Supporting Information

Regioselective One-pot Protection of D-Glucosamine

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Experimental Section:

Dichloromethane and acetonitrile were purified and dried from a safe purification system filled with anhydrous Al_2O_3. Flash column chromatography was carried out on Silica Gel 60 (230-400 mesh, E. Merk). TLC was performed on pre-coated glass plates of Silica Gel 60 F254 (0.25 mm, E. Merck); detection was executed by spraying with a solution of $Ce(NH_4)_2(NO_3)_6$, $(NH_4)_6Mo_7O_{24}$, and H_2SO_4 in water or ninhydrin and acetic acid solution in *n*-butanol and subsequent heating on a hot plate. Optical rotations were measured with a HORIBA Sepa-300 High sensitive polarimeter at ~25 °C. ^1H and ^{13}C NMR spectra were recorded with Bruker AMX400, 500 MHz and AVANCE-600 instruments. Chemical shifts are in ppm from Me_4Si, generated from the d-solvent lock signal. IR spectra were taken with a Perkin-Elmer Paragon 1000 FT-IR spectrometer. Elemental analyses were measured with a Perkin-Elmer 2400CHN instrument. Mass spectra were obtained with a FAB JMS-700 double focusing mass spectrometer (JEOL, Tokyo, Japan), MALDI Voyager DE-PRO (Applied Biosystem Houston, USA) and ESI Finnigan LCQ mass spectrometer (Thermo Finnigan, San Jose, CA, United States).

2-Azido-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-D-glucopyranose (3). Trifluoromethanesulfonic anhydride (0.94 mL, 5.57 mmol) was slowly added dropwise from an addition funnel to a solution of sodium azide (1.8 g, 27.8 mmol) in a mixture of water (4.7 mL) and dichloromethane (7.6 mL) at 0 °C. After stirring at the same temperature for 2 h, the organic layer was separated, and the aqueous layer was extracted with dichloromethane (2 x 10 mL). The combined organic layers were neutralized with saturated $NaHCO_{3(aq)}$. The generated trifluoromethanesulfonic azide (TfN_3) was directly used without further purification for the ensuing reaction. Sodium carbonate (0.59 g, 5.57 mmol) was added to a solution of D-glucosamine hydrochloride **4** (1.0 g, 4.6 mmol) in water (10 mL) until the *p*H value was around 10-11. After immersing this mixture in an ice-bath, $CuSO_4 \cdot 5H_2O$ (12 mg, 0.05 mmol) and the TfN_3 solution in dichloromethane were added sequentially. MeOH (10 mL) was added to the mixture until the phase became homogeneous. The ice-bath was, then, removed and the reaction was

kept stirring at room temperature for another 14 h. The mixture was filtered through celite, and the filtrate was concentrated *in vacuo*. The residue was purified by flash column chromatography (MeOH/$CHCl_3$ = 1/4) to afford the crude azido compound **5**. Triethylamine (Et_3N, 6.4 mL, 46 mmol) and chlorotrimethylsilane (2.8 mL, 22 mmol) were sequentially added to a solution of this crude 1,3,4,6-tetraol **5** in dichloromethane (11 mL) at 0 °C under nitrogen, and the resulting solution was kept stirring for 16 h. The solvent was evaporated *in vacuo*, and the residue was diluted with hexane followed by filtration. The filtrate was evaporated to get a solid, which was recrystallized in ethanol to afford the per-*O*-trimethylsilylated ether **3** (2.08 g, 91% in 2 steps, α/β = 1/3) as white crystallines. mp 57-58 °C; IR ($CHCl_3$) ν 2958, 2110, 1251 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 5.22 (d, *J* = 3.3 Hz, 1H), 4.49 (d, *J* = 7.7 Hz, 3H), 3.86 (dd, *J* = 9.8, 8.3 Hz, 1H), 3.73-3.64 (m, 9H), 3.56-3.51 (m, 4H), 3.21 (dd, *J* = 9.6, 8.5 Hz, 3H), 3.11 (ddd, *J* = 9.6, 4.3, 1.7 Hz, 3H), 3.07 (dd, *J* = 9.6, 7.7 Hz, 3H), 2.88 (dd, *J* = 9.8, 3.3 Hz, 1H), 0.20 (s, 9H), 0.17 (s, 27H), 0.16 (s, 36H), 0.15 (s, 9H), 0.12 (s, 27H), 0.08 (s, 27H), 0.07 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 97.1 (CH), 92.9 (CH), 76.8 (CH), 76.3 (CH), 73.1 (CH), 72.2 (CH), 72.0 (CH), 71.5 (CH), 69.3 (CH), 64.9 (CH), 61.7 (CH_2), 61.6 (CH_2), 0.8 (CH_3, TMS), 0.78 (CH_3, TMS), 0.76 (CH_3, TMS), 0.6 (CH_3, TMS), 0.0 (CH_3, TMS), -0.2 (CH_3, TMS), -0.4 (2 x CH_3, TMS); HRMS (FAB, MH^+) calcd for $C_{18}H_{44}N_3O_5Si_4$ 494.2358, found 494.2354.

General Procedure for the One-pot Synthesis of 3-Alcohols 6β, 10β, 14, and 15 from Compound 3. To a solution of compound **3** (0.265 g, 0.536 mmol) and ArCHO (0.562 mmol) in dichloromethane (3 mL) containing freshly dried 3Å molecular sieves (300 mg) was slowly added trimethylsilyl trifluoromethanesulfonate (TMSOTf, 80.4 μmoL) at 0 °C under nitrogen atmosphere. The mixture was stirred at the same temperature for 1.5 h, and acetic acid/benzoic acid (1.17 mmol) and tetra-*n*-butylammonium fluoride (TBAF 1.17 mmol) were sequentially added to the reaction solution. After stirring for another 4 h at 0 °C, acetic anhydride/benzoic anhydride (0.589 mmol) and Et_3N (5.36 mmol) were added to the solution, and the reaction mixture was kept stirring overnight at 0

°C. The whole mixture was filtered through a pad of celite, and the filtrate was washed with water (3 mL) and saturated $NaHCO_{3(aq.)}$ (3 mL). The aqueous layer was extracted with ethyl acetate (3 x 5 mL), and the combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (EtOAc/Hex = 1/3) to provide the desired 3-alcohol.

2-Azido-4,6-*O*-benzylidene-2-deoxy-β-D-glucopyranosyl Acetate (6β). (These data supersede the incorrect data provided for compound **10** on our paper found in *J. Org. Chem.* **2006**, *72*, 1226-1229.) $[\alpha]_D^{23}$ –37.7 (*c* 5.8, $CHCl_3$); IR ($CHCl_3$) ν 3472, 2114, 1762 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.50-7.43 (m, 2H, Ph-H) 7.40-7.33 (m, 3H, Ph-H), 5.50 (d, *J* = 8.5 Hz, 1H, H-1), 5.48 (s, 1H, PhCH), 4.30 (dd, *J* = 10.2, 4.8 Hz, 1H, H-6_{eq}), 3.68 (t, *J* = 10.2 Hz, 1H, H-6_{ax}), 3.66 (t, *J* = 9.5 Hz, 1H, H-3), 3.52-3.40 (m, 3H, H-2, H-3, H-5), 3.17 (br, 1H, OH-3), 2.15 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8 (C), 136.6 (C), 129.4 (C), 128.4 (2 x CH), 126.2 (2 x CH), 102.0 (CH), 93.0 (CH), 80.3 (CH), 72.1 (CH), 68.1 (CH_2), 66.8 (CH), 65.0 (CH), 20.8 (CH_3); HRMS (APCI, MH^+) calcd for $C_{15}H_{18}N_3O_6$ 336.1196, found 336.1205.

2-Azido-2-deoxy-4,6-*O*-(2-naphthylmethylidene)-β-D-glucopyranosyl Acetate (10β). $[\alpha]_D^{23}$ – 45.1 (*c* 5.0, $CHCl_3$); IR ($CHCl_3$) ν 3470, 2114, 1761, 1604, 1472 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 1H, Ar-H), 7.87-7.80 (m, 3H, Ar-H), 7.56 (d, *J* = 8.5 Hz, 1H, Ar-1), 7.51-7.47 (m, 2H, Ar-H), 5.62 (s, 1H, NaphCH), 5.51 (d, *J* = 8.4 Hz, 1H, H-1), 4.34 (dd, *J* = 10.0, 4.8 Hz, 1H, H-6_{eq}), 3.72 (t, *J* = 10.0 Hz, 1H, H-6_{ax}), 3.67 (t, *J* = 8.7 Hz, 1H, H-3), 3.55-3.43 (m, 3H, H2, H-4, H-5), 3.09 (s, 1H, OH-3), 2.17 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7 (C), 133.9 (C), 133.7 (C), 132.8 (C), 128.3 (3 x CH), 127.7 (2 x CH), 126.6 (2 x CH), 125.9 (2 x CH), 123.5 (2 x CH), 102.0 (CH), 93.0 (CH), 80.3 (CH), 72.2 (CH), 68.2 (CH_2), 66.8 (CH), 65.0 (CH), 20.8 (CH_3); HRMS (APCI, MH^+) calcd for $C_{19}H_{20}N_3O_6$ 386.1352, found 386.1356.

2-Azido-4,6-*O*-benzylidene-2-deoxy-β-D-glucopyranosyl Benzoate (14). $[\alpha]_D^{23}$ –95.0 (*c* 5.0, $CHCl_3$); IR ($CHCl_3$) ν 3478, 2114, 1739, 1601, 1493 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, *J* = 8.2, 1.0 Hz, 2H, Bz-H), 7.61 (t, *J* = 7.4 Hz, 1H, Bz-H) 7.52-7.45 (m, 4H, Bz-H, Ph-H), 7.40-7.36 (m, 3H, Ph-H), 5.80 (d, *J* = 8.4 Hz, 1H, H-1), 5.53 (s, 1H, PhCH), 4.36 (dd, *J* = 10.4, 4.5 Hz, 1H, H-6_{eq}), 3.86 (dt, *J* = 9.2, 2.6 Hz, 1H, H-3), 3.77 (dd, *J* = 9.2, 8.4 Hz, 1H, H-2), 3.74 (t, *J* = 10.2 Hz, 1H, H-6_{ax}), 3.64 (dd, *J* = 10.2, 9.2 Hz, 1H, H-4), 3.62 (dt, *J* = 10.2, 4.0 Hz, 1H, H-5); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4 (C), 136.6 (C), 134.0 (CH), 130.1 (2 x CH), 129.5 (2 x CH), 128.6 (2 x CH), 128.4 (2 x CH), 126.3 (2 x CH), 102.1 (CH), 93.7 (CH), 80.4 (CH), 72.2 (CH), 68.2 (CH_2), 66.9 (CH), 65.5 (CH); HRMS (APCI, MH^+) calcd for $C_{20}H_{20}N_3O_6$ 398.1352, found 398.1344.

2-Azido-2-deoxy-4,6-*O*-(2-naphthylmethylidene)-β-D-glucopyranosyl Benzoate (15). $[\alpha]_D^{23}$ – 114.2 (*c* 0.51, $CHCl_3$); IR ($CHCl_3$) ν 3345, 2945, 2107, 1741, 1267, 1081 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.11 (dd, *J* = 8.2, 1.0 Hz, 2H, Bz-H), 7.97 (s, 1H, Ar-H), 7.90-7.81 (m, 3H, Ar-H), 7.64-7.57 (m, 2H, Ar-H), 7.53-7.39 (m, 4H, Ar-H), 5.78 (d, *J* = 8.3 Hz, 2H, H-1), 5.66 (s, 1H, 2-NaphCH), 4.39 (dd, *J* = 10.5, 4.1 Hz, 1H, H-6a), 3.82-3.66 (m, 3H, H-6b, H-3, H-2), 3.64-3.50 (m, 2H, H-4, H-5), 3.12 (brs, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4 (C), 134.0 (CH), 133.8 (C), 132.8 (C), 130.1 (CH), 128.6 (CH), 128.5 (C), 128.3 (CH), 127.7 (CH), 126.6 (CH), 126.3 (CH), 125.9 (CH), 123.6 (CH), 102.2 (CH), 93.7 (CH), 80.4 (CH), 77.2 (C), 72.3 (CH), 68.3 (CH_2), 66. 9 (CH), 65.5 (CH); HRMS (FAB, MH^+) calcd for $C_{24}H_{22}N_3O_6$ 448.1508, found 448.1506.

General Procedure for the One-pot Synthesis of Fully Protected Derivatives 9, 13, 16, and 17 from Compound 3. TMSOTf (39.2 μmol) was added to a mixture of compound **3** (129 mg, 262 μmol), ArCHO (275 μmol), and freshly dried 3Å molecular sieves (150 mg) in dichloromethane (1.5 mL) at 0 °C under nitrogen atmosphere. The mixture was stirred at the same temperature for 1.5 h, and acetic acid/benzoic acid (575 μmol) and TBAF (575 μmol) were sequentially added to the reaction solution. After stirring for another 4 h at 0 °C, acetic anhydride/benzoic anhydride (785 μmol), Et_3N

(2.62 mmol) and 4-dimethylaminopyridine (DMAP, 26.2 μmol) were sequentially added to the mixture, the reaction flask was kept stirring for 12 h, and the temperature was gradually warmed up to room temperature. The whole mixture was filtered through a pad of celite, and the filtrate was consecutively washed with water (2 mL) and saturated $NaHCO_{3(aq.)}$ (2 mL). The aqueous layer was extracted with ethyl acetate (3 x 5 mL), and the combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (EtOAc/Hex = 1/4) to provide the expected fully protected derivative.

3-*O*-Acetyl-2-azido-4,6-*O*-benzylidene-2-deoxy-D-glucopyranosyl Acetate (9). α/β = 1/6. IR ($CHCl_3$) ν 2886, 2113, 1761, 1454, 1423 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43-7.37 (m, 14H, Ph-H), 7.36-7.30 (m, 21H, Ph-H), 6.26 (d, *J* = 3.7 Hz, 1H), 5.63 (d, *J* = 8.4 Hz, 6H), 5.59 (d, *J* = 3.6 Hz, 1H), 5.49 (s, 1H), 5.46 (s, 6H), 5.24 (t, *J* = 9.6 Hz, 6H), 4.3 (dd, *J* = 10.4, 4.4 Hz, 6H), 4.28 (dd, *J* = 10.4, 4.9 Hz, 1H), 4.15-4.06 (m, 1H), 3.97 (td, *J* = 9.8, 4.9 Hz, 1H), 3.78-3.55 (m, 25H), 3.52 (dd, *J* = 10.2, 3.7 Hz, 1H), 2.18 (s, 3H), 2.16 (s, 18H), 2.13 (s, 3H), 2.12 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7 (C), 169.4 (C), 168.8 (C), 168.4 (C), 136.5 (2 x C), 129.1 (2 x CH), 128.2 (4 x CH), 126.0 (4 x CH), 101.6 (CH), 101.5 (CH), 93.0 (CH), 90.8 (CH), 78.7 (CH), 78.2 (CH), 71.1 (CH), 69.1 (CH), 68.4 (CH_2), 68.1 (CH_2), 67.0 (CH), 64.8 (CH), 63.4 (CH), 60.8 (CH), 20.9 (CH_3), 20.8 (CH_3), 20.72 (CH_3), 20.68 (CH_3); HRMS (ESI, MNa^+) calcd for $C_{17}H_{19}N_3O_7Na$ 400.1121, found 400.1128.

3-*O*-Acetyl-2-azido-2-deoxy-4,6-*O*-(2-naphthylmethylidene)-D-glucopyranosyl Acetate (13). α/β = 1/6. IR ($CHCl_3$) ν 2872, 2113, 1760, 1604, 1424 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.92-7.78 (m, 29H), 7.56-7.43 (m, 20H), 6.28 (d, *J* = 3.7 Hz, 1H), 5.66 (d, *J* = 8.4 Hz, 1H), 5.64-5.58 (m, 8H), 5.29 (t, *J* = 9.5 Hz, 6H), 4.38 (dd, *J* = 10.4, 4.2 Hz, 6H), 4.33 (dd, *J* = 10.4, 4.8 Hz, 1H), 4.03 (td, *J* = 9.8, 4.8 Hz, 1H), 3.83-3.71 (m, 7H), 3.70-3.59 (m, 19H), 3.50 (dd, *J* = 10.4, 3.7 Hz, 1H), 2.18 (s, 21H), 2.14 (s, 21H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7 (C), 169.4 (C), 168.7 (C), 168.4 (C), 133.9 (2 x C), 133.6 (2 x C), 132.7 (2 x C), 128.3 (2 x CH), 128.0 (2 x CH), 127.6 (2 x CH), 126.4 (2 x CH), 126.1 (2 x CH), 125.6 (2 x CH), 123.5 (2 x CH), 101.8 (CH), 101.7 (CH), 93.0 (CH), 90.8 (CH), 78.7 (CH),

78.3 (CH), 71.1 (CH), 69.1 (CH), 68.4 (CH_2), 68.1 (CH_2), 67.0 (CH), 64.8 (CH), 63.4 (CH), 60.8 (CH), 20.9 (CH_3), 20.7 (CH_3), 20.69 (CH_3), 20.66 (CH_3); HRMS (ESI, MNa^+) calcd for $C_{21}H_{21}N_3O_7Na$ 450.1277, found 450.1287.

2-Azido-3-*O*-benzoyl-4,6-*O*-benzylidene-2-deoxy-β-D-glucopyranosyl Benzoate (16). $[\alpha]_D^{23}$ –85.6 (*c* 6.5, $CHCl_3$); IR ($CHCl_3$) ν 2876, 2112, 1736, 1602, 1452 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.14-8.09 (m, 4H, Bz-H), 7.64-7.55 (m, 2H, Bz-H), 7.50-7.43 (m, 4H, Bz-H), 7.42-7.38 (m, 2H, Ph-H), 7.32-7.28 (m, 3H, Ph-H), 6.00 (d, *J* = 8.4 Hz, 1H, H-1), 5.61 (t, *J* = 9.6 Hz, 1H, H-3), 5.53 (s, 1H, PhCH), 4.44, 4.41 (ABq, *J* = 10.3 Hz, 1H, H-6a), 4.03 (dd, *J* = 9.6, 8.4 Hz, 1H, H-2), 3.90 (t, *J* = 9.6 Hz, 1H, H-4), 3.85-3.77 (m, 2H, H-5, H-6b); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2 (C), 164.2 (C), 136.5 (C), 134.0 (CH), 133.4 (CH), 130.1 (2 x CH), 129.9 (2 x CH), 129.1 (CH), 128.6 (2 x CH), 128.4 (2 x CH), 128.2 (2 x CH), 126.1 (2 x CH), 101.6 (CH), 93.9 (CH), 78.5 (CH), 71.8 (CH), 68.2 (CH_2), 67.2 (CH), 64.1 (CH); HRMS (ESI, MNa^+) calcd for $C_{27}H_{23}N_3O_7Na$ 524.1434, found 524.1429.

2-Azido-3-*O*-benzoyl-2-deoxy-4,6-*O*-(2-naphthylmethylidene-β-D-glucopyranosyl Benzoate (17). $[\alpha]_D^{23}$ –127.4 (*c* 1.0, $CHCl_3$); IR ($CHCl_3$) ν 2111, 1744, 1725, 1645, 1451, 1258, 1087 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 8.12-8.08 (m, 4H, Bz-H), 7.87 (s, 1H, Ar-H), 7.80-7.76 (m, 3H, Ar-H), 7.62 (dd, *J* = 7.5, 1.1 Hz, 1H, Ar-H), 7.58 (dd, *J* = 7.5, 1.1 Hz, 1H, Ar-H), 7.50-7.42 (m, 7H, Ar-H), 6.00 (d, *J* = 8.4 Hz, 1H, H-1), 5.67 (s, 1H, NaphCH), 5.62 (t, *J* = 9.7 Hz, 1H, H-3), 4.48, 4.46 (ABq, *J* = 10.1 Hz, 1H, H-6a), 4.02 (dd, *J* = 9.7, 8.4 Hz, 1H, H-2), 3.95 (t, *J* = 9.7 Hz, 1H, H-4), 3.85 (m, 2H, H-5, H-6b); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.2 (C), 164.2 (C), 134.1 (CH), 133.9 (C), 133.7 (C), 133.4 (CH), 132.8 (C), 130.1 (CH), 129.9 (CH), 129.3 (C), 128.7 (CH), 128.5 (CH), 128.38 (C), 128.35 (CH), 128.1 (CH), 127.6 (CH), 126.4 (CH), 126.1 (CH), 125.7 (CH), 123.6 (CH), 101.9 (CH), 93.9 (CH), 78.7 (CH), 71.9 (CH), 68.4 (CH_2), 67.3 (CH), 64.2 (CH); HRMS (FAB, MH^+) calcd for $C_{31}H_{26}N_3O_7$ 552.1771, found 552.1764.

General Procedure for the One-pot Synthesis of 6-Alcohols 18, and 19 from Compound 3. Compound **3** (190 mg, 384 μmol) and ArCHO (404 μmol) were dissolved in dichloromethane (4 mL) under nitrogen atmosphere, the reaction flask was immersed in an ice bath, and TMSOTf (57.7 μmol) was added to the solution. The mixture was stirred at the same temperature for 1.5 h, and acetic anhydride (3.85 mmol) and $Cu(OTf)_2$ (76.8 μmol) were consecutively added to the solution. The ice bath was removed, the reaction flask was warmed up to 40 °C, and the solution was kept stirring for another 3 h. The reaction flask was cooled down to room temperature and then immersed in an ice bath. A 1 M solution of BH_3•THF complex in THF (1.93 mmol) and $Cu(OTf)_2$ (76.9 μmol) were sequentially added to the solution, the ice bath was removed, and the mixture was gradually warmed up to room temperature. After stirring for 16 h, the solution was cooled down to 0 °C, and the mixture was neutralized by Et_3N followed by slow quenching with methanol (5 mL). The resulting mixture was filtered through a pad of celite, and the filtrate was coevaporated with methanol under reduced pressure. Water (10 mL) was added to the mass, and the whole mixture was extracted with ethyl acetate (3 x 10 mL). The combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (EtOAc/Hex = 1/2.5) to give the desired 6-alcohol.

3-*O*-Acetyl-2-azido-4-*O*-benzyl-2-deoxy-D-glucopyranosyl Acetate (18). α/β = 1/1.8. IR ($CHCl_3$) ν 3499, 2112, 1756 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.34-7.22 (m, 14H), 6.20 (d, *J* = 2.4 Hz, 1H), 5.54-5.47 (m, 2.8H), 5.12 (t, *J* = 9.7 Hz, 1.8H), 4.67-4.52 (m, 5.6H), 3.88-3.63 (m, 9.2H), 3.52-3.44 (m, 3.8H), 3.39 (dd, *J* = 10.5, 3.4 Hz, 1H), 2.13 (s, 5.4H), 2.12 (s, 3H), 2.00 (s, 3H), 1.98 (s, 5.4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9 (C), 169.6 (C), 169.0 (C), 168.7 (C), 137.29 (C), 137.25 (C), 128.4 (4 x CH), 128.0 (2 x CH), 127.93 (3 x CH), 127.84 (CH), 92.6 (CH), 90.5 (CH), 76.1 (CH), 74.89 (CH), 74.84 (CH_2), 74.7 (CH), 74.6 (CH_2), 73.8 (CH), 73.3 (CH), 72.0 (CH), 63.0 (CH), 60.7 (2 x CH_2), 60.5 (CH), 20.9 (CH_3), 20.7 (3 x CH_3); HRMS (ESI, MNa^+) calcd for $C_{17}H_{21}N_3O_7Na$ 402.1277, found 402.1282.

3-*O*-Acetyl-2-azido-2-deoxy-4-*O*-(2-naphthylmethyl)-D-glucopyranosyl Acetate (19). α/β = 1/1.1**.** IR ($CHCl_3$) ν 3533, 2111, 1574 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.82-7.76 (m, 6.3H), 7.73-7.68 (m, 2.1H), 7.49-7.41 (m, 4.2H), 7.39-7.32 (m, 2.1H), 6.21 (d, *J* = 3.6 Hz, 1H), 5.55 (t, *J* = 9.4 Hz, 1.1H), 5.54 (d, *J* = 8.4 Hz, 1H), 5.16 (dd, *J* = 10.3, 9.4 Hz, 1.1H), 4.84-4.68 (m, 4.3H), 3.91-3.71 (m, 8.5 H), 3.55-3.45 (m, 2.2H), 3.42-3.33 (m, 2.1H), 2.14 (s, 6.3H), 1.95 (s, 3H), 1.92 (s, 3.3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9 (C), 169.6 (C), 169.0 (C), 168.7 (C), 134.8 (C), 134.7 (C), 133.1 (2 x C), 133.0 (2 x C), 128.3 (2 x CH), 127.9 (2 x CH), 127.6 (2 x CH), 126.8 (CH), 126.7 (CH), 126.2 (2 x CH), 126.1 (2 x CH), 125.8 (CH), 125.6 (CH), 92.7 (CH), 90.5 (CH), 76.2 (CH), 75.1 (CH), 75.0 (CH_2), 74.8 (CH), 75.0 (CH_2), 73.9 (CH), 73.4 (CH), 72.0 (CH), 63.1 (CH), 60.8 (2 x CH_2), 60.6 (CH), 20.9 (CH_3), 20.79 (CH_3), 20.74 (2 x CH_3); HRMS (ESI, MNa^+) calcd for $C_{21}H_{23}N_3O_7Na$ 452.1434, found 452.1443.

General Procedure for the One-pot Synthesis of Fully Protected Derivatives 20 and 21 from Compound 3. TMSOTf (60.7 μmol) was added to a solution of compound **3** (142 mg, 288 μmol) and ArCHO (302 μmol) in dichloromethane (4 mL) at 0 °C under nitrogen atmosphere. The mixture was stirred at same temperature for 1.5 h, and acetic anhydride (2.87 mmol) and $Cu(OTf)_2$ (57.7 μmol) were consecutively added to the solution. After the ice bath was removed, the reaction was warmed up and kept stirring at 40 °C for another 3 h. The flask was cooled down to room temperature and then immersed in an ice bath. Acetonitrile (4 mL), dimethylethylsilane (Me_2EtSiH, 2.87 mmol) and $Cu(OTf)_2$ (57.7 μmol) were consecutively added to the solution, and the reaction was kept stirring at the same temperature for another 2 h. The solution was neutralized by Et_3N, the whole mixture was filtered through a pad of celite, and the filtrate was washed with saturated $NaHCO_{3(aq.)}$ (3 mL). The aqueous layer was extracted with ethyl acetate (3 x 5 mL), and the combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (EtOAc/Hex = 1/3) to afford the fully protected derivative.

3,4-di-*O*-Acetyl-2-azido-6-*O*-benzyl-2-deoxy-D-glucopyranosyl Acetate (20). α/β = 1/1.9. IR ($CHCl_3$) ν 2113, 1756, 1075, 1042 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.32-7.28 (m, 5.8H), 7.27-7.23 (m, 8.7H), 6.27 (d, *J* = 3.6 Hz, 1H), 5.51 (d, *J* = 8.5 Hz, 1.9H), 5.40 (dd, *J* = 10.2, 9.6 Hz, 1H), 5.19 (t, *J* = 10.2 Hz, 1H), 5.13 (t, *J* = 9.8 Hz, 1.9H), 5.02 (t, *J* = 9.8 Hz, 1.9H), 4.56, 4.38 (ABq, *J* = 12.1 Hz, 3.8H), 4.53, 4.41 (ABq, *J* = 12.0 Hz, 2H), 3.97 (dt, *J* = 10.2, 3.4 Hz, 1H), 3.70 (dt, *J* = 9.8, 3.4 Hz, 1.9H), 3.64 (dd, *J* = 9.8, 8.5 Hz, 1.9H), 3.62 (dd, *J* = 10.2, 3.6 Hz, 1H), 3.55 (dd, *J* = 11.0, 3.4 Hz, 1.9H), 3.51 (dd, *J* = 11.0, 3.4 Hz, 1H), 3.46 (dd, *J* = 11.0, 3.4 Hz, 2.9H), 2.15 (s, 5.7H), 2.14 (s, 3H), 2.07 (s, 3H), 2.06 (s, 5.7H), 1.88 (s, 3H), 1.84 (s, 5.7H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2 (C), 169.9 (C), 169.5 (C), 169.4 (C), 168.7 (C), 168.6 (C), 137.32 (C), 137.30 (C), 128.3 (2 x CH), 128.0 (2 x CH), 127.9 (CH), 127.8 (CH), 92.6 (CH), 90.0 (CH), 73.8 (2 x CH), 73.5 (CH_2), 73.4 (CH_2), 72.9 (CH), 70.9 (CH), 68.4 (CH), 68.3 (CH), 67.5 (CH_2), 67.2 (CH_2), 62.5 (CH), 60.2 (CH), 20.9 (CH_3), 20.8 (CH_3), 20.7 (CH_3), 20.6 (CH_3), 20.5 (2 x CH_3); HRMS (ESI, MNa^+) calcd for $C_{19}H_{23}N_3O_8Na$ 444.1383, found 444.1380.

3,4-di-*O*-Acetyl-2-azido-2-deoxy-6-*O*-(2-naphthylmethyl)-D-glucopyranosyl Acetate (21). α/β = 1/2.5. IR ($CHCl_3$) ν 2113, 1756, 1075, 1042 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.82-7.77 (m, 10H), 7.71-7.69 (m, 3.5H), 7.47-7.43 (m, 7H), 7.41-7.37 (m, 3.5H), 6.29 (d, *J* = 3.5 Hz, 1H), 5.53 (d, *J* = 8.6 Hz, 2.5H), 5.41 (t, *J* = 10.0 Hz, 1H), 5.24 (t, *J* = 10.0 Hz, 1H), 5.17 (t, *J* = 9.9 Hz, 2.5H), 5.03 (t, *J* = 9.9 Hz, 2.5H), 4.76 (d, *J* = 12.2 Hz, 2.5H), 4.73, 4.57 (ABq, *J* = 12.5 Hz, 2H), 4.55 (d, *J* = 12.2 Hz, 2.5H), 3.98 (dt, *J* = 10.0, 3.0 Hz, 1H), 3.72 (dt, *J* = 9.9, 3.0 Hz, 2.5H), 3.65 (dd, *J* = 9.9, 8.6 Hz, 2.5H), 3.64 (dd, *J* = 10.0, 3.5 Hz, 1H), 3.60 (dd, *J* = 9.9, 3.0 Hz, 2.5H), 3.56 (dd, *J* = 10.0, 3.0 Hz, 1H), 3.50 (dd, *J* = 10.0, 3.0 Hz, 3.5H), 2.17 (s, 7.5H), 2.15 (s, 3H), 2.08 (s, 3H), 2.07 (s, 7.5H), 1.81 (s, 3H), 1.75 (s, 7.5H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2 (C), 169.9 (C), 169.6 (C), 169.5 (C), 168.7 (C), 168.6 (C), 134.8 (2 x C), 133.1 (2 x C), 133.0 (2 x C), 128.2 (2 x CH), 127.8 (2 x CH), 127.6 (2 x CH), 126.9 (CH), 126.8 (CH), 126.1 (2 x CH), 126.0 (2 x CH), 125.9 (CH), 125.8 (CH), 92.6 (CH), 90.1 (CH), 73.8 (2 x CH), 73.6 (CH_2), 73.5 (CH_2), 73.0 (CH), 71.0 (CH), 68.4 (CH), 68.2 (CH), 67.5 (CH_2), 67.2

(CH_2), 62.5 (CH), 60.2 (CH), 20.9 (2 x CH_3), 20.7 (CH_3), 20.6 (CH_3), 20.5 (CH_3), 20.4 (CH_3); HRMS (ESI, MNa^+) calcd for $C_{23}H_{25}N_3O_8Na$ 494.1539, found 464.1541.

General Procedure for the One-pot Synthesis of 4-Alcohols 22 and 23 from Compound 3. TMSOTf (60.7 μmol) was added to a solution of compound **3** (200 mg, 405 μmol) and ArCHO (425 μmol) in dichloromethane (4 mL) at 0 °C under nitrogen atmosphere. The mixture was stirred at the same temperature for 1.5 h, and acetic anhydride (1.01 mmol) and $Cu(OTf)_2$ (81.1 μmol) were sequentially added to the mixture. The ice bath was removed, and the reaction flask was warmed up and kept stirring at 40 °C for 3 h. After cooling to room temperature, the reaction was cooled down to 0 °C, and acetonitrile (4 mL), dimethylethylsilane (Me_2EtSiH, 1.62 mmol) and $Cu(OTf)_2$ (81.1 μmol) were consecutively added to the solution. The solution was continuously stirred for another 2 h at the same temperature, and Et_3N was added to neutralize the reaction. The whole mixture was filtered through a pad of celite, and the filtrate was washed with saturated $NaHCO_{3(aq.)}$ (3 mL). The aqueous layer was extracted with ethyl acetate (3 x 5 mL), and the combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (EtOAc/Hex = 1/2.5) to afford the expected 4-alcohol.

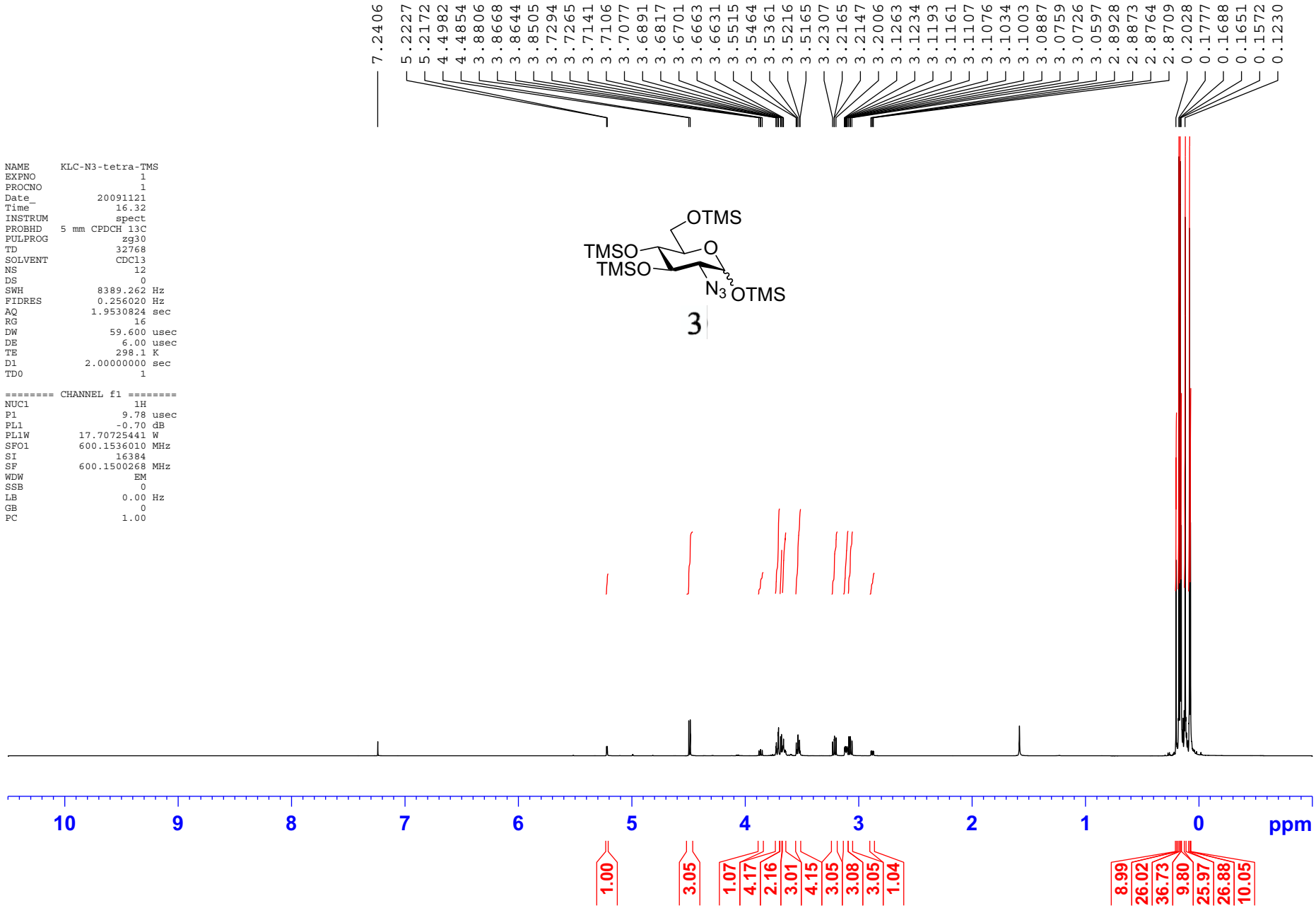
3-*O*-Acetyl-2-azido-6-*O*-benzyl-2-deoxy-D-glucopyranosyl Acetate (22). α/β = 1/1.1. IR ($CHCl_3$) ν 3469, 2112, 1574 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35-7.24 (m, 10.5H), 6.22 (d, *J* = 3.6 Hz, 1H), 5.50 (d, *J* = 8.4 Hz, 1.1H), 5.29 (dd, *J* = 10.4, 9.0 Hz, 1H), 4.91 (t, *J* = 9.7 Hz, 1.1H), 4.59-4.46 (m, 4.2H), 3.86-3.66 (m, 6.3H), 3.64 (dd, *J* = 10.4, 3.6 Hz, 1H), 3.57-3.46 (m, 3.2H), 3.17 (br, 2.1H), 2.15-2.11 (m, 12.6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2 (C), 171.0 (C), 168.8 (C), 168.7 (C), 137.4 (2 x C), 128.4 (4 x CH), 127.8 (2 x CH), 127.7 (4 x CH), 92.6 (CH), 90.3 (CH), 75.3 (CH_2), 75.2 (CH_2), 73.67 (CH), 73.58 (CH), 73.2 (CH), 72.6 (CH), 69.63 (CH), 69.57 (CH), 68.9 (CH_2), 68.7 (CH_2), 62.5 (CH), 60.1 (CH), 20.86 (CH_3), 20.79 (3 x CH_3); HRMS (ESI, MNa^+) calcd for $C_{17}H_{21}N_3O_7Na$ 402.1277, found 402.1285.

3-*O*-Acetyl-2-azido-2-deoxy-6-*O*-(2-naphthylmethyl)-D-glucopyranosyl Acetate (23). α/β = 1/1.4. IR ($CHCl_3$) ν 3476, 2112, 1754 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.83-7.76 (m, 7.2H), 7.75-7.69 (m, 2.4H), 7.50-7.33 (m, 7.2H), 6.25 (d, *J* = 3.5 Hz, 1H), 5.54 (d, *J* = 8.5 Hz, 1.4H), 5.31 (t, *J* = 10.0 Hz, 1H), 4.94 (t, *J* = 9.9 Hz, 1.4H), 4.74-4.61 (m, 4.8H), 3.91-3.64 (m, 8H), 3.60-3.49 (m, 3H), 3.46 (dd, *J* = 10.0, 3.5 Hz, 1H), 3.39-3.27 (m, 2.4H), 2.14 (s, 11.4H), 2.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1 (C), 171.0 (C), 168.8 (C), 168.6 (C), 134.9 (2 x C), 133.0 (2 x C), 132.8 (2 x C), 128.13 (CH), 128.08 (CH), 127.7 (CH), 127.5 (CH), 126.5 (CH), 126.05 (CH), 126.01 (CH), 125.86 (CH), 125.83 (CH), 125.5 (CH), 92.6 (CH), 90.2 (CH), 75.3 (CH), 75.2 (CH), 73.7 (CH_2), 73.5 (CH_2), 73.1 (CH), 72.7 (CH), 69.3 (CH), 69.2 (CH), 68.7 (CH_2), 68.6 (CH_2), 62.4 (CH), 60.0 (CH), 20.7 (4 x CH_3); HRMS (ESI, MNa^+) calcd for $C_{21}H_{23}N_3O_7Na$ 452.1434, found 452.1437.

General Procedure for the One-pot Synthesis of 1-Alcohols 24 and 25 from Compound 3. TMSOTf (56.8 μmol) was added to a solution of compound **3** (187 mg, 379 μmol) and ArCHO (398 μmol) in dichloromethane (4 mL) at 0 °C under nitrogen atmosphere, and the mixture was stirred at same temperature for 1.5 h. Acetic anhydride (3.79 mmol) and $Cu(OTf)_2$ (75.8 μmol) were consecutively added to the solution, the ice bath was removed, and the reaction flask was warmed up and kept stirring at 40 °C for 3 h. A mixed solvent of MeOH/THF (19 mL, v/v = 1/5) was added to the solution, the flask was immersed in an ice bath, and ammonia gas was passed through the mixture for 20 min. After stirring at the same temperature for another 2 h, the mixture was filtered through a pad of celite, and the filtrate was evaporated under reduced pressure. Saturated $NaHCO_{3(aq.)}$ (10 mL) was added to the resulting mass, and the mixture was extracted with ethyl acetate (3 x 10 mL). The combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (EtOAc/Hex = 1/1.8) to give the desired 1-alcohol.

3-*O*-Acetyl-2-azido-4,6-*O*-benzylidene-2-deoxy-D-glucopyranose (24). α/β = 1/1. $[\alpha]_D^{23}$ –139.3 (*c* 0.2, $CHCl_3$); IR ($CHCl_3$) ν 3422, 2112, 1742 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.44-7.31 (m, 6H), 5.62 (t, *J* = 10.0 Hz, 1H), 5.48 (s, 1H), 5.46 (s, 1H), 5.36 (d, *J* = 3.5 Hz, 1H), 5.15 (t, *J* = 9.8 Hz, 1H), 5.78 (d, *J* = 7.9 Hz, 1H), 4.30 (dd, *J* = 10.0, 4.9 Hz, 1H), 4.17 (td, *J* = 9.8, 4.6 Hz, 1H), 3.64-3.57 (m, 2H), 3.76 (t, *J* = 9.8 Hz, 1H), 3.71 (t, *J* = 9.8 Hz, 1H), 3.49 (td, *J* = 9.8, 4.9 Hz, 1H), 3.43 (dd, *J* = 9.8, 7.9 Hz, 1H), 3.32 (dd, *J* = 10.0, 3.5 Hz, 1H), 2.12 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.88 (C), 169.76 (C), 136.9 (C), 136.7 (C), 129.1 (2 x CH), 128.3 (4 x CH), 126.18 (2 x CH), 126.10 (2 x CH), 101.7 (CH), 101.5 (CH), 96.7 (CH), 93.2 (CH), 79.5 (CH), 78.6 (CH), 71.2 (CH), 69.1 (CH), 68.8 (CH_2), 68.4 (CH_2), 66.6 (CH), 65.9 (CH), 62.8 (CH), 62.3 (CH), 20.8 (2 x CH_3); HRMS (ESI, MNa^+) calcd for $C_{15}H_{17}N_3O_6Na$ 358.1015, found 358.1022.

3-*O*-Acetyl-2-azido-2-deoxy-4,6-*O*-(2-naphthylmethylidene)-D-glucopyranose (25). α/β = 2.3/1. $[\alpha]_D^{23}$ –20.9 (*c* 0.7, $CHCl_3$); IR ($CHCl_3$) ν 3417, 2111, 1741 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.90-7.81 (m, 13H), 7.56-7.43 (m, 10H), 5.70-5.60 (m, 5.6H), 5.37 (br, 2.3H), 5.18 (t, *J* = 9.8 Hz, 1H), 4.79 (d, *J* = 6.8 Hz, 1H), 4.36-4.18 (m, 6.4H), 4.07 (br, 1H), 3.84-3.73 (m, 3.3H), 3.70-3.62 (m, 3.3H), 3.56-3.48 (m, 1H), 3.48-3.41 (m, 3.3H), 3.36-3.29 (m, 2.3H), 2.12 (s, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0 (C), 169.8 (C), 134.2 (C), 134.1 (C), 133.7 (C), 132.8 (C), 128.4 (2 x CH), 128.1 (2 x CH), 127.7 (2 x CH), 126.5 (CH), 126.2 (CH), 125.8 (CH), 125.6 (CH), 123.7 (CH), 123.6 (CH), 101.9 (CH), 101.8 (CH), 96.7 (CH), 93.2 (CH), 79.5 (CH), 78.6 (CH), 71.2 (CH), 69.0 (CH), 68.9 (CH_2), 68.5 (CH_2), 66.6 (CH), 65.8 (CH), 62.8 (CH), 62.3 (CH), 20.9 (2 x CH_3); HRMS (ESI, MNa^+) calcd for $C_{19}H_{19}N_3O_6Na$ 408.1172, found 408.1179.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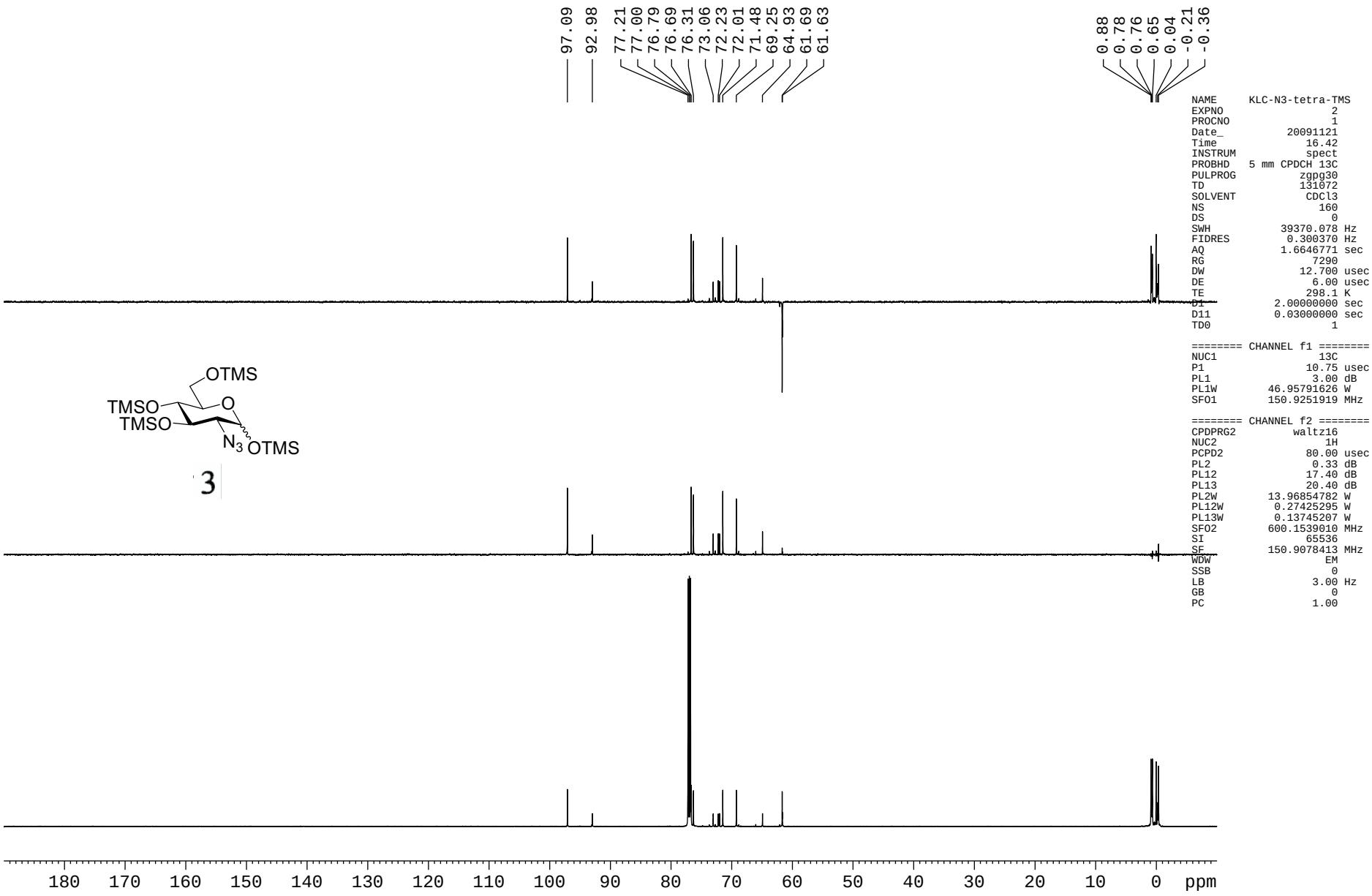
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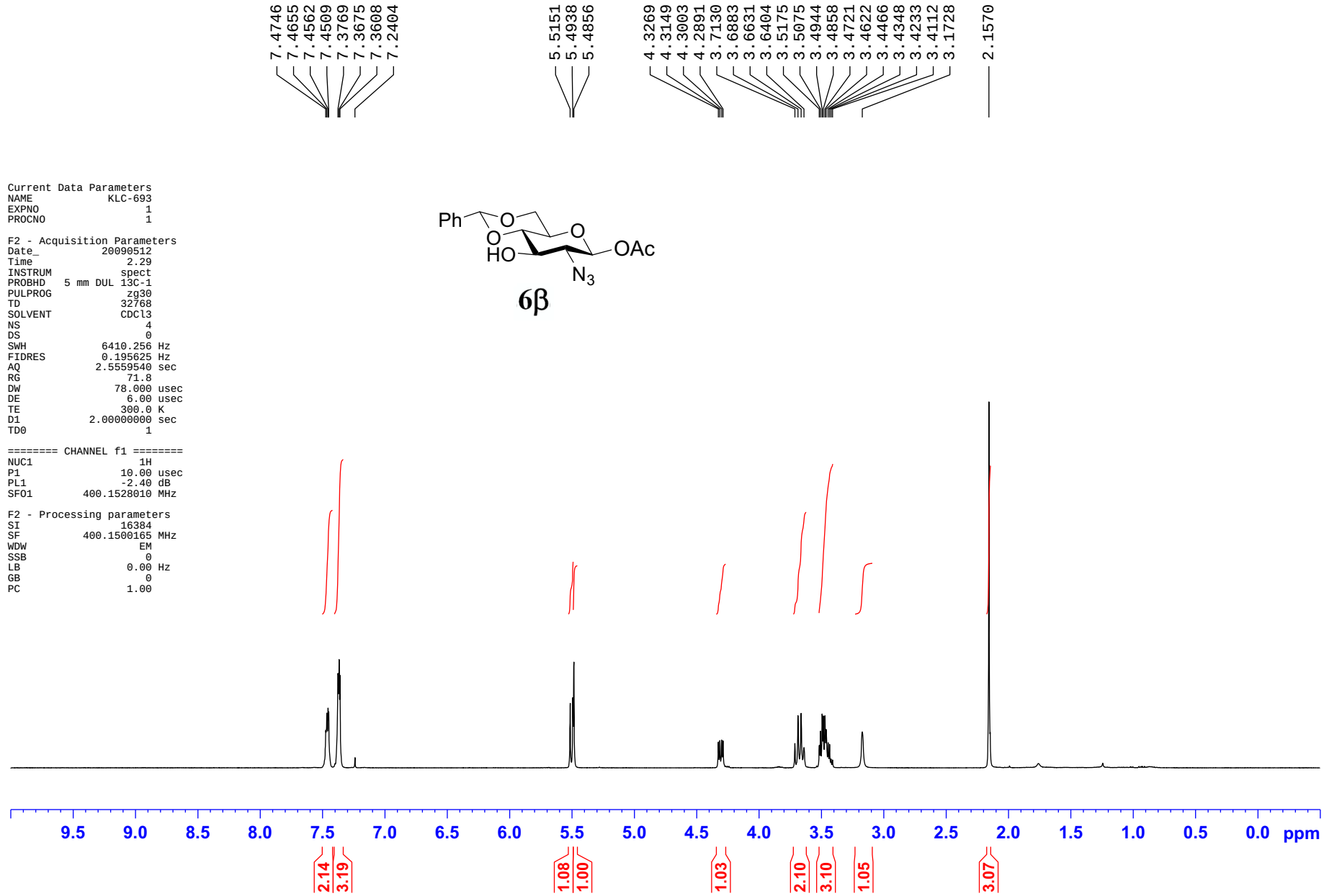
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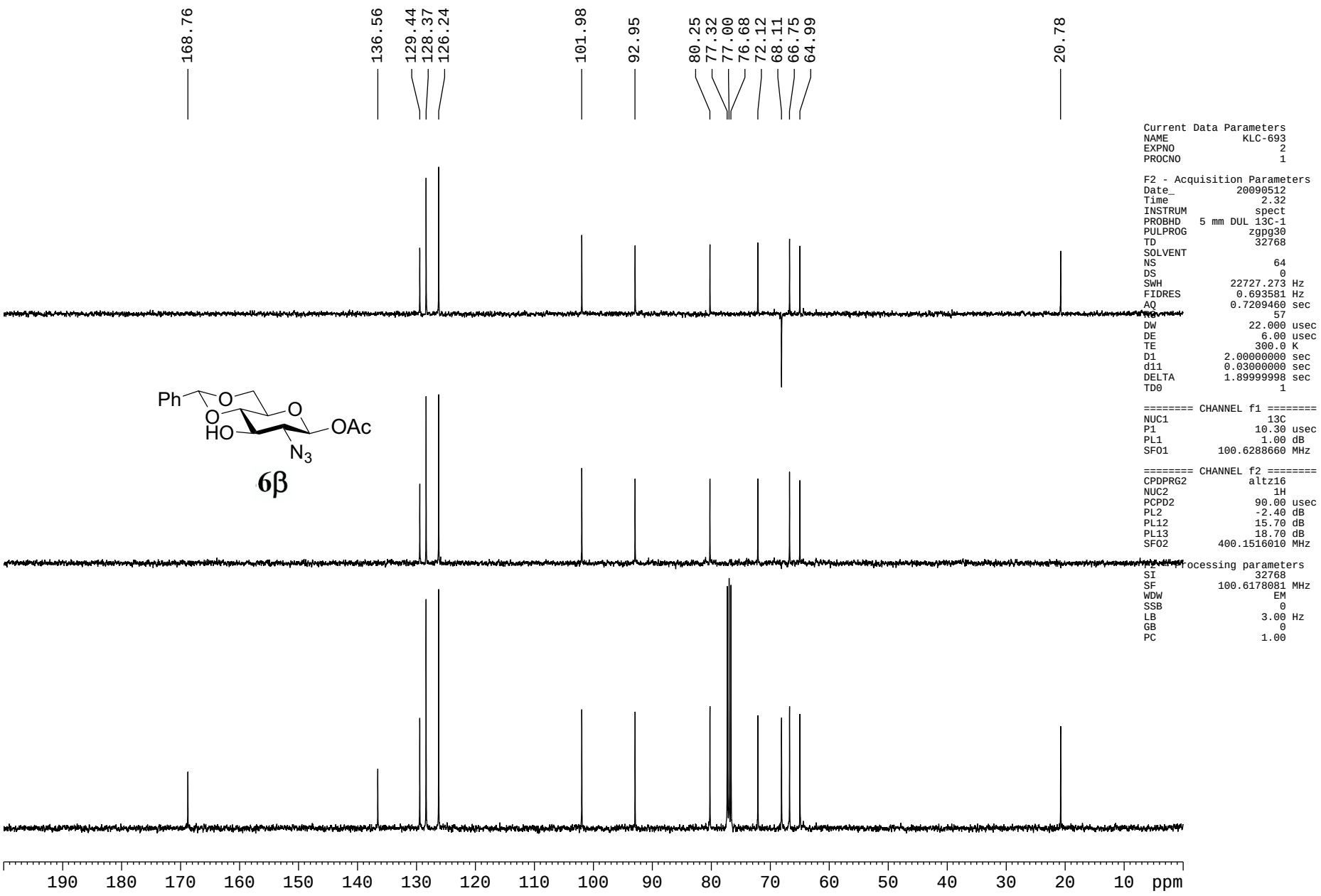
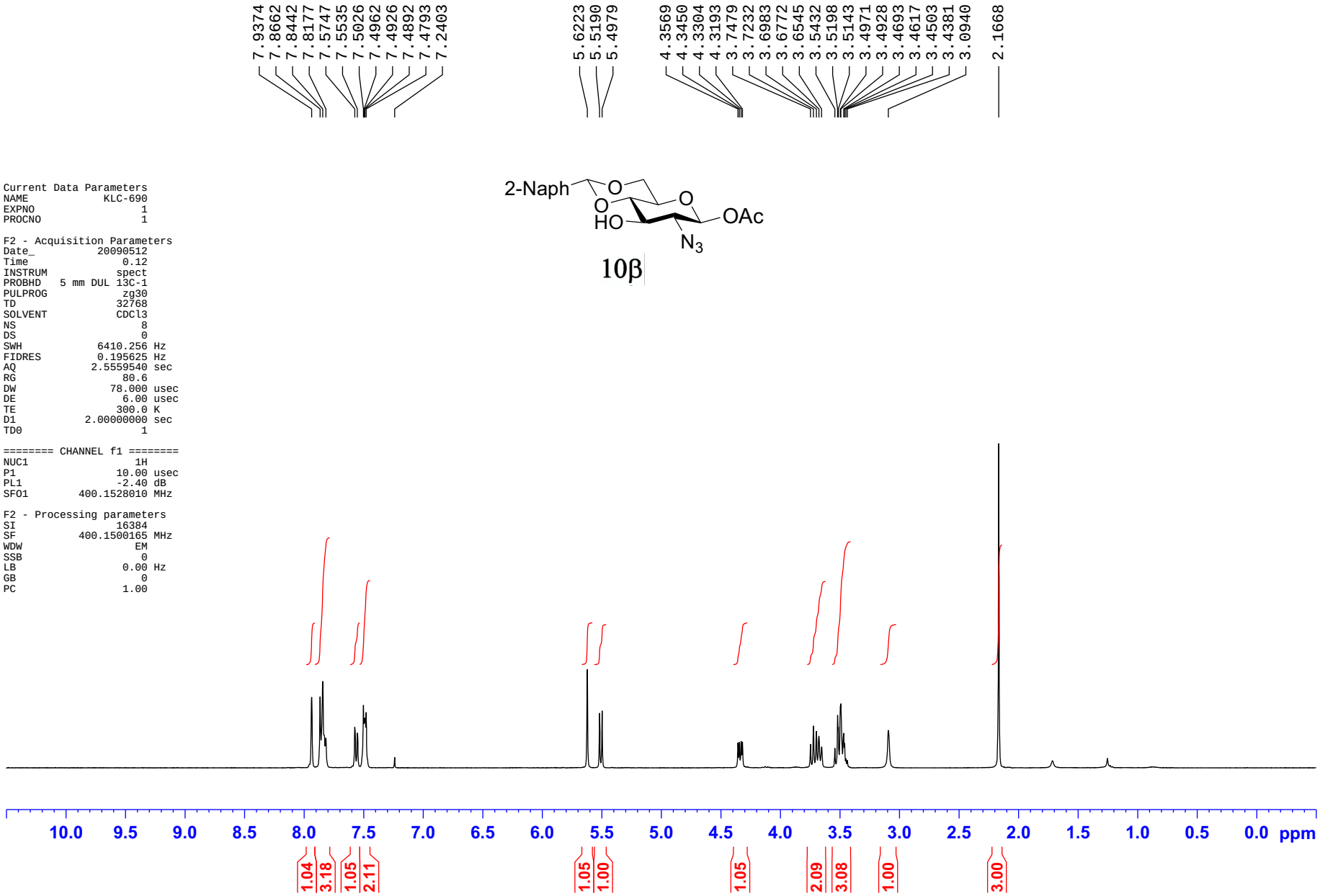
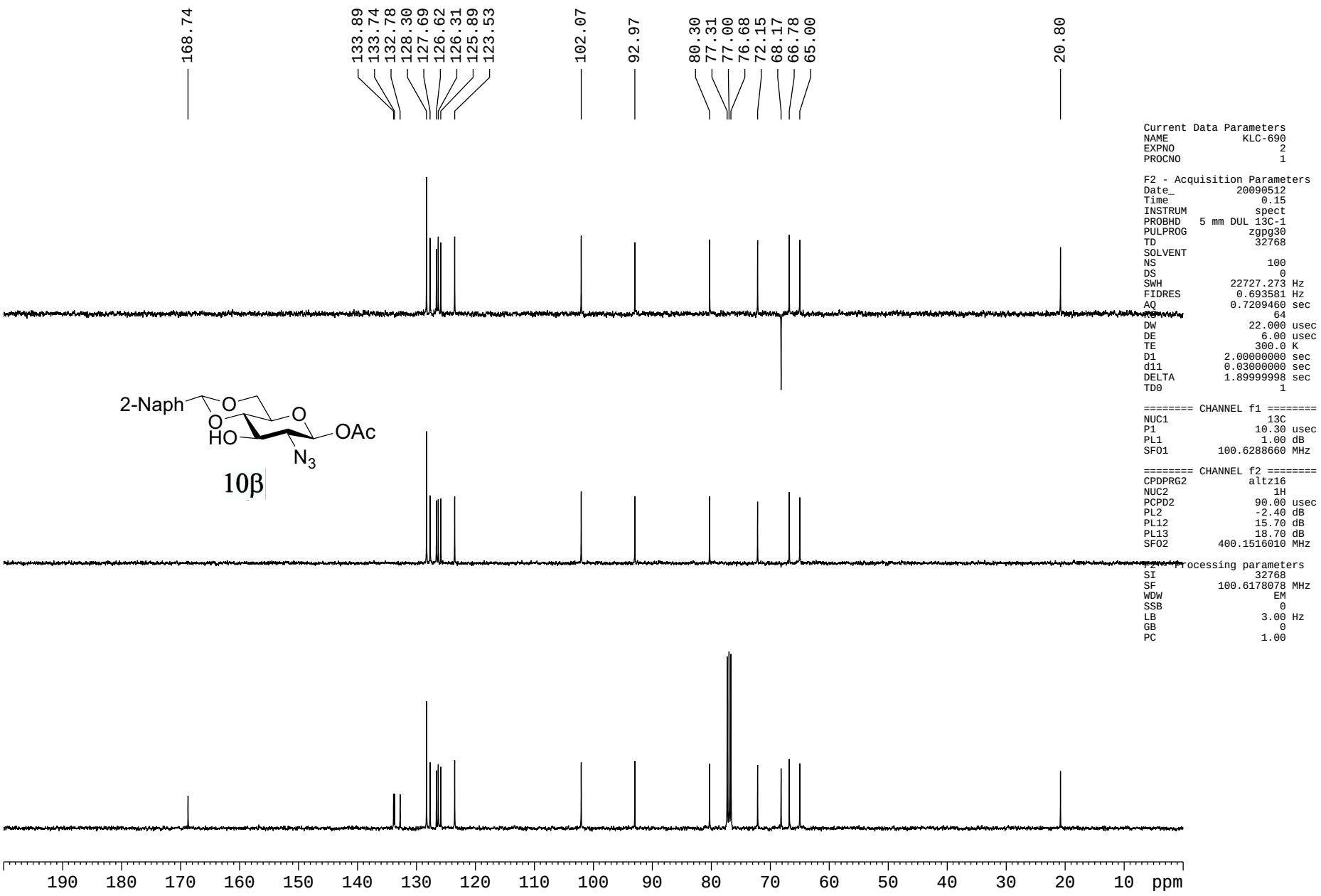
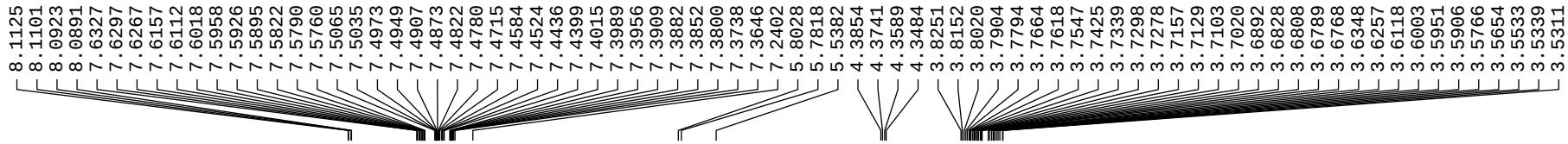
2.10
3.10
1.05
3.07

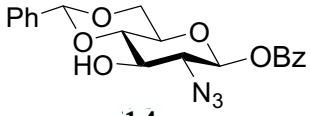

168.76
136.56
129.44
128.37
126.24
101.98
92.95
80.25
77.32
77.00
76.68
72.12
68.11
66.75
64.99
20.78
Ph
O
O
O
HO
OAc
N3
6β
Current Data Parameters
NAME KLC-693
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090512
Time 2.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 64
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178081 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.9374
7.8662
7.8442
7.8177
7.5747
7.5535
7.5026
7.4962
7.4926
7.4892
7.4793
7.2403
5.6223
5.5190
5.4979
4.3569
4.3450
4.3304
4.3193
3.7479
3.7232
3.6983
3.6772
3.6545
3.5432
3.5198
3.5143
3.4971
3.4928
3.4693
3.4617
3.4503
3.4381
3.0940
2.1668
Current Data Parameters
NAME KLC-690
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090512
Time 0.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 80.6
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500165 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2-Naph
O
O
O
HO
OAc
N3
10β
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.04
3.18
1.05
2.11
1.05
1.00
1.05
2.09
3.08
1.00
3.00

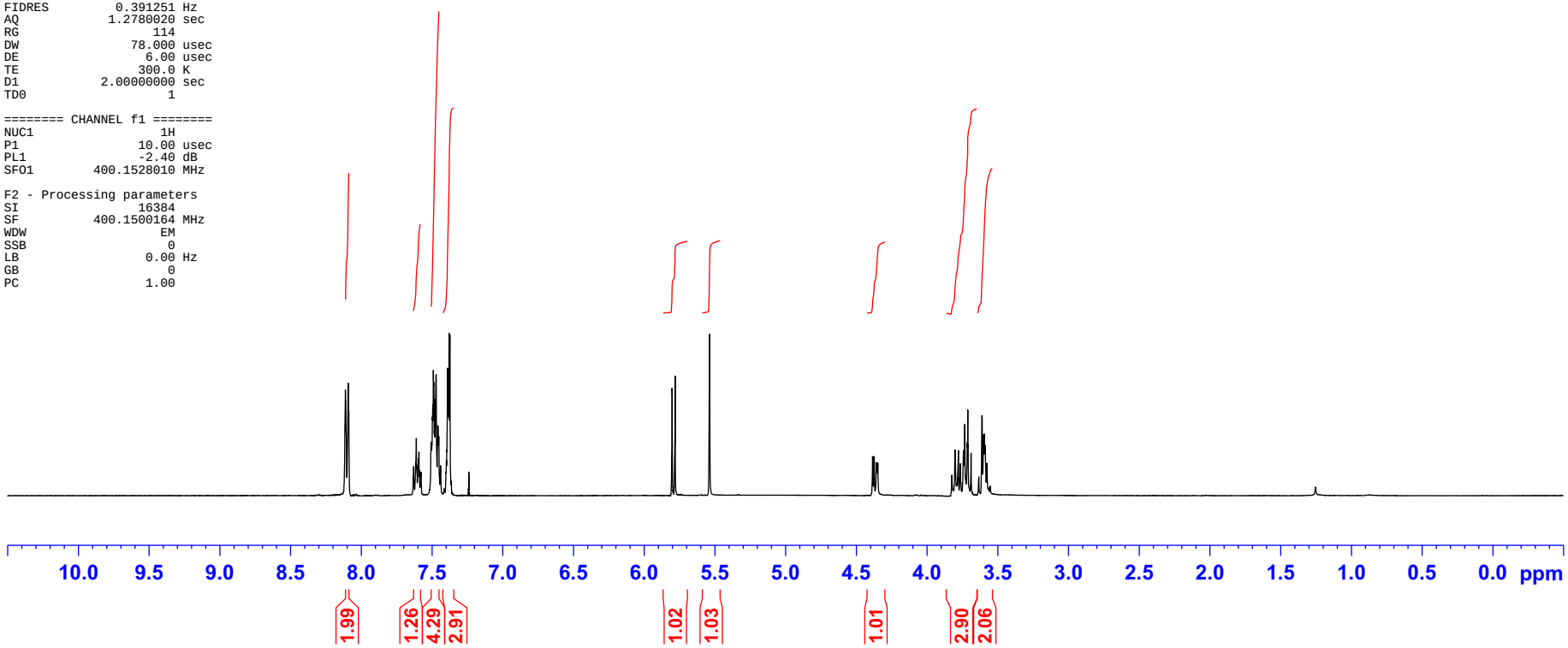

168.74
133.89
133.74
132.78
128.30
127.69
126.62
126.31
125.89
123.53
102.07
92.97
80.30
77.31
77.00
76.68
72.15
68.17
66.78
65.00
20.80
2-Naph
O
O
O
HO
OAc
N3
10β
Current Data Parameters
NAME KLC-690
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090512
Time 0.15
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 100
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178078 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

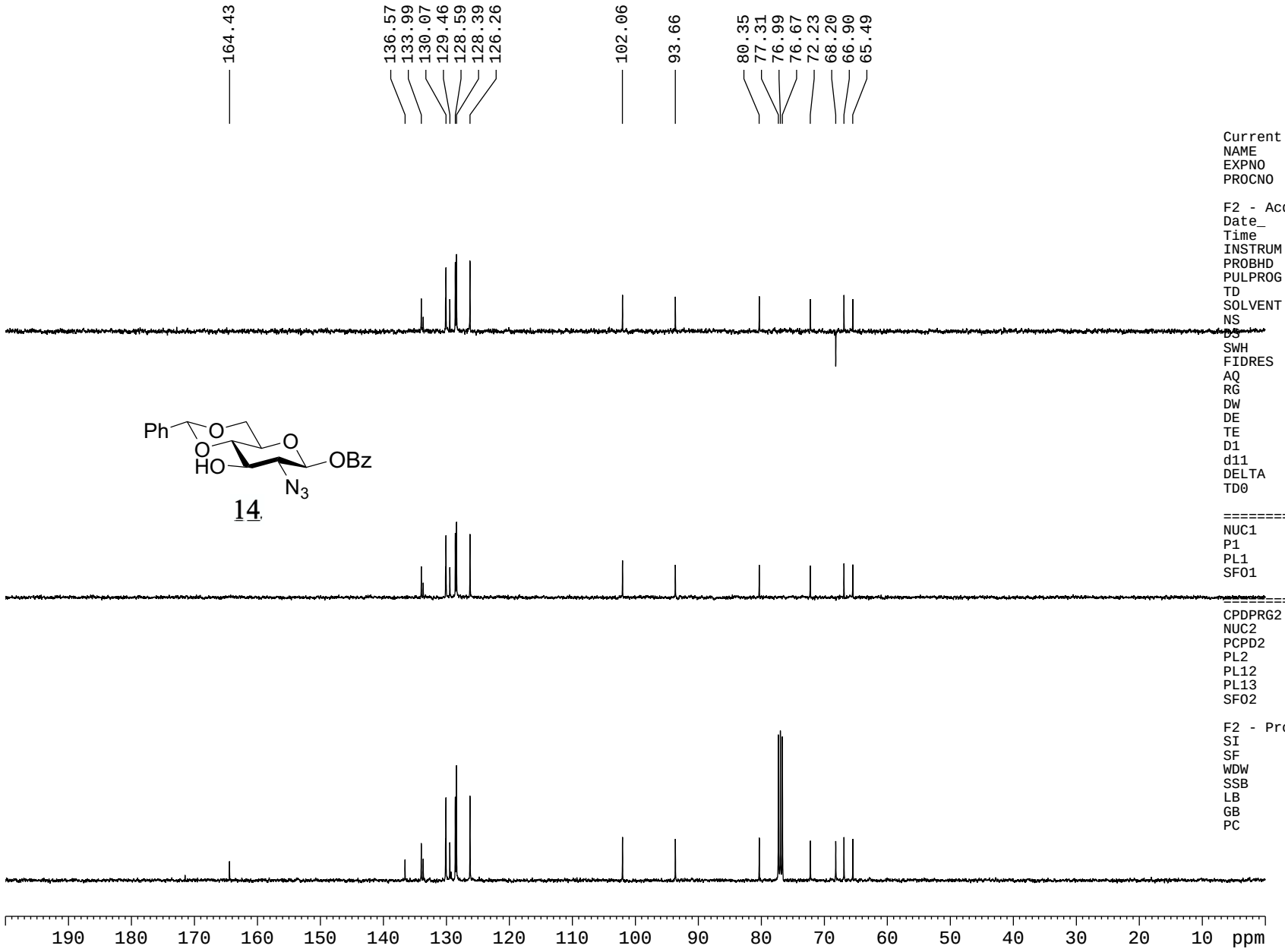


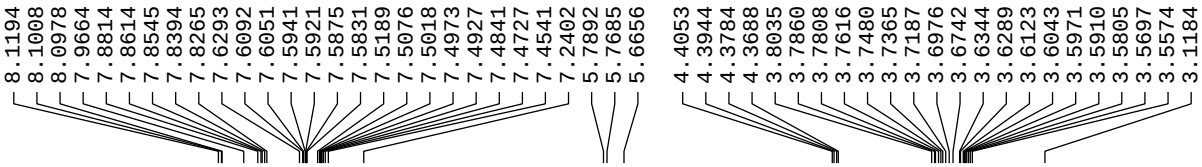

```
Current Data Parameters
NAME            KLC-695
EXPNO               100
PROCNO                1

F2 - Acquisition Parameters
Date_          20090513
Time               8.58
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                16384
SOLVENT
NS                    8
DS                    0
SWH            6410.256 Hz
FIDRES         0.391251 Hz
AQ            1.2780020 sec
RG                  114
DW                78.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500164 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

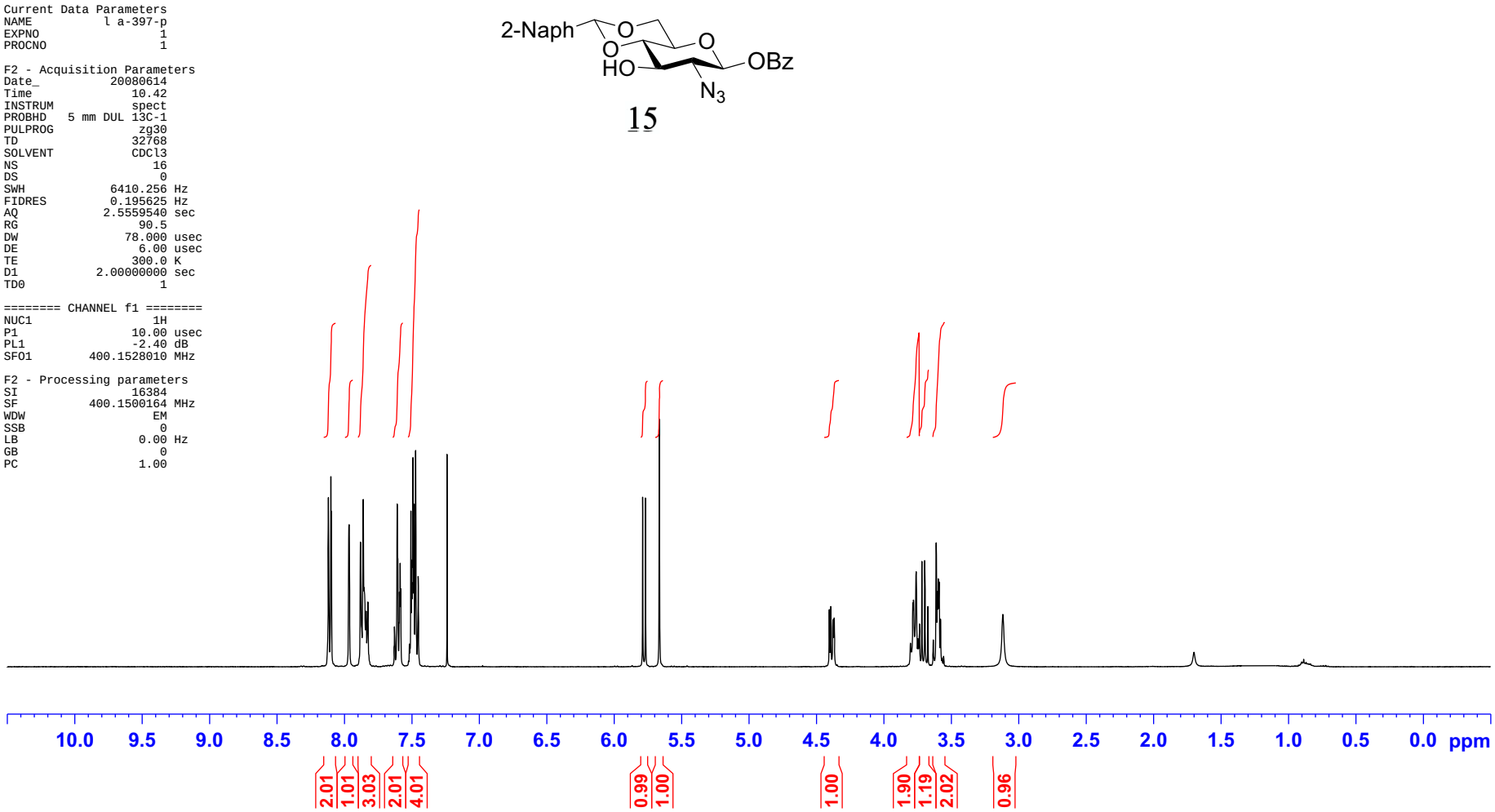


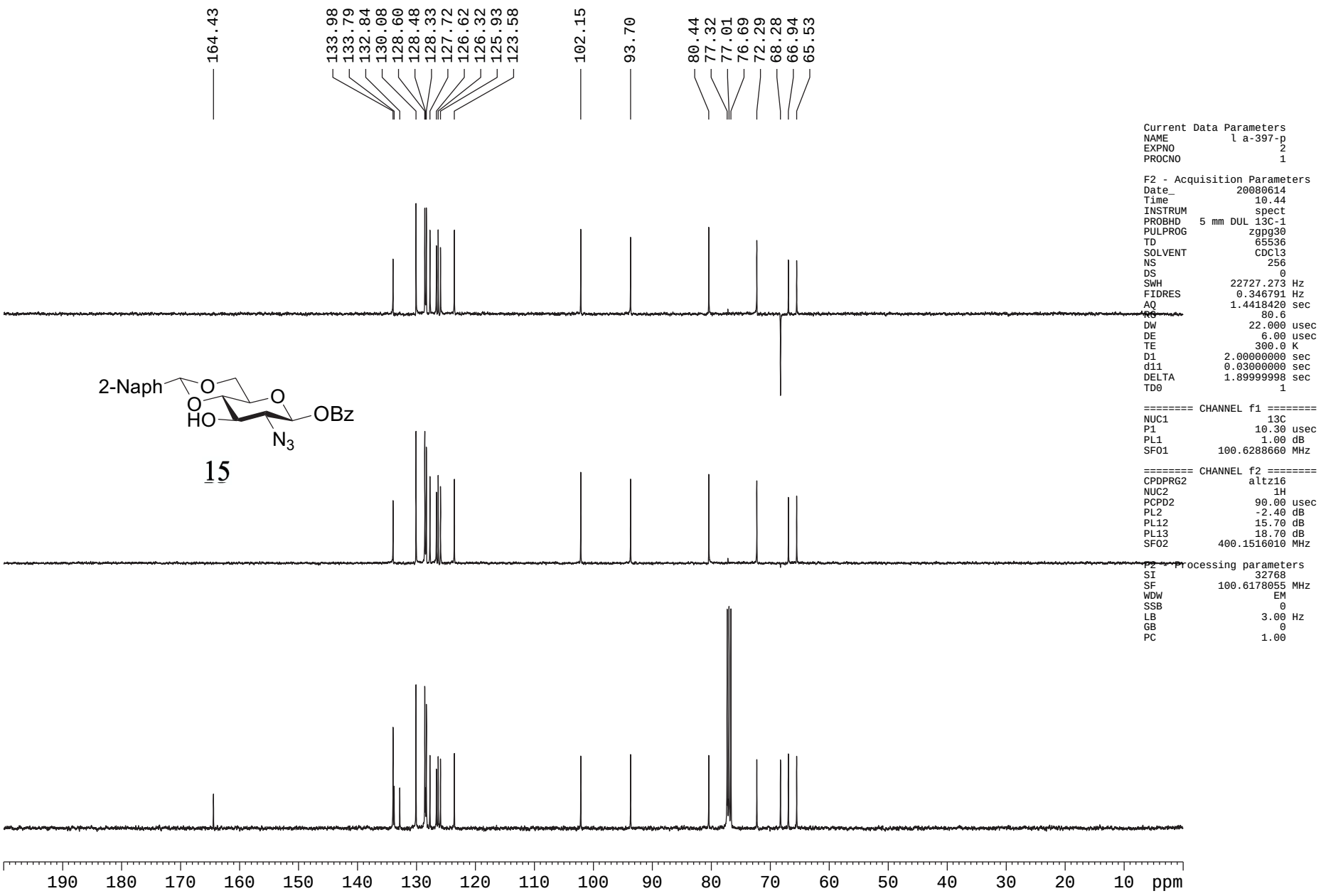
14

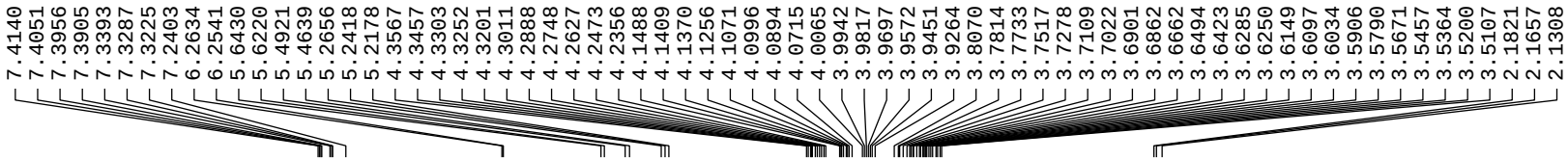



164.43
136.57
133.99
130.07
129.46
128.59
128.39
126.26
102.06
93.66
80.35
77.31
76.99
76.67
72.23
68.20
66.90
65.49
Ph
O
O
O
HO
OBz
N3
14
Current Data Parameters
NAME KLC-695
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090513
Time 8.14
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 64
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178074 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

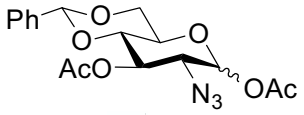

8.1194
8.1008
8.0978
7.9664
7.8814
7.8614
7.8545
7.8394
7.8265
7.6293
7.6092
7.6051
7.5941
7.5921
7.5875
7.5831
7.5189
7.5076
7.5018
7.4973
7.4927
7.4841
7.4727
7.4541
7.2402
5.7892
5.7685
5.6656
4.4053
4.3944
4.3784
4.3688
3.8035
3.7860
3.7808
3.7616
3.7480
3.7365
3.7187
3.6976
3.6742
3.6344
3.6289
3.6123
3.6043
3.5971
3.5910
3.5805
3.5697
3.5574
3.1184


Current Data Parameters
NAME l a-397-p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080614
Time 10.42
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500164 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2-Naph
O
O
O
HO
OBz
N3
15
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.01
1.01
3.03
2.01
4.01
0.99
1.00
1.00
1.90
1.19
2.02
0.96

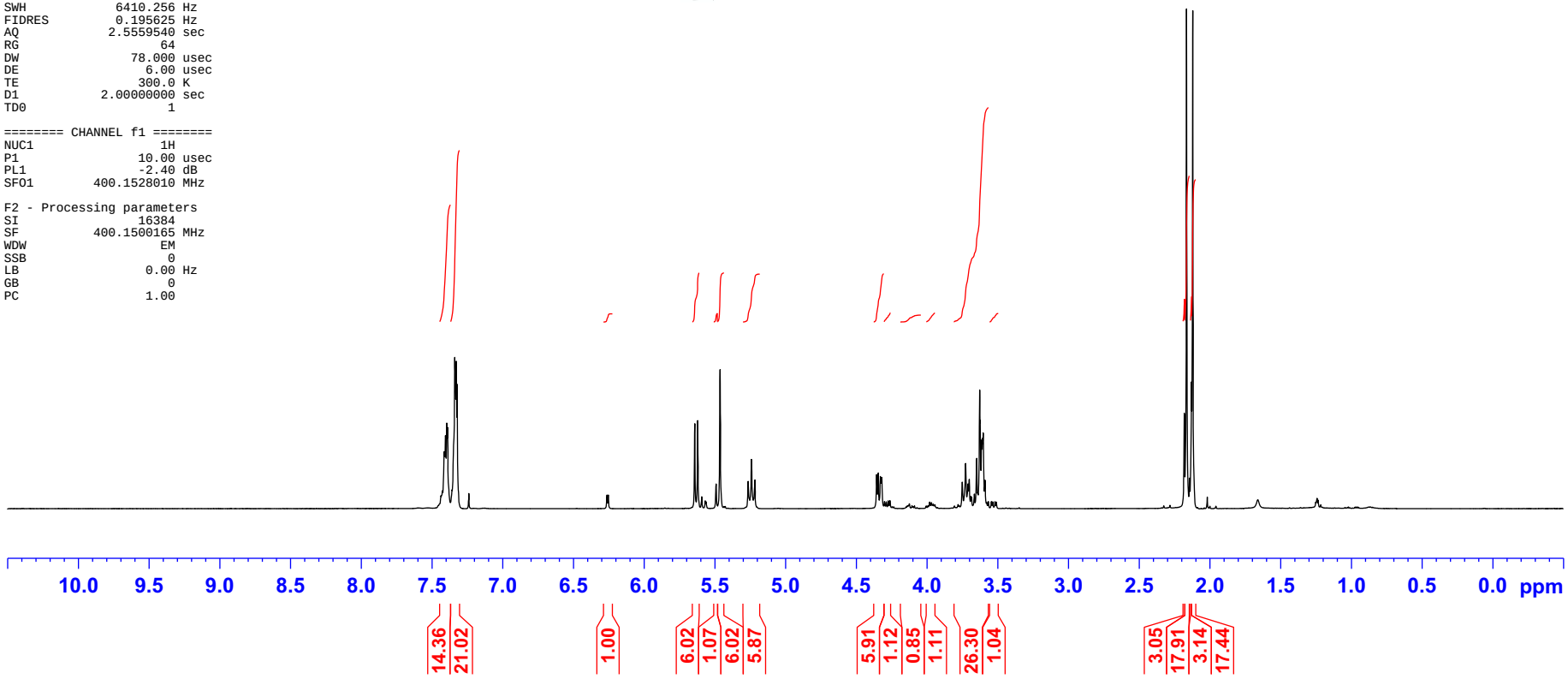

164.43
133.98
133.79
132.84
130.08
128.60
128.48
128.33
127.72
126.62
126.32
125.93
123.58
102.15
93.70
80.44
77.32
77.01
76.69
72.29
68.28
66.94
65.53
2-Naph
O
O
O
HO
OBz
N3
15
Current Data Parameters
NAME l a-397-p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080614
Time 10.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 80.6
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178055 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

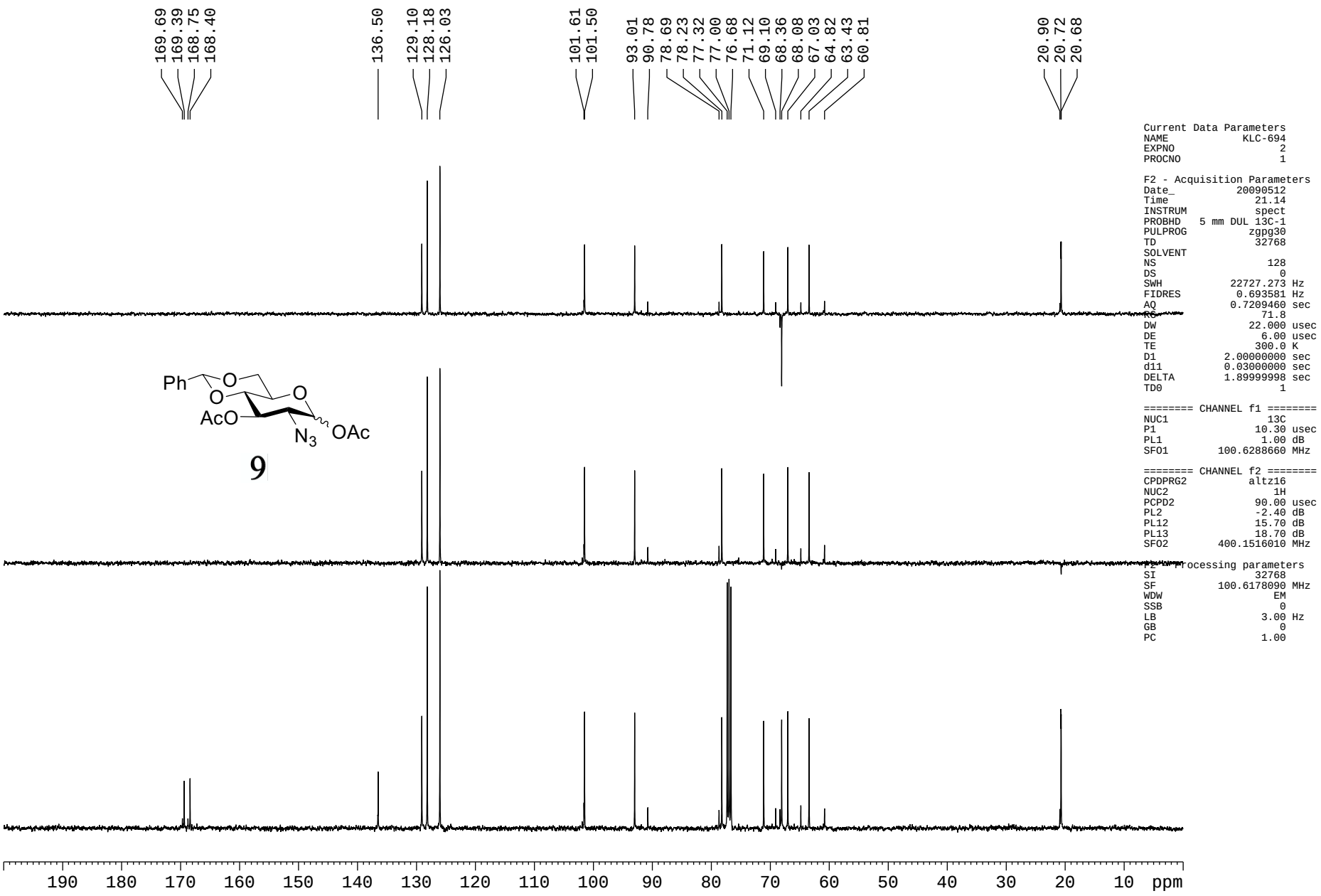


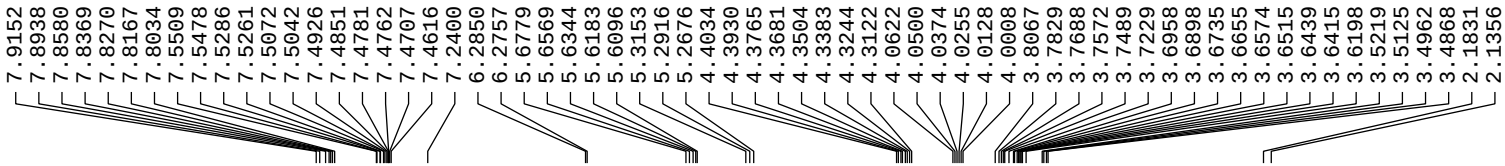

```
Current Data Parameters
NAME            KLC-694
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090512
Time              21.12
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   20
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                   64
DW               78.000 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500165 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



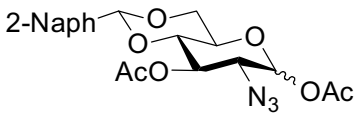
9



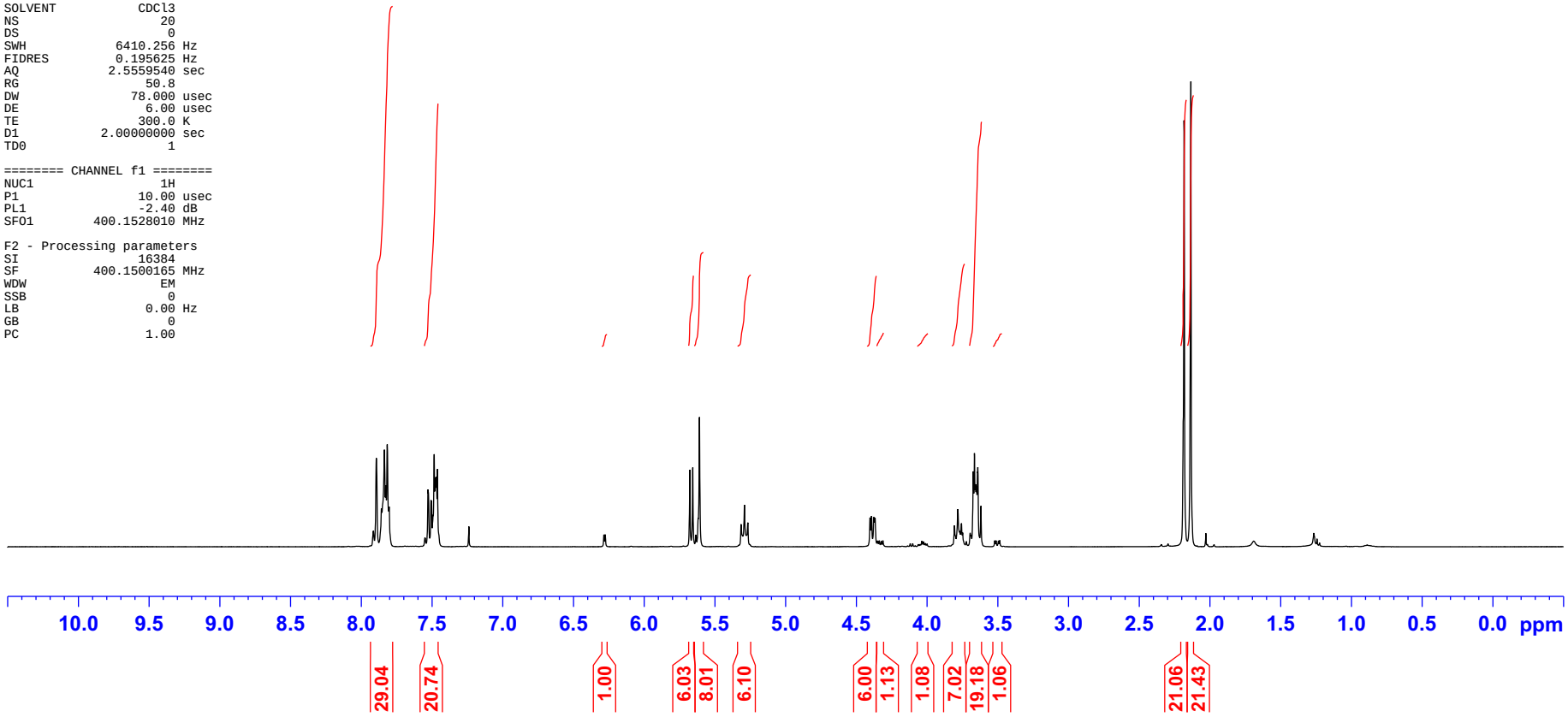

169.69
169.39
168.75
168.40
136.50
129.10
128.18
126.03
101.61
101.50
93.01
90.78
78.69
78.23
77.32
77.00
76.68
71.12
69.10
68.36
68.08
67.03
64.82
63.43
60.81
20.90
20.72
20.68
Ph
O
O
O
AcO
N3
OAc
9
Current Data Parameters
NAME KLC-694
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090512
Time 21.14
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 128
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 71.8
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178090 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

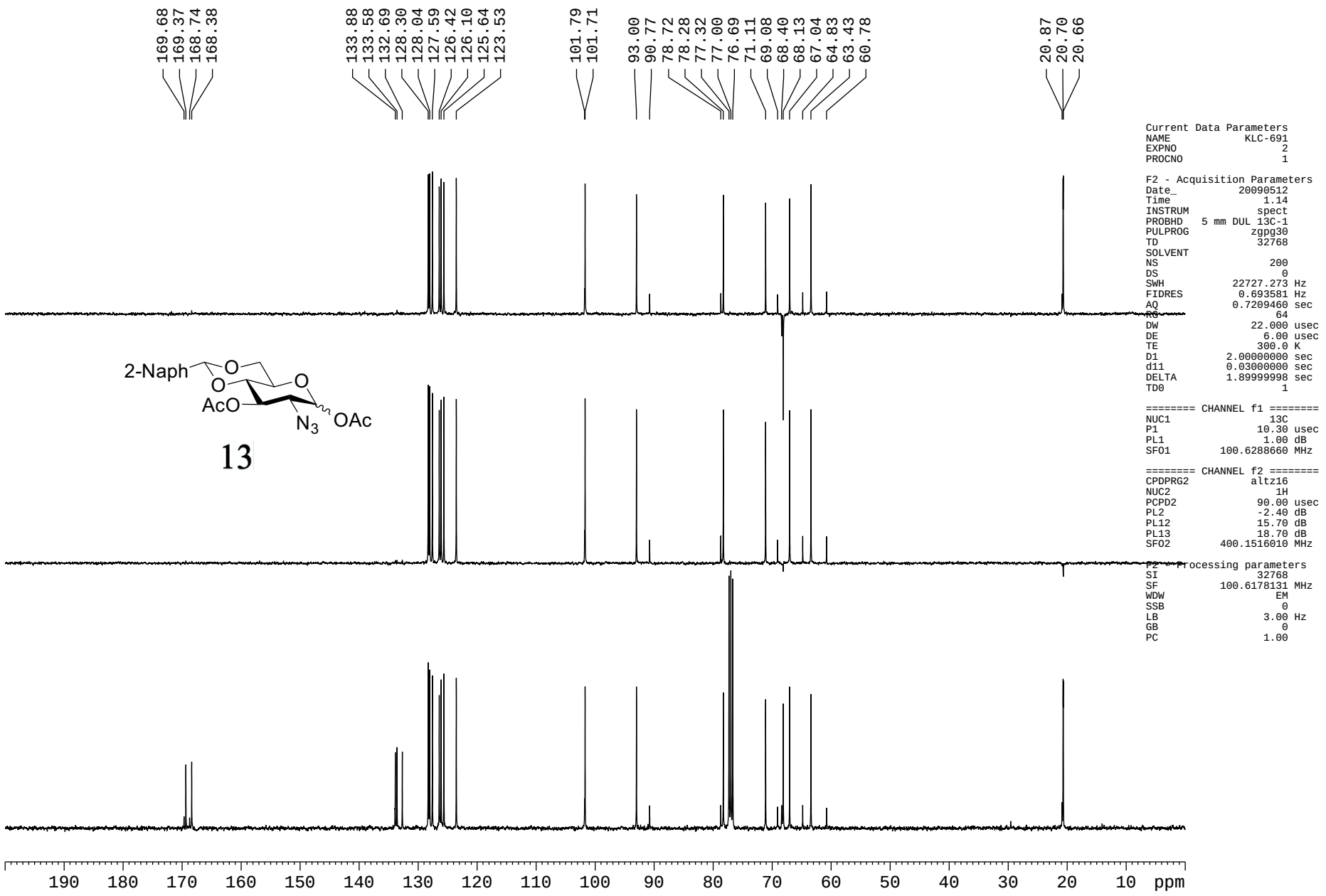


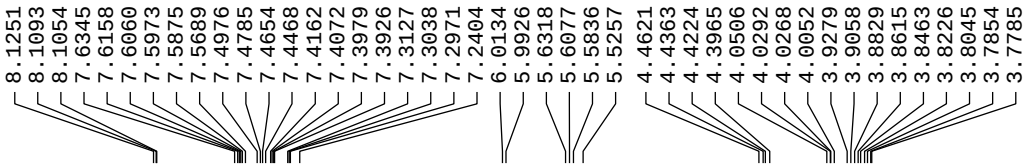

```
Current Data Parameters
NAME            KLC-691
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090512
Time               1.09
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   20
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                 50.8
DW               78.000 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500165 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

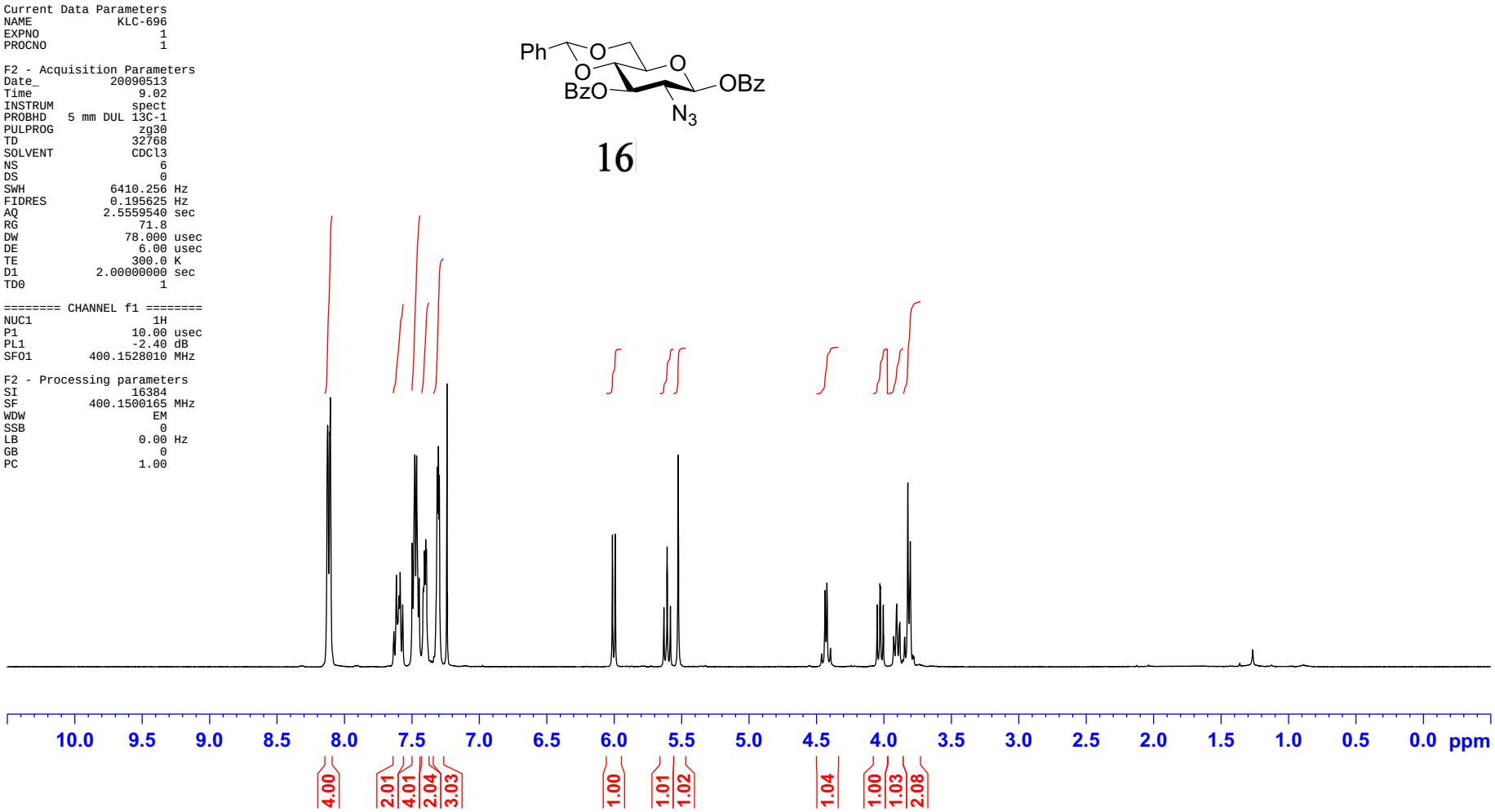


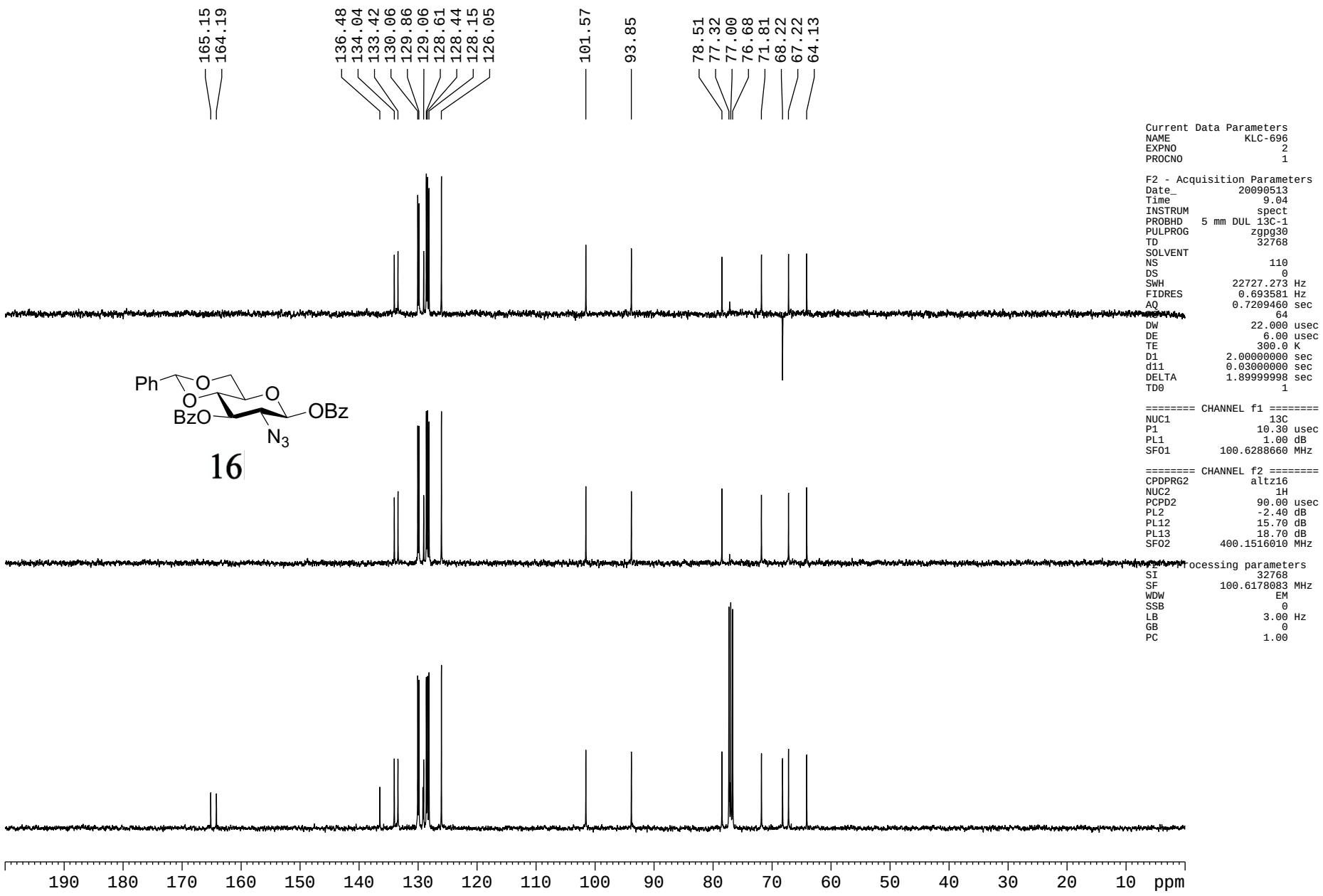
13

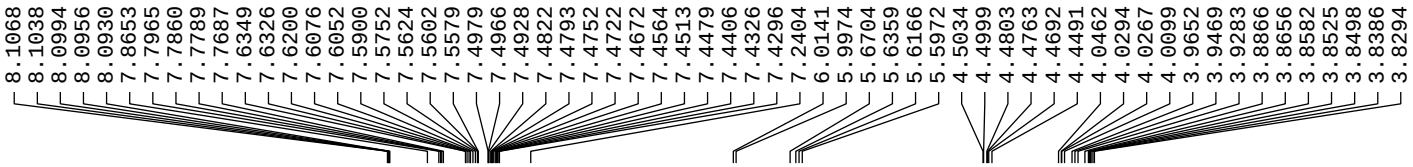



169.68
169.37
168.74
168.38
133.88
133.58
132.69
128.30
128.04
127.59
126.42
126.10
125.64
123.53
101.79
101.71
93.00
90.77
78.72
78.28
77.32
77.00
76.69
71.11
69.08
68.40
68.13
67.04
64.83
63.43
60.78
20.87
20.70
20.66
2-Naph
O
O
O
AcO
N3
OAc
13
Current Data Parameters
NAME KLC-691
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090512
Time 1.14
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 200
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178131 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

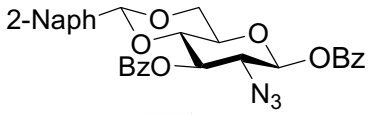

8.1251
8.1093
8.1054
7.6345
7.6158
7.6060
7.5973
7.5875
7.5689
7.4976
7.4785
7.4654
7.4468
7.4162
7.4072
7.3979
7.3926
7.3127
7.3038
7.2971
7.2404
6.0134
5.9926
5.6318
5.6077
5.5836
5.5257
4.4621
4.4363
4.4224
4.3965
4.0506
4.0292
4.0268
4.0052
3.9279
3.9058
3.8829
3.8615
3.8463
3.8226
3.8045
3.7854
3.7785


Current Data Parameters
NAME KLC-696
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090513
Time 9.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 71.8
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500165 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Ph
O
O
O
BzO
OBz
N3
16
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.00
2.01
4.01
2.04
3.03
1.00
1.01
1.02
1.04
1.00
1.03
2.08

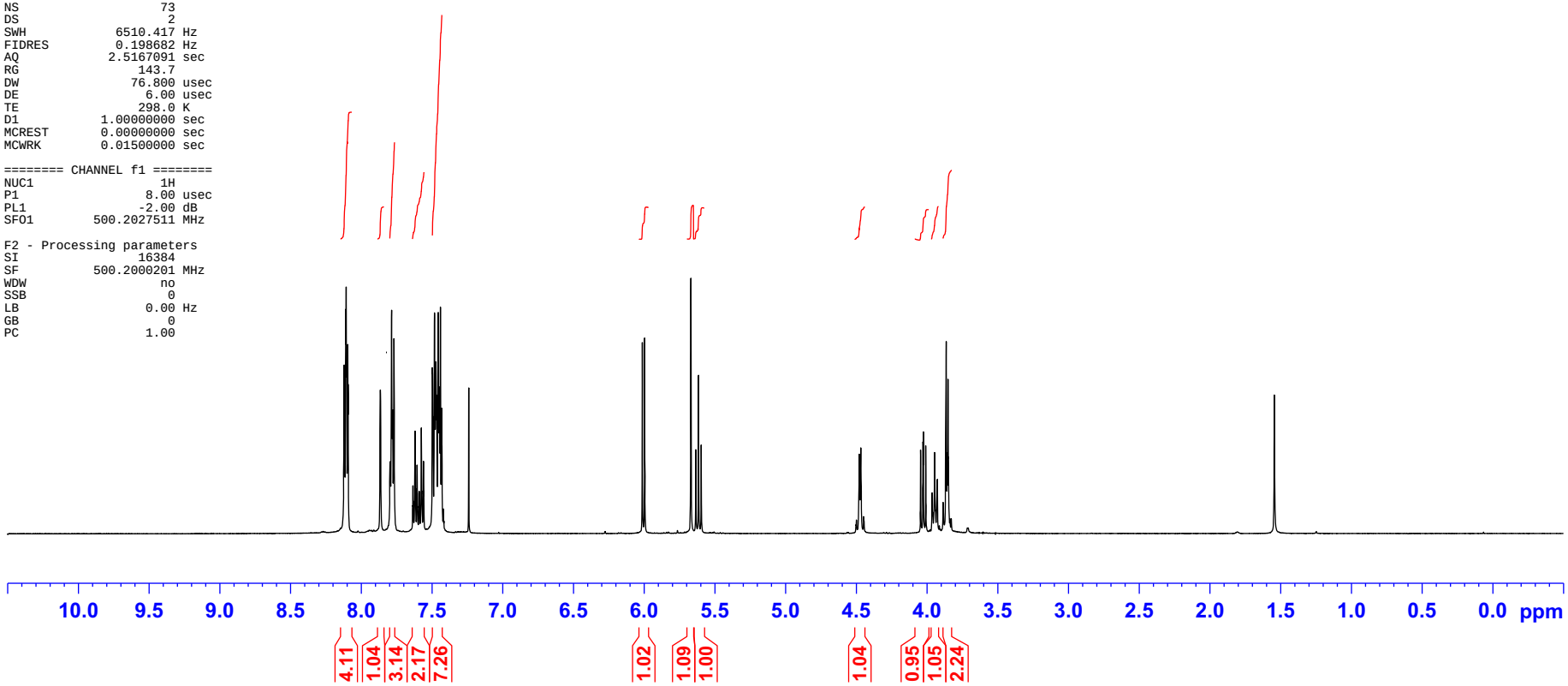

165.15
164.19
136.48
134.04
133.42
130.06
129.86
129.06
128.61
128.44
128.15
126.05
101.57
93.85
78.51
77.32
77.00
76.68
71.81
68.22
67.22
64.13
Ph
O
O
O
BzO
OBz
N3
16
Current Data Parameters
NAME KLC-696
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090513
Time 9.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 110
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178083 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

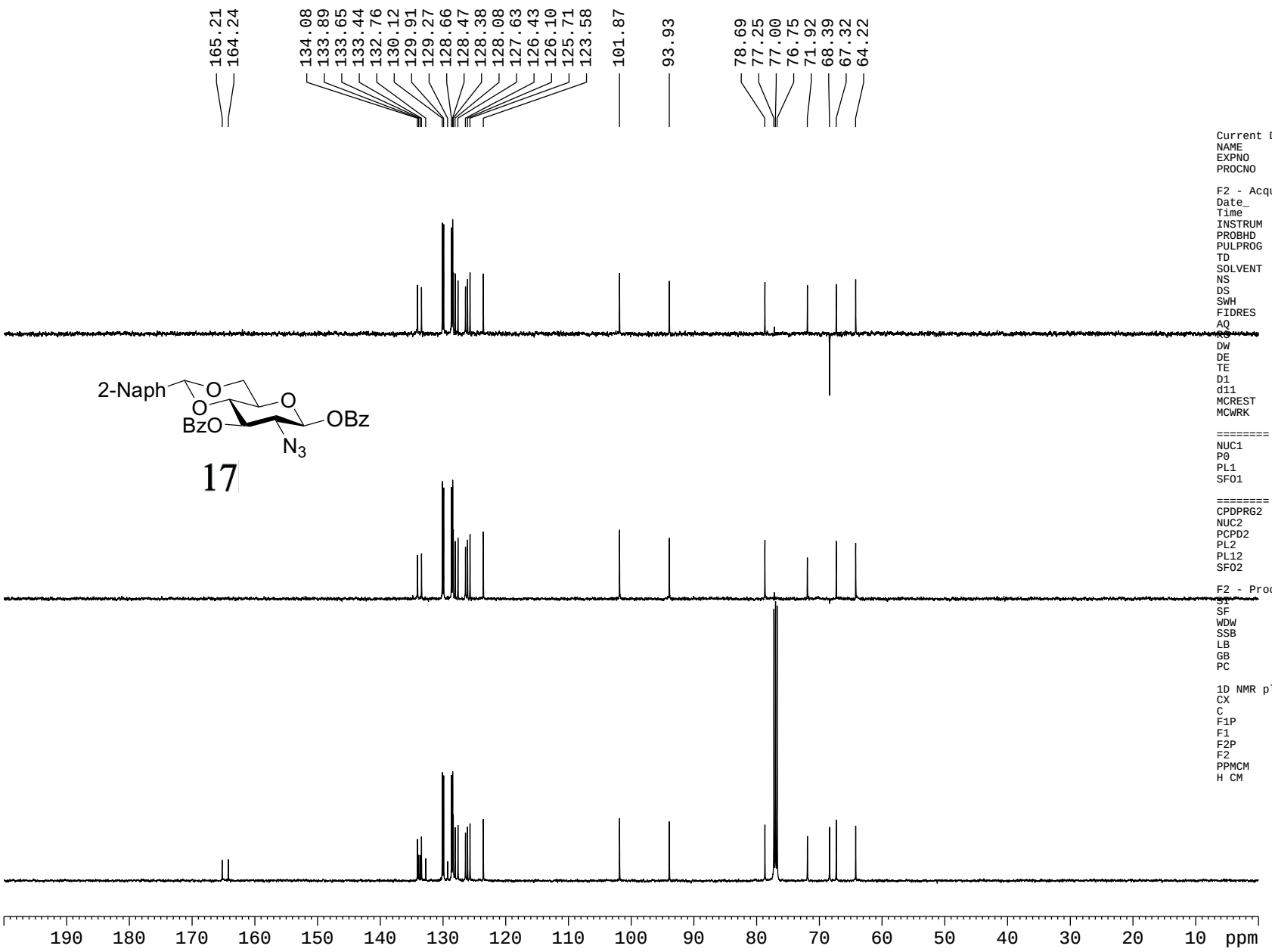


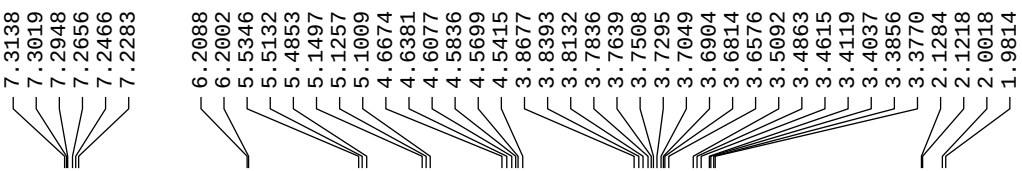

```
Current Data Parameters
NAME      CRS-047-Pa 500
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20031205
Time              16.57
INSTRUM           spect
PROBHD   5 mm QNP 1H 15
PULPROG             zg
TD                32768
SOLVENT           CDCl3
NS                   73
DS                    2
SWH            6510.417 Hz
FIDRES         0.198682 Hz
AQ            2.5167091 sec
RG                143.7
DW               76.800 usec
DE                6.00 usec
TE                298.0 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                8.00 usec
PL1              -2.00 dB
SFO1        500.2027511 MHz

F2 - Processing parameters
SI                16384
SF          500.2000201 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



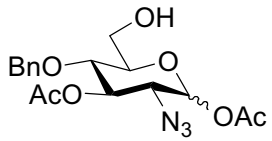
17



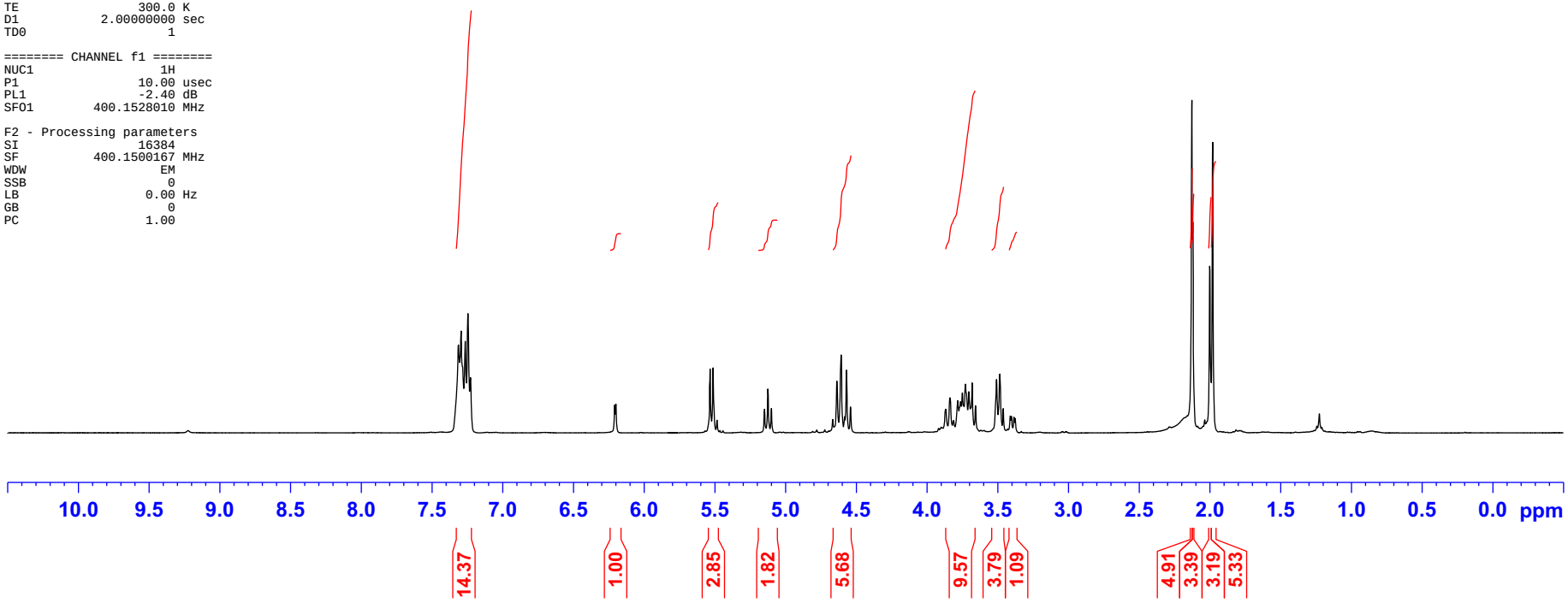

165.21
164.24
134.08
133.89
133.65
133.44
132.76
130.12
129.91
129.27
128.66
128.47
128.38
128.08
127.63
126.43
126.10
125.71
123.58
101.87
93.93
78.69
77.25
77.00
76.75
71.92
68.39
67.32
64.22
2-Naph
O
O
O
BzO
OBz
N3
17
Current Data Parameters
NAME CRS-047-Pa 500
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20031205
Time 18.04
INSTRUM spect
PROBHD 5 mm QNP 1H 15
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 500
DS 2
SWH 34013.605 Hz
FIDRES 1.038013 Hz
AQ 0.4817396 sec
RG 2048
DW 14.700 usec
DE 6.00 usec
TE 298.0 K
D1 3.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P0 8.00 usec
PL1 3.00 dB
SFO1 125.7898571 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 14.89 dB
SFO2 500.2020008 MHz
F2 - Processing parameters
SI 32768
SF 125.7753944 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 24.00 cm
C 25.00 cm
F1P 135.121 ppm
F1 16994.87 Hz
F2P 122.558 ppm
F2 15414.82 Hz
PPMCM 0.52344 ppm cm
H CM 65.83559 Hz cm
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

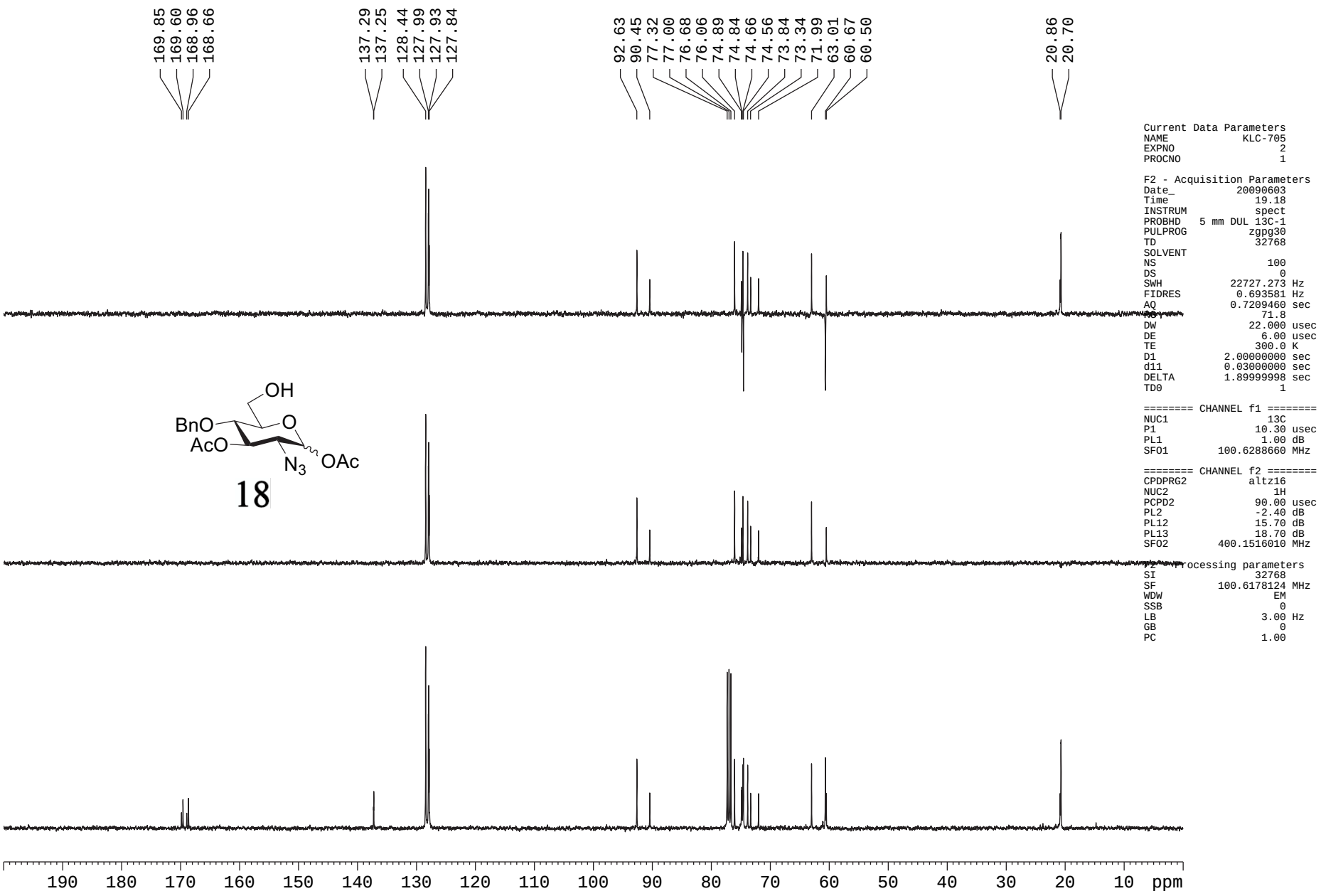


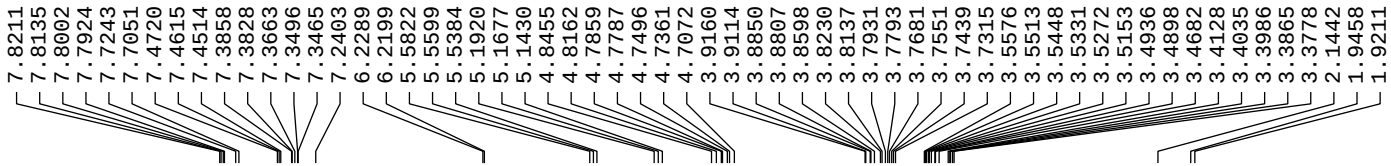

```
Current Data Parameters
NAME            KLC-705
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090603
Time              19.14
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   12
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                 50.8
DW               78.000 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500167 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

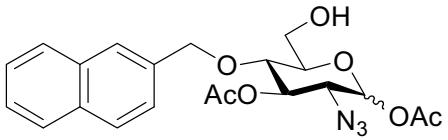


18



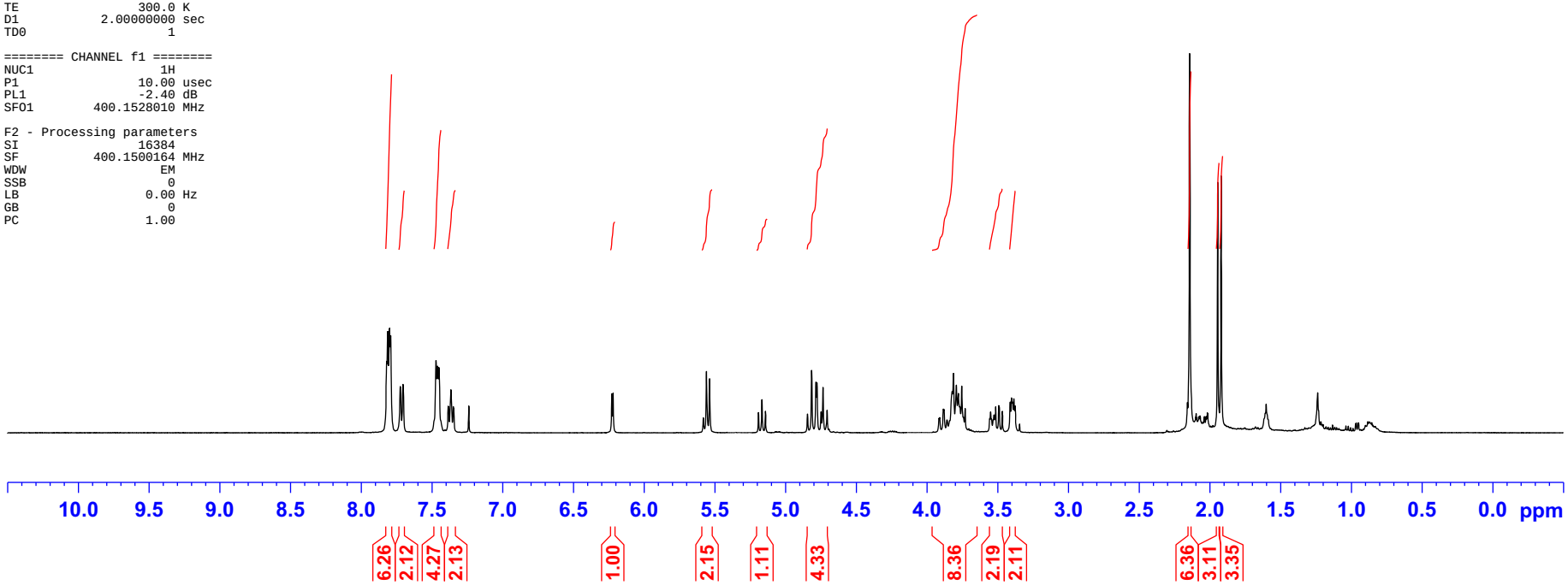

169.85
169.60
168.96
168.66
137.29
137.25
128.44
127.99
127.93
127.84
92.63
90.45
77.32
77.00
76.68
76.06
74.89
74.84
74.66
74.56
73.84
73.34
71.99
63.01
60.67
60.50
20.86
20.70
OH
BnO
O
AcO
N3
OAc
18
Current Data Parameters
NAME KLC-705
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090603
Time 19.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 100
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 71.8
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178124 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

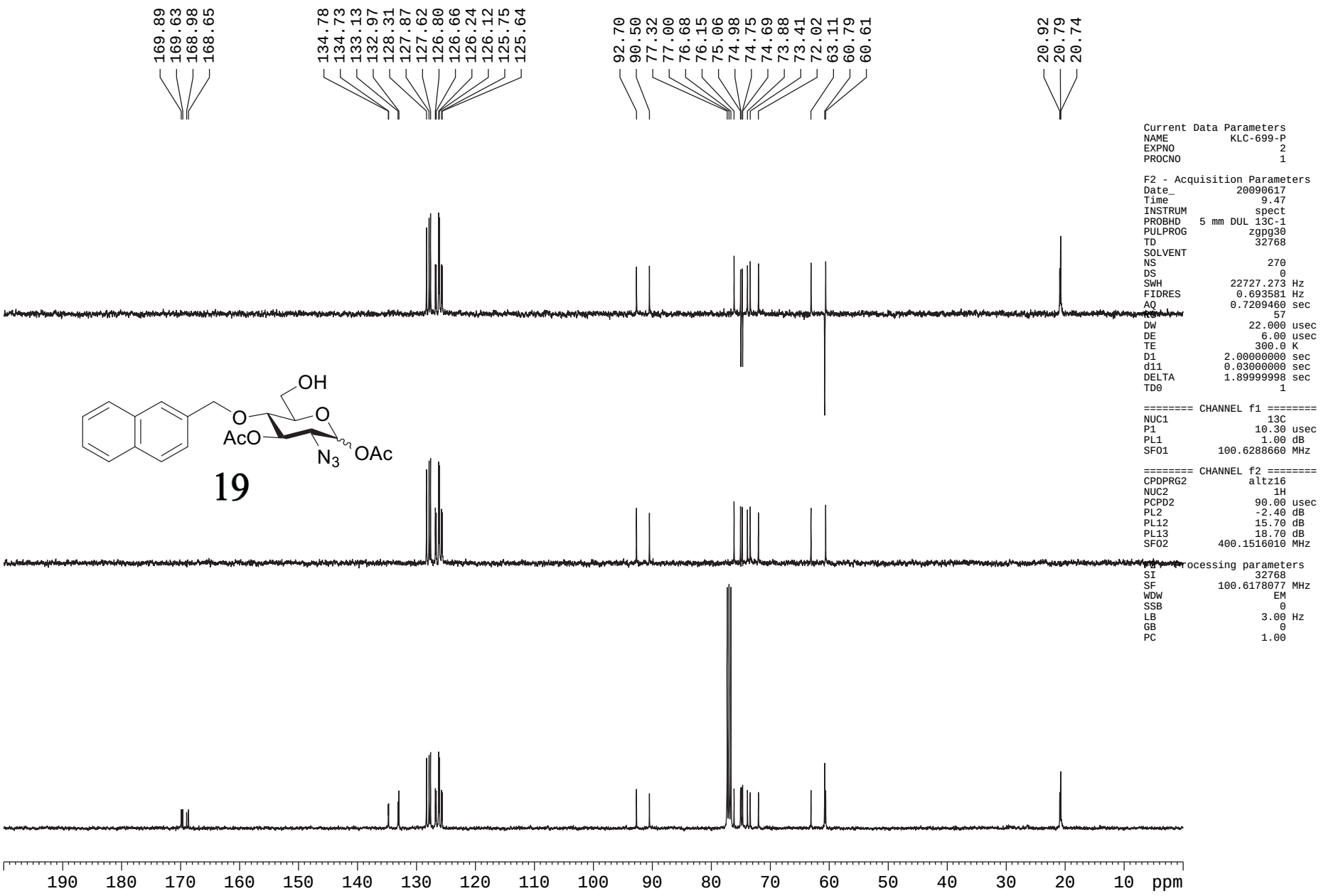


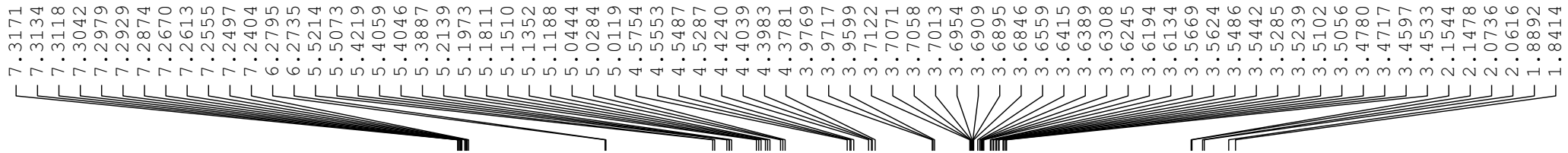


19

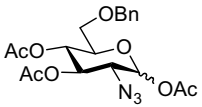
```
Current Data Parameters
NAME          KLC-699-P
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090617
Time               9.07
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   12
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                   64
DW               78.000 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500164 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```




169.89
169.63
168.98
168.65
134.78
134.73
133.13
132.97
128.31
127.87
127.62
126.80
126.66
126.24
126.12
125.75
125.64
92.70
90.50
77.32
77.00
76.68
76.15
75.06
74.98
74.75
74.69
73.88
73.41
72.02
63.11
60.79
60.61
20.92
20.79
20.74
OH
O
O
AcO
N3
OAc
19
Current Data Parameters
NAME KLC-699-P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090617
Time 9.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 270
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178077 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm




```
NAME            KLC-850
EXPNO                 1
PROCNO                1
Date_          20091203
Time              17.35
INSTRUM           spect
PROBHD   5 mm CPDCH 13C
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   10
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ            1.9530824 sec
RG                 20.2
DW                59.600 usec
DE                 6.00 usec
TE                298.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                9.45 usec
PL1               -0.70 dB
PL1W        17.70725441 W
SFO1        600.1536010 MHz
SI                16384
SF          600.1500279 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



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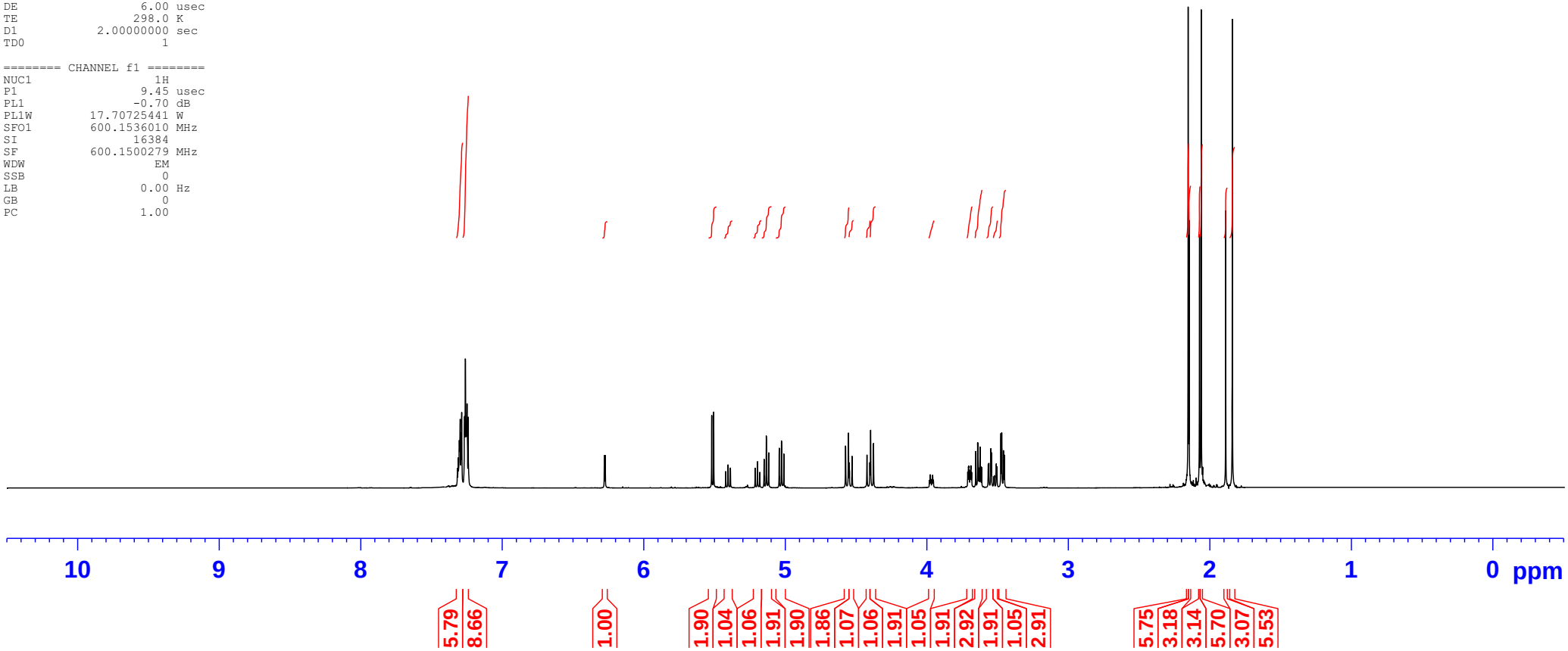
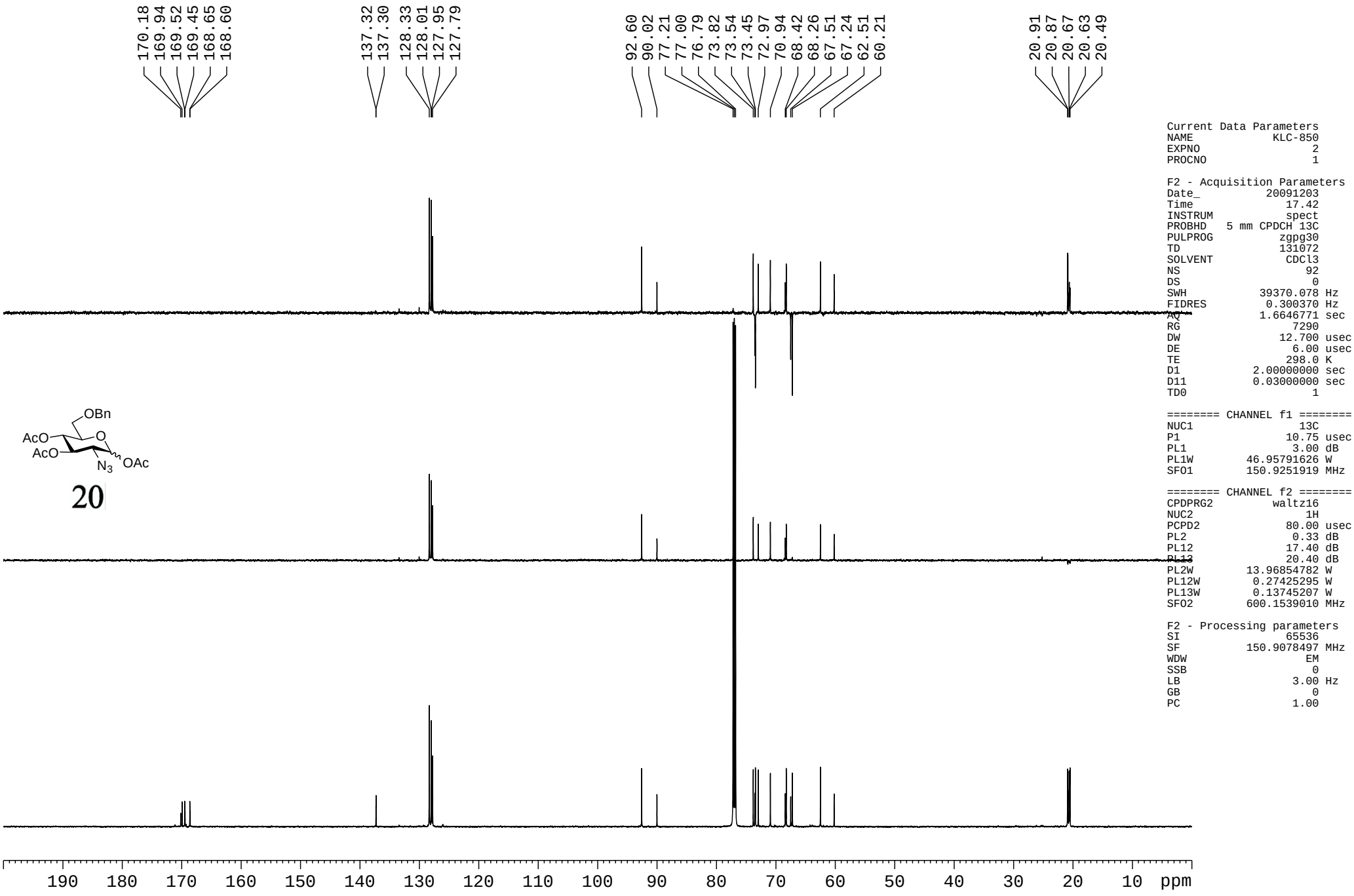
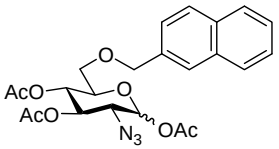


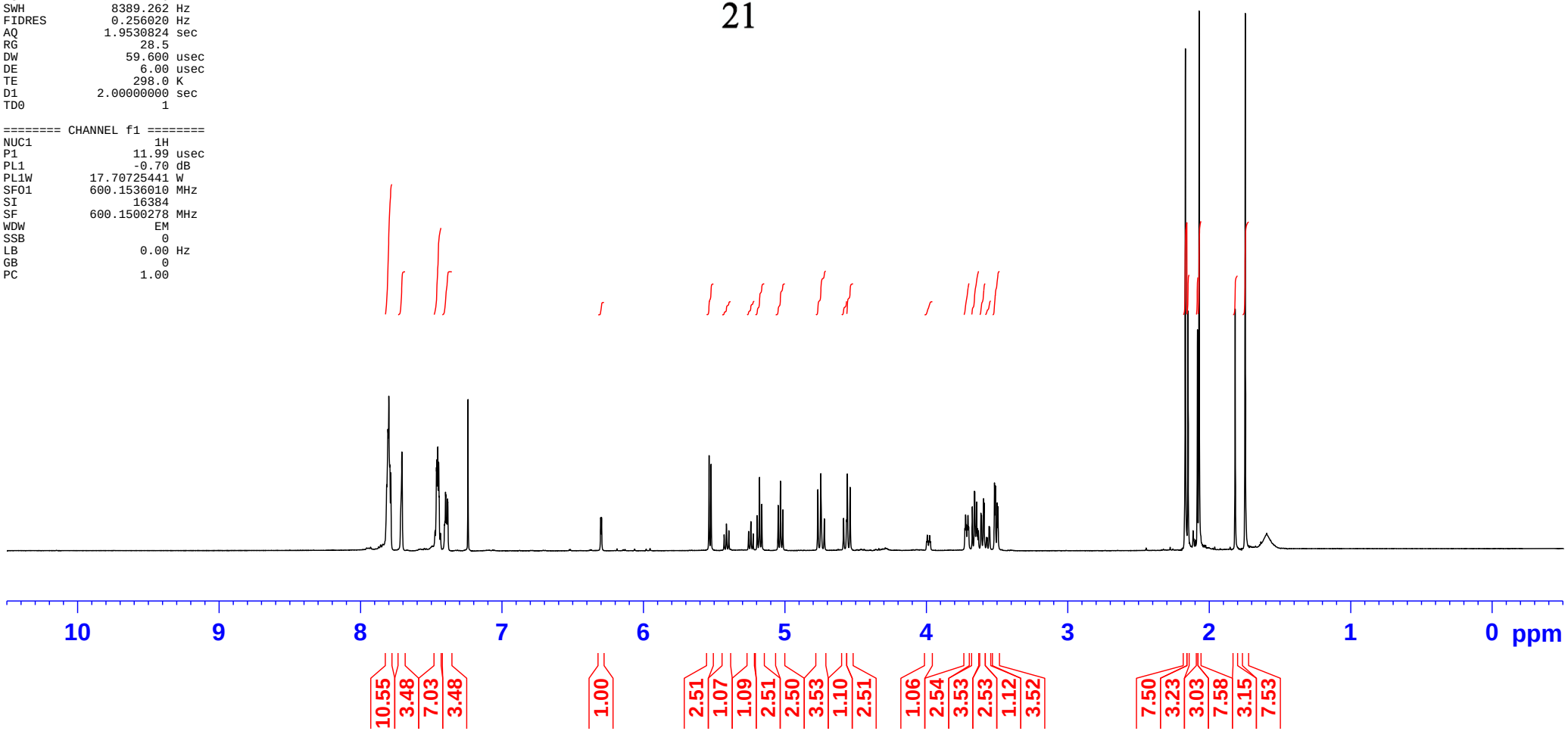

170.18
169.94
169.52
169.45
168.65
168.60
137.32
137.30
128.33
128.01
127.95
127.79
92.60
90.02
77.21
77.00
76.79
73.82
73.54
73.45
72.97
70.94
68.42
68.26
67.51
67.24
62.51
60.21
20.91
20.87
20.67
20.63
20.49
OBn
AcO
O
AcO
N3
OAc
20
Current Data Parameters
NAME KLC-850
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091203
Time 17.42
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 92
DS 0
SWH 39370.078 Hz
FIDRES 0.300370 Hz
AQ 1.6646771 sec
RG 7290
DW 12.700 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.75 usec
PL1 3.00 dB
PL1W 46.95791626 W
SFO1 150.9251919 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.33 dB
PL12 17.40 dB
PL13 20.40 dB
PL2W 13.96854782 W
PL12W 0.27425295 W
PL13W 0.13745207 W
SFO2 600.1539010 MHz
F2 - Processing parameters
SI 65536
SF 150.9078497 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

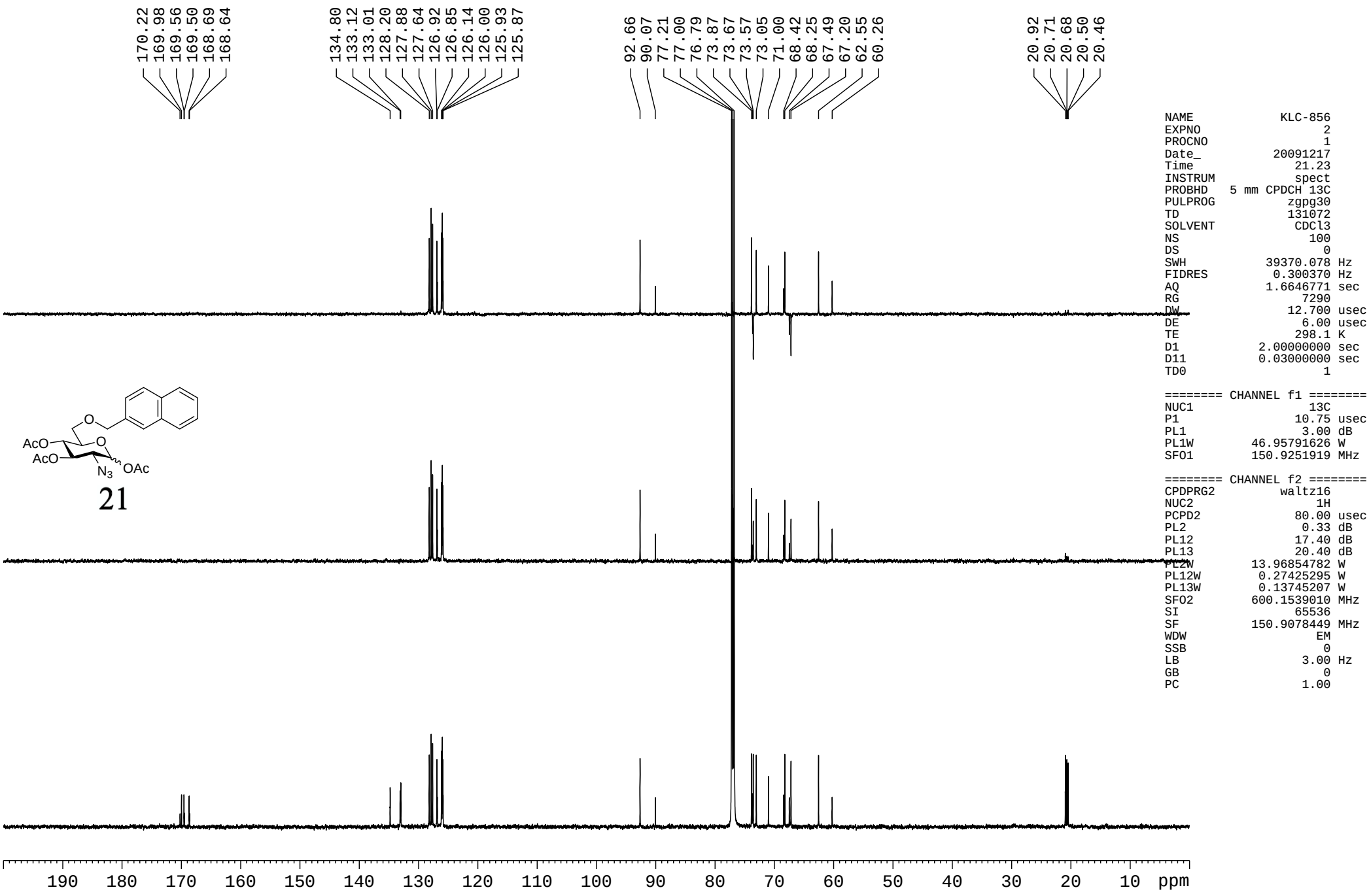
7.8160 7.8076 7.8009 7.7938 7.7870 7.7090 7.4749 7.4726 7.4637 7.4604 7.4559 7.4542 7.4483 7.4448 7.4067 7.4043 7.3998 7.3977 7.3934 7.3906 7.3860 7.3841 7.2405 6.3025 6.2966 5.5368 5.5225 5.4137 5.3966 5.2578 5.2414 5.1959 5.1798 5.1637 5.0482 5.0320 5.0156 4.7671 4.7466 4.7216 4.5859 4.5650 4.5595 4.5391 3.7290 3.7241 3.7187 3.7124 3.7070 3.7022 3.6765 3.6620 3.6596 3.6504 3.6452 3.6392 3.6331 3.6152 3.6111 3.5969 3.5928 3.5574 3.5531 3.5195 3.5133 3.5011 3.4950 2.1703 2.1513 2.0850 2.0726 1.8177 1.7470

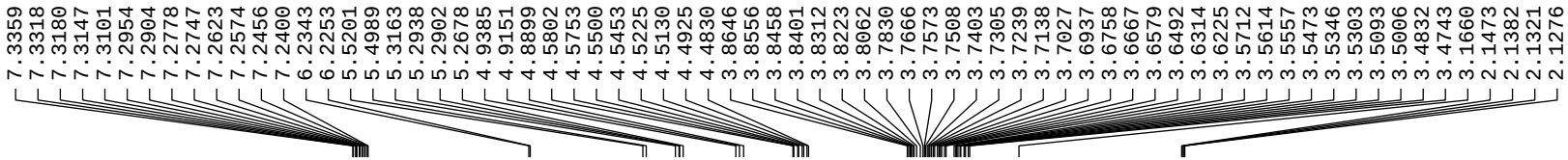
```
NAME            KLC-856
EXPNO                 1
PROCNO                1
Date_          20091217
Time              21.21
INSTRUM           spect
PROBHD   5 mm CPDCH 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     9
DS                     0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ            1.9530824 sec
RG                  28.5
DW                59.600 usec
DE                 6.00 usec
TE                298.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.99 usec
PL1                -0.70 dB
PL1W        17.70725441 W
SFO1        600.1536010 MHz
SI                 16384
SF          600.1500278 MHz
WDW                   EM
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```



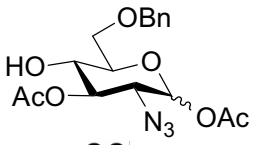
21



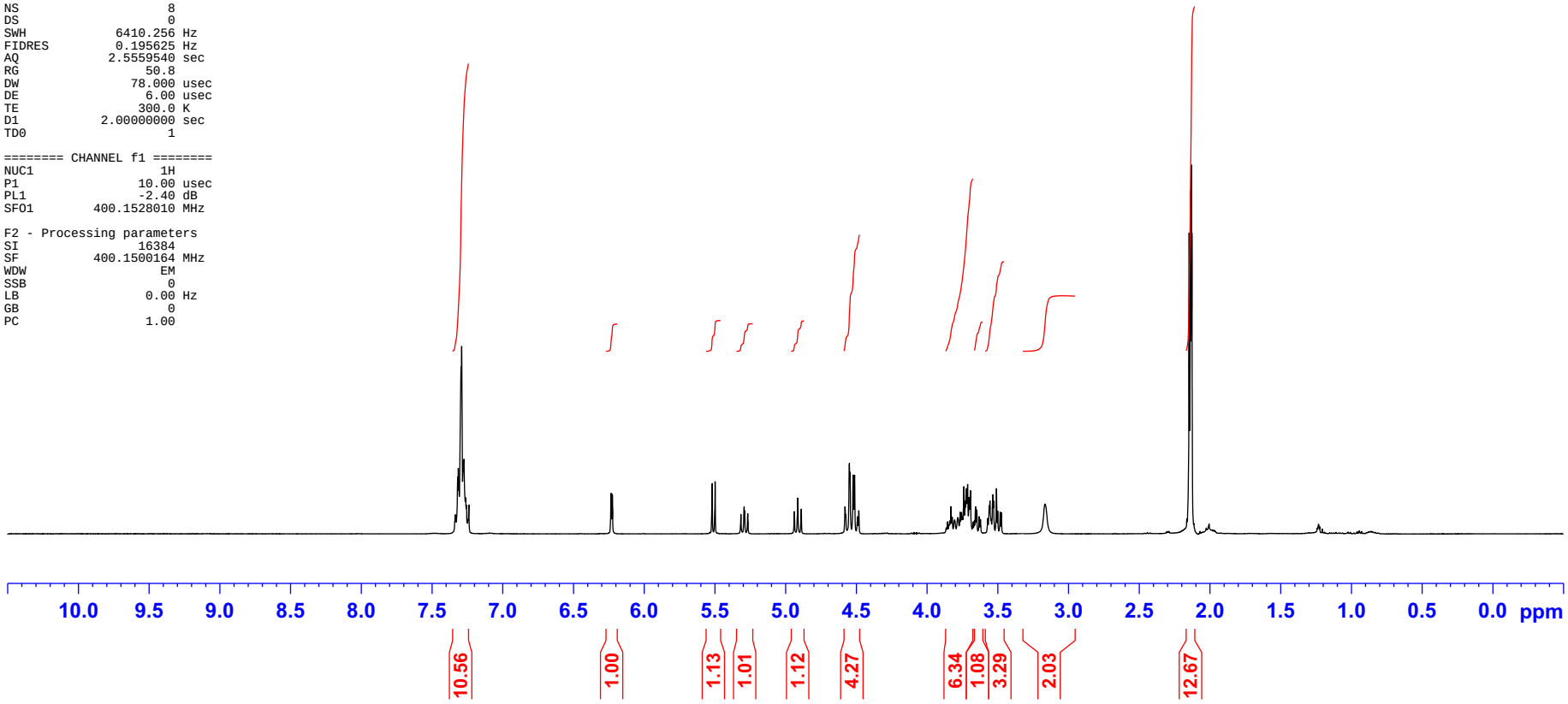

170.22
169.98
169.56
169.50
168.69
168.64
134.80
133.12
133.01
128.20
127.88
127.64
126.92
126.85
126.14
126.00
125.93
125.87
92.66
90.07
77.21
77.00
76.79
73.87
73.67
73.57
73.05
71.00
68.42
68.25
67.49
67.20
62.55
60.26
20.92
20.71
20.68
20.50
20.46
AcO
AcO
O
O
N3
OAc
21
NAME KLC-856
EXPNO 2
PROCNO 1
Date_ 20091217
Time 21.23
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 100
DS 0
SWH 39370.078 Hz
FIDRES 0.300370 Hz
AQ 1.6646771 sec
RG 7290
DW 12.700 usec
DE 6.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.75 usec
PL1 3.00 dB
PL1W 46.95791626 W
SFO1 150.9251919 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.33 dB
PL12 17.40 dB
PL13 20.40 dB
PL2W 13.96854782 W
PL12W 0.27425295 W
PL13W 0.13745207 W
SFO2 600.1539010 MHz
SI 65536
SF 150.9078449 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

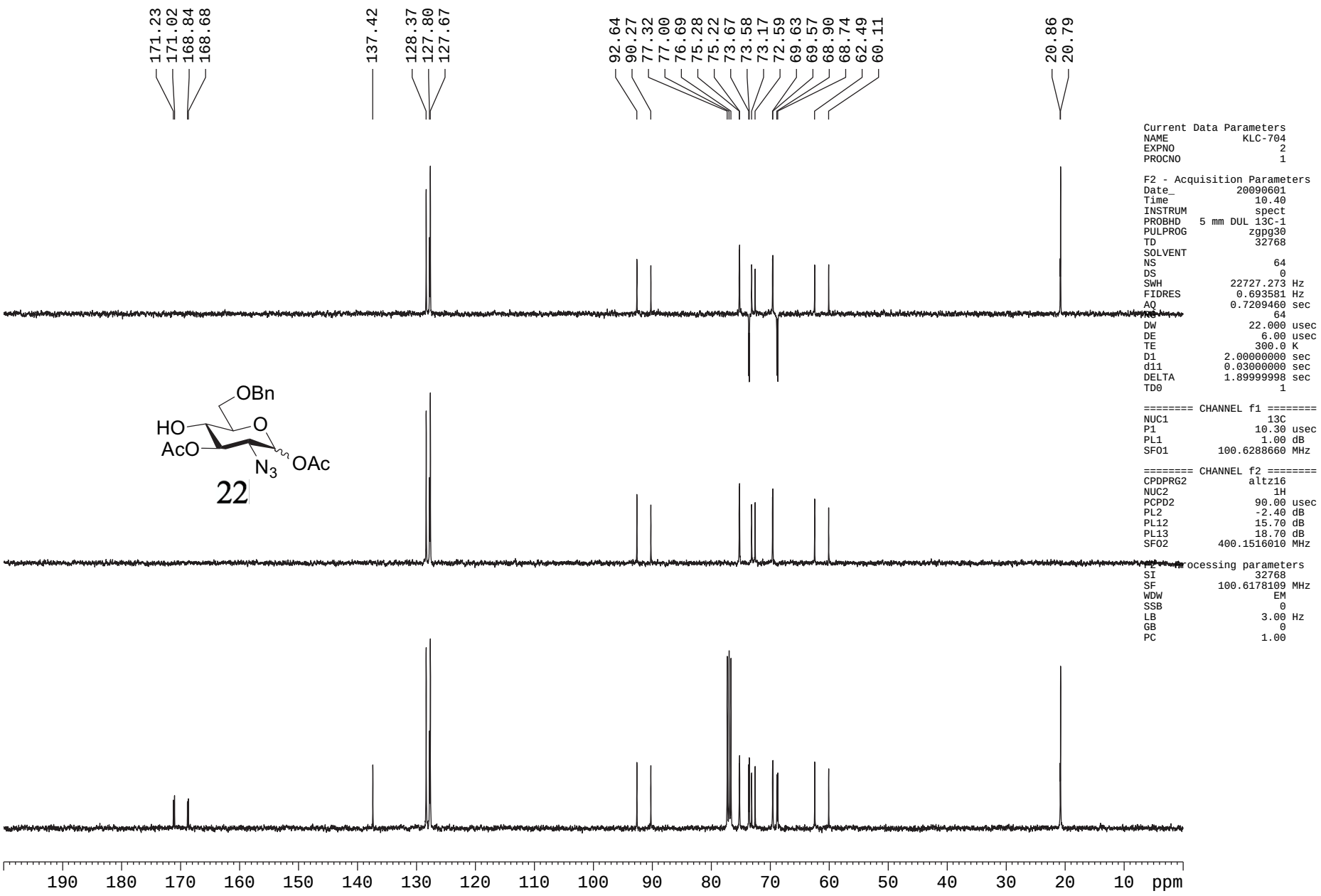


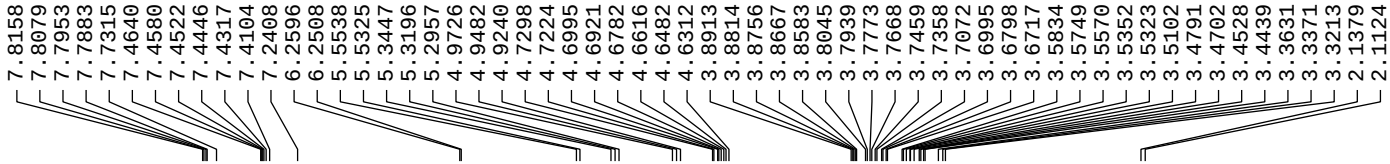

```
Current Data Parameters
NAME            KLC-704
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090601
Time              10.37
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                 50.8
DW               78.000 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500164 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



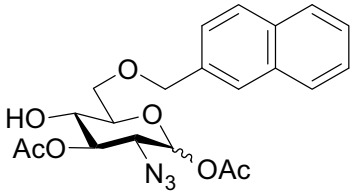
22



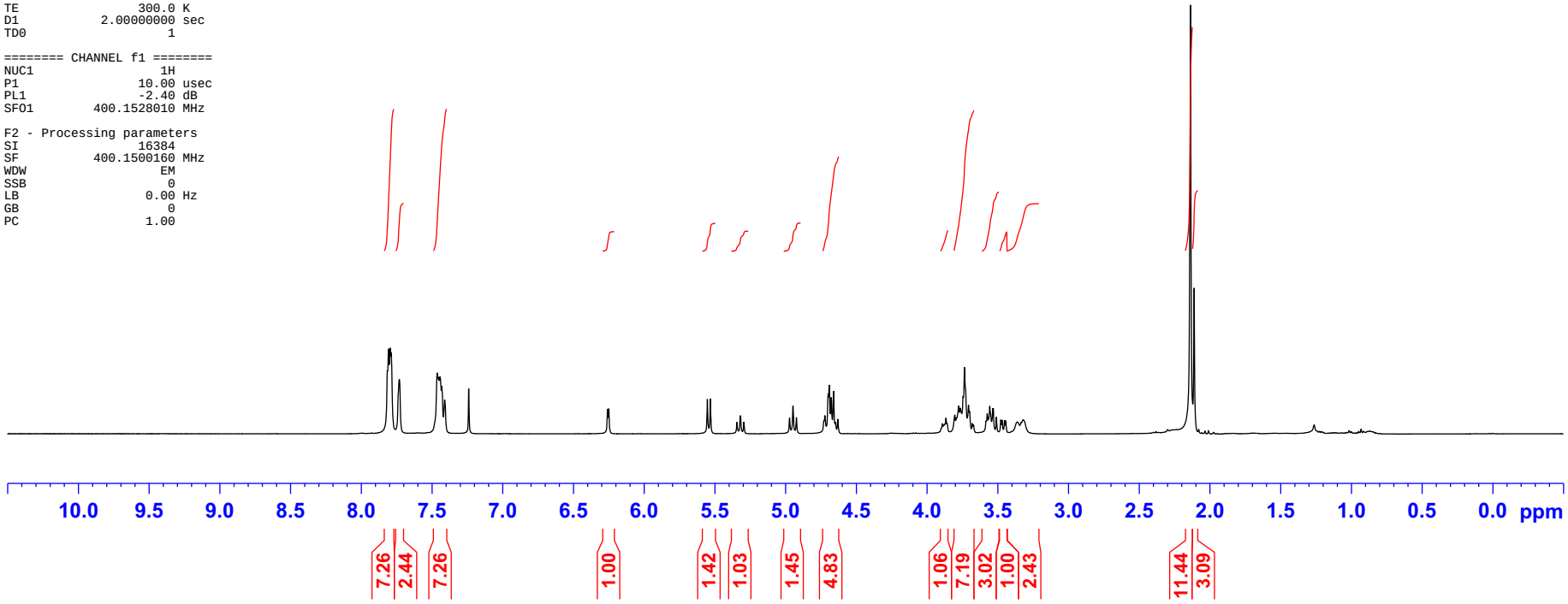

171.23
171.02
168.84
168.68
137.42
128.37
127.80
127.67
92.64
90.27
77.32
77.00
76.69
75.28
75.22
73.67
73.58
73.17
72.59
69.63
69.57
68.90
68.74
62.49
60.11
20.86
20.79
OBn
HO
O
AcO
N3
OAc
22
Current Data Parameters
NAME KLC-704
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090601
Time 10.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 64
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178109 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

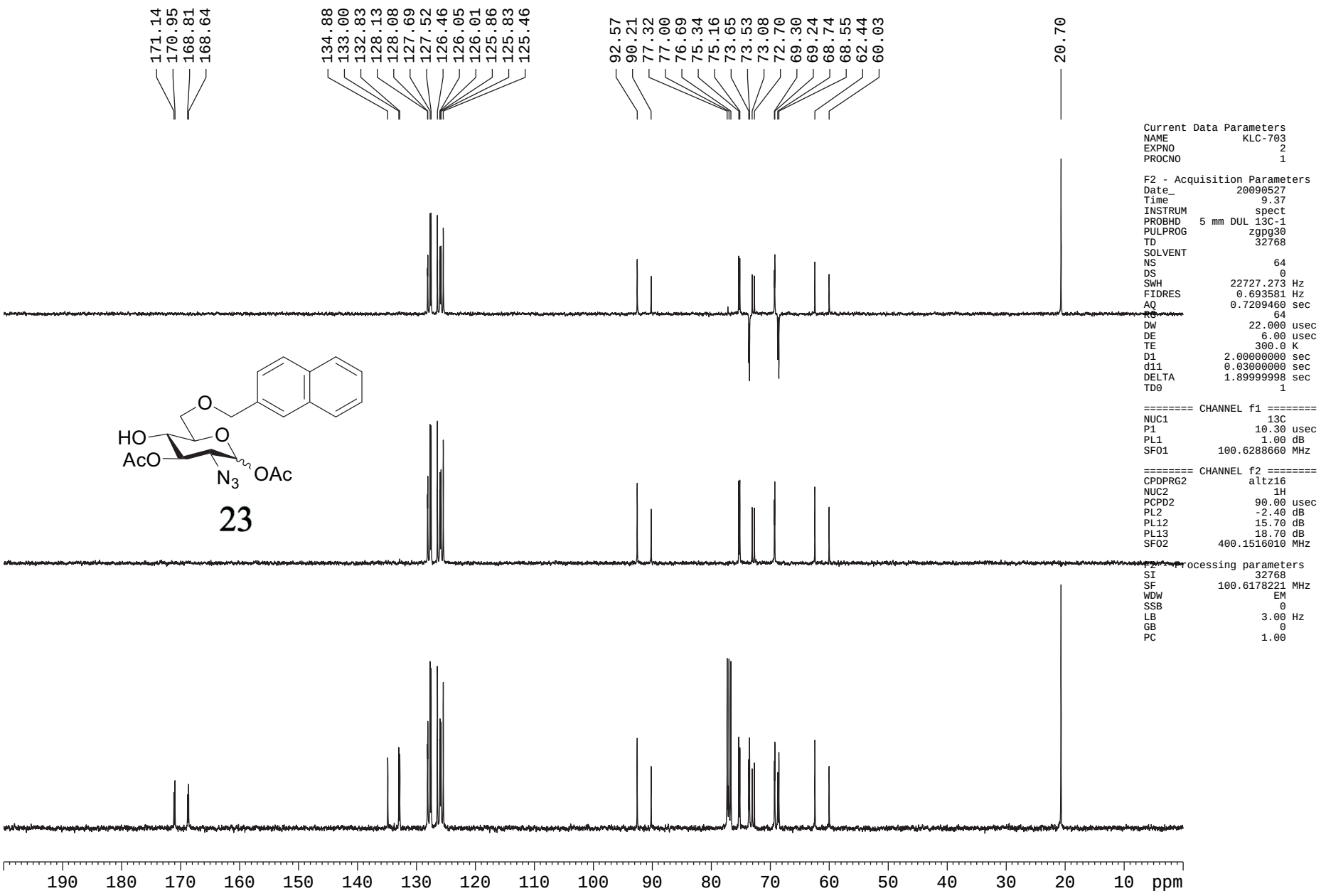


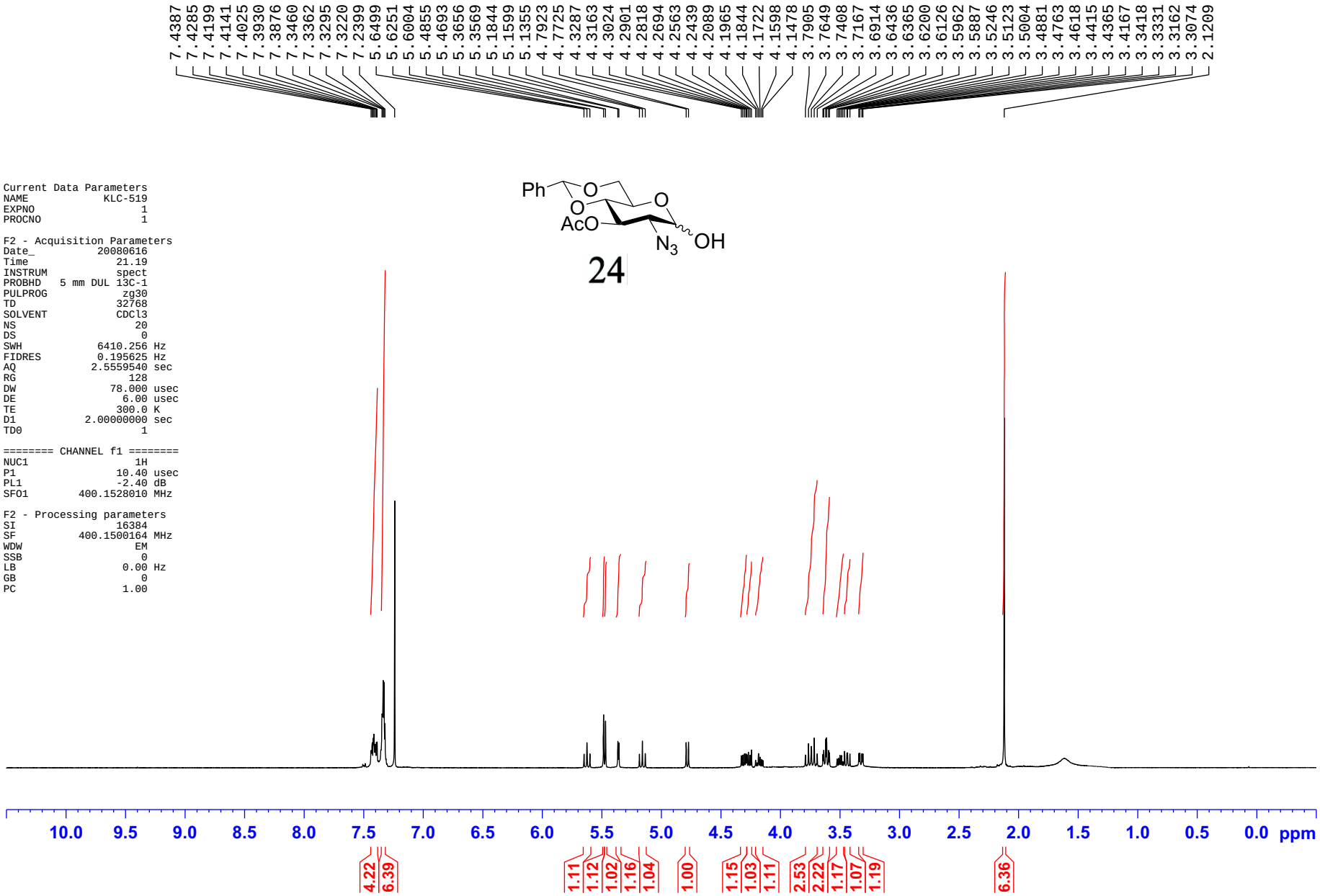

```
Current Data Parameters
NAME            KLC-703
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090527
Time               9.35
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                    32
DW                78.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1               -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500160 MHz
WDW                   EM
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```

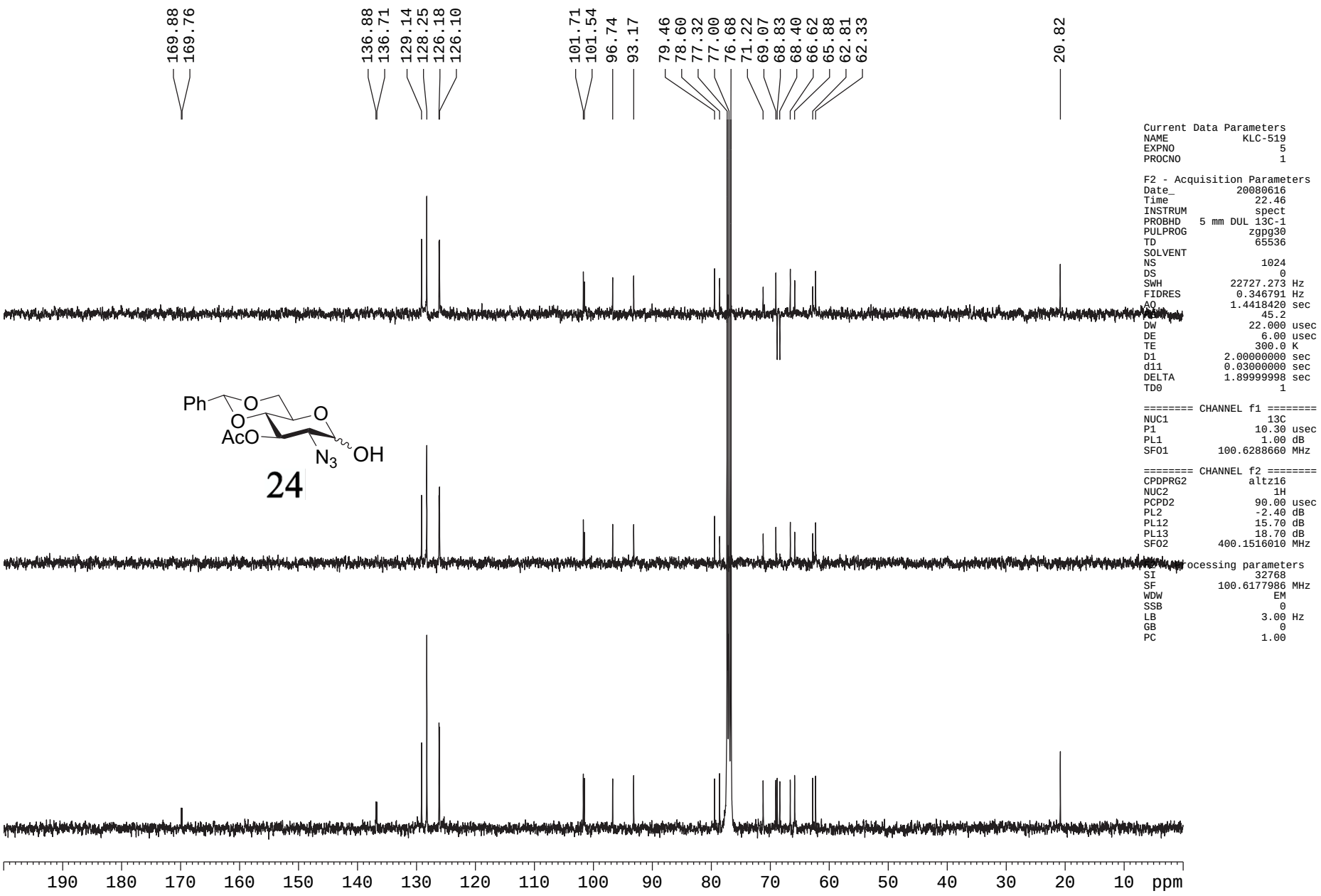


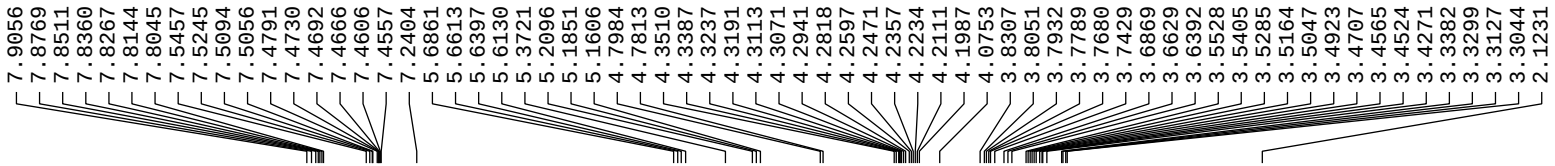
23



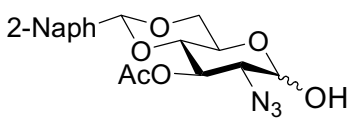

171.14
170.95
168.81
168.64
134.88
133.00
132.83
128.13
128.08
127.69
127.52
126.46
126.05
126.01
125.86
125.83
125.46
92.57
90.21
77.32
77.00
76.69
75.34
75.16
73.65
73.53
73.08
72.70
69.30
69.24
68.74
68.55
62.44
60.03
20.70
HO
AcO
O
O
N3
OAc
23
Current Data Parameters
NAME KLC-703
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090527
Time 9.37
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 64
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178221 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.4387
7.4285
7.4199
7.4141
7.4025
7.3930
7.3876
7.3460
7.3362
7.3295
7.3220
7.2399
5.6499
5.6251
5.6004
5.4855
5.4693
5.3656
5.3569
5.1844
5.1599
5.1355
4.7923
4.7725
4.3287
4.3163
4.3024
4.2901
4.2818
4.2694
4.2563
4.2439
4.2089
4.1965
4.1844
4.1722
4.1598
4.1478
3.7905
3.7649
3.7408
3.7167
3.6914
3.6436
3.6365
3.6200
3.6126
3.5962
3.5887
3.5246
3.5123
3.5004
3.4881
3.4763
3.4618
3.4415
3.4365
3.4167
3.3418
3.3331
3.3162
3.3074
2.1209
Current Data Parameters
NAME KLC-519
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080616
Time 21.19
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 20
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 128
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.40 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500164 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Ph
O
O
O
AcO
N3
OH
24
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.22
6.39
1.11
1.12
1.02
1.16
1.04
1.00
1.15
1.03
1.11
2.53
2.22
1.17
1.07
1.19
6.36


169.88
169.76
136.88
136.71
129.14
128.25
126.18
126.10
101.71
101.54
96.74
93.17
79.46
78.60
77.32
77.00
76.68
71.22
69.07
68.83
68.40
66.62
65.88
62.81
62.33
20.82
Ph
O
O
O
AcO
N3
OH
24
Current Data Parameters
NAME KLC-519
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080616
Time 22.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT
NS 1024
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 45.2
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6177986 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm




```
Current Data Parameters
NAME            KLC-712
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090609
Time              10.23
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   12
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                  203
DW               78.000 usec
DE                6.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500164 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```



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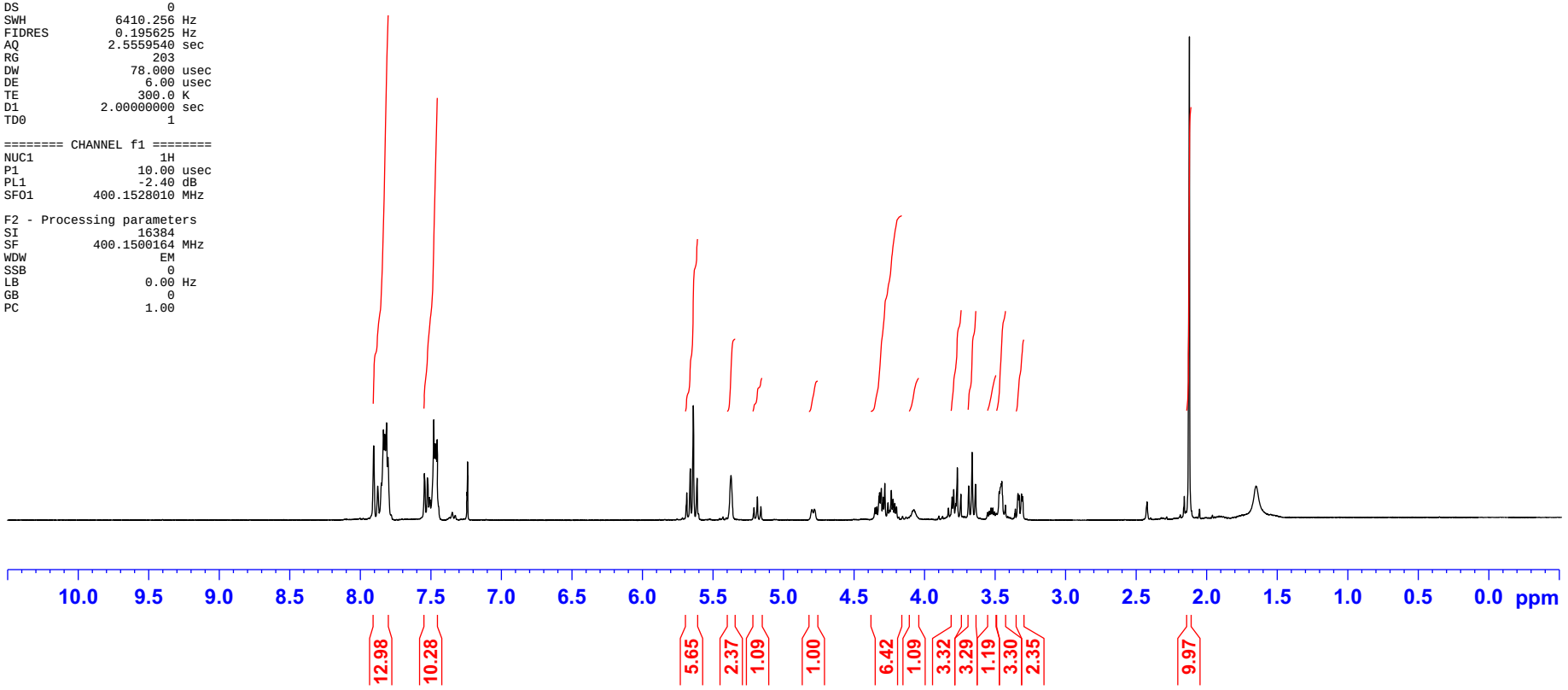

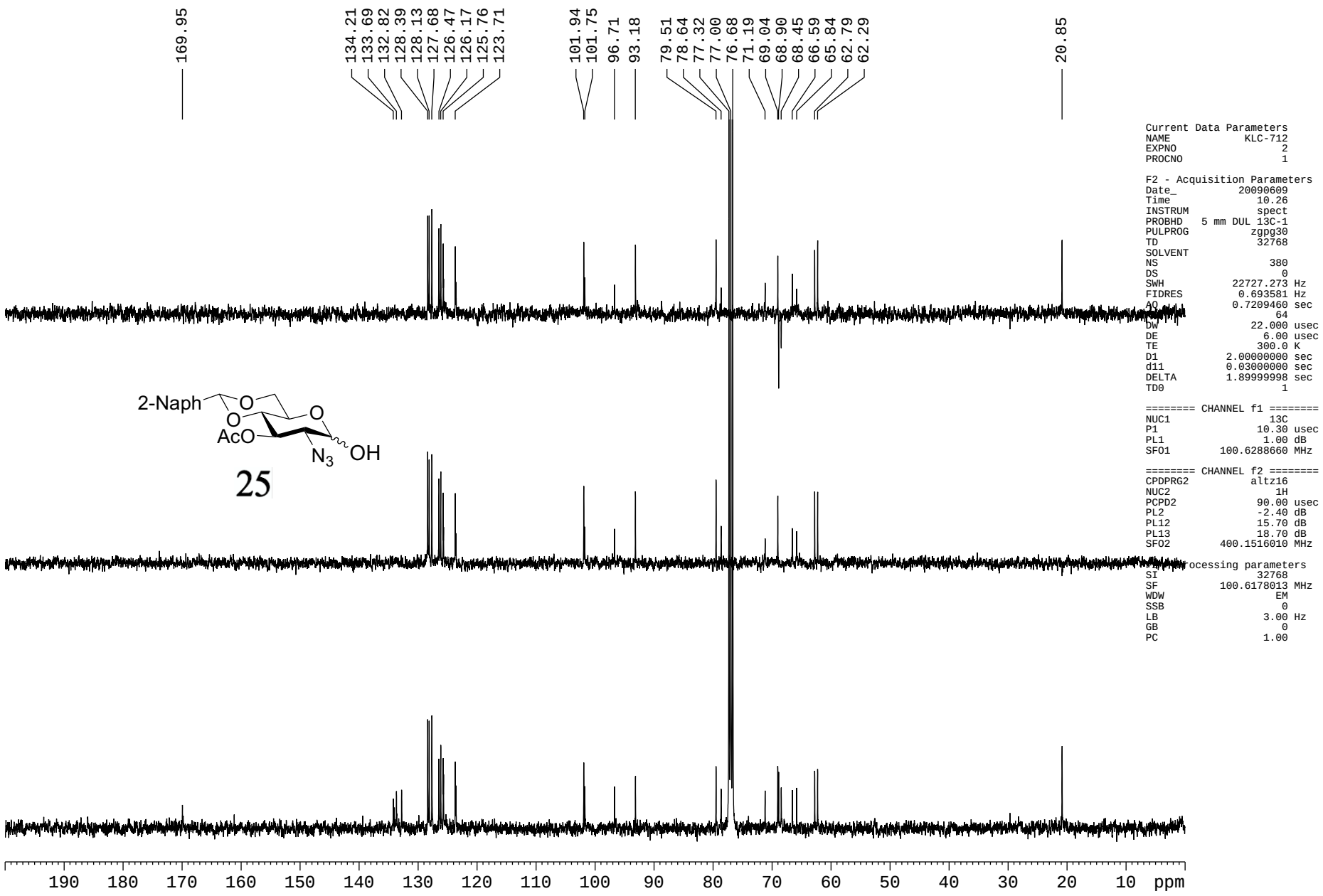
169.95
134.21
133.69
132.82
128.39
128.13
127.68
126.47
126.17
125.76
123.71
101.94
101.75
96.71
93.18
79.51
78.64
77.32
77.00
76.68
71.19
69.04
68.90
68.45
66.59
65.84
62.79
62.29
20.85
2-Naph
O
O
O
AcO
N3
OH
25
Current Data Parameters
NAME KLC-712
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090609
Time 10.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT
NS 380
DS 0
SWH 22727.273 Hz
FIDRES 0.693581 Hz
AQ 0.7209460 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178013 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm