Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Design and synthesis of new amino-modified iminocyclitols: Selective inhibitors of α-galactosidase

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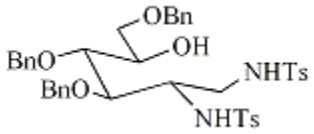
CONTENTS

General considerations:

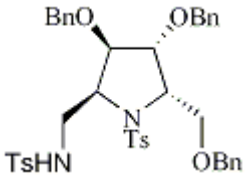
All solvents were purified by standard procedures. Anhydrous solvents were dried over sodium wire (benzene, hexane, THF) or molecular sieves (MeOH, DMF, CH_3CN). Thin-layer chromatography (TLC) was performed on Merck silica gel pre-coated on aluminium plates. Flash column chromatography was performed on 230–400 mesh silica gel. Optical rotations were recorded on an Autopol V (Rudolph Research Flanders, New Jersey) instrument. All the rotations were measured at 589 nm (sodium 'D line). Melting points of the compounds are uncorrected. IR spectra were taken over the 4000–400 cm^{-1} range as KBr pellets on a Nicolet (Madison, USA) FTIR spectrophotometer (Model protégé 460). All the ^1H and ^{13}C NMR spectra were recorded on a 300 MHz Bruker Spectrospin DPX FT-NMR spectrometer. Chemical shifts are reported as δ values (ppm) relative to Me_4Si as internal standard. Mass spectra were recorded on an Applied Biosystems Q-Star instrument. Freeze-drying of samples was done on a Freezone 2.5 (Labconco, USA) lyophilizer.

3,4,6-Tri-*O*-benzyl-1,2-dideoxy-1,2-(di-*p*-toluenesulfonamido)-D-glucitol 9:



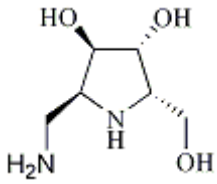
Compound **8**[1] (5.0 g, 6.6 mmol) was taken in a flame dried 100 mL three-necked round bottom flask and 50 mL of THF was added to it. $LiAlH_4$ (0.377 g, 9.9 mmol) was then added and the reaction mixture was refluxed at 66 °C under argon atmosphere. The progress of the reaction was monitored by TLC. After 20 min., when TLC indicated the disappearance of the starting material, the reaction was then quenched with 10% aqueous HCl (100 mL) and the reaction mixture was extracted with $CHCl_3$ (3 x 100 mL). The combined organic layer was dried over sodium sulphate, filtered and then concentrated to give **9** in 95% (4.763 g) as colourless low melting solid. $[\alpha]^{28}_D$ +0.54 (*c* 1.5, $CHCl_3$). ν_{max} (KBr)/cm^{-1}; 3496, 3280, 3032, 2920, 2866, 1451, 1330, 1159, 1088, 814, 742, 699, 666, 552; δ_H (300 MHz; $CDCl_3$): 7.66 (2H, d, *J* 7.8 Hz), 7.54 (2H, d, *J* 8.1 Hz), 7.35–7.13 (19H, m), 5.30 (1H, br s, exchangeable with D_2O), 4.71 (1H, br s, exchangeable with D_2O), 4.68 (2H, d, *J* 11.1 Hz), 4.57-4.44 (4H, m), 4.36 (1H, d, *J* 11.1 Hz), 3.82 (1H, m), 3.68 (1H, m), 3.55–3.52 (2H, m), 3.47-3.37 (2H, m), 2.88 (1H, m), 2.79 (1H, m), 2.38 (3H, s), 2.34 (3H, s); δ_C (300 MHz; $CDCl_3$) : 143.5 (C), 143.3 (C), 137.7 (C), 137.4 (C), 137.1 (C), 136.4 (C), 129.6 (CH), 128.5 (CH), 128.06 (CH), 127.9 (CH), 127.1 (CH), 126.9 (CH), 77.8 (2xCH), 74.7 (CH_2), 73.9 (CH_2), 73.3 (CH_2), 71.1 (CH), 70.7 (CH_2), 53.8 (CH), 44.4 (CH_2), 21.4 (2xCH_3); HRMS (ESI): $[M+H]^+$, Found: 759.2734, $C_{41}H_{47}N_2O_8S_2$ requires 759.2774.

(2*S*,3*R*,4*R*,5*S*)-2-(*N*-*p*-Toluenesulfonyl)aminomethyl-3,4-dibenzyloxy-5-benzyloxymethyl-1*N*-(*p*-toluenesulfonyl)pyrrolidine 10:



Compound **9** (5.0 g, 6.59 mmol) was taken in a 100 mL flame dried three necked round bottom flask and 30 mL of dry THF was added to it followed by PPh_3 (2.246 g, 8.56 mmol). The reaction mixture was cooled to 0 °C. Diethyl azodicarboxylate, DEAD (1.517 mL, 9.23 mmol) was injected into the reaction mixture drop-wise under argon atmosphere. After the addition was over, the reaction mixture was brought to room temperature. The reaction was found to be completed in 30 min. (vide TLC). The reaction was then stopped and the solvent was evaporated. Flash chromatography (hexane-ethyl acetate 6:1) of resulting residue provided **10** in 90% (4.393 g) yield as a colorless viscous liquid; $[\alpha]^{28}_{D}$ -1.1 (*c* 0.53, $CHCl_3$); ν_{max} (KBr)/cm^{-1}; 3378, 3031, 2923, 2867, 2359, 1497, 1456, 1405, 1334, 1160, 1094, 741, 700, 663, 554; δ_H (300MHz; $CDCl_3$) : 7.71 (2H,d, *J* 8.1 Hz), 7.67 (2H, d, *J* 8.1 Hz), 7.31–7.16 (17H, m), 6.91-6.89 (2H, m), 5.43 (1H, t, *J* = 7.2 Hz, exchangeable with D_2O), 4.63–4.44 (5H, m), 4.13–3.99 (4H, m), 3.88–3.82 (1H, m), 3.76 (1H, dd, *J* 9.9 Hz, 3.3 Hz), 3.48 (1H, d, *J* 9.9 Hz), 3.44–3.27 (2H, m), 2.39 (3H, s), 2.29 (3H, s); δ_C (300 MHz; $CDCl_3$) : 143.5 (C), 143.1 (C), 137.9 (C), 137.6 (C), 137.4 (C), 137.1 (C), 136.6 (C), 129.6 (CH), 129.5 (CH), 128.6 (CH), 128.5 (CH), 128.1 (CH), 128.1 (CH), 128.01 (CH), 127.9 (CH), 127.5 (CH), 127.4 (CH), 127.3 (CH), 127.2 (CH), 126.8 (CH), 80.1 (CH), 80.7 (CH), 73.5 (CH_2), 73.3 (CH_2), 72.6 (CH_2), 65.6 (CH_2), 58.4 (CH), 58.3 (CH), 43.8 (CH_2), 21.5 (CH_3), 21.5 (CH_3); HRMS (ESI): $[M+H]^+$, Found: 741.2635, $C_{41}H_{45}N_2O_7S_2$ requires 741.2668.

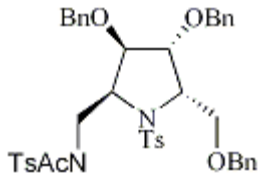
(2*S*,3*R*,4*R*,5*S*)-2-Aminomethyl-3,4-dihydroxy-5-hydroxymethyl-pyrrolidine 12:



Liquid ammonia (40 mL) was collected in a flame dried 100 mL three-necked round bottom flask at -78 °C. Sodium metal (0.140 g, 6.073 mmol) was added to it. Deep blue colour appeared. Pyrrolidine **10** (1.0 g, 1.35 mmol) dissolved in THF (2 mL) was added to the reaction mixture and stirred at -78 °C for 3 h. The reaction was quenched by the addition of benzene until the blue colour disappeared followed by addition of water (10 mL). The reaction mixture was allowed to slowly warm to room temperature. The crude reaction mixture was dried in a lyophilizer. Column chromatography (CH_3CN-NH_4OH 4:1) of the residue over deactivated silica yielded

ADMDP **12** in 86% (0.188 g) as a pale yellow low melting solid. $[\alpha]^{28}_{D}$ +2.3 (c 0.30, D_2O); ν_{max} (KBr)/cm^{-1}: 3451, 2924, 2361, 1717, 1640, 1452, 1330, 1269, 1158, 1095, 812, 754, 706, 667, 553; δ_H (300MHz; D_2O): 4.29 (2H, d, *J* 10.5 Hz) 4.05 (1H, s), 3.90-3.79 (3H, m), 3.43-3.39 (2H, m); δ_C (300 MHz; D_2O): 74.8 (CH), 74.4 (CH), 63.5 (CH), 58.2 (CH), 57.4 (CH_2), 36.2 (CH_2); HRMS (ESI): [M+H]+, Found: 163.1080, $C_6H_{15}N_2O_3$ requires 163.1083.

(2*S*,3*R*,4*R*,5*S*)-2-(*N*-Acetyl-*N*-*p*-toluenesulfonyl)aminomethyl-3,4-dibenzyloxy-5-benzyloxymethyl-1*N*-(*p*-toluenesulfonyl)pyrrolidine 13:



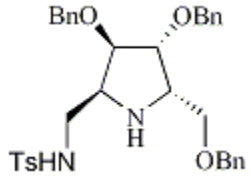
In a 50 mL single necked round bottom flask was taken compound **10** (3.0 g, 3.83 mmol) and dissolved in pyridine (30 mL). DMAP (0.467 g, 3.83 mmol) was then added and reaction mixture was cooled to 0°C. Ac_2O (0.724 mL, 7.66 mmol) was then added and the reaction mixture was allowed to come at room temperature and stirred for 24 h. The reaction was quenched with 10% aqueous HCl (100 mL) and the reaction mixture was extracted with EtOAc (3 x 150 mL). The organic layer then washed with brine solution and dried over sodium sulphate, filtered and then concentrated. Flash chromatography of crude reaction mixture was performed with hexane-ethyl acetate (6:1) to obtain **13** in 95% yield (3.012 g) as a colourless viscous liquid; $[\alpha]^{25}_{D}$ -1.9 (*c* 0.58, $CHCl_3$)**;** ν_{max} (KBr)/cm^{-1}: 3026, 2924, 2861, 2360, 1700, 1645, 1455, 1354, 1162, 1096, 762, 663, 581, 547; δ_H (300MHz; $CDCl_3$); 7.79 (2H, d, *J* 7.8 Hz), 7.77 (2H, d, *J* 7.5 Hz), 7.29–7.10 (17H, m), 6.93-6.90 (2H, m), 4.66–4.48 (5H, m), 4.43–4.21 (4H, m), 4.14-4.07 (2H, m), 3.96 (1H, dd, *J* 12 Hz), 3.80 (1H, dd, *J* 9.9, 4.2 Hz), 3.57 (1H, d, *J* 9.9 Hz), 2.39 (3H, s), 2.26 (3H, s), 2.18 (3H, s); δ_C (300 MHz; $CDCl_3$): 170.9 (C), 144.7 (C), 142.9 (C), 138.04 (C), 137.9 (C), 137.9 (C), 137.7 (C), 136.5 (C), 129.8 (CH), 129.6 (CH), 129.2 (CH), 129.02 (CH), 128.3 (CH), 128.2 (CH), 127.9 (CH), 127.8 (CH), 127.6 (CH), 127.4 (CH), 127.2 (CH), 127.1 (CH), 126.9 (CH), 81.3 (CH), 79.6 (CH), 73.3 (CH_2), 73.2 (CH_2), 72.5 (CH_2), 65.2 (CH_2), 58.8 (CH), 56.4 (CH), 46.2 (CH_2), 25.1 (CH_3), 21.6 (CH_3), 21.4 (CH_3); HRMS (ESI): $[M+Na]^+$, Found: 805.2584, $C_{43}H_{46}N_2O_8S_2Na$ requires 805.2593.

General procedure for Na-Hg mediated detosylation reactions.

In a flame dried 50 mL three necked round bottom flask were taken 3% Na/Hg and Na_2HPO_4 at room temperature under Ar atmosphere. Compound to be detosylated, dissolved in a solvent mixture (DMF-MeOH 8:1), was then added and the reaction mixture was heated at 60 °C for 3 h

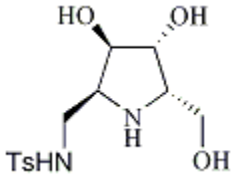
after which the reaction was stopped. The reaction mixture was then filtered through a celite pad. The filtrate was diluted with EtOAc and washed with brine solution, a few times, to remove DMF completely from the reaction mixture. The organic layer was dried over anhydrous sodium sulphate, filtered and then concentrated. Flash chromatography of crude reaction mixture afforded the corresponding detosylated compounds.

(2*S*,3*R*,4*R*,5*S*)-2-(*N*-*p*-Toluenesulfonyl)aminomethyl-3,4-dibenzyloxy-5-benzyloxymethyl-pyrrolidine 14:



Compound **14** (0.76 g, 96% yield) was obtained as a yellow gummy liquid from the reaction of compound **10** (1.0 g, 1.35 mmol) with 3% Na-Hg (52.0 g, 67.57 mmol) and Na_2HPO_4 (1.2 g, 6.76 mmol) following the general procedure described above. Product **14** was purified by column chromatography over silica using hexane-ethyl acetate (2:1). $[\alpha]^{28}_D$ -6.08 (*c* 0.44, $CHCl_3$); ν_{max} (KBr)/cm^{-1}: 3309, 3031, 2922, 2858, 1956, 1878, 1811, 1722, 1598, 1454, 1329, 1214, 1158, 1088, 910, 813, 741, 698, 664, 604, 552; δ_H (300MHz; $CDCl_3$) ; 7.65 (2H, d, *J* 8.1 Hz.) , 7.36- 7.22 (17H, m), 4.57- 4.53 (5H, m), 4.35 (1H, d, *J* 11.7 Hz), 4.0 – 3.97 (2H, m), 3.64 – 3.52 (4H, m), 3.12(1H, dd, *J* 12.6, 6.3 Hz), 2.97(1H, dd, *J* 18.6, 6.3 Hz), 2.44 (3H, s); δ_C (300 MHz; $CDCl_3$): 142.9 (C), 138.04 (C), 137.8 (C), 137.5 (C), 136.8 (C), 129.5 (CH) 128.5 (CH), 128.3 (CH), 128.3 (CH), 127.9 (CH), 127.7 (CH), 127.6 (CH), 127.6 (CH), 127.3 (CH), 126.9 (CH), 126.8 (CH), 82.8 (CH), 82.2 (CH), 73.2 (CH_2), 72.01 (CH_2), 71.9 (CH_2), 68.9 (CH_2), 64.8 (CH), 58.4 (CH), 57.6 (CH_2), 43.4 (CH_2), 21.3 (CH_3); HRMS (ESI): $[M+H]^+$, Found: 587.2586, $C_{34}H_{39}N_2O_5S$ requires 587.2580.

(2*S*,3*R*,4*R*,5*S*)-2-(*p*-Toluenesulfonyl)aminomethyl-3,4-dihydroxy-5-hydroxymethyl-pyrrolidine 15:



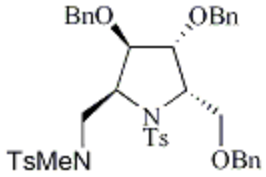
10% palladium on charcoal (1.5 g, 300% w/w) was taken in a 50 mL three-necked round bottom flask. Compound **14** (0.5 g, 0.85 mmol) dissolved in methanol (10 mL) was added to the reaction flask. H_2 gas was then bubbled slowly into reaction mixture for 5 h at 30 ^oC. The reaction mixture was then filtered through a celite pad. The filtrate was collected and the solvent

was evaporated to yield compound **15** as a white solid in 90% (0.243 g). mp 182 ^{o}C (recrystallized from MeOH/THF); $[\alpha]^{28}_{D}$ +7.4 (*c* 0.35, H_2O); ν_{max} (KBr)/cm^{-1}: 3150, 1745, 1632, 1406, 1325, 1158, 1089, 815, 666, 554; δ_H (300MHz; D_2O): 7.73 (2H, br s), 7.42 (2H, br s), 4.309 (1H, br s), 4.21 (1H, s), 3.94 (4H, m), 3.26 (2H, m), 2.37 (3H, s); δ_C (300 MHz; D_2O): 145.4 (C), 134.2 (C), 130.2 (CH), 126.8 (CH), 74.5 (CH), 74.4 (CH), 63.3 (CH), 61.05 (CH), 57.4 (CH_2), 39.5 (CH_2), 20.7 (CH_3); HRMS (ESI): $[M+H]^+$, Found: 317.1156, $C_{13}H_{21}N_2O_5S$ requires 317.1171.

General procedure for alkylation of compound 10:

Compound **10** was taken in a flame dried three-necked round bottom flask and dissolved in dry DMF. Sodium hydride was added to it at 0 ^{0}C followed by alkyl halide and the reaction mixture was stirred at room temperature for the specified time after which the reaction was stopped. The reaction was then quenched with iced water. The reaction mixture was diluted with EtOAc and washed with brine solution, a few times, to remove DMF completely from the reaction mixture. The organic layer was dried over anhydrous sodium sulphate, filtered and then concentrated. Flash chromatography of crude reaction mixture afforded the corresponding alkylated compounds.

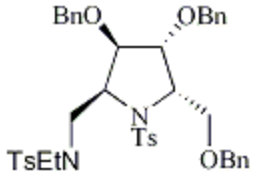
(2*S*,3*R*,4*R*,5*S*)-2-(*N*-Methyl-*N*-*p*-toluenesulfonyl)aminomethyl-3,4-dibenzyloxy-5-benzyloxymethyl-1*N*-(*p*-toluenesulfonyl)pyrrolidine 16:



Compound **16** (1.814 g, 89% yield) was obtained in 3 h as a white crystalline solid from the reaction of compound **10** (2.0 g, 2.69 mmol) with NaH (0.432 g of 60% suspension in mineral oil) and methyl iodide (0.252 mL, 4.05 mmol) following the general procedure described above. Product **16** was purified by column chromatography over silica using hexane-ethyl acetate (8:1). mp 110 ^{o}C (recrystallized from benzene/hexane); $[\alpha]^{27}_{D}$ +28.4 (*c* 0.69, $CHCl_3$); ν_{max} (KBr)/cm^{-1}:3029, 2918, 2865, 1717, 1594, 1491, 1453, 1338, 1157, 1088, 1018, 810, 747, 699, 659, 601, 546; δ_H (300MHz; $CDCl_3$) : 7.89 (2H, d, *J* 7.8 Hz.) , 7.84 (2H, d, *J* 8.1 Hz), 7.33 - 7.19 (17H, m), 6.89 (2H, m), 4.78 (1H, d, *J* 11.7 Hz), 4.64 (1H, d, *J* 11.7 Hz), 4.56 – 4.49 (4H, m), 4.11 – 3.88 (5H ,m), 3.79-3.76 (1H, m), 3.60 (1H, d, *J* 9.0 Hz), 2.88-2.80 (1H, m), 2.66 (3H, s), 2.43 (3H, s), 2.29 (3H , s); δ_C (300 MHz; $CDCl_3$): 143.5 (C), 142.9 (C), 137.9 (C), 137.8 (C), 137.5 (C), 132.2 (C), 129.6 (CH) 129.3 (CH), 128.3 (CH), 128.2 (CH), 127.9 (CH), 127.8 (CH), 127.6 (CH), 127.5 (CH), 127.3 (CH), 127.1 (CH), 126.8 (CH), 81.03

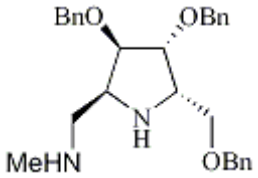
(CH), 79.1 (CH), 73.5 (CH_2), 73.01 (CH_2), 72.5 (CH_2), 65.2 (CH_2), 59.2 (CH), 58.8 (CH), 51.9 (CH_2), 38.9 (CH_3), 21.5 (CH_3), 21.4 (CH_3); HRMS (ESI): $[M+H]^+$, Found: 755.2818, $C_{42}H_{47}N_2O_7S_2$ requires 755.2825.

(2*S*,3*R*,4*R*,5*S*)-2-(*N*-Ethyl-*N*-*p*-toluenesulfonyl)aminomethyl-3,4-dibenzyloxy-5-benzyloxymethyl-1*N*-(*p*-toluenesulfonyl)pyrrolidine 17:



Compound **17** (2.413 g, 93% yield) was obtained in 5 h as colourless liquid from the reaction of compound **10** (2.5 g, 3.37 mmol) with NaH (0.404 g of 60% suspension in mineral oil) and ethyl iodide (0.329 mL, 4.05 mmol) following the general procedure described above. Product **17** was purified by column chromatography over silica using hexane-ethyl acetate (6:1). $[\alpha]^{28}_D$ +35.8 (*c* 0.48, $CHCl_3$); ν_{max}(KBr)/cm^{-1}: 3409, 3062, 3031, 2929, 2871, 1710, 1598, 1494, 1453, 1453, 1340, 1155, 1092, 908, 814, 731, 699, 662, 598, 548; δ_H (300MHz; $CDCl_3$) ; 7.86 (2H, d, *J* 8.4 Hz), 7.83 (2H,d, *J* 8.4 Hz), 7.30-7.13 (17H, m), 6.96-6.94 (2H, m), 4.71 (1H, d, *J* 11.7 Hz), 4.64-4.60 (2H, m), 4.53-4.47 (1H, dd, *J* 11.7, 4.5 Hz), 4.40 (1H, t, *J* 6.3Hz), 4.15 (1H, d, *J* 11.7Hz), 4.07-4.03 (2H, m), 3.95 (1H, d, *J* 14.4 Hz), 3.77-3.68 (4H ,m), 3.45-3.37 (1H, m), 3.25-3.14 (2H, m), 2.41 (3H, s), 2.29 (3H, s), 0.75 (3H, t, *J* 7.2 Hz); δ_C (300 MHz; $CDCl_3$): 143.3 (C), 143.1 (C), 138.0 (C), 137.9 (C), 137.9 (C), 137.8 (C), 136.4 (C), 129.7 (CH), 129.4 (CH), 128.4 (CH), 128.3 (CH), 128.1 (CH), 127.9 (CH) 127.8 (CH), 127.7 (CH), 127.6 (CH), 127.5 (CH), 127.4 (CH), 127.3 (CH), 127.04 (CH), 80.7 (CH), 78.9 (CH), 73.2 (CH_2), 73.04 (CH_2), 72.7 (CH_2), 65.5 (CH_2), 59.9 (CH), 59.7 (CH), 48.01 (CH_2), 45.3 (CH_2), 21.59 (CH_3), 21.53 (CH_3), 12.2 (CH_3); HRMS (ESI): $[M+Na]^+$, Found:791.2810, $C_{43}H_{48}N_2O_7S_2Na$ requires 791.2801.

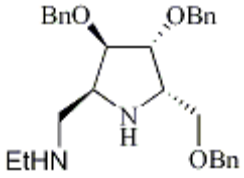
(2*S*,3*R*,4*R*,5*S*)-2-(*N*-Methyl)aminomethyl-3,4-dibenzyloxy-5-benzyloxymethyl-pyrrolidine 18:



Compound **18** (0.408 g, 69% yield) was obtained as a yellow gummy liquid from the reaction of compound **16** (1.0 g, 1.32 mmol) with 3% Na-Hg (126.939 gm, 165.57 mmol) and Na_2HPO_4 (2.948 gm, 16.56 mmol) following the general procedure described above. Product **18** was purified by column chromatography over silica using $CHCl_3$:MeOH:NH_4OH (100:2.5:2.5) as a solvent mixture. $[\alpha]^{27}_D$ 14.7 (*c* 0.43, $CHCl_3$); ν_{max}(KBr)/cm^{-1}: 3835, 3020, 2924, 2861, 2963, 1709, 1552, 1459, 1407, 1218, 1099, 764, 694; δ_H (300MHz; $CDCl_3$): 7.37-7.26

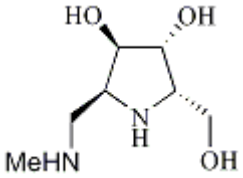
(15H, m), 4.58- 4.48 (5H, m), 4.40 (1H, d, *J* 12Hz), 4.03- 3.93 (2H, m), 3.68- 3.49 (4H, m), 2.73– 2.67 (2H, m), 2.41 (3H, s), 1.96 (2H, br s, N*H*, exchangeable with D_2O), δ_C (300 MHz; $CDCl_3$): 138.3 (C), 138.2 (C), 137.1 (C), 128.4 (CH), 127.3 (CH), 128.3 (CH), 127.8 (CH), 127.7 (CH), 127.7 (CH), 127.6 (CH), 127.5 (CH), 83.04 (CH), 82.4 (CH), 73.3 (CH_2), 72.2 (CH_2), 71.9 (CH_2), 69.6 (CH_2), 58.5 (2xCH), 51.7 (CH_2), 36.7 (CH_3); HRMS (ESI): $[M+H]^+$, Found: 447.2647, $C_{28}H_{35}N_2O_3$ requires 447.2648.

(2*S*,3*R*,4*R*,5*S*)-2-(N-Ethyl)aminomethyl-3,4-dibenzyloxy-5-benzyloxymethyl-pyrrolidine 19:



Compound **19** (0.449 g, 75% yield) was obtained as a yellow gummy liquid from the reaction of compound **17** (1.0 g, 1.3 mmol) with 3% Na-Hg (124.62 g, 162.55 mmol) and Na_2HPO_4 (2.893 g, 16.26 mmol) following the general procedure described above. Product **19** was purified by column chromatography over silica using $CHCl_3$:MeOH:NH_4OH (10:0.1:0.1) as a solvent mixture. $[\alpha]^{28}_D$ +8.9 (*c* 0.76, $CHCl_3$); ν_{max} (KBr)/cm^{-1}: 3323, 3061, 3031, 2863, 1955, 1877, 1813, 1649, 1608, 1491, 1455, 1362, 1309, 1208, 1095, 1208, 1095, 911, 808, 697, 608, 465 and; δ_H (300MHz; $CDCl_3$); 7.34-7.28 (15H, m), 4.60-4.50 (5H, m), 4.42 (d, 1H, *J* 12 Hz), 4.06-3.97 (2H, m), 3.72-3.52 (4H, m), 2.84-2.73 (2H, m), 2.65 (2H, q, *J* 7.2 Hz), 2.07 (2H, br s, N*H* exchangeable with D_2O), 1.08 (3H, t, *J* 7.2 Hz); δ_C (300 MHz; $CDCl_3$): 138.1 (C), 137.9 (C), 137.7 (C), 128.5 (CH), 128.4 (CH), 127.9 (CH), 127.8 (CH), 128.1 (CH), 127.8 (CH), 127.7 (CH), 127.3 (CH), 126.9 (CH), 82.7 (CH), 82.2 (CH), 73.4 (CH_2), 72.4 (2xCH_2), 69.1 (CH_2), 58.7 (CH), 57.1 (CH), 48.7 (CH_2), 43.9 (CH_2), 13.5 (CH_3); HRMS (ESI): $[M+H]^+$, Found: 461.2806, $C_{29}H_{37}N_2O_3$ requires 461.2804.

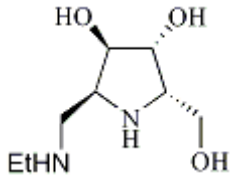
(2*S*,3*R*,4*R*,5*S*)-2-(*N*-Methyl)aminomethyl-3,4-dihydroxy-5-hydroxymethyl-pyrrolidine 20:



10% palladium on charcoal (0.4 g, 100% w/w) was taken in a 50 mL three-necked round bottom flask. Compound **18** (0.4 g, 0.89 mmol) dissolved in methanol (14 mL) was added to the reaction flask followed by the addition of 10% HCl in methanol (1.75 mL). H_2 gas was then bubbled slowly into reaction mixture for 12 h at 30 ^oC. The reaction mixture was then filtered through a celite pad and the filtrate was evaporated to get compound **20** as its hydrochloride salt which was then basified with triethylamine untill pH 12.0. Further purification of the product by

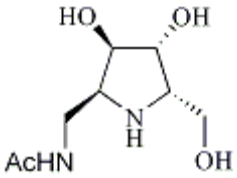
column chromatography with CH_3CN-NH_4OH (1:1) afforded the free base **20** in 94% (0.148 g) yield as a low melting white solid; $[\alpha]^{28}_D$ +3.8 (*c* 1.6, H_2O); ν_{max} (KBr)/cm^{-1}: 3379, 1631, 1406, 1082, 571; δ_H (300MHz; D_2O): 4.31 (2H, m), 4.11-4.08 (1H, m), 3.90-3.79 (3H, m), 3.51 (1H, dd, *J* 13.5, 6.0Hz), 3.41 (1H, dd, *J* 13.5, 6.6 Hz), 2.75 (3H, s); δ_C (300 MHz; D_2O): 75.2 (CH), 74.7 (CH), 63.5 (CH), 57.8 (CH_2), 57.3 (CH), 45.9 (CH_2), 33.8 (CH_3); HRMS (ESI): $[M+Na]^+$, Found: 199.1062, $C_7H_{16}N_2O_3Na$ requires 199.1059.

(2*S*,3*R*,4*R*,5*S*)-2-(*N*-ethyl)aminomethyl-3,4-dihydroxy-5-hydroxymethyl-pyrrolidine 21:



10% palladium on charcoal (0.4 g, 100% w/w) was taken in a 50 ml three necked round bottom flask. Compound **19** (0.4 g, 0.87 mmol) dissolved in methanol (14 mL) was added to the reaction flask followed by the addition of 10% HCl in methanol (1.75 mL). H_2 gas was then bubbled slowly into reaction mixture for 12 h at 30 ^oC. The reaction mixture was then filtered through a celite pad and the filtrate was evaporated to get compound **21** as its hydrochloride salt which was then basified with triethylamine untill pH 12.0. Further purification of the product by column chromatography with CH_3CN-NH_4OH (1:1) afforded **21** in 90% (0.149 g) as a low melting white solid; $[\alpha]^{28}_D$ +5.6 (*c* 0.90, H_2O); ν_{max} (KBr)/cm^{-1}: 3423, 2924, 2854, 2362, 1742, 1626, 1403, 1248, 1176, 1083, 555, 435, 403; δ_H (300MHz; D_2O): 4.22-4.18 (2H, m), 3.92-3.9 (1H, m), 3.79-3.67 (3H, m), 3.38 (1H, dd, *J* 13.5, 6.0 Hz), 3.27 (1H, dd, *J* 13.5, 6.6Hz), 3.03 (2H, q, *J* 7.5Hz), 1.15 (3H, t, *J* 7.5Hz); δ_C (300 MHz; D_2O): 75.3 (CH), 74.9 (CH), 62.9 (CH), 57.9 (CH_2), 57.1 (CH), 44.2 (CH_2), 44.02 (CH_2), 10.6 (CH_3); HRMS (ESI): $[M+H]^+$, Found: 191.1396, $C_8H_{19}N_2O_3$ requires 191.1396.

(2*S*,3*R*,4*R*,5*S*)-2-(*N*-Acetyl)aminomethyl-3,4-dihydroxy-5-hydroxymethyl-pyrrolidine 22:



In flame dried 50 mL three-necked round bottom flask was taken compound **10** (1.0 g, 6.17 mmol). Acetic anhydride (0.582mL, 6.17 mmol) was added to it at 0 ^oC under argon atmosphere. The reaction mixture was sonicated under solvent free condition for 15 minutes at room temperature, after which the reaction was quenched with liquor ammonia until the solution reaches a pH of 9. Column purification using CH_3CN-NH_4OH (1:1) afforded compound **22** in

85% (1.07 g) yield as low melting white solid. $[\alpha]^{28}_{D}$ -4.3 (*c* 0.73, H_2O); ν_{max} (KBr)/cm^{-1}: 3183, 2304, 1649, 1406, 1099, 618; δ_H (300MHz; D_2O): 4.27 (1H, br s), 4.21 (1H, br s), 3.94-3.79 (4H, m), 3.54-3.52 (2H, m), 1.94 (3H, s); δ_C (300 MHz; D_2O): 175.2 (C), 74.7 (CH), 74.3 (CH), 63.2 (CH), 60.9 (CH), 57.5 (CH_2), 36.3 (CH_2), 21.9 (CH_3); HRMS (ESI): $[M+1]^+$, Found: 205.1190, $C_8H_{17}N_2O_4$ requires 205.1188.

General procedure for enzyme assay and calculation of IC_{50} values:

Our experimental design was to measure enzyme velocity at constant substrate concentration with varying concentrations of an inhibitor,[2] spectrophotometrically,[3] by carrying out the inhibition assay of the glycosidases in the presence of the iminosugars as inhibitors utilizing the corresponding *p*-nitrophenyl glycosides as the substrates.

α-Glucosidase type I from Baker's yeast, α-galactosidase from green coffee beans, β-galactosidase from *Escherichia coli* and *p*-nitrophenyl-α-D-galactopyranoside were purchased from Sigma Chemicals Co. USA.

β-glucosidase from almond, *p*NP-α-D-glucopyranoside, *p*NP-β-D-glucopyranoside and *p*NP-β-D-galactopyranoside were purchased from SRL Chemicals Ltd., India.

Glycosidase was pre-incubated with various concentrations (0.5-12 mM) of inhibitor for 30 minutes at its optimum pH and temperature. 20μL of 25mM *p*-nitrophenyl glycopyranoside (*p*-NPG) in 0.1M phosphate buffer was added to the reaction mixture to initiate the reaction. In the case of β-glucosidase acetate buffer was used. The final volume of the reaction mixture was adjusted to 1.1 mL with buffer. Control was also run in parallel without inhibitor. The reaction was then incubated at the same pH and temperature for 10 minutes and quenched by adding 1mL of 1M Na_2CO_3 solution. The glycosidase activity was determined by measuring the *p*-nitrophenol released from *p*-nitrophenyl glycopyranosides at 405 nm using Shimadzu UV-Visible Spectrophotometer (Model UV-1800). IC_{50} values were determined by plotting experimental data as V_i/V_0 (fractional activity) versus the inhibitor concentration [I] at a constant concentration of substrate, where V_i and V_0 represent the enzyme velocity (activity) in the presence and absence of inhibitor, respectively. IC_{50} values were calculated from the inhibitor concentration [I] corresponding to fractional activity of 0.5. IC_{50} value was defined as the concentration of the inhibitor to inhibit 50% of enzyme activity under the assay conditions.

Each experiment was repeated **thrice** to get a range of IC_{50} values and the mean IC_{50} values were reported in the manuscript.

Preparations:

Buffer solutions:	0.1M phosphate buffer for all enzymes.
	For β-glucosidase 0.1M acetate buffer was used.
Quencher :	1M sodium carbonate for all experiments

Enzyme solution:	0.3 mg/mL for α- and β-glucosidase 0.045 mg/mL for α-galactosidase 0.1 mg/mL for β-galactosidase
Substrate concentration:	25 mM made from buffer in all cases
Inhibitor concentration:	Made from buffer in varying concentrations ranging from 0.5 mM to 15 mM

Standardisation: OD values were standardized in terms of amount of *p*-nitrophenol under given buffer condition,
1 OD = X micromoles of *p*-nitrophenol (*p*NP)

From experimental values, OD in terms of amount of *p*-nitrophenol were obtained as follows:
1 OD = 0.093183 micromoles of *p*-nitrophenol for α-glucosidase
1 OD = 0.105579 micromoles of *p*-nitrophenol for β-glucosidase
1 OD = 0.092232 micromoles of *p*-nitrophenol for α-galactosidase
1 OD = 0.079403 micromoles of *p*-nitrophenol for β-galactosidase

Activity: Enzyme activties were calculated as follows:
20 μL of Z mg/mL of Enzyme gives OD value of Y in 10 minutes of incubation period with 20μL of 25 mM Substrate
i.e.,

$$20\ \mu L \times Z\ mg/mL \times 10\ min = Y\ OD$$
$$= Y \times X \quad \{\text{as } 1\ OD = X\ \mu moles\ of\ p\text{-nitrophenol } (p\text{NP})\}$$
$$\text{Activity} = (Y \times X)/(20 \times Z \times 10 \times 10^{-3})$$
$$= (Y \times X \times 1000)/(20 \times Z \times 10)$$
$$= V\ moles/mg/min.$$

Y is the absorbance (@ 405 nm) of the *p*-nitrophenol released at the end of the reaction.
Z is the concentration of the enzyme solution (0.3 mg/mL for α- and β-glucosidase; 0.045 mg/mL for α-galactosidase, 0.1 mg/mL for β-galactosidase).
20 μL of enzyme solution and 20 μL of substrate solution were used in each experiment.

Activity at zero concentration of inhibitor (in the absence of inhibitor) = V_0
Activity at various concentrations of inhibitor = V_i
Fractional activity = V_i/V_0

Plot: (Concentration-response plot)
Fractional activity (V_i/V_0) (on the Y-axis) was plotted against inhibitor concentration [I] (on the X-axis)
IC_{50} values were calculated from the inhibitor concentration [I] corresponding to fractional activity of 0.5.

Result:

Compounds **12** and **22** did not inhibit any of the enzymes up to 12 mM concentration. The inhibition studies of compounds **15**, **20** and **21** are given in Table 1 below.

Table 1: Inhibition activities of compounds **15**, **20** and **21**

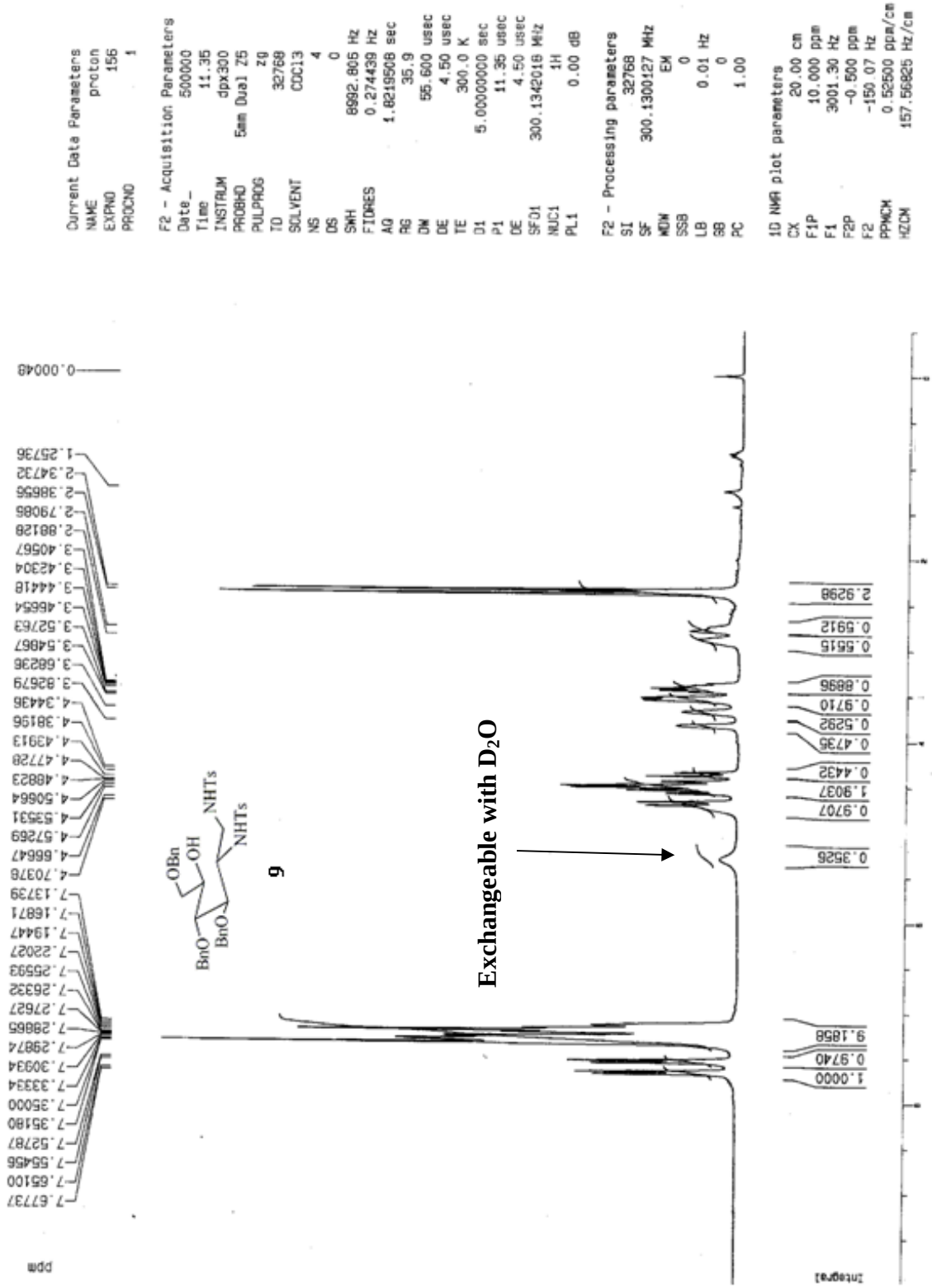
Inhibitor	Enxyme	Source	Condition	IC_{50} (mM)			Range (mM)	Mean IC_{50} (mM)
				Exp. 1	Exp. 2	Exp. 3		
15	α-glucosidase type 1	Baker's yeast	37 °C, 10 min, pH = 6.8	3.6	3.7	3.3	3.3-3.6	3.5
15	β-glucosidase	Almond	37 °C, 10 min, pH = 5	5.6	6.5	6.9	5.6-6.9	6.3
15	β-galactosidase	*Escherichia coli*	37 °C, 10 min, pH = 7.3	6.1	5.4	4.8	4.8-6.1	5.4
20	α-galactosidase	Green coffee beans	$25\ ^0$C, 10 min, pH = 6.5	6.3	6.7	7.8	6.3-7.8	6.9
21	α-galactosidase	Green coffee beans	$25\ ^0$C, 10 min, pH = 6.5	8.8	8.2	7.3	7.3-8.8	8.1

References:

(1) (a) Kumar, V.; Ramesh, N. G. *Org. Biomol. Chem.* **2007**, *5*, 3847. (b) Kumar, V.; Ramesh, N. G. *Chem. Commun.* **2006**, 4952

(2) Hamilton-Miller, J. M. T. *Biochem. J.* 1966, **101**, 40c.

(3) Li, Y.-T.; S.; Li, S.-C. in *Methods in Enzymology;* Ginsberg, V., Ed.; Academic Press, 1972, vol. 28, part B, p. 702.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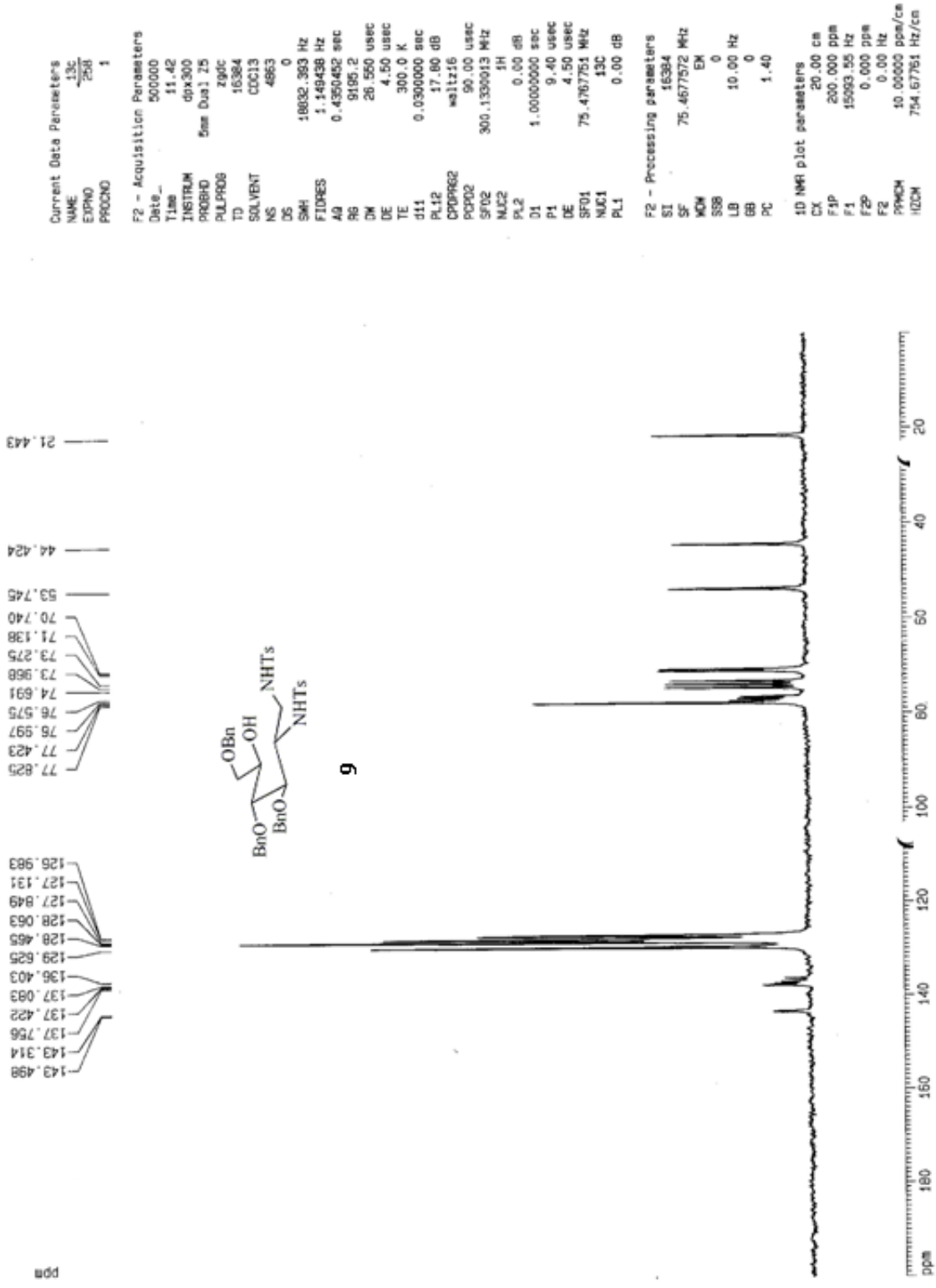
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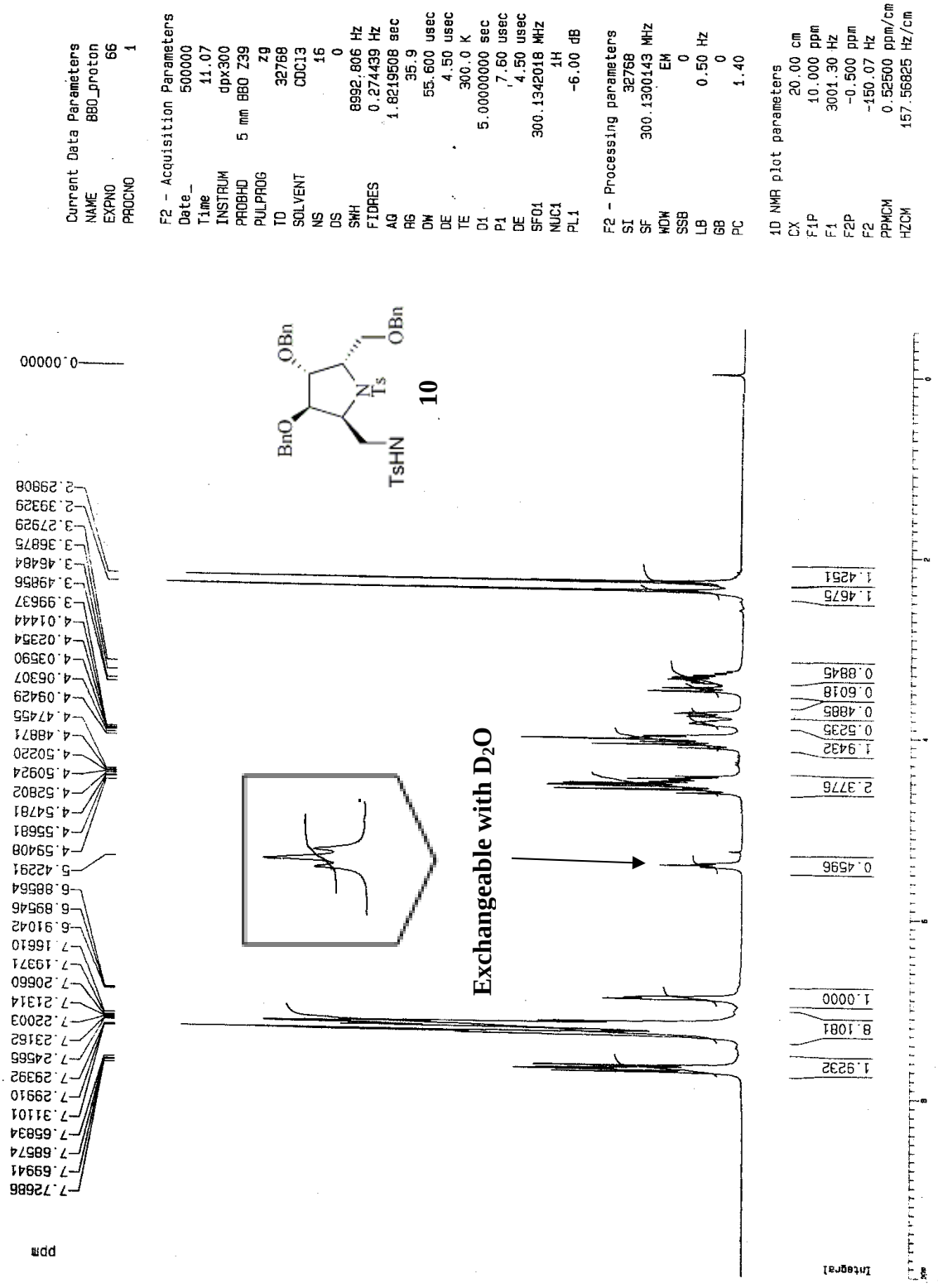
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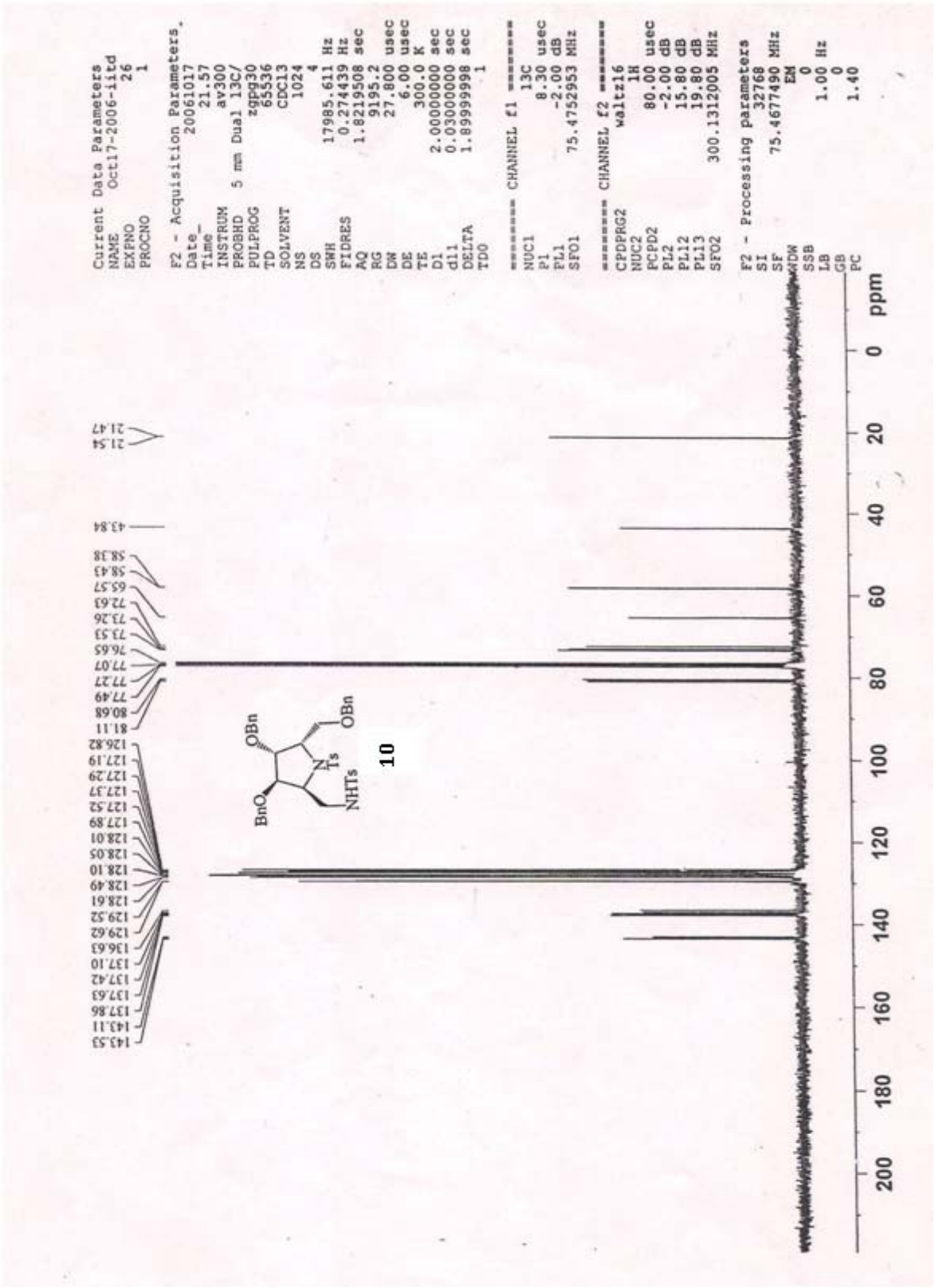

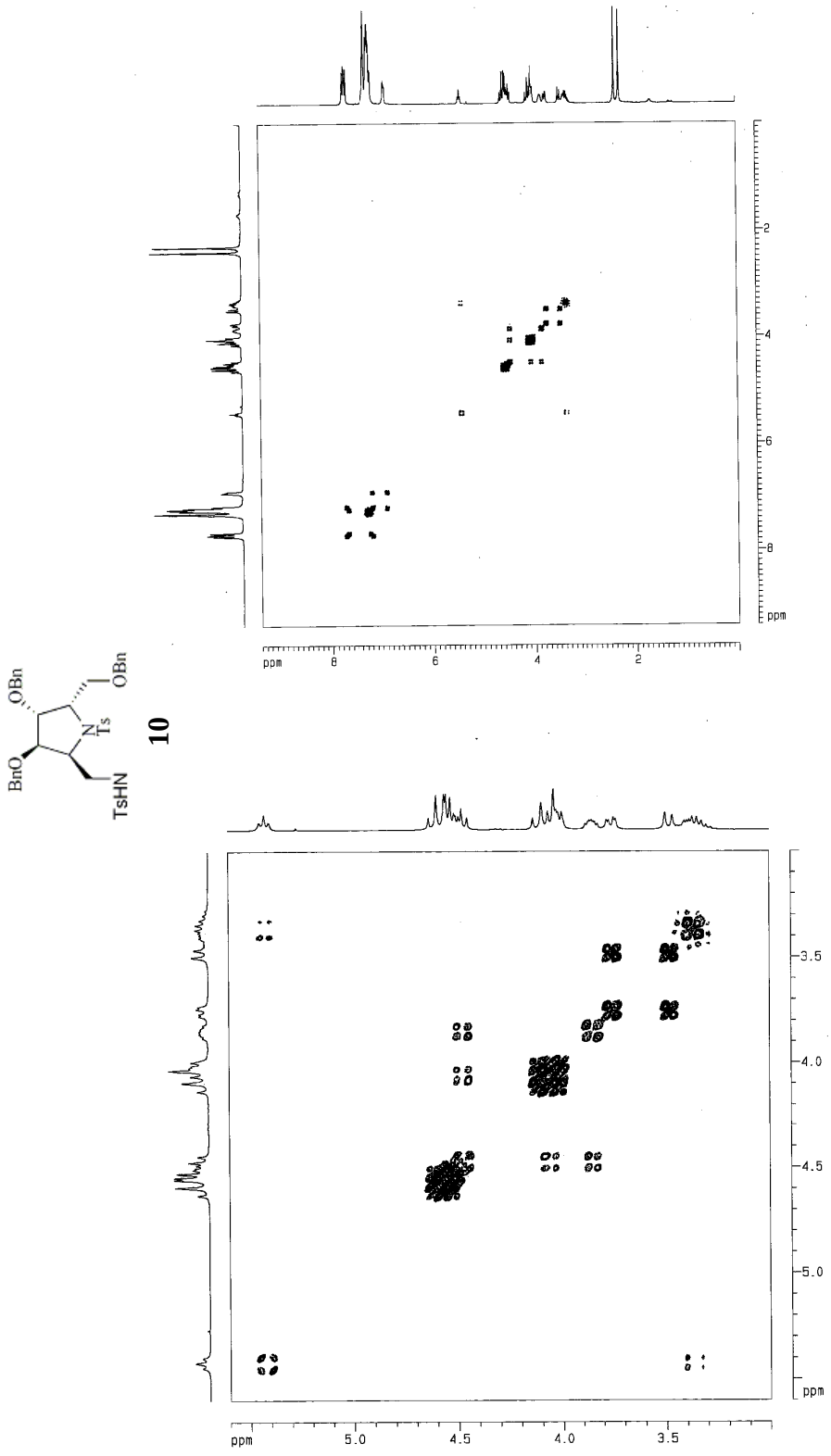

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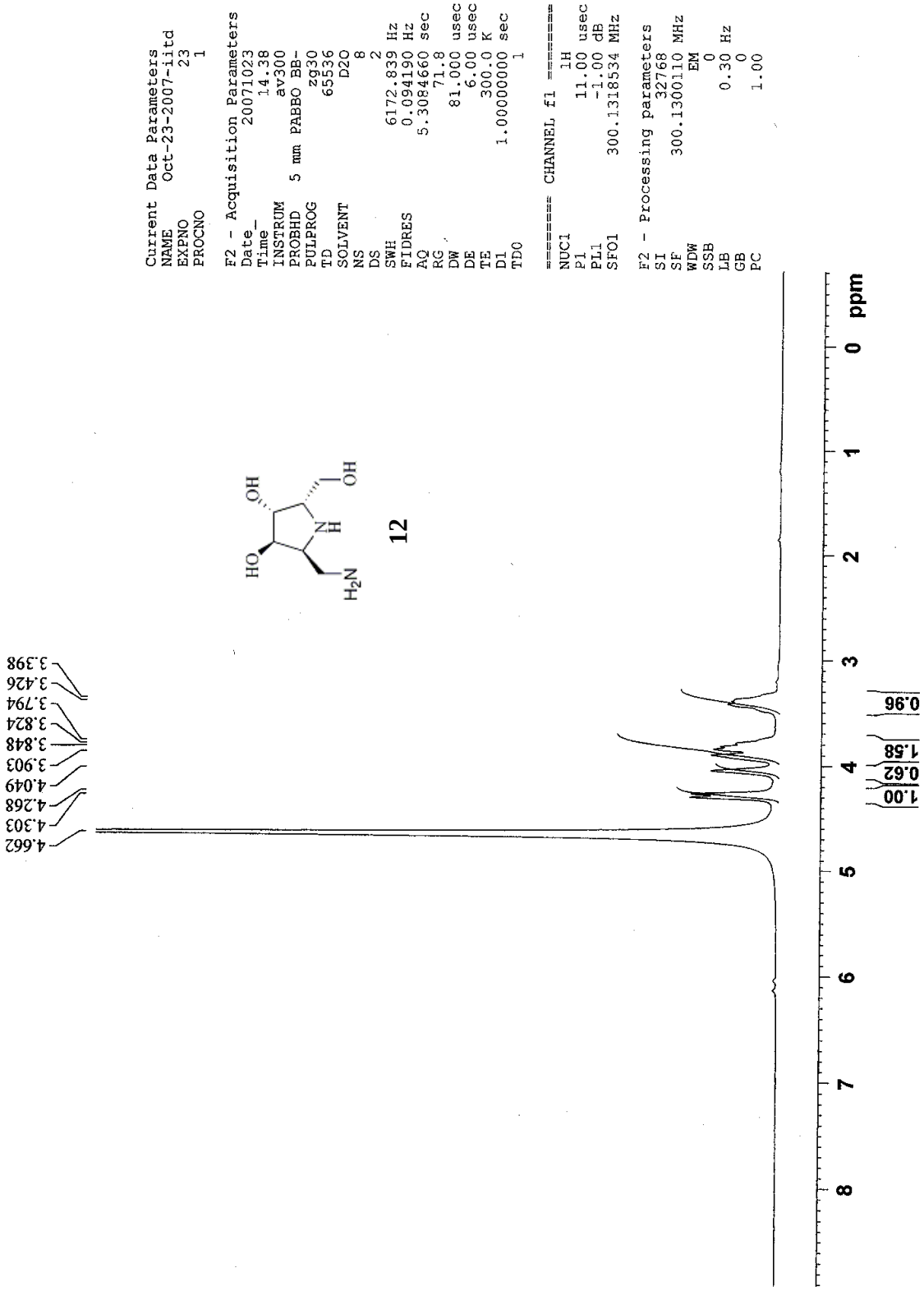
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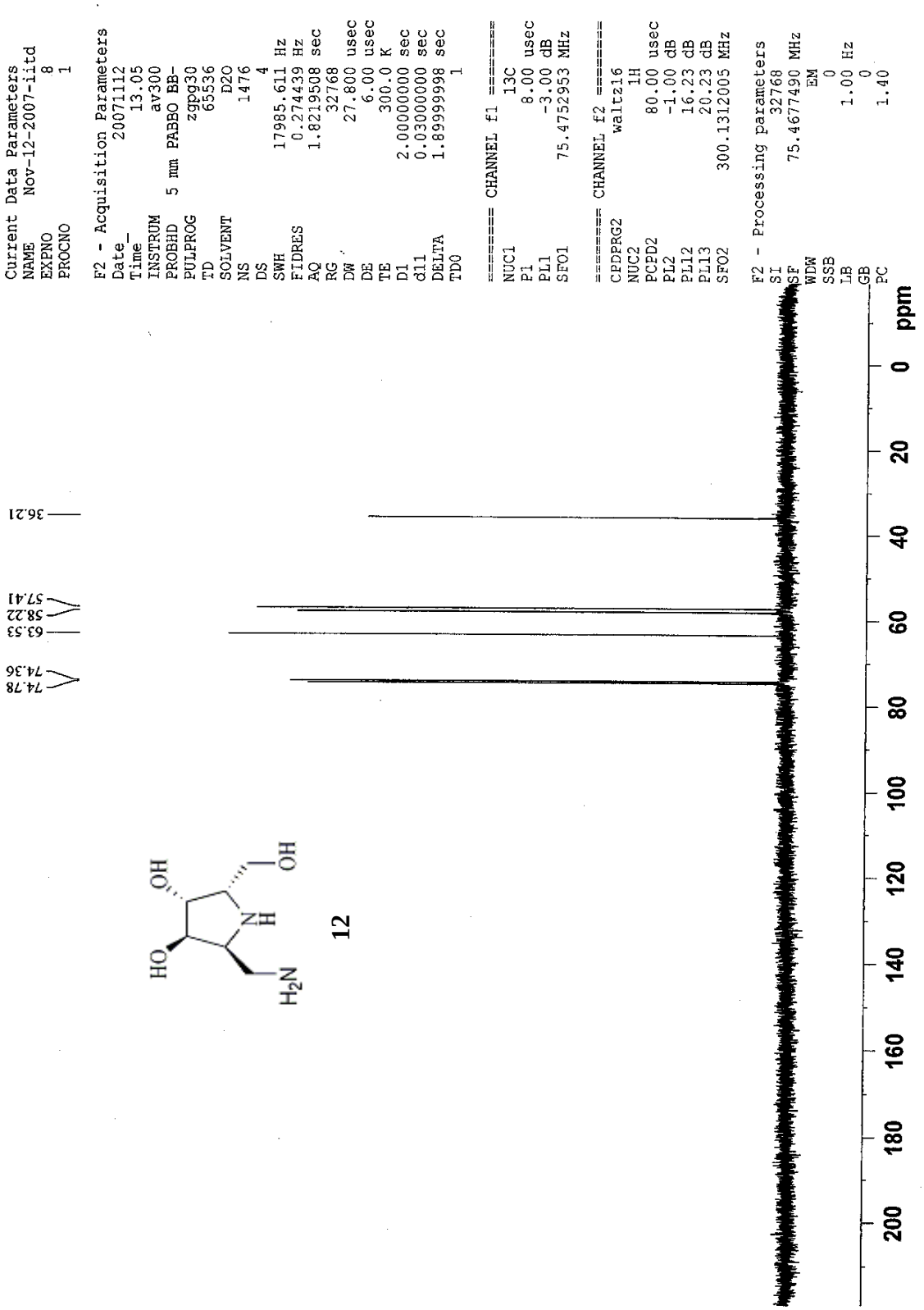
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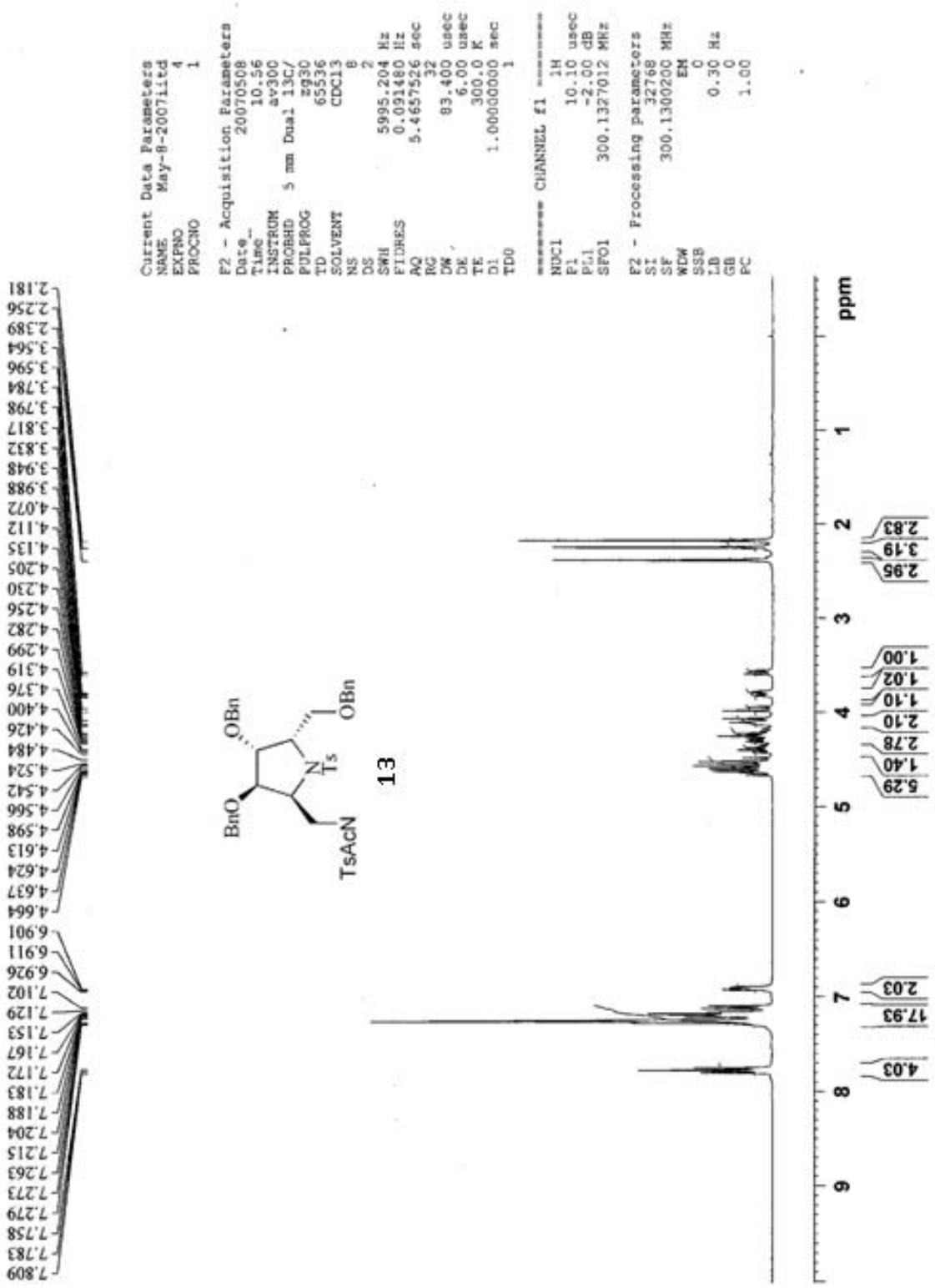

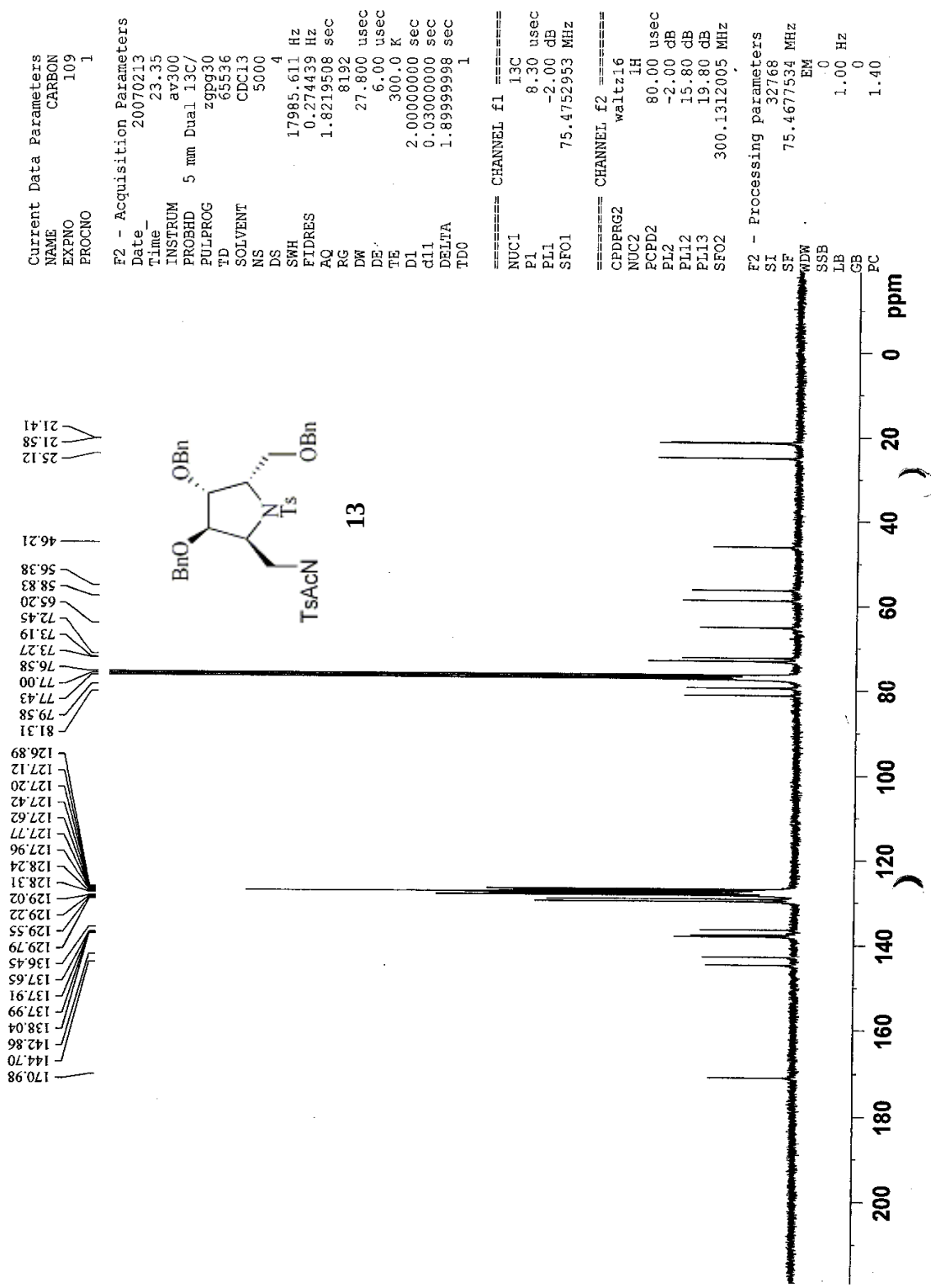
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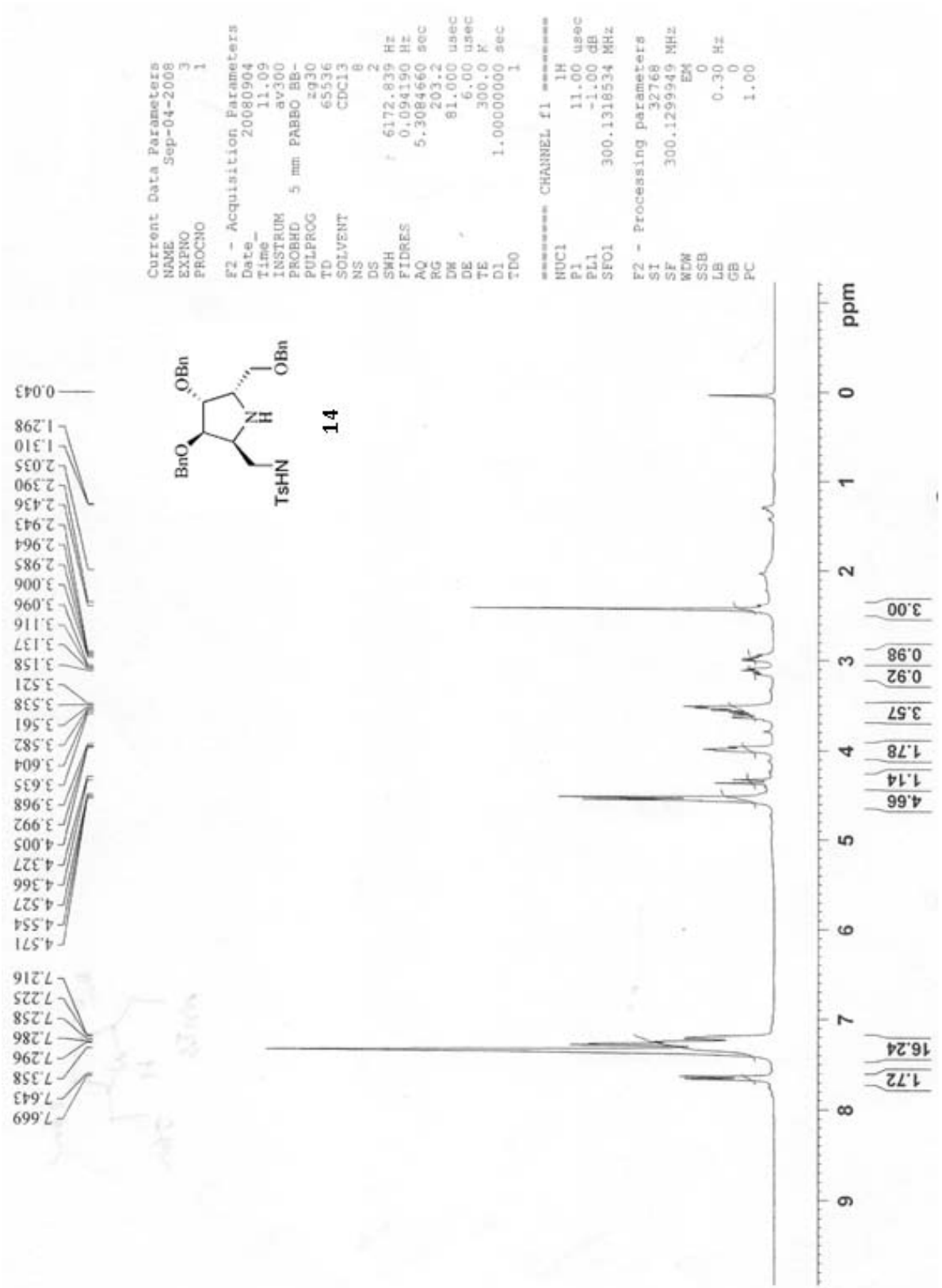
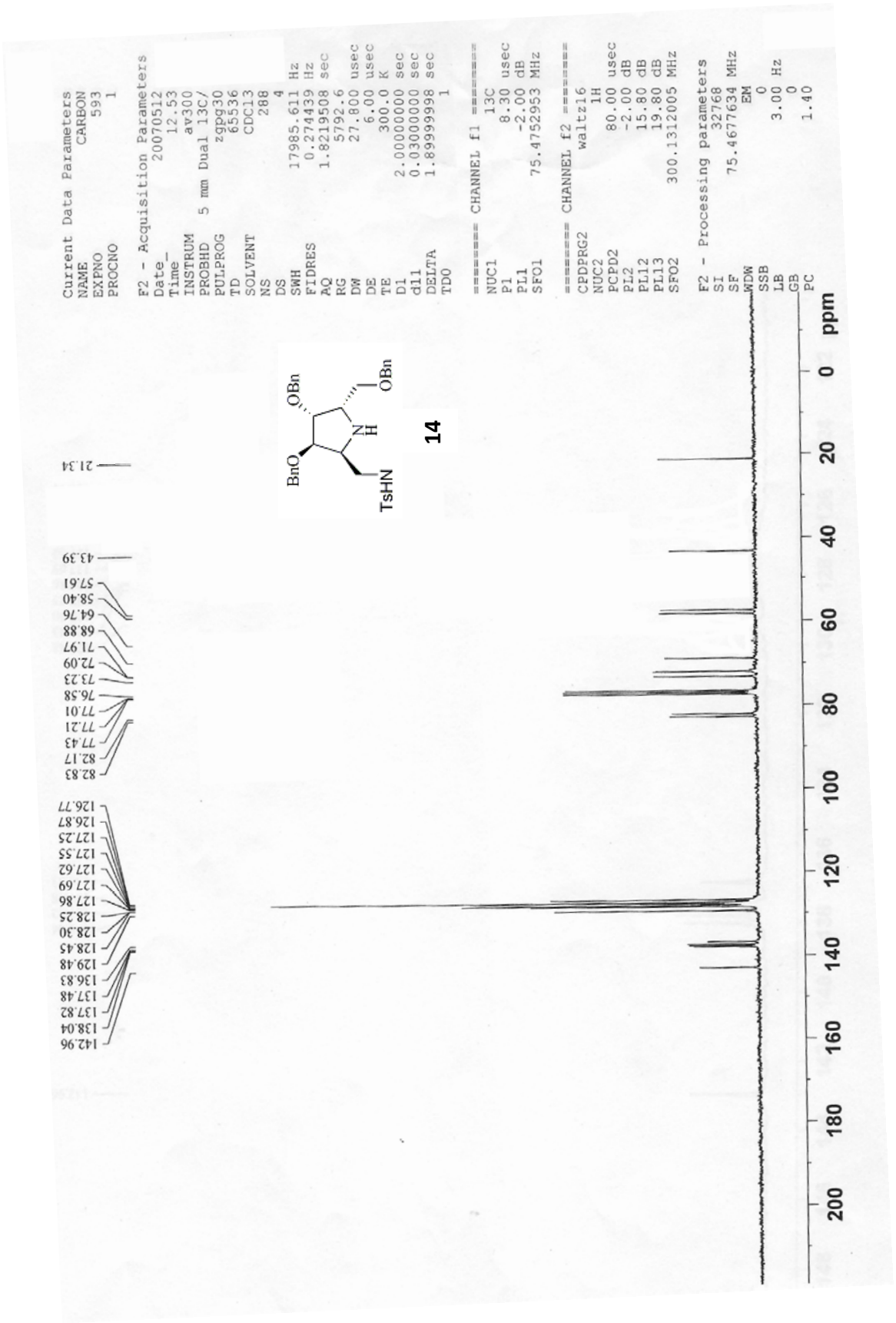
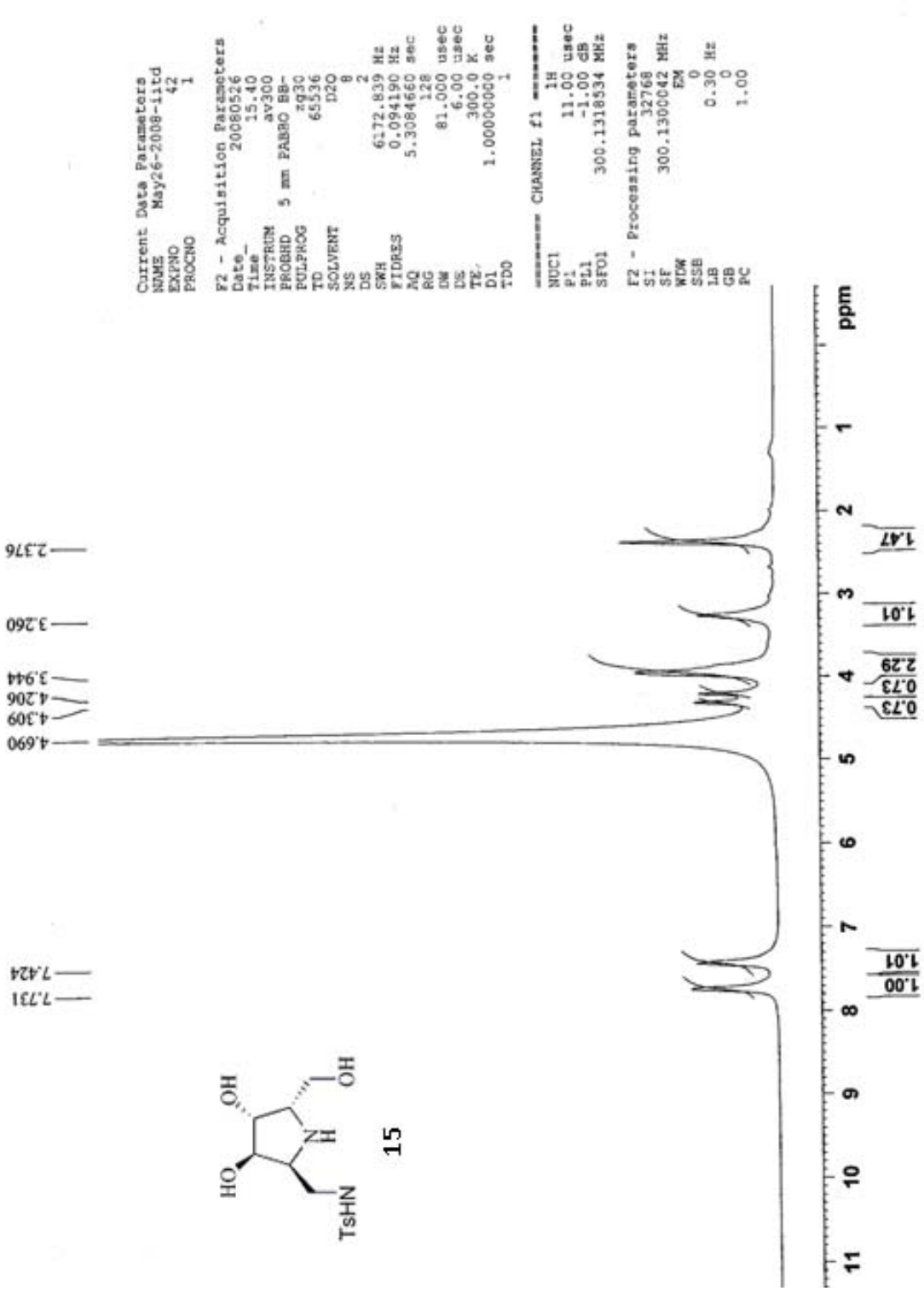
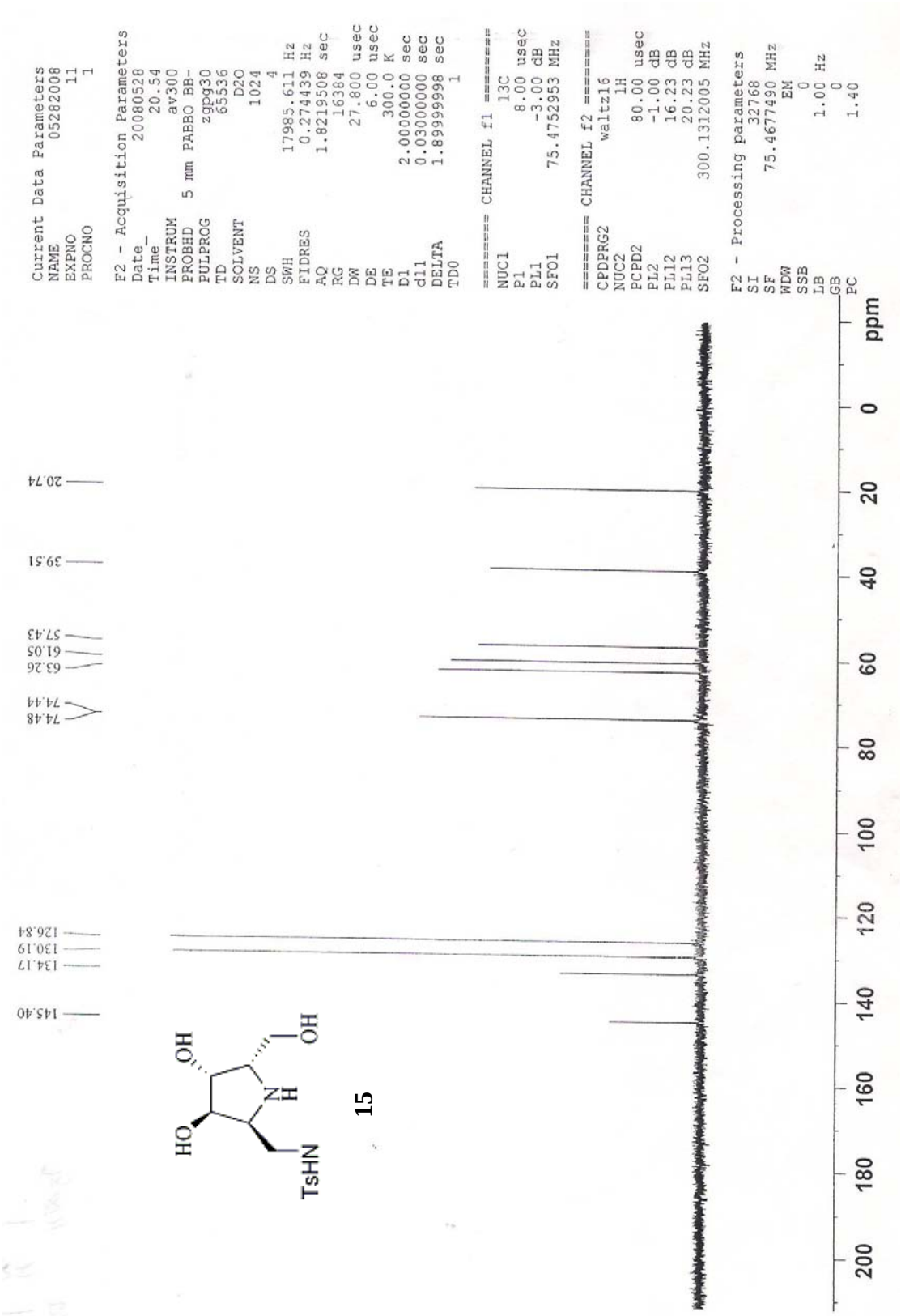
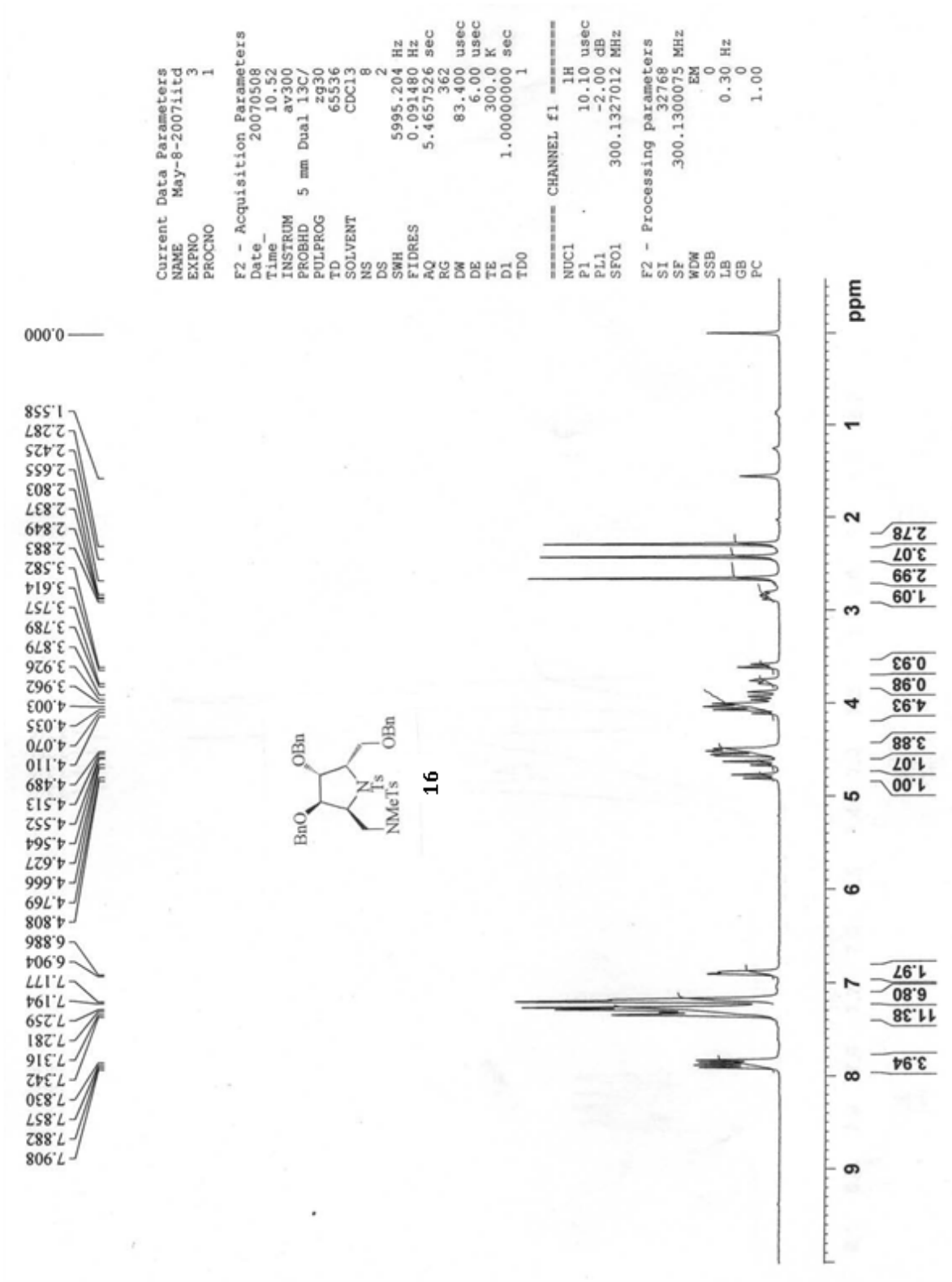
144.70
170.98
200
180
160
140
120
100
80
60
40
20
0
ppm

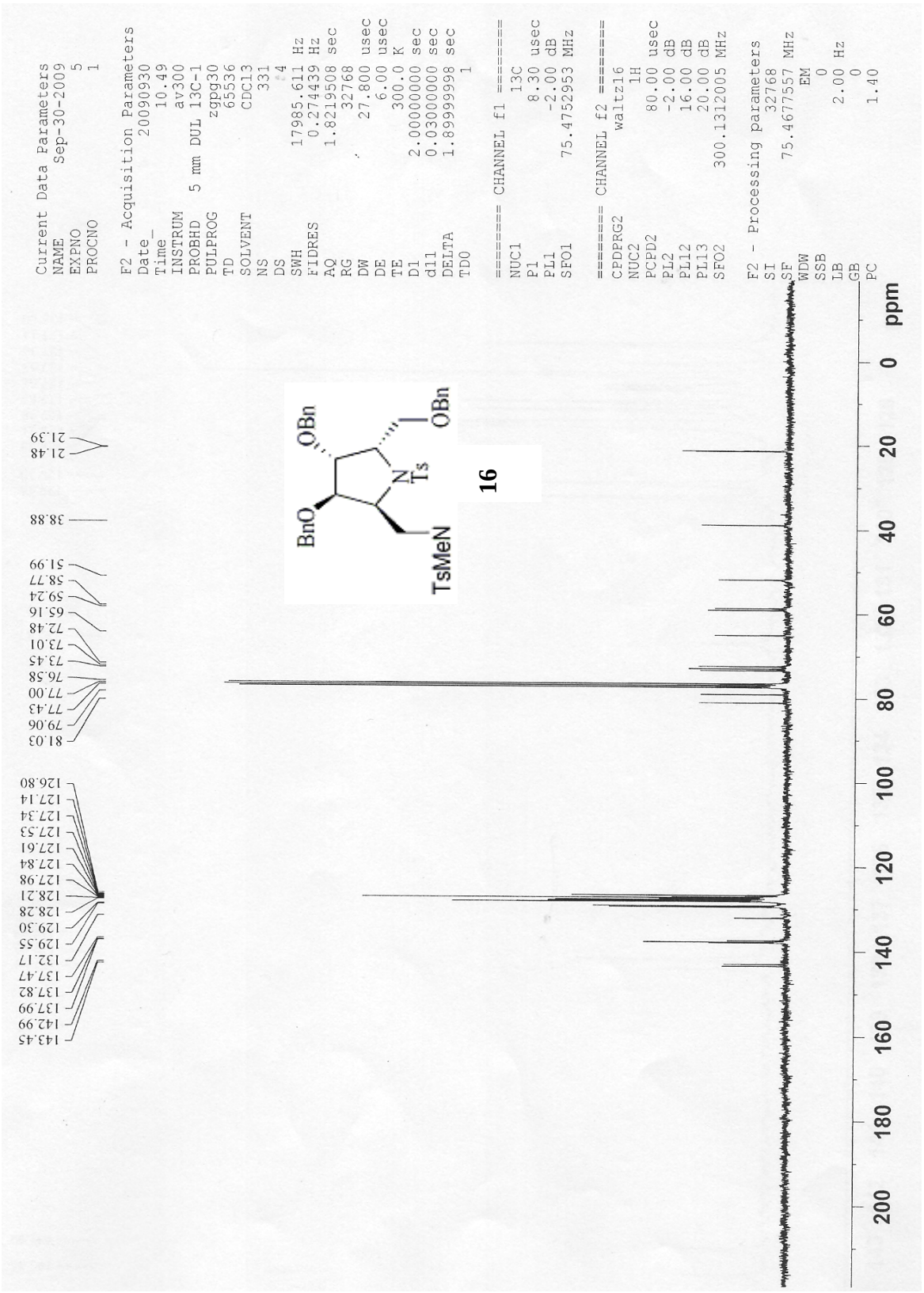


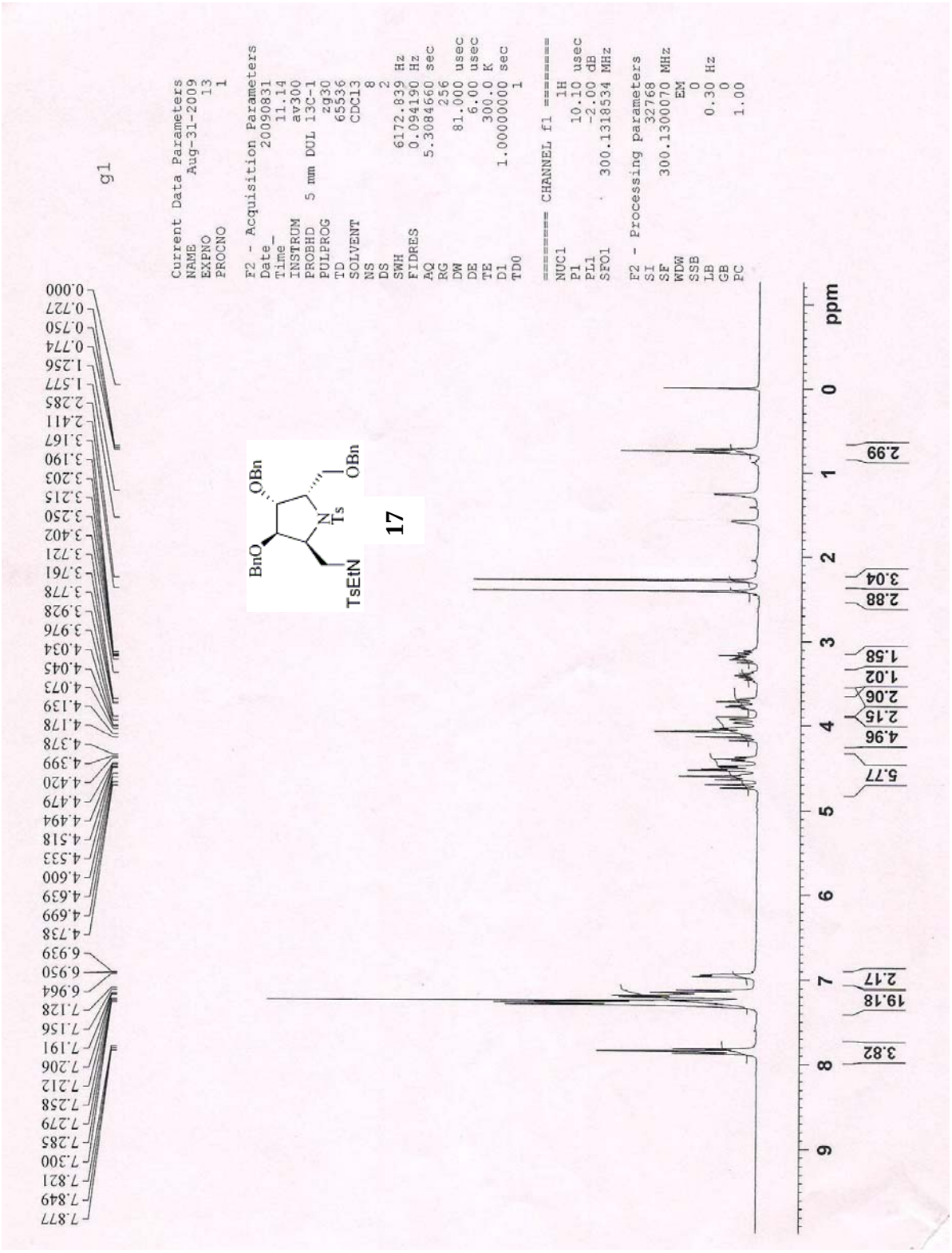

Current Data Parameters
NAME CARBON
EXPNO 593
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070512
Time_ 12.53
INSTRUM av300
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 288
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 5792.6
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.30 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.80 dB
PL13 19.80 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677634 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OBn
OBn
BnO
TsHN
14
21.34
43.39
57.61
58.40
64.76
68.88
71.97
72.09
73.23
76.58
77.01
77.21
77.43
82.17
82.83
126.77
126.87
127.25
127.55
127.62
127.69
127.86
128.25
128.30
128.45
129.48
136.83
137.48
137.82
138.04
142.96
200
180
160
140
120
100
80
60
40
20
0
ppm

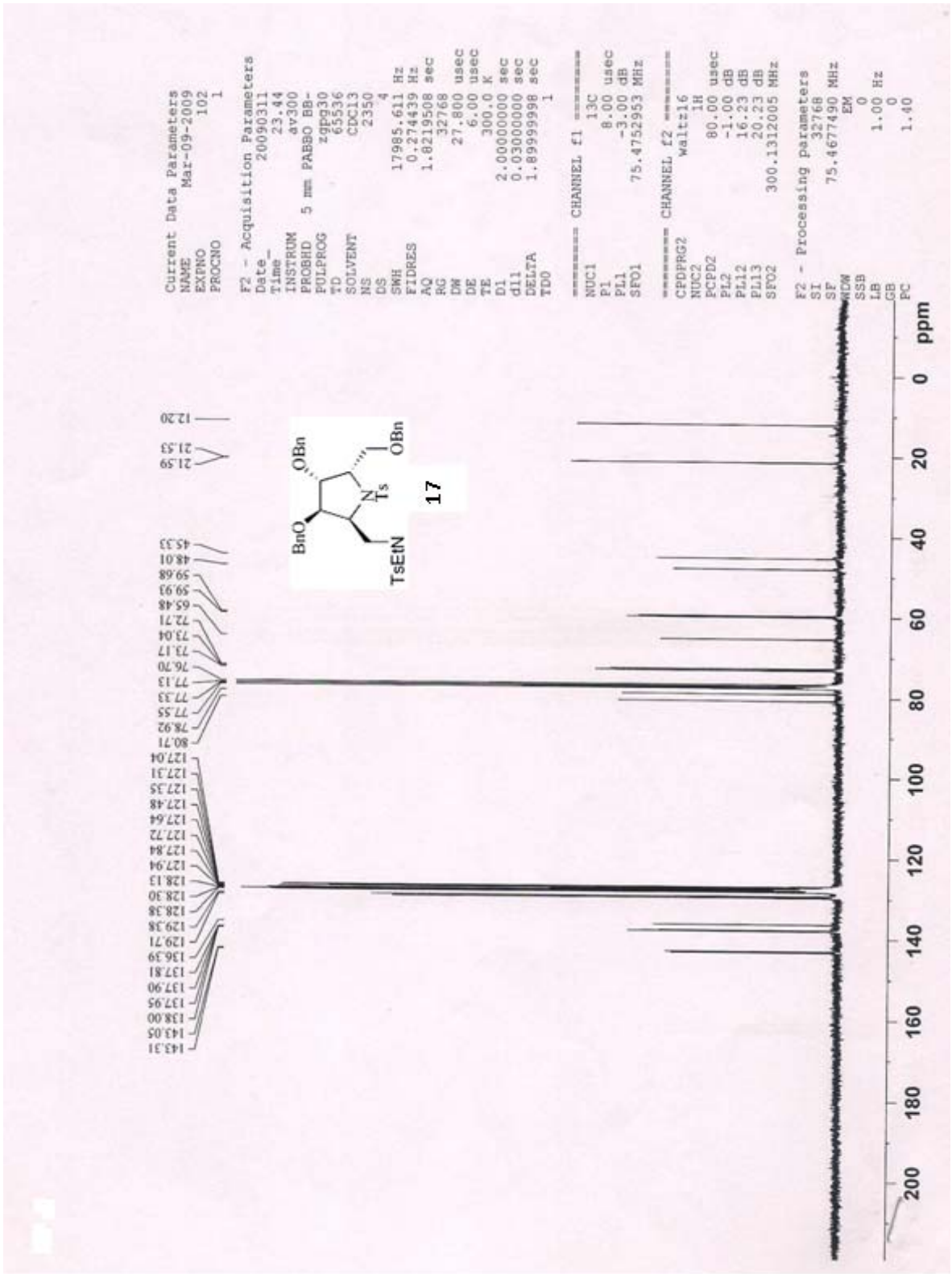


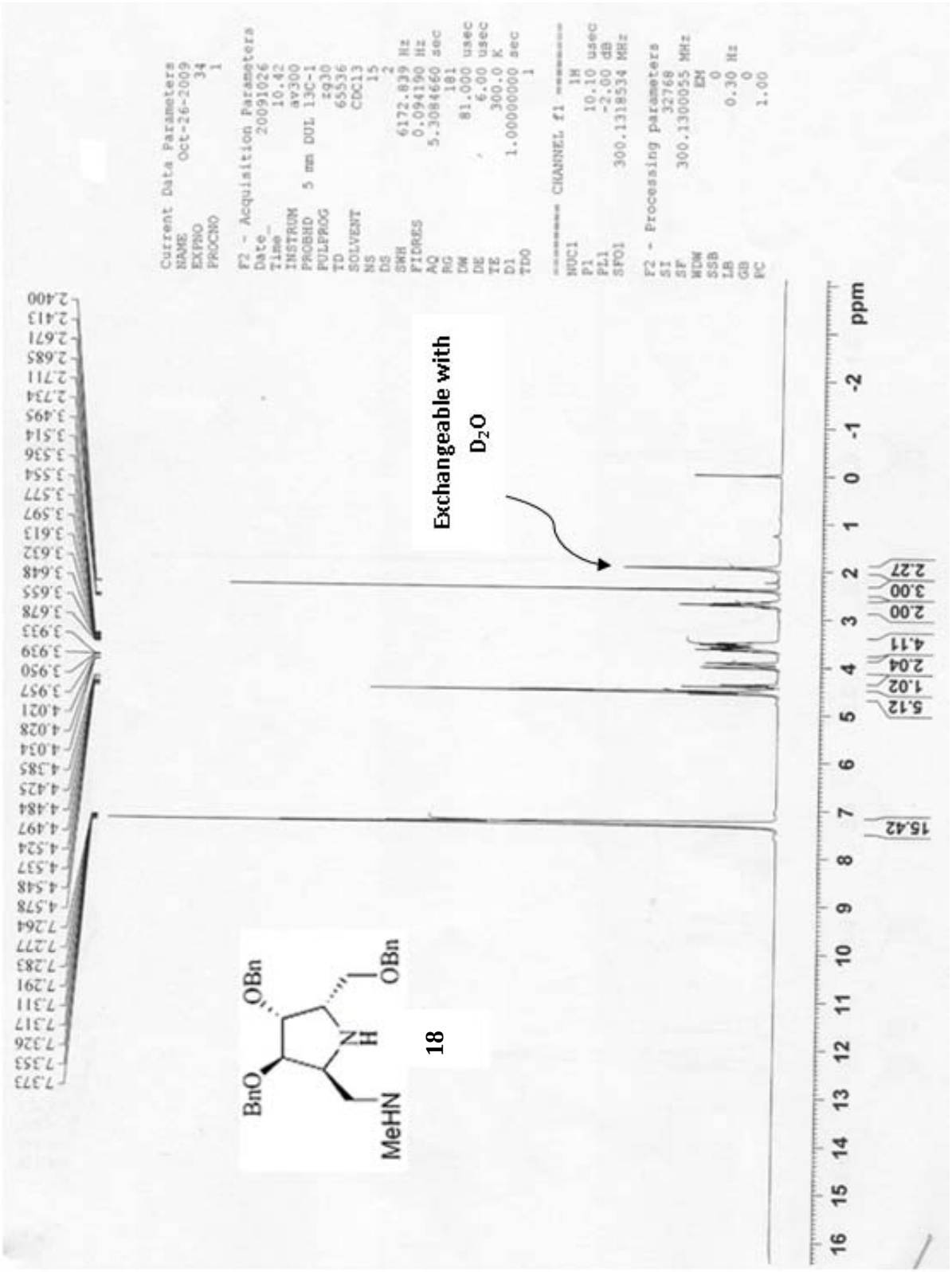

Current Data Parameters
NAME 05282008
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080528
Time 20.54
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 16.23 dB
PL13 20.23 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
20.74
39.51
57.43
61.05
63.26
74.44
74.48
126.84
130.19
134.17
145.40
OH
HO
N
H
OH
TsHN
15
200
180
160
140
120
100
80
60
40
20
0
ppm

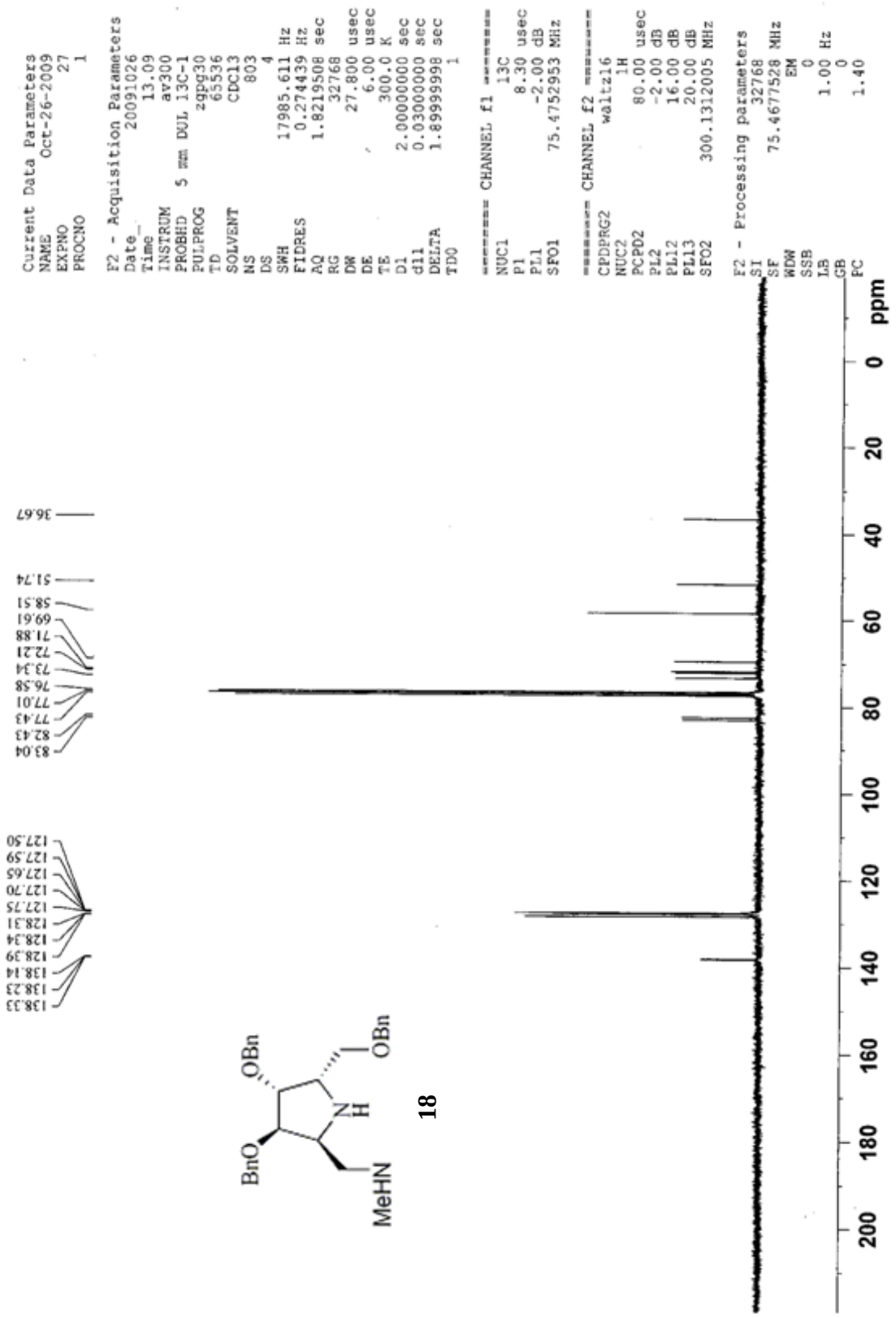

Current Data Parameters
NAME May-8-2007iitd
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070508
Time_ 10.52
INSTRUM av300
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 362
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
SFO1 300.1327012 MHz
F2 - Processing parameters
SI 32768
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OBn
OBn
BnO
NMeTs
Ts
16
ppm
2.78
3.07
2.99
1.09
0.93
0.98
4.93
3.88
1.07
1.00
1.97
6.80
11.38
3.94


Current Data Parameters
NAME Sep-30-2009
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090930
Time_ 10.49
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 331
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 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 8.30 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677557 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
OBn
OBn
BnO
N
Ts
TsMeN
16
21.39
21.48
38.88
51.99
58.77
59.24
65.16
72.48
73.01
73.45
76.58
77.00
77.43
79.06
81.03
126.80
127.14
127.34
127.53
127.61
127.84
127.98
128.21
128.28
129.30
129.55
132.17
137.47
137.82
137.99
142.99
143.45
200
180
160
140
120
100
80
60
40
20
0
ppm

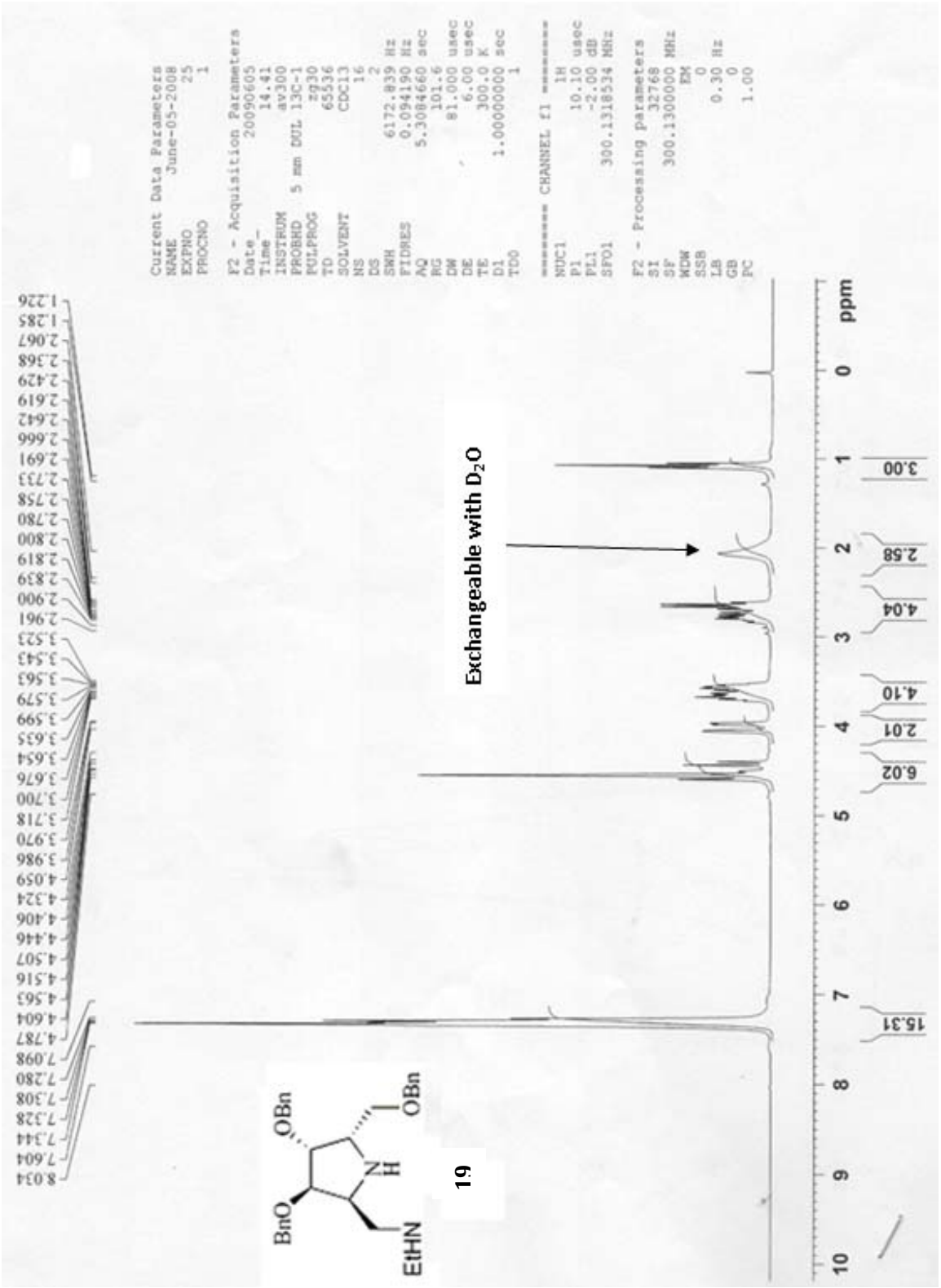

g1
Current Data Parameters
NAME Aug-31-2009
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090831
Time_ 11.14
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BnO
OBn
OBn
N
Ts
TsEtN
17
ppm
0.000
0.727
0.750
0.774
1.256
1.577
2.285
2.411
3.167
3.190
3.203
3.215
3.250
3.402
3.721
3.761
3.778
3.928
3.976
4.034
4.045
4.073
4.139
4.178
4.378
4.399
4.420
4.479
4.494
4.518
4.533
4.600
4.639
4.699
4.738
6.939
6.950
6.964
7.128
7.156
7.191
7.206
7.212
7.258
7.279
7.285
7.300
7.821
7.849
7.877
2.99
3.04
2.88
1.58
1.02
2.06
2.15
4.96
5.77
19.18
2.17
3.82

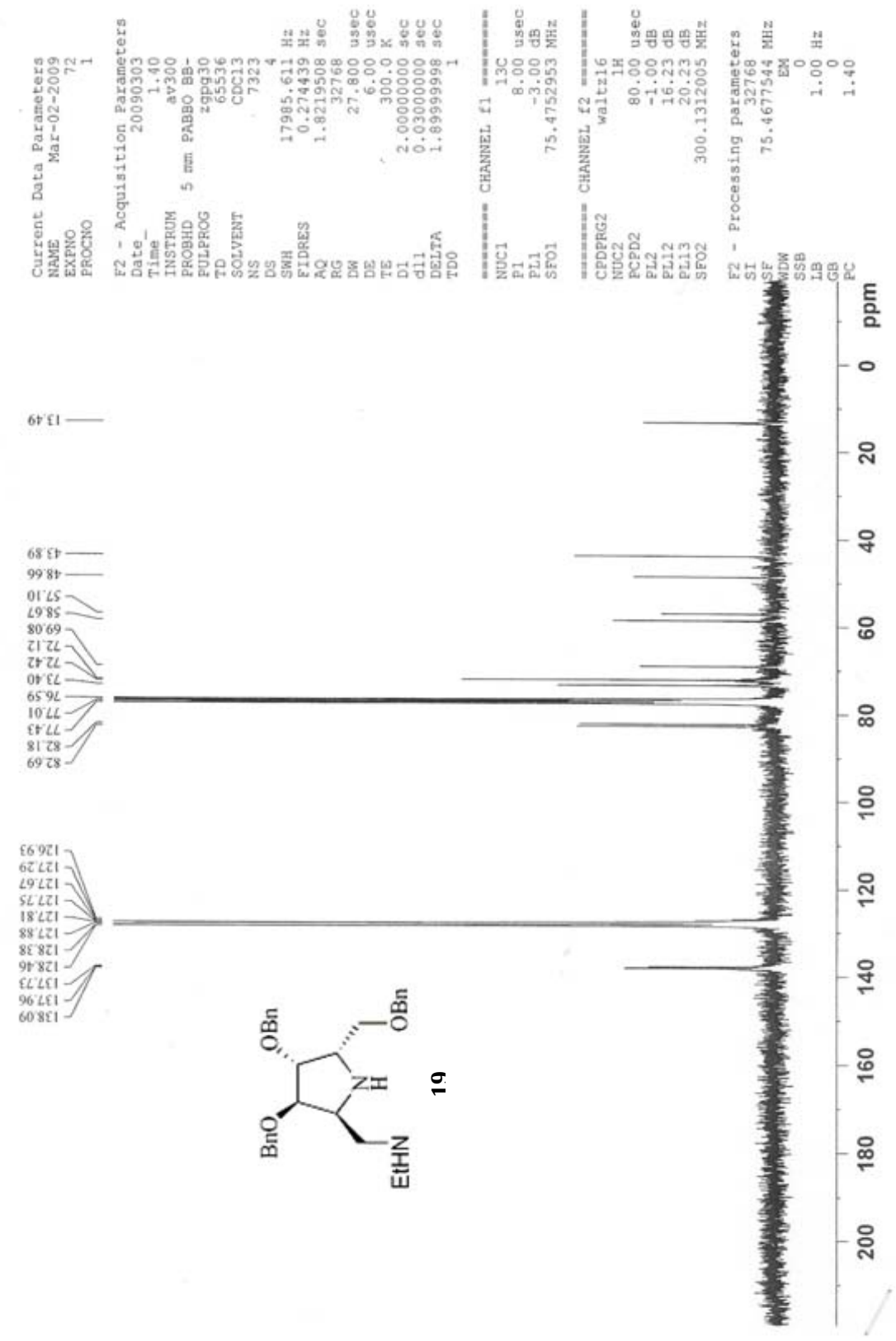

OBn
BnO
N
Ts
OBn
TsEtN
17

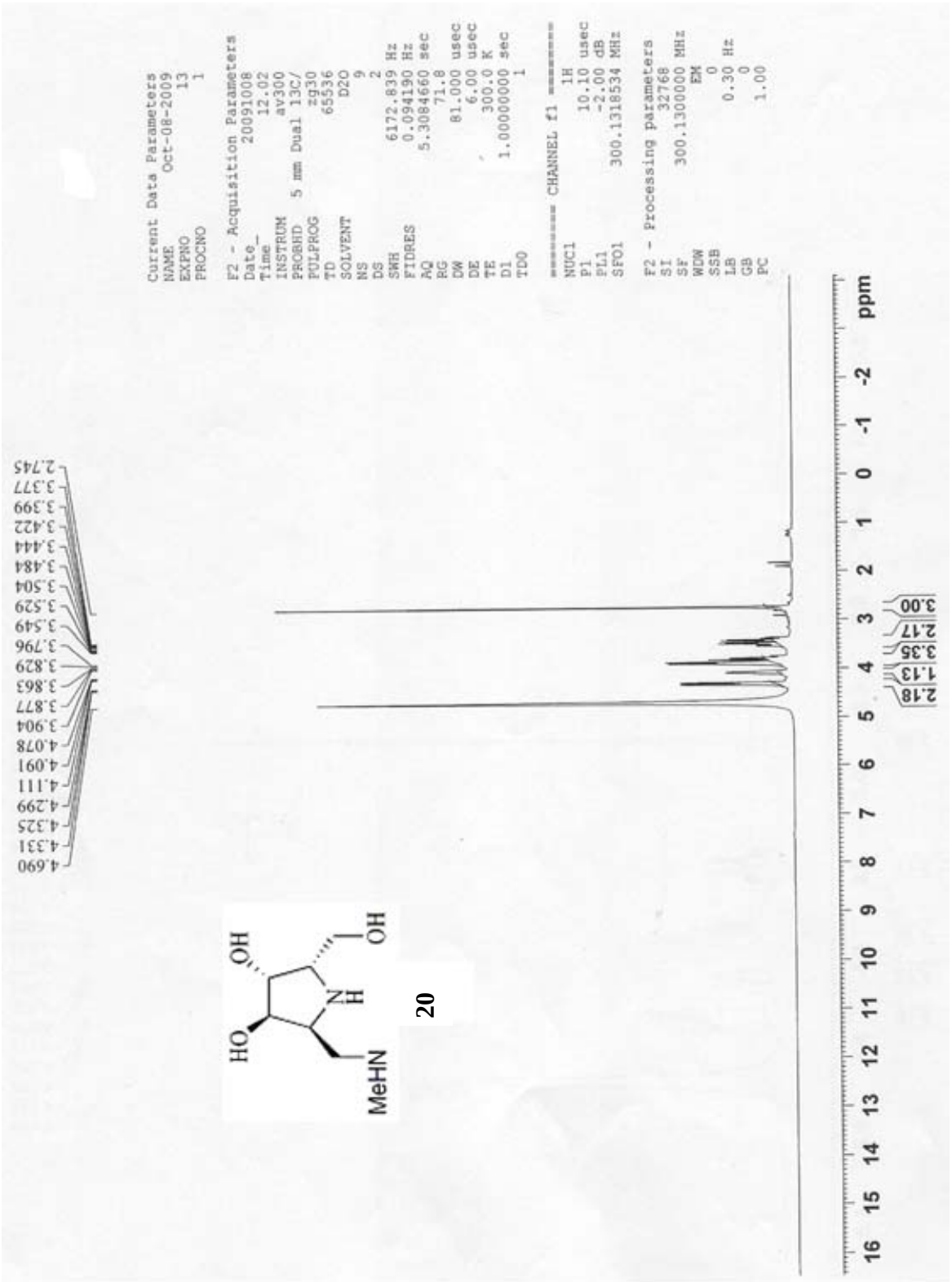

Exchangeable with
D2O
OBn
OBn
BnO
MeHN
18
ppm

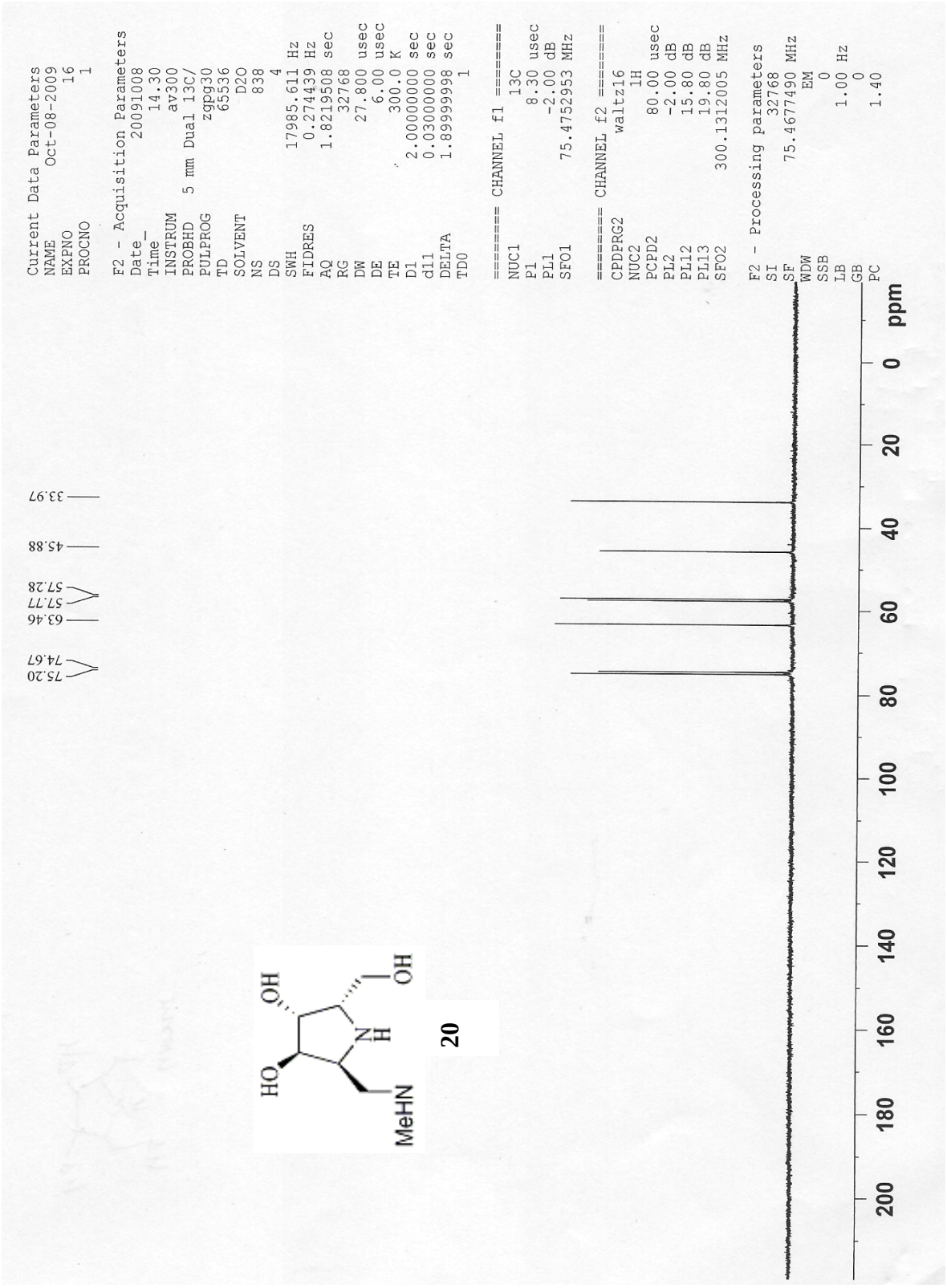

Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
d11
DELTA
TD0
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
======== CHANNEL f2 ========
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
ppm
36.67
51.74
58.51
69.61
71.88
72.21
73.34
76.58
77.01
77.43
82.43
83.04
127.50
127.59
127.65
127.70
127.75
128.31
128.34
128.39
138.14
138.23
138.33
OBn
OBn
BnO
MeHN
H
N

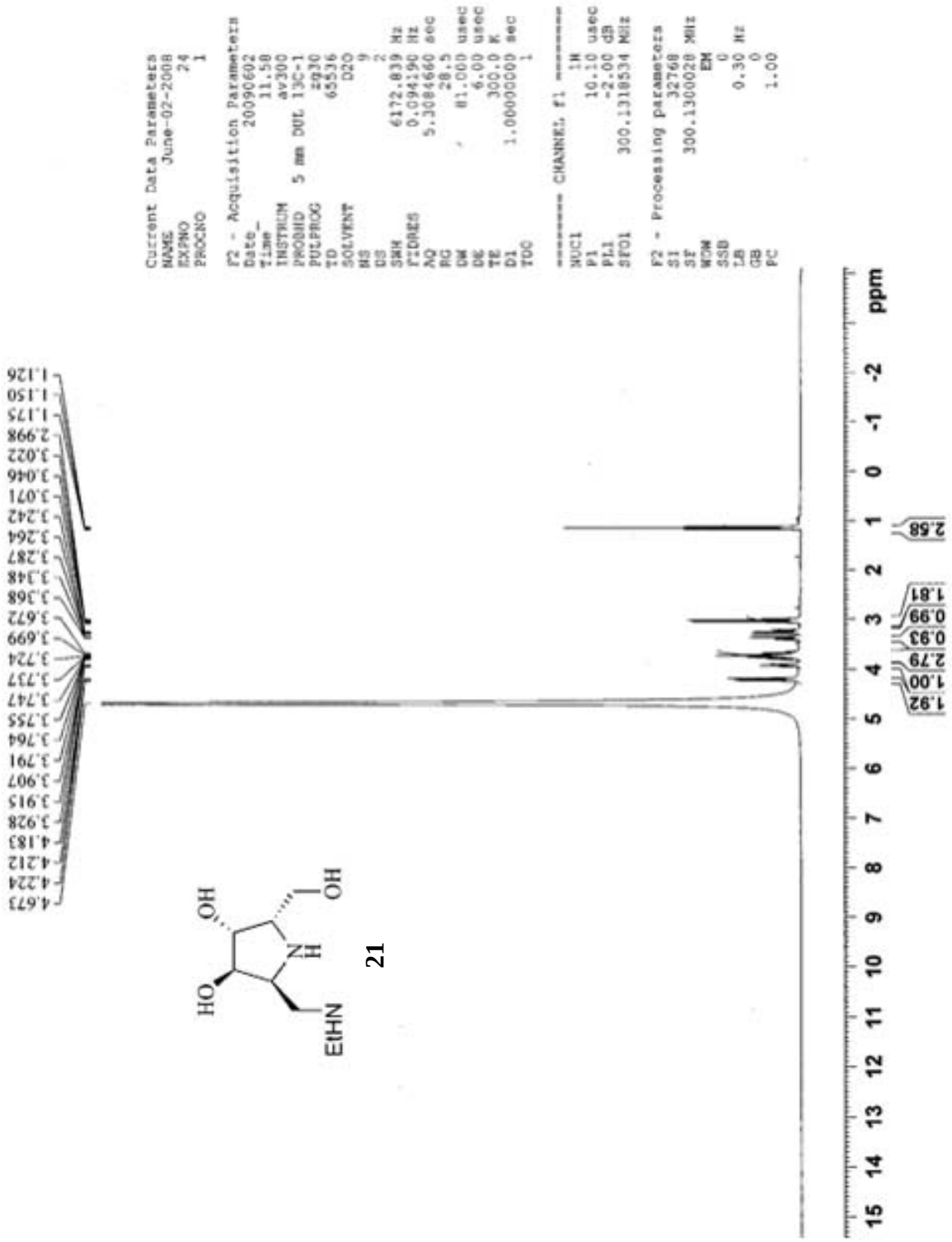
18

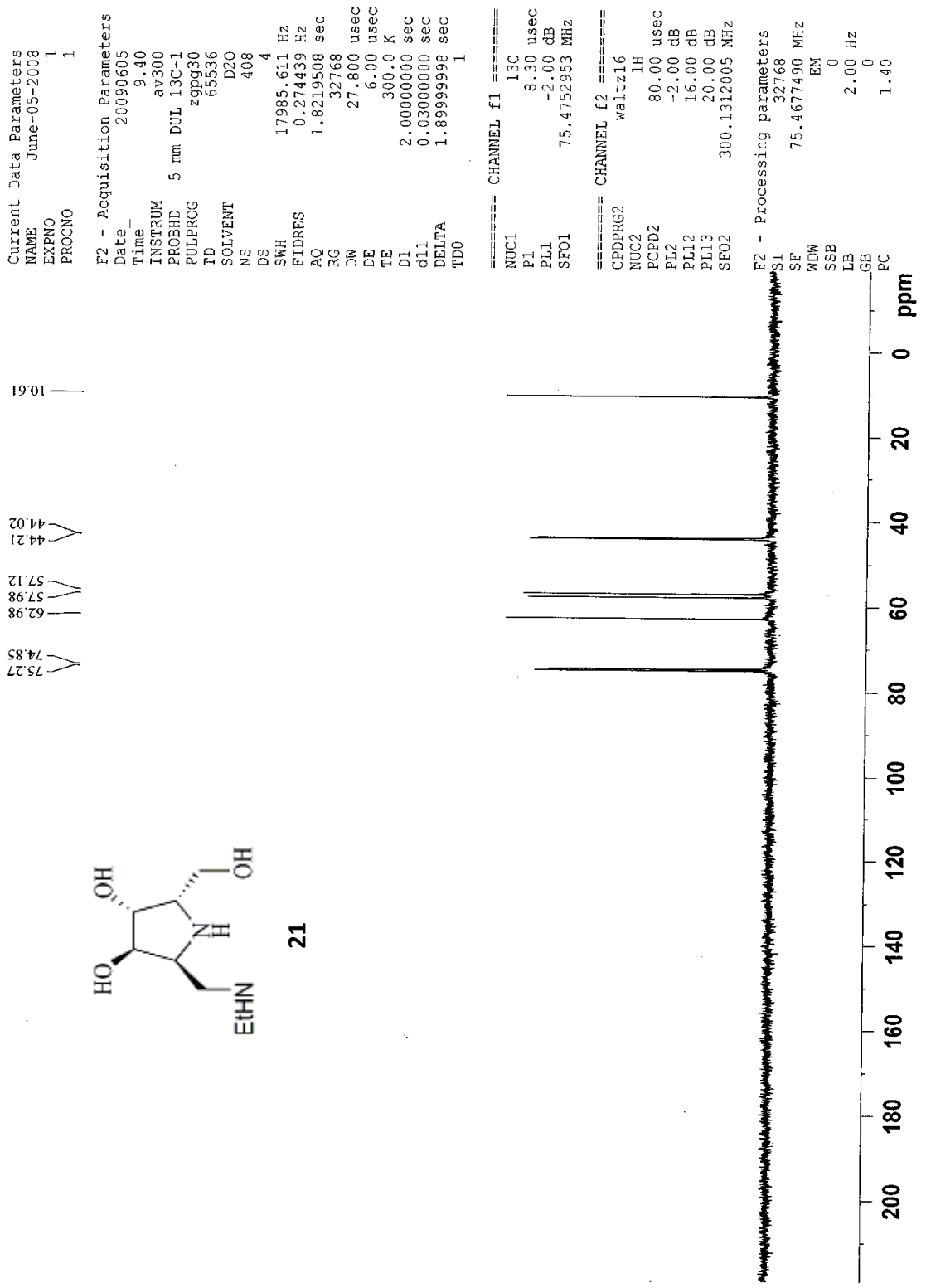

Exchangeable with D_2O
19
OBn
OBn
BnO
EtHN
N
H
ppm
3.00
2.58
4.04
4.10
2.01
6.02
15.31

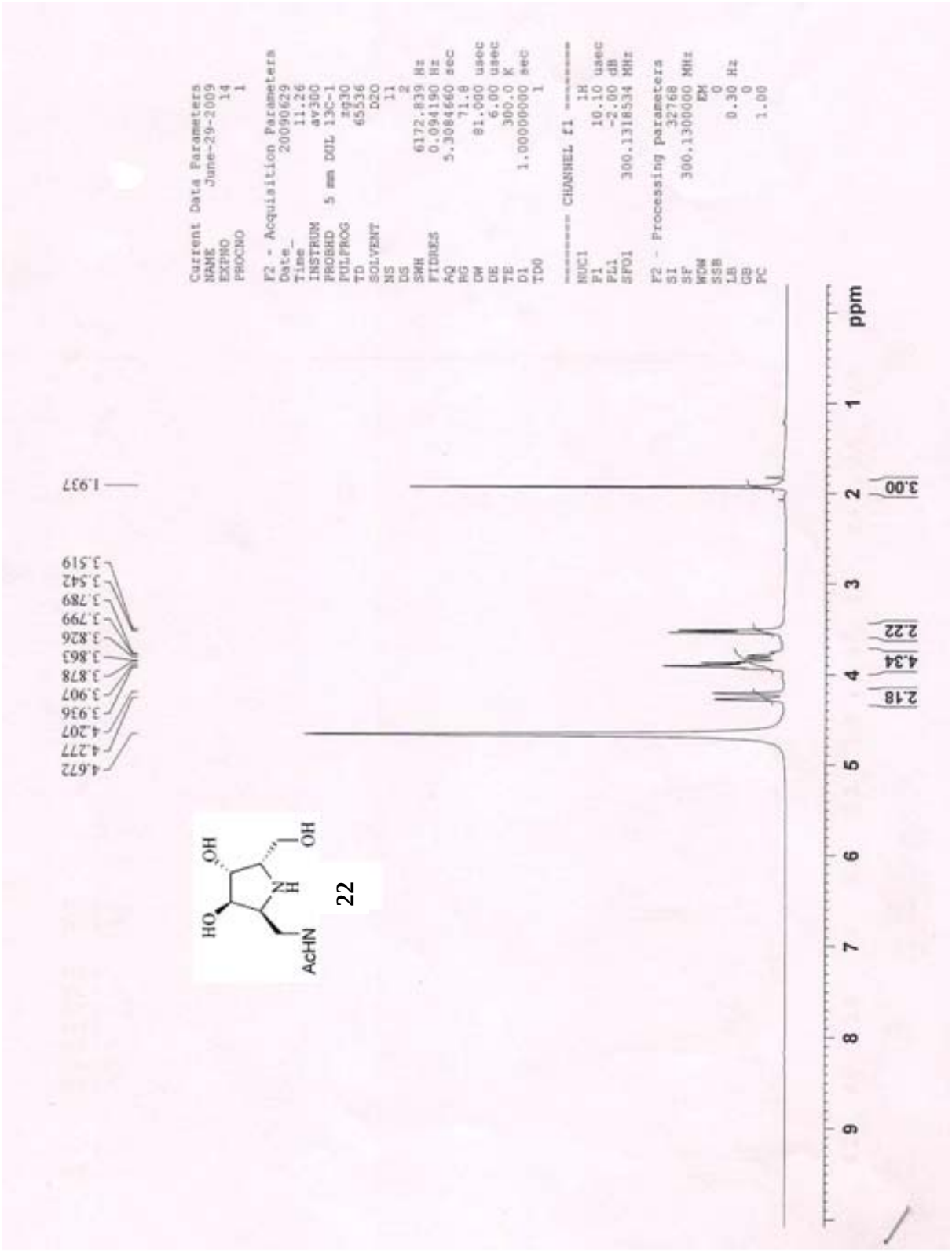


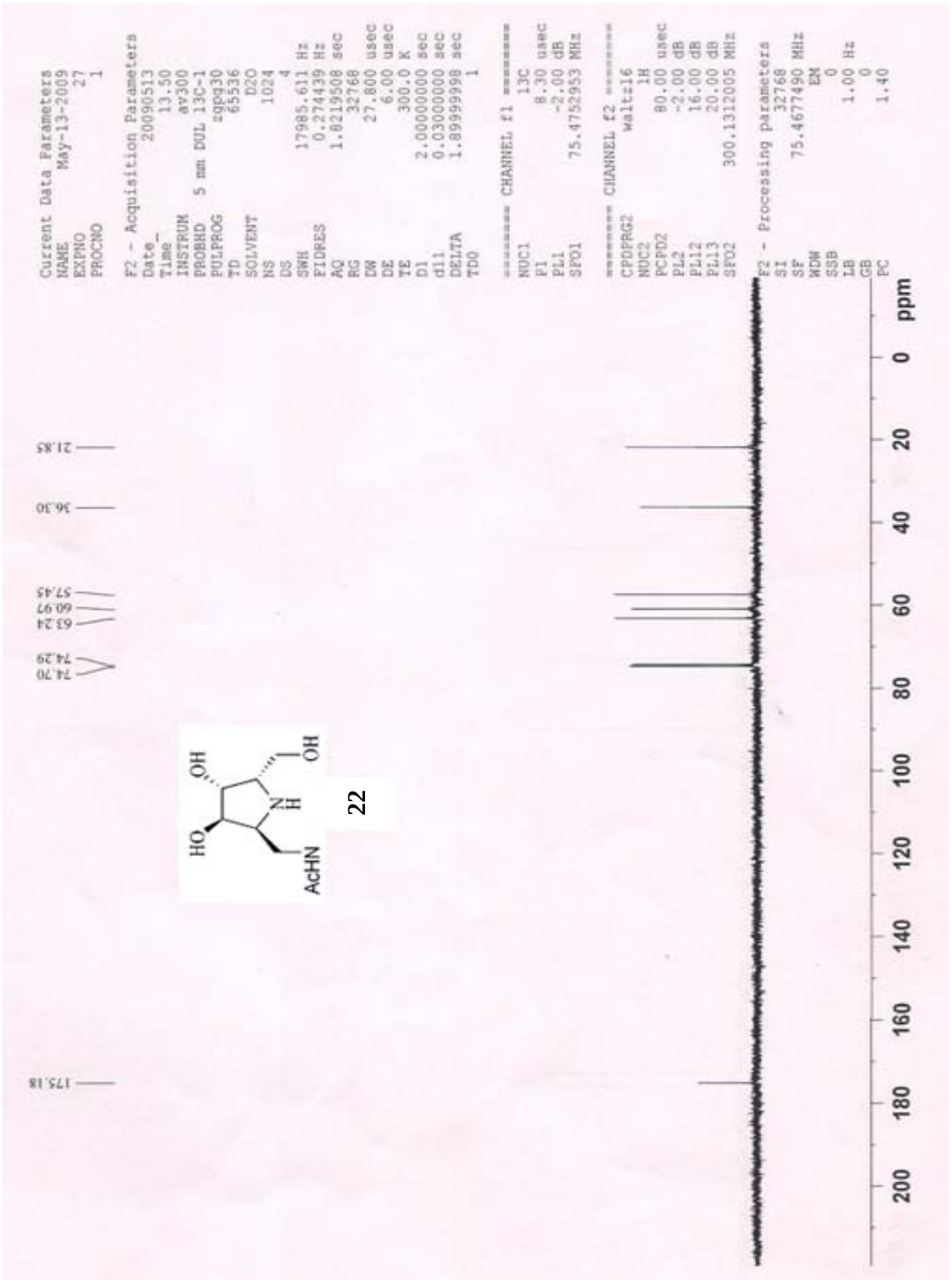

Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
TDO
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
ppm
HO
OH
N
H
OH
MeHN
20


Current Data Parameters
NAME Oct-08-2009
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091008
Time_ 14.30
INSTRUM av300
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 838
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 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 8.30 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.80 dB
PL13 19.80 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
33.97
45.88
57.28
57.77
63.46
74.67
75.20
200 180 160 140 120 100 80 60 40 20 0 ppm
OH
HO
N
H
OH
MeHN
20


Current Data Parameters
NAME June-02-2008
EXPNO 24
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090602
Time 11.50
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT D2O
NS 9
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 28.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
SFO1 300.1310534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.673
4.224
4.212
4.183
3.928
3.915
3.907
3.791
3.764
3.755
3.747
3.737
3.724
3.699
3.672
3.368
3.348
3.287
3.264
3.242
3.071
3.046
3.022
2.998
1.175
1.150
1.126
2.68
1.81
0.99
0.93
2.79
1.00
1.92
ppm
OH
OH
HO
N
H
EtHN
21


Current Data Parameters
NAME June-05-2008
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090605
Time_ 9.40
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 408
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 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 8.30 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
10.61
44.02
44.21
57.12
57.98
62.98
74.85
75.27
200
180
160
140
120
100
80
60
40
20
0
ppm
OH
HO
N
H
OH
EtHN
21


Current Data Parameters
NAME June-29-2009
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090629
Time_ 11:26
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT D2O
NS 11
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.937
3.519
3.542
3.789
3.799
3.826
3.863
3.878
3.907
3.936
4.207
4.277
4.672
3.00
2.22
4.34
2.18
ppm
1
2
3
4
5
6
7
8
9
HO
OH
OH
N
H
AcHN
22


Current Data Parameters
NAME May-13-2009
EXPNO 27
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090513
Time 13.50
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 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 8.30 usec
PL1 -2.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
175.18
74.70
74.29
63.24
60.97
57.45
36.30
21.85
OH
HO
N
H
OH
AcHN
22
200
180
160
140
120
100
80
60
40
20
0
ppm