

Supporting Information for

Influence of fluorination on the properties and performance of isoindigo- quaterthiophene-based polymers

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Additional Methodological Details

Device fabrication and characterization

Pre-patterned ITO-coated glass with a sheet resistance of $\sim 15\Omega$ per square was used as the substrate. It was cleaned by sequential sonications in soap DI water, DI water, acetone and isopropanol for 15 min at each step. After ultraviolet/ozone treatment for 60 min, a ZnO electron transport layer was prepared by spin coating at 5,000 r.p.m. from a ZnO precursor solution (To a 0.13 mL diethyl zinc 15 wt. % in toluene solution (purchased from sigma-aldrich) was added 0.87 mL anhydrous THF). Active layer were prepared at a 1:1.5 weight ratio in a 10 mg mL⁻¹ CB/DCB (6:4 volume ratio) solution (For PID-T4 and PffID-T4 polymers, a 3 v/v % DIO need to be added into the solution). To completely dissolve the polymer, the active layer solution should be stirred on a hot plate at 110 °C for at least 3 h. Before spin coating, both the polymer solution and ITO substrate are preheated on a hot plate at ~ 110 °C. Active layers were spin coated from the warm polymer solution on the preheated substrate at 600~700 r.p.m. to obtain thicknesses of ~ 190 nm (For PID-fft4 polymer, the substrate also needs to be attached onto a preheated metal chuck and spun together on the spincoater). The polymer/fullerene films were then annealed at 100 °C for 5 min before being transferred to the vacuum chamber. Then a thin layer (20 nm) of V₂O₅ was deposited as the anode interlayer under vacuum followed by 100 nm of Al as the top electrode. All cells were encapsulated using epoxy inside the glove box. For device characterization, $J-V$ characteristics were measured under AM1.5G (100 mW cm⁻²) using a Newport solar simulator. The light intensity was calibrated using a standard Si diode (with KG5 filter, purchased from PV Measurement) to bring spectral mismatch to unity. $J-V$ characteristics were recorded using a Keithley 236 source meter unit. Typical cells have devices area of 5.9 mm², which is defined by a metal mask with an aperture aligned with the device area. EQEs were characterized using a Newport EQE system equipped with a standard Si diode. Monochromatic light was generated from a Newport 300W lamp source.

AFM characterization. AFM measurements were performed by using a Scanning Probe Microscope-Dimension 3100 in tapping mode. All film samples were spin casted on ITO/ZnO substrates.

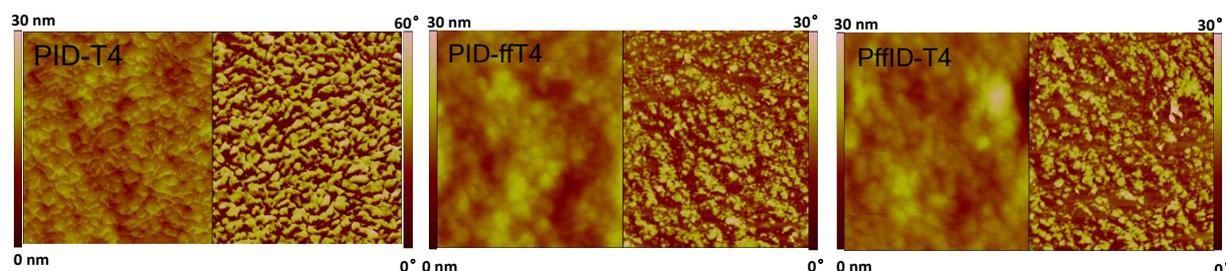


Fig. S1 AFM ($1\times 1\ \mu\text{m}$) images of Polymer:SMA blend films. The height and phase images are displayed on the left and right

sides, respectively.

Cyclic voltammetry. Cyclic voltammetry was performed in an electrolyte solution of 0.1 M tetrabutylammonium hexafluorophosphate in acetonitrile, both working and counter electrodes were platinum electrode. Ag/AgCl electrode was used as the reference electrode; the Fc/Fc⁺ redox couple was used as an external standard.

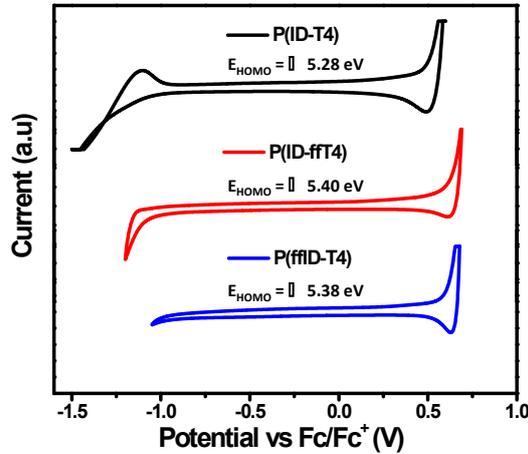


Fig. S2 Cyclic voltammetry results of the polymer

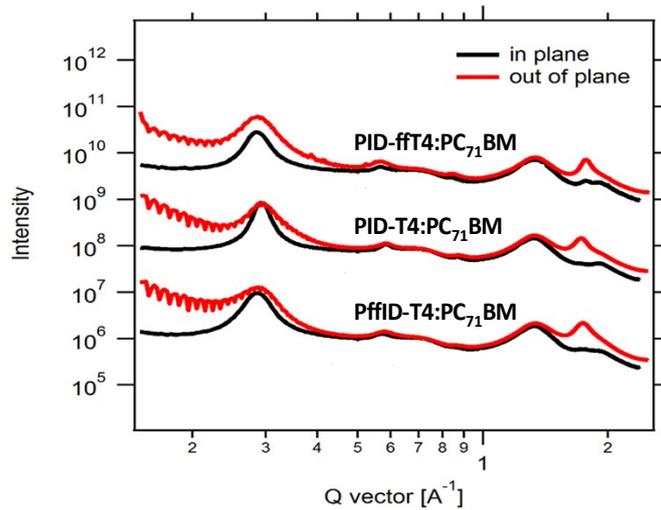


Fig. S3 1D line-curves of GIWAXS blend films.

Hole-only device. The hole mobility were measured using the space charge limited current (SCLC) method¹ by using a device architecture of ITO/V₂O₅/active layer (~100 nm)/V₂O₅/Al by taking current–voltage curves and fitting the results to a space

$$J = \frac{9\varepsilon_0\varepsilon_r\mu(V_{appl} - V_{bi} - V_s)^2}{8L^2}$$

charge limited form, where the SCLC is described by:

Where ε_0 is the permittivity of free space, ε_r is the dielectric constant of the polymer, μ is the hole mobility, V_{appl} is the applied voltage, V_{bi} is the built-in voltage (0 V)⁴, V_s is the voltage drop from the substrate's series resistance ($V_s = IR$, R is measured to be 10.8 Ω) and L is the thickness of the polymer. The dielectric constant ε_r is assumed to be 3, which is a typical value for conjugated polymers. The $J^{1/2} \sim (V_{appl} - V_{bi} - V_s)$ characteristics are obtained by linearly fitting $J^{1/2}$ with $(V_{appl} - V_{bi} - V_s)$, the mobilities were extracted from the slope and L :

$$\mu = \frac{slope^2 \times 8L^2}{9\varepsilon_0\varepsilon_r}$$

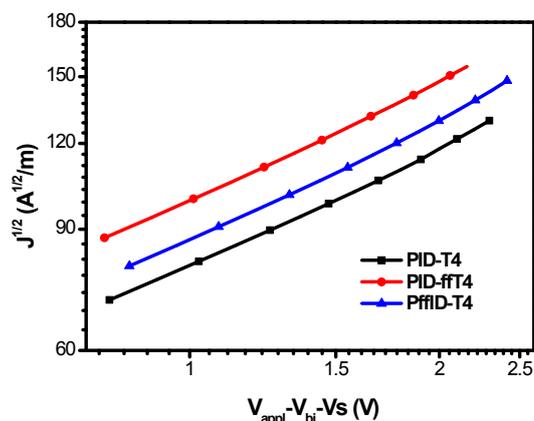


Fig. S4 J - V curves of the hole-only device of Polymers:SMA blend films.

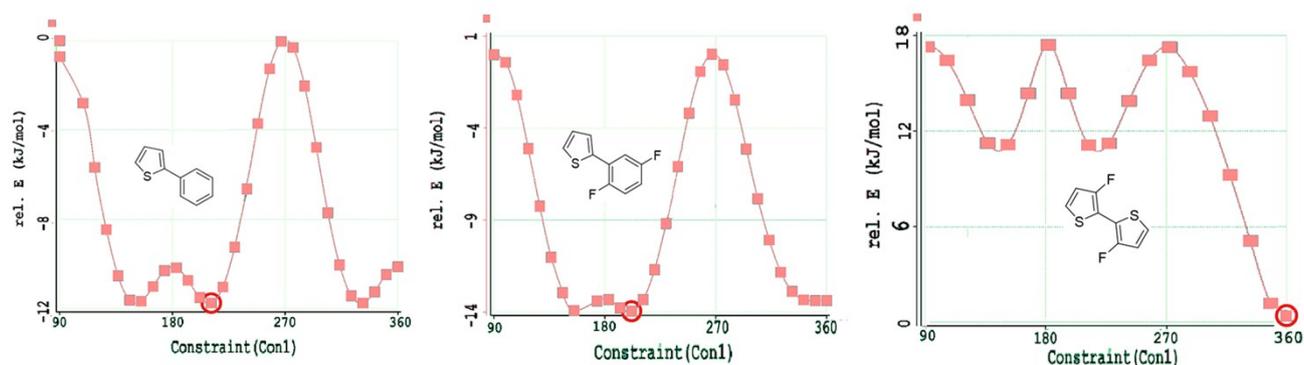


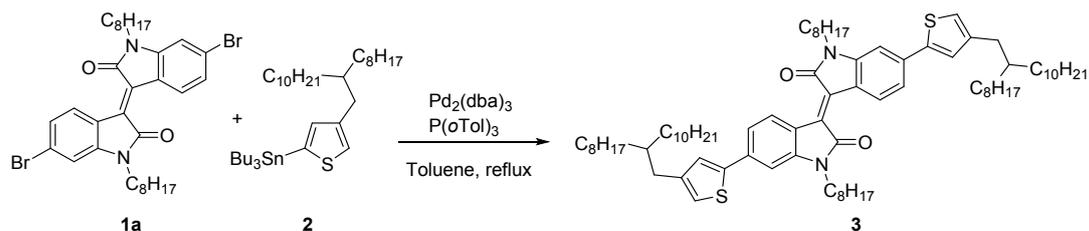
Fig. S5. Calculated energy profile of different conformations (DFT calculations were performed using the B3LYP functional and the 6-31 G* basis for the polymers).

General Information for polymer synthesis and characterization.

All reagents and chemicals were purchased from commercial sources and used without further purification unless stated otherwise. Toluene was freshly distilled before use from sodium. Anhydrous chlorobenzene were purchased from Sigma-Aldrich. ^1H and ^{13}C NMR spectra were recorded on a Bruker AV-400 MHz NMR spectrometer. Chemical shifts are reported in parts per million (ppm, δ). ^1H NMR and ^{13}C NMR spectra were referenced to tetramethylsilane (0 ppm) for CDCl_3 . Mass spectra were collected on a MALDI Micro MX mass spectrometer, or an API QSTAR XL System.

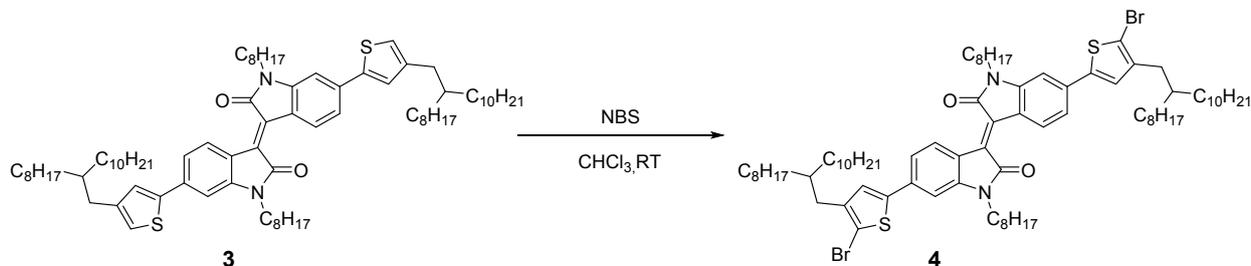
6,6'-Dibromoisindigo², (*E*)-6,6'-dibromo-1,1'-dioctyl-[3,3'-biindolinylidene]-2,2'-dione (**1a**)³ and (*E*)-6,6'-dibromo-7,7'-difluoro-1,1'-dioctyl-[3,3'-biindolinylidene]-2,2'-dione (**1b**)⁴ were synthesized according to literature procedures.

Monomer synthesis and characterization:



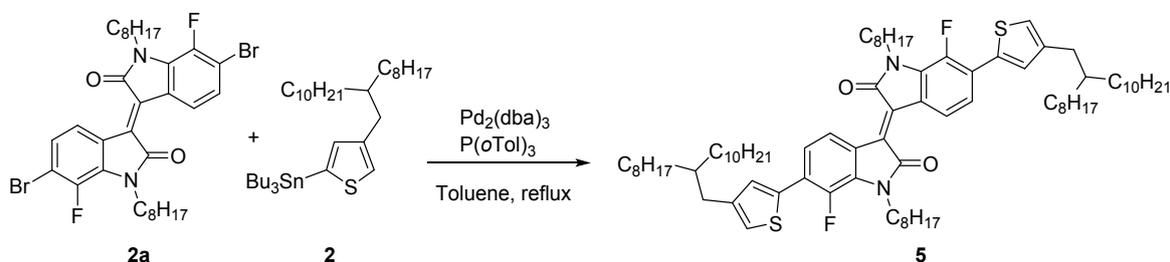
(E)-1,1'-dioctyl-6,6'-bis(4-(2-octyldodecyl)thiophen-2-yl)-[3,3'-biindolinylidene]-2,2'-dione (3).

To a solution of **1a** (258 mg, 0.4 mmol), Pd₂(dba)₃ (7 mg, 0.008 mmol) and P(*o*-tol)₃ (10 mg, 0.03 mmol) in 20 mL toluene was added **2** (654 mg, 1.0 mmol) under N₂, the reaction was refluxed overnight. After the reaction mixture was cooled to r.t., a solution of KF in water was added and the organic phase was washed with water for three times, then dried with Na₂SO₄. The solvent was evaporated and the residue was purified by flash column chromatography (eluent: *n*-hexane/DCM = 3:1) to give **3** as a dark solid (368 mg, 76 %). ¹H NMR (400 MHz, CDCl₃): δ 9.16 (d, *J* = 8.4 Hz, 2H), 7.28 (dd, *J* = 8.4 Hz, 1.6 Hz, 2H), 7.22 (d, *J* = 1.6 Hz, 2H), 6.95 (s, 2H), 6.92 (s, 2H), 3.81 (t, *J* = 7.2 Hz, 4H), 2.52 (d, *J* = 6.8 Hz, 4H), 1.75 – 1.65 (m, 6H), 1.43 – 1.15 (m, 84H), 0.92 – 0.75 (m, 18H). ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 145.3, 143.5, 143.4, 138.2, 131.9, 130.3, 126.2, 121.8, 121.0, 119.2, 104.6, 40.1, 38.8, 35.1, 33.3, 31.9, 30.1, 29.7, 29.6, 29.4, 29.3, 29.2, 27.6, 27.1, 26.6, 22.7, 22.6, 14.1, 14.0. HRMS (MALDI+) Calcd for C₈₀H₁₂₆N₂O₂S₂ (M⁺): 1210.9261, Found: 1210.9240.



(E)-6,6'-bis(5-bromo-4-(2-octyldodecyl)thiophen-2-yl)-1,1'-dioctyl-[3,3'-biindolinylidene]-2,2'-dione (4).

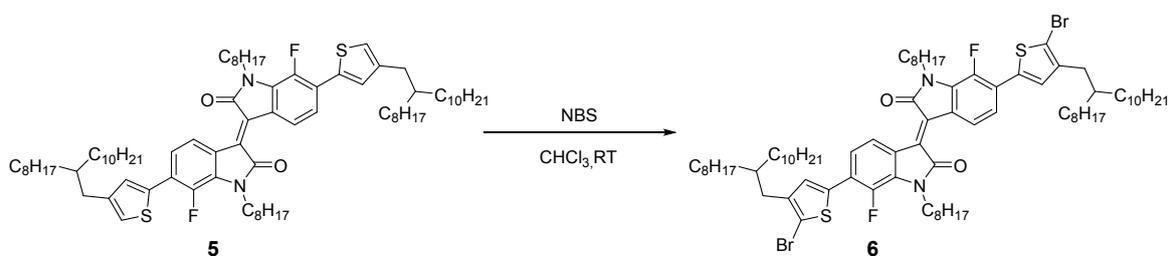
To a solution of **3** (243 mg, 0.2 mmol) in 10 mL CHCl₃ was added NBS (71 mg, 0.4 mmol) at 0 °C, the reaction was stirred overnight, the mixture was washed with water for three times, then dried with Na₂SO₄. The solvent was evaporated and the residue was purified by flash column chromatography (eluent: *n*-hexane/DCM = 3:1) to give **4** as a dark solid (250 mg, 80 %). ¹H NMR (400 MHz, CDCl₃) δ 9.17 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.06 (s, 2H), 6.85 (s, 2H), 3.81 (t, *J* = 7.2 Hz, 4H), 2.52 (d, *J* = 6.8 Hz, 4H), 1.75 – 1.65 (m, 6H), 1.43 – 1.15 (m, 84H), 0.92 – 0.75 (m, 18H). ¹³C NMR (100 MHz, CDCl₃) δ 168.2, 145.4, 143.0, 142.8, 137.2, 131.9, 130.5, 125.6, 121.2, 118.8, 110.6, 104.2, 40.1, 38.6, 34.4, 33.4, 31.9, 30.1, 29.7, 29.6, 29.4, 29.3, 29.2, 27.6, 27.0, 26.5, 22.7, 22.6, 14.1, 14.0; HRMS (MALDI+) Calcd for C₈₀H₁₂₄Br₂N₂O₂S₂ (M⁺): 1366.7471, Found: 1366.7428.



(E)-7,7'-difluoro-1,1'-dioctyl-6,6'-bis(4-(2-octyldodecyl)thiophen-2-yl)-[3,3'-biindolinylidene]-2,2'-dione (4).

To a solution of **2a** (272 mg, 0.4 mmol), Pd₂(dba)₃ (7 mg, 0.008 mmol) and P(*o*-tol)₃ (10 mg, 0.03 mmol) in 20 mL toluene was added **2** (654 mg, 1.0 mmol) under N₂, the reaction was refluxed overnight. After the reaction mixture was cooled to r.t., a solution of KF in water was added and the organic phase was washed with water for three times, then dried with

Na₂SO₄. The solvent was evaporated and the residue was purified by flash column chromatography (eluent: *n*-hexane/DCM = 3:1) to give **5** as a dark solid (359 mg, 72 %). ¹H NMR (400 MHz, CDCl₃): δ 8.94 (d, *J* = 8.4 Hz, 2H), 7.37 (s, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.01 (s, 2H), 3.99 (t, *J* = 7.2 Hz, 4H), 2.52 (d, *J* = 6.8 Hz, 4H), 1.75 – 1.65 (m, 6H), 1.43 – 1.15 (m, 84H), 0.92 – 0.75 (m, 18H). ¹³C NMR (100 MHz, CDCl₃): δ 167.7, 144.3 (d, *J* = 245.0 Hz), 142.5, 136.6, 133.2, 129.0 (d, *J* = 7.2 Hz), 126.0, 125.8, 123.5, 113.2, 112.5, 42.6, 38.4, 34.4, 33.5, 31.9, 31.8, 30.1, 29.8, 29.6, 29.4, 29.3, 29.2, 29.0, 26.9, 26.5, 22.7, 22.6, 14.1, 14.0; HRMS (MALDI⁺) Calcd for C₈₀H₁₂₄F₂N₂O₂S₂ (M⁺): 1246.9072, Found: 1246.9056.

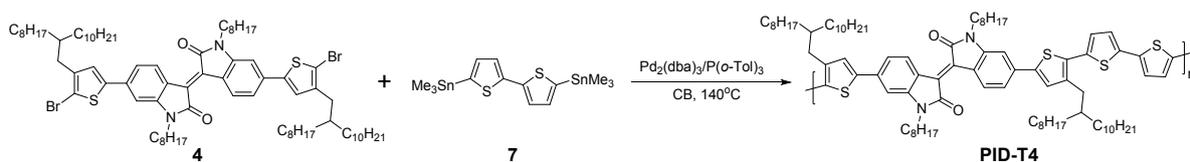


(*E*)-6,6'-bis(5-bromo-4-(2-octyldodecyl)thiophen-2-yl)-7,7'-difluoro-1,1'-dioctyl-[3,3'-biindolylidene]-2,2'-dione (6**).**

To a solution of **5** (250 mg, 0.2 mmol) in 10 mL CHCl₃ was added NBS (71 mg, 0.4 mmol) at 0 °C, the reaction was stirred overnight, the mixture was washed with water for three times, then dried with Na₂SO₄. The solvent was evaporated and the residue was purified by flash column chromatography (eluent: *n*-hexane/DCM = 3:1) to give **4** as a dark solid (233 mg, 85 %). ¹H NMR (400 MHz, CDCl₃) δ 8.92 (d, *J* = 8.4 Hz, 2H), 7.20 (s, 2H), 7.14 (dd, *J* = 8.4 Hz, 6.4 Hz, 2H), 3.97 (t, *J* = 7.2 Hz, 4H), 2.52 (d, *J* = 6.8 Hz, 4H), 1.75 – 1.65 (m, 6H), 1.43 – 1.15 (m, 84H), 0.92 – 0.75 (m, 18H). ¹³C NMR (100 MHz, CDCl₃) δ 167.6, 144.4 (d, *J* = 245.0 Hz), 142.3, 136.2, 132.4, 129.0 (d, *J* = 7.0 Hz), 126.1, 125.9, 123.1, 120.2, 112.4, 42.5, 38.5, 34.3, 33.4, 31.9, 31.8, 30.0, 29.7, 29.6, 29.4, 29.3, 29.2, 29.1, 26.8, 26.5, 22.7, 22.6, 14.0, 13.9; HRMS (MALDI⁺) Calcd for C₈₀H₁₂₂Br₂F₂N₂O₂S₂ (M⁺): 1402.7283, Found: 1402.7291.

Polymer synthesis and characterization:

Synthesis of PID-T4:

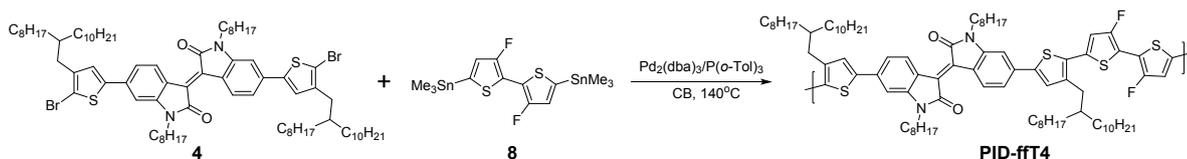


To a 10 mL of sealed tube were added monomer **4** (54.8 mg, 0.04 mmol), monomer **7** (19.7 mg, 0.04 mmol), tris(dibenzylideneacetone)dipalladium (1.0 mg), *tri-o*-tolylphosphine (2.0 mg) and CB (0.3 mL) under N₂. The mixture was vigorously stirred at 140 °C for 24 h. After cooling to room temperature, the reaction mixture was poured into 100 mL methanol. The precipitate was collected and further purified by Soxhlet extraction with methanol, acetone and chloroform successively. The polymer was recovered as a solid from the chlorobenzene fraction to afford the product as a dark green solid (38 mg, 70%).

¹H NMR (400 MHz, C₂D₂Cl₄, 120 °C). δ 9.25 (d, *J* = 8.8 Hz, 2H), 7.32 (d, *J* = 7.2 Hz, 2H), 7.27 (s, 2H), 7.20 (d, *J* = 2.8 Hz, 2H), 7.14 (d, *J* = 2.8 Hz, 2H), 6.99 (s, 2H), 3.95 – 3.75 (m, 4H), 2.85 – 2.75 (m, 4H), 1.90 – 1.75 (m, 6H), 1.55 – 1.10 (m, 84H), 0.95 – 0.80 (m, 18H).

GPC *M_n* = 55.6 kDa; *M_w* = 89.5 kDa; PDI = 1.6

Synthesis of PID-fft4:

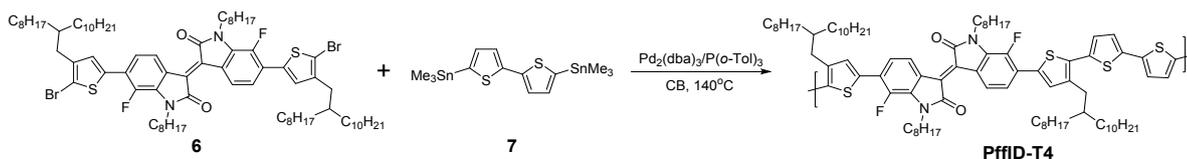


To a 10 mL of sealed tube were added monomer 4 (54.8 mg, 0.04 mmol), monomer 8 (121.1 mg, 0.04 mmol), tris(dibenzylideneacetone)dipalladium (1.0 mg), *tri-o*-tolylphosphine (2.0 mg) and CB (0.3 mL) under N₂. The mixture was vigorously stirred at 140 °C for 24 h. After cooling to room temperature, the reaction mixture was poured into 100 mL methanol. The precipitate was collected and further purified by Soxhlet extraction with methanol, acetone, chloroform and chlorobenzene successively. The polymer was recovered as a solid from the chlorobenzene fraction to afford the product as a dark green solid (38 mg, 70%).

¹H NMR (400 MHz, C₂D₂Cl₄, 120 °C). δ 9.08 (d, J = 7.6 Hz, 2H), 7.46 (s, 2H), 7.35 – 7.30 (m, 2H), 7.23 (s, 2H), 7.20 (d, 2H), 4.15 – 4.00 (m, 4H), 2.90 – 2.80(m, 4H), 1.95 – 1.75 (m, 6H), 1.55 – 1.10 (m, 84H), 0.95 – 0.80 (m, 18H).

GPC M_n = 52.4 kDa; M_w = 78.7 kDa; PDI = 1.5

Synthesis of PffID-T4:



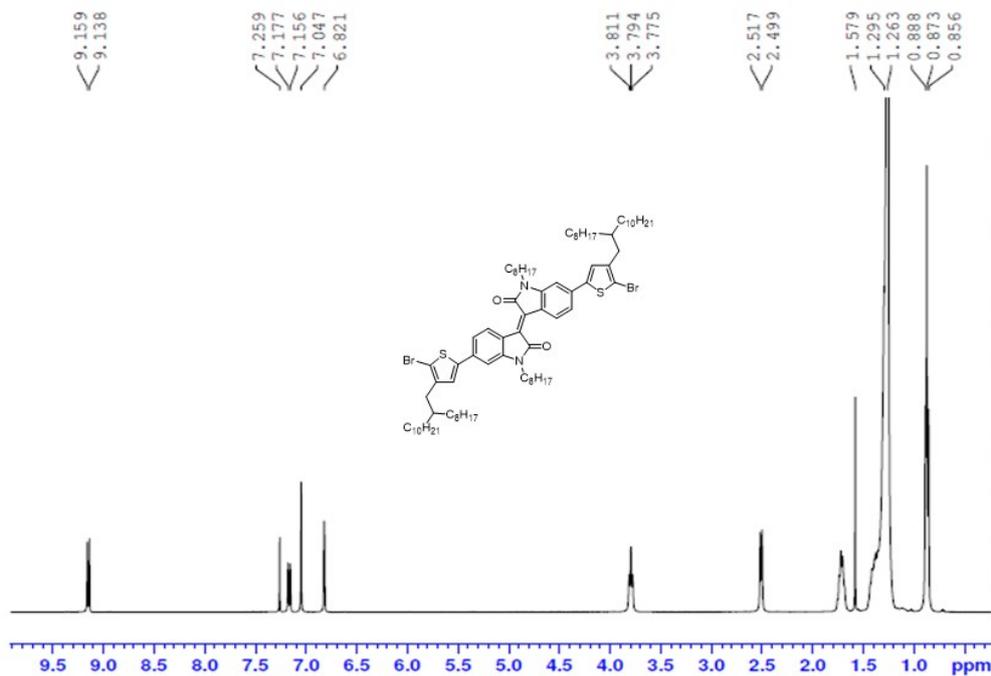
To a 10 mL of sealed tube were added monomer 4 (56.2 mg, 0.04 mmol), monomer 8 (19.7 mg, 0.04 mmol), tris(dibenzylideneacetone)dipalladium (1.0 mg), *tri-o*-tolylphosphine (2.0 mg) and CB (0.3 mL) under N₂. The mixture was vigorously stirred at 140 °C for 24 h. After cooling to room temperature, the reaction mixture was poured into 100 mL methanol. The precipitate was collected and further purified by Soxhlet extraction with methanol, acetone, chloroform and chlorobenzene successively. The polymer was recovered as a solid from the chlorobenzene fraction to afford the product as a dark green solid (34 mg, 60%).

¹H NMR (400 MHz, C₂D₂Cl₄, 120 °C). δ 8.35 (d, J = 4.0 Hz, 2H), 8.21 (s, 2H), 7.40 (d, J = 4.0 Hz, 2H), 7.21 (s, 2H), 2.94 (t, J = 6.8 Hz, 8H), 1.95 – 1.85 (m, 4H), 1.56 – 1.30 (m, 96H), 1.00 – 0.86 (m, 24H).

GPC M_n = 48.6 kDa; M_w = 78.0 kDa; PDI = 1.6

Supplementary References:

1. J. T. Bloking, T. Giovenzana, A. T. Higgs, A. J. Ponec, E. T. Hoke, K. Vandewal, S. Ko, Z. Bao, A. Sellinger and M. D. McGehee, *Adv. Energy Mater.*, 2014, **4**, 1301426.
2. T. Lei, Y. Cao, Y. Fan, C. J. Liu, S. C. Yuan and J. Pei, *J. Am. Chem. Soc.*, 2011, **133**, 6099-6101.
3. Z. Ma, W. Sun, S. Himmelberger, K. Vandewal, Z. Tang, J. Bergqvist, A. Salleo, J. W. Andreasen, O. Inganäs, M. R. Andersson, C. Müller, F. Zhang and E. Wang, *Energy Environ. Sci.*, 2014, **7**, 361-369.
4. T. Lei, J. H. Dou, Z. J. Ma, C. H. Yao, C. J. Liu, J. Y. Wang and J. Pei, *J. Am. Chem. Soc.*, 2012, **134**, 20025-20028.

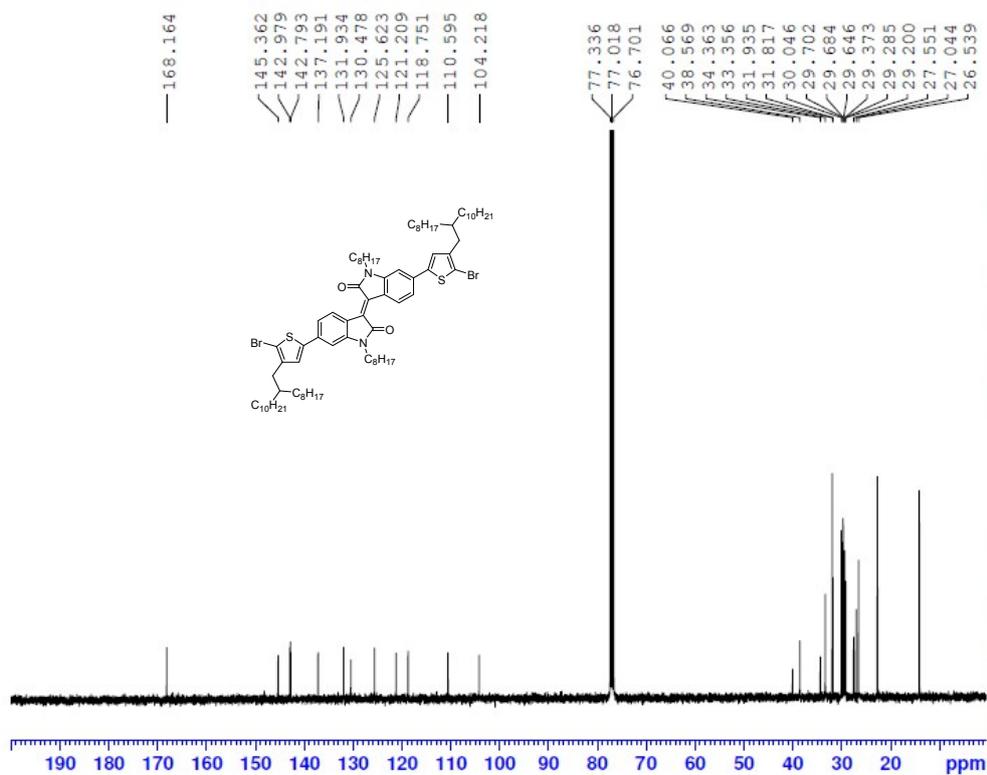


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 DE 6.50 usec
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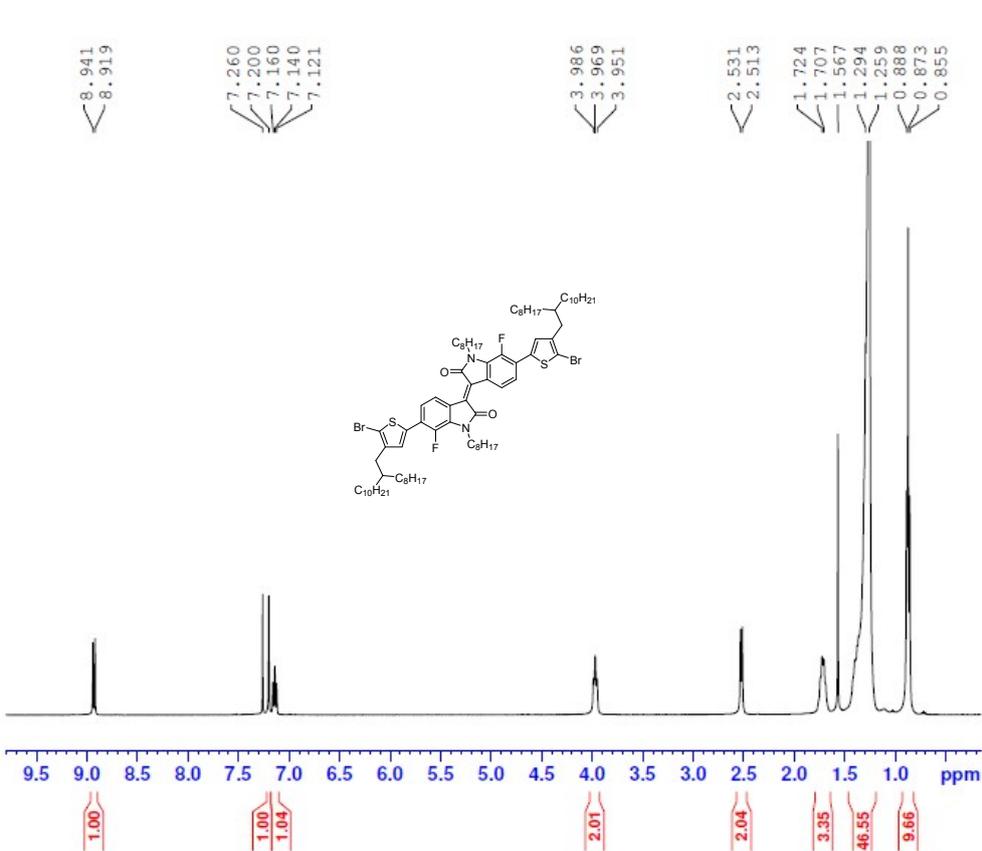
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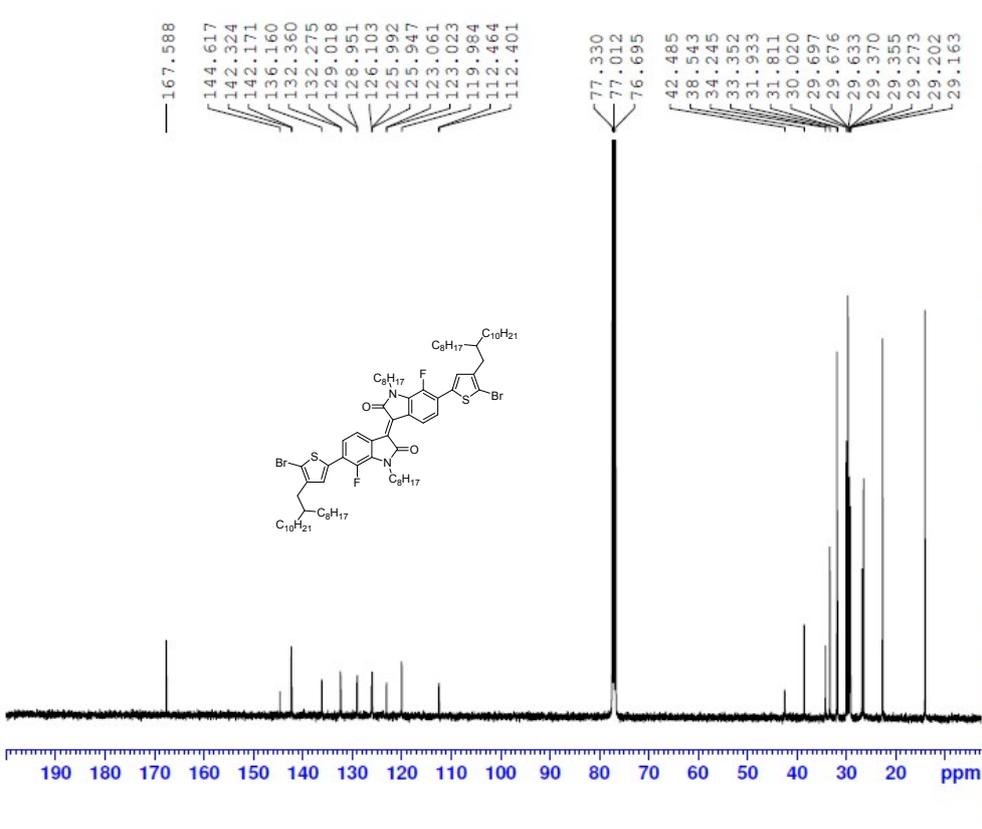
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BRUKER

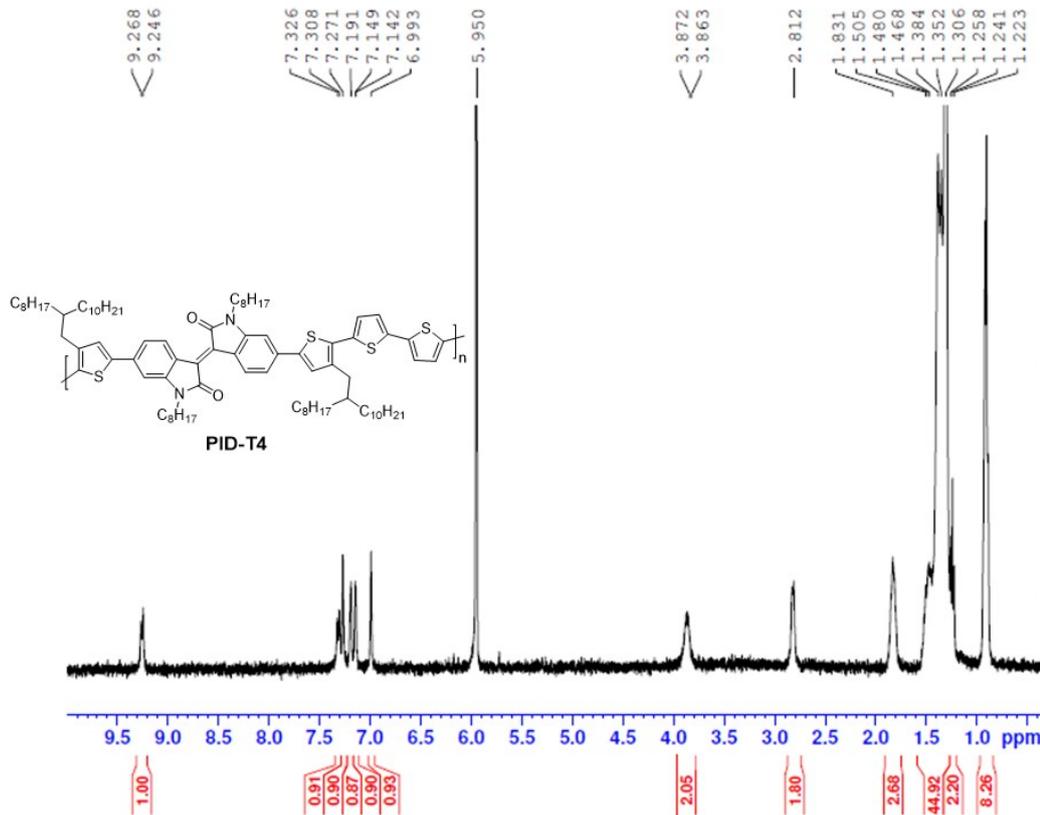
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 TDO 1

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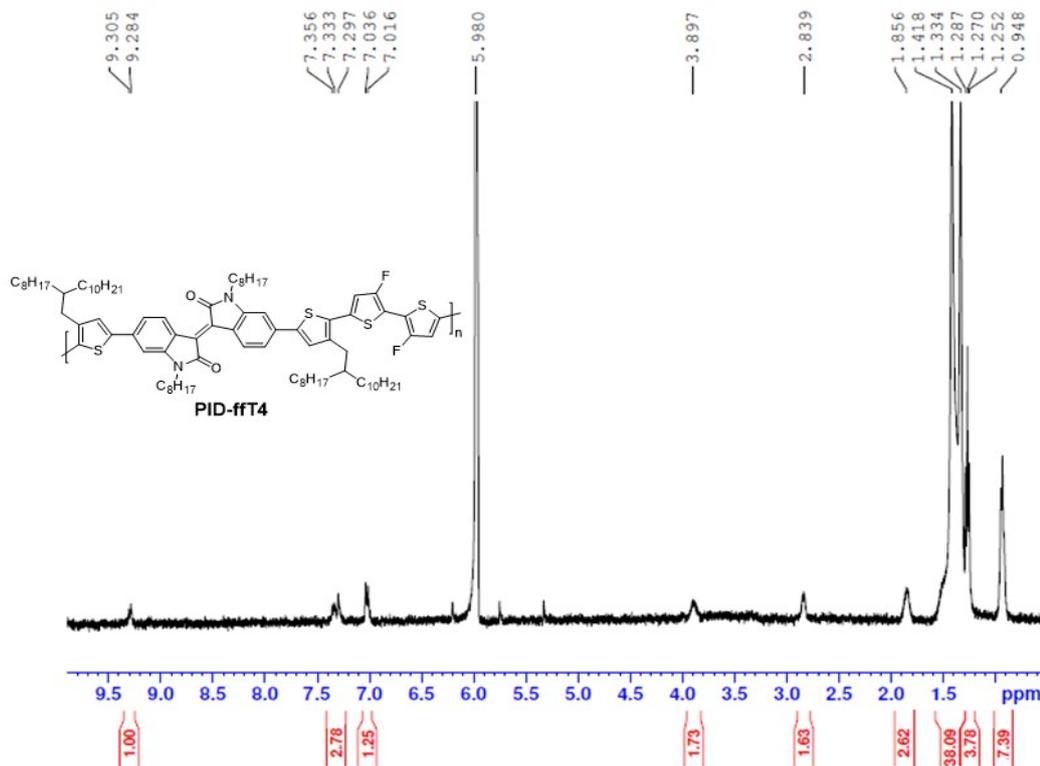


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 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 196.92
 DW 62.400 usec
 DE 6.50 usec
 TE 383.2 K
 D1 1.00000000 sec
 TD0 1

CHANNEL f1
 SF01 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1305297 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

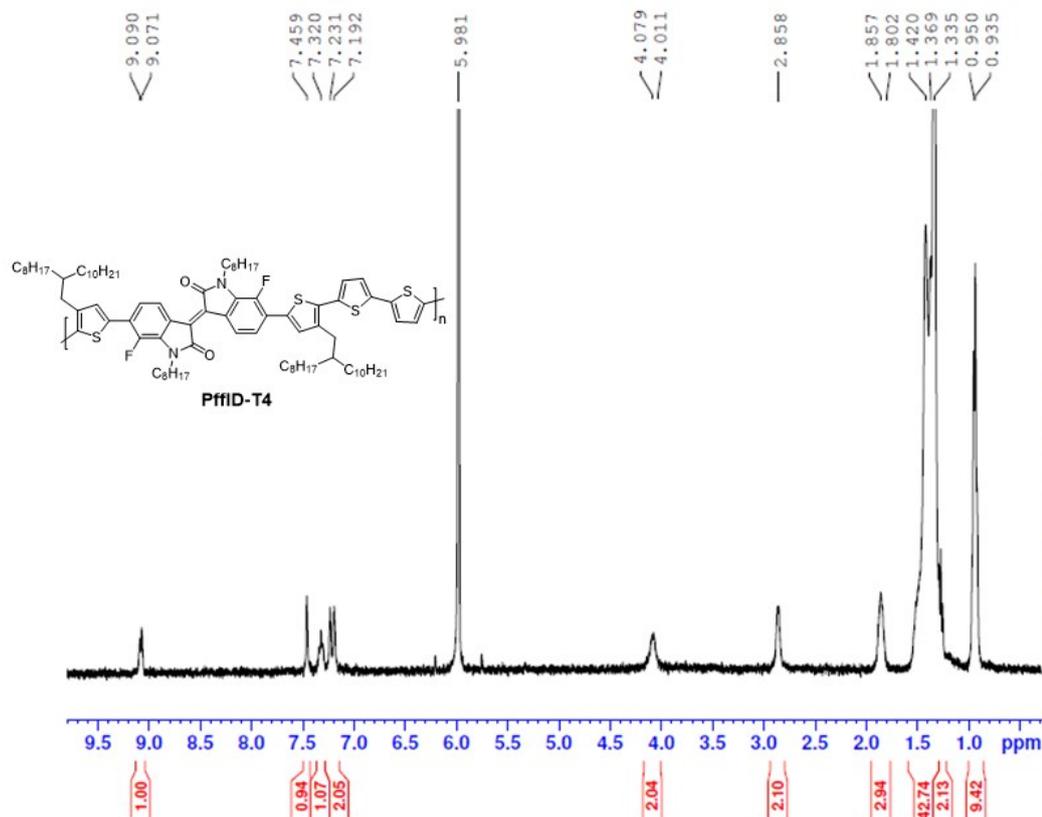


Current Data Parameters
 NAME S6
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160125
 Time 19.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 128
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 383.2 K
 D1 1.00000000 sec
 TD0 1

CHANNEL f1
 SF01 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1305185 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
NAME S20
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160120
Time 19.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 64
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 196.92
DW 62.400 usec
DE 6.50 usec
TE 383.2 K
D1 1.0000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLM1 11.99499989 W

F2 - Processing parameters
SI 65536
SF 400.1305179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00