

Supporting Information

Donor-acceptor π -conjugated polymers based on terthiophene-3, 4-dicarboxylate, dithienopyrrolbenzothiadiazole and thieno[3,4-*c*]pyrrole-4,6-dione units and their hole mobility

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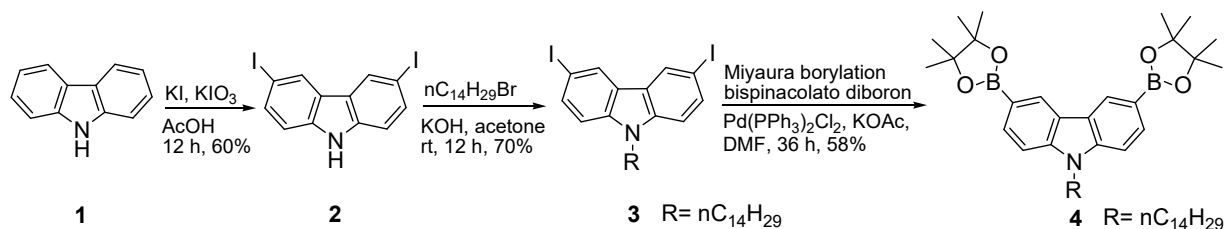
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Synthesis of monomers and intermediate compounds

Synthesis of 9-*n*-tetradecyl-3,6-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9*H*-carbazole (**4**)

To synthesize compound **4**, compound **1** was converted into diiodocarbazole **2** by reacting with an iodine-iodate mixture. Compound **2** was alkylated in the presence of a base to get compound **3**. The alkylated compound **3** was further treated with bis(pinacolato)diborane under Miyaura borylation reaction conditions to get desired compound **4** (Scheme 1).



Scheme 1 Synthesis of 9-*n*-tetradecyl-3,6-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9*H*-carbazole (**4**)

3, 6-Diiodo-9*H*-carbazole (**2**)¹

To the solution of carbazole (**1**) (3.0 g, 17 mmol) in 10 mL glacial acetic acid, the solution of KI (3.9 g, 23 mmol) and KIO₃ (5.68 g, 26 mmol) in 10 mL water was added. The reaction mixture was then heated to 80 °C for 48 hours. After completion of the reaction, the reaction mixture was cooled, filtered and washed with saturated sodium carbonate to yield the crude product. The crude product was purified by crystallization in methanol to yield 5.15 g (60%) of 3,6-diiodo-9*H*-carbazole (**2**) as a white solid.

¹H NMR (400 MHz CDCl₃): δ 7.22-7.24 (d, 2H), 7.68-7.71 (dd, 2H), 8.13 (s, 1H), 8.34 (s, 1H).

3, 6-Diiodo-9-*n*-tetradecyl-9*H*-carbazole (**3**)¹

In a 100 mL round bottom flask, KOH pellets (1.3 g, 23 mmol) were dissolved in 30 mL of acetone by stirring at room temperature for 15 minutes. To this solution, 1 g (6 mmol) 3,6-diiodo-9*H*-carbazole (**2**) was added, and the content was stirred for 30 minutes at room temperature. After that, 2.15 g (7 mmol) of *n*-tetradecyl bromide was added to the reaction mixture dropwise. The reaction mixture was stirred for 12 hours at room temperature. Excess of solvent was evaporated

under vacuum, and the crude product was repeatedly washed with water, extracted in DCM, dried and concentrated to yield a white solid product, 3,6-diiodo-9-*n*-tetradecyl-9*H*-carbazole (**7**), 1.02 g. (70%).

¹H NMR (400 MHz CDCl₃): δ 0.88-0.91 (t, 3H), 1.23-1.31 (m, 22H), 1.80-1.81 (m, 2H), 4.20-4.24 (t, 2H), 7.17-7.19 (d, 2H), 7.71-7.73 (d, 2H), 8.33 (s, 2H). ¹³C NMR (100 MHz CDCl₃): δ 14.1, 22.7, 27.2, 28.8, 29.3, 29.4, 29.4, 29.5, 29.6, 29.6, 29.7, 31.9, 43.2, 81.6, 110.9, 123.9, 129.3, 134.4, 139.4.

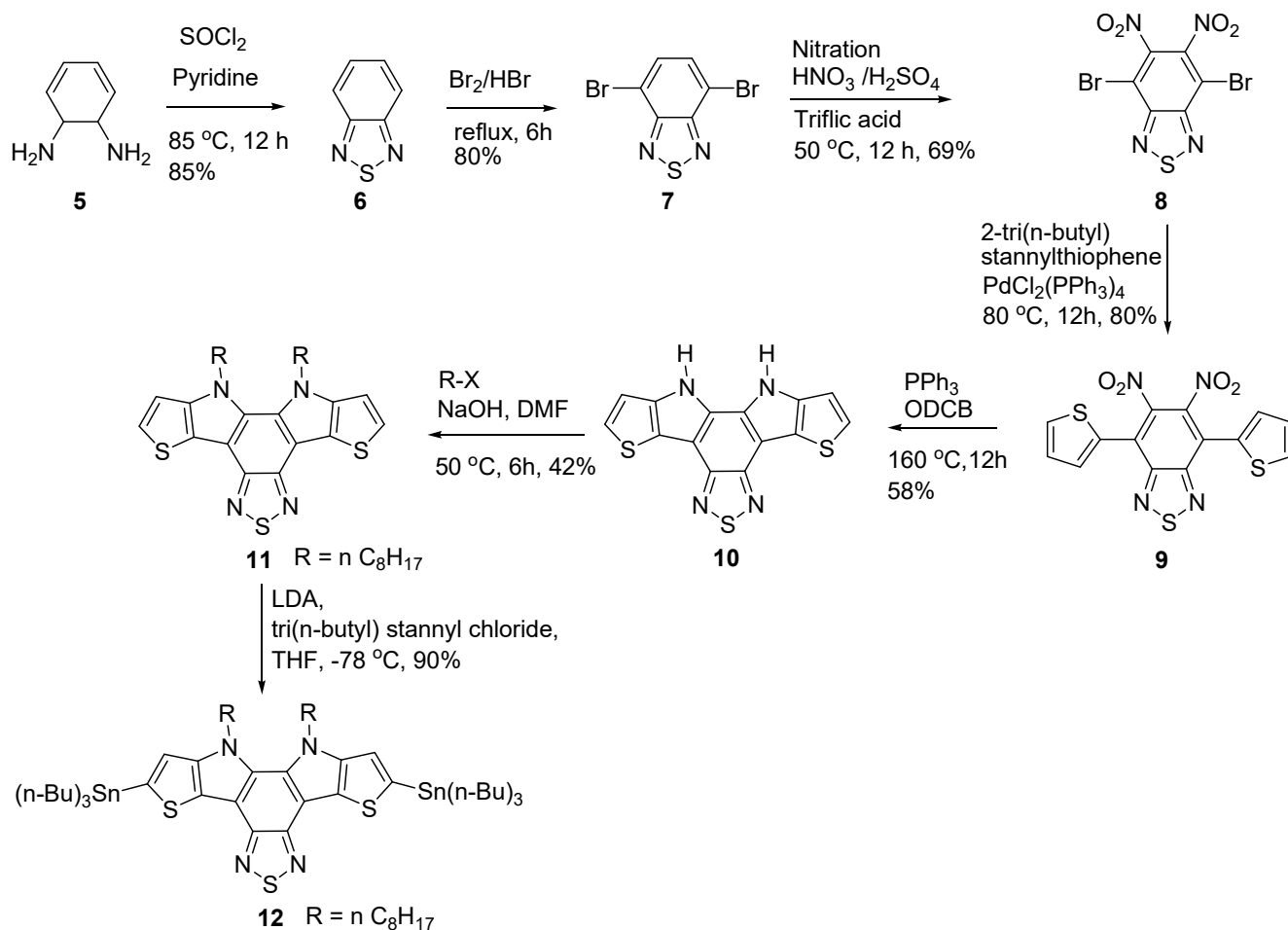
9-*n*-Tetradecyl-3, 6-bis(4, 4, 5, 5-tetramethyl-1, 3, 2-dioxaborolan-2-yl)-9*H*-carbazole (4**)¹**

In a dry three-neck round bottom flask attached with condenser under nitrogen atmosphere, 0.8 g (13 mmol) of 3,6-diiodo-9-*n*-tetradecyl-9*H*-carbazole (**3**) was taken. To this, 20 mL of dry DMF was added, and the solution was stirred to dissolve the compound. Bis(pinacolato)diboron (1.0 g, 39 mmol, 3 equivalents) and potassium acetate (0.50 g, 5.2 mmol, 4 equivalents) were added to the reaction mixture, and thereafter the reaction mixture was purged with nitrogen for 15 minutes. Pd(PPh₃)₄Cl₂ (91 mg, 1.3 mmol, 0.1 equivalents) was added to the reaction mixture and again purged with nitrogen for 15 minutes. The reaction mixture was heated at 120 °C for 36 hours under a nitrogen atmosphere. The completion of the reaction was confirmed by the disappearance of starting material on TLC. The reaction mixture was diluted with water, and then the crude product was extracted in DCM. The organic layer was washed with brine and dried over sodium sulfate, concentrated under vacuum to yield the crude product. Further purification was carried out by silica gel column chromatography (10% ethyl acetate in pet ether solvent system). The product was obtained as a colourless oil (**4**) with 58% (0.46 g) of yield.

¹H NMR (CDCl₃): δ 0.87-0.89 (t, 6H), 1.23-1.26 (m, 22H), 1.40 (s, 24H), 1.82-1.88 (m, 3H), 4.30-4.33 (t, 2H), 7.40-7.42 (d, 2H), 7.90- 7.93 (d, 2H), 8.68 (s, 2H).

Synthesis of 10,11-dioctyl-2,8-bis(tributylstannyl)-10,11-dihydro-[1,2,5]thiadiazolo[3,4-*e*]thieno[2',3':4,5]pyrrolo[3,2-*g*]thieno[3,2-*b*]indole (12)

Synthesis of compound **10** was carried out according to the method reported in the literature (Scheme 2).² *ortho*-Phenylenediamine (**5**) was reacted with thionyl chloride to yield benzothiadiazole **6**, which further brominated by using bromine in hydrobromic acid to get dibromo benzothiadiazole **7**. Compound **7** was nitrated by nitrating mixture (HNO₃/H₂SO₄:3/2) in triflic acid, giving product dinitro dibromo benzothiadiazole **8** in 69% yield. This compound **8** was reacted with 2-tributylstannylthiophene under nitrogen atmosphere in dry THF in presence of tetra(triphenyl)bispalladium dichloride as a catalyst to yield 5,6-dinitro-4,7-di(thiophen-2-yl)benzo[*c*]-2,1,3-thiadiazole (**9**) in 80%. Compound **9** was cyclized under Cadogen reaction conditions to form dithienopyrrolobenzothiadiazole **10** in a 58% yield.



Scheme 4 Synthesis of compound **12**

Compound **10** was alkylated by *n*-octyl bromide in DMF using NaOH as a base to form compound **11** with a yield of 42%. Compound **11** was first reacted with LDA at -78 °C and then reacted with tri-*n*-butylstannyl chloride to yield compound **12** in 90% (Scheme 4).³

Synthesis of benzo[*c*]-2,1,3-thiadiazole (**6**)⁴

o-Phenylene diamine (**5**) (5 g, 46 mmol) was taken in two-neck round bottom flask and 10 mL dichloroethane (DCE) and triethylamine (TEA) (19.3 mL, 139 mmol) were added sequentially. Slow dropwise addition of a solution of thionyl chloride (13.5 mL, 185.2 mmol) in DCE was carried out over 45 minutes using a pressure equalizer dropping funnel. The reaction mixture was refluxed at 85°C for 12 hours. Then it was cooled to room temperature, and water was added to quench excess thionyl chloride. The product was extracted in dichloromethane (DCM), the organic layer was dried over Na₂SO₄, and the solvent was evaporated under reduced pressure. The crude product thus obtained was subjected to steam distillation to obtain pure product as white crystals. The product was obtained in 85% yield (5.4g).

¹H NMR (CDCl₃): δ 8.00-8.05 (m, 2H), 7.59-7.63 (m, 2H).

Synthesis of 4, 7-dibromobenzo[*c*]-2,1,3-thiadiazole (**7**)⁴

A solution of bromine (7.06 g, 2.3 mL, 44 mmol) in HBr (20 mL) was added dropwise using pressure equalizer dropping funnel into the solution of compound **6** (2.0 g, 14.7 mmol) in 47 % HBr (30 mL) and then the reaction mixture was refluxed for 6 hours. After cooling to room temperature, NaHSO₃ solution was added to quench excess bromine. The crude product obtained was filtered and washed with water and then with cold diethyl ether carefully and dried. It was purified by silica gel column chromatography (10% ethyl acetate in petroleum ether). The product was obtained as white crystals in 80% yield (3.46g).

¹H NMR (CDCl₃): δ 7.75 (s, 2H).

Synthesis of 4,7-dibromo-5,6-dinitrobenzo[*c*]-2,1,3-thiadiazole (**8**)⁵

Triflic acid (trifluoromethane sulfonic acid) (11.1 mL, 128.6 mmol) was taken in a two neck round bottom flask and cooled in an ice-salt bath under a nitrogen atmosphere. To this fuming HNO₃ (2.5 mL conc. HNO₃ + 0.8 mL conc. H₂SO₄) was added, and this nitrating mixture was cooled for

15 minutes. Compound **7** (3.0 g, 10.2 mmol) was added into the nitrating mixture portion-wise over 30 minutes. The temperature was then raised to 50 °C and stirred overnight. Then the reaction mixture was poured slowly on crushed ice and neutralized with 2N NaOH. The resultant mixture was filtered and washed with water, dried and recrystallized from 95% EtOH. Crystals of compound **8** obtained in 69% (2.72 g) yield.

¹H NMR (400 MHz, CDCl₃): No proton signals observed. ¹³C NMR (100 MHz, CDCl₃): 151.5, 143.6, 111.3.

Synthesis of 5, 6-dinitro-4, 7-di(thiophen-2-yl)benzo[*c*]-2,1,3-thiadiazole (9**)⁶**

Compound **8** (0.8 g, 1.65 mmol) was dissolved in dry THF (20mL) under nitrogen atmosphere in two-neck round bottom flask and Pd(PPh₃)₂Cl₂ (0.058 g, 0.083 mmol) was added. Then 2-tributylstannylthiophene (1.02 mL, 4.13 mmol) was added *via* syringe and the reaction mixture was heated at 65 °C for 12 hours. The reaction was monitored by TLC. On completion of the reaction, the solvent was evaporated under reduced pressure and the reaction mixture was washed with water and extracted in DCM, which was later dried over anhydrous Na₂SO₄ and concentrated to yield the crude product. The crude product was further purified over silica gel column chromatography (20% ethyl acetate in petroleum ether). A bright red solid compound was obtained. Yield: 80% yield (0.65g).

¹H NMR (CDCl₃): δ 7.76-7.77 (dd, 2H), 7.53-7.54 (dd, 2H), 7.25-7.33 (m, 2H).

Synthesis of dithienopyrrolobenzothiadiazole (DTPBT) (10**)⁷**

Compound **9** (0.3g, 6.12 mmol) was dissolved in *o*-dichlorobenzene (5 mL) in a two neck round bottom flask under nitrogen atmosphere, and triphenylphosphine (1.604 g, 61.2 mmol) was added. The reaction mixture was then heated to 160 °C for 12 hours, cooled and purified by silica gel column chromatography (40% ethyl acetate in petroleum ether). The product was obtained as a mixture with triphenylphosphine oxide as orange solid (0.260g), and this slightly impure product was directly used for the next reaction.

¹H NMR (DMSO D₆): δ 11.90 (s, 2H), 7.61-7.63 (d, 2H), 7.43-7.44 (d, 2H).

Synthesis of 10,11-dioctyl-10,11-dihydro-[1,2,5]thiadiazolo[3,4-*e*]thieno[2',3':4,5]pyrrolo[3,2-*g*]thieno[3,2-*b*]indole (11)³

In a 100 mL round bottom flask, compound **10** (0.260 g, 0.79 mmol) was taken, followed by the addition of octyl bromide (0.69 mL, 3.98 mmol). To this reaction mixture, NaOH (0.316 g, 7.9 mmol) in DMF/water (2 mL) was added. The reaction mixture was stirred at 50°C for 6 hours. Then the excess water was added to the reaction mixture, and the product was extracted in DCM. The organic layer was dried over Na₂SO₄ and then evaporated under reduced pressure to get crude product which was purified by silica gel column chromatography (5% ethyl acetate in petroleum ether). The solid product was obtained in 42% yield (0.214 g).

¹H NMR (CDCl₃): δ 7.45-7.46 (d, 2H), 7.20-7.22 (d, 2H), 4.50-4.54 (t, 4H), 1.81-1.87 (m, 4H), 1.08-1.27 (m, 20H), 0.80-0.84 (m, 6H).

Synthesis of 10,11-dioctyl-2,8-bis(tributylstannyl)-10,11-dihydro-[1,2,5]thiadiazolo[3,4-*e*]thieno[2',3':4,5]pyrrolo[3,2-*g*]thieno[3,2-*b*]indole (12)³

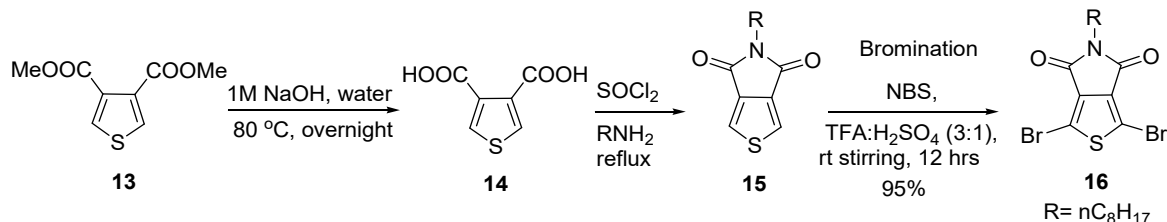
10, 11-Dioctyl-10, 11-dihydro-[1, 2, 5]thiadiazolo[3, 4-*e*]thieno[2', 3':4, 5]pyrrolo[3, 2-*g*]thieno[3, 2-*b*]indole (**11**) (0.4 g) (7 mmol) (1 equivalent) was taken in dry three neck round bottom flask attached with septum. 10 mL of dry THF was added to it and resultant solution was stirred at -78°C under nitrogen atmosphere. LDA (0.224 g) (21 mmol) (3 equivalents) was added to the reaction mixture and reaction mixture stirred at -78 °C for 3 hours. Tributyltin chloride was added to reaction mixture and reaction mixture was again stirred at -78 °C for 1 hour. Thereafter, the reaction mixture was allowed to reach at room temperature in overnight time. The reaction mixture was quenched with water addition. The organic contents was extracted in diethyl ether, ether layer was washed with brine and dried over sodium sulfate, concentrated to yield product 10, 11-dioctyl-2, 8-bis(tributylstannyl)-10, 11-dihydro-[1, 2, 5]thiadiazolo[3, 4-*e*]thieno[2', 3':4, 5]pyrrolo[3, 2-*g*]thieno[3, 2-*b*]indole (**12**) in 90% yield (0.738 g).

¹H NMR (CDCl₃): δ 0.77-0.83 (6H), 0.91-0.95 (18H), 1.10-1.69 (60H), 4.52-4.59 (4H), 7.17 (2H).

Synthesis of 1,3-dibromo-5-octylthieno[3,4-*c*]pyrrole-4,6-dione (16)

TPD was synthesized from dimethyl 3,4-thiophene dicarboxylate (**13**) in three steps. The first step involved hydrolysis of dimethyl 3,4-thiophene dicarboxylate in an aqueous NaOH solution to yield

thiophene diacid (**14**). Thiophene 3,4-dicarboxylic diacid was then converted into TPD through a one-pot reaction that included first its reaction with thionyl chloride to form acid chloride, which was further reacted with octyl amine to yield *n*-octylthieno[3,4-*c*]pyrrole-4,6-dione (TPD) (**15**).



Scheme 4 Synthesis of 1,3-dibromo-5-octylthieno[3,4-*c*]pyrrole-4,6-dione (**16**)

Synthesis of 5-*n*-octylthieno[3,4-*c*]pyrrole-4,6-dione (**15**)

Dimethyl thiophene-3,4-dicarboxylate (**13**) (4.0 g, 20 mmol) was taken in a round-bottom flask of 100 mL capacity. To this 20 mL of 1M solution of NaOH was added, and the mixture was stirred at 80 °C overnight. The solution was then acidified with HCl to pH = 3 and extracted with ethyl acetate, dried over sodium sulfate and concentrated to obtain the yellow solid compound (**14**) (3.44 g). The yield obtained was 80%. The obtained product was dissolved in toluene (50 mL), and 15 mL of thionyl chloride was added to it. The reaction mixture was refluxed for 5 hours. Thereafter, excess of thionyl chloride was distilled off and 3.3 mL of *n*-octyl amine was added to the reaction mixture. The reaction mixture was heated to 200 °C for 12 hours. The crude product was washed with water and brine, extracted in ethyl acetate. Dried over sodium sulfate and concentrated under reduced pressure. Further purification was performed by column chromatography on silica gel using 10% ethyl acetate in petroleum ether as an eluent to obtain white solid product 4.5 g (85% yield).

$^1\text{H NMR}$ (CDCl_3): δ 7.8 (s, 2H), 3.62 (t, 2H), 1.67-1.60 (m, 2H), 1.30 (m, 10H), 0.86 (t, 3H).

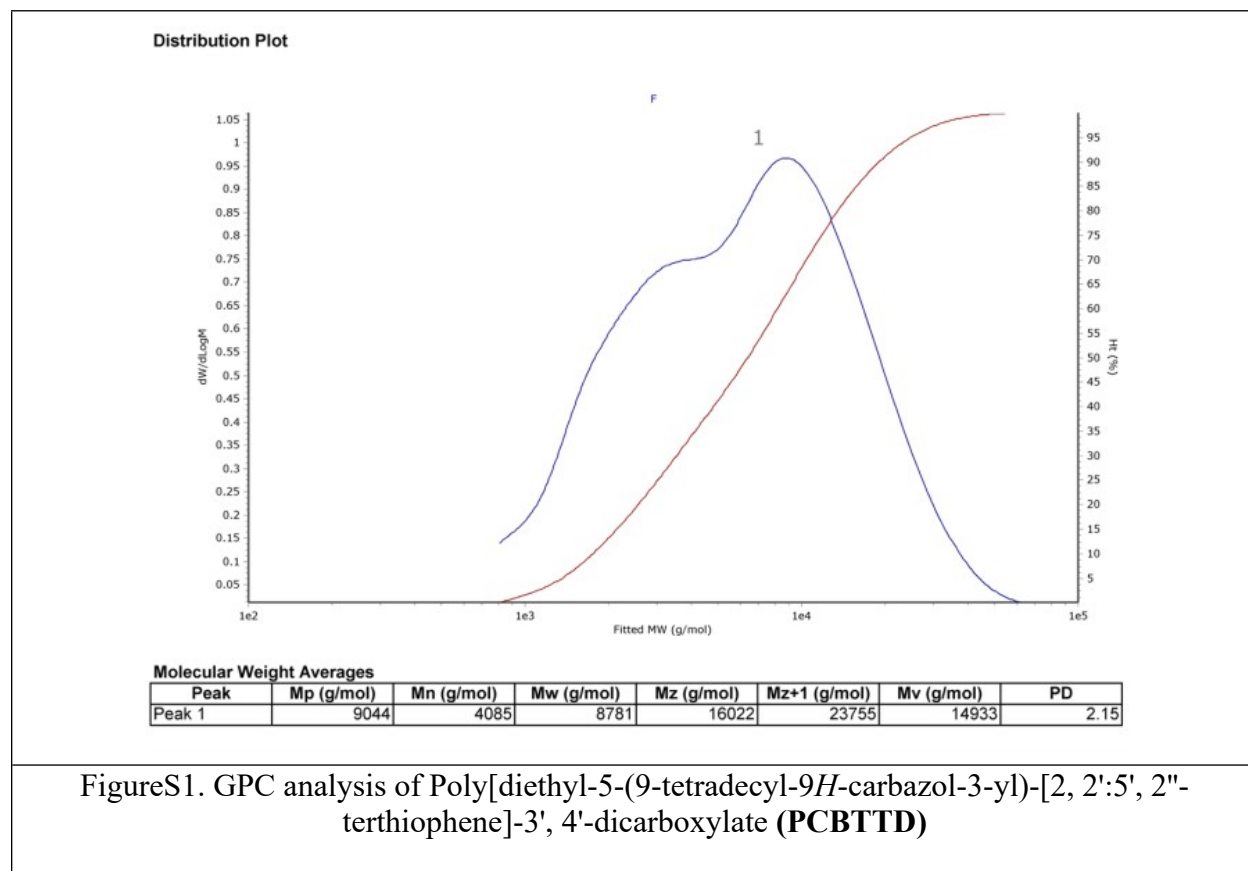
Synthesis of 1,3-dibromo-5-octylthieno[3,4-*c*]pyrrole-4,6-dione (**16**)

Compound **15** (2 g, 7.54 mmol) was dissolved in trifluoroacetic acid (20 mL) and sulfuric acid (7.97 mL). NBS (4.45 g, 25 mmol) was added in small portions with constant stirring. The reaction mixture was stirred at room temperature overnight. The reaction mixture was washed with aqueous sodium thiosulfate solution, and the organic compound was extracted in dichloromethane. The

crude product was purified by column chromatography on silica gel using 10% ethyl acetate in petroleum ether as an eluent to give compound **16** (2.69 g, 85%).

$^1\text{H NMR}$ (CDCl_3) δ 3.59 (t, 2H), 1.63 (m, 2H), 1.30-1.25 (m, 10H), 0.87 (t, 3H). $^{13}\text{C NMR}$ (CDCl_3) δ 14.1, 22.6, 26.8, 28.2, 29.1, 31.7, 38.8, 112.9, 134.7, 160.4.

GPC Analysis data of Polymers



FigureS1. GPC analysis of Poly[diethyl-5-(9-tetradecyl-9*H*-carbazol-3-yl)-[2, 2':5', 2''-terthiophene]-3', 4'-dicarboxylate (**PCBTDD**)

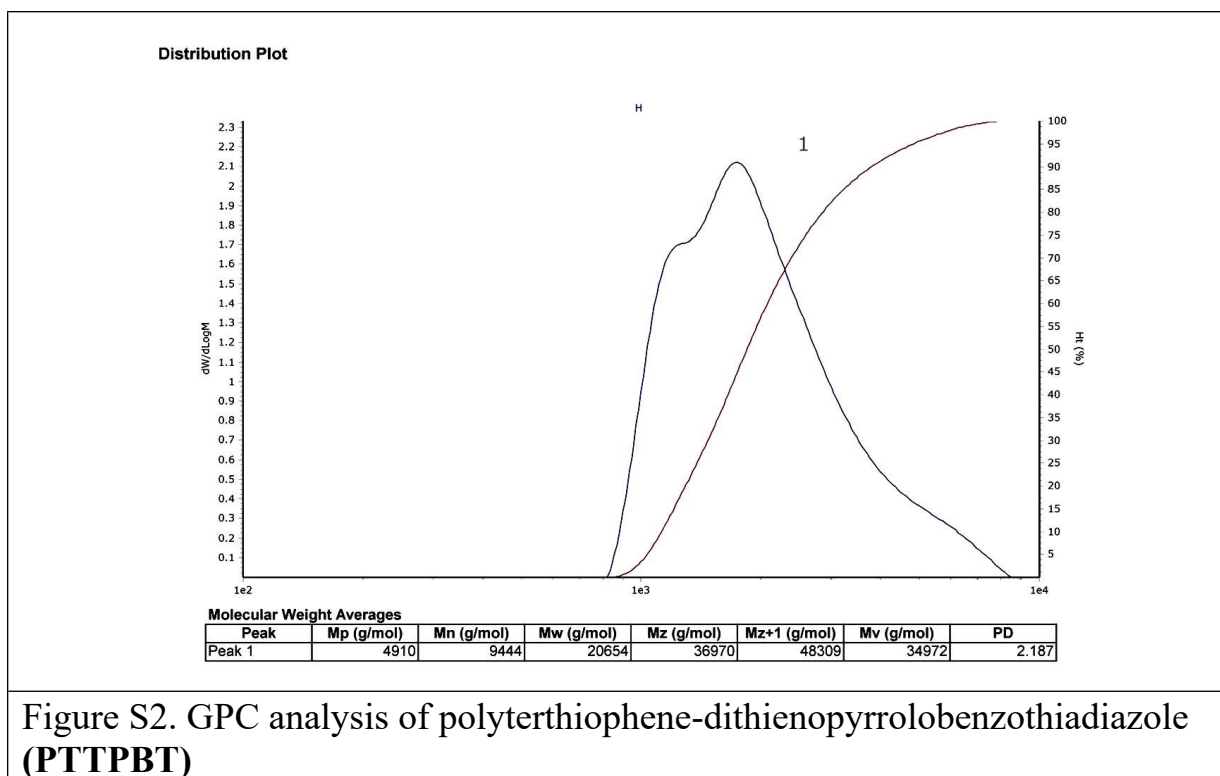


Figure S2. GPC analysis of polyterthiophene-dithienopyrrolobenzothiadiazole (PTTPBT)

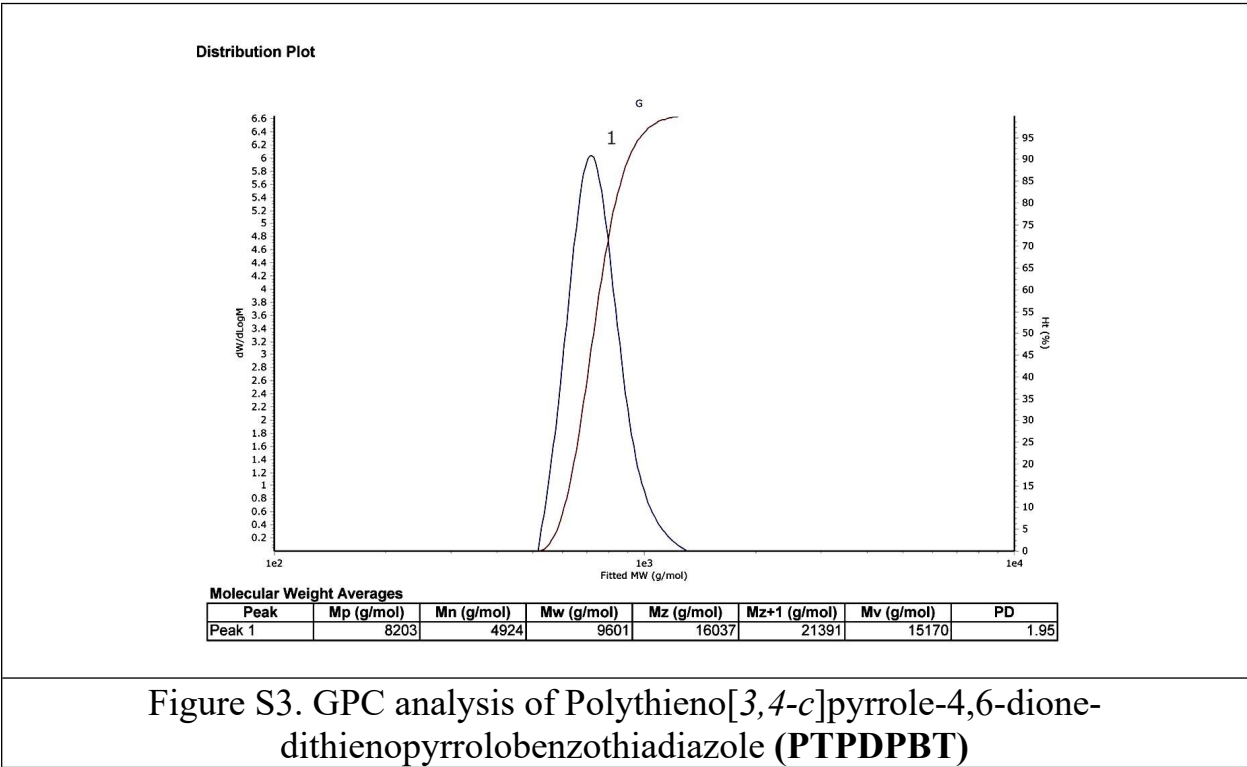
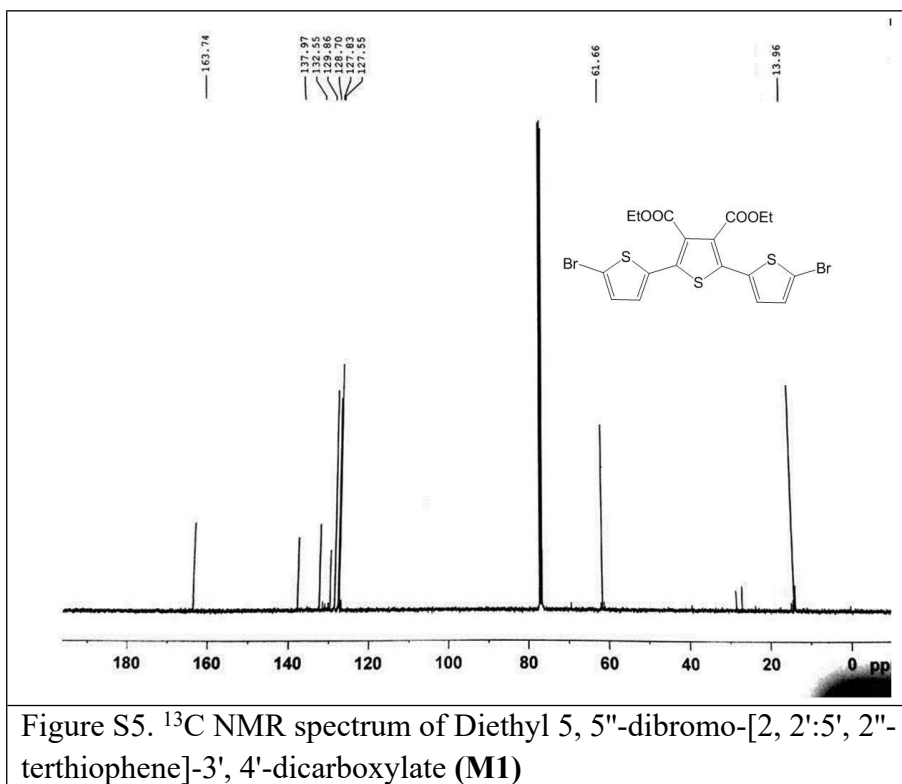
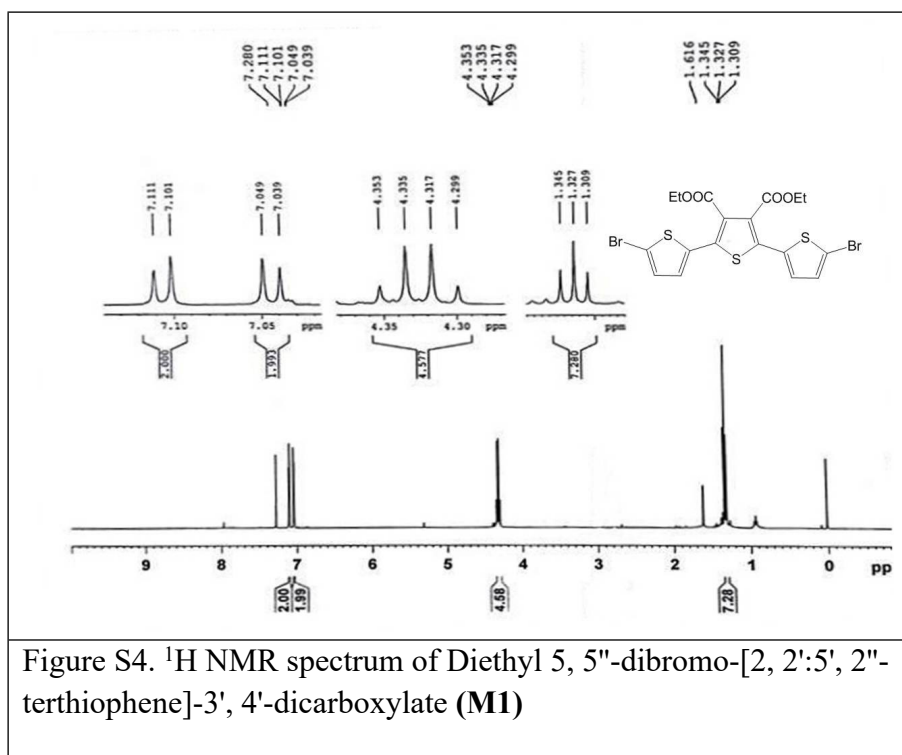
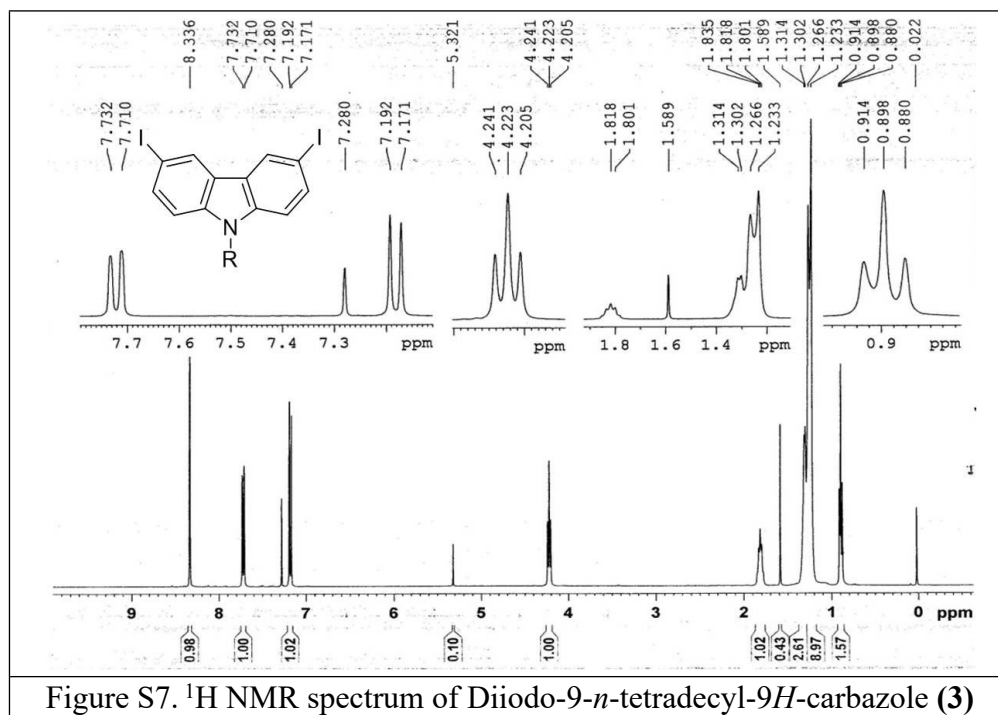
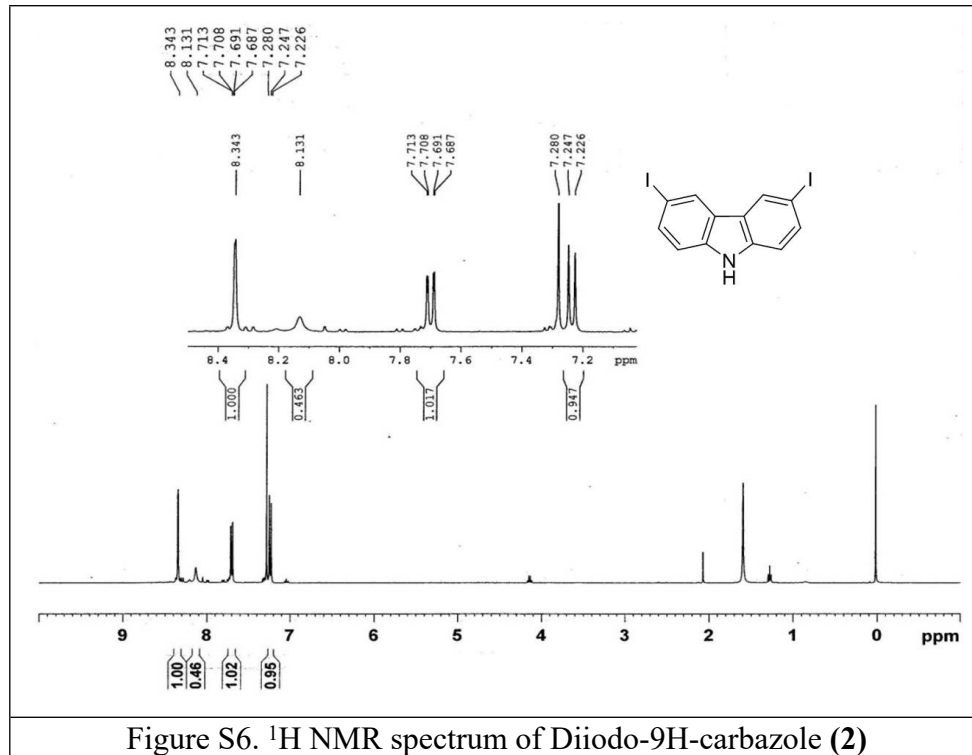
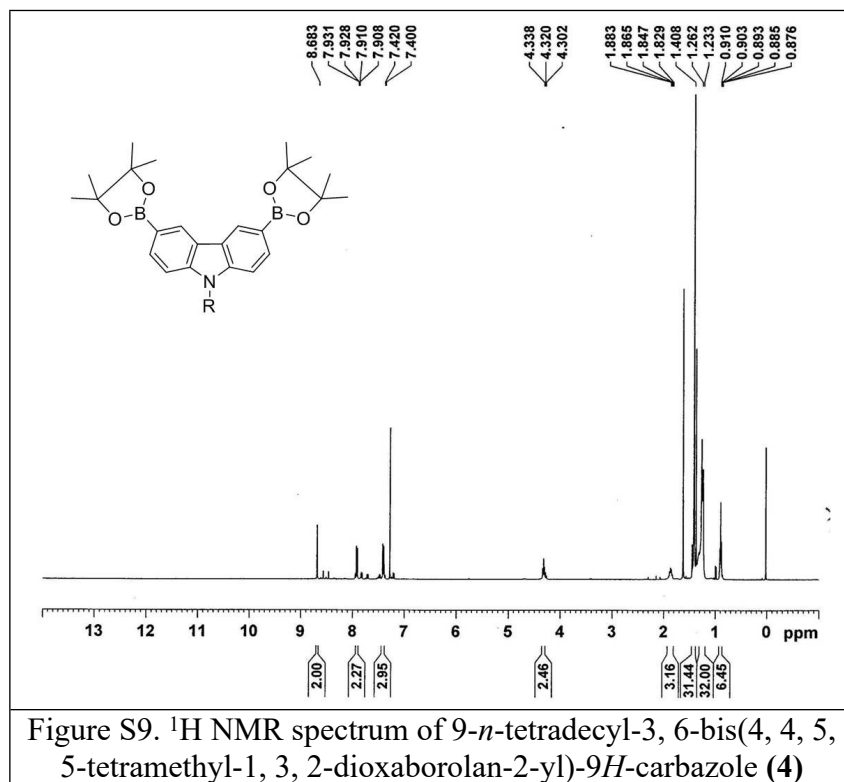
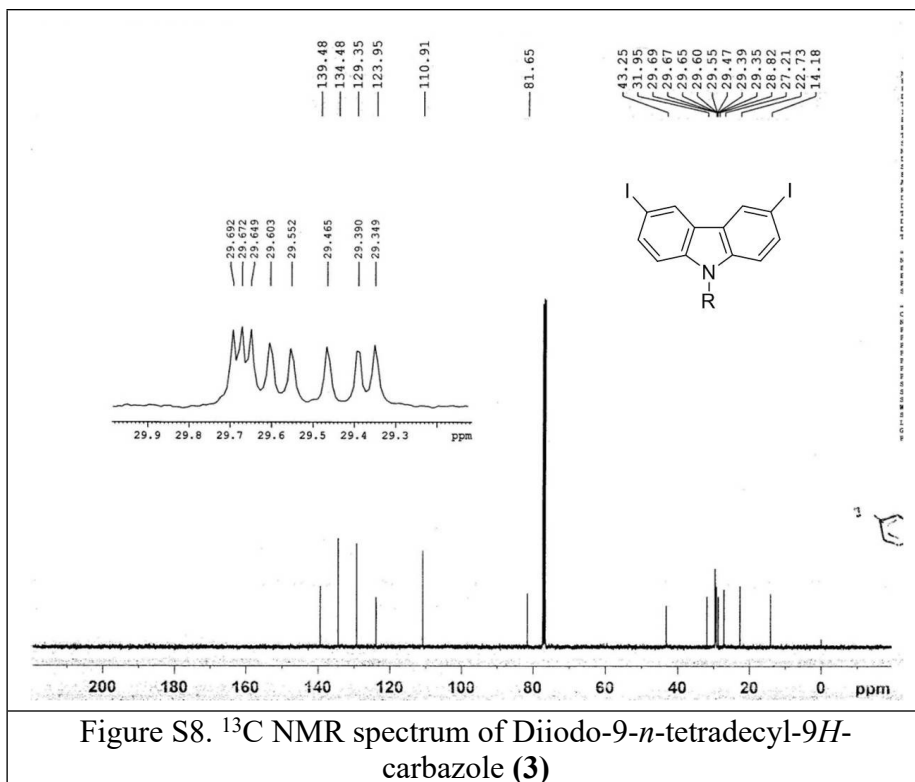


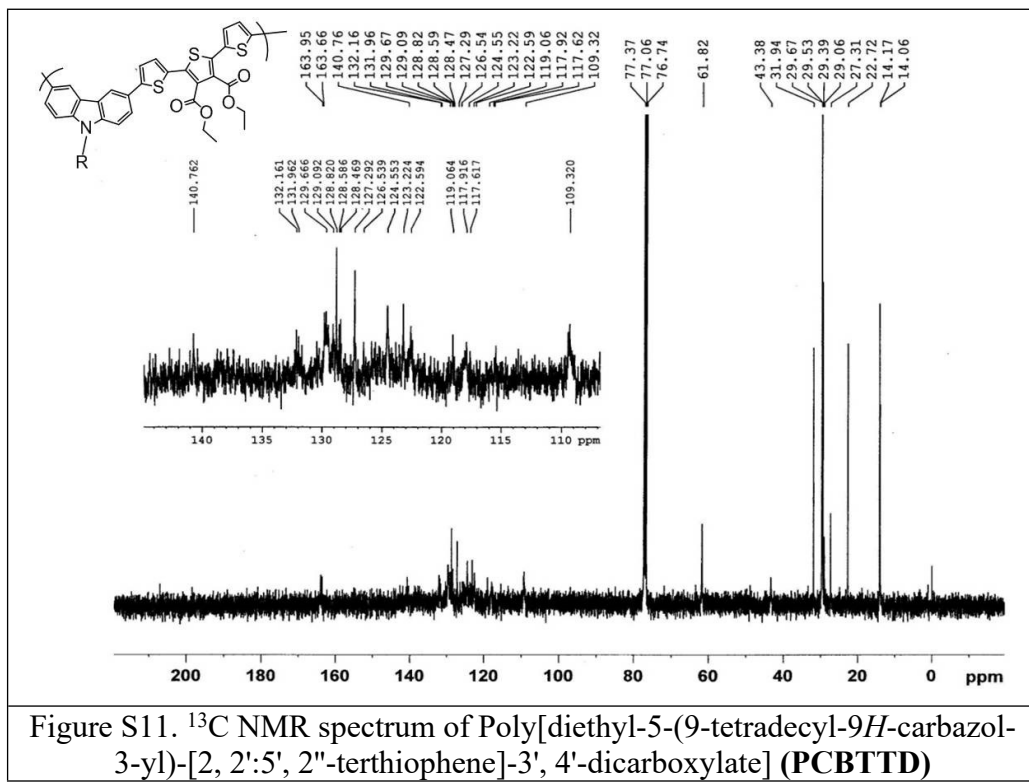
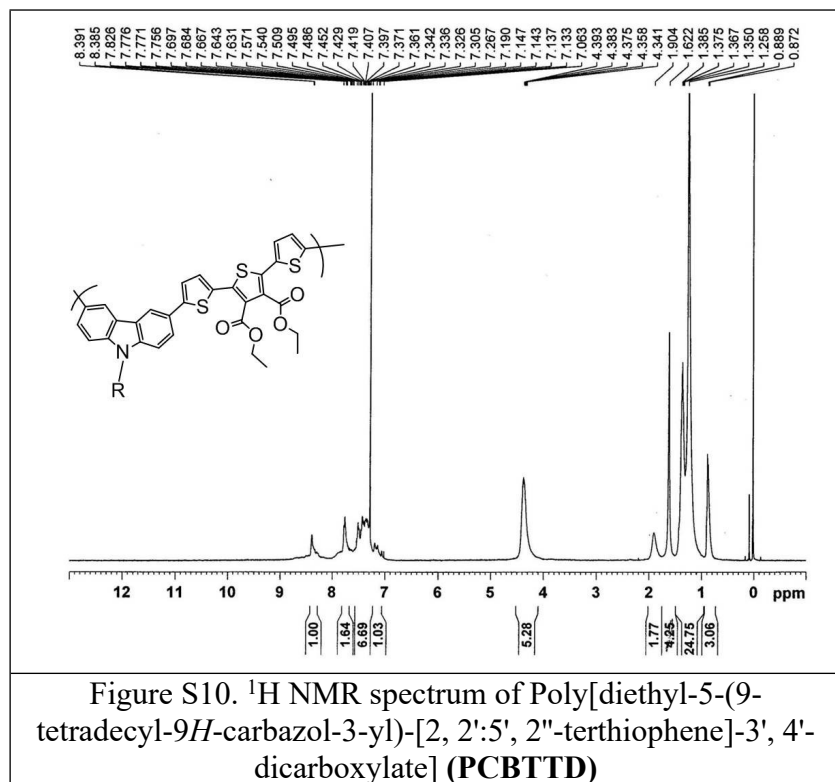
Figure S3. GPC analysis of Polythieno[3,4-*c*]pyrrole-4,6-dione-dithienopyrrolobenzothiadiazole (PTPDPBT)

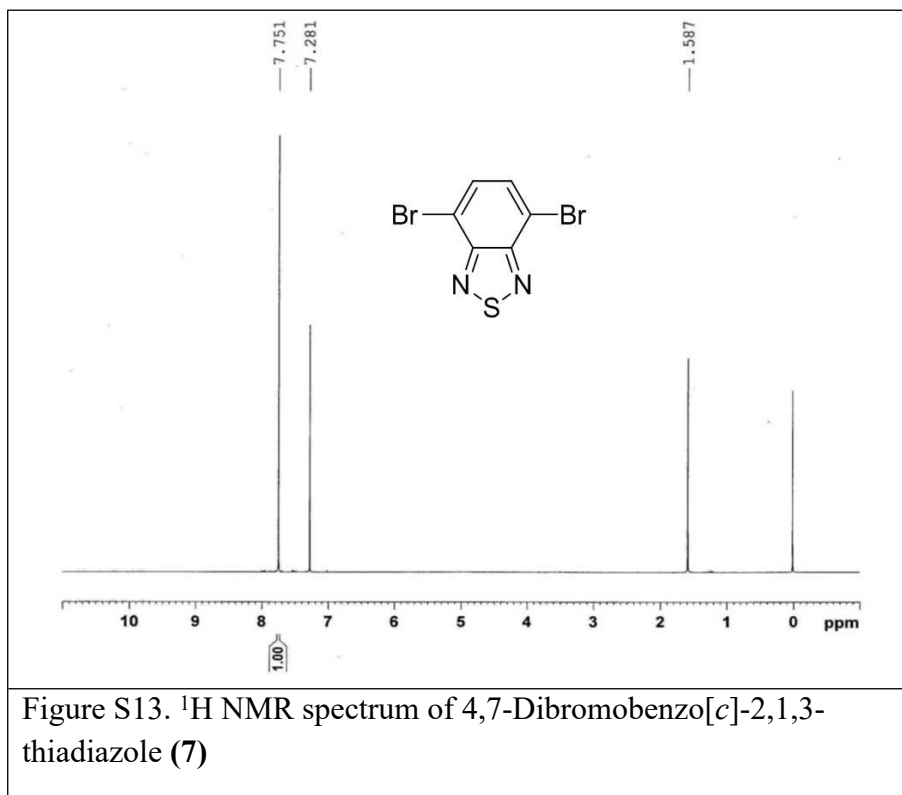
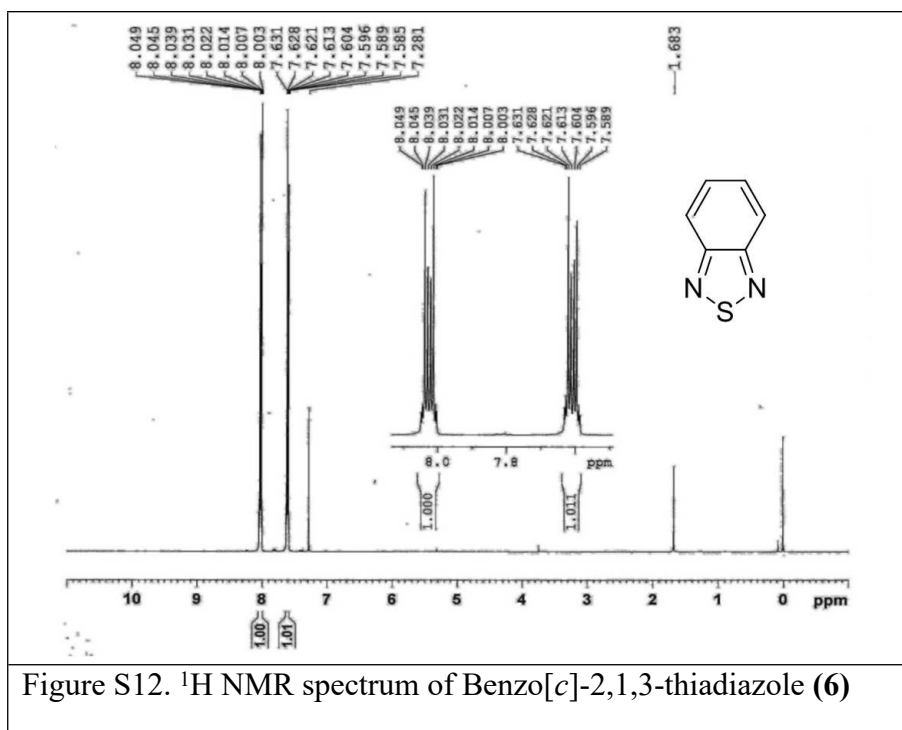
NMR spectra











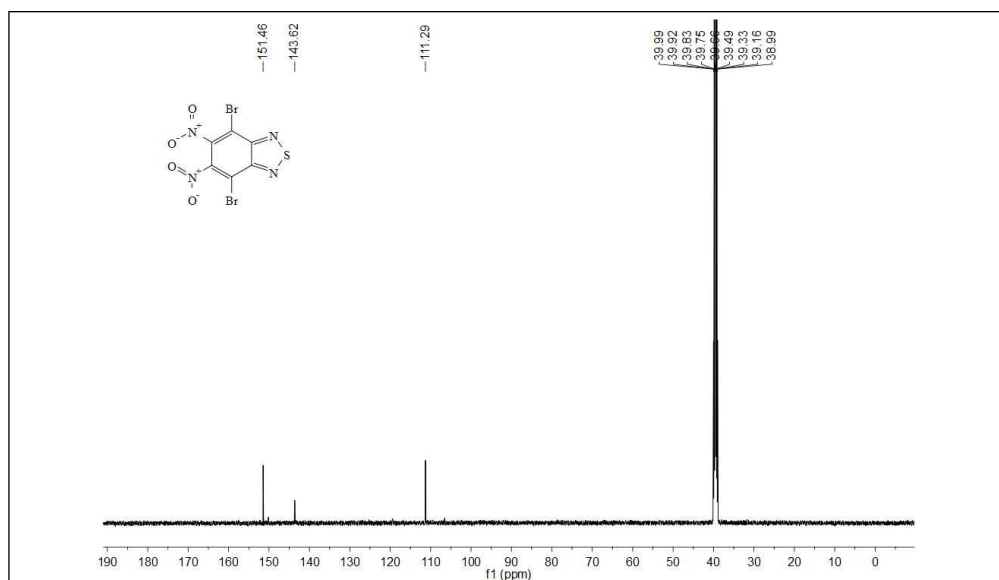


Figure S14. ¹³C NMR spectrum of 4,7-dibromo-5,6-dinitrobenzo[*c*]-2,1,3-thiadiazole (**8**)

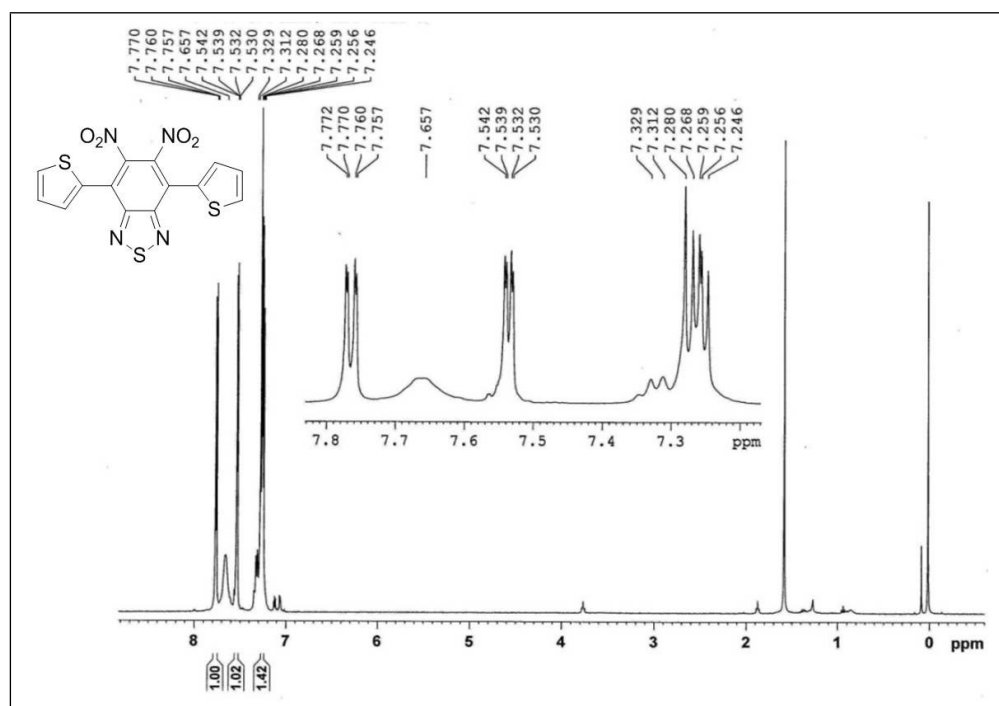


Figure S15. ¹H NMR spectrum of 5,6-Dinitro-4,7-di(thiophen-2-yl)benzo[*c*]-2,1,3-thiadiazole (**9**)

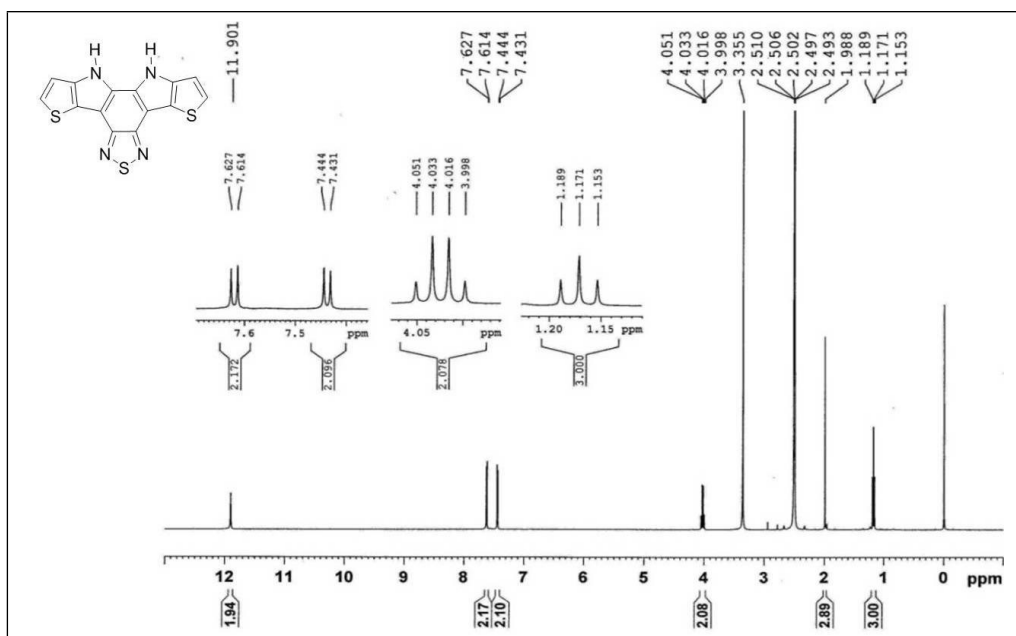


Figure S16. ^1H NMR spectrum of Dithienopyrrolobenzothiadiazole (**10**)

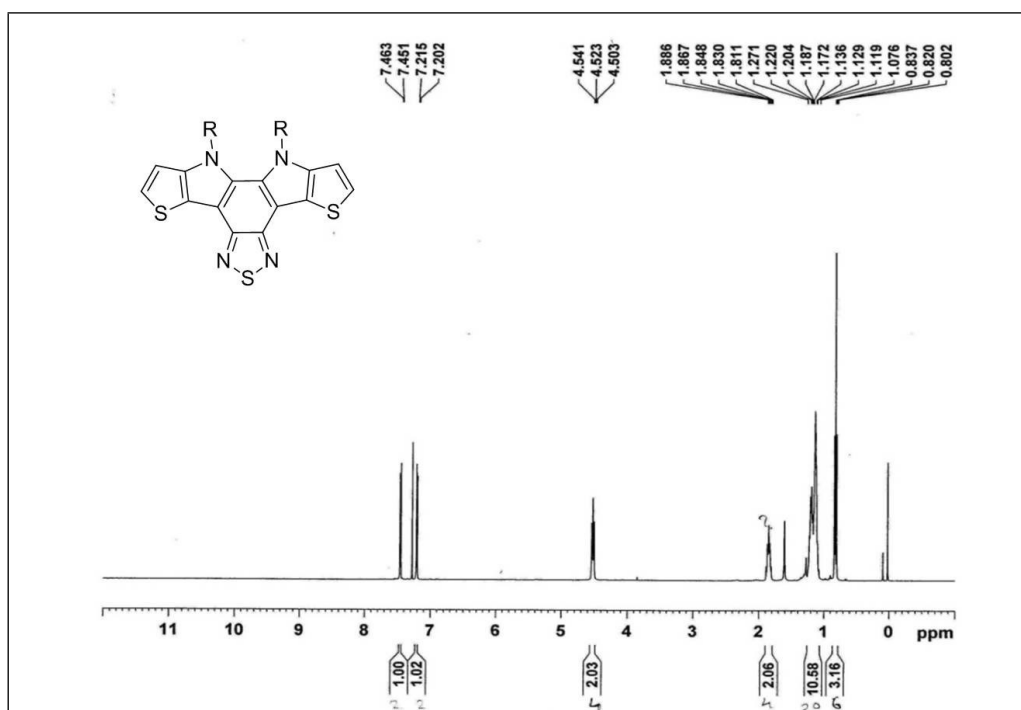
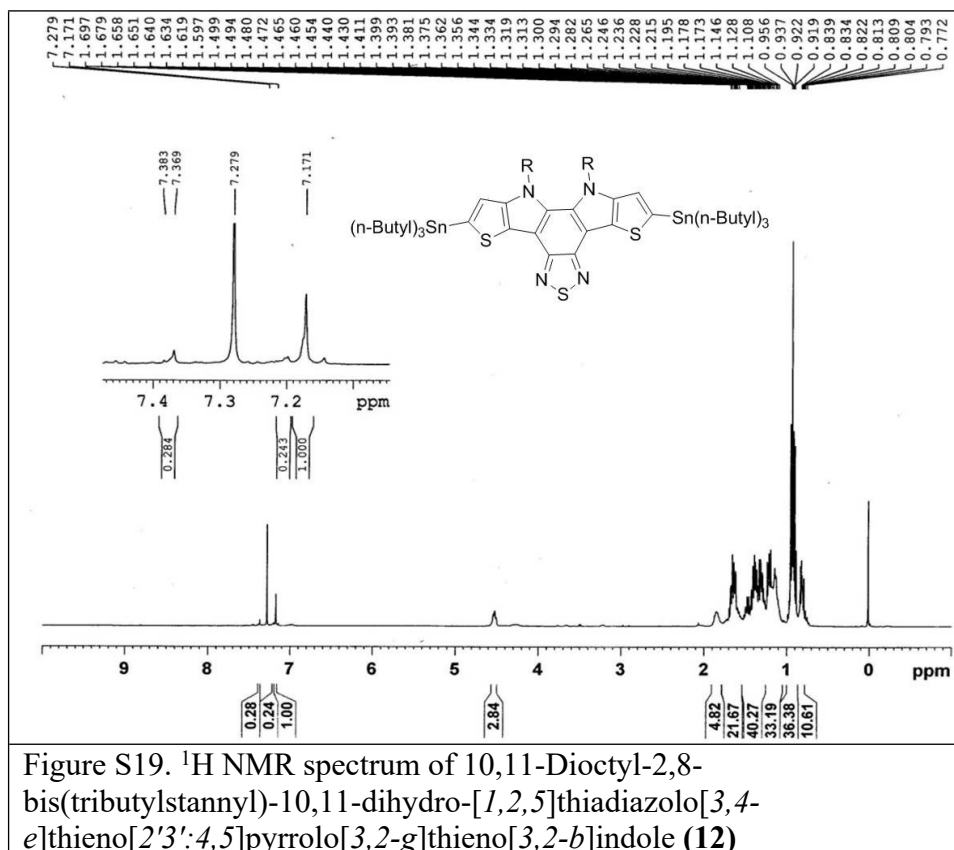
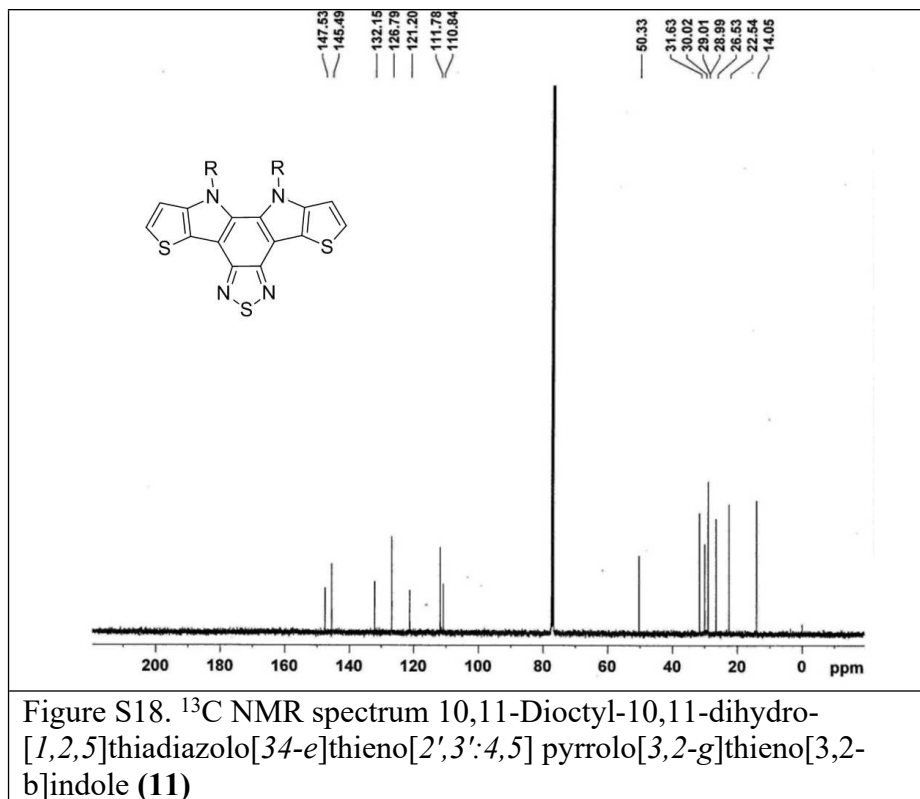
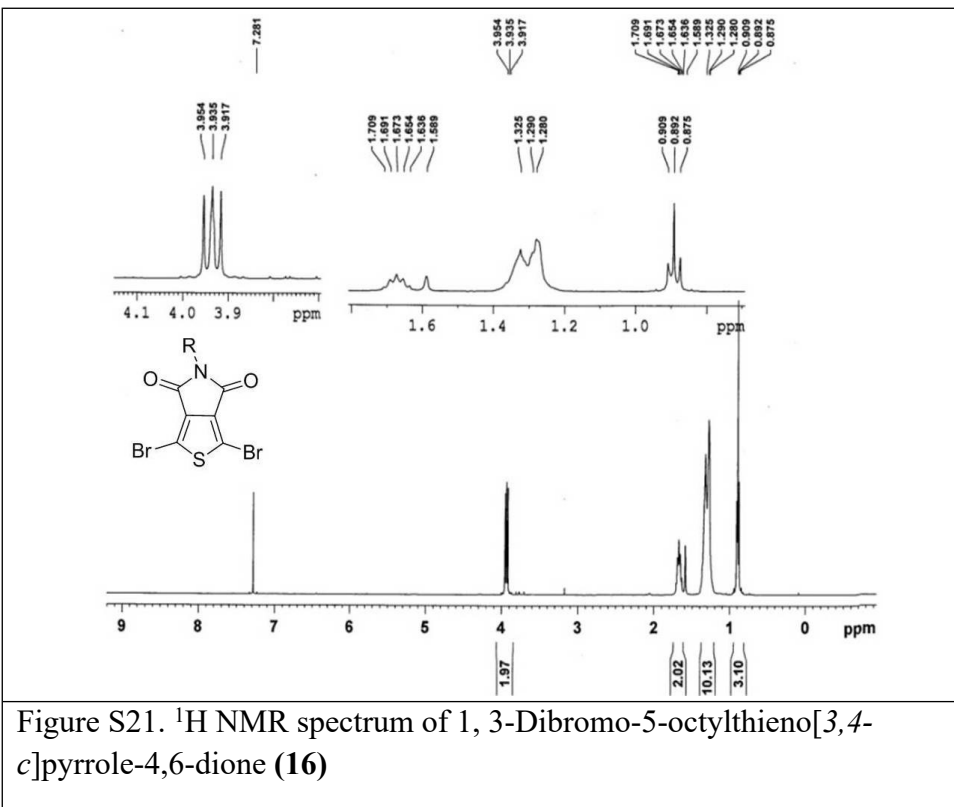
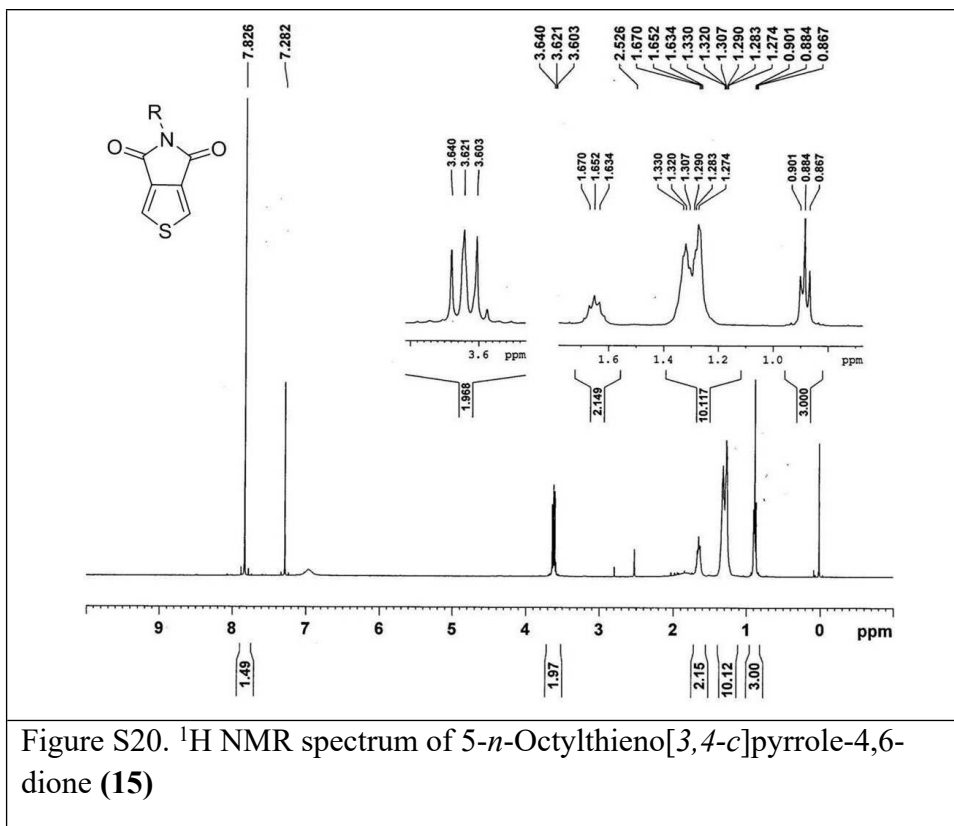
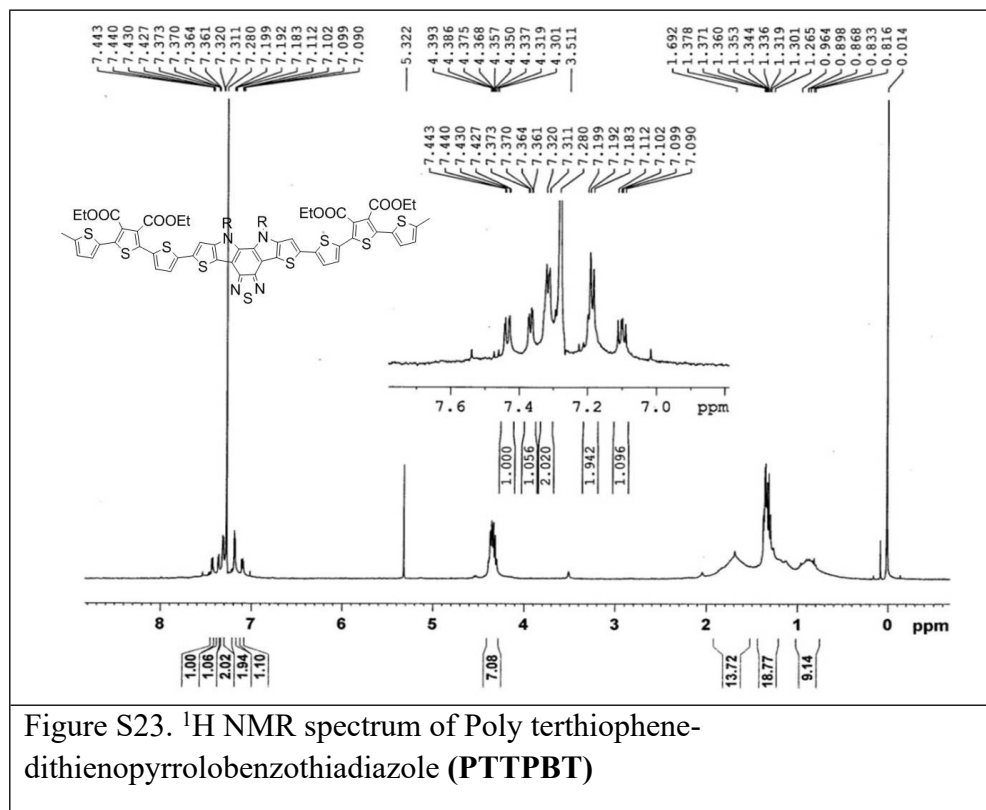
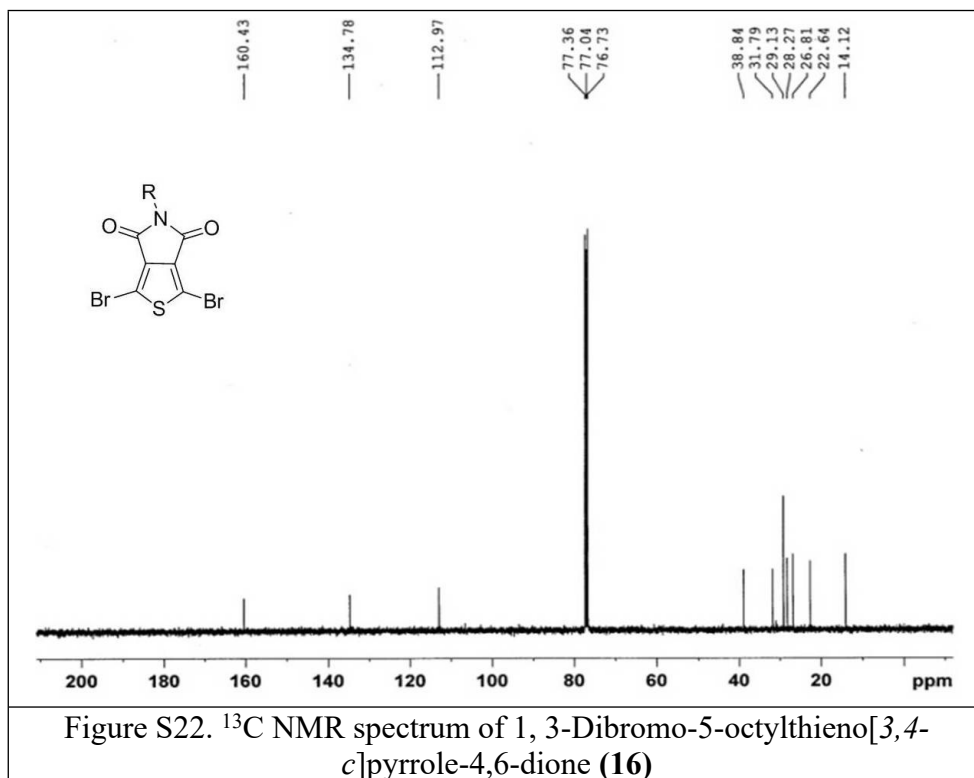
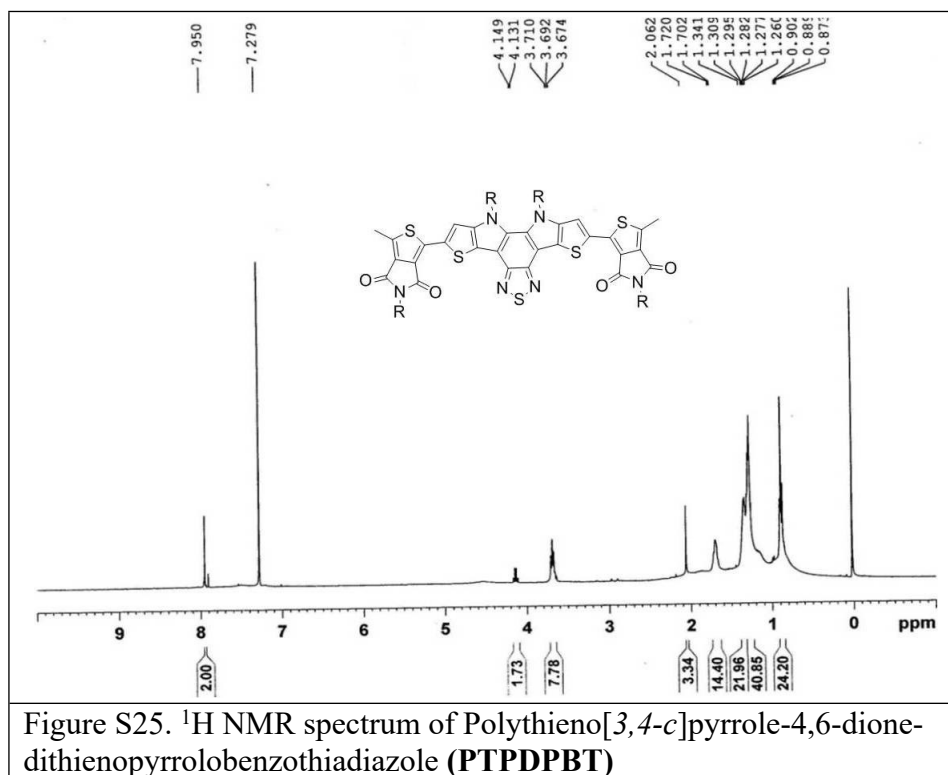
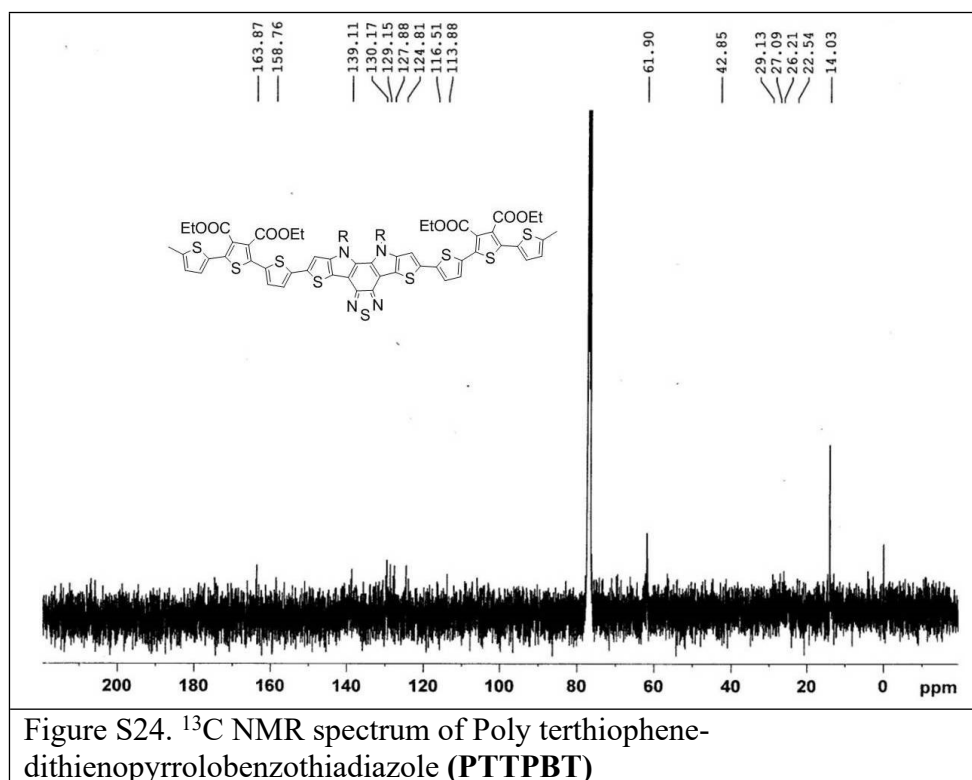


Figure S17. ^1H NMR spectrum of 10,11-Dioctyl-10,11-dihydro-[1,2,5]thiadiazolo[3,4-e]thieno[2',3':4,5] pyrrolo[3,2-g]thieno[3,2-b]indole (**11**)









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