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Fused H-shaped tetrathiafulvalene-oligothiophenes as charge transport materials for OFETs and OPVs

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General Experimental:

LDA was purchased from Sigma Aldrich and titrated against menthol with 2,2'-bipyridyl as indicator before use. *N*-Bromosuccinimide (NBS) was purchased from Sigma Aldrich and recrystallized from water and dried under vacuum. Tetrakis(triphenylphosphine)palladium(0) ($Pd(PPh_3)_4$) was prepared prior to use and stored under nitrogen. Unless otherwise stated, all other reagents were sourced commercially and used without further purification. Dry solvents were obtained from a solvent purification system (SPS 400 from Innovative Technologies) using alumina as the drying agent. Melting points were taken using a TA Instruments DSC Q1000 Differential Scanning Calorimeter and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX 500 apparatus at 500.13 and 125.76 MHz. Chemical shifts are given in ppm; all J values are in Hz. Elemental analyses were obtained on a Perkin-Elmer 2400 analyser. MS LDI-TOF spectra were run on a Shimadzu Axima-CFR spectrometer (mass range 1-150000 Da). Thermogravimetric analysis (TGA) was performed using a Perkin-Elmer Thermogravimetric Analyzer TGA7 under a constant flow of helium.

Cyclic voltammetry (CV) measurements were performed on a CH Instruments 660A electrochemical workstation with *iR* compensation using anhydrous dichloromethane as the solvent. The electrodes were glassy carbon, platinum wire and silver wire as the working, counter and reference electrodes, respectively. All solutions were degassed (Ar) and contained monomer substrates in concentrations of *ca.* 10^{-4} M, together with n-Bu_4NPF_6 (0.1 M) as the supporting electrolyte. All measurements are referenced against the $E_{1/2}$ of the Fc/Fc^+ redox couple. Spectroelectrochemical experiments were conducted on ITO glass. Absorption spectra and CIE coordinates were recorded on a Unicam UV 300 instrument.

Simultaneous solution EPR/UV-Vis-NIR spectroelectrochemistry has been carried using JEOL JES FA-200 X-band EPR spectrometer, equipped with JEOL ES-MCX3B transmission cavity. UV-Vis-NIR spectra were recorded with a combination of Ocean Optics QE 65000 and NIRQuest 512 diode array spectrometers, connected to the EPR cavity by means of fibre optic cables. Potential was applied using Autolab PGSTAT100N potentiostat. Measurements were performed in the same electrolytic environment as the electrochemical experiments. Platinum mesh was used as a working electrode, while suitably long platinum wire and a thin silver wire were used as counter and pseudoreference electrodes, with the latter calibrated versus the Fc^+/Fc formal redox couple potential. g-Factor of radicals was determined using JEOL internal standard – the $^{55}Mn^{2+}$ radical, whose 3rd hyperfine line has a g-factor of 2.03323.

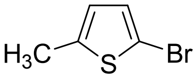
Commercially available (from Fraunhofer Institute) Silicon substrates (n-doped) with 200 nm of thermally grown SiO_2 with prefabricated interdigiated Au fingers having channel length of 10 µm, width of 1 cm and capacitance of $1.7x10^{-8}$ F/cm^2 were used as substrates. SiO_2 substrates were treated with octadecyltrichlorosilane (OTS) for 30 s and the gold electrodes were treated with pentafluorobenzenethiol. Chloroform solutions of quinquethiophene-TTF **2** (6 mg/cm^3) were spin cast to these Si/SiO_2 substrates followed by annealing at 120°C for 20 min. Current-voltage measurements were performed using a Keithley 4200 at room temperature in a glove box with water and oxygen levels lower than 0.1 ppm.

Solar cells were prepared as small area (0.04 cm^2) sandwich devices. The hole conductor PEDOT: PSS was spin coated on a pre-cleaned, patterned indium tin oxide (ITO)/glass substrate and baked for 120^{0}C for 20 minutes. The blend oligomer (quinquethiophene-TTF **2**) /$PC_{71}BM$ prepared in the solution of O-dichlorobenzene were spin coated on top of the annealed PEDOT: PSS layer. Then the devices once again subjected to annealing at 120^{0}C for 20 minutes. Then the fabrication was finalized by the deposition of a 40 nm thick calcium layer and a 40 nm of aluminium layer as top electrodes. The geometry of the electrode was defined by a shadow mask. Calcium and aluminium were thermally evaporated at a pressure of $6X10^{-6}$ mbar. Solar cell preparation and characterisation were performed in an oxygen free environment using Nitrogen filled glove box (Mbraun). Current–voltage characteristics were measured using a Keithley 4200 SMU while the solar cells were illuminated by a Newport Solar Simulator at 1 sun white light.

Synthetic Procedures:

Structures **3**, **8** and **11** were prepared according to our previously published procedure.[1]

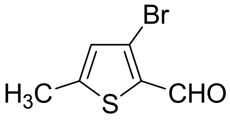
2-Bromo-5-methylthiophene (5):



5

Under exclusion of light, NBS (18.11 g, 101.86 mmol) was added portionwise to 2-methylthiophene (**4**) (10.00 g, 101.86 mmol) in DMF (150 mL). The reaction was then stirred overnight at room temperature before being poured into water (600 mL). The suspension was extracted with Et_2O (3 x 150 mL) before the combined organic extracts were washed with brine, dried (Na_2SO_4) and the solvent removed under reduced pressure. The material was purified *via* column chromatography (silica, hexane) to give a colourless oil (14.96 g, 83%), which was used immediately to avoid any possible decomposition.

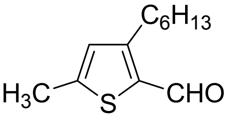
3-Bromo-5-methylthiophene-2-carbaldehyde (6):



6

Under N_2, 2-bromo-5-methylthiophene **5** (14.96 g, 84.49 mmol) was dissolved in anhydrous THF and cooled to -70 °C. LDA.THF (1.5 M solution in cyclohexane, 67.6 mL, 101.39 mmol) was added dropwise and the reaction was stirred for 2 h. Dry DMF (8.58 g, 109.84 mmol) was added and the reaction was stirred at -70 °C for 30 min before being allowed to warm to room temperature and stirred overnight. The reaction mixture was poured into water and extracted with Et_2O (3 × 200 mL) before being dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification was achieved by distillation under high vacuum (kugelrohr, b.p. 80 °C at 6.8 x 10^{-2} mbar) to give a black/brown oil, which was dissolved in CH_2Cl_2 and stirred with decolourising charcoal for 10 min before being filtered through a celite plug (eluting with CH_2Cl_2) to give a brown oil (11.00 g, 63%); ^{1}H NMR ($CDCl_3$): δ = 9.88 (1H, s), 6.86 (1H, s), 2.56 (3H, s).

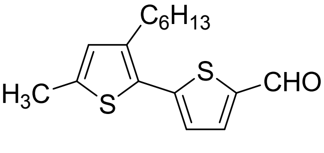
3-Hexyl-5-methylthiophene-2-carbaldehyde (7):



7

Under N_2, $ZnCl_2$ (11.58 g, 84.97 mmol) was suspended in dry THF (60 mL) and cooled to -60 °C. Hexyl lithium (2.3M in hexane, 33.58 mL, 77.24 mmol) was added slowly and the reaction was stirred for 30 min to generate hexylzinc chloride. The organozinc solution was then allowed to warm to room temperature before being added *via* cannula to a solution of **6** and $Pd(PPh_3)_4$ (1.55 g, 1.34 mmol) in THF (30 mL). The reaction was heated to reflux overnight then filtered prior to hydrolysis with 2N HCl (100 mL) and extraction with Et_2O (400 mL). The organic extract was washed with H_2O (2 x 150 mL), brine (2 x 150 mL), dried over $MgSO_4$ and the solvent was removed under reduced pressure. Purification was achieved by column chromatography (silica, CH_2Cl_2/hexane 2:1) to yield the desired product as a dark brown oil (7.60 g, 67 %); ^{1}H NMR ($CDCl_3$): δ = 9.93 (1H, s), 6.69 (1H, m), 2.88 (2H, t, *J* = 7.75), 2.51 (3H, d, *J* = 1.00), 1.64 (2H, quintet, *J* = 7.62), 1.34 (6H, m), 0.89 (3H, t, *J* = 7.00); ^{13}C NMR ($CDCl_3$): δ = 181.8, 154.0, 150.8, 136.1, 129.8, 31.8, 31.6, 29.2, 28.7, 22.7, 16.4, 14.3; MS (MALDI): m/z = 211 $[M + H^+]^+$.

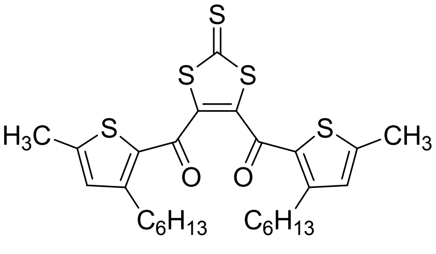
3'-Hexyl-5'-methyl-[2,2'-bithiophene]-5-carbaldehyde (10):



10

Under N_2, 3-hexyl-5-methyl-2,2'-bithiophene[1] (7.39 g, 27.94 mmol) was dissolved in dry THF (100 mL) and cooled to 0 °C. *n*-BuLi (2.36 M in hexanes, 15.29 mL, 36.33 mmol) was added drop wise and the reaction was stirred for 15 min. Dry DMF (3.01 mL, 36.33 mmol) was added and the reaction was stirred overnight at room temperature. The reaction mixture was then poured into water (200 mL) and extracted with ethyl acetate (3 x 100 mL) before the combined organic extracts were washed with water (3 x 150 mL), dried over $MgSO_4$ and the solvent removed under reduced pressure. The product was purified *via* column chromatography (silica, $CHCl_3$/ hexane 3:2), followed by distillation under high vacuum (kugelrohr, b.p. 175 °C at 6.8×10^{-2} mbar) to give the product as a yellow oil (6.35 g, 78 %); ^{1}H NMR ($CDCl_3$): δ = 9.87 (1H, s), 7.68 (1H, d, *J* = 4.00), 7.15 (1H, d, *J* = 4.00), 6.65 (1H, d, *J* = 0.50), 2.75 (2H, t, *J* = 7.75), 2.47 (3H, d, *J* = 1.00), 1.64 (2H, quintet, *J* = 7.75), 1.36 (6H, m), 0.90 (3H, t, *J* = 7.00); ^{13}C NMR ($CDCl_3$): δ = 182.8, 147.3, 142.5, 141.8, 140.8, 137.1, 129.4, 127.5, 125.6, 31.2, 30.5, 29.9, 29.4, 22.8, 15.5, 14.2; MS (MALDI): m/z = 291 $[M - H]^+$.

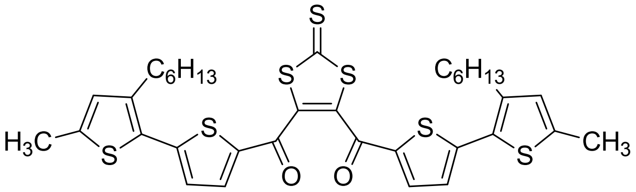
(2-Thioxo-1,3-dithiole-4,5-diyl)bis((3-hexyl-5-methylthiophen-2-yl)methanone) (12):



12

Under N_2, vinylene trithiocarbonate (**11**) (612 mg, 4.56 mmol) was dissolved in dry THF (20 mL) at -55 °C. LDA.THF (1.5 M solution in cyclohexane, 3.34 mL, 5.02 mmol) was added and the reaction was stirred for 20 min at -55 °C. Compound **7** (1.06 g, 5.03 mmol) was added dropwise as a solution in dry THF (15 mL) before the reaction was stirred for a further 20 min at -55 °C. A second equivalent of LDA.THF (1.5 M solution in cyclohexane, 3.34 mL, 5.02 mmol) was added and the reaction was stirred for 15 min at -55 °C. The cooling bath temperature was reduced to -80 °C, and a second equivalent of **7** (1.06 g, 5.02 mmol) in dry THF (15 mL) was added. The reaction was allowed to continue until the reaction temperature warmed to -50 °C, whereupon the reaction was allowed to attain room temperature without external cooling. The reaction mixture was then poured into a solution of saturated $NaHCO_3$ (200 mL) to which KBr (20 g) was added. The product was extracted with ethyl acetate (3 x 60 mL) and the combined organic layers were dried over Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified via column chromatography (silica, CH_2Cl_2 then ethyl acetate) to obtain the mono and diol products respectively in order of elution. After removal of solvent under reduced pressure the diol was obtained as a yellow/brown oil and was reacted immediately. The diol (3.51 g) was dissolved in CH_2Cl_2 (60 mL) and to this a ten-fold excess (w/w) of MnO_2 (35.1g) was added portion wise (yet quickly), and the mixture stirred for 1 min at room temperature before being filtered immediately through a plug of silica (ca. 2.5 x 5 cm thick), eluting with CH_2Cl_2 (approx. 200 mL). The solvent was removed under reduced pressure and purification was achieved *via* column chromatography (silica, CH_2Cl_2/hexane 1:1) prior to recrystallisation from hexane to give **12** as bright yellow crystals (1.32 g, 53 %); m.p. 89 – 90 °C; ^1H NMR ($CDCl_3$): δ = 6.62 (2H, s), 2.68 (4H, t, J = 7.76), 2.43 (6H, s), 1.39 (4H, broad quintet, J = 7.17), 1.25 (12H, m), 0.86 (6H, t, J = 7.02); ^{13}C NMR ($CDCl_3$): δ = 208.5, 175.6, 154.6, 149.8, 146.8, 131.8, 131.0, 31.9, 20.5, 30.3, 29.5, 22.8, 16.0, 14.3; MS (MALDI): m/z = 518 $[M - S]^+$, 549 $[M - H]^+$, 551 $[M + H]^+$; Elemental Analysis: Found C, 58.89; H, 6.20; S, 29.27 Calculated: C, 58.87; H, 6.22; S, 29.10.

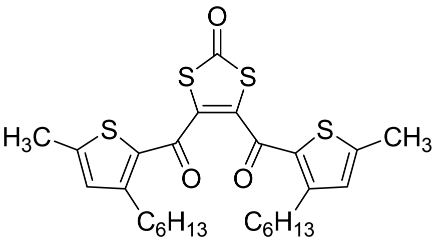
(2-Thioxo-1,3-dithiole-4,5-diyl)bis((3'-hexyl-5'-methyl-[2,2'-bithiophen]-5-yl)methanone) (13):



13

Under N_2, vinylene trithiocarbonate (**11**) (616 mg, 4.58 mmol) was dissolved in dry THF (20 mL) at -55 °C. LDA.THF (1.3 M solution in cyclohexane, 3.87 mL, 5.04 mmol) was added and the reaction was stirred for 20 min at -55 °C. Compound **10** (1.48 g, 5.04 mmol) was added dropwise as a solution in dry THF (15 mL) before the reaction was stirred for a further 20 min at -55 °C. A second equivalent of LDA.THF (1.3 M solution in cyclohexane, 3.87 mL, 5.04 mmol) was added and the reaction was stirred for 15 min at -55 °C. The cooling bath temperature was reduced to -80 °C, and a second equivalent of **10** (1.048 g, 5.04 mmol) in dry THF (15 mL) was added. The reaction was allowed to continue until the reaction temperature warmed to -50 °C, whereupon the reaction was allowed to attain room temperature without external cooling. The reaction mixture was then poured into a solution of saturated $NaHCO_3$ (200 mL) to which KBr (20 g) was added. The product was extracted with ethyl acetate (3 x 60 mL) and the combined organic layers were dried over Na_2SO_4. The solvent was removed in *vacuo* and the residue was purified *via* column chromatography (silica, CH_2Cl_2 then ethyl acetate) to obtain the mono and diol products respectively in order of elution. The solvent was removed under reduced pressure and the diol was obtained as a dark red/brown oil which was reacted immediately. The diol (4.03 g) was dissolved in CH_2Cl_2 (80 mL) and to this a ten-fold excess (w/w) of MnO_2 (40.3 g) was added portion wise (yet quickly), and the mixture stirred for 1 min at room temperature before being filtered immediately through a plug of silica (ca. 2.5 x 5 cm thick), eluting with CH_2Cl_2 (approx. 200 mL). Purification was achieved *via* column chromatography (silica, CH_2Cl_2/petroleum ether 1:1) to give a thick red oil (1.72 g, 53 %); ^{1}H NMR ($CDCl_3$): δ = 7.62 (2H, d, *J* = 4.00), 7.03 (2H, d, *J* = 4.00), 6.61 (2H, m), 2.61 (4H, t, *J* = 8.00), 2.44 (6H, d, *J* = 1.00), 1.55 (4H, broad quintet, *J* = 7.50), 1.30 (12H, brm), 0.89 (6H, t, *J* = 7.00); ^{13}C NMR ($CDCl_3$): δ = 207.9, 175.2, 149.8, 144.4, 143.2, 141.4, 139.8, 136.1, 129.4, 127.1, 125.5, 31.6, 30.1, 29.8, 29.2, 22.6, 15.4, 14.1; MS (MALDI): m/z = 683 $[M – S]^+$, 715 $[M]^+$.

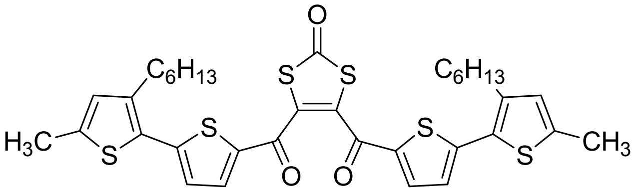
(2-Oxo-1,3-dithiole-4,5-diyl)bis((3-hexyl-5-methylthiophen-2-yl)methanone) (14):



14

1,3-Dithiol-2-thione **12** (1.31 g, 2.38 mmol) was dissolved in 3:1 CH_2Cl_2/acetic acid (100ml). $Hg(OAc)_2$ (1.05 g, 3.33 mmol) was added and the reaction was stirred overnight at room temperature. The reaction mixture was filtered through a plug of silica before the filtrate was washed with water (2 x 200 mL) and saturated $NaHCO_3$ solution (2 x 200 mL) and the solvent was removed under reduced pressure. Purification was achieved by column chromatography (silica, CH_2Cl_2/hexane 2:1), followed by recrystallisation from hexane to give **14** as round bright yellow crystals (1.12 g, 88 %). m.p. 54 – 55 °C; ^{1}H NMR ($CDCl_3$): δ = 6.64 (2H, s), 2.68 (4H, t, *J* = 7.75), 2.45 (6H, s), 1.42 (4H, broad quintet, *J* = 7.25), 1.29 (12H, m), 0.89 (6H, t, *J* = 6.90); ^{13}C NMR ($CDCl_3$): δ =187.5, 177.0, 153.9, 149.3, 137.7, 132.1, 130.6, 31.6, 30.2, 30.1, 29.3, 22.6, 15.8, 14.1; MS (MALDI): m/z = 535 $[M + H]^+$; Elemental Analysis: Found C, 60.54; H, 6.64; S, 23.82 Calculated: C, 60.64; H, 6.41; S, 23.98.

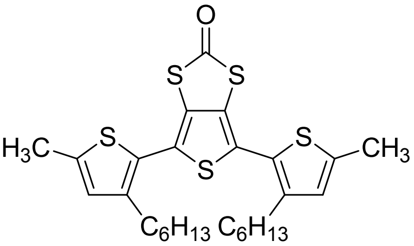
(2-Oxo-1,3-dithiole-4,5-diyl)bis((3'-hexyl-5'-methyl-[2,2'-bithiophen]-5-yl)methanone) (15):



15

1,3-Dithiol-2-thione **13** (1.72 g, 2.41 mmol) was dissolved in 3:1 CH_2Cl_2/acetic acid (100ml), $Hg(OAc)_2$ (1.07 g, 3.37 mmol) was added and the reaction was stirred overnight at room temperature. The reaction mixture was filtered through a plug of silica before the filtrate was washed with water (2 x 250 mL) and saturated $NaHCO_3$ solution (2 x 250 mL) and the solvent was removed under reduced pressure. Purification by column chromatography (silica, CH_2Cl_2/hexane 2:1) gave **15** as a thick red oil (1.41 g, 84 %); ^1H NMR ($CDCl_3$): $\delta = 7.59$ (2H, d, $J = 4.00$), 7.02 (2H, d, $J = 4.00$), 6.61, (2H, m), 2.61 (4H, t, $J = 7.75$), 2.44 (6H, d, $J = 1.00$), 1.54 (4H, broad quintet, $J = 7.63$), 1.19 (12H, brm), 0.89 (6H, brm); ^{13}C NMR ($CDCl_3$): $\delta = 187.3$, 176.9, 149.4, 143.0, 141.2, 140.1, 136.0, 135.6, 129.3, 127.1, 125.4, 31.6, 30.1, 29.8, 29.2, 22.6, 15.3, 14.1; MS (MALDI): m/z = 699 $[M]^+$.

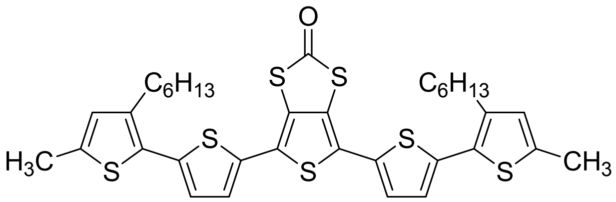
4,6-Bis(3-hexyl-5-methylthiophen-2-yl)thieno[3,4-d][1,3]dithiol-2-one (16):



16

1,3-Dithiole-2-one **14** (1.00 g, 1.87 mmol), P_4S_{10} (2.07 g, 4.66 mmol), and $NaHCO_3$ (786 mg, 9.35 mmol) were stirred together in 1,4-dioxane (40 mL) under N_2. The reaction was heated from 60 – 90 °C over 30 min, then stirred at 90 °C for 3 h. The reaction mixture was then allowed to cool to room temperature and water was added (10 x 10 mL portions) (CAUTION! H_2S and CO_2 gas evolution). The resulting suspension was heated to reflux for 1 h before being allowed to cool and stirred overnight at room temperature. The precipitate was filtered off over a glass sinter and washed with copious amounts of water before being dried under a stream of air. The precipitate was washed through the sinter with copious CS_2 before drying over Na_2SO_4 and removal of solvent under reduced pressure. Purification was achieved using column chromatography (silica, CS_2), then a further short silica column using pentane as the eluent. The product was isolated as yellow oil which solidified over time to give a brown solid (746 mg, 75 %); Td = 345 °C, m.p. 46 – 48 °C; ^1H NMR ($CDCl_3$): $\delta = 6.65$ (2H, m), 2.64 (4H, t, $J = 7.83$), 2.49 (6H, s), 1.60 (4H, broad quintet, $J = 7.56$), 1.30 (12H, m), 0.88 (6H, t, $J = 6.83$); ^{13}C NMR ($CDCl_3$): $\delta = 193.0$, 142.6, 140.6, 128.3, 126.5, 126.1, 124.5, 31.6, 30.7, 29.5, 29.2, 22.6, 15.4, 14.1; MS (MALDI): m/z = 533 [M – H]$^+$; Elemental Analysis: Found C, 60.57; H, 6.01; S, 29.94 Calculated: C, 60.63; H, 6.41; S, 29.97.

4,6-Bis(3'-hexyl-5'-methyl-[2,2'-bithiophen]-5-yl)thieno[3,4-d][1,3]dithiol-2-one (17):



17

1,3-Dithiole-2-one **15** (1.41 g, 2.02 mmol), P_4S_{10} (4.48 g, 10.08 mmol), and $NaHCO_3$ (848 mg, 10.08 mmol) were stirred together in 1,4-dioxane (60 mL) under N_2. The reaction was heated from 60 – 90 °C over 30 min then stirred at 90 °C for 3 h. The reaction mixture was then allowed to cool to room temperature and water was added (10 x 10 mL portions) (CAUTION! H_2S and CO_2 gas evolution). The resulting suspension was heated to reflux for 1 h before being allowed to cool and stirred overnight at room temperature. The precipitate was filtered off over a glass sinter and washed with copious amounts of water before being dried under a stream of air. The precipitate was washed through the sinter with copious CS_2 before drying over Na_2SO_4 and removal of solvent under reduced pressure. Purification was achieved using column chromatography (silica, CS_2), followed by recrystallisation from cyclohexane to obtain a shiny orange precipitate which was filtered and washed with cold methanol before drying in a desiccator (1.06 g, 75 %). Td = 419 °C, m.p. 47 – 48 °C; ^{1}H NMR ($CDCl_3$): δ = 7.14 (2H, d, J = 4.00), 7.02 (2H, d, J = 3.50), 6.63 (2H, m), 2.72 (4H, t, J = 7.75), 2.47 (6H, d, J = 0.50), 1.64 (4H, broad quintet, J = 7.38), 1.35 (12H, m), 0.91 (6H, m); ^{13}C NMR ($CDCl_3$): δ = 192.0, 140.5, 139.0, 137.9, 133.2, 128.7, 127.3, 125.9, 125.4, 125.3, 124.0, 31.7, 30.6, 29.5, 29.3, 22.7, 15.3, 14.1; MS (MALDI): m/z = 697 [M – H]$^+$, 669 [M – CO – H]$^+$; Elemental Analysis: Found C, 60.31; H, 5.40; S, 32.43 Calculated: C, 60.13; H, 5.48; S, 32.43.

Optical and electrochemical properties of half-units

Figure S1: cyclic voltammetry of half-units **16-18**; A, oxidation and B, reduction.

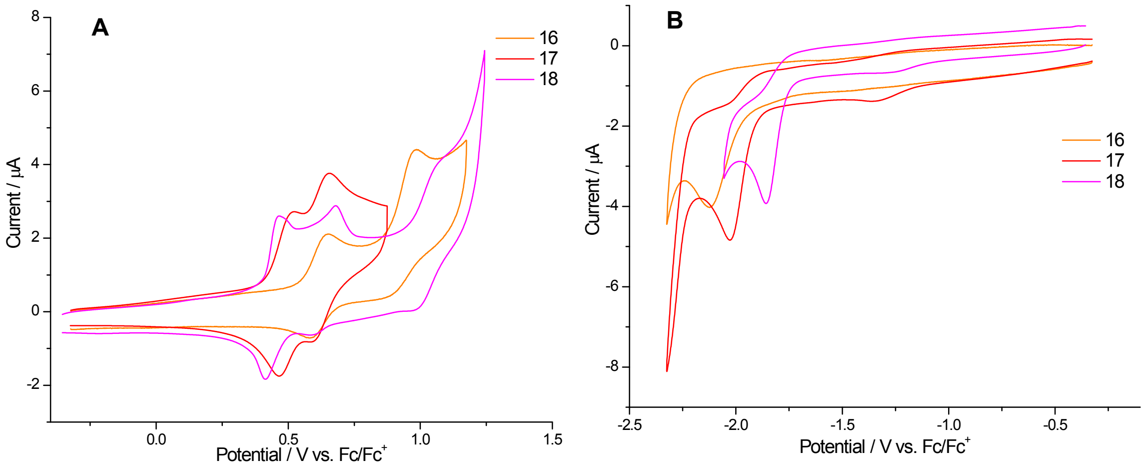
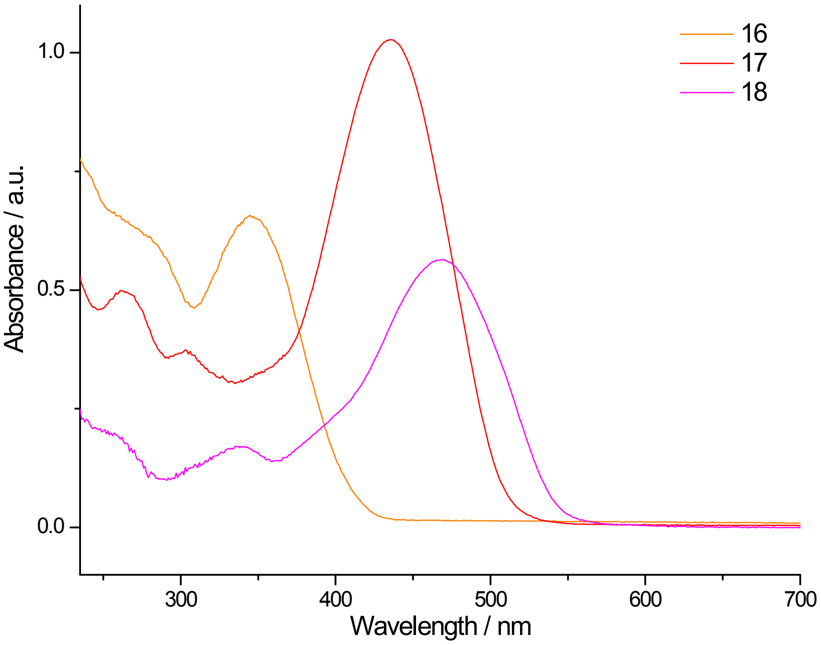


Table S1: UV-vis absorption and electrochemical results obtained for oligothiophene half-units **16-18**.

	λ_{max} (nm)	HOMO-LUMO gap (eV)	E_{1ox} (V)	E_{2ox} (V)	E_{3ox} (V)	E_{red} (V)	HOMO (eV)[a]	LUMO (eV)[a]	HOMO-LUMO gap (eV)[b]
16	345	3.03	+0.65/ +0.58	+0.99/ +0.88[q]	-	-2.12[irr]	-5.34	-2.84	2.50
17	435	2.44	+0.52/ +0.47	+0.66/ +0.58[q]	-	-2.03[irr]	-5.21	-2.88	2.33
18	467	2.29	+0.47/ +0.41	+0.68/ +0.59[q]	+1.05/ +0.98[q]	-1.86[irr]	-5.18	-3.08	2.10

[a] HOMO and LUMO levels are calculated from the onset of the first peak of the corresponding redox wave and are referenced to ferrocene which has a HOMO of -4.8 eV. [b] Electrochemical HOMO-LUMO gap is the energy gap between the HOMO and LUMO levels. [q] Quasi-reversible peak. [irr] Irreversible peak.

Figure S2: UV-vis absorption profiles of half-units **16-18**.



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