Electronic Supplementary Information

The interplay of conformation and photophysical properties in deep-blue fluorescent oligomers

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Materials and Characterisation. Materials obtained from commercial suppliers were used without further purification. Solvents were dried and degassed following standard procedures. ¹H NMR and ¹³C NMR spectra were recorded on Bruker Avance 400 or Varian VNMRS 700 spectrometers with TMS as an internal standard. MALDI-TOF mass spectra were measured on an Applied Biosystems Voyager-DE STR operating in reflector mode. Elemental analyses were performed on a CE-400 Elemental Analyser. Melting points were determined in open-ended capilliaries using a Stuart Scientific SMP3 melting point apparatus at a ramping rate of 5 °C/min and are quoted to the nearest 0.5 °C. Cyclic voltammetry experiments were performed on a BAS-CV50W electrochemical workstation with Auto iR compensation, using Ag/Ag⁺ (BAS-MF-2052, Ag/AgCl with Vycor Tip), PTE platinum voltammetry electrode (BAS-MF-2013) and platinum wire (BAS-MW-4130) as the reference, working and auxiliary electrodes, respectively. Oxidation potentials were measured in dry dichloromethane solution (0.2 M n-Bu₄NPF₆). The concentrations of the samples were ca 2-7 x 10⁻⁴ M. Scan rates were 100 and 50 mV s⁻¹ for oxidation and reduction scans, respectively.

Absorption spectra were obtained using a Perkin Elmer Lambda 19 double beam spectrophotometer in 1 cm path length quartz cells. Steady state photoluminescence spectra were recorded on a Fluorolog fluorescence spectrometer (Jobin Yvon) with double-excitation, double-emission monochromators using a right angle configuration. Time resolved fluorescence decays were collected using the picosecond time

correlated single photon counting technique (impulse response function, IRF = 21 ps). The excitation source, with vertical polarization, was a picosecond Ti:Sapphire laser from Coherent Inc., (wavelength range: 720 to 1000 nm, 76 MHz repetition rate) coupled to a second harmonic generator (360 to 500 nm). Emission collected at magic angle polarisation was detected through a SpectraPro-2300i double subtractive monochromator (Acton Research Corporation) by a Hamamatsu model R3809U-50 microchannel plate (MCPT). Signal acquisition was performed using a TCSPC module from Becker & Hickl (Model SPC-630) using 4096 channels in a 0.8 ps/channel time scale. Deconvolution of the fluorescence decays was performed using the Globals WE[®] software package.¹ Random flat residuals between [-2,2] were obtained in all cases with χ^2 values close to unity. Time resolved spectra were acquired using a streak camera model C5680 from Hamamatsu, having 10 ps IRF.

Work performed in solution was carried out using dilute solutions with optical density below 0.2 at the maximum absorption wavelength, corresponding to concentrations of $\sim 10^{-6}$ mol L⁻¹. The solvents with super purity grade were purchased from ROMIL[®] and used as supplied. Amorphous solid thin films of compounds **1-8** were spin coated from toluene solution with concentrations of ca. 10 mg/mL.

Synthesis. Compounds $\mathbf{1}^2$, $\mathbf{2}^3$, \mathbf{D}^2 , \mathbf{E}^4 , \mathbf{J}^5 and \mathbf{P}^2 were prepared according to literature routes. Compound **A** was purchased from Aldrich.

Synthesis of Compound 3 (Scheme 1)



Scheme 1. Reagents, conditions and yields: i, *n*-BuLi (2.5 M in hexanes), THF, CuCl₂, -78 °C to 20 °C (23% yield); ii, *N*-bromosuccinimide, conc. H₂SO₄, 20 °C (43% yield); iii, **D** (2.4 equiv.), Pd(PPh₃)₄ (5 mol%), Na₂CO₃ (1 M), dioxane, 85 °C (50% yield).

3,7-Bis(9,9-dihexylfluoren-2-yl)-2,8-difluorodibenzothiophene-*S,S***-dioxide 3.** A white solid (0.85 g, 50%). M.p. 189.5-190.0 °C. ¹H NMR (400 MHz) CDCl₃ δ 8.03 (d, *J* = 8.0 Hz, 2H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.77-7.75 (m, 2H), 7.59-7.54 (m, 6H), 7.39-7.35 (m, 6H), 2.04-2.00 (m, 8H), 1.12-1.06 (m, 32H), 0.77 (t, *J* = 8.0 Hz, 12H). ¹³C NMR (400 MHz) CDCl₃ δ 164.5, 162.5, 151.7, 151.4, 142.4, 140.4, 134.8, 133.0, 132.9, 132.2, 128.0, 127.2, 125.2, 123.5, 123.2, 120.4, 120.2, 110.5, 110.3, 55.6, 40.6, 31.7, 29.9, 24.0, 22.8, 14.3. ¹⁹F NMR (400 MHz) CDCl₃ δ -107.1. MS-MALDI *m/z* 916.6 ([M⁺], 100%). Anal. Calcd. for C₆₂H₇₀F₂O₂S: C 81.18; H 7.69; F 4.14; S 3.50. Found: C 81.04; H 7.64; F 4.13; S 3.62. Crystals for X-ray analysis were obtained by recrystallisation from ethanol.

Synthesis of Compound 4 (Scheme 2).



Scheme 2. Reagents, conditions and yields: i, glacial acetic acid, H₂O₂ (30% solution), 40 °C (79% yield); ii, 1-(bromomethyl)cyclohexane (2.5 equiv.), K₂CO₃, *N*,*N*-dimethylacetamide, 120 °C (76% yield); iii,

glacial acetic acid, H_2O_2 (30% solution), 125 °C (80% yield); iv, bromine, AcOH:CHCl₃ (1:1 v/v), 70 °C (71% yield); v, **D** (3.0 equiv.), Pd₂(dba)₃, tricyclohexylphosphine, CsF (aq.), dioxane, 110 °C (51% yield).

3,7-Bis(9,9-dihexylfluoren-2-yl)-2,8-di(cyclohexylmethoxy)dibenzothiophene-*S*,*S*-dioxide **4** (**F**-S(OR)₂-**F**). A white solid. M.p. 179.0-180.0 °C. ¹H NMR (700 MHz, CDCl₃): δ 7.83 (s, 2H), 7.75-7.73 (m, 4H), 7.54 (dd, *J* = 7.7, 1.4 Hz, 2H), 7.51 (d, *J* = 1.4 Hz, 2H), 7.37-7.30 (m, 6H), 7.28 (s, 2H), 3.98 (d, *J* = 5.6 Hz, 4H), 2.04-1.96 (m, 8H), 1.85-1.64 (m, 12H), 1.28-1.23 (m, 4H), 1.18-1.01 (m, 30H), 0.76 (t, *J* = 7.0 Hz, 12H), 0.60-0.70 (m, 8H). ¹³C NMR (175 MHz, CDCl₃): δ 161.2, 151.2, 150.9, 141.04, 140.97, 135.7, 134.0, 132.5, 130.8, 128.6, 127.4, 127.0, 124.6, 123.8, 123.2, 120.0, 119.4, 104.8, 75.2, 68.2, 55.4, 40.6, 37.8, 31.8, 30.1, 30.0, 26.6, 25.9, 25.8, 24.0, 22.8, 14.2. MS (MALDI-TOF): 1104.7 (100%), 1105.7 (85%), 1106.6 (50%); (calcd. M⁺ 1104.7). Anal. Calcd. for C₇₆H₉₆O₄S: C, 82.56; H, 8.75; S, 2.90. Found: C, 82.48; H, 8.72; S, 2.84. Crystals for X-ray analysis were obtained by recrystallisation from a mixture of dichloromethane and cyclohexane (7:3 v/v).

Synthesis of Compound 5 (Scheme 3).



Scheme 3. Reagents, conditions and yields: i, *n*-BuLi (2.5 M in hexanes), B(OMe)₃, -78 °C to 20 °C, H₂SO₄ (5% aq.) (88% yield); ii, glacial acetic acid, H₂O₂ (30% solution), 40 °C (77% yield); iii, 1-

(bromohexane (2.0 equiv.), K₂CO₃, *N*,*N*-dimethylacetamide, 120 °C (75% yield); iv, glacial acetic acid, H₂O₂ (30% solution), 125 °C (76% yield); v, bromine, AcOH:CHCl₃ (1:1 v/v), 70 °C (74% yield); vi, **P** (0.5 equiv.), Pd₂(dba)₃, tricyclohexylphosphine, CsF (aq.), dioxane, 110 °C (53% yield).

2,7-Bis(2-hexyloxy-*S,S***-dioxide-dibenzothiophen-3-yl)-9,9-dihexylfluorene 5** (S(OR')-F-S(OR')). A white solid. M.p. 222.5-223.5 °C. ¹H NMR (700 MHz, CDCl₃): δ 7.85 (s, 2H), 7.83 (d, *J* = 7 Hz, 2H), 7.80 (d, *J* = 7.7 Hz, 2H), 7.77 (d, *J* = 7.7 Hz, 2H), 7.64 (td, *J* = 7.7, 0.7 Hz, 2H), 7.547 (d, *J* = 1.4 Hz, 2H), 7.54-7.51 (m, 4H), 7.30 (s, 2H), 4.14 (t, *J* = 7 Hz, 4H), 2.04-2.01 (m, 4H), 1.83-1.78 (m, 4H), 1.45-1.40 (m, 4H), 1.34-1.28 (m, 8H), 1.14-1.02 (m, 12H), 0.87 (t, *J* = 7 Hz, 6H), 0.757 (t, *J* = 7.7 Hz, 6H), 0.72-0.65 (m, 4H). ¹³C NMR (175 MHz, CDCl₃): δ 161.0, 151.0, 140.7, 139.0, 135.7, 134.0, 133.9, 132.6, 131.8, 130.4, 127,8, 128.5, 124.6, 124.1, 122.3, 121.5, 119.8, 104.8, 69.5, 55.6, 40.9, 31.9, 31.6, 30.2, 29.1, 25.8, 24.2, 22.9, 22.8, 14.21, 14.16. MS (MALDI-TOF): 962.7 (100%), 963.6(80%), 964.6 (60%); (calcd for M⁺: 962.7). Anal. Calcd for C₆₁H₇₀O₆S₂: C, 76.05; H, 7.32. Found: C, 76.04; H, 7.29.

Synthesis of Compound 6 (Scheme 4).



Scheme 4. Reagents, conditions and yields: i, *n*-BuLi (2.5 M in hexanes), THF, 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaboralane, -78 °C to 20 °C (47% yield); ii, **I** (0.33 equiv.), $Pd_2(dba)_3$, tricyclohexylphosphine, CsF (aq.), dioxane:toluene (3:1 v/v), 110 °C (43% yield).

3,7-Bis(2-hexyloxy-*S***,***S***-dioxide-dibenzothiophen-3-yl)-2,8-di(cyclohexylmethoxy) dibenzothiophene-***S***,***S***-dioxide 6 (S(OR')-S(OR)₂-S(OR')). A white solid. M.p. 230.5-232.0 °C. ¹H NMR (700 MHz, CDCl₃): \delta 7.81 (d,** *J* **= 7.7 Hz, 2H), 7.79 (d,** *J* **= 7.7 Hz, 2H), 7.75 (s, 2H), 7.74 (s, 2H), 7.64 (td,** *J* **= 7.7, 1.4 Hz, 2H), 7.53 (td,** *J* **= 7.7, 1.4 Hz, 2H), 7.26 (s, 2H), 7.24 (s, 2H), 4.09 (t,** *J* **= 7.0 Hz, 4H), 3.91 (d,** *J* **= 5.6 Hz, 4H), 1.78-1.62 (m, 16H), 1.40-1.35 (m, 4H), 1.31-1.26 (m, 8H), 1.25-0.99 (m, 10H), 0.84 (t,** *J* **= 7.0 Hz, 6H). ¹³C NMR (175 MHz, CDCl₃): \delta 161.2, 161.0, 139.1, 133.8, 133.6, 133.4, 131.7, 130.6, 130.3, 129.0, 127.9, 127.7, 125.6, 125.4, 122.3, 121.6, 104.31, 104.27, 74.8, 69.5, 37.8, 31.6, 30.1, 29.0, 26.5, 25.9, 25.8, 22.7, 14.2. MS (MALDI-TOF): 1068.5 (100%), 1069.5(80%), 1070.5 (60%); (calcd for M⁺: 1068). Anal. Calcd for C₆₂H₆₈O₁₀S₃: C, 69.63; H, 6.41. Found: C, 69.52; H, 6.44. Crystals for X-ray analysis were obtained by recrystallisation from a mixture of THF and ethanol (3:2 v/v).**

Synthesis of Compound 7 (Scheme 5).



Scheme 5. Reagents, conditions and yields: i, 2,2,6,6-tetramethylpiperidine, *n*BuLi, B(OⁱPr)₃, 0 °C – -78 °C – 20 °C; ii, 3,7-dibromodibenzothiophene-*S*,*S*-dioxide **T**, Pd(PPh₃)₂Cl₂, K₂CO₃ (aq), dioxane, 100 °C

(65% yield of **U** from **R**); iii, 4-bromobutylbenzene, *n*BuLi, -78 °C – 20 °C (72% yield); iv, AcOH, HCl (7% yield).

Compound 7. A white solid. M.p. 253.5 – 254.5 °C (from ethanol). ¹H NMR (400 MHz, CDCl₃): δ 8.13 (d, *J* = 0.5 Hz, 2H), 7.77 (d, *J* = 7.6 Hz, 2H), 7.59 (d, *J* = 0.5 Hz, 2H), 7.42 – 7.27 (m, 6H), 7.11 – 6.94 (m, 16H), 2.59 – 2.44 (m, 8H), 1.54 (tt, *J* = 10.8, 7.6 Hz, 8H), 1.32 (dq, *J* = 14.5, 7.3 Hz, 8H), 0.89 (t, *J* = 7.3 Hz, 12H). ¹³C NMR (101 MHz, CDCl₃): δ 158.15, 152.48, 142.83, 142.09, 141.71, 138.11, 138.05, 131.74, 129.37, 128.72, 128.21, 128.21, 126.44, 120.98, 119.57, 114.03, 77.55, 77.23, 76.91, 65.55, 35.39, 33.65, 22.69, 14.15. MS-MALDI *m/z* 920.6 ([M⁺], 100%). HRMS [M⁺+H] calc: 921.4705; found: 921.4748.

Synthesis of Compound 8 (Scheme 6).



Scheme 6. Reagents, conditions and yields: Pd(PPh₃)₂Cl₂, NaCO₃ (aq), dioxane, 110 °C (30% yield).

Compound 8. A white solid. M.p. > 350 °C. ¹H NMR (500 MHz, CDCl₃): δ 8.03 (d, *J* = 0.6 Hz, 2H), 7.86 – 7.76 (m, 4H), 7.59 – 7.53 (m, 4H), 7.53 – 7.46 (m, 4H), 1.36 (s, 18H). ¹³C NMR (126 MHz, CDCl₃): δ 152.04, 143.65, 138.81, 136.07, 132.54, 130.17, 126.86, 126.36, 122.05, 120.62, 34.91, 31.51. MS-EI *m/z* 481.2 ([M⁺+H], 100%). Anal. Calcd. for C₃₂H₃₂O₂S: C, 79.96; H, 6.71. Found: C, 79.82; H, 6.67.

Photophysical Data.

These data are collated in Table S1. It should be noted that, in general, moderate or high photoluminescence quantum yield values are retained in the films. It is interesting to note the lack of significant solid state PL quenching. The implications of this fact for the nature of the ICT state will be investigated further in future work..

			p j =	,			
	Solvent	$\lambda_{\rm abs}/{\rm nm}$	$\lambda_{\rm PL}/\rm nm$	$arPhi_{ m PL}$ / %	$\tau_2 / \operatorname{ns}(A_2)$	$\tau_1 / \operatorname{ns}(A_1)$	χ^2
	/film						
1	MCH		385, 475		0.03 (0.45), 0.01 (-0.29)	0.71 (0.55), 0.72 (1)	0.99, 1.02
	PhMe	358	395, 416	0.8 ± 0.1			
	CHCl ₃	358	395, 416				
	EtOH		385, 475		0.03 (0.43), 0.05 (-0.1)	0.78 (0.57), 0.81 (1)	1.03, 1.06
	film	357	416, 440	0.7 ± 0.05			
2	MCH		410, 460			1.03 (1), 1.03 (1)	1.06, 1.04
	PhMe	352, 370	415, 434	0.9 ± 0.1			
	CHCl ₃	352,370	430				
	EtOH		410, 430, 520		0.04 (0.38), 0.03 (-0.29)	1.81 (0.2), 1.81 (0.62), 1.81 (1)	1.02, 1.02, 1.06
	film	360	441	0.5 ± 0.1			
3	MCH		390, 480		0.03 (0.49), 0.01 (-0.21)	0.87 (0.51), 0.88 (1)	1.06, 1.13
	PhMe	361	409, 429	1.0 ± 0.1			
	CHCl ₃	361	422				
	EtOH		410, 500		0.03 (0.44), 0.02 (-0.30)	1.35 (0.36), 1.36 (1)	1.18, 1.13
	film	360	435	0.7 ± 0.1			
4	MCH		400, 480		0.04 (0.30), 0.02 (-0.28)	0.96 (0.70), 0.96 (1)	0.98, 1.04
	PhMe	371	414, 433	0.8 ± 0.1			
	CHCl ₃	372	423, 440				
	EtOH		410, 500		0.04 (0.50), 0.03 (-0.26)	1.27 (0.50), 1.26 (1)	1.07, 1.06
_	film	375	443	0.5 ± 0.1			
5	MCH		390, 460		0.04 (0.38), 0.02 (-0.25)	0.83 (0.62), 0.84 (1)	1.14, 1.00
	PhMe	364	406, 427	0.9 ± 0.1			
	CHCl ₃	364	420				
	EtOH	<u></u>	410, 500		0.04 (0.66), 0.02 (-0.54)	1.61 (0.34), 1.61 (1)	1.22, 1.18
_	film	374	435	0.14 ± 0.05			
6	MCH	2.00	400, 450	0.7.01	0.04 (0.17)	1.00 (0.83), 1.04 (1)	1.05, 0.99
	PhMe	368	408, 429	0.7 ± 0.1			
	CHCl ₃	355	408				1.04.1.00
	EtOH	400	400, 475	0.72 + 0.05	0.03 (0.25), 0.15 (-0.06)	1.08 (0.75), 1.14 (1)	1.04, 1.08
-	nim	408	428	0.72 ± 0.05	0.02 (0.14) 0.07 (0.0()		1.04.1.02
7	MCH	250	400, 475	07.01	0.03 (0.14), 0.07 (0.06)	1./4 (0.86), 1./5 (0.94)	1.04, 1.02
	PhMe	350	396, 414	0.7 ± 0.1			
	CHCl ₃	333	425		0.02 (0.70)	2.75 (0.2) 2.02 (1)	1.05 1.02
	EIUH £1	252	400, 475	0.1 + 0.05	0.03 (0.70)	3.75 (0.3), 3.92 (1)	1.05, 1.02
0	MCU	555	423	0.1 ± 0.05	0.04 (0.07) 0.00 (0.05)	1.52 (0.02) 1.55 (0.05)	1.04.1.02
ð	MCH	225 260	3/3, 4/3	0.20 1.0.05	0.04 (0.07), 0.09 (0.05)	1.55 (0.95), 1.55 (0.95)	1.04, 1.02
		323,360	390, 410	0.38 ± 0.05			
		525, 500	410		0.02 (0.76) 0.01 (.0.2)	2.76(0.24), 2.76(1)	1.08 1.01
	film	360	<i>303,473</i> <i>420</i>	0.4 ± 0.1	0.05 (0.70), 0.01 (-0.2)	2.70 (0.24), 2.70 (1)	1.00, 1.01
	1 111111	500	1 447	10.4 ± 0.1			

Table S1. Photophysical data for 1-8, in different solvents and in film. Excitation wavelength: 380 nm



Figure SI:1 Normalised absorption (solid) and photoluminescence (dashed) spectra of compound **1** in toluene (black), chloroform (red) and film (blue).



Figure SI:2. Normalised absorption (solid) and photoluminescence (dashed) spectra of compound 2 in toluene (black), chloroform (red) and film (blue).



Figure SI:3. Normalised absorption (solid) and photoluminescence (dashed) spectra of compound 3 in toluene (black), chloroform (red) and film (blue).



Figure SI:4 Normalised absorption (solid) and photoluminescence (dashed) spectra of compound 4 in toluene (black), chloroform (red) and film (blue).



Figure SI:5 Normalised absorption (solid) and photoluminescence (dashed) spectra of compound **5** in toluene (black), chloroform (red) and film (blue).



Figure SI:6 Normalised absorption (solid) and photoluminescence (dashed) spectra of compound **6** in toluene (black), chloroform (red) and film (blue).



Figure SI:7 Normalised absorption (solid) and photoluminescence (dashed) spectra of compound **7** in toluene (black), chloroform (red) and film (blue).



Figure SI:8 Normalised absorption (solid) and photoluminescence (dashed) spectra of compound **8** in toluene (black), chloroform (red) and film (blue).

No dependence of the emission on concentration of the solution was found with the new oligomers, in agreement with previous data for compound 2.⁶ This confirms that that the red-shifted unresolved emission in polar solvents is not caused by intermolecular interactions, for example, aggregation or excimer formation (Figures S1:9 and 10).



Figure SI:9 Photoluminescence spectra for four different solutions of compound **7** in ethanol with OD values ranging from $0.32 (0.03 \text{ mg/cm}^3)$ to 0.012 at 355 nm. Excitation wavelength: 355 nm.



Figure SI:10 Normalised PL emission spectra shown in Figure SI:9. No new features were observed in the spectra with increasing concentration.

Solution Electrochemical Studies

Cyclic voltammetry experiments of the F-S systems showed reversible or quasi-reversible oxidation peaks (Table S2). In all cases the presence of the S units positively shifted the oxidation potentials compared to F-F-F **1**, indicating the reduced donor properties of F-S systems. A comparison of **4** $(E_1^{\text{ox}} 1.05 \text{ V}, \text{ vs. Fc/Fc}^+)$, **5** (1.07 V), and **6** (not observed, scanned up to 1.8 V) shows that, as predicted, the oxidation potential increases with an increasing number of S units – significantly so for the S-S-S trimer **6**. A comparison of the redox potentials of **2** and **3** reveals that the difluoro substituents on the S unit of **3** significantly reduce the donor ability of the trimer. The value of E_1^{ox} for **3** is raised by 0.22 V compared to **2**. For the reduction processes, it can be noted that in contrast to trimer **1**, all the compounds

2-8 show at least one wave due to the presence of the S unit(s). However, unlike the oxidation potentials, the reduction potential(s) cannot be correlated with the number of S units. It is notable that the S-S-S trimer **6** shows three distinct reduction peaks, consistent with sequential radical anion, dianion and radical trianion formation, although it is not clear why E_1^{red} for compound **5** is positively shifted compared to the S-S-S trimer **6** (i.e. **5** is easier to reduce than **6**) and the difluoro-S unit of **3** does not enhance the acceptor properties compared to **2**.

Compound	Oxidation; E^{ox}/V^a	Reduction E^{red}/V^a
1	+0.88, +1.12	Not observed
2	+1.19, +1.26	-2.16, -2.48
3	+1.41	-2.3
4	+1.05, +1.22	-2.21
5	+1.07	-2.06, -2.52
6	Not observed	-2.29, -2.61, -2.89
7	+0.98, +1.22, +1.48	-2.34
8	+1.46	-2.25, -2.38

 Table S2.
 Solution electrochemical data

^{*a*} vs. Fc/Fc. (See Page S1 for details).

X-Ray Crystallography







Figure SI:11. Four independent molecules in the crystal structure of **3**. Hydrogen atoms are omitted; thermal ellipsoids are drawn at the 50% probability level.



Figure SI:12. Molecular structure of **4**. Hydrogen atoms are omitted; thermal ellipsoids are drawn at the 50% probability level.



Figure SI:13. Molecular structure of **6**·MeOH. Thermal ellipsoids are drawn at the 50% probability level.

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