

Supporting Information

Chan-Lam Coupling Reaction of Sulfamoyl Azides with Arylboronic Acids for Synthesis of Unsymmetrical *N*-Arylsulfamides

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Experimental

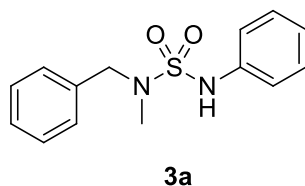
I . Materials and Methods

Unless otherwise indicated, all chemical reagents were purchased from commercial suppliers and were used without further purification. All reactions were carried out in oven-dried glassware equipped with a magnetic stir bar. Reactions were monitored by thin layer chromatography (TLC) with 0.25-mm pre-coated silica gel plates (Kieselgel 60F₂₅₄). Products were detected by UV light, by staining with *p*-anisaldehyde solution composed of acetic acid, sulfuric acid, and MeOH, or by staining with a KMnO₄ solution composed of potassium carbonate, sodium hydroxide, and water. Flash column chromatography was performed on silica gel (70-230 mesh). Yields refer to chromatographically and spectroscopically pure compounds unless otherwise noted. ¹H and ¹³C spectra were recorded on a 300 MHz NMR spectrometer. Chemical shifts are reported as δ values relative to internal SiMe₄ or chloroform (δ 0.00 for ¹H and δ 77.0 for ¹³C). IR spectra were measured as neat oils or solids on a FT-IR spectrometer. HRMS data were obtained by electron ionization with a double-focusing high-resolution magnetic sector mass analyzer.

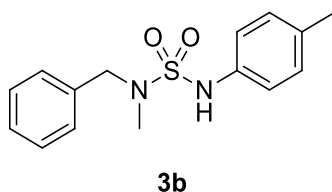
II. Experimental Section

General procedure

A sealed tube was charged with an arylboronic acid (2.0 mmol), CuCl (10 mol %) and the corresponding an organo azide (1.0 mmol). MeOH (1.0 mL) was then added to the flask. The reaction mixture was stirred at room temperature in an open flask. After completion of the reaction, the reaction mixture was filtered through Celite and was washed with EtOAc. The filtrate was collected and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel to obtain the desired product.

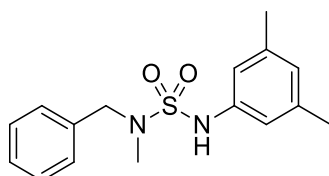


Compound 3a. General procedure was used employing phenylboronic acid (0.108 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 45 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3a** (0.111 g, 0.402 mmol, 91%) as a white solid. m.p: 76-78 °C; R_f 0.16 (hexanes/ethyl acetate = 5:1); IR (neat) 3270, 2361, 1967, 1601, 1412, 1342, 1222, 1152, 825 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.35-7.25 (m, 5H), 7.22-7.12 (m, 5H), 7.05 (s, 1H), 4.31 (s, 2H), 2.71 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 137.1, 135.6, 129.3, 128.6, 128.2, 127.8, 124.6, 120.3, 54.5, 34.5; HRMS-EI: m/z 276.0935 $[(M)^+]$; calcd for $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_2\text{S}^+$: 276.0932].



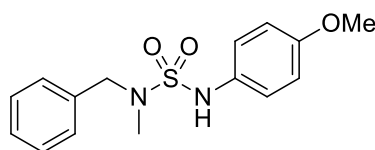
Compound 3b. General procedure was used employing *p*-tolylboronic acid (0.120 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 45 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3b** (0.105 g, 0.362 mmol, 82%) as a white solid. m.p: 100-102 °C; R_f 0.19 (hexanes/ethyl acetate = 5:1); IR (neat) 3393, 2360, 1967, 1634, 1512, 1454, 1226, 1152, 984, 726 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.32-7.26 (m, 3H), 7.22-7.18 (m, 2H), 7.15-7.07 (m, 4H), 6.45 (s, 1H), 4.29 (s, 2H), 2.70 (s, 3H), 2.34 (s, 3H); ^{13}C NMR

(75 MHz, CDCl₃) δ 135.8, 134.5, 134.4, 129.8, 128.5, 128.2, 127.7, 121.0, 54.5, 34.5, 20.7; HRMS-EI: m/z 290.1090 [(M)⁺; calcd for C₁₅H₁₈N₂O₂S⁺: 290.1089].



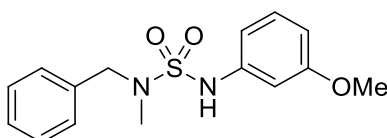
3c

Compound 3c. General procedure was used employing 3,5-dimethylphenylboronic acid (0.132 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 45 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3c** (0.121 g, 0.398 mmol, 90%) as a white solid. m.p: 66-68 °C; R_f 0.22 (hexanes/ethyl acetate = 5:1); IR (neat) 3270, 2921, 2361, 1603, 1398, 11150, 1044, 987, 728, 583 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.36 (s, 1H), 7.27-7.18 (m, 5H), 6.84 (s, 2H), 6.76 (s, 1H), 4.32 (s, 2H), 2.69 (s, 3H), 2.27 (s, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 138.9, 136.9, 135.8, 128.5, 128.2, 127.7, 126.1, 117.8, 54.4, 34.5, 21.2; HRMS-EI: m/z 304.1248 [(M)⁺; calcd for C₁₆H₂N₂O₂S⁺: 304.1245].



3d

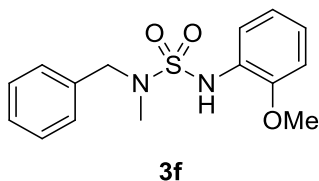
Compound 3d. General procedure was used employing 4-methoxyphenylboronic acid (0.134 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 45 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3d** (0.122 g, 0.398 mmol, 90%) as a white solid. m.p: 54-56 °C; R_f 0.10 (hexanes/ethyl acetate = 5:1); IR (neat) 3511, 2959, 2360, 1967, 1510, 1455, 1395, 1152, 937, 729 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.31-7.23 (m, 3H), 7.21-7.18 (m, 2H), 7.17-7.15 (m, 2H), 6.92 (s, 1H), 6.88-6.83 (m, 2H), 4.25 (s, 2H), 3.79 (s, 3H), 2.68 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 157.3, 135.8, 129.7, 128.5, 128.1, 127.7, 124.1, 114.4, 55.4, 54.5, 34.6; HRMS-EI: m/z 306.1039 [(M)⁺; calcd for C₁₅H₁₈N₂O₃S⁺: 306.1038].



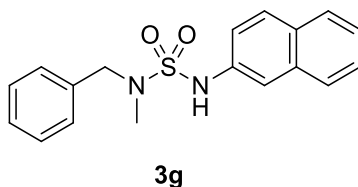
3e

Compound 3e. General procedure was used employing 3-methoxyphenylboronic acid (0.134 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 45 min. Flash column

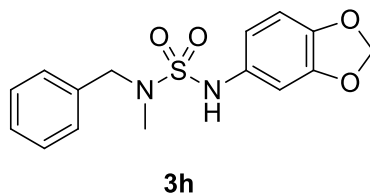
chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3e** (0.134 g, 0.437 mmol, 99%) as a white solid. m.p: 72-74 °C; R_f 0.19 (hexanes/ethyl acetate = 5:1); IR (neat) 3545, 3270, 2361, 1967, 1606, 1498, 1400, 1148, 973, 729 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.31-7.25 (m, 3H), 7.24-7.23 (m, 1H), 7.22-7.20 (m, 2H), 7.18 (s, 1H), 6.82-6.76 (m, 2H), 6.68 (dt, J = 0.8 Hz and J = 8.4 Hz, 1H), 4.32 (s, 2H), 3.77 (s, 3H), 2.71 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.4, 138.4, 135.6, 130.1, 128.6, 128.2, 127.8, 112.3, 110.1, 105.9, 55.3, 54.5, 34.6; HRMS-EI: m/z 306.1036 $[(\text{M})^+]$; calcd for $\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_3\text{S}^+$: 306.1038].



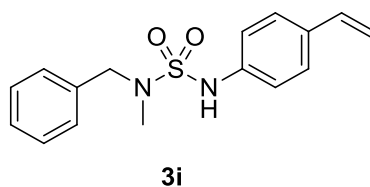
Compound 3f. General procedure was used employing 2-methoxyphenylboronic acid (0.134 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 3 h. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3f** (0.041 g, 0.133 mmol, 30%) as a yellow solid. m.p: 60-64 °C; R_f 0.22 (hexanes/ethyl acetate = 5:1); IR (neat) 3394, 2361, 1967, 1650, 1501, 1343, 1253, 1112, 821, 752 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.54 (dd, J = 1.6 Hz and J = 8.0 Hz, 1H), 7.31-7.24 (m, 3H), 7.18-7.15 (m, 2H), 7.10 (td, J = 1.6 Hz and J = 15.7 Hz, 1H), 6.99-6.93 (m, 1H), 6.96 (s, 1H), 6.89 (dd, J = 1.3 Hz and J = 8.1 Hz, 1H), 4.26 (s, 2H), 3.86 (s, 3H), 2.67 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 148.8, 135.8, 128.5, 128.2, 127.7, 126.6, 124.6, 121.1, 120.0, 110.5, 55.7, 54.6, 34.5; HRMS-EI: m/z 306.1036 $[(\text{M})^+]$; calcd for $\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_3\text{S}^+$: 306.1038]



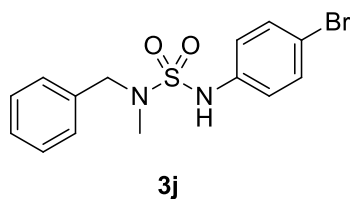
Compound 3g. General procedure was used employing 2-naphthylboronic acid (0.152 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 40 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3g** (0.128 g, 0.393 mmol, 89%) as a white solid. m.p: 113-116 °C; R_f 0.12 (hexanes/ethyl acetate = 5:1); IR (neat) 3749, 2361, 1967, 1422, 1266, 1156, 897, 742, 518 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.81-7.74 (m, 3H), 7.62 (d, J = 2.4 Hz, 1H), 7.51-7.40 (m, 2H), 7.34 (dd, J = 2.5 Hz and J = 8.8 Hz, 1H), 7.32 (s, 1H), 7.25-7.16 (m, 5H), 4.35 (s, 2H), 2.73 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 135.6, 134.6, 133.7, 130.7, 129.4, 128.6, 128.2, 127.9, 127.7, 127.4, 126.7, 125.3, 120.2, 116.9, 54.6, 34.7; HRMS-EI: m/z 326.1091 $[(\text{M})^+]$; calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_2\text{S}^+$: 326.1089].



Compound 3h. General procedure was used employing 3,4-methylenedioxyphenylboronic acid (0.147 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 55 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3h** (0.113 g, 0.354 mmol, 80%) as a white solid. m.p: 102-104 °C; R_f 0.14 (hexanes/ethyl acetate = 5:1); IR (neat) 3434, 2361, 2227, 1967, 1649, 1488, 1350, 1153, 1037, 729 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.31-7.25 (m, 3H), 7.24-7.18 (m, 2H), 6.90 (s, 1H), 6.83 (d, $J = 2.1$ Hz, 1H), 6.73 (d, $J = 8.2$ Hz, 1H), 6.65 (dd, $J = 2.2$ Hz and $J = 8.2$ Hz, 1H), 5.96 (s, 2H), 4.28 (s, 2H), 2.70 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 148.1, 145.4, 135.8, 130.9, 128.6, 128.2, 127.8, 115.7, 108.3, 104.5, 101.5, 54.6, 34.7; HRMS-EI: m/z 320.0834 $[(\text{M})^+]$; calcd for $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_4\text{S}^+$: 320.0831].

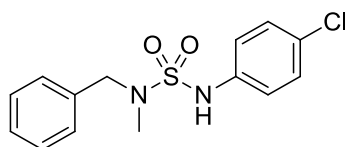


Compound 3i. General procedure was used employing 4-vinylphenylboronic acid (0.131 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 70 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3i** (0.118 g, 0.389 mmol, 88%) as a white solid. m.p: 84-86 °C; R_f 0.16 (hexanes/ethyl acetate = 5:1); IR (neat) 3441, 3270, 2360, 2223, 1967, 1649, 1230, 1154, 990, 728 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.36 (d, $J = 8.5$ Hz, 2H), 7.30-7.26 (m, 2H), 7.25-7.24 (m, 1H), 7.20-7.14 (m, 4H), 7.10 (s, 1H), 6.73-6.63 (m, 1H), 5.70 (dd, $J = 0.7$ Hz and $J = 17.5$ Hz, 1H), 5.22 (dd, $J = 0.7$ Hz and $J = 11.0$ Hz, 1H), 4.31 (s, 2H), 2.71 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 136.6, 135.8, 135.6, 134.0, 128.6, 128.2, 127.8, 127.1, 120.2, 113.4, 54.5, 34.6; HRMS-EI: m/z 302.1086 $[(\text{M})^+]$; calcd for $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2\text{S}^+$: 302.1089].



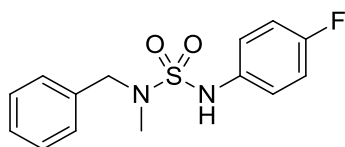
Compound 3j. General procedure was used employing 4-bromophenylboronic acid (0.177 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 12 h. Flash column

chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3j** (0.126 g, 0.354 mmol, 80%) as a white solid. m.p: 106-108 °C; R_f 0.23 (hexanes/ethyl acetate = 5:1); IR (neat) 3439, 2361, 1967, 1490, 1333, 1276, 1154, 903, 821, 749 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.43-7.39 (m, 2H), 7.38 (s, 1H), 7.31-7.24 (m, 3H), 7.18-7.15 (m, 2H), 7.10-7.05 (m, 2H), 4.30 (s, 2H), 2.70 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 136.3, 135.3, 132.3, 128.7, 128.2, 128.0, 121.7, 117.5, 54.5, 34.6; HRMS-EI: m/z 354.0035 $[(\text{M})^+]$; calcd for $\text{C}_{14}\text{H}_{15}\text{BrN}_2\text{O}_2\text{S}^+$: 354.0038].



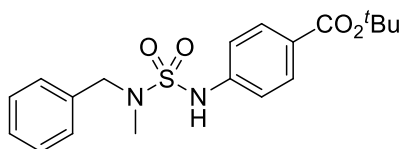
3k

Compound 3k. General procedure was used employing 4-chlorophenylboronic acid (0.138 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 12 h. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3k** (0.097 g, 0.314 mmol, 71%) as a white solid. m.p: 108-110 °C; R_f 0.20 (hexanes/ethyl acetate = 5:1); IR (neat) 3437, 3261, 2361, 1967, 1648, 1491, 1338, 1153, 985, 746 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.32 (s, 1H), 7.31-7.25 (m, 5H), 7.18-7.11 (m, 4H), 4.30 (s, 2H), 2.70 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 135.7, 135.4, 130.0, 129.4, 128.7, 128.2, 128.0, 121.5, 54.5, 34.6; HRMS-EI: m/z 310.0540 $[(\text{M})^+]$; calcd for $\text{C}_{14}\text{H}_{15}\text{ClN}_2\text{O}_2\text{S}^+$: 310.0543].



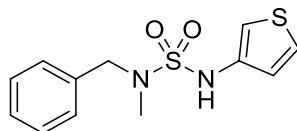
3l

Compound 3l. General procedure was used employing 4-fluorophenylboronic acid (0.124 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 3 h. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3l** (0.118 g, 0.402 mmol, 91%) as a yellow solid. m.p: 90-92 °C; R_f 0.31 (hexanes/ethyl acetate = 5:1); IR (neat) 3438, 3269, 2361, 1967, 1650, 1508, 1390, 1151, 905, 775 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.32-7.20 (m, 5H), 7.19-7.18 (m, 1H), 7.17 (s, 1H), 7.16-7.14 (m, 1H), 7.05-6.96 (m, 2H), 4.28 (s, 2H), 2.70 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 161.7, 158.5, 135.5, 133.0, 128.6, 128.2, 127.9, 123.1, 123.0, 116.2, 115.9, 54.5, 34.6; HRMS-EI: m/z 294.0839 $[(\text{M})^+]$; calcd for $\text{C}_{14}\text{H}_{15}\text{FN}_2\text{O}_2\text{S}^+$: 294.0838].



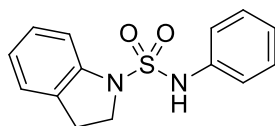
3m

Compound 3m. General procedure was used employing 4-methoxycarbonylphenylboronic acid (0.196 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 3 h. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3m** (0.133 g, 0.354 mmol, 80%) as a white solid. m.p: 108-110 °C; R_f 0.20 (hexanes/ethyl acetate = 5:1); IR (neat) 3398, 3270, 2361, 1967, 1709, 1511, 1297, 1155, 926, 770 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.95 (d, J = 8.8 Hz, 2H), 7.89 (s, 1H), 7.29-7.24 (m, 5H), 7.21-7.20 (m, 2H), 4.34 (s, 2H), 2.72 (s, 3H), 1.60 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 165.3, 141.2, 135.3, 130.9, 128.6, 128.2, 127.9, 127.2, 118.0, 81.0, 54.5, 34.5, 28.1; HRMS-EI: m/z 376.1456 $[(\text{M})^+]$; calcd for $\text{C}_{19}\text{H}_{24}\text{N}_2\text{O}_4\text{S}^+$: 376.1457].



3n

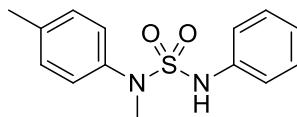
Compound 3n. General procedure was used employing 3-thiophenylphenylboronic acid (0.113 g, 0.884 mmol) and sulfamoyl azide (0.100 g, 0.442 mmol). The reaction was completed in 12 h. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **3n** (0.102 g, 0.362 mmol, 82%) as a yellow solid. m.p: 50-53 °C; R_f 0.20 (hexanes/ethyl acetate = 5:1); IR (neat) 3503, 3271, 2361, 1967, 1547, 1455, 1369, 1152, 971, 771 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.33-7.24 (m, 4H), 7.22-7.19 (m, 1H), 7.18-7.17 (m, 1H), 7.08 (s, 1H), 6.97 (d, J = 4.2 Hz, 2H), 4.28 (s, 2H), 2.70 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 135.7, 134.9, 128.6, 128.2, 127.9, 125.7, 122.9, 112.7, 54.6, 34.7; HRMS-EI: m/z 282.0497 $[(\text{M})^+]$; calcd for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_2\text{S}_2^+$: 282.0497].



4a

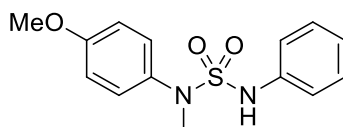
Compound 4a. General procedure was used employing phenylboronic acid (0.109 g, 0.892 mmol) and indoline-1-sulfonyl azide (0.100 g, 0.446 mmol). The reaction was completed in 50 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4a** (0.104 g, 0.380 mmol, 85%) as a white-pink solid. m.p: 110-112 °C; R_f 0.23 (hexanes/ethyl acetate = 5:1); IR (neat) 3271, 2361, 1967, 1649, 1479, 1351, 1243, 1104, 977, 752 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.41 (d, J =

8.0 Hz, 1H), 7.24-7.11 (m, 5H), 7.05-7.00 (m, 3H), 6.69 (s, 1H), 3.89 (t, $J = 8.4$ Hz, 2H), 2.85 (t, $J = 8.5$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.8, 136.2, 131.4, 129.2, 127.7, 125.5, 125.1, 123.4, 121.9, 114.0, 51.4, 27.9; HRMS-EI: m/z 274.0778 $[(\text{M})^+]$; calcd for $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2\text{S}^+$: 274.0776].



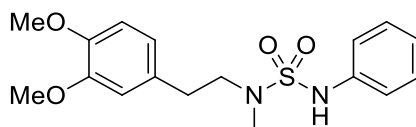
4b

Compound 4b. General procedure was used employing phenylboronic acid (0.108 g, 0.884 mmol) and 1-[(azidosulfonyl)(methyl)amino]-4-methylbenzene (0.100 g, 0.442 mmol). The reaction was completed in 85 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4b** (0.106 g, 0.384 mmol, 87%) as a white solid. m.p: 104-106 °C; R_f 0.26 (hexanes/ethyl acetate = 5:1); IR (neat) 3431, 3270, 2361, 2223, 1967, 1649, 1497, 1349, 1069, 878 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.33-7.24 (m, 2H), 7.15-7.12 (m, 3H), 7.10-7.08 (m, 4H), 6.96 (s, 1H), 3.21 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 139.0, 137.6, 137.3, 129.8, 129.2, 126.9, 124.3, 119.6, 39.8, 21.0; HRMS-EI: m/z 276.0934 $[(\text{M})^+]$; calcd for $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_2\text{S}^+$: 276.0932].



4c

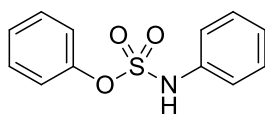
Compound 4c. General procedure was used employing phenylboronic acid (0.101 g, 0.825 mmol) and 1-[(azidosulfonyl)(methyl)amino]-4-methoxybenzene (0.100 g, 0.413 mmol). The reaction was completed in 1 h. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4c** (0.113 g, 0.388 mmol, 94%) as a white solid. m.p: 70-72 °C; R_f 0.14 (hexanes/ethyl acetate = 5:1); IR (neat) 3460, 3271, 2969, 2360, 1603, 1509, 1248, 1145, 926, 755 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.33-7.25 (m, 2H), 7.16-7.07 (m, 5H), 7.03 (s, 1H), 6.81-6.75 (m, 2H), 3.75 (s, 3H), 3.20 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 158.8, 137.4, 134.2, 129.2, 128.6, 124.1, 119.4, 114.3, 55.4, 40.0; HRMS-EI: m/z 292.0884 $[(\text{M})^+]$; calcd for $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_3\text{S}^+$: 292.0882].



4d

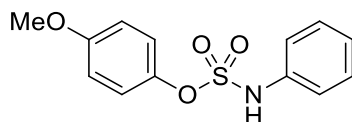
Compound 4d. General procedure was used employing phenylboronic acid (0.081 g, 0.666 mmol) and

4-[2-((azidosulfonyl)(methyl)amino)ethyl]-1,2-dimethoxybenzene (0.100 g, 0.333 mmol). The reaction was completed in 75 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4d** (0.113 g, 0.323 mmol, 97%) as a white solid. m.p: 110-112 °C; R_f 0.03 (hexanes/ethyl acetate = 5:1); IR (neat) 3398, 3270, 2361, 1967, 1624, 1517, 1342, 1150, 1029, 576 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.31-7.28 (m, 2H), 7.13-7.08 (m, 3H), 6.82 (s, 1H), 6.78-6.75 (m, 1H), 6.69-6.67 (m, 2H), 3.84 (s, 1H), 3.83 (s, 1H), 3.44-3.39 (m, 2H), 2.82 (s, 3H), 2.78-2.73 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 148.8, 147.6, 137.2, 130.7, 129.2, 124.3, 120.6, 119.9, 111.9, 111.2, 55.8, 55.7, 52.5, 35.3, 34.0; HRMS-EI: m/z 350.1301 $[(\text{M})^+]$; calcd for $\text{C}_{17}\text{H}_{22}\text{N}_2\text{O}_4\text{S}^+$: 350.1300].



4e

Compound 4e. General procedure was used employing phenylboronic acid (0.122 g, 1.004 mmol) and phenoxy sulfonyl azide (0.100 g, 0.502 mmol). The reaction was completed in 30 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4e** (0.084 g, 0.336 mmol, 67%) as a white solid. R_f 0.31 (hexanes/ethyl acetate = 5:1); ^1H NMR (300 MHz, CDCl_3) δ 7.39-7.26 (m, 5H), 7.24-7.12 (m, 5H), 6.92 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 149.8, 135.9, 129.8, 129.5, 127.3, 125.1, 122.0, 119.6; Data are consistent with those reported in the literature.¹

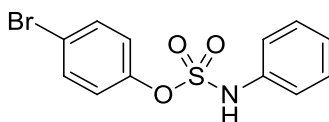


4f

Compound 4f. General procedure was used employing phenylboronic acid (0.106 g, 0.872 mmol) and 4-methoxyphenoxy sulfonyl azide (0.100 g, 0.436 mmol). The reaction was completed in 35 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4f** (0.092 g, 0.331 mmol, 76%) as a white oil. R_f 0.22 (hexanes/ethyl acetate = 5:1); ^1H NMR (300 MHz, CDCl_3) δ 7.40-7.34 (m, 2H), 7.25-7.16 (m, 3H), 7.07-7.01 (m, 2H), 6.82-6.77 (m, 2H), 6.70 (s, 1H), 3.75 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 158.3, 143.3, 136.1, 129.6, 125.0, 123.1, 119.4, 114.6, 55.6; Data are consistent with those reported in the literature.^{1b, 2}

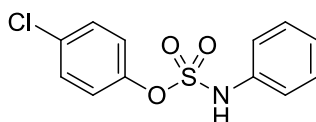
¹ (a) B. Yang, Z. Sun, C. Liu, Y. Cui, Z. Guo, Y. Ren, Z. Lu, S. Knapp, *Tetrahedron Lett.*, 2014, **49**, 6658; (b) V. Subbarayan, L.-M. Jin, C. Xin, X. P. Zhang, *Tetrahedron Lett.*, 2015, **56**, 3431.

² T. Yang, H. Cui, C. Zhang, Li. Zhang, C.-Y. Su, *ChemCatChem.*, 2013, **5**, 3131.



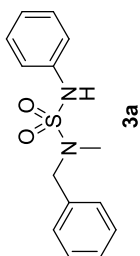
4g

Compound 4g. General procedure was used employing phenylboronic acid (0.088 g, 0.720 mmol) and 4-bromophenoxysulfonyl azide (0.100 g, 0.340 mmol). The reaction was completed in 40 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4g** (0.076 g, 0.231 mmol, 68%) as a white solid. R_f 0.41 (hexanes/ethyl acetate = 5:1); ^1H NMR (300 MHz, CDCl_3) δ 7.41-7.37 (m, 2H), 7.36-7.35 (m, 2H), 7.25-7.18 (m, 3H), 7.04-6.98 (m, 2H), 6.81 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 148.8, 135.6, 132.9, 129.7, 125.5, 123.8, 120.8, 119.8; Data are consistent with those reported in the literature.¹



4h

Compound 4h. General procedure was used employing phenylboronic acid (0.104 g, 0.856 mmol) and 4-chlorophenoxysulfonyl azide (0.100 g, 0.428 mmol). The reaction was completed in 40 min. Flash column chromatography on silica gel using hexanes/ethyl acetate (20:1) provided pure **4h** (0.080 g, 0.282 mmol, 66%) as a white solid. R_f 0.38 (hexanes/ethyl acetate = 5:1); ^1H NMR (300 MHz, CDCl_3) δ 7.43-7.40 (m, 2H), 7.39-7.30 (m, 2H), 7.25-7.20 (m, 3H), 7.12-7.08 (m, 2H), 6.70 (s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 148.3, 135.7, 133.0, 129.9, 129.7, 125.5, 123.4, 119.8; Data are consistent with those reported in the literature.¹



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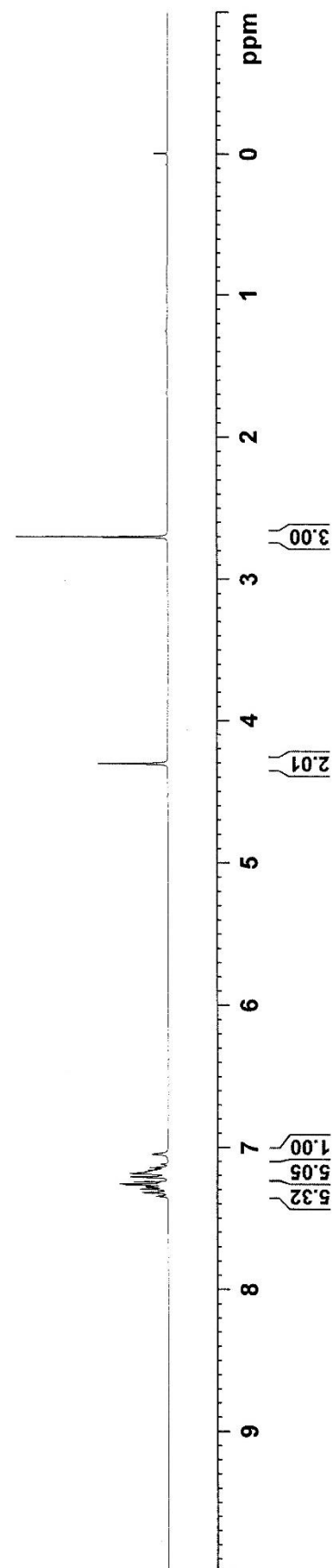
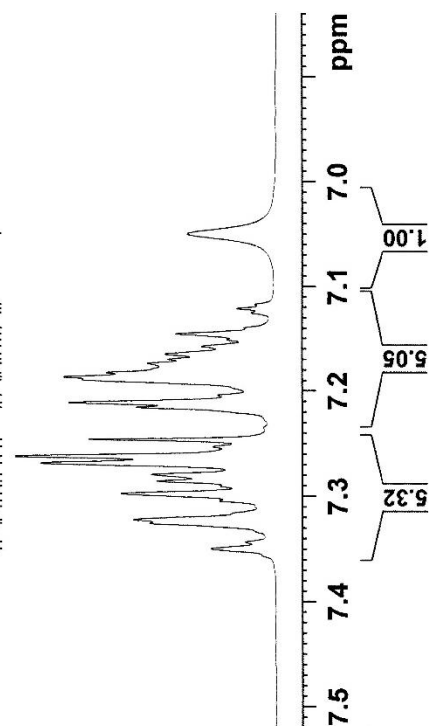
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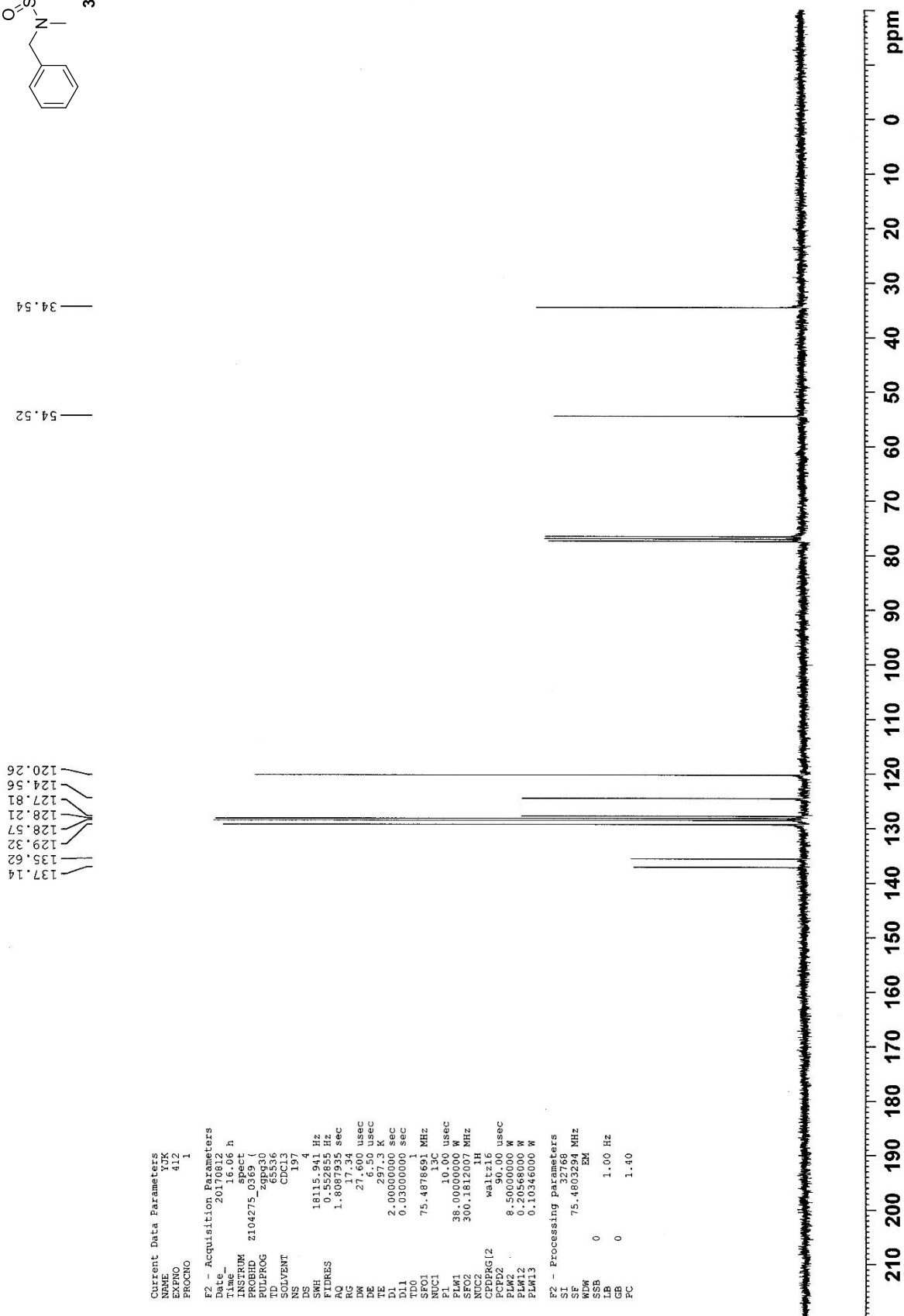
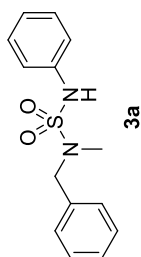
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TDO 0.00000000 sec
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P1 14.00 usec
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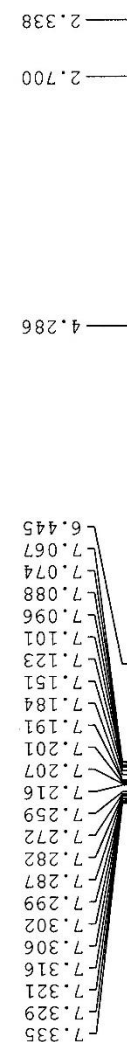
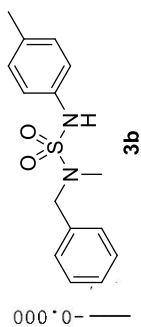




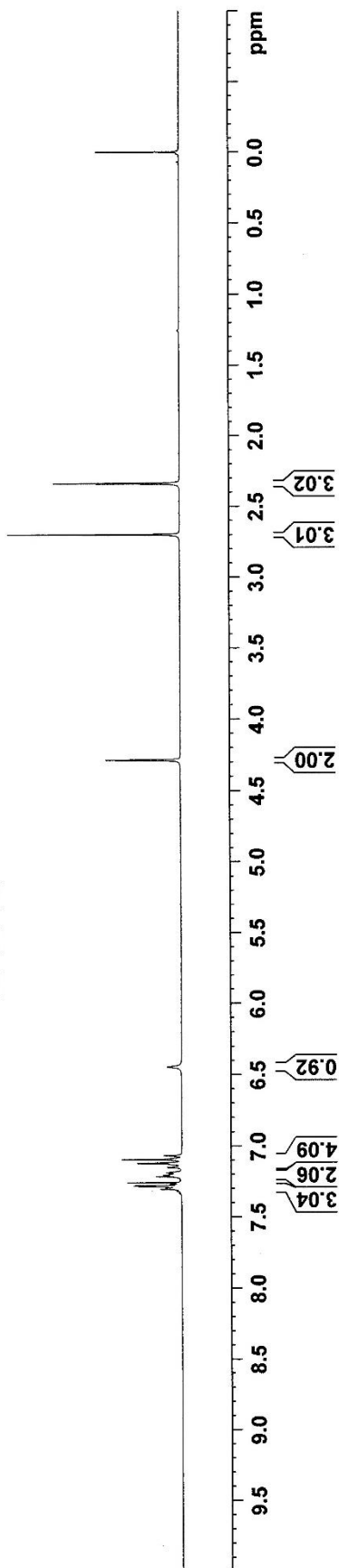
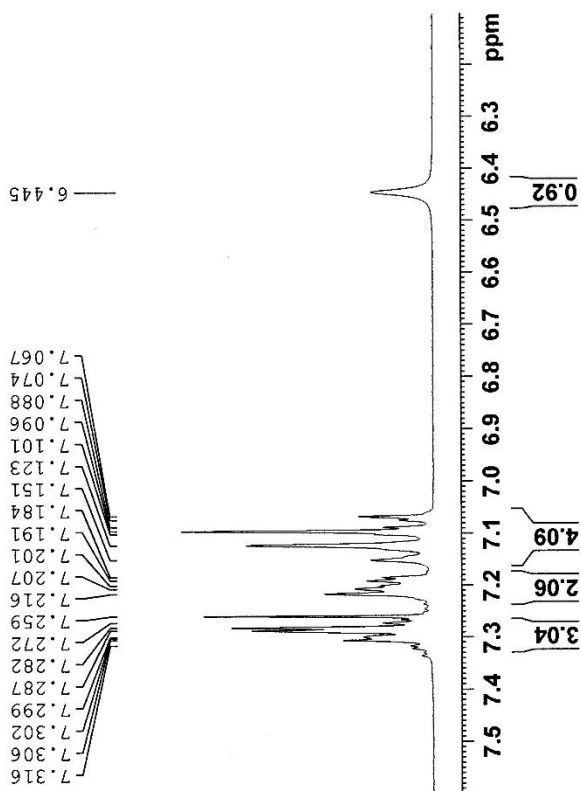
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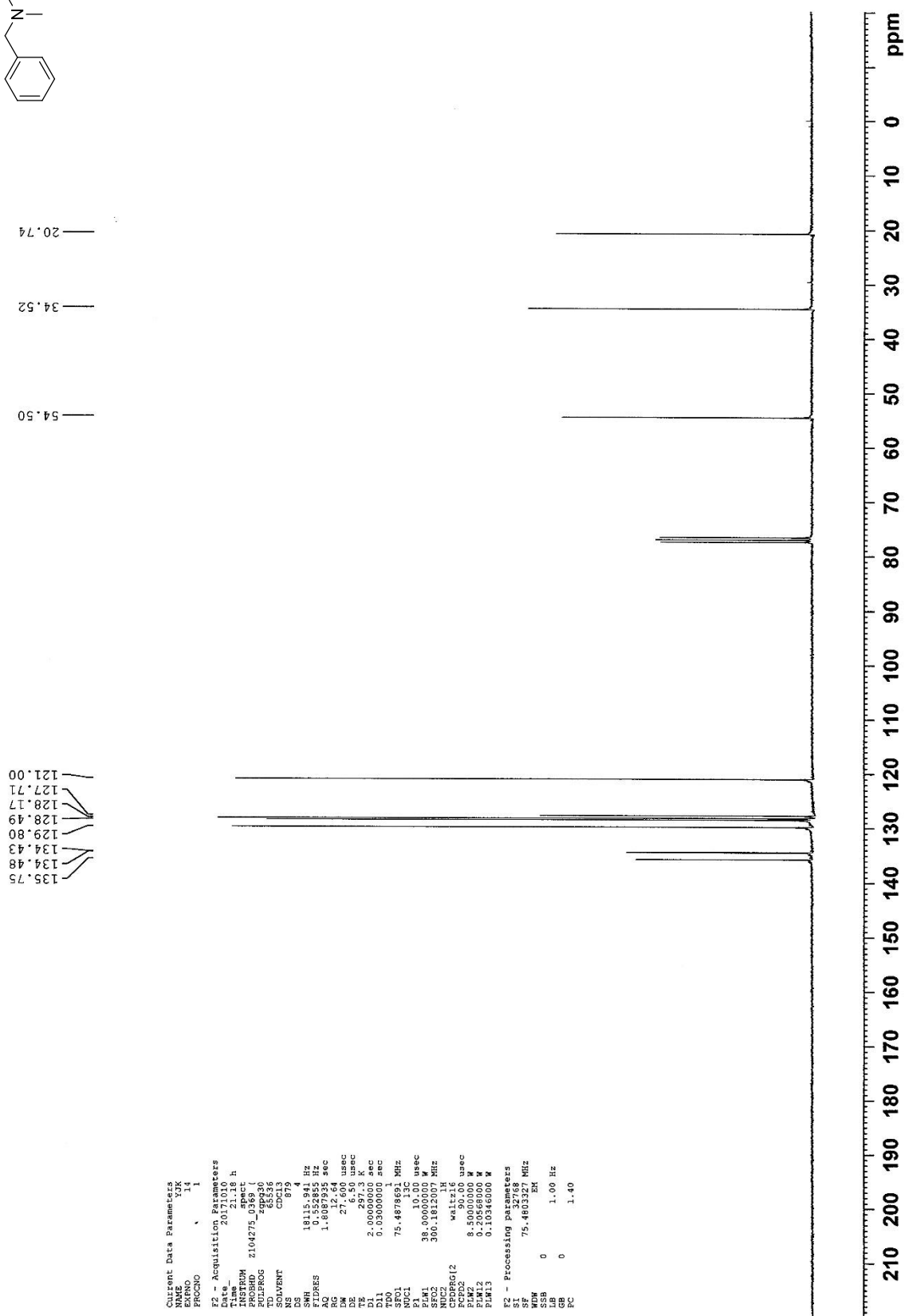
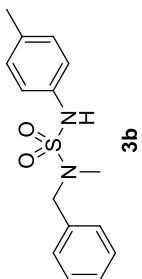
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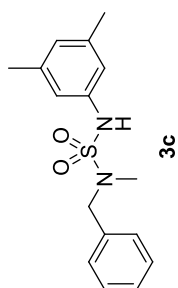
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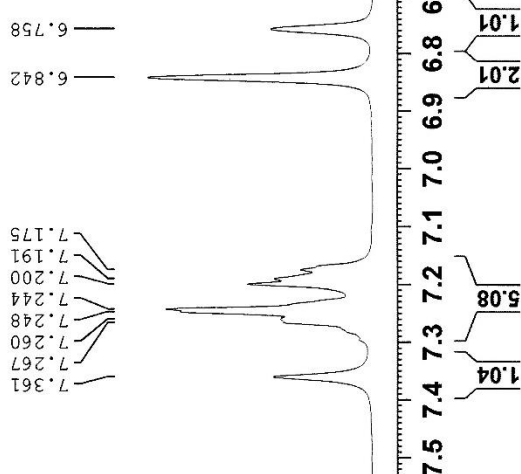
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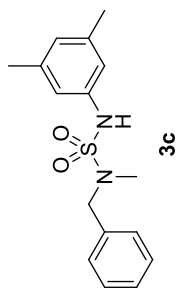
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6.842
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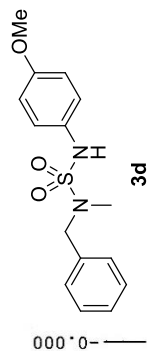
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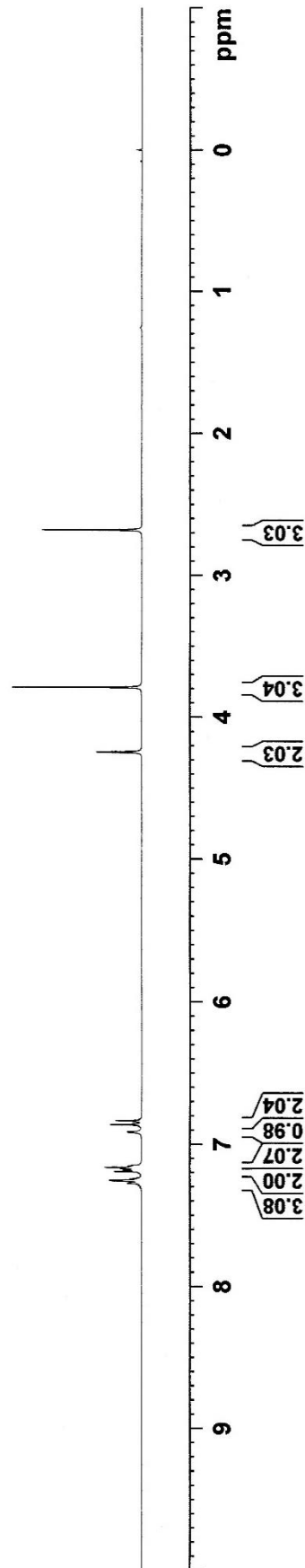
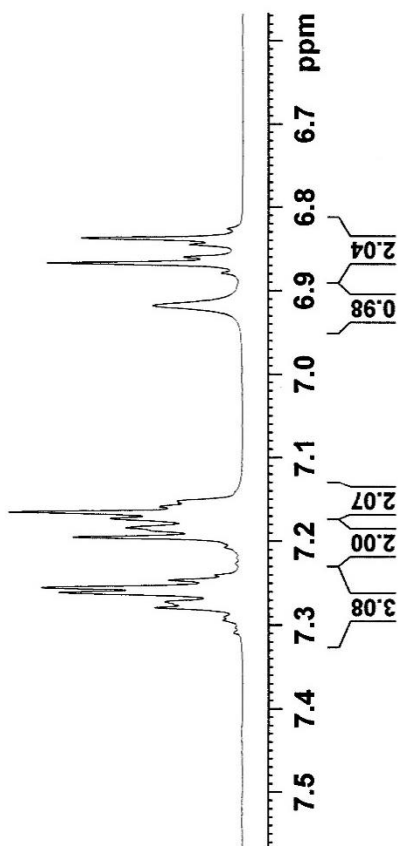
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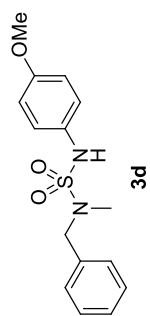
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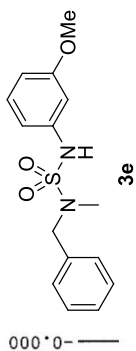
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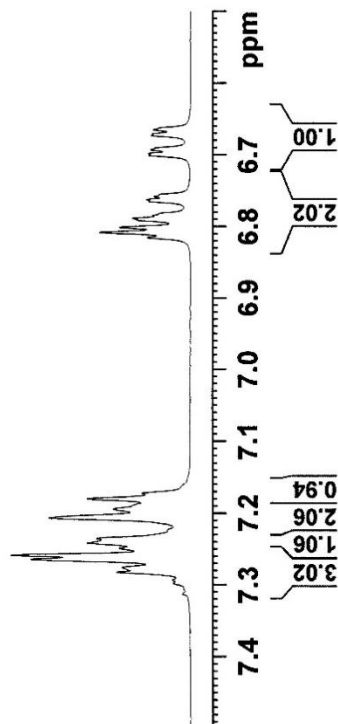
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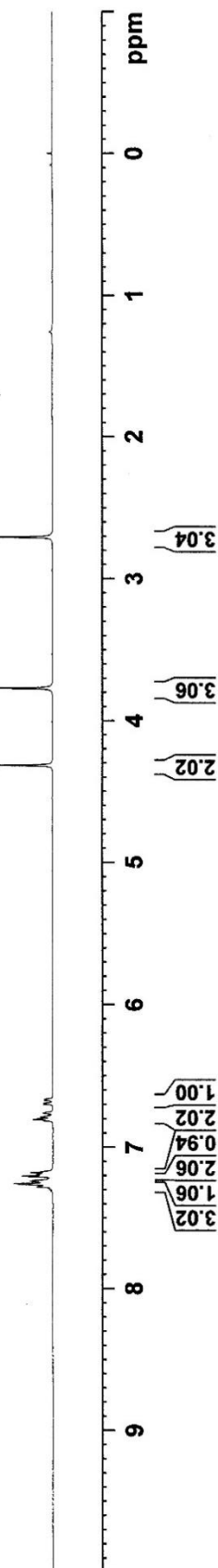
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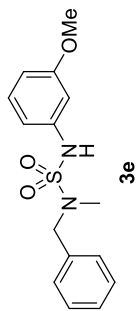
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TE 296.5 K
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6.691
6.673
6.671
6.665



7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 ppm





```

Current Data Parameters
NAME      3e
EXPNO     191
PROCNO    1

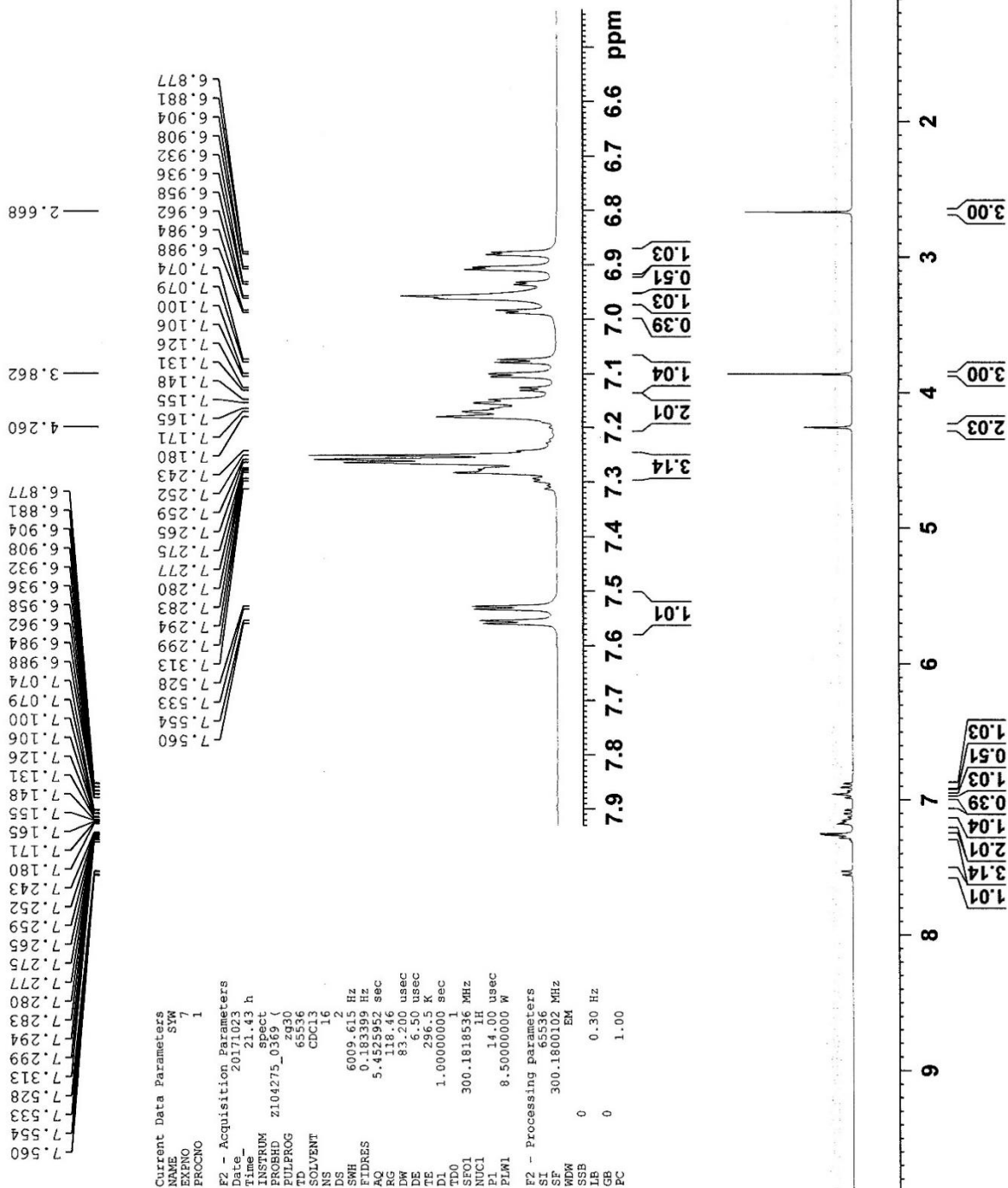
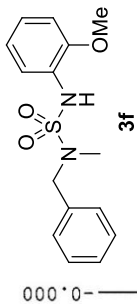
F2 - Acquisition Parameters
Date_     20170927
Time      20.54
INSTRUM   spect
PROBHD    5 mm PABBO BBO
PULPROG   zgpg30
TD         65536
FIDRES    0.275098 Hz
AQ         1.8175317 sec
RG         27.733 usec
DE         6.50 usec
TE         296.5 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

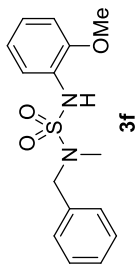
===== CHANNEL f1 =====
SFO1      125.762949 MHz
NUC1      13C
P1         9.00 usec
PLW1      54.95399857 W

===== CHANNEL f2 =====
SFO2      300.1312005 MHz
NUC2      1H
P2         12.00 usec
PLW2      14.9969974 W
PLW12     0.21596000 W
PLW13     0.17493001 W

F2 - Processing parameters
SI         32768
SF         75.4677571 MHz
RG         0
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```







148.77 —
135.81 —
128.53 —
128.17 —
127.74 —
126.61 —
124.57 —
121.10 —
120.04 —
110.47 —
54.55 —
54.67 —
34.45 —

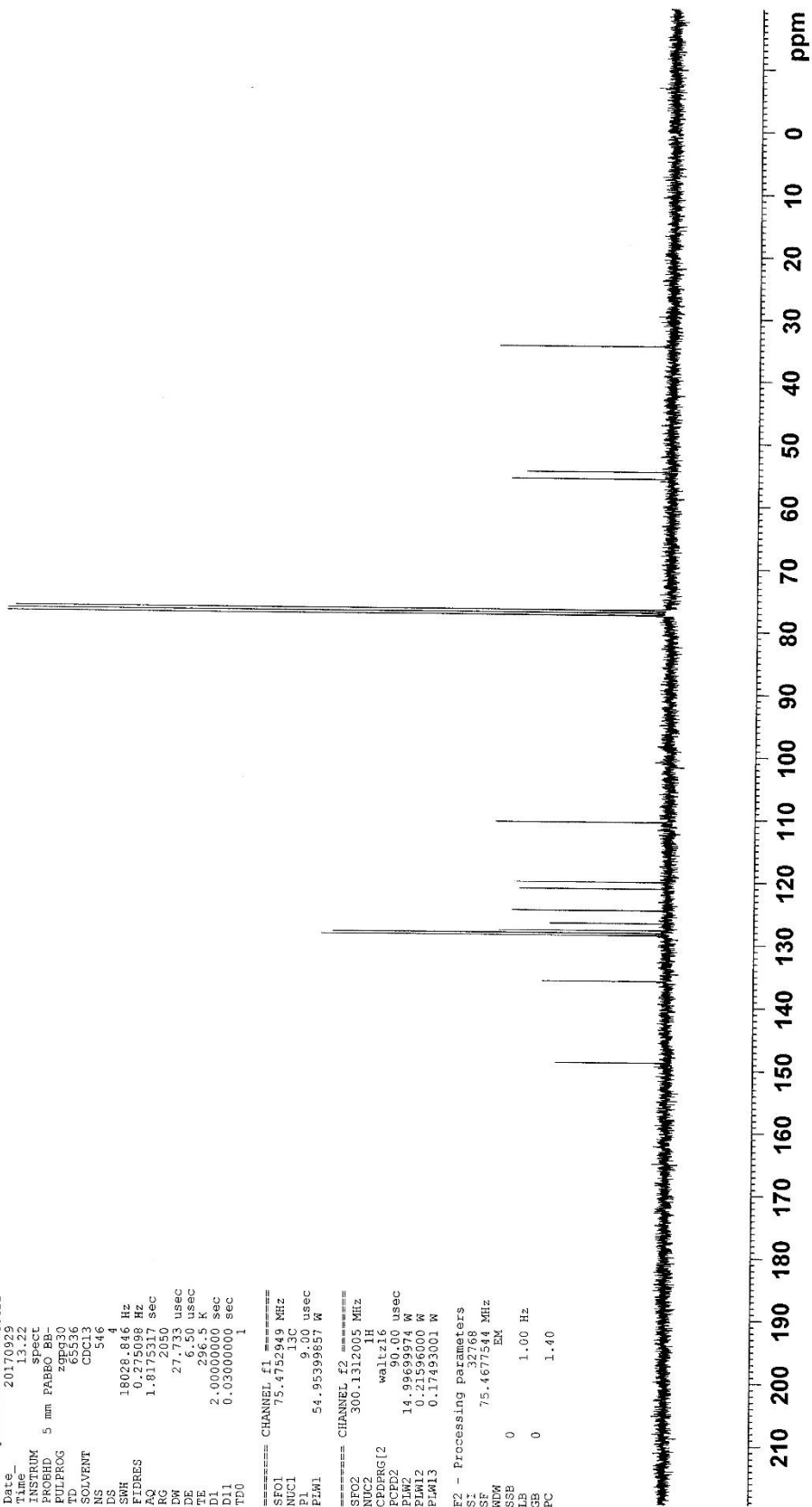
Current Data Parameters
NAME: 3f
EXPNO: 2
PROCNO: 1

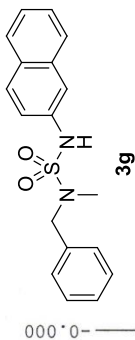
F2 - Acquisition Parameters
Date_: 20170929
Time_: 13.22
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 546
DS: 4
SWH: 18028.846 Hz
FIDRES: 0.275098 Hz
AQ: 1.8175317 sec
RG: 2050
IN: 27.733 usec
DE: 25.00 usec
TE: 296.2 K
D1: 2.00000000 sec
D11: 0.03000000 sec
TD0: 1

===== CHANNEL f1 =====
SFO1: 75.4752949 MHz
NUC1: 13C
P1: 9.00 usec
PL1: 54.9539657 W

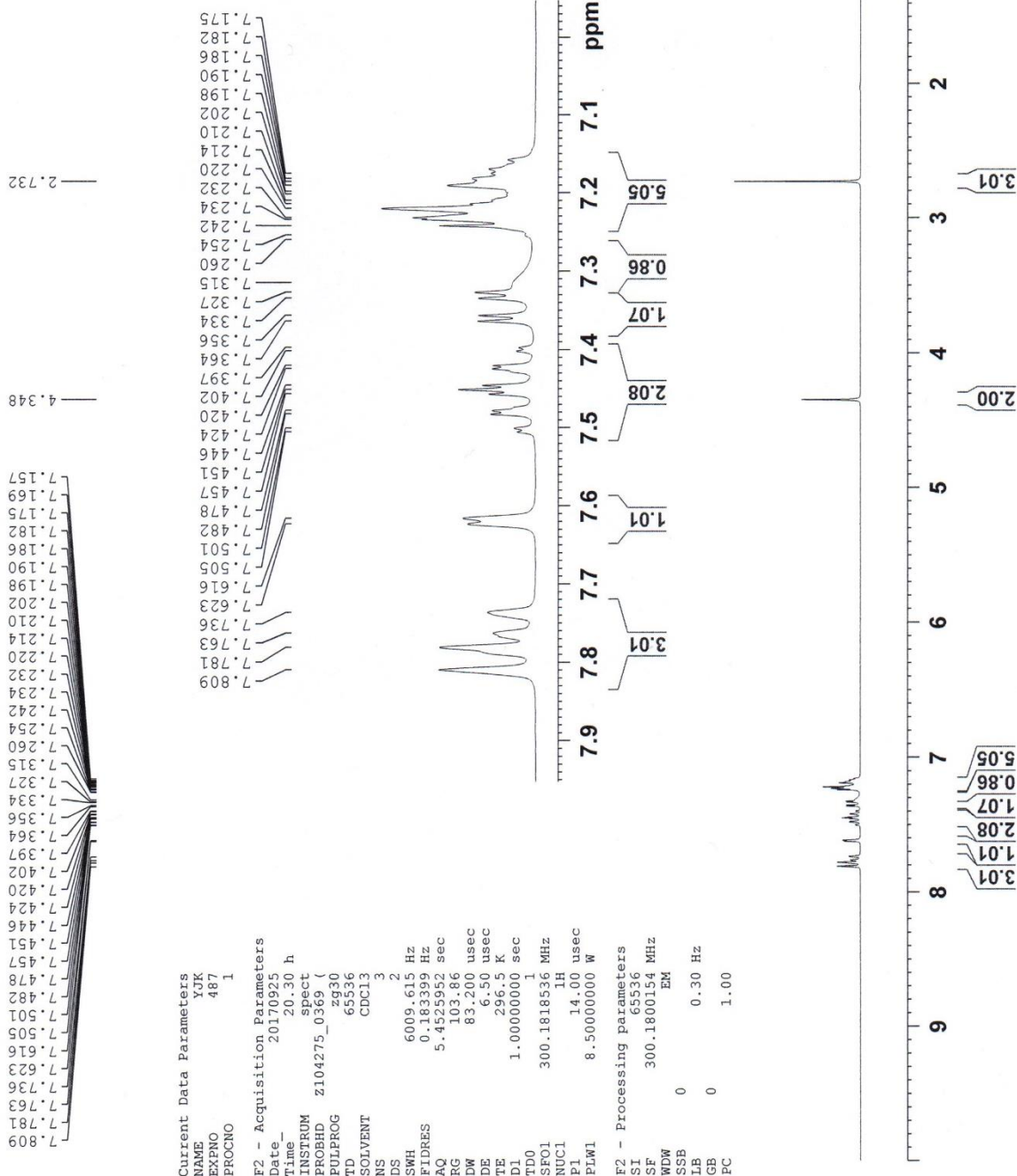
===== CHANNEL f2 =====
SFO2: 300.1312005 MHz
NUC2: 1H
P2: 90.00 usec
PL2: 14.9989974 W
PLW2: 0.21396000 W
PLM13: 0.17455001 W

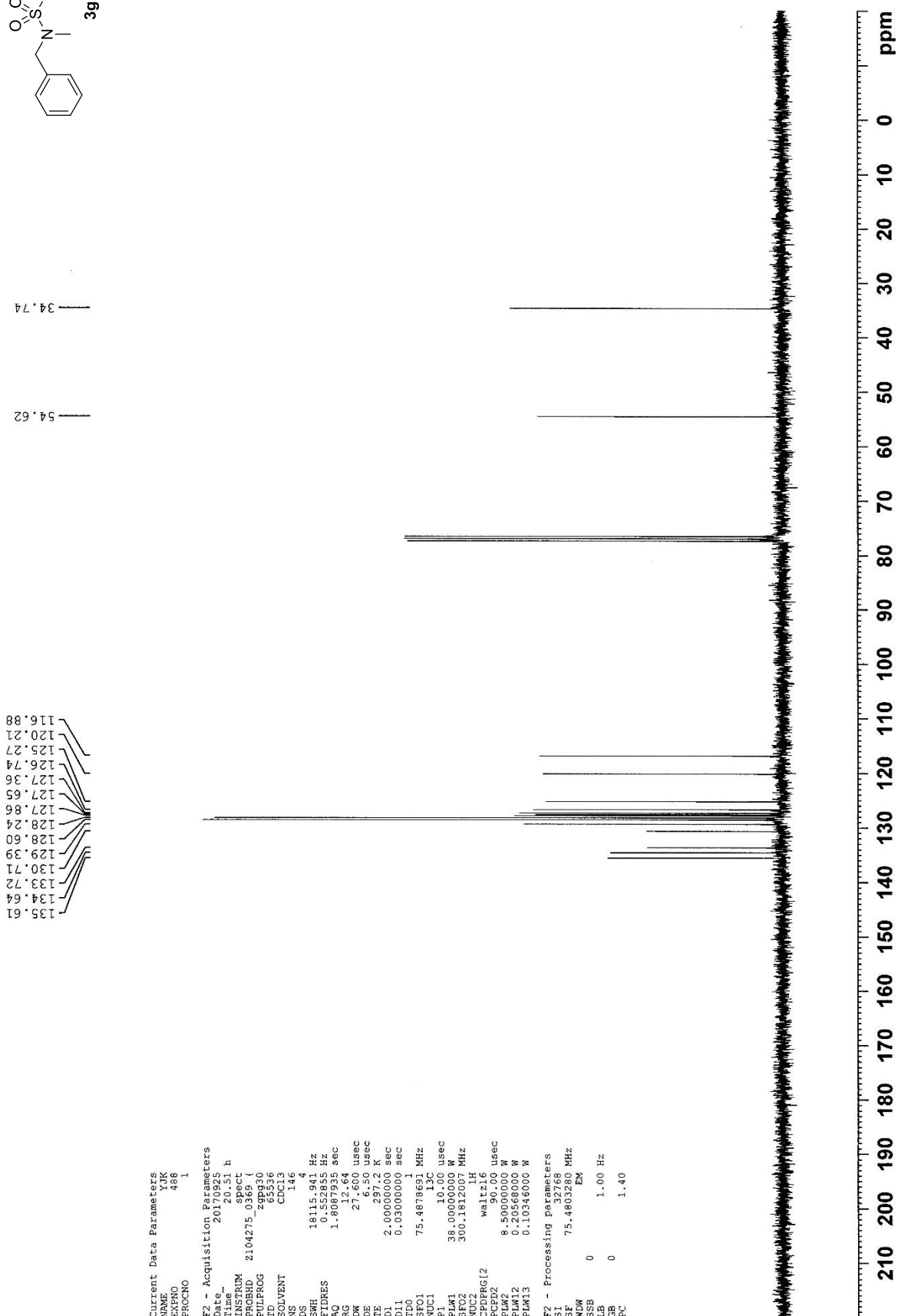
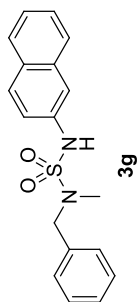
F2 - Processing parameters
SI: 32768
SF: 75.4677544 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

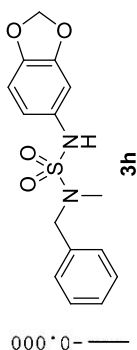




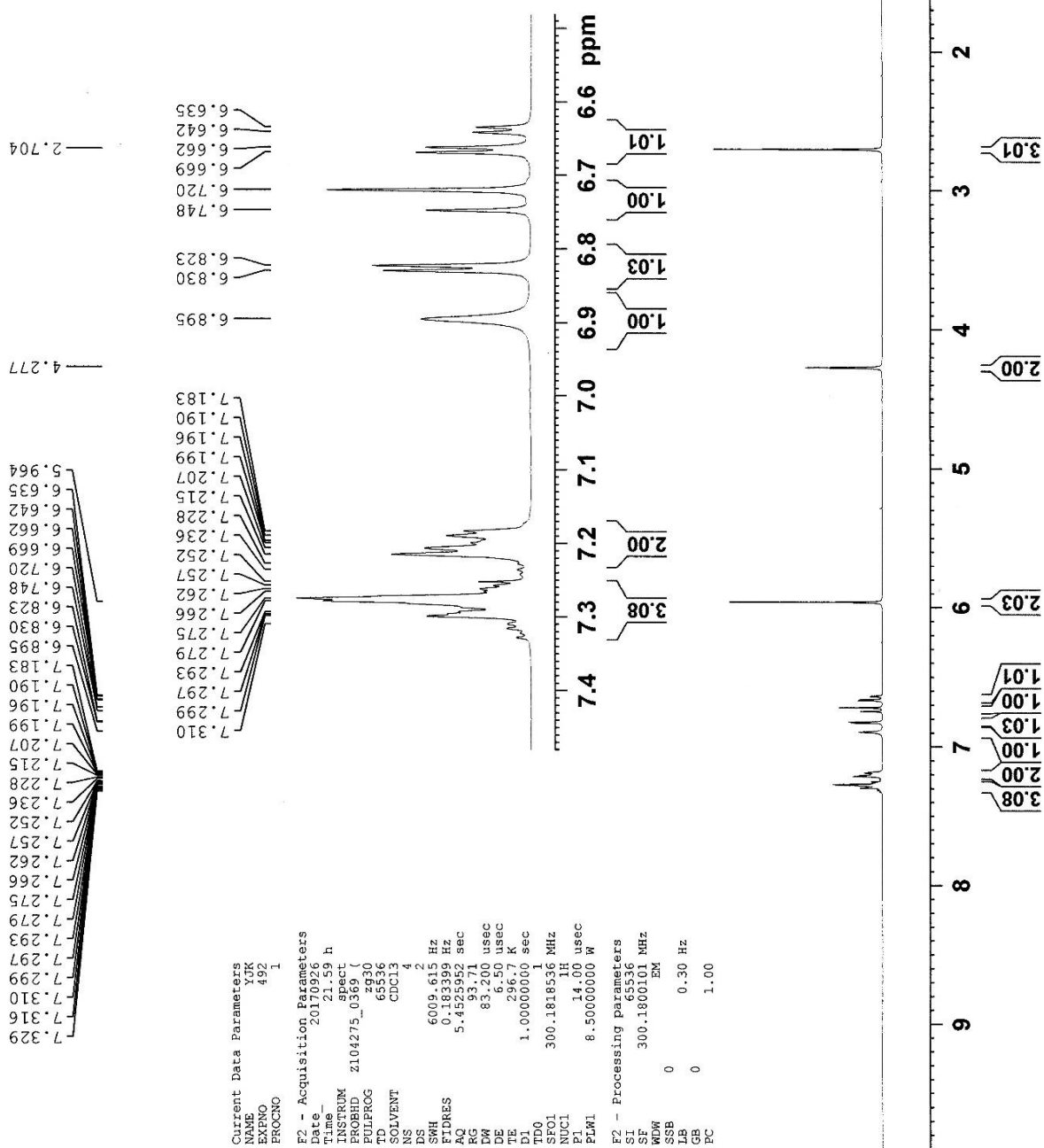
0.000

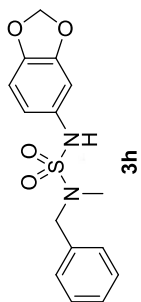






— -0.000

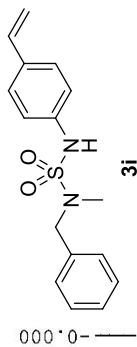




148.11
145.40
135.75
130.85
128.57
128.21
127.81
115.66
108.25
104.53
101.46
54.60
34.69

Current Data Parameters
NAME YJK
EXPNO 493
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170926
Time 22.50 h
INSTRUM spect
PROBHD 2104275_0369 (239956
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 595
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 1.8087935 sec
RG 15.81
DW 27.600 usec
DE 6.50 usec
TE 297.4 K
D1 2.0000000 sec
d11 0.0300000 sec
SFO1 75.4878691 MHz
NUC1 13C
P1 10.00 usec
PLW1 38.0000000 W
SFO2 300.1812007 MHz
NUC2 1H
CDEPRG[2] waltz16
PCPD2 90.00 usec
PLW2 8.5000000 W
PLW12 0.2036800 W
PLW13 0.1034600 W
F2 - Processing parameters
SI 32768
SF 75.4803282 MHz
MDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



— 0.000

— 2.705

— 4.310

7.376
7.347
7.301
7.297
7.287
7.281
7.270
7.264
7.255
7.246
7.195
7.184
7.176
7.168
7.145
7.139
7.102
6.726
6.690
6.667
6.631
5.728
5.726
5.669
5.667
5.297
5.287
5.246
5.244
5.209
5.207

7.376
7.347
7.301
7.297
7.287
7.281
7.270
7.264
7.255
7.246
7.195
7.184
7.176
7.168
7.145
7.139
7.102

Current Data Parameters
NAME SYM
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171024
Time_ 11:24 h
INSTRUM spect
PROBHD 2104275_0369 (2930
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 5.4525952 sec
RG 93.71
LW 83.200 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
D11 1
SFO1 300.181536 MHz
NUC1 1H
FI 14.00 usec
FLW1 8.50000000 W

F2 - Processing parameters
SI 65536
SF 300.180018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 ppm

2.03
2.02
1.09
4.05
0.98

ppm

0

1

2

3

4

5

6

7

8

9

3.05

2.04

1.00

1.00

1.00

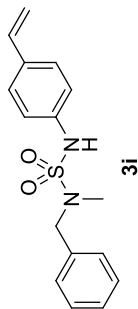
0.98

4.05

1.09

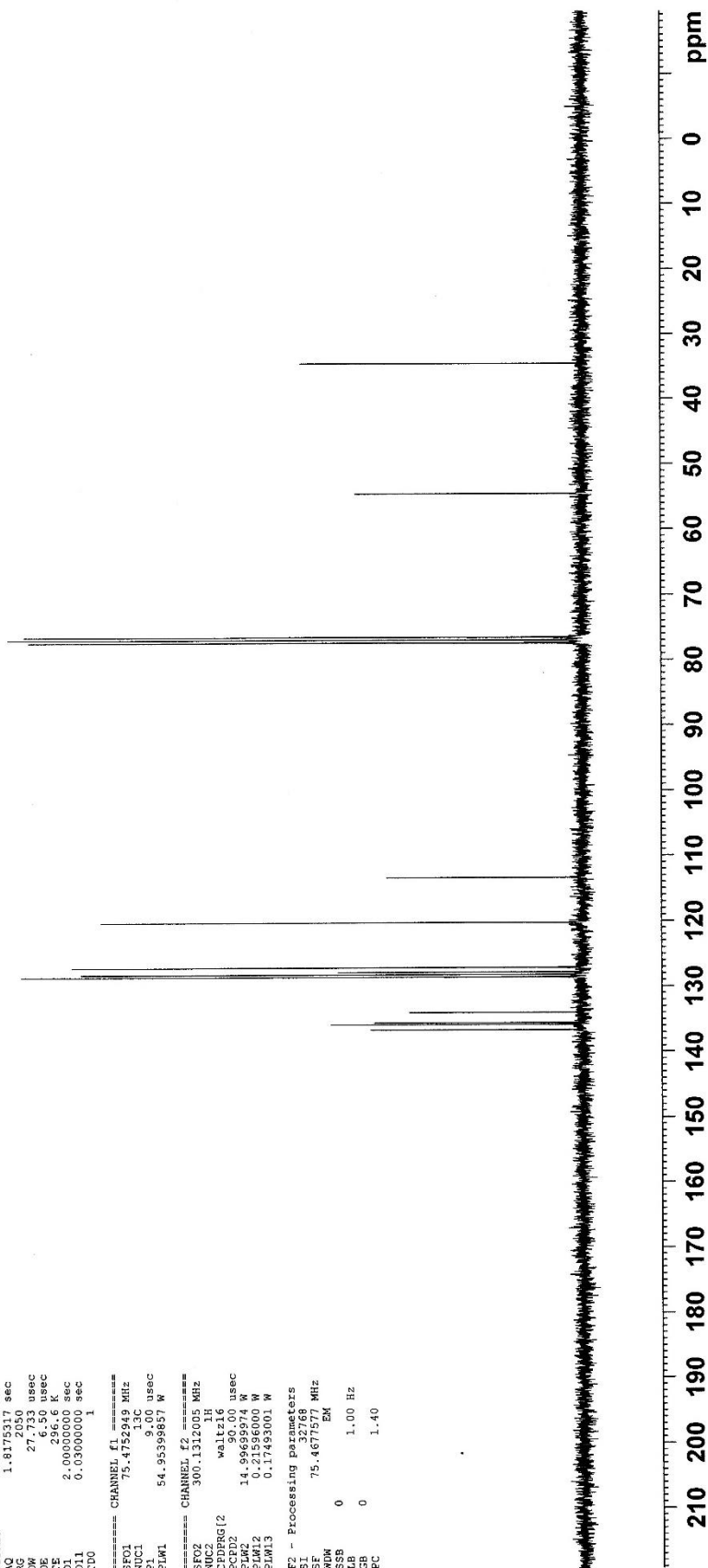
2.02

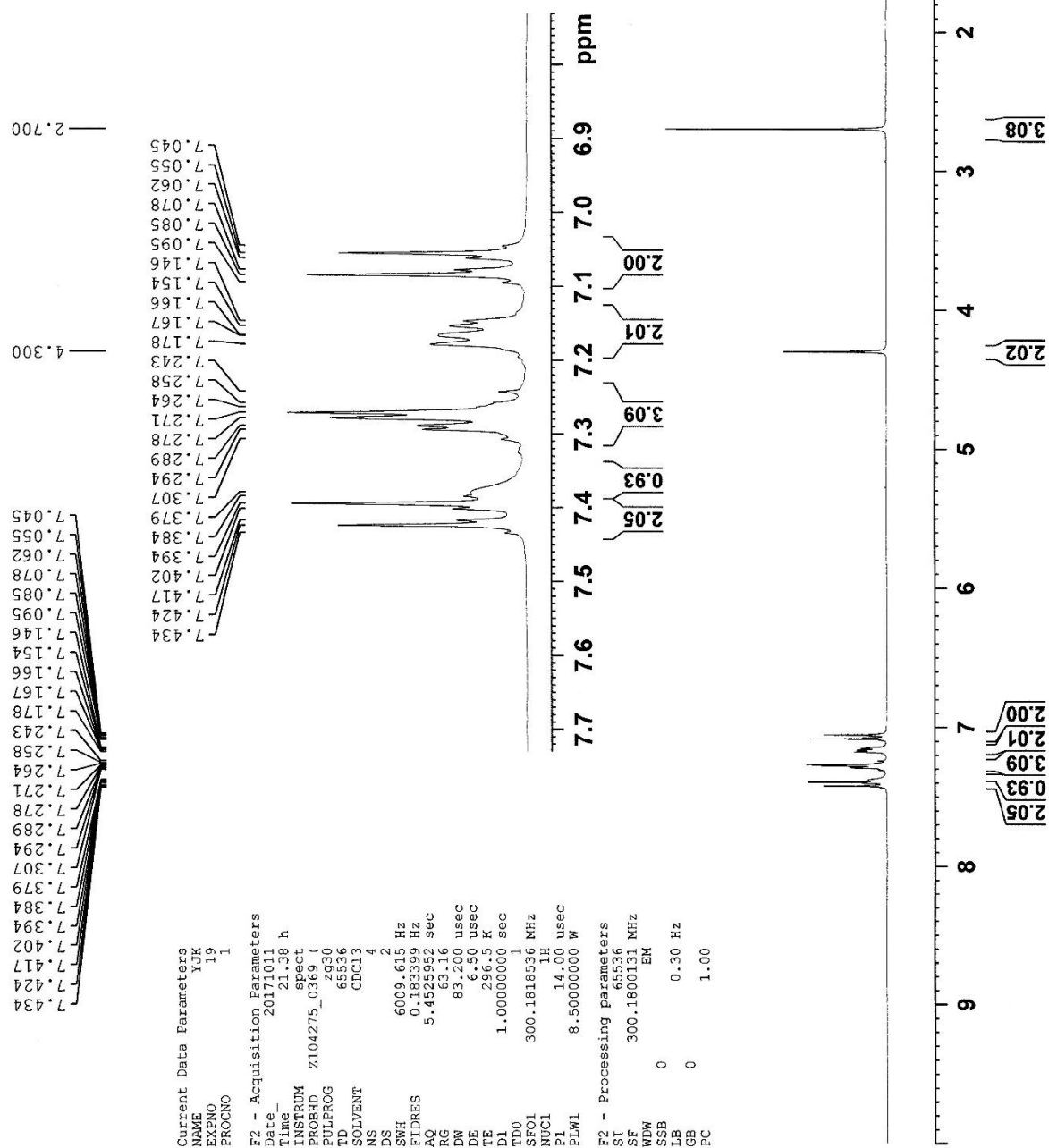
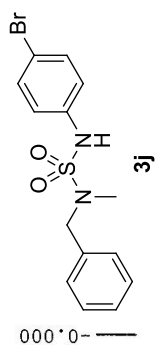
2.03

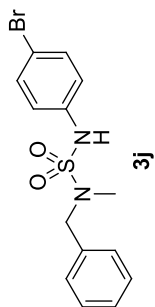


136.62
135.84
135.57
134.00
128.57
128.20
127.82
127.12
120.23
113.36
54.53
34.56

Current Data Parameters
NAME SWH
EXPNO 1.0
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170927
Time 20.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 504
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 655
DE 27.733 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
D10 1
===== CHANNEL f1 =====
SFO1 75.4752945 MHz
NUC1 13C
JC 9.00 usec
PL1 54.95398857 W
===== CHANNEL f2 =====
SFO2 300.1312005 MHz
NUC2 1H
JC 0.00 usec
PL2 0.00 usec
PCPD2 90.00 usec
PLW2 14.9869974 W
PLW12 0.21586000 W
PLW13 0.17455001 W
F2 - Processing parameters
SI 32768
SF 75.4671577 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







34.61

54.50

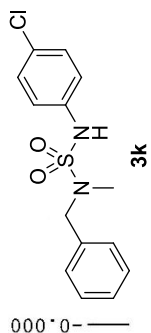
117.45
121.70
127.95
128.18
128.65
132.30
135.32
136.27

Current Data Parameters
NAME YJK
EXPNO 25
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171012
Time_ 20.38 h
INSTRUM spect
PROBHD Z104275_0369 (zgpg30)
PULPROG zgpg30
TD 65536
FIDRES 0.000000
SOLVENT CDCl3
NS 474
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 1.8087935 sec
RG 12.64
DW 27.600 usec
DE 6.50 usec
TE 297.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
SFO1 75.4878691 MHz
NUC1 13C
P1 10.00 usec
PL1 38.0000000 W
SFO2 300.1812007 MHz
NUC2 1H
PCPDPRG12 waltz16
PCPD2 90.00 usec
PLW2 8.5000000 W
PLW12 0.2056800 W
PLW13 0.1034600 W

F2 - Processing parameters
SI 32768
SF 75.4803300 MHz
WDW EM
SSB 0
GB 0
PC 1.40

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



— 0.000

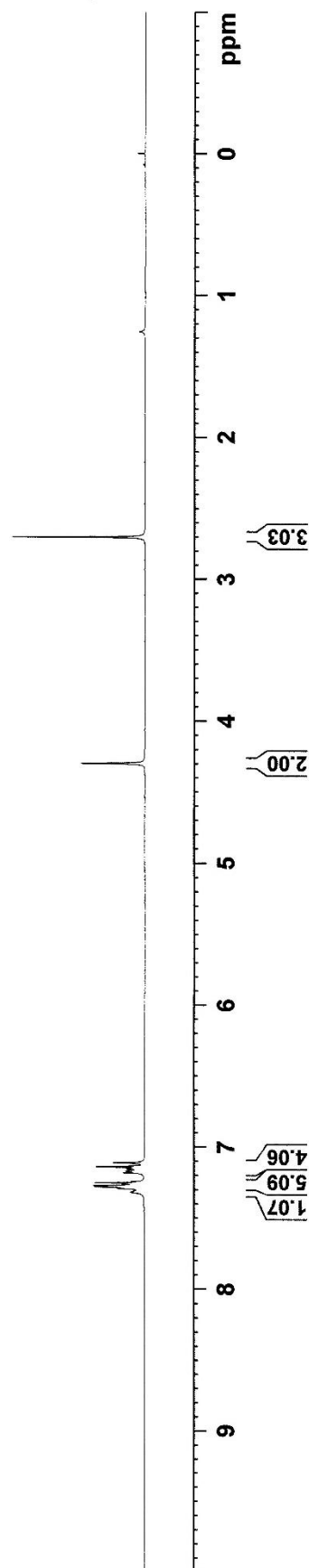
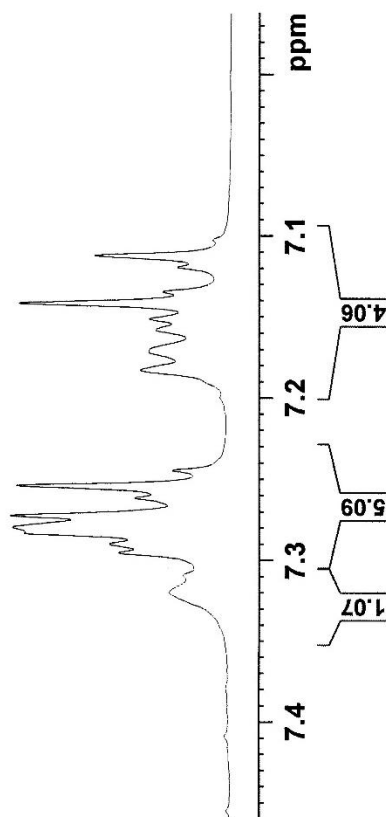
— 2.703

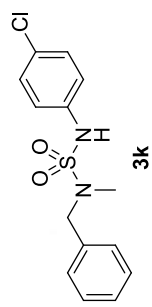
— 4.300

7.320
7.310
7.296
7.284
7.280
7.273
7.262
7.255
7.245
7.183
7.172
7.158
7.152
7.142
7.135
7.120
7.113

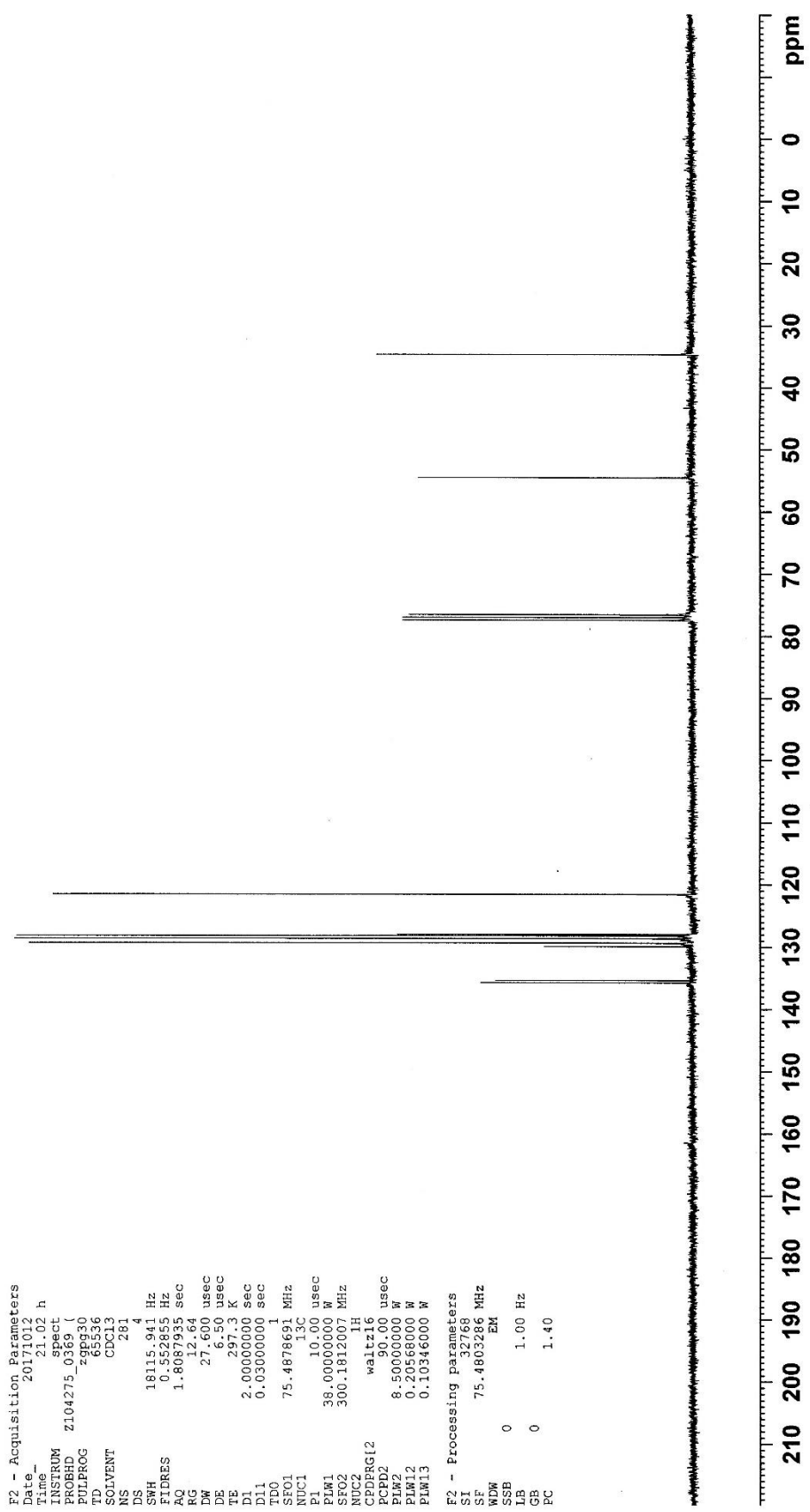
Current Data Parameters
NAME YK
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171011
Time_ 21.45 h
INSTRUM spect
PROBHD zg30
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 5.4525952 sec
RG 74.68
DW 83.200 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
SFO1 300.1818536 MHz
NUC1 1H
P1 14.00 usec
PL1 8.50000000 W
F2 - Processing parameters
SI 65536
SF 300.1800122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

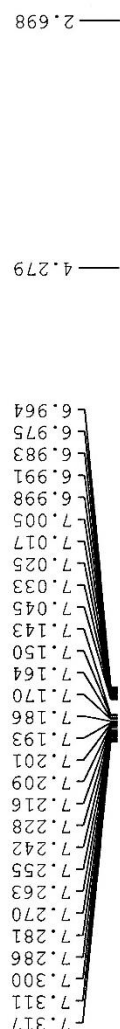
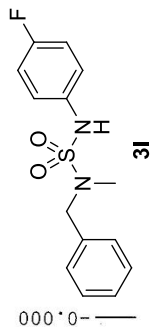
7.320
7.310
7.296
7.284
7.280
7.273
7.262
7.255
7.245
7.183
7.172
7.158
7.152
7.142
7.135
7.120
7.113





Current Data Parameters
 NAME YIK
 EXPNO 26
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20171012
 Time 21.02 h
 INSTRUM spect
 PROBD Z104275_0369
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 281
 DS 4
 SWH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 1.8087935 sec
 RG 12.64
 DW 27.600 usec
 DE 6.50 usec
 TE 297.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1
 SFO1 75.4878691 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 38.00000000 W
 SFO2 300.1812007 MHz
 NUC2 1H
 CPMRG12 waltz16
 PCPD2 90.00 usec
 PLW2 8.50000000 W
 PLW12 0.20568000 W
 PLW13 0.10346000 W
 F2 - Processing parameters
 SI 32768
 SF 75.4803286 MHz
 WDM EM
 SSB 0
 GB 0
 PC 1.40

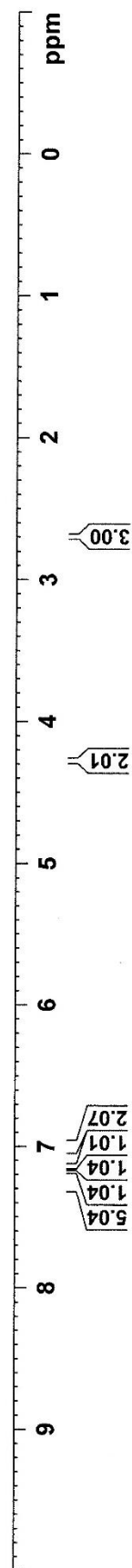
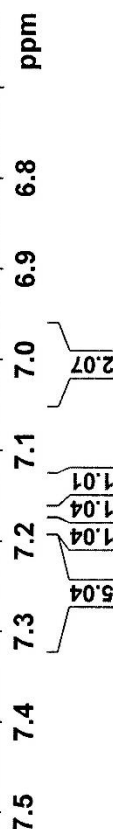
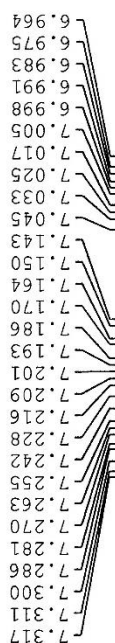


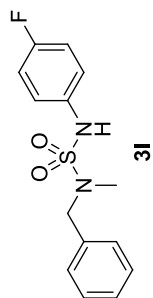


Current Data Parameters
 NAME SYW
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171023
 Time 20.20 h
 INSTRUM spect
 PROBHD z104275_0369 (zg30)
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6009.615 Hz
 FIDRES 0.183399 Hz
 AQ 5.4525952 sec
 RG 63.16
 DW 83.200 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.00000000 sec
 TD0 1
 SFO1 300.1818536 MHz
 NUC1 1H
 P1 14.00 usec
 PLW1 8.5000000 W

F2 - Processing parameters
 SI 65536
 SF 300.1800132 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





34.63

54.52

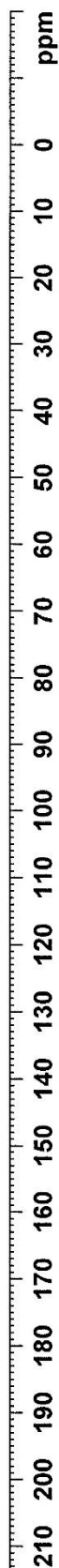
115.91
116.21
123.00
123.11
127.90
128.17
128.61
133.01
135.52

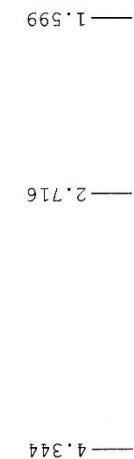
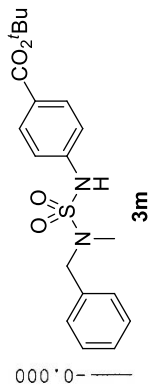
158.45
161.69

Current Data Parameters
NAME SYW
EXNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171023
Time_ 21.22 h
INSTRUM spect
PROBHD Z104275 0369 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 734
DS 4
SWH 18115.941 Hz
FIDRES 0.1552855 Hz
AQ 1.6081935 sec
RG 12.64
DW 27.600 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SF01 75.4878691 MHz
NUC1 13C
PCPD1 13C usec
PL1 38.00000000 W
SF02 300.1812007 MHz
NUC2 1H
PCPD2(2) waltz16
PCPD2 90.00 usec
PLW2 8.50000000 W
PLW12 0.20568000 W
PLW13 0.10346000 W

F2 - Processing parameters
SI 32768
SF 75.4803268 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

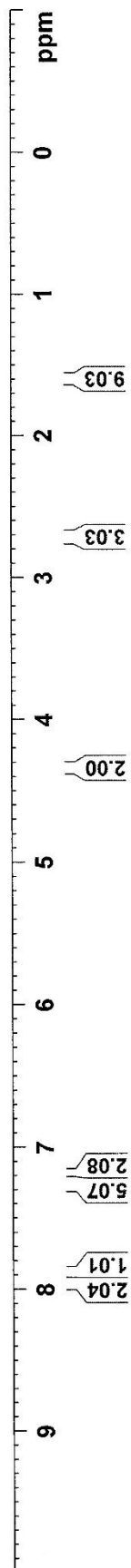


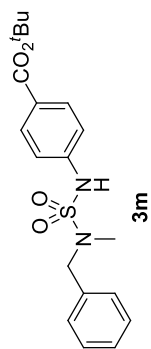


Current Data Parameters
 NAME YJK
 EXPNO 464
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20170922
 Time 21.09 h
 INSTRUM spect
 PROBHD z104275_0369 (2930
 PULPROG zgpg30
 F1 65536
 SOLVENT CDCl3
 NS 2
 DS 2
 SWH 6009.615 Hz
 FIDRES 0.183399 Hz
 AQ 5.4525952 sec
 RG 32.2
 DW 83.200 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.00000000 sec
 TDO 300.1818536 MHz
 SFOL 300.1800089 MHz
 NUCL 1H
 PL1 14.00 usec
 PLW1 8.5000000 W
 F2 - Processing parameters
 SI 65536
 SF 300.1800089 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 FC 1.00



8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm

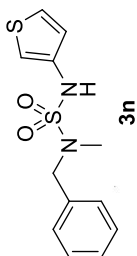




Current Data Parameters
 NAME 1
 EXPNO 485
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170922
 Time_ 21.36 h
 INSTRUM spect
 PROBHD z104275_0369 (zpgp30)
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 261
 DS 4
 SWH 18115.941 Hz
 FIDRES 0.352935 Hz
 AQ 1.800932 sec
 RG 14.34
 DE 27.600 usec
 TE 297.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1
 SFO1 75.4878691 MHz
 NUC1 13C
 P1 10.00 usec
 PL1 38.00000000 W
 SFO2 300.1612007 MHz
 NUC2 1H
 P2 10.00 usec
 PL2 8.50000000 W
 SFO3 0.20568000 W
 PL3 0.10346000 W

F2 - Processing parameters
 SI 32768
 SF 75.4803317 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



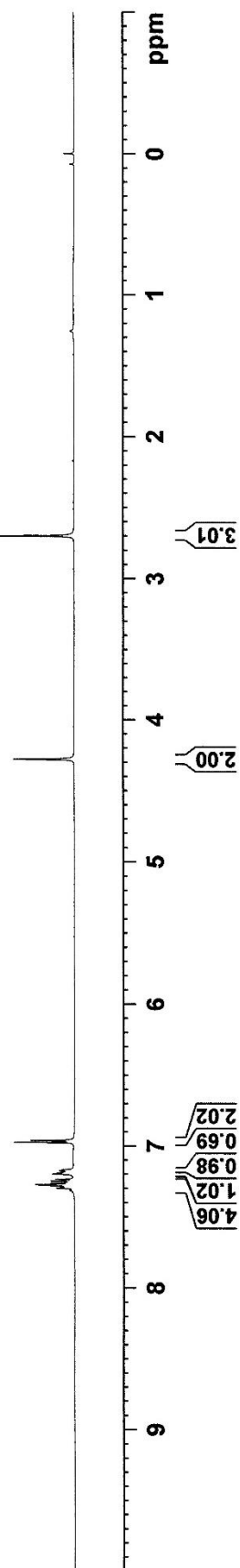
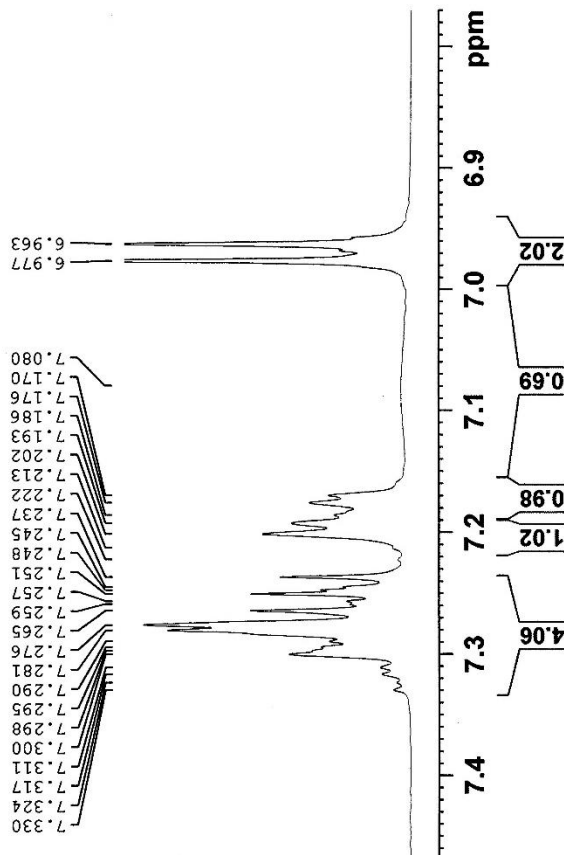
— 0.000

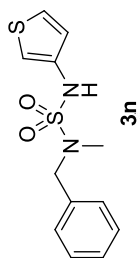
— 2.702

— 4.281

7.330
7.332
7.317
7.311
7.300
7.298
7.295
7.290
7.281
7.276
7.265
7.259
7.257
7.251
7.248
7.245
7.237
7.232
7.222
7.213
7.202
7.193
7.186
7.176
7.170
7.163
7.156
7.149
7.142
7.135
7.128
7.121
7.114
7.107
7.100
7.093
7.086
7.079
7.072
7.065
7.058
7.051
7.044
7.037
7.030
7.023
7.016
7.009
7.002
6.995
6.988
6.981
6.974
6.967
6.960
6.953

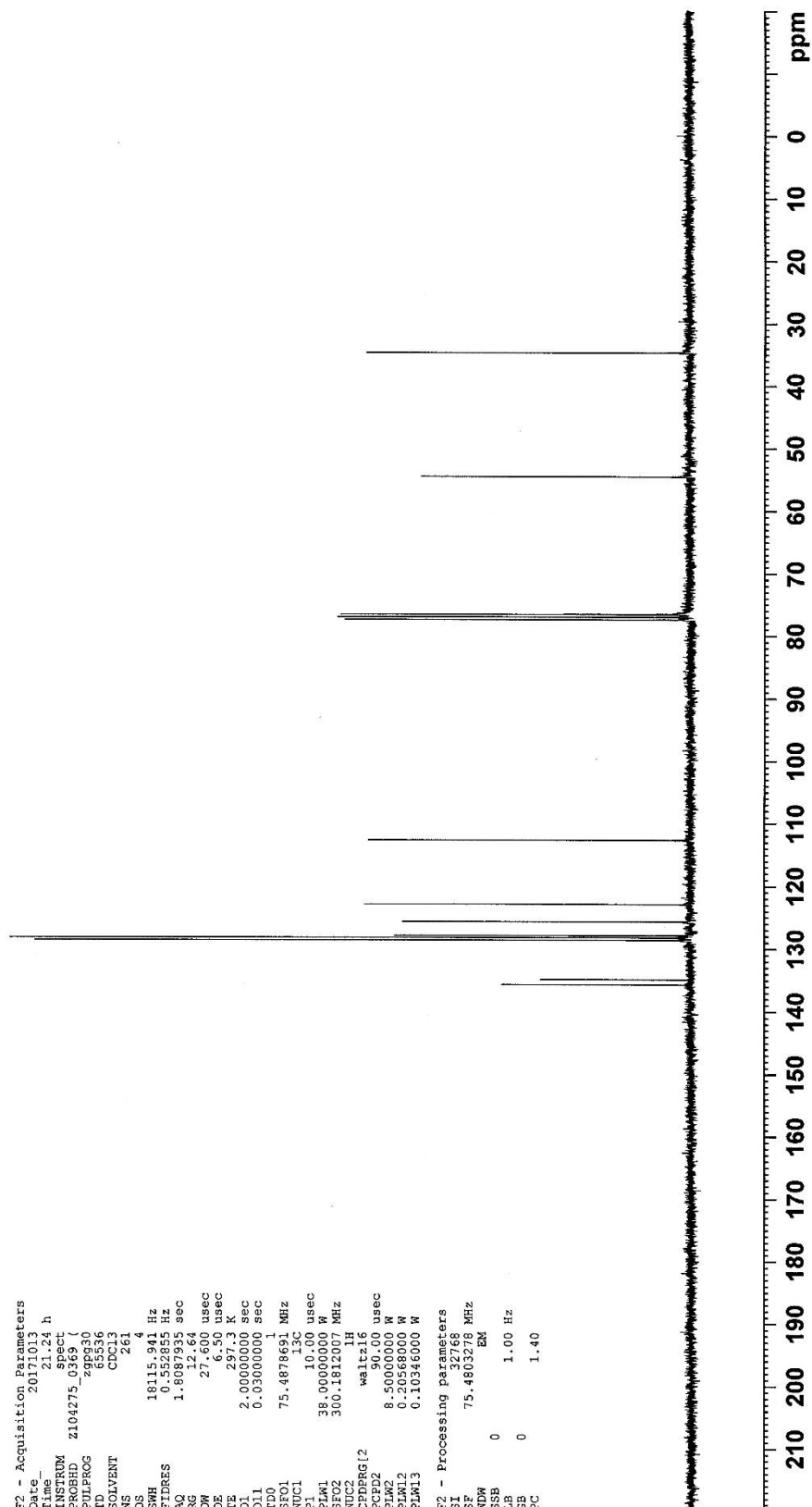
Current Data Parameters
NAME YUK
EXPNO 33
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171013
Time_ 20.53 h
INSTRUM spect
PROBHD Z104275_0369 (zg30)
PULPROG 65536
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 5.470392 sec
RG 126.500
DW 83.200 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
SFO1 300.1818536 MHz
NUC1 1H
P1 14.00 usec
PL1 8.50000000 W
F2 - Processing parameters
SI 65536
SF 300.180015 MHz
RG 126.500
EX 0
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

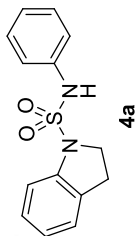




135.69
134.87
128.61
128.19
127.85
125.65
122.88
112.67
54.57
34.70

Current Data Parameters
NAME YJK
EXPNO 34
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171013
Time 21.24 h
INSTRUM spect
PROBHD z104275_0369
PULPROG zgpg30
TD 65536
FIDRES 0.552855 Hz
AQ 1.8087935 sec
RG 12.64
DW 27.600 usec
DE 6.50 usec
TE 297.3 K
D1 2.0000000 sec
D11 0.03000001 sec
TD0 1
SFO1 75.4878691 MHz
NUC1 13C
P1 10.00 usec
PLW1 38.00000000 W
SFO2 300.1812007 MHz
NUC2 1H
PCPG12 waltz16
PCPD2 50.00 usec
PLW2 8.50000000 W
PLW3 0.20568000 W
PLW13 0.10346000 W
F2 - Processing parameters
SI 32768
SF 75.4803278 MHz
WDW EM
SSB 0
GB 1.00 Hz
PC 1.40





—0.000

2.880
2.851
2.823

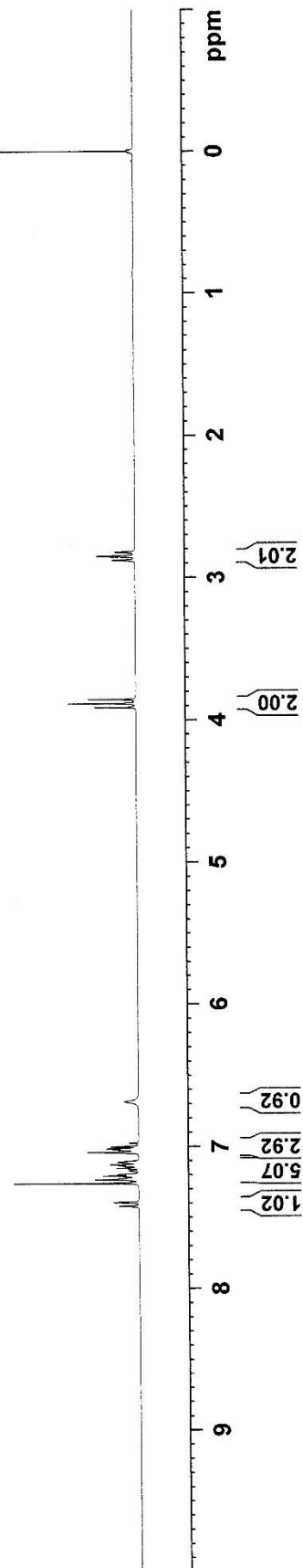
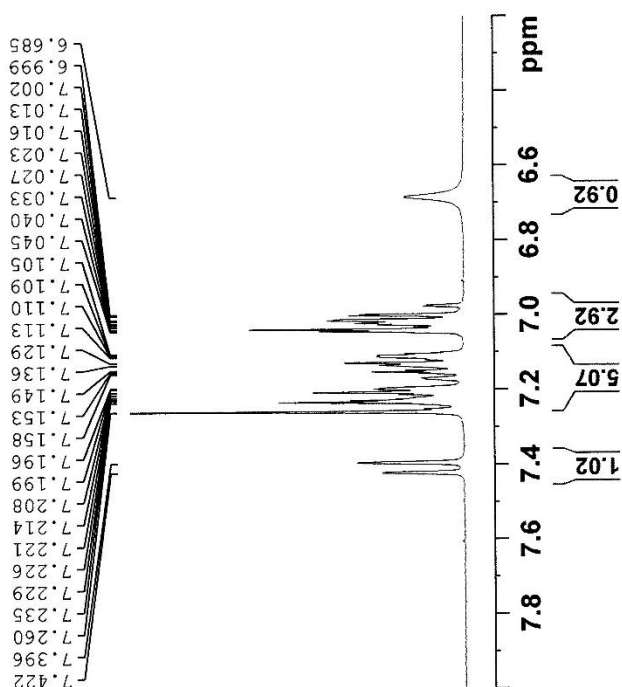
3.914
3.886
3.857

7.422
7.396
7.260
7.253
7.225
7.229
7.226
7.221
7.221
7.199
7.208
7.174
7.172
7.170
7.158
7.153
7.149
7.136
7.129
7.121
7.113
7.110
7.109
7.105
7.045
7.040
7.033
7.027
7.023
7.016
7.013
7.002
6.999
6.977
6.974
6.985

Current Data Parameters
NAME iJK
EXPNO 383
PROCNO 1

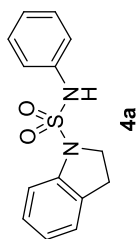
F2 - Acquisition Parameters
Date_ 20170720
Time 10.40 h
INSTRUM spect
PROBHD Z104275_0329
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 5.4525952 sec
RG 211.36
DW 83.200 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TDO 1
SFO1 300.1818536 MHz
NUC1 1H
P1 14.00 usec
PLW1 8.5000000 W

F2 - Processing parameters
SI 65536
SF 300.1800077 MHz
WDW EM
SSB 0
LB 0
GB 0
PC 1.00



141.77
136.18
131.39
129.24
127.68
125.45
125.07
123.40
121.87
113.98

51.44
27.89

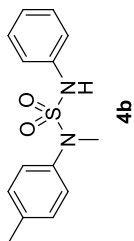


Current Data Parameters
NAME VJK
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171011
Time 20.35 h
INSTRUM spect
PROBHD Z104275_0389 (4
PULPROG zgpg30
TD 65536
F2 - Processing parameters
SI 32768
SF 75.4803283 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DS 4
NS 452
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 1.8087935 sec
RG 12.64
DW 27.600 usec
DE 6.50 usec
TE 297.4 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
SFO1 75.4878691 MHz
NUC1 13C
P1 10.00 usec
PLW1 38.0000000 W
SFO2 300.1812007 MHz
NUC2 1H
PCPD2 waltz16
PCPD2 90.00 usec
PLW2 8.5000000 W
PLW12 0.2056800 W
PLW13 0.1034600 W

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



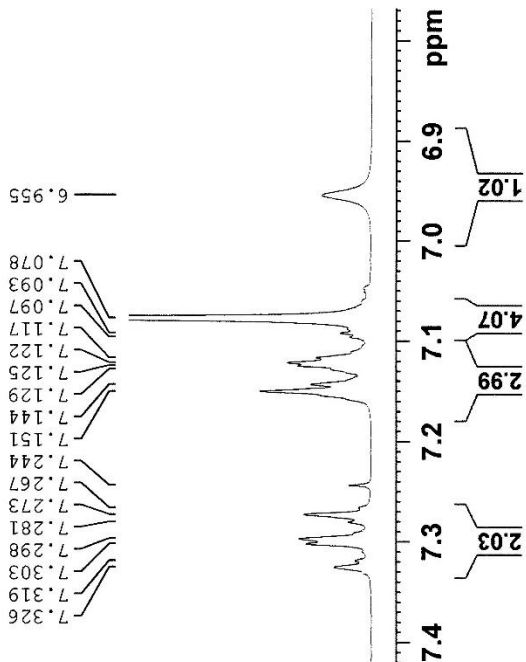
— 0.000

— 2.301

— 3.213

7.326
7.319
7.303
7.298
7.281
7.273
7.267
7.244
7.151
7.144
7.129
7.125
7.122
7.117
7.097
7.093
7.078
6.955

Current Data Parameters
NAME YJK
EXPNO 489
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170925
Time_ 21.10 h
INSTRUM spect
PROBHD Z104275_0369 (1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 4
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 5.4525952 sec
RG 93.71
DW 83.200 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TDO 1
SFO1 300.1818536 MHz
NUC1 1H
P1 14.00 usec
PLW1 8.50000000 W
F2 - Processing parameters
SI 65536
SF 300.1800125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



ppm

3.09

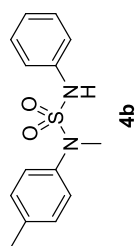
3.00

1.02

4.07

2.99

2.03



139.03
137.55
137.28
129.78
129.24
126.85
124.26
119.64

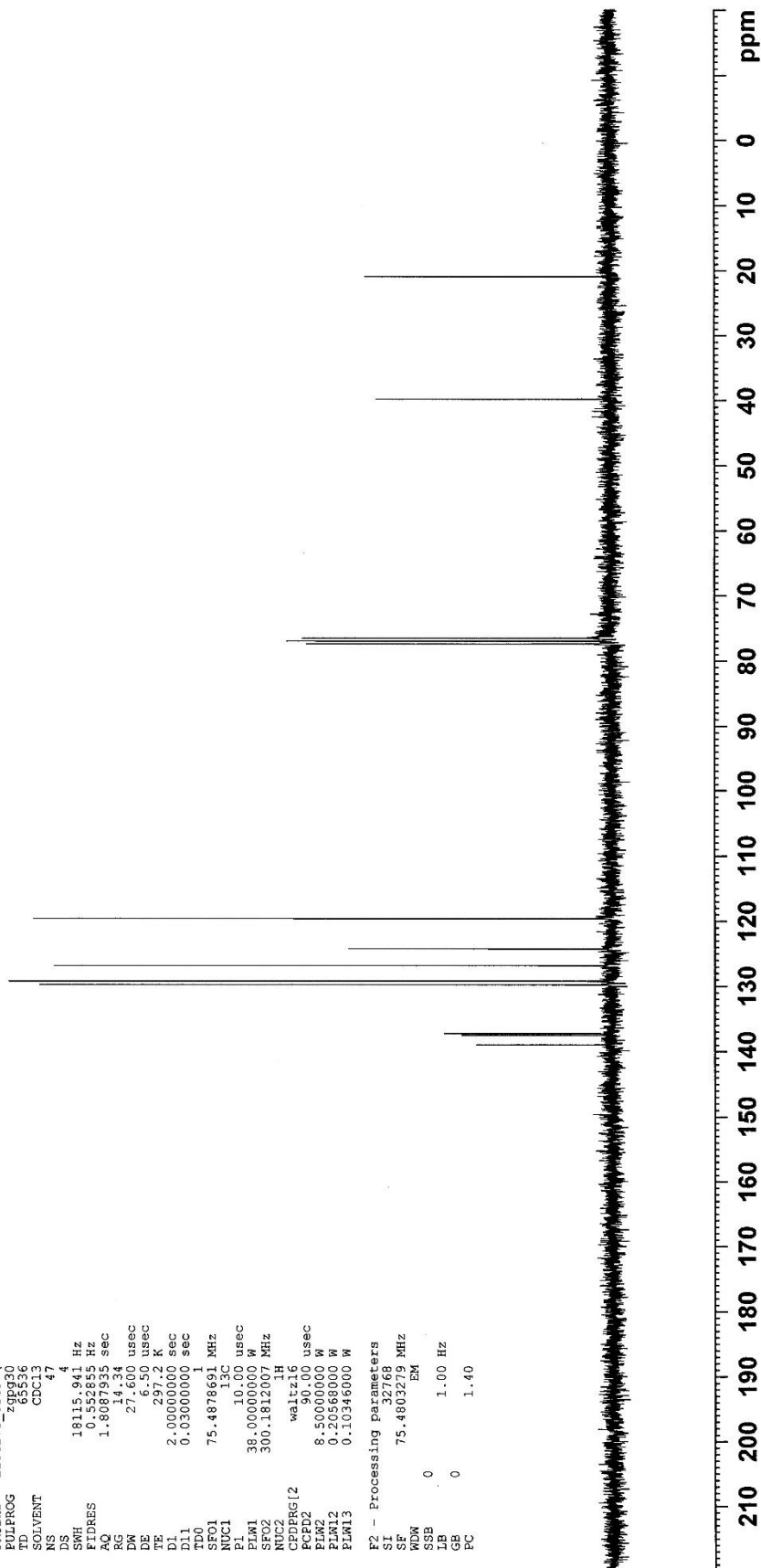
39.83

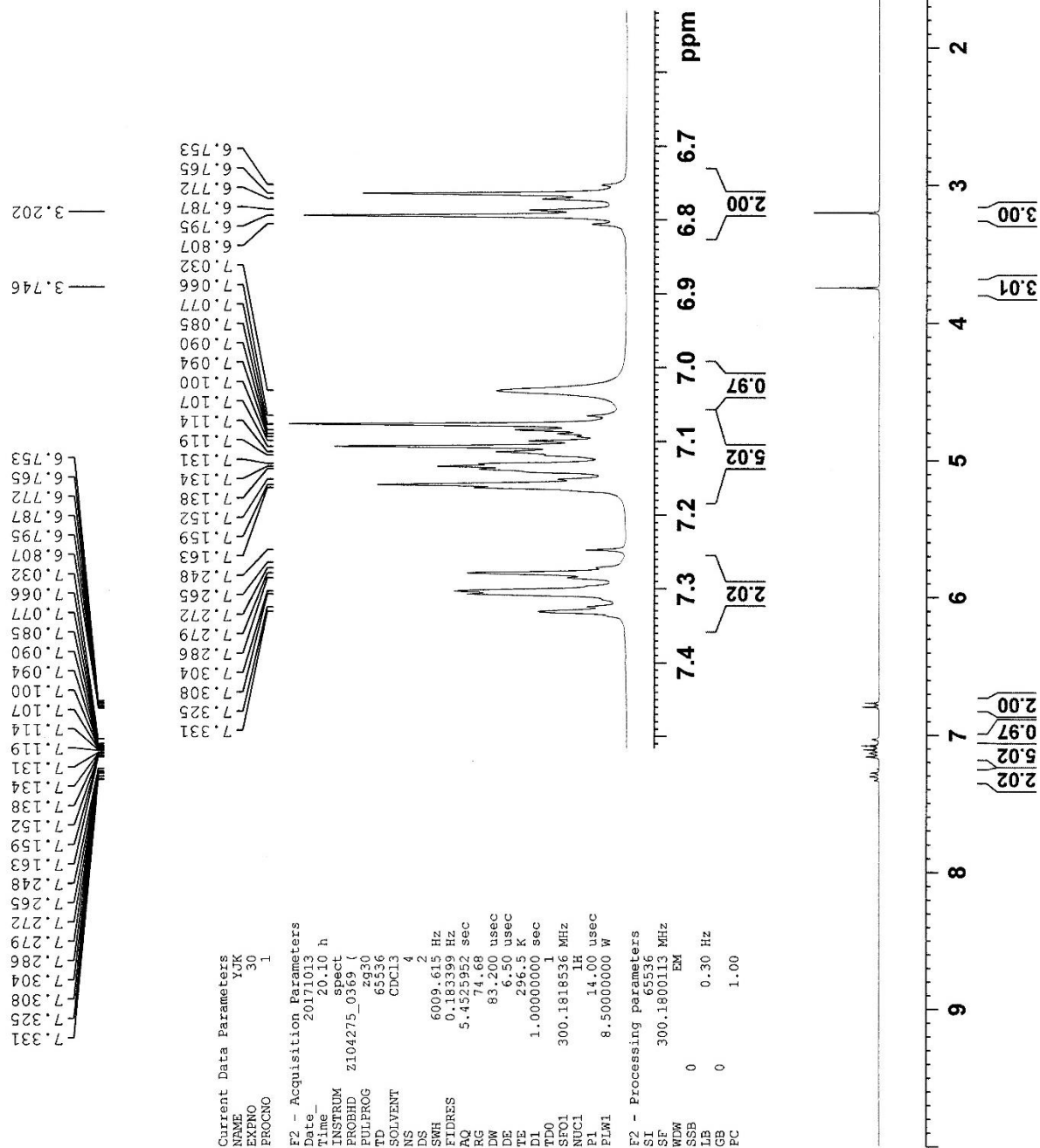
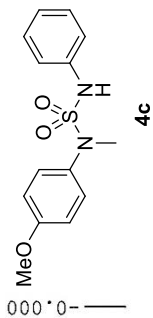
20.96

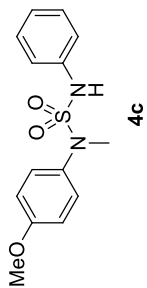
Current Data Parameters
NAME YJK
EXPNO 490
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170925
Time 21.27 h
INSTRUM spect
PROBHD zgpg30
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 47
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 1.8087935 sec
RG 14.34
DW 27.600 usec
DE 6.50 usec
TE 297.2 K
D1 2.0000000 sec
d11 0.0300000 sec
T1 1.1000000 sec
T2 0.0300000 sec
T20 1
SFO1 75.4878691 MHz
NUC1 13C
P1 10.00 usec
PLW1 38.0000000 W
SFO2 300.1812007 MHz
NUC2 1H
CDEPRG[2] waltz16
PCPD2 90.00 usec
PLW2 8.5000000 W
PLW12 0.2056800 W
PLW13 0.1034600 W

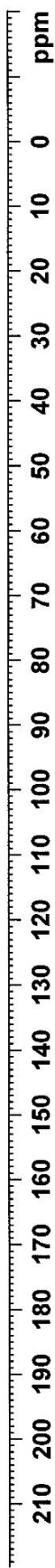
F2 - Processing Parameters
SI 32768
SF 75.4803279 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

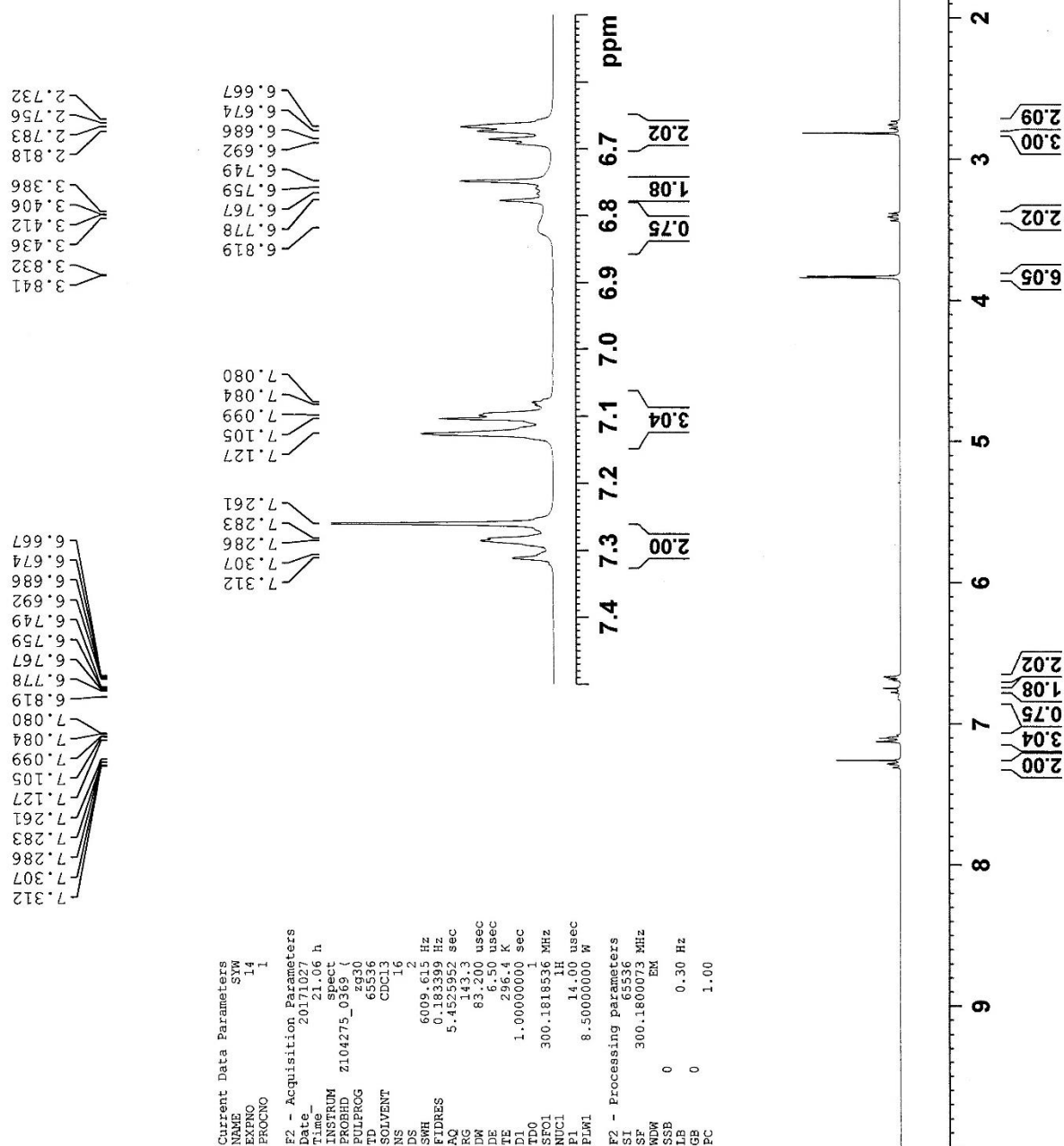
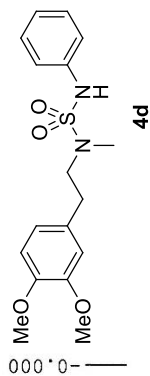


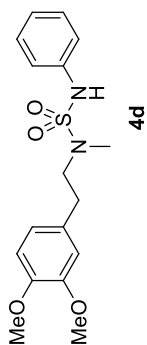




Current Data Parameters
NAME VJK
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171011
Time 21.11 h
INSTRUM spect
PROBHD z104275_Q369 (4mm) 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 443
DS 4
SWH 18115.941 Hz
FIDRES 0.352855 Hz
AQ 1.806932 sec
RG 12.62
DM 27.600 usec
DE 6.50 usec
TE 297.2 K
D1 2.0000000 sec
d11 0.0300000 sec
TDO
SFO1 75.4878691 MHz
NUC1 13C
P1 10.00 usec
PLW1 38.00000000 W
SFO2 300.1812007 MHz
NUC2 1H
PCPD2 waltz16
PCPD2 90.00 usec
PLW2 8.50000000 W
PLW12 0.20568000 W
PLW13 0.10346000 W
F2 - Processing Parameters
SI 32768
SF 75.4803300 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40







34.03
35.32
52.46
55.74
55.76

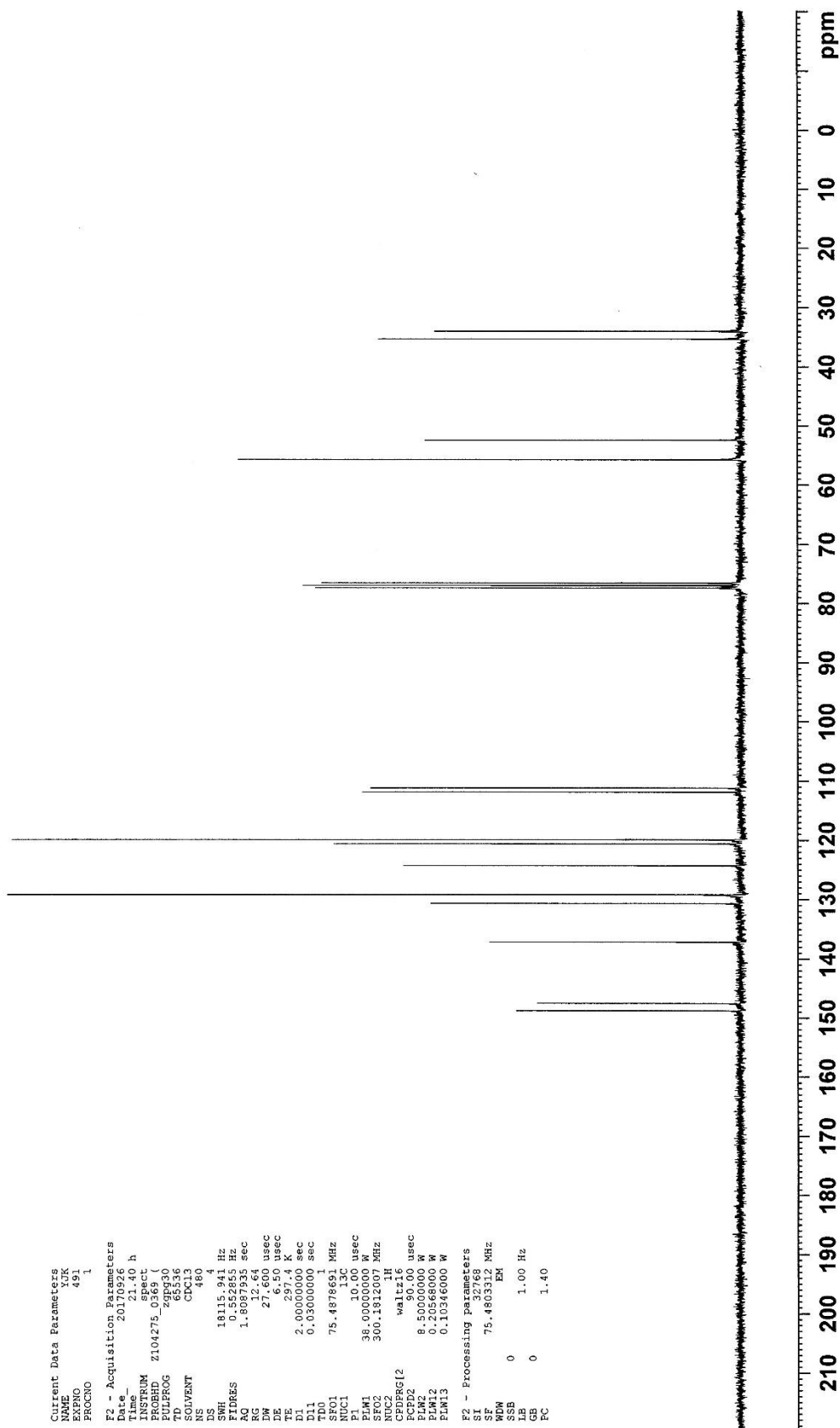
111.19
111.87
119.90
120.62
124.30
129.19
130.68
137.17

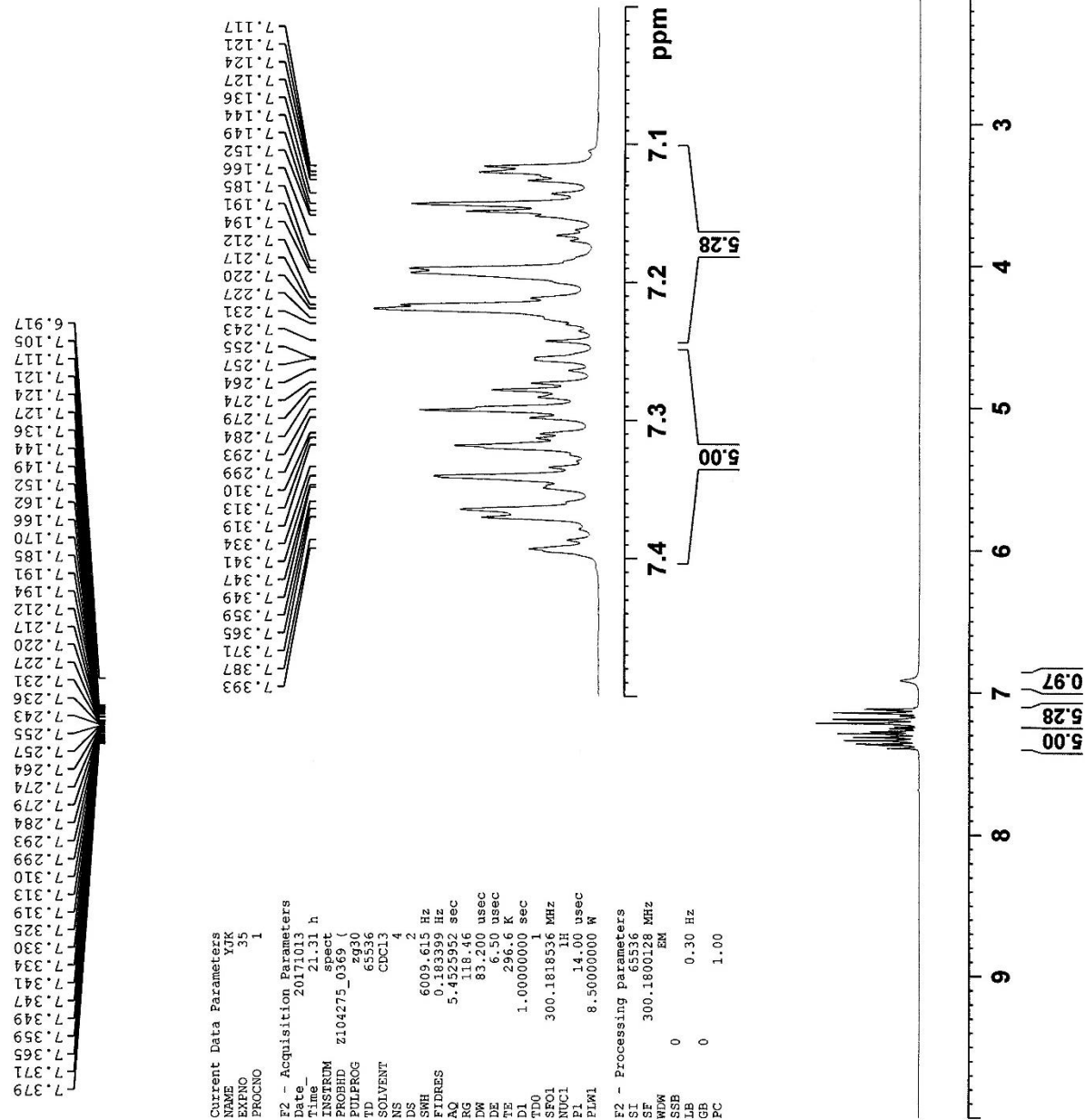
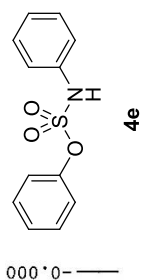
147.55
148.82

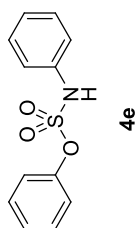
Current Data Parameters
NAME 50K
EXPNO 491
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170926
Time_ 21.40 h
INSTRUM spect
PROBHD Z104275.098
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 480
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 1.8087935 sec
RG 12.64
DW 27.600 usec
TE 29.50 usec
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 75.4878691 MHz
NUC1 13C
PL1 10.00 usec
SFO2 300.1912007 MHz
NUC2 1H
CPDPRG12 waltz16
PCPDW 8.50000000 usec
PLM2 0.20568000 W
PLM12 0.20568000 W
PLM13 0.10346000 W

F2 - Processing parameters
SI 32768
SF 75.480312 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





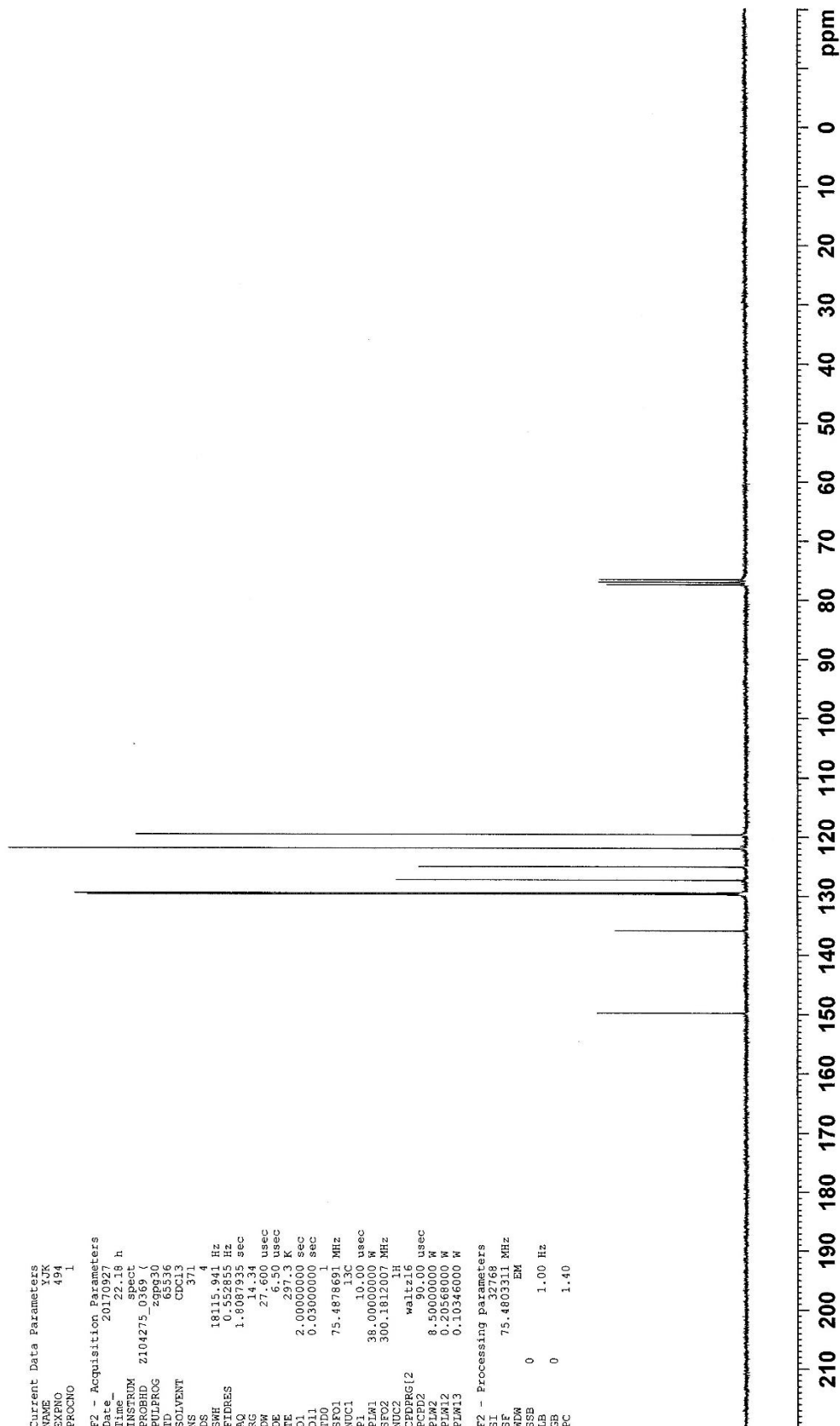


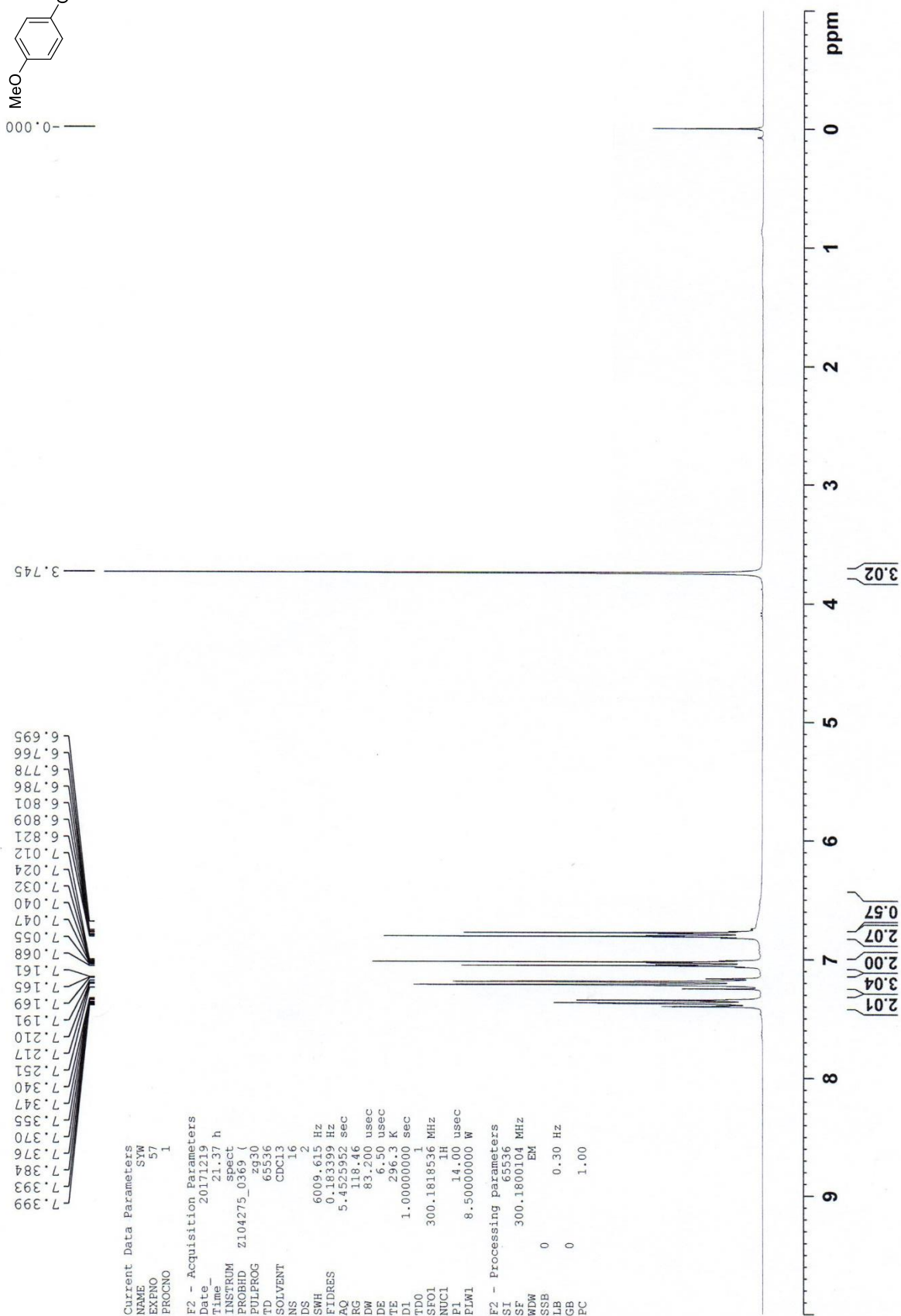
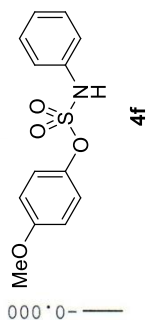
149.79
135.92
129.76
129.52
127.32
125.09
121.95
119.63

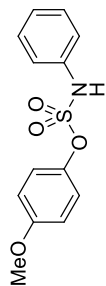
Current Data Parameters
NAME 1JK
EXPNO 494
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170927
Time 22.18 h
INSTRUM spect
PROBHD 2104275 0369 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 371
DS 4
SWH 18115.841 Hz
FIDRES 0.152851 Hz
AQ 1.6087935 sec
RG 14.34
DW 27.600 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 75.4878691 MHz
NUC1 13C
PL1 10.00 usec
PLW1 38.00000000 W
SFO2 300.1812007 MHz
NUC2 1H
PCPD2 waitz16
PCPD2 90.00 usec
PLW2 8.50000000 W
PLW12 0.20568000 W
PLW13 0.10346000 W

F2 - Processing Parameters
SI 32768
SF 75.4803311 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







4f

55.56

114.64

119.42

123.05

124.98

129.56

136.07

143.25

158.33

```

Current Data Parameters
NAME          SYW
EXPNO         115
PROCNO        1

F2 - Acquisition Parameters
Date_         20170810
Time          18.29
INSTRUM       spect
PROBHD        5 mm F4BBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            258
DS            4
SMH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG            2050
DW            27.733 usec
DE            6.50 usec
TE            296.3 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

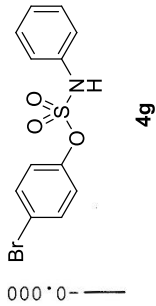
===== CHANNEL f1 =====
NUC1          13C
P1            9.00 usec
PL1           54.95398957 W
SFO1          75.4752950 MHz

===== CHANNEL f2 =====
CDEPRG2       waitz16
NUC2          1H
P2            90.00 usec
PL2           14.99699974 W
PLW12         0.21596000 W
PLW13         0.17493001 W
SFO2          300.1312005 MHz

F2 -- Processing parameters
SI            32768
SF            75.4677544 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

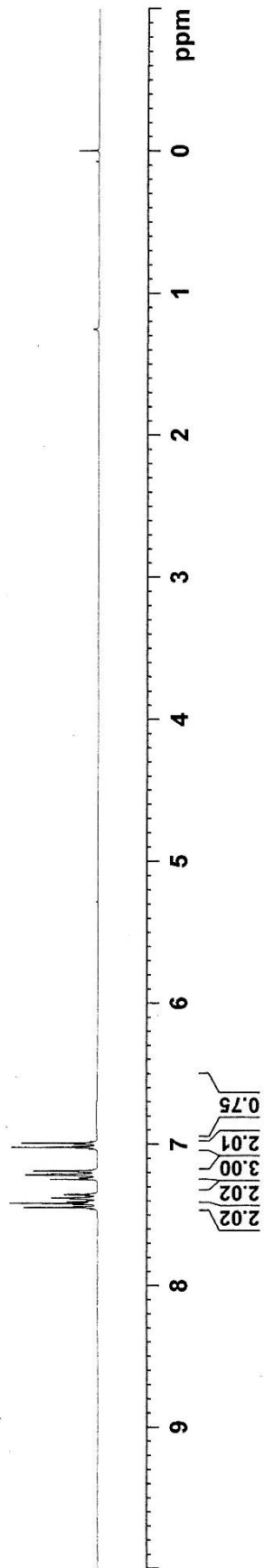
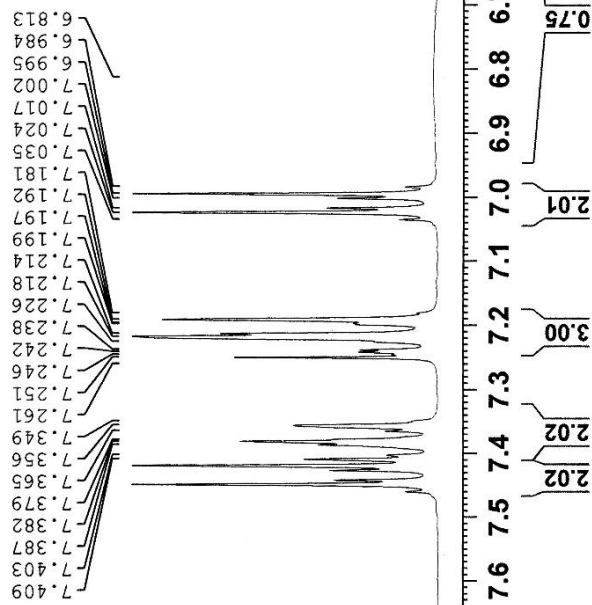
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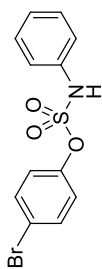
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



7.460
7.450
7.442
7.427
7.420
7.409
7.403
7.387
7.382
7.379
7.365
7.356
7.349
7.261
7.251
7.246
7.242
7.238
7.226
7.218
7.214
7.199
7.192
7.181
7.035
7.024
7.017
7.002
6.995
6.984
6.813

Current Data Parameters
NAME SW
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171024
Time_ 11.47 h
INSTRUM spect
PROBHD z104275_0369 (2950
PULPROG zgpg
ID 6536
SOLVENT CDC13
NS 16
DS 16
SWH 6009.615 Hz
FIDRES 0.183393 Hz
AQ 5.452922 sec
RG 127.02
DW 83.200 usec
DE 296.5 usec
TE 296.5 K
TD 1.00000000 sec
TDO 300.1818536 MHz
SFO1 300.1818536 MHz
NUC1 1H
P1 14.00 usec
PLW1 8.50000000 W
F2 - Processing parameters
SI 65536
SF 300.1800105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

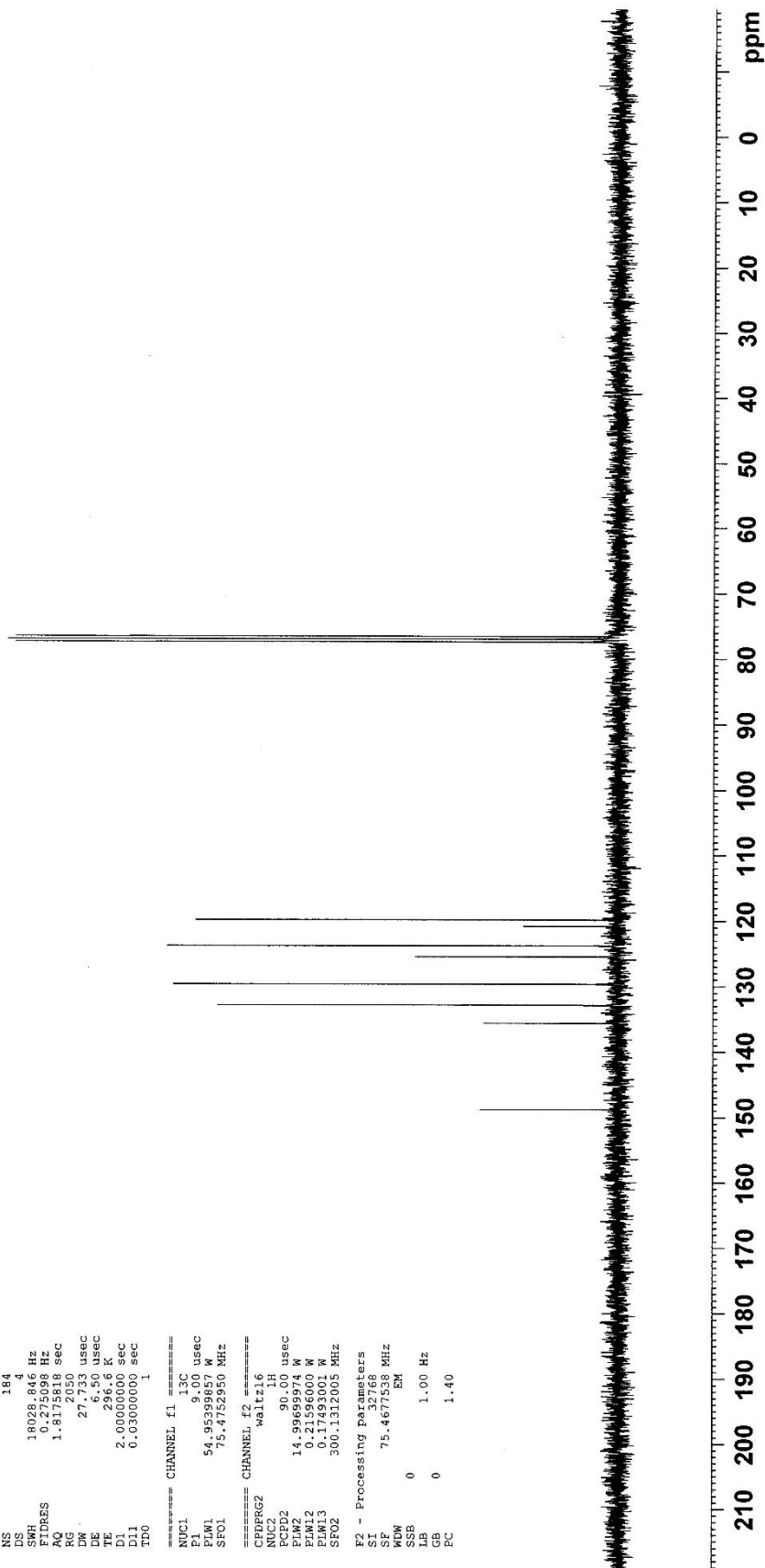


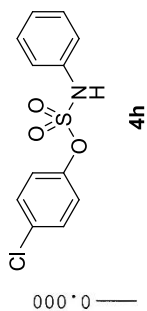


4g

148.80
135.62
132.88
129.67
125.46
123.80
120.79
119.83

Current Data Parameters
NAME_ 20170810
EXPNO 114
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170810
Time 18.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 184
DS 4
SWH 10028.844 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 Sec
RG 2050
DW 27.733 usec
DE 6.50 usec
TE 296.6 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 54.9539857 W
SFO1 75.4752950 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 14.9969974 W
PLW2 0.2159600 W
PLH3 0.0000000 W
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677538 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





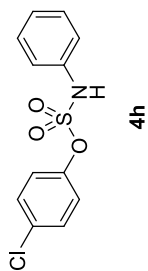
7.425
7.399
7.395
7.391
7.372
7.327
7.319
7.304
7.297
7.260
7.252
7.240
7.237
7.230
7.228
7.211
7.208
7.203
7.107
7.100
7.085
7.077
6.698

Current Data Parameters
NAME SWH
EXPNO 102
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170802
Time_ 16.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 14.9969974 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 65536
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7.425
7.399
7.395
7.391
7.372
7.327
7.319
7.304
7.297
7.260
7.252
7.240
7.237
7.230
7.228
7.211
7.208
7.203
7.107
7.100
7.085
7.077
6.698

7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 ppm
2.00 3.03 2.01 2.00 0.96

2.00 3.03 2.01 2.00 0.96



148.25
135.65
133.03
129.88
129.68
125.46
123.43
119.82

Current Data Parameters
NAME 105
EXPNO 435
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170927
Time_ 22:44 h
INSTRUM spect
PROBHD Z104275.0369 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 236
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 1.8087935 sec
RG 12.64
DW 27.600 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 75.4878691 MHz
NUC1 13C
P1 10.00 usec
PLW1 38.00000000 W
SFO2 300.1812007 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 8.50000000 W
PLW12 0.20568000 W
PLW13 0.10346000 W

F2 - Processing parameters
SI 32768
SF 75.4803261 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40

