Supporting Information

A Repetitive One-Step Method for Oligoarene Synthesis Using Catalyst-Controlled Chemoselective Cross-Coupling

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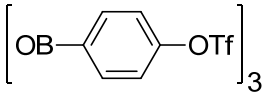
Experimental

General: Melting points were uncorrected. $CDCl_3$ and DMSO-d_6, were used as solvents for ^1H and ^{13}C spectra. For ^1H NMR, tetramethylsilane (TMS) ($\delta = 0$) in $CDCl_3$, DMSO ($\delta = 2.49$) in DMSO-d_6, and THF ($\delta = 1.73$) in THF-d_6 served as internal standards. For ^{13}C NMR, $CDCl_3$ ($\delta = 77.00$), DMSO-d_6 ($\delta = 39.50$), and THF-d_6 ($\delta = 25.2$) served as internal standards. Preparative thin-layer chromatography (TLC) was carried out using silica gel 60 F_{254}, 0.5 mm coated glass plates. Gel permeation chromatography (GPC) was performed using JALGEL-1H and 2H (Japan Analytical Industry). Boronic acid **17** was prepared according to a reported procedure.[1]

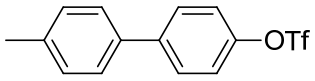
Procedures and Spectral Data



(**2**)

Under Ar, isopropylmagnesium chloride (a 2.0 M solution in Et_2O, 25.7 mL, 51.4 mmol) was added to a solution of 4-iodophenyl trifluoromethanesulfonate (15.1 g, 42.8 mmol) in THF (85 mL) at −20 °C for 10 min. The reaction mixture was warmed to −10 °C, stirred for 30 min, and then cooled to −78 °C. Triisopropyl borate (11.7 mL, 51.4 mmol) was added for 1 min, and the mixture was warmed to rt and stirred for 3 h. A cold aqueous HCl (1 M, 100 mL) was added. After 10 min, THF was removed in vacuo. The mixture was extracted with Et_2O, washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by recrystallization from CH_2Cl_2/hexane (1/10, twice) to give **2** (5.61 g, 52%) as a white solid.
Mp. 221.0-223.0 °C; ^1H NMR (500 MHz, DMSO-d_6 with 1 drop of D_2O) δ 7.35 (2H, d, J = 8.6 Hz), 7.86 (2H, d, J = 8.6 Hz) ppm; ^{13}C NMR (126 MHz, DMSO-d_6 with 1 drop of D_2O) δ 118.7 (q, J = 320.3 Hz),

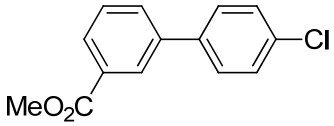
120.9, 136.9, 151.29 ppm (one carbon connected to the boron was not observed.); IR (ATR) 1404, 1348, 1211, 1136, 895 cm^{-1}; HRMS (ESI) m/z calcd for $C_7H_5BF_3O_5S$ (monomeric boronic acid form $[M-H]^-$) 268.9981; found: 268.9897; Anal. calcd. for $C_7H_4BF_3O_4S$: C, 33.37; H, 1.60, found: C, 33.73; H, 1.97.



(**3**)

4-Chlorotoluene (46.6 mg, 0.368 mmol), boronic anhydride **2** (188 mg, 0.745 mmol), tris(dibenzylideneacetone)dipalladium(0) (10.3 mg, 0.0112 mmol), tri-*t*-butylphosphonium tetrafluoroborate (6.6 mg, 0.023 mmol), and K_3PO_4 (237 mg, 1.12 mmol) were charged into a sealable tube under Ar, and then THF/H_2O (4/1) (0.40 mL) was added. The tube was sealed, and the mixture was stirred at 50 °C for 22 h. At rt, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (1/5)) to give **3** (98.4 mg, 85%) as a white solid.

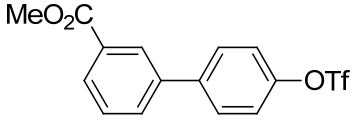
Mp. 70.0-71.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.43 (3H, s), 7.29 (2H, d, *J* = 8.6 Hz), 7.34 (2H, d, *J* = 8.6 Hz), 7.47 (2H, d, *J* = 8.6 Hz), 7.64 (2H, d, *J* = 8.6 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.2, 118.9 (q, *J* = 320.3 Hz), 121.7, 127.1, 128.7, 129.8, 136.5, 138.1, 141.7, 148.8 ppm; IR (ATR) 1423, 1215, 1138, 810, 615 cm^{-1}; MS (FAB) m/z 317 (M^+); Anal. calcd. for $C_{14}H_{11}F_3O_3S$: C, 53.16; H, 3.51, found: C, 53.24; H, 3.55.



(**6**)

Methyl 3-(trifluoromethanesulfonyloxy)benzoate[2] (2.78 g, 9.79 mmol), 4-chlorophenylboronic acid (1.70 g, 10.9 mmol), palladium(II) acetate (66.1 mg, 0.294 mmol), 1,1'-bis(diphenylphosphino)ferrocene (164 mg, 0.296 mmol), and KF (1.72 g, 29.5 mmol) were charged into a flask under Ar, and then THF (9.8 mL) was added. The mixture was stirred at rt for 24 h. H_2O (20 mL) was added, and the mixture was extracted with AcOEt (50 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (CH_2Cl_2/hexane (1/5 to 1/3)) to give **6** (2.09 g, 93%) as an oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.94 (3H, s), 7.41 (2H, d, *J* = 8.6 Hz), 7.49 (1H, t, *J* = 8.0 Hz), 7.53 (2H, d, *J* = 8.6 Hz), 7.72 (1H, d, *J* = 8.6 Hz), 8.02 (1H, dt, *J* = 1.1, 8.0 Hz), 8.23 (1H, t, *J* = 1.7 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 128.2, 128.5, 128.8, 129.1, 129.2, 130.9, 131.4, 134.0, 138.6, 140.3, 167.0 ppm; IR (neat) 2951, 1730, 1440, 1308, 1111, 754 cm^{-1}; MS (FAB) m/z 247 (M^+); Anal. calcd. for $C_{14}H_{11}ClO_2$: C, 68.16; H, 4.49; Cl, 14.37, found: C, 68.32; H, 4.31; Cl, 14.20.

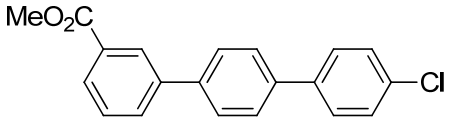


(**8**)

Methyl 3-chlorobenzoate (88.4 mg, 0.518 mmol), boronic anhydride **2** (157 mg, 0.623 mmol), tris(dibenzylideneacetone)dipalladium(0) (7.2 mg, 0.0079 mmol), tri-*t*-butylphosphonium tetrafluoroborate

(4.6 mg, 0.016 mmol), and K_3PO_4 (334 mg, 1.57 mmol) were charged into a flask under Ar, and then THF/H_2O (4/1) (0.50 mL) was added. The mixture was stirred at rt for 22 h. H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (CH_2Cl_2/hexane (1/8)) to give **8** (140 mg, 75%) as a white solid.

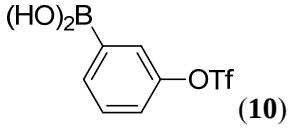
Mp. 29.2-32.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.94 (3H, s), 7.35 (2H, d, *J* = 8.6 Hz), 7.51 (1H, t, *J* = 7.7 Hz), 7.65 (2H, d, *J* = 9.2 Hz), 7.72 (1H, d, *J* = 7.5 Hz), 8.04 (1H, d, *J* = 8.1 Hz), 8.22 (1H, s) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.3, 118.9 (q, *J* = 320.3 Hz), 121.9, 128.4, 129.0, 129.18, 129.21, 131.0, 131.5, 139.6, 140.6, 149.3, 166.8 ppm; IR (neat) 2955, 1728, 1504, 1427, 1111, 887 cm^{-1}; MS (FAB) m/z 361 (M^+); Anal. calcd. for $C_{15}H_{11}F_3O_5S$: C, 50.00; H, 3.08, found: C, 49.83; H, 3.09.



(**9**)

Triflate **8** (0.149 g, 0.414 mmol), 4-chlorophenylboronic acid (97.2 mg, 0.622 mmol), palladium(II) acetate (5.8 mg, 0.026 mmol), 1,1'-bis(diphenylphosphino)ferrocene (14.0 mg, 0.0253 mmol), and KF (74.3 mg, 1.28 mmol) were charged into a flask under Ar, and then THF (0.41 mL) was added. The mixture was stirred at rt for 22 h. H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (1/6 to 1/5)) to give **9** (112 mg, 83%) as a white solid.

Mp. 155.5-157.2 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.96 (3H, s), 7.43 (2H, d, *J* = 8.6 Hz), 7.53 (1H, t, *J* = 7.4 Hz), 7.57 (2H, d, *J* = 8.0 Hz), 7.64 (2H, d, *J* = 8.6 Hz), 7.71 (2H, d, *J* = 8.6 Hz), 7.82 (1H, d, *J* = 8.6 Hz), 8.04 (1H, dt, *J* = 1.7, 7.5 Hz), 8.33 (1H, t, *J* = 1.8 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 127.6, 127.7, 128.2, 128.4, 128.6, 129.06, 129.11, 130.9, 131.5, 133.7, 139.0, 139.40, 139.43, 140.9, 167.1 ppm; IR (ATR) 1715, 1313, 1246, 810, 758 cm^{-1}; MS (FAB) m/z 323 (M^+); Anal. calcd. for $C_{20}H_{15}ClO_2$: C, 74.42; H, 4.68; Cl, 10.98, found: C, 74.56; H, 4.65; Cl, 11.19.

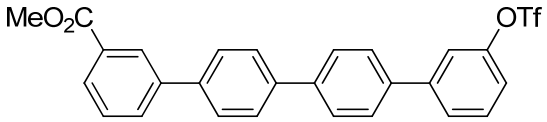


(**10**)

Under Ar, isopropylmagnesium chloride (a 2.0 M solution in Et_2O, 20.5 mL, 41.0 mmol) was added to a solution of 3-iodophenyl trifluoromethanesulfonate (12.0 g, 34.1 mmol) in THF (70 mL) at −20 °C for 40 min. The reaction mixture was warmed to −10 °C, stirred for 30 min, and then cooled to −78 °C. Triisopropyl borate (9.40 mL, 41.0 mmol) was added for 1 min, and the mixture was warmed to rt and stirred for 3 h. A cold aqueous HCl (2 M, 200 mL) was added. After 10 min, THF was removed in vacuo. The mixture was extracted with Et_2O, washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (1/5) to CH_2Cl_2/MeOH (20/1 to 10/1)) and by recrystallization from CH_2Cl_2/hexane/H_2O (10/100/1) to give **10** (3.08 g, 33%) as a white solid.

Mp. 105.0-106.0 °C; ^{1}H NMR (500 MHz, DMSO-d_6 with 1 drop of D_2O) δ 7.44 (1H, d, *J* = 8.0 Hz), 7.50

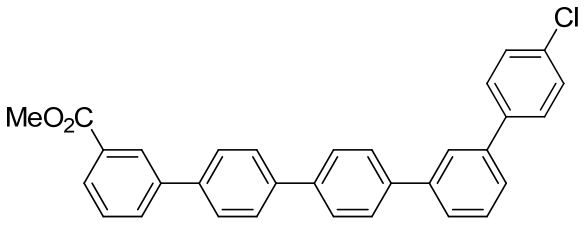
(1H, t, J = 8.0 Hz), 7,67 (1H, s), 7.81 (1H, d, J = 8.0 Hz) ppm; ^{13}C NMR (126 MHz, DMSO-d_6 with 1 drop of D_2O) δ 118.8 (q, J = 320.3 Hz), 123.6, 126.4, 130.8, 134.9, 149.7 ppm (one carbon connected to the boron was not observed.); IR (ATR) 3250, 1422, 1354, 1211, 910 cm^{-1}; HRMS (ESI) m/z calcd for $C_7H_5BF_3O_5S$ ($[M-H]^-$) 268.9981; found: 268.9908; Anal. calcd. for $C_7H_6BF_3O_5S$: C, 31.14; H, 2.24, found: C, 31.20; H, 2.07.



(**11**)

Chloride **9** (127 mg, 0.393 mmol), boronic acid **10** (161 mg, 0.596 mmol), tris(dibenzylideneacetone)dipalladium(0) (14.8 mg, 0.0162 mmol), tri-*t*-butylphosphonium tetrafluoroborate (9.4 mg, 0.032 mmol), and K_3PO_4 (253 mg, 1.19 mmol) were charged into a flask under Ar, and then THF/H_2O (4/1) (0.75 mL) was added. The mixture was stirred at 50 °C for 22 h. At rt, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (CH_2Cl_2/hexane (2/3)) and then by recrystallization from CH_2Cl_2/hexane (1/10) to give **11** (97.0 mg, 48%) as a white solid. The mother liquor was further purified by silica gel chromatography (CH_2Cl_2/hexane (2/1)) to give **11** (53.0 mg, total 75%).

Mp. 165.0-166.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.96 (3H, s), 7.27 (2H, dd, J = 1.7, 8.1 Hz), 7.53-7.56 (3H, m), 7.67 (3H, d, J = 8.0 Hz), 7.74-7.77 (5H, m), 7.85 (1H, dt, J = 2.3, 8.6 Hz), 8.04 (1H, dt, J = 1.2, 8.0 Hz), 8.34 (1H, t, J = 1.7 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 116.6 (q, J = 243.5 Hz), 119.95, 120.03, 127.0, 127.65, 127.74, 127.76, 127.78, 128.3, 128.6, 129.1, 130.7, 130.9, 131.5, 138.1, 139.5, 139.7, 140.6, 140.9, 143.5, 150.2, 167.1 ppm; IR (ATR) 1722, 1207, 1138, 758 cm^{-1}; MS (FAB) m/z 513 (M^+); Anal. calcd. for $C_{27}H_{19}F_3O_5S$: C, 63.28; H, 3.74, found: C, 63.48; H, 3.83.

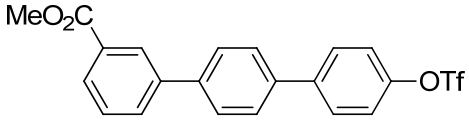


(**12**)

Triflate **11** (148 mg, 0.290 mmol), 4-chlorophenylboronic acid (69.5 mg, 0.444 mmol), palladium(II) acetate (4.1 mg, 0.018 mmol), 1,1'-bis(diphenylphosphino)ferrocene (10.1 mg, 0.0182 mmol), and KF (52.8 mg, 0.909 mmol) were charged into a flask under Ar, and then THF (0.43 mL) was added. The mixture was stirred at 50 °C for 20 h. At rt, H_2O (5 mL) and AcOEt (10 mL) were added. The resulting precipitate was collected on a sintered glass funnel and purified by silica gel chromatography (CH_2Cl_2/hexane (2/1)) to give **12** (74.9 mg) as a white solid. On the other hand, the organic layer of the mother liquor was washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (CH_2Cl_2/hexane (2/1)) to give **12** (24.5 mg, total 72%).

Mp. 238.0-239.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.94 (3H, s), 7.44 (2H, d, J = 8.0 Hz), 7.52-7.57 (3H, m),

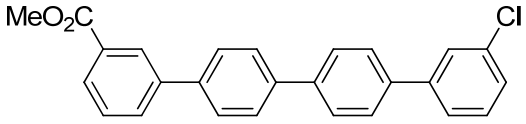
7.59 (2H, d, *J* = 8.6 Hz), 7.64-7.68 (1H, m), 7.73-7.77 (8H, m), 7.81 (1H, s), 7.85 (1H, dt, *J* = 1.2, 8.6 Hz), 8.04 (1H, dt, *J* = 1.2, 8.6 Hz), 8.35 (1H, t, *J* = 1.6 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 125.9, 126.2, 126.4, 127.58, 127.62, 127.7, 127.8, 128.2, 128.57, 128.61, 129.06, 129.09, 129.5, 130.9, 131.5, 133.7, 139.2, 139.7, 139.8, 140.1, 140.2, 140.7, 141.0, 141.5, 167.2 ppm; IR (ATR) 1722, 1246, 786, 756 cm^{-1}; MS (FAB) m/z 475 (M^+); Anal. calcd. for $C_{32}H_{23}ClO_2$: C, 80.92; H, 4.88; Cl, 7.46, found: C, 80.96; H, 4.92; Cl, 7.12.



(**13**)

Chloride **6** (127 mg, 0.520 mmol), boronic anhydride **2** (196 mg, 0.778 mmol), tris(dibenzylideneacetone)dipalladium(0) (7.4 mg, 0.0081 mmol), tri-*t*-butylphosphonium tetrafluoroborate (4.6 mg, 0.016 mmol), and K_3PO_4 (333 mg, 1.57 mmol) were charged into a flask under Ar, and then THF/H_2O (4/1) (0.50 mL) was added. The mixture was stirred at rt for 22 h. Then, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (CH_2Cl_2/hexane (1/3)) to give **13** (209 mg, 93%) as a white solid.

Mp. 105.0-106.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.96 (3H, s), 7.37 (2H, d, *J* = 8.6 Hz), 7.53 (1H, t, *J* = 8.0 Hz), 7.64 (2H, d, *J* = 8.6 Hz) , 7.68 (2H, d, *J* = 8.6 Hz), 7.72 (2H, d, *J* = 8.0 Hz), 7.82 (1H, d, *J* = 8.1 Hz), 8.05 (1H, d, *J* = 7.5 Hz), 8.33 (1H, s) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 49.3, 118.7 (q, *J* = 322.0 Hz), 121.7, 127.6, 127.7, 128.1, 128.6, 128.7, 129.0, 130.8, 131.3, 138.5, 139.7, 140.5, 140.9, 149.0, 166.9 ppm; IR (ATR) 2955, 1728, 1418, 1308, 1211, 1111, 887 cm^{-1}; MS (FAB) m/z 437 (M^+); Anal. calcd. for $C_{21}H_{15}F_3O_5S$: C, 57.80; H, 3.46, found: C, 58.06; H, 3.59.

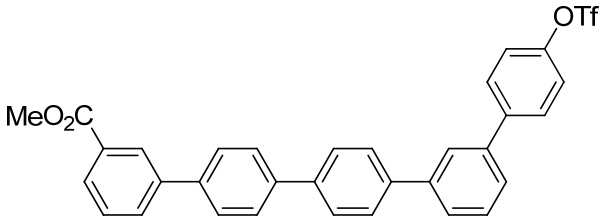


(**15**)

Triflate **13** (186 mg, 0.426 mmol), 3-chlorophenylboronic acid **14** (102 mg, 0.652 mmol), palladium(II) acetate (2.9 mg, 0.013 mmol), 1,1'-bis(diphenylphosphino)ferrocene (7.2 mg, 0.013 mmol), and KF (75.0 mg, 1.29 mmol) were charged into a flask under Ar, and then THF (0.43 mL) was added. The mixture was stirred at 50 °C for 22 h. Then, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (CH_2Cl_2) to give **15** (169 mg, 99%).

Mp. 166.0-167.2 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.96 (3H, s), 7.34 (1H, ddd, *J* = 8.0, 2.3, 1.1 Hz), 7.39 (1H, t, *J* = 7.5 Hz), 7.52 (1H, dt, *J* = 1.2, 8.1 Hz), 7.54 (1H, t, *J* = 7.5 Hz), 7.63-7.68 (3H, m), 7.72-7.75 (6H, m), 7.83 (1H, dt, *J* = 1.7, 7.5 Hz), 8.05 (1H, dt, *J* = 1.2, 8.1 Hz), 8.34 (1H, t, 1.8) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 125.3, 127.2, 127.5, 127.60, 127.64, 127.7, 128.2, 128.6, 129.1, 130.2, 130.9, 131.5, 134.8, 139.0, 139.3, 139.9, 140.1, 141.0, 142.6, 167.2 ppm (one carbon signal is overlapped with another.); IR (ATR) 1719, 1244, 787, 756 cm^{-1}; MS (FAB) m/z 399 (M^+); Anal. calcd. for $C_{26}H_{19}ClO_2$: C, 78.29; H, 4.80;

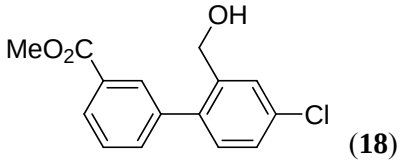
Cl, 8.89, found: C, 78.43; H, 4.95; Cl, 8.75.



(**16**)

Chloride **15** (154 mg, 0.387 mmol), boronic anhydride **2** (146 mg, 0.579 mmol), tris(dibenzylideneacetone)dipalladium(0) (11.0 mg, 0.0120 mmol), tri-*t*-butylphosphonium tetrafluoroborate (6.8 mg, 0.023 mmol), and K_3PO_4 (249 mg, 1.17 mmol) were charged into a sealable tube under Ar, and then THF/H_2O (4/1) (1.0 mL) was added. The tube was sealed, and the mixture was stirred at 50 ^oC for 22 h. Then, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (CH_2Cl_2/hexane (1/1)) to give **16** (216 mg, 95%) as a white solid.

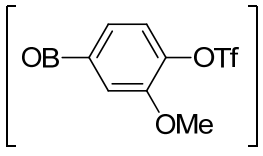
Mp. 192.5-193.0 ^oC; ^1H NMR (500 MHz, $CDCl_3$) δ 3.98 (3H, s) , 7.39 (2H, d, *J* = 9.2 Hz), 7.52-7.56 (3H, m), 7.71 (2H, d, *J* = 8.6 Hz) , 7.75 (9H, m), 7.82 (1H, s), 7.85 (1H, dt, *J* = 1.2, 8.6 Hz), 8.06 (1H, dt, *J* = 1.2, 8.1 Hz), 8.37 (1H, t, *J* = 1.2 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 118.9 (q, *J* = 320.3 Hz), 121.8, 126.1, 126.4, 126.9, 127.6, 127.7, 127.8, 128.2, 128.6, 129.08, 129.13, 129.7, 131.0, 131.5, 139.3, 139.85, 139.94, 140.0, 140.1, 141.0, 141.6, 141.7, 149.2, 167.2 ppm (one carbon signal is overlapped with another.); IR (ATR) 1719, 1425, 1211, 1138, 790, 758 cm^{-1}; MS (FAB) m/z 589 (M^+); Anal. calcd. for $C_{33}H_{23}F_3O_5S$: C, 67.34; H, 3.94, found: C, 67.43; H, 4.00.



(**18**)

Triflate **4** (169 mg, 0.593 mmol), boronic acid **17** (150 mg, 0.891 mmol), palladium(II) acetate (4.0 mg, 0.0178 mmol), 1,1'-bis(diphenylphosphino)ferrocene (9.9 mg, 0.0179 mmol), and KF (105 mg, 1.81 mmol) were charged into a flask under Ar, and then THF (0.59 mL) was added. The mixture was stirred at rt for 22 h. H_2O (10 mL) was added, and the mixture was extracted with AcOEt (20 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (3/5)) and by GPC (chloroform) to give **18** (158 mg, 96%) as a white solid.

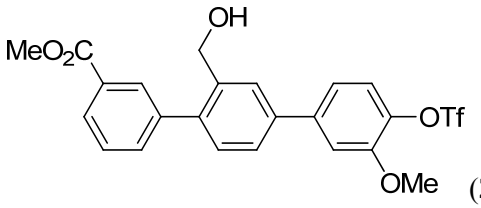
Mp. 49.3-50.5 ^oC; ^1H NMR (500 MHz, $CDCl_3$) δ 2,24 (1H, s), 3.90 (3H, s), 4.53 (2H, d, *J* = 3.4 Hz), 7.17 (1H, d, *J* = 8.0 Hz), 7.30 (1H, dd, *J* = 1.7, 8.6 Hz), 7.46-7.52 (2H, m), 7.58 (1H, d, *J* = 2.3 Hz), 7.97 (1H, t, *J* = 1.7 Hz), 8.02 (1H, dt, *J* = 1.7, 6.9 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 62.5, 127.8, 128.3, 128.6, 128.8, 130.2, 130.4, 131.3, 133.6, 134.1, 138.3, 139.9, 140.0, 167.1 ppm; IR (ATR) 3435, 2951, 1726, 1307, 1246, 756 cm^{-1}; MS (FAB) m/z 277 (M^+); Anal. calcd. for $C_{15}H_{13}ClO_3$: C, 65.11; H, 4.74, found: C, 65.09; H, 4.74.



(**19**)

Under Ar, isopropylmagnesium chloride (a 2.0 M solution in Et_2O, 23.0 mL, 46.0 mmol) was added to a solution of 4-iodo-2-methoxyphenyl trifluoromethanesulfonate (14.5 g, 37.9 mmol) in THF (110 mL) at −20 °C for 15 min. The reaction mixture was warmed to −10 °C, stirred for 30 min, and then cooled to −78 °C. Triisopropyl borate (10.6 mL, 46.0 mmol) was added for 1 min, and the mixture was warmed to rt and stirred for 3 h. A cold aqueous HCl (1 M, 500 mL) was added. After 10 min, THF was removed in vacuo. The mixture was extracted with AcOEt, washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by recrystallization from CH_2Cl_2/hexane (1/10, three times) to give **19** (4.47 g, 42%) as a white solid.

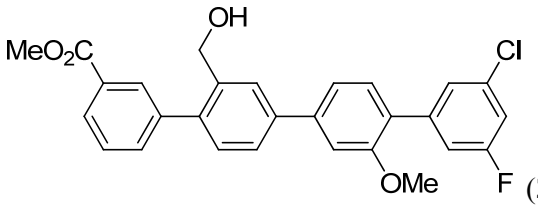
Mp. 193.4-198.0 °C; ^{1}H NMR (500 MHz, DMSO-d_6 with 1 drop of D_2O) δ 3.88 (3H, s), 7.35 (1H, d, J = 8.0 Hz), 7.44 (1H, dd, J = 1.2, 8.0 Hz), 7.63 (1H, s) ppm; ^{13}C NMR (126 MHz, DMSO-d_6 with 1 drop of D_2O) δ 57.1, 119.2 (q, J = 320.0 Hz), 119.8, 122.4, 128.0, 140.4, 150.9 ppm (one carbon connected to the boron was not observed.); IR (ATR) 1404, 1341, 1319, 1202, 1140, 1107, 874 cm^{-1}; HRMS (ESI) calcd for $C_8H_8BClF_3O_6S$ (monomeric boronic acid form $[M + Cl]^-$) 334.9782, found 334.9734; Anal. calcd. for $C_{24}H_{18}B_3F_9O_{15}S_3$: C, 34.07; H, 2.14, found: C, 34.09; H, 2.08.



(**20**)

Chloride **18** (358 mg, 1.30 mmol), boronic anhydride **19** (548 mg, 1.94 mmol), tris(dibenzylideneacetone)dipalladium(0) (59.3 mg, 0.0648 mmol), tri-*t*-butylphosphonium tetrafluoroborate (37.6 mg, 0.130 mmol), and KF (340 mg, 5.85 mmol) were charged into a flask under Ar, and then THF/H_2O (4/1) (3.25 mL) was added. The mixture was stirred at 50 °C for 22 h. Then, H_2O (10 mL) was added, and the mixture was extracted with AcOEt (20 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (1/3)) to give **20** (582 mg, 91%) as a white solid.

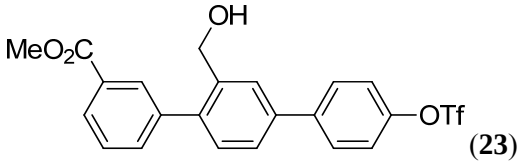
Mp. 87.4-89.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.72 (1H, t, J = 5.2 Hz), 3.94 (3H, s), 4.00 (3H, s), 4.68 (2H, d, J = 5.2 Hz), 7.22 (1H, dd, J = 2.2, 8.2 Hz), 7.26 (1H, d, J = 2.2 Hz), 7.30 (1H, d, J = 8.2 Hz), 7.38 (1H, d, J = 8.2 Hz), 7.53 (1H, t, J = 8.2 Hz), 7.54 (1H, dd, J = 2.2, 8.2 Hz), 7.61 (1H, dt, J = 1.6, 8.2 Hz), 7.79 (1H, d, J = 2.2 Hz), 8.06-8.08 (2H, m) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.3, 56.4, 63.0, 112.1, 118.8 (q, J = 320.1 Hz), 119.7, 122.8, 126.5, 127.3, 128.6, 128.7, 130.2, 130.4, 130.7, 133.6, 138.3, 138.7, 139.7, 140.0, 140.3, 142.3, 151.6, 167.0 ppm; IR (ATR) 1726, 1427, 1227, 1206, 1134, 1022, 1009, 883, 822 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{19}F_3NaO_7S$ ($[M + Na]^+$) 519.0696, found 519.0660.



(**22**)

Triflate **20** (192 mg, 0.386 mmol), boronic acid **21** (102 mg, 0.585 mmol), palladium(II) acetate (5.4 mg, 0.024 mmol), 1,1'-bis(diphenylphosphino)ferrocene (12.8 mg, 0.0231 mmol), and KF (70.1 mg, 1.21 mmol) were charged into a flask under Ar, and then THF (0.39 mL) was added. The mixture was stirred at 50 ^{o}C for 22 h. Then, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (Et_2O/hexane (1/1) then CH_2Cl_2) and by GPC (chloroform) to give **22** (138 mg, 75%).

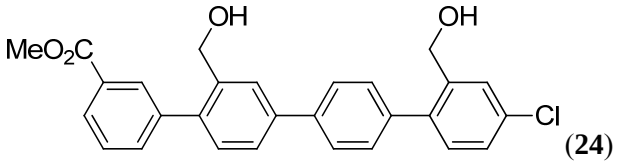
Mp. 127.6-134.4 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.72 (1H, t, *J* = 5.0 Hz), 3.93 (3H, s), 3.94 (3H, s), 4.70 (2H, d, *J* = 5.0 Hz), 7.06 (1H, dt, *J* = 2.0, 8.5 Hz), 7.22-7.24 (2H, m), 7.32 (1H, dd, *J* = 1.7, 7.9 Hz), 7.36-7.40 (3H, m), 7.53 (1H, t, *J* = 7.9 Hz), 7.64 (1H, d, *J* = 7.9 Hz), 7.64 (1H, d, *J* = 7.9 Hz), 7.86 (1H, d, *J* = 1.7 Hz), 8.07 (1H, dt, *J* = 1.7, 7.9 Hz), 8.10 (1H, t, *J* = 1.7 Hz) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 51.8, 55.2, 62.5, 109.7, 114.1 (d, *J* = 25.2 Hz), 114.5 (d, *J* = 21.6 Hz), 119.3, 125.0, 126.0, 126.7, 128.0, 128.1, 129.7, 129.8, 130.2, 130.4, 133.1, 133.9 (d, *J* = 12.0 Hz), 138.0, 139.1, 140.0, 140.7 (d, *J* = 6.0 Hz), 141.8, 156.2, 161.8 (d, *J* = 244.0 Hz), 166.4 ppm (two signals are overlapped); IR (ATR) 1722, 1302, 1221 cm^{-1}; HRMS (ESI) calcd for $C_{28}H_{22}ClFNaO_4$ ($[M + Na]^+$) 499.1083, found 499.1051.



(**23**)

Chloride **18** (106 mg, 0.383 mmol), boronic anhydride **2** (146 mg, 0.578 mmol), tris(dibenzylideneacetone)dipalladium(0) (10.8 mg, 0.0118 mmol), tri-*t*-butylphosphonium tetrafluoroborate (6.9 mg, 0.024 mmol), and KF (103 mg, 1.77 mmol) were charged into a flask under Ar, and then THF/H_2O (4/1) (0.40 mL) was added. The mixture was stirred at 50 ^{o}C for 22 h. Then, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (3/5)) to give **23** (141 mg, 79%) as a white solid.

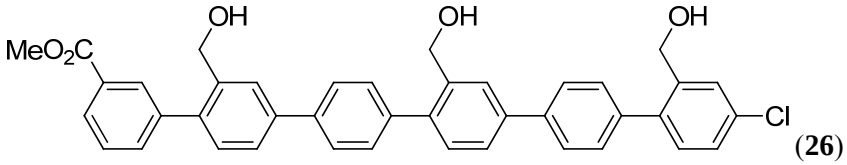
Mp. 79.8-80.6 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.75 (1H, t, *J* = 4.6 Hz), 3.94 (3H, s), 4.68 (2H, d, *J* = 5.2 Hz), 7.37 (3H, m), 7.51-7.57 (2H, m), 7.61 (1H, dd, *J* = 1.2, 7.5 Hz), 7.71 (2H, d, *J* = 8.6 Hz), 7.80 (1H, s), 8.06-8.08 (2H, m) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 52.4, 63.0, 118.9 (q, *J* = 320.3 Hz), 121.9, 126.5, 127.3, 128.6, 128.8, 129.0, 130.3, 130.5, 130.9, 133.7, 138.8, 139.2, 140.0, 140.4, 141.1, 149.2, 167.0 ppm; IR (ATR) 3204, 2369, 2320, 1717, 1226 cm^{-1}; HRMS (FAB) m/z calcd for $C_{22}H_{18}F_3O_6S$ ([M+H]+) 467.0776; found:467.0813.



(**24**)

Triflate **23** (168 mg, 0.360 mmol), boronic acid **17** (91.2 mg, 0.542 mmol), palladium(II) acetate (5.1 mg, 0.023 mmol), 1,1'-bis(diphenylphosphino)ferrocene (12.3 mg, 0.0222 mmol), and KF (67.5 mg, 1.16 mmol) were charged into a flask under Ar, and then THF (0.36 mL) was added. The mixture was stirred at 50 ^{o}C for 22 h. Then, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (1/1)) to give **24** (144 mg, 87%).

Mp. 51.2-52.0 ^{o}C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 3.85 (3H, s), 4.44 (4H, d, J = 4.6 Hz), 5.28 (1H, t, J = 5.2 Hz), 5.37 (1H, t, J = 5.2 Hz), 7.28 (1H, d, J = 8.1 Hz), 7.34-7.39 (2H, m), 7.46 (2H, d, J = 7.5 Hz), 7.58-7.59 (2H, m), 7.68 (1H, d, J = 8.0 Hz), 7.72 (1H, d, J = 7.5 Hz), 7.76 (2H, d, J = 8.1 Hz), 7.91-7.97 (3H, m) ppm; ^{13}C NMR (126 MHz, DMSO-d_6) δ 52.8, 61.0, 61.5, 125.9, 127.1, 127.3, 127.9, 128.5, 129.3, 130.1, 130.2, 130.3, 130.7, 131.8, 132.8, 134.4, 138.7, 138.8, 138.9, 139.4, 139.5, 140.4, 141.0, 142.4, 166.7 ppm (one carbon signal is overlapped with another.); IR (ATR) 3468, 3368, 2358, 2322, 1705, 1319, 819, 764 cm^{-1}; HRMS (ESI) m/z calcd for $C_{28}H_{23}ClNaO_4$ ([M+Na]+) 481.1177; found: 481.1203;



(**26**)

Chloride **24** (123 mg, 0.268 mmol), boronic anhydride **2** (107 mg, 0.425 mmol), tris(dibenzylideneacetone)dipalladium(0) (7.8 mg, 0.0085 mmol), tri-*t*-butylphosphonium tetrafluoroborate (5.0 mg, 0.017 mmol), and KF (75.3 mg, 1.30 mmol) were charged into a flask under Ar, and then THF/H_2O (4/1) (0.25 mL) was added. The mixture was stirred at 50 ^{o}C for 22 h. Then, H_2O (5 mL) was added, and the mixture was extracted with AcOEt (10 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography three times (AcOEt/hexane (3/2), benzene/AcOEt (5/2), then AcOEt/hexane (3/2)) to give a mixture of **24** and **25**. This mixture, boronic acid **17** (67.8 mg, 0.403 mmol), palladium(II) acetate (4.0 mg, 0.018 mmol), 1,1'-bis(diphenylphosphino)ferrocene (8.9 mg, 0.016 mmol), and KF (48.7 mg, 0.838 mmol) were charged into a flask under Ar, and then THF (0.21 mL) was added. The mixture was stirred at 50 ^{o}C for 22 h. Then, H_2O (4 mL) was added, and the mixture was extracted with AcOEt (6 mL), washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The crude product was purified by silica gel chromatography (AcOEt/hexane (3/2)) to give **26** (34.5 mg, 2 steps 18%).

Mp. 188.2-189.5 ^{o}C; ^{1}H NMR (500 MHz, THF-d_8) δ 3.86 (3H, s), 4.28 (2H, t, J = 5.2 Hz), 4.37 (1H, t, J = 5.7 Hz), 4.54 (4H, t, J = 5.7 Hz), 4.63 (2H, d, J = 5.2 Hz), 7.24 (1H, d, J = 8.0 Hz), 7.29 (1H, dd, J = 1.7, 8.0 Hz), 7.33 (1H, d, J = 7.5 Hz), 7.38 (1H, d, J = 8.0 Hz), 7.44 (2H, d, J = 8.0 Hz), 7.50 (1H, t, J = 8.1 Hz), 7.54 (2H, d, J = 8.1 Hz), 7.63-7.70 (4H, m) 7.77 (4H, t, J = 7.5 Hz), 7.99-8.00 (3H, m), 8.08 (1H, s) ppm; ^{13}C

NMR (126 MHz, THF-d_8) δ 52.1, 62.2, 62.6, 62.7, 125.9, 126.0, 127.35, 127.40, 127.43, 128.3, 128.8, 129.0, 130.3, 130.5, 130.85, 130.87, 131.2, 131.7, 133.9, 134.4, 139.5, 139.62, 139.64, 140.3, 140.4, 140.7, 140.9, 141.09, 141.13, 142.0, 143.0, 166.9 ppm (five carbon signals are overlapped.); IR (ATR) 3256, 3196, 1728, 1475, 1244, 1003, 814 cm^{-1}; HRMS (ESI) m/z calcd for $C_{41}H_{33}ClO_5$ ([M+Na]+) 663.1909; found: 663.1918;

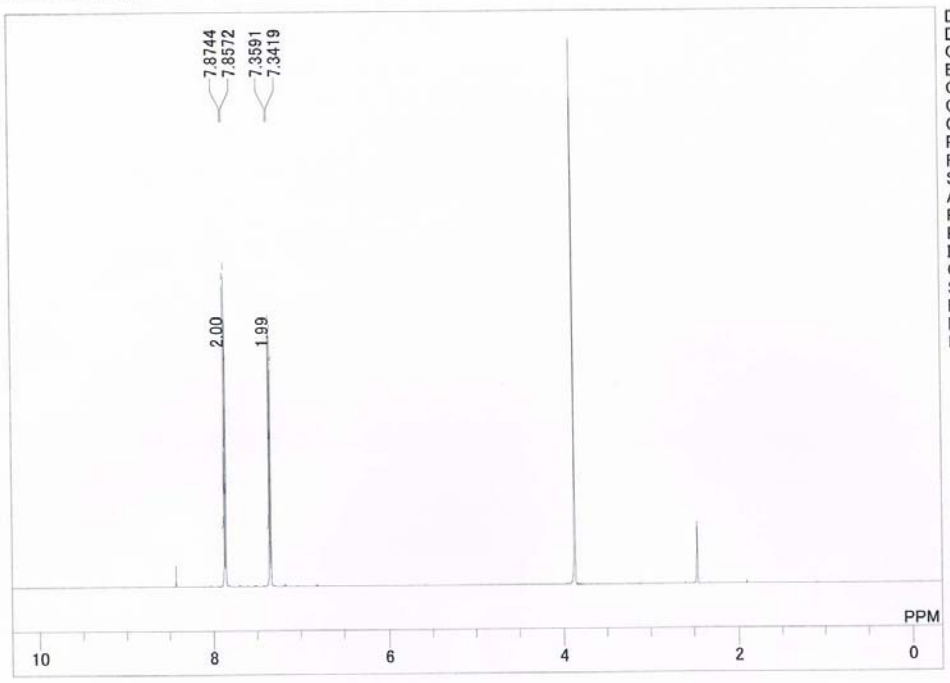
References

[1] S. J. Baker, Y-K. Zhang, T. Akama, A. Lau, H. Zhou, V. Hernandez, W. Mao, M. R. K. Alley, V. Sanders, J. J. Plattner, *J. Med. Chem*. **2006**, *49*, 4447.

[2] A. L. S. Thompson, G. W. Kabalka, M. R. Akula, J. W. Huffman, *Synthesis* **2005**, 547.

2

F:¥NMR data (大場)¥20091030 Tf BA.als



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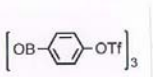

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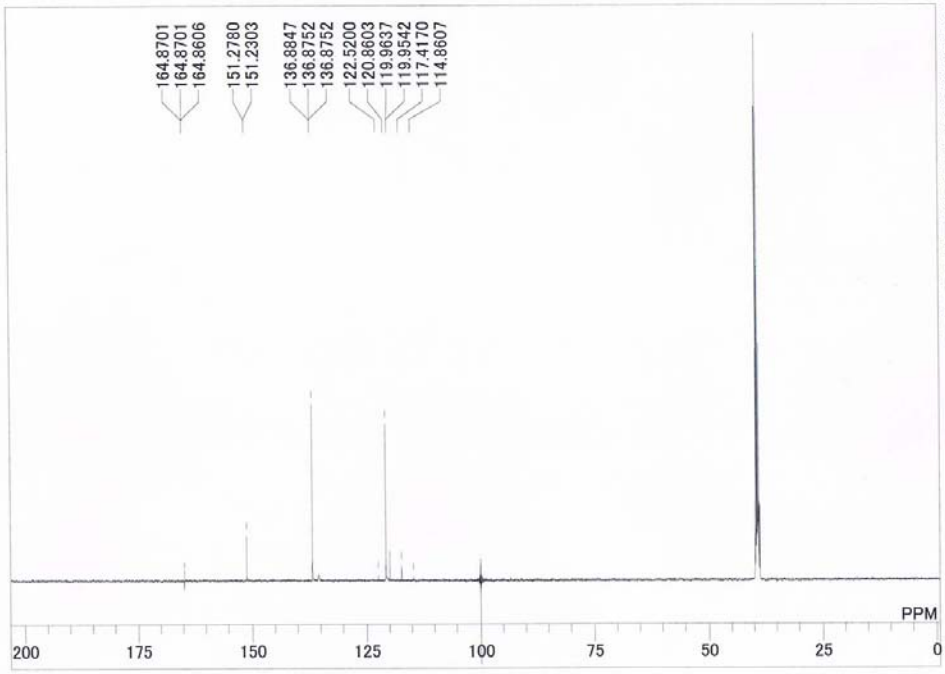

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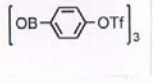
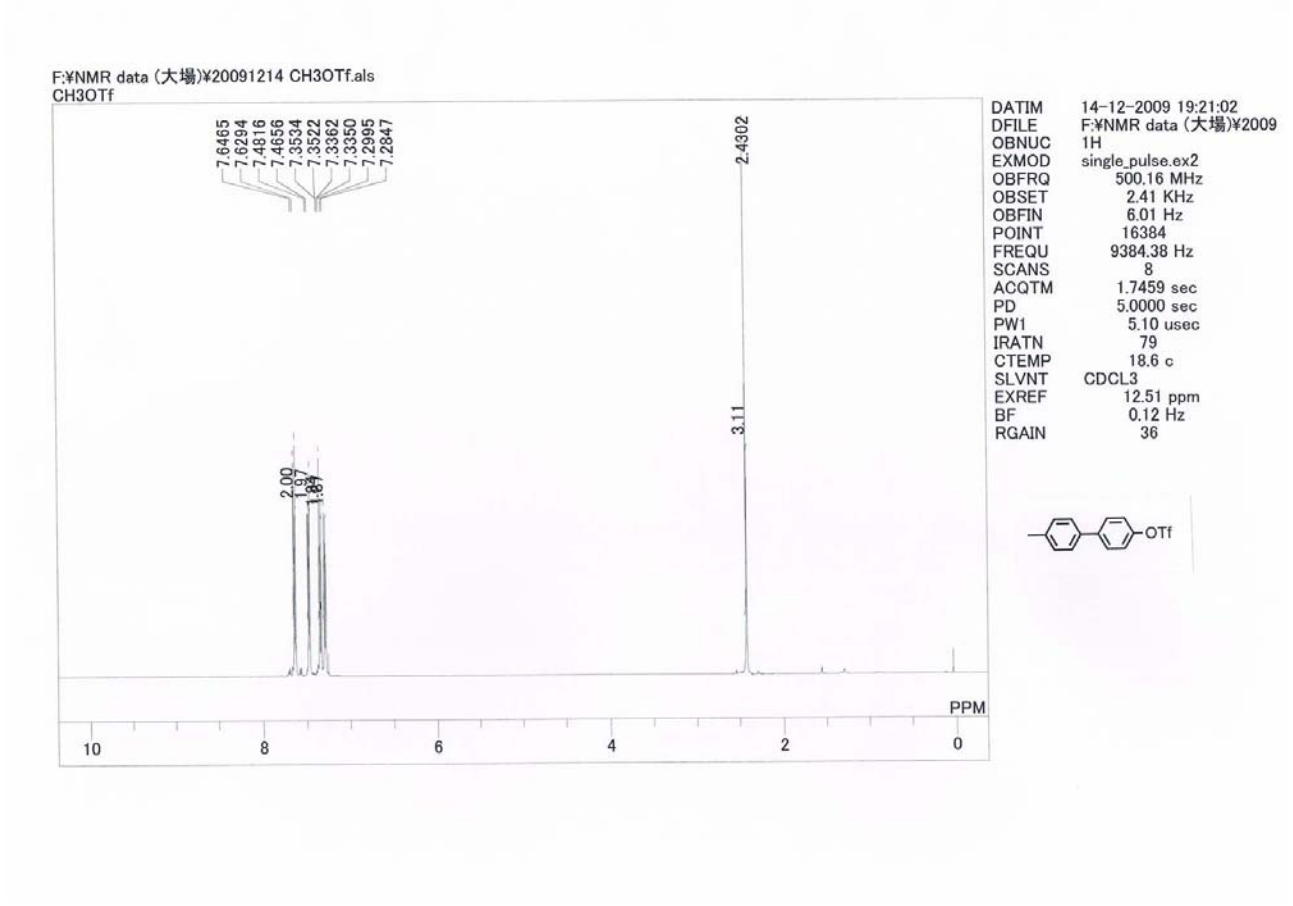


3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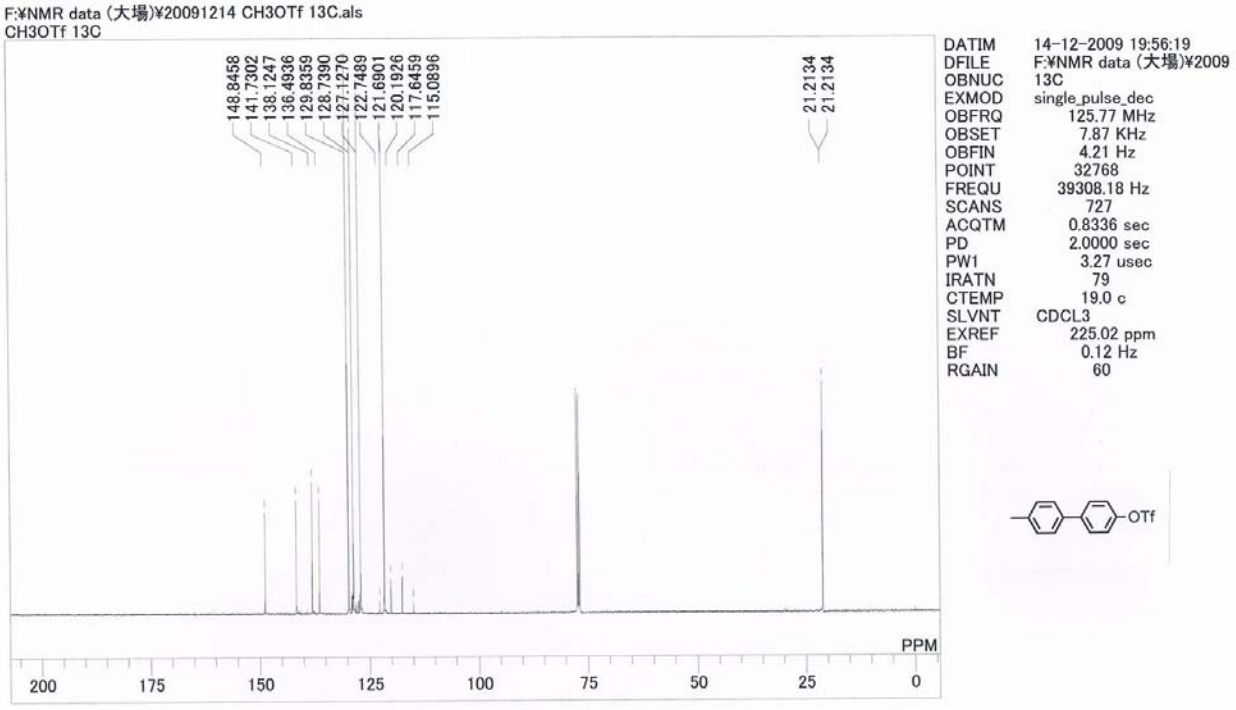
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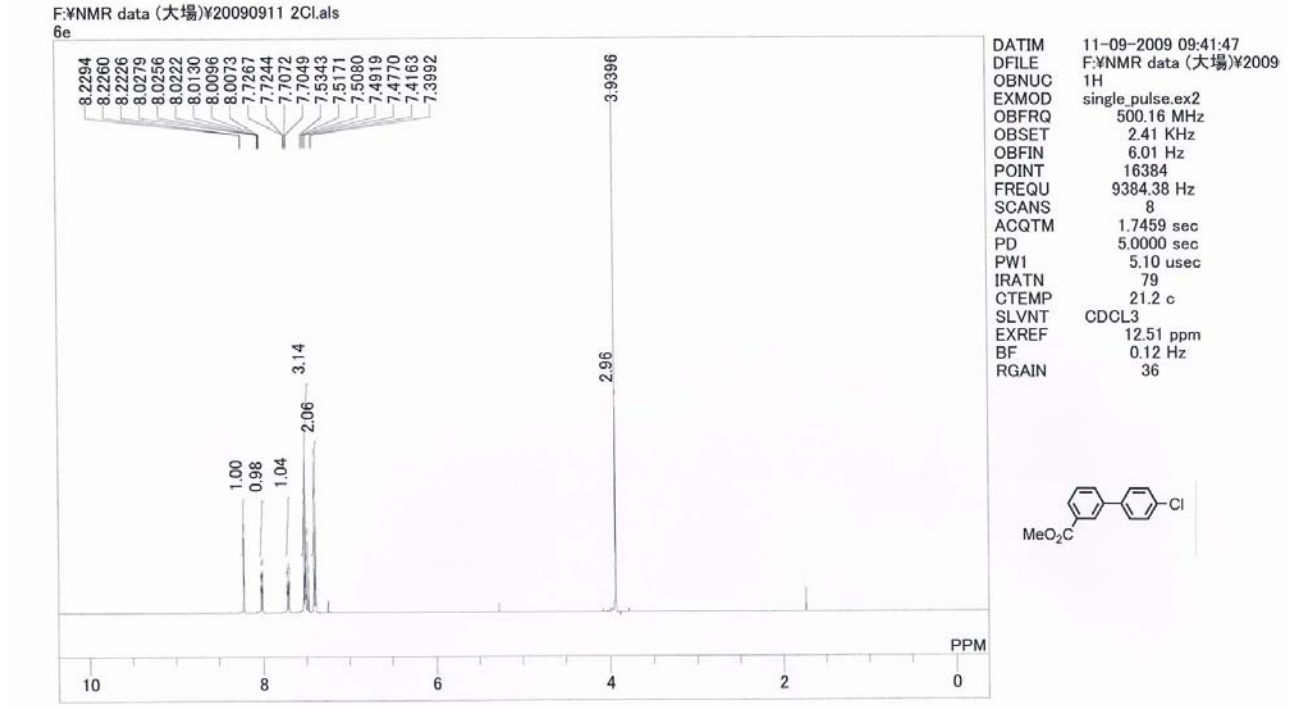
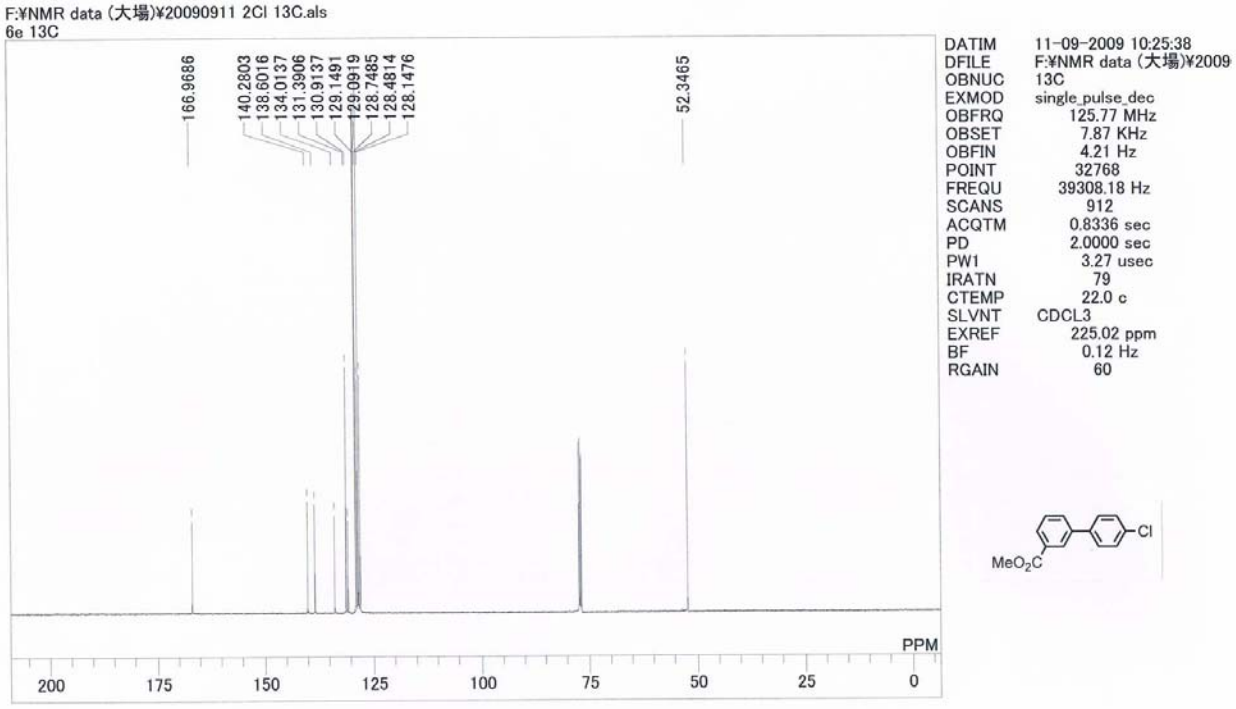
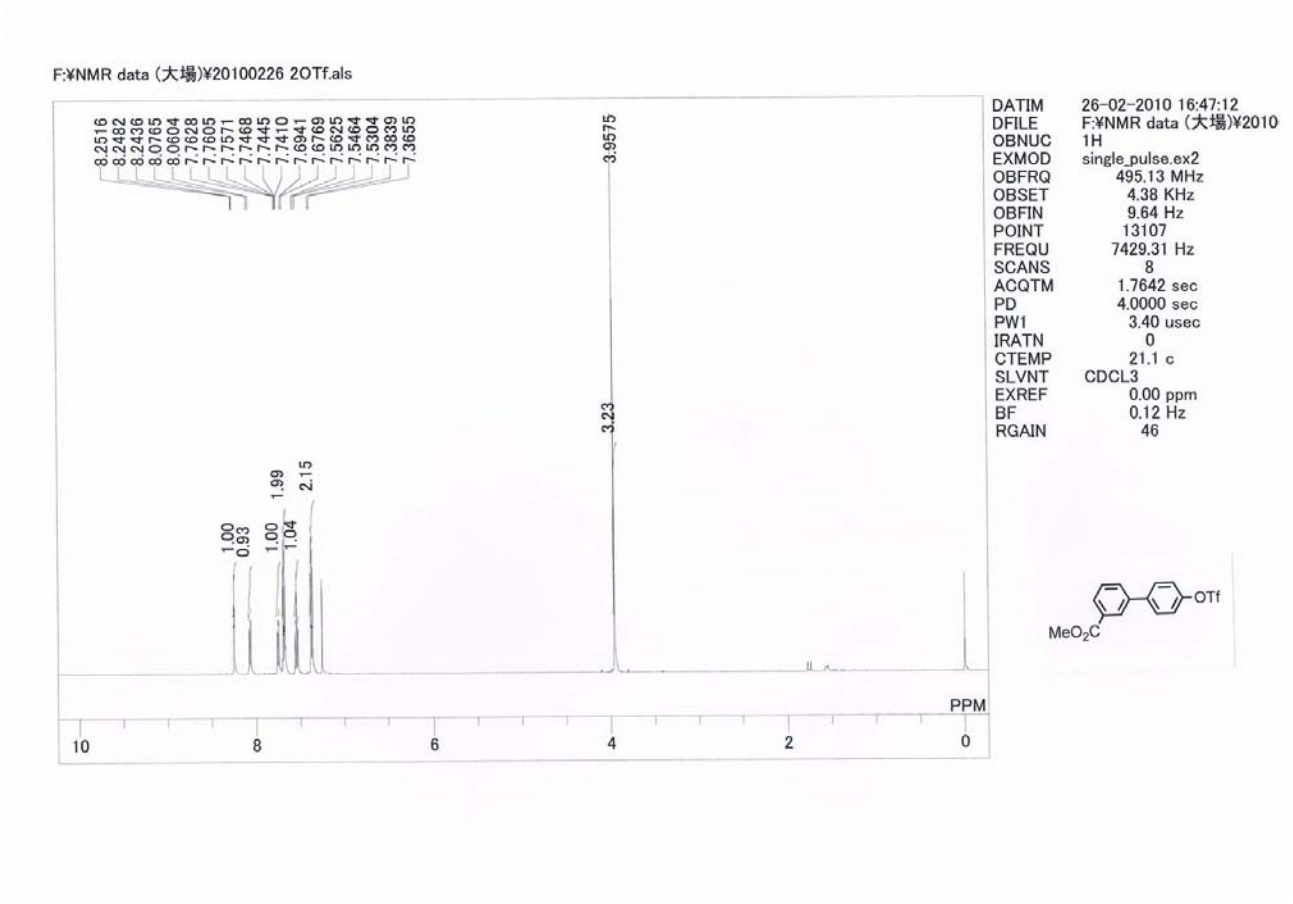
21.2134
21.2134
PPM
200
175
150
125
100
75
50
25
0
DATIM 14-12-2009 19:56:19
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 727
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 19.0 c
SLVNT CDCL3
EXREF 225.02 ppm
BF 0.12 Hz
RGAIN 60
OTf

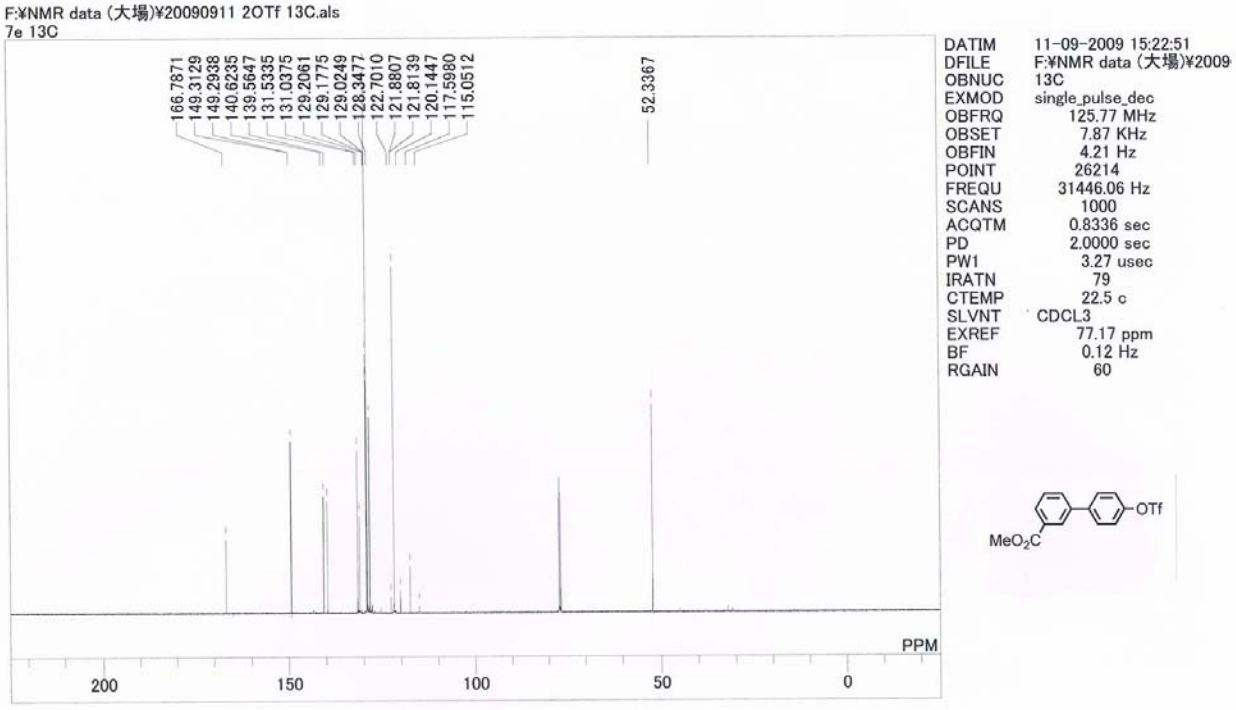
6


F:¥NMR data (大場)¥20090911 2Cl.als
6e
8.2294
8.2260
8.2226
8.0279
8.0256
8.0222
8.0130
8.0096
8.0073
7.7267
7.7244
7.7072
7.7049
7.5343
7.5171
7.5080
7.4919
7.4770
7.4163
7.3992
3.9396
3.14
2.06
1.00
0.98
1.04
2.96
PPM
10
8
6
4
2
0
DATIM 11-09-2009 09:41:47
DFILE F:¥NMR data (大場)¥2009
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.10 usec
IRATN 79
CTEMP 21.2 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 36
Cl
MeO2C


F:¥NMR data (大場)¥20090911 2Cl 13C.als
6e 13C
166.9686
140.2803
138.6016
134.0137
131.3906
130.9137
129.1491
129.0919
128.7485
128.4814
128.1476
52.3465
PPM
200
175
150
125
100
75
50
25
0
DATIM 11-09-2009 10:25:38
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 912
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 22.0 c
SLVNT CDCL3
EXREF 225.02 ppm
BF 0.12 Hz
RGAIN 60
Cl
MeO2C

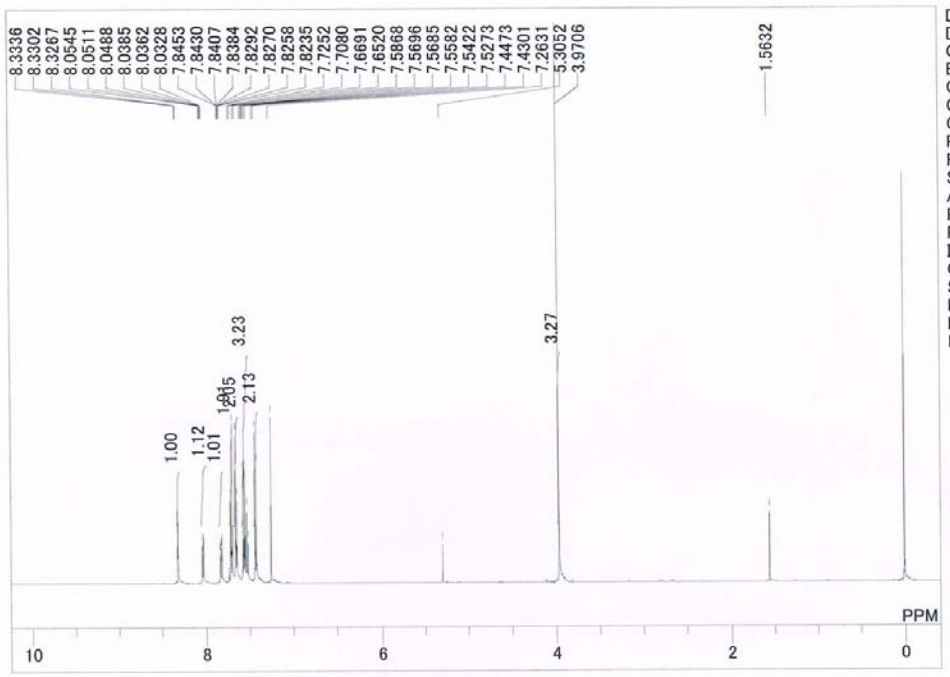
8


F:¥NMR data (大場)¥20100226 2OTf.als
8.2516
8.2482
8.2436
8.0765
8.0604
7.7628
7.7605
7.7571
7.7468
7.7445
7.7410
7.6941
7.6769
7.5625
7.5464
7.5304
7.3839
7.3655
3.9575
3.23
1.00
0.93
1.00
1.04
1.99
2.15
PPM
10
8
6
4
2
0
DATIM 26-02-2010 16:47:12
DFILE F:¥NMR data (大場)¥2010
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 4.0000 sec
PW1 3.40 usec
IRATN 0
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46
OTf
MeO2C

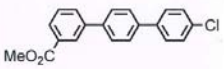

F:¥NMR data (大場)¥20090911 2OTf 13C.als
7e 13C
166.7871
149.3129
149.2938
140.6235
139.5647
131.5335
131.0375
129.2061
129.1775
129.0249
128.3477
122.7010
121.8807
121.8139
120.1447
117.5980
115.0512
52.3367
PPM
200
150
100
50
0
DATIM 11-09-2009 15:22:51
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 22.5 c
SLVNT CDCL3
EXREF 77.17 ppm
BF 0.12 Hz
RGAIN 60
OTf
MeO2C

9

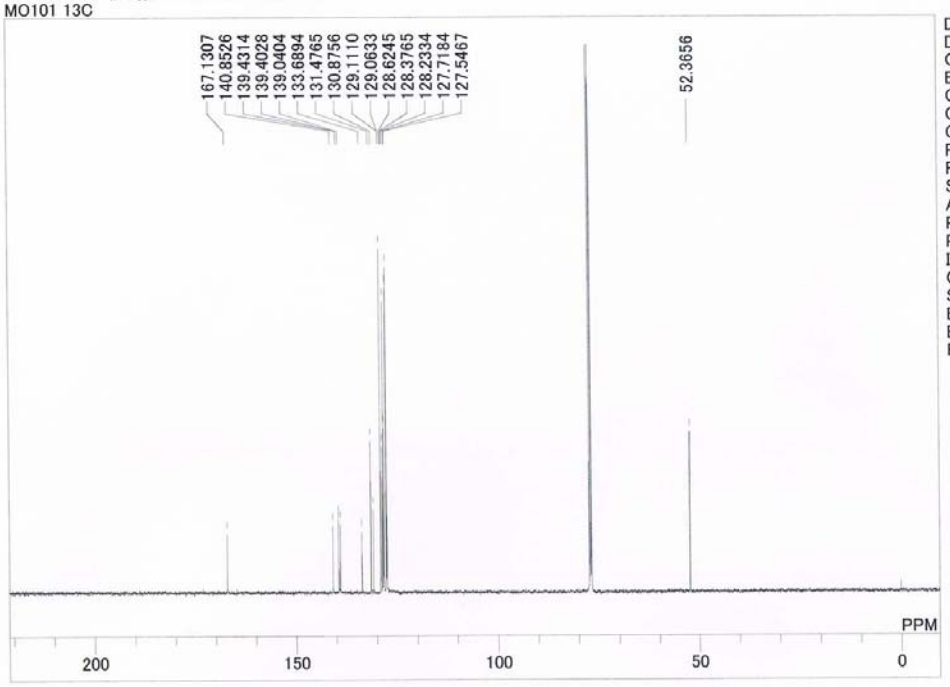
F:¥NMR data (大場)¥20100302 3Cl.als



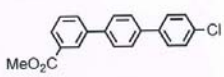
DATIM 02-03-2010 18:50:41
DFILE F:¥NMR data (大場)¥2010
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13120
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 4.0000 sec
PW1 3.40 usec
IRATN 10
CTEMP 20.8 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 50



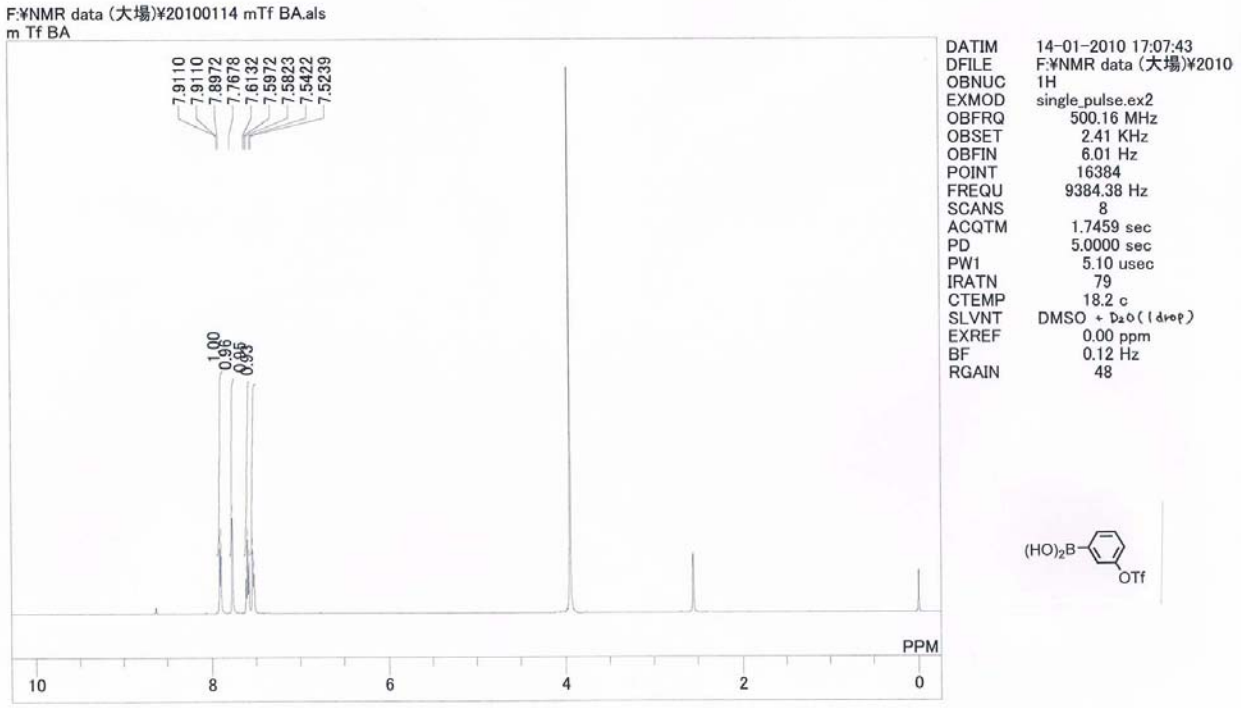
F:¥NMR data (大場)¥20091021 3Cl 13C.als
MO101 13C

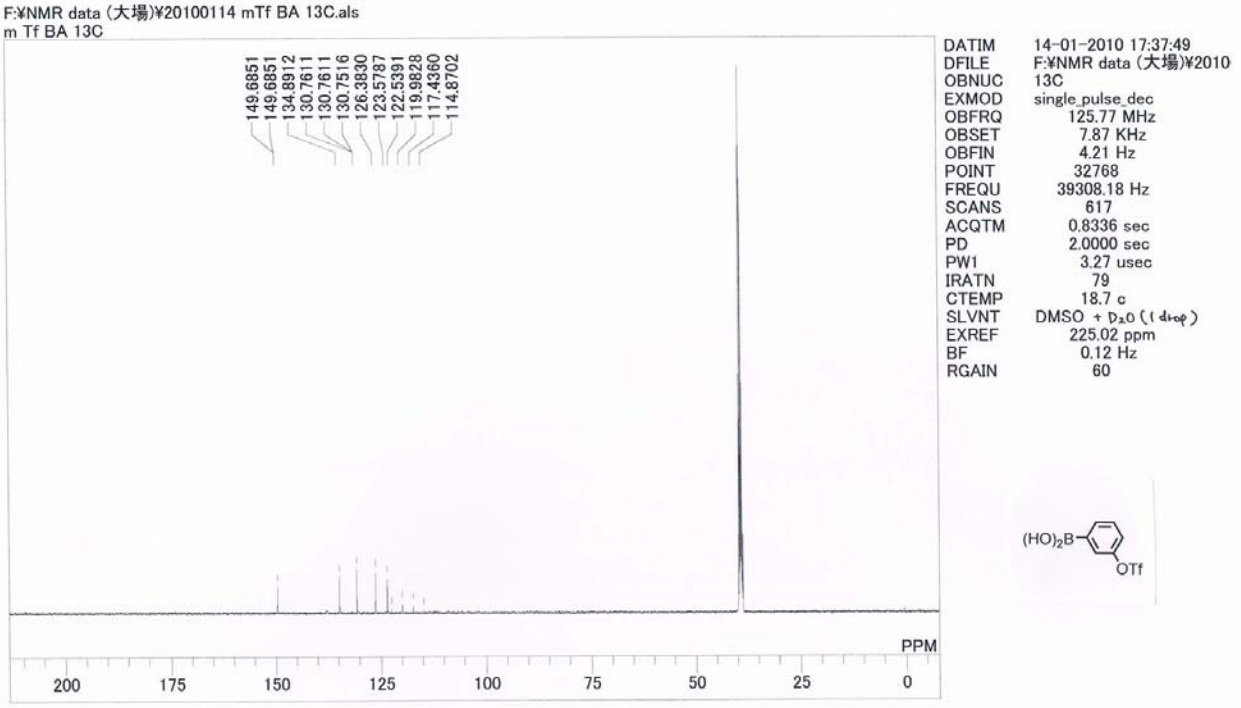


DATIM 21-10-2009 16:53:09
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 932
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 20.5 c
SLVNT CDCL3
EXREF 225.02 ppm
BF 0.12 Hz
RGAIN 60



10


F:¥NMR data (大場)¥20100114 mTf BA.als
m Tf BA
7.9110
7.9110
7.8972
7.7678
7.6132
7.5972
7.5823
7.5422
7.5239
1.00
0.96
0.95
0.93
PPM
10
8
6
4
2
0
DATIM 14-01-2010 17:07:43
DFILE F:¥NMR data (大場)¥2010
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.10 usec
IRATN 79
CTEMP 18.2 c
SLVNT DMSO + D2O (1 drop)
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48
(HO)2B
OTf


F:¥NMR data (大場)¥20100114 mTf BA 13C.als
m Tf BA 13C
149.6851
149.6851
134.8912
130.7611
130.7611
130.7516
126.3830
123.5787
122.5391
119.9828
117.4360
114.8702
PPM
200
175
150
125
100
75
50
25
0
DATIM 14-01-2010 17:37:49
DFILE F:¥NMR data (大場)¥2010
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 617
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 18.7 c
SLVNT DMSO + D2O (1 drop)
EXREF 225.02 ppm
BF 0.12 Hz
RGAIN 60
(HO)2B
OTf

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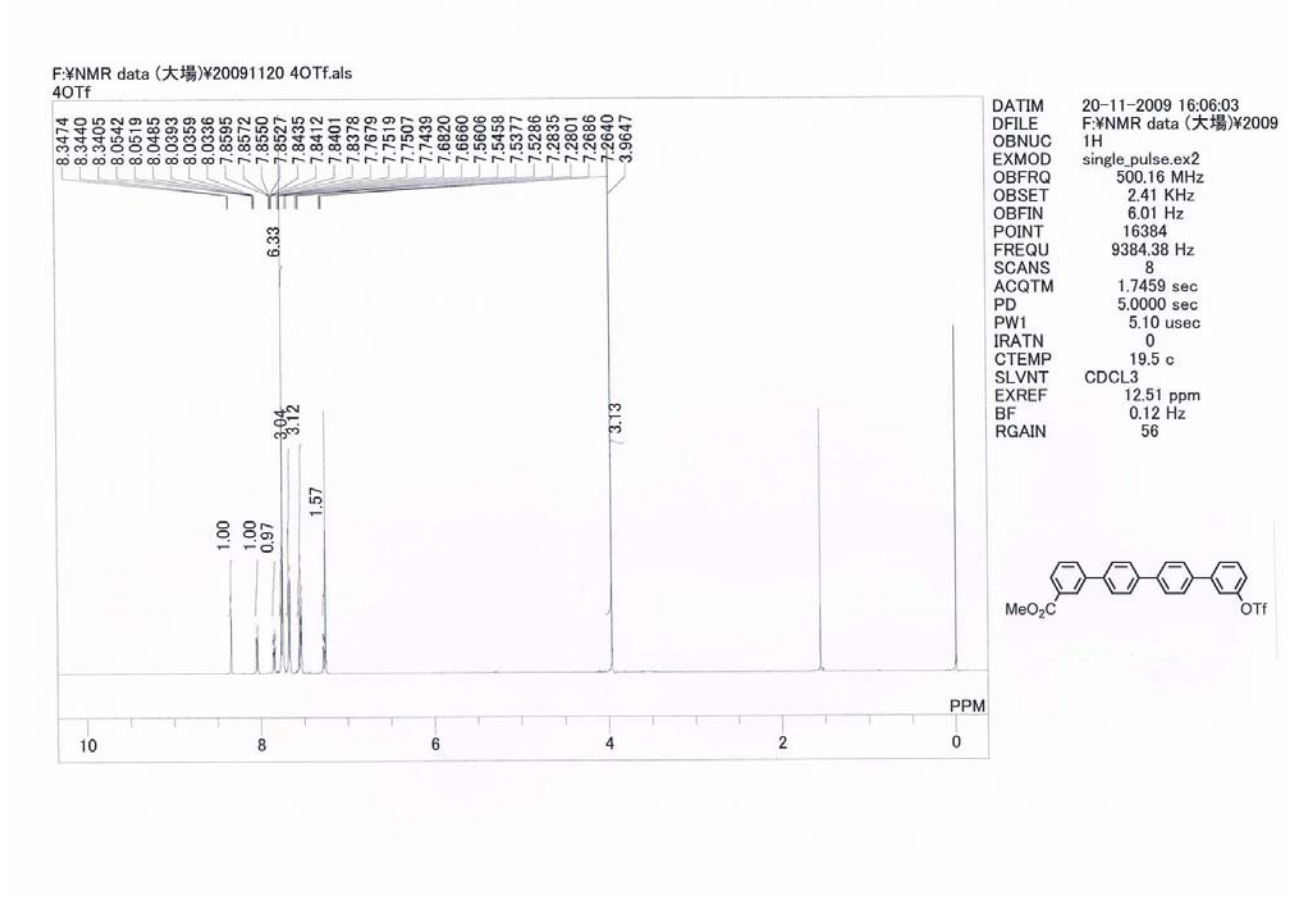
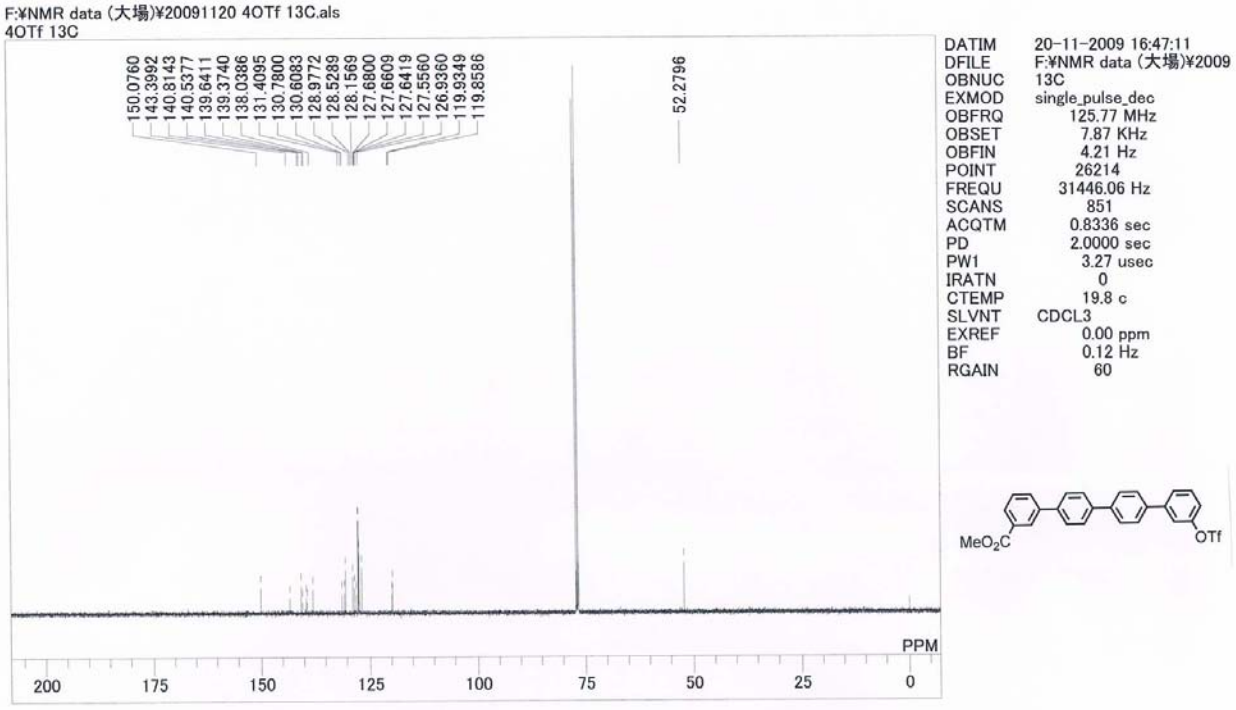
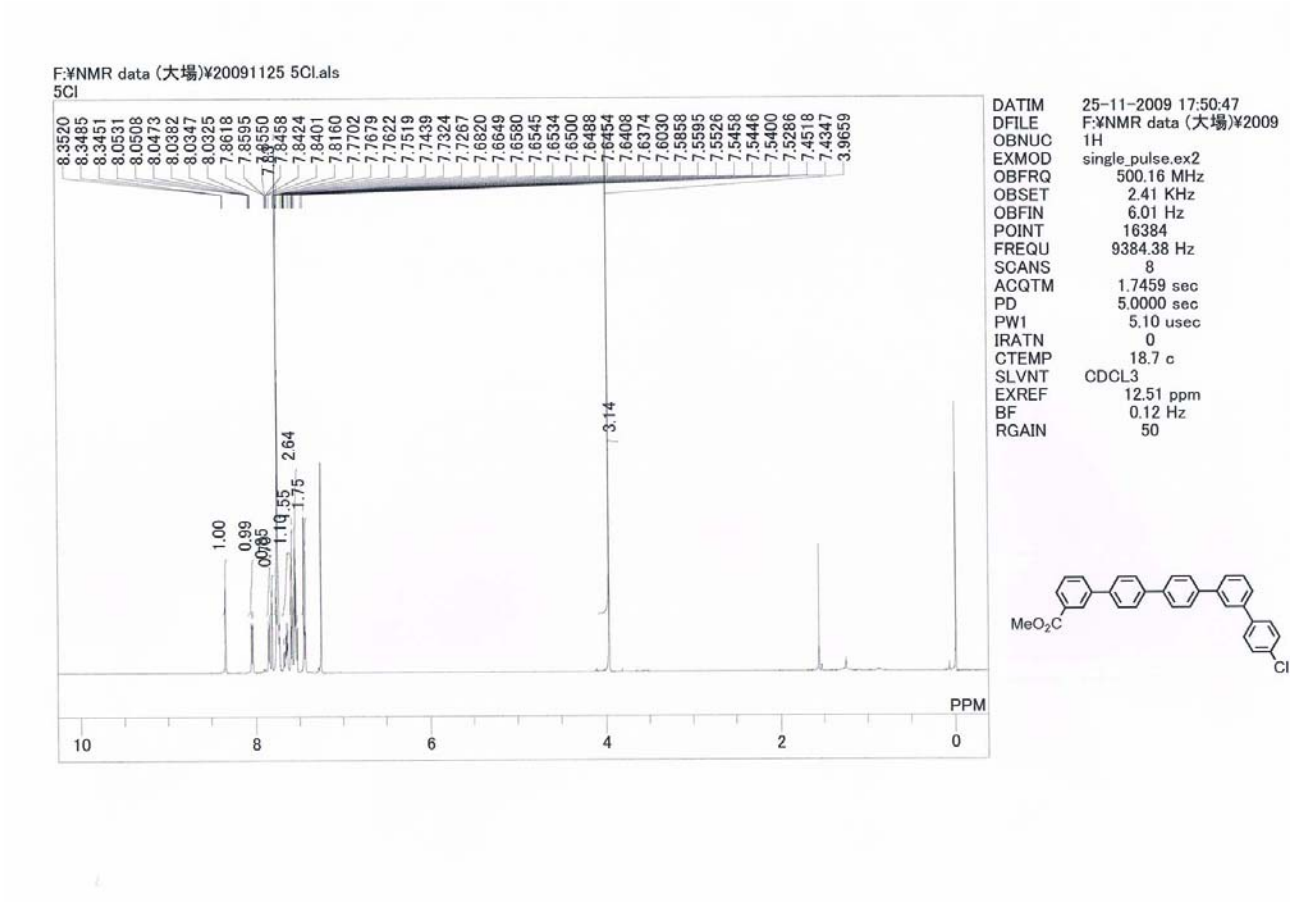
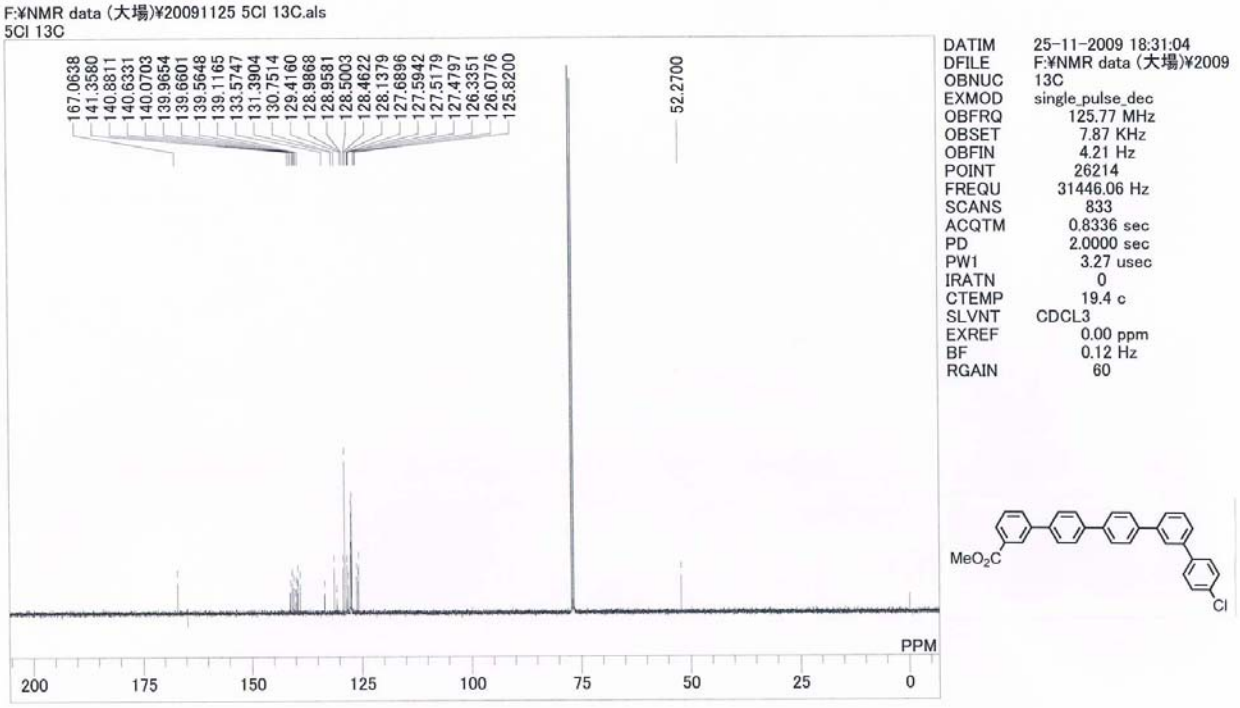

F:¥NMR data (大場)¥20091120 4OTf.als
4OTf
8.3474
8.3440
8.3405
8.0542
8.0519
8.0485
8.0393
8.0359
8.0336
7.8595
7.8572
7.8550
7.8527
7.8435
7.8412
7.8401
7.8378
7.7679
7.7519
7.7507
7.7439
7.6820
7.6660
7.5606
7.5458
7.5377
7.5286
7.2835
7.2801
7.2686
7.2640
3.9647
6.33
3.04
3.12
1.57
3.13
1.00
1.00
0.97
PPM
10
8
6
4
2
0
DATIM 20-11-2009 16:06:03
DFILE F:¥NMR data (大場)¥2009
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.10 usec
IRATN 0
CTEMP 19.5 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 56
MeO2C
OTf


F:¥NMR data (大場)¥20091120 4OTf 13C.als
4OTf 13C
150.0760
143.3992
140.8143
140.5377
139.6411
139.3740
138.0386
131.4095
130.7800
130.6083
128.9772
128.5289
128.1569
127.6800
127.6609
127.6419
127.5560
126.9360
119.9349
119.8586
52.2796
PPM
200
175
150
125
100
75
50
25
0
DATIM 20-11-2009 16:47:11
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 851
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 0
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60
MeO2C
OTf

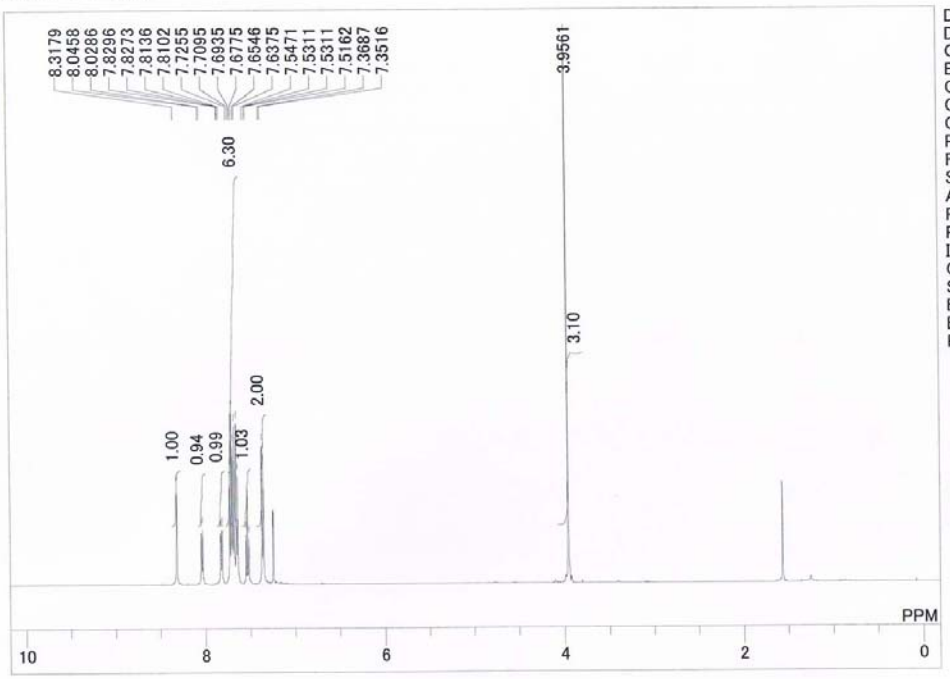
12


F:¥NMR data (大場)¥20091125 5Cl.als
5Cl
DATIM 25-11-2009 17:50:47
DFILE F:¥NMR data (大場)¥2009
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.10 usec
IRATN 0
CTEMP 18.7 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.12 Hz
RGAIN 50
3.14
2.64
1.75
1.10
1.00
0.99
PPM
MeO2C
Cl

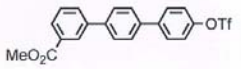

F:¥NMR data (大場)¥20091125 5Cl 13C.als
5Cl 13C
DATIM 25-11-2009 18:31:04
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 833
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 0
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60
52.2700
PPM
MeO2C
Cl

13

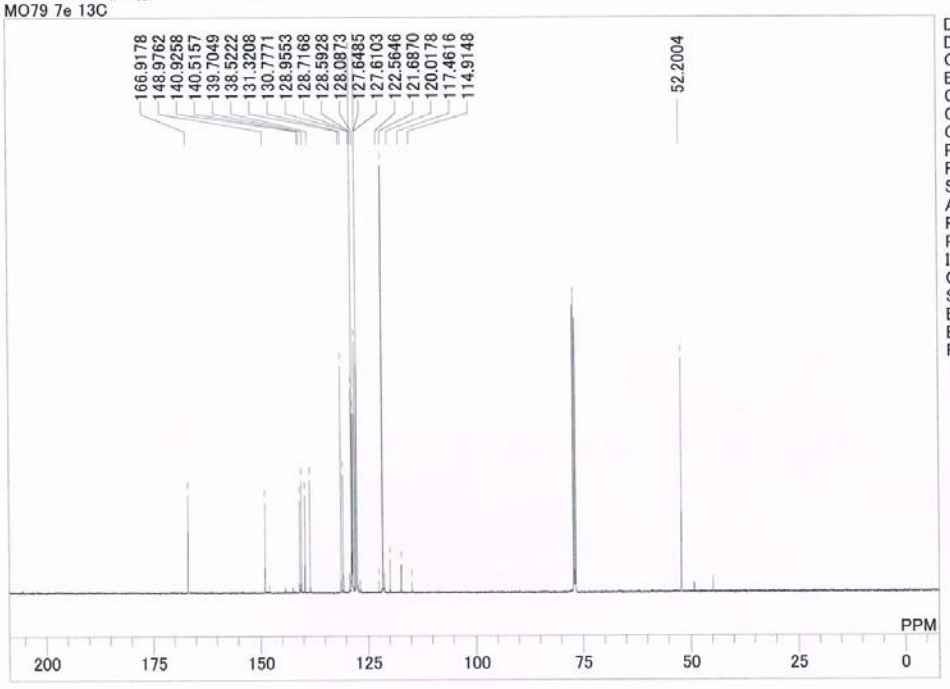
F:¥NMR data (大場)¥20090909 3OTf.als



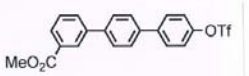
DATIM 09-09-2009 11:09:56
DFILE F:¥NMR data (大場)¥2009
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13120
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 4.0000 sec
PW1 3.30 usec
IRATN 10
CTEMP 23.9 c
SLVNT CDCL3
EXREF 12.49 ppm
BF 0.12 Hz
RGAIN 40



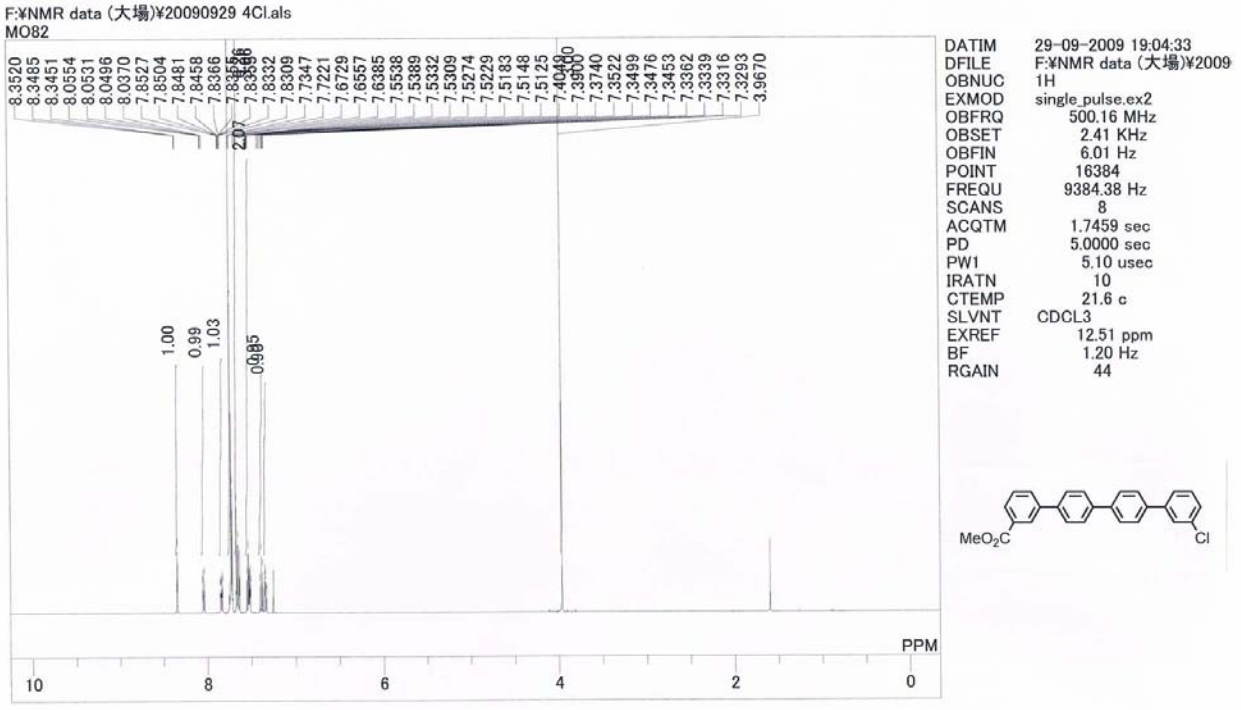
F:¥NMR data (大場)¥20090924 3OTf 13C.als
MO79 7e 13C

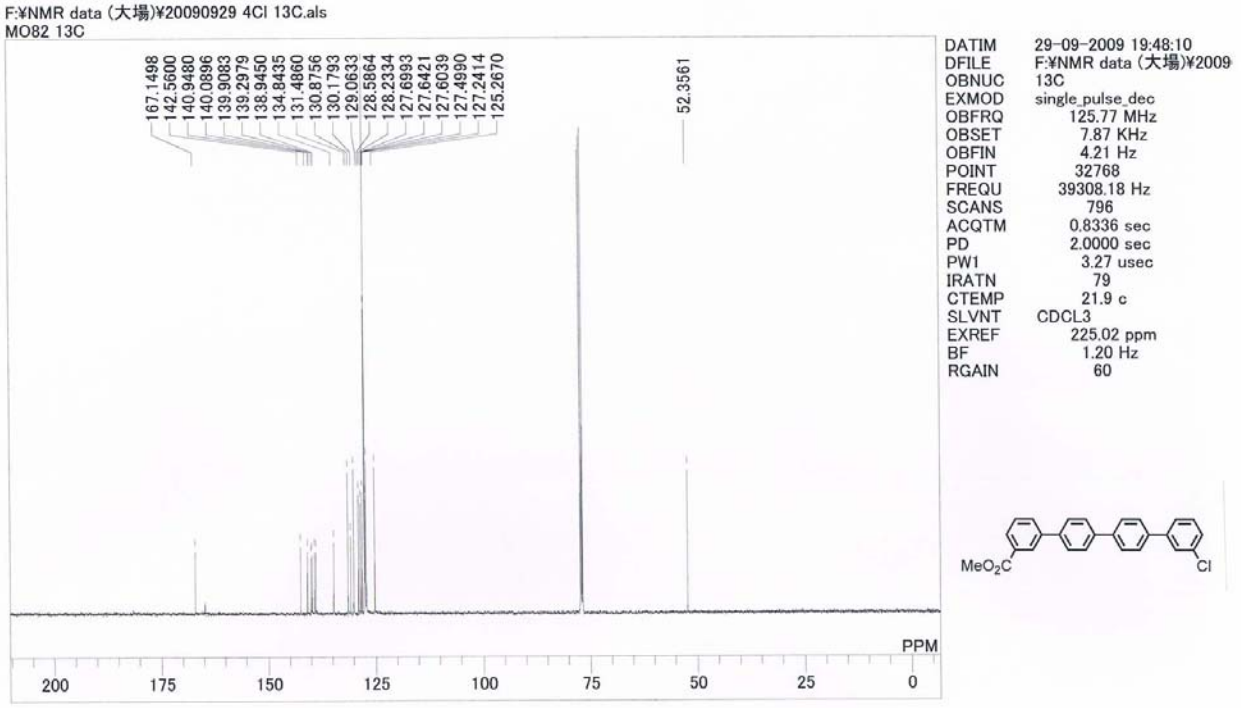


DATIM 24-09-2009 10:14:21
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 986
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

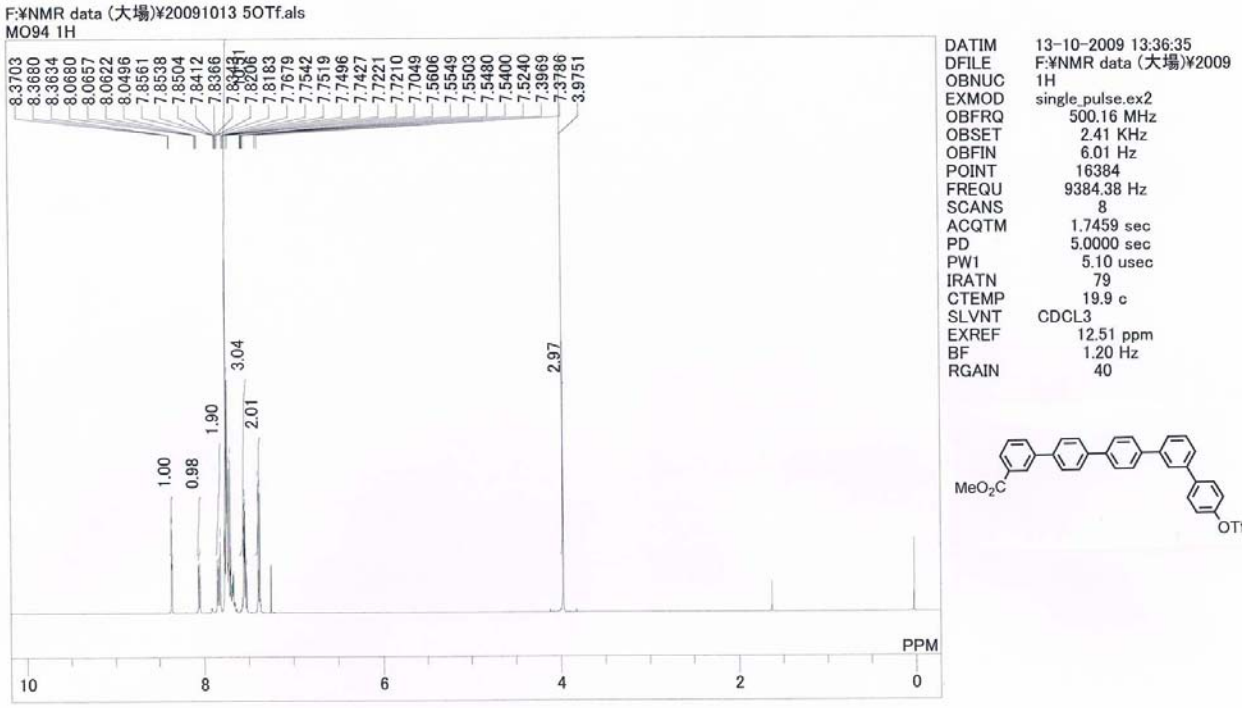


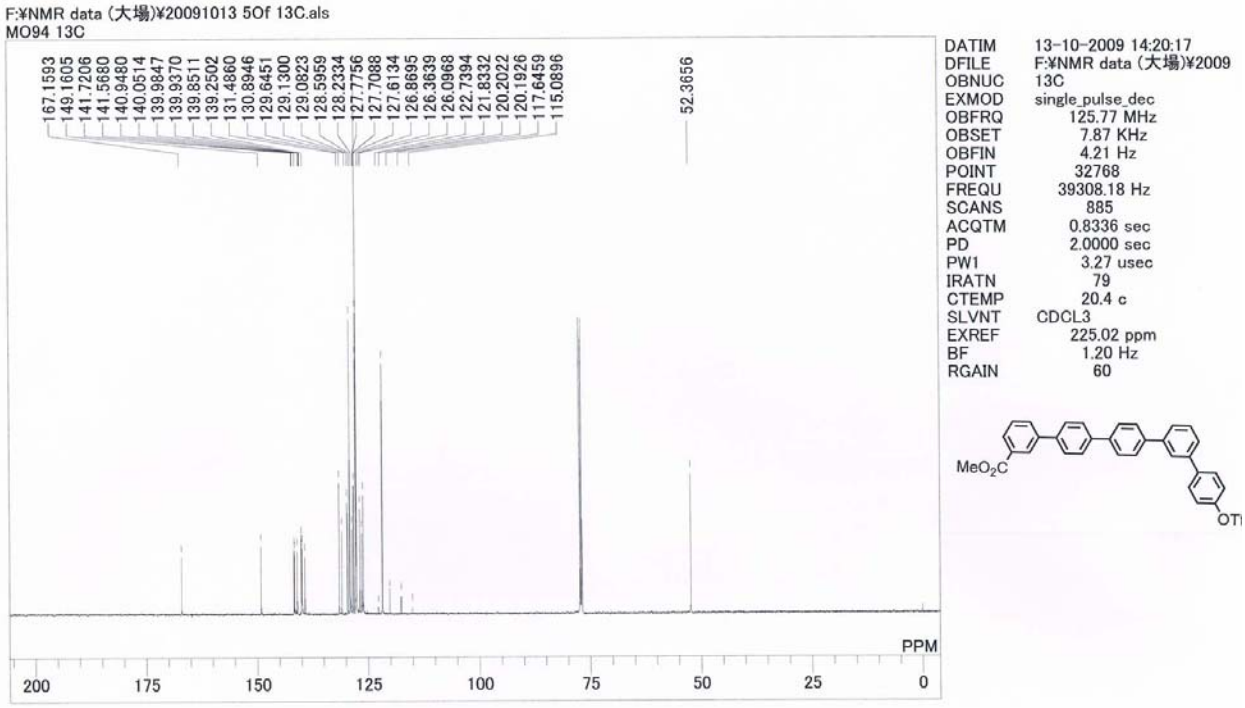
15


F:¥NMR data (大場)¥20090929 4Cl.als
MO82
8.3520
8.3485
8.3451
8.0554
8.0531
8.0496
8.0370
7.8527
7.8504
7.8481
7.8458
7.8366
7.8332
7.8309
7.7347
7.7221
7.6729
7.6557
7.6385
7.5538
7.5389
7.5332
7.5309
7.5274
7.5229
7.5183
7.5148
7.5125
7.4049
7.3900
7.3740
7.3522
7.3499
7.3476
7.3453
7.3362
7.3339
7.3316
7.3293
3.9670
2.07
1.00
0.99
1.03
0.85
PPM
10
8
6
4
2
0
DATIM 29-09-2009 19:04:33
DFILE F:¥NMR data (大場)¥2009
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.10 usec
IRATN 10
CTEMP 21.6 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 1.20 Hz
RGAIN 44
MeO2C
Cl

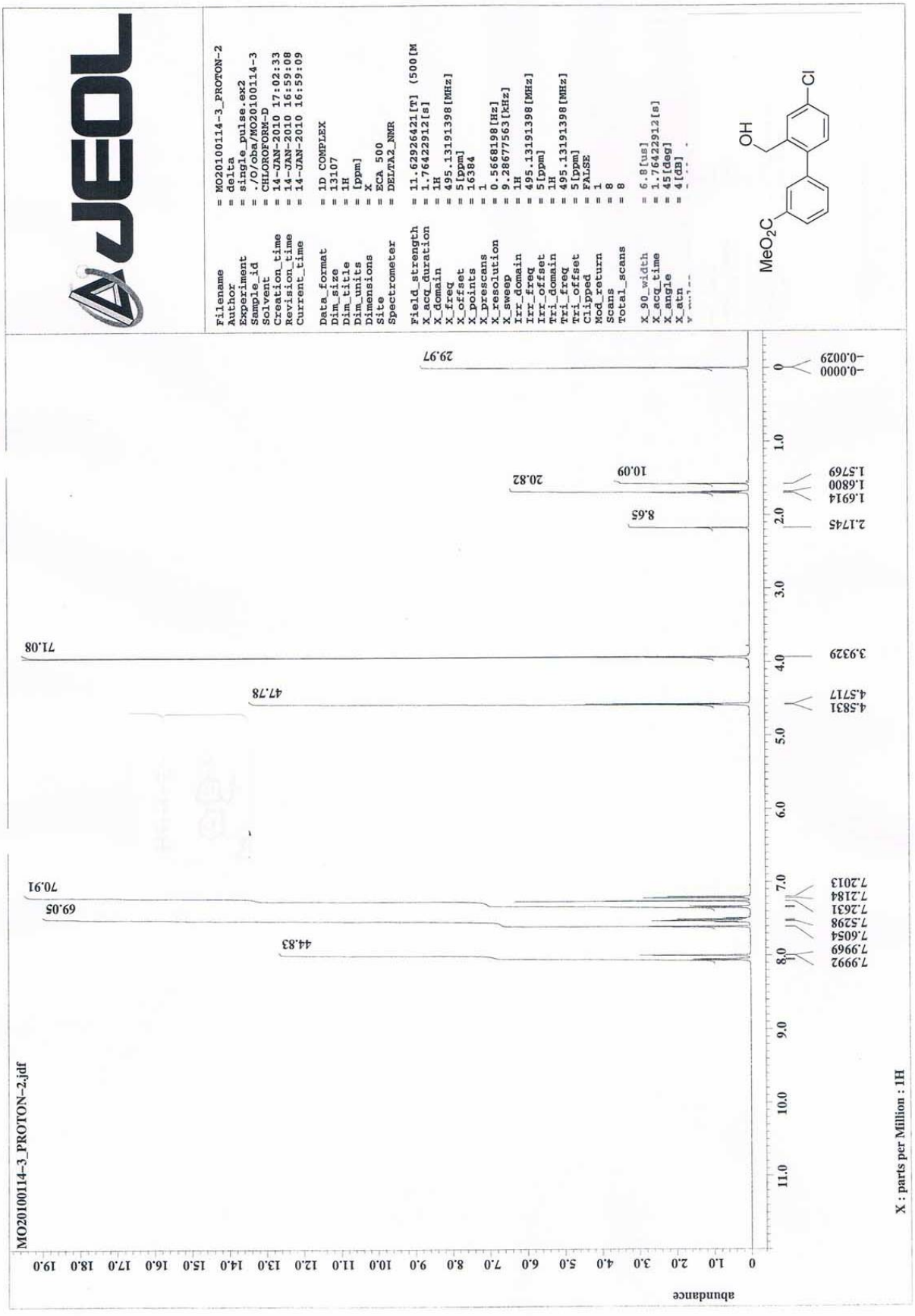

F:¥NMR data (大場)¥20090929 4Cl 13C.als
MO82 13C
167.1498
142.5600
140.9480
140.0896
139.9083
139.2979
138.9450
134.8435
131.4860
130.8756
130.1793
129.0633
128.5864
128.2334
127.6993
127.6421
127.6039
127.4990
127.2414
125.2670
52.3561
PPM
200
175
150
125
100
75
50
25
0
DATIM 29-09-2009 19:48:10
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 796
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 21.9 c
SLVNT CDCL3
EXREF 225.02 ppm
BF 1.20 Hz
RGAIN 60
MeO2C
Cl

16


F:¥NMR data (大場)¥20091013 5OTf.als
MO94 1H
8.3703
8.3680
8.3634
8.0680
8.0657
8.0622
8.0496
7.8561
7.8538
7.8504
7.8412
7.8366
7.8206
7.8183
7.7679
7.7542
7.7519
7.7496
7.7427
7.7221
7.7210
7.7049
7.5606
7.5549
7.5503
7.5480
7.5400
7.5240
7.3969
7.3786
3.9751
3.04
2.97
2.01
1.90
1.00
0.98
PPM
10
8
6
4
2
0
DATIM 13-10-2009 13:36:35
DFILE F:¥NMR data (大場)¥2009
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.10 usec
IRATN 79
CTEMP 19.9 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 1.20 Hz
RGAIN 40
MeO2C
OT


F:¥NMR data (大場)¥20091013 5Of 13C.als
MO94 13C
167.1593
149.1605
141.7206
141.5680
140.9480
140.0514
139.9847
139.9370
139.8511
139.2502
131.4860
130.8946
129.6451
129.1300
129.0823
128.5959
128.2334
127.7756
127.7088
127.6134
126.8695
126.3639
126.0968
122.7394
121.8332
120.2022
120.1926
117.6459
115.0896
52.3656
PPM
200
175
150
125
100
75
50
25
0
DATIM 13-10-2009 14:20:17
DFILE F:¥NMR data (大場)¥2009
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 885
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.27 usec
IRATN 79
CTEMP 20.4 c
SLVNT CDCL3
EXREF 225.02 ppm
BF 1.20 Hz
RGAIN 60
MeO2C
OT

18


JEOL
Filename = MO20100114-3_PROTON-2
Author = delta
Experiment = single_pulse.ex2
Sample_id = ./O/oba/MO20100114-3
Solvent = CHLOROFORM-D
Creation_time = 14-JAN-2010 17:02:33
Revision_time = 14-JAN-2010 16:59:08
Current_time = 14-JAN-2010 16:59:09
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.629264211[T] (500[M
X_acq_duration = 1.764229121[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 6.8[us]
X_acq_time = 1.764229121[s]
X_angle = 45[deg]
X_atn = 4[dB]
OH
Cl
MeO2C
MO20100114-3_PROTON-2.jdf
X : parts per Million : 1H
abundance
29.97
20.82
10.09
8.65
71.08
47.78
70.91
69.05
44.83
-0.0029
-0.0000
1.5769
1.6800
1.6914
2.1745
3.9329
4.5717
4.5831
7.2013
7.2184
7.2631
7.5298
7.6054
7.9969
7.9992


---- ACQUISITION PARAMETERS ----
Derived from: 20091215 MO116 CH2OH 13C.1
File Name = 20091215 MO116 CH2OH
Author = delta
Sample ID = 20091215 MO116 CH2OH
Content = MO116 CH2OH 13C
Creation Date = 15-DEC-2009 10:19:46
Revision Date = 15-DEC-2009 10:21:47
Spec Site = ECA500
Spec Type = DELTA2_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 26214
Dim Units = [ppm]
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 872
Total_scans = 872
X_90_width = 9.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.4[dB]
X_pulse = 3.26666667[us]
Irr_atn_dec = 21.8[dB]
Irr_atn_noe = 21.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
MeO2C
OH
Cl
JEOL
(Millions)
800.0
700.0
600.0
500.0
400.0
300.0
200.0
100.0
0
-100.0
-200.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
167.0544
140.0037
139.8797
138.3345
134.1091
133.6417
131.1143
130.3605
130.1602
128.8153
128.6245
128.2621
127.7470
77.4132
77.1653
76.9077
62.4476
52.4324
X : parts per Million : 13C

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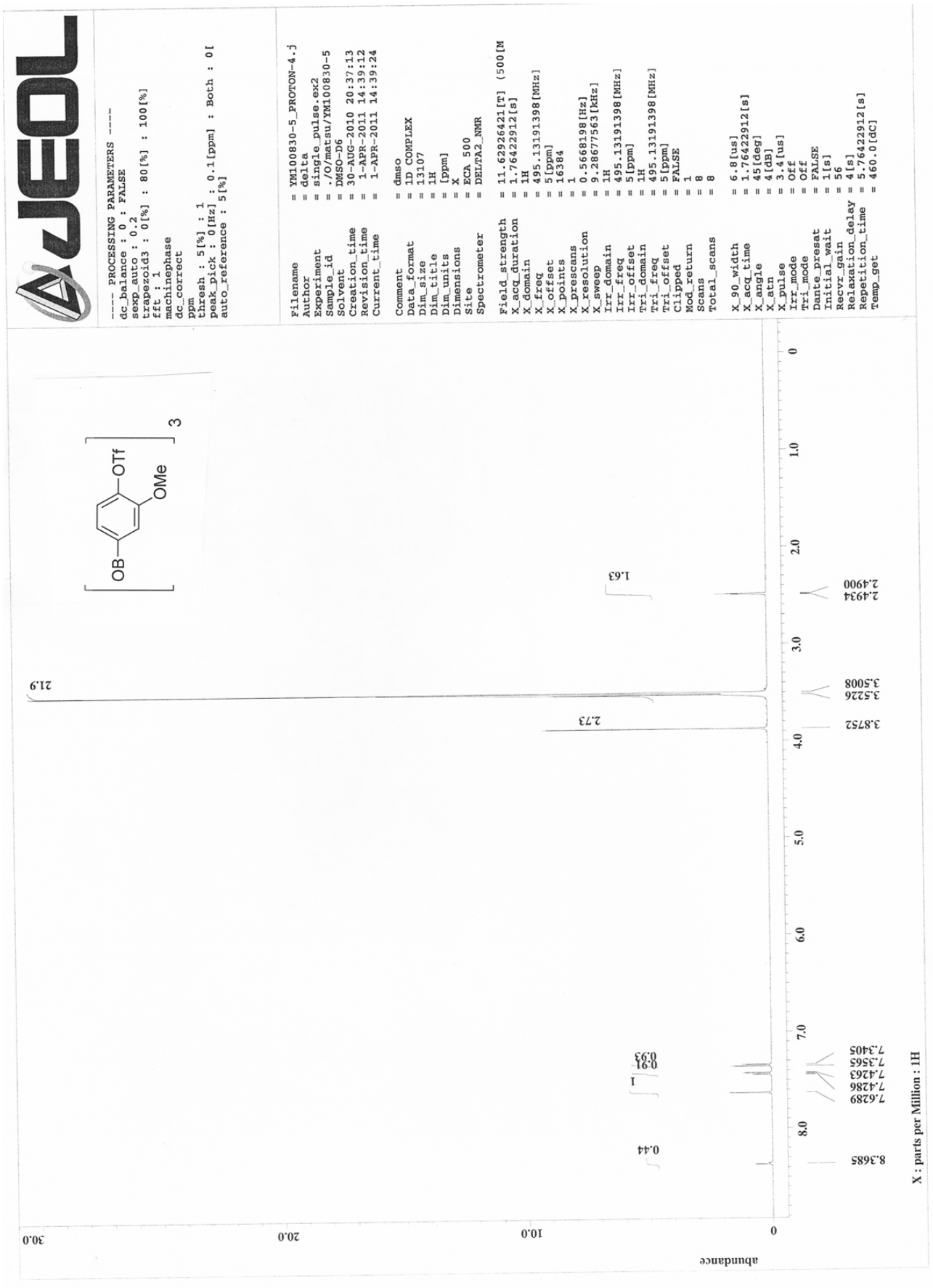
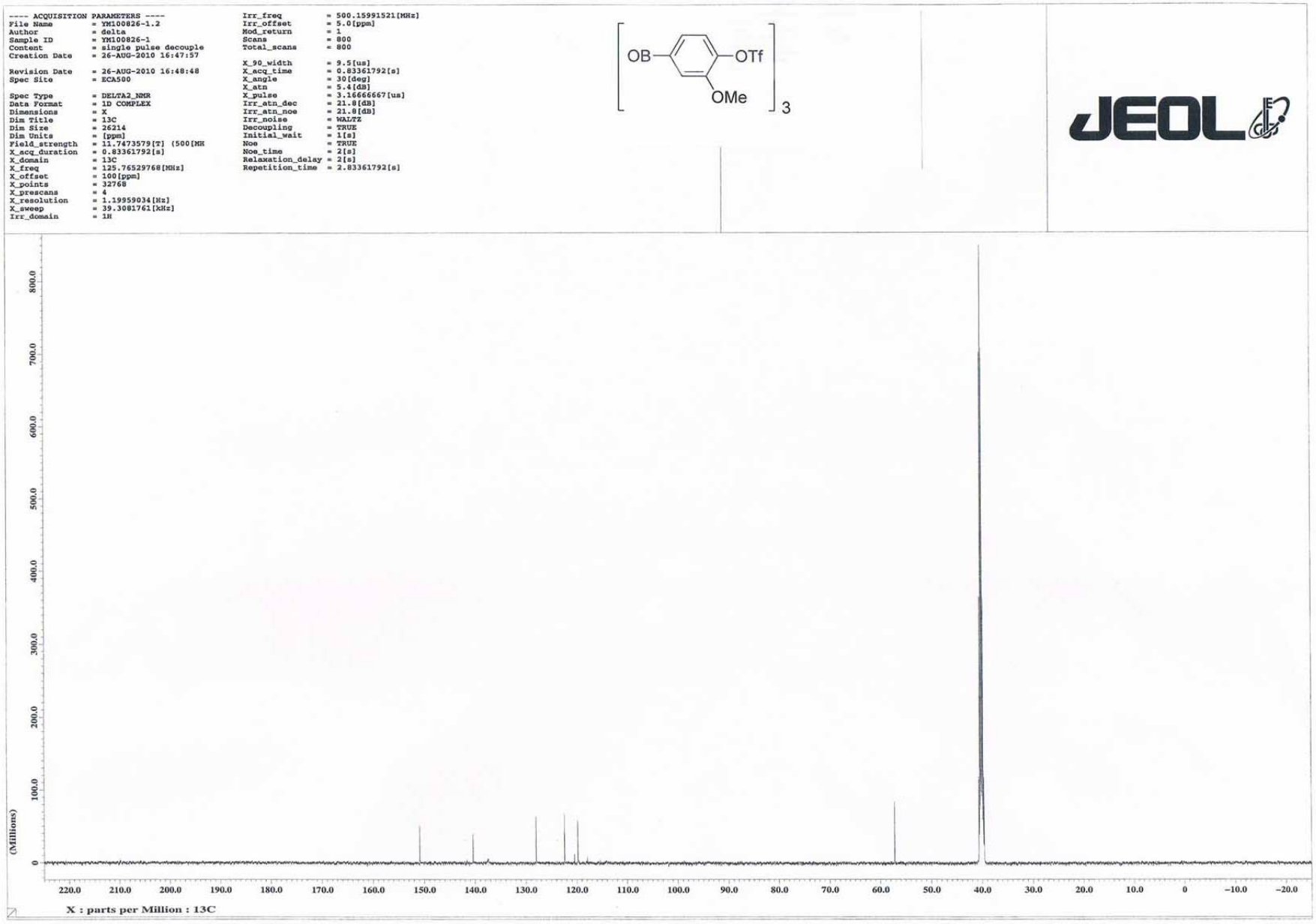

JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[
auto_reference : 5[%]
Filename = YM100830-5_PROTON-4.j
Author = delta
Experiment = single_pulse.ex2
Sample_id = ./O/matsu/YM100830-5
Solvent = DMSO-D6
Creation_time = 30-AUG-2010 20:37:13
Revision_time = 1-APR-2011 14:39:12
Current_time = 1-APR-2011 14:39:24
Comment = dmso
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 6.8[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 3.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 56
Relaxation_delay = 4[s]
Repetition_time = 5.76422912[s]
Temp_get = 460.0[dC]
OTf
OMe
OB
3
21.9
2.73
1.63
0.44
1
0.91
0.93
8.3685
7.6289
7.4286
7.4263
7.3565
7.3405
3.8752
3.5226
3.5008
2.4934
2.4900
abundance
X : parts per Million : 1H


---- ACQUISITION PARAMETERS ----
File Name = YM100826-1.2
Author = delta
Sample ID = YM100826-1
Content = single pulse decouple
Creation Date = 26-AUG-2010 16:47:57
Revision Date = 26-AUG-2010 16:48:48
Spec Site = ECA500
Spec Type = DELTA2_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 26214
Dim Units = [ppm]
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 800
Total_scans = 800
X_90_width = 9.5[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.4[dB]
X_pulse = 3.16666667[us]
Irr_atn_dec = 21.8[dB]
Irr_atn_noe = 21.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
OB
OTf
OMe
3
JEOL
(Millions)
800.0
700.0
600.0
500.0
400.0
300.0
200.0
100.0
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
−10.0
−20.0
X : parts per Million : 13C

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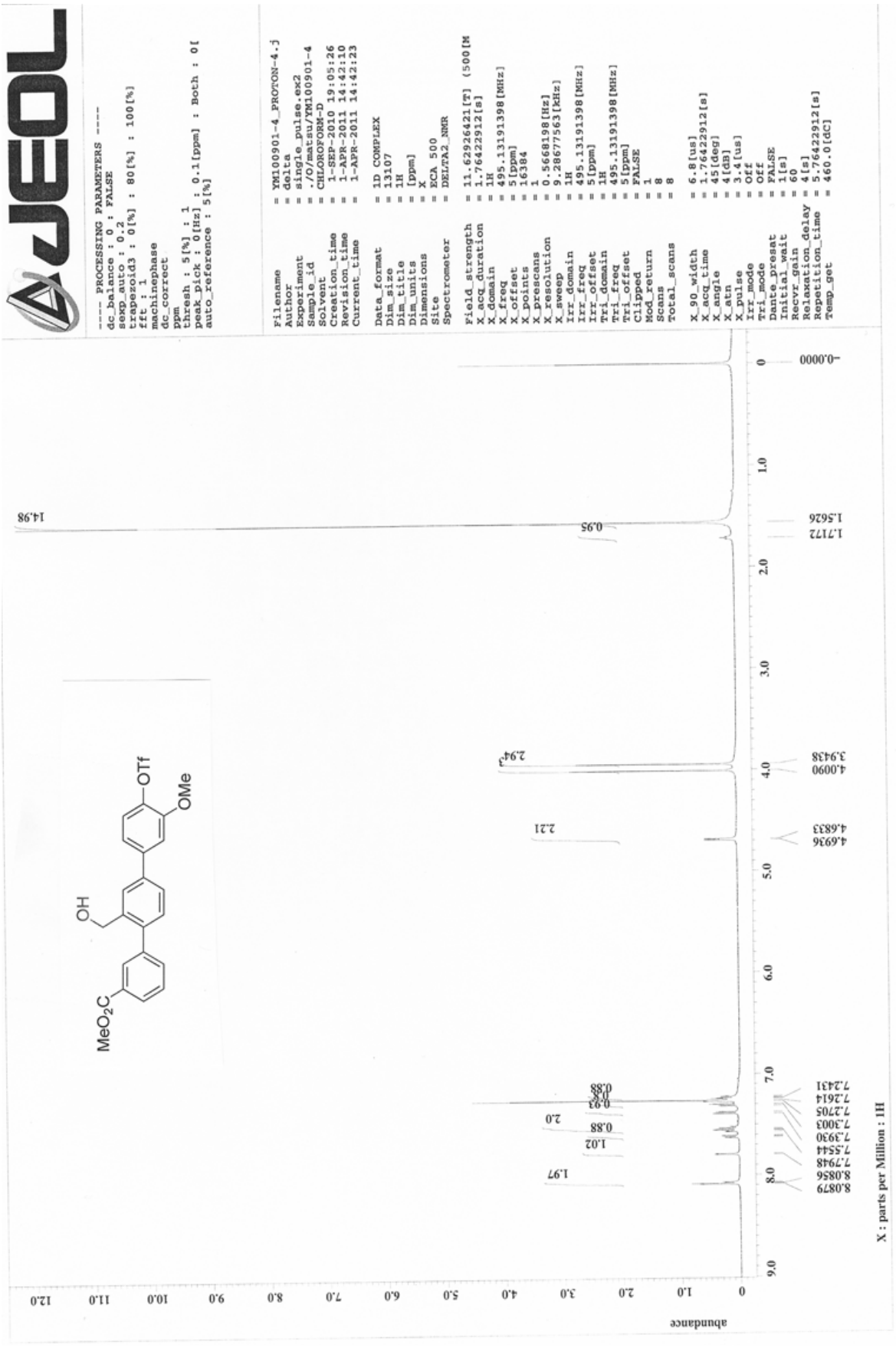
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
ffc_1a
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[
auto_reference : 5[%]
Filename = YM100901-4_PROTON-4.j
Author = delta
Experiment = single_pulse.ex2
Sample_id = ./O/matsu/YM100901-4
Solvent = CHLOROFORM-D
Creation_time = 1-SEP-2010 19:05:26
Revision_time = 1-APR-2011 14:42:10
Current_time = 1-APR-2011 14:42:23
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
total_scans = 8
X_90_width = 6.8[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 3.4[us]
Irr_mode = Off
Tri_mode = Off
Date_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 60
Relaxation_delay = 4[s]
Repetition_time = 5.76422912[s]
Temp_get = 460.0[dC]
14.98
0.95
2.94
2.21
1.97
2.0
1.02
0.88
0.93
0.88
1.5626
1.7172
3.9438
4.0090
4.6833
4.6936
7.2431
7.2614
7.2705
7.3003
7.3930
7.5544
7.7948
8.0856
8.0879
OTf
OMe
OH
MeO_2C
abundance
X : parts per Million : 1H

---- ACQUISITION PARAMETERS ----
File Name = YM100824-1-3-3.4
Author = daiba
Sample ID = YM100824-1-3-3
Content = single pulse decouple
Creation Date = 24-AUG-2010 15:19:38
Revision Date = 24-AUG-2010 15:19:06
Spec Site = ECA500
Spec Type = DELTA2_NMR
Data Format = 1D COMPLEX
Dimensions = 1
Dim Title = 13C
Dim Size = 26214
Dim Units = [ppm]
Field_strength = 11.7373578[T] (500[MH
X_acq_duration = 0.83361792[s] (
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100.00[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_scans = 36.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 900
Total_scans = 900
X_90_width = 9.5[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atten = 5.1[dB]
X_pulse = 3.16666667[us]
Irr_atn_dec = 21.8[dB]
Irr_atn_noe = 21.8[dB]
Irr_mode = NNESTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
---- PROCESSING PARAMETERS ----
dc_balance
exp_lb [Hz] : 0.0[s]
trapazoid3 : 0[%] : 80[%] : 100[%]
zerofill : 2
fft : FORWARD
machinephase
ppm
OH
MeO2C
OTf
OMe
JEOL
(Millions)
400.0
300.0
200.0
100.0
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
X : parts per Million : 13C

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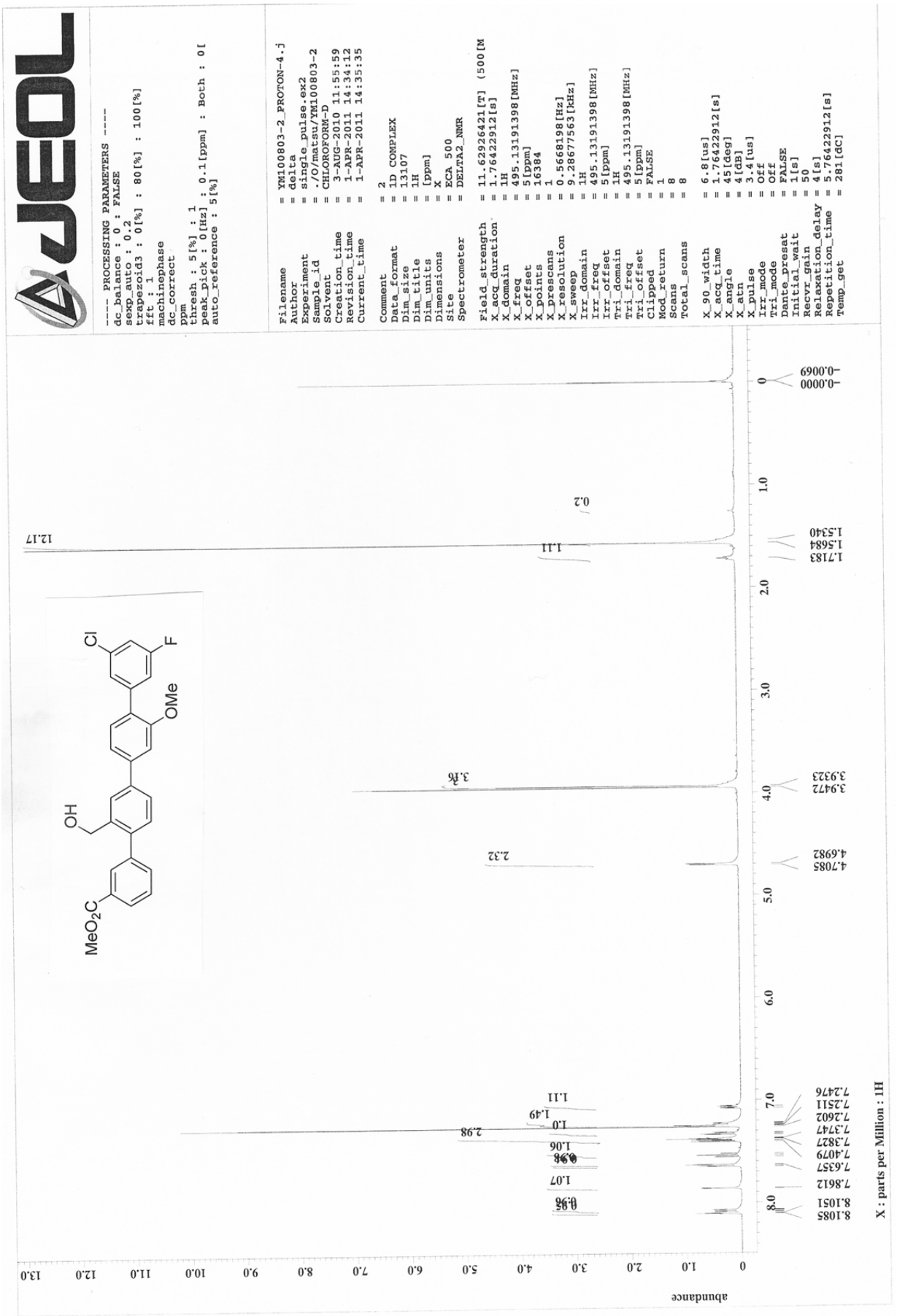
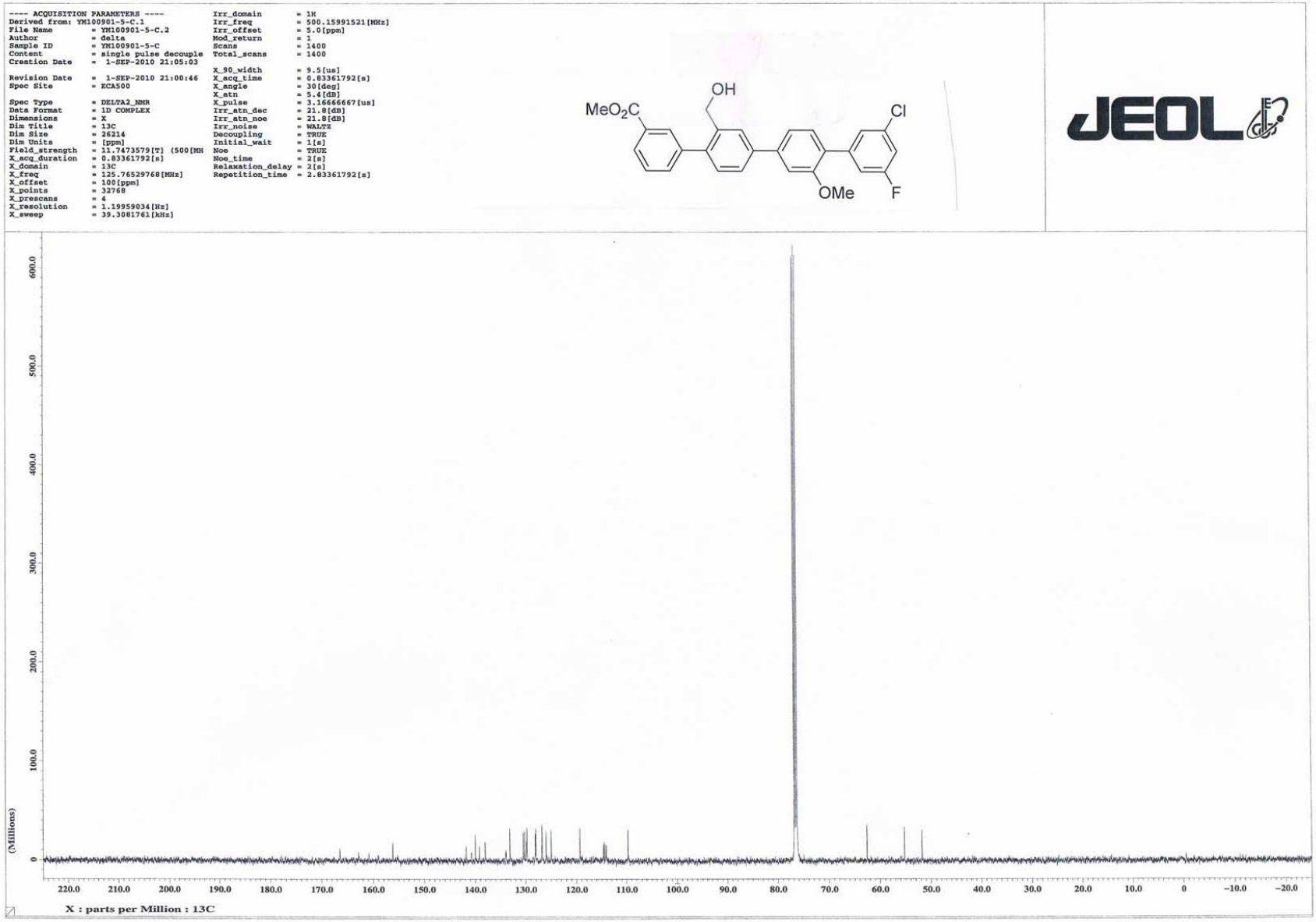

JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[
auto_reference : 5[%]
Filename = YM100803-2_PROTON-4.j
Author = delta
Experiment = single_pulse.ex2
Sample_id = ./O/matsu/YM100803-2
Solvent = CHLOROFORM-D
Creation_time = 3-AUG-2010 11:55:59
Revision_time = 1-APR-2011 14:34:12
Current_time = 1-APR-2011 14:35:35
Comment = 2
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 6.8[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 3.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 4[s]
Repetition_time = 5.76422912[s]
Temp_get = 281[dC]
Cl
F
OMe
OH
MeO2C
12.17
8.1085
8.1051
7.8612
7.6357
7.4079
7.3827
7.3747
7.2602
7.2511
7.2476
4.7085
4.6982
3.9472
3.9323
1.7183
1.5684
1.5340
-0.0000
-0.0069
X : parts per Million : 1H
abundance


---- ACQUISITION PARAMETERS ----
Derived from: YM100901-5-C.1
File Name = YM100901-5-C.2
Author = delta
Sample ID = YM100901-5-C
Content = single pulse decouple
Creation Date = 1-SEP-2010 21:05:03
Revision Date = 1-SEP-2010 21:00:46
Spec Site = ECA500
Spec Type = DELTA2_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 26214
Dim Units = [ppm]
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 1400
Total_scans = 1400
X_90_width = 9.5[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.4[dB]
X_pulse = 3.16666667[us]
Irr_atn_dec = 21.8[dB]
Irr_atn_noe = 21.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
MeO2C
OH
Cl
OMe
F
JEOL
(Millions)
600.0
500.0
400.0
300.0
200.0
100.0
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
X : parts per Million : 13C

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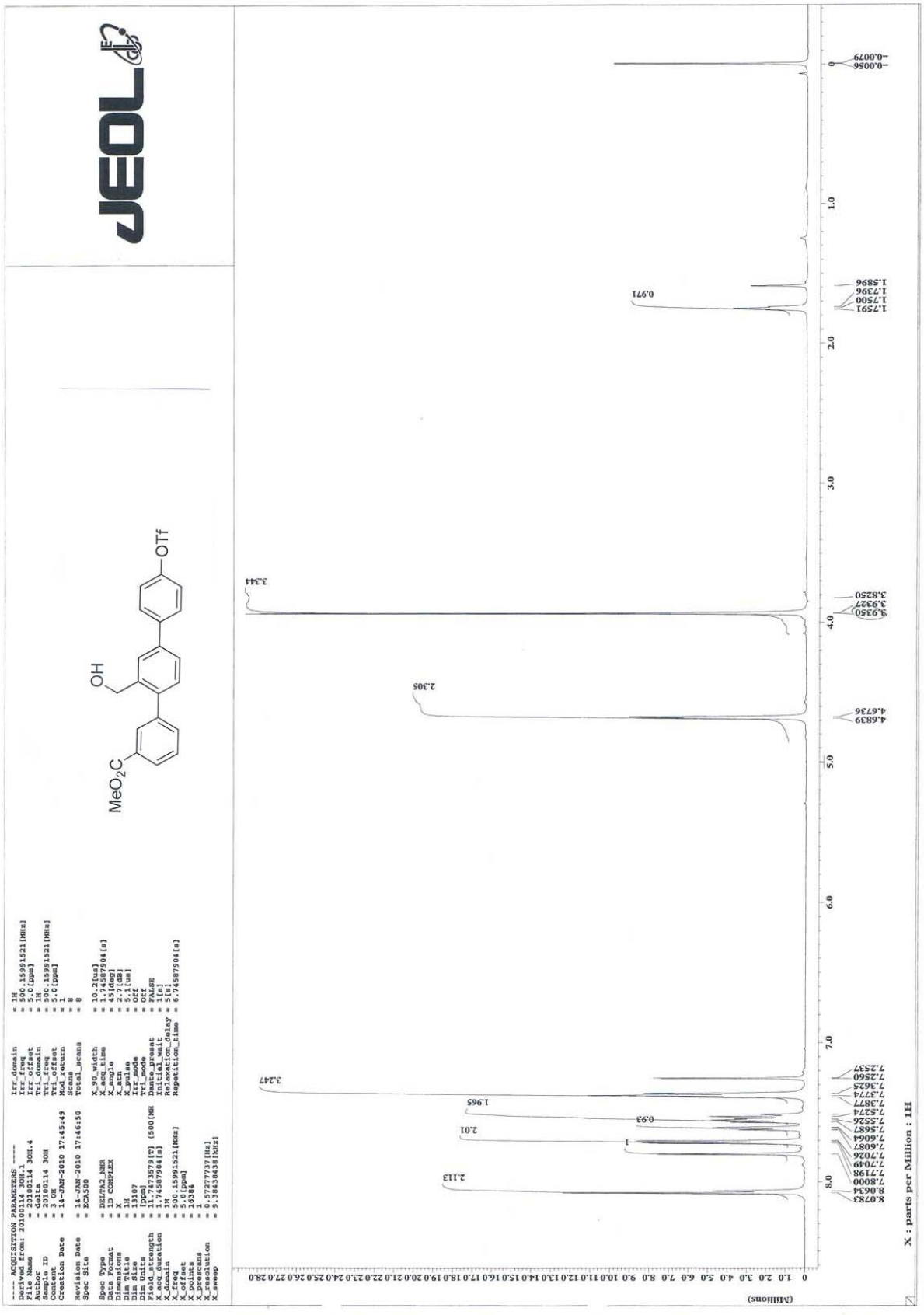
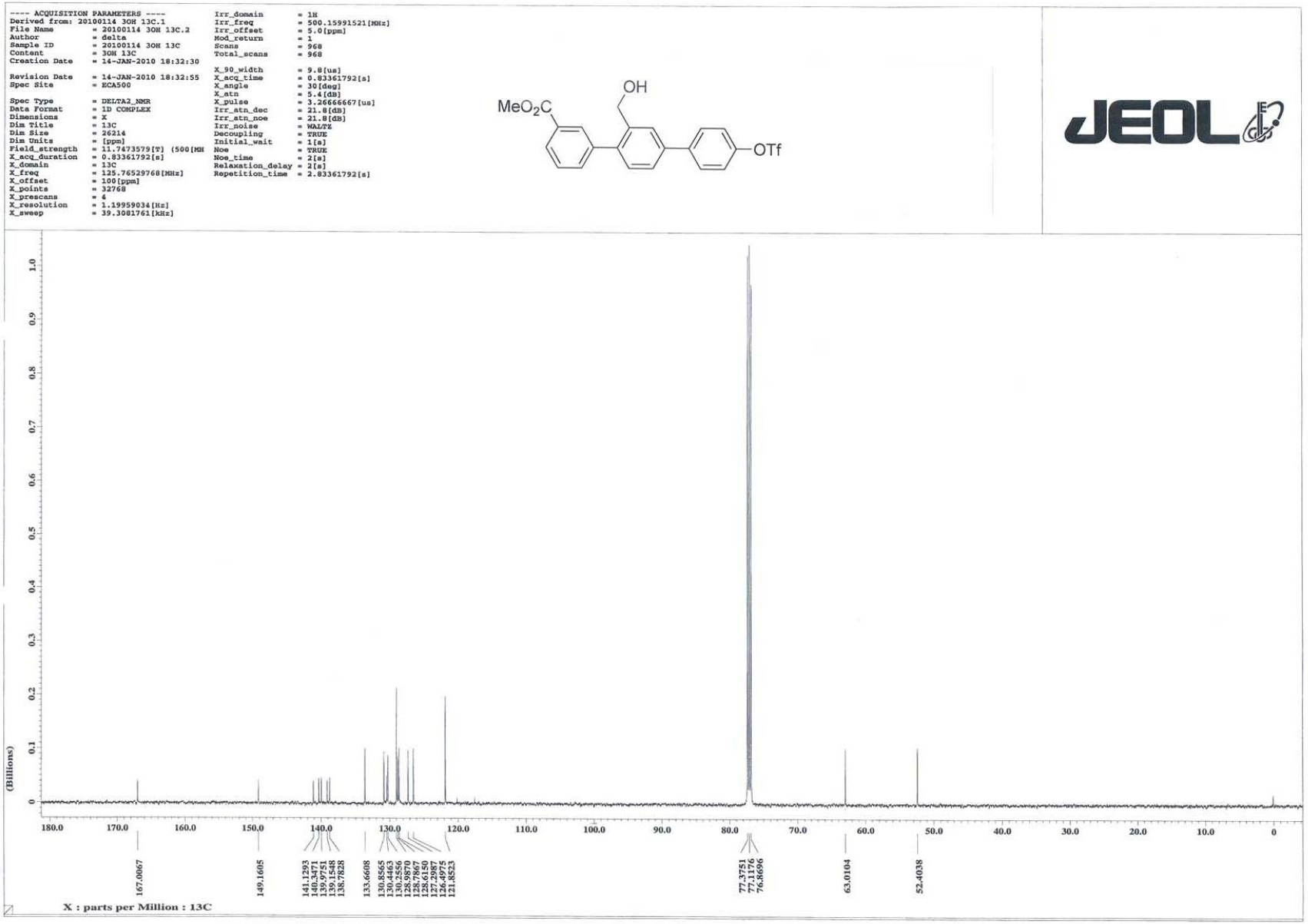

JEOL
OH
MeO2C
OTf
3.344
2.305
3.247
1.965
2.113
2.01
0.971
0.93
1
X : parts per Million : 1H
(Million)


---- ACQUISITION PARAMETERS ----
Derived from: 20100114 3OH 13C.1
File Name = 20100114 3OH 13C.2
Author = delta
Sample ID = 20100114 3OH 13C
Content = 3OH 13C
Creation Date = 14-JAN-2010 18:32:30
Revision Date = 14-JAN-2010 18:32:55
Spec Site = ECA500
Spec Type = DEL/TA2_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 26214
Dim Units = [ppm]
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Mod_return = 1
Scans = 968
Total_scans = 968
X_90_width = 9.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.4[dB]
X_pulse = 3.26666667[us]
Irr_atn_dec = 21.8[dB]
Irr_atn_noe = 21.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
MeO2C
OH
OTf
JEOL
(Billions)
167.0067
149.1605
141.1293
140.5471
139.3751
139.1548
138.7828
133.6608
130.8565
130.4463
130.1856
128.9870
128.7867
128.6150
127.2987
124.6975
121.8523
77.3751
77.1176
76.8696
63.0104
52.4038
X : parts per Million : 13C

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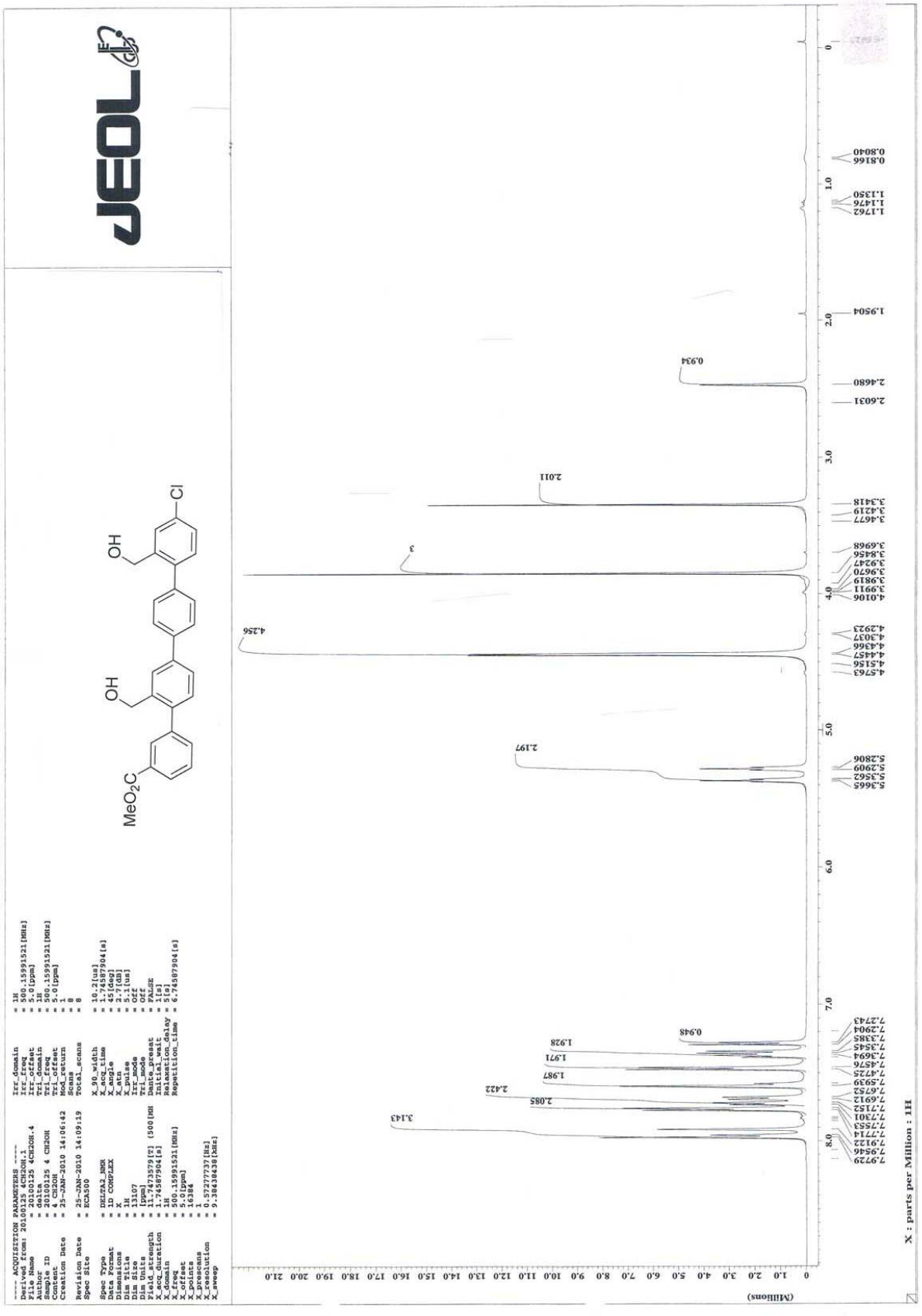
JEOL
OH
OH
Cl
MeO2C
3.143
2.422
2.085
1.987
1.971
1.928
0.948
2.197
4.256
3
2.011
0.934
X : parts per Million : 1H
(Millions)

JEOL
OH
OH
Cl
MeO2C
166.6920
142.4360
140.4043
138.7065
138.8114
134.4761
130.7420
130.2746
130.1602
130.0553
129.3399
127.9187
127.2605
127.0602
125.8584
61.4556
60.9787
52.8044
40.1852
40.0135
39.8513
(Billions)
X : parts per Million : 13C

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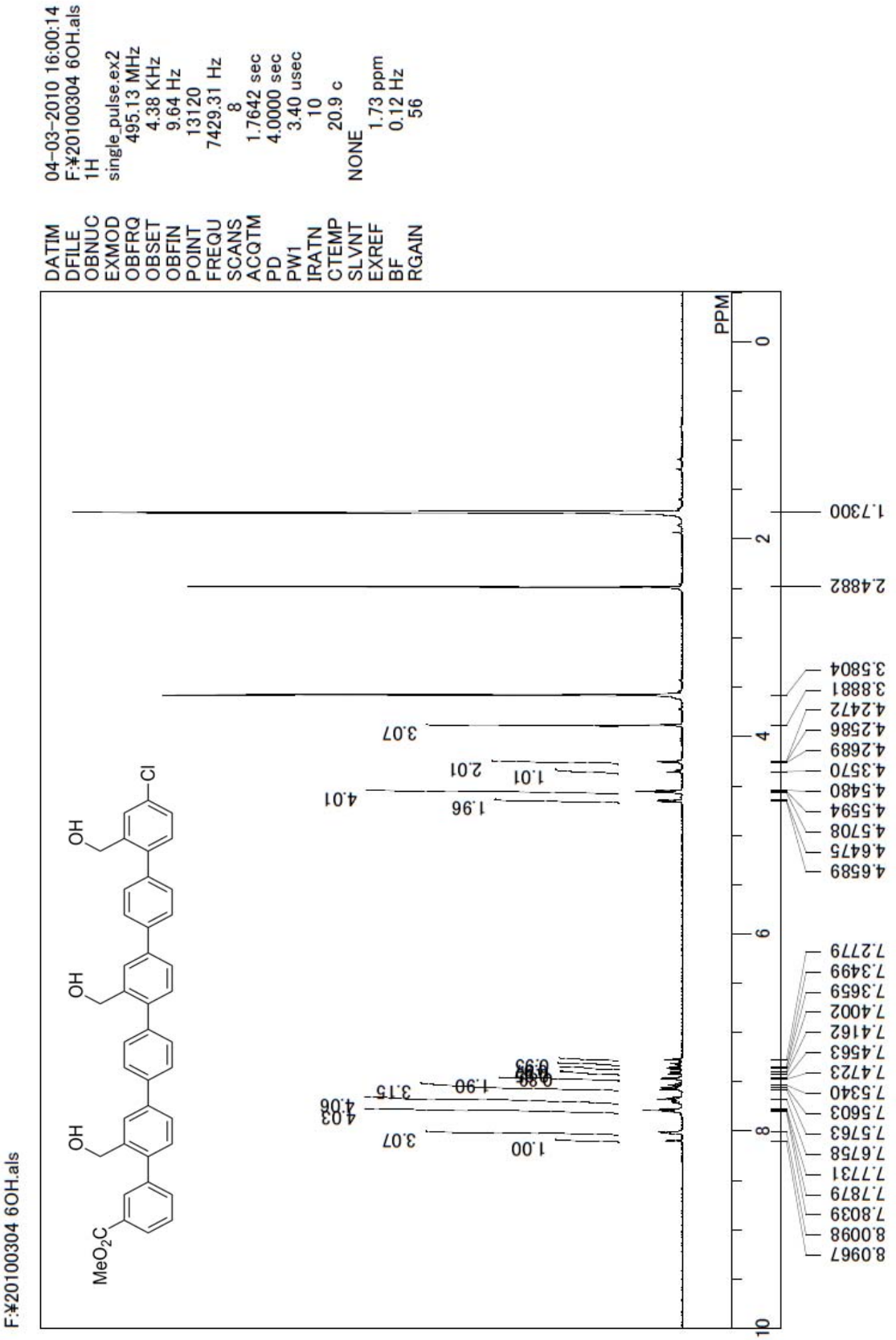
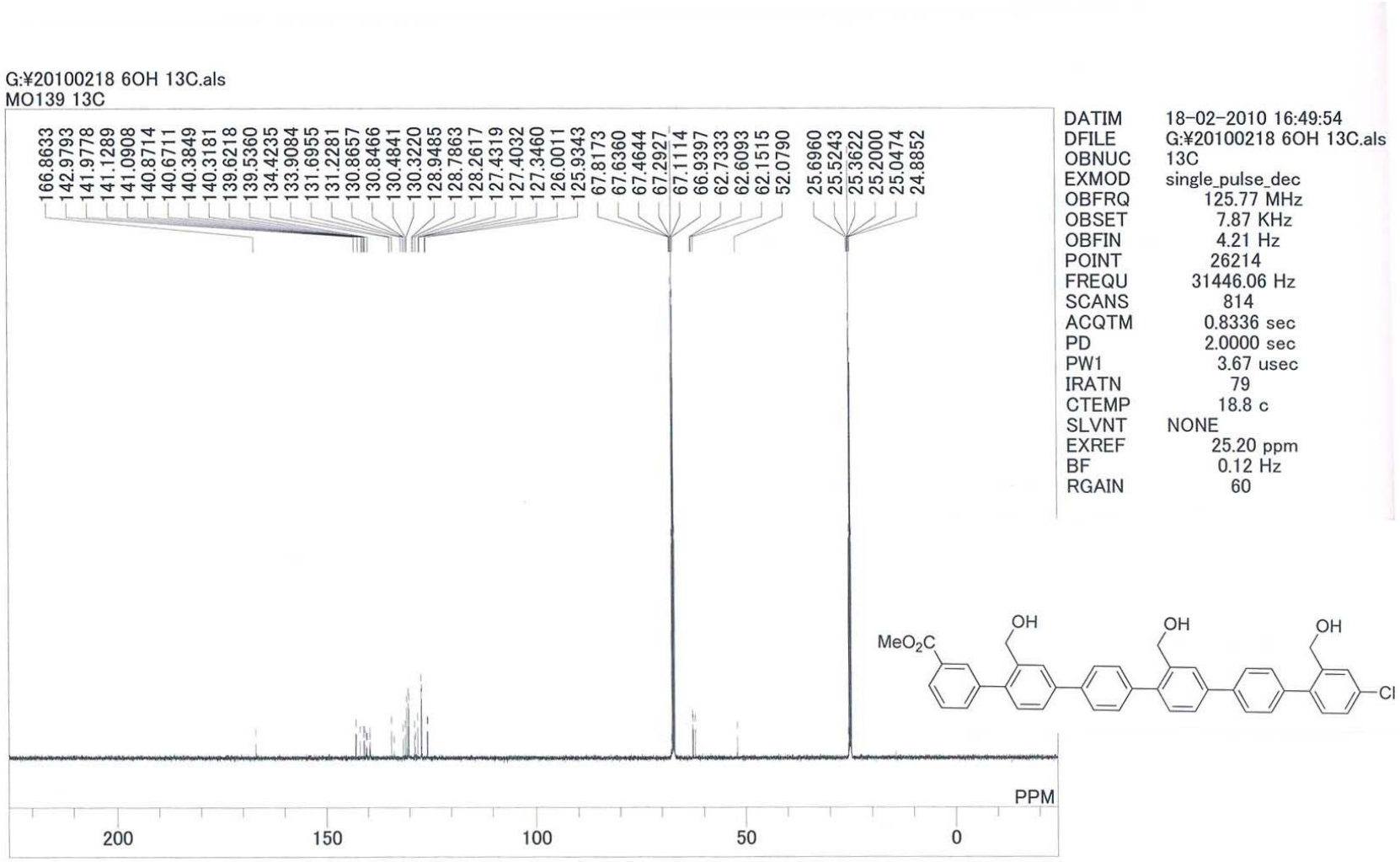

DATIM 04-03-2010 16:00:14
DFILE F:¥20100304 6OH.als
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13120
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 4.0000 sec
PW1 3.40 usec
IRATN 10
CTEMP 20.9 c
SLVNT NONE
EXREF 1.73 ppm
BF 0.12 Hz
RGAIN 56
F:¥20100304 6OH.als
PPM
1.7300
2.4882
3.5804
3.8881
4.2472
4.2586
4.2689
4.3570
4.5480
4.5594
4.5708
4.6475
4.6589
7.2779
7.3499
7.3659
7.4002
7.4162
7.4563
7.4723
7.5340
7.5603
7.5763
7.6758
7.7731
7.7879
7.8039
8.0098
8.0967
3.07
2.01
1.01
1.96
4.01
1.00
3.07
4.03
4.06
1.90
3.15
OH
OH
OH
Cl
MeO2C


G:¥20100218 6OH 13C.als
MO139 13C
166.8633
142.9793
141.9778
141.1289
141.0908
140.8714
140.6711
140.3849
140.3181
139.6218
139.5360
134.4235
133.9084
131.6955
131.2281
130.8657
130.8466
130.4841
130.3220
128.9485
128.7863
128.2617
127.4319
127.4032
127.3460
126.0011
125.9343
67.8173
67.6360
67.4644
67.2927
67.1114
66.9397
62.7333
62.6093
62.1515
52.0790
25.6960
25.5243
25.3622
25.2000
25.0474
24.8852
DATIM 18-02-2010 16:49:54
DFILE G:¥20100218 6OH 13C.als
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 814
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRATN 79
CTEMP 18.8 c
SLVNT NONE
EXREF 25.20 ppm
BF 0.12 Hz
RGAIN 60
MeO2C
OH
OH
OH
Cl
PPM
200
150
100
50
0