

Supporting Information

Alkyl substituted dithienothieno[2,3-d;2',3'-d']benzo[1,2-b:4,5-b']dithiophene, a solution-processable hexathiaheptacene

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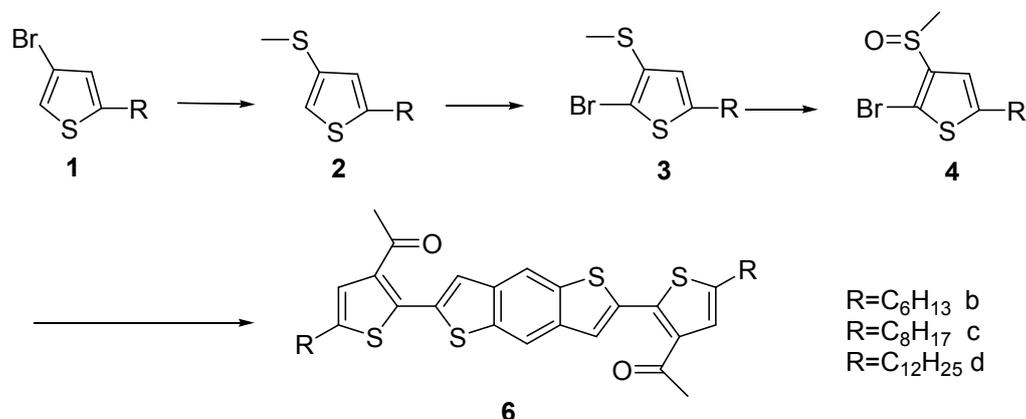
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1. General Methods:

Chemicals were obtained from Fluka, Aldrich, and Acros and used as received if not specified otherwise. ¹H NMR and ¹³C NMR spectra were recorded in deuterated solvents such as CD₂Cl₂, THF on a Bruker DPX 250 spectrometer using the solvent proton or carbon signal as an internal standard. Field desorption (FD) mass spectra were obtained from a VG Instruments ZAB 2-SE-FPD. UV/Vis/NIR spectra were recorded at room temperature on a Perkin-Elmer Lambda 9 spectrophotometer. Fluorescence spectra were recorded on a SPEX-Fluorolog II (212) spectrometer. Cyclic voltammetric measurements were carried out in a conventional three-electrode cell using gold working electrodes of 2 mm diameter, a platinum wire counter electrode, and a Ag/AgCl reference electrode on a computer-controlled PGSTAT12 at room temperature. MO calculations were carried out by DFT method at the B3LYP/6-31G(d) level using the Gaussian03 program package. XRD measurements were recorded using a Siemens D-500 powder diffractometer (Cu-Kα: 0.1541nm) with scan rate of 0.1°/20 s. 2D-WAXS measurements were performed using a custom setup consisting of the Siemens Kristalloflex X-ray source (copper anode X-ray tube, operated at 35 kV/20 mA), Osmic confocal MaxFlux optics, two collimating pinholes (1.0 and 0.5 mm – Owis, Germany) and an antiscattering pinhole (0.7 mm – Owis, Germany). The patterns were recorded on a MAR345 image plate detector (Marresearch, Germany). DSC was measured by Mettler DSC 30 with a heating rate of 10 K/min from 20 °C to 300°C. The optical textures of the hexathiaheptacenes were investigated using a Zeiss microscope with polarizing filters equipped with a Hitachi KP-D50 Colour digital CCD camera. The samples were sandwiched

between two glass slides and then thermally treated on a Linkam hot stage regulated with a Linkam TMS 91 temperature controller.

2. Synthetic Details:



Synthesis of 2-alkyl-4-methylsulfur thiophene (2)

A solution of 2-alkyl-4-bromothiophene (6.2mmol) in THF (10ml) under nitrogen was cooled to -78°C and n-butyllithium (4.25ml, 6.8mmol, 1.6M in hexane) added dropwise. After stirring for a further 1 hour at this temperature, the mixture was transferred via cannula to an ice cool solution of methyldisulfide (6.8mmol) in THF (10ml); the reaction mixture was warmed to room temperature and stirred at this temperature overnight. The mixture was poured into water and extracted with dichloromethane. The organic layer was separated, washed with water, dried (MgSO_4) and concentrated under reduced pressure. Purification by column chromatography (silica gel, hexane) gave compound **2** as colorless oil, yield (50-55%). Compound **2b**, ^1H NMR (250MHz, CD_2Cl_2 , δ ppm): 6.95 (s, 1H), 6.66 (s, 1H), 2.80 (t, 2H), 2.46 (s, 3H), 1.75-1.58(m,2H),1.47-1.24 (m,6H), 0.92(t, 3H). ^{13}C NMR (62.5 MHz, CD_2Cl_2 , δ ppm): 14.19, 22.69, 22.94, 29.11, 30.76, 31.91, 124.71, 132.02, 134.19, 149.72. Compound **2c**, ^1H NMR (250MHz, CD_2Cl_2 , δ ppm): 6.94 (s, 1H), 6.67 (s, 1H), 2.79 (t, 2H), 2.47 (s, 3H), 1.77-1.62(m,2H),1.45-1.23 (m,12H), 0.93(t, 3H). ^{13}C NMR (62.5 MHz, CD_2Cl_2 , δ ppm): 14.53, 22.70, 23.03, 29.45, 30.76, 32.23, 32.01, 124.71, 131.84, 134.08, 149.71. Compound **2d**, ^1H NMR (250MHz, CD_2Cl_2 , δ ppm): 6.94 (s, 1H), 6.68 (s, 1H), 2.82 (t, 2H), 2.48 (s, 3H), 1.82-1.62(m,2H),1.47-1.21 (m,20H), 0.94(t, 3H). ^{13}C NMR (62.5MHz, CD_2Cl_2 , δ ppm): 14.25, 22.69, 23.70, 29.43, 29.76,29.91, 30.03, 30.76, 31.95, 32.30, 124.71, 131.72, 133.97, 149.75.

Synthesis of 2-alkyl-4- methylsulfur-5-bromothiophene (3)

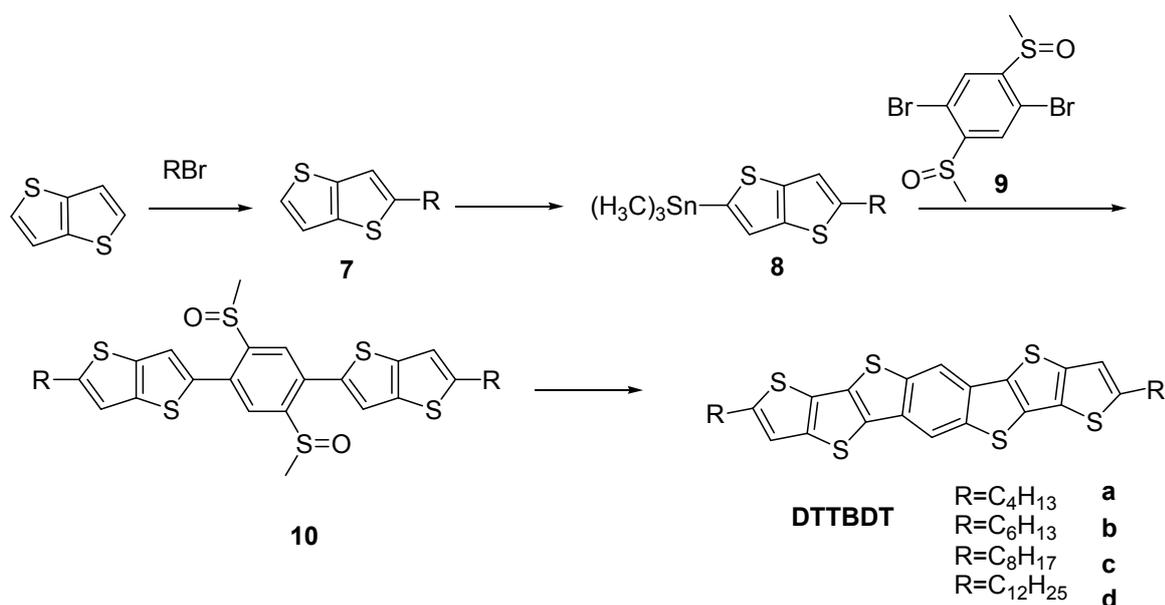
To a stirred solution of compound **2** (5.62mmol) in acetic acid (35ml) and chloroform (35ml) was added N-bromosuccinimide (1.0g, 5.62mmol) portion wise at the temperature between 15°C and 17°C. The resulting mixture was allowed to warm to room temperature overnight, poured into water and extracted with dichloromethane. The organic phase was washed with water, saturated aqueous NaHCO₃, dried (MgSO₄) and concentrated under reduced pressure. Purification by column chromatography (silica gel, hexane) gave compound **3** as colourless oil, yield (60-70%). Compound **3b**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 6.73 (s, 1H), 2.77(t, 2H), 2.46 (s, 3H), 1.74-1.60(m,2H), 1.47-1.28 (m,6H), 0.93(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.21, 20.95, 22.93, 29.01, 30.81, 31.87, 115.80, 127.98, 129.57, 149.24. Compound **3c**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 6.73 (s, 1H), 2.80 (t, 2H), 2.45 (s, 3H), 1.74-1.60(m,2H),1.47-1.25 (m,12H), 0.92(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.22, 20.94, 23.02, 29.31, 29.59, 30.78, 31.49, 32.20, 115.68, 128.10, 129.55, 149.54. Compound **3d**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 6.94 (s, 1H), 6.68 (d, 1H), 2.82 (t, 2H), 2.48 (s, 3H), 1.82-1.62(m,2H),1.47-1.21 (m,20H), 0.94(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.26, 20.94, 23.07, 29.32, 29.64, 29.73, 29.88, 30.03, 30.80, 31.50, 32.31, 115.79, 127.97, 129.57, 149.28.

Synthesis of 2-alkyl-4-methylsulfinyl-5-bromothiophene (4)

To a solution of compound **3** (4.03mmol) in dichloromethane (20ml) at 0°C, was added 3-chloro-perbenzoic acid (1.52g, 4.43mmol, 50-55%) portion wise and the resulting solution allowed to stir at room temperature for 12hrs. The reaction mixture was washed with aqueous Na₂CO₃, dried (MgSO₄). Purification by column chromatography (silica gel, hexane/ethyl acetate) gave compound **4** as a light yellow oil, yield (50-60%). Compound **4b**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 6.81 (s, 1H), 2.91 (s, 3H), 2.87(t, 2H), 1.78-1.65(m,2H), 1.49-1.31 (m,6H), 0.93(t, 3H). ¹³C NMR(62.5MHz, CD₂Cl₂, δ ppm) 14.17, 22.88, 28.95, 30.95, 31.45, 31.80, 111.23, 128.43, 140.78, 152.45. Compound **4c**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 6.68 (s, 1H), 2.79 (s, 3H), 2.75 (t, 2H), 1.68-1.53(m,2H),1.37-1.11 (m,12H), 0.83(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.23, 23.02, 29.28, 29.56, 30.96, 31.48, 32.19, 43.53, 111.23, 128.42, 140.76, 152.46. Compound **4d**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 6.81 (s, 1H), 2.91 (s, 3H), 2.87 (t, 2H), 1.84-1.63(m, 2H),1.51-1.24 (m, 20H), 0.94(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.25, 23.06, 29.29, 29.61, 29.72, 29.85, 30.01, 30.95, 31.49, 32.30, 43.54, 111.24, 128.42, 140.52, 152.47.

Synthesis of 2,2'-bis(2-alkyl-4-methylsulfinylthiophene) benzo[1,2-b;4,5-b']dithiophene (6)

The compound **4** (2.5 mmol) was added to a solution of compound **5** (0.52g, 1.0 mmol) in anhydrous DMF (8ml). The resulting mixture was purged with Ar for 30 min, and $[\text{Pd}(\text{PPh}_3)_4]$ (58mg) was added. The reaction mixture was heated overnight to 80°C. Excess DMF was removed under high vacuum, and the residue was dissolved in ethyl acetate and treated with 10% aqueous KF. The mixture was filtered through a pad of celite. The filtrate was dried over MgSO_4 , filtered, and the solvent removed in vacuo. The crude product was purified by chromatography on silica gel (eluent: hexane/THF) to afford compound **6** as a yellow solid, yield (>85%). Compound **6b**, ^1H NMR (250MHz, CD_2Cl_2 , δ ppm): 8.19 (s, 2H), 7.46 (s, 2H), 7.03 (s, 2H), 2.86 (s, 6H), 2.81(t, 4H), 1.75-1.58(m,4H), 1.41-1.16 (m,12H), 0.82(t, 6H). ^{13}C NMR (62.5MHz, CD_2Cl_2 , δ ppm): 14.21, 22.93, 29.10, 30.89, 31.65, 44.21, 117.28, 123.35, 126.69, 136.01, 137.28, 137.91, 138.52, 142.24, 151.68. FD-MS: m/z: 645.5 (M^+ ,100.0%). HRMS: $\text{C}_{32}\text{H}_{39}\text{O}_2\text{S}_6$, Calculate m/z: 647.1274, Found m/z: 647.1256. Compound **6c**, ^1H NMR (250MHz, CD_2Cl_2 , δ ppm): 8.32 (s, 2H), 7.58 (s, 2H), 7.15 (s, 2H), 2.99 (s, 6H), 2.90 (t, 4H), 1.82-1.69(m,4H),1.49-1.27 (m,20H), 0.93(t, 6H). ^{13}C NMR (62.5MHz, CD_2Cl_2): 14.25, 23.04, 29.48, 29.63, 30.89, 31.69, 32.23, 44.21, 117.30, 123.36, 126.70, 123.36, 126.70, 136.05, 137.18, 137.96, 138.39, 142.24, 151.67. FD-MS: m/z: 701.9 (M^+ ,100.0%), HRMS: $\text{C}_{36}\text{H}_{47}\text{O}_2\text{S}_6$, Calculate m/z: 703.1900, Found m/z: 703.1894. Compound **6d**, ^1H NMR (250MHz, CD_2Cl_2 , δ ppm): 8.19 (s, 2H), 7.46 (s, 2H), 7.02 (s, 2H), 2.86 (s, 6H), 2.81 (t, 4H), 1.73-1.58(m,4H),1.41-1.12(m,36H), 0.80(t, 6H). ^{13}C NMR (62.5MHz, CD_2Cl_2 , δ ppm) 14.26, 23.07, 29.43, 29.67, 29.74, 29.90, 30.02, 30.89, 31.69, 32.31, 44.21, 111.24, 128.42, 140.52, 152.47. FD-MS: m/z: 814.7 (M^+ ,100.0%), HRMS: $\text{C}_{44}\text{H}_{63}\text{O}_2\text{S}_6$, Calculate m/z: 815.3152, Found m/z: 815.3178.



Synthesis of 2-alkylthieno[3,2-b]thiophene (7a-d).

To a cooled (-78 °C) mixture of thieno[3,2-b]thiophene (1.0g, 7.13mmol) in anhydrous THF (20 mL) was added dropwise to a solution of n-BuLi (4 mL, 1.6 M, 6.41mmol) in hexane. After stirring for 1 hour at 0 °C, the mixture was cooled to -78 °C followed by addition of bromoalkane (7.0 mmol). The mixture was slowly warmed to room temperature overnight. Water (50 mL) was added and the mixture was extracted with diethylether. The combined organic fractions were dried over MgSO₄ and purified by chromatography on silica gel (eluent: hexane) to afford compound **7** as colorless liquid, yield (57-71%). Compound **7a**, ¹H NMR (250 MHz, CD₂Cl₂, δ ppm): 7.34 (d, 1H), 7.24 (d, 1H), 7.01 (s, 1H), 2.92 (t, 2H), 1.81-1.66(m,2H), 1.54-1.37 (m,2H), 0.98(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 13.97, 22.54, 31.19, 34.11, 116.52, 119.81, 125.69, 137.69, 139.11, 149.12. Compound **7b**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 7.21 (d, 1H), 7.12 (d, 1H), 6.89 (s, 1H), 2.80 (t, 2H), 1.73-1.53(m,2H), 1.42-1.17 (m,6H), 0.81(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.21, 22.99, 29.10, 31.49, 31.92, 31.94, 116.50, 119.79, 125.05, 137.67, 139.19, 149.19. Compound **7c**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 7.19 (d, 1H), 7.10 (d, 1H), 6.89 (s, 1H), 2.80 (t, 2H), 1.68-1.51(m,2H), 1.36-1.14 (m,10H), 0.80(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.23, 23.04, 29.44, 29.60, 29.69, 31.49, 31.97, 32.24, 116.28, 119.97, 125.58, 137.71, 139.09, 149.47. Compound **7d**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 7.31 (d, 1H), 7.21(d, 1H), 7.02 (s, 1H), 2.92 (t, 2H), 1.82-1.67(m,2H), 1.51-1.21 (m,18H), 0.92(t, 3H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.25, 23.07, 29.43, 29.64, 29.73, 29.76, 29.93, 30.01, 30.04, 31.49, 31.99, 32.30, 116.49, 119.79, 125.65, 137.56, 139.07, 149.18.

Synthesis of trimethyl(5-alkylthieno[3,2-b]thiophen-2-yl)stannane (8).

n-BuLi (1.67mL, 1.6 M, 2.68 mmol) was slowly dropwise to a solution of 2-alkylthieno[3,2-b]thiophene (2.23 mmol) in 10mL anhydrous THF at -78 °C under Ar. The mixture was stirred at this temperature for 30 min and then for 1.5h at room temperature followed, after cooling to -78 °C, by the addition of tributylstannyl chloride (2.79 mmol). After stirring for 6h at room temperature, the reaction was terminated by adding a saturated NH₄Cl aqueous solution. The mixture was extracted with CH₂Cl₂ and dried over MgSO₄. After the removal of solvent, the products were used directly to the next step.

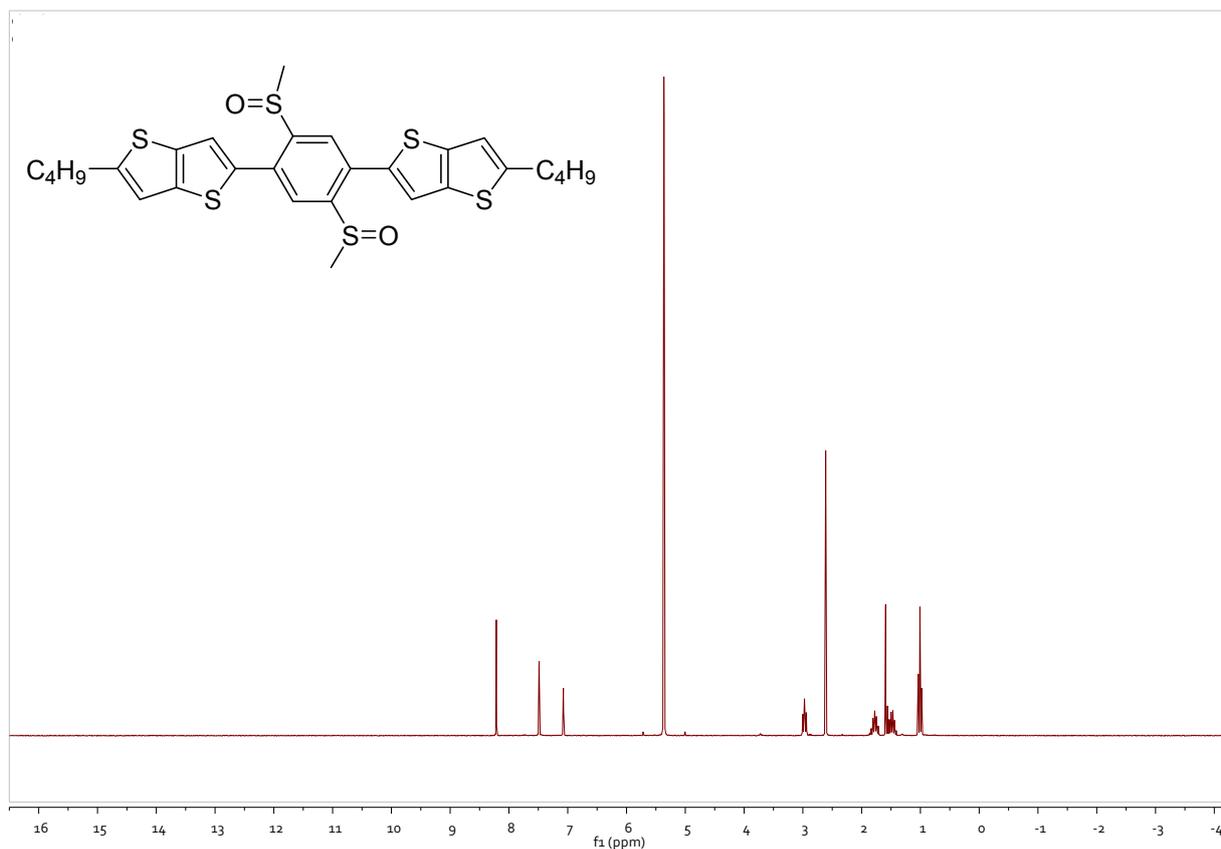
Synthesis of 5,5'-(2,5-bis(methylsulfinyl)-1,4-phenylene)bis(2-alkylthieno[3,2-b]thiophene) (10a-d) .

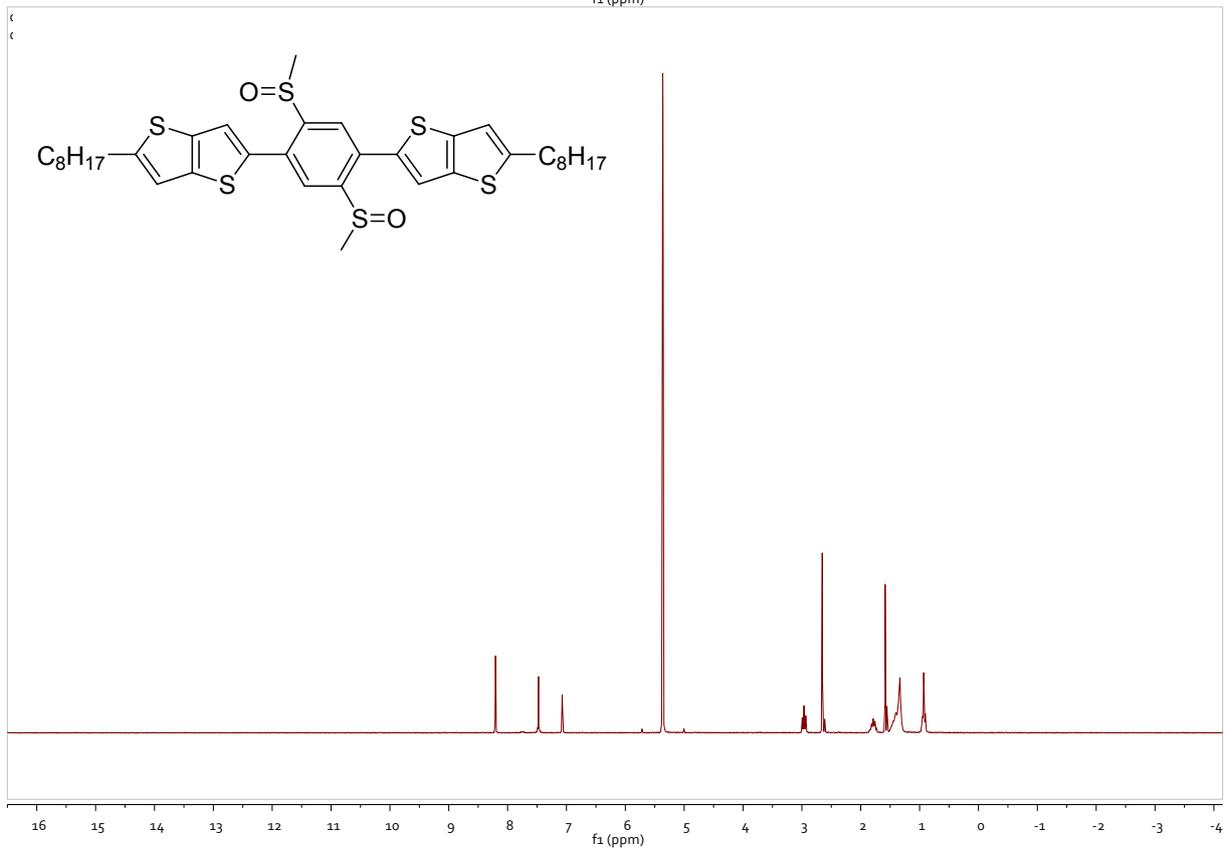
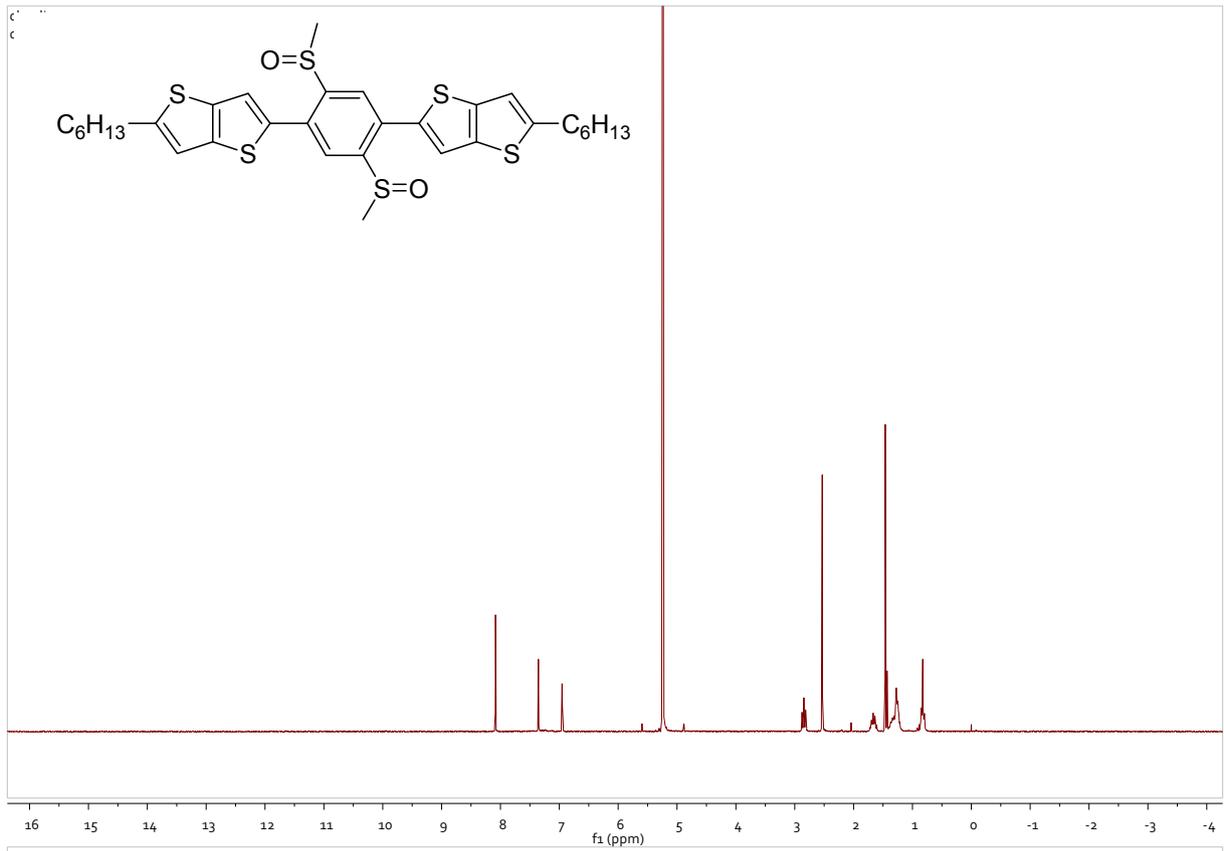
The compound **8** (2.5 mmol) was added to a solution of compound **9** (1.0 mmol) in anhydrous DMF(8ml). The resulting mixture was purged with Ar for 30 min, and [Pd(PPh₃)₄](58 mg) was added. The reaction mixture was heated overnight to 80°C. Excess DMF was removed under high vacuum, and the residue was dissolved in ethyl acetate and treated with 10% aqueous KF. The mixture was filtered through a pad of celite. The filtrate was dried over MgSO₄, filtered, and the solvent removed in vacuo. The crude product was purified by chromatography on silica gel (eluent: hexane/THF) to afford compound **10** as a yellow solid, yield 80-86%. **Compound 10a.** ¹H NMR(250MHz, CD₂Cl₂, δ ppm): 8.17 (s, 2H), 7.44 (s, 2H), 7.03 (s, 2H), 2.92 (t, 4H), 2.56(s, 6H), 1.83-1.66(m,4H), 1.54-1.36 (m,4H),0.99(t, 6H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 13.98, 22.56, 31.28, 34.03, 42.26, 116.70, 121.29, 126.42, 132.99, 137.61, 138.12, 140.98, 148.06, 150.70. FD-MS: m/z: 591.0 (M⁺,100.0%). HRMS: C₂₈H₃₁O₂S₆, Calculate m/z: 591.0648, Found m/z: 591.0654. **Compound 10b.** ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 8.08 (s, 2H), 7.36 (s, 2H), 6.94 (s, 2H), 2.85 (t, 4H), 2.49(s, 6H), 1.73-1.58(m,4H),1.42-1.16 (m,12H),0.82(t, 6H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm):14.21, 22.96, 29.13, 31.55, 31.93, 42.38, 116.66, 121.31, 126.42, 132.90, 137.40, 138.29, 140.74, 148.21, 150.86. FD-MS: m/z: 645.3 (M⁺,100.0%). HRMS: C₃₂H₃₉O₂S₆, Calculate m/z: 647.1274, Found m/z: 647.1264. **Compound 10c.** ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 8.08 (s, 2H), 7.35 (s, 2H), 6.94 (s, 2H), 2.85 (t,4H), 2.53(s, 6H), 1.77-1.58(m,4H),1.39-1.12 (m,20H),0.83(t, 6H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.24, 23.04, 29.47, 29.68, 31.54, 31.94, 32.23, 42.32, 116.58, 121.16, 126.29, 132.96, 137.31, 138.20, 140.98, 148.12, 150.84. FD-MS: m/z: 701.4 (M⁺,100.0%), HRMS: C₃₆H₄₇O₂S₆, Calculate m/z: 703.1900, Found m/z: 703.1887. **Compound 10d.** ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 8.17 (s, 2H), 7.44 (s, 2H), 7.02 (s, 2H), 2.90 (t, 4H), 2.56(s, 6H), 1.85-1.63(m, 4H),1.49-1.19 (m, 36H), 0.88(t, 6H). ¹³C NMR (62.5MHz, CD₂Cl₂, δ ppm): 14.30, 23.07, 29.47, 29.73, 30.01, 30.05, 31.55, 31.95, 32.31, 42.41, 116.58, 121.16, 126.29, 132.96, 137.31, 138.20, 140.98, 148.12, 150.84. FD-MS: m/z: 815.0 (M⁺,100.0%), HRMS: C₄₄H₆₃O₂S₆, Calculate m/z: 815.3152, Found m/z: 815.3127

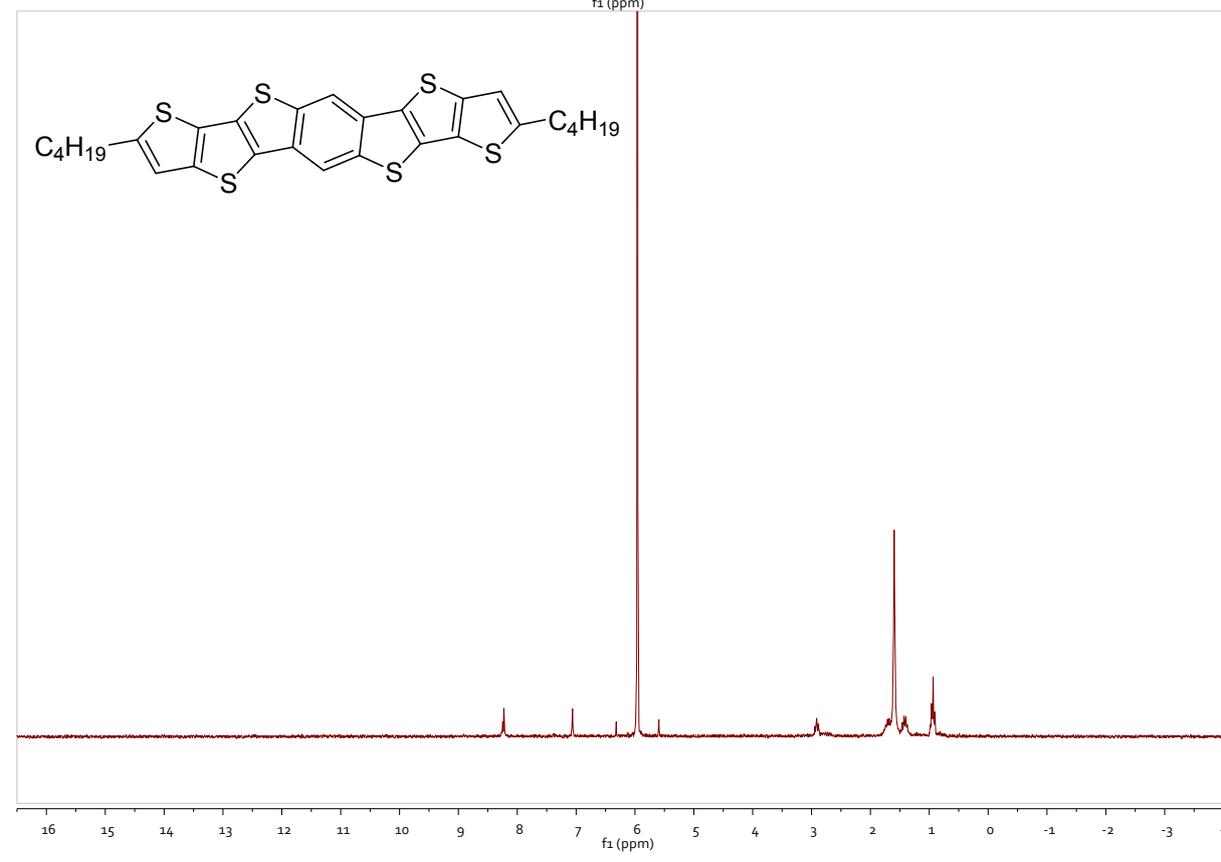
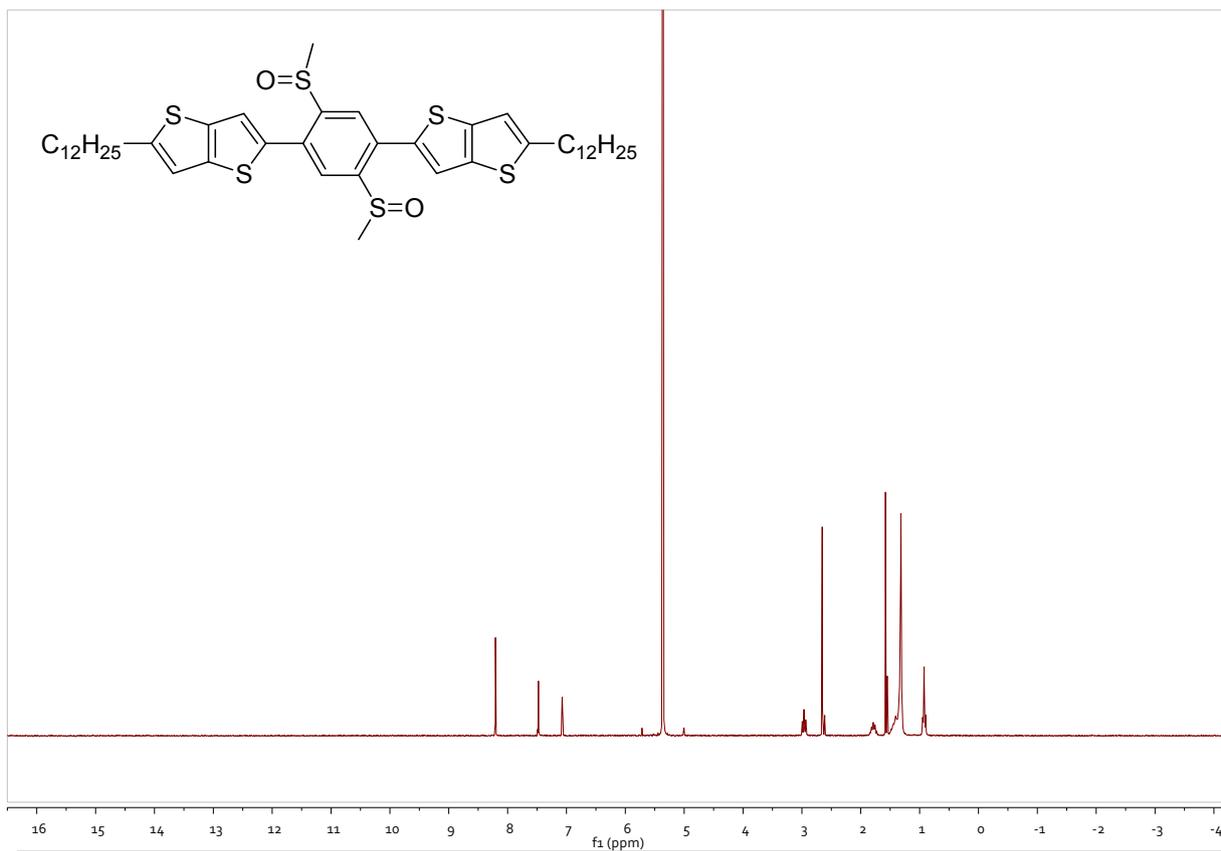
Synthesis of heteroheptacenes dithienothiophene[2,3-d;2',3'-d']benzo[1,2-b:4,5-b']dithiophene (DTTBDT a-d)

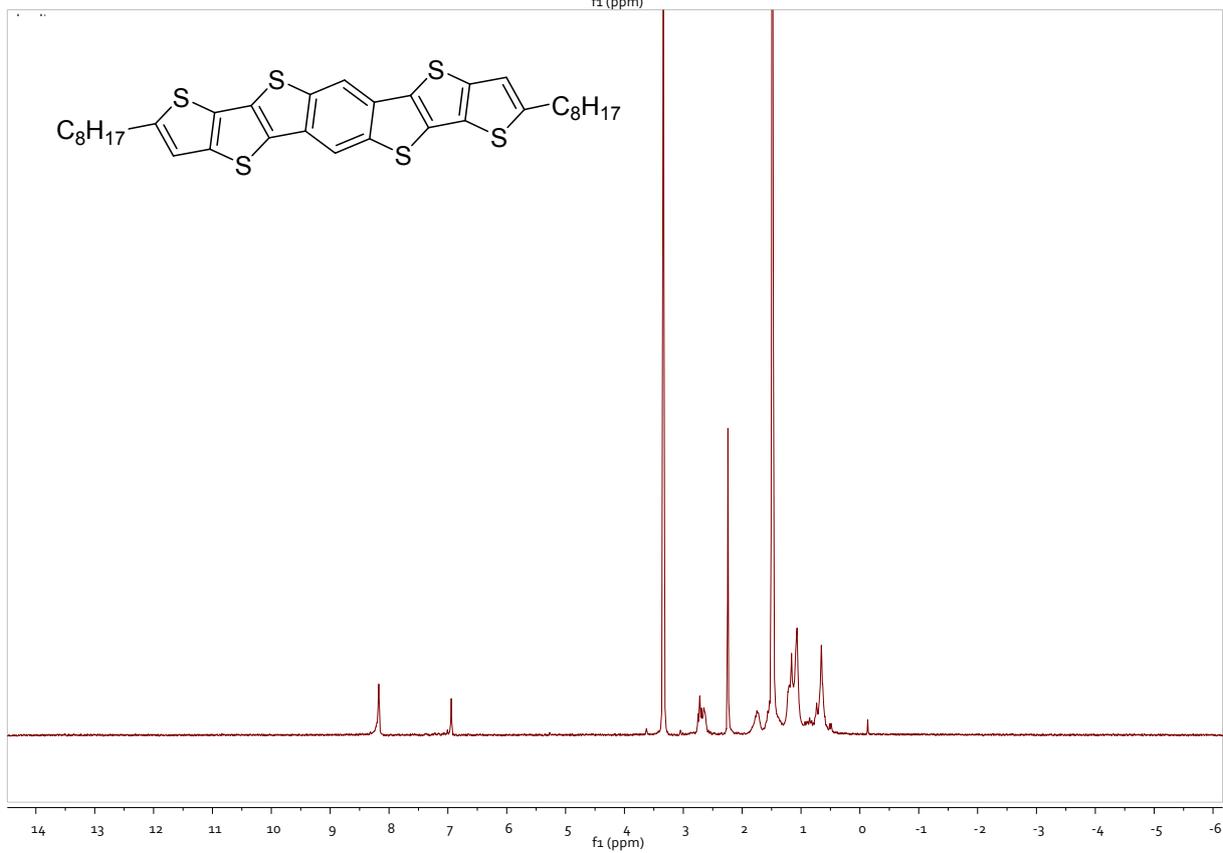
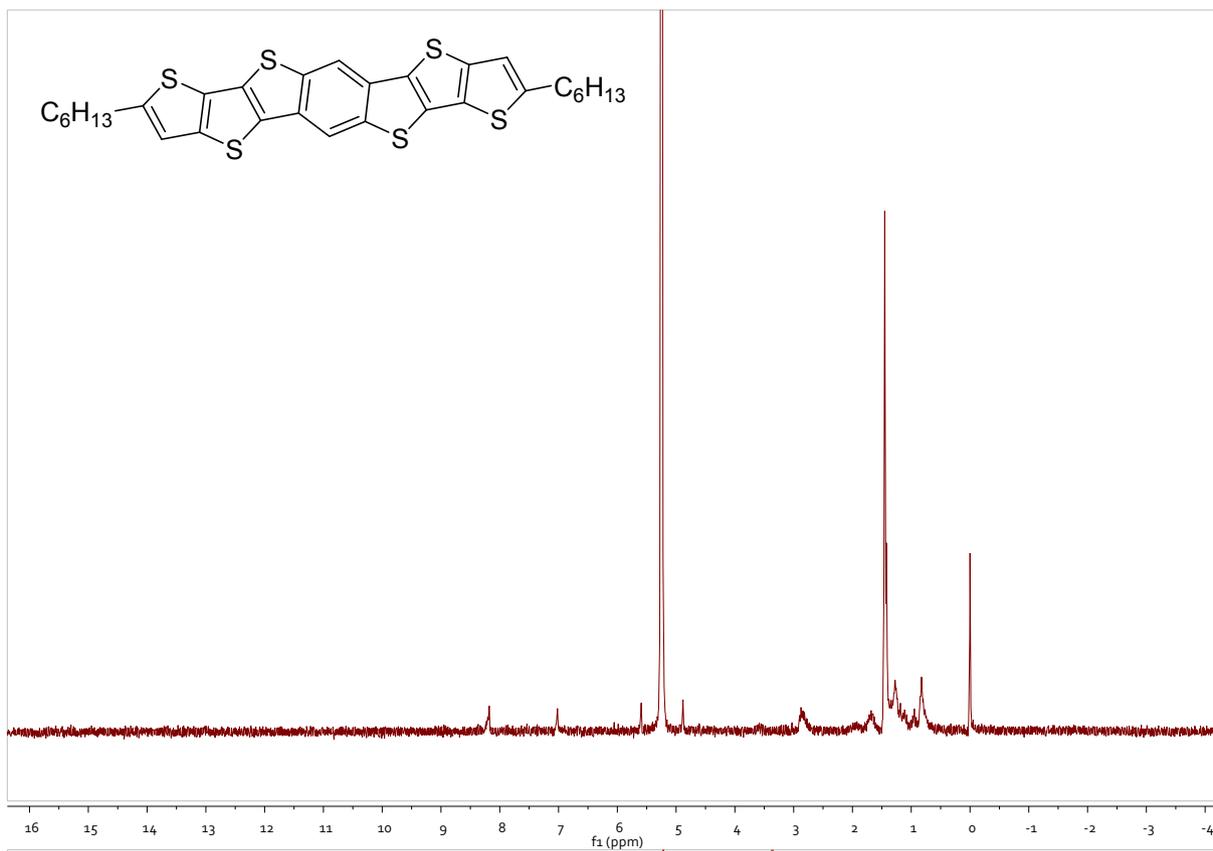
A 10-mL round-bottomed flask was filled with compound **10** (200 mg), phosphorus pentoxide (28 mg, 0.2mmol), and trifluoromethanesulfonic acid (6 ml). The mixture was stirred for 72h at room temperature to give a dark-blue solution, which was then poured into ice-water (100 ml). The yellow precipitate was collected by suction filtration and dried under

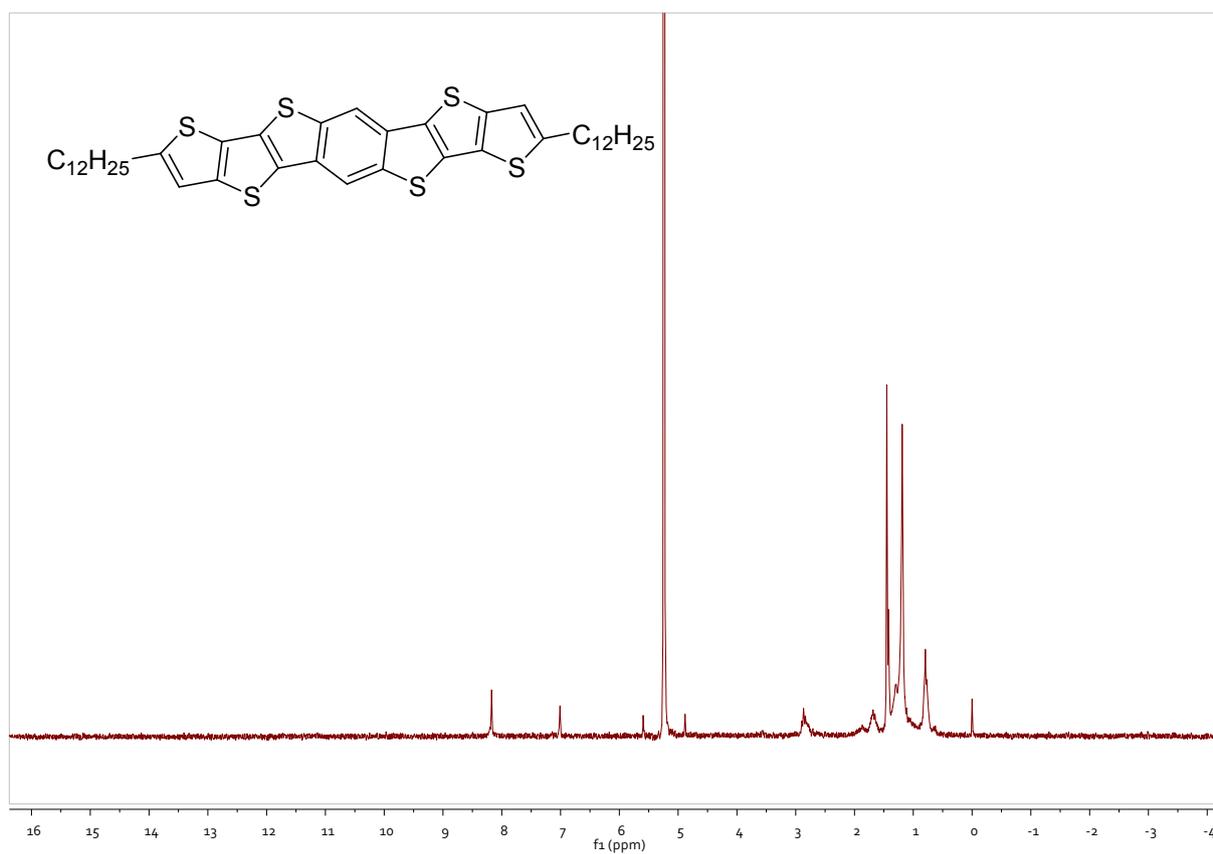
vacuum. The structure of this compound, which was insoluble in apolar organic solvents, was assumed to be the sulfonium salt. Demethylation of the solid was achieved by heating to reflux in pyridine (30ml) for 12h. When the suspension was cooled to room temperature, a large volume of CH₂Cl₂ was added to extract the product. Product **DTTBDT** was thus obtained as a yellow powder after chromatography on silica gel with THF/hexane as the eluent, followed by recrystallization in hexane/THF twice, yield (20-30%). The structures of products have been characterized by ¹H NMR, FD-MS and HRMS. ¹³C NMR spectra could not be obtained due to the relatively low solubility. **DTTBDT a**, ¹H NMR (250MHz, C₂D₂Cl₄, δ ppm): 8.22 (s, 2H), 7.06 (s, 2H), 2.90 (t, 4H), 1.83-1.50(m,4H), 1.48-1.34 (m,4H),0.98(t, 6H). FD-MS: m/z: 526.1 (M⁺,100.0%). HRMS: C₂₆H₂₂NaS₆, Calculate m/z: 548.9944, Found m/z: 548.9938. **DTTBDT b**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 8.17 (s, 2H), 7.02 (s, 2H), 2.85 (t, 4H), 1.82-1.55(m,4H), 1.46-1.18 (m,12H),0.85(t, 6H). FD-MS: m/z: 582.0 (M⁺,100.0%). HRMS: C₃₀H₃₁S₆, Calculate m/z: 583.0750, Found m/z: 583.0725. **DTTBDT c**, ¹H NMR (250MHz, THF, δ ppm): 8.17 (s, 2H), 6.94(s, 2H), 2.83 (t, 4H), 1.82-1.80(m, 4H), 1.39-1.10 (m,20H),0.86(t, 6H). FD-MS: m/z: 635.7 (M⁺,100.0%). HRMS: C₃₄H₃₉S₆, Calculate m/z: 639.1376, Found m/z: 639.1362. **DTTBDT d**, ¹H NMR (250MHz, CD₂Cl₂, δ ppm): 8.17 (s, 2H), 7.02 (s, 2H), 2.85 (t, 4H), 1.77-1.53(m,4H), 1.55-1.11 (m,36H),0.80(t, 6H). FD-MS: m/z:750.1 (M⁺,100.0%). HRMS: C₄₂H₅₅S₆, Calculate m/z: 751.2628, Found m/z: 751.2620.











3. UV/Vis absorption spectra

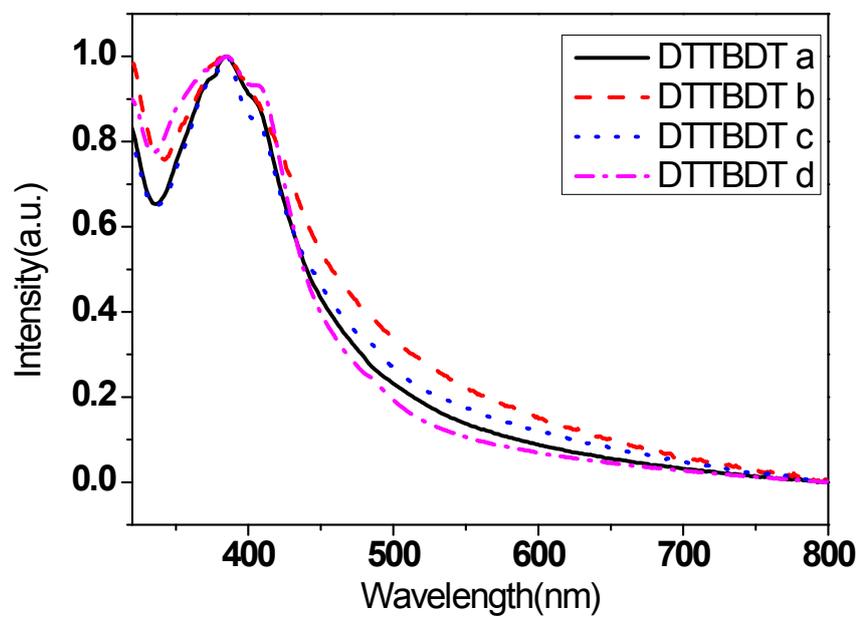


Figure S1. Normalized UV/Vis absorption spectra of **DTTBDT** films.

4. Cyclic voltammetry (CV) measurements:

CV measurements were performed on a computer-controlled PGSTAT12 with a three-electrode cell in a solution of 0.1 M tetrabutylammonium hexafluorophosphate (Bu_4NPF_6) dissolved in THF at a scan rate of 50 mV/s. A Pt wire was used as the counter electrode and an Ag/AgCl electrode was used as the reference electrode. HOMO energies was estimated by the empirical equation $\text{HOMO} = - (E_{\text{ox}} + 4.4) \text{ eV}$.

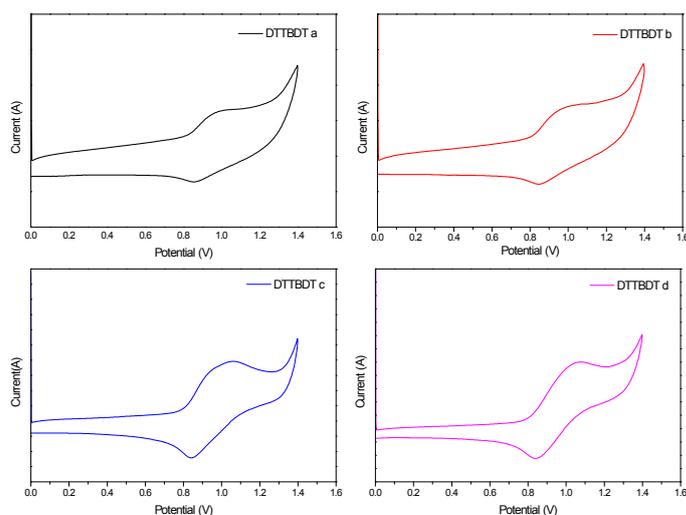


Figure S2. Cyclic voltammograms (CV) of DTTBDT a-d.

5. Thermogravimetric analysis (TGA)

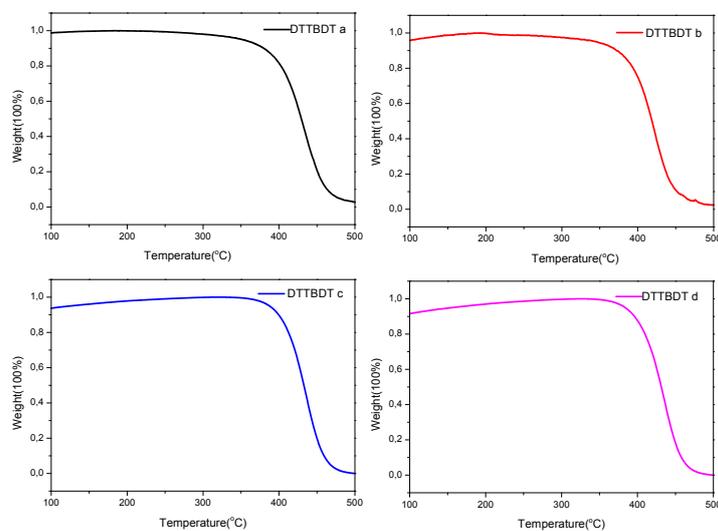


Figure S3. TG thermograms of DTTBDT a-d under nitrogen at a heating rate of 10 K/min.

6. Device Fabrication

FETs based on DTTBDT were fabricated by solution processing, except for DTTBDT a, due to its poor solubility. All FETs were prepared employing the bottom-gate, bottom-contact architecture. The heavily doped silicon wafers covered with about 200 nm thick SiO₂ dielectric with Au electrodes are used as the substrates. For the solution-processing of the semiconductor, 2 mg/mL chloroform solution was deposited by drop-casting on FET substrates, followed by annealing at 80 °C for 40 min. All electrical measurements were performed in a glovebox under nitrogen atmosphere. The device characteristics were measured using a Keithley 4200-SCS.

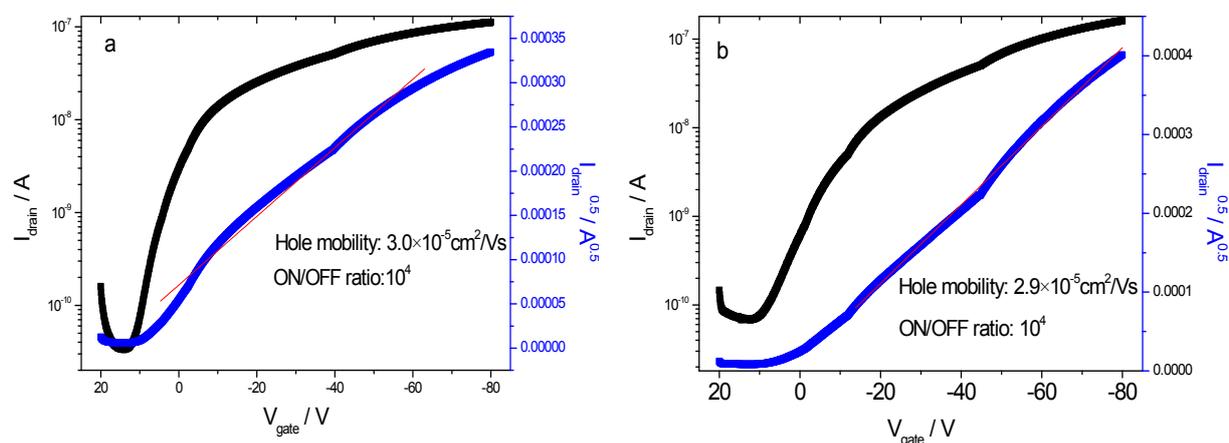


Figure S4. FET transfer characteristics of DTTBDT b (a) and d (b) at gate voltage of -80V.