Electronic Supplementary Material (ESI) for Energy & Environmental Science.

# Quinoxaline-benzothiadiazole Heterotrimer Enabling Organic Solar Cells with Extraordinary Efficiency and Stability

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## Materials and synthesis:

PM6 was purchased from Solarmer Materials Inc.(Beijing, China). Chloroform (CF) was purchased from Sigma Aldrich. Anhydrous tetrahydrofuran was purchased from Energy Chemical. 2-methoxynaphthalene (2-MN) was purchased from Aladdin. All reagents and chemicals were purchased from commercial sources and used without further purification. Compound 1[1] and L8-BO[2] were synthesized according to previously reported literatures. The detailed synthetic procedures and characterizations of the oligomers are described in the following sections.

### Synthesis of compound Tri-BT

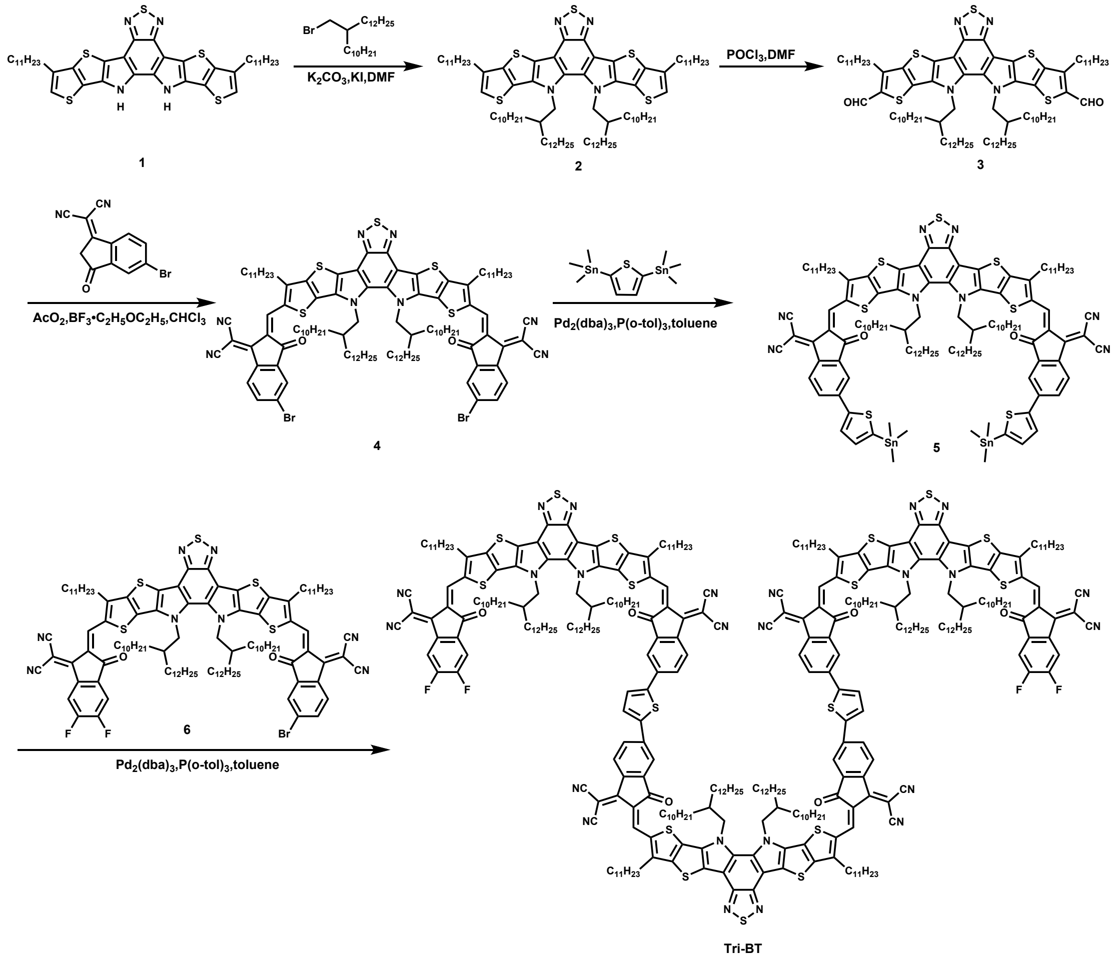

Figure S1. Synthetic route for Tri-BT acceptor.

**Compound 2:** Under nitrogen protection, compound1 (1 g, 1.34 mmol)，$K_2CO_3$ (8 eq, 1.48 g), KI (8 eq, 1.8 g) and 11-(bromomethyl)tricosane (8 eq, 4.47 g) were dissolved in N,N-dimethylformamide (15mL). The reaction mixture was heated at

80°C and stirred overnight. After cooling to room temperature, the mixture was added water and extracted with dichloromethane for three times. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using petroleum ether as eluent to give Compound 2 as a dark red solid (1.73 g, 91%).

**Compound 3:** Under nitrogen protection, $POCl_3$ (0.5 mL) was added dropwise to a solution of anhydrous N,N-dimethylformamide (DMF) (4 mL) at 0 °C and stirred at room temperature for 30 min. Then, compound 2 (347 mg, 0.25 mmol) dissolved in 1,2-dichloroethane (5 mL) was added to the mixture and stirred at 80ºC overnight. The reaction mixture is cooled to room temperature, and then $CH_3COOK$ (aq) is dropped into the reaction solution and was stirred for 30 minutes. The mixture was added water and extracted with dichloromethane for three times. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using petroleum ether: dichloromethane (5:1) as eluent to give Compound 3 as a yellow solid (288.5 mg, 80%).

**Compound 4:** To a solution of compound 3 (136 mg, 0.09 mmol) in toluene (2 mL) was added 2-(5-bromo-3-oxo-2, 3-dihydro-1H-inden-1-ylidene)malononitrile (2 eq, 50.3 mg), boron trifluoride diethyl etherate (0.09 mL) and acetic anhydride (0.09 mL) in turn at room temperature. The mixture was stirred for 30 min. Then the solution was cooled to room temperature and added into 150 mL methanol. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using trichloromethane: petroleum ether (1:1) as eluent to give Compound 4 as a blue solid (148.2 mg, 81%).

**Compound 5:** Under the protection of nitrogen, compound 4 (46 mg, 0.02 mmol), 2,5-bis(tributylstannanyl)thiophene (10 eq, 94.9 mg), $Pd_2(dba)_3$ (0.03 eq, 0.6 mg) and $P(o\text{-}tol)_3$ (0.3 eq, 2.1 mg) were dissolved in toluene (5 mL). After stirring at 70ºC for 1h, the reaction mixture was cooled to room temperature and then poured into methanol for precipitation. The sediment was filtered and collected, and the crude product (43 mg) was obtained by rinsing with ethanol.

**Compound 6:** To a solution of compound 3 (150 mg, 0.10 mmol) in toluene (10 mL) was added 2-(5-bromo-3-oxo-2, 3-dihydro-1H-inden-1-ylidene) malononitrile (1eq, 27.7 mg), 2-(5,6-difluoro-3-oxo-2,3-dihydro-1H-inden-1-ylidene)malononitrile (1eq, 23.4 mg), boron trifluoride diethyl etherate (0.1 mL) and acetic anhydride (0.1 mL) in turn at room temperature. The mixture was stirred for 30 min. Then the solution was cooled to room temperature and added into 150 mL methanol. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using trichloromethane: petroleum ether (1:1) as eluent to give Compound 4 as a blue solid (88.9 mg, 45%).

**Compound Tri-BT:** Under the protection of nitrogen, compound 5 (46 mg, 0.02 mmol), compound 6 (2 eq, 77.1 mg), $Pd_2(dba)_3$ (0.03 eq, 0.5 mg) and $P(o\text{-}tol)_3$ (0.3 eq, 1.8 mg) were dissolved in toluene (5 mL). The mixture was heated to reflux and stirred overnight. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using dichloromethane: petroleum ether (3:1) as eluent to give compound Tri-BT as a dark solid (24.1 mg, 21%).

$^1$H NMR (500 MHz, $CDCl_3$) δ 9.13 – 8.61 (m, 12H), 8.18 – 7.81 (m, 7H), 7.64 – 7.49 (m, 6H), 5.01– 4.74 (m, 12H), 3.41 – 2.80 (m, 12H), 2.38 – 2.13 (m, 8H), 1.87 – 1.68 (m, 16H), 1.25– 1.10 (m, 325H), 0.88 – 0.79 (m, 66H).

MS (MALDI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{368}H_{464}F_{10}N_{24}O_6S_{14}$, 5958.75, found: 5958.756.

## Synthesis of compound Tri-Qx

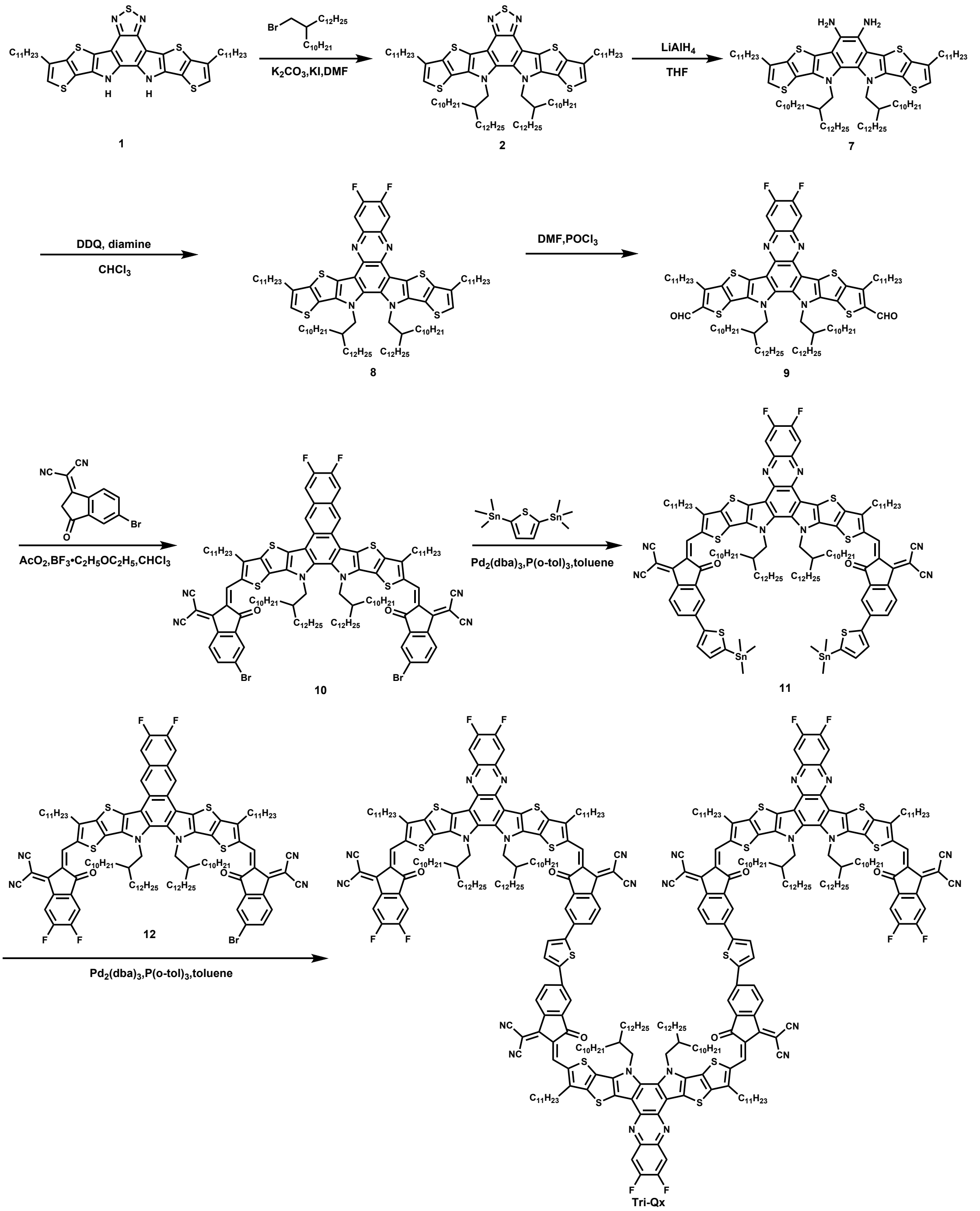

**Figure S2.** Synthetic route for Tri-Qx acceptor.

**Compound 7:** Under the protection of argon, $LiAlH_4$ (10 eq, 133.6 mg) was added to a solution of compound 2 (500 mg, 0.35 mmol) in tetrahydrofuran (THF, 10 mL). The resulting mixture was stirred and heated to 82ºC then refluxed for 12 h. After being cooled to 0ºC, water (35 mL) was slowly dropped into the mixture and extracted with dichloromethane. After removing the solvent, compound 7 was obtained as a black oily liquid.

**Compound 8:** The compound 7 was dissolved in chloroform (40 mL), then 3-dichloro-5, 6-dicyano-1, 4-141benzoquinone (1.15 eq, 91.9mg) and 1, 2-diaminobenzene (5.0 eq, 253.7 mg) were added to the solution. The reaction is stirred overnight at room temperature. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using dichloromethane: petroleum ether (1:2) as eluent to give compound 8 as a red solid (206.0 mg, 39%).

**Compound 9:** Under nitrogen protection, $POCl_3$ (0.5 mL) was added dropwise to a solution of anhydrous N,N-Dimethylformamide (DMF, 5 mL) at 0°C and stirred at room temperature for 40 min. Then, compound8 (110 mg, 0.07 mmol) dissolved in 1,2-dichloroethane (3 mL) was added to the mixture and stirred at 80ºC overnight. The reaction mixture was allowed to cool to room temperature and the mixture was poured into ice water, neutralized with $Na_2CO_3$ (aq), and then extracted with dichloromethane.

After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using petroleum ether: dichloromethane (5:1) as eluent to give Compound 9 as a red solid (114.1 mg, 79%).

**Compound 10:** To a solution of compound 9 (110 mg, 0.07 mmol) in toluene (10 mL) was added 2-(5-bromo-3-oxo-2, 3-dihydro-1H-inden-1-ylidene)malononitrile (2eq, 38.6 mg), boron trifluoride diethyl etherate (0.07 mL) and acetic anhydride (0.07 mL) in turn at room temperature. The mixture was stirred for 30 min. Then the solution was cooled to room temperature and added into 150 mL methanol. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using trichloromethane: petroleum ether (1:1) as eluent to give Compound 10 as a blue solid (119.6 mg, 82%).

**Compound 11:** Under the protection of nitrogen, compound 10 (50 mg, 0.02 mmol), 2,5-bis(tributylstannanyl) thiophene (10 eq, 99.2 mg), $Pd_2(dba)_3$ (0.03 eq, 0.7 mg) and $P(o\text{-}tol)_3$ (0.3 eq, 2.2 mg) were dissolved in toluene (15 mL). After stirring at 70ºC for 1h, the reaction mixture was cooled to room temperature and then poured into methanol for precipitation. The sediment was filtered and collected, and the crude product (49 mg) was obtained by rinsing with ethanol.

**Compound 12:** To a solution of compound 11 (300 mg, 0.20 mmol) in toluene (10 mL) was added 2-(5-bromo-3-oxo-2, 3-dihydro-1H-inden-1-ylidene) malononitrile (1eq, 52.6mg), 2-(5,6-difluoro-3-oxo-2,3-dihydro-1H-inden-1-ylidene)malononitrile (1eq, 44.3mg), boron trifluoride diethyl etherate (0.2 mL) and acetic anhydride (0.2 mL) in turn at room temperature. The mixture was stirred for 30 min. Then the solution was cooled to room temperature and added into 150 mL methanol. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using trichloromethane: petroleum ether (1:1) as eluent to give Compound 12 as a blue solid (183.1 mg, 47%).

**Compound Tri-Qx:** Under the protection of nitrogen, compound 7 (49 mg, 0.02 mmol), compound 8 (2 eq, 82.6 mg), $Pd_2(dba)_3$ (0.03 eq, 0.6 mg) and $P(o\text{-}tol)_3$ (0.3eq, 1.9 mg) were dissolved in toluene (5 mL). The mixture was heated to reflux and stirred overnight. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using $CH_2Cl_2$: petroleum ether (3:1) as eluent to give compound Tri-Qx as a dark solid. (29.6 mg, 24%).

$^1$H NMR (500 MHz, $CDCl_3$) δ 9.22 – 8.46 (m, 12H), 8.24 – 7.97 (m, 7H), 7.68 – 7.50 (m, 6H), 5.09– 4.74 (m, 12H), 3.41 – 2.88 (m, 12H), 2.45 – 2.12 (m, 8H), 1.98 – 1.79 (m, 16H), 1.20– 1.01 (m, 325H), 0.80 – 0.70 (m, 65H).

MS (MALDI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{350}H_{458}F_4N_{24}O_6S_{17}$, 5718.69, found: 5718.690.

## Synthesis of compound TQT

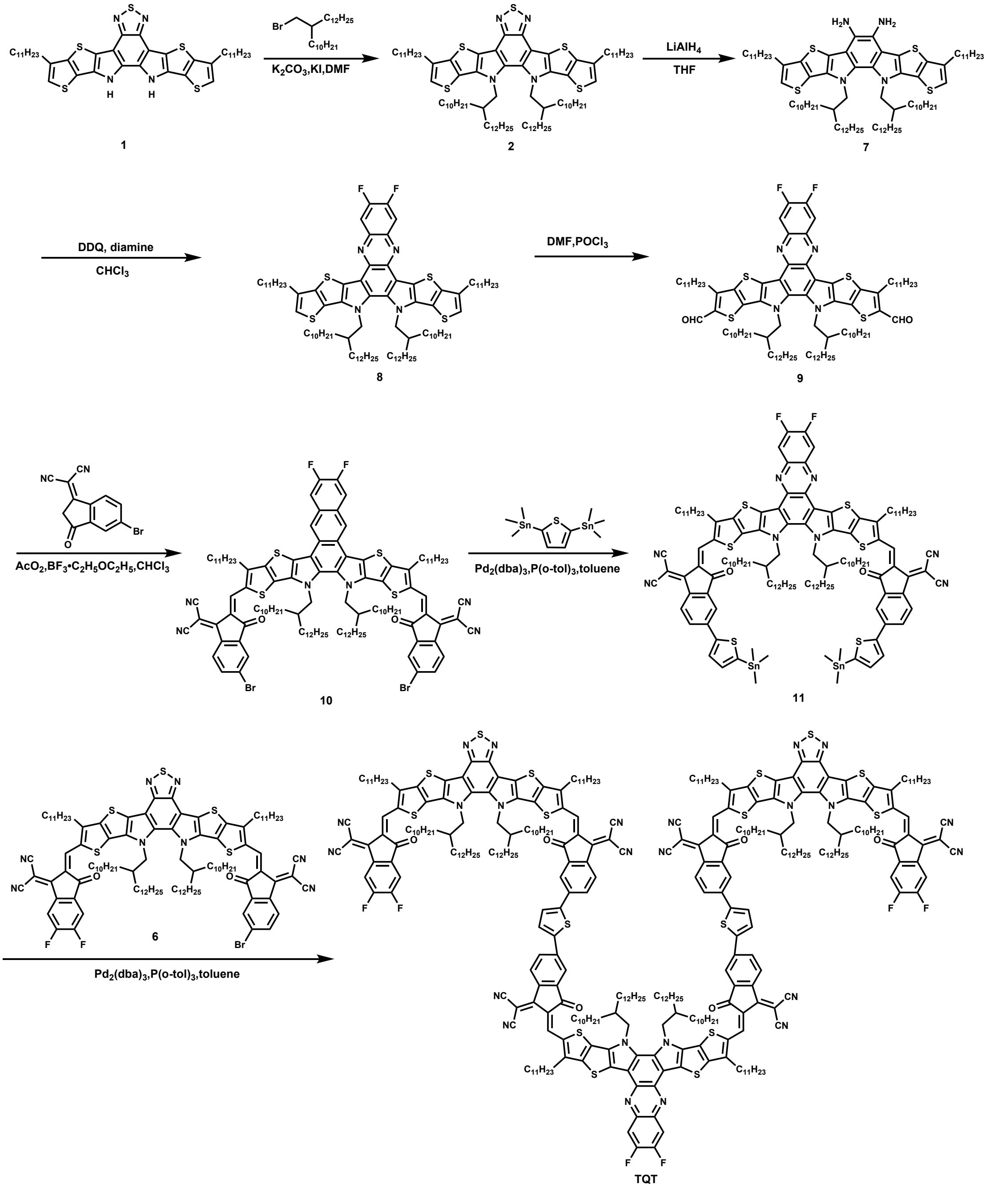

**Figure S3.** Synthetic route for TQT acceptor.

**Compound TQT:** Under the protection of nitrogen, compound 11 (46 mg, 0.02 mg), compound 6 (2 eq, 75 mg), $Pd_2(dba)_3$ (0.03eq, 0.5 mg) and $P(o\text{-}tol)_3$ (0.3 eq, 1.8 mg) were dissolved in toluene (5 mL). The mixture was heated to reflux and stirred overnight. After removal of the solvent, the crude product was purified via column chromatography (silica gel) by using dichloromethane: petroleum ether (3:1) as eluent to give compound TQT as a dark solid. (26.7 mg, 23%).

$^1$H NMR (500 MHz, $CDCl_3$) δ 9.13 – 8.54 (m, 12H), 8.24 – 7.91 (m, 7H), 7.71 – 7.45 (m, 6H), 4.97– 4.73 (m, 12H), 3.35 – 2.80 (m, 12H), 2.34 – 2.05 (m, 8H), 1.84 – 1.71 (m, 16H), 1.19– 1.01 (m, 325H), 0.79 – 0.70 (m, 65H).

MS (MALDI-TOF) *m/z*: $[M + H]^+$ calcd for $C_{356}H_{460}F_6N_{24}O_6S_{16}$, 5798.71, found: 5798.711.

## General characterization

$^1$H spectra were measured on a Bruker Avance-600 spectrometer. The mass spectra were measured using Bruker Daltonics Biflex III MALDI-TOF Analyzer in the MALDI mode. Absorption spectra were recorded on a Shimadzu UV-3700 spectrophotometer. Cyclic voltammetry was done by using a Shanghai Chenhua CHI660E voltammetric analyzer under argon in an anhydrous acetonitrile solution of tetra-n-butylammonium hexafluorophosphate (0.1 M). A glassy-carbon electrode was used as the working electrode, a platinum-wire was used as the counter electrode, and a $Ag/Ag^+$ electrode was used as the reference electrode. The materials was coated onto glassy-carbon electrode and all potentials were corrected against $Fc/Fc^+$. Device current density-voltage (*J-V*) characteristics were recorded using a Keithley 2400 Source Measure Unit. The photocurrent was tested under AM 1.5G illumination at 100 mW $cm^{-2}$ using a solar simulator (SS-F5-3A, Enlitech). The light intensity was calibrated using a standard Si solar cell (SRC2020, Enlitech). The EQE spectra were measured by using a solar-cell spectral-response measurement system (QE-R3011, Enlitech). AFM was performed on a Dimension Icon AFM (Bruker) using tapping mode.

## Device fabrication

Organic solar cells (OSCs) were fabricated with a conventional architecture of indium tin oxide (ITO)/ [2-(9H-carbazol-9-yl)ethyl] phosphonic acid (2PACz) /Active Layer/ poly[(9,9-bis(3’-(N,N-dimethylamino)propyl)-2,7-fluorene)- alt-5,5’-bis(2,2’-thiophene)-2,6-naphthalene-1,4,5,8-tetracaboxylic-N,N’-di(2-ethylhexyl)imide](PNDIT-F3N)/Ag. Initially, the ITO substrate is subjected to a pre-cleaning process using an ultrasonic bath, followed by 15-minute immersions in detergent, deionized water, acetone, and isopropanol. Subsequently, it is dried overnight in an oven. Prior to utilization, plasma treatment is employed for 2 minutes. The PEDOT:PSS is spin-coated on the top surface of the ITO at a speed of 4000 rpm for 30 seconds, resulting in a film thickness of approximately 40 nm. This is followed by a 15-minute thermal annealing at 150ºC. The PM6 and acceptor are dissolved in chloroform at a 1:1 mass ratio, and a film is prepared through spin-coating within a glovebox.

**Space-charge-limited current measurement**

The charge transport properties of the blend film were investigated by a space-charge-limited current method. The hole-only devices were fabricated with a configuration of ITO/PEDOT:PSS/active layer/$MoO_3$/Ag, and the electron-only devices were fabricated with a structure of ITO/ZnO/ active layer /PNDIT-F3N-Br/Ag. The mobility was determined by fitting the dark current to the model of a single carrier SCLC, which is described by: $J=9\varepsilon 0\varepsilon r\mu V^2/8L^3$, where $J$ is the current density, $\mu$ is the zero-field mobility of holes ($\mu_h$) or electrons ($\mu_e$), $\varepsilon_0$ is the permittivity of the vacuum, $\varepsilon_r$ is the relative permittivity of the material, $L$ is the thickness of the blend film, and $V$ is the effective voltage ($V = V_{appl} - V_{bi}$, where $V_{appl}$ is the applied voltage, and $V_{bi}$ is the built-in potential determined by electrode work function difference). Here, $V_{bi}$ = 0.1 V for hole-only devices, $V_{bi}$ = 0 V for electron-only devices. The mobility was calculated from the slope of $J^{1/2}$-$V$ plots.

**Grazing Incidence Wide-Angle X-ray Scattering (GIWAXS)**

GIWAXS measurements were conducted at beamline 7.3.3 of the Advanced Light Source. Samples were prepared on Si substrates utilizing blend solutions identical to those employed in the fabrication of devices. A 10 keV X-ray beam was incident at a grazing angle ranging from 0.12° to 0.16°, chosen to optimize the scattering intensity from the samples. The scattered X-rays were captured by a Dectris Pilatus 2M photon counting detector.

**Energy loss characterization**

EQEEL was obtained based on a home-built EQE measurement system where a Keithley 2400 SourceMeter was used for the current injection. The emission photon-flux from the solar cells was recorded by an Ocean Optics QE Pro spectrometer.

FTPSEQE spectra were obtained using a highly sensitive EQE measurement system (PECT-600, Enlitech Taiwan).

**Photo-induced Force Microscopy (PiFM)**

PiFM is a multimodal Atomic Force Microscopy (AFM) technique that integrates the high resolution of AFM with infrared spectroscopy to acquire topographical and molecular information with sub-10 nanometer resolution. All measurements were conducted using Molecular Vista's Visascope microscope and Block Engineering's Laser Tune QCL, with an adjustable range from 775 to 1885 $cm^{-1}$ and a spectral linewidth of 2 $cm^{-1}$. PiFM images were created with a resolution of 256x256 pixels, line speed of 0.89 lines/s, and a size of 1 $\mu m^2$, using wavenumbers 1650 $cm^{-1}$ and 1700$cm^{-1}$ to emphasize the copolymer donor and acceptor.

**Stability measurement**

Photostability of devices were tested under Maximum Power Point Tracking (MPP) with 1-sun illumination (white LED lamp). At the commencement of the test, the bias voltage is typically initially set to 0 V, with an increment step size of 0.01 V. As the test progresses, the settings for the bias voltage and increment can be automatically adjusted until the bias voltage approaches the maximum power point voltage *V*max.

**Estimation of glass-transition temperature (*T*g)**

The glass transition temperature (*T*g) was determined using ultraviolet-visible spectroscopy. Absorption spectra measurements were conducted on the receptor thin film, with the annealing temperature ranging from 20°C to 240°C. Then, the deviation metric (DMT) of the each absorption spectra was calculated, following the method from Samuel E. Root et al.[3] $DM_T = \sum^{\lambda max}{}_{\lambda min} [I_{RT} (\lambda) - I_T (\lambda)]^2$where λ is the wavelength, λmax and λmin are the upper and lower bounds of the optical sweep, respectively, IRT(λ) and IT(λ) are the normalized absorption intensities of the as-cast (room temperature) and annealed films, respectively. Then, *T*gs were determined to points where the two interpolated lines in low- and high-temperature regions intersect.

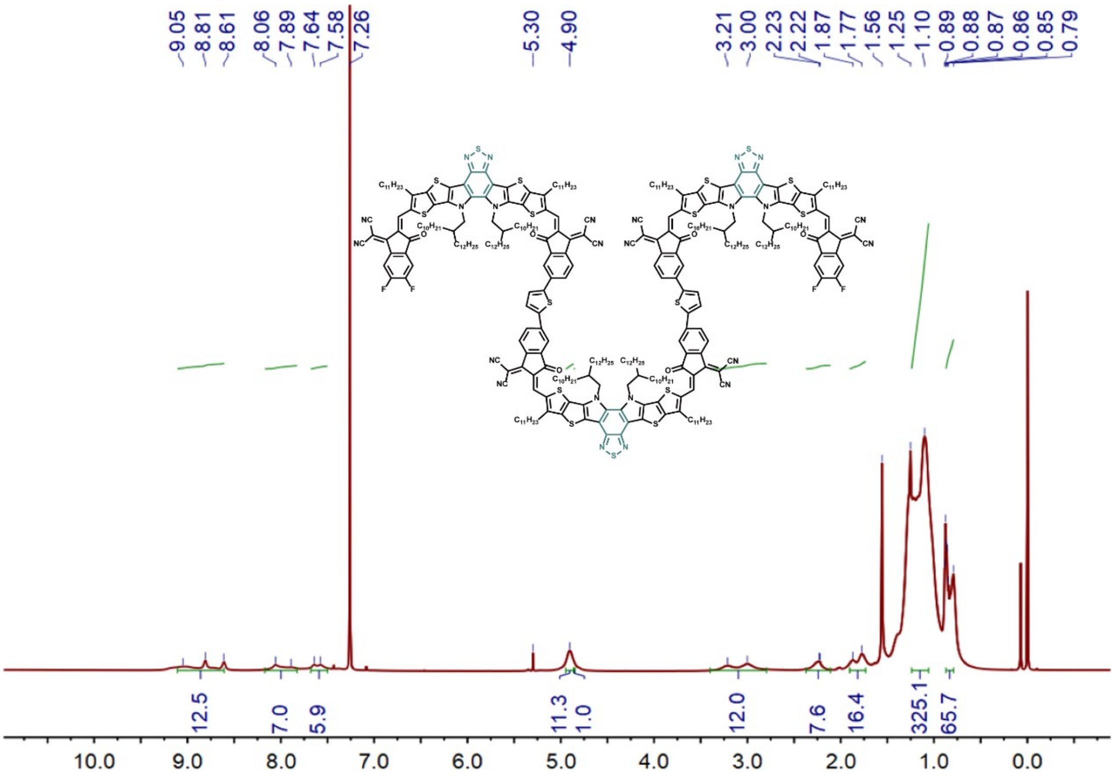

**Figure S4.** $^1$H-NMR spectrum of Tri-BT in $CDCl_3$.

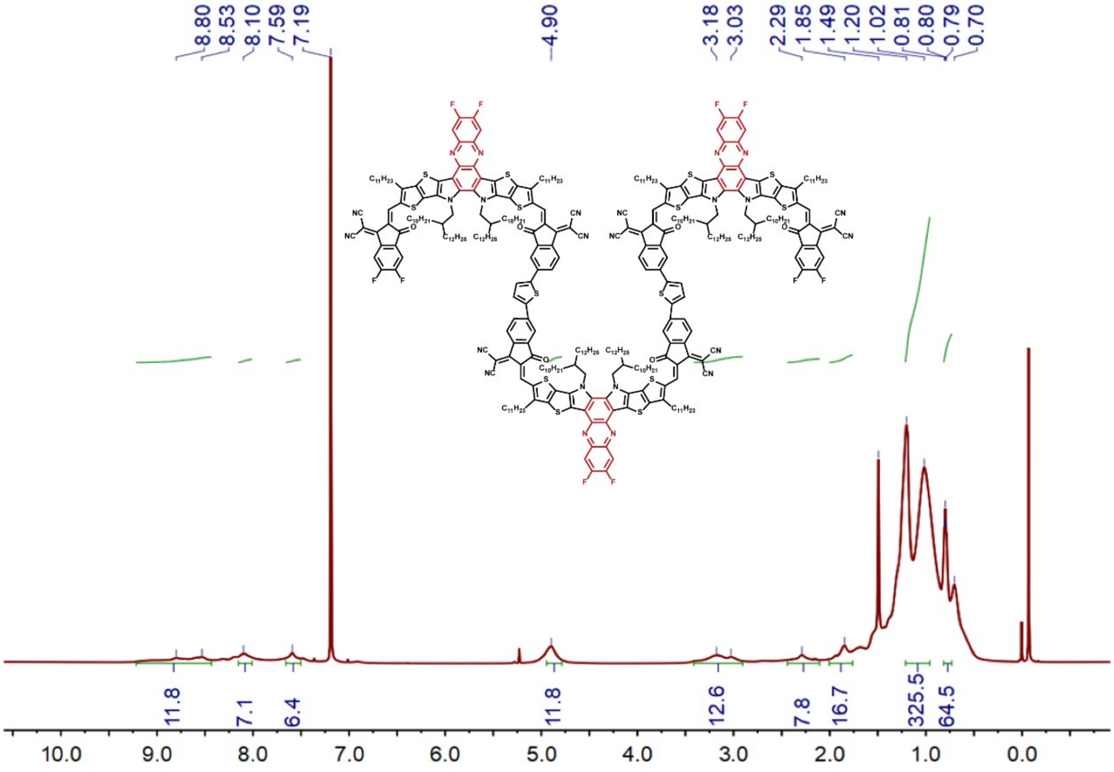

**Figure S5.** $^1$H-NMR spectrum of Tri-Qx in $CDCl_3$.

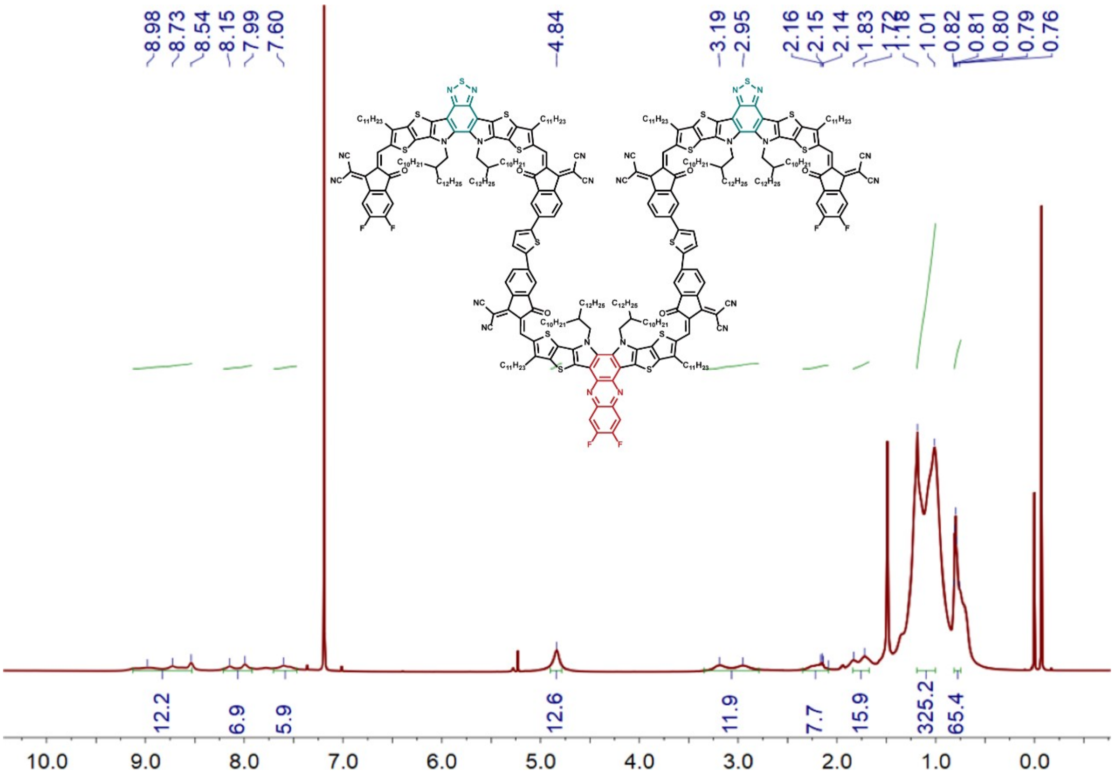

**Figure S6.** $^1$H-NMR spectrum of Tri-Qx in $CDCl_3$.

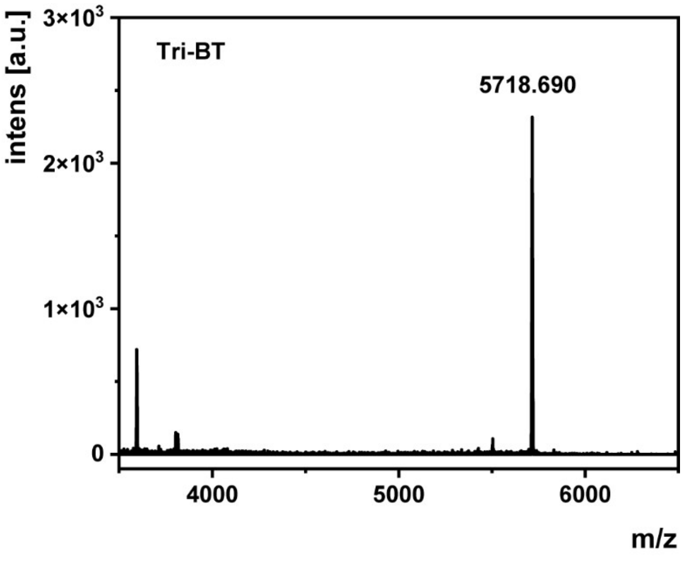

**Figure S7.** MS (MALDI-TOF) spectrum of Tri-BT.

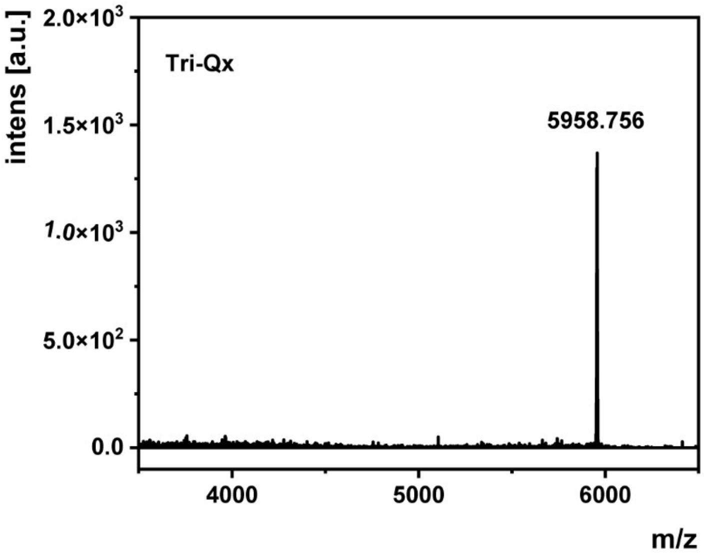

**Figure S8.** MS (MALDI-TOF) spectrum of Tri-Qx.

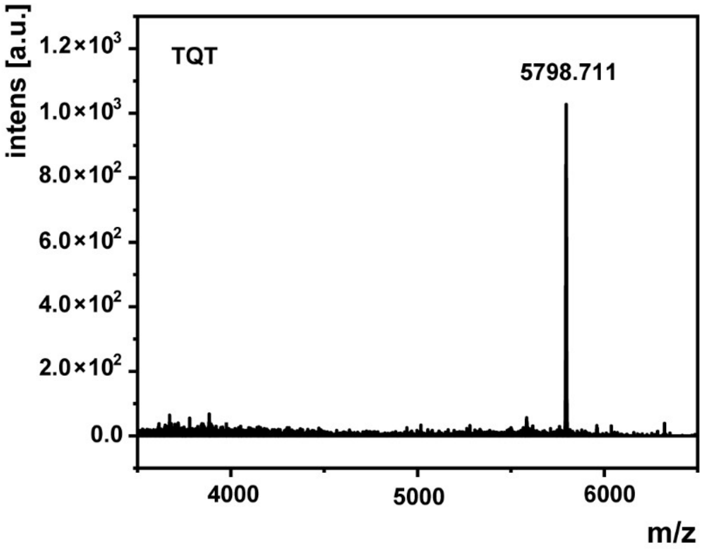

**Figure S9.** MS (MALDI-TOF) spectrum of TQT.

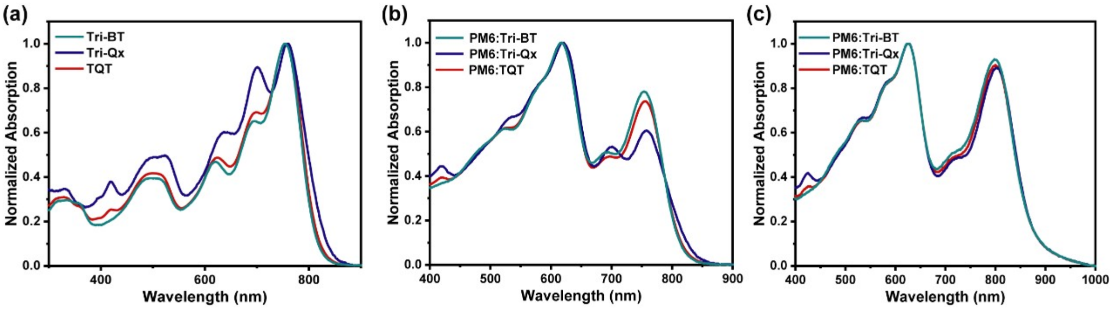

**Figure S10.** (a) Normalized absorption of Tri-BT, Tri-Qx and TQT neat films. Normalized absorption of Tri-BT, Tri-Qx and TQT in (a) diluted chloroform solution and (b) neat films.

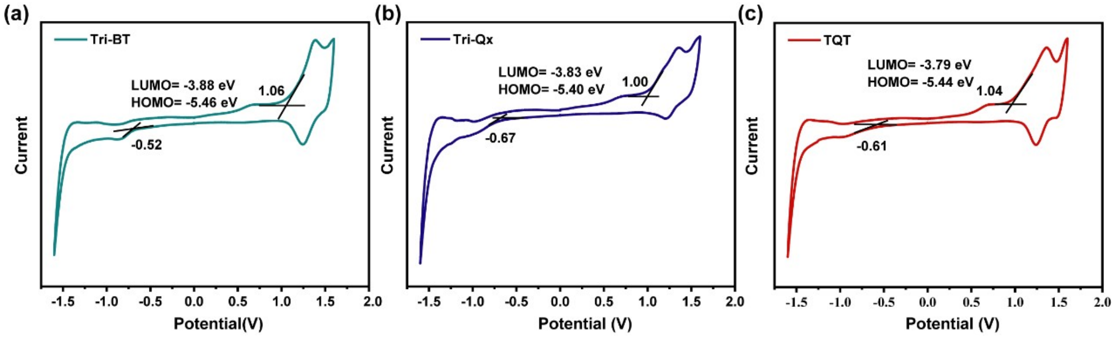

**Figure S11.** Cyclic voltammetry curves of Tri-BT, Tri-Qx and TQT.

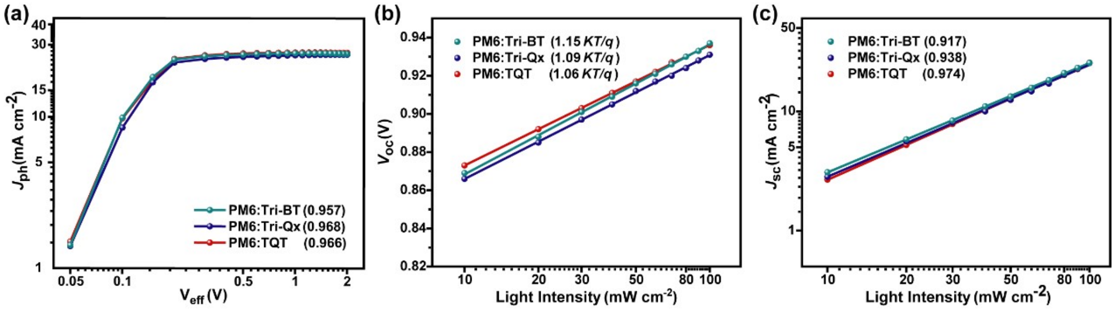

**Figure S12.** (a) $J_{\mathrm{ph}}$ versus $V_{\mathrm{eff}}$ characteristics. (b) $V_{\mathrm{oc}}$ versus light intensity curves. (c) $J_{\mathrm{sc}}$ versus light intensity curves.

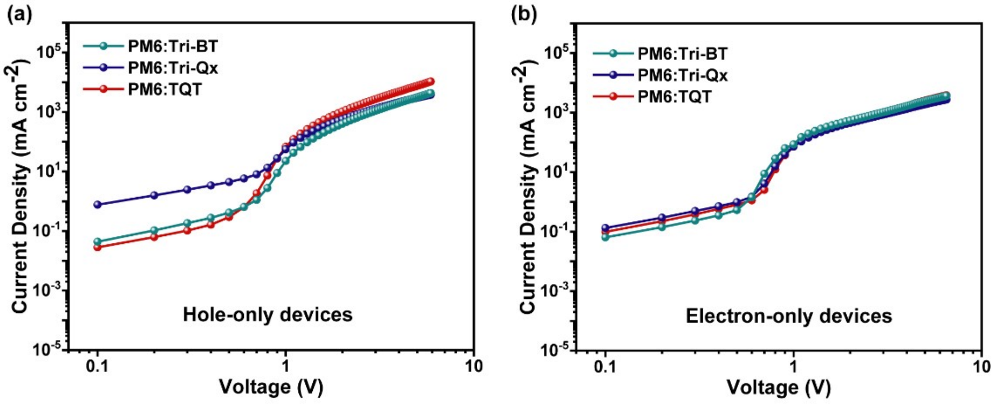

**Figure S13.** (a) SCLC curves of hole-only devices based on blend films. (b) SCLC curves of electron-only devices based on blend films.

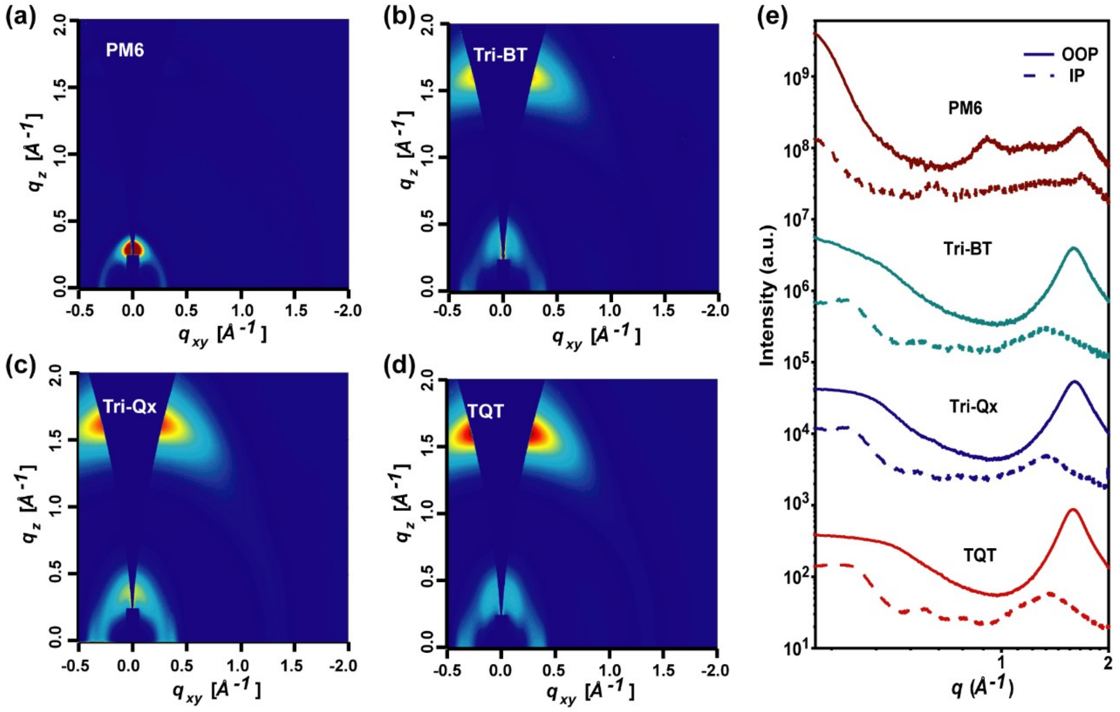

**Figure S14.** (a-d) 2D-GIWAXS patterns of PM6, Tri-BT, Tri-Qx and TQT neat film. (e) Corresponding in-plane and out-of-plane line cuts of PM6, Tri-BT, Tri-Qx and TQT neat films.

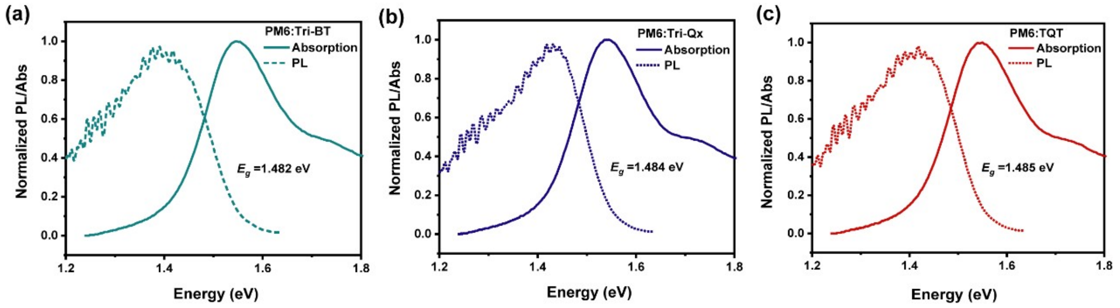

**Figure S15.** Normalized absorption and photoluminescence spectra of PM6:Tri-BT (a), PM6:Tri-Qx (b), and PM6:TQT (c) films.

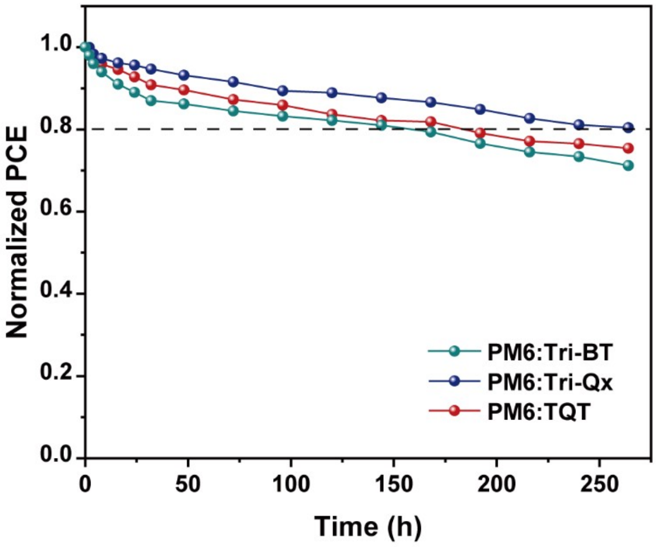

**Figure S16.** Thermal stability of PM6:Tri-BT, PM6:Tri-Qx, PM6:TQT devices..

**Table S1**. Linear-shaped trimer acceptor-based OSCs reported in the past several years

| All Polymer Blends | $V_{oc}$ (V) | $J_{sc}$ (mA cm$^{-2}$) | FF (%) | PCE (%) | Reference |
|---|---|---|---|---|---|
| PM6:tBTICγ-BO | 0.90 | 21.25 | 68.76 | 13.16 | 4 |
| PBDB-T:OY3 | 0.839 | 23.76 | 74.58 | 14.87 | 5 |
| PM6:TYT | 0.964 | 25.07 | 75.0 | 18.15 | 6 |
| PM6:Tri-Y6-OD | 0.916 | 25.30 | 77.8 | 18.03 | 7 |
| PM6:TY | 0.953 | 23.35 | 73.36 | 16.32 | 8 |
| **PM6:TQT** | **0.944** | **25.78** | **76.1** | **18.52** | **Our work** |

**Table S2**. The electron and hole mobility results measured by SCLC method.

| Devices | $\mu_e$ (cm$^2$ V$^{-1}$ s$^{-1}$) | $\mu_h$ (cm$^2$ V$^{-1}$ s$^{-1}$) | $\mu_h/\mu_e$ |
|---|---|---|---|
| Tri-BT | $6.29 \times 10^{-4}$ | $4.55 \times 10^{-4}$ | 1.38 |
| Tri-Qx | $4.22 \times 10^{-4}$ | $3.68 \times 10^{-4}$ | 1.15 |
| TQT | $5.96 \times 10^{-4}$ | $5.26 \times 10^{-4}$ | 1.13 |

**Table S3**. Crystal coherence length and the d-spacing for the neat and blend film.

| Samples | In plane |  |  |  | Out of plane |  |  |  |
|---|---|---|---|---|---|---|---|---|
|  | $q$ (Å$^{-1}$) | *d*-spacing (Å) | FWHM | CCL (Å) | $q$ (Å$^{-1}$) | *d*-spacing (Å) | FWHM | CCL (Å) |
| PM6 | 0.274 | 22.89 | 0.129 | 43.36 | 1.656 | 3.79 | 0.294 | 19.01 |
| Tri-BT | 0.323 | 19.41 | 0.186 | 30.02 | 1.586 | 3.96 | 0.258 | 21.68 |
| Tri-Qx | 0.314 | 19.98 | 0.203 | 27.55 | 1.574 | 3.99 | 0.268 | 20.83 |
| TQT | 0.331 | 18.95 | 0.184 | 30.38 | 1.598 | 3.93 | 0.251 | 22.28 |
| PM6:Tri-BT | 0.291 | 21.55 | 0.077 | 72.21 | 1.639 | 3.83 | 0.266 | 21.01 |
| PM6:Tri-Qx | 0.288 | 21.75 | 0.069 | 81.48 | 1.624 | 3.86 | 0.264 | 21.17 |
| PM6:TQT | 0.289 | 21.67 | 0.070 | 80.30 | 1.640 | 3.83 | 0.258 | 21.70 |

## References

[1] J. Yuan, Y. Zhang, L. Zhou, G. Zhang, H.-L. Yip, T.-K. Lau, X. Lu, C. Zhu, H. Peng, P. A. J. J. Johnson, *Joule* **2019**, 3, 1140.

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