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Conjugated polymers based on benzodithiophene and fluorinated quinoxaline for bulk heterojunction solar cells: thiophene *versus* thieno[3,2-*b*]thiophene as π -conjugated spacers

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Contents

Synthesis of monomers and polymersS2
Synthesis of 6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)-5,8-di(thiophen-2-yl)quinoxaline (2)S2
Synthesis of 5,8-bis(5-bromothiophen-2-yl)-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline (3)S3
Synthesis of 6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)-5,8-bis(thieno[3,2-b]thiophen-2-yl)quinoxaline (4) S3
Synthesis of 5,8-bis(5-bromothieno[3,2-b]thiophen-2-yl)-6,7-difluoro-2,3-bis(3- (octyloxy)phenyl)quinoxaline (5)S4
Synthesis of 5-ethynylundecane (7)S5
Synthesis of 4,8-bis(3-butylnon-1-yn-1-yl)benzo[1,2-b:4,5-b']dithiophene (8)S5
Synthesis 4,8-bis(3-butylnonyl)benzo[1,2- <i>b</i> :4,5- <i>b'</i>]dithiophene (9)S6
Synthesis of (4,8-bis(3-butylnonyl)benzo[1,2-b:4,5-b']dithiophene-2,6-diyl)bis(trimethylstannane) (10)S7
Synthesis of PBDTFQ-T
Synthesis of PBDTFQ-TT
NMR spectraS10
DFT calculations
Device optimizations
References

Synthesis of monomers and polymers

5,8-Dibromo-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline $(1)^1$ and 4,8-bis(3-butylnonyl)benzo[1,2-*b*:4,5-*b*]dithiophene-2,6-diyl)bis(trimethylstannane) $(10)^2$ was prepared according to previously reported procedures. Tetrahydrofuran (THF) was dried over Na/benzophenone and freshly distilled prior to use. Other reagents and solvents were in commercial grade and used as received without further purification.

Synthesis of 6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)-5,8-di(thiophen-2-yl)quinoxaline (2)



5,8-Dibromo-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline (1) (1.3 g, 1.775 mmol), $Pd_2(dba)_3$ (38 mg), P(o-tolyl)₃ (45 mg) were dissolved in THF (35 mL) and heated to reflux. 2-(Tributylstannyl) thiophene (1.66 g, 1.41 mL, 4.44 mmol) was added drop by drop to the reaction mixture. The reaction mixture was heated overnight. The solvent was removed and hexane was added to the crude product. The solid formed was separated by suction filtration and recrystallized from isopropanol to yield compound **2** (0.8 g, 62%); MALDI (m/z) 741.45 (M+).

¹H NMR (400 MHz, CDCl₃, δ): 8.05 (1H, d, *J*= 4 Hz), 7.62 (1H, d, *J*= 4 Hz), 7.36 (1H, s), 7.26 (4H, m), 6.95 (1H, m), 3.92 (2H, t), 1.76 (2H, m), 1.43 (10H, m), 0.90 (3H, t).

¹³C NMR (100 MHz, CDCl₃, δ): 159.01, 139.23, 130.80, 130.74, 129.90, 129.19, 126.60, 126.58, 122.73, 116.59, 115.61, 109.99, 68.12, 31.83, 29.33, 29.99, 29.13, 26.05, 22.68.

Synthesis of 5,8-bis(5-bromothiophen-2-yl)-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline (3)



6,7-Difluoro-2,3-bis(3-(octyloxy)phenyl)-5,8-di(thiophen-2-yl)quinoxaline (**2**) (0.8 g, 1.08 mmol) was dissolved in THF (40 mL) and *n*-bromosuccinimide (NBS) (0.38 g, 2.16 mmol) was added. After being stirred at room temperature and in the dark for 3 hours, the mixture was poured onto water and the orange solid formed was collected by filtration. The solid was recrystallized from isopropanol to yield the desired compound as fluffy orange solid (**3**) (0.5 g, 52%); MALDI (m/z) 895.89 (M+).

¹H NMR (400 MHz, CDCl₃, δ): 7.77 (1H, d, *J*= 4 Hz), 7.51 (1H, s), 7.23 (1H, t, *J*=8 Hz), 7.16 (1H, d, *J*= 4 Hz), 7.08 (1H, d, *J*= 8 Hz), 7.00 (1H, dd, *J*= 4 Hz, 8 Hz), 4.05 (2H, t), 1.82 (2H, m), 1.51 (10H, m), 0.91 (3H, t).

¹³C NMR (100 MHz, CDCl₃, δ): 159.38, 151.62, 138.71, 132.37, 130.86, 129.34, 129.08, 122.90, 118.88, 117.29, 115.09, 68.30, 31.85, 29.42, 29.32, 29.30, 26.17, 22.69, 14.13.

Synthesis of 6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)-5,8-bis(thieno[3,2-*b*]thiophen-2-yl)quinoxaline (4)



5,8-Dibromo-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline (1) (1.53g, 2.1 mmol), $Pd_2(dba)_3$ (44 mg, 0.048 mmol) and P(o-Tolyl)₃ (53 mg, 0.17 mmol) were dissolved in THF (45 mL) and refluxed. To the refluxing mixture, tributyl(thieno[3,2-*b*]thiophen-2-yl)stannane (2.28 g, 5.3 mmol) was added drop by drop. After being refluxed for about 24 hours, the reaction mixture

was concentrated on a rotary evaporator and hexane was added. The orange solid formed was collected by suction filtration to yield compound 4 (0.75 g, 42%); MALDI (m/z) 849.76 (M+).

¹H NMR (400 MHz, CDCl₃, δ): 8.24 (2H, s), 7.47 (2H, s), 7.23 (2H, d, *J*= 4Hz), 7.43 (2H, t), 7.30 (2H, d, *J*= 4 Hz), 7.28 (2H, t), 7.21 (2H, t), 6.98 (2H, m), 3.96 (4H, t), 1.78 (4H, m), 1.43-1.26 (20H, m), 0.91 (6H, t).

¹³C NMR (100 MHz, CDCl3, δ): 159.11, 151.35, 143.84, 139.13, 139.05, 132.99, 129.20, 128.81, 123.13, 122.81, 119.35, 116.75, 115.55, 109.99, 68.14, 31.83, 29.36, 29.30, 29.22, 26.12, 22.69, 14.12.

Synthesis of 5,8-bis(5-bromothieno[3,2-*b*]thiophen-2-yl)-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline (5)



NBS (0.31g, 1.76 mmol) was added to a solution of compound 4 (0.75 g, 0.88 mmol) in THF (35 mL) and the mixture was stirred in the dark and at room temperature for overnight. The volume of the THF was reduced on a rotary evaporator and water was added to the crude product. The orange solid formed was collected by filtration and recrystallized from isopropanol to give the desired compound as a fluffy orange solid (5) (0.7 g, 78.6%); MALDI (m/z) 1007.6 (M+).

¹H NMR (400 MHz, CDCl₃, δ): 8.04 (2H, s), 7.36 (2H, t), 7.28 (4H, m), 7.17 (2H, d, *J*= 8Hz), 6.96 (2H, d, *J*= 8 Hz), 3.94(4H, t), 1.81(4H, m), 1.45-1.31 (20H, m), 0.91 (6H, t).

¹³C NMR (100 MHz, CDCl₃, δ): 159.11, 151.38, 142.83, 139.21, 138.83, 132.29, 129.24, 122.79, 122.37, 122.13, 116.73, 115.63, 115.45, 68.16, 31.85, 29.39, 29.32, 29.23, 26.17, 22.69, 14.13.

Synthesis of 5-ethynylundecane (7)



1-Heptyne (6) (6.39 mL, 47.7 mmol) was dissolved in hexane (47 mL) and cooled in an ice bath. *n*-BuLi, 2.5 M in hexanes (42 mL, 105 mmol) was added gradually through a septum with a syringe over 100 minutes. During the addition of the *n*-BuLi, a thick mass of white solid was formed and extra hexane (15 mL) was added and the reaction mixture was shaken intermittently. As addition of the *n*-BuLi was continued, the solid gradually disappeared and a yellowish solution was formed. The reaction mixture was stirred in the bath for a total of 5 hours. 1-Bromohexane (6.84 mL, 47.7 mmol) was added drop by drop and the reaction mixture was stirred overnight. The reaction mixture (white suspension after overnight stirring) was further stirred for 1 day and quenched with 6M hydrochloric acid. The crude product was purified with column chromatography using hexane as eluent and was further purified with Kugelrohr distillation to yield compound **7** (2.38 g, 27.1%).

¹H NMR (400 MHz, CDCl₃, δ): 2.31-2.27 (1H, m), 2.2 (1H, d, *J* = 4Hz), 1.45-1.28 (20H, m), 0.91-0.86 (6H, m).

¹³C NMR (100 MHz, CDCl₃, δ):88.20, 68.85, 34.98, 34.69, 31.78, 31.47, 29.46, 29.16, 27.22, 22.62, 22.55, 14.05, 13.99.

Synthesis of 4,8-bis(3-butylnon-1-yn-1-yl)benzo[1,2-b:4,5-b']dithiophene (8)



Ethynylundecane (7) (2.38 g, 13.2 mmol) was dissolved in THF (18 mL) and stirred at room temperature under nitrogen atmosphere. *i*-PrMgCl, 2M in THF (6.3 mL, 12.6 mmol) was added drop by drop at room temperature and heated to 55 °C for 2 hours. The reaction mixture was cooled and benzo[1,2-*b*:4,5-*b*]dithiophene-4,8-dione (0.97 g, 4.4 mmol) was added. The reaction mixture

was heated at 55 °C for overnight. The reaction mixture was cooled to room temperature and $SnCl_2 2H_2O$ (6.4 g) in 10% HCl (16.1 mL) was added. After being heated at 55 °C for 2 hours, the reaction mixture was cooled to room temperature and poured on water. The organic compound was extracted with diethyl ether and dried with anhydrous sodium sulfate. The solvent was removed to yield a crude product which was dissolved in hot isopropanol and refrigerated. The solid formed was collected by filtration and washed with cold isopropanol. The solid was dried to give compound **8** (1.04 g, 43.2%); MALDI (m/z) 545.9 (M+).¹H NMR (400 MHz, CDCl₃, δ):7.56 (2H, d, *J* = 4Hz), 7.51 (2H, d, *J* = 4Hz), 2.78 (2H, m), 1.71-1.32 (32H, m), 0.97-0.89 (12H, m).

¹³C NMR (100 MHz, CDCl₃, δ):140.23, 138.12, 127.53, 123.17, 112.18, 103.92, 78.20, 35.30, 34.98, 32.95, 31.85, 29.79, 29.23, 27.57, 22.67, 22.63, 14.13, 14.12.

Synthesis 4,8-bis(3-butylnonyl)benzo[1,2-*b*:4,5-*b'*]dithiophene (9)



4,8-Bis(3-butylnon-1-yn-1-yl)benzo[1,2-*b*:4,5-*b*']dithiophene (**8**) (1.5 g, 2.94 mmol) was dissolved in THF (50 mL), to which 10% Pd/C (0.31 g, 0.29 mmol) was added. The mixture was stirred under a hydrogen atmosphere at room temperature for 22 hours. After filtration, the solvent was removed and purified by column chromatography using hexane as eluent to yield compound **9** (1.04 g, 70.1%); MALDI (m/z) 545.9 (M+). ¹H NMR (400 MHz, CDCl₃, δ): 7.46 (4H, s), 3.16 (4H, m), 1.76 (4H, m), 1.51-1.31 (34H, m), 0.95 (12H, m).

¹³C NMR (100 MHz, CDCl₃, δ):137.15, 135.70, 129.23, 125.88, 121.67, 37.88, 33.57, 33.29, 33.24, 31.97, 30.71, 29.80, 28.99, 26.71, 23.17, 22.72, 14.22, 14.16.

Synthesis of (4,8-bis(3-butylnonyl)benzo[1,2-*b*:4,5-*b'*]dithiophene-2,6-diyl)bis(trimethylstannane) (10)



4,8-Bis(3-butylnonyl)benzo[1,2-*b*:4,5-*b*']dithiophene (**9**) (1.04 g, 2.00 mmol) was dissolved in dry THF (20 mL) in nitrogen atmosphere. The solution was cooled to -78 °C and *n*-BuLi, 2.5 M in THF (5.5 mmol, 2.2 mL) was added drop-wise. After stirring at -78 °C for 30 minutes, the bath was removed and stirred for 30 minutes. Then the solution was cooled to -78 °C again and trimethyl tin chloride, 1 M in hexane (6 mL, 6 mmol) was added. The reaction mixture was stirred for about 24 hours and the temperature was allowed to rose to room temperature gradually. The reaction mixture was poured on water and extracted with hexanes. The organic extract was washed with distilled water and dried with anhydrous sodium sulfate. The solid formed upon removal of the solvent was recrystallized with isopropanol to give compound **10** (1.22 g, 73.3%); MALDI (m/z) 884.59 (M+). ¹H NMR (400 MHz, CDCl₃, δ): 7.49 (2H, s), 3.17 (4H, m), 1.77 (4H, m), 1.43-1.31(34H, m), 0.96 (12H, m), 0.52 (18H, s).

¹³C NMR (100 MHz, CDCl₃, δ):141.37, 140.10, 136.64, 129.62, 127.74, 37.78, 33.60, 33.30, 33.02, 32.06, 30.56, 29.86, 29.06, 26.76, 23.21, 22.75, 14.26, 14.17, -8.39.

Synthesis of PBDTFQ-T



PBDTFQ-T

5,8-Bis(5-bromothiophen-2-yl)-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline (3) (0.179 g, 0.2 (4,8-bis(3-butylnonyl)benzo[1,2-b:4,5-b]dithiophene-2,6mmol) and diyl)bis(trimethylstannane) (6) (0.176 g, 0.2 mmol) were dissolved in toluene (8 mL), which was aerated with nitrogen gas for 15 minutes. Pd₂(dba)₃ (4 mg) and P(o-Tolyl)₃ (6 mg) were added and aerated with nitrogen gas for 25 minutes and heated at 90 °C for 30 minutes which resulted in a viscous solution. The polymer was added to methanol and stirred. The solid was separated by filtration and redissolved in chloroform by heating at 60 °C for 1 hour. 10% Sodium dithiocarbamate trihydrate aqueous solution (100 mL) was added and stirred overnight at room temperature. The polymer solution was washed with distilled water. The chloroform solution was concentrated to a small volume and precipitated on methanol. The solid was collected by filtration and subjected to Soxhlet extraction successively with methanol, hexane, diethyl ether, dichloromethane and finally chloroform. The chloroform solution was reduced to small volume and precipitated and the solid was isolated by filtration and dried to give **PBDTFQ-T** (242 mg, 94%).

¹H NMR (400 MHz, CDCl₃, δ):7.5-6.5 (aromatic protons), 4.0-3.5 (-OCH₂-), 2.0-0.5 (aliphatic protons).

Synthesis of PBDTFQ-TT



5,8-Bis(5-bromothieno[3,2-b]thiophen-2-yl)-6,7-difluoro-2,3-bis(3-(octyloxy)phenyl)quinoxaline (5) (0.202 g, 0.2 mmol) and (4,8-bis(3-butylnonyl)benzo[1,2-b:4,5-b']dithiophene-2,6diyl)bis(trimethylstannane) (6) (0.176 g, 0.2 mmol) were dissolved in toluene (8 mL) and DMF (0.5 (mL). The mixture was heated to 75 °C for complete dissolution of the monomers and nitrogen gas was bubbled for 10 minutes. $Pd_2(dba)_3$ (4 mg) and $P(o-Tolyl)_3$ (6 mg) were added and aerated with nitrogen gas for 25 minutes and heated at 90 °C for 30 minutes. The polymer was added to methanol and stirred. The solid was separated by filtration and redissolved in chloroform by heating at 60 °C for 1 hour. 10% Sodium dithiocarbamate trihydrate aq. solution (100 mL) was added and stirred overnight at room temperature. The polymer solution was washed with distilled water. The chloroform solution was concentrated to a small volume and precipitated on methanol. The solid was separated and subjected to Soxhlet extraction successively with methanol, hexane, diethyl ether, dichloromethane and finally chloroform. The chloroform solution was reduced to small volume and precipitated and the solid was isolated by filtration and dried to give **PBDTFQ-TT** (106 mg, 37.8%).

¹H NMR (400 MHz, CDCl₃, δ):7.5-6.5 (aromatic protons), 4.0-3.5 (-OCH₂-), 2.0-0.5 (aliphatic protons).

NMR spectra



Figure S1. ¹H NMR spectrum (400 MHz, CDCl₃) of compound 1.



Figure S2. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound **1**.



Figure S3. ¹H NMR spectrum (400 MHz, CDCl₃) of compound 2.



Figure S4. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 2.



Figure S5. ¹H NMR spectrum (400 MHz, CDCl₃) of compound 3.



Figure S6. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 3.



Figure S7. ¹H NMR spectrum (400 MHz, CDCl₃) of compound 4.



Figure S8. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 4.



Figure S9. ¹H NMR spectrum (400 MHz, CDCl₃) of compound **5**.



Figure S10. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 5.



Figure S11. ¹H NMR spectrum (400 MHz, CDCl₃) of compound **7**.



Figure S12. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 7.



Figure S13. ¹H NMR spectrum (400 MHz, CDCl₃) of compound 8.



Figure S14. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 8.



Figure S15. ¹H NMR spectrum (400 MHz, CDCl₃) of compound **9**.



Figure S16. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 9.



Figure S17. ¹H NMR spectrum (400 MHz, CDCl₃) of compound 10.



Figure S18. ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 10.



Figure S19. ¹H NMR spectrum (400 MHz, CDCl₃) of **PBDTFQ-T**.



Figure S20. ¹H NMR spectrum (400 MHz, CDCl₃) of PBDTFQ-TT.

DFT calculations



Figure S21. Optimized geometries for the two-repeating-unit models calculated by DFT. The structures in the frames show the preferred conformations for the two polymers.



Figure S22. Illustrations of the frontier orbitals of the polymers and their molecular geometries from side view (isovalue surface 0.02 au) evaluated by DFT at the B3LYP/6-31G(d) level.

Device optimizations

	Ratio (D/A)	Additives ^a	Annealing	$J_{\rm SC}$ (mA/cm ²)	$V_{\rm OC}$ (V)	FF (%)	PCE (%)
PBDTFQ-TT :PC ₆₁ BM Conc.tot. = 30mg/mL In ODCB	1:1	/	No	-10.87	0.82	59.11	5.29
		/	110°C (10 min)	-8.97	0.80	60.66	4.35
		/	110°C (20 min)	-7.90	0.81	64.26	4.10
		/	80°C (10 min)	-9.35	0.82	58.91	4.51
		3%	No	-9.53	0.75	44.64	3.19
		3%	110°C (10 min)	-10.11	0.78	48.72	3.84
	1:1.5	/	No	-9.27	0.81	62.07	4.62
		/	110°C (10 min)	-8.35	0.81	60.19	4.08
		3%	No	-10.09	0.75	52.70	3.99
		3%	110°C (10 min)	-9.04	0.77	52.19	3.64
	1.5:1	/	No	-10.48	0.83	58.16	5.05
		/	110°C (10 min)	-9.26	0.81	62.07	4.64
		3%	No	-10.93	0.77	50.76	4.25
		3%	110°C (10 min)	-9.53	0.78	56.64	4.21
PBDTFQ-TT	1:1	/	No	-6.56	0.76	45.35	2.25
C_{71} DM		/	110°C (10 min)	-6.32	0.80	52.14	2.64
30 mg/mL		3%	No	-9.45	0.75	46.43	3.29
In ODCB		3%	110°C (10 min)	-9.63	0.76	50.63	3.69
σρητεή τ		/	No	-5.46	0.87	45.92	2.18
PBDTFQ-T :PC ₆₁ BM		/	110°C (10 min)	-4.33	0.88	53.92	2.05
Conc.tot. = 20mg/mI	1:1	3%	No	-9.18	0.84	66.53	5.30
In ODCB		3%	110°C (5 min)	-8.52	0.83	64.62	4.59
-		3%	140°C (10 min)	-7.52	0.84	68.52	4.32
		3%	110°C (10 min)	-7.98	0.81	66.40	4.31

Table S1. Device performances at different processing conditions

	1:1.5	/	No	-5.09	0.88	64.88	2.89
		/	110°C (10 min)	-4.72	0.87	64.02	2.63
		3%	No	-9.29	0.81	67.81	5.12
-		3%	110°C (10 min)	-6.64	0.81	58.18	3.12
		/	No	-1.94	0.85	35.51	0.59
	1 5.1	/	110°C (10 min)	-1.87	0.86	39.84	0.64
	1.011	3%	No	-9.40	0.84	60.07	4.76
		3%	110°C (10 min)	-8.64	0.85	66.64	4.91
	1:2	/	No	-4.44	0.87	67.75	2.61
		/	110°C (10 min)	-3.97	0.85	68.71	2.32
		3%	No	-8.30	0.80	63.79	4.25
		3%	110°C (10 min)	-5.78	0.82	68.15	3.23
	2.1	/	No	-3.32	0.85	31.42	0.88
		/	110°C (10 min)	-1.95	0.81	35.75	0.57
		3%	No	-6.19	0.86	54.22	2.87
		3%	110°C (10 min)	-2.81	0.87	51.42	1.26
PBDTFQ-T $:PC_{71}BM$	1:1	/	No	-1.68	0.82	45.70	0.63
		/	110°C (10 min)	-1.91	0.84	52.00	0.83
20mg/mL		3%	No	-6.92	0.78	57.35	3.10
In ODCB		3%	110°C (10 min)	-7.08	0.80	67.68	3.83

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