Supporting Information

For

Palladium-Catalyzed Perarylation of 3-Thiophene- and 3-Furancarboxilic Acids Accompanied by C-H Bond Cleavage and Decarboxylation

Masaya Nakano, Hayato Tsurugi, Tetsuya Satoh, and Masahiro Miura*

Department of Applied Chemistry, Faculty of Engineering, Osaka University, Suita, Osaka 565-0871, Japan

e-mail: miura@chem.eng.osaka-u.ac.jp

List of Contents for Experimental Section

Experimental Section

General: ^{1}H and ^{13}C NMR spectra were recorded at 400 MHz and 100 MHz, respectively, for $CDCl_3$ solutions. MS data were obtained by EI. GC analysis was carried out using a silicon OV-17 column (i. d. 2.6 mm x 1.5 m) or a CBP-1 capillary column (i. d. 0.5 mm x 25 m). GC-MS analysis was carried out using a CBP-1 capillary column (i. d. 0.25 mm x 25 m).

N-Phenyl-3-thiophenecarboxamide (**5**) was prepared by the reaction of 3-thiophenecarbonyl chloride with aniline in the presence of pyridine in ether. Ethyl 3-thiophenecarboxylate (**7**) was prepared by esterification of 3-thiophenecarboxylic acid (**2**) in the presence of a catalytic amount of sulfuric acid in refluxing ethanol. Other starting materials were commercially available.

The following experimental procedure may be regarded as typical in methodology and scale.

Tetraphenylation of 3-Thiophenecarboxylic acid (2) with Bromobenzene (1a) (Table 1, entry 8): In a 20 cm^3 two-necked flask were added the bromide **1a** (2.5 mmol, 393 mg), the acid **2** (0.5 mmol, 64 mg), $Pd(OAc)_2$ (0.05 mmol, 11.2 mg), PCy_3 (0.2 mmol, 56 mg), Cs_2CO_3 (2.5 mmol, 815 mg), MS4A (150 mg), 1-methylnaphthalene (94 mg) as internal standard, and mesitylene (5 mL). The resulting mixture was stirred under N_2 (balloon) at 170 °C (bath temperature) for 8 h. After cooling, analysis of the mixture by GC and GC-MS confirmed formation of 2,3,4,5-tetraphenylthiophene (**4a**) (163 mg, 83%) together with 2,3,5-triphenylthiophene (**3**) (25 mg, 16%). The tetraphenylthiophene **4a** (161 mg, 82 %) was also isolated by filtration of the mixture with a filter paper with ether, evaporation of the solvents, and chromatography on silca gel using hexane-ethyl acetate (99:1, v/v).

Triphenylation of *N*-Phenyl-3-thiophenecarboxamide (5) with Bromobenzene (1a) (Scheme 2): In a 20 cm^3 two-necked flask were added the bromide

1a (3 mmol, 471 mg), the amide **5** (0.5 mmol, 102 mg), $Pd(OAc)_2$ (0.05 mmol, 11.2 mg), PPh_3 (0.1 mmol, 26 mg), Cs_2CO_3 (3 mmol, 978 mg), 1-methylnaphthalene (55 mg) as internal standard, and *o*-xylene (2.5 mL). The resulting mixture was stirred under N_2 (balloon) at 170 °C (bath temperature) for 10 h. After cooling, *N*,2,4,5-tetraphenyl-3-thiophenecarboxamide **6** (182 mg, 84 %) was isolated by filtration of the mixture with a filter paper with ether, evaporation of the solvents, and chromatography on silca gel using hexane-ethyl acetate (94:6, v/v).

Diphenylation of Ethyl 3-Thiophenecarboxylate (7) with Bromobenzene (1a) (Scheme 3): In a 100 cm^3 two-necked flask were added the bromide **1a** (12 mmol, 1.88 g), the ester **7** (4 mmol, 625 mg), $Pd(OAc)_2$ (0.4 mmol, 90 mg), P(biphenyl-2-yl)^{t}Bu$_2$ (0.8 mmol, 239 mg), Cs_2CO_3 (12 mmol, 3.90 g), MS4A (1.2 g), and DMF (40 mL). The resulting mixture was stirred under N_2 (balloon) at 150 °C (bath temperature) for 11 h. After cooling, the mixture was poured into water and extracted with diethyl ether. The solvent was dried over sodium sulfate and evaporated. The residue was chromatographed on silica gel using hexane-ethyl acetate (99.5:0.5, v/v) to afford ethyl 2,5-diphenyl-3-thiophenecarboxylate (**8**) (1.13 g, 91%). Hydrolysis of **8** (2 mmol) with KOH (10 mmol) in refluxing ethanol (15 mL) for 10 h and acidification with dilute HCl gave 2,5-diphenyl-3-thiophenecarboxylic acid (**11**) quantitatively.

Diarylation of 2,5-Diphenyl-3-thiophenecarboxylic acid (11) with 4-Bromoanisole (1d) (Table 3): In a 20 cm^3 two-necked flask were added the bromide **1d** (1.5 mmol, 281 mg), the acid **11** (0.5 mmol, 140 mg), $Pd(OAc)_2$ (0.05 mmol, 11.2 mg), PCy_3 (0.2 mmol, 56 mg), Cs_2CO_3 (1.5 mmol, 487 mg), MS4A (150 mg), dibenzyl (56 mg) as internal standard, and mesitylene (5 mL). The resulting mixture was stirred under N_2 (balloon) at 170 °C (bath temperature) for 9 h. After cooling, 3,4-bis(4-methoxyphenyl)-2,5-diphenylthiophene (**12**) (106 mg, 47%) was isolated by filtration of the mixture with a filter paper with ether, evaporation of the solvents, and chromatography on silca gel using hexane-ethyl acetate (99:1, v/v).

Characterization Data of Products.

2,3,4,5-Tetraphenylthiophene (4a):[1] mp 184-185 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95-6.98 (m, 4H), 7.08-7.14 (m, 6H), 7.19-7.25 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 126.58, 127.20, 127.82, 128.29, 129.20, 130.86, 134.25, 136.46, 138.55, 139.48; HRMS *m/z* (M^+) Calcd for $C_{28}H_{20}S$ 388.1286. Found 388.1283.

2,3,4,5-Tetrakis(4-methylphenyl)thiophene (4b):[2] mp 227-228 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.26 (s, 6H), 2.29 (s, 6H), 6.84 (d, *J* = 8.1 Hz, 4H), 6.91 (d, *J* = 8.1 Hz, 4H), 7.01 (d, *J* = 8.1 Hz, 4H), 7.12 (d, *J* = 8.1 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.15, 21.22, 128.52, 128.96, 129.00, 130.67, 131.62, 133.66, 135.86, 136.74, 137.96, 139.09; HRMS *m/z* (M^+) Calcd for $C_{32}H_{28}S$ 444.1912. Found 444.1913.

2,3,4,5-Tetrakis(4-*tert*-butylphenyl)thiophene (4c): mp 193-194 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.24 (s, 18H), 1.28 (s, 18H), 6.87 (d, *J* = 8.4 Hz, 4H), 7.09 (d, *J* = 8.4 Hz, 4H), 7.17-7.23 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.26, 31.30, 34.36, 34.49, 124.40, 125.11, 128.49, 130.41, 131.61, 133.87, 137.55, 139.63, 149.10, 149.80; HRMS *m/z* (M^+) Calcd for $C_{44}H_{52}S$ 612.3790. Found 612.3792.

2,3,4,5-Tetrakis(4-methoxyphenyl)thiophene (4d):[3] mp 217-218 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.74 (s, 6H), 3.77 (s, 6H), 6.67 (d, *J* = 8.8 Hz, 4H), 6.76 (d, *J* = 9.2 Hz, 4H), 6.87 (d, *J* = 8.8 Hz, 4H), 7.15 (d, *J* = 9.2 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.03, 55.18, 113.30, 113.72, 127.08, 129.12, 130.30, 131.95, 137.18, 138.35, 158.06, 158.64; HRMS *m/z* (M^+) Calcd for $C_{32}H_{28}O_4S$ 508.1708. Found 508.1712.

2,3,4,5-Tetrakis(4-trifluoromethylphenyl)thiophene (4e): mp 166-167 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (d, *J* = 8.1 Hz, 4H), 7.30 (d, *J* = 8.1 Hz, 4H), 7.44 (d, *J* = 8.1 Hz, 4H), 7.53 (d, *J* = 8.1 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 123.88 (q, *J* = 272.3 Hz), 123.91 (q, *J* = 272.4 Hz), 125.68 (q, *J* = 4 Hz), 125.37 (q, *J* = 4 Hz), 129.17, 129.66 (q, *J* = 32.1 Hz), 129.98 (q, *J* = 32.8 Hz), 130.92, 133.66, 138.76, 138.89, 139.18; HRMS *m/z* (M^+) Calcd for $C_{32}H_{16}F_{12}S$ 660.0781. Found 660.0780.

2,3,4,5-Tetrakis(4-ethoxycarbonylphenyl)thiophene (4f):[4] mp 81-82 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.371 (t, *J* = 7.0 Hz, 6H), 1.374 (t, *J* = 7.0 Hz, 6H), 4.34 (q, *J* = 7.0 Hz, 4H), 4.36 (q, *J* = 7.0 Hz, 4H), 7.01 (d, *J* = 8.1 Hz, 4H), 7.26 (d, *J* = 8.4 Hz, 4H), 7.82 (d, *J* = 8.1 Hz, 4H), 7.91 (d, *J* = 8.4 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.28, 14.30, 61.05, 61.06, 128.97, 129.28, 129.47, 129.61, 129.78, 130.63, 137.73, 139.34, 139.36, 140.25, 166.08, 166.26; HRMS *m/z* (M^+) Calcd for $C_{40}H_{36}O_8S$ 676.2131. Found 676.2170.

2,3,4,5-Tetrakis(biphenyl-4-yl)thiophene (4g): mp 180-181 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.4 Hz, 4H), 7.27-7.34 (m, 4H), 7.35-7.44 (m,16H), 7.49 (d, *J* = 8.4 Hz, 4H), 7.58 (d, *J* = 8.1 Hz, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 126.54, 126.84, 126.90, 127.01, 127.21, 127.36, 128.70, 128.77, 129.52, 131.32, 133.22, 135.47, 138.48, 139.11, 139.23, 139.88, 140.40, 140.51; HRMS *m/z* (M^+) Calcd for $C_{52}H_{36}S$ 692.2538. Found 692.2545.

***N*,2,4,5-Tetraphenyl-3-thiophenecarboxamide (6)**: mp 214-215 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00-7.11 (m, 3H), 7.18-7.23 (m, 7H), 7.27-7.43 (m, 8H), 7.66 (d, *J* = 7.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 120.18, 124.56, 127.69, 127.71, 128.46, 128.49, 128.56, 128.58, 128.81, 128.91, 129.18, 129.90, 132.74, 133.30, 135.03, 135.48, 137.36, 137.65, 139.73, 142.25, 163.78; HRMS *m/z* (M^+) Calcd for $C_{29}H_{21}NOS$ 431.1344. Found 431.1349.

Ethyl 2,5-Diphenyl-3-thiophenecarboxylate (8):[5] mp 45-46 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.20 (t, *J* = 7.1 Hz, 3H), 4.22 (q, *J* = 7.1 Hz, 2H), 7.32 (t, *J* = 7.3 Hz, 1H), 7.38-7.43 (m, 5H), 7.52-7.55 (m, 2H), 7.62 (d, J = 7.33 Hz, 2H), 7.71 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.02, 60.57, 125.42, 125.72, 127.92, 128.01, 128.58, 128.96, 129.01, 129.78, 133.34, 133.38, 142.46, 149.71, 163.27; HRMS *m/z* (M^+) Calcd for $C_{19}H_{16}O_2S$ 308.0871. Found 308.0878.

Ethyl 2,5-Bis(4-methoxyphenyl)-3-thiophenecarboxylate (9): mp 92-93 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.24 (t, *J* = 7.1 Hz, 3H), 3.84 (s, 3H), 3.85 (s, 3H), 4.23

(q, *J* = 7.1 Hz, 2H), 6.93 (d, *J* = 9.2 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 7.48 (d, *J* = 8.8 Hz, 2H), 7.53 (d, *J* = 9.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) 14.14, 55.32, 55.36, 60.45, 113.37, 114.38, 124.35, 125.72, 126.29, 126.97, 128.21, 131.07, 141.73, 149.09, 159.51, 159.92, 163.41; HRMS *m/z* (M^+) Calcd for $C_{21}H_{20}O_4S$ 368.1082. Found 368.1086.

2,5-Diphenyl-3-thiophenecarboxylic Acid (10):[6] mp 196-197 °C; ^{1}H NMR (400 MHz, DMSO-d_6) 7.36 (t, *J* = 7.3 1H), 7.41-7.47 (m, 5H), 7.53-7.56 (m, 2H), 7.7 (d, *J* = 7.3 Hz, 2H), 7.79 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) 125.29, 125.84, 128.06, 128.16, 128.53, 129.17, 129.32, 129.98, 132.53, 132.77, 141.56, 147.76, 163.92; HRMS *m/z* (M^+) Calcd for $C_{17}H_{12}O_2S$ 280.0558. Found 280.0551.

2,5-Bis(4-methoxyphenyl)-3-thiophenecarboxylic Acid (11): mp 223-224 °C; ^{1}H NMR (400 MHz, DMSO-d_6) 3.79 (s, 3H), 3.80 (s, 3H), 6.96-7.01 (m, 4H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.61 (d, *J* = 9.2 Hz, 2H), 7.62 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) 55.17, 55.19, 113.52, 114.56, 124.61, 125.10, 125.32, 126.62, 129.15, 130.65, 140.81, 147.05, 159.22, 159.5, 164.08; HRMS *m/z* (M^+) Calcd for $C_{19}H_{16}O_4S$ 340.0769. Found 340.0778.

3,4-Bis(4-methoxyphenyl)-2,5-diphenylthiophene (12): mp 210-211 °C; ^{1}H NMR (400 MHz, $CDCl_3$) 3.74 (s, 6H), 6.67 (d, *J* = 9.0 Hz, 4H), 8.79 (d, *J* = 9.0 Hz, 4H), 7.17-7.26 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) 55.04, 113.33, 127.05, 128.27, 128.86, 129.17, 131.93, 134.50, 138.05, 139.14, 158.18; HRMS *m/z* (M^+) Calcd for $C_{30}H_{24}O_2S$ 448.1497. Found 448.1494.

2,5-Diphenyl-3,4-bis(4-trifluoromethyl-phenyl)thiophene (13): mp 141-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) 7.05 (d, *J* = 8.1 Hz, 4H), 7.18-7.21 (m, 4H), 7.24-7.27 (m, 6H), 7.39 (d, *J* = 8.1 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) 124.09 (q, *J* = 272 Hz), 125.02 (q, *J* = 4 Hz), 127.81, 128.58, 129.00 (q, *J* = 32 Hz), 129.31, 131.10, 133.37, 137.37, 139.77, 140.16; HRMS *m/z* (M^+) Calcd for $C_{30}H_{18}F_6S$ 524.1033. Found 524.1029.

2,5-Bis(4-methoxyphenyl)-3,4-diphenylthiophene (14):[7] mp 196-197 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.76 (s, 6H), 6.75 (d, J = 8.8 Hz, 4H), 6.94-6.98 (m, 4H), 7.08-7.13 (m, 6H), 7.15 (d, J = 8.8 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.17, 113.73, 126.42, 126.81, 127.78, 130.32, 130.88, 136.71, 137.68, 138.70, 158.75; HRMS m/z (M^+) Calcd for $C_{30}H_{24}O_2S$ 448.1497. Found 448.1490.

2,5-Bis(4-methoxyphenyl)-3,4-bis(4-trifluoromethylphenyl)thiophene (15): mp 172-173 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.78 (s, 6H), 6.78 (d, J = 8.8 Hz, 4H), 7.04 (d, J = 7.9 Hz, 4H), 7.10 (d, J = 8.8 Hz, 4H), 7.39 (d, J = 7.9 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.22, 114.0, 124.13 (q, J = 272 Hz), 124.98 (q, J = 4 Hz), 125.83, 128.81 (q, J = 32 Hz), 130.48, 131.13, 136.62, 139.37, 140.05, 159.20; HRMS m/z (M^+) Calcd for $C_{32}H_{22}F_6O_2S$ 584.1245. Found 584.1247.

2,3,4,5-Tetraphenylfuran (17a):[8] mp 172-173 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14-7.28 (m, 16H), 7.50-7.51 (m, 2H), 7.52-7.53 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 125.13, 125.86, 127.14, 127.29, 128.34, 128.36, 130.41, 130.91, 133.19, 147.72; HRMS m/z (M^+) Calcd for $C_{28}H_{20}O$ 372.1514. Found 372.1517.

2,3,4,5-Tetrakis(4-methoxyphenyl)furan (17d):[9] mp 208-209 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.79 (s, 6H), 3.79 (s, 6H), 6.79 (d, J = 8.8 Hz, 4H), 6.80 (d, J = 9.2 Hz, 4H), 7.06 (d, J = 8.8 Hz, 4H), 7.44 (d, J = 9.2 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.10, 55.22, 113.82, 113.83, 123.24, 124.15, 125.80, 127.14, 131.53, 147.10, 158.53, 158.72; HRMS m/z (M^+) Calcd for $C_{32}H_{28}O_5$ 492.1937. Found 492.1934.

2,3,4,5-Tetrakis(4-trifluoromethyl-phenyl)furan (17e): mp 196-197 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, J = 8.1 Hz, 4H), 7.57-7.59 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$:DMSO-d_6 = 4:1) δ 123.57 (q, J = 272 Hz), 123.58 (q, J = 272 Hz), 124.89, 125.22 (q, J = 4 Hz), 125.38 (q, J = 4 Hz), 125.80, 128.95 (q, J = 32 Hz), 129.33 (q, J = 32 Hz), 130.26, 132.66, 135.30, 147.35; HRMS m/z (M^+) Calcd for $C_{32}H_{16}F_{12}O$ 644.1010. Found 644.1004.

References in Supporting Information

(1) Braye, E. H.; Hübel, W.; Caplier, I. *J. Am. Chem. Soc.* **1961**, *83*, 4406.

(2) Xing, Y.; Xu, X.; Wang, F.; Lu, P. *Opt. Mat.* **2006**, *29*, 407.

(3) Arumugam, K.; Bollinger, J. E.; Fink, M.; Donahue, J. P. *Inorg. Chem.* **2007**, *46*, 3283.

(4) Hopff, H.; von der Crone, J. *Chimia* **1959**, *13*, 107.

(5) Potts, K. T.; Datta, S. K.; Marshall, J. L. *J. Org. Chem.* **1979**, *44*. 622.

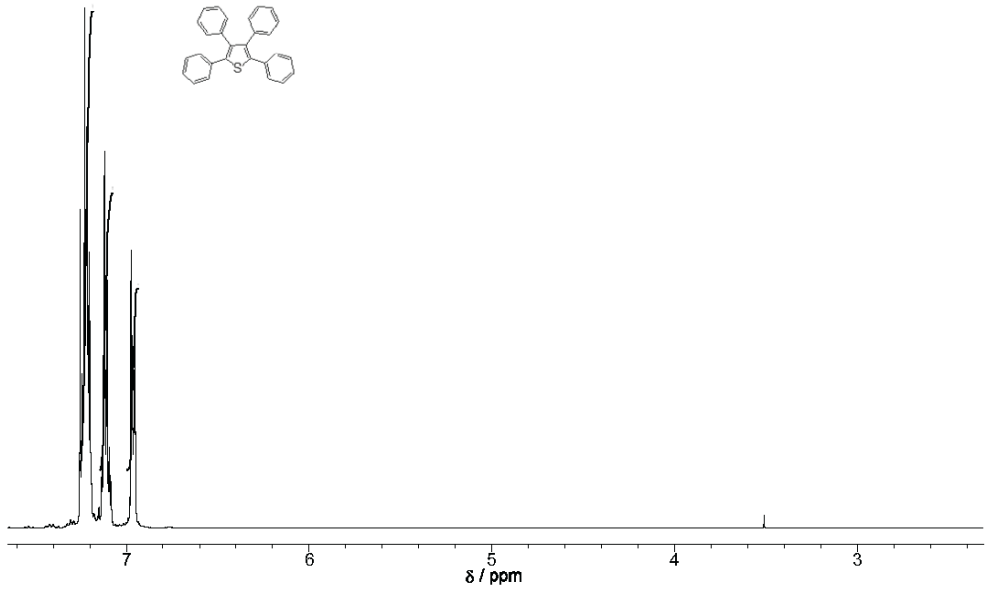
(6) Gotthart, H.; Weisshuhn, M. C.; Christl, B. *Chem. Ber.* **1976**, *109*, 753.

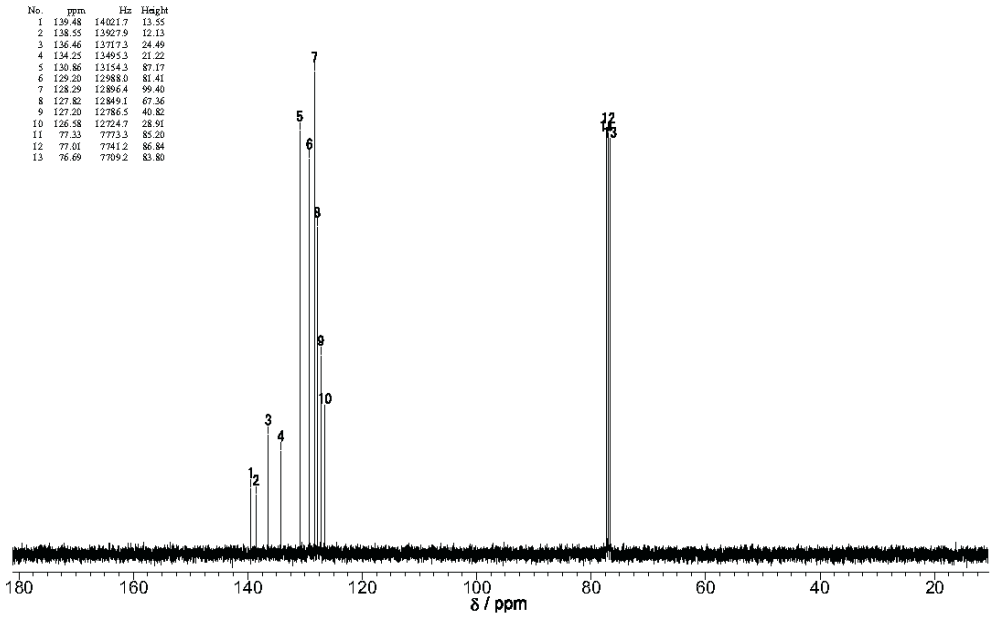
(7) Dang, Y.; Chen, Y. *J. Org. Chem.* **2007**, *72*, 6901.

(8) Tseng, J.-C.; Chen, J.-H.; Luh, T.-Y. *Synlett* **2006**, 1209.

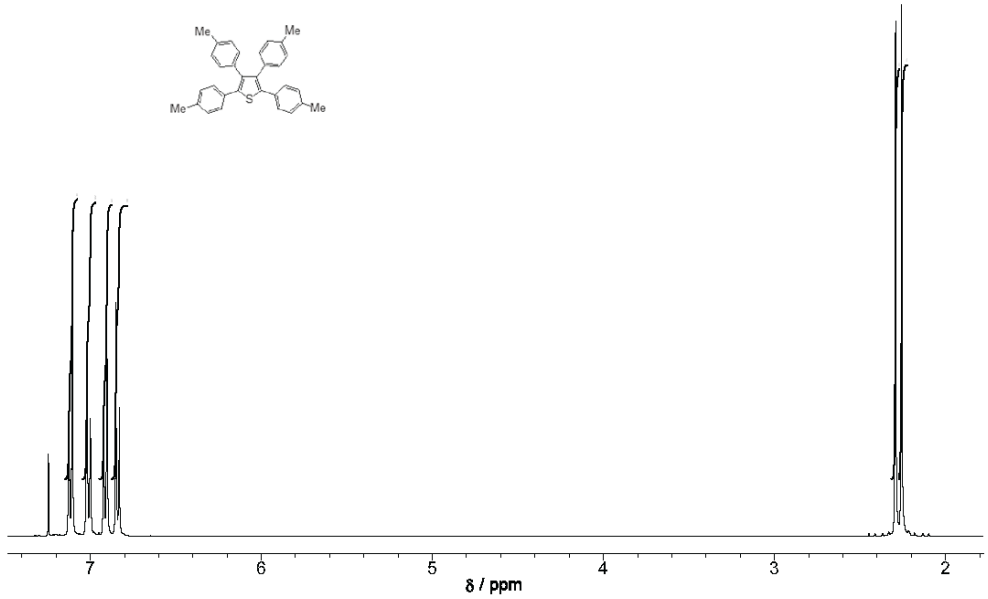
(9) Mortensen, D. S.; Rodriguez, A. L.; Carlson, K. E.; Sun, J.; Katzenellenbogen, B. S.; Katzenellenbogen, J. A. *J. Med. Chem.* **2001**, *44*, 3838.

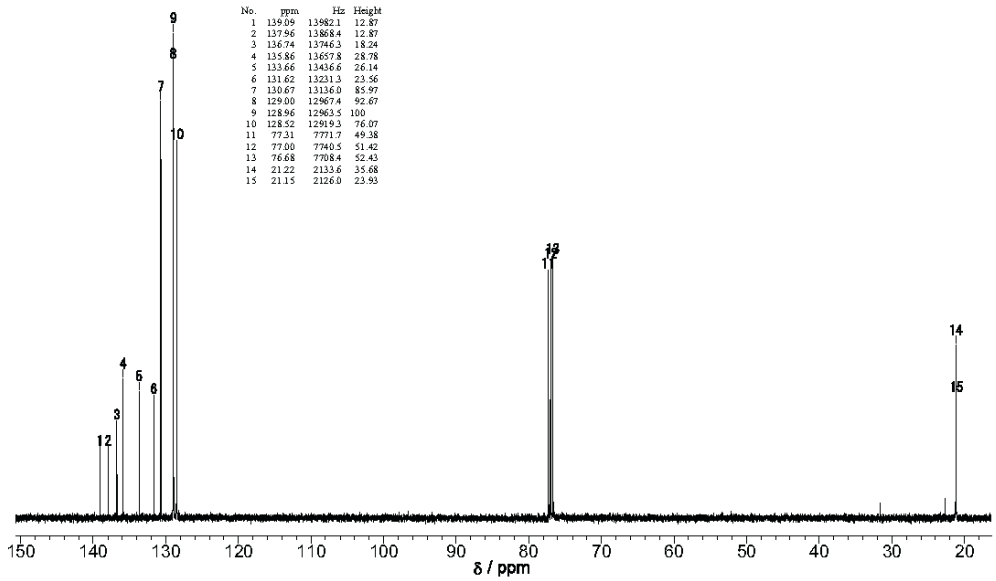
^{1}H and ^{13}C NMR of Compound **4a**



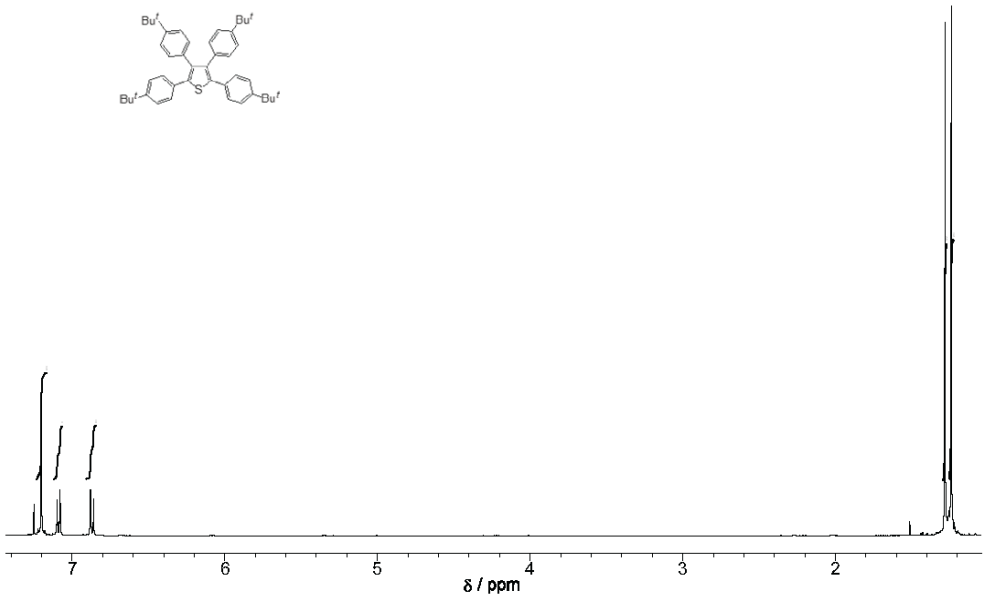


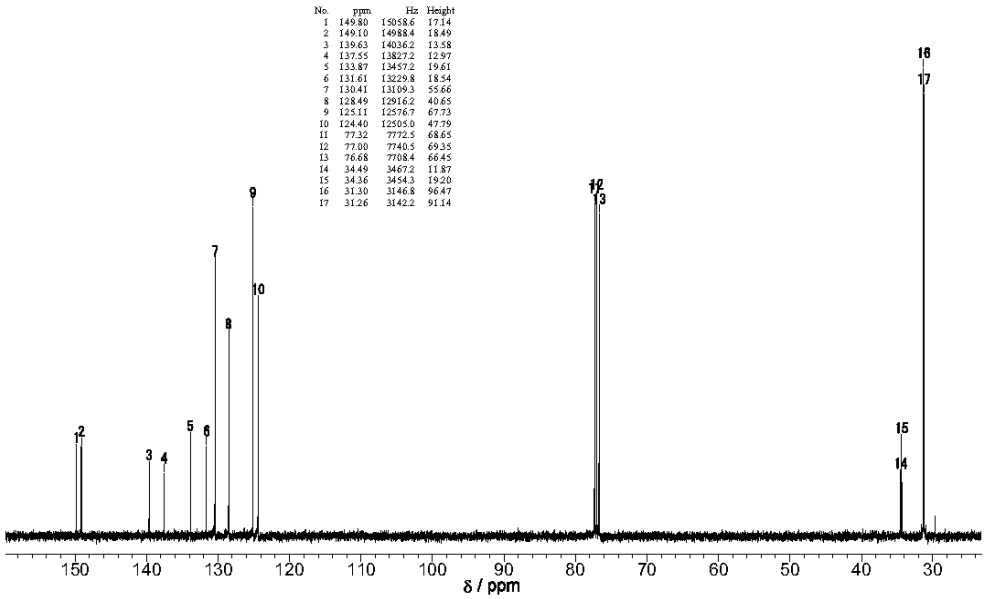
^{1}H and ^{13}C NMR of Compound **4b**



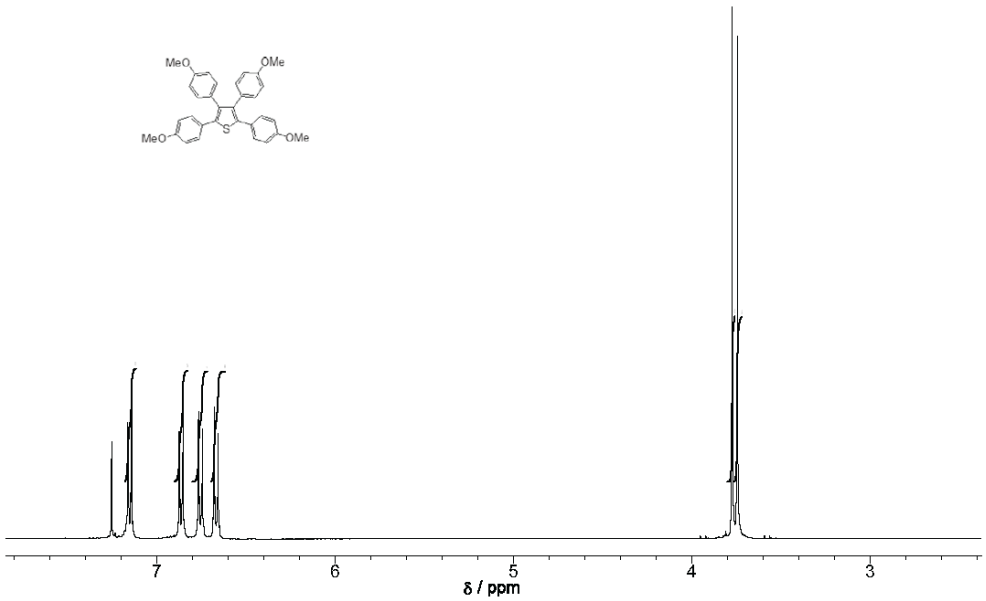


^{1}H and ^{13}C NMR of Compound **4c**

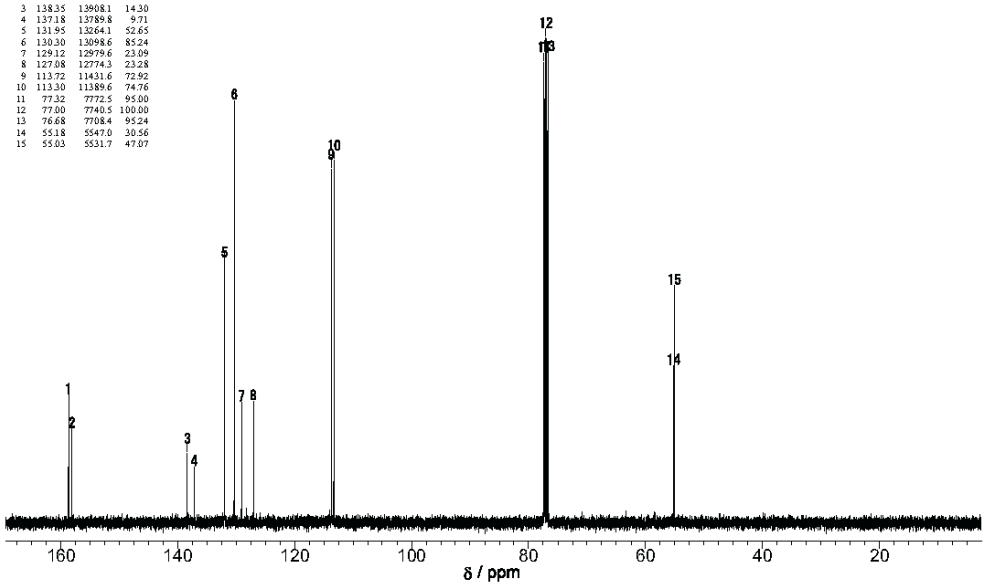




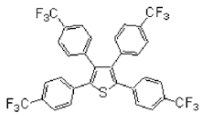
^{1}H and ^{13}C NMR of Compound **4d**

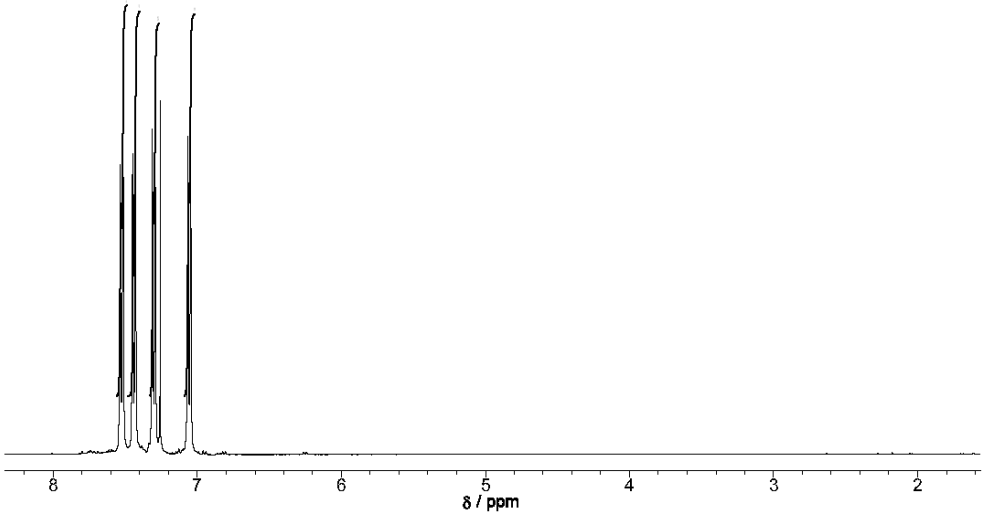


No.	ppm	Hz	Height
1	158.64	15947.4	24.59
2	158.06	15888.6	17.59
3	138.35	13908.1	14.30
4	137.18	13789.8	9.71
5	131.95	13264.1	52.65
6	130.30	13098.6	85.24
7	129.12	12979.6	23.09
8	127.08	12774.3	23.28
9	113.72	11431.6	72.92
10	113.30	11389.6	74.76
11	77.32	7772.5	95.00
12	77.00	7740.5	100.00
13	76.68	7708.4	95.24
14	55.18	5547.0	30.56
15	55.03	5531.7	47.07

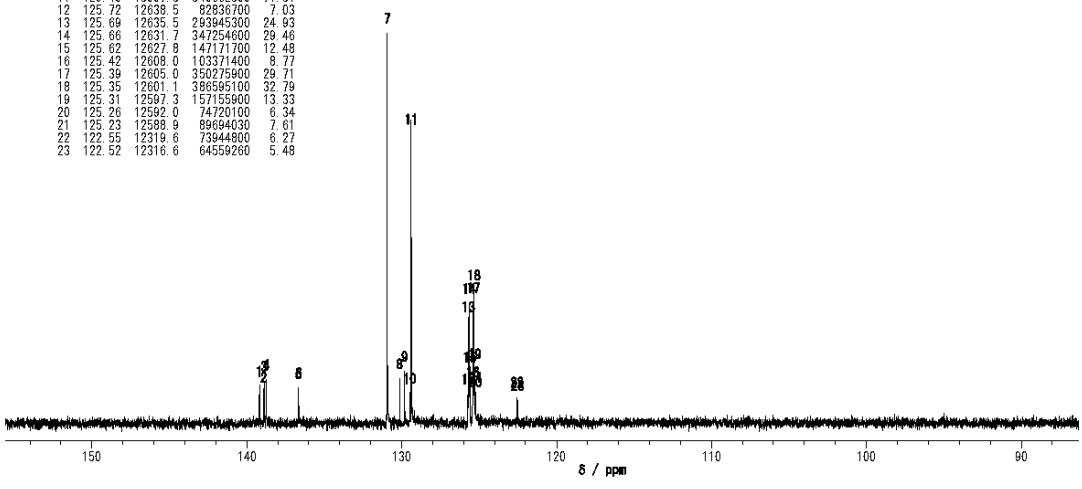


^{1}H and ^{13}C NMR of Compound **4e**

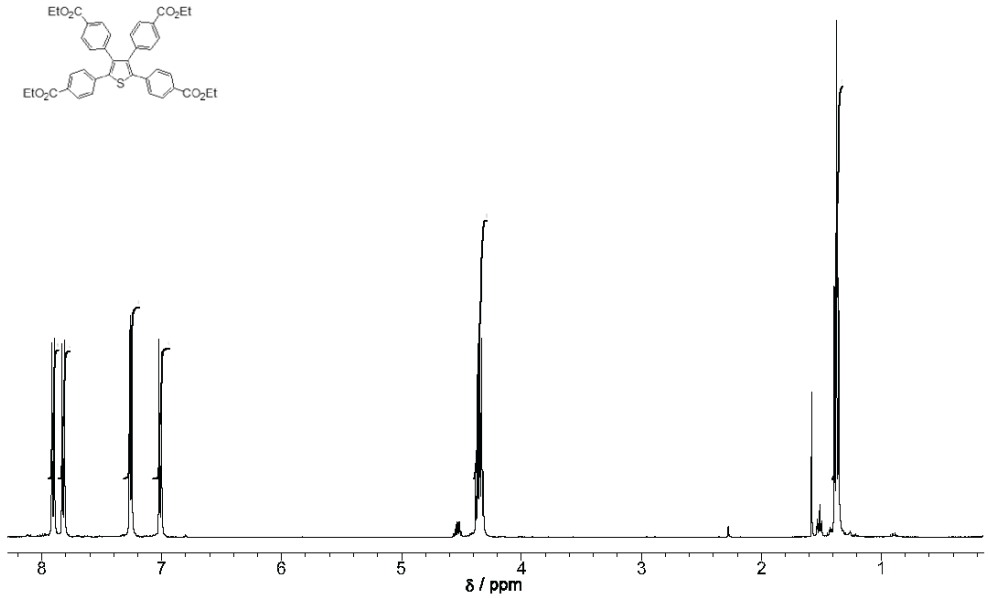




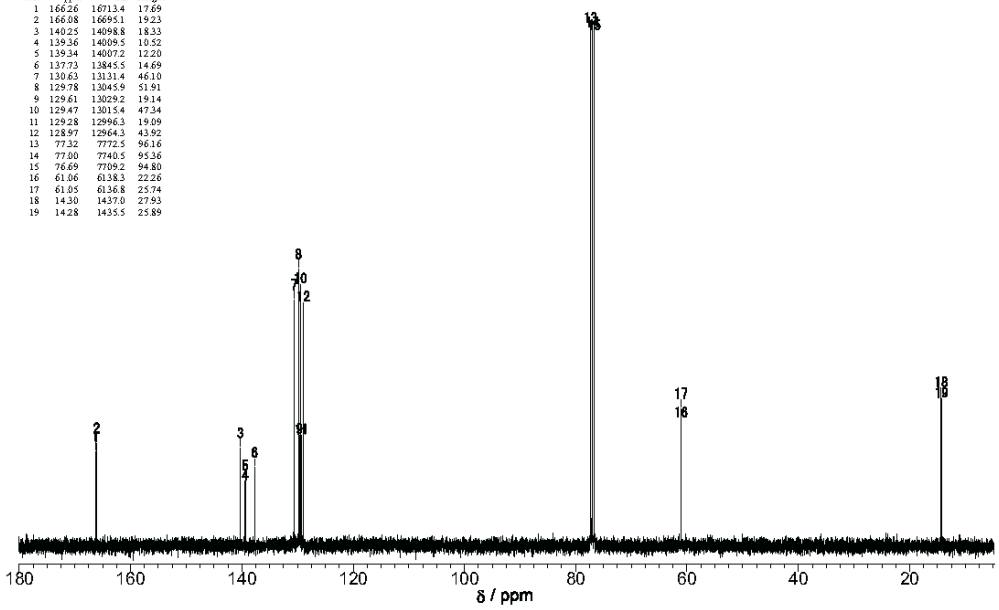
No.	ppm	Hz	Amp.	Height
1	139.18	13991.2	106556600	9.04
2	138.90	13963.0	88061510	7.47
3	138.88	13961.5	121376100	10.30
4	138.76	13948.5	125140100	10.61
5	136.66	13737.9	99477820	8.44
6	136.65	13736.4	101308000	8.59
7	130.92	13161.1	1135967000	96.36
8	130.14	13082.6	127628700	10.83
9	129.82	13049.8	149230300	12.66
10	129.50	13017.7	85011210	7.21
11	129.40	13007.8	840662300	71.31
12	125.72	12638.5	82836700	7.03
13	125.69	12635.5	293945300	24.93
14	125.66	12631.7	347254600	29.46
15	125.62	12627.8	147171700	12.48
16	125.42	12608.0	103371400	8.77
17	125.39	12605.0	350275900	29.71
18	125.35	12601.1	386595100	32.79
19	125.31	12597.3	157155900	13.33
20	125.26	12592.0	74720100	6.34
21	125.23	12588.9	89694030	7.61
22	122.55	12319.6	73944800	6.27
23	122.52	12316.6	64559260	5.48



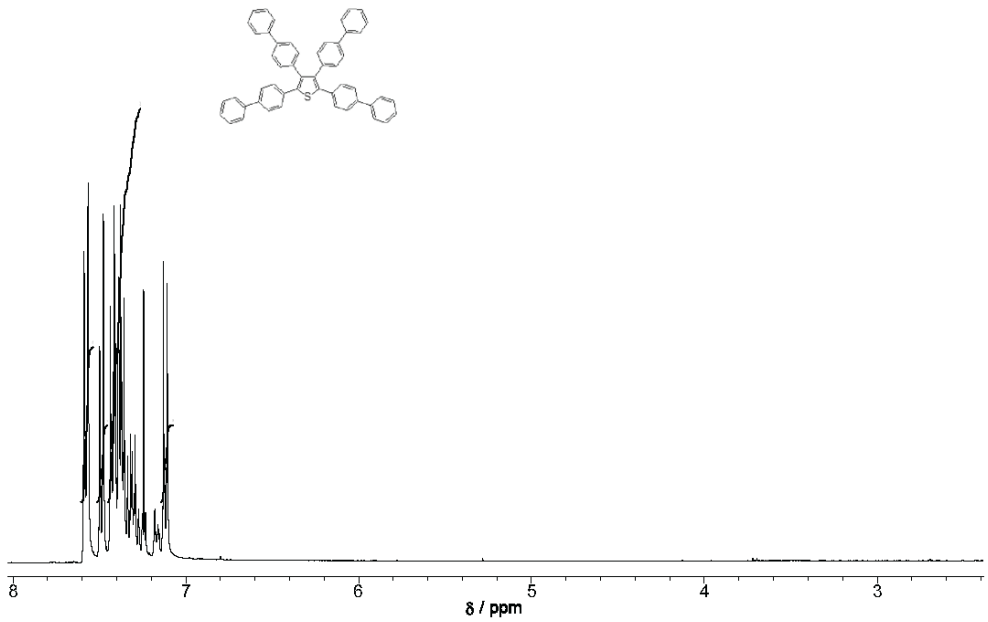
^{1}H and ^{13}C NMR of Compound **4f**



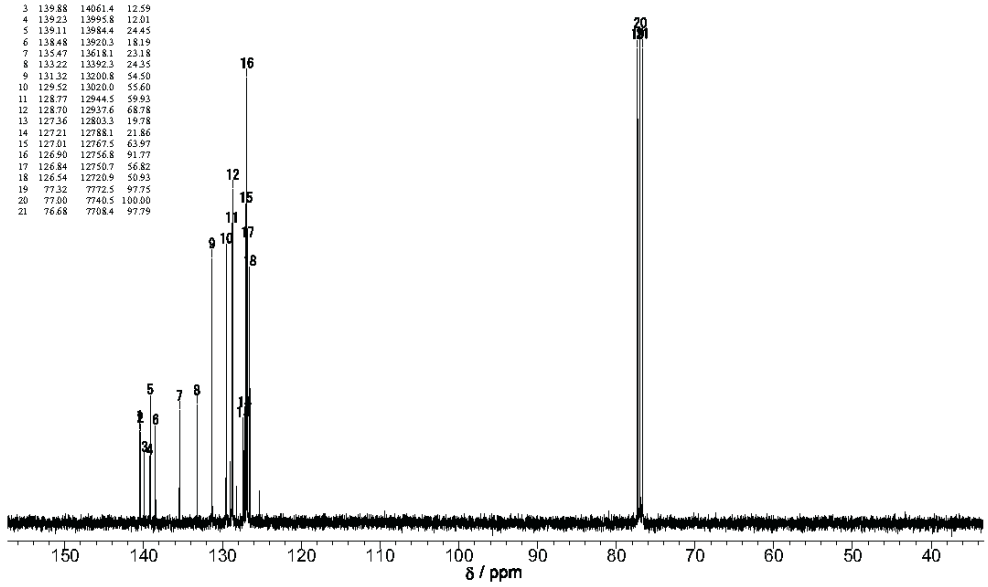
No.	ppm	Hz	Height
1	166.26	16713.4	17.69
2	166.08	16695.1	19.23
3	140.25	14098.8	18.33
4	139.36	14009.5	10.52
5	139.34	14007.2	12.20
6	137.73	13845.5	14.69
7	130.63	13131.4	46.10
8	129.78	13045.9	51.91
9	129.61	13029.2	19.14
10	129.47	13015.4	47.34
11	129.28	12996.3	19.09
12	128.97	12964.3	43.92
13	77.32	7772.5	96.16
14	77.00	7740.5	95.36
15	76.69	7709.2	94.80
16	61.06	6138.3	22.26
17	61.05	6136.8	25.74
18	14.30	1437.0	27.93
19	14.28	1435.5	25.89



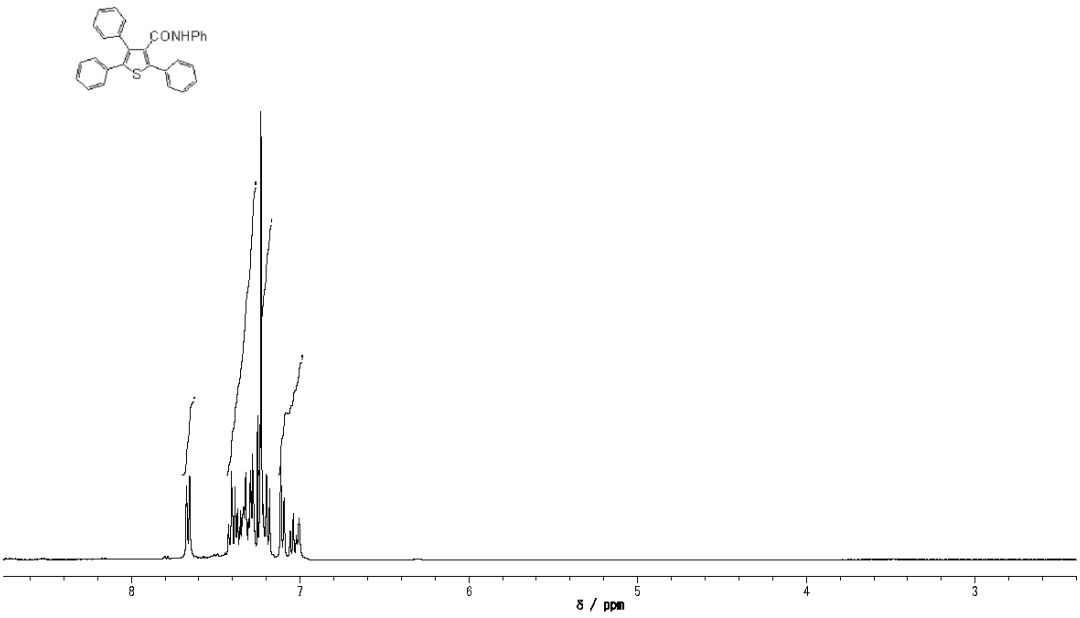
^{1}H and ^{13}C NMR of Compound **4g**

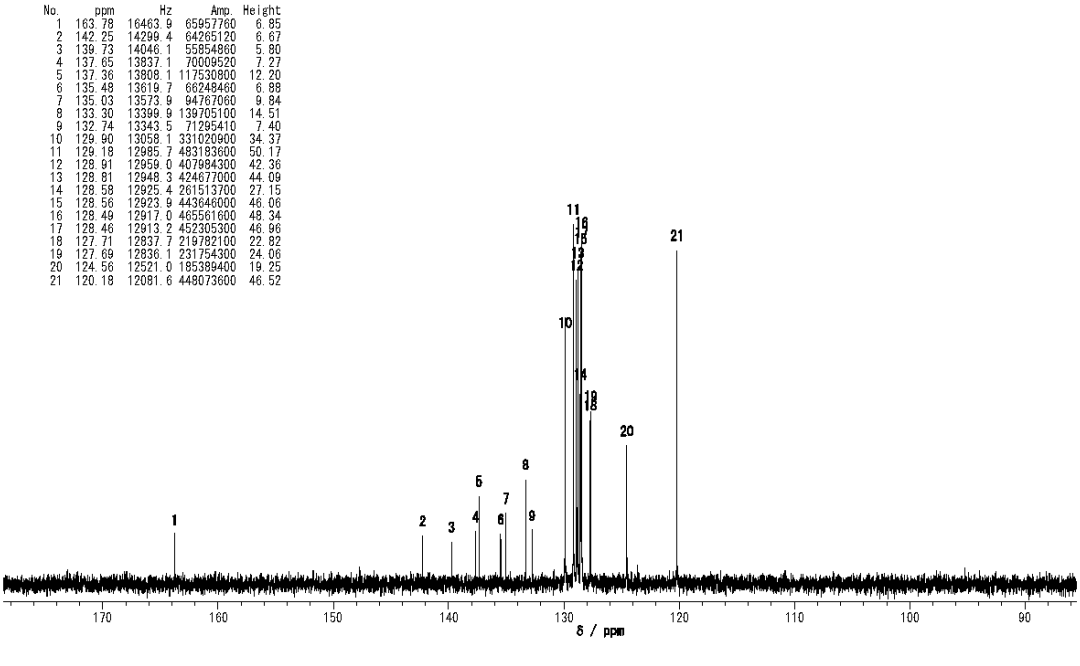


No.	ppm	Hz	Height
1	140.51	14124.7	19.03
2	140.40	14113.3	18.63
3	139.88	14061.4	12.59
4	139.23	13995.8	12.01
5	139.11	13984.4	24.45
6	138.48	13920.3	18.19
7	135.47	13618.1	23.18
8	133.22	13392.3	24.35
9	131.32	13200.8	54.50
10	129.52	13020.0	55.60
11	128.77	12944.5	59.93
12	128.70	12937.6	68.78
13	127.36	12803.3	19.78
14	127.21	12788.1	21.86
15	127.01	12767.5	63.97
16	126.90	12756.8	91.77
17	126.84	12750.7	56.82
18	126.54	12720.9	50.93
19	77.32	7772.5	97.75
20	77.00	7740.5	100.00
21	76.68	7708.4	97.79



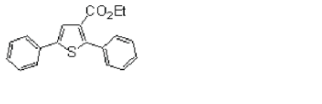
^{1}H and ^{13}C NMR of Compound **6**

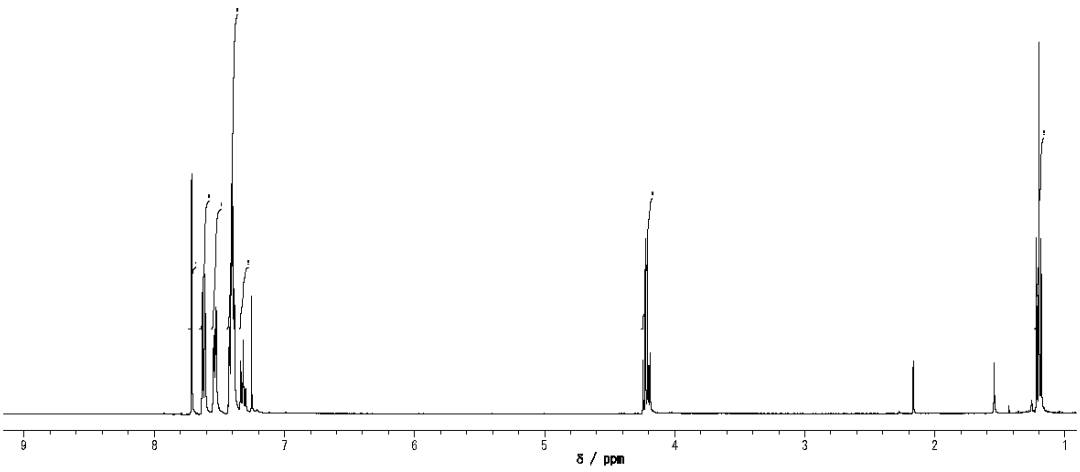




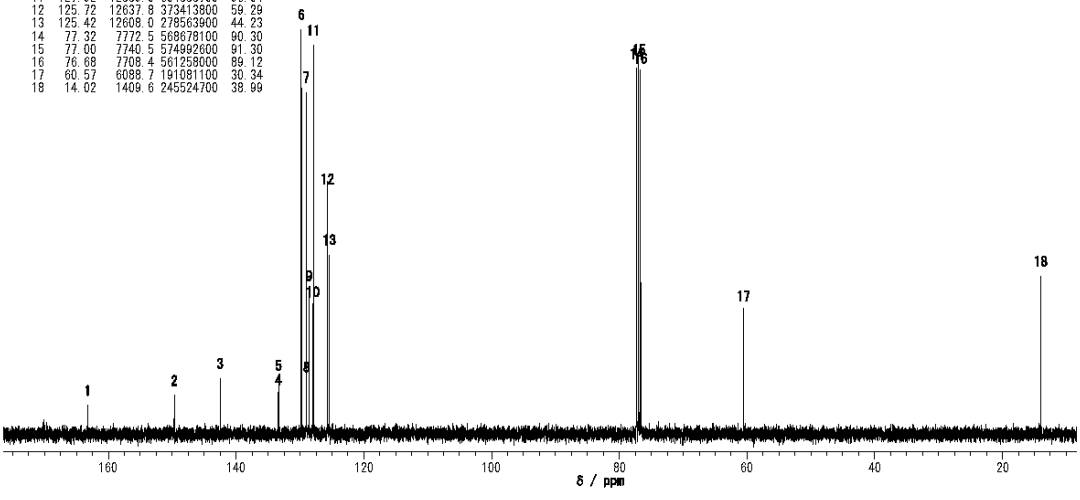
No.	ppm	Hz	Amp.	Height
1	163.78	16463.9	65957760	6.85
2	142.25	14299.4	64265120	6.67
3	139.73	14046.1	55854860	5.80
4	137.65	13837.1	70009520	7.27
5	137.36	13808.1	117530800	12.20
6	135.48	13619.7	68248460	6.88
7	135.03	13573.9	94767060	9.84
8	133.30	13399.9	139705100	14.51
9	132.74	13343.5	71295410	7.40
10	129.90	13058.1	331020900	34.37
11	129.18	12985.7	483183600	50.17
12	128.91	12959.0	407984300	42.36
13	128.81	12948.3	424677000	44.09
14	128.58	12925.4	261513700	27.15
15	128.56	12923.9	443646000	46.06
16	128.49	12917.0	465561600	48.34
17	128.46	12913.2	452305300	46.96
18	127.71	12837.7	219782100	22.82
19	127.69	12836.1	231754300	24.06
20	124.56	12521.0	185389400	19.25
21	120.18	12081.6	448073600	46.52

^{1}H and ^{13}C NMR of Compound **8**

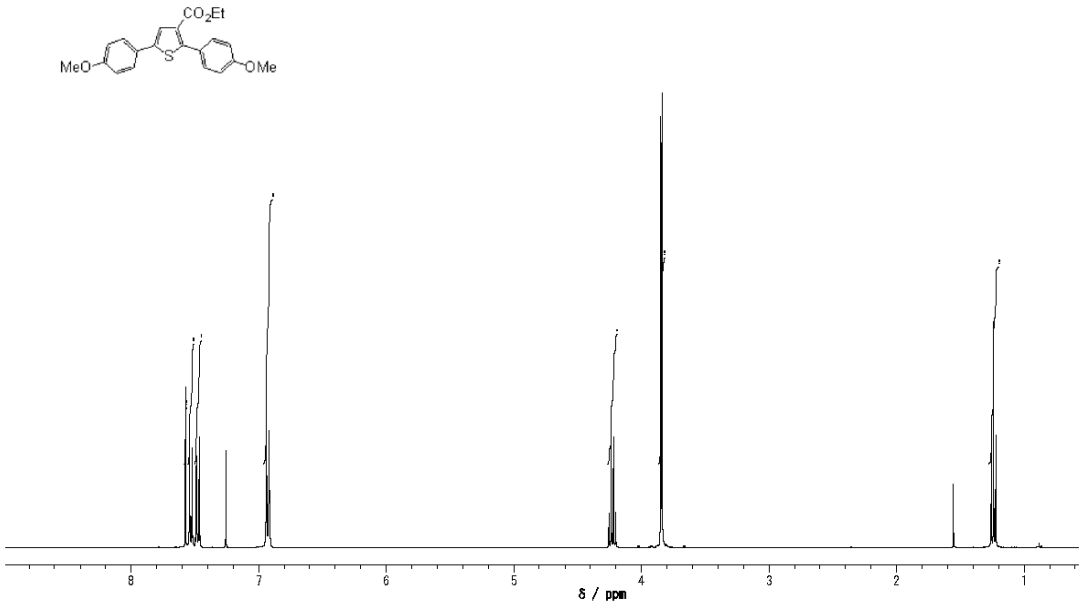




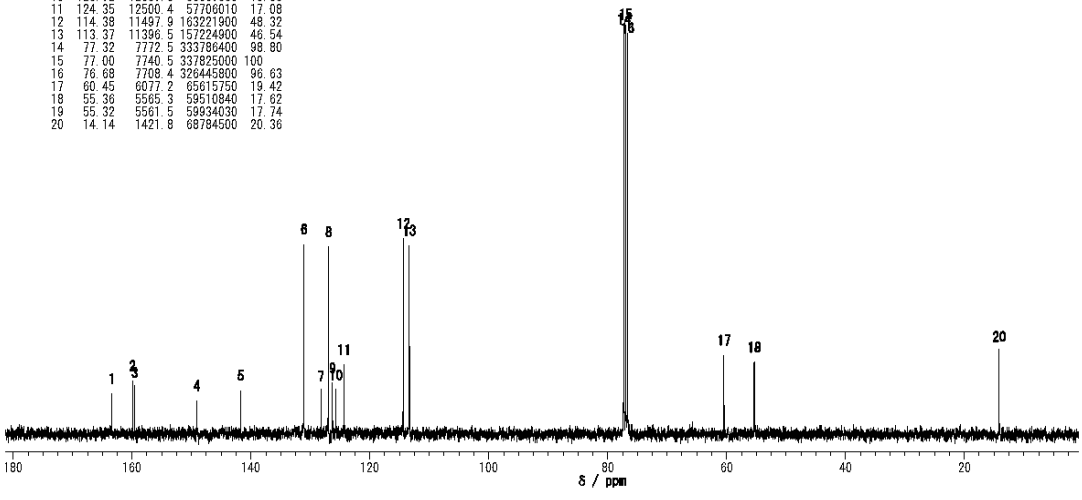
No.	ppm	Hz	Amp	Height
1	163.27	16412.8	43903230	6.97
2	149.71	15049.4	59823200	9.50
3	142.46	14320.8	86408830	13.72
4	133.38	13408.3	61078430	9.70
5	133.34	13403.8	83454310	13.25
6	129.78	13045.9	629766200	100
7	129.01	12968.9	531596700	84.41
8	128.96	12963.5	80539420	12.79
9	128.58	12925.4	222852900	35.39
10	128.01	12868.2	197807900	31.41
11	127.92	12859.0	604855700	96.04
12	125.72	12637.8	373413800	59.29
13	125.42	12608.0	278563900	44.23
14	77.32	7772.5	568678100	90.30
15	77.00	7740.5	574992600	91.30
16	76.68	7708.4	561258000	89.12
17	60.57	6088.7	191081100	30.34
18	14.02	1409.6	245524700	38.99



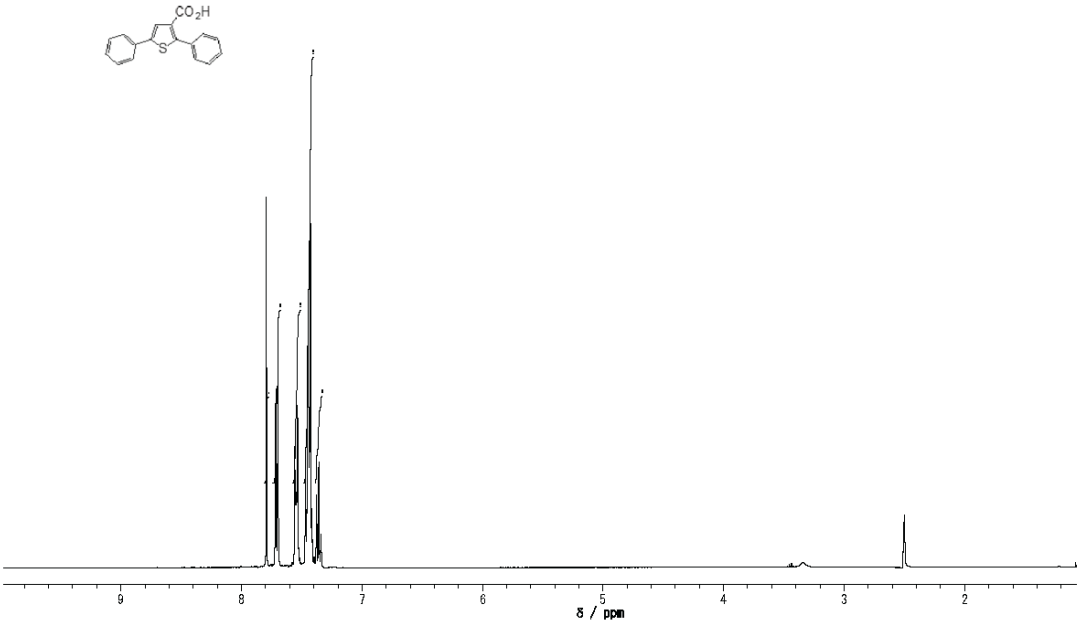
^{1}H and ^{13}C NMR of Compound **9**



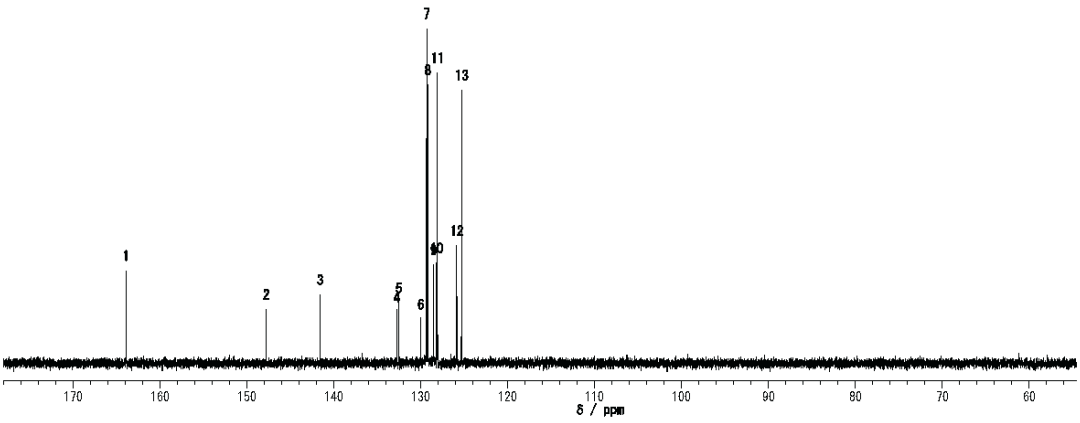
No.	ppm	Hz	Amp.	Height
1	163.41	16426.5	33404620	9.89
2	159.92	16076.3	44354460	13.13
3	159.51	16035.1	38567400	11.42
4	149.09	14986.9	27514130	8.14
5	141.73	14247.6	36064480	10.68
6	131.07	13175.6	158180400	46.82
7	128.21	12888.8	35648050	10.55
8	126.97	12763.6	156224500	46.24
9	126.29	12695.7	42467140	12.57
10	125.72	12637.8	36667350	10.85
11	124.35	12500.4	57706010	17.08
12	114.38	11497.9	163221900	48.32
13	113.37	11396.5	157224900	46.54
14	77.32	7772.5	333786400	98.80
15	77.00	7740.5	337825000	100
16	76.68	7709.4	326445800	96.63
17	60.45	6077.2	65615750	19.42
18	55.36	5565.3	59510840	17.62
19	55.32	5561.5	59934030	17.74
20	14.14	1421.8	68784500	20.36



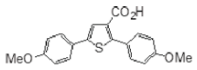
^{1}H and ^{13}C NMR of Compound **10**

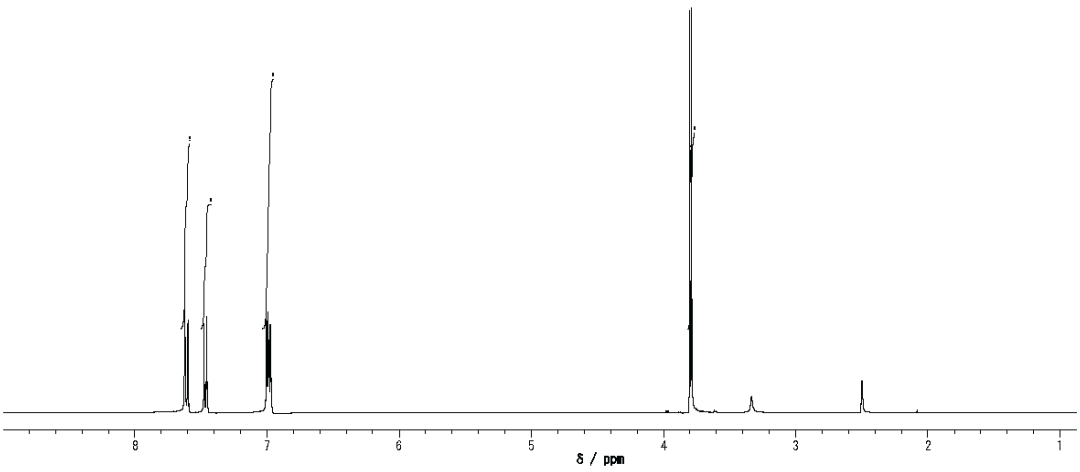


No.	ppm	Hz	Amp.	Height
1	163.92	16477.9	176295800	27.53
2	147.76	14853.6	102747600	16.04
3	141.56	14230.2	130635000	20.40
4	132.77	13346.8	96530330	15.07
5	132.53	13322.3	115039400	17.96
6	129.98	13066.8	83396770	13.02
7	129.32	12999.6	640462600	100
8	129.17	12985.1	532248600	83.10
9	128.53	12920.3	187167300	29.22
10	128.16	12883.7	191298100	29.87
11	128.06	12873.7	555610600	86.75
12	125.84	12650.2	224651700	35.08
13	125.29	12594.5	522083600	81.52

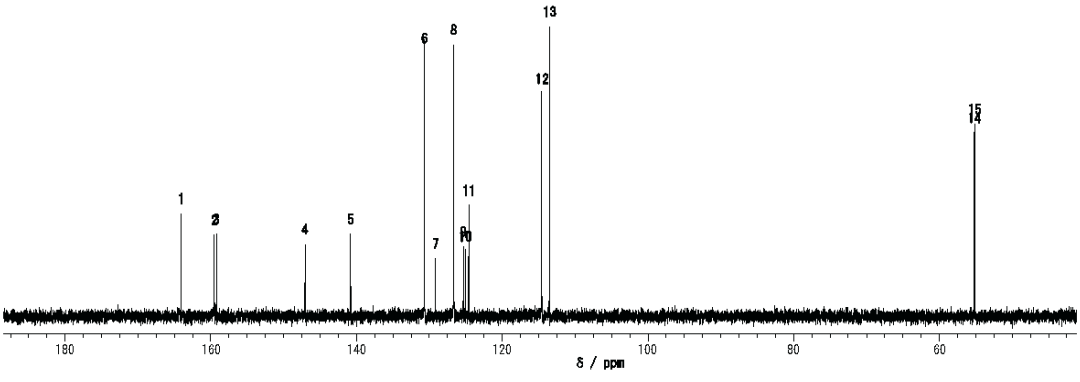


^{1}H and ^{13}C NMR of Compound **11**

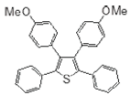


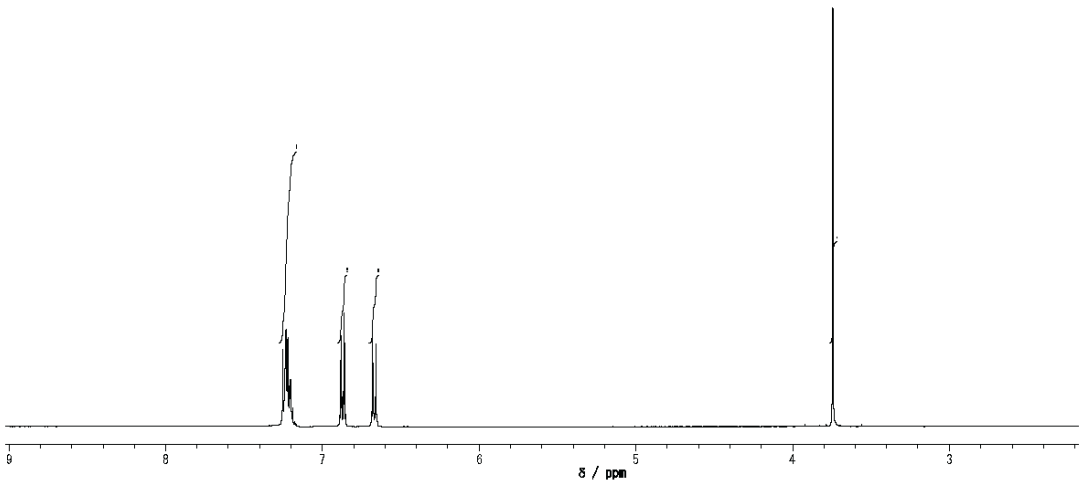


No.	ppm	Hz	Amp.	Height
1	164.08	16494.7	193930200	22.98
2	159.50	16033.8	152897500	18.12
3	159.22	16005.6	155534400	18.43
4	147.05	14781.9	135701100	16.08
5	140.81	14154.7	155127100	18.38
6	130.65	13133.9	498198400	59.03
7	129.15	12982.8	108524100	12.86
8	126.62	12728.8	514216000	60.93
9	125.32	12598.3	131272400	15.55
10	125.10	12576.2	121618100	14.41
11	124.61	12526.6	209420000	24.81
12	114.58	11516.5	421195500	49.91
13	113.52	11411.9	548336500	64.97
14	55.19	5548.0	347541100	41.18
15	55.17	5545.7	363483800	43.07

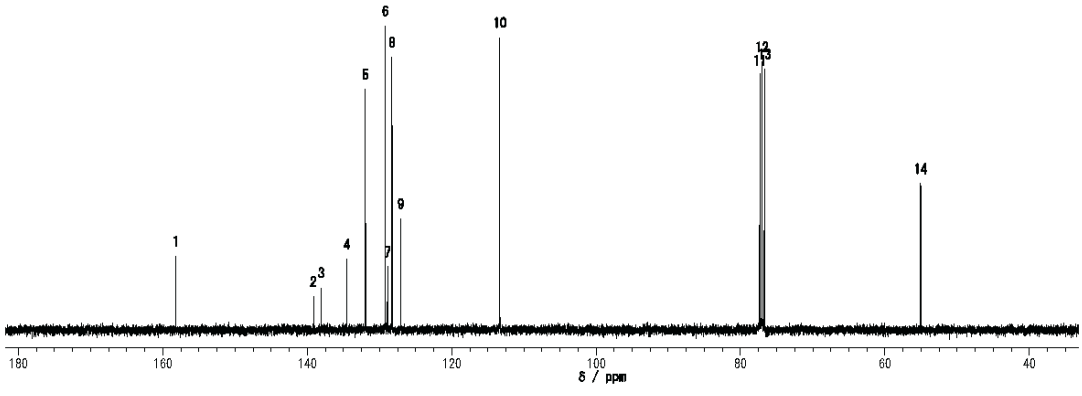


^{1}H and ^{13}C NMR of Compound **12**

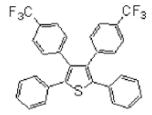


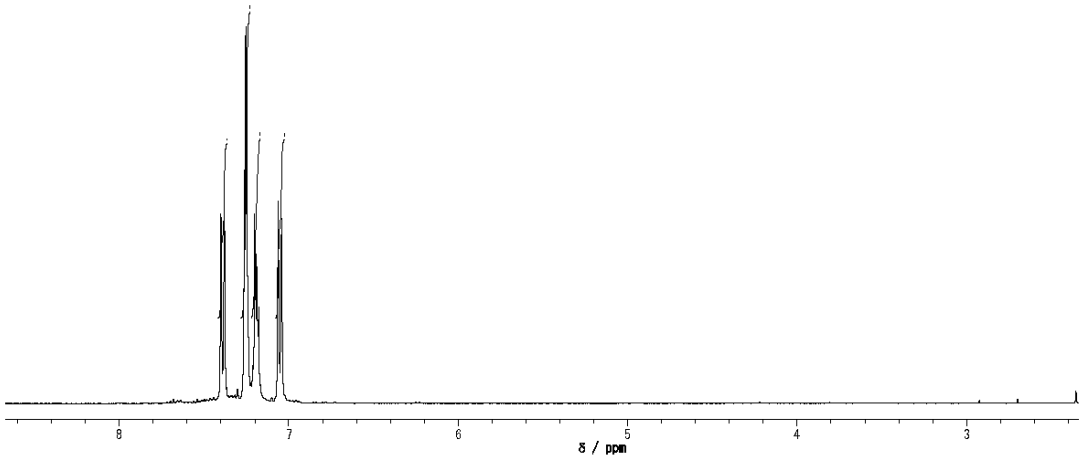


No.	ppm	Hz	Amp.	Height
1	158.18	15900.9	164051600	24.21
2	139.14	13987.4	74038950	10.93
3	138.05	13877.5	93331520	13.78
4	134.50	13520.5	157241600	23.21
5	131.93	13261.8	535652200	79.06
6	129.17	12984.9	677525100	100
7	128.86	12953.6	140834200	20.79
8	128.27	12894.9	606844700	89.57
9	127.05	12771.3	247581900	36.54
10	113.33	11392.6	651386900	96.14
11	77.32	7772.5	566697500	83.64
12	77.00	7740.5	597056100	88.12
13	76.68	7708.4	581161700	85.78
14	55.04	5532.5	325888300	48.10

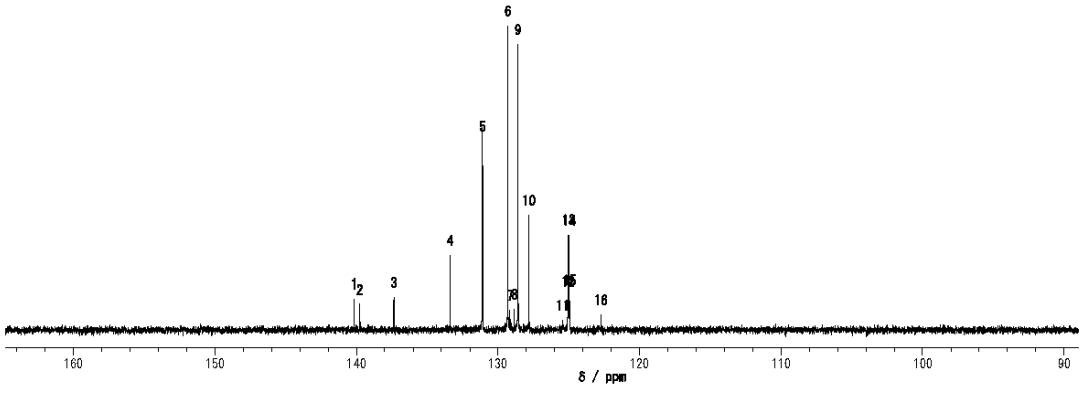


^{1}H and ^{13}C NMR of Compound **13**

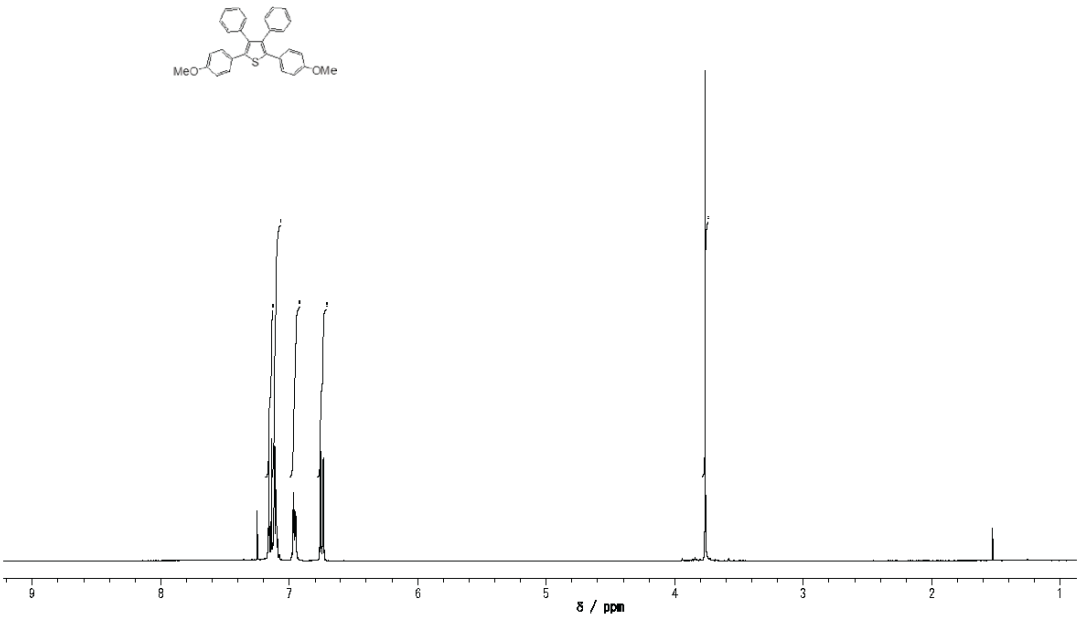




No.	ppm	Hz	Amp.	Height
1	140.16	14089.6	123325400	10.16
2	139.77	14050.0	100465300	8.28
3	137.37	13808.9	129088100	10.64
4	133.37	13406.8	298559000	24.60
5	131.10	13178.7	752327500	61.98
6	129.31	12998.6	1213729000	100
7	129.16	12983.4	77410300	6.38
8	128.84	12951.3	82471960	6.79
9	128.58	12925.4	1138976000	93.84
10	127.81	12848.3	456565900	37.62
11	125.44	12610.3	36812180	3.03
12	125.07	12572.9	132743800	10.94
13	125.03	12569.1	379228800	31.24
14	125.00	12565.3	376618700	31.03
15	124.96	12561.5	138720100	11.43
16	122.74	12338.7	58886070	4.85

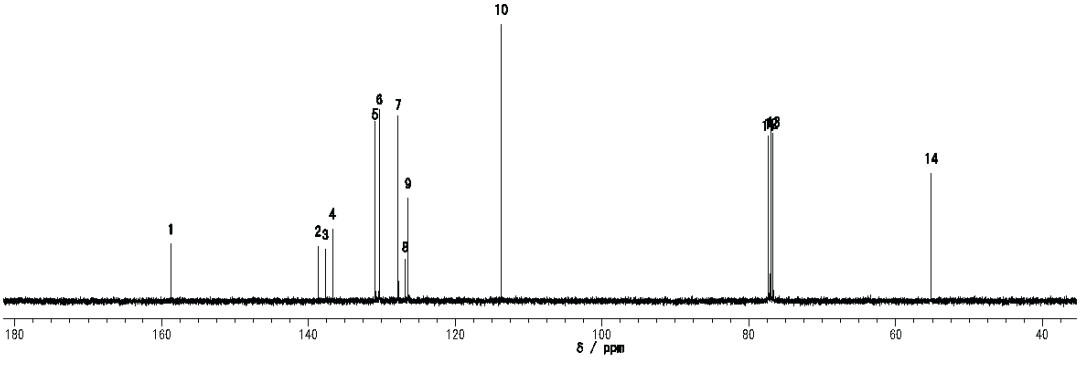


^{1}H and ^{13}C NMR of Compound **14**



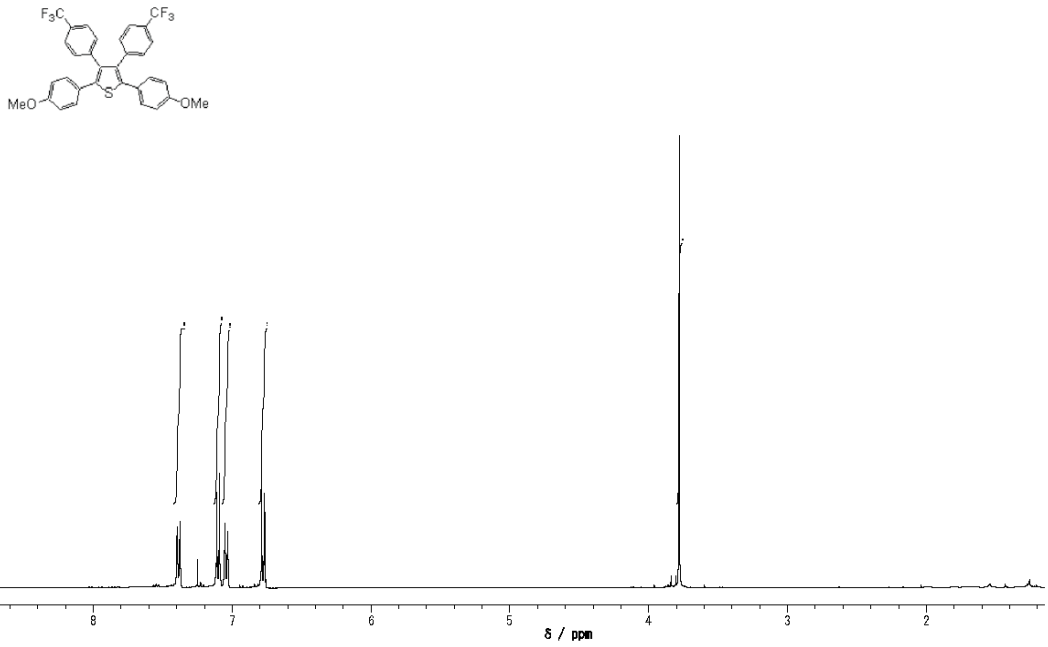
7

No.	ppm	Hz	Amp.	Height
1	158.75	15958.8	191382800	20.53
2	138.70	13943.2	184724700	19.81
3	137.69	13840.9	172437600	18.50
4	136.71	13743.3	243296600	26.10
5	130.89	13157.3	578735000	62.08
6	130.33	13101.6	627850900	67.35
7	127.79	12846.0	610195000	65.45
8	126.82	12748.4	131541900	14.11
9	126.43	12709.5	344658500	36.97
10	113.73	11433.1	932267600	100
11	77.32	7772.5	540829100	58.01
12	77.01	7741.2	547688300	58.75
13	76.69	7709.2	553399400	59.36
14	55.18	5547.0	429077300	46.03

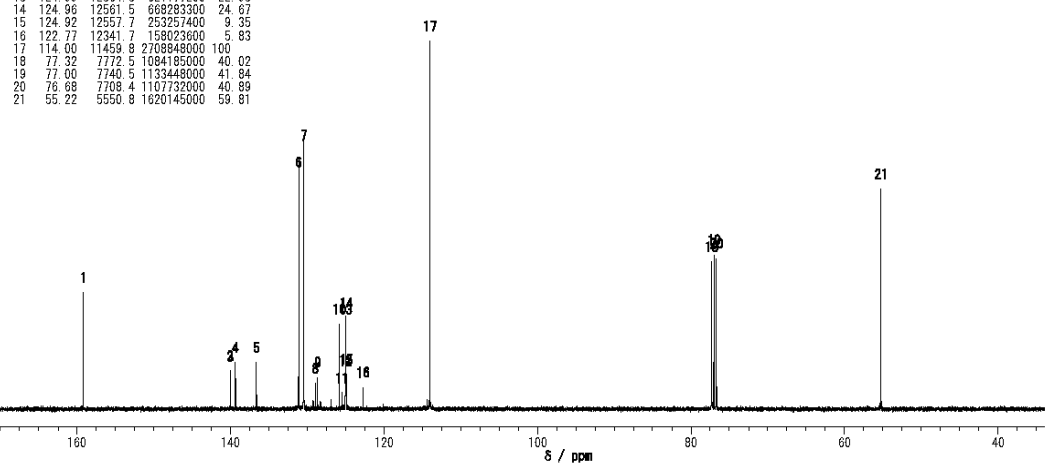


7

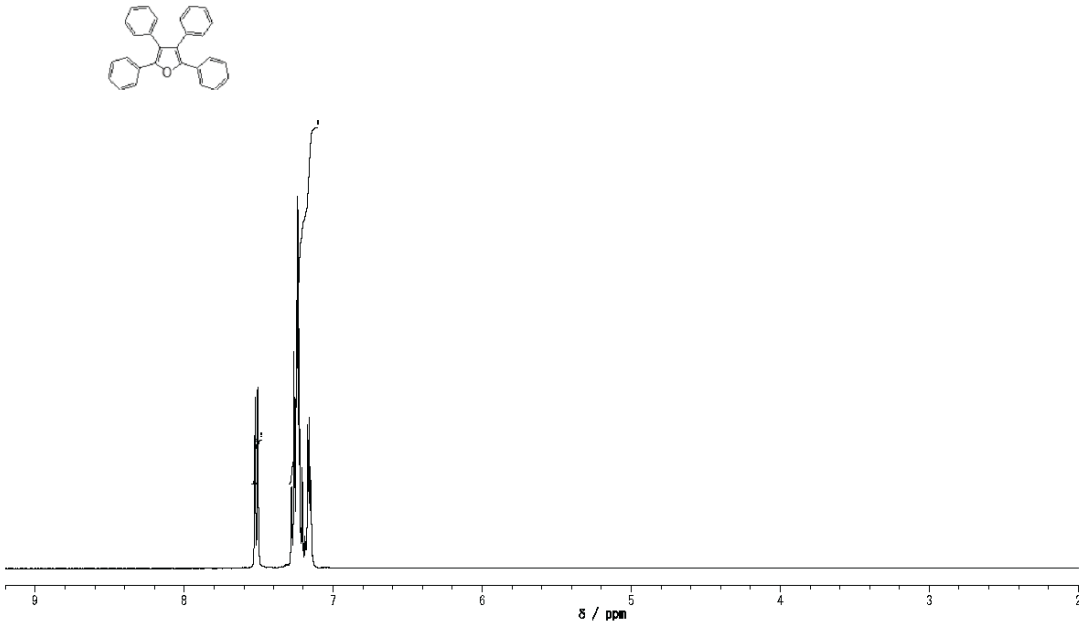
^{1}H and ^{13}C NMR of Compound **15**



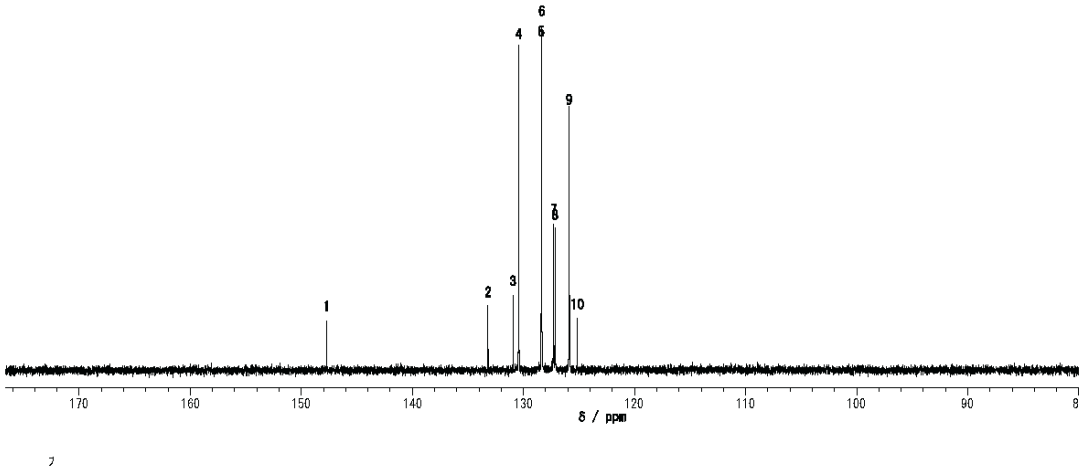
No.	ppm	Hz	Amp.	Height
1	159.20	16003.9	855243000	31.57
2	140.05	14079.0	280729600	10.36
3	140.04	14077.4	274982500	10.15
4	139.37	14010.3	341014700	12.59
5	136.62	13734.1	341655700	12.61
6	131.13	13181.7	1706289000	62.99
7	130.48	13116.1	1903447000	70.27
8	128.97	12964.3	187879600	6.94
9	128.65	12932.3	231471200	8.55
10	125.83	12649.2	624688200	23.06
11	125.48	12614.1	115970300	4.28
12	125.03	12568.3	246890900	9.11
13	124.99	12564.5	621177200	22.93
14	124.96	12561.5	668283300	24.67
15	124.92	12557.7	253257400	9.35
16	122.77	12341.7	158023600	5.83
17	114.00	11459.8	2708848000	100
18	77.32	7772.5	1084185000	40.02
19	77.00	7740.5	1133448000	41.84
20	76.68	7708.4	1107732000	40.89
21	55.22	5550.8	1620145000	59.81



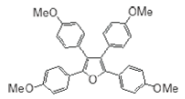
^{1}H and ^{13}C NMR of Compound **17a**

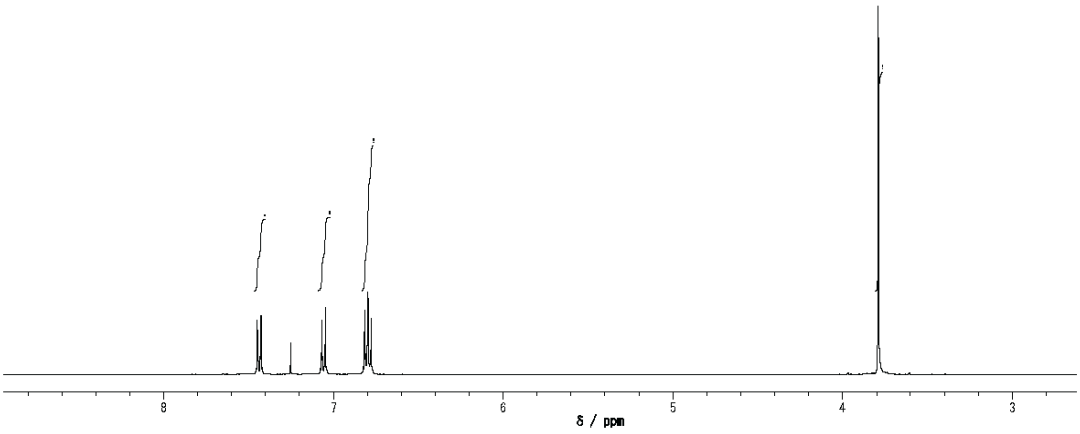


No.	ppm	Hz	Amp.	Height
1	147.73	14850.3	129737600	13.24
2	133.19	13389.3	167989200	17.14
3	130.92	13160.4	197429200	20.14
4	130.42	13110.0	856513000	87.38
5	128.37	12904.0	862321700	87.97
6	128.34	12901.0	916233000	93.47
7	127.30	12796.5	388944300	39.68
8	127.15	12782.0	373946400	38.15
9	125.87	12653.0	67991850	69.36
10	125.13	12579.0	135394000	13.81

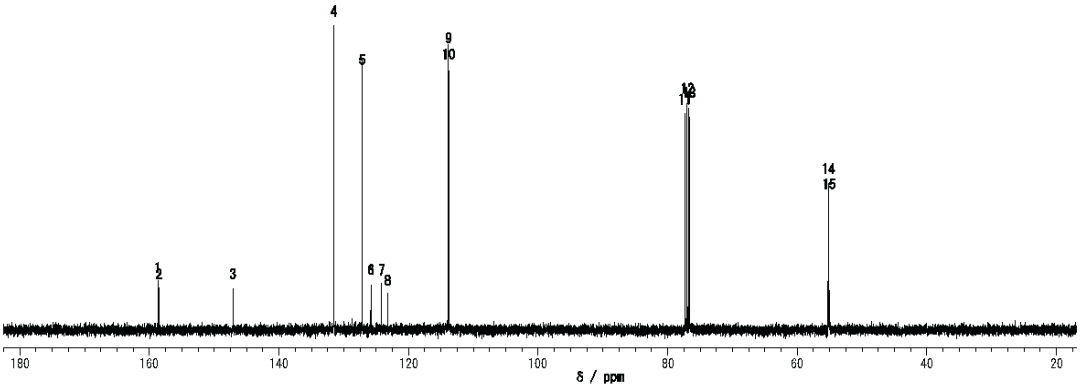


^{1}H and ^{13}C NMR of Compound **17d**

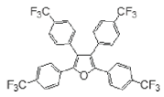


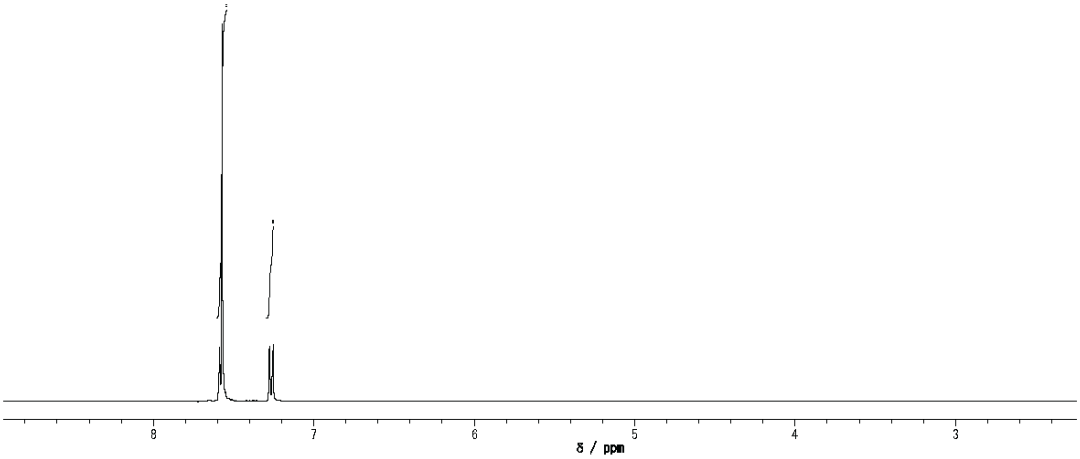


No.	ppm	Hz	Amp	Height
1	158.72	15955.8	81986510	15.55
2	158.53	15936.7	70783280	13.42
3	147.10	14787.7	71262330	13.51
4	131.53	13222.2	527294700	100
5	127.14	12781.2	442710100	83.96
6	125.80	12646.2	77994020	14.79
7	124.15	12480.6	78157090	14.82
8	123.24	12388.3	60277570	11.43
9	113.83	11443.0	480628700	91.15
10	113.82	11441.5	452491100	85.81
11	77.32	7772.5	374463200	71.02
12	77.00	7740.5	392985300	74.53
13	76.68	7708.4	385402400	73.09
14	55.22	5550.8	253591100	48.09
15	55.10	5538.6	227155300	43.08



^{1}H and ^{13}C NMR of Compound **17e**





No.	ppm	Hz	Amp.	Height
1	147.35	14812.5	204315000	13.84
2	135.30	13601.4	161258600	10.92
3	132.67	13336.2	170992300	11.58
4	132.65	13335.1	174869800	11.85
5	130.26	13094.8	1344570000	91.09
6	129.49	13016.9	101352200	6.87
7	129.16	12984.1	98762910	6.69
8	129.11	12979.2	105169900	7.12
9	128.79	12946.8	107643300	7.29
10	127.64	12831.2	63794620	4.32
11	127.62	12829.3	62011030	4.20
12	125.80	12645.8	1475954000	99.99
13	125.44	12609.9	155406300	10.53
14	125.40	12606.1	503599900	34.12
15	125.36	12602.3	542476200	36.75
16	125.33	12598.5	208593600	14.13
17	125.27	12593.1	161264800	10.93
18	125.24	12589.3	470497900	31.87
19	125.20	12585.5	475823600	32.24
20	125.16	12582.1	175467500	11.89
21	124.93	12558.8	136074300	9.22
22	124.92	12557.3	140484800	9.52
23	124.89	12554.6	218171000	14.78
24	122.23	12286.8	129604700	8.78
25	122.21	12285.7	132306400	8.96