Supporting Information

Structure-Property Relationship of Two Structural Isomers of Thiophene-
Flanked Diazaisoindigo on Carrier Transport Properties.

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I. General

All the chemicals and solvents were of reagent grade purchased from Tokyo Kasei Chemical Industries,
Wako Pure Chemical Industries, and Aldrich and used without further purification. All reactions were
carried out under an argon atmosphere. The 1H and 13C NMR spectra were recorded using a JEOL JNM-
AL300 spectrometer (300 MHz) in deuterated chloroform at 298 K and tetramethylsilane was used as the
internal standard. Mass spectrograms were recorded by a Shimadzu QP-5000 (EI-MS) or Shimadzu MALDI-AXIMA time of flight mass spectrometer (MALDI-TOF) operated in the linear mode. The UV-Vis-NIR absorption spectra of the dilute solution and spin-coated thin film of the present compounds were collected using a Jasco Corporation V-630 UV-VIS-NIR spectrophotometer. The optical energy gaps $E_g^{\text{opt}}$ were estimated from the low-energy band edge of the absorption spectra of the thin films. Elemental analyses were performed at the Center for Advanced Materials Analysis, Tokyo Institute of Technology.

II DFT calculations

Density Functional Theory (DFT) calculations were performed using the Gaussian 09 program\textsuperscript{1} based on the $\omega$B97X-D/6-31G(d,p) for calculation of the optimized molecular geometry and ground-state electronic structures and $\omega$B97X-D/6-311++G(d,p) for TD-DFT calculation.
Fig. S1. Optimized molecular geometry depending on nitrogen positions on the diazaisoindigo core at (a) 4,4’-positions, (b) 5,5’-positions, (c) 6,6’-positions, and (d) 7,7’-positions.

III. Material Synthesis

6-Bromo-7-azaindole 1 was prepared according to the previous report.\textsuperscript{2}

1-Hexyl-6-bromo-7-azaindole (2)
To a solution of 6-Bromo-7-azaindole (0.5 g, 2.54 mmol) dissolved in dry DMF (10 ml), NaH (55 wt% dispersion in oil, 0.16 g, 3.71 mmol) was added in one portion, and the solution was stirred at r.t. for 30 min. Hexylbromide (0.4 ml, 2.77 mmol) was added to the solution, and the mixture was stirred at r.t. for 12 h. The reaction mixture was poured into water, and the mixture was extracted with CH$_2$Cl$_2$ and was washed with brine. After drying over MgSO$_4$, the solvent was removed and the residue was purified by silica-gel chromatography eluting with CH$_2$Cl$_2$-hexane (2/1 = v/v) to provide 2 as a clear oil (1.15 g, 63%).

$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC): $\delta$ 7.73 (d, $J$ = 8.4 Hz, 1H), 7.18 (dd, $J$ = 5.1, 3.6 Hz, 2H), $\delta$ 6.43 (d, $J$ = 3.6 Hz, 1H), 4.24 (t, $J$ = 7.5 Hz, 2H) 1.90-1.80 (m, 4H), 1.45-1.25 (m, 6H), 0.87 (t, $J$ = 6.6 Hz, 3H).  $^{13}$C NMR (300 MHz, CDCl$_3$, 25 ºC): $\delta$ 146.99, 134.49, 130.70, 127.95, 119.13, 99.63, 44.57, 31.35, 30.22, 26.41, 22.51, 13.97, one aromatic peak is overlapped. MS (EI) m/z: 282 [M]$^+$.

1-Hexyl-3,3,6-tribromo-7-azaoxiindole (3)

To a solution of 2 (0.45 g, 1.60 mmol) dissolved in tert-butanol (20 ml), pyridinium bromide perbromide (2.90 g, 7.40 mmol) was added in small potions at r.t., and the mixture was stirred at r.t. for 12 h. The solvent removed under reduced pressure, and the residue was extracted with EtOAc and washed with brine. After drying over MgSO$_4$, the solvent was removed and the residue was purified by silica-gel chromatography eluting with CH$_2$Cl$_2$-hexane (2/1 = v/v) to provide 3 as a red oil (0.45 g, 62%).

$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC): $\delta$ 7.66 (d, $J$ = 7.5 Hz, 1H), 7.28 (d, $J$ = 7.8 Hz 1H), 3.83 (t, $J$ = 7.5 Hz, 2H), 1.85-1.73 (m, 2H), 1.45-1.22 (m, 6H), 0.88 (t, $J$ = 7.8 Hz, 3H).  $^{13}$C NMR (300 MHz, CDCl$_3$, 25 ºC): $\delta$ 169.06, 152.53, 142.80, 134.83, 124.56, 123.09, 40.30, 31.21, 27.08, 26.11, 22.46, 13.96, one
aromatic peak is overlapped. MS(EI) m/z: 454 [M]+.

1-Hexyl-6-bromo-7-azoxiindole (7a)

To a solution of 2 (1.20 g, 2.64 mmol) dissolved in acetic acid (20 ml), zinc dust (1.73 g, 26.4 mmol) was added, and the mixture was stirred at r.t. for 12 h. The solvent was removed under reduced pressure, and the residue was extracted with EtOAc and washed with brine. After drying over MgSO₄, the solvent was removed to provide 7a as a pale white solid (0.74 g, 95%), which was used without further purification.

1H NMR (300 MHz, CDCl₃, 25 °C): δ 7.32 (d, J = 7.8 Hz, 1H), 7.11 (d, J = 7.8 Hz 1H), 3.78 (t, J = 7.5 Hz, 2H), 3.48 (s, 2H), 1.75-1.63 (m, 2H), 1.38-1.26 (m, 6H), 0.88 (t, J = 7.8 Hz, 3H).

13C NMR (300 MHz, CDCl₃, 25 °C): δ 174.00, 158.42, 137.50, 133.32, 121.04, 111.66, 39.49, 34.46, 31.37, 27.51, 26.47, 22.54, 14.02. MS(EI) m/z: 296 [M]+

1-Hexyl-6-bromo-7-azaisatin (8a)

To a solution of 7a (0.55g, 1.85 mmol) dissolved in dry DMSO (7 ml), N-bromosuccinimide (0.34 g, 1.92 mmol) was added at r.t., and the mixture was stirred under 60 °C for 12 h. The reaction mixture was poured into water, and the mixture was extracted with ethylacetate and was washed with brine. After drying over MgSO₄, the solvent was removed and the residue was purified by silica-gel chromatography eluting with CH₂Cl₂-hexane (2/1 = v/v) to provide 8a as an oily orange solid (0.46 g, 80%).

1H NMR (300 MHz, CDCl₃, 25 °C): δ 7.90 (d, J = 7.5 Hz, 1H), 7.27 (d, J = 7.5 Hz 1H), 3.83 (t, J = 7.5 Hz, 2H), 1.76-1.67 (m, 2H), 1.40-1.26 (m, 6H), 0.89 (t, J = 7.2 Hz, 3H).

13C NMR (300 MHz, CDCl₃, 25 °C): δ 164.17, 158.10, 150.15, 133.97, 123.27, 110.25, 39.56, 31.25, 27.38, 26.41, 22.50, 13.98, 14.02.
aromatic peak is overlapped. MS(EI) m/z: 311 [M]+.

1,1’-Dihexyl-6,6’-dibromo-7,7’-diazaisoindigo (9a)

To a solution of 7a (0.52 g, 1.75 mmol) and 8a (0.50 g, 0.61 mmol) dissolved in acetic acid (15 ml), catalytic amount of conc. HCl 0.1 ml was added at r.t., and the mixture was stirred at 110 °C for 12 h. The resulting precipitate was filtered and washed with MeOH, H2O, and hexane. After drying in vacuum, 9a was obtained as a red solid (0.97 g, 94%).

1H NMR (300 MHz, CDCl3, 25 °C): δ 9.30 (d, J = 8.1 Hz, 2H), 7.22 (d, J = 8.1 Hz 2H), 3.87 (t, J = 7.2 Hz, 4H), 1.79-1.70 (m, 4H), 1.48-1.37 (m, 12H), 0.89 (t, J = 6.9 Hz, 6H). 13C NMR (300 MHz, CDCl3, 25 °C): δ 167.90, 157.81, 143.78, 139.09, 121.90, 114.50, 39.60, 31.36, 27.56, 26.52, 22.54, 14.02, one aromatic peak is overlapped. MS(MALDI-TOF) m/z: 590.81 [M]+. Anal. Calcd for C26H30Br2N4O2: C, 52.90; H, 5.12; N, 9.49; found: C, 52.92; H, 4.95; N, 9.45.

1,1’-dihexyl-6,6’-Dithienyl-7,7’-diazaisoindigo (6-6’-T-DAII)

The mixture of 9a (0.12 g, 0.2 mmol), tributhylstanylthiophene (0.2 ml, 6.1 mmol), and Pd(dppf)Cl2 (0.02 g, 0.028 mmol) dissolved in dry toluene (8 ml) was stirred to reflux for 24 h. The reaction mixture was poured into water, and the mixture was extracted with CH2Cl2 and was washed with brine. After drying over Na2SO4, the solvent was removed and the residue was purified by silica-gel chromatography eluting with CH2Cl2-hexane (2/1 = v/v) to provide 1 as a dark purple solid (0.09 g, 76%).

1H NMR (300 MHz, CDCl3, 25 °C): δ 9.44 (d, J = 8.4 Hz 2H), 7.71 (dd, J = 2.0, 1.5 Hz , 2H), 7.46 (dd, J = 2.5, 1.1 Hz, 2H), 7.36 (d, J = 8.4 Hz 2H), 7.15 (dd, J = 3.6, 2.4 Hz 2H), 3.97 (t, J = 7.7 Hz, 4H), 1.90-1.81
(m, 4H), 1.44-1.31 (m, 12H), 0.89 (t, \(J = 7.0\) Hz, 6H). The \(^{13}\)C NMR failed to obtain a spectrum because of the limited solubility of the product in chloroform. MS (MALDI-TOF) \(m/z\): 597.46 [M+H]+. Anal. Caled for C\(_{34}\)H\(_{36}\)N\(_4\)O\(_2\)S\(_2\): C, 68.43; H, 6.08; N, 9.39; found: C, 68.17; H, 5.99; N, 9.28.

1-Hexyl-7-azaindole (5)

The compound 5 was prepared according to the same procedure for 1 using 7-azaindole (1.0 g, 8.46 mmol), NaH (55wt% dispersion in oil, 0.55 g, 12.4 mmol), and hexylbromide (1.4 ml, 9.23 mmol) to provide 5 as a clear oil (1.74 g, quant.).

\(^1\)H NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 8.33-8.31 (m, 1H), 7.90-7.88 (m, 1H), 7.21 (d, \(J = 3.3\) Hz, 1H), 7.06-7.02 (m, 1H), 6.44 (d, \(J = 3.3\) Hz, 1H), 4.28 (t, \(J = 7.2\) Hz, 2H), 1.92-1.82 (m, 2H), 1.45-1.22 (m, 6H), 0.86 (t, \(J = 6.9\) Hz, 3H). \(^{13}\)C NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 142.63, 128.61, 127.89, 120.52, 115.43, 99.13, 44.58, 31.42 30.38, 26.54, 26.52, 22.51, 13.97, one aromatic peak is overlapped. MS(EI) \(m/z\): 202 [M]+.

1-Hexyl-3,3,5-tribromo-7-azaoxiindole (6)

To a solution of 6 (1.60 g, 7.91 mmol) dissolved in tert-butanol (20 ml) and H\(_2\)O (20 ml), bromine (16 g, 100 mmol) was added dropwise, and the mixture was stirred at r.t. for 12 h. The solvent was removed under reduced pressure, the residue was purified by silica-gel chromatography eluting with CH\(_2\)Cl\(_2\)-hexane (2/1 = v/v) to provide 6 as a red oil (3.24 g, 70%).

\(^1\)H NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 8.31 (d, \(J = 8.1\) Hz, 1H), 7.94 (d, \(J = 8.1\) Hz, 1H), 3.82 (t, \(J = 7.2\) Hz, 2H), 1.81-1.69 (m, 2H), 1.42-1.27 (m, 6H), 0.88 (t, \(J = 6.6\) Hz, 3H). \(^{13}\)C NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 168.69, 151.14, 150.87, 135.82, 127.18, 114.64, 40.30, 31.27 27.11, 26.18, 22.64, 14.11, one aromatic
peak is overlapped. MS (MALDI-TOF) m/z: 451.16 [M]^+

1-Hexyl-5-bromo-7-azoiindole (7b)

The compound 7b was prepared according to the same procedure for 7a using 6 (3.10 g, 6.81 mmol) and zinc dust (4.45 g, 68.1 mmol) to provide 7b as a pale pink oil (2.02 g, quant.). \(^1\)H NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 8.25 (s, 1H), 7.59 (s, 1H), 3.81 (t, \(J = 7.5\) Hz, 2H), 3.55 (s, 2H), 1.71-1.63 (m, 2H), 1.31-1.23 (m, 6H), 0.87 (t, \(J = 7.2\) Hz, 3H). \(^{13}\)C NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 181.16, 174.60, 156.31, 144.80, 135.06, 124.20, 114.96, 39.51, 31.31, 27.40, 26.47, 22.48, 13.98. MS(EI) m/z: 296 [M]^+.

1-Hexyl-5-bromo-7-azaisatin (8b)

The compound 8b was prepared according to the same procedure for 8a using 7b (1.3 g, 4.37 mmol) and N-bromosuccinimide (0.81 g, 4.53 mmol) to provide 8b as a dark red oily solid (0.28 g, 21%). \(^1\)H NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 8.52 (s, 1H), 7.91 (s, 1H), 3.83 (t, \(J = 7.2\) Hz, 2H), 1.75-1.68 (m, 2H), 1.43-1.31 (m, 6H), 0.88 (t, \(J = 6.6\) Hz, 3H). \(^{13}\)C NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 177.03, 173.70, 156.80, 147.40, 134.40, 120.92, 113.26, 39.52, 31.39, 27.54, 26.49, 22.50, 13.97. MS(EI) m/z: 310 [M]^+.

1,1'-Dihexyl-5,5'-dibromo-7,7'-diazaisoindigo (9b)

The compound 9b was prepared according to the same procedure for 9a using 7b (0.27 g, 0.9 mmol) and 8b (0.28 g, 0.9 mmol) to provide 9b as a red solid (0.31 g, 58%). \(^1\)H NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 9.66 (s, 1H), 8.33 (s, 1H), 3.88 (t, \(J = 6.9\) Hz, 2H), 1.78-1.69 (m, 2H), 1.40-1.29 (m, 6H), 0.89 (t, \(J = 7.2\) Hz, 3H). \(^{13}\)C NMR (300 MHz, CDCl\(_3\), 25 °C): \(\delta\) 166.96, 156.41, 151.14, 139.95, 132.21, 117.07, 113.91, 39.64, 31.46, 27.62, 26.60, 22.56, 14.03. MS (MALDI-TOF) m/z: 590.07 [M]^+. Anal. Calcd for
C$_{30}$H$_{36}$Br$_2$N$_4$O$_2$: C, 52.90; H, 5.12; N, 9.49; found: C, 52.88; H, 4.88; N, 9.43.

1,1'-Dihexyl-5,5'-dithienyl-7,7'-diazaisoindigo (5-5'-T-DAII)

The compound 5-5'-T-DAII was prepared according the same procedure for 6-6'-T-DAII using 9b (0.12 g, 0.20 mmol), tributhylstanylthiophene (0.2 ml, 6.1 mmol), Pd(PPh$_3$)$_4$ (0.02 g, 0.02 mmol) to provide I as a purple solid (0.09 g, 75%).

$^1$H NMR (300 MHz, CDCl$_3$, 25 °C): $\delta$ 9.82 (d, $J = 2.2$ Hz, 2H), 8.51 (d, $J = 1.8$ Hz, 2H), 7.39 (dd, $J = 1.8$, 1.5 Hz, 2H), 7.33 (dd, $J = 2.6$, 1.1 Hz, 2H), 7.137 (dd, $J = 3.7$, 2.6 Hz, 2H), 3.94 (t, $J = 7.3$ Hz, 4H), 1.85-1.72 (m, 4H), 1.46-1.32 (m, 12H), 0.89 (t, $J = 7.0$ Hz, 6H). The $^{13}$C NMR failed to obtain a spectrum because of the limited solubility of the product in chloroform. MS (MALDI-TOF) $m/z$: 597.49 [M+H]$^+$. Anal. Calcd for C$_{34}$H$_{36}$N$_4$O$_2$: C, 68.43; H, 6.08; N, 9.39; found: C, 68.49; H, 6.02; N, 9.01.

$^1$H NMR spectra for compound 9a
$^{13}$C NMR spectra for compound 9a
$^1$H NMR spectra for 6-6'-T-DAII
MS chart for 6-6'-T-DAII
$^1$H NMR spectra for compound 9b
13C NMR spectra for compound 9b.
MS chart for 5-5'-T-DAII
IV. Thermal properties

Shimadzu Biotech Axima Performance 2.8.4.20081127: Mode Linear, Power: 45, P.Ext. @ 596 (bin 46)

%Int. 27 mV Profile 200

%Int. 34 mV [sum= 6855 mV] Profiles 1-200 Unsmoothed

%Int. 204 mV Profiles 1-200: Threshold Gradient
Thermal gravimetric analyses (TGA) were performed using a RIGAKU Thermo plus EVO TG8120 thermal analyzer at the heating rate of 10 °C min⁻¹ under a N₂ atmosphere. Differential scanning calorimetric (DSC) measurements were performed with an SII EXSTAR DSC-6100 at the heating rate of 5 °C min⁻¹ under a N₂ atmosphere. For the DSC measurements, the second heating-cooling cycles were analyzed.

**Fig. S2.** TGA curves of 6,6'-T-DAII and 5,5'-T-DAII at the heating rate of 10 °C / min under N₂ atmosphere.
V. X-ray Single Crystal Structure Analysis

The crystal structures were determined from single-crystal X-ray diffraction data. The measurements were made by a RIGAKU R-AXIS RAPID II imaging plate with Cu-Kα radiation from a rotation anode source with a confocal multilayer X-ray mirror (RIGAKU VM-spider, \( \lambda = 1.54187 \ \text{Å} \)). The diffraction data were corrected under -100 °C. The structures were solved by the direct method (SHELX-97). The structures were refined by the full-matrix least-squares procedure by applying anisotropic temperature factors.

Fig. S3. DSC curves of 6,6'-T-DAII and 5,5'-T-DAII at the heating rate of 5 °C / min under N₂ atmosphere.
factors. The positions of the hydrogen atoms were calculated. The disordered sulfur and carbon atoms of 6,6'-T-DAII are analyzed population analysis.

Table S1 Crystallographic data

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\[ a R_t = \frac{\sum |F_o| - |F_i|}{\sum |F_o|}, \quad b R_w = \left[ \frac{\sum w(F_o^2 - |F_i|^2)}{\sum wF_o^2} \right]^{1/2} \]
VI. Electrochemical properties

Cyclic voltammetry (CV) was carried out using an ALS/CH Instruments Electrochemical Analyzer Model 700C. The measurement was performed in dehydrated dichloromethane solution containing 0.1 M $n$-Bu$_4$NPF$_6$ as the supporting electrolyte using a glassy carbon working electrode, a platinum counter electrode, and an Ag/AgNO$_3$ electrode as a reference electrode at the scan rate of 100 mV s$^{-1}$. The films of the present compounds were formed on a glassy carbon working electrode. From the onset reduction potentials and $E_{\text{red, onset}}$, LUMO levels were estimated by assuming the ferrocene/ferrocenium energy level to be -4.8 eV under the vacuum level. The HOMO levels are estimated from the LUMO value and the optical bandgap.
Fig. S4. Cyclic voltamograms of (a) 6,6'-DAII and (b) 5,5'-DAII. In the positive scan, no noticeable peaks were observed.

VIII. Transistor fabrication and thin-film characterization

A 300-nm thermally grown SiO$_2$ on a highly-doped silicon wafer (capacitance ($C_i$) = 11.5 nF cm$^{-2}$) was cleaned in a piranha solution (70% H$_2$SO$_4$ + 30% H$_2$O$_2$) for 15 min, and then washed with distilled water. As a gate insulator layer, the TTC was thermally evaporated to form a 20-nm-thick TTC layer on the Si substrate. The active layer was formed by thermal deposition under a vacuum of ca. 1.0 × 10$^{-4}$ Pa, in
which the resulting thickness was 45 nm. Finally, the source and drain electrodes were formed by the vapor deposition of Au (50 nm). The standard channel length (L) and width (W) were 50 μm and 1000 μm, respectively. The organic field-effect transistor (OFET) properties were measured by a Keithley 4200 semiconductor parameter analyzer under vacuum conditions. The field-effect mobility (μ) and threshold voltage (Vth) were calculated in the saturation region using the equation, \( I_{DS} = \mu (W/C_i) L (V_G - V_{th})^2 \), where \( I_{DS} \) and \( V_G \) are the drain current and gate voltage, respectively. The capacitance including the TTC layer on the SiO₂ substrate was calculated to be \( 1.25 \times 10^{-8} \text{ F cm}^{-2} \). Atomic force microscopic (AFM) images were obtained using an SII Nanonavi SPA400 scanning probe microscope with an SII SI-DF40 cantilever. Two-dimensional grazing incidence wide-angle X-ray scattering (GIWAXS) patterns were obtained at SPring-8 on beamline BL45XU. The samples were irradiated at a fixed incident angle on the order of 0.10 ° and the GIWAXS patterns were recorded with a 2-D image detector (PILATUS3X 2M). The wavelength of the X-ray beam was 0.1 nm (energy of 12.39 KeV) and the camera length was 395 mm.
Figure S4. Typical output curves of (a) and (c) p-channel and (b) and (d) n-channel ((a) and (b) for 6,6'-T-DAII and (c) and (d) for 5,5'-T-DAII). Gate voltages ($V_g$) are changed from 20 V to -100 V for p-channel scan and from -20 V to 100 V for n-channel scan with 20 V steps.

References


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