

–Supporting Information –

Lamello-Columnar Mesophase Formation in a Side-Chain Liquid Crystal π -Conjugated Polymer Architecture

Ibtissam Tahar-Djebbar,^a Fabien Nekelson,^{a,b} Benoît Heinrich,^c Bertrand Donnio,^c Daniel Guillon,^c David Kreher,^a Fabrice Mathevet,^{*,a} and André-Jean Attias^{*,a}

^a Laboratoire de Chimie des polymères, Université Pierre et Marie Curie, 4 Place Jussieu 75005, Paris, France

^b DRT/LETI/DIHS/LIMN, CEA-Grenoble, 17 Rue des Martyrs, Grenoble, France

^c Groupe des Matériaux Organiques, IPCMS, 43 rue du Loess, Strasbourg, France

fabrice.mathevet@upmc.fr
andre-jean@upmc.fr

Experimental

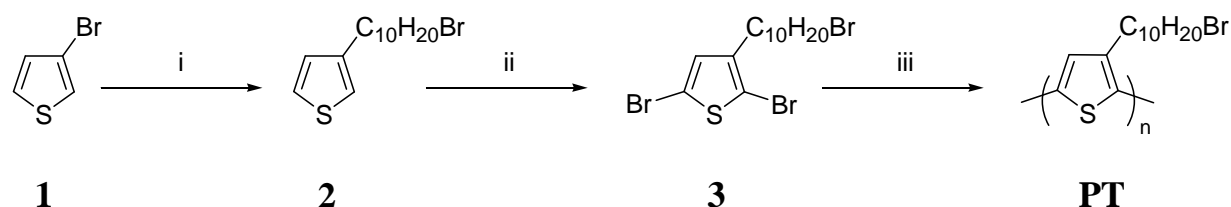
General methods:

All commercially available chemicals were used without further purification and solvents were carefully dried and distilled prior to use. All glassware was previously heat-dried under vacuum with a hot air gun before using. Reactions were performed under an inert atmosphere. Column chromatography was performed on Geduran Silica 60 (40-63 μm , Merck). ^1H NMR spectra were recorded at 200, 250 or 300 MHz on Bruker spectrometers in CDCl_3 . Tetramethylsilane was used as internal standard and the positions of the peaks are reported in ppm. J values are given in Hz. Proton decoupled ^{13}C NMR spectra were recorded at 50, 66 or 75 MHz on the same spectrometer. Gel permeation chromatography (GPC) was performed using an Agilent 1100 Series GPC equipped with a 300 mm x 7.5 PLgel Mixed-D 5 μm 10^{-4} Å, a refractive-index (RI) detector and a diode-array UV-vis detector (DAD) (calibration: polystyrene standards). The column temperature and the flow rate were fixed to 40°C and 1 mL.min⁻¹ in THF (HPLC-Grade). The THF solution used was filtered using cellulose filter (pore size: 0.20 μm) before sample injection. Mass spectra were performed on a MALDI-TOF mass spectrometer (4700 Proteomic analyzer, Applied Biosystems). Low mass positive ions ($m/z < 4000$) and high mass positive ions ($m/z > 4000$) were analyzed in reflector and linear mode, respectively. Samples (< 1mg) were dissolved in 100 μL THF and 1 μL were mixed with 10 μL of matrix (dithranol saturated solution in THF). 1 μL was deposited on the sample holder. Calibration in the high mass range was obtained using the singly and multiply charged ions of Bovine Serum Albumin (BSA) using a regular detector (MultiChannel Plate) and or a high mass detector (CovalX). Polarization optical microscopy (POM) was carried out using a Leica microscope equipped with a Linkam THMS 350 heating plate connected to a Linkam TMS 93 processor. Differential scanning calorimetry (DSC) was performed using TA instruments Q2000 under N_2 flow in aluminium hermetic pans. The XRD patterns were obtained with three different experimental set-ups. In all cases, a linear

monochromatic Cu-K α 1 beam ($\lambda = 1.5405 \text{ \AA}$) was obtained using a sealed-tube generator (900 W) equipped with a bent quartz monochromator. The 3 setups are based on the Guinier transmission geometry, with different focalization distances. In all cases, the crude powder was filled in Lindemann capillaries of 1 mm diameter. An initial set of diffraction patterns was recorded on an image plate; periodicities up to 80 \AA can be measured, and the sample temperature controlled to within $\pm 0.3^\circ\text{C}$ from the ambient up to 300°C . The second set of diffraction patterns was recorded with a curved Inel CPS 120 counter gas-filled detector linked to a data acquisition computer; periodicities up to 60 \AA can be measured, and the sample temperature controlled to within $\pm 0.05^\circ\text{C}$ from the ambient up to 200°C . Finally, the last set of diffraction patterns was recorded on image plate, and periodicities up to 350 \AA can be measured, and the sample temperature controlled to within $\pm 0.01^\circ\text{C}$ from the ambient up to 200°C . In each case, exposure times were varied from 1 to 24 h.

Synthesis

Scheme S1. Synthesis of polymer **PT**^{1,2}



Conditions: i) a) BuLi, hexane/THF, -40°C , 1h b) 1,10-Dibromodecane, $-10^\circ\text{C}/\text{RT}$, 2h ii) NBS, DMF, RT iii) a) $i\text{PrMgCl}$, THF, 0°C , 1h b) Ni(dppp)Cl_2 , $0^\circ\text{C}/\text{RT}$.

Synthesis of 3-(10-bromodecyl)thiophene (2)

3-Bromothiophene (3mL, $3.1 \cdot 10^{-2}$ mol) was dissolved in 50 mL of freshly distilled hexane under an atmosphere of argon. The resulting solution was cooled down at -40°C and the solution stirred for 10 minutes. $n\text{-BuLi}$ (19.4 mL, $3.1 \cdot 10^{-2}$ mol, 1.6 M in hexane) was added dropwise *via* a syringe at this temperature. 5 mL of THF was added to the pale yellow solution which was further left stirring for 1h, at which time, the resulting white suspension was allowed to warm to -10°C . 1,10-Dibromodecane (28.7 mL, 0.12 mol) was added in one portion at -10°C and the mixture was warmed to room temperature and stirred for 2h. The reaction was quenched by pouring 15 mL of water into the flask and extracted with diethyl ether (2 x 30 mL). The organic phase was washed with water (3 x 30 mL) and dried over anhydrous MgSO_4 . A pale yellow oil was obtained as a crude product after solvent removal which was purified by fractional distillation under reduced pressure. The excess of dibromodecane was first isolated (B.p. 105°C @ 0.08 mm Hg) followed by the *title compound* (B.p. 135°C @ 0.08 mm Hg) which was filtered over silica gel using petroleum ether to yield a colorless oil (3.52 g, 37%); δ_{H} (200 MHz, CDCl_3) 7.24 (1H, dd, J 2.9 and 4.9, 5- H_{arom}), 6.93 (2H, m, 4- H_{arom} , 2- H_{arom}), 3.41 (2H, t, J 6.8, $-\text{CH}_2\text{-Br}$), 2.63 (2H, t, J 7.6, $-\text{CH}_2\text{-Ar}$), 1.86 (2H, m, H_{aliph}), 1.62 (2H, m, H_{aliph}), 1.40-1.25 (12H, m, H_{aliph}); δ_{C} (50 MHz, CDCl_3) 143.31, 128.38, 125.16, 119.88, 34.20, 32.95, 30.66, 30.39, 29.58, 29.53, 29.42, 28.87, 28.83, 28.28. Anal. Calcd for $\text{C}_{14}\text{H}_{23}\text{BrS}$: C, 55.44; H, 7.64; Br, 26.34, Found: C, 55.80; H, 7.68; Br, 25.98.

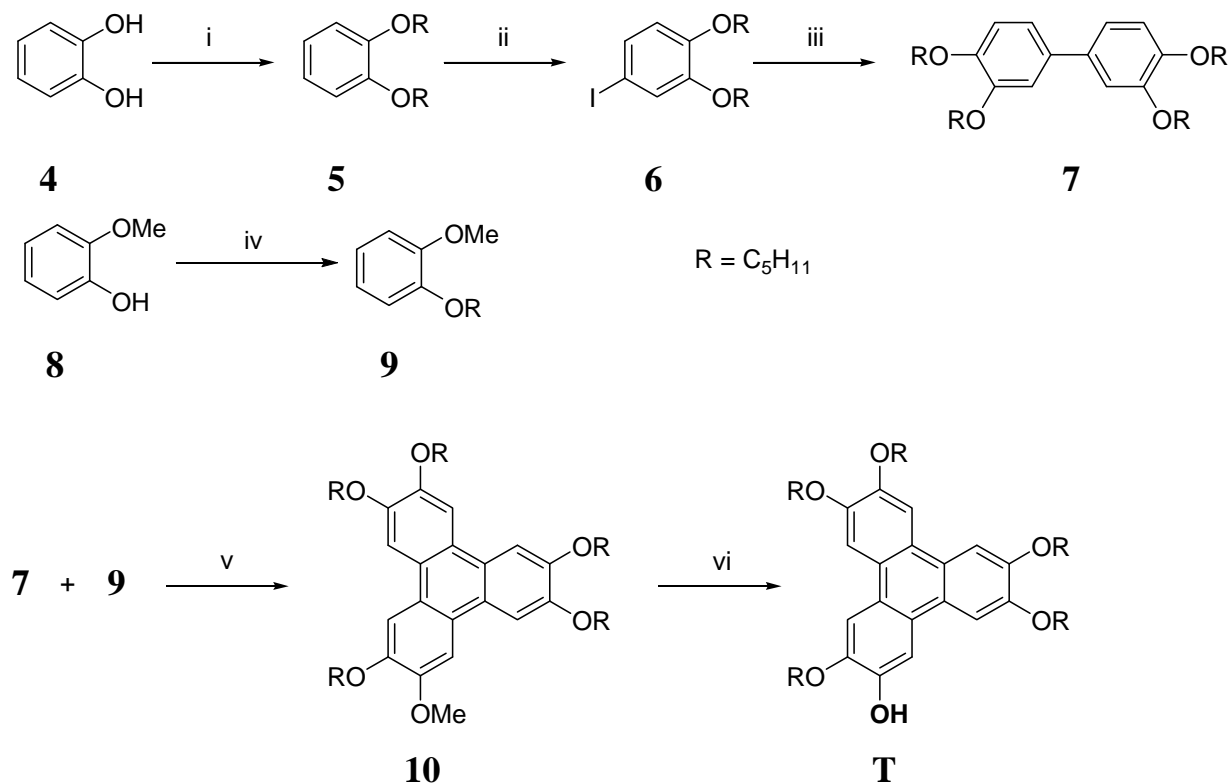
Synthesis of 2,5-Dibromo-3-(10-bromodecyl)thiophene (3)

3-(10-Bromodecyl)thiophene (1.61 g, 5×10^{-3} mol) was dissolved in 5 mL of dimethylformamide under an atmosphere of nitrogen. *N*-Bromosuccinimide (2.3 g, 1.3×10^{-2} mol) in 10 mL of DMF was added dropwise to the clear solution at room temperature. After stirring the reaction mixture overnight, the reaction mixture was poured into cold water (50 mL) and extracted with 3 x 30 mL of diethyl ether. The organic phase was washed with water (3 x 30 mL), dried over anhydrous MgSO_4 and concentrated under reduced pressure. The crude product was submitted to column chromatography over silica gel using hexane as eluant to yield the *title compound* (1.98 g, 81%); δ_{H} (200 MHz, CDCl_3) 6.77 (1H, s, 4- H_{arom}), 3.41 (2H, t, J 6.8, $-\text{CH}_2\text{-Br}$), 2.50 (2H, t, J 7.5, $-\text{CH}_2\text{-Ar}$), 1.86 (2H, m, H_{aliph}), 1.65-1.25 (14H, m, H_{aliph}); δ_{C} (75 MHz, CDCl_3) 142.00, 131.97, 109.05, 107.64, 34.20, 32.96, 30.56, 29.56, 29.50, 29.48, 29.32, 29.01, 28.87, 28.30. Anal. Calcd for $\text{C}_{14}\text{H}_{21}\text{Br}_3\text{S}$: C, 36.47; H, 4.59; Br, 51.99, Found: C, 36.70; H, 4.42; Br: 51.15.

Synthesis of HT-poly(3-(10-bromodecyl)thiophene) (PT)

All glassware and solvent were carefully dried before use. 2,5-Dibromo-3-(10-bromodecyl)thiophene (4 g, 8.7×10^{-3} mol) was poured into 100 mL of dry THF at 0°C under an atmosphere of argon. A solution of isopropyl magnesium chloride (4.3 mL, 8.7×10^{-3} mol, 2.0 M in THF) was added through a syringe and the resulting pale yellow solution was stirred for 1h at 0°C . A suspension of Ni(dppp)Cl_2 (75 mg, 1.48×10^{-4} mol, 1.6 mol %) in THF (5 mL) was quickly injected into the flask. The resulting red solution was gently warmed to room temperature and left overnight under constant stirring. The polymerization was quenched by pouring a 6 M solution of HCl (100 mL) followed by extraction with chloroform (100 mL). The organic layer was successively washed with water (3 x 100 mL), dried over MgSO_4 and concentrated under reduced pressure. The crude product was precipitated using methanol and the resulting purple solid was filtered through a Soxhlet thimble. The *title compound* was obtained (1.31 g, 50%) after successive extraction with methanol; $M_n = 14200$, $M_w/M_n = 1.15$ (GPC, PS Standard); δ_{H} (300 MHz, CDCl_3) 6.98 (1H, s, H_{arom}), 3.39 (2H, t, J 6.8, $-\text{CH}_2\text{-Br}$), 2.80 (2H, m, $-\text{CH}_2\text{-Ar}$), 1.90-1.60 (4H, m, H_{aliph}), 1.50-1.31 (12H, m, H_{aliph}); δ_{C} (75 MHz, CDCl_3) 139.98, 133.80, 130.59, 128.73, 34.16, 34.14, 32.95, 30.64, 29.64, 29.59, 29.56, 28.91, 28.31. Anal. Calcd for $\text{C}_{14}\text{H}_{21}\text{BrS}$: C, 55.81; H, 7.03; Br, 26.52, Found: C, 55.43; H, 6.94; Br: 26.21.

Scheme S2. Synthesis of unsymmetrical triphenylene **T**³



Conditions: i) 1-Bromopentane, K₂CO₃, KI, TBAB, EtOH, Reflux ; ii) I₂, HIO₃, H₂SO₄/AcOH/H₂O/CHCl₃, 40°C ; iii) Zn, 10% Pd /C, Acetone/H₂O, 50°C ; iv) 1-Bromopentane, K₂CO₃, KI, Acetone, Reflux ; v) a) FeCl₃, CH₂Cl₂ b) MeOH ; vi) a) Ph₂PH, BuLi, THF, 0°C b) Reflux for 3h c) HCl 3M

Synthesis of 1,2-bis(pentyloxy)benzene (5)

Catechol **4** (10 g, 0.09 mol), 1-bromopentane (25 mL, 0.20 mol), potassium carbonate (50 g, 0.36 mol), potassium iodide (0.94 g, 6 × 10⁻³ mol) and a crystal of tetrabutylammonium bromide were added to absolute ethanol (200 mL) under an atmosphere of argon. The light pink mixture was heated at reflux for 24h, cooled down to room temperature and extracted with dichloromethane (100 mL). The organic layer was successively washed with water (3 × 50 mL), dried over MgSO₄, filtered and the solvent removed under reduced pressure. The crude product was filtered through a short plug of silica gel using a 1:1 mixture of DCM/Hexane to yield the *title compound* (18.79 g, 83%) as a yellow oil; δ_H (250 MHz, CDCl₃) 6.90 (4H, s, H_{arom}), 4.01 (4H, t, *J* 6.6, -OCH₂-), 1.84 (4H, m, H_{aliph}), 1.44 (8H, m, H_{aliph}), 0.94 (6H, t, *J* 6.9, -CH₃); δ_C (66 MHz, CDCl₃) 148.78, 120.56, 113.57, 68.80, 28.60, 27.82, 22.09, 13.65.

Synthesis of 1,2-bis(pentyloxy)-4-iodobenzene (**6**)

1,2-Bis(pentyloxy)benzene (14 g, 0.056 mol) was added to a mixture of acetic acid (60 mL), distilled water (20 mL), chloroform (15 mL) and concentrated H₂SO₄ (1 mL). Iodine (5.3 g, 0.02 mol) was added to the solution which was then warmed to 40°C. Iodic acid (2.1 g, 0.01 mol) was added in three portions, the first on reaching 40°C, the second after 1h and the third after a further 1h. The reaction was left stirring overnight at 40°C and chloroform (50 mL) and a 30wt% solution of Na₂SO₃ (50 mL) were added to the purple mixture. The organic phase was separated and successively washed with water (3 x 50 mL), dried over MgSO₄, filtered and the solvent removed under reduced pressure to afford the *title compound* (21.04 g, 94%) as a yellow oil; δ_{H} (250 MHz, CDCl₃) 7.18 (1H, dd, *J* 2.1 & 8.3, 5-H_{arom}), 7.13 (1H, d, *J* 2.1, 3-H_{arom}), 6.61 (1H, d, *J* 8.5, 6-H_{arom}), 3.96 (2H, t, *J* 6.6, -O-CH₂-), 1.81 (2H, m, H_{aliph}), 1.42 (8H, m, H_{aliph}), 0.94 (3H, t, *J* 6.9, -CH₃); δ_{C} (66 MHz, CDCl₃) 149.67, 148.78, 129.36, 122.11, 115.19, 82.07, 68.94, 68.88, 28.65, 28.43, 27.73, 27.71, 22.05, 22.02, 19.03, 11.04.

Synthesis of 3,4,3',4'-tetrakis(pentyloxy)biphenyl (**7**)

Zinc powder (4.7 g, 7.2 10⁻² mol) and 10% palladium (2.0 g, 8 mol%, 1.9 10⁻³ mol) on charcoal were added to a mixture of water (100 mL) and acetone (100 mL). The resulting suspension was heated at 50°C with stirring for 30 minutes in open atmosphere. 1,2-Bis(pentyloxy)-4-iodobenzene (9 g, 2.4 10⁻² mol) in 10 mL of acetone was slowly added. The mixture was stirred for 12h. The reaction mixture was cooled to room temperature, filtered through a short plug of silica, and eluted with diethyl ether. The organic fraction was washed with water (2 x 50 mL), saturated NaCl solution (50 mL), dried with MgSO₄ and the solvent was evaporated under reduced pressure. The *title compound* (3.3 g, 55%) was obtained as a white solid after recrystallisation from a 1:1 mixture of DCM/EtOH; δ_{H} (250 MHz, CDCl₃) 7.06 (4H, dd, *J* 2.2 & 6.5, 3-H_{arom} & 5-H_{arom}), 6.92 (2H, d, *J* 8.9, H_{arom}), 4.04 (8H, m, -O-CH₂-), 1.85 (8H, m, H_{aliph}), 1.56-1.37 (16H, m, H_{aliph}), 0.94 (12H, t, *J* 6.9, -CH₃).

Synthesis of 1-pentyloxy-2-methoxybenzene (**9**)

Guaiacol **8** (5 g, 0.04 mol), 1-bromopentane (6 mL, 0.05 mol), potassium carbonate (22.3 g, 0.16 mol) and potassium iodide (0.4 g, 2.5 10⁻³ mol) were added to acetone (100 mL) under an atmosphere of argon. The reaction mixture was heated at reflux for 24h, cooled down to room temperature and extracted with dichloromethane (100 mL). The organic layer was successively washed with water (3 x 50 mL), dried over MgSO₄, filtered and the solvent removed under reduced pressure to give the *title compound* (6.12g, 78%) as a colorless oil; δ_{H} (250 MHz, CDCl₃) 6.91 (4H, s, H_{arom}), 4.02 (2H, t, *J* 7.0, -O-CH₂-), 3.88 (3H, s, -O-CH₃), 1.87 (2H, m, H_{aliph}), 1.45 (4H, m, H_{aliph}), 0.94 (3H, t, *J* 6.9, -CH₃); δ_{C} (66 MHz, CDCl₃) 149.53, 148.7, 120.93, 120.88, 113.11, 111.89, 69.05, 56.05, 29.02, 28.23, 22.61, 14.15.

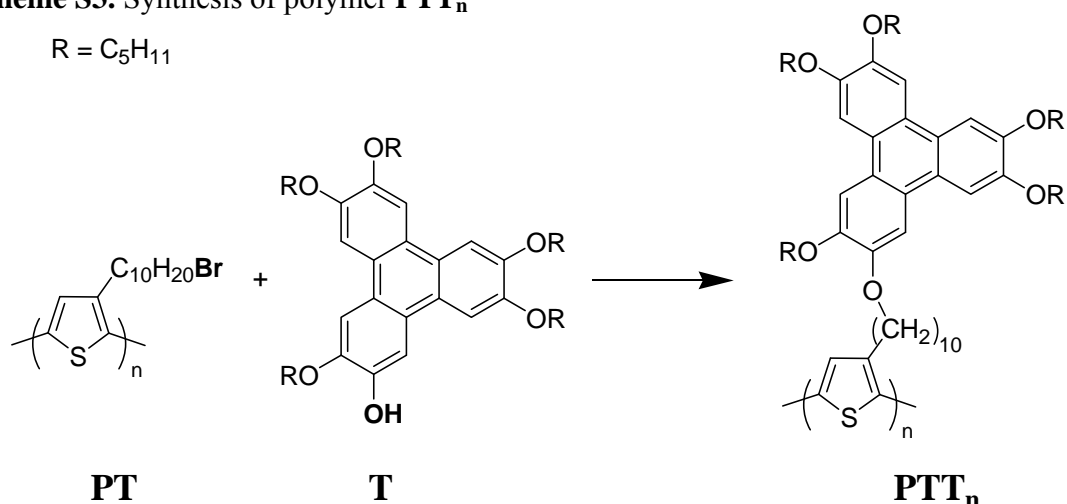
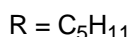
Synthesis of 2,3,6,7,10-pentakis(pentyloxy)-11-methoxytriphenylene (10)

3,4,3',4'-Tetrakis(pentyloxy)biphenyl (3.0 g, 6×10^{-3} mol) and 1-pentyloxy-2-methoxybenzene (2.3 g, 1.2×10^{-2} mol) were added to anhydrous dichloromethane (30 mL) under an atmosphere of argon. Iron (III) trichloride (3.9 g, 2.4×10^{-2} mol) was added slowly to the reaction mixture to avoid strong evolution of acidic gas. The reaction was monitored by TLC (1:1 hexane/dichloromethane) until completion was reached. The dark blue mixture was poured carefully into stirred methanol (100 mL), left for 1h stirring and then allowed to cool overnight in a fridge. The grey solid was filtered off and washed with cold methanol. After drying under vacuum, the crude solid was chromatographed over silica gel using a 1:9 mixture of hexane/Et₂O to yield the *title compound* (2.24 g, 54%) as a light yellow solid after solvent removal and recrystallisation from DCM/EtOH; δ_{H} (250 MHz, CDCl₃) 7.83 (6H, m, H_{arom}), 4.26 (10H, m, -O-CH₂-), 4.11 (3H, s, -O-CH₃), 1.96 (10H, m, H_{aliph}), 1.61-1.41 (24H, m, H_{aliph}), 0.98 (15H, t, *J* 6.9, -CH₃).

Synthesis of 2,3,6,7,10-pentakis(pentyloxy)-11-hydroxytriphenylene (T)

Diphenylphosphine (0.9 mL, 4.9×10^{-3} mol) was added to anhydrous THF (30 mL) under an atmosphere of argon at 0°C. *n*-BuLi (4.1 mL, 1.6 M in hexane, 6.6×10^{-3} mol) was added to the well-stirred solution over 30 minutes. After a further 30 minutes, 2,3,6,7,10-pentakis(pentyloxy)-11-methoxytriphenylene (2.27 g, 3.3×10^{-3} mol) was added and the solution was heated at reflux for 3 hours. 3 M solution of HCl (30 mL) was added and the organic phase was extracted with ethyl acetate, washed with water (3 x 30 mL) and dried over MgSO₄. After solvent removal, the crude product was chromatographed over silica gel using 2:1 mixture of petroleum ether/DCM to recover the diphenylphosphine derivatives followed by the *title compound* (1.62 g, 73%) using a 1:1 mixture of DCM/petroleum ether followed by recrystallisation from DCM/MeOH; δ_{H} (200 MHz, CDCl₃) 7.98-7.75 (6H, m, H_{arom}), 5.91 (1H, s, OH), 4.23 (10H, m, -O-CH₂-), 1.95 (10H, m, H_{aliph}), 1.65-1.35 (20H, m, H_{aliph}), 0.97 (15H, t, *J* 6.9, -CH₃); δ_{C} (50 MHz, CDCl₃) 149.23, 149.08, 148.90, 148.83, 145.94, 145.35, 124.03, 123.78, 123.72, 123.65, 123.32, 123.08, 107.6, 107.42, 107.25, 106.51, 104.39, 69.97, 69.94, 69.68, 69.21, 69.17, 29.30, 19.25, 29.16, 29.12, 28.51, 28.48, 28.45, 28.42, 22.71, 22.68, 22.67, 14.26, 14.23, 14.20.

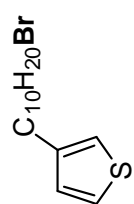
Scheme S3. Synthesis of polymer **PTT_n**



Conditions: a) **T**, K₂CO₃, DMF, 80°C for 1h, N₂ b) HT-poly(3-bromodecylthiophene) **PT** dissolved in THF, 130°C for 3 days

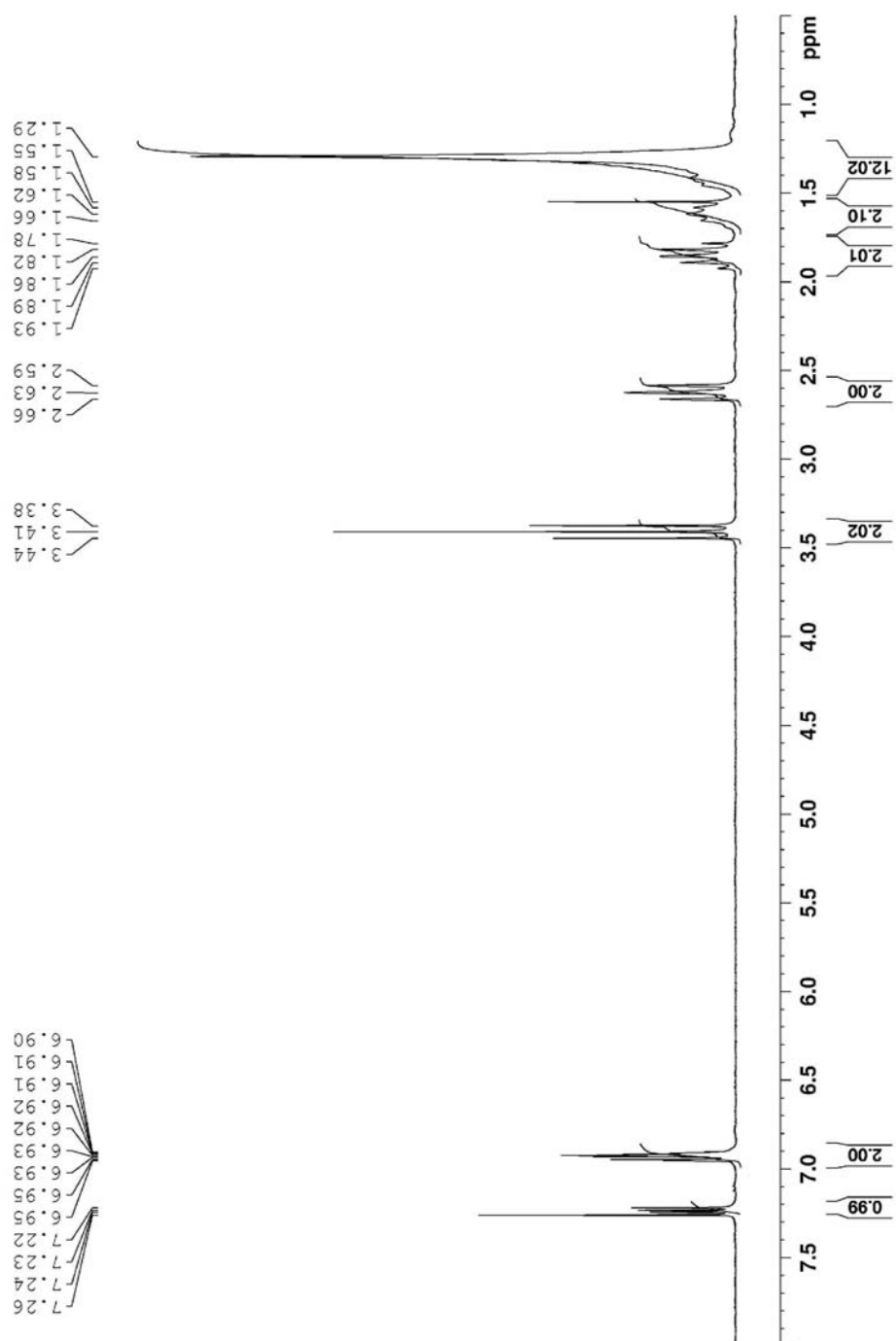
*Synthesis of HT poly[3-(10-(2,3,6,7,10-pentakis(pentyloxy)triphenyl)decyloxy)thiophene] (**PTT_n**)*

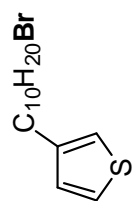
2,3,6,7,10-Pentakis(pentyloxy)-11-hydroxytriphenylene (0.45 g, 6.6 · 10⁻⁴ mol), potassium carbonate (0.18 g, 1.3 · 10⁻³ mol) in DMF (20 mL) was heated at 80°C for 1 h under an atmosphere of nitrogen. At which time, HT poly[3-(10-bromodecyl)thiophene] (0.1 g, 3.3 · 10⁻⁴ mol) in THF (15 mL) was added slowly to the green mixture. The resulting orange solution was left under heating at 130 °C for 3 days. The reaction mixture was then cooled down and filtered to remove the excess of base followed by extraction with chloroform (100 mL). The organic layer was successively washed with water (3 x 100 mL), dried over MgSO₄ and concentrated under reduced pressure. The crude product was precipitated using methanol (50mL) and the resulting purple solid was filtered through a Soxhlet thimble. The *title compound* was obtained (0.22 g, 73%) as a dark red powder after successive extraction with methanol; M.p. 107 °C (Lcol - Iso), δ_H (250 MHz, CDCl₃) 7.73 (6H, s, H_{arom}), 6.94 (1H, s, H_{arom}), 4.16 (12H, br. s, OCH₂), 2.76 (2H, br. s, CH₂-Ar), 1.90 (12H, br. s, H_{aliph}), 1.50-1.30 (34H, m, H_{aliph}), 0.93 (15H, br. s, -CH₃).



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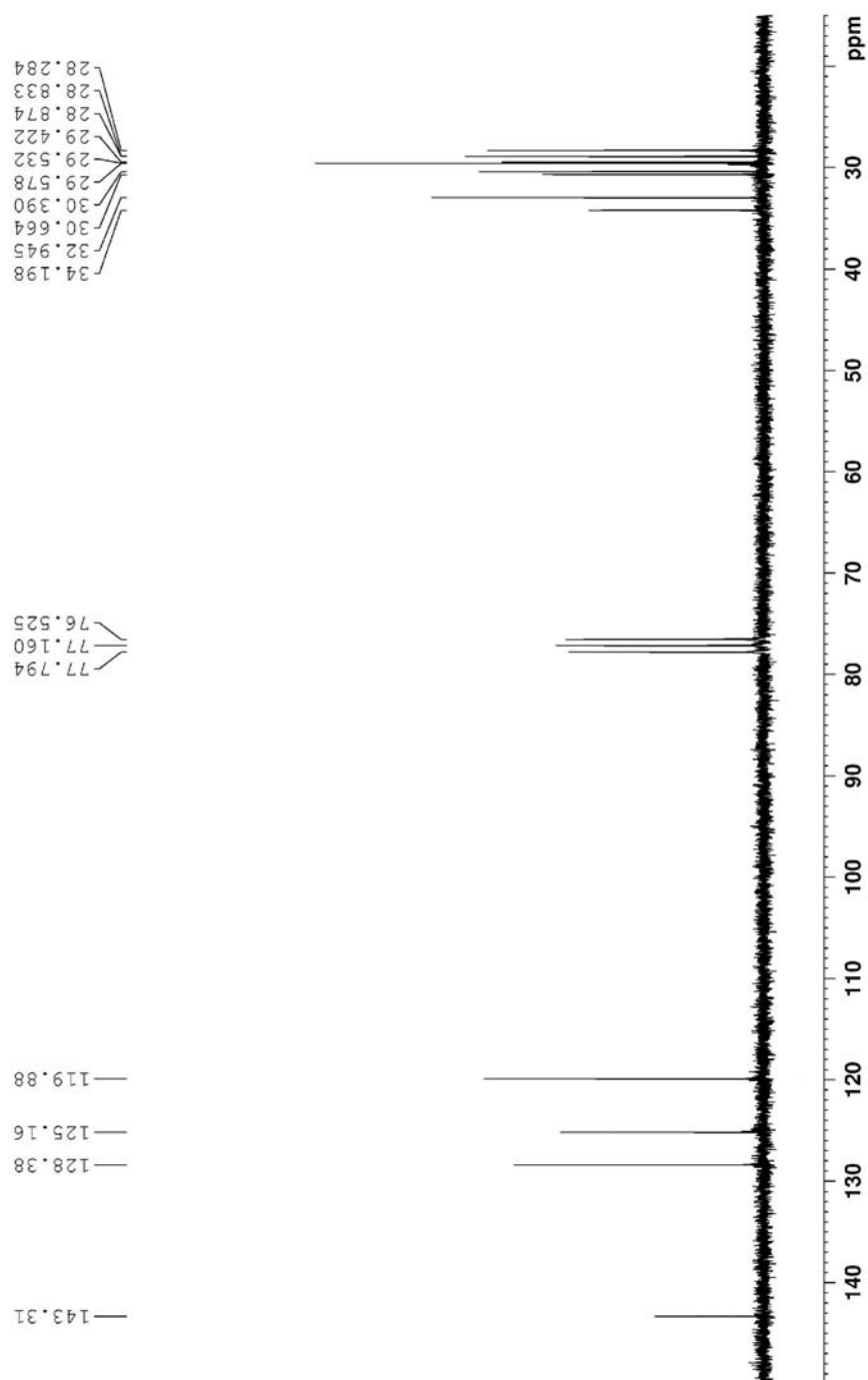
^1H NMR spectrum (200 MHz)

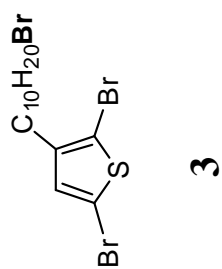




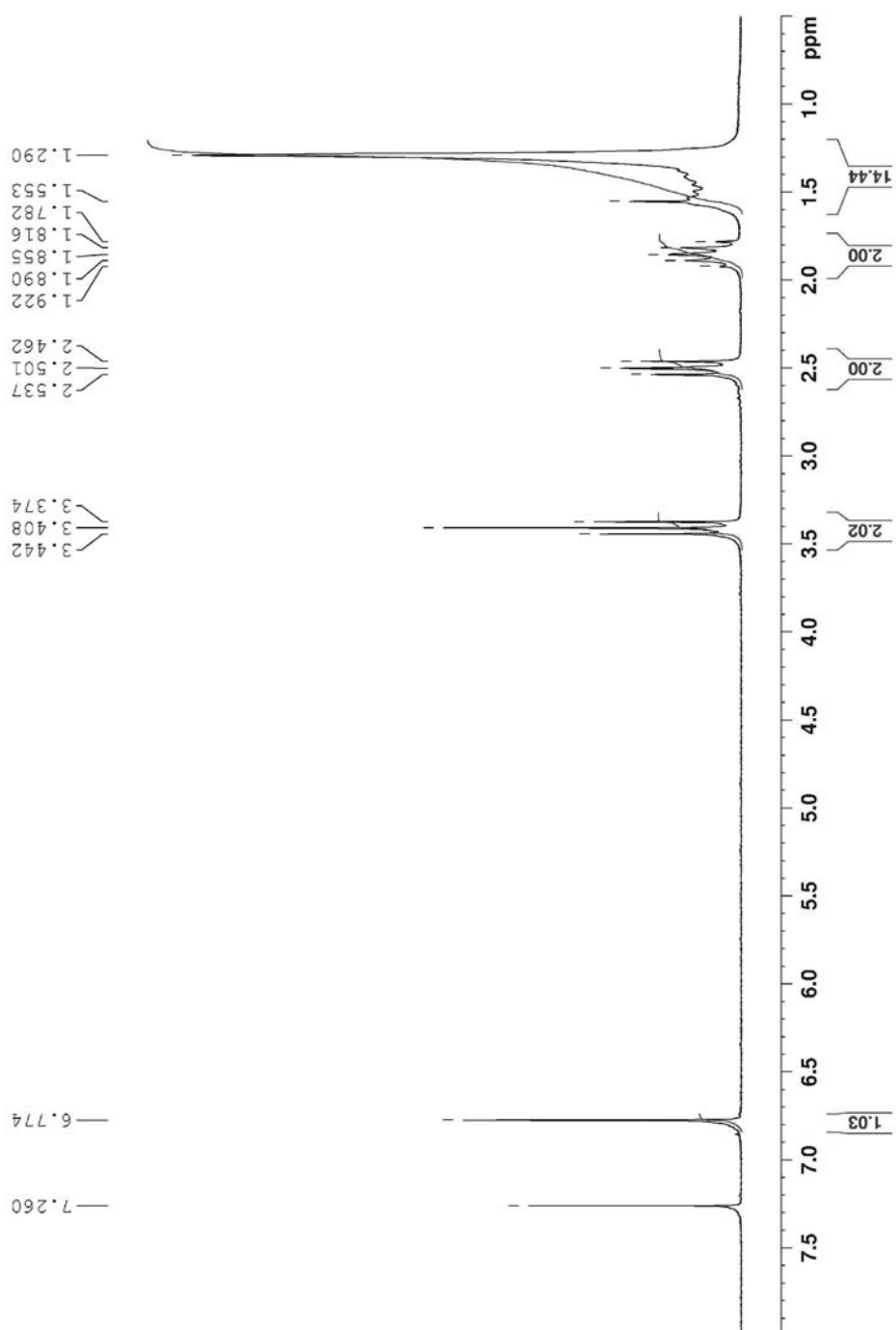
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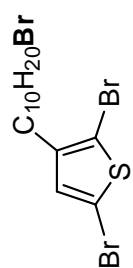
^{13}C NMR spectrum (50 MHz)





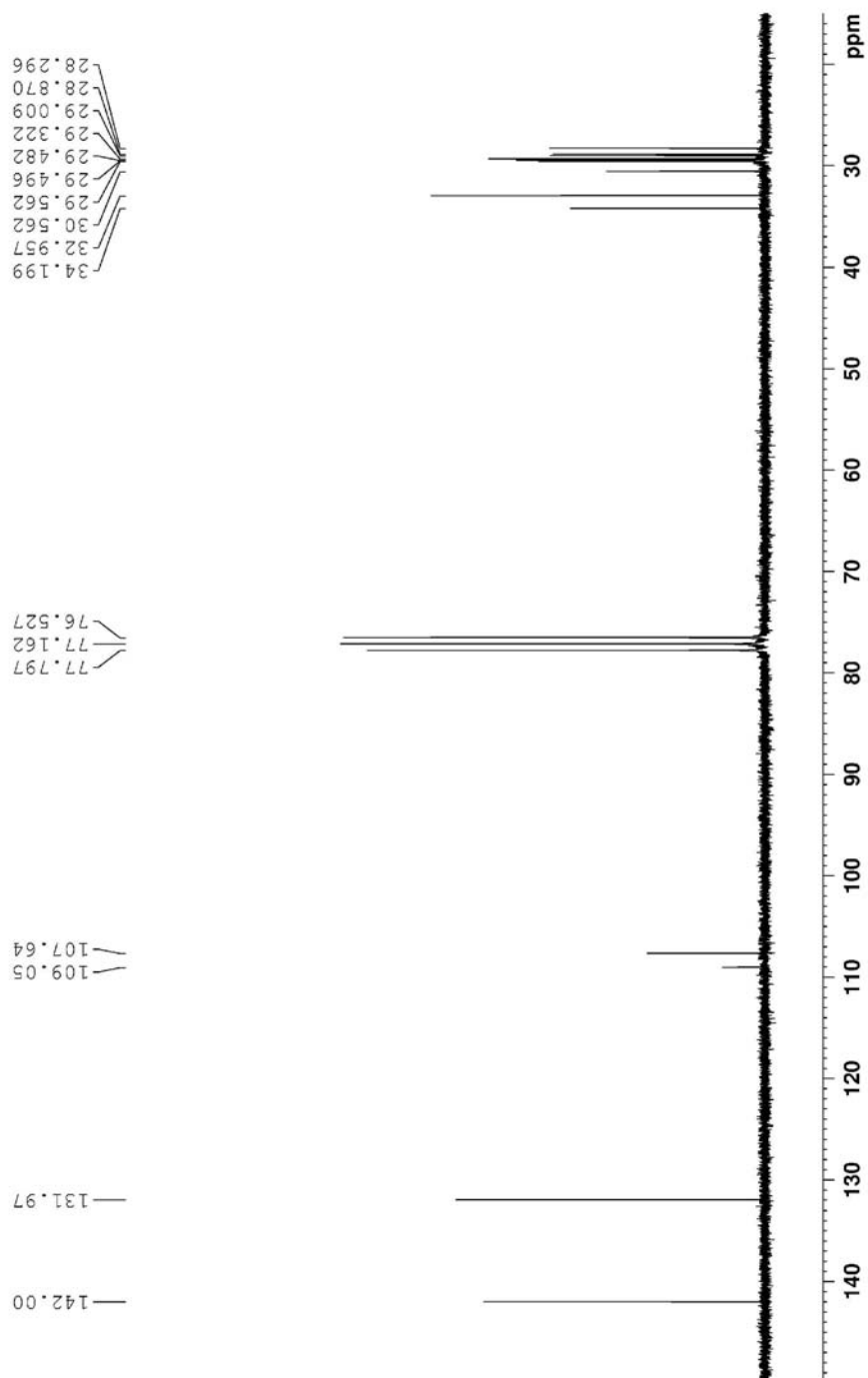
¹H NMR spectrum (200 MHz)

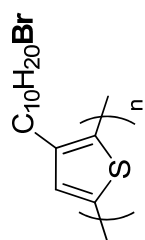




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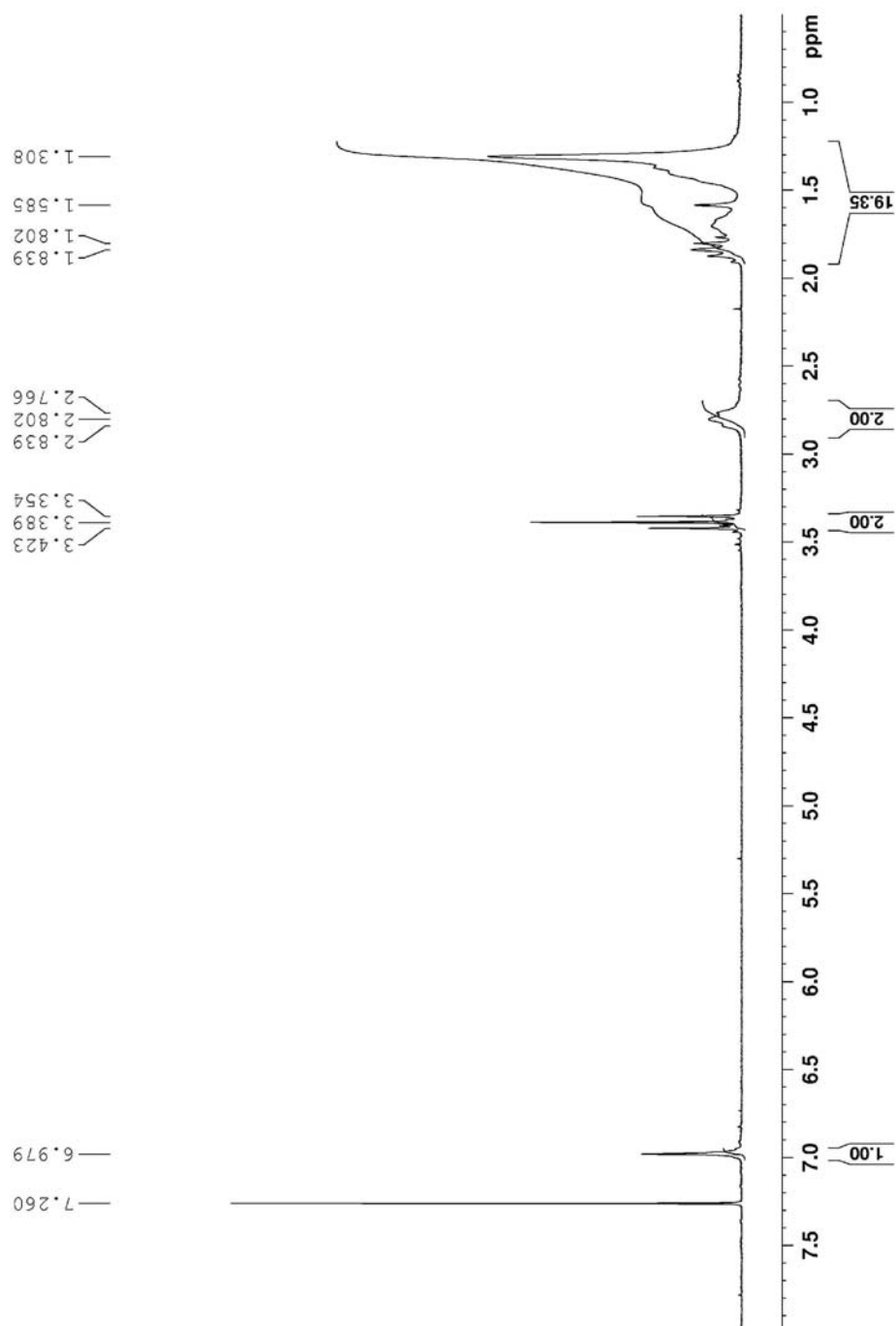
^{13}C NMR spectrum (75 MHz)

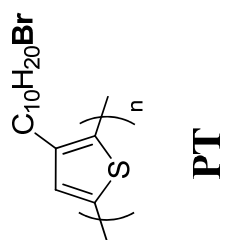




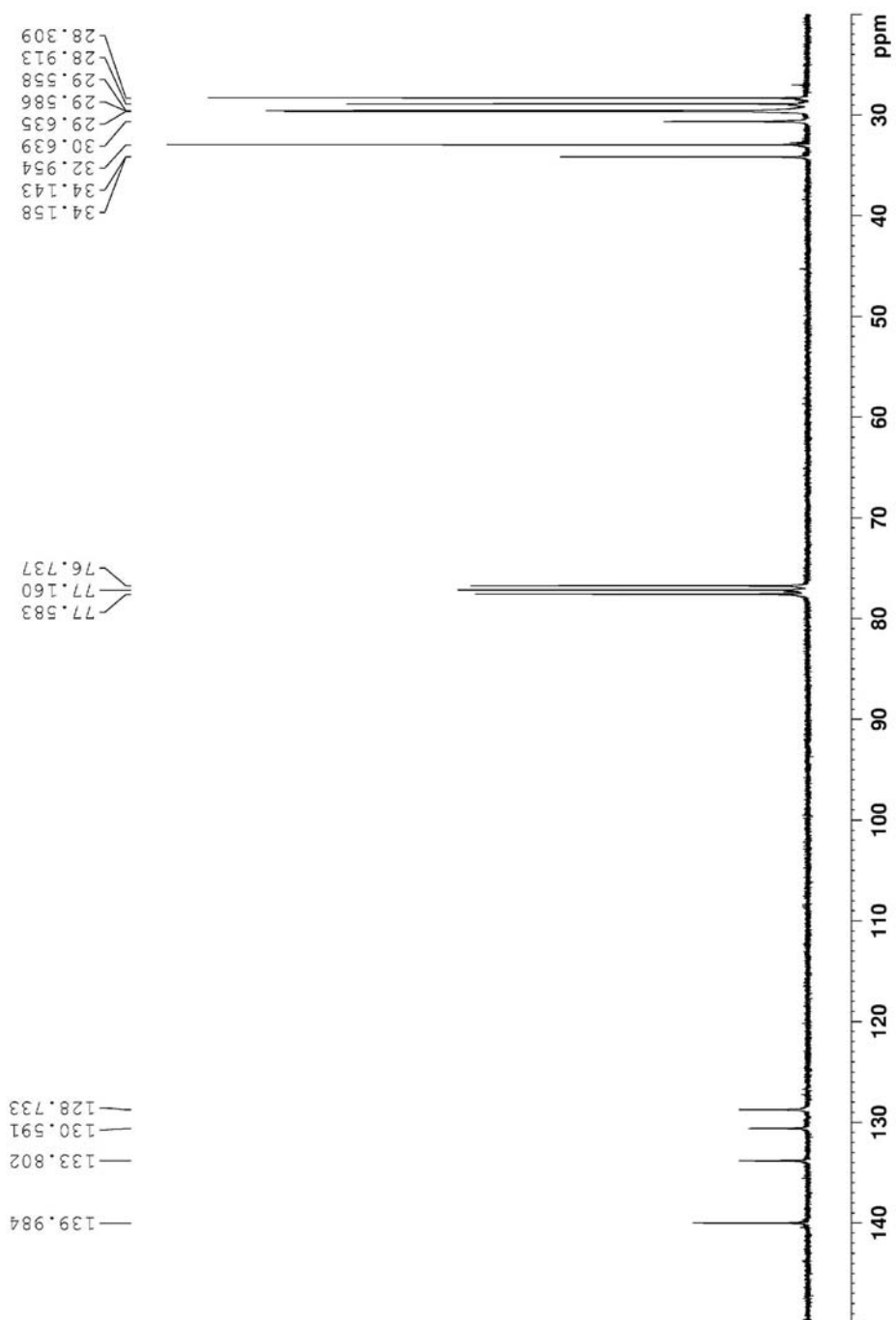
PT

^1H NMR spectrum (300 MHz)

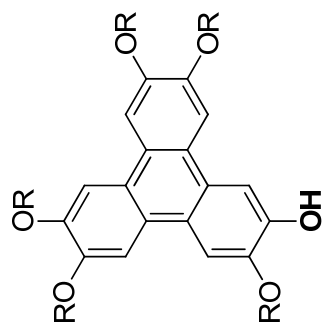




^{13}C NMR spectrum (75 MHz)

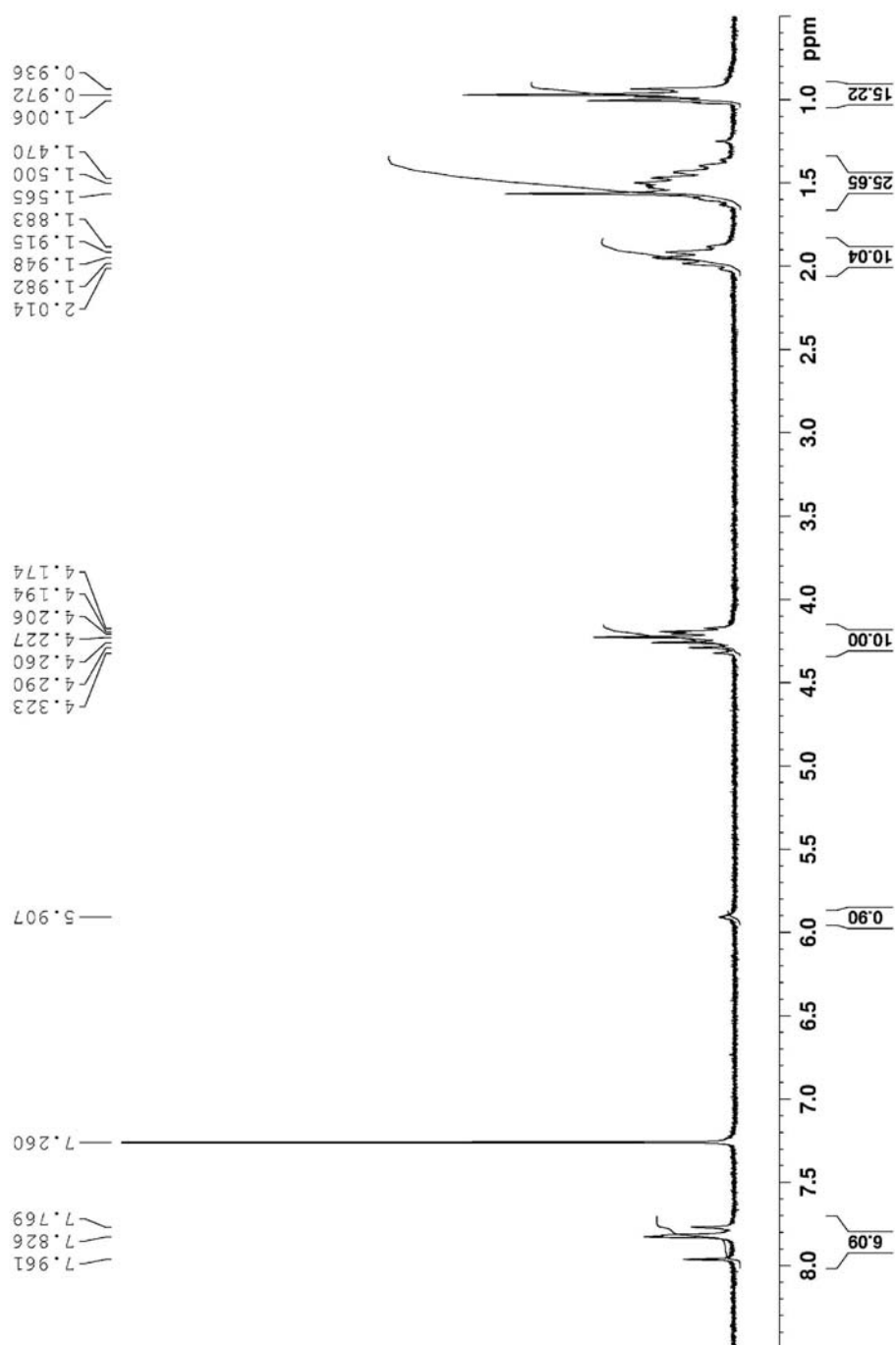


R = C₅H₁₁

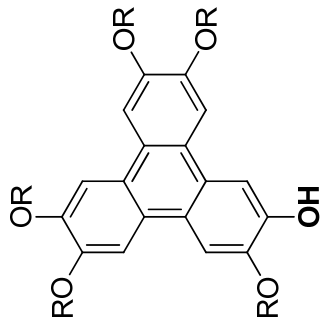


T

¹H NMR spectrum (200 MHz)

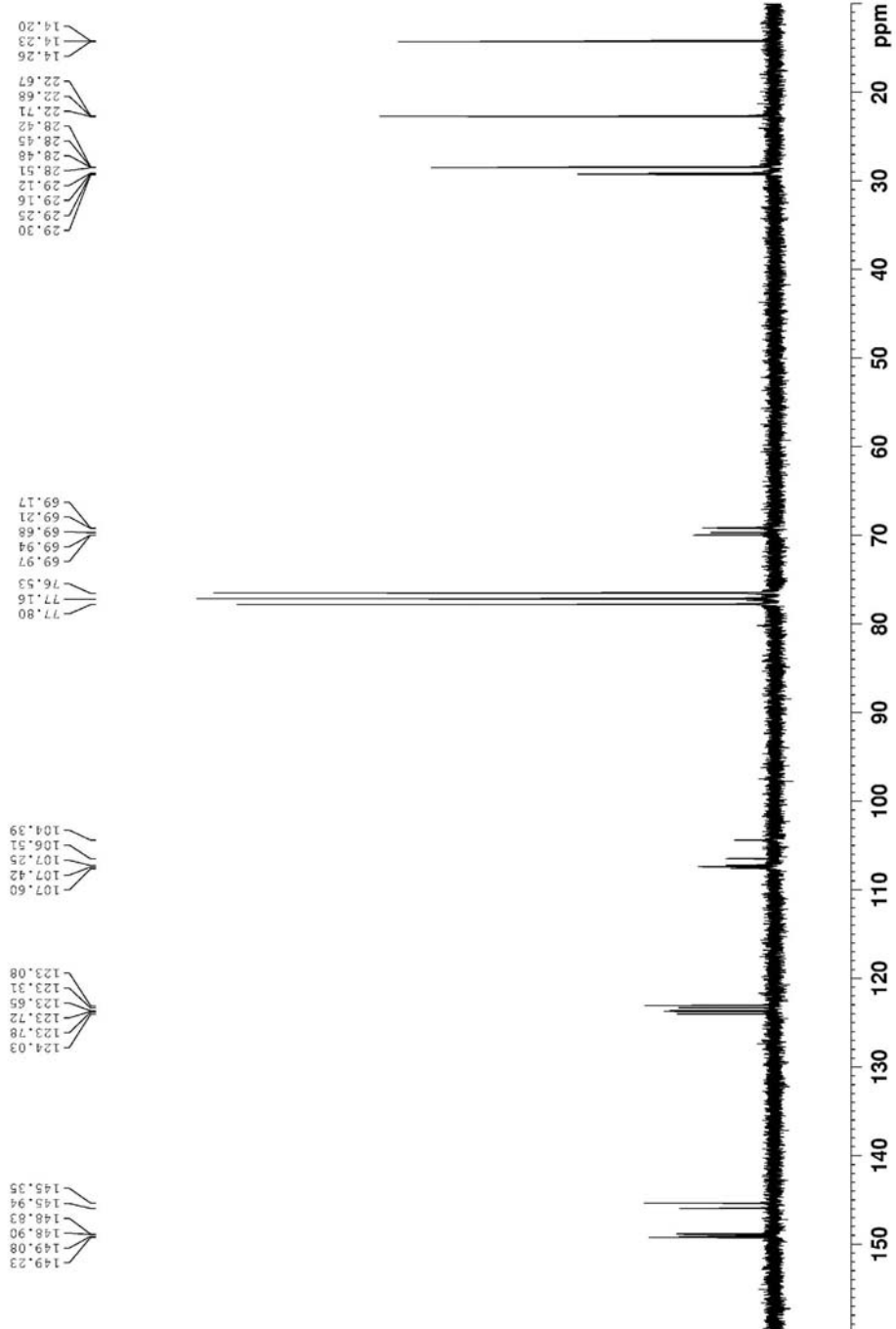


R = C₅H₁₁

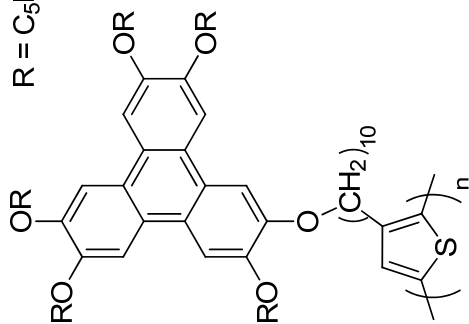


T

¹³C NMR spectrum (50 MHz)

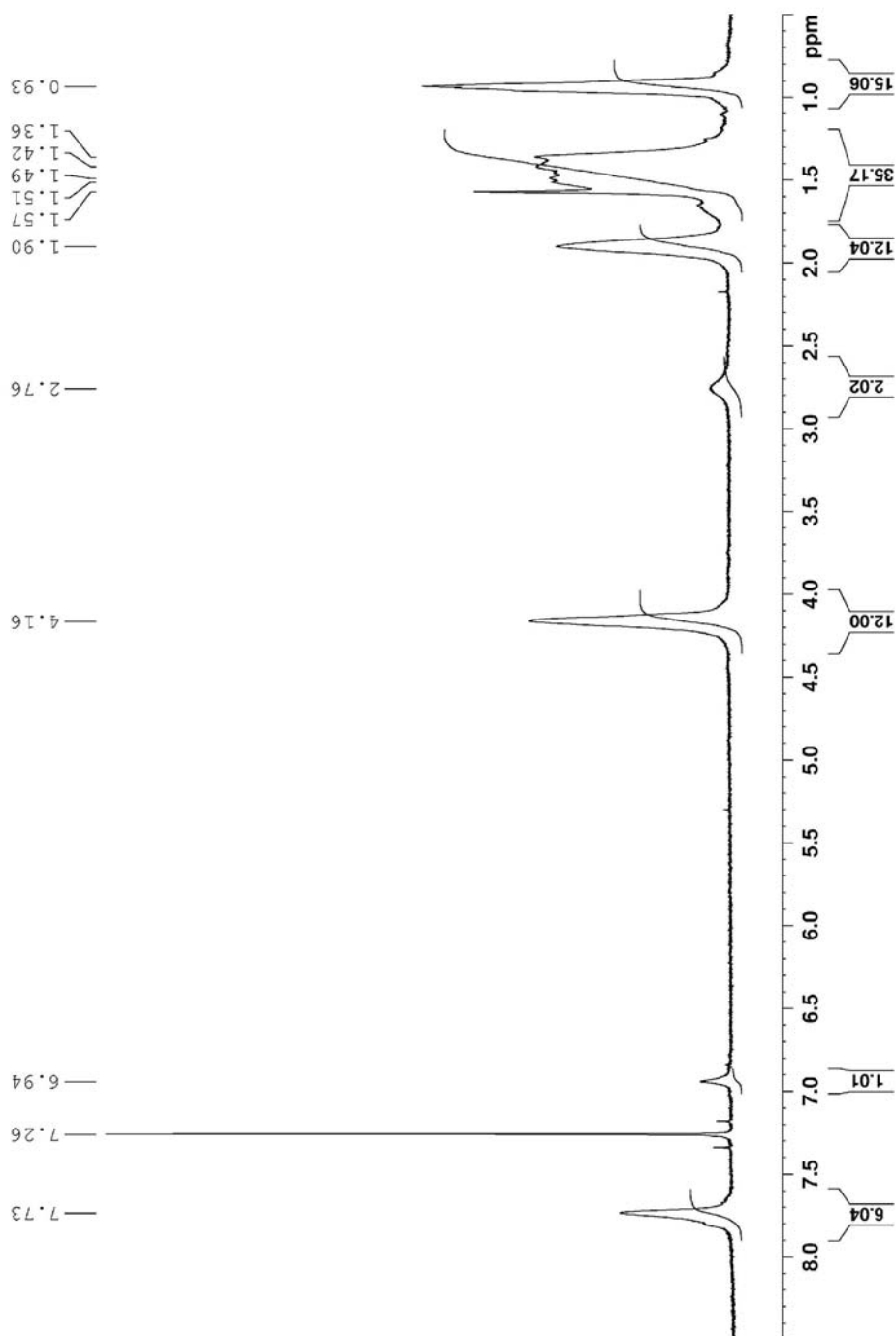


R = C₅H₁₁

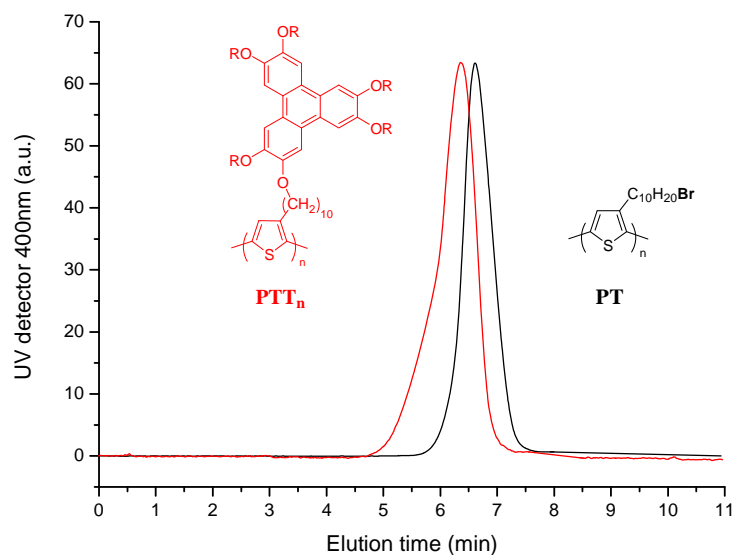


PTT_n

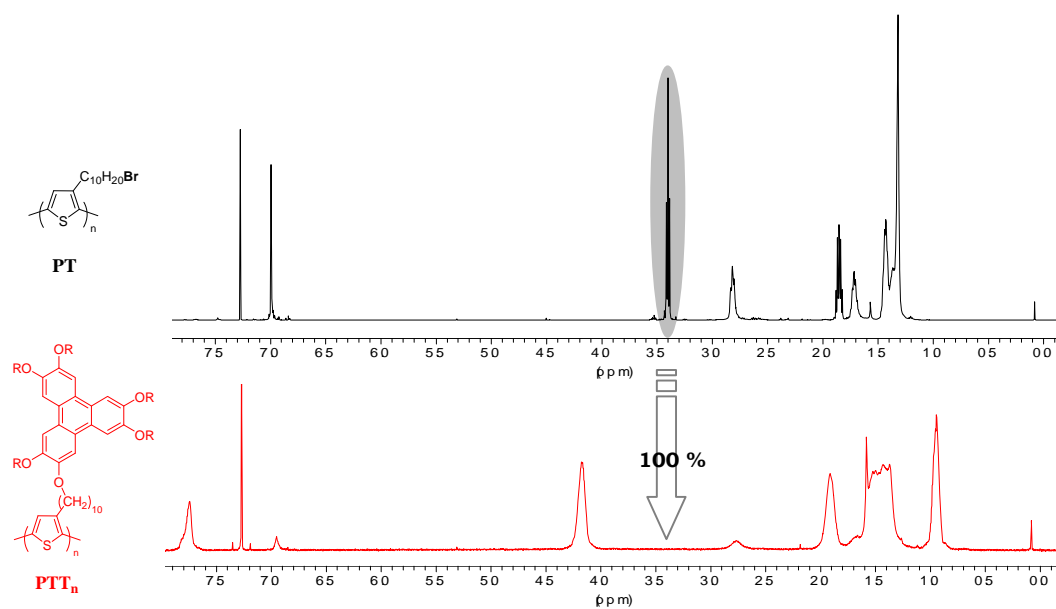
¹H NMR spectrum (250 MHz)



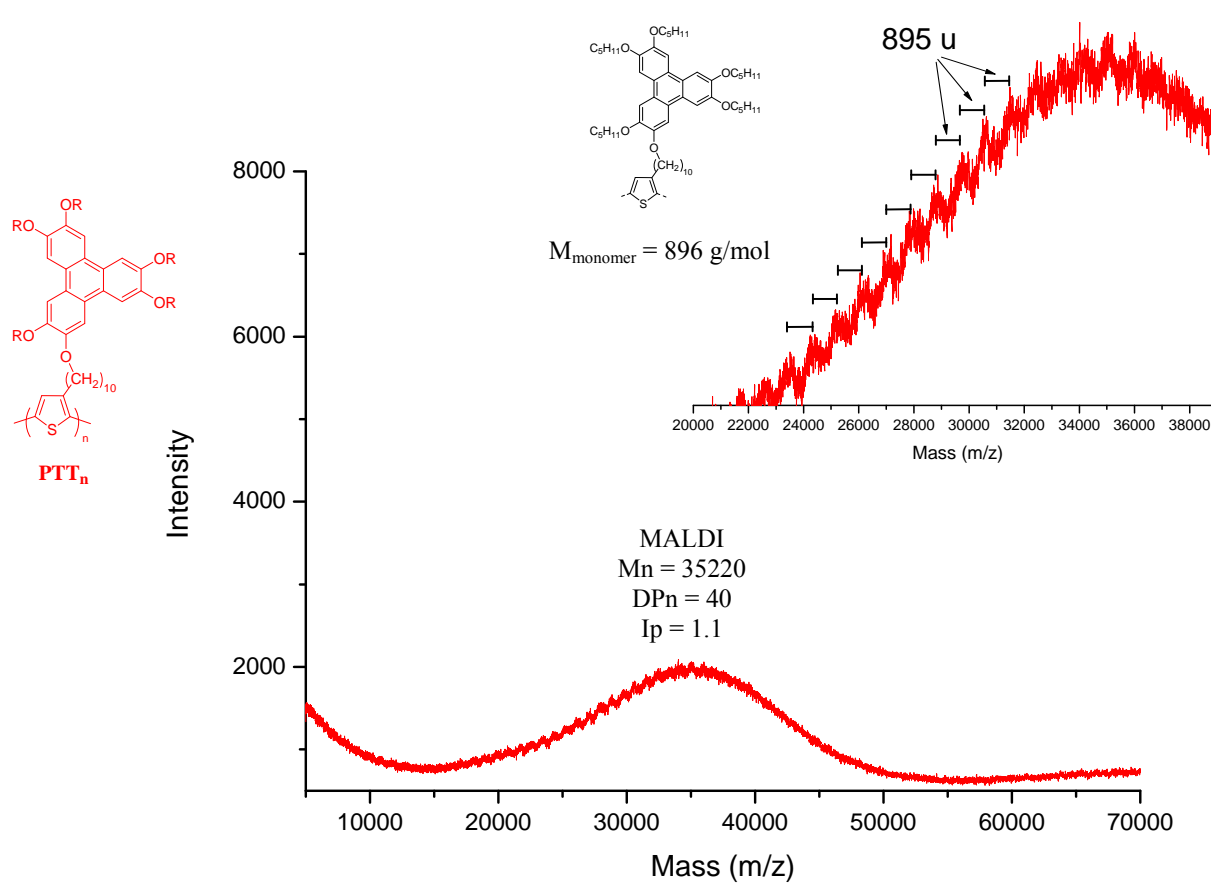
Scheme S4. GPC profiles of **PT** (black line) and **PTT_n** (red line) after complete substitution by the triphenylene derivatives.



Scheme S5. NMR spectra of **PT** (black) and **PTT_n** (red) after complete substitution by the triphenylene derivatives.



Scheme S6. MALDI-TOF mass spectrum of **PTT_n**



Scheme S7. Long time exposure X-ray powder diffraction patterns (Image plate profile) of discotic side-chain polymer **PTT_n** in the lamello-columnar Lcol phase at 50°C.

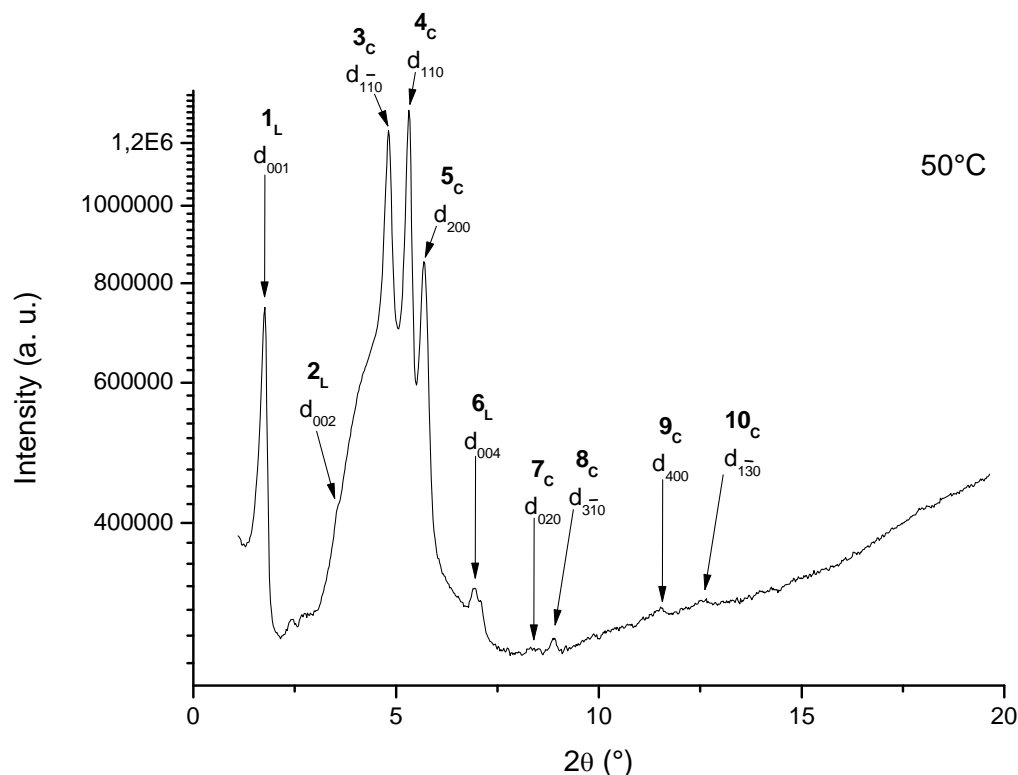


Table S2. Data collected from Powder-XRD Diffraction Patterns of polymer **PTT_n** in the lamello-columnar phase at 50°C.

^a reflection	^a 2θ _{meas} (°)	^b d _{meas} (Å)	^c I	hkl	^a 2θ _{calc} (°)	^b d _{calc} (Å)	assignment	Parameters
1	1.76	50.2	VS (sh)	001	1.74	50.8	lamellar lattice L	Lcol Oblique lattice a = 31.5 Å b = 21.1 Å γ = 97° S = 658 Å ² Z = 2 Lamellar lattice d = 50.8 Å
2	3.45	25.6	W (sh)	002	3.48	25.4	lamellar lattice L	
3	4.82	18.3	VS (sh)	1 $\bar{1}$ 0	4.79	18.43	Oblique lattice C	
4	5.32	16.6	VS (sh)	110	5.35	16.51	Oblique lattice C	
5	5.70	15.5	VS (sh)	200	5.65	15.62	Oblique lattice C	
6	7.0	12.7	W (sh)	004	6.95	12.7	lamellar lattice L	
7	8.4	10.5	VW (sh)	020	8.45	10.46	Oblique lattice C	
8	8.9	9.94	W (sh)	3 $\bar{1}$ 0	9.02	9.80	Oblique lattice C	
9	11.5	7.66	VW (sh)	400	11.32	7.81	Oblique lattice C	
10	12.5	7.07	VW (sh)	1 $\bar{3}$ 0	12.65	6.99	Oblique lattice C	
11	20	4.4	VS (br)				D	
12	25.3	3.5	M (br)				h ₀	

^a Reflection number (see Figure 2 and scheme S6 in the ESI). ^b d_{meas} and d_{calc} are the measured and calculated diffraction spacing. ^c I is the intensity of the reflections: VS, very strong; M, medium; W, weak; sh, sharp reflection; br broad reflection. hkl are the indexations of the reflections. D₀, lateral distances between chains and mesogens; h₀, distance between stacked plates.

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