Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Synthesis and Structure-Property Relationships of Phthalimide and Napthalimide based Organic π-Conjugated Small Molecules

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SUPPORTING INFORMATION

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Materials and methods

Materials: Pivalic acid (PivOH) and 4-bromophthalic anhydride were purchased from TCI America. Tetrakis(triphenylphosphine) palladium(0) catalyst ($Pd(PPh_3)_4$) and SiliaCat® heterogeneous catalyst DPP-Pd were bought from Strem Chemicals and SiliCycle respectively.. Anhydrous potassium carbonate was purchased from ACP chemicals, glacial acetic acid (AcOH) from Fisherbrand, and anhydrous *N*,*N*'-dimethylformamide (DMF) from EMD Millipore. The remaining reagents were bought from Sigma-Aldrich. All solvents and materials purchased were used without further purification.

Microwave-Assisted Synthesis: All microwave reactions were carried out using a Biotage® Initiator+ microwave reactor. The operational power range of the instrument is 0–400 W, using a 2.45 GHz magnetron. Pressurized air is used to cool each reaction after microwave heating.

Nuclear Magnetic Resonance (NMR): ^1H and $^{13}C\{^1H\}$ NMR spectroscopy spectra were recorded on a Bruker Avance-300 MHz spectrometer at 300K. Chemical shifts are reported in parts per million (ppm) and are referenced to the external standard $SiMe_4$. Multiplicities are reported as: singlet (s), doublet (d), triplet (t), multiplet (m), and overlapping (ov).

Mass Spectrometry (MS): Mass spectrometry measurements were performed courtesy of Xiao Feng at the Dalhousie University Analytical Laboratory with a Bruker-Daltronics Micro TOF Mass Spectrometer. Atmospheric pressure chemical ionization (APCI) was used to ionize samples with a corona discharge voltage of 4000 V and a vaporizer temperature of 375 ºC.

UV-Visible Spectroscopy (UV-Vis): All absorption measurements were recorded using Agilent Technologies Cary 60 UV-Vis spectrometer at room temperature. All solution UV-Vis experiments were run in $CHCl_3$ using 10 mm quartz cuvettes. Neat films were prepared by spin-coating solutions from $CHCl_3$ onto glass substrates cut from Corning Micro slides. Films were annealed by direct mounting on a VWR hotplate.

Photoluminescence (PL): All emission measurements were recorded using Agilent Technologies Cary Eclipse Fluorescence spectrophotometer at room temperature. All solution PL experiments were run in $CHCl_3$ using 10 mm quartz cuvettes.

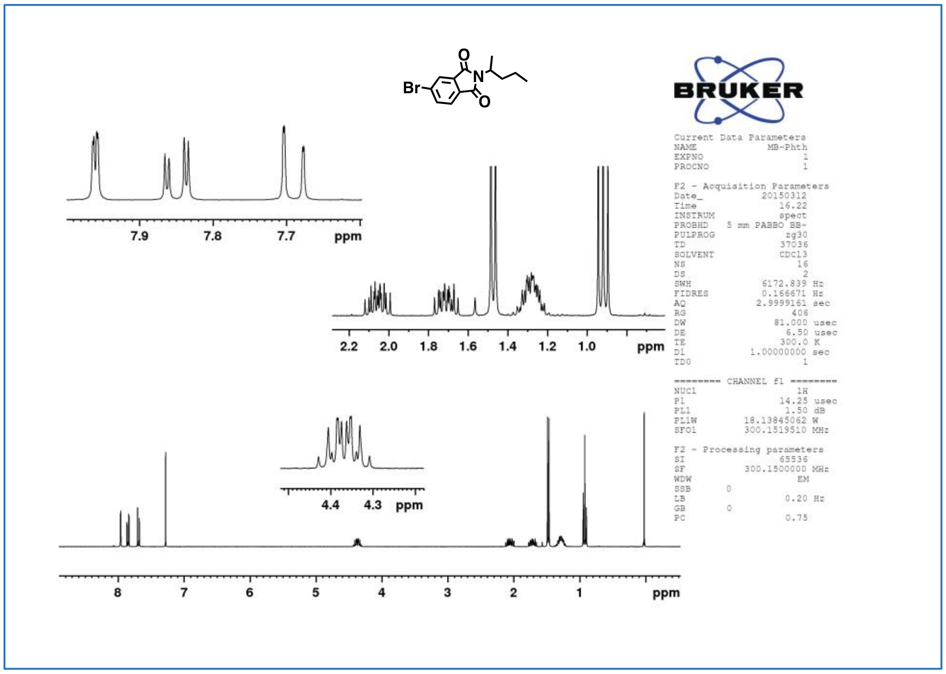
Differential Scanning Calorimeter (DSC): DSC was performed on a TA instruments Q-1000 DSC instrument with compressed air as the purging gas. Samples were heated from 50 °C to 300 °C for 3 cycles under air unless otherwise specified.

Ellipsometry: In this work, a VASE setup, M2000 by JA Wollam was employed.Thin film samples for VASE analysis were prepared by spin casting hot 10 mg/mL solutions of **1-6** in $CHCl_3$ onto heated, pre-cleaned ~300 nm SiO_2 layer terminated silicon wafers at 6000 RPM for 30s.

Ellipsometry data was collected over the 250-1000 nm range for each of the six films at three different incident angles (55 °, 65 °, 75 °). Using the CompleteEASE software package provided with the ellipsometry equipment, the substrate was modelled as a SiO_2 terminated silicon wafer and the organic layer was modelled using a uniaxial anisotropic B-Spline model. Representative fits of the model to the data are provided in **Figure S21**.

Single Crystal X-Ray Diffraction. X-ray crystallographic data collection was carried out using APEX2, cell refinement using SAINT, and data reduction using SAINT.[2] Structure identification was completed using SHELXS97.[3] Structure refinement software packages used included SHELXL97 and Olex2.[4] Molecular graphics were created using the Mercury software package. Intermolecular π-π stacking distances between adjacent molecules were calculated using the Olex2 software package by calculating the plane to plane centroid distances for the mean planes defined by the 4 carbon atoms in each of the symmetry equivalent thiophene units on adjacent (π-stacked) molecules for **1-6**.The angles at which the end-capping units and the bithiophene cores are twisted were calculated in a similar manner, first the mean plane of each ring was defined using all carbon atoms in the ring, followed by the use of the 'esd' command to generate the plane to plane angles.

Solution ^{1}H & ^{13}C NMR spectra



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EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
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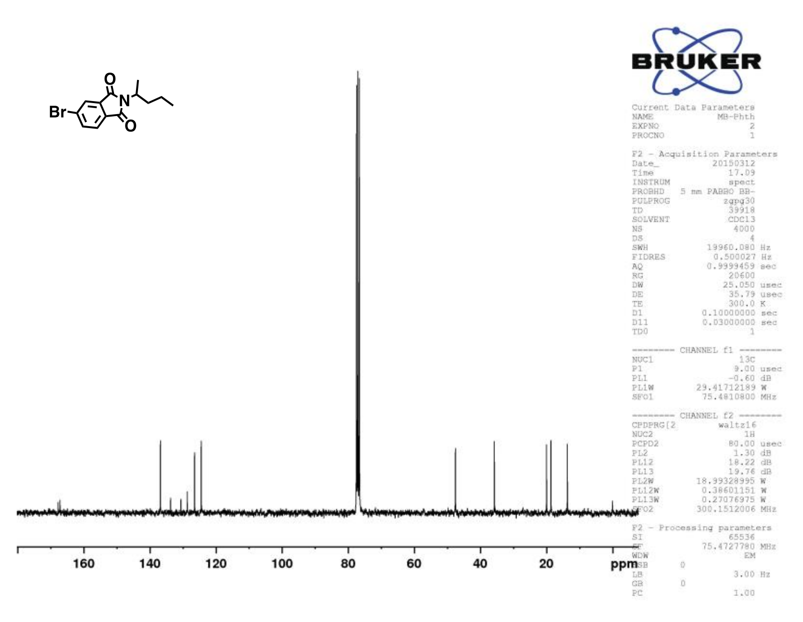
PULPROG              zg30
TD                  37036
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.166671 Hz
AQ              2.9999161 sec
RG                    406
DW                 81.000 usec
DE                   6.50 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.25 usec
PL1                  1.50 dB
PL1W          18.13845062 W
SFO1          300.1519510 MHz

F2 - Processing parameters
SI                  65536
SF            300.1500000 MHz
WDW                    EM
SSB      0
LB                   0.20 Hz
GB       0
PC                   0.75
```

Figure S1. ^{1}H NMR spectra of **MB-Phth** in $CDCl_3$.



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EXPNO                    2
PROCNO                   1

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TE                   300.0 K
D1             0.10000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.00 usec
PL1                  -0.60 dB
PL1W           29.41712189 W
SFO1            75.4810800 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.30 dB
PL12                 18.22 dB
PL13                 19.76 dB
PL2W           18.99328995 W
PL12W           0.38601151 W
PL13W           0.27076975 W
SFO2           300.1512006 MHz

F2 - Processing parameters
SI                   65536
SF              75.4727780 MHz
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PC                    1.00
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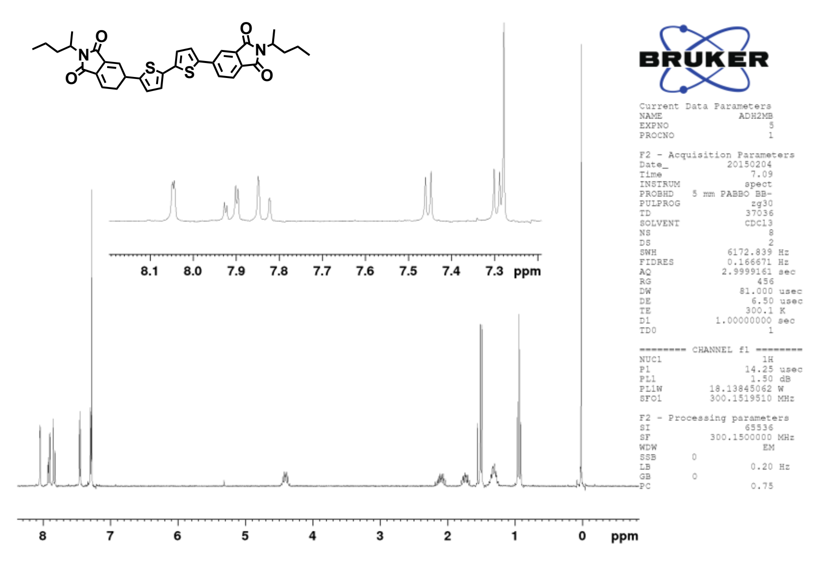
Figure S2. ^{13}C NMR spectra of **MB-Phth** in $CDCl_3$.



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PROCNO                   1

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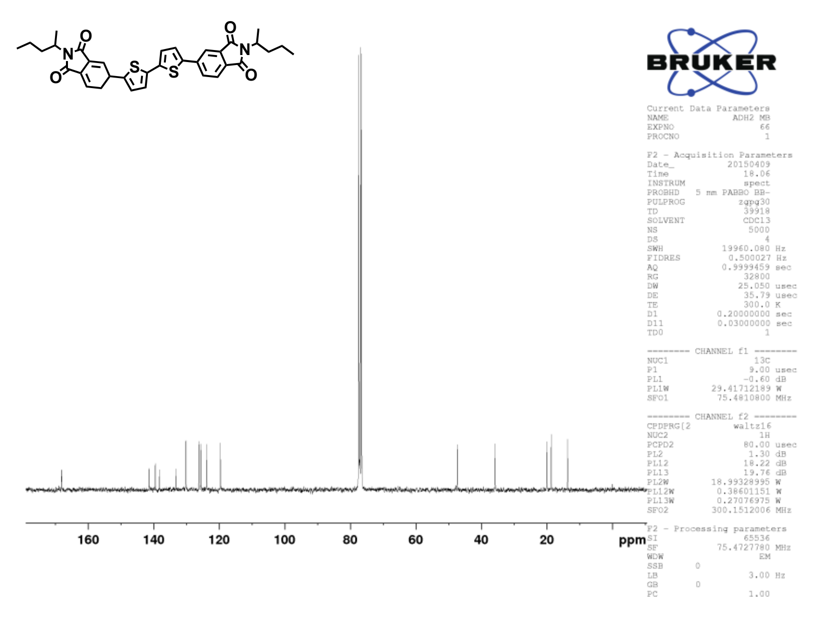
SOLVENT              CDCl3
NS                       8
DS                       2
SWH               6172.839 Hz
FIDRES            0.166671 Hz
AQ               2.9999161 sec
RG                     456
DW                  81.000 usec
DE                    6.50 usec
TE                   300.1 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.25 usec
PL1                   1.50 dB
PL1W           18.13845062 W
SFO1           300.1519510 MHz

F2 - Processing parameters
SI                   65536
SF             300.1500000 MHz
WDW                     EM
SSB      0
LB                    0.20 Hz
GB       0
PC                    0.75
```

Figure S3. ^{1}H NMR spectra of **2** in $CDCl_3$.



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PROCNO                  1

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TD                  39918
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NS                   5000
DS                      4
SWH             19960.080 Hz
FIDRES           0.500027 Hz
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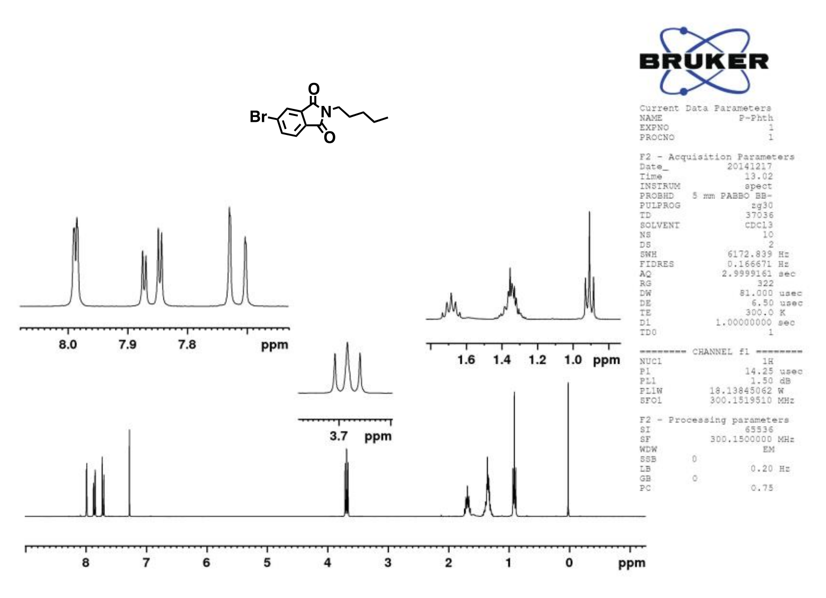
RG                  32800
DW                 25.050 usec
DE                  35.79 usec
TE                  300.0 K
D1             0.20000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                 -0.60 dB
PL1W          29.41712189 W
SFO1           75.4810800 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  1.30 dB
PL12                18.22 dB
PL13                19.76 dB
PL2W          18.99328995 W
PL12W          0.38601151 W
PL13W          0.27076975 W
SFO2          300.1512006 MHz

F2 - Processing parameters
SI                  65536
SF             75.4727780 MHz
WDW                    EM
SSB      0
LB                   3.00 Hz
GB       0
PC                   1.00
```

Figure S4. ^{13}C NMR spectra of **2** in $CDCl_3$.



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Current Data Parameters
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EXPNO                   1
PROCNO                  1

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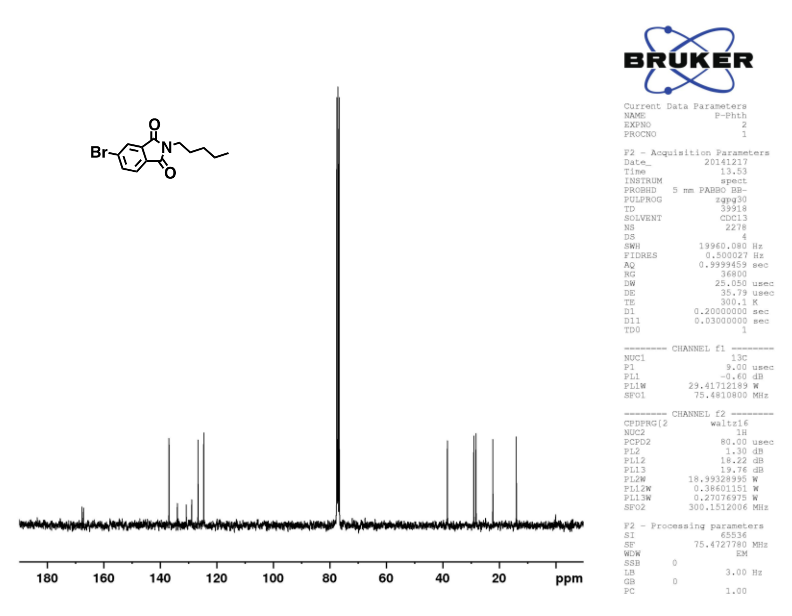
PULPROG              zg30
TD                  37036
SOLVENT             CDCl3
NS                     10
DS                      2
SWH              6172.839 Hz
FIDRES           0.166671 Hz
AQ              2.9999161 sec
RG                    322
DW                 81.000 usec
DE                   6.50 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.25 usec
PL1                  1.50 dB
PL1W          18.13845062 W
SFO1          300.1519510 MHz

F2 - Processing parameters
SI                  65536
SF            300.1500000 MHz
WDW                    EM
SSB      0
LB                   0.20 Hz
GB       0
PC                   0.75
```

Figure S5. ^{1}H NMR spectra of **P-Phth** in $CDCl_3$.



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EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
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PULPROG             zgpg30
TD                   39918
SOLVENT              CDCl3
NS                    2278
DS                       4
SWH             19960.080 Hz
FIDRES           0.500027 Hz
AQ              0.9999459 sec
RG                   36800
DW                  25.050 usec
DE                   35.79 usec
TE                   300.1 K
D1             0.20000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.00 usec
PL1                  -0.60 dB
PL1W          29.41712189 W
SFO1          75.4810800 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.30 dB
PL12                 18.22 dB
PL13                 19.76 dB
PL2W          18.99328995 W
PL12W          0.38601151 W
PL13W          0.27076975 W
SFO2         300.1512006 MHz

F2 - Processing parameters
SI                   65536
SF             75.4727780 MHz
WDW                     EM
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GB       0
PC                    1.00
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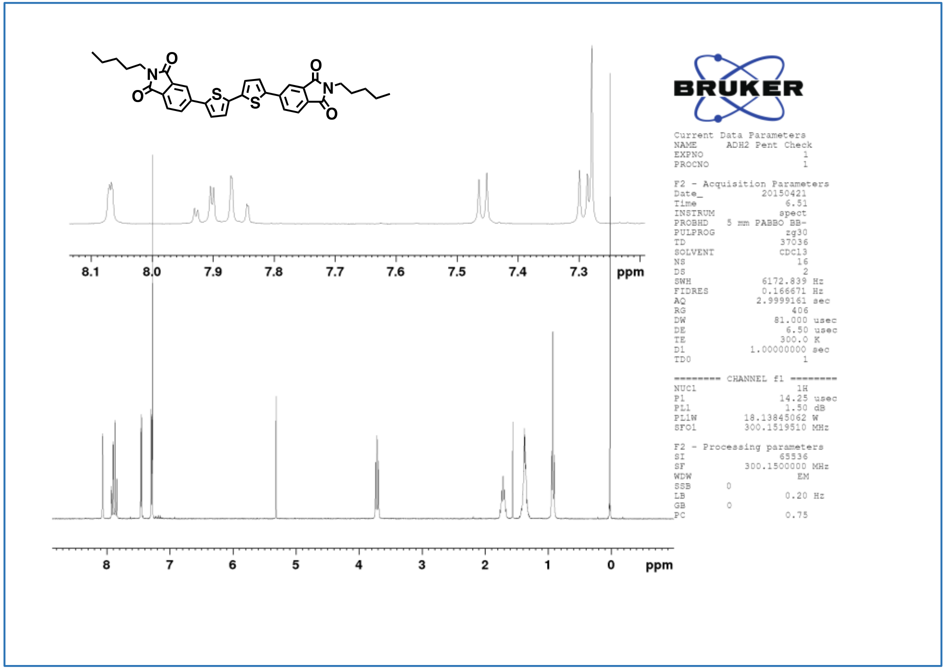
Figure S6. ^{13}C NMR spectra of **P-Phth** in $CDCl_3$.



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PROCNO                    1

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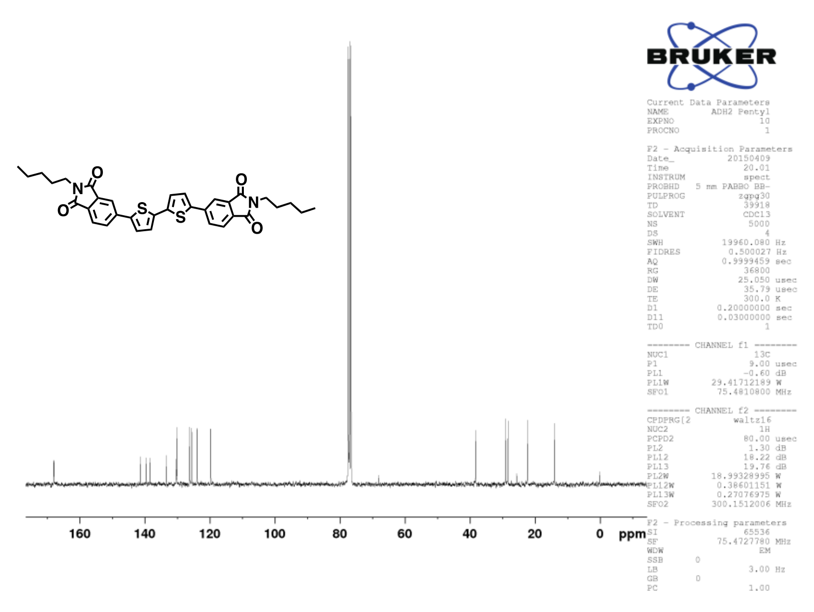
TD                    37036
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6172.839 Hz
FIDRES             0.166671 Hz
AQ                2.9999161 sec
RG                      406
DW                   81.000 usec
DE                     6.50 usec
TE                    300.0 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.25 usec
PL1                    1.50 dB
PL1W            18.13845062 W
SFO1            300.1519510 MHz

F2 - Processing parameters
SI                    65536
SF              300.1500000 MHz
WDW                      EM
SSB       0
LB                     0.20 Hz
GB        0
PC                     0.75
```

Figure S7. ^{1}H NMR spectra of **3** in $CDCl_3$.



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Current Data Parameters
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EXPNO                  10
PROCNO                  1

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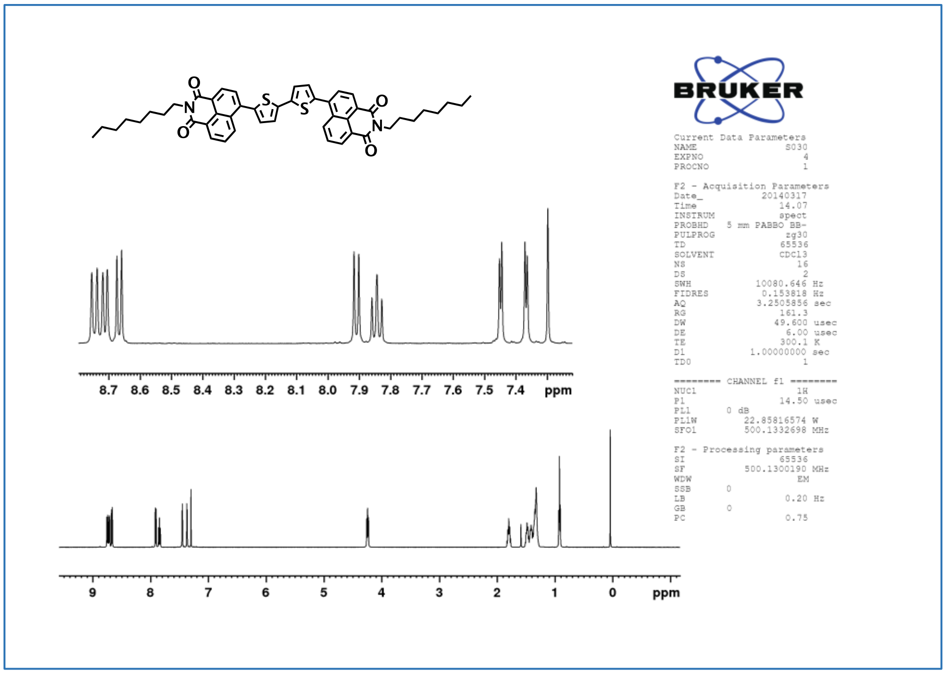
TD                  39918
SOLVENT             CDCl3
NS                   5000
DS                      4
SWH             19960.080 Hz
FIDRES           0.500027 Hz
AQ              0.9999459 sec
RG                  36800
DW                 25.050 usec
DE                  35.79 usec
TE                  300.0 K
D1             0.20000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                 -0.60 dB
PL1W          29.41712189 W
SFO1          75.4810800 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  1.30 dB
PL12                18.22 dB
PL13                19.76 dB
PL2W          18.99328995 W
PL12W          0.38601151 W
PL13W          0.27076975 W
SFO2         300.1512006 MHz

F2 - Processing parameters
SI                  65536
SF             75.4727780 MHz
WDW                    EM
SSB       0
LB                   3.00 Hz
GB        0
PC                   1.00
```

Figure S8. ^{13}C NMR spectra of **3** in CDCl$_3$.



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EXPNO                   4
PROCNO                  1

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TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10080.646 Hz
FIDRES           0.153818 Hz
AQ              3.2505856 sec
RG                  161.3
DW                 49.600 usec
DE                   6.00 usec
TE                  300.1 K
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TD0                     1

======== CHANNEL f1 ========
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P1                  14.50 usec
PL1       0 dB
PL1W          22.85816574 W
SFO1         500.1332698 MHz

F2 - Processing parameters
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SF            500.1300190 MHz
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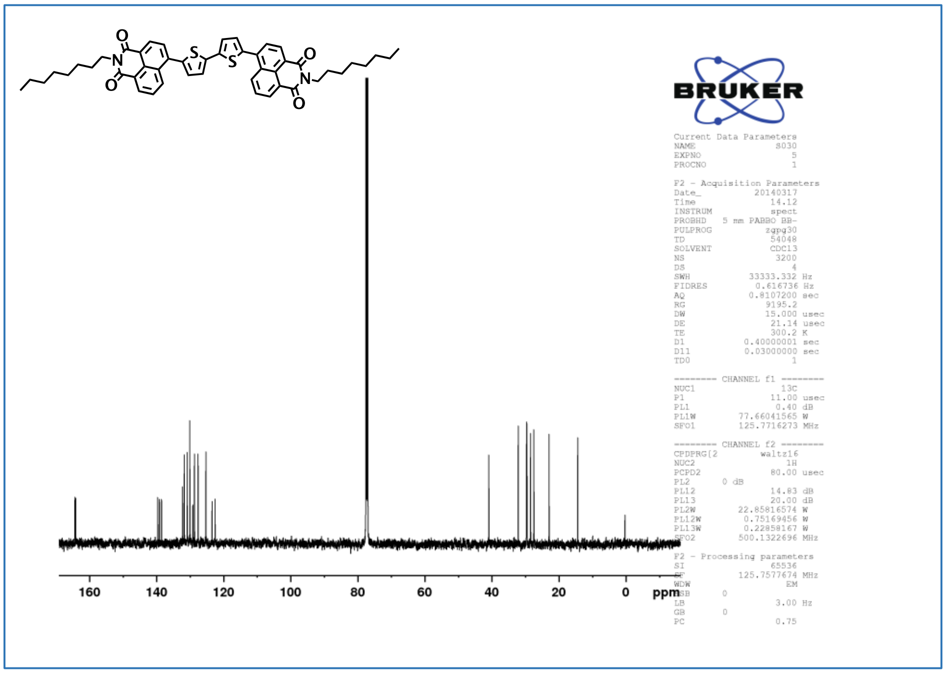
PC                   0.75
```

Figure S9. ^{1}H NMR spectra of **6** in CDCl$_3$.



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Current Data Parameters
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EXPNO                   5
PROCNO                  1

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TD                  54048
SOLVENT             CDCl3
NS                   3200
DS                      4
SWH             33333.332 Hz
FIDRES           0.616736 Hz
AQ              0.8107200 sec
RG                 9195.2
DW                 15.000 usec
DE                  21.14 usec
TE                  300.2 K
D1             0.40000001 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.00 usec
PL1                  0.40 dB
PL1W          77.66041565 W
SFO1          125.7716273 MHz

======== CHANNEL f2 ========
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NUC2                   1H
PCPD2               80.00 usec
PL2       0 dB
PL12                14.83 dB
PL13                20.00 dB
PL2W          22.85816574 W
PL12W          0.75169456 W
PL13W          0.22858167 W
SFO2          500.1322696 MHz

F2 - Processing parameters
SI                  65536
SF            125.7577674 MHz
WDW                    EM
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GB        0
PC                   0.75
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Figure S10. ^{13}C NMR spectra of **6** in CDCl$_3$.

UV-vis absorption thin film annealing

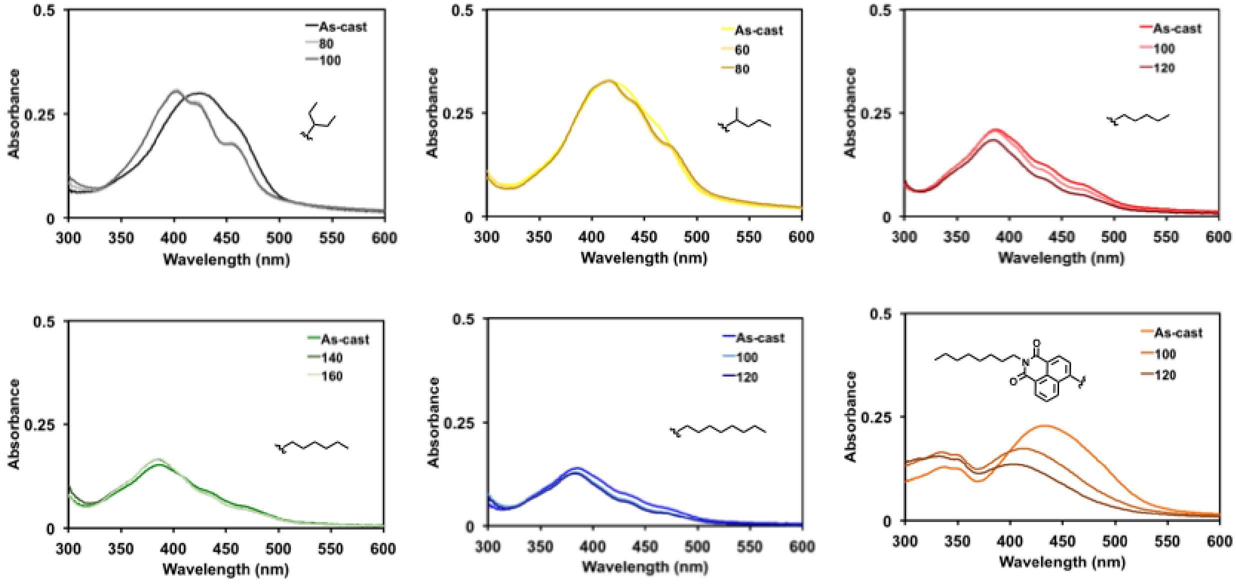


Figure S11. Thermal annealing of spun cast films of compounds **1-6**.

Optical Microscopy

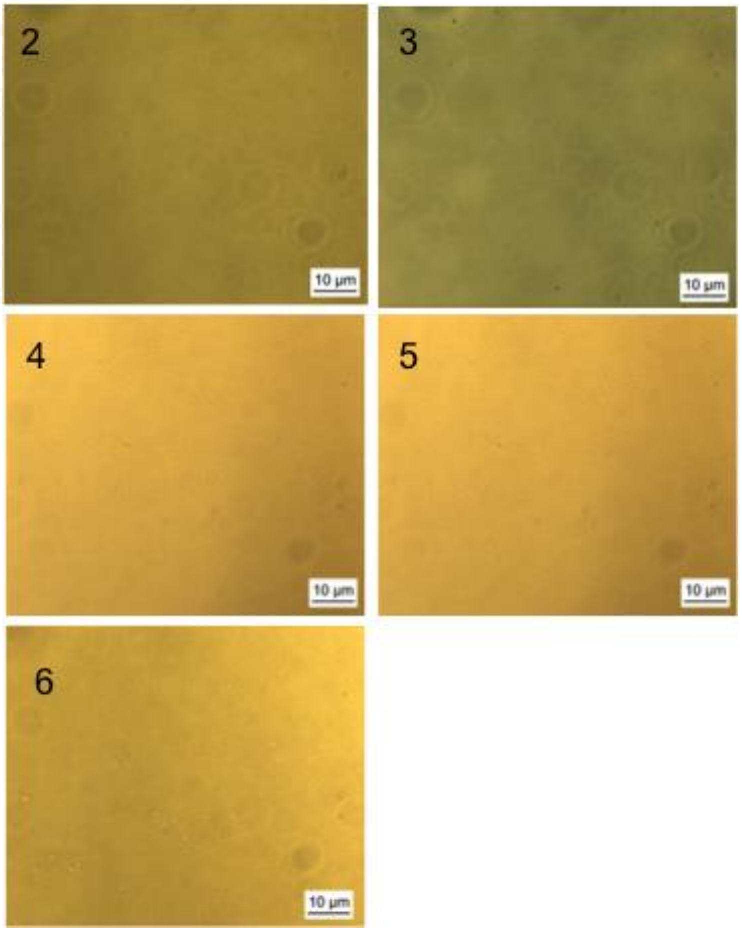


Figure S12. An Axio Imager A2 optical microscope was used to visualize compounds **2-6** as-cast up to 100x magnification, no changes in the thin films were observable upon thermal annealing up to 200 ºC.

UV-vis photoluminescence

Solution:

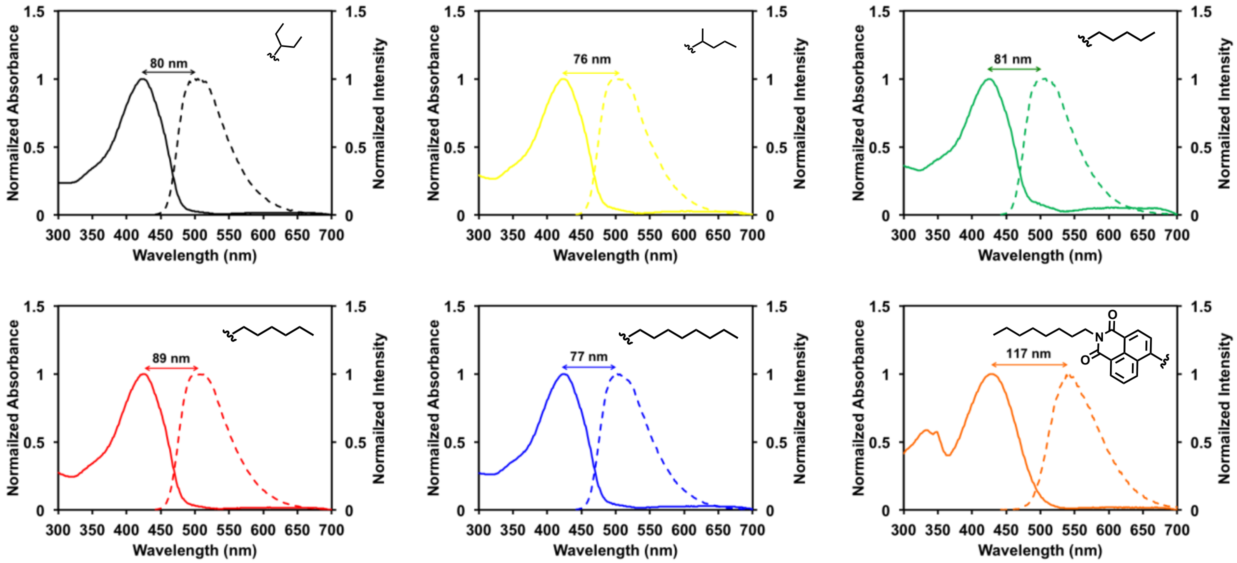


Figure S13. Absorption (dark solid lines) and fluorescence profiles (light solid lines) in solution ($CHCl_3$) for compounds **1-6**.

Thin Film:

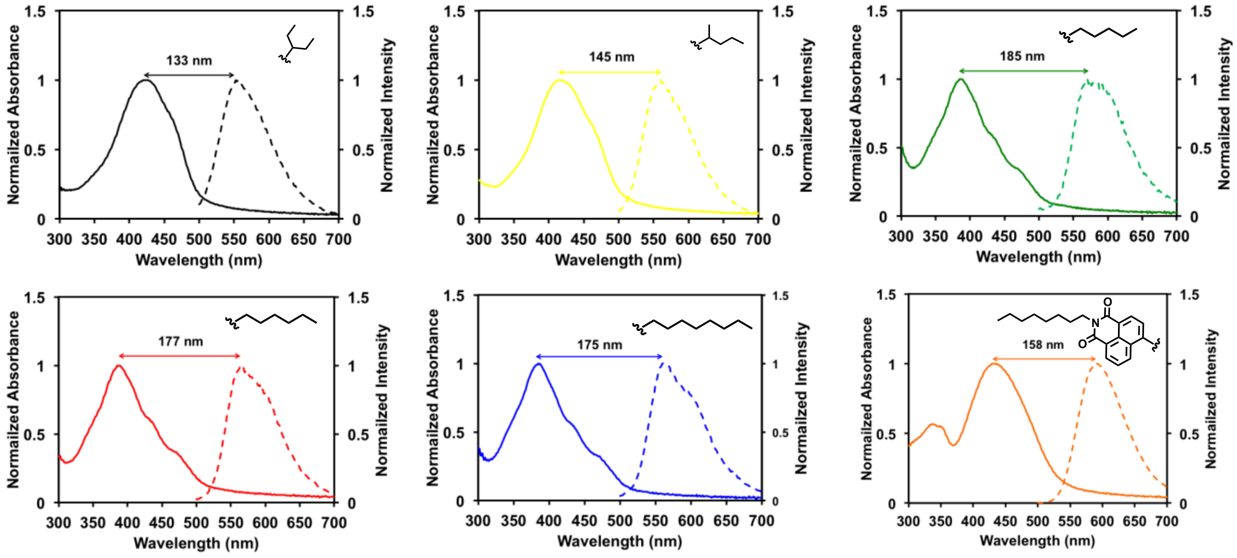
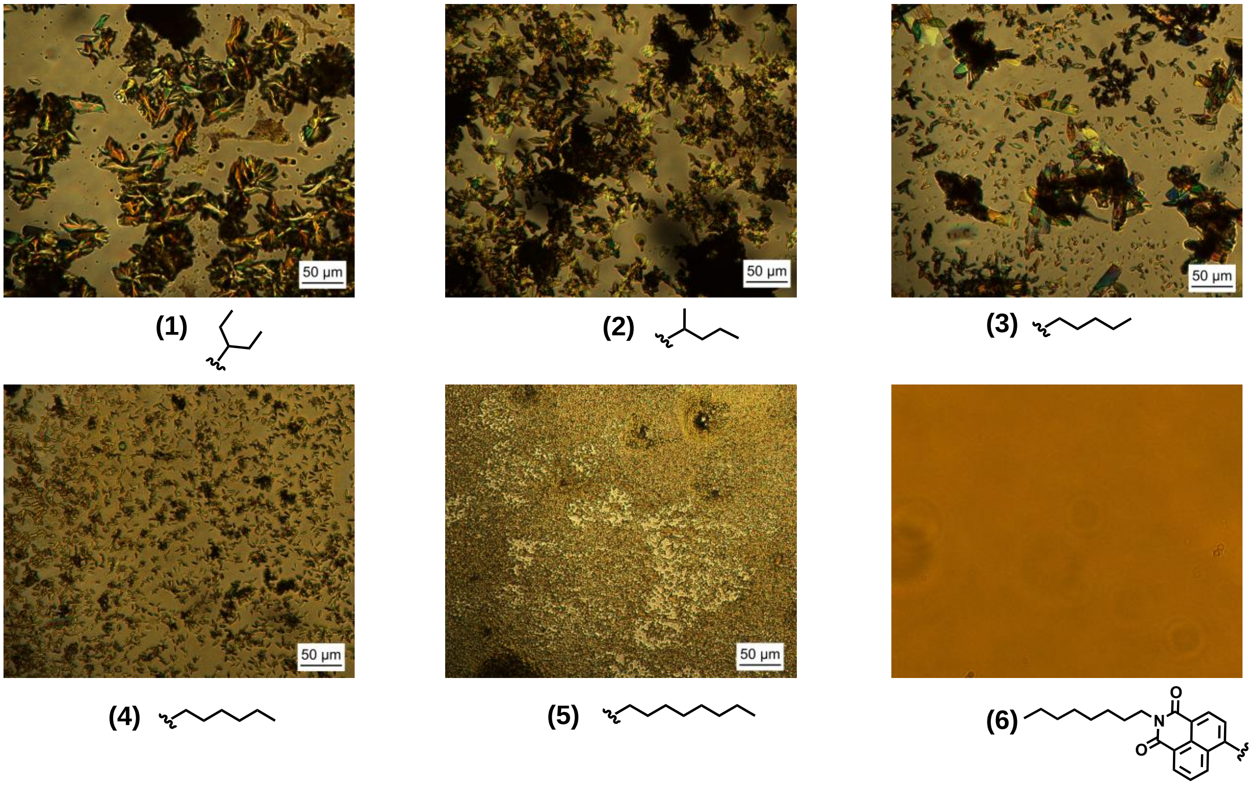


Figure S14. Absorption (dark solid lines) and fluorescence profiles (light solid lines) of as-cast films spun from $CHCl_3$ for compounds **1-6**.

Drop-cast thin films



(1) (2) (3) (4) (5) (6)

Figure S15. Optical microscopy images of drop-cast films of **1-6** from $CHCl_3$ solutions on top glass.

Differential Scanning Calorimetry

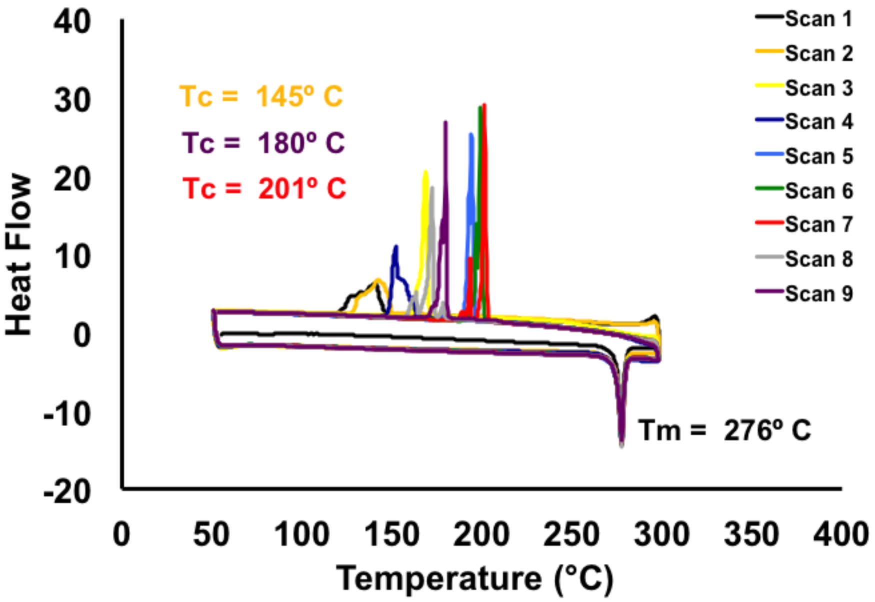


Figure S16. DSC thermogram of compound **1** with nine scans at a heating and cooling rate of 10 ºC per minute.

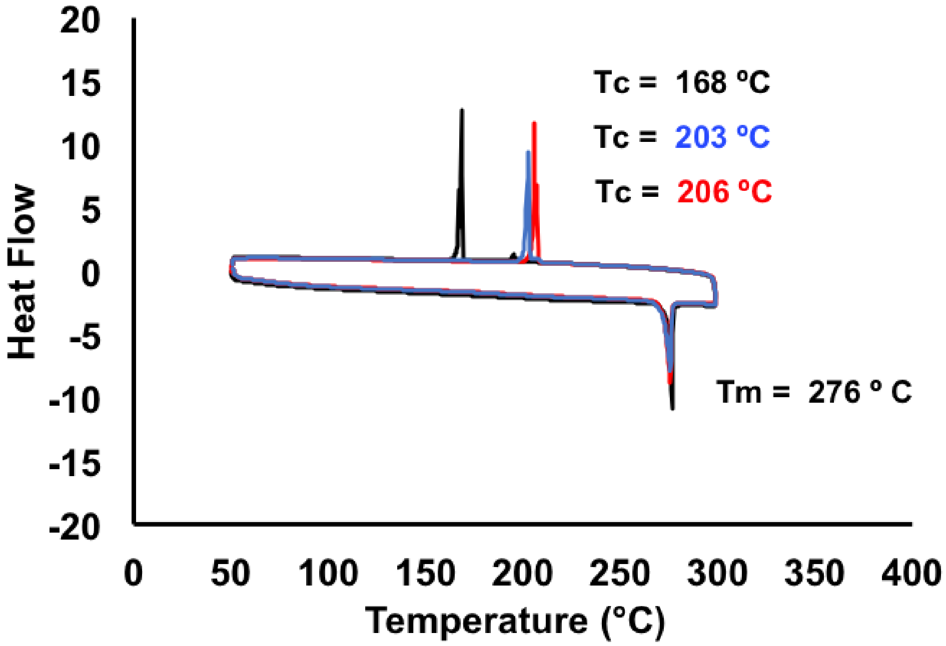


Figure S17. DSC thermogram of compound **1** with three scans at a heating and cooling rate of 5 ºC per minute.

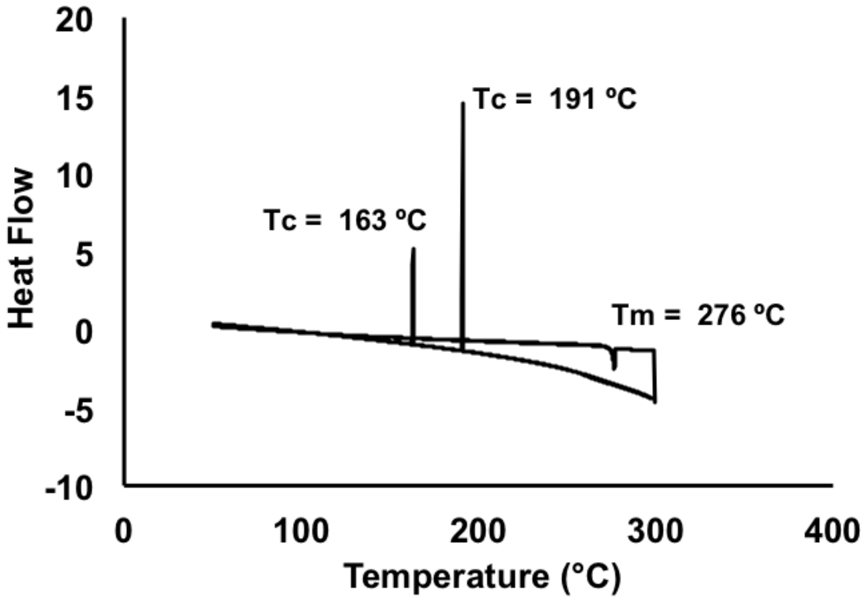


Figure S18. DSC thermogram of compound **1** with 3 scans at a heating and cooling rate of 1 ºC per minute.

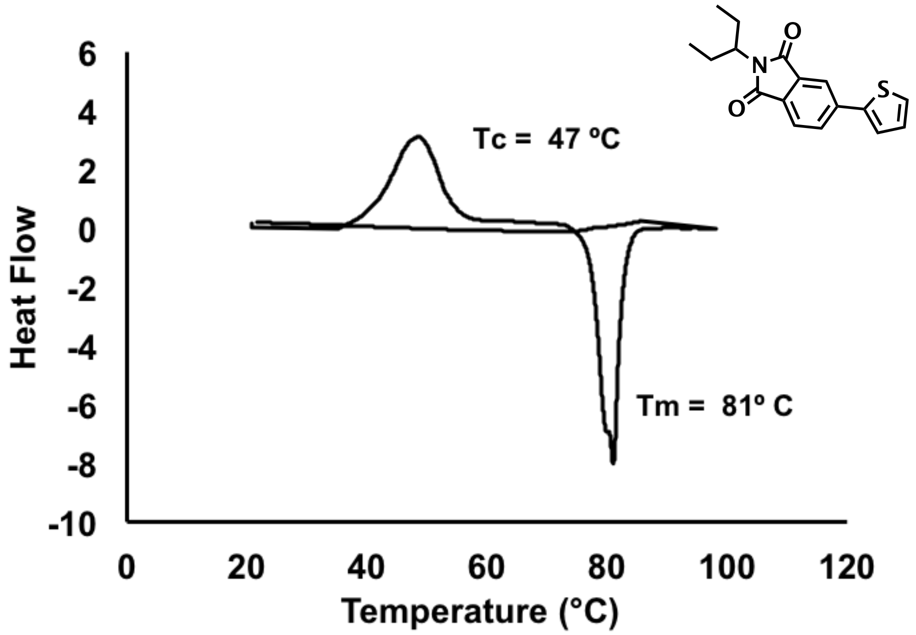


Figure S19. DSC thermogram of the half molecule of **1.**

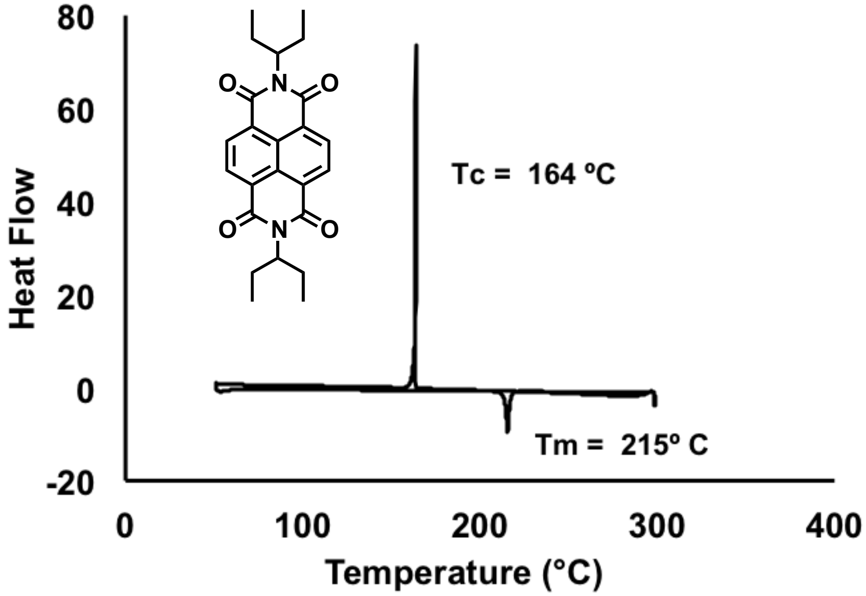


Figure S20. DSC thermogram of 1-ethylpropyl NDI**.**

Ellipsometry:

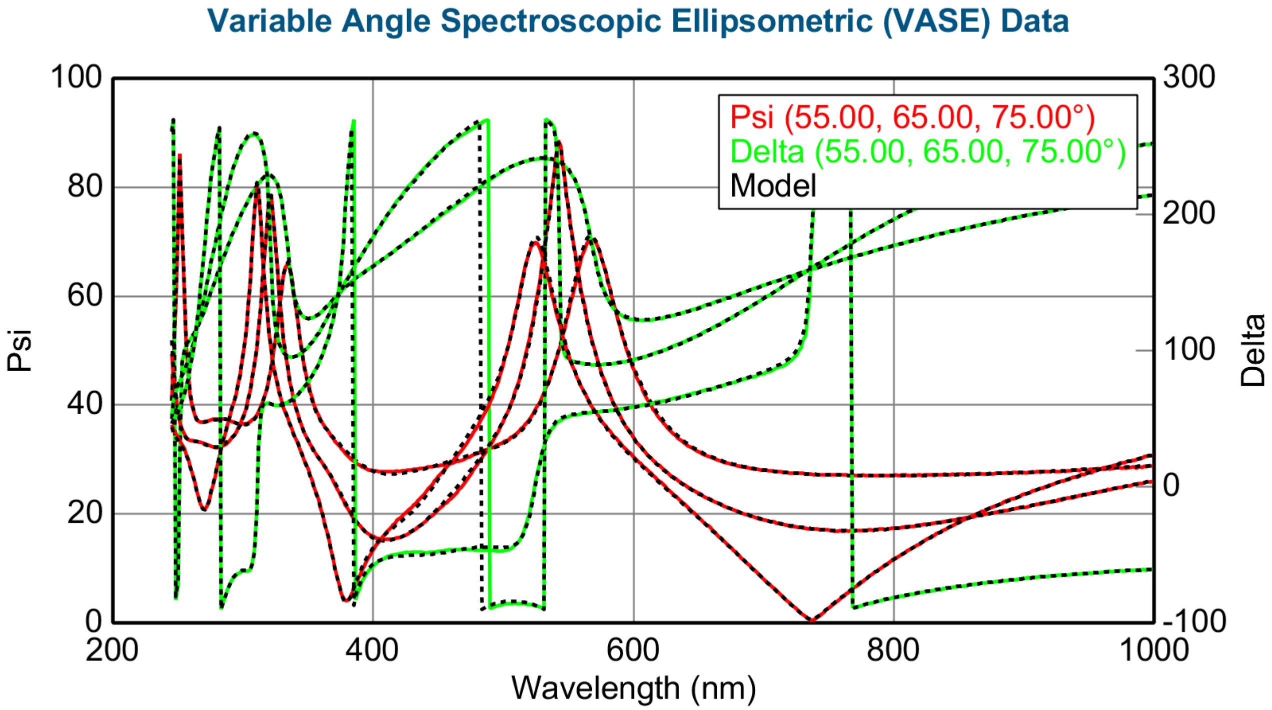


Figure S21. Representative fit of uniaxial anisotropic model (Si/SiO_2/Uniaxial layer) to ellipsometry data (data shown for compound **1**, as-cast film). MSE = 8.82.

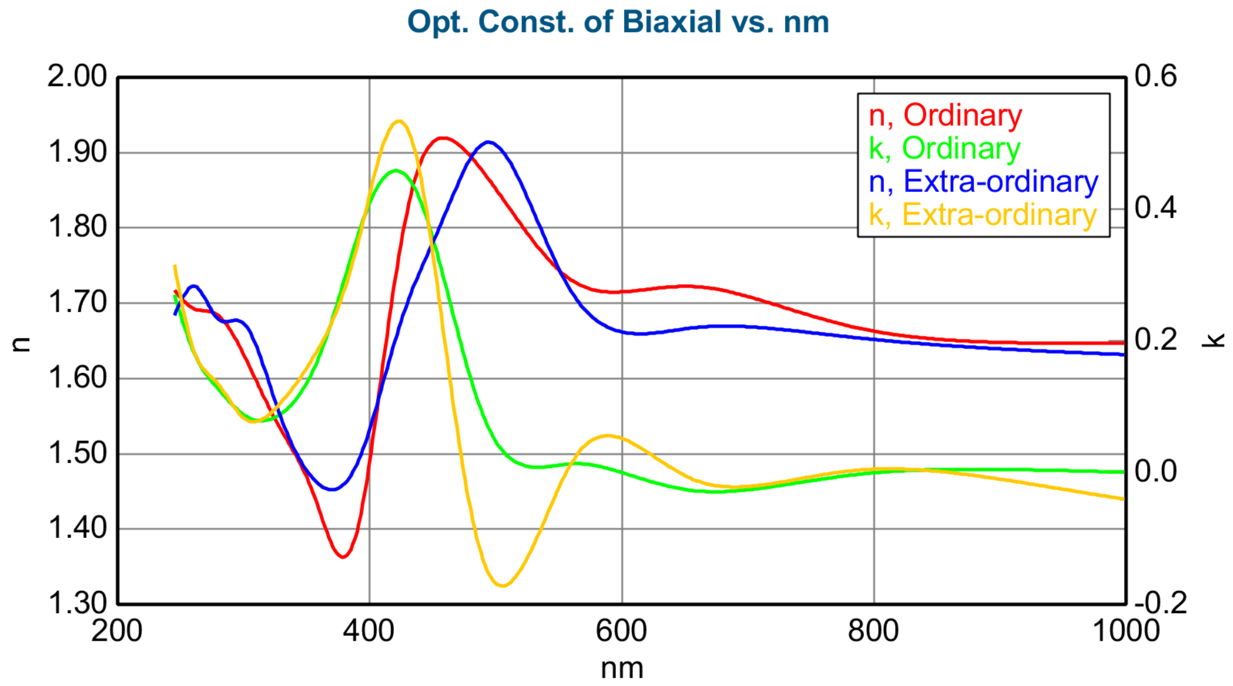


Figure S22. Representative example of the extracted optical parameters for compound **1** data (data shown for compound **1**, as-cast film). from the VASE experiments.

Organic Electronic Devices

Bottom-gate, bottom-contact configuration (Bin Sun, Li Research Group, University of Waterloo)

Compounds **1** to **6** were evaluated in OTFTs with bottom gate bottom contact configuration. Heavily doped n-type Si wafer was used as substrate with 300nm of SiO_2 as dielectric (Ci=11 nF cm^{-2}). Au source and drain pairs (channel length is 30um, and channel width is 1000um) were patterned on SiO_2/Si substrate by conventional lithography methods. The patterned substrate was cleaned by air plasma, acetone, and isopropanol sequent, followed by DDTS modification. Then the material film was formed by spin-coating a solution (20 mg mL^{-1}) in chloroform at 5000RPM for 60s, resulting a BGBC OTFT device. After annealing at 50 to 100ºC, the device was tested in dark by Agilent B2912 Source measure unit. All the fabrication process was operated in N2 fill glove box. Device performance was unreliable and is not reported.

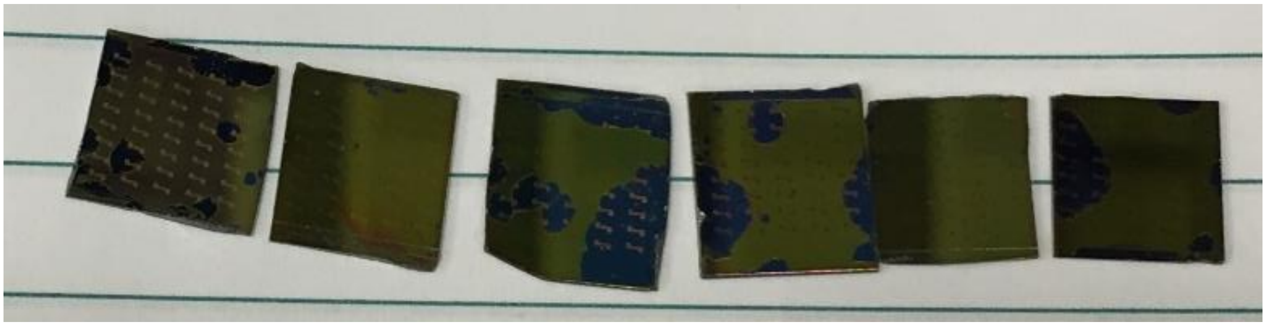


Figure S23. OTFT devices with compounds **1** to **6** from left to right; the film did not fully cover the bare substrate due to the low surface energy of DDTS modified substrate. A concentration of 20 mg mL^{-1} was required to even form partial films. Lower concentrations resulted in very poor substrate wetting.

Bottom-contact, top-gate (BCTG) (Ossila Ltd.)

Initially bottom-contact, top-gate (BCTG) devices were attempted, utilizing pre-patterned interdigitated ITO substrates on soda lime glass (Ossila Limited, S161) as the bottom contact and substrate, PMMA as the gate dielectric and PEDOT:PSS as the top gate electrode. The thin films of compounds 1 – 6 were annealed at either 80 °C or 120 °C. However the thin films of compounds 1 – 6 were all dissolved by PMMA solution that was spin cast on top.

Bottom-gate, top-contact (BGTC) (Ossila Ltd.)

Bottom-gate, top-contact (BGTC) transistors were fabricated from polished <100> p-doped silicon wafers (Ossila S146, 0.001 Ω cm resistivity) that acted as the gate electrode, and with a 300 nm thermally grown silicon oxide on the surface (1.09 nF cm^{-2} capacitance) that acted as the gate dielectric. Substrates were cleaned by sonication successively in hot 1 v/v % Hellmanex III solution in water and warm isopropyl alcohol, rinsing twice with deionised water between sonication steps. Cleaning culminated with 5 min UV Ozone treatment (Ossila UV Ozone cleaner). There were no surface treatments of the silicon oxide substrates. This was in order to improve surface wetting and to enable comparison of the FET characteristics to similarly produced thin films of material analysed elsewhere in the paper.

Solutions of compounds 1-6 in chloroform (10 mg/ml) were filtered through 0.45 µm PTFE syringe filters and kept on a hotplate at 50 °C until use to prevent reaggregration. The thin films were deposited by dynamically spin casting 50 µl of the warm solution at 5000 rpm for 30 s. The active layer was wiped clean from the substrate surface, where electrode contact pads would be evaporated, using cleanroom swabs soaked in chloroform. The area between devices was wiped with high-precision cleanroom swabs prior to measurement to avoid potential electrical crosstalk. The finished devices were either annealed for 5 minutes at 120 °C or not annealed at all.

Devices were completed by thermal evaporation of 60 nm of aluminium (at 3 x 10^{-6} mbar and 0.2 Å/s) as the top source-drain electrodes through a shadow mask (Ossila low-density FET mask E291 and evaporation stack E281). The transistor channels had a width of $W = 1$ mm and a length of $L = 30$ um, with 5 transistors per substrate. The area between devices was wiped with high-precision cleanroom swabs prior to measurement to avoid potential electrical crosstalk.

FET measurements (Ossila Ltd.)

All FET measurements were carried out at room temperature. The electrical characterisation of the transistors was performed using the Ossila FACT1 OFET Mobility Testing System (Electronic multiplexor, Keithley 2612B system dual source-measure unit and control software). The average calculated values and the standard deviation for each OFET performance parameter (mobility, μ, threshold voltage, V_T and ON/OFF ratio, $I_{ON/OFF}$) were calculated. The threshold voltage was extracted from the linear extrapolation of the square root of source-drain current, $(I_{SD})^{1/2}$ versus gate voltage, V_G curve. The linear mobility was used opposed to the saturation mobility due to lack of observed saturation in the output characteristics, and was calculated from the relationship shown in Equation 1.

$$\mu_{\text{lin}} = \frac{\delta I_{\text{DS}}}{\delta V_{\text{G}}} \cdot \frac{L}{W C_{\text{i}} V_{\text{DS}}} \tag{1}$$

where L is the transistor length, W is the transistor width, and C_i is the dielectric capacitance per unit area (1.09 × 10^{-8} for 300 nm of silicon dioxide). V_{DS} was set at + 5 V. The mobility values reported are an average of the highest points from the mobility versus gate voltage curve calculated for each transistor. The $I_{ON/OFF}$ was determined from the ratio of I_{ON} at $V_G = 0$ V to I_{OFF} at $V_G = +100$ V for the transfer curves in the linear region ($V_{DS} = +5$ V).

Device List

Substrate	Material	Solvent	Conc. / mg ml^{-1}	Spin speed / rpm	Annealing Temp / °C	Result
S146	1 – EP	Chloroform	10	5000	None	No FET behaviour
S146	2 – MB	Chloroform	10	5000	None	No FET behaviour
S146	3 - Pentyl	Chloroform	10	5000	None	No FET behaviour
S146	4 - Hexyl	Chloroform	10	5000	None	No FET behaviour
S146	5 - Octyl	Chloroform	10	5000	None	No FET behaviour
S146	6 - Nap	Chloroform	10	5000	None	No FET behaviour
S146	1 – EP	Chloroform	10	5000	120 °C	No FET behaviour

S146	2 – MB	Chloroform	10	5000	120 °C	No FET behaviour
S146	3 - Pentyl	Chloroform	10	5000	120 °C	No FET behaviour
S146	4 - Hexyl	Chloroform	10	5000	120 °C	No FET behaviour
S146	5 - Octyl	Chloroform	10	5000	120 °C	No FET behaviour
S146	6 - Nap	Chloroform	10	5000	120 °C	FET behaviour

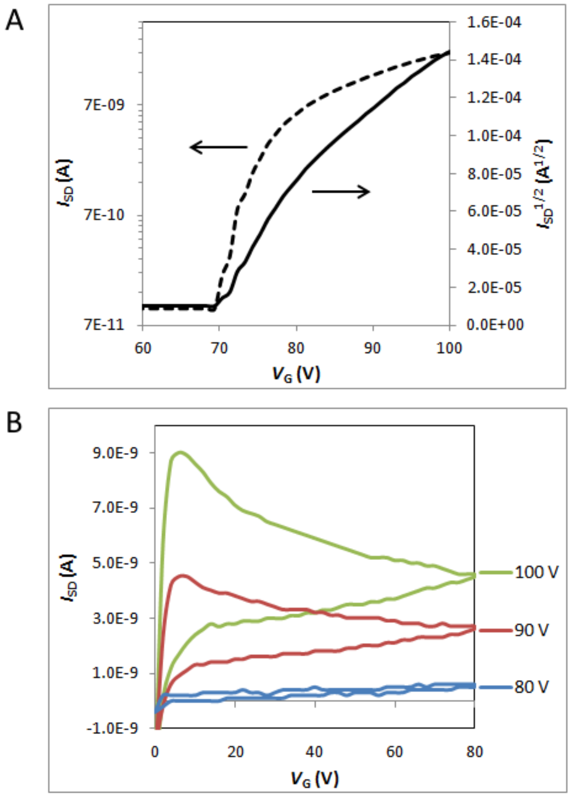


Figure S24. A) Transfer characteristics of compound 6 thin film annealed at 120 °C. B) Output characteristics of compound 6 thin film annealed at 120 °C.

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