Total Synthesis of Largamide B**

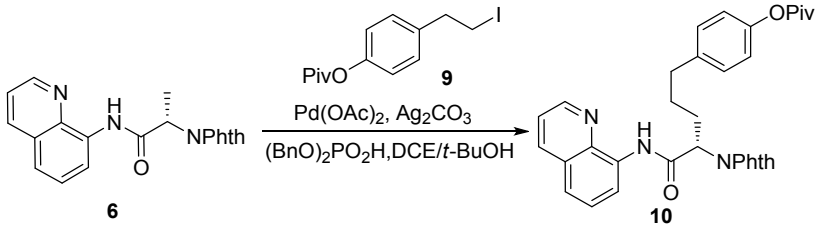
Shiwei Qu, Ying Chen, Xiaoji Wang, Shipeng Chen, Zhengshuang Xu, and Tao Ye**

Index

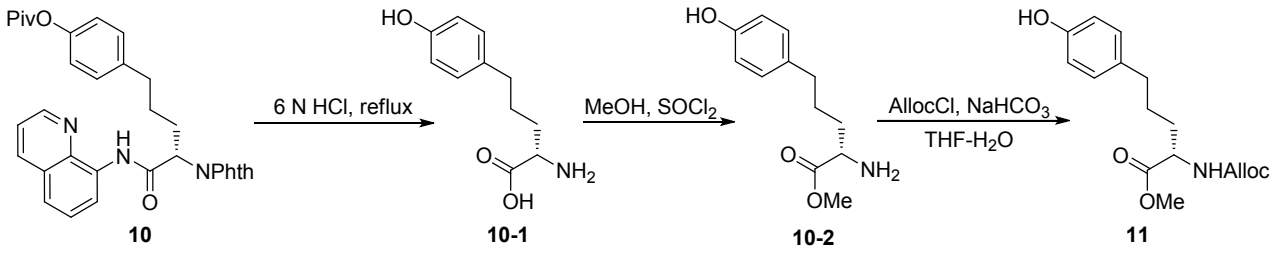
General Experimental

Non-aqueous reactions were carried out under inert atmosphere (nitrogen or argon) with rigid exclusion of moisture from reagents in oven-dried reaction vessels. Solvents were distilled prior to use: THF (tetrahydrofuran) from Na/benzophenone, MeOH (methanol) from Mg/I_2, DCM (dichloromethane), DMF (dimethylformamide), DEA (diethylamine), TEA (triethylamine) and DIPEA (diisoproylethylamine) from CaH_2. Flash column chromatography was performed using the indicated solvents on E. Qingdao silica gel 60 (230 – 400 mesh ASTM). TLC was carried out using pre-coated sheets (Qingdao silica gel 60-F250, 0.2 mm) which, after development, were visualized at 254 nm, and/or staining in *p*-anisole, ninhydrin or phosphomolybdic acid solution followed by heating. NMR spectra were recorded on Bruker DPX 300 MHz, Avance 400 MHz or AV 500 MHz spectrometers. Chemical shifts were reported in parts per million (ppm), relative to either a tetramethylsilane (TMS) internal standard or the signals due to the solvent. Data were reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad) and coupling constants (Hz). Low- and high- resolution EI and ESI mass spectra were obtained using a AB QSTAR Elite mass spectrometer. Optical rotations were recorded on a Rudolph AutoPol I Polarimeter.

Experimental Procedures



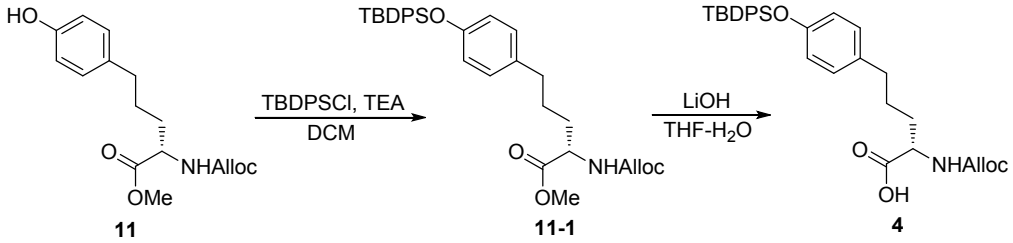
N-phthaloyl-alanine 8-aminoquinoline amide (**6**) (28 mg, 0.08 mmol), alkyl iodide (**9**) (80 mg, 0.24 mmol), $Pd(OAc)_2$ (1.8 mg, 0.008 mmol), Ag_2CO_3 (33mg, 0.12 mmol), $(BnO)_2POOH$ (11mg, 0.04 mmol) and DCE/t-BuOH (1.5/0.75 mL) were added to a 25 mL round bottom flask. The flask was then charged with Argon. The mixture was stirred at 80 °C for 12 h under Argon. After being cooled to room temperature, the reaction was diluted with dichloromethane (5 mL) and then filtered through a pad of Celite. Volatiles were removed *in vacuo*. and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:3) to afford **10** (29 mg, 66%) as a colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 1:3). $[\alpha]_D^{20}$ = -5.4 (*c* 1.5, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.35 (s, 1H), 8.76 – 8.67 (m, 2H), 8.15 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.91 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.54 – 7.50 (m, 2H), 7.43 (dd, *J* = 8.2, 4.2 Hz, 1H), 7.19 (d, *J* = 8.5 Hz, 2H), 6.95 (d, *J* = 8.4 Hz,, 2H), 5.18 (dd, *J* = 10.9, 5.4 Hz, 1H), 2.83 – 2.59 (m, 3H), 2.52 – 2.40 (m, 1H), 1.81 – 1.70 (m, 2H), 1.34 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 177.18, 168.11, 166.88, 149.28, 148.28, 138.90, 138.38, 136.46, 134.29, 133.84, 131.81, 129.27, 127.90, 127.37, 123.68, 121.97, 121.62, 121.33, 116.91, 54.95, 39.03, 34.61, 28.54, 28.34, 27.15 ppm. **HRMS** (*m/z*) calculated for $C_{33}H_{32}N_3O_5^+$ $[M + H]^+$: 550.2336, found: 550.2333.



Compound **10** (200 mg, 0.36 mmol) was dissolved in 10 mL of aqueous HCl (6 N) and refluxed for 20 h and then cooled to room temperature. The reaction mixture was concentrated *in vacuo* to afford **10-1**, which was used in next step of reaction without further purification.

To a solution of compound **10-1** in dry MeOH (10 mL), $SOCl_2$ (0.3 mL, 3.6 mmol) was added at 0 °C. The reaction mixture was refluxed for 6 h and then concentrated *in vacuo* to afford **10-2**, which was used in next step of reaction without further purification.

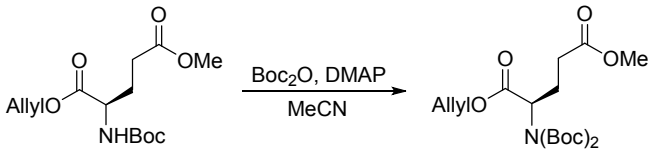
To a solution of the **10-2** in THF/H_2O (10 mL, 1:1) was added $NaHCO_3$ (306 mg, 3.6 mmol) and AllocCl (65 μL, 0.62 mmol) at 0 °C. The reaction mixture was stirred at room temperature for 12 h. The reaction was diluted with ethyl acetate (50 mL), washed with saturated NH_4Cl (aq.) (20 mL) and brine (20 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford **11** (96 mg, 86% 3 steps) as a colorless oil. **TLC**: R_f = 0.4 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = +18.9 (*c* 2.5, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.97 (d, *J* = 8.5 Hz, 2H), 6.75 (d, *J* = 8.5 Hz, 2H), 5.93 – 5.86 (m, 1H), 5.39 (d, *J* = 8.3 Hz, 1H), 5.30 (d, *J* = 17.2 Hz, 1H), 5.21 (dd, *J* = 10.4, 1.2 Hz, 1H), 4.57 (d, *J* = 5.5 Hz, 2H), 4.38 (t, *J* = 6.9 Hz, 1H), 3.72 (s, 3H), 2.60 – 2.47 (m, 2H), 1.89 – 1.78 (m, 1H), 1.72 – 1.56 (m, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.20, 156.10, 154.27, 133.19, 132.43, 129.35, 118.00, 115.29, 66.04, 53.79, 52.45, 34.35, 32.10, 27.26 ppm. **HRMS** (*m/z*) calculated for $C_{16}H_{21}NO_5Na^+$ [M + Na]$^+$: 330.1312, found: 330.1313.



To a solution of compound **11** (1.029 g, 3.35 mmol) in dry DCM (30 mL), TEA (2.4 mL, 16.74 mmol) was added, followed by addition of *t*-Butyl-diphenylsilyl chloride (1.7 mL, 6.70 mmol) in one portion at 0°C. After being stirred for 12 h at room temperature, the reaction mixture was diluted with ethyl ether (80 mL), washed successively with H_2O (20 mL), saturated NH_4Cl (aq.) (2 × 20mL) and brine (20 mL), dried over Na_2SO_4 (s) and concentrated *in vacuo*. The crude residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:5) to give rise to **11-1** (1.74 g, 95%) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:2). $[\alpha]_D^{20}$ = +8.9 (*c* 2.0, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (dd, *J* = 7.9, 1.4 Hz, 4H), 7.46 – 7.36 (m, 6H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.70 (d, *J* = 8.4 Hz, 2H), 5.93 – 5.86 (m, 1H), 5.32 (d, *J* = 17.2 Hz, 1H), 5.22 (d, *J* = 10.0 Hz, 2H), 4.58 (d, *J* = 5.6 Hz, 2H), 4.38 (d, *J* = 6.5 Hz, 1H), 3.72 (s, 3H), 2.59 – 2.43 (m, 2H), 1.82 (t, *J* = 11.4 Hz, 1H), 1.69 – 1.53 (m, 3H), 1.12 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.01, 155.75, 153.76, 135.56, 133.96, 133.14, 132.65, 129.84, 129.01, 127.73, 119.53, 117.83, 65.83, 53.70, 52.32, 34.41, 32.17, 27.07, 26.58, 19.47 ppm. **HRMS** (*m/z*): calculated for $C_{32}H_{40}NO_5Si^+$ [M + H]$^+$: 546.2670, found: 546.2666.

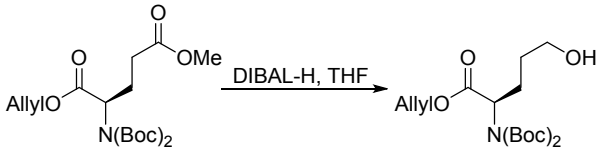
To a solution of compound **11-1** (426 mg, 0.781 mmol) in THF/H_2O (10 mL, 1/1) at 0 °C, $LiOH{\cdot}H_2O$ (164 mg, 3.9 mmol) was added. The reaction mixture was stirred at room temperature for 2 h. Volatiles were

evaporated *in vacuo*. The aqueous layer was washed with diethyl ether (5 mL), then acidified to pH = 3 with 1 N HCl and extracted with ethyl acetate (3 × 30 mL). The combined organic layers were washed successively with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* to provide **4** (395 mg, 95%) as a colorless oil, which was used in next step of reaction without further purification.



12 **12-1**

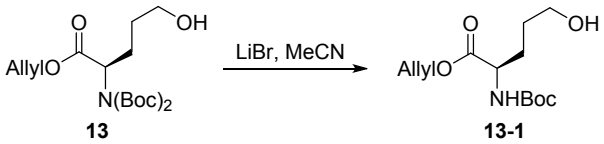
To a solution of compound **12** (678 mg, 2.25 mmol) and DMAP (56 mg, 0.45 mmol) in acetonitrile (9 mL), a solution of Boc_2O (1.06 mL, 4.95 mmol) in acetonitrile (3 mL) was added. The resulting mixture was stirred at room temperature for 22 h. Volatiles were evaporated under reduced pressure. The residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:6) to give **12-1** (845 mg, 94%) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:4). $[\alpha]_D^{20}$ = +27.8 (*c* 2.0, $CHCl_3$). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.92 – 5.78 (m, 1H), 5.32 – 5.23 (m, 1H), 5.17 (dd, *J* = 10.5, 1.3 Hz, 1H), 4.91 (dd, *J* = 9.6, 4.9 Hz, 1H), 4.57 (d, *J* = 5.5 Hz, 2H), 3.63 (s, 3H), 2.49 – 2.42 (m, 1H), 2.41 – 2.33 (m, 2H), 2.21 – 2.13 (m, 1H), 1.45 (s, 18H) ppm. **^{13}C NMR** (125 MHz, $CDCl_3$) δ 173.06, 170.00, 152.01, 131.80, 118.09, 83.24, 65.72, 57.49, 51.59, 30.66, 27.96, 27.92, 25.04 ppm. **HRMS** (*m/z*): calculated for $C_{19}H_{32}NO_8^+$ $[M + H]^+$: 402.2122, found: 402.2120.



12-1 **13**

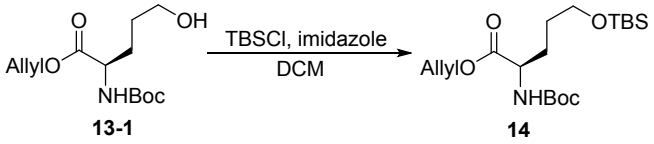
To a solution of **12-1** (584 mg, 1.45 mmol) in THF (15 mL), DIBAL-H (2.9 mL, 4.365 mmol, 1.5 M in toluene) was added at -78 ºC. The resulting mixture was stirred at -50 ºC for 4 h and then quenched with methanol (2 mL). Aqueous solution of Rochelle's salt (50 mL) was added, and the solution was stirred vigorously for 6 h. The reaction mixture was stirred at room temperature for additional 2 h. Volatiles were evaporated under reduced pressure and the aqueous layer was extracted with ethyl acetate (2 × 50 mL). The combined organic extracts were washed successively with H_2O (20 mL), saturated $NaHCO_3$ (aq.) (20 mL) and brine (20 mL), dried over anhydrous Na_2SO_4(s) and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:4) to provide **13** (454 mg, 84%) as a

colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 1:4). $[\alpha]_D^{20}$ = +24.0 (*c* 1.0, $CHCl_3$). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.95 – 5.84 (m, 1H), 5.34 – 5.28 (m, 1H), 5.21 (dd, *J* = 10.5, 1.2 Hz, 1H), 4.89 (dd, *J* = 9.1, 5.4 Hz, 1H), 4.61 (d, *J* = 5.5 Hz, 2H), 3.67 (dd, *J* = 11.9, 6.0 Hz, 2H), 2.28 – 2.20 (m, 1H), 1.98 – 1.91 (m, 1H), 1.67 – 1.60 (m, 3H), 1.49 (s, 18H) ppm. **^{13}C NMR** (125 MHz, $CDCl_3$) δ 170.57, 152.28, 131.88, 118.07, 83.17, 65.72, 62.32, 58.02, 29.44, 28.01, 26.26 ppm. **HRMS** (*m/z*): calculated for $C_{18}H_{32}NO_7^+$ $[M + H]^+$: 374.2173, found: 374.2170.



13 **13-1**

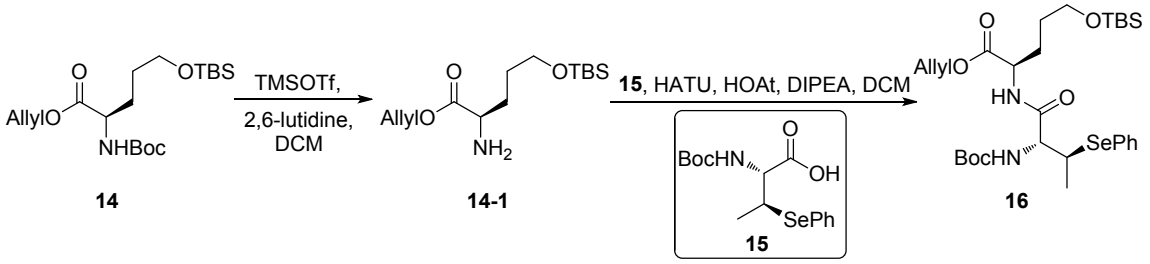
To a solution of compound **13** (1.0 g, 2.68 mmol) in acetonitrile (50 mL), LiBr (698 mg, 8.03 mmol) was added in one portion. The resulting solution was stirred at 70 ºC for 6 h. After removal of the solvent under reduced pressure, the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to give **13-1** (658 mg, 90%) as a colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 1:2). $[\alpha]_D^{20}$ = -1.0 (*c* 3.0, $CHCl_3$). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.94 – 5.86 (m, 1H), 5.32 (dd, *J* = 17.2, 1.2 Hz, 1H), 5.24 (dd, *J* = 10.5, 0.7 Hz, 1H), 5.21 (d, *J* = 6.5 Hz, 1H), 4.67 – 4.58 (m, 2H), 4.34 (d, *J* = 4.3 Hz, 1H), 3.66 (t, *J* = 6.0 Hz, 2H), 2.07 (s, 1H), 1.92 (dd, *J* = 13.2, 6.5 Hz, 1H), 1.78 – 1.70 (m, 1H), 1.67 – 1.59 (m, 2H), 1.43 (s, 9H) ppm. **^{13}C NMR** (125 MHz, $CDCl_3$) δ 172.48, 155.56, 131.66, 118.81, 79.98, 65.85, 62.04, 53.25, 29.46, 28.33 ppm. **HRMS** (*m/z*): calculated for $C_{13}H_{23}NO_5Na^+$ $[M + Na]^+$: 296.1468, found: 296.1467.



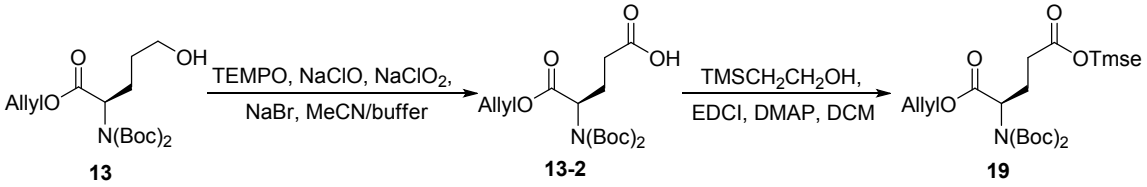
13-1 **14**

To a solution of **13-1** (658 mg, 2.41 mmol) in dry DCM (20 mL), Imidazole (656 mg, 9.64 mmol) was added, followed by the addition of *t*-Butyl-dimethylsilyl chloride (726 mg, 4.82 mmol) in one portion at 0°C. The reaction mixture was stirred for 12 h at room temperature. The reaction mixture was then diluted with ethyl acetate (60 mL), and the organic solution was washed successively with H_2O (20 mL), saturated NH_4Cl (aq.) (20mL) and brine (20 mL), dried over Na_2SO_4 (s) and concentrated under reduced pressure. The crude residue was purified by flash chromatography (silica gel, EtOAc/hexane, 1:5) to give rise to **14** (756 mg, 81%) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:4). $[\alpha]_D^{20}$ = +0.5 (*c* 3.0, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.93 – 5.83 (m 1H), 5.34 – 5.26 (m, 1H), 5.27 – 5.15 (m, 2H), 4.63 – 4.57 (m, 2H), 4.29 (dd, *J* = 12.7, 7.7 Hz, 1H), 3.60 (t, *J* = 6.1 Hz, 2H), 1.93 – 1.82 (m, 1H), 1.76 – 1.67 (m, 1H), 1.60

– 1.51 (m, 2H), 1.42 (s, 9H), 0.87 (s, 9H), 0.03 (s, 6H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.54, 155.42, 131.70, 118.58, 79.69, 65.69, 62.27, 53.30, 29.05, 28.41, 28.31, 25.91, 18.28, -5.37 ppm. **HRMS** (*m/z*): calculated for $C_{19}H_{38}NO_5Si^+$ $[M + H]^+$: 388.2514, found: 388.2516.

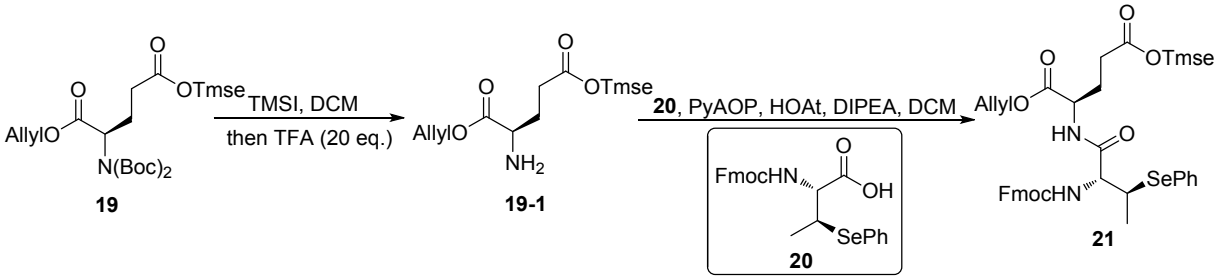


To a solution of compound **14** (174 mg, 0.45 mmol) in dry DCM (5 mL), 2,6-lutidine (0.32 mL, 2.7 mmol) and TMSOTf (0.24 mL, 1.35 mmol) were added at 0 °C. After being stirred at room temperature for 3 h, the reaction mixture was allowed to cool to 0 °C and quenched by addition of saturated $NaHCO_3$ (aq.) (5 mL). Layers were separated, and the aqueous layer was extracted with ethyl acetate (3 × 10 mL). The combined organic layers were washed with brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* to afford **14-1**, which was used in the next step of reaction without further purification. To a solution of the carboxylic acid **15** (161 mg, 0.45 mmol) and **14-1** in dry DCM (6 mL), HOAt (122 mg, 0.9 mmol), HATU (342 mg, 0.9 mmol) and DIPEA (0.47 mL, 2.7 mmol) were added successively at 0°C. After being warmed to room temperature and stirred for additional 12 h, the reaction mixture was quenched with H_2O (1 mL) and diluted with ethyl acetate (50 mL). The organic solution was washed successively with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane =1:4) to afford dipeptide **16** (225 mg, 80% 2 steps) as a colorless oil. **TLC**: R_f = 0.4 (silica gel, EtOAc/hexane = 1:4). $[\alpha]_D^{20}$ = -32.8 (*c* 1.0, $CHCl_3$). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.61 – 7.55 (m, 2H), 7.33 – 7.27 (m, 3H), 6.72 (d, *J* = 8.0 Hz, 1H), 5.95 – 5.85 (m, 1H), 5.38 – 5.29 (m, 1H), 5.25 (dd, *J* = 10.4, 1.1 Hz, 2H), 4.68 – 4.56 (m, 3H), 4.19 (s, 1H), 3.73 (s, 1H), 3.60 (t, *J* = 6.1 Hz, 2H), 2.02 – 1.91 (m, 1H), 1.80 – 1.70 (m, 1H), 1.58 – 1.51 (m, 2H), 1.48 – 1.41 (m, 12H), 0.88 (s, 9H), 0.03 (d, *J* = 2.2 Hz, 6H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.79, 169.97, 155.63, 135.01, 131.51, 129.22, 128.69, 128.07, 118.92, 80.64, 65.97, 62.08, 58.36, 51.98, 40.75, 28.80, 28.37, 28.25, 25.93, 18.29, 17.77, -5.35, -5.36 ppm. **HRMS** (*m/z*): calculated for $C_{29}H_{49}N_2O_6SeSi^+$ $[M + H]^+$: 629.2520, found: 629.2516.



Compound **13** (433 mg, 1.16 mmol) was dissolved in MeCN (10 mL) and aqueous solution of NaH_2PO_4-Na_2HPO_4 (10 mL, pH = 6.7). To this solution, 2,2,6,6-tetramethylpiperidin-1-oxyl (9 mg, 0.058 mmol), sodium chlorite (535 mg, 5.8 mmol), NaBr (119 mg, 1.16 mmol), and a 10% Bleach solution (0.87 mL) were added sequentially at room temperature. After being stirred at room temperature for 1 h, the reaction mixture was acidified to pH = 1-2 with 1 M HCl (aq.) at 0°C. The aqueous layer was extracted with ethyl acetate (2 × 50 mL). The combined organic layers were washed with brine (10 mL) and finally dried over anhydrous Na_2SO_4 (s). The solvent was concentrated *in vacuo* to afford **13-2**, which was used in the next step without further purification.

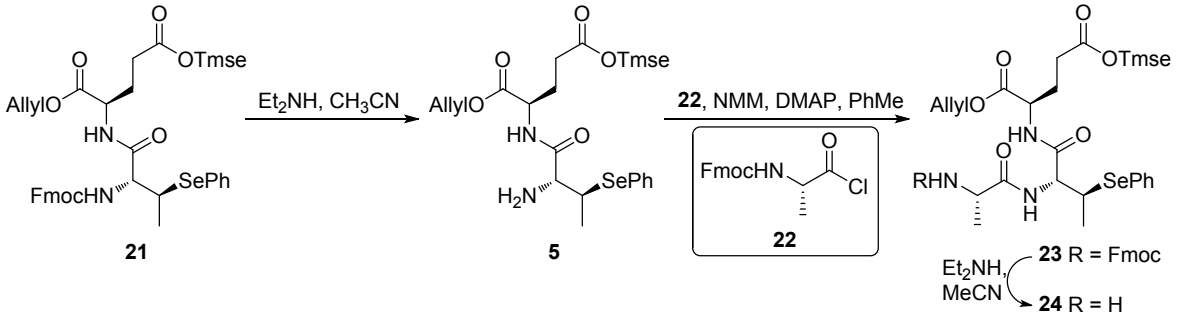
To a solution of the **13-2** and 2-(trimethylsilyl)-ethanol (248 µL, 1.74 mmol) in dry DCM (10 mL), DMAP (425 mg, 3.48 mmol) and EDCI (665 mg, 3.48 mmol) were successively added at 0 °C. After being warmed to room temperature and stirred for additional 12 h, the reaction mixture was quenched with H_2O (5 mL), diluted with ethyl acetate (100 mL), washed successively with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:6) to afford **19** (398 mg, 70% 2 steps) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:4). $[\alpha]_D^{20}$ = +27.0 (*c* 0.5, $CHCl_3$). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.95 – 5.85 (m, 1H), 5.32 (dq, *J* = 17.2, 1.5 Hz, 1H), 5.22 (ddd, *J* = 10.5, 2.6, 1.3 Hz, 1H), 4.96 (dd, *J* = 9.5, 5.2 Hz, 1H), 4.62 (d, *J* = 5.5 Hz, 2H), 4.20 – 4.15 (m, 2H), 2.53 – 2.46 (m, 1H), 2.43 – 2.34 (m, 2H), 2.22 – 2.15 (m, 1H), 1.50 (s, 18H), 1.03 – 0.95 (m, 2H), 0.04 (s, 9H) ppm. **^{13}C NMR** (125 MHz, $CDCl_3$): δ 172.75, 170.06, 152.08, 131.86, 118.07, 83.20, 65.71, 62.65, 57.57, 31.11, 28.00, 25.13, 17.38, -1.52 ppm. **HRMS** (*m/z*): calculated for $C_{23}H_{42}NO_8Si^+$ $[M + H]^+$: 488.2674, found: 488.2664.



To a solution of compound **19** (163 mg, 0.335 mmol) in dry DCM (3 mL), Iodotrimethylsilane (53 µL, 0.369 mmol) was added at 0 °C. The reaction mixture was stirred at 0 °C and folllowed by TLC. The first Boc

protecting group was removed (approx. 15 minutes), and the reaction was quenched by the addition of MeOH (68 μL) at 0 °C. Trifluoroacetic acid (260 μL, 3.35 mmol) was added to the solution. After being stirred at room temperature for 1 h, additional trifluoroacetic acid (260 μL, 3.35 mmol) was added to the reaction mixture, which was stirred for additional 1 h. Volatiles were removed *in vacuo*. The residue was dried under high vacuum for 2 h to afford amine **19-1**, which was used in the next step of reaction without further purification.

To the solution of **20** (340 mg, 0.709 mmol) in DCM (6 mL), HOAt (95 mg, 0.709 mmol), PyAOP (1.1 g, 2.227 mmol) and DIPEA (0.74 mL, 4.254 mmol) were added successively at 0 °C. To this mixture, a solution of amine **19-1** in DCM (2 mL) was added. After being stirred at 0 °C for 4 h, the reaction mixture was warmed to room temperature and stirred for additional 12 h. The reaction was quenched with H_2O (1 mL), diluted with ethyl acetate (50 mL). The organic solution was washed successively with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane =1:4) to afford dipeptide **21** (181 mg, 72% 2 steps) as a colorless oil. **TLC**: R_f = 0.4 (silica gel, EtOAc/hexane = 1:2). $[\alpha]_D^{20}$ = -4.4 (*c* 1.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.59 (dd, *J* = 7.2, 2.1 Hz, 4H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.34 – 7.26 (m, 5H), 7.02 (d, *J* = 7.6 Hz, 1H), 5.92 – 5.85 (m, 1H), 5.59 (s, 1H), 5.36-5.28 (m, 1H), 5.25 (dd, *J* = 10.4, 1.1 Hz, 1H), 4.65 – 4.57 (m, 3H), 4.40 (dd, *J* = 15.8, 8.7 Hz, 3H), 4.25 (s, 1H), 4.18 – 4.08 (m, 2H), 3.71 (d, *J* = 5.5 Hz, 1H), 2.41 (dd, *J* = 15.1, 7.6 Hz, 2H), 2.22 (dd, *J* = 13.4, 6.0 Hz, 1H), 2.07 – 1.96 (m, 1H), 1.46 (d, *J* = 6.9 Hz, 3H), 0.92 (dd, *J* = 13.8, 7.5 Hz, 2H), 0.01 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.96, 171.09, 169.88, 156.24, 143.77, 143.70, 141.32, 141.30, 135.02, 131.37, 129.26, 128.61, 128.12, 127.78, 127.14, 125.15, 120.02, 119.15, 67.53, 66.24, 63.07, 58.88, 52.07, 47.10, 40.93, 30.44, 29.70, 26.93, 17.93, 17.24, -1.52 ppm. **HRMS** (*m/z*): calculated for $C_{38}H_{47}N_2O_7SeSi^+$ $[M + H]^+$: 751.2312, found: 751.2306.

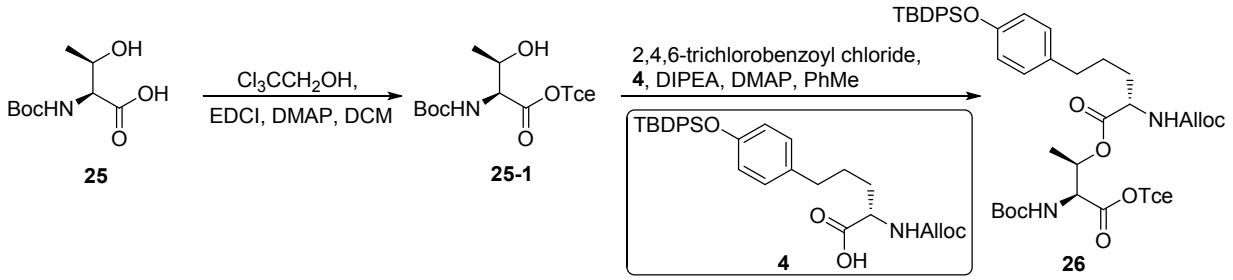


To a solution of compound **21** (93 mg, 0.124 mmol) in dry MeCN (2 mL), diethylamine (1 mL) was added at

0 °C. After being stirred at 0 °C for 30 min, volatiles were removed *in vacuo*. The residue was dried under high vacuum for 2 h to provide **5**, which was used in the next step of reaction without further purification.

To a solution of amine **5** in dry toluene (3 mL), *N*-methylmorpholine (0.34 mL, 3.1 mmol) and *N*-Fmoc-*L*-Alanyl Chloride **22** (in 3 ml of dry toluene) were added slowly at 0 ^{o}C. The reaction mixture was stirred at 0 ^{o}C for 15 min followed by addition of a solution of DMAP (76 mg, 0.62 mmol) in dry toluene (1 mL). After being stirred at room temperature for 4 h, the reaction mixture was quenched with MeOH (1 mL), diluted with ethyl acetate (50 mL), washed successively with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane =1:2) to afford tripeptide **23** (73 mg, 72% 2 steps) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -13.4 (*c* 0.5, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.4 Hz, 2H), 7.60 (t, *J* = 7.0 Hz, 2H), 7.52 (dd, J = 7.9, 1.5 Hz, 2H), 7.40 (t, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.26 – 7.21 (m, 3H), 6.83 (d, *J* = 7.9 Hz, 1H), 5.90 – 5.83 (m, 1H), 5.29 (dd, *J* = 17.2, 1.4 Hz, 1H), 5.25 – 5.15 (m, 2H), 4.63 (dd, *J* = 8.3, 4.5 Hz, 1H), 4.61 – 4.52 (m, 3H), 4.48 (dt, *J* = 10.6, 4.0 Hz, 2H), 4.24 (t, *J* = 6.5 Hz, 2H), 4.19 – 4.11 (m, 2H), 3.83-3.73 (m, 1H), 2.42 (dd, *J* = 16.0, 8.4 Hz, 2H), 2.22 (dt, *J* = 12.4, 7.4 Hz, 1H), 2.05 (dt, *J* = 14.4, 7.4 Hz, 1H), 1.43 (t, *J* = 8.4 Hz, 6H), 0.99-0.93 (m, 2H), 0.03 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ173.09, 172.23, 171.20, 169.54, 156.58, 143.75, 143.62, 141.36, 141.33, 134.87, 131.48, 129.47, 129.24, 128.70, 128.03, 127.80, 127.16, 127.12, 124.95, 120.04, 118.94, 67.36, 66.07, 63.03, 56.82, 52.08, 51.20, 47.08, 40.56, 30.52, 26.63, 17.69, 17.27, -1.50 ppm. **HRMS** (*m/z*): calculated for $C_{41}H_{52}N_3O_8SeSi^+$ $[M + H]^+$: 822.2683, found: 822.2681.

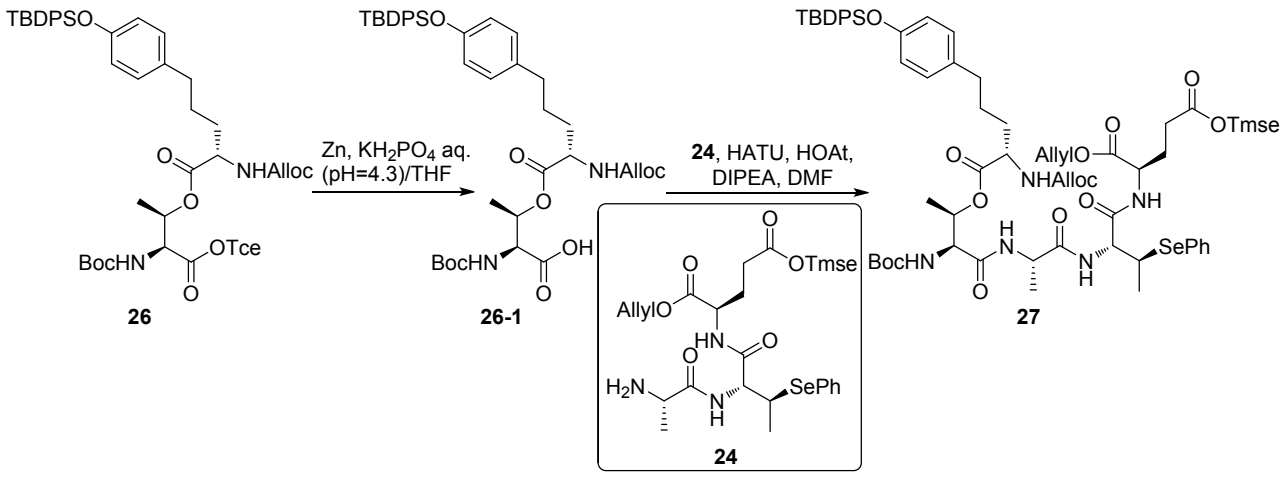
To a solution of compound **23** (53 mg, 0.0646 mmol) in dry MeCN (2 mL), diethylamine (1 mL) was added at 0 °C. After being stirred at room temperature for 30 min, volatiles in the reaction mixture were removed *in vacuo*. The residue was dried under high vacuum for 2 h to provide amine **24**, which was used in next step without further purification.



To a solution of *L*-Boc-Thr-OH (**25**, 2.94 g, 13.41 mmol) and 2,2,2-trichloroethanol (2.6 mL, 26.82 mmol) in dry DCM (30 mL), DMAP (8.2 g, 67.05 mmol) and EDCI (6.4 g, 33.50 mmol) were added successively at 0

°C. After being warmed to room temperature and stirred for 12 h, the reaction mixture was quenched with H_2O (10 mL), diluted with ethyl acetate (100 mL), washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford **25-1** (3.86 g, 82%) as a colorless oil.

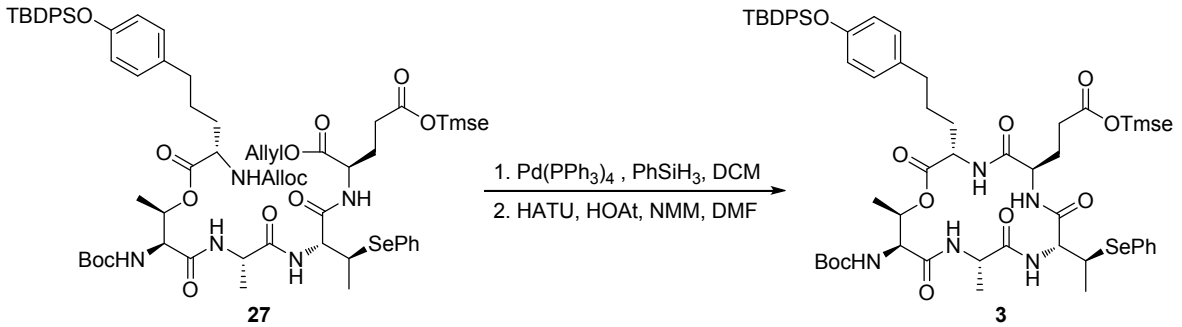
To a solution of **25-1** (410 mg, 1.17 mmol) and **4** in dry toluene (10 mL), DIPEA (1.4 mL, 7.80 mmol) and 2,4,6-trichlorobenzoyl chloride (0.37 mL, 2.34 mmol) were added at 0°C. The reaction mixture was stirred at 0 °C for 15 min followed by addition of DMAP (476 mg, 3.90 mmol) in dry toluene (2.5 mL). After being stirred at room temperature for 10 h, the reaction was quenched with MeOH (0.5 mL), diluted with ethyl acetate (50 mL). The organic solution was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:4) to afford **26** (506 mg, 75%) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:2). $[\alpha]_D^{20}$ = -1.5 (*c* 1.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 – 7.68 (m, 4H), 7.43 – 7.34 (m, 6H), 6.90 – 6.79 (m, 2H), 6.71 – 6.63 (m, 2H), 5.87 – 5.95 (m, 1H), 5.56 – 5.41 (m, 1H), 5.33 – 5.16 (m, 3H), 5.06 (d, *J* = 7.8 Hz, 1H), 4.81 – 4.77 (m, 2H), 4.66 – 4.52 (m, 3H), 4.31 – 4.17 (m, 1H), 2.57 – 2.39 (m, 2H), 1.75 (d, *J* = 12.9 Hz, 1H), 1.65 (s, 1H), 1.57 (dd, *J* = 13.7, 7.3 Hz, 2H), 1.51-1.40 (m, 9H), 1.32 (d, *J* = 6.4 Hz, 3H), 1.10 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.36, 168.55, 155.82, 155.65, 153.81, 135.53, 133.75, 133.08, 132.55, 129.84, 128.95, 127.73, 119.57, 118.03, 94.23, 80.69, 74.94, 71.50, 65.92, 57.06, 53.67, 34.42, 31.75, 28.28, 27.09, 26.55, 19.46, 16.82 ppm. HRMS(ESI) *m/z* calculated for $C_{42}H_{53}Cl_3N_2O_9Si$ $[M+H]^+$: 863.2659, found: 863.2657. **HRMS** (*m/z*): calculated for $C_{42}H_{54}Cl_3N_2O_9Si^+$ $[M + H]^+$: 863.2659, found: 863.2657.



Compound **26** (158 mg, 0.18 mmol) was dissolved in THF (2 mL) and aqueous solution of KH_2PO_4 (2 mL,

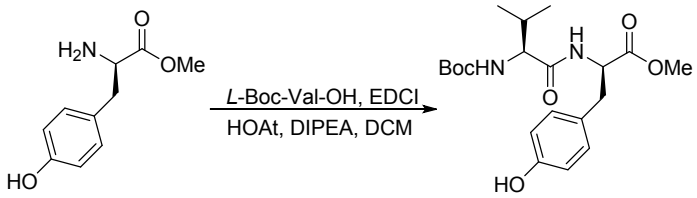
pH = 4.3). To this solution, zinc powder (cat.) was added at room temperature. After being stirred at room temperature for 10 h, the reaction mixture was filtered through a pad of Celite and washed with ethyl acetate. The aqueous layer was extracted with ethyl acetate (2 × 25 mL). The combined organic layers were washed with brine (10 mL) and dried over anhydrous Na_2SO_4 (s). The solvent was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford **26-1** (108 mg, 81%) as a colorless oil.

To a solution of **26-1** (60 mg, 0.08 mmol) and amine **24** in dry DMF (4 mL), HOAt (44 mg, 0.32 mmol), HATU (123 mg, 0.32 mmol) and DIPEA (113 μL, 0.65 mmol) were added at 0°C. After being warmed to room temperature and stirred for 12 h, the reaction mixture was quenched with H_2O (1 mL), diluted with ethyl acetate (50 mL). The organic solution was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford **27** (59 mg, 69% 2 steps) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -4.2 (*c* 0.5, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 6.7 Hz, 4H), 7.58 – 7.46 (m, 2H), 7.43 – 7.34 (m, 6H), 7.31 – 7.19 (m, 3H), 7.13 (d, *J* = 6.9 Hz, 1H), 6.90-6.76 (m, 3H), 6.66 (t, *J* = 8.0 Hz, 2H), 5.96 – 5.81 (m, 2H), 5.49 (d, *J* = 6.9 Hz, 1H), 5.37-5.19 (m, 5H), 4.65 – 4.45 (m, 6H), 4.41 – 4.30 (m, 2H), 4.21 – 4.12 (m, 3H), 3.97 – 3.86 (m, 1H), 2.55-2.35 (m, 4H), 2.22 (dt, *J* = 13.0, 8.3 Hz, 1H), 2.11-2.01 (m, 1H), 1.78 (dd, *J* = 13.7, 7.0 Hz, 1H), 1.65-1.54 (m, 3H), 1.50 – 1.38 (m, 12H), 1.32 – 1.13 (m, 6H), 1.09 (s, 9H), 1.00-0.93 (m, 2H), 0.03 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.01, 172.14, 171.51, 171.17, 169.80, 169.66, 156.32, 155.29, 153.84, 135.53, 134.73, 133.05, 132.21, 131.61, 129.84, 129.24, 128.95, 128.06, 127.72, 119.58, 118.83, 118.32, 80.66, 70.34, 66.24, 65.96, 62.89, 56.77, 56.56, 54.56, 52.21, 50.95, 39.89, 34.48, 30.96, 30.54, 29.70, 28.30, 28.24, 27.29, 26.54, 19.45, 17.27, 17.12, 15.66, -1.50 ppm. **HRMS** (*m/z*): calculated for $C_{66}H_{91}N_5O_{14}SeSi_2Na^+$ [M + Na]$^+$: 1336.5158, found: 1336.5159.



27 **3**

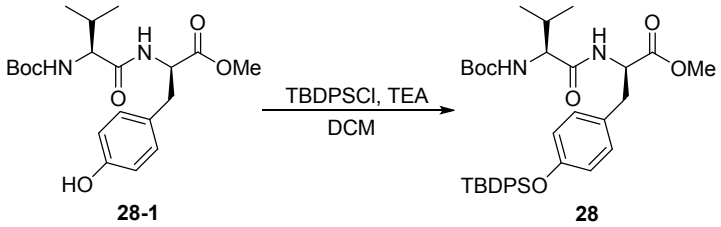
To a solution of $Pd(PPh_3)_4$ (2 mg, 0.002 mmol) in CH_2Cl_2 (1 mL) and phenylsilane (8 μL, 0.06 mmol), compound **27** 12 mg, 0.009 mmol) was added. After being stirred for 3 h at room temperature, volatiles of the reaction mixture were removed *in vacuo*. The residue was dried under high vacuum for 2 h and dissolved in dry DMF (10 mL). After HATU (35 mg, 0.09 mmol), HOAt (12 mg, 0.09 mmol) and *N*-methylmorpholine (30 μL, 0.27 mmol) were added sequentially. After being stirred at room temperature for 48 h, solvent of the reaction mixture was evaporated under high vacuum. The residue was dissolved with ethyl acetate (50 mL), and the organic layer was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford **3** (5 mg, 58%) as a colorless oil. **TLC**: R_f = 0.4 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -59.3 (*c* 1.0, MeOH). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.74 – 7.68 (m, 4H), 7.57 (dd, *J* = 7.1, 2.2 Hz, 2H), 7.44 – 7.34 (m, 6H), 7.32 – 7.28 (m, 3H), 7.18 (d, *J* = 9.7 Hz, 1H), 7.01 (d, *J* = 8.8 Hz, 1H), 6.95 (d, *J* = 7.9 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.5 Hz, 2H), 6.26 (s, 1H), 5.52 (d, *J* = 9.5 Hz, 1H), 5.24 (dd, *J* = 6.4, 1.7 Hz, 1H), 4.56 (d, *J* = 5.8 Hz, 1H), 4.50 (d, *J* = 5.2 Hz, 1H), 4.37 (d, *J* = 9.5 Hz, 2H), 4.16-4.11 (m, 2H), 4.08 (dd, *J* = 7.2, 3.8 Hz, 2H), 2.51 – 2.29 (m, 5H), 2.07 – 1.98 (m, 1H), 1.77 (s, 1H), 1.65 – 1.51 (m, 3H), 1.45 (s, 9H), 1.40 – 1.37 (m, 6H), 1.29 (d, *J* = 6.3 Hz, 3H), 1.09 (s, 9H), 1.00 – 0.94 (m, 2H), 0.03 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.60, 173.26, 172.22, 171.27, 170.69, 169.39, 156.93, 153.60, 135.53, 134.70, 134.09, 133.15, 129.81, 129.31, 129.03, 128.85, 127.95, 127.73, 119.41, 80.51, 72.32, 62.87, 57.38, 57.07, 52.38, 51.82, 51.75, 40.98, 34.48, 31.15, 30.44, 28.35, 27.06, 26.57, 25.14, 19.48, 17.99, 17.57, 17.30, 17.00, -1.47 ppm. **HRMS** (*m/z*): calculated for $C_{59}H_{81}N_5O_{11}SeSi_2Na^+$ $[M + Na]^+$: 1194.4529, found: 1194.4526.



***D*-Tyr-OMe** **28-1**

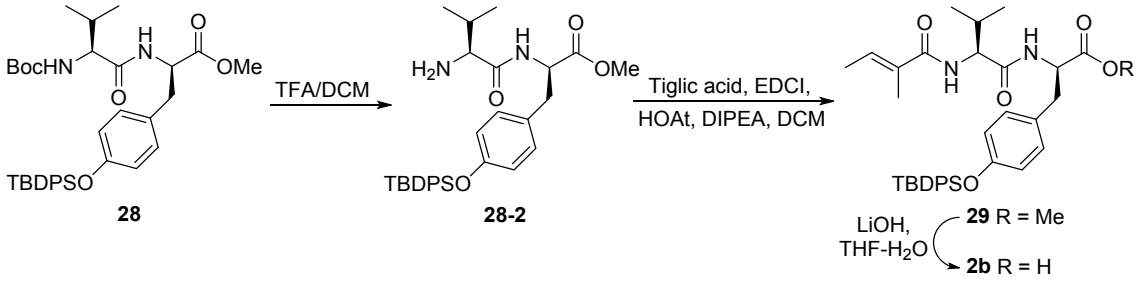
To a solution of *D*-Tyr-OMe (2.30 g, 9.93 mmol) and *L*-Boc-Val-OH (2.20 g, 9.93 mmol) in dry DCM (100 mL), DIPEA (10.4 mL, 59.58 mmol), HOAt (4.0 g, 29.79 mmol) and EDCI (5.7 g, 29.79 mmol) were added at 0 ^oC. After being warmed to room temperature and stirred for 12 h, the reaction was quenched with H_2O (100 mL), diluted with ethyl acetate (100 mL). The organic solution was washed with saturated NH_4Cl (aq.) (50 mL) and brine (50 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo*

and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:4) to afford **28-1** (3.37 g, 86%) as a colorless oil. **TLC**: R_f = 0.4 (silica gel, EtOAc/hexane = 1:2). $[\alpha]_D^{20}$ = -34.8 (*c* 2.0, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.94 (d, *J* = 8.4 Hz, 2H), 6.78 – 6.63 (m, 3H), 5.12 (d, *J* = 8.5 Hz, 1H), 4.86 (dd, *J* = 14.0, 6.1 Hz, 1H), 4.00 (s, 1H), 3.72 (s, 3H), 3.09 – 2.92 (m, 2H), 2.14 (dq, *J* = 13.2, 6.7 Hz, 1H), 1.45 (s, 9H), 0.88 (d, J = 6.2 Hz, 3H), 0.80 (d, *J* = 6.7 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.21, 171.56, 156.01, 155.45, 130.20, 126.96, 115.67, 80.24, 59.70, 53.20, 52.43, 37.27, 30.64, 29.70, 28.31, 19.27, 17.17 ppm. **HRMS** (*m/z*): calculated for $C_{20}H_{31}N_2O_6^+$ [M + H]$^+$: 395.2177, found: 395.2171.



28-1 **28**

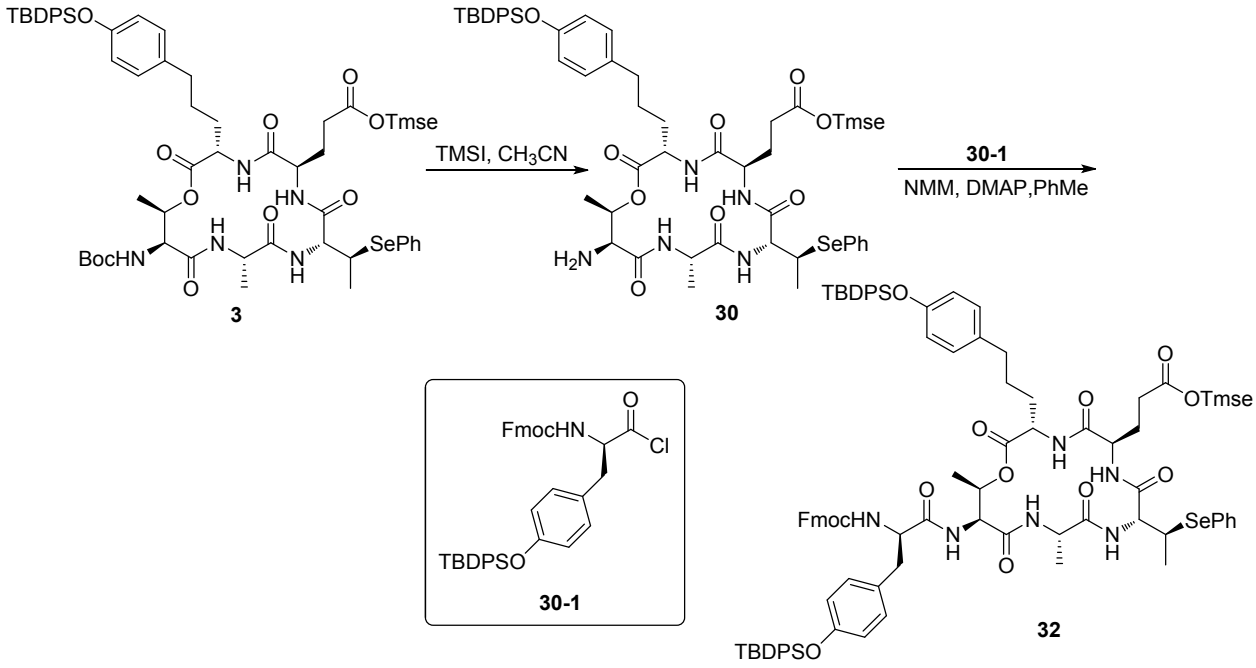
To a solution of dipeptide **28-1** (3.37 g, 8.54 mmol) in dry DCM (50 mL), triethylamine (7.2 mL, 51.24 mmol) and *t*-Butyl-diphenylsilyl chloride (6.6 mL, 25.62 mmol) were added at 0°C. After being stirred at room temperature for 12 h, the reaction mixture was diluted with ethyl ether (100 mL), washed successively with H_2O (25 mL), saturated NH_4Cl (aq.) (2 × 50mL) and brine (50 mL), dried over Na_2SO_4 (s) and concentrated *in vacuo*. The residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:5) to provide **28** (5.13 g, 95%) as a colorless oil. **TLC**: R_f = 0.6 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -23.2 (*c* 8.0, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 – 7.59 (m, 4H), 7.53 – 7.30 (m, 6H), 6.85 (d, *J* = 8.5 Hz, 2H), 6.76 – 6.61 (m, 2H), 6.51 (d, *J* = 8.0 Hz, 1H), 5.07 (d, *J* = 8.3 Hz, 1H), 4.79 (dd, *J* = 14.1, 6.3 Hz, 1H), 3.99 (s, 1H), 3.62 (s, 3H), 3.09 – 2.80 (m, 2H), 2.11 – 2.02 (m, 1H), 1.43 (s, 9H), 1.10 (s, 9H), 0.87 (d, *J* = 5.8 Hz, 3H), 0.79 (d, *J* = 6.8 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.93, 171.25, 155.79, 154.74, 135.51, 132.89, 132.86, 129.94, 129.92, 128.20, 127.77, 119.87, 79.79, 59.56, 53.18, 52.20, 37.29, 30.90, 28.31, 26.53, 19.46, 19.25, 17.31 ppm. **HRMS** (*m/z*): calculated for $C_{36}H_{49}N_2O_6Si^+$ [M + H]$^+$: 633.3354, found: 633.3352.



To a solution of compound **28** (950 mg, 1.50 mmol) in dry DCM (5 mL), trifluoroacetic acid (5 mL) was added at 0 °C. After being stirred at room temperature for 2 h, volatiles of the reaction mixture were removed *in vacuo*. The residue was dried under high vacuum for 2 h to afford amine **28-2**.

To a solution of **28-2** in DCM (20 mL), tiglic acid (300 mg, 3.00 mmol) was added in one portion at 0°C. After EDCI (863 mg, 4.50 mmol), HOAt (612 mg, 4.50 mmol) and DIPEA (1.6 mL, 9.00 mmol) were added. After being stirred at room temperature for 8 h, the reaction was quenched with H_2O (20 mL) and diluted with ethyl acetate (60 mL). The organic solution was washed with saturated NH_4Cl (aq.) (30 mL) and brine (30 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford **29** (719 mg, 78% 2 steps) as a colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 1:2). $[\alpha]_D^{20}$ = -19.6 (*c* 3.0, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 (ddd, *J* = 8.0, 3.0, 1.4 Hz, 4H), 7.47 – 7.41 (m, 2H), 7.41 – 7.35 (m, 4H), 6.89 (d, *J* = 8.5 Hz, 2H), 6.82 (d, *J* = 8.1 Hz, 1H), 6.69 (d, *J* = 8.5 Hz, 2H), 6.50 – 6.42 (m, 1H), 6.38 (d, *J* = 8.6 Hz, 1H), 4.79 (td, *J* = 7.8, 5.6 Hz, 1H), 4.41 (dd, *J* = 8.5, 6.0 Hz, 1H), 3.62 (s, 3H), 3.02 (dd, *J* = 14.1, 5.5 Hz, 1H), 2.92 (dd, *J* = 14.1, 7.7 Hz, 1H), 2.08 – 2.00 (m, 1H), 1.84 (s, 3H), 1.75 (dd, *J* = 6.9, 0.8 Hz, 3H), 1.11 (s, 9H), 0.83 (d, *J* = 6.8 Hz, 3H), 0.79 (d, *J* = 6.8 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.85, 171.11, 169.28, 154.69, 135.50, 132.89, 132.84, 131.57, 131.36, 129.95, 129.92, 128.35, 127.78, 119.79, 58.04, 53.31, 52.25, 37.22, 31.38, 26.52, 19.46, 19.22, 17.77, 13.94, 12.38 ppm. **HRMS** (*m/z*): calculated for $C_{36}H_{47}N_2O_5Si^+$ $[M + H]^+$: 615.3249, found: 615.3240.

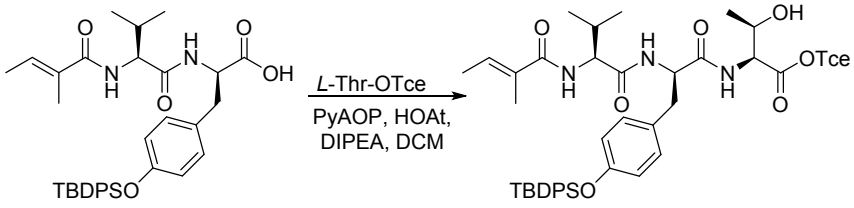
To a solution of compound **29** (492 mg, 0.80 mmol) in THF/H_2O (10 mL, 1/1), $LiOH{\cdot}H_2O$ (168 mg, 4.00 mmol) was added at 0 °C. After being stirred at room temperature for 2 h, volatiles of the reaction mixture were evaporated *in vacuo*. The aqueous layer was washed with diethyl ether (5 mL), then acidified to pH = 3 with 1 N HCl and extracted with ethyl acetate (3 × 30 mL). The combined organic layers were washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* to provide **2b** (456 mg, 95%) as a colorless oil, which was used in the next step of reaction without further purification.



To a solution of compound **3** (17 mg, 0.02 mmol) in dry MeCN (2 mL), iodotrimethylsilane (4.2 μL, 0.03 mmol) was added at 0 °C. After being stirred at 0 °C for 15 min., volatiles of the reaction mixture were removed *in vacuo*. The residue was dried under high vacuum for 2 h to provide amine **30** as a colorless oil, which was used in next step of reaction without further purification.

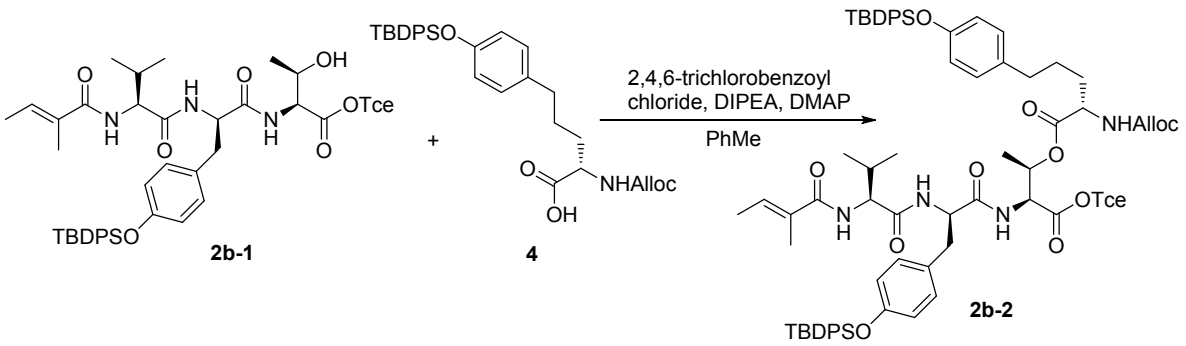
To a solution of amine **30** in dry toluene (2 mL), *N*-methylmorpholine (18 μL, 0.17 mmol) and *O*-TBDPS-*N*-Fmoc-*D*-Tyrosyl Chloride (**30-1**, in 1 mL of dry toluene) were added slowly at 0 °C. This solution was stirred at 0 °C for 15 min. followed by addition of DMAP (7 mg, 0.06 mmol, in 1 mL of dry toluene), and stirred at room temperature for additional 4 h. The reaction was quenched with MeOH (1 mL) and diluted with ethyl acetate (50 mL). The organic solution was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:1) to afford **32** (7.5 mg, 29% 2 steps) as a colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 1:1). $\mathbf{[\alpha]_D^{20}}$ = -17.4 (*c* 1.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.65 (m, 10H), 7.58 (dd, *J* = 6.3, 3.0 Hz, 2H), 7.54 – 7.27 (m, 21H), 7.25 – 7.16 (m, 3H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.89 (d, *J* = 5.3 Hz, 1H), 6.79 (d, *J* = 8.3 Hz, 2H), 6.70 (d, *J* = 8.2 Hz, 2H), 6.63 (d, *J* = 8.4 Hz, 2H), 6.05 (s, 1H), 5.28 (d, *J* = 5.9 Hz, 1H), 4.78 (s, 1H), 4.50 – 4.22 (m, 6H), 4.17 – 4.07 (m, 4H), 3.94 – 3.85 (m, 1H), 3.21 (d, *J* = 12.5 Hz, 1H), 2.84 – 2.71 (m, 1H), 2.49 – 2.26 (m, 5H), 2.05 (d, *J* = 4.5 Hz, 1H), 1.69 (s, 1H), 1.59 – 1.43 (m, 3H), 1.39 – 1.29 (m, 6H), 1.20 (d, *J* = 6.0 Hz, 3H), 1.09 (s, 9H), 1.08 (s, 9H), 0.93 (dd, *J* = 9.9, 6.7 Hz, 2H), -0.01 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.69,

173.60, 173.29, 172.55, 171.84, 170.44, 170.01, 156.30, 154.59, 153.62, 143.93, 143.69, 141.19, 135.53, 135.48, 134.78, 133.89, 133.14, 132.90, 132.85, 129.93, 129.89, 129.82, 129.25, 129.08, 128.98, 127.90, 127.74, 127.66, 127.07, 125.15, 119.89, 119.84, 119.42, 72.38, 67.12, 63.08, 56.97, 55.58, 52.52, 52.34, 51.92, 47.08, 41.01, 37.17, 34.44, 30.78, 30.13, 27.09, 26.57, 26.51, 24.89, 19.48, 19.46, 17.95, 17.53, 17.23, 17.14, -1.48 ppm. **HRMS** (*m/z*): calculated for $C_{94}H_{110}N_6O_{13}SeSi_3Na^+$ [M + Na]$^+$: 1717.6496, found: 1717.6493.

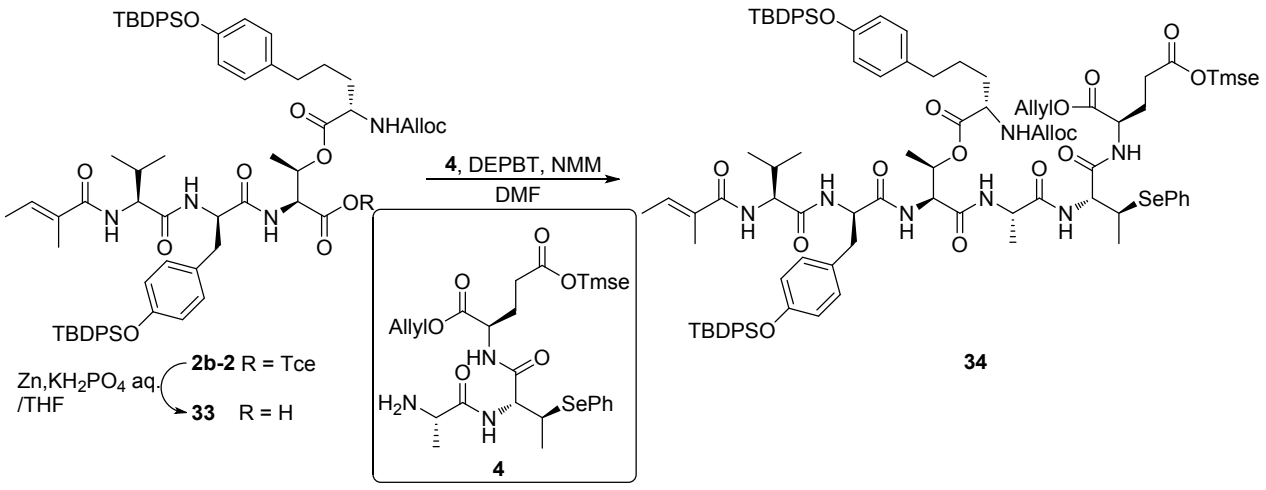


2b **2b-1**

To a solution of acid **2b** (2.68 g, 4.46 mmol) in DCM (20 mL), PyAOP (6.98 g, 13.38 mmol), HOAt (1.82 g, 13.38 mmol) and DIPEA (4.7 mL, 26.76 mmol) *L*-Thr-OTce (1.05g, 4.42 mmol) were added sequentially at 0°C. After being stirred at room temperature for 12 h, the reaction was quenched with H_2O (5 mL) and diluted with ethyl acetate (60 mL). The organic solution was washed with saturated NH_4Cl (aq.) (30 mL) and brine (30 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford **2b-1** (3.13g, 85%) as a colorless oil. **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = +4.4 (*c* 2.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 – 7.67 (m, 4H), 7.60 (d, *J* = 9.1 Hz, 1H), 7.45 – 7.33 (m, 6H), 6.93 (d, *J* = 8.5 Hz, 2H), 6.67 (d, *J* = 8.5 Hz, 2H), 6.51 (d, *J* = 8.1 Hz, 1H), 6.47 – 6.39 (m, 1H), 6.32 (d, *J* = 3.0 Hz, 1H), 4.85 – 4.69 (m, 4H), 4.43 (qd, *J* = 6.5, 2.6 Hz, 1H), 3.48 (dd, *J* = 8.9, 5.0 Hz, 1H), 3.23 (dd, *J* = 14.4, 4.6 Hz, 1H), 2.90 (dd, *J* = 14.4, 9.5 Hz, 1H), 1.90 – 1.82 (m, 1H), 1.79 (s, 3H), 1.73 (dd, *J* = 6.9, 0.8 Hz, 3H), 1.09 (s, 9H), 1.09 (d, *J* = 4.6 Hz, 3H), 0.89 (d, *J* = 6.7 Hz, 3H), 0.53 (d, *J* = 6.7 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.66, 171.54, 171.43, 169.70, 154.65, 135.54, 135.51, 133.47, 132.88, 132.83, 130.37, 130.02, 129.91, 128.90, 127.78, 127.76, 119.88, 94.60, 74.67, 67.91, 62.06, 58.51, 54.34, 36.37, 29.43, 26.54, 19.66, 19.62, 19.44, 18.81, 14.05, 12.44 ppm. **HRMS** (*m/z*): calculated for $C_{41}H_{52}Cl_3N_3O_7SiNa^+$ [M + Na]$^+$: 854.2532, found: 854.2531.



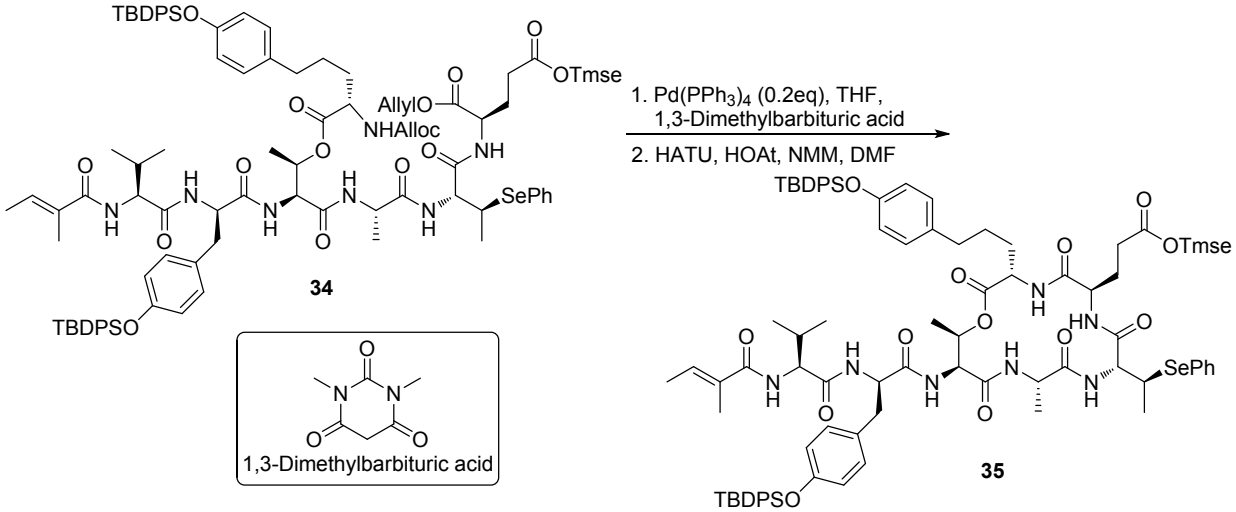
To a solution of alcohol **2b-1** (376 mg, 0.45 mmol) and acid **4** (288 mg, 0.54 mmol) in dry toluene (10 mL) DIPEA (0.4 mL, 2.26 mmol) and 2,4,6-trichlorobenzoyl chloride (0.18 mL, 1.13 mmol) were added at 0°C. After being stirred at 0 °C for 15 min, DMAP (138 mg, 1.13 mmol, in 4mL of tuluene) was added to the solution and then stirred at room temperature for 4 h. The reaction was quenched with MeOH (1 mL) and diluted with ethyl acetate (50 mL). The organic solution was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:1) to afford **2b-2** (432 mg, 71%) as a colorless oil. **TLC**: R_f = 0.6 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = +0.6 (*c* 1.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 – 7.66 (m, 8H), 7.56 (d, *J* = 8.9 Hz, 1H), 7.46 – 7.32 (m, 12H), 6.93 (d, *J* = 8.2 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 6.70 (dd, *J* = 24.8, 8.2 Hz, 5H), 6.45 – 6.29 (m, 2H), 5.93 – 5.83 (m, 1H), 5.52 (d, *J* = 8.0 Hz, 1H), 5.41 (dd, *J* = 6.2, 3.0 Hz, 1H), 5.36 – 5.22 (m, 1H), 5.17 (d, *J* = 10.5 Hz, 1H), 4.75 – 4.47 (m, 6H), 4.26 (d, *J* = 6.7 Hz, 1H), 4.11 (dd, *J* = 13.4, 6.4 Hz, 1H), 3.16 (dd, *J* = 14.5, 5.2 Hz, 1H), 2.85 (dd, *J* = 13.2, 3.6 Hz, 1H), 2.55 – 2.38 (m, 2H), 1.91 (dd, *J* = 13.5, 6.8 Hz, 1H), 1.81 – 1.66 (m, 7H), 1.60 (d, *J* = 19.6 Hz, 3H), 1.28 – 1.21 (m, 3H), 1.09 (s, 9H), 1.09 (s, 9H), 0.78 (d, *J* = 6.7 Hz, 3H), 0.65 (d, *J* = 6.5 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.05, 171.76, 171.40, 169.66, 168.00, 156.13, 154.56, 153.75, 135.53, 135.48, 134.08, 133.92, 133.07, 132.88, 132.85, 132.63, 132.49, 131.81, 131.25, 129.93, 129.85, 128.97, 128.87, 128.21, 127.78, 127.74, 119.75, 119.50, 117.96, 94.25, 74.91, 70.46, 65.99, 59.15, 55.56, 54.28, 53.82, 36.36, 34.46, 31.39, 30.48, 27.25, 26.54, 19.46, 19.02, 18.42, 17.44, 13.98, 12.30 ppm. HRMS(ESI) *m/z* calculated for $C_{72}H_{87}Cl_3N_4O_{11}Si_2$ $[M+Na]^+$: 1367.4868, found: 1367.4874. **HRMS** (*m/z*): calculated for $C_{72}H_{87}Cl_3N_4O_{11}Si_2Na^+$ $[M + Na]^+$: 1367.4868, found: 1367.4874.



To a solution of compound **2b-2** (360 mg, 0.27 mmol) in THF (5 mL) and a aqueous solution of KH_2PO_4 (pH = 4.3), zinc powder (cat.) was added at room temperature. After being stirred at room temperature for 10 h, the reaction mixture was filtered through a pad of Celite and washed with ethyl acetate. The aqueous layer was extracted with ethyl acetate (2 × 30 mL), and the combined organic layers were washed with brine (10 mL) and dried over anhydrous Na_2SO_4 (s). The solvent was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 1:2) to afford acid **33** (267 mg, 81%) as a colorless oil.

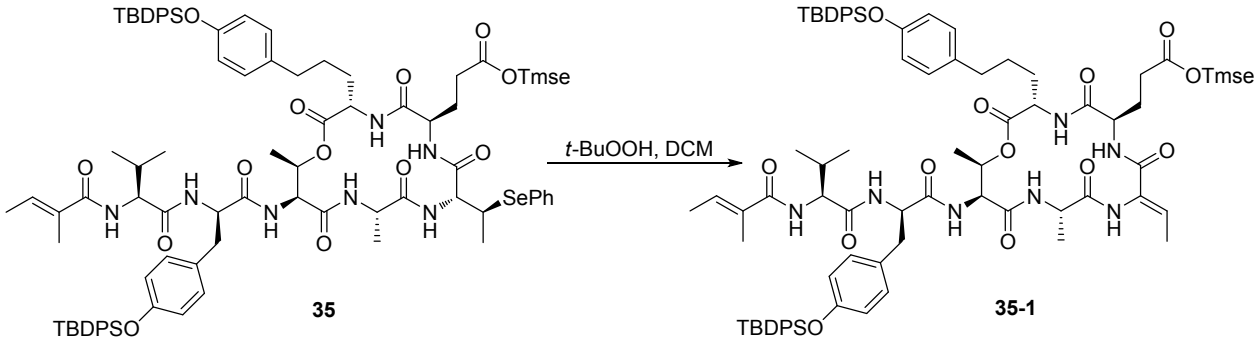
To a solution of acid **33** (72 mg, 0.06 mmol) and amine 2**4** obtained above in dry DMF (3 mL), DEPBT (53 mg, 0.18 mmol) and NMM (39 μL, 0.35 mmol) was added at 0 °C. After being warmed to room temperature and stirred for 12 h, the reaction was quenched with H_2O (1 mL) and diluted with ethyl acetate (60 mL). The organic solution was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 3:2) to afford **34** (63 mg, 60% over 2 steps from **2b-2**) as a colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -14.6 (*c* 1.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 6.1 Hz, 1H), 7.70 (dd, *J* = 10.8, 4.1 Hz, 8H), 7.50 (dd, *J* = 7.6, 1.6 Hz, 2H), 7.44 – 7.39 (m, 4H), 7.39 – 7.33 (m, 8H), 7.26 – 7.18 (m, 3H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.94 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 6.69 – 6.65 (m, 4H), 6.33 (dd, *J* = 13.5, 6.7 Hz, 1H), 6.22 (d, *J* = 6.7 Hz, 1H), 5.91-5.80 (m, 2H), 5.77 (d, *J* = 7.8 Hz, 1H), 5.33 – 5.12 (m, 5H), 4.68 – 4.44 (m, 8H), 4.40 – 4.32 (m, 1H), 4.22 (d, *J* = 7.0 Hz, 1H), 4.18 – 4.11 (m, 2H), 4.01 (t, *J* = 7.2 Hz, 1H), 3.85 – 3.77 (m, 1H), 3.22 (dd, *J* = 14.2, 5.2 Hz, 1H), 2.80 (dd, *J* = 14.2, 10.1 Hz, 1H), 2.54 – 2.36 (m, 4H), 2.21 (dd, *J* = 13.2, 6.2 Hz, 1H), 2.12 – 2.05 (m, 1H), 1.91 (dd, *J* = 13.7, 6.8 Hz, 1H), 1.84-1.66 (m, 7H), 1.60 (d, *J* = 15.0 Hz, 3H), 1.48 – 1.38 (m, 6H), 1.17 (d, *J* = 6.4 Hz, 3H), 1.10 (s, 9H), 1.09 (s, 9H), 0.96 (dd, *J* = 9.3, 7.8 Hz, 2H), 0.78 (d, *J* = 6.8 Hz, 3H), 0.66

(d, J = 6.6 Hz, 3H), 0.02 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.07, 172.94, 172.34, 172.13, 171.33, 171.24, 170.96, 170.37, 169.98, 169.64, 156.34, 154.55, 153.74, 135.53, 135.47, 134.88, 133.99, 133.11, 133.07, 133.04, 132.86, 132.62, 131.56, 130.98, 129.94, 129.86, 129.17, 128.98, 128.45, 127.97, 127.79, 127.74, 119.75, 119.48, 118.92, 117.97, 70.13, 66.06, 62.93, 59.78, 57.55, 56.99, 55.23, 54.04, 52.18, 52.07, 50.59, 40.30, 36.10, 34.59, 31.18, 30.51, 29.86, 27.21, 26.65, 26.55, 19.46, 19.08, 18.74, 17.53, 17.26, 16.98, 14.07, 12.38, -1.48 ppm. **HRMS** (*m/z*): calculated for $C_{96}H_{125}N_7O_{16}SeSi_3Na^+$ $[M + Na]^+$: 1818.7548, found: 1818.7546.



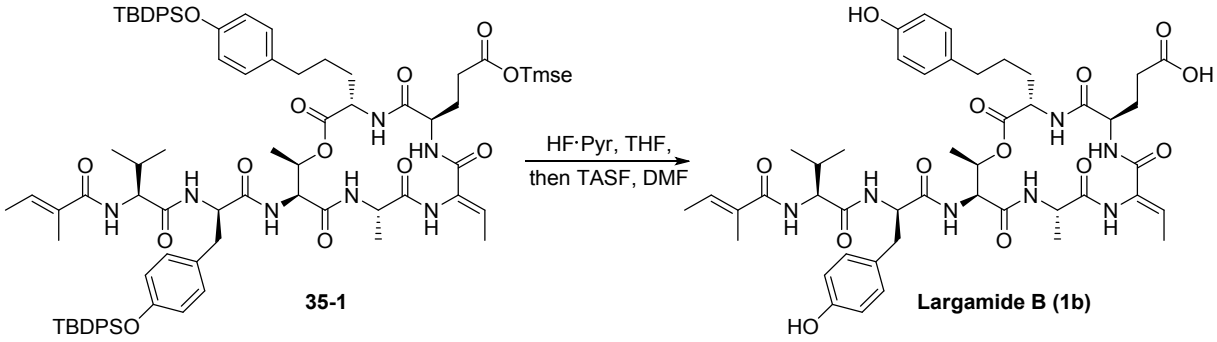
To a solution of **34** (24 mg, 0.01 mmol) and 1,3-dimethylbarbituric acid (5 mg, 0.03 mmol) in anhydrous THF (4 mL), $Pd(PPh_3)_4$ (3 mg, 0.003 mmol) in THF (1 mL) was added. After being stirred for 2 h at room temperature, volatiles of the reaction mixture were removed *in vacuo*. The residue was dried under high vacuum for 2 h and dissolved in dry DMF (15 mL). To this solution, HATU (51 mg, 0.13 mmol), HOAt (18 mg, 0.13 mmol) and *N*-methylmorpholine (44 μL, 0.40 mmol) were added sequentially, and the reaction mixture was stirred at room temperature for 48 h. Solvent was evaporated under high vacuum, and the residue was dissolved with ethyl acetate (60 mL). After being washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s), the organic phase was concentrated *in vacuo* to afford the crude product, which was purified by flash chromatography (silica gel, EtOAc/hexane = 3:2) to afford **35** (14 mg, 63% 2 steps) as a colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 3:2). $[\alpha]_D^{20}$ = -20.0 (*c* 1.0, MeOH). **^{1}H NMR** (400 MHz, CD_3OD) δ 7.74 – 7.64 (m, 8H), 7.63 – 7.51 (m, 2H), 7.46 – 7.39 (m, 4H), 7.38 – 7.34 (m, 8H), 7.31 – 7.26 (m, 3H), 6.98 (d, J = 8.5 Hz, 2H), 6.88 (d, J = 8.5 Hz, 2H), 6.62 (dd, J = 10.9, 8.5 Hz, 4H), 6.37 (dd, J = 7.5, 6.1 Hz, 1H), 5.19 (dd, J = 6.4, 2.9 Hz, 1H), 4.66 (dd, J = 9.4, 6.0 Hz, 1H), 4.58 (dd, J = 7.7, 4.7 Hz, 1H), 4.53 (dd, J = 8.2, 4.5 Hz, 1H), 4.24 (dd, J = 9.2, 5.5 Hz, 1H), 4.19 – 4.00 (m, 6H),

3.04 (dd, *J* = 14.0, 5.9 Hz, 1H), 2.77 (dd, *J* = 14.0, 9.6 Hz, 1H), 2.56 – 2.35 (m, 5H), 2.20 – 2.06 (m, 1H), 1.92 – 1.82 (m, 1H), 1.78 (s, 4H), 1.71 (d, *J* = 6.1 Hz, 3H), 1.59 (dd, *J* = 10.7, 6.0 Hz, 3H), 1.49 (d, *J* = 6.8 Hz, 1H), 1.33 (d, *J* = 7.1 Hz, 3H), 1.19 (d, *J* = 6.6 Hz, 3H), 1.07 (s, 9H), 1.06 (s, 9H), 1.03 (s, 3H), 0.97 – 0.90 (m, 2H), 0.75 (d, *J* = 6.7 Hz, 3H), 0.66 (d, *J* = 6.7 Hz, 3H), -0.02 (s, 9H) ppm. **^{13}C NMR** (100 MHz, CD_3OD) δ 174.10, 172.66, 172.42, 171.56, 170.79, 170.44, 169.82, 169.39, 168.86, 154.40, 153.46, 135.24, 135.21, 134.75, 132.87, 132.58, 131.43, 130.93, 129.78, 129.71, 129.66, 129.41, 128.88, 128.74, 127.53, 127.47, 119.26, 119.04, 72.53, 62.62, 60.16, 59.01, 58.43, 56.12, 55.32, 54.82, 53.18, 51.13, 50.42, 40.22, 36.88, 34.24, 30.65, 30.55, 30.07, 26.68, 25.63, 25.56, 20.78, 18.78, 18.75, 18.39, 17.62, 16.75, 15.56, 15.14, 13.06, 12.62, 11.20, -2.74 ppm. **HRMS** (*m/z*): calculated for $C_{89}H_{115}N_7O_{13}SeSi_3Na^+$ [M + Na]$^+$: 1676.6918, found: 1676.6916.



To a solution of **35** (8 mg, 0.01 mmol) in CH_2Cl_2 (2 mL), 5.5 M *t*-BuOOH in decane (18 μL, 0.10 mmol) was added at 0 °C. Five minutes later, the reaction mixture was allowed to reach room temperature and then stirred for 2 h. The reaction was quenched with $NaHCO_3$ (aq.)/$Na_2S_2O_3$ (aq.) (6 mL, 1:1) and diluted with ethyl acetate (50 mL). The organic solution was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was purified by flash chromatography (silica gel, EtOAc/hexane = 2:1) to afford **35-1** (5 mg, 62%) as a colorless oil. **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 2:1). **$[\alpha]_D^{20}$** = -46.7 (*c* 0.5, MeOH). **^{1}H NMR** (400 MHz, CD_3OD) δ 8.71 (d, *J* = 2.4 Hz, 1H), 8.09 (d, *J* = 7.9 Hz, 1H), 7.80 (d, *J* = 8.8 Hz, 1H), 7.72 – 7.66 (m, 9H), 7.62 (d, *J* = 8.7 Hz, 1H), 7.45 – 7.39 (m, 4H), 7.39 – 7.33 (m, 8H), 7.29 (d, *J* = 8.0 Hz, 1H), 6.99 (d, *J* = 8.5 Hz, 2H), 6.88 (d, *J* = 8.5 Hz, 2H), 6.68-6.58 (m, 5H), 6.39 – 6.32 (m, 1H), 5.19 (dd, *J* = 6.3, 2.8 Hz, 1H), 4.72 – 4.65 (m, 1H), 4.58 (dt, *J* = 11.9, 5.9 Hz, 2H), 4.44 (td, *J* = 9.4, 4.5 Hz, 1H), 4.20 – 4.03 (m, 4H), 3.05 (dd, *J* = 14.0, 5.9 Hz, 1H), 2.77 (dd, *J* = 14.0, 9.5 Hz, 1H), 2.58 – 2.38 (m, 5H), 2.17 (dt, *J* = 14.2, 7.1 Hz, 1H), 1.86 (dd, *J* = 17.0, 10.2 Hz, 2H), 1.81 – 1.69 (m, 10H), 1.60 – 1.48 (m, 3H), 1.37 (d, *J* = 7.0 Hz, 3H),

1.07 (s, 9H), 1.06 (s, 9H), 1.04 (s, 3H), 0.97 – 0.91 (m, 2H), 0.76 (d, J = 6.7 Hz, 3H), 0.67 (d, J = 6.8 Hz, 3H), -0.02 (s, 9H) ppm. **^{13}C NMR** (100 MHz, CD_3OD) δ 175.45, 174.06, 172.75, 172.68, 172.39, 171.77, 170.43, 170.39, 169.70, 164.61, 154.39, 153.44, 135.24, 135.22, 134.69, 132.88, 132.59, 131.50, 131.47, 130.87, 129.79, 129.73, 129.66, 129.44, 129.24, 128.75, 127.54, 127.45, 119.26, 119.02, 72.73, 62.59, 60.14, 59.07, 58.96, 55.48, 55.40, 54.86, 54.81, 52.84, 50.85, 50.10, 36.91, 33.89, 30.71, 29.79, 26.73, 25.96, 25.62, 25.58, 19.47, 18.78, 18.76, 18.41, 17.56, 16.75, 15.19, 15.15, 13.08, 12.63, 11.59, 11.24, -2.73 ppm. **HRMS** (*m/z*): calculated for $C_{83}H_{109}N_7O_{13}Si_3Na^+$ $[M + Na]^+$: 1518.7283, found: 1518.7288.



To a solution of **35-1** (10 mg, 0.01 mmol) in anhydrous THF (0.5 mL), pyridine (120 µL) and HF·Py (60 µL) were added at 0 °C. Five minutes later, the reaction mixture was allowed to reach room temperature, stirred for additional 2 h, and then diluted with ethyl acetate (50 mL). The organic solution was washed with saturated NH_4Cl (aq.) (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* and the residue was dissolved in dry DMF (2 mL) at 0 °C. To this solution, TASF (37 mg, 0.134 mmol) in dry DMF (0.5 mL) was added at 0 °C. After being allowed to warm to room temperature and stirred for additional 4 h, the solvents of the reaction mixture were evaporated under high vacuum. The residue was dissolved with ethyl acetate (50 mL), washed with brine (10 mL), dried over anhydrous Na_2SO_4 (s). The organic phase was concentrated *in vacuo* to provide the crude product, which was purified by HPLC. HPLC purification was performed on Agilent 1200 system, equipped with a reverse-phase C18 S G300 column (S-5uM, 10.0 mm i.d. × 150 mm length, from Fine Chemicals, Shiseido CAPCELL PAK). A linear elution gradient consisting of 45:55 H_2O/MeOH brought to 30:70 H_2O/MeOH over 35 min, at a flow rate of 0.8 mL/min was employed. The temperature was 25 °C and the DAD detector was set at 220 nm and 254 nm wave length. Fraction whose retention time is between 10.1-10.5 min was collected and concentrated in *vacuo* to afford the revised structure of Largamide B **1b** (3 mg, 50%) as an colorless amorphous solid.

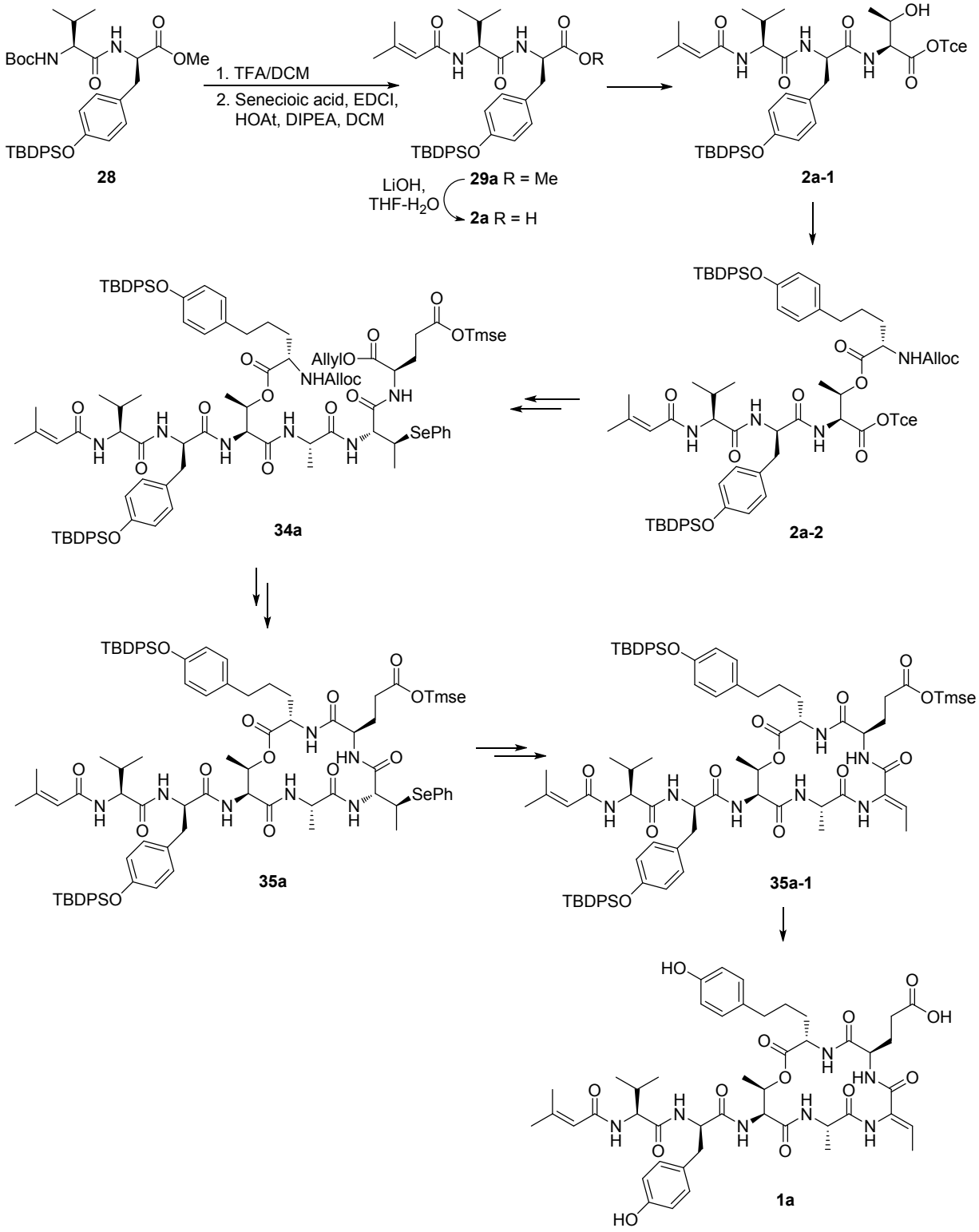
Analytical data for Revised Structure of Largamide B (1b) $[\alpha]_D^{20}$ = -72.6 (*c* 0.12, MeOH). **^{1}H NMR** (400 MHz, CD_3OD) δ 7.70 (d, J = 9.5 Hz, 1H), 7.08 (d, J = 8.4 Hz, 2H), 6.98 (d, J = 8.4 Hz, 2H), 6.74 – 6.63 (m,

5H), 6.38 (q, *J* = 6.7 Hz, 1H), 5.21 (dd, *J* = 6.3, 2.8 Hz, 1H), 4.70 (dd, *J* = 9.4, 5.9 Hz, 1H), 4.61 (t, *J* = 6.8 Hz, 2H), 4.51 (dd, *J* = 10.0, 4.1 Hz, 1H), 4.19 (dd, *J* = 13.5, 7.1 Hz, 2H), 3.06 (dd, *J* = 13.9, 5.8 Hz, 1H), 2.82 (dd, *J* = 13.9, 9.6 Hz, 1H), 2.59 – 2.44 (m, 5H), 2.19 (dd, *J* = 17.1, 6.8 Hz, 1H), 1.97 – 1.85 (m, 2H), 1.84 – 1.78 (m, 6H), 1.75 (d, *J* = 6.9 Hz, 3H), 1.65 – 1.53 (m, 3H), 1.40 (d, *J* = 7.1 Hz, 3H), 1.11 (d, *J* = 6.3 Hz, 3H), 0.78 (d, *J* = 6.7 Hz, 3H), 0.75 (d, *J* = 6.8 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, CD_3OD) δ 176.63, 176.60, 174.03, 173.62, 173.10, 171.73, 171.63, 170.84, 165.80, 157.15, 156.00, 134.04, 132.64, 132.16, 132.10, 131.13, 130.38, 130.10, 128.59, 116.09, 115.75, 73.88, 60.06, 56.56, 56.35, 53.90, 52.08, 51.27, 38.20, 35.14, 31.92, 31.37, 31.02, 28.15, 27.11, 19.48, 18.45, 16.37, 16.23, 13.74, 12.72, 12.34 ppm.

^{1}H NMR (500 MHz, DMF-d_7) δ 12.42 (s, 1H), 10.13 (s, 1H), 9.29 (s, 1H), 9.24 (s, 1H), 8.80 (s, 1H), 8.10 (s, 1H), 7.87 (d, *J* = 8.5 Hz, 1H), 7.64 (d, *J* = 9.4 Hz, 1H), 7.49 (d, *J* = 8.7 Hz, 1H), 7.25 (d, *J* = 8.5 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 6.73 (d, *J* = 8.4 Hz, 2H), 6.70 (d, *J* = 8.4 Hz, 2H), 6.56 (q, *J* = 7.1 Hz, 1H), 6.42 (qq, *J*= 6.7, 1.5 Hz, 1H), 5.37 (qd, *J* = 6.3, 2.9 Hz, 1H), 4.79 (m, 1H), 4.73 (m, 1H), 4.65 (ddd, *J* = 9.5, 9.5, 5.5 Hz, 1H), 4.50 (ddd, *J* = 10.0, 9.0, 5.0 Hz, 1H), 4.30 (qd, *J* = 7.0, 2.5 Hz, 1H) 4.28 (dd, *J* = 8.5, 7.0 Hz, 1H), 3.06 (dd, *J* = -13.9, 4.7 Hz, 1H), 2.83 (dd, *J* = -13.8, 9.9 Hz, 1H), 2.60 – 2.42 (m, 5H), 2.18 (m, 1H), 2.02 (m, 1H), 1.89 (m, 1H), 1.80 (s, 3H), 1.77 (d, *J* = 7.2 Hz, 3H), 1.71 (dq, *J* = 6.7, 1.5 Hz, 3H), 1.60 (m, 1H), 1.58 (m, 1H), 1.56 (m, 1H), 1.38 (d, *J* = 7.0 Hz, 3H), 1.17 (d, *J* = 6.3 Hz, 3H), 0.75 (d, *J* = 6.8 Hz, 3H), 0.72 (d, *J* = 6.7 Hz, 3H) ppm. **^{13}C NMR** (125 MHz, DMF-d_7) δ 175.84, 175.18, 172.71, 172.10, 171.75, 171.72, 170.35, 169.38, 164.04, 157.20, 156.56, 133.18, 132.83, 131.17, 131.03, 130.44, 129.90, 129.28, 128.67, 115.75, 115.67, 73.46, 59.29, 55.88, 55.59, 53.19, 51.15, 50.65, 38.16, 34.63, 31.66, 31.26, 31.10, 27.93, 27.04, 19.81, 18.31, 16.74, 16.27, 13.86, 12.76, 12.59 ppm.

HRMS (*m/z*): calculated for $C_{46}H_{62}N_7O_{13}^+$ $[M + H]^+$: 920.4400, found: 920.4398.

Synthesis Scheme and Analytical Data for the Structure of Largamide B (1a)



Analytical data for 29a: (Yield = 74%, 2 steps) **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -31.7 (*c* 3.0, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.67 (m, 4H), 7.45 – 7.33 (m, 6H), 6.84 (d, *J* = 8.5 Hz, 2H), 6.67 (d, *J* = 8.5 Hz, 2H), 6.54 (d, *J* = 8.0 Hz, 1H), 6.00 (d, *J* = 8.6 Hz, 1H), 5.58 – 5.53 (m, 1H),

4.77 (dd, *J* = 14.2, 6.4 Hz, 1H), 4.32 (dd, *J* = 8.5, 6.0 Hz, 1H), 3.61 (s, 3H), 3.01 – 2.89 (m, 2H), 2.13 (d, *J* = 1.0 Hz, 3H), 2.08 – 2.02 (m, 1H), 1.80 (d, *J* = 0.9 Hz, 3H), 1.10 (s, 9H), 0.83 (d, *J* = 6.8 Hz, 3H), 0.80 (d, *J* = 6.8 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 171.79, 171.07, 166.78, 154.72, 151.89, 135.52, 135.50, 132.89, 132.84, 129.96, 129.92, 128.20, 127.78, 127.76, 119.84, 118.17, 57.82, 53.18, 52.22, 37.23, 30.98, 27.16, 26.50, 19.89, 19.46, 19.25, 17.79 ppm. **HRMS** (*m/z*): calculated for $C_{36}H_{47}N_2O_5Si^+$ $[M + H]^+$: 615.3249, found: 615.3236.

Analytical data for 2a-1: (Yield = 87%) **TLC**: R_f = 0.5 (silica gel, EtOAc/hexane = 2:1). $[\alpha]_D^{20}$ = -5.3 (*c* 3.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.70 (ddd, *J* = 7.9, 4.0, 1.4 Hz, 4H), 7.55 (d, *J* = 9.0 Hz, 1H), 7.44 – 7.34 (m, 6H), 6.93 (d, *J* = 8.5 Hz, 2H), 6.67 (d, *J* = 8.5 Hz, 2H), 6.54 (d, *J* = 8.1 Hz, 1H), 6.21 (d, *J* = 5.1 Hz, 1H), 5.62 (s, 1H), 4.78 (dd, *J* = 25.5, 11.9 Hz, 2H), 4.72 – 4.62 (m, 3H), 4.43 – 4.35 (m, 1H), 3.49 (dd, *J* = 8.9, 5.2 Hz, 1H), 3.26 (dd, *J* = 14.4, 4.3 Hz, 1H), 2.83 (dd, *J* = 14.4, 10.0 Hz, 1H), 1.98 (s, 3H), 1.87 – 1.79 (m, 4H), 1.09 (s, 9H), 1.04 (d, *J* = 6.5 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 3H), 0.50 (d, *J* = 6.7 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): *δ* 172.01, 171.56, 169.75, 169.21, 154.65, 151.61, 135.54, 135.51, 132.86, 132.82, 129.95, 129.92, 128.94, 127.78, 127.76, 119.91, 117.86, 94.59, 74.65, 67.78, 61.77, 58.42, 54.78, 36.38, 29.34, 26.92, 26.54, 20.38, 19.66, 19.61, 19.45, 18.82 ppm. **HRMS** (*m/z*): calculated for $C_{41}H_{53}Cl_3N_3O_7Si^+$ $[M + H]^+$: 832.2713, found: 832.2710.

Analytical data for 2a-2: (Yield = 73%) **TLC**: R_f = 0.6 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -11.9 (*c* 2.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.74 – 7.68 (m, 8H), 7.64 (d, *J* = 8.9 Hz, 1H), 7.45 – 7.39 (m, 4H), 7.39 – 7.34 (m, 8H), 6.94 (d, *J* = 8.3 Hz, 2H), 6.88 – 6.80 (m, 3H), 6.71 – 6.63 (m, 5H), 6.21 (d, *J* = 8.0 Hz, 1H), 6.00 (s, 1H), 5.92 – 5.81 (m, 1H), 5.54 (dd, *J* = 20.4, 11.9 Hz, 2H), 5.41 (dd, *J* = 6.2, 3.7 Hz, 1H), 5.28 (d, *J* = 17.0 Hz, 1H), 5.18 (dd, *J* = 10.4, 1.2 Hz, 1H), 4.75 – 4.51 (m, 6H), 4.35 – 4.23 (m, 1H), 4.07 (t, *J* = 7.8 Hz, 1H), 3.16 (dd, *J* = 14.5, 5.0 Hz, 1H), 2.84 (dd, *J* = 14.4, 9.6 Hz, 1H), 2.56 – 2.38 (m, 2H), 2.08 (s, 3H), 1.96 – 1.85 (m, 1H), 1.82 – 1.72 (m, 4H), 1.60 (dd, *J* = 22.1, 6.2 Hz, 3H), 1.26 (d, *J* = 6.5 Hz, 3H), 1.09 (s, 9H), 1.08 (s, 9H), 0.78 (d, *J* = 6.7 Hz, 3H), 0.64 (d, *J* = 6.6 Hz, 3H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): *δ* 172.53, 171.78, 171.35, 167.85, 167.27, 156.17, 154.52, 153.76, 151.83, 135.53, 135.48, 134.17, 133.98, 133.05, 132.90, 132.86, 132.61, 132.49, 129.92, 129.86, 129.00, 128.19, 127.79, 127.75, 119.74, 119.52, 118.13, 118.02, 94.28, 74.83, 70.41, 66.01, 58.86, 55.62, 54.30, 53.75, 35.96, 34.50, 31.41, 30.24, 27.21, 26.55, 26.53, 19.88, 19.46, 19.03, 18.43, 17.49 ppm. **HRMS** (*m/z*): calculated for $C_{72}H_{88}Cl_3N_4O_{11}Si_2^+$ $[M + H]^+$: 1345.5048, found: 1345.5050.

Analytical data for 34a: (Yield = 64%, 2 steps) **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 1:1). $[\alpha]_D^{20}$ = -

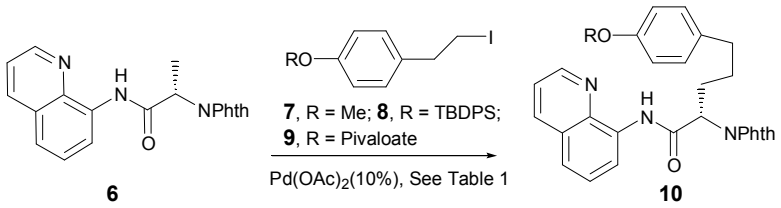
22.1 (*c* 1.0, MeOH). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.75 – 7.65 (m, 8H), 7.51 (dd, *J* = 10.8, 5.5 Hz, 2H), 7.44 – 7.33 (m, 12H), 7.23 (t, *J* = 6.1 Hz, 3H), 7.04 (s, 1H), 6.93 (d, *J* = 8.3 Hz, 2H), 6.87 – 6.82 (m, 2H), 6.68 – 6.64 (m, 4H), 6.24 (s, 1H), 5.95 – 5.78 (m, 2H), 5.67 – 5.49 (m, 2H), 5.34 – 5.12 (m, 5H), 4.76 – 4.46 (m, 8H), 4.35 – 4.24 (m, 1H), 4.22 – 4.02 (m, 4H), 3.92 – 3.74 (m, 1H), 3.11 (dd, *J* = 12.2, 4.4 Hz, 1H), 2.96 – 2.79 (m, 1H), 2.52 – 2.38 (m, 4H), 2.27 – 2.16 (m, 2H), 2.11-2.01 (m, 5H), 1.74 (d, *J* = 15.8 Hz, 3H), 1.64 – 1.53 (m, 3H), 1.48 – 1.38 (m, 6H), 1.09 (s, 9H), 1.08 (s, 9H), 0.98 – 0.93 (m, 2H), 0.90 – 0.67 (m, 6H), 0.02 (s, 9H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.10, 172.98, 172.59, 172.44, 172.02, 171.73, 171.18, 169.97, 169.85, 169.74, 167.74, 156.33, 154.62, 153.78, 153.75, 152.72, 135.53, 135.46, 134.90, 134.63, 133.84, 133.05, 132.81, 132.46, 132.37, 131.61, 131.57, 129.96, 129.86, 129.18, 129.02, 128.99, 128.77, 128.68, 128.59, 128.00, 127.80, 127.75, 120.00, 119.80, 119.53, 118.82, 118.22, 118.03, 117.86, 70.13, 66.19, 66.09, 65.97, 62.96, 62.90, 58.82, 57.08, 56.73, 55.65, 54.07, 52.09, 50.74, 40.50, 40.08, 37.11, 36.07, 34.53, 31.21, 30.52, 29.71, 27.50, 27.25, 26.54, 26.52, 20.03, 19.46, 19.25, 18.60, 17.25, 17.11, -1.48 ppm. **HRMS** (*m/z*): calculated for $C_{96}H_{125}N_7O_{16}SeSi_3Na^+$ $[M + Na]^+$: 1818.7548, found: 1818.7546.

Analytical data for 35a: (Yield = 60%, 2 steps) **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 3:2). **$[\alpha]_D^{20}$** = -44.1 (*c* 0.5, MeOH). **^{1}H NMR** (400 MHz, CD_3OD) δ 7.72 – 7.66 (m, 8H), 7.58 (dd, *J* = 7.3, 2.1 Hz, 2H), 7.44 – 7.33 (m, 12H), 7.30 – 7.28 (m, 3H), 6.97 (d, *J* = 8.5 Hz, 2H), 6.88 (d, *J* = 8.5 Hz, 2H), 6.62 (t, *J* = 8.4 Hz, 4H), 5.77 – 5.75 (m, 1H), 5.19 (dd, *J* = 6.3, 2.8 Hz, 1H), 4.65 (dd, *J* = 10.0, 7.6 Hz, 2H), 4.58 – 4.50 (m, 2H), 4.25 (dd, *J* = 9.2, 5.4 Hz, 1H), 4.15 – 4.01 (m, 6H), 2.99 (dd, *J* = 13.9, 6.3 Hz, 1H), 2.79 (dd, *J* = 13.9, 9.0 Hz, 1H), 2.56 – 2.37 (m, 5H), 2.10 (dd, *J* = 14.8, 8.1 Hz, 1H), 2.05 (d, *J* = 0.9 Hz, 3H), 1.89 (dd, *J* = 13.5, 6.7 Hz, 1H), 1.79 (d, *J* = 16.6 Hz, 4H), 1.65 – 1.51 (m, 3H), 1.33 (d, *J* = 7.1 Hz, 3H), 1.19 (d, *J* = 6.8 Hz, 3H), 1.07 (s, 9H), 1.06 (s, 9H), 1.03 (d, *J* = 6.4 Hz, 3H), 0.96 – 0.91 (m, 2H), 0.74 (dd, *J* = 11.9, 6.8 Hz, 6H), -0.02 (s, 9H) ppm. **^{13}C NMR** (100 MHz, CD_3OD): δ175.42, 174.03, 172.62, 172.31, 171.60, 170.71, 169.69, 169.33, 167.67, 154.38, 153.45, 150.87, 135.24, 135.21, 134.71, 134.66, 132.87, 132.58, 129.78, 129.74, 129.66, 129.36, 128.89, 128.81, 128.75, 127.60, 127.53, 127.47, 119.25, 119.03, 117.94, 72.57, 62.58, 60.15, 58.44, 58.26, 55.28, 54.85, 53.17, 51.06, 50.41, 40.31, 37.01, 34.26, 30.66, 30.57, 30.06, 26.66, 25.90, 25.63, 25.57, 19.47, 18.82, 18.79, 18.76, 18.40, 17.64, 17.18, 16.76, 15.55, 15.21, 13.07, -2.74 ppm. **HRMS** (*m/z*): calculated for $C_{89}H_{115}N_7O_{13}SeSi_3Na^+$ $[M + Na]^+$: 1676.6918, found: 1676.6916.

Analytical data for 35a-1: (Yield = 62%) **TLC**: R_f = 0.2 (silica gel, EtOAc/hexane = 2:1). **$[\alpha]_D^{20}$** = -40.8 (*c* 0.5, MeOH). **^{1}H NMR** (400 MHz, CD_3OD) δ 7.71 – 7.67 (m, 8H), 7.44 – 7.33 (m, 12H), 6.98 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 6.70 – 6.55 (m, 5H), 5.79 – 5.75 (m, 1H), 5.19 (qd, *J* = 6.2, 2.8 Hz, 1H), 4.66

(dd, *J* = 8.9, 6.4 Hz, 1H), 4.61 – 4.54 (m, 2H), 4.45 (dd, *J* = 9.8, 4.9 Hz, 1H), 4.19 – 4.09 (m, 4H), 3.00 (dd, *J* = 13.9, 6.3 Hz, 1H), 2.80 (dd, *J* = 14.0, 9.0 Hz, 1H), 2.60 – 2.37 (m, 5H), 2.21 – 2.11 (m, 1H), 2.05 (d, *J* = 1.0 Hz, 3H), 1.91 (dd, *J* = 13.6, 6.8 Hz, 1H), 1.81 (d, *J* = 1.0 Hz, 4H), 1.74 (d, *J* = 7.2 Hz, 3H), 1.61 – 1.48 (m, 3H), 1.37 (d, *J* = 7.1 Hz, 3H), 1.07 (s, 9H), 1.06 (s, 9H), 1.03 (d, *J* = 6.4 Hz, 3H), 0.98 – 0.91 (m, 2H), 0.75 (dd, *J* = 11.6, 6.8 Hz, 6H), -0.02 (s, 9H) ppm. **^{13}C NMR** (100 MHz, CD_3OD): δ 175.43, 174.00, 172.64, 172.30, 171.75, 170.37, 170.35, 169.64, 167.67, 164.50, 154.39, 153.44, 150.83, 135.24, 135.22, 134.67, 132.88, 132.59, 130.89, 129.79, 129.76, 129.67, 129.38, 129.22, 128.76, 127.54, 127.47, 119.28, 119.03, 117.99, 72.75, 62.55, 60.15, 58.23, 55.35, 54.88, 52.71, 50.84, 50.74, 50.10, 37.03, 33.92, 30.70, 30.67, 29.76, 26.71, 25.93, 25.65, 25.61, 19.50, 18.85, 18.80, 18.78, 18.43, 17.21, 16.78, 15.27, 15.22, 13.10, 11.62, -2.70 ppm. **HRMS** (*m/z*): calculated for $C_{83}H_{109}N_7O_{13}Si_3Na^+$ $[M + Na]^+$: 1518.7283, found: 1518.7288.

Analytical data for largamide B (1a) (Yield = 56%, 2 steps) **$[\alpha]_D^{20}$** = -96.0 (*c* 0.12, MeOH). **^{1}H NMR** (500 MHz, DMF-d_7) δ 12.44 (s, 1H), 10.13 (s, 1H), 9.29 (s, 1H), 9.24 (s, 1H), 8.80 (s, 1H), 7.90 (d, *J* = 8.2 Hz, 1H), 7.84 (d, *J* = 8.5 Hz, 1H), 7.64 (d, *J* = 9.5 Hz, 1H), 7.49 (dd, *J* = 8.9, 4.0 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 6.73 (d, *J* = 8.3 Hz, 2H), 6.70 (d, *J* = 8.4 Hz, 2H), 6.56 (q, *J* = 7.2 Hz, 1H), 5.91 (s, 1H), 5.36 (qd, *J* = 6.4, 2.8 Hz, 1H), 4.79 (m, 1H), 4.73 (m, 1H), 4.65 (ddd, *J* = 9.5, 9.5, 5.5 Hz, 1H), 4.50 (ddd, *J* = 10.0, 9.0, 5.0 Hz, 1H), 4.30 (qd, *J* = 7.0, 2.5 Hz, 1H), 4.28 (dd, *J* = 8.5, 7.0 Hz, 1H), 3.04 (dd, *J* = -13.8, 4.7 Hz, 1H), 2.86 (dd, *J* = -13.8, 9.6 Hz, 1H), 2.58 – 2.42 (m, 5H), 2.19 – 2.14 (m, 1H), 2.12 (s, 3H), 2.03 (m, 1H), 1.89 (m, 1H), 1.80 (s, 3H), 1.77 (d, *J* = 7.2 Hz, 3H), 1.60 (m, 1H), 1.58 (m, 1H), 1.56 (m, 1H), 1.38 (d, *J* = 7.0 Hz, 3H), 1.17 (d, J = 6.1 Hz, 3H), 0.76 (d, *J* = 6.8 Hz, 3H), 0.73 (d, *J* = 6.8 Hz, 3H) ppm. **^{13}C NMR** (125 MHz, DMF-d_7) δ 175.70, 174.97, 172.59, 171.98, 171.61, 171.55, 170.34, 166.88, 163.98, 157.07, 156.44, 150.07, 133.05, 131.08, 130.90, 129.77, 129.18, 128.50, 119.62, 115.63, 115.59, 73.40, 58.41, 55.81, 55.64, 53.16, 51.05, 50.63, 38.05, 31.27, 31.12, 31.04, 27.78, 27.00, 26.98, 19.66, 19.51, 17.90, 16.68, 16.19, 12.62 ppm. **HRMS** (*m/z*): calculated for $C_{46}H_{61}N_7O_{13}Na^+$ $[M + Na]^+$: 942.4220, found: 942.4216.



Scheme 1: Synthesis of **10** via C-H functionalization.

Table 1: Optimization of reaction conditions for the synthesis of 10.

Entry	Alkyl Iodide	Ag(I) (eq)	Additive (eq)	Solvents	T (°C)	Y/C[a] (%)
1	**7**（3.0）	AgOAc（1.5）	None	Toluene	80	23/24
2	**8**（3.0）	AgOAc（1.5）	None	Toluene	80	29/30
3	**9**（3.0）	AgOAc（1.5）	None	Toluene	80	46/59
4	**8**（3.0）	Ag_2CO_3（1.25）	$(BnO)_2PO_2H$（0.5）	DCE/t-BuOH (2/1)	80	42/45
5	**9**（3.0）	AgOAc（1.5）	$(BnO)_2PO_2H$（0.5）	DCE/t-BuOH (2/1)	80	54/59
6	**9**（3.0）	Ag_2CO_3（1.5）	$(BnO)_2PO_2H$（0.5）	DCE/t-BuOH (2/1)	80	66/90
7	**9**（3.0）	Ag_2CO_3（1.5）	$(BnO)_2PO_2H$（0.5）	DCE/t-BuOH (2/1)	110	43/74

[a] Y refers to an isolated yield of product **10**; C refers conversion of **6**.

As shown in Scheme 1, we initially surveyed the alkylation of alanine derivative **6** with alkyl iodides **7**, **8**, **9** (entries 1-3) employing catalytic $Pd(OAc)_2$ in the presence of 1.5 equivalents of AgOAc with toluene as the solvent at 80 °C. Alkylation with both alkyl iodides **7** and **8** produced **10** in less than 30% yields and suffered from low conversion (entries 1, 2). When **9** was employed as the alkylating reagent, the reaction resulted in a significant improvement of the yield and conversion (entry 3). Upon examining the alkylation with alkyl iodide **8**, we were pleased to find when the reaction was conducted with Ag_2CO_3/$(BnO)_2PO_2H$ as additives in a dichloroethane–*tert*-butanol mixed solvent system, the desired L-ahppa derivative **10** could be obtained in 42% yield (entry 4). Further exploration of the reaction with alkyl iodide **9** under the identical conditions revealed the efficiency of the alkylation could be further improved (entries 5, 6). As shown in entry 6, the desired L-ahppa derivative **10** could be obtained in 66% yield. Further experimentation indicated the yield of the alkylation also depended on the reaction temperature. Thus, when the reaction temperature was raised from 80 °C to 110 °C, the alkylation resulted in reduced yield (entry 7).

Table 2: Comparison of ^{1}H NMR Data of Largamide B (Reported Data and Synthetic 1a)

Amino unit	No.	δ H in ppm (J in Hz)		$\Delta\delta$H (ppm)
		Reported Data (500 MHz)	Synthetic 1a (500 MHz)	
Ahppa	1			
	2	4.65, ddd (9.5, 9.5, 5.5)	4.65, ddd (9.5, 9.5, 5.5)	0.00
	3	1.88, m; 1.56, m	1.89, m; 1.56, m	+0.01
	4	1.60, m; 1.58, m	1.60, m; 1.58, m	0.00
	5	2.56, m; 2.48, m	2.56, m; 2.48, m	0.00
	6			
	7/11	7.01, d (8.5)	7.01, d (8.4)	0.00
	8/10	6.73, d (8.5)	6.73, d (8.3)	0.00
	9			
	OH	9.29, s	9.24, s	-0.05
	NH	7.65, d (9.5)	7.64, d (9.5)	-0.01
Glu	1			
	2	4.50, ddd (10.0, 9.0, 5.0)	4.50, ddd (10.0, 9.0, 5.0)	0.00
	3	2.51, m; 2.18, m	2.51, m; 2.17, m	-0.01
	4	2.59, m; 2.47, m	2.59, m; 2.47, m	0.00
	5			
	OH	12.51, br s	12.44, br s	-0.07
	NH	7.50, d (9.0)	7.49, dd (8.9, 4.0)	-0.01
Abu	1			
	2			
	3	6.55, qd (7.0, 1.0)	6.56, q (7.2)	+0.01
	4	1.77, d (7.0)	1.77, d (7.2)	0.00
	NH	10.16, s	10.13, s	-0.03
Ala	1			
	2	4.30, qd (7.0, 2.5)	4.30, qd (7.0, 2.5)	0.00
	3	1.37, d (7.0)	1.38, d (7.0)	+0.01
	NH	8.83, d (2.5)	8.80, s	-0.03
Thr	1			
	2	4.73, dd (8.5, 2.5)	4.73, m	0.00
	3	5.37, qd (6.5, 2.5)	5.36, qd (6.4, 2.8)	-0.01
	4	1.18, d (6.5)	1.17, d (6.1)	-0.01

	NH	7.96, d (8.5)	7.84, d (8.5)	-0.12
	1			
Tyr	2	4.79, ddd (10.5, 8.5, 4.5)	4.79, m	0.00
	3	3.07, dd (-14.0, 4.5); 2.82, dd(-14.0, 10.0)	3.04, dd (-13.8, 4.7); 2.86, dd(-13.8, 9.6)	-0.03; +0.04
	4			
	5/9	7.11, d (8.5)	7.09, d (8.4)	-0.02
	6/8	6.70, d (8.5)	6.70, d (8.4)	0.00
	7			
	OH	9.34, s	9.29, s	-0.05
	NH	8.09, d (8.5)	7.90, d (8.2)	-0.19
Val	1			
	2	4.28, dd (8.5, 7.0)	4.28, dd (8.5, 7.0)	0.00
	3	2.01, m	2.03, m	+0.02
	4	0.74, d (7.0)	0.76, d (6.8)	+0.02
	5	0.71, d (7.0)	0.73, d (6.8)	+0.02
	NH	7.25, d (8.5)	7.25, d (8.5)	0.00
Tig/Sen	1			
	2		5.91, s	-
	3	6.42, qq (6.7, 1.5)		-
	4	1.71, dq (6.7, 1.5)	2.12, s	+0.41
	5	1.79, br s	1.80, s	+0.01

Table 3: Comparison of ^{13}C NMR Data of Largamide B (Reported Data and Synthetic 1a)

Amino unit	No.	δ C in ppm		ΔδH (ppm)
		Reported Data (500 MHz)	Synthetic 1a (500 MHz)	
Ahppa	1	171.7, qC	171.61	0.05
	2	51.1, CH	51.05	-0.05
	3	31.2, CH_2	31.12	-0.08
	4	27.9, CH_2	27.78	-0.12
	5	34.7, CH_2	*	-
	6	133.1, qC	133.05	-0.05
	7/11	129.9, CH	129.77	-0.13
	8/10	115.7, CH	115.63	-0.07
	9	156.5, qC	156.44	-0.06
	OH			
	NH			
Glu	1	171.6, qC	171.55	-0.05
	2	53.2, CH	53.16	-0.04
	3	27.0, CH_2	27.00	0.00
	4	31.0, CH_2	31.04	0.04
	5	175.1, qC	174.97	-0.13
	OH			
	NH			
Abu	1	164.1, qC	163.98	-0.12
	2	131.1, qC	131.08	-0.02
	3	129.2, CH	129.18	-0.02
	4	12.7, CH_3	12.62	-0.08
	NH			
Ala	1	175.8, qC	175.70	-0.10
	2	50.7, CH	50.63	-0.07
	3	16.8, CH_3	16.68	-0.12
	NH			
Thr	1	170.4, qC	170.34	-0.06
	2	56.0, CH	55.81	-0.19
	3	73.4, CH	73.40	0.00
	4	16.2, CH_3	16.19	-0.01
	NH			
Tyr	1	172.8, qC	172.59	-0.21
	2	55.5, CH	55.64	0.14
	3	38.2, CH_2	38.05	-0.15
	4	128.6, qC	128.50	-0.10

	5/9	131.0, CH	130.90	-0.10
	6/8	115.6, CH	115.59	-0.01
	7	157.1, qC	157.07	-0.03
	OH			
	NH			
Val	1	172.0, qC	171.98	-0.02
	2	59.2, CH	58.41	0.79
	3	31.6, CH	31.27	-0.33
	4	19.8, CH_3	19.66	-0.14
	5	18.3, CH_3	17.9	-0.40
	NH			
Tig/Sen	1	169.3, qC	166.88	-2.42
	2	132.7, qC	150.07	17.37
	3	130.4, CH	119.62	-10.78
	4	13.8, CH_3	26.98	13.18
	5	12.6, CH_3	19.51	6.91

Table 4: Comparison of ^{1}H NMR Data of Largamide B (Reported Data and Synthetic 1b)

Amino unit	No.	δ H in ppm (J in Hz)		$\Delta\delta$H (ppm)
		Reported Data (500 MHz)	Synthetic 1b (500 MHz)	
Ahppa	1			
	2	4.65, ddd (9.5, 9.5, 5.5)	4.65, ddd (9.5, 9.5, 5.5)	0.00
	3	1.88, m; 1.56, m	1.89, m; 1.56, m	+0.01
	4	1.60, m; 1.58, m	1.60, m; 1.58, m	0.00
	5	2.56, m; 2.48, m	2.56, m; 2.48, m	0.00
	6			
	7/11	7.01, d (8.5)	7.01, d (8.4)	0.00
	8/10	6.73, d (8.5)	6.73, d (8.4)	0.00
	9			
	OH	9.29, s	9.24, s	-0.05
	NH	7.65, d (9.5)	7.64, d (9.4)	-0.01
Glu	1			
	2	4.50, ddd (10.0, 9.0, 5.0)	4.50, ddd (10.0, 9.0, 5.0)	0.00
	3	2.51, m; 2.18, m	2.51, m; 2.17, m	-0.01
	4	2.59, m; 2.47, m	2.59, m; 2.47, m	0.00
	5			
	OH	12.51, br s	12.42, br s	-0.09
	NH	7.50, d (9.0)	7.49, d (8.7)	-0.01
Abu	1			
	2			
	3	6.55, qd (7.0, 1.0)	6.56, q (7.1)	+0.01
	4	1.77, d (7.0)	1.77, d (7.2)	0.00
	NH	10.16, s	10.13, s	-0.03
Ala	1			
	2	4.30, qd (7.0, 2.5)	4.30, qd (7.0, 2.5)	0.00
	3	1.37, d (7.0)	1.38, d (7.0)	+0.01
	NH	8.83, d (2.5)	8.80, s	-0.03
Thr	1			
	2	4.73, dd (8.5, 2.5)	4.73, m	0.00
	3	5.37, qd (6.5, 2.5)	5.37, qd (6.3, 2.9)	0.00
	4	1.18, d (6.5)	1.17, d (6.3)	-0.01
	NH	7.96, d (8.5)	7.87, d (8.5)	-0.09
	1			
Tyr	2	4.79, ddd (10.5, 8.5,	4.79, m	0.00

		4.5)		
	3	3.07, dd (-14.0, 4.5); 2.82, dd(-14.0, 10.0)	3.06, dd (-13.9, 4.7); 2.83, dd(-13.8, 9.9)	-0.01; +0.01
	4			
	5/9	7.11, d (8.5)	7.11, d (8.4)	0.00
	6/8	6.70, d (8.5)	6.70, d (8.4)	0.00
	7			
	OH	9.34, s	9.29, s	-0.05
	NH	8.09, d (8.5)	8.10, s	+0.01
Val	1			
	2	4.28, dd (8.5, 7.0)	4.28, dd (8.5, 7.0)	0.00
	3	2.01, m	2.02, m	+0.01
	4	0.74, d (7.0)	0.75, d (6.8)	+0.01
	5	0.71, d (7.0)	0.72, d (6.7)	+0.01
	NH	7.25, d (8.5)	7.25, d (8.5)	0.00
Tig	1			
	2			
	3	6.42, qq (6.7, 1.5)	6.42, qq (6.7, 1.5)	0.00
	4	1.71, dq (6.7, 1.5)	1.71, dq (6.7, 1.5)	0.00
	5	1.79, br s	1.80, s	+0.01

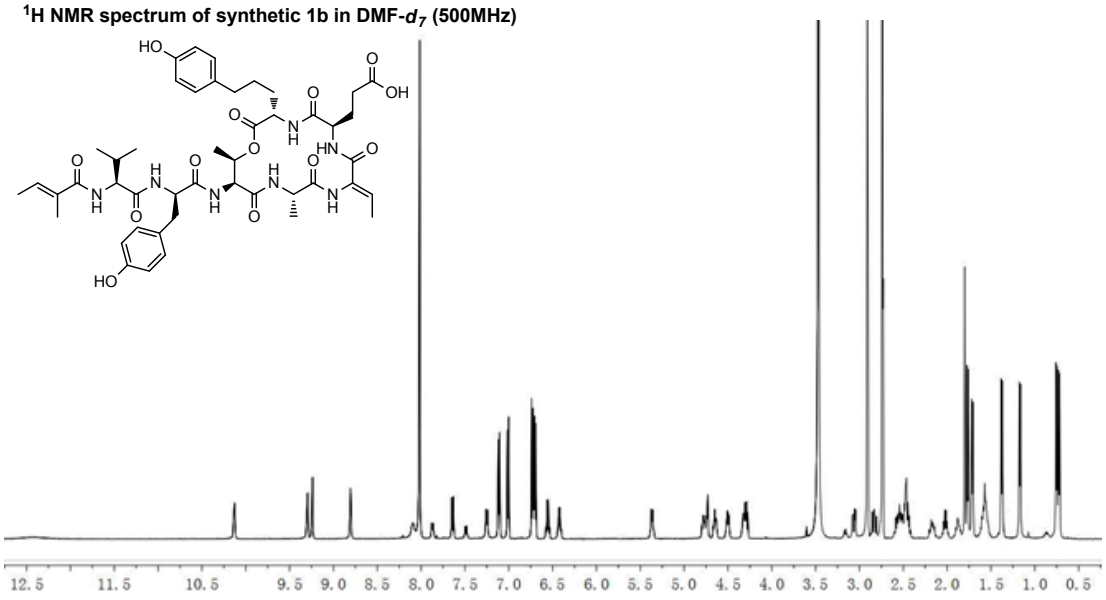
Table 5: Comparison of ^{13}C NMR Data of Largamide B (Reported Data and Synthetic 1b)

Amino unit	No.	δ C in ppm		$\Delta\delta$H (ppm)
		Reported Data (500 MHz)	Synthetic 1b (500 MHz)	
Ahppa	1	171.7, qC	171.75	0.05
	2	51.1, CH	51.15	0.05
	3	31.2, CH_2	31.26	0.06
	4	27.9, CH_2	27.93	0.03
	5	34.7, CH_2	34.63	-0.07
	6	133.1, qC	133.18	0.08
	7/11	129.9, CH	129.90	0.00
	8/10	115.7, CH	115.75	0.05
	9	156.5, qC	156.56	0.06
	OH			
	NH			
Glu	1	171.6, qC	171.72	0.12
	2	53.2, CH	53.19	-0.01
	3	27.0, CH_2	27.04	0.04
	4	31.0, CH_2	31.10	0.10
	5	175.1, qC	175.18	0.08
	OH			
	NH			
Abu	1	164.1, qC	164.04	-0.06
	2	131.1, qC	131.17	0.07
	3	129.2, CH	129.28	0.08
	4	12.7, CH_3	12.76	0.06
	NH			
Ala	1	175.8, qC	175.84	0.04
	2	50.7, CH	50.65	-0.05
	3	16.8, CH_3	16.74	-0.06
	NH			
Thr	1	170.4, qC	170.35	-0.05
	2	56.0, CH	55.88	-0.12
	3	73.4, CH	73.46	0.06
	4	16.2, CH_3	16.27	0.07
	NH			
Tyr	1	172.8, qC	172.71	-0.09
	2	55.5, CH	55.59	0.09
	3	38.2, CH_2	38.16	-0.04

	4	128.6, qC	128.67	0.07
	5/9	131.0, CH	131.03	0.03
	6/8	115.6, CH	115.67	0.07
	7	157.1, qC	157.20	0.10
	OH			
	NH			
Val	1	172.0, qC	172.10	0.10
	2	59.2, CH	59.29	0.09
	3	31.6, CH	31.66	0.06
	4	19.8, CH_3	19.81	0.01
	5	18.3, CH_3	18.31	0.01
	NH			
Tig	1	169.3, qC	169.38	0.08
	2	132.7, qC	132.83	0.13
	3	130.4, CH	130.44	0.04
	4	13.8, CH_3	13.86	0.06
	5	12.6, CH_3	12.59	-0.01

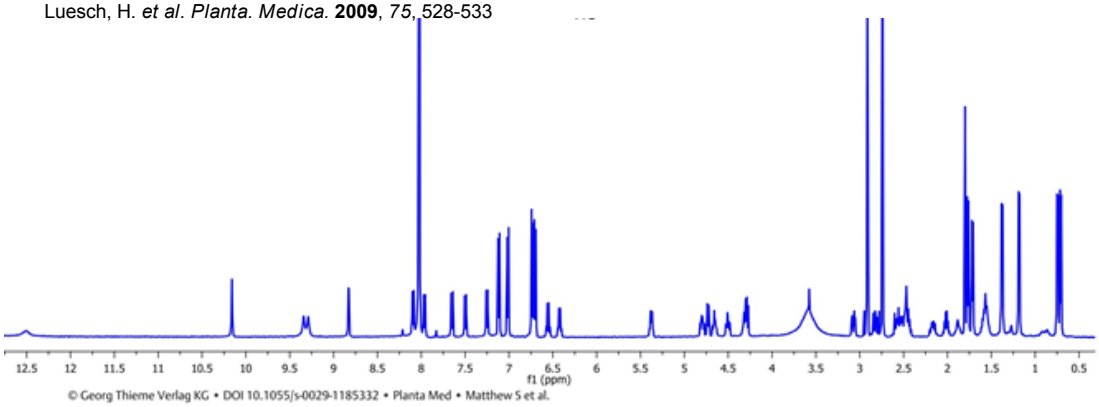
Figure 1: Comparison of ^{1}H NMR Spectra of Nature and Synthetic Largamide B (1a and 1b)

^{1}H NMR spectrum of synthetic 1b in DMF-d_7 (500MHz)



^{1}H NMR spectrum of natural product in DMF-d_7 (500MHz)

Luesch, H. *et al*. *Planta. Medica.* **2009**, *75*, 528-533



^{1}H NMR spectrum of synthetic 1a in DMF-d_7 (500MHz)

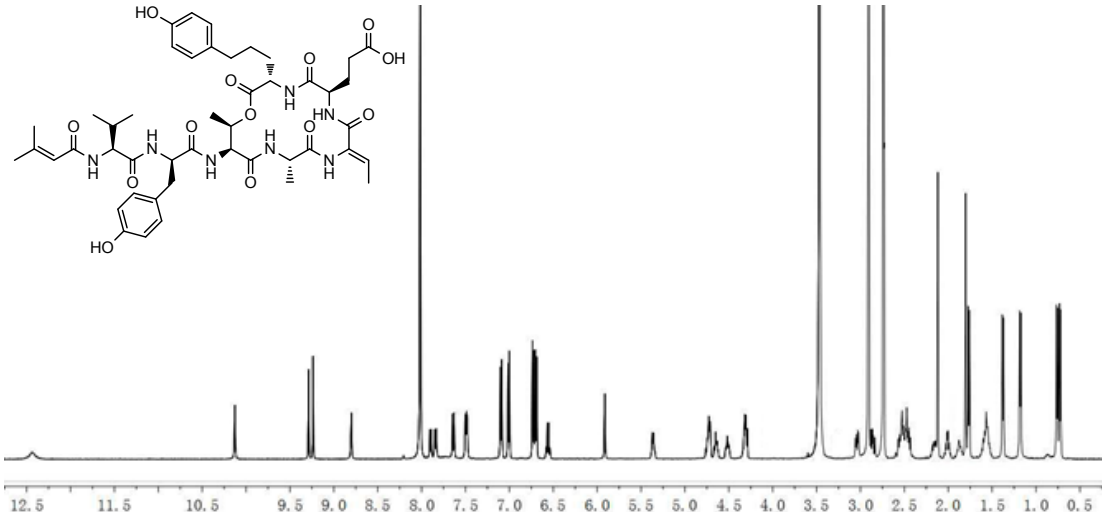
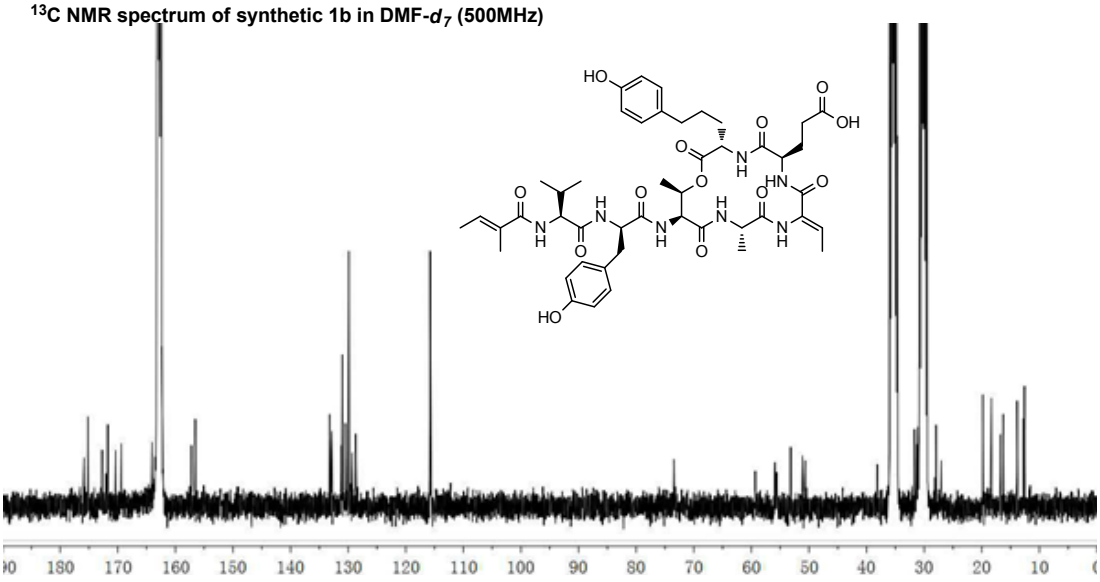


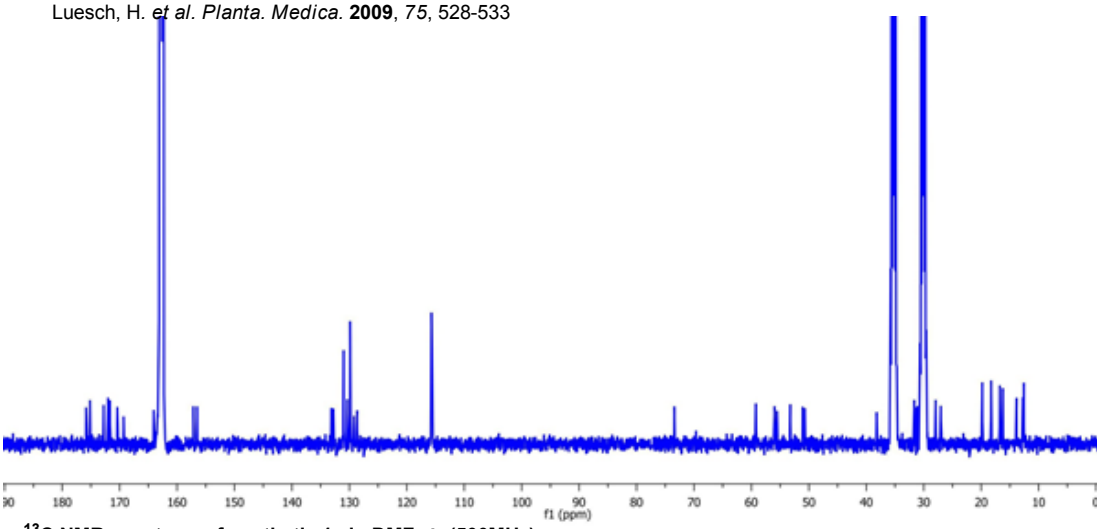
Figure 2: Comparison of ^{13}C NMR Spectra of Nature and Synthetic Largamide B (1a and 1b)

^{13}C NMR spectrum of synthetic 1b in DMF-d_7 (500MHz)

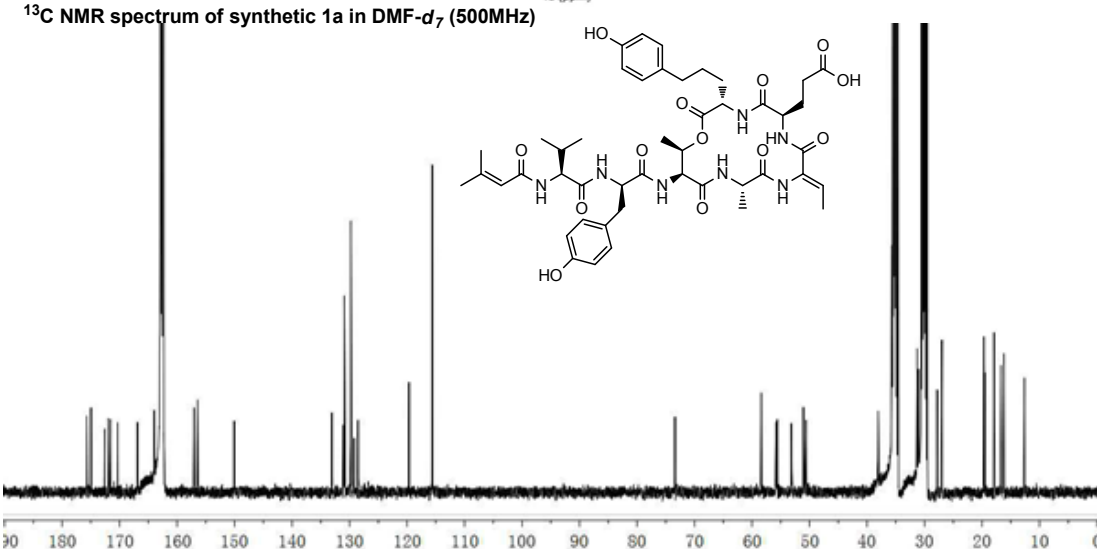


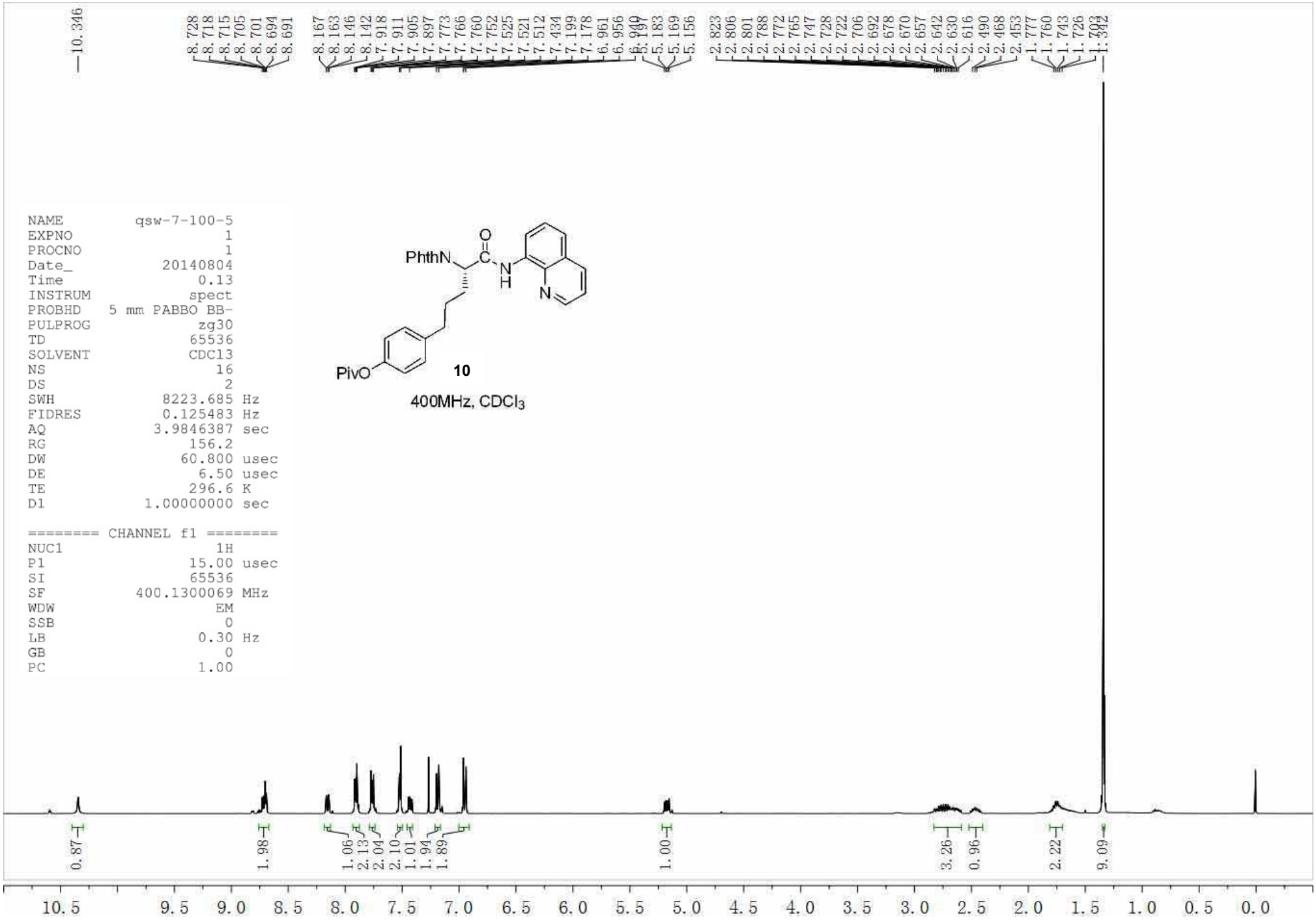
^{13}C NMR spectrum of natural product in DMF-d_7 (500MHz)

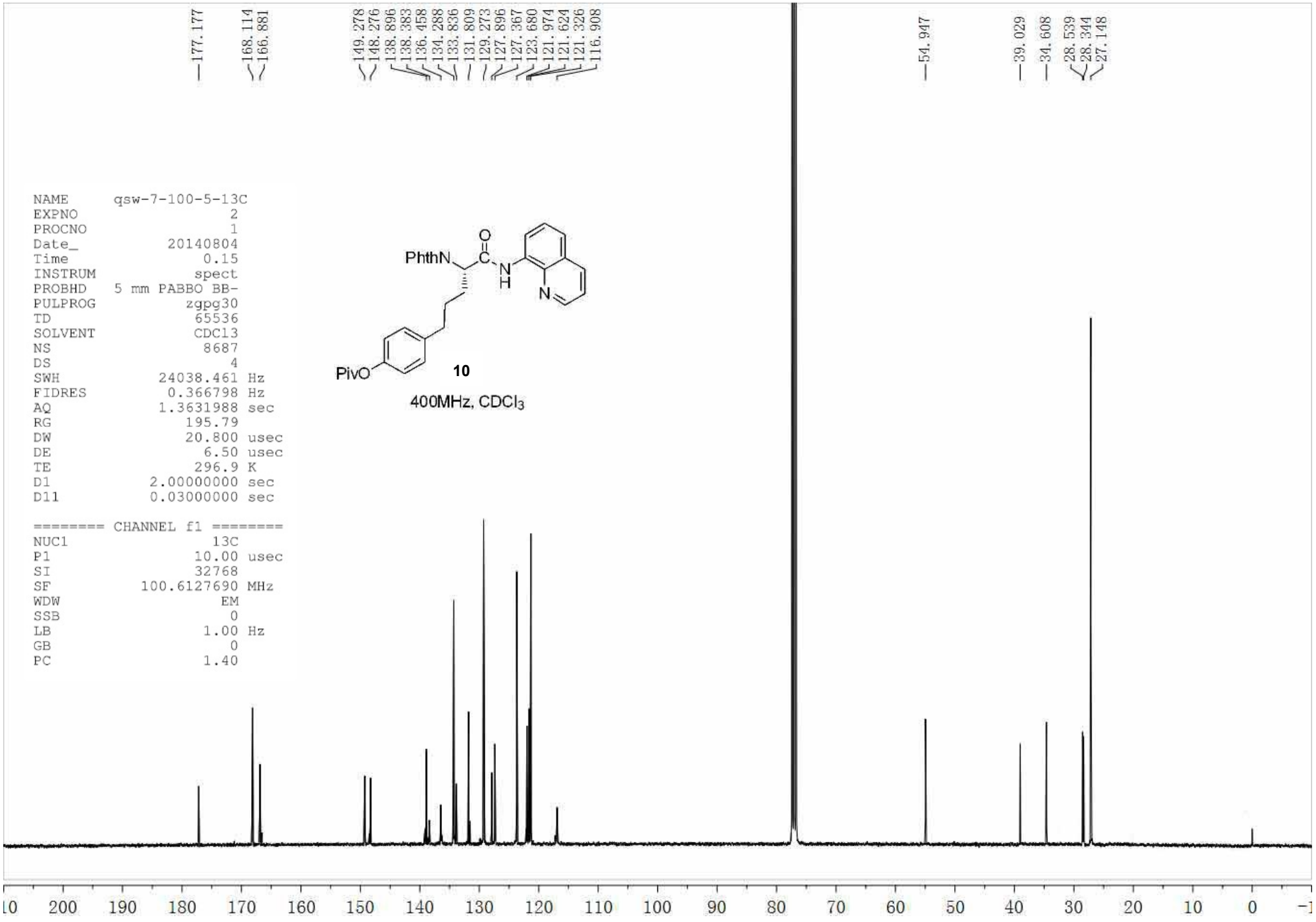
Luesch, H. *et al. Planta. Medica.* **2009**, *75*, 528-533

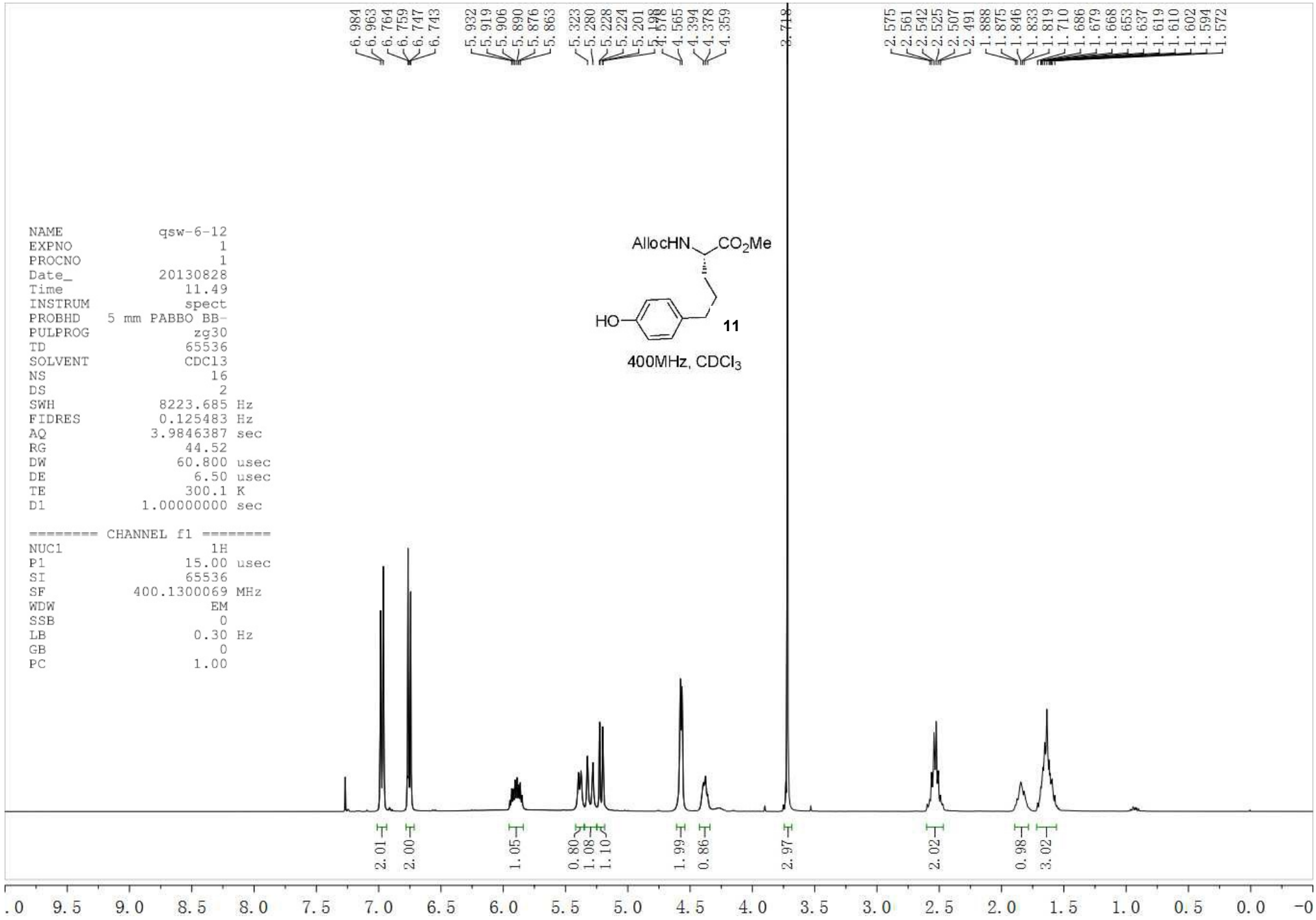


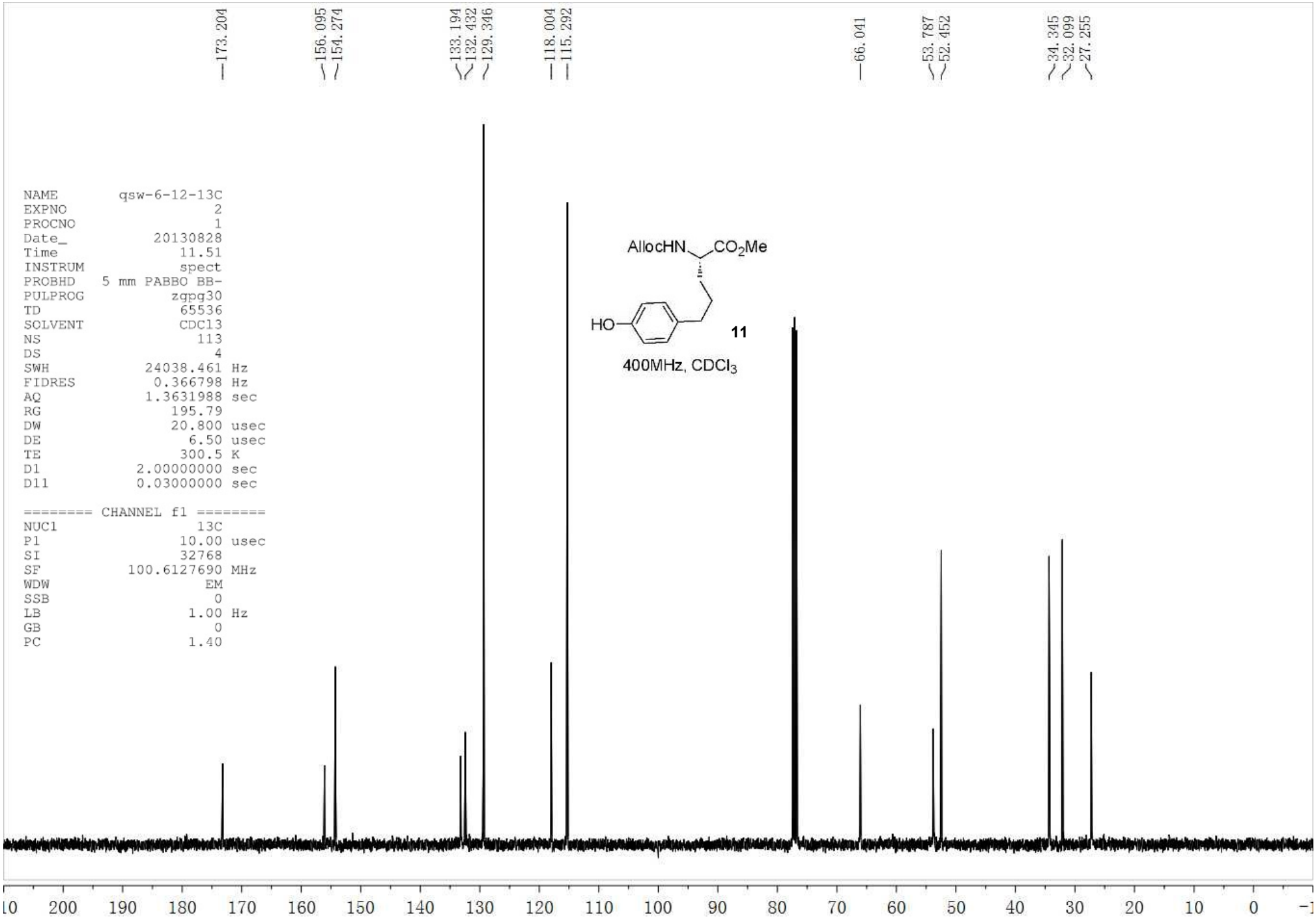
^{13}C NMR spectrum of synthetic 1a in DMF-d_7 (500MHz)

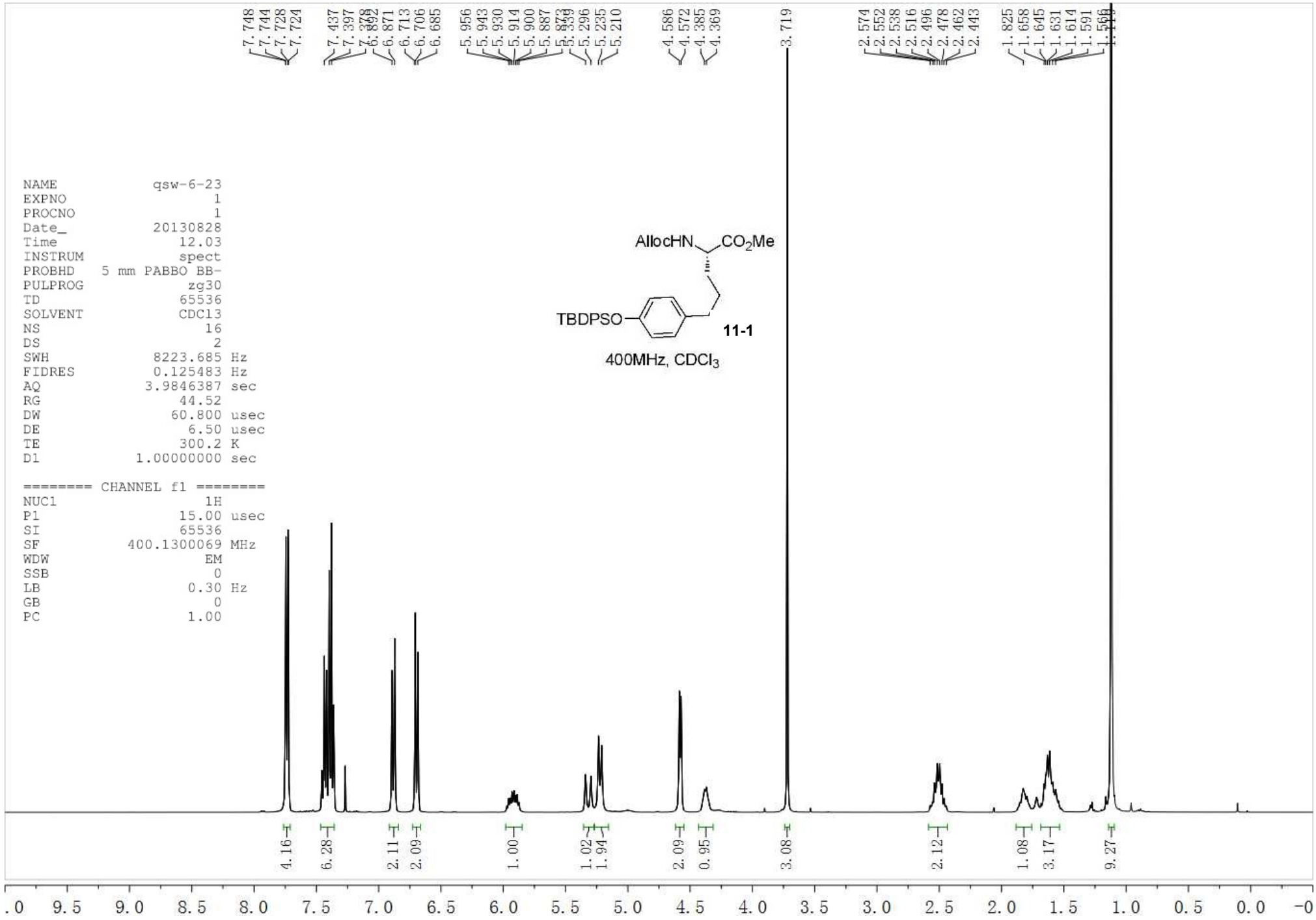


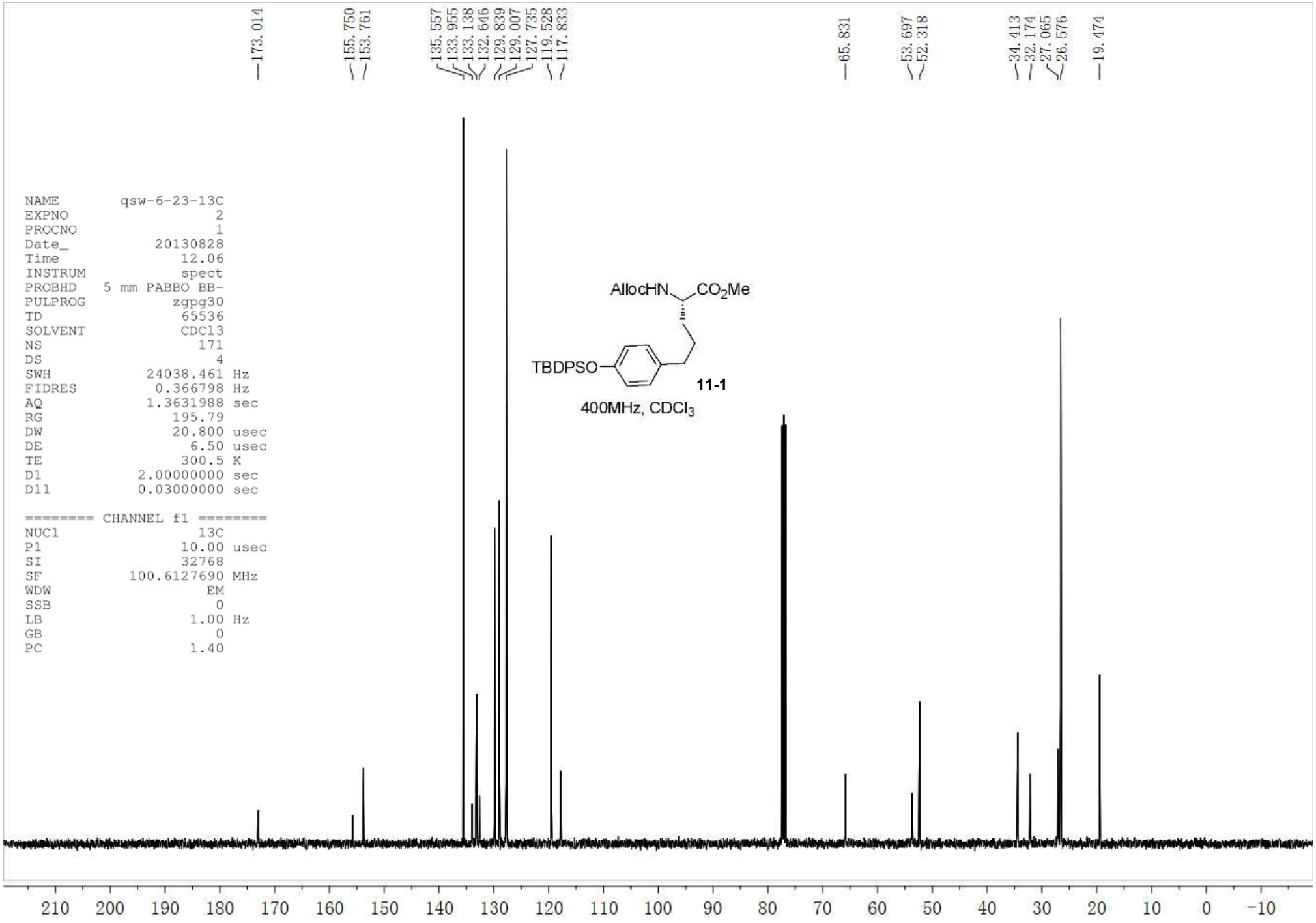

10.346
8.728 8.718 8.715 8.705 8.701 8.694 8.691
8.167 8.163 8.146 8.142 7.918 7.911 7.905 7.897 7.773 7.766 7.760 7.752 7.525 7.521 7.512 7.434 7.199 7.178 6.961 6.956 6.940 6.937 5.183 5.169 5.156
2.823 2.806 2.801 2.788 2.772 2.765 2.747 2.728 2.722 2.706 2.692 2.678 2.670 2.657 2.642 2.630 2.616 2.490 2.468 2.453
1.777 1.760 1.743 1.726 1.703 1.342
NAME qsw-7-100-5
EXPNO 1
PROCNO 1
Date_ 20140804
Time 0.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 156.2
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
PhthN
O
C
N
H
N
PivO
10
400MHz, CDCl3
0.87
1.98
1.06
2.13
2.04
2.10
1.01
1.94
1.89
1.00
3.26
0.96
2.22
9.09
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

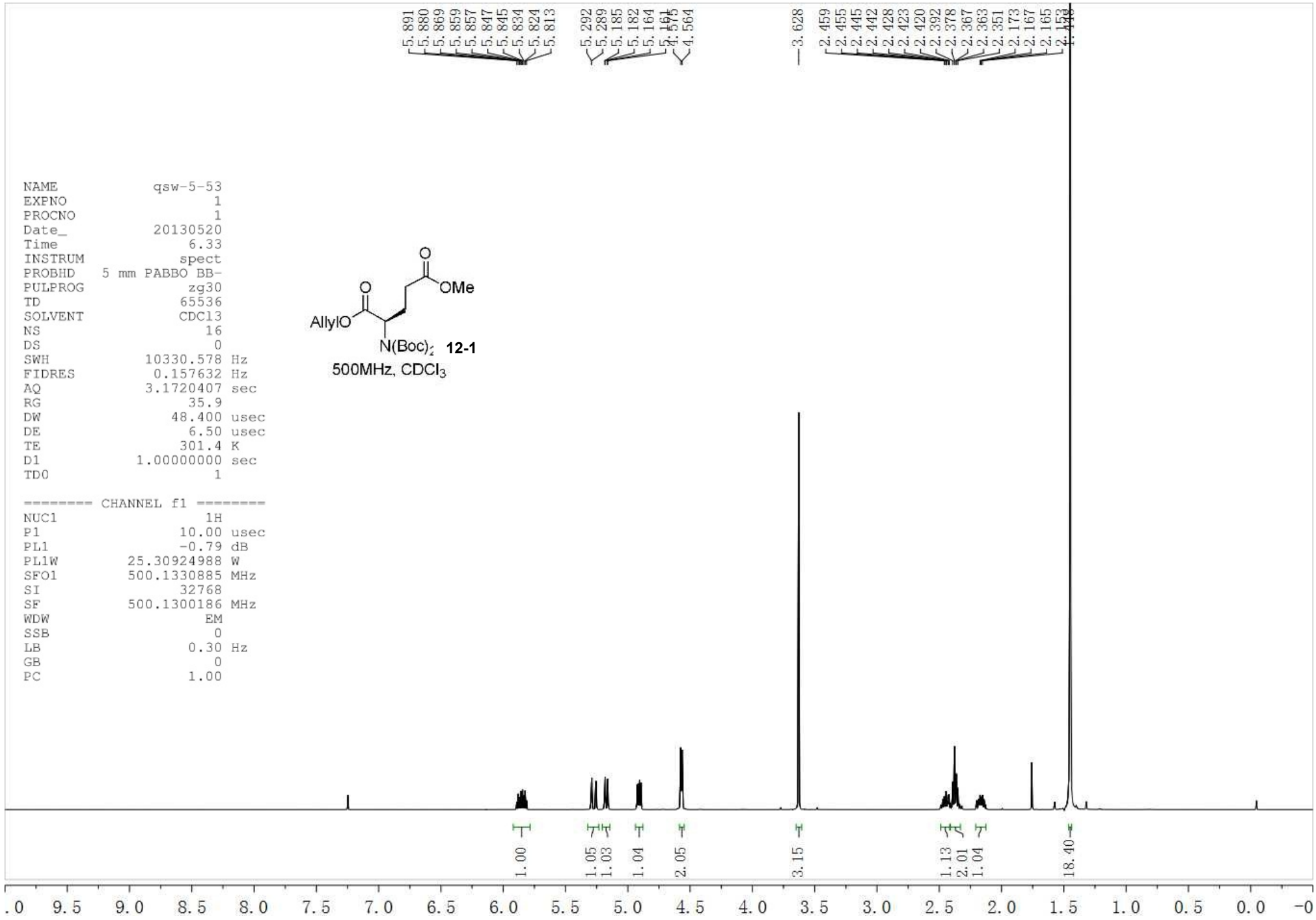

177.177
168.114
166.881
149.278
148.276
138.896
138.383
136.458
134.288
133.836
131.809
129.273
127.896
127.367
123.680
121.974
121.624
121.326
116.908
54.947
39.029
34.608
28.539
28.344
27.148
NAME qsw-7-100-5-13C
EXPNO 2
PROCNO 1
Date_ 20140804
Time 0.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8687
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
PhthN
O
C
N
H
N
PivO
10
400MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

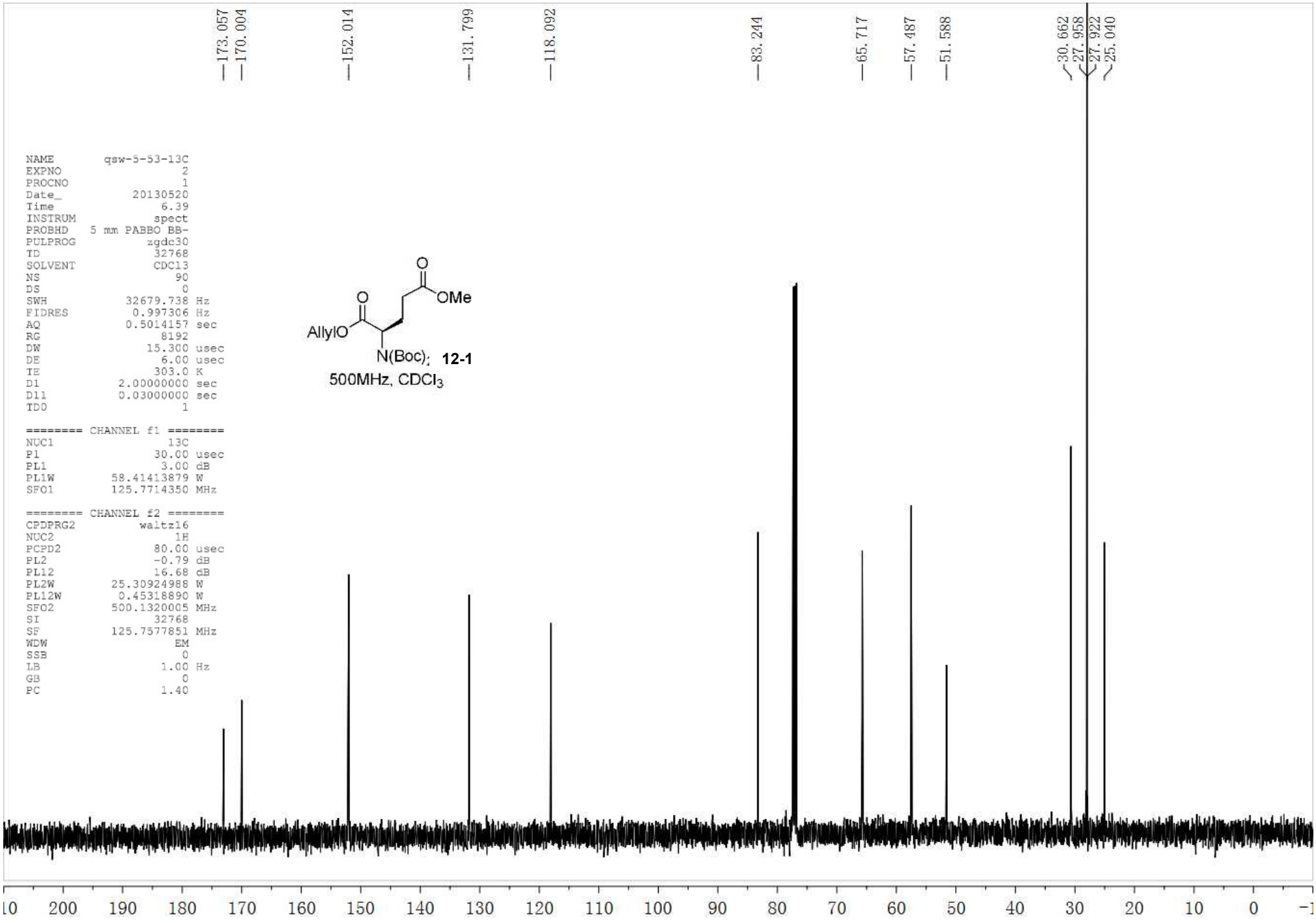

6.984
6.963
6.764
6.759
6.747
6.743
5.932
5.919
5.906
5.890
5.876
5.863
5.323
5.280
5.228
5.224
5.201
4.565
4.394
4.378
4.359
3.718
2.575
2.561
2.542
2.525
2.507
2.491
1.888
1.875
1.846
1.833
1.819
1.710
1.686
1.679
1.668
1.653
1.637
1.619
1.610
1.602
1.594
1.572
NAME qsw-6-12
EXPNO 1
PROCNO 1
Date_ 20130828
Time 11.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 44.52
DW 60.800 usec
DE 6.50 usec
TE 300.1 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AllocHN
CO2Me
HO
11
400MHz, CDCl3
2.01
2.00
1.05
0.80
1.08
1.10
1.99
0.86
2.97
2.02
0.98
3.02
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0

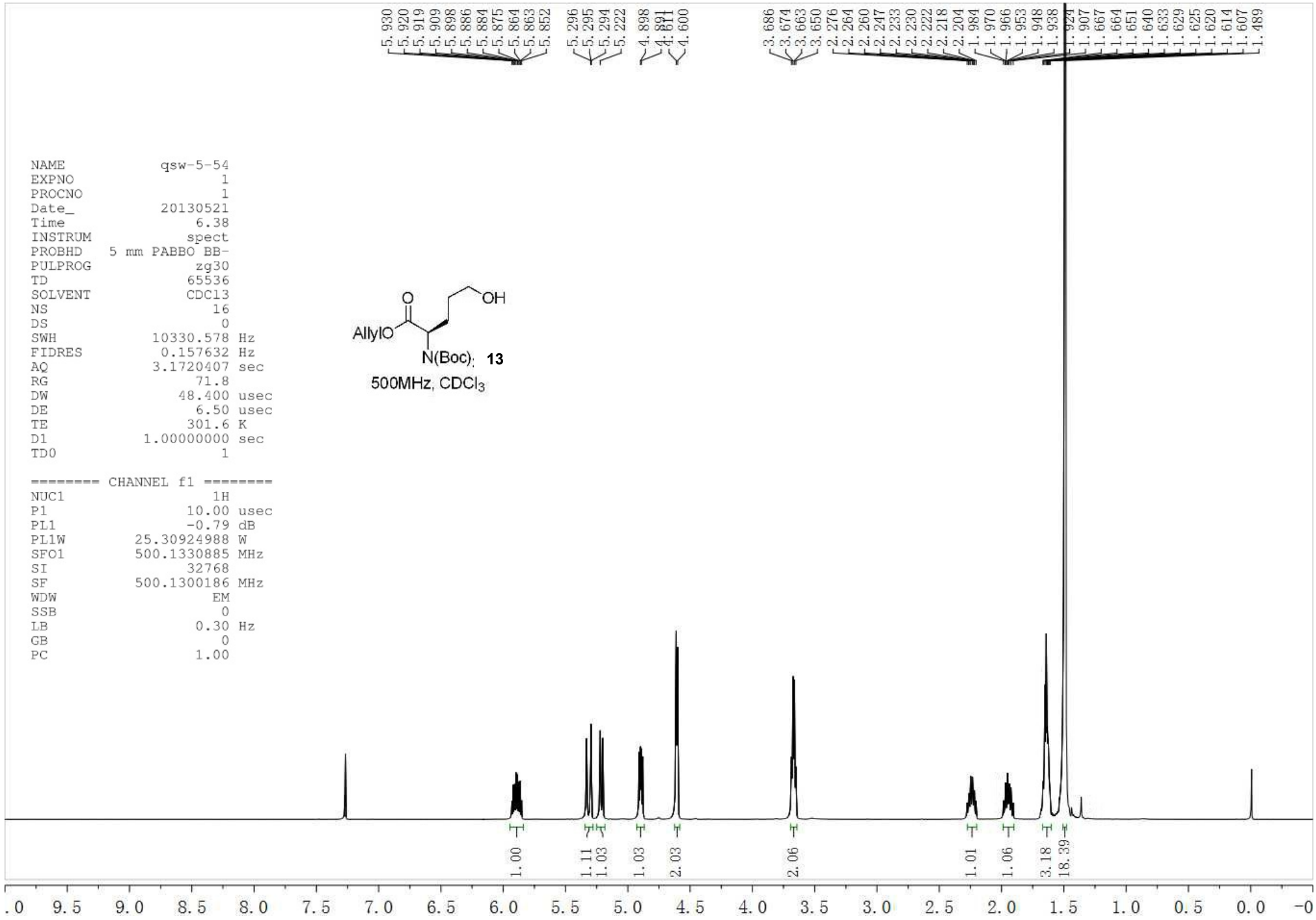

173.204
156.095
154.274
133.194
132.432
129.346
118.004
115.292
66.041
53.787
52.452
34.345
32.099
27.255
NAME qsw-6-12-13C
EXPNO 2
PROCNO 1
Date_ 20130828
Time 11.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 113
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 300.5 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AllocHN
CO2Me
HO
11
400MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

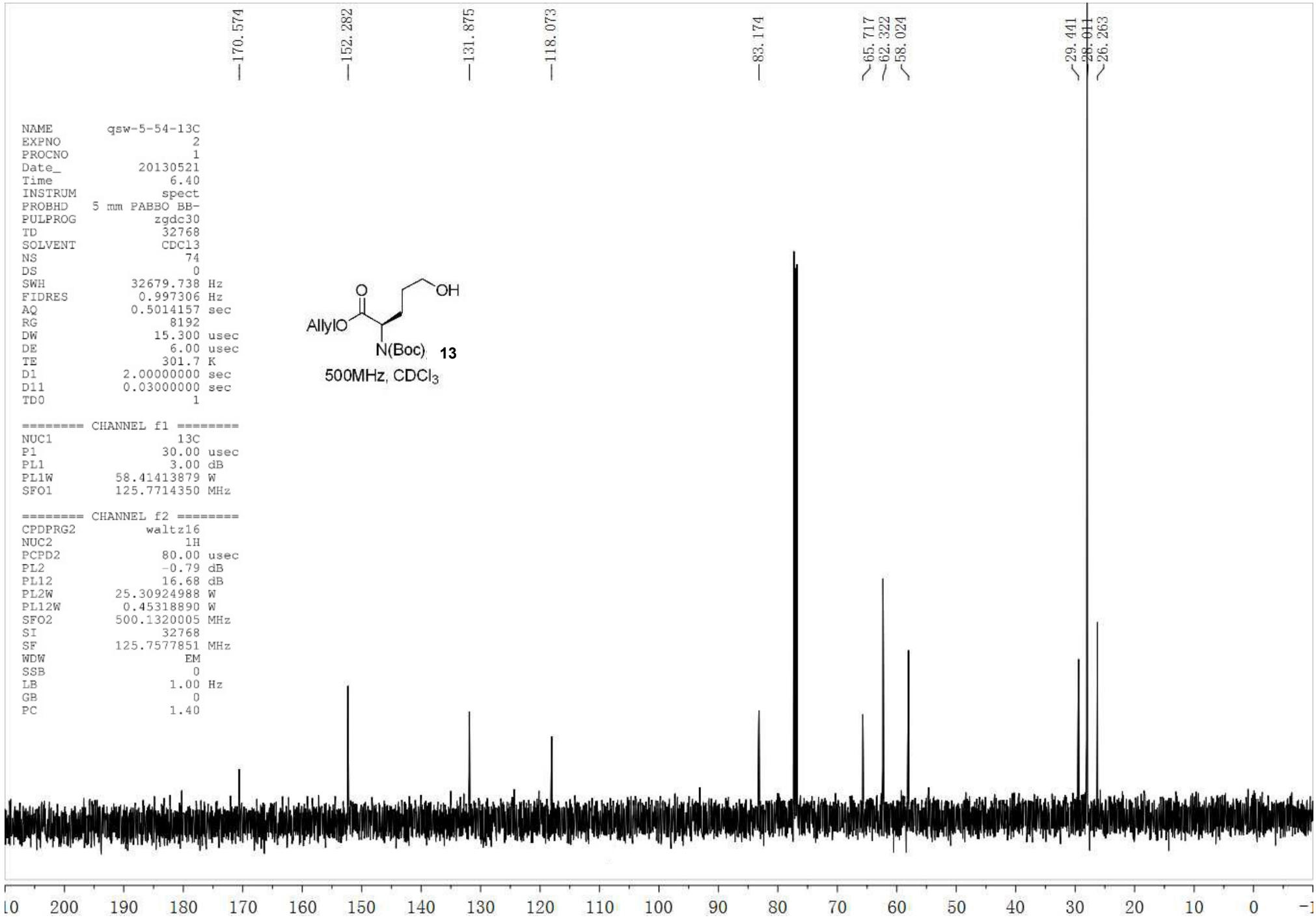

7.748
7.744
7.728
7.724
7.437
7.397
6.871
6.713
6.706
6.685
5.956
5.943
5.930
5.914
5.900
5.887
5.296
5.235
5.210
4.586
4.572
4.385
4.369
3.719
2.574
2.552
2.538
2.516
2.496
2.478
2.462
2.443
1.825
1.658
1.645
1.631
1.614
1.591
1.566
NAME qsw-6-23
EXPNO 1
PROCNO 1
Date_ 20130828
Time 12.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 44.52
DW 60.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AllocHN
CO2Me
TBDPSO
11-1
400MHz, CDCl3
4.16
6.28
2.11
2.09
1.00
1.02
1.94
2.09
0.95
3.08
2.12
1.08
3.17
9.27
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

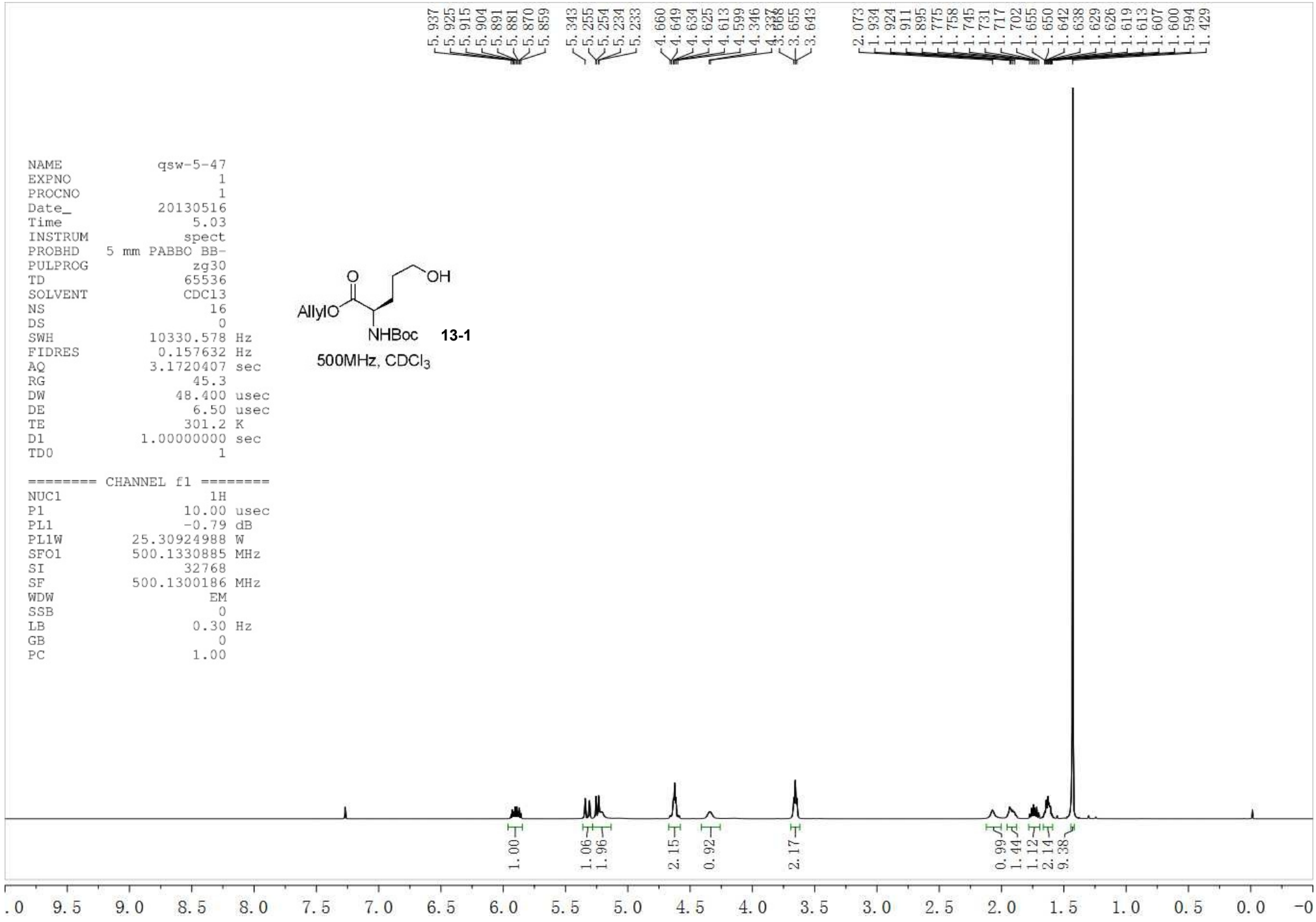

173.014
155.750
153.761
135.557
133.955
133.138
132.646
129.839
129.007
127.735
119.528
117.833
65.831
53.697
52.318
34.413
32.174
27.065
26.576
19.474
NAME qsw-6-23-13C
EXPNO 2
PROCNO 1
Date_ 20130828
Time 12.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 171
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 300.5 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AllocHN
CO2Me
TBDPSO
11-1
400MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

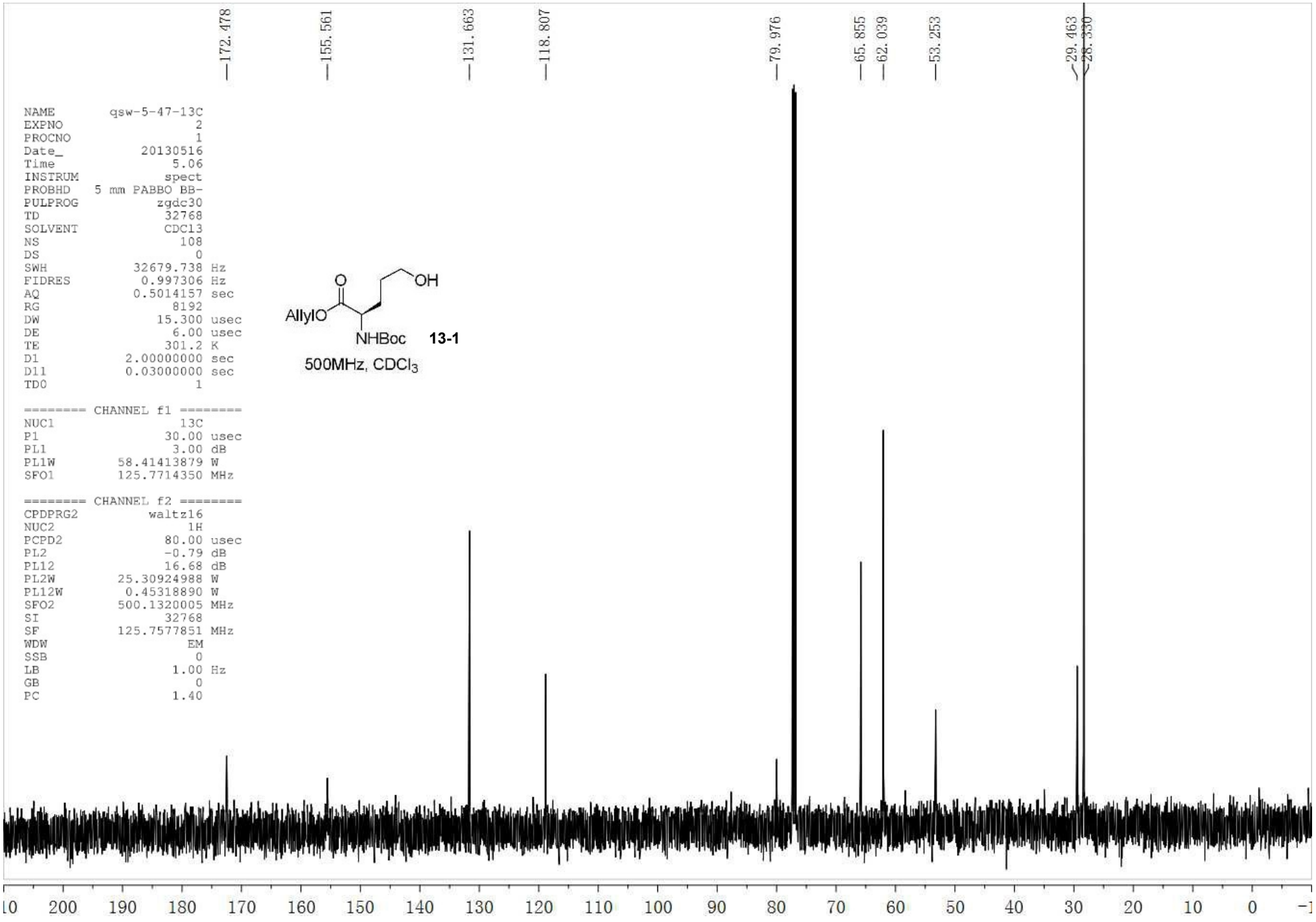

5.891
5.880
5.869
5.859
5.857
5.847
5.845
5.834
5.824
5.813
5.292
5.289
5.185
5.182
5.164
4.575
4.564
3.628
2.459
2.455
2.445
2.442
2.428
2.423
2.420
2.392
2.378
2.367
2.363
2.351
2.173
2.167
2.165
NAME qsw-5-53
EXPNO 1
PROCNO 1
Date_ 20130520
Time 6.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 35.9
DW 48.400 usec
DE 6.50 usec
TE 301.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AllylO
OMe
N(Boc)2
12-1
500MHz, CDCl3
1.00
1.05
1.03
1.04
2.05
3.15
1.13
2.01
1.04
18.40
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0

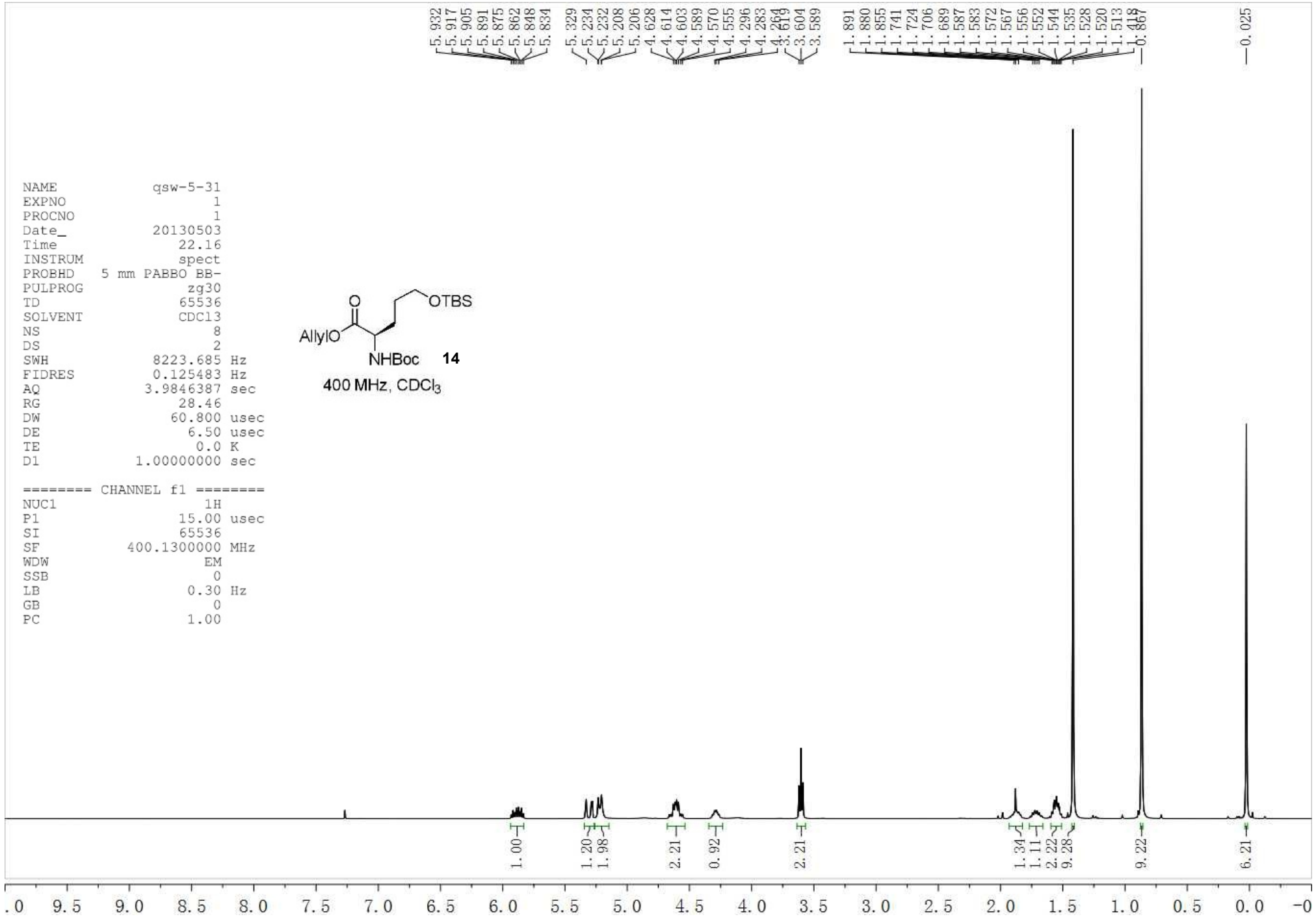

173.057
170.004
152.014
131.799
118.092
83.244
65.717
57.487
51.588
30.662
27.958
27.922
25.040
NAME qsw-5-53-13C
EXPNO 2
PROCNO 1
Date_ 20130520
Time 6.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT CDC13
NS 90
DS 0
SWH 32679.738 Hz
FIDRES 0.997306 Hz
AQ 0.5014157 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 30.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7714350 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.79 dB
PL12 16.68 dB
PL2W 25.30924988 W
PL12W 0.45318890 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577851 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
OMe
AllylO
N(Boc)
12-1
500MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

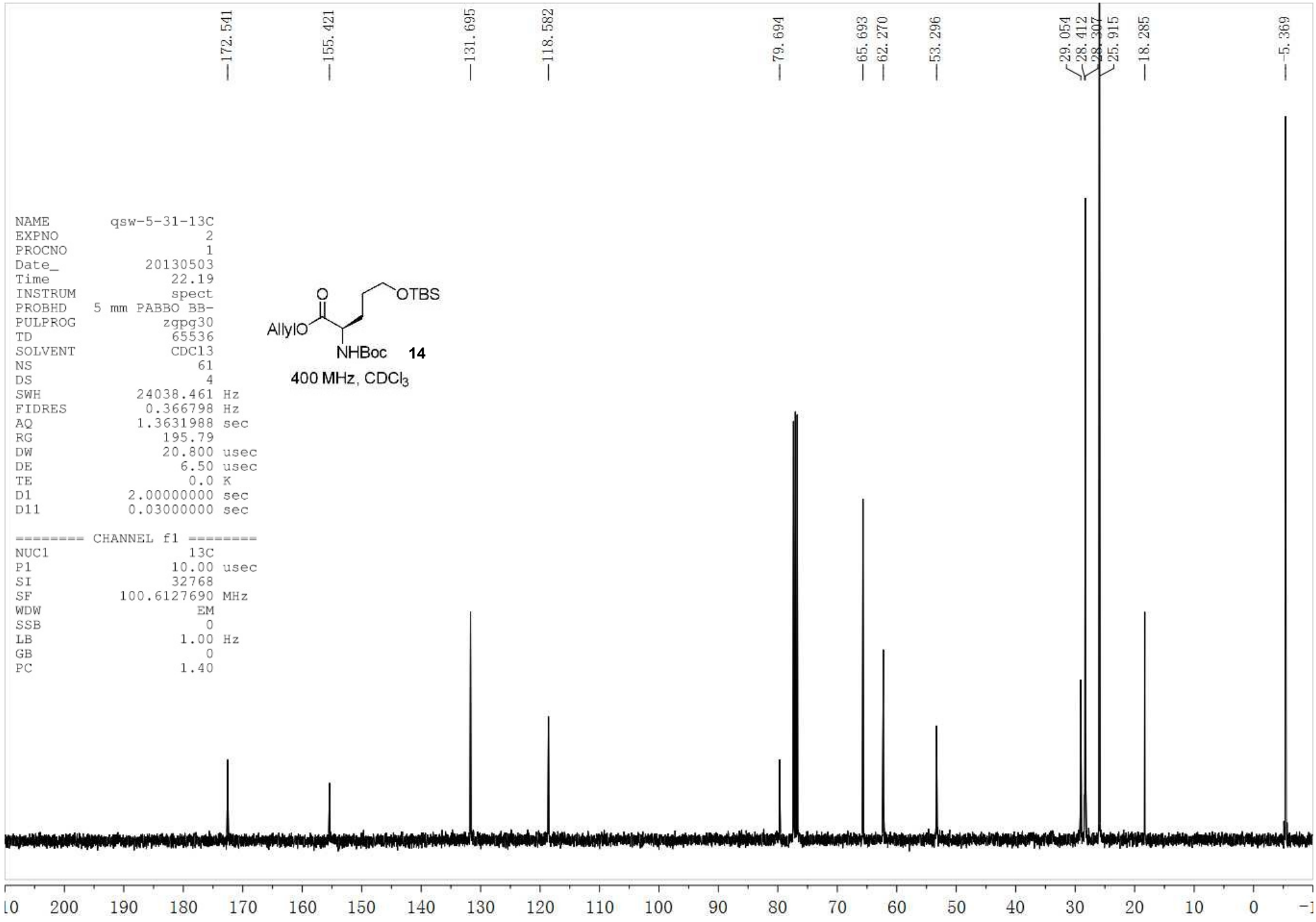

5.930
5.920
5.919
5.909
5.898
5.886
5.884
5.875
5.864
5.863
5.852
5.296
5.295
5.294
5.222
4.898
4.891
4.611
4.600
3.686
3.674
3.663
3.650
2.276
2.264
2.260
2.247
2.233
2.230
2.222
2.218
2.204
1.984
1.970
1.966
1.953
1.948
1.938
1.924
1.907
1.667
1.664
1.651
1.640
1.633
1.629
1.625
1.620
1.614
1.607
1.489
NAME qsw-5-54
EXPNO 1
PROCNO 1
Date_ 20130521
Time 6.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 301.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OH
AllylO
N(Boc)
13
500MHz, CDCl3
1.00
1.11
1.03
1.03
2.03
2.06
1.01
1.06
3.18
18.39
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0

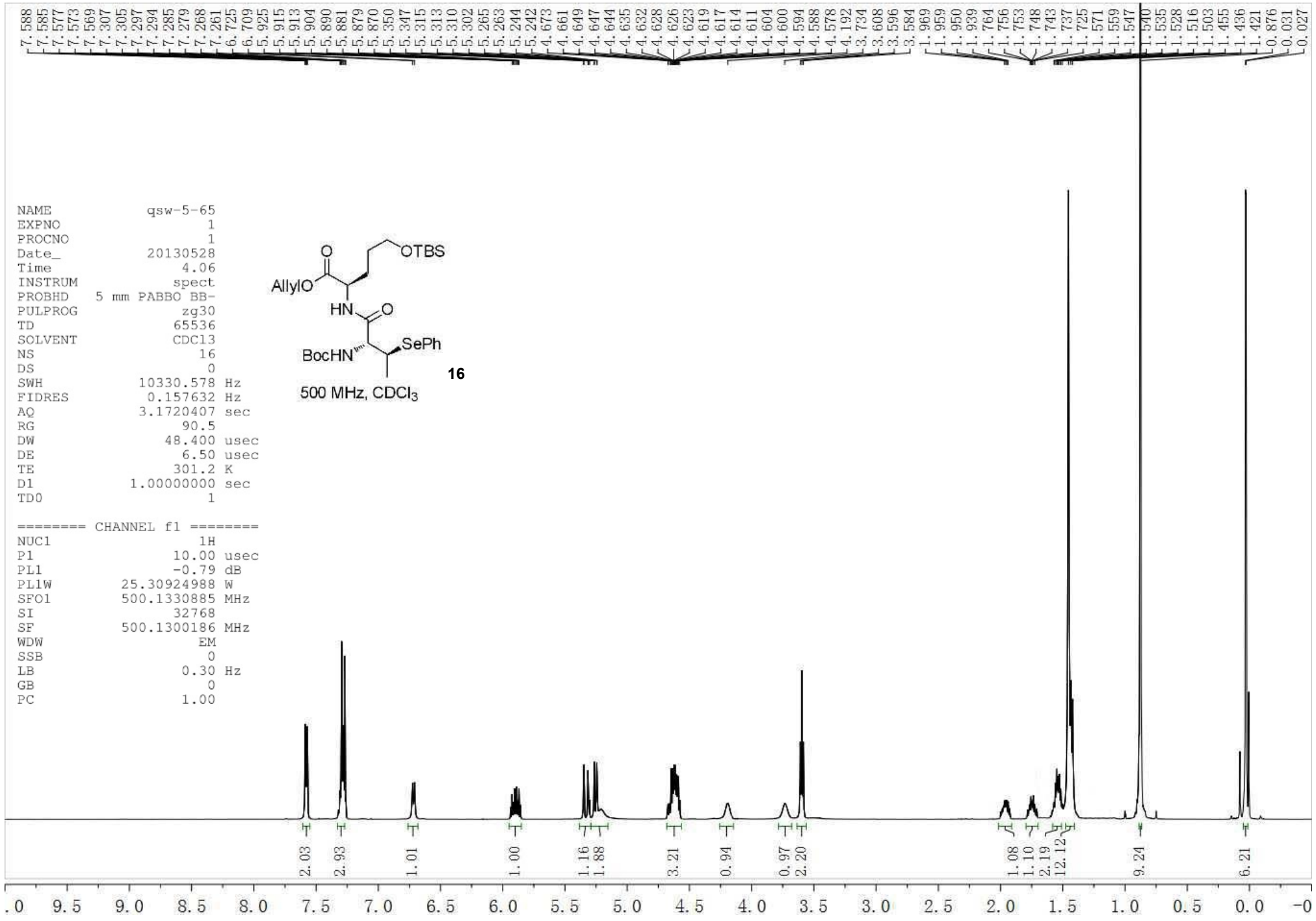

170.574
152.282
131.875
118.073
83.174
65.717
62.322
58.024
29.441
28.011
26.263
NAME qsw-5-54-13C
EXPNO 2
PROCNO 1
Date_ 20130521
Time 6.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT CDCl3
NS 74
DS 0
SWH 32679.738 Hz
FIDRES 0.997306 Hz
AQ 0.5014157 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 301.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 30.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7714350 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.79 dB
PL12 16.68 dB
PL2W 25.30924988 W
PL12W 0.45318890 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577851 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OH
AllylO
N(Boc)
13
500MHz, CDCl3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

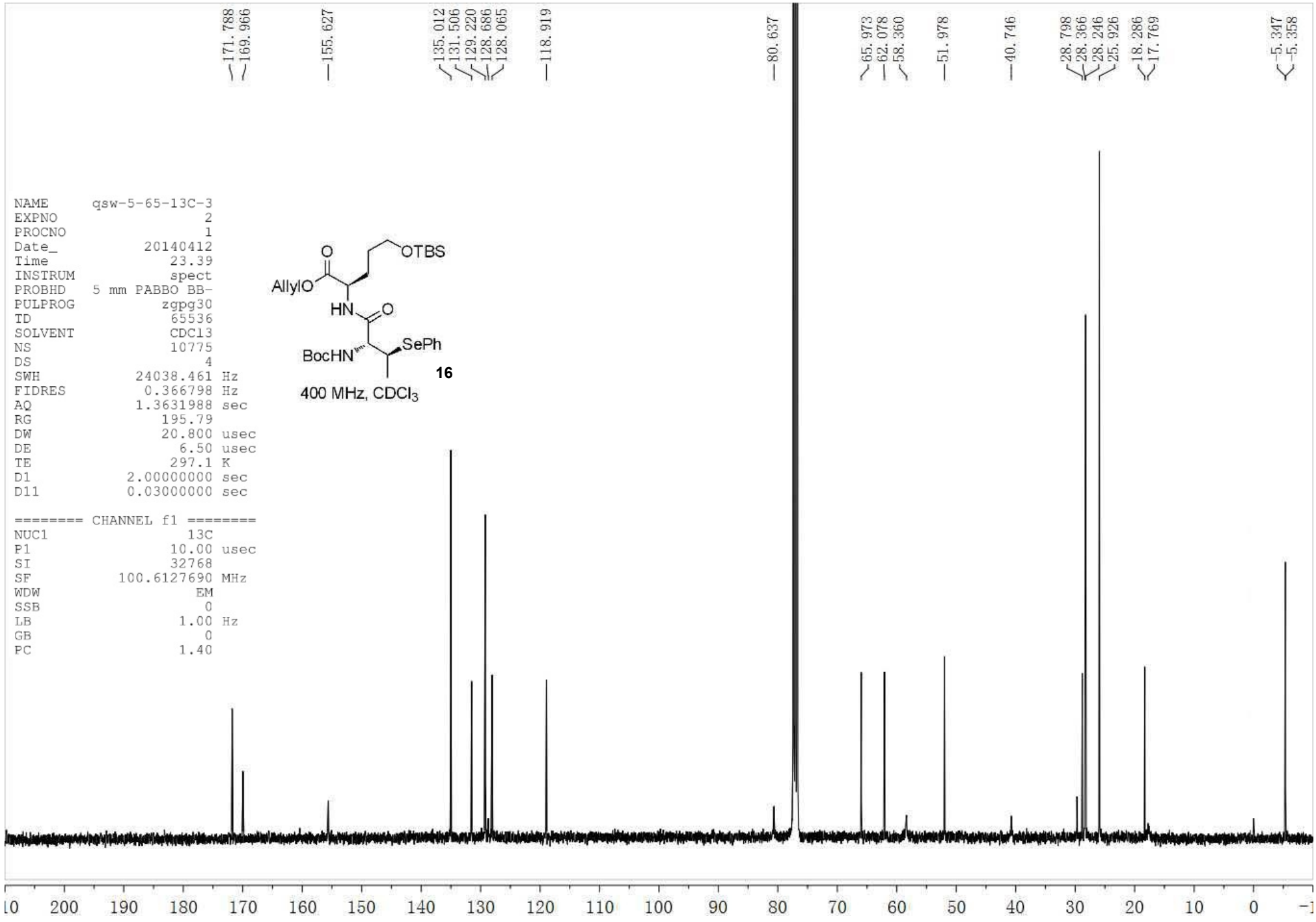

5.937
5.925
5.915
5.904
5.891
5.881
5.870
5.859
5.343
5.255
5.254
5.234
5.233
4.660
4.649
4.634
4.625
4.613
4.599
4.346
3.668
3.655
3.643
2.073
1.934
1.924
1.911
1.895
1.775
1.758
1.745
1.731
1.717
1.702
1.655
1.650
1.642
1.638
1.629
1.626
1.619
1.613
1.607
1.600
1.594
1.429
NAME qsw-5-47
EXPNO 1
PROCNO 1
Date_ 20130516
Time 5.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 45.3
DW 48.400 usec
DE 6.50 usec
TE 301.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OH
AllylO
NHBoc
13-1
500MHz, CDCl3
1.00
1.06
1.96
2.15
0.92
2.17
0.99
1.44
1.12
2.14
9.38
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0

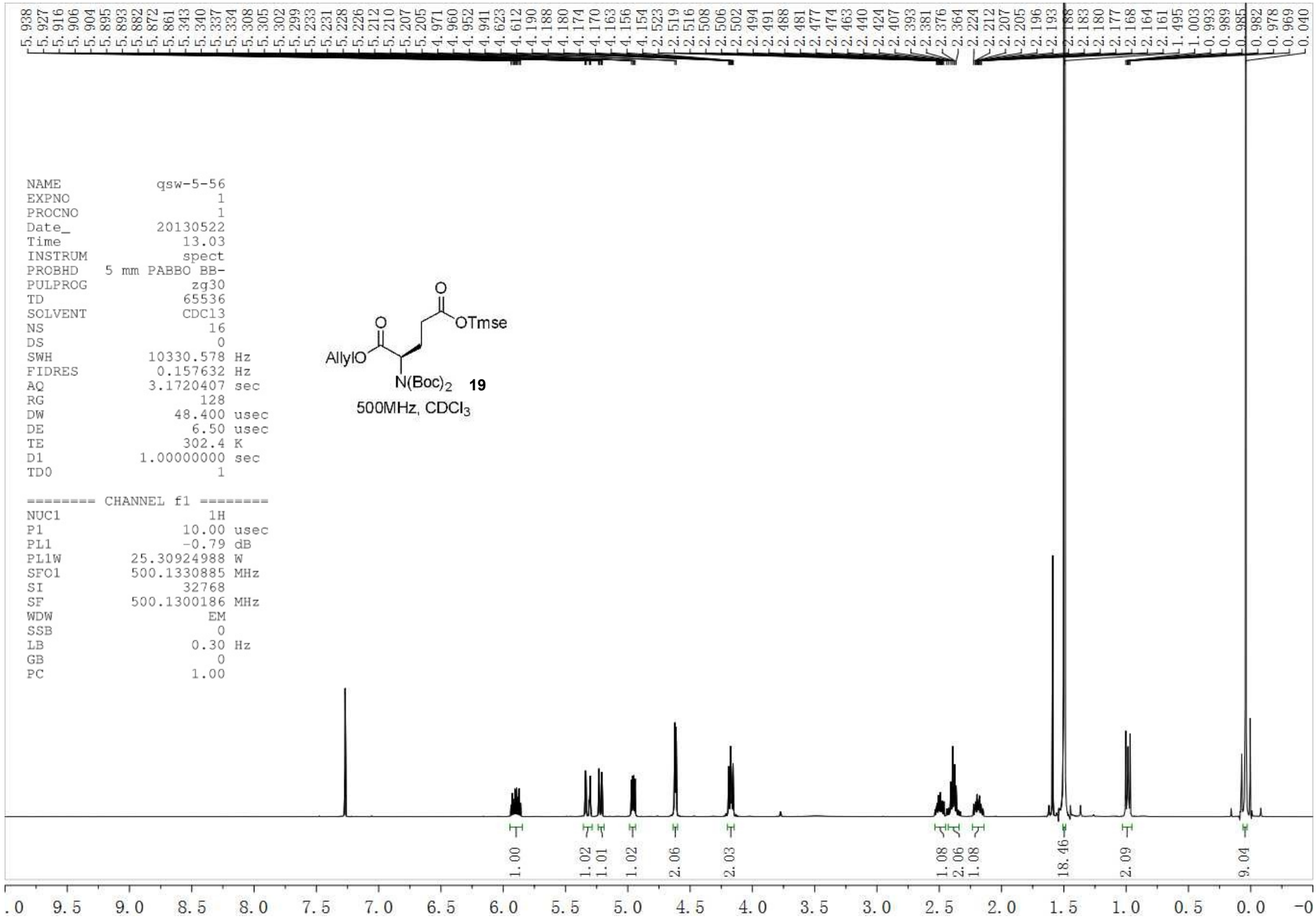

172.478
155.561
131.663
118.807
79.976
65.855
62.039
53.253
29.463
28.330
NAME qsw-5-47-13C
EXPNO 2
PROCNO 1
Date_ 20130516
Time 5.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT CDC13
NS 108
DS 0
SWH 32679.738 Hz
FIDRES 0.997306 Hz
AQ 0.5014157 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 301.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 30.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7714350 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.79 dB
PL12 16.68 dB
PL2W 25.30924988 W
PL12W 0.45318890 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577851 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OH
AllylO
NHBoc
13-1
500MHz, CDCl3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

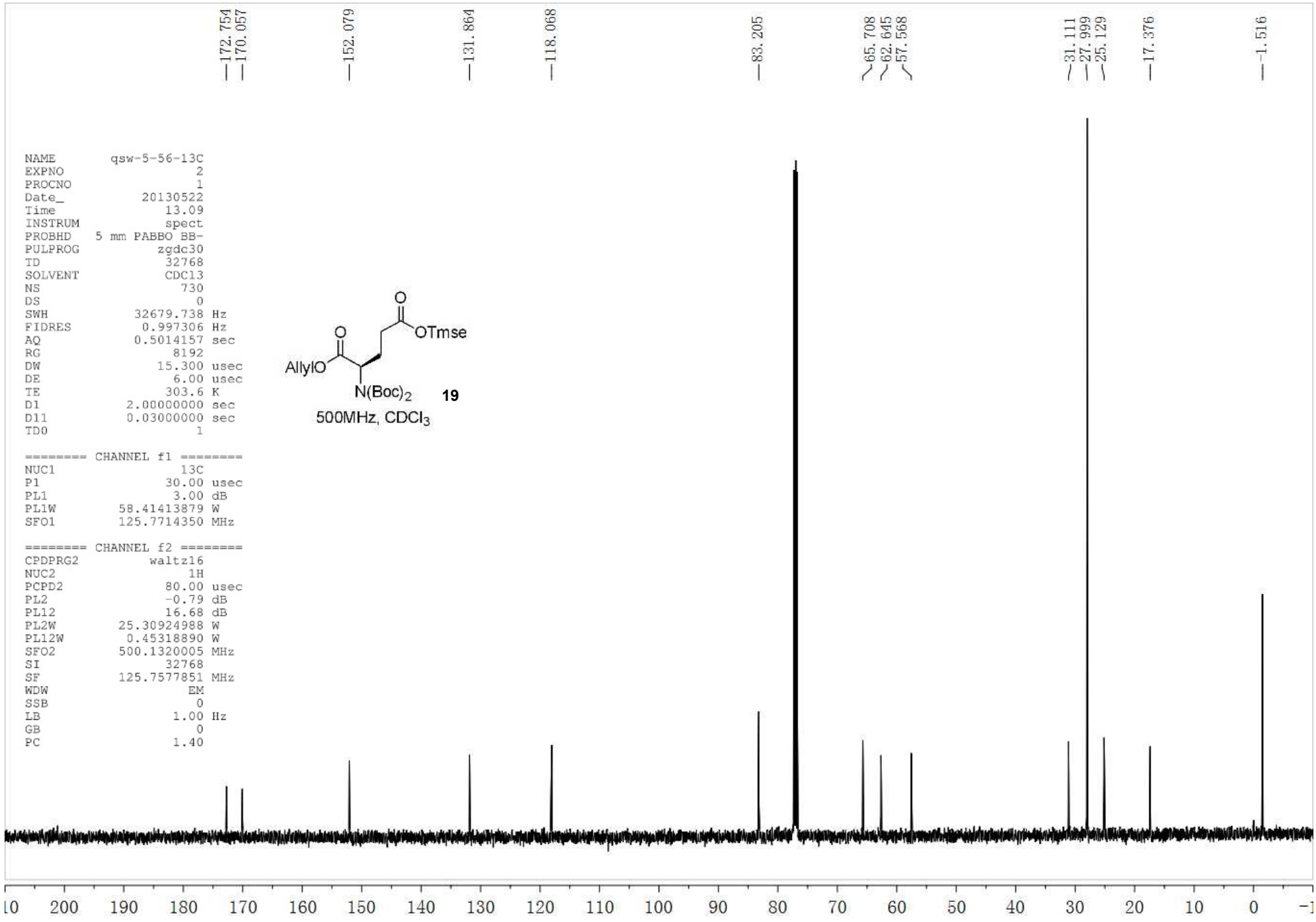

5.932
5.917
5.905
5.891
5.875
5.862
5.848
5.834
5.329
5.234
5.232
5.208
5.206
4.628
4.614
4.603
4.589
4.570
4.555
4.296
4.283
3.604
3.589
1.891
1.880
1.855
1.741
1.724
1.706
1.689
1.587
1.583
1.572
1.567
1.556
1.552
1.544
1.535
1.528
1.520
1.513
0.867
0.025
NAME qsw-5-31
EXPNO 1
PROCNO 1
Date_ 20130503
Time 22.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 28.46
DW 60.800 usec
DE 6.50 usec
TE 0.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OTBS
AllylO
NHBoc
14
400 MHz, CDCl3
1.00
1.20
1.98
2.21
0.92
2.21
1.34
1.11
2.22
9.28
9.22
6.21
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0

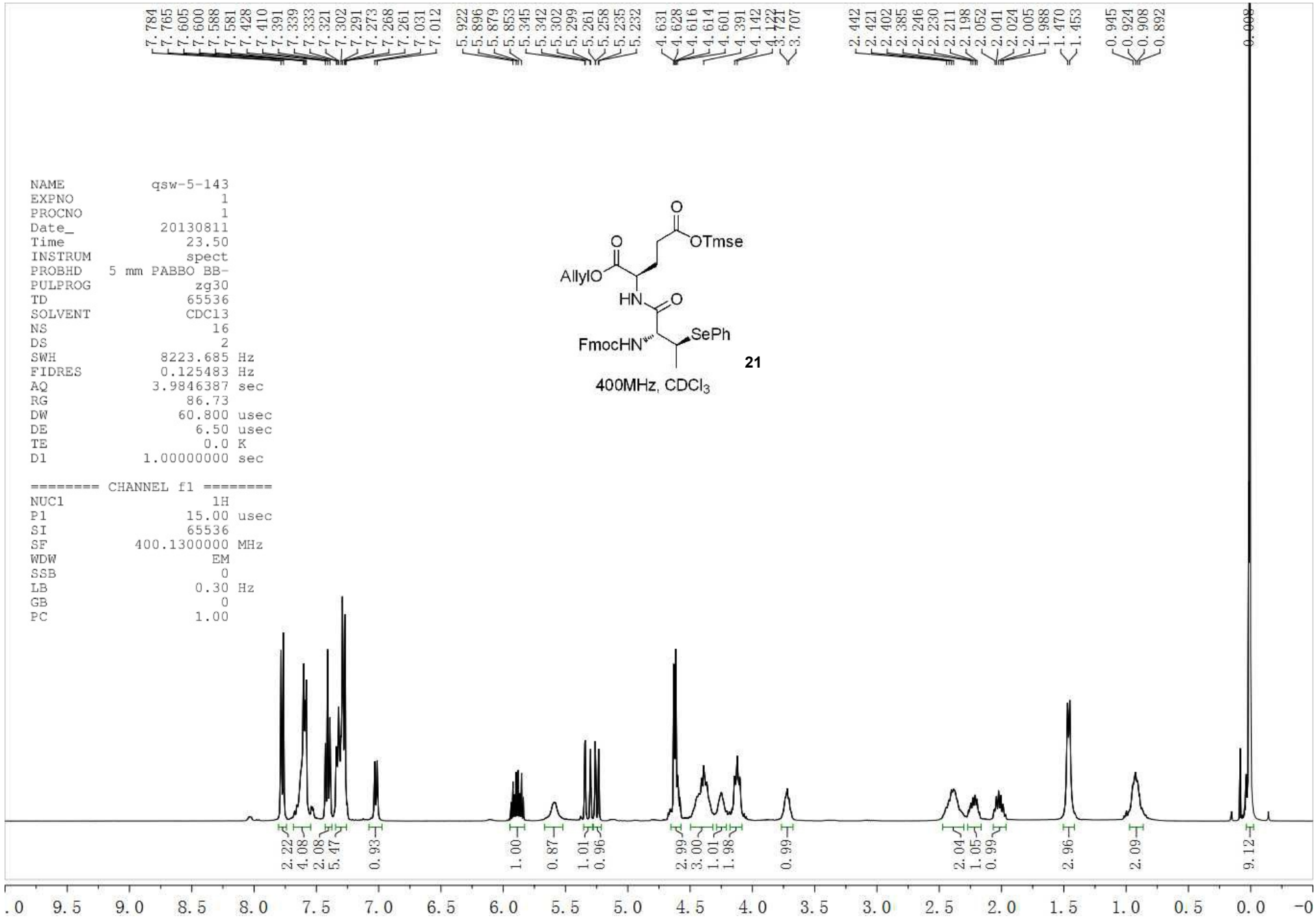

172.541
155.421
131.695
118.582
79.694
65.693
62.270
53.296
29.054
28.412
28.307
25.915
18.285
-5.369
NAME qsw-5-31-13C
EXPNO 2
PROCNO 1
Date_ 20130503
Time 22.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 61
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 0.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
OTBS
AllylO
NHBoc
14
400 MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

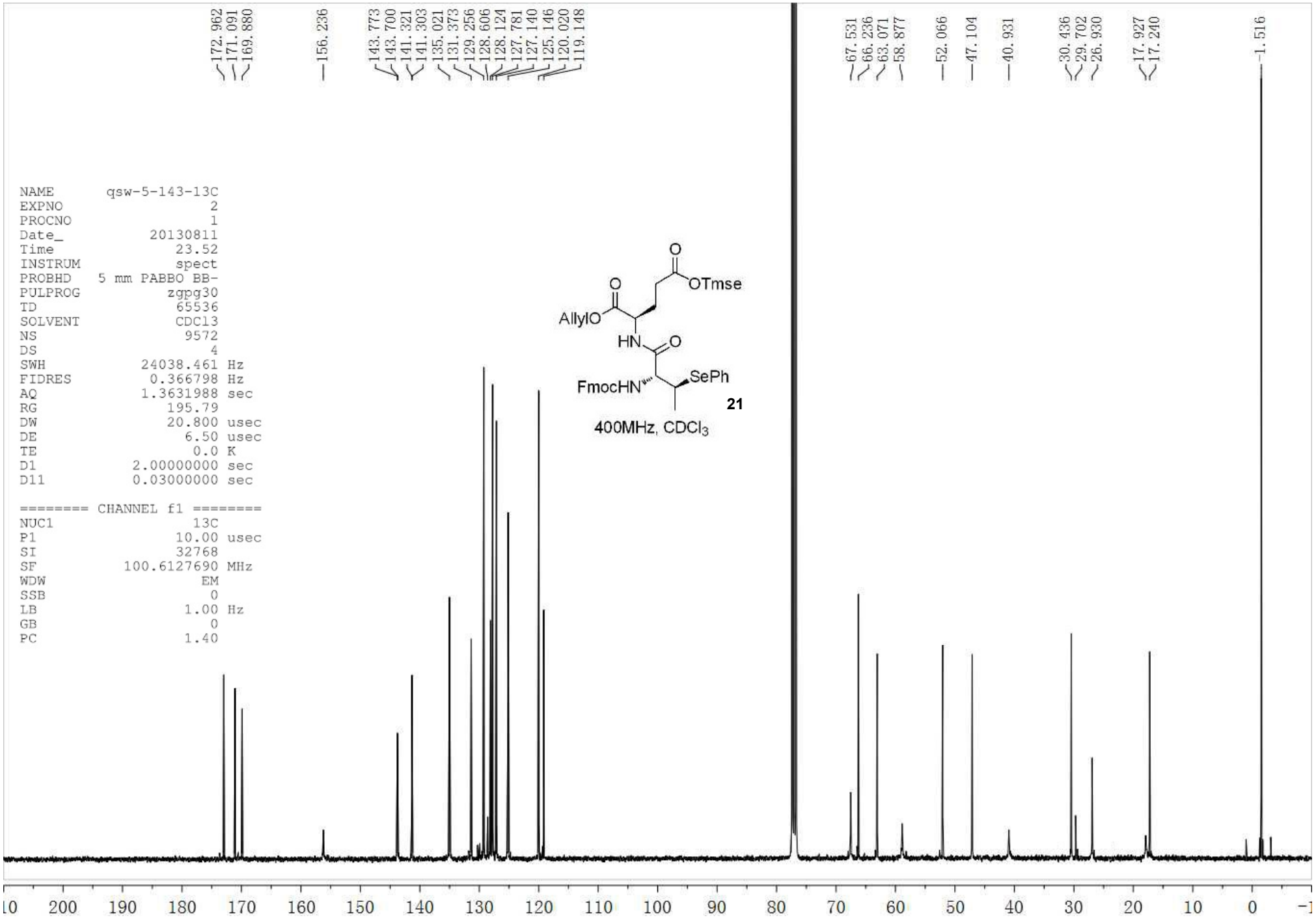

7.588 7.585 7.577 7.573 7.569 7.307 7.305 7.297 7.294 7.285 7.279 7.268 7.261 6.725 6.709 5.925 5.915 5.913 5.904 5.890 5.881 5.879 5.870 5.350 5.347 5.315 5.313 5.310 5.302 5.265 5.263 5.244 5.242 4.673 4.661 4.649 4.647 4.644 4.635 4.632 4.628 4.626 4.623 4.619 4.617 4.614 4.611 4.604 4.600 4.594 4.588 4.578 4.192 3.734 3.608 3.596 3.584 1.969 1.959 1.950 1.939 1.764 1.756 1.753 1.748 1.743 1.737 1.725 1.571 1.559 1.547 1.540 1.535 1.528 1.516 1.503 1.455 1.436 1.421 0.876 0.031 0.027
NAME qsw-5-65
EXPNO 1
PROCNO 1
Date_ 20130528
Time 4.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.50 usec
TE 301.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AllylO
O
OTBS
HN
O
BocHN
SePh
16
500 MHz, CDCl3
2.03 2.93 1.01 1.00 1.16 1.88 3.21 0.94 0.97 2.20 1.08 1.10 2.19 12.12 9.24 6.21
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

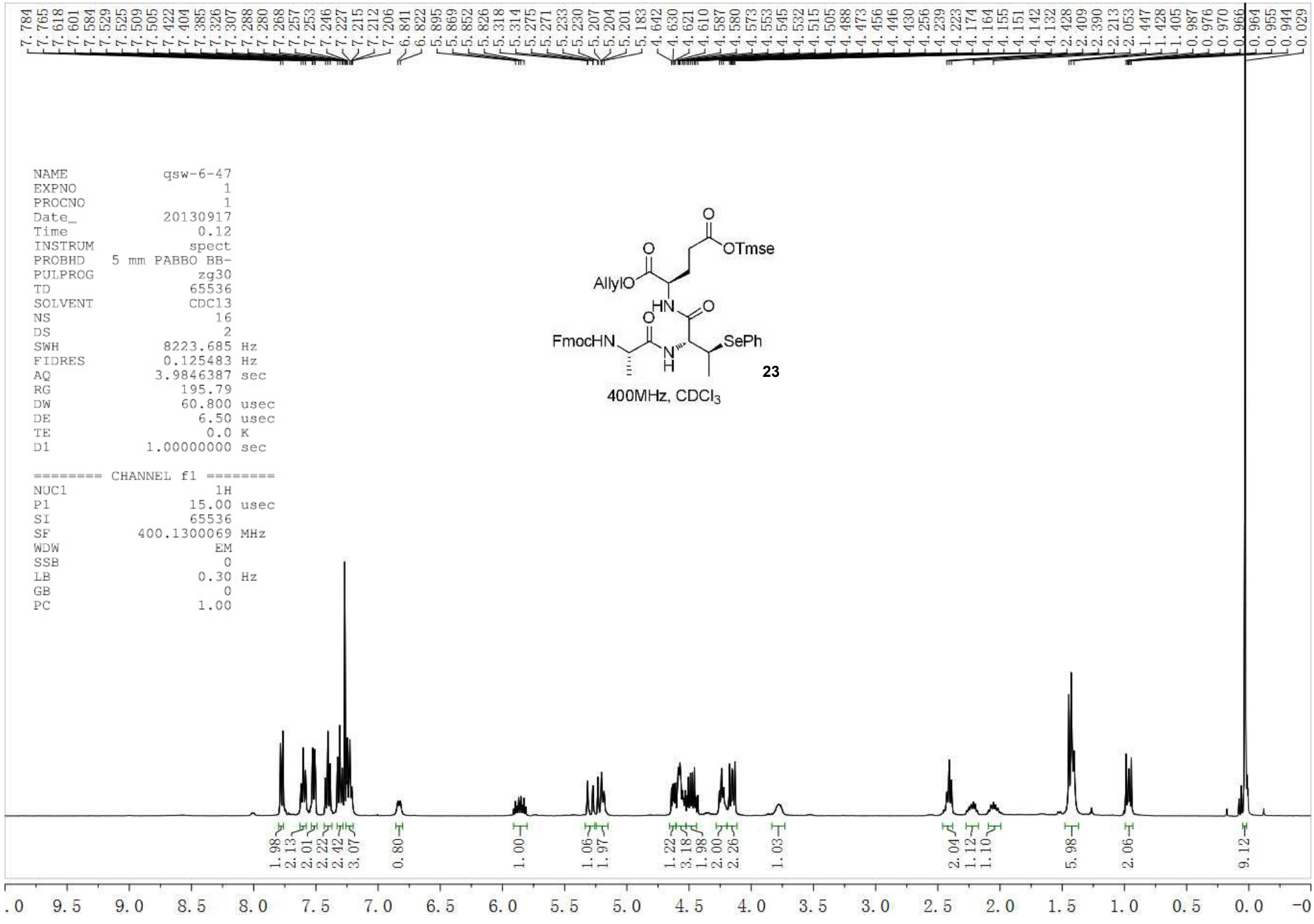

171.788
169.966
155.627
135.012
131.506
129.220
128.686
128.065
118.919
80.637
65.973
62.078
58.360
51.978
40.746
28.798
28.366
28.246
25.926
18.286
17.769
-5.347
-5.358
NAME qsw-5-65-13C-3
EXPNO 2
PROCNO 1
Date_ 20140412
Time 23.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10775
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AllylO
O
OTBS
HN
O
BocHN
SePh
16
400 MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1

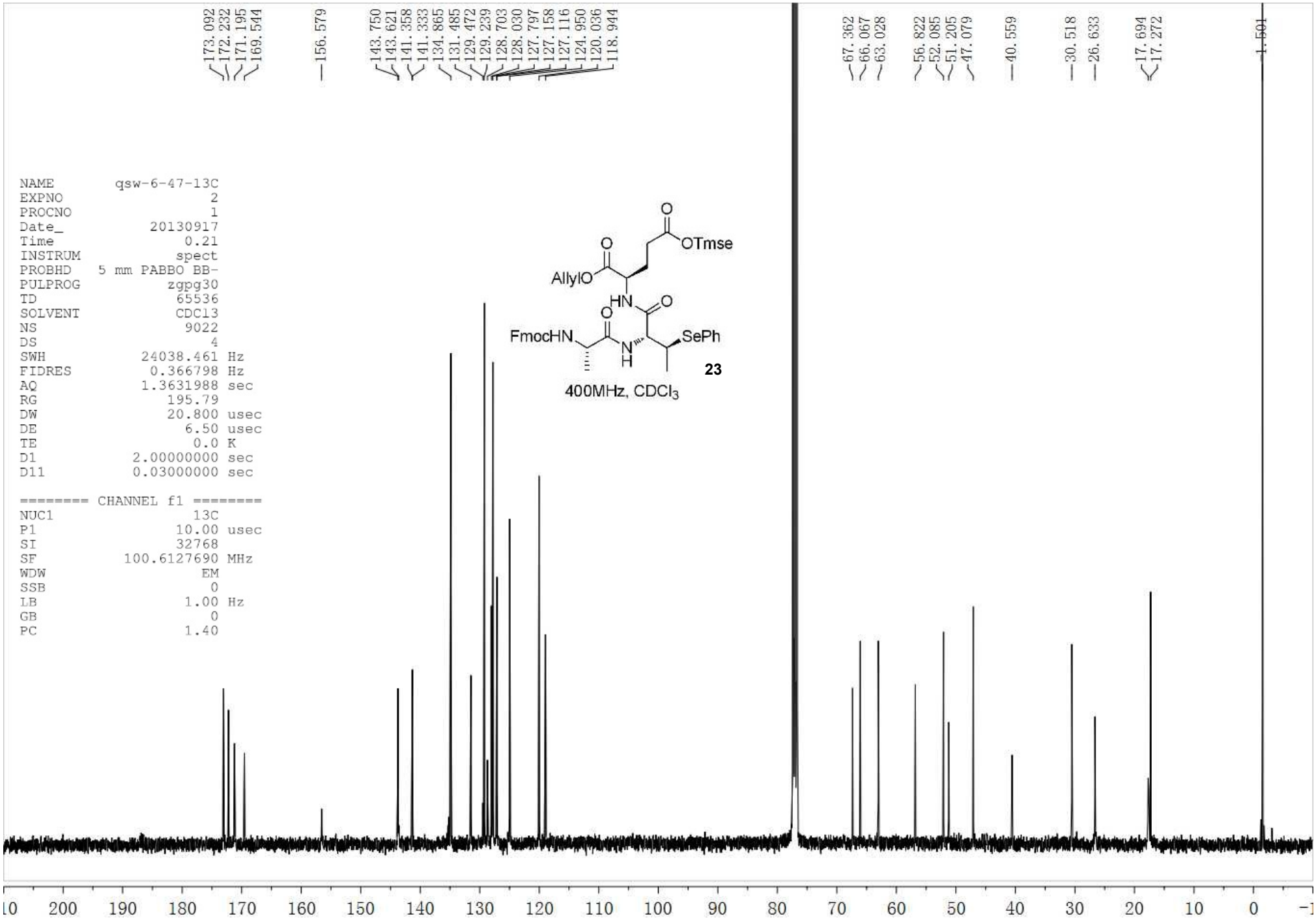

5.938 5.927 5.916 5.906 5.904 5.895 5.893 5.882 5.872 5.861 5.343 5.340 5.337 5.334 5.308 5.305 5.302 5.299 5.233 5.231 5.228 5.226 5.212 5.210 5.207 5.205 4.971 4.960 4.952 4.941 4.623 4.612 4.190 4.188 4.180 4.174 4.170 4.163 4.156 4.154 2.523 2.519 2.516 2.508 2.506 2.502 2.494 2.491 2.488 2.481 2.477 2.474 2.463 2.440 2.424 2.407 2.393 2.381 2.376 2.364 2.224 2.212 2.207 2.205 2.196 2.193 2.188 2.183 2.180 2.177 2.168 2.164 2.161 1.495 1.003 0.993 0.989 0.985 0.982 0.978 0.969 0.040
NAME qsw-5-56
EXPNO 1
PROCNO 1
Date_ 20130522
Time 13.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 302.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
OTmse
AllylO
N(Boc)2
19
500MHz, CDCl3
1.00 1.02 1.01 1.02 2.06 2.03 1.08 2.06 1.08 18.46 2.09 9.04
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

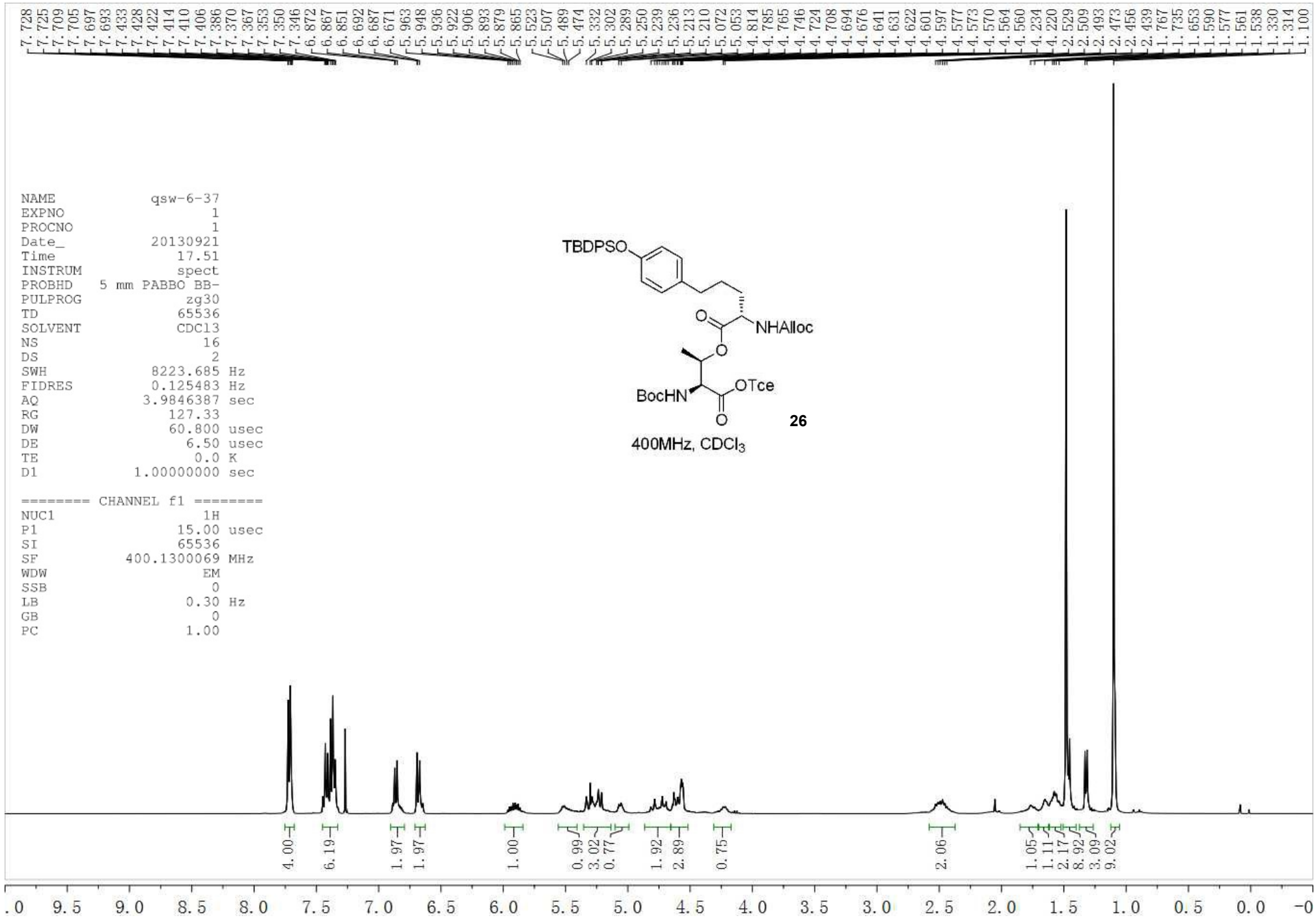

172.754
170.057
152.079
131.864
118.068
83.205
65.708
62.645
57.568
31.111
27.999
25.129
17.376
-1.516
NAME qsw-5-56-13C
EXPNO 2
PROCNO 1
Date_ 20130522
Time 13.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT CDCl3
NS 730
DS 0
SWH 32679.738 Hz
FIDRES 0.997306 Hz
AQ 0.5014157 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 303.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 30.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7714350 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.79 dB
PL12 16.68 dB
PL2W 25.30924988 W
PL12W 0.45318890 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577851 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AllylO
OTmse
N(Boc)2
19
500MHz, CDCl3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

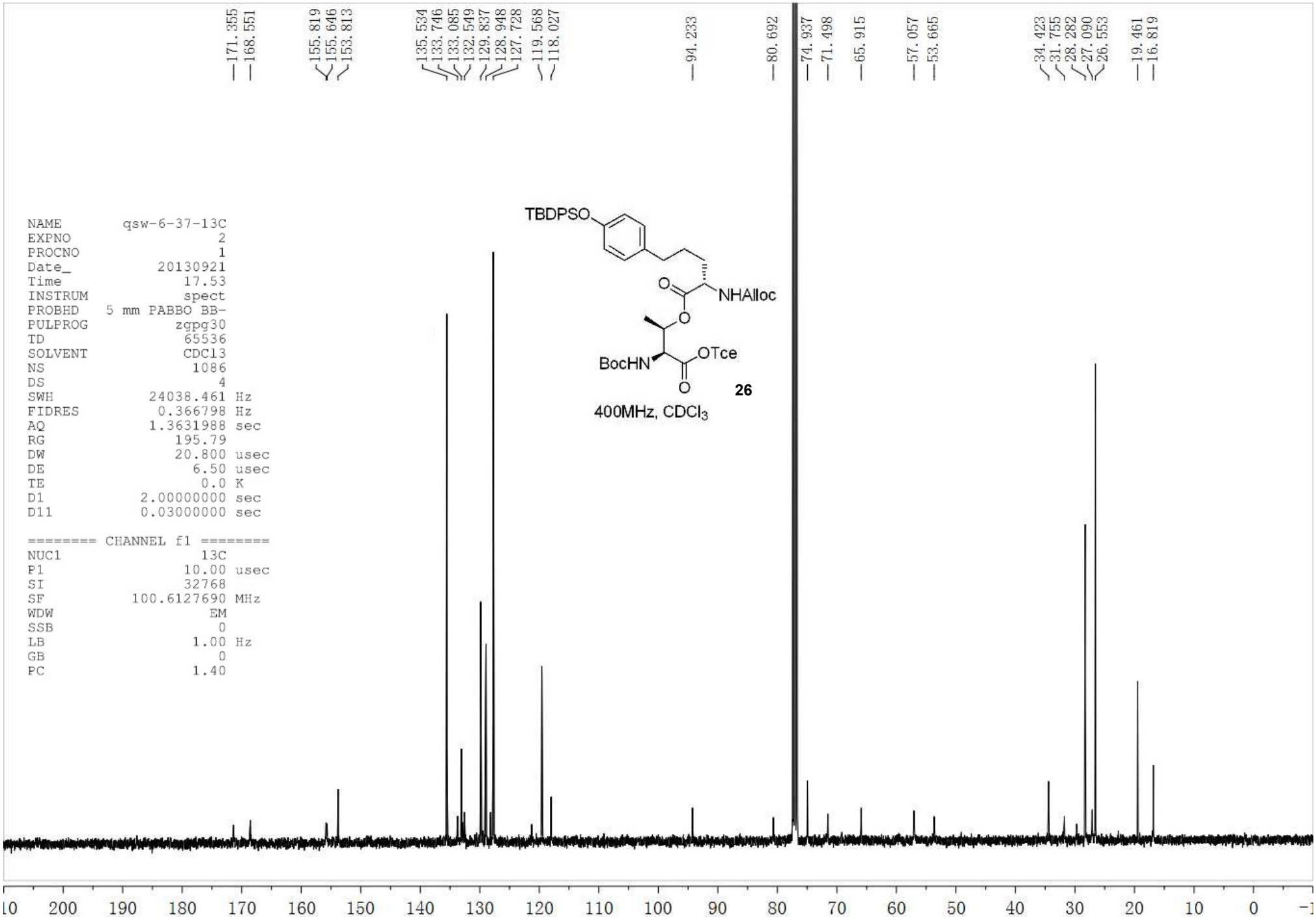

7.784 7.765 7.605 7.600 7.588 7.581 7.428 7.410 7.391 7.339 7.333 7.321 7.302 7.291 7.273 7.268 7.261 7.031 7.012
5.922 5.896 5.879 5.853 5.345 5.342 5.302 5.299 5.261 5.258 5.235 5.232
4.631 4.628 4.616 4.614 4.601 4.391 4.142 3.707
2.442 2.421 2.402 2.385 2.246 2.230 2.211 2.198 2.052 2.041 2.024 2.005 1.988 1.470 1.453
0.945 0.924 0.908 0.892
0.000
NAME qsw-5-143
EXPNO 1
PROCNO 1
Date_ 20130811
Time 23.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 86.73
DW 60.800 usec
DE 6.50 usec
TE 0.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OTmse
AllylO
HN
O
FmocHN
SePh
21
400MHz, CDCl3
2.22 4.08 2.08 5.47 0.93 1.00 0.87 1.01 0.96 2.99 3.00 1.01 1.98 0.99 2.04 1.05 0.99 2.96 2.09 9.12
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

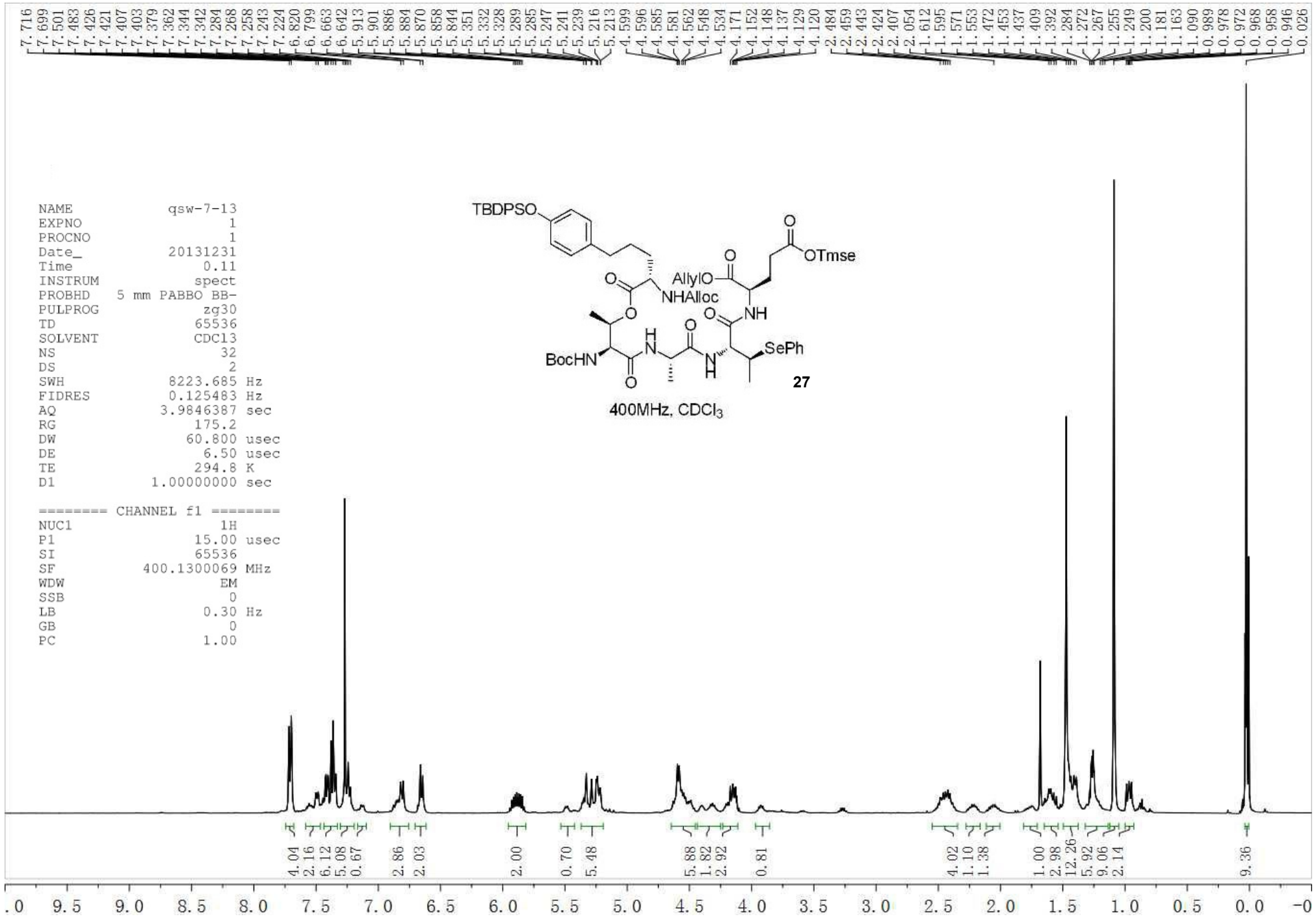

172.962
171.091
169.880
156.236
143.773
143.700
141.321
141.303
135.021
131.373
129.256
128.606
128.124
127.781
127.140
125.146
120.020
119.148
67.531
66.236
63.071
58.877
52.066
47.104
40.931
30.436
29.702
26.930
17.927
17.240
1.516
NAME qsw-5-143-13C
EXPNO 2
PROCNO 1
Date_ 20130811
Time 23.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9572
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 0.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AllylO
OTmse
HN
FmocHN
SePh
21
400MHz, CDCl3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

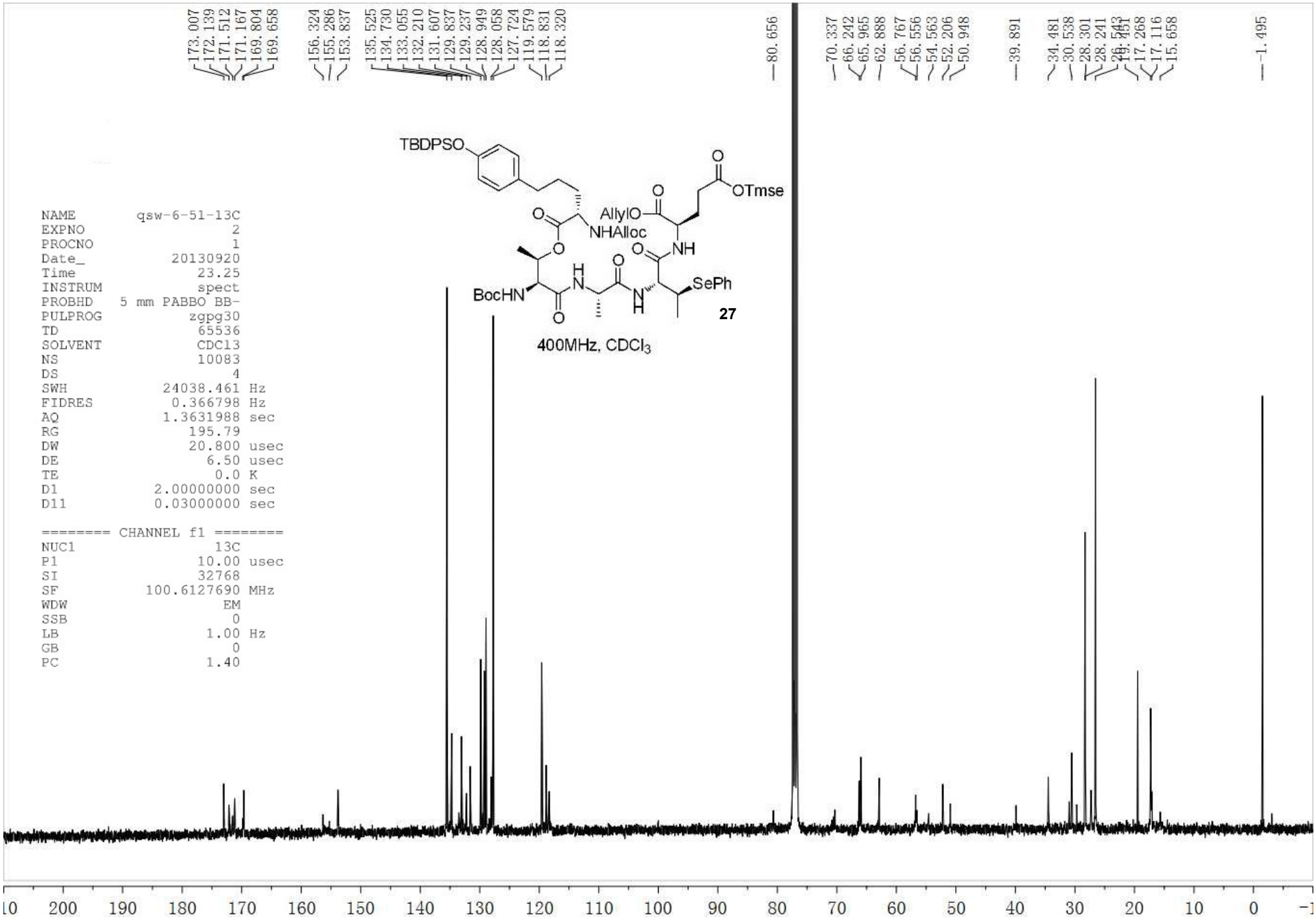

7.784 7.765 7.618 7.601 7.584 7.529 7.525 7.509 7.505 7.422 7.404 7.385 7.326 7.307 7.288 7.280 7.268 7.257 7.253 7.246 7.227 7.215 7.212 7.206 6.841 6.822 5.895 5.869 5.852 5.826 5.318 5.314 5.275 5.271 5.233 5.230 5.207 5.204 5.201 5.183 4.642 4.630 4.621 4.610 4.587 4.580 4.573 4.553 4.545 4.532 4.515 4.505 4.488 4.473 4.456 4.446 4.430 4.256 4.239 4.223 4.174 4.164 4.155 4.151 4.142 4.132 2.428 2.409 2.390 2.213 2.053 1.447 1.428 1.405 0.987 0.976 0.970 0.966 0.964 0.955 0.944 0.029
NAME qsw-6-47
EXPNO 1
PROCNO 1
Date_ 20130917
Time 0.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 195.79
DW 60.800 usec
DE 6.50 usec
TE 0.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
OTmse
AllylO
HN
O
O
FmocHN
N
H
SePh
23
400MHz, CDCl3
1.98 2.13 2.01 2.22 2.42 3.07 0.80 1.00 1.06 1.97 1.22 3.18 1.98 2.00 2.26 1.03 2.04 1.12 1.10 5.98 2.06 9.12
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

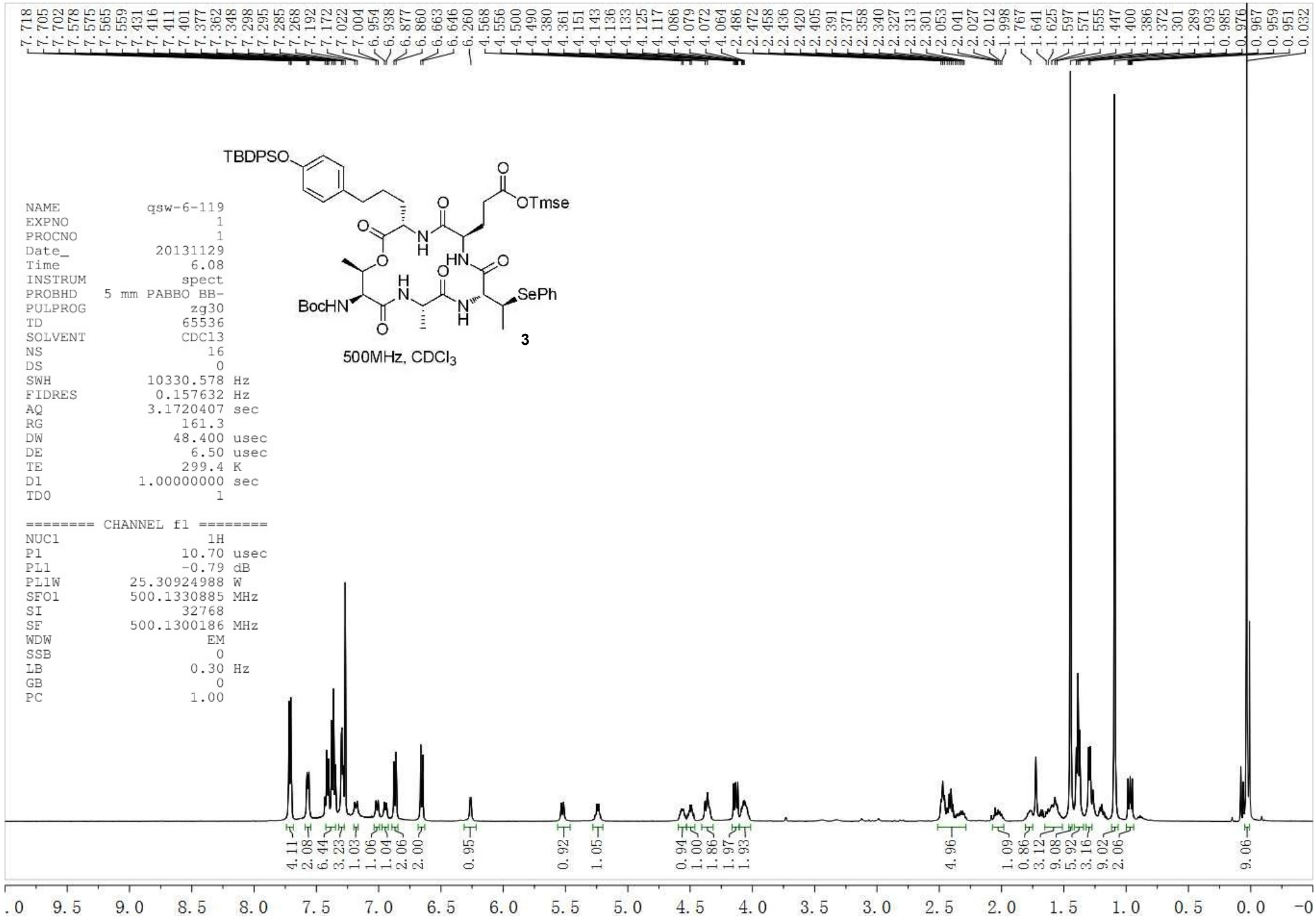

173.092
172.232
171.195
169.544
156.579
143.750
143.621
141.358
141.333
134.865
131.485
129.472
129.239
128.703
128.030
127.797
127.158
127.116
124.950
120.036
118.944
67.362
66.067
63.028
56.822
52.085
51.205
47.079
40.559
30.518
26.633
17.694
17.272
1.501
NAME qsw-6-47-13C
EXPNO 2
PROCNO 1
Date_ 20130917
Time 0.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9022
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 0.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AllylO
OTmse
FmocHN
SePh
23
400MHz, CDCl3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

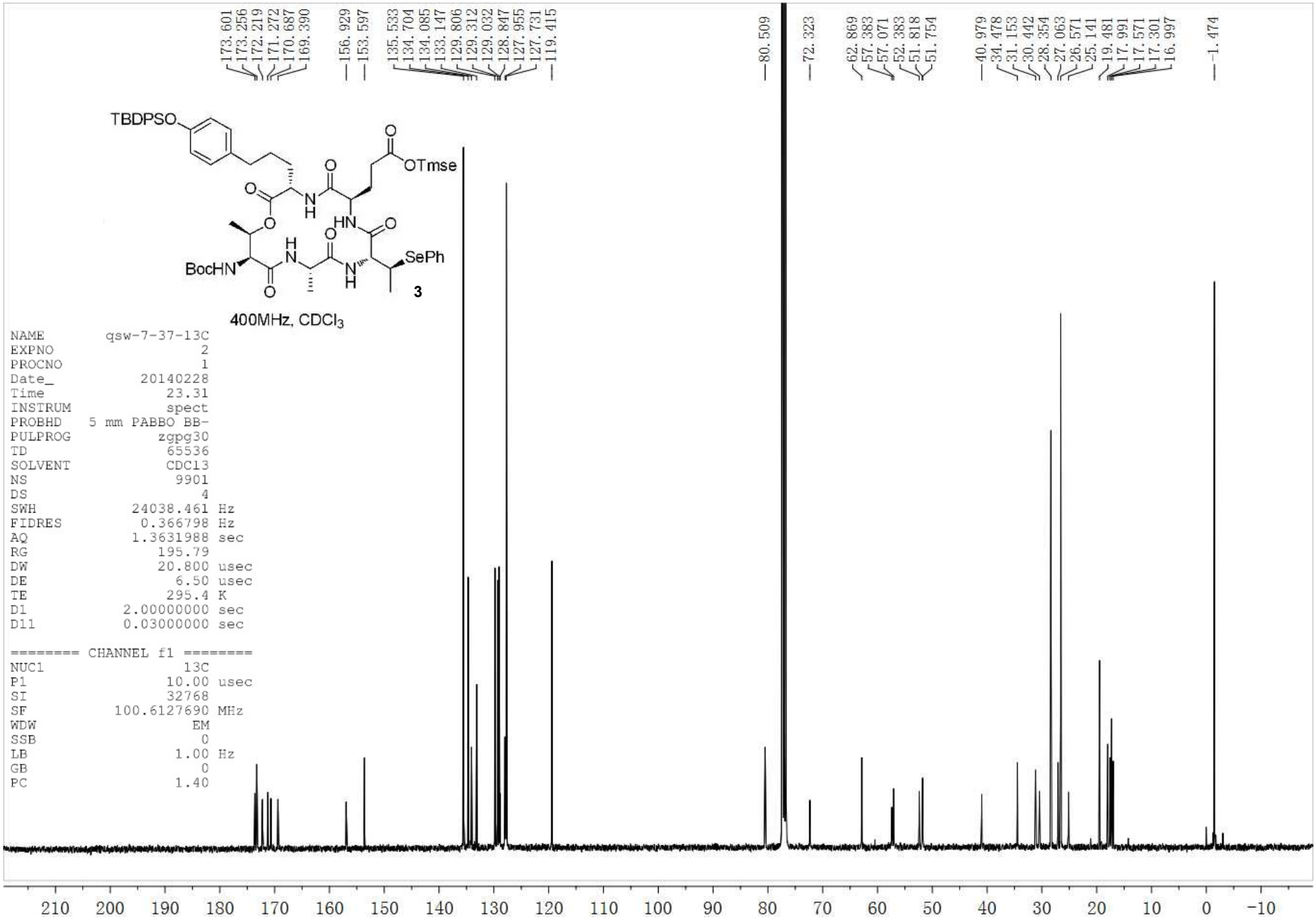

7.728 7.725 7.709 7.705 7.697 7.693 7.433 7.428 7.422 7.414 7.410 7.406 7.386 7.370 7.367 7.353 7.350 7.346 6.872 6.867 6.851 6.692 6.687 6.671 5.963 5.948 5.936 5.922 5.906 5.893 5.879 5.865 5.523 5.507 5.489 5.474 5.332 5.302 5.289 5.250 5.239 5.236 5.213 5.210 5.072 5.053 4.814 4.785 4.765 4.746 4.724 4.708 4.694 4.676 4.641 4.631 4.622 4.601 4.597 4.577 4.573 4.570 4.564 4.560 4.234 4.220 2.529 2.509 2.493 2.473 2.456 2.439 1.767 1.735 1.653 1.590 1.577 1.561 1.538 1.330 1.314 1.100
NAME qsw-6-37
EXPNO 1
PROCNO 1
Date_ 20130921
Time 17.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 127.33
DW 60.800 usec
DE 6.50 usec
TE 0.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBDPSO
NHAlloc
BocHN
OTce
26
400MHz, CDCl3
4.00 6.19 1.97 1.97 1.00 0.99 3.02 0.77 1.92 2.89 0.75 2.06 1.05 1.11 2.17 8.92 3.09 9.02
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

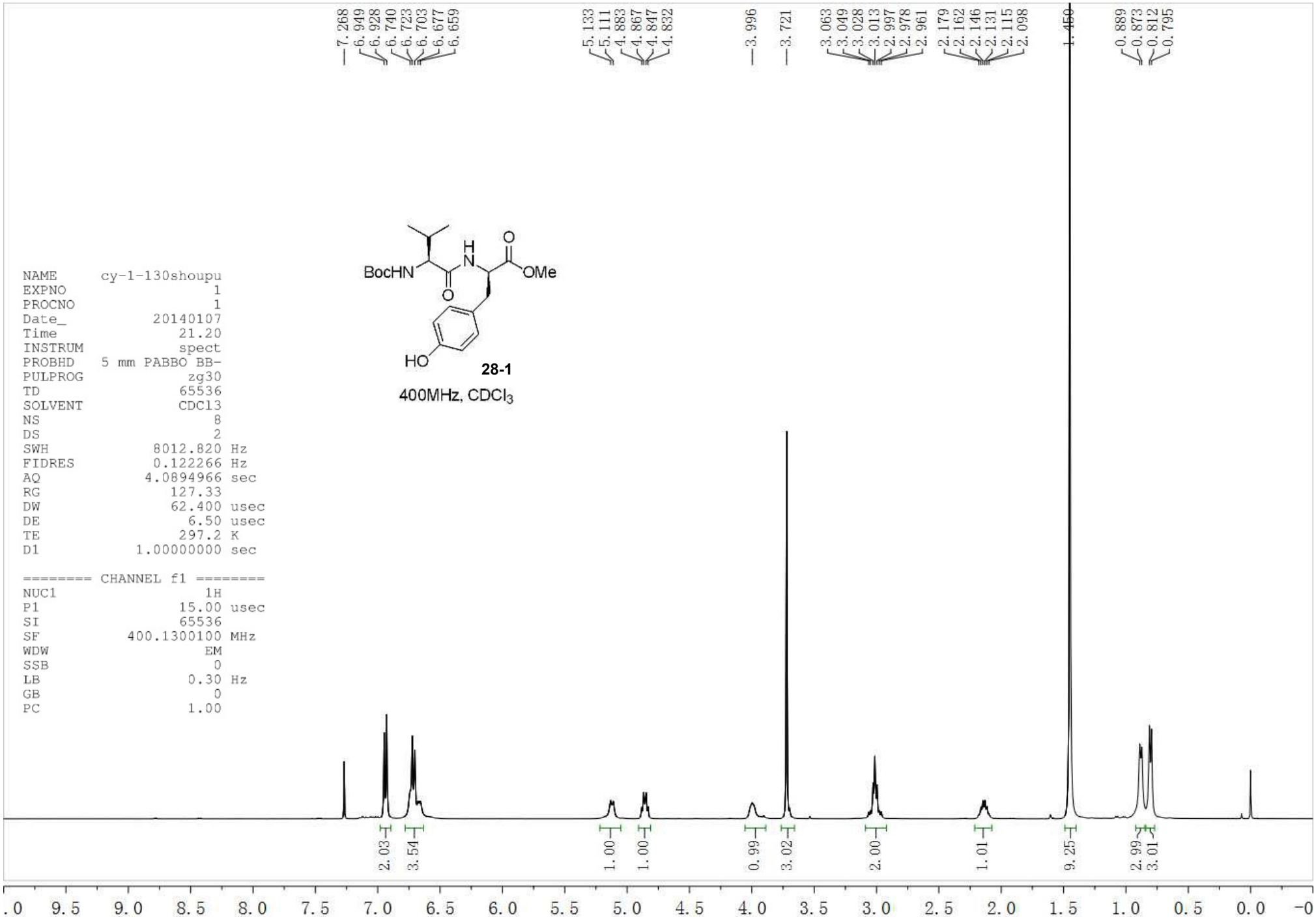

171.355
168.551
155.819
155.646
153.813
135.534
133.746
133.085
132.549
129.837
128.948
127.728
119.568
118.027
94.233
80.692
74.937
71.498
65.915
57.057
53.665
34.423
31.755
28.282
27.090
26.553
19.461
16.819
NAME qsw-6-37-13C
EXPNO 2
PROCNO 1
Date_ 20130921
Time 17.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1086
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 0.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
TBDPSO
NHAlloc
BocHN
OTce
26
400MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

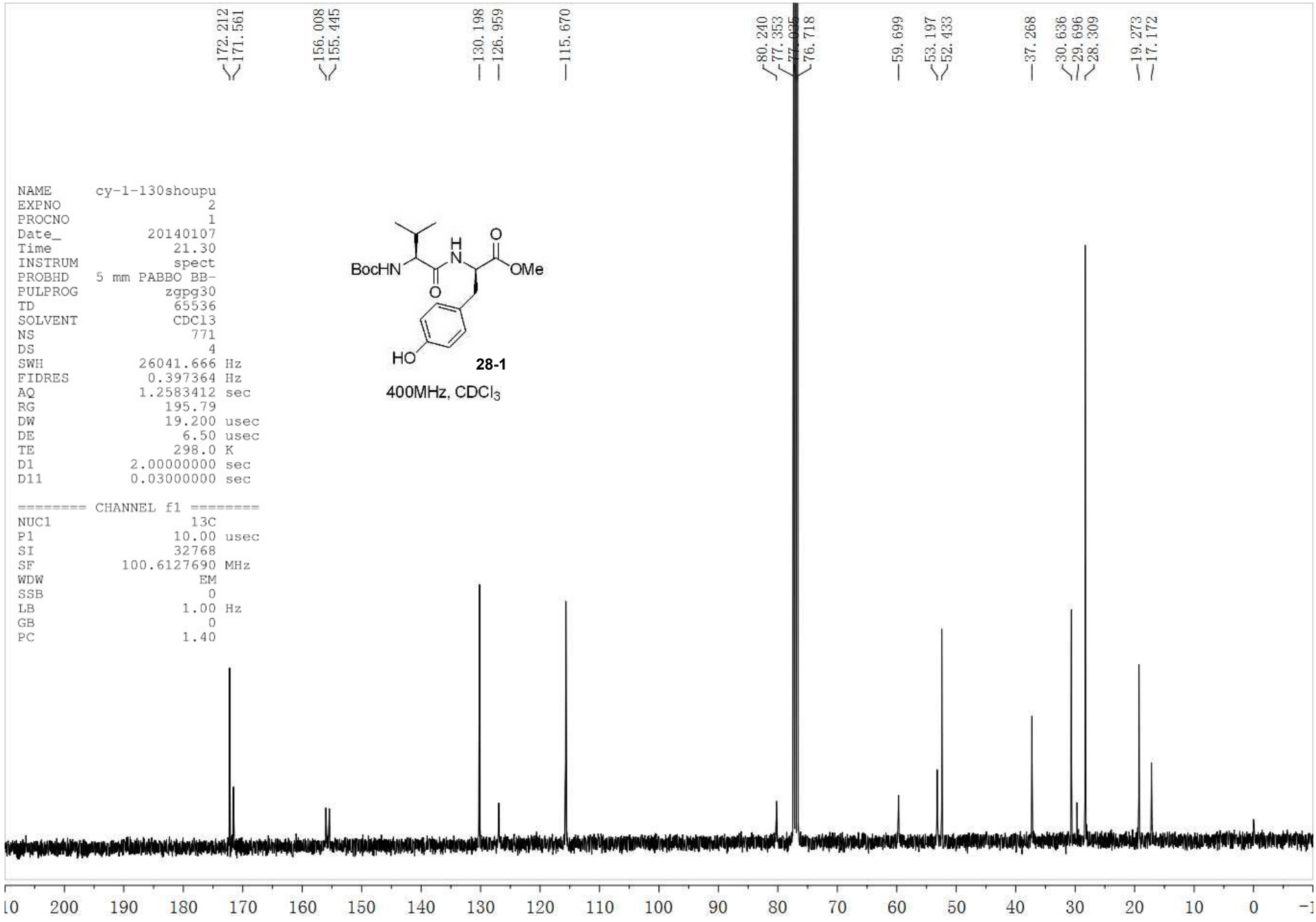

7.716 7.699 7.501 7.483 7.426 7.421 7.407 7.403 7.379 7.362 7.344 7.342 7.284 7.268 7.258 7.243 7.224 6.820 6.799 6.663 6.642 5.913 5.901 5.886 5.884 5.870 5.858 5.844 5.351 5.332 5.328 5.289 5.285 5.247 5.241 5.239 5.216 5.213 4.599 4.596 4.585 4.581 4.562 4.548 4.534 4.171 4.152 4.148 4.137 4.129 4.120 2.484 2.459 2.443 2.424 2.407 2.054 1.612 1.595 1.571 1.553 1.472 1.453 1.437 1.409 1.392 1.284 1.272 1.267 1.255 1.249 1.200 1.181 1.163 1.090 0.989 0.978 0.972 0.968 0.958 0.946 0.026
NAME qsw-7-13
EXPNO 1
PROCNO 1
Date_ 20131231
Time 0.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 175.2
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBDPSO
AllylO
NHAlloc
OTmse
BocHN
SePh
27
400MHz, CDCl3
4.04 2.16 6.12 5.08 0.67 2.86 2.03 2.00 0.70 5.48 5.88 1.82 2.92 0.81 4.02 1.10 1.38 1.00 2.98 12.26 5.92 9.06 2.14 9.36
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

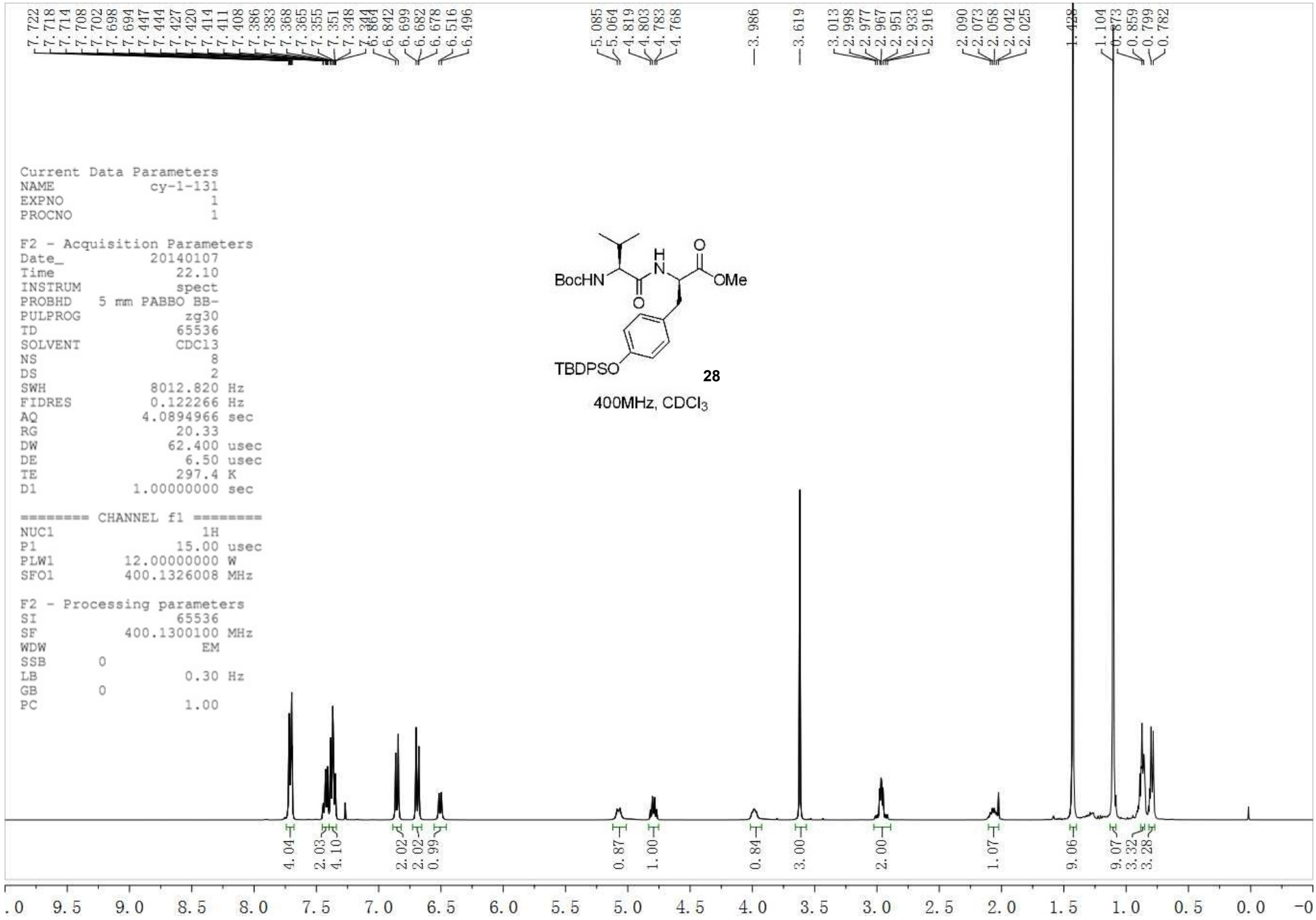

173.007
172.139
171.512
171.167
169.804
169.658
156.324
155.286
153.837
135.525
134.730
133.055
132.210
131.607
129.837
129.237
128.949
128.058
127.724
119.579
118.831
118.320
80.656
70.337
66.242
65.965
62.888
56.767
56.556
54.563
52.206
50.948
39.891
34.481
30.538
28.301
28.241
17.268
17.116
15.658
-1.495
TBDPSO
AllylO
NHAlloc
OTmse
BocHN
SePh
27
400MHz, CDCl3
NAME qsw-6-51-13C
EXPNO 2
PROCNO 1
Date_ 20130920
Time 23.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10083
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 0.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

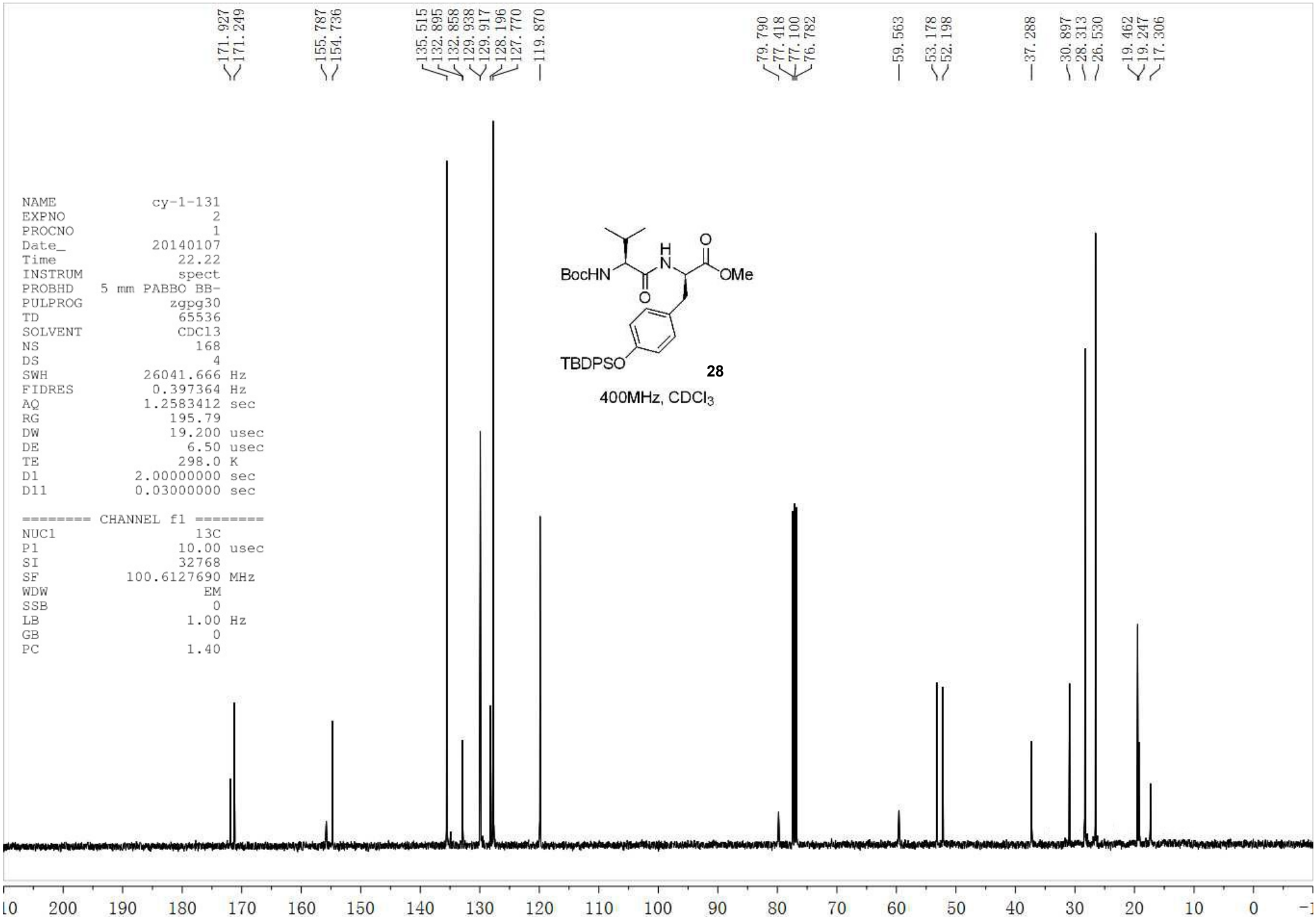

TBDPSO
OTmse
BocHN
SePh
3
500MHz, CDCl3
NAME qsw-6-119
EXPNO 1
PROCNO 1
Date_ 20131129
Time 6.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 161.3
DW 48.400 usec
DE 6.50 usec
TE 299.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.70 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

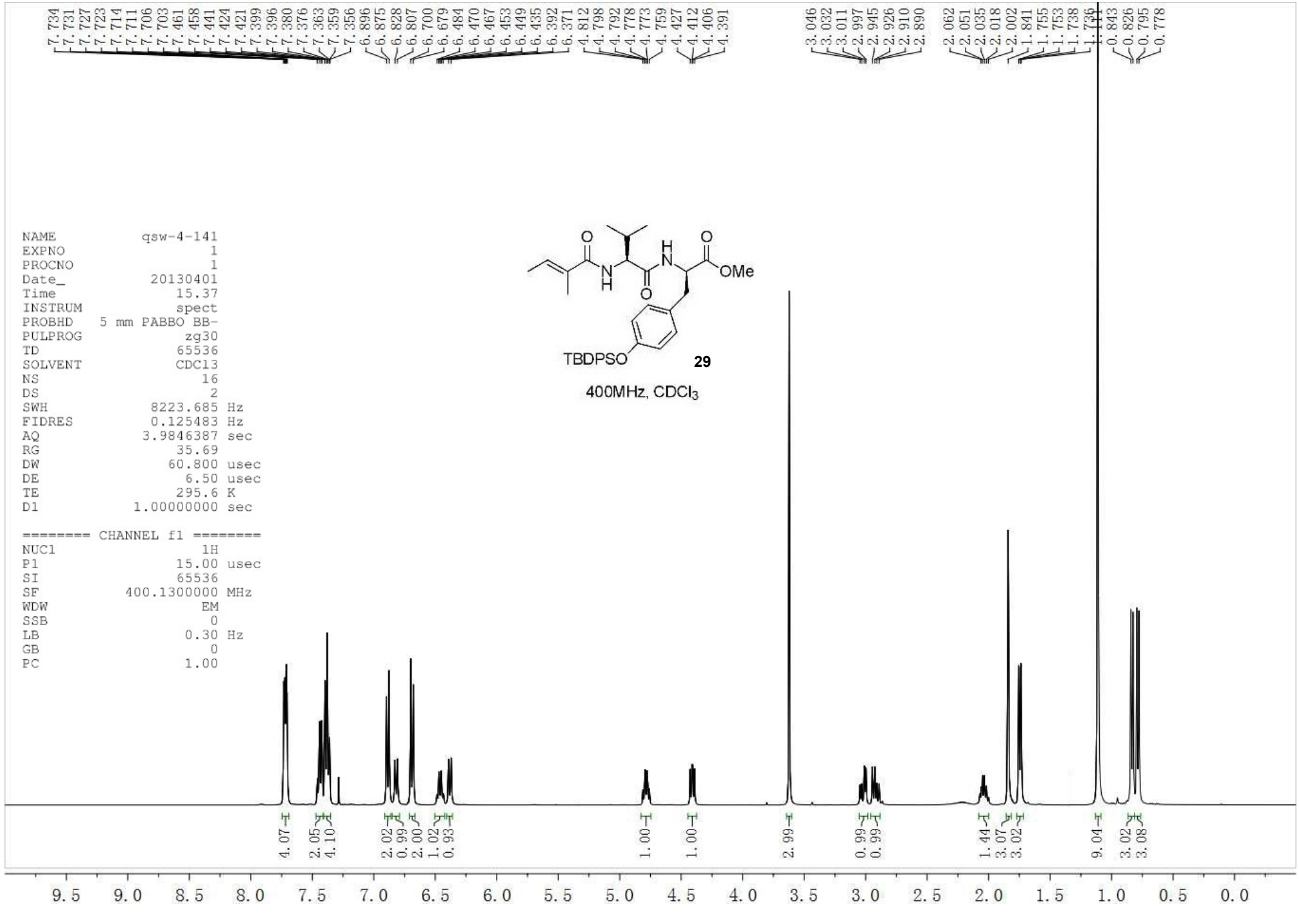

173.601
173.256
172.219
171.272
170.687
169.390
156.929
153.597
135.533
134.704
134.085
133.147
129.806
129.312
129.032
128.847
127.955
127.731
119.415
80.509
72.323
62.869
57.383
57.071
52.383
51.818
51.754
40.979
34.478
31.153
30.442
28.354
27.063
26.571
25.141
19.481
17.991
17.571
17.301
16.997
-1.474
TBDPSO
OTmse
BocHN
SePh
3
400MHz, CDCl3
NAME qsw-7-37-13C
EXPNO 2
PROCNO 1
Date_ 20140228
Time 23.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 9901
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 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 -10

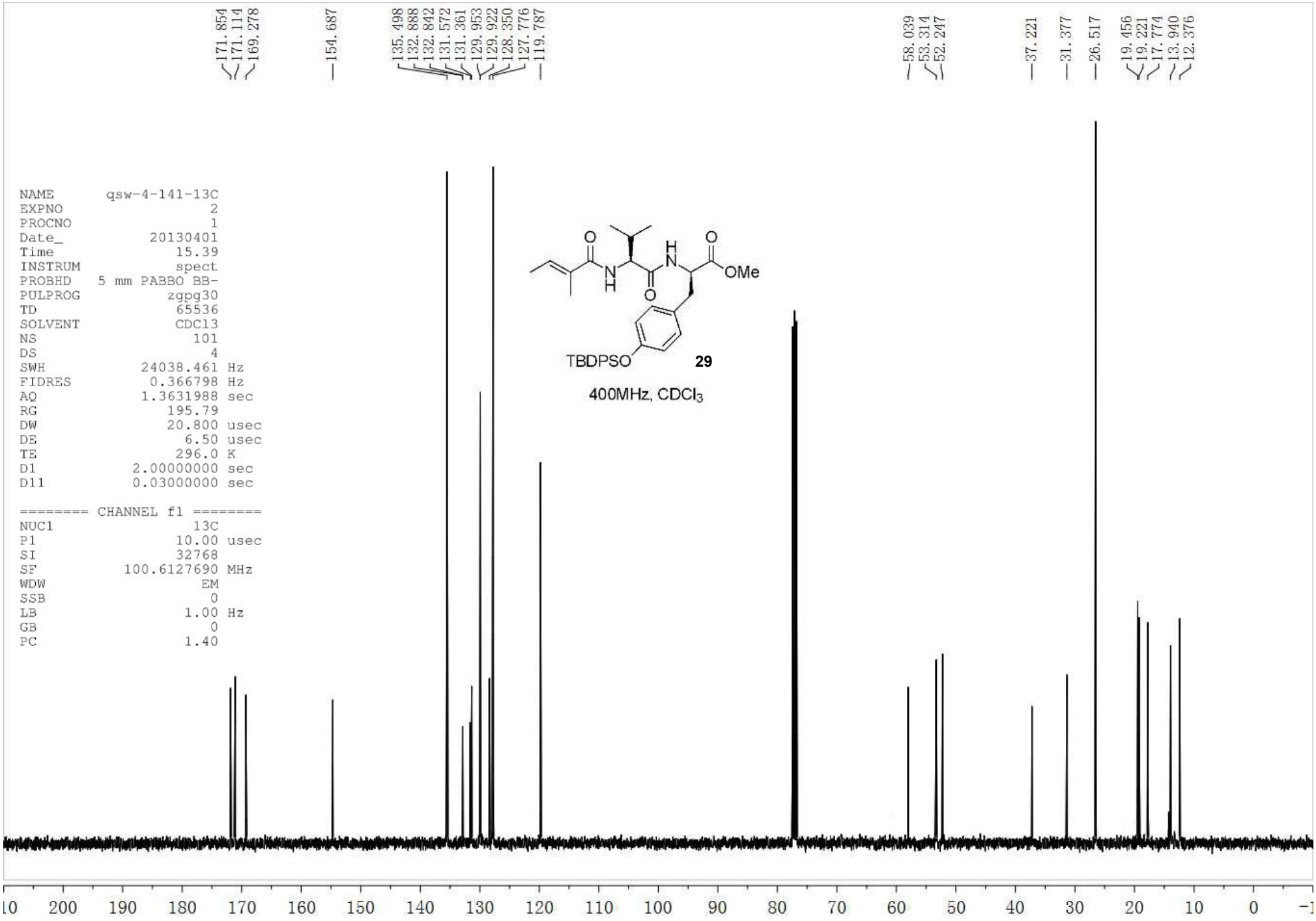

7.268
6.949
6.928
6.740
6.723
6.703
6.677
6.659
5.133
5.111
4.883
4.867
4.847
4.832
3.996
3.721
3.063
3.049
3.028
3.013
2.997
2.978
2.961
2.179
2.162
2.146
2.131
2.115
2.098
1.450
0.889
0.873
0.812
0.795
BocHN
O
N
H
O
OMe
HO
28-1
400MHz, CDCl3
NAME cy-1-130shoupu
EXPNO 1
PROCNO 1
Date_ 20140107
Time 21.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 127.33
DW 62.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.03
3.54
1.00
1.00
0.99
3.02
2.00
1.01
9.25
2.99
3.01
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

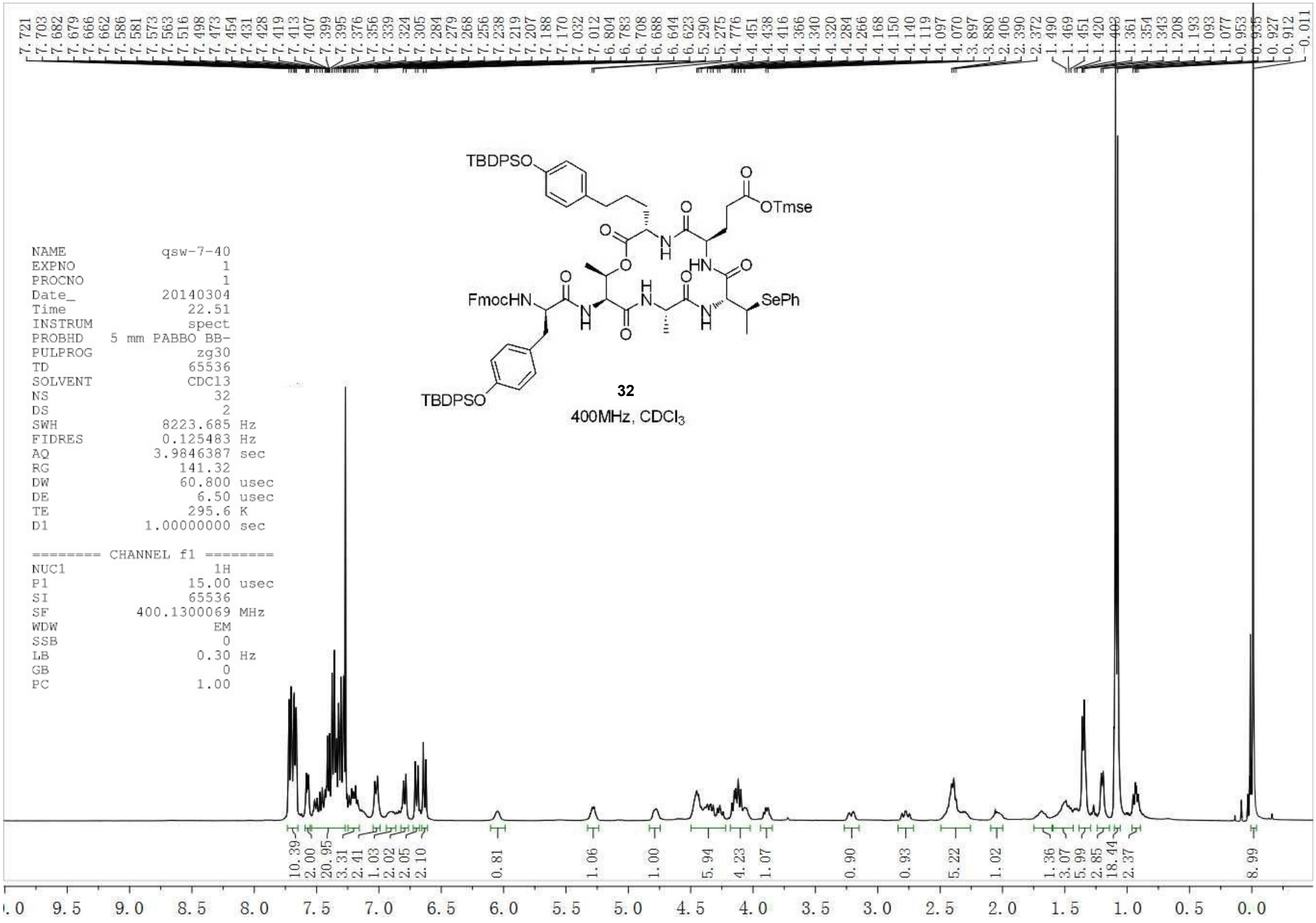

172.212
171.561
156.008
155.445
130.198
126.959
115.670
80.240
77.353
76.718
59.699
53.197
52.433
37.268
30.636
29.696
28.309
19.273
17.172
NAME cy-1-130shoupu
EXPNO 2
PROCNO 1
Date_ 20140107
Time 21.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 771
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
BocHN
O
H
N
O
O
OMe
HO
28-1
400MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

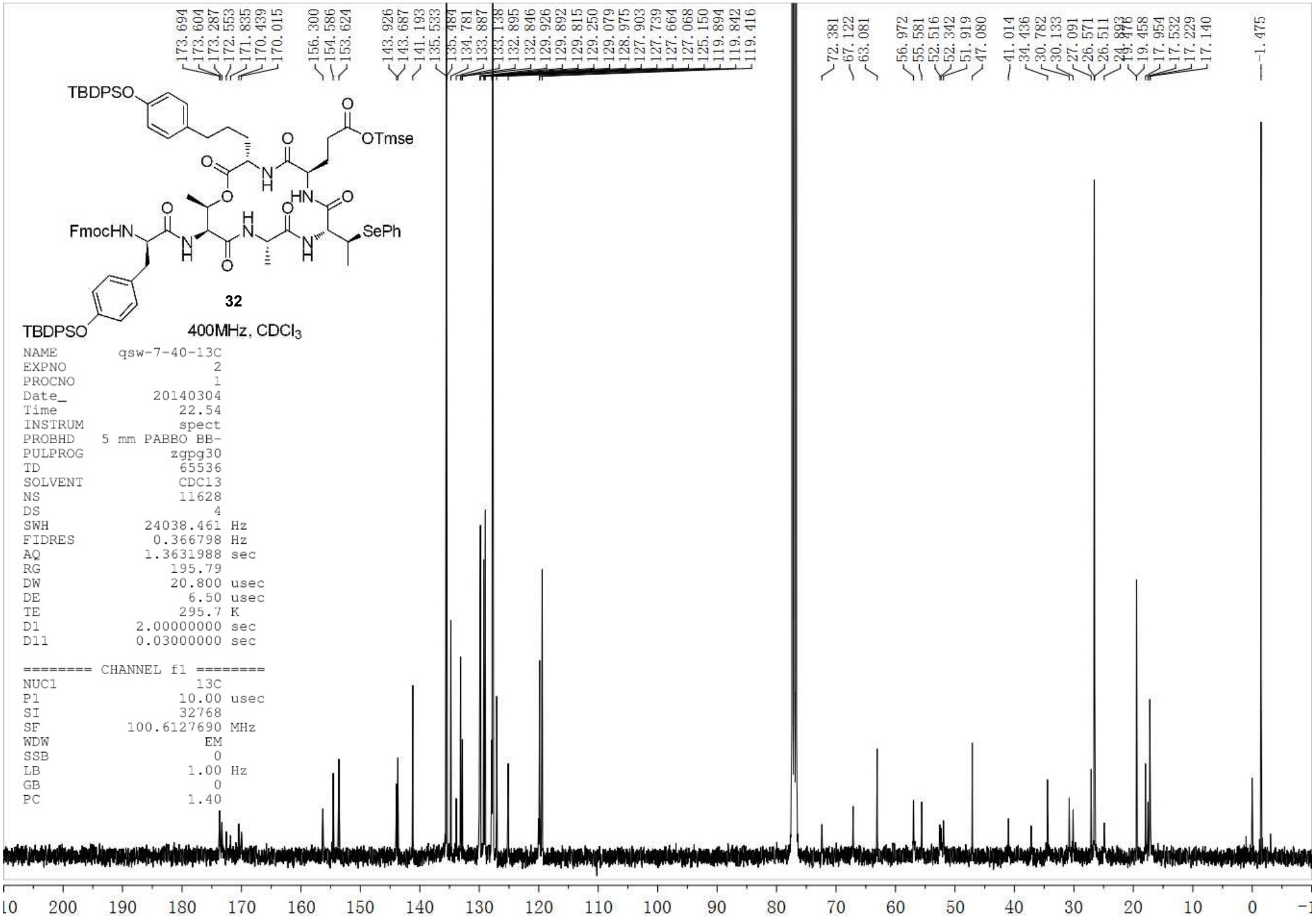

7.722
7.718
7.714
7.708
7.702
7.698
7.694
7.447
7.444
7.427
7.420
7.414
7.411
7.408
7.386
7.383
7.368
7.365
7.355
7.351
7.348
6.842
6.699
6.682
6.678
6.516
6.496
5.085
5.064
4.819
4.803
4.783
4.768
3.986
3.619
3.013
2.998
2.977
2.967
2.951
2.933
2.916
2.090
2.073
2.058
2.042
2.025
1.428
1.104
0.873
0.859
0.799
0.782
Current Data Parameters
NAME cy-1-131
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140107
Time 22.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 20.33
DW 62.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BocHN
OMe
TBDPSO
28
400MHz, CDCl3
4.04
2.03
4.10
2.02
2.02
0.99
0.87
1.00
0.84
3.00
2.00
1.07
9.06
9.07
3.32
3.28
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0

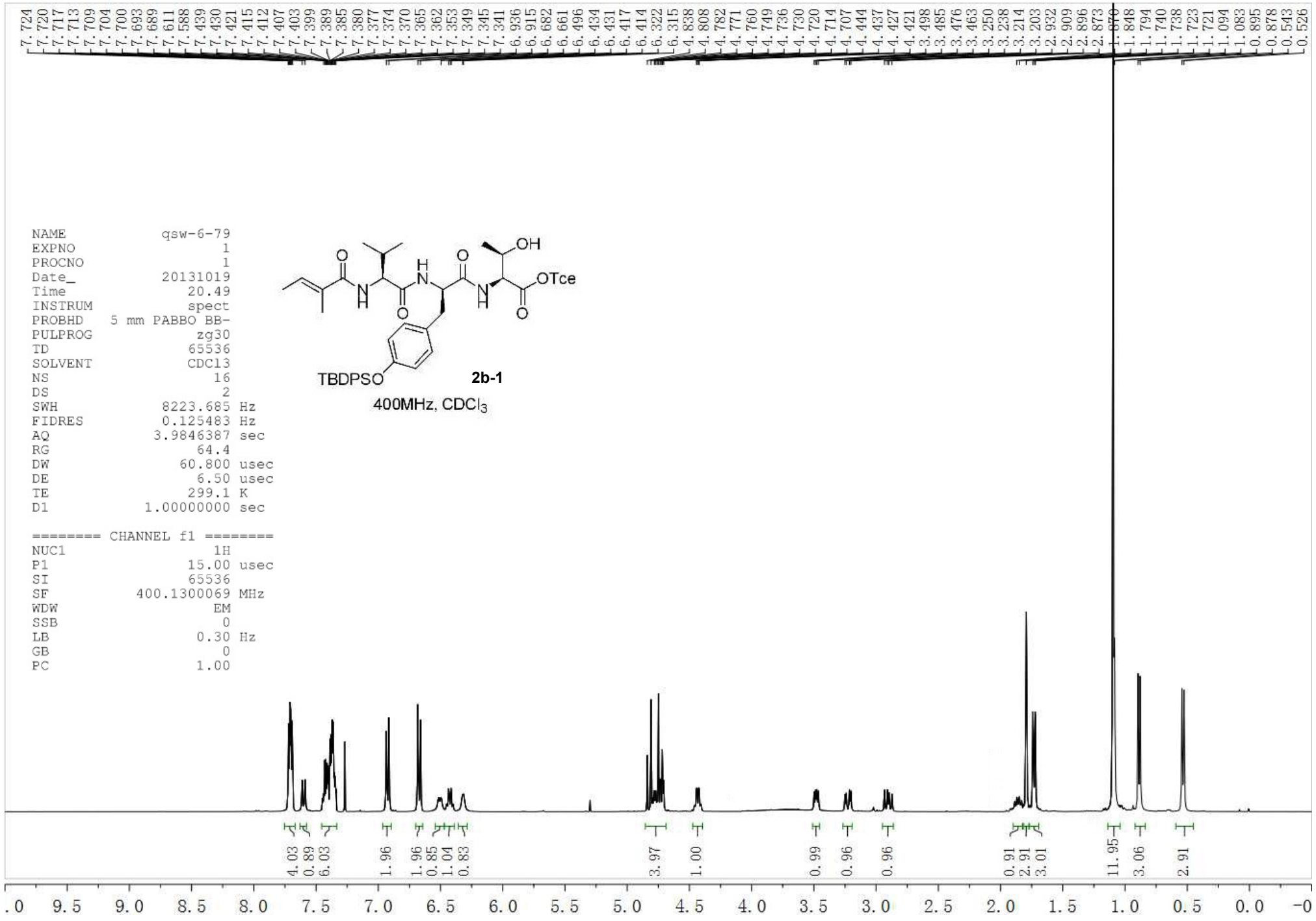

171.927
171.249
155.787
154.736
135.515
132.895
132.858
129.938
129.917
128.196
127.770
119.870
79.790
77.418
77.100
76.782
59.563
53.178
52.198
37.288
30.897
28.313
26.530
19.462
19.247
17.306
NAME cy-1-131
EXPNO 2
PROCNO 1
Date_ 20140107
Time 22.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 168
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
BocHN
H
N
O
O
OMe
TBDPSO
28
400MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

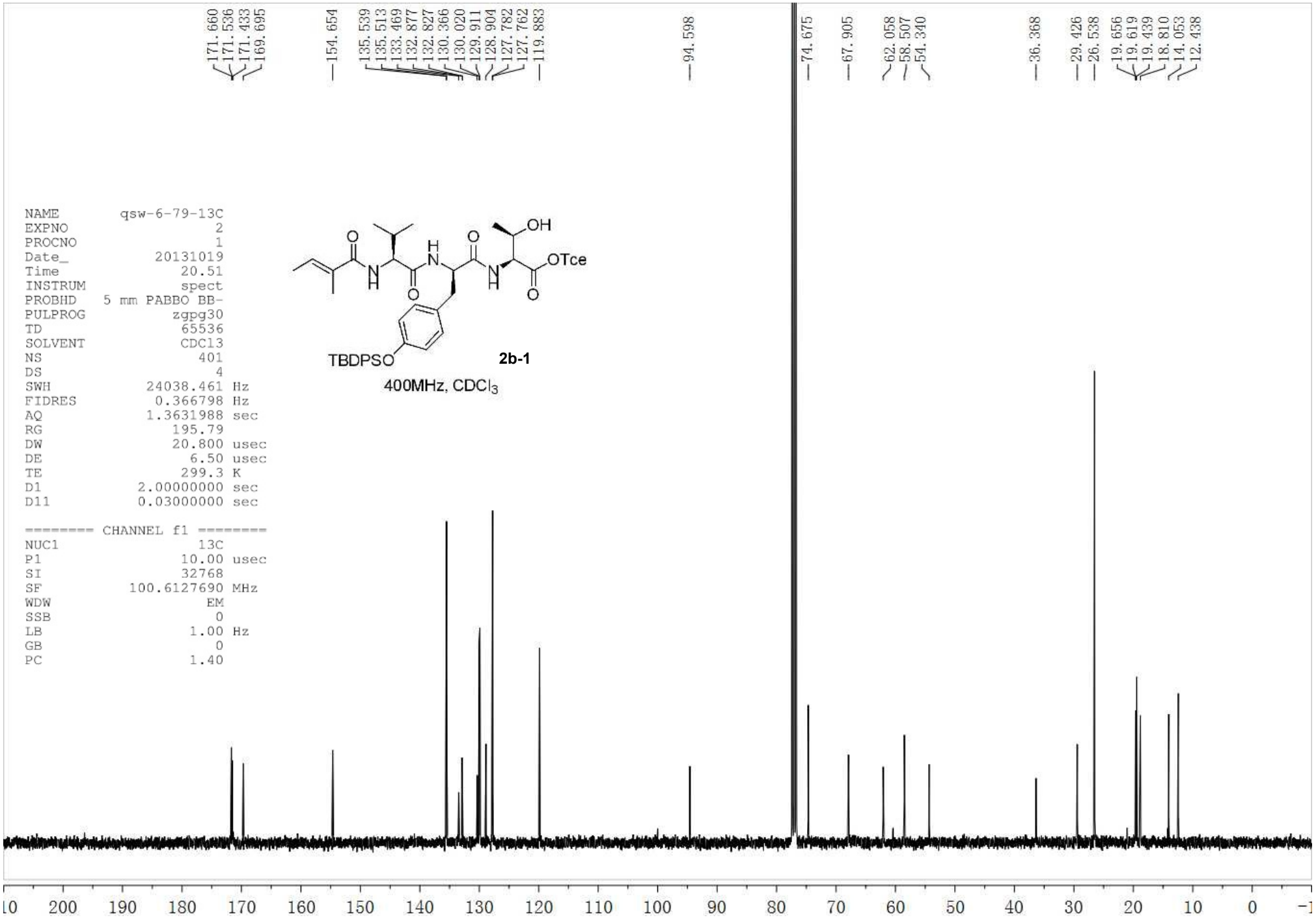

7.734 7.731 7.727 7.723 7.714 7.711 7.706 7.703 7.461 7.458 7.441 7.424 7.421 7.399 7.396 7.380 7.376 7.363 7.359 7.356
6.896 6.875 6.828 6.807 6.700 6.679 6.484 6.470 6.467 6.453 6.449 6.435 6.392 6.371
4.812 4.798 4.792 4.778 4.773 4.759 4.427 4.412 4.406 4.391
3.046 3.032 3.011 2.997 2.945 2.926 2.910 2.890
2.062 2.051 2.035 2.018 2.002 1.841 1.755 1.753 1.738 1.736 1.411
0.843 0.826 0.795 0.778
NAME qsw-4-141
EXPNO 1
PROCNO 1
Date_ 20130401
Time 15.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 35.69
DW 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
TBDPSO
29
400MHz, CDCl3
4.07 2.05 4.10 2.02 0.99 2.00 1.02 0.93 1.00 1.00 2.99 0.99 0.99 1.44 3.07 3.02 9.04 3.02 3.08
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

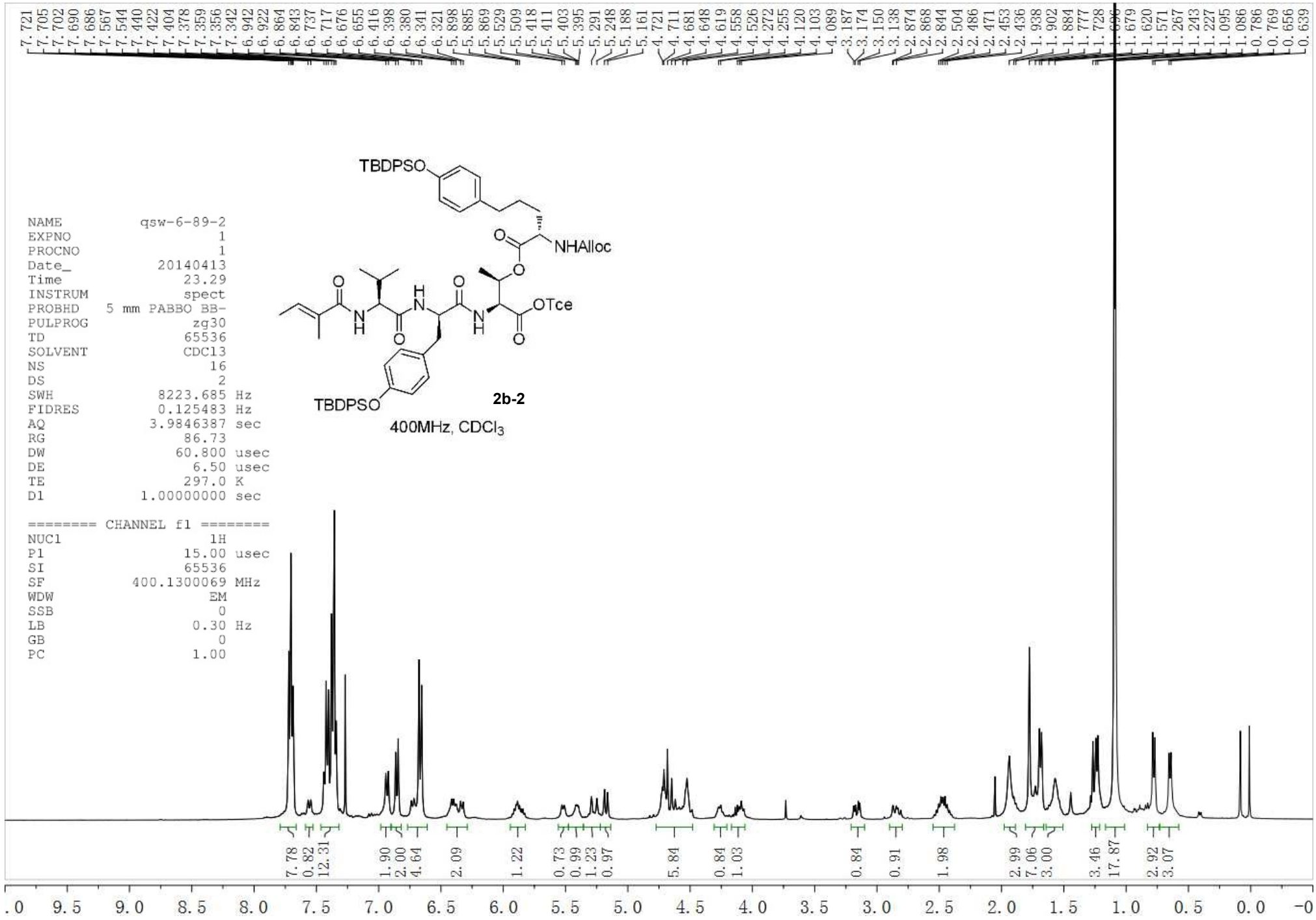

171.854
171.114
169.278
154.687
135.498
132.888
132.842
131.572
131.361
129.953
129.922
128.350
127.776
119.787
58.039
53.314
52.247
37.221
31.377
26.517
19.456
19.221
17.774
13.940
12.376
NAME qsw-4-141-13C
EXPNO 2
PROCNO 1
Date_ 20130401
Time 15.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 101
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
TBDPSO
OMe
29
400MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

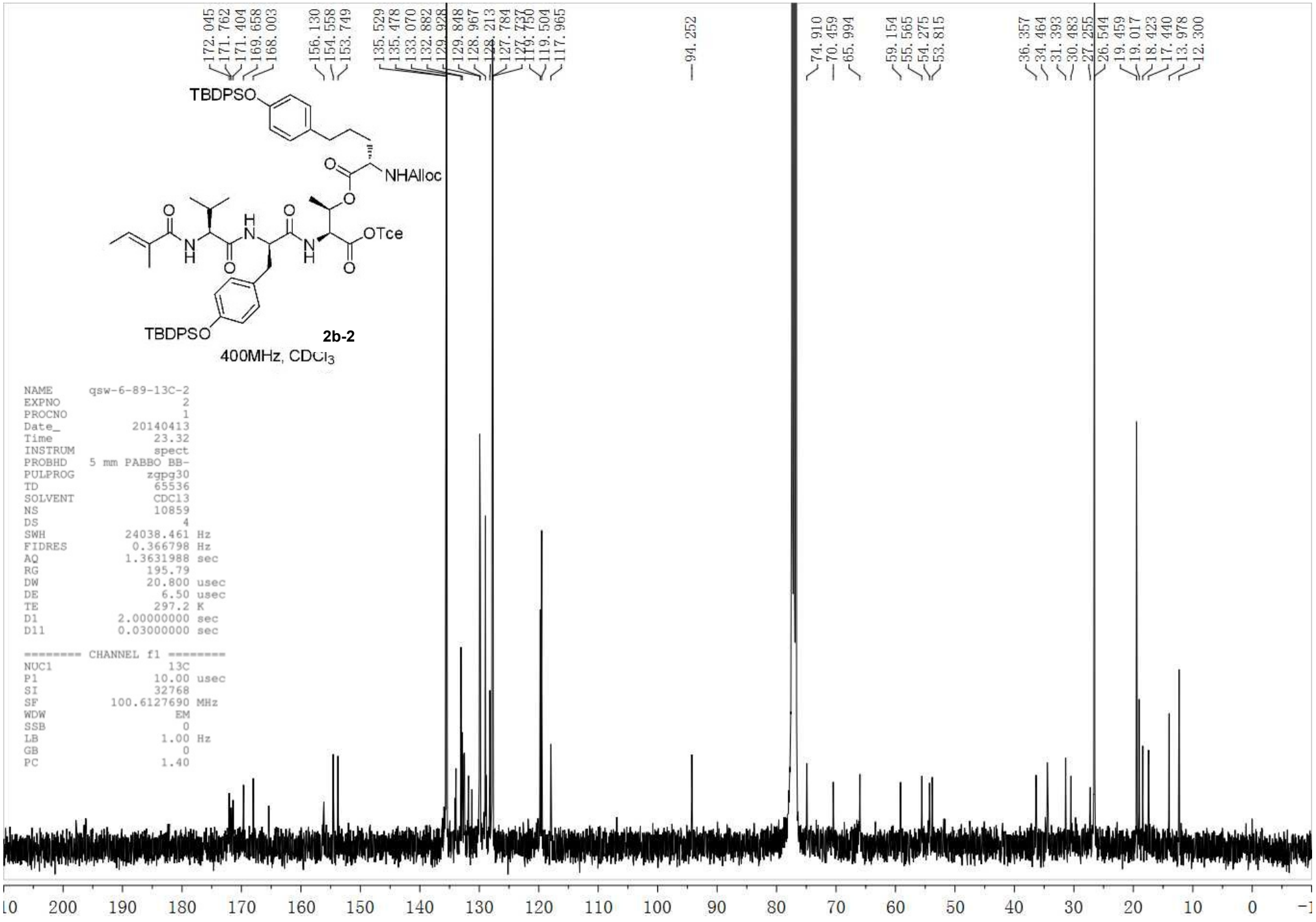

7.721 7.703 7.682 7.679 7.666 7.662 7.586 7.581 7.573 7.563 7.516 7.498 7.473 7.454 7.431 7.428 7.419 7.413 7.407 7.399 7.395 7.376 7.356 7.339 7.324 7.305 7.284 7.279 7.268 7.256 7.238 7.219 7.207 7.188 7.170 7.032 7.012 6.804 6.783 6.708 6.688 6.644 6.623 5.290 5.275 4.776 4.451 4.438 4.416 4.366 4.340 4.320 4.284 4.266 4.168 4.150 4.140 4.119 4.097 4.070 3.897 3.880 2.406 2.390 2.372 1.490 1.469 1.451 1.420 1.403 1.361 1.354 1.343 1.208 1.193 1.093 1.077 0.953 0.935 0.927 0.912 -0.011
TBDPSO
OTmse
FmocHN
SePh
TBDPSO
32
400MHz, CDCl3
NAME qsw-7-40
EXPNO 1
PROCNO 1
Date_ 20140304
Time 22.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 141.32
DW 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.39 2.00 20.95 3.31 2.41 1.03 2.02 2.05 2.10 0.81 1.06 1.00 5.94 4.23 1.07 0.90 0.93 5.22 1.02 1.36 3.07 5.99 2.85 18.44 2.37 8.99
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

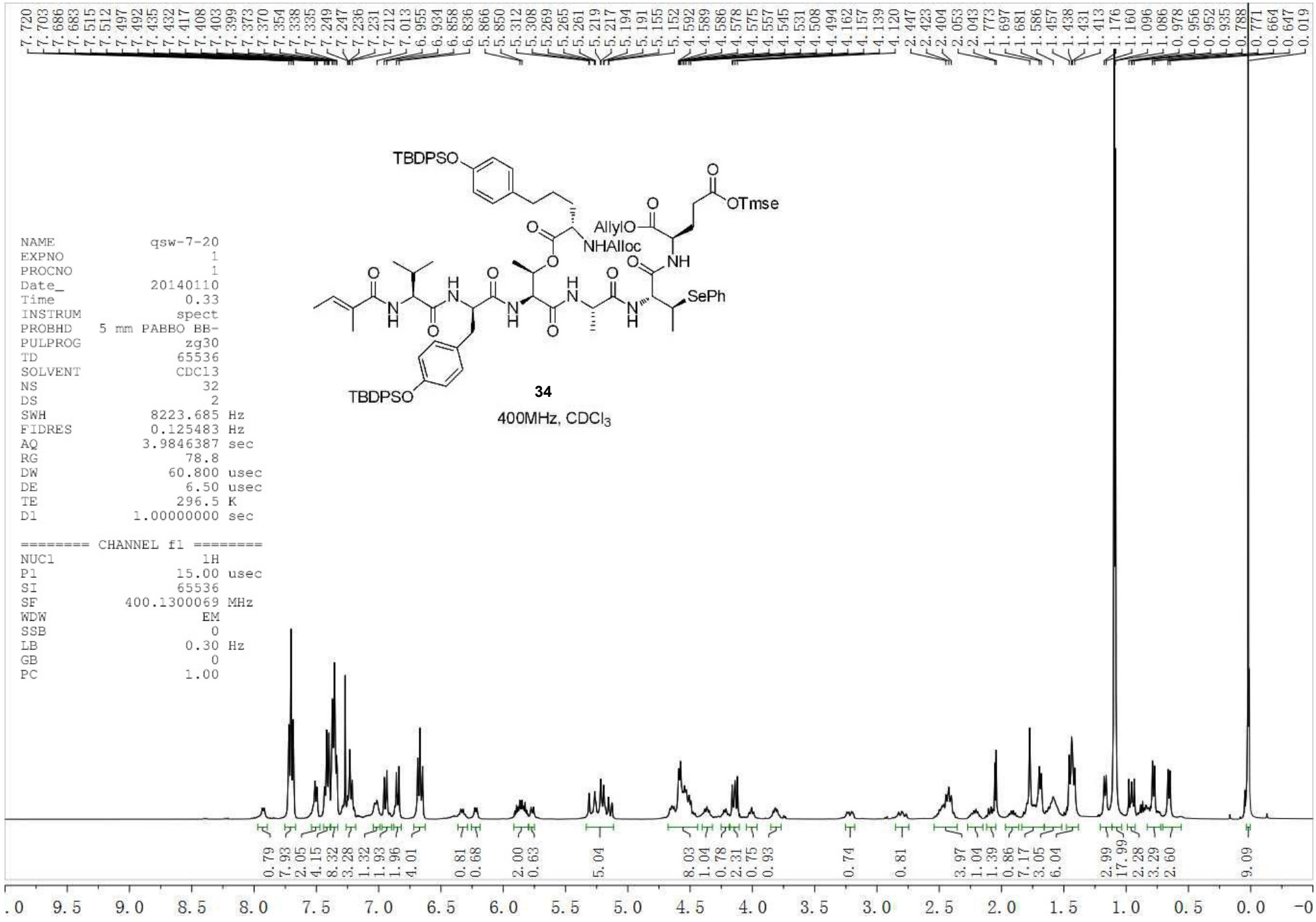

TBDPSO
OTmse
FmocHN
SePh
TBDPSO
32
400MHz, CDCl3
NAME qsw-7-40-13C
EXPNO 2
PROCNO 1
Date_ 20140304
Time 22.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 11628
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

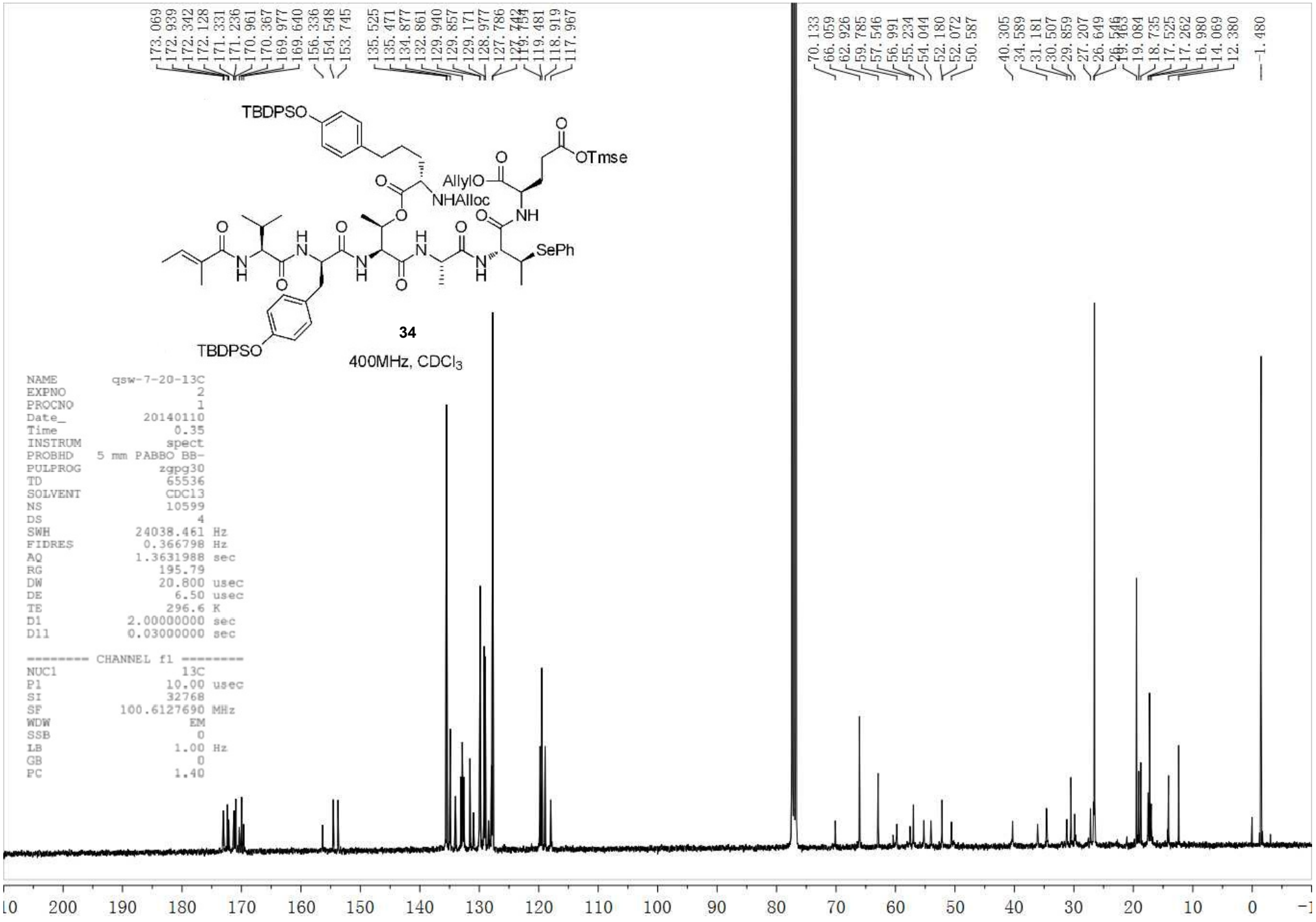

7.724 7.720 7.717 7.713 7.709 7.704 7.700 7.693 7.689 7.611 7.588 7.439 7.430 7.421 7.415 7.412 7.407 7.403 7.399 7.389 7.385 7.380 7.377 7.374 7.370 7.365 7.362 7.353 7.349 7.345 7.341 6.936 6.915 6.682 6.661 6.496 6.434 6.431 6.417 6.414 6.322 6.315 4.838 4.808 4.782 4.771 4.760 4.749 4.736 4.730 4.720 4.714 4.707 4.444 4.437 4.427 4.421 3.498 3.485 3.476 3.463 3.250 3.238 3.214 3.203 2.932 2.909 2.896 2.873 1.870 1.848 1.794 1.740 1.738 1.723 1.721 1.094 1.083 0.895 0.878 0.543 0.526
NAME qsw-6-79
EXPNO 1
PROCNO 1
Date_ 20131019
Time 20.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 64.4
DW 60.800 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
OTce
TBDPSO
2b-1
400MHz, CDCl3
4.03 0.89 6.03 1.96 1.96 0.85 1.04 0.83 3.97 1.00 0.99 0.96 0.96 0.91 2.91 3.01 11.95 3.06 2.91
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

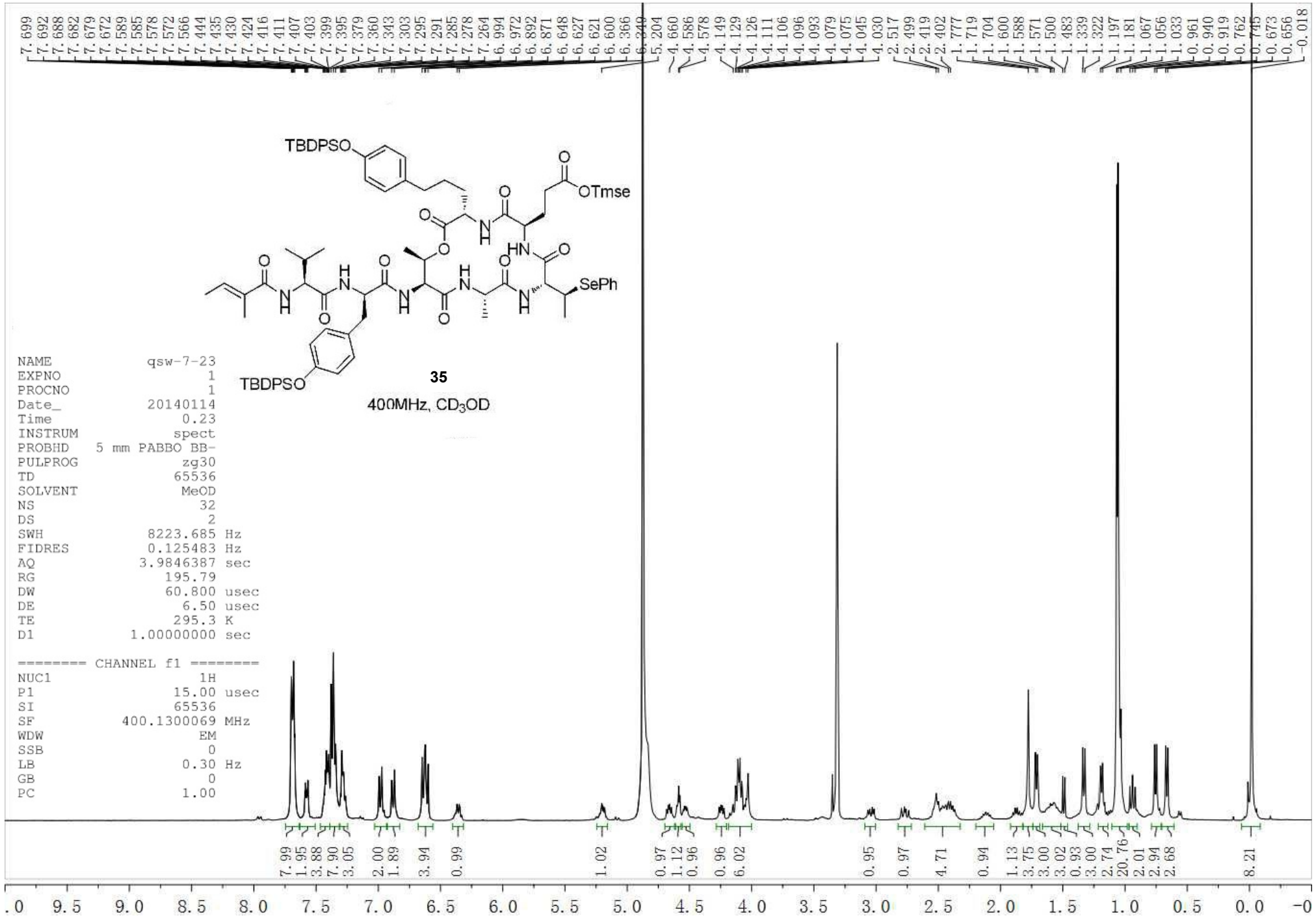

171.660
171.536
171.433
169.695
154.654
135.539
135.513
133.469
132.877
132.827
130.366
130.020
129.911
128.904
127.782
127.762
119.883
94.598
74.675
67.905
62.058
58.507
54.340
36.368
29.426
26.538
19.656
19.619
19.439
18.810
14.053
12.438
NAME qsw-6-79-13C
EXPNO 2
PROCNO 1
Date_ 20131019
Time 20.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 401
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
OTce
TBDPSO
2b-1
400MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

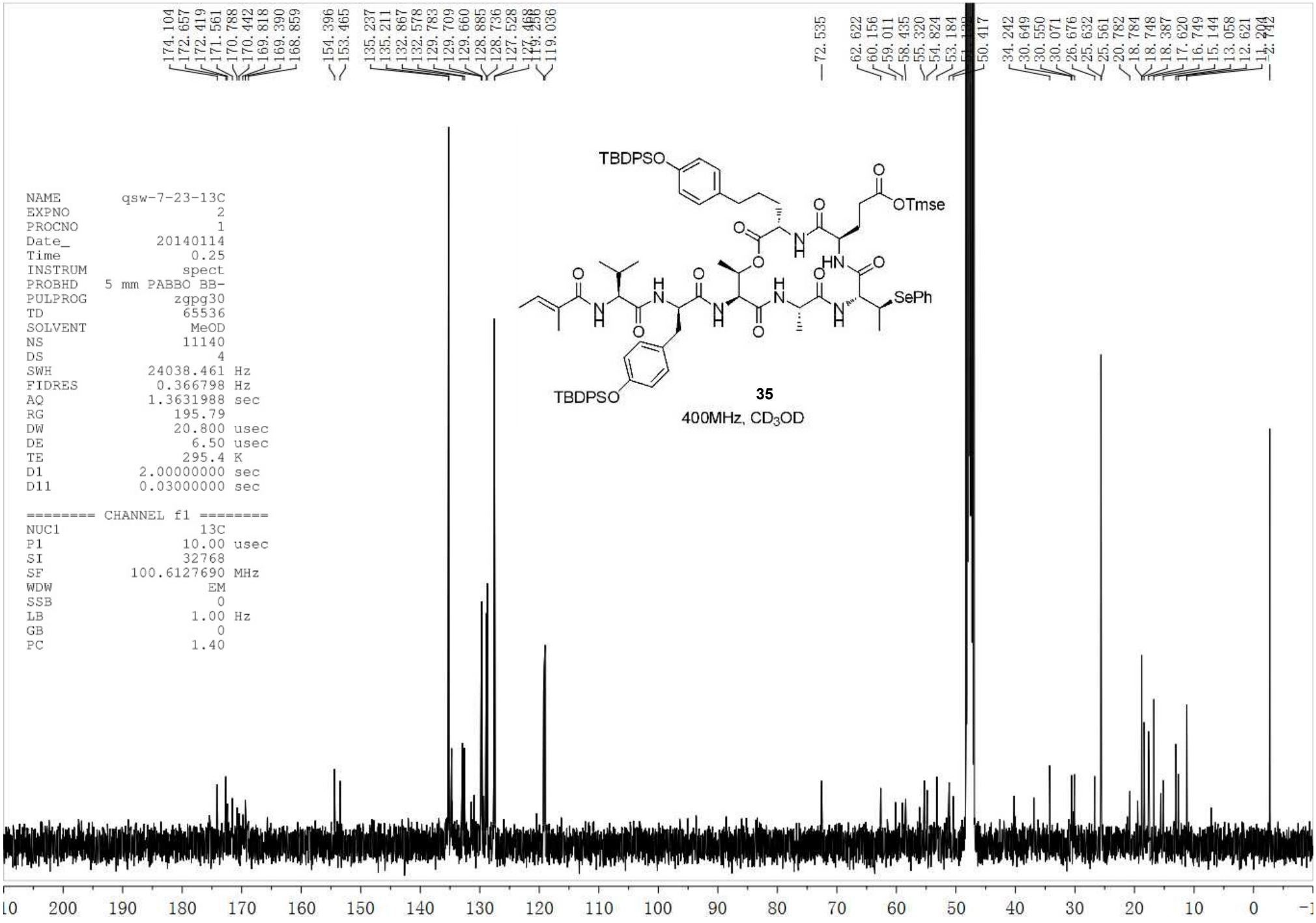

7.721 7.705 7.702 7.690 7.686 7.567 7.544 7.440 7.422 7.404 7.378 7.359 7.356 7.342 6.942 6.922 6.864 6.843 6.737 6.717 6.676 6.655 6.416 6.398 6.380 6.341 6.321 5.898 5.885 5.869 5.529 5.509 5.418 5.411 5.403 5.395 5.291 5.248 5.188 5.161 4.721 4.711 4.681 4.648 4.619 4.558 4.526 4.272 4.255 4.120 4.103 4.089 3.187 3.174 3.150 3.138 2.874 2.868 2.844 2.504 2.486 2.471 2.453 2.436 1.938 1.902 1.884 1.777 1.728 1.679 1.620 1.571 1.267 1.243 1.227 1.095 1.086 0.786 0.769 0.656 0.639
TBDPSO
NHAlloc
OTce
TBDPSO
2b-2
400MHz, CDCl3
NAME qsw-6-89-2
EXPNO 1
PROCNO 1
Date_ 20140413
Time 23.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 86.73
DW 60.800 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.78 0.82 12.31 1.90 2.00 4.64 2.09 1.22 0.73 0.99 1.23 0.97 5.84 0.84 1.03 0.84 0.91 1.98 2.99 7.06 3.00 3.46 17.87 2.92 3.07
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

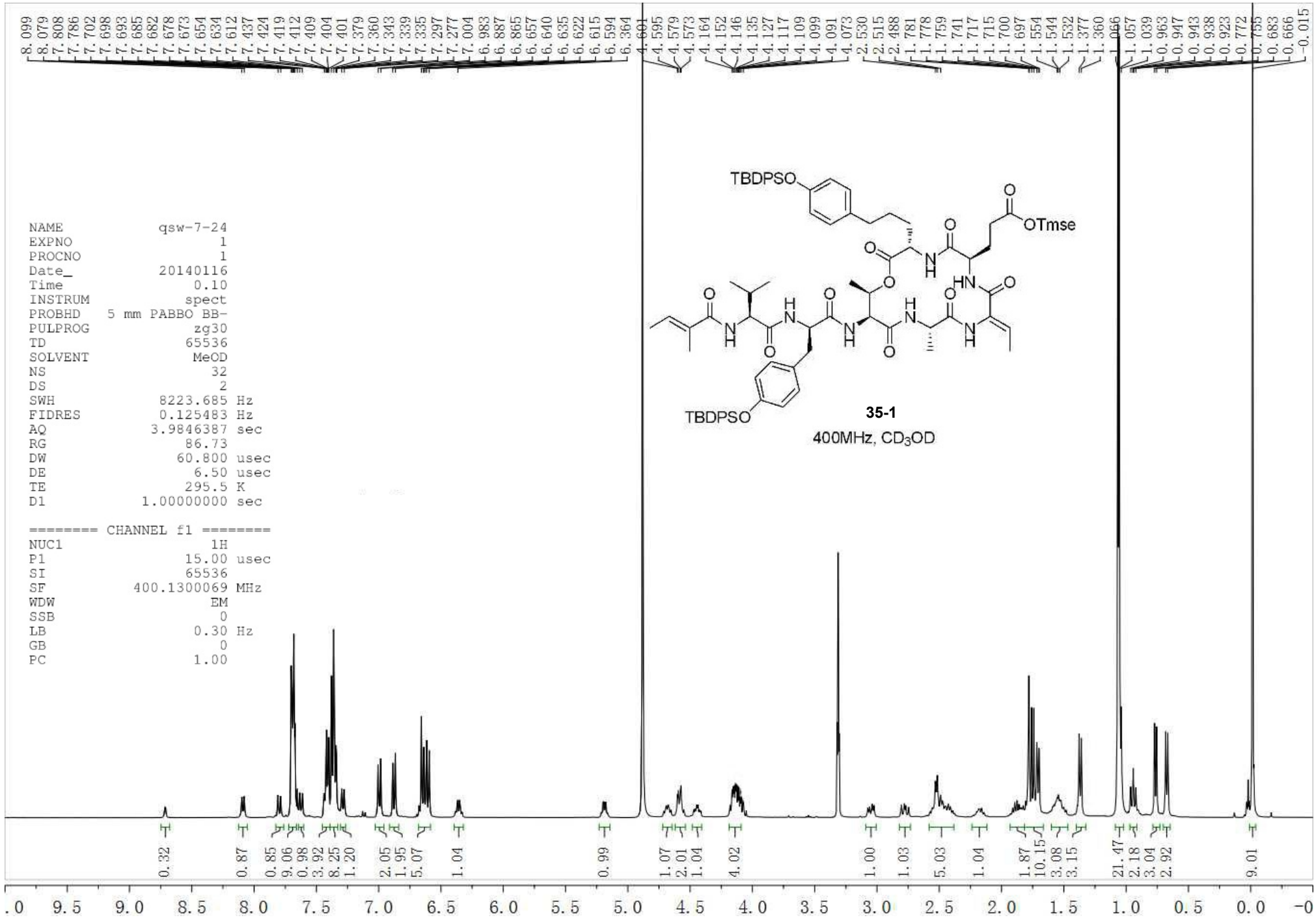

172.045
171.762
171.404
169.658
168.003
156.130
154.558
153.749
135.529
135.478
133.070
132.882
129.928
129.848
128.967
128.213
127.784
127.737
119.750
119.504
117.965
94.252
74.910
70.459
65.994
59.154
55.565
54.275
53.815
36.357
34.464
31.393
30.483
27.255
26.544
19.459
19.017
18.423
17.440
13.978
12.300
TBDPSO
NHAlloc
OTce
TBDPSO
2b-2
400MHz, CDCl3
NAME qsw-6-89-13C-2
EXPNO 2
PROCNO 1
Date_ 20140413
Time 23.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10859
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

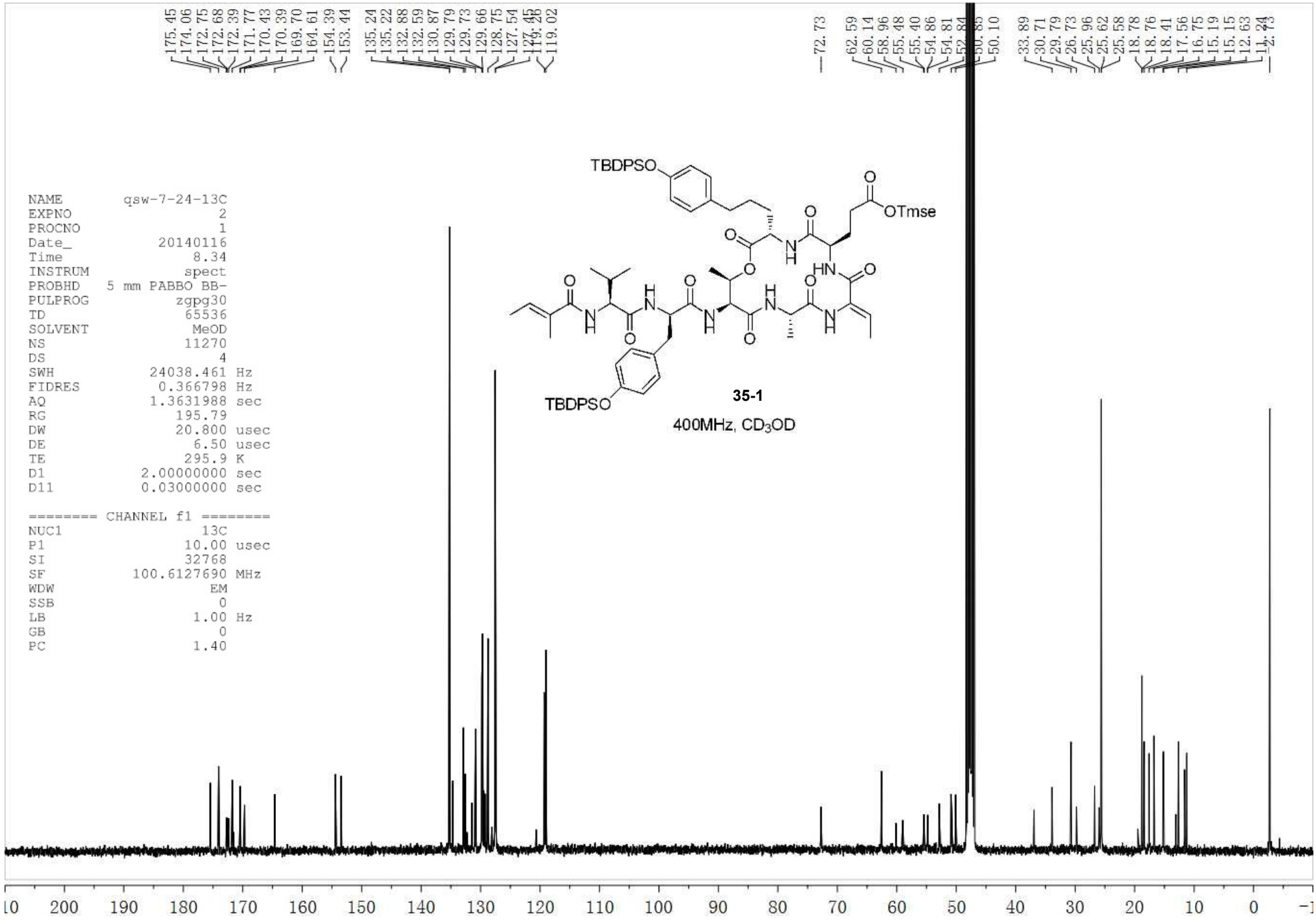

7.720 7.703 7.686 7.683 7.515 7.512 7.497 7.492 7.435 7.432 7.417 7.408 7.403 7.399 7.373 7.370 7.354 7.338 7.335 7.249 7.247 7.236 7.231 7.212 7.013 6.955 6.934 6.858 6.836 5.866 5.850 5.312 5.308 5.269 5.265 5.261 5.219 5.217 5.194 5.191 5.155 5.152 4.592 4.589 4.586 4.578 4.575 4.557 4.545 4.531 4.508 4.494 4.162 4.157 4.139 4.120 2.447 2.423 2.404 2.053 2.043 1.773 1.697 1.681 1.586 1.457 1.438 1.431 1.413 1.176 1.160 1.096 1.086 0.978 0.956 0.952 0.935 0.788 0.771 0.664 0.647 0.019
TBDPSO
AllylO
NHAlloc
OTmse
NH
SePh
TBDPSO
34
400MHz, CDCl3
NAME qsw-7-20
EXPNO 1
PROCNO 1
Date_ 20140110
Time 0.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 78.8
DW 60.800 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.79 7.93 2.05 4.15 8.32 3.28 1.32 1.93 1.96 4.01 0.81 0.68 2.00 0.63 5.04 8.03 1.04 0.78 2.31 0.75 0.93 0.74 0.81 3.97 1.04 1.39 0.86 7.17 3.05 6.04 2.99 17.99 2.28 3.29 2.60 9.09
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

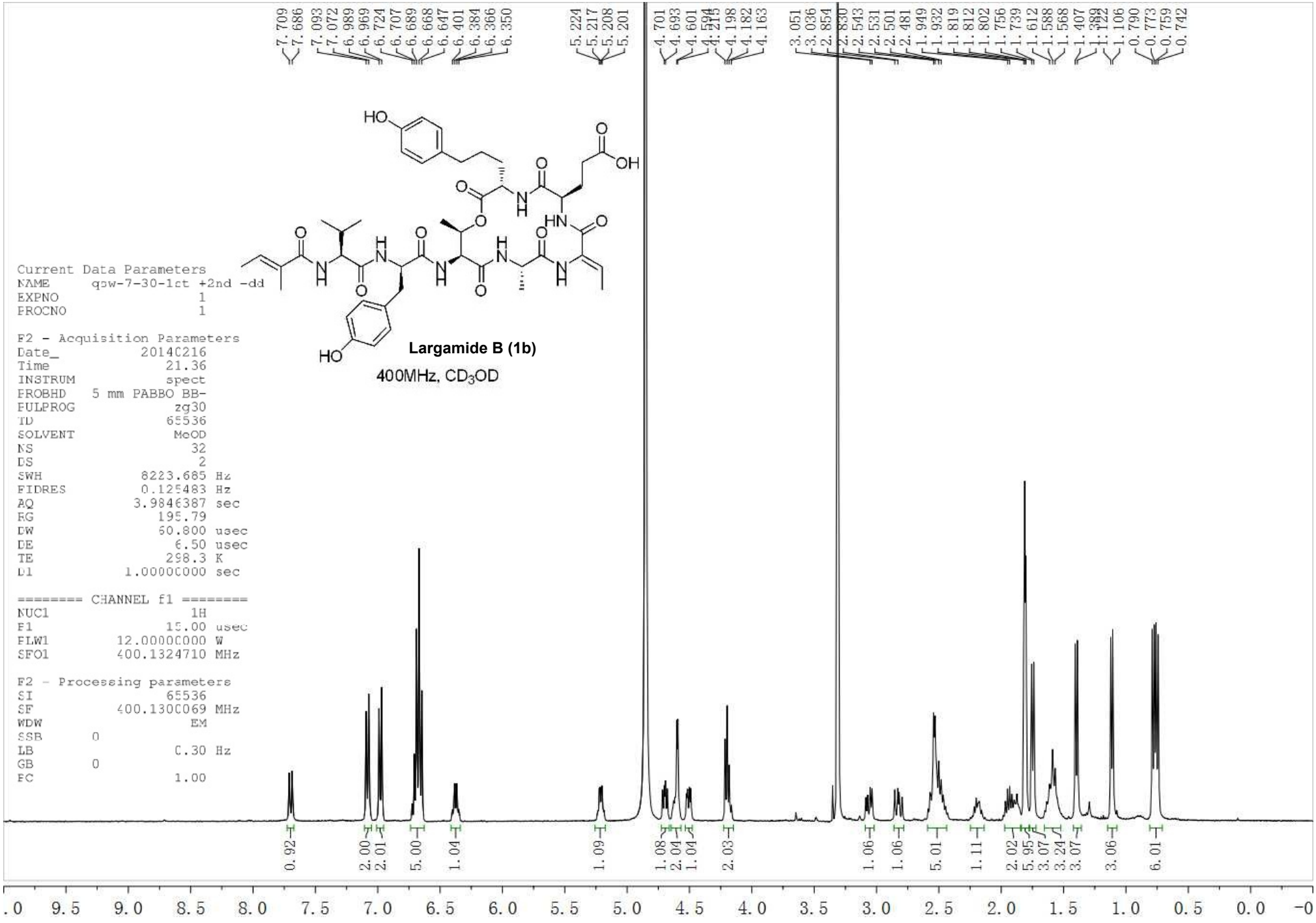

TBDPSO
AllylO
NHAlloc
OTmse
NH
SePh
TBDPSO
34
400MHz, CDCl3
NAME qsw-7-20-13C
EXPNO 2
PROCNO 1
Date_ 20140110
Time 0.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10599
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
-------- CHANNEL f1 --------
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

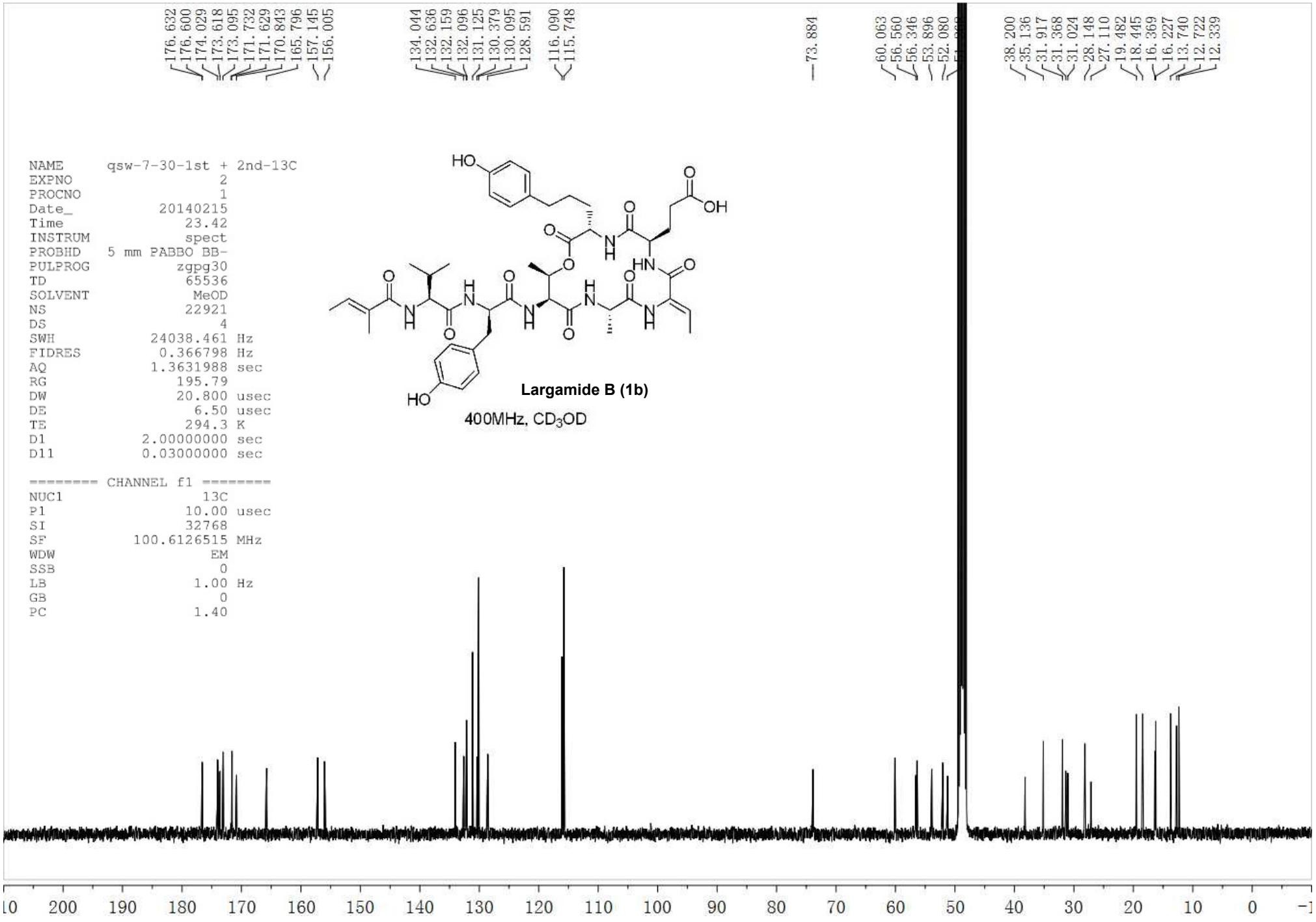

TBDPSO
OTmse
HN
SePh
TBDPSO
35
400MHz, CD3OD
NAME qsw-7-23
EXPNO 1
PROCNO 1
Date_ 20140114
Time 0.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 195.79
DW 60.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

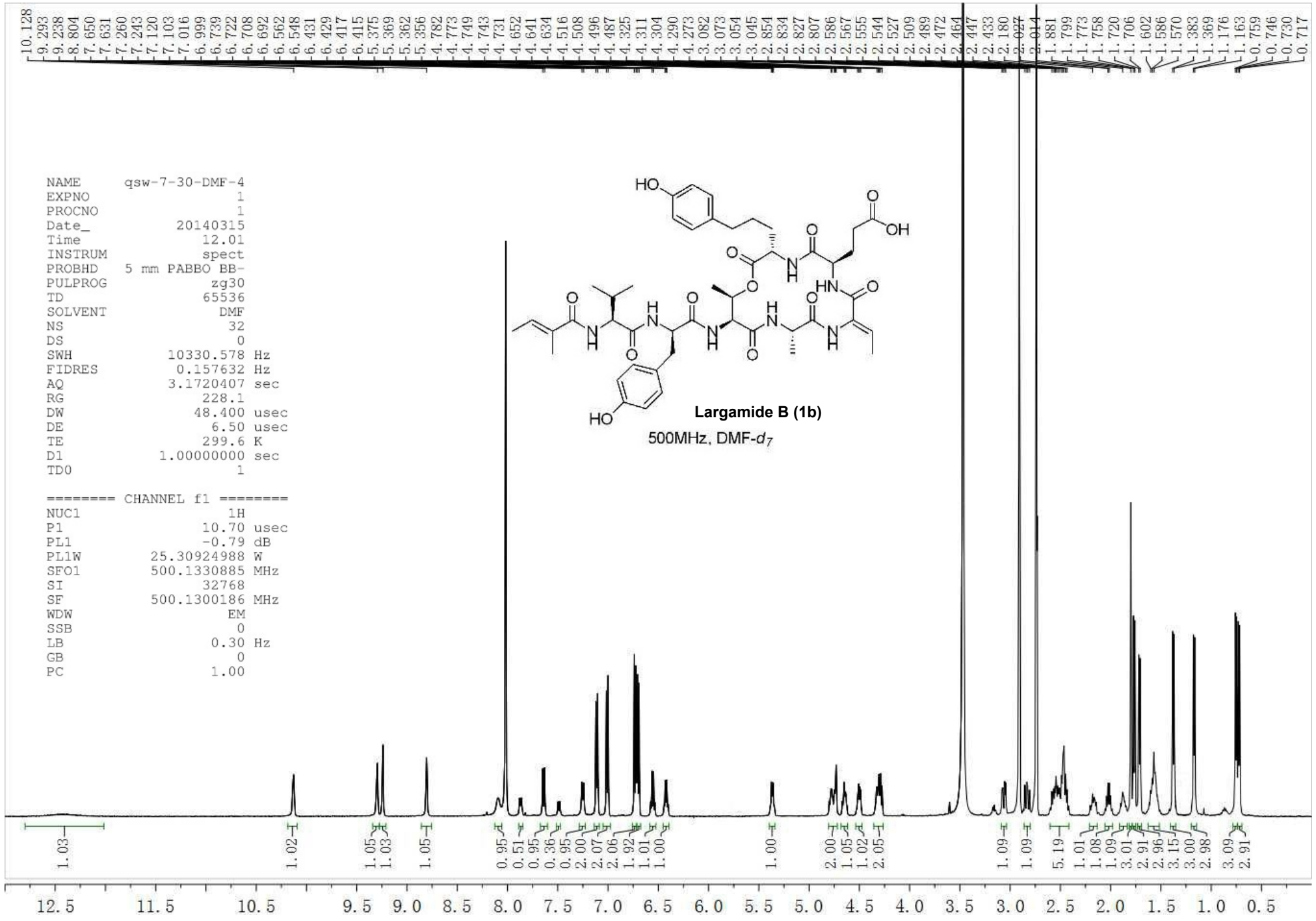

174.104
172.657
172.419
171.561
170.788
170.442
169.818
169.390
168.859
154.396
153.465
135.237
135.211
132.867
132.578
129.783
129.709
129.660
128.885
128.736
127.528
119.036
72.535
62.622
60.156
59.011
58.435
55.320
54.824
53.184
50.417
34.242
30.649
30.550
30.071
26.676
25.632
25.561
20.782
18.784
18.748
18.387
17.620
16.749
15.144
13.058
12.621
NAME qsw-7-23-13C
EXPNO 2
PROCNO 1
Date_ 20140114
Time 0.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 11140
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
TBDPSO
OTmse
HN
SePh
TBDPSO
35
400MHz, CD3OD
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

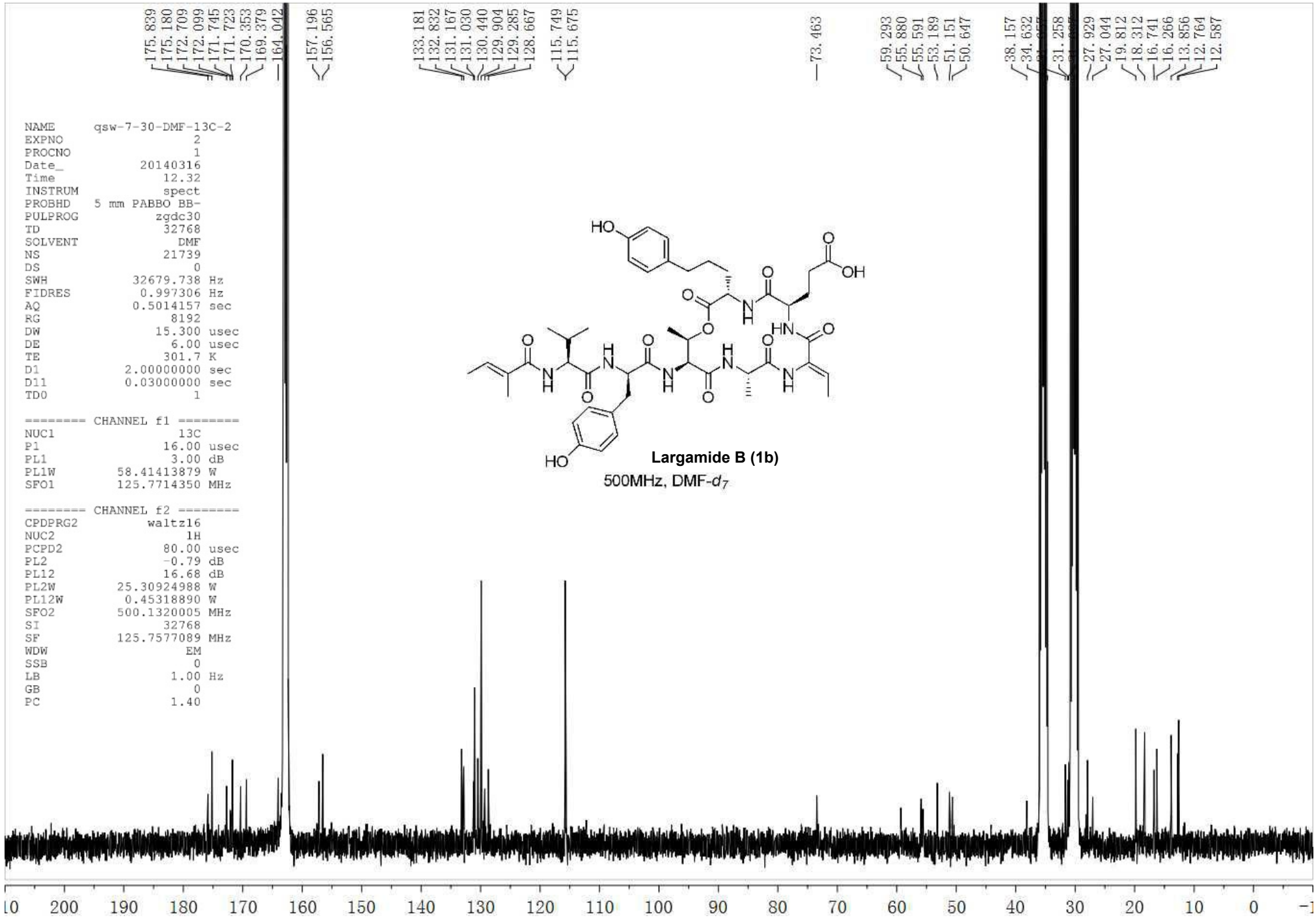

NAME qsw-7-24
EXPNO 1
PROCNO 1
Date_ 20140116
Time 0.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 86.73
DW 60.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBDPSO
OTmse
TBDPSO
35-1
400MHz, CD3OD

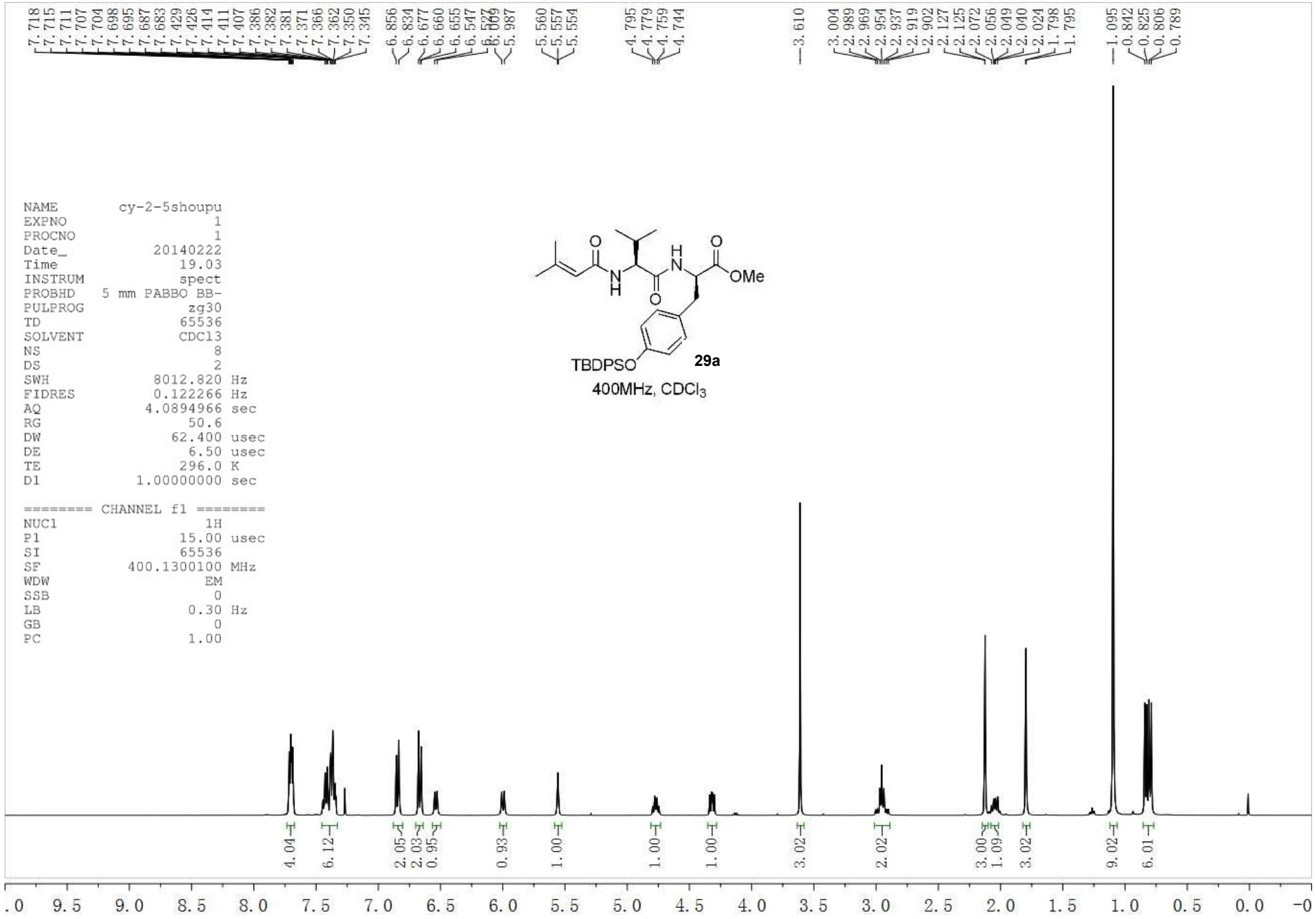

175.45
174.06
172.75
172.68
172.39
171.77
170.43
170.39
169.70
164.61
154.39
153.44
135.24
135.22
132.88
132.59
130.87
129.79
129.73
129.66
128.75
127.54
119.02
72.73
62.59
60.14
58.96
55.48
55.40
54.86
54.81
50.85
50.10
33.89
30.71
29.79
26.73
25.96
25.62
25.58
18.78
18.76
18.41
17.56
16.75
15.19
15.15
12.63
TBDPSO
OTmse
HN
TBDPSO
35-1
400MHz, CD3OD
NAME qsw-7-24-13C
EXPNO 2
PROCNO 1
Date_ 20140116
Time 8.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 11270
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

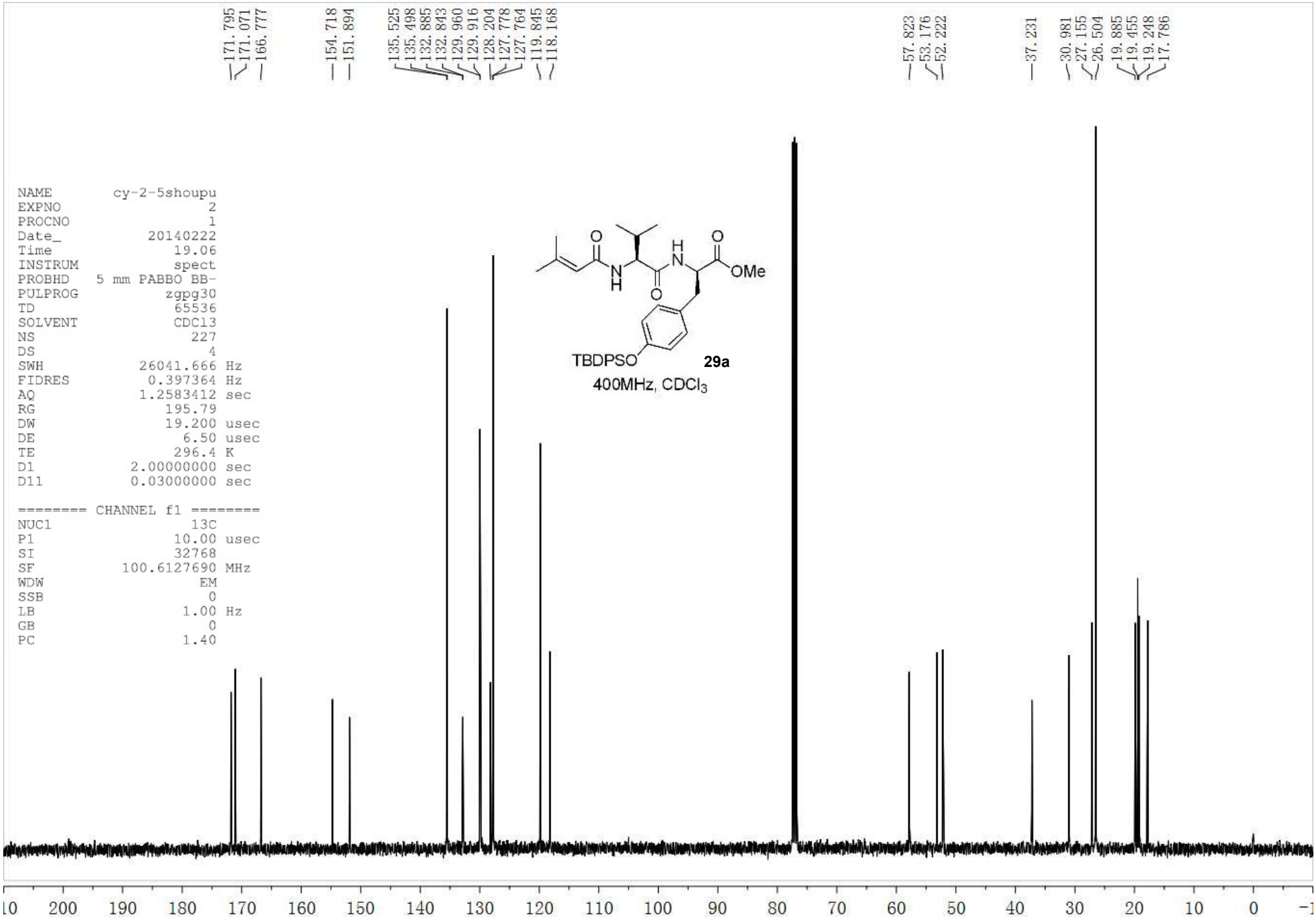

Current Data Parameters
NAME qsw-7-30-1ct +2nd -dd
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140216
Time 21.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 195.79
DW 60.800 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Largamide B (1b)
400MHz, CD3OD

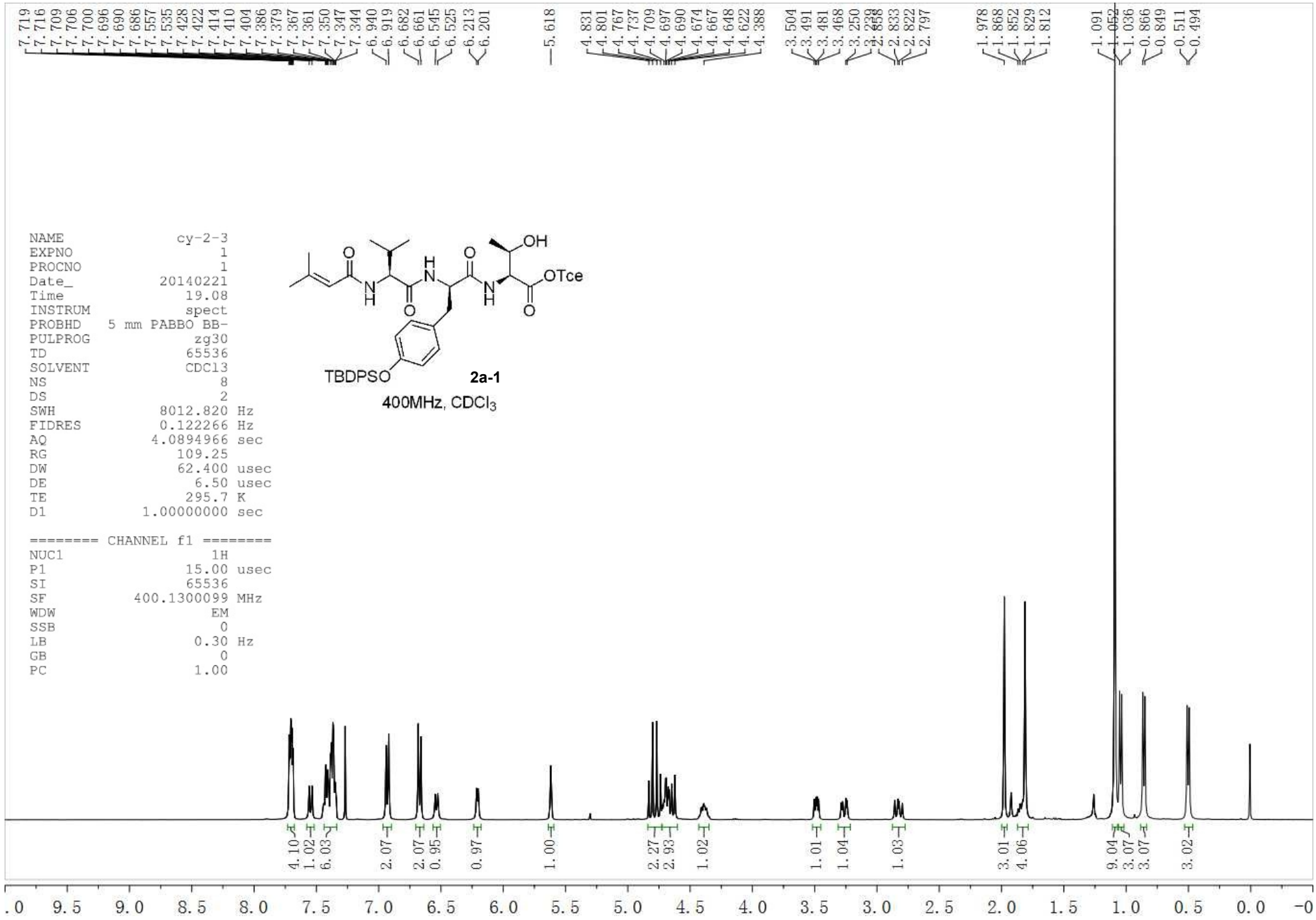

176.632
176.600
174.029
173.618
173.095
171.732
171.629
170.843
165.796
157.145
156.005
134.044
132.636
132.159
132.096
131.125
130.379
130.095
128.591
116.090
115.748
73.884
60.063
56.560
56.346
53.896
52.080
38.200
35.136
31.917
31.368
31.024
28.148
27.110
19.482
18.445
16.369
16.227
13.740
12.722
12.339
NAME qsw-7-30-1st + 2nd-13C
EXPNO 2
PROCNO 1
Date_ 20140215
Time 23.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 22921
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6126515 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Largamide B (1b)
400MHz, CD3OD
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

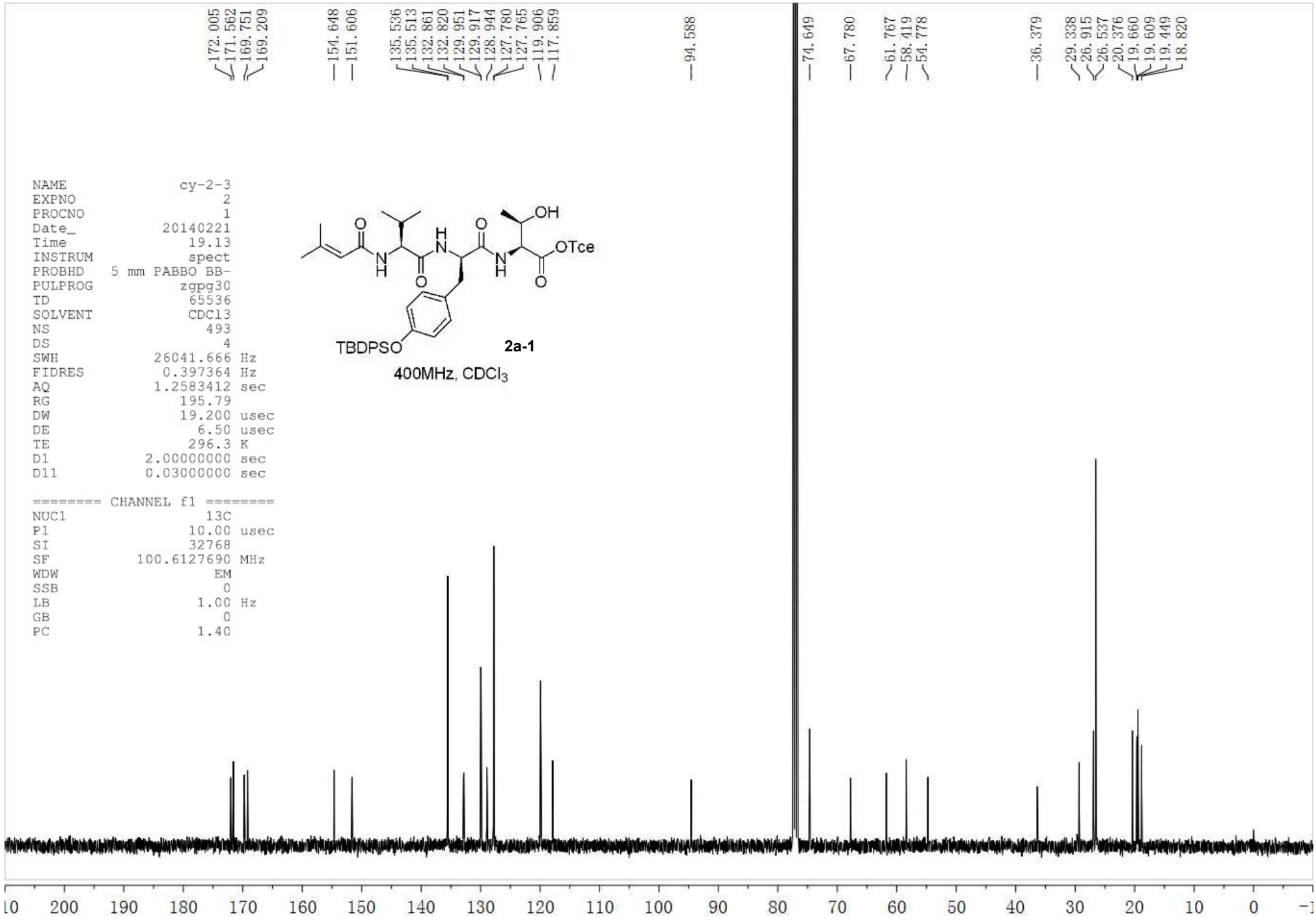

NAME qsw-7-30-DMF-4
EXPNO 1
PROCNO 1
Date_ 20140315
Time 12.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMF
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 228.1
DW 48.400 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.70 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Largamide B (1b)
500MHz, DMF-d7

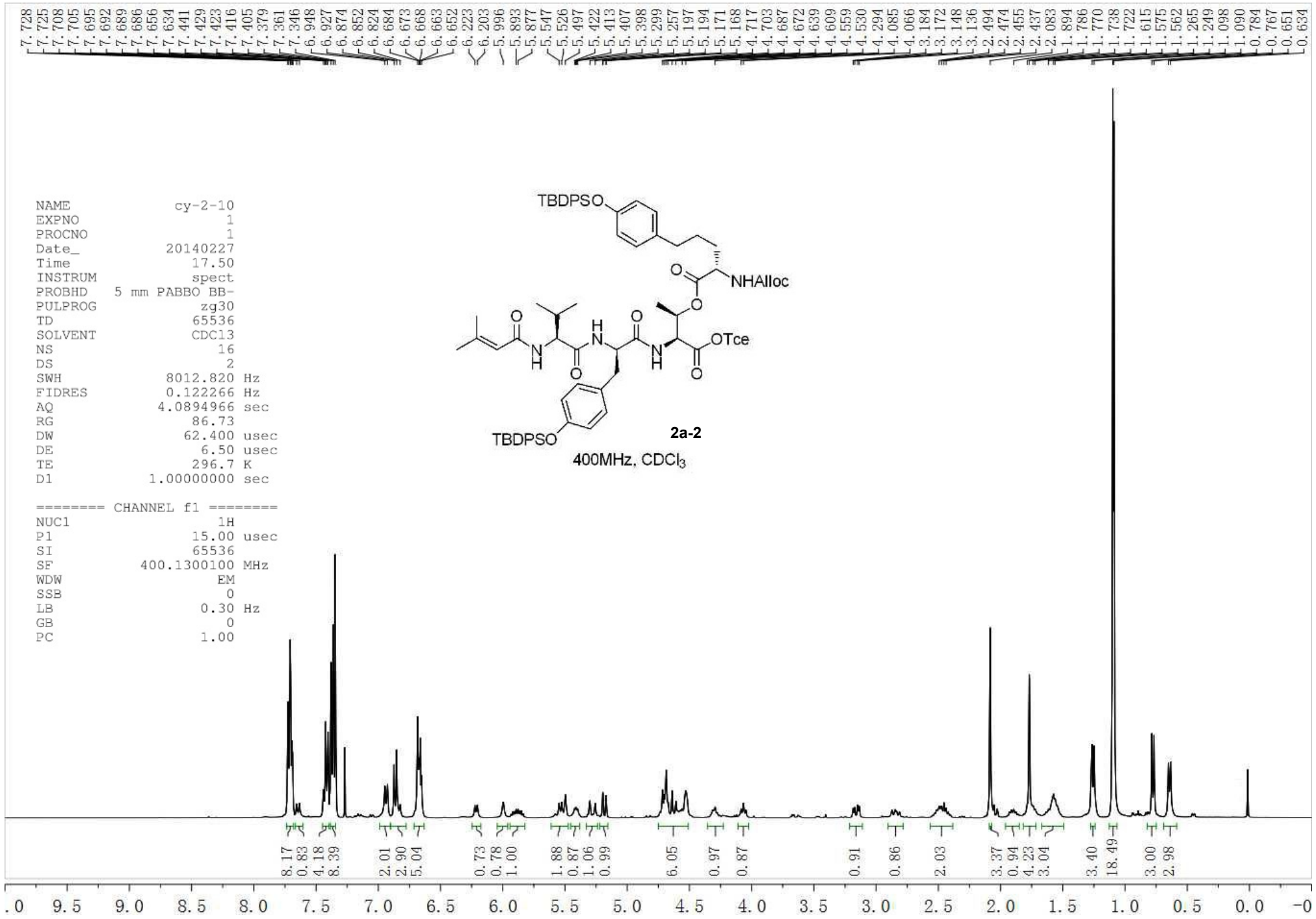

175.839
175.180
172.709
172.099
171.745
171.723
170.353
169.379
164.042
157.196
156.565
133.181
132.832
131.167
131.030
130.440
129.904
129.285
128.667
115.749
115.675
73.463
59.293
55.880
55.591
53.189
51.151
50.647
38.157
34.632
31.258
27.929
27.044
19.812
18.312
16.741
16.266
13.856
12.764
12.587
NAME qsw-7-30-DMF-13C-2
EXPNO 2
PROCNO 1
Date_ 20140316
Time 12.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT DMF
NS 21739
DS 0
SWH 32679.738 Hz
FIDRES 0.997306 Hz
AQ 0.5014157 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 301.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 16.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7714350 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.79 dB
PL12 16.68 dB
PL2W 25.30924988 W
PL12W 0.45318890 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577089 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Largamide B (1b)
500MHz, DMF-d_7

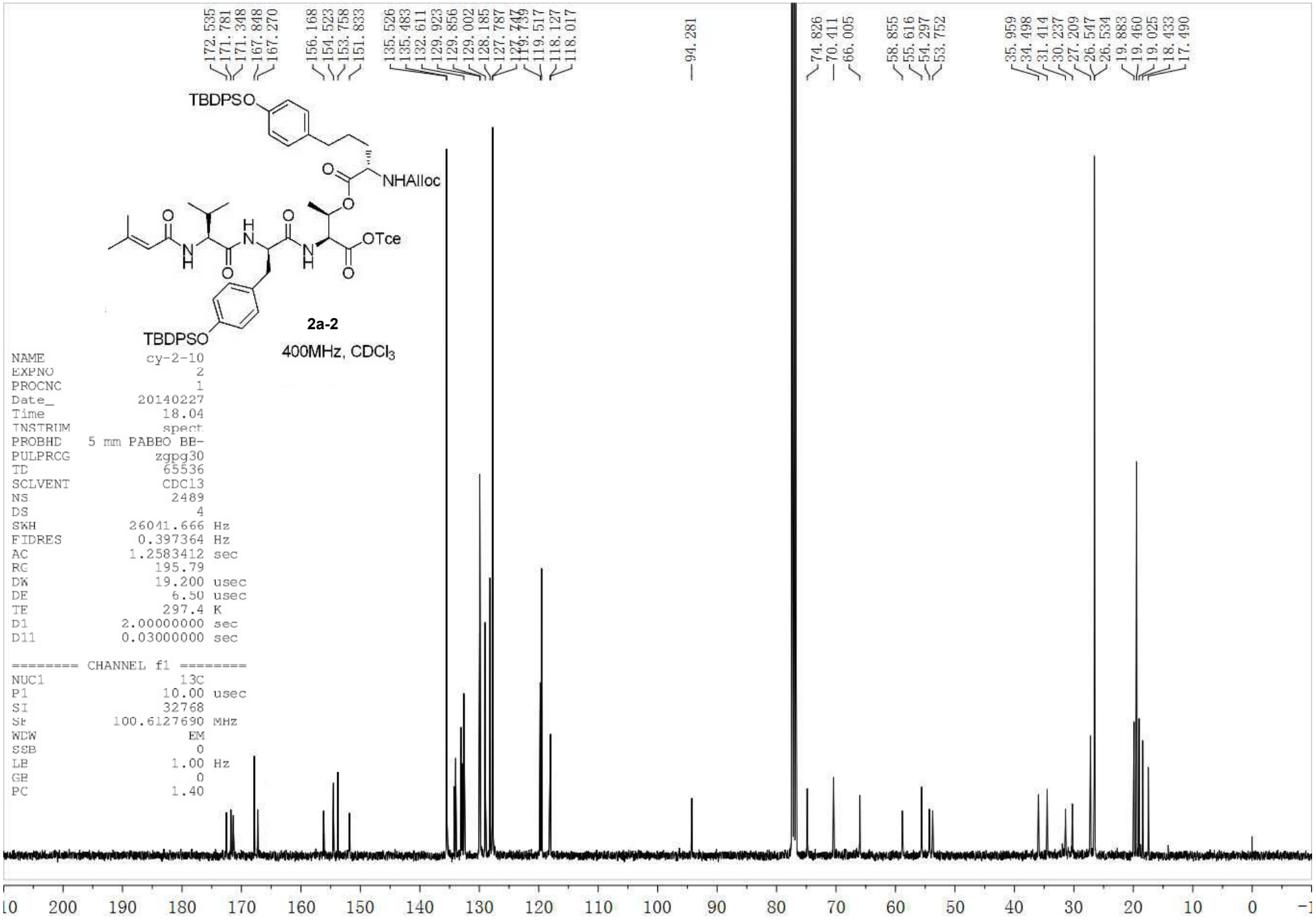

7.718 7.715 7.711 7.707 7.704 7.698 7.695 7.687 7.683 7.429 7.426 7.414 7.411 7.407 7.386 7.382 7.381 7.371 7.366 7.362 7.350 7.345
6.856 6.834 6.677 6.660 6.655 6.547 6.069 5.987
5.560 5.557 5.554
4.795 4.779 4.759 4.744
3.610
3.004 2.989 2.969 2.954 2.937 2.919 2.902
2.127 2.125 2.072 2.056 2.049 2.040 2.024
1.798 1.795
1.095 0.842 0.825 0.806 0.789
NAME cy-2-5shoupu
EXPNO 1
PROCNO 1
Date_ 20140222
Time 19.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 50.6
DW 62.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
TBDPSO
29a
400MHz, CDCl3
4.04 6.12 2.05 2.03 0.95 0.93 1.00 1.00 1.00 3.02 2.02 3.00 1.09 3.02 9.02 6.01
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

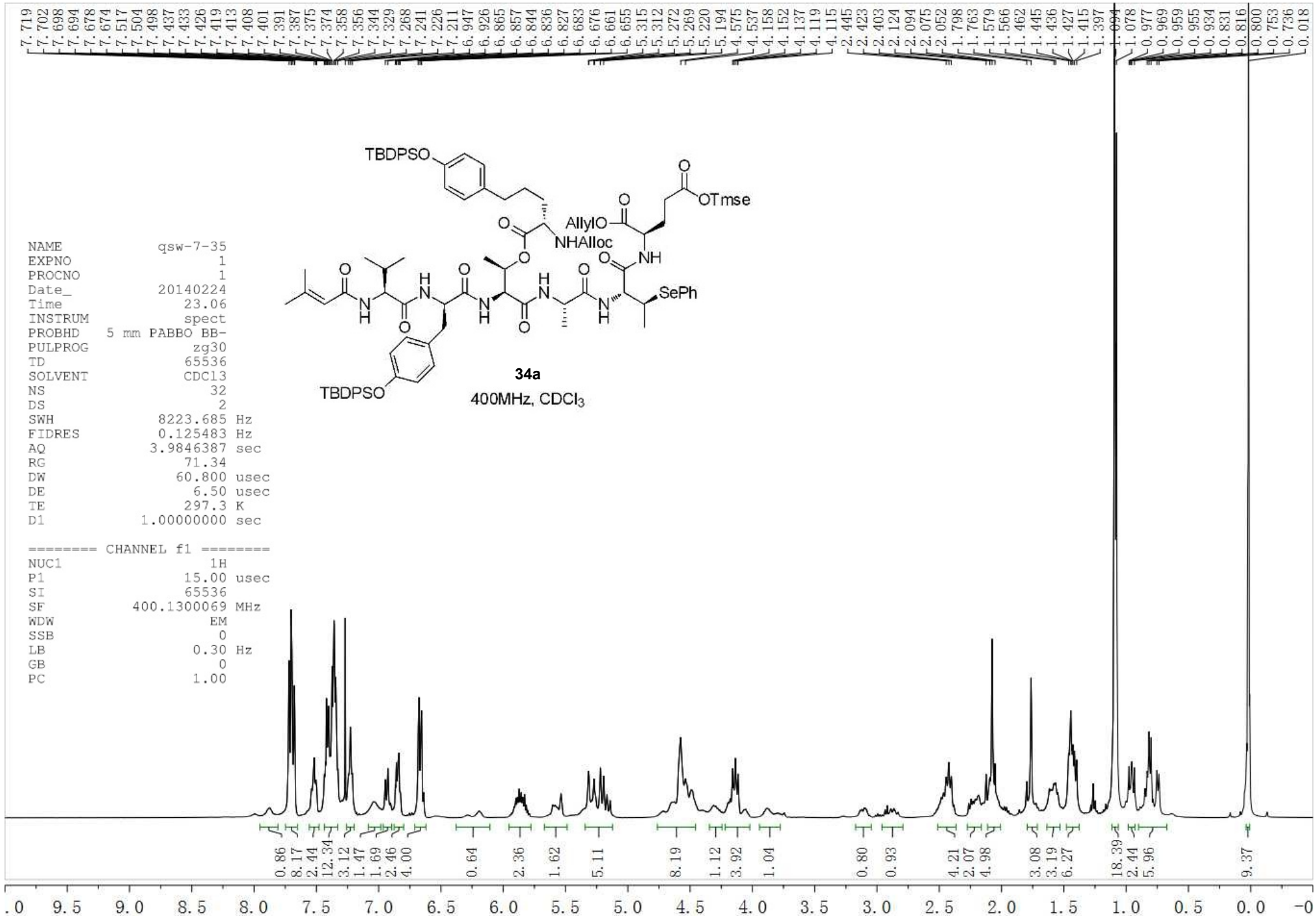

171.795
171.071
166.777
154.718
151.894
135.525
135.498
132.885
132.843
129.960
129.916
128.204
127.778
127.764
119.845
118.168
57.823
53.176
52.222
37.231
30.981
27.155
26.504
19.885
19.455
19.248
17.786
NAME cy-2-5shoupu
EXPNO 2
PROCNO 1
Date_ 20140222
Time 19.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 227
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
TBDPSO
OMe
29a
400MHz, CDCl3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

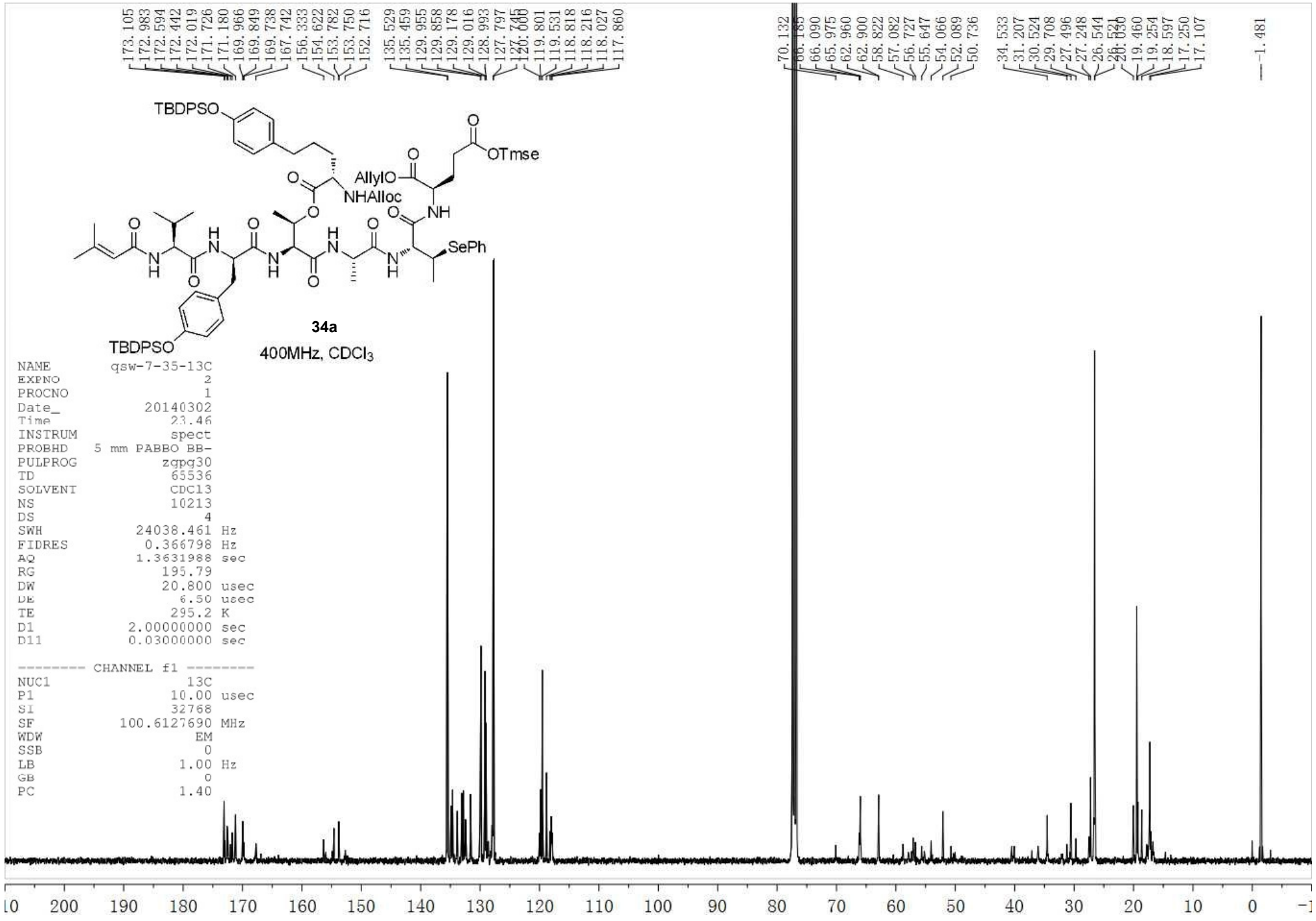

7.719 7.716 7.709 7.706 7.700 7.696 7.690 7.686 7.557 7.535 7.428 7.422 7.414 7.410 7.404 7.386 7.379 7.367 7.361 7.350 7.347 7.344 6.940 6.919 6.682 6.661 6.545 6.525 6.213 6.201 5.618 4.831 4.801 4.767 4.737 4.709 4.697 4.690 4.674 4.667 4.648 4.622 4.388 3.504 3.491 3.481 3.468 3.250 2.833 2.822 2.797 1.978 1.868 1.852 1.829 1.812 1.091 1.052 1.036 0.866 0.849 0.511 0.494
NAME cy-2-3
EXPNO 1
PROCNO 1
Date_ 20140221
Time 19.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 109.25
DW 62.400 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
OTce
TBDPSO
2a-1
400MHz, CDCl3
4.10 1.02 6.03 2.07 2.07 0.95 0.97 1.00 2.27 2.93 1.02 1.01 1.04 1.03 3.01 4.06 9.04 3.07 3.07 3.02
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

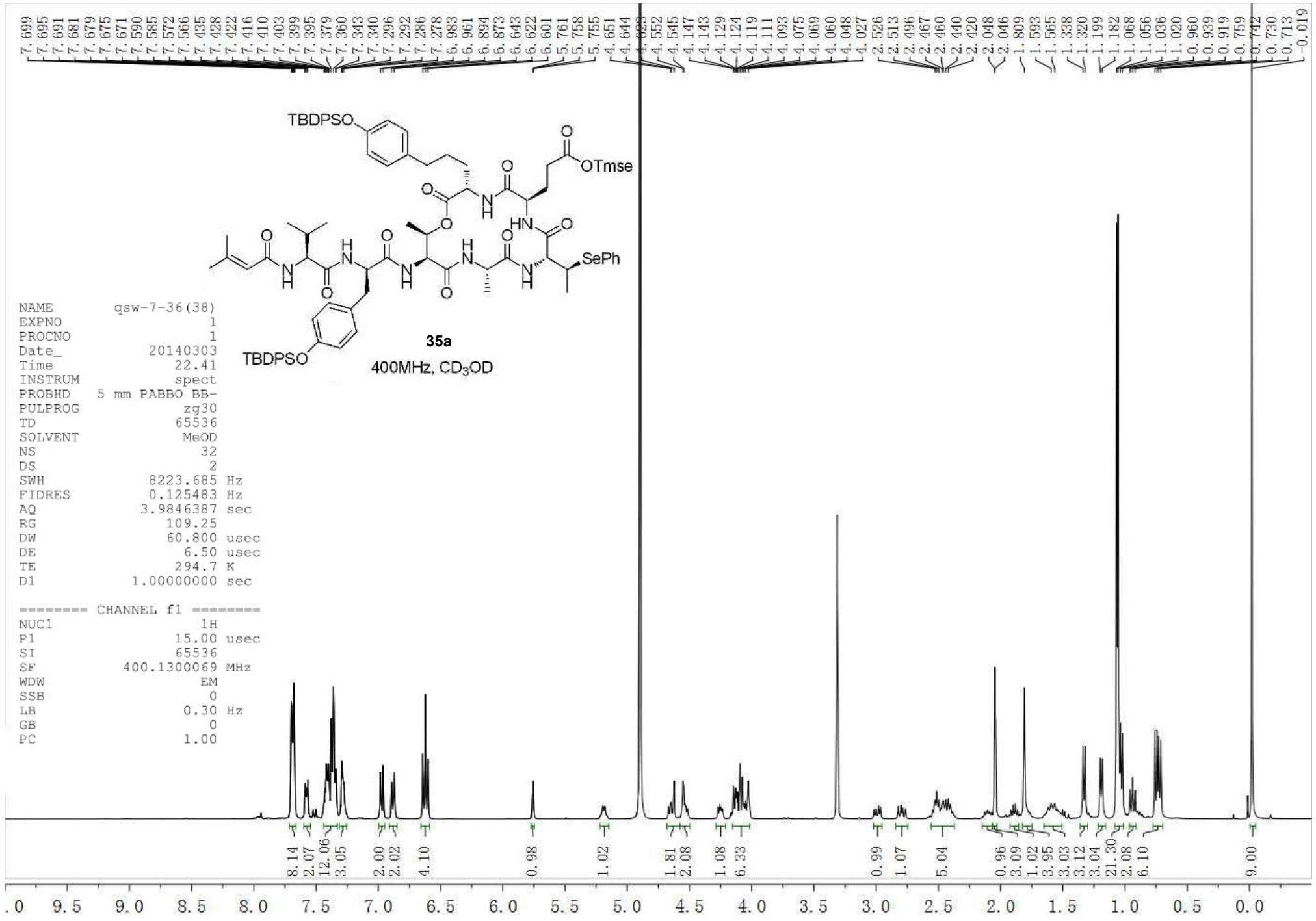

172.005
171.562
169.751
169.209
154.648
151.606
135.536
135.513
132.861
132.820
129.951
129.917
128.944
127.780
127.765
119.906
117.859
94.588
74.649
67.780
61.767
58.419
54.778
36.379
29.338
26.915
26.537
20.376
19.660
19.609
19.449
18.820
NAME cy-2-3
EXPNO 2
PROCNO 1
Date_ 20140221
Time 19.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 493
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
OTce
TBDPSO
2a-1
400MHz, CDCl3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

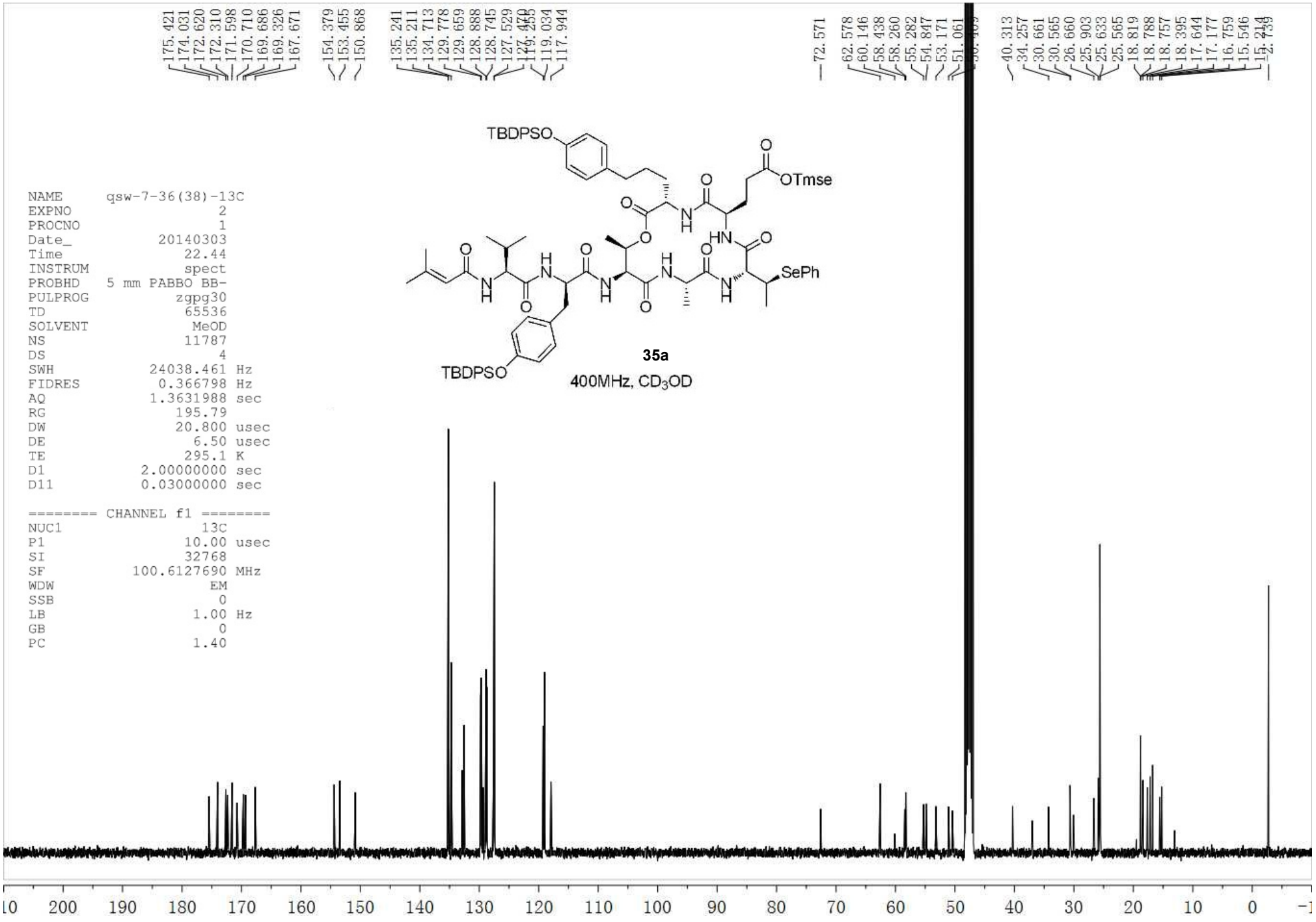

NAME cy-2-10
EXPNO 1
PROCNO 1
Date_ 20140227
Time 17.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 86.73
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBDPSO
NHAlloc
OTce
TBDPSO
2a-2
400MHz, CDCl3

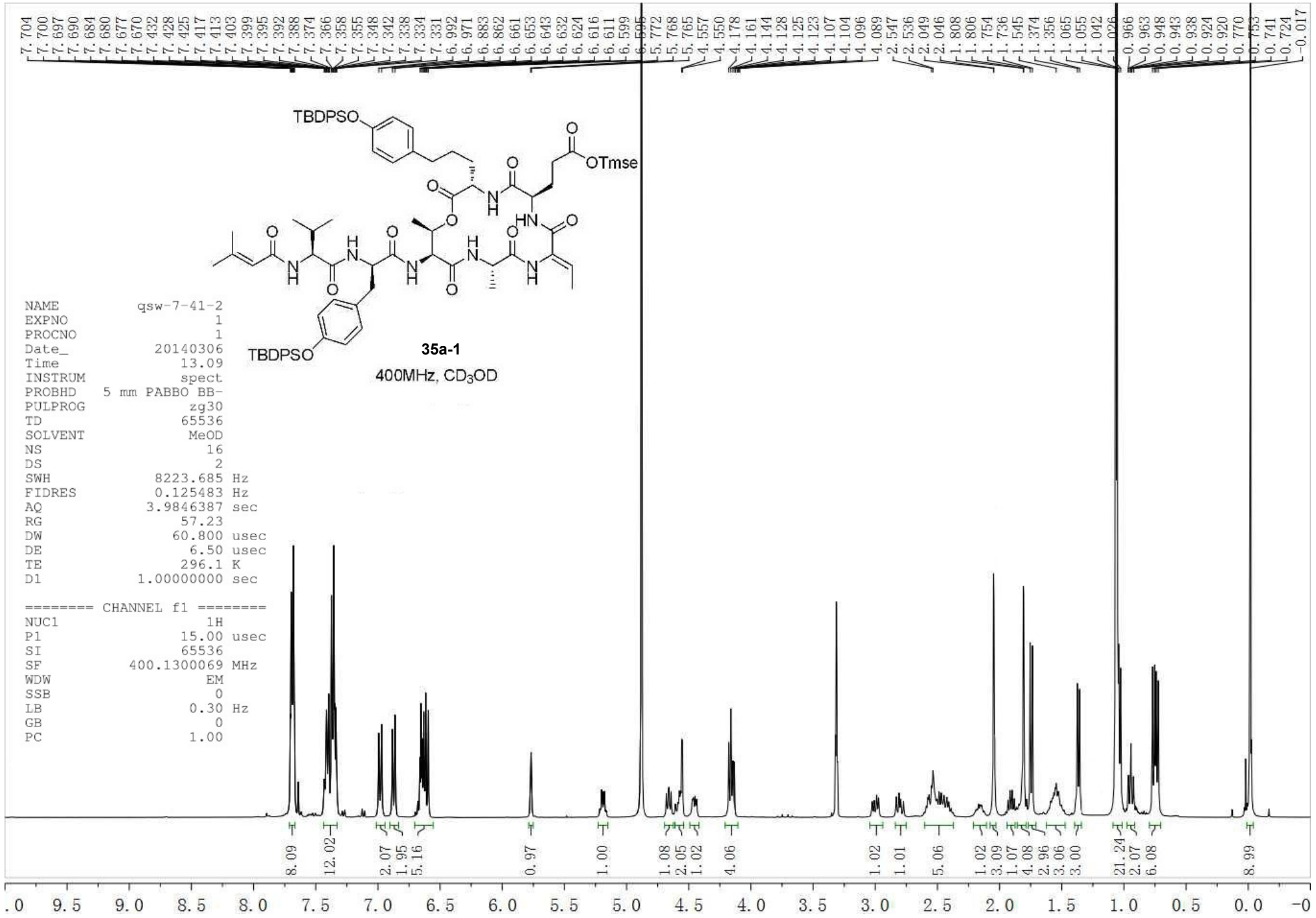

172.535
171.781
171.348
167.848
167.270
156.168
154.523
153.758
151.833
135.526
135.483
132.611
129.923
129.856
129.002
128.185
127.787
119.517
118.127
118.017
94.281
74.826
70.411
66.005
58.855
55.616
54.297
53.752
35.959
34.498
31.414
30.237
27.209
26.547
26.534
19.883
19.460
19.025
18.433
17.490
TBDPSO
NHAlloc
OTce
TBDPSO
2a-2
400MHz, CDCl3
NAME cy-2-10
EXPNO 2
PROCNC 1
Date_ 20140227
Time 18.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPRCG zgpg30
TD 65536
SCLVENT CDCl3
NS 2489
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AC 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

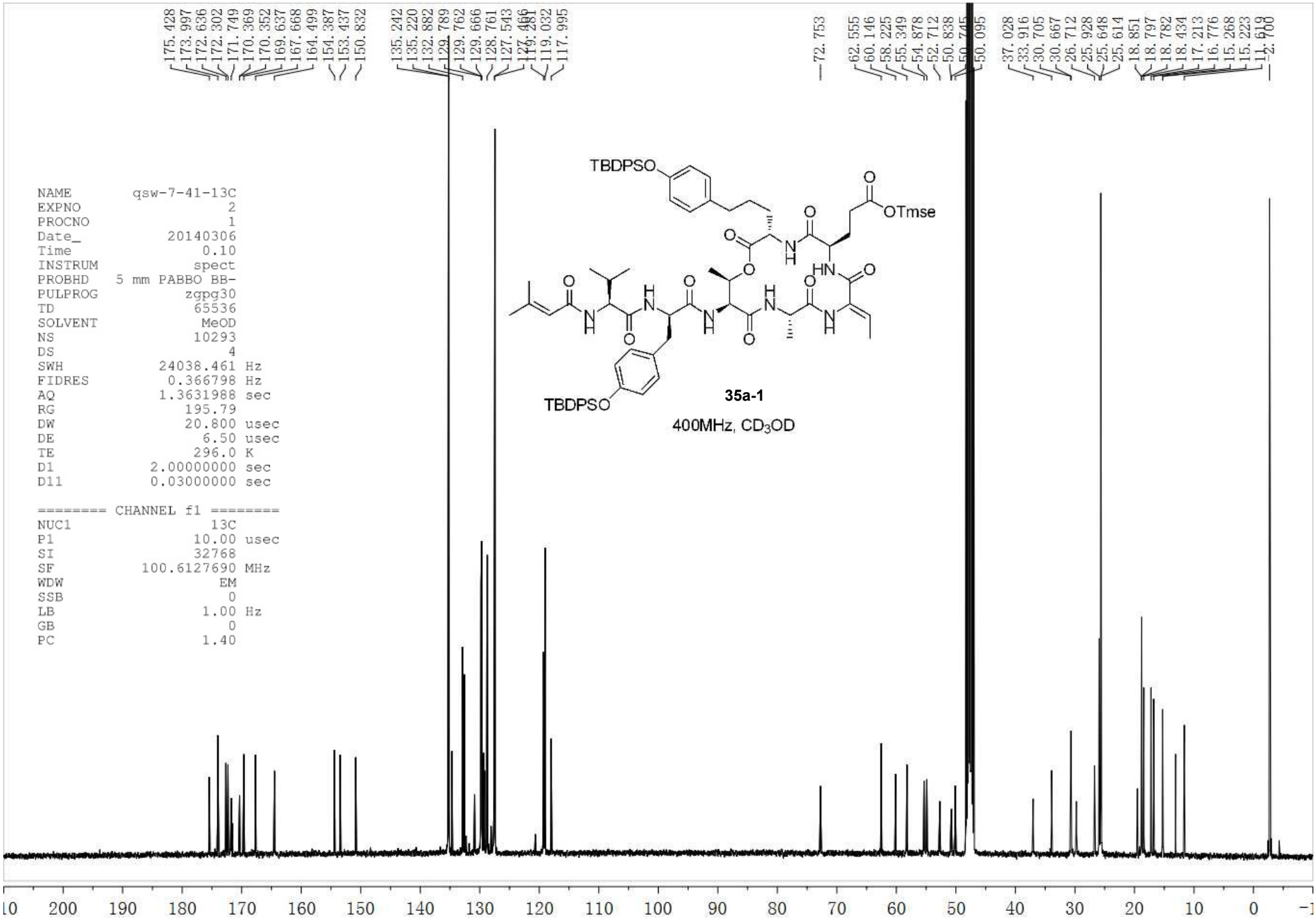

TBDPSO
AllylO
NHAlloc
OTmse
NH
SePh
TBDPSO
34a
400MHz, CDCl3
NAME qsw-7-35
EXPNO 1
PROCNO 1
Date_ 20140224
Time 23.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.34
DW 60.800 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

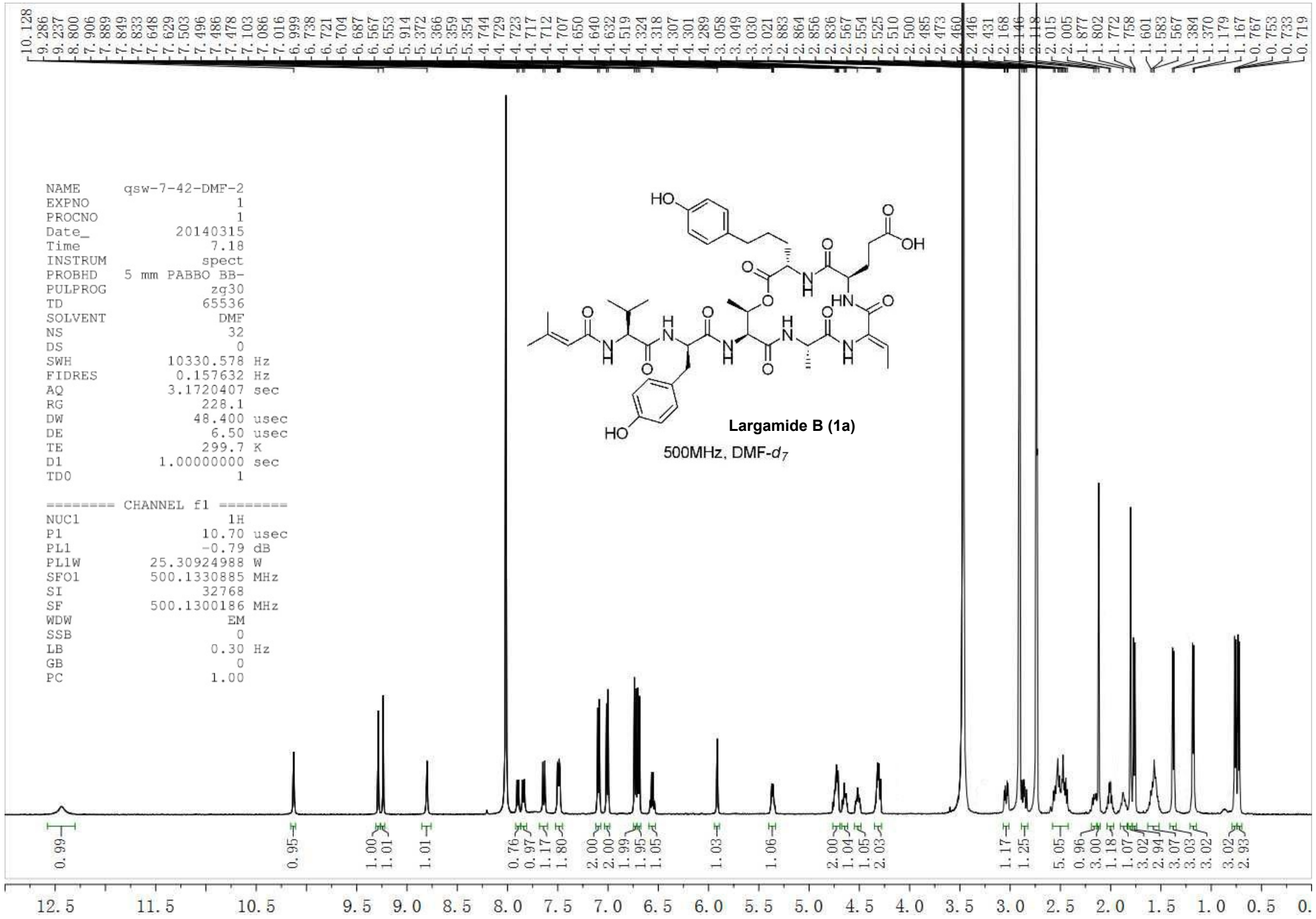

TBDPSO
AllylO
NHAlloc
OTmse
NH
SePh
TBDPSO
34a
400MHz, CDCl3
NAME qsw-7-35-13C
EXPNO 2
PROCNO 1
Date_ 20140302
Time 23.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10213
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
-------- CHANNEL f1 --------
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

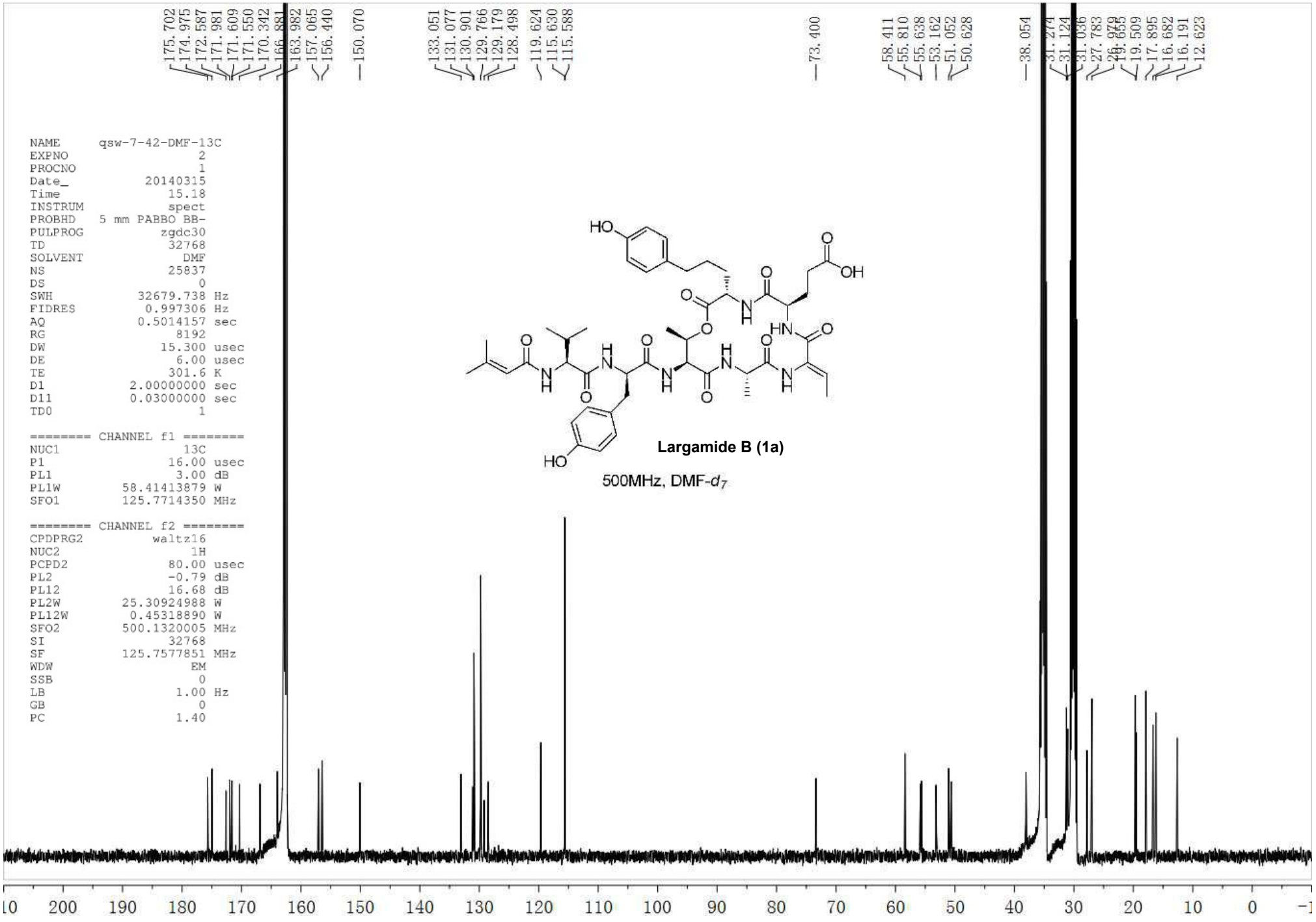

TBDPSO
OTmse
SePh
TBDPSO
35a
400MHz, CD_3OD
NAME qsw-7-36(38)
EXPNO 1
PROCNO 1
Date_ 20140303
Time 22.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 109.25
DW 60.800 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


175.421
174.031
172.620
172.310
171.598
170.710
169.686
169.326
167.671
154.379
153.455
150.868
135.241
135.211
134.713
129.778
129.659
128.888
128.745
127.529
119.034
117.944
72.571
62.578
60.146
58.438
58.260
55.282
54.847
53.171
51.061
40.313
34.257
30.661
30.565
26.660
25.903
25.633
25.565
18.819
18.788
18.757
18.395
17.644
17.177
16.759
15.546
TBDPSO
OTmse
SePh
TBDPSO
NAME qsw-7-36(38)-13C
EXPNO 2
PROCNO 1
Date_ 20140303
Time 22.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 11787
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
35a
400MHz, CD3OD
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


TBDPSO
OTmse
TBDPSO
35a-1
400MHz, CD3OD
NAME qsw-7-41-2
EXPNO 1
PROCNO 1
Date_ 20140306
Time 13.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57.23
DW 60.800 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
SI 65536
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


175.428
173.997
172.636
172.302
171.749
170.369
170.352
169.637
167.668
164.499
154.387
153.437
150.832
135.242
135.220
132.882
129.789
129.762
129.666
128.761
127.543
119.032
117.995
72.753
62.555
60.146
58.225
55.349
54.878
52.712
50.838
50.095
37.028
33.916
30.705
30.667
26.712
25.928
25.648
25.614
18.851
18.797
18.782
18.434
17.213
16.776
15.268
15.223
2.700
NAME qsw-7-41-13C
EXPNO 2
PROCNO 1
Date_ 20140306
Time 0.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 10293
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 195.79
DW 20.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
TBDPSO
OTmse
HN
TBDPSO
35a-1
400MHz, CD3OD
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1


NAME qsw-7-42-DMF-2
EXPNO 1
PROCNO 1
Date_ 20140315
Time 7.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMF
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 228.1
DW 48.400 usec
DE 6.50 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.70 usec
PL1 -0.79 dB
PL1W 25.30924988 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Largamide B (1a)
500MHz, DMF-d7


175.702
174.975
172.587
171.981
171.609
171.550
170.342
166.881
163.982
157.065
156.440
150.070
133.051
131.077
130.901
129.766
129.179
128.498
119.624
115.630
115.588
73.400
58.411
55.810
55.638
53.162
51.052
50.628
38.054
31.274
31.124
31.036
27.783
26.979
26.655
19.509
17.895
16.682
16.191
12.623
NAME qsw-7-42-DMF-13C
EXPNO 2
PROCNO 1
Date_ 20140315
Time 15.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 32768
SOLVENT DMF
NS 25837
DS 0
SWH 32679.738 Hz
FIDRES 0.997306 Hz
AQ 0.5014157 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 301.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 16.00 usec
PL1 3.00 dB
PL1W 58.41413879 W
SFO1 125.7714350 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.79 dB
PL12 16.68 dB
PL2W 25.30924988 W
PL12W 0.45318890 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577851 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
OH
HN
HO
Largamide B (1a)
500MHz, DMF-d_7
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1