

Supporting Information

Total Synthesis and Stereochemistry Establishment of Tumescenamide A

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Tables

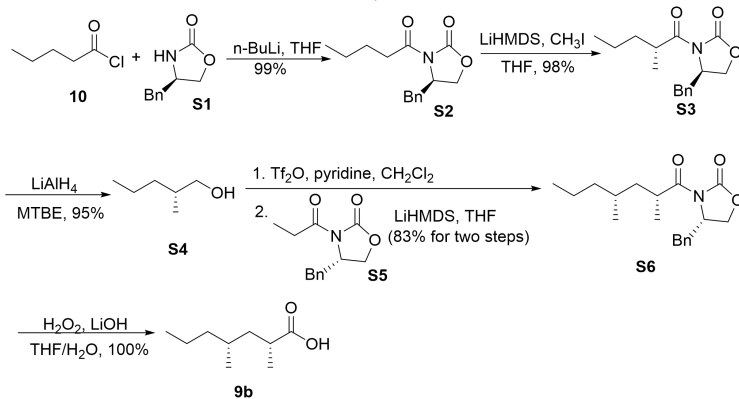
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1. General Information

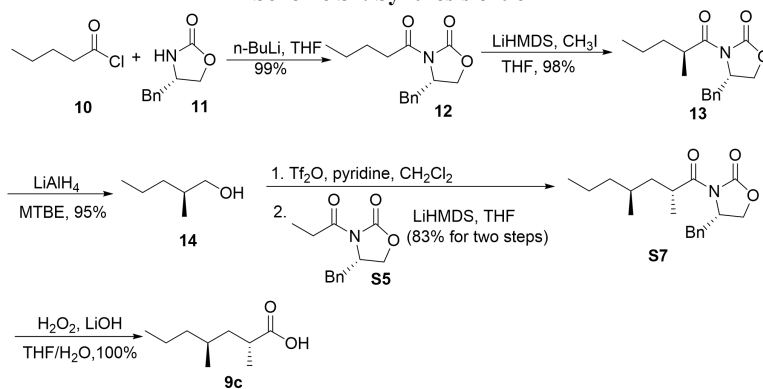
Chemicals were purchased from commercial sources without further purification. The solvents were dried using standard conditions. Glassware was dried in oven and cooled before use. ^1H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectras are recorded on a Bruker AV 500 spectrometer in CDCl_3 and Acetone- d_6 . For ^1H NMR (500 MHz), CDCl_3 ($\delta = 7.26$ ppm) and Acetone- d_6 ($\delta = 2.05$ ppm) served as internal standard. The chemical shift (δ) of each signal is reported in parts per million (ppm) and all coupling constants (J) are reported in Hertz (Hz). The multiplicities of the signals are described using the following abbreviations: s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet, br = broad. For ^{13}C NMR (125 MHz), CDCl_3 ($\delta = 77.16$ ppm) and Acetone- d_6 ($\delta = 29.84$ ppm) served as internal standard. High-resolution Mass spectral (HRMS) datas were measured on a Bruker Apex II. Optical rotation was measured with a Anton Paar MCP 200. Analytical thin layer chromatography (TLC) was performed using silica gel plate (0.2 mm thickness), visualized using UV light and Phosphomolybdic acid staining solutions followed by heating. Column chromatography was carried out on silica gel (200-300 mesh).

2. Synthesis Schemes and Tables

Scheme S1. Synthesis of 9b



Scheme S2. Synthesis of 9c



Scheme S3. Synthesis of 9d

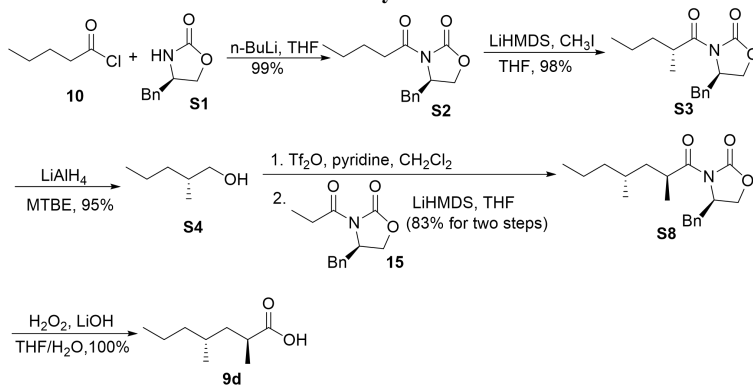
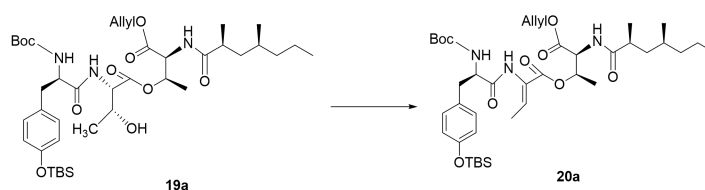
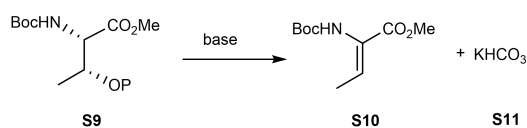


Table S1. β -elimination of 19a¹⁻⁴

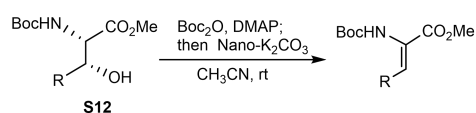
Entry	Conditions	Yield(%) ^[a]
1	(Boc) ₂ O, DMAP; then TMG	decomposition
2	EDC, CuCl ₂	15
3	TsCl, DMAP, DBU	6
4	Et ₃ N, MsCl	11
5	(Boc) ₂ O, DMAP; then Nano K ₂ CO ₃	85

[a] Isolated yields by column chromatography

Table S2. β -Elimination reaction of threonine derivatives ^[a]

Entry	P	Base	T (°C)	Solvent	Yield (%) ^[b]
1	Cbz	Normal K ₂ CO ₃	25	DMF	52
2	Cbz	Normal K ₂ CO ₃	65	DMF	89
3	Cbz	Nano-K ₂ CO ₃	25	DMF	91
4	Boc	Normal K ₂ CO ₃	25	DMF	4
5	Boc	Normal K ₂ CO ₃	65	DMF	81
6	Boc	Nano-K ₂ CO ₃	25	DMF	88
7	Boc	Nano-K ₂ CO ₃	25	THF	73
8	Boc	Nano-K ₂ CO ₃	25	CH ₃ CN	93

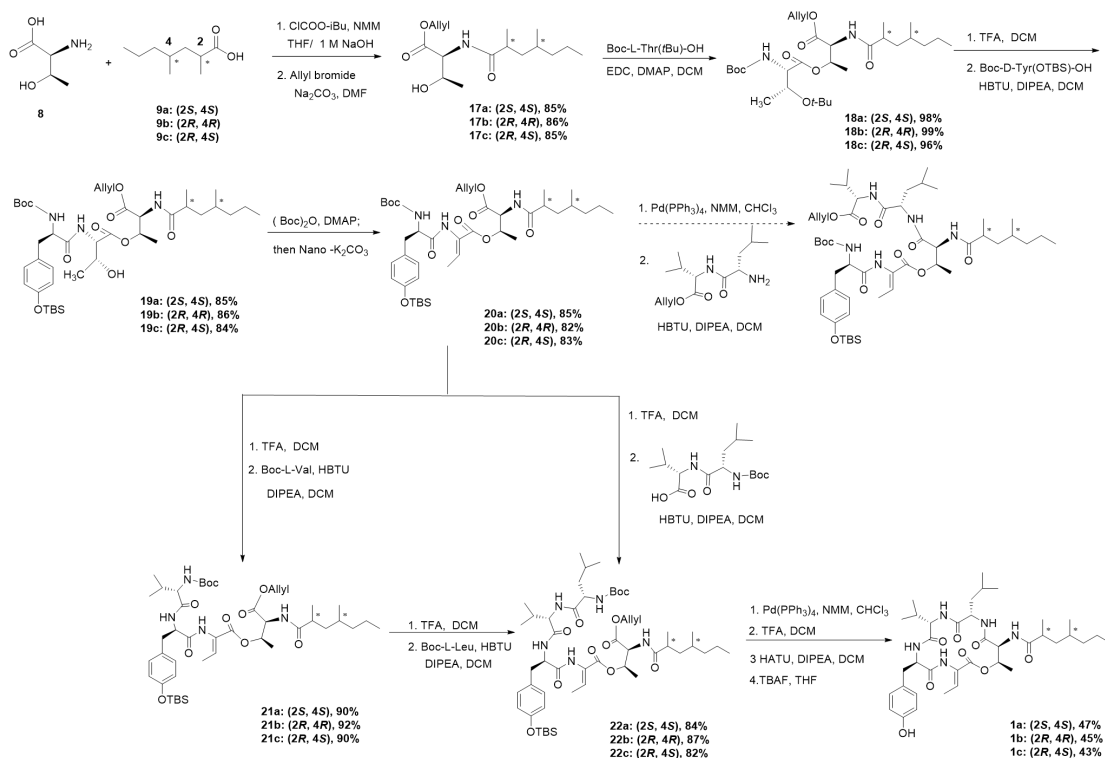
[a] Base (2 equiv), reaction time: 5h. [b] Isolated yields of compounds isolated after column chromatography.

Scheme S4. One-pot synthesis of Boc- Δ Abu-OMe and Boc- Δ Ala-OMe

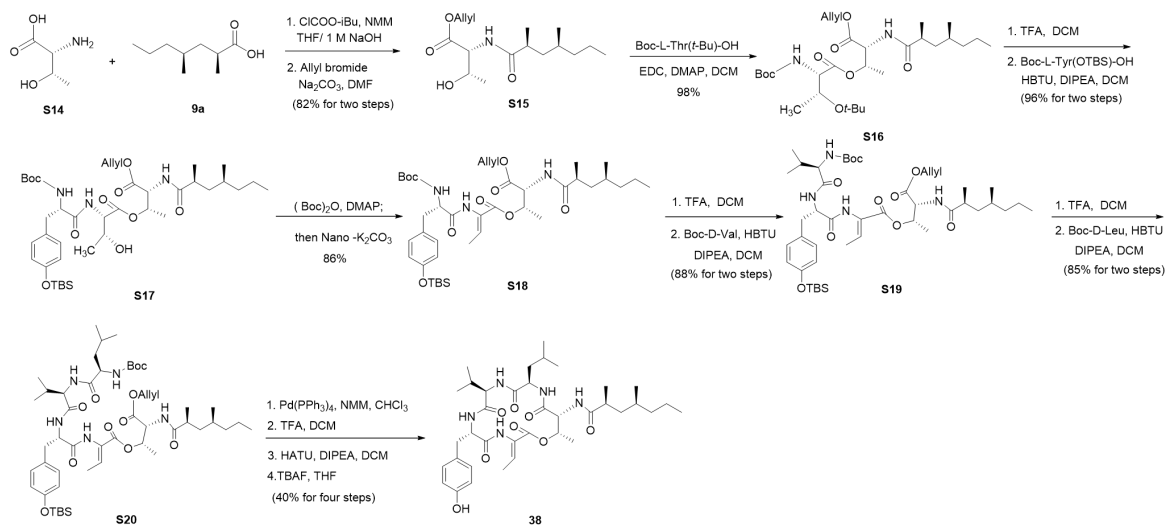
S10 R = CH₃, 84% yield on a 1g scale; 81% yield on a 100 g scale

S13 R = H, 81% yield on a 1g scale; 78% yield on a 100 g scale

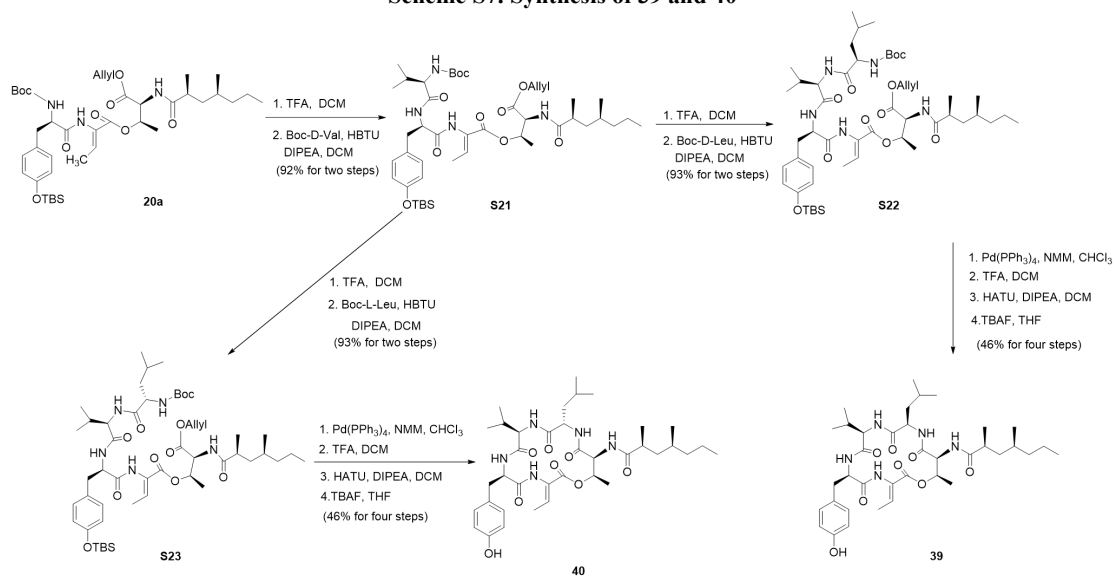
Scheme S5. Synthesis of 1



Scheme S6. Synthesis of 38

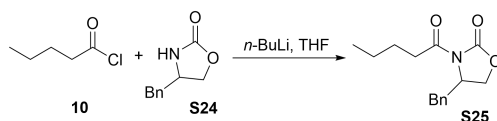


Scheme S7. Synthesis of 39 and 40

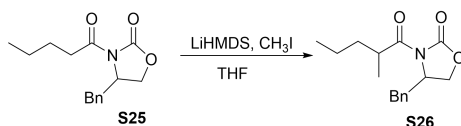


3. Experimental Procedures

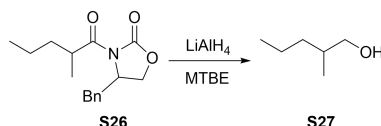
3.1 Preparation of Dmh



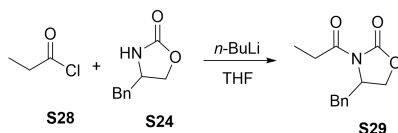
To a cooled (-78 °C) solution of 4-benzyl-2-oxazolidinone **S24** (17.72 g, 100.0 mmol) in THF (500 mL) was added *n*-BuLi (42.0 mL, 2.5 M, 105.0 mmol) dropwise over a 15 mins period. The resulting solution was allowed to stir at -78 °C for 15 mins and then treated with valeryl chloride **10** (12.46 mL, 105.0 mmol). After an additional 15 mins of stirring, the resulting cold (-78 °C) solution was allowed to warm to 25 °C and then quenched with aqueous NH₄Cl. The layers were separated, and the aqueous phase was extracted with EtOAc (3 x 100 mL). The combined organic extracts were dried (MgSO₄), concentrated in vacuo. The crude product was purified via flash chromatographed (SiO₂, 5-10% gradient, EtOAc-hexane) to provide compound **S25**.



To a cooled (-78 °C) suspension of the imide **S25** (13.06 g, 50.0 mmol) in THF (200 mL) was added LiHMDS (60.0 mL, 60.0 mmol, 1 M) dropwise over a 30 mins period. After 1 h of stirring, the resulting cold (-78 °C) solution was treated with methyl iodide (3.70 mL, 60.0 mmol) and allowed to stir at -78 °C for 3 h before being warmed to 25 °C overnight. The reaction was quenched with aqueous NH₄Cl (50 mL), and the aqueous layer was extracted with EtOAc (3 x 50 mL). The combined organic extracts were dried (MgSO₄), concentrated in vacuo. The crude product was purified via flash chromatographed (SiO₂, 5-15% gradient, EtOAc-hexane) to provide compound **S26**.

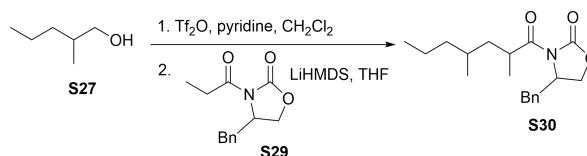


To a cooled (0 °C) suspension of compound **S26** (11.01 g, 40.0 mmol) in MTBE (50 mL) was added LiAlH₄ (2.28 g, 60.0 mmol) in small portions over a 15 mins period. After an additional 30 mins of stirring, the cold (0 °C) reaction was slowly quenched with brine (10 mL). MTBE (50 mL) was added to precipitate the aluminum salts, which were then filtered and dried (MgSO₄), and the solution was concentrated in vacuo. The crude product was purified via flash chromatographed (SiO₂, 15% MTBE-pentane) to provide compound **S27**.



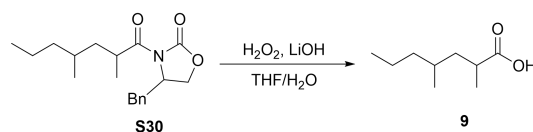
To a cooled (-78 °C) solution of 4-benzyl-2-oxazolidinone **S24** (17.72 g, 100.0 mmol) in THF (500 mL) was added *n*-BuLi (42.0 mL, 2.5 M, 105.0 mmol) dropwise over a 15 mins period. The resulting solution was allowed to stir at -78 °C for 15 mins and then treated with propionyl chloride **S28** (9.81 mL, 105.0 mmol). After an additional 15 mins of stirring, the resulting cold (-78 °C) solution was allowed to warm to

25 °C and then quenched with aqueous NH₄Cl. The layers were separated, and the aqueous phase was extracted with EtOAc (3 x 100 mL). The combined organic extracts were dried (MgSO₄), concentrated in vacuo. The crude product was purified via flash chromatography (SiO₂, 5-10% gradient, EtOAc-hexane) to provide compound **S29**.



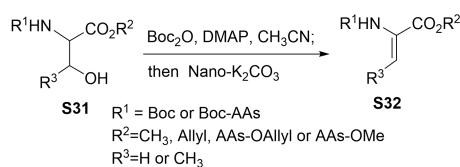
A stirred solution of compound **S27** (3.68 g, 36.0 mmol) in CH₂Cl₂ (50 mL) under nitrogen atmosphere was cooled to -78 °C, to which pyridine (2.90 mL, 36.0 mmol) and Tf₂O (6.06 mL, 36.0 mmol) were added. After being stirred for 50 mins at -78 °C, the reaction was quenched with aqueous NH₄Cl (10 mL). The aqueous layer was extracted with CH₂Cl₂ (20 mL), and the combined organic layers were dried (MgSO₄) and concentrated in vacuo. The residue was used for the next step quickly.

A stirred solution of compound **S29** (6.99 g, 30.0 mmol) in THF (100 mL) under nitrogen atmosphere was cooled to -78 °C, to which LiHMDS (36.0 mL, 36.0 mmol, 1 M) was added. After being stirred for 30 mins at -78 °C, the obtained material in THF (5 mL) was added. The reaction mixture was warmed to 0 °C and stirred for 3 h, and then quenched with aqueous NH₄Cl (15 mL). The aqueous layer was extracted with EtOAc (3 x 50 mL), and the combined organic layers were dried (MgSO₄) and concentrated in vacuo. The crude product was purified via flash chromatography (SiO₂, 10-15% gradient, EtOAc-hexane) to give compound **S30**.



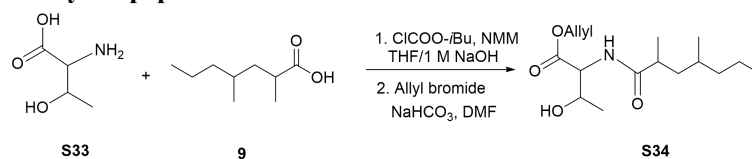
Compound **S30** (6.34 g, 20.0 mmol) was dissolved in 1:2 THF/water (200 mL) and cooled to 0 °C. To the resulting solution was added 30% aqueous H₂O₂ (4.90 mL, 160.0 mmol) followed by LiOH (1.92 g, 80.0 mmol). The resulting mixture was stirred at 0 °C for 2 h. The reaction was slowly warmed to ambient temperature. THF was removed on a rotary evaporator. The aqueous solution was washed with EtOAc (20 mL). The solution was acidified with 2 M HCl to pH = 2 and extracted with EtOAc (3 x 50 mL). The extracts were combined, dried (MgSO₄). The crude product was purified via flash chromatography (SiO₂, 15-20% EtOAc-hexane) to give compound **9**.

3.2 Preparation of dehydropeptides



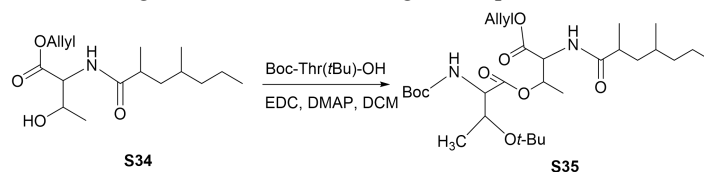
Compound **S31** (5.0 mmol) was dissolved in acetonitrile (5 mL), then DMAP (0.06 g, 0.5 mmol) and (Boc)₂O (1.26 mL, 5.5 mmol) were added, the reaction mixture was stirred at room temperature for 2 h. Then nano-K₂CO₃ (1.38 g, 10.0 mmol) was added, the reaction was stirred at room temperature for 5 h. The nano-K₂CO₃ was recycled by filtration. The solvent was removed in vacuo and water was added. The aqueous solution was extracted with EtOAc (3 x 30 mL). The organics were combined and washed with 1 M KHSO₄ (2 x 20 mL), 1 M NaHCO₃ (2 x 20 mL), dried (MgSO₄), concentrated in vacuo to give crude product. Pure product **S32** was obtained by recrystallization using EtOAc and n-hexane.

3.3 Preparation of cyclic peptides

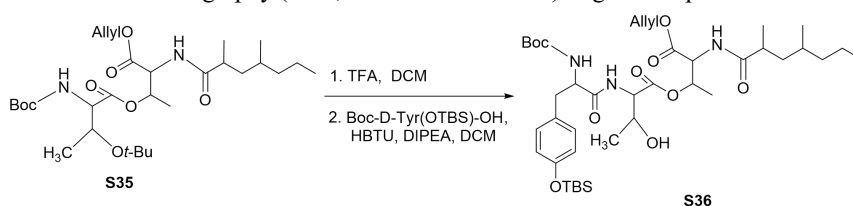


A stirred solution of compound **9** (1.58 g, 10.0 mmol) and 4-methylmorpholine (1.12 mL, 10.0 mmol) in THF (50 mL) was cooled to -10 °C. Isobutyl chloroformate (1.26 mL, 10.0 mmol) was added and the reaction was allowed to stir for 30 minutes. The reaction was allowed to warm to 0 °C at which time a solution of compound **S33** (1.43 g, 12.0 mmol) in 1 M NaOH (12 mL) was added. The reaction was allowed to warm to room temperature and stir for 24 hours at which time it was diluted with H₂O (30 mL). The layers were separated and the aqueous layer was washed with EtOAc (2 x 50 mL). The combined organic layers were then extracted with saturated NaHCO₃ solution (3 x 50 mL). All aqueous layers were combined and acidified to pH = 2 with 1 M HCl and extracted with EtOAc (3 x 100 mL). The combined extractions were then dried (MgSO₄) and concentrated to give the crude product as a colorless oil which was used without further purification for the next step.

Allyl bromide (1.04 mL, 12.0 mmol) was added to a stirred solution of the crude product and NaHCO₃ (1.68 g, 20.0 mmol) in DMF (30 mL). The reaction was stirred for 24 hours at which time it was diluted with H₂O (50 mL). The aqueous layer was extracted with EtOAc (3 x 50 mL). The combined organic layers were then washed with H₂O (2 x 20 mL), dried (MgSO₄), and concentrated in vacuo. The crude product was purified by recrystallization using EtOAc and n-hexane to give compound **S34**.



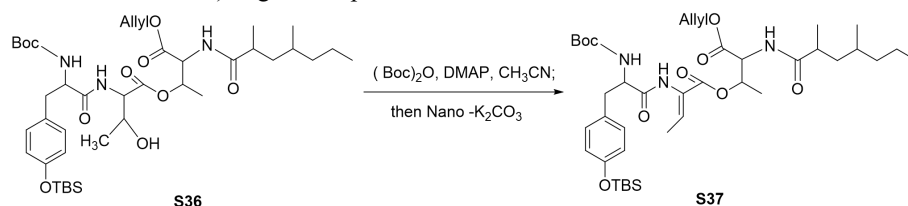
EDC (1.84 g, 9.6 mmol) and 4-dimethylaminopyridine (1.17 g, 9.6 mmol) were dissolved in CH₂Cl₂ (50 mL). The solution was then added to a mixture of compound **S34** (2.39 g, 8.0 mmol) and Boc-Thr(*t*Bu)-OH (2.64 g, 9.6 mmol) in a reaction flask cooled to 0 °C. This reaction mixture was allowed to warm to room temperature and stir for 24 hours at which time it was diluted with CH₂Cl₂ (50 mL) and washed with a saturated NaHCO₃ solution (3 x 10 mL). The combined aqueous layers were back extracted with CH₂Cl₂ (2 x 10 mL). All organic layers were combined, dried (MgSO₄), and concentrated in vacuo. The crude product was purified via flash chromatography (SiO₂, 15% EtOAc-hexane) to give compound **S35**.



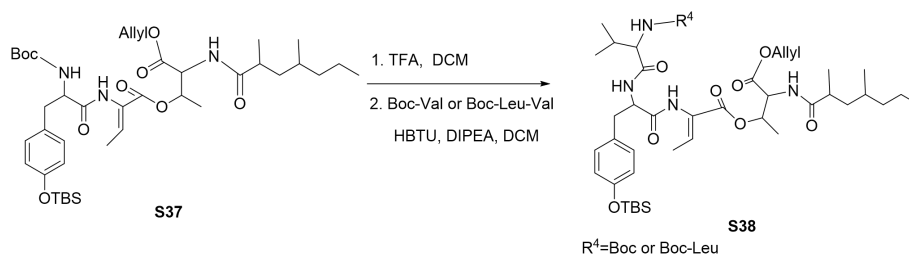
TFA (7.0 mL) was added to a stirred solution of compound **S35** (3.89 g, 7.0 mmol) in CH₂Cl₂ (70 mL). Reaction was stirred at room temperature for 30 mins after which the solvent was removed under reduced pressure. The crude material was dissolved in CH₂Cl₂ and concentrated three times to ensure all TFA was removed. The deprotected amine was used in the next reaction without further purification.

To a stirred solution of Boc-D-Tyr(OTBS)-OH (3.32 g, 8.4 mmol) in CH₂Cl₂ (50 mL) were added HBTU (3.19 g, 8.4 mmol), the deprotected amine, and DIPEA (2.68 mL, 15.4 mmol) at room temperature. After being stirred at room temperature for 12 h, the reaction mixture was concentrated in vacuo and diluted with EtOAc (100 mL). The organic layer was washed with 1 M HCl (2 x 20 mL), saturated aqueous NaHCO₃ (2 x

20 mL), dried (MgSO_4), and concentrated in vacuo. The residue was purified via flash chromatography (SiO_2 , 20-30% EtOAc-hexane) to give compound **S36**.

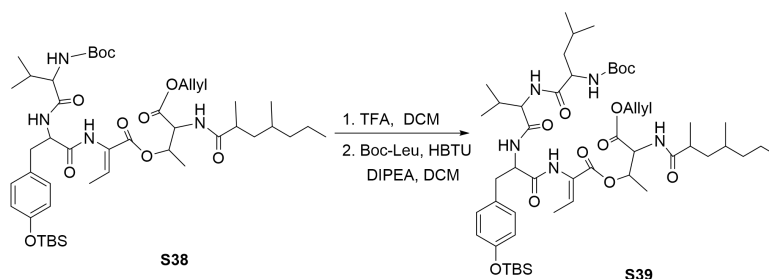


Compound **S36** (4.66 g, 6.0 mmol) was dissolved in acetonitrile (6 ml), then DMAP (0.07 g, 0.6 mmol) and $(\text{Boc})_2\text{O}$ (1.52 mL, 6.6 mmol) were added, the reaction mixture was stirred at room temperature for 2 h. Then nano- K_2CO_3 (1.66 g, 12.0 mmol) was added, the reaction was stirred at room temperature for 5 h. The nano- K_2CO_3 was recycled by filtration. The solvent was removed in vacuo and water was added. The aqueous solution was extracted with EtOAc (3 x 30 mL). The organics were combined and washed with 1 M KHSO_4 (2 x 20 mL), 1 M NaHCO_3 (2 x 20 mL), dried (MgSO_4), concentrated in vacuo to give crude product. Pure product **S37** was obtained by recrystallization using EtOAc and n-hexane.



TFA (5 mL) was added to a stirred solution of compound **S37** (3.80 g, 5.0 mmol) in CH_2Cl_2 (50 mL). Reaction was stirred at room temperature for 30 mins after which the solvent was removed under reduced pressure. The crude material was dissolved in CH_2Cl_2 and concentrated three times to ensure all TFA was removed. The deprotected amine was used in the next reaction without further purification.

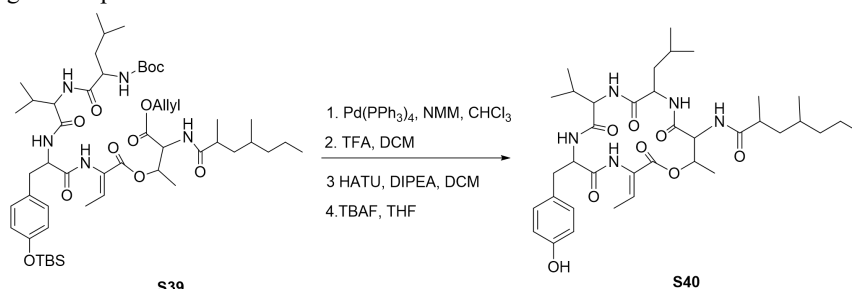
To a stirred solution of Boc-Val or Boc-Leu-Val (6 mmol) in CH_2Cl_2 (30 mL) were added HBTU (2.28 g, 6 mmol), the deprotected amine, and DIPEA (1.92 mL, 11.0 mmol) at room temperature. After being stirred at room temperature for 12 h, the reaction mixture was concentrated in vacuo and diluted with EtOAc (100 mL). The organic layer was washed with 1 M HCl (2 x 20 mL), saturated aqueous NaHCO_3 (2 x 20 mL), dried (MgSO_4), and concentrated in vacuo. The residue was purified by recrystallization using EtOAc and n-hexane to give compound **S38**.



TFA (3 mL) was added to a stirred solution of compound **S38** (3.43 g, 4.0 mmol) in CH_2Cl_2 (30 mL). Reaction was stirred at room temperature for 30 mins after which the solvent was removed under reduced pressure. The crude material was dissolved in CH_2Cl_2 and concentrated three times to ensure all TFA was removed. The deprotected amine was used in the next reaction without further purification.

To a stirred solution of Boc-Leu (1.11 g, 4.8 mmol) in CH_2Cl_2 (30 mL) were added HBTU (1.82 g, 4.8 mmol), the deprotected amine, and DIPEA (1.53 mL, 8.8 mmol) at room temperature. After being stirred at

room temperature for 12 h, the reaction mixture was concentrated in vacuo and diluted with EtOAc (100 mL). The organic layer was washed with 1 M HCl (2 x 20 mL), saturated aqueous NaHCO₃ (2 x 20 mL), dried (MgSO₄), and concentrated in vacuo. The residue was purified by recrystallization using EtOAc and n-hexane to give compound **S39**.



To a stirred solution of compound **S39** (485.8 mg, 0.5 mmol) in CHCl₃ (10 mL) were added 4-Methylmorpholine (331.6 μL, 3.0 mmol) and Pd(PPh₃)₄ (231.1 mg, 0.2 mmol) at room temperature. After being stirred at the same temperature for 3 h, the reaction mixture was concentrated in vacuo, and the deprotected product was used for the next reaction without further purification.

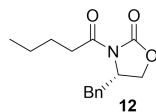
To a stirred solution of the above crude product in CH₂Cl₂ (5 mL) were added TFA (0.5 mL). Reaction was stirred at room temperature for 30 mins after which the solvent was removed under reduced pressure. The crude material was dissolved in CH₂Cl₂ and concentrated three times to ensure all TFA was removed. The deprotected product was used in the next reaction without further purification.

To a stirred solution of the deprotected product in CH₂Cl₂ (5 mL) were added HATU (228.1 mg, 0.6 mmol) and DIPEA (191.6 μL, 1.1 mmol) at room temperature. After being stirred at the same temperature for 12 h, the reaction mixture was concentrated in vacuo and diluted with EtOAc (10 mL). The organic layer was washed with 1 M HCl (2 x 2 mL), saturated aqueous NaHCO₃ (2 x 2 mL), dried (MgSO₄), and concentrated in vacuo. The crude product was used without further purification for the next step.

To a stirred solution of the crude product in THF (3 mL) was added 1 M TBAF (0.6 mL, 0.6 mmol) at 0 °C. After being stirred at 0 °C to room temperature for 2 h, the reaction mixture was diluted with EtOAc (10 mL). The organic layer was washed with water, dried (MgSO₄), and concentrated in vacuo. The residue was purified via flash chromatography (SiO₂, 1-2% MeOH-CH₂Cl₂) to give compound **S40**

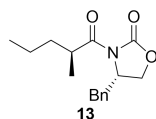
4. Data of Products

4.1 Data of Dmh



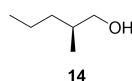
(S)-4-benzyl-3-pentanoyloxazolidin-2-one (12):

Colorless oil, 25.85 g, 99.0% yield. R_f = 0.5 (petroleum/EtOAc = 5:1); ^1H NMR (500 MHz, CDCl_3) δ 7.39 (2H, t, J = 7.0 Hz), 7.35-7.32 (1H, m), 7.27 (2H, d, J = 7.0 Hz), 4.75-4.71 (1H, m), 4.27-4.21 (2H, m), 3.34 (1H, dd, J = 3.0, 13.0 Hz), 3.07-2.93 (2H, m), 2.83 (1H, dd, J = 9.5, 13.0 Hz), 1.79-1.70 (2H, m), 1.51-1.44 (2H, m), 1.02 (3H, t, J = 7.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 173.55, 153.58, 135.48, 129.54, 129.06, 127.45, 66.27, 55.27, 38.07, 35.37, 26.49, 22.38, 13.96; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ found for 284.1264, calcd for $\text{C}_{15}\text{H}_{19}\text{NO}_3\text{Na}$ 284.1263.



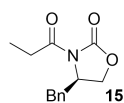
(S)-4-benzyl-3-((S)-2-methylpentanoyl)oxazolidin-2-one (13):

Yellow oil, 13.48 g, 98.0% yield; R_f = 0.6 (petroleum/EtOAc = 5:1); ^1H NMR (500 MHz, CDCl_3) δ 7.33 (2H, t, J = 7.0 Hz), 7.29-7.26 (1H, m), 7.21 (2H, d, J = 7.5 Hz), 4.69-4.66 (1H, m), 4.21-4.16 (2H, m), 3.76-3.70 (1H, m), 3.27 (1H, dd, J = 2.5, 13.0 Hz), 2.77 (1H, dd, J = 9.5, 13.0 Hz), 1.76-1.69 (1H, m), 1.44-1.30 (3H, m), 1.22 (3H, d, J = 6.5 Hz), 0.91 (3H, t, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 177.51, 153.21, 135.51, 129.58, 129.06, 127.46, 66.15, 55.51, 38.07, 37.59, 35.72, 20.54, 17.43, 14.19; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ found for 298.1420, calcd for $\text{C}_{16}\text{H}_{21}\text{NO}_3\text{Na}$ 298.1419.



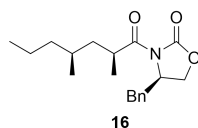
(S)-2-methylpentan-1-ol (14):

Colorless liquid, 3.88 g, 95.0% yield; R_f = 0.6 (petroleum/EtOAc = 3:1); ^1H NMR (500 MHz, CDCl_3) δ 3.52-3.49 (1H, m), 3.43-3.39 (1H, m), 1.69-1.59 (2H, m), 1.42-1.33 (3H, m), 1.32-1.25 (2H, m), 1.12-1.06 (1H, m), 0.91-0.88 (6H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 68.50, 35.61, 35.54, 20.19, 16.65, 14.44; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 103.1120, calcd for $\text{C}_6\text{H}_{15}\text{O}$ 103.1123.



(R)-4-benzyl-3-propionylloxazolidin-2-one (15):

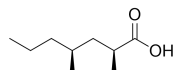
Colorless oil, 23.08 g, 99.0% yield. R_f = 0.6 (petroleum/EtOAc = 5:1); ^1H NMR (500 MHz, CDCl_3) δ 7.33 (2H, t, J = 7.5 Hz), 7.29-7.26 (1H, m), 7.21 (2H, d, J = 7.5 Hz), 4.70-4.65 (1H, m), 4.22-4.15 (2H, m), 3.31 (1H, dd, J = 3.0, 13.5 Hz), 3.03-2.89 (2H, m), 2.78 (1H, dd, J = 9.5, 13.0 Hz), 1.21 (3H, t, J = 7.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 174.21, 153.63, 135.46, 129.53, 129.07, 127.45, 66.34, 55.28, 38.05, 29.30, 8.42.



(R)-4-benzyl-3-((2S, 4S)-2, 4-dimethylheptanoyl)oxazolidin-2-one (16):

Colorless oil, 7.90 g, 83% yield; R_f = 0.5 (petroleum/EtOAc = 4:1); ^1H NMR (500 MHz, CDCl_3) δ 7.34-7.30 (2H, m), 7.28-7.25 (1H, m), 7.21 (2H, d, J = 7.0 Hz), 4.71-4.66 (1H, m), 4.20-4.13 (2H, m), 3.98-3.91 (1H, m), 3.30

(1H, dd, $J = 3.0, 13.0$ Hz), 2.75-2.70 (1H, m), 1.90-1.84 (1H, m), 1.50-1.42 (1H, m), 1.41-1.25 (3H, m), 1.22-1.19 (1H, m), 1.16 (3H, d, $J = 7.0$ Hz), 1.14-1.08 (1H, m), 0.91 (3H, d, $J = 6.5$ Hz), 0.89 (3H, t, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 178.00, 153.17, 135.56, 129.59, 129.56, 129.07, 127.46, 66.09, 55.49, 41.01, 39.84, 38.19, 35.50, 30.33, 20.18, 19.25, 16.85, 14.43; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 318.2071, calcd for $\text{C}_{19}\text{H}_{28}\text{NO}_3$ 318.2069.

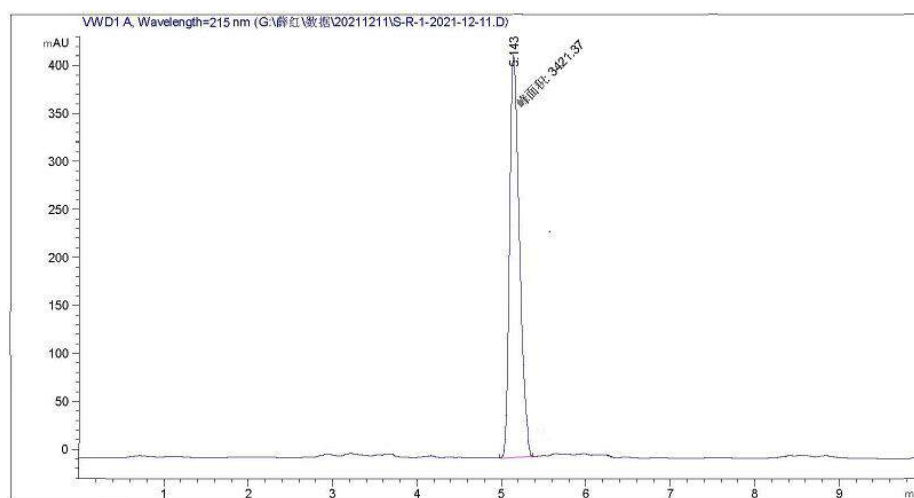


9a

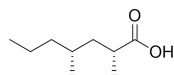
(2S, 4S)-2, 4-dimethylheptanoic acid (9a):

Colorless oil, 3.16 g, quantitative; $R_f = 0.6$ (petroleum/EtOAc = 4:1); $[\alpha]_D^{20} +27.86$ (c 0.14, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 2.60-2.52 (1H, m), 1.76-1.70 (1H, m), 1.55-1.44 (1H, m), 1.40-1.32 (1H, m), 1.31-1.22 (2H, m), 1.18 (3H, d, $J = 7.0$ Hz), 1.17-1.06 (2H, m), 0.90-0.86 (6H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 183.80, 41.37, 39.42, 37.46, 30.58, 19.99, 19.68, 17.94, 14.42; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 159.1382, calcd for $\text{C}_9\text{H}_{19}\text{O}_2$ 159.1385.

The pure **9a** was analyzed by chiral HPLC (Chiralcel IG column, 25°C , 1.0 mL/min, n-hexane:isopropanol = 90:10). Elution time = 5.143 min, ee% > 99%.



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	5.143	MF	0.1361	3421.37329	418.90640	100.0000



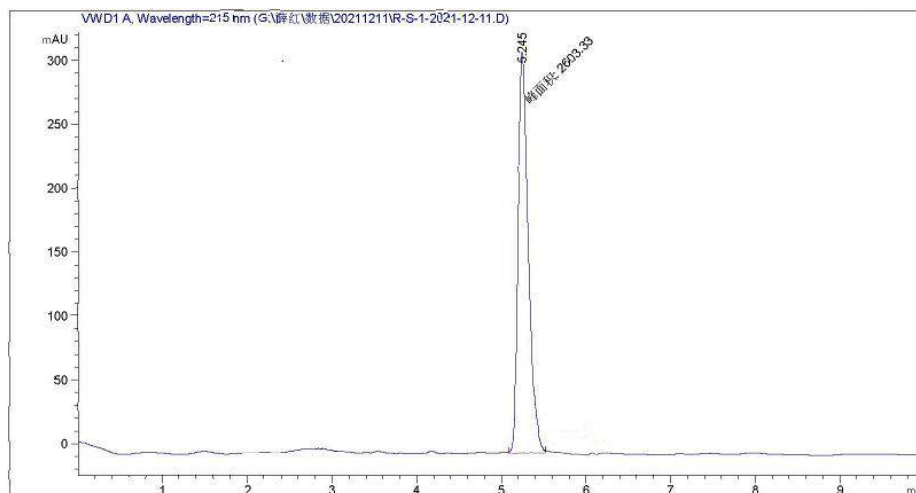
9b

(2R, 4R)-2, 4-dimethylheptanoic acid (9b):

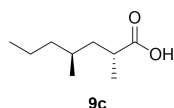
Colorless oil, 3.16 g, quantitative; $R_f = 0.6$ (petroleum/EtOAc = 4:1); $[\alpha]_D^{20} -27.33$ (c 0.14, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 2.60-2.52 (1H, m), 1.76-1.70 (1H, m), 1.53-1.44 (1H, m), 1.40-1.31 (1H, m), 1.30-1.23 (2H, m), 1.18 (3H, d, $J = 7.0$ Hz), 1.16-1.08 (2H, m), 0.90-0.86 (6H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 184.00, 41.36, 39.41, 37.47, 30.57, 19.98, 19.67, 17.93, 14.42; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 159.1391, calcd for $\text{C}_9\text{H}_{19}\text{O}_2$ 159.1385.

The pure **9b** was analyzed by chiral HPLC (Chiralcel IG column, 25°C , 1.0 mL/min, n-hexane:isopropanol =

90:10). Elution time = 5.245 min, ee% > 99%.



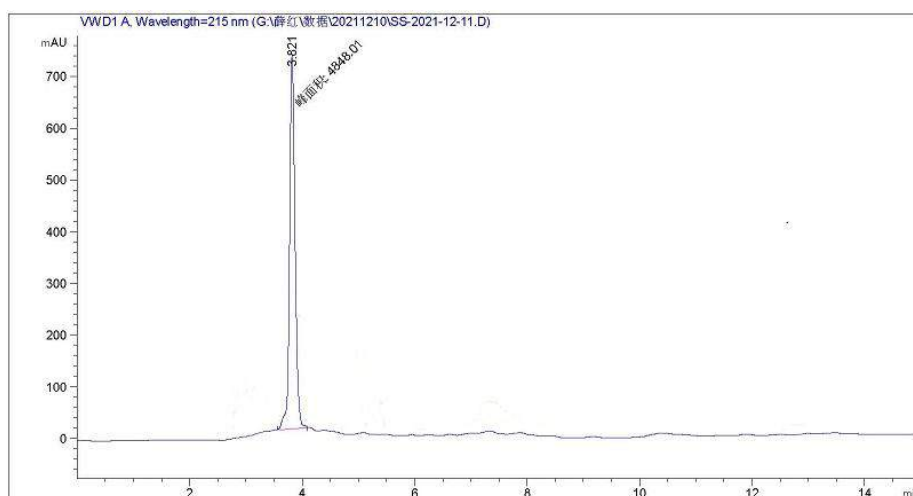
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	5.245	MM	0.1393	2603.32593	311.38516	100.0000



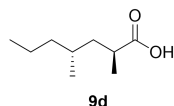
(2R, 4S)-2, 4-dimethylheptanoic acid (9c):

Colorless oil, 3.16 g, quantitative; $R_f = 0.6$ (petroleum/EtOAc = 4:1); $[\alpha]_D^{20} -12.71$ (c 0.14, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 2.58-2.50 (1H, m), 1.57-1.46 (2H, m), 1.40-1.33 (2H, m), 1.32-1.25 (2H, m), 1.15 (3H, d, $J = 7.0$ Hz), 1.13-1.09 (1H, m), 0.89-0.85 (6H, m); ¹³C NMR (125 MHz, CDCl₃) δ 183.86, 40.95, 39.44, 37.30, 30.33, 20.08, 19.44, 16.97, 14.42; HRMS (ESI) m/z : $[M + H]^+$ found for 159.1393, calcd for C₉H₁₉O₂ 159.1385.

The pure **9c** was analyzed by chiral HPLC (Chiralcel IG column, 25°C, 1.0 mL/min, n-hexane:isopropanol = 90:10). Elution time = 3.821 min, ee% > 99%.



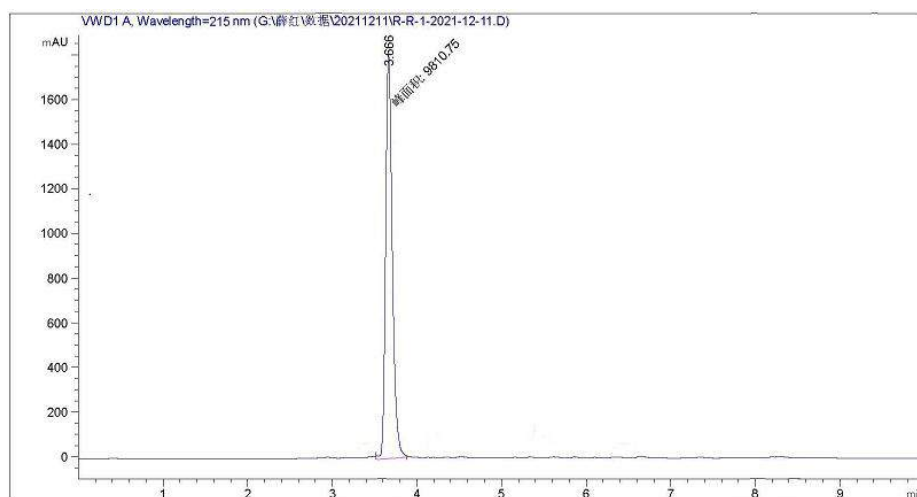
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	3.821	MM	0.1117	4848.01416	723.32825	100.0000



(2S, 4R)-2, 4-dimethylheptanoic acid (9d):

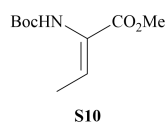
Colorless oil, 3.16 g, quantitative; $R_f = 0.6$ (petroleum/EtOAc = 4:1); $[\alpha]_D^{20} +13.17$ (c 0.14, CHCl₃); The data of ¹H NMR and ¹³C NMR are in the agreement with the reported data.⁶

The pure **9d** was analyzed by chiral HPLC (Chiralcel IG column, 25°C, 1.0 mL/min, n-hexane:isopropanol = 90:10). Elution time = 3.666 min, ee% > 99%.



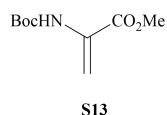
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	3.666	MM	0.0903	9810.75293	1811.06958	100.0000

4.2 Data of dehydropeptides



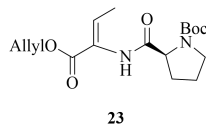
methyl (Z)-2-((tert-butoxycarbonyl)amino)but-2-enoate (S10):

White solid, 0.90 g, 84% yield; $R_f = 0.4$ (petroleum/EtOAc = 4:1); ¹H NMR (500 MHz, CDCl₃) δ 6.68 (1H, q, $J = 7.0$ Hz), 5.98 (1H, br), 3.77 (3H, s), 1.81 (3H, d, $J = 7.0$ Hz), 1.47 (9H, s); ¹³C NMR (125 MHz, CDCl₃) δ 165.45, 153.25, 132.18, 80.54, 52.34, 28.29, 14.34; HRMS (ESI) m/z : $[M + H]^+$ found for 216.1238, calcd for C₁₀H₁₈NO₄ 216.1236.



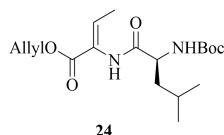
methyl 2-((*tert*-butoxycarbonyl)amino)acrylate (S13):

White solid, 0.81 g, 81% yield; $R_f = 0.5$ (petroleum/EtOAc = 4:1); $^1\text{H NMR}$ (500 MHz, CD_3OD) δ 6.01 (1H, br), 5.67 (1H, s), 4.72 (1H, s), 3.81 (3H, s), 1.48 (9H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 164.61, 152.71, 131.43, 105.32, 80.85, 53.02, 28.40; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 202.1073, calcd for $\text{C}_9\text{H}_{16}\text{NO}_4$ 202.1079.



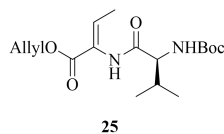
***tert*-butyl (*S, Z*)-2-((1-(allyloxy)-1-oxobut-2-en-2-yl)carbamoyl)pyrrolidine-1-carboxylate (23):**

White solid, 1.40 g, 83% yield; $R_f = 0.3$ (petroleum/EtOAc = 5:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 6.82 (1H, d, $J = 45.0$ Hz), 5.96-5.89 (1H, m), 5.33 (1H, d, $J = 17.0$ Hz), 5.24 (1H, d, $J = 10.5$ Hz), 4.69-4.62 (2H, m), 4.38 (1H, d, $J = 40.5$ Hz), 3.51-3.37 (2H, m), 2.37-1.93 (5H, m), 1.78 (3H, d, $J = 6.5$ Hz), 1.48 (9H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 171.25, 164.01, 155.97, 134.65, 132.04, 126.70, 118.76, 80.91, 65.86, 61.56, 47.23, 31.43, 28.43, 24.68, 14.82; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 339.1921, calcd for $\text{C}_{17}\text{H}_{27}\text{N}_2\text{O}_5$ 339.1920.



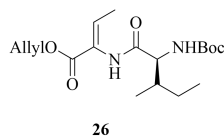
allyl (*S, Z*)-2-((*tert*-butoxycarbonyl)amino)-4-methylpentanamido)but-2-enoate (24):

White solid, 1.58 g, 89% yield; $R_f = 0.2$ (petroleum/EtOAc = 5:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.41 (1H, br), 6.86 (1H, q, $J = 7.0$ Hz), 5.96-5.88 (1H, m), 5.33 (1H, d, $J = 17.0$ Hz), 5.25 (1H, d, $J = 10.0$ Hz), 5.02 (1H, s), 4.66 (2H, d, $J = 5.5$ Hz), 4.22 (1H, s), 2.05 (1H, s), 1.78 (3H, d, $J = 7.5$ Hz), 1.74 (1H, s), 1.45 (9H, s), 1.26 (1H, s), 0.97-0.92 (6H, m); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 171.12, 164.07, 155.93, 134.58, 131.97, 126.08, 118.61, 80.36, 65.99, 53.35, 41.09, 28.39, 24.84, 23.03, 22.15, 14.65; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 355.2230, calcd for $\text{C}_{18}\text{H}_{31}\text{N}_2\text{O}_5$ 355.2233.



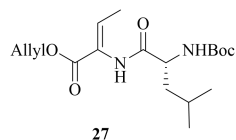
allyl (*S, Z*)-2-((*tert*-butoxycarbonyl)amino)-3-methylbutanamido)but-2-enoate (25):

White solid, 1.33 g, 78% yield; $R_f = 0.3$ (petroleum/EtOAc = 5:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.73 (1H, br), 6.84 (1H, q, $J = 7.0$ Hz), 5.94-5.89 (1H, m), 5.32 (1H, d, $J = 17.0$ Hz), 5.23 (2H, d, $J = 10.0$ Hz), 4.64 (2H, d, $J = 5.5$ Hz), 4.12 (1H, s), 2.20 (1H, s), 1.76 (3H, d, $J = 7.0$ Hz), 1.44 (9H, s), 1.03 (3H, d, $J = 6.5$ Hz), 0.98 (3H, d, $J = 6.5$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 170.51, 164.01, 156.05, 134.73, 131.90, 126.21, 118.57, 79.99, 65.93, 60.04, 30.94, 28.33, 19.35, 17.90, 14.53; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 341.2077, calcd for $\text{C}_{17}\text{H}_{29}\text{N}_2\text{O}_5$ 341.2076.



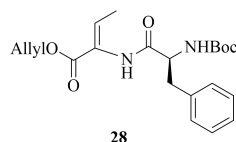
allyl (*Z*)-2-((2*S*, 3*S*)-2-((*tert*-butoxycarbonyl)amino)-3-methylpentanamido)but-2-enoate (26):

White solid, 1.31 g, 74% yield; $R_f = 0.3$ (petroleum/EtOAc = 5:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 6.83 (1H, br), 5.95-5.87 (1H, m), 5.34 (1H, d, $J = 17.0$ Hz), 5.26 (1H, d, $J = 10.0$ Hz), 4.94 (1H, d, $J = 6.5$ Hz), 4.66 (2H, t, $J = 5.0$ Hz), 4.63 (1H, dd, $J = 1.5, 9.0$ Hz), 4.39 (1H, s), 4.21-4.18 (1H, m), 2.10-2.05 (1H, m), 1.63 (1H, s), 1.46 (9H, s), 1.24 (3H, d, $J = 6.5$ Hz), 0.95 (3H, t, $J = 7.5$ Hz), 0.89 (3H, d, $J = 7.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 170.50, 164.11, 156.03, 134.80, 131.93, 125.93, 118.73, 80.28, 66.07, 58.54, 37.18, 28.39, 26.48, 14.83, 14.42, 11.82; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 355.2236, calcd for $\text{C}_{18}\text{H}_{31}\text{N}_2\text{O}_5$ 355.2233.



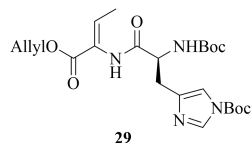
allyl (*R*, *Z*)-2-((2-((*tert*-butoxycarbonyl)amino)-4-methylpentanamido)but-2-enoate (27):

White solid, 1.35 g, 76% yield; $R_f = 0.2$ (petroleum/EtOAc = 5:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.48 (1H, br), 6.84 (1H, q, $J = 7.5$ Hz), 5.96-5.88 (1H, m), 5.33 (1H, dd, $J = 1.5, 17.0$ Hz), 5.24 (1H, d, $J = 10.5$ Hz), 4.90 (1H, s), 4.66 (2H, d, $J = 5.5$ Hz), 4.22 (1H, s), 1.77 (3H, d, $J = 7.0$ Hz), 1.66 (1H, s), 1.57 (1H, d, $J = 6.0$ Hz), 1.45 (9H, s), 1.21 (1H, s), 0.96 (6H, t, $J = 6.5$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 171.12, 164.06, 155.92, 134.57, 131.96, 126.07, 118.60, 80.35, 65.98, 53.34, 41.08, 28.38, 24.83, 23.02, 22.14, 14.64; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 355.2233, calcd for $\text{C}_{18}\text{H}_{31}\text{N}_2\text{O}_5$ 355.2233.



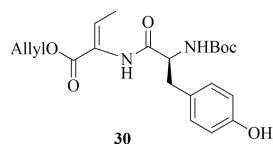
allyl (*S*, *Z*)-2-((2-((*tert*-butoxycarbonyl)amino)-3-phenylpropanamido)but-2-enoate (28):

White solid, 1.57 g, 81% yield; $R_f = 0.4$ (petroleum/EtOAc = 1:1); $^1\text{H NMR}$ (500 MHz, CD_2Cl_2) δ 7.31-7.28 (2H, m), 7.25-7.20 (3H, m), 6.77 (1H, q, $J = 7.0$ Hz), 5.97-5.89 (1H, m), 5.34-5.30 (1H, m), 5.23 (1H, dd, $J = 1.0, 10.5$ Hz), 4.63 (2H, d, $J = 5.5$ Hz), 4.47 (1H, s), 3.20-3.16 (1H, m), 3.02-2.98 (1H, m), 1.67 (3H, d, $J = 6.5$ Hz), 1.43 (1H, s), 1.39 (1H, s), 1.38 (9H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 169.72, 164.63, 155.55, 136.45, 134.54, 129.40, 128.69, 126.99, 125.75, 80.38, 70.26, 55.90, 52.31, 38.63, 28.24, 14.55; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 389.2073, calcd for $\text{C}_{21}\text{H}_{29}\text{N}_2\text{O}_5$ 389.2076.



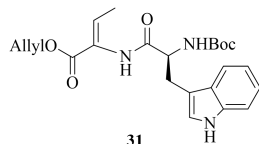
allyl (*S*, *Z*)-2-((2-((*tert*-butoxycarbonyl)amino)-3-(1*H*-imidazol-4-yl)propanamido)but-2-enoate (29):

White solid, 1.63 g, 86% yield; $R_f = 0.2$ (petroleum/EtOAc = 3:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.83 (1H, s), 7.26 (1H, q, $J = 7.0$ Hz), 7.20 (1H, s), 7.07 (1H, s), 5.87-5.79 (1H, m), 5.24 (1H, d, $J = 5.5$ Hz), 5.16 (1H, d, $J = 10.0$ Hz), 4.67 (1H, d, $J = 5.0$ Hz), 4.59 (2H, s), 3.51-3.46 (1H, m), 3.26 (1H, d, $J = 14.5$ Hz), 1.67 (3H, d, $J = 6.5$ Hz), 1.52 (9H, s), 1.51 (9H, s), 1.41-1.34 (2H, m); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 169.44, 161.82, 150.61, 148.62, 144.74, 137.20, 136.64, 131.71, 122.61, 118.71, 115.55, 85.90, 84.52, 66.35, 59.34, 28.16, 27.99, 27.85, 13.98; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 479.2508, calcd for $\text{C}_{23}\text{H}_{35}\text{N}_4\text{O}_7$ 479.2506.

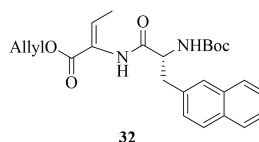


allyl (S, Z)-2-(2-((tert-butoxycarbonyl)amino)-3-(4-hydroxyphenyl)propanamido)but-2-enoate (30):

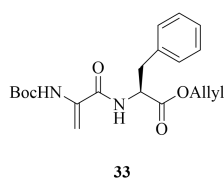
White solid, 1.64 g, 81% yield; $R_f = 0.3$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.04 (2H, d, $J = 8.5$ Hz), 6.73 (2H, d, $J = 8.5$ Hz), 6.07 (1H, br), 5.93-5.85 (1H, m), 5.33 (1H, d, $J = 17.0$ Hz), 5.25 (1H, d, $J = 10.5$ Hz), 5.14 (1H, s), 4.63 (1H, d, $J = 4.5$ Hz), 4.56 (1H, d, $J = 7.0$ Hz), 4.35-4.29 (1H, m), 2.99 (2H, t, $J = 8.0$ Hz), 2.81 (1H, s), 1.75 (1H, s), 1.41 (9H, s), 1.16 (3H, d, $J = 6.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 169.71, 163.92, 151.95, 150.20, 134.85, 134.03, 131.96, 130.48, 125.88, 121.59, 118.65, 83.69, 66.03, 55.87, 37.24, 28.36, 27.81, 14.70; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 405.2028, calcd for $\text{C}_{21}\text{H}_{29}\text{N}_2\text{O}_6$ 405.2026.

**allyl (S, Z)-2-(2-((tert-butoxycarbonyl)amino)-3-(1H-indol-3-yl)propanamido)but-2-enoate (31):**

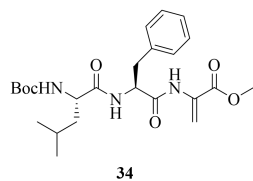
White solid, 1.88 g, 88% yield; $R_f = 0.3$ (petroleum/EtOAc = 1:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.10 (1H, br), 7.68 (1H, d, $J = 7.5$ Hz), 7.36 (1H, d, $J = 8.0$ Hz), 7.29 (1H, s), 7.21 (1H, t, $J = 7.0$ Hz), 7.15-7.12 (2H, m), 6.80 (1H, q, $J = 7.0$ Hz), 5.93-5.85 (1H, m), 5.31 (1H, d, $J = 17.0$ Hz), 5.23 (1H, d, $J = 10.5$ Hz), 5.13 (1H, s), 4.60 (3H, d, $J = 5.5$ Hz), 1.68-1.66 (3H, m), 1.42 (9H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 170.26, 170.14, 163.95, 136.34, 134.63, 132.01, 129.02, 127.67, 125.91, 123.50, 122.44, 119.96, 119.00, 118.59, 111.32, 65.99, 61.79, 55.44, 29.84, 28.40, 14.72; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 428.2158, calcd for $\text{C}_{23}\text{H}_{30}\text{N}_3\text{O}_5$ 428.2158.

**allyl (R, Z)-2-(2-((tert-butoxycarbonyl)amino)-3-(naphthalen-2-yl)propanamido)but-2-enoate (32):**

White solid, 1.88 g, 86% yield; $R_f = 0.4$ (petroleum/EtOAc = 1:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.78-7.74 (3H, m), 7.72-7.70 (1H, m), 7.59 (1H, s), 7.46-7.42 (3H, m), 7.20 (1H, dd, $J = 1.0, 8.0$ Hz), 7.05 (1H, q, $J = 7.0$ Hz), 5.90-5.82 (1H, m), 5.29-5.21 (2H, m), 4.90 (1H, dd, $J = 2.5, 5.0$ Hz), 4.62 (2H, t, $J = 7.0$ Hz), 3.79-3.75 (1H, m), 3.51-3.48 (1H, m), 1.67 (9H, s), 0.64 (3H, d, $J = 7.5$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 171.28, 169.08, 161.54, 149.97, 148.87, 144.53, 133.51, 131.60, 129.25, 127.82, 127.66, 126.48, 122.20, 118.73, 84.88, 66.31, 61.02, 60.52, 34.97, 28.24, 21.18, 14.31, 13.07; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 439.2231, calcd for $\text{C}_{25}\text{H}_{31}\text{N}_2\text{O}_5$ 439.2233.

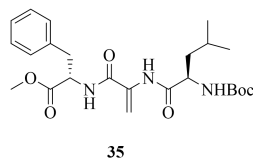
**allyl (2-((tert-butoxycarbonyl)amino)acryloyl)-L-phenylalaninate (33):**

White solid, 1.53 g, 82% yield; $R_f = 0.4$ (petroleum/EtOAc = 1:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.37 (1H, br), 7.28-7.25 (2H, m), 7.20-7.19 (3H, m), 6.62 (1H, s), 5.93-5.86 (2H, m), 5.43 (1H, s), 5.31 (1H, dd, $J = 1.0, 17.0$ Hz), 5.26-5.24 (1H, m), 4.65 (2H, d, $J = 5.5$ Hz), 4.52 (1H, s), 3.20-3.16 (1H, m), 3.04 (1H, s), 1.38 (9H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 170.18, 162.82, 136.22, 130.96, 130.43, 128.82, 128.22, 126.50, 118.45, 109.02, 66.03, 37.66, 27.80; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 375.1917, calcd for $\text{C}_{20}\text{H}_{27}\text{N}_2\text{O}_5$ 375.1920.



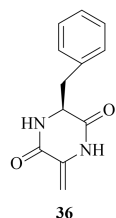
methyl (6*S*, 9*S*)-9-benzyl-6-isobutyl-2, 2-dimethyl-12-methylene-4, 7, 10-trioxo-3-oxa-5, 8, 11-triazatridecan-13-oate (34):

White solid, 1.85 g, 80% yield; R_f = 0.2 (petroleum/EtOAc = 1:1); ^1H NMR (500 MHz, CDCl_3) δ 8.19 (1H, br), 7.28-7.25 (2H, m), 7.23-7.18 (4H, m), 7.00 (1H, br), 6.53 (1H, s), 5.88 (1H, s), 5.07 (1H, s), 4.84-4.77 (1H, m), 3.77 (3H, s), 3.14-3.08 (2H, m), 1.64-1.55 (2H, m), 1.46 (1H, s), 1.42 (9H, s), 0.90 (6H, d, J = 6.5 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 169.70, 163.84, 155.68, 136.17, 130.80, 129.26, 128.64, 127.02, 109.83, 60.40, 55.06, 52.87, 41.21, 38.16, 28.29, 27.67, 24.70, 22.94; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 462.2067, calcd for $\text{C}_{24}\text{H}_{36}\text{N}_3\text{O}_6$ 462.2064.



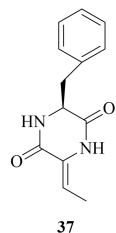
methyl (2-((*R*)-2-((*tert*-butoxycarbonyl)amino)-4-methylpentanamido)acryloyl)-*L*-phenylalaninate (35):

White solid, 1.87 g, 81% yield; R_f = 0.2 (petroleum/EtOAc = 1:1); ^1H NMR (500 MHz, DMSO-d_6) δ 7.25-7.17 (6H, m), 6.85 (1H, d, J = 8.0 Hz), 6.19 (1H, s), 5.73 (1H, d, J = 9.0 Hz), 4.75 (1H, q, J = 8.0 Hz), 3.75-3.62 (3H, m), 3.06-2.99 (1H, m), 2.88-2.75 (1H, m), 1.48-1.44 (1H, m), 1.42 (1H, s), 1.36 (9H, s), 1.31-1.23 (3H, m), 0.81 (3H, d, J = 6.5 Hz), 0.78 (3H, d, J = 6.0 Hz); ^{13}C NMR (125 MHz, DMSO-d_6) δ 173.06, 171.26, 164.12, 155.62, 137.81, 132.83, 129.72, 128.74, 126.74, 110.25, 78.54, 54.40, 53.42, 53.09, 41.30, 28.63, 27.78, 24.62, 23.29, 22.03; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 462.2065, calcd for $\text{C}_{24}\text{H}_{36}\text{N}_3\text{O}_6$ 462.2064.



(*S*)-3-benzyl-6-methylenepiperazine-2, 5-dione (36):

White solid, 0.85 g, 79% yield; R_f = 0.2 (petroleum/EtOAc = 4:1); ^1H NMR (500 MHz, DMSO-d_6) δ 7.25-7.20 (3H, m), 7.12 (2H, d, J = 6.5 Hz), 4.90 (1H, s), 4.50 (1H, s), 4.38 (1H, t, J = 4.0 Hz), 4.04 (1H, s), 3.31 (1H, s), 3.14 (1H, dd, J = 3.5, 14.0 Hz), 2.90 (1H, dd, J = 5.0, 13.0 Hz); ^{13}C NMR (125 MHz, DMSO-d_6) δ 165.11, 158.06, 135.40, 134.21, 130.03, 128.04, 126.72, 98.50, 56.09, 55.99; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 217.0975, calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2\text{O}_2$ 217.0977.

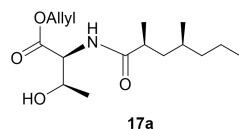


(*S*, *Z*)-3-benzyl-6-ethylidenepiperazine-2, 5-dione (37):

White solid, 0.94 g, 82% yield; R_f = 0.4 (petroleum/EtOAc = 3:1); ^1H NMR (500 MHz, CD_3OD) δ 7.23-7.21 (3H,

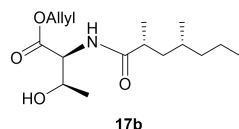
m), 7.17-7.15 (2H, m), 5.63 (1H, q, $J = 7.5$ Hz), 4.35 (1H, t, $J = 4.5$ Hz), 3.20 (1H, dd, $J = 4.5, 13.5$ Hz), 3.00 (1H, dd, $J = 4.5, 13.5$ Hz), 1.51 (3H, d, $J = 7.5$ Hz); ^{13}C NMR (125 MHz, CD_3OD) δ 168.16, 162.46, 135.93, 131.43, 129.41, 128.31, 128.26, 115.11, 57.98, 41.39, 10.81; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 231.1138, calcd for $\text{C}_{13}\text{H}_{15}\text{N}_2\text{O}_2$ 231.1134.

4.3 Data of compound 1



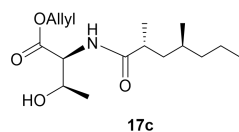
allyl ((2*S*, 4*S*)-2, 4-dimethylheptanoyl)-*L*-threoninate (17a):

White solid, 2.54 g, 85.0% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); ^1H NMR (500 MHz, CDCl_3) δ 6.35 (1H, br), 5.91-5.83 (1H, m), 5.31 (1H, d, $J = 17.0$ Hz), 5.22 (1H, d, $J = 10.5$ Hz), 4.63-4.60 (3H, m), 4.37-4.35 (1H, m), 2.87 (1H, br), 2.46-2.38 (1H, m), 1.73-1.67 (1H, m), 1.48-1.42 (1H, m), 1.35-1.20 (3H, m), 1.18 (3H, d, $J = 6.5$ Hz), 1.14 (3H, d, $J = 7.0$ Hz), 1.12-1.02 (2H, m), 0.86 (3H, d, $J = 6.5$ Hz), 0.83 (3H, t, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 177.53, 170.95, 131.60, 119.00, 68.12, 66.22, 57.01, 41.88, 39.63, 39.35, 30.40, 20.02, 19.70, 19.01, 14.42; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 300.2179, calcd for $\text{C}_{16}\text{H}_{30}\text{NO}_4$ 300.2175.



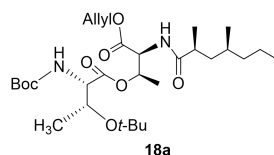
allyl ((2*R*, 4*R*)-2, 4-dimethylheptanoyl)-*L*-threoninate (17b):

White solid, 2.57 g, 86% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); ^1H NMR (500 MHz, CDCl_3) δ 6.25-6.22 (1H, m), 5.94-5.86 (1H, m), 5.36-5.24 (2H, m), 4.69-4.64 (2H, m), 4.63 (1H, d, $J = 2.5$ Hz), 4.41-4.32 (1H, m), 2.47-2.40 (1H, m), 2.27-2.21 (1H, m), 1.76-1.70 (1H, m), 1.54-1.39 (1H, m), 1.37-1.31 (1H, m), 1.29-1.23 (2H, m), 1.21 (3H, d, $J = 6.5$ Hz), 1.16 (3H, d, $J = 7.0$ Hz), 1.14-1.05 (1H, m), 0.92 (1H, d, $J = 6.5$ Hz), 0.89 (2H, d, $J = 6.5$ Hz), 0.86 (3H, t, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 177.45, 171.05, 131.59, 119.05, 68.13, 66.26, 57.08, 41.84, 39.63, 39.43, 30.59, 20.10, 20.05, 19.64, 18.81, 14.40; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 300.2178, calcd for $\text{C}_{16}\text{H}_{30}\text{NO}_4$ 300.2175.



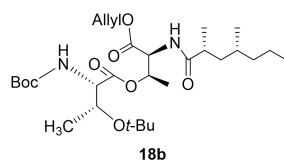
allyl ((2*R*, 4*S*)-2, 4-dimethylheptanoyl)-*L*-threoninate (17c):

White solid, 2.54 g, 85% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); ^1H NMR (500 MHz, CDCl_3) δ 6.30 (1H, d, $J = 8.5$ Hz), 5.94-5.86 (1H, m), 5.35-5.31 (1H, m), 5.26-5.23 (1H, m), 4.67-4.64 (2H, m), 4.62 (1H, dd, $J = 2.5, 9.0$ Hz), 4.40-4.34 (1H, m), 2.52-2.48 (1H, m), 2.41 (1H, q, $J = 7.0$ Hz), 1.53-1.45 (2H, m), 1.39-1.33 (1H, m), 1.32-1.23 (2H, m), 1.21 (3H, d, $J = 6.5$ Hz), 1.13 (3H, d, $J = 7.0$ Hz), 1.11-1.07 (1H, m), 0.92 (1H, d, $J = 6.5$ Hz), 0.88-0.84 (6H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 177.83, 171.02, 131.62, 118.90, 67.99, 66.17, 57.19, 41.48, 39.34, 39.20, 30.34, 20.10, 20.03, 19.60, 17.82, 14.43; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 300.2176, calcd for $\text{C}_{16}\text{H}_{30}\text{NO}_4$ 300.2175.



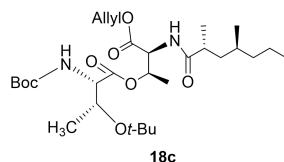
allyl O-(N-(tert-butoxycarbonyl)-O-(tert-butyl)-L-threonyl)-N-((2S, 4S)-2, 4-dimethylheptanoyl)-L-threoninate (18a):

Colorless oil, 4.36 g, 98% yield; R_f = 0.3 (petroleum/EtOAc = 5:1); ^1H NMR (500 MHz, CDCl_3) δ 6.20 (1H, d, J = 9.0 Hz), 5.91-5.83 (1H, m), 5.42-5.41 (1H, m), 5.30 (1H, d, J = 17.0 Hz), 5.22 (2H, d, J = 10.5 Hz), 4.84-4.82 (1H, m), 4.64-4.60 (2H, m), 4.10-4.03 (2H, m), 2.47-2.41 (1H, m), 1.75-1.71 (1H, m), 1.43 (9H, s), 1.32 (1H, d, J = 6.5 Hz), 1.27 (3H, d, J = 6.5 Hz), 1.25-1.20 (2H, m), 1.18-1.16 (6H, m), 1.12 (9H, s), 0.92 (1H, d, J = 6.5 Hz), 0.88 (3H, d, J = 6.0 Hz), 0.84 (3H, t, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 177.23, 170.64, 169.40, 156.04, 131.61, 119.25, 79.85, 74.07, 71.96, 67.08, 66.59, 59.41, 55.28, 41.69, 39.56, 39.24, 30.40, 28.61, 28.41, 20.66, 20.02, 19.77, 19.08, 17.30, 14.40; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ found for 579.3625, calcd for $\text{C}_{29}\text{H}_{52}\text{N}_2\text{O}_8\text{Na}$ 579.3621.



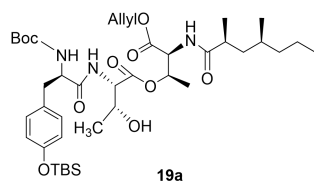
allyl O-(N-(tert-butoxycarbonyl)-O-(tert-butyl)-L-threonyl)-N-((2R, 4R)-2, 4-dimethylheptanoyl)-L-threoninate (18b):

Colorless oil, 4.41 g, 99% yield; R_f = 0.3 (petroleum/EtOAc = 5:1); ^1H NMR (500 MHz, CDCl_3) δ 6.26 (1H, d, J = 9.0 Hz), 5.92-5.84 (1H, m), 5.45-5.41 (1H, m), 5.31 (1H, d, J = 17.0 Hz), 5.25-5.22 (2H, m), 4.83 (1H, dd, J = 2.5, 9.0 Hz), 4.62 (2H, d, J = 6.0 Hz), 4.05 (2H, d, J = 7.0 Hz), 2.49-2.45 (1H, m), 1.71-1.72 (1H, m), 1.44 (11H, s), 1.29 (3H, d, J = 6.5 Hz), 1.26-1.22 (2H, m), 1.19-1.15 (7H, m), 1.13 (10H, s), 1.78 (3H, d, J = 6.5 Hz), 0.88 (3H, t, J = 7.0 Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 177.23, 170.59, 169.51, 156.03, 131.66, 119.29, 79.91, 74.14, 72.05, 67.18, 66.63, 59.40, 55.34, 41.82, 39.63, 39.32, 30.62, 28.66, 28.45, 20.57, 20.06, 19.62, 18.75, 17.39, 14.39; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ found for 579.3621, calcd for $\text{C}_{29}\text{H}_{52}\text{N}_2\text{O}_8\text{Na}$ 579.3621.



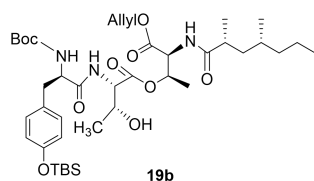
allyl O-(N-(tert-butoxycarbonyl)-O-(tert-butyl)-L-threonyl)-N-((2R, 4S)-2, 4-dimethylheptanoyl)-L-threoninate (18c):

Colorless oil, 4.27 g, 96% yield; R_f = 0.3 (petroleum/EtOAc = 5:1); ^1H NMR (500 MHz, CDCl_3) δ 6.26 (1H, d, J = 9.0 Hz), 5.95-5.84 (1H, m), 5.45-5.41 (1H, m), 5.34-5.29 (1H, m), 5.25-5.23 (2H, m), 4.83 (1H, dd, J = 2.5, 9.0 Hz), 4.65-4.61 (2H, m), 4.07-4.04 (2H, m), 2.47-2.42 (1H, m), 1.55-1.47 (2H, m), 1.44 (9H, s), 1.41-1.32 (2H, m), 1.29 (3H, d, J = 6.5 Hz), 1.27-1.22 (2H, m), 1.17 (3H, d, J = 6.0 Hz), 1.14 (3H, s), 1.13 (9H, s), 0.96-0.92 (1H, m), 0.89-0.86 (6H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 177.55, 170.60, 169.52, 156.04, 131.65, 119.27, 79.89, 74.11, 71.99, 67.11, 66.63, 59.38, 55.34, 41.49, 39.31, 39.14, 30.39, 28.63, 28.42, 20.60, 20.04, 19.68, 17.83, 17.37, 14.47; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ found for 579.3621, calcd for $\text{C}_{29}\text{H}_{52}\text{N}_2\text{O}_8\text{Na}$ 579.3621.



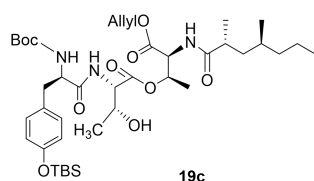
allyl *O*-(((*R*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-((*tert*-butyldimethylsilyl)oxy)phenyl)propanoyl)-*L*-threonyl)-*N*-((2*S*, 4*S*)-2, 4-dimethylheptanoyl)-*L*-threoninate (19a):

White amorphous solid, 4.63 g, 85% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.08 (2H, d, $J = 8.5$ Hz), 6.77 (2H, d, $J = 8.0$ Hz), 6.70 (1H, s), 6.58 (1H, d, $J = 7.5$ Hz), 5.93-5.85 (1H, m), 5.48-5.46 (1H, m), 5.35-5.30 (1H, m), 5.27-5.25 (1H, m), 5.08 (1H, br), 4.76 (1H, d, $J = 8.0$ Hz), 4.60 (2H, d, $J = 6.0$ Hz), 4.35-4.28 (2H, m), 4.24-4.22 (1H, m), 3.09-3.05 (1H, m), 2.96-2.91 (1H, m), 1.76-1.70 (1H, m), 1.41 (2H, d, $J = 3.0$ Hz), 1.40 (9H, s), 1.29 (4H, d, $J = 7.0$ Hz), 1.27-1.21 (3H, m), 1.16 (3H, d, $J = 7.0$ Hz), 1.14-1.06 (2H, m), 1.03 (3H, d, $J = 5.0$ Hz), 0.97 (9H, s), 0.90 (3H, d, $J = 6.5$ Hz), 0.85 (3H, t, $J = 7.0$ Hz), 0.18 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 177.94, 172.46, 170.69, 169.92, 154.90, 131.48, 130.49, 130.30, 129.17, 120.45, 120.28, 119.47, 71.57, 67.49, 66.81, 57.75, 55.51, 41.97, 39.67, 38.74, 37.60, 30.42, 28.43, 25.80, 20.08, 19.80, 19.78, 19.01, 18.32, 17.08, 14.45, -4.31; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 778.4672, calcd for $\text{C}_{40}\text{H}_{68}\text{N}_3\text{O}_{10}\text{Si}$ 778.4674.



allyl *O*-(((*R*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-((*tert*-butyldimethylsilyl)oxy)phenyl)propanoyl)-*L*-threonyl)-*N*-((2*R*, 4*R*)-2, 4-dimethylheptanoyl)-*L*-threoninate (19b):

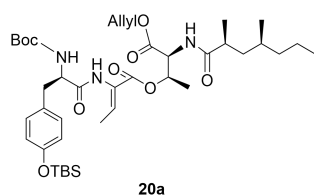
White amorphous solid, 4.68 g, 86% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.09-7.06 (2H, m), 6.77-6.73 (3H, m), 6.54 (1H, s), 5.92-5.84 (1H, m), 5.49-5.45 (1H, m), 5.34-5.31 (1H, m), 5.25 (1H, d, $J = 10.5$ Hz), 5.09 (1H, d, $J = 7.5$ Hz), 4.78 (1H, d, $J = 7.5$ Hz), 4.60 (2H, d, $J = 6.0$ Hz), 4.40-4.31 (2H, m), 4.21-4.20 (1H, m), 3.13-3.09 (1H, m), 2.95-2.91 (1H, m), 2.56-2.50 (1H, m), 1.77-1.71 (1H, m), 1.38 (9H, s), 1.34-1.22 (5H, m), 2.30 (3H, d, $J = 7.0$ Hz), 1.13-1.05 (4H, m), 0.96 (9H, s), 0.91-0.88 (5H, m), 0.84 (3H, t, $J = 7.0$ Hz), 0.18 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 177.67, 172.45, 172.39, 170.61, 169.97, 169.85, 154.81, 154.79, 131.45, 130.33, 130.30, 129.31, 120.38, 119.51, 67.49, 66.85, 57.67, 55.32, 41.68, 39.66, 39.04, 30.64, 28.38, 25.78, 20.07, 19.72, 19.61, 19.14, 18.89, 18.29, 17.14, 14.41, -4.33; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 778.4658, calcd for $\text{C}_{40}\text{H}_{68}\text{N}_3\text{O}_{10}\text{Si}$ 778.4674.



allyl *O*-(((*R*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-((*tert*-butyldimethylsilyl)oxy)phenyl)propanoyl)-*L*-threonyl)-*N*-((2*R*, 4*S*)-2, 4-dimethylheptanoyl)-*L*-threoninate (19c):

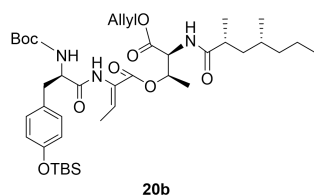
White amorphous solid, 4.57 g, 84% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.07 (2H, d, $J = 8.0$ Hz), 6.76 (2H, d, $J = 8.0$ Hz), 6.71 (1H, d, $J = 8.0$ Hz), 6.51 (1H, s), 5.92-5.84 (1H, m), 5.49-5.45 (1H, m), 5.33 (1H, dd, $J = 1.5, 17.0$ Hz), 5.25 (1H, dd, $J = 1.0, 10.5$ Hz), 5.07 (1H, s), 4.77 (1H, d, $J = 7.5$ Hz), 4.61 (2H, d, $J = 6.0$ Hz), 4.40-4.32 (2H, m), 4.21 (1H, d, $J = 6.5$ Hz), 3.14-3.10 (1H, m), 2.95-2.90 (1H, m), 2.63 (1H, s), 2.53-2.47 (1H, m), 1.54-1.47 (1H, m), 1.46-1.41 (2H, m), 1.38 (9H, s), 1.35-1.31 (2H, m), 1.29 (3H, d, $J = 6.5$ Hz), 1.26-1.21 (1H, m), 1.14 (3H, d, $J = 6.5$ Hz), 1.07 (3H, d, $J = 6.5$ Hz), 0.96 (9H, s), 0.91-0.88 (2H, m), 0.87-0.84 (5H, m), 0.17 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 178.01, 172.44, 172.37, 170.55, 169.99, 169.85, 154.80, 131.45, 130.30, 129.30, 120.37, 119.50, 80.40, 71.67, 67.42, 66.83, 57.64, 55.38, 41.30, 39.44, 38.95, 37.53, 30.43, 28.39, 25.77, 20.10, 19.73, 19.61, 19.13, 18.29, 17.95, 17.15, 14.46, -4.34; HRMS (ESI)

m/z: [M + H]⁺ found for 778.4673, calcd for C₄₀H₆₈N₃O₁₀Si 778.4674.



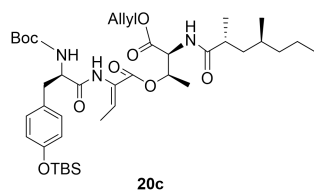
(2R, 3S)-4-(allyloxy)-3-((2S, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (Z)-2-((R)-2-((tert-butoxycarbonyl)amino)-3-(4-((tert-butyl dimethylsilyl)oxy)phenyl)propanamido)but-2-enoate (20a):

Yellow solid, 3.87 g, 85%; *R_f* = 0.5 (petroleum/EtOAc = 2:1); ¹H NMR (500 MHz, CDCl₃) δ 7.20 (1H, s), 7.10 (2H, d, *J* = 8.5 Hz), 6.88 (1H, s), 6.79-6.77 (3H, m), 5.91-5.83 (1H, m), 5.45 (1H, s), 5.33-5.29 (1H, m), 5.21 (1H, d, *J* = 10.0 Hz), 4.94 (1H, s), 4.91 (1H, dd, *J* = 3.0, 9.0 Hz), 4.63 (2H, d, *J* = 6.0 Hz), 4.44 (1H, d, *J* = 6.0 Hz), 3.09-3.04 (2H, m), 1.79-1.74 (1H, m), 1.69-1.66 (4H, m), 1.43 (9H, s), 1.35-1.31 (2H, m), 1.28 (3H, d, *J* = 6.5 Hz), 1.26-1.21 (2H, m), 1.15 (3H, d, *J* = 7.0 Hz), 1.11-1.06 (2H, m), 0.97 (9H, s), 0.91 (3H, d, *J* = 6.5 Hz), 0.86 (3H, t, *J* = 7.0 Hz), 0.18 (6H, s); ¹³C NMR (125 MHz, CDCl₃) δ 177.82, 170.48, 169.68, 162.96, 154.95, 135.73, 131.72, 130.49, 130.44, 128.98, 126.33, 120.52, 119.33, 119.24, 80.77, 72.23, 66.58, 55.47, 41.96, 39.83, 38.77, 36.59, 30.51, 28.38, 25.80, 20.10, 19.73, 19.30, 18.33, 16.92, 14.47, 14.41, -4.30; HRMS (ESI) m/z: [M + H]⁺ found for 760.4564, calcd for C₄₀H₆₆N₃O₉Si 760.4568.



(2R, 3S)-4-(allyloxy)-3-((2R, 4R)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (Z)-2-((R)-2-((tert-butoxycarbonyl)amino)-3-(4-((tert-butyl dimethylsilyl)oxy)phenyl)propanamido)but-2-enoate (20b):

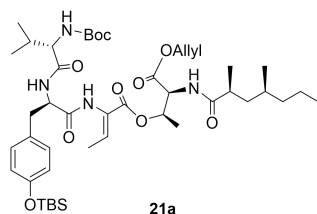
Yellow solid, 3.74 g, 82%; *R_f* = 0.6 (petroleum/EtOAc = 2:1); ¹H NMR (500 MHz, CDCl₃) δ 7.28 (1H, s), 7.10 (2H, d, *J* = 8.0 Hz), 6.78-6.70 (3H, m), 5.91-5.83 (1H, m), 5.43-5.41 (1H, m), 5.33-5.29 (1H, m), 5.26-5.21 (1H, m), 4.95 (1H, s), 4.87 (1H, dd, *J* = 3.0, 9.0 Hz), 4.66-4.56 (2H, m), 4.43 (1H, s), 3.88-3.07 (2H, m), 1.83-1.74 (1H, m), 1.70-1.66 (4H, m), 1.42 (9H, s), 1.37 (2H, d, *J* = 6.5 Hz), 1.35-1.31 (1H, m), 1.29 (3H, d, *J* = 6.5 Hz), 1.27-1.21 (2H, m), 1.17 (3H, d, *J* = 6.5 Hz), 1.13-1.07 (1H, m), 0.97 (9H, s), 0.92 (2H, d, *J* = 6.5 Hz), 0.88-0.83 (5H, m), 0.18 (6H, s); ¹³C NMR (125 MHz, CDCl₃) δ 177.57, 170.47, 169.74, 163.06, 154.90, 135.10, 131.65, 130.45, 130.36, 130.30, 129.08, 120.49, 120.43, 120.31, 119.27, 72.23, 66.62, 55.56, 41.60, 39.76, 39.10, 30.69, 28.40, 25.80, 20.09, 19.70, 19.17, 19.09, 18.32, 17.05, 16.88, 14.44, -4.30; HRMS (ESI) m/z: [M + H]⁺ found for 760.4577, calcd for C₄₀H₆₆N₃O₉Si 760.4568.



(2R, 3S)-4-(allyloxy)-3-((2R, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (Z)-2-((R)-2-((tert-butoxycarbonyl)amino)-3-(4-((tert-butyl dimethylsilyl)oxy)phenyl)propanamido)but-2-enoate (20c):

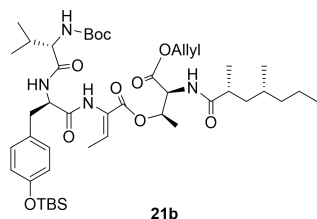
Yellow solid, 3.78 g, 83%; *R_f* = 0.5 (petroleum/EtOAc = 2:1); ¹H NMR (500 MHz, CDCl₃) δ 7.29 (1H, s), 7.10 (2H, d, *J* = 8.0 Hz), 6.77 (2H, d, *J* = 8.0 Hz), 6.72 (1H, q, *J* = 7.0 Hz), 6.55 (1H, s), 5.91-5.83 (1H, m), 5.42-5.40 (1H, m), 5.31 (1H, dd, *J* = 1.5, 17.0 Hz), 5.22 (1H, dd, *J* = 1.0, 10.0 Hz), 4.98 (1H, s), 4.86 (1H, dd, *J* = 3.5, 9.5 Hz), 4.64-4.62 (2H, m), 4.43 (1H, s), 3.11-3.03 (2H, m), 2.53-2.48 (1H, m), 1.87-1.74 (1H, m), 1.69 (3H, d, *J* = 7.5

(Hz), 1.57-1.51 (1H, m), 1.49-1.44 (1H, m), 1.41 (9H, s), 1.38-1.31 (1H, m), 1.29 (3H, d, $J = 6.0$ Hz), 1.27-1.17 (1H, m), 1.15 (3H, d, $J = 7.0$ Hz), 1.10-1.03 (1H, m), 0.97 (9H, s), 0.91 (1H, d, $J = 7.0$ Hz), 0.88-0.84 (6H, m), 0.17 (6H, s); ^{13}C NMR (125 MHz, CDCl_3) δ 177.89, 170.46, 169.74, 163.05, 154.87, 135.06, 131.61, 130.43, 129.09, 126.24, 120.46, 119.29, 80.62, 72.22, 66.60, 55.60, 41.27, 39.41, 39.02, 36.87, 30.50, 28.39, 25.78, 20.10, 20.07, 19.73, 19.15, 18.30, 18.23, 17.07, 14.47, 14.43, -4.33; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 760.4561, calcd for $\text{C}_{40}\text{H}_{66}\text{N}_3\text{O}_9\text{Si}$ 760.4568.



(2R, 3S)-4-(allyloxy)-3-((2S, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6S, 9R, Z)-9-(4-(tert-butylidimethylsilyloxy)benzyl)-12-ethylidene-6-isopropyl-2,2-dimethyl-4, 7, 10-trioxo-3-oxa-5, 8, 11-triazatridecan-13-oate (21a):

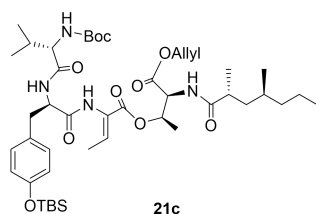
White amorphous solid, 4.08 g, 90% over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); ^1H NMR (500 MHz, CDCl_3) δ 8.18 (1H, s), 7.78 (1H, d, $J = 10.0$ Hz), 7.08 (2H, d, $J = 8.5$ Hz), 6.76-6.73 (3H, m), 6.13 (1H, d, $J = 8.0$ Hz), 5.97-5.89 (1H, m), 5.47-5.44 (1H, m), 5.33-5.29 (1H, m), 5.20 (1H, d, $J = 11.0$ Hz), 4.95 (1H, dd, $J = 2.5, 9.5$ Hz), 4.90 (1H, d, $J = 6.0$ Hz), 4.85 (1H, q, $J = 7.0$ Hz), 4.71 (2H, d, $J = 5.5$ Hz), 3.39 (1H, t, $J = 7.0$ Hz), 3.16-3.10 (2H, m), 2.83 (1H, s), 1.85-1.77 (2H, m), 1.68 (3H, d, $J = 7.0$ Hz), 1.45-1.40 (2H, m), 1.32 (2H, s), 1.28 (9H, s), 1.19 (3H, d, $J = 6.5$ Hz), 1.16 (3H, d, $J = 7.0$ Hz), 1.09-1.00 (2H, m), 0.96 (9H, s), 0.92 (3H, d, $J = 6.5$ Hz), 0.86-0.82 (6H, m), 0.77 (3H, d, $J = 6.5$ Hz), 0.17 (6H, s); ^{13}C NMR (125 MHz, CDCl_3) δ 178.08, 172.35, 170.62, 169.45, 163.29, 156.75, 155.03, 135.49, 132.22, 130.34, 129.04, 127.90, 120.59, 118.56, 80.79, 72.46, 66.60, 62.32, 55.09, 54.25, 41.56, 39.93, 38.15, 35.93, 30.51, 29.93, 28.34, 28.29, 25.78, 20.11, 19.74, 19.63, 19.22, 19.19, 18.96, 18.31, 16.75, 14.47, 13.48, -4.33; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 859.5255, calcd for $\text{C}_{40}\text{H}_{75}\text{N}_6\text{O}_{12}\text{Si}$ 859.5252.



(2R, 3S)-4-(allyloxy)-3-((2R, 4R)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6S, 9R, Z)-9-(4-(tert-butylidimethylsilyloxy)benzyl)-12-ethylidene-6-isopropyl-2, 2-dimethyl-4, 7, 10-trioxo-3-oxa-5, 8, 11-triazatridecan-13-oate (21b):

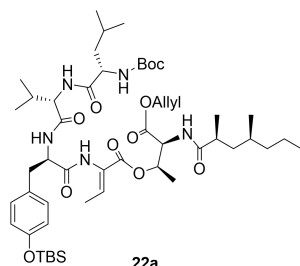
White amorphous solid, 3.95 g, 92% over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); ^1H NMR (500 MHz, CDCl_3) δ 8.27 (1H, s), 7.81 (1H, d, $J = 9.5$ Hz), 7.09 (2H, d, $J = 8.5$ Hz), 6.75-6.71 (3H, m), 6.24-6.23 (1H, m), 5.95-5.90 (1H, m), 5.47-5.45 (1H, m), 5.30 (1H, d, $J = 17.5$ Hz), 5.19 (1H, d, $J = 10.5$ Hz), 4.94 (1H, d, $J = 9.5$ Hz), 4.86 (2H, d, $J = 5.0$ Hz), 4.72 (2H, d, $J = 5.5$ Hz), 3.39 (1H, t, $J = 6.5$ Hz), 3.12 (2H, d, $J = 5.5$ Hz), 1.81-1.76 (2H, m), 1.68 (3H, d, $J = 7.0$ Hz), 1.44-1.36 (3H, m), 1.31 (2H, s), 1.26 (9H, s), 1.18 (3H, d, $J = 6.5$ Hz), 1.12 (3H, d, $J = 6.5$ Hz), 1.09-0.99 (2H, m), 0.95 (9H, s), 0.91 (6H, d, $J = 6.5$ Hz), 0.83 (3H, t, $J = 7.0$ Hz), 0.77 (3H, d, $J = 6.5$ Hz), 0.16 (6H, s); ^{13}C NMR (125 MHz, CDCl_3) δ 178.35, 172.36, 170.86, 169.42, 165.91, 163.32, 156.74, 154.94, 135.41, 132.26, 130.32, 129.23, 128.09, 120.52, 118.43, 80.66, 72.00, 66.61, 62.25, 55.22, 54.30, 41.57, 39.92, 38.72, 38.22, 35.75, 30.67, 29.89, 28.22, 25.75, 20.13, 19.58, 19.11, 18.81, 18.28, 16.67, 14.38, -4.37;

HRMS (ESI) m/z : $[M + H]^+$ found for 859.5250, calcd for $C_{40}H_{75}N_6O_{12}Si$ 859.5252.



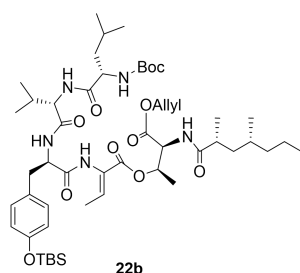
(2*R*, 3*S*)-4-(allyloxy)-3-((2*R*, 4*S*)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6*S*, 9*R*, *Z*)-9-(4-((*tert*-butyldimethylsilyloxy)benzyl)-12-ethylidene-6-isopropyl-2, 2-dimethyl-4, 7, 10-trioxo-3-oxa-5, 8, 11-triazatridecan-13-oate (21c):

White amorphous solid, 4.08 g, 90% over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); 1H NMR (500 MHz, $CDCl_3$) δ 8.22 (1H, s), 7.54 (1H, d, $J = 9.0$ Hz), 7.09 (2H, d, $J = 8.5$ Hz), 6.94 (1H, s), 6.77-6.73 (3H, m), 6.24 (1H, d, $J = 7.5$ Hz), 5.96-5.86 (1H, m), 5.47-5.43 (1H, m), 5.34-5.29 (1H, m), 5.22-5.19 (1H, m), 4.95-4.90 (1H, m), 4.83 (1H, q, $J = 7.0$ Hz), 4.71 (2H, d, $J = 5.5$ Hz), 4.65-4.58 (1H, m), 4.20-4.19 (1H, m), 3.43 (1H, t, $J = 7.5$ Hz), 3.20-3.09 (1H, m), 2.72 (1H, q, $J = 6.5$ Hz), 2.26-2.20 (1H, m), 2.06-1.81 (2H, m), 1.69 (2H, d, $J = 7.0$ Hz), 1.54-1.46 (1H, m), 1.44-1.39 (1H, m), 1.37-1.32 (3H, m), 1.30 (9H, s), 1.22 (3H, d, $J = 6.5$ Hz), 1.13 (3H, d, $J = 6.5$ Hz), 1.07 (3H, d, $J = 7.0$ Hz), 1.01 (3H, d, $J = 6.5$ Hz), 0.97 (9H, s), 0.91 (3H, t, $J = 9.0$ Hz), 0.87 (3H, d, $J = 6.5$ Hz), 0.17 (6H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.66, 172.35, 170.70, 169.57, 163.33, 156.76, 155.05, 135.40, 132.23, 130.38, 129.11, 127.99, 120.61, 120.52, 118.62, 80.81, 72.22, 66.67, 62.47, 55.32, 54.27, 41.04, 39.64, 38.35, 35.91, 30.39, 29.91, 28.36, 28.29, 25.80, 20.08, 19.68, 19.20, 19.05, 18.33, 17.98, 16.80, 14.54, 13.49, -4.31; HRMS (ESI) m/z : $[M + H]^+$ found for 859.5255, calcd for $C_{40}H_{75}N_6O_{12}Si$ 859.5252.



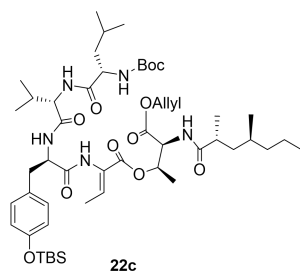
(2*R*, 3*S*)-4-(allyloxy)-3-((2*S*, 4*S*)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6*S*, 9*S*, 12*R*, *Z*)-12-(4-((*tert*-butyldimethylsilyloxy)benzyl)-15-ethylidene-6-isobutyl-9-isopropyl-2, 2-dimethyl-4, 7, 10, 13-tetraoxo-3-oxa-5, 8, 11, 14-tetraazahexadecan-16-oate (22a):

White amorphous solid, 3.26 g, 84% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); 1H NMR (500 MHz, $CDCl_3$) δ 8.05 (1H, s), 7.63 (1H, d, $J = 9.0$ Hz), 7.08 (2H, d, $J = 8.0$ Hz), 6.83 (1H, s), 6.75-6.70 (3H, m), 6.56 (1H, d, $J = 8.0$ Hz), 5.93-5.86 (1H, m), 5.47-5.46 (1H, m), 5.30 (1H, dd, $J = 1.5, 17.0$ Hz), 5.22-5.15 (1H, m), 4.94 (1H, d, $J = 2.0, 9.5$ Hz), 4.89 (1H, d, $J = 6.5$ Hz), 4.73 (1H, d, $J = 5.5$ Hz), 4.66 (2H, d, $J = 5.5$ Hz), 4.02 (1H, d, $J = 6.0$ Hz), 3.87 (1H, d, $J = 7.0$ Hz), 3.75 (1H, t, $J = 6.5$ Hz), 3.26-3.18 (1H, m), 3.10-2.99 (1H, m), 2.73 (1H, s), 2.07-2.01 (2H, m), 1.93-1.89 (2H, m), 1.81-1.76 (1H, m), 1.67-1.64 (3H, m), 1.53-1.43 (4H, m), 1.40 (9H, s), 1.35-1.30 (2H, m), 1.23 (3H, d, $J = 6.5$ Hz), 1.16 (2H, d, $J = 7.0$ Hz), 1.09-1.02 (2H, m), 0.96 (9H, s), 0.91 (2H, d, $J = 7.0$ Hz), 0.87-0.85 (8H, m), 0.84-0.83 (4H, m), 0.77 (2H, d, $J = 6.5$ Hz), 0.16 (6H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.21, 174.66, 170.74, 170.59, 169.42, 163.42, 156.51, 154.58, 134.03, 132.32, 130.60, 130.17, 127.82, 120.34, 118.53, 81.25, 72.29, 66.52, 60.28, 55.09, 54.85, 54.28, 41.78, 40.05, 38.16, 35.37, 30.48, 28.96, 28.62, 25.79, 24.78, 22.71, 22.31, 20.13, 19.69, 19.59, 19.43, 18.31, 16.90, 16.78, 14.48, 13.71, -4.34; HRMS (ESI) m/z : $[M + H]^+$ found for 972.6093, calcd for $C_{51}H_{86}N_5O_{11}Si$ 972.6093.



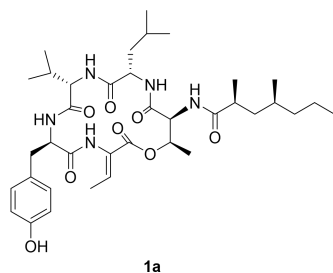
(2*R*, 3*S*)-4-(allyloxy)-3-((2*R*, 4*R*)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6*S*, 9*S*, 12*R*, *Z*)-12-(4-((*tert*-butyldimethylsilyl)oxy)benzyl)-15-ethylidene-6-isobutyl-9-isopropyl-2, 2-dimethyl-4, 7, 10, 13-tetraoxo-3-oxa-5, 8, 11, 14-tetraazahexadecan-16-oate (22b):

White amorphous solid, 3.38 g, 87% yield over two steps; R_f = 0.5 (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.09 (1H, s), 7.62 (1H, s), 7.10-7.06 (3H, m), 6.83-6.74 (4H, m), 5.95-5.87 (1H, m), 5.51 (1H, s), 5.31 (1H, d, J = 18.5 Hz), 5.21 (1H, d, J = 10.0 Hz), 5.07 (1H, s), 4.93-4.90 (1H, m), 4.74 (1H, s), 4.68-4.54 (3H, m), 4.05 (1H, s), 3.94-3.87 (1H, m), 3.76-3.68 (1H, m), 3.27-3.23 (1H, m), 3.07-3.03 (1H, m), 2.67 (1H, s), 2.27-2.22 (1H, m), 2.08-2.02 (2H, m), 1.92-1.81 (1H, m), 1.69 (3H, d, J = 7.0 Hz), 1.65 (2H, d, J = 7.0 Hz), 1.43-1.41 (6H, m), 1.40 (9H, s), 1.36-1.31 (2H, m), 1.26 (3H, d, J = 6.5 Hz), 1.14 (3H, d, J = 7.0 Hz), 1.09-1.03 (1H, m), 0.97 (9H, s), 0.95-0.91 (5H, m), 0.89-0.83 (5H, m), 0.76 (2H, d, J = 6.0 Hz), 0.17 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 178.16, 173.99, 171.75, 169.92, 163.15, 156.20, 154.89, 135.48, 131.96, 130.23, 129.41, 127.06, 120.50, 120.42, 118.90, 80.81, 71.72, 66.62, 61.01, 57.16, 55.40, 55.10, 41.60, 41.51, 39.94, 39.91, 38.79, 38.60, 30.72, 28.41, 25.80, 24.71, 22.88, 22.07, 20.13, 19.65, 19.20, 19.16, 19.09, 18.32, 17.09, 14.42, 14.08, -4.32; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 972.6092, calcd for $\text{C}_{51}\text{H}_{86}\text{N}_5\text{O}_{11}\text{Si}$ 972.6093.



(2*R*, 3*S*)-4-(allyloxy)-3-((2*R*, 4*S*)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6*S*, 9*S*, 12*R*, *Z*)-12-(4-((*tert*-butyldimethylsilyl)oxy)benzyl)-15-ethylidene-6-isobutyl-9-isopropyl-2, 2-dimethyl-4, 7, 10, 13-tetraoxo-3-oxa-5, 8, 11, 14-tetraazahexadecan-16-oate (22c):

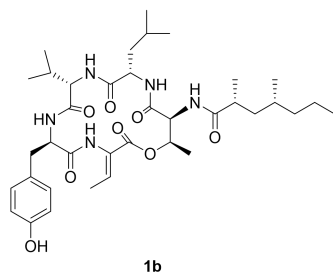
White amorphous solid, 3.19 g, 82% yield over two steps; R_f = 0.5 (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.03 (1H, s), 7.41 (1H, s), 7.09 (2H, d, J = 9.0 Hz), 6.83 (1H, s), 6.75-6.71 (3H, m), 6.61 (1H, d, J = 8.5 Hz), 5.93-5.85 (1H, m), 5.48-5.47 (1H, m), 5.30 (1H, dd, J = 1.5, 17.0 Hz), 5.20 (1H, d, J = 10.0 Hz), 5.08-5.03 (1H, m), 4.90 (1H, dd, J = 2.0, 9.0 Hz), 4.73 (1H, d, J = 5.0 Hz), 4.64 (2H, d, J = 5.5 Hz), 4.57-4.52 (1H, m), 4.11 (1H, q, J = 7.0 Hz), 4.04 (1H, s), 3.88-3.79 (1H, m), 3.26-3.22 (1H, m), 3.06-3.02 (1H, m), 2.63-2.59 (1H, m), 2.25-2.22 (1H, m), 2.07-2.01 (2H, m), 1.67 (3H, d, J = 6.5 Hz), 1.53-1.47 (2H, m), 1.43 (6H, s), 1.39 (9H, s), 1.36-1.29 (2H, m), 1.27-1.24 (4H, m), 1.13 (3H, d, J = 7.0 Hz), 0.96 (9H, s), 0.93-0.89 (4H, m), 0.88-0.81 (7H, m), 0.75 (3H, d, J = 6.5 Hz), 0.16 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 178.55, 173.80, 171.73, 170.00, 169.88, 163.13, 156.18, 154.83, 135.27, 131.89, 130.25, 129.43, 127.03, 120.44, 120.38, 118.96, 80.75, 71.87, 66.59, 60.70, 57.18, 55.44, 55.08, 41.53, 41.00, 39.62, 38.64, 30.46, 28.40, 25.79, 24.80, 24.73, 22.88, 22.10, 20.11, 19.67, 19.15, 18.30, 18.07, 17.70, 17.11, 14.51, 14.06, -4.33; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 972.6099, calcd for $\text{C}_{51}\text{H}_{86}\text{N}_5\text{O}_{11}\text{Si}$ 972.6093.



(2*S*, 4*S*)-*N*-((6*R*, 9*S*, 12*S*, 15*S*, 16*R*, *Z*)-3-ethylidene-6-(4-hydroxybenzyl)-12-isobutyl-9-isopropyl-16-methyl-, 5, 8, 11, 14-pentaoxo-1-oxa-4, 7, 10, 13-tetraazacyclohexadecan-15-yl)-2, 4-dimethylheptanamide (1a):

White solid, 164.4 mg, 47% yield over four steps; $R_f = 0.5$ ($\text{CH}_2\text{Cl}_2/\text{MeOH} = 40:1$); $[\alpha]_D^{20} -30.77$ (c 0.65, MeOH); $^1\text{H NMR}$ (500 MHz, Acetone- d_6) δ 8.50-8.46 (1H, m), 8.42 (1H, s), 8.19 (1H, s), 7.91-7.87 (1H, m), 7.38 (1H, t, $J = 7.0$ Hz), 7.22 (2H, t, $J = 8.5$ Hz), 6.76 (2H, d, $J = 8.0$ Hz), 6.61-6.55 (1H, m), 5.22-5.19 (1H, m), 4.95-4.56 (1H, m), 4.43-4.32 (1H, m), 4.22-4.20 (1H, m), 3.81-3.77 (1H, m), 3.35-3.32 (1H, m), 3.30-3.25 (1H, m), 3.03-2.94 (1H, m), 2.82 (3H, s), 2.48-2.43 (1H, m), 1.97-1.77 (1H, m), 1.70 (3H, d, $J = 7.0$ Hz), 1.69-1.64 (2H, m), 1.59-1.54 (2H, m), 1.42-1.39 (2H, m), 1.35 (3H, d, $J = 6.0$ Hz), 1.20-1.14 (2H, m), 1.06 (2H, d, $J = 7.0$ Hz), 1.05-1.01 (1H, m), 0.99-0.94 (9H, m), 0.89-0.86 (2H, m), 0.83-0.78 (6H, m), 0.75 (3H, d, $J = 7.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, Acetone- d_6) δ 177.25, 175.33, 171.37, 170.42, 170.38, 156.86, 134.61, 134.28, 131.00, 130.82, 128.57, 116.04, 74.91, 71.61, 60.67, 58.36, 57.46, 57.09, 55.56, 54.90, 42.56, 41.13, 39.94, 39.29, 36.92, 31.04, 28.79, 25.33, 23.19, 21.97, 20.61, 20.25, 19.77, 19.15, 16.37; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 700.4277, calcd for $\text{C}_{37}\text{H}_{58}\text{N}_5\text{O}_8$ 700.4285.

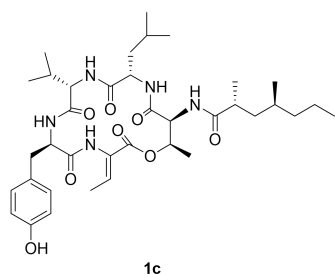
The proton signals 4.95-4.56 (1H, m) in the $^1\text{H NMR}$ spectrum of compound 1a clearly depicted two sets of signals, which is because of either rotamerism or epimerization during macrolactamization.



(2*R*, 4*R*)-*N*-((6*R*, 9*S*, 12*S*, 15*S*, 16*R*, *Z*)-3-ethylidene-6-(4-hydroxybenzyl)-12-isobutyl-9-isopropyl-16-methyl-, 2, 5, 8, 11, 14-pentaoxo-1-oxa-4, 7, 10, 13-tetraazacyclohexadecan-15-yl)-2, 4-dimethylheptanamide (1b):

White solid, 157.4 mg, 45% yield over four steps; $R_f = 0.5$ ($\text{CH}_2\text{Cl}_2/\text{MeOH} = 40:1$); $[\alpha]_D^{20} -30.7$ (c 0.65, MeOH); $^1\text{H NMR}$ (500 MHz, Acetone- d_6) δ 8.47 (1H, d, $J = 7.0$ Hz), 8.42 (1H, s), 8.20-8.19 (1H, m), 7.91-7.88 (1H, m), 7.41-7.38 (1H, m), 7.23-7.20 (2H, m), 7.08 (1H, d, $J = 9.0$ Hz), 6.76 (2H, d, $J = 8.5$ Hz), 6.61-6.56 (1H, m), 5.23-5.20 (1H, m), 4.89-4.52 (1H, m), 4.43-4.34 (1H, m), 4.22-4.18 (1H, m), 3.83-3.77 (1H, m), 3.34 (1H, t, $J = 6.5$ Hz), 3.27 (1H, dd, $J = 3.5, 14.5$ Hz), 3.03-2.94 (1H, m), 2.83 (3H, s), 2.67-2.62 (1H, m), 2.47-2.43 (1H, m), 1.89-1.83 (1H, m), 1.71 (3H, d, $J = 7.0$ Hz), 1.68-1.64 (1H, m), 1.59-1.53 (1H, m), 1.42-1.39 (1H, m), 1.37 (3H, d, $J = 6.0$ Hz), 1.24-1.16 (2H, m), 1.07 (3H, d, $J = 7.0$ Hz), 0.99-0.94 (10H, m), 0.89-0.86 (1H, m), 0.83-0.81 (4H, m), 0.80 (3H, d, $J = 4.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, Acetone- d_6) δ 177.28, 175.36, 171.44, 170.45, 169.72, 165.46, 156.88, 134.44, 130.88, 130.82, 130.42, 128.57, 116.09, 116.00, 74.56, 71.61, 60.65, 57.36, 55.96, 55.00, 42.50, 41.09, 40.28, 39.19, 36.83, 31.19, 28.79, 25.34, 23.17, 22.00, 21.77, 20.57, 19.86, 19.77, 19.27, 16.57, 14.79, 14.51; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 700.4288, calcd for $\text{C}_{37}\text{H}_{58}\text{N}_5\text{O}_8$ 700.4285.

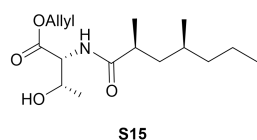
The proton signals 4.89-4.52 (1H, m) in the $^1\text{H NMR}$ spectrum of compound 1b clearly depicted two sets of signals, which is because of either rotamerism or epimerization during macrolactamization.



(2*R*, 4*S*)-*N*-((6*R*, 9*S*, 12*S*, 15*S*, 16*R*, *Z*)-3-ethylidene-6-(4-hydroxybenzyl)-12-isobutyl-9-isopropyl-16-methyl-2, 5, 8, 11, 14-pentaoxo-1-oxa-4, 7, 10, 13-tetraazacyclohexadecan-15-yl)-2, 4-dimethylheptanamide (1c):

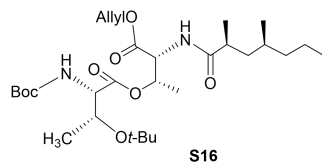
White solid, 150.4 mg, 43% yield over four steps; $R_f = 0.5$ ($\text{CH}_2\text{Cl}_2/\text{MeOH} = 40:1$); $[\alpha]_D^{20} -16.4$ (c 0.65, MeOH); $^1\text{H NMR}$ (500 MHz, Acetone- d_6) δ 8.46 (1H, d, $J = 7.5$ Hz), 8.42 (1H, s), 8.20-8.18 (1H, m), 7.90-7.88 (1H, m), 7.38 (1H, d, $J = 8.0$ Hz), 7.20 (2H, d, $J = 8.0$ Hz), 6.76 (2H, d, $J = 8.0$ Hz), 6.59 (1H, q, $J = 7.0$ Hz), 5.94 (1H, d, $J = 9.0$ Hz), 5.36-5.32 (1H, m), 5.22-5.19 (1H, m), 4.57 (1H, dd, $J = 3.0, 9.0$ Hz), 4.43-4.39 (1H, m), 4.23-4.19 (1H, m), 3.80-3.79 (2H, m), 3.35-3.30 (2H, m), 3.04-2.94 (1H, m), 2.81 (6H, s), 2.48-2.42 (1H, m), 2.14 (1H, t, $J = 7.5$ Hz), 1.96 (1H, d, $J = 8.0$ Hz), 1.89-1.77 (1H, m), 1.70 (3H, d, $J = 7.0$ Hz), 1.68-1.64 (1H, m), 1.59-1.53 (1H, m), 1.40 (3H, d, $J = 6.0$ Hz), 1.38-1.36 (1H, m), 1.05 (1H, dd, $J = 1.5, 7.0$ Hz), 0.98 (6H, t, $J = 6.0$ Hz), 0.95 (3H, d, $J = 6.5$ Hz), 0.88 (3H, t, $J = 7.0$ Hz), 0.84-0.80 (4H, m), 0.78 (2H, d, $J = 6.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, Acetone- d_6) δ 175.28, 171.39, 170.49, 169.98, 165.54, 157.83, 156.84, 134.77, 130.81, 130.58, 130.27, 128.42, 116.05, 74.69, 71.62, 60.67, 57.19, 55.05, 41.02, 30.58, 28.79, 27.77, 26.33, 25.33, 23.32, 23.13, 22.00, 21.74, 20.04, 19.75, 19.19, 16.43, 14.73, 14.30; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 700.4279, calcd for $\text{C}_{37}\text{H}_{58}\text{N}_5\text{O}_8$ 700.4285.

4.4 Data of compound 38



allyl ((2*S*, 4*S*)-2,4-dimethylheptanoyl)-*D*-threoninate (S15):

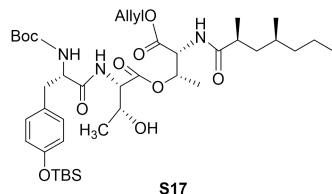
White solid, 2.45 g, 82% yield over two steps; $R_f = 0.4$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 6.38-6.36 (1H, m), 5.92-5.85 (1H, m), 5.34-5.30 (1H, m), 5.24-5.22 (1H, m), 4.64-4.60 (3H, m), 4.36 (1H, s), 3.86-2.68 (1H, m), 2.47-2.40 (1H, m), 1.74-1.68 (1H, m), 1.46-1.37 (1H, m), 1.35-1.29 (1H, m), 1.27-1.21 (2H, m), 1.19 (3H, d, $J = 6.5$ Hz), 1.14 (3H, d, $J = 7.0$ Hz), 1.12-1.05 (2H, m), 0.91 (1H, d, $J = 6.5$ Hz), 0.87 (2H, d, $J = 6.5$ Hz), 0.84 (3H, t, $J = 7.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 177.53, 171.02, 131.60, 118.94, 68.00, 66.20, 57.14, 41.79, 39.61, 39.36, 30.55, 20.05, 20.01, 19.59, 18.78, 14.36; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 300.2178, calcd for $\text{C}_{16}\text{H}_{30}\text{NO}_4$ 300.2175.



allyl *O*-(*N*-(*tert*-butoxycarbonyl)-*O*-(*tert*-butyl)-*L*-threonyl)-*N*-((2*S*, 4*S*)-2, 4-dimethylheptanoyl)-*D*-threoninate (S16):

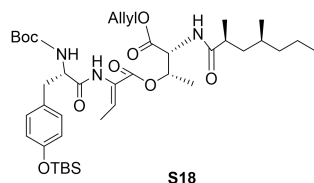
Colorless oil, 4.36 g, 98% yield; $R_f = 0.3$ (petroleum/EtOAc = 5:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 6.24 (1H, d, $J = 9.0$ Hz), 5.91-5.80 (1H, m), 5.39 (1H, dd, $J = 2.0, 6.0$ Hz), 5.30-5.19 (3H, m), 4.72 (1H, d, $J = 7.5$ Hz), 4.61-4.51 (2H, m), 4.07-4.04 (2H, m), 2.48-2.40 (1H, m), 1.75-1.68 (1H, m), 1.42 (9H, s), 1.32-1.29 (1H, m), 1.26 (3H, d, $J = 6.0$ Hz), 1.24-1.16 (3H, m), 1.14-1.10 (5H, m), 1.09 (9H, s), 1.06-1.02 (1H, m), 0.85 (3H, d, $J = 6.5$ Hz), 0.81

(3H, t, $J = 7.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 177.34, 170.42, 169.37, 155.87, 131.51, 119.00, 79.87, 74.12, 71.69, 67.32, 66.40, 59.41, 55.33, 41.47, 39.50, 39.16, 30.53, 28.56, 28.42, 20.40, 19.98, 19.65, 17.72, 17.38, 14.32; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ found for 579.3619, calcd for $\text{C}_{29}\text{H}_{52}\text{N}_2\text{O}_8\text{Na}$ 579.3621.



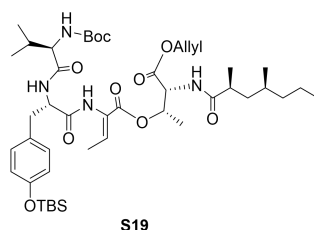
allyl O-(((S)-2-((tert-butoxycarbonyl)amino)-3-(4-((tert-butyl)dimethylsilyloxy)phenyl)propanoyl)-L-threonyl)-N-((2S, 4S)-2, 4-dimethylheptanoyl)-D-threoninate (S17):

White amorphous solid, 5.22 g, 96% yield over two steps; $R_f = 0.4$ (petroleum/EtOAc = 2:1); ^1H NMR (500 MHz, CDCl_3) δ 7.02 (2H, d, $J = 6.5$ Hz), 6.92 (1H, d, $J = 9.0$ Hz), 6.71 (2H, d, $J = 8.0$ Hz), 6.66 (1H, s), 5.90-5.82 (1H, m), 5.47-5.45 (1H, m), 5.29 (1H, d, $J = 17.0$ Hz), 5.21 (1H, d, $J = 10.5$ Hz), 5.11 (1H, s), 4.81 (1H, dd, $J = 2.0, 9.0$ Hz), 4.61-4.53 (2H, m), 4.49 (1H, d, $J = 7.0$ Hz), 4.38 (1H, s), 4.25 (1H, s), 3.24 (1H, s), 3.06-3.02 (1H, m), 2.96 (1H, s), 2.54-2.49 (1H, m), 1.76-1.70 (1H, m), 1.40 (1H, d, $J = 7.5$ Hz), 1.36 (9H, s), 1.32-1.30 (1H, m), 1.26 (3H, d, $J = 6.5$ Hz), 1.24-1.19 (2H, m), 1.13-1.08 (6H, m), 1.07-0.97 (1H, m), 0.93 (9H, s), 0.90-0.86 (4H, m), 0.83 (3H, t, $J = 7.0$ Hz), 0.14 (6H, s); ^{13}C NMR (125 MHz, CDCl_3) δ 177.56, 172.34, 170.18, 169.35, 155.60, 154.67, 131.50, 130.34, 129.07, 120.21, 119.16, 80.27, 71.89, 67.79, 66.73, 57.66, 55.78, 55.15, 41.57, 39.62, 38.99, 36.95, 30.59, 28.28, 25.70, 20.00, 19.57, 19.05, 18.87, 18.19, 17.22, 14.33, -4.42; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 778.4677, calcd for $\text{C}_{40}\text{H}_{68}\text{N}_3\text{O}_{10}\text{Si}$ 778.4674.



(2S, 3R)-4-(allyloxy)-3-(((S)-2-((tert-butoxycarbonyl)amino)-3-(4-((tert-butyl)dimethylsilyloxy)phenyl)propanamido)but-2-enoate (S18):

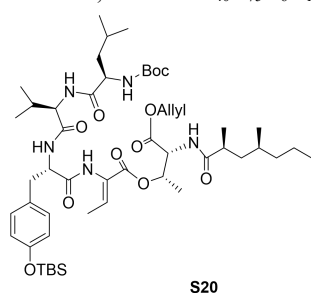
Yellow solid, 2.87 g, 86% yield; $R_f = 0.3$ (petroleum/EtOAc = 3:1); ^1H NMR (500 MHz, CDCl_3) δ 7.33 (1H, s), 7.09 (2H, d, $J = 8.5$ Hz), 6.76 (2H, d, $J = 8.0$ Hz), 6.71 (1H, q, $J = 7.0$ Hz), 6.60 (1H, s), 5.90-5.82 (1H, m), 5.42-5.40 (1H, m), 5.29 (1H, dd, $J = 1.5, 17.0$ Hz), 5.21 (1H, dd, $J = 1.0, 10.5$ Hz), 5.00 (1H, d, $J = 7.5$ Hz), 4.86 (1H, dd, $J = 3.0, 9.0$ Hz), 4.62 (2H, d, $J = 5.0$ Hz), 4.43 (1H, s), 3.10-3.05 (2H, m), 2.53 (1H, s), 1.78-1.73 (1H, m), 1.68 (3H, d, $J = 7.0$ Hz), 1.40 (9H, s), 1.35-1.31 (1H, m), 1.28 (3H, d, $J = 6.5$ Hz), 1.25-1.20 (2H, m), 1.15 (3H, d, $J = 7.0$ Hz), 1.12-1.02 (2H, m), 0.95 (9H, s), 0.90 (1H, d, $J = 6.5$ Hz), 0.86 (3H, d, $J = 6.5$ Hz), 0.83 (3H, t, $J = 7.0$ Hz), 0.16 (6H, s); ^{13}C NMR (125 MHz, CDCl_3) δ 177.54, 170.48, 169.70, 163.02, 154.84, 135.03, 131.62, 130.41, 129.09, 126.27, 120.42, 119.20, 80.59, 72.15, 66.56, 55.53, 41.56, 39.72, 39.04, 36.81, 30.65, 28.36, 25.76, 20.04, 19.66, 19.13, 19.04, 18.28, 17.02, 14.39, -4.35; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 760.4568, calcd for $\text{C}_{40}\text{H}_{66}\text{N}_3\text{O}_9\text{Si}$ 760.4568.



(2S, 3R)-4-(allyloxy)-3-(((2S, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl)-9-(4-((tert-butyl)dimethylsilyloxy)phenyl)propanamide (S19):

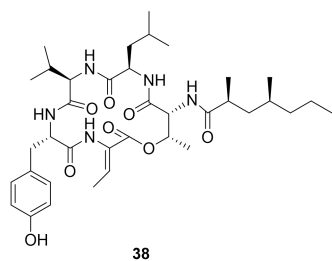
butyldimethylsilyloxy)benzyl)-12-ethylidene-6-isopropyl-2,2-dimethyl-4, 7, 10-trioxo-3-oxa-5, 8, 11-triazatridecan-13-oate (S19):

White amorphous solid, 3.78 g, 88% over two steps; $R_f = 0.4$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.23 (1H, s), 7.76 (1H, d, $J = 10.0$ Hz), 7.09 (2H, d, $J = 8.5$ Hz), 6.76 (2H, d, $J = 8.5$ Hz), 6.75-6.73 (1H, m), 6.03 (1H, d, $J = 8.5$ Hz), 5.97-5.90 (1H, m), 5.48-5.45 (1H, m), 5.32 (1H, dd, $J = 1.5, 17.0$ Hz), 5.21 (1H, dd, $J = 1.0, 10.5$ Hz), 4.95 (1H, dd, $J = 2.0, 9.5$ Hz), 4.89-4.84 (2H, m), 4.73 (2H, d, $J = 5.5$ Hz), 3.37 (1H, t, $J = 6.5$ Hz), 3.20-3.16 (1H, m), 3.11-3.08 (1H, m), 2.83-2.80 (1H, m), 1.86-1.78 (2H, m), 1.69 (3H, d, $J = 7.0$ Hz), 1.45-1.41 (2H, m), 1.33 (2H, s), 1.28 (9H, s), 1.25-1.22 (1H, m), 1.19 (3H, d, $J = 6.0$ Hz), 1.14 (3H, d, $J = 7.0$ Hz), 1.10-1.00 (2H, m), 0.97 (9H, s), 0.94-0.92 (5H, m), 0.85 (3H, t, $J = 7.0$ Hz), 0.80 (3H, d, $J = 6.5$ Hz), 0.17 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 178.29, 172.31, 170.77, 169.51, 163.34, 156.79, 155.05, 135.49, 132.29, 130.38, 129.10, 128.08, 120.62, 118.49, 80.80, 72.11, 66.66, 62.39, 55.26, 54.23, 41.63, 39.97, 38.31, 35.84, 30.72, 29.91, 28.27, 25.80, 20.18, 19.66, 19.25, 19.15, 18.89, 18.33, 16.72, 14.43, 13.42, -4.30; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 859.5245, calcd for $\text{C}_{40}\text{H}_{75}\text{N}_6\text{O}_{12}\text{Si}$ 859.5252.



(2S, 3R)-4-(allyloxy)-3-((2S, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6R, 9R, 12S, Z)-12-(4-((tert-butylidimethylsilyloxy)benzyl)-15-ethylidene-6-isobutyl-9-isopropyl-2, 2-dimethyl-4, 7, 10, 13-tetraoxo-3-oxa-5, 8, 11, 14-tetraazahexadecan-16-oate (S20):

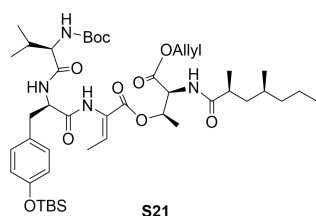
White amorphous solid, 3.30 g, 85% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.08 (1H, s), 7.58 (1H, d, $J = 10.0$ Hz), 7.08 (2H, d, $J = 8.0$ Hz), 6.78 (1H, s), 6.75-6.71 (3H, m), 6.50 (1H, d, $J = 7.5$ Hz), 5.93-5.86 (1H, m), 5.50 (1H, d, $J = 4.5$ Hz), 5.30 (1H, dd, $J = 1.5, 17.0$ Hz), 5.20 (1H, d, $J = 10.0$ Hz), 5.05 (1H, d, $J = 7.0$ Hz), 4.91 (1H, dd, $J = 2.0, 9.5$ Hz), 4.73 (1H, d, $J = 5.0$ Hz), 4.65 (2H, d, $J = 5.5$ Hz), 4.03 (1H, d, $J = 7.0$ Hz), 3.74 (1H, t, $J = 6.5$ Hz), 3.24 (1H, dd, $J = 5.0, 14.0$ Hz), 3.06-3.02 (1H, m), 2.65 (1H, s), 2.03 (2H, s), 1.83-1.77 (1H, m), 1.68 (3H, d, $J = 7.0$ Hz), 1.57-1.51 (1H, m), 1.48-1.43 (2H, m), 1.38 (9H, s), 1.36-1.30 (2H, m), 1.24 (3H, d, $J = 6.0$ Hz), 1.23 (1H, s), 1.13 (3H, d, $J = 7.0$ Hz), 1.09-1.02 (2H, m), 0.96 (9H, s), 0.92-0.90 (4H, m), 0.87-0.82 (11H, m), 0.75 (3H, d, $J = 6.0$ Hz), 0.15 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 177.97, 173.87, 171.67, 169.88, 169.84, 163.16, 156.16, 154.84, 135.21, 131.99, 130.22, 129.48, 127.17, 120.44, 118.81, 80.69, 71.71, 66.55, 60.94, 55.35, 55.07, 52.99, 41.53, 40.27, 39.92, 38.57, 35.92, 30.70, 29.63, 28.39, 25.78, 24.69, 22.87, 22.07, 20.10, 19.63, 19.17, 19.07, 18.50, 18.29, 17.06, 14.40, 14.00, -4.34; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 972.6094, calcd for $\text{C}_{51}\text{H}_{86}\text{N}_5\text{O}_{11}\text{Si}$ 972.6093.



(2S, 4S)-N-((6S, 9R, 12R, 15R, 16S, Z)-3-ethylidene-6-(4-hydroxybenzyl)-12-isobutyl-9-isopropyl-16-methyl-2, 5, 8, 11, 14-pentaoxo-1-oxa-4, 7, 10, 13-tetraazacyclohexadecan-15-yl)-2, 4-dimethylheptanamide (38):

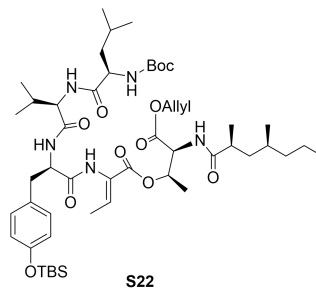
White solid, 139.9 mg, 40% yield over four steps; $R_f = 0.4$ ($\text{CH}_2\text{Cl}_2/\text{MeOH} = 40:1$); $[\alpha]_D^{20} +17.95$ (c 0.65, MeOH); $^1\text{H NMR}$ (500 MHz, Acetone- d_6) δ 8.46 (1H, d, $J = 7.0$ Hz), 8.42 (1H, s), 8.20 (1H, br), 7.89 (1H, s), 7.40 (1H, d, $J = 7.5$ Hz), 7.22 (2H, d, $J = 8.5$ Hz), 7.06 (1H, d, $J = 9.0$ Hz), 6.76 (2H, d, $J = 8.5$ Hz), 6.58 (1H, q, $J = 7.0$ Hz), 5.35 (1H, t, $J = 4.5$ Hz), 5.23-5.21 (1H, m), 4.88 (1H, dd, $J = 3.0, 9.0$ Hz), 4.40-4.35 (1H, m), 4.22-4.18 (1H, m), 3.35 (1H, t, $J = 6.5$ Hz), 3.28 (1H, dd, $J = 3.0, 14.0$ Hz), 3.00 (1H, t, $J = 13.0$ Hz), 2.67-2.62 (1H, m), 2.48-2.41 (1H, m), 2.15 (1H, t, $J = 7.0$ Hz), 1.92-1.85 (1H, m), 1.71 (3H, d, $J = 7.0$ Hz), 1.68-1.64 (1H, m), 1.59-1.54 (2H, m), 1.37 (3H, d, $J = 6.0$ Hz), 1.23-1.17 (2H, m), 1.06 (3H, d, $J = 7.0$ Hz), 1.04-1.01 (1H, m), 0.98 (6H, t, $J = 6.5$ Hz), 0.95 (3H, d, $J = 6.5$ Hz), 0.92-0.87 (4H, m), 0.82 (3H, t, $J = 4.0$ Hz), 0.80 (3H, d, $J = 4.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, Acetone- d_6) δ 177.29, 175.36, 171.46, 170.47, 169.78, 165.46, 156.90, 134.48, 130.88, 130.59, 128.59, 116.10, 74.56, 60.67, 57.34, 55.97, 55.03, 42.52, 41.12, 40.30, 39.23, 36.84, 31.21, 27.79, 25.35, 23.18, 22.02, 21.77, 20.58, 19.87, 19.77, 19.27, 16.57, 14.77, 14.51; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 700.4280, calcd for $\text{C}_{37}\text{H}_{58}\text{N}_5\text{O}_8$ 700.4285.

4.5 Data of compound 39



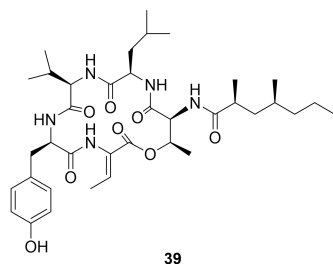
(2R, 3S)-4-(allyloxy)-3-((2S, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6R, 9R, Z)-9-(4-((tert-butyl)dimethylsilyloxy)benzyl)-12-ethylidene-6-isopropyl-2, 2-dimethyl-4, 7, 10-trioxo-3-oxa-5, 8, 11-triazatridecan-13-oate (S21):

White amorphous solid, 3.95 g, 92% over two steps; $R_f = 0.4$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.78 (1H, d, $J = 9.0$ Hz), 7.68 (1H, s), 7.06 (2H, d, $J = 8.5$ Hz), 6.81 (1H, q, $J = 7.0$ Hz), 6.75 (2H, d, $J = 8.5$ Hz), 6.45 (1H, d, $J = 7.5$ Hz), 5.93-5.85 (1H, m), 5.52-5.51 (1H, m), 5.30 (1H, dd, $J = 1.5, 17.0$ Hz), 5.19 (1H, dd, $J = 1.0, 10.5$ Hz), 4.97 (1H, dd, $J = 2.5, 9.5$ Hz), 4.79-4.76 (2H, m), 4.64 (2H, d, $J = 9.5$ Hz), 3.79 (1H, t, $J = 4.5$ Hz), 3.27-3.25 (1H, m), 3.01-2.99 (1H, m), 2.82 (1H, s), 2.17-2.11 (1H, m), 1.83-1.77 (1H, m), 1.62 (3H, d, $J = 7.0$ Hz), 1.43-1.35 (2H, m), 1.31 (9H, s), 1.29-1.26 (2H, m), 1.24 (3H, d, $J = 6.5$ Hz), 1.22-1.19 (1H, m), 1.16 (3H, d, $J = 7.0$ Hz), 1.09-1.01 (2H, m), 0.96 (9H, s), 0.94 (3H, d, $J = 7.0$ Hz), 0.89-0.87 (5H, m), 0.84 (3H, t, $J = 7.5$ Hz), 0.16 (6H, d, $J = 3.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 178.10, 171.33, 169.79, 169.46, 163.13, 156.70, 155.09, 136.00, 132.07, 130.36, 128.73, 126.89, 120.58, 118.73, 81.32, 72.30, 66.45, 61.58, 55.22, 53.82, 41.72, 39.98, 38.25, 35.98, 30.51, 29.98, 28.20, 25.74, 20.10, 19.67, 19.62, 19.34, 18.26, 17.73, 16.60, 14.48, 14.10, -4.33; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 859.5251, calcd for $\text{C}_{40}\text{H}_{75}\text{N}_6\text{O}_{12}\text{Si}$ 859.5252.



(2R, 3S)-4-(allyloxy)-3-((2S, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6R, 9R, 12R, Z)-12-(4-((tert-butyl)dimethylsilyloxy)benzyl)-15-ethylidene-6-isobutyl-9-isopropyl-2, 2-dimethyl-4, 7, 10, 13-tetraoxo-3-oxa-5, 8, 11, 14-tetraazahexadecan-16-oate (S22):

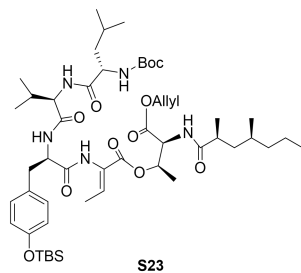
White amorphous solid, 3.61 g, 93% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.07 (1H, s), 7.80 (1H, d, $J = 9.0$ Hz), 7.09 (2H, d, $J = 8.0$ Hz), 6.88 (1H, d, $J = 8.0$ Hz), 6.76-6.72 (2H, m), 6.70 (2H, d, $J = 8.0$ Hz), 5.94-5.86 (1H, m), 5.51 (1H, q, $J = 6.0$ Hz), 5.28 (1H, d, $J = 17.5$ Hz), 5.15 (1H, d, $J = 10.5$ Hz), 5.05 (1H, s), 4.96 (2H, d, $J = 9.0$ Hz), 4.72-4.63 (2H, m), 3.99 (1H, t, $J = 4.0$ Hz), 3.90-3.86 (1H, m), 3.38 (1H, d, $J = 12.0$ Hz), 2.94-2.89 (2H, m), 2.15-2.11 (1H, m), 2.05-2.02 (1H, m), 1.82-1.77 (1H, m), 1.73 (3H, d, $J = 7.0$ Hz), 1.68-1.63 (1H, m), 1.57-1.52 (1H, m), 1.50-1.43 (1H, m), 1.42 (9H, s), 1.34-1.28 (2H, m), 1.23 (3H, d, $J = 6.5$ Hz), 1.15 (3H, d, $J = 6.5$ Hz), 1.08-0.99 (2H, m), 0.95 (9H, s), 0.92 (3H, d, $J = 6.5$ Hz), 0.89 (3H, d, $J = 6.0$ Hz), 0.87 (3H, d, $J = 6.5$ Hz), 0.84-0.81 (3H, m), 0.76 (3H, d, $J = 7.0$ Hz), 0.13 (6H, s); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 178.24, 174.53, 170.80, 170.75, 169.37, 163.42, 156.75, 154.50, 134.82, 132.28, 130.05, 129.75, 127.78, 120.24, 118.45, 81.50, 72.16, 66.51, 60.68, 55.13, 54.48, 53.96, 41.74, 39.98, 38.08, 35.69, 30.48, 29.28, 28.37, 25.77, 24.89, 22.95, 21.69, 20.09, 19.65, 19.61, 19.19, 18.30, 17.54, 16.77, 14.45, 13.64, -4.37; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 972.6094, calcd for $\text{C}_{51}\text{H}_{86}\text{N}_5\text{O}_{11}\text{Si}$ 972.6093.



(2S, 4S)-N-((6R, 9R, 12R, 15S, 16R, Z)-3-ethylidene-6-(4-hydroxybenzyl)-12-isobutyl-9-isopropyl-16-methyl-2, 5, 8, 11, 14-pentaoxo-1-oxa-4, 7, 10, 13-tetraazacyclohexadecan-15-yl)-2, 4-dimethylheptanamide (39):

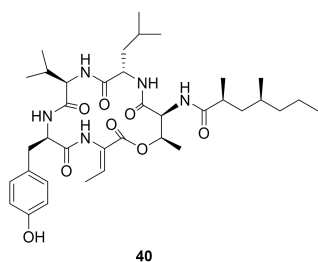
White solid, 160.9 mg, 46% yield over four steps; $R_f = 0.4$ ($\text{CH}_2\text{Cl}_2/\text{MeOH} = 40:1$); $[\alpha]_D^{20} +6.8$ (c 0.65, MeOH); $^1\text{H NMR}$ (500 MHz, Acetone- d_6) δ 8.14 (1H, d, $J = 25.0$ Hz), 8.07 (1H, s), 7.79 (1H, s), 7.48 (1H, s), 7.20-7.18 (3H, m), 6.76 (2H, d, $J = 8.0$ Hz), 6.61 (1H, q, $J = 7.0$ Hz), 5.23 (1H, s), 4.77 (1H, s), 4.48 (1H, s), 4.05 (1H, s), 3.86 (1H, s), 3.33 (1H, dd, $J = 3.5, 14.0$ Hz), 3.00 (1H, t, $J = 13.0$ Hz), 2.70 (2H, q, $J = 7.0$ Hz), 2.03-2.01 (2H, m), 1.76-1.76 (2H, m), 1.66 (3H, d, $J = 7.0$ Hz), 1.61-1.49 (1H, m), 1.42-1.37 (1H, m), 1.33 (3H, d, $J = 6.0$ Hz), 1.25-1.19 (2H, m), 1.15 (3H, d, $J = 6.5$ Hz), 1.03 (1H, s), 0.95 (3H, d, $J = 6.5$ Hz), 0.92 (3H, d, $J = 6.5$ Hz), 0.90-0.87 (4H, m), 0.84 (3H, t, $J = 7.0$ Hz), 0.72 (3H, d, $J = 6.5$ Hz), 0.67 (3H, d, $J = 6.5$ Hz); $^{13}\text{C NMR}$ (125 MHz, Acetone- d_6) δ 173.96, 171.40, 170.40, 169.25, 165.10, 156.99, 133.93, 130.94, 130.10, 128.26, 116.04, 74.00, 62.02, 56.87, 56.12, 54.36, 42.67, 40.06, 39.49, 36.73, 32.63, 31.12, 25.72, 23.80, 21.31, 20.69, 20.31, 19.35, 19.10, 18.46, 16.72, 14.88, 14.56; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 700.4282, calcd for $\text{C}_{37}\text{H}_{58}\text{N}_5\text{O}_8$ 700.4285.

4.6 Data of compound 40



(2R, 3S)-4-(allyloxy)-3-((2S, 4S)-2, 4-dimethylheptanamido)-4-oxobutan-2-yl (6S, 9R, 12R, Z)-12-(4-((tert-butyl dimethylsilyloxy)benzyl)-15-ethylidene-6-isobutyl-9-isopropyl-2, 2-dimethyl-4, 7, 10, 13-tetraoxo-3-oxa-5, 8, 11, 14-tetraazahexadecan-16-oate (S23):

White amorphous solid, 3.61 g, 93% yield over two steps; $R_f = 0.5$ (petroleum/EtOAc = 2:1); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.95 (1H, s), 7.84 (1H, d, $J = 9.5$ Hz), 7.16 (2H, d, $J = 8.0$ Hz), 7.04 (1H, d, $J = 6.5$ Hz), 6.75 (2H, d, $J = 8.0$ Hz), 6.70 (1H, q, $J = 7.0$ Hz), 6.34 (1H, s), 5.96-5.88 (1H, m), 5.53-5.50 (1H, m), 5.30 (1H, dd, $J = 1.5, 17.0$ Hz), 5.19 (1H, dd, $J = 1.0, 10.5$ Hz), 4.98 (1H, dd, $J = 2.5, 9.5$ Hz), 4.89 (1H, s), 4.84-4.80 (1H, m), 4.70 (2H, d, $J = 5.5$ Hz), 4.06-4.04 (1H, m), 3.88 (1H, s), 3.33 (1H, d, $J = 12.0$ Hz), 2.97 (1H, t, $J = 11.0$ Hz), 2.85-2.81 (1H, m), 2.26-2.23 (1H, m), 1.83 (1H, s), 1.81-1.77 (1H, m), 1.74 (3H, d, $J = 7.5$ Hz), 1.63-1.56 (2H, m), 1.45 (9H, s), 1.36-1.26 (3H, m), 1.24 (3H, d, $J = 6.0$ Hz), 1.21-1.18 (1H, m), 1.15 (3H, d, $J = 7.0$ Hz), 1.09-0.99 (2H, m), 0.97 (9H, s), 0.96-0.95 (3H, m), 0.91 (3H, d, $J = 6.0$ Hz), 0.87 (3H, d, $J = 7.0$ Hz), 0.84 (6H, t, $J = 7.0$ Hz), 0.63 (3H, d, $J = 6.5$ Hz), 0.15 (6H, d, $J = 2.5$ Hz); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 178.21, 174.66, 170.74, 170.59, 169.42, 163.42, 156.51, 154.58, 134.03, 132.32, 130.60, 130.17, 127.82, 120.34, 118.53, 81.25, 72.29, 66.52, 60.28, 55.09, 54.85, 54.28, 41.78, 40.05, 38.16, 35.37, 30.48, 28.96, 28.62, 25.79, 24.78, 22.71, 22.31, 20.13, 19.69, 19.59, 19.43, 18.31, 16.90, 16.78, 14.48, 13.71, -4.34; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 972.6090, calcd for $\text{C}_{51}\text{H}_{86}\text{N}_5\text{O}_{11}\text{Si}$ 972.6093.



(2S, 4S)-N-((6R, 9R, 12S, 15S, 16R,Z)-3-ethylidene-6-(4-hydroxybenzyl)-12-isobutyl-9-isopropyl-16-methyl-2, 5, 8, 11, 14-pentaoxo-1-oxa-4, 7, 10, 13-tetraazacyclohexadecan-15-yl)-2, 4-dimethylheptanamide (40):

White solid, 160.9 mg, 46% yield over four steps; $R_f = 0.4$ ($\text{CH}_2\text{Cl}_2/\text{MeOH} = 40:1$); $[\alpha]_D^{20} +29.18$ (c 0.65, MeOH); $^1\text{H NMR}$ (500 MHz, Acetone- d_6) δ 8.14 (1H, br), 8.03-8.00 (2H, m), 7.94 (1H, s), 7.48 (1H, d, $J = 9.0$ Hz), 7.20 (2H, d, $J = 8.5$ Hz), 7.01 (1H, d, $J = 9.0$ Hz), 6.73 (2H, d, $J = 8.5$ Hz), 6.59 (1H, q, $J = 7.0$ Hz), 5.18-5.13 (1H, m), 4.94 (1H, dd, $J = 3.0, 9.0$ Hz), 4.48-4.43 (1H, m), 4.33-4.30 (1H, m), 3.86 (1H, t, $J = 4.5$ Hz), 3.31 (1H, dd, $J = 3.5, 14.0$ Hz), 3.01 (1H, t, $J = 13.5$ Hz), 2.67-2.63 (1H, m), 2.16-2.13 (1H, m), 1.78-1.71 (1H, m), 1.68 (3H, d, $J = 7.0$ Hz), 1.63-1.52 (3H, m), 1.42-1.37 (1H, m), 1.35 (3H, d, $J = 6.5$ Hz), 1.21-1.14 (3H, m), 1.06 (3H, d, $J = 7.0$ Hz), 1.04-1.02 (1H, m), 0.98 (3H, d, $J = 6.5$ Hz), 0.96-0.92 (1H, m), 0.89 (3H, d, $J = 6.5$ Hz), 0.86 (3H, d, $J = 7.0$ Hz), 0.79 (3H, t, $J = 7.0$ Hz), 0.76 (3H, d, $J = 6.5$ Hz), 0.60 (3H, d, $J = 7.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, Acetone- d_6) δ 177.27, 176.66, 171.43, 171.27, 169.47, 165.33, 156.89, 134.75, 130.98, 130.34, 128.46, 115.93, 75.21, 61.81, 56.74, 55.59, 54.51, 42.51, 40.82, 40.00, 39.24, 36.69, 31.06, 29.38, 25.25, 23.00, 22.59, 20.63, 20.28, 19.26, 19.17, 17.17, 16.32, 14.75, 14.62; $^1\text{H NMR}$ (500 MHz, CD_3OD) δ 7.19 (2H, d, $J = 8.5$ Hz), 6.80 (1H, q, $J = 7.0$ Hz), 6.70 (2H, d, $J = 8.5$ Hz), 5.18-5.13 (1H, m), 4.72 (1H, d, $J = 3.0$ Hz), 4.49 (1H, dd, $J = 3.5, 13.0$ Hz), 4.32 (1H, t, $J = 7.5$ Hz), 3.83 (1H, d, $J = 4.0$ Hz), 3.39-3.35 (1H, m), 3.09 (1H, t, $J = 13.0$ Hz), 2.64-2.58 (1H, m), 2.10-2.07 (1H, m), 1.74 (3H, d, $J = 7.0$ Hz), 1.69-1.64 (2H, m), 1.61-1.46 (2H, m), 1.43 (3H, d, $J = 6.0$ Hz), 1.32 (1H, s), 1.24-1.14 (2H, m), 1.10 (3H, d, $J = 7.0$ Hz), 1.07-1.03 (1H, m), 1.01 (3H, d, $J = 6.5$ Hz), 0.95 (3H, d, $J = 6.5$ Hz), 0.92-0.85 (2H, m), 0.83-0.79 (6H, m), 0.70 (3H, d, $J = 6.5$ Hz), 0.61 (3H, d, $J = 7.0$ Hz); $^{13}\text{C NMR}$ (125 MHz, CD_3OD) δ 180.25, 178.57, 173.80, 172.08, 171.50, 165.43, 157.38, 138.10, 131.45, 129.91, 127.63, 116.35, 75.55, 62.42, 57.29, 56.84, 54.55, 42.51, 41.02, 40.33, 39.56, 36.72, 31.51, 29.99, 25.81, 22.99, 22.81, 20.99, 20.36, 19.27, 19.21, 17.28, 16.33, 14.66, 14.55; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ found for 700.4282, calcd for $\text{C}_{37}\text{H}_{58}\text{N}_5\text{O}_8$ 700.4285.

5. NMR Comparison Tables of Tumescenamide A

Table S3. ¹H NMR comparison for Tumescenamide A

Natural Tumescenamide A (500 MHz, acetone-d ₆)	Synthetic Tumescenamide A (compound 40) (500 MHz, acetone-d ₆)
8.20, br	8.14, br
8.02, d (J = 3.9 Hz)	8.02, m
7.98, s	
7.92, d (J = 2.0 Hz)	7.94, s
7.48, d (J = 8.6 Hz)	7.48, d (J = 8.6 Hz)
7.19, d (J = 8.4 Hz)	7.20, d (J = 8.4 Hz)
7.04, d (J = 9.0 Hz)	7.01, d (J = 9.0 Hz)
6.71, d (J = 8.4 Hz)	6.73, d (J = 8.4 Hz)
6.57, q (J = 7.1 Hz)	6.58, q (J = 7.1 Hz)
5.15, dq (J = 3.2, 6.1 Hz)	5.15, dq (J = 3.1, 6.1 Hz)
4.92, dd (J = 3.2, 9.0 Hz)	4.93, dd (J = 3.0, 9.0 Hz)
4.45, m	4.45, m
4.29, dd (J = 7.6, 7.6 Hz)	4.30, dd (J = 7.6, 7.6 Hz)
3.85, dd (J = 3.9, 3.9 Hz)	3.85, dd (J = 4.0, 5.1 Hz)
3.29, dd (J = 3.4, 13.9 Hz)	3.30, dd (J = 3.4, 14.0 Hz)
2.99, dd (J = 13.7, 13.7 Hz)	3.00, t (J = 13.6 Hz)
2.65, m	2.65, m
2.12, m	2.13, m
1.72, m	1.72, m
1.67, d (J = 7.1 Hz)	1.68, d (J = 7.0 Hz)
1.67, m	1.58, m
1.58, m	
1.52, m	
1.38, m	1.38, m
1.34, d (J = 6.4 Hz)	1.35, d (J = 6.3 Hz)
1.20, m	1.18, m
1.18, m	
1.14, m	
1.04, d (J = 6.8 Hz)	1.06, d (J = 6.9 Hz)
1.02, m	1.02, m
0.98, d (J = 6.6 Hz)	0.98, d (J = 6.6 Hz)
0.92, m	0.92, m
0.88, d (J = 7.1 Hz)	0.89, d (J = 6.6 Hz)
0.86, d (J = 7.1 Hz)	0.86, d (J = 7.1 Hz)
0.78, t (J = 7.1 Hz)	0.79, t (J = 7.1 Hz)
0.76, d (J = 6.6 Hz)	0.76, d (J = 6.6 Hz)
0.58, d (J = 7.1 Hz)	0.60, d (J = 7.1 Hz)

Table S4. ¹³C NMR comparison for Tumescenamide A

Natural Tumescenamide A (125 MHz, CD ₃ OD)	Synthetic Tumescenamide A (compound 40) (125 MHz, CD ₃ OD)
177.3	177.3
176.6	176.6
171.3	171.3
171.2	171.2
169.5	169.5
165.3	165.2
156.8	156.8
134.8	134.8
130.9	130.9
130.0	130.2
128.0	128.4
115.8	115.9
75.1	75.1
61.7	61.7
56.7	56.7
55.5	55.5
54.4	54.4
42.7	42.4
40.7	40.7
39.9	39.9
39.1	39.2
36.6	36.6
31.0	31.0
29.2	29.3
25.2	25.2
22.9	22.9
22.5	22.5
20.6	20.6
20.2	20.2
19.2	19.2
19.1	19.1
17.0	17.1
16.2	16.2
14.7	14.7
14.6	14.5

6. NMR Comparison Tables of Tumescenamide C

Table S5. ¹H NMR comparison for Tumescenamide C

Reported Tumescenamide C ⁷ (500 MHz, CD ₃ OD)	Synthetic Tumescenamide C (compound 40) (500 MHz, CD ₃ OD)
7.18, d (J = 8.5 Hz)	7.19, d (J = 8.5 Hz)
6.79, q (J = 7.0 Hz)	6.80, q (J = 7.0 Hz)
6.69, d (J = 8.5 Hz)	6.70, d (J = 8.5 Hz)
5.15, dq (J = 3.5, 6.5 Hz)	5.18-5.13, m
4.72, dd (J = 3.0, 8.5 Hz)	4.72, d (J = 3.0 Hz)
4.48, dd (J = 3.5, 12.5 Hz)	4.49, dd (J = 3.5, 13.0 Hz)
4.31, dd (J = 7.5, 8.0 Hz)	4.32, t (J = 7.5 Hz)
3.82, d (J = 4.0 Hz)	3.83, d (J = 4.0 Hz)
3.37, m	3.39-3.35, m
3.20, m	
3.08, dd (J = 13.0, 13.5 Hz)	3.09, t (J = 13.0 Hz)
2.61, m	2.64-2.58, m
2.08, m	2.10-2.07, m
1.73, d (J = 6.5 Hz)	1.74, d (J = 7.0 Hz)
1.65, m	1.69-1.64, m
1.60-1.47, overlapped	1.61-1.46, m
1.42, d (J = 9.0 Hz)	1.43, d (J = 6.0 Hz)
1.32, m	1.32, s
1.24-1.10, overlapped	1.24-1.14, m
1.09, d (J = 7.0 Hz)	1.10, d (J = 7.0 Hz)
1.05, m	1.07-1.03, m
1.00, d (J = 6.0 Hz)	1.01, d (J = 6.5 Hz)
0.94, d (J = 6.5 Hz)	0.95, d (J = 6.5 Hz)
0.88, m	0.92-0.85, m
0.81, d (J = 7.0 Hz)	0.83-0.79, m
0.79, t (J = 7.0 Hz)	
0.69, d (J = 7.0 Hz)	0.70, d (J = 6.5 Hz)
0.60, d (J = 7.0 Hz)	0.61, d (J = 7.0 Hz)

Table S6. ¹³C NMR comparison for Tumescenamide C

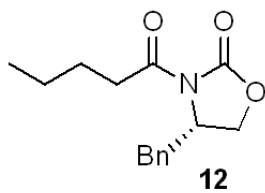
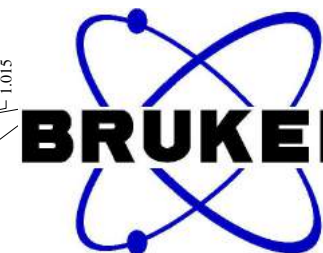
Reported Tumescenamide C ⁷ (125 MHz, CD ₃ OD)	Synthetic Tumescenamide C (compound 40) (125 MHz, CD ₃ OD)
180.3	180.3
178.6	178.6
173.8	173.8
172.1	172.1
171.5	171.5
165.4	165.4
157.4	157.4
138.1	138.1
131.5	131.5
129.9	129.9
127.6	127.6
116.3	116.4
75.5	75.6
62.4	62.4
57.3	57.3
56.9	56.8
54.5	54.6
42.5	42.5
41.0	41.0
40.3	40.3
39.5	39.6
36.7	36.7
31.5	31.5
30.0	30.0
25.8	25.8
23.0	23.0
22.8	22.8
21.0	21.0
20.4	20.4
19.3	19.3
19.2	19.2
17.2	17.3
16.3	16.3
14.7	14.7
14.6	14.6

7. NMR Spectra of Products

liushouxin-XH

7.405
7.391
7.376
7.346
7.331
7.321
7.317
7.274
7.260

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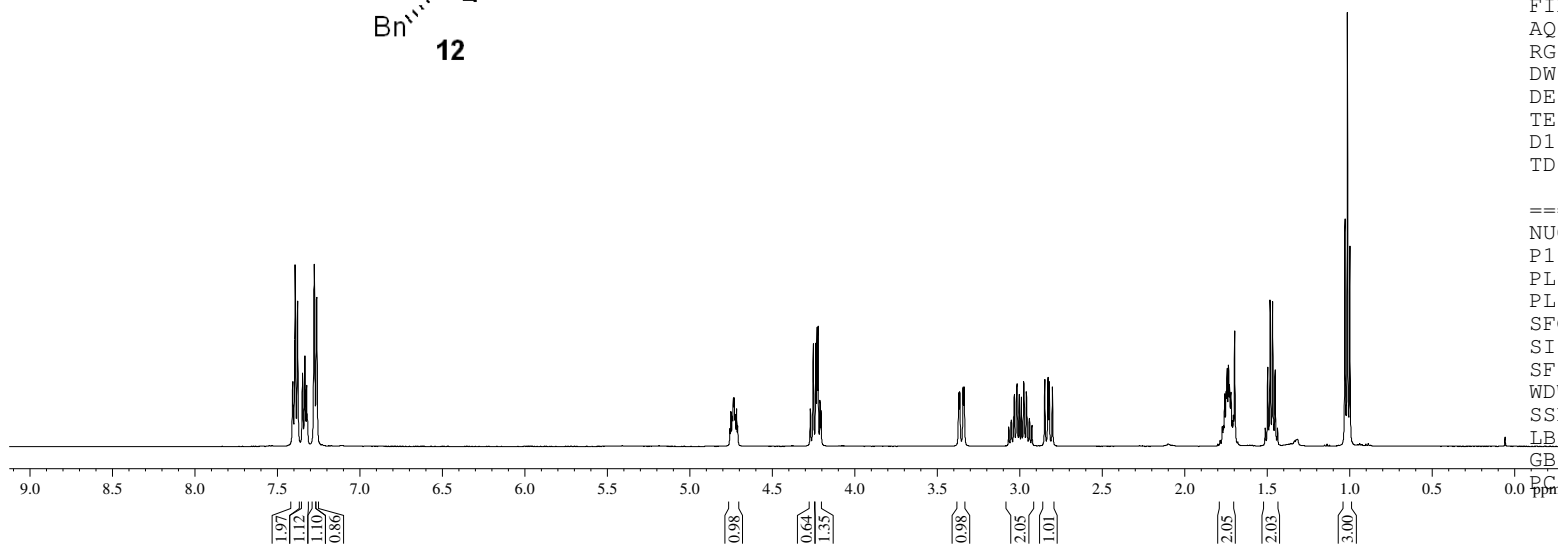


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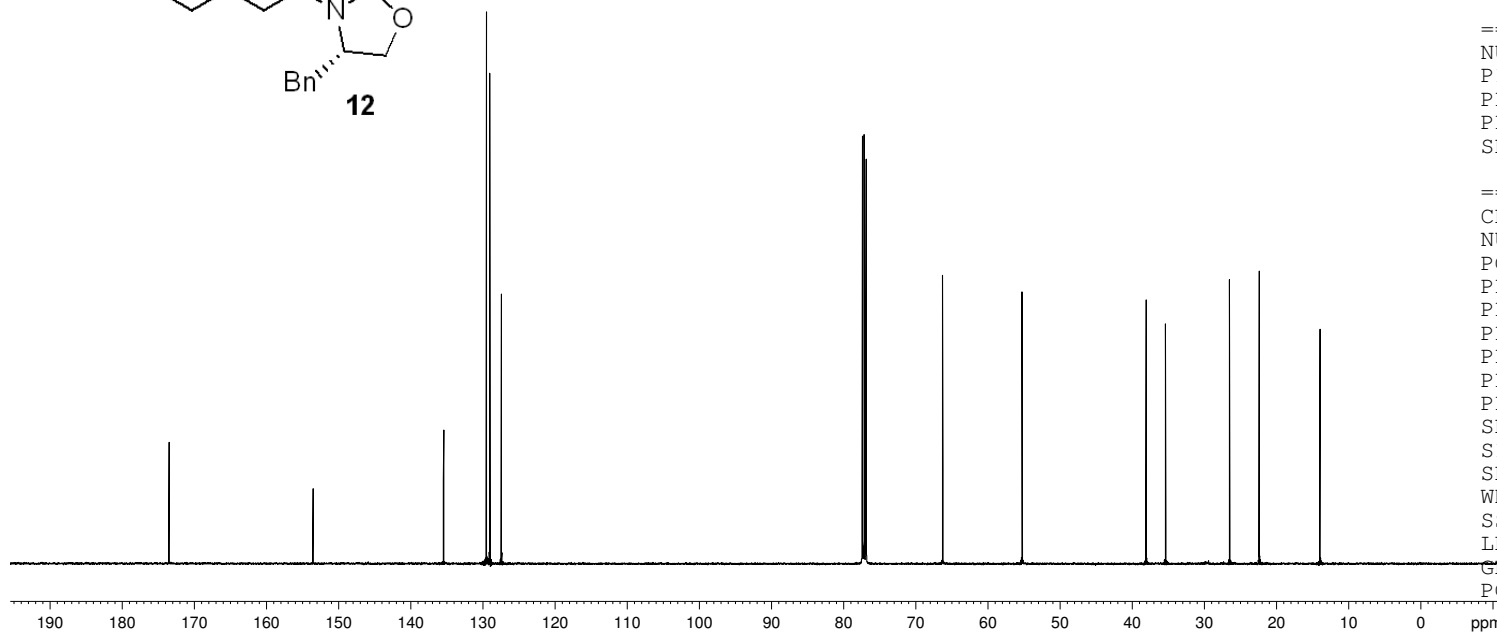
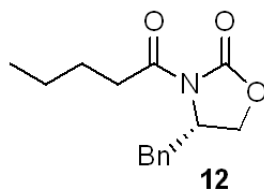
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— 135.48
— 129.54
— 129.06
— 127.45

77.41
77.16
76.91
— 66.27
— 55.27
— 38.07
— 35.37
— 26.49
— 22.38
— 13.96



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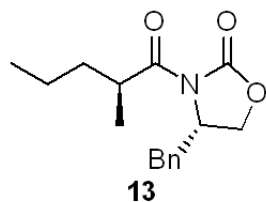
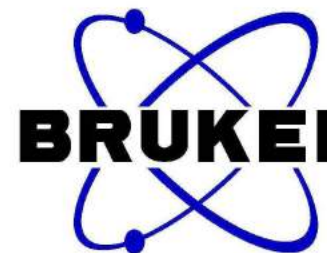
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PL13W 0.47786582 W
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liushouxin-xh-Me

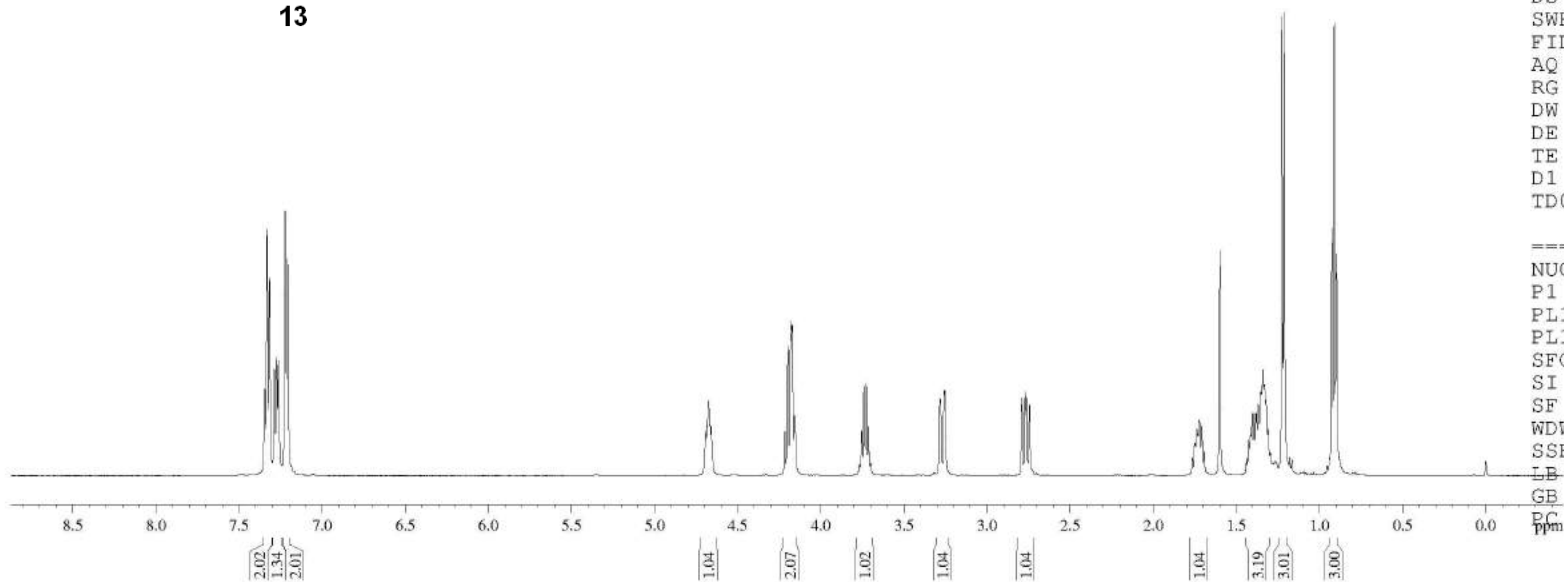
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liushouxin-xh-Me

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— 135.51

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— 129.06

— 127.46

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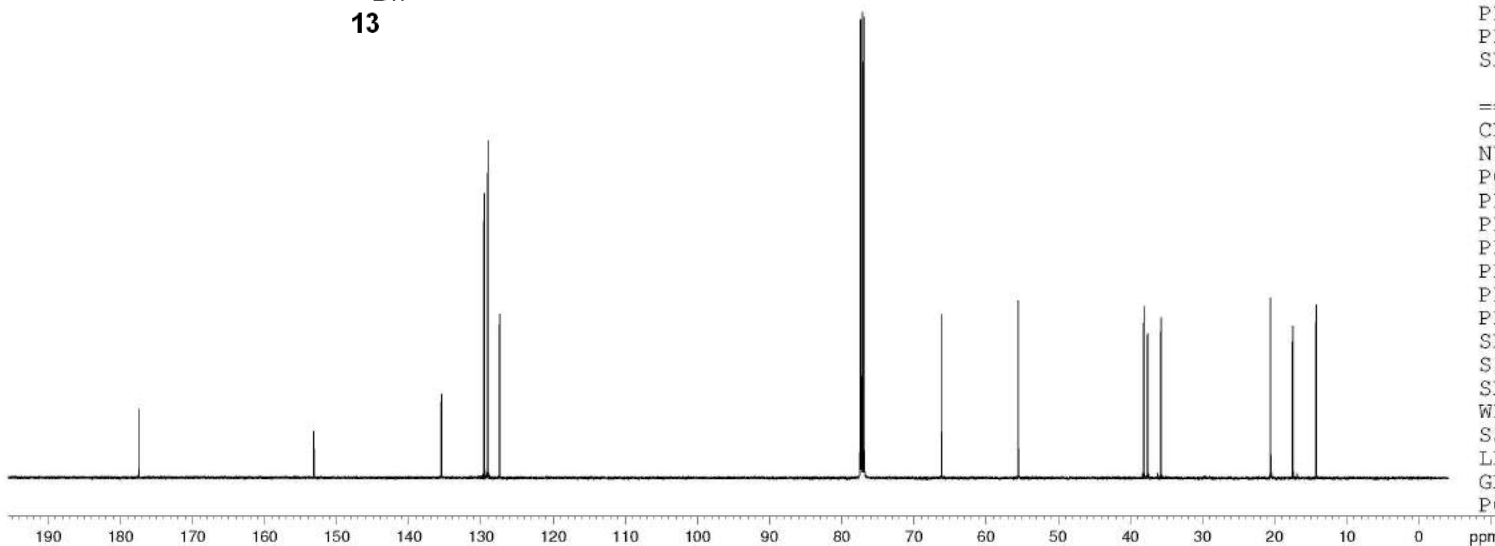
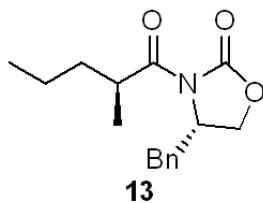
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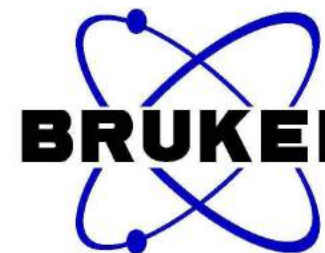
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PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7728799 MHz

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PL12 17.46 dB
PL13 17.46 dB
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PL12W 0.47786582 W
PL13W 0.47786582 W
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GB 0
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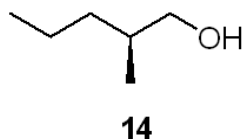
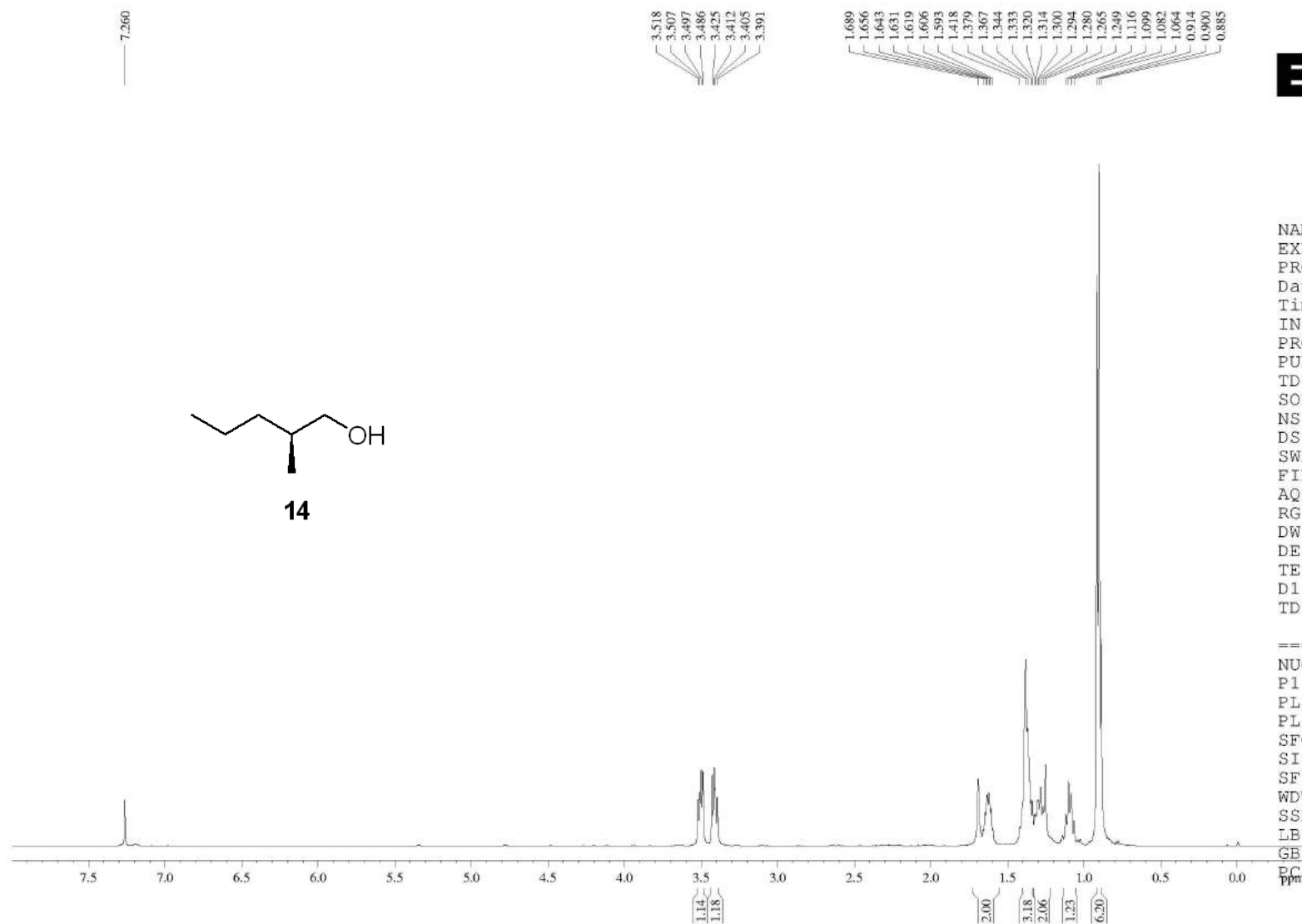


liushouxin-xh-OH



NAME 2019.08.03
EXPNO 1
PROCNO 1
Date_ 20190803
Time 10.38
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 7500.000
FIDRES 0.114441
AQ 4.3691168
RG 144
DW 66.667
DE 6.50
TE 298.7
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1332508
SI 32768
SF 500.1300228
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00

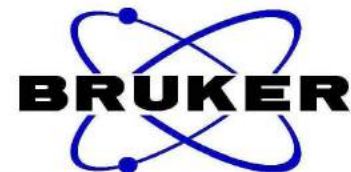


liushouxin-xh-OH

77.41
77.16
76.91
68.50

35.61
35.54

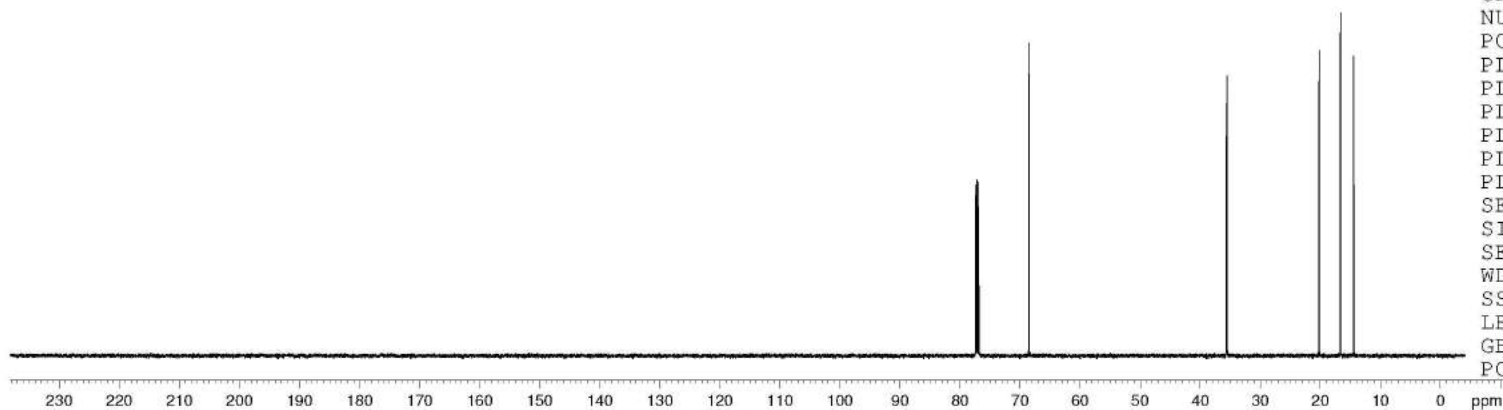
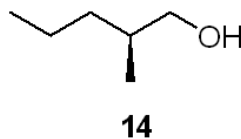
20.19
16.65
14.44



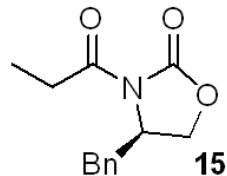
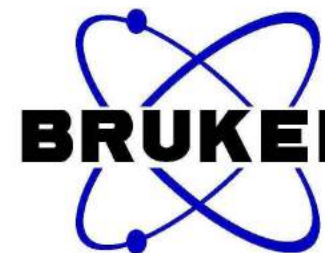
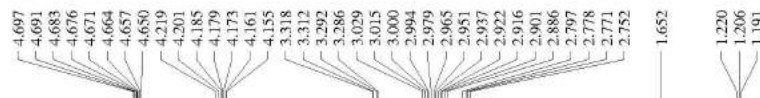
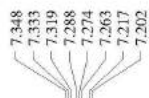
NAME 2019.08.10
EXPNO 6
PROCNO 1
Date_ 20190810
Time 15.49
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 343
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 0.5243380 sec
RG 2050
DW 16.000 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7728799 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577746 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



liushouxin-xh

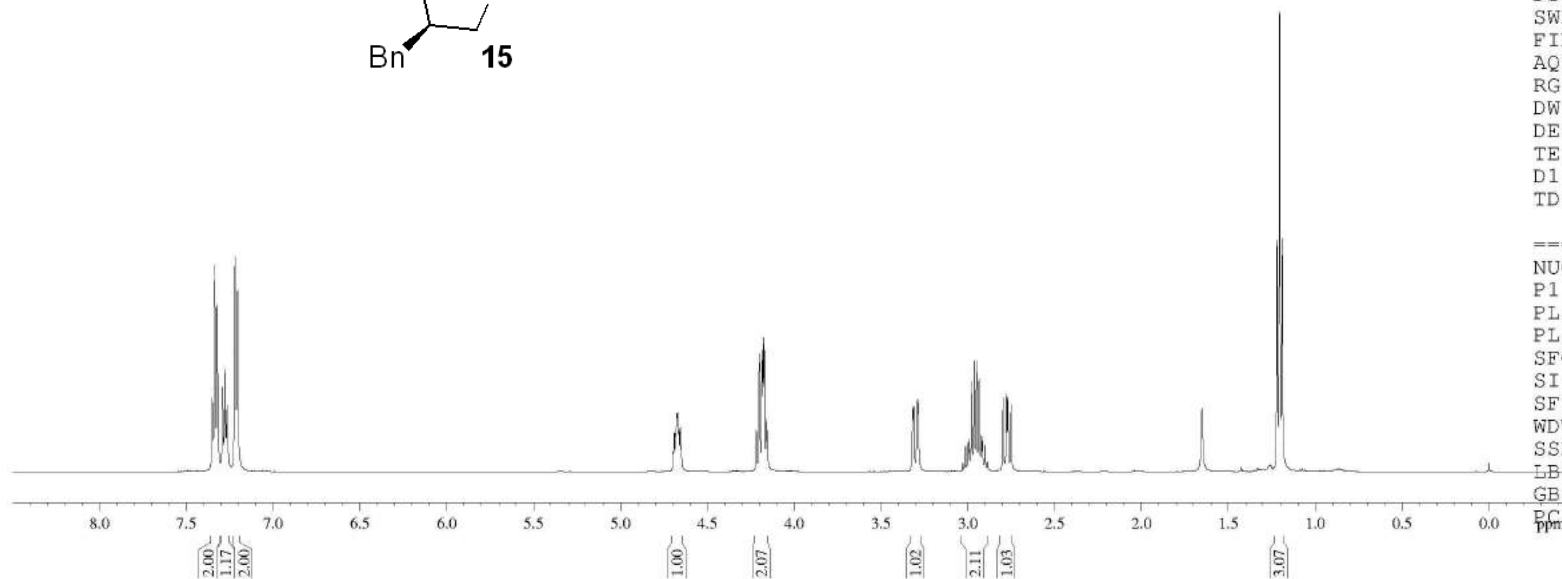


```

NAME          2019.07.27
EXPNO         2
PROCNO        1
Date_         20190727
Time          8.29
INSTRUM       spect
PROBHD        5 mm PADUL 13C
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            0
SWH           7500.000
FIDRES        0.114441
AQ            4.3691168
RG            128
DW            66.667
DE            6.50
TE            298.2
D1            1.00000000
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.50
PL1           2.00
PL1W         16.79986763
SFO1         500.1332508
SI            32768
SF           500.1300213
WDW           EM
SSB           0
LB            1.00
GB            0
PC            1.00
  
```



liushouxin-xh

— 174.21

— 153.63

— 135.46

— 129.53

— 129.07

— 127.45

— 77.41

— 77.16

— 76.90

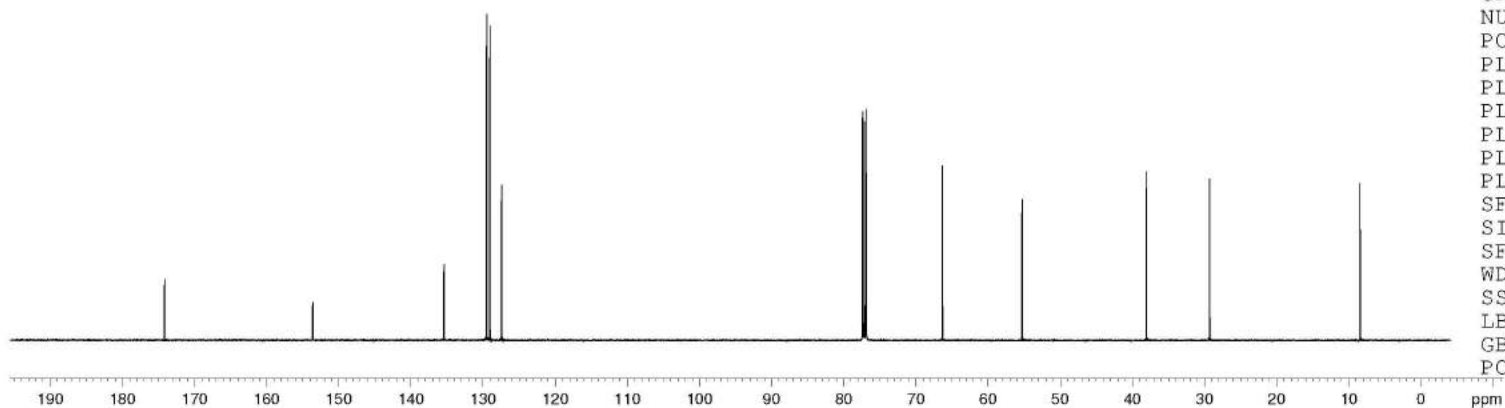
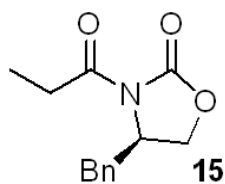
— 66.34

— 55.28

— 38.05

— 29.30

— 8.42

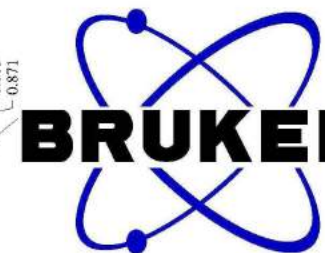


NAME 2019.07.27
EXPNO 3
PROCNO 2
Date_ 20190727
Time 8.50
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 1851
DS 4
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 0.5243380 sec
RG 2050
DW 16.000 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

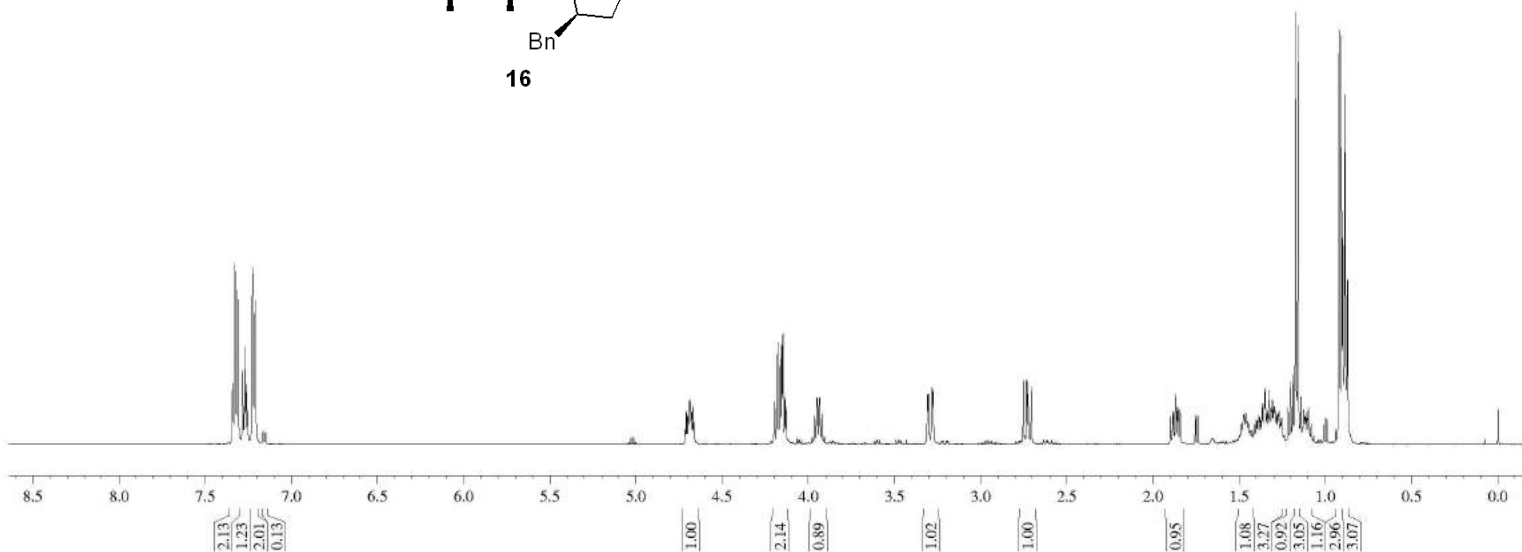
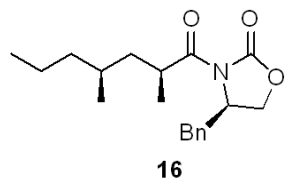
===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7728799 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577772 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

xh-sr



7.538
7.524
7.509
7.281
7.267
7.260
7.252
7.223
7.208
4.706
4.699
4.691
4.686
4.679
4.671
4.665
4.196
4.178
4.165
4.154
4.148
4.136
4.130
3.964
3.951
3.935
3.921
3.310
3.303
3.283
3.277
2.753
2.734
2.727
2.707
1.899
1.887
1.883
1.872
1.860
1.856
1.844
1.475
1.462
1.449
1.400
1.395
1.390
1.386
1.382
1.375
1.367
1.355
1.350
1.340
1.335
1.331
1.325
1.315
1.308
1.300
1.294
1.283
1.274
1.269
1.260
1.255
1.255
1.216
1.204
1.200
1.188
1.173
1.160
1.143
1.125
1.119
1.115
1.109
1.103
1.099
0.920
0.907
0.899
0.885
0.871



NAME 2020.12.23
EXPNO 25
PROCNO 1
Date_ 20201223
Time 17.37
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 80.6
DW 50.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

==== CHANNEL f1 ===
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1340010
SI 32768
SF 500.1300227
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

liushouxin-XH-celian-5

— 178.00 — 153.17 — 135.56 — 129.56 — 129.07 — 127.46

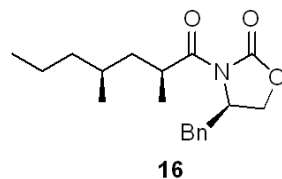
77.41 77.16 76.91 — 66.09 — 55.49

41.01 39.84 38.19 35.50 30.33

20.18 19.25 16.85 14.43

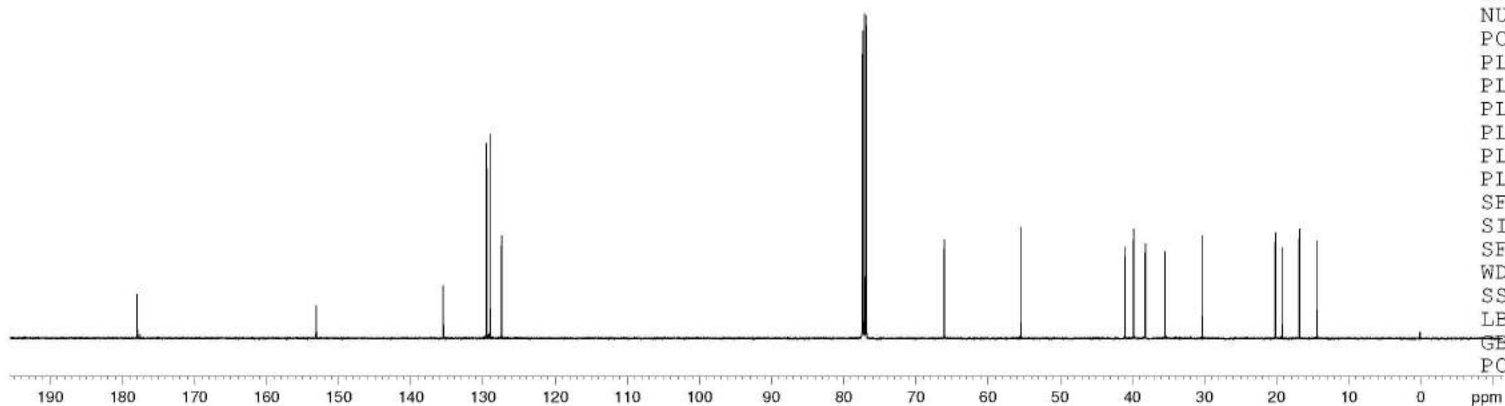


NAME 20190921
 EXPNO 13
 PROCNO 1
 Date_ 20190921
 Time 15.09
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zgpg30
 TD 32768
 SOLVENT CDC13
 NS 2662
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.908261 Hz
 AQ 0.5505524 sec
 RG 1820
 DW 16.800 usec
 DE 6.50 usec
 TE 299.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

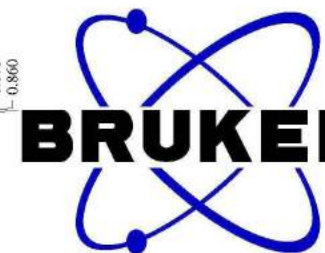


==== CHANNEL f1 =====
 NUC1 13C
 P1 12.00 usec
 PL1 4.50 dB
 PL1W 33.60015869 W
 SFO1 125.7703643 MHz

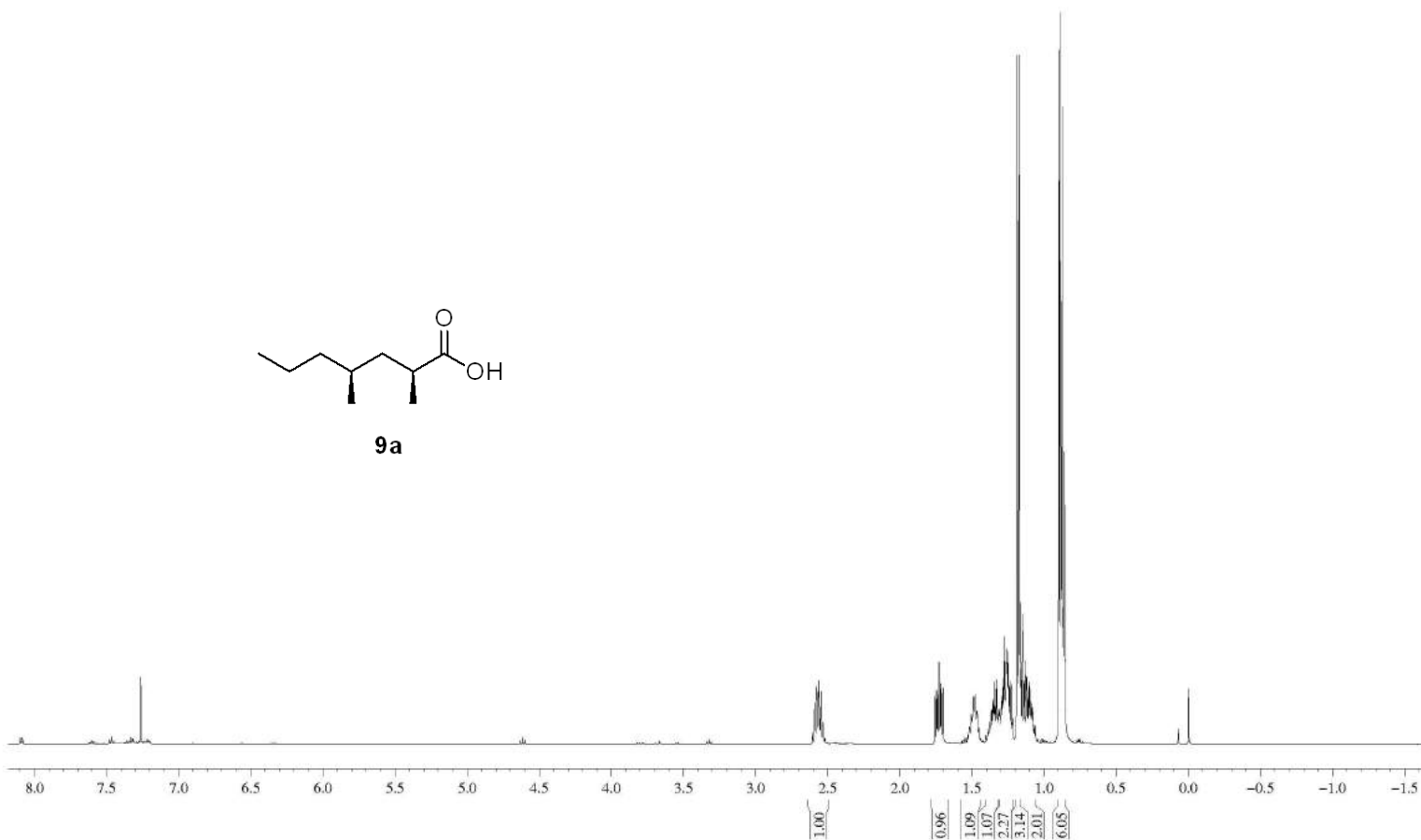
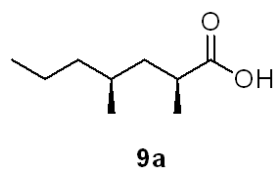
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PL12 17.46 dB
 PL13 17.46 dB
 PL2W 16.79986763 W
 PL12W 0.47786582 W
 PL13W 0.47786582 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7577739 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



xh-sr-cooh



8.082 7.463 7.529 7.260 4.612 2.604 2.591 2.587 2.577 2.573 2.565 2.561 2.547 2.533 2.533 1.746 1.739 1.729 1.719 1.712 1.701 1.554 1.541 1.529 1.517 1.505 1.492 1.479 1.466 1.452 1.452 1.404 1.393 1.389 1.378 1.372 1.368 1.363 1.357 1.353 1.349 1.343 1.338 1.329 1.320 1.315 1.310 1.306 1.300 1.295 1.291 1.286 1.280 1.275 1.265 1.258 1.253 1.248 1.244 1.238 1.234 1.228 1.221 1.217 1.188 1.174 1.166 1.160 1.151 1.151 1.139 1.133 1.121 1.111 1.105 1.101 1.095 1.090 1.085 1.079 1.068 1.059 0.898 0.889 0.885 0.875 0.860



NAME 2020.12.23
EXPNO 22
PROCNO 1
Date_ 20201223
Time 17.20
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 80.6
DW 50.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

==== CHANNEL f1 ===
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1340010
SI 32768
SF 500.1300227
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

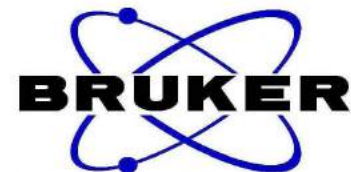
liushouxin-xh--SR-COOH

183.80

77.41
77.16
76.91

41.37
39.42
37.46
30.58

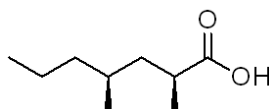
19.99
19.68
17.94
14.42



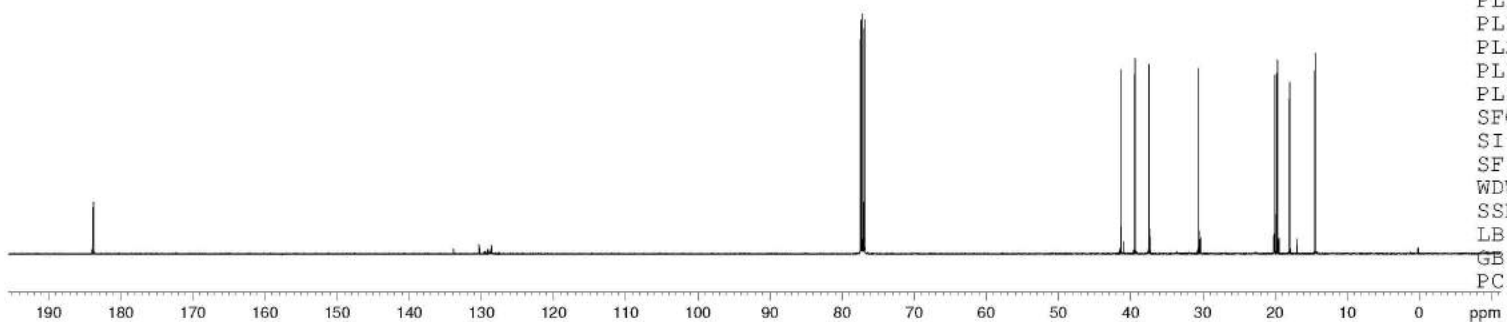
NAME 2021.03.26
EXPNO 10
PROCNO 1
Date_ 20210326
Time 12.33
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 5120
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

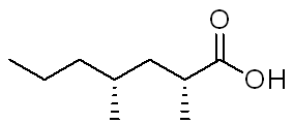


9a

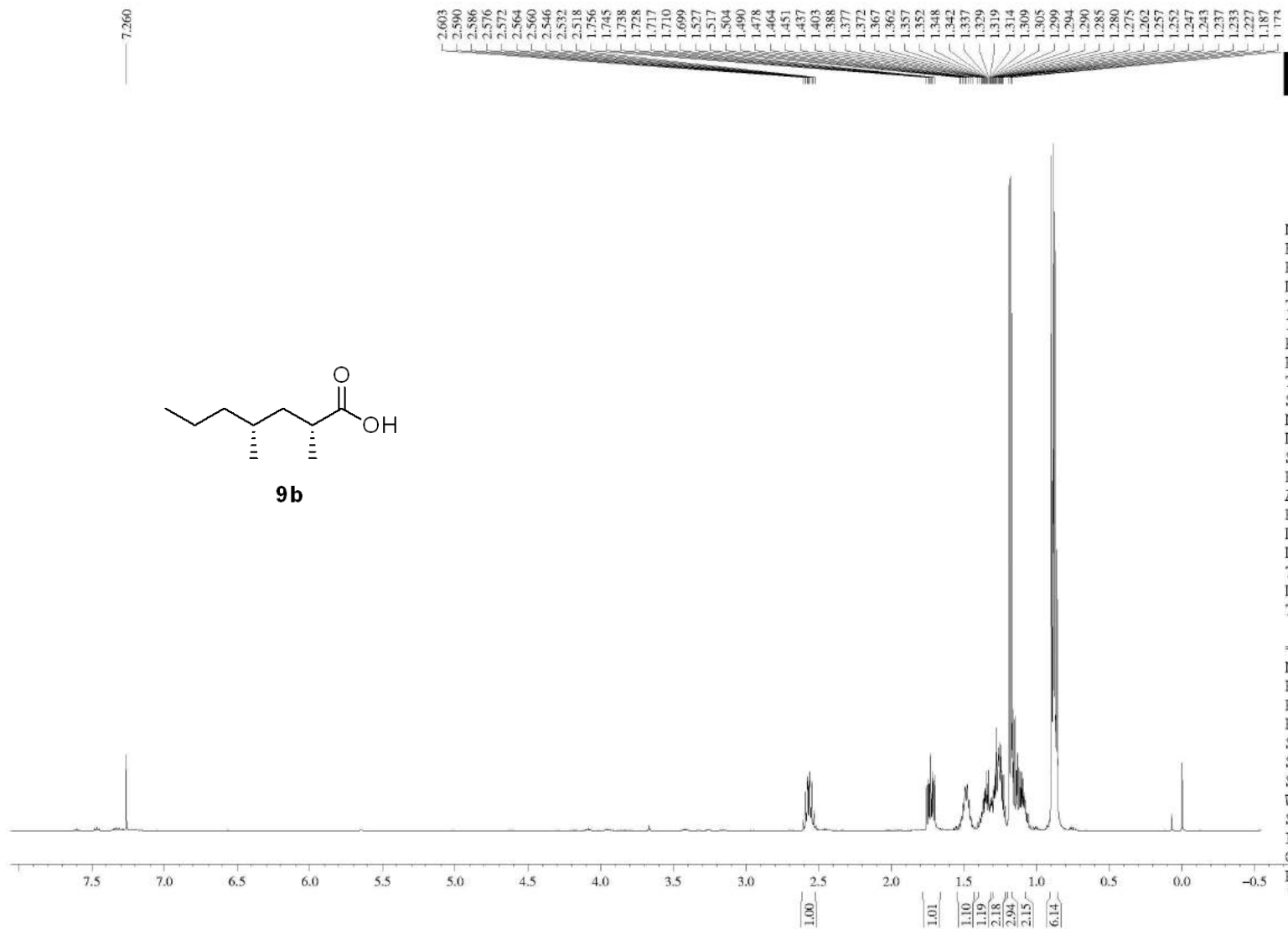


xh-rs-cooh

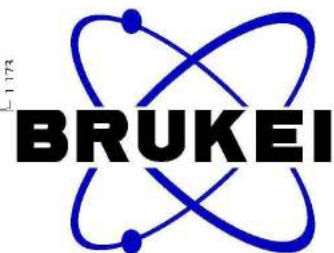
7.260



9b



2.603
2.590
2.586
2.576
2.572
2.564
2.560
2.546
2.532
2.518
1.756
1.745
1.738
1.728
1.717
1.710
1.699
1.627
1.517
1.504
1.490
1.478
1.464
1.451
1.437
1.403
1.388
1.377
1.372
1.367
1.362
1.357
1.352
1.348
1.342
1.337
1.329
1.319
1.314
1.309
1.305
1.299
1.294
1.290
1.285
1.280
1.275
1.262
1.257
1.252
1.247
1.243
1.237
1.233
1.227
1.187
1.173



NAME 2020.12.18
EXPNO 11
PROCNO 1
Date_ 20201218
Time 19.23
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 0
SWH 5000.000
FIDRES 0.076294
AQ 6.5536499
RG 90.5
DW 100.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1322506
SI 32768
SF 500.1300228
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

liushouxin-xh-RS-COOH

184.00

77.41
77.16
76.91

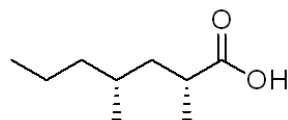
41.36
39.41
37.47
30.57
19.98
19.67
17.93
14.42



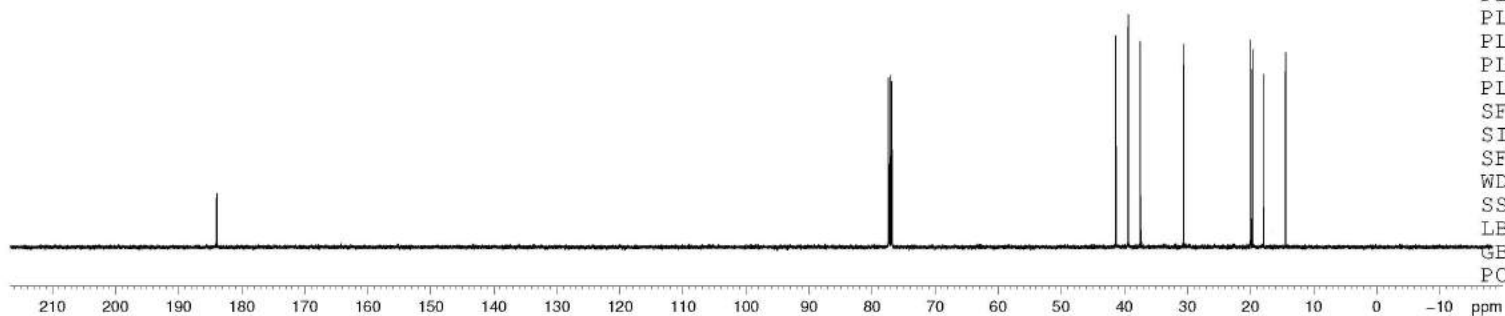
NAME 2021.03.11
EXPNO 14
PROCNO 1
Date_ 20210319
Time 16.54
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 125
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1150
DW 16.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577730 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



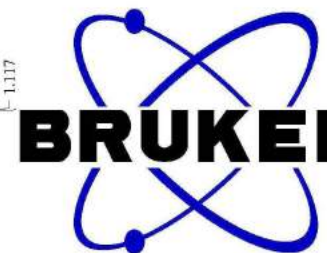
9b



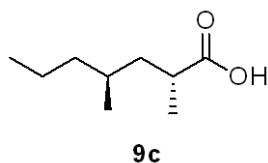
xh-cooh

7.260

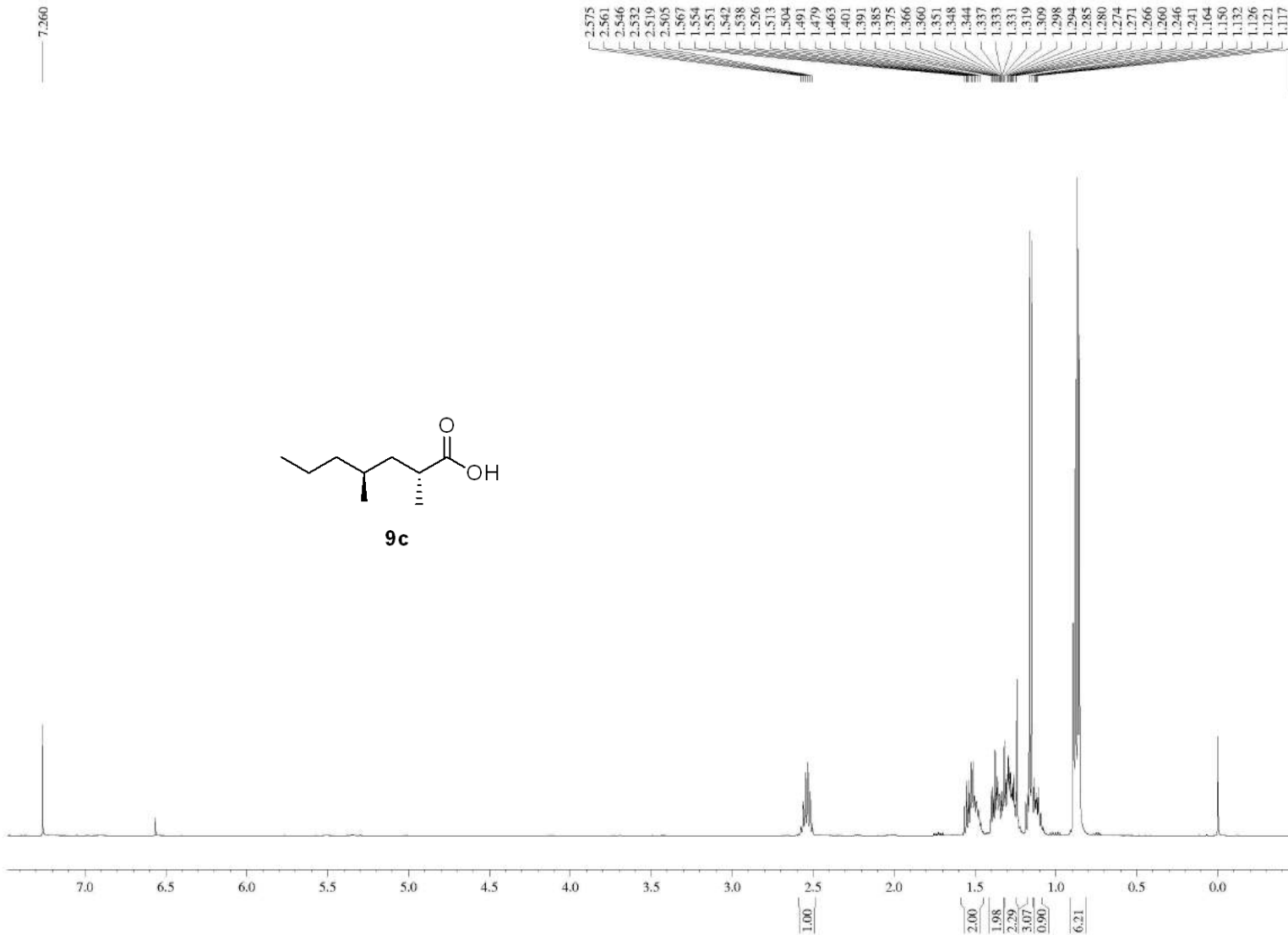
2.575
2.561
2.546
2.532
2.519
2.505
1.567
1.554
1.551
1.542
1.538
1.526
1.513
1.504
1.491
1.479
1.463
1.401
1.391
1.385
1.375
1.366
1.360
1.351
1.344
1.344
1.337
1.333
1.331
1.319
1.309
1.298
1.294
1.285
1.280
1.274
1.271
1.266
1.260
1.246
1.241
1.164
1.150
1.132
1.126
1.121
1.117



NAME 2020.12.01
EXPNO 15
PROCNO 1
Date_ 20201201
Time 20.00
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 114
DW 50.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1



==== CHANNEL f1 ===
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1340010
SI 32768
SF 500.1300230
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



liushouxin XH-COOH-SS

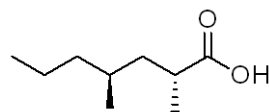
183.86

77.42
77.16
76.91

40.95
39.44
37.30
30.33
20.08
19.44
16.97
14.42



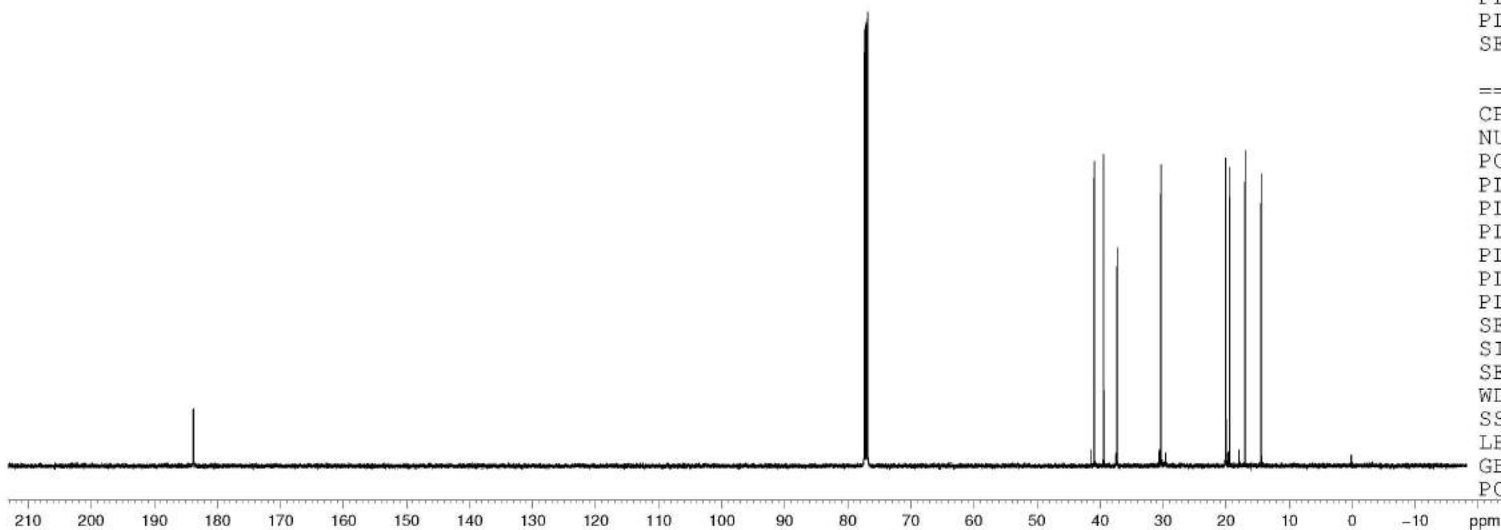
NAME 2021.05.07
EXPNO 15
PROCNO 1
Date_ 20210510
Time 22.13
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 805
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1440
DW 16.800 usec
DE 6.50 usec
TE 299.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



9c

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577713 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

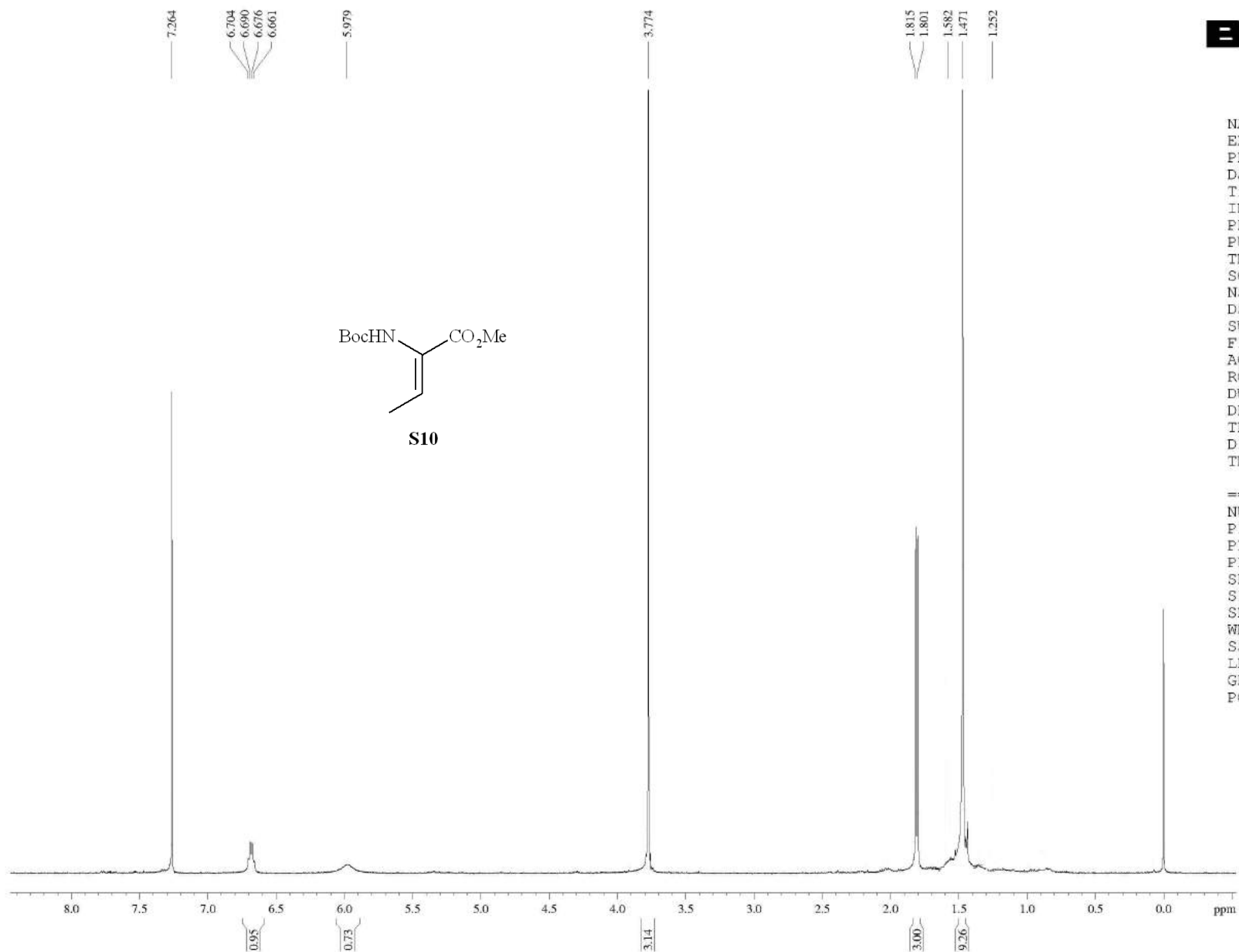


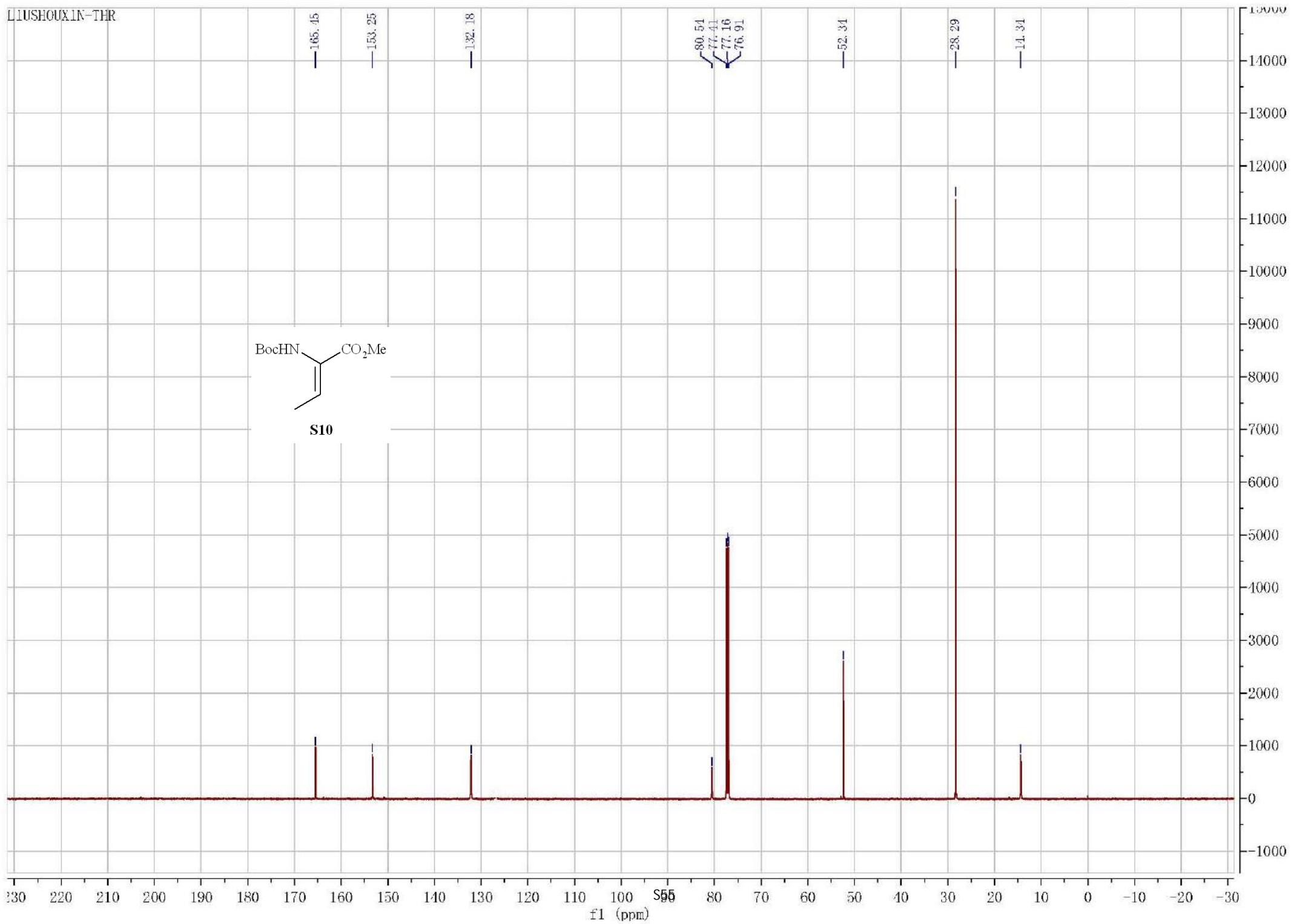
liushouxin-f-2013-4-10b



NAME 2013.04.13
EXPNO 5
PROCNO 1
Date_ 20130413
Time 16.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 7500.000
FIDRES 0.228882
AQ 2.1845834
RG 90.5
DW 66.667
DE 6.50
TE 293.4
D1 1.00000000
TD0 1

==== CHANNEL f1 ====
NUC1 1H
P1 13.00
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1300116
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



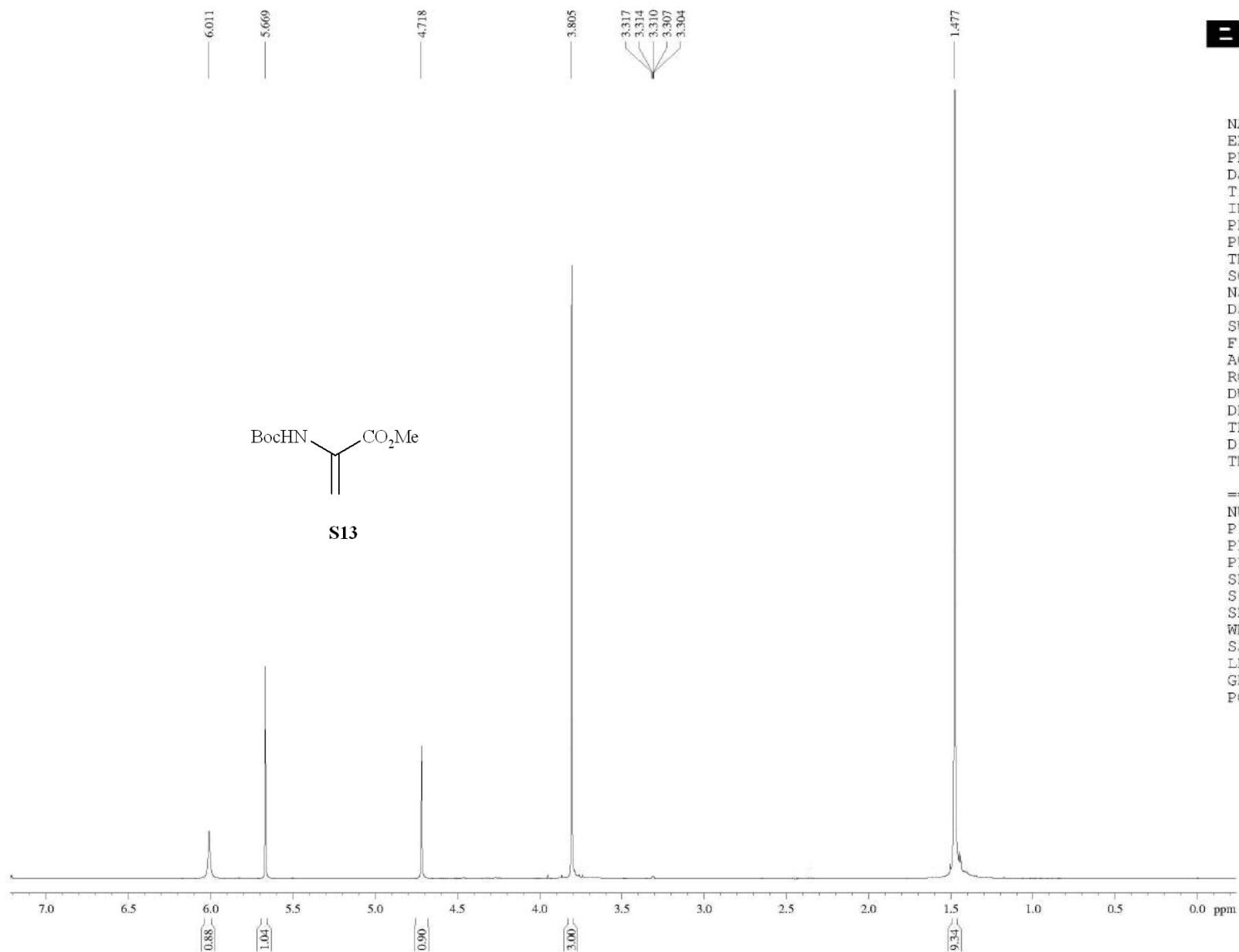
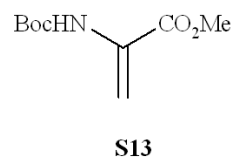


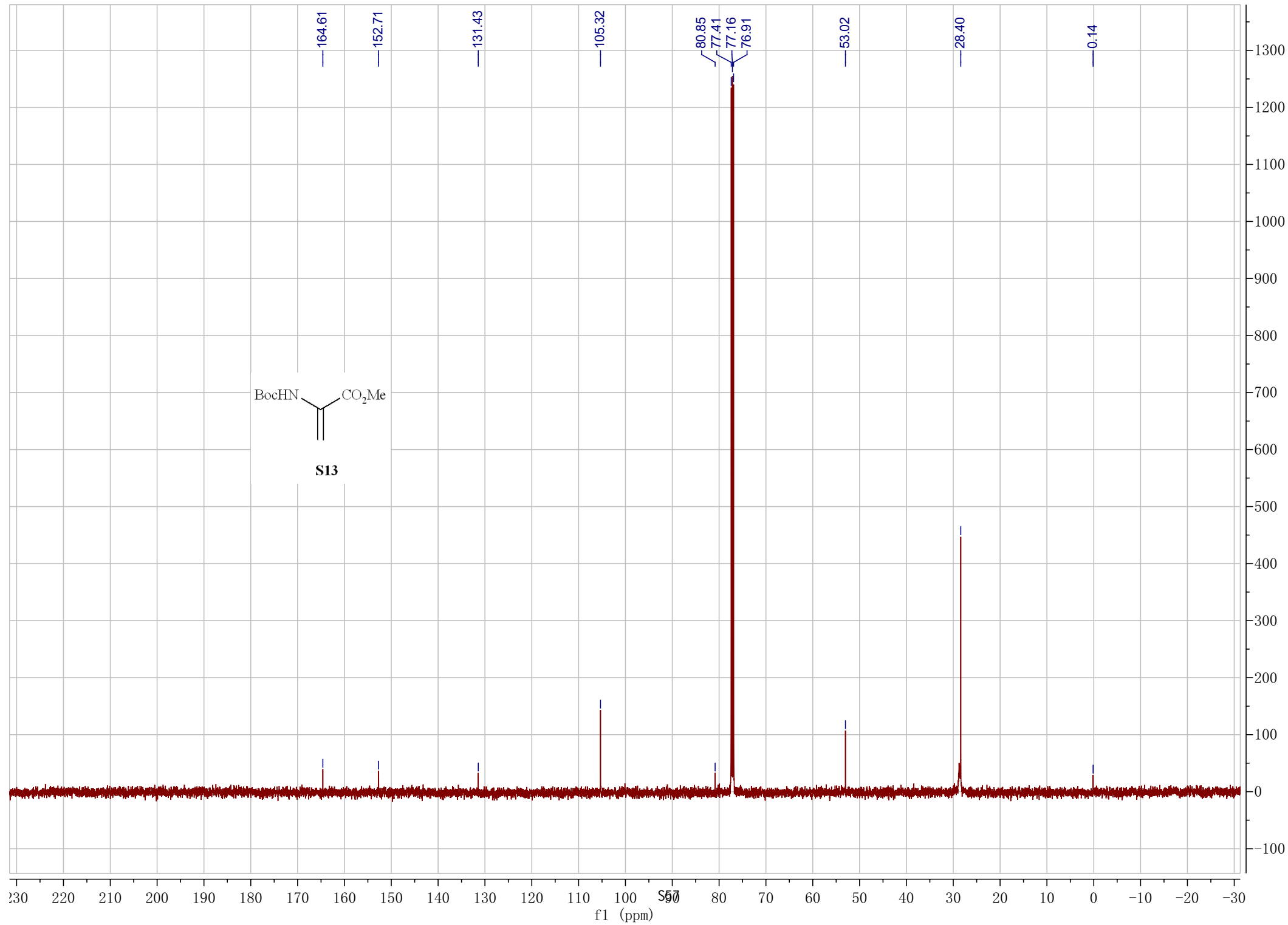
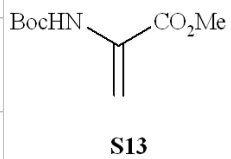
liushouxin-2018-3-14-ZY



NAME 2018.03.15
EXPNO 10
PROCNO 1
Date_ 20180315
Time 15.05
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 0
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 18
DW 48.400
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1300153
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



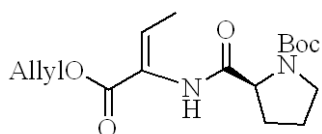


liu shou xin THR-Pro-H2o

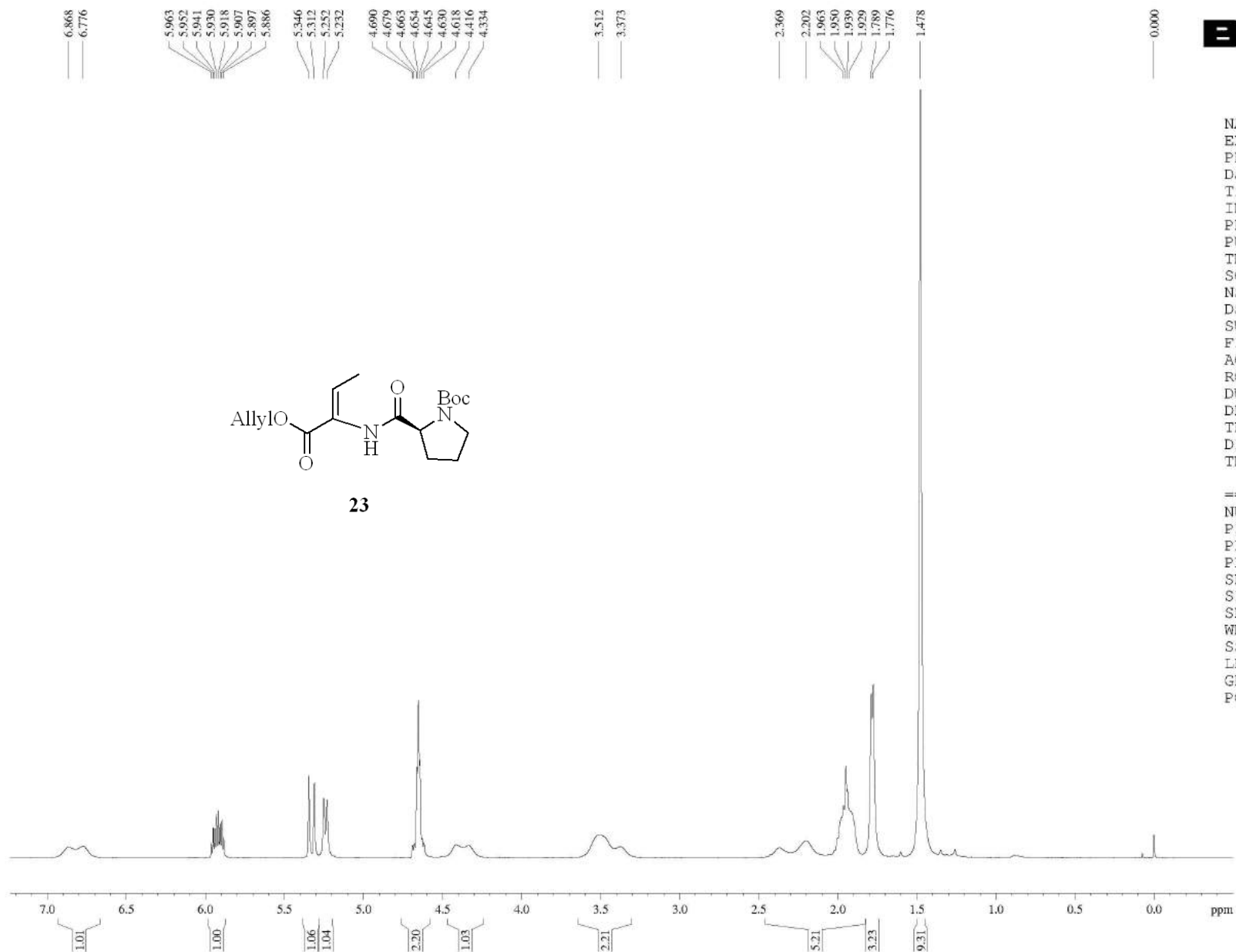


NAME 2012.08.08
EXPNO 4
PROCNO 1
Date_ 20120808
Time 15.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 64
DW 48.400
DE 6.50
TE 299.9
D1 1.00000000
TD0 1

==== CHANNEL f1 ====
NUC1 1H
P1 13.00
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1299975
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00



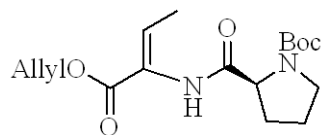
23



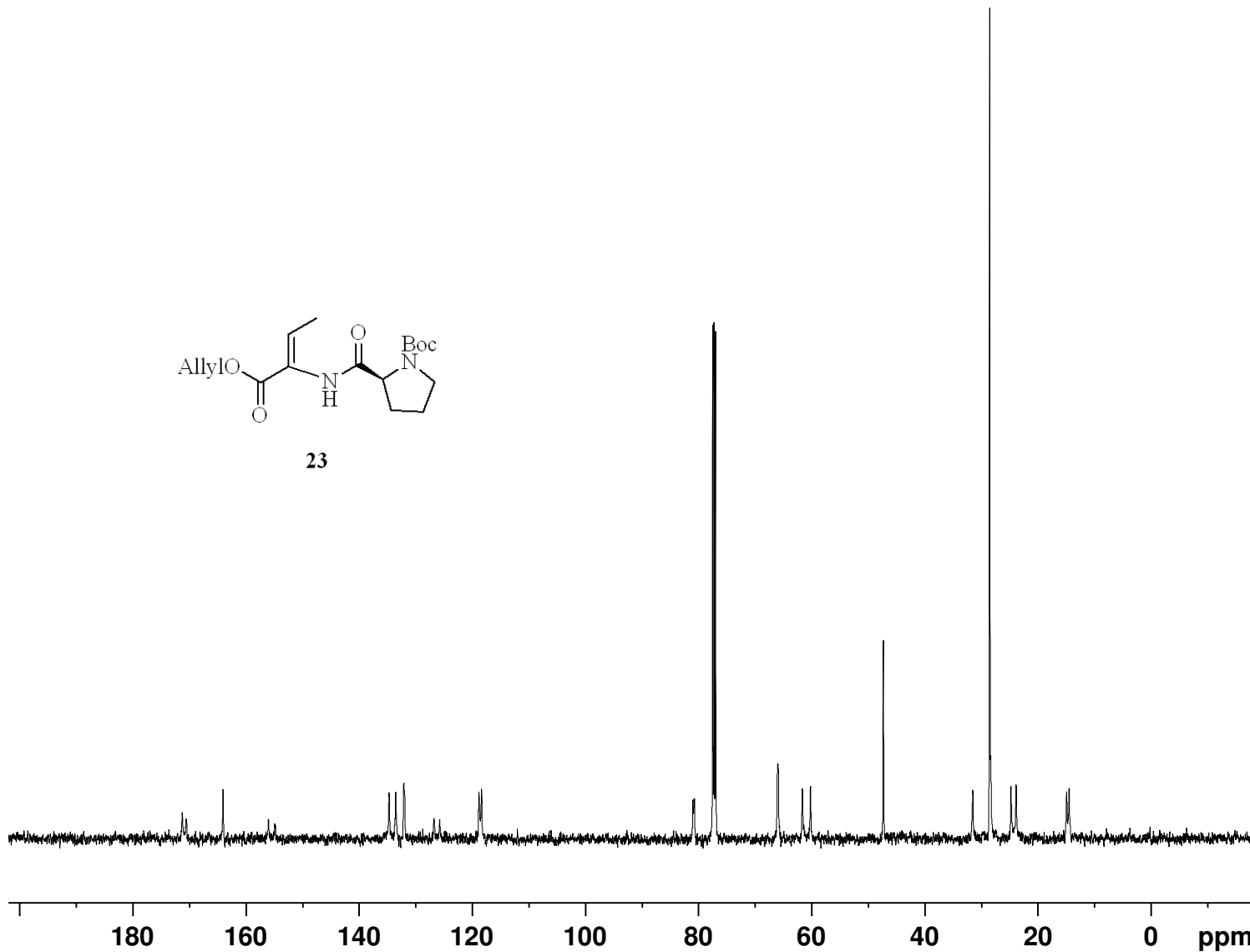
liushouxin-Thr-L-Pro

171.25
170.47
164.01
155.97
154.86
134.65
133.47
132.04
126.70
125.68
118.76
118.30

80.91
80.64
77.41
77.15
76.90
65.86
61.56
60.11
47.23
31.43
28.43
24.68
23.77
14.82
14.38



23



S59



Current Data Parameters
NAME xuehong
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20211102
Time 12.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 4096
SOLVENT CDCl3
NS 396
DS 0
SWH 29761.904 Hz
FIDRES 3.633045 Hz
AQ 0.1376256 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577799 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

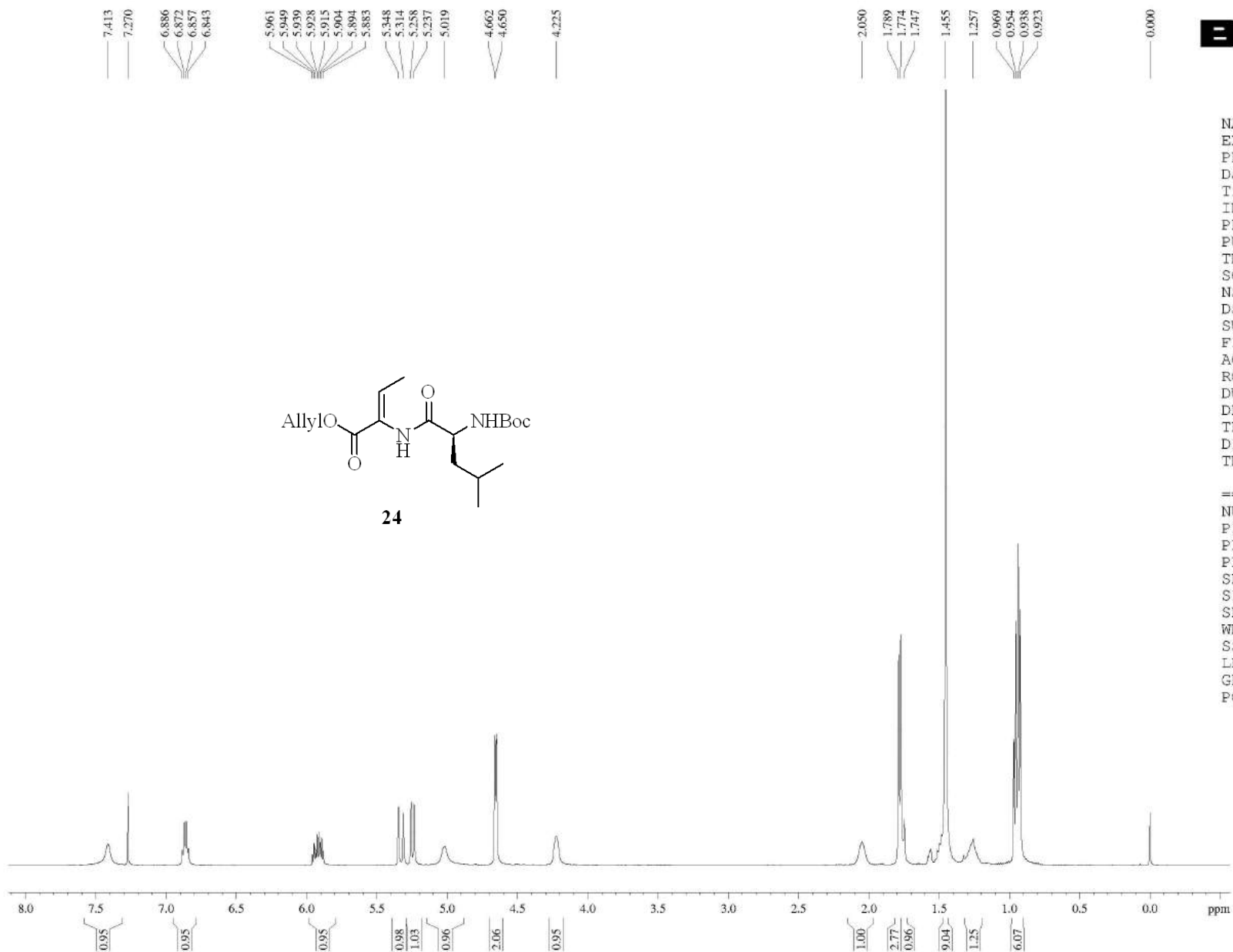
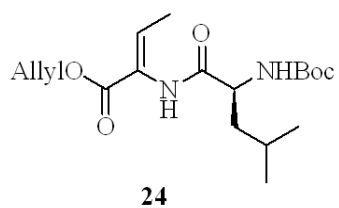
liu shou xin

Thr-I-Leu2

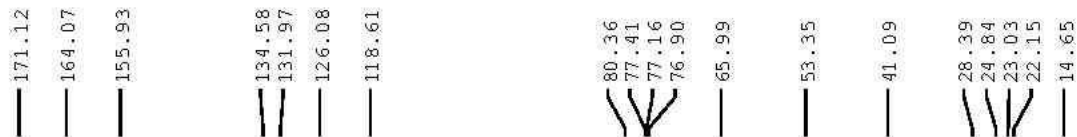


NAME 2012.07.25
EXPNO 5
PROCNO 1
Date_ 20120724
Time 21.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 90.5
DW 48.400
DE 6.50
TE 299.3
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1300083
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00

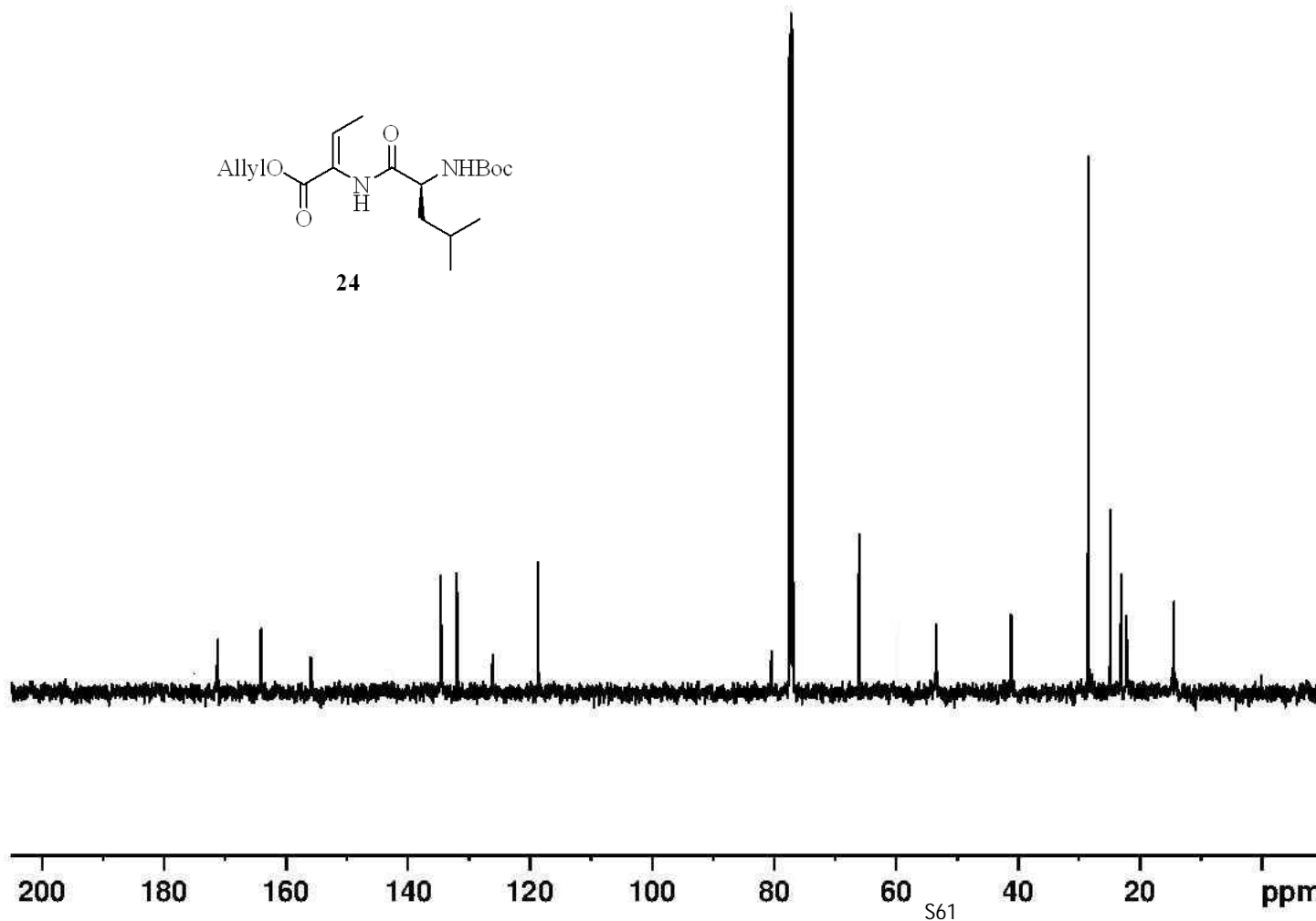
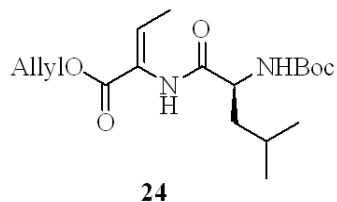


liushouxin-Thr-L-Leu



Current Data Parameters
 NAME xuehong
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20211108
 Time 11.47
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 2048
 SOLVENT CDCl3
 NS 198
 DS 0
 SWH 29761.904 Hz
 FIDRES 3.633045 Hz
 AQ 0.1376256 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 293.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1



----- CHANNEL f1 -----
 NUC1 13C
 P1 12.30 usec
 PL1 4.50 dB
 PL1W 33.60015869 W
 SFO1 125.7703643 MHz

----- CHANNEL f2 -----
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PL12 17.78 dB
 PL13 17.78 dB
 PL2W 16.79986763 W
 PL12W 0.44392112 W
 PL13W 0.44392112 W
 SFO2 500.1320005 MHz

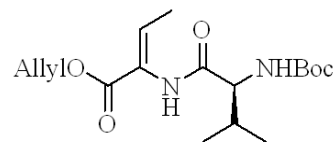
F2 - Processing parameters
 SI 32768
 SF 125.7577754 MHz
 WDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.40

liu shou xin THR-Val-H2O2

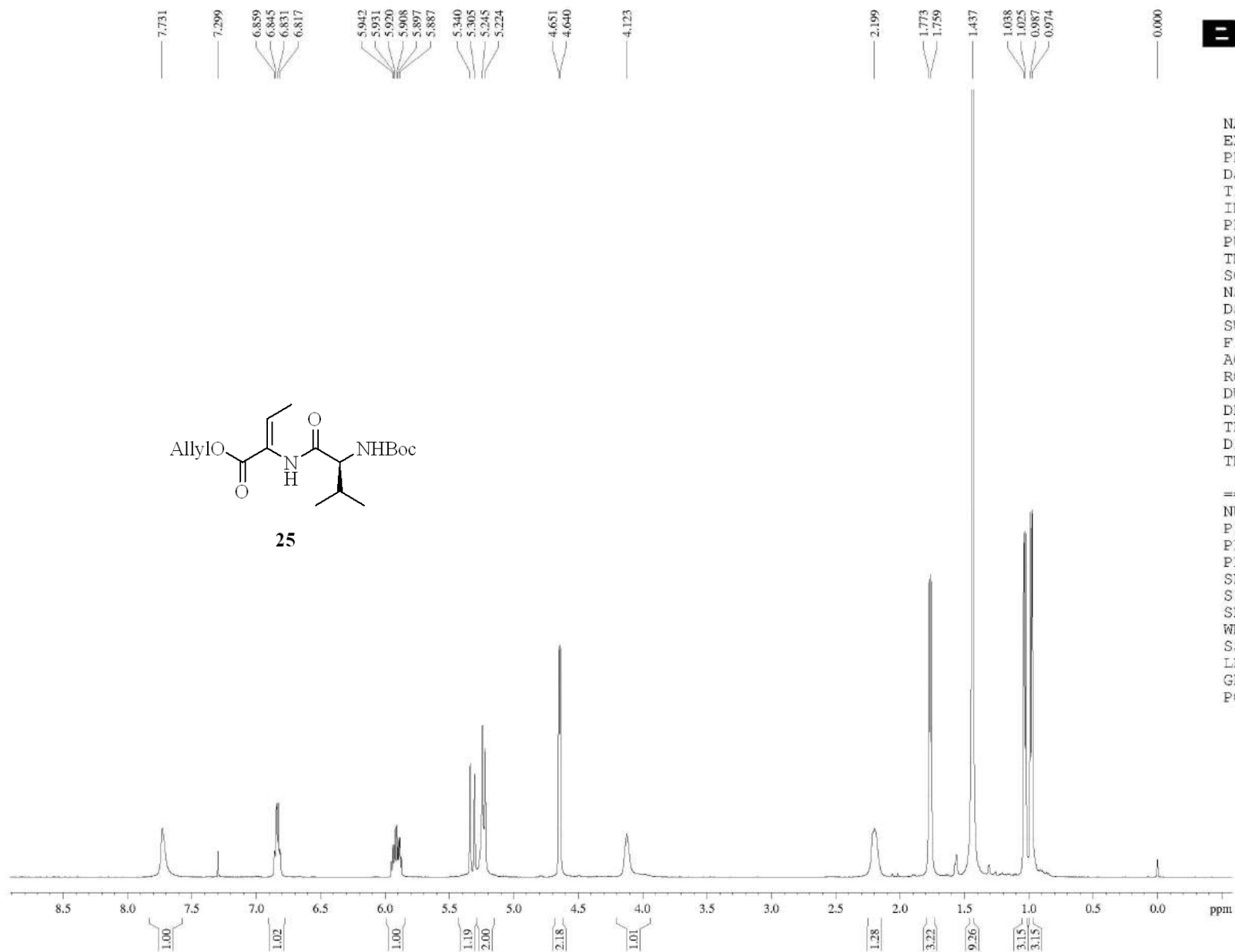


NAME 2012.07.24
EXPNO 5
PROCNO 1
Date_ 20120723
Time 21.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 144
DW 48.400
DE 6.50
TE 299.1
D1 1.00000000
TD0 1

=====
CHANNEL f1
=====
NUC1 1H
P1 13.00
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1299938
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



25

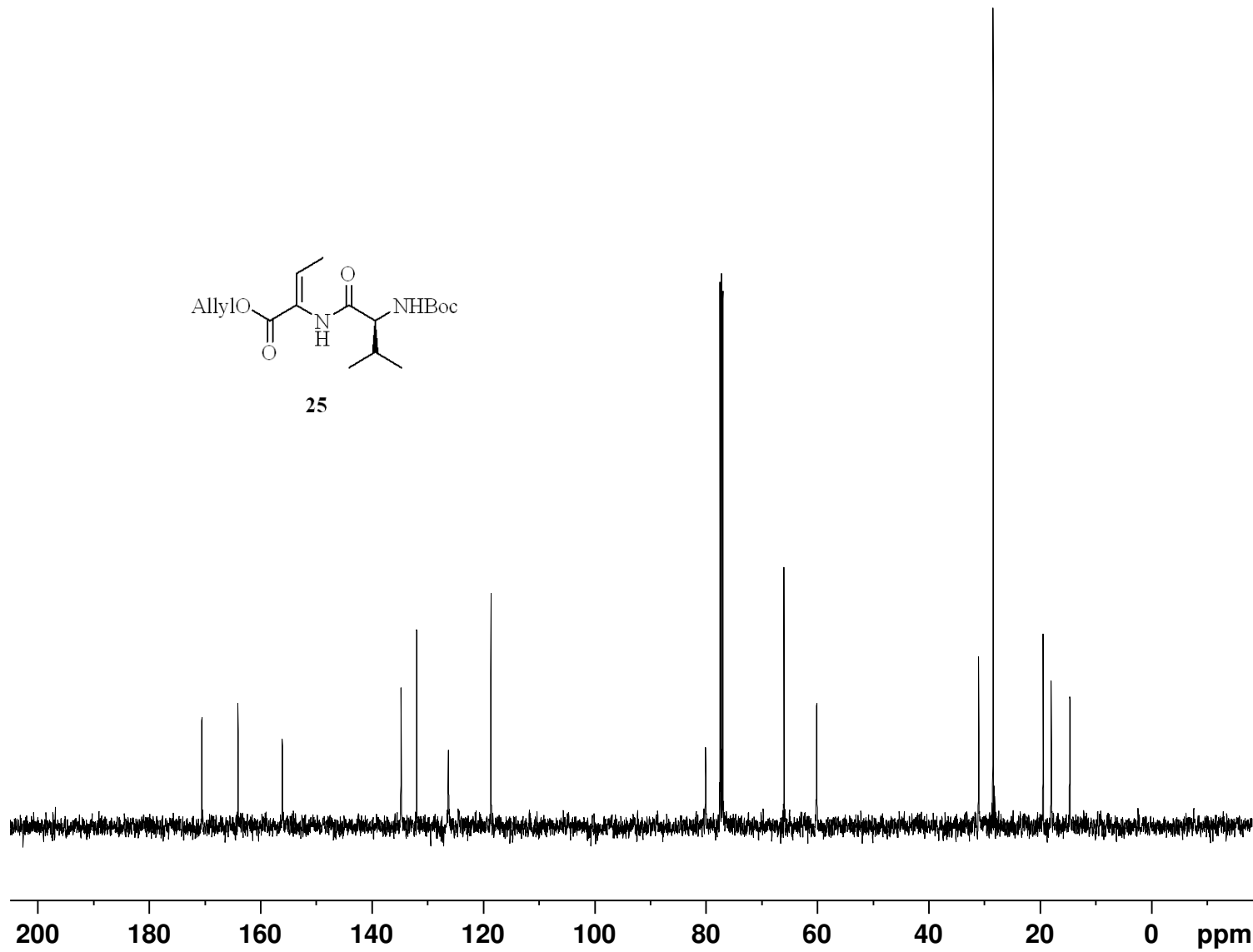
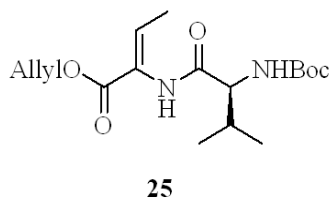


liushouxin-Thr-L-Val

170.51
164.01
156.05
134.73
131.90
126.21
118.57

79.99
77.41
77.16
76.90
65.93
60.04

30.94
28.33
19.35
17.90
14.53



Current Data Parameters
NAME xuehong
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20211102
Time 14.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 8192
SOLVENT CDC13
NS 152
DS 0
SWH 29761.904 Hz
FIDRES 3.633045 Hz
AQ 0.1376256 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577813 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

liu shou xin Thr-I-Leu

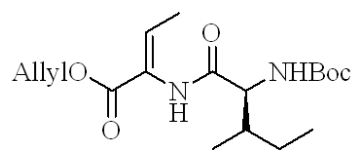


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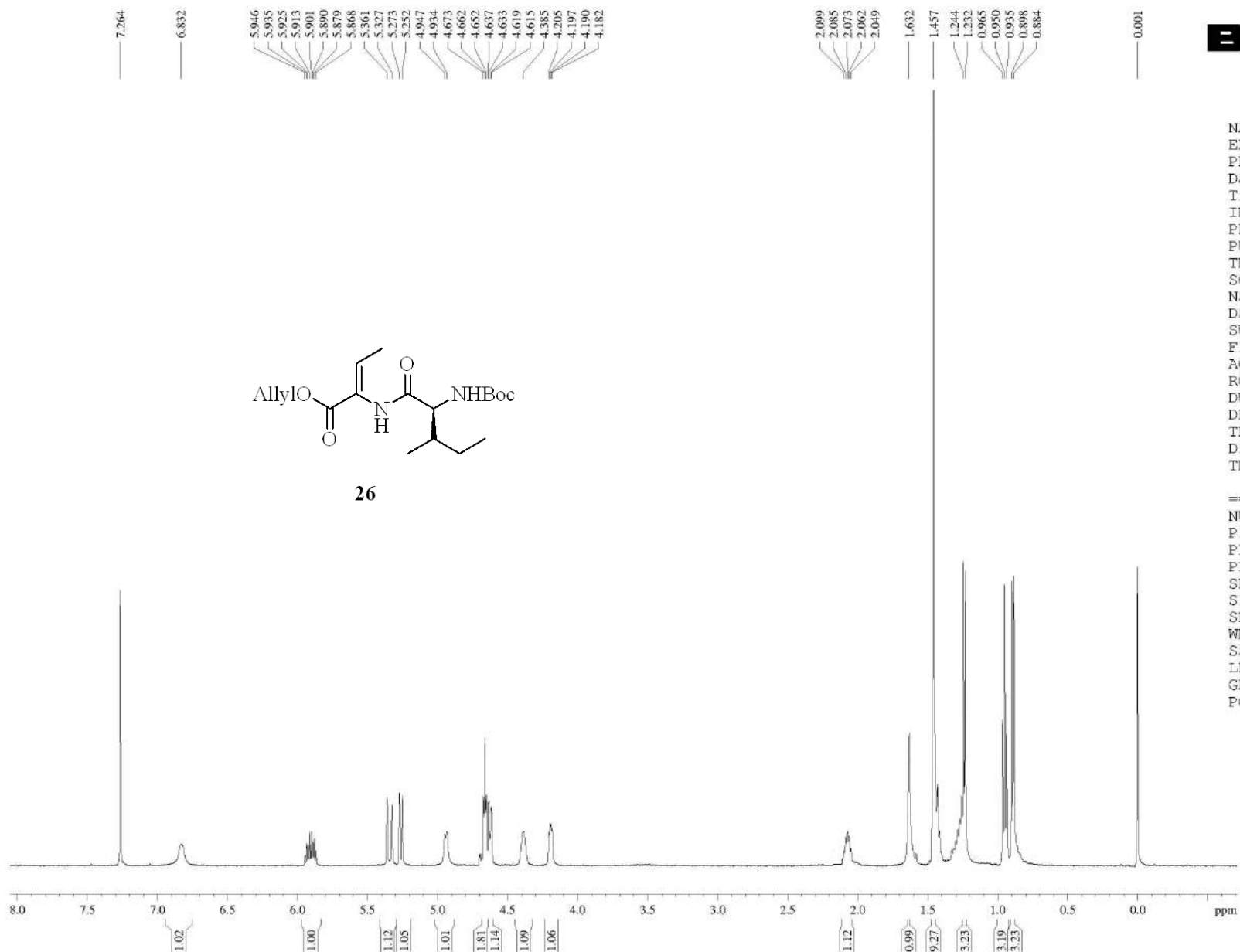
NAME      2012.07.24
EXPNO     9
PROCNO    1
Date_     20120723
Time      21.52
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        4
DS        2
SWH       10330.578
FIDRES    0.157632
AQ        3.1719923
RG        322
DW        48.400
DE        6.50
TE        299.1
D1        1.00000000
TD0       1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        13.00
PL1       2.00
PL1W      16.79986763
SFO1      500.1330885
SI        32768
SF        500.1300115
WDW       EM
SSB       0
LB        0.30
GB        0
PC        1.00
    
```

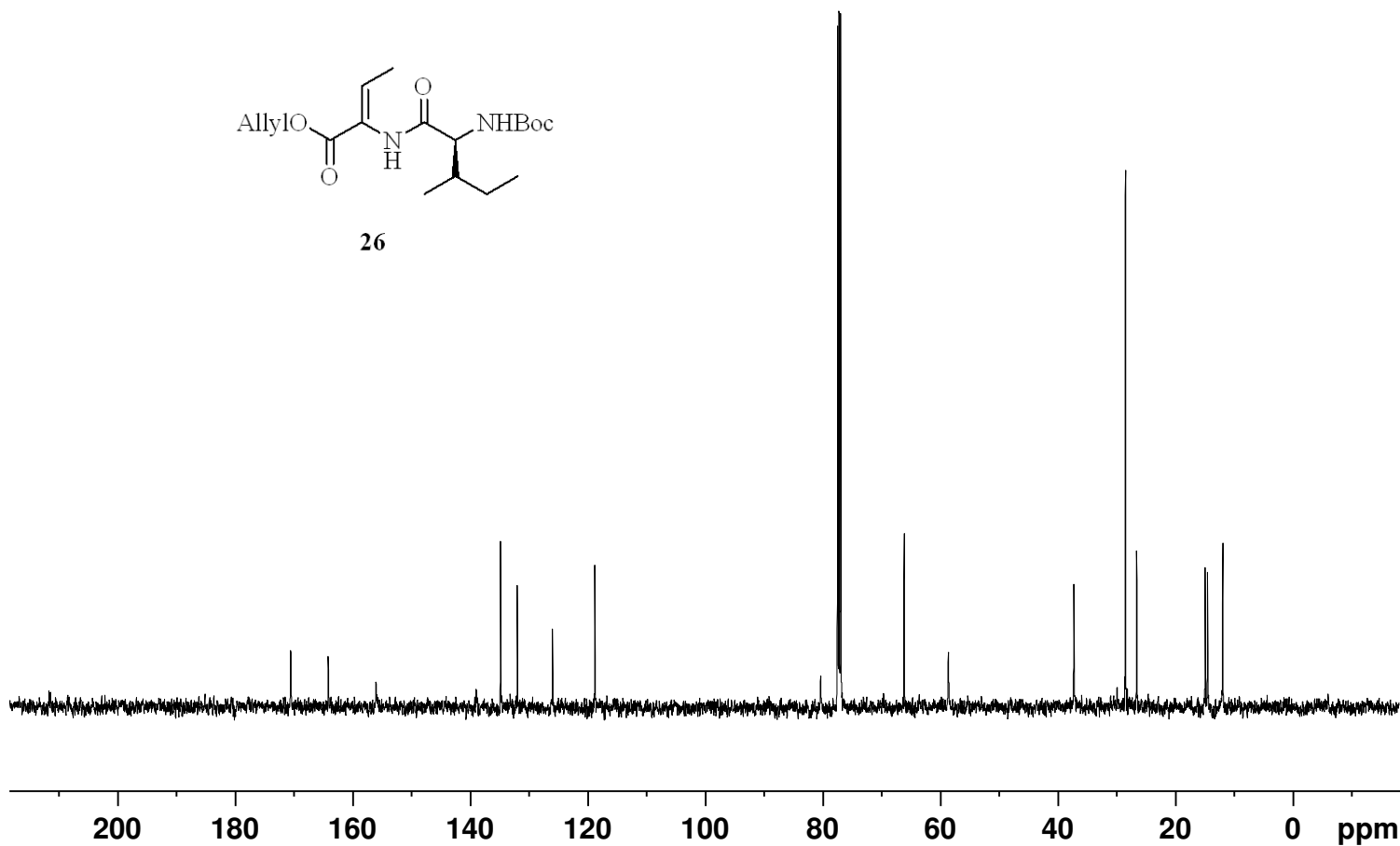
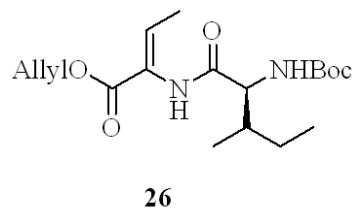


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liushouxin-Thr-L-Ileu

170.50
164.11
156.03
134.80
131.93
125.93
118.73
80.28
77.41
77.16
76.90
66.07
58.54
37.18
28.39
26.48
14.83
14.42
11.82



S65



Current Data Parameters
NAME xuehong
EXPNO 5
PROCNO 1

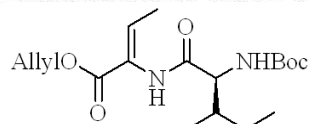
F2 - Acquisition Parameters
Date_ 20211103
Time 10.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 8192
SOLVENT CDC13
NS 399
DS 0
SWH 29761.904 Hz
FIDRES 3.633045 Hz
AQ 0.1376256 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

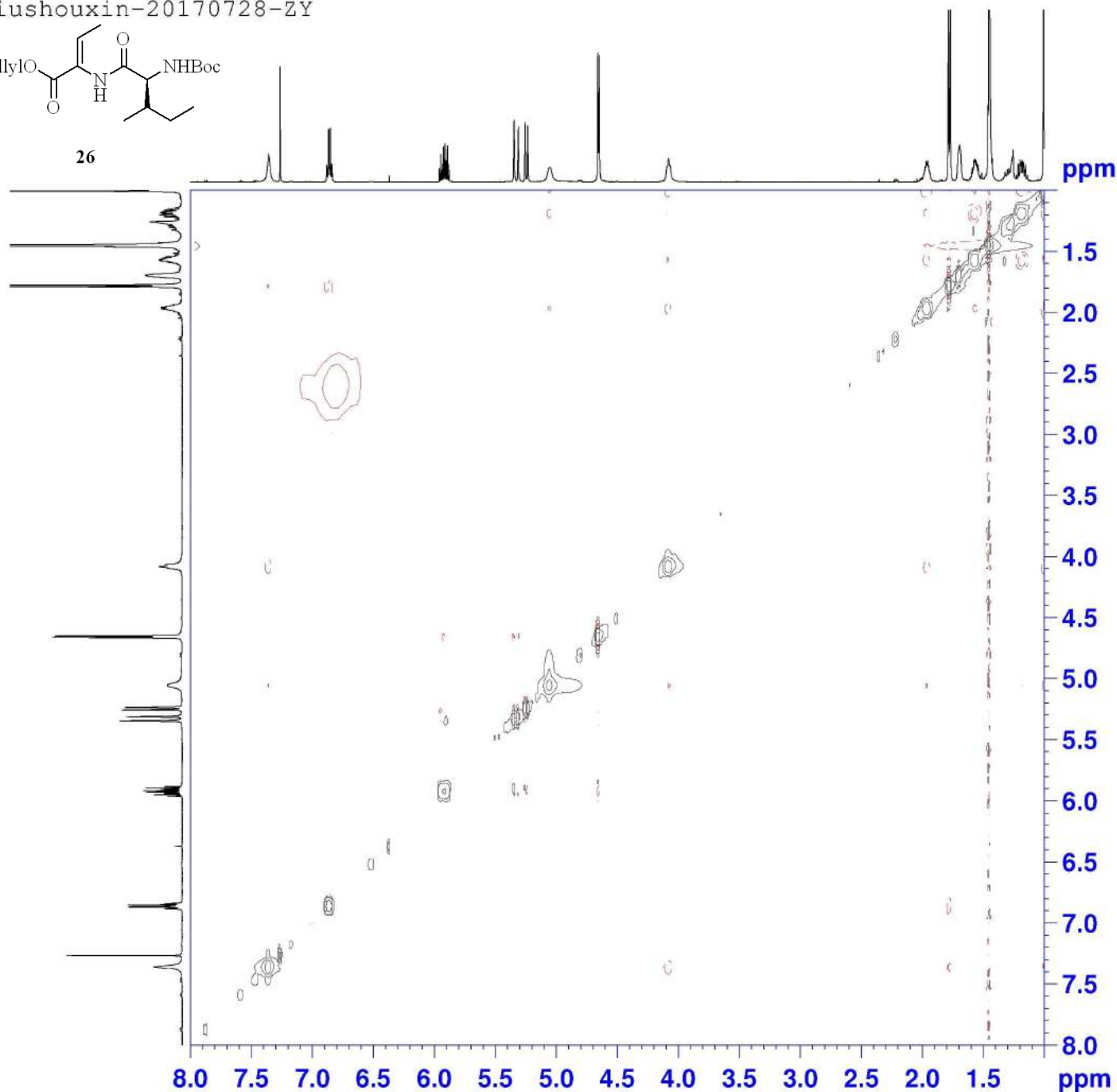
==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577750 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

liushouxin-20170728-ZY



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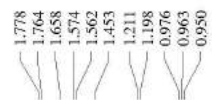
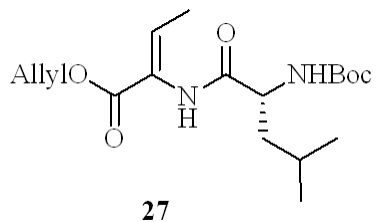
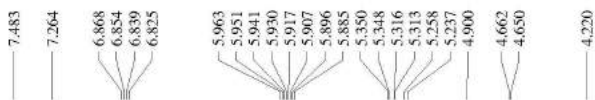


NAME 2017.07.28
EXPNO 10
PROCNO 1
Date_ 20170728
Time 14.38
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG noesygpph
TD 2048
SOLVENT CDCl3
NS 16
DS 8
SWH 4504.504 Hz
FIDRES 2.199465 Hz
AQ 0.2273780 sec
RG 456
DW 111.000 usec
DE 6.50 usec
TE 298.3 K
D0 0.00009453 sec
D1 1.00000000 sec
D8 0.60000002 sec
D16 0.00020000 sec
IN0 0.00022215 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
P2 26.00 usec
PL1 2.00 dB
PL1W 16.79986763 W
SFO1 500.1317505 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 40.00 %
GPZ2 -40.00 %
P16 1500.00 usec
ND0 1
TD 256
SFO1 500.1318 MHz
FIDRES 17.582756 Hz
SW 9.000 ppm
FnMODE States-IPPI
SI 1024
SF 500.1300190 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-IPPI
SF 500.1300168 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

liu shou xin Thr-D-Leu-H2o(2)

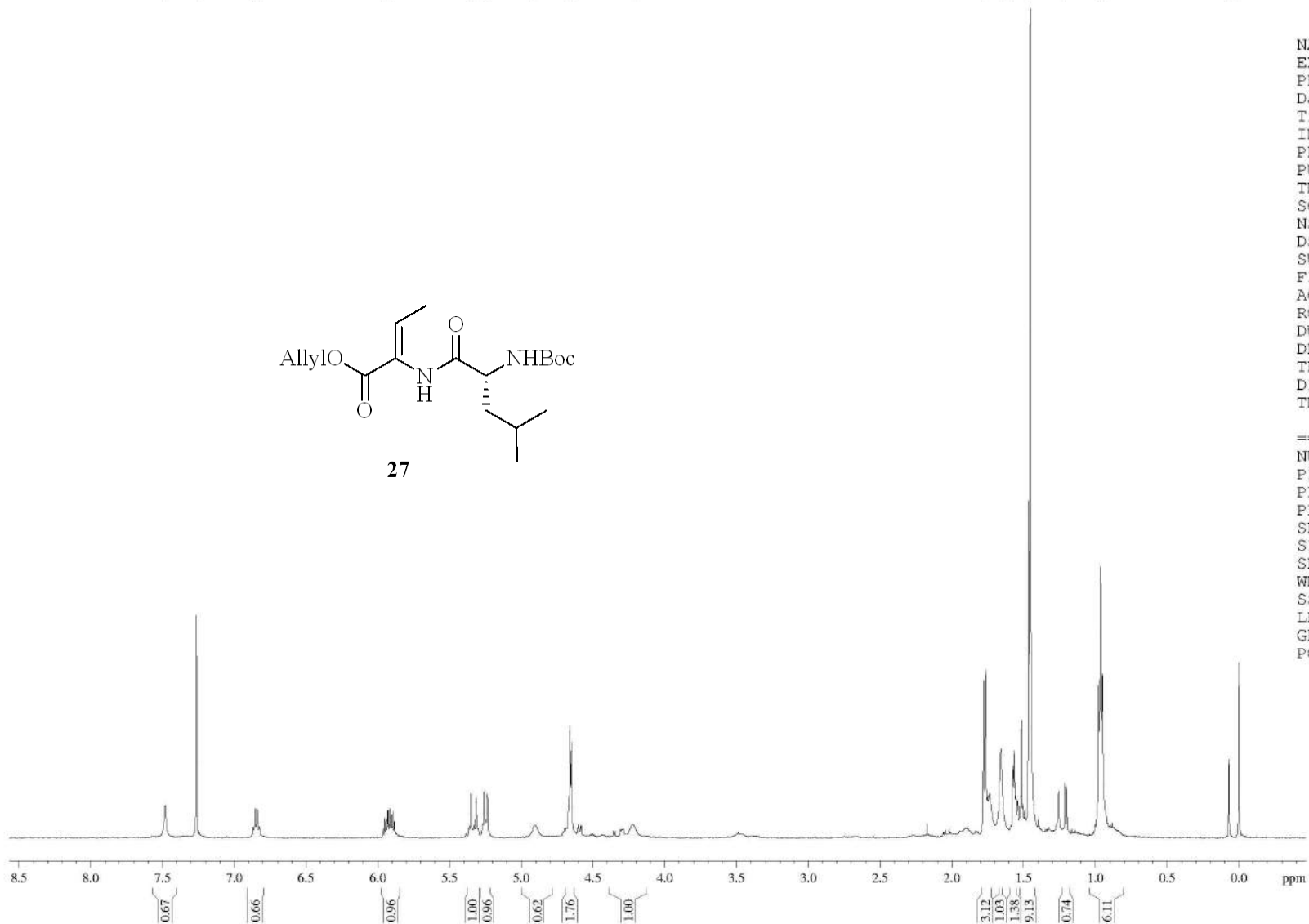


```

NAME          2012.08.06
EXPNO         1
PROCNO        1
Date_         20120806
Time          9.39
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            1
DS            2
SWH           10330.578
FIDRES        0.157632
AQ            3.1719923
RG            256
DW            48.400
DE            6.50
TE            298.6
D1            1.00000000
TD0           1
  
```

```

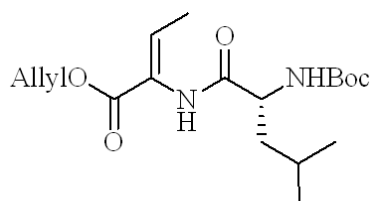
===== CHANNEL f1 =====
NUC1          1H
P1            13.00
PL1           2.00
PL1W          16.79986763
SFO1          500.1330885
SI            32768
SF            500.1300111
WDW           EM
SSB           0
LB            1.00
GB            0
PC            1.00
  
```



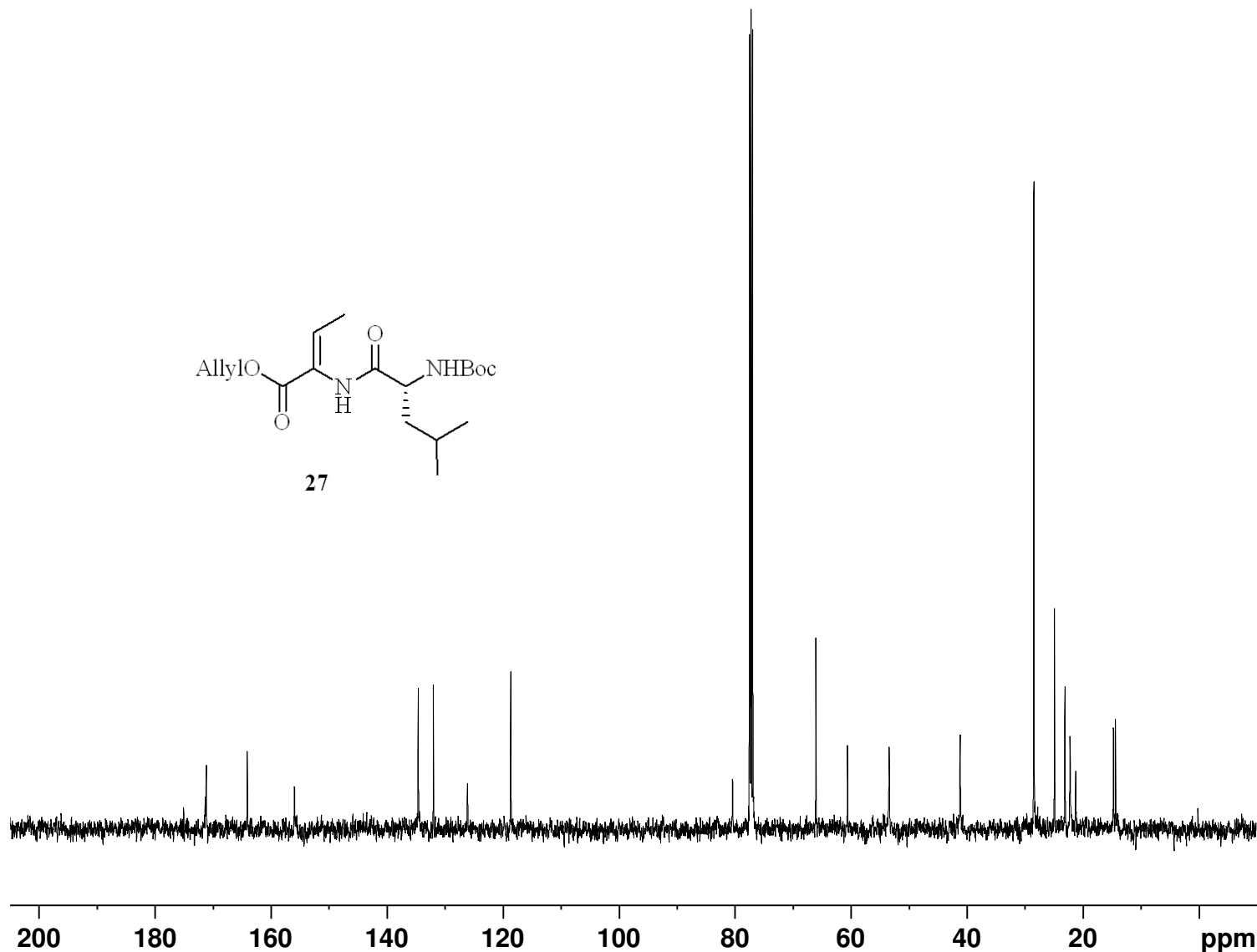
liushouxin-Thr-D-Leu

— 171.12
 — 164.06
 — 155.92
 — 134.57
 — 131.96
 — 126.07
 — 118.60

80.35
 77.40
 77.15
 76.89
 — 65.98
 — 53.34
 — 41.08
 28.38
 24.83
 23.02
 22.14
 — 14.64



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S68



Current Data Parameters
 NAME xuehong
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20211202
 Time 17.32
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 1024
 SOLVENT CDCl3
 NS 209
 DS 0
 SWH 29761.904 Hz
 FIDRES 3.633045 Hz
 AQ 0.1376256 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 293.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

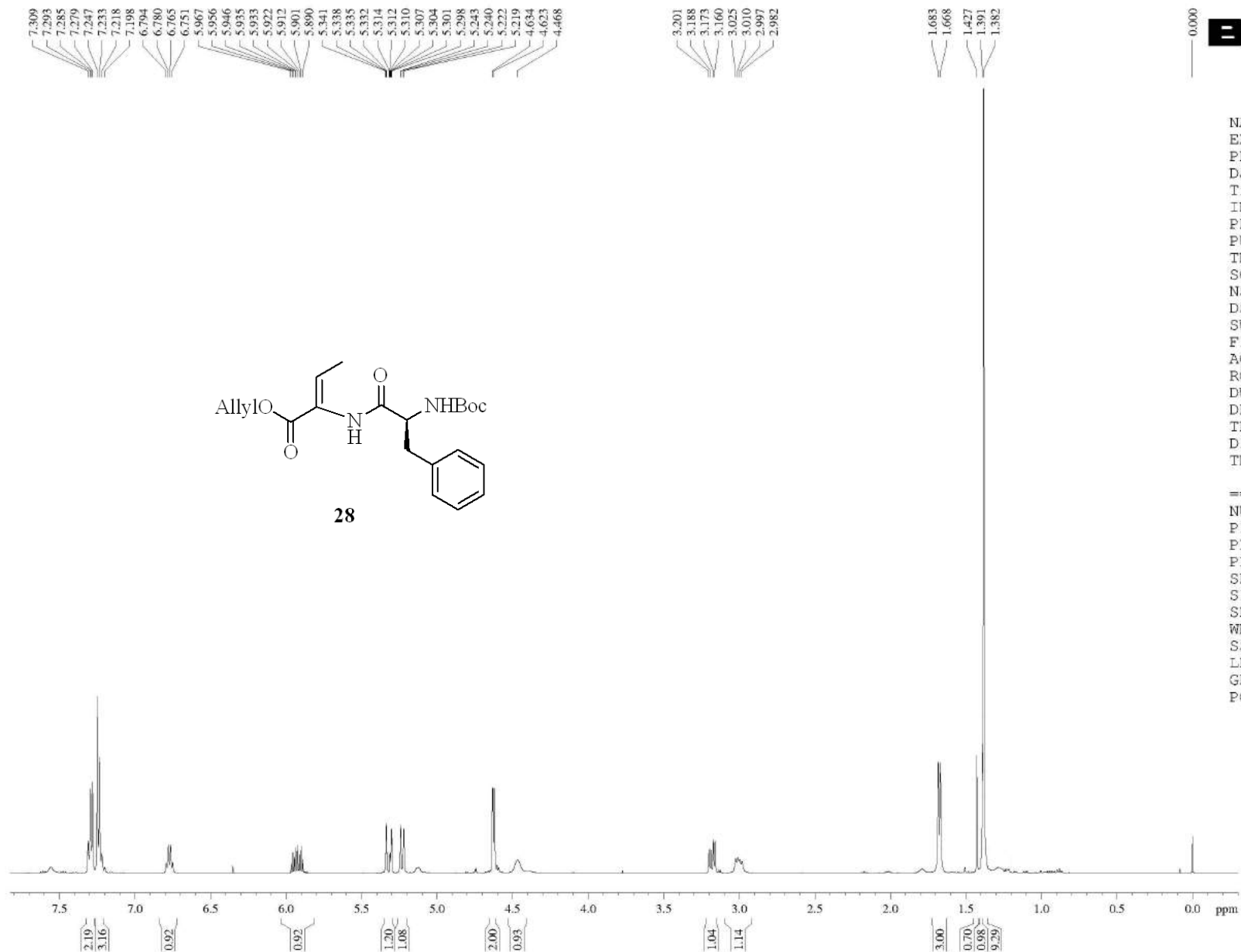
==== CHANNEL f1 =====
 NUC1 13C
 P1 12.30 usec
 PL1 4.50 dB
 PL1W 33.60015869 W
 SFO1 125.7703643 MHz

==== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PL12 17.78 dB
 PL13 17.78 dB
 PL2W 16.79986763 W
 PL12W 0.44392112 W
 PL13W 0.44392112 W
 SFO2 500.1320005 MHz

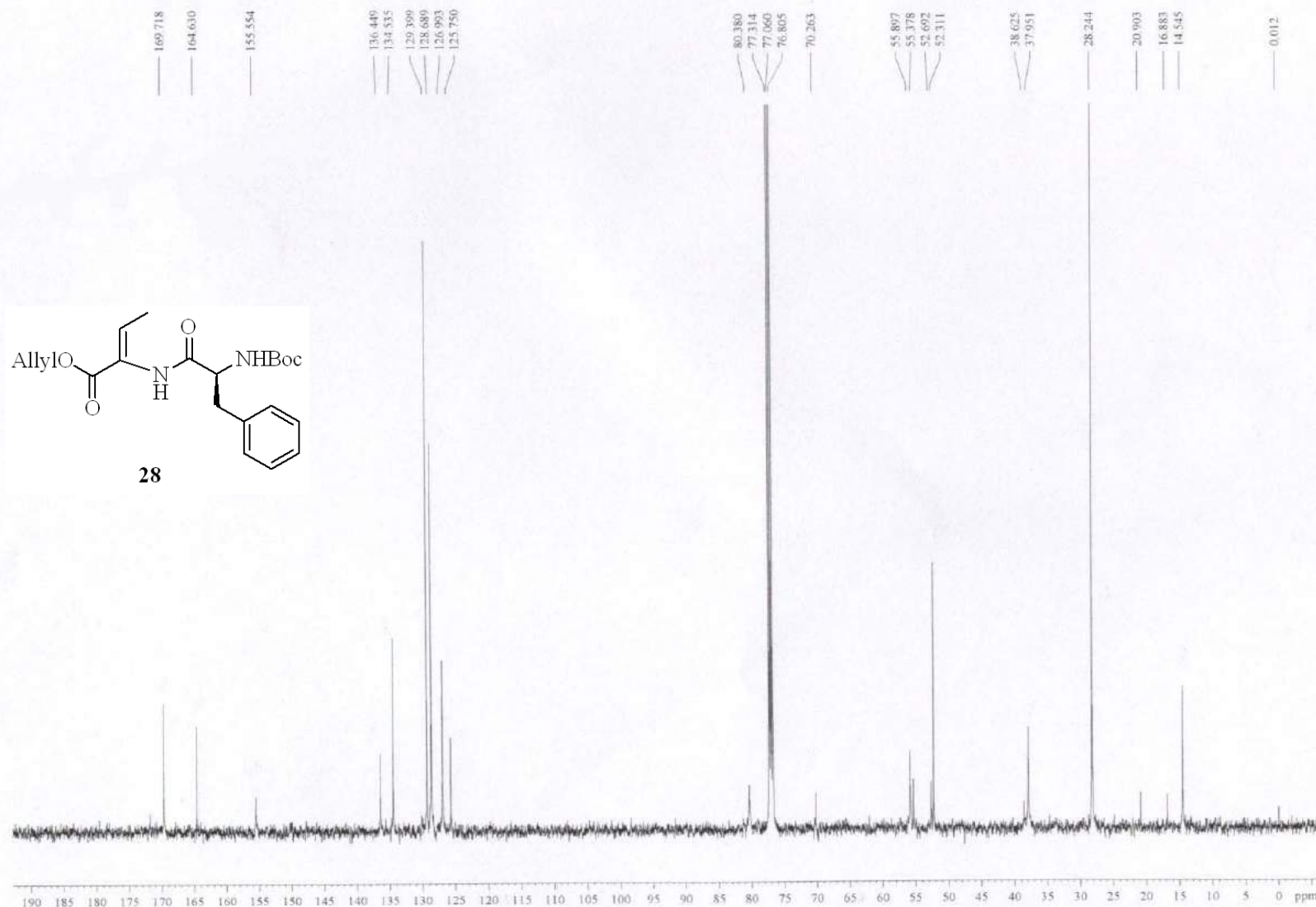
F2 - Processing parameters

SI 32768
 SF 125.7577765 MHz
 WDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.40

liushouxin-20170629-ZY



liushouxin-ertaituoshui



```

NAME      2013.04.18
EXPNO     21
PROCNO    1
Date_     20130418
Time      16.02
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         16384
SOLVENT   CDC13
NS         450
DS         4
SWH       29761.904 Hz
FIDRES    1.816522 Hz
AQ         0.2753012 sec
RG         2050
DW         16.800 usec
DE         6.50 usec
TE         293.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        12.30 usec
PL1       4.50 dB
PL1W      33.60015869 W
SFO1      125.7703643 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.00 dB
PL12      17.78 dB
PL13      17.78 dB
PL2W      16.79986763 W
PL12W     0.44392112 W
PL13W     0.44392112 W
SFO2      500.1320005 MHz
SI        32768
SF        125.7577890 MHz
WDW       EM
SSB       0
LB        3.00 Hz
GB        0
PC        1.40
    
```

liu shou xin Thr-His-H2O

7.83
 7.28
7.27
7.25
7.24
7.20
7.07
 5.87
5.86
5.84
5.83
5.82
5.81
5.80
5.79
5.26
5.22
5.18
5.16
4.68
4.67
4.59

3.51
3.49
3.48
3.46
3.28
3.25

1.68
1.66
1.52
1.51
1.41
1.40
1.39
1.37
1.35
1.34

-0.00
-0.07

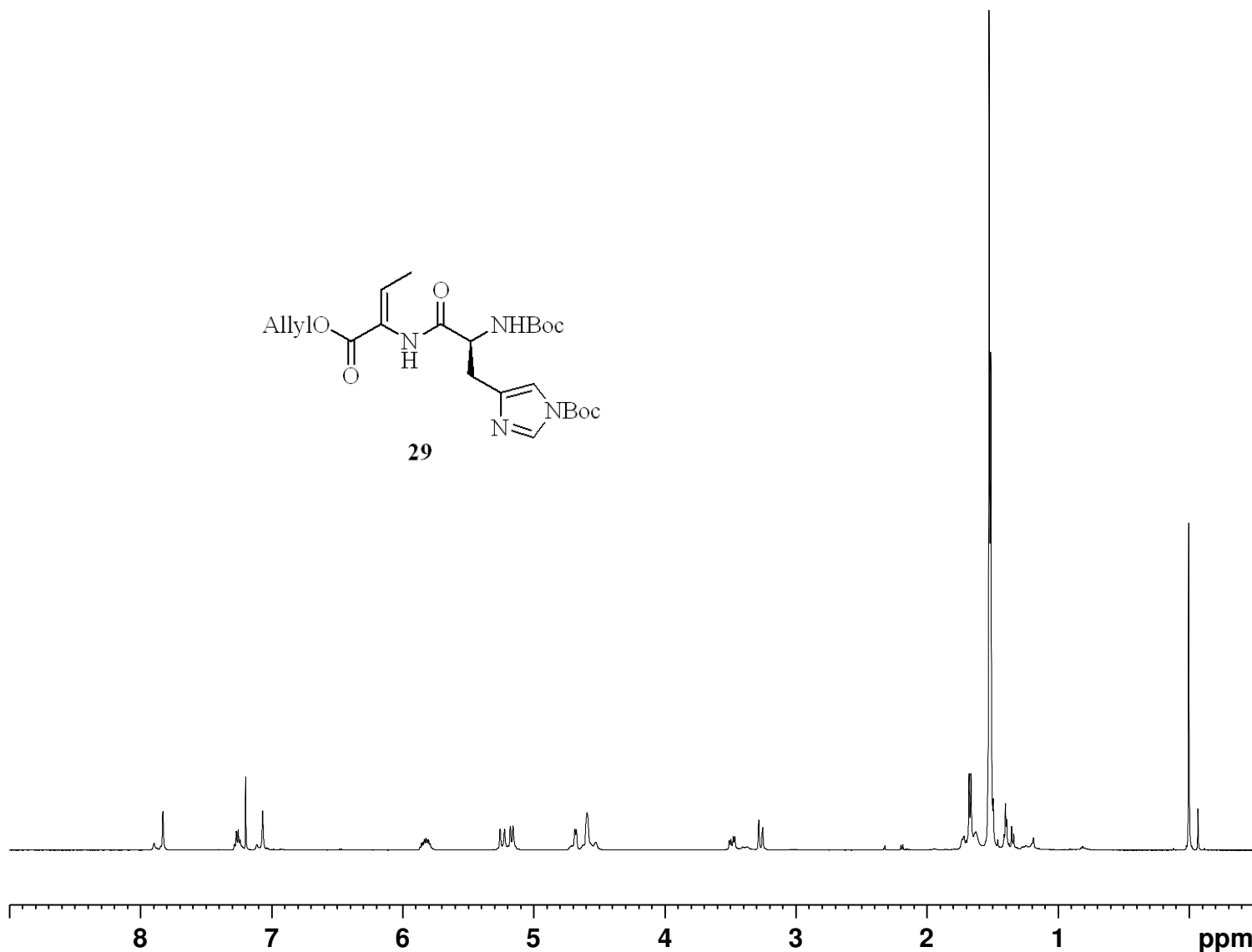
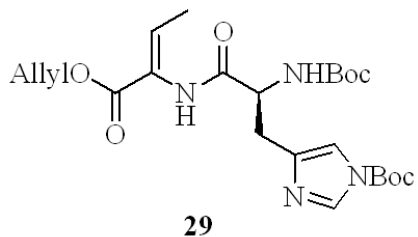


Current Data Parameters
 NAME xuehong
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170802
 Time 17.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719425 sec
 RG 181
 DW 48.400 usec
 DE 6.50 usec
 TE 297.7 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 2.00 dB
 PL1W 16.79986763 W
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300451 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



0.73
0.86
0.66
0.80

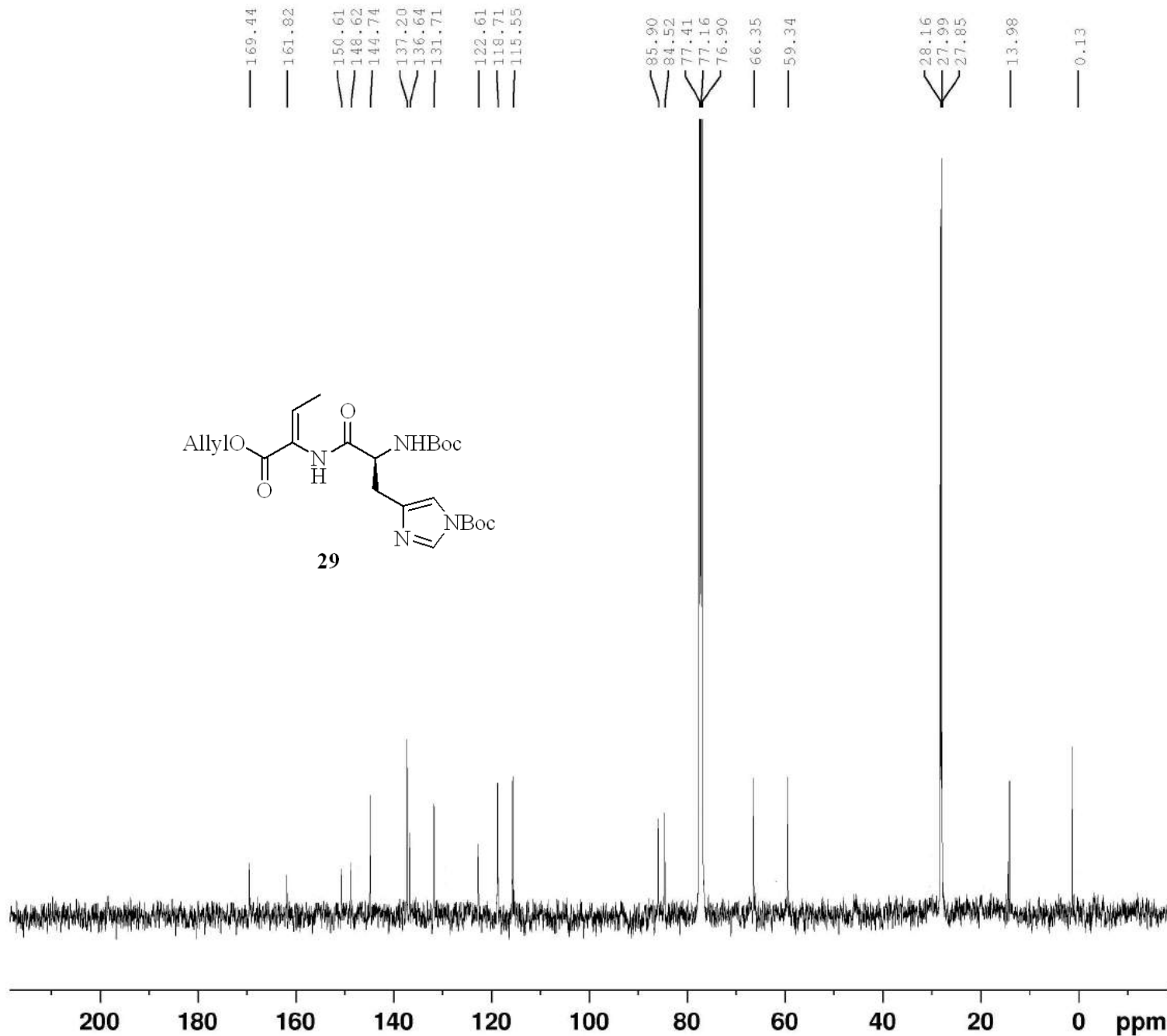
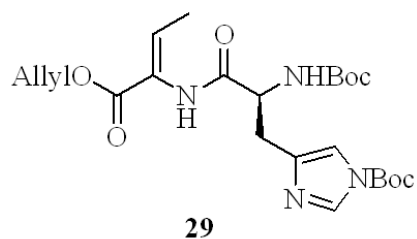
1.00
0.95
1.02
0.95
2.07

0.99
1.01

S71

3.25
18.11
1.89

liushouxin-Thr-L-His



```

Current Data Parameters
NAME                xuehong
EXPNO                8
PROCNO              1

F2 - Acquisition Parameters
Date_                20211202
Time                 10.48
INSTRUM              spect
PROBHD               5 mm PABBO BB-
PULPROG              zgpg30
TD                   2048
SOLVENT              CDCl3
NS                   597
DS                   0
SWH                  29761.904 Hz
FIDRES               1.816522 Hz
AQ                   0.2752512 sec
RG                   2050
DW                   16.800 usec
DE                   6.50 usec
TE                   293.7 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                  1

===== CHANNEL f1 =====
NUC1                  13C
P1                    12.30 usec
PL1                   4.50 dB
PL1W                  33.60015869 W
SFO1                  125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG[2]             waltz16
NUC2                   1H
PCPD2                 80.00 usec
PL2                   2.00 dB
PL12                  17.78 dB
PL13                  17.78 dB
PL2W                  16.79986763 W
PL12W                 0.44392112 W
PL13W                 0.44392112 W
SFO2                  500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF                    125.7577739 MHz
WDW                   EM
SSE                   0
LB                    5.00 Hz
GB                   0
PC                    1.40
    
```

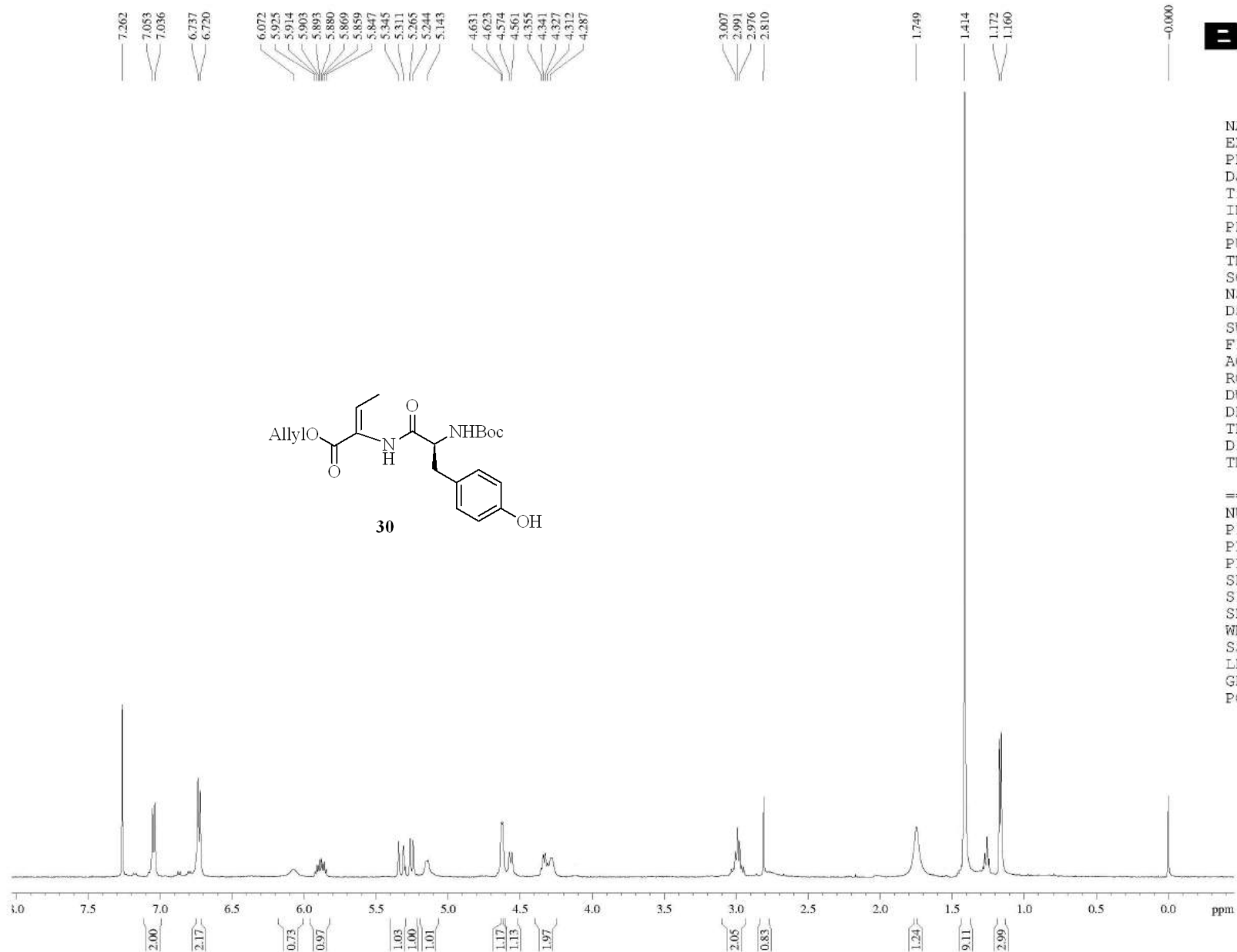
liu shou xin

Boc-Tyr-Thr-Allyl



NAME 2012.07.25
EXPNO 7
PROCNO 1
Date_ 20120724
Time 21.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 2
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 90.5
DW 48.400
DE 6.50
TE 299.4
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1300124
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00

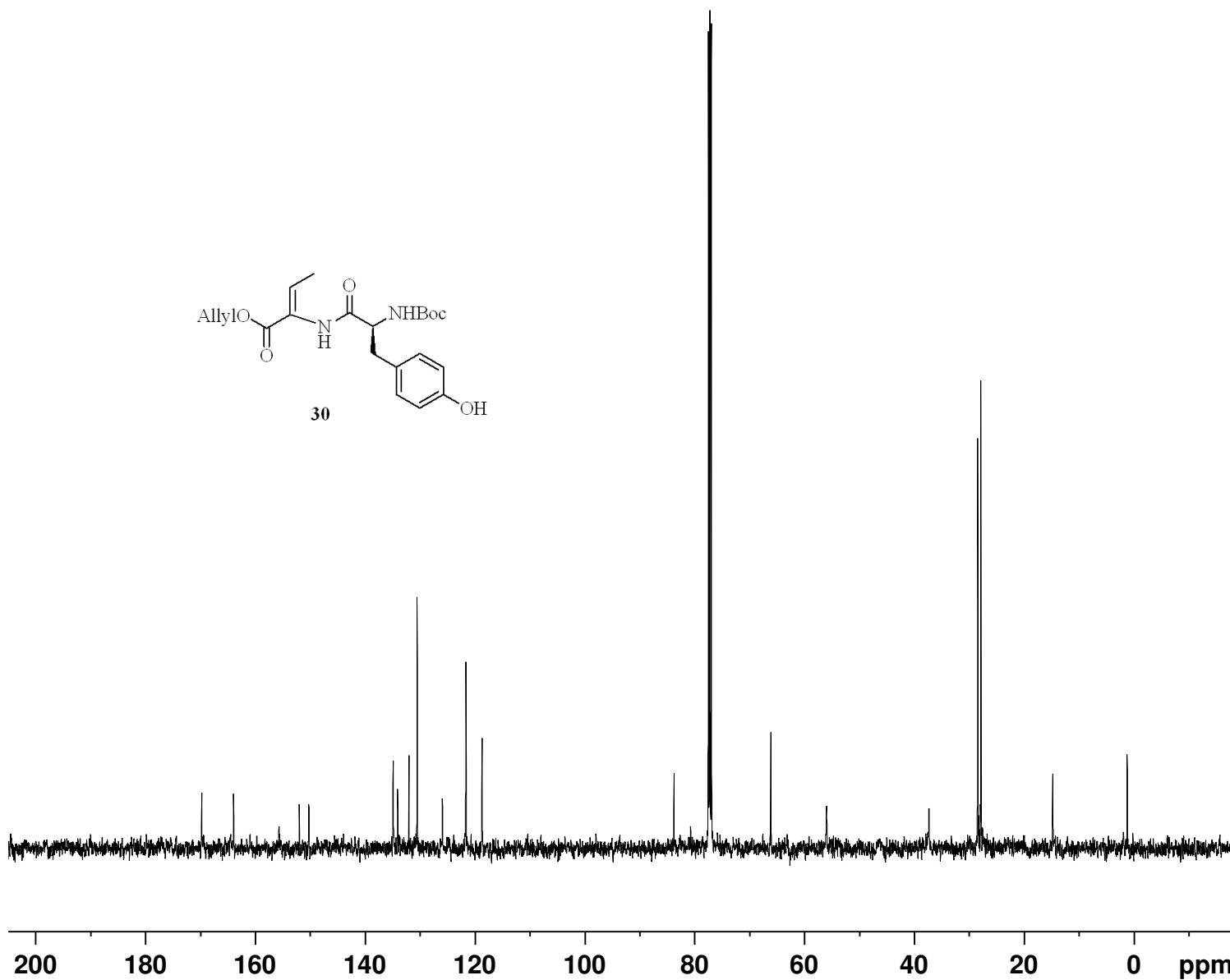
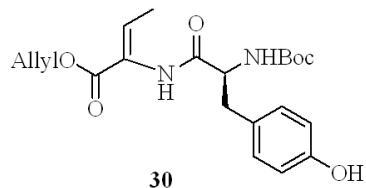


liushouxin-Thr-L-Tyr



169.71
163.92
151.95
150.20
134.85
134.03
131.96
130.48
125.88
121.59
118.65

83.69
77.41
77.16
76.90
66.03
55.87
37.24
28.36
27.81
14.70



Current Data Parameters
NAME xuehong
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20211103
Time 9.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 2048
SOLVENT CDC13
NS 205
DS 0
SWH 29761.904 Hz
FIDRES 3.633045 Hz
AQ 0.1376256 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577754 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

liu shou xin Thr-Pro

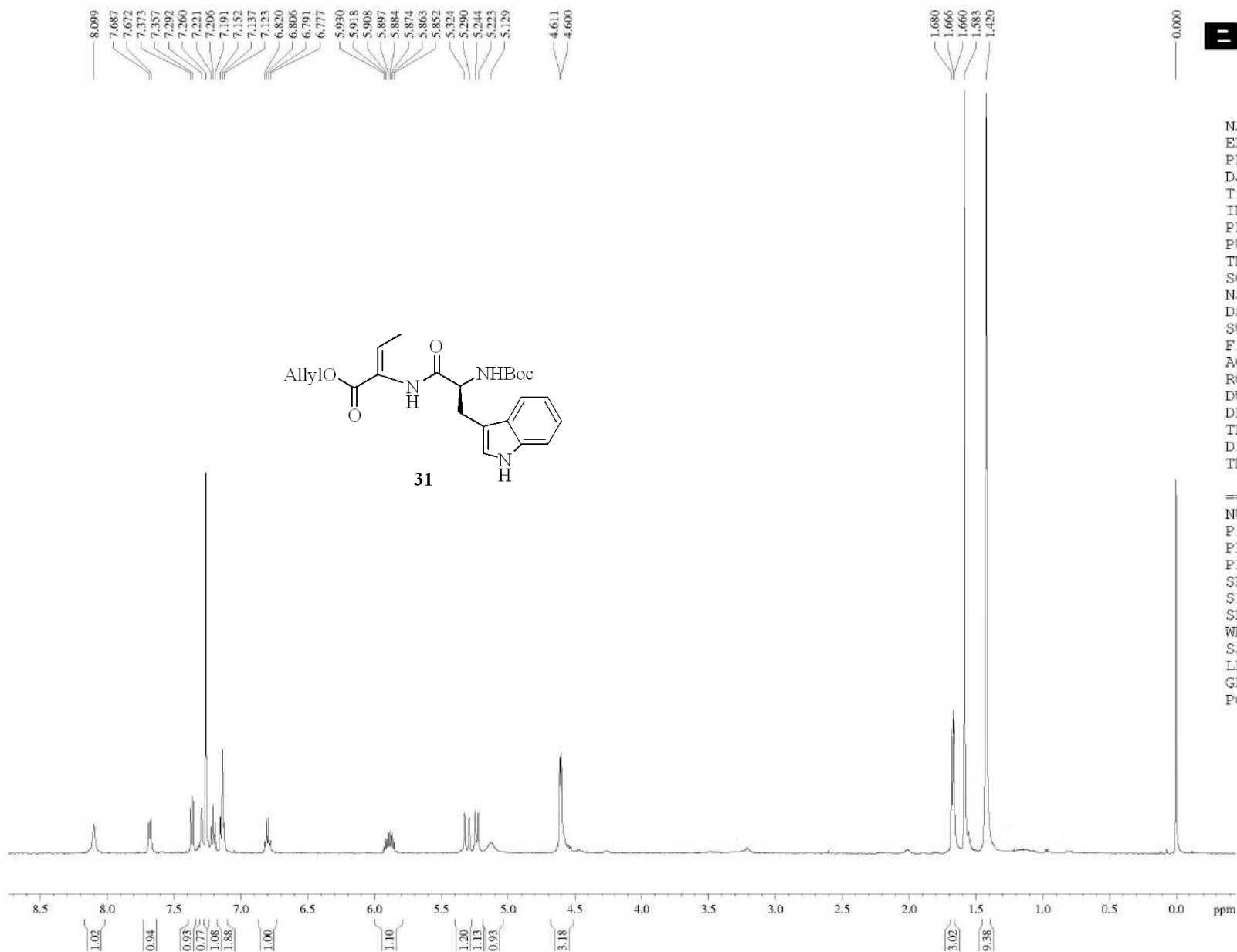
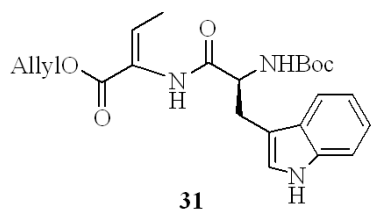


```

NAME      2012.08.06
EXPNO     5
PROCNO    1
Date_     20120806
Time      10.16
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS         8
DS         2
SWH       10330.578
FIDRES    0.157632
AQ        3.1719923
RG         406
DW        48.400
DE         6.50
TE        298.8
D1        1.00000000
TD0       1
    
```

```

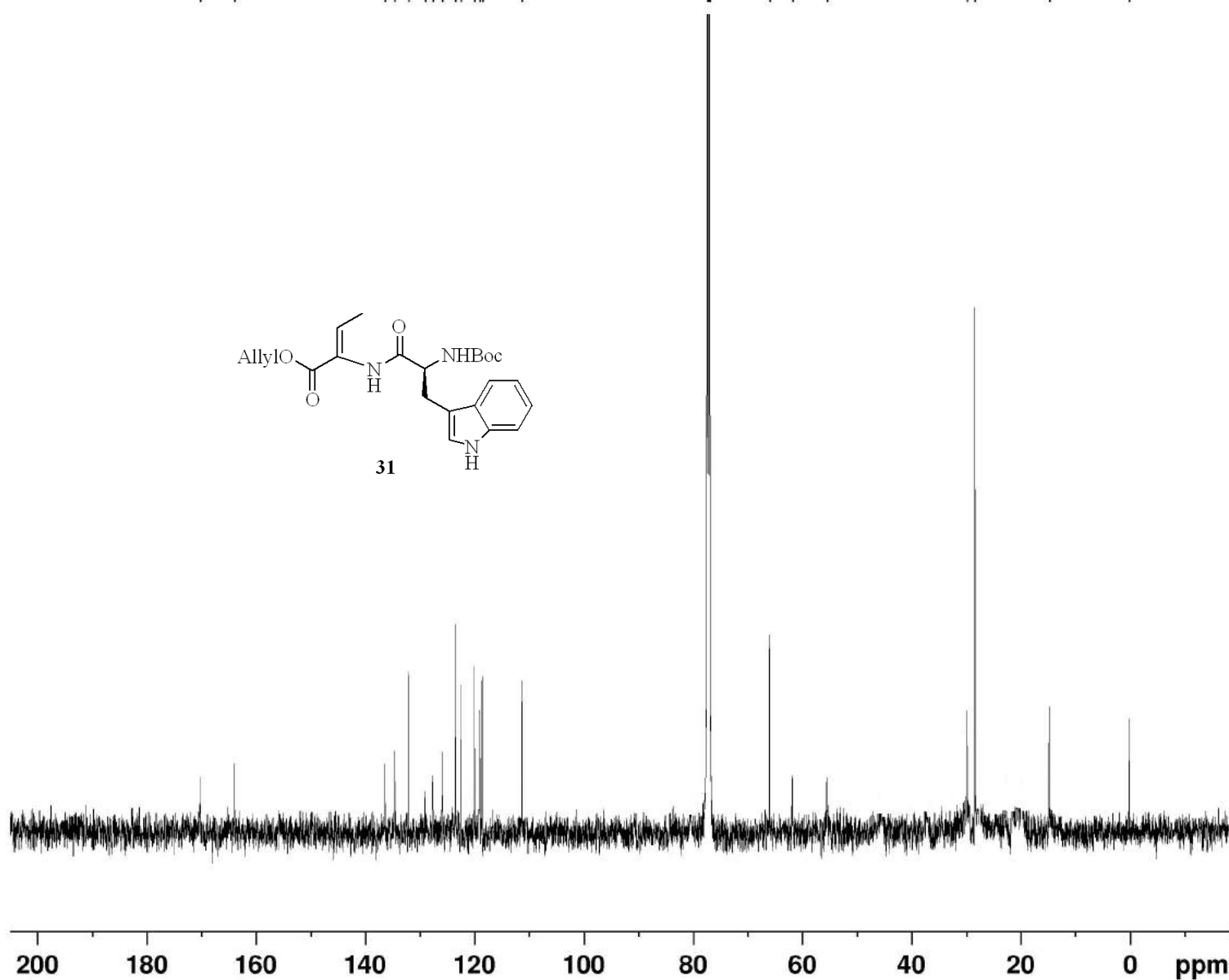
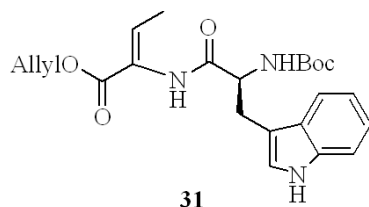
===== CHANNEL f1 =====
NUC1      1H
P1        13.00
PL1       2.00
PL1W     16.79986763
SFO1     500.1330885
SI        32768
SF        500.1300133
WDW       EM
SSB       0
LB        1.00
GB        0
PC        1.00
    
```



liushouxin--Thr-L-Trp

170.26
170.14
163.95
136.34
134.63
132.01
129.02
127.67
125.91
123.50
122.44
119.96
119.00
118.59
111.32

77.41
77.16
76.90
65.99
61.79
55.44
29.84
28.40
14.72
0.14



Current Data Parameters
NAME xuehong
EXPNO 9
PROCNO 1

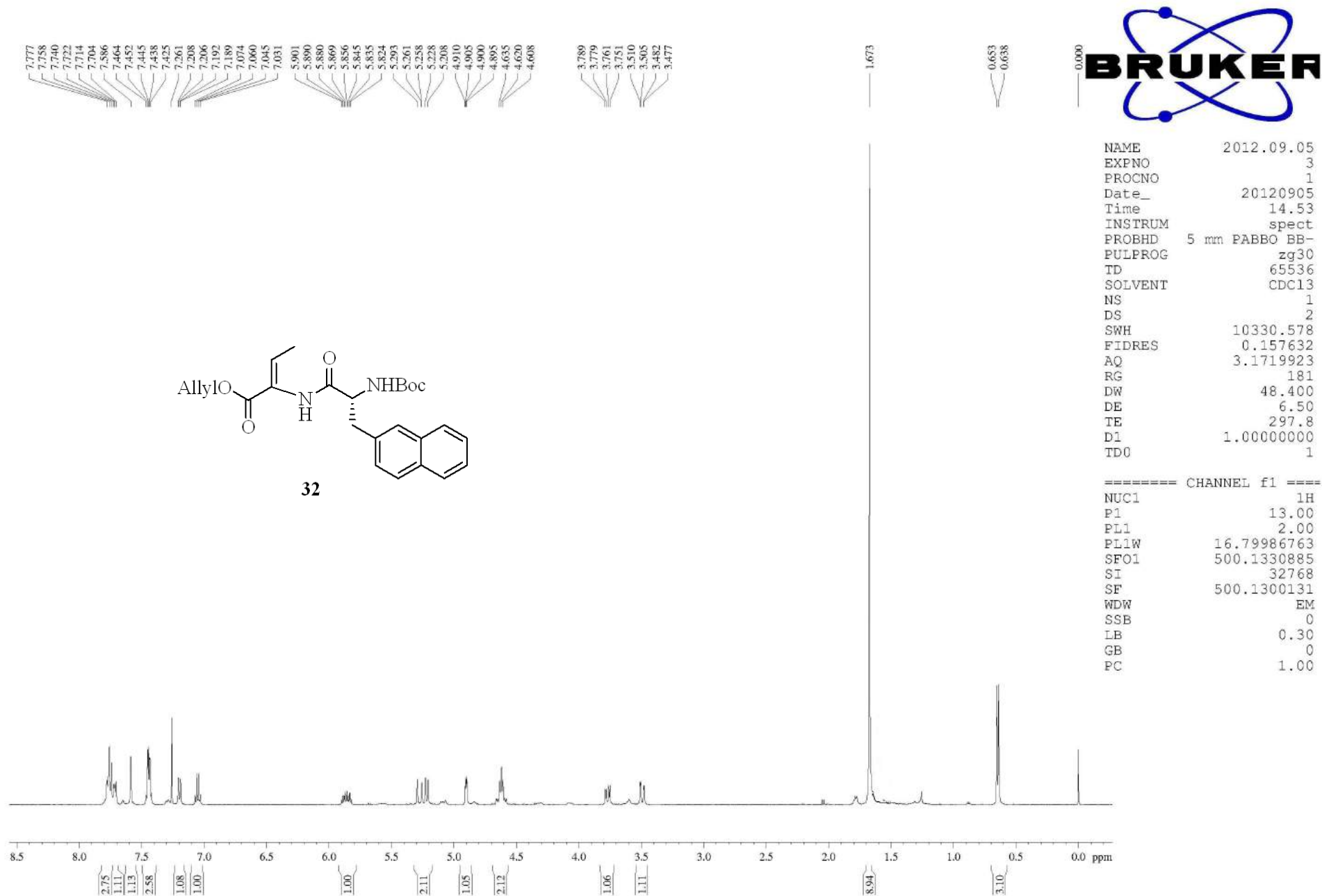
F2 - Acquisition Parameters
Date_ 20211202
Time 14.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 2048
SOLVENT CDCl3
NS 2048
DS 0
SWH 29761.904 Hz
FIDRES 1.816522 Hz
AQ 0.2752512 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG12 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577734 MHz
WDW EM
SSE 0
LB 3.00 Hz
GB 0
PC 1.40

liu shou xin Thr-D-Nap-H2O1

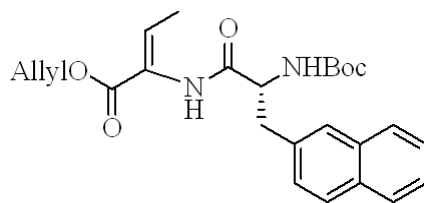


liushouxin-Thr-D-Nap

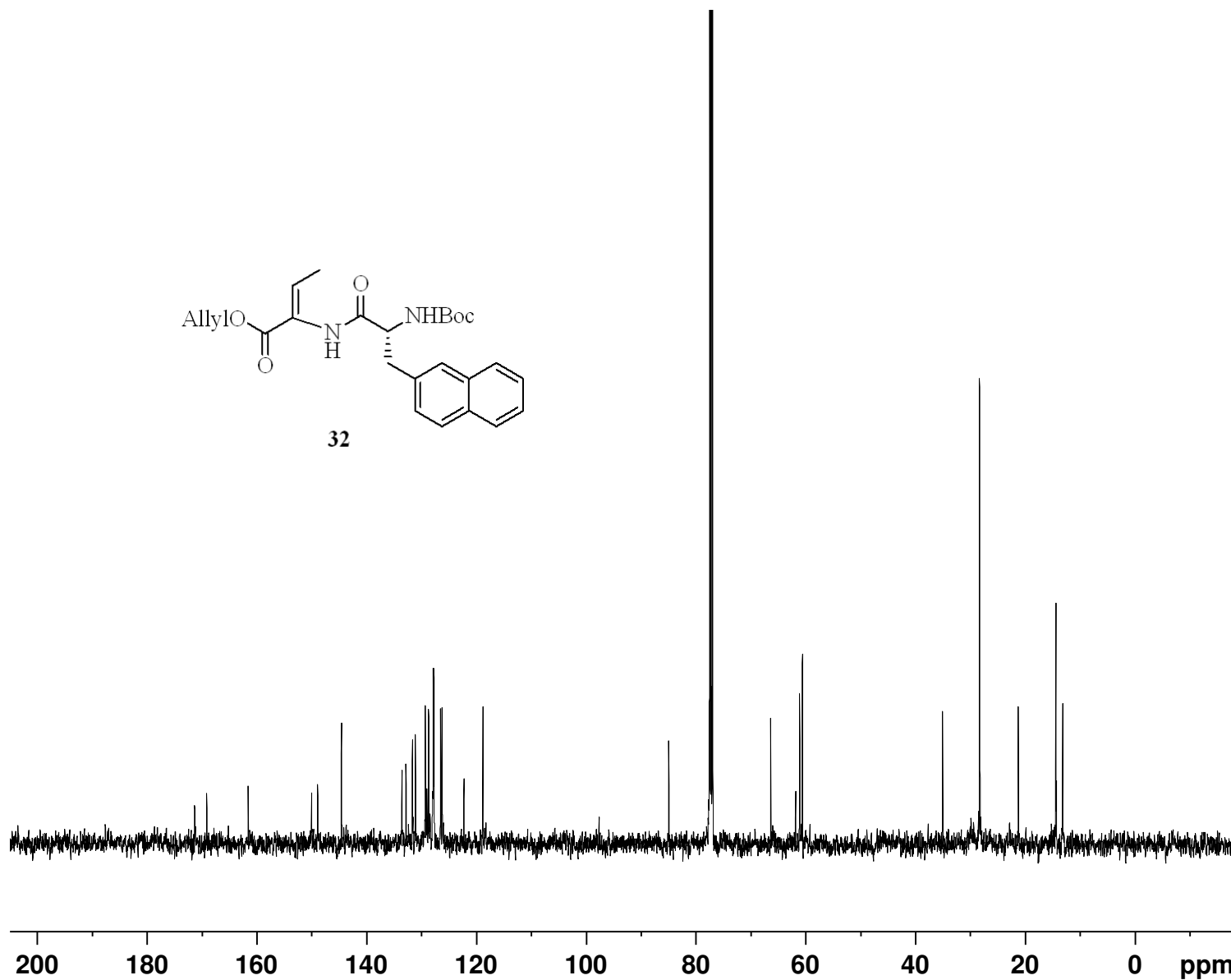


171.28
169.08
161.54
149.97
148.87
144.53
133.51
132.78
131.60
131.06
129.25
128.64
127.82
127.73
127.66
126.48
126.21
122.20
118.73

84.88
77.41
77.16
76.90
66.31
61.02
60.52
34.97
28.24
21.18
14.31
13.07



32



Current Data Parameters
NAME xuehong
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20211103
Time 9.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 4096
SOLVENT CDC13
NS 153
DS 0
SWH 29761.904 Hz
FIDRES 3.633045 Hz
AQ 0.1376256 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz

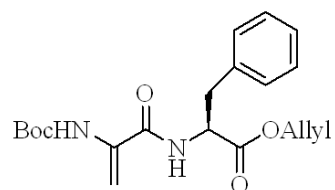
F2 - Processing parameters
SI 32768
SF 125.7577763 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

liushouxin-2018.6.11-ZY

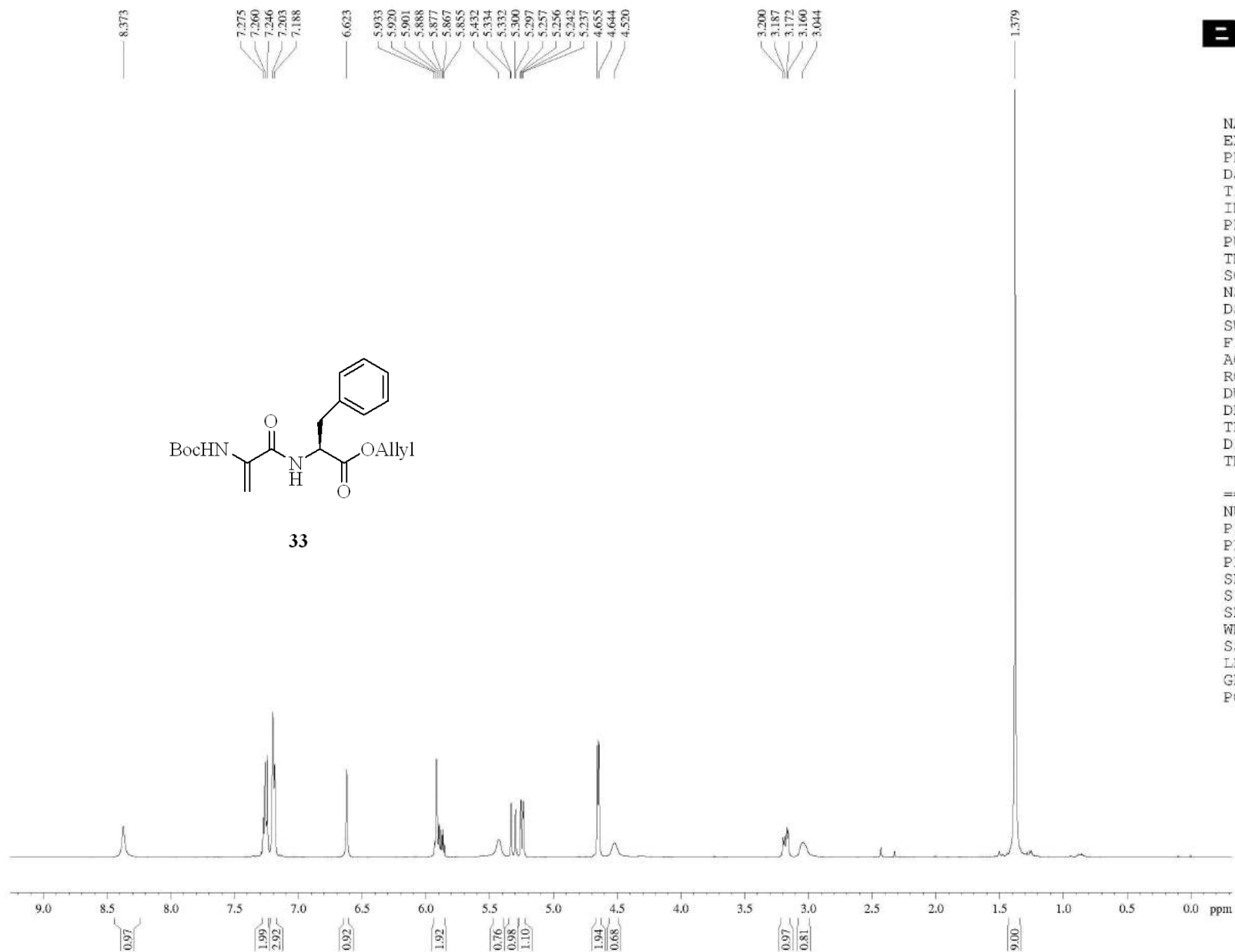


NAME 2018.06.14
EXPNO 12
PROCNO 1
Date_ 20180614
Time 20.44
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 14.2
DW 48.400
DE 6.50
TE 297.7
D1 1.00000000
TD0 1

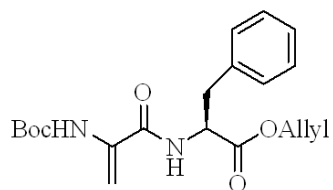
===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1300063
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00



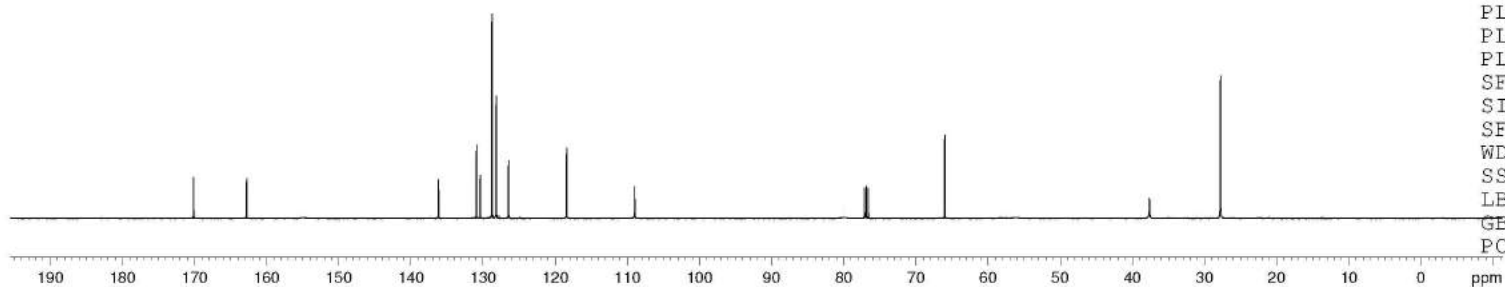
33



liushouxin-2018-6-11-zy



33



NAME 2018.06.23
EXPNO 10
PROCNO 1
Date_ 20180623
Time 16.51
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 512
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

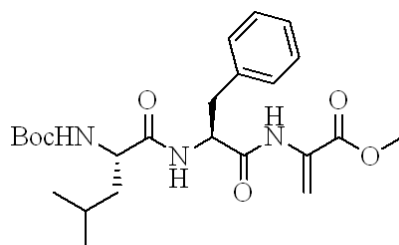
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578395 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

liushouxin-2018-5-26-ZY-3

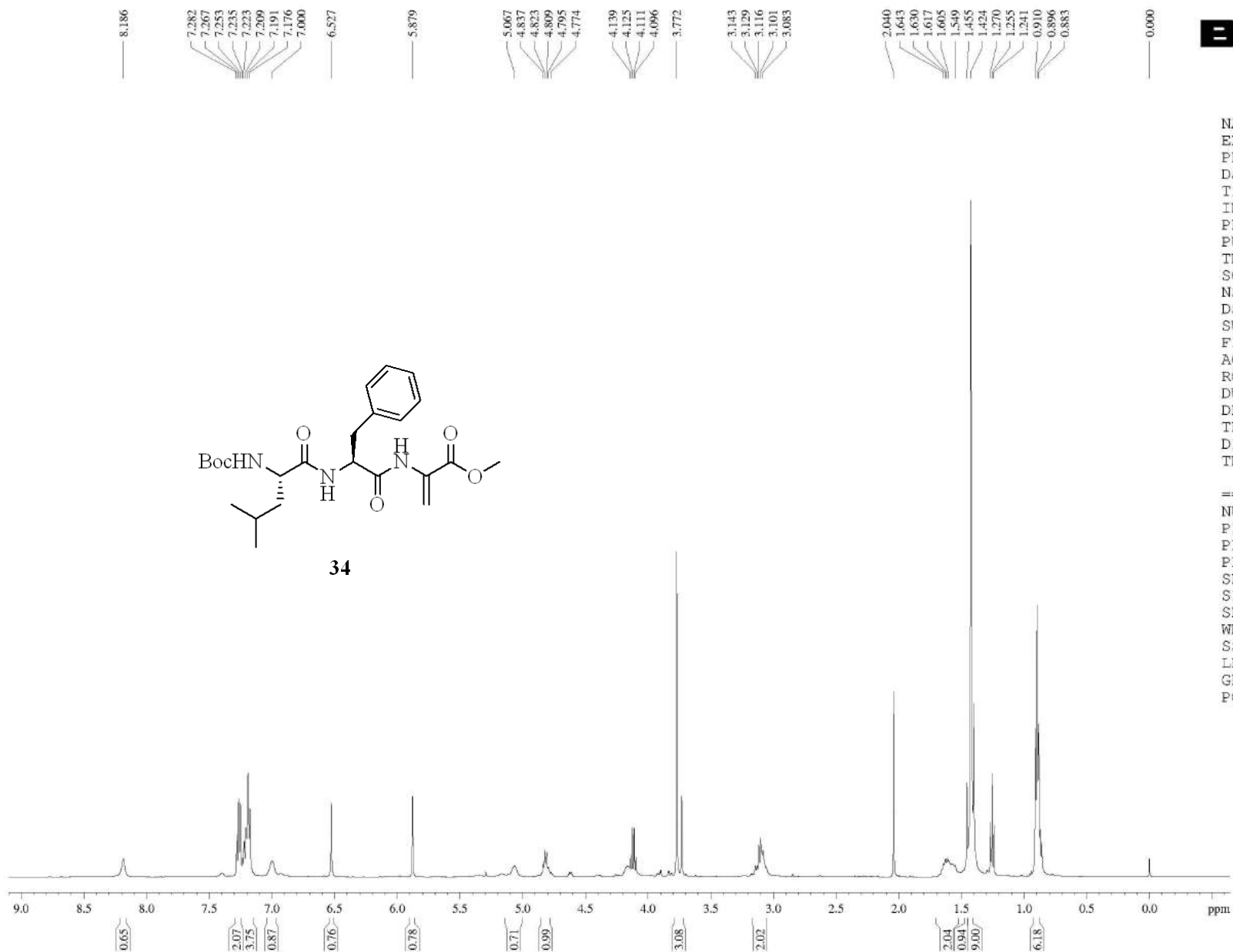


NAME 2018.05.30
EXPNO 21
PROCNO 1
Date_ 20180530
Time 12.29
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 45.2
DW 48.400
DE 6.50
TE 297.8
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1300115
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00

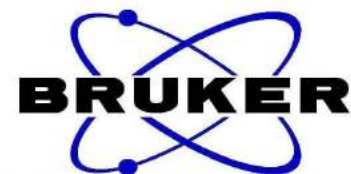


34

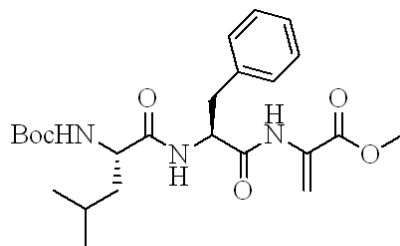


1-liushouxin-2018-5-26-zy-3

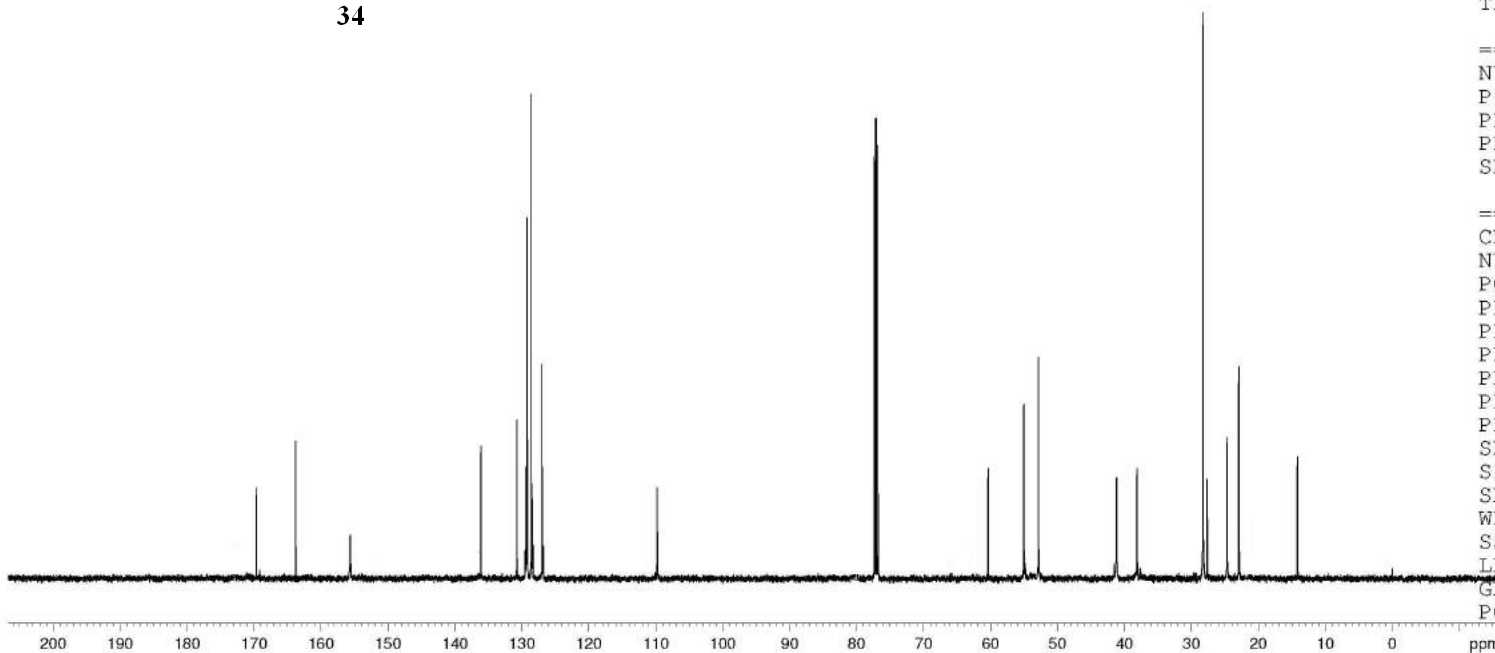
169.70
163.84
155.68
136.17
130.80
129.26
128.64
127.02
109.83
77.36
77.10
76.85
60.40
55.06
52.87
41.21
38.16
28.29
27.67
24.70
22.94
14.19



NAME 2018.06.08
EXPNO 20
PROCNO 1
Date_ 20180608
Time 17.58
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 512
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1



34



==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

liushouxin-ZY-2019.1.22

7.248
7.236
7.222
7.199
7.191
7.186
7.174
6.858
6.841

6.185
5.744
5.726

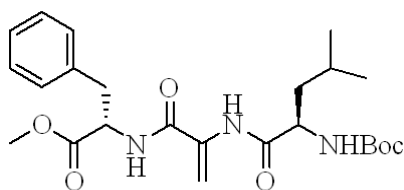
4.771
4.754
4.745
4.728

3.780
3.649
3.633
3.620
3.354
3.061
3.051
3.033
3.023
3.003
2.995
2.883
2.864
2.856
2.836
2.799
2.788
2.779
2.771
2.761
2.752
2.500

1.482
1.454
1.443
1.415
1.359
1.305
1.295
1.286
1.275
1.247
1.234
0.828
0.815
0.790
0.778

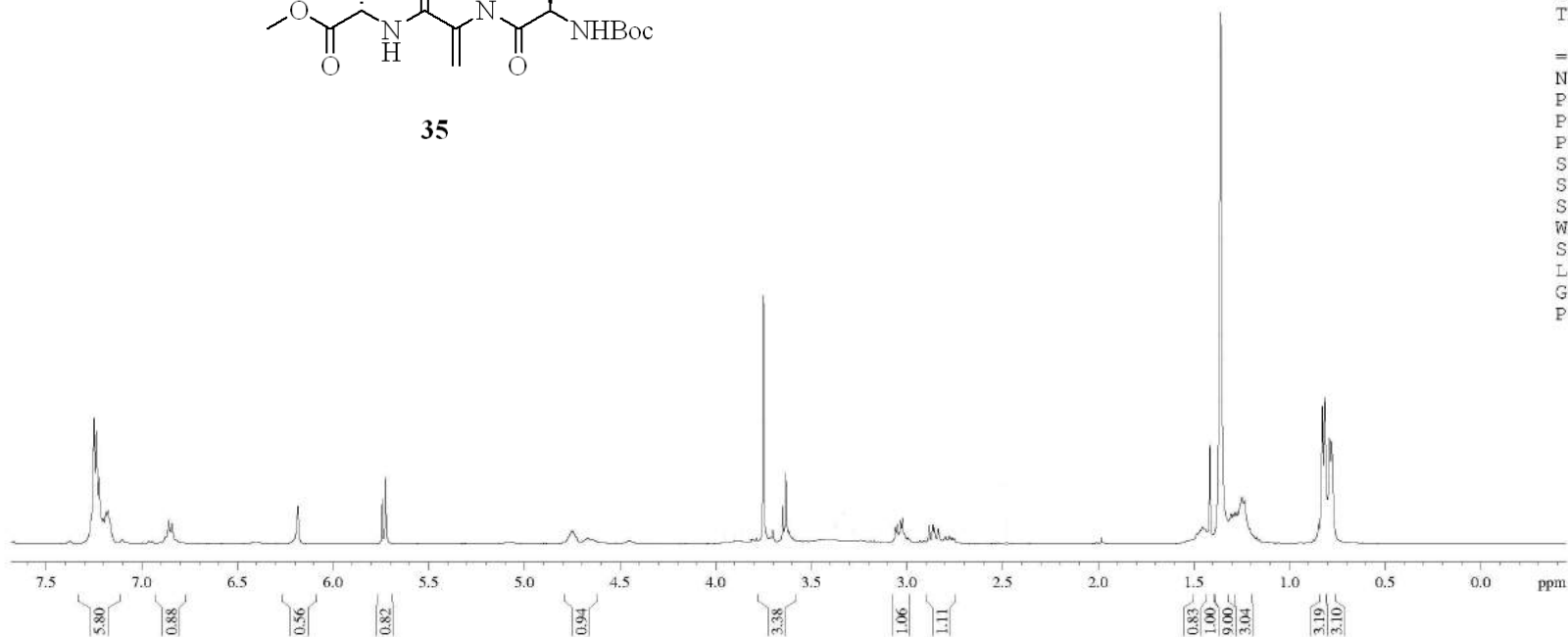


NAME 2019.01.22
EXPNO 5
PROCNO 1
Date_ 20190122
Time 17.29
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 10330.578
FIDRES 0.157632
AQ 3.1719923
RG 80.6
DW 48.400
DE 6.50
TE 298.0
D1 1.00000000
TD0 1



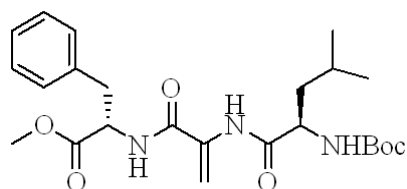
35

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1330885
SI 32768
SF 500.1300092
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00



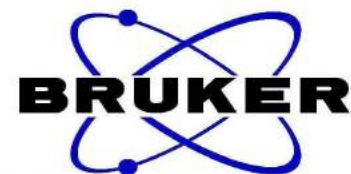
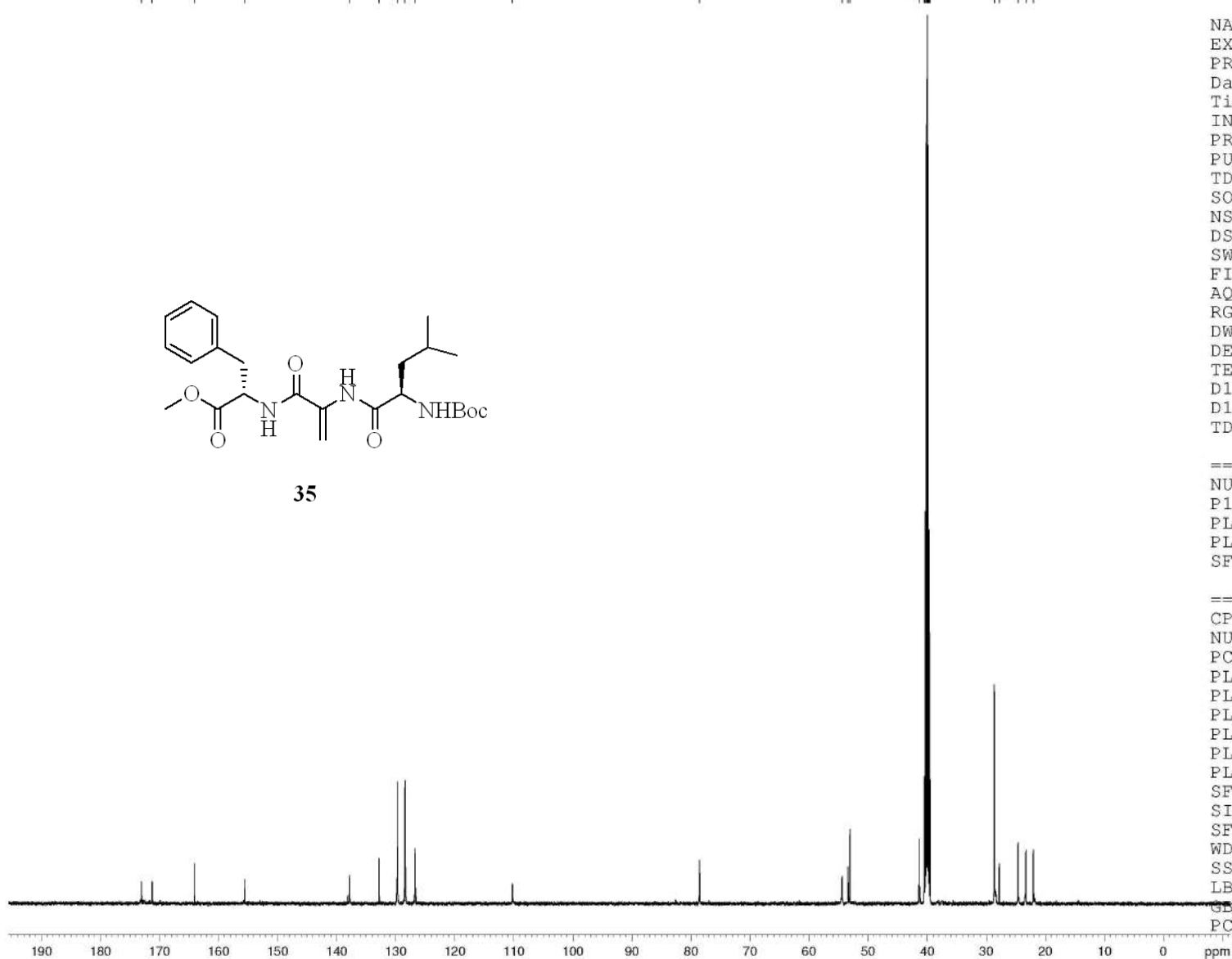
liushouxin-ZY-2019.1.22

173.06
171.26
164.12
155.62
137.81
132.83
129.72
128.44
126.74
110.25
78.54



35

54.40
53.42
53.09
41.30
40.49
40.32
40.15
39.99
39.82
39.65
39.49
28.63
27.78
24.62
23.29
22.03



NAME 2019.01.22
EXPNO 7
PROCNO 1
Date_ 20190122
Time 18.59
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 1279
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 912
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

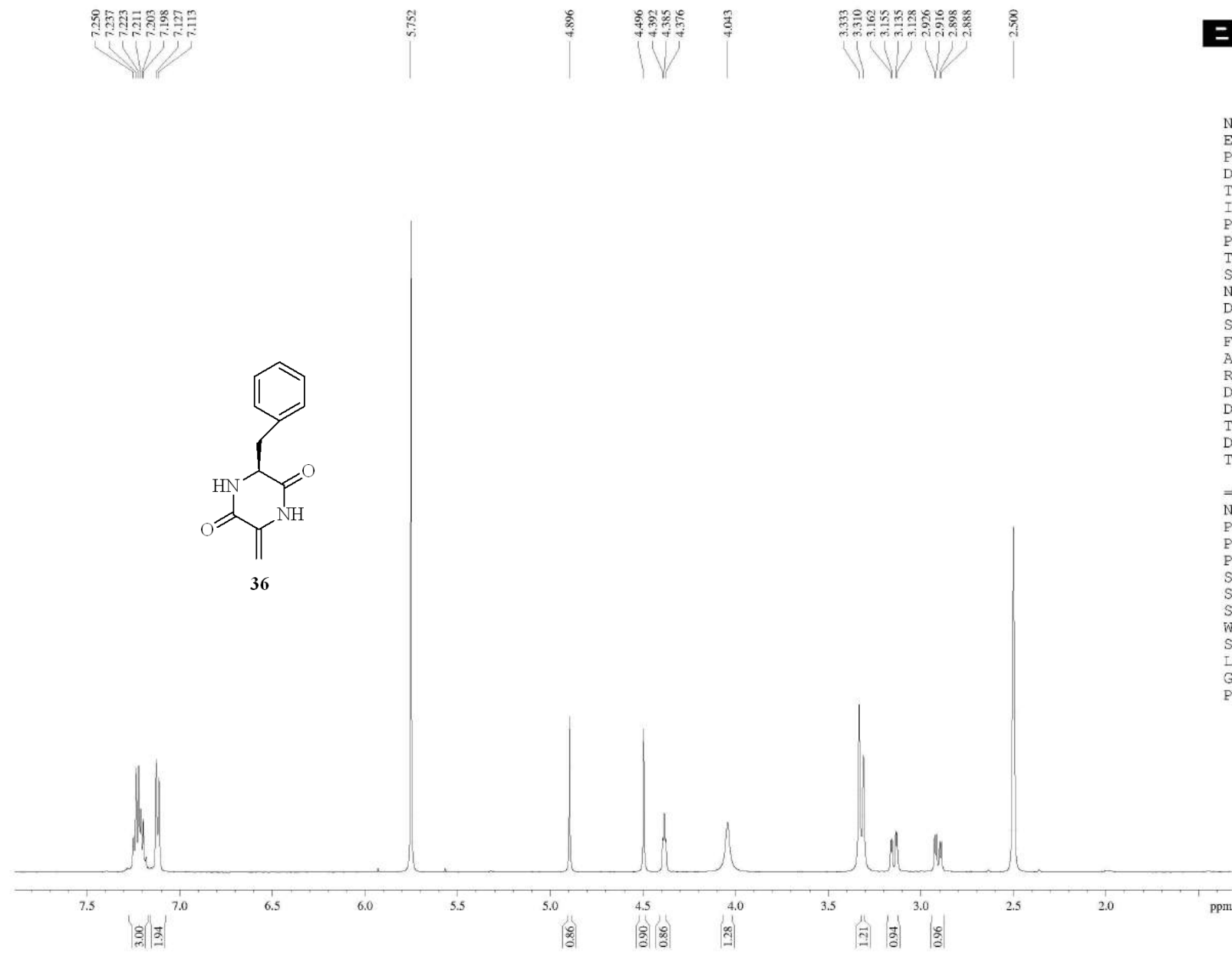
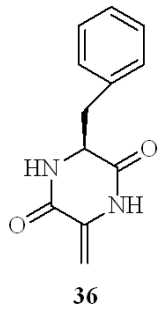
==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

2018-10-11-zy



NAME 2018.10.11
EXPNO 3
PROCNO 1
Date_ 20181011
Time 18.19
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 7500.000
FIDRES 0.114441
AQ 4.3691168
RG 322
DW 66.667
DE 6.50
TE 296.5
D1 1.00000000
TD0 1

==== CHANNEL f1 ====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1330008
SI 32768
SF 500.1330094
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00

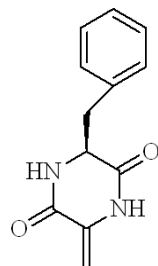


liushouxin-2018-10-11-zy

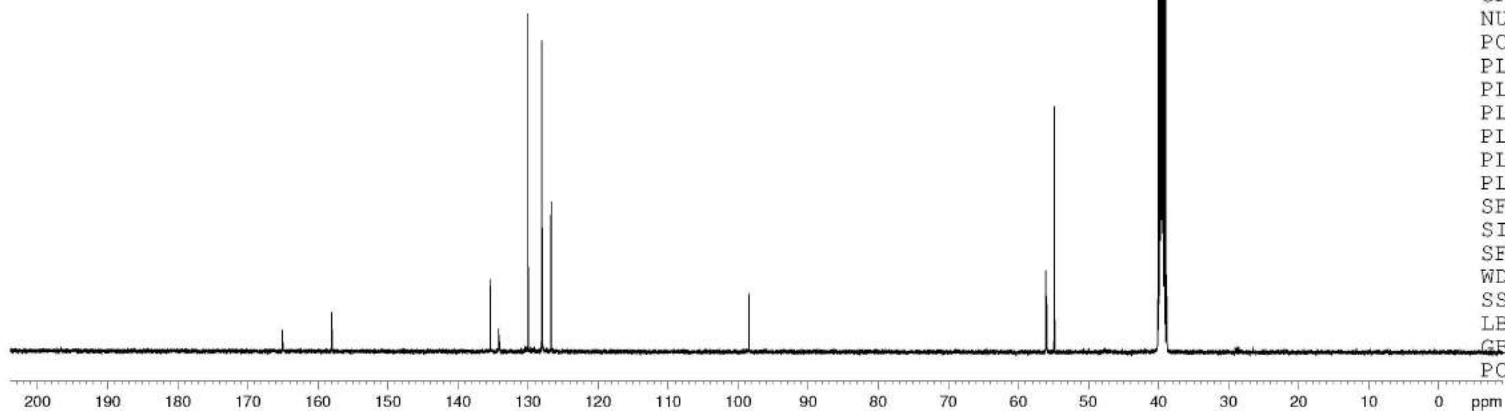
165.11
158.06
135.40
134.21
130.03
128.04
126.72

98.50

56.09
55.99
54.90
40.02
39.86
39.69
39.52
39.36
39.19
39.02



36



NAME 2018.10.19
EXPNO 1
PROCNO 1
Date_ 20181020
Time 6.54
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 13312
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1150
DW 16.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

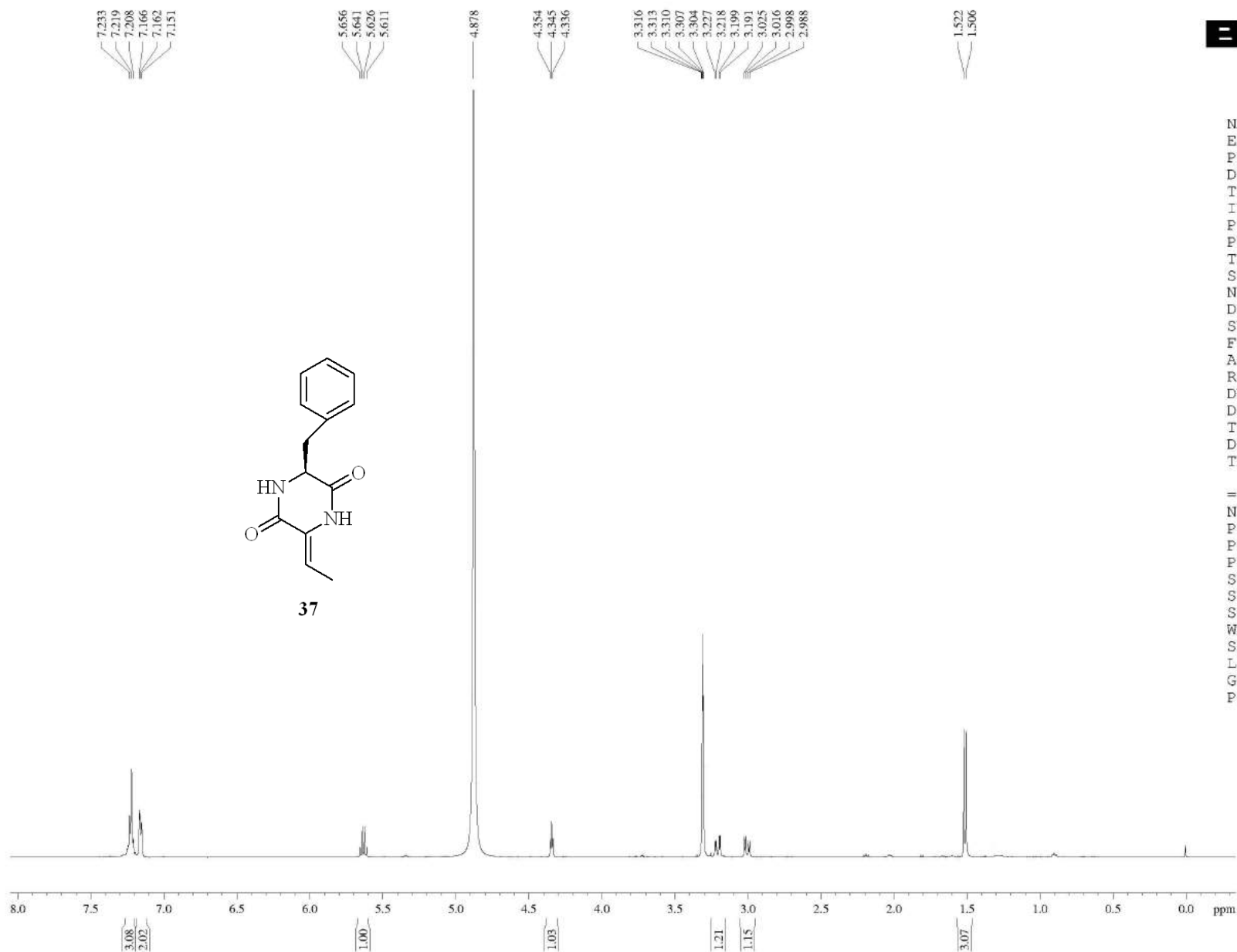
==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578489 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ZY-2018-10-29

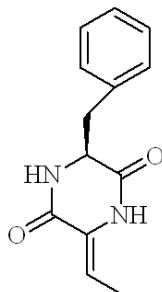


NAME 2018.10.30
EXPNO 6
PROCNO 1
Date_ 20181030
Time 13.12
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 256
DW 50.000
DE 6.50
TE 294.9
D1 1.00000000
TD0 1

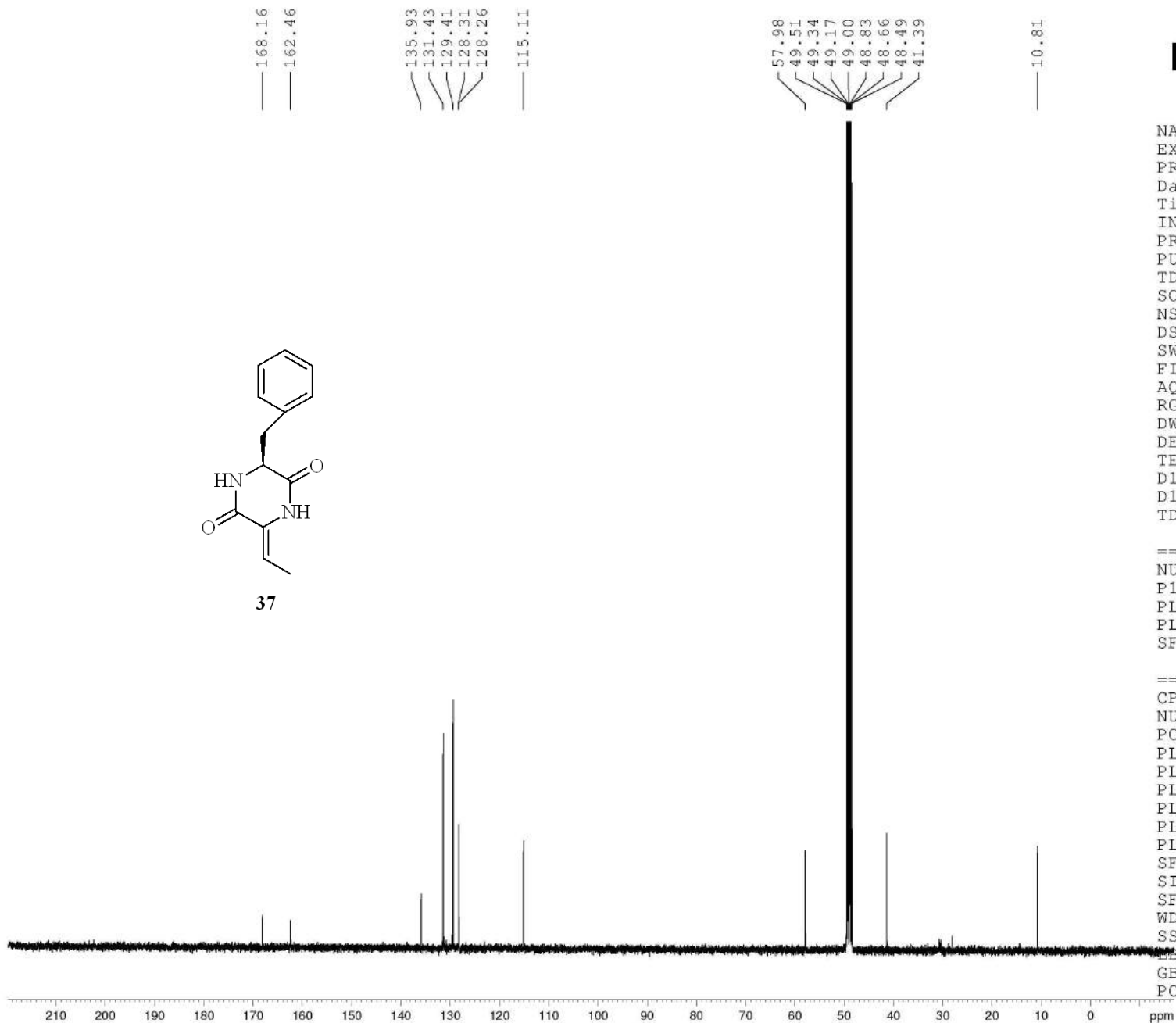
===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1330008
SI 32768
SF 500.1300156
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.00



liushouxin-ZY-2018-10-29



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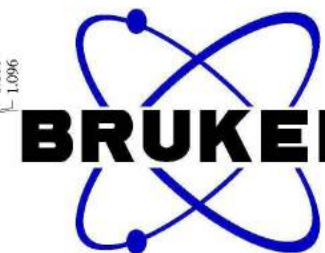


NAME 2018.11.21
EXPNO 15
PROCNO 1
Date_ 20181121
Time 22.04
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT MeOD
NS 8192
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1030
DW 16.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

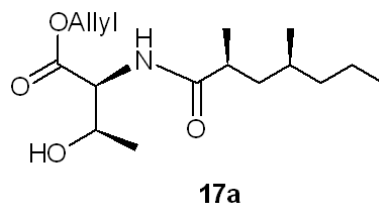
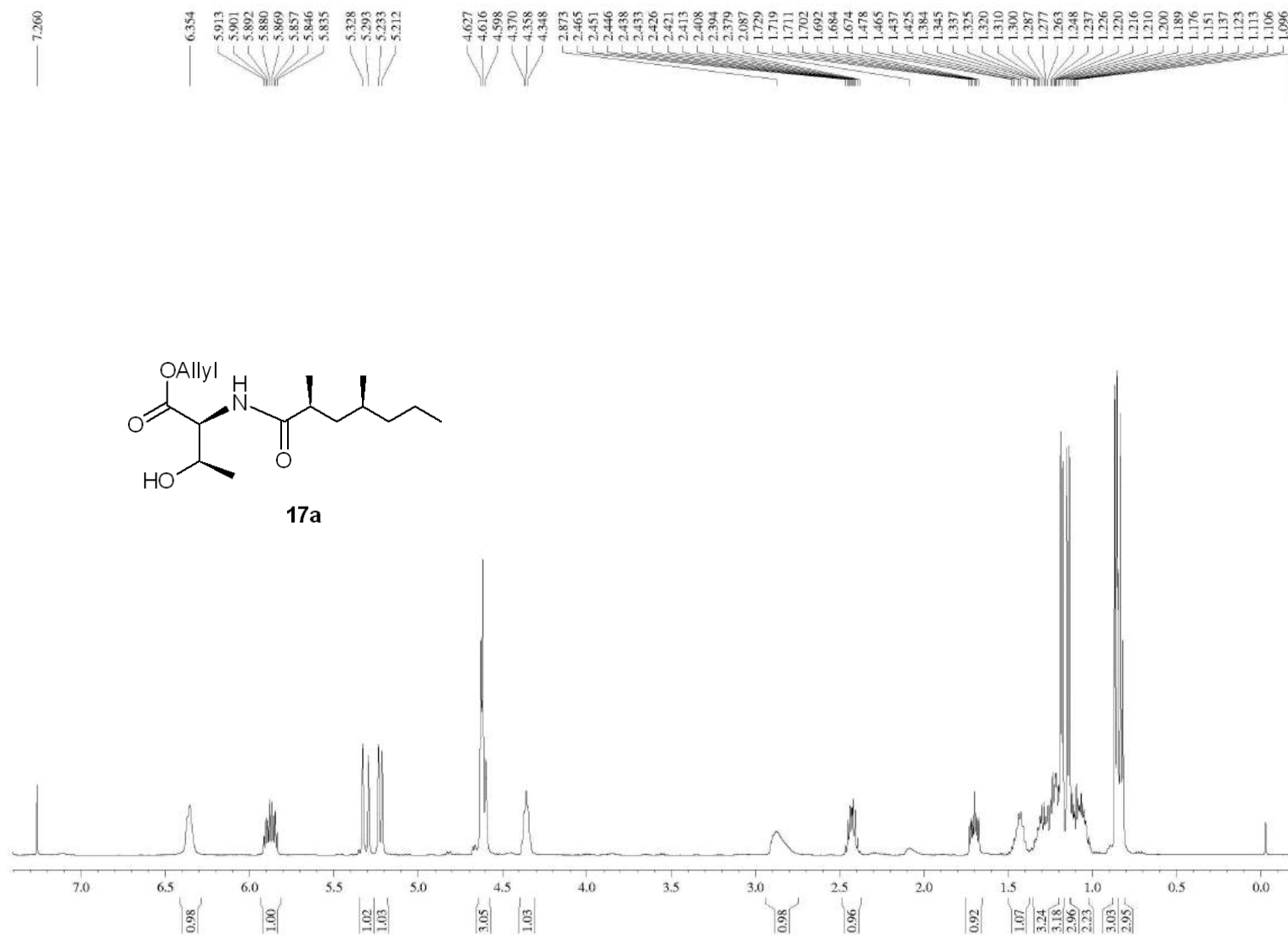
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7576132 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

liushouxin-xh-SR-T



NAME 2020.12.29
 EXPNO 9
 PROCNO 1
 Date_ 20201229
 Time 11.24
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 50.8
 DW 50.000
 DE 6.50
 TE 298.0
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300224
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



liushouxin-xh-SR-T

— 177.53
— 170.95

— 131.60
— 119.00

77.42
77.16
76.91
68.12
66.22
57.01

41.88
39.63
39.35
30.40

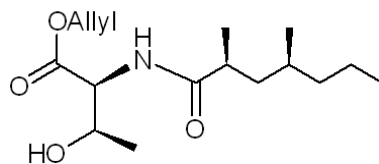
20.02
19.70
19.01
14.42



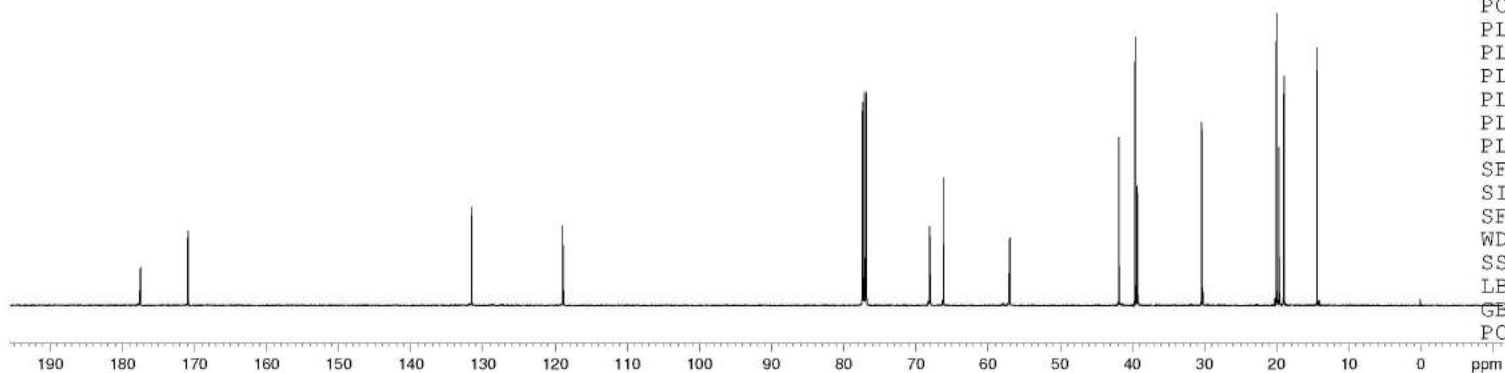
NAME 2021.03.11
EXPNO 11
PROCNO 1
Date_ 20210317
Time 17.18
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1150
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

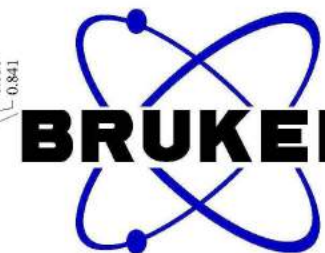
==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577768 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



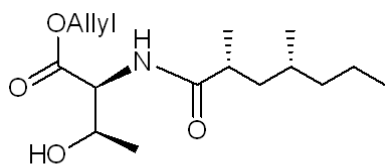
17a



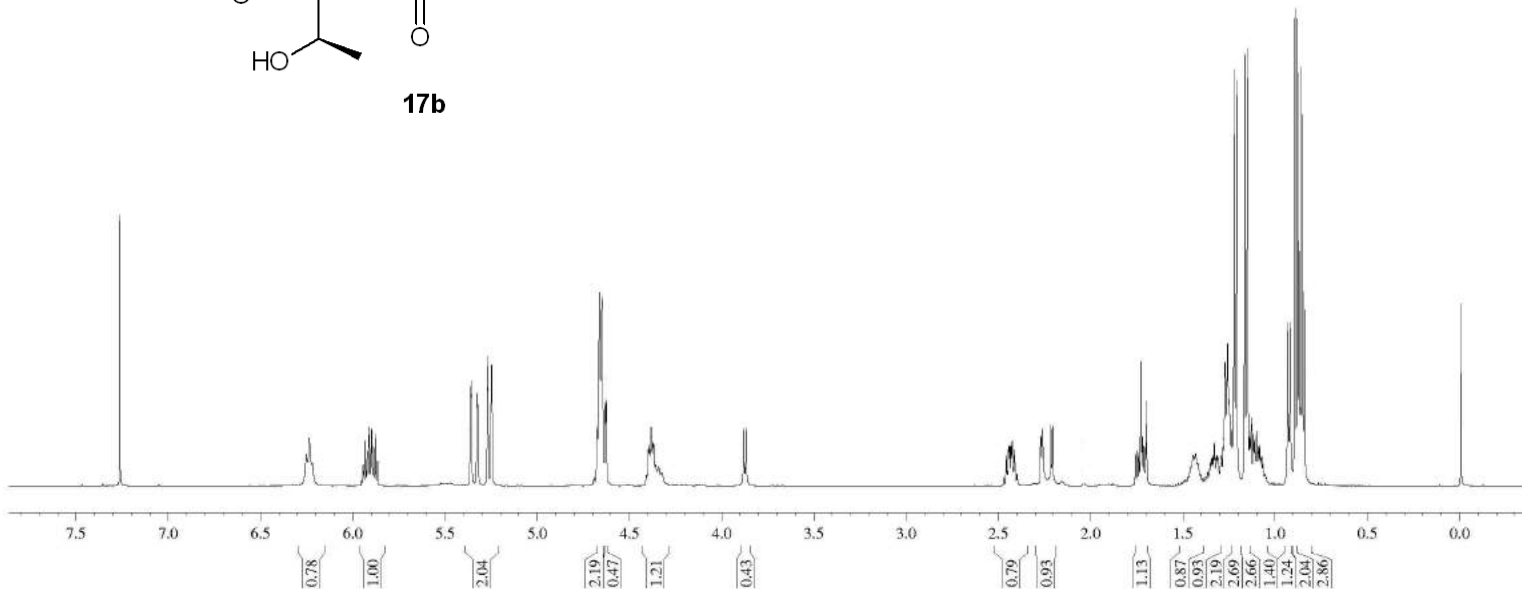
xh-rs-thr



7.260 6.250 6.233 5.930 5.920 5.918 5.909 5.895 5.885 5.884 5.874 5.356 5.354 5.322 5.319 5.268 5.265 5.263 5.247 5.245 5.242 4.671 4.660 4.646 4.641 4.628 4.623 4.393 4.388 4.380 4.371 4.366 4.366 3.877 3.864 2.456 2.445 2.442 2.437 2.431 2.426 2.424 2.413 2.413 2.270 2.261 2.215 2.205 1.757 1.747 1.738 1.738 1.728 1.720 1.711 1.701 1.695 1.444 1.431 1.431 1.338 1.329 1.291 1.282 1.278 1.270 1.257 1.250 1.235 1.220 1.207 1.207 1.164 1.151 1.138 1.134 1.128 1.117 1.111 1.099 1.092 1.088 1.082 0.931 0.918 0.892 0.879 0.870 0.856 0.841



17b



NAME 2020.12.18
EXPNO 13
PROCNO 1
Date_ 20201218
Time 19.36
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 22
DS 0
SWH 5000.000
FIDRES 0.076294
AQ 6.5536499
RG 161
DW 100.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1322506
SI 32768
SF 500.1300228
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

liushouxin-xh-RS-Thr

— 177.45
— 171.05

— 131.59
— 119.05

77.41
77.16
76.90
68.13
66.26
57.08

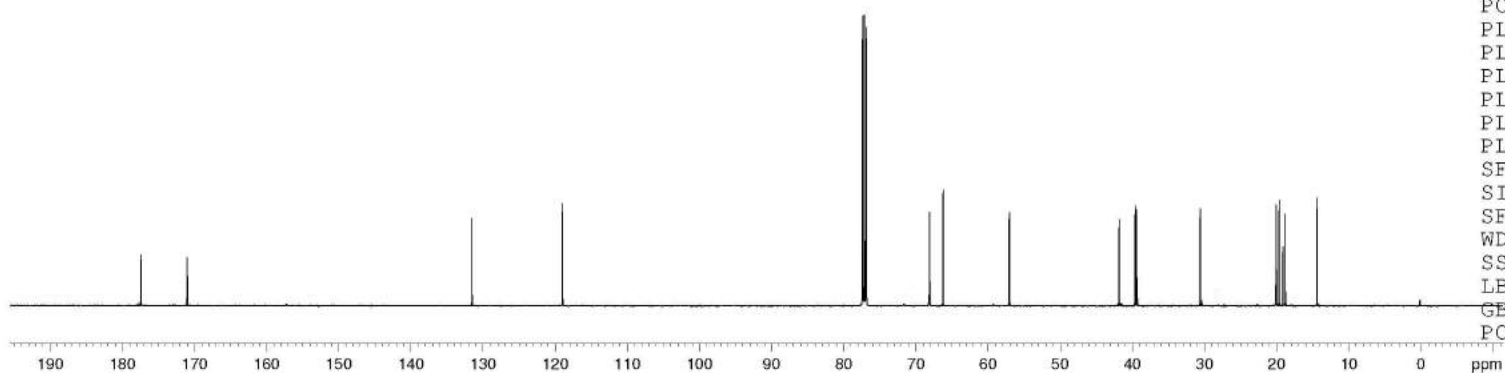
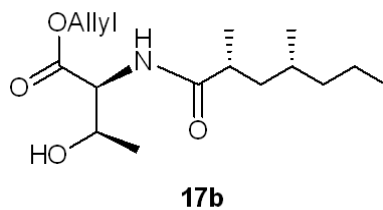
41.84
39.63
39.43
30.59
20.10
20.05
19.64
18.81
14.40



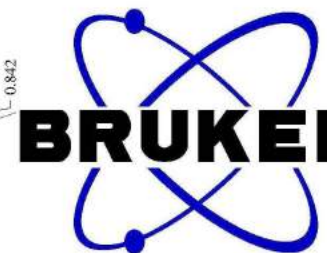
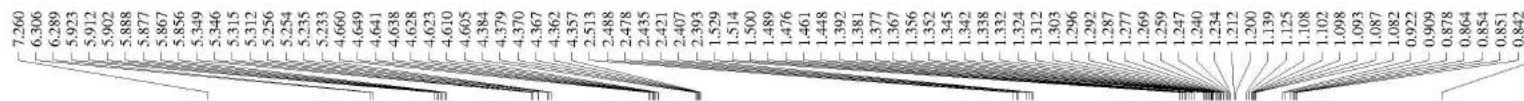
NAME 2021.03.11
EXPNO 18
PROCNO 1
Date_ 20210324
Time 17.18
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1820
DW 16.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

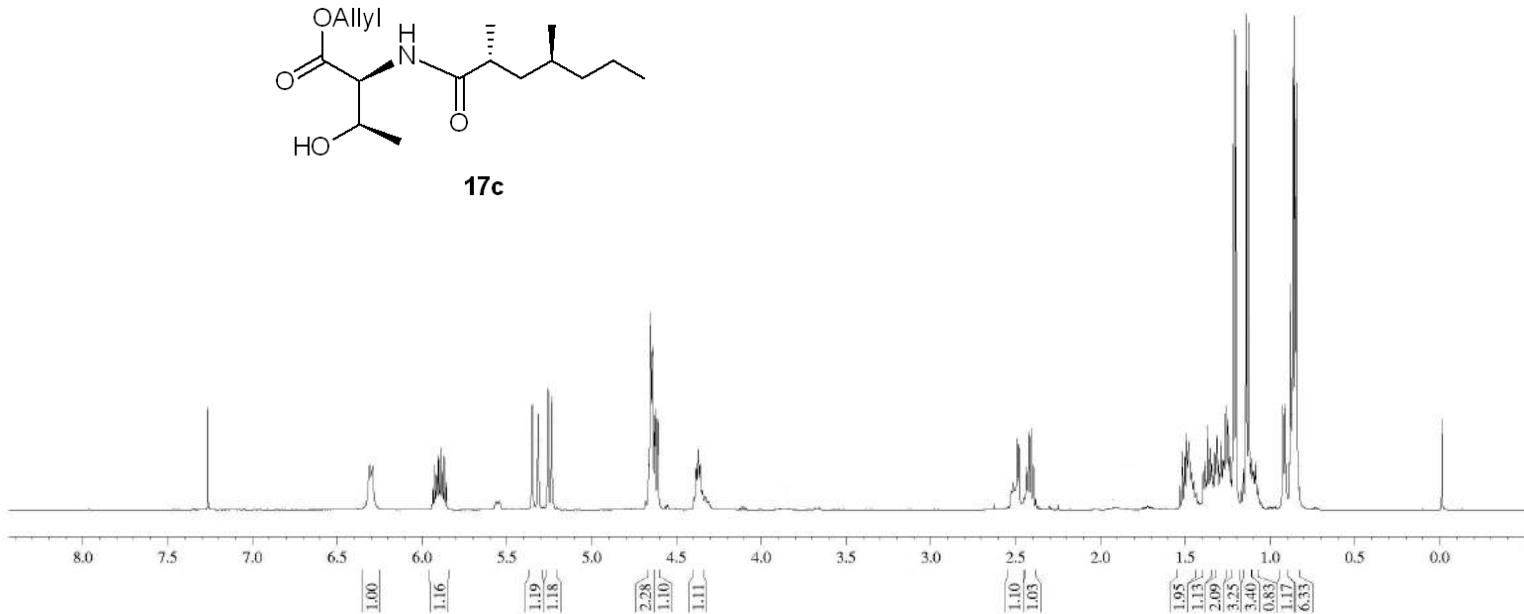
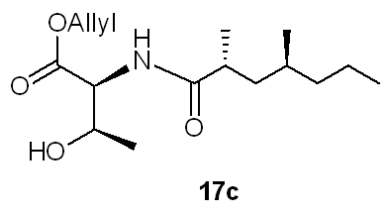
==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577748 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



xh-thr-ss



NAME 2020.12.03
EXPNO 13
PROCNO 1
Date_ 20201203
Time 18.12
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 20
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 114
DW 50.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1



===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1340010
SI 32768
SF 500.1300230
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

liushouxin-xh--Thr-SS

— 177.83
— 171.02

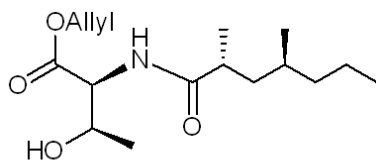
— 131.62
— 118.90

77.41
77.16
76.90
67.99
66.17
— 57.19

41.48
39.34
39.20
— 30.34
20.12
20.03
19.60
17.82
14.43



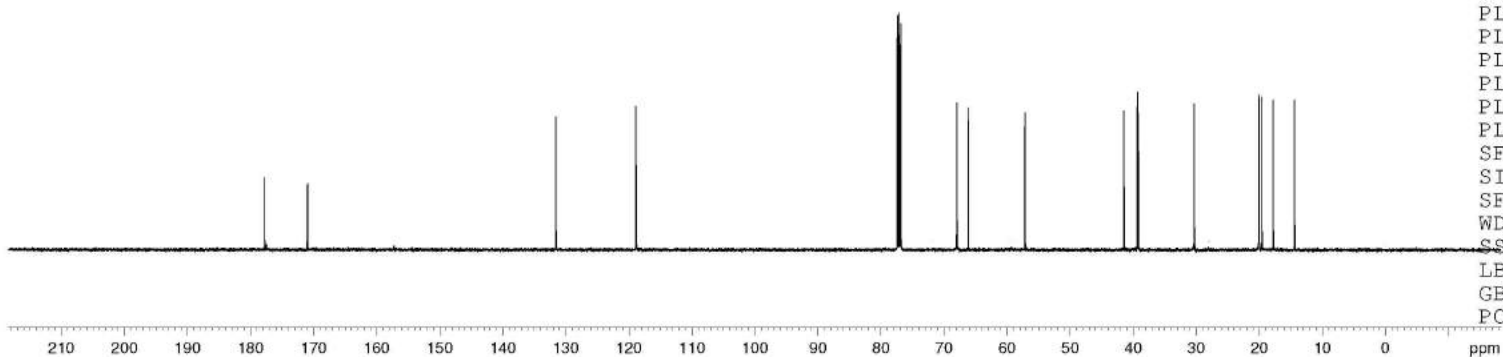
NAME 2021.03.11
EXPNO 19
PROCNO 1
Date_ 20210325
Time 16.45
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 401
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1820
DW 16.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



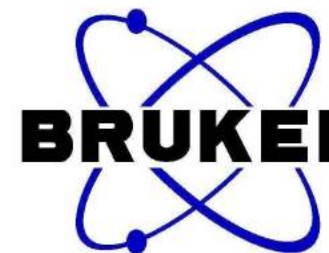
17c

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577786 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

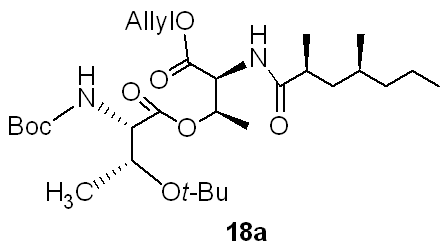
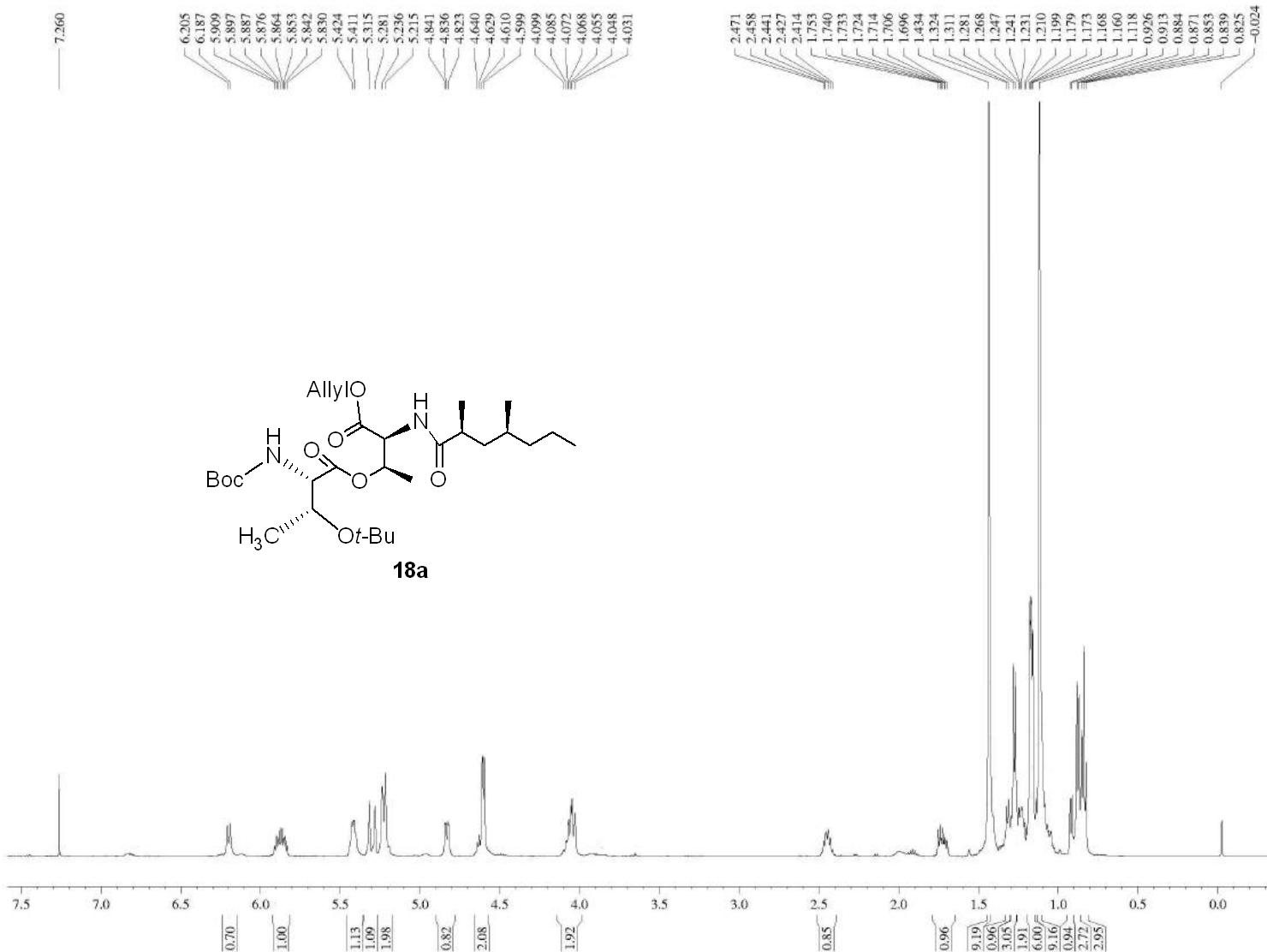


liushouxin-xh-SR-T-T



NAME 2020.12.29
 EXPNO 10
 PROCNO 1
 Date_ 20201229
 Time 11.31
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 50.8
 DW 50.000
 DE 6.50
 TE 298.0
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300224
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



liushouxin-xh-SR-T-T

177.23
170.64
169.40
156.04
131.61
119.25

79.85
77.42
77.16
76.91
74.07
71.96
67.08
66.59
59.41
55.28

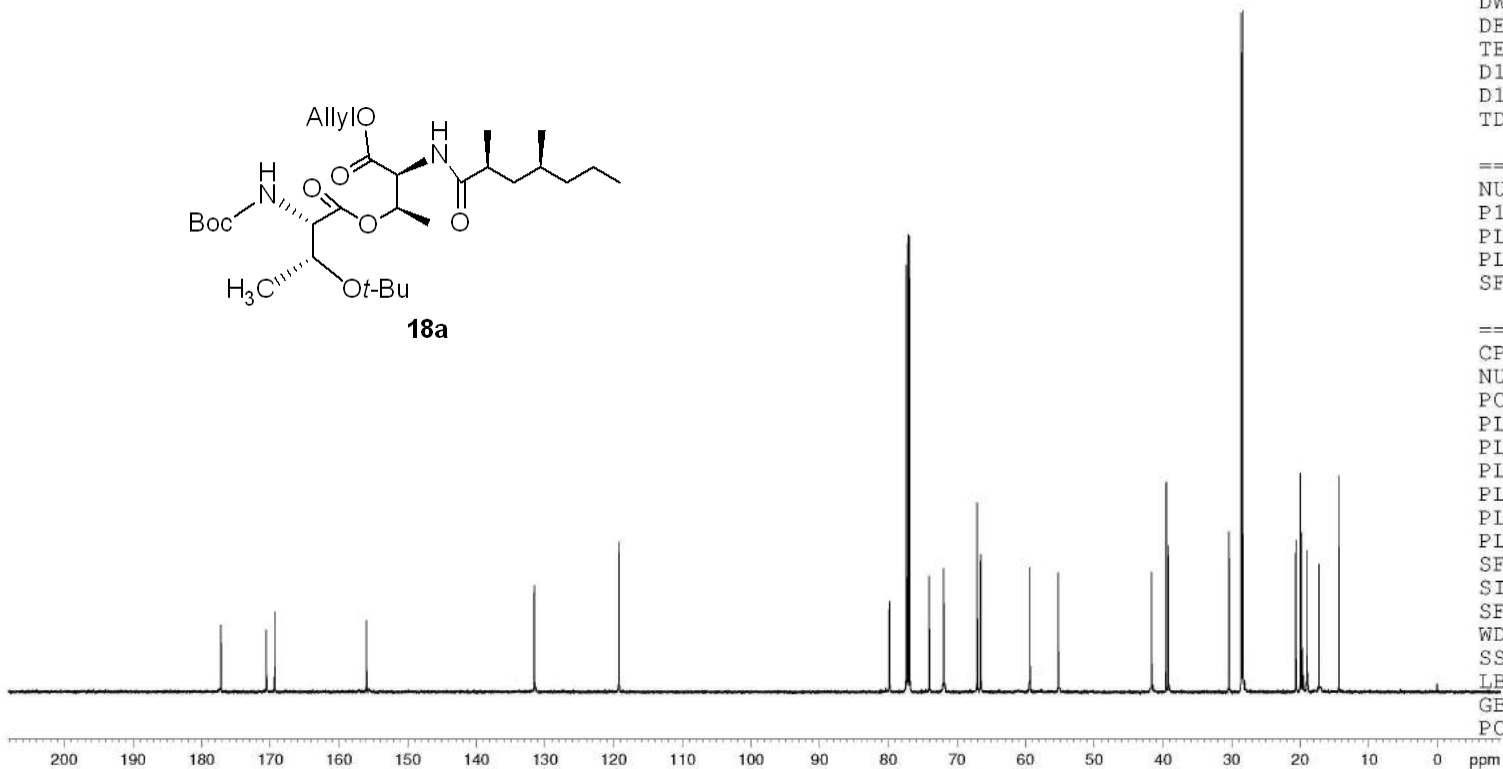
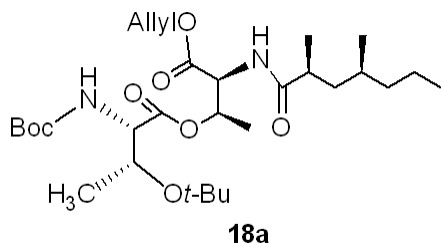
41.69
39.56
39.24
30.40
28.61
28.41
20.66
20.02
19.77
19.08
17.30
14.40



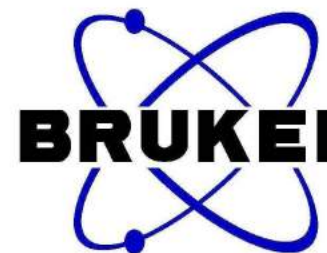
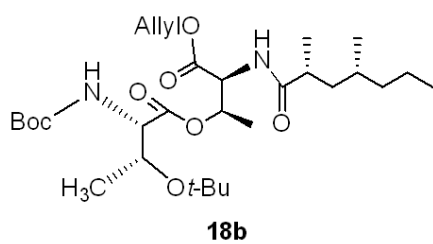
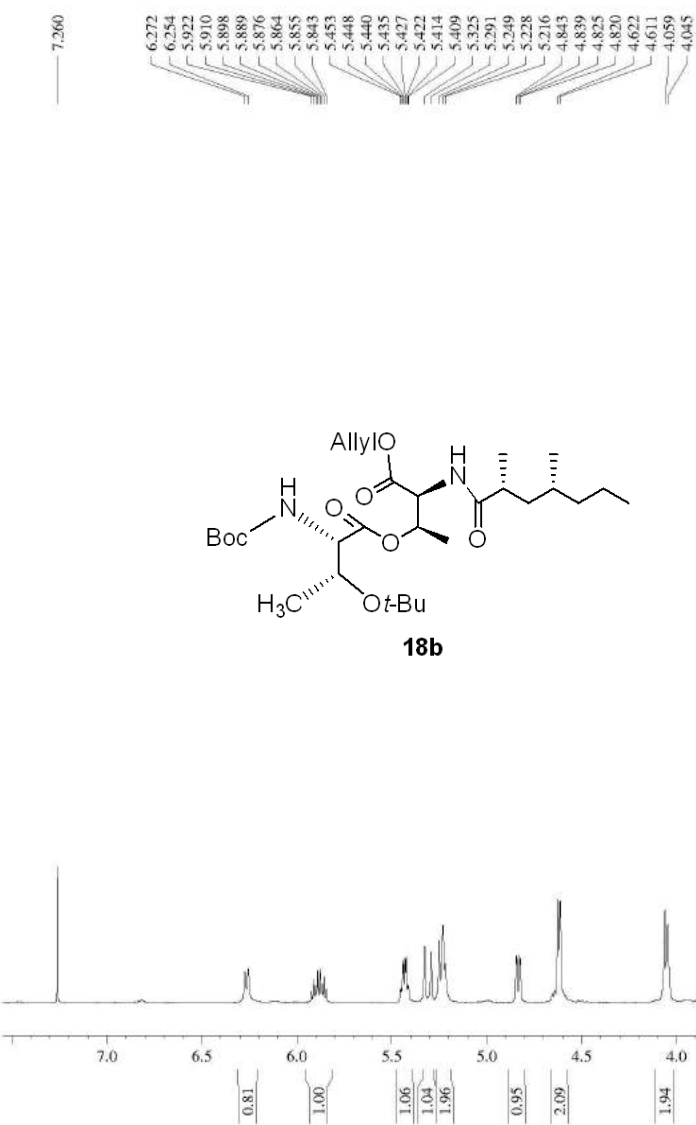
NAME 2021.03.11
EXPNO 12
PROCNO 1
Date_ 20210318
Time 9.01
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 2391
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1150
DW 16.800 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577776 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



xh-rs-t-t



NAME 2020.12.23
 EXPNO 24
 PROCNO 1
 Date_ 20201223
 Time 17.32
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 80.6
 DW 50.000
 DE 6.50
 TE 298.0
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300227
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

liushouxin-xh--RS-T-T

— 177.23
— 170.59
— 169.51
— 156.03
— 131.66
— 119.29

79.91
77.42
77.16
76.91
74.14
72.05
67.18
66.63
59.40
55.34

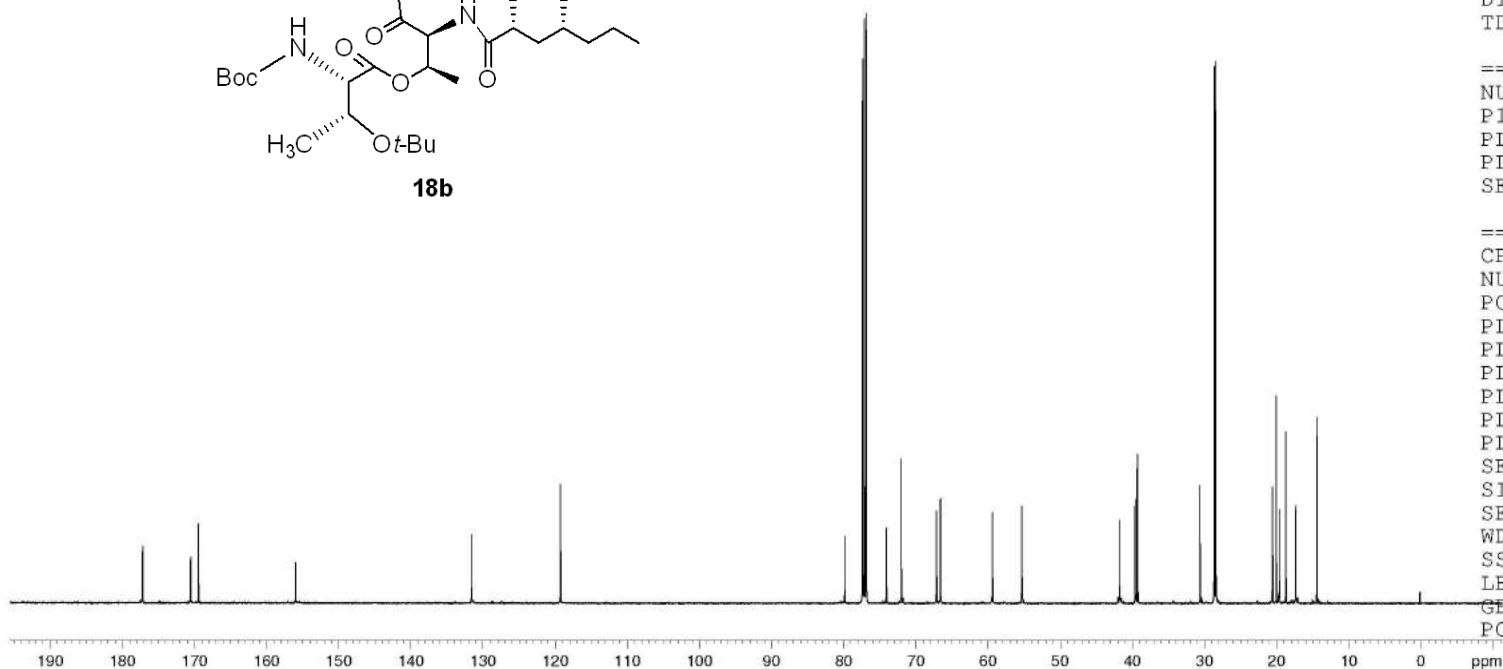
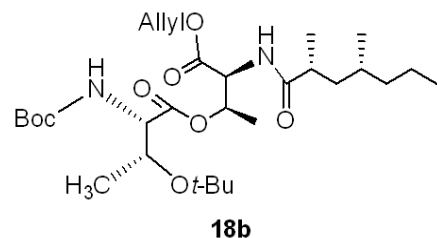
41.82
39.63
39.32
30.62
28.66
28.45
20.57
20.06
19.62
18.75
17.39
14.39



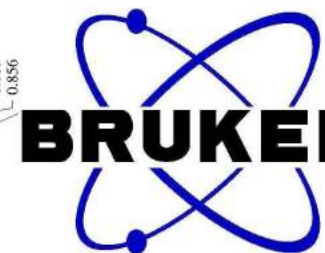
NAME 2021.03.11
EXPNO 20
PROCNO 1
Date_ 20210325
Time 17.08
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1820
DW 16.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577748 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

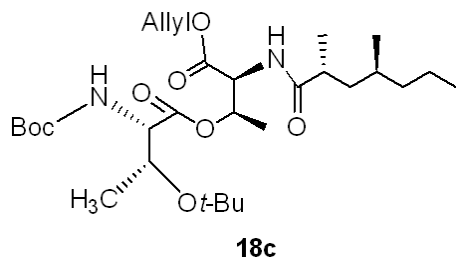
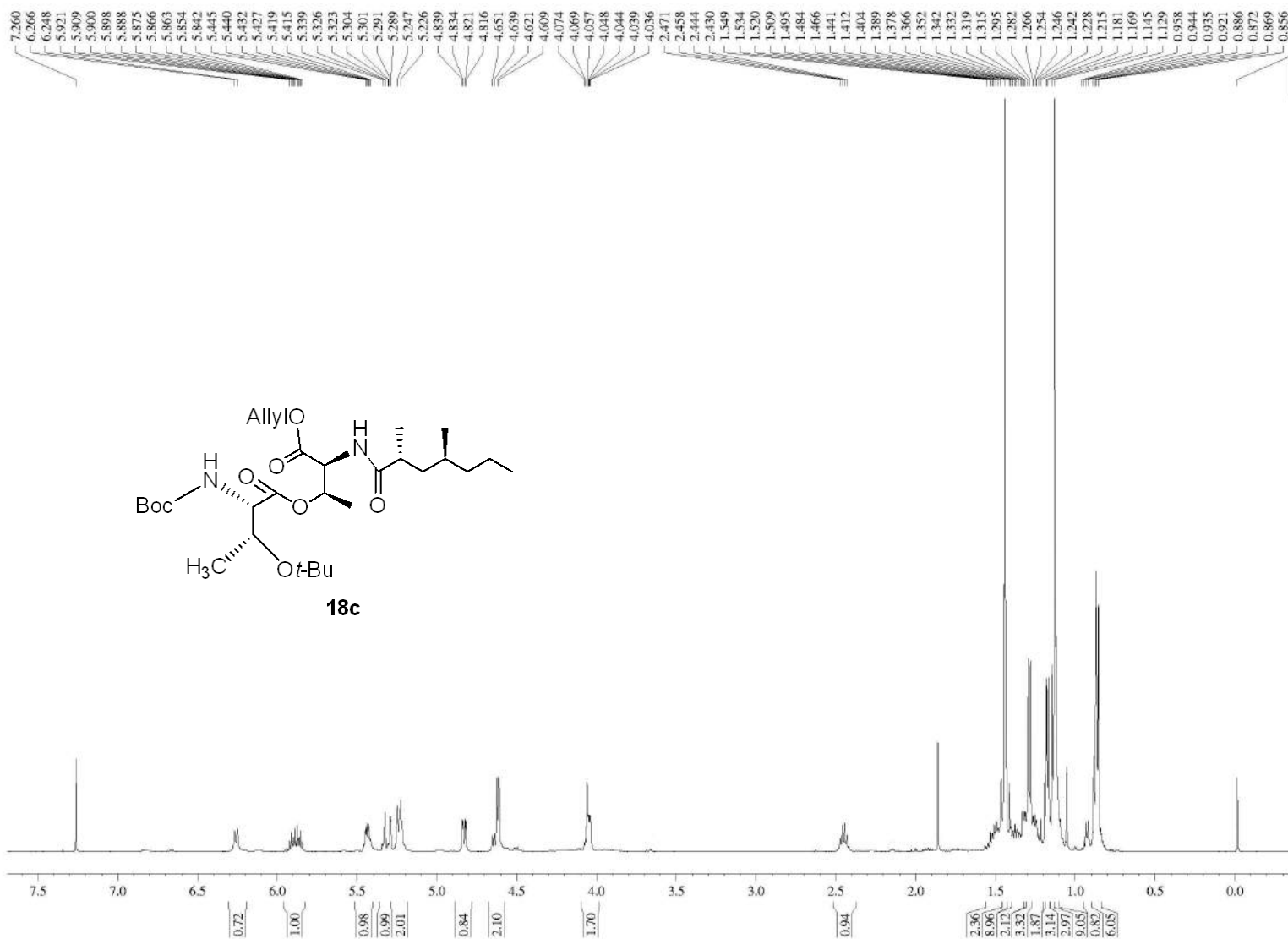


xh-ss-t-t



NAME 2020.12.09
EXPNO 30
PROCNO 1
Date_ 20201209
Time 18.09
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 80.6
DW 50.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1340010
SI 32768
SF 500.1300226
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



liushouxin-xh-SS-T-T

— 177.55
 — 170.60
 — 169.52
 — 156.04
 — 131.65
 — 119.27

79.89
 77.41
 77.16
 76.91
 74.11
 71.99
 67.11
 66.63
 59.38
 55.34

41.49
 39.31
 39.14
 30.39
 28.63
 28.42
 20.60
 20.04
 19.68
 17.83
 17.37
 14.47



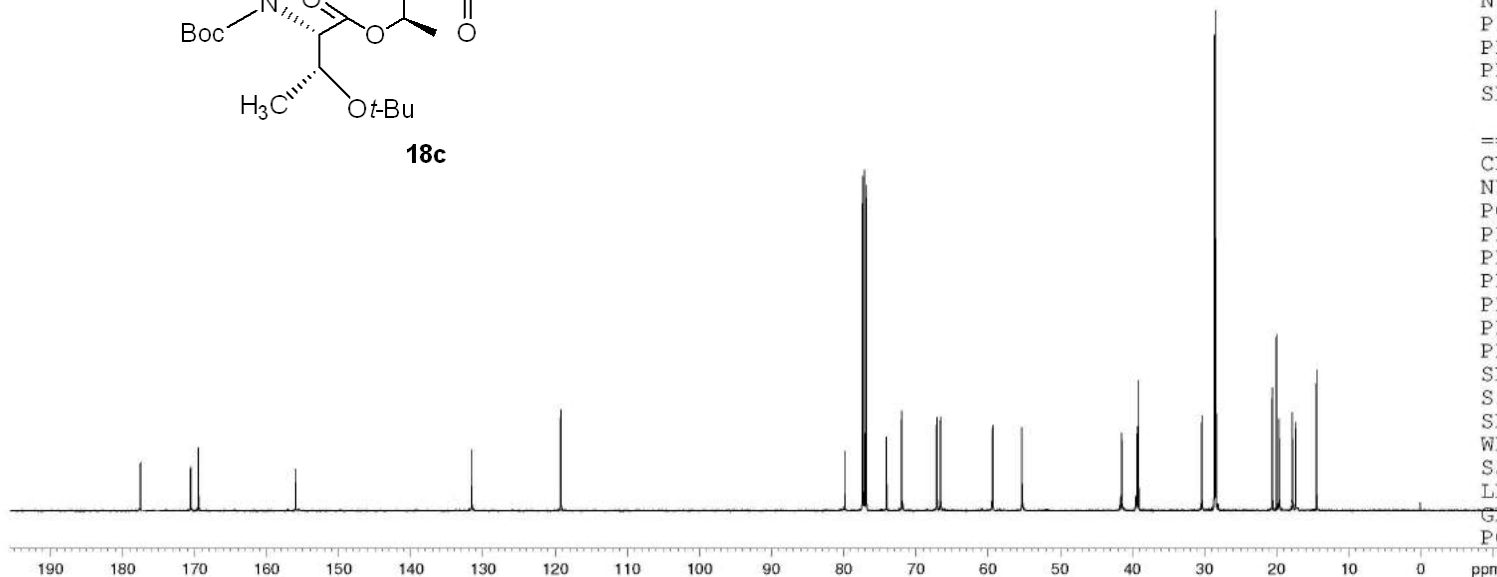
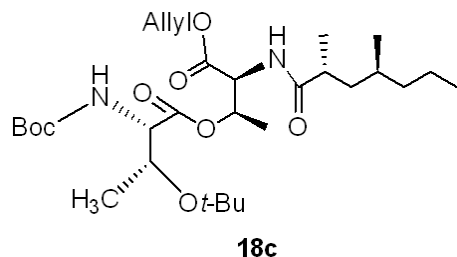
NAME 2021.03.11
 EXPNO 13
 PROCNO 1
 Date_ 20210318
 Time 17.34
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zgpg30
 TD 32768
 SOLVENT CDC13
 NS 10240
 DS 0
 SWH 29761.904 Hz
 FIDRES 0.908261 Hz
 AQ 0.5505524 sec
 RG 1150
 DW 16.800 usec
 DE 6.50 usec
 TE 296.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====

NUC1 13C
 P1 12.00 usec
 PL1 4.50 dB
 PL1W 33.60015869 W
 SFO1 125.7703643 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.00 dB
 PL12 17.46 dB
 PL13 17.46 dB
 PL2W 16.79986763 W
 PL12W 0.47786582 W
 PL13W 0.47786582 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7577767 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



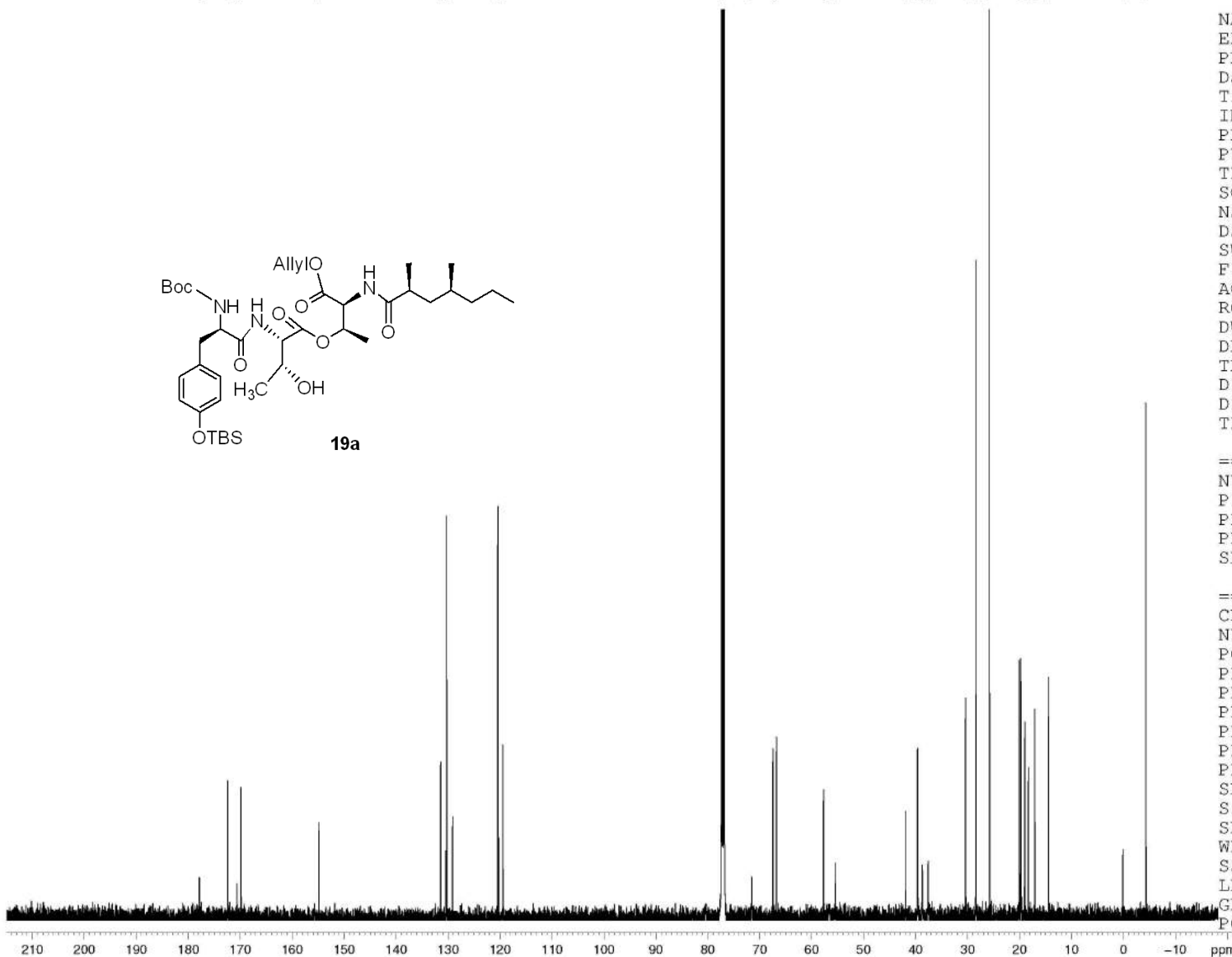
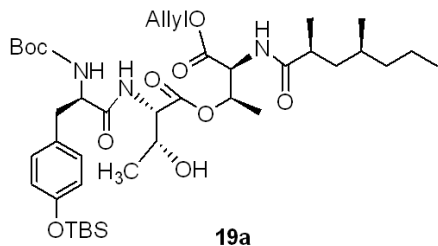
liushouxin XH-SR-3

177.94
172.46
170.69
169.92

154.90

131.48
130.49
130.30
129.17
120.45
120.28
119.47

77.41
77.16
76.91
71.57
67.49
66.81
57.75
55.51
41.97
39.67
38.74
37.60
30.42
28.43
25.80
20.08
19.80
19.78
19.01
18.32
17.08
14.45
0.14
-4.31

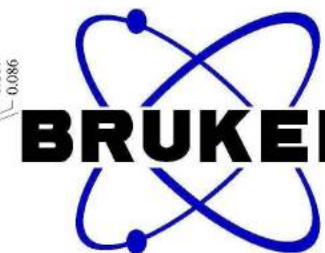


NAME 2021.05.07
EXPNO 6
PROCNO 1
Date_ 20210507
Time 13.00
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 2048
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 912
DW 16.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

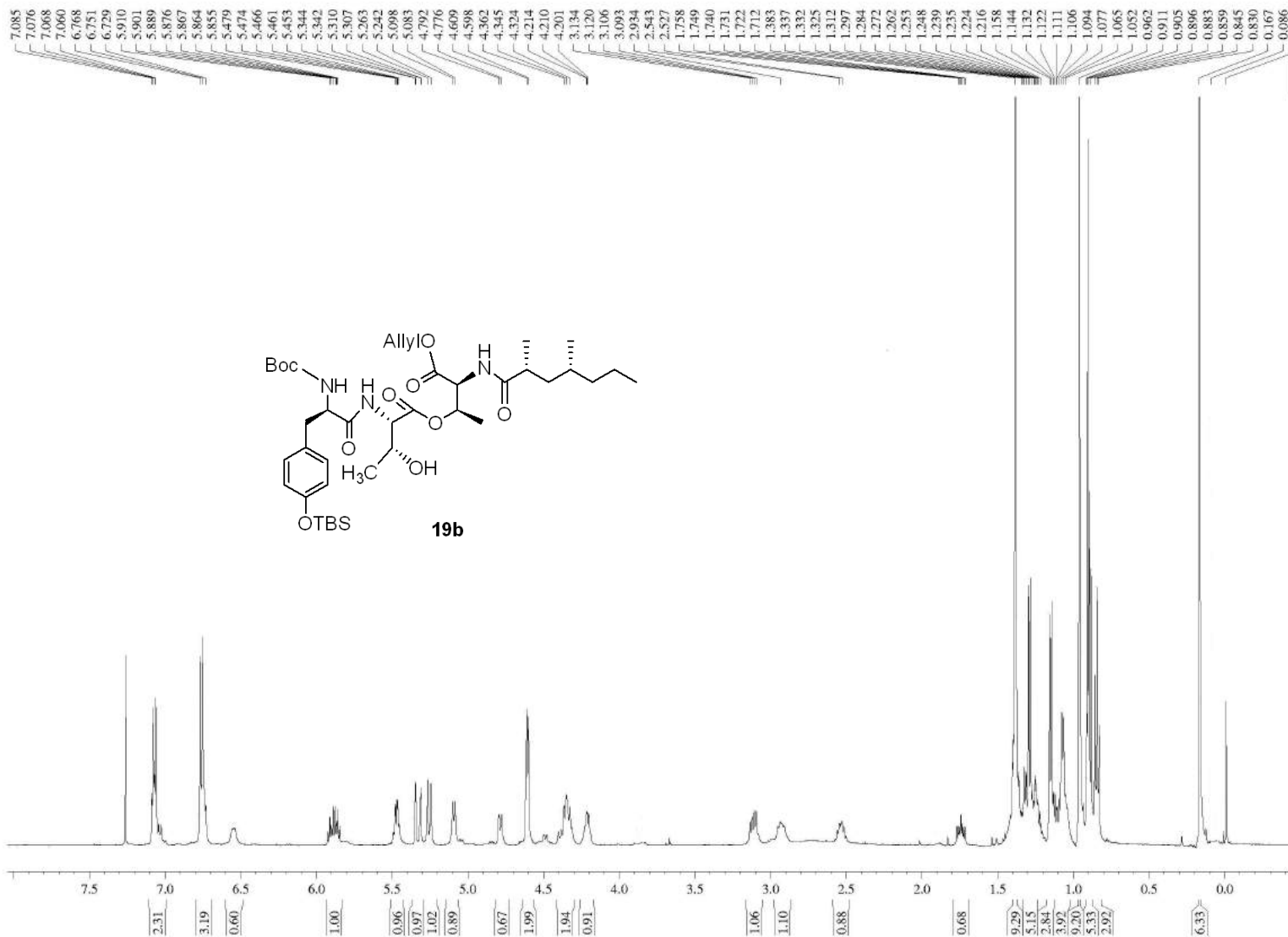
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

liushouxin-xh-RS-3



NAME 2020.12.29
EXPNO 11
PROCNO 1
Date_ 20201229
Time 11.39
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 80.6
DW 50.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1340010
SI 32768
SF 500.1300227
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



liushouxin-xh-RS-3

177.67
172.45
172.39
170.61
169.97
169.85

154.81
154.79

131.45
130.33
130.30
129.31
120.38
119.50

77.41
77.16
76.91

67.49
66.85

57.67
55.32

41.68
39.66
39.04

30.64
28.38
25.78

20.07
19.72
19.61
19.14

18.89
18.29
17.14
14.41

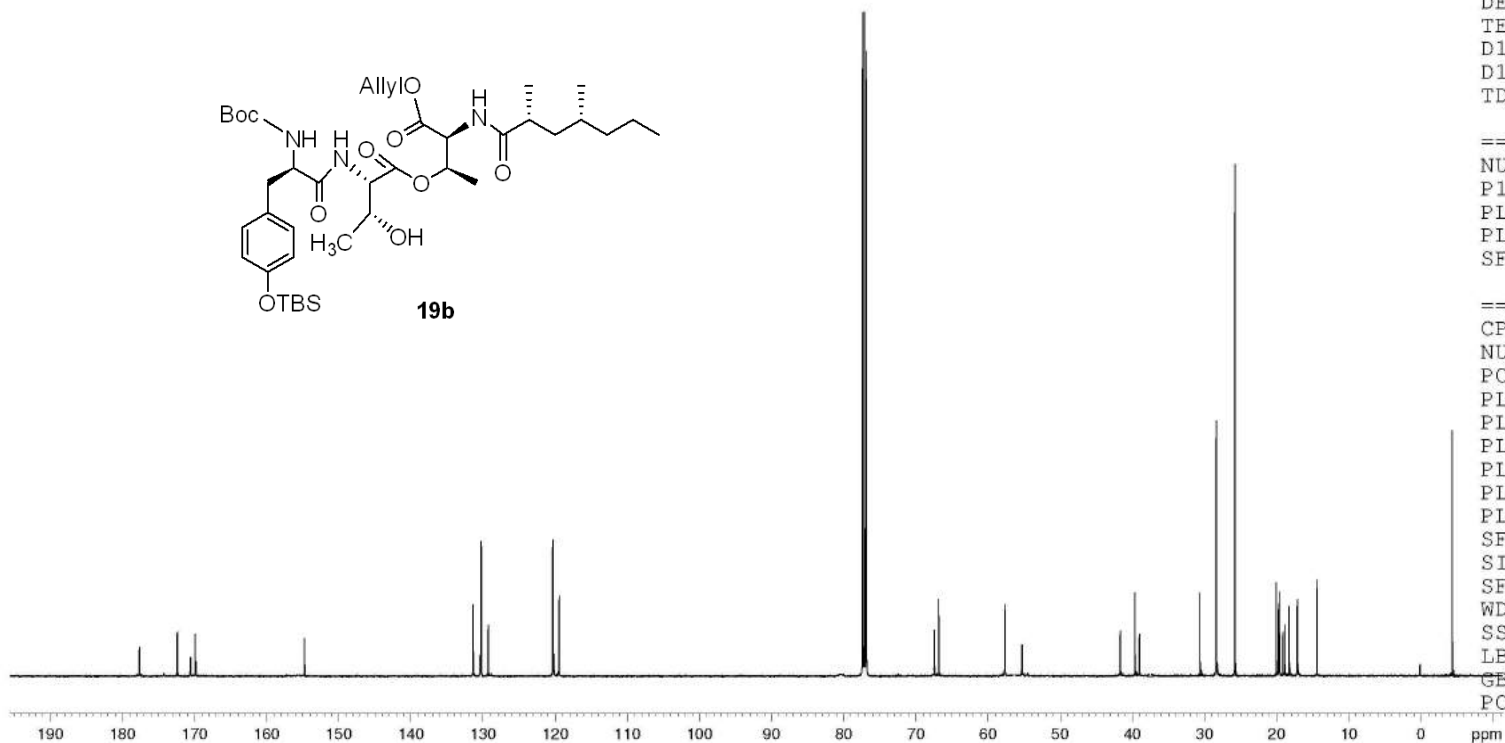
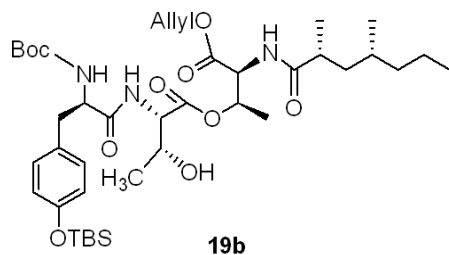
0.12
-4.33



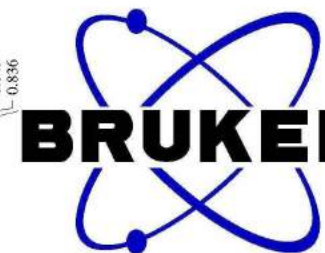
NAME 2021.03.11
EXPNO 10
PROCNO 1
Date_ 20210317
Time 9.24
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1150
DW 16.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577756 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

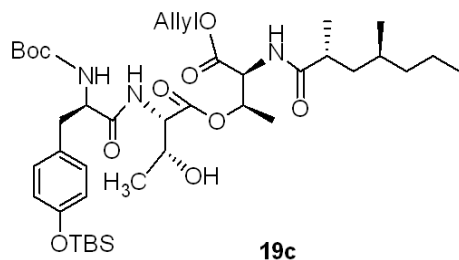
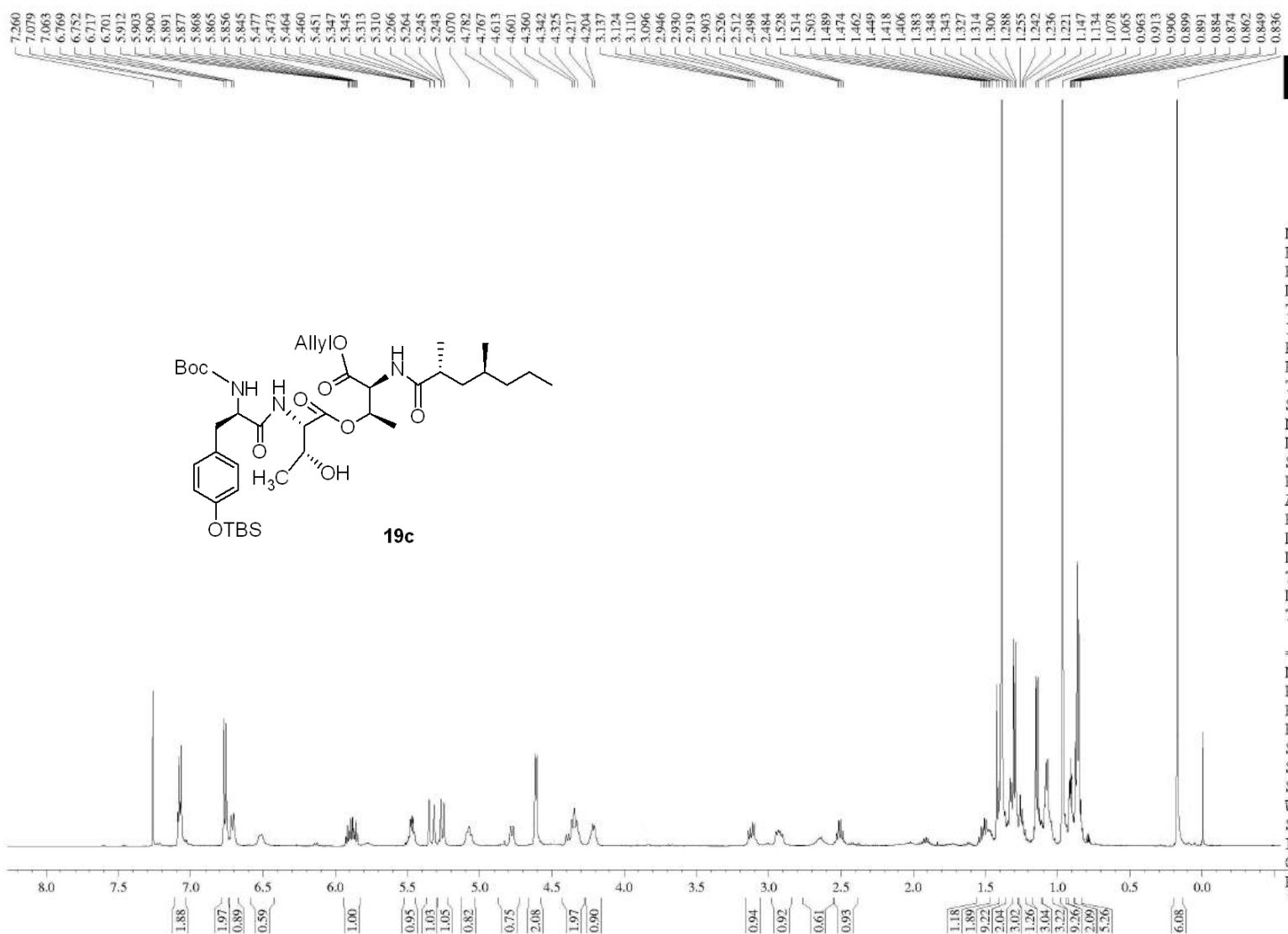


xh-ss-3



NAME 2020.12.18
EXPNO 12
PROCNO 1
Date_ 20201218
Time 19.29
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 5000.000
FIDRES 0.076294
AQ 6.5536499
RG 90.5
DW 100.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1322506
SI 32768
SF 500.1300228
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



liushouxin-xh-SS-3

178.01
172.44
172.37
170.55
169.99
169.85

154.80

131.45
130.30
129.30
120.37
119.50

80.40
77.42
77.16
76.91
71.67
67.42
66.83

57.64
55.38

41.30
39.44
38.95
37.53
30.43
28.39
25.77
20.10
19.73
19.61
19.13
18.29
17.95
17.15
14.46

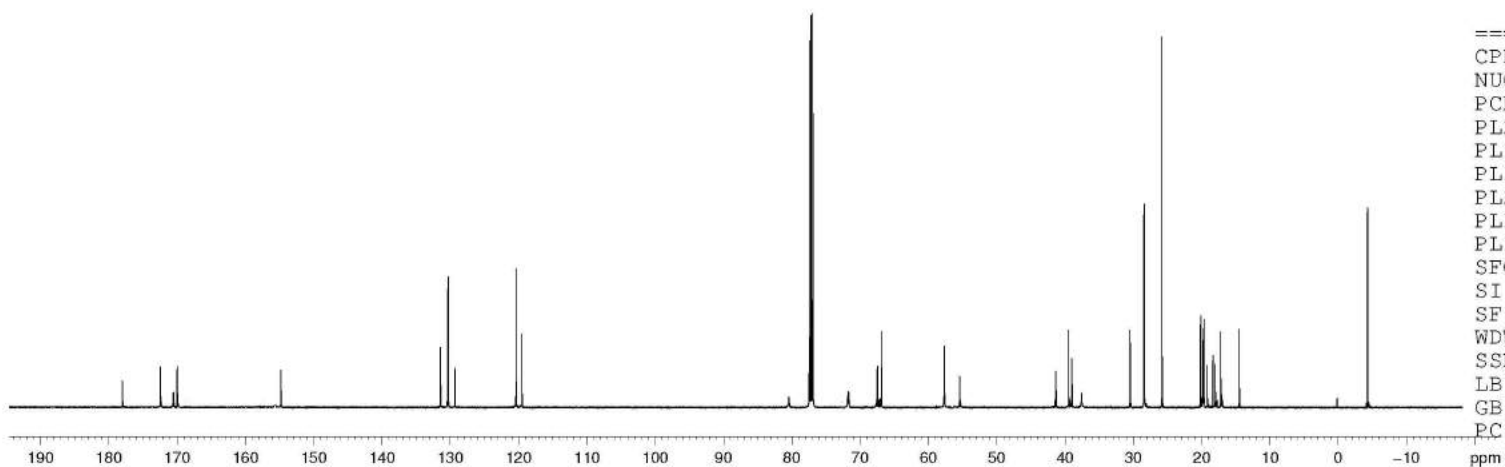
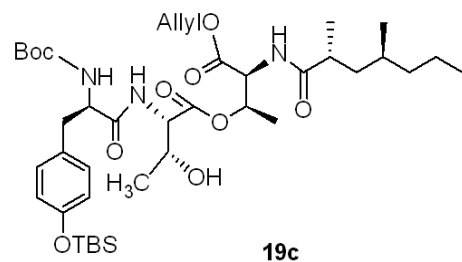
-4.34



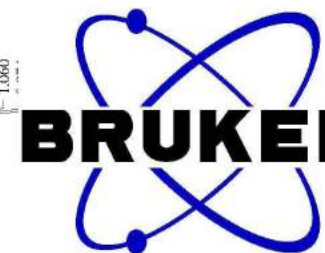
NAME 2021.03.11
EXPNO 16
PROCNO 1
Date_ 20210322
Time 13.01
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1820
DW 16.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577758 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



liushouxin-xh-SR-3-H2O

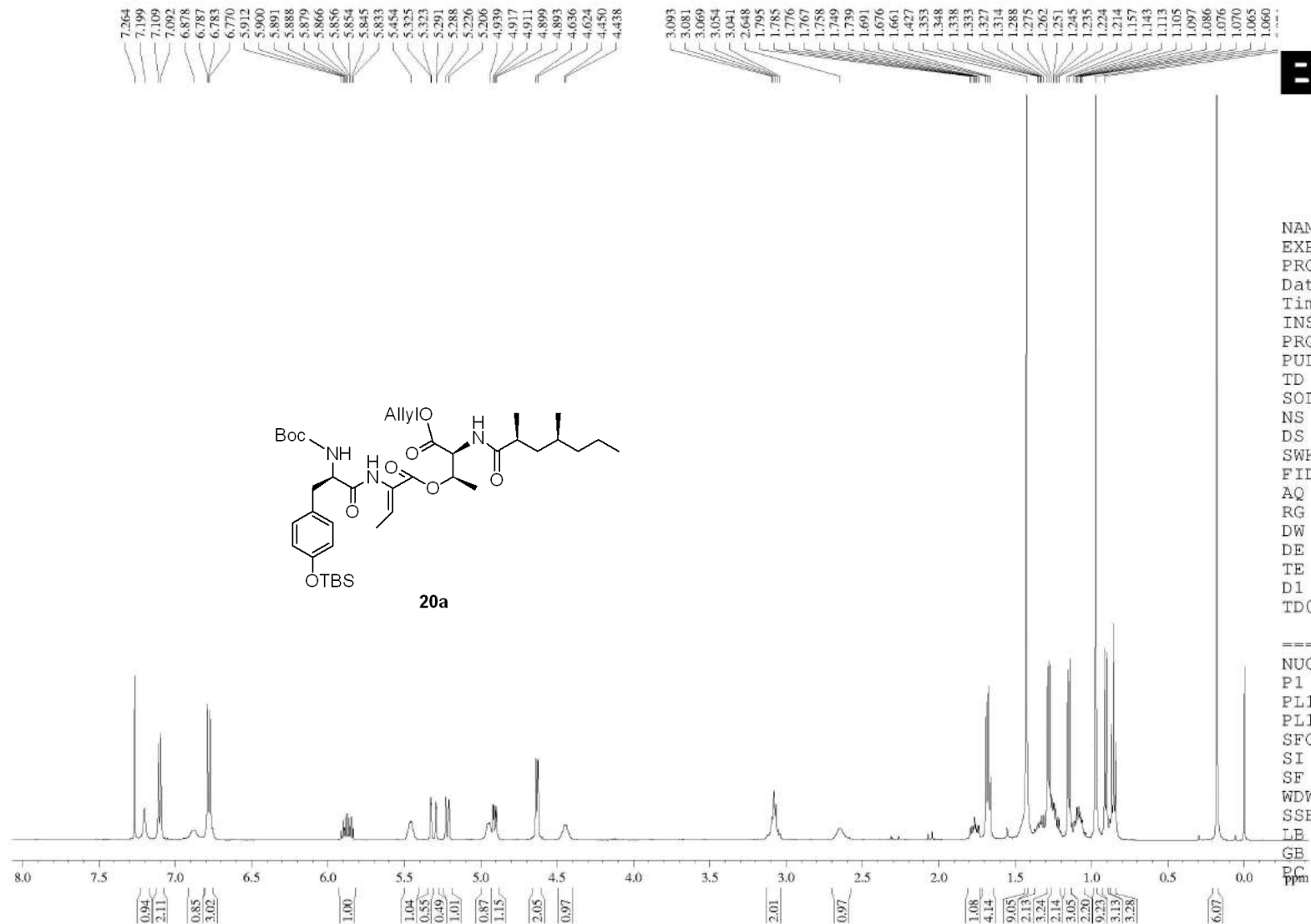
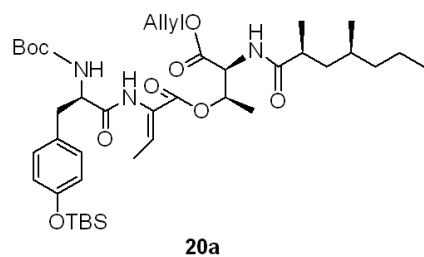


NAME 2021.05.06
 EXPNO 15
 PROCNO 1
 Date_ 20210506
 Time 16.25
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 181
 DW 50.000
 DE 6.50
 TE 296.9
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1345012
 SI 32768
 SF 500.1300207
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

7.264
7.199
7.109
7.092
6.878
6.787
6.783
6.770
5.912
5.900
5.891
5.888
5.879
5.866
5.856
5.854
5.845
5.833
5.454
5.325
5.323
5.291
5.288
5.226
5.206
4.939
4.917
4.911
4.899
4.893
4.636
4.624
4.450
4.438

3.093
3.081
3.069
3.054
3.041
2.648
1.795
1.785
1.776
1.767
1.758
1.749
1.739
1.691
1.676
1.661
1.427
1.353
1.348
1.338
1.333
1.327
1.314
1.288
1.275
1.262
1.251
1.245
1.235
1.224
1.214
1.157
1.143
1.113
1.105
1.097
1.086
1.076
1.065
1.060

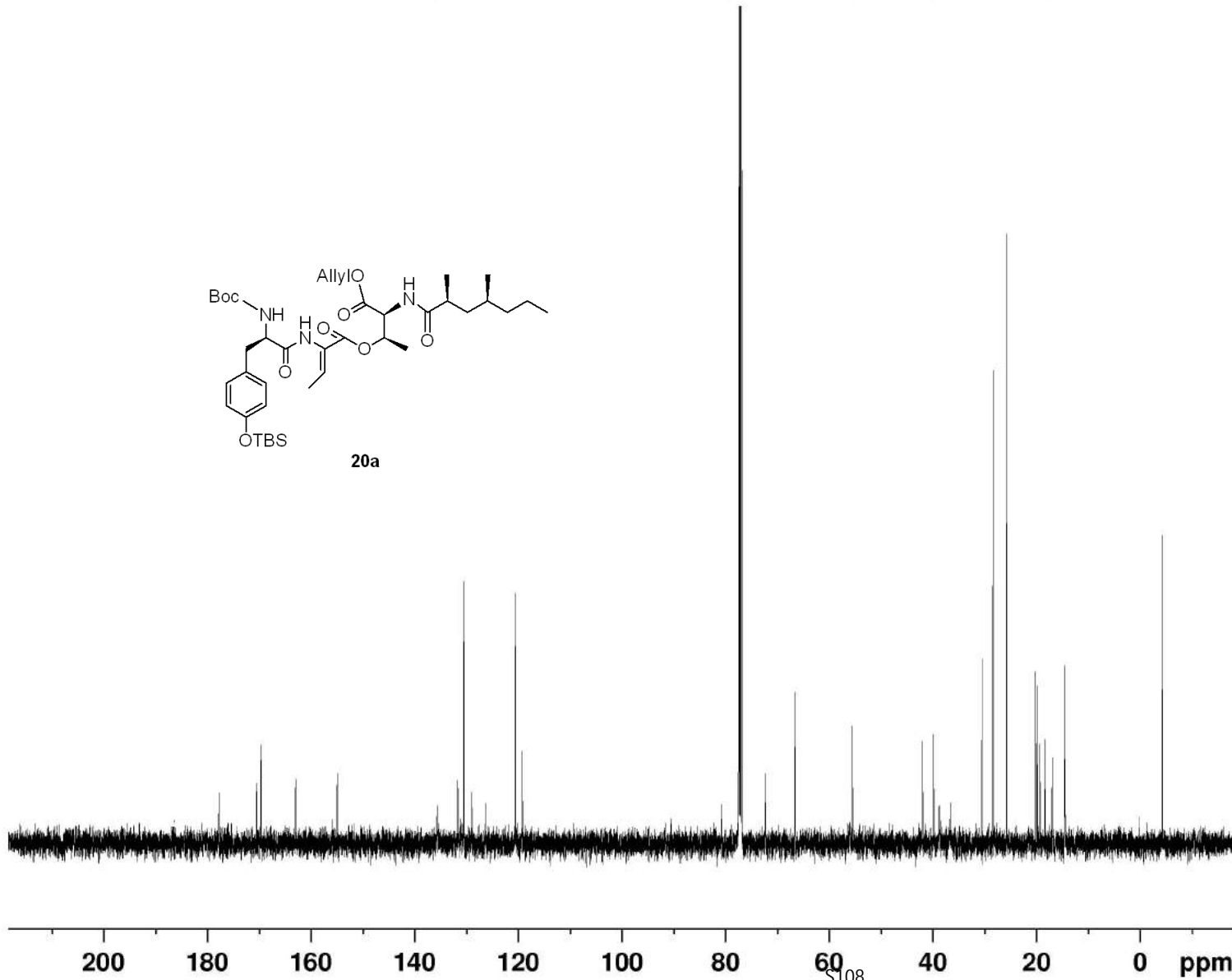
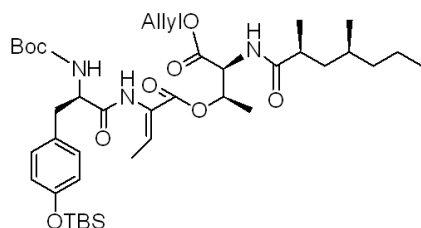


liushouxin XH-SR-3-h20



177.82
170.48
169.68
162.96
154.95
135.73
131.72
130.49
130.44
128.98
126.33
120.52
119.33
119.24

80.77
77.42
77.16
76.91
72.23
66.58
55.47
41.96
39.83
38.77
36.59
30.51
28.38
25.80
20.10
19.73
19.30
18.33
16.92
14.47
14.41
-4.30

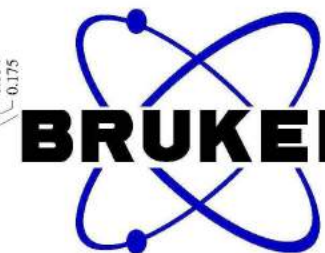


```
NAME 2021.05.07
EXPNO 7
PROCNO 1
Date_ 20210507
Time 17.37
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 262
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 912
DW 16.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

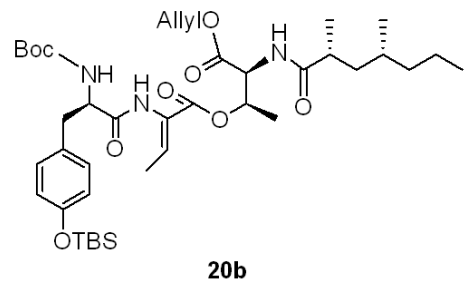
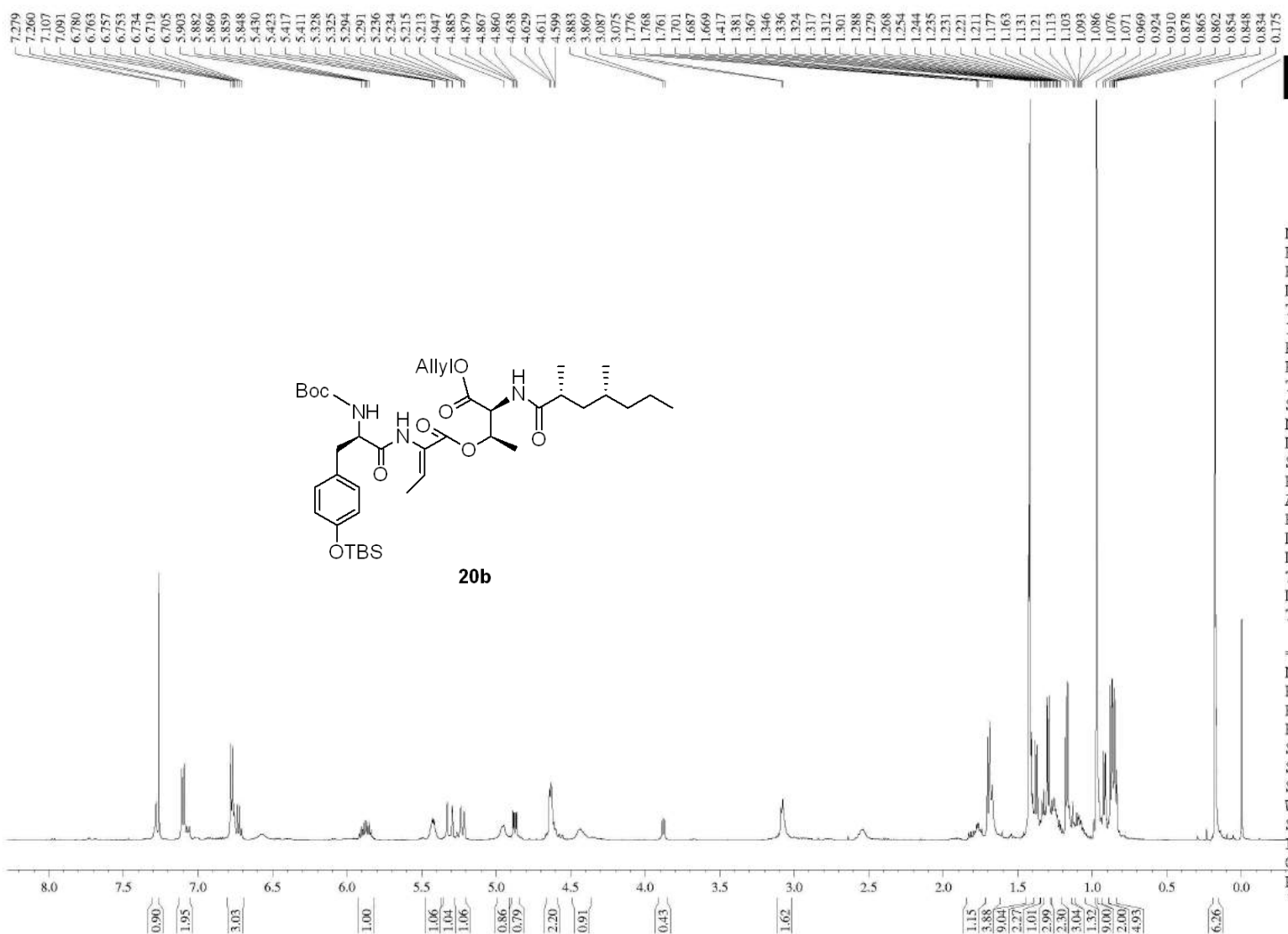
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```


liushouxin-xh-RS-3-H2O



NAME 2020.12.29
 EXPNO 12
 PROCNO 1
 Date_ 20201229
 Time 11.51
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 203
 DW 50.000
 DE 6.50
 TE 298.0
 D1 1.00000000
 TD0 1

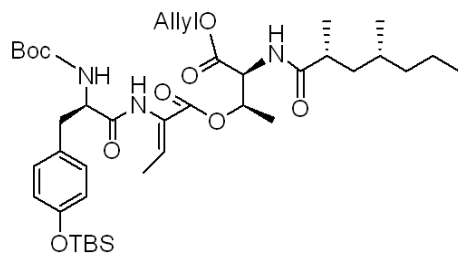
===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300226
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



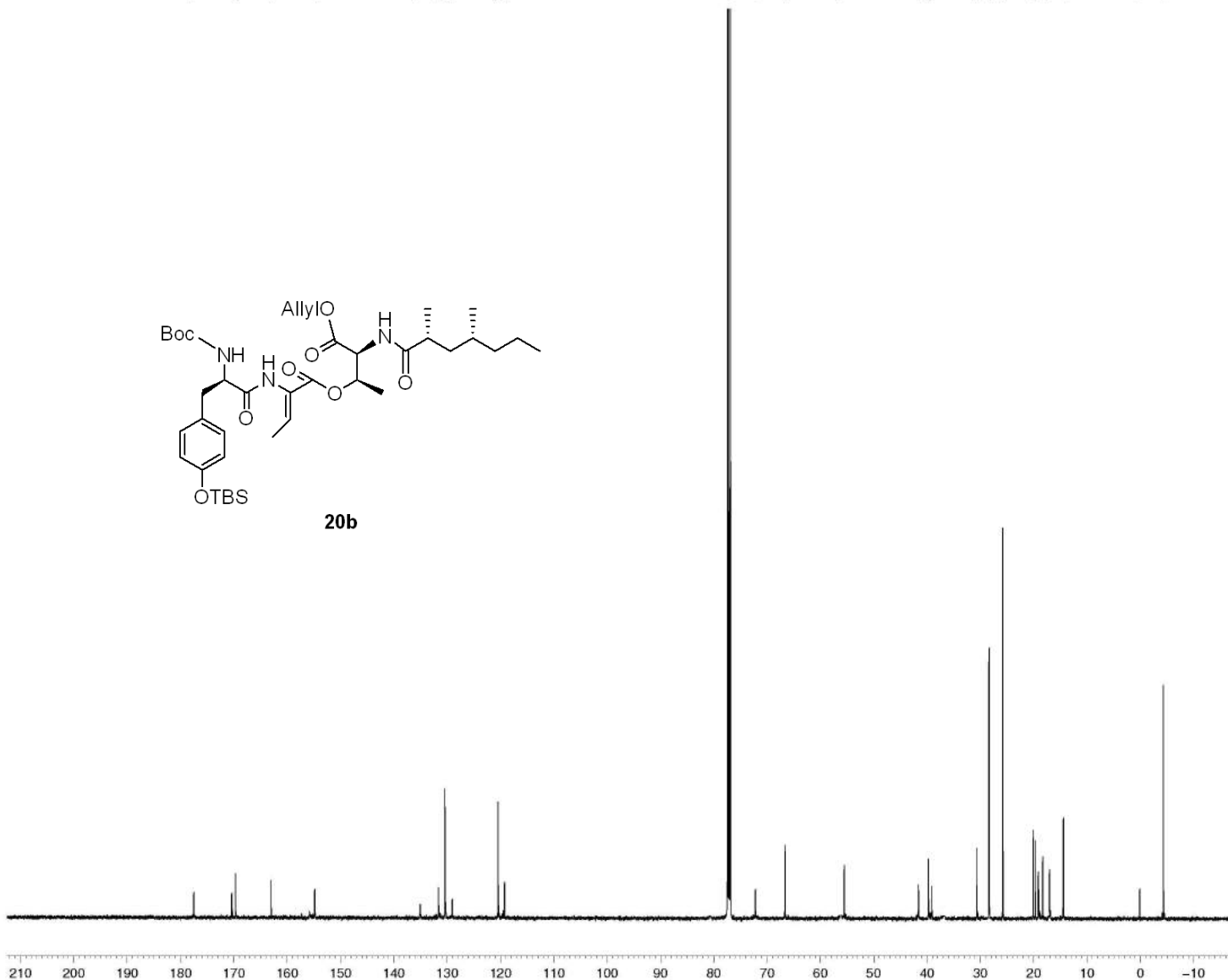
liushouxin-xh-RS--3-h2O

177.57
170.47
169.74
163.06
154.90
135.10
131.65
130.45
130.36
130.30
129.08
120.49
120.43
120.31
119.27

77.41
77.16
76.90
72.23
66.62
55.56
41.60
39.76
39.10
30.69
28.40
25.80
20.09
19.70
19.17
19.09
18.32
17.05
16.88
14.44
0.13
-4.31



20b

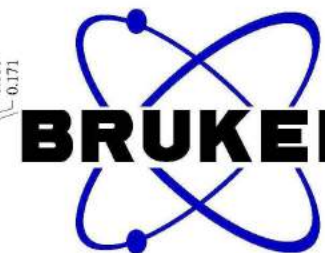


NAME 2021.03.11
EXPNO 15
PROCNO 1
Date_ 20210319
Time 17.05
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 912
DW 16.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

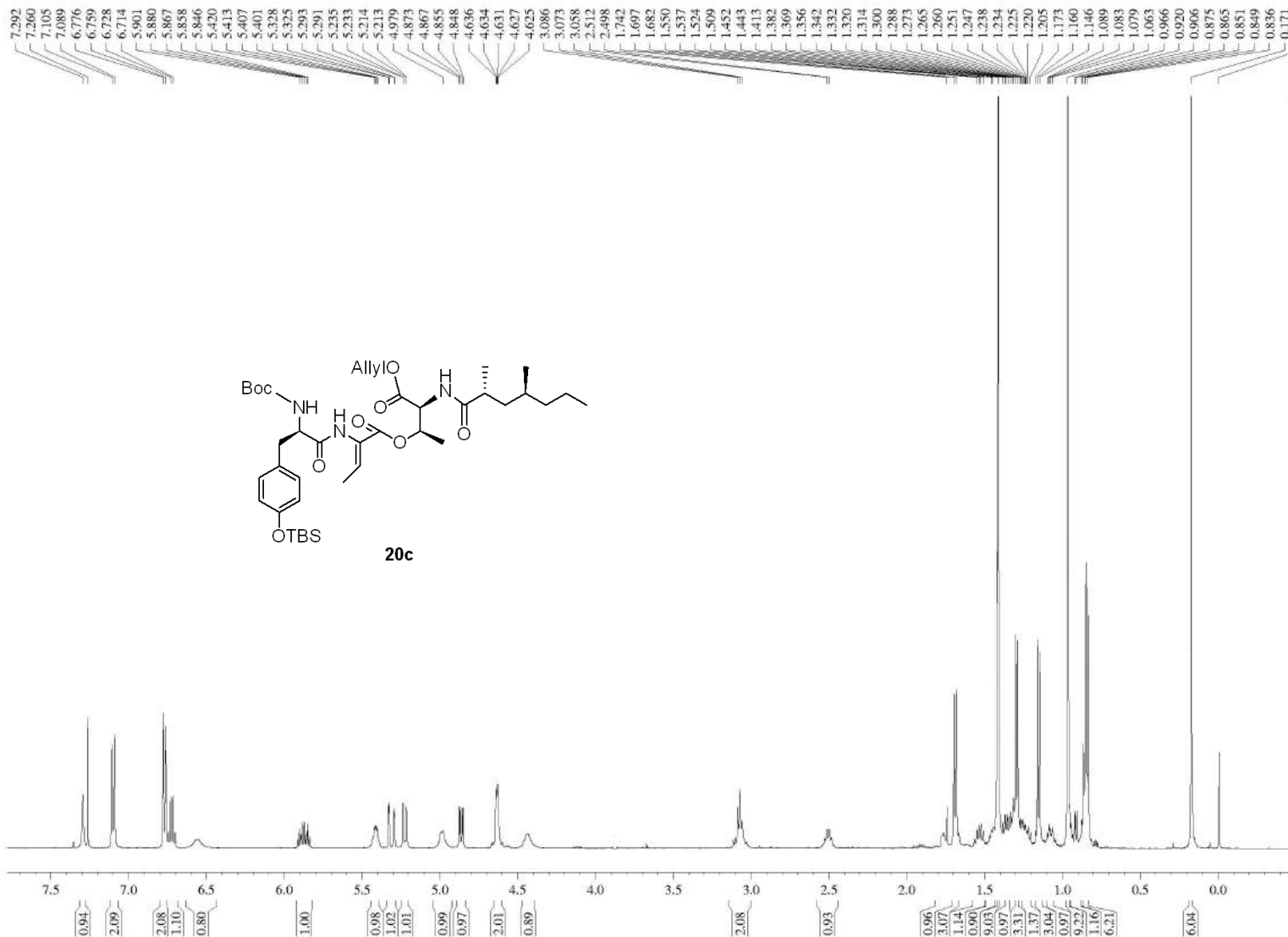
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577740 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

xh-ss-3-h2o



NAME 2020.12.23
EXPNO 23
PROCNO 1
Date_ 20201223
Time 17.26
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000
FIDRES 0.152588
AQ 3.2768500
RG 80.6
DW 50.000
DE 6.50
TE 298.0
D1 1.00000000
TD0 1

==== CHANNEL f1 ===
NUC1 1H
P1 13.50
PL1 2.00
PL1W 16.79986763
SFO1 500.1340010
SI 32768
SF 500.1300227
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



liushouxin-xh-SS-3-h2O

177.89
170.46
169.74
163.05
154.87
135.06
131.61
130.43
129.09
126.24
120.46
119.29

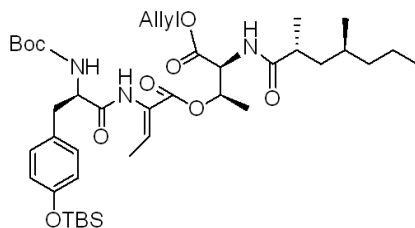
80.62
77.41
77.16
76.90
72.22
66.60
55.60
41.27
39.41
39.02
36.87
30.50
28.39
25.78
20.10
20.07
19.73
19.15
18.30
18.23
17.07
14.47
14.43
0.11
-4.33



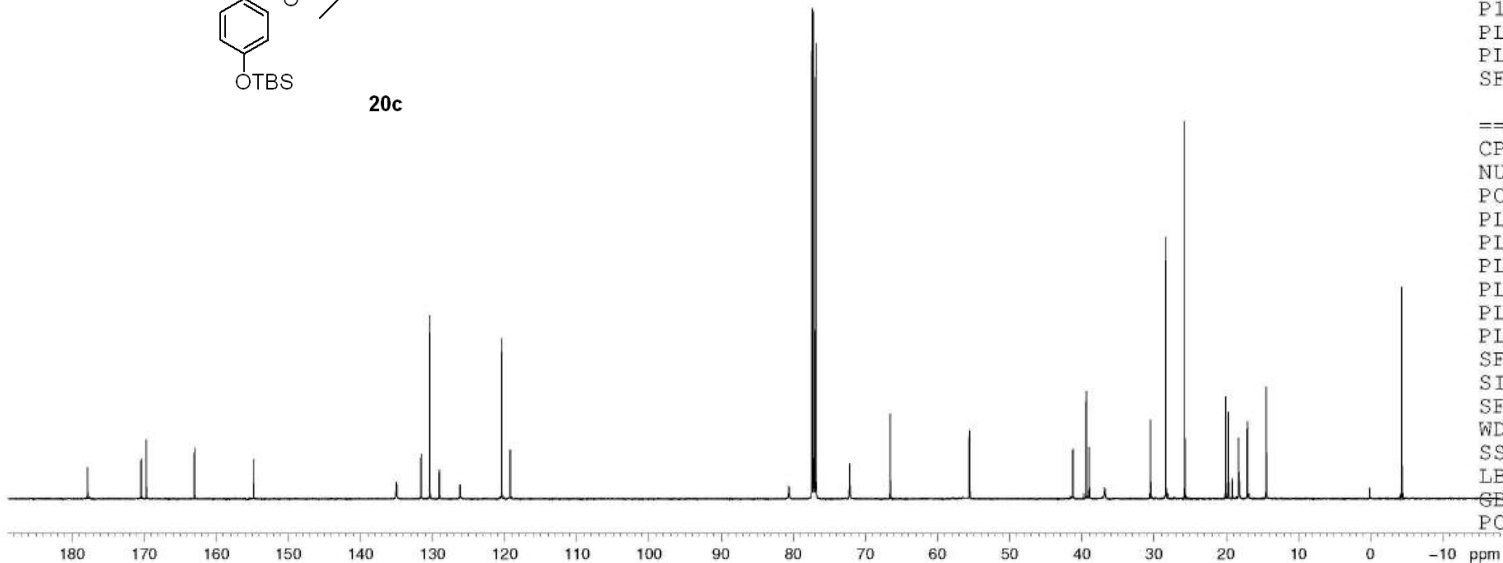
NAME 2021.03.11
EXPNO 17
PROCNO 1
Date_ 20210323
Time 16.52
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 1820
DW 16.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

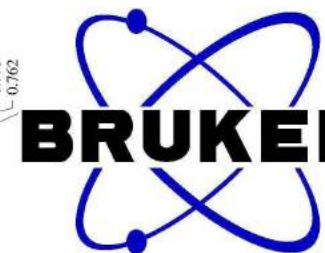
==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577758 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



20c

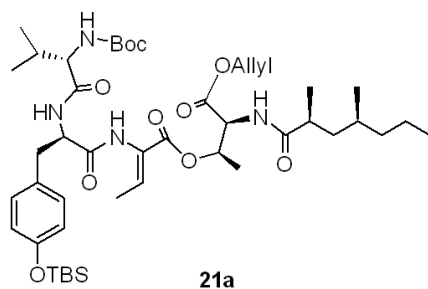
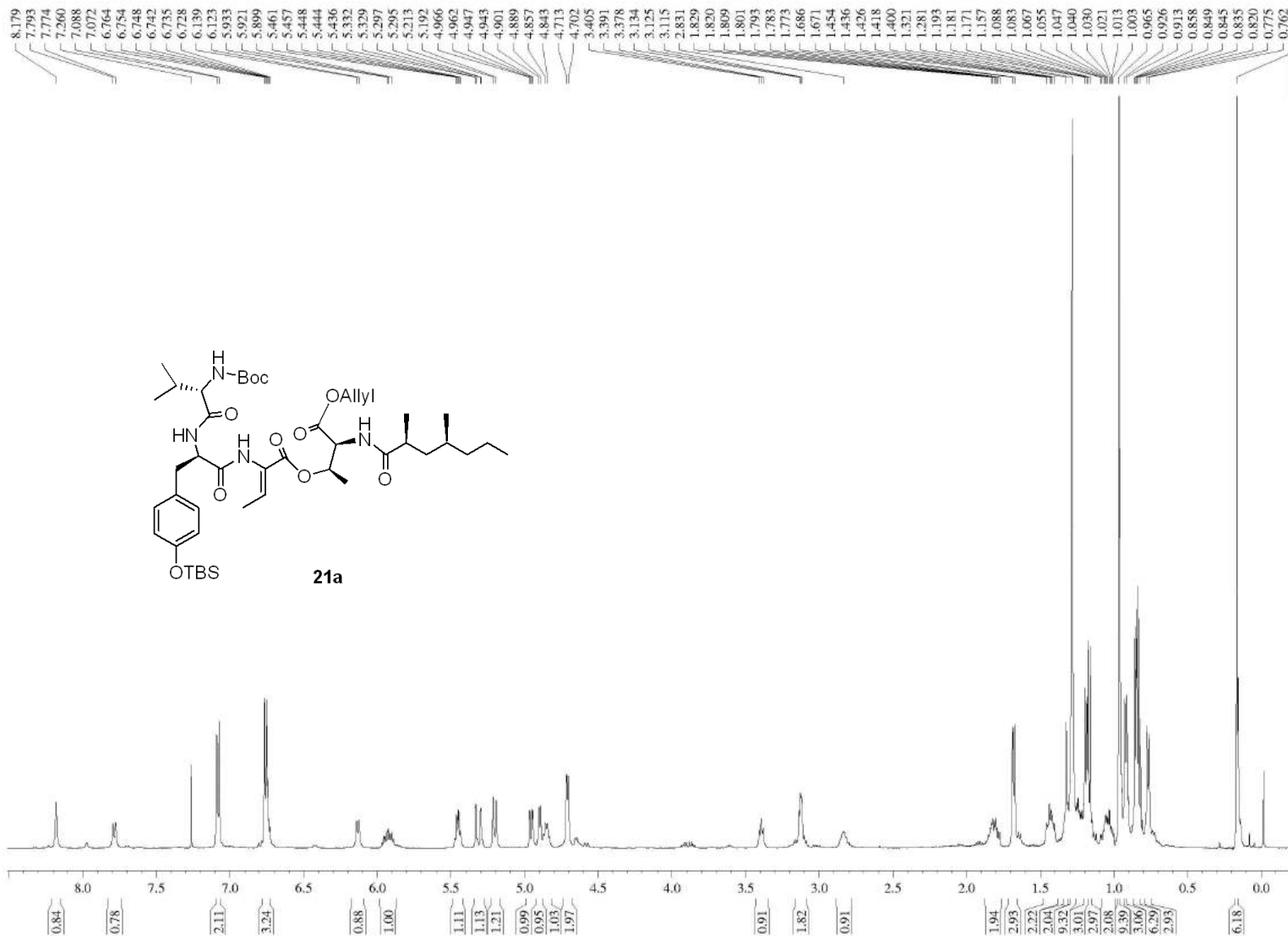


liushouxin-xh-SR-4



NAME 2021.03.11
 EXPNO 1
 PROCNO 2
 Date_ 20210630
 Time 17.41
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 80.6
 DW 50.000
 DE 6.50
 TE 296.4
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300227
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



liushouxin-xh-SR-4

178.08
172.35
170.62
169.45
163.29
156.75
155.03
135.49
132.22
130.34
129.04
127.90
120.59
118.56

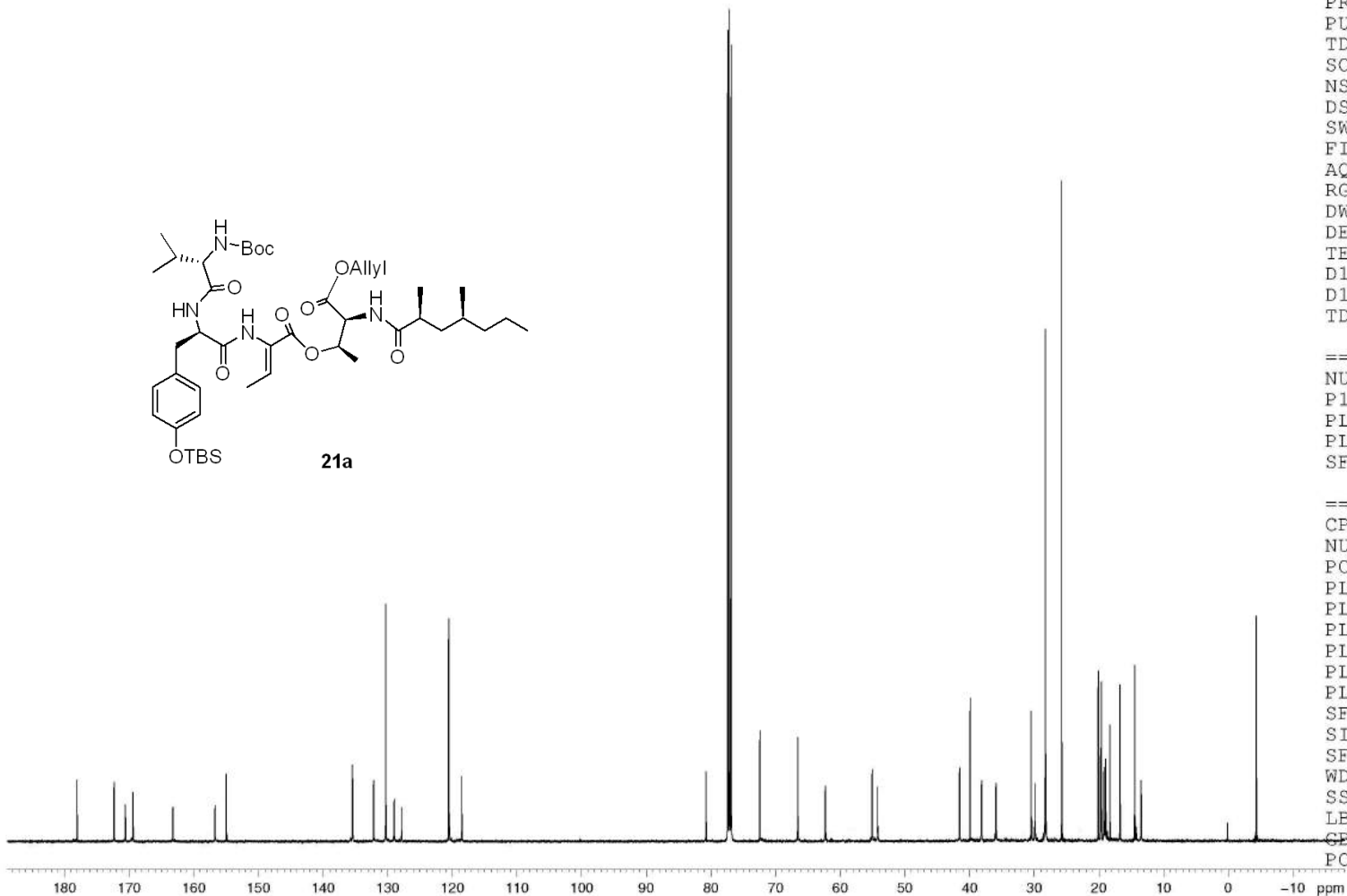
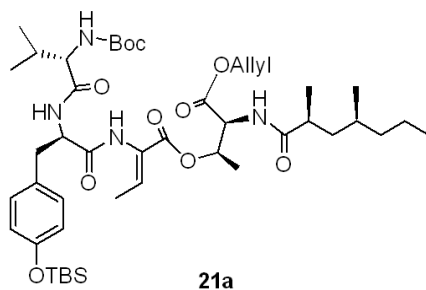
80.79
77.41
77.16
76.91
72.46
66.60
62.32
55.09
54.25
41.56
39.93
38.15
35.93
30.51
29.93
28.34
28.29
25.78
20.11
19.74
19.63
19.22
19.19
18.96
18.31
16.75
14.47
13.48
0.11
-4.33



NAME 2021.03.11
EXPNO 4
PROCNO 1
Date_ 20210312
Time 15.13
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577757 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



liushouxin-xh-RS-4

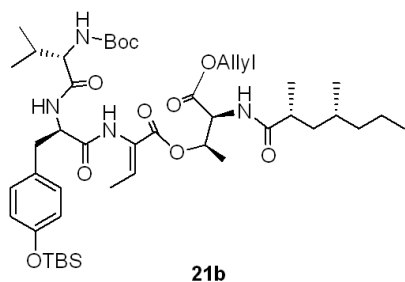
178.35
172.36
170.86
169.42
165.91
163.32
156.74
154.94

135.41
132.26
130.32
129.23
128.09
120.52
118.43

80.66
77.41
77.16
76.90
72.00
66.61
62.25
55.22
54.30
41.57
39.92
38.72
38.22
35.75
30.67
29.89
28.22
25.75
20.13
19.58
19.11
18.81
18.28
16.67
14.38
0.08
-4.37

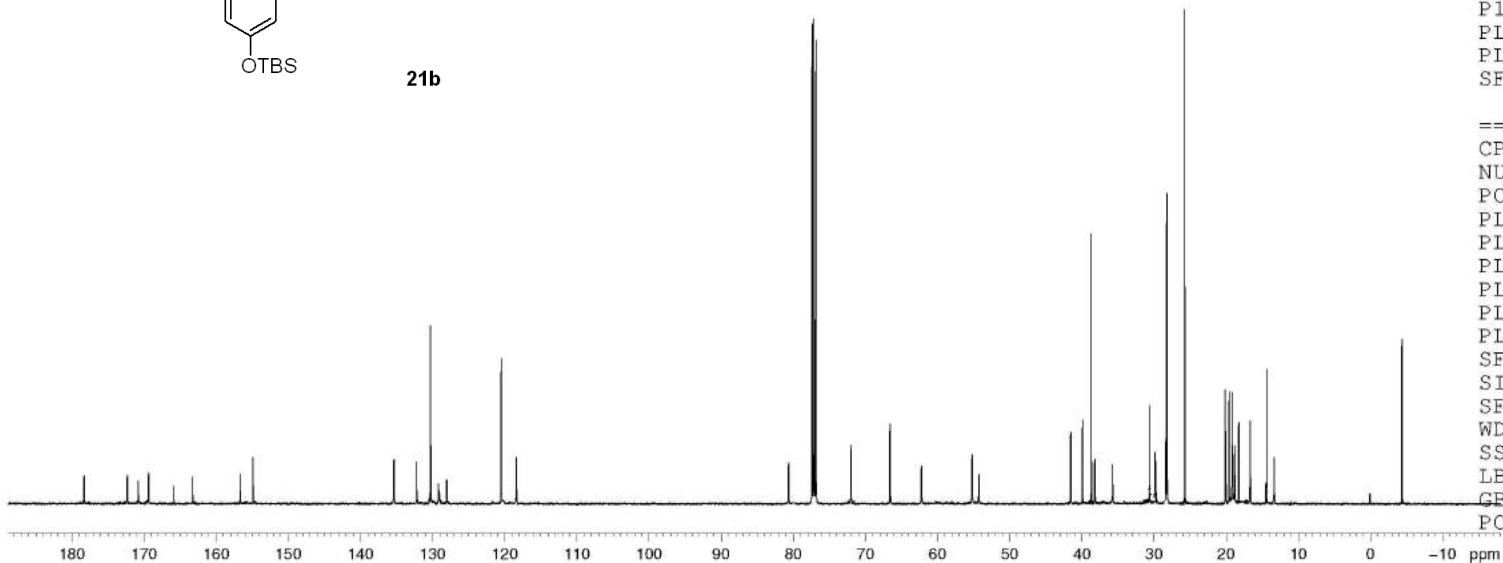


NAME 2021.03.11
EXPNO 8
PROCNO 1
Date_ 20210315
Time 17.03
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

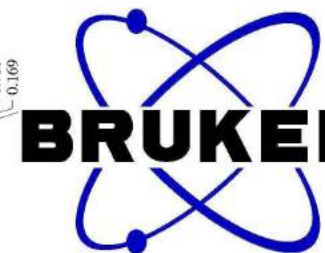


==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577784 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

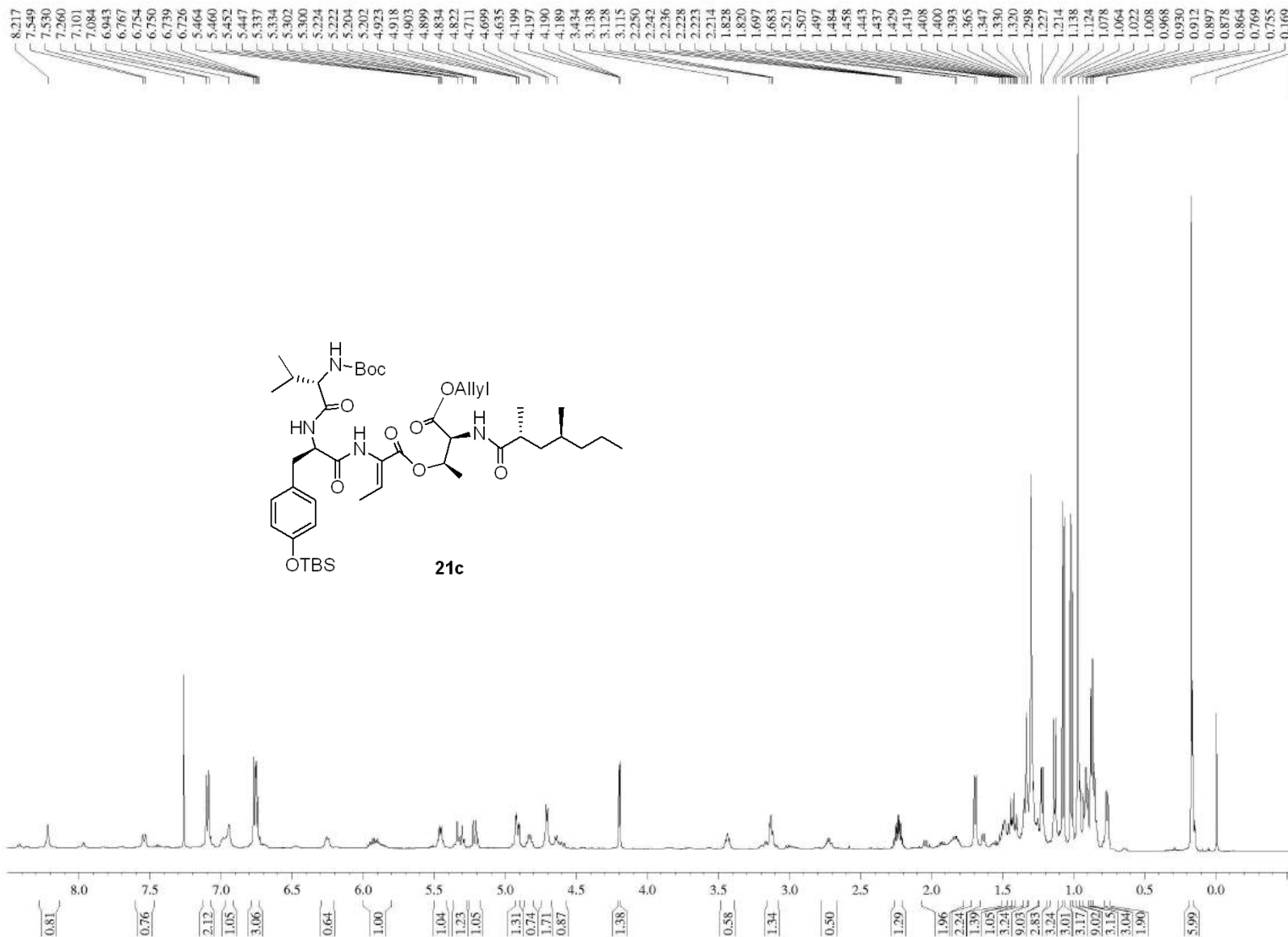


liushouxin-xh-SS-4

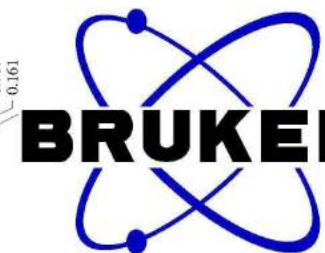


NAME 2020.12.29
 EXPNO 7
 PROCNO 1
 Date_ 20201229
 Time 11.02
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 512
 DW 50.000
 DE 6.50
 TE 298.0
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300227
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



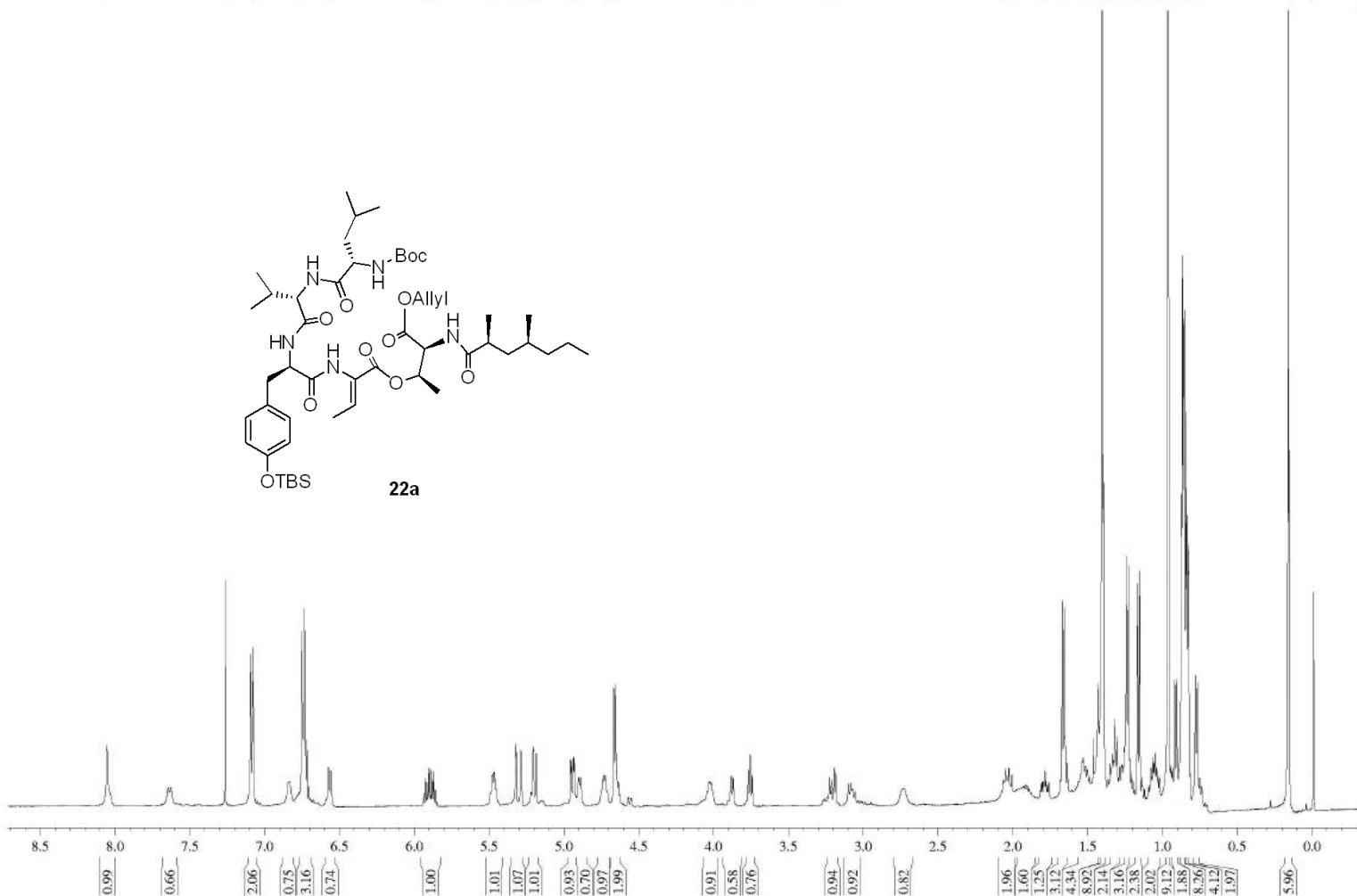
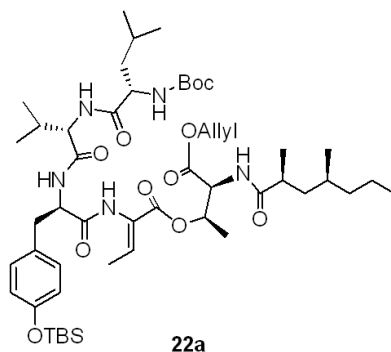
liushouxin-xh-SR-5



NAME 2021.03.11
 EXPNO 2
 PROCNO 2
 Date_ 20210630
 Time 17.45
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 80.6
 DW 50.000
 DE 6.50
 TE 296.6
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300229
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

8.051 7.260 7.092 7.075 6.748 6.731 6.716 6.572 6.556 5.902 5.888 5.887 5.473 5.465 5.461 5.319 5.316 5.285 5.282 5.204 5.202 5.183 4.953 4.948 4.934 4.929 4.900 4.734 4.722 4.664 4.652 3.879 3.866 3.765 3.752 3.739 3.221 3.193 3.182 2.050 2.026 2.007 1.786 1.671 1.656 1.650 1.635 1.529 1.515 1.503 1.488 1.462 1.430 1.404 1.350 1.332 1.326 1.317 1.303 1.240 1.227 1.167 1.153 1.076 1.071 1.065 1.056 1.048 1.039 1.030 1.020 0.963 0.918 0.905 0.872 0.866 0.858 0.853 0.843 0.837 0.828 0.825 0.780 0.767 0.161



liushouxin-xh-SR-5

178.01
173.58
171.64
170.02
169.66
163.18
154.88

134.95
132.02
130.29
129.34
127.28
120.47
120.39
118.81

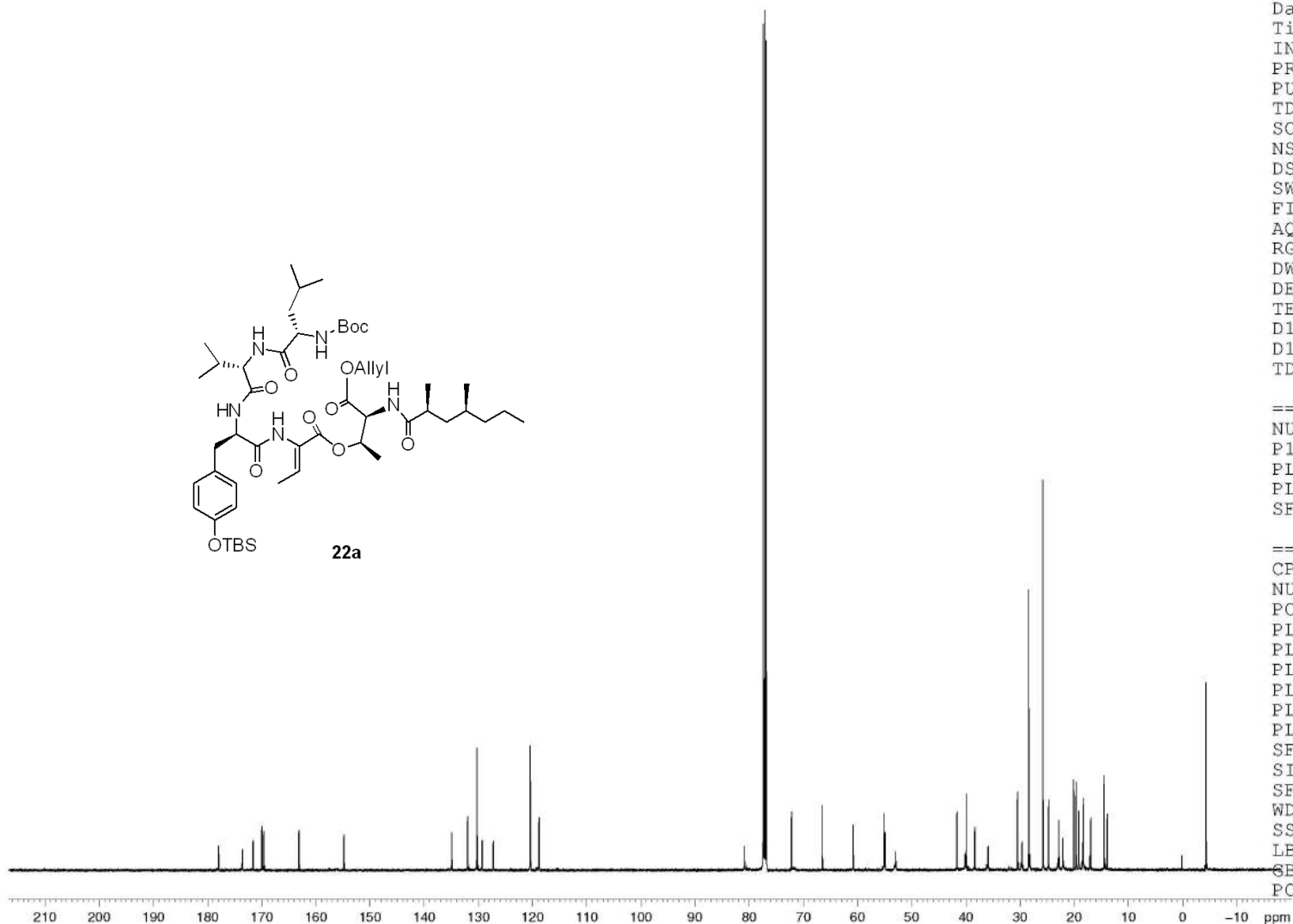
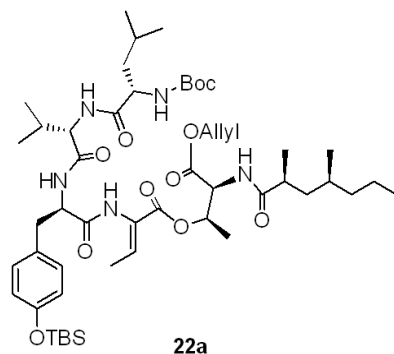
80.89
77.42
77.16
76.91
72.19
66.51
60.83
55.16
54.95
52.97
41.68
39.93
38.35
35.91
30.48
29.64
28.41
25.79
24.73
22.84
22.13
20.10
19.71
19.62
19.19
19.16
18.40
18.31
16.96
14.47
13.91



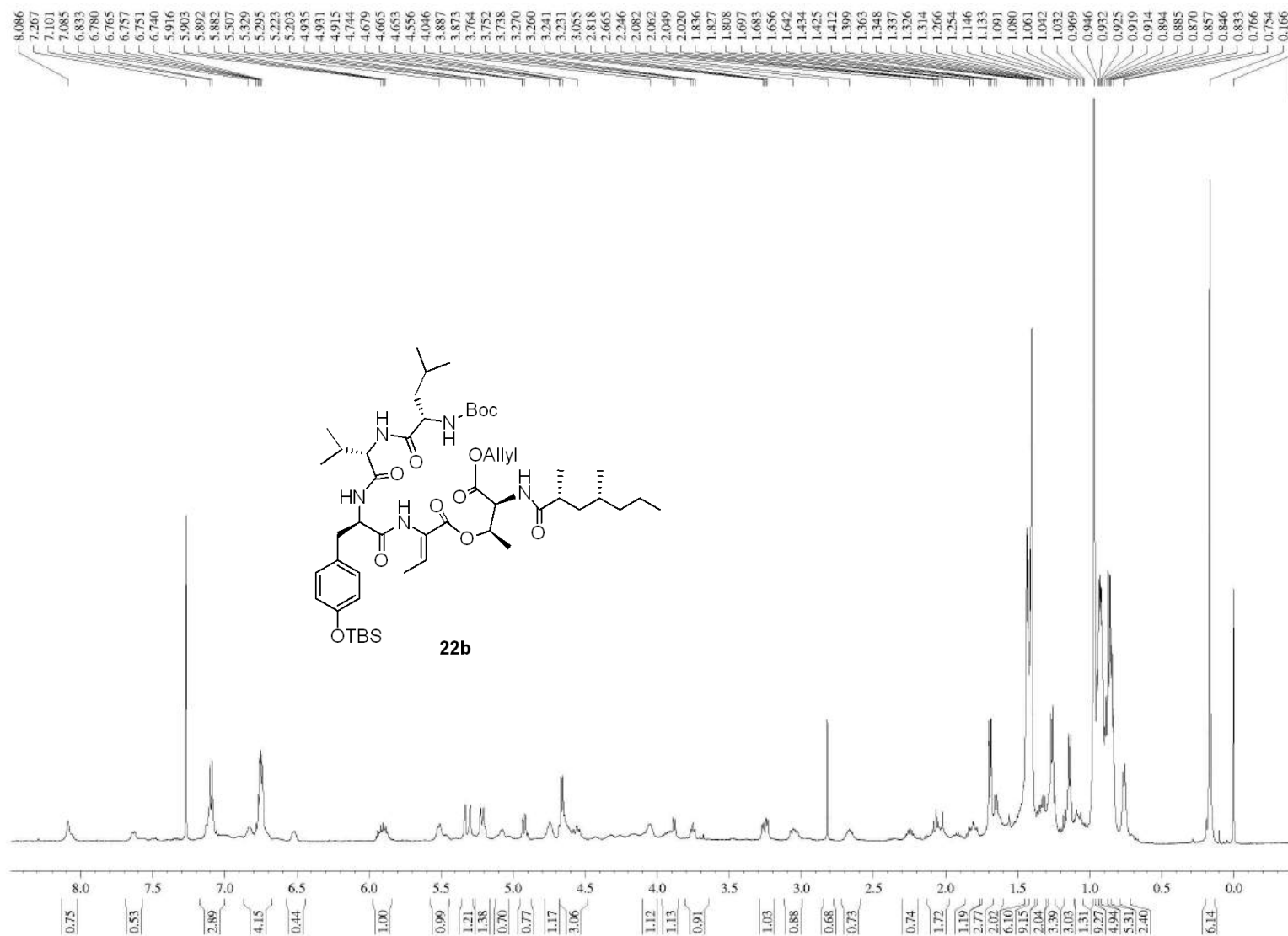
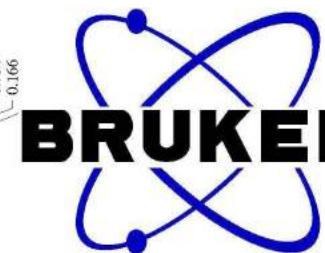
NAME 2021.03.11
EXPNO 3
PROCNO 1
Date_ 20210311
Time 17.14
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577746 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



liushouxin-xh-RS-5



```

NAME          2021.03.11
EXPNO         5
PROCNO        1
Date_         20210313
Time          15.31
INSTRUM       spect
PROBHD        5 mm PADUL 13C
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            0
SWH           10000.000
FIDRES        0.152588
AQ            3.2768500
RG            80.6
DW            50.000
DE            6.50
TE            295.1
D1            1.00000000
TD0           1
  
```

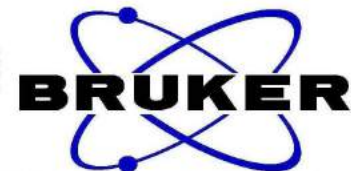
```

===== CHANNEL f1 =====
NUC1          1H
P1            13.50
PL1           2.00
PL1W         16.79986763
SFO1         500.1340010
SI            32768
SF           500.1300193
WDW           EM
SSB           0
LB            0.30
GB            0
RG            1.00
  
```


liushouxin-xh-RS-5

178.16
173.99
171.75
169.92
163.15
156.20
154.89
135.48
131.96
130.23
129.41
127.06
120.50
120.42
118.90

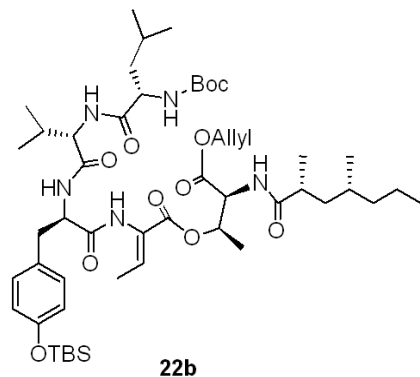
80.81
77.42
77.16
76.91
71.72
66.62
61.01
57.16
55.40
55.10
41.60
41.51
39.94
39.91
38.79
38.60
30.72
28.41
25.80
24.71
22.88
22.07
20.13
19.65
19.20
19.16
19.09
18.32
17.09
14.42
14.08
0.14
-4.32



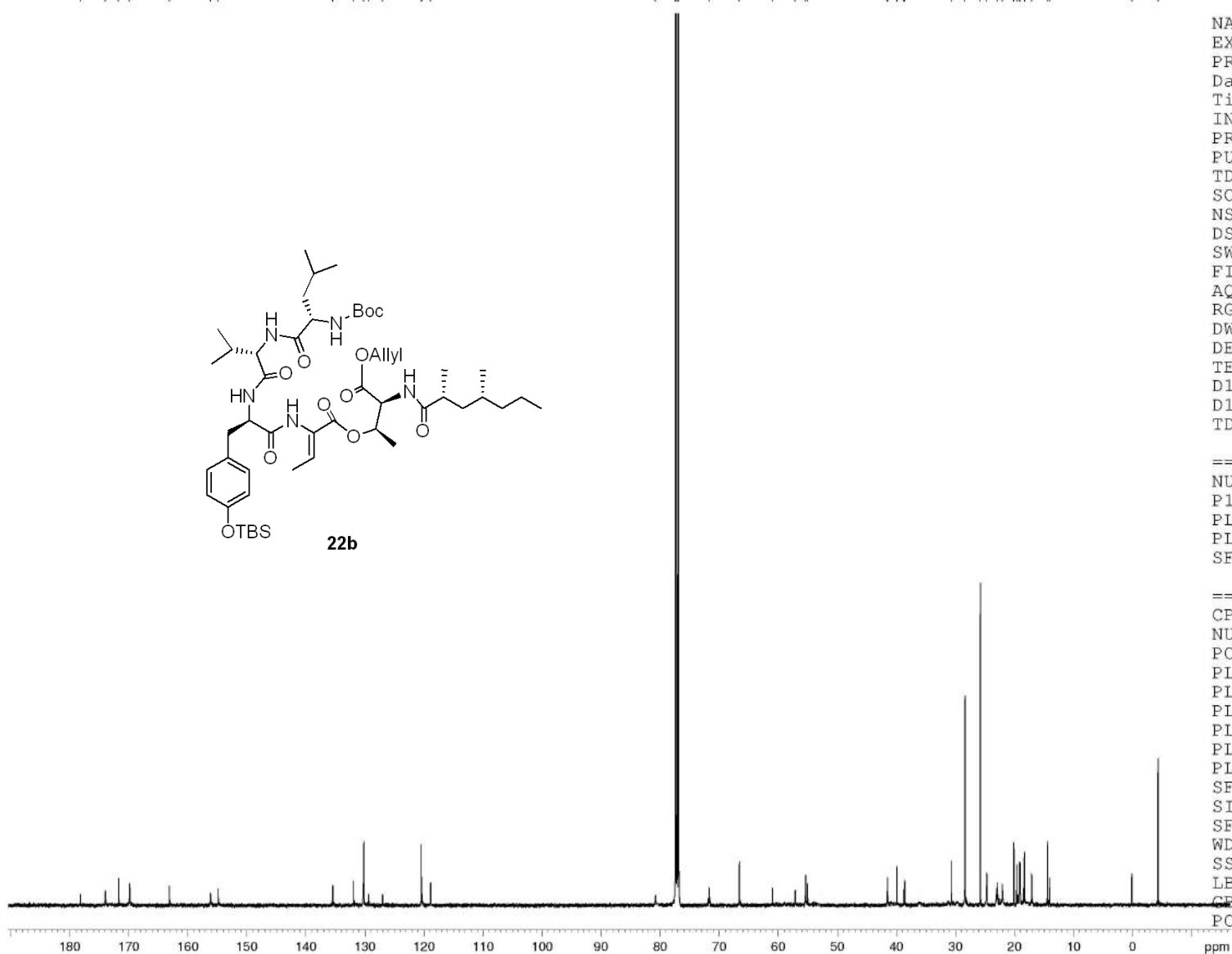
NAME 2021.03.11
EXPNO 6
PROCNO 1
Date_ 20210313
Time 15.33
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577735 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



22b



liushouxin-xh-SS-5

178.55
173.80
171.73
170.00
169.88
163.13
156.18
154.83

135.27
131.89
130.25
129.43
127.03
120.44
120.38
118.96

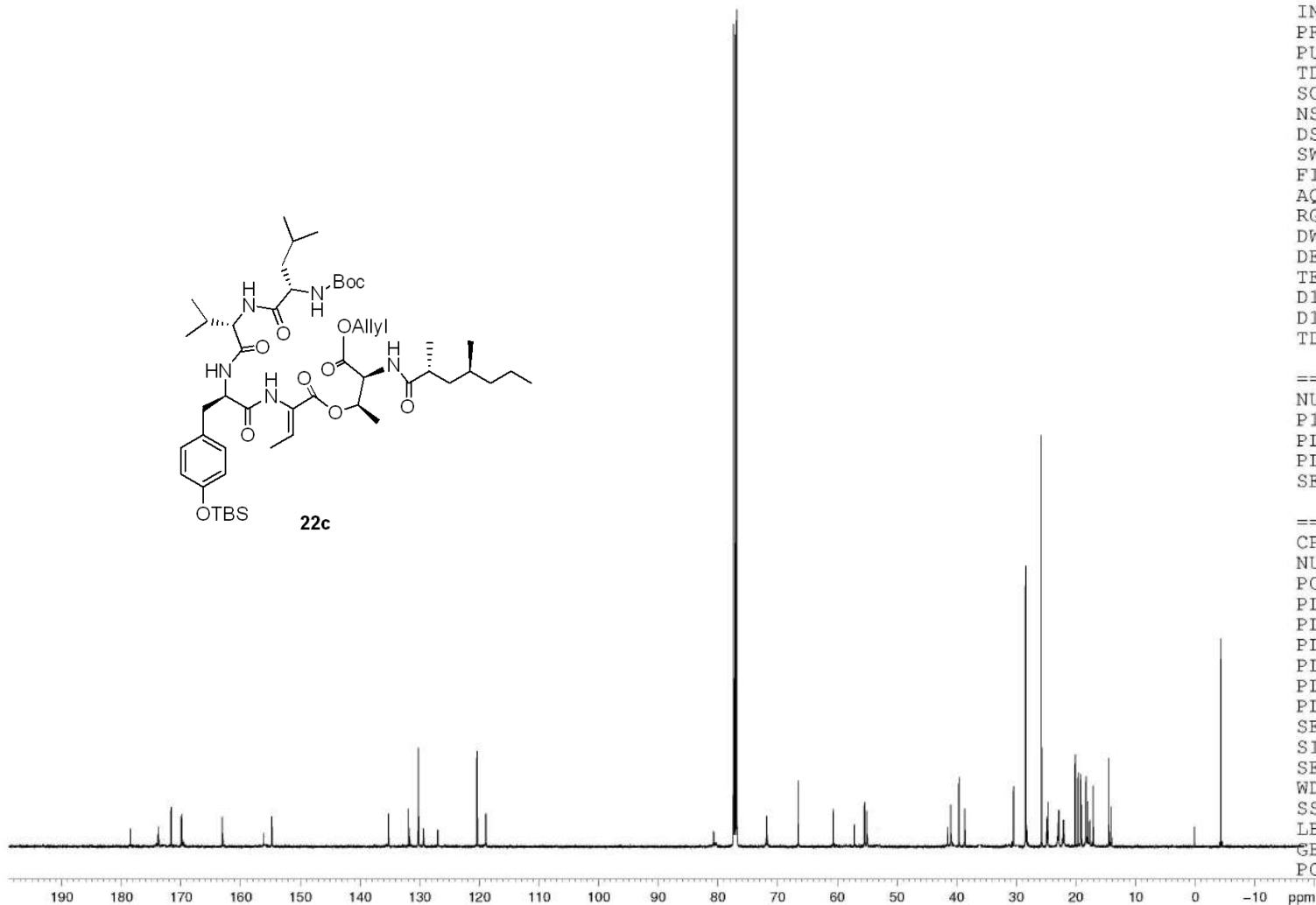
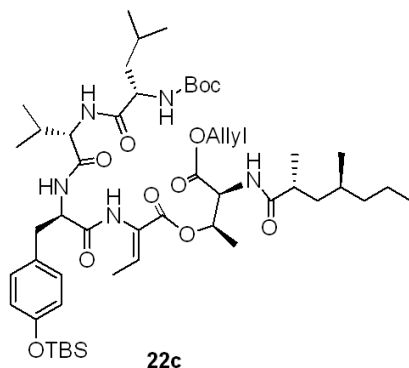
80.75
77.42
77.16
76.91
71.87
66.59
60.70
57.18
55.44
55.08
41.53
41.00
39.62
38.64
30.46
28.40
25.79
24.80
24.73
22.88
22.10
20.11
19.67
19.15
18.30
18.07
17.70
17.11
14.51
14.06
0.12
-4.33



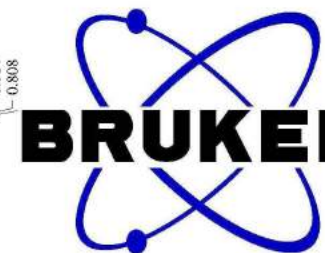
NAME 2021.03.11
EXPNO 9
PROCNO 1
Date_ 20210316
Time 16.53
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 10240
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

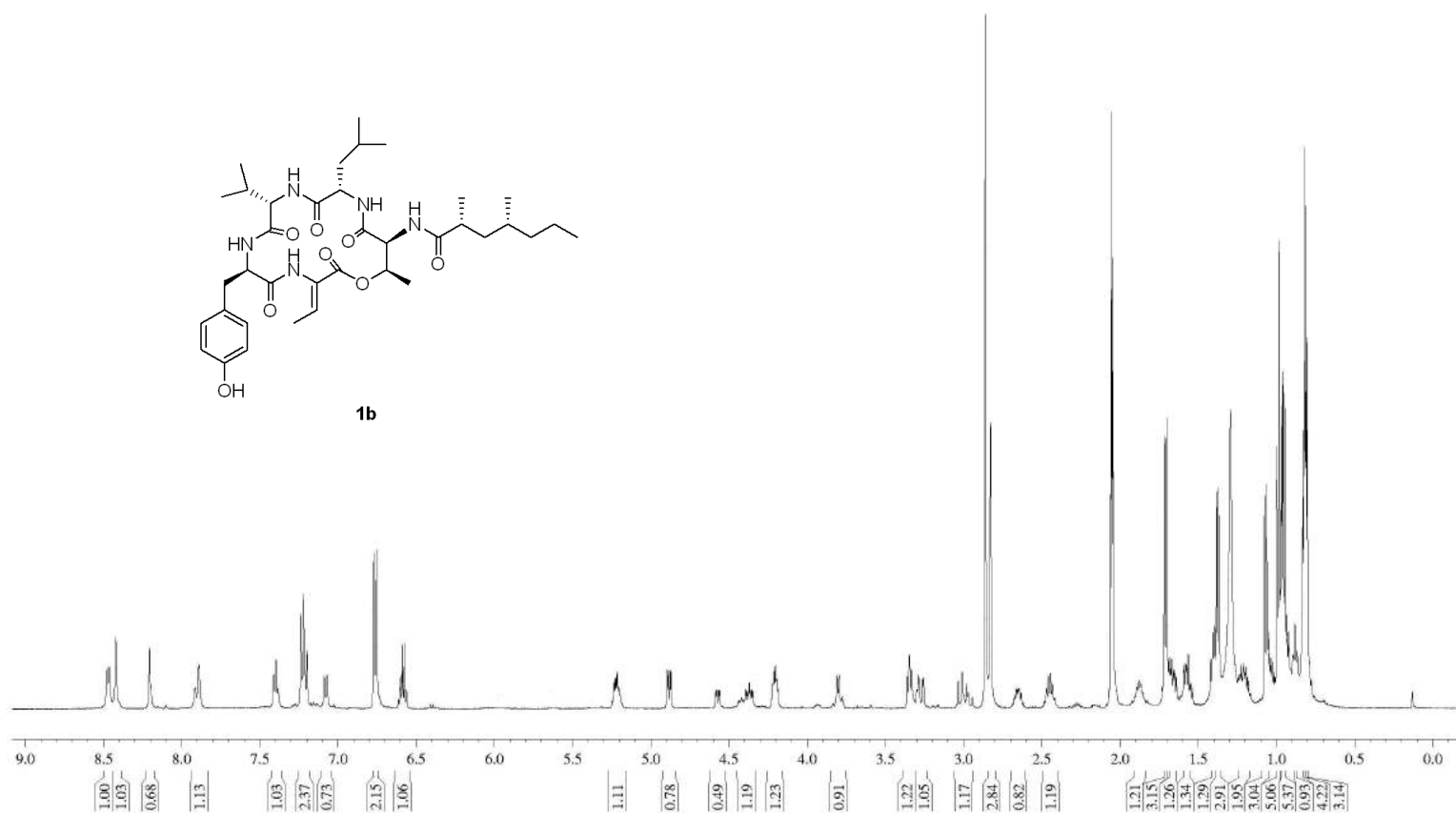
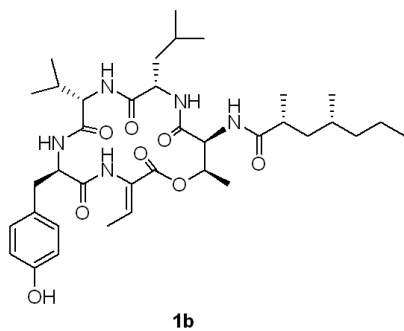
==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577751 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



liushouxin-xh-RS-TBAF-TLC-1



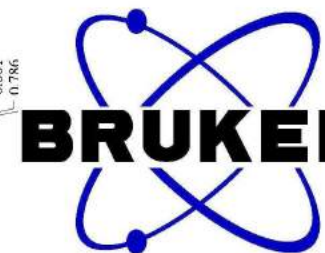
8.474 8.460 8.419 8.203 7.890 7.885 7.409 7.394 7.234 7.217 7.196 7.086 7.068 6.769 6.752 6.597 6.588 6.583 6.573 5.220 5.214 4.893 4.886 4.875 4.869 4.200 4.202 3.805 3.793 3.358 3.344 3.332 3.288 3.259 3.253 3.007 2.859 2.825 2.454 2.441 2.059 2.055 2.050 2.046 2.041 1.713 1.699 1.684 1.674 1.674 1.656 1.649 1.638 1.588 1.577 1.571 1.561 1.418 1.404 1.392 1.379 1.367 1.288 1.243 1.229 1.222 1.211 1.204 1.199 1.193 1.184 1.072 1.059 0.994 0.979 0.965 0.957 0.944 0.890 0.878 0.864 0.821 0.814 0.808



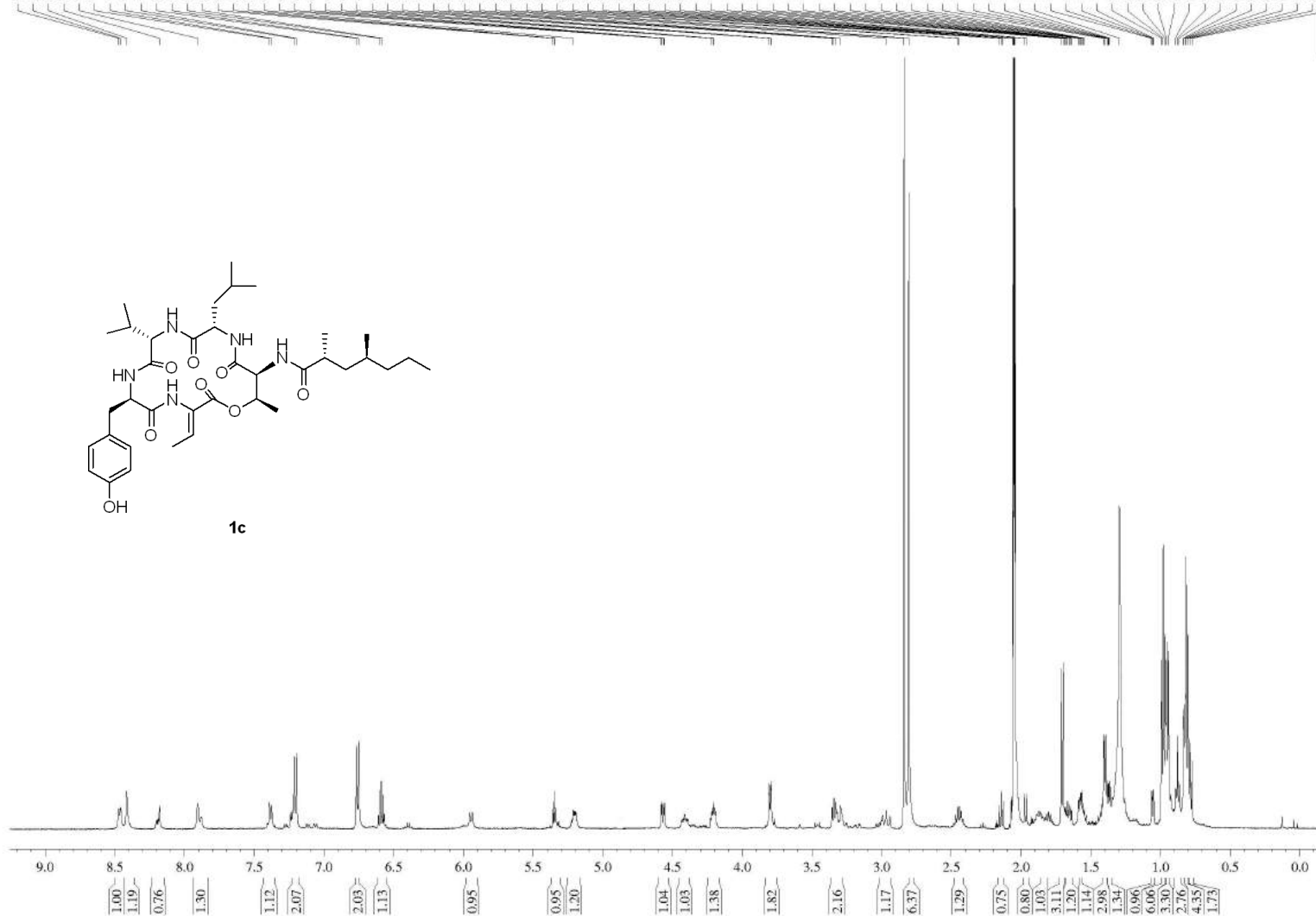
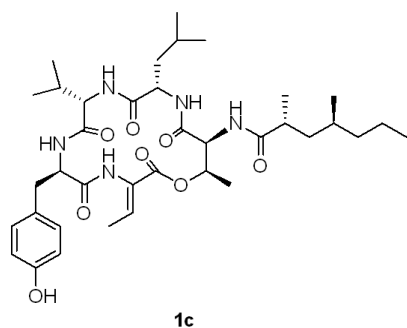
NAME 2021.04.08
 EXPNO 4
 PROCNO 1
 Date_ 20210408
 Time 14.20
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 32
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 256
 DW 50.000
 DE 6.50
 TE 294.9
 D1 1.00000000
 TD0 1

==== CHANNEL f1 ===
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300146
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 GC 1.00

liushouxin-xh-SS-TBAF-TLC



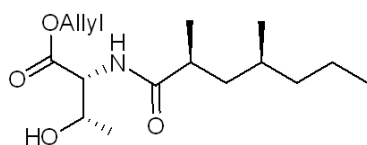
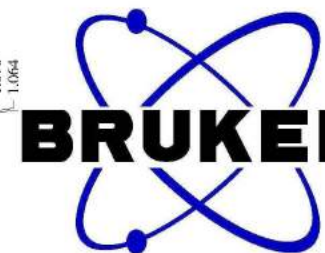
8.472 8.457 8.415 8.179 7.904 7.391 7.375 7.213 7.196 6.768 6.751 6.595 6.581 5.358 5.348 5.339 5.211 4.580 4.562 4.575 4.556 4.216 4.209 4.201 4.201 3.805 3.795 3.792 3.355 3.341 3.329 3.295 2.967 2.839 2.805 2.455 2.443 2.158 2.143 2.128 2.059 2.054 2.050 2.046 2.041 2.041 1.974 1.959 1.712 1.698 1.683 1.676 1.667 1.655 1.648 1.637 1.588 1.577 1.571 1.561 1.550 1.544 1.405 1.392 1.382 1.374 1.369 1.361 1.294 1.063 1.060 1.049 1.046 0.992 0.979 0.966 0.956 0.943 0.892 0.879 0.865 0.835 0.830 0.821 0.815 0.801 0.786



NAME 2021.04.08
 EXPNO 6
 PROCNO 1
 Date_ 20210408
 Time 14.40
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 64
 DS 0
 SWH 10000.000
 FIDRES 0.152588
 AQ 3.2768500
 RG 456
 DW 50.000
 DE 6.50
 TE 294.9
 D1 1.00000000
 TD0 1

==== CHANNEL f1 ===
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300147
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

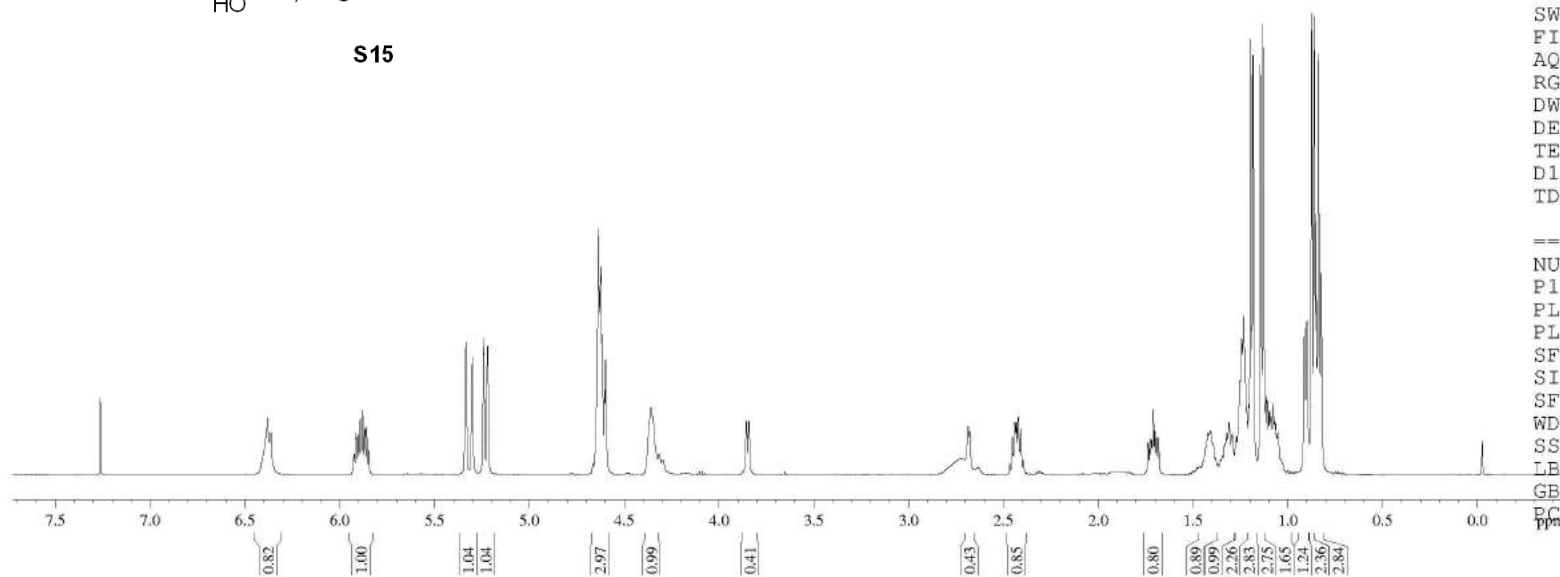
liushouxin-xh-SR-DT



S15

NAME 2021.05.27
 EXPNO 1
 PROCNO 1
 Date_ 20210527
 Time 14.35
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9014.423
 FIDRES 0.137549
 AQ 3.6351135
 RG 64
 DW 55.467
 DE 6.50
 TE 296.8
 D1 1.00000000
 TD0 1

==== CHANNEL f1 ===
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300229
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



liushouxin-xh-SR-DT

— 177.53
— 171.02

— 131.60

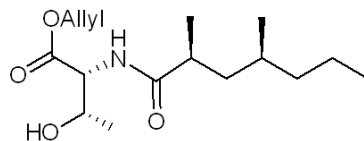
— 118.94

77.41
77.16
76.90
68.00
66.20
57.14

41.79
39.61
39.36
30.55
20.05
20.01
19.59
18.78
14.36



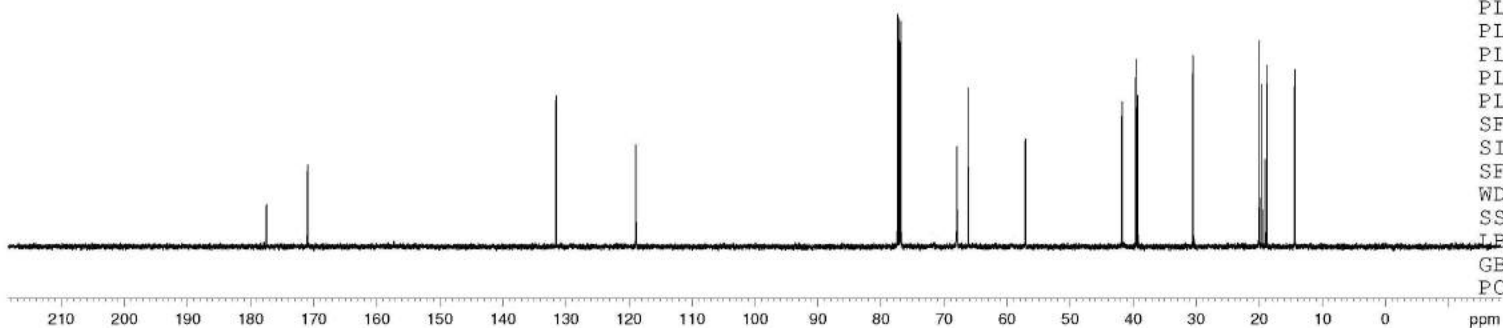
NAME 2021.05.27
EXPNO 10
PROCNO 1
Date_ 20210527
Time 15.52
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 182
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



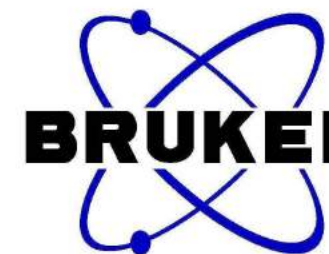
S15

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577778 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



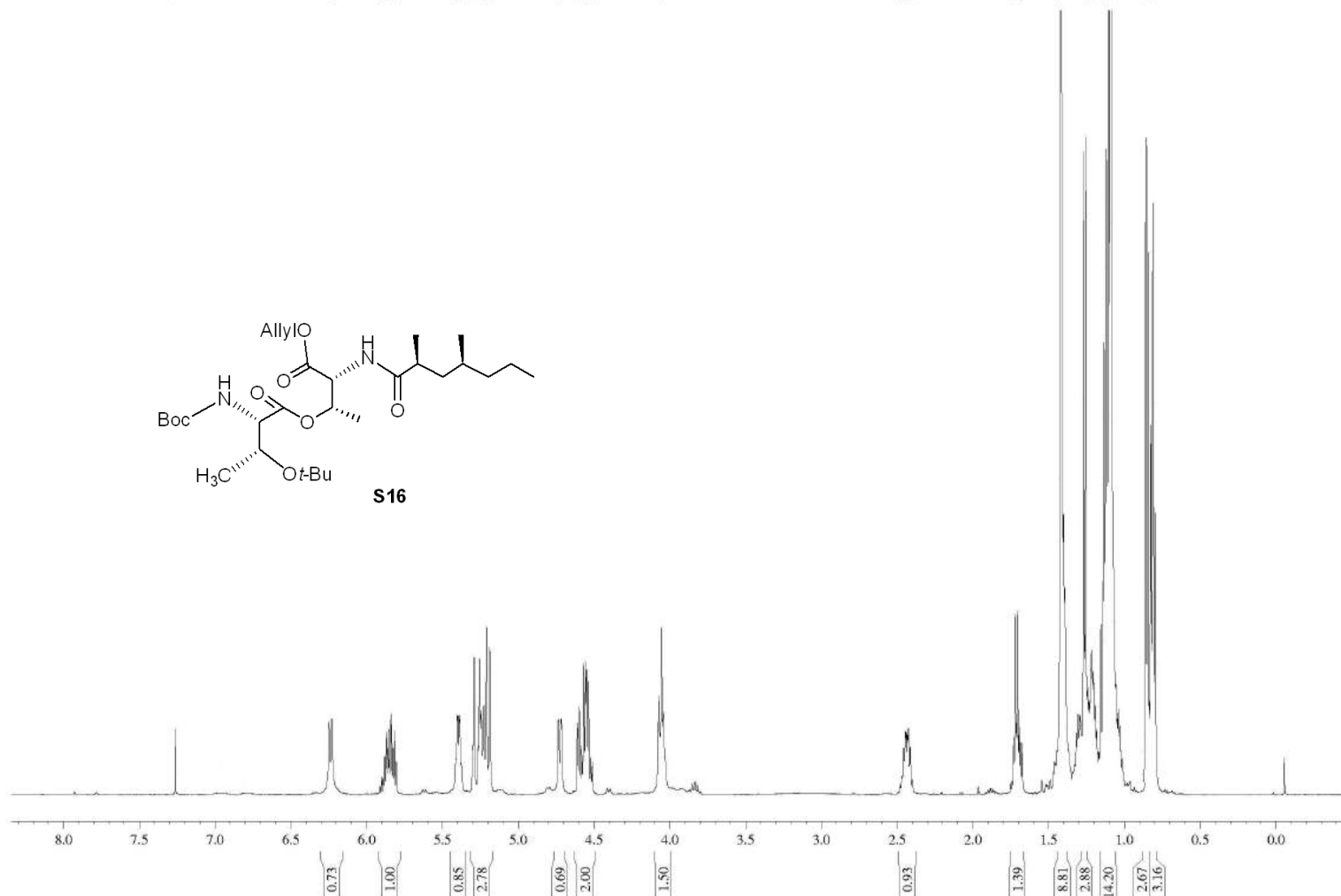
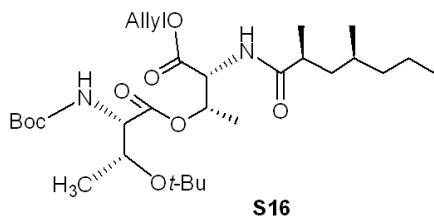
liushouxin-xh-SR-DT-T



NAME 2021.05.27
 EXPNO 2
 PROCNO 1
 Date_ 20210527
 Time 14.41
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 2
 DS 0
 SWH 9014.423
 FIDRES 0.137549
 AQ 3.6351135
 RG 20.2
 DW 55.467
 DE 6.50
 TE 296.8
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300225
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

7.260 6.246 6.228 5.899 5.888 5.879 5.868 5.857 5.847 5.834 5.823 5.813 5.802 5.400 5.396 5.387 5.383 5.297 5.289 5.287 5.262 5.255 5.252 5.243 5.225 5.207 5.207 5.186 4.731 4.716 4.607 4.596 4.580 4.566 4.554 4.546 4.534 4.520 4.508 4.069 4.052 4.041 2.478 2.455 2.444 2.437 2.430 2.424 2.412 2.398 1.732 1.720 1.713 1.706 1.694 1.686 1.675 1.421 1.270 1.257 1.157 1.142 1.135 1.121 1.103 1.092 0.860 0.847 0.828 0.813 0.799



liushouxin-xh-SR-DT-T

177.34
170.42
169.37
155.87
131.51
119.00

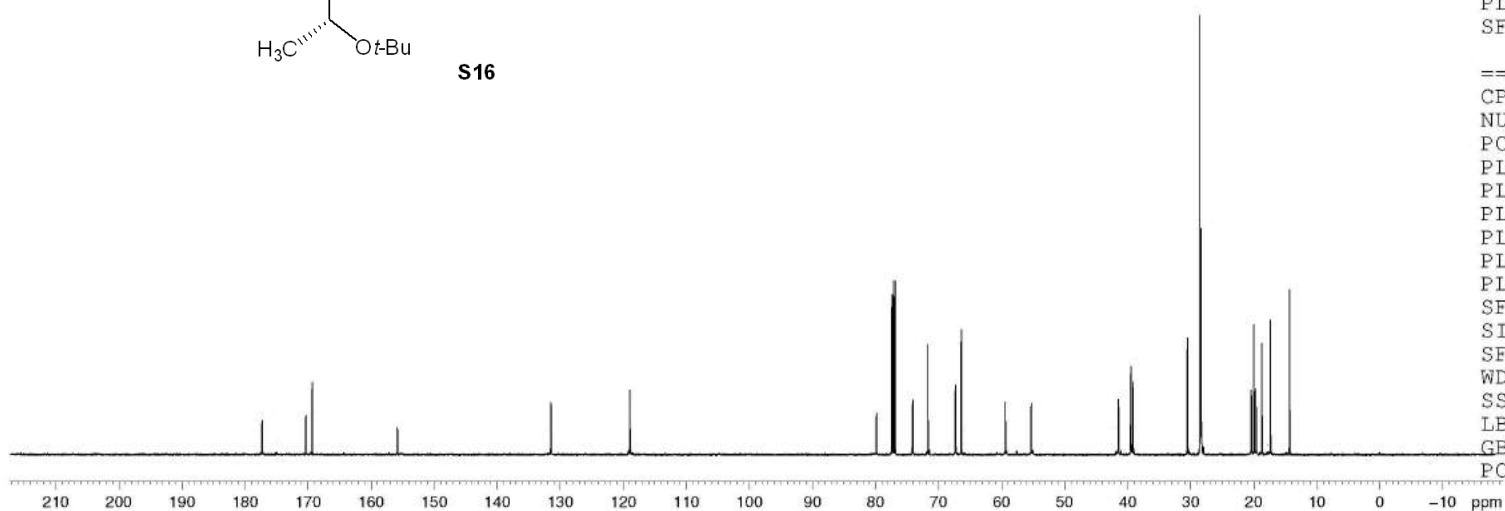
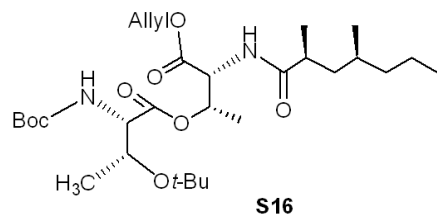
79.87
77.42
77.16
76.91
74.12
71.69
67.32
66.40
59.41
55.33
41.47
39.50
39.16
30.53
28.56
28.42
20.40
19.98
19.65
18.72
17.38
14.32



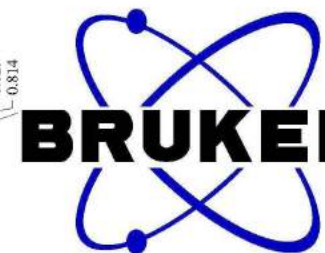
NAME 2021.05.27
EXPNO 11
PROCNO 1
Date_ 20210527
Time 15.59
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 940
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577803 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

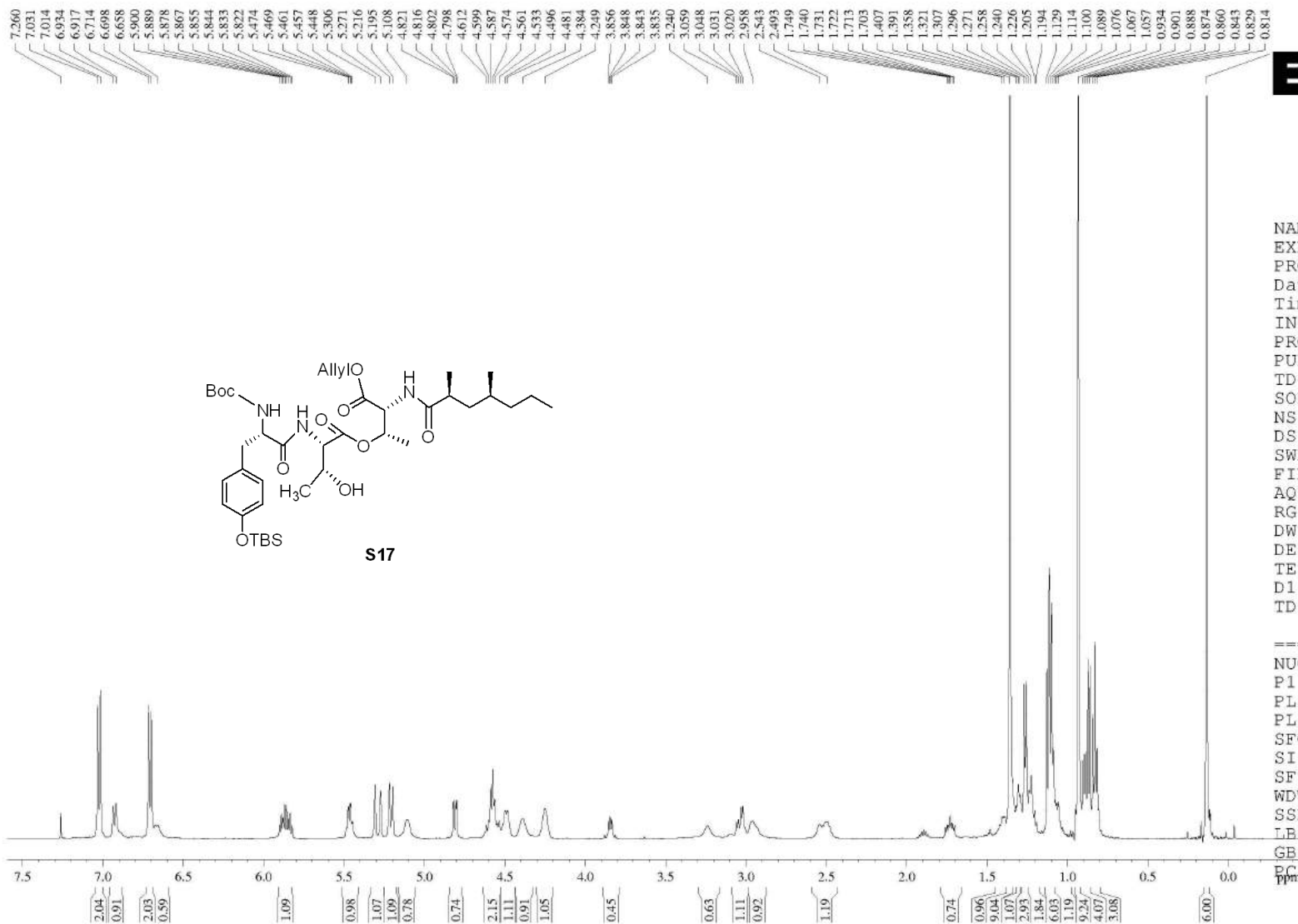


liushouxin-xh-SR-DT-3



NAME 2021.05.27
 EXPNO 3
 PROCNO 1
 Date_ 20210527
 Time 14.47
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 2
 DS 0
 SWH 9014.423
 FIDRES 0.137549
 AQ 3.6351135
 RG 20.2
 DW 55.467
 DE 6.50
 TE 296.7
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300225
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



liushouxin XH-SR-DT-3

177.56
172.34
170.18
169.35
155.60
154.67
131.50
130.34
129.07
120.21
119.16

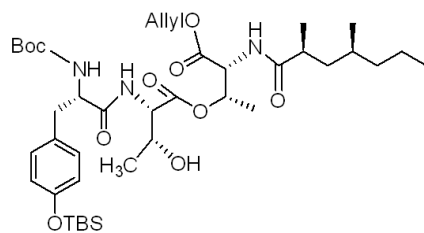
80.27
77.41
77.16
76.90
71.89
67.79
66.73
57.66
55.78
55.15
41.57
39.62
38.99
36.95
30.59
28.28
25.70
20.00
19.57
19.05
18.87
18.19
17.22
14.33
-4.42



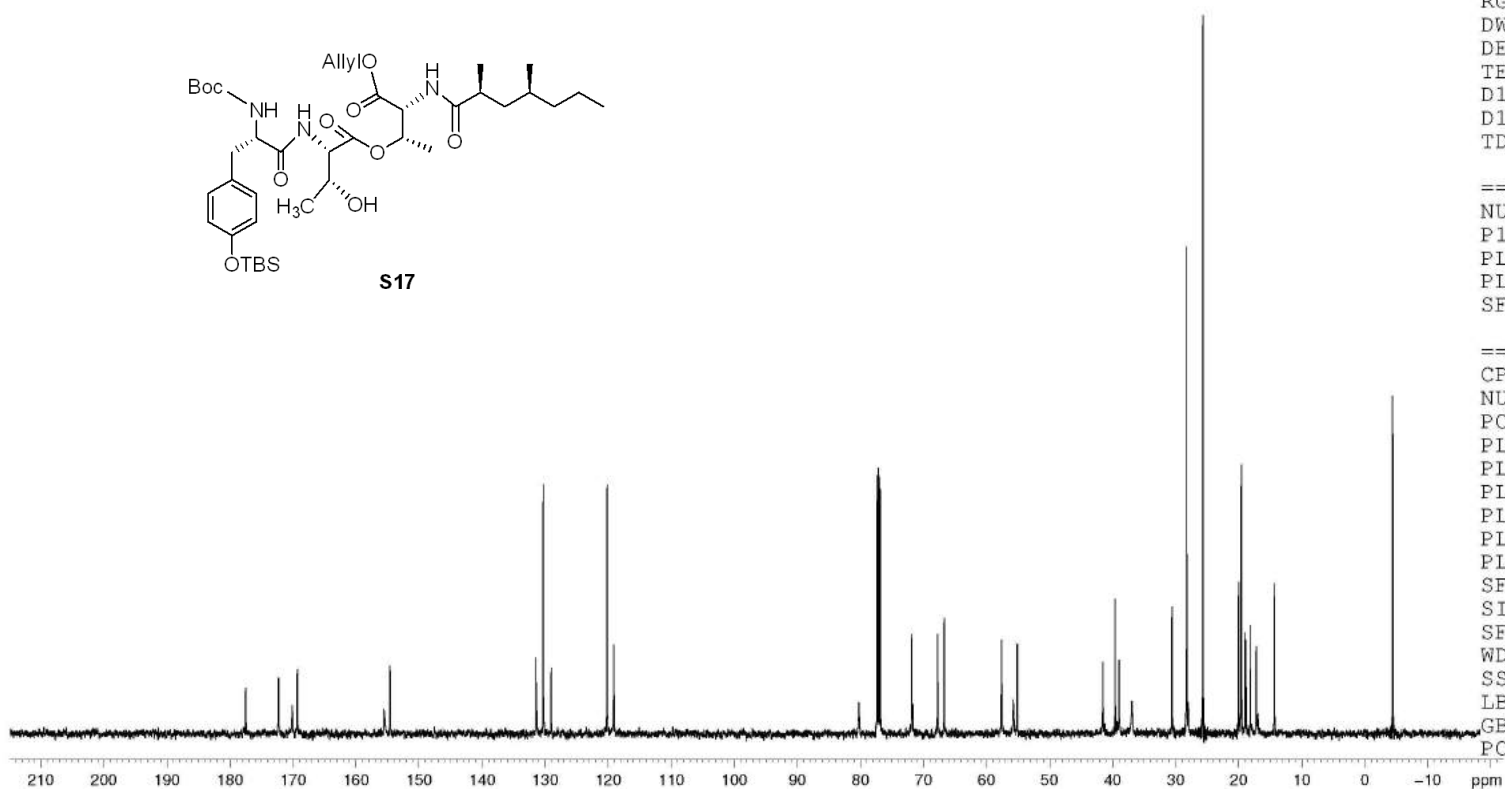
NAME 2021.05.27
EXPNO 41
PROCNO 1
Date_ 20210528
Time 11.38
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 10240
SOLVENT CDC13
NS 114
DS 4
SWH 29761.904 Hz
FIDRES 2.906436 Hz
AQ 0.1720820 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

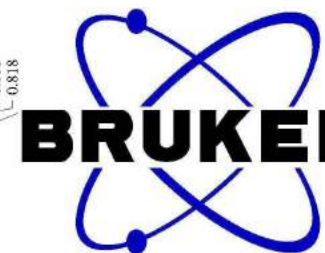
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577832 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



S17

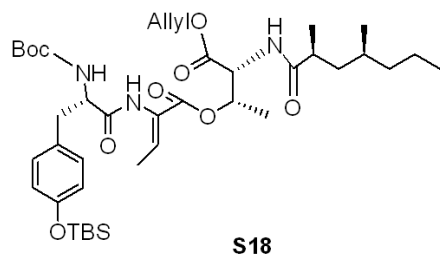
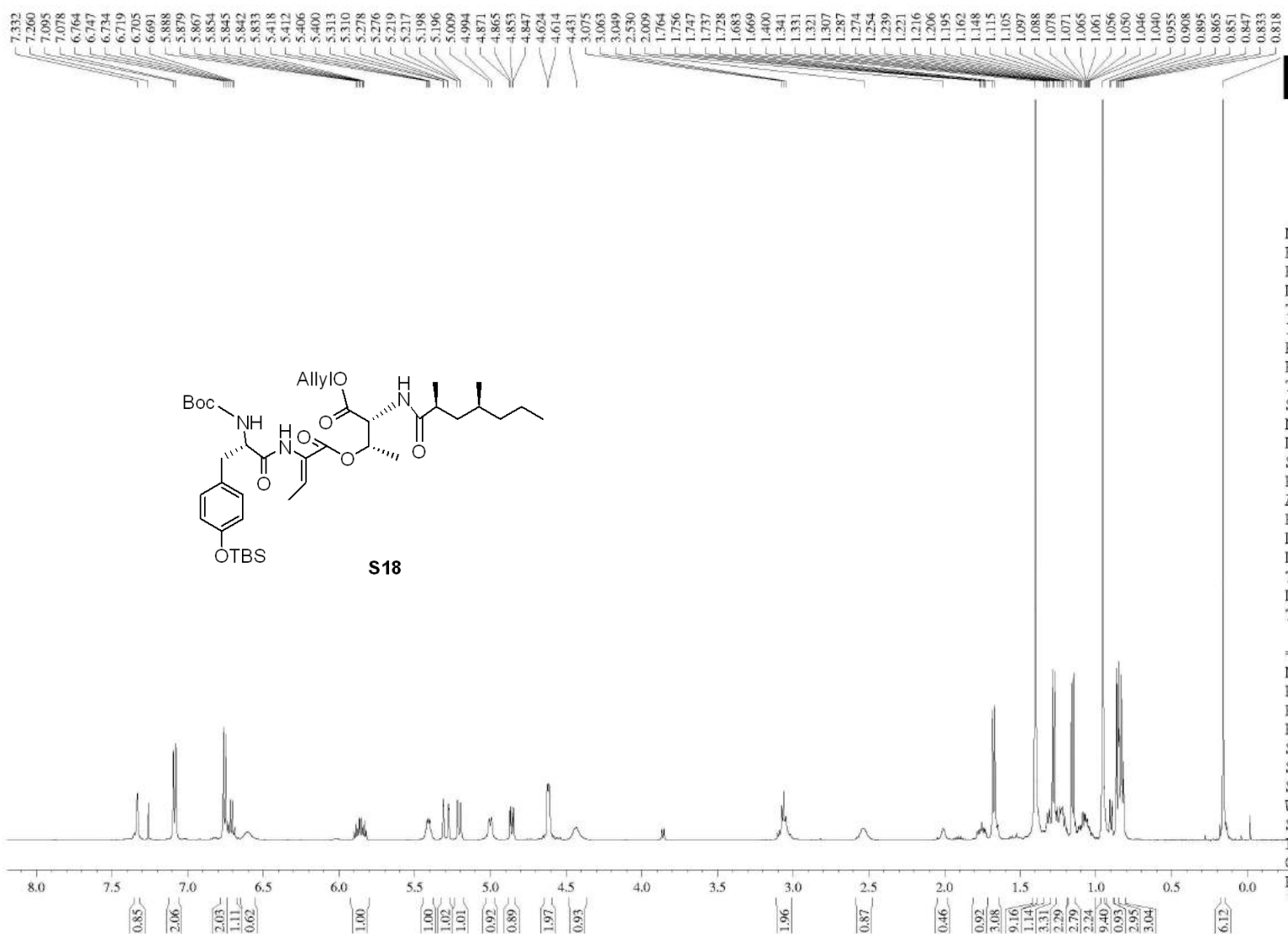


liushouxin-xh-SR-DT-3-H20



NAME 2021.05.27
 EXPNO 4
 PROCNO 1
 Date_ 20210527
 Time 14.57
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 9014.423
 FIDRES 0.137549
 AQ 3.6351135
 RG 50.8
 DW 55.467
 DE 6.50
 TE 296.8
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300227
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



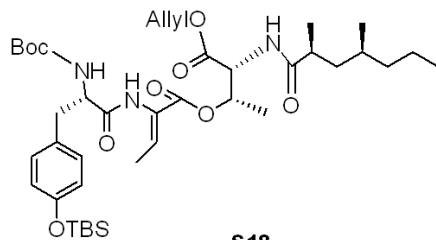
liushouxin XH-SR-DT-3-H2O

177.54
170.48
169.70
163.02
154.84
135.03
131.62
130.41
129.09
126.27
120.42
119.20

80.59
77.41
77.16
76.91
72.15
66.56
55.53
41.56
39.72
39.04
36.81
30.65
28.36
25.76
20.04
19.66
19.13
19.04
18.28
17.02
14.39
-4.35

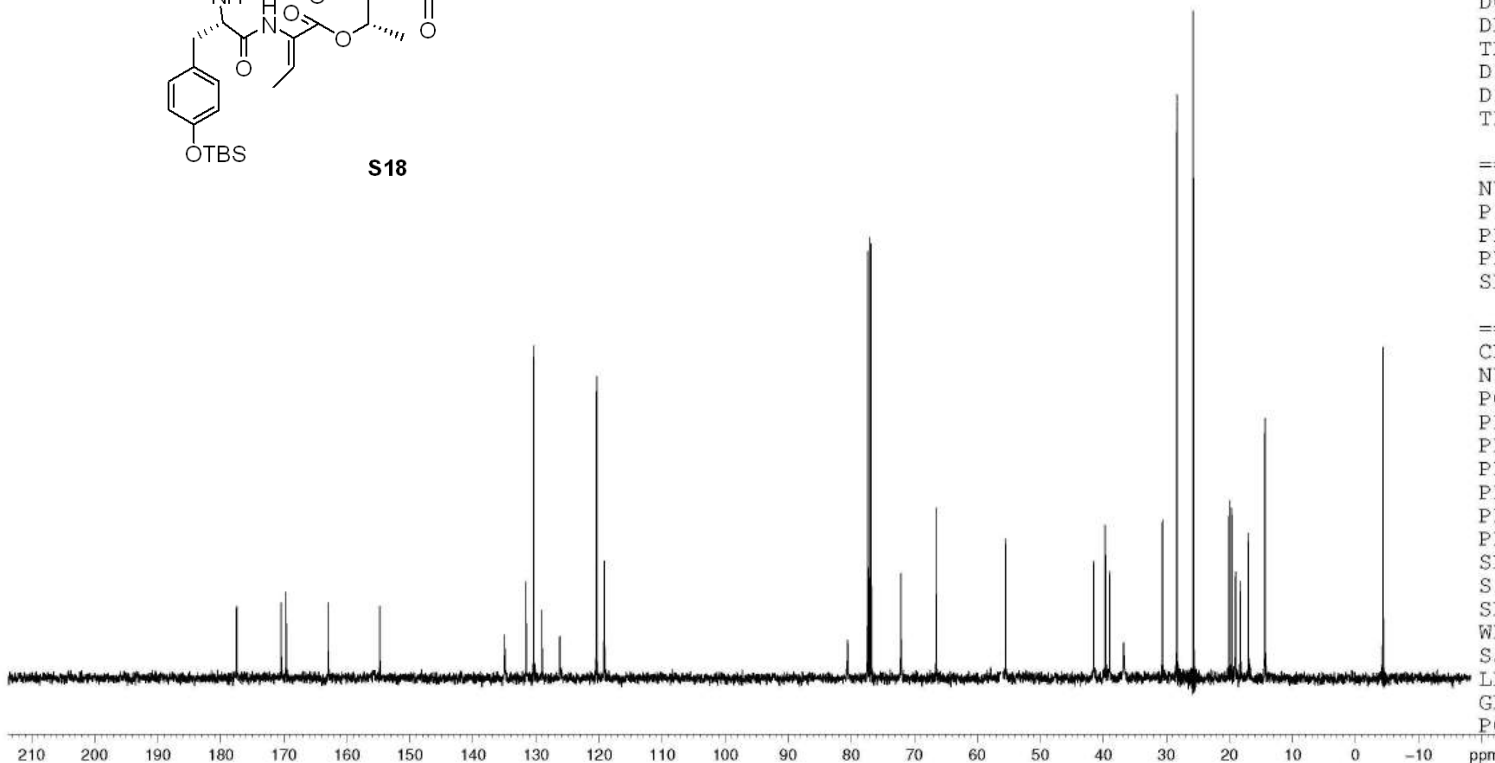


NAME 2021.05.27
EXPNO 38
PROCNO 1
Date_ 20210528
Time 9.58
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 10240
SOLVENT CDC13
NS 200
DS 0
SWH 29761.904 Hz
FIDRES 2.906436 Hz
AQ 0.1720820 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



==== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577777 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



liushouxin XH-SR-DT-4

178.29
172.31
170.77
169.51
163.34
156.79
155.05

135.49
132.29
130.38
129.10
128.08
120.62
118.49

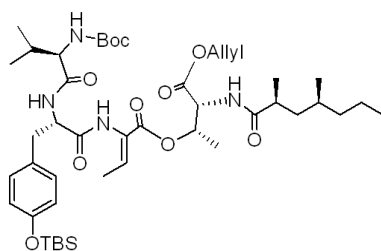
80.80
77.41
77.16
76.91
66.66
62.39
55.26
54.23
41.63
39.97
38.31
35.84
30.72
29.91
28.27
25.80
20.18
19.66
19.25
19.15
18.89
18.33
16.72
14.43
13.42
-4.30



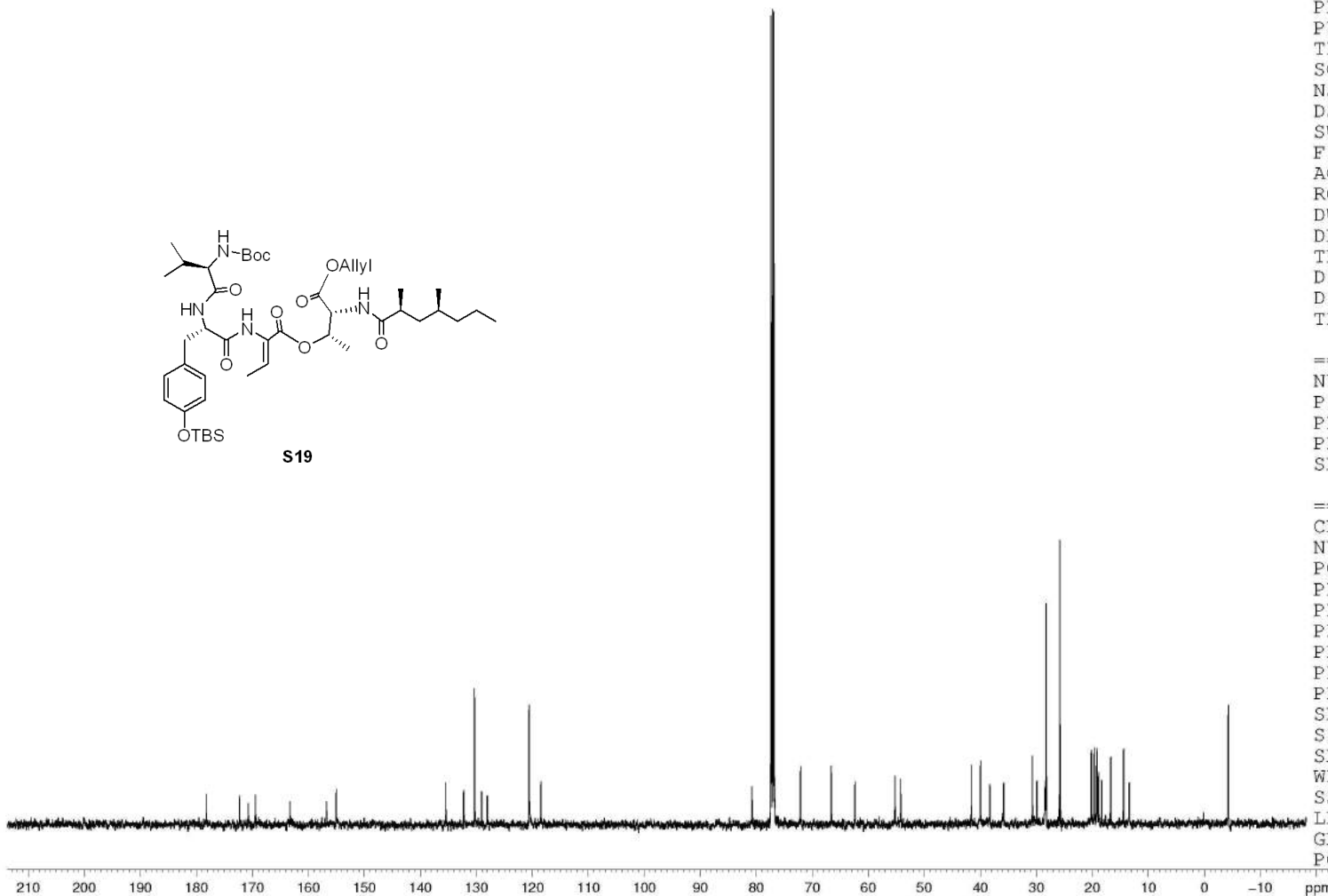
NAME 2021.05.27
EXPNO 40
PROCNO 1
Date_ 20210528
Time 11.04
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 10240
SOLVENT CDC13
NS 775
DS 4
SWH 29761.904 Hz
FIDRES 2.906436 Hz
AQ 0.1720820 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

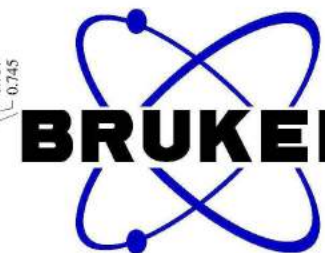
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577736 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



S19



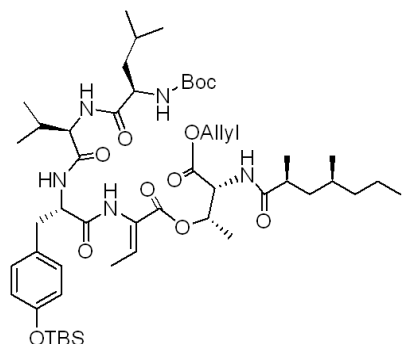
liushouxin-xh-SR-DT-5



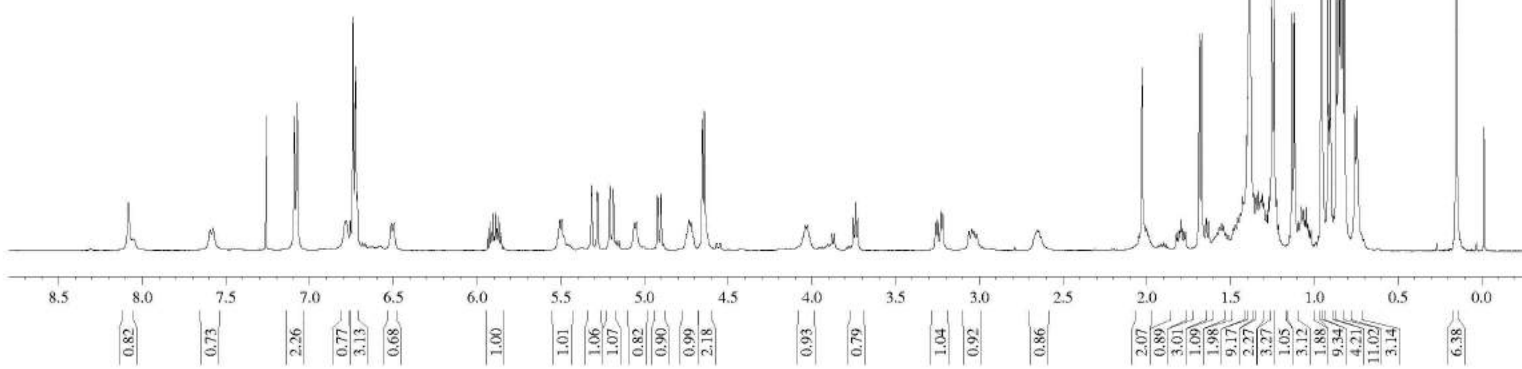
NAME 2021.05.27
 EXPNO 6
 PROCNO 1
 Date_ 20210527
 Time 15.07
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9014.423
 FIDRES 0.137549
 AQ 3.6351135
 RG 64
 DW 55.467
 DE 6.50
 TE 296.8
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300228
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

8.081 7.260 7.089 7.072 6.780 6.755 6.740 6.726 6.723 6.713 6.511 6.496 5.923 5.902 5.888 5.868 5.803 5.494 5.315 5.312 5.280 5.277 5.206 5.185 5.060 5.046 4.921 4.916 4.901 4.897 4.732 4.721 4.652 4.640 4.038 4.025 3.751 3.738 3.724 3.260 3.249 3.231 3.220 2.029 1.797 1.688 1.673 1.554 1.541 1.472 1.457 1.429 1.385 1.362 1.350 1.345 1.334 1.321 1.312 1.305 1.299 1.252 1.239 1.225 1.133 1.119 1.093 1.077 1.066 1.056 1.047 1.039 1.029 0.956 0.920 0.915 0.907 0.902 0.868 0.855 0.843 0.832 0.819 0.757 0.745



S20



liushouxin-xh-SR-DT-5

177.97
173.87
171.67
169.88
169.84
163.16
156.16
154.84

135.21
131.99
130.22
129.48
127.17
120.44
118.81

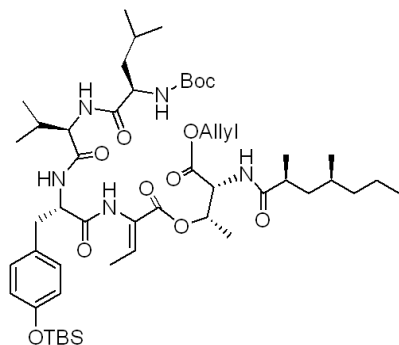
80.69
77.41
77.16
76.90
71.71
66.55
60.94
55.35
55.07
52.99
41.53
40.27
39.92
38.57
35.92
30.70
29.63
28.39
25.78
24.69
22.87
22.07
20.10
19.63
19.17
19.07
18.50
18.29
17.06
14.40
14.00
-4.34



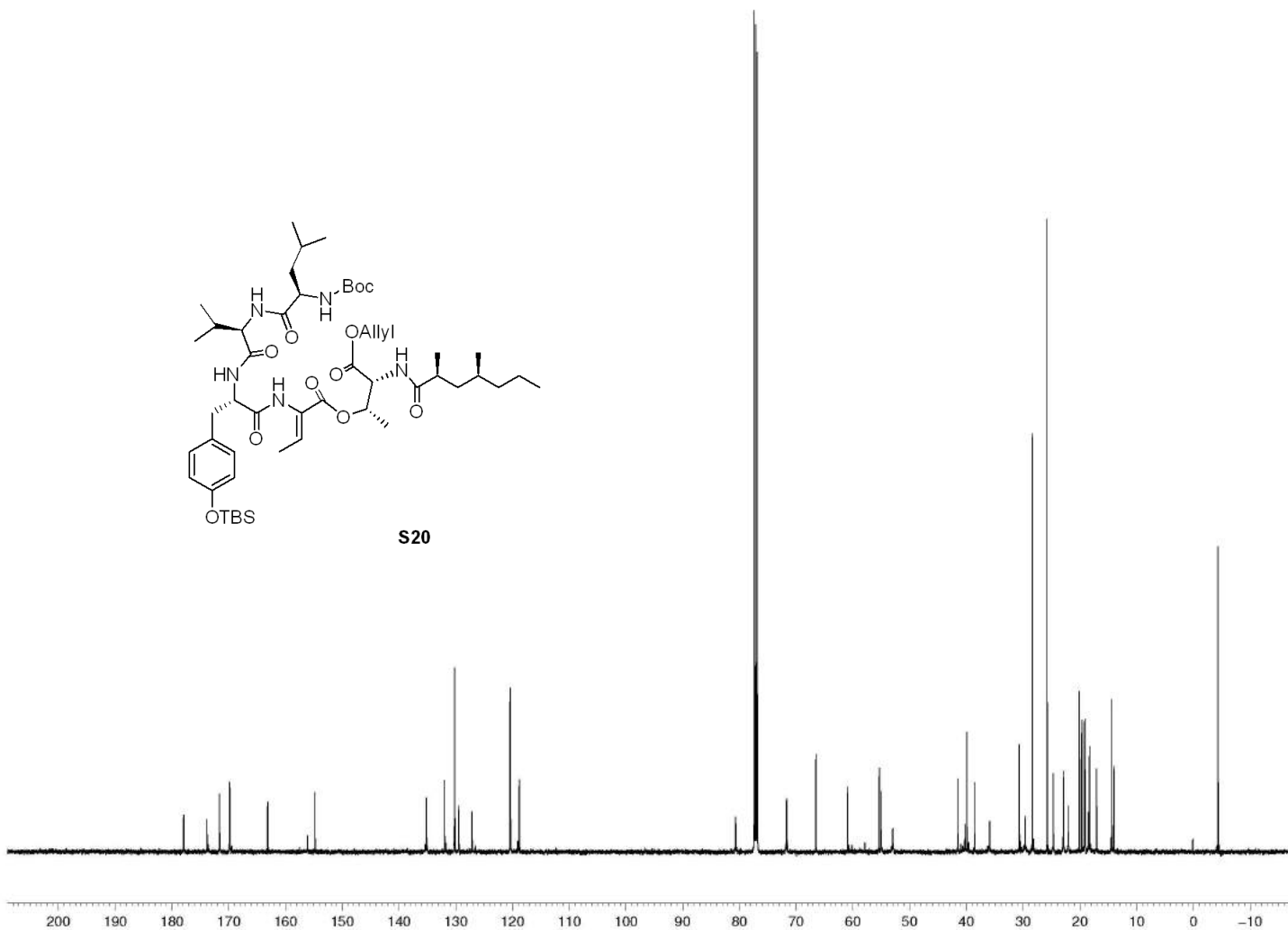
NAME 2021.05.27
EXPNO 17
PROCNO 1
Date_ 20210527
Time 20.03
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 2048
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 299.5 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

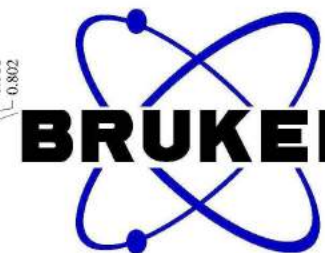
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577756 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



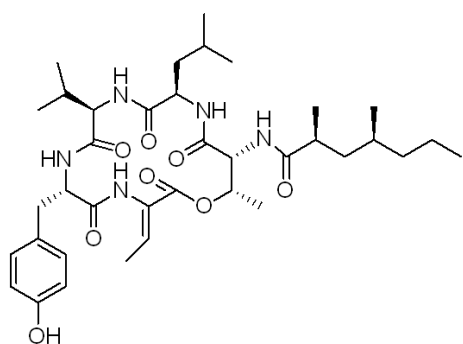
S20



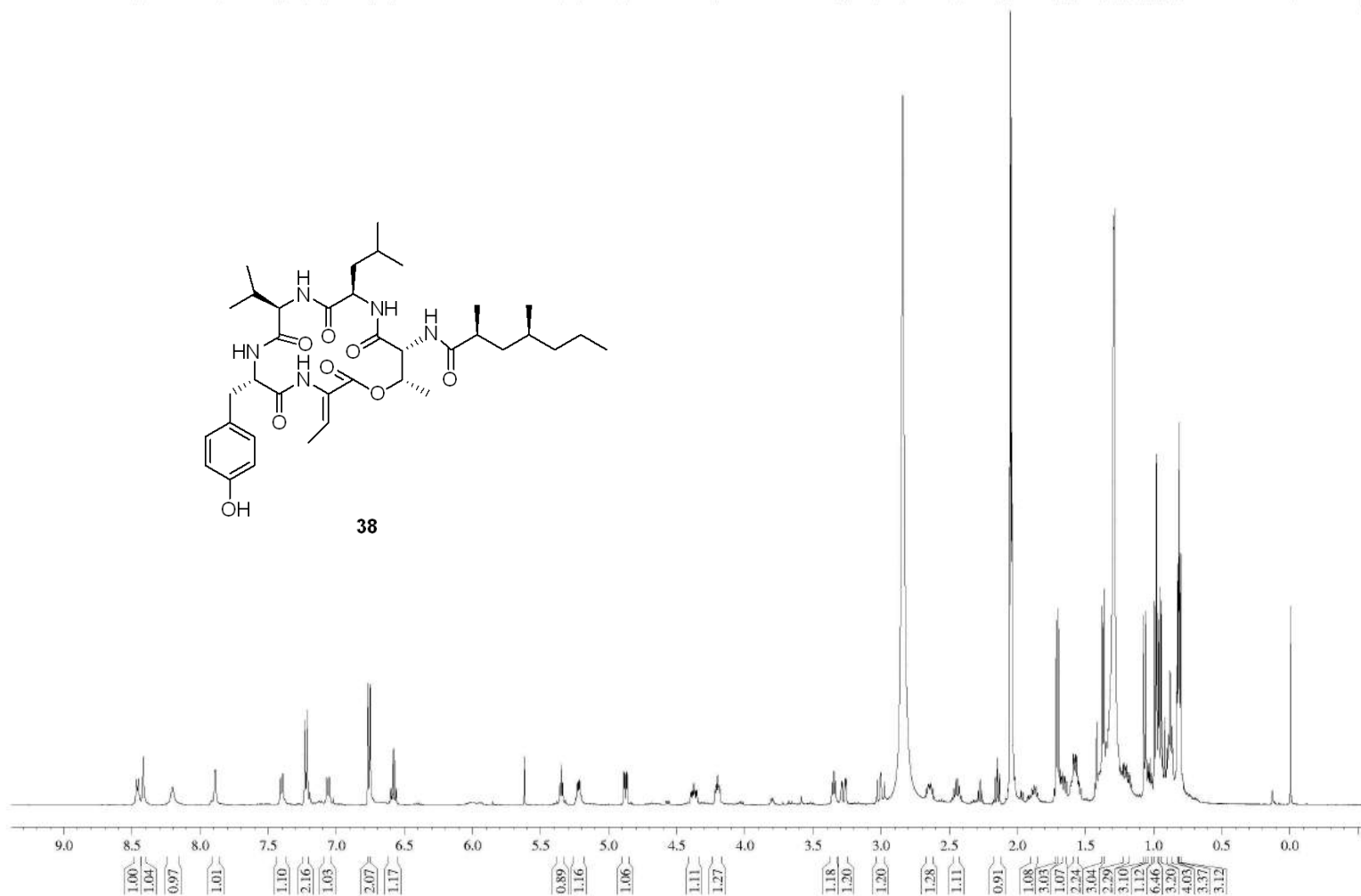
liushouxin-xh-SR-1D-TBAF



8.465 8.451 8.415 7.886 7.410 7.395 7.232 7.215 7.071 7.053 6.769 6.752 6.590 6.576 5.349 5.215 4.890 4.884 4.872 4.866 4.202 3.349 3.337 3.294 3.287 3.265 3.259 3.030 3.005 2.842 2.454 2.441 2.162 2.147 2.132 2.059 2.055 2.050 2.046 2.042 2.042 1.714 1.700 1.685 1.676 1.669 1.658 1.651 1.590 1.580 1.574 1.563 1.552 1.379 1.366 1.290 1.224 1.224 1.213 1.206 1.196 1.186 1.176 1.073 1.060 1.044 1.037 1.028 1.018 1.011 0.994 0.981 0.967 0.958 0.945 0.933 0.919 0.904 0.891 0.879 0.865 0.829 0.824 0.815 0.811 0.802



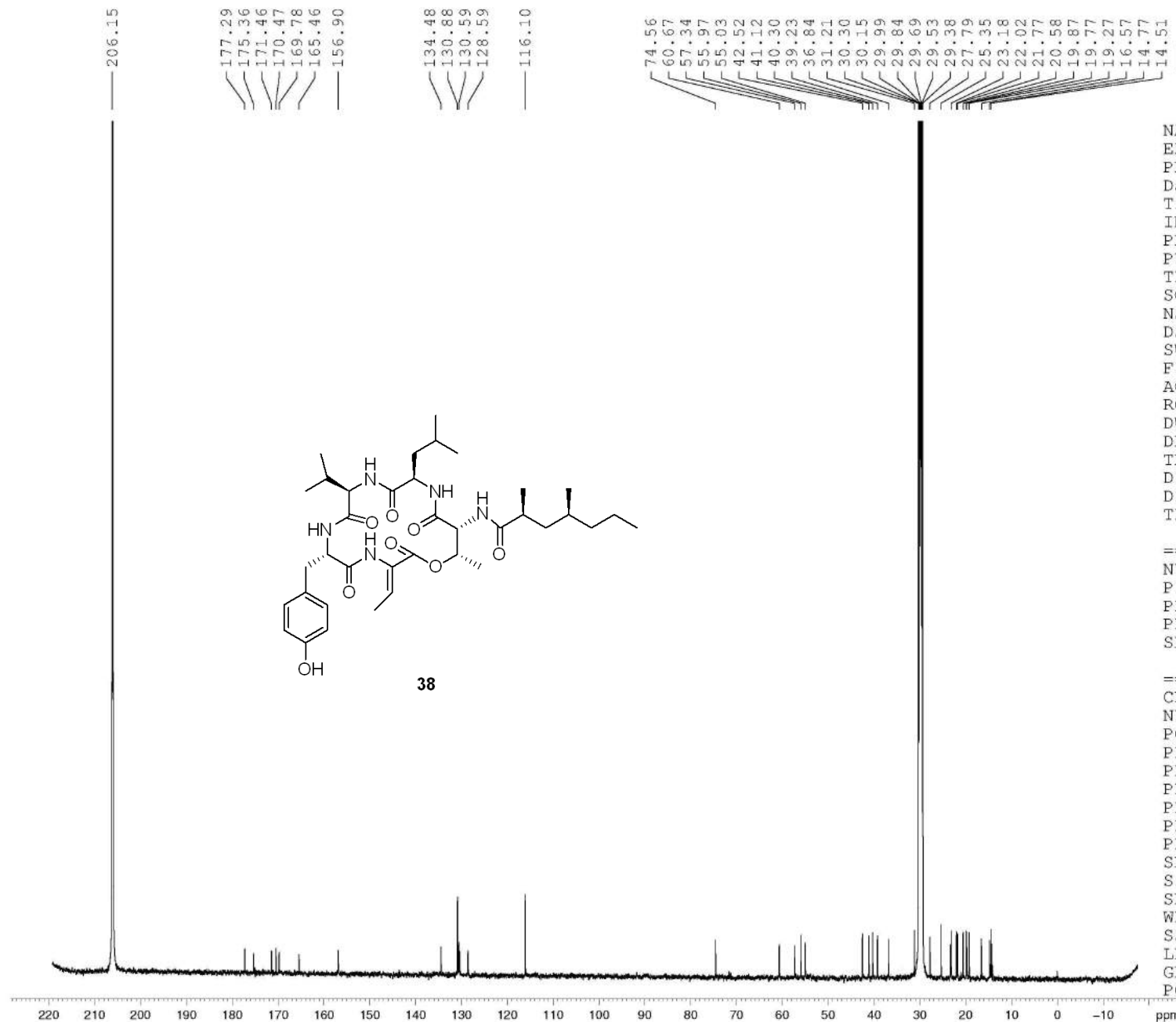
38



NAME 2021.05.31
 EXPNO 2
 PROCNO 1
 Date_ 20210531
 Time 13.11
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 128
 DS 0
 SWH 9014.423
 FIDRES 0.137549
 AQ 3.6351135
 RG 287
 DW 55.467
 DE 6.50
 TE 297.2
 D1 1.00000000
 TD0 1

==== CHANNEL f1 ===
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300145
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

liushouxin XH-SR-1D-TBAF



```

NAME      2021.05.31
EXPNO     19
PROCNO    1
Date_     20210602
Time      8.18
INSTRUM   spect
PROBHD    5 mm PADUL 13C
PULPROG   zgpg30
TD        32768
SOLVENT   Acetone
NS        15360
DS        0
SWH       29761.904 Hz
FIDRES    0.908261 Hz
AQ        0.5505524 sec
RG        2050
DW        16.800 usec
DE        6.50 usec
TE        300.2 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        12.00 usec
PL1       4.50 dB
PL1W      33.60015869 W
SFO1      125.7703643 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.00 dB
PL12      17.46 dB
PL13      17.46 dB
PL2W      16.79986763 W
PL12W     0.47786582 W
PL13W     0.47786582 W
SFO2      500.1320005 MHz
SI        32768
SF        125.7576749 MHz
WDW       EM
SSB       0
LB        3.00 Hz
GB        0
PC        1.40
    
```

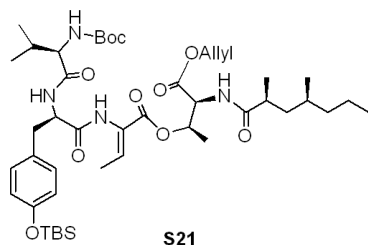

liushouxin XH-SR-4D-4

178.10
171.33
169.79
169.46
163.13
156.70
155.09
136.00
132.07
130.36
128.73
126.89
120.58
118.73

81.32
77.42
77.16
76.91
72.30
66.45
61.58
55.22
53.82
41.72
39.98
38.25
35.98
30.51
29.98
28.20
25.74
20.10
19.67
19.62
19.34
18.26
17.73
16.60
14.48
14.10
-4.33

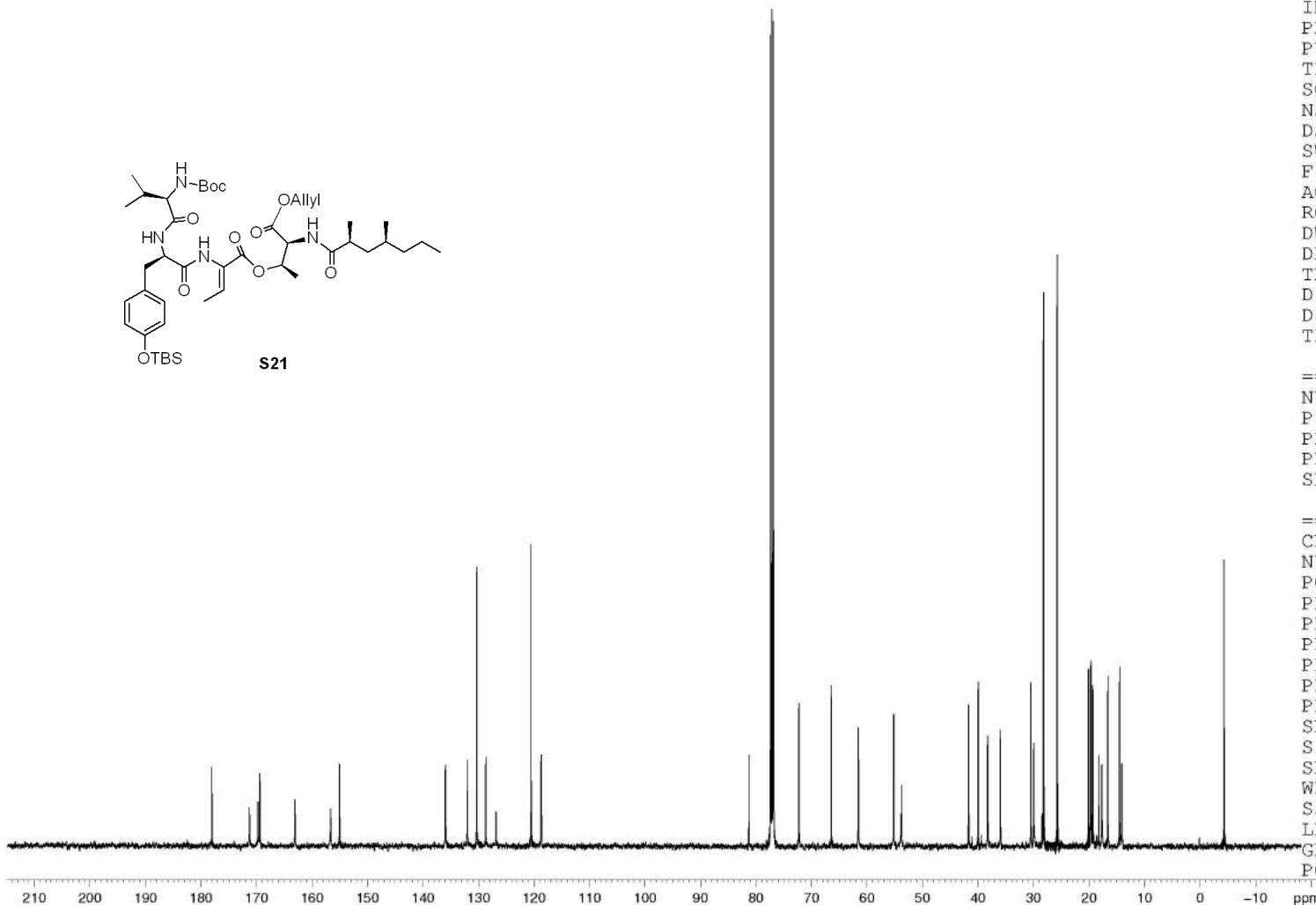


NAME 2021.05.27
EXPNO 42
PROCNO 1
Date_ 20210528
Time 13.00
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 10240
SOLVENT CDC13
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 2.906436 Hz
AQ 0.1720820 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577747 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



liushouxin-xh-SR-4D-5D

178.24
174.53
170.80
170.75
169.37
163.42
156.75
154.50
134.82
132.28
130.05
129.75
127.78
120.24
118.45

81.50
77.41
77.16
76.90
72.16
66.51
60.68
55.13
54.48
53.96
41.74
39.98
38.08
35.69
30.48
29.28
28.37
25.77
24.89
22.95
21.69
20.09
19.65
19.61
19.19
18.30
17.54
16.77
14.45
13.64
-4.37



Current Data Parameters
NAME Desktop
EXPNO 2
PROCNO 1

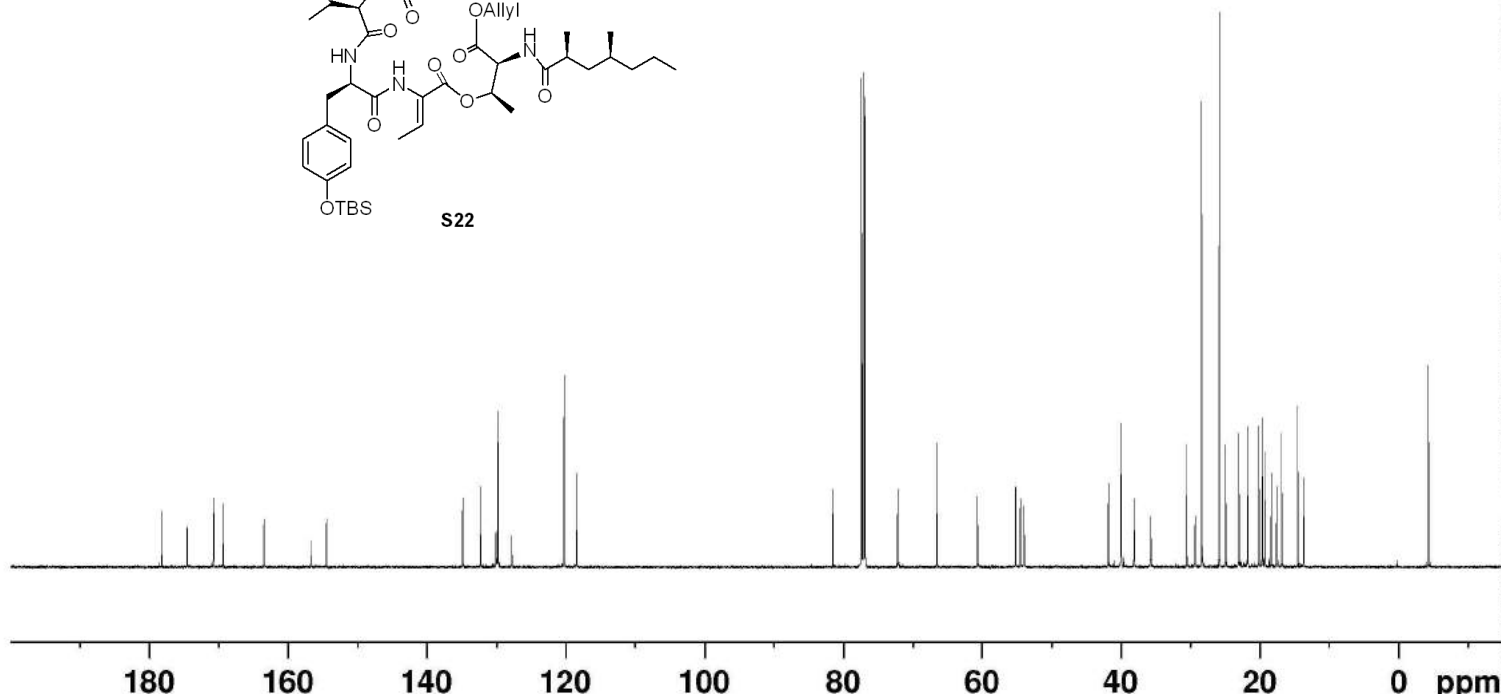
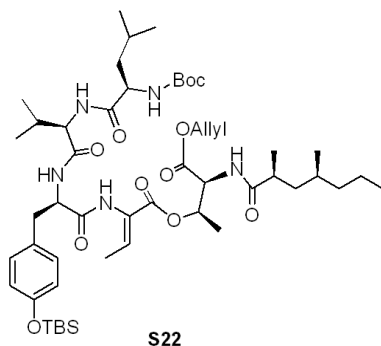
F2 - Acquisition Parameters

Date_ 20210627
Time 12.12
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4864
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577776 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



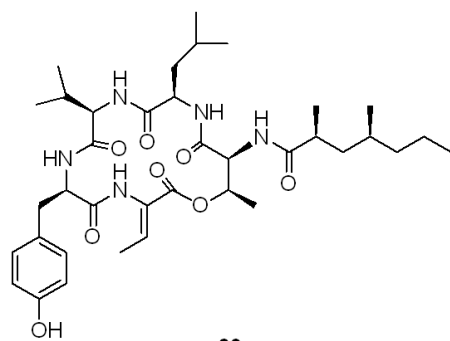
liushouxin-xh-SR-4D-5D-TBAF



8.16
8.11
8.07
7.79
7.20
7.18
6.77
6.75
6.64
6.62
6.61
5.23
4.77
4.48
4.05
3.86
3.35
3.32
3.32
3.03
3.00
2.97
2.86
2.72
2.70
2.05
2.05
2.03
2.01
1.76
1.69
1.67
1.66
1.61
1.60
1.49
1.42
1.38
1.37
1.35
1.33
1.29
1.25
1.24
1.22
1.21
1.19
1.16
1.15
1.03
0.96
0.94
0.93
0.92
0.90
0.88
0.87
0.85
0.84
0.82
0.73
0.72
0.68
0.67
-0.00

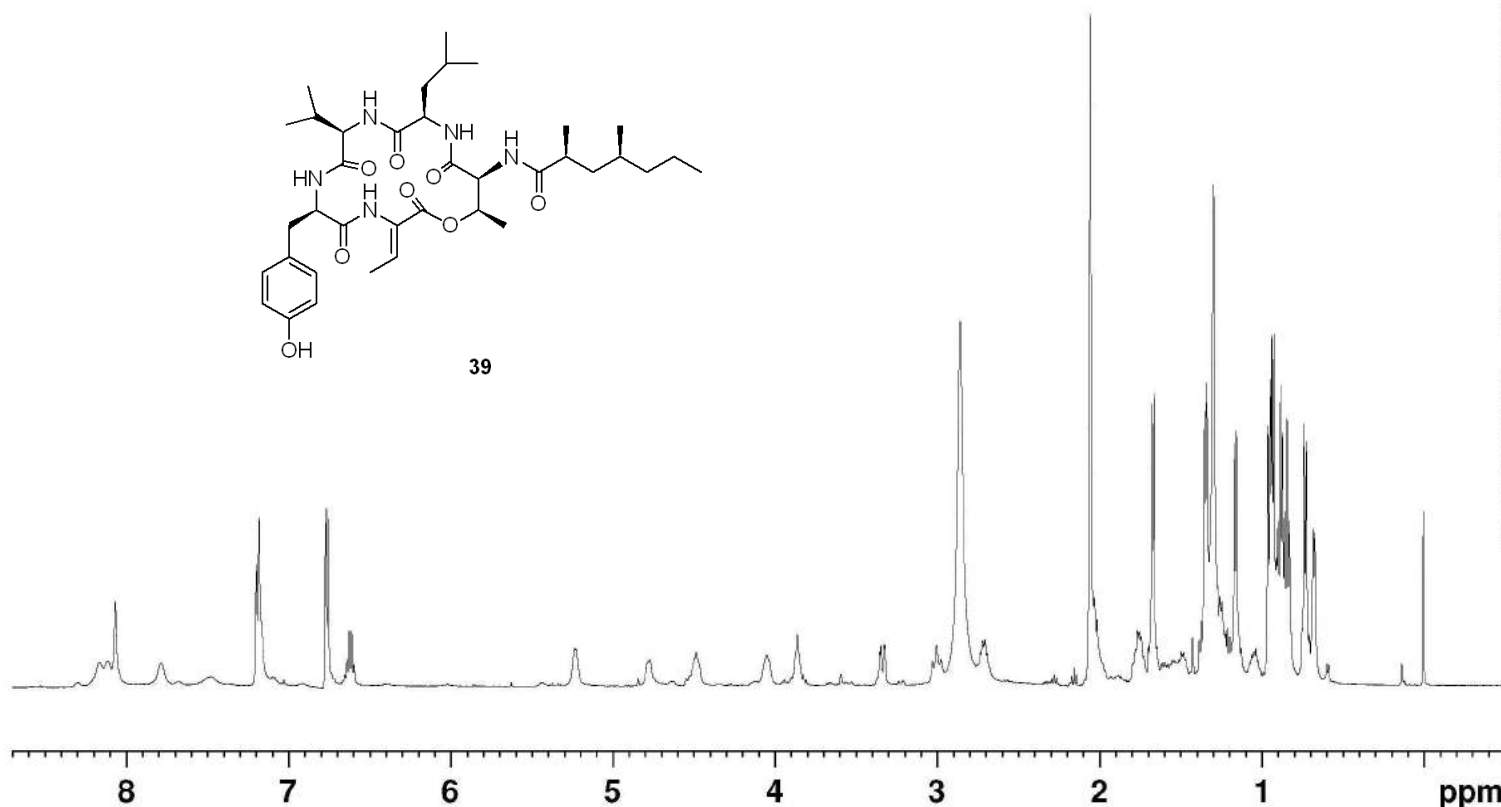
Current Data Parameters
NAME Desktop
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210625
Time 17.40
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 64
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 256
DW 62.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1



===== CHANNEL f1 =====
NUC1 1H
P1 13.50 usec
PL1 2.00 dB
PL1W 16.79986763 W
SFO1 500.1335009 MHz

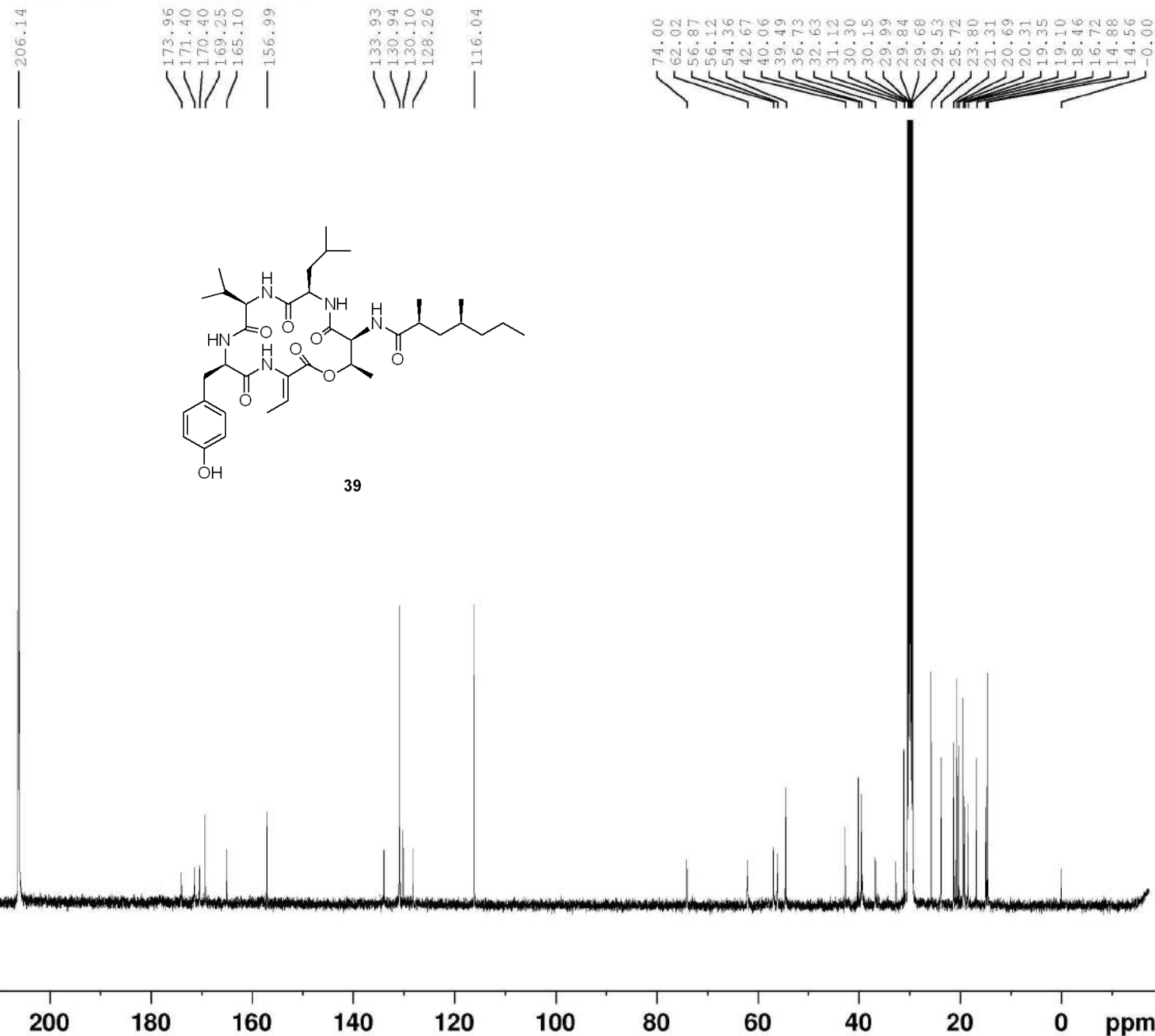
F2 - Processing parameters
SI 32768
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



1.30
1.35
0.80
0.68
2.97
2.34
1.03
0.96
0.77
1.14
1.00
1.16
1.01
1.39
1.69
2.00
1.82
2.13
2.87
1.19
1.39
2.93
2.12
2.80
1.19
3.01
3.23
4.10
3.22
2.91
2.91

S149

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206.14
173.96
171.40
170.40
169.25
165.10
156.99
133.93
130.94
130.10
128.26
116.04
74.00
62.02
56.87
56.12
54.36
42.67
40.06
39.49
36.73
32.63
31.12
30.30
30.15
29.99
29.84
29.68
29.53
25.72
23.80
21.31
20.69
20.31
19.35
19.10
18.46
16.72
14.88
14.56
-0.00

Current Data Parameters
NAME Desktop
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

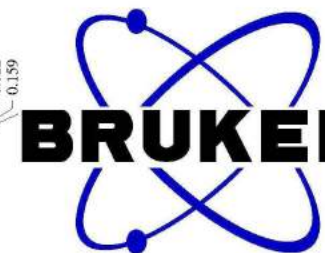
Date_ 20210625
Time 17.58
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT Acetone
NS 15360
DS 0
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505024 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7576759 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

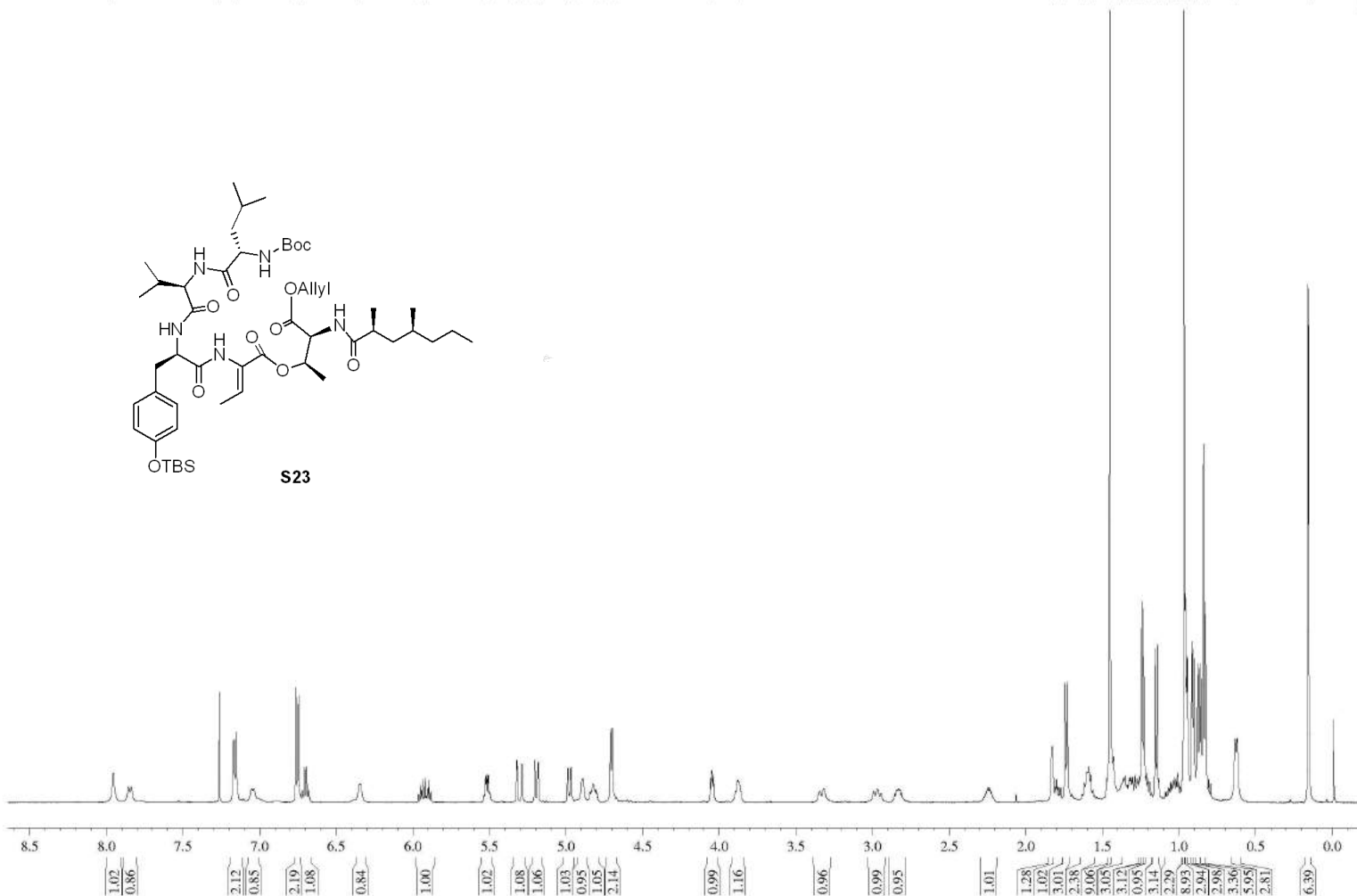
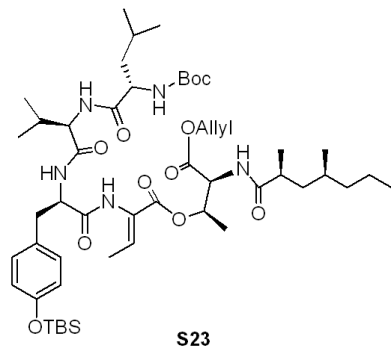
liushouxin-xh-SR-4D-5



NAME 2021.05.27
 EXPNO 8
 PROCNO 1
 Date_ 20210527
 Time 15.19
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9014.423
 FIDRES 0.137549
 AQ 3.6351135
 RG 64
 DW 55.467
 DE 6.50
 TE 296.8
 D1 1.00000000
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50
 PL1 2.00
 PL1W 16.79986763
 SFO1 500.1340010
 SI 32768
 SF 500.1300227
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00

7.952 7.260 7.168 7.152 6.760 6.743 6.705 6.690 6.338 5.950 5.929 5.916 5.895 5.826 5.521 5.513 5.509 5.323 5.320 5.289 5.286 5.205 5.202 5.184 5.181 4.992 4.987 4.972 4.968 4.891 4.824 4.710 4.710 4.699 4.059 4.051 4.048 4.040 3.880 1.829 1.800 1.744 1.729 1.619 1.603 1.589 1.577 1.577 1.452 1.363 1.352 1.322 1.317 1.308 1.303 1.289 1.289 1.275 1.264 1.259 1.244 1.231 1.213 1.203 1.192 1.156 1.142 1.055 1.045 1.038 1.028 1.019 1.010 1.001 0.967 0.961 0.953 0.947 0.917 0.905 0.879 0.866 0.855 0.841 0.828 0.622 0.622 0.159



liushouxin XH-SR-4D-5

178.21
174.66
170.74
170.59
169.42
163.42
156.51
154.58

134.03
132.32
130.60
130.17
127.82
120.34
118.53

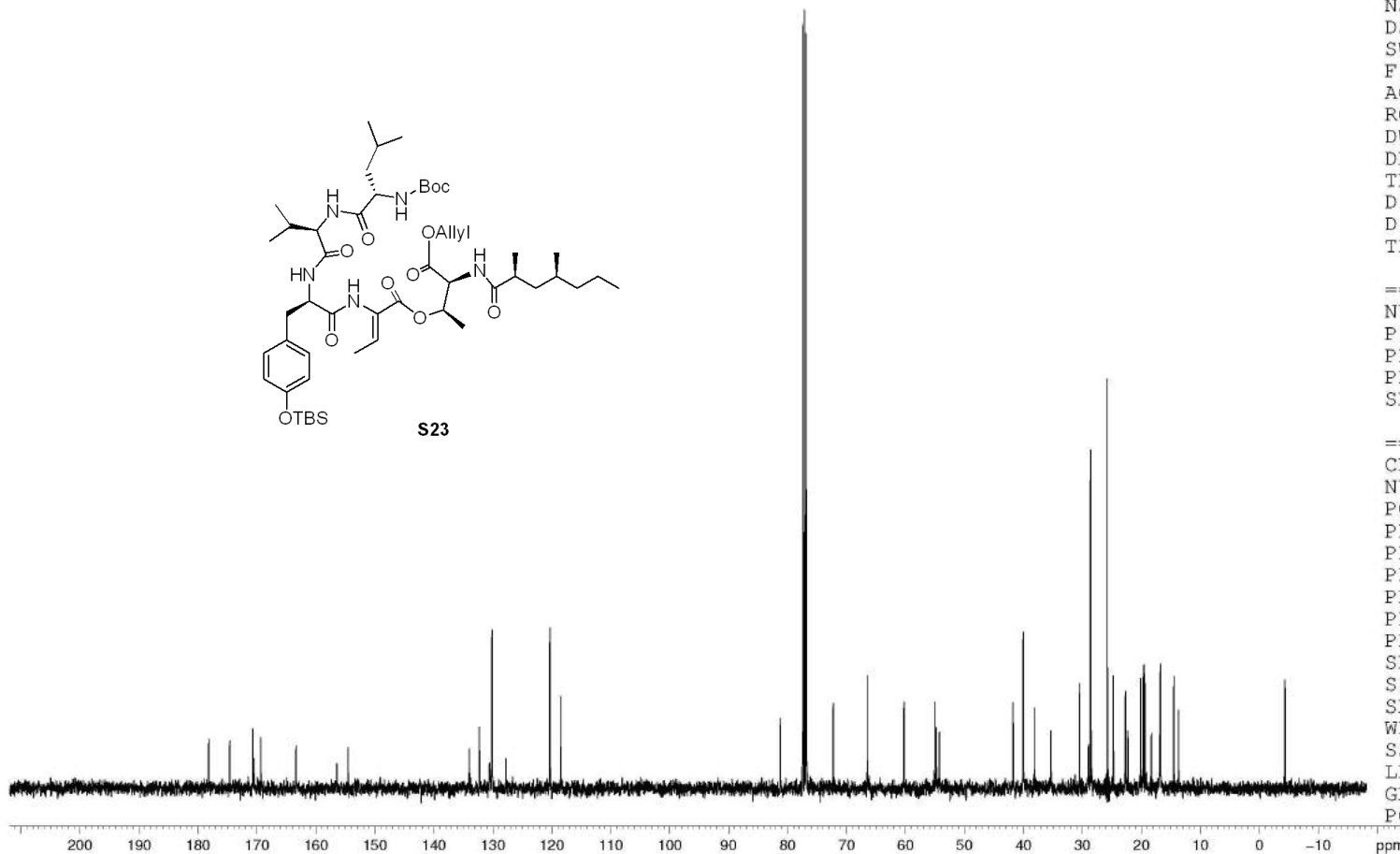
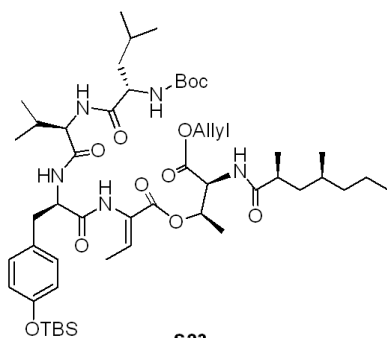
81.25
77.41
77.16
76.90
72.29
66.52
60.28
55.09
54.85
54.28
41.78
40.05
38.16
35.37
30.48
28.96
28.62
25.79
24.78
22.71
22.31
20.13
19.69
19.59
19.43
18.31
16.90
16.78
14.48
13.71
-4.34



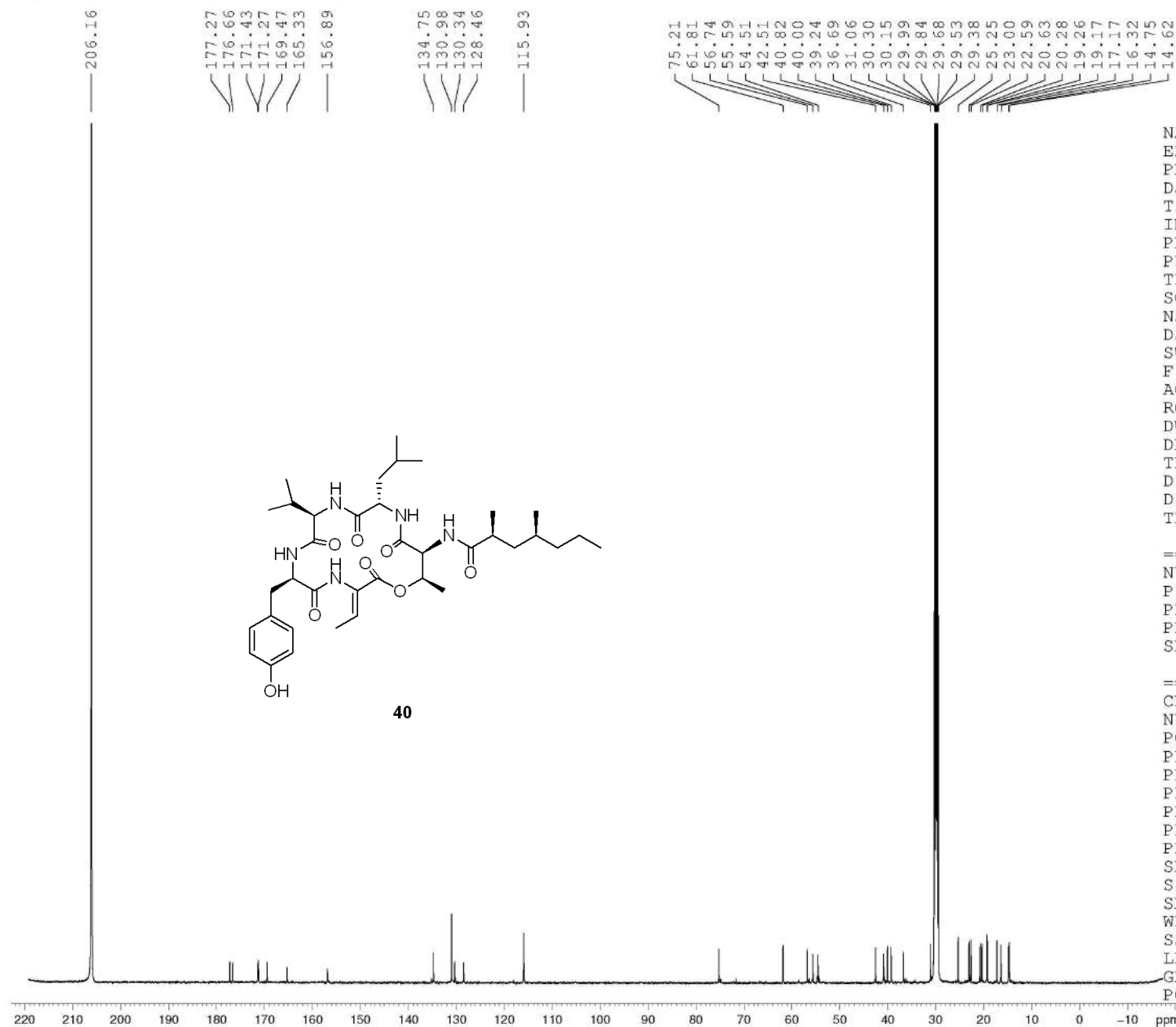
NAME 2021.05.27
EXPNO 39
PROCNO 1
Date_ 20210528
Time 10.49
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 10240
SOLVENT CDC13
NS 294
DS 4
SWH 29761.904 Hz
FIDRES 2.906436 Hz
AQ 0.1720820 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.46 dB
PL13 17.46 dB
PL2W 16.79986763 W
PL12W 0.47786582 W
PL13W 0.47786582 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577749 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



liushouxin XH-SR-4D-TBAF



```

NAME      2021.05.31
EXPNO     17
PROCNO    1
Date_     20210601
Time      6.42
INSTRUM   spect
PROBHD    5 mm PADUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   Acetone
NS         15360
DS         0
SWH       29761.904 Hz
FIDRES    0.908261 Hz
AQ        0.5505524 sec
RG         2050
DW         16.800 usec
DE         6.50 usec
TE         300.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

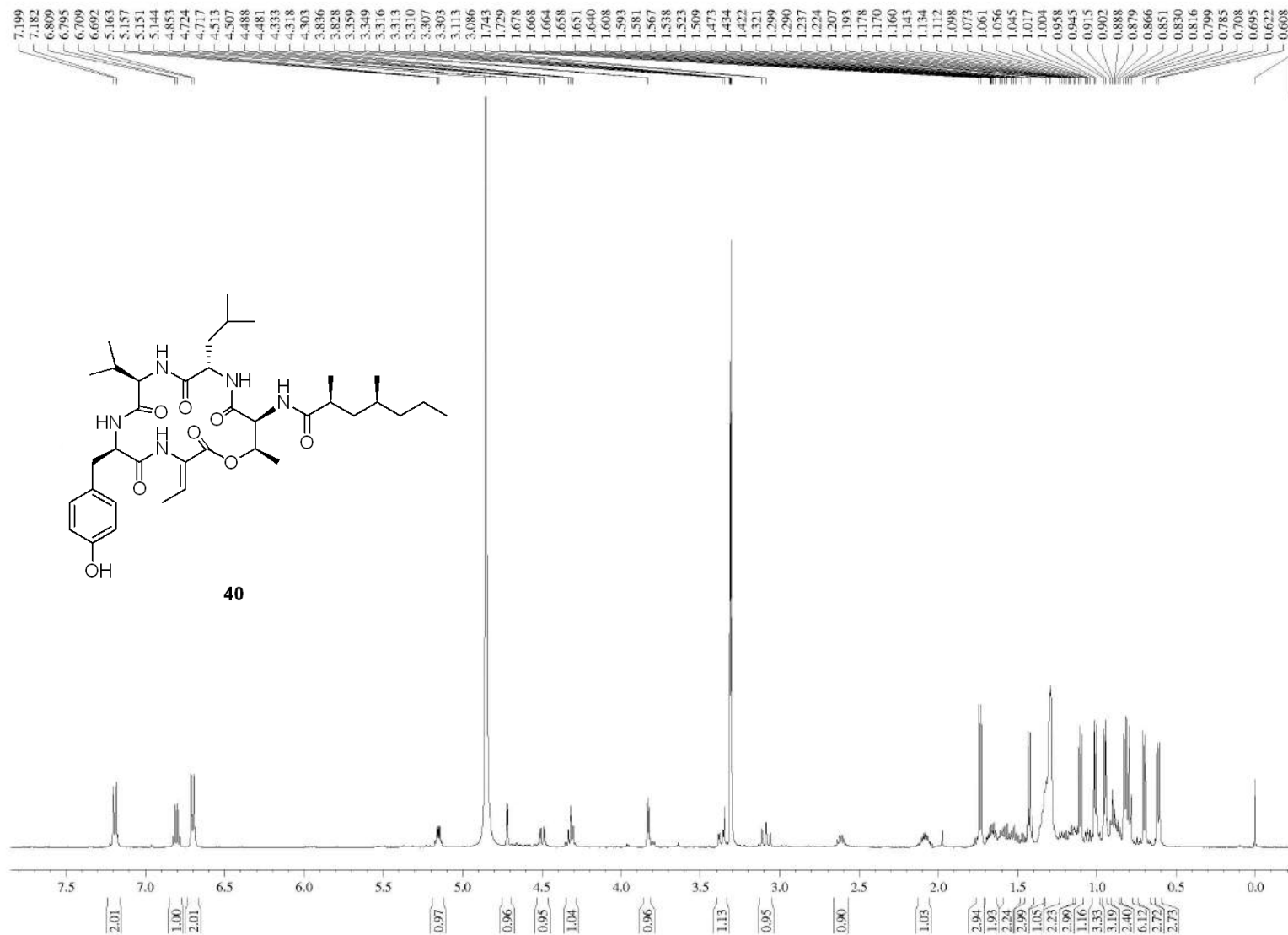
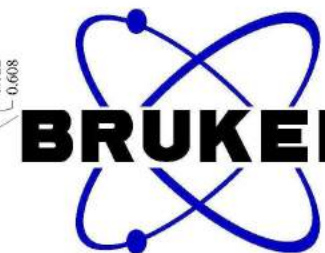
```

===== CHANNEL f1 =====
NUC1      13C
P1         12.00 usec
PL1        4.50 dB
PL1W      33.60015869 W
SFO1      125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        2.00 dB
PL12       17.46 dB
PL13       17.46 dB
PL2W      16.79986763 W
PL12W     0.47786582 W
PL13W     0.47786582 W
SFO2      500.1320005 MHz
SI         32768
SF        125.7576758 MHz
WDW        EM
SSB         0
LB         3.00 Hz
GB         0
PC         1.40
  
```

liushouxin-xh-SR-4D-TBAF-MeOD



```

NAME          2021.06.2
EXPNO         13
PROCNO        1
Date_         20210602
Time          14.35
INSTRUM       spect
PROBHD        5 mm PADUL 13C
PULPROG       zg30
TD            65536
SOLVENT       MeOD
NS            128
DS            0
SWH           9014.423
FIDRES        0.137549
AQ            3.6351135
RG            322
DW            55.467
DE            6.50
TE            297.6
D1            1.00000000
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            13.50
PL1           2.00
PL1W         16.79986763
SFO1         500.1340010
SI            32768
SF           500.1300160
WDW           EM
SSB           0
LB            0.30
GB            0
PC            1.00
  
```


8. References

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- [6] S. Kishimoto, Y. Tsunematsu, S. Nishimura, Y. Hayashi, A. Hattori and H. Kakeya, Tumescenamides C, an antimicrobial cyclic lipodepsipeptide from *Streptomyces* sp. *Tetrahedron.*, 2012, **68**, 5572-5578.
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