Supporting Information

Dithieno[3,2-b:2′,3′-d]pyran-Containing Organic D-π-A Sensitizers for Dye-Sensitized Solar Cells and the Effect of Dipole Moment

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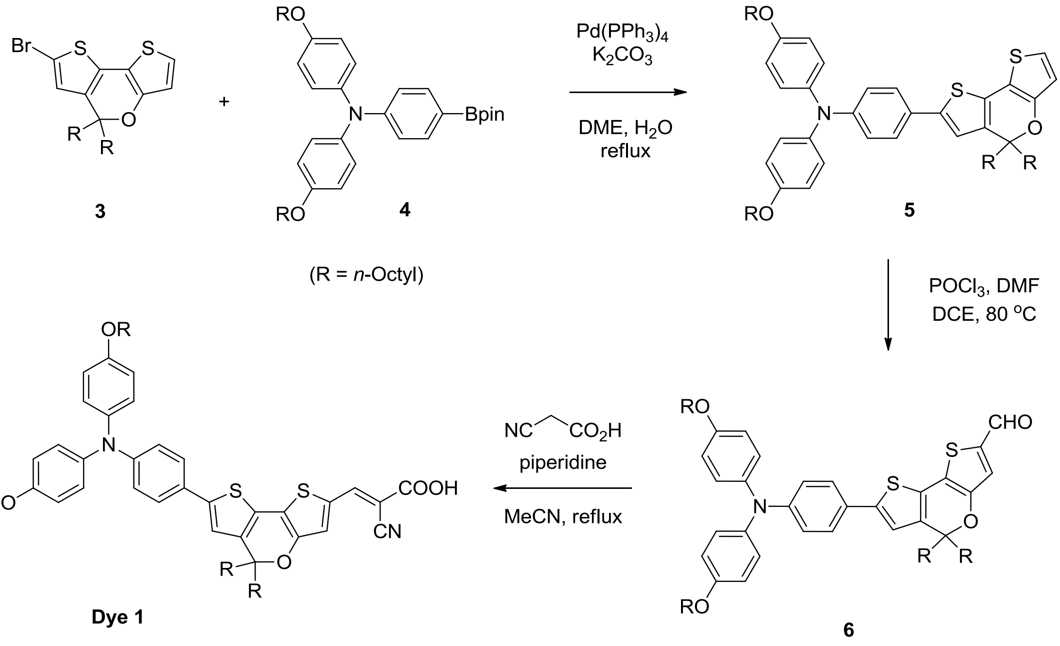
^{1}H NMR and ^{13}C NMR Spectra

I. General Information

All air or moisture sensitive reactions were conducted in oven-dried glassware under nitrogen atmosphere using dry solvents. Flash column chromatography was performed over silica gel (230-400 mesh) purchased from Qingdao Puke Co., China. Dichloromethane, tetrahydrofuran, diethyl ether, *N*,*N*-dimethylformamide (DMF), and acetonitrile were dried with a solvent purification system by Innovative Technology®. *tert*-Butanol was distilled before use. All other reagents were purchased and used as received without purification. ^{1}H and ^{13}C NMR spectra were collected on a Bruker AV 400 MHz NMR spectrometer using residue solvent peaks as an internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm, ^{13}C NMR: $CDCl_3$ at 77.0 ppm). Mass spectra were collected on an Agilent GC/MS 5975C system, or a MALDI Micro MX mass spectrometer, or an API QSTAR XL System.

II. Synthesis of Dyes 1 and 2

Compounds **3** and **4** are known compounds and their literature procedures were followed.[1]



Scheme S1. Synthesis of Dye 1.

4-(5,5-Dioctyl-5H-dithieno[3,2-b:2',3'-d]pyran-7-yl)-*N*,*N*-bis(4-(octyloxy)phenyl)aniline (5). Under N_2, a flask was charged with a mixture of the bromide **3** (436 mg, 0.88 mmol),[1] the boronic ester **4** (550 mg, 0.88 mmol), an aqueous solution of K_2CO_3 (1.0 M, 1.3 mL), $Pd(PPh_3)_4$ (101 mg, 0.088 mmol), and ethylene glycol (26 mL). The reaction mixture was heated to reflux for 18 hours and cooled to room temperature. Next, the solvent was removed under

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[1] a) K. Yoshimura, K. Ohya, T. Kato, US Patent, US 2012/0205644, **2012**; b) J. Li, X. Deng, Z. Zhang, Y. Wang, Y. Liu, K. He, Y. Huang, Q. Tao, L. Quan, W. Zhu, *J. Polym. Sci. Pol. Chem.* **2012**, *50*, 4686–4694.

reduced pressure. Water (30 mL) and DCM (50 mL) were added. The layers were separated, and the aqueous layer was extracted with DCM (2×50 mL). The organic layers were combined, dried with anhydrous Na_2SO_4, and concentrated. The residue was subjected to flash column chromatography (eluent: hexanes/DCM = 10/1 to 5/1) to afford the pure product **5** as red oil (509 mg, 63% yield).

^{1}HNMR (400MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.8 Hz, 2H), 7.07-7.04 (m, 4H), 6.95 (d, *J* = 5.2 Hz, 1H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.85-6.81 (m, 4H), 6.78 (s, 1H), 6.67 (d, *J* = 5.2 Hz, 2H), 3.94 (dd, J_1 = 6.5 Hz, J_2 = 6.6 Hz, 4H), 1.89-1.87 (m, 4H), 1.82-1.75 (m, 4H), 1.49-1.43 (m, 4H), 1.36-1.23 (m, 40H), 0.92-0.85 (m, 12H).

^{13}CNMR (100 MHz, $CDCl_3$) δ 155.5, 151.1, 148.1, 140.4, 140.1, 133.6, 120.6, 126.9, 126.5, 126.2, 125.8, 120.8, 120.6, 120.3, 119.0, 118.5, 117.5, 115.4, 115.2, 110.2, 86.2, 68.2, 40.0, 31.8, 31.8, 30.0, 29.5, 29.4, 29.34, 29.26, 29.2, 26.1, 23.8, 22.7, 22.6, 14.1.

LRMS (LD+) calcd. for $C_{59}H_{83}NO_3S_2$ [M^+]: 917.6; found: 917.7.

7-(4-(Bis(4-(octyloxy)phenyl)amino)phenyl)-5,5-dioctyl-5H-dithieno[3,2-b:2', 3'-d]pyran-2-carbaldehyde (6). To a solution of **5** (400 mg, 0.44 mmol) in DCE (7.2 mL) were added DMF (50 μL, 0.65 mmol) and $POCl_3$ (61 μL, 0.65 mmol). The mixture was heated to 80 °C for 18 hours and cooled to room temperature. A saturated aqueous NaOAc solution (2.4 mL) and dichloromethane (1.2 mL) were added. Then, the mixture was extracted with DCM (3×50 mL). The organic layers were combined, dried with Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (eluent: hexanes/EA = 50/1) to give pure product **6** as red oil (342 mg, 82% yield).

^{1}HNMR (400 MHz, $CDCl_3$) δ 9.74 (s, 1H), 7.36 (d, *J* = 8.7 Hz, 2H), 7.26 (d, *J* = 2.0 Hz, 1H), 7.06 (d, *J* = 8.9 Hz, 4H), 6.90 (d, *J* = 8.7 Hz, 2H), 6.83 (d, *J* = 8.9 Hz, 4H), 6.79 (s, 1H), 3.94 (dd, J_1 = 6.5 Hz, J_2 = 6.6 Hz, 4H), 1.88-1.86 (m, 4H), 1.81-1.74 (m, 4H), 1.58-1.42 (m, 4H), 1.35-1.22 (m, 40H), 0.91-0.83 (m, 12H).

^{13}CNMR (100 MHz, $CDCl_3$) δ 181.3, 155.9, 155.1, 149.2, 144.7, 140.2, 139.8, 137.7, 133.5, 132.6, 130.8, 128.8, 127.0, 126.0, 119.5, 115.3, 112.5, 86.2, 68.2, 40.0, 31.8, 29.8, 29.4, 29.34, 29.29, 29.21, 29.19, 26.0, 23.7, 22.62, 22.59, 14.07, 14.06.

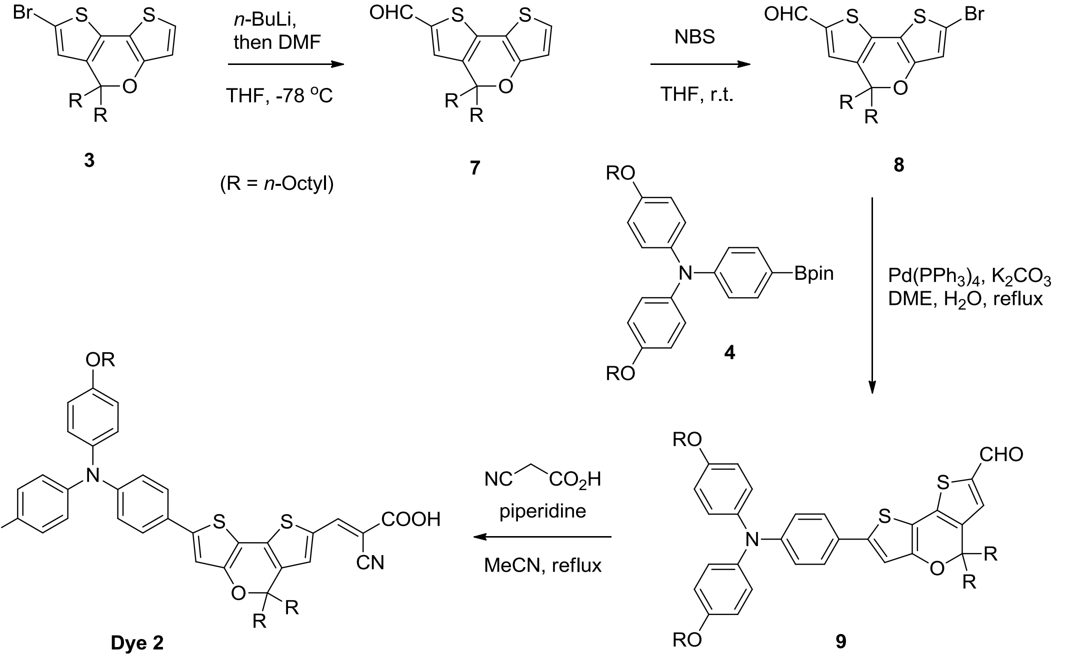
HRMS (LD+) calcd. for $C_{60}H_{83}NO_4S_2$ [M^+]: 945.5763; found: 945.5776.

3-(7-(4-(Bis(4-(octyloxy)phenyl)amino)phenyl)-5,5-dioctyl-5H-dithieno[3,2-b:2',3'-d]pyran-2-yl)-2-cyanoacrylic acid (Dye 1). A flask was charged with compound **6** (342 mg, 0.36 mmol), cyanoacetic acid (308 mg, 3.6 mmol), piperidine (0.1 mL, 1.1 mmol), and $CHCl_3$ (36 mL). The mixture was heated to reflux for 20 hours and cooled to room temperature. Water (30 mL) was added, and the layers were separated. The aqueous layer was extracted with DCM (2 × 50 mL). The combined organic layers were washed with an aqueous solution of HCl (1 M, 30 mL) and brine (30 mL), dried with Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (eluent: DCM/methanol = 10/1) to form the desired pure dye **1** (120 mg, 33% yield) as purple solid.

^{1}HNMR (400MHz, acetone-*d$_6$*) δ 8.31 (s, 1H), 7.61 (s, 1H), 7.54-7.49 (m, 2H), 7.33 (s, 1H), 7.08 (d, *J* = 6.4 Hz, 4H), 6.93 (d, *J* = 7.9 Hz, 4H), 6.86 (d, *J* = 7.5 Hz, 2H), 3.99 (dd, J_1 = 6.2 Hz, J_2 = 6.1 Hz, 4H), 1.77-1.81 (m, 4H), 1.46-1.50 (m, 4H), 1.23-1.36 (m, 44H), 0.88-0.90 (m, 6H), 0.82-0.86 (m, 6H).

^{13}CNMR (100 MHz, acetone-*d$_6$*) δ 172.0, 156.4, 156.2, 145.4, 139.9, 139.7, 133.0, 130.7, 129.5, 127.4, 127.1, 126.3, 126.1, 119.9, 119.3, 118.7, 115.5, 115.4, 112.7, 87.4, 86.8, 67.9, 40.5, 40.1, 31.7, 31.6, 25.9, 23.6, 22.42, 22.39, 13.46.

HRMS (LD+) calcd. for $C_{63}H_{84}N_2O_5S_2$ [M$^+$]: 1012.5822; found: 1012.5909.



Scheme S2. Synthesis of Dye 2.

5,5-Dioctyl-5H-dithieno[3,2-b:2',3'-d]pyran-7-carbaldehyde (7). At -78 °C, to a solution of bromide **3** (1.5 g, 3.0 mmol) in anhydrous THF (30 mL) was added *n*-BuLi (1.25 mL, 2.4 M in hexane, 3.0 mmol) dropwise. The reaction mixture was stirred at -78 °C for 35 min, and anhydrous DMF (1.2 mL, 15 mmol) was added. The reaction mixture was stirred for 15 min, then warmed to room temperature and kept stirring for 1 h. Water (50 mL) was added and the mixture was extracted with DCM (3×100 mL). The combined organic layers were washed with water (100 mL) and brine (100 mL), dried over Na_2SO_4, and concentrated. The residue was subjected to flash column chromatography (eluent: hexane/ethyl acetate = 50/1) to afford the desired product **7** as red oil (1.2 g, 90% yield).

^{1}HNMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 7.33 (s, 1H), 7.18 (d, *J* = 5.3 Hz, 1H), 6.68 (d, *J* = 5.3 Hz, 1H), 1.95-1.81 (m, 4H), 1.38-1.22 (m, 24H), 0.85 (dd, J_1 = 6.7 Hz, J_2 = 7.0 Hz, 6H).

^{13}CNMR (100 MHz, $CDCl_3$) δ 181.7, 154.6, 139.8, 138.5, 133.5, 133.2, 125.3, 118.7, 109.3, 86.3, 40.0, 31.8, 29.8, 29.4, 29.2, 23.7, 22.6, 14.1.

HRMS (LD+) calcd. for $C_{26}H_{38}O_2S_2$ [M$^+$]: 446.2313; found: 446.2335.

2-Bromo-5,5-dioctyl-5H-dithieno[3,2-b:2',3'-d]pyran-7-carbaldehyde (8). At room temperature, to a solution of compound **7** (688 mg, 1.54 mmol) in THF (15 mL) was added NBS (411 mg, 2.3 mmol). The reaction mixture was stirred overnight and quenched with water (15 mL). The mixture was extracted with

DCM (3×50 mL). The organic layers were combined, washed with water (30 mL) and brine (30 mL), dried with Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (eluent: hexanes/ethyl acetate = 50/1) to give the desired product **8** as yellow crystal (700 mg, 86% yield).

^{1}HNMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 7.31 (s, 1H), 6.69 (s, 1H), 1.90-1.84 (m, 4H), 1.35-1.21 (m, 24H), 0.86-0.83 (m, 6H).

^{13}CNMR (100 MHz, $CDCl_3$) δ 181.5, 153.3, 138.8, 138.2, 133.3, 133.0, 121.9, 113.9, 110.8, 86.8, 39.9, 31.7, 29.7, 29.4, 29.2, 23.6, 22.6, 14.0.

HRMS (LD+) calcd. for $C_{26}H_{37}BrO_2S_2$ [M^+]: 524.1418; found: 524.1417.

2-(4-(Bis(4-(octyloxy)phenyl)amino)phenyl)-5,5-dioctyl-5H-dithieno[3,2-b:2',3'-d]pyran-7-carbaldehyde (9). A flask was charged with bromide **8** (1.0 g, 1.91 mmol), boronic ester **4** (1.2 g, 1.91 mmol), $Pd(PPh_3)_4$ (220 mg, 0.19 mmol), an aqueous solution of K_2CO_3 (4 mL, 1 M), and DME (40 mL). The mixture was refluxed for 24 hours, then cooled to room temperature and concentrated under reduced pressure. Water (30 mL) was added, and the mixture was extracted with DCM (3×50 mL). The organic layers were combined, dried with Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (hexanes/ethyl acetate = 20/1) to afford the desired product **9** as red oil (420 mg, 24% yield).

^{1}HNMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 7.35 (d, *J* = 8.2 Hz, 2H), 7.32 (s, 1H), 7.06 (d, *J* = 7.3 Hz, 4H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 4H), 6.78 (s, 1H), 3.94 (dd, J_1 = 6.0 Hz, J_2 = 6.2 Hz, 4H), 1.93-1.88 (m, 4H), 1.82-1.75 (m, 4H), 1.47-1.43 (m, 4H), 1.34-1.24 (m, 40H), 0.92-0.85 (m, 12H).

^{13}CNMR (100 MHz, $CDCl_3$) δ 181.3, 155.9, 155.1, 149.2, 144.7, 140.2, 139.9, 137.7, 133.5, 132.6, 130.8, 128.8, 127.0, 126.0, 119.5, 115.3, 112.5, 86.2, 68.2, 40.0, 31.8, 29.8, 29.7, 29.4, 29.34, 29.29, 29.21, 29.19, 26.0, 23.7, 22.62, 22.59, 14.07, 14.06.

HRMS (LD+) calcd. for $C_{60}H_{83}NO_4S_2$ $[M]^+$: 945.5764; found: 945.5742.

3-(2-(4-(Bis(4-(octyloxy)phenyl)amino)phenyl)-5,5-dioctyl-5H-dithieno[3,2-b:2',3'-d]pyran-7-yl)-2-cyanoacrylic acid (Dye 2). A flask was charged with compound **9** (420 mg, 0.44 mmol), cyanoacetic acid (374 mg, 4.4 mmol), piperidine (0.13 mL, 1.32 mmol), and $CHCl_3$ (20 mL). The mixture was heated to reflux for 24 hours and cooled to room temperature. Water (20 mL) was added, and the layers were separated. The aqueous layer was extracted with DCM (3×50 mL). Next, the organic layer was then washed with an aqueous solution of HCl (1 M, 30 mL) and brine (30 mL), dried with Na_2SO_4, and concentrated. The residue was purified by flash column chromatography (eluent: DCM/methanol = 10/1) to form the desired pure dye **2** (170 mg, 38% yield) as purple solid.

^{1}HNMR (400 MHz, CD_2Cl_2) δ 8.31 (s, 1H), 7.38 (s, 1H), 7.32 (d, J = 7.8Hz, 2H), 7.08 (d, J = 8.4 Hz, 4H), 6.88-6.86 (m, 4H), 6.86-6.84 (m, 2H), 6.75 (s, 1H), 3.97 (dd, J_1 = 6.4 Hz, J_2 = 6.5 Hz, 4H), 1.97-1.95 (m, 4H), 1.83-1.7 (m, 4H), 1.50-1.46 (m, 4H), 1.38-1.26 (m, 40H), 0.95-0.86 (m, 12H).

^{13}CNMR (100 MHz, CD_2Cl_2) δ 169.2, 156.5, 156.2, 149.6, 146.6, 142.8, 139.7, 137.1, 133.9, 133.5, 130.6, 127.2, 126.0, 124.3, 119.0, 116.6, 115.3, 112.3, 107.7, 86.8, 68.3, 40.3, 31.8, 29.8, 29.5, 29.4, 29.32, 29.27, 26.1, 23.7, 22.67, 22.65, 13.9.

HRMS (LD+) calcd. for $C_{63}H_{84}N_2O_5S_2$ [M^+]: 1012.5822; found: 1012.5918.

III. Optical and Cyclic Voltammetry Measurement

UV-vis absorptions in solution were measured in 1 cm cuvettes with Milton Roy 3000 spectrometer at room temperature. UV-vis spectra of dyes loaded on TiO_2 film were recorded in a Perkin Elmer Lambda 20 spectrometer at room temperature. Photoluminescence (PL) spectra was recorded in a Perkin Elmer LS 55 luminescence spectrometer.

Cyclic voltammetry was carried out on a CH Instruments Electrochemical Analyzer at a scan rate of 100 mV s^{-1}. The oxidation potential of the sensitizers was measured in DMF with $TBAPF_6$ (0.1 M) as electrolyte (working electrode: glassy carbon; reference electrode: Ag/Ag^+; calibrated with Fc/Fc^+ as an internal reference and converted to NHE by addition of 630 mV; counter electrode: Pt).

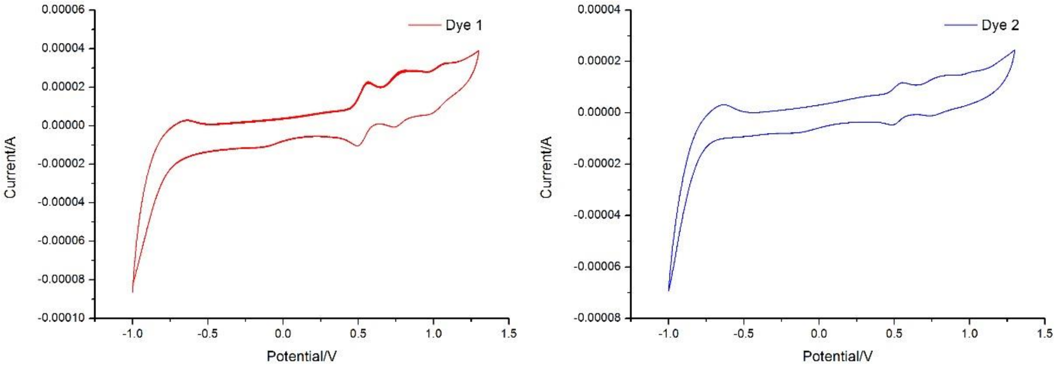


Figure S1. Cyclic voltammogram of dyes **1** (left) and **2** (right) in DMF solution with ferrocene as the internal standard.

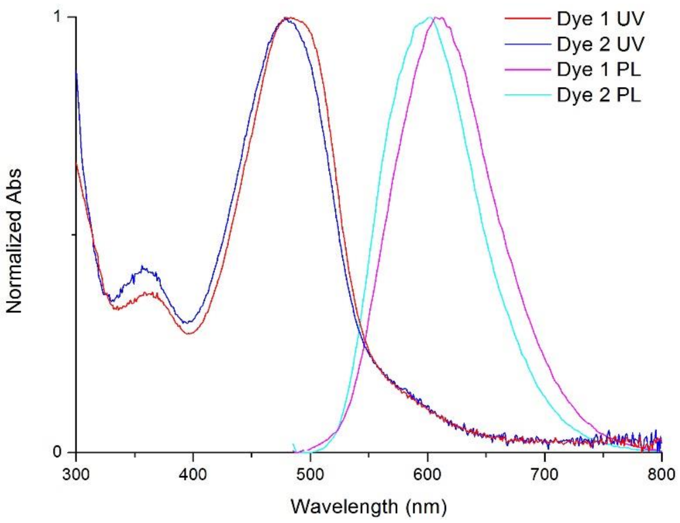


Figure S2. Normalized absorption and PL spectra of dyes **1** and **2**. E_{0-0} was determined by the onset wavelength in DMF solution.

IV. Solar Cell Fabrication and Characterization

TiO_2 films (12 μm) composed of 6 μm nanoparticle (20 nm) layer in direct contact with the FTO substrate and 6 μm light scattering particle [20 nm TiO_2 (80%) + 100 nm TiO_2 (20%)] layer were fabricated with a screen printing method and used in this study.[2] The films were sintered at 500 °C for 2 h to achieve good necking of neighboring TiO_2 particles. The sintered films were then treated with $TiCl_4$ aqueous solution (0.05 M) at 70 °C for 30 min followed by calcinations at 450 °C for 30 min. When TiO_2 electrodes were cooled down at around 120 °C, the electrodes were dipped in dye solutions (0.3 mM in toluene) for 24 h at room temperature for complete dye adsorption. The dye-loaded TiO_2 film as working electrode and the Pt-coated FTO as counter electrode were separated by a hot-melt Surlyn film (30 μm) and sealed together by pressing them under heating. The redox electrolyte [0.6 M 1,2-dimethyl-3-n-propylimidazolium iodide (DMPImI), 0.1 M LiI, 0.05 M I_2, and 0.5 M 4-tertbutylpyridine (TBP) in acetonitrile] was introduced through a hole in the counter electrode via suction through another drilled hole. Finally, the two holes were sealed using additional hot melt Surlyn film covered with a thin glass slide.

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[2] Z.-S. Wang, H. Kawauchi, T. Kashima, H. Arakawa, *Coord. Chem. Rev.* **2004**, *248*, 1381–1389.

The working performance of DSSCs was tested by recording the current density–voltage (J–V) curves with a Keithley 2400 source meter (Oriel) under illumination of simulated AM1.5G solar light coming from a solar simulator (Oriel-94043A equipped with a 450 W Xe lamp and an AM1.5G filter). The light intensity was calibrated using a standard Si solar cell (Newport 91150). A black mask with aperture area of 0.2304 cm^2 was used during measurement to avoid stray light. Charge extraction analysis was carried out on an electrochemical workstation (Zahner Zennium), which includes a green light emitting diode (LED, 555 nm) and the corresponding control system.

V. DFT Calculations

The molecular geometries were fully optimized using the B3LYP functionals and the 6-31G(d) basis set.[3] On these structures, the electronic spectra were computed using the TD-DFT approach[4] and the CAM-B3LYP functionals,[5] which have been shown to provide reliable results for CT states.[6] The obtained vertical transitions were then convoluted using Gaussian functions, so to simulate the experimental UV-vis spectra.[7] All the DFT and TD-DFT calculations were carried out using the Gaussian 09 program.[8]

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[3] a) C. T. Lee, W. T. Yang, R. G. Parr, *Phys. Rev. B* **1988**, *37*, 785–789; b) A. D. Becke, *Phys. Rev. A* **1988**, *38*, 3098–3100; c) B. Miehlich, A. Savin, H. Stoll, H. Preuss, *Chem. Phys. Lett.* **1989**, *157*, 200–206; d) A. D. Becke, *J. Chem. Phys.* **1993**, *98*, 5648–5652; e) P. J. Stephens, F. J. Devlin, C. F. Chabalowski, M. J. Frisch, *J. Phys. Chem.* **1994**, *98*, 11623–11627.

[4] C. Adamo, D. Jacquemin, *Chem. Soc. Rev.* **2013**, *42*, 845–856.

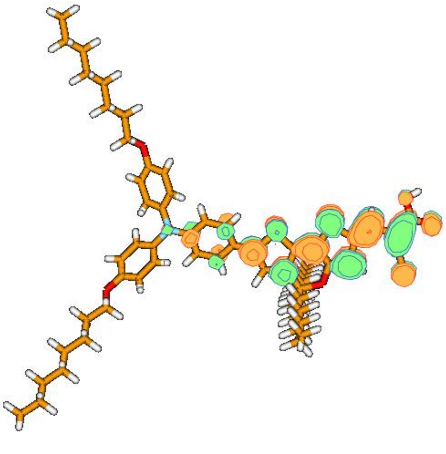
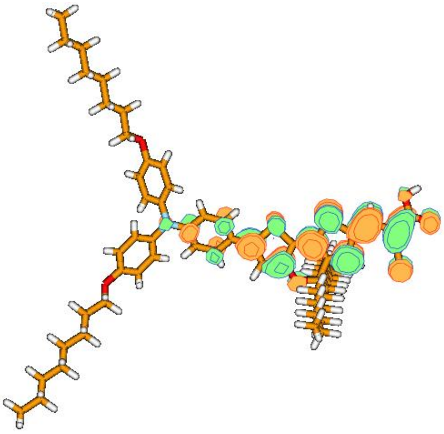
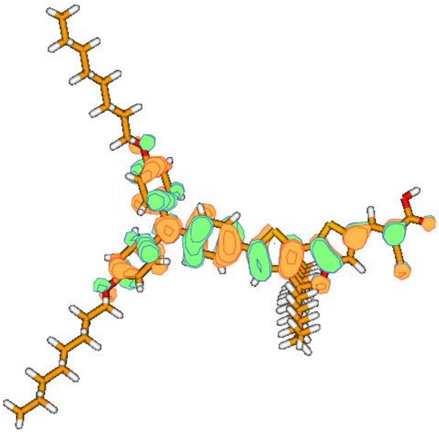
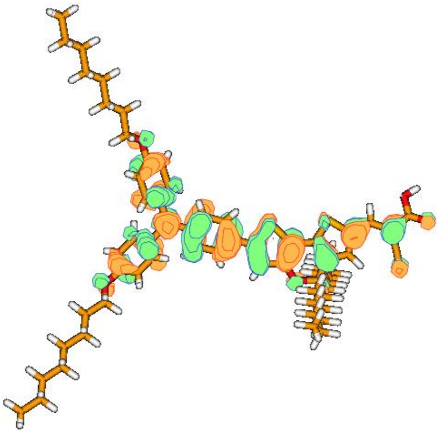
[5] T. Yanai, D. P. Tew, N. C. Handy, *Chem. Phys. Lett.* **2004**, *393*, 51–57.

[6] D. Jacquemin, E.A. Perpéte, G. Scalmani, R. Kobayashi, M.J. Frisch, C. Adamo, *J. Chem. Phys.* **2007**, *126*, 144105.

[7] E.A. Brémond, J. Kieffer, C. Adamo, *THEOCHEM* **2010**, *954*, 52–56.

[8] Gaussian 09, Revision A.1, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, Ö. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.

Table S1. HOMOs and LUMOs calculated for dyes **1** and **2**.

	Dye 1	**Dye 2**
HOMO-LUMO gap	2.18 eV	2.27 eV
LUMO	 -2.57 eV	 -2.49 eV
HOMO	 -4.75 eV	 -4.76 eV

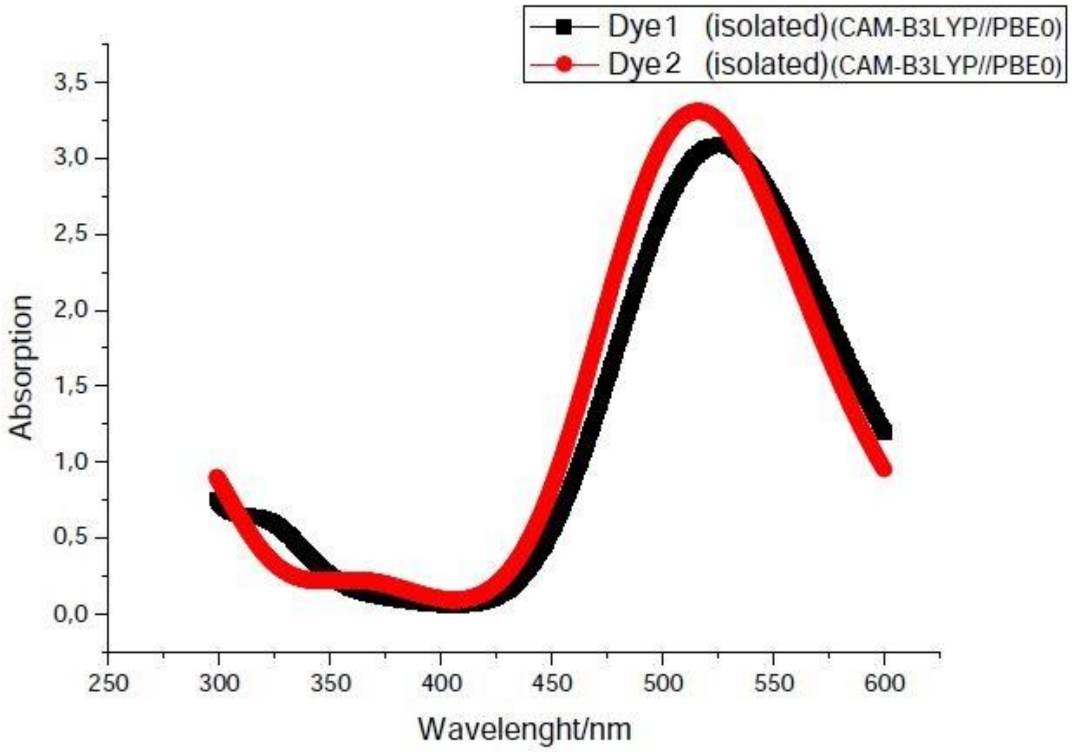
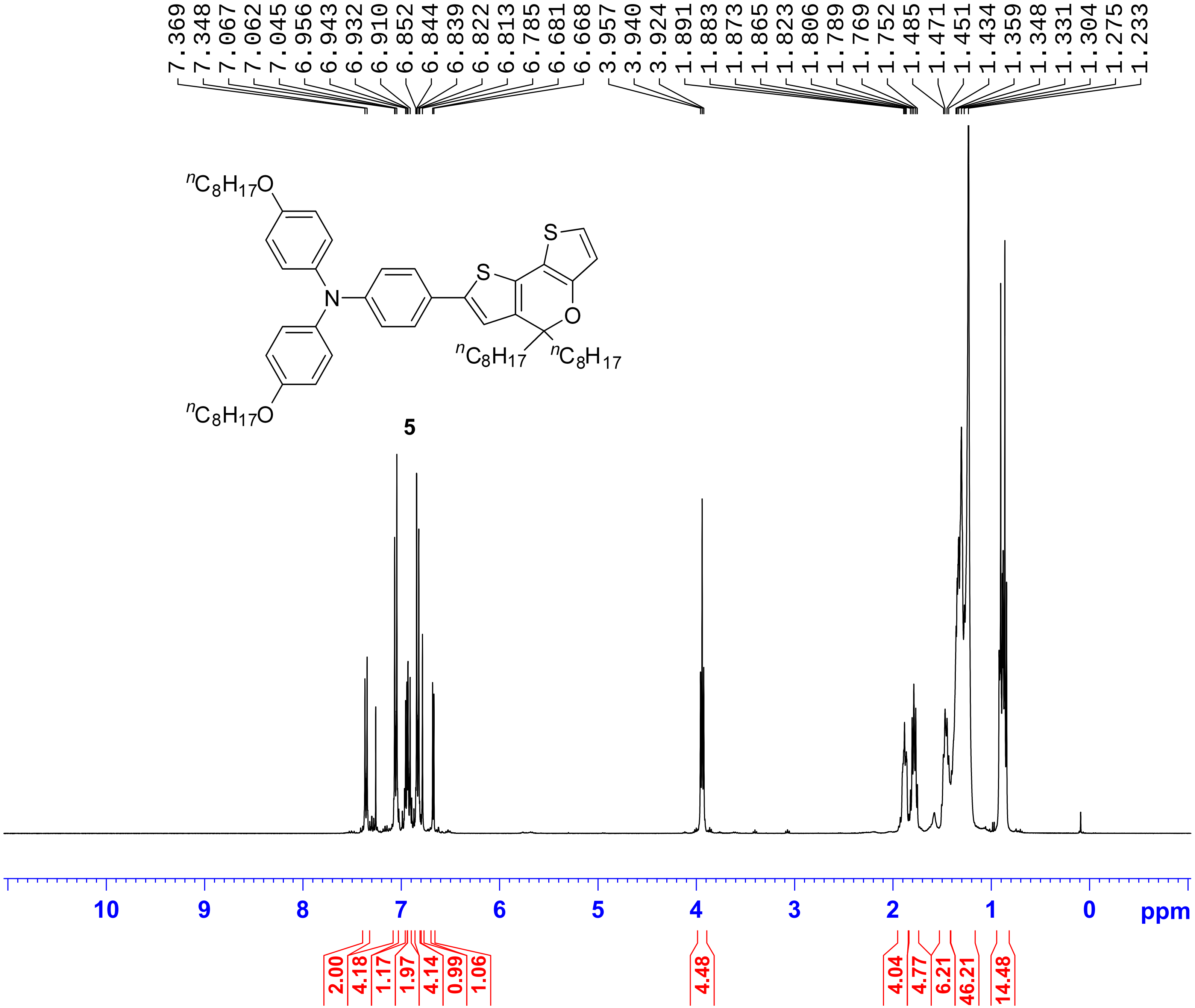


Figure S3. UV absorption spectra of dyes **1** and **2** with the PCM-CAM-B3LYP/6-31+G(d,p)//PBE0/6-31+G(d,p) level. The solvent used in the PCE is the THF.

Table S2. Electronic transition of dyes **1** and **2** with the geometrical parameters determined with the PBE0 functional and 6-31+g(d,p) basis set.

Dye1			**Dye 2**		
λ	f	Transition	λ	f	Transition
526	1.64	HOMO>LUMO	516	1.76	HOMO>LUMO
324	0.24	HOMO>LUMO+1, HOMO>LUMO	366	0.11	HOMO-1>LUMO, HOMO>LUMO
284	0.23	HOMO-1>LUMO+1,	298	0.28	HOMO-4>LUMO,

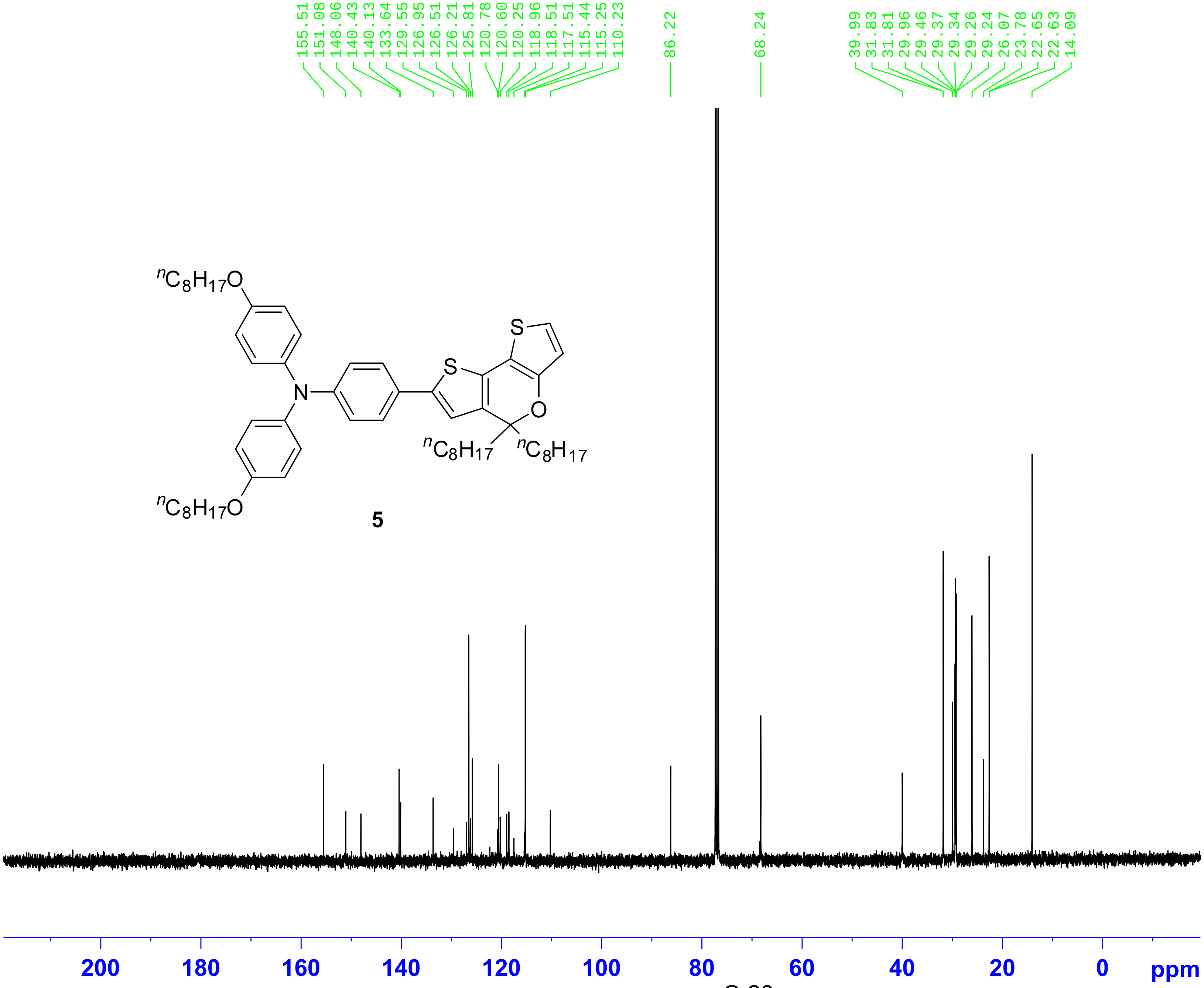
		HOMO-3>LUMO			HOMO>LUMO+1, HOMO-1>LUMO+1
279	0.16	HOMO>LUMO+6, HOMO>LUMO+5, HOMO-1>LUMO+6	285	0.12	HOMO-1>LUMO+1, HOMO>LUMO+4, HOMO>LUMO+5
275	0.14	HOMO>LUMO+5, HOMO>LUMO+3, HOMO>LUMO+6	276	0.18	HOMO>LUMO+6, HOMO-8>LUMO, HOMO>LUMO+3



NAME	bc2190	
EXPNO	1	
PROCNO	1	
Date_	20130411	
Time	21.10	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	8	
DS	0	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894966	sec
RG	31.55	
DW	62.400	usec
DE	6.50	usec
TE	295.8	K
D1	1.00000000	sec
TD0	1	

======== CHANNEL f1 ========

SFO1	400.1324710	MHz
NUC1	1H	
P1	14.30	usec
SI	65536	
SF	400.1300097	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

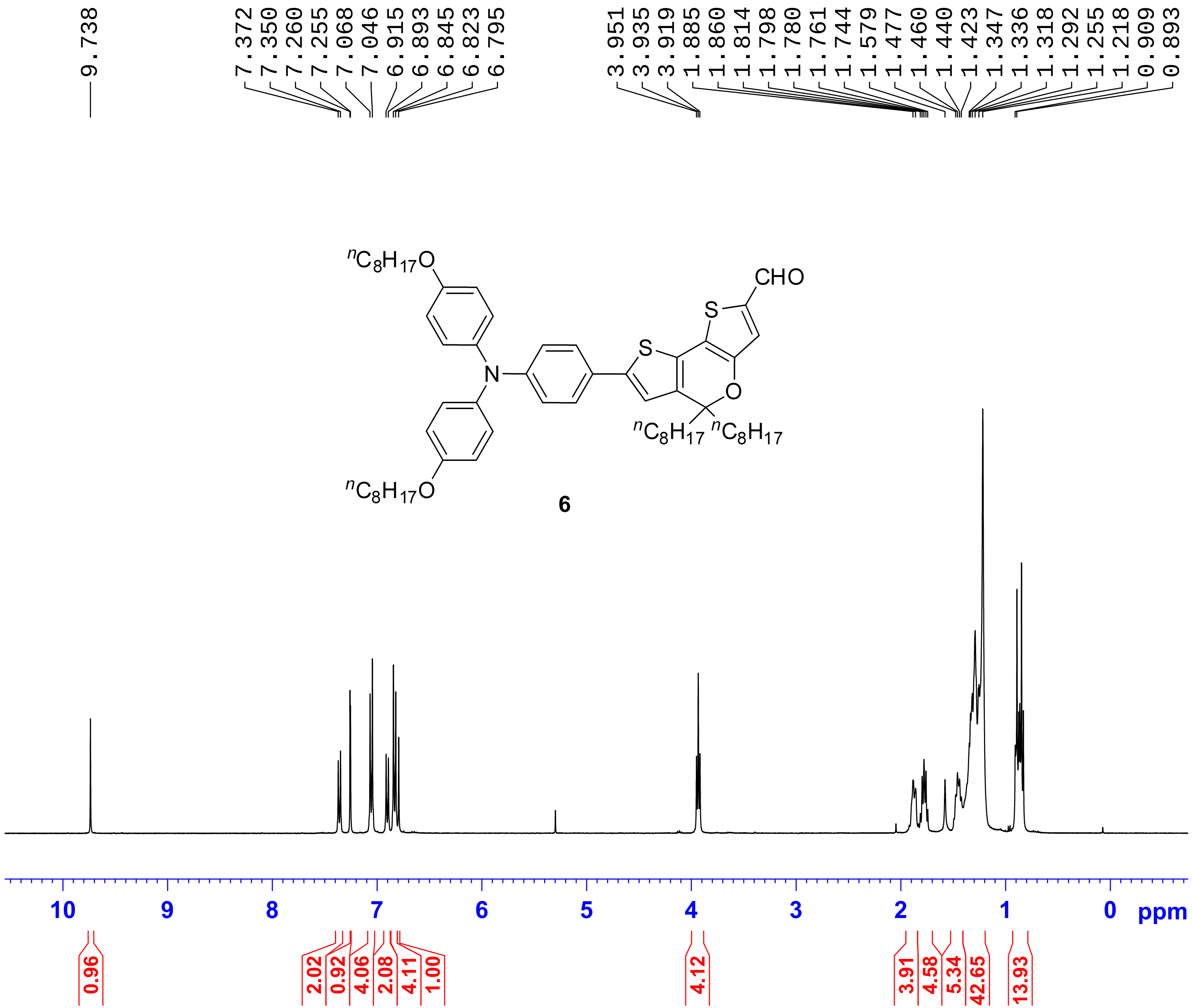


5

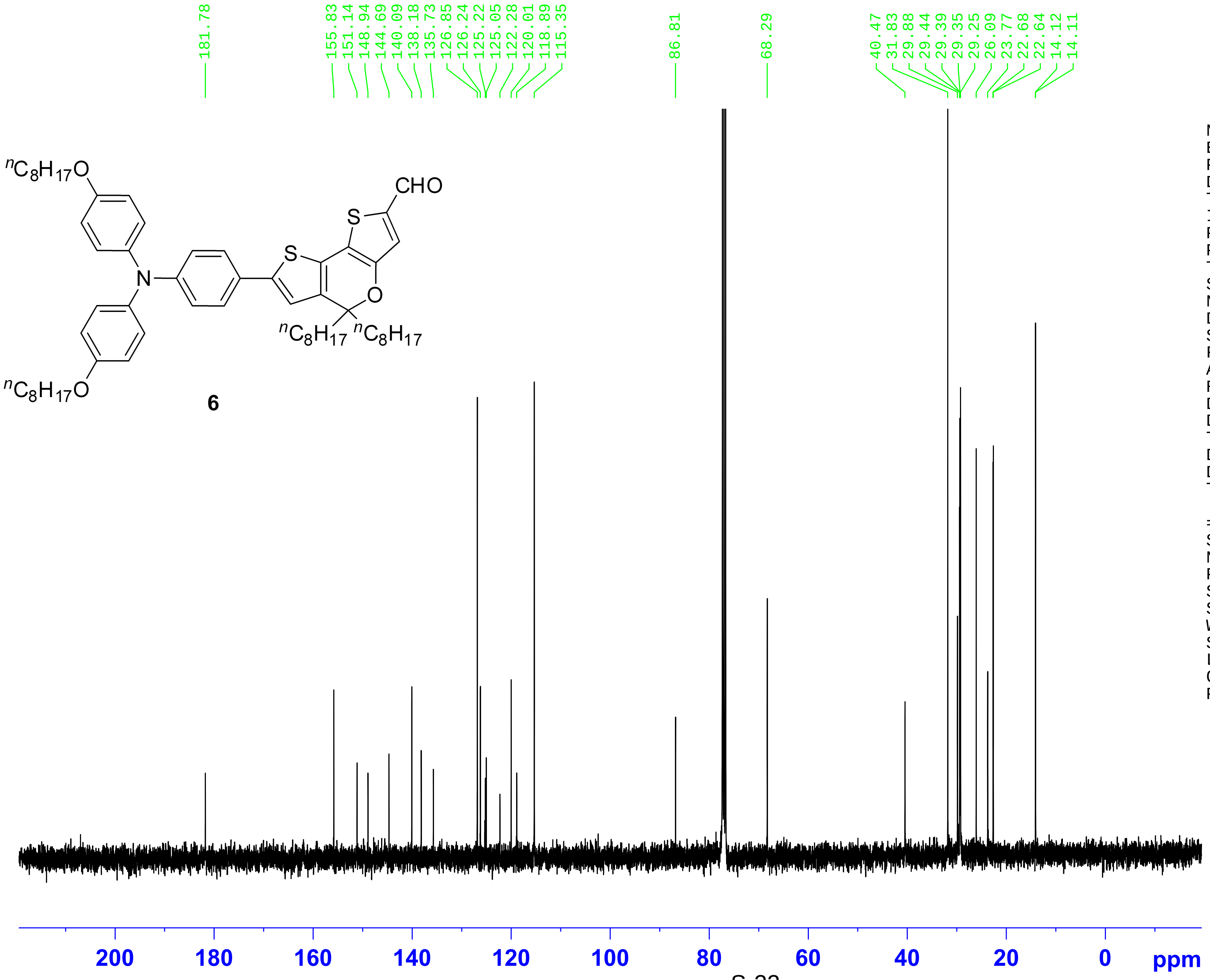
NAME	bc2190
EXPNO	2
PROCNO	1
Date_	20130411
Time	21.19
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	204
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	196.92
DW	20.800 usec
DE	6.50 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

SFO1	100.6228298 MHz
NUC1	13C
P1	9.60 usec
SI	32768
SF	100.6127737 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

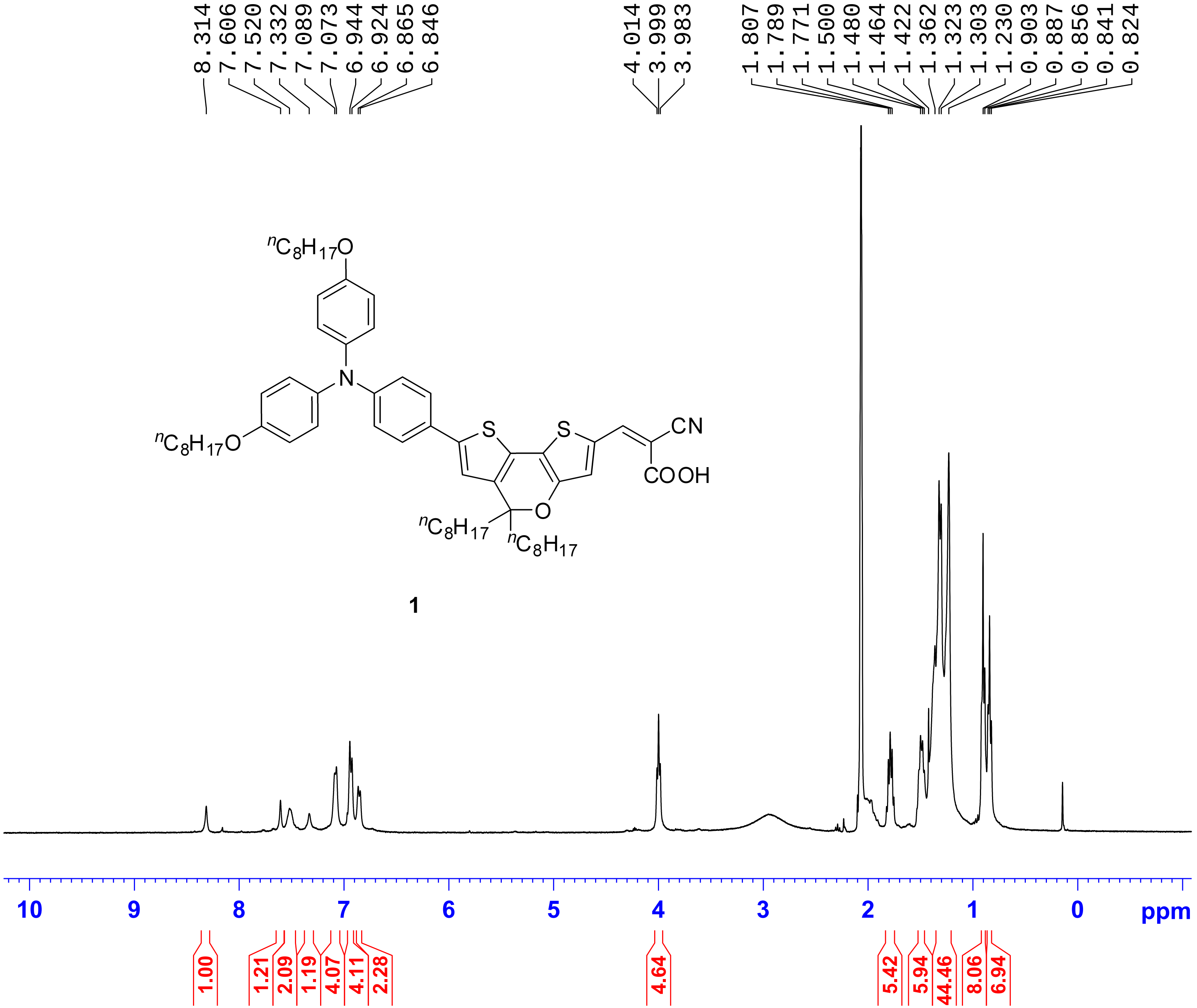


```
NAME              bc2191
EXPNO                  2
PROCNO                 1
Date_           20130417
Time               14.17
INSTRUM            spect
PROBHD    5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     3
DS                     0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894966 sec
RG                 54.81
DW               62.400 usec
DE                 6.50 usec
TE                295.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                  1H
P1                14.30 usec
SI                 65536
SF          400.1300097 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



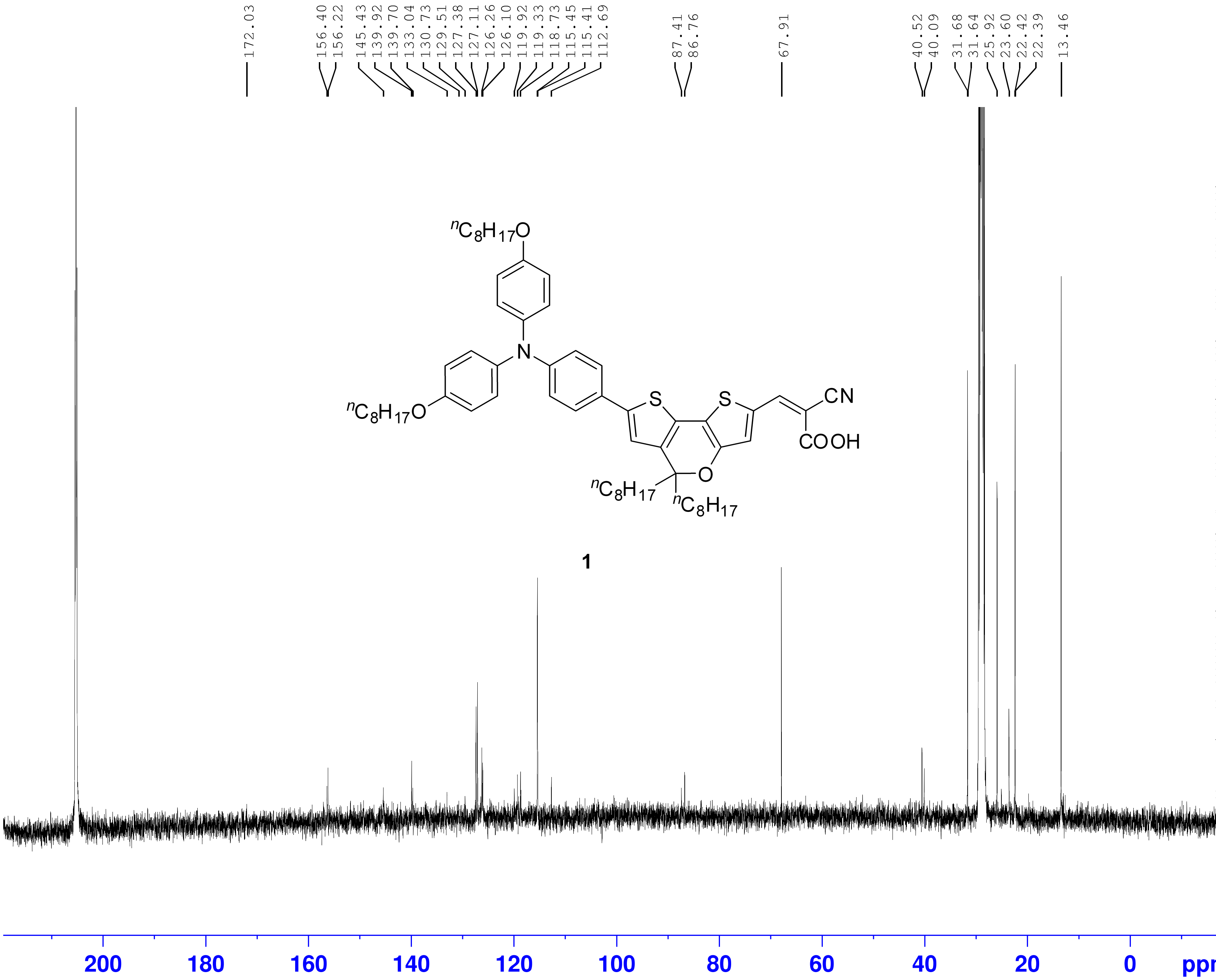
6

```
NAME               bc2191
EXPNO                   4
PROCNO                  1
Date_            20130417
Time                19.49
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   916
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                196.92
DW                20.800 usec
DE                  6.50 usec
TE                 296.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.60 usec
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



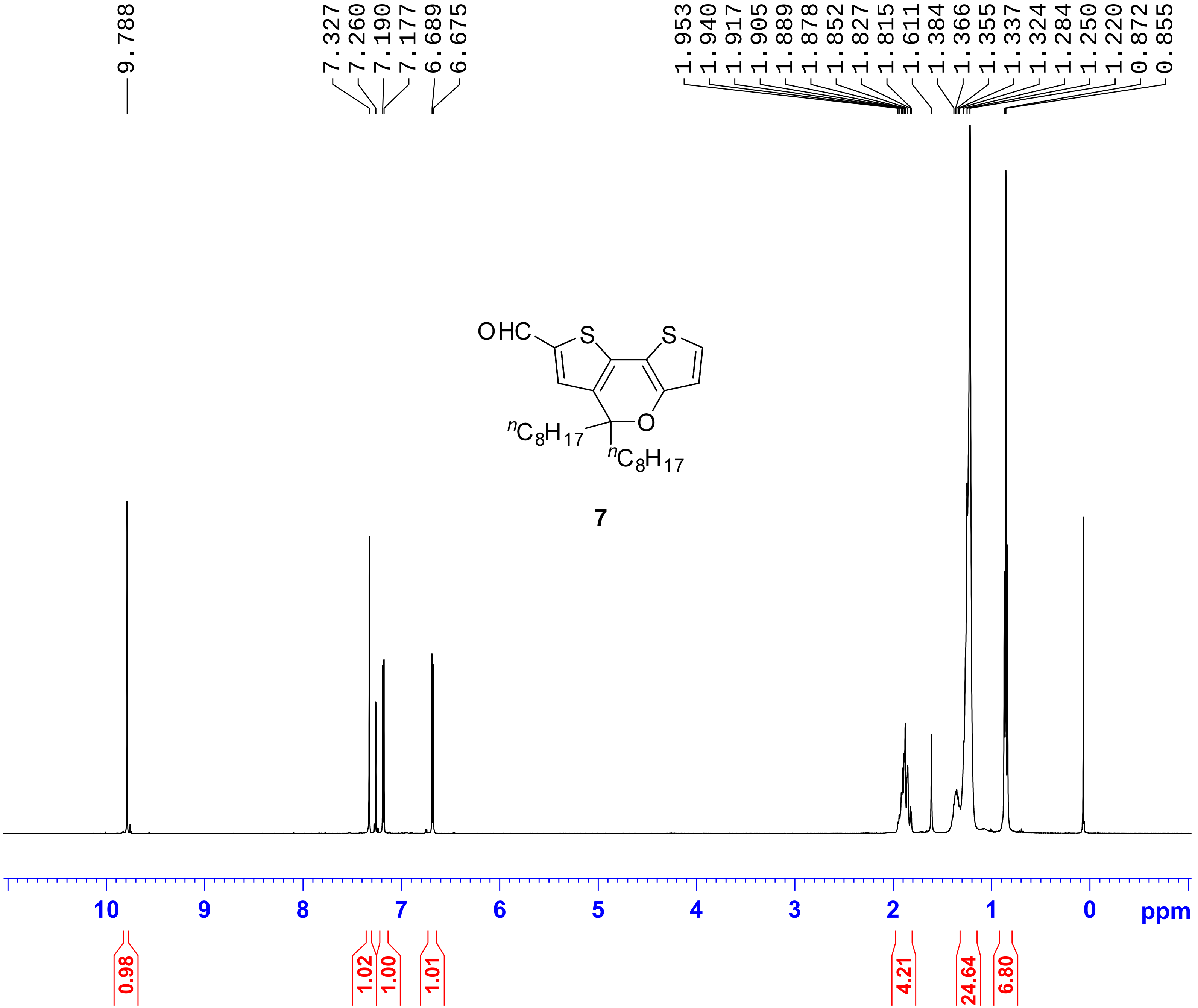
1

```
NAME             bc-dye28-F
EXPNO                     1
PROCNO                    1
Date_              20140212
Time                  15.12
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT             Acetone
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                4.0894966 sec
RG                   126.97
DW                   62.400 usec
DE                     6.50 usec
TE                    295.6 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                     1H
P1                    14.30 usec
SI                    65536
SF              400.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



1

```
Current Data Parameters
NAME          bc-d28-rec2
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140619
Time                10.22
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 12363
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                  6.50 usec
TE                 297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.60 usec
PLW1         31.98900032 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          9.10000038 W
PLW12         0.24608000 W
PLW13         0.19933000 W

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

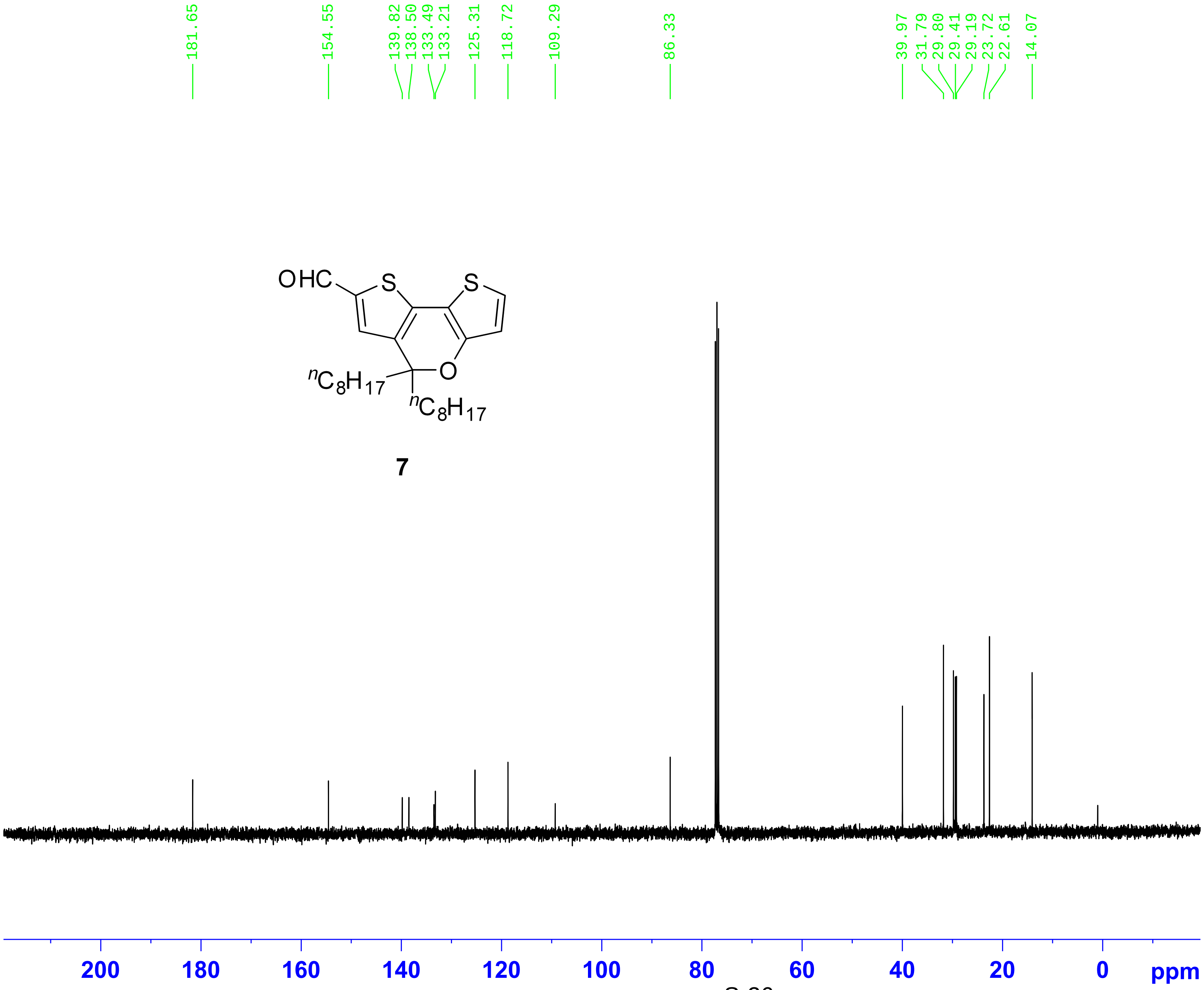


7

NAME	bc2188-3
EXPNO	1
PROCNO	1
Date_	20130412
Time	21.04
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	45.67
DW	62.400 usec
DE	6.50 usec
TE	295.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

SFO1	400.1324710 MHz
NUC1	1H
P1	14.30 usec
SI	65536
SF	400.1300097 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

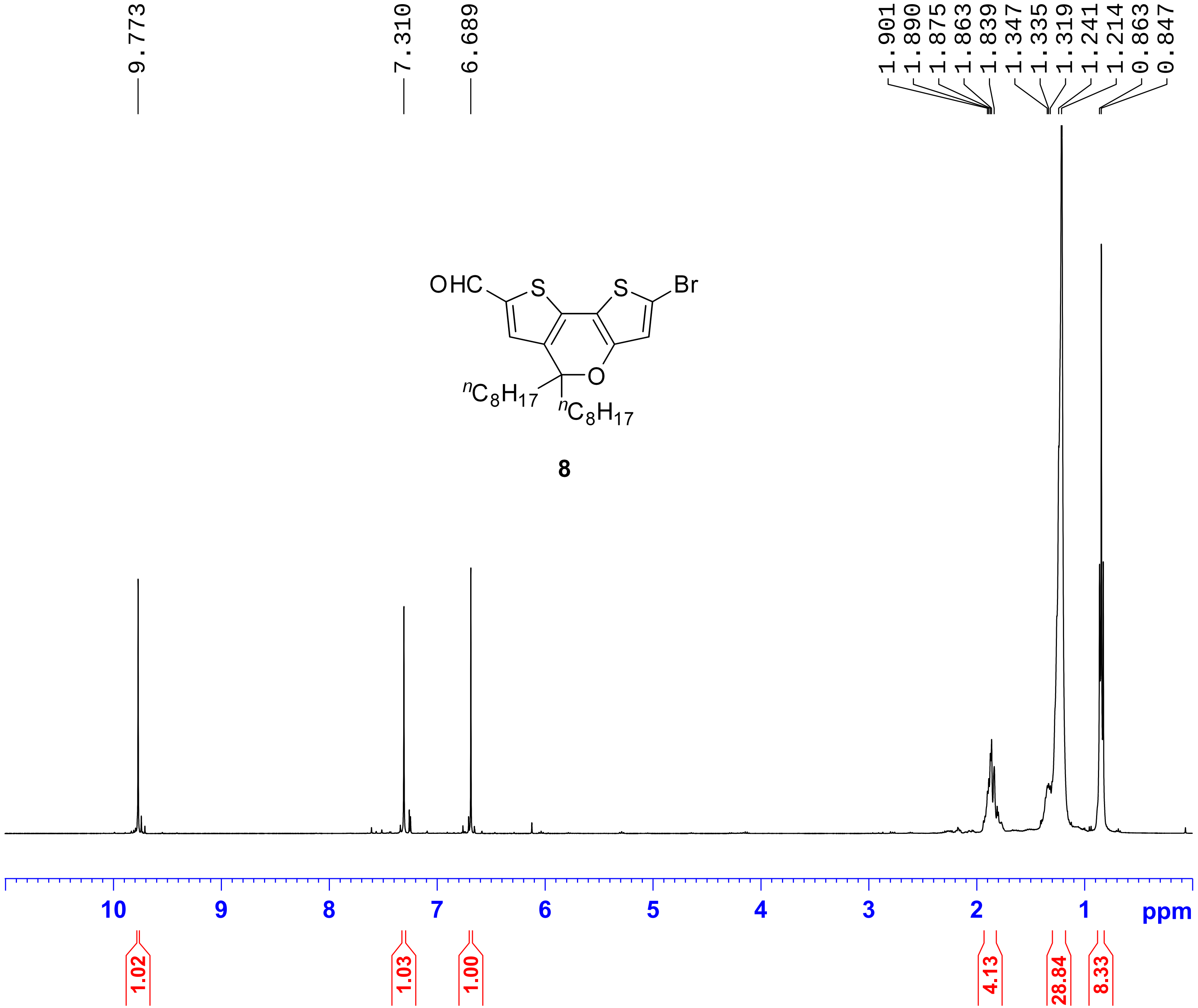


7

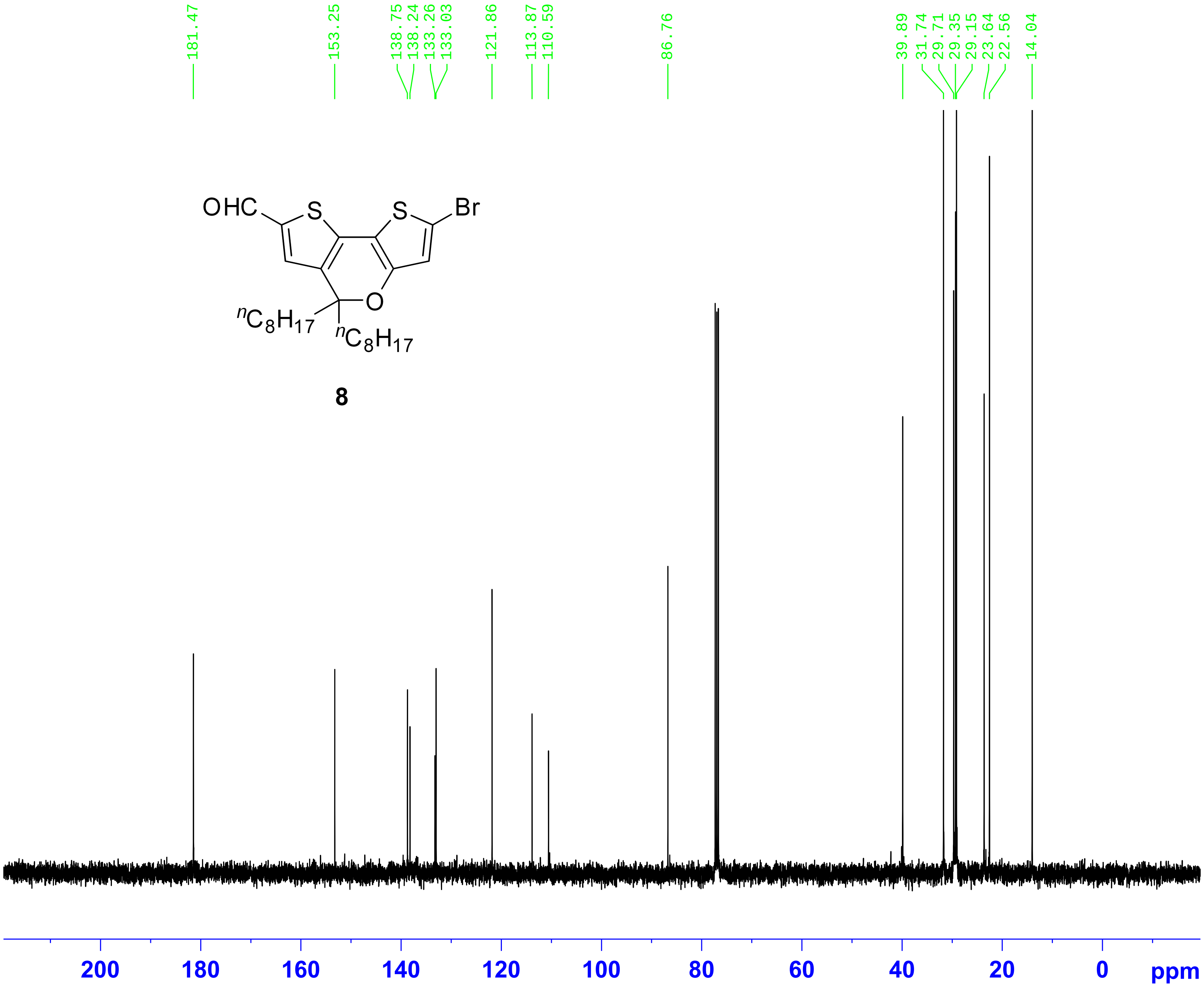
NAME	bc2188-3
EXPNO	2
PROCNO	1
Date_	20130412
Time	21.09
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	92
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	196.92
DW	20.800 usec
DE	6.50 usec
TE	296.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

SFO1	100.6228298 MHz
NUC1	13C
P1	9.60 usec
SI	32768
SF	100.6127722 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

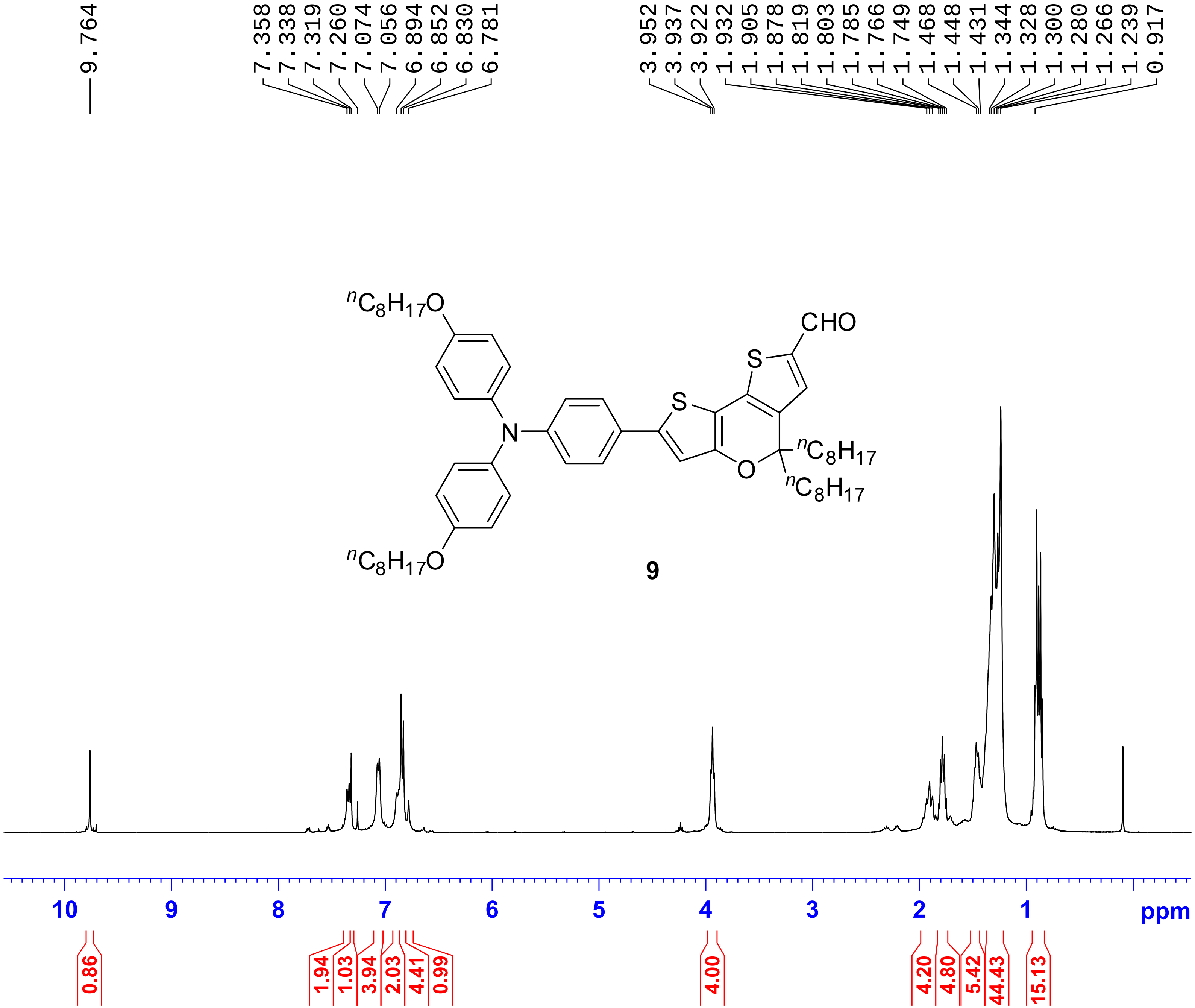


```
NAME            bc3004-2
EXPNO                  1
PROCNO                 1
Date_           20130424
Time               14.42
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894966 sec
RG                 15.71
DW                62.400 usec
DE                  6.50 usec
TE                 295.7 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 14.30 usec
SI                 65536
SF           400.1300096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



8

```
NAME               bc3004-2
EXPNO                     2
PROCNO                    1
Date_              20130424
Time                  14.44
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       38
DS                        2
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631988 sec
RG                   196.92
DW                   20.800 usec
DE                     6.50 usec
TE                    296.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                    13C
P1                     9.60 usec
SI                    32768
SF              100.6127774 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

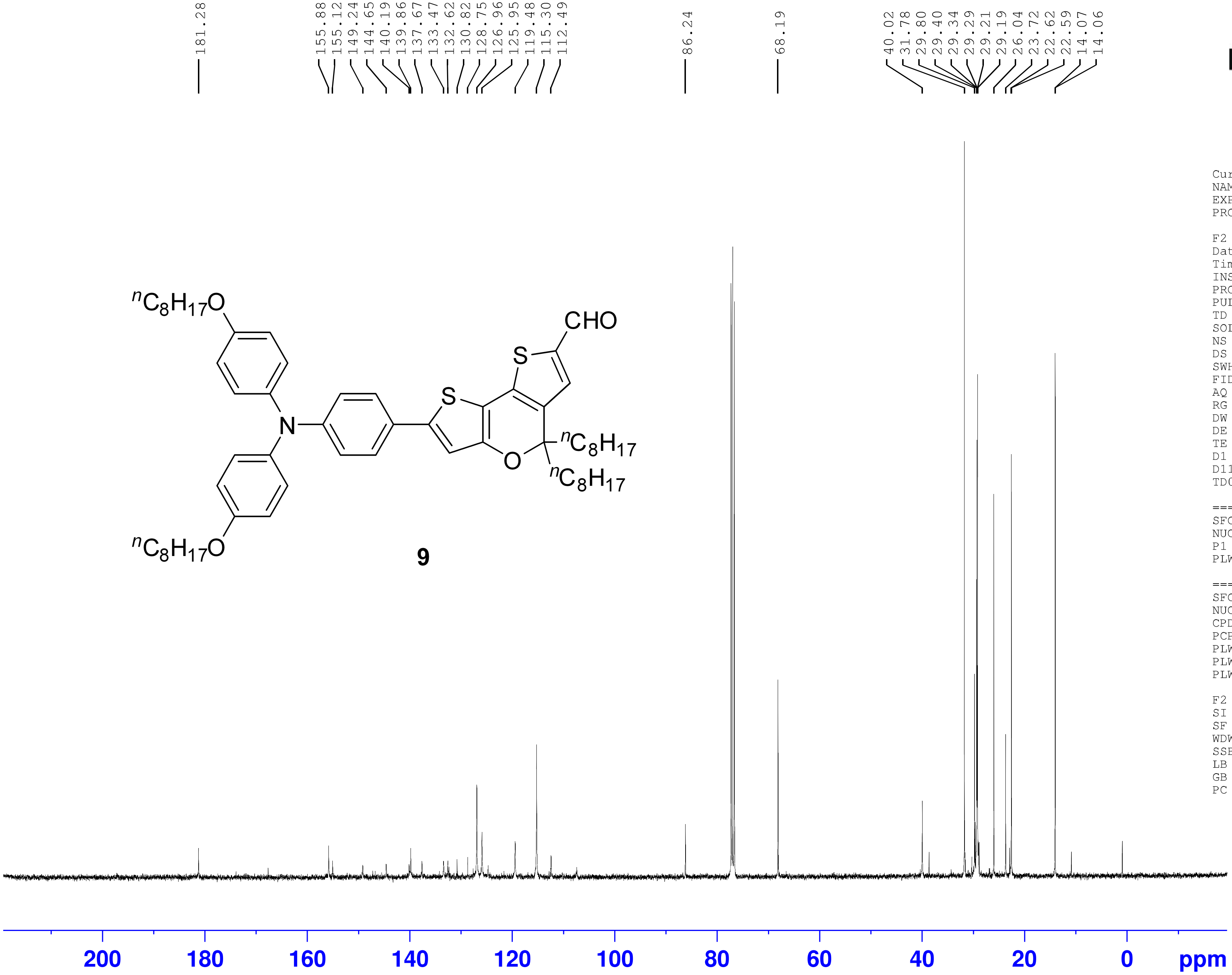


9

NAME	bc3010
EXPNO	3
PROCNO	1
Date_	20130503
Time	13.13
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	17.38
DW	62.400 usec
DE	6.50 usec
TE	295.8 K
D1	1.00000000 sec
TD0	1

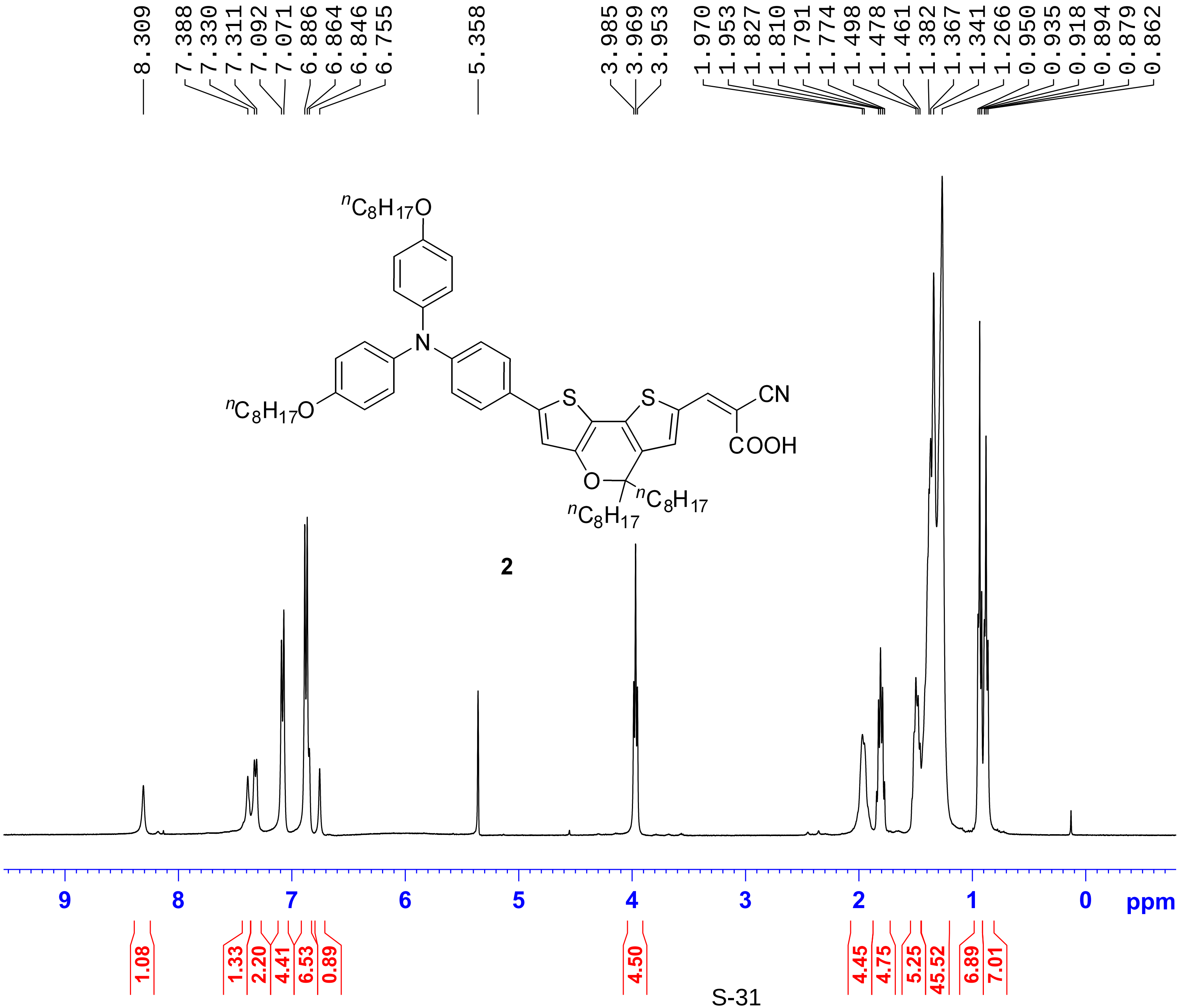
======== CHANNEL f1 ========

SFO1	400.1324710 MHz
NUC1	1H
P1	14.30 usec
SI	65536
SF	400.1300094 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



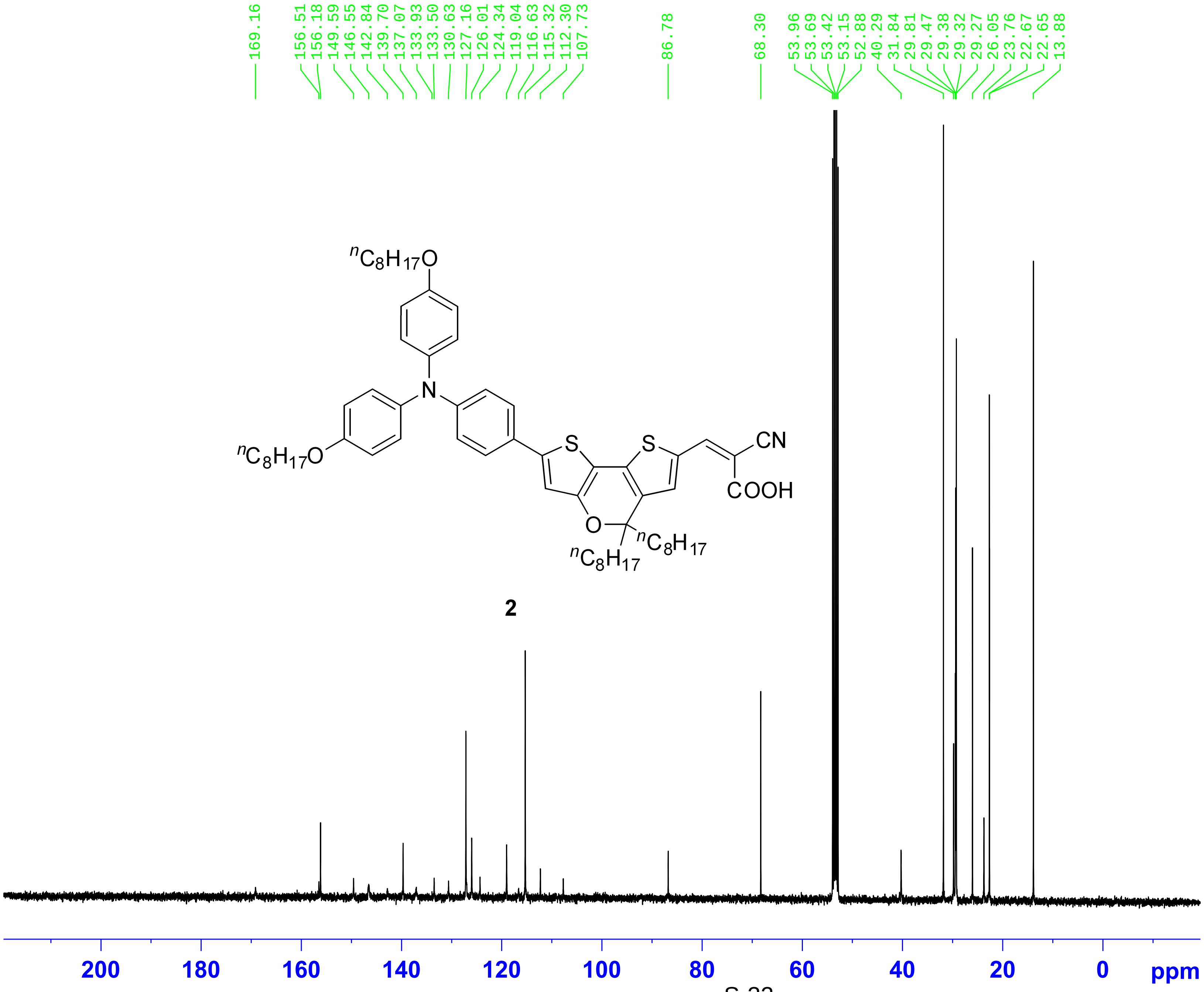
9

```
Current Data Parameters
NAME              bc3010
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130503
Time               14.40
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1207
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                196.92
DW                20.800 usec
DE                  6.50 usec
TE                 296.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                  9.60 usec
PLW1         31.98900032 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          9.10000038 W
PLW12         0.24608000 W
PLW13         0.19933000 W

F2 - Processing parameters
SI                 32768
SF           100.6127766 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



2

```
NAME           BC-d29-F2
EXPNO                  1
PROCNO                 1
Date_           20140123
Time               17.26
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT           CD2Cl2
NS                     3
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894966 sec
RG                 27.78
DW                62.400 usec
DE                  6.50 usec
TE                 295.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                  1H
P1                 14.30 usec
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



2

```
NAME             BC-d29-F2
EXPNO                    2
PROCNO                   1
Date_             20140123
Time                 19.20
INSTRUM              spect
PROBHD     5 mm DUL 13C-1
PULPROG            zgpg30
TD                   65536
SOLVENT             CD2Cl2
NS                    2000
DS                       2
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  196.92
DW                  20.800 usec
DE                    6.50 usec
TE                   297.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6228298 MHz
NUC1                   13C
P1                    9.60 usec
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```