinction coefficient in different wavelength.

6. IPCE spectra

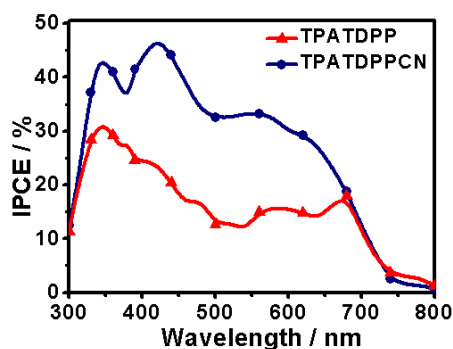


Fig. S11 The IPCE spectra of TPATDPP and TPATDPPCN.

7. X-ray Diffraction (XRD) patterns

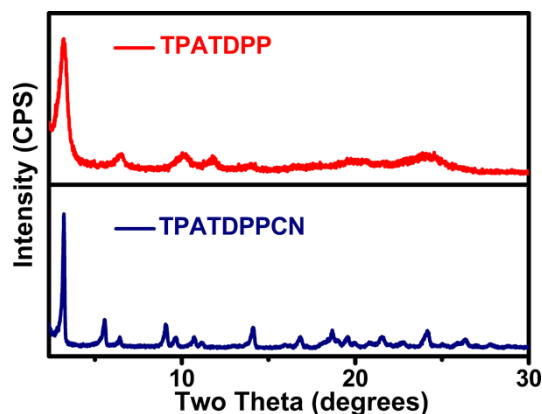


Fig. S12 XRD patterns of TPATDPP and TPATDPPCN.

8. AFM images

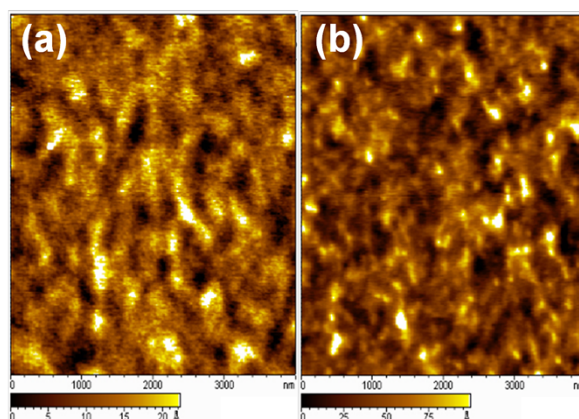


Fig. S13 AFM images of blended films based on TPATDPP:PC₆₁BM (1:2, w/w) (a) and TPATDPPCN:PC₆₁BM (1:2, w/w) (b).

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