Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Electronic Supplementary Information

Thieno[2,3,*a*]carbazole donor-based organic dyes for high efficient dye-sensitized solar cells

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General information

^{1}H NMR and ^{13}C NMR spectra were recorded on JEOL JMTC-270/54/SS (JASTEC, 400 MHz) and (JASTEC (700 MHz) spectrometers. ^{1}H NMR spectra are reported as follows: chemical shift in ppm (δ) relative to the chemical shifts of $CDCl_3$ at 7.26 ppm and DMSO-d_6 at 2.49 ppm, respectively. ^{13}C NMR spectra reported in ppm (δ) relative to the central line for $CDCl_3$ at 77 ppm and DMSO-d_6 at 39.7 ppm, respectively. High-resolution mass spectra were obtained on a BRUKER APEXIII spectrometer. The ionization potential (IP) of dyes bound to the nanocrystalline TiO_2 film was measured using the photoelectron spectrometer surface analyser (Riken Keiki, AC-3E). Optimized ground state geometry of the lowest-energy conformer of K-dyes were calculated using the hybrid DFT energy functional B3LYP and the basis set of DGDZVP (density Gauss double-ζ with polarization functions). Column chromatography was carried out employing Silica gel 60N (spherical, neutral, 40~100 μm, KANTO Chemical Co.). All other reagents and solvents commercially available were used without further purification unless otherwise noted. Intermediates **2a** and **2b** were prepared according to the reported method.[1,2]

[1] M. Melucci, G. Barbarella, M. Zambianchi, P. Di Pietro, and A. Bongini, *J. Org. Chem*. **2004**, *69*, 4821-4828.

[2] Y. Liu, X. Wan, F. Wang, J. Zhou, G. Long, J. Tian, and Y. Chen, *Adv. Mater*. **2011**, *23*, 5387-5391.

Fabrication of the DSCs

A double-layer TiO_2 photoelectrode (10 + 5) μm in thickness with a 10 μm thick nanoporous layer and a 5 μm thick scattering layer (area: 0.25 cm^2) was prepared by screen printing on conducting glass substrate. A dye solution of K-dyes with 3 x 10^{-4} M concentration in acetonitrile/*tert*-butyl alcohol (1/1, v/v) was used to up take the dye on to the TiO_2 film. Deoxycholic acid (DCA) (20 mM) as a co-adsorbent was added into the dye solution to prevent aggregation of the dye molecules. The TiO_2 films were immersed into the dye solution and then kept at 25 ^{o}C for 30 h. Photovoltaic measurements were performed in a sandwich type solar cell in conjunction with an electrolyte consisting of a solution of 0.6 M dimethylpropyl-imidazolium iodide (DMPII), 0.05 M I_2, 0.1M LiI and 0.5 M *tert*-butylpyridine (TBP) in acetonitrile (AN). The dye-deposited TiO_2 film and a platinum-coated conducting glass were separated by a Surlyn spacer (40 μm thick) and sealed by heating the polymer frame. Photocurrent density-voltage (I-V) of sealed solar cells was measured under AM 1.5 G simulated solar light at a light intensity of 100 mW cm^{-2} with a metal

mask of 0.25 cm^2. The photovoltaic parameters, *i.e.* short circuit current *(J_{sc})*, open circuit voltage (V_{oc}), fill factor (FF), and power conversion efficiency (η) were estimated from I-V characteristics under illumination. The photoemission yield curves were measured with a Riken Keiki Co. Ltd. model AC-3E photoelectron spectrometer surface analyser under atmosphere with a 0.2 mL min^{-1} N_2 flow. The adsorption amounts of dyes on the surface of TiO_2 films that were prepared under the same conditions with those fabricated into cells. TiO_2 films sensitized with K-dyes were immersed into 0.1 M NaOH solution for desorption of the dyes. The amounts of desorbed dyes were evaluated by their corresponding absorbance spectra.

Electrochemical impedance spectroscopy (EIS) and intensity modulated photo-voltage spectroscopy (IMVS) measurements

The IMVS spectra were measured with a potentiostat (Solartron 1287) equipped with a frequency response analyser (Solartron 1255B) at an open-circuit condition, based on a monochromatic illumination (420 nm) controlled by Lab-view system, to obtain the photovoltaic response induced by the modulated light. The modulated light was driven with a 10% AC perturbation current superimposed on a DC current in a frequency range from 0.1 to 10^6 Hz. The charge extraction method (CEM) was performed with the same monochromatic light source. The solar cell was illuminated at an open-circuit condition for 5s to attain a steady state and then the light source was switched off when the device simultaneously switched to a short-circuit condition to extract the charges generated at that light intensity. The electrochemical impedance spectra were measured with an impedance analyser (Solartron Analytical, 1255B) connected with a potentiostat (Solartron Analytical, 1287) under illumination using a solar simulator (WXS-155S-10: Wacom Denso Co. Japan). EIS spectra were recorded over a frequency range of 10^{-2} to 10^6 Hz at 298 K. The applied bias voltage and AC amplitude were set at the V_{oc} of the DSCs. The electrical impedance spectra were characterized using Z-View software (Solartron Analytical).

Ionization Potentials (IP) of K-dyes

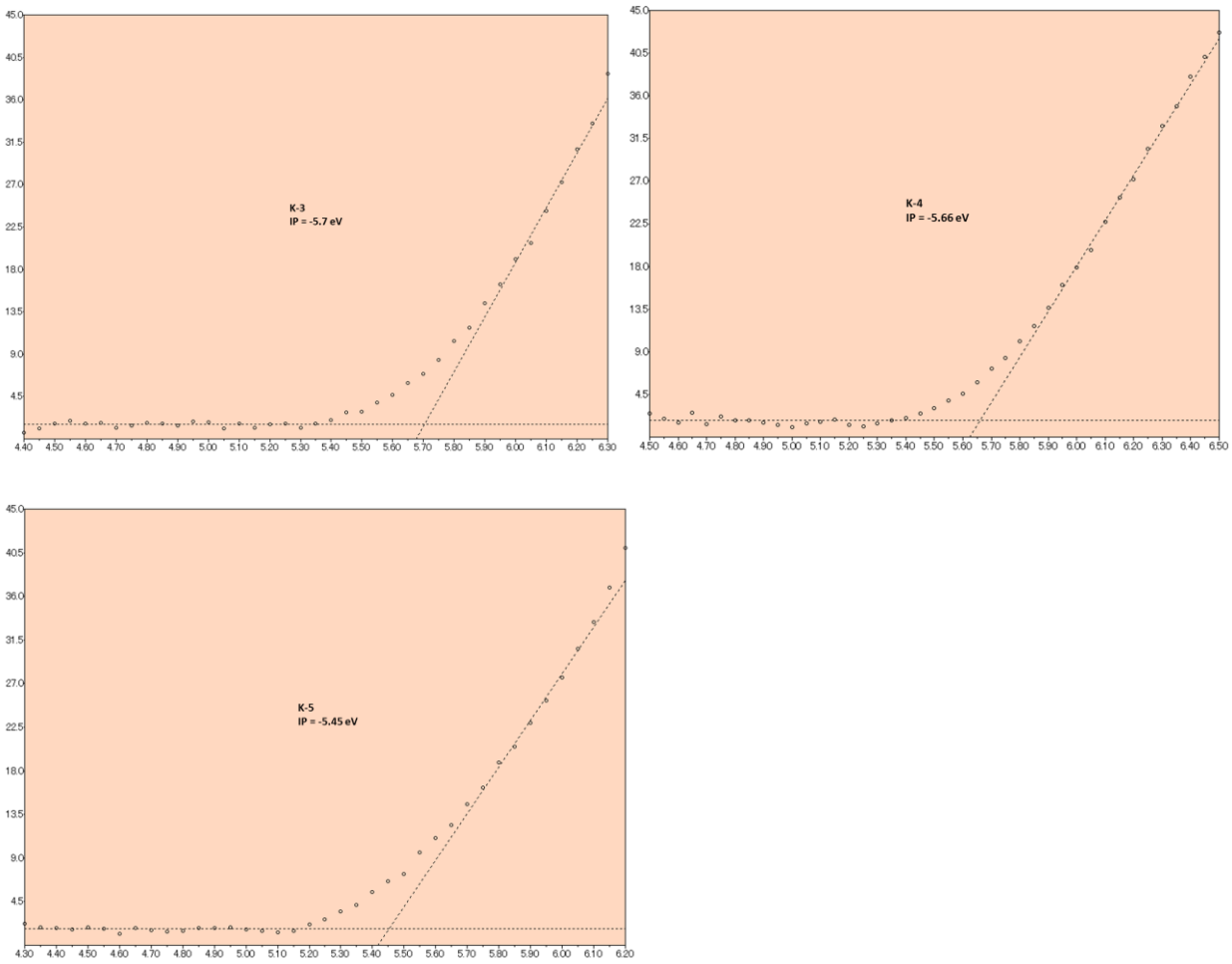


Fig S1 Ionization potentials of K-dyes.

Electrochemical cyclic voltammetry (CV) measurement of K-dyes

Potential values are versus Ag/AgCl reference electrode; 0.1 M $TBAPF_6$ as a supporting electrolyte in dichloromethane; redox potential of ferrocene is 0.37 V (vs Ag/AgCl) and we take the energy level of Fc/Fc+ as -4.8 eV. The HOMO energy levels of K-dyes were calculated from the onset of the first oxidation potential (E^1_{ox}) according to the equation, HOMO = -(4.43 + E^1_{ox}) (eV);

K-3: E^1_{ox}: 1.24 eV; HOMO: 5.67 eV

K-4: E^1_{ox}: 1.16 eV; HOMO: 5.59 eV

K-5: E^1_{ox}: 1.12 eV; HOMO: 5.55 eV

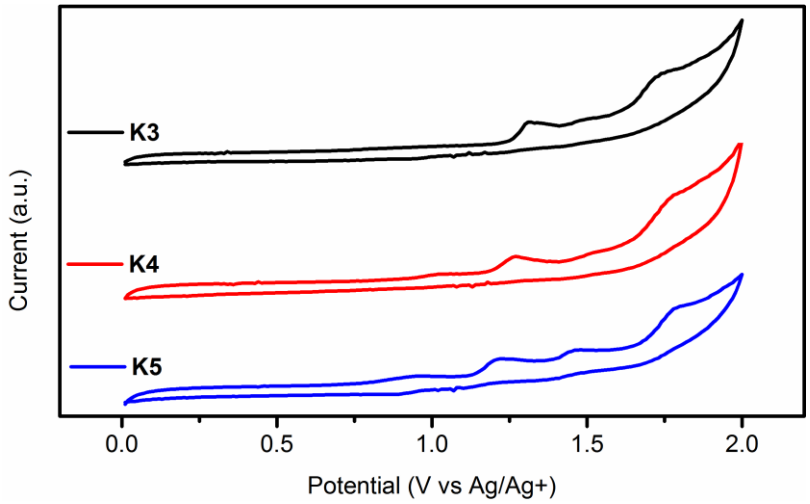


Fig. S2 Cyclic voltammograms of K-dyes.

DFT calculation of K-dyes

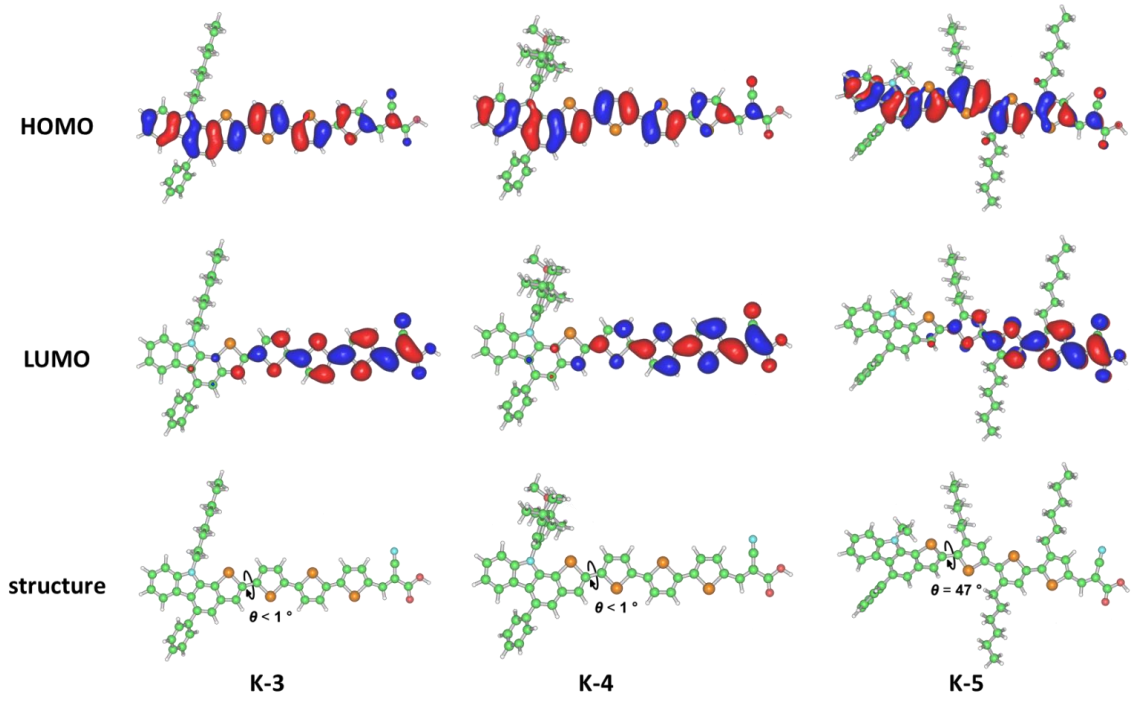
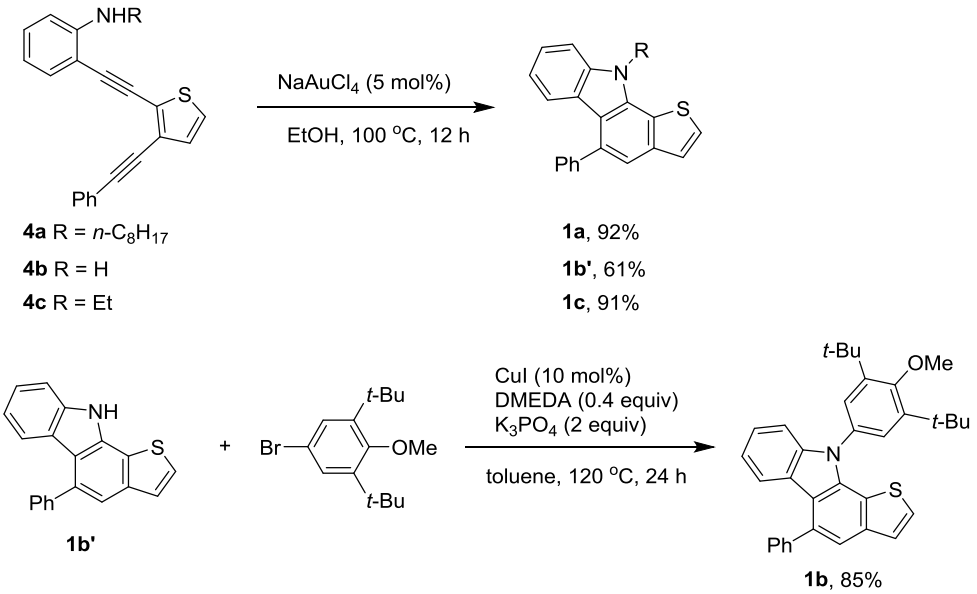


Fig. S3 The DFT calculation of the frontier orbitals and the optimized ground state structures of K-dyes.

Synthesis of K-dyes and analytic data



10-Octyl-5-phenyl-2-(thiophen-2-yl)-10*H*-thieno[2,3-a]carbazole (1a)

A mixture of **4a** (2.9 mmol) and $NaAuCl_4$ (5 mol%) in dry ethanol was heated at 100 °C in sealed vial overnight. After TLC monitor, the reaction mixture was filtered by Florisil, and the filtrate was concentrated in vacuum. The crude mixture was purified with silica-gel column chromatography (hexane/CH_2Cl_2 = 5/1) to produce the desired compound **1a** in a 92% yield as a yellow oil. **1a**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77–7.40 (m, 11H), 7.06 (dd, *J* = 7.6, 7.6 Hz, 1H), 4.69 (t, *J* = 7.6 Hz, 2H), 2.10–1.95 (m, 2H), 1.67–1.55 (m, 2H), 1.53–1.25 (m, 8H), 0.95 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.4, 139.9, 139.1, 135.4, 135.1, 129.4, 128.3, 127.3, 125.0, 124.5, 124.4, 123.0, 122.1, 120.5, 118.9, 116.5, 116.4, 108.7, 44.8, 31.9, 30.8, 29.4. 29.3, 27.1, 22.7, 14.2; HRMS (ESI): m/z: calcd for $C_{28}H_{29}NSNa$ ($M+Na^+$), 434.1913; found, 434.1913.

10-(3,5-Di-*tert*-butyl-4-methoxyphenyl)-5-phenyl-10*H*-thieno[2,3-a]carbazole (1b)

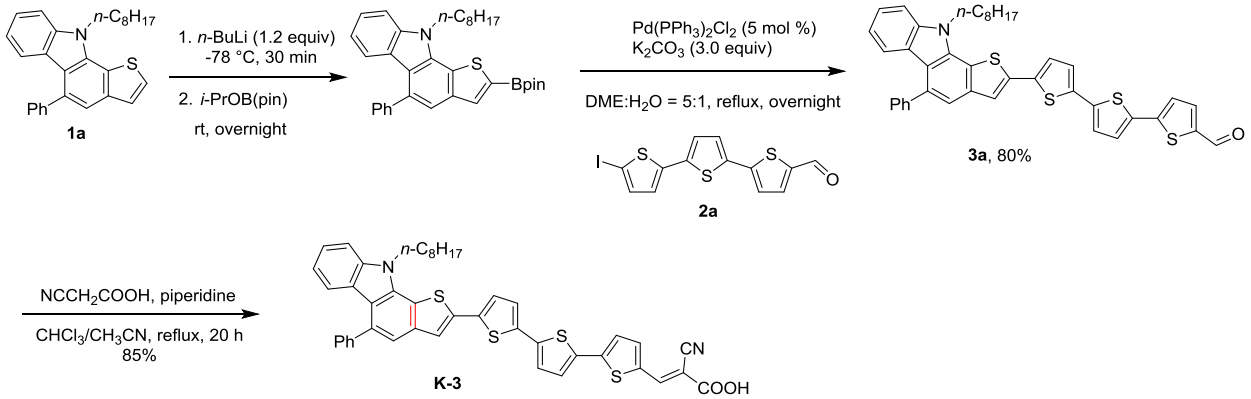
1b' (R = H) was prepared in 61% yield under the same conditions with **1a** by using **4b** a starting substrate. To the mixture of **1b'** (202 mg, 0.675 mmol), 5-bromo-1,3-di-*tert*-butyl-2-methoxybenzene (182 mg, 1.08 mmol), CuI (13 mg, 0.0675 mmol), *N*,*N*-dimethylethylene diamine (DMEDA, 30 μL, 0.27 mmol), and K_3PO_4 (286 mg, 1.35 mmol) was added toluene (3 mL) at room temperature under argon. The reaction mixture was then heated to 120 °C for 24 h. After removal of the solvent, the residue was directly transferred into silica gel chromatography using hexane/ethyl acetate (10:1) as eluent, yielding the corresponding product **1b** (297 mg, 85%) as a yellow solid. **1b**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77-7.72 (m, 2H), 7.63-7.51 (m, 4H), 7.50-7.45 (m, 4H), 7.40-7.30 (m, 3H), 7.10-7.05 (m, 1H), 3.92 (s, 3H), 1.53 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2, 145.1, 141.5, 141.3, 139.3, 136.0, 134.9, 130.9, 129.4,

128.3, 127.7, 127.3, 125.9, 124.7, 124.2, 122.9, 121.9, 121.2, 119.4, 116.7, 116.2, 109.9, 64.8, 36.1, 32.0; HRMS (ESI): m/z: calcd for $C_{35}H_{35}ONS$ ($M+H^+$), 518.2512; found, 518.2511.

10-Ethyl-5-phenyl-10*H*-thieno[2,3-a]carbazole (1c)

1c was prepared in a 91% yield under the same conditions with **1a** by using **4c** a starting substrate. **1c**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10-8.03 (m, 2H), 7.95-7.80 (m, 5H), 7.78-7.68 (m, 3H), 7.64 (d, *J* = 5.6 Hz, 1H), 7.47-7.40 (m, 1H), 4.90 (q, *J* = 7.6 Hz, 2H), 1.80 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.2, 139.3, 138.9, 135.0, 134.9, 129.2, 128.1, 127.2, 124.8, 124.5, 124.2, 123.0, 122.0, 120.3, 118.9, 116.4, 108.3, 39.0, 15.4; HRMS (ESI): m/z: calcd for $C_{22}H_{17}NS$ ($M+H^+$), 328.1154; found, 328.1154.

Synthesis of K-3



5''-(10-Octyl-5-phenyl-10*H*-thieno[2,3-a]carbazol-2-yl)-[2,2':5',2''-terthiophene]-5-carbaldehyde (3a)

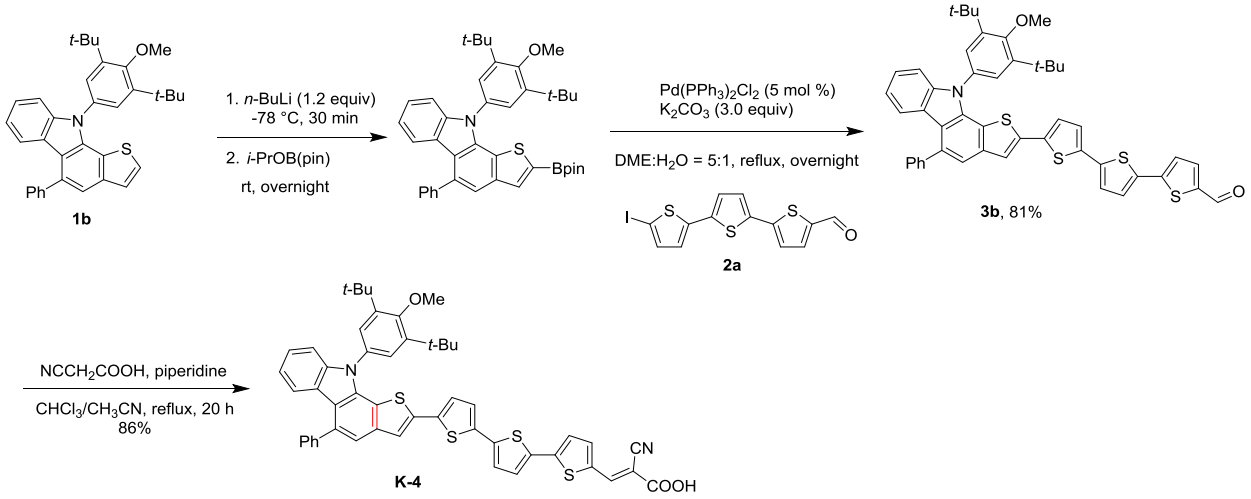
Under an argon atmosphere, 1.6 M of *n*-BuLi (0.52 mL, 0.84 mmol) in hexane was added to **1a** (288 mg, 0.7 mmol) in dry THF (10 mL) at -78 °C. The reaction mixture was stirred for 30 min at -78 °C before 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxabororane (195 mg, 1.05 mmol) was added. After stirring for 12 h at room temperature, the reaction was quenched with a saturated NH_4Cl aqueous solution and was extracted with Et_2O for 3 times. The organic layer was combined and dried with Na_2SO_4. After concentration, the yellow residue (boronate) was directly used to the next step without further purification. To a solution of the above boronate, the trithiophene **2a** (233 mg, 0.58 mmol),[1] and $PdCl_2(PPh_3)_2$ (20.3 mg, 0.029 mmol) in DME (10 mL) was added K_2CO_3 (240 mg, 1.74 mmol) in H_2O (2 ml). The reaction mixture was refluxed under an argon atmosphere for 12 h. The reaction mixture was extracted with EtOAc and the organic layer was washed with brine. The aqueous phase was extracted with EtOAc (3 times). The combined organic phases were washed with water then brine, and dried with Na_2SO_4. After concentration, the residue was purified by silica gel chromatography using hexane/EtOAc (10:1) as eluents, giving the corresponding aldehyde **3a** (318 mg, 80% from **2a**) as a red solid. **3a**: ^{1}H NMR (400

MHz, $CDCl_3$) δ 9.75 (s, 1H), 7.65-7.60 (m, 2H), 7.59-7.47 (m, 5H), 7.45-7.30 (m, 4H), 7.20-7.15 (m, 2H), 7.10-6.95 (m, 4H), 4.54 (t, *J* = 7.6 Hz, 2H), 2.00-1.90 (m, 2H), 1.60-1.50 (m, 2H), 1.50-1.20 (m, 8H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.1, 146.3, 141.3, 141.0, 140.0, 139.5, 138.4, 137.1, 136.8, 135.8, 135.5, 134.9, 134.9, 134.5, 129.2, 128.3, 127.4, 126.7, 125.2, 124.9, 124.8, 124.5, 123.8, 122.9, 122.0, 121.1, 119.4, 119.0, 117.1, 116.3, 108.7, 44.7, 31.8, 30.7, 29.4, 29.2, 27.0, 22.7, 14.2; HRMS (ESI): m/z: calcd for $C_{41}H_{35}ONS_4$ ($M+H^+$), 686.1674; found, 686.1673.

K-3 dye

To a solution of **3a** (103 mg, 0.15 mmol) and cyanoacetic acid (38 mg, 0.45 mmol) in chloroform (1.5 mL) and acetonitrile (1.5 mL) was added piperidine (104 μL, 1.05 mmol). The reaction mixture then refluxed for 20 h. After removal of the solvent, the residue was purified by siliga gel column chromatography using $CHCl_3$:EtOH (10:1 v/v) as eluents, giving **K-3** as a dark red solid (85%, 96 mg). **K-3:** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.66-7.22 (m, 15H), 7.13 (d, *J* = 8.0 Hz, 1H), 6.88 (dd, *J* = 7.6, 7.6 Hz, 1H), 4.42 (broad, 3H), 1.78-1.75 (m, 2H), 1.41-1.20 (m, 10H), 0.81 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR is not shown due to low solubility; HRMS (ESI): m/z: calcd for $C_{44}H_{35}O_2N_2S_4$ ($M-H^+$), 751.1586; found, 751.1585.

Synthesis of K-4 dye



5''-(10-(3,5-Di-tert-butyl-4-methoxyphenyl)-5-phenyl-10*H*-thieno[2,3-a]carbazol-2-yl)-[2,2':5',2''-terthiophene]-5-carbaldehyde (3b)

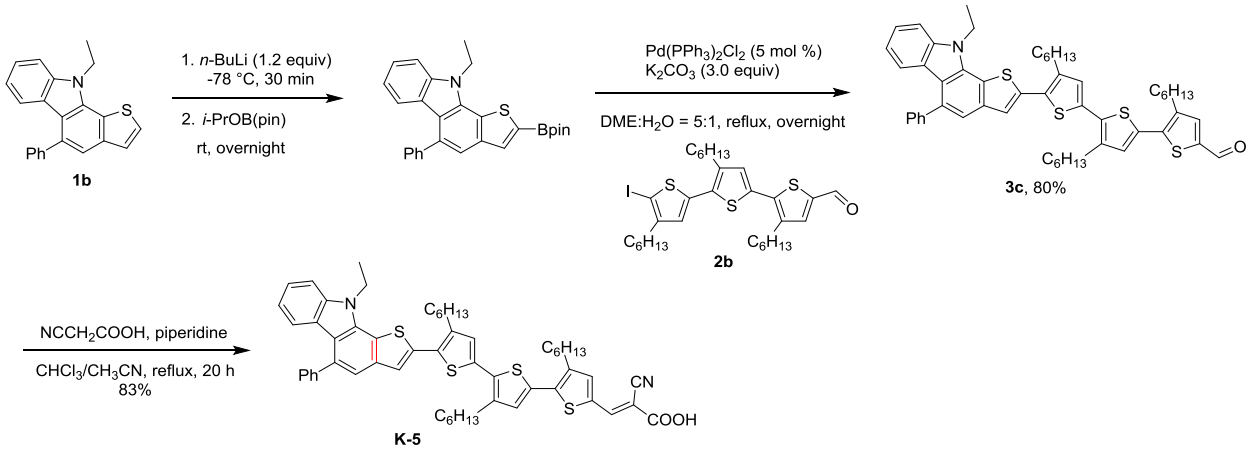
3b was prepared in 81% yield as a red solid by using the similar procedure of **3a** from **1b**. **3b**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.75 (s, 1H), 7.79-7.68 (m, 2H), 7.63-7.55 (m, 3H), 7.52 (d, *J* = 0.8 Hz, 1H), 7.50-7.45 (m, 4H), 7.44 (s, 1H), 7.42-7.33 (m, 2H), 7.15 (d, *J* = 3.6 Hz, 1H), 7.11-7.05 (m, 2H), 7.04 (d, *J* = 4.0 Hz, 1H), 6.99 (d, *J* = 4.0 Hz, 1H), 6.96 (d, *J* = 3.6 Hz, 1H), 3.96 (s, 1H), 1.57 (s, 18H); ^{13}C NMR (100

MHz, $CDCl_3$) δ 182.0, 160.3, 146.2, 145.1, 141.6, 141.3, 141.0, 139.7, 138.3, 137.3, 137.0, 135.9, 135.5, 135.5, 135.4, 134.4, 130.7,129.3, 128.3, 127.5, 127.4, 126.7, 125.0, 124.9, 124.7, 124.4, 123.8, 122.9, 121.9, 120.5, 120.1, 119.6, 116.9, 116.7, 109.9, 64.9, 36.1, 32.0; HRMS (ESI): m/z: calcd for $C_{48}H_{41}O_2NS_4$ (M^+), 791.2014; found, 791. 2014.

K-4 dye

K-4 dye was synthesized in 86% yield using the similar procedure of **K-3** from **3b**. **K-4**: red solid; ^{1}H NMR (400 MHz, DMSO-d_6/$CDCl_3$) δ 8.22 (s, 1H), 7.86 (d, *J* = 4.0 Hz, 1H), 7.73 (s, 1H), 7.63-7.45 (m, 8H), 7.42 (s, 2H), 7.35-7.20 (m, 5H), 7.14 (d, *J* = 3.6 Hz, 1H), 6.99 (t, *J* = 8.0 Hz, 1H), 3.88 (s, 3H), 1.47 (s, 18H); ^{13}C NMR (100 MHz, DMSO-d_6/$CDCl_3$) δ 163.3, 160.0, 144.8, 141.1, 140.4, 140.0, 139.6, 137.3, 136.2, 135.2, 135.1, 135.1, 134.9, 134.4, 134.1, 130.0, 128.9, 128.4, 127.6, 127.1, 125.9, 125.5, 125.4, 125.3, 124.9, 122.3, 121.2, 120.9, 119.6, 119.3, 116.7, 116.6, 116.3, 109.7, 64.7, 39.1, 31.6; HRMS (ESI): m/z: calcd for $C_{51}H_{41}O_3N_2S_4$ ($M\text{-}H^+$), 857.2005; found, 857.2004.

Synthesis of K-5



3,4',4''-Trihexyl-5''-iodo-[2,2':5',2''-terthiophene]-5-carbaldehyde (2b)[2]

2b: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 7.52 (s, 1H), 7.06 (s, 1H), 6.78 (s, 1H), 2.76 (t, *J* = 8.0 Hz, 2H), 2.68 (t, *J* = 8.0 Hz, 2H), 2.52 (t, *J* = 8.0 Hz, 2H), 1.75-1.55 (m, 6H), 1.50-1.30 (m, 18H), 0.90 (t, *J* = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.8, 147.4, 140.5, 140.0, 139.9, 139.8, 139.5, 138.6, 132.5, 132.1, 129.8, 126.3, 74.6, 32.1, 31.5, 31.5, 30.3, 30.0, 29.8, 29.3, 29.1, 29.0, 28.8, 22.5, 14.0, 14.0; HRMS (ESI): m/z: calcd for $C_{31}H_{43}IOS_3$ (M^+), 654.1515; found, 654.1515.

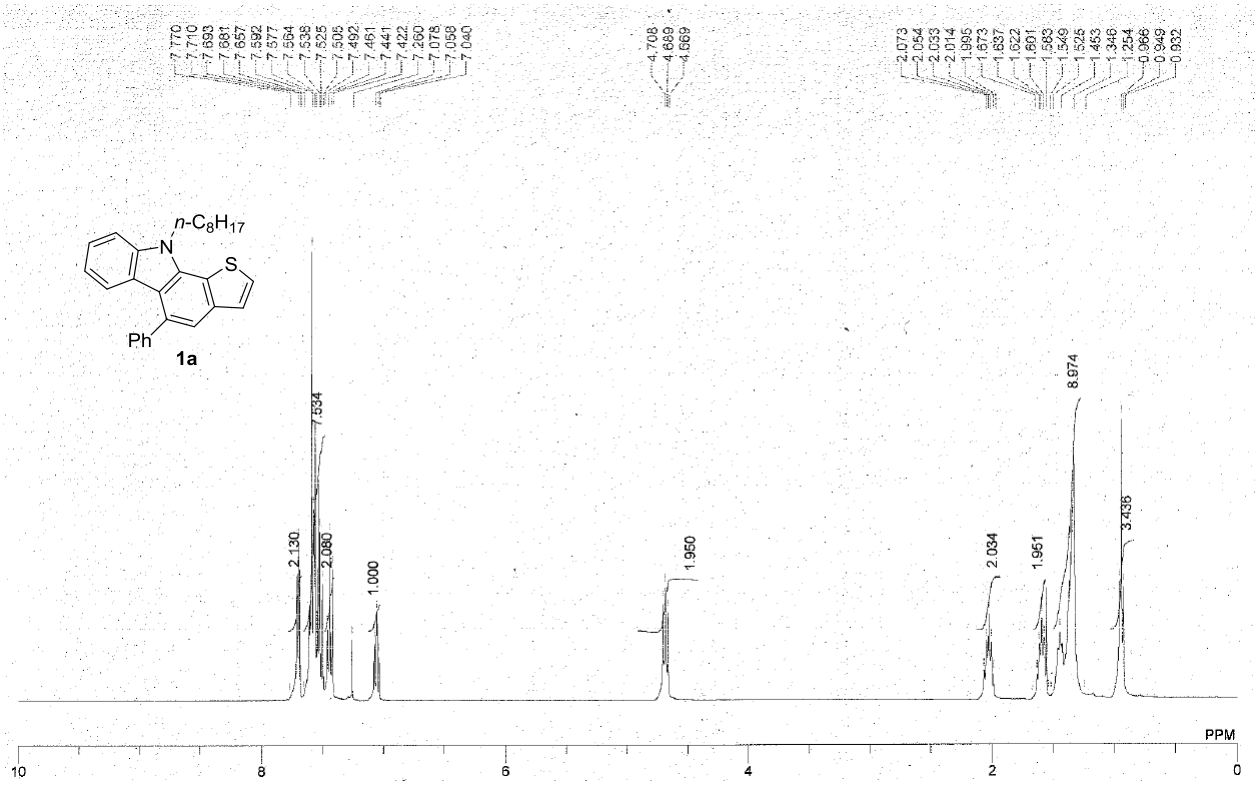
5''-(10-Ethyl-5-phenyl-10*H*-thieno[2,3-a]carbazol-2-yl)-3,4',4''-trihexyl-[2,2':5',2''-terthiophene]-5-carbaldehyde (3c)

3c was prepared in 80% yield under the similar procedure with the **3a** and **3b** using **2b** as an aldehyde source instead of **2a** from **1c**. **3c**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 (s, 1H), 7.72-7.65 (m, 2H), 7.62-7.47 (m, 8H), 7.46-7.38 (m, 2H), 7.16 (s, 1H), 7.10 (s, 1H), 7.05 (dd, *J* = 7.2, 7.2 Hz, 1H), 4.76 (q, *J* = 7.2 Hz, 2H), 2.94 (dd, *J* = 7.6, 7.6 Hz, 2H), 2.86 (dd, *J* = 7.2, 7.2 Hz, 2H), 2.84 (dd, *J* = 7.2, 7.2 Hz, 2H), 1.85-1.68 (m, 6H), 1.63 (t, *J* =7.2 Hz, 3H), 1.55-1.35 (m, 18H), 1.00-0.93 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.2, 141.1, 141.0, 140.9, 140.2, 140.1, 139.9, 139.6, 139.2, 138.8, 135.5, 134.6, 134.3, 134.1, 132.6, 132.5, 130.8, 130.3, 129.2, 129.0, 128.2, 127.4, 124.7, 123.3, 123.1, 122.1, 120.2, 119.0, 116.8, 116.3, 108.4, 39.4, 31.6, 31.6, 31.6, 30.7, 30.4, 30.2, 29.5, 29.4, 29.4, 29.3, 29.2, 29.1, 22.6, 22.6, 22.6, 15.7, 14.1, 14.1; HRMS (ESI): m/z: calcd for $C_{53}H_{59}ONS_4$ ($M+H^+$), 854.3552; found, 854.3551.

K-5 dye

K-5 dye was synthesized in 83% yield under the similar procedure with **K-3** and **K-4** dyes from **3c**. **K-5:** red solid; ^{1}H NMR (400 MHz, DMSO-d_6/$CDCl_3$) δ 8.00 (s, 1H), 7.67 (s, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.60-7.50 (m, 7H), 7.36 (t, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.14 (s, 1H), 6.94 (dd, *J* = 7.6, 7.6 Hz, 1H). 4.70 (q, *J* = 7.6 Hz, 2H), 2.87-2.72 (m, 6H), 1.75-1.50 (m, 6H), 1.50-1.20 (m, 21H), 0.90-0.82 (m, 9H); ^{13}C NMR (100 MHz, DMSO-d_6/$CDCl_3$) δ 163.8, 143.7, 141.0, 140.5, 140.3, 139.9, 139.3, 139.0, 137.8, 135.0, 134.0, 133.6, 133.4, 133.3, 132.1, 131.4, 130.2, 130.0, 129.1, 128.8, 128.3, 127.6, 127.5, 124.8, 123.2, 122.3, 121.5, 121.2, 119.4, 118.9, 117.4, 117.0, 116.2, 116.2, 109.1, 56.0, 30.9, 30.9, 29.9, 29.7, 29.4, 28.8, 28.6, 28.5, 28.4, 28.4, 21.9, 18.3, 15.3, 13.7; HRMS (ESI): m/z: calcd for $C_{56}H_{59}O_2N_2S_4$ (M-H+), 919.3464; found, 919.3464.

NMR spectra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7.441
7.422
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7.078
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7.040
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1.525
1.453
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0.966
0.949
0.932
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N
S
Ph
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7.534
2.080
1.000
1.950
2.034
1.951
8.974
3.436
PPM
10
8
6
4
2
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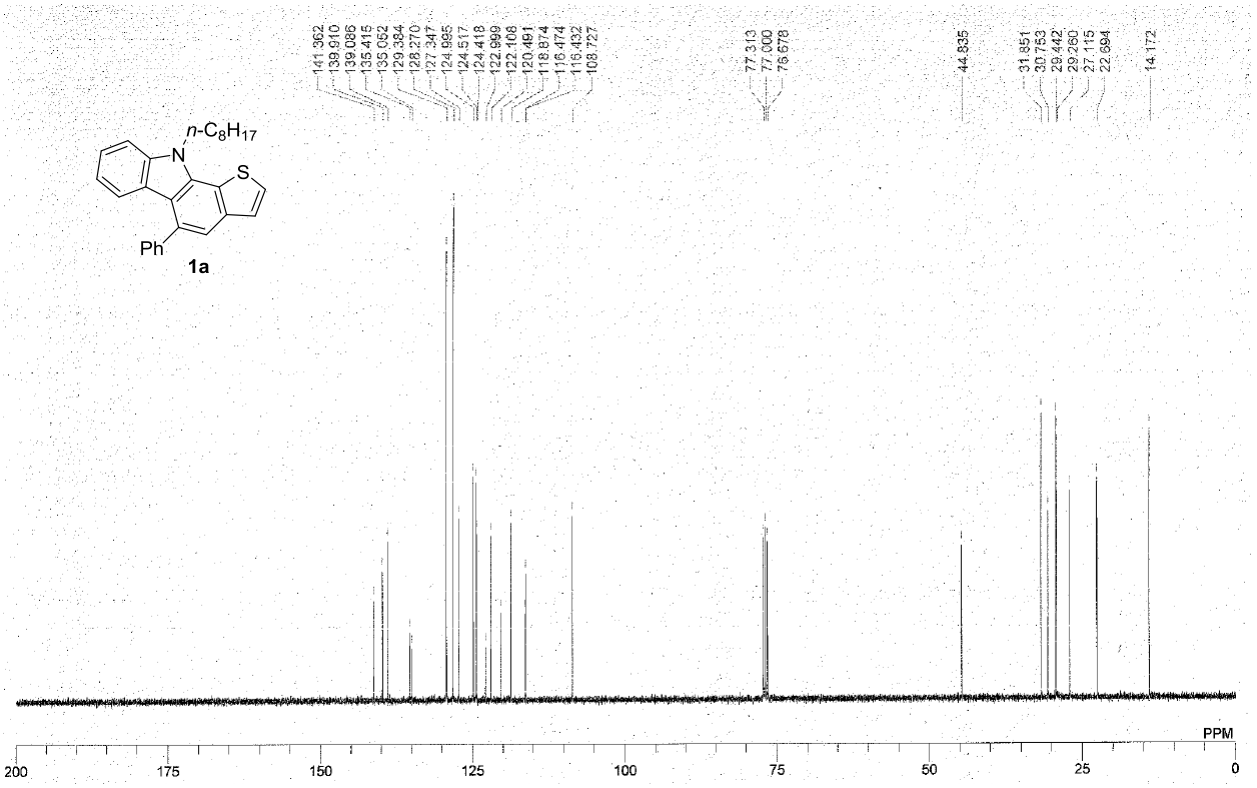
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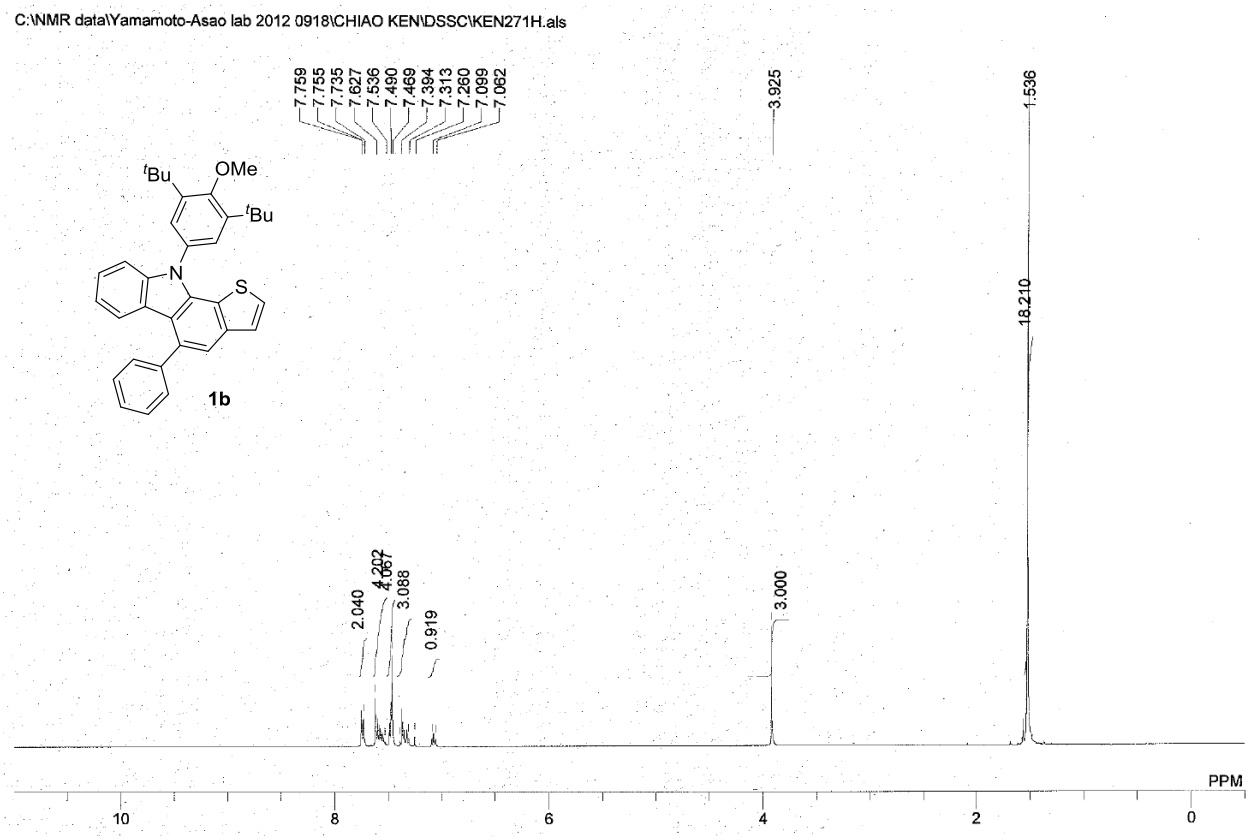
139.910
139.086
135.415
135.052
129.384
128.270
127.347
124.985
124.517
124.418
122.989
122.108
120.491
118.874
116.474
116.432
108.727
77.313
77.000
76.678
44.835
31.851
30.753
29.442
29.260
27.115
22.594
14.172
n-C8H17
N
S
Ph
1a
PPM
200
175
150
125
100
75
50
25
0


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN271H.als
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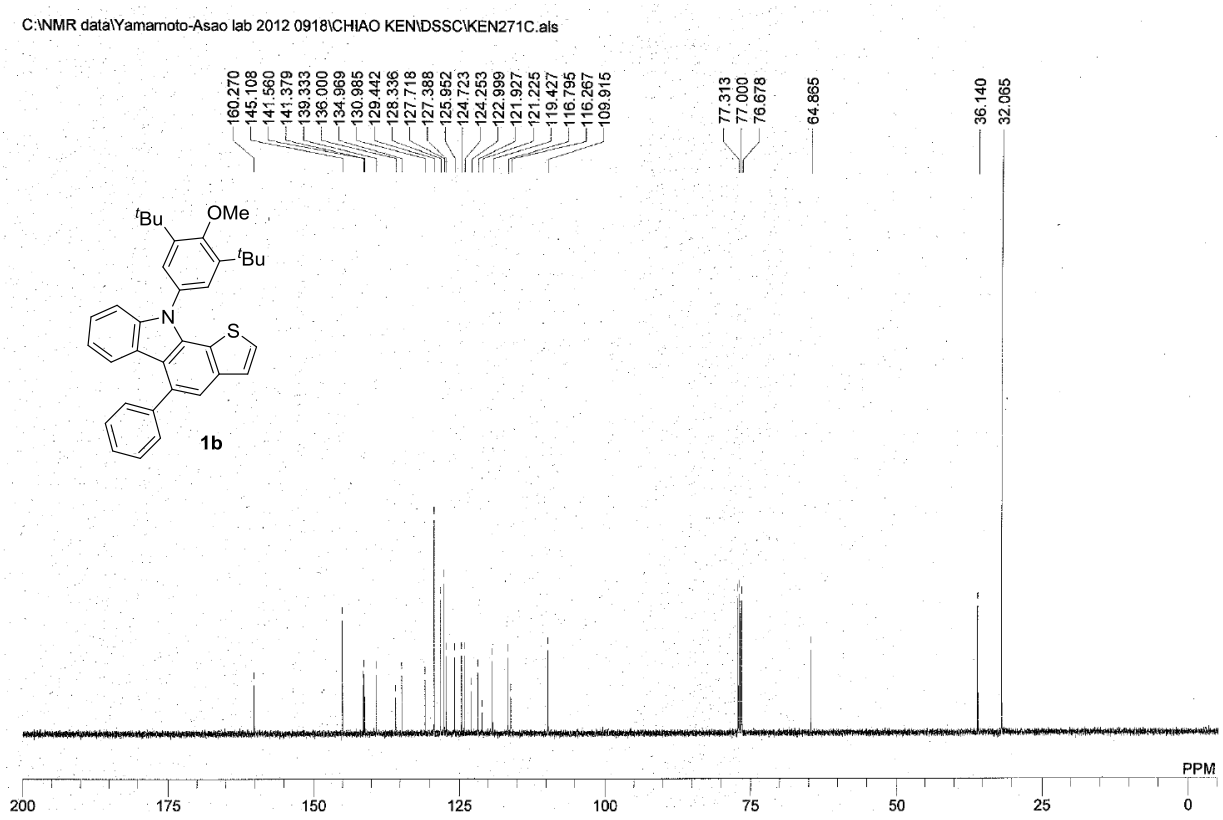
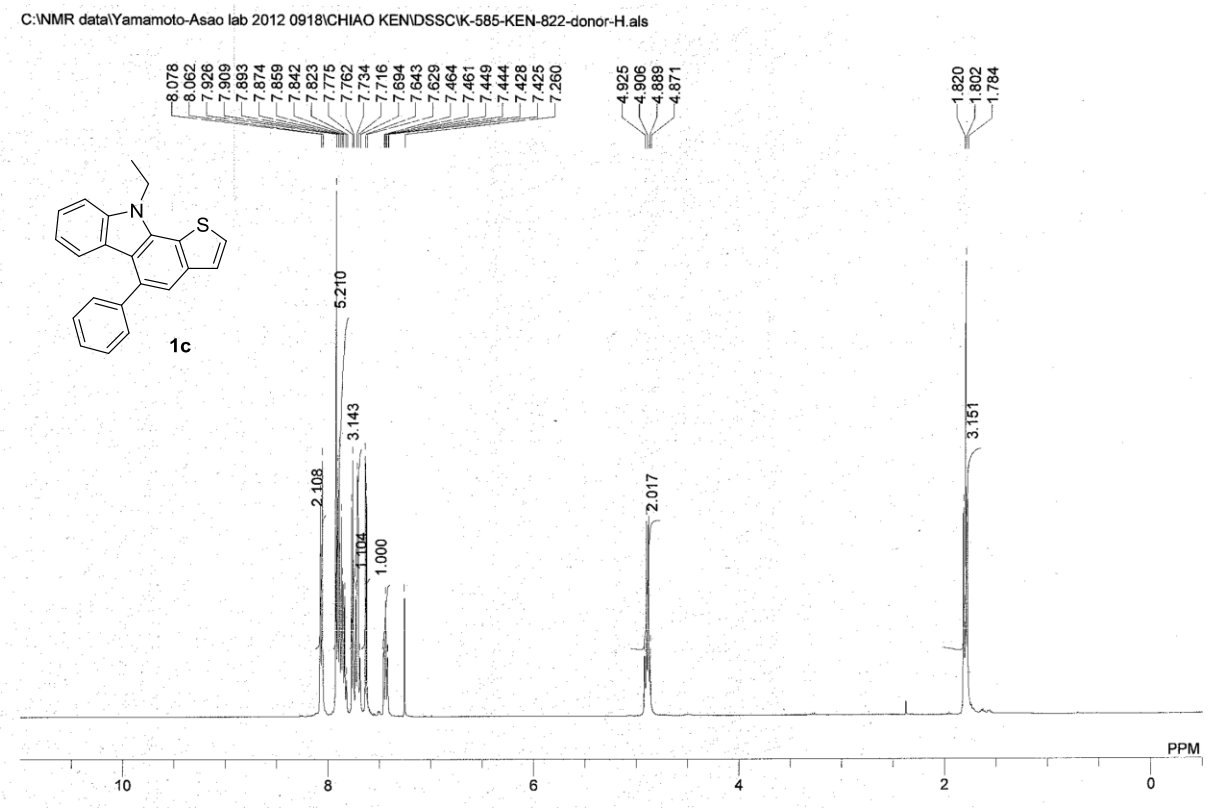
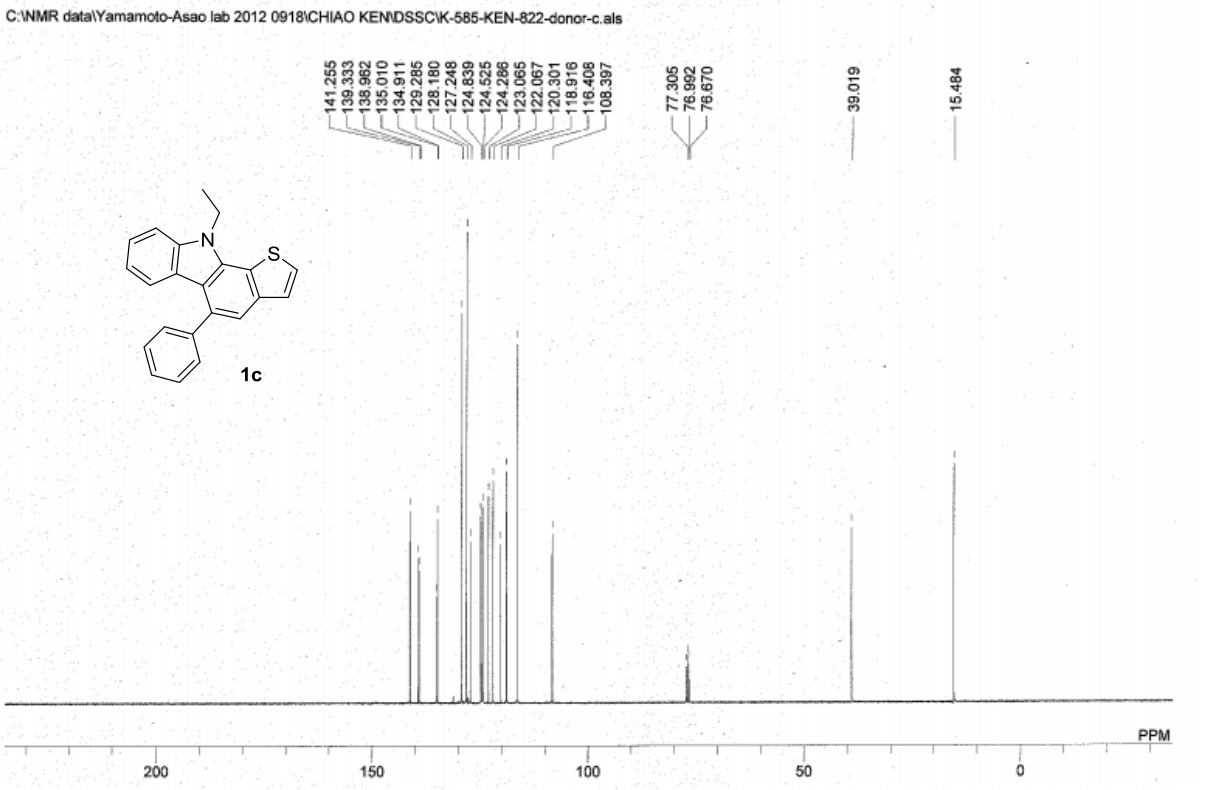
7.755
7.735
7.627
7.536
7.490
7.469
7.384
7.313
7.260
7.099
7.062
3.925
1.536
18.210
2.040
4.202
4.067
3.088
0.919
3.000
tBu
OMe
tBu
N
S
1b
PPM
10
8
6
4
2
0


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN271C.als
160.270
145.108
141.560
141.379
139.333
136.000
134.969
130.985
129.442
128.336
127.718
127.388
125.952
124.723
124.253
122.999
121.927
121.225
119.427
116.795
116.267
109.915
77.313
77.000
76.678
64.865
36.140
32.065
tBu
OMe
tBu
N
S
1b
PPM
200
175
150
125
100
75
50
25
0


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\K-585-KEN-822-donor-H.als
8.078
8.062
7.926
7.909
7.893
7.874
7.859
7.842
7.823
7.775
7.762
7.734
7.716
7.694
7.643
7.629
7.464
7.461
7.449
7.444
7.428
7.425
7.260
4.925
4.906
4.889
4.871
1.820
1.802
1.784
1c
5.210
3.143
2.108
1.104
1.000
2.017
3.151
PPM
10
8
6
4
2
0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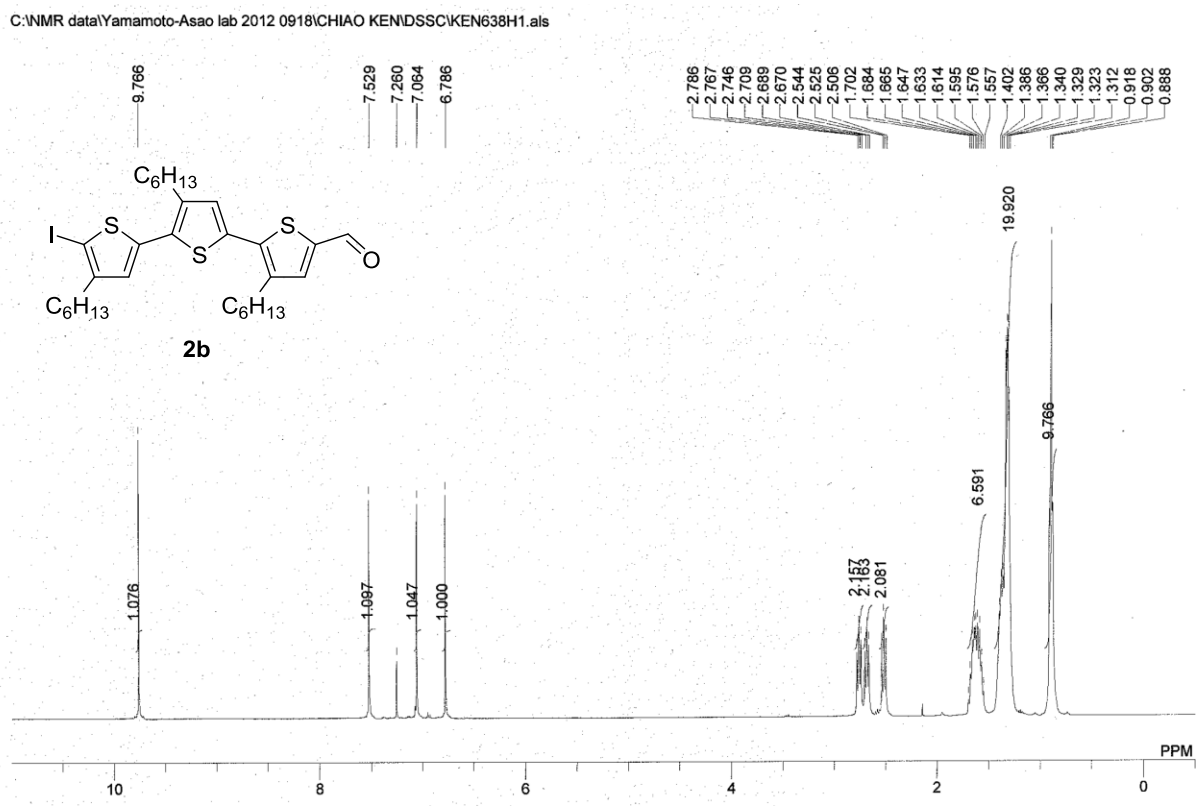
139.333
138.962
135.010
134.911
129.285
128.180
127.248
124.839
124.525
124.286
123.065
122.067
120.301
118.916
116.408
108.397
77.305
76.992
76.670
39.019
15.484
1c
PPM
200
150
100
50
0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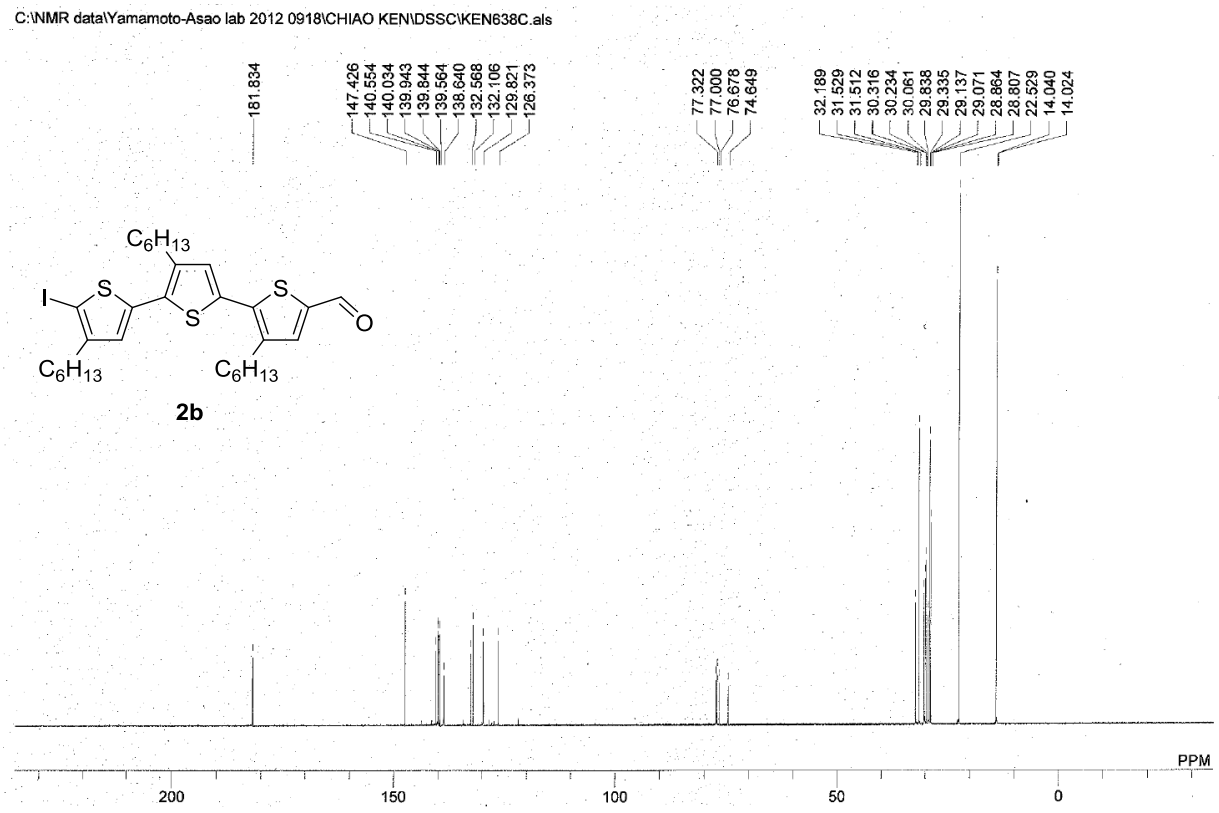
7.529
7.260
7.064
6.786
2.786
2.767
2.746
2.709
2.689
2.670
2.544
2.525
2.506
1.702
1.684
1.665
1.647
1.633
1.614
1.595
1.576
1.557
1.402
1.386
1.366
1.340
1.329
1.323
1.312
0.918
0.902
0.888
C6H13
I
S
S
S
O
C6H13
C6H13
2b
19.920
9.766
6.591
2.167
2.163
2.081
1.076
1.097
1.047
1.000
PPM
10
8
6
4
2
0


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN638C.als
181.834
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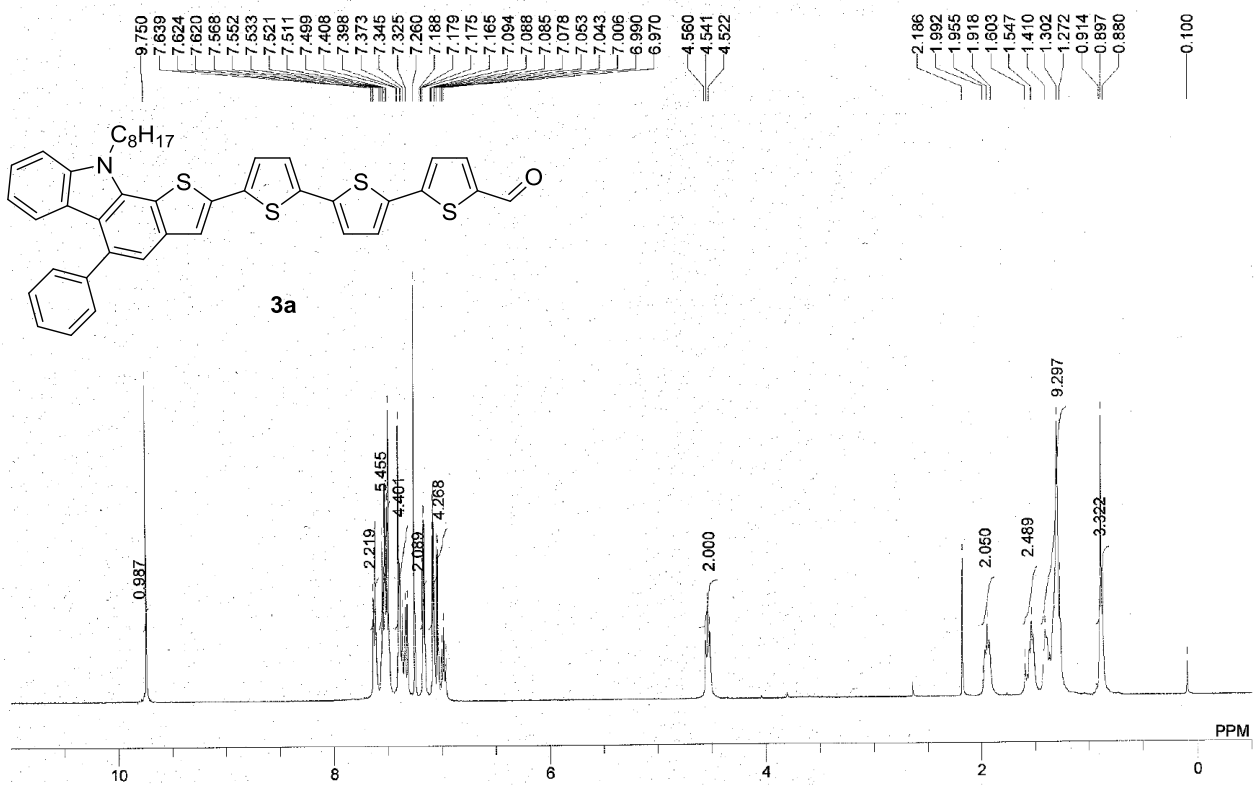
140.554
140.034
139.943
139.844
139.564
138.640
132.568
132.106
129.821
126.373
77.322
77.000
76.678
74.649
32.189
31.529
31.512
30.316
30.234
30.061
29.838
29.335
29.137
29.071
28.864
28.807
22.529
14.040
14.024
C6H13
I
S
S
S
O
C6H13
C6H13
2b
PPM
200
150
100
50
0

C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN-857-H donor aldhyde .als



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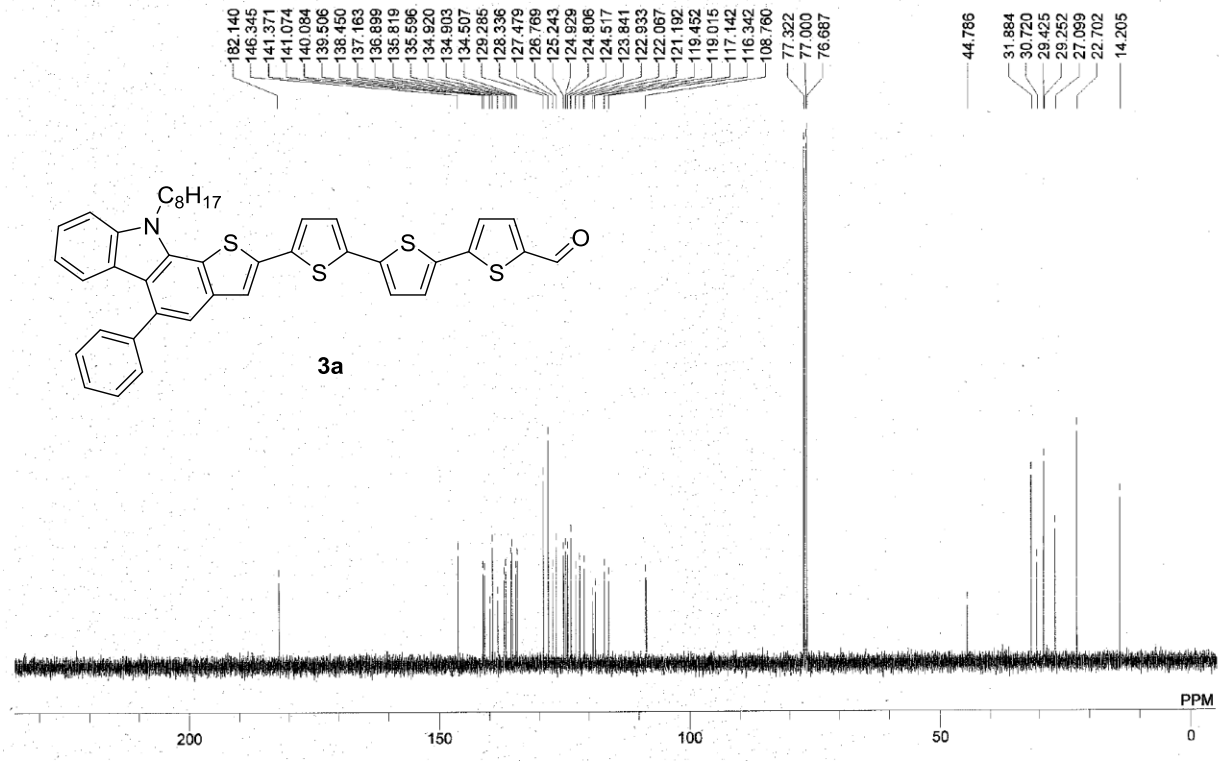

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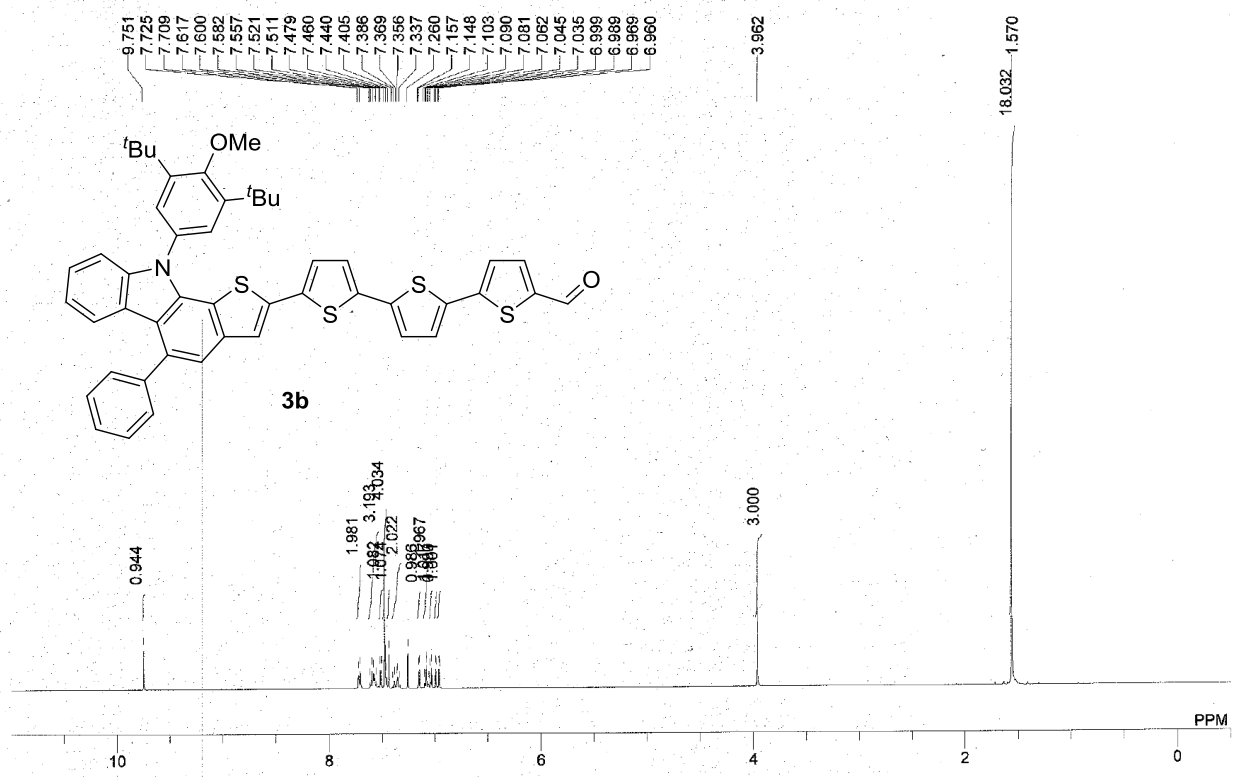

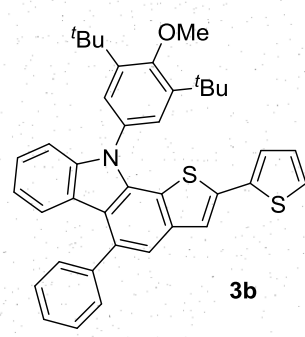

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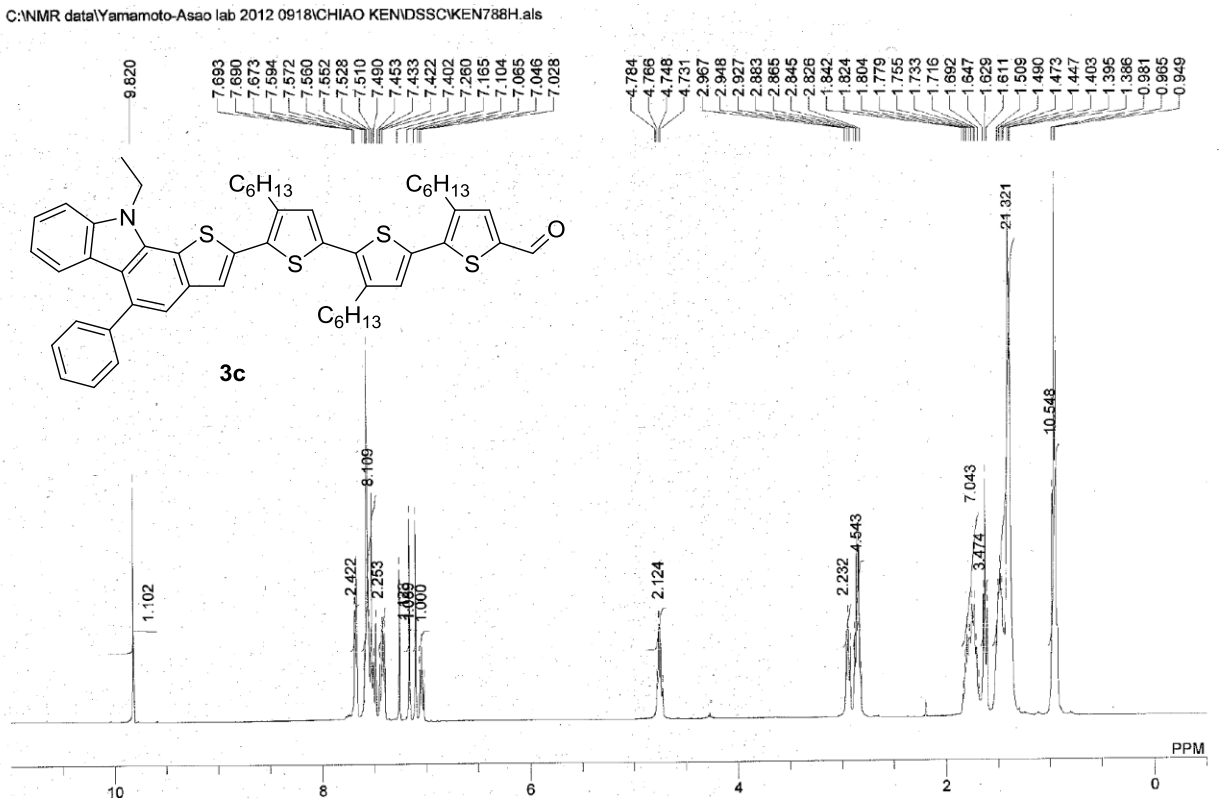
C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN788H.als
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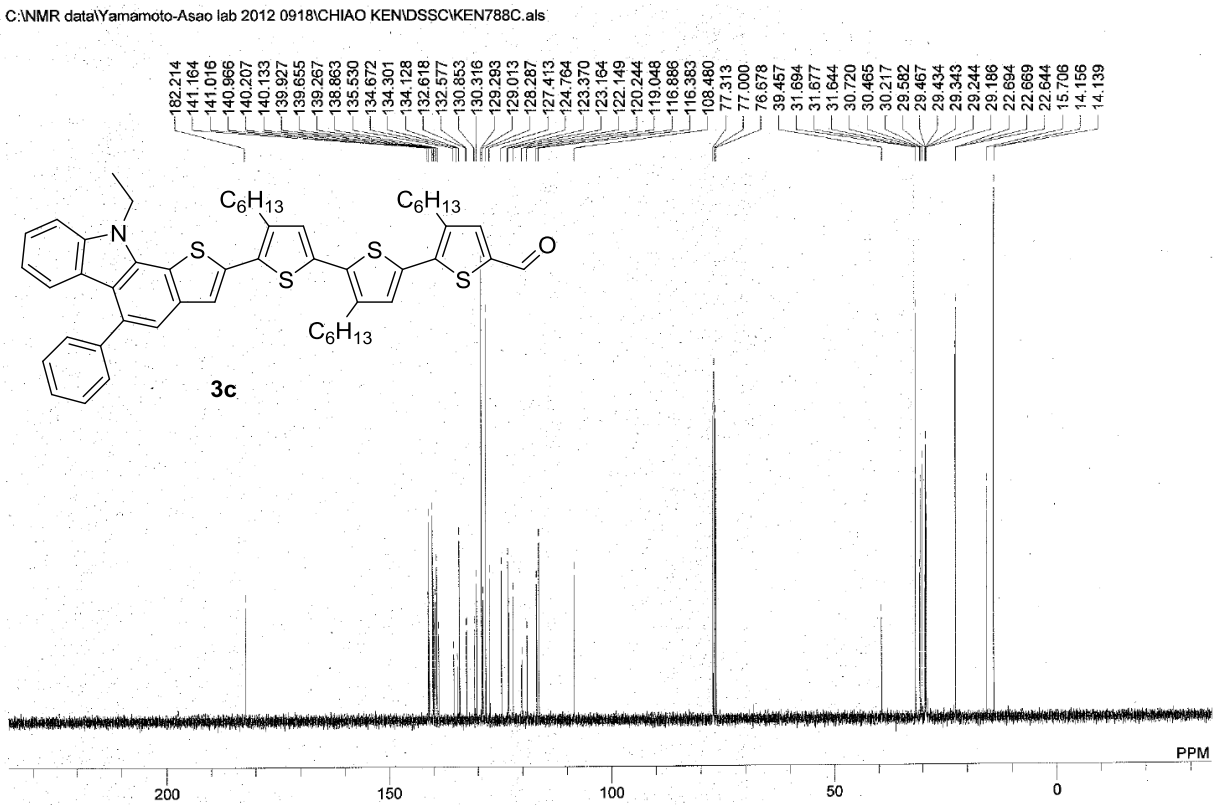
7.690
7.673
7.594
7.572
7.560
7.552
7.528
7.510
7.490
7.453
7.433
7.422
7.402
7.260
7.165
7.104
7.065
7.046
7.028
4.784
4.766
4.748
4.731
2.967
2.948
2.927
2.883
2.865
2.845
2.828
1.842
1.824
1.804
1.779
1.755
1.733
1.716
1.692
1.647
1.629
1.611
1.509
1.490
1.473
1.447
1.403
1.395
1.386
0.981
0.965
0.949
C6H13
C6H13
C6H13
N
S
S
S
S
O
3c
21.321
10.548
8.109
7.043
3.474
2.422
2.253
2.232
4.543
2.124
1.102
1.000
PPM
10
8
6
4
2
0


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN788C.als
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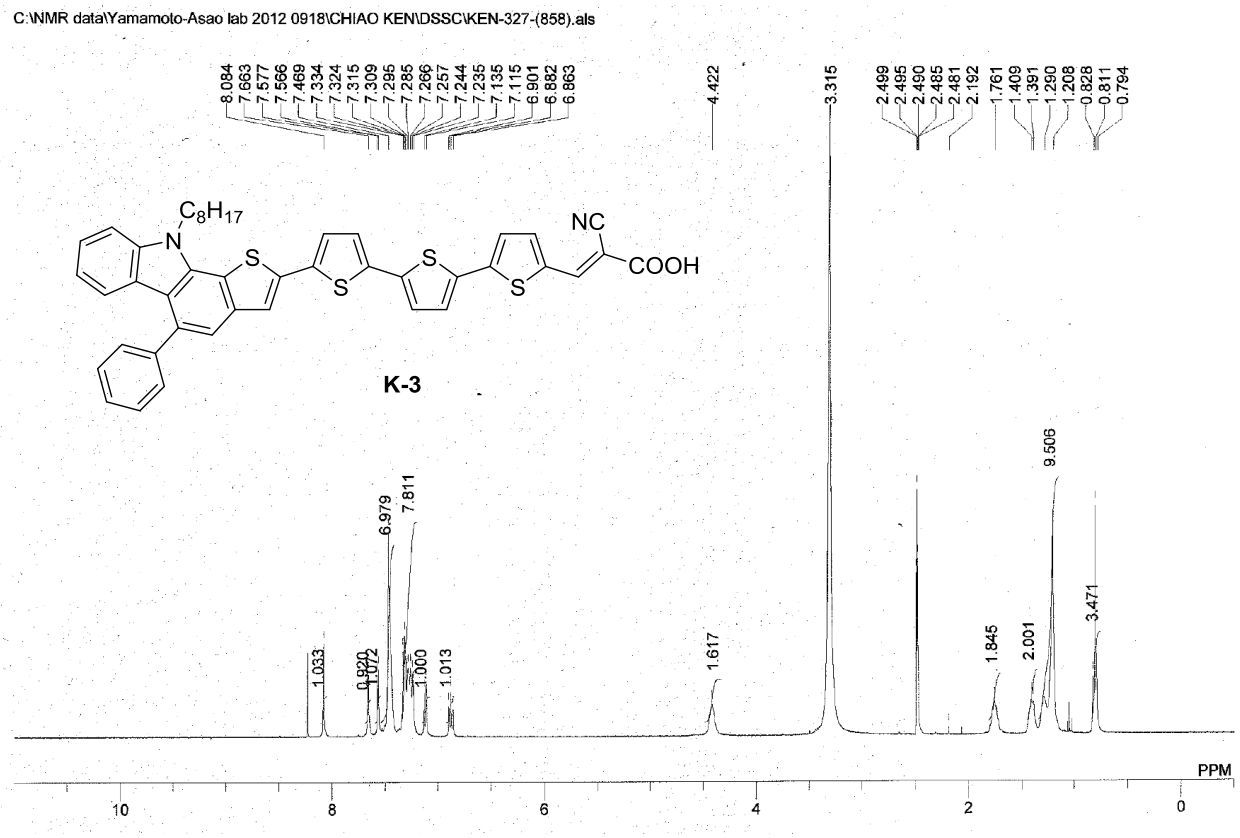
141.164
141.016
140.966
140.207
140.133
139.927
139.655
139.267
138.863
135.530
134.672
134.301
134.128
132.618
132.577
130.853
130.316
129.293
129.013
128.287
127.413
124.764
123.370
123.164
122.149
120.244
119.048
116.886
116.383
108.480
77.313
77.000
76.678
39.457
31.694
31.677
31.644
30.720
30.465
30.217
29.582
29.467
29.434
29.343
29.244
29.186
22.694
22.669
22.644
15.706
14.156
14.139
C6H13
C6H13
C6H13
N
S
S
S
S
O
3c
PPM
200
150
100
50
0


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN-327-(858).als
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7.577
7.566
7.469
7.334
7.324
7.315
7.309
7.296
7.285
7.266
7.257
7.244
7.235
7.135
7.115
6.901
6.882
6.863
4.422
3.315
2.499
2.495
2.490
2.485
2.481
2.192
1.761
1.409
1.391
1.290
1.208
0.828
0.811
0.794
C8H17
N
S
S
S
S
NC
COOH
K-3
9.506
6.979
7.811
3.471
1.033
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1.000
1.013
1.617
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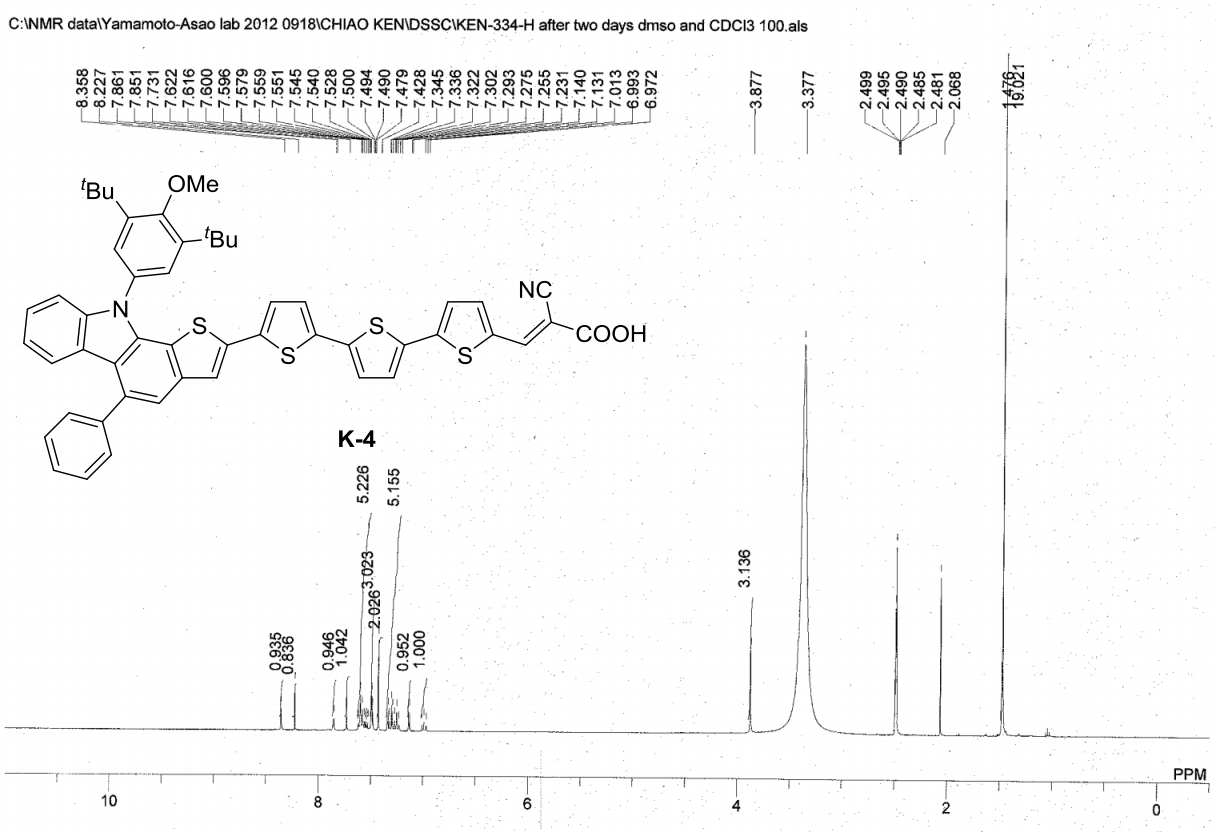
2.001
PPM
10
8
6
4
2
0


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN-334-H after two days dmso and CDCl3 100.als
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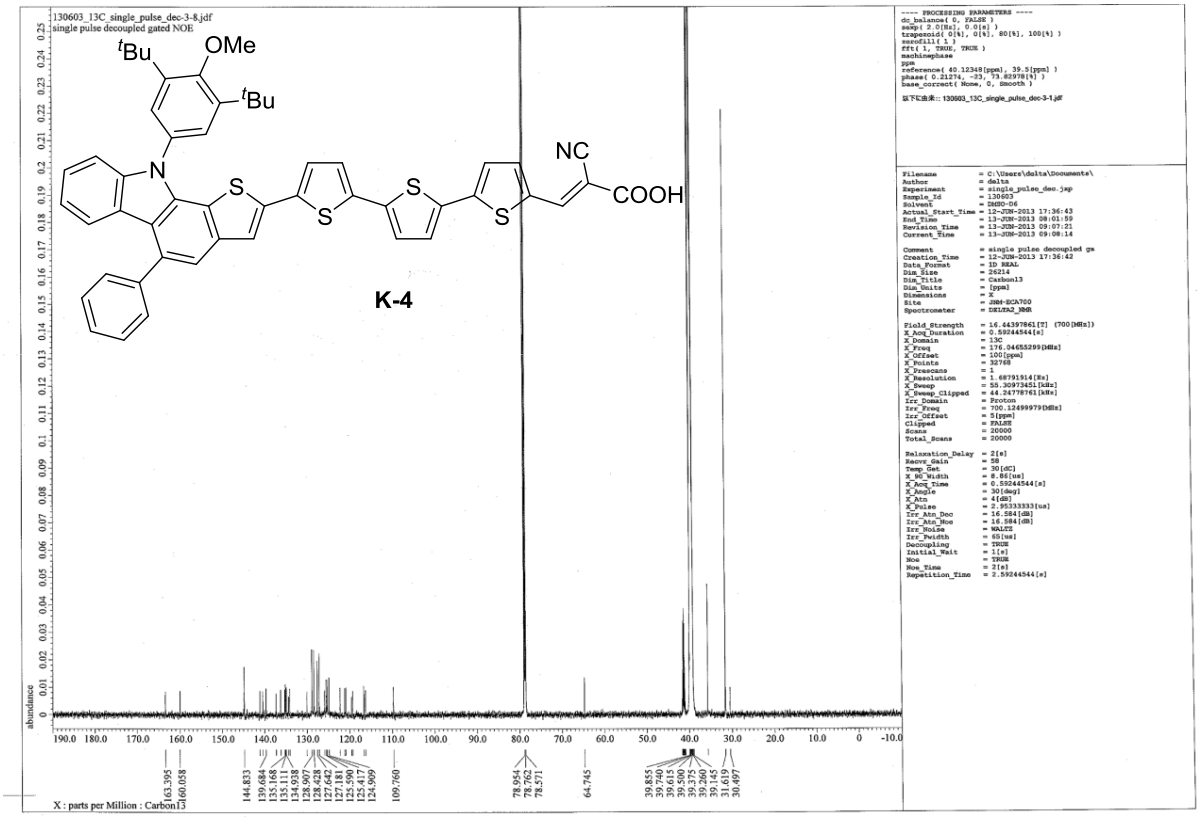
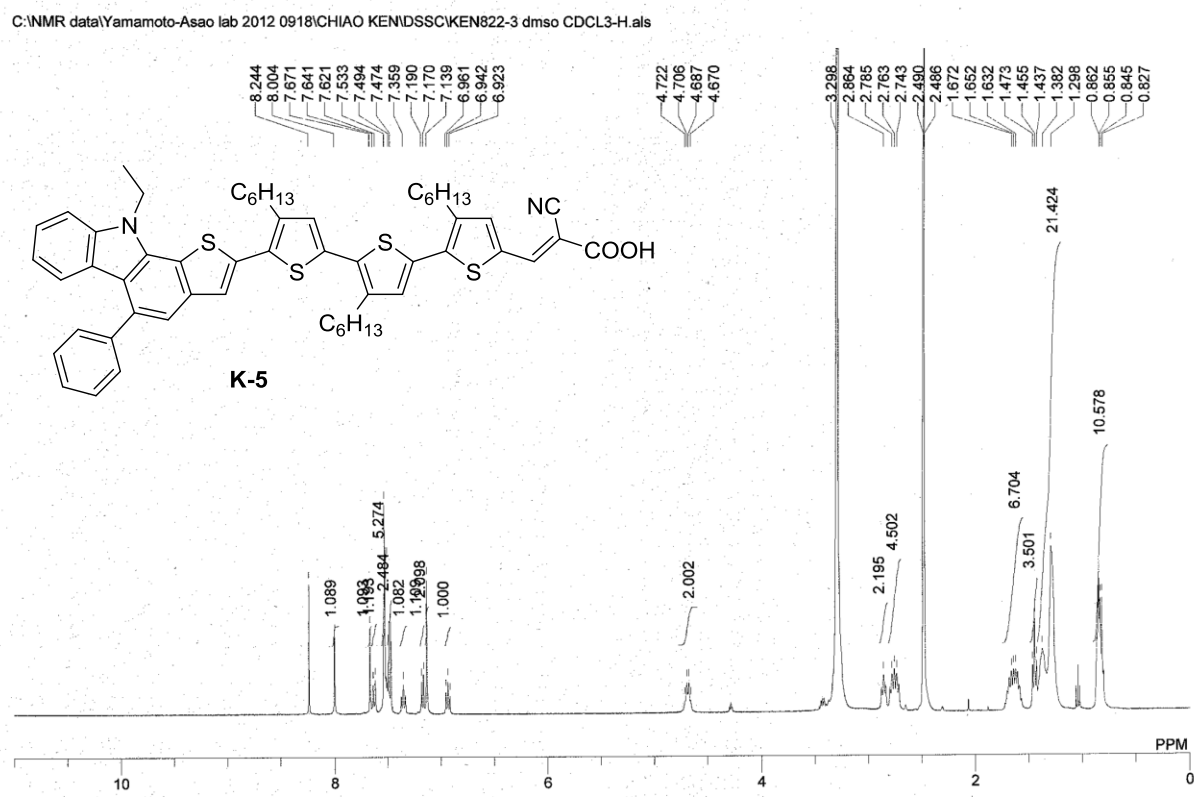
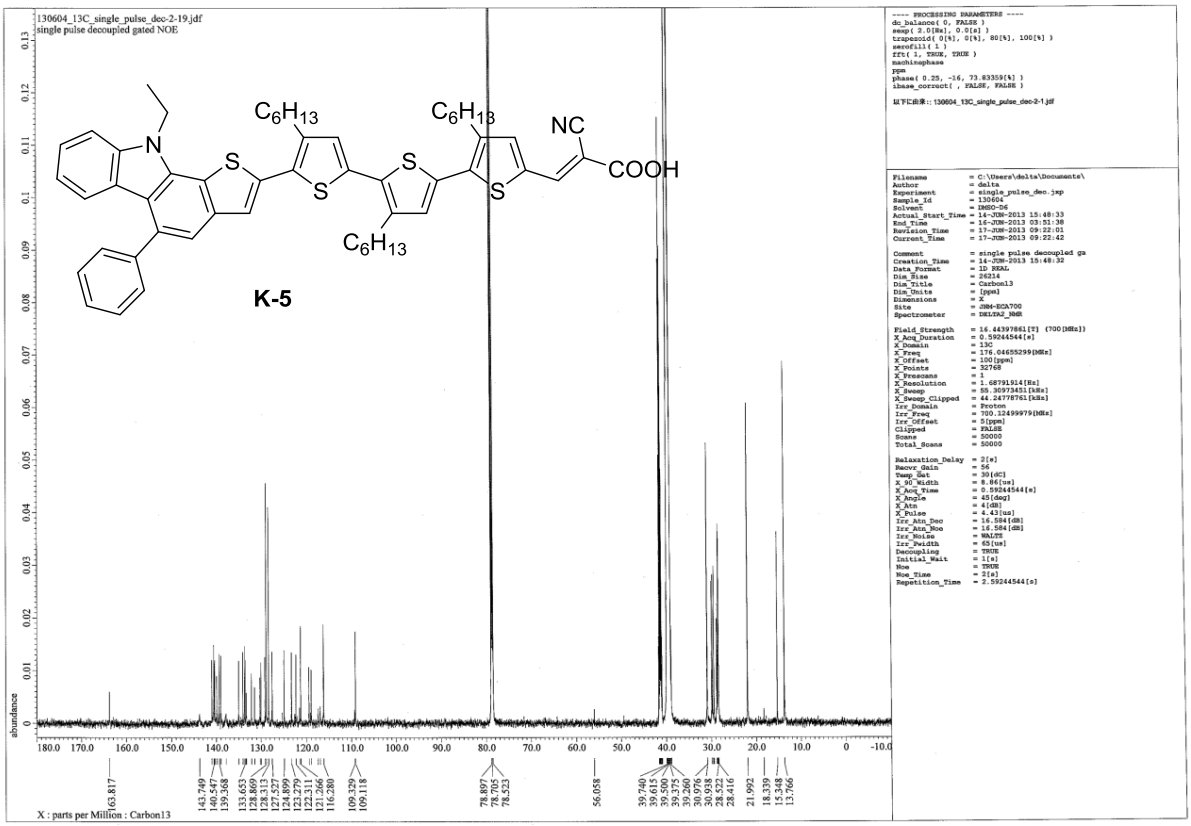
8.227
7.861
7.851
7.731
7.622
7.616
7.600
7.596
7.579
7.559
7.551
7.545
7.540
7.528
7.500
7.494
7.490
7.479
7.428
7.345
7.336
7.322
7.302
7.293
7.275
7.255
7.231
7.140
7.131
7.013
6.993
6.972
3.877
3.377
2.499
2.495
2.490
2.485
2.481
2.068
1.476
19.021
tBu
OMe
tBu
N
S
S
S
S
NC
COOH
K-4
5.226
5.155
3.023
2.026
3.136
0.935
0.836
0.946
1.042
0.952
1.000
PPM
10
8
6
4
2
0


130603_13C_single_pulse_dec-3-8.jdf
single pulse decoupled gated NOE
tBu
OMe
tBu
N
S
S
S
S
NC
COOH
K-4
abundance
X : parts per Million : Carbon13
163.395
160.058
144.833
139.684
136.145
135.111
134.938
128.907
128.428
127.647
127.181
125.590
125.417
124.909
109.760
78.954
78.762
78.571
64.745
39.855
39.740
39.615
39.500
39.375
39.260
39.145
31.619
30.497
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(40.12348[ppm], 39.5[ppm])
phase(0.21274, -23, 73.82978[%])
base_correct(None, 0, Smooth)
Filename = C:\Users\delta\Documents\
Author = delta
Experiment = single_pulse_dec.jxp
Sample_Id = 130603
Solvent = DMSO-D6
Actual_Start_Time = 12-JUN-2013 17:36:43
End_Time = 13-JUN-2013 08:01:59
Revision_Time = 13-JUN-2013 09:07:21
Current_Time = 13-JUN-2013 09:09:14
Comment = single pulse decoupled ga
Creation_Time = 12-JUN-2013 17:36:42
Data_Format = 1D REAL
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-GSX700
Spectrometer = DELTA2_NMR
Field_Strength = 16.44397861[T] (700[MHz])
X_Acq_Duration = 0.59244544[s]
X_Domain = 13C
X_Freq = 176.04655299[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 1.68791916[Hz]
X_Sweep = 55.30973451[kHz]
X_Sweep_Clipped = 44.24778761[kHz]
Irr_Domain = Proton
Irr_Freq = 700.12499979[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 20000
Total_Scans = 20000
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Set = 30[dC]
X_90_Width = 8.86[us]
X_Acq_Time = 0.59244544[s]
X_Angle = 30[deg]
X_Atn = 4[dB]
X_Pulse = 2.95333333[us]
Irr_Atn_Dec = 16.58[dB]
Irr_Atn_Noe = 16.58[dB]
Irr_Noise = WALTZ
Irr_Width = 65[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.59244544[s]


C:\NMR data\Yamamoto-Asao lab 2012 0918\CHIAO KEN\DSSC\KEN822-3 dmso CDCL3-H.als
K-5
PPM


130604_13C_single_pulse_dec-2-19.jdf
single pulse decoupled gated NOE
K-5
X : parts per Million : Carbon13