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Electronic Supplementary Information for Chemical Communications

Solution-processed new porphyrin-based small-molecules as electron donors for highly efficient organic photovoltaics

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Contents

1. Experimental Section

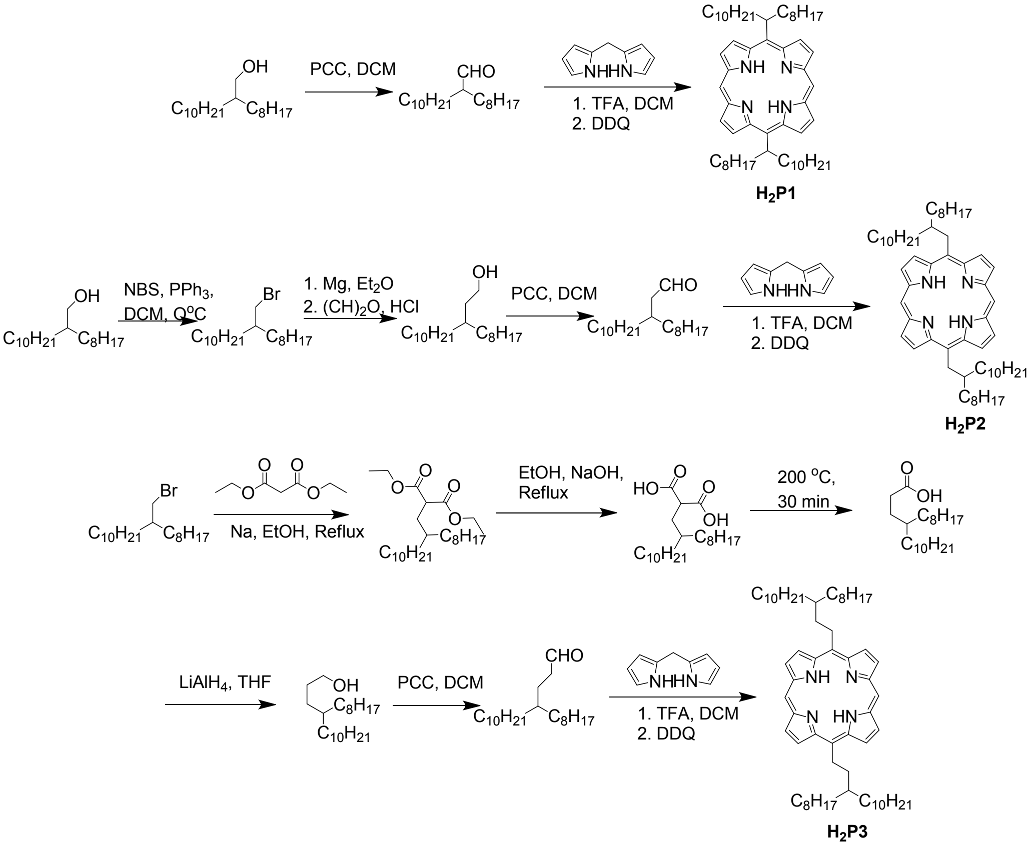
Characterizations: ^{1}H NMR spectra were recorded using a Bruker Ultrashield 400 Plus NMR spectrometer. UV-vis spectra of dilute solutions (1×10^{-5} M) of the samples in $CHCl_3$ were recorded at room temperature (ca. 25°C) using a Varian Cary 100 UV-vis spectrophotometer. Solid films for UV-vis spectroscopic analysis were obtained by spin-coating the molecule solutions onto a quartz substrate. Cyclic voltammetry (CV) of the molecule films was performed using a Versastat II electrochemical workstation operated at a scan rate of 50 mV s^{-1}; the solvent used was anhydrous CH_2Cl_2, containing 0.1 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$) as the supporting electrolyte. The potentials were measured against a Ag/Ag^+ (0.01 M $AgNO_3$) reference electrode; the ferrocene/ferrocenium ion (Fc/Fc^+) pair was used as the internal standard (0.09 V). The onset potentials were determined from the intersection of two tangents drawn at the rising and background currents of the cyclic voltammograms. HOMO and LUMO energy levels were estimated relative to the energy level of the ferrocene reference (4.8 eV below vacuum level). High-resolution matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) mass spectra were obtained with a Bruker Autoflex MALDI-TOF mass spectrometer. Topographic and phase images of the donor:$PC_{71}BM$ films (surface area: 5×5 μm^2) were obtained using a Digital Nanoscope III atomic force microscope operated in the tapping mode under ambient conditions. The thickness of the active layer of the device was measured using a Veeco Dektak 150 surface profiler. The *J-V* characteristics were measured under AM 1.5 solar simulator (Japan, SAN-EI, XES-40S1) at 100 mW cm^{-2}, and data was collected using a Keithley 2400 digital source meter. The spectral response was measured with a DSR100UV-B spectrometer with a SR830 lock-in amplifier. A calibrated Si photodiode was used as a reference before each measurement.

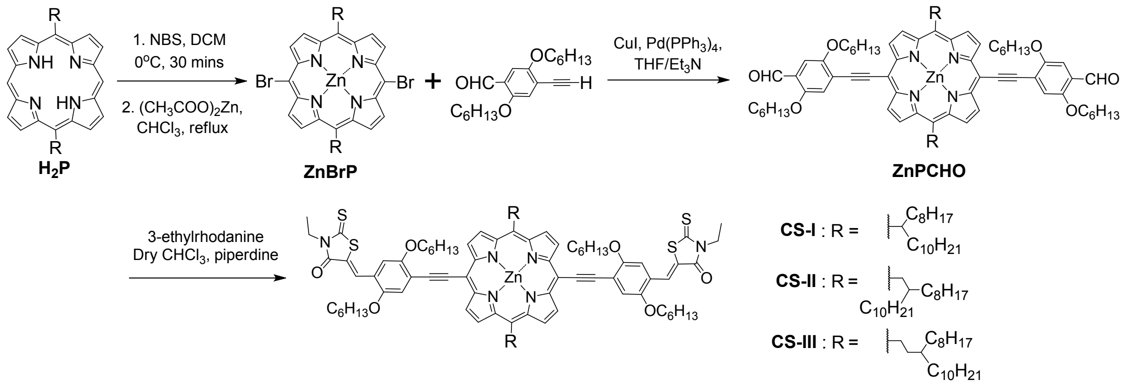
OSC device fabrication: Solution-processed bulk-heterojunction solar cells were fabricated as follows: Indium tin oxide (ITO) coated glass substrates were cleaned prior to device fabrication by sonication in acetone, detergent, distilled water, and isopropyl alcohol. After treated with an oxygen plasma for 5 min, 40 nm thick poly(styrene sulfonate)-doped poly(ethylene-dioxythiophene) (PEDOT:PSS) (Bayer Baytron 4083) layer was spin-coated on the ITO-coated glass substrates at 2500 rpm for 30s, the substrates were subsequently dried at 150℃ for 10 min in air and then transferred to a N_2-glovebox. The active layers were spun from solution of donor:$PC_{71}BM$ at the weight ratio of 1:1 with an overall concentration of 20 mg/mL and the volume of pyridine additive was 3%. The thicknesses of active layers were measured by a profilometer. The ultra-thin PFN layer was deposited by spin casting from a 0.02% (w/v) solution in methanol (from 2000 rpm for 30 s). Finally, Al (~80 nm) was evaporated with a shadow mask as the top electrode. The effective area was measured to be 0.16 cm^2.

Atomic force microscopy: The atomic force microscopy (AFM) measurements of the surface morphology of blend films were conducted on a NanoScope NS3A system (Digital Instrument). The *J-V* characteristics were measured under AM 1.5 solar simulator (Japan, SAN-EI, XES-40S1) at 100 mW cm^{-2}, and data was collected using a Keithley 2400 digital source meter. The spectral response was measured with a DSR100UV-B spectrometer with a SR830 lock-in amplifier. A calibrated Si photodiode was used as a reference before each measurement.

2. Synthesis procedures

All of the chemicals were purchased from Dieckmann Chemical Ltd, China. Bis(hexyloxy)benzene, 1,4-dibromo-2,5-bis(hexyloxy)benzene, 4-bromo-2,5-bis(hexyloxy)benzaldehyde, 3-octyl-1-tridecanol and 4-octyl-1-tetradecanol were prepared according to the literature procedures and characterized by comparing their ^{1}H NMR and ^{13}C NMR spectra with those found in the literature.[1-3] Organic solvents used in this work were purified using standard process. Other chemicals and reagents were used as received from commercial sources without further purification, unless stated otherwise. [6,6]-Phenyl-C_{71}-butyric acid methyl ester ($PC_{71}BM$) was purchased from Nano-C.





Scheme S1. Synthetic routes to **CS-I**, **CS-II** and **CS-III**.

4-Ethynyl-2,5-bis(hexyloxy)benzaldehyde: 4-Bromo-2,5-bis(hexyloxy)benzaldehyde (7.706 g, 20.00 mmol), CuI (0.052 g, 0.28 mmol), and $Pd(PPh_3)_2Cl_2$ (0.475 g, 0.68 mmol) were combined in a mixture of toluene (200 mL) and diisopropylamine (60 mL) to yield a yellow solution under a nitrogen atmosphere. A solution of trimethysilyl acetylene (4.32 g, 6.22 mL, 44.0 mmol) in diisopropylamine (30 mL) was slowly added to the reaction mixture at room temperature. The reaction mixture was then stirred at 70 °C for 6 h to give a brown suspension. The completion of the reaction was verified by spot TLC. The solvent was then removed under reduced pressure, and the residue was chromatographed on silica gel using hexane as eluent to give 2,5-bis(hexyloxy)-4-(2-(trimethylsilyl)ethynyl)benaldehyde (7.36 g, 92%) as a light yellow solid. A KOH aqueous solution (15.0 mL, 20.0%) was diluted with methanol (50 mL) and added to a stirred solution of trimethylsilyl protected aldehyde (7.01 g, 12.0 mmol) in THF (100 mL) under a nitrogen atmosphere. The mixture was stirred at room temperature for 1 h and extracted with dichloromethane for several times. The organic fraction was washed with water and dried over anhydrous $MgSO_4$. The crude product (5.28 g, 80%) was isolated as a light yellow solid. ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.76-0.77 (t, *J* = 7.6Hz, 6H, CH_3), 1.20-1.30 (m, 8H), 1.3-1.5 (m, 4H), 1.65-1.80 (m, 4H), 3.08 (s, 1H, C≡CH), 4.01 (t, *J* = 6.4Hz, 2H), 4.02 (t, *J* = 6.4Hz, 2H), 7.22 (s, 1H), 7.30 (s, 1H), 10.41 (s, 1H).

General procedure for the synthesis of aldehyde derivatives with alkyl side-chain: To the mixture of side-chain alkyl alcohol components (15 mmol) and dichloromethane (35 mL), Pyridinium chlorochromate (PCC) (4.52 g, 21 mmol) was added at room temperature. After stirring the mixture for another 4 hours, it was filtered and the filtrate was distilled under reduced pressure to give the crude products, which was flash chromatographed on short silica column with CH_2Cl_2 as eluent to afford the title compound as a colorless oil.

2-octyl-dodecanal: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.82 (m, 6H), 1.16−1.25 (m, 28H), 1.37−1.42 (m, 2H), 1.50−1.55 (m, 2H), 2.25−2.31 (m, 1H), 9.70 (s, 1H).

3-octyl-tridecanal: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): δ 0.88 (t, *J* = 6.8 Hz, 6H), 1.26 (m, 32H), 1. 94 (m, 1H), 2.40 (m, 2H), 9.83 (s, 1H).

4-octyl-tetradecanal: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.87 (m, 6H), 1.25 (m, 29H), 1.54−1.60 (m, 2H), 2.41 (m, 2H), 9.77 (s, 1H).

General procedure for the synthesis of 5,15-dialkylated porphyrin (H_2P): A solution of dipyrromethane (3 g, 21 mmol) and aldehyde derivatives (21 mmol) in CH_2Cl_2 (1.5 L) was degassed by bubbling with nitrogen for 30 min. Then trifluoroacetic acid (TFA) (145 μL, 2.0 mmol) was added. After the solution was stirred overnight at room temperature under nitrogen, 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) (7.28 g, 32 mmol) was added, and the reaction mixture was stirred for a further 2h. Triethylamine (5 mL) was added. The solvent was evaporated and the crude product was purified by silica chromatography using CH_2Cl_2/n-hexane (1:4) as the eluent, to give a purple product.

H_2P-I: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): −2.44 (s, 2H), 0.73 (m, 12H), 1.03 (m, 38H), 1.18−1.40 (m, 12H), 1.53 (m, 6H), 2.76 (m, 4H), 2.98 (m, 4H), 5.20 (m, 2H), 9.43 (m, 4H), 9.66 (m, 2H), 9.74 (m, 2H), 10.22 (m, 2H).

H_2P-II: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): −2.26 (s, 2H), 0.82 (m, 6H), 0.90 (m, 6H), 1.00−1.26 (m, 56H), 1.47 (m, 8H), 2.14 (m, 2H), 4.14 (m, 4H), 8.82 (d, *J* = 4.4 Hz, 4H), 9.24 (d, *J* = 4.8 Hz, 4H), 10.28 (s, 2H).

H_2P-III: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): −2.32 (s, 2H), 0.92 (m, 12H), 1.08−1.50 (m, 66H), 1.78 (m, 4H), 3.18 (m, 4H), 8.05 (d, *J* = 4.4 Hz, 4H), 8.64 (d, *J* = 4.8 Hz, 4H), 10.18 (s, 2H).

General procedure for the synthesis brominated 5,15-dialkylated metalloporphyrins (ZnBrP): Solution of H_2P (1.00 mmol), NBS (373 mg, 2.10 mmol) and pyridine (0.1 mL) in CH_2Cl_2 (200 mL) was stirred at 0°C for 30 min. Acetone (5 mL) was added, and the solvent was removed in vacuo. The residue was purified via chromatography on silica gel using CH_2Cl_2/n-hexane (1:4) as the eluent. Then, **BrH_2P** (1.00 mmol) and zinc acetate (1.095 g, 5 mmol) were refluxed in $CHCl_3$ (200 mL) for 4 h. The solvent was removed and the residue was purified via chromatography on silica gel using CH_2Cl_2/n-hexane (1:4) as the eluent to give the product in quantitative yield.

ZnBrP-I: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.73 (m, 12H), 0.98−1.08 (m, 38H), 1.20−1.32 (m, 12H), 1.52 (m, 6H), 2.72 (m, 4H), 2.89 (m, 4H), 5.16 (m, 2H), 9.73 (m, 8H).

ZnBrP-II: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.77 (m, 6H), 0.83 (m, 6H), 0.96−1.22 (m, 56H), 1.41 (m, 8H), 2.08 (m, 2H), 4.08 (m, 4H), 8.76 (d, *J* = 4.4 Hz, 4H), 9.17 (d, *J* = 4.8 Hz, 4H).

ZnBrP-III: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.99 (m, 12H), 1.13−1.64 (m, 66H), 1.84 (m, 4H), 3.23 (m, 4H), 8.11 (d, *J* = 4.4 Hz, 4H), 8.75 (d, *J* = 4.8 Hz, 4H).

General procedure for the synthesis of precursors ZnPCHO: A mixture of **ZnBrP** (0.150 mmol) and 4-ethynyl-2,5-bis(hexyloxy)benzaldehyde (115.7 mg, 0.350 mmol) in THF (15 mL) and Et_3N (5 ml) was degassed with nitrogen for 10 min, and then $Pd(PPh_3)_4$ (25 mg, 0.022 mmol) and CuI (5 mg, 0.022 mmol) were added to the mixture, and the solution was refluxed for 12 h under nitrogen. The solvent was removed under vacuum, and the solid residue was purified by preparative thin layer chromatography using a $CHCl_3$/hexane (3:1) mixture as eluents. Recrystallization from $CHCl_3$/methanol gave **ZnPCHO** as a green solid.

ZnPCHO-I: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.68−0.76 (m, 12H, CH_3), 0.89 (t, *J* = 7.2 Hz, 6 H), 0.98 (t, *J* = 7.2 Hz, 6H), 1.05−1.45 (m, 64H, CH_2), 1.59 (m, 12H, CH_2), 1.78−1.86 (m, 4H, CH_2), 1.92−1.99 (m, 4H, CH_2), 2.34 (m, 4H, CH_2), 2.76 (m, 4H, CH_2), 2.95 (m, 4H, CH_2), 4.25 (m, 4H, OCH_2), 4.35 (m, 4H, OCH_2), 5.18 (m, 2H, CH), 7.44−7.51 (m, 4H, ArH), 9.64 (m, 2H, *β*-pyrrolic H), 9.73 (m, 2H, *β*-pyrrolic H), 9.86−9.94 (m, 4H, *β*-pyrrolic H), 10.50 (s, 2H, CHO).

ZnPCHO-II: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.72 (t, *J* = 7.2 Hz, 6H), 0.78 (t, *J* = 7.2 Hz, 6H), 0.92−1.18 (m, 68H), 1.32−1.58 (m, 22H, CH_2), 1.68 (m, 8H, CH_2), 1.86 (m, 4H, CH_2), 2.04 (m, 4H, CH_2), 2.37 (m, 4H, CH_2), 4.17 (d, *J* = 6.8 Hz, 4H), 4.27 (t, *J* = 6.4 Hz, 4H, OCH_2), 4.36 (t, *J* = 6.4 Hz, 4H, OCH_2), 7.34 (s, 2H, ArH), 7.49 (s, 2H, ArH), 8.71 (d, *J* = 4 Hz, 2H, *β*-pyrrolic H), 9.22 (d, *J* = 4.4 Hz, 4H, *β*-pyrrolic H), 10.53 (s, 2H, CHO).

ZnPCHO-III: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.87−1.01(m, 24H), 1.21−1.56(m, 84H), 1.83 (m, 6H, CH_2), 1.96 (m, 4H, CH_2), 2.20−2.34 (m, 8H, CH_2), 4.23 (t, *J* = 5.6 Hz, 4H, OCH_2), 4.32 (t, *J* = 5.6 Hz, 4H, OCH_2), 4.44 (s, 4H, CH_2), 7.40 (s, 2H, ArH), 7.47 (s, 2H, ArH), 8.98 (d, J = 4 Hz, 4H, *β*-pyrrolic H), 9.51 (d, J = 4.4 Hz, 4H, *β*-pyrrolic H), 10.50 (s, 2H, CHO).

General procedure for the synthesis CS-X (X = I, II, III): Compound **ZnPCHO** (0.066 mmol) was dissolved in a solution of dry $CHCl_3$, two drops of piperdine and then 3-ethylrhodanine (106 mg, 0.66 mmol) were added, and the resulting solution was refluxed and stirred for 12 h under argon. The reaction was quenched into water (30 mL). The aqueous layers were extracted with $CHCl_3$ (3 × 20 mL). The organic layer was dried over $NaSO_4$. After removal of the solvent, it was purified by chromatography on a silica gel column using $CHCl_3$ as eluent and was purified by preparative thin layer chromatography using a $CHCl_3$ as eluents. Then the crude solid was recrystallized from a mixture of $CHCl_3$ and methanol to afford the final product as a gray green solid.

CS-I: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.72−0.79 (m, 12H, CH_3), 0.90−0.94 (m, 6H, CH2), 0.96−1.38 (m, 62H, CH_2), 1.44−1.50 (m, 16H), 1.60−1.66 (m, 8H), 1.81−1.91 (m, 10H), 1.92−2.01 (m, 4H), 2.31−2.40 (m, 4H), 2.63−2.76 (m, 4H), 2.87−2.99 (m, 4H), 4.21−4.26 (m, 8H), 4.31−4.38 (m, 4H), 5.09−5.20 (m, 2H), 7.08 (s, 2H), 7.45 (s, 2H), 8.28 (s, 2H), 9.54 (d, *J* = 5.2 Hz, 2H), 9.63 (d, *J* = 5.2 Hz, 2H), 9.81 (m, 4H). (MALDI-TOF, m/z) calculated for $C_{110}H_{154}N_{6}0_{6}S_{4}Zn$: 1849.0115; found: 1849.0006.

CS-II: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.82 (m, 12H), 0.96 (m, 20H), 1.11−1.28 (m, 48H)1.33 (m, 6H), 1.49 (m, 20H), 1.63 (m, 8H), 1.81 (m, 4H), 1.96 (m, 4H), 2.28 (m, 4H), 2.38 (m, 4H), 4.17−4.24 (m, 12H), 4.68 (d, *J* = 3.2 Hz, 2H), 6.87 (s, 2H), 7.23 (s, 2H), 7.31 (s, 2H), 8.19 (s, 2H) 9.22 (d, *J* = 4.4 Hz, 4H), 9.66 (d, *J* = 4.4 Hz, 4H). (MALDI−TOF, m/z) calculated for $C_{112}H_{158}N_{6}0_{6}S_{4}Zn$: 1877.0428; found: 1877.0422.

CS-III: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 0.86 (m, 18H), 0.92 (m, 6H), 1.24−1.42 (m, 72H), 1.56−1.65 (m, 16H), 1.79 (m, 4H), 1.95 (m, 4H), 2.23 (m, 4H), 2.37 (m, 4H), 4.19 (m, 12H), 4.71 (m, 2H), 6.84 (s, 2H), 7.19 (s, 2H), 7.28 (s, 2H), 8.15 (s, 2H) 9.22 (d, *J* = 4.4 Hz, 4H), 9.65 (d, *J* = 4.4 Hz, 4H). (MALDI−TOF, m/z) calculated for $C_{114}H_{162}N_{6}0_{6}S_{4}Zn$: 1905.0743; found: 1905.0756.

3. ^{1}H NMR spectra and MALDI-TOF spectra

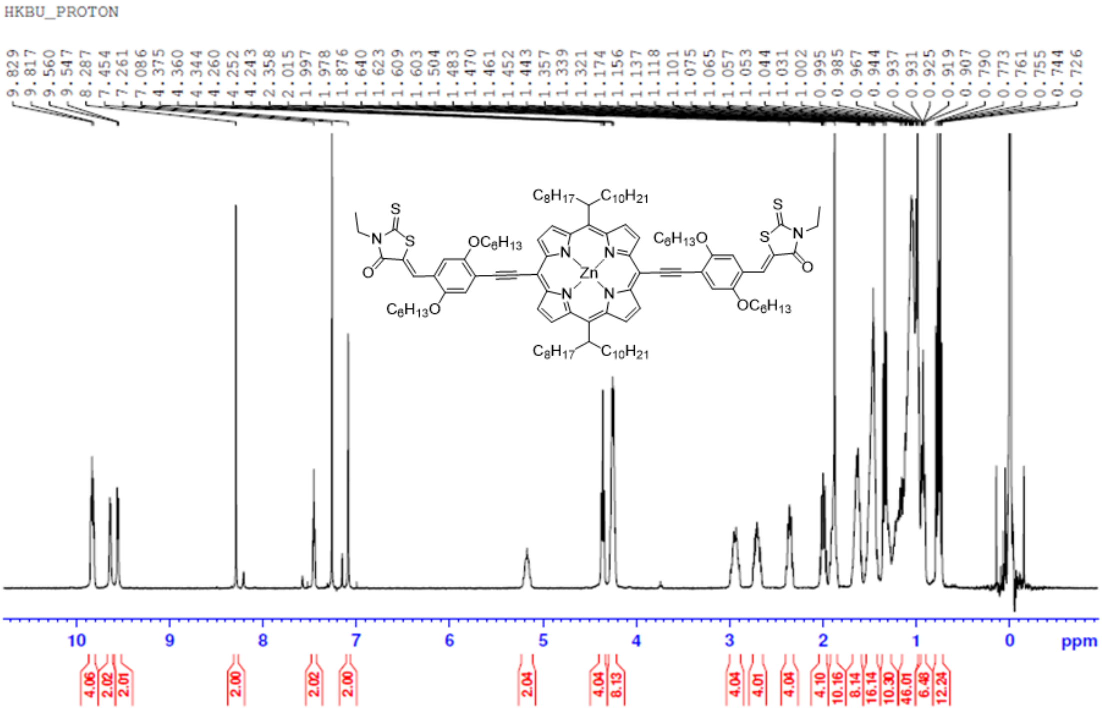


Figure S1. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **CS-I**.

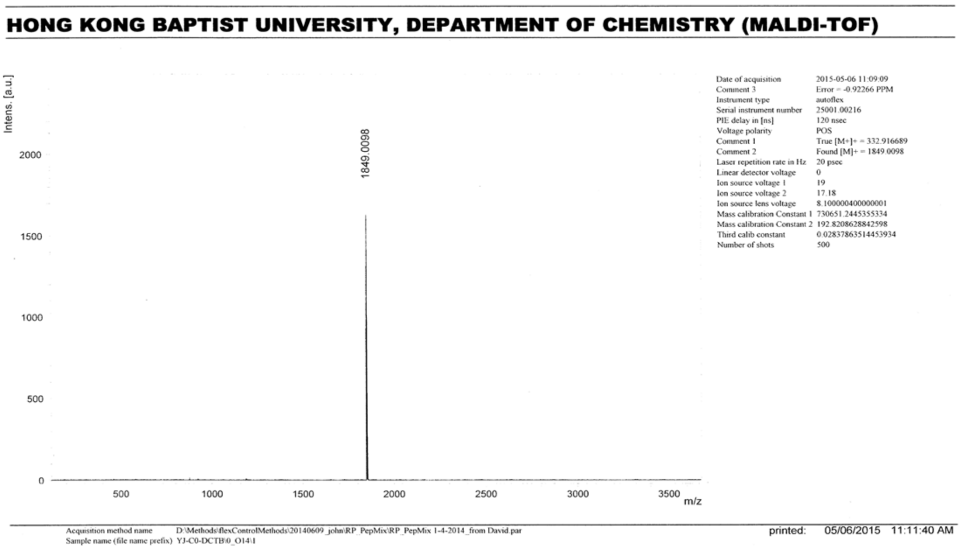


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Figure S2. The whole MALDI-TOF plot of compound **CS-I**.

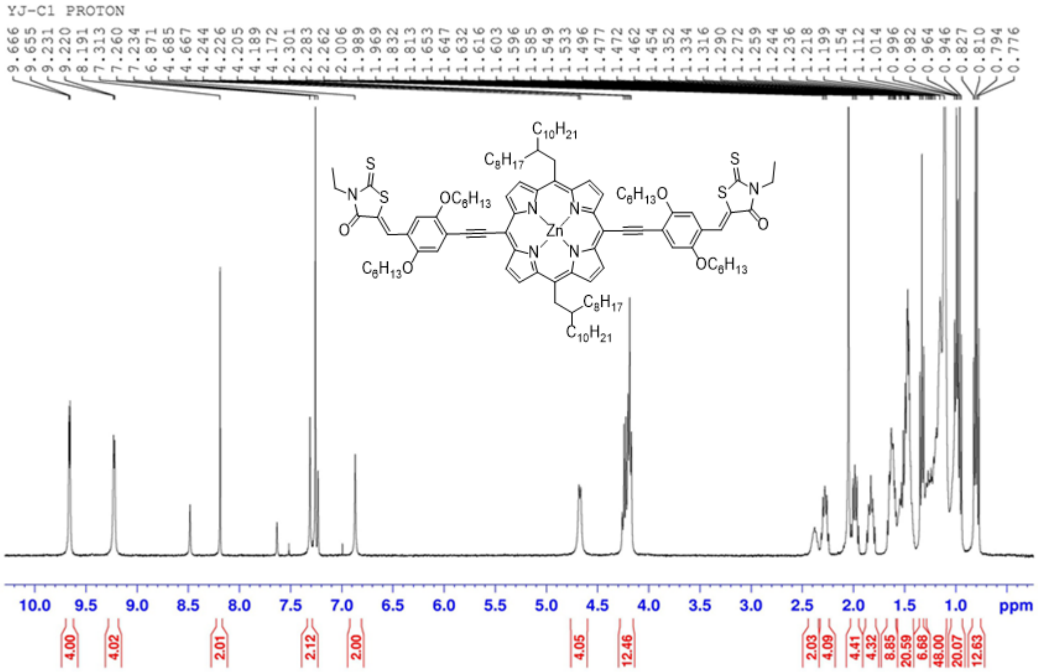

Figure S3. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **CS-II**.

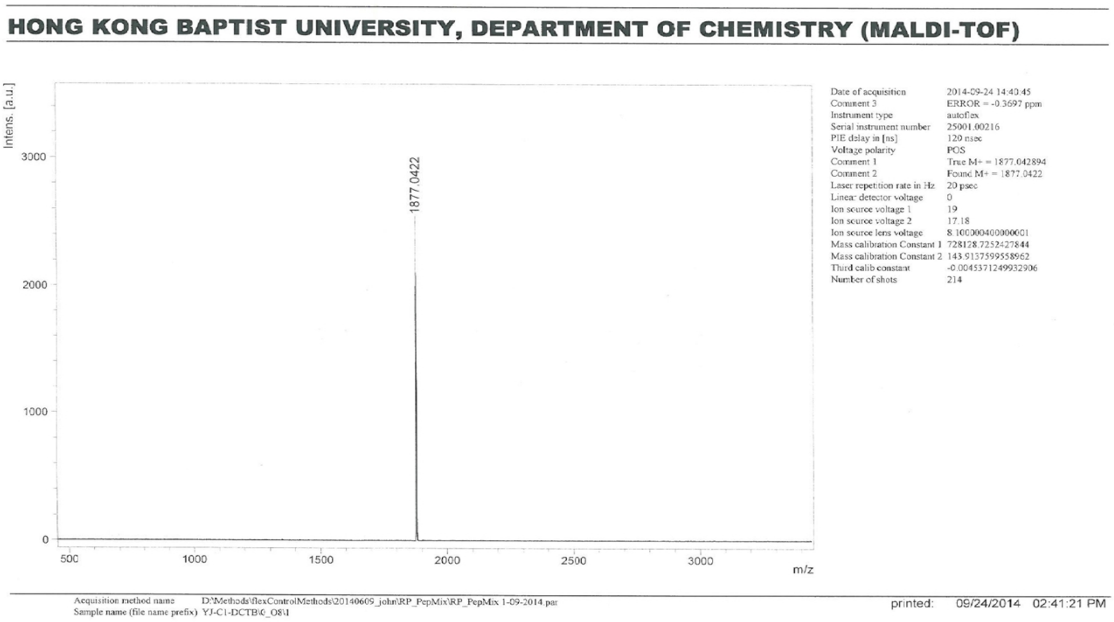

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Figure S4. The whole MALDI-TOF plot of compound **CS-II**.

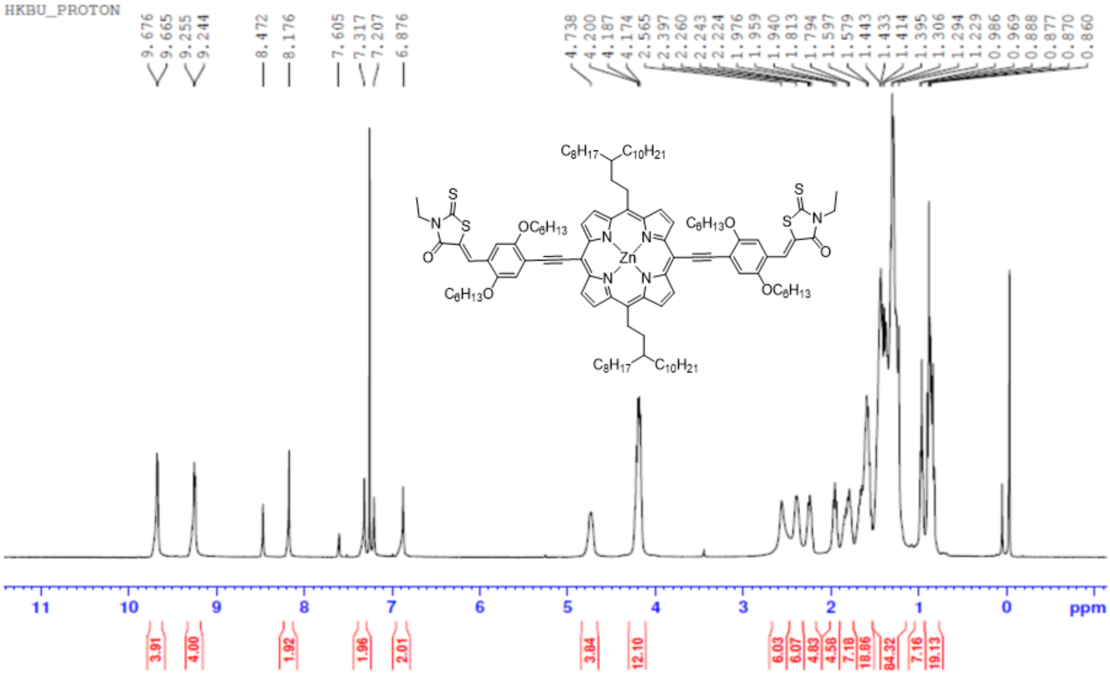
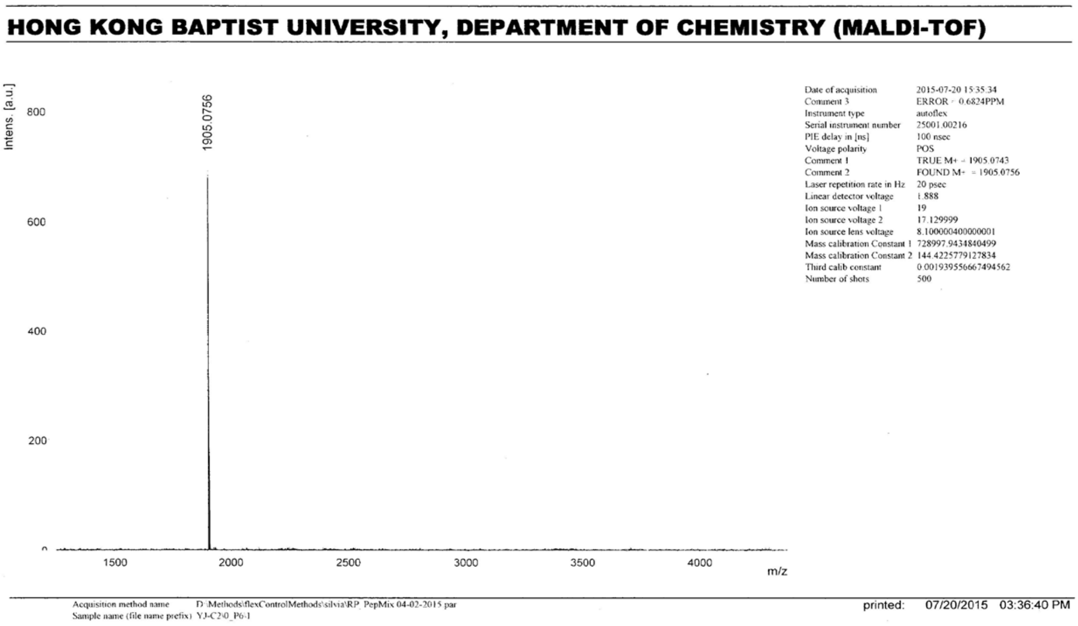


Figure S5. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **CS-III**.



HONG KONG BAPTIST UNIVERSITY, DEPARTMENT OF CHEMISTRY (MALDI-TOF)

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Figure S6. The whole MALDI-TOF plot of compound **CS-III**.

4. Cyclic voltammogram of the objective compounds

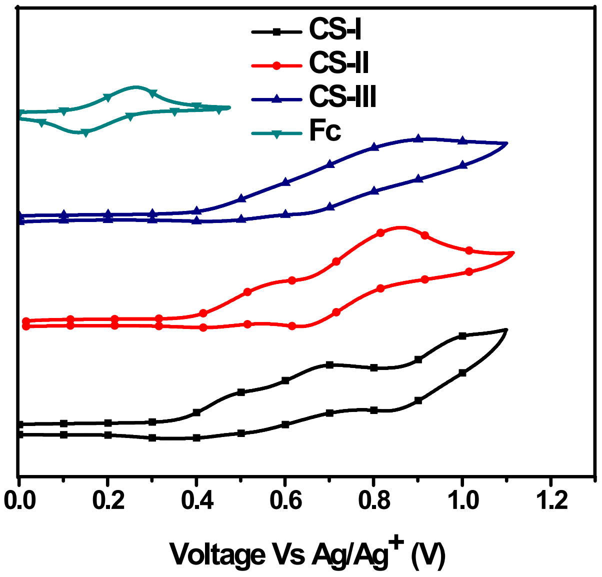

Figure S7. Cyclic voltammogram of **CS-I CS-II** and **CS-III** films on a glassy carbon disk measured in 0.1 mol L^{-1} Bu_4NPF_6 CH_2Cl_2 solutions with scan rate of 50 mV s^{-1}.

5. OPV characteristics of CS-I without additive

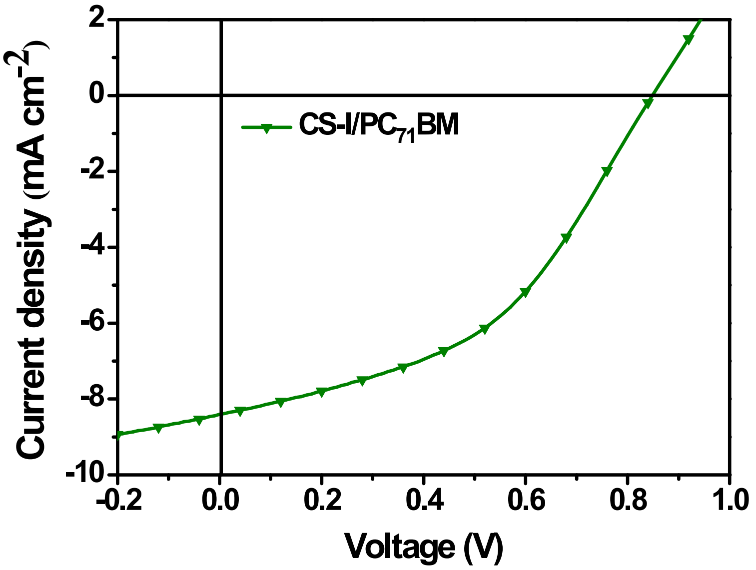

Figure S8. J-V curves of the OSCs based on **CS-I**/PC$_{71}$BM without additive under the illumination of AM 1.5G at 100 mW cm^{-2}.

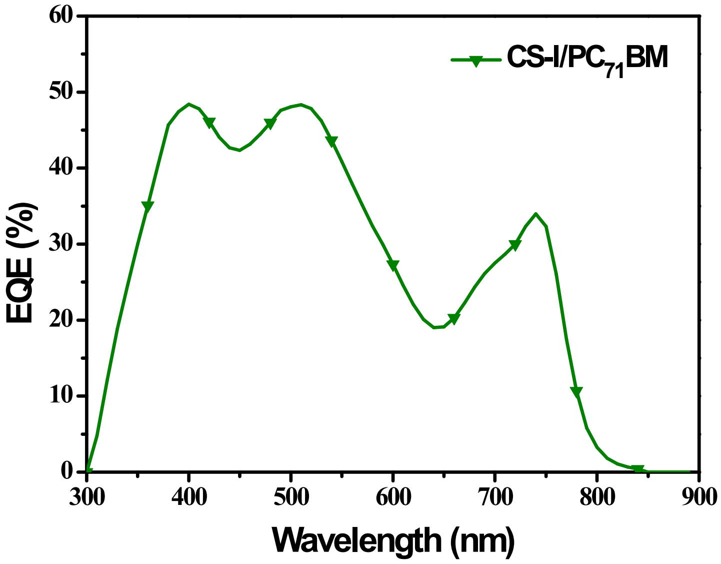

Figure S9. EQE curve of the devices based on **CS-I**/$PC_{71}BM$ (1 : 1) without additive.

6. Charge-carrier mobility measurement results

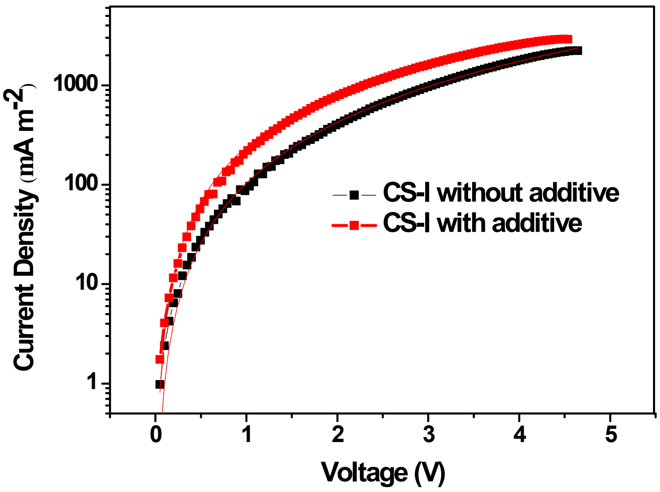

Figure S10. The space charge limited current (SCLC) curves of **CS-I**: mobility of **CS-1** without additive and mobility of **CS-I** with additive

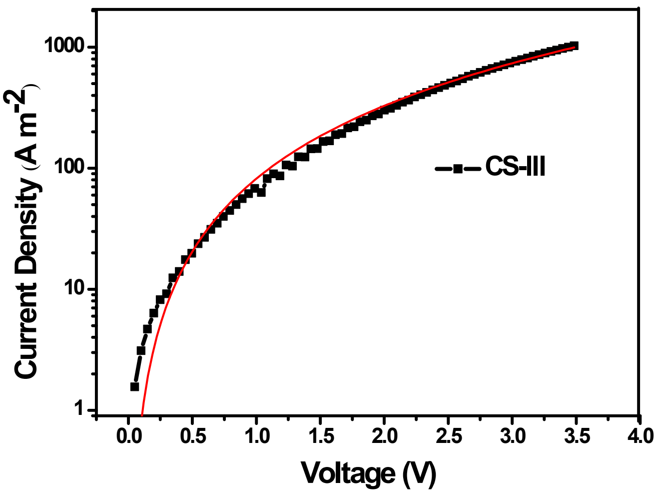

Figure S11. The space charge limited current (SCLC) curves of **CS-III** with additives

7. AFM images of the CS-II/PC$_{71}$BM and CS-III/PC$_{71}$BM composite films

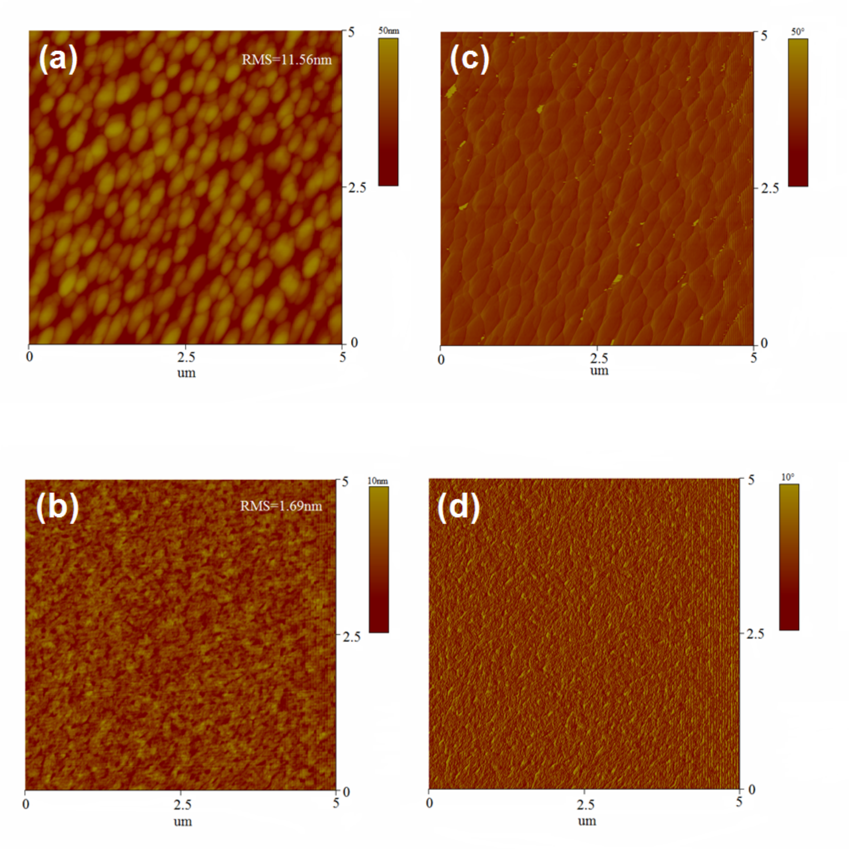

Figure S12. AFM height images (a) and (b) and AFM images (c) and (d) (5 × 5 µm, tapping-mode) of **CS-II**/PC$_{71}$BM (1:1) and **CS-III**/PC$_{71}$BM (1:1) composite films prepared with additive, respectively.

8. References

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