

## Supporting Information

# Solution-Processed Benzotrithiophene-Based Donor Molecules for Efficient Bulk Heterojunction Solar Cells

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## Synthesis

### 2,3-Dibromo-5-nonanoylthiophene (1)

Under N<sub>2</sub>, anhydrous AlCl<sub>3</sub> (8.3 g, 62 mmol) was added portion-wise to an ice-cooled solution of 2,3-dibromothiophene (10.0 g, 41.3 mmol) and nonanoyl chloride (8.9 mL, 49.6 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (200 mL) for 15 min. The cooling bath was removed and then the mixture was stirred for 2 h at room temperature before being quenched with ice-cold 2 M HCl (400 mL). The aqueous phase was extracted with CH<sub>2</sub>Cl<sub>2</sub> and dried (MgSO<sub>4</sub>); the solvent was evaporated under reduced pressure to give a brown oil (15.4 g, 97.2%), which was used in the next step without further purification. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 7.47 (s, 1H), 2.80 (t, *J* = 7.2 Hz, 2H), 1.72–1.66 (m, 2H), 1.27 (m, 10H), 0.87 (t, *J* = 6.9 Hz, 3H). EIMS (*m/z*): calcd. for C<sub>13</sub>H<sub>18</sub>Br<sub>2</sub>OS 381.92, found 382 [M<sup>+</sup>].

### 5-Nonanoyl-2,3-bis(3-thienyl)thiophene (2).

Pd(PPh<sub>3</sub>)<sub>4</sub> (0.755 g, 0.909 mmol) was added under N<sub>2</sub> to a suspension of **1** (5.00 g, 13.1 mmol) and thiophene-3-boronic acid (3.70 g, 28.8 mmol) in 2 M Na<sub>2</sub>CO<sub>3</sub> (20 mL), toluene (60 mL), and EtOH (10 mL) and then the mixture was heated under reflux for 24 h. The reaction was quenched with water and then the aqueous phase was extracted with CH<sub>2</sub>Cl<sub>2</sub>, dried (MgSO<sub>4</sub>), and concentrated under reduced pressure. The residue was purified through column chromatography (SiO<sub>2</sub>, toluene). The title compound was obtained as a pale-yellow solid (10.8 g, 80%) after recrystallization (toluene/MeOH). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 7.68 (s, 1H), 7.30 (dd, *J* = 2.7, 3.0 Hz 1H), 7.29–7.27 (m, 2H), 7.21 (d, *J* = 2.4 Hz, 1H), 7.02–6.99 (m, 2H), 2.88 (t, *J* = 7.5 Hz, 2H), 1.78–1.74 (m, 2H), 1.33–1.26 (m, 10H), 0.86 (t, *J* = 6.9 Hz, 3H). EIMS (*m/z*): calcd for C<sub>21</sub>H<sub>24</sub>OS<sub>3</sub> 388.09, found 389 [M<sup>+</sup>].

### 5-Nonanoylbenzo[1,2-*b*:3,4-*b'*:5,6-*d''*]trithiophene (3)

Boron trifluoride diethyl etherate (1.25 mL, 10.0 mmol) was added to an ice-cooled solution of **2** (3.0 g, 7.71 mmol) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (300 mL) and then 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (2.10 g, 9.26 mmol) was added portion-wise over 10 min. The mixture was stirred at room temperature overnight and then the reaction was quenched through the addition of Zn (5.00 g, 77.1 mmol) and MeOH (150 mL); the mixture was again stirred overnight. After filtering, the filtrate was washed with water, dried (MgSO<sub>4</sub>), and concentrated under reduced pressure. The residue was purified through column chromatography (SiO<sub>2</sub>, toluene); subsequent recrystallization (EtOH) afforded the title compound as a yellow-orange solid (3.92 g, 58%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 8.29 (s, 1H), 7.74 (d, *J* = 5.4 Hz, 1H), 7.61 (d, *J* = 5.4 Hz, 1H), 7.55–7.51 (m, 2H), 3.06 (t, *J* = 7.2 Hz, 2H), 1.85–1.78 (m, 2H), 1.32–1.22 (m, 10H), 0.87 (t, *J* = 6.4 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 194.48, 142.06, 136.18, 133.51, 133.22, 132.21, 131.63, 131.12, 127.67, 125.46, 125.26, 122.63, 122.47, 39.39, 31.83, 29.68, 29.42, 29.39, 29.16, 24.83, 22.64, 14.09. EIMS (*m/z*): calcd for C<sub>21</sub>H<sub>22</sub>OS<sub>3</sub> 386.08, found 387 [M<sup>+</sup>].

### 5-Nonylbenzo[1,2-*b*:3,4-*b'*:5,6-*d''*]trithiophene (4)

KOH (4.35 g, 77.6 mmol) and hydrazine monohydrate (6.27 mL, 129 mmol) were added to a suspension of **3** (1.0 g, 2.58 mmol) in ethylene glycol (100 mL) and then the mixture was stirred at 190 °C over night. After cooling to room temperature, the mixture was poured into water and extracted with CH<sub>2</sub>Cl<sub>2</sub>; the combined extracts were dried (MgSO<sub>4</sub>) and concentrated. The residue was purified through column chromatography (SiO<sub>2</sub>, hexane) to give a white solid (0.81 g,

84.4%).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.69 (d,  $J = 5.4$  Hz, 1H), 7.56 (d,  $J = 5.4$  Hz, 1H), 7.49–7.46 (m, 2H), 7.42 (s, 1H), 3.0 (t,  $J = 4.8$  Hz, 2H), 1.84–1.76 (m, 2H), 1.32–1.26 (m, 10H), 0.87 (t,  $J = 6.4$  Hz, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  145.39, 132.85, 132.35, 131.50, 131.02, 130.78, 129.76, 124.77, 124.05, 122.76, 122.30, 119.37, 31.87, 31.53, 30.76, 29.69, 29.50, 29.39, 29.30, 29.12, 22.66, 14.10. EIMS ( $m/z$ ): calcd for  $\text{C}_{21}\text{H}_{24}\text{S}_3$  372.10, found 373 [ $\text{M}^+$ ].

### **2,8-Bis(trimethylstannyl)-5-nonylbenzo[1,2-*b*:3,4-*b'*:5,6-*d''*]trithiophene (5)**

A solution of *n*-BuLi in hexanes (1.6 M, 3.85 mL, 7.00 mmol) was injected slowly via syringe into a solution of **4** (1.00 g, 2.68 mmol) in anhydrous THF (100 mL) at  $-78$  °C under  $\text{N}_2$ . After stirring for 1 h at low temperature, the mixture was stirred at room temperature for 2 h. The mixture was cooled once again to  $-78$  °C and then a solution of trimethyltin chloride (1 M, 6.50 mL, 6.44 mmol) in THF was added; the mixture was then warmed slowly to room temperature and stirred overnight. The reaction was quenched through the addition of water and the aqueous phase extracted with hexane. The combined organic extracts were dried ( $\text{MgSO}_4$ ) and concentrated to afford a light-yellow oil (1.40 g, 75%), which was used in the next step without further purification.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.75 (s, 1H), 7.60 (s, 1H), 7.46 (s, 1H), 3.01 (t,  $J = 7.2$  Hz, 2H), 1.84–1.79 (m, 2H), 1.40–1.27 (m, 10H), 0.88 (t,  $J = 6.4$  Hz, 3H), 0.48 (s, 18H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  144.78, 138.86, 137.69, 134.62, 134.51, 132.64, 132.37, 130.60, 130.27, 129.92, 123.95, 119.53, 31.87, 31.52, 30.75, 29.69, 29.08, 25.59, 22.66, 14.10, – 8.17. MALDI-TOF MS ( $m/z$ ): calcd for  $\text{C}_{27}\text{H}_{40}\text{S}_3\text{Sn}_2$  700.03, found 700.03.

### **3,4'-Dioctyl-2,2'-bithiophene-5-carbaldehyde (6)**

Pd(PPh<sub>3</sub>)<sub>4</sub> (2 mol %) was added a solution of 5-bromo-4-octylthiophene-2-carbaldehyde (10.0 g, 33.0 mmol) and trimethyl(4-octylthien-2-yl)stannane (14.2 g, 39.6 mmol) in toluene (100 mL) under N<sub>2</sub> and then the mixture was heated under reflux overnight. After pouring the mixture into water (200 mL), the organic phase was extracted with CH<sub>2</sub>Cl<sub>2</sub>; the combined extracts were washed three times with distilled water, dried (MgSO<sub>4</sub>), and concentrated. The residue was purified through column chromatography (SiO<sub>2</sub>; hexanes/CH<sub>2</sub>Cl<sub>2</sub>, 6:1) to provide a yellow solid (10.9 g, 79.3%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 9.81 (s, 1H), 7.57 (s, 1H), 7.10 (s, 1H), 7.00 (s, 1H), 2.78 (t, *J* = 7.8 Hz, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 1.68–1.57 (m, 4H), 1.27 (m, 20H), 0.88 (t, *J* = 6.6 Hz, 6H). EIMS (*m/z*): calcd for C<sub>25</sub>H<sub>38</sub>OS<sub>2</sub> 418.23, found 419 [M<sup>+</sup>].

#### **5'-Bromo-3,4'-dioctyl-2,2'-bithiophene-5-carbaldehyde (7)**

Under a N<sub>2</sub> atmosphere and protection from light, *N*-bromosuccinimide (NBS, 4.70 g, 26.3 mmol) was added portion-wise to a solution of **6** (10.0 g, 23.9 mmol) in CHCl<sub>3</sub> (80 mL) and AcOH (80 mL) at 0 °C. The mixture was stirred for 4 h at room temperature and then water was added to quench the reaction. The aqueous phase was extracted three times with CH<sub>2</sub>Cl<sub>2</sub>; the combined extracts were washed sequentially with NaHCO<sub>3</sub> solution, brine, and water, dried (MgSO<sub>4</sub>), and then concentrated under reduced pressure. The residue was purified through column chromatography (SiO<sub>2</sub>; hexane/CH<sub>2</sub>Cl<sub>2</sub>, 6:1) to afford a yellow oil (8.5 g, 71.5%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 9.82 (s, 1H), 7.57 (s, 1H), 6.97 (s, 1H), 2.74 (t, *J* = 7.8 Hz, 2H), 2.57 (t, *J* = 7.6 Hz, 2H), 1.61–1.55 (m, 4H), 1.30–1.55 (m, 20H), 0.88 (t, *J* = 6.6 Hz, 6H). EIMS (*m/z*): calcd for C<sub>25</sub>H<sub>38</sub>OS<sub>2</sub>Br 496.214, found 497 [M<sup>+</sup>].

#### **3,4',4''-Trioctyl-2,2':5',2''-trithiophene-5-carbaldehyde (8)**

Compound **8** was prepared from **7** (5.00 g, 10.0 mmol), trimethyl(4-octylthien-2-yl)stannane (4.33 g, 12.1 mmol), and Pd(PPh<sub>3</sub>)<sub>4</sub> (2 mol %) using the procedure described for the preparation of **6**; purification through column chromatography (SiO<sub>2</sub>; hexane/CH<sub>2</sub>Cl<sub>2</sub>, 4:1) gave a yellow oil (4.98 g, 81.2%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 9.81 (s, 1H), 7.58 (s, 1H), 7.10 (s, 1H), 6.99 (s, 1H), 6.93 (s, 1H), 2.81 (t, *J* = 8.4 Hz, 2H), 2.74 (t, *J* = 7.8 Hz, 2H), 2.59 (t, *J* = 7.5 Hz, 2H), 1.70–1.58 (m, 6H), 1.32–1.27 (m, 30H), 0.89–0.85 (m, 9H). EIMS (*m/z*): calcd for C<sub>37</sub>H<sub>56</sub>OS<sub>3</sub> 612.34, found 613 [M<sup>+</sup>].

#### **5''-Bromo-3,4',4''-trioctyl-2,2':5',2''-trithiophene-5-carbaldehyde (9)**

Compound **9** was prepared from **8** (4.00 g, 6.52 mmol) and NBS (1.27 g, 7.17 mmol) using the same procedure as that described for the preparation of **7**; purification through column chromatography (SiO<sub>2</sub>; hexane/CH<sub>2</sub>Cl<sub>2</sub>, 4:1) gave a yellow oil (3.85 g, 85.3%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 9.82 (s, 1H), 7.58 (s, 1H), 7.09 (s, 1H), 6.84 (s, 1H), 2.80 (t, *J* = 8.1 Hz, 2H), 2.74 (t, *J* = 7.8 Hz, 2H), 2.59 (t, *J* = 7.5 Hz, 2H), 1.64–1.58 (m, 6H), 1.33–1.27 (m, 30H), 0.88–0.86 (m, 9H). EIMS (*m/z*): calcd for C<sub>37</sub>H<sub>55</sub>OS<sub>2</sub>Br 690.25, found 691 [M<sup>+</sup>].

#### **Compound 10**

Octyl cyanoacetate (8.20 mL, 60.20 mmol) was added under N<sub>2</sub> to a solution of **7** (3.00 g, 6.02 mmol) in dry CHCl<sub>3</sub> (30 mL) and piperidine (3 drops) and then the resulting solution was stirred for 36 h at room temperature. The reaction mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> and washed with water, dried (MgSO<sub>4</sub>), and concentrated under reduced pressure. The residue was purified through column chromatography (SiO<sub>2</sub>; hexane/CH<sub>2</sub>Cl<sub>2</sub>, 6:1) to afford a yellow solid (4.1 g, 90.5%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 8.19 (s, 1H), 7.54 (s, 1H), 7.02 (s, 1H), 4.28 (t, *J* = 6.6 Hz,

2H), 2.74 (t,  $J = 7.8$  Hz, 2H), 2.56 (t,  $J = 7.6$  Hz, 2H), 1.71–1.54 (m, 6H), 1.31–1.27 (m, 30H), 0.89–0.85 (m, 9H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  163.01, 147.0, 144.57, 143.08, 141.10, 140.67, 139.51, 137.80, 133.02, 128.59, 115.88, 111.75, 98.04, 66.58, 31.85, 30.15, 29.66, 29.46, 29.32, 29.21, 29.18, 29.12, 28.52, 25.77, 22.63, 14.08. MALDI-TOF MS ( $m/z$ ): calcd for  $\text{C}_{36}\text{H}_{56}\text{BrNO}_2\text{S}_2$  677.29, found 677.2.

### Compound 11

Compound **11** was prepared from **9** (3.00 g, 4.33 mmol) and octyl cyanoacetate (5.90 mL, 43.4 mmol) using the same procedure as that described for the preparation of **10**; purification through column chromatography ( $\text{SiO}_2$ ; hexane/ $\text{CH}_2\text{Cl}_2$ , 4:1) gave a red liquid (3.10 g, 82.2%).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ): 8.19 (s, 1H), 7.55 (s, 1H), 7.15 (s, 1H), 6.85 (s, 1H), 4.28 (t,  $J = 6.9$  Hz, 2H), 2.80 (t,  $J = 7.5$  Hz, 2H), 2.71 (t,  $J = 7.5$  Hz, 2H), 2.56 (t,  $J = 7.8$  Hz, 2H), 1.77–1.64 (m, 8H), 1.33–1.27 (m, 36H), 0.89–0.86 (m, 12H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  163.11, 146.0, 142.67, 141.69, 140.92, 140.58, 140.43, 134.62, 132.82, 132.77, 132.45, 130.62, 127.15, 115.98, 109.49, 97.63, 66.52, 31.82, 31.74, 30.56, 30.12, 29.66, 29.51, 29.43, 29.34, 29.22, 29.15, 29.13, 28.53, 28.46, 25.77, 22.63, 14.07. MALDI-TOF MS ( $m/z$ ): calcd for  $\text{C}_{48}\text{H}_{74}\text{BrNO}_2\text{S}_3$  681.21, found 871.2.

### BT4OT

A 50-mL two-neck flask charged with **5** (0.30 g, 0.40 mmol), **10** (0.73 g, 1.0 mmol), and  $\text{Pd}(\text{PPh}_3)_4$  (5 mol %) was subjected to three cycles of vacuum purging followed by refilling with  $\text{N}_2$ . Anhydrous toluene (20 mL) was added via syringe and then the mixture was heated under reflux for 36 h. After cooling, the mixture was filtered through Celite to remove the metal

catalyst and then the solids were washed with CH<sub>2</sub>Cl<sub>2</sub>. The combined solvents were concentrated under reduced pressure; the residue was purified through column chromatography (SiO<sub>2</sub>; hexane/CH<sub>2</sub>Cl<sub>2</sub>, 6:1) to obtain a product, which was washed with hexane and acetone to afford a dark powder (0.34 g, 50.6%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 8.22 (s, 2H), 7.72 (s, 1H), 7.60–7.57 (m, 3H), 7.57 (m, 1H), 7.46 (s, 1H), 7.30 (s, 2H), 4.33 (t, *J* = 7.2 Hz, 4H), 3.07 (t, *J* = 7.8 Hz, 2H), 2.94–2.86 (m, 8H), 1.86–1.79 (m, 14H), 1.34 (m, 72H), 0.94–0.91 (m, 21H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 163.13, 145.94, 145.87, 141.86, 141.17, 140.99, 140.52, 140.47, 140.31, 134.95, 134.17, 133.99, 133.18, 132.83, 132.69, 132.61, 132.31, 131.83, 131.23, 130.97, 130.91, 130.83, 130.01, 129.18, 128.89, 120.89, 120.16, 119.36, 116.62, 97.41, 66.50, 31.87, 31.84, 31.74, 31.57, 30.82, 30.53, 30.50, 30.08, 29.62, 29.56, 29.51, 29.48, 29.44, 29.42, 29.36, 29.29, 29.23, 29.16, 29.43, 28.54, 25.78, 22.64, 22.62, 14.08. MALDI-TOF MS (*m/z*): calcd for C<sub>93</sub>H<sub>130</sub>N<sub>2</sub>O<sub>4</sub>S<sub>7</sub> [M]<sup>+</sup> 1563.807, found 1563.81. Anal. calcd for C<sub>93</sub>H<sub>130</sub>N<sub>2</sub>O<sub>4</sub>S<sub>7</sub>: C, 71.40; H, 8.38; N, 1.79; found: C, 71.61; H, 8.20; N, 1.81.

## BT6OT

**BT6OT** was prepared from **5** (0.30 g, 0.40 mmol), **11** (0.87 g, 1.0 mmol), and Pd(PPh<sub>3</sub>)<sub>4</sub> (5 mol %), using the same procedure as that described for the preparation of BT4OT, as a dark powder (0.40 g, 47.7%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): 8.19 (s, 2H), 7.67 (s, 1H), 7.57–7.52(m, 3H), 7.42 (s, 1H), 7.19 (s, 2H), 7.06 (s, 2H), 4.28 (t, *J* = 6.9 Hz, 4H), 2.80 (t, *J* = 7.5 Hz, 4H), 2.71 (t, *J* = 7.5 Hz, 4H), 2.56 (t, *J* = 7.8 Hz, 4H), 1.84–1.73 (m, 16H), 1.50–1.10 (m, 96H), 0.89–0.87 (m, 27H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 163.13, 145.94, 145.67, 141.86, 141.17, 140.99, 140.52, 140.47, 140.31, 134.95, 134.17, 133.99, 133.22, 133.18, 132.83, 132.69, 132.61, 132.31, 131.83, 131.23, 130.97, 130.91, 130.83, 130.01, 129.18, 128.89, 120.89, 120.16, 119.36, 116.02, 97.41,

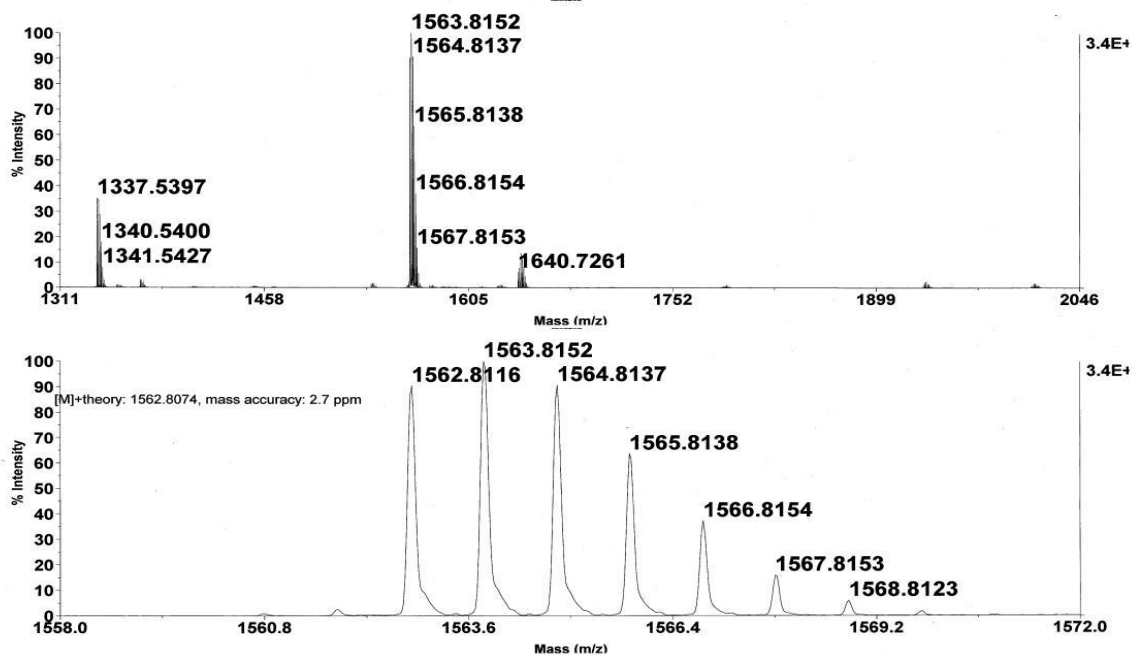






Fig. S3. Mass spectra of (a) BT4OT and (b) BT6OT.

(a)



(b)

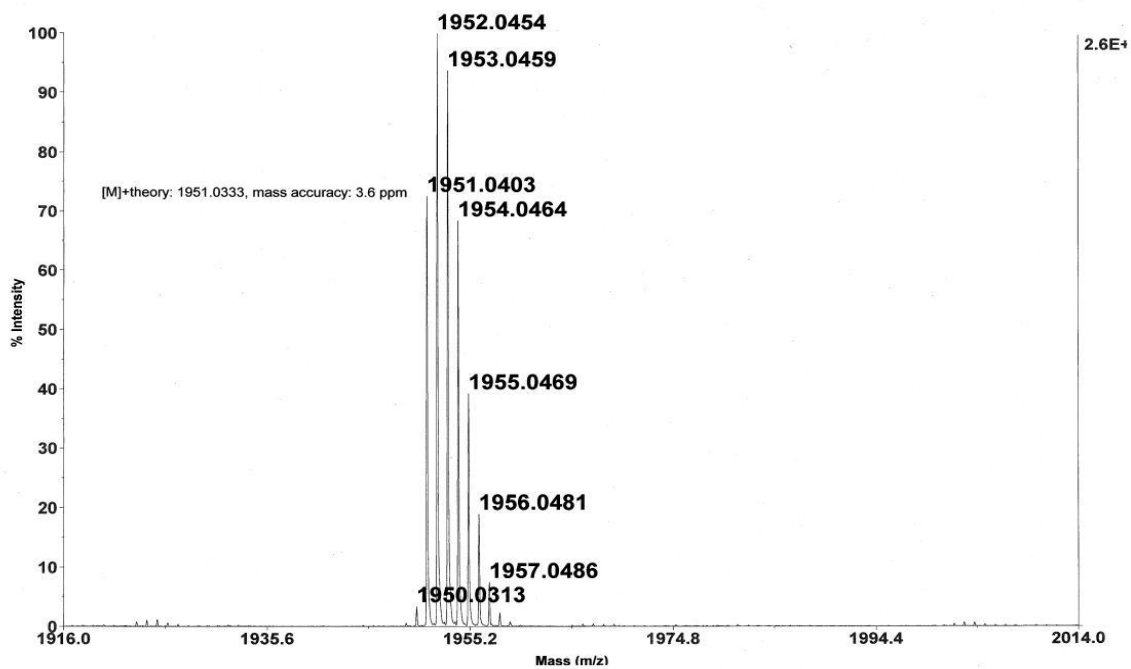


Fig. S4 Absorption spectra of BT4OT (1 wt%) and BT4OT/PC<sub>61</sub>BM (1:0.75, w/w) spin-coated from CHCl<sub>3</sub> on glass substrates.

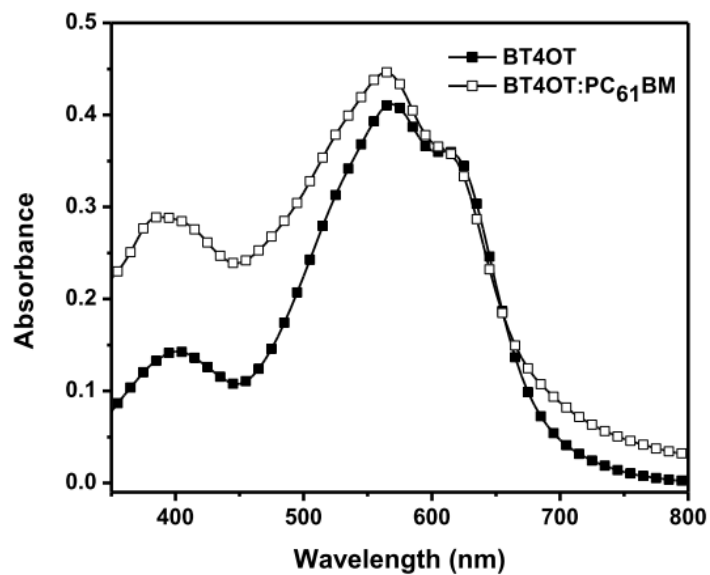


Fig. S5 Absorption spectra of BT6OT (1 wt%), BT6OT:PC<sub>61</sub>BM (1:0.75, w/w) and BT6OT:PC<sub>71</sub>BM (1:0.75, w/w) spin-coated from CHCl<sub>3</sub> on glass substrates.

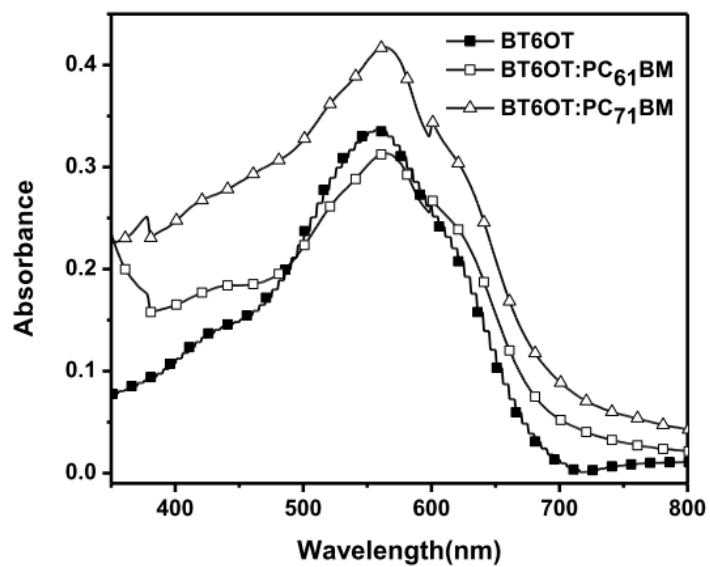


Fig. S6  $J$ - $V$  curves of BHJ solar cells incorporating BT4OT:PC<sub>61</sub>BM films with blend ratios of 1:0.5, 1:0.75, and 1:1 (w/w) under AM 1.5 G illumination (100 mW/cm<sup>2</sup>).

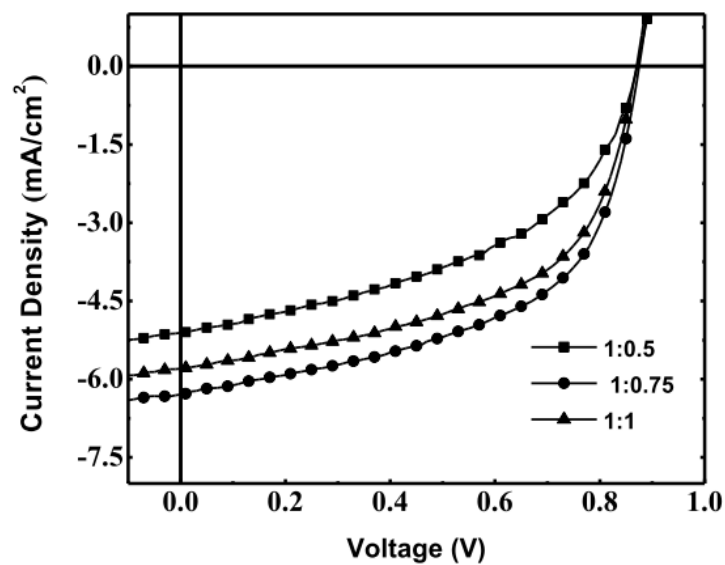


Fig. S7.  $J$ - $V$  curves of BHJ solar cells incorporating BT6OT:PC<sub>61</sub>BM films with blend ratios of 1:0.5, 1:0.75, and 1:1 (w/w) under AM 1.5 G illumination (100 mW/cm<sup>2</sup>).

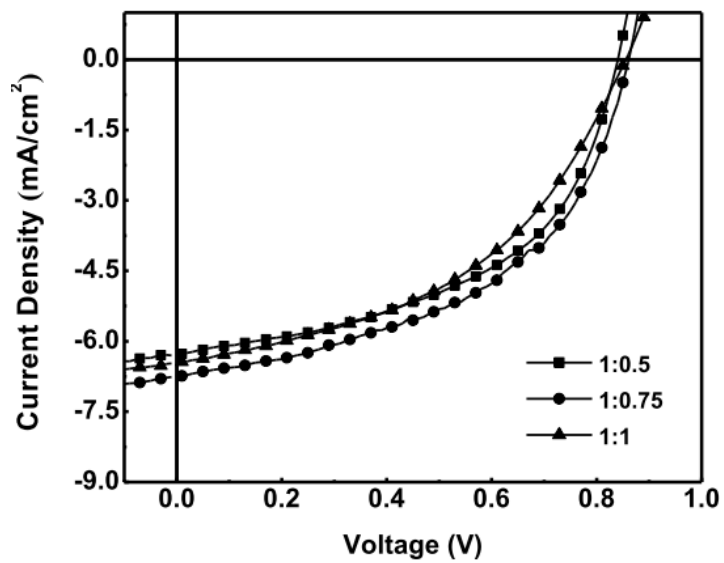


Fig. S8  $J$ - $V$  curves of (a) hole and (b) electron mobility devices based on BT6OT films incorporating various weight ratios of PC<sub>71</sub>BM and various amounts (vol%) of CN.

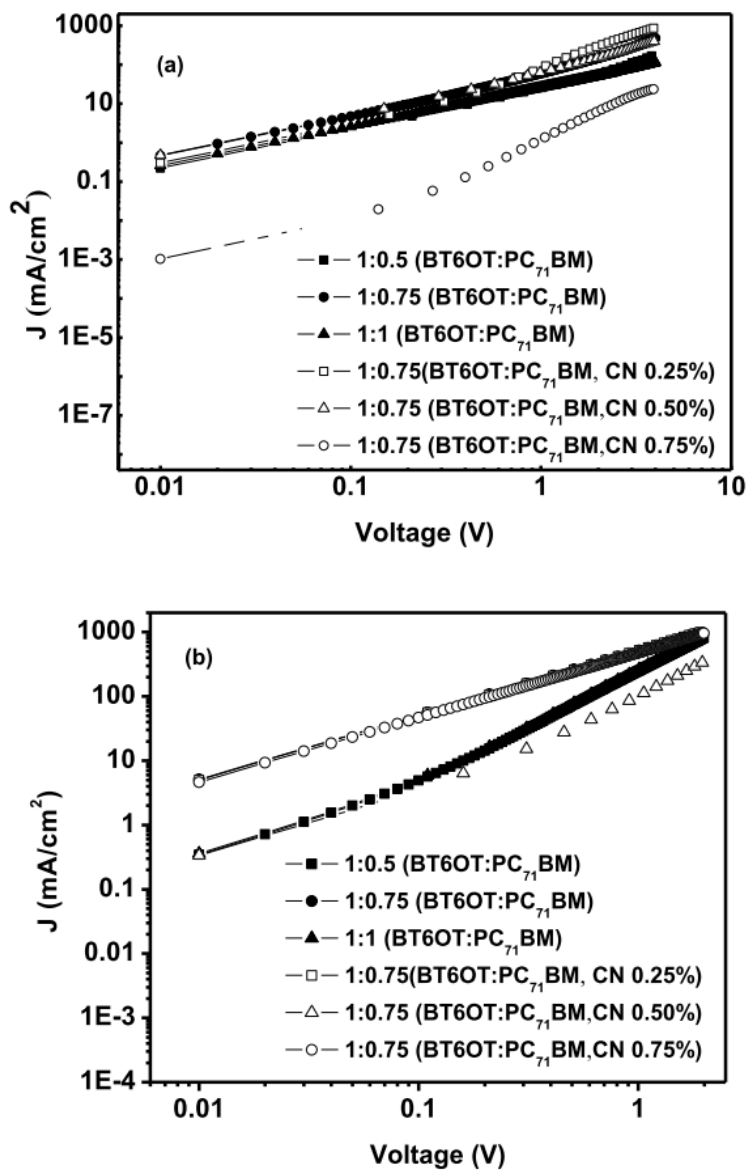
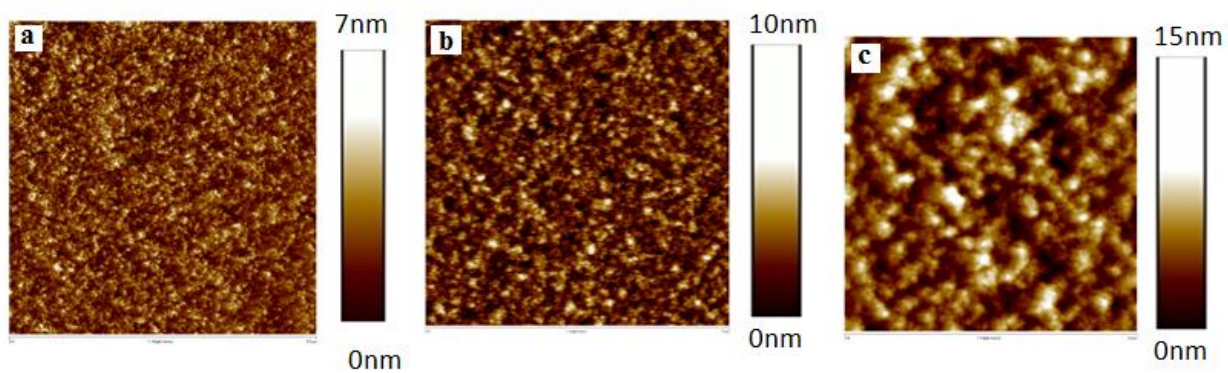




Fig. S9 AFM images of BT6OT:PC<sub>71</sub>BM film spin-coated from CHCl<sub>3</sub> solutions at blend ratios of (a) 1:0.5, (b) 1:0.75, and (c) 1:1 (w/w); root mean square roughnesses ( $R_{\text{rms}}$ ) were (a) 0.79, (b) 1.54, and (c) 2.61 nm.



**Table S1.** Performance of BHJ photovoltaic devices incorporating films of BT4OT or BT6OT blended with PC<sub>61</sub>BM at various weight ratios

Weight ratio of small molecule to PC <sub>61</sub> BM <sup>a</sup>	Blend ratio (w/w)	V <sub>oc</sub> (V)	J <sub>sc</sub> (mA/cm <sup>2</sup> )	FF (%)	PCE (%)
BT4OT	1:0.50	0.87	5.10	49.4	2.19
BT4OT	1:0.75	0.88	6.32	53.6	2.98
BT4OT	1:1	0.87	5.79	51.2	2.58
BT6OT	1:0.50	0.84	6.26	51.9	2.72
BT6OT	1:0.75	0.86	6.78	54.8	3.19
BT6OT	1:1	0.85	6.47	49.2	2.70

<sup>a</sup> Device with Ca/Al as cathode.