

Supporting Information

Self-doping small molecular conjugated electrolytes enabled by n-type side chains for highly efficient non-fullerene polymer solar cells

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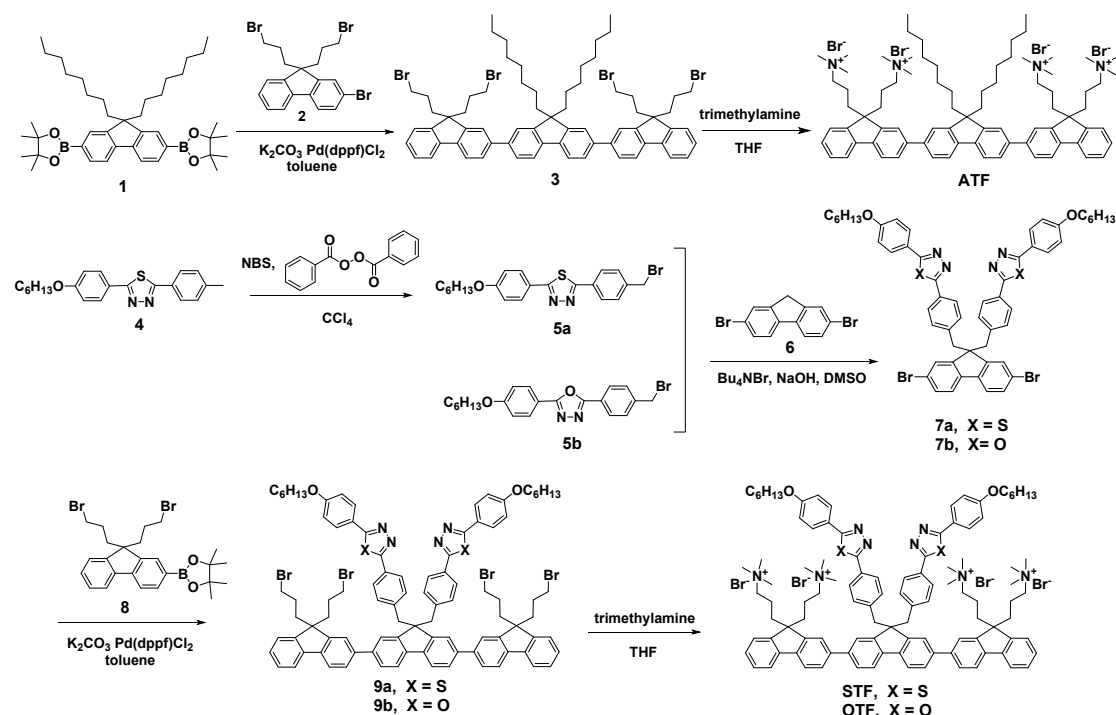
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1. Synthetic routes of the intermediates and SMCEs



Scheme S1 Synthetic routes of the SMCEs

2. Synthetic details of the intermediates and cathode interlayer materials

All reagents were purchased from Aladdin Co., Alfa Aesar Co., and Aldrich Chemical Co., and used as received without further purification. Compounds **1**, **2**, **4**, **6**, **5b** and **8** and were synthesized according to previous reported procedures.¹⁻⁴ The synthetic routes of the monomers and copolymers are shown in Scheme S1. The detailed synthetic processes are as follows.

Compound 3

Compound **1** (6.42g, 10.0 mmol), compound **2** (9.68g, 20.0 mmol) and Pd(PPh₃)₄ (73 mg, 0.1 mmol) were first added to a degassed solution of toluene (20 mL) and Na₂CO₃ aqueous solution (2 M, 5 mL) in a 50 mL Schlenk flask. The reaction was stirred at 110 °C for 24 h. The reaction mixture was then cooled down to room temperature. Dichloromethane (DCM) and water was added for extraction. The organic layer was washed with brine and water, and then dried with anhydrous

MgSO₄. After removal of the solvent under rotary evaporation, the crude product was purified by column chromatography to obtain a white solid (yield: 56%). ¹H NMR (400 MHz, CDCl₃, δ/ppm): 7.75-7.41 (m, 14H, -ArH), 7.41-7.23 (m, 6H, -ArH), 3.16-3.12 (s, 8H, -CH₂-Br), 2.28-2.10 (s, 12H, -CH₂-), 1.22-1.11 (m, 30H, -CH₂-), 0.85-0.78 (m, 10H, -CH₃). ¹³C NMR (100 MHz, CDCl₃, δ/ppm): 151.9, 149.7, 149.1, 141.1, 140.8, 140.2, 127.6, 126.8, 126.2, 122.9, 121.6, 121.4, 120.3, 120.1, 55.4, 54.2, 40.4, 39.0, 38.7, 36.1, 34.5, 31.8, 30.1, 29.3, 29.1, 27.2, 27.0, 24.0, 22.7, 19.5, 18.8, 14.2, 11.5. Anal. Calcd for C₆₇H₇₈Br₄: C, 66.90; H, 6.54; found: C, 66.82; H, 6.60.

Compound ATF

A mixture of compound **3** (0.60 g, 0.5 mmol) and trimethylamine solution (33%, 5 mL) was dissolved in anhydrous tetrahydrofuran (THF, 50 mL). The mixture was refluxed under argon atmosphere for 10 h. After removal of the solvent under rotary evaporation, the residue was recrystallized from chloroform to give compound **ATF** as a pale solid (yield: 62%). ¹H NMR (400 MHz, CD₃OD, δ/ppm): 7.98-7.87 (m, 14H, -ArH), 7.84-7.78 (m, 2H, -ArH), 7.65-7.42 (s, 4H, -ArH), 3.21-3.17 (s, 8H, -CH₂-), 2.81-2.92 (s, 36H, -CH₃), 2.36-2.21 (m, 12H, -CH₂-), 1.21-1.08 (m, 30H, -CH₃), 0.81-0.78 (m, 6H, -CH₃), 0.75-0.66 (m, 4H, -CH₃). ¹³C NMR (100 MHz, CD₃OD, δ/ppm): 151.6, 149.1, 148.3, 141.3, 140.8, 140.5, 140.2, 139.9, 127.9, 127.7, 126.9, 126.3, 123.4, 121.4, 120.9, 121.4, 120.9, 120.4, 120.1, 119.9, 66.3, 55.4, 54.2, 51.9, 48.2, 48.1, 47.9, 47.6, 47.4, 47.2, 47.0, 40.1, 35.6, 31.5, 29.7, 29.1, 28.9, 23.7, 22.3, 24.0, 17.8, 13.1. Anal. Calcd for C₇₉H₁₁₄Br₄N₄: C, 65.92; H, 7.98; N, 3.89; found: C, 66.00; H, 7.90; N, 3.92.

Compound 5a

A mixture of compound **4** (3.52 g, 10.0 mmol), N-bromosuccinimide (NBS, 1.96 g, 11.0 mmol), benzoyl peroxide (0.02 g, 0.08 mmol) and CCl₄ (40 mL) in a 100 mL flask was refluxed for 5 h. Then the mixture was filtered while it was still hot. The solid was washed with hot chloroform. The residual solid was recrystallized from THF/methanol to give compound **5a** as white needle-like crystals (yield: 45%). ¹H NMR (400 MHz, CDCl₃, δ/ppm): 7.98-7.91 (m, 4H, -ArH), 7.53-7.49 (m, 2H, -ArH), 7.10-6.82 (m, 2H, -ArH), 4.01-4.67 (t, 2H, -CH₂-), 1.59-1.86 (m, 2H, -CH₂-), 1.52-1.39 (m, 6H, -CH₂-), 0.95-0.91 (t, 3H, -CH₃). ¹³C NMR (100 MHz, CDCl₃, δ/ppm): 167.6, 167.4, 161.5, 141.3, 129.8, 129.4, 127.7, 127.6, 122.7, 115.0, 77.4, 77.1, 76.8, 68.2, 53.5, 31.6, 29.1, 25.7, 22.6, 21.5, 14.1. Anal. Calcd for C₂₁H₂₃N₂BrOS: C, 58.47; H, 5.37; N, 6.49; Br, 18.52; O, 3.71; S, 7.43; found: C, 58.26; H, 5.22; N, 6.62.

Compound 7a

Compound **6** (3.22 g, 10.0 mmol), potassium hydroxide (1.68 g, 30.0 mmol) and a catalytic amount of tetrabutylammonium bromide (0.77 g) were first dissolved in dimethylsulfoxide (DMSO, 20 mL). A DMSO (5 mL) solution of compound **5a** (12.9 g, 30.0 mmol) was then added dropwise in 0.5 h. The reaction mixture was stirred at room temperature for 24 h and poured into water (300 mL). The product was extracted with DCM. The combined organic layers organic layer was washed with brine and water and then dried with anhydrous MgSO₄. After removal of the solvent under rotary evaporation, the crude product was purified by column chromatography to obtain compound **7a** as a white solid (yield: 47.2%). ¹H NMR (400 MHz, CDCl₃,

δ /ppm): 7.98-7.87 (m, 14H, -ArH), 7.84-7.78 (m, 2H, -ArH), 7.92-7.89 (m, 3H, -ArH), 7.76-7.74 (m, 3H, -ArH), 7.50-7.44 (m, 10H, -ArH), 7.02-6.96 (m, 6H, -ArH), 4.04-4.00 (t, 4H, -CH₂-), 2.06-2.03 (s, 4H, -CH₂-), 1.49-1.46 (m, 4H, -CH₂-), 1.37-1.34 (m, 6H, -CH₂-), 1.27-1.24 (m, 6H, -CH₂-), 0.93-0.91 (t, 6H, -CH₃). ¹³C NMR (100 MHz, CDCl₃, δ /ppm): 167.0, 161.6, 161.5, 149.3, 146.5, 141.3, 139.1, 138.9, 138.5, 138.3, 132.7, 131.7, 130.9, 130.8, 129.4, 128.8, 128.4, 127.9, 127.8, 120.1, 127.6, 126.9, 122.5, 121.6, 120.8, 115.0, 68.2, 65.9, 57.2, 53.5, 46.1, 45.1, 31.6, 29.4, 29.1, 25.7, 22.6, 14.1. Anal. Calcd for C₅₅H₅₂Br₂N₄O₂S₂: C, 64.45; H, 5.11; N, 5.47; found: C, 64.31; H, 5.21; N, 5.52.

Compound 7b

Compound **7b** was synthesized similar to **7a** with a yield of 48.0%. ¹H NMR (400 MHz, CDCl₃, δ /ppm): 7.99-7.94 (m, 4H, -ArH), 7.70-7.64 (m, 1H, -ArH), 7.55-7.47 (s, 4H, -ArH), 7.46-7.42 (m, 1H, -ArH), 7.39-7.36 (m, 2H, -ArH), 7.22-7.19 (m, 2H, -ArH), 7.01-6.74 (m, 8H, -ArH), 4.12-3.99 (m, 4H, -CH₂-), 3.49-3.45 (m, 4H, -CH₂-), 1.83-1.75 (m, 4H, -CH₂-), 1.50-1.31 (m, 12H, -CH₂-), 0.94-0.86 (m, 6H, -CH₃). ¹³C NMR (100 MHz, CDCl₃, δ /ppm): 164.4, 163.9, 161.9, 149.0, 139.7, 138.9, 131.0, 130.6, 128.6, 127.6, 125.9, 122.2, 121.5, 120.9, 116.1, 114.9, 68.2, 57.2, 45.3, 31.6, 29.1, 25.7, 22.6, 14.0. Anal. Calcd for C₅₅H₅₂Br₂N₄O₄: C, 66.54; H, 5.28; N, 5.64; found: C, 66.46; H, 5.25; N, 5.52.

Compound 9a

Compound **7a** (0.63 g, 0.62 mmol), compound **8** (0.83 g, 1.56 mmol), Pd(PPh₃)₄ (0.025 g), toluene (30 ml), and Na₂CO₃ aqueous solution (2M 20 mL) were first

added into an oven-dried 100 mL flask. The solution was degassed with bubbling argon and then heated to reflux for 12 h under argon. After cooling to room temperature, the reaction mixture was moved to a separatory funnel and 20 mL of diethyl ether was added. The organic layer was washed with brine. After drying over sodium sulfate, the organic layer was removed and the residue was purified by column chromatography to give compound **9a** as a faint yellow powder (yield: 46.3%). ¹H NMR (400 MHz, CDCl₃, δ/ppm): 7.90-7.59 (m, 22H, -ArH), 7.39-7.36 (m, 6H, -ArH), 6.97-6.95 (s, 8H, -ArH), 4.02-3.99 (s, 4H, -CH₂-), 3.62-3.58 (s, 4H, -CH₂-), 3.18-3.11 (m, 6H, -CH₂-), 2.45-2.18 (m, 6H, -CH₂-), 1.83-1.75 (m, 4H, -CH₂-), 1.55-1.16 (m, 20H, -CH₂-), 0.94-0.86 (m, 6H, -CH₃). ¹³C NMR (100 MHz, CDCl₃, δ/ppm): 149.8, 149.4, 148.7, 140.8, 140.5, 140.4, 140.2, 139.9, 133.6, 131.2, 129.4, 128.3, 127.6, 127.4, 126.9, 126.8, 126.6, 123.3, 122.6, 121.8, 121.4, 120.5, 120.4, 120.2, 120.1, 117.9, 115.0, 66.3, 56.6, 54.3, 54.2, 45.4, 44.9, 38.6, 34.7, 31.5, 29.1, 27.4, 27.2, 25.7, 22.6, 14.1. Anal. Calcd for C₉₃H₉₀Br₄N₄O₂S₂: C, 66.51; H, 5.40; N, 3.34; found: C, 66.47; H, 5.35; N, 3.32.

Compound 9b

Compound **9b** was synthesized similar to **9a** with a yield of 49.0%. ¹H NMR (400 MHz, CDCl₃, δ/ppm): 7.90-7.59 (m, 22H, -ArH), 7.39-7.36 (m, 6H, -ArH), 6.99-6.93 (s, 8H, -ArH), 4.04-3.96 (s, 4H, -CH₂-), 3.62-3.55 (s, 4H, -CH₂-), 3.19-3.11 (m, 6H, -CH₂-), 2.45-2.18 (m, 6H, -CH₂-), 1.83-1.75 (m, 4H, -CH₂-), 1.56-1.21 (m, 20H, -CH₂-), 0.93-0.86 (m, 6H, -CH₃). ¹³C NMR (100 MHz, CDCl₃, δ/ppm): 164.4, 164.0, 161.9, 150.3, 150.0, 149.6, 149.4, 148.5, 140.9, 140.5, 140.4, 140.3, 139.9, 139.7, 133.8,

133.5, 131.0, 128.6, 127.4, 127.4, 127.3, 127.0, 126.6, 126.4, 125.8, 123.8, 123.4, 123.2, 122.2, 121.8, 120.5, 120.4, 120.2, 120.1, 119.9, 117.9, 68.2, 56.8, 54.3, 54.2, 45.6, 43.5, 37.2, 34.7, 31.5, 29.1, 27.4, 25.7, 22.6, 14.1. Anal. Calcd for $C_{93}H_{90}Br_4N_4O_4$: C, 67.81; H, 5.51; N, 3.40; found: C, 67.77; H, 5.45; N, 3.33.

Compound STF

Compound **9a** (0.47 g, 0.28 mmol) was dissolved in THF (20 mL) and was then diluted with water (5 mL). After the mixture was cooled to 0 °C, condensed trimethylamine (1 mL) was added dropwise. The mixture was stirred at 0 °C for 12 h and was then gradually warmed to room temperature. After an additional 24 h, the THF and water were removed, and the residue was recrystallized from chloroform to give compound **STF** as a yellow solid (yield: 49.2%). 1H NMR (400 MHz, $CDCl_3$, δ/ppm): 8.21-8.09 (m, 4H, -ArH), 7.85-7.63 (m, 14H, -ArH), 7.55-7.40 (m, 10H, -ArH), 7.02-6.90 (m, 8H, -ArH), 4.00-3.80 (s, 8H, -CH₂-), 3.35-3.11 (m, 4H, -CH₂-), 2.92-2.84 (s, 36H, -CH₃-Br), 2.13-2.45 (m, 8H, -CH₂-), 1.65-1.86 (m, 4H, -CH₂-), 1.14-1.56 (m, 20H, -CH₂-), 0.94-0.86 (m, 6H, -CH₃). ^{13}C NMR (100 MHz, CD_3OD , δ/ppm): 168.2, 167.7, 161.9, 149.4, 148.5, 141.8, 140.8, 140.7, 140.2, 139.1, 131.2, 129.1, 127.9, 127.7, 127.4, 126.7, 126.5, 126.2, 123.4, 121.8, 121.5, 120.6, 120.2, 114.9, 68.0, 66.3, 58.8, 57.4, 54.3, 54.2, 52.0, 35.6, 31.4, 29.4, 28.9, 25.5, 22.3, 18.0, 13.1. Anal. Calcd for $C_{105}H_{126}Br_4N_8O_2S_2$: C, 65.82; H, 6.63; N, 5.85; found: C, 65.77; H, 6.55; N, 5.82.

Compound OTF

Compound **OTF** was synthesized similar to **STF** with a yield of 48.2% 1H NMR (400

MHz, CDCl₃, δ /ppm): 8.27-8.09 (m, 4H, -ArH), 8.05-7.83 (m, 10H, -ArH), 7.65-7.50 (m, 8H, -ArH), 7.49-7.40 (m, 6H, -ArH), 7.12-6.90 (m, 6H, -ArH), 4.10-3.90 (s, 8H, -CH₂-), 3.25-3.11 (m, 4H, -CH₂-), 2.92-2.84 (s, 36H, -CH₃-Br), 2.43-2.15 (m, 8H, -CH₂-), 1.86-1.65 (m, 4H, -CH₂-), 1.56-1.14 (m, 20H, -CH₂-), 0.94-0.86 (m, 6H, -CH₃). ¹³C NMR (100 MHz, CD₃OD, δ /ppm): 164.6, 164.2, 162.4, 149.3, 148.5, 142.7, 140.8, 140.7, 140.2, 139.2, 131.0, 128.4, 127.8, 127.7, 126.8, 126.5, 125.2, 123.4, 121.6, 120.9, 120.5, 120.2, 114.9, 69.2, 66.3, 58.6, 57.5, 54.7, 54.2, 52.0, 35.6, 31.4, 30.8, 29.4, 28.8, 25.4, 22.3, 18.0, 13.0. Anal. Calcd for C₁₀₅H₁₂₆Br₄N₈O₄: C, 66.95; H, 6.74; N, 5.95. Found: C, 65.91; H, 6.65; N, 6.02.

3. Measurements and characterizations

¹H and ¹³C NMR spectra were recorded on a Bruker Avance-400 spectrometer with CDCl₃ or CD₃OD as the solvent. The chemical shifts were reported as δ value (ppm) relative to an internal tetramethylsilane (TMS) standard. The elemental analysis was performed on a Thermo Electron SPA Flash EA 1112 series analyzer. UV-vis spectra were obtained on a Hitachi U2910 spectrophotometer. Cyclic voltammetry measurements were made on a CHI660 potentiostat/galvanostat electrochemical workstation at a scan rate of 50 mV s⁻¹, with a platinum wire counter electrode and an Ag/AgCl reference electrode in an anhydrous and nitrogen-saturated 0.1 mol L⁻¹ acetonitrile solution of tetrabutylammonium perchlorate. The UPS measurements were conducted on a Thermo Scientific ESCALAB 250Xi photoelectron spectroscope. AFM images were obtained by using a Bruker Inova atomic microscope in tapping

mode. The TEM images were obtained by using a ZEISS LIBRA 200 FE transmission electron microscope.

Device fabrication: The device structure was ITO/PEDOT:PSS/active layer/CIL/Al. The pre-patterned ITO glass substrates (sheet resistance = $15 \Omega \text{ sq}^{-1}$) were ultrasonicated in detergent, deionized water, acetone, and isopropanol, subsequently. After dried by high-pressure air flow, the substrates were further cleaned by UV-ozone exposure for 30 min. After filtration through a $0.45 \mu\text{m}$ filter, PEDOT:PSS (Bay P VP AI 4083, Bayer AG) was spin-coated at 5000 rpm for 60 s to form a thickness of 35 nm thin layer on the cleaned ITO substrate, and baked on a hot plate at 150°C for about 15 min. A blend film of PBDTS-TDZ:IT-4F (1:1 w/w) was prepared by spin-coating its mixed solvent of MeTHF/1,8-diiodoctane (DIO) (99.5:0.5, v/v) solution (concentration: 15 mg mL^{-1}) at 2000 rpm for 60 s, followed by baked on a hot plate at 100°C for 5 min. The thickness of the active layer was around 100 nm, which was detected by a Dektak 6 M surface profilometer. A thin layer of the CILs was prepared by spin-coating their solutions ($0.5\text{-}5 \text{ mg/mL}$ in MeOH) at 4000 rpm to form different film thicknesses. Finally, Al (100 nm) was deposited on the top of surface in a high vacuum chamber ($\sim 10^{-4} \text{ Pa}$). The device area was 4.0 mm^2 .

Device characterization: The I - V characterization of the devices was carried out on a computer-controlled Keithley 2400 Source Measurement system under 100 mW cm^{-2} AM 1.5G light source, provided by an AAA solar simulator (XES-70S1, SAN-EI Electric Co., Ltd), which was calibrated with a standard Si solar cell (AK-200, KONICA MINOLTA, INC.). The EQE values were measured with a Newport QE test

system (Newport Co., Ltd.) during illumination with monochromatic light from a xenon lamp, monitored by a traceable silicon photodiode. The AC impedances of the devices were characterized with CHI604E electrochemical workstation at the impedance mode under AM 1.5 G 100 mW cm⁻² illumination.

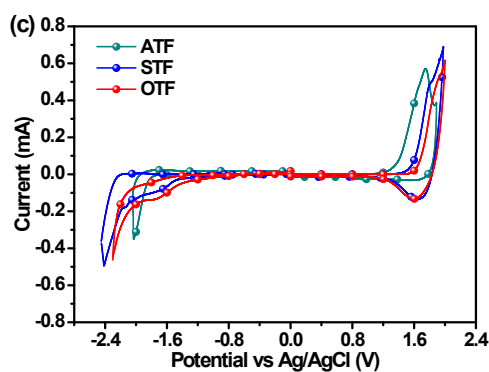


Fig. S1 CV curves of the SMCEs.

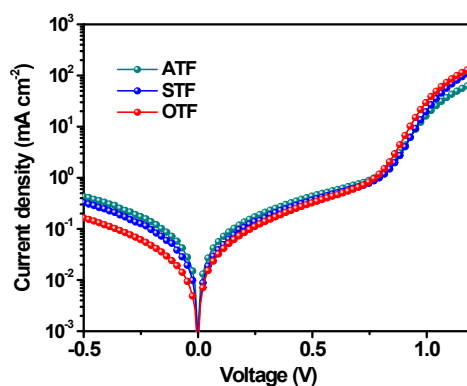


Fig. S2 *J-V* curves of PBDTS-TDZ:IT4F devices under dark.

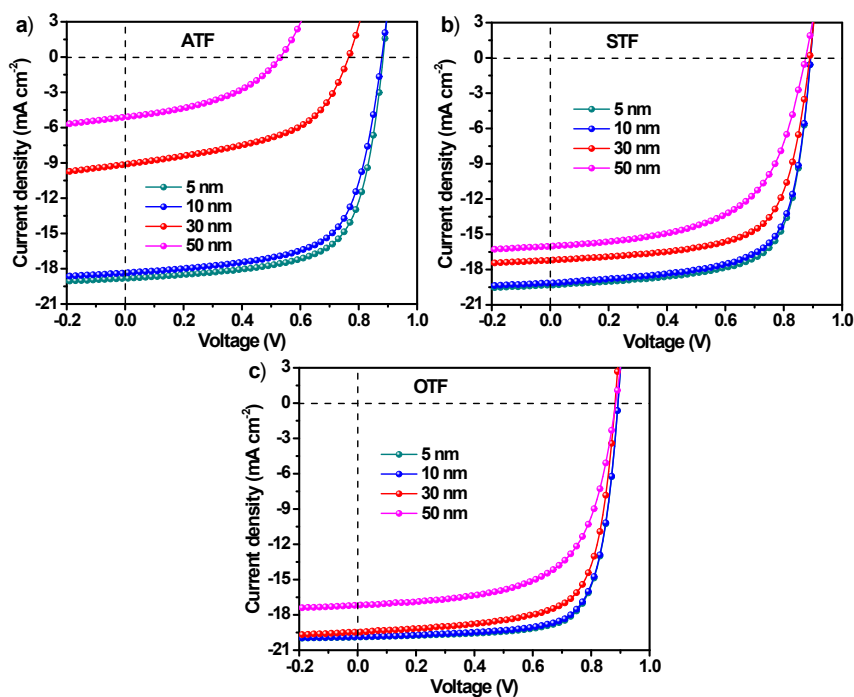


Fig. S3 J - V curves of PBDTS-TDZ:IT-4F devices with various CIL thickness.

Table S1 Optical and electrochemical data of the SMCEs.

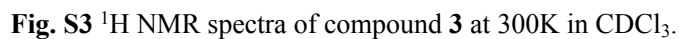
SMCE	Abs. (nm)	Abs. (nm)	$E_g^{\text{opt a)}$ (eV)	HOMO ^{b)} (eV)	LUMO ^{c)} (eV)
	$\lambda_{\text{max}}^{\text{Sol}}$	$\lambda_{\text{max}}^{\text{film}}$			
ATF	351	352	3.19	-6.33	-3.14
STF	341	342	3.20	-7.14	-3.94
OTF	351, 308	352, 310	3.18	-7.23	-4.05

^{a)}Optical band gap (E_g^{opt}) was estimated from the wavelength of the optical absorption edge of the SMCE film. ^{b)} HOMO level was calculated from the UPS results. ^{c)} LUMO level was calculated from the equation LUMO = E_g - HOMO.

Table S2 Parameters of the equivalent circuit for the of the NF-PSCs with various CILs.

CIL	R_s (Ω)	R_1 (Ω)	C_1 (F)	R_2 (Ω)	C_2 (F)	R_3 (Ω)	C_3 (F)
ATF	42.1	107.1	8.5e-9	40.2	3.3e-9	11.5	3.4e-9
STF	40.2	99.9	9.2e-9	19.6	5.1e-8	5.2	2.3e-7
OTF	38.6	96.7	9.6e-9	17.6	4.7e-8	3.5	5.4e-6

CIL	Thickness (nm)	V_{oc} (V)	J_{sc} (mA cm ⁻²)	FF (%)	PCE (%)
ATF	5	0.88	18.83	68.2	11.30
	10	0.87	18.35	66.6	10.63
	30	0.76	9.12	51.4	4.50
	50	0.53	5.10	42.5	1.15
STF	5	0.89	19.32	70.5	12.12
	10	0.88	19.14	70.1	11.81
	30	0.88	17.21	68.0	10.30
	50	0.87	16.02	58.7	8.18
OTF	5	0.89	19.89	74.6	13.21
	10	0.89	19.86	74.4	13.15
	30	0.88	19.47	70.8	12.13
	50	0.88	17.18	62.9	9.51



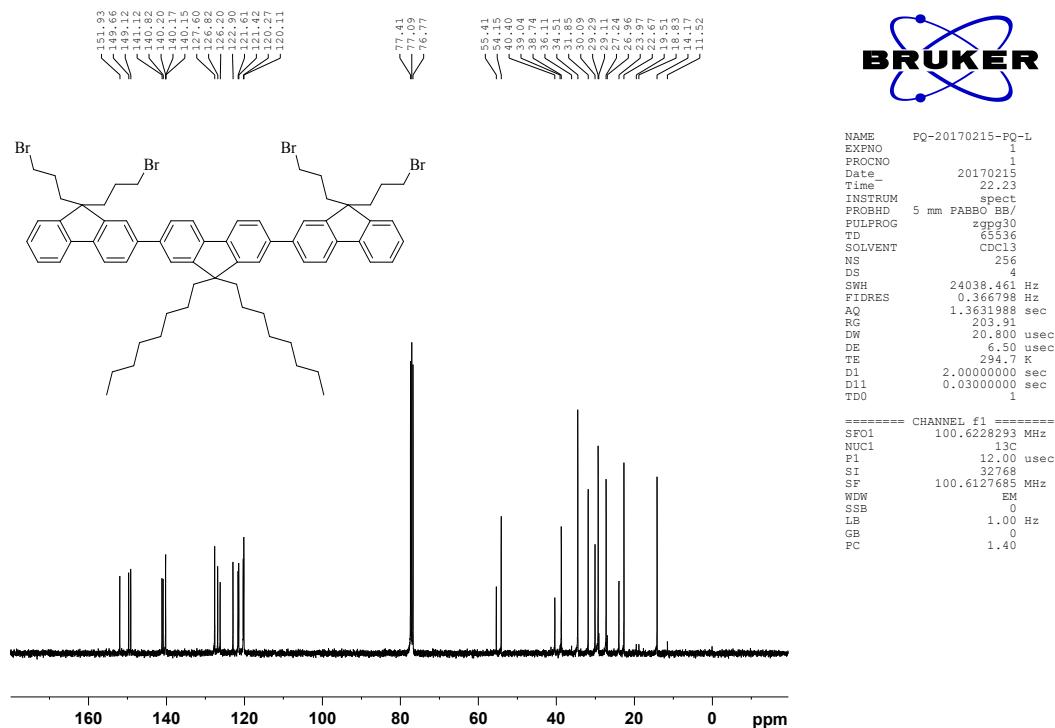


Fig. S4 ¹³C NMR spectra of compound **3** at 300K in CDCl₃.

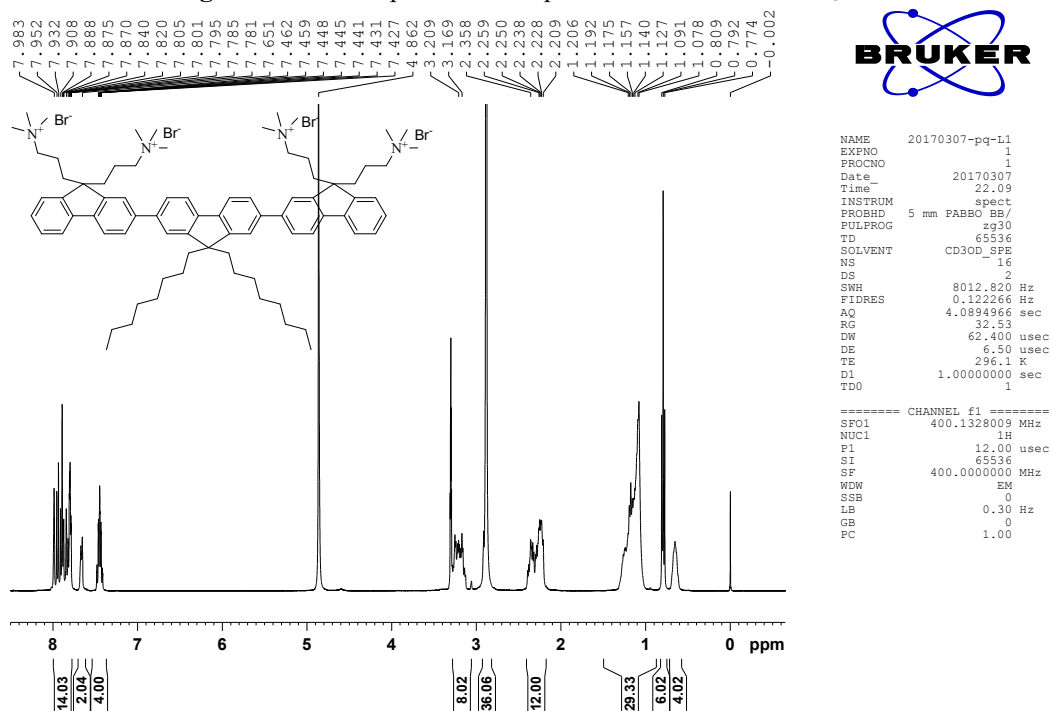
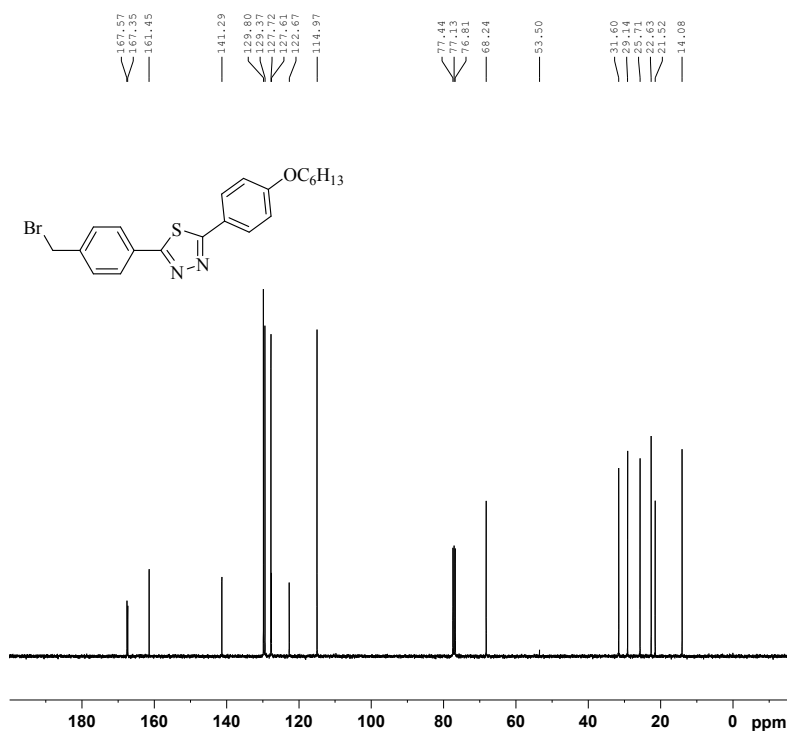


Fig. S5 ¹H NMR spectra of compound **ATF** at 300K in CD₃OD.



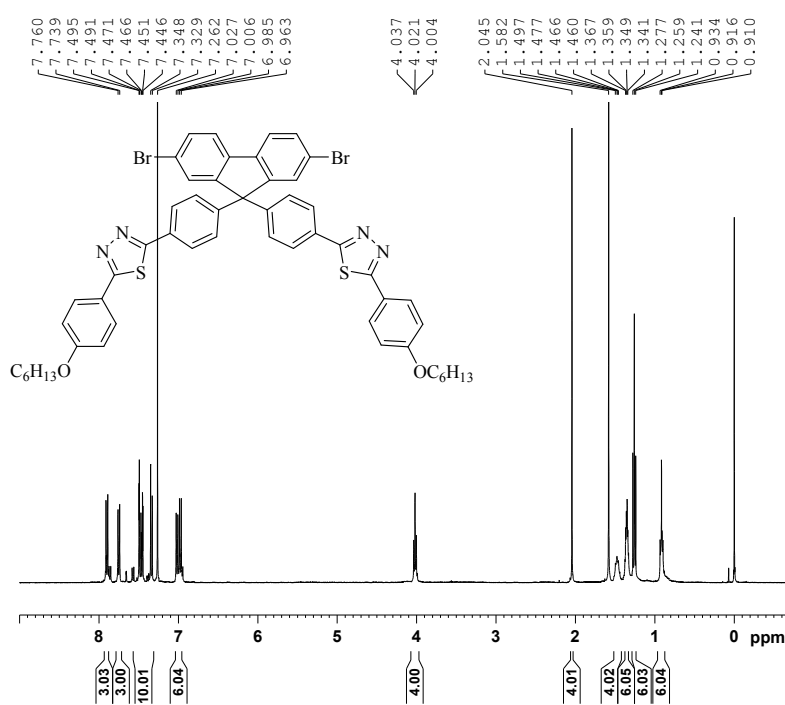
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RG         203.91
DW         20.800 usec
DE         6.50 usec
TE         294.7 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

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NUC1      13C
P1        12.00 usec
SI        32768
SF        100.6127685 MHz
WDW       EM
SSB       0
LB        1.00 Hz
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PC        1.40

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Fig. S8 ¹³C NMR spectra of compound 5a at 300K in CDCl₃.



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Fig. S19 ¹H NMR spectra of compound 7a at 300K in CDCl₃.

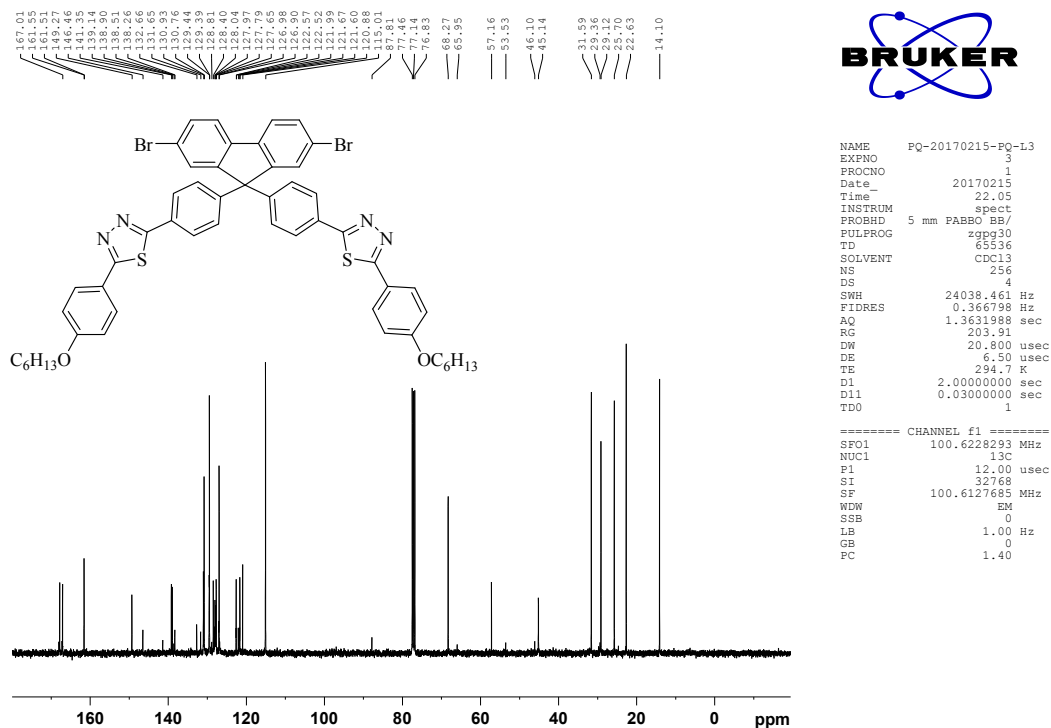


Fig. S10 ¹³C NMR spectra of compound **7a** at 300K in CDCl₃.

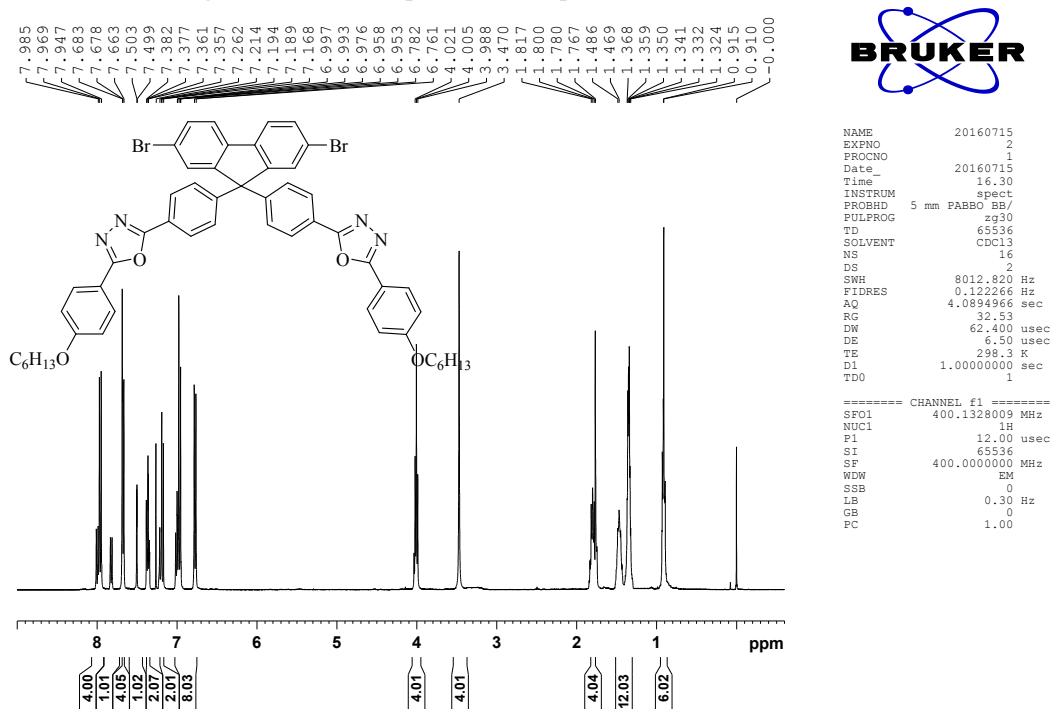


Fig. S11 ¹H NMR spectra of compound **7b** at 300K in CDCl₃.

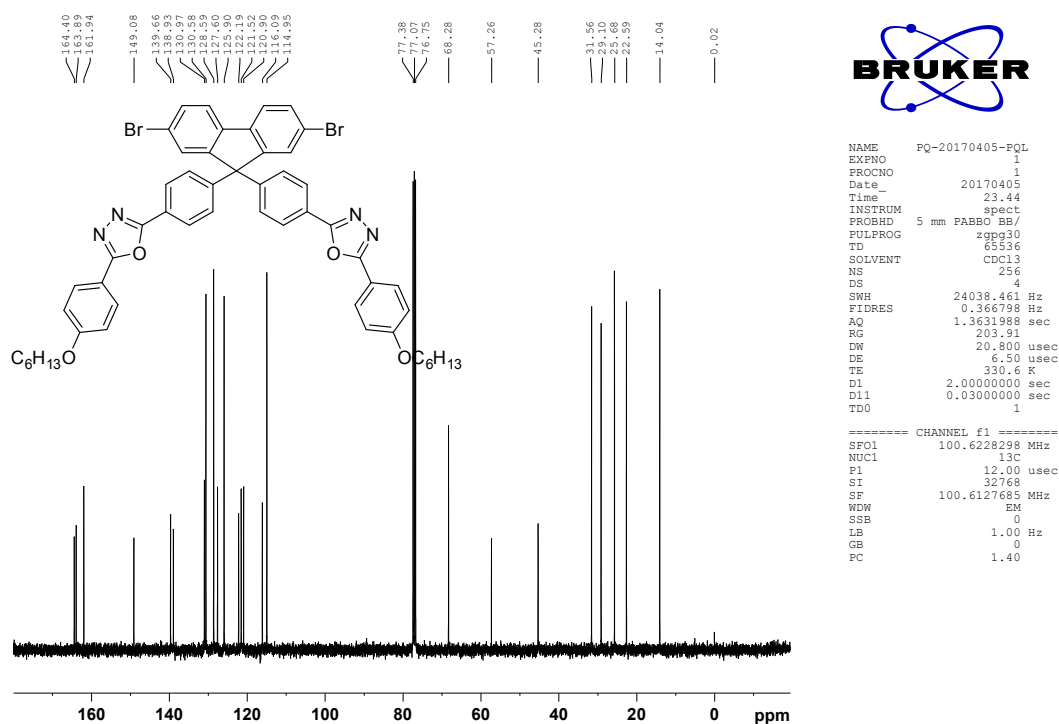


Fig. S12 ^{13}C NMR spectra of compound **7b** at 300K in CDCl_3 .

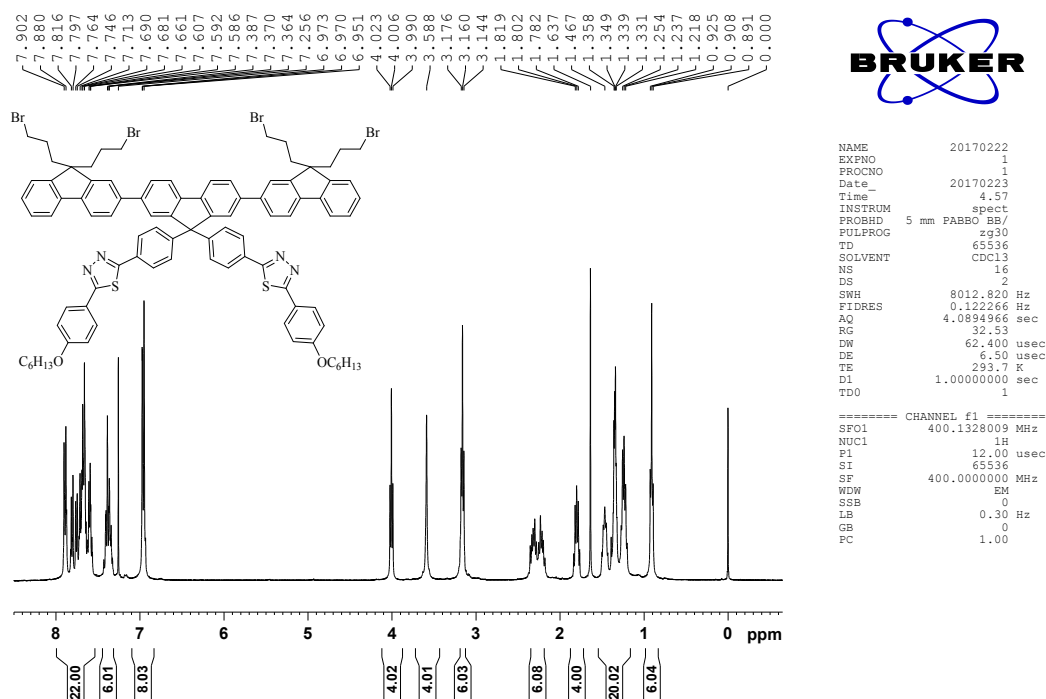
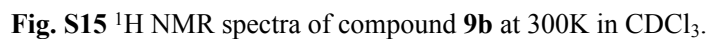


Fig. S13 ^1H NMR spectra of compound **9a** at 300K in CDCl_3 .



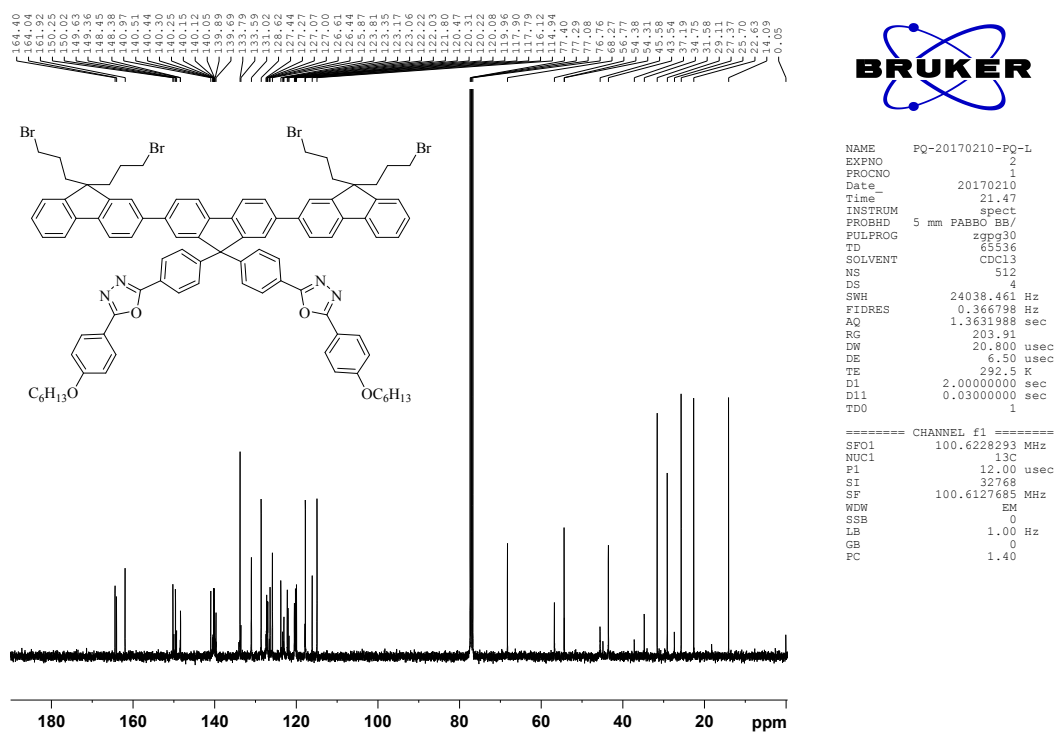


Fig. S16 ^{13}C NMR spectra of compound **9b** at 300K in CDCl_3 .

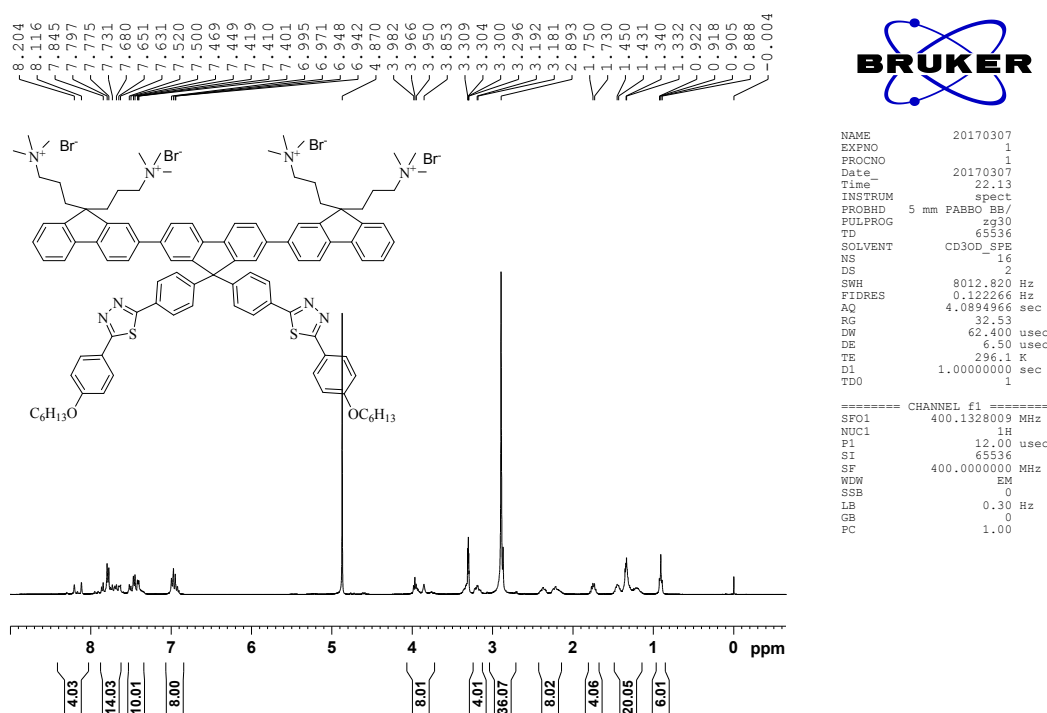


Fig. S17 ^1H NMR spectra of STF at 300K in CD_3OD .

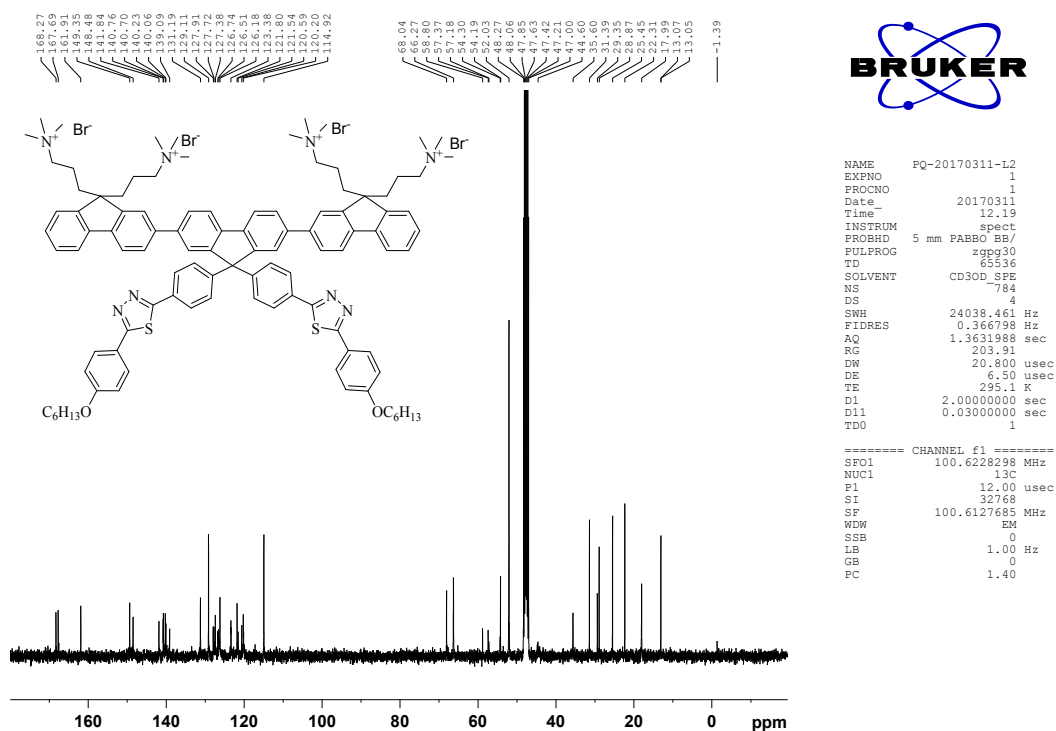


Fig. S18 ^{13}C NMR spectra of STF at 300K in CD_3OD .

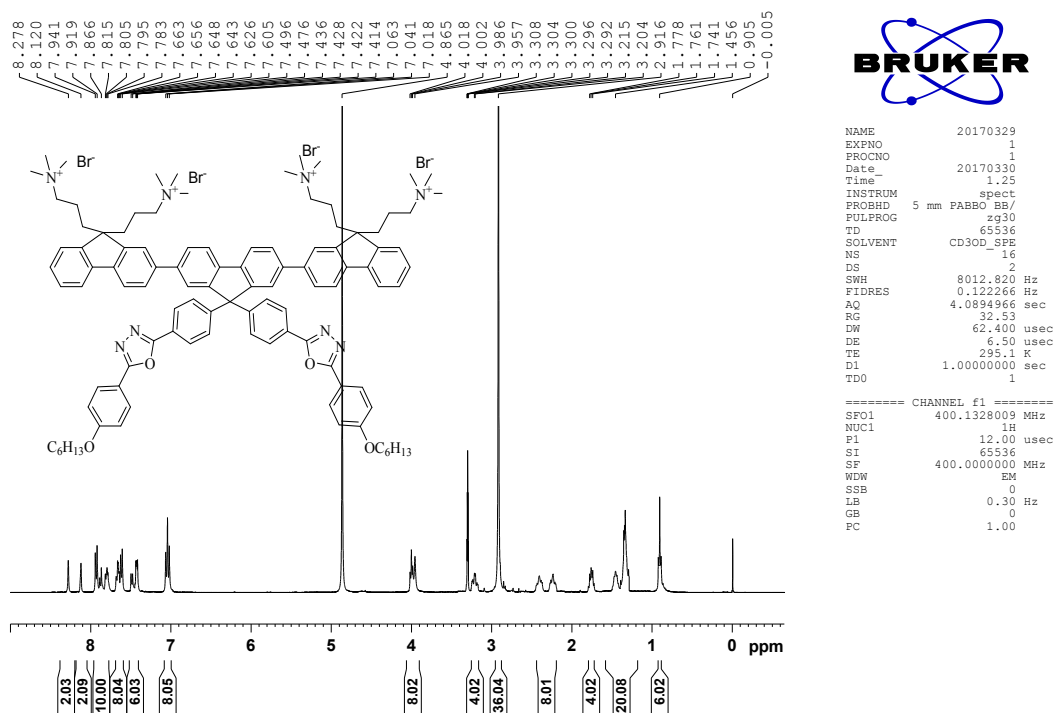


Fig. S19 ^1H NMR spectra of OTF at 300K in CD_3OD .

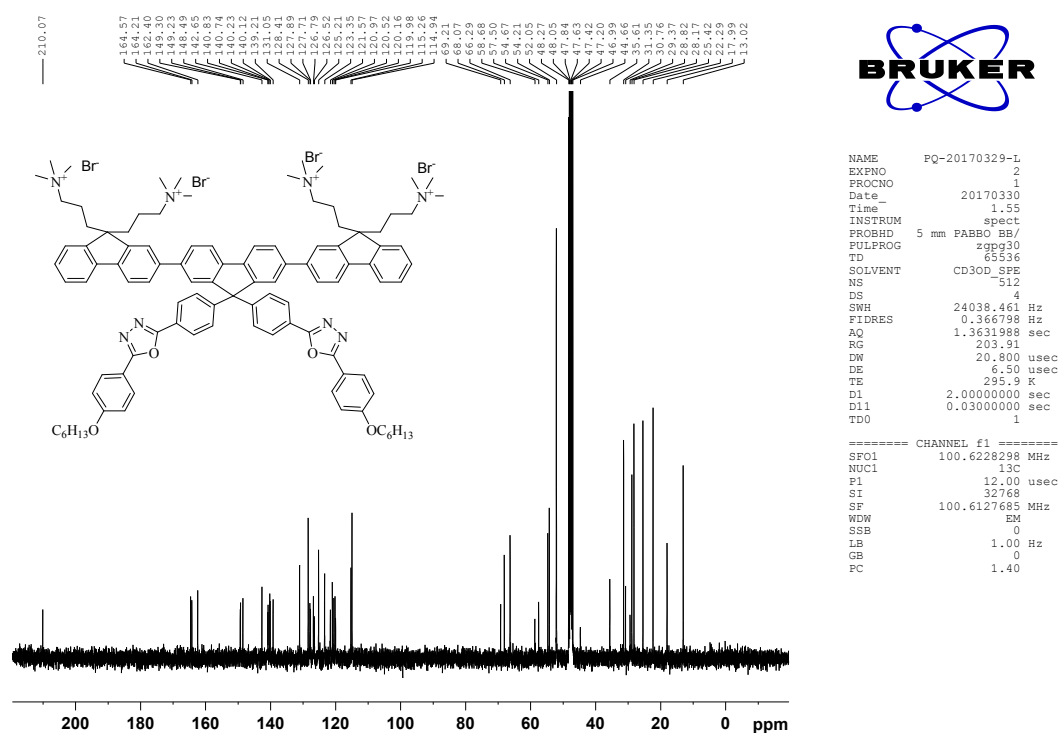


Fig. S20 ^{13}C NMR spectra of **OTF** at 300K in CD_3OD .

4. References

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