Electronic Supplementary Information for

Engineered co-sensitization system for highly efficient dye solar cells

by


**General remarks:** all reactants were purchased from standard commercial sources and used without any further purification. All solvents used were carefully dried and freshly distilled according to standard laboratory practice. All manipulations were carried out under inert nitrogen atmosphere. Flash chromatography was performed using a silica gel of 230-400 mesh. $^1$H and $^{13}$C{$_{^1}$H} NMR spectra were recorded on a Bruker Avance 700 MHz instrument. Melting points were measured on a Büchi B-545 instrument. Elemental analyses were obtained on a EuroVector CHNS EA3000 elemental analyser on the basis of three replicates. The high resolution electrospray ionization mass spectrometry (HR ESI-MS) analyses were performed using a Bruker microTOF QII mass spectrometer equipped with an electrospray ion source operated in positive ion mode. The sample solutions (CH$_2$Cl$_2$/MeOH) were introduced by continuous infusion at a flow rate of 180 $\mu$L/min with the aid of a syringe pump. The instrument was operated with end-plate offset and capillary voltages set to $-500$ V (500 V) and $-4500$ V (3500 V), respectively. The nebulizer pressure was 0.4 bar (N$_2$), and the drying gas (N$_2$) flow rate was 4.0 L/min. The capillary exit and skimmer 1 voltages were 90 V and 30 V, respectively. The drying gas temperature was set at 180°C. The calibration was carried out with sodium formate. FT-IR measurements were carried out on a JASCO FT/IR 4200 spectrophotometer. UV-Vis spectra were recorded on a Jasco V-670 instrument. Cyclic voltammetry was carried out on a Metrohm Autolab PGSTAT 302-N potentiostat. The materials were drop cast on a platinum working electrode from a 1 mg/mL THF (tetrahydrofuran) solution. Measurements were carried out at 25 °C in acetonitrile solution containing tetrabutylammonium tetrafluoroborate (0.025 M) as supporting electrolyte.
with a scan rate of 50 mV/s. The potentials were measured versus Ag/Ag⁺ as the quasi-reference electrode. After each experiment, the potential of the Ag/Ag⁺ electrode was calibrated against the ferrocene/ferrocenium (Fc/Fc⁺) redox couple. The electrochemical energy gap was determined as the difference between the onsets of the oxidation and the reduction potentials ($E_{\text{g,elc}} = E_{\text{onset,ox}} - E_{\text{onset,red}}$). The HOMO and LUMO energy values were estimated from the onset potentials of the first oxidation and reduction event, respectively. After calibration of the measurements against Fc/Fc⁺, the HOMO and LUMO energy levels were calculated according to the following equations:

$$E_{\text{HOMO}} (\text{eV}) = -\left[ E_{\text{onset,ox}} - E_{1/2}(\text{Fc/Fc}^+) + 4.8 \right]$$

$$E_{\text{LUMO}} (\text{eV}) = -\left[ E_{\text{onset,red}} - E_{1/2}(\text{Fc/Fc}^+) + 4.8 \right]$$

where $E_{1/2}(\text{Fc/Fc}^+)$ is the half-wave potential of the Fc/Fc⁺ couple (the oxidation potential of which is assumed at 4.8 eV) against the Ag/Ag⁺ electrode. The HOMO and LUMO levels evaluated for DTB-B and DTB-T were very similar (–5.4 eV and –3.4 eV, respectively) for both dyes. Density functional theory (DFT) and time-dependent DFT (TD-DFT) calculations were carried out with the Gaussian09 program package.
Synthesis of DTB-B and DTB-T

The synthetic sequence followed for the obtainment of DTB-B and DTB-T is illustrated in Scheme 1S. Reaction of 4,7-bis-thiophen-2-yl-benzo[2,1,3]thiadiazole with an equimolar amount of N-bromo-succinimide (NBS) allowed the obtainment of the corresponding mono-bromo derivative 2. A Pd-catalyzed Sonogashira cross-coupling between 2 and trimethylsilyl-acetylene resulted in the functionalization with the ethynylene moiety, yielding the alkyne 3. The trimethylsilyl protecting group in 3 could be easily removed by reaction with potassium fluoride affording the terminal alkyne 4. At this stage of the synthetic procedure, the assembly of the \( \pi \)-bridges was completed by a further Sonogashira cross-coupling of 4 with either 4-bromobenzaldehyde or 5-bromo-thiophene-2-carbaldehyde to obtain aldehydes 5 and 6, respectively. The synthesis was completed by introduction of the cyano-acrylic functionality by submitting 5 and 6 to a Knoevenagel condensation with cyanoacetic acid in the presence of ammonium acetate eventually yielding the target molecules DTB-B and DTB-T, respectively. The synthetic procedure is depicted in Scheme 1S and the characterization of the intermediates is described below.

\[ \text{Scheme 1S. Synthetic approach for the obtainment of DTB-B and DTB-T.} \]
4-(5-Bromo-thiophen-2-yl)-7-(thiophen-2-yl)-benzo[2,1,3]thiadiazole (2). A solution of N-bromo-succinimide (0.89 g, 5.00 mmol) in DMF (30 mL) was added dropwise to a solution of 1 (1.51 g, 5.00 mmol) in DMF (45 mL) kept at 0 °C. After the addition, the obtained reaction mixture was warmed to room temperature and allowed to react for further 2 h before quenching with water (50 mL). The products was extracted with diethyl ether (3 × 50 mL) and the combined organic phases were washed with brine and dried over Na$_2$SO$_4$. After solvent removal, the crude product was purified by column chromatography (SiO$_2$, petroleum ether 40-60 °C/CH$_2$Cl$_2$ = 4/1 v/v) and, subsequently, by crystallization with ethanol to afford 2 (0.65 g, 35%) as a red solid. m. p.: 117.5 ± 0.5 °C. $^1$H NMR (700 MHz, CDCl$_3$): δ 8.11 (dd, J = 3.7, 1.1 Hz, 1H), 7.86-7.73 (m, 3H), 7.46 (dd, J = 5.0, 1.0 Hz, 1H), 7.20 (dd, J = 5.0, 3.9 Hz, 1H), 7.14 (d, J = 3.9 Hz, 1H) ppm. $^{13}$C($^1$H) NMR (176 MHz, CDCl$_3$): δ 152.6, 152.2, 140.8, 139.3, 130.8, 128.1, 127.6, 127.4, 126.9, 126.4, 125.8, 125.1, 125.0, 114.6 ppm. HR-MS (ESI) calcd for C$_{14}$H$_7$BrN$_2$S$_3$Na (M+Na)$^+$: 402.8825 m/z; found: 402.8873 m/z. IR (KBr): $\nu_{\text{max}}$ 3090, 1481, 1423, 1215, 879, 833 cm$^{-1}$. Elem. Anal. calcd for C$_{14}$H$_7$BrN$_2$S$_3$: C, 44.33; H, 1.86, N, 7.39; found: C, 44.17; H, 1.86; N, 7.36.

4-(5-Trimethylsilylthiophen-2-yl)-7-(thiophen-2-yl)-benzo[2,1,3]thiadiazole (3). A mixture of 2 (0.39 g, 1.02 mmol), Pd(PPh$_3$)$_4$ (58.9 mg, 5.1 × 10$^{-2}$ mmol), CuI (9.7 mg, 5.1 × 10$^{-2}$ mmol) and trimethylsilylacetylene (0.11 g, 1.12 mmol) in triethylamine (15 mL) was refluxed for 24 h under vigorous stirring. After cooling down the reaction mixture to room temperature, methylene chloride (50 mL) was added and the resulting organic phase was washed with water (50 mL) and dried over Na$_2$SO$_4$. Upon the solvent removal, the crude product was purified by column chromatography (SiO$_2$, petroleum ether 40-60 °C/CH$_2$Cl$_2$ = 4/1 v/v) to give 3 (0.36 g, 89%) as an orange solid. m. p.: 120.7 ± 0.5 °C. $^1$H NMR (700 MHz, CDCl$_3$): δ 8.10 (dd, J = 4.1, 1.1 Hz, 1H), 7.92 (d, J = 4.1 Hz, 1H), 7.84-7.79 (m, 2H), 7.46 (dd, J = 5.2, 1.1 Hz, 1H), 7.30 (d, J = 4.1 Hz, 1H), 7.20 (dd, J = 5.2, 4.1 Hz, 1H), 0.29 (s, 9H) ppm. $^{13}$C($^1$H) NMR (176 MHz, CDCl$_3$): δ 152.8, 152.7, 140.9, 139.5, 132.5, 132.4, 128.9, 128.8, 128.3, 127.8, 127.4, 126.8, 125.4, 124.7, 101.2, 98.0, 0.2 ppm. HR-MS (ESI) calcd for C$_{19}$H$_{16}$N$_2$S$_3$SiNa (M+Na)$^+$: 419.0137 m/z; found: 419.0182 m/z. IR (KBr): $\nu_{\text{max}}$ 3078, 2952, 2920, 2849, 2138, 1482, 1247, 843, 801, 756, 683 cm$^{-1}$. Elem. Anal. calcd for C$_{19}$H$_{16}$N$_2$S$_3$Si: C, 57.54; H, 4.07; N, 7.06; found: C, 57.57; H, 4.10; N, 7.02.
4-(5-Ethynyl-thiophen-2-yl)-7-(thiophen-2-yl)-benzo[2,1,3]thiadiazole (4). A mixture of 3 (0.36 g, 0.90 mmol) and KF (0.26 g, 4.50 mmol) in methylene chloride (5 mL) and methanol (5 mL) was stirred for 30 min at 55 °C. After cooling down the reaction mixture to room temperature, methylene chloride (50 mL) was added and the resulting organic phase was washed with water (50 mL) and dried over Na2SO4. After solvent removal, the crude product was purified by column chromatography (SiO2, petroleum ether 40-60 °C/CH2Cl2 = 4/1 v/v) yielding 4 (0.26 g, 88%) as an orange solid. m. p.: 136.3 ± 0.5 °C. 1H NMR (700 MHz, CDCl3): δ 8.13 (dd, J = 4.1, 1.1 Hz, 1H), 7.95 (d, J = 4.1 Hz, 1H), 7.88-7.84 (m, 2H), 7.47 (dd, J = 5.2, 1.1 Hz, 1H), 7.35 (d, J = 4.1 Hz, 1H), 7.21 (dd, J = 5.2, 4.1 Hz, 1H), 3.48 (s, 1H) ppm. 13C{1H} NMR (176 MHz, CDCl3): δ 152.9, 152.8, 141.2, 139.5, 134.2, 128.4, 128.3, 128.1, 127.8, 127.5, 127.2, 127.0, 126.3, 126.1, 125.9, 125.4, 123.5, 83.3, 77.4 ppm. HR-MS (ESI) calcd for C16H8N2S3Na (M+Na)+: 346.9742 m/z; found: 346.9772 m/z. IR (KBr): νmax 3080, 2922, 2851, 2089, 1484, 1213, 876, 830, 801, 786, 699, 661 cm–1. Elem. Anal. calcd for C16H8N2S3: C, 59.23; H, 2.49; N, 8.63; found: C, 59.27; H, 2.50; N, 8.62.

4-((5-(7-(Thiophen-2-yl)-benzo[2,1,3]thiadazol-4-yl)-thiophen-2-yl-ethynyl)-benzaldehyde (5). A mixture of 4 (0.12 g, 0.37 mmol), 4-bromo-benzaleide (68.5 mg, 0.37 mmol), Pd(PPh3)4 (42.7 mg, 3.7 × 10–2 mmol) and CuI (7.1 mg, 3.7 × 10–2 mmol) in triethylamine (15 mL) was refluxed for 24 h under vigorous stirring. After cooling down the reaction mixture to room temperature, methylene chloride (50 mL) was added and the resulting organic phase was washed with water (50 mL) and dried over Na2SO4. After solvent removal, the crude product was purified by column chromatography (SiO2, CH2Cl2) yielding 5 (64.3 mg, 41%) as a red solid. m. p.: 181.3±0.9 °C. 1H-NMR (700 MHz, CDCl3): δ 10.5 (s, 1H), 8.17 (dd, J = 3.9, 1.2 Hz, 1H), 8.05 (d, J = 3.9 Hz, 1H), 7.93-7.90 (m, 4H), 7.71 (d, J = 8.5 Hz, 2H), 7.51 (dd, J = 5.0, 1.2 Hz, 1H), 7.45 (d, J = 3.9 Hz, 1H), 7.25 (dd, J = 5.0, 3.9 Hz, 1H) ppm. 13C{1H} NMR (176 MHz, CDCl3): δ 191.3, 152.6, 152.5, 141.8, 139.2, 135.5, 133.7, 131.8, 129.7, 129.2, 128.1, 127.9, 127.2, 127.2, 126.8, 126.0, 125.6, 125.0, 123.6 ppm. HR-MS (ESI) calcd for C23H12N2S3Na (M+Na)+: 451.0004 m/z; found: 450.9965
m/z. IR (KBr): $\nu_{\text{max}}$ 3070, 2920, 2857, 2187, 1682, 1600, 1210, 818 cm$^{-1}$. Anal. calcd for C$_{23}$H$_{12}$N$_2$O$_3$: C, 64.46; H, 2.82; N, 6.54; found: C, 64.44; H, 2.80; N, 6.53.

5-((5-(7-(Thiophen-2-yl)-benzo[2,1,3]thiadiazol-4-yl)-thiophen-2-yl-ethynyl)-thiophene-2-carbaldehyde (6). A mixture of 4 (0.13 g, 0.40 mmol), 5-bromo-thiophene-2-carbaldehyde (0.08 g, 0.44 mmol), Pd(PPh$_3$)$_4$ (46.2 g, 4.0 $\times$ 10$^{-2}$ mmol) and CuI (7.6 mg, 4.0 $\times$ 10$^{-2}$ mmol) in triethylamine (15 mL) was refluxed for 24 h under vigorous stirring. After cooling down the reaction mixture to room temperature, methylene chloride (50 mL) was added and the resulting organic phase was washed with water (50 mL) and dried over Na$_2$SO$_4$. After solvent removal, the crude product was purified by column chromatography (SiO$_2$, CH$_2$Cl$_2$) yielding 6 (95.6 mg, 55%) as a red solid. m. p.: 189.4±0.8 °C. $^1$H NMR (700 MHz, CDCl$_3$): $\delta$ 9.90 (s, 1H), 8.17 (d, J = 3.5 Hz, 1H), 8.04 (d, J = 4.2 Hz, 1H), 7.92 (s, 2H), 7.71 (d, J = 3.5 Hz, 1H), 7.51 (d, J = 4.8 Hz, 1H), 7.45 (d, J = 4.2 Hz, 1H), 7.36 (d, J = 3.5 Hz, 1H), 7.26-7.24 (m, 1H) ppm. $^{13}$C{$_1^H$} NMR (176 MHz, CDCl$_3$): $\delta$ 182.3, 152.6, 152.5, 144.2, 142.4, 139.2, 136.0, 134.0, 132.5, 132.4, 128.2, 127.9, 127.3, 127.2, 126.9, 126.1, 125.6, 124.8, 122.8, 91.6, 87.5 ppm. HR-MS (ESI) calcd for C$_{21}$H$_{11}$N$_2$O$_3$ (M+H)$^+$: 434.9749 m/z; found: 434.9676 m/z. IR (KBr): $\nu_{\text{max}}$ 3097, 2920, 2852, 2182, 1653, 1419, 1226, 834, 807 cm$^{-1}$. Anal. calcd for C$_{21}$H$_{10}$N$_2$O$_3$: C, 58.04; H, 2.32; N, 6.45; found: C, 58.00; H, 2.29; N, 6.43.

2-Cyano-3-(4-((5-(7-(Thiophen-2-yl)-benzo[2,1,3]thiadiazol-4-yl)-thiophen-2-yl)ethynyl)phenyl)-acrylic acid (DTB-B). A mixture of 5 (59.4 mg, 0.12 mmol), cyano-acetic acid (0.10 g, 1.20 mmol) and ammonium acetate (9.2 mg, 0.12 mmol) in acetic acid (10 mL) was refluxed overnight. The mixture was allowed to reach room temperature and then poured into water (200 mL) to obtain a precipitate that was collected by filtration and washed with water. The crude product was purified by column chromatography (SiO$_2$, CH$_2$Cl$_2$/MeOH = 10/1 v/v) affording DTB-B (54.7 mg, 92%) as a red solid. $^1$H NMR (700 MHz, DMSO-d$_6$): $\delta$ 8.42-8.01 (br, 8H), 7.85-7.73 (br, 2H), 7.64 (br, 1H), 7.30 (br, 1H) ppm. HR-MS (ESI) calcd for C$_{26}$H$_{12}$N$_3$O$_2$S$_3$ (M–H)$^-$: 494.0092 m/z; found: 494.0211 m/z. IR (KBr): $\nu_{\text{max}}$ 3097, 2921, 2226, 2183, 1696,
2-Cyano-3-(5-(7-(thiophen-2-yl)-benzo[2,1,3]thiadiazol-4-yl)-thiophen-2-yl)ethynyl)thiophen-2-yl)-acrylic acid (DTB-T). A mixture of 6 (73.9 mg, 0.17 mmol), cyano-acetic acid (0.14 g, 1.70 mmol) and ammonium acetate (13.1 mg, 0.17 mmol) in acetic acid (10 mL) was refluxed overnight. The mixture was allowed to reach room temperature and then poured into water (200 mL) to obtain a precipitate that was collected by filtration and washed with water. The crude product was purified by column chromatography (SiO$_2$, CH$_2$Cl$_2$/MeOH = 10/1 v/v) affording DTB-T (77.5 mg, 91%) as a red solid. $^1$H NMR (700 MHz, DMSO-d$_6$): $\delta$ 8.30-8.13 (br, 5H), 8.00 (br, 1H), 7.81 (d, J = 4.5 Hz, 1H), 7.71-7.53 (br, 2H), 7.30-7.28 (br, 1H) ppm. HR-MS (ESI) calcd for C$_{24}$H$_{11}$N$_3$O$_2$S$_4$ (M–H)$^-$: 499.9656 m/z; found: 499.9448 m/z. IR (KBr): $\nu_{\text{max}}$ 3097, 2920, 2220, 2176, 1690, 1575, 1410, 1270, 1235, 806 cm$^{-1}$. Anal. calcd for C$_{24}$H$_{11}$N$_3$O$_2$S$_4$: C, 57.46; H, 2.21; N, 8.38; found: C, 57.43; H, 2.23; N, 8.41.
**Figure S1.** Theoretical HOMO (red) and LUMO (black) energy levels of the sensitizers **G2**, **DTB-B** and **DTB-T** calculated at the B3LYP/6-311G(d,p) level of the theory in THF (CPCM method). The relevant chemical structures are also shown (R = 2-ethyl-hexyl).

**Figure S2.** Simulated absorption spectra and oscillator strengths of **G2**, **DTB-B** and **DTB-T** calculated *in vacuo* at the CAM-B3LYP/6-31G(d,p) level of the theory.
Figure 3S. Optimized molecular geometries of DTB-B (left) and DTB-T (right) calculated \textit{in vacuo} at the B3LYP/6-31G(d,p) level of the theory.

Figure 4S. Natural transition orbitals defining the photo-excitation dynamics of the main electronic transition in DTB-B and DTB-T calculated \textit{in vacuo} at the CAM-B3LYP/6-31G(d,p) level of the theory.
Figure 5S. HR-MS (ESI) spectrogram of DTB-B.

Figure 6S. HR-MS (ESI) spectrogram of DTB-T.
**Figure 7S.** Absorption spectra of G2, DTB-B and DTB-T recorded in THF.

**Figure 8S:** Absorption spectra recorded for G2, G2/DTB-B and G2/DTB-T sensitized 6 μm-thick TiO2 mesoporous films.
Figure 9S. Cyclic voltammogram of DTB-B.

Figure 10S. Cyclic voltammogram of DTB-T.
Device fabrication and characterization: Fluorine-doped tin oxide (FTO, 10 Ω/sq, provided by Solaronix S.A.) glass plates were cleaned in a detergent solution using an ultrasonic bath for 15 min and subsequently rinsed with water and ethanol. Double-layer photo-anodes (thickness 17 μm) were prepared as follows: i) a layer of commercial colloidal paste (Dyesol 18NR-T) was deposited onto the FTO glass and dried at 125°C for 15 min to obtain a transparent nano-crystalline film (~12 μm); ii) a scattering layer (~5 μm, Solaronix D/SP colloidal paste) was deposited onto the transparent layer; iii) a sintering process was performed at 450°C for 30 min. The thickness and the active area (0.16 cm²) of the sintered photo-anodes was measured using a profilometer (Tencor Alpha-Step 500 Surface Profiler). The dye loading was performed by immersing the photoanodes in 0.2 mM THF solutions of the individual dyes (G2, DTB-B or DTB-T for devices A, B and C, respectively) containing chenodeoxycholic acid (CDCA, 10 mM) and kept for 14 h in dark at room temperature. The co-sensitized photo-anodes were prepared by dyeing them into appropriate solutions of G2 (0.1 mM) and DTB-B (0.05 mM, device D) or DTB-T (0.05 mM, device E) in THF containing CDCA (10 mM). The counter-electrodes were prepared by sputtering a 50 nm Pt layer on a suitably cleaned FTO plate. The two electrodes were faced and assembled by means of a gasket of 50 mm-thick Surlyn® foil (Dyesol Ltd) interposed between them. The redox electrolyte (0.1 M LiI, 0.02 M I₂, 0.6 M 1-methyl-3-propylimidazolium iodide, and 0.5 M tert-butylpyridine in dry acetonitrile) was vacuum-injected into the space between the electrodes through holes suitably pre-drilled through the counter-electrodes.

The surface concentrations (dye loading) of the dyes were assessed by spectrophotometric determination as follows: double layered photoanodes (12 ± 5 μm, 1 cm²) were sensitized with the same solutions used for devices A-E; then the dyes were completely desorbed from the TiO₂ surface by immersing the substrates in a 0.01 M tetrabutylammonium hydroxide in DMF solution (20 ml). The molar concentration of each dye was estimated by the Lambert-Beer’s law from the absorption spectra of the resulting desorption solutions taking into account the molar extinction coefficient of the individual dyes at the corresponding λ max.

Photocurrent-voltage measurements were performed using a Keithley Model 2400 Source Meter. A Newport 91160A AM 1.5 Solar Simulator equipped with a 1000W xenon arc lamp served as a light
source. The light intensity (or radiant power) was calibrated to 100 mWcm$^{-2}$ using as reference a Si solar cell. A mask (0.25 cm$^2$ aperture) was applied to the devices before the measurements. The incident photon-to-current conversion efficiency (IPCE) was measured by the DC method. IPCE measurements were carried out with a computerized setup consisting of a xenon arc lamp (140 W, Newport, 67005) coupled to a Cornerstore 260 Oriel 74125 monochromator. Light intensity was measured by a calibrated UV silicon photodetector (Oriel 71675) and the short-circuit currents were measured by using a Newport 2936-C dual-channel optical power/energy meter.

Electrochemical impedance spectroscopy (EIS) was performed using a Metrohm Autolab PGSTAT 302N (Eco Chemie B.V.) potentiostat in a frequency range between 100 kHz and 10 mHz. The impedance measurements were carried out at different voltage biases under 1.0 sun illumination. The resulting impedance spectra were fitted with the ZView software (Scribner Associates). EIS spectra were analyzed through the well-known equivalent circuit. All electrochemical parameters have been plotted as a function of the corrected potential in order to take into account the losses due to the total series resistance, which lead to a potential drop, that is not associated with the displacement of the Fermi level.
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<th>$J_{sc}$ [mA/cm²]</th>
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**Table 1S.** Photovoltaic parameters of devices based on different dye mixtures measured under 1.0 sun illumination (AM 1.5, 100 mW/cm²).
Figure 11S. J-V curves for devices A-E.