Supporting Information

for

An ADMET route to low band gap poly(3-hexadecylthiénylene vinylene): A systematic study of molecular weight on photovoltaic performance

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Experimental Section

Materials and General Methods. Commercially available solvents and reagents were purchased and used as received from Aldrich and Acros. Degassed THF was purified by passage through an activated alumina column and collected in flame-dried, air-free flasks. All reactions were run under argon or vacuum using standard Schlenk techniques. $^1$H and $^{13}$C NMR spectra were acquired on either a Varian INOVA 500 or 300 MHz spectrometer. Spectra were referenced internally to tetramethylsilane ($^1$H) or to residual solvent peaks ($^{13}$C). High resolution mass spectrometry was performed on a Finnigan MAT 95 mass spectrometer operating in EI (electron impact) mode. Samples were introduced using a solid probe. Size exclusion chromatography (SEC) analysis was performed in CHCl$_3$ (1 mL/min, 35 °C) using a Hewlett Packard (Agilent Technologies) 1100 Series liquid chromatograph equipped with three PlGel 5 $\mu$m Mixed-C (Polymer Laboratories) columns with pore sizes of 500, $1\times10^3$, and $1\times10^4$ Å. The columns were calibrated using polystyrene standards (Polymer Laboratories), and the refractive index signal was recorded with a Hewlett Packard 1047A refractive index detector. Ultraviolet-visible (UV-Vis) absorption spectra for polymer solutions and thin films were acquired on a Spectronic Genesys 5 spectrometer over a wavelength range of 300–1000 nm. The solution spectra were obtained in a 1 cm quartz cuvette, and the film spectra were obtained by spin casting a CHCl$_3$ solution (10 mg/mL, 1500 rpm, 40 s) on a glass substrate. Elemental analysis was performed by Atlantic Microlab Inc. in Norcross, GA using combustion coupled with thermal conductivity detection. Polymer decomposition temperatures were determined by thermogravimetric analysis (TGA) using a PerkinElmer Pyris Diamond TG/DTA 6300 with aluminum sample pans. Polymer melting and crystallization transitions were quantified by differential scanning calorimetry (DSC) using either a TA Instruments Q1000 or Discovery DSC
both calibrated with indium. Cyclic voltammetry (CV) was run using a Pine Instruments bipotentiostat with a Pt wire counter electrode, Ag/AgCl reference electrode, and Au-coated Si working electrode. C16-PTV was spin coated onto the working electrode from a 1,2-dichlorobenzene solution, and voltammograms were recorded in acetonitrile with 0.1 M [Bu4N][PF6] as the supporting electrolyte at a scan rate of 20 mV/sec. The HOMO level was estimated from the oxidation onset relative to ferrocene using the equation: \( E_{\text{HOMO}} = -q(E_{\text{ox, onset}} \text{ vs. fer} + 4.8) \) \( (E_{\text{fer}} = 0.45 \text{ V vs. Ag/AgCl}) \). Temperature-dependent wide-angle X-ray scattering (TWAXS) data was collected at various temperatures in an N2 atmosphere using a Bruker D8 Advance and a temperature-controlled sample cell. WAXS thin film analysis was performed on quartz substrates using a Bruker-AXS Microdiffractometer. Poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS, Clevios P VP AI 4083) aqueous dispersion and [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) were purchased from Heraeus Materials Technology (West Conshohocken, PA) and American Dye Source (Baie-d’Urfé, Quebec) respectively. Atomic force microscopy (AFM) was performed on an Agilent 5500 environmental SPM with an open-loop scanner operating in tapping mode in the repulsive regime. AFM samples were prepared by spin coating a 1:4 wt% (C16-PTV:PCBM) solution in 1,2-dichlorobenzene onto a PEDOT:PSS coated glass slide.

**Device Fabrication and Characterization.** Photovoltaic devices were fabricated on patterned indium tin oxide (ITO) coated glass substrates (Delta Technologies (Stillwater, MN), sheet resistance 8–12 ohms/sq), cleaned with successive acetone, methanol, and isopropyl alcohol sonications followed by an UV/O3 exposure. PEDOT:PSS was spin-coated from a filtered water suspension twice at 4000 rpm, forming ca. 30-40 nm films as measured by profilometry (KLA Tencor P-10). Coated substrates were then transferred to nitrogen atmosphere glove box and
annealed at 135 °C for 10 minutes. All remaining fabrication steps were carried out in a N₂
glove box. PTV:PCBM active layers were spin-coated from 1,2-dichlorobenzene solutions,
varying spin speed and concentration to control active layer thickness. Wet spun devices were
placed in covered Petri dishes to slowly dry. Aluminum cathodes, *ca.* 100 nm as measured by
profilometry and quartz crystal microbalance, were formed via thermal evaporation at a rate of 2-
3 Å/min. Silver paste was applied to the anode and cathode contact pads facilitating ohmic
contact with the testing apparatus.

Bottom gate bottom contact geometry transistors were fabricated on doped Si wafers with
3000 Å of thermally grown oxide. Gold source-drain contacts, *ca.* 250 Å as measured by
 profilometry and quartz crystal microbalance, were deposited via electron beam evaporation
(Temescal) of a 25 Å chromium adhesion layer and 225 Å of gold, and patterned via the lift-off
technique. Substrates were cleaned with successive acetone, methanol, and isopropyl alcohol
sonications followed by an UV/O₃ exposure. In a N₂ glove box, PTV active layers were spin-
coated from 10 mg/ml 1,2-dichlorobenzene solutions at 2000 rpm, and baked on a hot plate at
105 °C for 10 minutes to drive off residual solvent.

PV current-voltage characteristics were collected using an Agilent 4155C Semiconductor
Parameter Analyzer, under dark conditions and simulated solar illumination using a 150 W Xe-
arc lamp (Oriel) with an AM 1.5 G filter, attenuated to 100 mW/cm². External quantum
efficiency spectra were obtained using a monochromator (Cornerstone 130 1/8 m) equipped with
gratings and filters (Newport Corp.) in conjunction SRB 10 DSP lock-in amplifier (Stanford
Research Systems) controlled by customized LabView code.
Transistors were tested in Desert Cryogenics vacuum probe station at room temperature housed within a N\textsubscript{2} glove box. Output and transfer curves were collected with Keithly 236, 237, and 6517A source meters controlled by customized LabView code.

Scheme S1. Synthesis of M1–M5.

Synthesis of 3-hexadecylthiophene. A 250 mL 3-neck round bottom flask was equipped with a magnetic stir bar, reflux condenser, glass stopcock, liquid addition funnel, and two rubber septa. Magnesium turnings (3.3 g, 136.5 mmol) were added and the apparatus was flame-dried under vacuum. Anhydrous THF (ca. 125 mL) was transferred via cannula into the flask followed by the addition of 1,2-dibromoethane (ca. 0.5 mL) to activate the magnesium. A solution of 1-bromohexadecane (20.0 mL, 65.5 mmol) in anhydrous THF (ca. 25 mL) was added to the liquid addition funnel via syringe and dripped into the Mg/THF mixture over the course of 20 min. Stirred at room temperature under argon for 2 h. The Grignard solution was transferred via cannula into a second flame-dried flask containing a magnetic stir bar, 3-bromothiophene (5.1 mL, 54.6 mmol), 1,3-bis(diphenylphosphino)propane nickel (II) chloride [Ni(dppp)Cl\textsubscript{2}] (0.60 g, 1.1 mmol), and anhydrous THF (ca. 25 mL) held at 0°C. The reaction was stirred under argon for 16 h. Reaction was quenched with 1 M NH\textsubscript{4}HCO\textsubscript{3} (ca. 50 mL), H\textsubscript{2}O (ca. 75 mL) was added, and extracted with diethyl ether (2 \times 75 mL). The combined organics were washed with H\textsubscript{2}O, saturated NaHCO\textsubscript{3}, and brine (ca. 75 mL each) before drying over Na\textsubscript{2}SO\textsubscript{4}. The solvent was removed under reduced pressure. The remaining residue was dissolved in minimal amount of THF (ca. 15 mL) and precipitated into MeOH (ca. 250 mL, 0 °C). The product was filtered and
dried under vacuum overnight to give an off-white solid (15.6 g, 93%). $^1$H NMR (500 MHz, CDCl$_3$): $\delta_H$ (ppm) = 7.23 (dd, 1H, $J = 4.80$, 3.22 Hz, Th-H5), 6.93 (d, 1H, $J = 4.93$ Hz, Th-H4), 6.91 (d, 1H, $J = 2.20$ Hz, Th-H2), 2.61 (t, 2H, $J = 7.71$ Hz, –CH$_2$–), 1.61 (quintet, 2H, $J = 7.34$, –CH$_2$–), 1.25 (bs, 26H, –C$_{12}$H$_{26}$–), 0.90 (t, 3H, $J = 6.89$ Hz, –CH$_3$). $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta_C$ (ppm) = 143.5 (Th-C3), 128.5 (Th-C4), 125.2 (Th-C5), 119.9 (Th-C2), 32.6, 30.8, 30.5, 29.9, 29.8, 29.7, 29.6 (2), 22.9 (–CH$_2$CH$_3$), 14.4 (–CH$_3$). HRGC-MS (EI): $M_{\text{calcd.}} = 308.2538$, $M_{\text{found}} = 308.2555$. Anal. calcd. for C$_{20}$H$_{36}$S: C 77.85, H 11.76, S 10.39; Found: C 78.64, H 11.96, S 9.23.

**Synthesis of 2,5-dicarbaldehyde-3-hexadecylthiophene.** A 3-neck 500 mL round bottom flask was equipped with a magnetic stir bar, reflux condenser, glass stopcock, liquid addition funnel, and two rubber septa. The apparatus was flame-dried under vacuum and 3-hexadecylthiophene (10.0 g, 32.4 mmol) was added as a solid under argon purge. Anhydrous hexanes (ca. 200 mL) were transferred via cannula into the flask followed by N,N,N’,N’-tetramethylethylenediamine (TMEDA) (12.1 mL, 81.0 mmol) addition via syringe. A 2.5 M solution of n-butyllithium in hexanes (32.4 mL, 81.0 mmol) was added to the liquid addition funnel via syringe. The n-butyllithium was added to the solution of 3-hexadecylthiophene, TMEDA, and hexanes over the course of 20 min. The solution was refluxed for 1 h following addition. Anhydrous THF (ca. 100 mL) was added to the flask and cooled to 0 °C on an ice bath, at which point anhydrous N,N’-dimethylformamide (DMF) (10.0 mL, 29.6 mmol) was added via syringe. The reaction was stirred for 1 h before adding 1 M HCl (ca. 150 mL) to quench. The aqueous phase was extracted with diethyl ether (2 × 75 mL), and the combined organics were washed with H$_2$O (ca. 100 mL) and brine (ca. 100 mL). The solution was dried over Na$_2$SO$_4$ and the solvent was removed under reduced pressure to give an orange-brown oil that solidified on standing (11.1 g, 94%). The product was used without further purification. $^1$H NMR (300 MHz, CDCl$_3$): $\delta_H$ (ppm) = 10.14 (s,
1H, Th-5CHO), 9.97 (s, 1H, Th-2CHO), 7.65 (s, 1H, Th-H4), 3.00 (t, 2H, \(J = 7.68\) Hz, \(-\text{CH}_2\)),
1.70 (quintet, 2H, \(J = 7.46\) Hz, \(-\text{CH}_2\)), 1.25 (bs, 26H, \(-\text{CH}_3\)), 0.88 (t, 3H, \(J = 6.70\) Hz, \(-\text{CH}_3\)).

\(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \(\delta\)C (ppm) = 183.7 (–CHO), 183.3 (–CHO), 152.3 (Th-C3), 148.1
(Th-C5), 143.6 (Th-C2), 137.5 (Th-C4), 31.5, 30.0–29.9 (m), 29.8, 29.6 (2), 29.5, 28.8, 23.0 (–CH\(_2\)CH\(_3\)), 14.4 (–CH\(_3\)). HRGC-MS (EI): M\(_{\text{calcd.}}\) = 364.2436, M\(_{\text{found}}\) = 364.2409. Anal. calcd. for
C\(_{22}\)H\(_{36}\)O\(_2\)S: C 72.48, H 9.95, O 8.78, S 8.79; Found: C 73.47, H 10.10, O 8.00, S 8.22.

**Synthesis of 2,5-dipropenyl-3-hexadecylthiophene (M1–M5).** A 2-neck 500 mL round bottom flask was equipped with a magnetic stir bar, reflux condenser, glass stopcock, and rubber septum. The apparatus was flame dried under vacuum before adding sodium bis(trimethylsilyl)amide (10.5 g, 57.5 mmol) and (ethyl)triphenylphosphonium bromide (21.3 g, 57.5 mmol) under an argon purge. The apparatus was placed under reduced pressure for 30 minutes to dry the solids. Anhydrous hexanes (ca. 300 mL) was transferred into the flask and refluxed for 1 h under argon to form the Wittig reagent (bright orange-red solution). The reaction was cooled to -78 °C on acetone/dry ice bath and the hexanes were removed with a cannula taking care not to remove precipitated Wittig reagent. The cold hexanes wash was repeated with another 300 mL. Residual hexanes were removed under vacuum and anhydrous THF (ca. 300 mL) was added to dissolve the remaining Wittig reagent. This solution was transferred via cannula into a 500 mL round bottom flask containing anhydrous THF (ca. 100 mL) and 2,5-dicarbaldehyde-3-hexadecylthiophene (10.0 g, 27.4 mmol) held at 0 °C. The reaction was stirred for 30 min. before filtering off Ph\(_3\)P=O. The filtrate was concentrated under reduced pressure to give a dark brown residue, which was dissolved in an ether:hexanes (10:90) solution (ca. 500 mL) and flushed through a silica gel plug to remove residual Ph\(_3\)P=O. The eluent was concentrated and dissolved in hexanes before flushing through a second silica gel plug. The solvent was removed under
reduced pressure to give a yellow oil that solidified on standing. The oil was dissolved in THF (ca. 30 mL) and precipitated into MeOH (ca. 300 mL) at -78 °C. The precipitation procedure was repeated and the pale yellow solid was dried under vacuum overnight (8.0 g, 75%). Highest Z:E from $^1$H NMR was 83:17. $^1$H NMR (500 MHz, CDCl$_3$): δ$_H$ (ppm) = 6.76, 6.67, 6.62 (s, 1H, Th-$H_4$ isomers), 6.54–6.37 (m, 2H, Th–$CH$–$CH$–$CH_3$), 6.07–5.90 (E), 5.70–5.58 (Z) (m, 2H, Th–$CH$–$CH$–$CH_3$), 2.56–2.49 (m, 2H, –$CH_2$–), 1.98 (Z), 1.85 (E) (m, 6H, Th–$CH$–$CH$–$CH_3$), 1.54 (m, 2H, –$CH_2$–), 1.25 (bs, 26H, –$CH_2$–), 0.88 (t, 3H, $J = 6.95$ Hz, –$CH_3$). $^{13}$C NMR (125 MHz, CDCl$_3$): δ$_C$ (ppm) = 141.3, 141.1, 140.7, 139.1, 138.6, 134.2, 132.0, 130.1, 129.3, 126.4, 125.4, 125.1, 124.8, 124.5 (2), 124.4, 124.1, 123.6, 123.5, 123.1, 121.6 (aromatic and olefinic Cs), 32.3, 31.1, 30.0 (3), 29.9, 29.8 (2), 29.7 (2), 23.0, 19.0, 18.7, 15.5 (3), 14.5 (aliphatic Cs). HRGC-MS (EI): $M_{\text{calc.}} = 388.3164$, $M_{\text{found}} = 388.3159$. Anal. calcd. for C$_{26}$H$_{44}$S: C 80.34, H 11.41, S 8.25; Found: C 81.35, H 11.39, S 7.45.

**General synthetic procedure for C16-PTV (P7).** An apparatus like that shown in the Figure S1 was assembled and flame-dried under vacuum. The Schlenk tube was charged with M5 (1.0 g, 2.57 mmol, Z:E = 83:17) in a solution of anhydrous 1,2,4-trichlorobenzene (TCB) (15 mL, ca. 0.17 M). The solution was heated to 80 °C and dynamic vacuum was applied for 15 min. to remove oxygen. The flask was placed under an argon atmosphere and G3 (23.0 mg, 0.0257 mmol) was added as a solution in TCB (0.5 mL, ca. 0.05 M). Dynamic vacuum was reapplied. Polymerization progress was observed by color change (yellow→orange→red→purple→dark blue). After 96 h the polymer was precipitated into a 20-fold excess of acetone held at 0 °C and filtered through a Soxhlet thimble. The polymer was purified by Soxhlet extraction with acetone (ca. 100 mL) and chloroform (ca. 100 mL). The chloroform fraction was concentrated to ~10 mL under reduced pressure and precipitated in 20-fold excess of acetone (0 °C). The polymer was
filtered and dried under vacuum to obtain the target material as a black solid (0.70 g, 82%). $^1$H NMR (500 MHz, CDCl$_3$): $\delta_H (ppm) = 7.00–6.80$ (m, 2H, olefinic Hs), 6.78 (bs, 1H, Th-H4), 6.44 (m, $E$ end group Th–CH=CH–CH$_3$), 6.01 (m, $E$ end group Th–CH=CH–CH$_3$), 2.62 (bs, 2H, –CH$_2$–), 2.19 (m, $E$ end group Th–CH=CH–CH$_3$), 1.60 (bs, 2H, –CH$_2$–), 1.25 (bs, 26H, –CH$_2$–), 0.87 (t, 3H, $J = 6.80$ Hz, –CH$_3$). SEC (CHCl$_3$, 1 mL/min, RI): $M_n = 33.3$ kg/mol, $M_w = 72.5$ kg/mol, PDI = 2.17. Anal. calcd. for C$_{22}$H$_{36}$S: C 79.45, H 10.91, S 9.64; Found: C 79.67, H 11.07, S 9.46.

**Figure S1.** ADMET polymerization apparatus

**C16-PTV (P1).** M1 (0.50 g, 1.3 mmol, $Z:E = 47:53$) in TCB (6 mL). Added G3 (11.5 mg, 0.013 mmol) in TCB (1 mL) and polymerized for 16 h before workup (0.29 g, 67%).

**C16-PTV (P2).** M1 (0.30 g, 0.77 mmol, $Z:E = 47:53$) in TCB (4 mL). Added G3 (7.0 mg, 0.0077 mmol) in TCB (1 mL) and polymerized for 24 h before workup (0.19 g, 73%).

**C16-PTV (P3).** M2 (0.30 g, 0.77 mmol, $Z:E = 67:33$) in TCB (5 mL). Added G3 (7.0 mg, 0.0077 mmol) in TCB (1 mL) and polymerized for 20 h before workup (0.22 g, 85%).
C16-PTV (P4). M3 (0.30 g, 0.77 mmol, Z:E = 82:18) in TCB (5 mL). Added G3 (7.0 mg, 0.0077 mmol) in TCB (1 mL) and polymerized for 24 h before workup (0.25 g, 96%).

C16-PTV (P5). M4 (0.30 g, 0.77 mmol, Z:E = 81:19) in TCB (5 mL). Added G3 (7.0 mg, 0.0077 mmol) in TCB (1 mL) and polymerized for 27 h before workup (0.23 g, 88%).

C16-PTV (P6). M5 (0.50 g, 1.29 mmol, Z:E = 83:17) in TCB (6 mL). Degassed with three freeze-pump-thaw cycles before adding G3 (11.0 mg, 0.0129 mmol) in TCB (0.5 mL). Polymerized for 48 h before workup (0.40 g, 93%).

Figure S2. $^{13}$C and $^1$H NMR spectra of 3-hexadecylthiophene in CDCl$_3$. 
Figure S3. $^{13}$C and $^1$H NMR spectra of 2,5-dicarbaldehyde-3-hexadecylthiophene in CDCl$_3$. 
Figure S4. $^{13}$C and $^1$H NMR spectra of 2,5-dipropenyl-3-hexadecylthiophene.
**Figure S5.** $^1$H NMR spectra of M1–M5 illustrating different Z:E ratios.

**Figure S6.** Representative $^1$H NMR spectra of C16-PTV (P7).
**Figure S7.** ¹H NMR spectrum of P1 highlighting end group and repeat unit integrals. Integral values are arbitrary.

Propenyl (c) and aldehyde (a) end groups, when compared to main chain resonances, can be used to quantify polymer conversion. The aldehyde resonances were propenyl groups before reaction with oxygen, and though unreactive, are still counted as chain ends. At 0% conversion the propenyl and α-methylene integrals are 1:1, and at 50% conversion they are 1:2. Following this trend and assuming no converted monomer is lost during workup, conversion (p) can be determined by dividing the end group integrals by repeat unit integrals (d).

\[
p = 1 - \frac{a + c}{d} = 1 - \frac{13.2}{266.2} = 0.95
\]

ADMET is a step polymerization, and as such, molecular weight estimation is made possible by the Carothers equation (Odian, G. *Principles of Polymerization*; John Wiley & Sons, Inc.: Hoboken, NJ, 2004; pp 50–79.) In this case it is assumed that G3 acts as a monofunctional contaminant that limits molecular weight. From this the stoichiometric imbalance (r) can be calculated using the monomer:catalyst mole ratio (100:1) where NA refers to the total number of reactive groups (2 per monomer) and NB refers the monofunctional impurity (G3). The coefficient of 2 in front of NB is necessary because one catalyst molecule has the same quantitative effect as one monomer molecule.

\[
r = \frac{N_A}{N_A + 2N_B} = \frac{200}{200 + 2} = 0.99
\]

Combining p and r into the Carothers equation for stoichiometric imbalance gives the predicted number-average degree of polymerization (Xn).
\[ X_n = \frac{1 + r}{1 + r - 2rp} = \frac{1.99}{1.99 - 2(0.99)(0.95)} = 18 \]

Multiplying \( X_n \) by the repeat unit molecular weight (\( M_0 \)), and neglecting end groups, gives the number-average molecular weight (\( M_n \)).

\[ M_n = X_n \times M_0 = 18 \times 332.59 \text{ g} \cdot \text{mol}^{-1} = 6.0 \text{ kg} \cdot \text{mol}^{-1} \]

**Figure S8.** McMurry coupling of aldehyde terminated monomer. SEC shows post reaction increase in molecular weight. Inset illustrates disappearance of aldehyde resonances in \(^1\text{H} \) NMR.

**Figure S9.** Cyclic voltammogram of P7 as a thin film spin coated onto an Au-coated silicon wafer.
Figure S10. Thermogravimetric analysis of P7 in an N2 and oxidizing (air) atmosphere. Decomposition temperature (T_d) was determined as the temperature where 5% of the polymer weight (dashed line) was lost.

Figure S11. (a) WAXS of P1 and P7 as thin films on quartz substrates. (b) Schematic illustration of crystalline C16-PTV domain. L refers to lamellar spacing, C to chain-to-chain, and π to π-stacking.
Figure S12. Powder TWAXS of (a) P1 and (b) P7. L refers to lamellar spacing, C to chain-to-chain, and π to π-stack.

Figure S13. DSC (a) second heating and (b) first cooling thermograms of P1–P7. All sweeps were performed at 10 °C/min. LC denotes the liquid crystalline region. Sweep direction (→), π-stack side chain melt (●), and π-stack melt (▼).
Figure S14. Thin film UV-Vis spectra of P7:PCBM containing 10-90% PCBM.

Figure S15. Thin film UV-Vis spectra of 1:4 PTV:PCBM containing P1–P5 & P7.
Figure S16. (a) UV-Vis spectra of P6 films with various thickness and (b) plot of absorbance vs. film thickness as $\lambda = 630$ nm from which absorption coefficient was extracted.
Figure S17. External quantum efficiency of 1:4 P6:PCBM device.
Figure S18. DSC thermograms of P1-P7 in 1:4 PTV:PCBM blends at a scan rate of 10 °C/min: (a) first cooling; (b) second heating.
Figure S19. Fourier transform of AFM phase image and P.S.D plot of 20:80 (a & b), 35:65 (c & d), and 50:50 (e & f) P7:PCBM samples. This data was obtained from the images shown in Figure 10.
Figure S20. AFM (a) height and (b) phase images of 1:4 P1:PCBM. (c) Fourier transform of AFM phase image and (d) P.S.D plot.