Electronic Supplementary Information for

Cleavage of unactivated amide bonds by ammonium salt-accelerated hydrazinolysis

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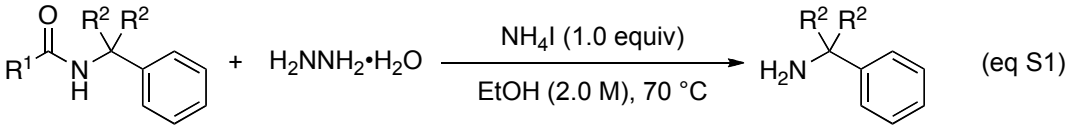
1. Preliminary Experiments for Understanding Steric and Electronic Effects

To understand steric and electronic effects for the hydrazinolysis of amide bonds under our reaction conditions, we performed preliminary experiments described below.

First, we performed reactions of amides with different steric hindrance (eq S1). As a result, sterically less crowded amide **1a** reacted faster than sterically more crowded amides **1u** and **1v** (Fig. S1). Therefore, steric effects are important for the hydrazinolysis of amide bonds under our reaction conditions.



(eq S1)

1a: R^1 = Me, R^2 = H
1u: R^1 = Me, R^2 = Me
1v: R^1 = *t*-Bu, R^2 = H

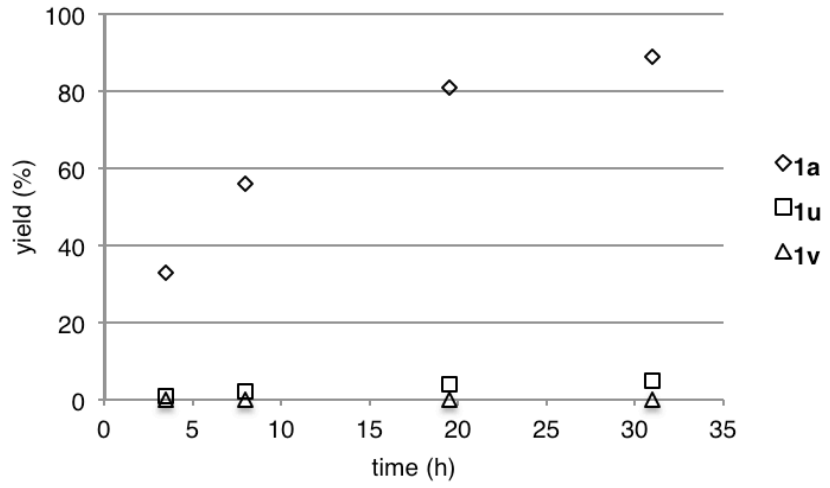
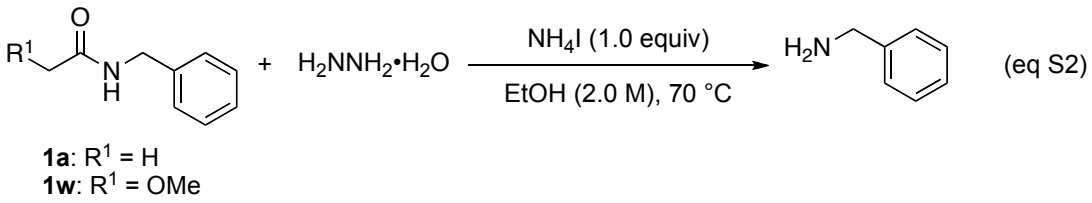


Fig. S1 Time-course experiments for amides with different steric hindrance. Yields were determined by ^{1}H NMR analysis of the crude mixture.

Next, we performed reactions of amides with different electronic properties (eq S2). As a result, electronically more deficient amide **1w** (Taft's polar substituent constant σ^* = 0.60) reacted faster than electronically more rich amide **1a** (σ^* = 0), although steric hindrance of **1w** (Taft's steric substituent constant E_s = –0.19) is larger than that of **1a** (E_s = 0) (Fig. S2). Therefore, electronic effects are also important for the hydrazinolysis of amide

bonds under our reaction conditions.



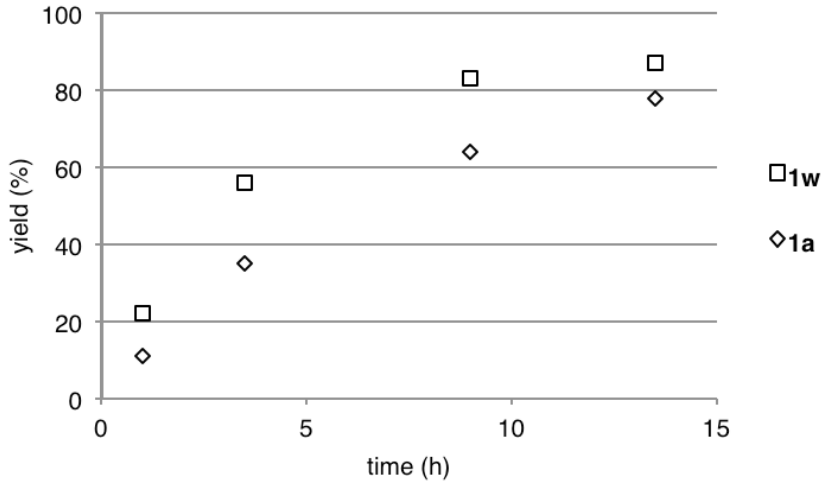


Fig. S2 Time-course experiments for amides with different electronic properties. Yields were determined by ^{1}H NMR analysis of the crude mixture.

Taken together, these results indicate that both steric and electronic effects are important for the hydrazinolysis of amide bonds under our reaction conditions.

2. General Experimental Details

All reactions were conducted in flame-dried glassware under an argon atmosphere unless otherwise noted. Reagents and solvents were obtained from commercial sources and used as received unless otherwise stated. Microwave irradiation reactions were performed with CEM Discover LabMate. Flash silica gel column chromatography was conducted with Merck silica gel 60 (230–400 mesh ASTM) or Kanto Chemical silica gel 60N (spherical neutral, particle size 40–50 μm).

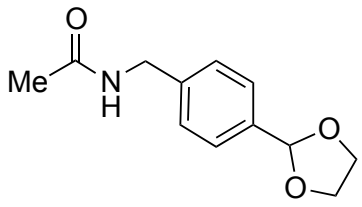
Nuclear magnetic resonance (NMR) spectra were acquired on 400 MHz Varian Unity and 500 MHz Bruker Avance III spectrometers. Chemical shifts are reported in ppm and referenced to tetramethylsilane or residual solvent peaks as internal standards (for $CDCl_3$, tetramethylsilane 0 ppm for ^1H and $CDCl_3$ 77.0 ppm for ^{13}C; for DMSO-*d*6, 2.50 ppm for ^1H and 39.5 ppm for ^{13}C; for C_6D_6, 7.16 ppm for ^1H and 128.06 ppm for ^{13}C). Coupling constants are reported in hertz. The following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Infrared (IR) spectra were recorded with Shimadzu FTIR-8400. High-resolution mass spectroscopy (HRMS) was obtained with Waters ACQUITY UPLC®–LCT-Premier™ XE system. High performance liquid chromatography (HPLC) was performed with JASCO PU-2089plus pump and UV-2075plus detector. Chiral HPLC analysis was performed with DAICEL CHIRALPAK AD-3 column. Optical rotation was measured with JASCO P2200 polarimeter. Melting points were measured by Yanaco Micro Melting Point System MP-J3 and are uncorrected.

3. Preparation of Substrates

Amide **1a** [CAS Registry No. 588-46-5] and anilides **1j** [CAS Registry No. 104-04-1], **1n** [CAS Registry No. 103-90-2] and **1o** [CAS Registry No. 122-80-5] were purchased from Tokyo Chemical Industry Co. Ltd. Amides **1e** [CAS Registry No. 14028-67-2][1], **1f** [CAS Registry No. 82342-56-1][1], **1g** [CAS Registry No. 796053-27-5][2], **1i** [CAS Registry No. 5464-77-7][3], **1t** [CAS Registry No. 13343-62-9][4], **1u** [CAS Registry No. 79649-68-6], **1v** [CAS Registry No. 26209-45-0][5], **1w** [CAS Registry No. 2945-05-3] and anilides **1h** [CAS Registry No. 864424-24-8][6], **1l** [CAS Registry No. 16375-88-5][2], **1m** [CAS Registry No. N/A][7] were prepared by conventional procedure from the corresponding amines or anilines with carboxylic acid chlorides or anhydrides in the presence of base, or according to the procedure known in the literature.

Preparation Method and Characterization Data of Substrates Unknown in the Literature

***N*-(4-(1,3-Dioxolan-2-yl)benzyl)acetamide (1b) [CAS Registry No. 738615-92-4][8]**



1b

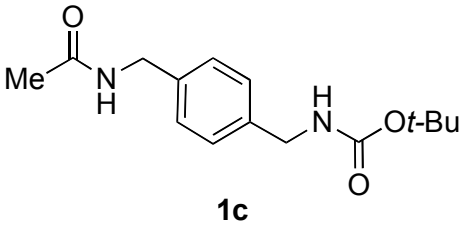
(4-(1,3-Dioxolan-2-yl)phenyl)methanamine[9] (1.31 g, 7.30 mmol) and Et_3N (1.1 mL, 8.0 mmol) were dissolved in dichloromethane (20 mL) followed by dropwise addition of acetyl chloride (0.52 mL, 7.3 mmol) at 0 ºC. The resulting mixture was stirred at room temperature overnight. The reaction mixture was diluted with dichloromethane and washed three times with saturated aqueous $NaHCO_3$. The combined organic layer was dried over Na_2SO_4, filtered and purified by flash silica gel column

[1] C. Aubert, C. Huard-Perrio and M.-C. Lasne, *J. Chem .Soc., Perkin Trans. 1*, 1997, 2837.
[2] Y. Shimizu, H. Morimoto, M. Zhang and T. Ohshima, *Angew. Chem. Int. Ed*., 2012, **51**, 8564.
[3] C. Borel, L. S. Hegedus, J. Krebs and Y. Satoh, *J. Am. Chem. Soc*., 1987, **109**, 1101.
[4] S. Dumbre, A. Derouaux, E. Lescrinier, A. Piette, B. Joris, M. Terrak and P. Herdewijin, *J. Am. Chem. Soc.*, 2012, **134**, 9343.
[5] H. Ueki and V. A. Soloshonok, *Org. Lett*., 2009, **11**, 1797.
[6] E. T. Nadres and O. Daugulis, *J. Am. Chem. Soc.*, 2012, **134**, 7.
[7] H. Suga, N. Tanimoto, A. J. Sinskey and S. Masamune, *J. Am. Chem. Soc.*, 1994, **116**, 11197.
[8] A. Obreza, M. Stegnar and U. Urleb, *Pharmazie*, 2004, **59**, 659.
[9] C. Salomé, E. Salomé-Grosjean, K. D. Park, P. Morieux, R. Swendiman, E. DeMarco, J. P. Stables and H. Kohn, *J. Med. Chem.* 2010, **53**, 1288.

chromatography using hexane/EtOAc = 1/4 to EtOAc as eluent to give *N*-(4-(1,3-dioxolan-2-yl)benzyl)acetamide (**1b**) as a white solid (1.40 g, 86% yield).

mp 81–84 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.0 Hz, 2H), 7.33–7.22 (m, 2H), 5.81 (s, 1H), 5.66 (br, 1H), 4.45 (d, *J* = 6.0 Hz, 2H), 4.15–4.00 (m, 4H), 2.02 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9, 139.3, 137.3, 127.9, 126.8, 103.4, 65.3, 43.5, 23.2. IR (KBr disc) 3310, 3063, 2886, 2808, 2762, 1651, 1551, 1435, 1397, 1366, 1296, 1250, 1227, 1080, 1026, 972, 941, 864, 826, 787, 709, 656, 617, 579, 532 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{12}H_{16}NO_3$ [M + H^+] 222.1130, found 222.1122.

tert-Butyl 4-(acetamidomethyl)benzylcarbamate (1c) [CAS Registry No. 1257585-74-2][10]



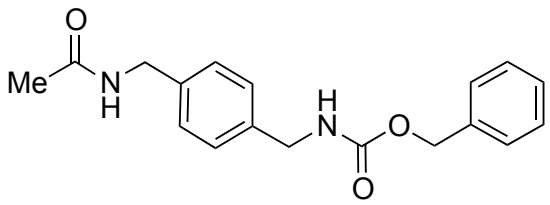
1c

tert-Butyl 4-(aminomethyl)benzylcarbamate [11] (2.36 g, 10.0 mmol) and Et_3N (2.8 mL, 20 mmol) were dissolved in dichloromethane (20 mL) followed by dropwise addition of acetic anhydride (1.1 mL, 12 mmol) at 0 ºC. The resulting mixture was stirred at room temperature overnight. The solvent was removed under reduced pressure. The crude residue was diluted with EtOAc and washed three times with saturated aqueous $NaHCO_3$ solution and once with brine. The combined organic layer was dried over Na_2SO_4, filtered and purified by flash silica gel column chromatography using EtOAc as eluent to give *tert*-butyl 4-(acetamidomethyl)benzylcarbamate (**1c**) as a white solid (1.92 g, 69% yield).

mp 115–117 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.19 (m, 4H), 5.67 (br, 1H), 4.82 (br, 1H), 4.42 (d, *J* = 5.6 Hz, 2H), 4.29 (d, *J* = 6.8 Hz, 2H), 2.02 (s, 3H), 1.46 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9, 155.9, 138.4, 137.3, 128.1, 127.7, 79.5, 44.3, 43.4, 28.4, 23.2. IR (KBr disc) 3333, 3287, 3078, 2978, 2932, 2886, 1682, 1643, 1505, 1443, 1366, 1296, 1250, 1180, 1049, 1018, 872, 826, 733, 625, 602 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{15}H_{21}N_2O_3$ [M – H^+] 277.1552, found 277.1556.

[10] Nissan Chemical Industires, Ltd., *Eur. Pat.*, EP2439204 A1, 2012.
[11] C. L. M. Goodyera, E. C. Chinjeb, M. Jaffarb, I. J. Stratfordb and M. D. Threadgilla, *Bioorg. Med. Chem.*, 2003, **11**, 4189.

Benzyl 4-(Acetamidomethyl)benzylcarbamate (1d) [CAS Registry No. N/A]

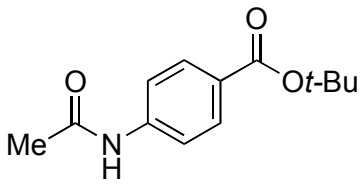


1d

p-Xylylendiamine (4.08 g, 30.0 mmol) and Et_3N (1.4 mL, 10 mmol) were dissolved in dichloromethane (80 mL) followed by dropwise addition of benzyl chloroformate (1.4 mL, 9.9 mmol) dissolved in dichloromethane (30 mL) at 0 ºC. The resulting mixture was stirred at room temperature for 3 h. The solvent was removed under reduced pressure. The crude residue was diluted with EtOAc and washed with 1 M aqueous NaOH solution. The solution was filtered and washed three times with 1 M aqueous NaOH solution and once with brine. The combined organic layer was dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure to give a white solid which contained benzyl 4-(aminomethyl)benzylcarbamate.[12] The crude benzyl 4-(aminomethyl)benzylcarbamate and Et_3N (2.5 mL, 18 mmol) were dissolved in dichloromethane (30 mL) followed by dropwise addition of acetic anhydride (0.99 mL, 10 mmol) at 0 ºC. The resulting mixture was stirred at room temperature for 4 h. The solvent was removed under reduced pressure and the residue was partitioned between EtOAc and 1 M aqueous HCl solution. The resulting mixture was filtered, and the layers were separated. The organic phase was dried over Na_2SO_4, filtered and purified by flash silica gel column chromatography using EtOAc as eluent to give benzyl 4-(acetamidomethyl)benzylcarbamate (**1d**) as a white solid (726 mg, 23% yield for 2 steps). mp 149–155 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.10 (m, 10H), 5.68 (br, 1H), 5.13 (s, 2H), 5.05 (br, 1H), 4.42 (d, J = 6.0 Hz, 2H), 4.37 (d, J = 6.0 Hz, 2H), 2.02 (s, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 169.0, 156.3, 138.2, 138.1, 137.2, 128.3, 127.8, 127.7, 127.3, 127.0, 65.3, 43.6, 41.9, 22.5. IR (KBr disc) 3301, 1690, 1636, 1543, 1427, 1381, 1265, 1219, 1142, 1057, 972, 825, 748, 671, 563 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{18}H_{21}N_2O_3$[M + H$^+$] 313.1552, found 313.1556.

[12] Pfizer Limited, *Eur. Pat.*, EP1577292 A1, 2005.

***tert*-Butyl 4-(acetamido)benzoate (1k) [CAS Registry No. N/A]**

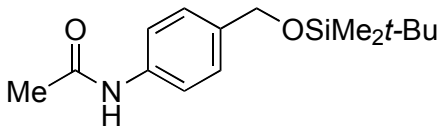


1k

To a stirred solution of *tert*-butyl 4-aminobenzoate (1.93 g, 10.0 mmol) in CH_2Cl_2 (20 mL) at 0 °C was added acetic anhydride (0.99 mL, 11 mmol) and the resulting mixture was stirred at room temperature for 8 h. The solvent was evaporated, and the crude mixture was extracted with ethyl acetate, and the organic phase was washed with 1 M aqueous HCl solution, saturated aqueous $NaHCO_3$ solution and brine. The organic phase was dried over Na_2SO_4, filtered and evaporated to give *tert*-butyl 4-(acetamido)benzoate (**1k**) as a white solid (2.35 g, >99% yield).

mp 105–109 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, J = 8.5 Hz, 2H), 7.88 (br s, 1H), 7.58 (d, J = 8.5 Hz, 2H), 2.19 (s, 3H), 1.58 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.7, 165.4, 141.8, 130.5, 127.4, 118.7, 80.9, 24.7. IR (KBr disc) 3263, 3187, 3117, 3055, 2978, 2932, 1705, 1674, 1597, 1535, 1404, 1375, 1296, 1265, 1165, 1111, 1010, 856, 772, 748, 702, 601, 579 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{13}H_{18}NO_3$ [M + H$^+$] 236.1287, found 236.1282.

***N*-(4-(((*tert*-Butyldimethylsilyl)oxy)methyl)phenyl)acetamide (1p) [CAS Registry No. N/A]**



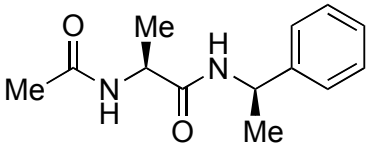
1p

To a stirred solution of 4-(acetamido)benzyl alcohol[13] (1.65 g, 10.0 mmol) in DMF (24 mL) were added imidazole (1.00 g, 14.7 mmol) and *tert*-butyldimethylsilyl chloride (1.50 g, 9.95 mmol) at 0 ºC, and the reaction mixture was stirred at room temperature overnight. After addition of water and Et_2O, the mixture was washed trice with water. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered, evaporated and purified by flash silica gel column chromatography using hexane/EtOAc = 1/1 as eluent to give *N*-(4-(((*tert*-butyldimethylsilyl)oxy)methyl)phenyl)acetamide (**1p**) as a white solid (1.59 g, 57 % yield).

[13] R. P. Iyer, and S. Padmanabhan, *US Pat*., US2007/149462 A1, 2007.

mp 98–100 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.11 (br, 1H), 4.69 (s, 2H), 2.16 (s, 3H), 0.93 (m, 9H), 0.09 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.2, 137.5, 136.6, 126.8, 119.7, 64.6, 25.9, 24.6, 18.4, –5.3. IR (KBr disc) 3256, 3194, 3125, 3071, 2940, 2862, 1659, 1605, 1512, 1466, 1373, 1319, 1057, 1011, 880, 810, 679 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{18}H_{32}NO_2Si$ [M + H$^+$] 322.2202, found 322.2196.

(*S*)-2-Acetamido-*N*-((*R*)-1-phenylethyl)propanamide (1q) [CAS Registry No. N/A]

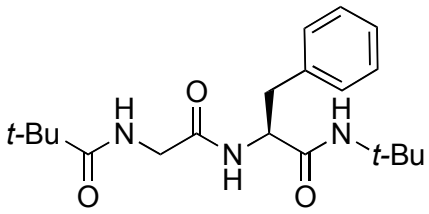


1q

tert-Butyl (*S*)-1-oxo-1-((*R*)-1-phenylethylamino)propane-2-ylcarbamate (1.463 g, 5.00 mmol) was dissolved in trifluoroacetic acid (10 mL) and CH_2Cl_2 (40 mL) at 0 °C. The reaction mixture was stirred at room temperature for 3 h. The solvent was removed under reduced pressure. The crude TFA salt was diluted with dichloromethane and Et_3N (1.4 mL, 10 mmol) was added, followed by dropwise addition of acetic anhydride (0.47 mL, 5.0 mmol) at 0 °C. The mixture was stirred for 3 h at room temperature, washed twice with 1 M aqueous HCl solution, once with saturated aqueous $NaHCO_3$ solution and once with brine. The combined organic layer was dried over Na_2SO_4, filtered and purified by flash silica gel column chromatography using dichloromethane/MeOH = 20/1 as eluent to give (*S*)-2-acetamido-*N*-((*R*)-1-phenylethyl)propanamide (**1q**) as a white solid (987.9 mg, 84% yield).

mp 180–182 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.16 (m, 5H), 6.96 (d, *J* = 7.0 Hz, 1H), 6.32 (d, *J* = 7.0 Hz, 1H), 5.03 (dq, *J* = 7.0, 7.0 Hz, 1H), 4.53 (dq, *J* = 7.0, 7.0 Hz, 1H), 1.90 (s, 3H), 1.47 (d, *J* = 7.0 Hz, 3H), 1.38 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.4, 170.0, 124.1, 128.6, 127.3, 126.0, 49.1, 48.9, 23.1, 22.1, 18.5. IR (KBr disc) 3287, 3078, 2970, 2932, 2816, 2724, 2639, 1636, 1551, 1443, 1373, 1281, 1242, 1157, 1126, 1018, 926, 733, 702, 610 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{13}H_{19}N_2O_2$ [M + H$^+$] 235.1447, found 235.1457. $[\alpha]^{27}_D$ –2.3 (*c* 1.00, $CHCl_3$).

(*S*)-*N*-(*tert*-Butyl)-3-phenyl-2-(2-pivalamidoacetamido)propanamide (1r) [CAS Registry No. N/A]



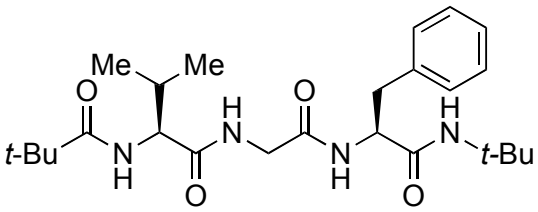
1r

(*S*)-*tert*-Butyl (1-(*tert*-butylamino)-1-oxo-3-phenylpropan-2-yl)carbamate[14] (1.03 g, 3.21 mmol) was dissolved 20% trifluoroacetic acid in CH_2Cl_2 (32 mL) at 0 °C. The reaction mixture was stirred at room temperature for 3 h. The solvent was removed under reduced pressure to give crude TFA salt (1.20 g). The crude TFA salt (1.00 g), 2-pivalamidoacetic acid[15] (448 mg, 2.81 mmol) and HBTU (1.14 g, 3.01 mmol) were dissolved in dichloromethane (15 mL) followed by dropwise addition of 2,4,6-trimethylpyridine (1.2 mL, 9.1 mmol) at 0 ºC. The resulting mixture was stirred at room temperature for 3 h. The solvent was removed under reduced pressure. The crude residue was diluted with EtOAc and washed three times with 1 M aqueous HCl solution, three times with saturated aqueous $NaHCO_3$ solution and once with brine. The combined organic layer was dried over Na_2SO_4, filtered and purified by flash silica gel column chromatography using hexane/EtOAc = 1/4 to EtOAc as eluent to give (*S*)-*N*-(*tert*-butyl)-3-phenyl-2-(2-pivalamidoacetamido)propanamide (**1r**) as a white solid (894.6 mg, 93% yield in 2 steps). Enantiomeric excess of **1r** was determined to be 99% by chiral HPLC analysis.

mp 155–158 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.20 (m, 5H), 6.56 (d, J = 7.5 Hz, 1H), 6.32 (br s, 1H), 5.14 (s, 1H), 4.49–4.42 (m, 1H), 3.90 (d, J = 5.0 Hz, 2H), 3.20 (dd, J = 5.5, 13.5 Hz, 1H), 2.84 (dd, J = 9.0, 13.5 Hz, 2H), 1.21 (s, 9H), 1.19 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 179.1, 169.2, 168.7, 136.8, 129.4, 128.7, 127.0, 55.1, 51.4, 43.3, 38.9, 38.7, 28.4, 27.4. IR (KBr disc) 3279, 3078, 2970, 2870, 1651, 1551, 1458, 1397, 1366, 1258, 1219, 1011, 941, 849, 795, 741, 702 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{20}H_{32}N_3O_3$ [M + H$^+$] 362.2444, found 362.2429. HPLC conditions: DAICEL CHIRALCEL AD-3, eluent: Hexane/2-Propanol 9.0/1.0, flow: 1.0 mL/min, detection: 210 nm, t_R: 8.8 min (minor), 11.9 min (major). $[\alpha]^{26}_D$ –4.9 (*c* 1.02, $CHCl_3$).

[14] Y. Lu, C. Zheng, Y. Yang, G. Zhao and G. Zou, *Adv. Synth. Catal.*, 2011, **353**, 3129.
[15] N. M. Maier, E. Greco, J. Petrovaj and W. Lindner, *Acta Chim. Slov.*, 2012, **59**, 454.

(*S*)-*N*-(2-((*S*)-1-(*tert*-Butylamino)-1-oxo-3-phenylpropan-2-ylamino)-2-oxoethyl)-3-methyl-2-pivalamidobutanamide (1s) [CAS Registry No. N/A]



1s

To a solution of (*S*)-*tert*-butyl (1-(*tert*-butylamino)-1-oxo-3-phenylpropan-2-yl)carbamate[14] (641 mg, 2.00 mmol) in CH_2Cl_2 (5.0 mL) at 0 °C was added 4 M HCl in AcOEt (5.0 mL, 20 mmol). The mixture was stirred at room temperature for 1 h, and the solvent was removed under reduced pressure. The residue was diluted with CH_2Cl_2 and the organic phase was washed with 1 M aqueous NaOH solution and the aqueous phase was extracted with CH_2Cl_2. The combined organic phase was dried over Na_2SO_4, filtered and evaporated to give crude (*S*)-2-amino-*N*-*tert*-butyl-3-phenylpropanamide[16] (459 mg) as pale yellow oil, which was used without further purification. The crude oil was dissolved in CH_2Cl_2 (10 mL) and transferred to the mixture of (*S*)-2-(3-methyl-2-pivalamidobutanamido)acetic acid (**S1**) (516 mg, 2.00 mmol) and HOBt (297 mg, 2.20 mmol). To the mixture at 0 °C was added EDCI·HCl (422 mg, 2.20 mmol) and Et_3N (0.31 mL, 2.2 mmol), and the mixture was stirred at room temperature for 16 h. The solvent was evaporated and the crude residue was diluted with EtOAc. The organic phase was washed twice with 1 M aqueous HCl solution/brine mixture and twice with saturated aqueous $NaHCO_3$ solution/brine mixture, dried over Na_2SO_4, filtered and purified by flash silica gel column chromatography using hexane/EtOAc = 1/1 to EtOAc only to EtOAc/MeOH = 9/1 to 2/1 as eluent to give (*S*)-*N*-(2-((*S*)-1-(*tert*-butylamino)-1-oxo-3-phenylpropan-2-ylamino)-2-oxoethyl)-3-methyl-2-pivalamidobutanamide (**1s**) as a white solid (847.3 mg, 92% yield in 2 steps).

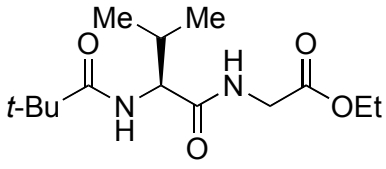
mp 108–112 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.11 (m, 6H), 6.98 (d, J = 7.5 Hz, 1H), 6.31 (d, J = 8.0, Hz, 1H), 5.66 (s, 1H), 4.59 (ddd, J = 5.5, 7.5, 8.5 Hz, 1H), 4.39 (dd, J = 7.0, 8.0 Hz, 1H), 4.03 (dd, J = 5.5, 17 Hz, 1H), 3.88 (dd, J = 5.0, 17 Hz, 1H), 3.10 (dd, J = 5.5,

[16] D. Obrecht, U. Bohdal, C. Broger, D. Bur, C. Lehmann, R. Ruffieux, P. Schönholzer, C. Spiegler, and K. Müller, *Helv. Chim. Acta*, 1995, **78**, 563.

13.5 Hz, 1H), 2.94 (dd, *J* = 8.5, 13.5 Hz, 1H), 2.10 (qqd, *J* = 6.0, 6.5, 7.0 Hz, 1H), 1.22 (s, 9H), 1.17 (s, 9H), 0.95 (d, *J* = 6.0 Hz, 3H), 0.94 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.7, 172.0, 169.4, 168.1, 136.8, 129.5, 128.6, 126.9, 58.1, 55.1, 51.3, 42.8, 39.1, 38.9, 31.2, 28.5, 27.6, 19.3, 18.3. IR (KBr disc) 3316, 3298, 3082, 2997, 2934, 2872, 1640, 1533, 1506, 1456, 1393, 1366, 1261, 1225, 1032, 743, 698 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{25}H_{41}N_4O_4$ [M + H$^+$] 461.3128, found 461.3128. $[\alpha]^{27}_D$ 2.8 (*c* 0.99, $CHCl_3$).

Preparation of (*S*)-2-(3-Methyl-2-pivalamidobutanamido)acetic Acid (S1)

(*S*)-Ethyl 2-(3-methyl-2-pivalamidobutanamido)acetate (S2) [CAS Registry No. N/A]



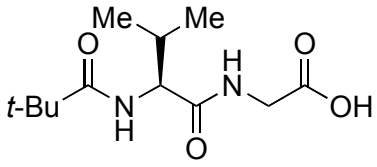
S2

To a solution of (*S*)-ethyl 2-(2-amino-3-methylbutanamido)acetate hydrochloride[17] (2.607 g, 10.92 mmol) in CH_2Cl_2 (34 mL) at 0 °C was added Et_3N (3.4 mL, 24 mmol) and pivaloyl chloride (1.3 mL, 11 mmol), and the mixture was stirred at room temperature for 12 h. The solvent was evaporated and the crude residue was diluted with EtOAc. The organic phase was washed twice with 1 M aqueous HCl solution/brine mixture and twice with saturated aqueous $NaHCO_3$ solution/brine mixture, dried over Na_2SO_4, filtered and evaporated to give (*S*)-ethyl 2-(3-methyl-2-pivalamidobutanamido)acetate (**S2**) as a white solid (2.983 g, 95% yield).

mp 103–105 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.53 (br s, 1H), 6.22 (d, *J* = 8.5 Hz, 1H), 4.32 (dd, *J* = 6.5, 8.5 Hz, 1H), 4.21 (q, *J* = 7.0 Hz, 2H), 4.10 (dd, *J* = 5.5, 18 Hz, 1H), 3.94 (dd, *J* = 5.0, 18 Hz, 1H), 2.16 (dqq, *J* = 6.5, 6.5, 7.0 Hz, 1H), 1.28 (t, *J* = 7.0 Hz, 3H), 0.98 (d, *J* = 7.0 Hz, 3H), 0.95 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.6, 171.6, 169.5, 61.5, 58.0, 41.3, 38.9, 31.0, 27.5, 19.2, 18.1, 14.1. IR (KBr disc) 3337, 3279, 3094, 2974, 2872, 1759, 1678, 1632, 1564, 1532, 1481, 1468, 1449, 1402, 1379, 1204, 1186, 1099, 1026, 964, 930, 864, 712, 669, 559 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{14}H_{27}N_2O_4$ [M + H$^+$] 287.1971, found 287.1973. $[\alpha]^{28}_D$ –42.7 (*c* 1.00, $CHCl_3$).

[17] L. Ayres, K. Koch, P. H. H. M. Adams, J. C. M. van Hest, *Macromolecules*, 2005, **38**, 1699.

(*S*)-2-(3-Methyl-2-pivalamidobutanamido)acetic acid (S1) [CAS Registry No. N/A]



S1

To a solution of **S2** (859 mg, 3.00 mmol) in MeOH (6.0 mL) at 0 °C was added 1 M aqueous NaOH solution (6.0 mL, 6.0 mmol), and the mixture was stirred at 0 °C for 1 h and acidified to pH ~ 4–5 with 1 M aqueous HCl solution (4.5 mL). The organic solvent was evaporated and the residue was further acidified to pH ~ 1 by addition of 1 M aqueous HCl solution (2.5 mL), and the aqueous phase was extracted twice with EtOAc. The combined organic phase was dried over Na_2SO_4, filtered and evaporated to give (*S*)-2-(3-methyl-2-pivalamidobutanamido)acetic acid (**S1**) as a white solid (750 mg, 97% yield).

mp 175–178 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26 (br s, 1H), 6.48 (d, J = 9.0 Hz, 1H), 4.62 (dd, J = 8.0, 9.0 Hz, 1H), 4.15 (dd, J = 5.0, 18.5 Hz, 1H), 3.99 (dd, J = 4.0, 18.5 Hz, 1H), 2.04 (qqd, J = 6.5, 6.5, 8.0 Hz, 1H), 0.97 (d, J = 6.5 Hz, 3H), 0.94 (d, J = 6.5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 179.6, 171.62, 171.57, 58.0, 41.4, 39.0, 31.6, 27.4, 19.3, 18.2. IR (KBr disc) 3327, 3291, 3084, 2970, 2876, 1728, 1713, 1678, 1562, 1526, 1227, 1204, 1040, 934, 889, 706 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{12}H_{23}N_2O_4$ [M + H$^+$] 259.1658, found 259.1648. $[\alpha]^{26}_D$ –48.7 (*c* 1.00, MeOH).

4. General Procedure for Deacylation of Amines (Tables 1 and 2)

Conventional heating conditions: To a 4.0 mL of a vial equipped with a magnetic stir bar, ammonium iodide (144 mg, 1.00 mmol), amide or anilide **1** (1.00 mmol) and hydrazine monohydrate (0.50 mL, 10 mmol) were added and the vial was sealed with a Teflon-lined screw cap. The vial was heated with stirring at the indicated temperature and time. The resulting mixture was purified by extraction or flush silica gel column chromatography to give amine **2**.

Microwave heating conditions: To a 10 mL of a glass test tube equipped with a magnetic stir bar, ammonium iodide (144 mg, 1.00 mmol), amide or anilide **1** (1.00 mmol) and hydrazine monohydrate (0.50 mL, 10 mmol) were added and the tube was sealed with a cap. The test tube was heated with stirring at the indicated temperature and time under microwave irradiation conditions (maximum power 250 W). The crude mixture was purified by the indicated methods.

Experimental Details of Control Experiments Under Literature Conditions (Table 1)

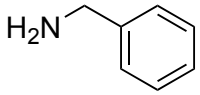
Entry 1: Amide **1a** (1.00 mmol) and hydrazine monohydrate (0.50 mL, 10 mmol) were mixed and stirred at 60 °C for 6 h. ^{1}H NMR analysis of the crude mixture showed that **2a** was not detected.

Entry 2: Acetic acid (0.086 mL, 1.5 mmol), amide **1a** (74.5 mg, 0.500 mmol) and hydrazine monohydrate (0.075 mL, 1.5 mmol) were dissolved in EtOH (0.50 mL). The resulting mixture was heated with stirring at 60 ºC for 6 h. ^{1}H NMR analysis of the crude mixture showed that **2a** was formed in 7% yield.

Details for Each Substrate and Characterization Data of Products

Amines **2a**, **2e**, **2i** and anilines **2h**, **2j**, **2k**, **2l**, **2n** and **2o** were commercially available.

Benzylamine (2a) (Table 1, entry 7) [CAS Registry No. 100-46-9]

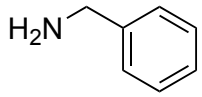


2a

After the reaction, the crude mixture was diluted with dichloromethane and deprotected amine was extracted with 1 M aqueous HCl solution. The aqueous layer was basified with 1 M aqueous NaOH solution and

back-extracted with Et_2O. The organic layer was dried over Na_2SO_4, filtered and evaporated to give benzylamine (**2a**) as pale yellow liquid (96.3 mg, 90% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.25 (m, 5H), 3.87 (s, 2H), 1.55 (br, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.3, 128.5, 127.1, 126.8, 46.5.

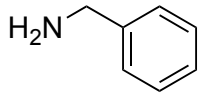
Benzylamine (2a) (Table 1, entry 8) [CAS Registry No. 100-46-9]



2a

After the reaction, the crude mixture was diluted with dichloromethane and deprotected amine was extracted with 1 M aqueous HCl solution. The aqueous layer was basified with 1 M aqueous NaOH solution and back-extracted with Et_2O. The organic layer was dried over Na_2SO_4, filtered and evaporated to give benzylamine (**2a**) as pale yellow liquid (94.9 mg, 89% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.21 (m, 5H), 3.87 (s, 2H), 1.55 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.2, 128.5, 127.0, 126.8, 46.5.

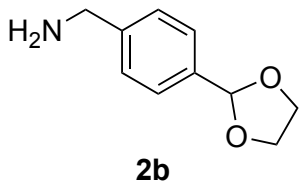
Benzylamine (2a) (Table 1, entry 9) [CAS Registry No. 100-46-9]



2a

After the reaction, the crude mixture was diluted with dichloromethane and deprotected amine was extracted with 1 M aqueous HCl solution. The aqueous layer was basified with 1 M aqueous NaOH solution and back-extracted with Et_2O. The organic layer was dried over Na_2SO_4, filtered and evaporated to give benzylamine (**2a**) as pale yellow liquid (97.0 mg, 90% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.22 (m, 5H), 3.88 (s, 2H), 1.52 (br s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.2, 128.6, 127.1, 126.8, 46.5.

(4-(1,3-Dioxolan-2-yl)phenyl)methanamine (2b) (Table 2, entry 1) [CAS Registry No. 104566-44-1][9]



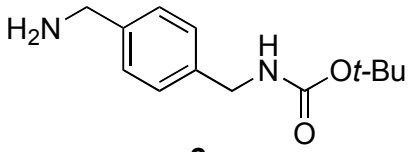
2b

This reaction was performed on a 0.50 mmol scale for **1b**. After the reaction, the crude reaction mixture was diluted with saturated aqueous $NaHCO_3$ solution. The aqueous layer was extracted with AcOEt and combined organic layer was dried over Na_2SO_4, filtered and purified by flash silica gel column chromatography using

dichloromethane/MeOH = 20/1 to dichloromethane/MeOH/diethylamine = 20/1/1 as eluent. After evaporation of the solvent, the residue was washed with Et_2O and filtered to give (4-(1,3-dioxolan-2-yl)phenyl)methanamine (**2b**) as a white solid (69.8 mg, 78% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 5.81 (s, 1H), 4.17–4.00 (m, 4H), 3.88 (s, 1H), 1.48 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.0, 136.5, 127.1, 126.7, 103.6, 65.2, 46.1.

Under the conditions using ethylenediamine as cleaving agent: To a 10 mL of a glass test tube equipped with a magnetic stir bar, ammonium bromide (98 mg, 1.0 mmol), amide **1b** (221 mg, 1.00 mmol) and ethylenediamine (0.27 mL, 4.0 mmol) were added and the tube was sealed with a cap. The test tube was heated with stirring for 5 h at the 80 ºC under microwave irradiation conditions (maximum power 250 W). The crude mixture was diluted with saturated aqueous $NaHCO_3$ solution and the deprotected amine was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4, filtered, evaporated and purified by flash silica gel column chromatography using dichloromethane/MeOH = 100/5 to dichloromethane/MeOH/diethylamine = 100/5/1 as eluent to give crude **2b** as a yellow liquid. Yield was determined by ^{1}H NMR analysis using 1,2,4,5-tetramethylbenzene as an internal standard (<40% yield).

***tert*-Butyl 4-(aminomethyl)benzylcarbamate (2c) (Table 2, entry 2) [CAS Registry No. 108468-00-4][18]**



2c

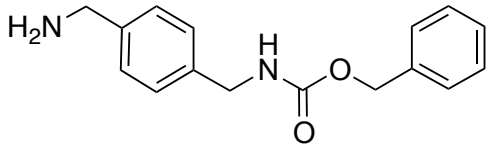
This reaction was performed in EtOH (0.50 mL), and after the reaction the crude mixture was directly purified by flash silica gel column chromatography using dichloromethane/MeOH = 10/1 to dichloromethane/MeOH/diethylamine = 20/2/1 as eluent to give *tert*-butyl 4-(aminomethyl)benzylcarbamate (**2c**) as a white solid (200 mg, 85% yield).

[18] N. Y. Mok, J. Chadwick, K. A. B. Kellett, E. Casas-Arce, N. M. Hooper, A. P. Johnson and C. W. G. Fishwick, *J. Med. Chem.*, 2013, **56**, 1843.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.21 (m, 4H), 4.81 (br s, 1H), 4.30 (d, J = 5.5 Hz, 2H), 3.86 (s, 2H), 1.46 (s, 9H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 155.8, 141.6, 138.3, 127.0, 126.8, 77.7, 45.0, 43.1, 28.2.

Under the conditions using ethylenediamine as cleaving agent: To a 10 mL of a glass test tube equipped with a magnetic stir bar, ammonium bromide (98 mg, 1.0 mmol), amide **1c** (278 mg, 1.00 mmol) and ethylenediamine (0.27 mL, 4.0 mmol) were added and the tube was sealed with a cap. The test tube was heated with stirring for 5 h at the 80 ºC under microwave irradiation conditions (maximum power 250 W). The crude mixture was diluted with saturated aqueous $NaHCO_3$ solution and the deprotected amine was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4, filtered, evaporated and purified by flash silica gel column chromatography using dichloromethane/MeOH = 100/5 to dichloromethane/MeOH/diethylamine = 100/5/1 as eluent to give **2c** containing minor impurities as a white solid (94 mg, <40% yield).

Benzyl 4-(aminomethyl)benzylcarbamate (2d) (Table 2, entry 3) [CAS Registry No. 164648-85-5][19]



2d

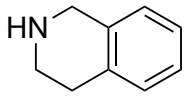
This reaction was performed on a 0.250 mmol scale for **1d** in EtOH (0.25 mL). The crude reaction mixture was directly purified by flash silica gel column chromatography using dichloromethane/MeOH = 10/1 to dichloromethane/MeOH/diethylamine = 20/2/1 as eluent. After evaporation of the solvent, the crude **2d** was dissolved in dichloromethane and 1 M aqueous NaOH solution. The aqueous layer was extracted with dichloromethane. The combined organic layers were dried over Na_2SO_4, filtered and evaporated to give pure **2d** as a white solid (53.6 mg, 79 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.17 (m, 10H), 5.14 (s, 2H), 5.04 (br, 1H), 4.37 (d, J = 6.0 Hz, 2H), 3.85 (s, 2H), 1.45 (br s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.3, 142.7, 137.6, 137.2, 128.3, 127.8, 127.7, 126.9, 126.8, 65.3, 45.3, 43.6.

[19] Pfizer Limited, *Eur. Pat*., EP1577292 A1, 2005.

Under the conditions using ethylenediamine as cleaving agent: To a 10 mL of a glass test tube equipped with a magnetic stir bar, ammonium bromide (98 mg, 1.0 mmol), amide **1d** (312 mg, 1.00 mmol) and ethylenediamine (0.27 mL, 4.0 mmol) were added and the tube was sealed with a cap. The test tube was heated with stirring for 5 h at the 80 ºC under microwave irradiation conditions (maximum power 250 W). However, **2d** was not detected on ^{1}H NMR spectrum of the crude mixture.

1,2,3,4-Tetrahydroisoquinoline (2e) (Table 2, entry 4) [CAS Registry No. 91-21-4]

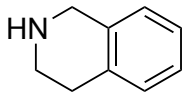


2e

After the reaction, the crude mixture was diluted with Et_2O and washed with 1 M aqueous NaOH solution. The aqueous layer was extracted with Et_2O, and combined organic layers were dried over Na_2SO_4, filtered and evaporated to give 1,2,3,4-tetrahydroisoquinoline (**2e**) as pale yellow liquid (122.5 mg, 92% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18–6.94 (m, 4H), 4.01 (s, 2H), 3.14 (t, J = 6.0 Hz, 2H), 2.80 (t, J = 6.0 Hz, 2H), 1.58 (br s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 134.8, 129.3, 126.2, 126.0, 125.7, 48.3, 43.9, 29.2.

1,2,3,4-Tetrahydroisoquinoline (2e) (Table 2, entry 5) [CAS Registry No. 91-21-4]

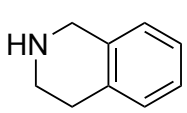


2e

This reaction was performed on a 0.500 mmol scale for **1f** in EtOH (0.25 mL). After the reaction, the crude reaction mixture was diluted with dichloromethane and washed with 1 M aqueous NaOH solution. The aqueous layer was extracted with dichloromethane and combined organic layer was dried over Na_2SO_4, filtered and purified by flash silica gel column chromatography using dichloromethane/MeOH = 100/5 as eluent to give 1,2,3,4-tetrahydroisoquinoline (**2e**) as pale yellow liquid (55.9 mg, 84% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17–6.97 (m, 4H), 4.02 (s, 2H), 3.15 (t, J = 6.0 Hz, 2H), 2.80 (t, J = 6.0 Hz, 2H), 1.63 (br s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.1, 134.9, 129.5, 126.4, 126.2, 125.9, 48.9, 44.1, 29.4.

1,2,3,4-Tetrahydroisoquinoline (2e) (Table 2, entry 6) [CAS Registry No. 91-21-4]

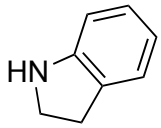


2e

After the reaction, the crude mixture was diluted with dichloromethane and deprotected amine was extracted with 1 M aqueous HCl solution. The aqueous layer was basified with 1 M aqueous NaOH solution and back-extracted with Et_2O. The organic layer was dried over Na_2SO_4, filtered and evaporated. The residue was treated with hexane, filtered and evaporated to give 1,2,3,4-tetrahydroisoquinoline (**2e**) as yellow liquid (110.5 mg, 83% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.05 (m, 4H), 4.01 (s, 2H), 3.14 (t, J = 6.0 Hz, 2H), 2.80 (t, J = 6.0 Hz, 2H), 1.60 (br s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 134.8, 129.3, 127.0, 125.7, 48.3, 43.9, 29.2.

Indoline (2h) (Table 2, entry 7) [CAS Registry No. 496-15-1]

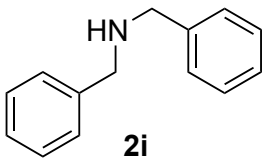


2h

After the reaction in EtOH (0.50 mL), the crude reaction mixture was directly purified by flash silica gel column chromatography using hexane/ EtOAc = 4/1 as eluent to give to indoline (**2h**) as colorless liquid (101.0 mg, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, J = 7.6 Hz, 1H), 7.03–6.92 (m, 1H), 6.74–6.77 (m, 1H), 6.64 (d, J = 7.6 Hz, 1H), 3.74 (br s, 1H), 3.55 (t, J = 8.4 Hz, 2H), 3.03 (t, J = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.6, 129.3, 127.2, 124.6, 118.6, 109.4, 47.3, 29.8.

Dibenzylamine (2i) (Table 2, entry 8) [CAS Registry No. 103-49-1]



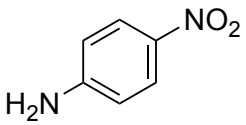
2i

This reaction was performed on a 0.50 mmol scale for **1i** in EtOH (0.25 mL). After the reaction, the crude mixture was diluted with Et_2O and washed with saturated aqueous $NaHCO_3$ solution. The aqueous layer was extracted with Et_2O and combined organic layer was dried over Na_2SO_4, filtered, evaporated and purified by flash silica gel column chromatography using hexane/ EtOAc = 1/1 as eluent to give dibenzylamine (**2i**) as colorless liquid (90.6 mg, 92% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.20 (m, 10H), 3.82 (s, 4H), 1.57 (br s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.3, 128.4, 128.1, 126.9, 53.2.

The above reaction was performed without addition of ammonium iodide under identical conditions to give **2i** in only 26% yield.

4-Nitoroaniline (2j) (Table 2, entry 9) [CAS Registry No. 100-01-6]

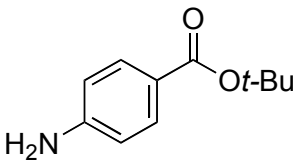


2j

This reaction was performed in EtOH (0.50 mL). After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using hexane/EtOAc = 3/1 to 2/1 as eluent to give 4-nitoroaniline (**2j**) as a yellow solid (133.7 mg, 97% yield).

^{1}H NMR (500 MHz, DMSO-d_6) δ 7.98–7.90 (m, 2H), 6.70 (br, 2H), 6.63–6.56 (d, J = 8.4 Hz, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 155.7, 135.6, 126.4, 112.4.

***tert*-Butyl 4-aminobenzoate (2k) (Table 2, entry 10) [CAS Registry No. 18144-47-3]**

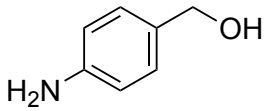


2k

This reaction was performed on a 0.25 mmol scale for **1l** in EtOH (0.25 mL). After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using hexane/EtOAc = 1/1 to 1/4 as eluent to give *tert*-butyl 4-aminobenzoate (**2k**) as a white solid (80.9 mg, 84% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77–7.83 (m, 2H), 6.59–6.65 (m, 2H), 3.82 (br s, 2H), 1.57 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 150.4, 131.3, 121.8, 113.7, 80.0, 28.3.

4-Aminobenzyl alcohol (2l) (Table 2, entry 11) [CAS Registry No. 623-04-1]

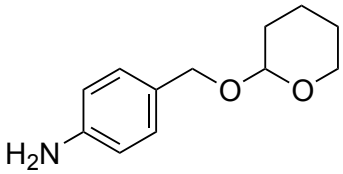


2l

After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using hexane/EtOAc = 2/1 as eluent to give 4-aminobenzyl alcohol (**2l**) as a pale yellow solid (115.9 mg, 94% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.10 (m, 2H), 6.72–6.62 (m, 2H), 4.55 (s, 2H), 3.67 (br s, 2H), 1.59 (br s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.0, 131.0, 128.7, 115.1, 65.3.

4-[(2-Tetrahydropyranyloxy)methyl]aniline (2m) (Table 2, entry 12) [CAS Registry No. 18484-05-4][2]

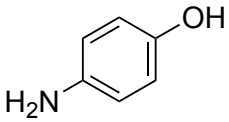


2m

This reaction was performed in EtOH (0.50 mL). After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using hexane/AcOEt = 1/2 as eluent to give 4-[(2-tetrahydropyranyloxy)methyl]aniline (**2m**) as pale yellow liquid (194.8 mg, 94% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19–7.13 (m, 2H), 6.69–6.64 (m, 2H), 4.70–4.65 (m, 1H), 4.66 (d, *J* = 11.2 Hz, 2H), 4.39 (d, *J* = 11.2 Hz, 2H), 3.96–3.84 (m, 1H), 3.64 (br, 2H), 3.58–3.50 (m, 1H), 1.90–1.45 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.9, 129.6, 128.0, 114.9, 97.3, 68.7, 62.1, 30.6, 25.5, 19.4.

4-Aminophenol (2n) (Table 2, entry 13) [CAS Registry No. 123-30-8]

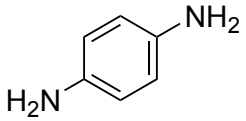


2n

After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using hexane/EtOAc = 2/1 to 1/1 as eluent to give 4-aminophenol (**2n**) as a white solid (106.2 mg, 97% yield).

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.29 (s, 1H), 6.50–6.38 (m, 4H), 4.34 (s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 148.2, 140.7, 115.6, 115.3.

***p*-Phenylenediamine (2o) (Table 2, entry 14) [CAS Registry No. 106-50-3]**

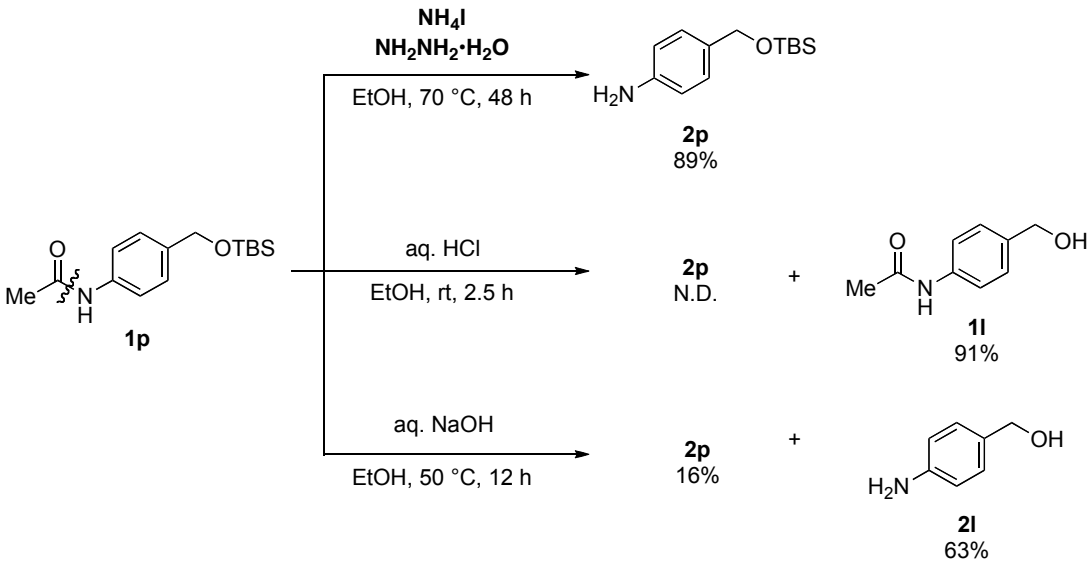


2o

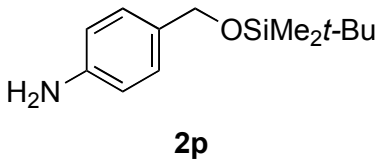
After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using hexane/ EtOAc = 1/2 to EtOAc only as eluent to give *p*-phenylenediamine (**2o**) as a pale red solid (106.6 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.57 (s, 4H), 3.32 (br s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 116.7.

5. Comparison with Conventional Acidic/Basic Hydrolysis Conditions (Scheme 1)



Under our conditions: **4-[(*tert*-Butyldimethylsilyloxy)methyl]aniline (2p) [CAS Registry No. 131230-76-7]**[20]

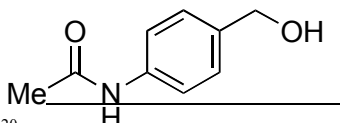


2p

According to the general procedure for Table 2, this reaction was performed in EtOH (0.50 mL), and the crude mixture was directly purified by flash silica gel column chromatography using hexane/EtOAc = 1/3 as eluent to give 4-[(*tert*-Butyldimethylsilyloxy)methyl]aniline (**2p**) as colorless liquid (210.5 mg, 89% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.4 Hz, 2H), 4.62 (s, 2H), 3.56 (br, 2H), 0.92 (s, 9H), 0.07 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.3, 131.5, 127.7, 115.0, 65.0, 26.0, 18.4, –5.2.

Under acidic conditions: **4-(Acetamido)benzyl alcohol (1l) [CAS Registry No. 16375-88-5]**[2]



1l

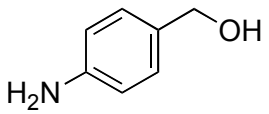
A solution of **1p** (55.9 mg, 0.200 mmol) in 1 M aqueous HCl solution (0.20 mL, 0.20 mmol) was stirred for 2.5 h at room

[20] H. Lee, M. Suzuki, J. Cui and S. A. Kozmin, *J. Org. Chem.*, 2010, **75**, 1756.

temperature. After the reaction, the crude mixture was diluted with CH_2Cl_2 and washed with saturated aqueous $NaHCO_3$ solution. The aqueous layer was extracted with CH_2Cl_2, and combined organic layer was dried over Na_2SO_4, filtered, evaporated and purified by silica gel column chromatography using hexane/EtOAc = 1/2 to EtOAc only as eluent to give 4-(acetamido)benzyl alcohol **1l** as a white solid (30.0 mg, 91 % yield). The desired product **2p** was not detected based on TLC analysis.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.65 (d, *J* = 5.2 Hz, 2H), 2.18 (s, 3H), 1.63 (m, 1H).

Under basic conditions: **4-Aminobenzyl alcohol (2l) [CAS Registry No. 623-04-1]**

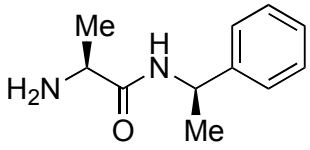


2l

A mixture of **1p** (139.8 mg, 0.500 mmol) and NaOH (200.7 mg, 5.00 mmol) in H_2O/EtOH (1/1, 0.50 mL) was stirred for 12 h at 50 ºC. The crude mixture was diluted with EtOH, quenched with solid NH_4Cl, filtered, evaporated and purified by flash silica gel column chromatography using hexane/EtOAc = 1/1 to 1/2 to EtOAc only as eluent to give crude 4-aminobenzyl alcohol (**2l**) as a pale yellow solid. The yield of **2l** was determined by ^{1}H NMR analysis using 1,2,4,5-tetramethylbenzene as an internal standard (63% yield). 4-[(*tert*-Butyldimethylsilyloxy)methyl]aniline (**2p**) was also obtained as yellow liquid (18.9 mg, 16% yield).

6. Application to Peptide And Amino Sugar Derivatives (*Scheme 2)*

(*S*)-2-Amino-*N*-((*R*)-1-phenylethyl)propanamide (2q) [CAS Registry No. N/A]

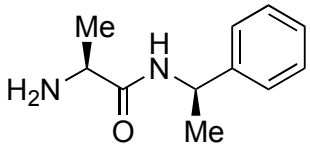


2q

According to the general procedure for Table 2, this reaction was performed on a 0.250 mmol scale for **1q** (58.6 mg) in EtOH (0.25 mL). After the reaction, the crude mixture was first directly purified by flash silica gel column chromatography using dichloromethane/MeOH = 100/5, and the crude product was further purified by flash silica gel column chromatography using dichloromethane/MeOH = 100/3 to dichloromethane/MeOH/Et_2NH =100/10/1 as eluent to give (*S*)-2-amino-*N*-((*R*)-1-phenylethyl)propanamide (**2q**) as yellow liquid (39.6 mg, 82% yield).

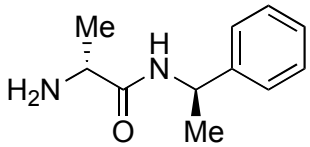
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (br s, 1H), 7.36–7.23 (m, 5H), 5.10 (dq, *J* = 7.0, 7.0 Hz, 1H), 3.51 (q, *J* = 7.0 Hz, 1H), 1.53 (br s, 2H), 1.49 (d, *J* = 7.0 Hz, 3H), 1.32 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.6, 143.5, 128.6, 127.2, 126.0, 50.8, 48.1, 22.1, 21.8. $[\alpha]^{27}_D$ +112.4 (*c* 1.00, $CHCl_3$).

Determination of diastereomeric purity of **2q**: Diastereomeric purity of **2q** was determined by ^{1}H NMR analysis of **2q** and its diastereomer **2q´** in C_6D_6 based on the peaks of α-Me (underlined below). Peaks corresponding to **2q´** were well below the detection limit (>20/1) on ^{1}H NMR chart of **2q** (Fig. S3)**.**



2q

Data for **2q**: ^{1}H NMR (500 MHz, C_6D_6) δ 7.35 (br s, 1H), 7.25–7.09 (m, 4H), 7.09–7.01 (m, 1H), 5.27 (qd, *J* = 7.0, 7.5 Hz, 1H), 3.07 (br q, *J* = 7.0 Hz, 1H), 1.28 (d, *J* = 7.0 Hz, 3H), <u>1.04</u> (d, *J* = 7.0 Hz, 3H), 0.63 (br s, 2H). ^{13}C NMR (125 MHz, C_6D_6) δ 173.8, 144.6, 128.8, 127.3, 126.4, 50.9, 48.3, 22.23, 21.7. HRMS (ESI-TOF) *m/z* calcd. $C_{11}H_{17}N_2O$ [M + H^+] 193.1341, found 193.1337.



2q´

Data for **2q´**: ^{1}H NMR (500 MHz, C_6D_6) δ 7.33 (br s, 1H), 7.23–7.08 (m, 4H), 7.08–7.02 (m, 1H), 5.29 (qd, *J* = 7.0, 8.0 Hz, 1H), 3.02 (q, *J* = 7.0 Hz, 1H), 1.26 (d, *J* = 7.0 Hz, 3H), <u>1.10</u> (d, *J* = 7.0 Hz, 3H), 0.51 (br s, 2H). ^{13}C NMR (125 MHz, C_6D_6) δ 173.8, 144.6, 128.8, 127.3, 126.6, 50.9, 48.3, 22.15, 21.7.

HRMS (ESI-TOF) *m/z* calcd. for $C_{11}H_{17}N_2O$ [M + H^+] 193.1341, found 193.1336.

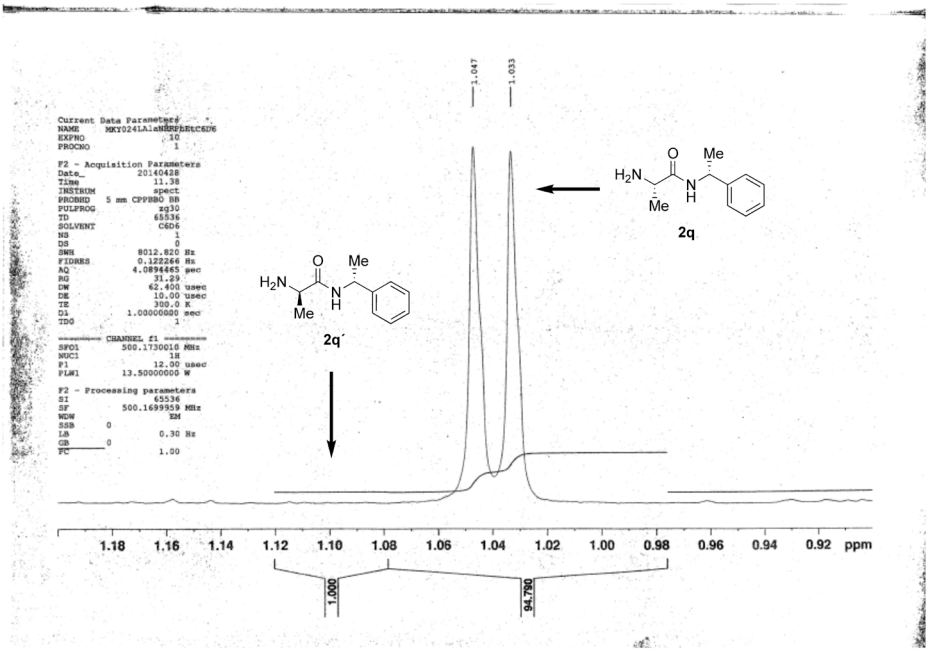
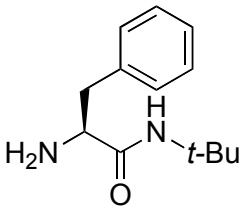


Fig. S3 Diastereomeric purity of **2q**.

(*S*)-2-Amino-*N*-(*tert*-butyl)-3-phenylpropanamide (2r) [CAS Registry No. 40847-05-0][21]



2r

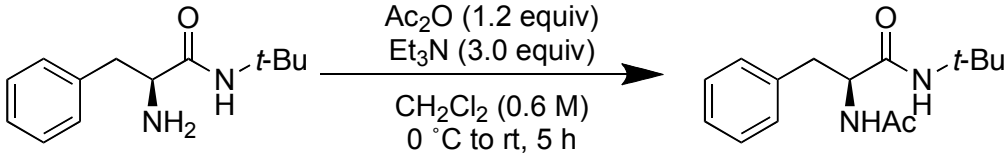
According to the general procedure for Table 2, this reaction was performed on a 0.250 mmol scale for **1r** (90.4 mg) in EtOH (0.13 mL). After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using EtOAc only to EtOAc/MeOH =10/1 as eluent. After evaporation of the solvent, the residue was diluted with dichloromethane and deprotected amine was extracted with 1 M aqueous HCl solution. The aqueous layer was basified with 1 M aqueous NaOH solution and back-extracted with Et_2O. The organic layer was dried over

[21] D. Obrecht, U. Bohdal, C. Broger, D. Bur, C. Lehmann, R. Ruffeux, P. Schönholzer, C. Spiegler and K. Müller, *Helv. Chim. Acta*, 1995, **78**, 563.

Na_2SO_4, filtered and evaporated to give (*S*)-2-amino-*N*-(*tert*-butyl)-3-phenylpropanamide (**2r**) as yellow liquid (46.2 mg, 84% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34–7.18 (m, 5H), 7.01 (br s, 1H), 3.47 (dd, *J* = 4.5, 9.0 Hz, 1H), 3.21 (dd, *J* = 4.5, 13.8 Hz, 1H), 2.71 (dd, *J* = 9.0, 13.8 Hz, 1H), 1.34 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.3, 138.2, 129.3, 128.6, 126.7, 56.9, 50.4, 41.1, 28.7. $[\alpha]^{27}_D$ –64.4 (*c* 1.01, $CHCl_3$).

Enantiomeric excess of **2r** was determined to be 99% after conversion of **2r** as shown below:

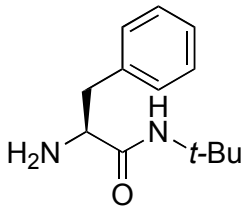


2r
(0.10 mmol)

3
99% ee

HPLC conditions of **3** [CAS Registry No. N/A][22]: DAICEL CHIRALCEL AD-3, eluent: Hexane/2-Propanol = 9.0/1.0, flow: 1.0 mL/min, detection: 220 nm, t_R: 5.4 min (minor), 7.7 min (major).

(*S*)-2-Amino-*N*-(*tert*-butyl)-3-phenylpropanamide (2r) [CAS Registry No. 40847-05-0][21]



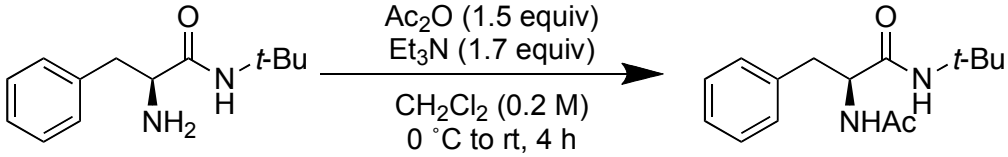
2r

According to the general procedure for Table 2, this reaction was performed on a 0.200 mmol scale for **1s** (92.3 mg) in EtOH (0.20 mL). After the reaction, the crude mixture was directly purified by flash silica gel column chromatography using EtOAc only to EtOAc/MeOH =100/5 to 100/7 as eluent. After evaporation of the solvent, the residue was diluted with Et_2O and washed with 1 M aqueous NaOH solution. The aqueous layer was extracted with Et_2O and combined organic layer was dried over Na_2SO_4 and evaporated to give (*S*)-2-amino-*N*-(*tert*-butyl)-3-phenylpropanamide (**2r**) as yellow liquid (32.6 mg, 74% isolated yield; 92% yield based on recovered starting material described below).

[22] A. R. Ekkati and J. J. Kodanko, *J. Am. Chem. Soc.*, 2007, **129**, 12390.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.18 (m, 5H), 7.04 (br s, 1H), 3.48 (dd, J = 4.5, 9.0 Hz, 1H), 3.22 (dd, J = 4.5, 14.0 Hz, 1H), 2.70 (dd, J = 9.0, 14.0 Hz, 2H), 1.34 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.5, 138.3, 129.5, 128.8, 126.8, 57.1, 50.6, 41.2, 28.8. $[\alpha]^{24}_D$ – 63.5 (c 0.94, $CHCl_3$).

Enantiomeric excess of **2r** was determined to be 99% after conversion of **2r** as shown below:



2r
(0.0426 mmol)

3
99% ee

HPLC conditions of **3** [CAS Registry No. N/A]: DAICEL CHIRALCEL AD-3, eluent: Hexane/2-Propanol = 9.0/1.0, flow: 1.0 mL/min, detection: 210 nm, t_R: 5.2 min (minor), 7.4 min (major).

*Recovery of starting material **1s***: After purification of the crude mixture by flash silica gel column chromatography as described above, fractions containing **1s** were collected and the solvent was evaporated. The residue was diluted with EtOAc and washed with 1 M aqueous HCl solution, saturated aqueous $NaHCO_3$ solution and brine. The organic phase was dried over Na_2SO_4, filtered and evaporated to give **1s** (18.4 mg, 0.040 mmol, 20% recovery).

Determination of site-selectivity: To clarify site-selectivity of hydrazinolysis of **1s**, a time-course experiment was performed. ^{1}H NMR analysis of the crude mixture showed that **2r**, product after cleavage of Gly-Phe bond, was observed as major species, while (*S*)-2-(2-aminoacetamido)-*N*-*tert*-butyl-3-phenylpropanamide (**S3**), product after cleavage of Val-Gly bond, was not observed as major species throughout the experiment (Fig. S4). These results suggest that Gly-Phe bond was selectively cleaved over Val-Gly bond.

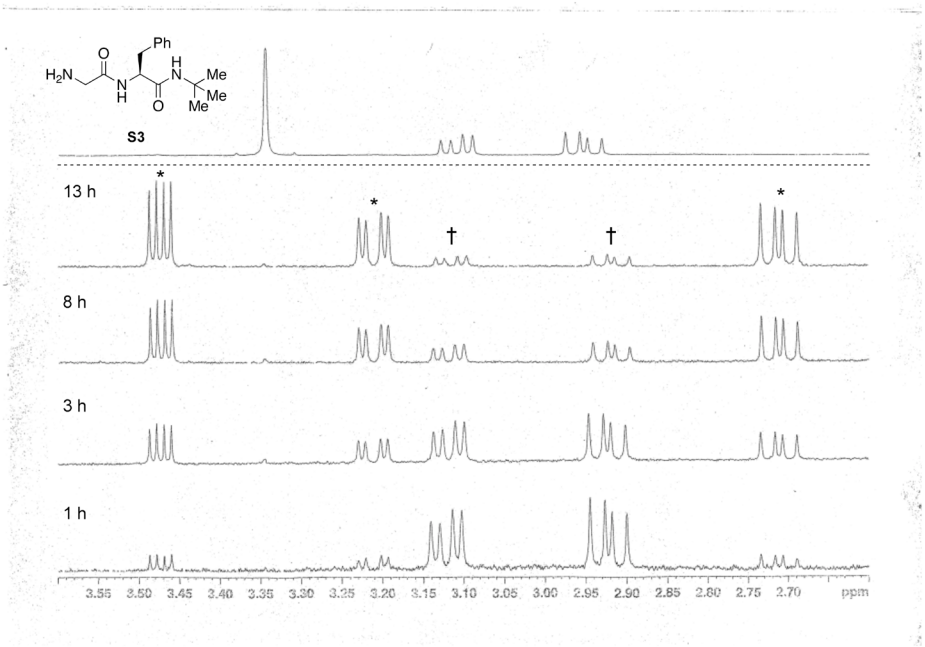
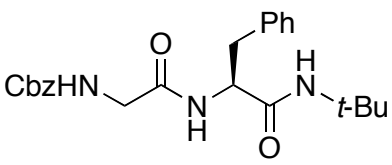


Fig. S4 Time-course experiment of hydrazinolysis of **1s**. † and * indicate peaks of **1s** and **2r**, respectively.

Preparation of (*S*)-2-(2-Aminoacetamido)-*N*-*tert*-butyl-3-phenylpropanamide (S3)

(*S*)-Benzyl 2-(1-(*tert*-butylamino)-1-oxo-3-phenylpropan-2-ylamino)-2-oxoethyl carbamate (S4) [CAS Registry No. N/A]



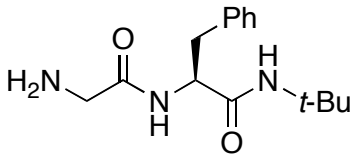
S4

To a solution of (*S*)-*tert*-butyl (1-(*tert*-butylamino)-1-oxo-3-phenylpropan-2-yl)carbamate[13] (160 mg, 0.500 mmol) in CH_2Cl_2 (2.5 mL) at 0 °C was added 4 M HCl in AcOEt (2.5 mL, 10 mmol). The mixture was stirred at room temperature for 1.5 h, and the solvent was removed under reduced pressure. To the crude HCl salt was added *N*-carbobenzoxyglycine (115 mg, 0.550 mmol), HOBt (74 mg, 0.55 mmol) and CH_2Cl_2 (2.5 mL). To the mixture at 0 °C was added EDCI·HCl (105 mg, 0.550 mmol) and Et_3N (0.15 mL, 1.1 mmol). The mixture was stirred at room temperature for 17 h and the solvent was removed under reduced pressure. The residue was diluted with EtOAc and washed twice

with 1 M aqueous HCl solution/brine mixture and twice with saturated aqueous $NaHCO_3$ solution/brine mixture, dried over Na_2SO_4, filtered, evaporated and purified by flash silica gel column chromatography using hexane/EtOAc = 2/1 to 1/4 to give (*S*)-benzyl 2-(1-(*tert*-butylamino)-1-oxo-3-phenylpropan-2-ylamino)-2-oxoethyl carbamate (**S4**) as a white solid (156 mg, 76% yield in 2 steps).

mp 146–148 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45–7.10 (m, 5H), 6.68 (br s, 1H), 5.33 (br s, 1H), 5.13 (s, 2H), 5.09 (br s, 1H), 4.45 (ddd, *J* = 4.0, 8.0, 9.0 Hz, 1H), 3.90 (dd, *J* = 5.5, 17 Hz, 1H), 3.85 (dd, *J* = 6.5, 17 Hz, 1H), 3.14 (dd, *J* = 4.0, 13 Hz, 1H), 2.86 (dd, *J* = 9.0, 13 Hz, 1H), 1.17 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.0, 168.3, 156.5, 136.7, 136.1, 129.4, 128.7, 128.6, 128.3, 128.1, 127.1, 67.3, 55.1, 51.5, 44.5, 39.1, 28.4. IR (KBr disc) 3343, 3291, 3065, 2967, 1707, 1649, 1528, 1456, 1393, 1366, 1234, 1101, 1057, 735, 698 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{23}H_{30}N_3O_4$ [M + H^+] 412.2236, found 412.2232. $[\alpha]^{29}_D$ –0.5 (*c* 1.00, $CHCl_3$).

(*S*)-2-(2-Aminoacetamido)-*N*-*tert*-butyl-3-phenylpropanamide (S3) [CAS Registry No. N/A]

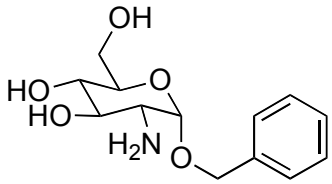


S3

A mixture of **S4** (112.3 mg, 0.273 mmol) and 10% Pd/C (12.4 mg, 0.0117 mmol) in EtOH (2.7 mL) was stirred under H_2 atmosphere (1 atm) at room temperature for 2 h. The mixture was filtered through a pad of Celite, and the filtrate was evaporated and purified by flash silica gel column chromatography using EtOAc/MeOH = 4/1 to 1/1 as eluent to give (*S*)-2-(2-aminoacetamido)-*N*-*tert*-butyl-3-phenylpropanamide (**S3**) as colorless oil (55.3 mg, 73% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.0 Hz, 1H), 7.37–7.18 (m, 5H), 5.28 (br s, 1H), 4.45 (ddd, *J* = 6.0, 8.0, 9.0 Hz, 1H), 3.35 (s, 2H), 3.11 (dd, *J* = 6.0, 13.5 Hz, 1H), 2.95 (dd, *J* = 9.0, 13.5 Hz, 1H), 1.55 (br s, 2H), 1.19 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.6, 169.5, 137.1, 129.4, 128.6, 126.9, 54.9, 51.3, 44.8, 38.9, 28.5. IR (neat, NaCl) 3291, 2969, 1651, 1557, 1454, 1362, 1225, 745, 700 cm^{-1}. HRMS (ESI-TOF) *m/z* calcd. for $C_{15}H_{24}N_3O_2$ [M + H^+] 278.1869, found 278.1862. $[\alpha]^{30}_D$ –8.6 (*c* 0.98, $CHCl_3$).

Benzyl 2-amino-2-deoxy-α-D-glucopyranoside (2t) [CAS Registry No. 50692-69-8][23]



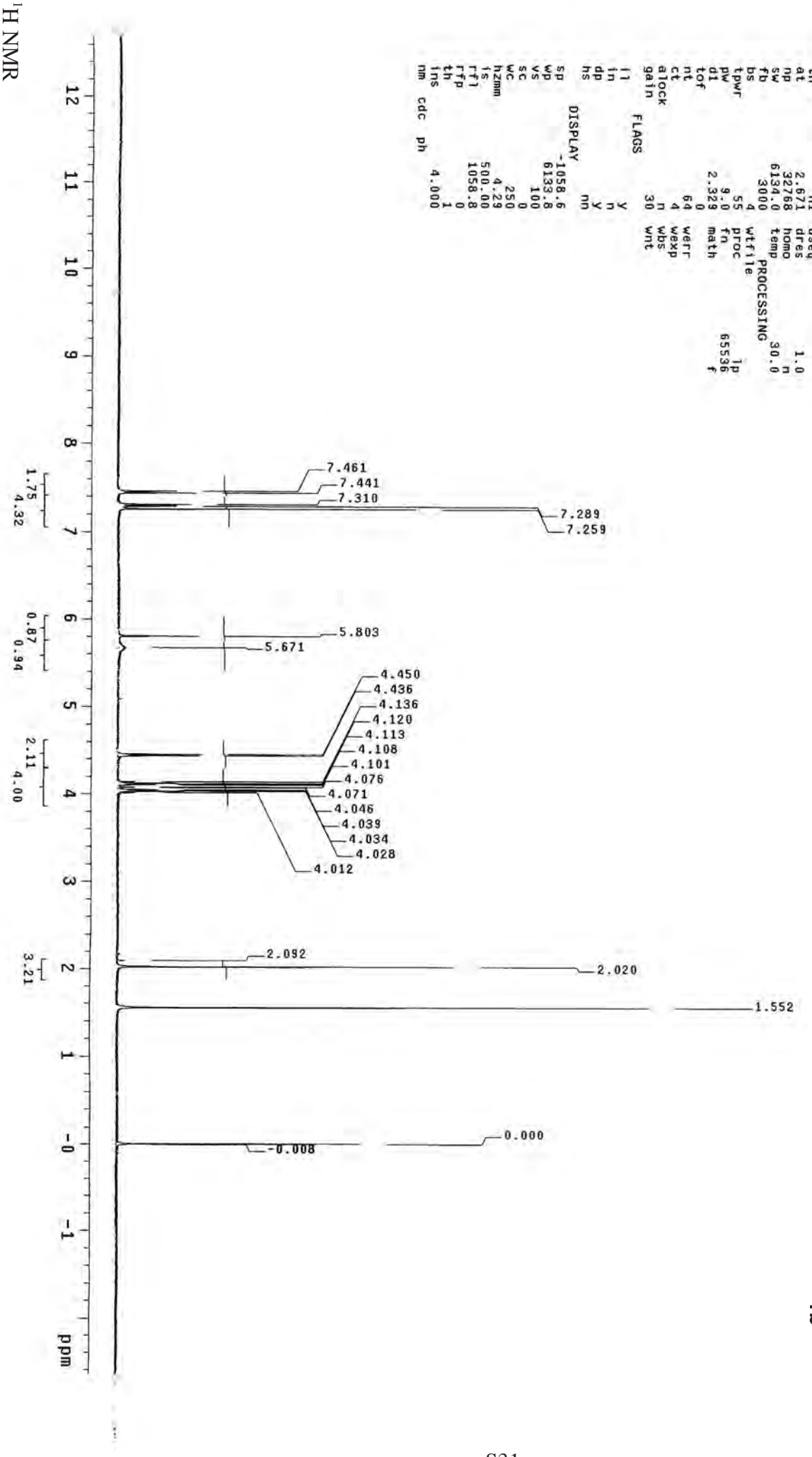
2t

To a 10 mL of a glass test tube equipped with a magnetic stir bar, ammonium iodide (36.2 mg, 0.250 mmol), **1t** (0.250 mmol) and hydrazine monohydrate (0.25 mL, 2.5 mmol) were added and the tube was sealed with a cap. The test tube was heated with stirring at the indicated temperature and time under microwave irradiation conditions. The crude mixture was directly purified by flash silica gel column chromatography using dichloromethane/MeOH/diethylamine = 270/30/1 to 240/60/1 as eluent. After evaporation of the solvent, the residue was treated with Et_2O and filtered to give benzyl 2-amino-2-deoxy-α-D-glucopyranoside (**2t**) as a white solid (54.3 mg, 81% yield).

^{1}H NMR (500 MHz, DMSO-d_6) δ 4.93–4.82 (m, 2H), 4.75 (d, J = 4.0 Hz, 1H), 4.68 (d, J = 12.0 Hz, 1H), 3.70 (t, J = 6.0 Hz, 1H), 4.43 (d, J = 12.0 Hz, 1H), 3.65 (ddd, J = 6.0, 6.0, 6.0 Hz, 1H), 3.52–3.41 (m, 2H), 3.24 (ddd, J = 4.0, 9.5, 9.5 Hz, 1H), 3.07 (ddd, J = 5.0, 9.5, 9.5 Hz, 1H), 2.43 (dd, J = 4.0, 9.5 Hz, 1H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 151.5, 129.3, 127.2, 124.6, 118.6, 109.4, 47.3, 29.8. $[\alpha]^{27}_D$ +142.9 (*c* 1.00, MeOH).

[23] A. Burkhardt, H. Görls and W. Plass, *Carbohyd. Res.*, 2008, **343**, 1266.

^{1}H NMR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d1 2.329
tof 0
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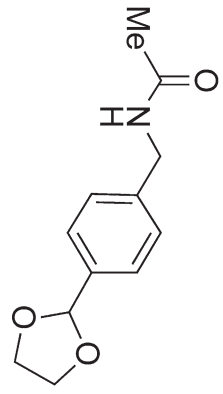
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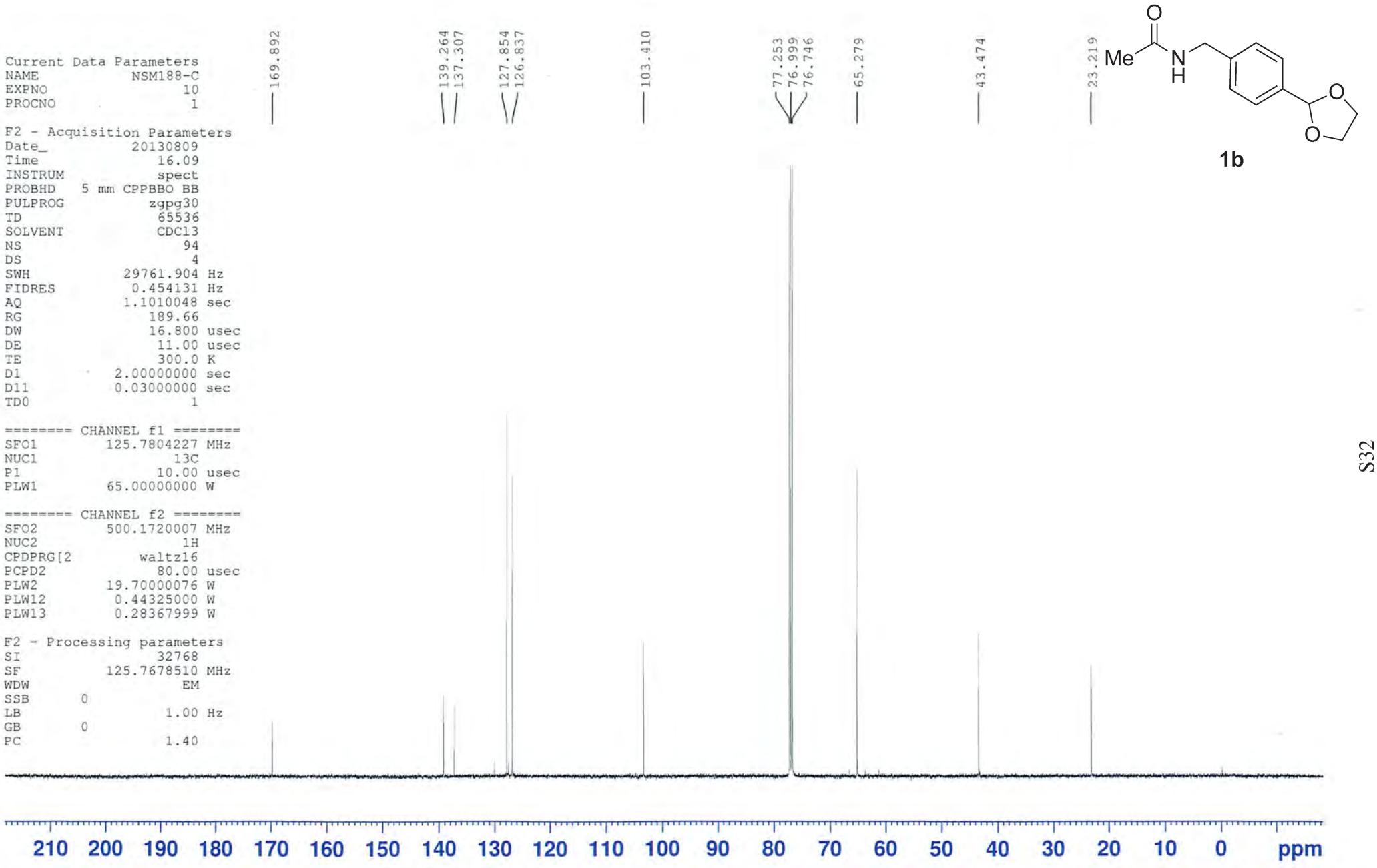
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NUC1 13C
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PLW1 65.00000000 W
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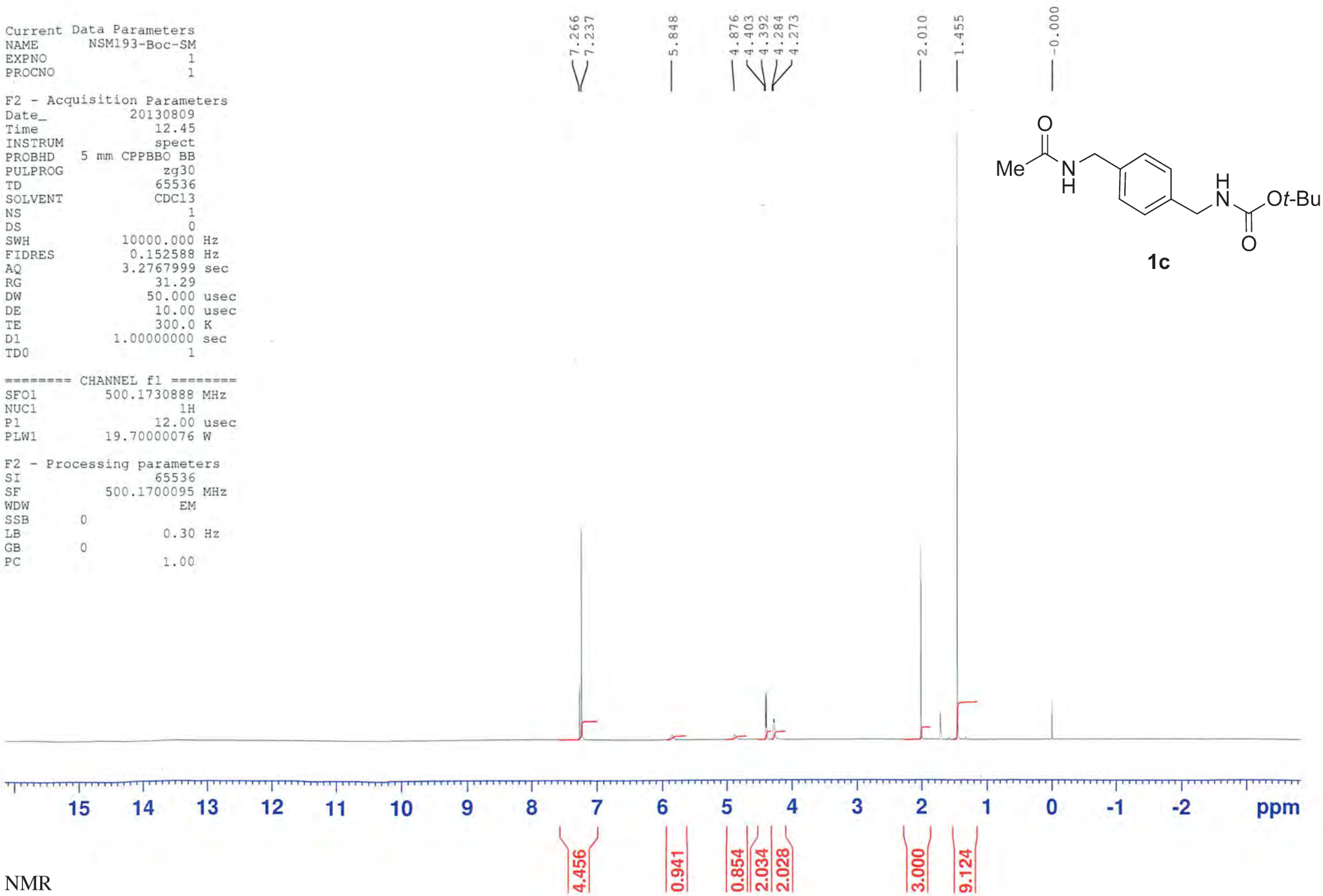
^{13}C NMR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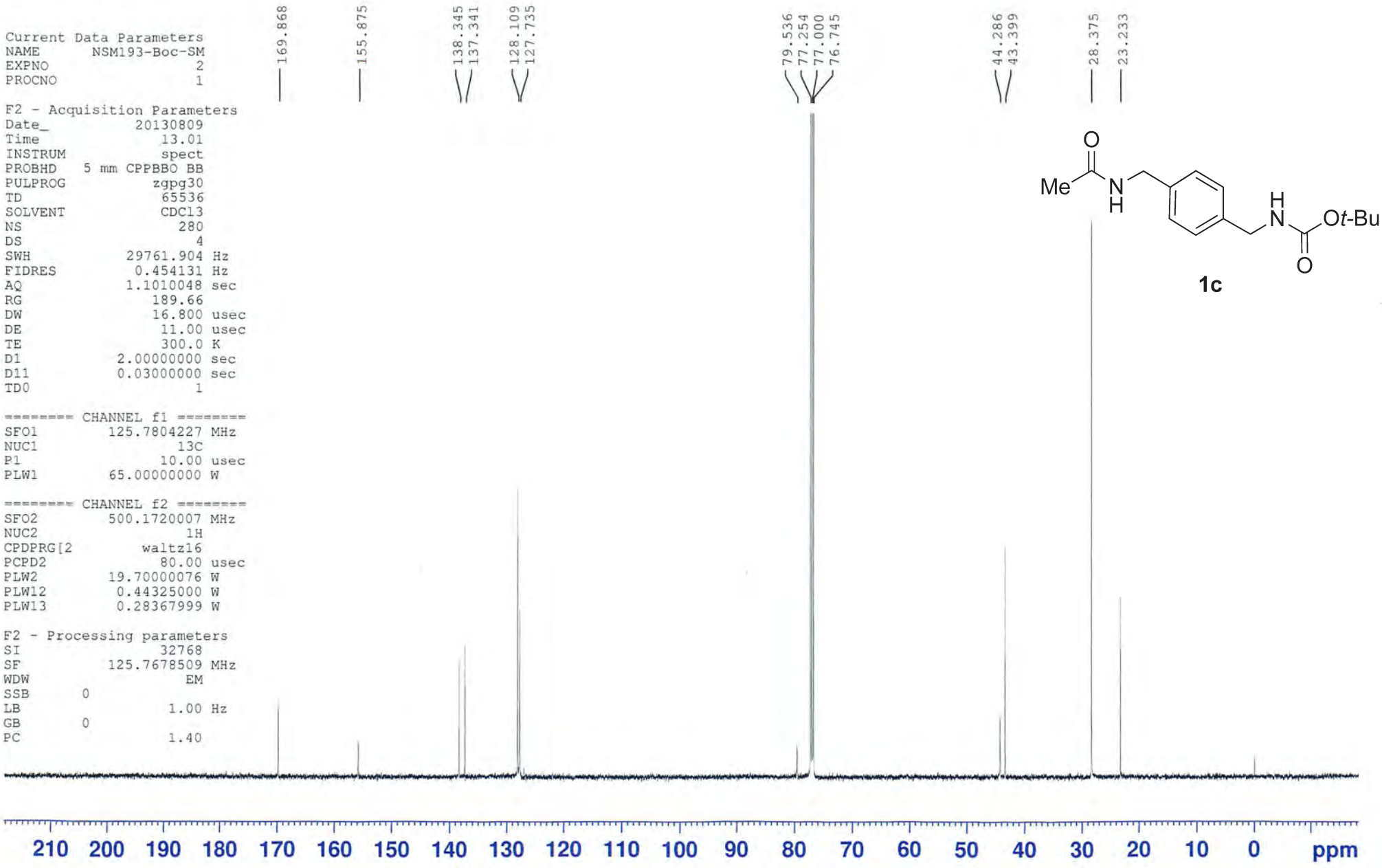
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NUC2 1H
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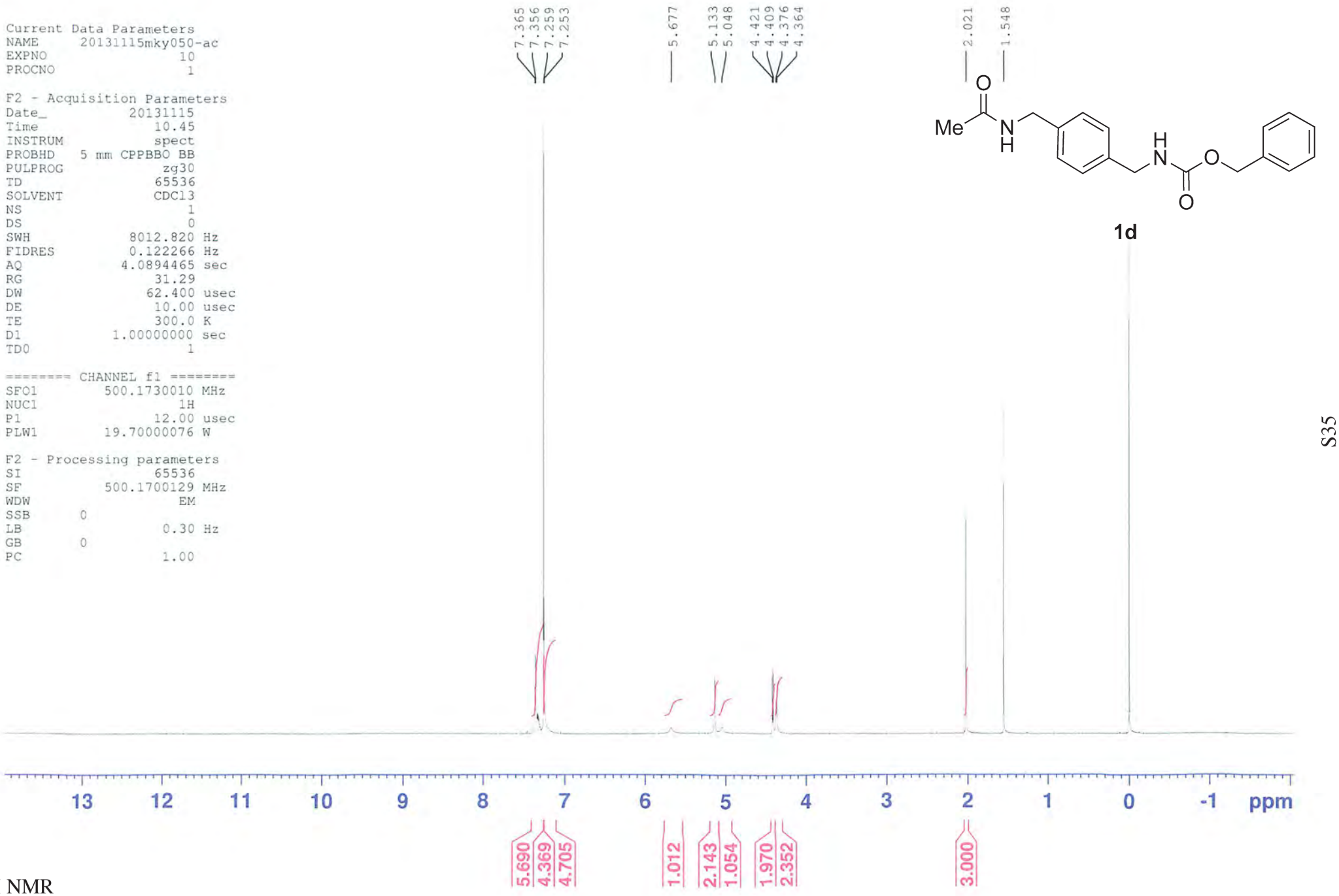
^{13}C NMR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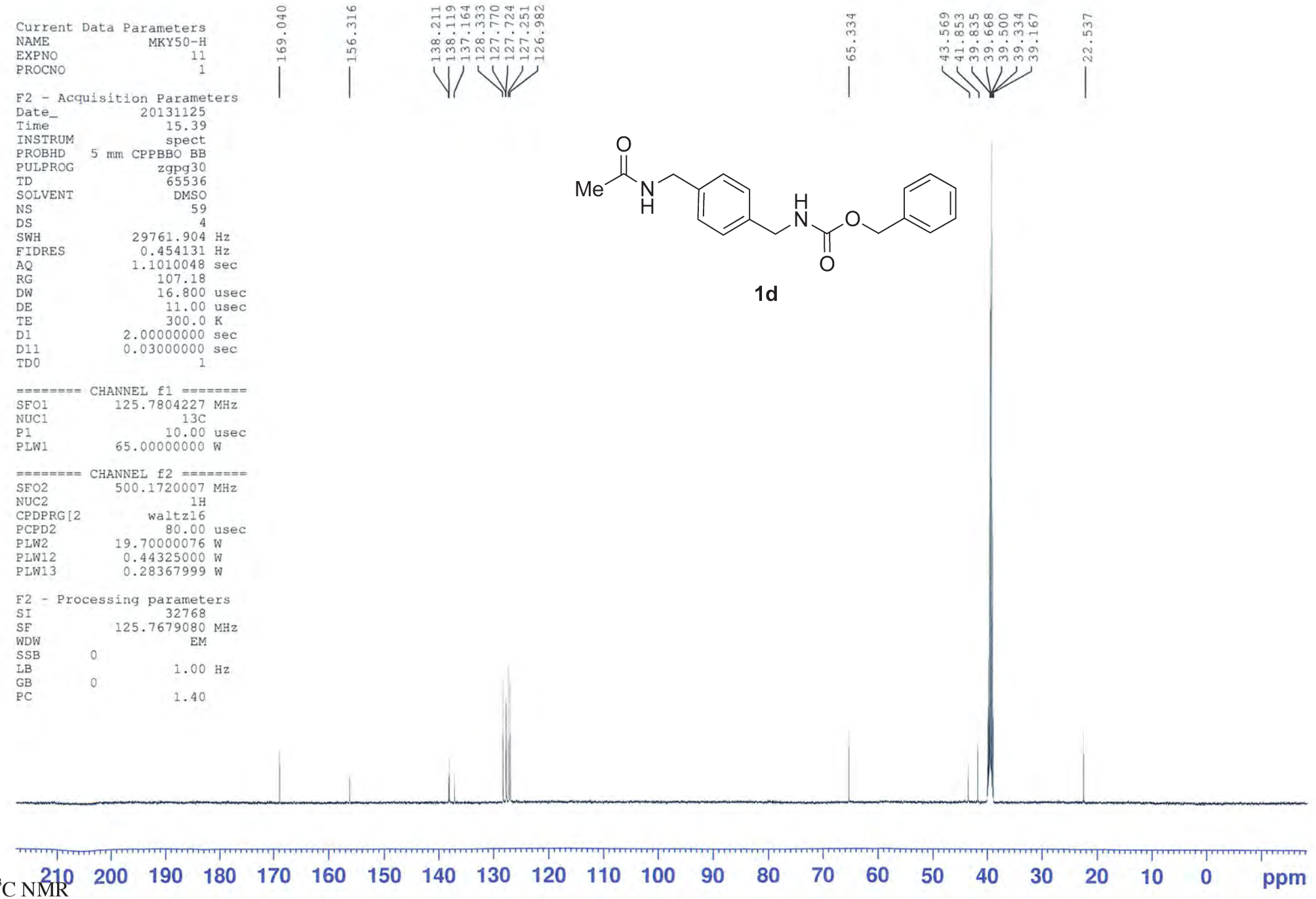
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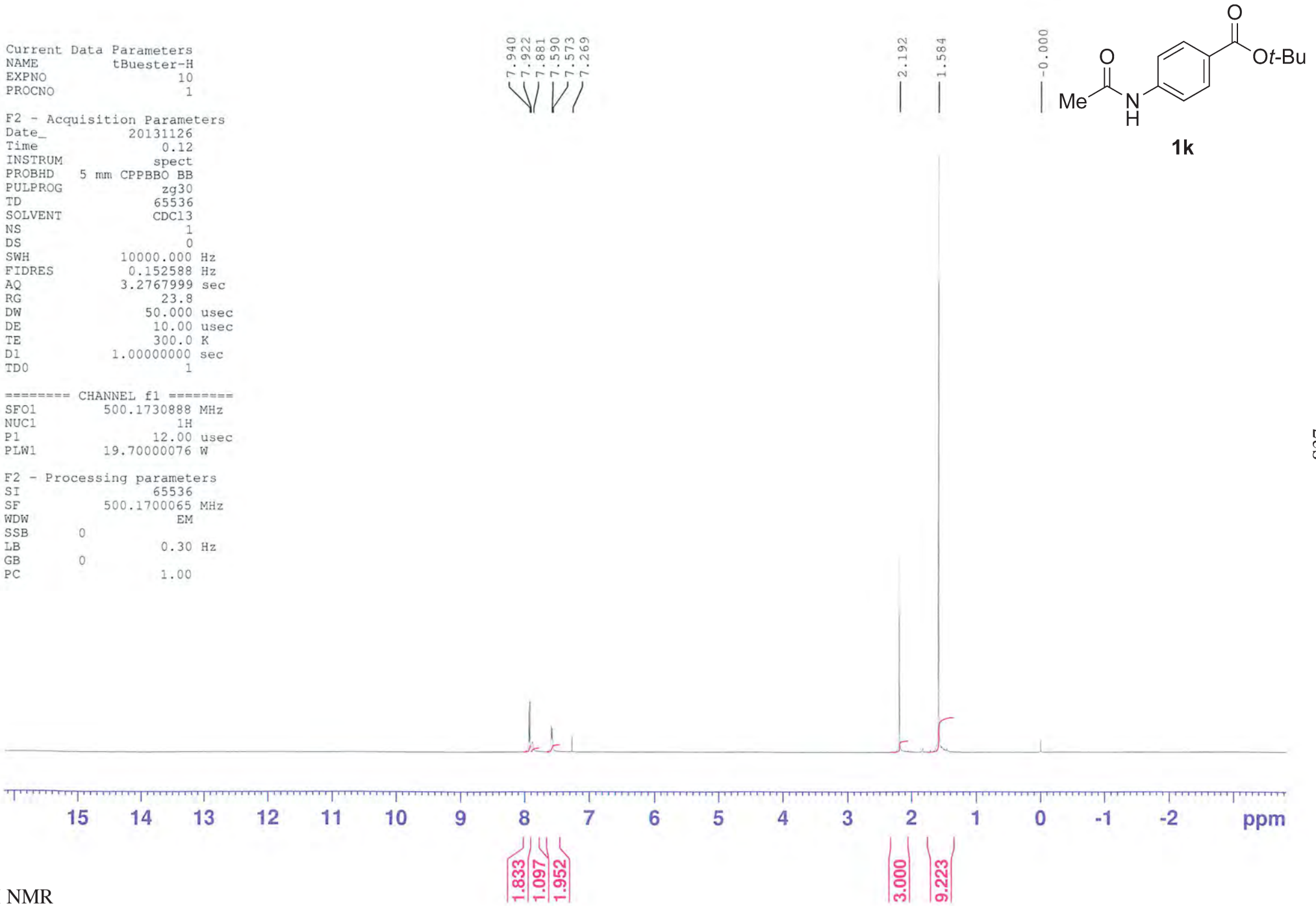
^{13}C NMR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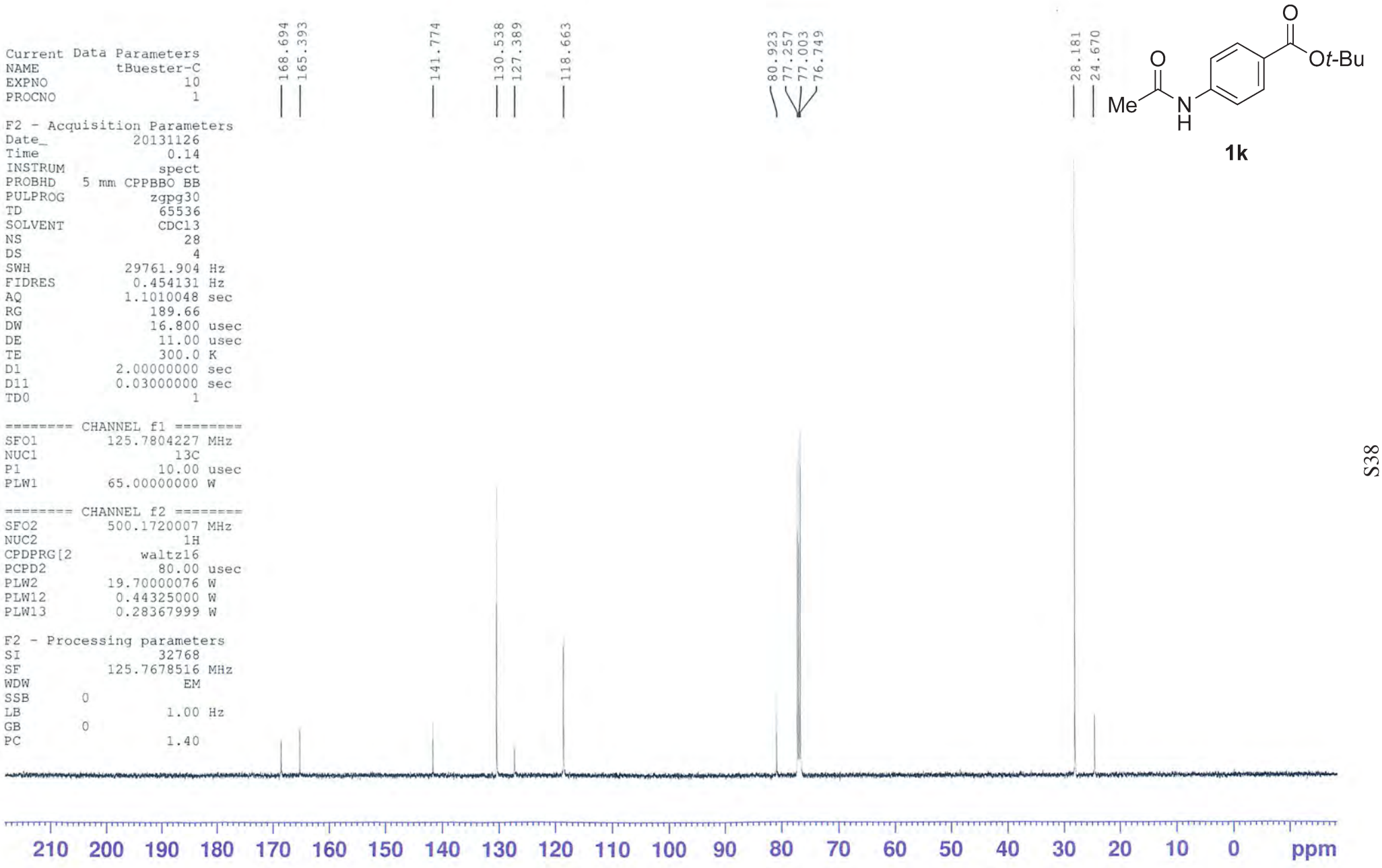
^{1}H NMR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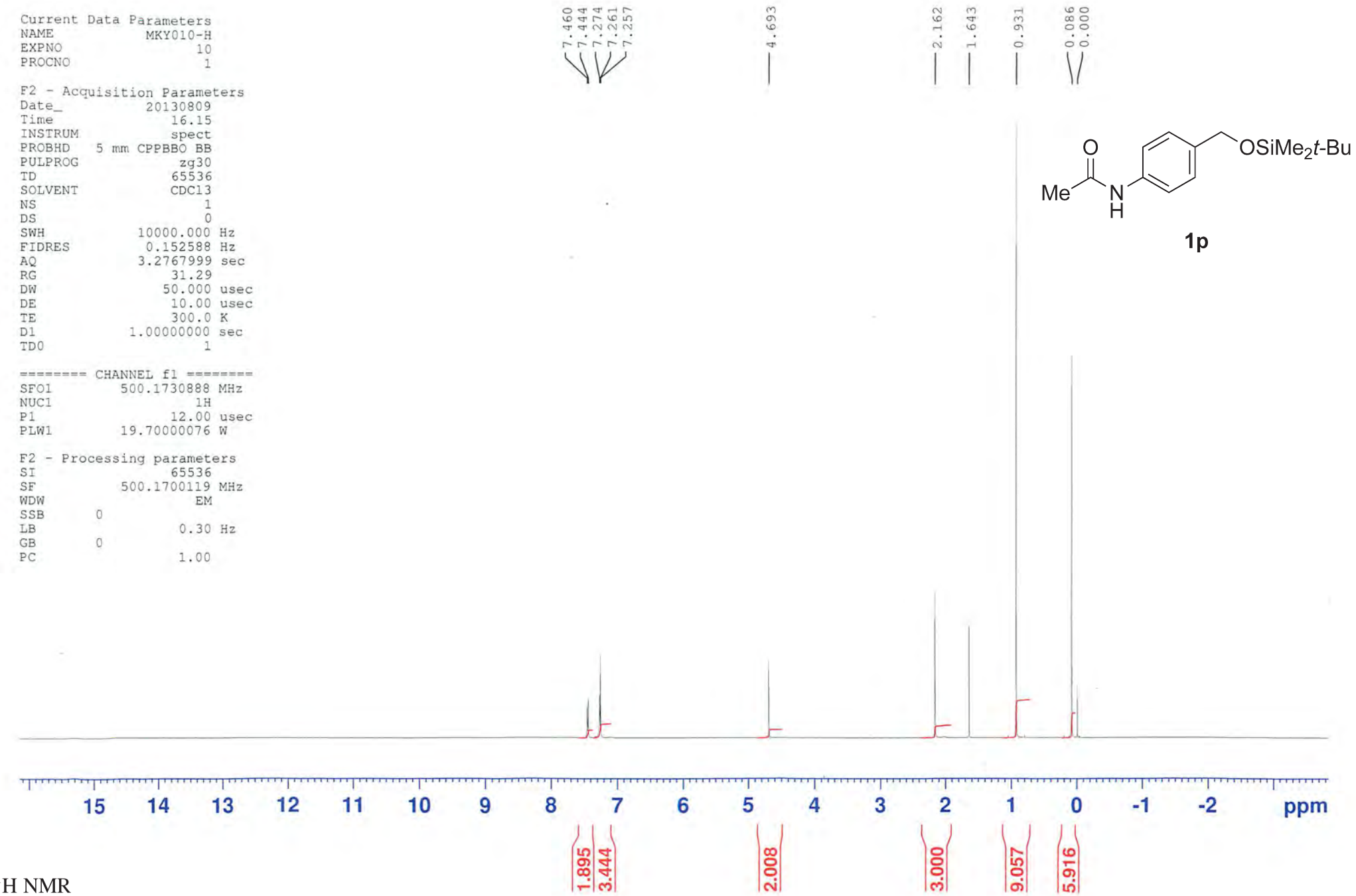
^{13}C NMR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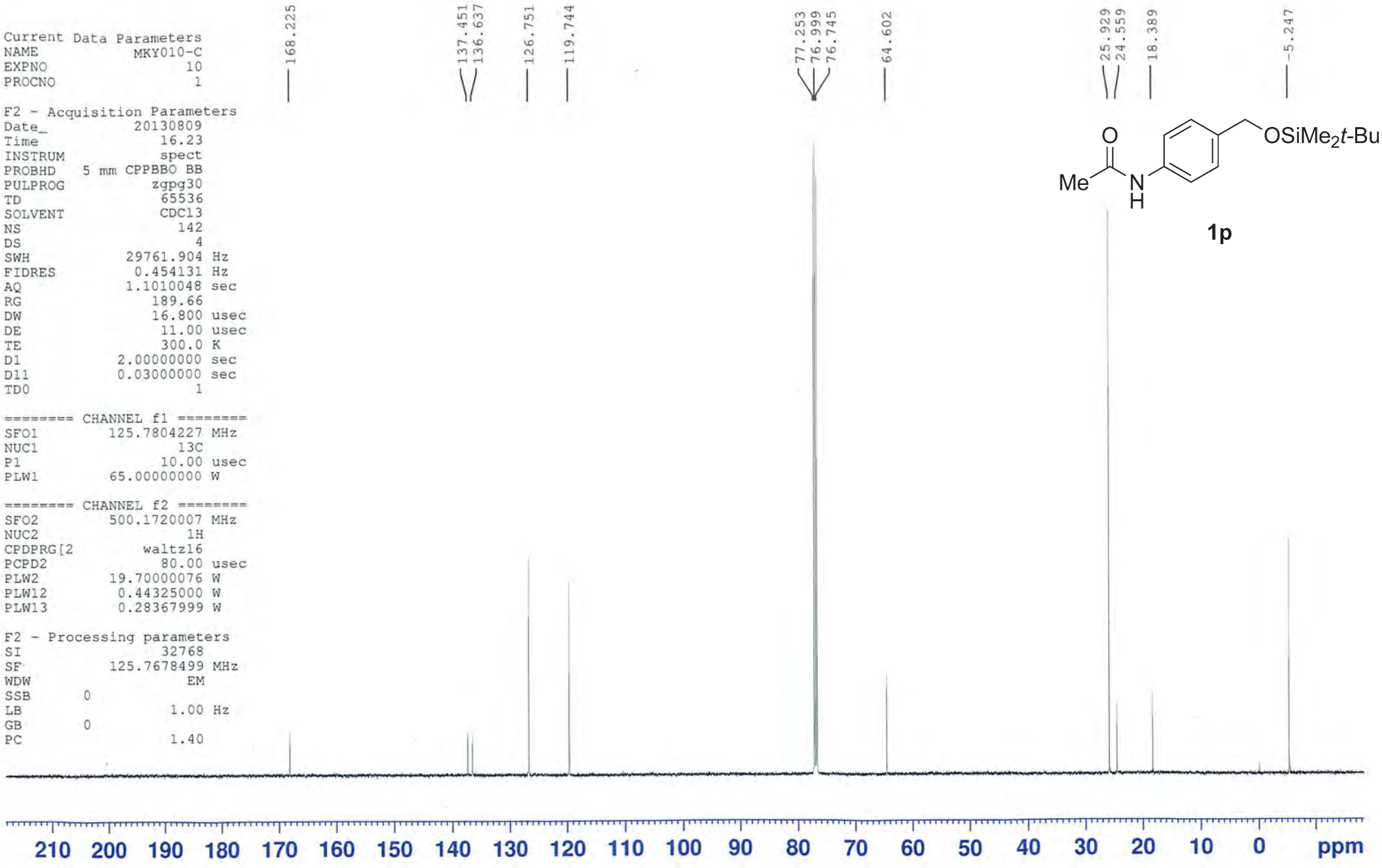
^{1}H NMR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NUC1 13C
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NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
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PLW12 0.44325000 W
PLW13 0.28367999 W
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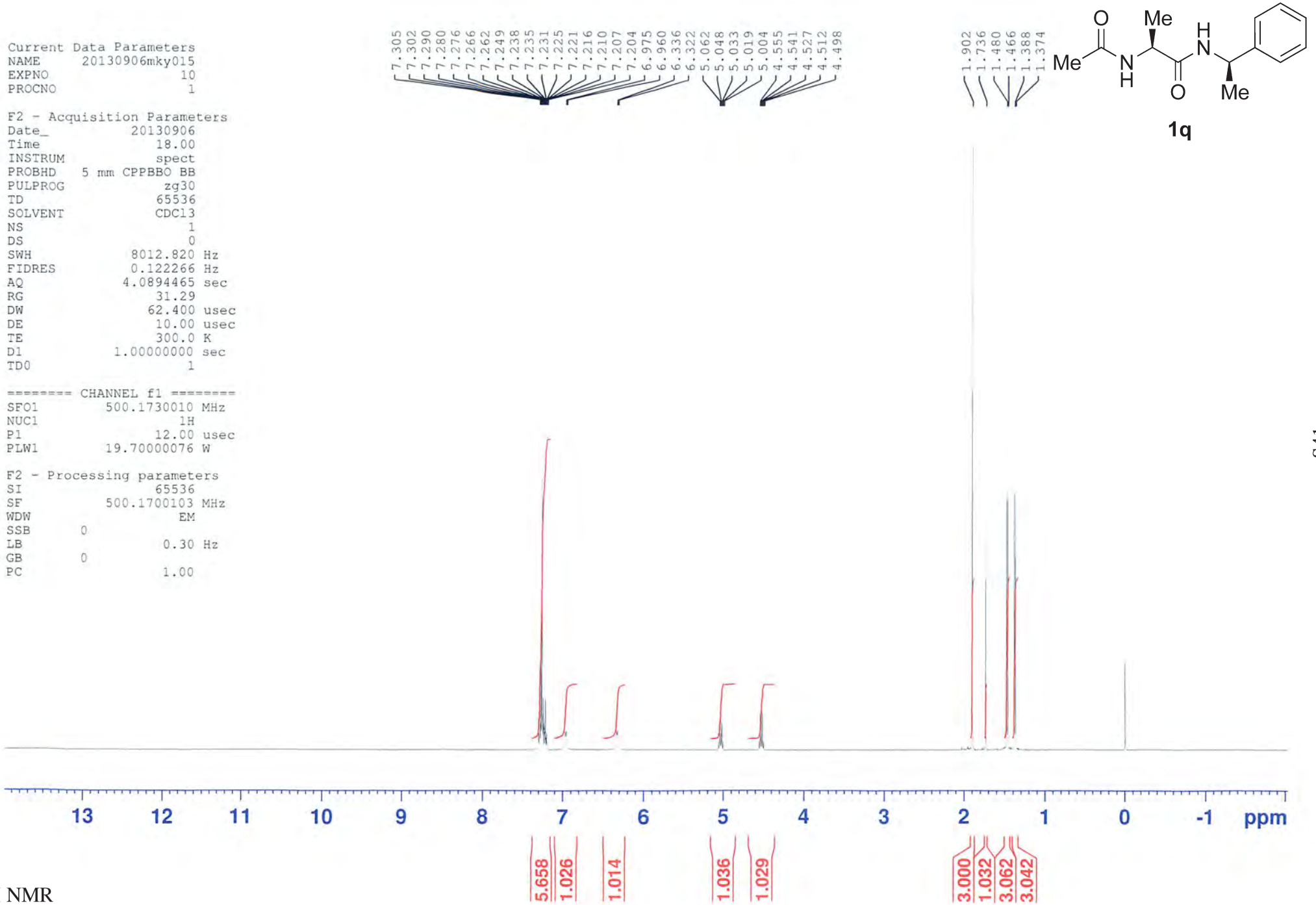
168.225
137.451
136.637
126.751
119.744
77.253
76.999
76.745
64.602
25.929
24.559
18.389
-5.247
O
Me
N
H
OSiMe2t-Bu
1p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR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NAME 20130906mky015
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130906
Time 18.00
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PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.305
7.302
7.290
7.280
7.276
7.266
7.262
7.249
7.238
7.235
7.231
7.225
7.221
7.216
7.210
7.207
7.204
6.975
6.960
6.336
6.322
5.062
5.048
5.033
5.019
5.004
4.555
4.541
4.527
4.512
4.498
1.902
1.736
1.480
1.466
1.388
1.374
O
Me
H
N
Me
N
H
O
Me
1q
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
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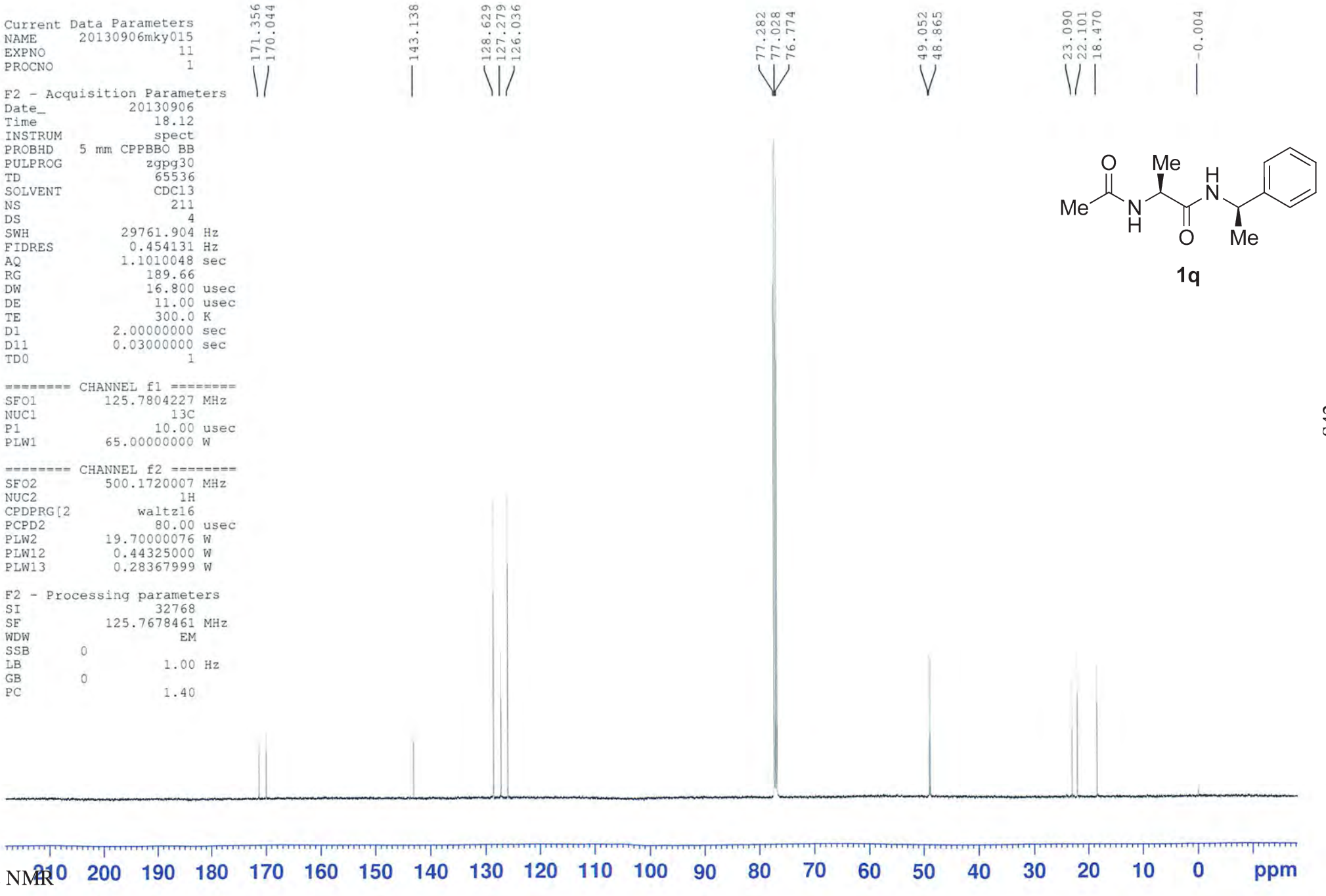
1.026
1.014
1.036
1.029
3.000
1.032
3.062
3.042

^{1}H NMR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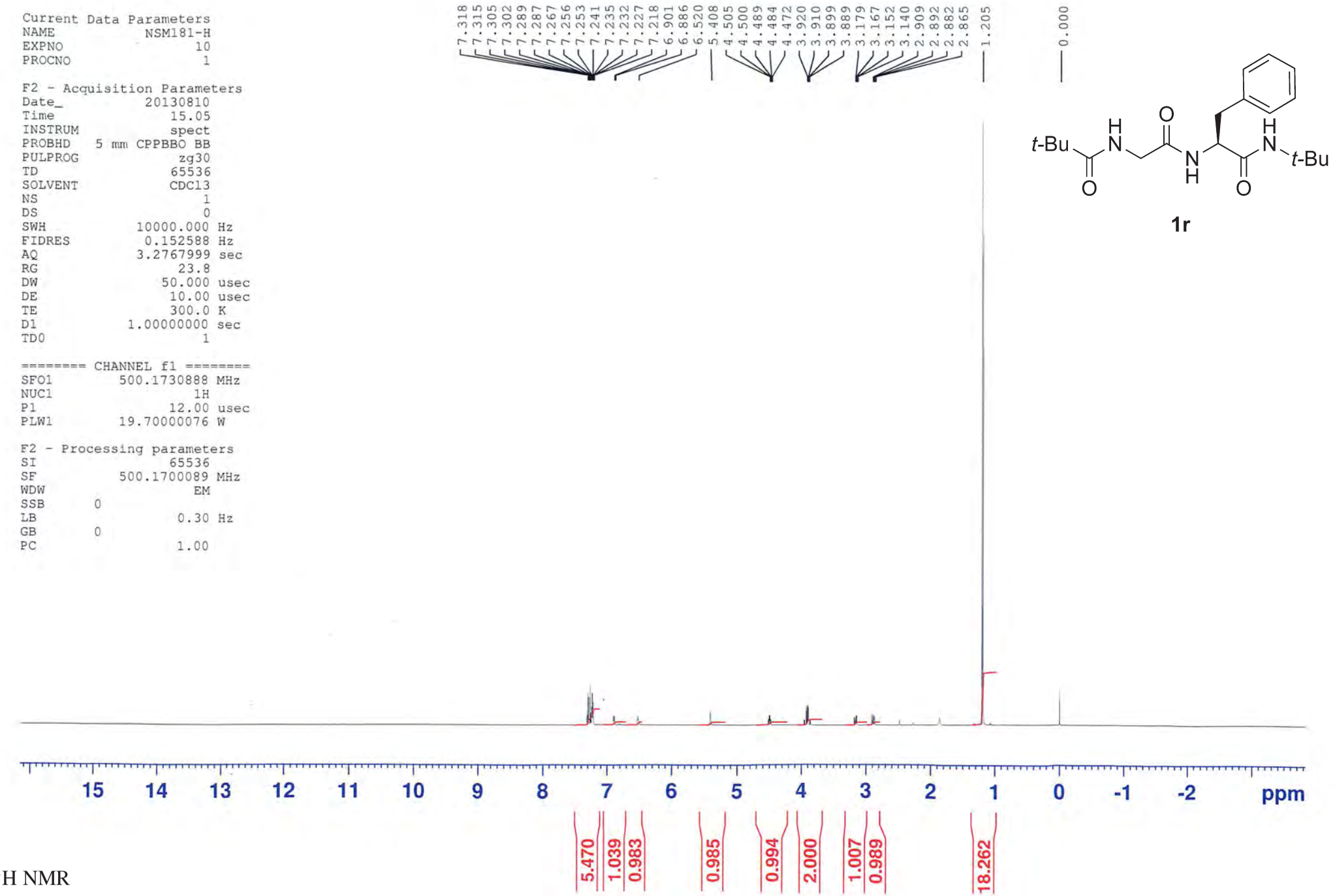
NAME 20130906mky015
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130906
Time 18.12
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 211
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7678461 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
171.356
170.044
143.138
128.629
127.279
126.036
77.282
77.028
76.774
49.052
48.865
23.090
22.101
18.470
-0.004
O
Me
H
N
Me
N
H
O
Me
1q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR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NAME NSM181-H
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130810
Time 15.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 23.8
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.318
7.315
7.305
7.302
7.289
7.287
7.267
7.256
7.253
7.241
7.235
7.232
7.227
7.218
6.901
6.886
6.520
5.408
4.505
4.500
4.489
4.484
4.472
3.920
3.910
3.899
3.889
3.179
3.167
3.152
3.140
2.909
2.892
2.882
2.865
1.205
0.000
t-Bu
H
N
O
N
H
O
H
N
t-Bu
O
1r
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
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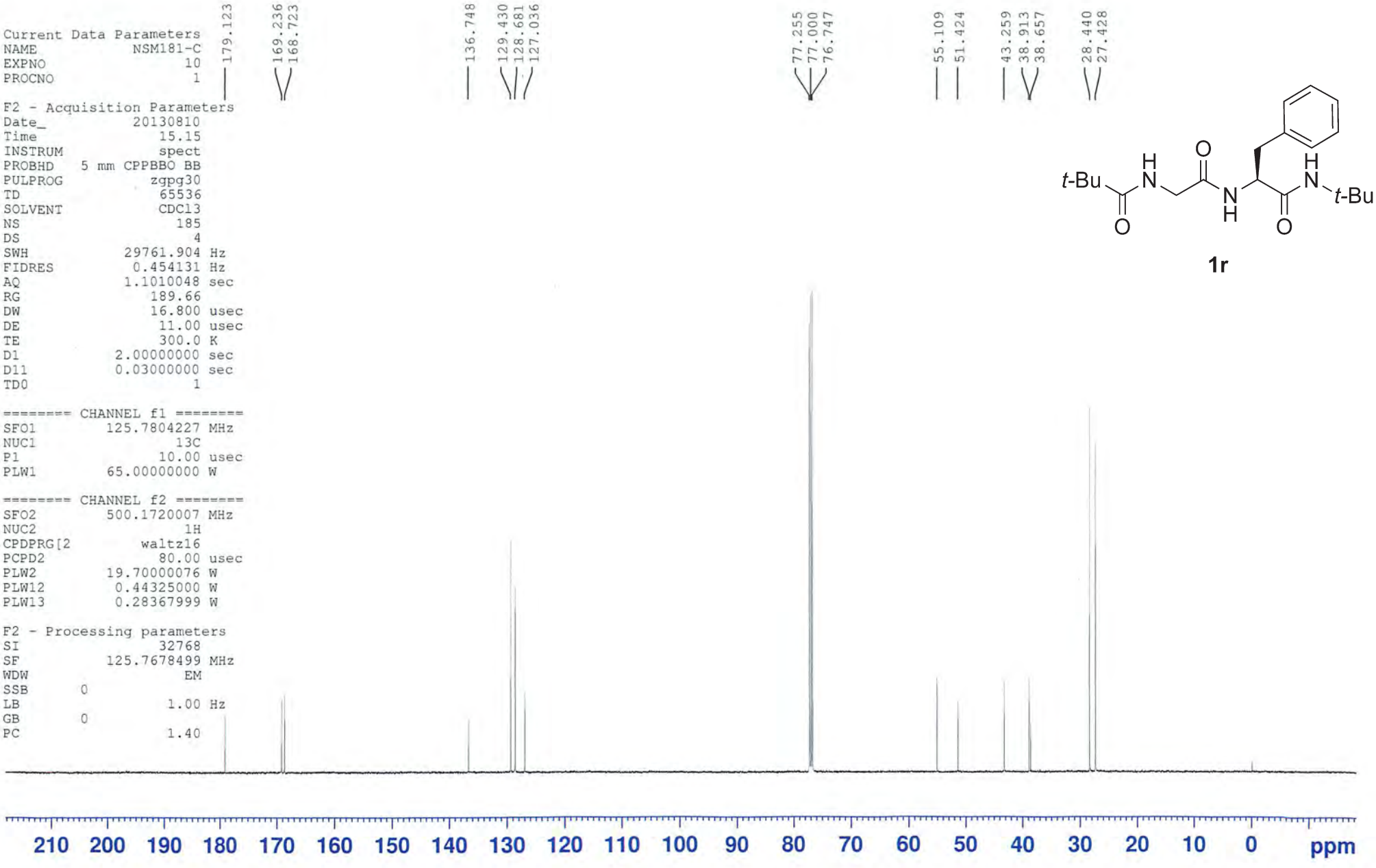
1.039
0.983
0.985
0.994
2.000
1.007
0.989
18.262

^{1}H NMR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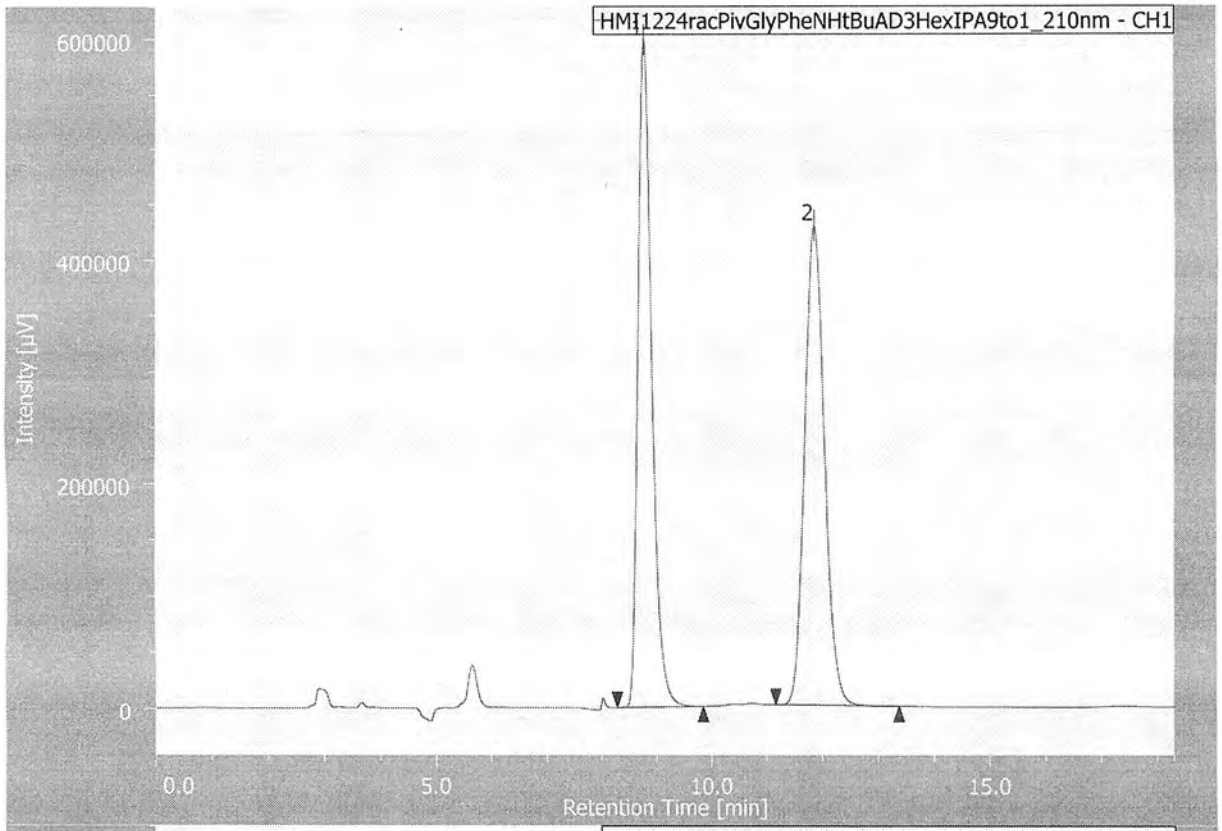
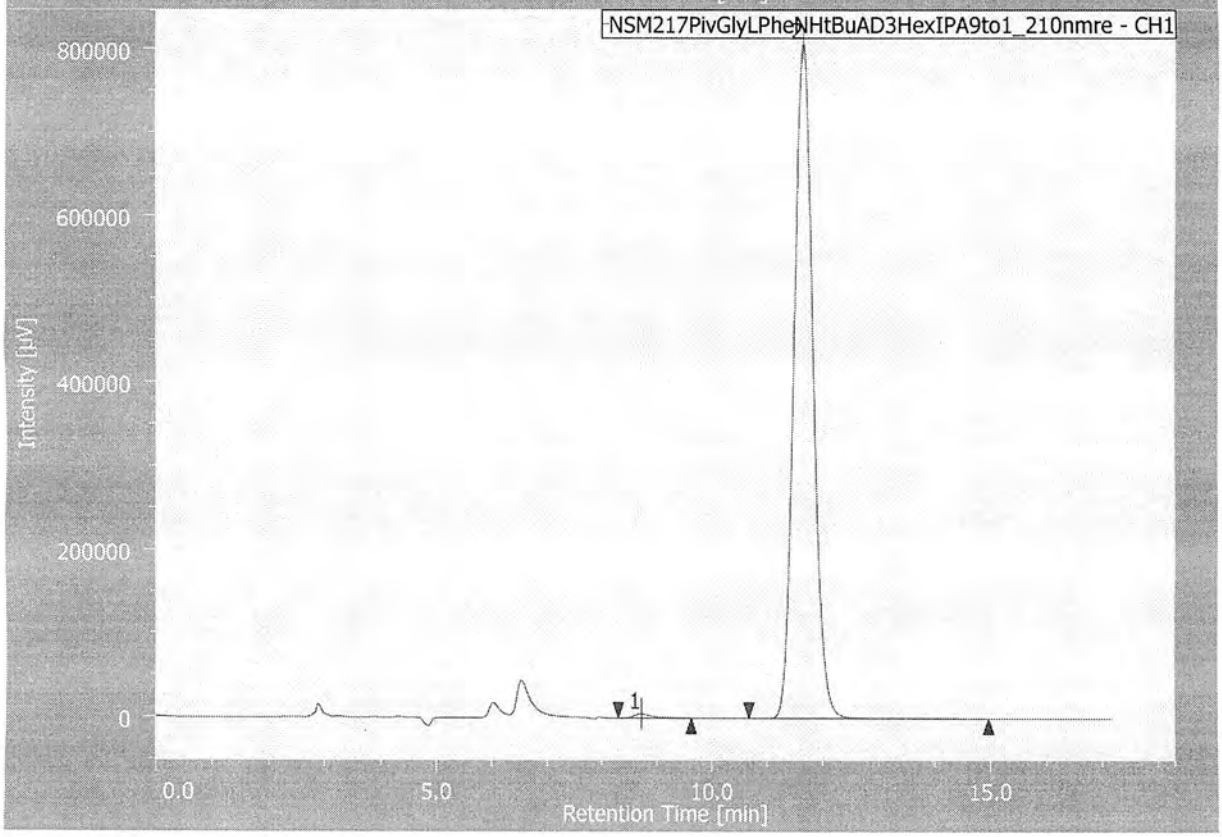
NAME NSM181-C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130810
Time 15.15
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 185
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7678499 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
179.123
169.236
168.723
136.748
129.430
128.681
127.036
77.255
77.000
76.747
55.109
51.424
43.259
38.913
38.657
28.440
27.428
t-Bu
H
N
O
N
H
O
H
N
t-Bu
O
1r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

HPLC





チャンネル情報＋ピーク情報

クロマトグラム名　HMI1224racPivGlyPheNHtBuAD3HexIPA9to1_210nm-CH1

サンプル名

チャンネル名　CH1

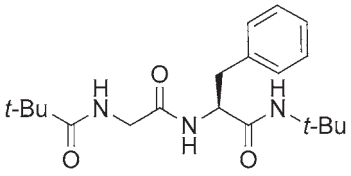
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	8.77	10428114	596845	49.756	58.273	N/A	6165	5.663	1.364	
2	Unknown	1	11.85	10530357	427369	50.244	41.727	N/A	5398	N/A	1.103	

クロマトグラム名　NSM217PivGlyLPheNHtBuAD3HexIPA9to1_210nmre-CH1

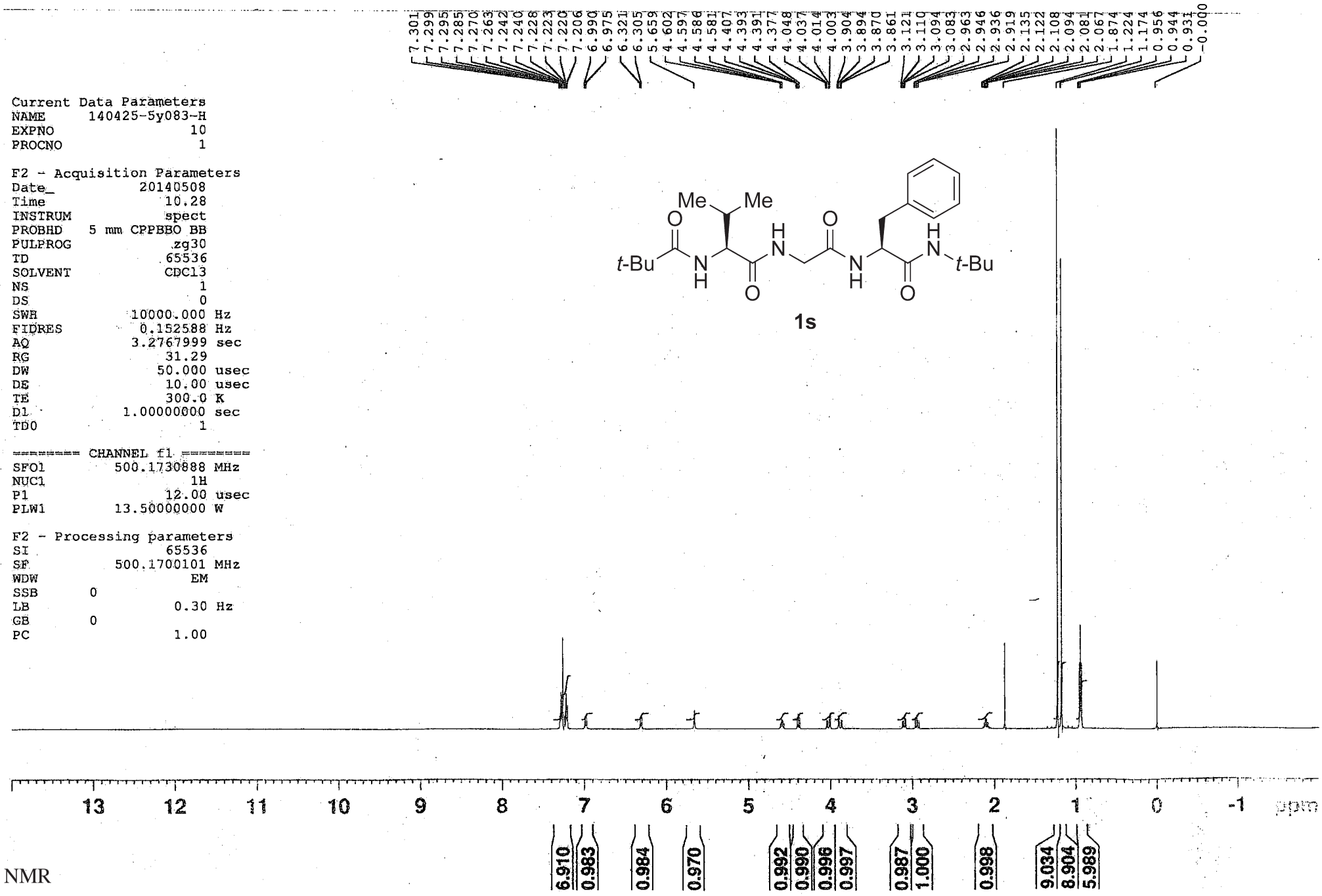
サンプル名

チャンネル名　CH1

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	8.72	112909	4795	0.583	0.591	N/A	4015	5.003	1.652	
2	Unknown	1	11.65	19245585	806470	99.417	99.409	N/A	5552	N/A	1.077	



1r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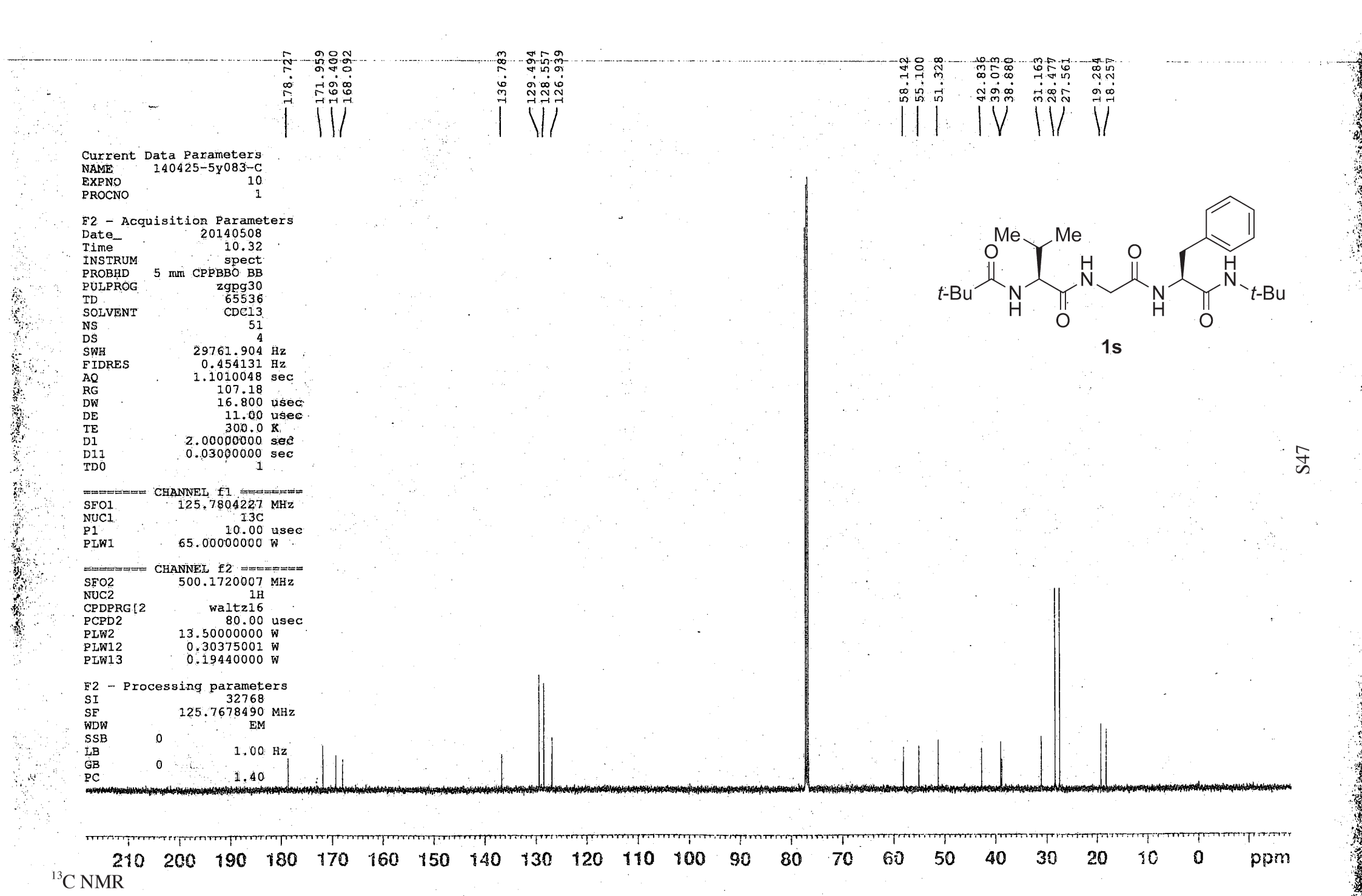
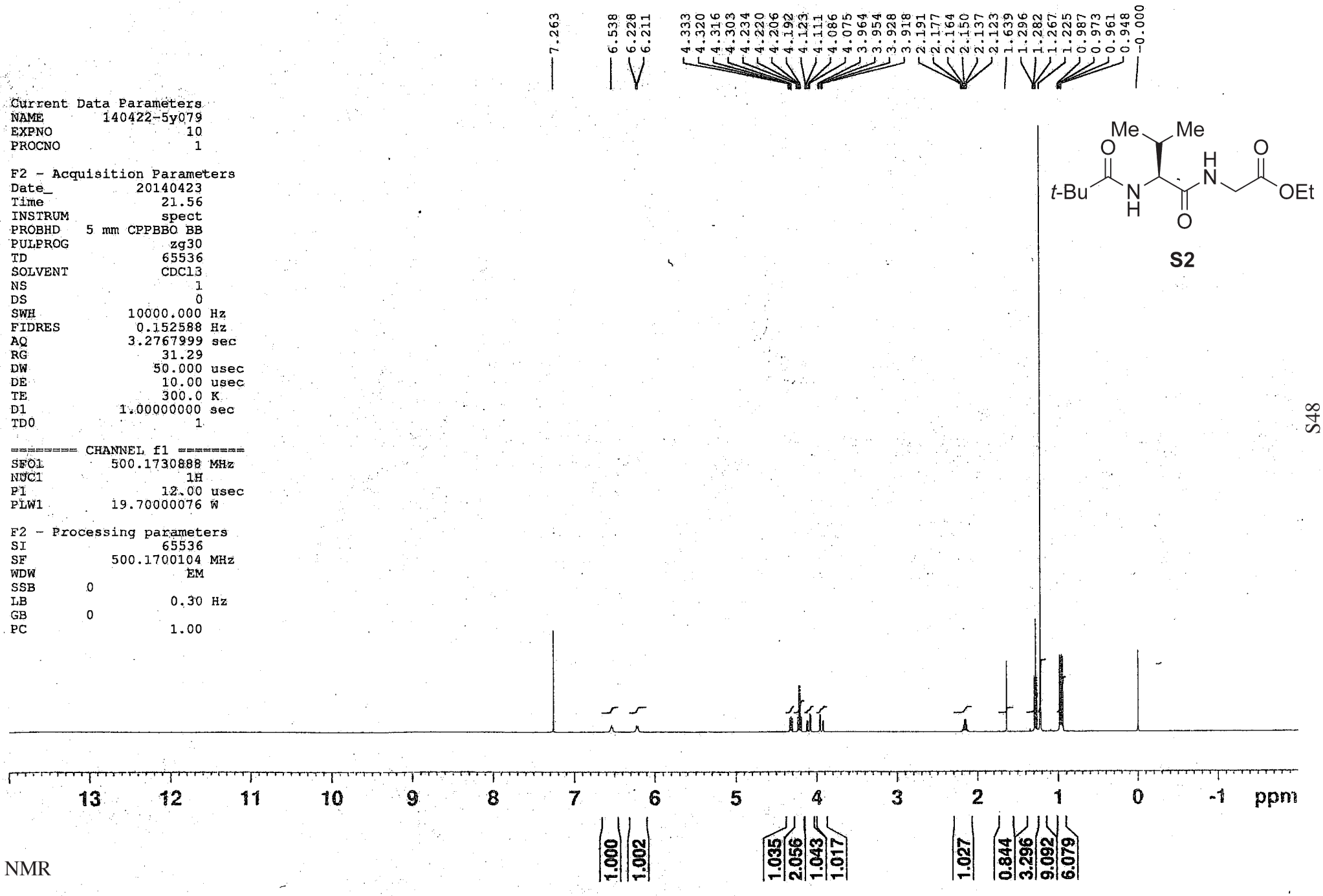
7.295
7.285
7.270
7.263
7.242
7.240
7.228
7.223
7.220
7.206
6.990
6.975
6.321
6.305
5.659
4.602
4.597
4.586
4.581
4.407
4.393
4.391
4.377
4.048
4.037
4.014
4.003
3.904
3.894
3.870
3.861
3.121
3.110
3.094
3.083
2.963
2.946
2.936
2.919
2.135
2.122
2.108
2.094
2.081
2.067
1.874
1.224
1.174
0.956
0.944
0.931
-0.000
Current Data Parameters
NAME 140425-5y083-H
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140508
Time 10.28
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 31.29
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
O
H
N
O
t-Bu
N
H
O
H
N
O
N
H
t-Bu
1s
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
6.910
0.983
0.984
0.970
0.992
0.990
0.996
0.997
0.987
1.000
0.998
9.034
8.904
5.989

^{1}H NMR


178.727
171.959
169.400
168.092
136.783
129.494
128.557
126.939
58.142
55.100
51.328
42.836
39.073
38.880
31.163
28.477
27.561
19.284
18.257
Current Data Parameters
NAME 140425-5y083-C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140508
Time 10.32
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.18
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
Me
O
t-Bu
N
H
O
H
N
O
H
N
O
N
H
O
t-Bu
1s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR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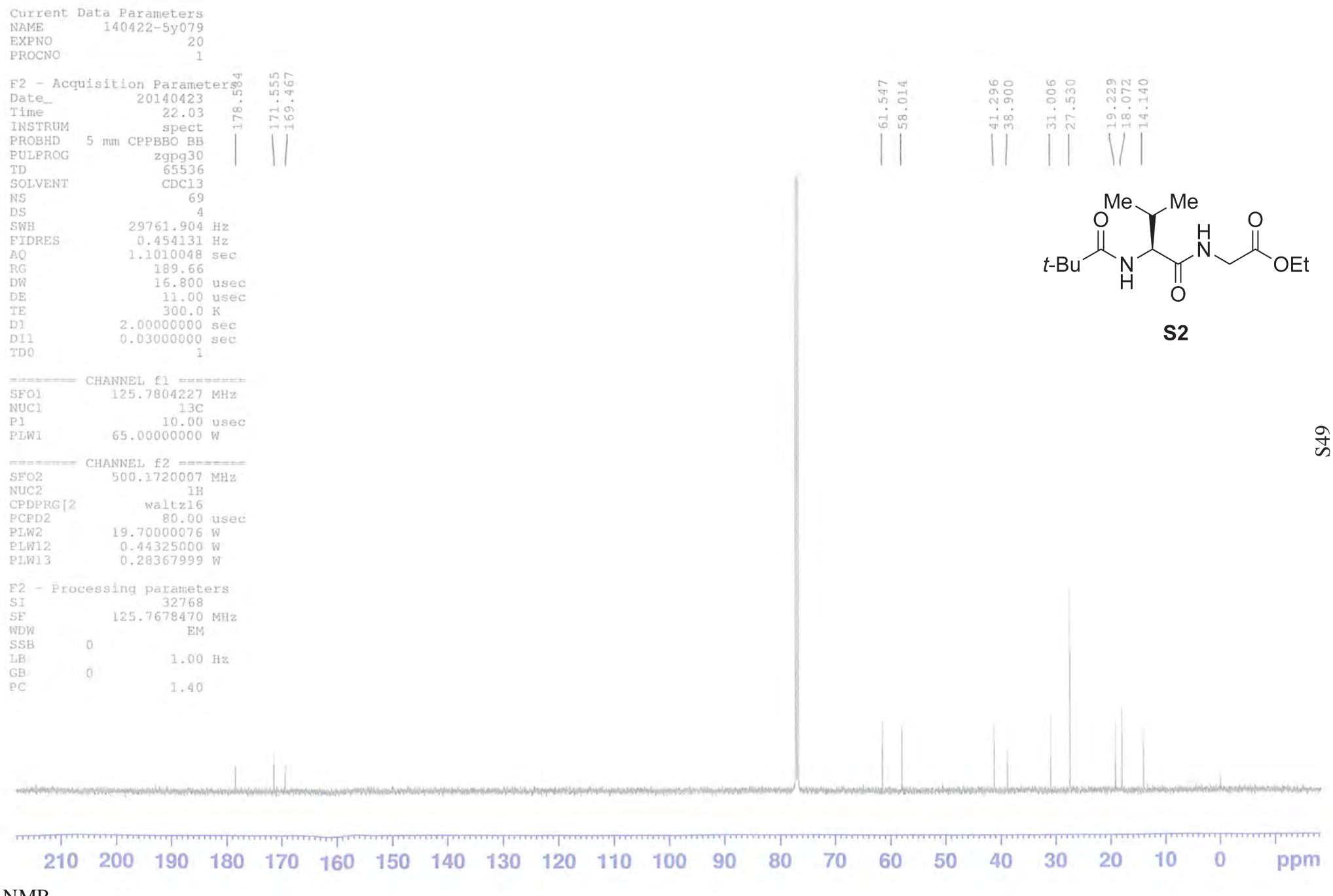
6.228
6.211
4.333
4.320
4.316
4.303
4.234
4.220
4.206
4.192
4.123
4.111
4.086
4.075
3.964
3.954
3.928
3.918
2.191
2.177
2.164
2.150
2.137
2.123
1.639
1.296
1.282
1.267
1.225
0.987
0.973
0.961
0.948
-0.000
Current Data Parameters
NAME 140422-5y079
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140423
Time 21.56
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 31.29
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
O
O
H
N
t-Bu
N
O
OEt
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.000
1.002
1.035
2.056
1.043
1.017
1.027
0.844
3.296
9.092
6.079

S2

^{1}H NMR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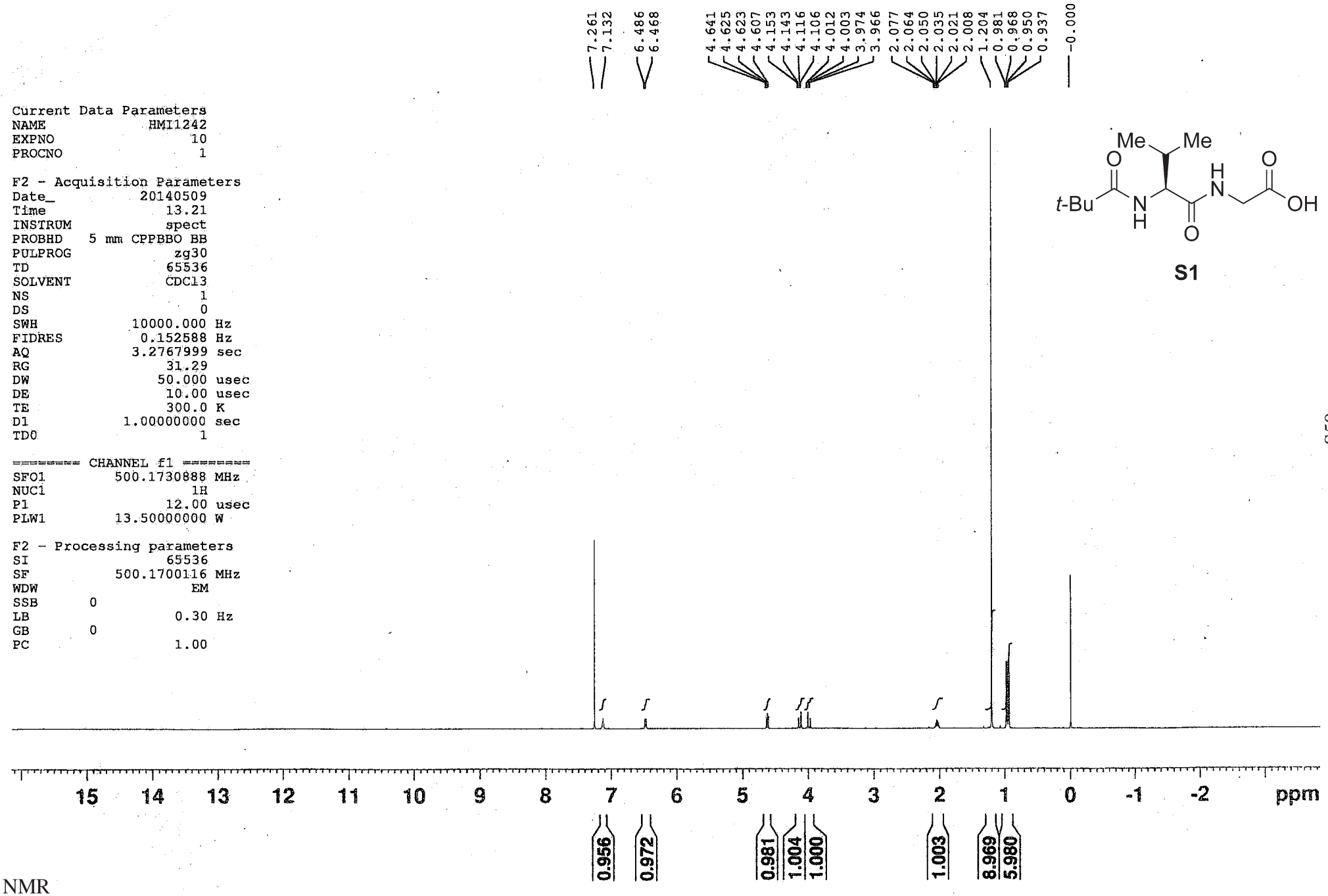
NAME 140422-5y079
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140423
Time 22.03
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 69
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
178.584
171.555
169.467
61.547
58.014
41.296
38.900
31.006
27.530
19.229
18.072
14.140
Me
Me
O
H
N
O
t-Bu
N
H
O
OEt
S2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR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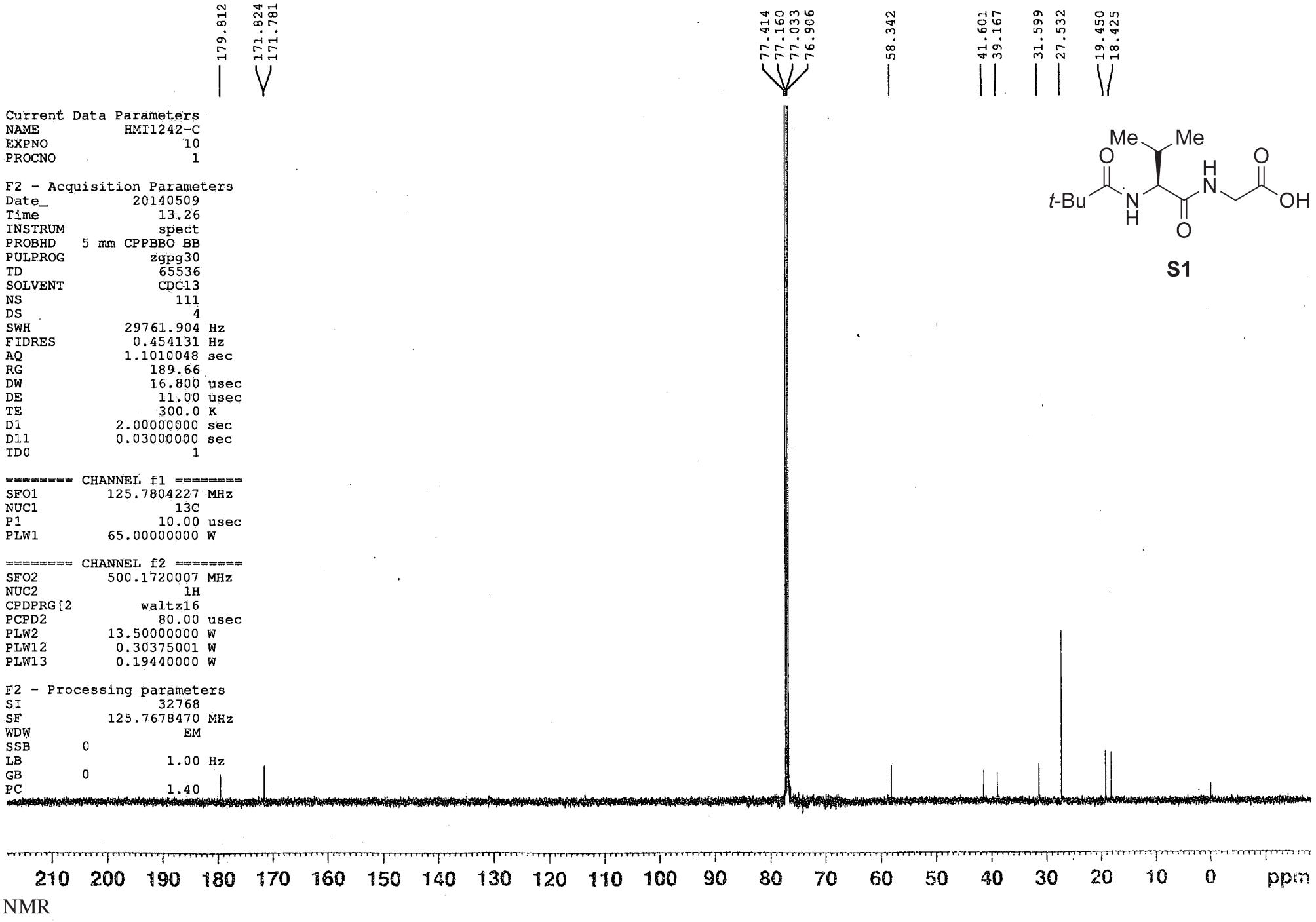
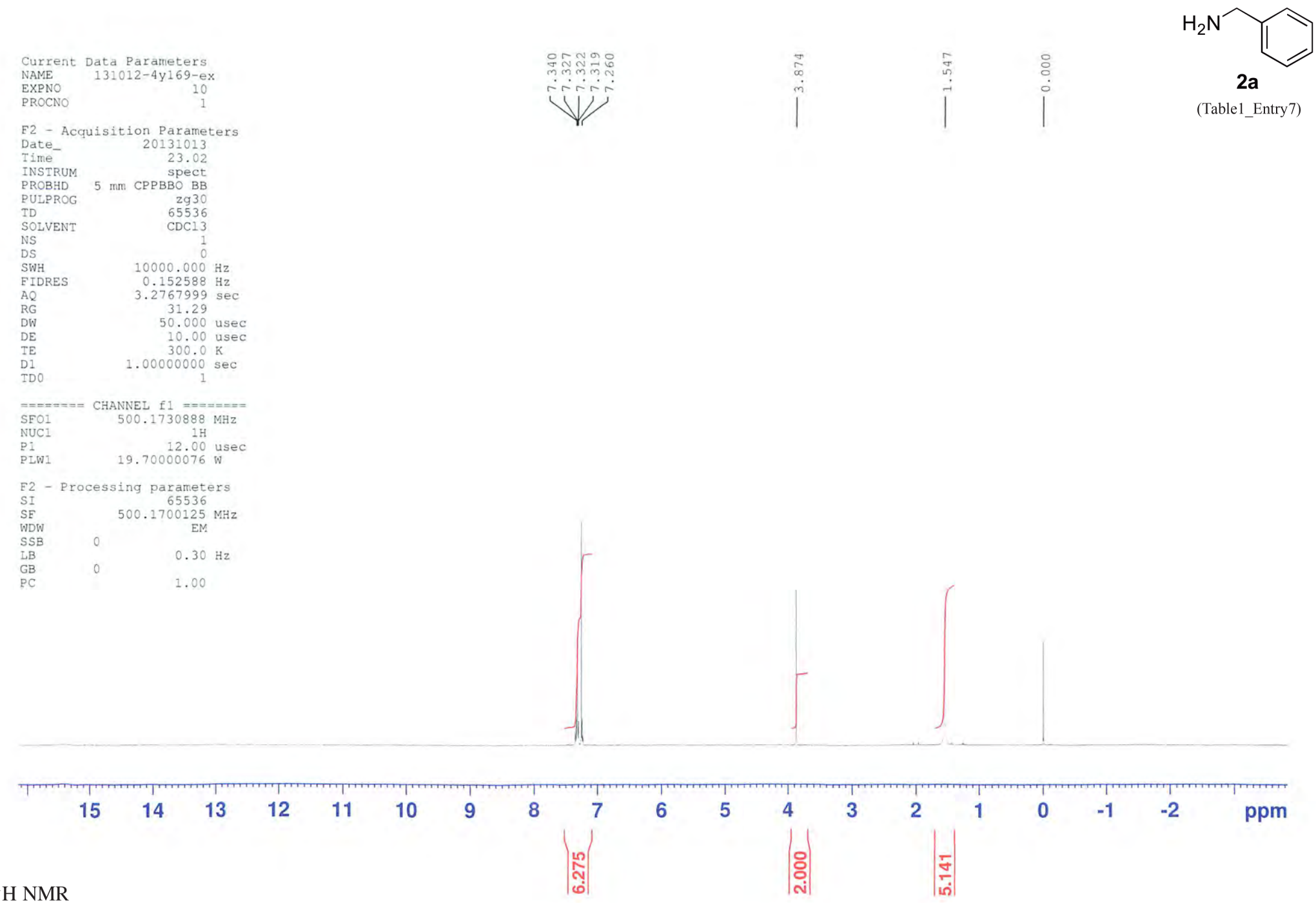
7.132
6.486
6.468
4.641
4.625
4.623
4.607
4.153
4.143
4.116
4.106
4.012
4.003
3.974
3.966
2.077
2.064
2.050
2.035
2.021
2.008
1.204
0.981
0.968
0.950
0.937
-0.000
Current Data Parameters
NAME HMI1242
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140509
Time 13.21
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 31.29
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1700116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
O
H
N
O
t-Bu
N
H
O
OH
O
S1
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
0.956
0.972
0.981
1.004
1.000
1.003
8.969
5.980

^{1}H NMR


179.812
171.824
171.781
77.414
77.160
77.033
76.906
58.342
41.601
39.167
31.599
27.532
19.450
18.425
Current Data Parameters
NAME HMI1242-C
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140509
Time 13.26
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 111
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
Me
O
H
N
O
t-Bu
N
H
N
OH
O
S1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

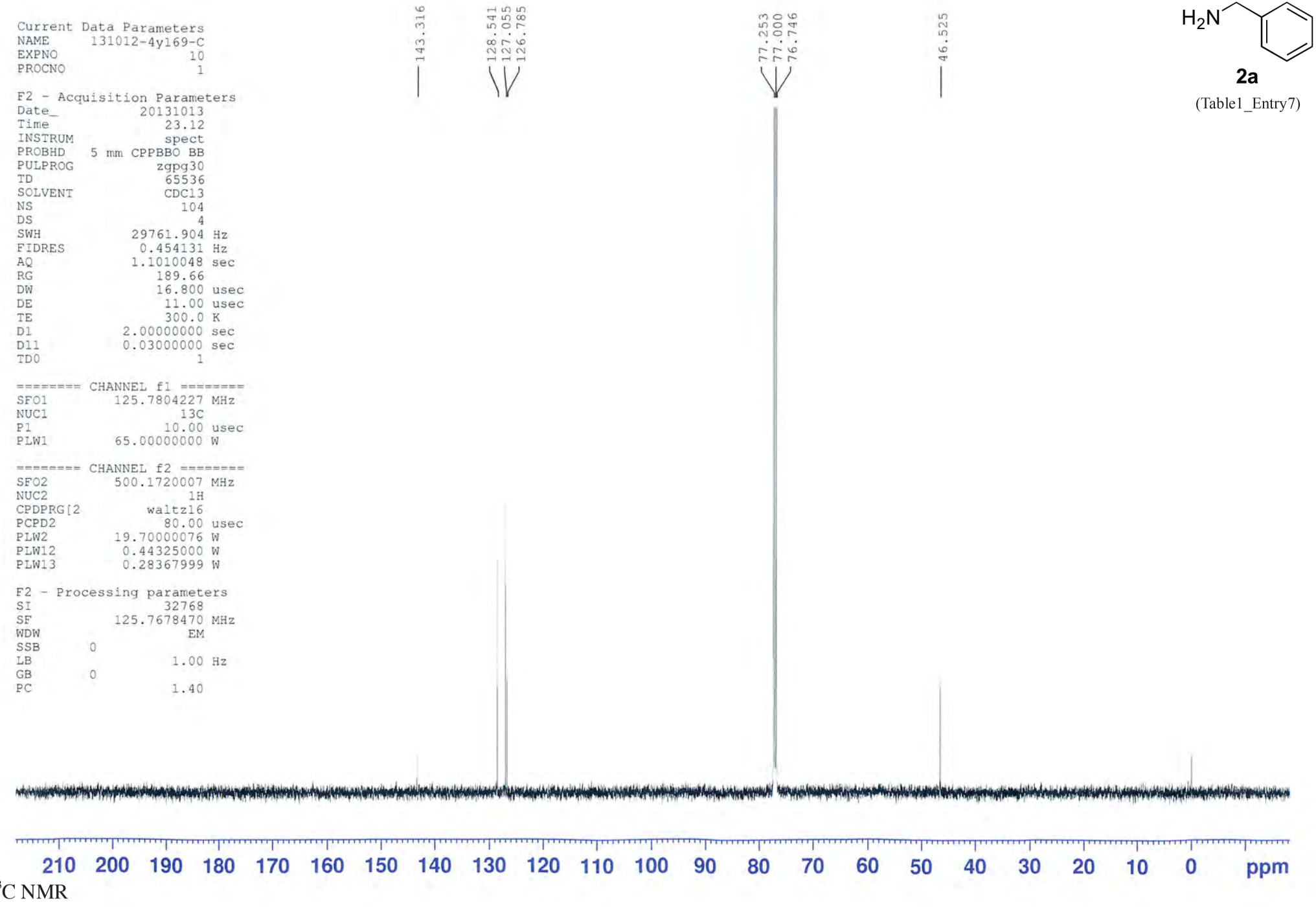


H_2N

2a

(Table1_Entry7)

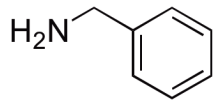
^{1}H NMR



2a

(Table1_Entry7)

^{13}C NMR



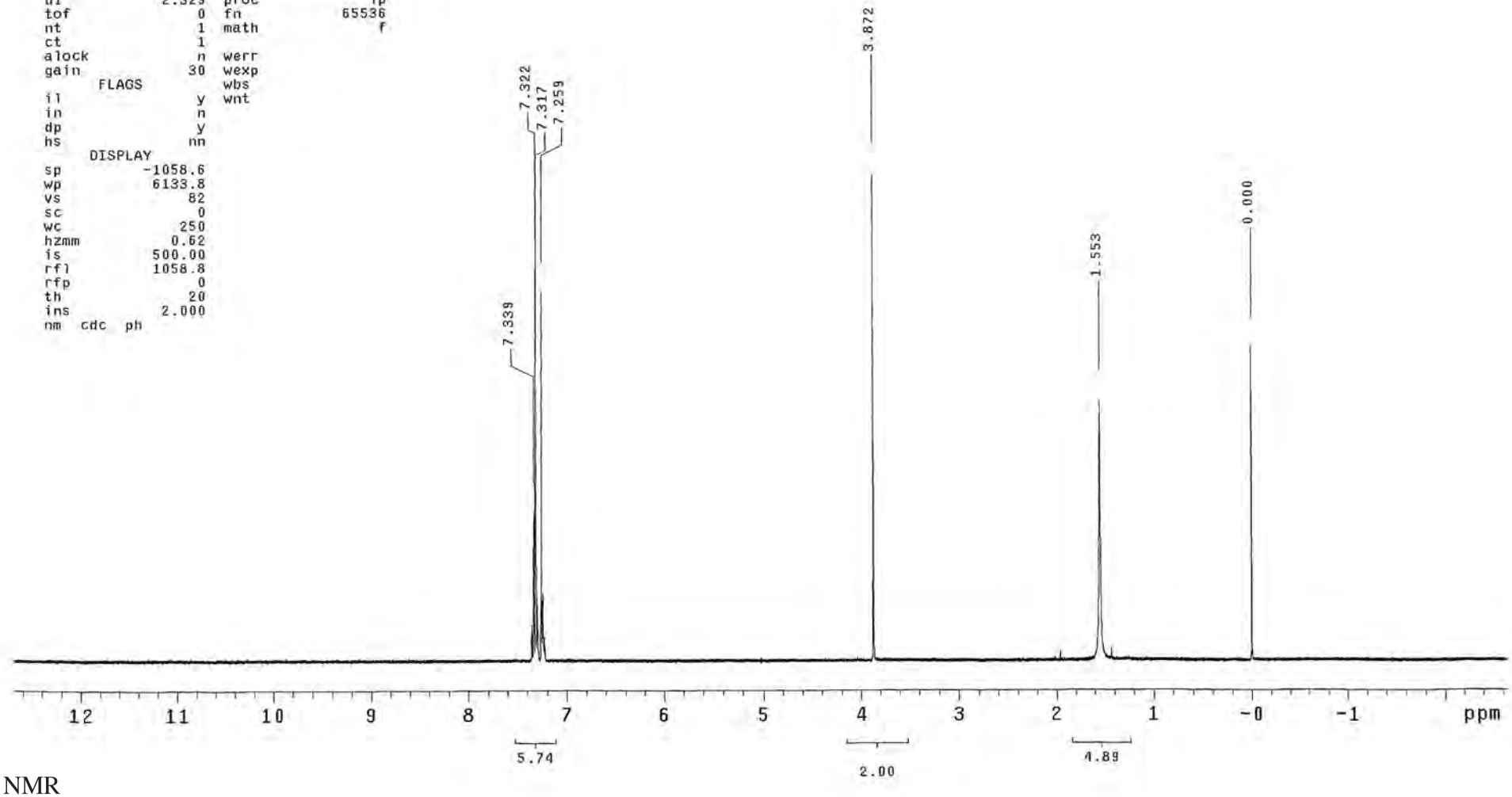
2a

(Table1_Entry8)

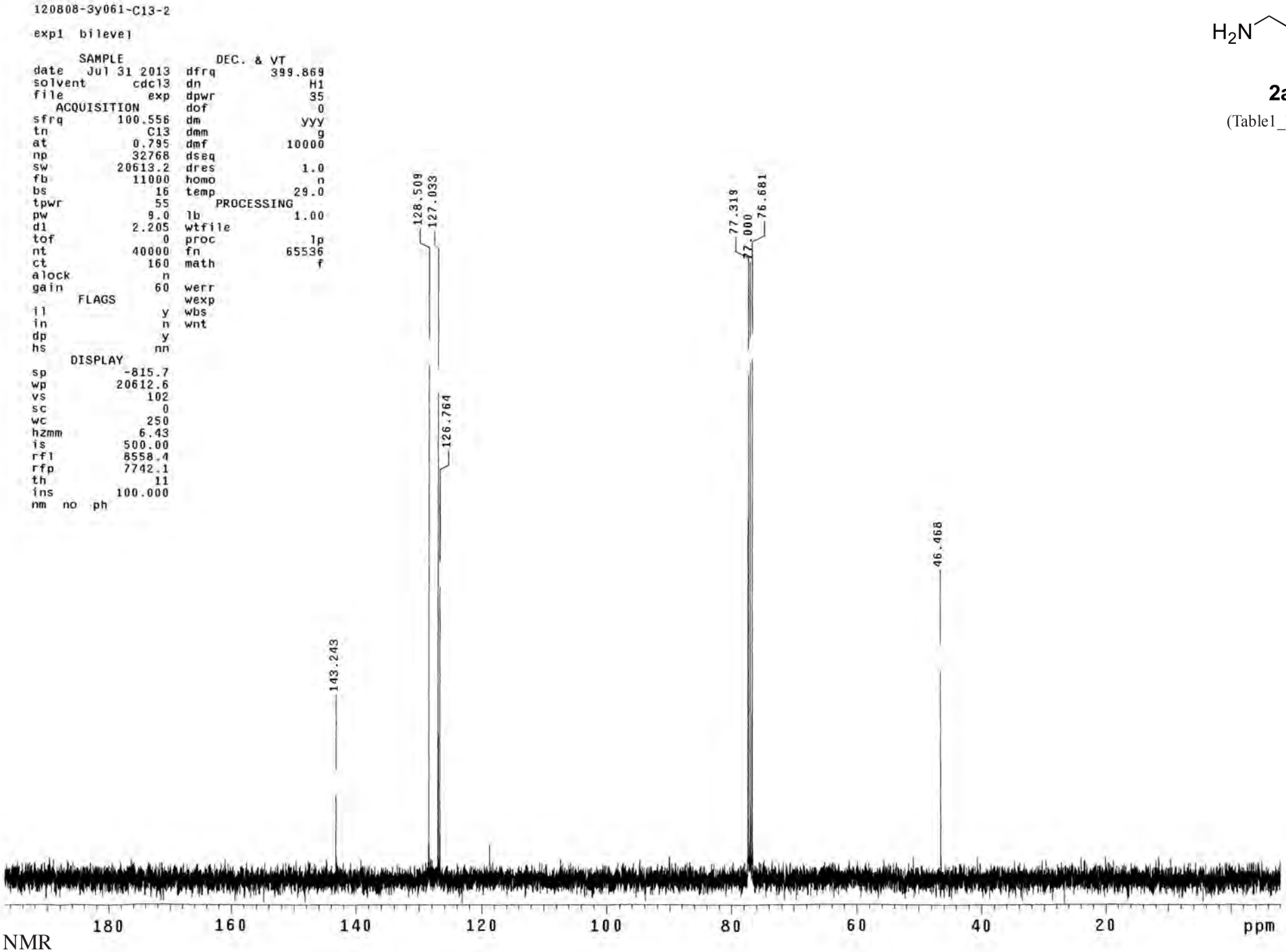
```
120808-3y061-ex

exp1  std1h

      SAMPLE              DEC. & VT
date    Aug 12 2012  dfrq        399.869
solvent      cdcl3   dn             H1
file           exp   dpwr           30
   ACQUISITION       dof             0
sfrq       399.869   dm            nnn
tn              H1   dmm             c
at           2.671   dmf           200
np           32768   dseq
sw          6134.0   dres          1.0
fb            3000   homo            n
bs               4   temp         30.0
tpwr            54       PROCESSING
pw             6.0   wtfile
d1           2.329   proc           lp
tof              0   fn          65536
nt               1   math            f
ct               1
alock            n   werr
gain            30   wexp
      FLAGS          wbs
il               y   wnt
in               n
dp               y
hs              nn
     DISPLAY
sp         -1058.6
wp          6133.8
vs              82
sc               0
wc             250
hzmm          0.62
is          500.00
rfl         1058.8
rfp              0
th              20
ins          2.000
nm  cdc  ph
```



^{1}H NMR

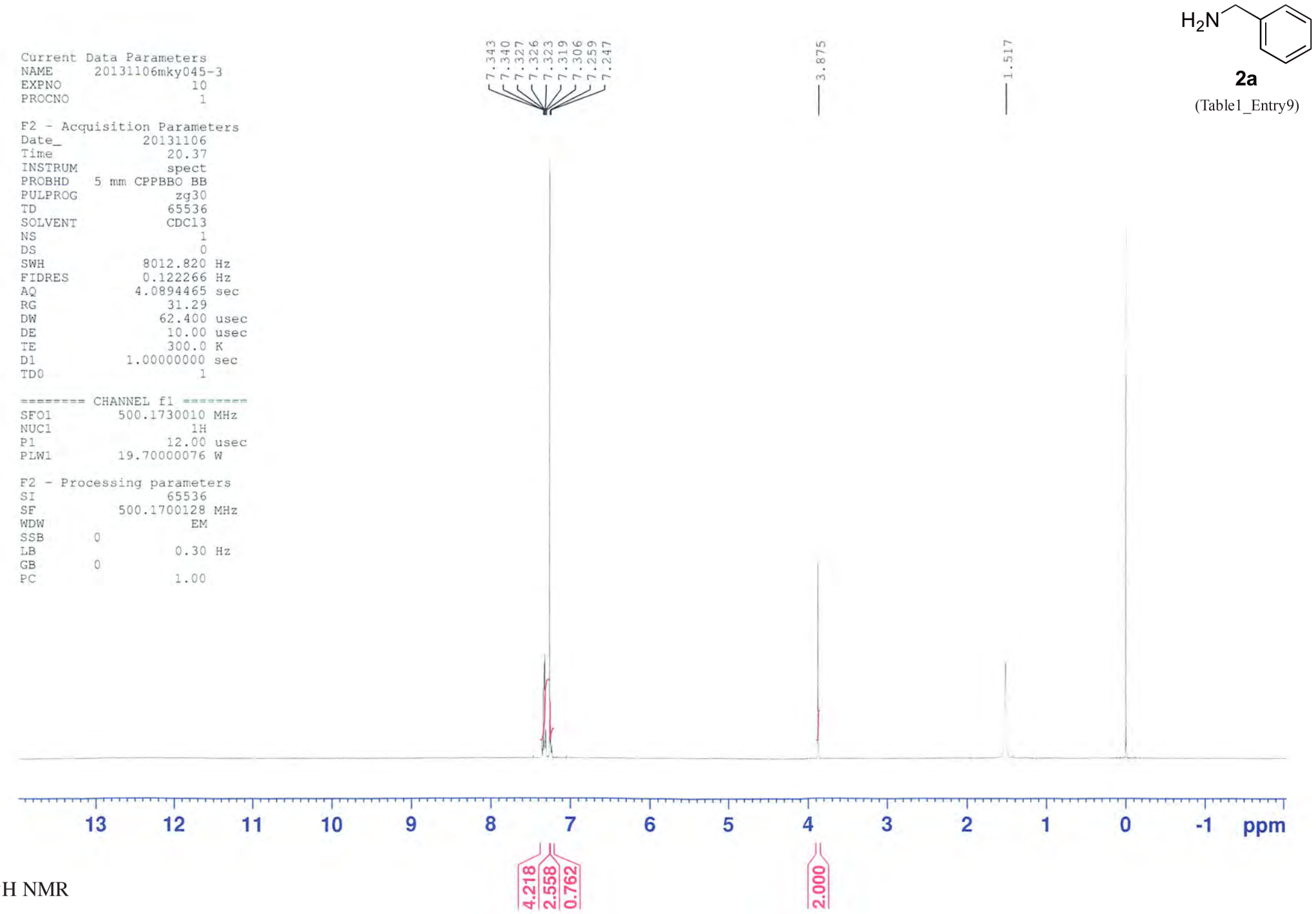


H_2N

2a

(Table1_Entry8)

^{13}C NMR

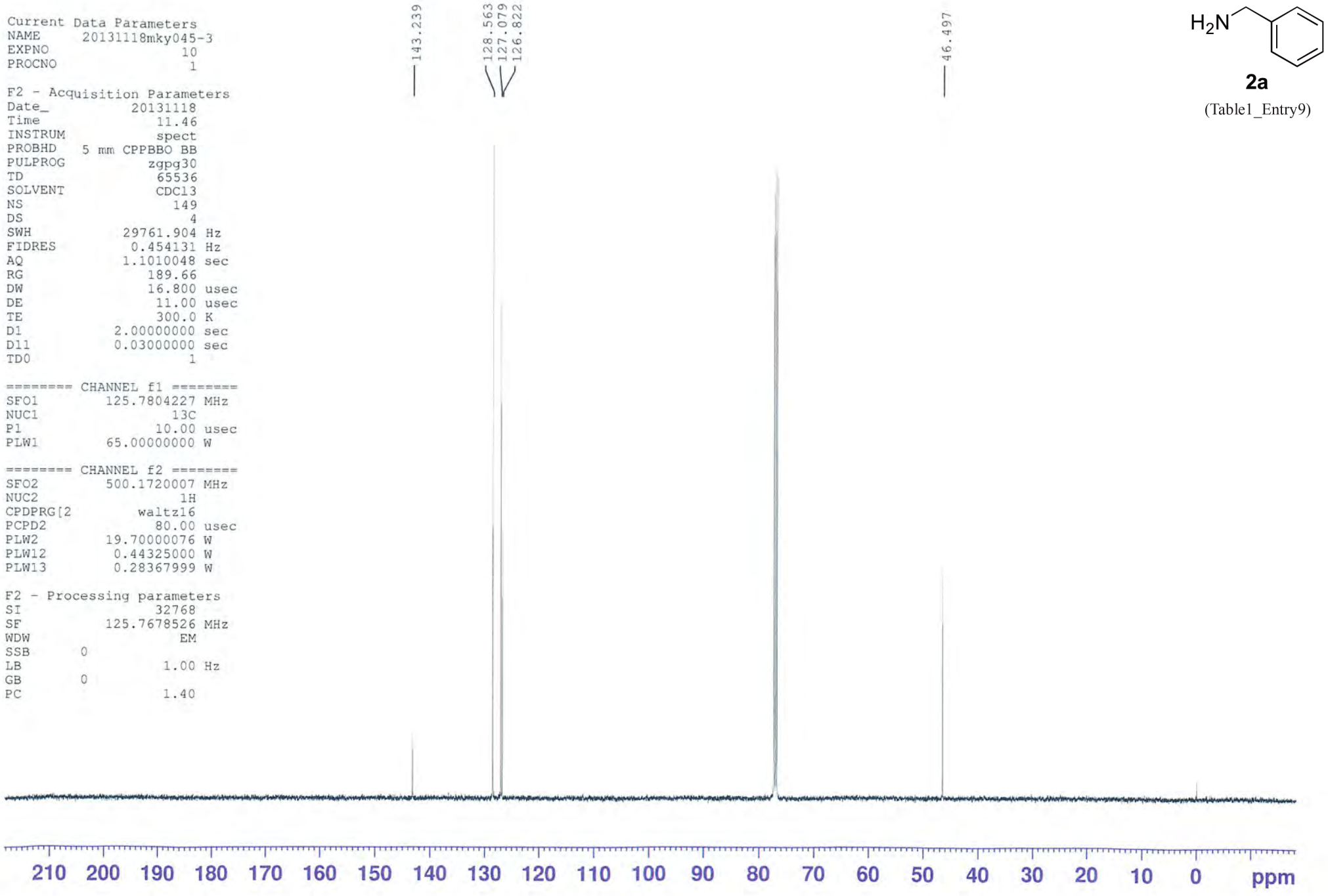


H_2N

2a

(Table1_Entry9)

^{1}H NMR

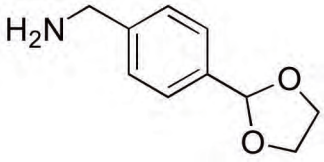


H_2N

2a

(Table1_Entry9)

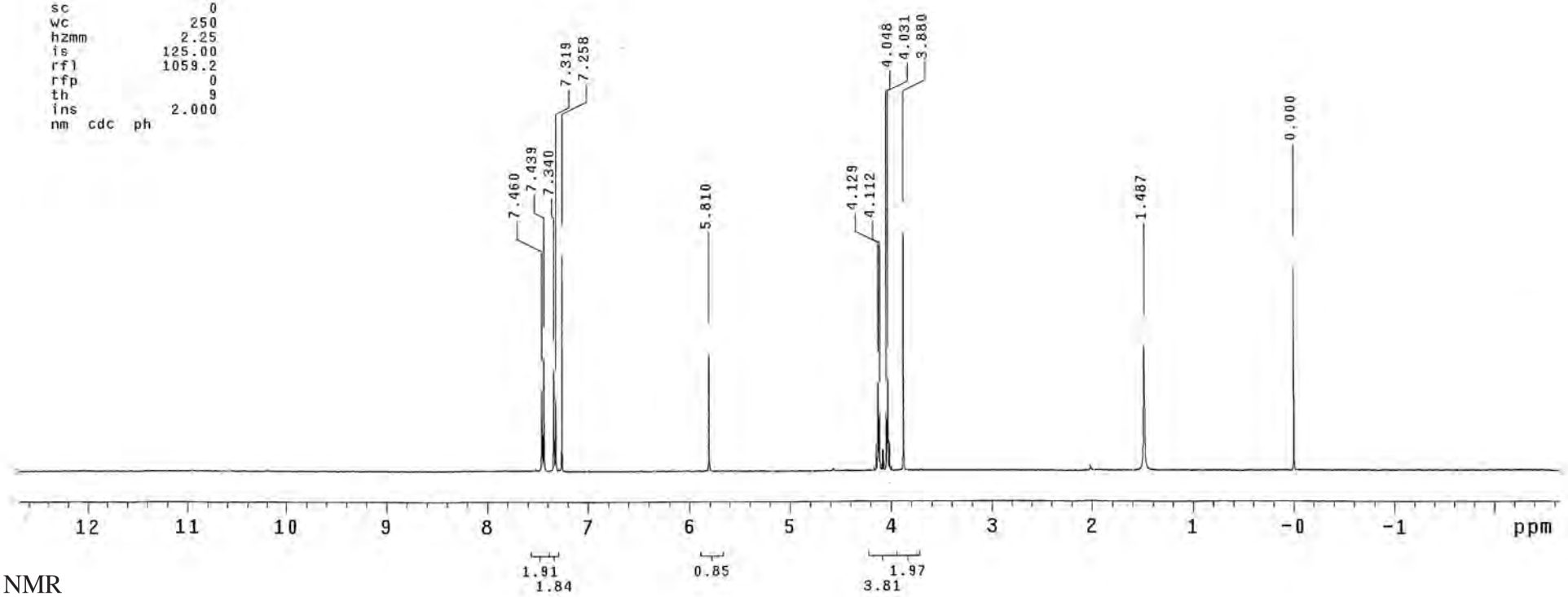
^{13}C NMR



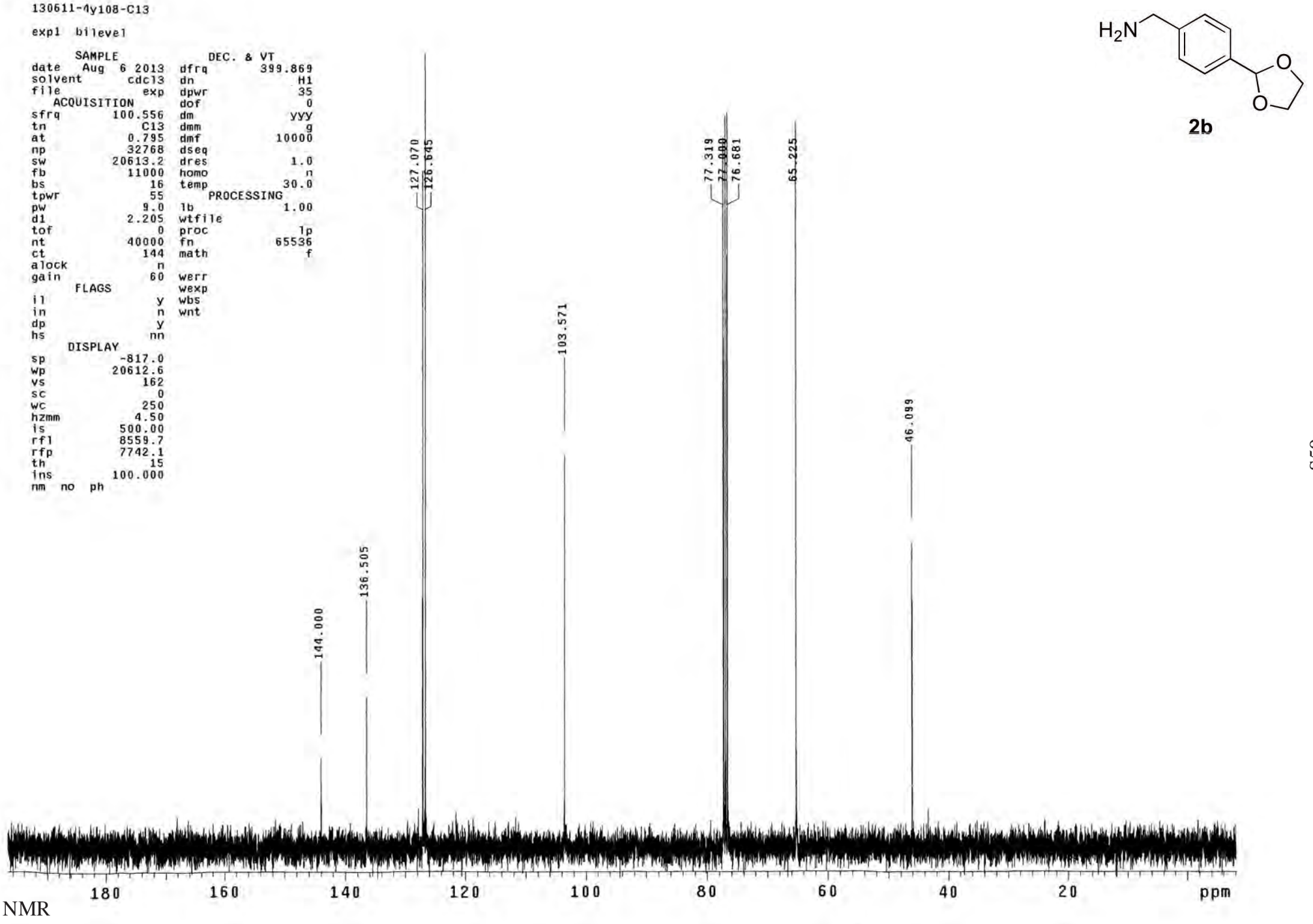
2b

```
exp2  std1h

      SAMPLE                DEC. & VT
date    Dec  6 2012   dfrq          399.869
solvent       cdcl3   dn                H1
file /data/molrec/~   dpwr              30
Dec1/121202-3y182-~   dof                0
        culm-3.fid    dm               nnn
   ACQUISITION        dmm                c
sfrq         399.869   dmf              200
tn                H1   dseq
at             2.671   dres             1.0
np             32768   homo               n
sw            6134.0   temp            30.0
fb              3000        PROCESSING
bs                 4   wtfile
tpwr              55   proc              lp
pw               9.0   fn             65536
d1             2.329   math               f
tof                0
nt                64   werr
ct                 0   wexp
alock              n   wbs
gain              30   wnt
      FLAGS
il                 y
in                 n
dp                 y
hs                nn
     DISPLAY
sp           -1059.0
wp            6133.8
vs                39
sc                 0
wc               250
hzmm            2.25
is            125.00
rfl           1059.2
rfp                0
th                 9
ins            2.000
nm  cdc  ph
```



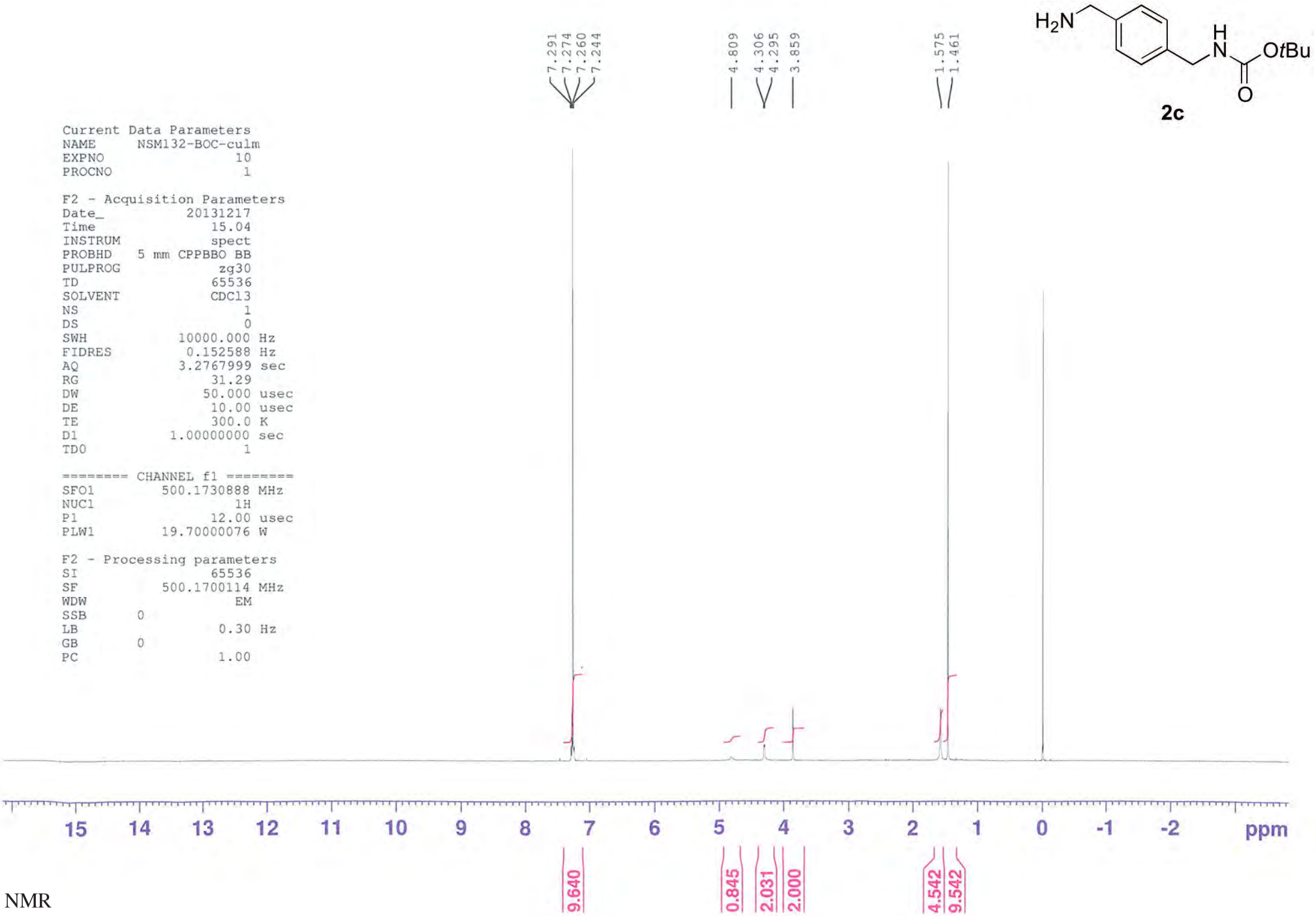
^{1}H NMR


130611-4y108-C13
exp1 bilevel
SAMPLE
date Aug 6 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 144
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -817.0
wp 20612.6
vs 162
sc 0
wc 250
hzmm 4.50
is 500.00
rfl 8559.7
rfp 7742.1
th 15
ins 100.000
nm no ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
dm yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
144.000
136.505
127.070
126.645
103.571
77.319
77.000
76.681
65.225
46.099
180
160
140
120
100
80
60
40
20
ppm

H2N
O
O

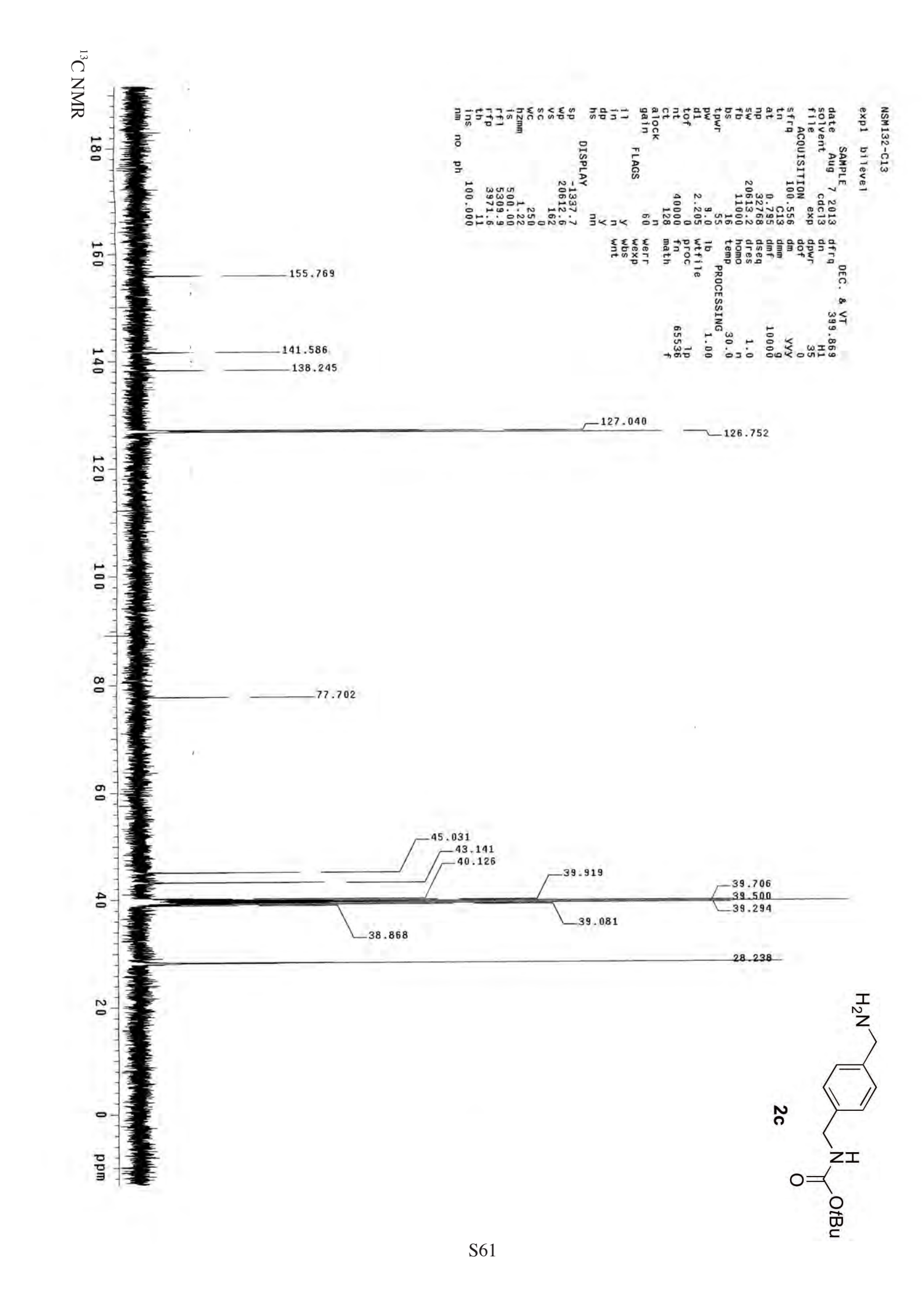
2b

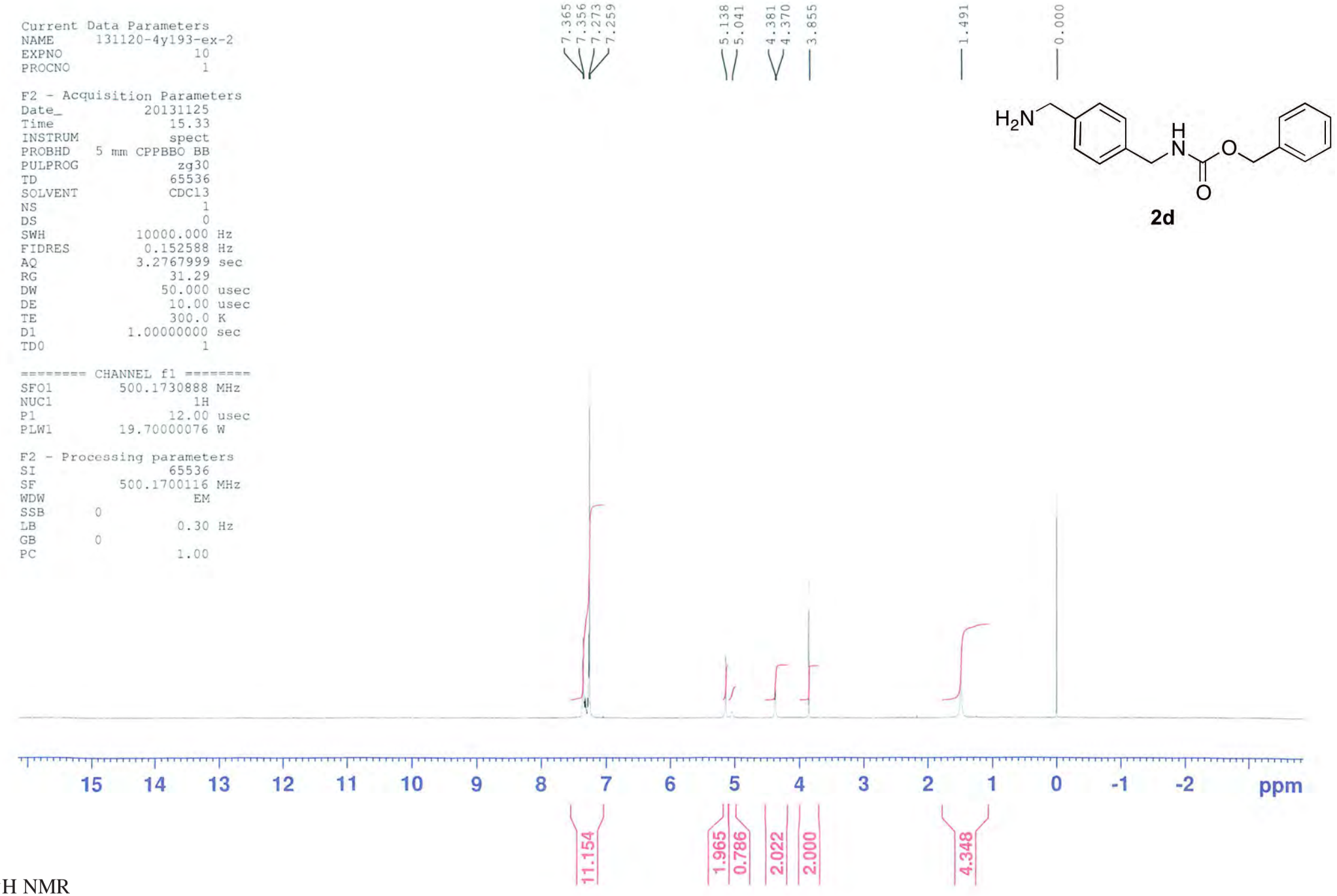
^{13}C NMR


7.291
7.274
7.260
7.244
4.809
4.306
4.295
3.859
1.575
1.461
H2N
H
N
OtBu
O
2c
Current Data Parameters
NAME NSM132-BOC-culm
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131217
Time 15.04
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 31.29
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
9.640
0.845
2.031
2.000
4.542
9.542

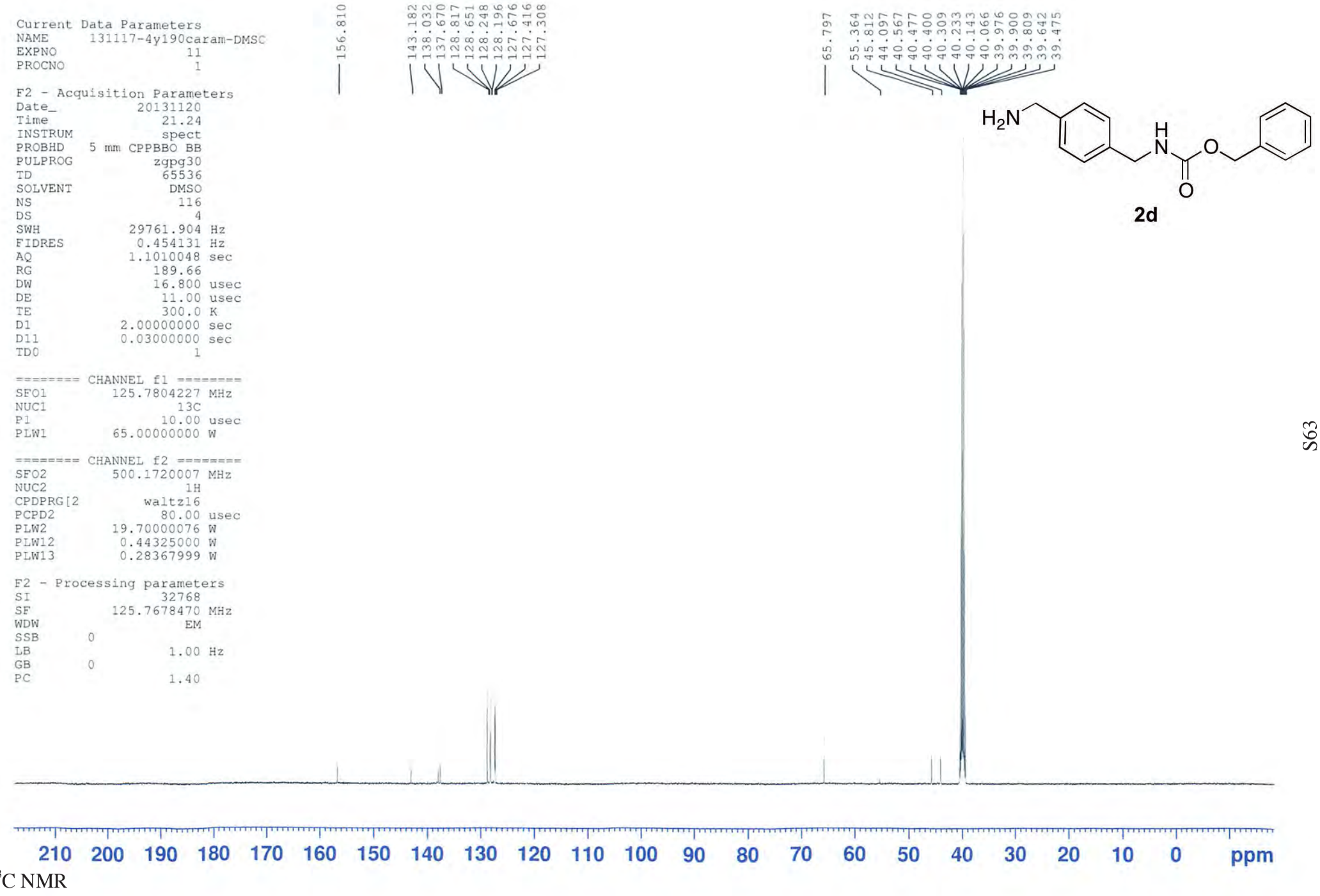
^{1}H NMR

^{13}C NMR

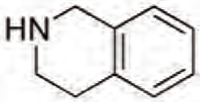

NSM132-C13
exp1 bilevel
SAMPLE
date Aug 7 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 128
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1337.7
wp 20612.6
vs 162
sc 0
wc 250
hzmm 1.22
is 500.00
rfl 5309.9
rfp 3971.6
th 11
ins 100.000
nm no ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
155.769
141.586
138.245
127.040
126.752
77.702
45.031
43.141
40.126
39.919
39.706
39.500
39.294
39.081
38.868
28.238
180
160
140
120
100
80
60
40
20
0
ppm
H2N
N
H
O
OtBu
2c


Current Data Parameters
NAME 131120-4y193-ex-2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131125
Time 15.33
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 31.29
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.365
7.356
7.273
7.259
5.138
5.041
4.381
4.370
3.855
1.491
0.000
H2N
H
N
O
O
2d
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
11.154
1.965
0.786
2.022
2.000
4.348

^{1}H NMR


Current Data Parameters
NAME 131117-4y190caram-DMSO
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131120
Time 21.24
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 116
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7678470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
156.810
143.182
138.032
137.670
128.817
128.651
128.248
128.196
127.676
127.416
127.308
65.797
55.364
45.812
44.097
40.567
40.477
40.400
40.309
40.233
40.143
40.066
39.976
39.900
39.809
39.642
39.475
H2N
H
N
O
O
2d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

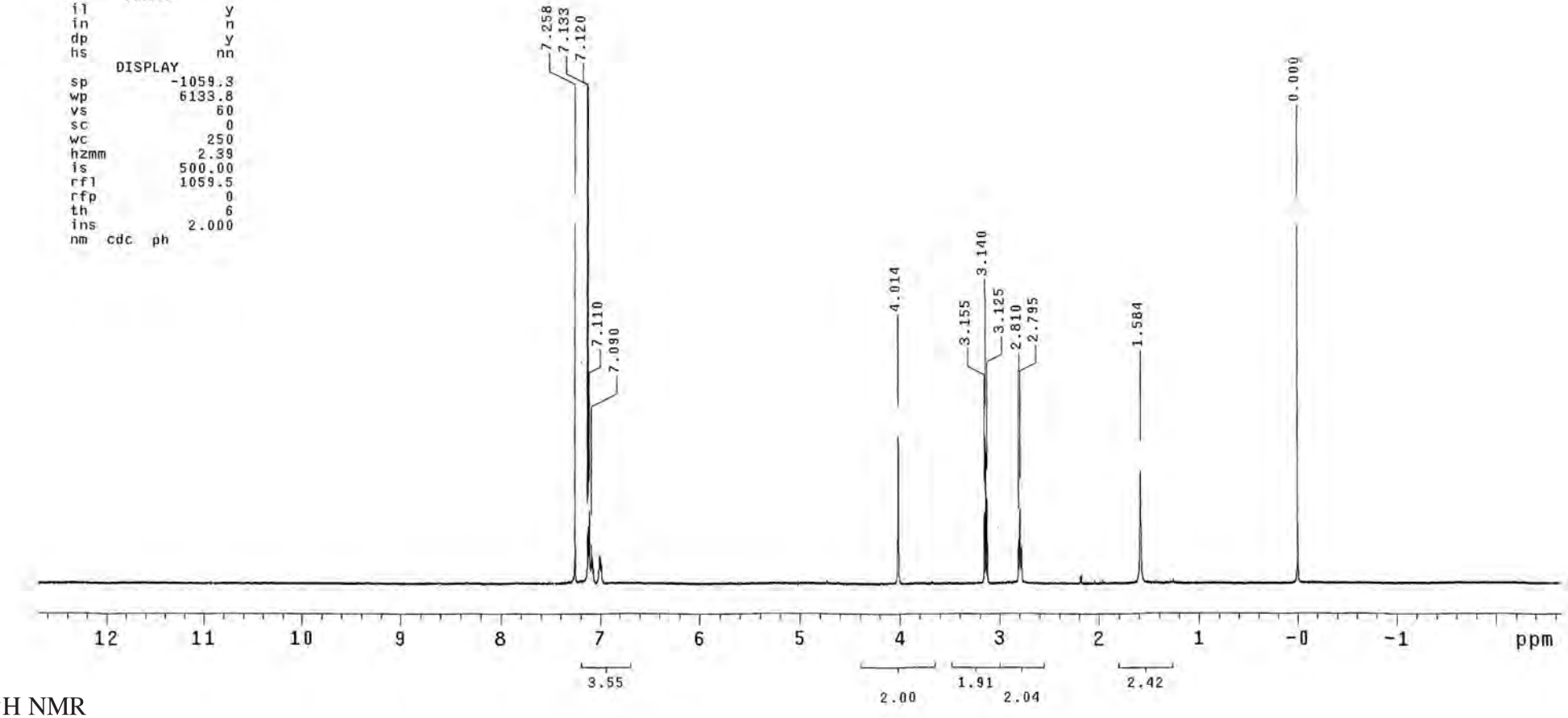


2e

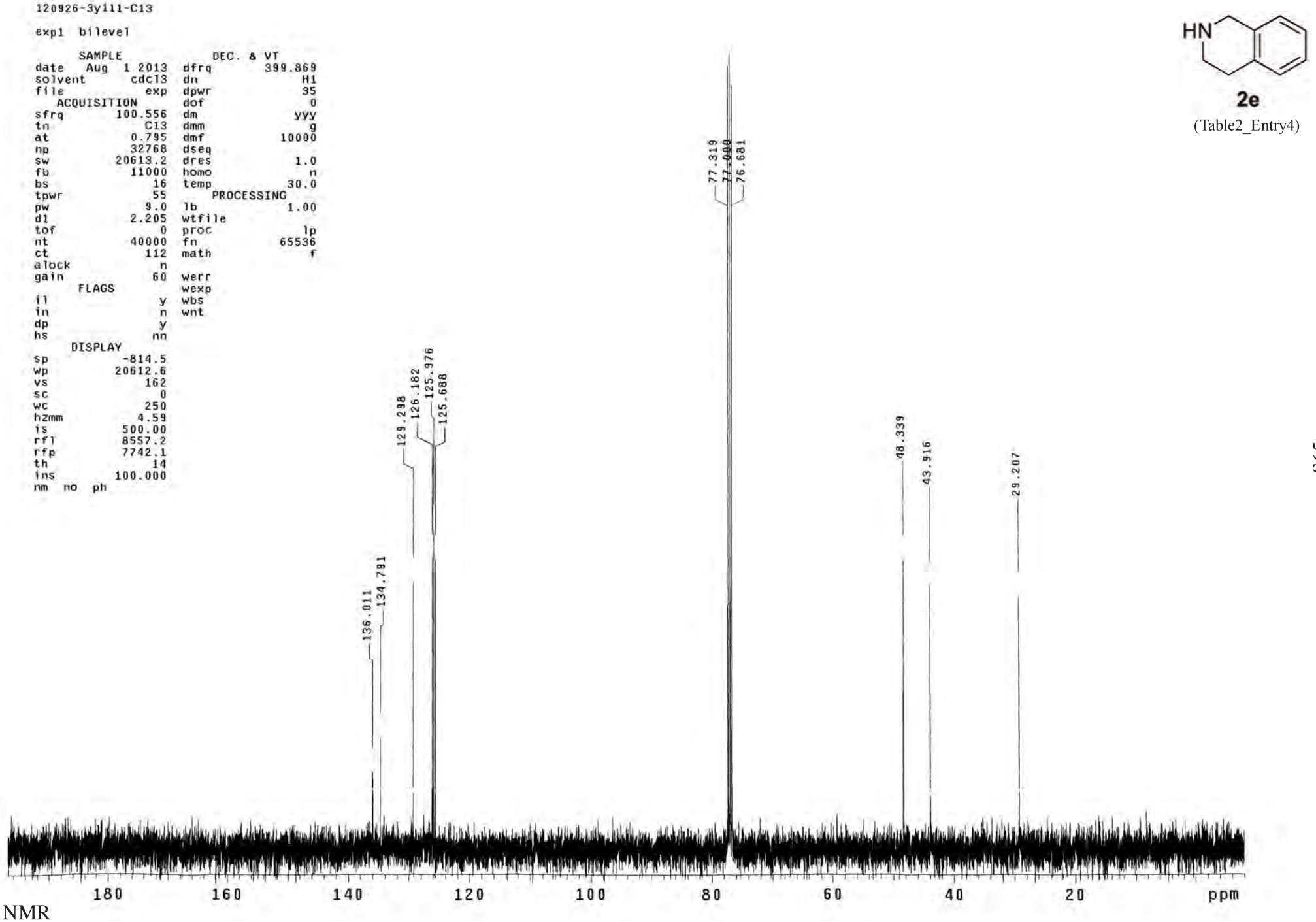
(Table2_Entry4)

```
exp2  std1h

      SAMPLE                DEC. & VT
date    Sep 26 2012   dfrq        399.869
solvent       cdcl3   dn               H1
file /data/molrec/~   dpwr             30
Sep2/120926-3y111-~   dof               0
             ex.fid   dm              nnn
   ACQUISITION        dmm               c
sfrq        399.869   dmf             200
tn               H1   dseq
at            2.671   dres            1.0
np            32768   homo              n
sw           6134.0   temp           30.0
fb             3000       PROCESSING
bs                4   wtfile
tpwr             54   proc             lp
pw              6.0   fn            65536
d1            2.329   math              f
tof               0
nt               64   werr
ct                4   wexp
alock             n   wbs
gain             30   wnt
      FLAGS
il                y
in                n
dp                y
hs               nn
      DISPLAY
sp          -1059.3
wp           6133.8
vs               60
sc                0
wc              250
hzmm           2.39
is           500.00
rfl          1059.5
rfp               0
th                6
ins           2.000
nm  cdc  ph
```



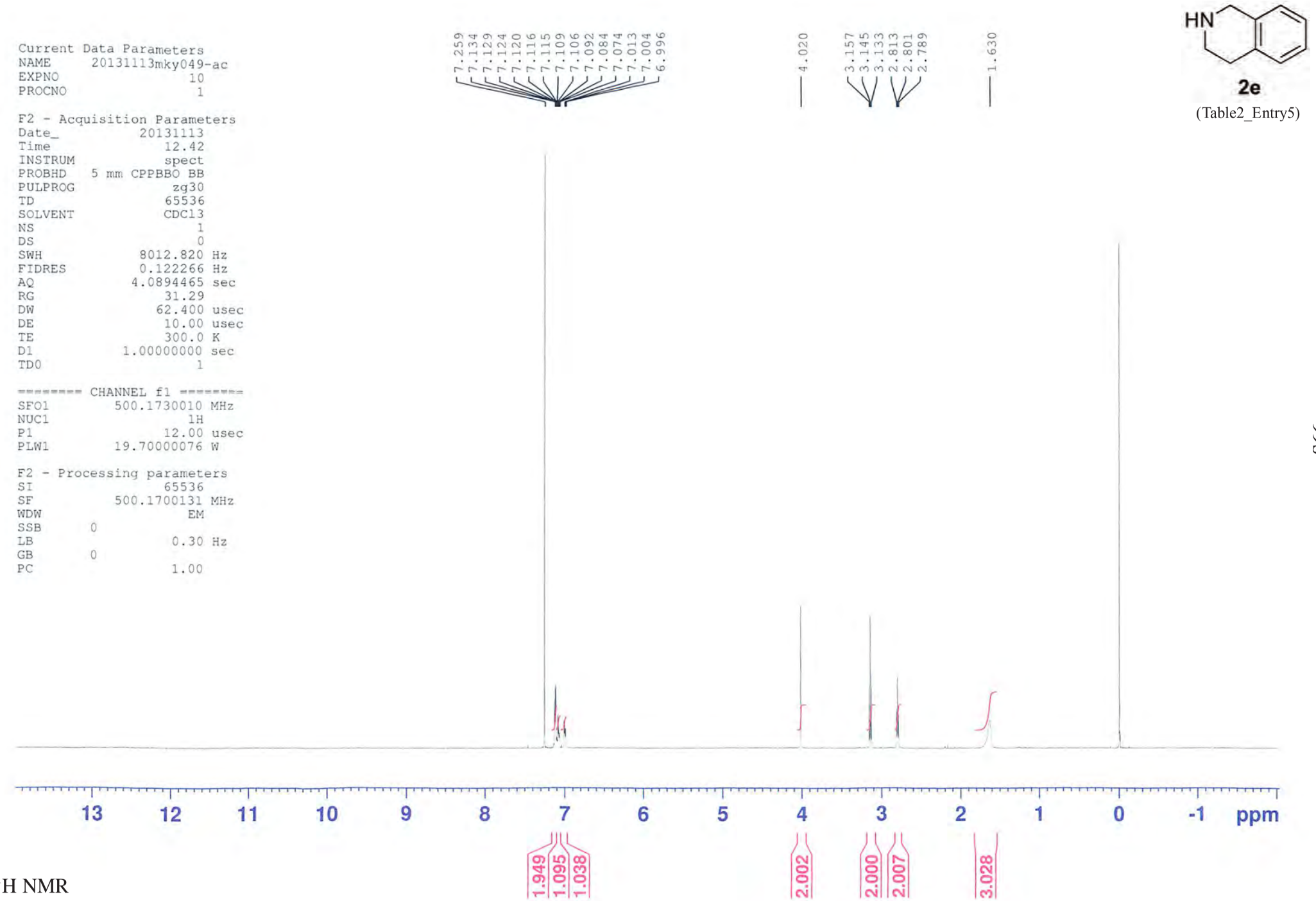
^{1}H NMR



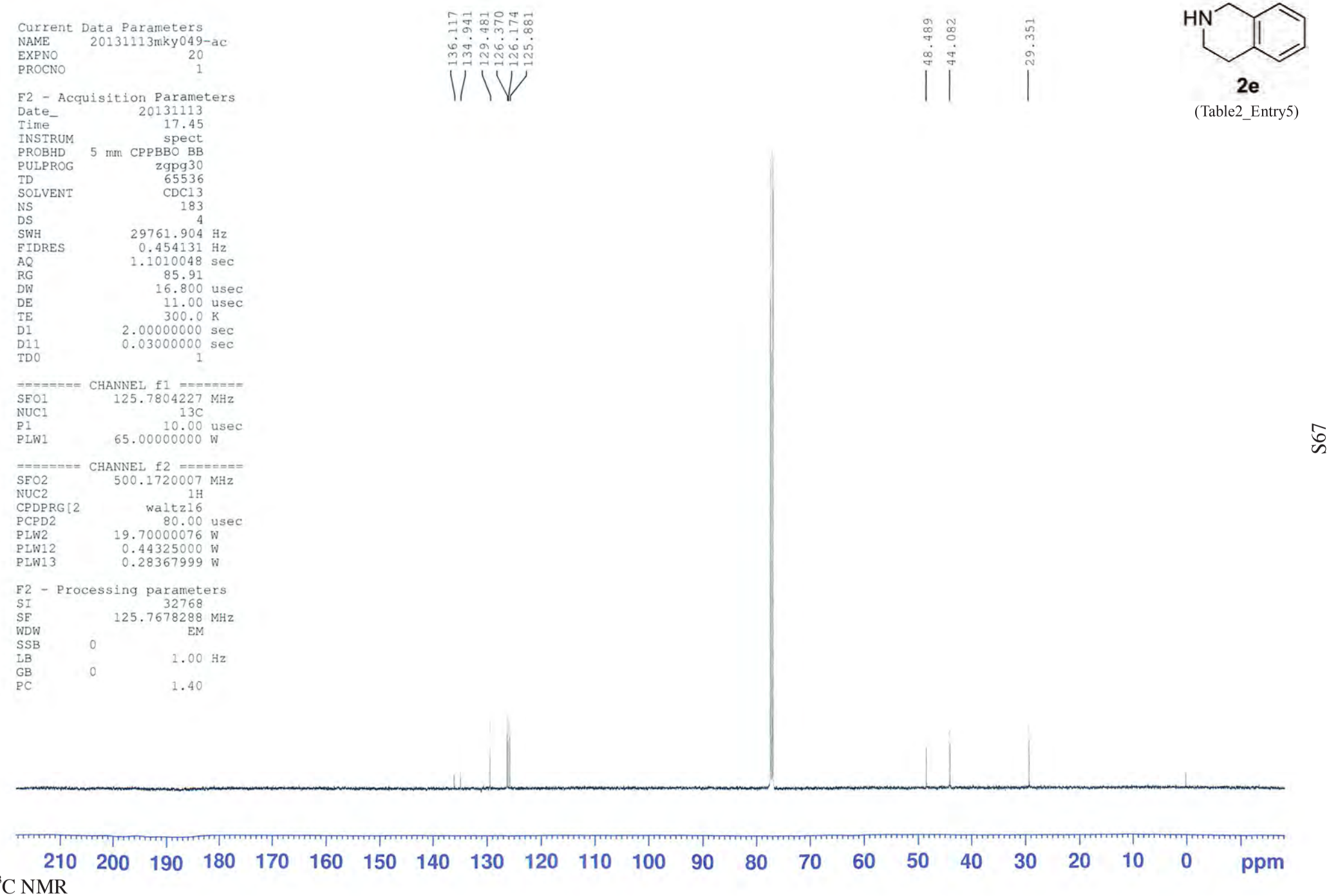
2e

(Table2_Entry4)

^{13}C NMR

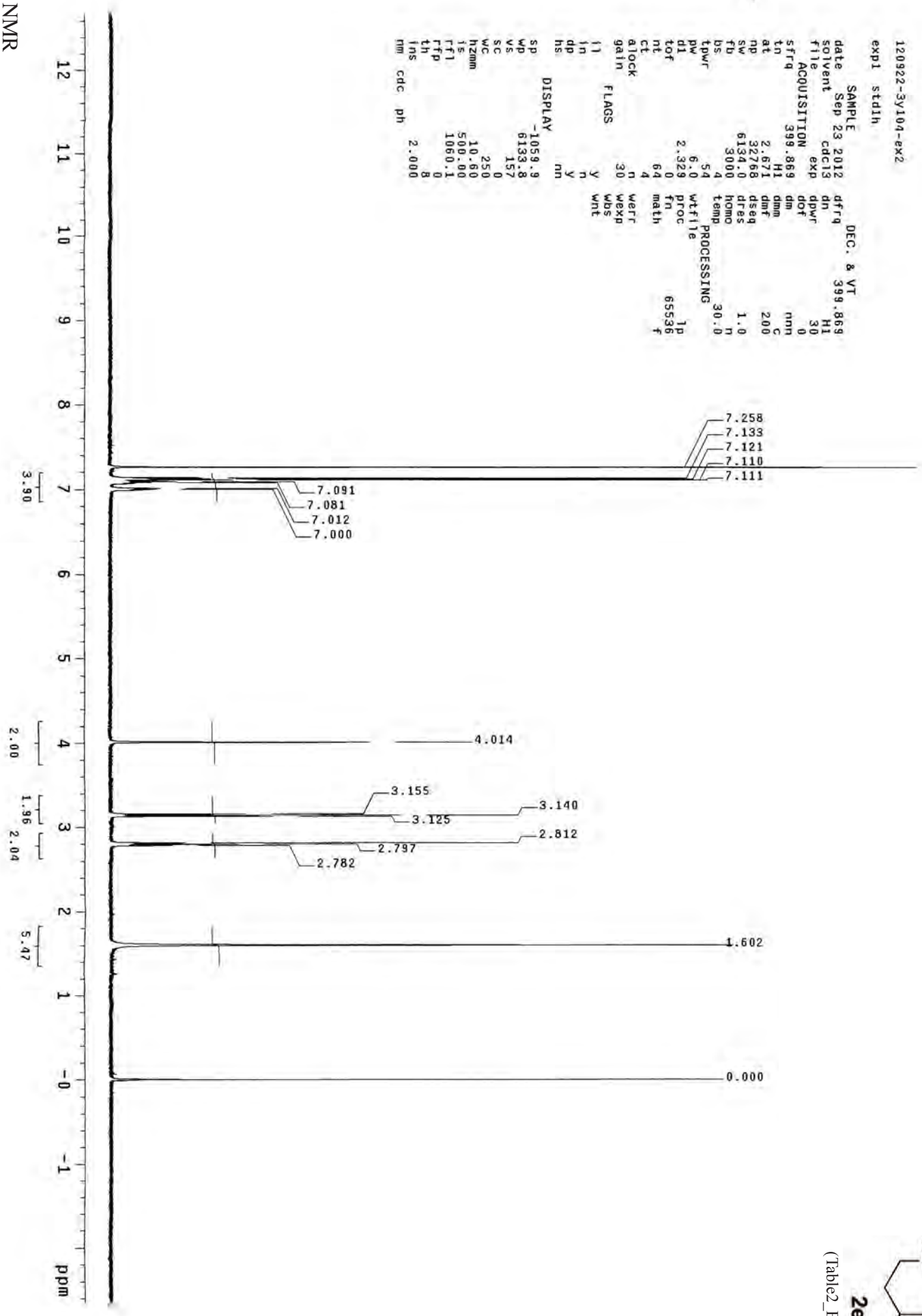

Current Data Parameters
NAME 20131113mky049-ac
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131113
Time 12.42
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.259
7.134
7.129
7.124
7.120
7.116
7.115
7.109
7.106
7.092
7.084
7.074
7.013
7.004
6.996
4.020
3.157
3.145
3.133
2.813
2.801
2.789
1.630
HN
2e
(Table2_Entry5)
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.949
1.095
1.038
2.002
2.000
2.007
3.028

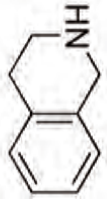
^{1}H NMR


Current Data Parameters
NAME 20131113mky049-ac
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131113
Time 17.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 183
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 85.91
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7678288 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
136.117
134.941
129.481
126.370
126.174
125.881
48.489
44.082
29.351
HN
2e
(Table2_Entry5)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

^{1}H NMR





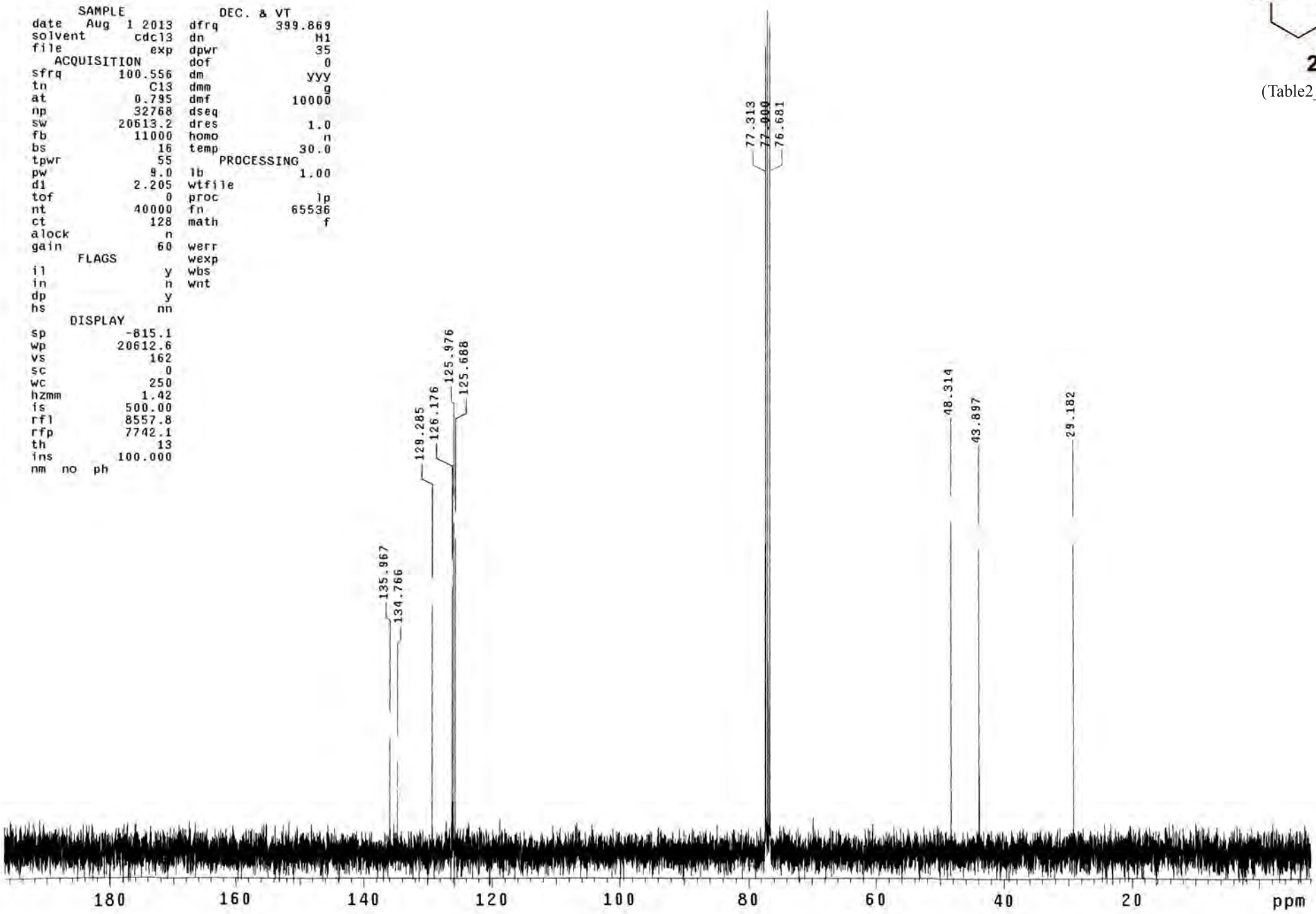
2e

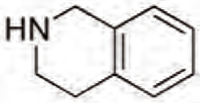
(Table2_Entry6)

120922-3y103-C13

exp1 bilevel

SAMPLE		DEC. & VT	
date	Aug 1 2013	dfrq	399.869
solvent	cdcl3	dn	H1
file	exp	dpwr	35
ACQUISITION		dof	0
sfrq	100.556	dm	yyy
tn	C13	dmm	g
at	0.795	dmf	10000
np	32768	dseq	
sw	20613.2	dres	1.0
fb	11000	homo	n
bs	16	temp	30.0
tpwr	55	PROCESSING	
pw	9.0	lb	1.00
d1	2.205	wtfile	
tof	0	proc	lp
nt	40000	fn	65536
ct	128	math	f
alock	n		
gain	60	werr	
FLAGS		wexp	
il	y	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-815.1		
wp	20612.6		
vs	162		
sc	0		
wc	250		
hzmm	1.42		
is	500.00		
rfl	8557.8		
rfp	7742.1		
th	13		
ins	100.000		
nm no ph			



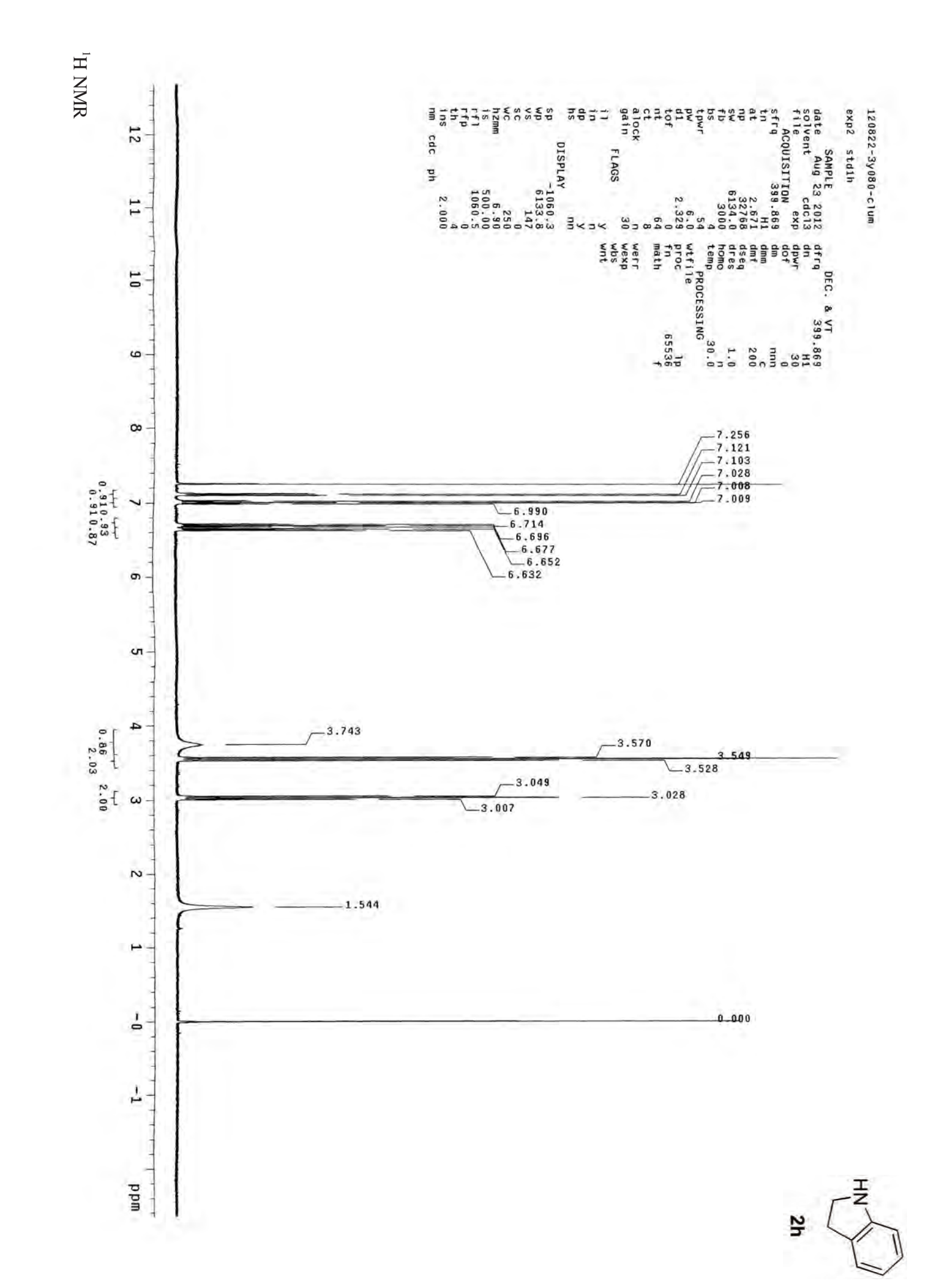


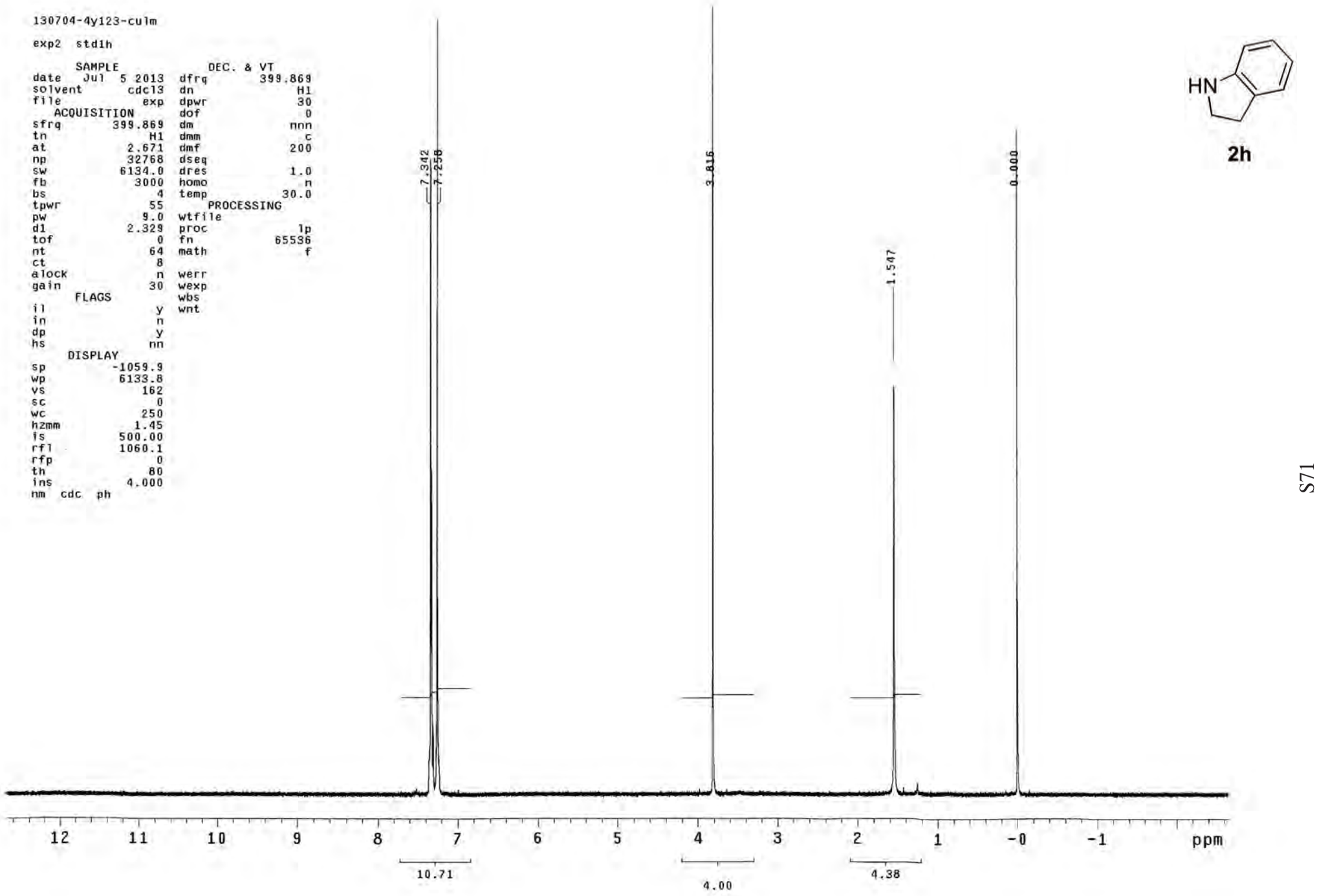
2e

(Table2_Entry6)

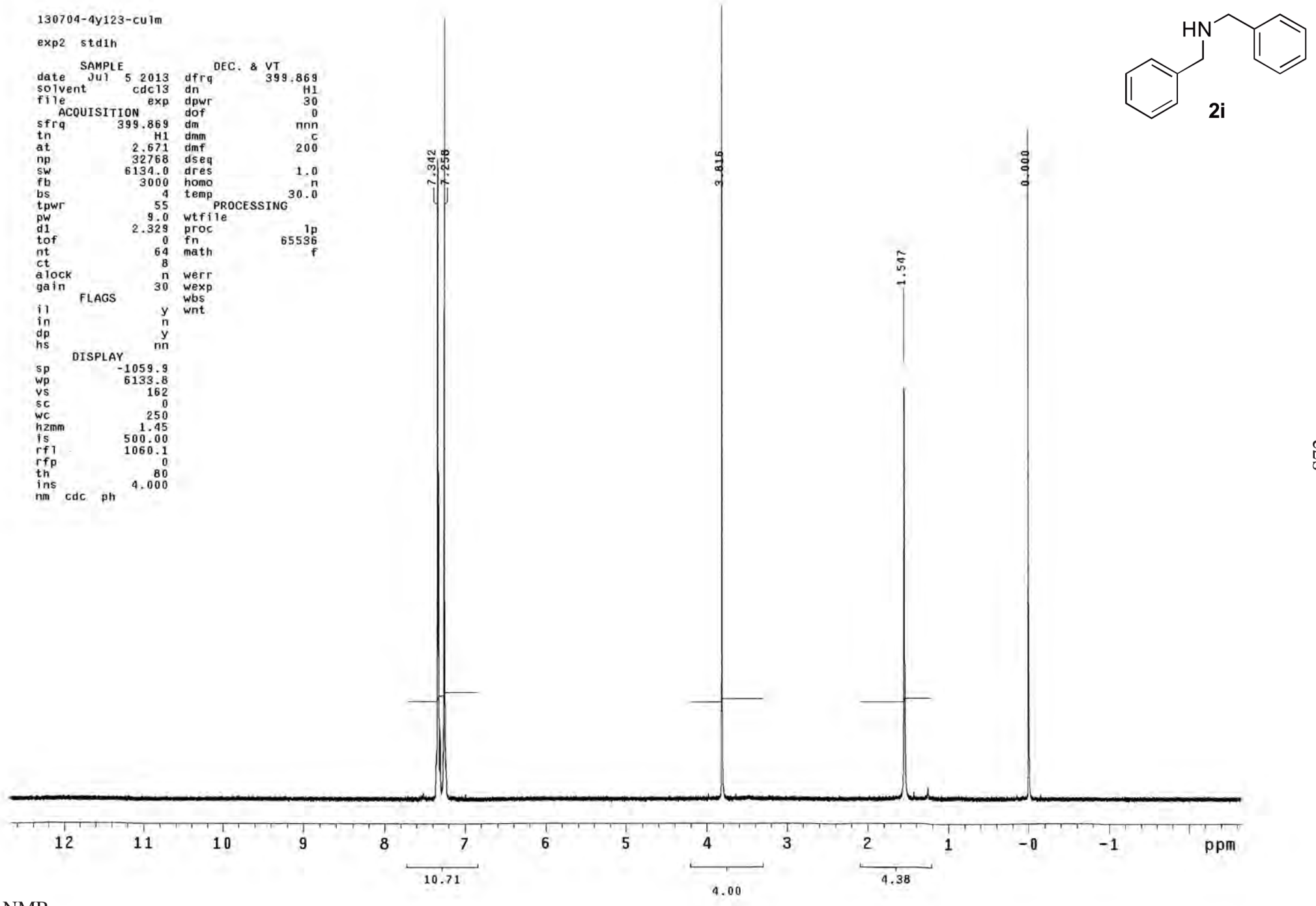
^{13}C NMR

^{1}H NMR

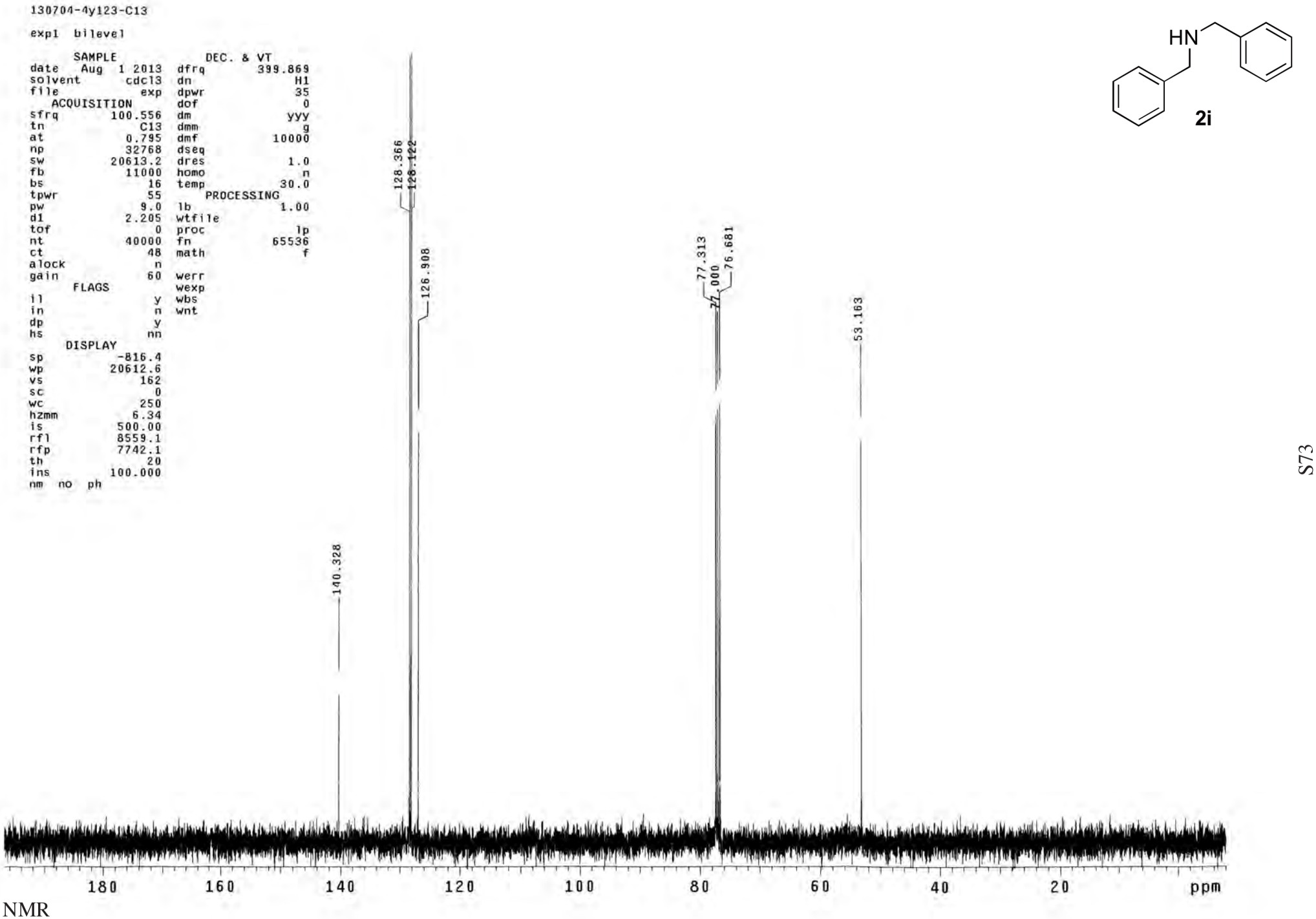

120822-3y080-clum
exp2 std1h
SAMPLE
date Aug 23 2012
solvent cdcl3
file exp
ACQUISITION
sfrq 399.869
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 54
pw 6.0
d1 2.329
tof 0
nt 64
ct 8
alock n
gain 30
FLAGS
ii y
in n
dp y
hs nn
DISPLAY
sp -1060.3
wp 6133.8
vs 147
sc 0
wc 250
hzmm 6.90
is 500.00
rfl 1060.5
rfp 0
th 4
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
wexp
wbs
wnt
7.256
7.121
7.103
7.028
7.008
7.009
6.990
6.714
6.696
6.677
6.652
6.632
3.743
3.570
3.549
3.528
3.049
3.028
3.007
1.544
0.000
0.91
0.93
0.91
0.87
0.86
2.03
2.00
ppm
HN
2h


130704-4y123-culm
exp2 stdlh
SAMPLE
date Jul 5 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 399.869
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 55
pw 9.0
d1 2.329
tof 0
nt 64
ct 8
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1059.9
wp 6133.8
vs 162
sc 0
wc 250
hzmm 1.45
fs 500.00
rfl 1060.1
rfp 0
th 80
ins 4.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
7.342
7.258
3.816
1.547
0.000
HN
2h
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
10.71
4.00
4.38

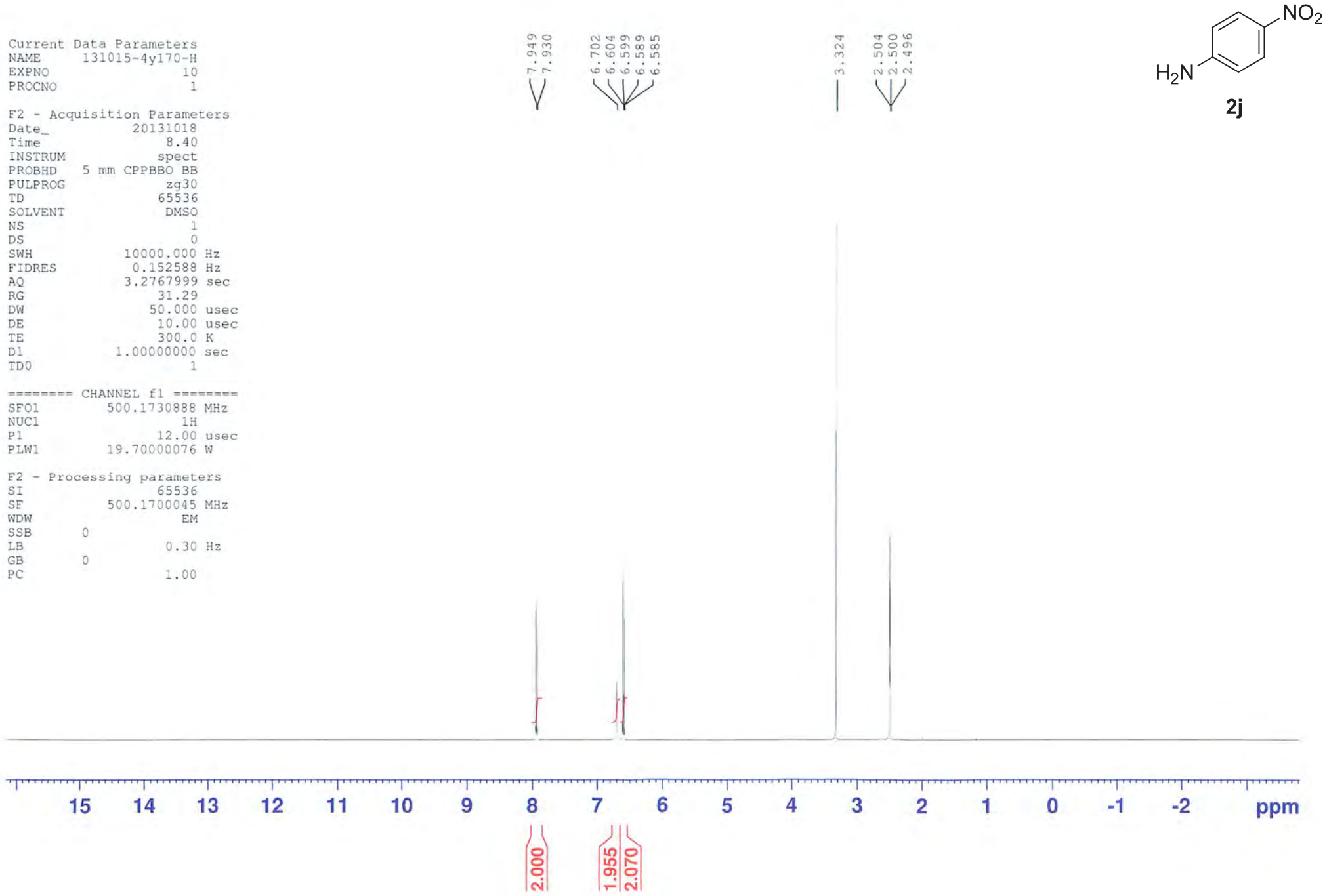
^{13}C NMR


130704-4y123-culm
exp2 stdlh
SAMPLE
date Jul 5 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 399.869
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 55
pw 9.0
d1 2.329
tof 0
nt 64
ct 8
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1059.9
wp 6133.8
vs 162
sc 0
wc 250
hzmm 1.45
fs 500.00
rfl 1060.1
rfp 0
th 80
ins 4.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
7.342
7.258
3.816
1.547
0.000
HN
2i
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
10.71
4.00
4.38

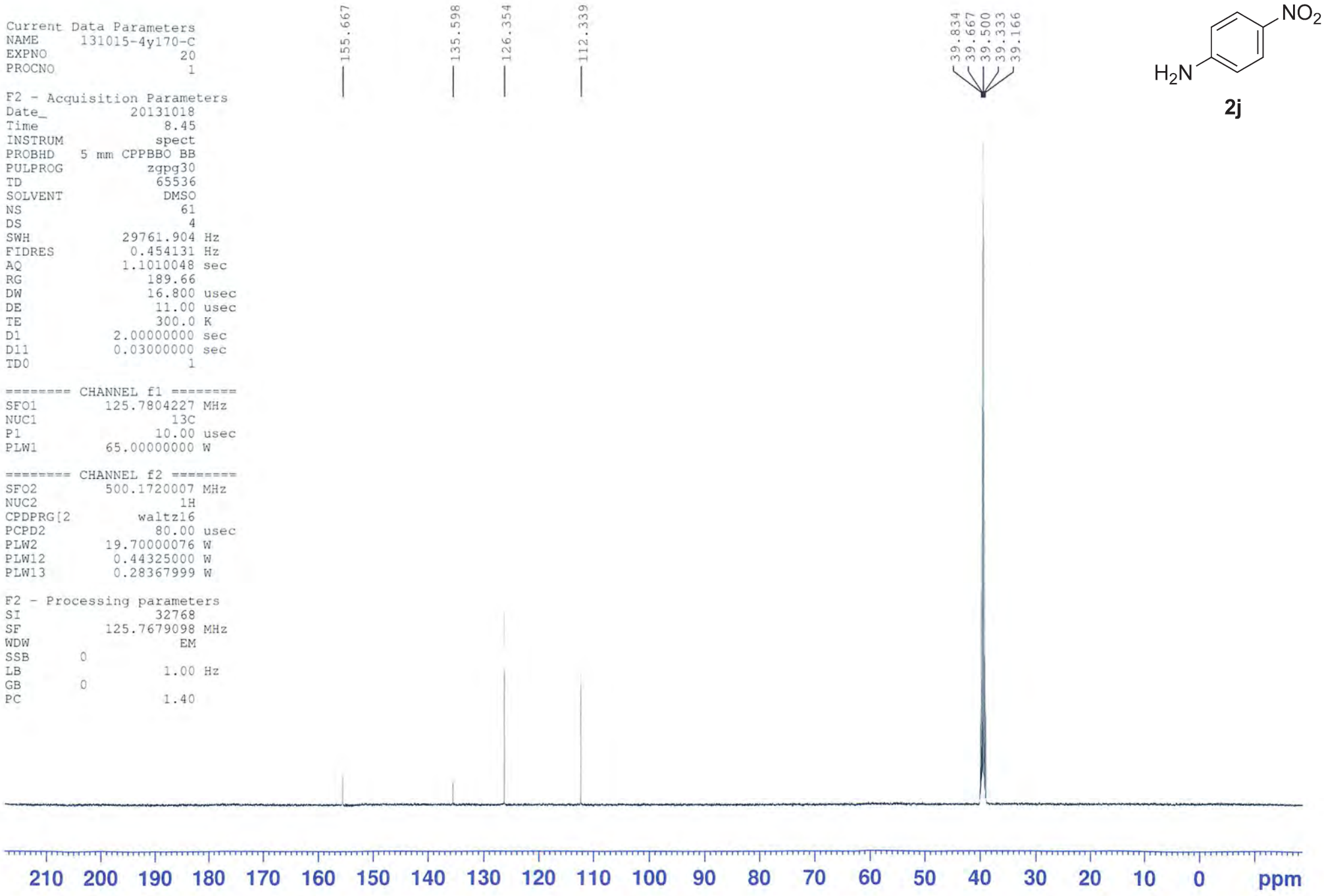
^{1}H NMR


130704-4y123-C13
exp1 bilevel
SAMPLE
date Aug 1 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 48
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -816.4
wp 20612.6
vs 162
sc 0
wc 250
hzmm 6.34
is 500.00
rfl 8559.1
rfp 7742.1
th 20
ins 100.000
nm no ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
dm yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
128.366
128.122
126.908
77.313
77.000
76.681
53.163
140.328
HN
2i
180
160
140
120
100
80
60
40
20
ppm

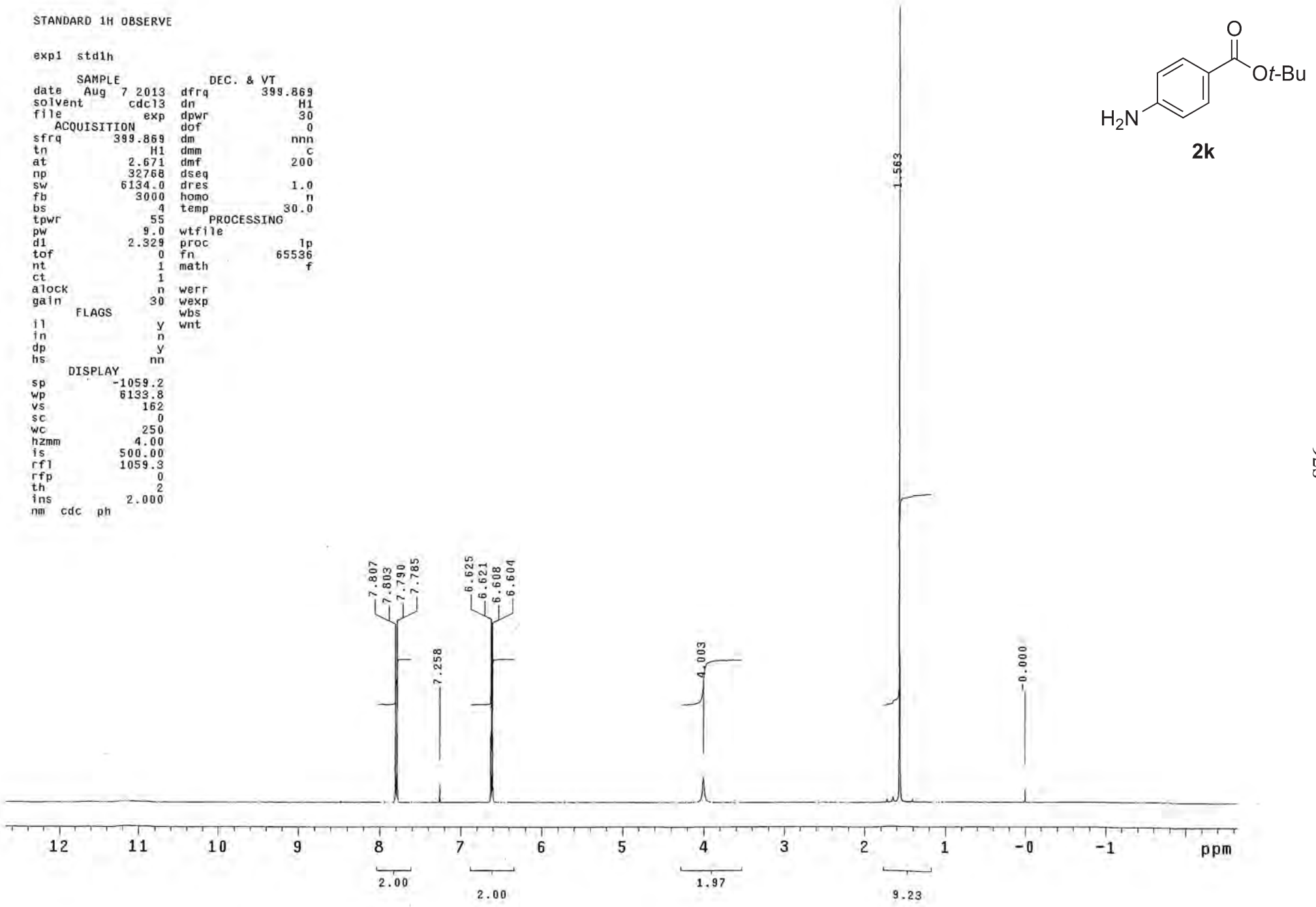
^{13}C NMR


Current Data Parameters
NAME 131015-4y170-H
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131018
Time 8.40
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 31.29
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.949
7.930
6.702
6.604
6.599
6.589
6.585
3.324
2.504
2.500
2.496
NO2
H2N
2j
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
2.000
1.955
2.070

^{1}H NMR

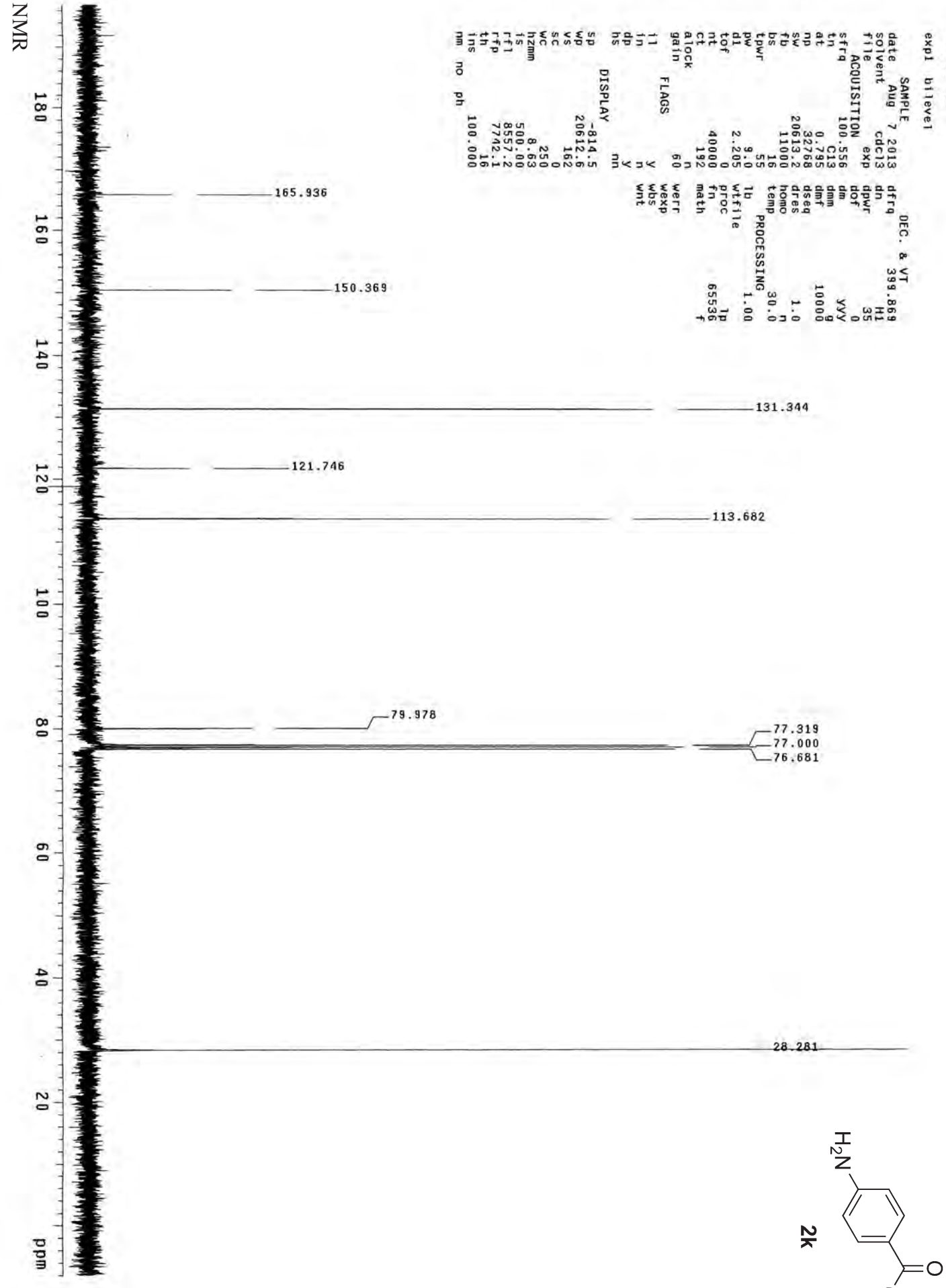

Current Data Parameters
NAME 131015-4y170-C
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131018
Time 8.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 61
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7679098 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
155.667
135.598
126.354
112.339
39.834
39.667
39.500
39.333
39.166
NO2
H2N
2j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR


STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Aug 7 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 399.869
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 55
pw 9.0
d1 2.329
tof 0
nt 1
ct 1
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1059.2
wp 6133.8
vs 162
sc 0
wc 250
hzmm 4.00
is 500.00
rfl 1059.3
rfp 0
th 2
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
7.807
7.803
7.790
7.785
7.258
6.625
6.621
6.608
6.604
4.003
1.563
0.000
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
2.00
2.00
1.97
9.23
O
Ot-Bu
H2N
2k

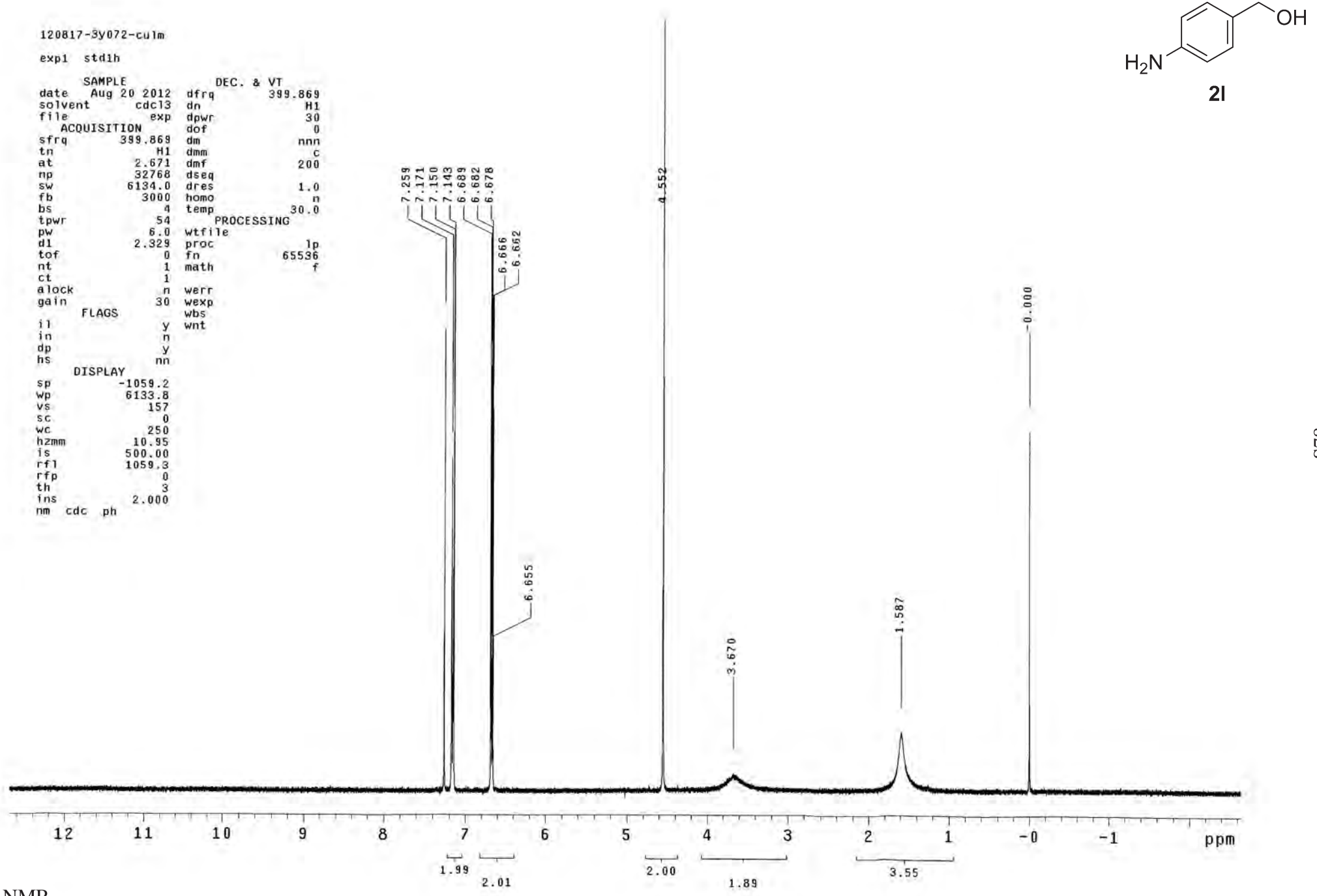
^{1}H NMR

^{13}C NMR


NSM192-C13
expl bilevel
SAMPLE
date Aug 7 2013
solvent cdcl3
file
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 192
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -814.5
wp 20612.6
vs 16
sc 0
wc 250
hzmm 8.63
is 500.00
rfl 8557.2
rfp 7742.1
th 16
ins 100.000
nm no ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
dm yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
165.936
150.369
131.344
121.746
113.682
79.978
77.319
77.000
76.681
28.281
180
160
140
120
100
80
60
40
20
ppm

H2N
O
Ot-Bu

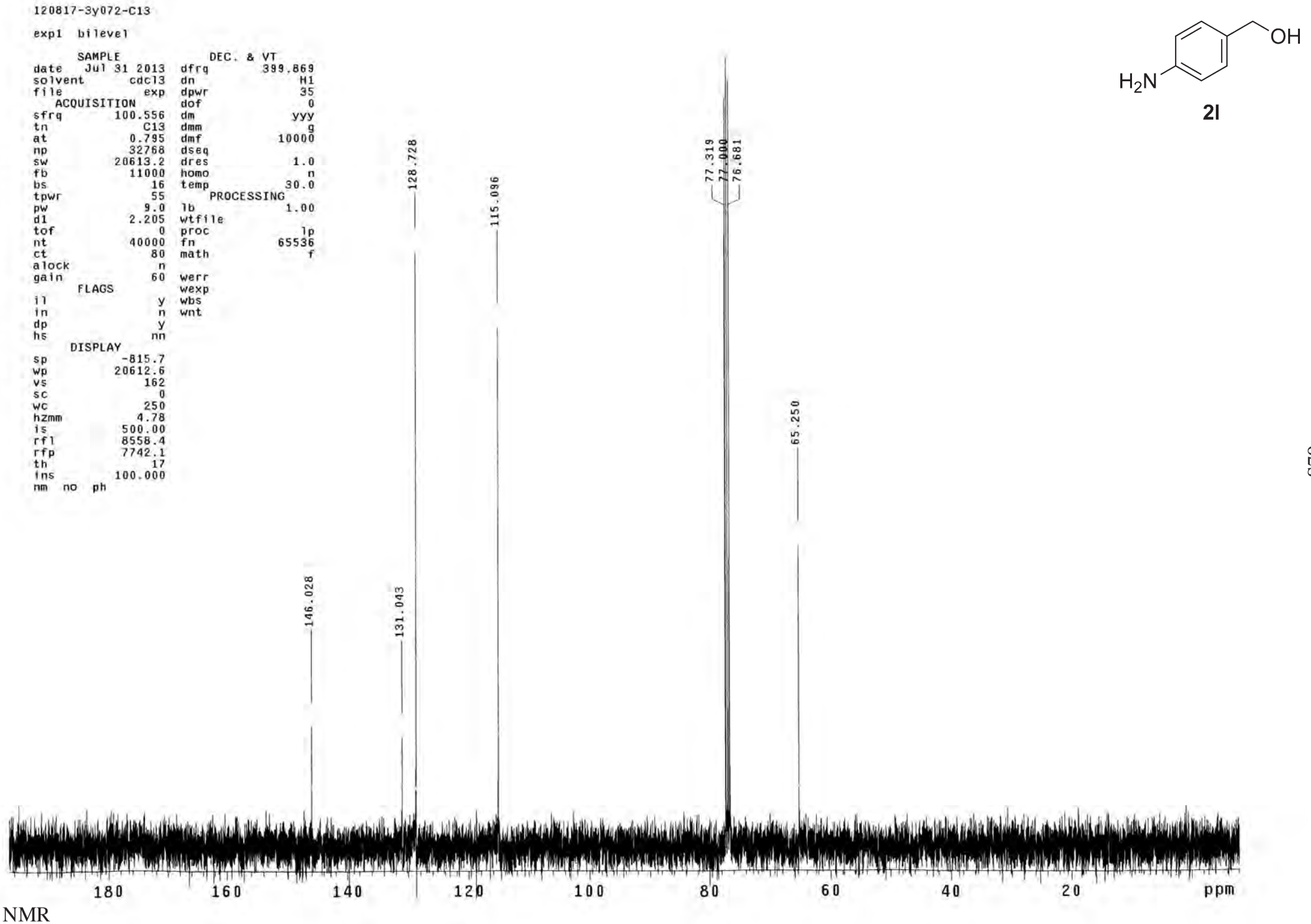
2k


120817-3y072-cu1m
exp1 stdlh
SAMPLE
date Aug 20 2012
solvent cdcl3
file exp
ACQUISITION
sfrq 399.869
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 54
pw 6.0
d1 2.329
tof 0
nt 1
ct 1
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1059.2
wp 6133.8
vs 157
sc 0
wc 250
hzmm 10.95
is 500.00
rfl 1059.3
rfp 0
th 3
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
7.259
7.171
7.150
7.143
6.689
6.682
6.678
6.666
6.662
6.655
4.552
3.670
1.587
0.000
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.99
2.01
2.00
1.89
3.55

^{1}H NMR

OH
H2N

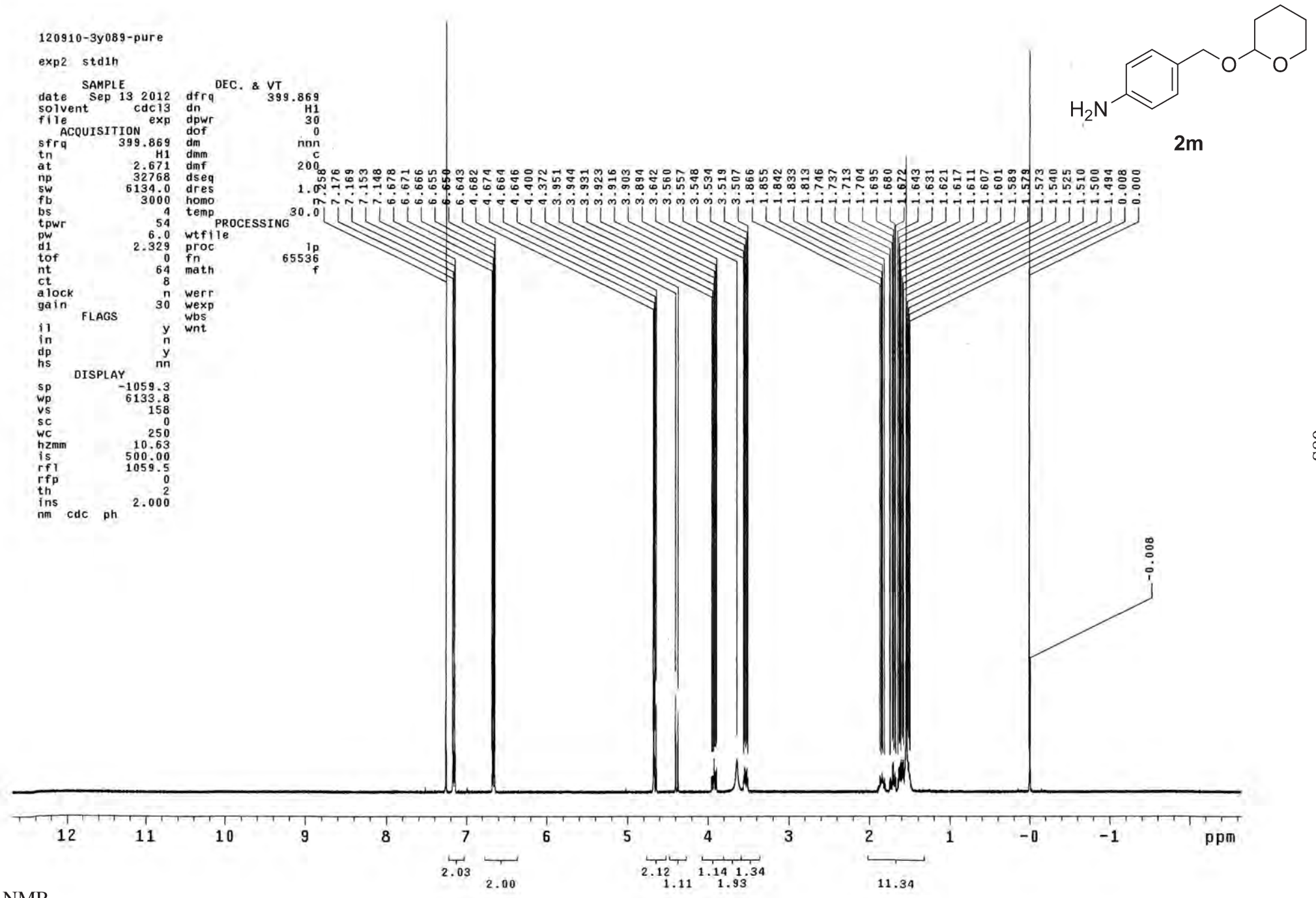
2l


120817-3y072-C13
expl bilevel
SAMPLE
date Jul 31 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 80
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -815.7
wp 20612.6
vs 162
sc 0
wc 250
hzmm 4.78
is 500.00
rfl 8558.4
rfp 7742.1
th 17
ins 100.000
nm no ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
dm yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
146.028
131.043
128.728
115.096
77.319
77.000
76.681
65.250
180
160
140
120
100
80
60
40
20
ppm

OH
H2N

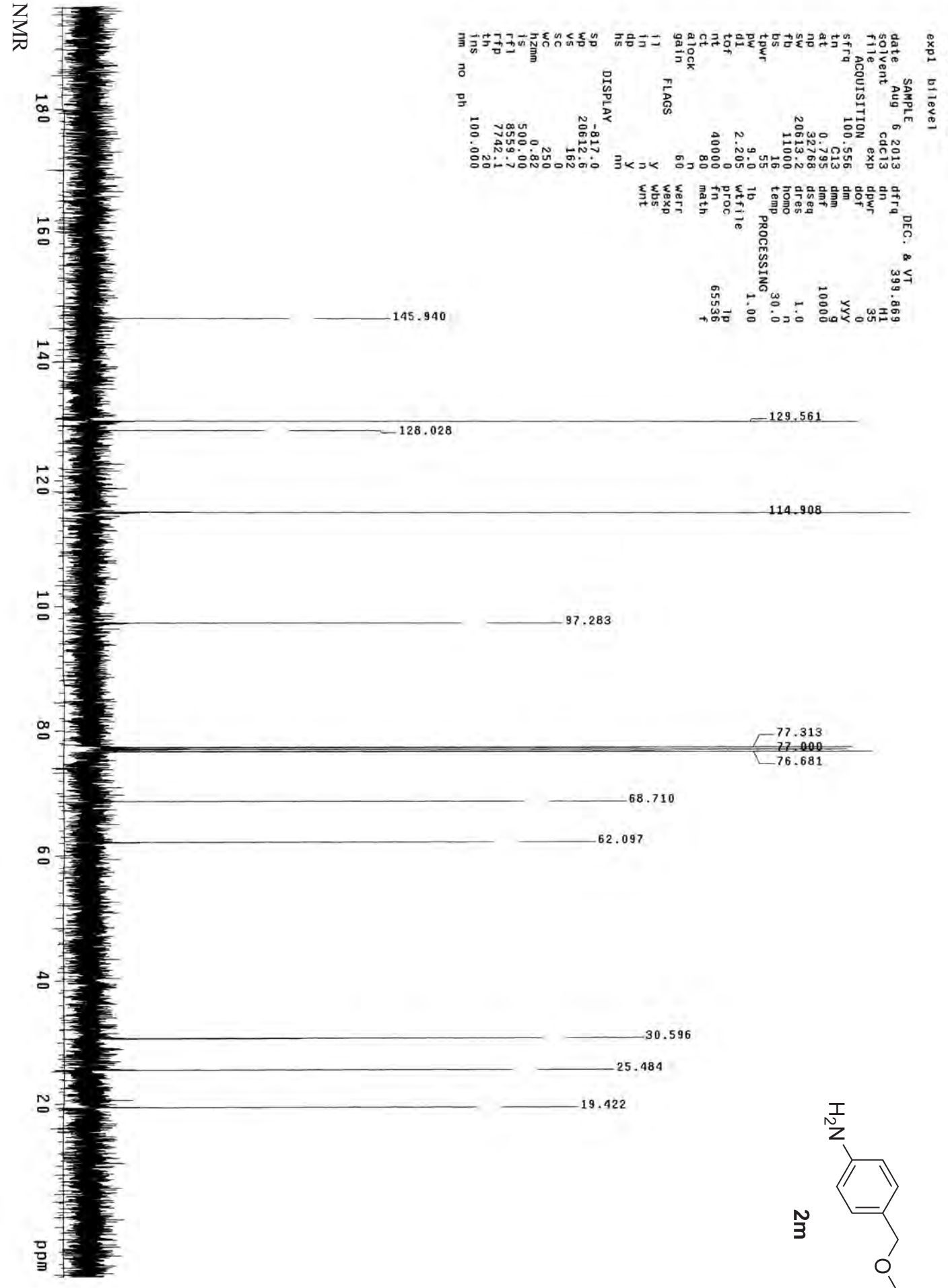
2l

^{13}C NMR


120910-3y089-pure
exp2 std1h
SAMPLE
date Sep 13 2012
solvent cdcl3
file exp
ACQUISITION
sfrq 399.869
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 54
pw 6.0
d1 2.329
tof 0
nt 64
ct 8
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1059.3
wp 6133.8
vs 158
sc 0
wc 250
hzmm 10.63
is 500.00
rfl 1059.5
rfp 0
th 2
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
7.258
7.176
7.169
7.153
7.148
6.678
6.671
6.666
6.655
6.650
6.643
4.682
4.674
4.664
4.646
4.400
4.372
3.951
3.944
3.931
3.923
3.916
3.903
3.894
3.642
3.560
3.557
3.548
3.534
3.519
3.507
1.866
1.855
1.842
1.833
1.813
1.746
1.737
1.713
1.704
1.695
1.680
1.622
1.643
1.631
1.621
1.617
1.611
1.607
1.601
1.589
1.573
1.573
1.540
1.525
1.510
1.500
1.494
0.008
0.000
-0.008
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
2.03
2.00
2.12
1.11
1.14
1.34
1.93
11.34
H_2N
O
O
2m

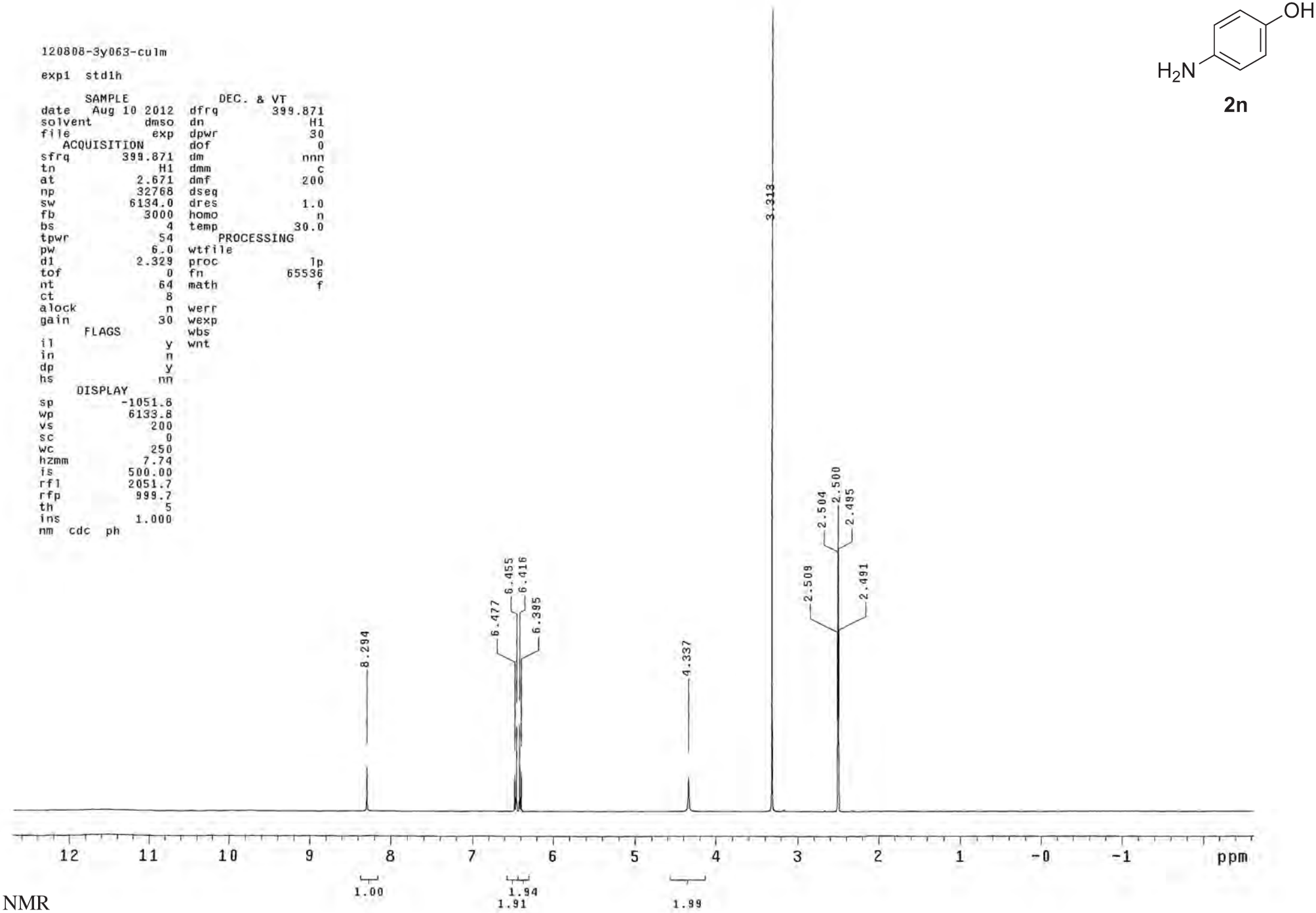
^{1}H NMR

^{13}C NMR


120910-3y089-C13
exp1 bilevel
SAMPLE
date Aug 6 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 80
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -817.0
wp 20612.6
vs 162
sc 0
wc 250
hzmm 0.82
is 500.00
rfl 8559.7
rfp 7742.1
th 20
ins 100.000
nm no ph
dfrq DEC.
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
temp
PROCESSING
lb
wtfile
proc
fn
math
werr
wexp
wbs
wnt
399.869 & VT
H1
35
0
yyy
9
10000
1.0
n
30.0
1.00
lp
65536
f
145.940
129.561
128.028
114.908
97.283
77.313
77.000
76.681
68.710
62.097
30.596
25.484
19.422
180
160
140
120
100
80
60
40
20
ppm

H2N
O
O

2m

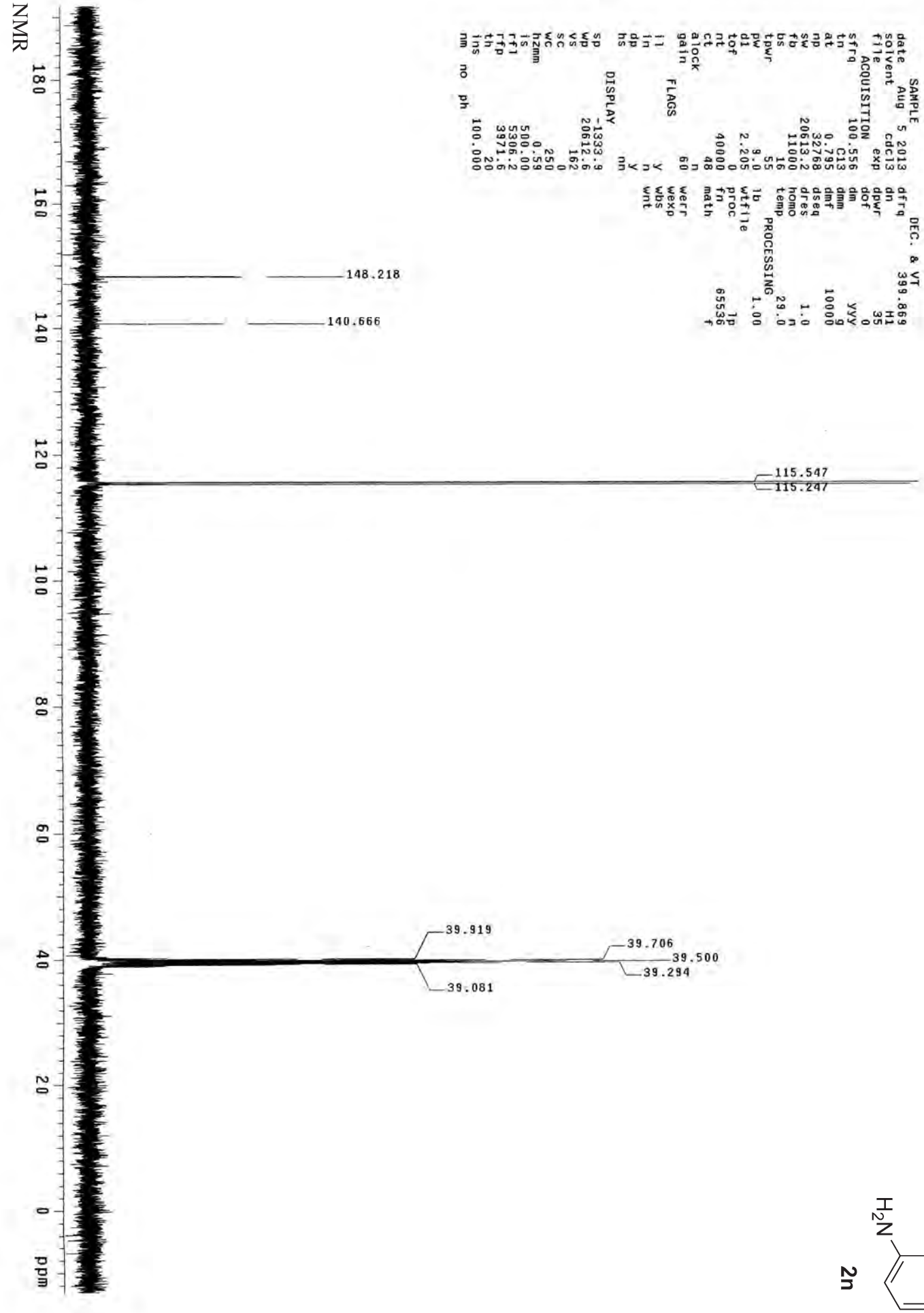

120808-3y063-culm
exp1 std1h
SAMPLE
date Aug 10 2012
solvent dmso
file exp
ACQUISITION
sfrq 399.871
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 54
pw 6.0
d1 2.329
tof 0
nt 64
ct 8
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1051.8
wp 6133.8
vs 200
sc 0
wc 250
hzmm 7.74
is 500.00
rfl 2051.7
rfp 999.7
th 5
ins 1.000
nm cdc ph
DEC. & VT
dfrq 399.871
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
8.294
6.477
6.455
6.416
6.395
4.337
3.318
2.509
2.504
2.500
2.495
2.491
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.00
1.94
1.91
1.99

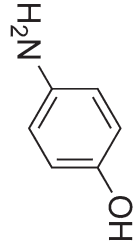
OH
H2N

2n

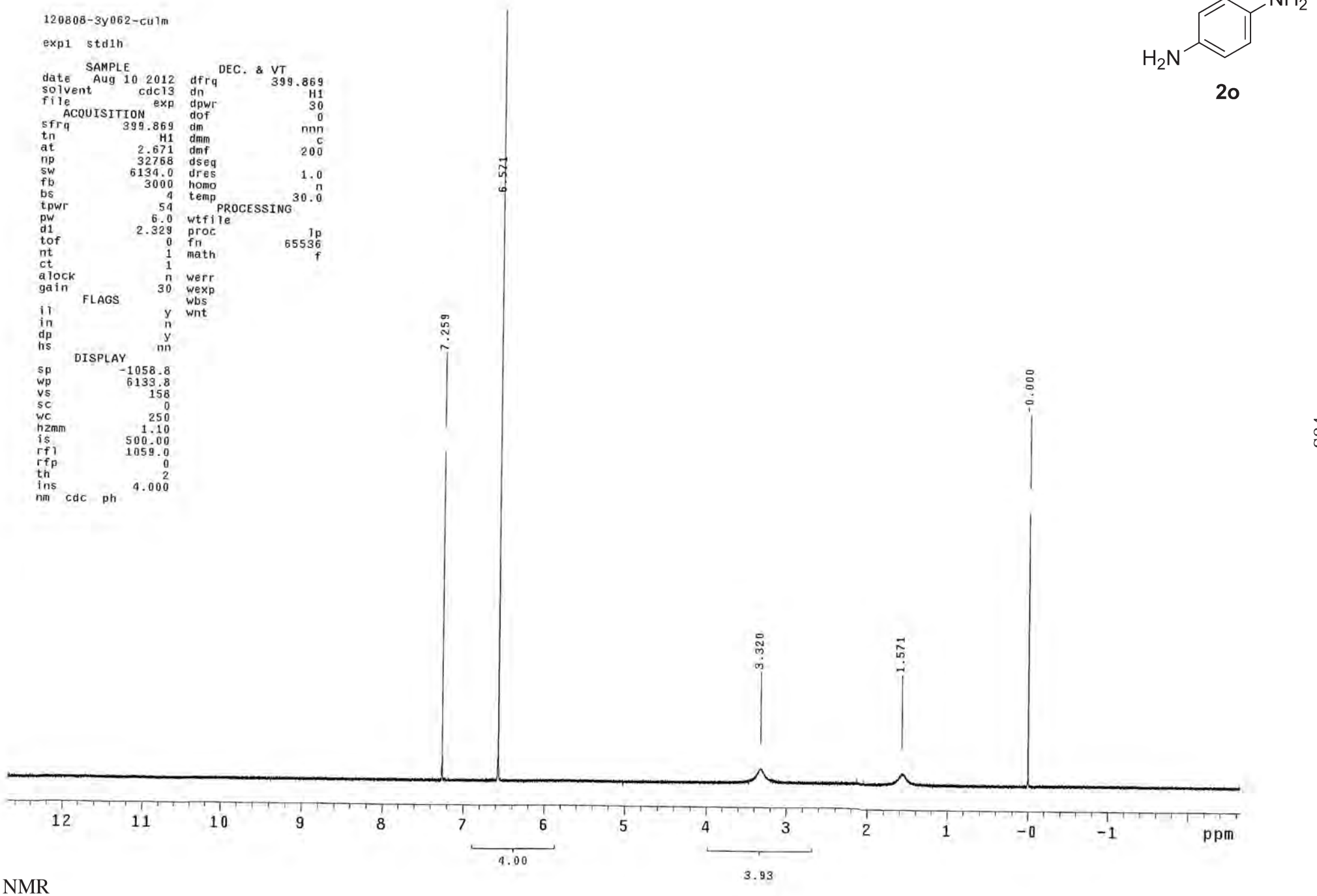
^{1}H NMR

^{13}C NMR


120808-3y063-C13
exp2 bilevel
SAMPLE
date Aug 5 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 48
alock
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1333.9
wp 20612.6
vs 162
sc 0
wc 250
hzmm 0.59
is 500.00
rfl 5306.2
rfp 3971.6
th 20
ins
nm no ph 100.000
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
dm yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 29.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
148.218
140.666
115.547
115.247
39.919
39.706
39.500
39.294
39.081
180
160
140
120
100
80
60
40
20
0
ppm


H2N
OH

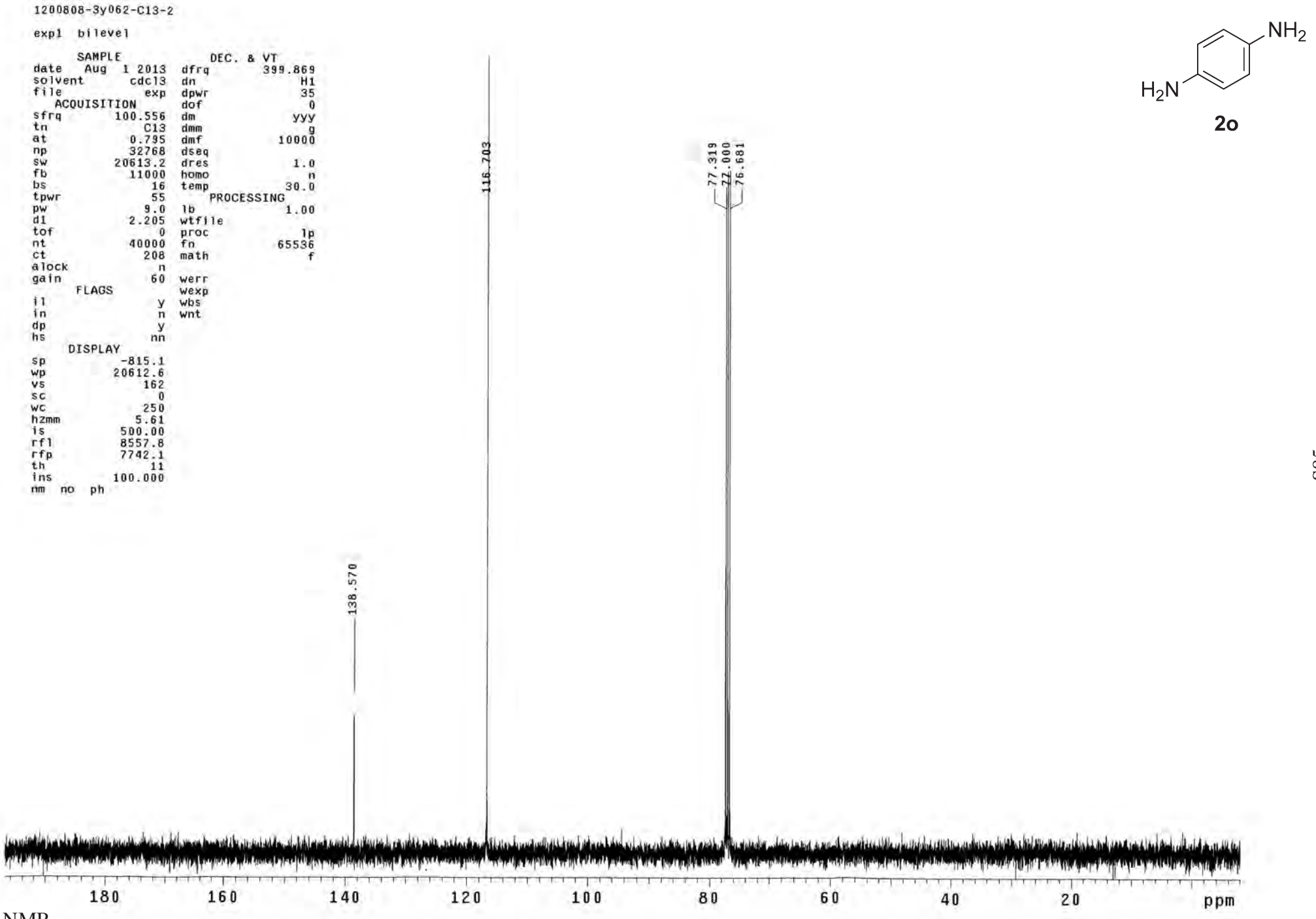
2n


120808-3y062-culm
exp1 stdlh
SAMPLE
date Aug 10 2012
solvent cdcl3
file exp
ACQUISITION
sfrq 399.869
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 54
pw 6.0
d1 2.329
tof 0
nt 1
ct 1
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1058.8
wp 6133.8
vs 158
sc 0
wc 250
hzmm 1.10
is 500.00
rfl 1059.0
rfp 0
th 2
ins 4.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
7.259
6.571
3.320
1.571
-0.000
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
4.00
3.93

NH2
H2N

2o

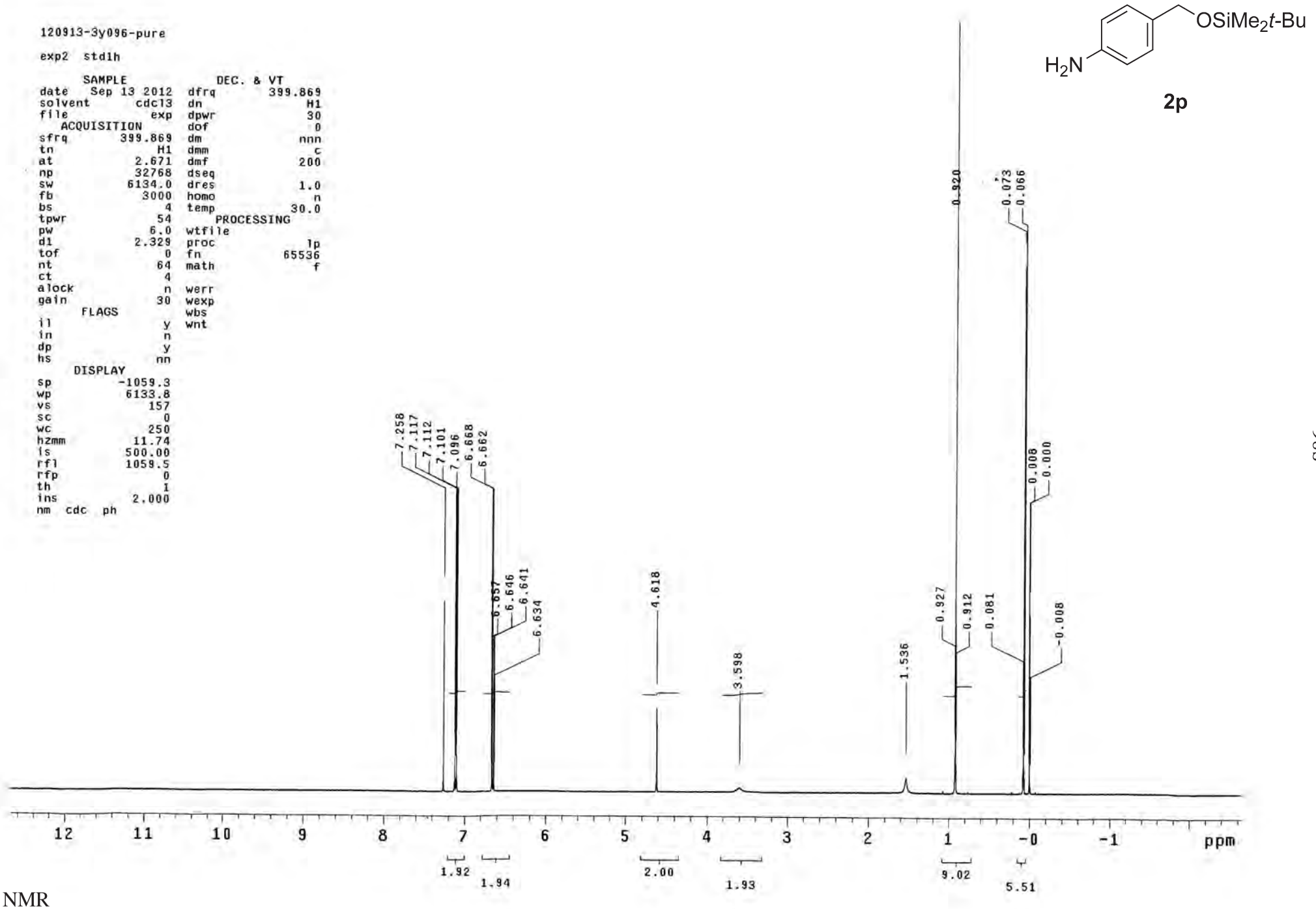
^{1}H NMR


1200808-3y062-C13-2
expl bilevel
SAMPLE
date Aug 1 2013
solvent cdcl3
file exp
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 208
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -815.1
wp 20612.6
vs 162
sc 0
wc 250
hzmm 5.61
is 500.00
rfl 8557.8
rfp 7742.1
th 11
ins 100.000
nm no ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
dm yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
138.570
116.703
77.319
77.000
76.681
180
160
140
120
100
80
60
40
20
ppm

NH2
H2N

2o

^{13}C NMR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exp2 std1h
SAMPLE
date Sep 13 2012
solvent cdcl3
file exp
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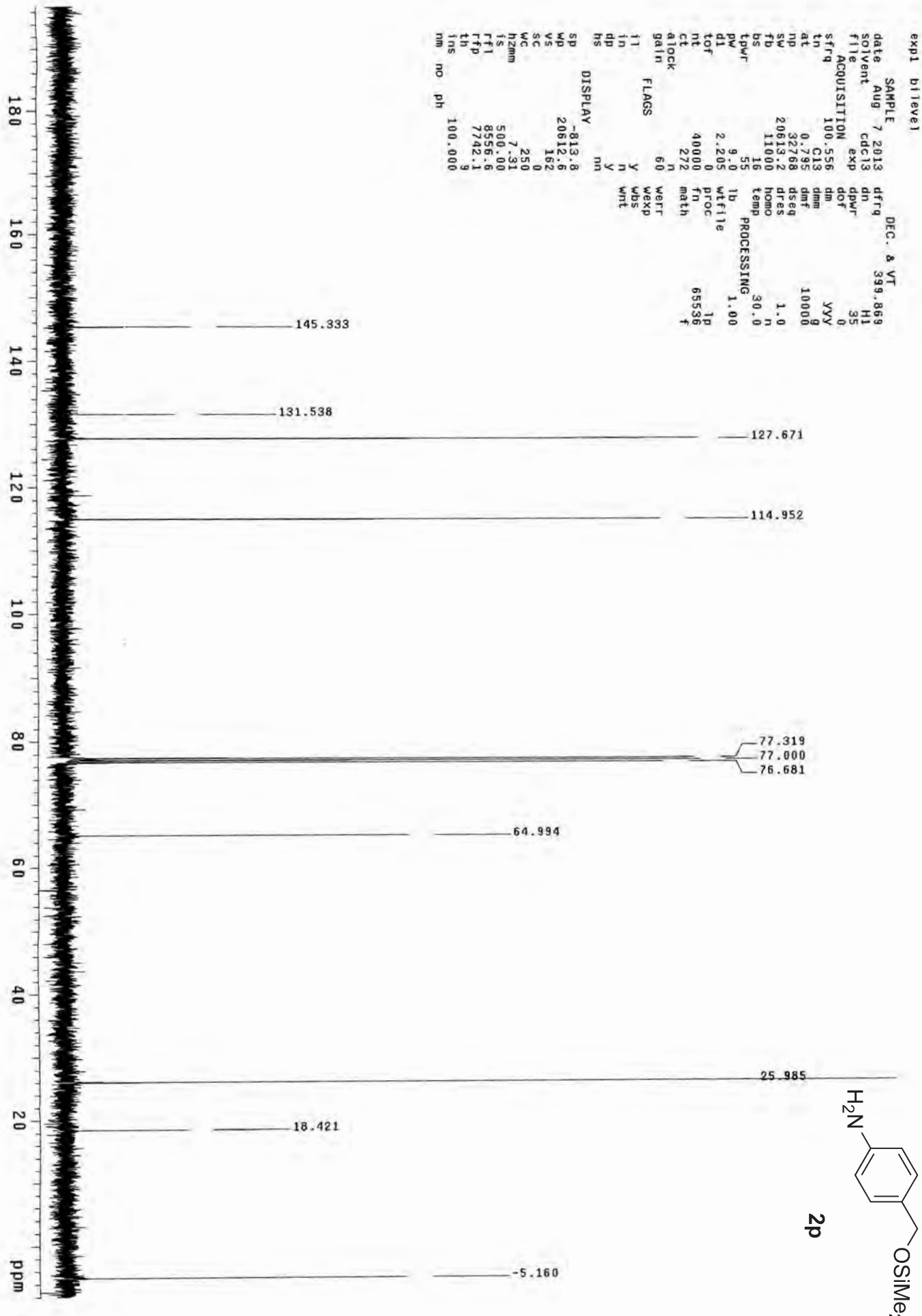
tn H1
at 2.671
np 32768
sw 6134.0
fb 3000
bs 4
tpwr 54
pw 6.0
d1 2.329
tof 0
nt 64
ct 4
alock n
gain 30
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -1059.3
wp 6133.8
vs 157
sc 0
wc 250
hzmm 11.74
is 500.00
rfl 1059.5
rfp 0
th 1
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.869
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
7.258
7.117
7.112
7.101
7.096
6.668
6.662
6.657
6.646
6.641
6.634
4.618
3.598
1.536
0.920
0.927
0.912
0.081
0.073
0.066
0.008
0.000
-0.008
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.92
1.94
2.00
1.93
9.02
5.51

OSiMe2t-Bu
H2N

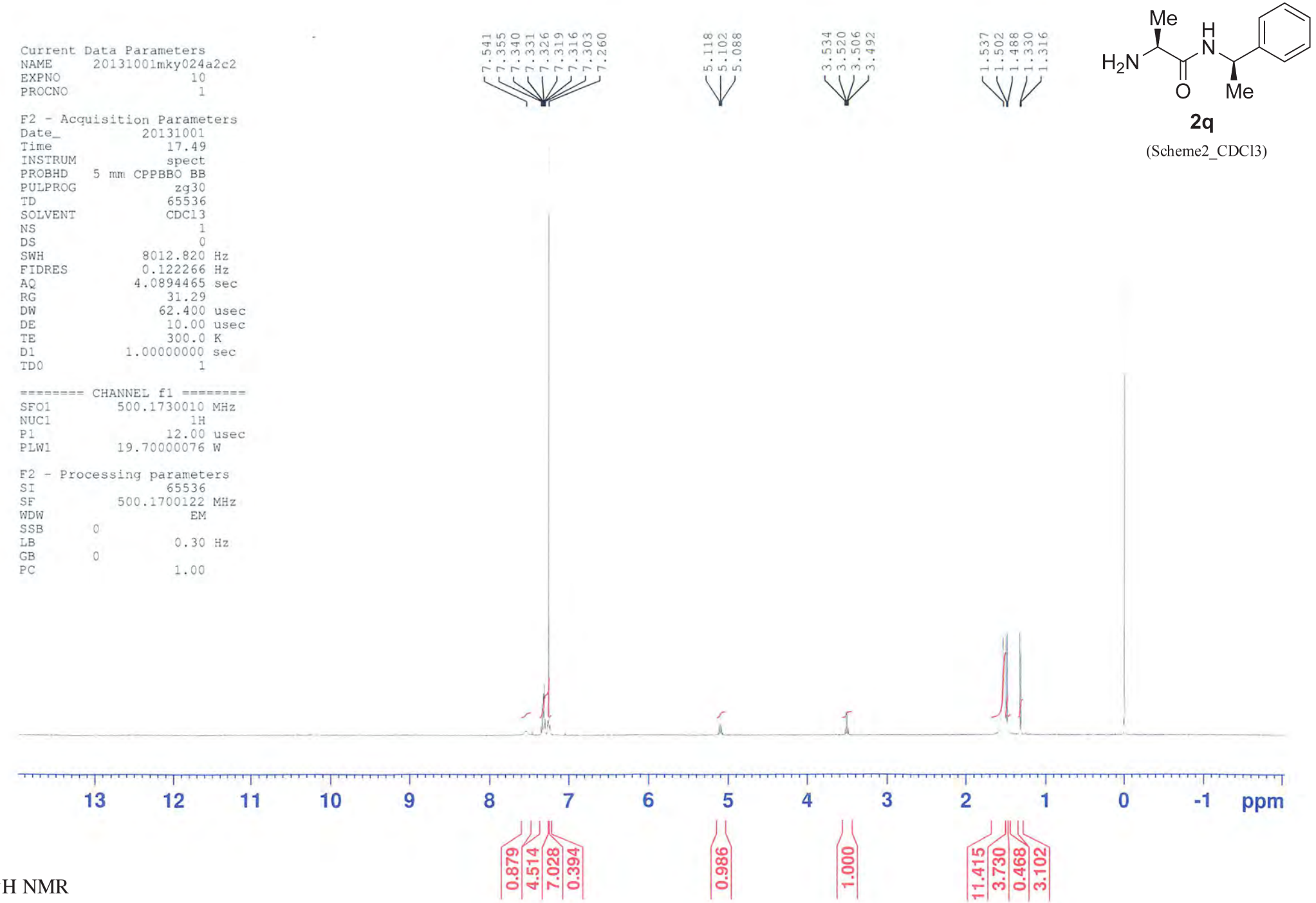
2p

^{1}H NMR

^{13}C NMR


120913-3y096-C13
exp1 bileve1
SAMPLE
date Aug 7 2013
solvent cdcl3
file
ACQUISITION
sfrq 100.556
tn C13
at 0.795
np 32768
sw 20613.2
fb 11000
bs 16
tpwr 55
pw 9.0
d1 2.205
tof 0
nt 40000
ct 272
alock n
gain 60
FLAGS
il y
in n
dp y
hs nn
DISPLAY
sp -813.8
wp 20612.6
vs 162
sc 0
wc 250
hzmm 7.31
is 500.00
rfl 8556.6
rfp 7742.1
th 9
ins
nm no ph
100.000
DEC. & VT
dfrq 399.869
dn H1
dpwr 35
dof 0
dm yyy
dmm g
dmf 10000
dseq
dres 1.0
homo n
temp 30.0
PROCESSING
lb 1.00
wtfile
proc lp
fn 65536
math f
werr
wexp
wbs
wnt
145.333
131.538
127.671
114.952
77.319
77.000
76.681
64.994
25.985
18.421
-5.160
180
160
140
120
100
80
60
40
20
ppm
H2N
OSiMe2t-Bu

2p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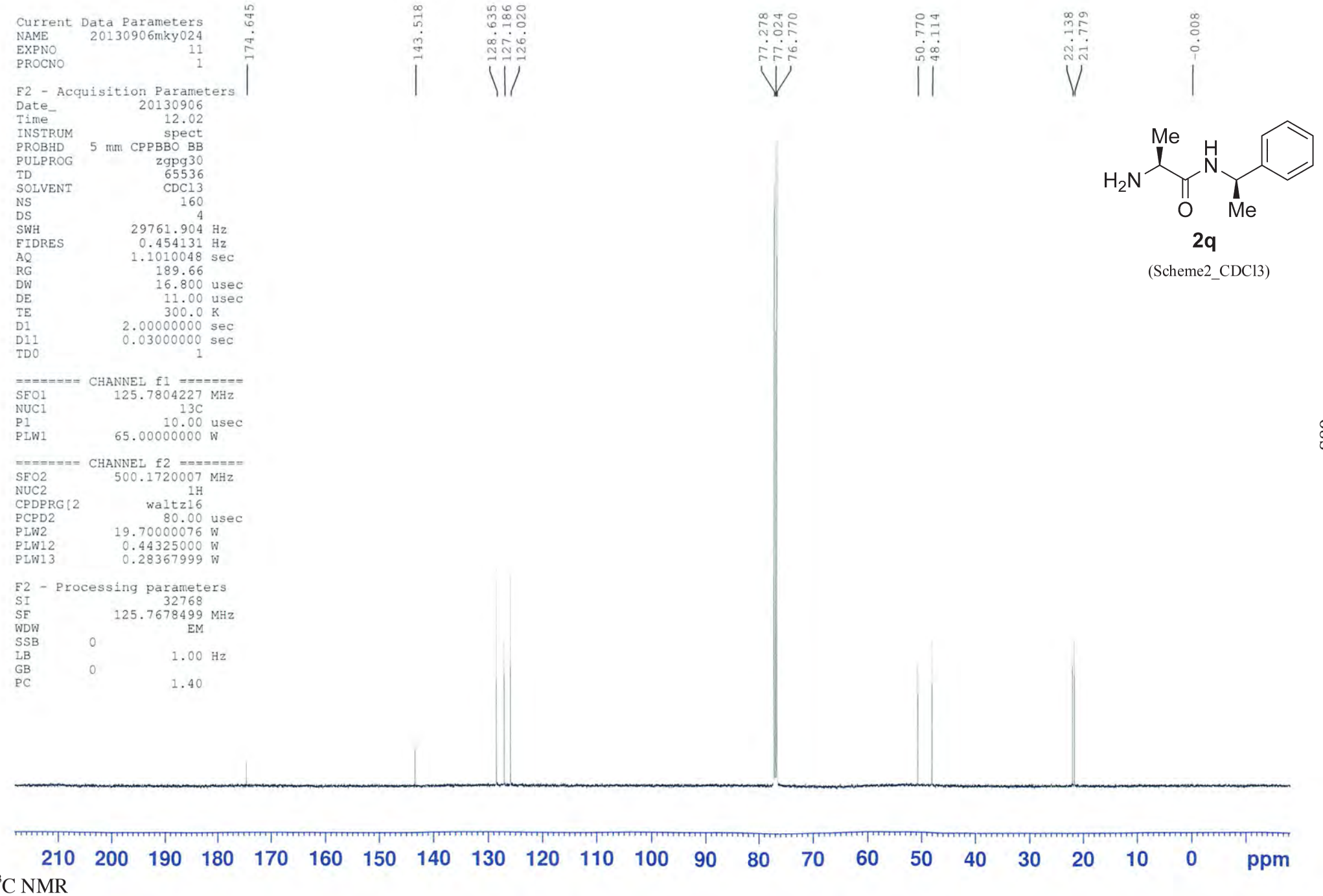
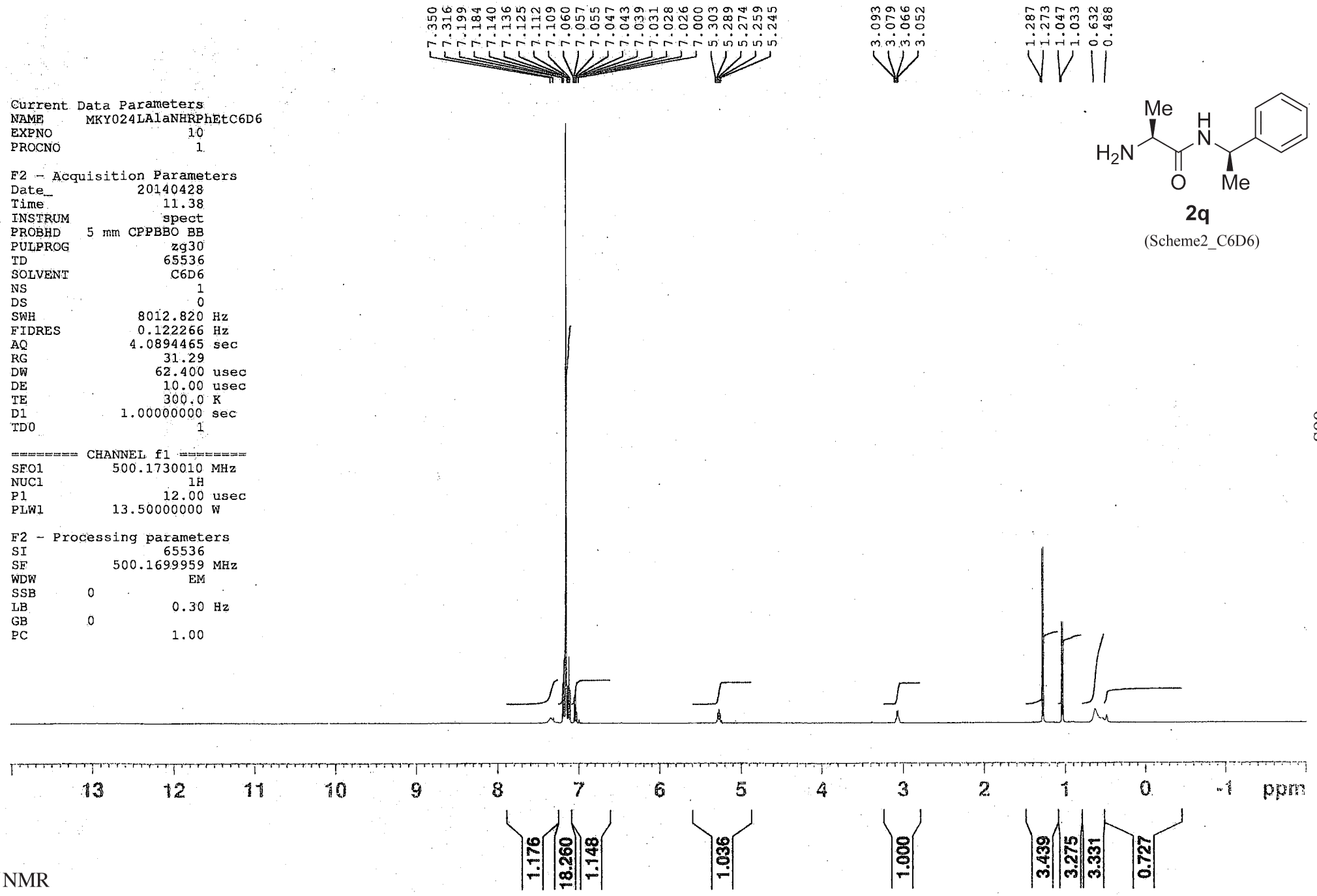
NAME 20131001mky024a2c2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131001
Time 17.49
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.541
7.355
7.340
7.331
7.326
7.319
7.316
7.303
7.260
5.118
5.102
5.088
3.534
3.520
3.506
3.492
1.537
1.502
1.488
1.330
1.316
Me
H
N
H2N
O
Me
2q
(Scheme2_CDCl3)
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.879
4.514
7.028
0.394
0.986
1.000
11.415
3.730
0.468
3.102

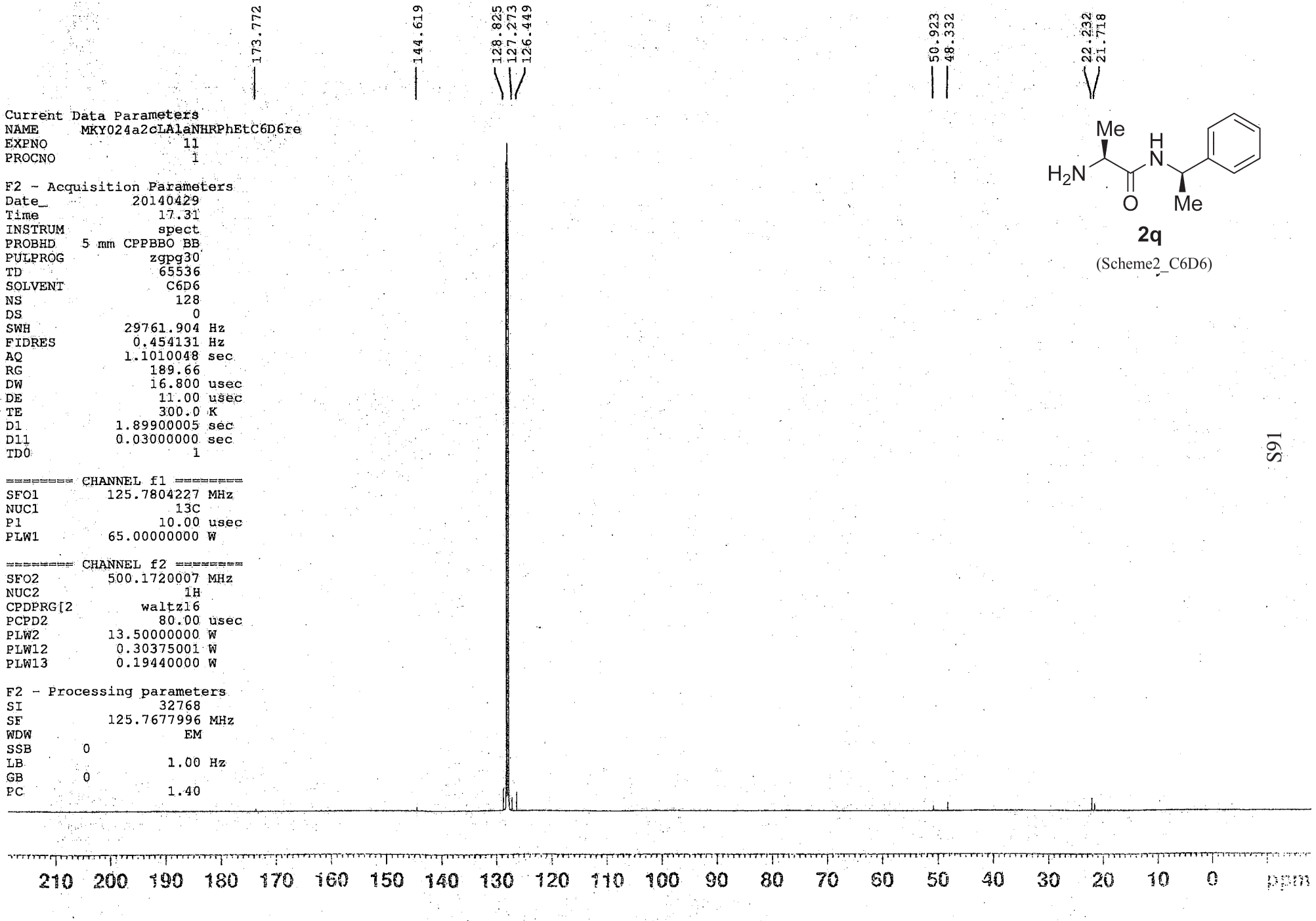
^{1}H NMR


Current Data Parameters
NAME 20130906mky024
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130906
Time 12.02
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7678499 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
174.645
143.518
128.635
127.186
126.020
77.278
77.024
76.770
50.770
48.114
22.138
21.779
-0.008
Me
H
N
H2N
O
Me
2q
(Scheme2_CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

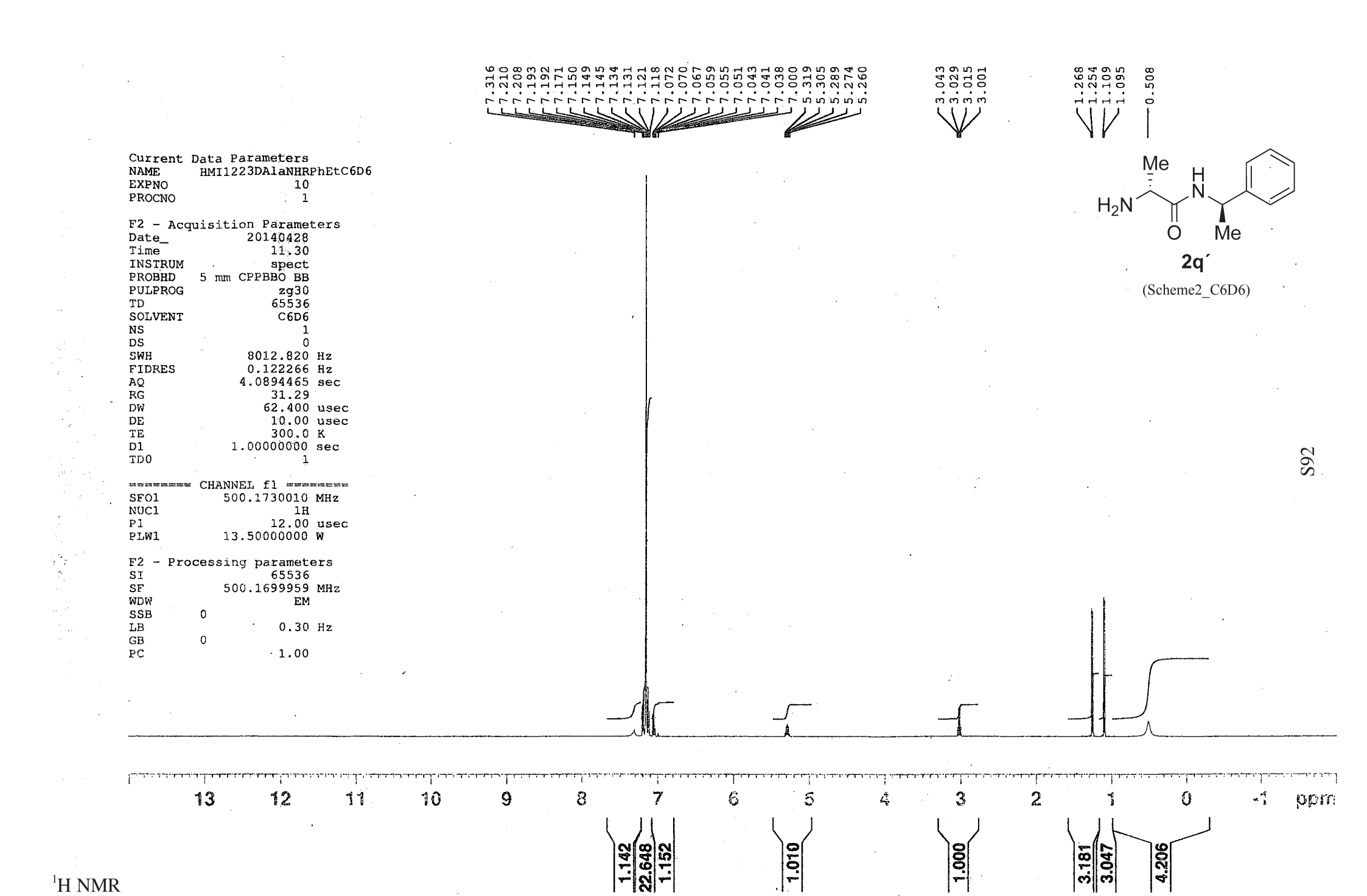
^{13}C NMR


7.350
7.316
7.199
7.184
7.140
7.136
7.125
7.112
7.109
7.060
7.057
7.055
7.047
7.043
7.039
7.031
7.028
7.026
7.000
5.303
5.289
5.274
5.259
5.245
3.093
3.079
3.066
3.052
1.287
1.273
1.047
1.033
0.632
0.488
Current Data Parameters
NAME MKY024LAlaNHRPhEtC6D6
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140428
Time 11.38
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1699959 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
H
N
H2N
O
Me
2q
(Scheme2_C6D6)
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.176
18.260
1.148
1.036
1.000
3.439
3.275
3.331
0.727

^{1}H NMR

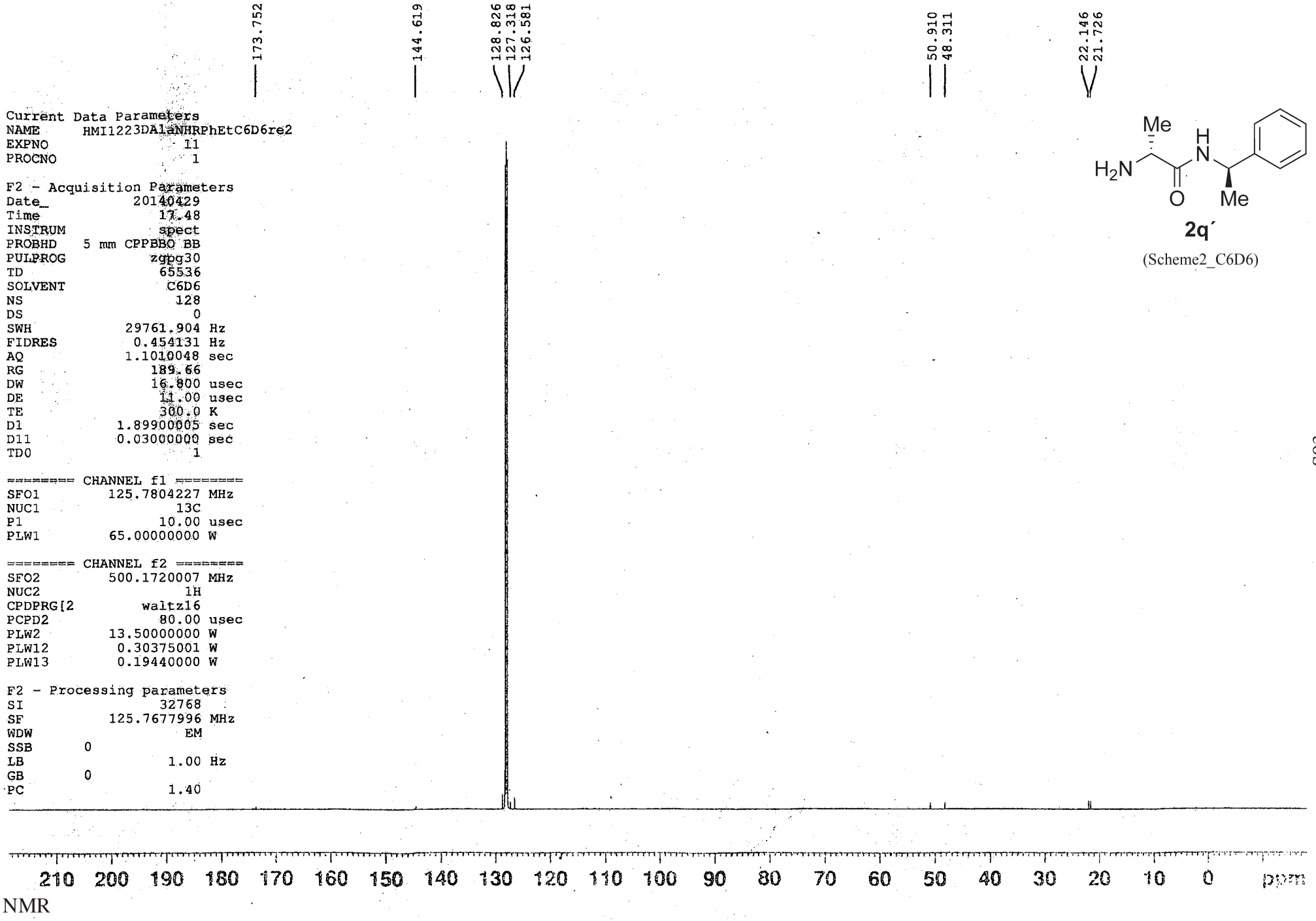

173.772
144.619
128.825
127.273
126.449
50.923
48.332
22.232
21.718
Current Data Parameters
NAME MKY024a2cLAlaNHRPhEtC6D6re
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140429
Time 17.31
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7677996 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
H
N
H2N
O
Me
2q
(Scheme2_C6D6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

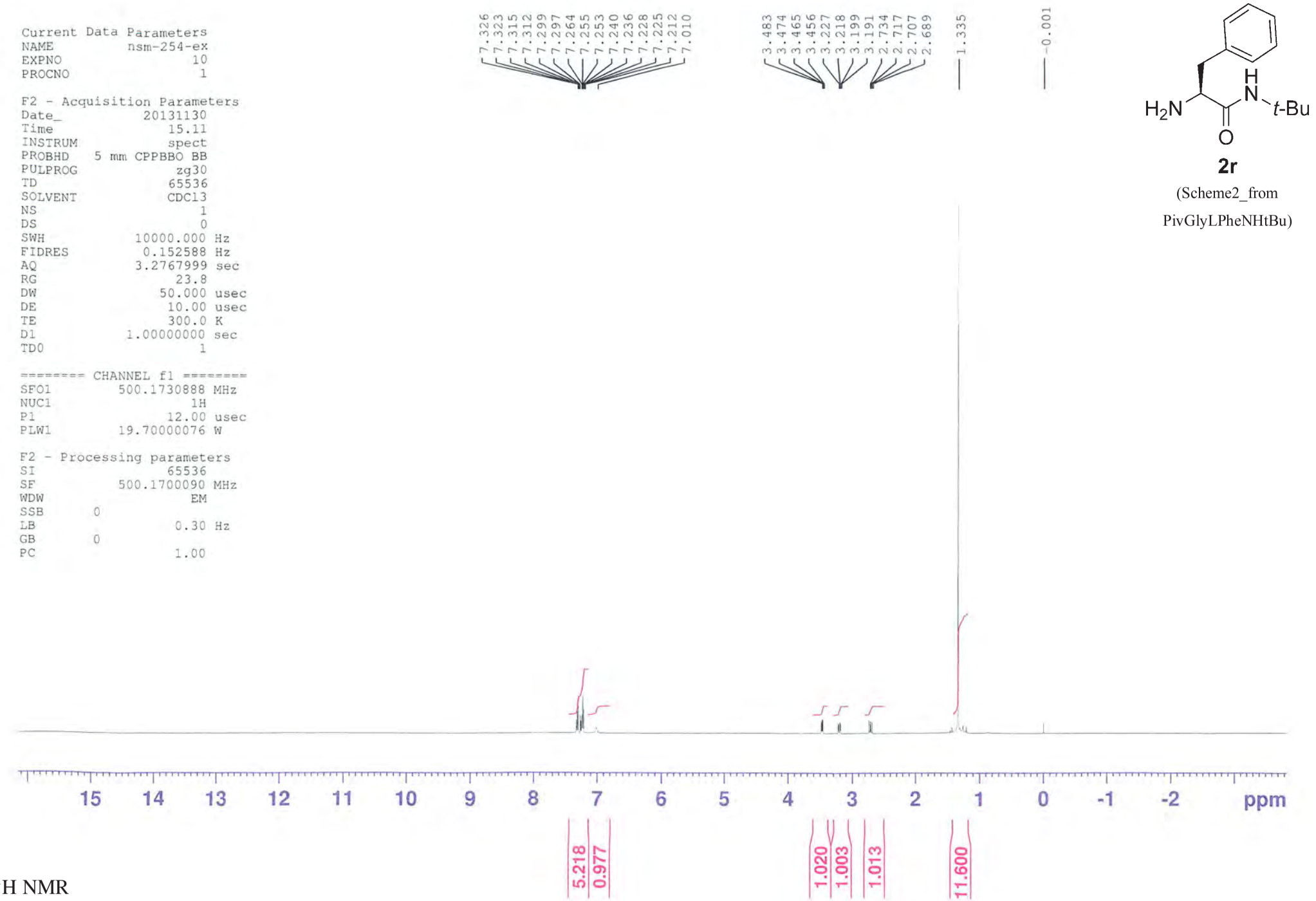

Current Data Parameters
NAME HMI1223DAlaNHRPhEtC6D6
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140428
Time 11.30
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.29
DW 62.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730010 MHz
NUC1 1H
P1 12.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1699959 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.316
7.210
7.208
7.193
7.192
7.171
7.150
7.149
7.145
7.134
7.131
7.121
7.118
7.072
7.070
7.067
7.059
7.055
7.051
7.043
7.041
7.038
7.000
5.319
5.305
5.289
5.274
5.260
3.043
3.029
3.015
3.001
1.268
1.254
1.109
1.095
0.508
Me
H
N
H2N
O
Me
2q´
(Scheme2_C6D6)
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.142
22.648
1.152
1.010
1.000
3.181
3.047
4.206

S92

^{1}H NMR


173.752
144.619
128.826
127.318
126.581
50.910
48.311
22.146
21.726
Current Data Parameters
NAME HMI1223DAlaNHRPhEtC6D6re2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140429
Time 17.48
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 128
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7677996 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
H
N
H2N
O
Me
2q´
(Scheme2_C6D6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

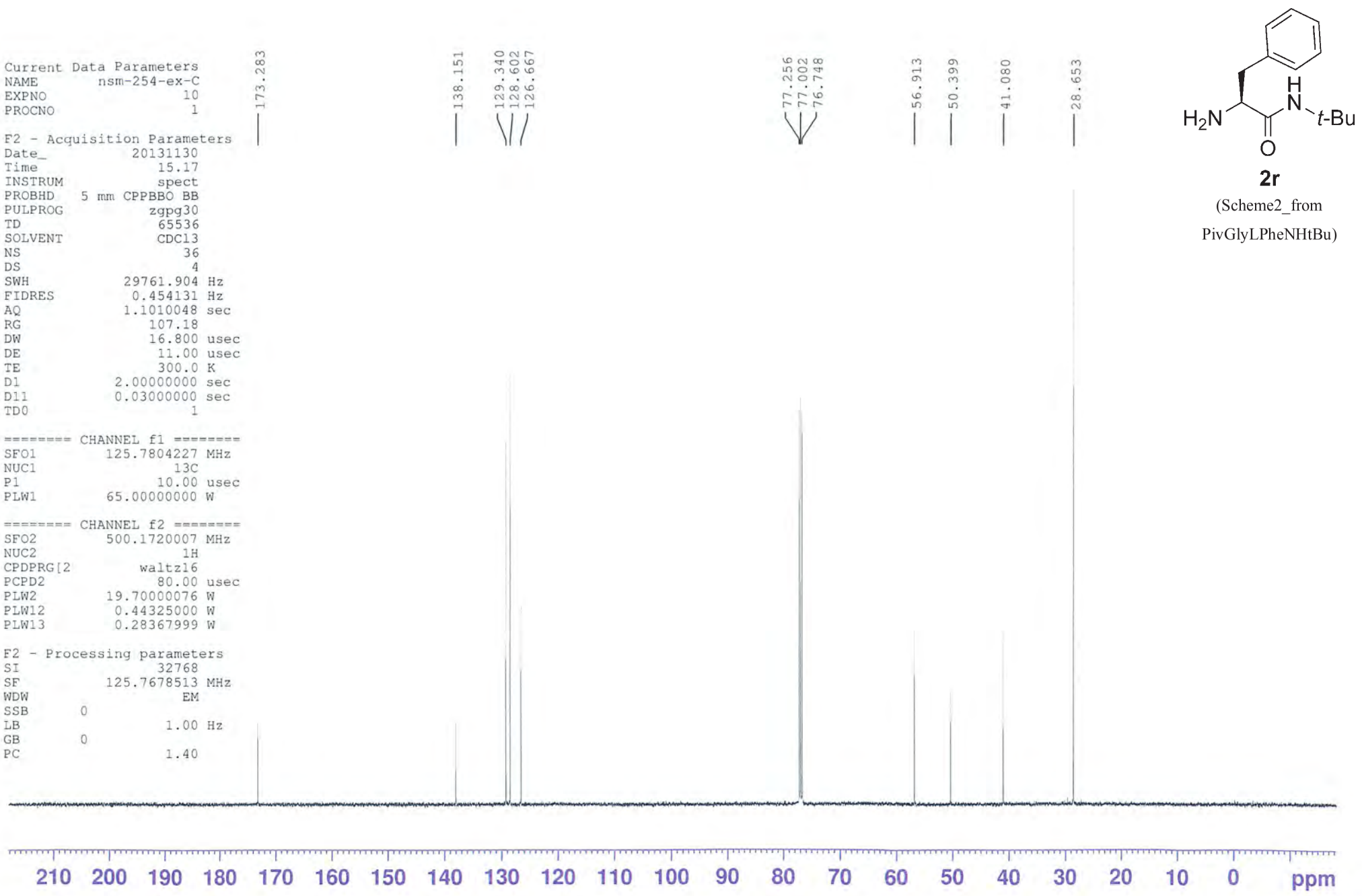
^{13}C NMR



2r

(Scheme2_from PivGlyLPheNHtBu)

^{1}H NMR

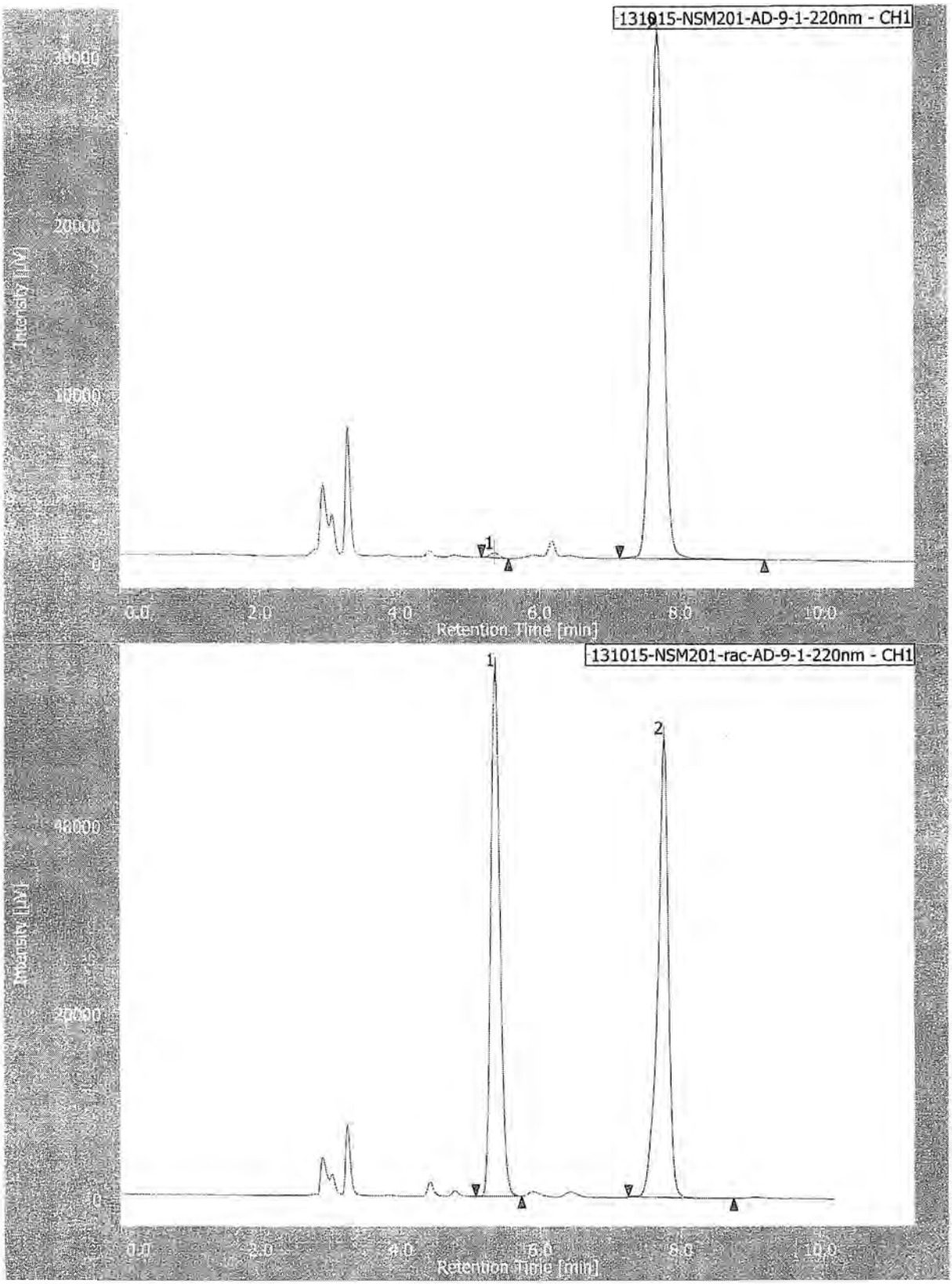


2r

(Scheme2_from PivGlyLPheNHtBu)

^{13}C NMR

HPLC



チャンネル情報＋ピーク情報

クロマトグラム名　131015-NSM201-AD-9-1-220nm-CH1

サンプル名

チャンネル名　CH1

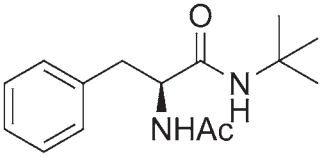
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.350	2700	295	0.675	0.942	N/A	7695	8.031	1.057	
2	Unknown	1	7.675	397325	30975	99.325	99.058	N/A	8320	N/A	0.972	

クロマトグラム名　131015-NSM201-rac-AD-9-1-220nm-CH1

サンプル名

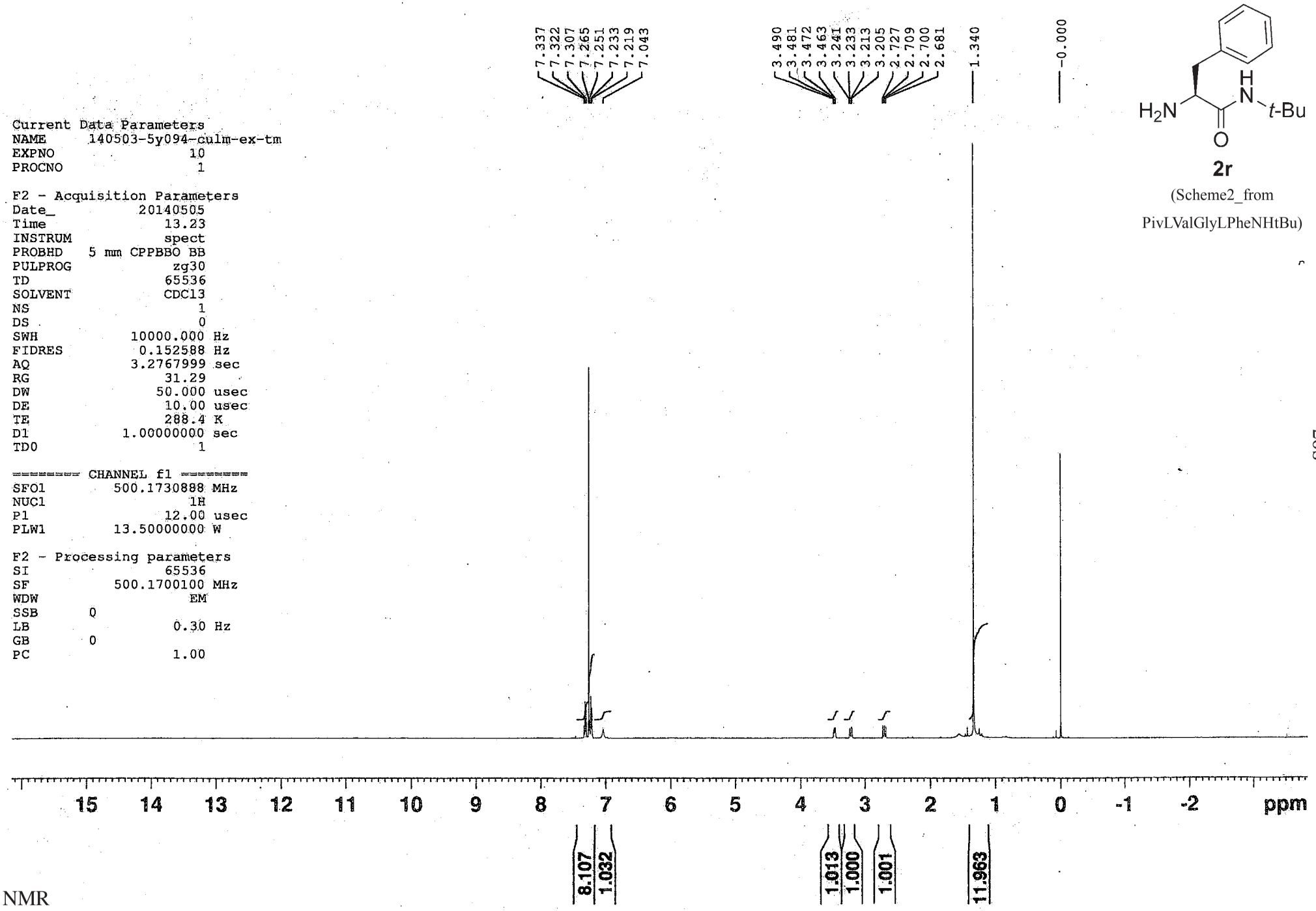
チャンネル名　CH1

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.358	519234	56310	49.849	53.405	N/A	7738	9.666	1.121	
2	Unknown	1	7.758	522381	49130	50.151	46.595	N/A	14898	N/A	0.806	



3

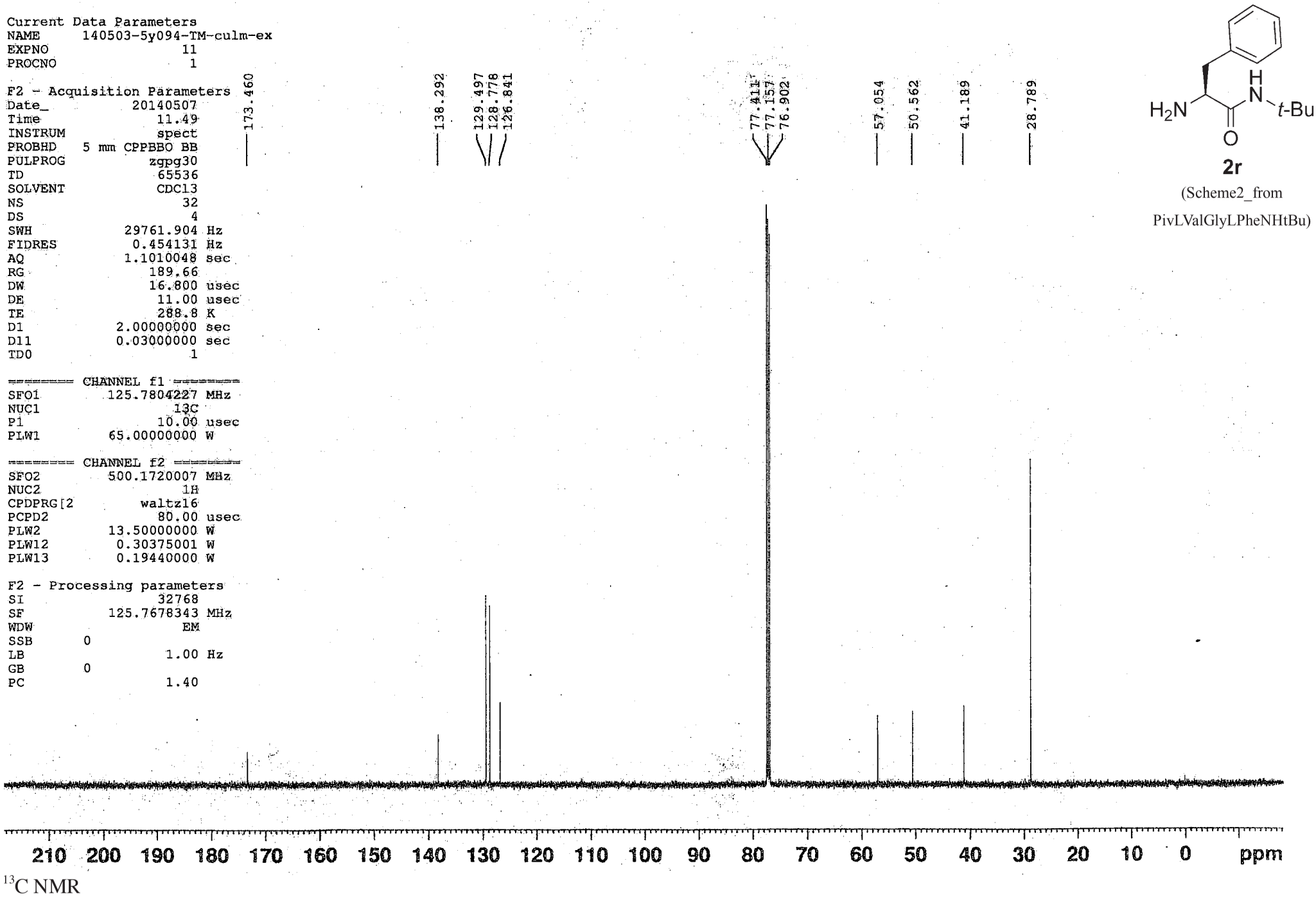
(Scheme2_from PivGlyLPheNHtBu)



2r

(Scheme2_from PivLValGlyLPheNHtBu)

^{1}H NMR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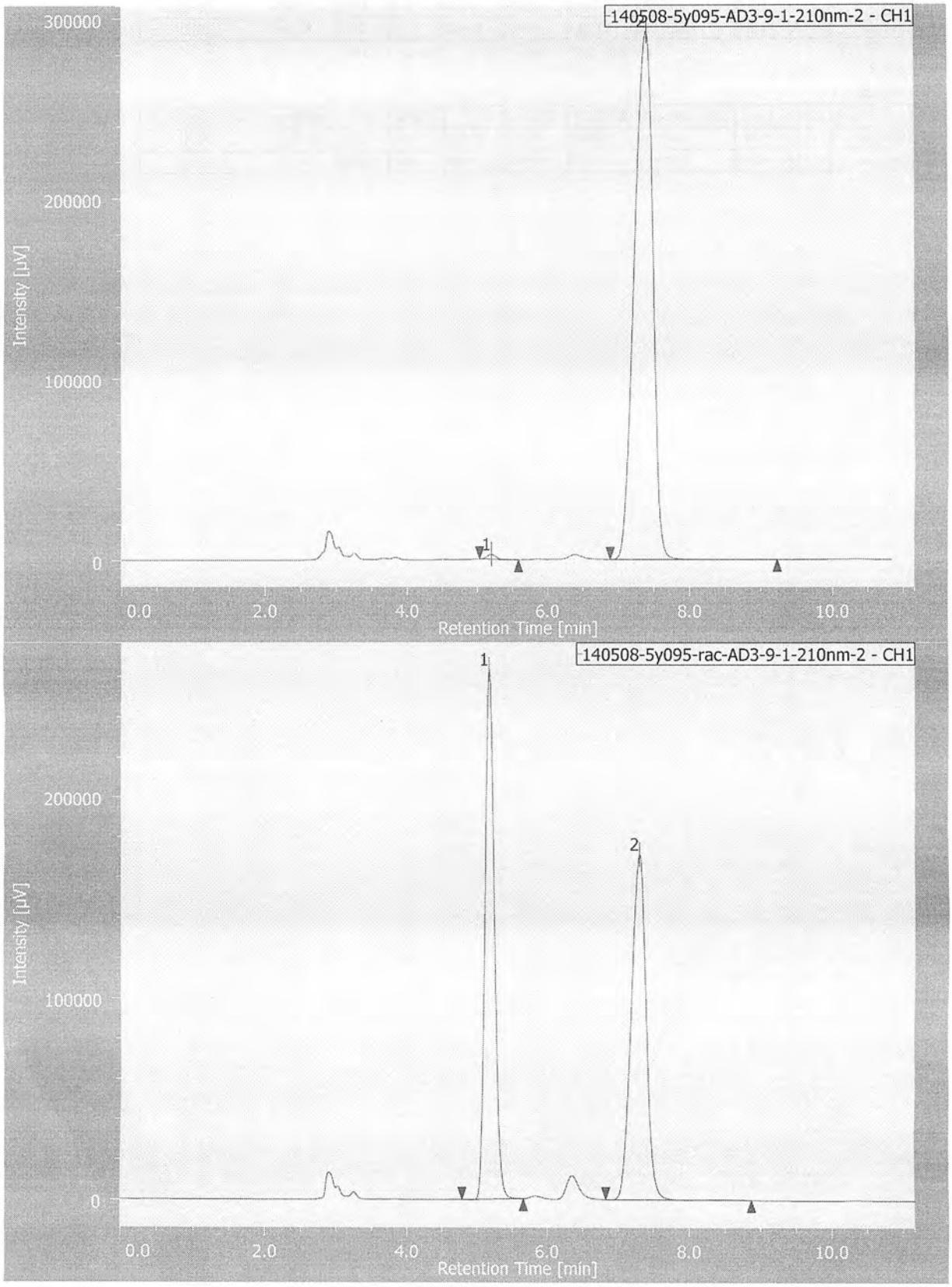
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140507
Time 11.49
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 288.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678343 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
173.460
138.292
129.497
128.778
126.841
77.411
77.157
76.902
57.054
50.562
41.189
28.789
H2N
H
N
t-Bu
O
2r
(Scheme2_from
PivLValGlyLPheNHtBu)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR

HPLC



チャンネル情報＋ピーク情報

クロマトグラム名 140508-5y095-AD3-9-1-210nm-2-CH1

サンプル名

チャンネル名 CH1

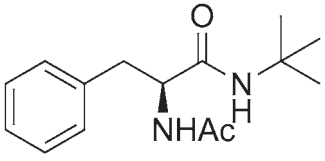
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.200	20836	2581	0.396	0.878	N/A	8672	6.148	1.016	
2	Unknown	1	7.400	5238160	291554	99.604	99.122	N/A	3587	N/A	0.812	

クロマトグラム名 140508-5y095-rac-AD3-9-1-210nm-2-CH1

サンプル名

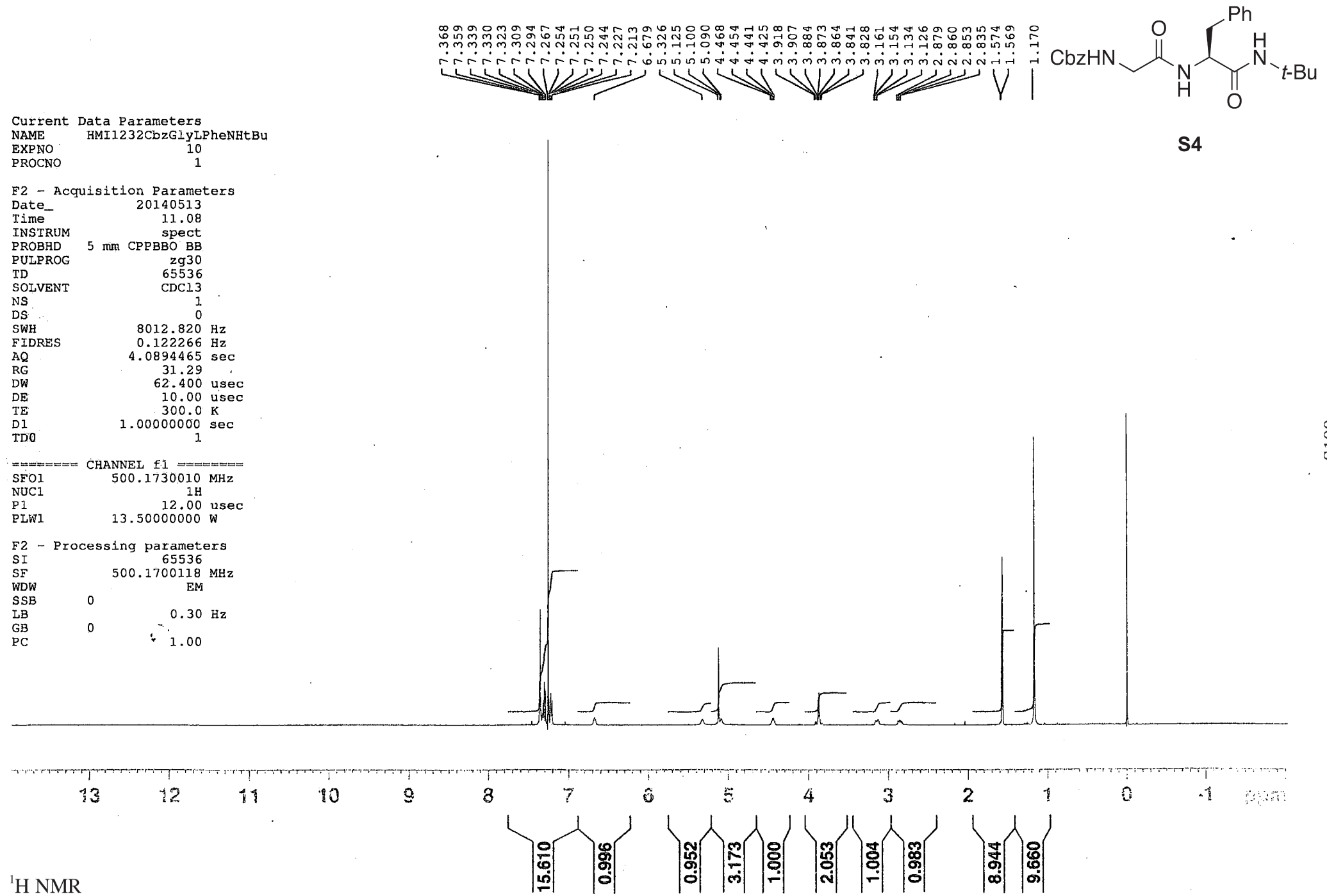
チャンネル名 CH1

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	5.167	2440794	262702	49.615	60.596	N/A	7152	6.788	1.221	
2	Unknown	1	7.292	2478653	170829	50.385	39.404	N/A	5788	N/A	0.979	



3

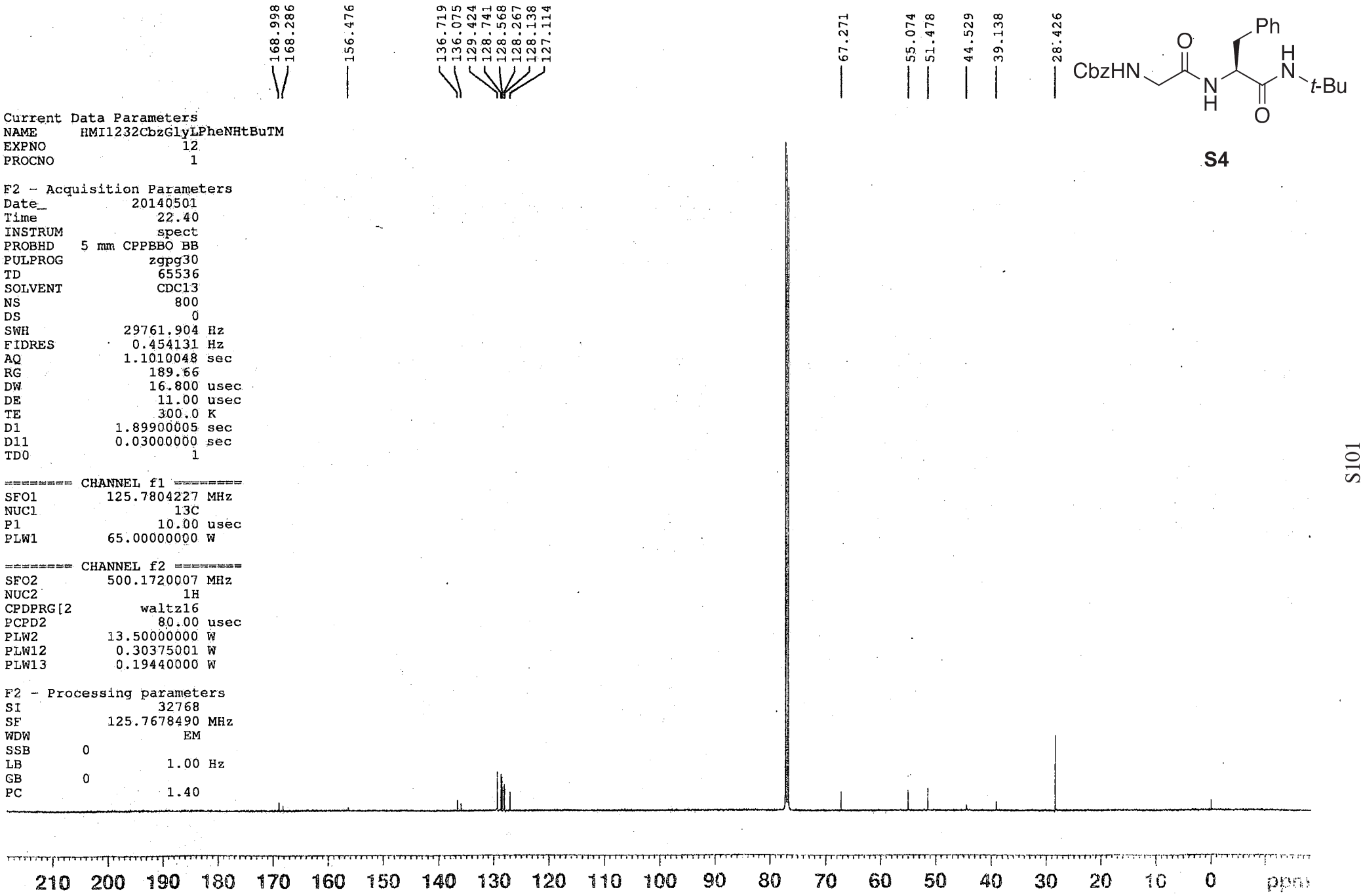
(Scheme2_from PivLValGlyLPheNHtBu)



S4

S100

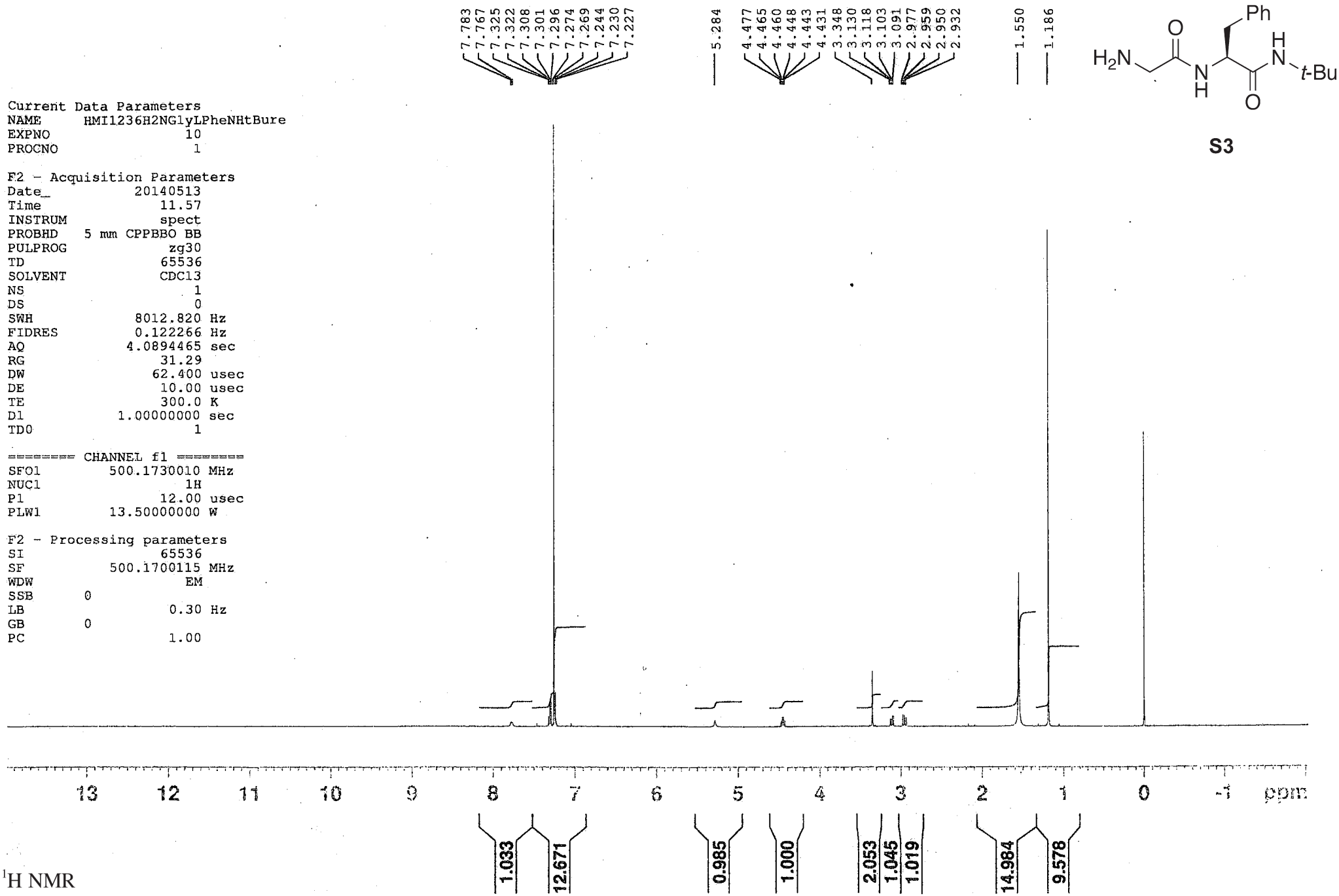
^{1}H NMR



S4

S101

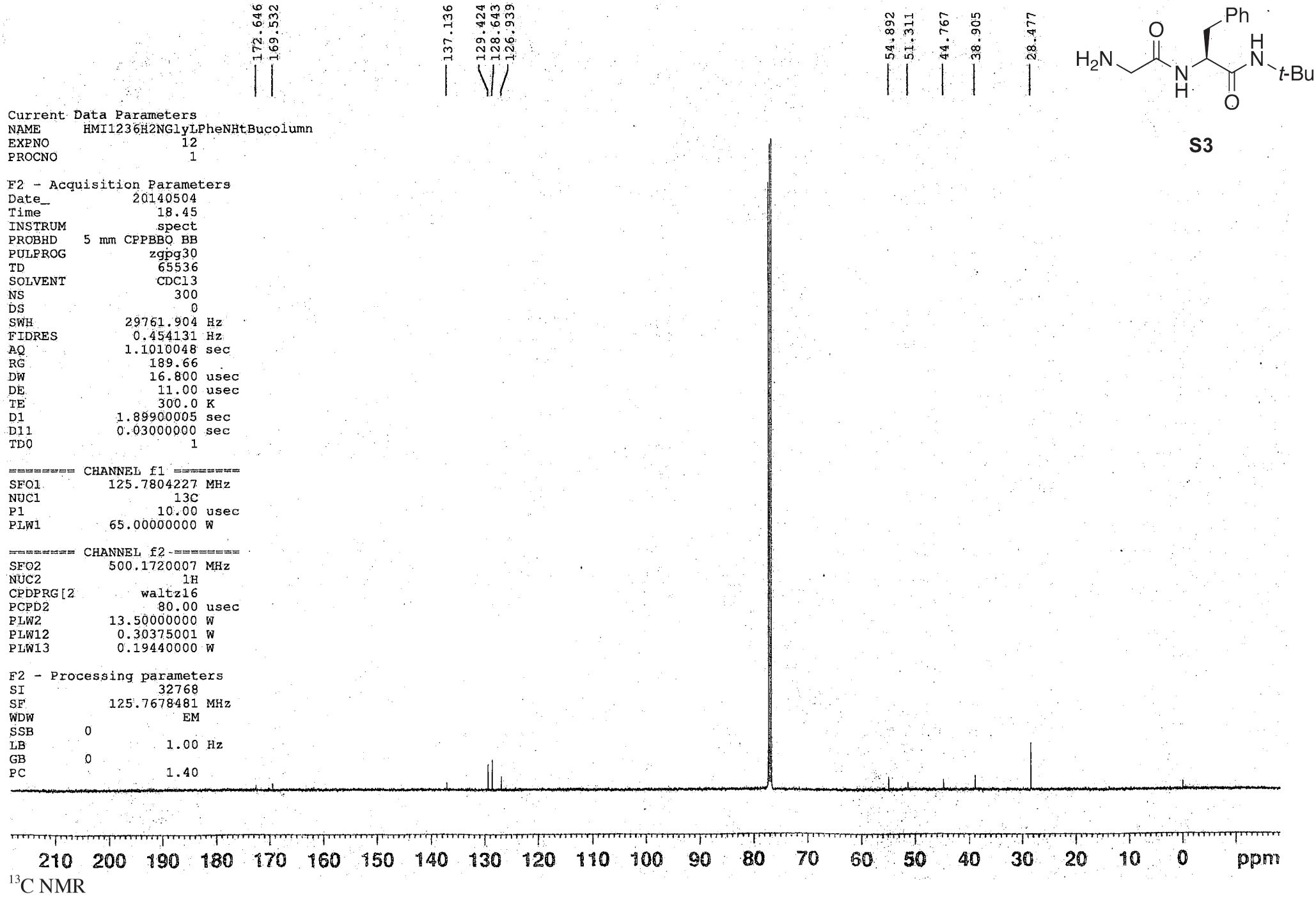
^{13}C NMR



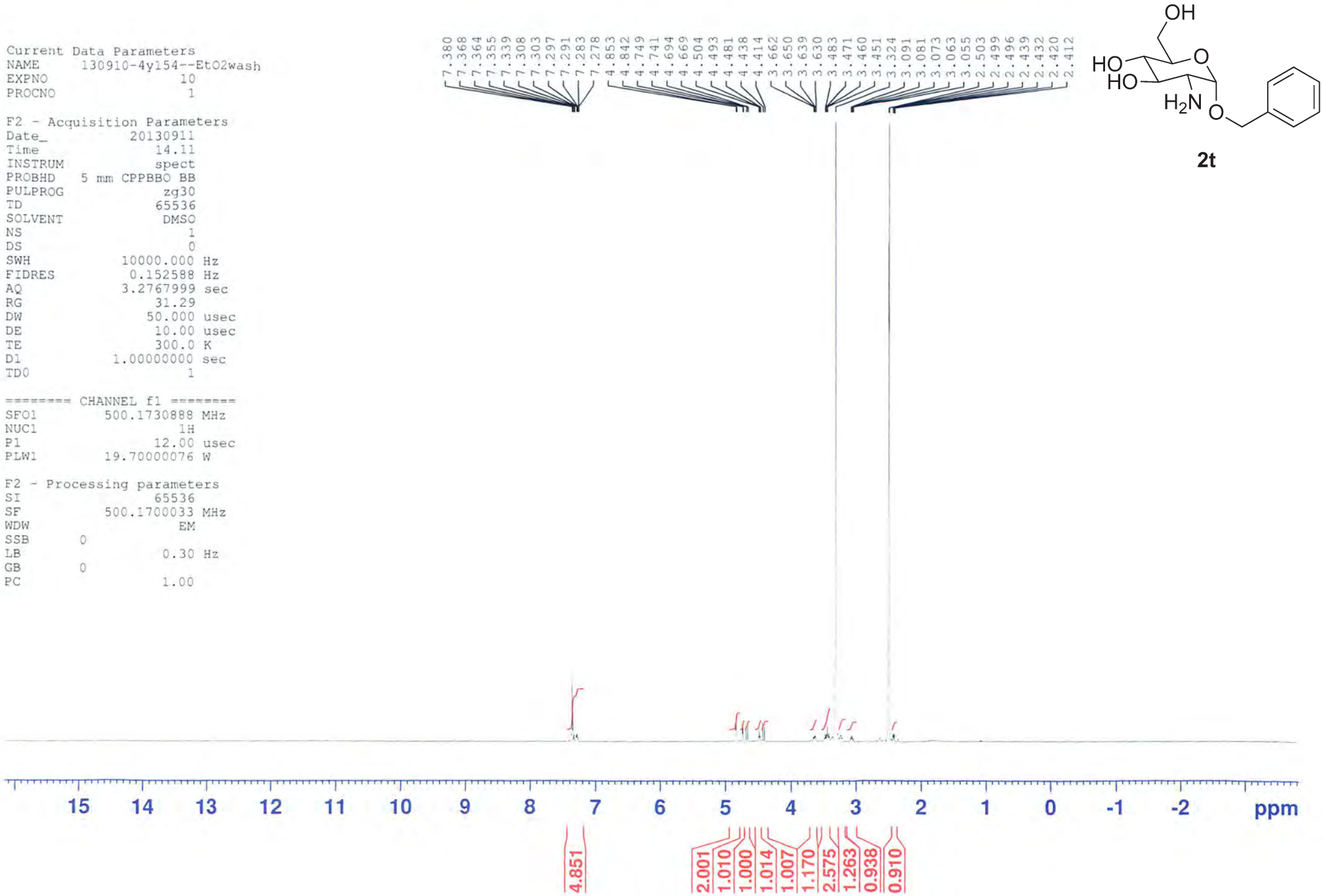
S3

S102

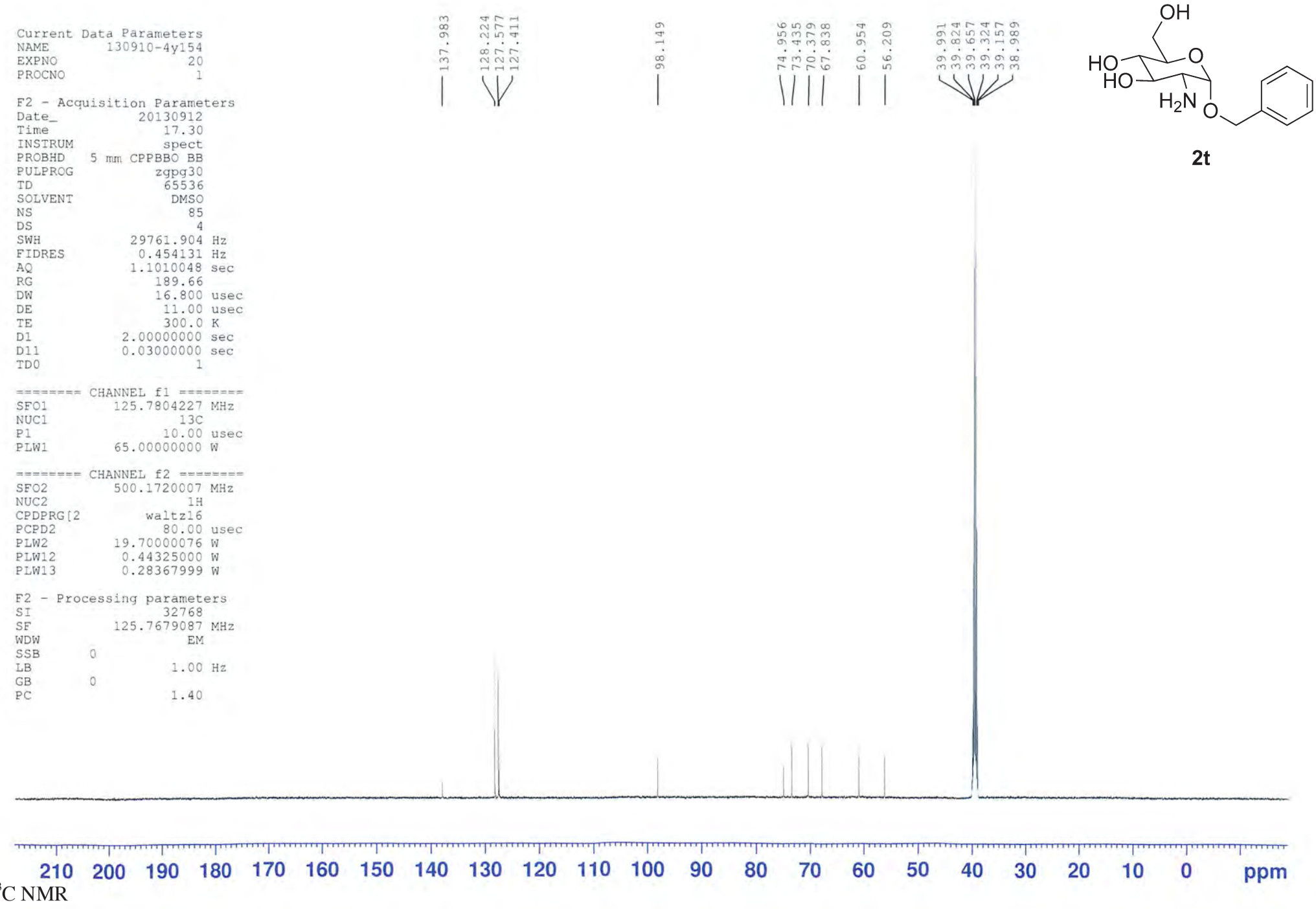
^{1}H NMR


172.646
169.532
137.136
129.424
128.643
126.939
54.892
51.311
44.767
38.905
28.477
Ph
O
H
N
H2N
N
t-Bu
H
O
S3
Current Data Parameters
NAME HMI1236H2NGlyLPheNHtBucolumn
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140504
Time 18.45
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 1.89900005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.30375001 W
PLW13 0.19440000 W
F2 - Processing parameters
SI 32768
SF 125.7678481 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR


Current Data Parameters
NAME 130910-4y154--EtO2wash
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130911
Time 14.11
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 31.29
DW 50.000 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1730888 MHz
NUC1 1H
P1 12.00 usec
PLW1 19.70000076 W
F2 - Processing parameters
SI 65536
SF 500.1700033 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.380
7.368
7.364
7.355
7.339
7.308
7.303
7.297
7.291
7.283
7.278
4.853
4.842
4.749
4.741
4.694
4.669
4.504
4.493
4.481
4.438
4.414
3.662
3.650
3.639
3.630
3.483
3.471
3.460
3.451
3.324
3.091
3.081
3.073
3.063
3.055
2.503
2.499
2.496
2.439
2.432
2.420
2.412
OH
HO
HO
O
H_2N
O
2t
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
4.851
2.001
1.010
1.000
1.014
1.007
1.170
2.575
1.263
0.938
0.910

^{1}H NMR


Current Data Parameters
NAME 130910-4y154
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130912
Time 17.30
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 85
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 189.66
DW 16.800 usec
DE 11.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7804227 MHz
NUC1 13C
P1 10.00 usec
PLW1 65.00000000 W
======== CHANNEL f2 ========
SFO2 500.1720007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 19.70000076 W
PLW12 0.44325000 W
PLW13 0.28367999 W
F2 - Processing parameters
SI 32768
SF 125.7679087 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
137.983
128.224
127.577
127.411
98.149
74.956
73.435
70.379
67.838
60.954
56.209
39.991
39.824
39.657
39.324
39.157
38.989
OH
HO
HO
O
H_2N
O
2t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{13}C NMR