Electronic Supplementary Material (ESI) for Chemical Science.

# Supplementary Information for

## An Economycal Approach for Peptide Synthesis *via* Regioselective C–N Bond Cleavage of Lactams

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Materials and Methods
References
HPLC data
NMR data

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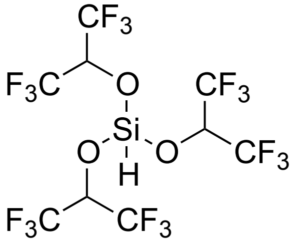
# 1. General

NMR spectra were recorded on a JEOL 400SS spectrometer operating at 400 MHz and 100 MHz, for $^{1}$H and $^{13}$C acquisitions, respectively. Chemical shifts are reported in ppm with a solvent resonance as an internal standard ($^{1}$H NMR; tetramethylsilane, $CD_3OD$, $D_2O$, and $CDCl_3$ as internal standard, indicating 0, 3.31, 4.79, and 7.26 ppm, respectively, $^{13}$C NMR; $(CD_3)_2SO$, $CD_3OD$, and $CDCl_3$ as internal standard, indicating 39.52, 49.00, and 77.00 ppm, $^{29}$Si NMR; tetramethylsilane as internal standard, indicating 0 ppm). Data is reported as follows: s = singlet, br = broad, d = doublet, t = triplet, q = quartet, quin = quintet, m = multiplet; coupling constants in Hz; integration. FT-IR spectra were recorded with a Bruker ALPHA (Eco-ATR) spectrometer. Specific rotation was measured with an ATAGO AP-300 digital polarimeter. MS spectra were recorded with a JEOL JMS-T100CS "AccuTOF CS" mass spectrometer with electrospray ionization time-of flight (ESI-TOF) for HRMS measurements. E.r. values were determined by high performance liquid chromatography (HPLC) using a Shimadzu CBM 20A HPLC equipped with Shimadzu SPD-M20A photodiode array detector and DAICEL IA-3, IE-3, and IG-3 columns (4.6 mm x 25 cm). Removal of amino acid esters were performed by using ULVAC KIKO GLD-137CC small oil rotary vacuum pump and ULVAC KIKO VPC-051A oil diffusion pump. TLC analysis was performed on commercial glass plates bearing a 0.25 mm layer of Merck KGaA TLC silica gel 60 F254. Silica gel chromatography was carried out Merck KGaA silica gel 60 (230–400 mesh ASTM). Dry solvents, DCM and $CHCl_3$, were purchased from FUJIFILM Wako Pure Chemical Co. These solvents were used without further treatment. Amino acids and their derivatives were purchased from Accela ChemBio Inc., Acros Organics, Advanced ChemBlocks Inc., AmBeed, Inc., Apollo Scientific Ltd., BLD Pharmatech Ltd., Cayman Chemical Company, Chem-Impex Int'l Inc. Chemspace LLC., Combi-Blocks, Inc., Sigma-Aldrich Co. LLC., Synthonic, Inc., Tokyo Chemical Industry Co., Ltd., Toronto Research Chemicals, Inc., and Watanabe Chemical Ind., Ltd. Trichlorosilane and 1,1,1,3,3,3-hexafluoro-2-propanol were purchased from Tokyo Chemical Industry Co., Ltd. Amberlyst$^{TM}$ A21 was purchased from Organo corporation.

## 2. General procedure for the neutralization of amino acid HCl salts

A flame-dried 500 mL round-bottom flask with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with amino acid *tert*-butyl ester HCl salt (5.0 g) and Amberlyst A21 (10 g, 200 wt%), and DCM or $CHCl_3$ (150 ~ 400 mL: choice of the *solvents and their volumes are depended on the solubility of the amino acid ester HCl salts*). After the resulting mixture was stirred under nitrogen atmosphere for 4 h ~ overnight at 0 °C to ambient temperature, the mixture was filtered on celite with DCM, then the filtrate was concentrated in *vacuo* (70 Torr) with the aid of a rotary evaporator and water bath without heating. The residue was transferred into 50 mL vials by a pipette, and further concentrated in *vacuo* (70 Torr) at ambient temperature for overnight. 3Å MS was added to the free amine, then it was stored in a freezer (See, E. L. Baker, M. M. Yamano, Y. Zhou, S. M. Anthony and N. K Garg, *Nat. Commun.* 2016, **7**, 11554).

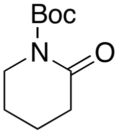
## 3. Preparation of tris[(1,1,1,3,3,3-hexafluoropropan-2-yl)oxy]silane



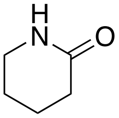
**Tris[(1,1,1,3,3,3-hexafluoropropan-2-yl)oxy]silane (CAS No. 2473688-54-7)[1]:** Tris[(1,1,1,3,3,3-hexafluoropropan-2-yl)oxy]silane was prepared from commercially avairable trichlorosilane (Tokyo Chemical Industry Co., Ltd., T0398) and 1,1,1,3,3,3-hexafluoro-2-propanol (Tokyo Chemical Industry Co., Ltd., H0424) according to the literature method.[1]

$^{1}$H NMR (400 MHz, $CD_2Cl_2$) δ 4.79 (s, 1H, Si*H*), 4.75–4.60 (m, 3H, OC*H*($CF_3$)$_2$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 120.6 (q, 6C, *J* = 280.6 Hz), 70.3 (quin, 3C, *J* = 35.0 Hz); $^{29}$Si NMR (80 MHz, $CD_2Cl_2$) δ -63.1. IR (thin film, cm$^{-1}$) 2253, 1377, 1301, 1231, 1198, 1151, 1104, 900, 859, 732, 686

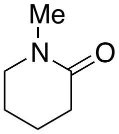
## 4. Properties of lactams 1a–k (Table 1)



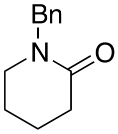
***N*-Boc-2-piperidone (1a, CAS No. 85908-96-9):** *N*-Boc-2-piperidone was purchased from Tokyo Chemical Industry Co., Ltd. (B3788) and used as delivered.



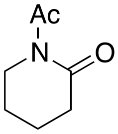
**2-Piperidone (1b, CAS No. 675-20-7):** 2-Piperidone was purchased from Tokyo Chemical Industry Co., Ltd. (P0455) and used as delivered.



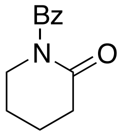
***N*-Me-2-Piperidone (1c, CAS No. 931-20-4):** *N*-Bn-2-piperidone was purchased from Sigma-Aldrich Co. LLC. (M73788) and used as delivered.



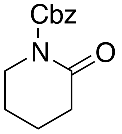
***N*-Bn-2-Piperidone (1d, CAS No. 4783-65-7):** *N*-Bn-2-piperidone was purchased from Sigma-Aldrich Co. LLC. (496898) and used as delivered.



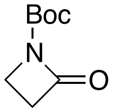
***N*-Ac-2-piperidone (1e, CAS No. 3326-13-4):** *N*-Ac-2-piperidone was purchased from BLD Pharmatech Ltd. (BD142931) and used as delivered.



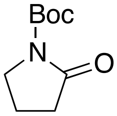
**_N_-Bz-2-piperidone (1f, CAS No. 4252-56-6):** _N_-Bz-2-piperidone was purchased from Enamine Ltd. (EN300-207021) and used as delivered.



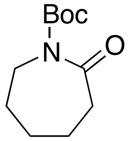
**_N_-Cbz-2-piperidone (1g, CAS No. 106412-35-5):** _N_-Cbz-2-piperidone was purchased from Sigma-Aldrich Co. LLC. (737534) and used as delivered.



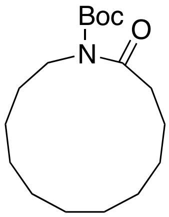
**_N_-Boc-2-azetidinone (1h, CAS No. 1140510-99-1):** _N_-Boc-2-azetidinone was purchased from Apollo Scientific Ltd. (OR925839) and used as delivered.



**_N_-Boc-2-pyrrolidinone (1i, CAS No. 85909-08-6):** _N_-Boc-2-pyrrolidinone was purchased from Combi-Blocks, Inc. (SS-7905) and used as delivered.



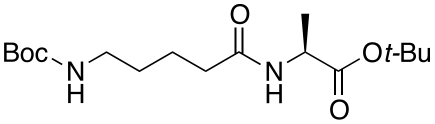
**_N_-Boc-ε-caprolactam (1j, CAS No. 106412-36-6):** _N_-Boc-ε-caprolactam was purchased from Sigma-Aldrich Co. LLC. (719706) and used as delivered.



***N*-Boc-λ-caprolactam (1k, CAS No. 370861-81-7)[2]:** *N*-Boc-λ-caprolactam was prepared from commercially avairable *ω*-laurinlactam (Tokyo Chemical Industry Co., Ltd., L0092), Boc$_2$O (Tokyo Chemical Industry Co., Ltd., D1547), DMAP (FUJIFILM Wako Pure Chemical Co., 042-19212), and triethylamine (FUJIFILM Wako Pure Chemical Co., 202-02646) according to the literature method.[2]

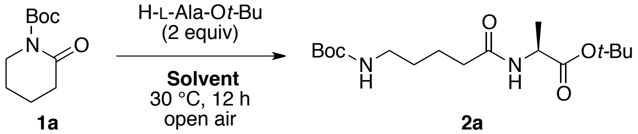
$R_f$ = 0.33 (10% AcOEt in hexane). $^1$H NMR (400 MHz, CDCl$_3$) δ 3.90–3.75 (m, 2H, $C\underline{H}_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2$), 3.00–3.85 (m, 2H, $CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2C\underline{H}_2$), 1.80–1.65 (m, 2H, $CH_2C\underline{H}_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2$), 1.65–1.50 (m, 2H, $CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2C\underline{H}_2CH_2$), 1.53 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCON$), 1.40–1.20 (m, 14H, $CH_2CH_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2CH_2CH_2$). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 177.0, 153.7, 82.6, 43.9, 37.9, 28.0 (3C), 27.6, 26.9, 26.5, 25.7, 25.2, 25.0 (2C), 24.9, 24.1. IR (thin film, cm$^{-1}$) 2930, 2862, 21728, 1689, 1459, 1367, 1351, 1337, 1282, 1257, 1220.

## 5. Properties of dipeptides 2a–k (Table 1)

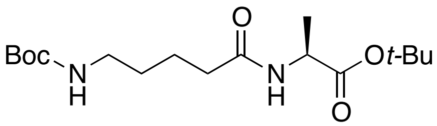


**Boc-δ-HoGly-L-Ala-O*t*-Bu (2a, entry 1):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-2-piperidone (**1a**, 99.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **2a** (171.4 mg, >99% yield) as a colorless oil with >99:1 er.

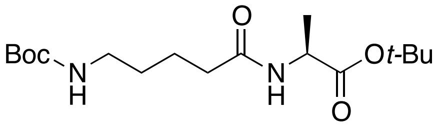
$R_f$ = 0.56 (80% AcOEt in hexane). $[\alpha]_D^{22} = -34.8$ (*c* 1.18, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.20 (br d, *J* = 7.1 Hz, 1H, N*H*), 4.74 (br t, *J* = 6.0 Hz, 1H, $(C\underline{H}_3)_3COCON\underline{H}$), 4.46 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 3.13 (q, *J* = 6.0 Hz, 2H, $C\underline{H}_2CH_2CH_2CH_2$), 2.23 (t, *J* = 7.3 Hz, 2H, $CH_2CH_2CH_2C\underline{H}_2$), 1.75–1.40 (m, 4H, $CH_2C\underline{H}_2C\underline{H}_2CH_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.36 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.4, 172.1, 156.0, 81.9, 79.1, 48.5, 39.9, 35.8, 29.4, 28.4 (3C), 27.9 (3C), 22.6, 18.7. IR (thin film, cm$^{-1}$) 3316, 2977, 2933, 1713, 1692, 1651, 1523, 1454, 1366, 1246, 1147. HRMS (ESI) calculated for $C_{17}H_{32}N_2O_5Na$ $[M+Na]^+$ *m/z* 367.2209, found, 367.2223.



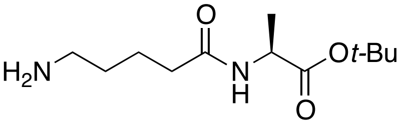
| solvent | yield of **2a** (%) | solvent | yield of **2a** (%) | solvent | yield of **2a** (%) |
|---|---|---|---|---|---|
| no solvent | 99 | 2-Me-THF | 69 | DMA | 42 |
| toluene | 74 | 1,4-dioxane | 46 | DMSO | 29 |
| benzene | 58 | *i*-$Pr_2O$ | 81 | NBP | 65 |
| PhCl | 53 | TBME | 76 | $H_2O$ | 32 |
| $CHCl_3$ | 47 | DME | 58 | EtOH | 48 |
| DCM | 52 | AcOEt | 55 | *i*-PrOH | 46 |
| CPME | 88 | MeCN | 55 | *t*-BuOH | 74 |
| THF | 61 | DMF | 52 | $CF_3CH_2OH$ | 17 |



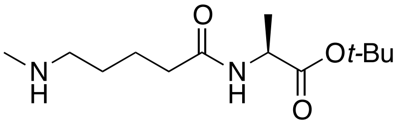
**Boc-δ-HoGly-L-Ala-O*t*-Bu (2a, entry 2):** A flame-dried 20 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-2-piperidone (**1a**, 1.20 g, 6.0 mmol) and H-L-Ala-O*t*-Bu (1.74 g, 12 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (6.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **2a** (2.00 g, 97% yield) as a white solid with >99:1 er.



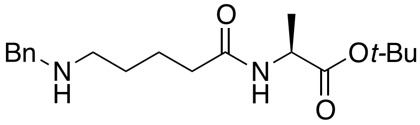
**Boc-δ-HoGly-L-Ala-O*t*-Bu (2a, entry 3):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-2-piperidone (**1a**, 199.3 mg, 1.0 mmol) and H-L-Ala-O*t*-Bu (72.6 mg, 0.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **2a** (157.9 mg, 92% yield) as a white solid with >99:1 er.



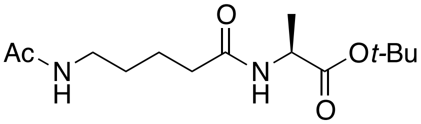
**H-δ-HoGly-L-Ala-O*t*-Bu (2b, entry 19):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with 2-piperidone (**1b**, 49.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The title compound **2b** was not produced.



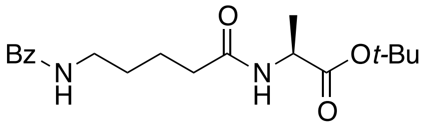
**Me-δ-HoGly-L-Ala-O*t*-Bu (2c, entry 20):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Me-2-piperidone (**1c**, 56.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The title compound **2c** was not produced.



**Bn-δ-HoGly-L-Ala-O*t*-Bu (2d, entry 21):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Bn-2-piperidone (**1d**, 94.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The title compound **2d** was not produced.

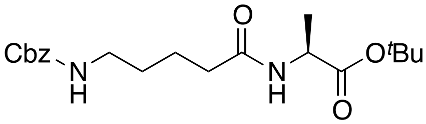


**Ac-δ-HoGly-L-Ala-O*t*-Bu (2e, entry 22):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Ac-2-piperidone (**1e**, 70.6 mg, 0.5 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the undesired compound Ac-L-Ala-O*t*-Bu (CAS No. 75552-90-8). The title compound **2e** was not produced.



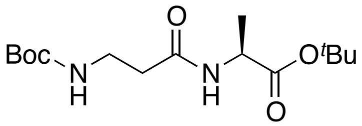
**Bz-δ-HoGly-L-Ala-O*t*-Bu (2f, entry 23):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Bz-2-piperidone (**1f**, 101.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **2f** (8.8 mg, 5% yield) as a colorless oil and undesired Bz-L-Ala-O*t*-Bu (CAS No. 67617-34-9).

$R_f = 0.35$ (AcOEt). $[\alpha]_D^{20} = -40.6$ (*c* 1.06, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.90–7.75 (m, 2H, $C_6\underline{H}_5CONH$), 7.55–7.35 (m, 3H, $C_6\underline{H}_5CONH$), 6.76 (br t, $J = 6.0$ Hz, 1H, $C_6H_5CON\underline{H}$), 6.21 (br d, $J = 7.3$ Hz, 1H, $N\underline{H}$), 4.45 (quin, $J = 7.3$ Hz, 1H, $C\underline{H}CH_3$), 3.46 (td, $J = 6.4, 6.0$ Hz, 2H, $C\underline{H}_2CH_2CH_2CH_2$), 2.40–2.20 (m, 2H, $CH_2CH_2CH_2C\underline{H}_2$), 1.80–1.60 (m, 4H, $CH_2C\underline{H}_2C\underline{H}_2CH_2$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.36 (d, $J = 7.3$ Hz, 3H, $CHC\underline{H}_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 172.3 (2C), 167.5, 134.6, 131.2, 128.4 (2C), 126.9 (2C), 81.9, 48.5, 39.2, 35.5, 28.7, 27.9 (3C), 22.3, 18.5. IR (thin film, $cm^{-1}$) 3291, 3064, 2978, 1734, 1637, 1536, 1454, 1367, 1306, 1224, 1146. HRMS (ESI) calculated for $C_{19}H_{28}N_2O_4Na$ $[M+Na]^+$ *m/z* 371.1947, found, 371.1903.



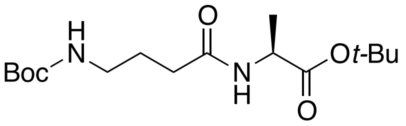
**Cbz-δ-HoGly-L-Ala-O*t*-Bu (2g, entry 24):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Cbz-2-piperidone (**1g**, 116.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **2g** (81.6 mg, 43% yield) as a white solid with >99:1 er and undesired Cbz-L-Ala-O*t*-Bu (CAS No. 50300-96-4).

$R_f$ = 0.48 (80% AcOEt in hexane). M.p. 61–62 °C. $[\alpha]_D^{28}$ = –35.3 (*c* 1.22, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.25 (m, 5H, $C_6\underline{H}_5CH_2OCONH$), 6.16 (br d, *J* = 7.3 Hz, 1H, $N\underline{H}$), 5.09 (s, 2H, $C_6H_5C\underline{H}_2OCONH$), 5.02 (br t, *J* = 6.4 Hz, 1H, $C_6H_5CH_2OCON\underline{H}$), 4.45 (quin, *J* = 7.3 Hz, 1H, $C\underline{H}CH_3$), 3.20 (q, *J* = 6.4 Hz, 2H, $C\underline{H}_2CH_2CH_2CH_2$), 2.25 (dd, *J* = 15.5, 7.3 Hz, 1H, $CH_2CH_2CH_2C\underline{H}_2$), 2.19 (dd, *J* = 15.5, 6.8 Hz, 1H, $CH_2CH_2CH_2C\underline{H}_2$), 1.75–1.60 (m, 2H, $CH_2CH_2C\underline{H}_2CH_2$), 1.60–1.45 (m, 2H, $CH_2C\underline{H}_2CH_2CH_2$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.35 (d, *J* = 7.3 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.4, 172.0, 156.4, 136.6, 128.4 (2C), 128.0, 128.0 (2C), 81.9, 66.5, 48.4, 40.4, 35.7, 29.2, 27.9 (3C), 22.4, 18.6. IR (thin film, cm$^{-1}$) 3344, 3307, 2977, 2948, 1736, 1679, 1652, 1538, 1517, 1210, 1155. HRMS (ESI) calculated for $C_{20}H_{30}N_2O_5Na$ $[M+Na]^+$ *m/z* 401.2052, found, 401.2044.



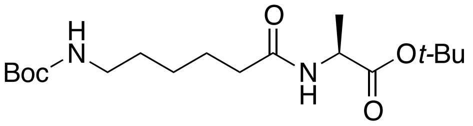
**Boc-β-HoGly-L-Ala-O*t*-Bu (2h, entry 25):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-2-azetidinone (**1h**, 85.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **2h** (143.9 mg, 91% yield) as a colorless oil with >99:1 er.

$R_f$ = 0.60 (80% AcOEt in hexane). $[\alpha]_D^{16}$ = –39.8 (*c* 1.03, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.19 (br d, *J* = 7.1 Hz, 1H, $N\underline{H}$), 5.22 (br t, *J* = 6.2 Hz, 1H, $(C\underline{H}_3)_3COCON\underline{H}$), 4.45 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 3.41 (dt, *J* = 6.2, 6.0 Hz, 2H, $C\underline{H}_2CH_2$), 2.42 (t, *J* = 6.0 Hz, 2H, $CH_2C\underline{H}_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.37 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.2, 170.9, 156.0, 82.0, 79.2, 48.6, 36.5, 36.1, 28.4 (3C), 27.9 (3C), 18.5. IR (thin film, cm$^{-1}$) 3323, 2978, 2935, 1714, 1651, 1516, 1454, 1366, 1246, 1147, 1049. HRMS (ESI) calculated for $C_{15}H_{28}N_2O_5Na$ $[M+Na]^+$ *m/z* 339.1896, found, 339.1944.



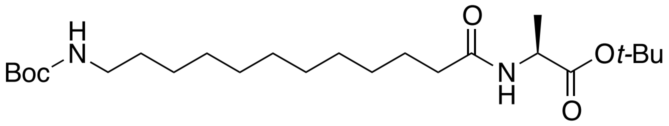
**Boc-γ-HoGly-L-Ala-O*t*-Bu (2i, entry 26):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-2-pyrrolidinone (**1i**, 92.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (217.8 mg, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **2i** (120.6 mg, 73% yield) as a colorless oil with >99:1 er.

$R_f$ = 0.43 (80% AcOEt in hexane). $[\alpha]_D^{25}$ = –25.0 (*c* 1.16, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.43 (br d, *J* = 7.1 Hz, 1H, N*H*), 4.90–4.70 (m, 1H, $(CH_3)_3COCONH$), 4.45 (quin, *J* = 7.1 Hz, 1H, C*H*$CH_3$), 3.30–3.10 (m, 2H, $CH_2CH_2CH_2$), 2.30–2.20 (m, 2H, $CH_2CH_2CH_2$), 1.90–1.75 (m, 2H, $CH_2CH_2CH_2$), 1.47 (s, 9H, $CO_2C(CH_3)_3$), 1.44 (s, 9H, $(CH_3)_3COCONH$), 1.37 (d, *J* = 7.1 Hz, 3H, $CHCH_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.4, 171.9, 156.2, 81.9, 79.2, 48.6, 39.8, 33.5, 28.4 (3C), 27.9 (3C), 26.0, 18.5. IR (thin film, cm$^{-1}$) 3314, 2978, 2934, 1692, 1651, 1523, 1453, 1366, 1249, 1147, 1048. HRMS (ESI) calculated for $C_{16}H_{30}N_2O_5Na$ $[M+Na]^+$ *m/z* 353.2052, found, 353.2053.



**Boc-ε-HoGly-L-Ala-O*t*-Bu (2j, entry 27):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-ε-caprolactam (**1j**, 106.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **2j** (162.2 mg, 90% yield) as a colorless oil with >99:1 er.

$R_f$ = 0.50 (80% AcOEt in hexane). $[\alpha]_D^{25}$ = –28.1 (*c* 1.14, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.05 (br d, *J* = 7.1 Hz, 1H, $N\underline{H}$), 4.65–4.50 (m, 1H, $(C\underline{H}_3)_3COCON\underline{H}$), 4.47 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 3.20–3.00 (m, 2H, $C\underline{H}_2CH_2CH_2CH_2CH_2$), 2.30–2.15 (m, 2H, $CH_2CH_2CH_2CH_2C\underline{H}_2$), 1.75–1.60 (m, 2H, $CH_2C\underline{H}_2CH_2CH_2CH_2$), 1.60–1.25 (m, 4H, $CH_2CH_2C\underline{H}_2C\underline{H}_2CH_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.36 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.5, 172.1, 156.0, 81.9, 79.0, 48.4, 40.3, 36.3, 29.7, 28.4 (3C), 27.9 (3C), 25.1, 18.7. IR (thin film, cm$^{-1}$) 3320, 2978, 2934, 1693, 1652, 1525, 1454, 1366, 1247, 1148, 1046. HRMS (ESI) calculated for $C_{18}H_{34}N_2O_5Na$ $[M+Na]^+$ *m/z* 381.2365, found, 381.2358.

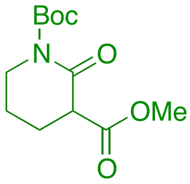


**Boc-λ-HoGly-L-Ala-O*t*-Bu (2k, entry 28):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-λ-caprolactam (**1k**, 146.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (217.8 mg, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **2k** (143.8 mg, 65% yield) as a white solid with >99:1 er.

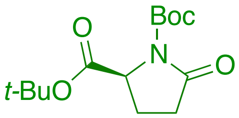
$R_f$ = 0.61 (50% AcOEt in hexane). M.p. 72–74 °C. $[\alpha]_D^{22}$ = –48.2 (*c* 1.64, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.08 (br d, *J* = 7.1 Hz, 1H, $N\underline{H}$), 4.60–4.50 (m, 1H, $(C\underline{H}_3)_3COCON\underline{H}$), 4.47 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 3.10 (q, *J* = 6.4 Hz, 2H, $C\underline{H}_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2$), 2.30–2.10 (m, 2H, $CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2C\underline{H}_2$), 1.70–1.55 (m, 2H, $CH_2C\underline{H}_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2$), 1.55–1.20 (m, 16H, $CH_2CH_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2CH_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.36 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.5, 172.4, 155.9, 81.9, 78.9, 48.4, 40.6, 36.6, 30.0, 29.4, 29.4, 29.3, 29.2, 29.2, 29.2, 28.4 (3C), 27.9

(3C), 26.7, 25.5, 18.8. IR (thin film, cm$^{-1}$) 3338, 2920, 2850, 1737, 1696, 1661, 1524, 1455, 1365, 1235, 1151. HRMS (ESI) calculated for $C_{24}H_{46}N_2O_5Na$ $[M+Na]^+$ *m/z* 465.3304, found, 465.3308.

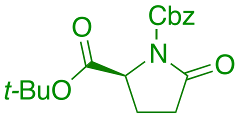
## 6. Properties of lactams 3a–m (Scheme 2)



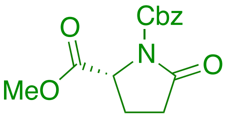
***rac*-Methyl *N*-Boc-2-oxopiperidine-3-carboxylate (3a, CAS No. 400073-68-9):** *rac*-Methyl *N*-Boc-2-oxopiperidine-3-carboxylate was purchased from Accela ChemBio Inc. (SY026045) and used as delivered.



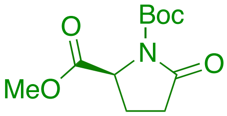
**Boc-L-Pyr-O*t*-Bu (3b, CAS No. 91229-91-3):** Boc-L-Pyr-O*t*-Bu was purchased from Tokyo Chemical Industry Co., Ltd. (B5216) and used as delivered.



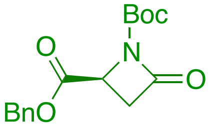
**Cbz-L-Pyr-O*t*-Bu (3c, CAS No. 81470-51-1):** Cbz-L-Pyr-O*t*-Bu was purchased from AmBeed, Inc. (A346058) and used as delivered.



**Cbz-D-Pyr-OMe (3d, CAS No. 690211-34-8):** Cbz-D-Pyr-OMe was purchased from Combi-Blocks, Inc. (YC-2555) and used as delivered.

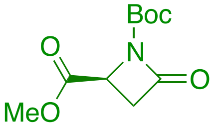


**Boc-L-Pyr-OMe (3e, CAS No. 108963-96-8):** Boc-L-Pyr-OMe was purchased from Tokyo Chemical Industry Co., Ltd. (M2803) and used as delivered.



**(*S*)-Benzyl *N*-Boc-4-oxoazetidine-2-carboxylate (3f, CAS No. 146849-54-9)[3]:** (*S*)-Benzyl *N*-Boc-4-oxoazetidine-2-carboxylate was prepared from commercially avairable (*S*)-benzyl 4-oxoazetidine-2-carboxylate (Sigma-Aldrich Co. LLC., 468975), $Boc_2O$ (Tokyo Chemical Industry Co., Ltd., D1547), DMAP (FUJIFILM Wako Pure Chemical Co., 042-19212), and triethylamine (FUJIFILM Wako Pure Chemical Co., 202-02646) according to the literature method.[3]

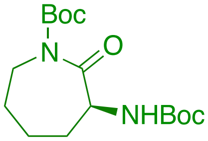
$R_f$ = 0.33 (20% AcOEt in hexane). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.45–7.30 (m, 5H, $CO_2CH_2C_6\underline{H}_5$), 5.27 (d, *J* = 12.1 Hz, 1H, $CO_2C\underline{H}_2C_6H_5$), 5.20 (d, *J* = 12.1 Hz, 1H, $CO_2C\underline{H}_2C_6H_5$), 4.42 (dd, *J* = 6.6, 3.2 Hz, 1H, $C\underline{H}CH_2CO$), 3.25 (dd, *J* = 15.8, 6.6 Hz, 1H, $CHC\underline{H}_2CO$), 2.98 (dd, *J* = 15.8, 3.2 Hz, 1H, $CHC\underline{H}_2CO$), 1.45 (s, 9H, $(C\underline{H}_3)_3COCON$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.1, 162.1, 146.8, 134.8, 128.7 (3C), 128.5 (2C), 84.0, 67.6, 49.6, 41.3, 27.8. IR (thin film, cm$^{-1}$) 2979, 1813, 1723, 1456, 1324, 1253, 1187, 1145, 1094, 1044, 1032.



**(*S*)-Methyl *N*-Boc-4-oxoazetidine-2-carboxylate (3g, CAS No. 107020-13-3)[4]:** (*S*)-Methyl *N*-Boc-4-oxoazetidine-2-carboxylate was prepared from commercially avairable (*S*)-1-Boc-azetidine-2-carboxylic acid methyl ester (Synthonic, Inc., A8195), $NaIO_4$ (FUJIFILM Wako Pure Chemical Co., 199-02401), and $RuO_2$ hydrate (Sigma-Aldrich Co. LLC., 208833-1G) according to the literature method.[4]

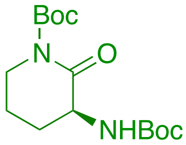
$R_f$ = 0.40 (30% AcOEt in hexane). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.39 (dd, *J* = 6.6, 3.2 Hz, 1H, $C\underline{H}CH_2CO$), 3.82 (s, 3H, $CO_2C\underline{H}_3$), 3.26 (dd, *J* = 15.8, 6.6 Hz, 1H, $CHC\underline{H}_2CO$), 3.00 (dd, *J* = 15.8, 3.2 Hz, 1H, $CHC\underline{H}_2CO$), 1.51 (s, 9H, $(C\underline{H}_3)_3COCON$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ

169.7, 162.2, 146.9, 84.0, 52.7, 49.4, 41.2, 27.9. IR (thin film, cm$^{-1}$) 2982, 1814, 1720, 1439, 1324, 1254, 1217, 1144, 1098, 1045, 1031.



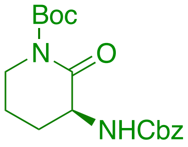
**(*S*)-*N,N′*-di-Boc-3-aminoazepan-2-one (3h):** A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable (*S*)-3-aminoazepan-2-one hydrochloride (1.0 g, 6.07 mmol), DMAP (2.23 g, 18.2 mmol), Boc$_2$O (3.98 g, 18.2 mmol), and DCM–CHCl$_3$ (5:2, 70 mL). After Et$_3$N (2.54 mL, 18.2 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. Next, the reaction mixture was transferred to a separatory funnel with CHCl$_3$ (200 mL) and saturated aqueous solution of NH$_4$Cl (100 mL) was added. The phases were separated and aqueous layer was extracted with CHCl$_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over MgSO$_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with CHCl$_3$ (15 mL), transferred onto SiO$_2$ column by a pipette, and used round-bottom flask and pipette were washed with CHCl$_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (30–100% AcOEt in hexane) to provide (*S*)-*N*-Boc-3-aminoazepan-2-one (CAS No. 76944-95-1, 824.6 mg, 59% yield). A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with (*S*)-*N*-Boc-3-aminoazepan-2-one (CAS No. 76944-95-1, 804.6 mg, 3.52 mmol), DMAP (861.2 mg, 7.05 mmol), Boc$_2$O (1.54 g, 7.05 mmol), and DCM (50 mL). After Et$_3$N (0.49 mL, 3.52 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. Next, the reaction mixture was transferred to a separatory funnel with CHCl$_3$ (200 mL) and saturated aqueous solution of NH$_4$Cl (100 mL) was added. The phases were separated and aqueous layer was extracted with CHCl$_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over MgSO$_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with CHCl$_3$ (15 mL), transferred onto SiO$_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (10–30% AcOEt in hexane) to provide the title compound **3h** (1.10 g, 95% yield) as a colorless oil.

$R_f = 0.40$ (20% AcOEt in hexane). $[\alpha]_D^{24} = +6.7$ (*c* 1.20, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) δ 5.83 (d, *J* = 6.6 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.54 (ddd, *J* = 11.0, 6.6, 1.8 Hz, 1H, $C\underline{H}CH_2CH_2CH_2CH_2NCO_2C(CH_3)_3$), 4.29 (ddt, *J* = 15.4, 5.0, 1.6 Hz, 1H, $CHCH_2CH_2CH_2C\underline{H}_2NCO_2C(CH_3)_3$), 3.31 (dd, *J* = 15.4, 11.2 Hz, 1H, $CHCH_2CH_2CH_2C\underline{H}_2NCO_2C(CH_3)_3$), 2.15–1.40 (m, 6H, $CHC\underline{H}_2C\underline{H}_2C\underline{H}_2CH_2NCO_2C(CH_3)_3$), 1.53 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.44 (s, 9H, $CHCH_2CH_2CH_2CH_2NCO_2C(C\underline{H}_3)_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 173.2, 155.1, 152.5, 83.6, 79.6, 54.9, 45.4, 33.0, 28.3 (3C), 27.9 (3C), 27.7, 27.2. IR (thin film, $cm^{-1}$) 3429, 2978, 2933, 1774, 1698, 1486, 1366, 1279, 1254, 1136, 1055. HRMS (ESI) calculated for $C_{16}H_{28}N_2O_5Na$ $[M+Na]^+$ *m/z* 351.1896, found, 351.1900.

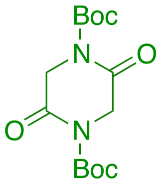


***tert*-Butyl (3*S*)-3-{[(*tert*-butoxy)carbonyl]amino}-2-oxopiperidine-1-carboxylate (3i, CAS No. 1277168-39-4):** A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable (*S*)-*tert* butyl (2-oxopiperidin-3-yl)carbamate (1.50 g, 7.0 mmol), DMAP (1.71 g, 14.0 mmol), $Boc_2O$ (3.06 g, 14.0 mmol), and DCM (70 mL). After $Et_3N$ (0.98 mL, 7.0 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. Next, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (20–50% AcOEt in hexane) to provide the title compound **3i** (1.67 g, 76% yield) as a colorless oil.

$R_f$ = 0.45 (30% AcOEt in hexane). $^1$H NMR (400 MHz, $CDCl_3$) δ 5.50 (br s, 1H, $(CH_3)_3COCON\underline{H}$), 4.35–4.20 (m, 1H, $C\underline{H}CH_2CH_2CH_2NCO_2C(CH_3)_3$), 3.99 (dt, $J$ = 13.5, 5.9 Hz, 1H, $CHCH_2CH_2C\underline{H_2}NCO_2C(CH_3)_3$), 3.52 (ddd, $J$ = 13.5, 8.4, 5.3 Hz, 1H, $CHCH_2CH_2C\underline{H_2}NCO_2C(CH_3)_3$), 2.60–2.45 (m, 1H, $CHCH_2C\underline{H_2}CH_2NCO_2C(CH_3)_3$), 2.00–1.80 (m, 2H, $CHC\underline{H_2}C\underline{H_2}CH_2NCO_2C(CH_3)_3$), 1.60–1.45 (m, 1H, $CHC\underline{H_2}CH_2CH_2NCO_2C(CH_3)_3$), 1.53 (s, 9H, $(C\underline{H_3})_3COCONH$), 1.45 (s, 9H, $CHCH_2CH_2CH_2NCO_2C(C\underline{H_3})_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.2, 155.5, 151.9, 83.5, 79.8, 52.6, 43.8, 28.3 (3C), 27.9 (3C), 26.8, 20.3. IR (thin film, $cm^{-1}$) 3389, 2978, 2933, 1773, 1705, 1495, 1366, 1279, 1242, 1142, 1054.

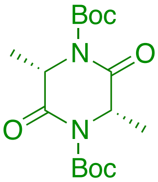


**_tert_-Butyl (3_S_)-3-{[(benzyloxy)carbonyl]amino}-2-oxopiperidine-1-carboxylate (3j, CAS No. 1277168-36-1):** *tert*-Butyl (3*S*)-3-{[(benzyloxy)carbonyl]amino}-2-oxopiperidine-1-carboxylate was purchased from Chemspace LLC. (BBV-300822848) and used as delivered.



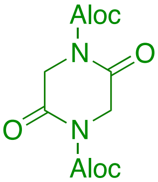
**Cyclo(-Boc-Gly-Boc-Gly-) (3k, CAS No. 151692-07-8)[5]:** Cyclo(-Boc-Gly-Boc-Gly-) was prepared from commercially avairable Cyclo(-Gly-Gly-) (Kanto Chemical Co. Inc., 17568-31), $Boc_2O$ (Tokyo Chemical Industry Co., Ltd., D1547), DMAP (FUJIFILM Wako Pure Chemical Co., 042-19212), and triethylamine (FUJIFILM Wako Pure Chemical Co., 202-02646) according to the literature method.[5]

$R_f$ = 0.36 (30% AcOEt in hexane). M.p. 140–143 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 4.43 (s, 4H, $C\underline{H_2}$), 1.54 (s, 18H, $(C\underline{H_3})_3COCON$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 164.4 (2C), 149.5 (2C), 85.1 (2C), 49.5 (2C), 27.9 (6C). IR (thin film, $cm^{-1}$) 2982, 1787, 1726, 1715, 1366, 1293, 1248, 1206, 1139, 1085, 846.



**Cyclo(-Boc-L-Ala-Boc-L-Ala-) (3l, CAS No. 959865-87-3)[5]:** A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable Cyclo(-L-Ala-L-Ala-) (4.26 g, 30.0 mmol), DMAP (733.0 mg, 6.0 mmol), $Boc_2O$ (26.2 g, 120.0 mmol), and $CHCl_3$–DMF (35:1, 36 mL). After $Et_3N$ (8.33 mL, 60.0 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. Next, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (10–50% AcOEt in hexane) to provide the title compound **3l** (9.45 g, 92% yield) as a white solid.

$R_f$ = 0.52 (30% AcOEt in hexane). M.p. 101–102 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ $^1$H NMR (400 MHz, $CDCl_3$) δ 4.87 (q, *J* = 7.4 Hz, 2H, $C\underline{H}CH_3$), 1.64 (d, *J* = 7.4 Hz, 6H, $CHC\underline{H}_3$), 1.55 (s, 18H, $(C\underline{H}_3)_3COCON$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 168.0 (2C), 149.8 (2C), 84.8 (2C), 56.0 (2C), 27.9 (6C), 20.4 (2C). IR (thin film, cm$^{-1}$) 2979, 2942, 1768, 1722, 1696, 1459, 1368, 1238, 1142, 1080, 1029.

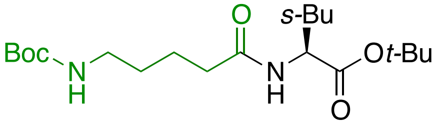


**Cyclo(-Aloc-Gly-Aloc-Gly-) (3m):** A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable Cyclo(-Gly-Gly-) (1.39 g, 12.2 mmol), DMAP (149.2 g, 1.22 mmol), and DMF (50 mL). After

$Aloc_2O$ (5.0 g, 26.9 mmol) was added to the mixture at room temperature, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. Next, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **3m** (355.3 mg, 10% yield) as a colorless oil.

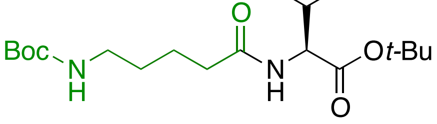
$R_f = 0.42$ (50% AcOEt in hexane). $^1H$ NMR (400 MHz, $CDCl_3$) δ $^1H$ NMR (400 MHz, $CDCl_3$) δ 5.96 (ddt, $J = 17.2, 10.3, 5.7$ Hz, 2H, $CH_2C\underline{H}CH_2OCON$), 5.45 (ddd, $J = 17.2, 2.5, 1.2$ Hz, 2H, $C\underline{H}_2CHCH_2OCON$), 5.33 (ddd, $J = 10.3, 2.5, 1.2$ Hz, 2H, $C\underline{H}_2CHCH_2OCON$), 4.79 (dt, $J = 5.7, 1.2$ Hz, 4H, $CH_2CHC\underline{H}_2OCON$), 4.55 (s, 4H, $C\underline{H}_2$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 163.8 (2C), 151.2 (2C), 130.5 (2C), 119.9 (2C), 68.5 (2C), 49.5 (2C). IR (thin film, $cm^{-1}$) 2945, 1782, 1717, 1438, 1363, 1297, 1276, 1085, 985, 933, 768. HRMS (ESI) calculated for $C_{12}H_{14}N_2O_6Na$ $[M+Na]^+$ *m/z* 305.0750, found, 305.0799.

## 7. Properties of peptides 2aa–ab, 4a–m (Scheme 2)



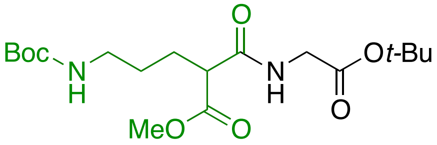
**Boc-δ-HoGly-L-Ile-O*t*-Bu (2aa):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-2-piperidone (**1a**, 99.6 mg, 0.50 mmol) and H-L-Ile-O*t*-Bu (187.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **2aa** (192.5 mg, >99% yield) as a colorless oil with >99:1 dr.

$R_f = 0.71$ (80% AcOEt in hexane). $[\alpha]_D^{29} = -19.5$ (*c* 1.08, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) δ 6.07 (br d, *J* = 8.5 Hz, 1H, $N\underline{H}$), 4.68 (br t, *J* = 6.4 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.50 (dd, *J* = 8.5, 4.6 Hz, 1H, $C\underline{H}CH(CH_3)CH_2CH_3$), 3.13 (q, *J* = 6.4 Hz, 2H, $C\underline{H_2}CH_2CH_2CH_2$), 2.28 (dd, *J* = 15.0, 7.5 Hz, 1H, $CH_2CH_2CH_2C\underline{H_2}$), 2.22 (dd, *J* = 15.0, 7.5 Hz, 1H, $CH_2CH_2CH_2C\underline{H_2}$), 1.90–1.80 (m, 1H, $CHC\underline{H}(CH_3)CH_2CH_3$), 1.75–1.40 (m, 5H, $CH_2C\underline{H_2}C\underline{H_2}CH_2$ and $CHCH(CH_3)C\underline{H_2}CH_3$), 1.47 (s, 9H, $CO_2C(C\underline{H_3})_3$), 1.44 (s, 9H, $(C\underline{H_3})_3COCONH$), 1.30–1.10 (m, 1H, $CHCH(CH_3)C\underline{H_2}CH_3$), 0.93 (t, *J* = 7.5 Hz, 3H, $CHCH(CH_3)CH_2C\underline{H_3}$), 0.90 (d, *J* = 6.9 Hz, 3H, $CHCH(C\underline{H_3})CH_2CH_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 172.3, 171.2, 156.0, 82.0, 79.0, 56.5, 39.9, 38.1, 36.0, 29.4, 28.4 (3C), 28.0 (3C), 25.3, 22.6, 15.3, 11.7. IR (thin film, $cm^{-1}$) 3626, 2969, 2933, 1714, 1694, 1652, 1520, 1366, 1247, 1161, 1144. HRMS (ESI) calculated for $C_{20}H_{38}N_2O_5Na$ $[M+Na]^+$ *m/z* 409.2678, found, 409.2661.



**Boc-δ-HoGly-L-Thr(*t*-Bu)-O*t*-Bu (2ab):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *N*-Boc-2-piperidone (**1a**, 99.6 mg, 0.50 mmol) and H-L-Thr(*t*-Bu)-O*t*-Bu (231.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **2ab** (214.5 mg, >99% yield) as a colorless oil with >99:1 dr.

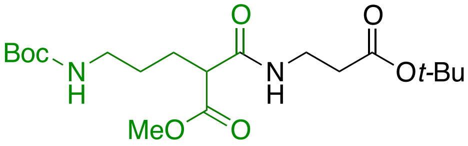
$R_f$ = 0.72 (80% AcOEt in hexane). $[\alpha]_D^{30} = -1.0$ (*c* 1.04, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) δ 6.16 (br d, *J* = 9.3 Hz, 1H, N$\underline{H}$), 4.67 (br t, *J* = 6.3 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.38 (dd, *J* = 9.3, 2.0 Hz, 1H, $C\underline{H}CH(CH_3)OC(CH_3)_3$), 4.20 (qd, *J* = 6.2 Hz, 1H, $CHC\underline{H}(CH_3)OC(CH_3)_3$), 3.14 (q, *J* = 6.3 Hz, 2H, $C\underline{H_2}CH_2CH_2CH_2$), 2.30 (t, *J* = 7.5 Hz, 2H, $CH_2CH_2CH_2C\underline{H_2}$), 1.80–1.45 (m, 4H, $CH_2C\underline{H_2}C\underline{H_2}CH_2$), 1.46 (s, 9H, $CO_2C(C\underline{H_3})_3$), 1.44 (s, 9H, $(C\underline{H_3})_3COCONH$), 1.17 (s, 9H, $CHCH(CH_3)OC(C\underline{H_3})_3$), 1.13 (d, *J* = 6.2 Hz, 3H, $CHCH(C\underline{H_3})OC(CH_3)_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 173.0, 170.0, 156.0, 81.8, 79.0, 73.8, 67.2, 58.2, 40.0, 35.9, 29.4, 28.7 (3C), 28.4 (3C), 28.1 (3C), 22.6, 21.0. IR (thin film, $cm^{-1}$) 3366, 2976, 2933, 1713, 1662, 1506, 1365, 1247, 1160, 1146, 1086. HRMS (ESI) calculated for $C_{22}H_{42}N_2O_6Na$ $[M+Na]^+$ *m/z* 453.2941, found, 453.2930.



**4aa:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *rac*-methyl *N*-Boc-2-oxopiperodine-3-carboxylate (**3a**, 128.6 mg, 0.50 mmol) and H-Gly-O*t*-Bu (131.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4aa** (184.5 mg, 95% yield) as a colorless oil.

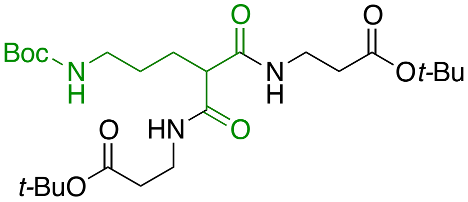
$R_f$ = 0.33 (50% AcOEt in hexane). $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.02 (br t, *J* = 5.0 Hz, 1H, N$\underline{H}$), 4.70 (br t, *J* = 5.7 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 3.93 (d, *J* = 5.0 Hz, 2H, $C\underline{H_2}$), 3.75 (s, 3H, $CH_2CH_2CH_2CHCO_2C\underline{H_3}$), 3.40–3.25 (m, 1H, $CH_2CH_2CH_2C\underline{H}CO_2CH_3$), 3.25–3.05 (m, 2H, $C\underline{H_2}CH_2CH_2CHCO_2CH_3$), 2.05–1.90 (m, 2H, $CH_2CH_2C\underline{H_2}CHCO_2CH_3$), 1.60–1.45 (m, 2H,

$CH_2C\underline{H}_2CH_2CHCO_2CH_3$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H, $(C\underline{H}_3)_3COCONH$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 171.7, 168.6, 168.3, 156.1, 82.3, 79.2, 52.5, 51.9, 42.1, 39.6, 28.4 (3C), 28.0 (3C), 27.6, 27.5. IR (thin film, $cm^{-1}$) 3341, 2976, 2933, 1739, 1714, 1668, 1519, 1366, 1247, 1150, 1036. HRMS (ESI) calculated for $C_{18}H_{32}N_2O_7Na$ $[M+Na]^+$ *m/z* 411.2107, found, 411.2102.



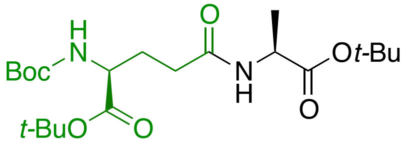
**4ab:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *rac*-methyl *N*-Boc-2-oxopiperodine-3-carboxylate (**3a**, 128.6 mg, 0.50 mmol) and H-β-HoGly-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **4ab** (199.1 mg, 99% yield) as a colorless oil.

$R_f$ = 0.31 (50% AcOEt in hexane). $^1H$ NMR (400 MHz, $CDCl_3$) δ 6.93 (br t, *J* = 6.2 Hz, 1H, $N\underline{H}$), 4.64 (br t, *J* = 6.1 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 3.60–3.40 (m, 2H, $CH_2C\underline{H}_2$), 3.73 (s, 3H, $CH_2CH_2CH_2CHCO_2C\underline{H}_3$), 3.23 (t, *J* = 7.3 Hz, 1H, $CH_2CH_2CH_2C\underline{H}CO_2CH_3$), 3.12 (q, *J* = 6.1 Hz, 2H, $C\underline{H}_2CH_2CH_2CHCO_2CH_3$), 2.45 (t, *J* = 6.2 Hz, 2H, $C\underline{H}_2CH_2$), 2.00–1.85 (m, 2H, $CH_2CH_2C\underline{H}_2CHCO_2CH_3$), 1.60–1.40 (m, 2H, $CH_2C\underline{H}_2CH_2CHCO_2CH_3$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 171.7, 171.5, 168.1, 156.0, 81.1, 79.2, 52.4, 52.2, 39.7, 35.3, 35.0, 28.4 (3C), 28.1 (3C), 27.6, 27.4. IR (thin film, $cm^{-1}$) 3300, 2978, 2937, 1727, 1715, 1695, 1661, 1520, 1366, 1248, 1154. HRMS (ESI) calculated for $C_{19}H_{34}N_2O_7Na$ $[M+Na]^+$ *m/z* 425.2264, found, 425.2253.



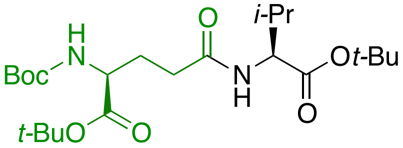
**4ac:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *rac*-methyl *N*-Boc-2-oxopiperodine-3-carboxylate (**3a**, 128.6 mg, 0.50 mmol), H-β-HoGly-O*t*-Bu (290.4 mg, 2.0 mmol), and Ta(OMe)$_5$ (16.8 mg, 0.050 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 60 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **4ac** (247.5 mg, 96% yield) as a white solid.

$R_f$ = 0.15 (50% AcOEt in hexane). M.p. 75–77 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.23 (br t, *J* = 6.0 Hz, 2H, $N\underline{H}$), 4.77 (br t, *J* = 6.4 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 3.60–3.35 (m, 4H, $C\underline{H}_2CH_2CO_2C(CH_3)_3$), 3.13 (q, *J* = 6.4 Hz, 2H, $C\underline{H}_2CH_2CH_2CH$), 3.01 (t, *J* = 7.3 Hz, 1H, $CH_2CH_2CH_2C\underline{H}$), 2.44 (t, *J* = 6.3 Hz, 4H, $CH_2C\underline{H}_2CO_2C(CH_3)_3$), 1.84 (q, *J* = 7.3 Hz, 2H, $CH_2CH_2C\underline{H}_2CH$), 1.60–1.35 (m, 2H, $CH_2C\underline{H}_2CH_2CH$), 1.45 (s, 18H, $CH_2CH_2CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H, $(C\underline{H}_3)_3COCONH$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.2 (2C), 170.6 (2C), 156.0, 81.1 (2C), 79.1, 53.8, 39.6, 35.2 (2C), 35.1 (2C), 29.8, 28.4 (3C), 28.0 (6C), 27.6. IR (thin film, cm$^{-1}$) 3296, 2977, 2932, 1727, 1683, 1666, 1531, 1365, 1278, 1249, 1149. HRMS (ESI) calculated for $C_{25}H_{45}N_3O_8Na$ [M+Na]$^+$ *m/z* 538.3104, found, 538.3092.



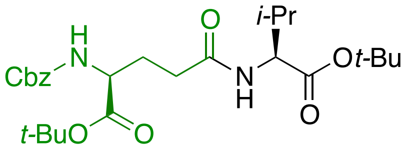
**Boc-L-Glu(L-Ala-O*t*-Bu)-O*t*-Bu (4ba, CAS No. 2374223-99-9)[6]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Boc-L-Pyr-O*t*-Bu (**3b**, 142.7 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (217.8 mg, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 60 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (1–10% MeOH in $CHCl_3$) to provide the title compound **4ba** (208.9 mg, 97% yield) as a white solid with >99:1 dr.

$R_f$ = 0.44 (5% MeOH in $CHCl_3$). M.p. 64–65 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 6.42 (br d, *J* = 7.1 Hz, 1H, $N\underline{H}$), 5.25 (br d, *J* = 7.8 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.45 (quin, *J* = 7.1 Hz, 1H, C*H*CH₃), 4.25–4.10 (m, 1H, C*H*CH₂CH₂), 2.35–2.25 (m, 2H, CHCH₂C*H*₂), 2.25–2.10 (m, 1H, CHC*H*₂CH₂), 2.00–1.80 (m, 1H, CHC*H*₂CH₂), 1.47 (s, 9H, CO₂C(C*H*₃)₃), 1.46 (s, 9H, CO₂C(C*H*₃)₃), 1.44 (s, 9H, (C*H*₃)₃COCONH), 1.38 (d, *J* = 7.1 Hz, 3H, CHC*H*₃). $^{13}$C NMR (100 MHz, CDCl₃) δ 172.2, 171.4, 171.3, 155.6, 82.1, 81.8, 79.7, 53.4, 48.6, 32.4, 29.0, 28.2 (3C), 27.9 (3C), 27.9 (3C), 18.5. IR (thin film, cm$^{-1}$) 3365, 3301, 2977, 2936, 1733, 1704, 1661, 1525, 1366, 1151, 1057.



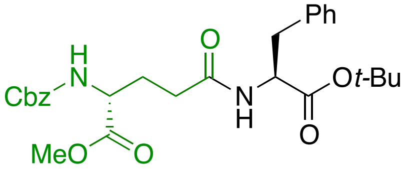
**Boc-L-Glu(L-Val-O*t*-Bu)-O*t*-Bu (4bb):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Boc-L-Pyr-O*t*-Bu (**3b**, 142.7 mg, 0.50 mmol) and H-L-Val-O*t*-Bu (260.0 mg, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 60 °C for 24 h. The reaction mixture was diluted with CHCl₃ (3.0 mL), transferred onto SiO₂ column by a pipette, and used vial and pipette were washed with CHCl₃ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–50% AcOEt in hexane) to provide the title compound **4bb** (178.4 mg, 78% yield) as a white solid with >99:1 dr.

$R_f$ = 0.70 (50% AcOEt in hexane). M.p. 105–107 °C. $[\alpha]_D^{27}$ = –52.9 (*c* 1.02, MeOH). $^1$H NMR (400 MHz, CDCl₃) δ 6.38 (br d, *J* = 8.7 Hz, 1H, N*H*), 5.24 (br d, *J* = 8.4 Hz, 1H, (CH₃)₃COCON*H*), 4.45 (dd, *J* = 8.7, 4.6 Hz, 1H, C*H*CH(CH₃)₂), 4.18 (td, *J* = 8.4, 3.6 Hz, 1H, C*H*CH₂CH₂), 2.40–2.10 (m, 4H, CHC*H*₂C*H*₂), 1.95–1.80 (m, 1H, CHC*H*(CH₃)₂), 1.47 (s, 9H, CO₂C(C*H*₃)₃), 1.46 (s, 9H, CO₂C(C*H*₃)₃), 1.45 (s, 9H, (C*H*₃)₃COCONH), 0.95 (d, *J* = 6.9 Hz, 3H, CHCH(C*H*₃)₂), 0.92 (d, *J* = 7.1 Hz, 3H, CHCH(C*H*₃)₂). $^{13}$C NMR (100 MHz, CDCl₃) δ 171.8, 171.4, 171.1, 155.7, 82.1, 81.8, 79.8, 57.4, 53.4, 32.6, 31.3, 29.4, 28.3 (3C), 28.0 (3C), 27.9 (3C), 18.8, 17.6. IR (thin film, cm$^{-1}$) 2968, 1733, 1706, 1665, 1521, 1452, 1365, 1249, 1147, 1053, 847. HRMS (ESI) calculated for $C_{23}H_{42}N_2O_7Na$ [M+Na]$^+$ *m/z* 481.2890, found, 481.2864.



**Cbz-L-Glu(L-Val-O*t*-Bu)-O*t*-Bu (4c, CAS No. 161878-63-3)[7]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cbz-L-Pyr-O*t*-Bu (**3c**, 159.7 mg, 0.50 mmol) and H-L-Val-O*t*-Bu (173.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–100% AcOEt in hexane) to provide the title compound **4c** (240.1 mg, 97% yield) as a white solid with >99:1 dr.

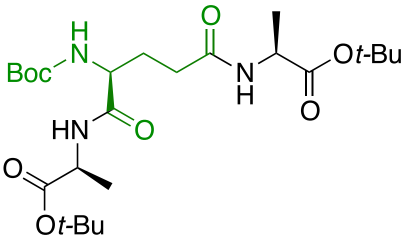
$R_f$ = 0.44 (50% AcOEt in hexane). M.p. 105–106 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.25 (m, 5H, $C_6\underline{H}_5CH_2OCONH$), 6.29 (br d, $J$ = 8.7 Hz, 1H, $N\underline{H}$), 5.57 (br d, $J$ = 8.5 Hz, 1H, $C_6H_5CH_2OCON\underline{H}$), 5.11 (s, 2H, $C_6H_5C\underline{H}_2OCONH$), 4.46 (dd, $J$ = 8.7, 4.6 Hz, 1H, $C\underline{H}CH(CH_3)_2$), 4.26 (td, $J$ = 8.5, 3.7 Hz, 1H, $C\underline{H}CH_2CH_2$), 2.40–2.10 (m, 4H, $CHC\underline{H}_2C\underline{H}_2$), 2.00–1.80 (m, 1H, $CHC\underline{H}(CH_3)_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 0.94 (d, $J$ = 6.9 Hz, 3H, $CHC\underline{H}_3$), 0.91 (d, $J$ = 6.8 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.7, 171.1, 171.0, 156.2, 136.2, 128.5 (2C), 128.1 (2C), 82.4, 81.9, 67.0, 57.4, 53.9, 32.5, 31.4, 29.1, 28.0 (3C), 27.9 (3C), 18.8, 17.6. IR (thin film, cm$^{-1}$) 3365, 3292, 2968, 2937, 1736, 1706, 1666, 1529, 1253, 1157, 1048.



**Cbz-D-Glu(L-Phe-O*t*-Bu)-OMe (4d):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cbz-D-Pyr-OMe (**3d**, 138.6 mg, 0.50 mmol) and H-L-Phe-O*t*-Bu (221.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography

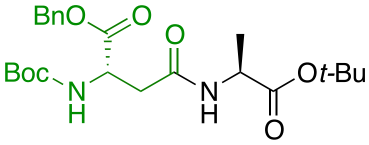
(10–100% AcOEt in hexane) to provide the title compound **4d** (178.0 mg, 76% yield) as a white solid with >99:1 dr.

$R_f$ = 0.43 (50% AcOEt in hexane). M.p. 90–91 °C. $[\alpha]_D^{23}$ = +18.2 (*c* 1.21, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.10 (m, 10H, $C_6\underline{H}_5CH_2OCONH$ and $CHCH_2C_6\underline{H}_5$), 6.28 (br d, *J* = 7.6 Hz, 1H, $N\underline{H}$), 5.70 (br d, *J* = 8.0 Hz, 1H, $C_6H_5CH_2OCON\underline{H}$), 5.10 (s, 2H, $C_6H_5C\underline{H}_2OCONH$), 4.73 (dt, *J* = 7.6, 6.2 Hz, 1H, $C\underline{H}CH_2C_6H_5$), 4.50–4.30 (m, 1H, $C\underline{H}CH_2CH_2$), 3.72 (s, 3H, $CO_2C\underline{H}_3$), 3.06 (d, *J* = 6.2 Hz, 2H, $CHC\underline{H}_2C_6H_5$), 2.40–2.10 (m, 3H, $CHC\underline{H}_2C\underline{H}_2$), 2.10–1.90 (m, 1H, $CHCH_2C\underline{H}_2$), 1.39 (s, 9H, $CO_2C(C\underline{H}_3)_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.4, 171.2, 170.7, 156.2, 136.2, 136.1, 129.4 (2C), 128.5 (2C), 128.3 (2C), 128.1, 128.1 (2C), 126.9, 82.3, 67.0, 53.6, 53.5, 52.5, 38.0, 32.2, 28.2, 27.9 (3C). IR (thin film, cm$^{-1}$) 3334, 2988, 1731, 1718, 1703, 1650, 1538, 1454, 1253, 1152, 1041. HRMS (ESI) calculated for $C_{27}H_{34}N_2O_7Na$ $[M+Na]^+$ *m/z* 521.2264, found, 521.2286.



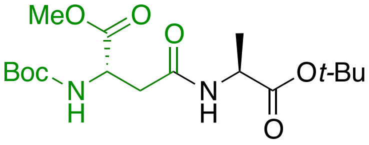
**Boc-L-Glu(L-Ala-O*t*-Bu)-L-Ala-O*t*-Bu (4e):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Boc-L-Pyr-OMe (**3e**, 121.6 mg, 0.50 mmol), H-L-Ala-O*t*-Bu (290.4 mg, 2.0 mmol), and $Ta(OMe)_5$ (16.8 mg, 0.050 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **4e** (206.4 mg, 80% yield) as a white solid with >99:1 dr.

$R_f$ = 0.62 (80% AcOEt in hexane). M.p. 42–43 °C. $[\alpha]_D^{28}$ = –69.4 (*c* 1.08, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.05–7.85 (m, 1H, $N\underline{H}$), 7.70–7.50 (m, 1H, $N\underline{H}$), 5.20–5.00 (m, 1H, $(CH_3)_3COCON\underline{H}$), 4.80–4.40 (m, 1H, $C\underline{H}CH_2CH_2$), 4.53 (quin, *J* = 7.6 Hz, 1H, $C\underline{H}CH_3$), 4.10–3.80 (m, 1H, $C\underline{H}CH_3$), 2.40–2.10 (m, 3H, $CHC\underline{H}_2C\underline{H}_2$), 1.90–1.70 (m, 1H, $CHC\underline{H}_2CH_2$), 1.48 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.42 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.39 (d, *J* = 7.6 Hz, 3H, CHC*H*$_3$), 1.35 (d, *J* = 7.6 Hz, 3H, CHC*H*$_3$). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 174.1, 172.1 (2C), 171.6, 154.6, 82.4 (2C), 79.4, 52.9, 48.4 (2C), 32.0, 29.4, 28.3 (3C), 27.9 (6C), 17.7, 17.5. IR (thin film, cm$^{-1}$) 3308, 2981, 2931, 1718, 1655, 1524, 1452, 1366, 1232, 1145, 1050. HRMS (ESI) calculated for $C_{24}H_{43}N_3O_8Na$ [M+Na]$^+$ *m/z* 524.2948, found, 524.2918.



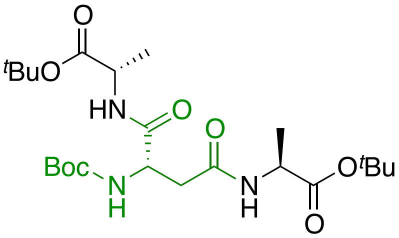
**Boc-L-Asp(L-Ala-O*t*-Bu)-OBn (4f):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with (*S*)-Benzyl *N*-Boc-4-oxoazetidine-2-carboxylate (**3f**, 152.7 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (217.8 mg, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 60 °C for 24 h. The reaction mixture was diluted with CHCl$_3$ (3.0 mL), transferred onto SiO$_2$ column by a pipette, and used vial and pipette were washed with CHCl$_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4f** (249.9 mg, 60% yield) as a white solid with >99:1 dr.

R$_f$ = 0.50 (30% AcOEt in hexane). M.p. 103–104 °C. $[\alpha]_D^{27}$ = –38.3 (*c* 1.07, MeOH). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.40–7.20 (m, 5H, CO$_2$CH$_2$C$_6$*H*$_5$), 6.20 (br d, *J* = 7.1 Hz, 1H, N*H*), 5.80 (br d, *J* = 8.5 Hz, 1H, (CH$_3$)$_3$COCON*H*), 5.21 (d, *J* = 12.4 Hz, 1H, CO$_2$C*H*$_2$C$_6$H$_5$), 5.14 (d, *J* = 12.4 Hz, 1H, CO$_2$C*H*$_2$C$_6$H$_5$), 4.57 (dt, *J* = 8.5, 4.4 Hz, 1H, C*H*CH$_2$), 4.39 (quin, *J* = 7.1 Hz, 1H, C*H*CH$_3$), 2.93 (dd, *J* = 15.8, 4.4 Hz, 1H, CHC*H*$_2$), 2.71 (dd, *J* = 15.8, 4.4 Hz, 1H, CHC*H*$_2$), 1.46 (s, 9H, CO$_2$C(C*H*$_3$)$_3$), 1.42 (s, 9H, (C*H*$_3$)$_3$COCONH), 1.29 (d, *J* = 7.1 Hz, 3H, CHC*H*$_3$). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 172.0, 171.3, 169.2, 155.6, 128.4 (2C), 128.2, 128.1 (2C), 82.1, 79.9, 67.2, 50.4, 48.6, 37.7, 28.2 (3C), 27.9 (3C), 18.5. IR (thin film, cm$^{-1}$) 3309, 3264, 2982, 2730, 1699, 1653, 1542, 1286, 1240, 1158, 1057. HRMS (ESI) calculated for $C_{23}H_{34}N_2O_7Na$ [M+Na]$^+$ *m/z* 473.2264, found, 473.2240.



**Boc-L-Asp(L-Ala-O*t*-Bu)-OMe (4ga):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with (*S*)-methyl *N*-Boc-4-oxoazetidine-2-carboxylate (**3g**, 114.6 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **4ga** (147.8 mg, 79% yield) as a white solid with >99:1 dr.

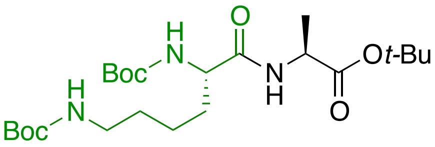
$R_f$ = 0.32 (50% AcOEt in hexane). M.p. 72–73 °C. $[\alpha]_D^{24}$ = –52.0 (*c* 1.23, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.22 (br d, *J* = 7.1 Hz, 1H, N$\underline{H}$), 5.74 (br d, *J* = 8.2 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.53 (ddd, *J* = 8.2, 4.6, 4.2 Hz, 1H, $C\underline{H}CH_2$), 4.42 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 3.75 (s, 3H, $CO_2C\underline{H}_3$), 2.92 (dd, *J* = 15.6, 4.2 Hz, 1H, $CHC\underline{H}_2$), 2.70 (dd, *J* = 15.6, 4.6 Hz, 1H, $CHC\underline{H}_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.35 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.0, 171.9, 169.2, 155.6, 82.1, 79.9, 52.5, 50.3, 48.6, 37.8, 28.3 (3C), 27.9 (3C), 18.5. IR (thin film, cm$^{-1}$) 3296, 2979, 1732, 1697, 1657, 1542, 1365, 1286, 1239, 1159, 1058. HRMS (ESI) calculated for $C_{17}H_{30}N_2O_7Na$ $[M+Na]^+$ *m/z* 397.1951, found, 397.1932.



**Boc-L-Asp(L-Ala-O*t*-Bu)-L-Ala-O*t*-Bu (4gb):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with (*S*)-methyl *N*-Boc-4-oxoazetidine-2-carboxylate (**3g**, 114.6 mg, 0.50 mmol), H-L-Ala-O*t*-Bu (290.4 mg, 2.0 mmol), and $Ta(OMe)_5$ (16.8 mg, 0.050 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100%

AcOEt in hexane) to provide the title compound **4gb** (102.9 mg, 41% yield) as a white solid with >99:1 dr.

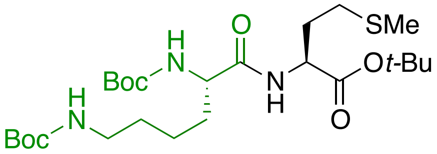
$R_f$ = 0.45 (50% AcOEt in hexane). M.p. 53–54 °C. $[\alpha]_D^{24}$ = –51.2 (*c* 1.27, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.47 (br d , *J* = 7.1 Hz, 1H, N*H*), 6.57 (br d, *J* = 7.3 Hz, 1H, N*H*), 6.11 (br d, *J* = 7.1, 1H, $(CH_3)_3COCONH$), 4.50–4.30 (m, 2H, C*H*$CH_2$ and C*H*$CH_3$), 4.40 (quin, *J* = 7.3 Hz, 1H, C*H*$CH_3$), 2.87 (dd, *J* = 15.1, 2.5 Hz, 1H, CHC*H*$_2$), 2.57 (dd, *J* = 15.1, 6.6 Hz, 1H, CHC*H*$_2$), 1.46 (s, 9H, $CO_2C(CH_3)_3$), 1.45 (s, 18H, $(CH_3)_3COCONH$ and $CO_2C(CH_3)_3$), 1.35 (d, *J* = 7.1 Hz, 3H, CHC*H*$_3$), 1.35 (d, *J* = 7.3 Hz, 3H, CHC*H*$_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.1, 171.8, 170.6, 170.4, 155.5, 82.1, 81.8, 80.1, 51.1, 48.9, 48.8, 38.0, 28.3 (3C), 27.9 (6C), 18.1, 18.0. IR (thin film, cm$^{-1}$) 3305, 2976, 1731, 1684, 1649, 1530, 1454, 1367, 1253, 1146, 1050. HRMS (ESI) calculated for $C_{23}H_{41}N_3O_8Na$ $[M+Na]^+$ *m/z* 510.2791, found, 510.2748.



**Boc-L-Lys(Boc)-L-Ala-O*t*-Bu (4ha, CAS No. 2374224-03-8)[6]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with (*S*)-*N,N'*-di-Boc-3-aminoazepan-2-one (**3h**, 164.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4ha** (234.4 mg, 99% yield) as a white solid with >99:1 dr.

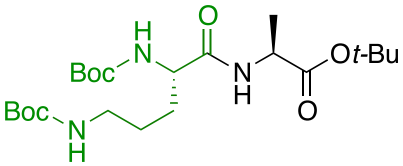
$R_f$ = 0.52 (50% AcOEt in hexane). M.p. 81–83 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 6.64 (br d, *J* = 7.1 Hz, 1H, N*H*), 5.19 (br d, *J* = 5.3 Hz, 1H, $(CH_3)_3COCONH$), 4.80–4.60 (m, 1H, $CHCH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 4.42 (quin, *J* = 7.1 Hz, 1H, C*H*$CH_3$), 4.08 (br td, *J* = 7.6, 5.3 Hz, 1H, $CHCH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 3.20–3.00 (m, 2H, $CHCH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.90–1.75 (m, 1H, $CHCH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.70–1.55 (m, 1H, $CHCH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.60–1.30 (m, 4H, $CHCH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.46 (s, 9H, $CO_2C(CH_3)_3$), 1.44 (s, 18H, $(CH_3)_3COCONH$ and $CHCH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.37 (d, *J* = 7.1 Hz, 3H, CHC*H*$_3$). $^{13}$C NMR (100

MHz, $CDCl_3$) δ 171.9, 171.4, 156.1, 155.7, 81.9, 79.9, 79.0, 54.3, 48.6, 39.9, 32.1, 29.5, 28.4 (3C), 28.3 (3C), 27.9 (3C), 22.5, 18.4. IR (thin film, $cm^{-1}$) 2974, 2938, 1686, 1655, 1509, 1456, 1365, 1246, 1148, 1046, 1018.



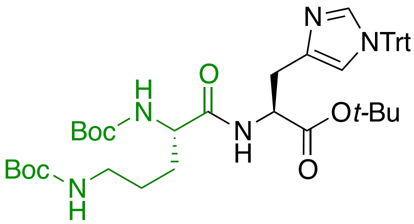
**Boc-L-Lys(Boc)-L-Met-O*t*-Bu (4hb):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with (*S*)-*N*,*N'*-di-Boc-3-aminoazepan-2-one (**3h**, 164.2 mg, 0.50 mmol) and H-L-Met-O*t*-Bu (205.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 36 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4hb** (258.9 mg, 97% yield) as a white solid with >99:1 dr.

$R_f$ = 0.58 (50% AcOEt in hexane). M.p. 35–36 °C. $[\alpha]_D^{24}$ = –32.3 (*c* 1.55, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) δ 6.75 (br d, *J* = 7.6 Hz, 1H, $N\underline{H}$), 5.17 (br d, *J* = 5.5 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.68 (br t, *J* = 6.0 Hz, 1H, $CHCH_2CH_2CH_2CH_2N\underline{H}CO_2C(CH_3)_3$), 4.56 (td, *J* = 7.6, 5.1 Hz, 1H, $C\underline{H}CH_2CH_2SCH_3$), 4.06 (br td, *J* = 8.0, 5.5 Hz, 1H, $C\underline{H}CH_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 3.11 (br td, *J* = 6.9, 6.0 Hz, 2H, $CHCH_2CH_2CH_2C\underline{H_2}NHCO_2C(CH_3)_3$), 2.60–2.40 (m, 2H, $CHCH_2C\underline{H_2}SCH_3$), 2.20–2.05 (m, 1H, $CHC\underline{H_2}CH_2SCH_3$), 2.09 (s, 3H, $CHCH_2CH_2SC\underline{H_3}$), 2.05–1.75 (m, 2H, $CHC\underline{H_2}CH_2CH_2CH_2NHCO_2C(CH_3)_3$ and $CHC\underline{H_2}CH_2SCH_3$), 1.70–1.55 (m, 1H, $CHC\underline{H_2}CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.60–1.30 (m, 4H, $CHCH_2C\underline{H_2}C\underline{H_2}CH_2NHCO_2C(CH_3)_3$), 1.47 (s, 9H, $CO_2C(C\underline{H_3})_3$), 1.44 (s, 18H, $(C\underline{H_3})_3COCONH$ and $CHCH_2CH_2CH_2CH_2NHCO_2C(C\underline{H_3})_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 171.8, 170.6, 156.1, 155.7, 82.4, 80.0, 79.1, 54.4, 52.1, 39.8, 31.9, 31.8, 29.8, 29.6, 28.4 (3C), 28.3 (3C), 28.0 (3C), 22.5, 15.4. IR (thin film, $cm^{-1}$) 3329, 2977, 2932, 1690, 1661, 1513, 1455, 1365, 1246, 1151, 1044. HRMS (ESI) calculated for $C_{25}H_{47}N_3O_7SiNa$ $[M+Na]^+$ *m/z* 556.3032, found, 556.3027.



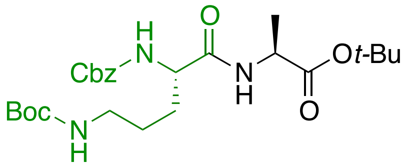
**Boc-L-Orn(Boc)-L-Ala-O*t*-Bu (4ia):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *tert*-butyl (3*S*)-3-{[(*tert*-butoxy)carbonyl]amino}-2-oxopiperidine-1-carboxylate (**3i**, 157.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **4ia** (225.1 mg, 98% yield) as a white solid with >99:1 dr.

$R_f$ = 0.50 (50% AcOEt in hexane). M.p. 41–42 °C. $[\alpha]_D^{25}$ = –25.0 (*c* 1.00, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.81 (br d, *J* = 7.3 Hz, 1H, N*H*), 5.22 (br d, *J* = 6.2 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.78 (br t, *J* = 5.7 Hz, 1H, $CHCH_2CH_2CH_2N\underline{H}CO_2C(CH_3)_3$), 4.42 (quin, *J* = 7.3 Hz, 1H, $C\underline{H}CH_3$), 4.30–4.15 (m, 1H, $C\underline{H}CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 3.40–3.20 (m, 1H, $CHCH_2CH_2C\underline{H}_2NHCO_2C(CH_3)_3$), 3.20–3.00 (m, 1H, $CHCH_2CH_2C\underline{H}_2NHCO_2C(CH_3)_3$), 1.95–1.80 (m, 1H, $CHC\underline{H}_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.70–1.50 (m, 3H, $CHC\underline{H}_2C\underline{H}_2CH_2NHCO_2C(CH_3)_3$), 1.45 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H, $CHCH_2CH_2CH_2NHCO_2C(C\underline{H}_3)_3$), 1.37 (d, *J* = 7.3 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.8, 171.6, 156.4, 155.7, 81.8, 79.8, 79.2, 53.2, 48.6, 39.4, 30.2, 28.4 (3C), 28.3 (3C), 27.9 (3C), 26.1, 18.1. IR (thin film, cm$^{-1}$) 3309, 2978, 2933, 1686, 1659, 1515, 1365, 1247, 1148, 1048, 1015. HRMS (ESI) calculated for $C_{22}H_{41}N_3O_7Na$ $[M+Na]^+$ *m/z* 482.2842, found, 482.2842.



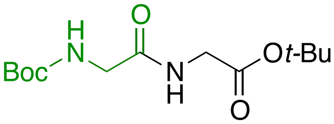
**Boc-L-Orn(Boc)-L-His(Trt)-O*t*-Bu (4ib):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *tert*-butyl (3*S*)-3-{[(*tert*-butoxy)carbonyl]amino}-2-oxopiperidine-1-carboxylate (**3i**, 157.2 mg, 0.50 mmol) and H-L-His-O*t*-Bu (680.4 mg, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 60 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **4ib** (268.8 mg, 70% yield) as a white solid with >99:1 dr.

$R_f$ = 0.38 (50% AcOEt in hexane). M.p. 58–60 °C. $[\alpha]_D^{25}$ = –10.0 (*c* 1.00, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.72 (br d, *J* = 7.8 Hz, 1H, $N\underline{H}$), 7.40–7.25 (m, 10H, $CHCH_2CNCHN(C(C_6\underline{H}_5)_3)CH$), 7.20–7.00 (m, 6H, $CHCH_2CNC\underline{H}N(C(C_6\underline{H}_5)_3)CH$), 6.59 (s, 1H, $CHCH_2CNCHN(C(C_6H_5)_3)C\underline{H}$), 5.44 (br d, *J* = 7.6 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.82 (br t, *J* = 5.5 Hz, 1H, $CHCH_2CH_2CH_2N\underline{H}CO_2C(CH_3)_3$), 4.64 (dt, *J* = 7.8, 5.4 Hz, 1H, $C\underline{H}CH_2CNCHN(C(C_6H_5)_3)CH$), 4.30–4.15 (m, 1H, $C\underline{H}CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 3.20–3.00 (m, 2H, $CHCH_2CH_2C\underline{H}_2NHCO_2C(CH_3)_3$), 2.98 (d, *J* = 5.4 Hz, 2H, $CHC\underline{H}_2CNCHN(C(C_6H_5)_3)CH$), 1.95–1.80 (m, 1H, $CHC\underline{H}_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.70–1.50 (m, 3H, $CHC\underline{H}_2C\underline{H}_2CH_2NHCO_2C(CH_3)_3$), 1.40 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.40 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.34 (s, 9H, $CHCH_2CH_2CH_2NHCO_2C(C\underline{H}_3)_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.4, 170.0, 156.0, 155.3, 142.2 (3C), 138.6, 136.3, 129.7 (6C), 128.0 (9C), 119.4, 81.5, 79.4, 78.8, 75.2, 53.7, 52.8, 39.9, 30.5, 29.5, 28.4 (3C), 28.3 (3C), 28.0 (3C), 25.4. IR (thin film, $cm^{-1}$) 3278, 2973, 2928, 1711, 1695, 1680, 1494, 1365, 1245, 1153, 1037. HRMS (ESI) calculated for $C_{23}H_{34}N_2O_7Na$ $[M+Na]^+$ *m/z* 790.4156, found, 790.4143.



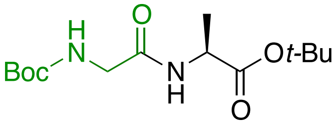
**Cbz-L-Orn(Boc)-L-Ala-O*t*-Bu (4j):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with *tert*-butyl (3*S*)-3-{[(benzyloxy)carbonyl]amino}-2-oxopiperidine-1-carboxylate (**3j**, 174.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (217.8 mg, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 36 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4j** (234.5 mg, 95% yield) as a white solid with >99:1 dr.

$R_f$ = 0.43 (50% AcOEt in hexane). M.p. 95–96 °C. $[\alpha]_D^{24}$ = –49.1 (*c* 1.14, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.25 (m, 5H, $C_6\underline{H}_5CH_2OCONH$), 6.79 (br d, *J* = 7.1 Hz, 1H, $N\underline{H}$), 5.54 (br d, *J* = 7.6 Hz, 1H, $C_6H_5CH_2COCON\underline{H}$), 5.10 (s, 2H, $C_6H_5C\underline{H}_2OCONH$), 4.75 (br t, *J* = 6.1 Hz, 1H, $CHCH_2CH_2CH_2N\underline{H}CO_2C(CH_3)_3$), 4.50–4.30 (m, 1H, $C\underline{H}CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 4.42 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 3.40–3.20 (m, 1H, $CHCH_2CH_2C\underline{H}_2NHCO_2C(CH_3)_3$), 3.15–3.00 (m, 1H, $CHCH_2CH_2C\underline{H}_2NHCO_2C(CH_3)_3$), 2.00–1.80 (m, 1H, $CHC\underline{H}_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.70–1.20 (m, 3H, $CHC\underline{H}_2C\underline{H}_2CH_2NHCO_2C(CH_3)_3$), 1.45 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H, $CHCH_2CH_2CH_2NHCO_2C(C\underline{H}_3)_3$), 1.36 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.7, 171.3, 156.5, 156.2, 136.3, 128.5 (2C), 128.1, 128.0 (2C), 81.8, 79.3, 66.9, 53.5, 48.6, 39.2, 30.4, 28.4 (3C), 27.9 (3C), 26.1, 18.0. IR (thin film, $cm^{-1}$) 3316, 2978, 1715, 1683, 1657, 1530, 1453, 1367, 1246, 1151, 1054. HRMS (ESI) calculated for $C_{25}H_{39}N_3O_7Na$ $[M+Na]^+$ *m/z* 516.2686, found, 516.2653.



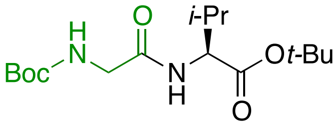
**Boc-Gly-Gly-O*t*-Bu (4ka, CAS No. 5845-68-1)[8]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-Boc-Gly-) (**3k**, 157.2 mg, 0.50 mmol) and H-Gly-O*t*-Bu (262.4 mg, 2.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **4ka** (288.0 mg, >99% yield) as a colorless oil.

$R_f$ = 0.35 (50% AcOEt in hexane). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.60 (br t, *J* = 5.3 Hz, 1H, N*H*), 5.22 (br t, *J* = 5.7 Hz, 1H, $(CH_3)_3COCONH$), 3.95 (d, *J* = 5.3 Hz, 2H, $CH_2$), 3.85 (br d, *J* = 5.7 Hz, 2H, $CH_2$), 1.47 (s, 9H, $CO_2C(CH_3)_3$), 1.46 (s, 9H, $(CH_3)_3COCONH$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.5, 168.8, 156.0, 82.4, 80.3, 44.2, 41.9, 28.3 (3C), 28.0 (3C). IR (thin film, cm$^{-1}$) 3318, 2979, 2933, 1732, 1668, 1515, 1366, 1224, 1151, 1049, 1030.



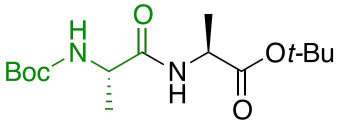
**Boc-Gly-L-Ala-O*t*-Bu (4kb, CAS No. 58177-79-0)[6]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-Boc-Gly-) (**3k**, 157.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (290.4 mg, 2.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **4kb** (296.4 mg, 98% yield) as a colorless oil with >99:1 er.

$R_f$ = 0.37 (50% AcOEt in hexane). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.66 (br d, *J* = 7.1 Hz, 1H, N*H*), 5.22 (br t, *J* = 5.5 Hz, 1H, $(CH_3)_3COCONH$), 4.47 (quin, *J* = 7.1 Hz, 1H, $CHCH_3$), 3.85 (dd, *J* = 17.0, 5.5 Hz, 1H, $CH_2$), 3.79 (dd, *J* = 17.0, 5.5 Hz, 1H, $CH_2$), 1.47 (s, 9H, $CO_2C(CH_3)_3$), 1.46 (s, 9H, $(CH_3)_3COCONH$), 1.38 (d, *J* = 7.1 Hz, 3H, $CHCH_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.0, 168.7, 155.9, 82.1, 80.1, 48.6, 44.2, 28.2 (3C), 27.9 (3C), 18.6. IR (thin film, cm$^{-1}$) 3314, 2979, 2933, 1721, 1664, 1514, 1367, 1246, 1144, 1050, 1030.



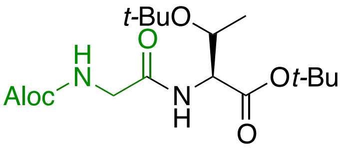
**Boc-Gly-L-Val-O*t*-Bu (4kc, CAS No. 1202014-17-2)[9]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-Boc-Gly-) (**3k**, 157.2 mg, 0.50 mmol) and H-L-Val-O*t*-Bu (346.6 mg, 2.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4kc** (301.0 mg, 91% yield) as a colorless oil with >99:1 er.

$R_f$ = 0.54 (50% AcOEt in hexane). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.57 (br d, *J* = 8.8 Hz, 1H, N*H*), 5.18 (br dd, *J* = 5.7, 5.5 Hz, 1H, $(CH_3)_3$COCON*H*), 4.46 (dd, *J* = 8.8, 4.5 Hz, 1H, C*H*CH$(CH_3)_2$), 3.87 (dd, *J* = 16.7, 5.5 Hz, 1H, C*H*$_2$), 3.78 (dd, *J* = 16.7, 5.7 Hz, 1H, C*H*$_2$), 1.47 (s, 9H, $CO_2$C(C*H*$_3$)$_3$), 1.46 (s, 9H, (C*H*$_3$)$_3$COCONH), 0.94 (d, *J* = 6.9 Hz, 3H, CHCH(C*H*$_3$)$_2$), 0.90 (d, *J* = 6.9 Hz, 3H, CHCH(C*H*$_3$)$_2$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 170.8, 169.2, 156.0, 82.1, 80.2, 57.2, 44.4, 31.4, 28.2 (3C), 28.0 (3C), 18.8, 17.5. IR (thin film, cm$^{-1}$) 3321, 2974, 2934, 1721, 1665, 1514, 1367, 1247, 1142, 1049, 1029.



**Boc-L-Ala-L-Ala-O*t*-Bu (4l, CAS No. 93836-34-1)[6]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-Boc-L-Ala-) (**3l**, 171.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (290.4 mg, 2.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4l** (94.9 mg, 30% yield) as a colorless oil with >99:1 dr.

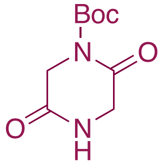
$R_f$ = 0.54 (50% AcOEt in hexane). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.66 (br d, *J* = 7.1 Hz, 1H, N*H*), 5.20–5.05 (m, 1H, $(CH_3)_3$COCON*H*), 4.43 (quin, *J* = 7.1 Hz, 1H, C*H*$CH_3$), 4.30–4.10 (m, 1H, C*H*$CH_3$), 1.46 (s, 9H, $CO_2$C(C*H*$_3$)$_3$), 1.45 (s, 9H, (C*H*$_3$)$_3$COCONH), 1.37 (d, *J* = 7.1 Hz, 3H, CHC*H*$_3$), 1.36 (d, *J* = 7.1 Hz, 3H, CHC*H*$_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.0, 171.9, 155.3, 81.9, 79.9, 50.0, 48.6, 28.3 (3C), 27.9 (3C), 18.5 (2C). IR (thin film, cm$^{-1}$) 3310, 2978, 2935, 1718, 1659, 1518, 1453, 1366, 1247, 1146, 1049.



**Aloc-Gly-L-Thr(*t*-Bu)-O*t*-Bu (4m):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Aloc-Gly-Aloc-Gly-) (**3m**, 141.1 mg, 0.50 mmol) and H-L-Thr(*t*-Bu)-O*t*-Bu (462.7 mg, 2.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **4m** (261.5 mg, 91% yield) as a colorless oil with >99:1 dr.

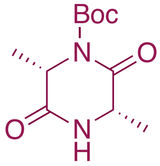
$R_f$ = 0.38 (50% AcOEt in hexane). $[\alpha]_D^{26}$ = –5.2 (*c* 1.36, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.53 (br d, *J* = 9.2 Hz, 1H, N$\underline{H}$), 5.92 (ddt, *J* = 17.2, 10.3, 5.3 Hz, 1H, $CH_2CC\underline{H}CH_2OCONH$), 5.60–5.40 (m, 1H, $CH_2CCHCH_2OCON\underline{H}$), 5.31 (ddd, *J* = 17.2, 3.2, 1.6 Hz, 1H, $C\underline{H}_2CCHCH_2OCONH$), 5.22 (ddd, *J* = 10.3, 2.5, 1.2 Hz, 1H, $C\underline{H}_2CCHCH_2OCONH$), 4.62 (dd, *J* = 13.8, 5.3 Hz, 1H, $CH_2CCHC\underline{H}_2OCONH$), 4.58 (dd, *J* = 13.8, 5.3 Hz, 1H, $CH_2CCHC\underline{H}_2OCONH$), 4.37 (dd, *J* = 9.2, 2.0 Hz, 1H, $C\underline{H}CH(CH_3)OC(CH_3)_3$), 4.20 (qd, *J* = 6.3, 2.0 Hz, 1H, $CHC\underline{H}(CH_3)OC(CH_3)_3$), 3.99 (dd, *J* = 17.2, 5.5 Hz, 1H, $C\underline{H}_2$), 3.94 (dd, *J* = 17.2, 6.0 Hz, 1H, $C\underline{H}_2$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.16 (s, 9H, $CHCH(CH_3)OC(C\underline{H}_3)_3$), 1.16 (d, *J* = 6.3 Hz, 3H, $CHCH(C\underline{H}_3)OC(CH_3)_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.6, 169.0, 156.2, 132.6, 117.8, 82.1, 73.8, 67.1, 65.9, 58.3, 44.3, 28.6 (3C), 28.0 (3C), 21.0. IR (thin film, cm$^{-1}$) 3326, 2976, 2935, 1726, 1669, 1514, 1366, 1247, 1143, 1081, 990. HRMS (ESI) calculated for $C_{18}H_{32}N_2O_6Na$ $[M+Na]^+$ *m/z* 395.2158, found, 395.2175.

## 8. Properties of lactams 5a–k (Scheme 3)



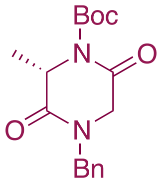
**Cyclo(-Boc-Gly-Gly-) (5a):** A flame-dried 300 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with **3k** (5.0 g, 15.9 mmol) and DCM (160 mL). After TFA (1.22 mL, 15.9 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. Next, the reaction mixture was filtered on filter paper to recover Cyclo(-Gly-Gly-) (544.2 mg, 30% yield) and the filtrate was concentrated in *vacuo* with the aid of a rotary evaporator and water bath. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The reaction mixture was purified by flash column chromatography (50–100% AcOEt in hexane → 5% MeOH in $CHCl_3$) to provide the title compound **5a** (1.26 g, 37% yield) as a white solid and recover **3k** (1.30 g, 26% yield). The recovered Cyclo(-Gly-Gly-) and **3k** were reused to synthesize **5a**.

$R_f = 0.29$ (AcOEt). M.p. >300 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.55 (br d, $J = 2.1$ Hz, 1H, N$\underline{H}$), 4.32 (s, 2H, C$\underline{H}_2$), 4.06 (br d, $J = 2.1$ Hz, 2H, C$\underline{H}_2$), 1.54 (s, 9H, (C$\underline{H}_3$)$_3$COCON). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.2, 164.7, 149.8, 84.9, 47.8, 46.5, 27.8 (3C). IR (thin film, cm$^{-1}$) 3240, 2977, 1736, 1697, 1682, 1447, 1367, 1292, 1212, 1150, 1058. HRMS (ESI) calculated for $C_9H_{14}N_2O_4Na$ $[M+Na]^+$ *m/z* 237.0851, found, 237.0895.



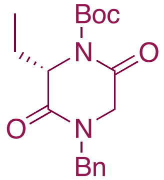
**Cyclo(-Boc-L-Ala-L-Ala-) (5b):** A flame-dried 300 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with **3l** (6.05 g, 17.7 mmol) and DCM (170 mL). After TFA (1.35 mL, 17.7 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at 0 °C for 2 h and at room temperature for 2.5 h. Next, the reaction mixture concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The reaction mixture was purified by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **5b** (1.60 g, 37% yield) as a white solid and recover **3l** (3.14 g, 52% yield). The recovered **3l** was reused to synthesize **5b**.

$R_f$ = 0.46 (AcOEt). M.p. 140–141 °C. $[\alpha]_D^{24}$ = +100.0 (*c* 1.08, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.63 (br d, *J* = 3.2 Hz, 1H, N$\underline{H}$), 4.66 (q, *J* = 7.1 Hz, 1H, C$\underline{H}$$CH_3$), 4.21 (qd, *J* = 7.3, 3.2 Hz, 1H, C$\underline{H}$$CH_3$), 1.61 (d, *J* = 7.1 Hz, 3H, CHC$\underline{H}_3$), 1.58 (d, *J* = 7.3 Hz, 3H, CHC$\underline{H}_3$), 1.55 (s, 9H, $(C\underline{H}_3)_3$COCON). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.5, 167.8, 150.2, 84.6, 54.8, 52.7, 27.8 (3C), 21.9, 20.9. IR (thin film, cm$^{-1}$) 2941, 1769, 1733, 1715, 1448, 1370, 1289, 1241, 1149, 1135, 1089. HRMS (ESI) calculated for $C_{11}H_{18}N_2O_4Na$ $[M+Na]^+$ *m/z* 265.1164, found, 265.1199.



**Cyclo(-Boc-L-Ala-Bn-Gly-) (5c):** A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable Cyclo(-L-Ala-Bn-Gly-) (1.0 g, 4.58 mmol), DMAP (1.12 g, 9.16 mmol), $Boc_2O$ (2.0 g, 9.16 mmol), and DCM (46 mL). After $Et_3N$ (0.64 mL, 4.58 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (10–50% AcOEt in hexane) to provide the title compound **5c** (1.30 g, 89% yield) as a white solid.

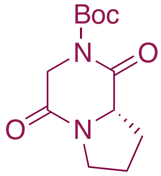
$R_f$ = 0.38 (30% AcOEt in hexane). M.p. 130–132 °C. $[\alpha]_D^{25}$ = +70.4 (*c* 1.15, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.20 (m, 5H, $C_6\underline{\mathit{H}}_5CH_2N$), 4.83 (q, *J* = 7.1 Hz, 1H, $C\underline{\mathit{H}}CH_3$), 4.71 (d, *J* = 14.6 Hz, 1H, $C_6H_5C\underline{\mathit{H}}_2N$), 4.50 (d, *J* = 14.6 Hz, 1H, $C_6H_5C\underline{\mathit{H}}_2N$), 3.94 (d, *J* = 18.3 Hz, 1H, $C\underline{\mathit{H}}_2$), 3.85 (d, *J* = 18.3 Hz, 1H, $C\underline{\mathit{H}}_2$), 1.54 (s, 9H, $(C\underline{\mathit{H}}_3)_3COCON$), 1.53 (d, *J* = 7.1 Hz, 3H, $CHCH_2C\underline{\mathit{H}}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.3, 164.6, 149.6, 134.9, 129.0 (2C), 128.2 (3C), 84.6, 55.3, 50.2, 49.2, 27.9 (3C), 18.5. IR (thin film, cm$^{-1}$) 2973, 1735, 1718, 1647, 1494, 1368, 1291, 1256, 1232, 1149, 1100. HRMS (ESI) calculated for $C_{17}H_{22}N_2O_4Na$ $[M+Na]^+$ *m/z* 341.1477, found, 341.1526.



**Cyclo(-Boc-L-Abu-Bn-Gly-) (5d, CAS No. 1932055-28-1):** A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable Cyclo(-L-Abu-Bn-Gly-) (1.0 g, 4.31 mmol), DMAP (1.05 g, 8.62 mmol), $Boc_2O$ (1.88 g, 8.62 mmol), and DCM (43 mL). After $Et_3N$ (0.60 mL, 4.31 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for overnight. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **5d** (1.29 g, 90% yield) as a white solid.

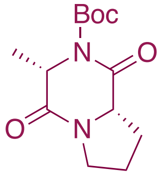
$R_f$ = 0.36 (30% AcOEt in hexane). M.p. 109–110 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.20 (m, 5H, $C_6\underline{\mathit{H}}_5CH_2N$), 4.81 (d, *J* = 14.4 Hz, 1H, $C_6H_5C\underline{\mathit{H}}_2N$), 4.75 (t, *J* = 6.9 Hz, 1H, $C\underline{\mathit{H}}CH_2CH_3$), 4.36 (d, *J* = 14.4 Hz, 1H, $C_6H_5C\underline{\mathit{H}}_2N$), 3.95 (d, *J* = 18.3 Hz, 1H, $C\underline{\mathit{H}}_2$), 3.85 (d, *J* = 18.3 Hz, 1H, $C\underline{\mathit{H}}_2$), 1.90 (qd, *J* = 7.6, 6.9 Hz, 2H, $CHC\underline{\mathit{H}}_2CH_3$), 1.54 (s, 9H, $(C\underline{\mathit{H}}_3)_3COCON$), 1.03 (t, *J* = 7.6

Hz, 3H, $CHCH_2C\underline{H}_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 166.4, 164.9, 149.8, 135.0, 129.0 (2C), 128.3 (2C), 128.2, 84.6, 60.2, 50.4, 49.2, 27.8 (3C), 26.6, 10.0. IR (thin film, $cm^{-1}$) 2973, 2935, 1778, 1668, 1493, 1391, 1296, 1247, 1234, 1146, 1109.



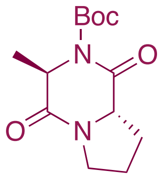
**Cyclo(-Boc-Gly-L-Pro-) (5e, CAS No. 1174732-58-1)[10]:** Cyclo(-Boc-Gly-L-Pro-) was prepared from commercially avairable Cyclo(-Gly-L-Pro-) (BLD Pharmatech Ltd., BD173941), $Boc_2O$ (Tokyo Chemical Industry Co., Ltd., D1547), DMAP (FUJIFILM Wako Pure Chemical Co., 042-19212), and triethylamine (FUJIFILM Wako Pure Chemical Co., 202-02646) according to the literature method.[10]

$R_f$ = 0.50 (AcOEt). M.p. 104–105 °C. $^1H$ NMR (400 MHz, $CDCl_3$) δ 4.67 (d, $J$ = 16.5 Hz, 1H, $C\underline{H}_2$), 4.17 (t, $J$ = 8.0 Hz, 1H, $C\underline{H}CH_2CH_2CH_2$), 4.10 (dd, $J$ = 16.5, 0.7 Hz, 1H, $C\underline{H}_2$), 3.58 (dd, $J$ = 8.1, 5.9 Hz, 2H, $CHCH_2CH_2C\underline{H}_2$), 2.45–2.20 (m, 2H, $CHC\underline{H}_2CH_2CH_2$), 2.10–1.85 (m, 2H, $CHCH_2C\underline{H}_2CH_2$), 1.54 (s, 9H, $(C\underline{H}_3)_3COCON$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 167.4, 163.2, 150.0, 84.5, 60.3, 49.7, 45.1, 28.0, 27.8 (3C), 23.1. IR (thin film, $cm^{-1}$) 2985, 2880, 1742, 1714, 1664, 1448, 1360, 1295, 1255, 1195, 1143.



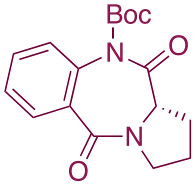
**Cyclo(-Boc-L-Ala-L-Pro-) (5f):** A flame-dried 50 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with commercially avairable Cyclo(-L-Ala-L-Pro-) (500 mg, 2.97 mmol), DMAP (72.6 mg, 0.60 mmol), $Boc_2O$ (1.30 g, 5.95 mmol), and DCM (30 mL), and $Et_3N$ (0.41 mL, 2.97 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 0 °C to room temperature for 2 h. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **5f** (256.9 mg, 32% yield) as a white solid.

$R_f = 0.59$ (AcOEt). M.p. 93–95 °C. $[\alpha]_D^{24} = -2.6$ (*c* 1.14, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.50 (q, *J* = 6.6 Hz, 1H, C$\underline{H}$CH$_3$), 4.15 (dd, *J* = 9.2, 6.6 Hz, 1H, C$\underline{H}$CH$_2$CH$_2$CH$_2$), 3.73 (dt, *J* = 11.9, 8.1 Hz, 1H, CHCH$_2$CH$_2$C$\underline{H}_2$), 3.51 (ddd, *J* = 11.9, 9.0, 3.2 Hz, 1H, CHCH$_2$CH$_2$C$\underline{H}_2$), 2.50–2.30 (m, 1H, CHC$\underline{H}_2$CH$_2$CH$_2$), 2.20–1.80 (m, 3H, CHC$\underline{H}_2$C$\underline{H}_2$CH$_2$), 1.57 (d, *J* = 6.6 Hz, 3H, CHC$\underline{H}_3$), 1.55 (s, 9H, (C$\underline{H}_3$)$_3$COCON). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.6, 164.9, 151.2, 84.9, 59.7, 55.2, 45.5, 28.9, 27.7 (3C), 22.1, 1 7.1. IR (thin film, cm$^{-1}$) 2992, 2950, 1738, 1698, 1651, 1468, 1367, 1325, 1272, 1248, 1153. HRMS (ESI) calculated for $C_{13}H_{20}N_2O_4Na$ $[M+Na]^+$ *m/z* 291.1321, found, 291.1365.



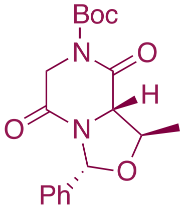
**Cyclo(-Boc-D-Ala-L-Pro-) (5g):** A flame-dried 50 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with commercially avairable Cyclo(-D-Ala-L-Pro-) (500 mg, 2.97 mmol), DMAP (72.6 mg, 0.60 mmol), $Boc_2O$ (1.30 g, 5.95 mmol), and $CHCl_3$–DMF (30:1, 31 mL), and $Et_3N$ (0.41 mL, 2.97 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at room temperature for overnight. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **5g** (524.7 mg, 66% yield) as a white solid.

$R_f$ = 0.54 (AcOEt). M.p. 112–113 °C. $[\alpha]_D^{24}$ = –105.4 (*c* 1.30, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.79 (q, *J* = 7.3 Hz, 1H, $C\underline{H}CH_3$), 4.19 (dd, *J* = 9.4, 7.1 Hz, 1H, $C\underline{H}CH_2CH_2CH_2$), 3.70–3.50 (m, 2H, $CHCH_2CH_2C\underline{H_2}$), 2.50–1.85 (m, 4H, $CHC\underline{H_2}C\underline{H_2}CH_2$), 1.54 (d, *J* = 7.3 Hz, 3H, $CHC\underline{H_3}$), 1.54 (s, 9H, $(C\underline{H_3})_3COCON$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.4, 166.1, 150.0, 84.4, 59.3, 57.3, 45.5, 29.0, 27.9 (3C), 22.6, 17.2. IR (thin film, cm$^{-1}$) 2981, 2888, 1776, 1728, 1667, 1459, 1384, 1287, 1250, 1222, 1148. HRMS (ESI) calculated for $C_{13}H_{20}N_2O_4Na$ $[M+Na]^+$ *m/z* 291.1321, found, 291.1302.



**Cyclo(-Boc-Abz-L-Pro-) (5h):** A flame-dried 50 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with commercially avairable Cyclo(-Abz-L-Pro)-) (1.0 g, 4.62 mmol), DMAP (113.0 mg, 0.93 mmol), $Boc_2O$ (2.02 g, 9.25 mmol), and DCM (46 mL), and $Et_3N$ (0.64 mL, 4.62 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 0 °C for 1.5 h. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **5h** (1.43 g, 98% yield) as a white solid.

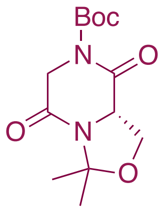
$R_f$ = 0.60 (AcOEt). M.p. 167–168 °C. $[\alpha]_D^{25}$ = –17.0 (*c* 1.06, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 7.8, 1.6 Hz, 1H, $CCHCHCHC\underline{H}C$), 7.50 (ddd, *J* = 8.0, 7.3, 1.6 Hz, 1H, $CCHC\underline{H}CHCHC$), 7.41 (ddd, *J* = 7.8, 7.3, 1.0 Hz, 1H, $CCHCHC\underline{H}CHC$), 7.23 (dd, *J* = 8.0, 1.0 Hz, 1H, $CC\underline{H}CHCHCHC$), 4.10–4.00 (m, 1H, $C\underline{H}CH_2CH_2CH_2$), 3.85 (ddd, *J* = 11.7, 8.6, 3.0 Hz, 1H, $CHCH_2CH_2C\underline{H}_2$), 3.56 (ddd, *J* = 11.7, 10.1, 6.9 Hz, 1H, $CHCH_2CH_2C\underline{H}_2$), 2.80–2.65 (m, 1H, $CHC\underline{H}_2CH_2CH_2$), 2.20–1.90 (m, 3H, $CHC\underline{H}_2C\underline{H}_2CH_2$), 1.46 (s, 9H, $(C\underline{H}_3)_3COCON$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.2, 165.0, 150.7, 135.4, 131.2, 131.1, 129.8, 127.5, 126.2, 84.8, 58.9, 46.7, 27.6 (3C), 26.4, 23.6. IR (thin film, cm$^{-1}$) 2980, 2927, 1776, 1634, 1600, 1454, 1417, 1369, 1230, 1140, 1108. HRMS (ESI) calculated for $C_{17}H_{20}N_2O_4Na$ $[M+Na]^+$ *m/z* 339.1321, found, 339.1352.



**Cyclo(-Boc-Gly-L-Thr(ψ$^{Ph,H}$pro)-) (5i):** A flame-dried 50 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with commercially avairable Cyclo(-Gly-L-Thr(ψ$^{Ph,H}$pro)-) (500 mg, 2.03 mmol), DMAP (744.2 mg, 6.09 mmol), $Boc_2O$ (1.33 g, 6.09 mmol), and DCM (20 mL), and $Et_3N$ (0.28 mL, 2.03 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at room temperature for overnight. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (10–50% AcOEt in hexane) to provide the title compound **5i** (618.8 mg, 88% yield) as a white solid.

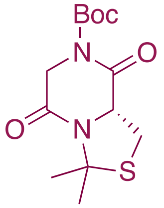
$R_f$ = 0.83 (AcOEt). M.p. 147–148 °C. $[\alpha]_D^{24}$ = –37.2 (*c* 1.05, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.45–7.30 (m, 5H, $CHCH(CH_3)OCHC_6\underline{H}_5$), 6.31 (s, 1H, $CHCH(CH_3)OC\underline{H}C_6H_5$), 4.73 (d, *J* = 17.1 Hz, 1H, $C\underline{H}_2$), 4.50 (dq, *J* = 8.9, 6.0 Hz, 1H, $CHC\underline{H}(CH_3)OCHC_6H_5$), 4.17 (d, *J* = 17.1 Hz, 1H, $C\underline{H}_2$), 3.99 (d, *J* = 8.9 Hz, 1H, $C\underline{H}CH(CH_3)OCHC_6H_5$), 1.56 (s, 9H, $(C\underline{H}_3)_3COCON$), 1.56 (d, *J* = 6.0 Hz, 3H, $CHCH(C\underline{H}_3)OCHC_6H_5$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.2, 162.0, 149.7, 136.8, 129.3, 128.7 (2C), 126.2 (2C), 88.5, 85.2, 74.0, 63.7, 50.2, 27.9

(3C), 18.1. IR (thin film, cm$^{-1}$) 2985, 2932, 1764, 1683, 1428, 1368, 1285, 1237, 1145, 1127, 1011. HRMS (ESI) calculated for $C_{18}H_{22}N_2O_5Na$ $[M+Na]^+$ *m/z* 369.1426, found, 369.1459.



**Cyclo(-Boc-Gly-L-Ser(ψ$^{Me,Me}$pro)-) (5j):** A flame-dried 500 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable Fmoc-Gly-L-Ser(ψ$^{Me,Me}$pro)-OH (10.0 g, 23.56 mmol), MeOH (9.53 mL, 235.60 mmol), WSC•HCl (5.87 g, 30.63 mmol), HOBt (4.14 g, 30.63 mmol), and $CHCl_3$ (200 mL). After $^i$Pr$_2$NEt (6.16 mL, 35.34 mmol) was added to the mixture at -5 °C, the resulting mixture was stirred under nitrogen atmosphere at -5 °C for overnight. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (300 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 200 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (30 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 10 mL). The crude product was purified by flash column chromatography (30–100% AcOEt in hexane) to provide Fmoc-Gly-L-Ser(ψ$^{Me,Me}$pro)-OMe (9.88 g, 95% yield) as a white solid. A flame-dried 300 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with Fmoc-Gly-L-Ser(ψ$^{Me,Me}$pro)-OMe (6.37 g, 14.5 mmol) and DCM (116 mL). After piperidine (29 mL) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for 2 h. After completion, the reaction mixture was concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (200 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (3 x 200 mL). The organic phase was dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (30 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 10 mL). The crude product was purified by flash column chromatography (1–5% MeOH in $CHCl_3$) to provide Cyclo(-Gly-L-Ser($\psi^{\mathrm{Me,Me}}$pro)-) (2.41 g, 90% yield) as a white solid. A flame-dried 300 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with Cyclo(-Gly-L-Ser($\psi^{\mathrm{Me,Me}}$pro)-) (2.12 g, 11.51 mmol), DMAP (281.2 mg, 2.30 mmol), $Boc_2O$ (5.02 g, 23.02 mmol), DCM (115 mL), and $Et_3N$ (1.6 mL, 11.51 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 0 °C to room temperature for overnight. After completion, the reaction mixture was concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (30 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 10 mL). The crude product was purified diretly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **5j** (2.95 g, 90% yield) as a white solid.

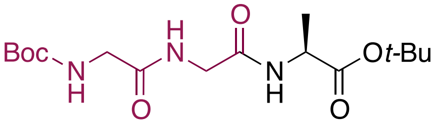
$R_f = 0.54$ (50% AcOEt in hexane). M.p. 114–115 °C. $[\alpha]_D^{25} = -122.4$ (*c* 1.07, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) δ 4.61 (d, *J* = 16.5 Hz, 1H, $C\underline{H}_2$), 4.50–4.30 (m, 2H, $C\underline{H}C\underline{H}_2OC(CH_3)_2$), 4.20–4.10 (m, 1H, $CHC\underline{H}_2OC(CH_3)_2$), 4.11 (d, *J* = 16.5 Hz, 1H, $C\underline{H}_2$), 1.65 (s, 3H, $CHCH_2OC(C\underline{H}_3)_2$), 1.60 (s, 3H, $CHCH_2OC(C\underline{H}_3)_2$), 1.55 (s, 9H, $(C\underline{H}_3)_3COCON$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 165.6, 160.8, 149.6, 95.9, 85.0, 65.2, 58.3, 50.3, 27.8 (3C), 25.2, 23.5. IR (thin film, $cm^{-1}$) 2980, 1760, 1674, 1430, 1384, 1366, 1290, 1237, 1149, 1098, 1015. HRMS (ESI) calculated for $C_{13}H_{20}N_2O_5Na$ $[M+Na]^+$ *m/z* 307.1270, found, 307.1223.



**Cyclo(-Boc-Gly-L-Cys($\psi^{\mathrm{Me,Me}}$pro)-) (5k):** A flame-dried 300 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with commercially avairable Fmoc-Gly-L-Cys($\psi^{\mathrm{Me,Me}}$pro)-OH (5.0 g, 11.35 mmol), MeOH (4.59 mL, 113.51 mmol), WSC•HCl (2.83 g, 14.76 mmol), HOBt (1.99 g, 14.76 mmol), and $CHCl_3$ (110 mL). After $Et_3N$ (2.37 mL, 17.03 mmol) was added to the mixture at -5 °C, the resulting mixture was stirred under nitrogen atmosphere at -5 °C for overnight. After completion, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (200 mL) and saturated aqueous solution of $NH_4Cl$ (100 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 100 mL). The organic phase was washed with water (50 mL) and brine (50 mL), and then dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (50% AcOEt in hexane) to provide Fmoc-Gly-L-Cys($\psi^{Me,Me}$pro)-OMe (4.73 g, 92% yield) as a white solid. A flame-dried 200 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) and nitrogen balloon was charged with Fmoc-Gly-L-Cys($\psi^{Me,Me}$pro)-OMe (2.50 g, 5.50 mmol) and DCM (55 mL). After piperidine (5.5 mL, 55.68 mmol) was added to the mixture at 0 °C, the resulting mixture was stirred under nitrogen atmosphere at room temperature for 2 h. After completion, the reaction mixture was concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (1–7.5% MeOH in $CHCl_3$) to provide Cyclo(-Gly-L-Cys($\psi^{Me,Me}$pro)-) (1.09 g, 99% yield) as a white solid. A flame-dried 50 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with commercially avairable Cyclo(-Gly-L-Cys($\psi^{Me,Me}$pro)-) (500 mg, 2.50 mmol), DMAP (61.0 mg, 0.50 mmol), $Boc_2O$ (1.90 g, 4.99 mmol), DCM (25 mL), and $Et_3N$ (0.28 mL, 2.50 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 0 °C to room temperature for overnight. After completion, the reaction mixture was concentrated in *vacuo* with the aid of a rotary evaporator and water bath without heating. The crude product was then diluted with $CHCl_3$ (15 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with $CHCl_3$ (2 x 5 mL). The crude product was purified by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **5k** (666.6 mg, 89% yield) as a white solid.

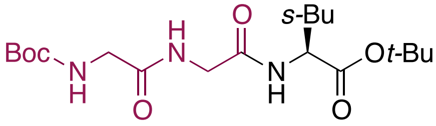
$R_f = 0.52$ (50% AcOEt in hexane). M.p. 154–155 °C. $[\alpha]_D^{20} = -55.9$ (*c* 1.11, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 4.60 (dd, $J = 9.5, 6.3$ Hz, 1H, $C\underline{H}CH_2SC(CH_3)_2$), 4.59 (d, $J = 16.5$ Hz, 1H, $C\underline{H}_2$), 4.08 (d, $J = 16.5$ Hz, 1H, $C\underline{H}_2$), 4.11 (d, $J = 16.5$ Hz, 1H, $C\underline{H}_2$), 3.43 (d, $J = 12.5, 9.5$ Hz, 1H, $\mathrm{CHC}\underline{H}_2\mathrm{SC(CH_3)_2}$), 3.31 (d, *J* = 12.5, 6.3 Hz, 1H, $\mathrm{CHC}\underline{H}_2\mathrm{SC(CH_3)_2}$), 1.90 (s, 3H, $\mathrm{CHCH_2SC(C}\underline{H}_3\mathrm{)_2}$), 1.84 (s, 3H, $\mathrm{CHCH_2SC(C}\underline{H}_3\mathrm{)_2}$), 1.55 (s, 9H, $\mathrm{(C}\underline{H}_3\mathrm{)_3COCON}$). $^{13}$C NMR (100 MHz, $\mathrm{CDCl_3}$) δ 165.9, 162.0, 149.7, 85.0, 73.2, 65.4, 50.2, 30.1, 29.2, 27.9, 27.8 (3C). IR (thin film, cm$^{-1}$) 2980, 1768, 1723, 1667, 1415, 1370, 1312, 1282, 1236, 1143, 1071. HRMS (ESI) calculated for $\mathrm{C_{13}H_{20}N_2O_4SNa}$ $[\mathrm{M+Na}]^+$ *m/z* 323.1042, found, 323.1049.

## 9. Properties of peptides 6a–k (Scheme 3)



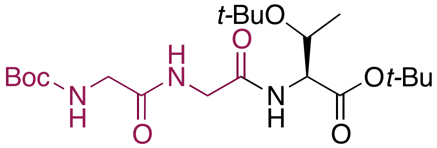
**Boc-Gly-Gly-L-Ala-O*t*-Bu (6aa):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-Gly-) (**5a**, 107.1 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (AcOEt → 10% MeOH in $CHCl_3$) to provide the title compound **6aa** (163.5 mg, 91% yield) as a white solid with >99:1 er.

$R_f$ = 0.30 (10% MeOH in $CHCl_3$). M.p. 30–31 °C. $[\alpha]_D^{23}$ = –23.5 (*c* 1.02, MeOH). $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.16 (br t, *J* = 5.4 Hz, 1H, N*H*), 6.95 (br d, *J* = 7.3 Hz, 1H, N*H*), 5.50 (br t, *J* = 5.4 Hz, 1H, $(CH_3)_3COCONH$), 4.43 (quin, *J* = 7.3 Hz, 1H, C*H*$CH_3$), 4.04 (dd, *J* = 16.7, 5.4 Hz, 1H, C$H_2$), 3.97 (dd, *J* = 16.7, 5.4 Hz, 1H, C$H_2$), 3.85 (br d, *J* = 5.4 Hz, 2H, C$H_2$), 1.46 (s, 9H, $CO_2C(CH_3)_3$), 1.45 (s, 9H, $(CH_3)_3COCONH$), 1.38 (d, *J* = 7.3 Hz, 3H, CHC$H_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 171.9, 170.1, 168.2, 156.1, 82.0, 80.2, 48.7, 44.2, 42.8, 28.3 (3C), 27.9 (3C), 18.1. IR (thin film, $cm^{-1}$) 3309, 2979, 2934, 1731, 1652, 1519, 1454, 1367, 1247, 1146, 1051. HRMS (ESI) calculated for $C_{16}H_{29}N_3O_6Na$ $[M+Na]^+$ *m/z* 382.1954, found, 382.1963.



**Boc-Gly-Gly-L-Ile-O*t*-Bu (6ab):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-Gly-) (**5a**, 107.1 mg, 0.50 mmol) and H-L-Ile-O*t*-Bu (187.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ 12 mL). The reaction mixture was purified directly by flash column chromatography (50–100% AcOEt/hexane → 20% MeOH in $CHCl_3$) to provide the title compound **6ab** (186.7 mg, 93% yield) as a white solid with >99:1 dr.

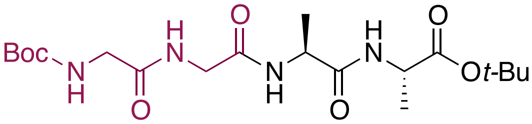
$R_f = 0.68$ (AcOEt). M.p. 28–29 °C. $[\alpha]_D^{27} = -41.5$ (*c* 1.06, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.08 (br t, *J* = 5.3 Hz, 1H, N*H*), 6.81 (br d, *J* = 8.5 Hz, 1H, N*H*), 5.41 (br t, *J* = 5.5 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.47 (dd, *J* = 8.5, 4.6 Hz, 1H, $C\underline{H}CH(CH_3)CH_2CH_3$), 4.04 (dd, *J* = 16.7, 5.3 Hz, 1H, $C\underline{H}_2$), 3.99 (dd, *J* = 16.7, 5.3 Hz, 1H, $C\underline{H}_2$), 3.86 (br d, *J* = 5.5 Hz, 2H, $C\underline{H}_2$), 1.95–1.80 ($CHC\underline{H}(CH_3)CH_2CH_3$), 1.60–1.35 (m, 1H, $CHCH(CH_3)C\underline{H}_2CH_3$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.30–1.10 (m, 1H, $CHCH(CH_3)C\underline{H}_2CH_3$), 0.92 (t, *J* = 7.5 Hz, 3H, $CHCH(CH_3)CH_2C\underline{H}_3$), 0.90 (d, *J* = 6.9 Hz, 3H, $CHCH(C\underline{H}_3)CH_2CH_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 170.7, 170.0, 168.3, 156.0, 82.1, 80.2, 56.9, 44.1, 42.9, 38.0, 28.3 (3C), 28.0 (3C), 25.2, 15.3, 11.7. IR (thin film, cm$^{-1}$) 3304, 2973, 2934, 1731, 1652, 1518, 1456, 1366, 1247, 1143, 1049. HRMS (ESI) calculated for $C_{19}H_{35}N_3O_6Na$ $[M+Na]^+$ *m/z* 424.2424, found, 424.2403.



**Boc-Gly-Gly-L-Thr(*t*-Bu)-O*t*-Bu (6ac):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-Gly-) (**5a**,107.1 mg, 0.50 mmol) and H-L-Thr(*t*-Bu)-O*t*-Bu (200.6 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 60 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (1–10% MeOH in $CHCl_3$) to provide a mixture of the title compound **6ac** and H-L-Thr(*t*-Bu)-O*t*-Bu. Finally, H-L-Thr(*t*-Bu)-O*t*-Bu was evaporated *in vacuo* with the aid of an oil difusion pump to provide **6ac** (200.6 mg, 90% yield) as a white solid with >99:1 dr.

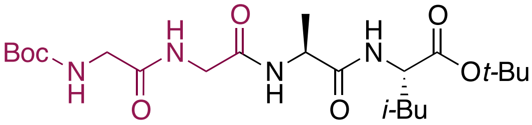
$R_f = 0.59$ (AcOEt). M.p. 31–32 °C. $[\alpha]_D^{28} = +2.9$ (*c* 1.03, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.95 (br dd, *J* = 5.3, 5.0 Hz, 1H, N*H*), 6.53 (br d, *J* = 9.2 Hz, 1H, N*H*), 5.31 (br t, *J* = 5.3 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.36 (dd, *J* = 9.2, 2.1 Hz, 1H, $C\underline{H}CH(CH_3)OC(CH_3)_3$), 4.20 (qd, *J* = 6.2, 2.1 Hz, 1H, $CHC\underline{H}(CH_3)OC(CH_3)_3$), 4.09 (dd, *J* = 16.8, 5.3 Hz, 1H, $C\underline{H}_2$), 4.30 (dd, *J* = 16.8, 5.0 Hz, 1H, $C\underline{H}_2$), 3.87 (br d, *J* = 5.3 Hz, 2H, $C\underline{H}_2$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.16 (s, 9H, $CHCH(CH_3)OC(C\underline{H}_3)_3$), 1.16 (d, *J* = 6.2 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C

NMR (100 MHz, $CDCl_3$) δ 169.7, 169.5, 168.7, 155.9, 82.1, 80.1, 73.9, 67.0, 58.4, 44.0, 42.6, 28.6 (3C), 28.2 (3C), 28.0 (3C), 20.9. IR (thin film, $cm^{-1}$) 3340, 2977, 2932, 1723, 1660, 1515, 1506, 1366, 1244, 1147, 1082. HRMS (ESI) calculated for $C_{21}H_{39}N_3O_7Na$ $[M+Na]^+$ *m/z* 468.2686, found, 468.2659.



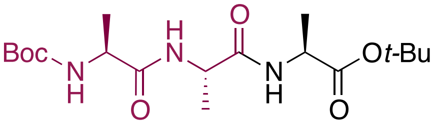
**Boc-Gly-Gly-L-Ala-L-Ala-O*t*-Bu (6ad):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Nd) was charged with Cyclo(-Boc-Gly-Gly-) (**5a**, 107.1 mg, 0.50 mmol) and H-L-Ala-L-Ala-O*t*-Bu (216.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (80–100% AcOEt/hexane → 10–20% MeOH in $CHCl_3$) to provide the title compound **6ad** (206.7 mg, 96% yield) as a white solid with >99:1 dr.

$R_f = 0.35$ (AcOEt). M.p. 69–70 °C. $[\alpha]_D^{25} = -48.7$ (*c* 1.23, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.75 (br t, *J* = 4.4 Hz, 1H, N*H*), 7.52 (br d, *J* = 7.1 Hz, 1H, N*H*), 7.41 (br d, *J* = 7.4 Hz, 1H, N*H*), 5.79 (br t, *J* = 4.4 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.79 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 4.47 (quin, *J* = 7.4 Hz, 1H, $C\underline{H}CH_3$), 4.14 (dd, *J* = 17.2, 4.4 Hz, 1H, $C\underline{H}_2$), 4.06 (dd, *J* = 17.2, 4.4 Hz, 1H, $C\underline{H}_2$), 3.98 (d, *J* = 4.4 Hz, 2H, $C\underline{H}_2$), 1.45 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.40 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$), 1.37 (d, *J* = 7.4 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.9, 171.7, 169.8, 168.3, 156.1, 81.7, 79.8, 48.8, 48.6, 43.9, 43.0, 28.3 (3C), 27.9 (3C), 19.1, 18.0. IR (thin film, $cm^{-1}$) 3309, 2979, 1730, 1702, 1667, 1634, 1504, 1447, 1366, 1244, 1151. HRMS (ESI) calculated for $C_{19}H_{34}N_4O_7Na$ $[M+Na]^+$ *m/z* 453.2325, found, 453.2295.



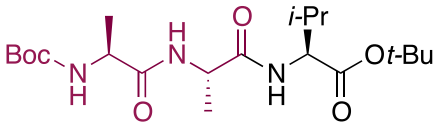
**Boc-Gly-Gly-L-Ala-L-Leu-O*t*-Bu (6ae):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Nd) was charged with Cyclo(-Boc-Gly-Gly-) (**5a**, 107.1 mg, 0.50 mmol) and H-L-Ala-L-Leu-O*t*-Bu (258.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (80–100% AcOEt/hexane → 10% MeOH in $CHCl_3$) to provide the title compound **6ae** (229.2 mg, 97% yield) as a white solid with >99:1 dr.

$R_f$ = 0.50 (AcOEt). M.p. 135–137 °C. $[\alpha]_D^{25}$ = –52.5 (*c* 1.18, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.58 (br t, *J* = 4.8 Hz, 1H, N$\underline{H}$), 7.19 (br d, *J* = 7.1 Hz, 1H, N$\underline{H}$), 7.09 (br d, *J* = 8.6 Hz, 1H, N$\underline{H}$), 5.62 (br t, *J* = 4.4 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.73 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 4.45 (td, *J* = 8.6, 5.1 Hz, 1H, $C\underline{H}CH_2CH(CH_3)_2$), 4.05 (d, *J* = 4.8 Hz, 2H, $C\underline{H}_2$), 3.96 (dd, *J* = 4.4 Hz, 2H, $C\underline{H}_2$), 1.75–1.40 (m, 3H, $CHC\underline{H}_2C\underline{H}(CH_3)_2$), 1.45 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.40 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$), 0.92 (d, *J* = 6.0 Hz, 3H, $CHCH_2CH(C\underline{H}_3)_2$), 0.91 (d, *J* = 6.0 Hz, 3H, $CHCH_2CH(C\underline{H}_3)_2$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.0, 171.7, 169.8, 168.2, 156.1, 81.7, 80.0, 51.5, 48.8, 44.0, 42.9, 41.1, 28.3 (3C), 27.9 (3C), 24.8, 22.7, 21.9, 18.9. IR (thin film, cm$^{-1}$) 3297, 2974, 1727, 1702, 1667, 1637, 1505, 1453, 1366, 1247, 1149. HRMS (ESI) calculated for $C_{22}H_{40}N_4O_7Na$ $[M+Na]^+$ *m/z* 495.2795, found, 495.2768.



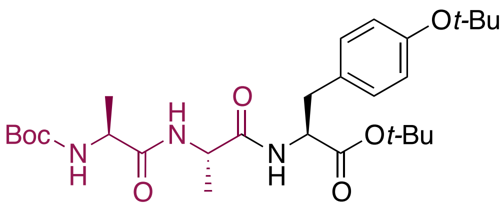
**Boc-L-Ala-L-Ala-L-Ala-O*t*-Bu (6ba, CAS No. 2249882-15-1)$^6$:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-L-Ala-) (**5b**, 121.1 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 30 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (1–10% MeOH in $CHCl_3$) to provide a mixture of the title compound **6ba** and H-L-Ala-O*t*-Bu. Finally, H-L-Ala-O*t*-Bu was evaporated *in vacuo* with the aid of an oil rotary vacuum pump to provide **6ba** (158.9 mg, 82% yield) as a white solid with >99:1 dr.

$R_f$ = 0.68 (AcOEt). M.p. 180–182 °C. $^1H$ NMR (400 MHz, $CDCl_3$) δ 6.89 (br d, $J$ = 6.8 Hz, 1H, $N\underline{H}$), 6.87 (br d, $J$ = 7.1 Hz, 1H, $N\underline{H}$), 5.20 (br d, $J$ = 7.1 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.53 (quin, $J$ = 6.8 Hz, 1H, $C\underline{H}CH_3$), 4.42 (quin, $J$ = 7.1 Hz, 1H, $C\underline{H}CH_3$), 4.20 (br quin, 1H, $C\underline{H}CH_3$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.39 (d, $J$ = 6.8 Hz, 3H, $CHC\underline{H}_3$), 1.36 (d, $J$ = 7.1 Hz, 3H, $CHC\underline{H}_3$), 1.36 (d, $J$ = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 172.5, 171.8, 171.4, 155.5, 81.9, 80.1, 50.1, 48.7, 48.7, 28.3 (3C), 27.9 (3C), 18.5 (2C), 18.3. IR (thin film, $cm^{-1}$) 3309, 2977, 1737, 1674, 1637, 1516, 1454, 1368, 1251, 1151, 1050.



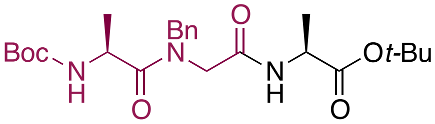
**Boc-L-Ala-L-Ala-L-Val-O*t*-Bu (6bb, CAS No. 2249882-18-4)[6]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-L-Ala-) (**5b**, 121.1 mg, 0.50 mmol) and H-L-Val-O*t*-Bu (173.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–100% AcOEt in hexane) to provide the title compound **6bb** (191.1 mg, 92% yield) as a white solid with >99:1 dr.

$R_f$ = 0.63 (80% AcOEt in hexane). M.p. 73–75 °C. $^1H$ NMR (400 MHz, $CDCl_3$) δ 6.93 (br d, $J$ = 7.1 Hz, 1H, $N\underline{H}$), 6.86 (br d, $J$ = 8.7 Hz, 1H, $N\underline{H}$), 5.26 (br d, $J$ = 7.1 Hz, 1H, $(C\underline{H}_3)_3COCON\underline{H}$), 4.58 (quin, $J$ = 7.1 Hz, 1H, $C\underline{H}CH_3$), 4.40 (dd, $J$ = 8.7, 4.6 Hz, 1H, $C\underline{H}CH(CH_3)_2$), 4.22 (br quin, $J$ = 7.1 Hz, 1H, $C\underline{H}CH_3$), 2.25–2.10 (m, 1H, $CHC\underline{H}(CH_3)_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.39 (d, $J$ = 7.1 Hz, 3H, $CHC\underline{H}_3$), 1.35 (d, $J$ = 7.1 Hz, 3H, $CHC\underline{H}_3$), 0.91 (dd, $J$ = 7.1 Hz, 3H, $CHCH(C\underline{H}_3)_2$), 0.90 (dd, $J$ = 7.1 Hz, 3H, $CHCH(C\underline{H}_3)_2$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 172.6, 171.8, 170.6, 155.4, 81.9, 80.0, 57.5, 50.0, 48.8, 31.2, 28.3 (3C), 28.0 (3C), 18.8, 18.5, 18.1, 17.5. IR (thin film, $cm^{-1}$) 3281, 2977, 1706, 1641, 1505, 1453, 1391, 1366, 1312, 1225, 1151.



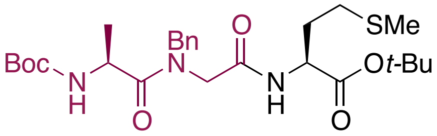
**Boc-L-Ala-L-Ala-L-Tyr(*t*-Bu)-O*t*-Bu (6bc):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-L-Ala-) (**5b**, 121.1 mg, 0.50 mmol) and H-L-Tyr(*t*-Bu)-O*t*-Bu (205.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **6bc** (222.2 mg, 83% yield) as a white solid with >99:1 dr.

$R_f$ = 0.57 (80% AcOEt in hexane). M.p. 60–62 °C. $[\alpha]_D^{22}$ = –23.4 (*c* 1.07, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.10–7.00 (m, 2H, $CHCH_2C_6\underline{H}_4OC(CH_3)_3$), 6.95–6.85 (m, 2H, $CHCH_2C_6\underline{H}_4OC(CH_3)_3$), 6.82 (br d, *J* = 7.1 Hz, 1H, N$\underline{H}$), 6.67 (br d, *J* = 6.1 Hz, 1H, N$\underline{H}$), 5.15 (br d, *J* = 7.1 Hz, 1H, $(CH_3)_3COCON\underline{H}$), 4.75–4.60 (m, 1H, $C\underline{H}CH_3$), 4.48 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 4.25–4.10 (m, 1H, $C\underline{H}CH_2C_6H_4OC(CH_3)_3$), 3.05 (dd, *J* = 13.9, 5.8 Hz, 1H, $CHC\underline{H}_2C_6H_4OC(CH_3)_3$), 3.00 (dd, *J* = 13.9, 6.3 Hz, 1H, $CHC\underline{H}_2C_6H_4OC(CH_3)_3$), 1.45 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.37 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.34 (d, *J* = 7.1 Hz, 6H, $CHC\underline{H}_3$ and $CHC\underline{H}_3$), 1.32 (s, 9H, $CHCH_2C_6H_4OC(C\underline{H}_3)_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.4, 171.3, 170.2, 155.5, 154.3, 130.9, 129.9 (2C), 124.0 (2C), 82.3, 80.1, 78.3, 53.7, 50.0, 48.8, 37.4, 28.8 (3C), 28.3 (3C), 27.9 (3C), 18.3 (2C). IR (thin film, cm$^{-1}$) 3286, 2977, 2931, 1704, 1637, 1504, 1446, 1364, 1234, 1152, 1047. HRMS (ESI) calculated for $C_{28}H_{45}N_3O_7Na$ $[M+Na]^+$ *m/z* 558.3155, found, 558.3136.



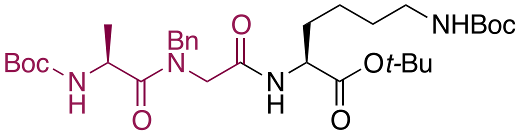
**Boc-L-Ala-Bn-Gly-L-Ala-O*t*-Bu (6ca):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-Bn-Gly-) (**5c**, 159.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–80% AcOEt in hexane) to provide the title compound **6ca** (220.2 mg, 95% yield, ratio of rotamers = 60:40) as a white solid with >99:1 dr.

$R_f$ = 0.55 (50% AcOEt in hexane). M.p. 38–40 °C. $[\alpha]_D^{24}$ = –13.0 (*c* 1.00, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.45 (br d, *J* = 7.2 Hz, 1H x 40/100, $N\underline{H}$), 7.40–7.15 (m, 5H, $C_6\underline{H}_5CH_2N$), 6.85 (br d, *J* = 7.2 Hz, 1H x 60/100, $N\underline{H}$), 5.29 (br d, *J* = 7.1 Hz, 1H x 60/100, $(CH_3)_3COCON\underline{H}$), 5.20 (br d, *J* = 7.1 Hz, 1H x 40/100, $(CH_3)_3COCON\underline{H}$), 4.95–4.20 (m, 4H + 1H x 60/100, $C\underline{H}CH_3$, $C_6H_5C\underline{H}_2N$, $C\underline{H}_2$, and $C\underline{H}CH_3$), 4.03 (d, *J* = 18.1 Hz, 1H x 60/100, $C\underline{H}_2$), 3.98 (d, *J* = 18.1 Hz, 1H x 60/100, $C\underline{H}_2$), 3.84 (d, *J* = 16.3, 1H x 40/100, $C\underline{H}_2$), 1.47 (s, 9H x 60/100, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 18H x 40/100, $(C\underline{H}_3)_3COCONH$ and $CO_2C(C\underline{H}_3)_3$), 1.42 (s, 9H x 60/100, $(C\underline{H}_3)_3COCONH$), 1.36 (d, *J* = 7.2 Hz, 3H x 60/100, $CHC\underline{H}_3$), 1.34 (d, *J* = 7.2 Hz, 3H x 40/100, $CHC\underline{H}_3$), 1.29 (d, *J* = 7.1 Hz, 3H x 40/100, $CHC\underline{H}_3$), 1.24 (d, *J* = 7.1 Hz, 3H x 60/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 174.3 (1C x 60/100), 173.7 (1C x 40/100), 171.8 (1C x 60/100), 171.5 (1C x 40/100), 167.8 (1C x 60/100), 167.6 (1C x 40/100), 156.0 (1C x 40/100), 155.4 (1C x 60/100), 136.4 (1C x 40/100), 135.6 (1C x 60/100), 128.9 (2C x 60/100), 128.6 (2C x 40/100), 128.5 (1C x 60/100), 128.0 (2C x 40/100), 127.7 (1C x 40/100), 126.9 (2C x 60/100), 81.8 (1C x 60/100), 81.5 (1C x 40/100), 80.2 (1C x 60/100), 79.9 (1C x 40/100), 52.0 (1C x 60/100), 50.8 (1C x 40/100), 50.5 (1C x 40/100), 49.7 (1C x 60/100), 48.9 (1C x 40/100), 48.6 (1C x 60/100), 46.9 (1C x 40/100), 46.3 (1C x 60/100), 28.3 (3C x 60/100), 28.2 (3C x 40/100), 27.9 (3C x 60/100), 27.9 (3C x 40/100), 18.3 (1C x 40/100), 18.2 (1C x 60/100), 17.3 (1C x 60/100), 17.1 (1C x 40/100). IR (thin film, $cm^{-1}$) 3292, 2977, 2932, 1732, 1686, 1645, 1520, 1452, 1366, 1222, 1147. HRMS (ESI) calculated for $C_{24}H_{37}N_3O_6Na$ $[M+Na]^+$ *m/z* 486.2580, found, 486.2563.



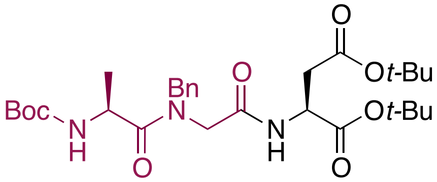
**Boc-L-Ala-Bn-Gly-L-Met-O*t*-Bu (6cb):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-Bn-Gly-) (**5c**, 159.2 mg, 0.50 mmol) and H-L-Met-O*t*-Bu (205.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **6cb** (251.3 mg, 96% yield, ratio of rotamers = 60:40) as a white solid with >99:1 dr.

$R_f$ = 0.36 (50% AcOEt in hexane). M.p. 29–30 °C. $[\alpha]_D^{22}$ = –21.2 (*c* 1.18, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.60 (br d, *J* = 7.8 Hz, 1H x 40/100, $N\underline{H}$), 7.40–7.15 (m, 5H, $C_6\underline{H}_5CH_2N$), 6.88 (br d, *J* = 7.3 Hz, 1H x 60/100, $N\underline{H}$), 5.33 (br d, *J* = 6.6 Hz, 1H x 60/100, $(CH_3)_3COCON\underline{H}$), 5.20 (br d, *J* = 6.9 Hz, 1H x 40/100, $(CH_3)_3COCON\underline{H}$), 4.90–4.40 (m, 4H, $C\underline{H}CH_3$, $C_6H_5C\underline{H}_2N$, and $C\underline{H}CH_2CH_2SCH_3$), 4.22 (d, *J* = 15.8 Hz, 1H x 60/100, $C\underline{H}_2$), 4.02 (s, 1H x 40/100, $C\underline{H}_2$), 4.01 (s, 1H x 40/100, $C\underline{H}_2$), 3.87 (d, *J* = 15.8, 1H x 60/100, $C\underline{H}_2$), 2.55–2.35 (m, 2H, $CHCH_2C\underline{H}_2SCH_3$), 2.20–2.00 (m, 1H, $CHC\underline{H}_2CH_2SCH_3$), 2.09 (s, 3H, $CHCH_2CH_2SC\underline{H}_3$), 2.00–1.85 (m, 1H, $CHC\underline{H}_2CH_2SCH_3$), 1.48 (s, 9H x 60/100, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H x 40/100, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H x 40/100, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H x 60/100, $(C\underline{H}_3)_3COCONH$), 1.35 (d, *J* = 6.9 Hz, 3H x 40/100, $CHC\underline{H}_3$), 1.28 (d, *J* = 6.6 Hz, 3H x 60/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 174.2 (1C x 60/100), 173.6 (1C x 40/100), 170.6 (1C x 60/100), 170.5 (1C x 40/100), 168.2 (1C x 40/100), 168.1 (1C x 60/100), 156.1 (1C x 60/100), 155.3 (1C x 40/100), 136.3 (1C x 40/100), 135.5 (1C x 60/100), 129.0 (2C x 60/100), 128.7 (2C x 40/100), 128.4 (1C x 60/100), 128.0 (2C x 40/100), 127.8 (1C x 40/100), 127.0 (2C x 60/100), 82.3 (1C x 60/100), 81.9 (1C x 40/100), 80.3 (1C x 40/100), 79.9 (1C x 60/100), 52.6 (1C x 40/100), 52.1 (1C x 60/100), 50.4 (1C x 60/100), 50.7 (1C x 40/100), 50.4 (1C x 40/100), 49.8 (1C x 60/100), 46.9 (1C x 60/100), 46.3 (1C x 40/100), 31.9 (1C x 60/100), 30.6 (1C x 40/100), 30.4 (1C x 40/100), 30.0 (1C x 60/100), 28.3 (3C x 60/100), 28.3 (3C x 40/100), 28.0 (3C x 60/100), 27.9 (3C x 40/100), 18.6 (1C x 60/100), 17.3 (1C x 40/100), 15.5 (1C x 60/100), 15.4 (1C x 40/100). IR (thin film, cm$^{-1}$) 3207, 2979, 2931, 1682, 1651, 1645, 1518, 1447, 1366, 1226, 1151. HRMS (ESI) calculated for $C_{26}H_{41}N_3O_6SNa$ $[M+Na]^+$ *m/z* 546.2614, found, 546.2622.



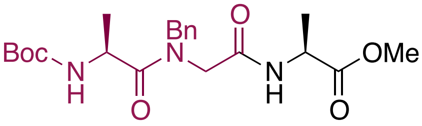
**Boc-L-Ala-Bn-Gly-L-Lys(Boc)-O*t*-Bu (6cc):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-Bn-Gly-) (**5c**, 159.2 mg, 0.50 mmol) and H-L-Lys(Boc)-O*t*-Bu (302.4 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **6cc** (304.1 mg, 98% yield, ratio of rotamers = 60:40) as a white solid with >99:1 dr.

$R_f$ = 0.29 (50% AcOEt in hexane). M.p. 44–45 °C. $[\alpha]_D^{23}$ = –28.0 (*c* 1.18, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.50–7.20 (m, 5H + 1H x 40/100, $C_6\underline{H}_5CH_2N$ and $N\underline{H}$), 6.73 (br d, $J$ = 6.9 Hz, 1H x 60/100, $N\underline{H}$), 5.45 (br d, $J$ = 4.8 Hz, 1H x 60/100, $(CH_3)_3COCON\underline{H}$), 5.26 (br d, $J$ = 7.4 Hz, 1H x 40/100, $(CH_3)_3COCON\underline{H}$), 5.10–4.25 (m, 5H, $C\underline{H}CH_3$, $C_6H_5C\underline{H}_2N$, and $C\underline{H}CH_2CH_2CH_2CH_2N\underline{H}CO_2C(CH_3)_3$), 4.16 (d, $J$ = 15.7 Hz, 1H x 60/100, $C\underline{H}_2$), 4.05 (d, $J$ = 17.9 Hz, 1H x 40/100, $C\underline{H}_2$), 3.98 (d, $J$ = 17.9 Hz, 1H x 40/100, $C\underline{H}_2$), 3.85 (d, $J$ = 15.7 Hz, 1H x 60/100, $C\underline{H}_2$), 3.20–3.00 (m, 2H, $CHCH_2CH_2CH_2C\underline{H}_2NHCO_2C(CH_3)_3$), 1.90–1.70 (m, 1H, $CHC\underline{H}_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.70–1.55 (m, 1H, $CHC\underline{H}_2CH_2CH_2CH_2NHCO_2C(CH_3)_3$), 1.55–1.20 (m, 4H, $CHCH_2C\underline{H}_2C\underline{H}_2CH_2NHCO_2C(CH_3)_3$), 1.46 (s, 9H x 60/100, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H x 40/100, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 18H x 40/100, $(C\underline{H}_3)_3COCONH$ and $CHCH_2CH_2CH_2CH_2NHCO_2C(C\underline{H}_3)_3$), 1.42 (s, 18H x 60/100, $(C\underline{H}_3)_3COCONH$ and $CHCH_2CH_2CH_2CH_2NHCO_2C(C\underline{H}_3)_3$), 1.35 (d, $J$ = 6.9 Hz, 3H x 40/100, $CHC\underline{H}_3$), 1.28 (d, $J$ = 6.9 Hz, 3H x 60/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 174.5 (1C x 40/100), 173.7 (1C x 60/100), 171.0 (1C x 60/100), 170.0 (1C x 40/100), 168.0 (1C x 40/100), 167.8 (1C x 60/100), 156.0 (1C x 60/100), 155.9 (1C x 40/100), 155.3, 136.3 (1C x 40/100), 135.5 (1C x 60/100), 129.0 (2C x 60/100), 128.6 (2C x 40/100), 128.3 (1C x 60/100), 128.0 (2C x 40/100), 127.6 (1C x 40/100), 127.0 (2C x 60/100), 81.9 (1C x 60/100), 81.8 (1C x 40/100), 80.1 (1C x 40/100), 79.8 (1C x 60/100), 79.0, 53.1 (1C x 40/100), 52.3 (1C x 60/100), 52.3 (1C x 60/100), 50.4 (1C x 40/100), 50.3 (1C x 60/100), 50.0 (1C x 40/100), 40.1 (1C x 40/100), 40.0 (1C x 60/100), 31.8 (1C x 60/100), 31.0 (1C x 40/100), 29.5 (1C x 40/100), 29.2 (1C x 60/100), 28.4 (3C x 60/100), 28.3 (3C), 28.2 (3C x 40/100), 28.0 (3C x 60/100), 27.9 (3C x 40/100), 22.8 (1C x 40/100), 22.1 (1C x 60/100), 18.6 (1C x 60/100), 17.6 (1C x 40/100). IR (thin film, cm$^{-1}$) 3321, 2977, 2932, 1682, 1651, 1517, 1454, 1365, 1247, 1156, 1027. HRMS (ESI) calculated for $C_{32}H_{52}N_4O_8Na$ $[M+Na]^+$ *m/z* 643.3683, found, 643.3648.



**Boc-L-Ala-Bn-Gly-L-Asp(*t*-Bu)-O*t*-Bu (6cd):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-Bn-Gly-) (**5c**, 159.2 mg, 0.50 mmol) and H-L-Asp(*t*-Bu)-O*t*-Bu (245.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **6cd** (259.3 mg, 92% yield, ratio of rotamers = 60:40) as a white solid with >99:1 dr.

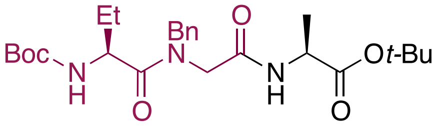
$R_f$ = 0.36 (50% AcOEt in hexane). M.p. 40–41 °C. $[\alpha]_D^{21}$ = –25.2 (*c* 1.11, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.15 (m, 5H + 1H x 40/100, $C_6\underline{H}_5CH_2N$ and $N\underline{H}$), 6.98 (br d, *J* = 8.3 Hz, 1H x 60/100, $N\underline{H}$), 5.51 (br d, *J* = 6.6 Hz, 1H x 60/100, $(CH_3)_3COCON\underline{H}$), 5.31 (br d, *J* = 6.8 Hz, 1H x 40/100, $(CH_3)_3COCON\underline{H}$), 4.90–4.40 (m, 4H, $C\underline{H}CH_3$, $C_6H_5C\underline{H}_2N$, and $C\underline{H}CH_2CO_2C(CH_3)_3$), 4.20 (d, *J* = 18.2, 1H x 40/100, $C\underline{H}_2$), 4.03 (d, *J* = 15.9, 1H x 60/100, $C\underline{H}_2$), 3.97 (d, *J* = 15.9, 1H x 60/100, $C\underline{H}_2$), 3.86 (d, *J* = 18.2, 1H x 40/100, $C\underline{H}_2$), 2.85 (dd, *J* = 17.0, 4.6 Hz, 1H x 60/100, $CHC\underline{H}_2CO_2C(CH_3)_3$), 2.82 (dd, *J* = 17.0, 4.6 Hz, 1H x 40/100, $CHC\underline{H}_2CO_2C(CH_3)_3$), 2.71 (dd, *J* = 17.0, 4.6 Hz, 1H x 40/100, $CHC\underline{H}_2CO_2C(CH_3)_3$), 2.69 (dd, *J* = 17.0, 4.6 Hz, 1H x 60/100, $CHC\underline{H}_2CO_2C(CH_3)_3$), 1.46 (s, 18H x 40/100, $CHCH_2CO_2C(C\underline{H}_3)_3$ and $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 18H x 60/100, $CHCH_2CO_2C(C\underline{H}_3)_3$ and $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H x 40/100, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H x 60/100, $(C\underline{H}_3)_3COCONH$), 1.35 (d, *J* = 6.6 Hz, 3H x 60/100, $CHC\underline{H}_3$), 1.35 (d, *J* = 6.8 Hz, 3H x 40/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 173.9 (1C x 40/100), 173.9 (1C x 60/100), 171.1 (1C x 60/100), 170.0 (1C x 40/100), 169.4 (1C x 60/100), 169.3 (1C x 30/100), 167.8 (1C x 60/100), 167.5 (1C x 40/100), 155.6 (1C x 40/100), 155.1 (1C x 60/100), 136.1 (1C x 40/100), 135.3 (1C x 60/100), 128.6 (2C x 60/100), 128.6 (2C x 40/100), 128.2 (1C x 60/100), 128.0 (2C x 40/100), 127.6 (1C x 40/100), 127.1 (2C x 60/100), 82.3 (1C x 40/100), 82.2 (1C x 60/100), 81.5, 79.9 (1C x 40/100), 79.6 (1C x 60/100), 51.8 (1C x 60/100), 49.9 (1C x 40/100), 49.8 (1C x 40/100), 49.4 (1C x 40/100), 49.0 (1C x 60/100), 49.0 (1C x 60/100), 46.5 (1C x 40/100), 46.4 (1C x 60/100), 37.3 (1C x 60/100), 37.1 (1C x 40/100), 28.3 (3C x 60/100), 28.3 (3C x 40/100), 28.0 (3C), 27.9 (3C), 19.0 (1C x 60/100), 18.2 (1C x 40/100). IR (thin film, cm$^{-1}$) 3311, 2979, 2934, 1731, 1682, 1651, 1515, 1454, 1366, 1246, 1149. HRMS (ESI) calculated for $C_{29}H_{45}N_3O_8Na$ $[M+Na]^+$ *m/z* 586.3104, found, 586.3074.



**Boc-L-Ala-Bn-Gly-L-Ala-OMe (6ce):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-Bn-Gly-) (**5c**, 159.2 mg, 0.50 mmol), H-L-Ala-OMe•HCl (154.7 mg, 1.5 mmol), and $Et_3N$ (0.21 mL, 1.5 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (50–80% AcOEt in hexane) to provide the title compound **6ce** (200.3 mg, 95% yield, ratio of rotamers = 64:36) as a white solid with >99:1 dr.

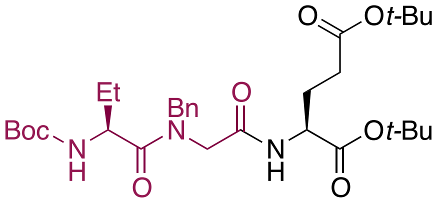
$R_f$ = 0.69 (AcOEt). M.p. 31–33 °C. $[\alpha]_D^{21}$ = –21.8 (*c* 1.01, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.64 (br d, *J* = 7.4 Hz, 1H x 36/100, $N\underline{H}$), 7.40–7.20 (m, 5H, $C_6\underline{H}_5CH_2N$), 6.90 (br d, *J* = 7.4 Hz, 1H x 64/100, $N\underline{H}$), 5.26 (br d, *J* = 6.6 Hz, 1H x 64/100, $(CH_3)_3COCON\underline{H}$), 5.20 (br d, *J* = 6.9 Hz, 1H x 36/100, $(CH_3)_3COCON\underline{H}$), 4.87 (d, *J* = 16.4 Hz, 1H x 36/100, $C\underline{H}_2$), 4.70–4.30 (m, 4H, $C\underline{H}CH_3$, $C_6H_5C\underline{H}_2N$, and $C\underline{H}CH_3$), 4.07 (d, *J* = 17.9 Hz, 1H x 64/100, $C\underline{H}_2$), 3.95 (d, *J* = 17.9 Hz, 1H x 64/100, $C\underline{H}_2$), 3.83 (d, *J* = 16.4 Hz, 1H x 36/100, $C\underline{H}_2$), 3.75 (s, 3H x 64/100, $CO_2C\underline{H}_3$), 3.69 (s, 3H x 36/100, $CO_2C\underline{H}_3$), 1.42 (s, 9H x 64/100, $(C\underline{H}_3)_3COCONH$), 1.40 (s, 9H x 36/100, $(C\underline{H}_3)_3COCONH$), 1.35 (d, *J* = 6.6 Hz, 3H x 64/100, $CHC\underline{H}_3$), 1.31 (d, *J* = 7.3 Hz, 3H x 64/100, $CHC\underline{H}_3$), 1.24 (d, *J* = 6.9 Hz, 6H x 36/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 174.3 (1C x 64/100), 173.6 (1C x 36/100), 173.1 (1C x 64/100), 172.8 (1C x 36/100), 168.1 (1C x 64/100), 167.9 (1C x 36/100), 156.1 (1C x 64/100), 155.5 (1C x 36/100), 136.5 (1C x 36/100), 135.6 (1C x 64/100), 129.0 (2C x 64/100), 128.6 (2C x 36/100), 128.5 (1C x 64/100), 128.0 (2C x 36/100), 127.7 (1C x 36/100), 126.9 (2C x 64/100), 80.2 (1C x 36/100), 79.9 (1C x 64/100), 52.4 (1C x

64/100), 52.2 (1C x 36/100), 51.9 (1C x 64/100), 50.9 (1C x 36/100), 50.6 (1C x 36/100), 49.6 (1C x 64/100), 48.2 (1C x 36/100), 48.0 (1C x 64/100), 47.0 (1C x 64/100), 46.3 (1C x 36/100), 28.3 (3C x 64/100), 28.2 (3C x 36/100), 18.2 (1C x 36/100), 17.9 (1C x 64/100), 17.2 (1C x 64/100), 16.9 (1C x 36/100). IR (thin film, cm$^{-1}$) 3302, 2983, 2930, 1744, 1686, 1643, 1524, 1450, 1366, 1212, 1157. HRMS (ESI) calculated for $C_{21}H_{31}N_3O_6Na$ [M+Na]$^+$ *m/z* 444.2111, found, 444.2069.



**Boc-L-Abu-Bn-Gly-L-Ala-O*t*-Bu (6da):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Abu-Bn-Gly-) (**5d**, 166.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–100% AcOEt in hexane) to provide the title compound **6da** (226.9 mg, 95% yield, ratio of rotamers = 60:40) as a white solid with >99:1 dr.

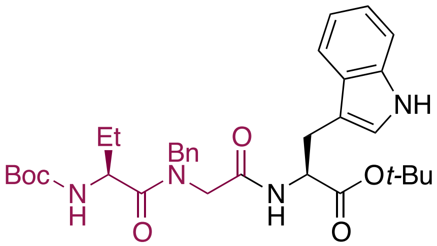
$R_f$ = 0.50 (50% AcOEt in hexane). M.p. 41–42 °C. $[\alpha]_D^{28}$ = –13.9 (*c* 1.01, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.41 (br d, *J* = 7.1 Hz, 1H x 40/100, N$\underline{H}$), 7.40–7.20 (m, 5H, $C_6\underline{H}_5CH_2N$), 6.76 (br d, *J* = 7.1 Hz, 1H x 60/100, N$\underline{H}$), 5.25 (br d, *J* = 7.6 Hz, 1H x 60/100, $(CH_3)_3COCON\underline{H}$), 5.11 (br d, *J* = 7.6 Hz, 1H x 40/100, $(CH_3)_3COCON\underline{H}$), 4.90–4.20 (m, 3H, $C\underline{H}CH_2CH_3$ and $C_6H_5C\underline{H}_2N$), 4.43 (quin, *J* = 7.1 Hz, 1H x 60/100, $C\underline{H}CH_3$), 4.30 (quin, *J* = 7.1 Hz, 1H x 40/100, $C\underline{H}CH_3$), 4.14 (d, *J* = 15.7 Hz, 1H x 40/100, $C\underline{H}_2$), 4.07 (d, *J* = 17.6 Hz, 1H x 60/100, $C\underline{H}_2$), 3.98 (d, *J* = 17.6 Hz, 1H x 60/100, $C\underline{H}_2$), 3.93 (d, *J* = 15.7 Hz, 1H x 40/100, $C\underline{H}_2$), 1.90–1.50 (m, 2H, $CHC\underline{H}_2CH_3$), 1.47 (s, 9H x 60/100, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H x 40/100, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H x 60/100, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H x 40/100, $(C\underline{H}_3)_3COCONH$), 1.36 (d, *J* = 7.1 Hz, 3H x 60/100, $CHC\underline{H}_3$), 1.30 (d, *J* = 7.1 Hz, 3H x 40/100, $CHC\underline{H}_3$), 0.98 (t, *J* = 7.4 Hz, 3H x 40/100, $CHCH_2C\underline{H}_3$), 0.89 (t, *J* = 7.4 Hz, 3H x 60/100, $CHCH_2C\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 173.7 (1C x 60/100), 173.1 (1C x 40/100), 171.8 (1C x 60/100), 171.6 (1C x 40/100), 167.8 (1C x 60/100), 167.6 (1C x 40/100), 156.3 (1C x 40/100), 155.7 (1C x 60/100), 136.5 (1C x 40/100), 135.6 (1C x 60/100), 128.9 (2C x 60/100), 128.6 (2C x 40/100), 128.5 (1C x 60/100), 128.0 (2C x 40/100), 127.6 (1C x 40/100), 127.2 (2C x 60/100), 81.8 (1C x 60/100), 81.5 (1C x 40/100), 80.1 (1C x 60/100), 79.7 (1C x 40/100), 52.2 (1C x 40/100), 52.1 (1C x 60/100), 51.6 (1C x 60/100), 50.7 (1C x 40/100), 50.2 (1C x 40/100), 49.5 (1C x 60/100), 49.0 (1C x 40/100), 48.6 (1C x 60/100), 28.3 (3C x 40/100), 28.2 (3C x 60/100), 27.9 (3C x 60/100), 27.9 (3C x 40/100), 26.0 (1C x 60/100), 25.2 (1C x 40/100), 18.3 (1C x 60/100), 17.3 (1C x 40/100), 10.0 (1C x 40/100), 9.8 (1C x 60/100). IR (thin film, cm$^{-1}$) 3297, 2973, 2936, 1733, 1686, 1640, 1522, 1451, 1366, 1218, 1146. HRMS (ESI) calculated for $C_{25}H_{39}N_3O_6Na$ [M+Na]$^+$ *m/z* 500.2737, found, 500.2750.



**Boc-L-Abu-Bn-Gly-L-Glu(*t*-Bu)-O*t*-Bu (6db):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Abu-Bn-Gly-) (**5d**, 166.2 mg, 0.50 mmol) and H-L-Glu(*t*-Bu)-O*t*-Bu (259.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–40% AcOEt in hexane) to provide the title compound **6db** (245.6 mg, 83% yield, ratio of rotamers = 65:35) as a white solid with >99:1 dr.

$R_f$ = 0.20 (30% AcOEt in hexane). M.p. 28–30 °C. $[\alpha]_D^{27}$ = –21.6 (*c* 1.02, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.54 (br d, *J* = 7.8 Hz, 1H x 35/100, N$\underline{H}$), 7.40–7.20 (m, 5H, $C_6\underline{H}_5CH_2N$), 6.78 (br d, *J* = 8.0 Hz, 1H x 65/100, N$\underline{H}$), 5.32 (br d, *J* = 7.6 Hz, 1H x 65/100, $(CH_3)_3COCON\underline{H}$), 5.14 (br d, *J* = 7.6 Hz, 1H x 35/100, $(CH_3)_3COCON\underline{H}$), 4.85–4.30 (m, 4H, $C\underline{H}CH_2CH_3$, $C_6H_5C\underline{H}_2N$, and $C\underline{H}CH_2CH_2CO_2C(CH_3)_3$), 4.06 (d, *J* = 15.6 Hz, 1H x 35/100, $C\underline{H}_2$), 4.03 (s, 2H x 65/100, $C\underline{H}_2$), 3.95 (d, *J* = 15.6 Hz, 1H x 35/100, $C\underline{H}_2$), 2.40–2.00 (m, 3H, $CHC\underline{H}_2C\underline{H}_2CO_2C(CH_3)_3$), 2.00–1.55 (m, 3H, $CHC\underline{H}_2CH_3$ and $CHC\underline{H}_2CH_2CO_2C(CH_3)_3$), 1.47 (s, 9H x 65/100, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H x 65/100, $CHCH_2CH_2CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H x 35/100, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H x 35/100,

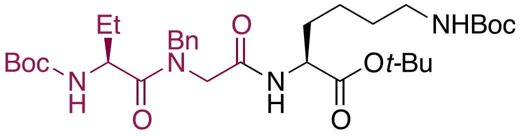
$\mathrm{CHCH_2CH_2CO_2C(C}\underline{H}_3)_3)$, 0.98 (t, $J$ = 7.5 Hz, 3H x 35/100, $\mathrm{CHCH_2C}\underline{H}_3$), 0.90 (t, $J$ = 7.5 Hz, 3H x 65/100, $\mathrm{CHCH_2C}\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 173.6 (1C x 65/100), 173.0 (1C x 35/100), 172.0 (1C x 65/100), 172.0 (1C x 35/100), 170.7 (1C x 65/100), 170.5 (1C x 35/100), 168.2 (1C x 35/100), 168.2 (1C x 65/100), 156.4 (1C x 35/100), 155.5 (1C x 65/100), 136.4 (1C x 35/100), 135.5 (1C x 65/100), 128.9 (2C x 65/100), 128.7 (2C x 35/100), 128.4 (1C x 65/100), 128.0 (2C x 35/100), 127.7 (1C x 35/100), 127.3 (2C x 65/100), 82.2 (1C x 65/100), 81.9 (1C x 35/100), 80.6 (1C x 35/100), 80.6 (1C x 35/100), 80.2 (1C x 35/100), 79.7 (1C x 65/100), 52.8 (1C x 35/100), 52.2 (1C x 35/100), 52.1 (1C x 65/100), 52.1 (1C x 65/100), 51.6 (1C x 65/100), 50.5 (1C x 65/100), 5fD-0.1 (1C x 35/100), 49.5 (1C x 65/100), 31.8 (1C x 35/100), 31.4 (1C x 65/100), 28.3 (3C), 28.0 (3C), 28.0 (3C), 27.7 (1C x 65/100), 26.5 (1C x 35/100), 26.2 (1C x 65/100), 25.2 (1C x 35/100), 10.0 (1C x 35/100), 9.8 (1C x 65/100). IR (thin film, cm$^{-1}$) 3202, 2975, 2931, 1730, 1693, 1645, 1518, 1453, 1366, 1246, 1148. HRMS (ESI) calculated for $C_{31}H_{49}N_3O_8Na$ $[M+Na]^+$ *m/z* 614.3417, found, 614.3405.



**Boc-L-Abu-Bn-Gly-L-Trp-O*t*-Bu (6dc):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Nd) was charged with Cyclo(-Boc-L-Abu-Bn-Gly-) (**5d**, 166.2 mg, 0.50 mmol) and H-L-Trp-O*t*-Bu (260.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **6dc** (272.7 mg, 92% yield, ratio of rotamers = 60:40) as a white solid with >99:1 dr.

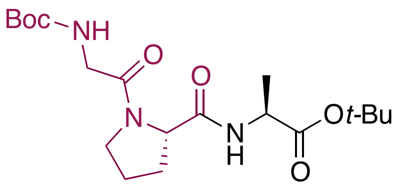
$R_f$ = 0.47 (50% AcOEt in hexane). M.p. 60–62 °C. $[\alpha]_D^{26}$ = +3.6 (*c* 1.12, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.41 (br s, 1H x 60/100, $\mathrm{CHCH_2CCHN}\underline{H}\mathrm{CCHCHCHCHC}$), 8.37 (br s, 1H x 40/100, $\mathrm{CHCH_2CCHN}\underline{H}\mathrm{CCHCHCHCHC}$), 7.62 (br d, $J$ = 7.8 Hz, 1H x 40/100, $\mathrm{N}\underline{H}$), 7.58 (br d, $J$ = 7.8 Hz, 1H x 60/100, $\mathrm{N}\underline{H}$), 7.40–6.90 (m, 9H + 1H x 40/100, $\mathrm{C_6}\underline{H}_5\mathrm{CH_2N}$ and

$CHCH_2CC\underline{H}NHCC\underline{H}C\underline{H}C\underline{H}C\underline{H}C$), 6.60 (br d, $J$ = 7.8 Hz, 1H x 60/100, $CHCH_2CC\underline{H}NHCCHCHCHCHC$), 5.34 (br d, $J$ = 8.0 Hz, 1H x 60/100, $(CH_3)_3COCON\underline{H}$), 5.23 (br d, $J$ = 7.3 Hz, 1H x 40/100, $(CH_3)_3COCON\underline{H}$), 4.85–3.60 (m, 6H, $C\underline{H}CH_2CH_3$, $C_6H_5C\underline{H}_2N$, and $C\underline{H}_2$), 3.41 (d, $J$ = 14.7, 5.0 Hz, 1H x 40/100, $CHC\underline{H}_2CCHNHCCHCHCHCHC$), 3.30 (d, $J$ = 14.7, 5.5 Hz, 1H x 60/100, $CHC\underline{H}_2CCHNHCCHCHCHCHC$), 3.24 (d, $J$ = 14.7, 6.4 Hz, 1H x 60/100, $CHC\underline{H}_2CCHNHCCHCHCHCHC$), 3.18 (d, $J$ = 14.7, 8.9 Hz, 1H x 40/100, $CHC\underline{H}_2CCHNHCCHCHCHCHC$), 1.80–1.30 (m, 2H, $CHC\underline{H}_2CH_3$), 1.47 (s, 9H x 40/100, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H x 60/100, $CO_2C(C\underline{H}_3)_3$), 1.41 (s, 9H x 40/100, $(C\underline{H}_3)_3COCONH$), 1.37 (s, 9H x 60/100, $(C\underline{H}_3)_3COCONH$), 0.90 (t, $J$ = 7.3 Hz, 3H x 40/100, $CHCH_2C\underline{H}_3$), 0.84 (t, $J$ = 7.3 Hz, 3H x 60/100, $CHCH_2C\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 173.5 (1C x 60/100), 172.9 (1C x 40/100), 170.8 (1C x 60/100), 170.6 (1C x 40/100), 167.8 (1C x 60/100), 167.6 (1C x 40/100), 156.2 (1C x 40/100), 155.5 (1C x 60/100), 136.3 (1C x 40/100), 136.1 (1C x 40/100), 136.0 (1C x 60/100), 135.4 (1C x 60/100), 128.9 (2C x 60/100), 128.5 (2C x 40/100), 128.2 (1C x 60/100), 128.0 (2C x 40/100), 127.8 (1C x 40/100), 127.6 (1C x 40/100), 127.5 (1C x 60/100), 127.1 (2C x 60/100), 122.9, 122.1 (1C x 40/100), 122.0 (1C x 60/100), 119.6 (1C x 40/100), 119.4 (1C x 60/100), 118.9 (1C x 60/100), 118.7 (1C x 40/100), 111.2 (1C x 40/100), 111.1 (1C x 60/100), 110.8 (1C x 40/100), 110.1 (1C x 60/100), 82.1 (1C x 60/100), 81.8 (1C x 40/100), 80.1 (1C x 60/100), 79.7 (1C x 40/100), 53.7 (1C x 40/100), 53.1 (1C x 40/100), 52.1 (1C x 40/100), 51.8 (1C x 60/100), 51.5 (1C x 40/100), 49.8 (1C x 60/100), 49.2 (1C x 40/100), 49.1 (1C x 60/100), 28.4 (3C x 40/100), 28.3 (3C x 60/100), 27.9 (3C x 40/100), 27.9 (3C x 60/100), 27.2 (1C x 40/100), 27.1 (1C x 60/100), 26.3 (1C x 60/100), 25.3 (1C x 40/100), 9.9 (1C x 40/100), 9.7 (1C x 60/100). IR (thin film, cm$^{-1}$) 3299, 2973, 2931, 1730, 1693, 1640, 1509, 1453, 1365, 1240, 1152. HRMS (ESI) calculated for $C_{33}H_{44}N_4O_6Na$ $[M+Na]^+$ *m/z* 615.3159, found, 615.3139.



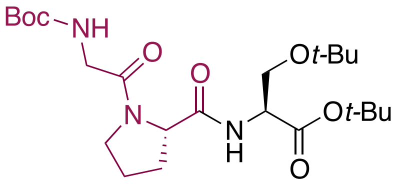
**Boc-L-Abu-Bn-Gly-L-Lys(Boc)-O*t*-Bu (6dd):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Abu-Bn-Gly-) (**5d**, 166.2 mg, 0.50 mmol) and H-L-Lys(Boc)-O*t*-Bu (302.4 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **6dd** (288.7 mg, 91% yield, ratio of rotamers = 67:33) as a white solid with >99:1 dr.

$R_f$ = 0.38 (50% AcOEt in hexane). M.p. 45–46 °C. $[\alpha]_D^{26}$ = –20.4 (*c* 1.08, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40–7.20 (m, 5H + 1H x 33/100, $C_6\underline{H}_5CH_2N$ and $N\underline{H}$), 6.73 (br d, *J* = 6.2 Hz, 1H x 67/100, $N\underline{H}$), 5.45–4.25 (m, 8H, $(CH_3)_3COCON\underline{H}$, $C\underline{H}CH_2CH_3$, $C_6H_5C\underline{H}_2N$, and $C\underline{H}CH_2CH_2CH_2CH_2N\underline{H}CO_2C(CH_3)_3$), 4.10–3.80 (m, 2H, $C\underline{H}_2$), 3.20–3.00 (m, 2H, $CHCH_2CH_2CH_2C\underline{H}_2NHCO_2C(CH_3)_3$), 1.90–1.25 (m, 8H, $CHC\underline{H}_2CH_3$ and $CHC\underline{H}_2C\underline{H}_2C\underline{H}_2CH_2NHCO_2C(CH_3)_3$), 1.46 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H x 33/100, $CHCH_2CH_2CH_2CH_2NHCO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H x 67/100, $CHCH_2CH_2CH_2CH_2NHCO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H x 33/100, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H x 67/100, $(C\underline{H}_3)_3COCONH$), 0.97 (t, *J* = 7.3 Hz, 3H x 33/100, $CHCH_2C\underline{H}_3$), 0.89 (t, *J* = 7.3 Hz, 3H x 67/100, $CHCH_2C\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 173.8 (1C x 67/100), 173.1 (1C x 33/100), 171.0 (1C x 67/100), 171.0 (1C x 33/100), 167.9 (1C x 33/100), 167.9 (1C x 67/100), 156.2 (1C x 33/100), 156.0 (1C x 67/100), 155.6 (1C x 67/100), 155.5 (1C x 33/100), 136.4 (1C x 33/100), 135.6 (1C x 67/100), 128.9 (2C x 67/100), 128.6 (2C x 33/100), 128.4 (1C x 67/100), 128.0 (2C x 33/100), 127.6 (1C x 33/100), 127.3 (2C x 67/100), 82.0 (1C x 67/100), 81.8 (1C x 33/100), 80.0 (1C x 33/100), 79.7 (1C x 67/100), 79.0, 53.1 (1C x 67/100), 52.4, 52.1 (1C x 33/100), 51.6 (1C x 67/100), 50.4 (1C x 33/100), 50.0 (1C x 33/100), 49.8 (1C x 67/100), 40.2 (1C x 33/100), 40.0 (1C x 67/100), 31.8 (1C x 67/100), 31.2 (1C x 33/100), 29.5 (1C x 33/100), 29.3 (1C x 67/100), 28.4 (3C x 67/100), 28.4 (3C x 33/100), 28.3 (3C x 67/100), 28.2 (3C x 33/100), 28.0 (3C), 26.1 (1C x 67/100), 25.4 (1C x 33/100), 22.8 (1C x 33/100), 22.1 (1C x 67/100), 10.0 (1C x 33/100), 9.8 (1C x 67/100). IR (thin film, $cm^{-1}$) 3335, 2977, 2934, 1687, 1646, 1517, 1453, 1366, 1243, 1156, 1079. HRMS (ESI) calculated for $C_{33}H_{54}N_4O_8Na$ $[M+Na]^+$ *m/z* 657.3839, found, 657.3842.

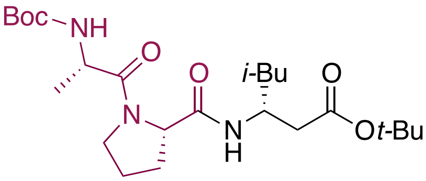


**Boc-Gly-L-Pro-L-Ala-O*t*-Bu (6ea):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-L-Pro-) (**5e**, 127.1 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (1–10% MeOH in $CHCl_3$) to provide the title compound **6ea** (193.8 mg, 97% yield, ratio of rotamers = 90:10) as a white solid with >99:1 dr.

$R_f$ = 0.42 (10% MeOH in $CHCl_3$). M.p. 180–183 °C. $[\alpha]_D^{27}$ = –130.5 (*c* 1.05, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.11 (br d, *J* = 7.3 Hz, 1H x 90/100, N$\underline{H}$), 6.78 (br d, *J* = 7.3 Hz, 1H x 10/100, N$\underline{H}$), 5.48 (br dd, *J* = 4.8, 4.1 Hz, 1H x 90/100, $(CH_3)_3COCON\underline{H}$), 5.28 (br dd, *J* = 4.8, 4.1 Hz, 1H x 10/100, $(CH_3)_3COCON\underline{H}$), 4.55 (dd, *J* = 8.1, 2.2 Hz, 1H, $C\underline{H}CH_2CH_2CH_2$), 4.44 (quin, *J* = 7.3 Hz, 1H x 10/100, $C\underline{H}CH_3$), 4.40 (quin, *J* = 7.3 Hz, 1H x 90/100, $C\underline{H}CH_3$), 4.00 (dd, *J* = 17.3, 4.8 Hz, 1H, $C\underline{H}_2$), 3.91 (dd, *J* = 17.3, 4.1 Hz, 1H, $C\underline{H}_2$), 3.80–3.30 (m, 2H, $CHCH_2CH_2C\underline{H}_2$), 2.40–1.85 (m, 4H, $CHC\underline{H}_2C\underline{H}_2CH_2$), 1.46 (s, 9H x 90/100, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H x 10/100, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H x 90/100, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H x 10/100, $(C\underline{H}_3)_3COCONH$), 1.39 (d, *J* = 7.3 Hz, 3H x 10/100, $CHC\underline{H}_3$), 1.36 (d, *J* = 7.3 Hz, 3H x 90/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.9 (1C x 90/100), 171.6 (1C x 10/100), 170.5 (1C x 10/100), 170.2 (1C x 90/100), 168.4 (1C x 10/100), 168.4 (1C x 90/100), 155.9 (1C x 10/100), 155.7 (1C x 90/100), 82.1 (1C x 10/100), 81.7 (1C x 90/100), 79.6, 60.1 (1C x 10/100), 60.0 (1C x 90/100), 48.8 (1C x 90/100), 48.7 (1C x 10/100), 47.1 (1C x 10/100), 46.2 (1C x 90/100), 43.0 (1C x 90/100), 42.9 (1C x 10/100), 31.9 (1C x 10/100), 28.3 (3C), 27.9 (3C), 27.7 (1C x 90/100), 24.8 (1C x 90/100), 22.3 (1C x 10/100), 18.2 (1C x 90/100), 18.0 (1C x 10/100). IR (thin film, cm$^{-1}$) 3351, 2968, 1720, 1703, 1678, 1635, 1531, 1450, 1366, 1220, 1148. HRMS (ESI) calculated for $C_{19}H_{33}N_3O_6Na$ $[M+Na]^+$ *m/z* 422.2267, found, 422.2246.

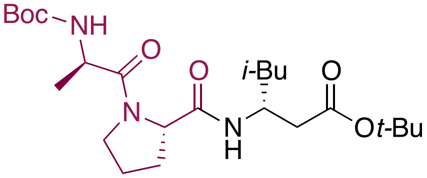


**Boc-Gly-L-Pro-L-Ser(*t*-Bu)-O*t*-Bu (6eb, CAS No. 23828-63-9)[11]:** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-L-Pro-) (**5e**, 127.1 mg, 0.50 mmol) and H-L-Ser(*t*-Bu)-O*t*-Bu (217.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (10–100% AcOEt in hexane) to provide the title compound **6eb** (235.1 mg, >99% yield, ratio of rotamers = 84:16) as a white solid with >99:1 dr. $R_f$ = 0.58 (AcOEt). M.p. 69–70 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.07 (br d, $J$ = 8.1 Hz, 1H x 86/100, $N\underline{H}$), 6.61 (br d, $J$ = 8.5 Hz, 1H x 14/100, $N\underline{H}$), 5.47 (br dd, $J$ = 4.7, 4.1 Hz, 1H x 86/100, $(CH_3)_3COCON\underline{H}$), 5.47–5.35 (m, 1H x 14/100, $(CH_3)_3COCON\underline{H}$), 4.65–4.55 (m, 1H, $C\underline{H}CH_2CH_2CH_2$), 4.53 (dt, $J$ = 8.1, 3.0 Hz, 1H x 86/100, $C\underline{H}CH_2OC(CH_3)_3$), 4.40–4.30 (m, 1H x 14/100, $C\underline{H}CH_2OC(CH_3)_3$), 4.10–3.85 (m, 2H x 14/100, $C\underline{H}_2$), 4.00 (dd, $J$ = 17.2, 4.7 Hz, 1H x 86/100, $C\underline{H}_2$), 3.92 (dd, $J$ = 17.2, 4.1 Hz, 1H x 86/100, $C\underline{H}_2$), 3.85–3.30 (m, 4H x 128/400, $CHCH_2CH_2C\underline{H}_2$ and $CHC\underline{H}_2OC(CH_3)_3$), 3.79 (dd, $J$ = 8.7, 3.0 Hz, 1H x 14/100, $CHC\underline{H}_2OC(CH_3)_3$), 3.76 (dd, $J$ = 8.7, 3.0 Hz, 1H x 86/100, $CHC\underline{H}_2OC(CH_3)_3$), 3.51 (dd, $J$ = 8.7, 3.0 Hz, 1H x 86/100, $CHC\underline{H}_2OC(CH_3)_3$), 3.41 (dt, $J$ = 9.2, 7.1 Hz, 1H x 86/100, $CHCH_2CH_2C\underline{H}_2$), 2.40–1.85 (m, 4H, $CHC\underline{H}_2C\underline{H}_2CH_2$), 1.46 (s, 9H x 86/100, $CO_2C(C\underline{H}_3)_3$), 1.46 (s, 9H x 14/100, $CO_2C(C\underline{H}_3)_3$), 1.44 (s, 9H x 86/100, $(C\underline{H}_3)_3COCONH$), 1.44 (s, 9H x 14/100, $(C\underline{H}_3)_3COCONH$), 1.14 (s, 9H, $CHCH_2OC(C\underline{H}_3)_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 170.5, 169.3 (1C x 86/100), 168.9 (1C x 14/100), 168.6 (1C x 14/100), 168.0 (1C x 86/100), 155.7, 82.2 (1C x 14/100), 81.7 (1C x 86/100), 79.5, 73.2 (1C x 14/100), 73.0 (1C x 86/100), 61.9 (1C x 86/100), 61.9 (1C x 14/100), 60.4 (1C x 14/100), 60.0 (1C x 86/100), 53.3 (1C x 86/100), 53.0 (1C x 14/100), 47.0 (1C x 14/100), 46.1 (1C x 86/100), 43.0, 32.0 (1C x 14/100), 28.3 (3C), 28.0 (1C x 86/100), 28.0 (3C), 27.3 (3C), 24.7 (1C x 86/100), 22.2 (1C x 14/100). IR (thin film, cm$^{-1}$) 3274, 2977, 1749, 1719, 1702, 1665, 1652, 1636, 1546, 1453, 1165.



**Boc-L-Ala-L-Pro-D-β-HoLeu-O*t*-Bu (6f):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-L-Ala-L-Pro-) (**5f**, 134.2 mg, 0.50 mmol) and H-D-β-HoLeu-O*t*-Bu (201.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (50–100% AcOEt in hexane) to provide the title compound **6fa** (225.4 mg, 96% yield, ratio of rotamers = 85:15) as a white solid with >99:1 dr.

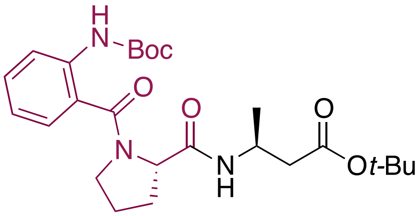
$R_f$ = 0.64 (AcOEt). M.p. 27–28 °C. $[\alpha]_D^{20}$ = –67.9 (*c* 1.09, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.21 (br d, *J* = 8.7 Hz, 1H x 15/100, N*H*), 6.92 (br d, *J* = 9.2 Hz, 1H x 85/100, N*H*), 5.35 (br d, *J* = 6.9 Hz, 1H x 85/100, $(CH_3)_3COCON\underline{H}$), 5.12 (br d, *J* = 6.9 Hz, 1H x 15/100, $(CH_3)_3COCON\underline{H}$), 4.56 (br dd, *J* = 8.0. 2.5 Hz, 1H, $C\underline{H}CH_2CH_2CH_2$), 4.48 (quin, *J* = 6.9 Hz, 1H x 85/100, $C\underline{H}CH_3$), 4.45–4.30 (m, 1H x 15/100, $C\underline{H}CH_3$), 4.30–4.20 (m, 1H x 85/100, $C\underline{H}(CH_2CH(CH_3)_2)CH_2$), 4.15–4.00 (m, 1H x 15/100, $C\underline{H}(CH_2CH(CH_3)_2)CH_2$), 3.70–3.50 (m, 2H, $CHCH_2CH_2C\underline{H}_2$), 2.40–1.80 (m, 4H, $CHC\underline{H}_2C\underline{H}_2CH_2$), 2.34 (d, *J* = 5.5 Hz, 2H, $CH(CH_2CH(CH_3)_2)C\underline{H}_2$), 1.70–1.20 (m, 3H, $CH(C\underline{H}_2C\underline{H}(CH_3)_2)CH_2$), 1.43 (s, 18H, $(C\underline{H}_3)_3COCONH$ and $CO_2C(C\underline{H}_3)_3$), 1.38 (d, *J* = 6.9 Hz, 3H, $CHC\underline{H}_3$), 0.92 (d, *J* = 6.4 Hz, 3H, $CH(CH_2CH(C\underline{H}_3)_2)CH_2$), 0.90 (d, *J* = 6.6 Hz, 3H, $CH(CH_2CH(C\underline{H}_3)_2)CH_2$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 173.0 (1C x 85/100), 172.3 (1C x 15/100), 170.9 (1C x 85/100), 170.6 (1C x 15/100), 170.0 (1C x 85/100), 169.6 (1C x 15/100), 155.5 (1C x 15/100), 155.1 (1C x 85/100), 80.8 (1C x 85/100), 80.7 (1C x 15/100), 79.9 (1C x 15/100) 79.6 (1C x 85/100), 61.0 (1C x 15/100), 60.0 (1C x 85/100), 48.7 (1C x 15/100), 47.7 (1C x 85/100), 47.0 (1C x 85/100), 46.6 (1C x 15/100), 45.2 (1C x 15/100), 44.4 (1C x 85/100), 43.3 (1C x 85/100), 42.8 (1C x 15/100), 40.5, 32.5 (1C x 15/100), 28.3 (3C), 28.0 (3C), 27.5 (1C x 85/100), 25.0 (1C x 15/100), 24.9 (2C x 85/100), 23.0 (1C x 85/100), 22.7 (1C x 15/100), 22.3 (1C x 15/100), 22.1 (1C x 85/100), 21.8 (1C x 15/100), 18.6 (1C x 85/100), 17.3 (1C x 15/100). IR (thin film, cm$^{-1}$) 3341, 2977, 1713, 1694, 1682, 1644, 1519, 1448, 1366, 1249, 1157. HRMS (ESI) calculated for $C_{24}H_{43}N_3O_6Na$ $[M+Na]^+$ *m/z* 492.3050, found, 492.3007.



**Boc-D-Ala-L-Pro-D-β-HoLeu-O*t*-Bu (6g):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-D-Ala-L-Pro-) (**5g**, 134.2 mg, 0.50 mmol) and H-D-β-HoLeu-O*t*-Bu (201.3 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–100% AcOEt in hexane) to provide the title compound **6g** (230.1 mg, 98% yield, ratio of rotamers = 91:9) as a white solid with >99:1 dr.

$R_f = 0.58$ (AcOEt). M.p. 50–51 °C. $[\alpha]_D^{25} = -17.0$ (*c* 1.06, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.03 (br d, *J* = 8.8 Hz, 1H x 91/100, N*H*), 6.68 (br d, *J* = 8.8 Hz, 1H x 9/100, N*H*), 5.53 (br d, *J* = 6.6 Hz, 1H x 91/100, $(CH_3)_3COCONH$), 5.16 (br d, *J* = 6.6 Hz, 1H x 9/100, $(CH_3)_3COCONH$), 4.74 (br dd, *J* = 8.0. 1.4 Hz, 1H x 9/100, $CHCH_2CH_2CH_2$), 4.55 (br dd, *J* = 8.0. 1.4 Hz, 1H x 91/100, $CHCH_2CH_2CH_2$), 4.41 (quin, *J* = 6.6 Hz, 1H, $CHCH_3$), 4.35–4.20 (m, 1H, $CH(CH_2CH(CH_3)_2)CH_2$), 3.72 (ddd, *J* = 9.6, 8.5, 3.0 Hz, 1H, $CHCH_2CH_2CH_2$), 3.70–3.50 (m, 1H x 9/100, $CHCH_2CH_2CH_2$), 3.45 (td, *J* = 9.6, 7.2 Hz, 1H x 91/100, $CHCH_2CH_2CH_2$), 2.50–2.25 (m, 2H x 9/100, $CH(CH_2CH(CH_3)_2)CH_2$), 2.40 (dd, *J* = 14.7, 5.5 Hz, 1H x 91/100, $CH(CH_2CH(CH_3)_2)CH_2$), 2.32 (dd, *J* = 14.7, 6.9 Hz, 1H x 91/100, $CH(CH_2CH(CH_3)_2)CH_2$), 2.20–1.20 (m, 7H, $CHCH_2CH_2CH_2$ and $CH(CH_2CH(CH_3)_2)CH_2$), 1.44 (s, 9H, $CO_2C(CH_3)_3$), 1.43 (s, 9H, $(CH_3)_3COCONH$), 1.32 (d, *J* = 6.6 Hz, 3H, $CHCH_3$), 0.92 (d, *J* = 6.8 Hz, 3H, $CH(CH_2CH(CH_3)_2)CH_2$), 0.91 (d, *J* = 6.8 Hz, 3H, $CH(CH_2CH(CH_3)_2)CH_2$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 172.9 (1C x 9/100), 172.2 (1C x 91/100), 171.0 (1C x 9/100), 170.8 (1C x 91/100), 169.9 (1C x 91/100), 169.8 (1C x 9/100), 155.3 (1C x 91/100), 155.3 (1C x 9/100), 81.2 (1C x 9/100), 80.6 (1C x 91/100), 79.6, 61.0 (1C x 9/100), 60.2 (1C x 91/100), 48.1 (1C x 91/100), 47.5 (1C x 9/100), 46.8, 44.9 (1C x 91/100), 44.5 (1C x 9/100), 43.7 (1C x 91/100), 42.9 (1C x

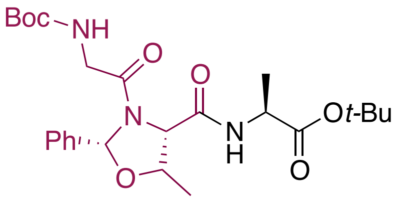
9/100), 41.0 (1C x 91/100), 39.5 (1C x 9/100), 32.1 (1C x 9/100), 28.4 (3C x 91/100), 28.3 (3C x 9/100), 28.0 (3C), 27.6 (1C x 91/100), 25.1 (1C x 9/100), 24.8 (1C x 91/100), 24.5, 22.9 (1C x 91/100), 22.7 (1C x 9/100), 22.4 (1C x 9/100), 22.1 (1C x 91/100), 18.6 (1C x 9/100), 17.6 (1C x 91/100). IR (thin film, $cm^{-1}$) 3334, 2977, 1737, 1681, 1631, 1522, 1452, 1367, 1249, 1159, 748. HRMS (ESI) calculated for $C_{24}H_{43}N_3O_6Na$ $[M+Na]^+$ *m/z* 492.3050, found, 492.3061.



**Boc-Abz-L-Pro-L-β-HoAla-O*t*-Bu (6h):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Abz-L-Pro-) (**5h**, 158.2 mg, 0.50 mmol) and H-L-β-HoAla-O*t*-Bu (159.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at room temperature for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (50–80% AcOEt in hexane) to provide the title compound **6h** (178.3 mg, 75% yield, ratio of rotamers = 56:44) as a white solid with >99:1 dr.

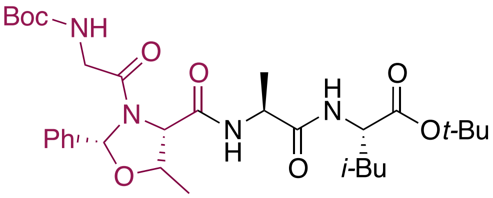
$R_f$ = 0.71 (AcOEt). M.p. 47–48 °C. $[\alpha]_D^{24}$ = +6.0 (*c* 1.00, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.44 (br s, 1H, $(CH_3)_3COCON\underline{H}$), 8.15 (br d, *J* = 8.2 Hz, 1H, $N\underline{H}$), 7.50–6.90 (m, 4H, $CC\underline{H}C\underline{H}C\underline{H}C\underline{H}C$), 4.71 (br t, *J* = 6.6 Hz, 1H x 44/100, $C\underline{H}CH_2CH_2CH_2$), 4.65 (br t, *J* = 7.0 Hz, 1H x 56/100, $C\underline{H}CH_2CH_2CH_2$), 4.40–4.20 (m, 1H, $C\underline{H}(CH_3)CH_2$), 3.70–3.40 (m, 2H, $CH(CH_3)C\underline{H}_2$), 2.50–1.75 (m, 6H, $CHC\underline{H}_2C\underline{H}_2C\underline{H}_2$), 1.50 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H x 44/100, $(C\underline{H}_3)_3COCONH$), 1.43 (s, 9H x 56/100, $(C\underline{H}_3)_3COCONH$), 1.24 (d, *J* = 6.5 Hz, 3H x 56/100, $CH(C\underline{H}_3)CH_2$), 1.23 (d, *J* = 6.5 Hz, 3H x 44/100, $CH(C\underline{H}_3)CH_2$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.0, 170.3 (1C x 56/100), 170.2 (1C x 44/100), 169.8 (1C x 44/100), 169.8 (1C x 56/100), 153.0, 137.4 (1C x 56/100), 137.3 (1C x 44/100), 131.1, 127.7, 123.7 (1C x 44/100), 123.5 (1C x 56/100), 121.7 (1C x 44/100), 121.7 (1C x 56/100), 120.5 (1C x 44/100), 120.4 (1C x 56/100), 81.1, 80.4, 60.3 (1C x 56/100), 60.1 (1C x 44/100), 50.6 (1C x 56/100), 50.5 (1C x 44/100), 42.4, 41.1 (1C x 44/100), 41.1 (1C x 56/100), 28.5 (1C x 56/100), 28.3 (3C), 28.1 (1C x 44/100), 28.0 (3C), 25.4 (1C x 56/100), 25.3 (1C x 44/100), 19.9 (1C x 56/100), 19.9 (1C x

44/100). IR (thin film, cm$^{-1}$) 2981, 2969, 1723, 1666, 1620, 1588, 1522, 1411, 1365, 1235, 1152. HRMS (ESI) calculated for $C_{25}H_{37}N_3O_6Na$ $[M+Na]^+$ *m/z* 498.2580, found, 498.2579.



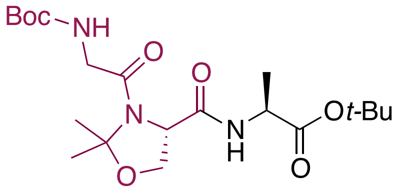
**Boc-Gly-L-Thr($\psi^{Ph,H}$pro)-L-Ala-O*t*-Bu (6ia):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-L-Thr($\psi^{Ph,H}$pro)-) (**5i**, 173.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (20–100% AcOEt in hexane) to provide the title compound **6ia** (243.8 mg, >99% yield, ratio of rotamers = 70:30) as a white solid with >99:1 dr.

$R_f$ = 0.50 (50% AcOEt in hexane). M.p. 60–61 °C. $[\alpha]_D^{24}$ = –17.1 (*c* 1.17, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, *J* = 7.6, 1.6 Hz, 2H, $CHCH(CH_3)OCHC_6\underline{H}_5$), 7.50–7.20 (m, 3H + 1H x 70/100, $CHCH(CH_3)OCHC_6\underline{H}_5$, and $N\underline{H}$), 6.95–6.75 (m, 1H x 30/100, $N\underline{H}$), 6.71 (s, 1H x 30/100, $CHCH(CH_3)OC\underline{H}C_6H_5$), 6.24 (s, 1H x 70/100, $CHCH(CH_3)OC\underline{H}C_6H_5$), 5.50–5.30 (m, 1H, $(CH_3)_3COCON\underline{H}$), 4.80–3.30 (m, 5H, $C\underline{H}_2$, $C\underline{H}C\underline{H}(CH_3)OCHC_6H_5$, and $C\underline{H}CH_3$), 1.70–1.30 (m, 3H + 3H x 70/100, $CHCH(C\underline{H}_3)OCHC_6H_5$ and $CHC\underline{H}_3$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.41 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.10 (br s, 3H x 30/100, $CHCH(C\underline{H}_3)OCHC_6H_5$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.6 (1C x 70/100), 171.2 (1C x 30/100), 169.5 (1C x 30/100), 168.5 (1C x 70/100), 167.8, 156.0 (1C x 30/100), 155.6 (1C x 70/100), 138.2 (1C x 30/100), 136.9 (1C x 70/100), 129.6 (2C x 30/100), 128.9 (2C x 70/100), 128.6, 127.1 (2C x 70/100), 126.3 (2C x 30/100), 89.4 (1C x 70/100), 89.2 (1C x 30/100), 82.0, 80.0 (1C x 30/100), 79.8 (1C x 70/100), 78.4 (1C x 30/100), 74.3 (1C x 70/100), 65.4, 48.9 (1C x 70/100), 48.4 (1C x 30/100), 43.3, 28.2 (3C), 27.9 (3C), 19.2 (1C x 30/100), 18.3 (1C x 70/100), 18.0 (1C x 70/100), 17.3 (1C x 30/100). IR (thin film, cm$^{-1}$) 3330, 2979, 1731, 1662, 1520, 1451, 1367, 1239, 1150, 1047, 845. HRMS (ESI) calculated for $C_{25}H_{37}N_3O_7Na$ $[M+Na]^+$ *m/z* 514.2529, found, 514.2538.



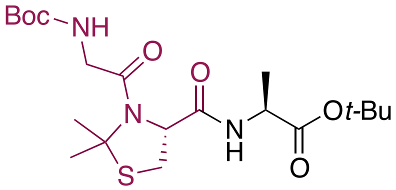
**Boc-Gly-L-Thr($\psi^{Ph,H}$pro)-L-Ala-L-Leu-O*t*-Bu (6ib):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Nd) was charged with Cyclo(-Boc-Gly-L-Thr($\psi^{Ph,H}$pro)-) (**5i**, 173.2 mg, 0.50 mmol) and H-L-Ala-L-Leu-O*t*-Bu (258.4 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (20–100% AcOEt in hexane) to provide the title compound **6ib** (207.0 mg, 68% yield, ratio of rotamers = 70:30) as a white solid with >99:1 dr.

$R_f$ = 0.31 (50% AcOEt in hexane). M.p. 62–63 °C. $[\alpha]_D^{24}$ = –49.2 (*c* 1.24, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.70–7.50 (m, 2H, $CHCH(CH_3)OCHC_6\underline{H}_5$), 7.50–6.50 (m, 5H, $CHCH(CH_3)OCHC_6\underline{H}_5$, $N\underline{H}$, and $N\underline{H}$), 6.95–6.75 (m, 1H x 30/100, $N\underline{H}$), 6.48 (s, 1H x 30/100, $CHCH(CH_3)OC\underline{H}C_6H_5$), 6.26 (s, 1H x 70/100, $CHCH(CH_3)OC\underline{H}C_6H_5$), 5.50–5.30 (m, 1H, $(CH_3)_3COCON\underline{H}$), 4.60–3.40 (m, 6H, $C\underline{H}_2$, $C\underline{H}C\underline{H}(CH_3)OCHC_6H_5$, $C\underline{H}CH_3$, and $C\underline{H}CH_2CH(CH_3)_2$), 2.20–2.05 (m, 1H, $CHC\underline{H}_2CH(CH_3)_2$), 1.70–1.00 (m, 8H, $CHCH(C\underline{H}_3)OCHC_6H_5$, $CHC\underline{H}_3$, and $CHC\underline{H}_2C\underline{H}(CH_3)_2$), 1.45 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.41 (s, 9H, $(C\underline{H}_3)_3COCONH$), 0.91 (d, *J* = 6.1 Hz, 3H, $CHCH_2CH(C\underline{H}_3)_2$), 0.91 (d, *J* = 6.1 Hz, 3H, $CHCH_2CH(C\underline{H}_3)_2$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.8, 171.3, 169.6 (1C x 30/100), 168.6 (1C x 70/100), 168.3 (1C x 30/100), 168.2 (1C x 70/100), 156.3 (1C x 30/100), 155.6 (1C x 70/100), 138.2 (1C x 30/100), 136.9 (1C x 70/100), 129.6, 128.9 (2C x 70/100), 128.6 (2C x 30/100), 127.0 (2C x 70/100), 126.2 (2C x 30/100), 89.4 (1C x 70/100), 89.2 (1C x 30/100), 81.9, 80.1 (1C x 30/100), 79.9 (1C x 70/100), 78.4 (1C x 30/100), 74.5 (1C x 70/100), 65.6 (1C x 70/100), 65.3 (1C x 30/100), 51.4 49.2 (1C x 70/100), 49.0 (1C x 30/100), 43.2, 41.5, 28.2 (3C), 27.9 (3C), 24.8, 22.7, 22.0, 19.1 (1C x 30/100), 17.9 (1C x 70/100), 17.9 (1C x 70/100), 17.4 (1C x 30/100). IR (thin film, $cm^{-1}$) 3322, 2978, 1719, 1649, 1518, 1450, 1366, 1244, 1150, 1050, 843. HRMS (ESI) calculated for $C_{31}H_{48}N_4O_8Na$ $[M+Na]^+$ *m/z* 627.3370, found, 627.3358.



**Boc-Gly-L-Ser($\psi^{Me,Me}$pro)-L-Ala-O*t*-Bu (6j):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-L-Ser($\psi^{Me,Me}$pro)-) (**5j**, 142.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 12 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (30–80% AcOEt in hexane) to provide the title compound **6j** (210.5 mg, 98% yield, ratio of rotamers = 77:23) as a white solid with >99:1 dr.

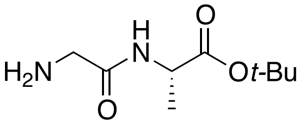
$R_f$ = 0.30 (80% AcOEt in hexane). M.p. 31–32 °C. $[\alpha]_D^{25}$ = –81.2 (*c* 1.01, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.95 (br d, *J* = 7.2 Hz, 1H, $N\underline{H}$), 5.45–5.30 (m, 1H, $(CH_3)_3COCON\underline{H}$), 4.44 (quin, *J* = 7.2 Hz, 1H, $C\underline{H}CH_3$), 4.35 (d, *J* = 6.6 Hz, 1H, $C\underline{H}CH_2OC(CH_3)_2$), 4.29 (d, *J* = 9.4 Hz, 1H, $CHC\underline{H}_2OC(CH_3)_2$), 4.18 (dd, *J* = 9.4, 6.6 Hz, 1H, $CHC\underline{H}_2OC(CH_3)_2$), 3.90 (dd, *J* = 17.0, 5.9 Hz, 1H, $C\underline{H}_2$), 3.79 (dd, *J* = 17.0, 4.3 Hz, 1H x 77/100, $C\underline{H}_2$), 3.71 (dd, *J* = 17.0, 4.3 Hz, 1H x 23/100, $C\underline{H}_2$), 1.76 (s, 3H, $CHCH_2OC(C\underline{H}_3)_2$), 1.58 (s, 3H, $CHCH_2OC(C\underline{H}_3)_2$), 1.46 (s, 9H x 23/100, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H x 77/100, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.41 (d, *J* = 7.2 Hz, 3H x 77/100, $CHC\underline{H}_3$), 1.40 (d, *J* = 7.2 Hz, 3H x 23/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.4, 169.2 (1C x 77/100), 168.9 (1C x 23/100), 166.8 (1C x 77/100), 166.7 (1C x 23/100), 156.0 (1C x 77/100), 155.8 (1C x 23/100), 97.1 (1C x 23/100), 97.0 (1C x 77/100), 82.4 (1C x 23/100), 82.3 (1C x 77/100), 80.0 (1C x 23/100), 79.9 (1C x 77/100), 67.9, 59.9, 49.0 (1C x 23/100), 49.0 (1C x 77/100), 43.9 (1C x 23/100), 43.8 (1C x 77/100), 28.2 (3C), 27.9 (3C), 25.8 (1C x 23/100), 25.7 (1C x 77/100), 22.8, 18.2 (1C x 23/100), 17.9 (1C x 77/100). IR (thin film, cm$^{-1}$) 3303, 2977, 1649, 1521, 1424, 1366, 1246, 1147, 1077, 1051, 841. HRMS (ESI) calculated for $C_{20}H_{35}N_3O_7Na$ $[M+Na]^+$ *m/z* 452.2373, found, 452.2388.



**Boc-Gly-L-Cys($\psi^{Me,Me}$pro)-L-Ala-O*t*-Bu (6k):** A flame-dried 5.0 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Cyclo(-Boc-Gly-L-Cys($\psi^{Me,Me}$pro)-) (**5k**, 150.2 mg, 0.50 mmol) and H-L-Ala-O*t*-Bu (145.2 mg, 1.0 mmol). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 24 h. The reaction mixture was diluted with $CHCl_3$ (3.0 mL), transferred onto $SiO_2$ column by a pipette, and used vial and pipette were washed with $CHCl_3$ (12 mL). The reaction mixture was purified directly by flash column chromatography (AcOEt) to provide a mixture of the title compound **6k** and H-L-Ala-O*t*-Bu. Finally, H-L-Ala-O*t*-Bu was evaporated *in vacuo* with the aid of an oil rotary vacuum pump to provide **6k** (207.2 mg, 93% yield, ratio of rotamers = 63:37) as a white solid with >99:1 dr.

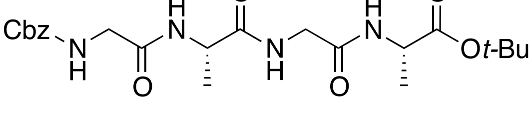
$R_f$ = 0.26 (50% AcOEt in hexane). M.p. 54–55 °C. $[\alpha]_D^{20}$ = –39.0 (*c* 1.00, MeOH). $^1$H NMR (400 MHz, $CDCl_3$) δ 6.96 (br d, *J* = 7.1 Hz, 1H, $N\underline{H}$), 5.45–5.25 (m, 1H, $(CH_3)_3COCON\underline{H}$), 4.66 (br s, 1H, $C\underline{H}CH_2SC(CH_3)_2$), 4.44 (quin, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 4.00–3.60 (m, 2H, $C\underline{H}_2$), 3.36 (d, *J* = 11.8 Hz, 1H, $CHC\underline{H}_2SC(CH_3)_2$), 3.31 (d, *J* = 11.8 Hz, 1H, $CHC\underline{H}_2SC(CH_3)_2$), 2.04 (s, 3H x 37/100, $CHCH_2SC(C\underline{H}_3)_2$), 1.98 (s, 3H x 63/100, $CHCH_2SC(C\underline{H}_3)_2$), 1.85 (s, 3H x 63/100, $CHCH_2SC(C\underline{H}_3)_2$), 1.84 (s, 3H x 37/100, $CHCH_2SC(C\underline{H}_3)_2$), 1.47 (s, 9H x 37/100, $CO_2C(C\underline{H}_3)_3$), 1.45 (s, 9H x 63/100, $CO_2C(C\underline{H}_3)_3$), 1.43 (s, 9H, $(C\underline{H}_3)_3COCONH$), 1.42 (d, *J* = 7.1 Hz, 3H x 63/100, $CHC\underline{H}_3$), 1.40 (d, *J* = 7.1 Hz, 3H x 37/100, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 171.4, 168.8 (1C x 63/100), 168.7 (1C x 37/100), 167.5 (1C x 63/100), 167.4 (1C x 37/100), 156.0 (1C x 63/100), 155.7 (1C x 37/100), 82.4 (1C x 37/100), 82.2 (1C x 63/100), 79.8, 74.5 (1C x 37/100), 74.5 (1C x 63/100), 66.0, 49.2, 44.6, 32.4, 28.6, 28.2 (3C), 27.9 (3C x 37/100), 27.9 (3C x 63/100), 27.7, 18.3 (1C x 37/100), 18.0 (1C x 63/100). IR (thin film, cm$^{-1}$) 3319, 2979, 1670, 1651, 1517, 1453, 1392, 1366, 1246, 1147, 1052. HRMS (ESI) calculated for $C_{20}H_{35}N_3O_6SNa$ $[M+Na]^+$ *m/z* 468.2144, found, 468.2139.

## 10. Synthesis of hexapeptides (Scheme 4)



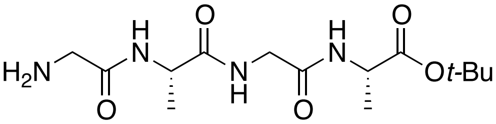
**H-Gly-L-Ala-O*t*-Bu (7, CAS No. 58177-80-3)[12]:** A flame-dried 300 mL two-necked round-bottom flask equipped with a magnetic stirring bar (Nd) and nitrogen balloon was charged with H-Gly-OH (2.63 g, 35 mmol), CsF (531.7 mg, 3.5 mmol), imidazole (238.3 mg, 3.5 mmol), and $CHCl_3$ (70 mL). After $HSi[OCH(CF_3)_2]_3$ (37.11 g, 70 mmol) was added to the mixture at room and then the resulting mixture was stirred vigorously under nitrogen atmosphere at 50 °C for 1 h. Next, *N*-(*tert*-butyldimethylsilyl)-*N*-methyltrifluoroacetamide (MTBSTFA, 8.45 g, 35 mmol) was added to the reaction mixture and the resulting mixture was stirred vigorously under nitrogen atmosphere at 30 °C for 1 h. Then H-L-Ala-O*t*-Bu (10.16 g, 70 mmol) was added to the reaction mixture and the resulting mixture was stirred vigorously under nitrogen atmosphere at 30 °C for 24 h. The reaction mixture was transferred onto $SiO_2$ column by a pipette, and used two-necked round-bottom flask and pipette were washed with $CHCl_3$ (20 mL). The reaction mixture was purified directly by flash column chromatography (5–20% MeOH in $CHCl_3$) to provide the title compound **7** (5.53 g, 78% yield).

$R_f = 0.53$ (20% MeOH in $CHCl_3$). $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.74 (br d, *J* = 7.1 Hz, 1H, N$\underline{H}$), 4.49 (quin, *J* = 7.1 Hz, 1H, C$\underline{H}$CH$_3$), 3.43 (s, 2H, C$\underline{H}_2$), 2.42 (br s, 2H, N$\underline{H}_2$), 1.47 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.39 (d, *J* = 7.1 Hz, 3H, CHC$\underline{H}_3$). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 172.2, 171.7, 81.8, 48.2, 44.4, 27.9 (3C), 18.6. IR (thin film, cm$^{-1}$) 3301, 2980, 2935, 1732, 1655, 1521, 1454, 1368, 1225, 1146, 846.



**Cbz-Gly-L-Ala-Gly-L-Ala-O*t*-Bu (8):** A flame-dried 100 mL schlenk flask equipped with a magnetic stirring bar (Nd) and nitrogen balloon was charged with H-Gly-Ala-O*t*-Bu (5.53 g, 27.3 mmol), Cbz-Gly-L-Ala-OH (5.11 g, 18.2 mmol), and $CHCl_3$ (18.2 mL). After $HSi[OCH(CF_3)_2]_3$ (12.56 g, 23.7 mmol) was added to the mixture at room temperature, the resulting mixture was stirred vigorously under nitrogen atmosphere at 50 °C for 24 h. Next, the reaction mixture was transferred to a separatory funnel with $CHCl_3$ (300 mL) and saturated aqueous solution of $NH_4Cl$ (150 mL) was added. The phases were separated and aqueous layer was extracted with $CHCl_3$ (2 x 200 mL). The organic phase dried over $MgSO_4$, filtered, concentrated in *vacuo* with the aid of a rotary evaporator and water bath. The crude product was then diluted with 5% MeOH in $CHCl_3$ (30 mL), transferred onto $SiO_2$ column by a pipette, and used round-bottom flask and pipette were washed with 5% MeOH in $CHCl_3$ (2 x 20 mL). The crude product was purified by flash column chromatography (5–20% MeOH in $CHCl_3$) to provide the title compound **8** (8.43 g, >99% yield) as a white solid with >99:1 dr.

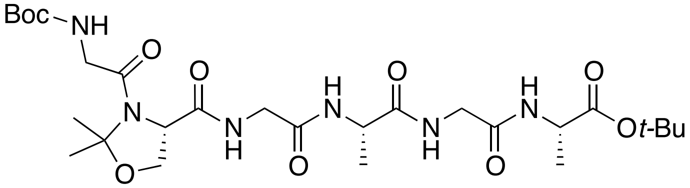
$R_f$ = 0.30 (10% MeOH in $CHCl_3$). M.p. 145–147 °C. $[\alpha]_D^{21}$ = –32.2 (*c* 1.15, MeOH). $^1$H NMR (400 MHz, $CD_3OD$) δ 7.40–7.20 (m, 5H, $C_6\underline{H}_5CH_2OCONH$), 5.08 (s, 2H, $C_6H_5C\underline{H}_2OCONH$), 4.31 (q, *J* = 7.1 Hz, 1H, $C\underline{H}CH_3$), 4.23 (q, *J* = 7.3 Hz, 1H, $C\underline{H}CH_3$), 4.00–3.70 (m, 4H, $C\underline{H}_2$ and $C\underline{H}_2$), 1.44 (s, 9H, $CO_2C(C\underline{H}_3)_3$), 1.37 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$), 1.33 (d, *J* = 7.1 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $CD_3OD$) δ 175.4, 173.4, 172.4, 171.3, 159.2, 138.0, 129.5 (2C), 129.1, 128.9 (2C), 82.7, 67.9, 51.0, 50.3, 44.8, 43.2, 28.2 (3C), 17.4 (2C). IR (thin film, cm$^{-1}$) 3277, 1733, 1711, 1673, 1631, 1514, 1447, 1366, 1231, 1148, 1048. HRMS (ESI) calculated for $C_{22}H_{32}N_4O_7Na$ $[M+Na]^+$ *m/z* 487.2169, found, 487.2164.



**H-Gly-L-Ala-Gly-L-Ala-O*t*-Bu (9):** A flame-dried 300 mL round-bottom flask equipped with a magnetic stirring bar (Sm-Co) was charged with **8** (8.42 g, 18.16 mmol), 5% Pd/C type NX (843.4 mg, 10 wt%), and MeOH (180 mL), then evacuated and backfilled with hydrogen (this process was repeated a total of 15 times). The resulting mixture was stirred under hydrogen atmosphere at ambient temperature for 5 h. After completion, the reaction mixture was filtrated by filter paper with MeOH (200 mL), and the solvents were removed in *vacuo* with the aid of a rotary evaporator. The title compound **9** (5.98 g, >99% yield) was obtained as a white solid with >99:1 dr.

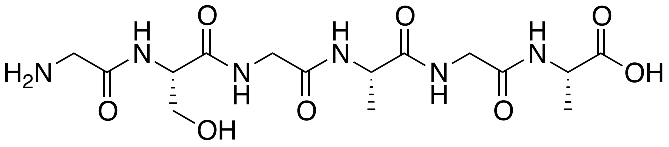
$R_f$ = 0.20 (50% MeOH in $CHCl_3$). M.p. 27–28 °C. $[\alpha]_D^{23}$ = –38.1 (*c* 1.05, MeOH). $^1$H NMR (400 MHz, $CD_3OD$) δ 4.35 (q, *J* = 7.3 Hz, 1H, $C\underline{H}CH_3$), 4.28 (q, *J* = 7.3 Hz, 1H, $C\underline{H}CH_3$), 3.92 (d, *J* =

16.7 Hz, 1H, C*H*$_2$), 3.85 (d, *J* = 16.7 Hz, 1H, C*H*$_2$), 3.57 (d, *J* = 16.5 Hz, 1H, C*H*$_2$), 3.53 (d, *J* = 16.5 Hz, 1H, C*H*$_2$), 1.46 (s, 9H, CO$_2$C(C*H*$_3$)$_3$), 1.39 (d, *J* = 7.3 Hz, 3H, CHC*H*$_3$), 1.37 (d, *J* = 7.3 Hz, 3H, CHC*H*$_3$). $^{13}$C NMR (100 MHz, CD$_3$OD) δ 175.3, 173.5, 171.1, 170.9, 82.8, 50.8, 50.3, 43.2, 43.0, 28.2 (3C), 17.7, 17.5. IR (thin film, cm$^{-1}$) 3309, 2980, 2935, 2476, 1732, 1643, 1532, 1447, 1331, 1228, 1146. HRMS (ESI) calculated for $C_{14}H_{26}N_4O_5Na$ [M+Na]$^+$ *m/z* 353.1801, found, 353.1833.



**Boc-Gly-L-Ser(ψ$^{Me,Me}$pro)-Gly-L-Ala-Gly-L-Ala-O*t*-Bu (10):** A flame-dried 50 mL screw-cap vial equipped with a magnetic stirring bar (Nd) was charged with Cyclo(-Boc-Gly-L-Ser(ψ$^{Me,Me}$pro)-) (**5j**, 1.71 g, 6.0 mmol), H-Gly-L-Ala-Gly-L-Ala-O*t*-Bu (**9**, 3.96 g, 12.0 mmol), and CPME (9 mL). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at 50 °C for 48 h. The reaction mixture was diluted with 10% MeOH in CHCl$_3$ (20 mL), transferred onto SiO$_2$ column by a pipette, and used vial and pipette were washed with 10% MeOH in CHCl$_3$ (100 mL). The reaction mixture was purified directly by flash column chromatography (10% MeOH in CHCl$_3$) to provide the title compound **10** (3.13 g, 85% yield, >99:1 dr) as a white solid with >99:1 dr.

R$_f$ = 0.31 (10% MeOH in CHCl$_3$). M.p. 119–120 °C. $[\alpha]_D^{19}$ = –53.3 (*c* 1.05, MeOH). $^1$H NMR (400 MHz, CD$_3$OD) δ 4.70–3.50 (m, 11H, C*H*$_2$, C*H*C*H*$_2$OC(CH$_3$)$_2$, C*H*$_2$, C*H*CH$_3$, C*H*$_2$, and C*H*CH$_3$), 1.67 (s, 3H, CHCH$_2$OC(C*H*$_3$)$_2$), 1.55 (s, 3H, CHCH$_2$OC(C*H*$_3$)$_2$), 1.46 (s, 9H, CO$_2$C(C*H*$_3$)$_3$), 1.44 (s, 9H, (C*H*$_3$)$_3$COCONH), 1.38 (d, *J* = 7.3 Hz, 3H, CHC*H*$_3$), 1.36 (d, *J* = 7.3 Hz, 3H, CHC*H*$_3$). $^{13}$C NMR (100 MHz, CD$_3$OD) δ 175.4, 173.4, 172.8, 171.1 (2C), 169.0, 158.5, 98.1, 82.7, 80.6, 69.1, 60.7, 50.8, 50.3, 44.5, 43.7, 43.3, 28.7 (3C), 28.2 (3C), 25.5, 23.5, 17.6 (2C). IR (thin film, cm$^{-1}$) 3301, 2979, 1701, 1660, 1630, 1520, 1366, 1230, 1146, 1079, 1053. HRMS (ESI) calculated for $C_{27}H_{46}N_6O_{10}Na$ [M+Na]$^+$ *m/z* 637.3173, found, 637.3135.



**H-Gly-L-Ser-Gly-L-Ala-Gly-L-Ala-OH (11, CAS No. 844644-62-8):** A 20 mL screw-cap vial equipped with a magnetic stirring bar (Sm-Co) was charged with Boc-Gly-L-Ser($\psi^{Me,Me}$pro)-Gly-L-Ala-Gly-L-Ala-O*t*-Bu (**10**, 307.3 mg, 0.50 mmol) and 1 *N* HCl aq. (5.0 mL). After the vial was sealed with screw-cap, the resulting mixture was stirred vigorously at room temperature for 2 h. The reaction mixture was concentrated to provide the title compound **11** (209.0 mg, >99% yield) as a white solid with >99:1 dr.

M.p. 200–201 °C. $^1$H NMR (400 MHz, $D_2O$) δ 4.50 (t, *J* = 5.2 Hz, 1H, $C\underline{H}CH_2OH$), 4.45–4.20 (m, 2H, $C\underline{H}CH_3$ and $C\underline{H}CH_3$), 4.05–3.80 (m, 8H, $C\underline{H}_2$, $CHC\underline{H}_2OH$, $C\underline{H}_2$, and $C\underline{H}_2$), 1.38 (d, *J* = 7.3 Hz, 3H, $CHC\underline{H}_3$), 1.36 (d, *J* = 7.8 Hz, 3H, $CHC\underline{H}_3$). $^{13}$C NMR (100 MHz, $(CD_3)_2SO$) δ 174.0, 172.4, 170.0, 168.7, 168.5, 166.3, 61.6, 55.6, 48.5, 47.5, 42.2, 41.7, 30.7, 17.9, 17.2. IR (thin film, cm$^{-1}$) 3284, 1701, 1660, 1633, 1526, 1366, 1198, 1169, 1130. HRMS (ESI) calculated for $C_{15}H_{26}N_6O_8Na$ $[M+Na]^+$ *m/z* 441.1710, found, 441.1712.

# 11. References

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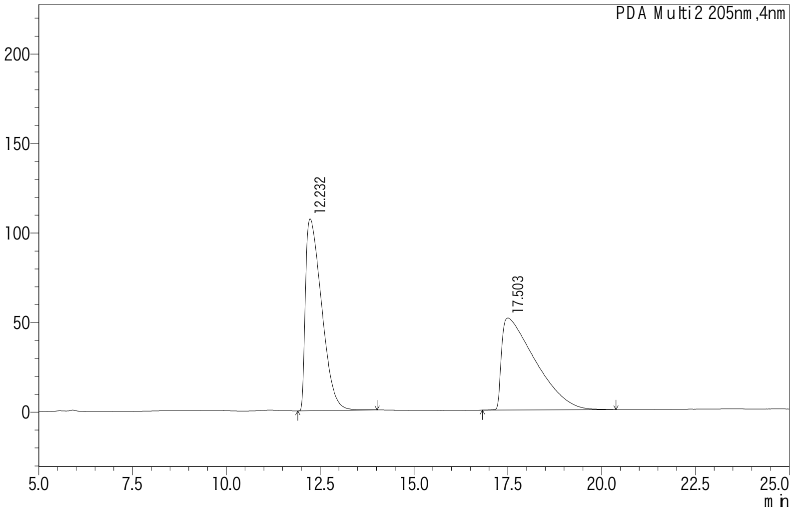
## 12. HPLC data

### Boc-δ-HoGly-*rac*-Ala-O*t*-Bu



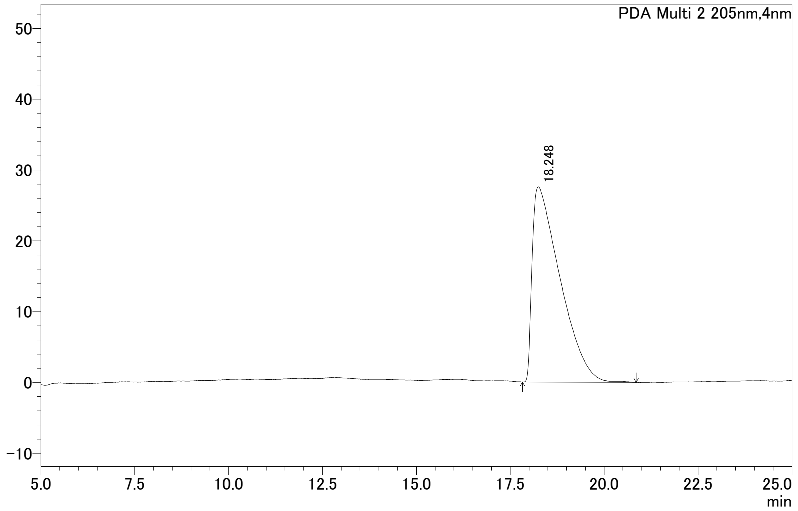
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 12.232 | 3212531 | 49.996 |
| 2 | 17.503 | 3213053 | 50.004 |
| Total | | 6425584 | 100.000 |

12.232 min = Boc-δ-HoGly-D-Ala-O*t*-Bu; 17.503 min = Boc-δ-HoGly-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 3:97, *v* = 1.5 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

### Boc-δ-HoGly-L-Ala-O*t*-Bu (2a, Table 1)

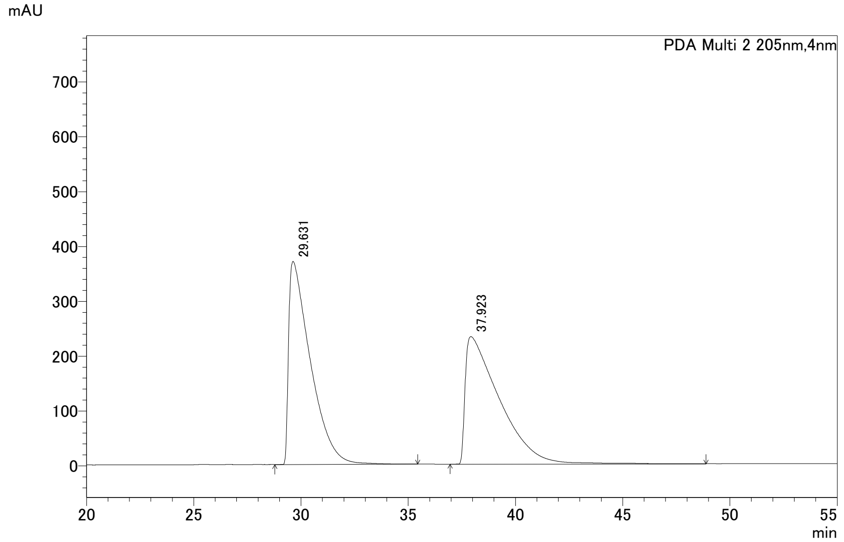


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 18.248 | 1423592 | 100.000 |
| Total | | 1423592 | 100.000 |

## Cbz-δ-HoGly-*rac*-Ala-O*t*-Bu



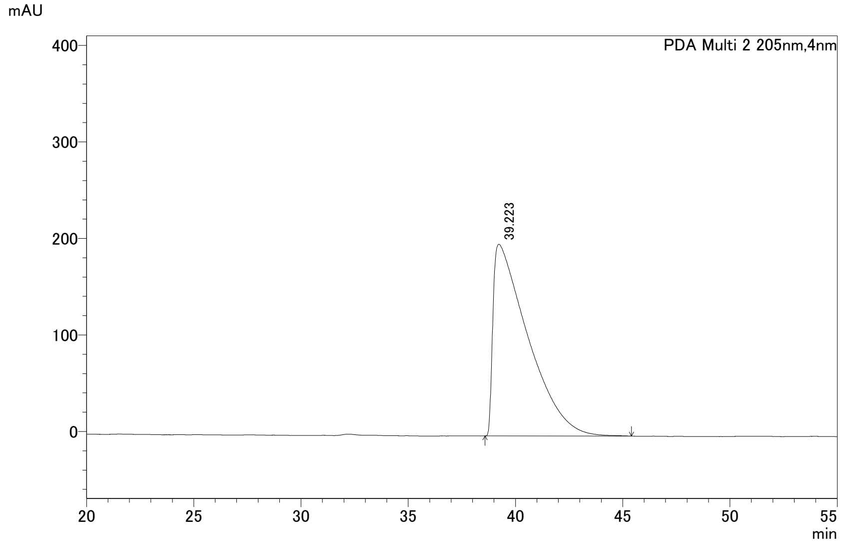
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 29.631 | 26085163 | 50.012 |
| 2 | 37.923 | 26073079 | 49.988 |
| Total | | 52158241 | 100.000 |

29.631 min = Cbz-δ-HoGly-D-Ala-O*t*-Bu; 37.923 min = Cbz-δ-HoGly-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 3:97, *v* = 1.5 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

## Cbz-δ-HoGly-L-Ala-O*t*-Bu (2b, Table 1)

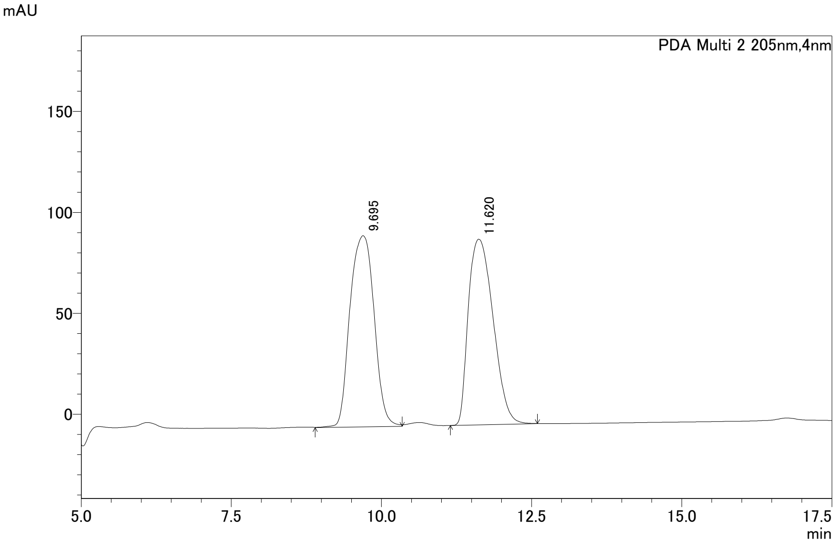


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 39.223 | 22498903 | 100.000 |
| Total | | 22498903 | 100.000 |

### Boc-β-HoGly-*rac*-Ala-O*t*-Bu



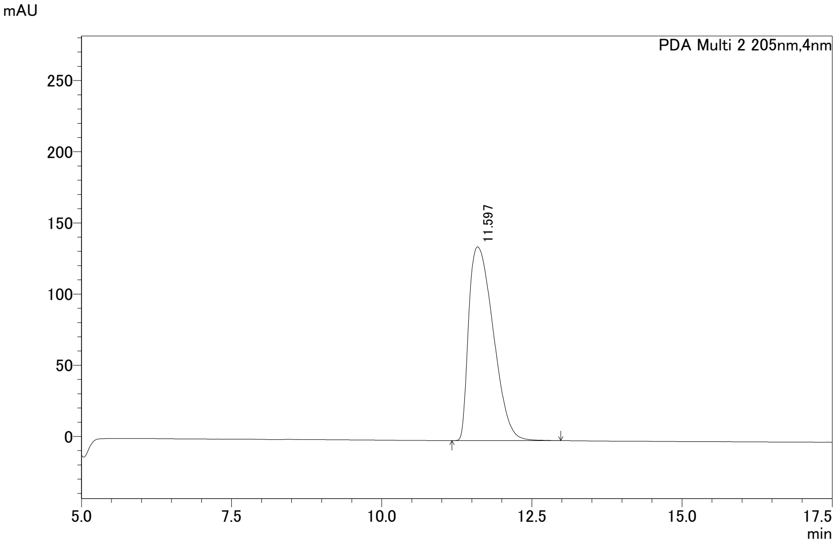
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 9.695 | 2657873 | 50.141 |
| 2 | 11.620 | 2642949 | 49.859 |
| Total | | 5300822 | 100.000 |

9.977 min = Boc-β-HoGly-D-Ala-O*t*-Bu; 12.652 min = Boc-β-HoGly-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 4:96, *v* = 0.8 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

### Boc-β-HoGly-L-Ala-O*t*-Bu (2h, Table 1)

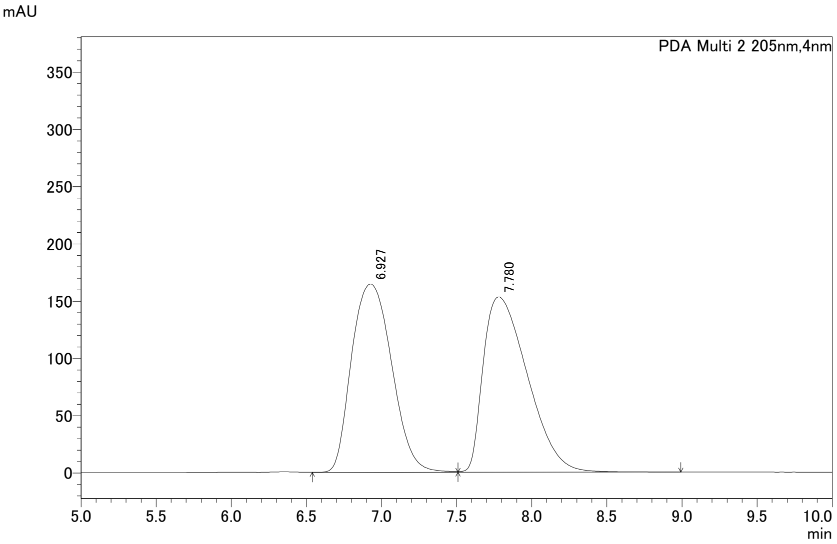


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 11.597 | 3876838 | 100.000 |
| Total | | 3876838 | 100.000 |

### **Boc-γ-HoGly-*rac*-Ala-O*t*-Bu**



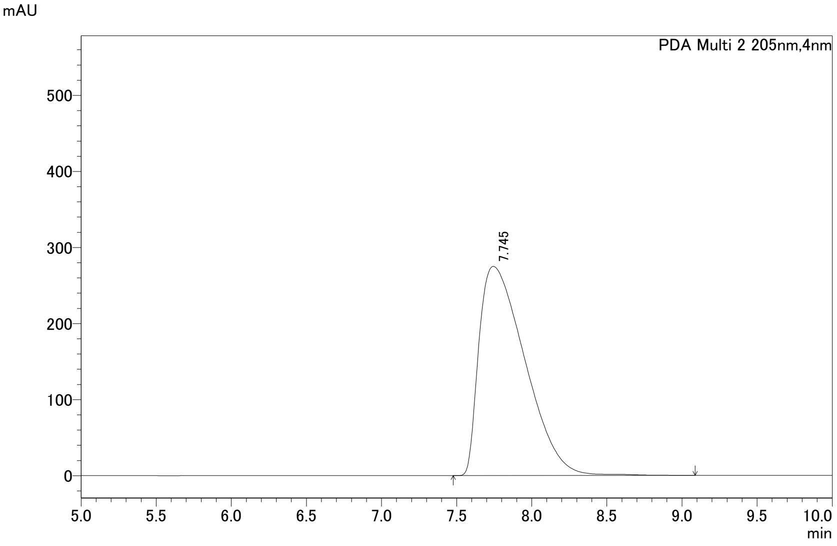
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 6.927 | 3069679 | 49.427 |
| 2 | 7.780 | 3140874 | 50.573 |
| Total | | 6210553 | 100.000 |

6.927 min = Boc-γ-HoGly-D-Ala-O*t*-Bu; 7.780 min = Boc-γ-HoGly-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 6:94, *v* = 1.0 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

### **Boc-γ-HoGly-L-Ala-O*t*-Bu (2i, Table 1)**

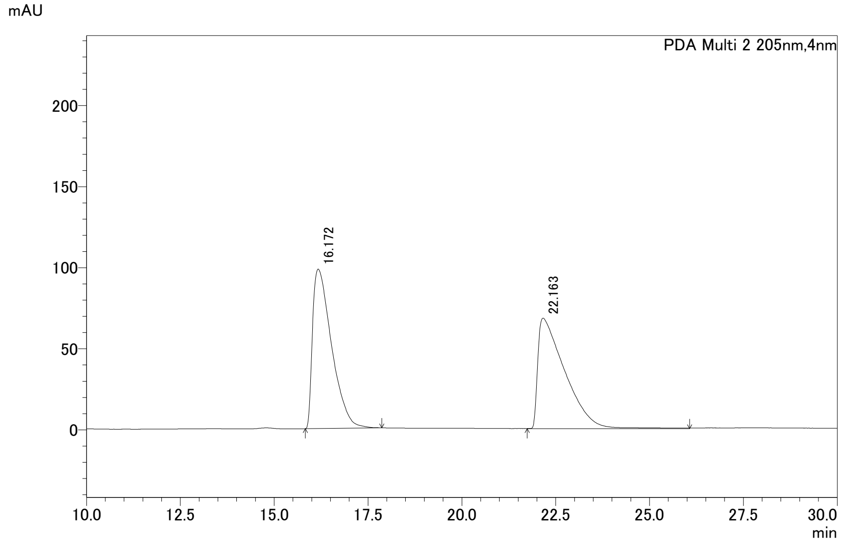


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 7.745 | 5860385 | 100.000 |
| Total | | 5860385 | 100.000 |

### Boc-ε-HoGly-*rac*-Ala-O*t*-Bu



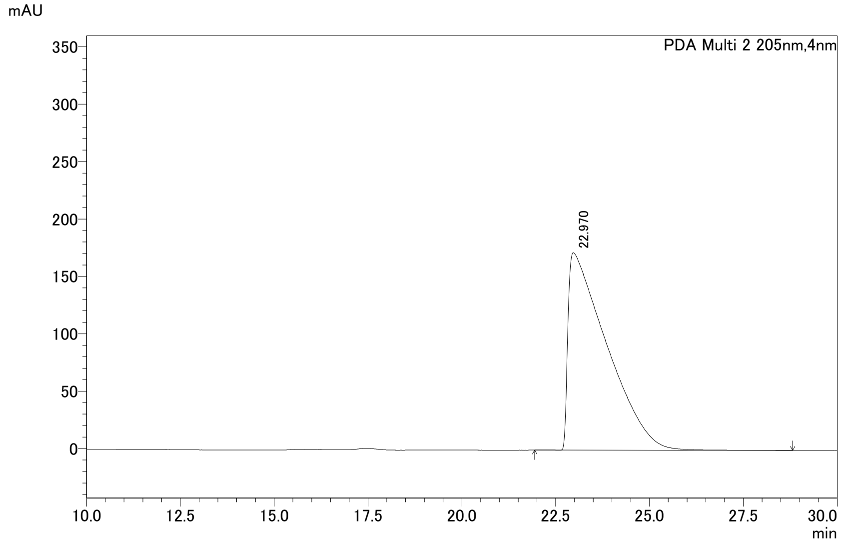
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 16.172 | 3466668 | 50.248 |
| 2 | 22.163 | 3432415 | 49.752 |
| Total | | 6899083 | 100.000 |

16.172 min = Boc-ε-HoGly-D-Ala-O*t*-Bu; 22.163 min = Boc-ε-HoGly-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 4:96, *v* = 0.8 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

### Boc-ε-HoGly-L-Ala-O*t*-Bu (2j, Table 1)

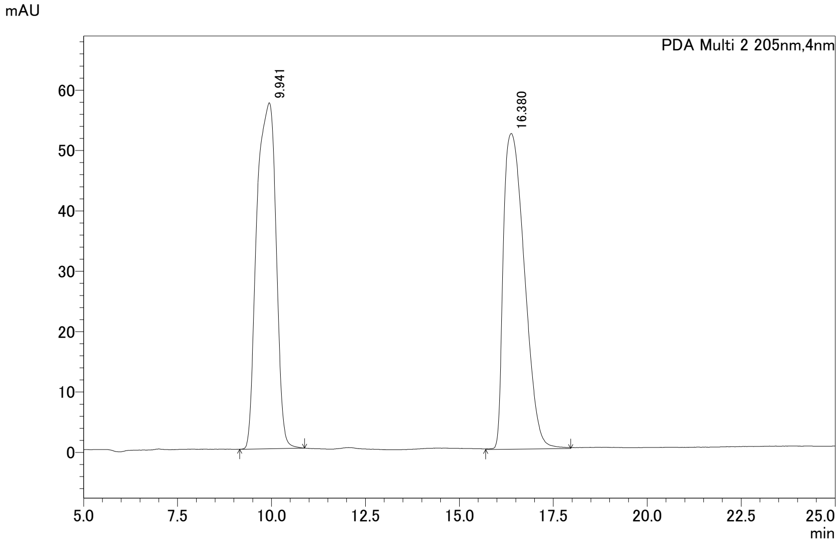


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 22.970 | 12357955 | 100.000 |
| Total | | 12357955 | 100.000 |

**Boc-λ-HoGly-*rac*-Ala-O*t*-Bu**



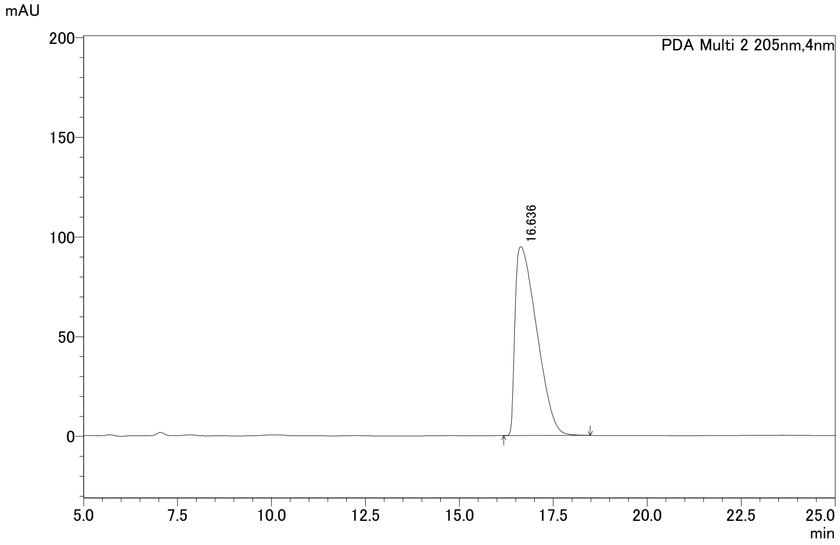
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 9.941 | 2043000 | 49.988 |
| 2 | 16.380 | 2043954 | 50.012 |
| Total | | 4086955 | 100.000 |

9.941 min = Boc-λ-HoGly-ᴅ-Ala-O*t*-Bu; 16.380 min = Boc-λ-HoGly-ʟ-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 5:95, *v* = 1.0 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

**Boc-λ-HoGly-ʟ-Ala-O*t*-Bu (2k, Table 1)**

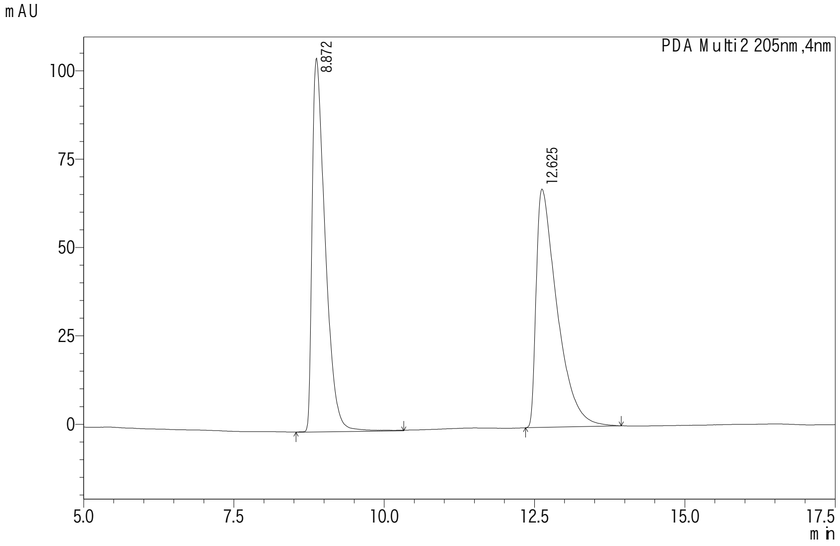


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 16.636 | 3789938 | 100.000 |
| Total | | 3789938 | 100.000 |

### Boc-Gly-*rac*-Ala-O*t*-Bu



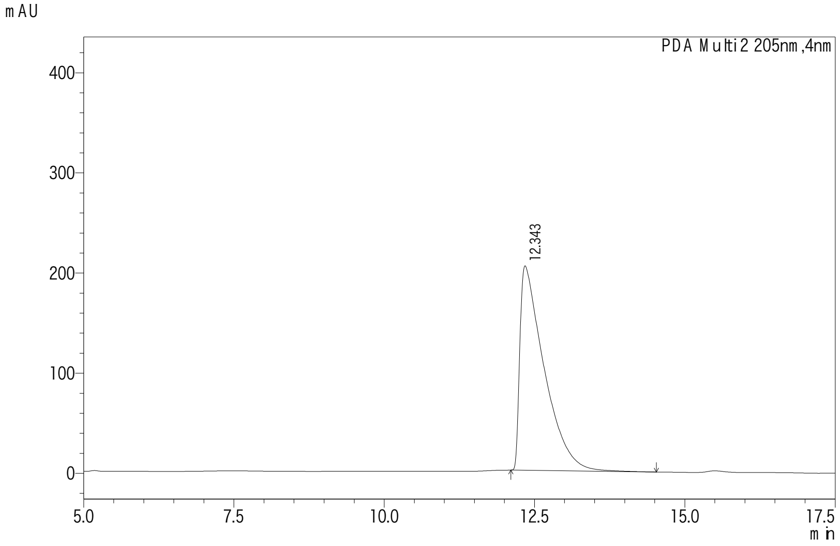
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 8.872 | 1605779 | 49.560 |
| 2 | 12.625 | 1634285 | 50.440 |
| Total |  | 3240064 | 100.000 |

8.872 min = Boc-Gly-D-Ala-O*t*-Bu; 12.625 min = Boc-Gly-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 3:97, *v* = 1.5 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

### Boc-Gly-L-Ala-O*t*-Bu (4kb, Scheme 2)

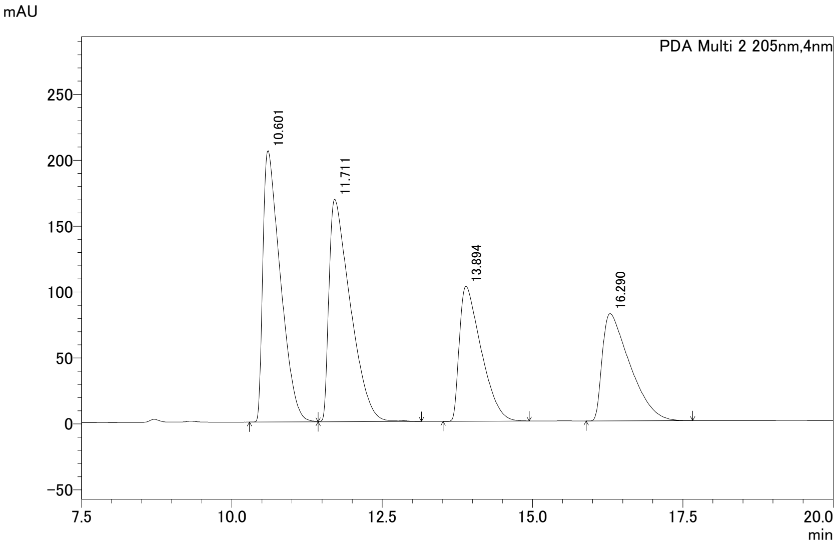


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 12.343 | 5733022 | 100.000 |
| Total |  | 5733022 | 100.000 |

**Boc-*rac*-Ala-*rac*-Ala-O*t*-Bu**



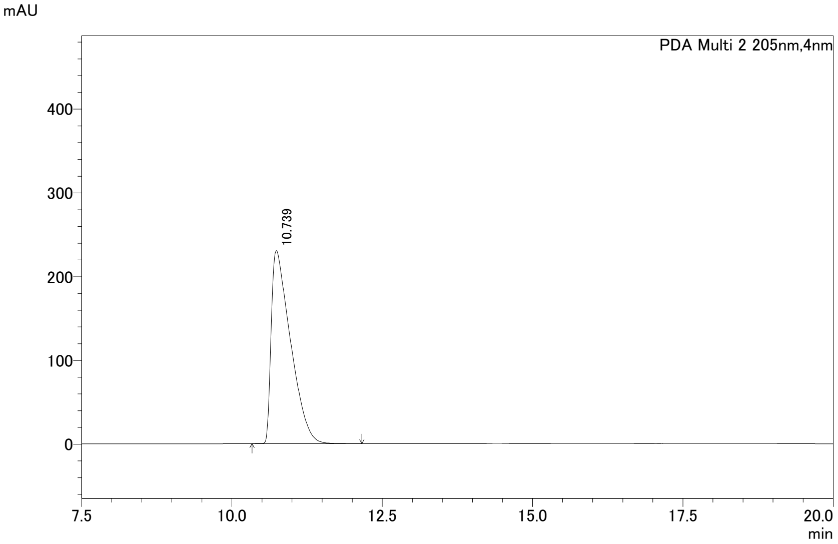
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 10.601 | 4167761 | 30.863 |
| 2 | 11.711 | 4161467 | 30.816 |
| 3 | 13.894 | 2572053 | 19.047 |
| 4 | 16.290 | 2602764 | 19.274 |
| Total | | 13504045 | 100.000 |

10.601 min = Boc-L-Ala-L-Ala-O*t*-Bu; 11.711 min = Boc-D-Ala-D-Ala-O*t*-Bu
13.894 min = Boc-L-Ala-D-Ala-O*t*-Bu; 16.290 min = Boc-D-Ala-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 5:95, *v* = 1.5 mL/min, λ = 205 nm, 40 °C
Chiral Column: IE-3 column from Daicel Chemical Ind., Ltd

**Boc-L-Ala-L-Ala-O*t*-Bu (4l, Scheme 2)**

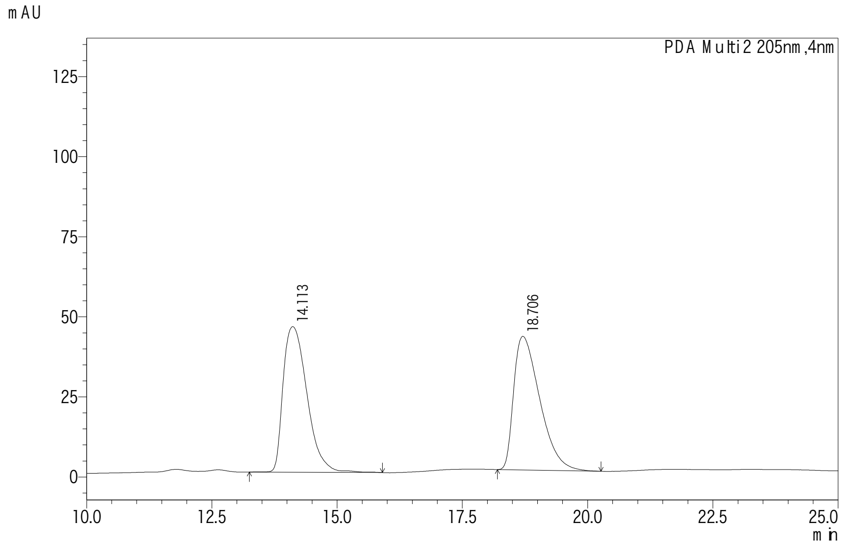


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 10.739 | 5153218 | 100.000 |
| Total | | 5153218 | 100.000 |

## Boc-Gly-*rac*-Val-O*t*-Bu



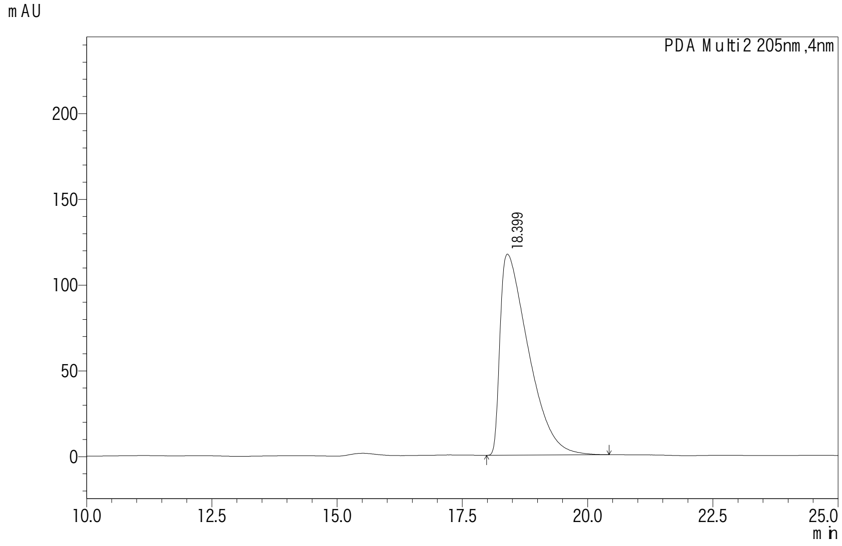
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 14.113 | 1510265 | 49.939 |
| 2 | 18.706 | 1513968 | 50.061 |
| Total | | 3024233 | 100.000 |

14.113 min = Boc-Gly-D-Val-O*t*-Bu; 18.706 min = Boc-Gly-L-Val-O*t*-Bu
Conditions: 2-propanol/hexane = 2:98, *v* = 1.5 mL/min, λ = 205 nm, 40 °C
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

## Boc-Gly-L-Val-O*t*-Bu (4kc, Scheme 2)

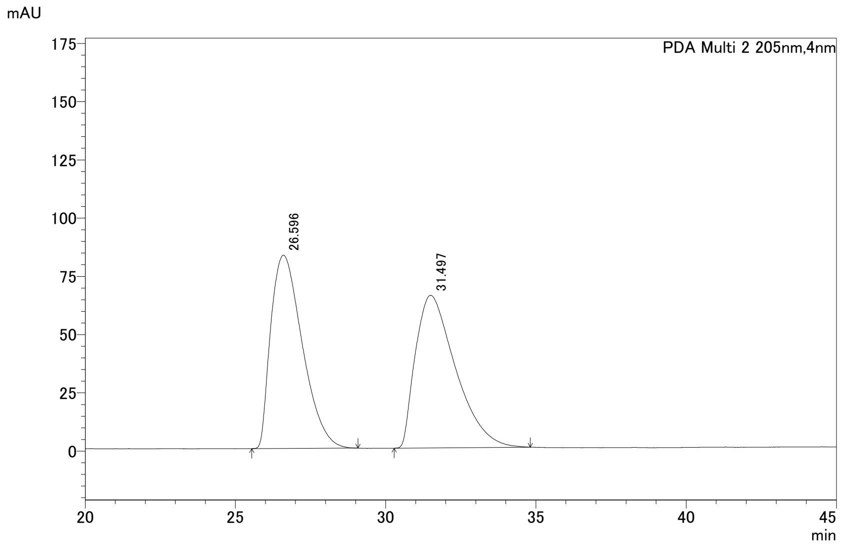


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 18.399 | 4621090 | 100.000 |
| Total | | 4621090 | 100.000 |

**Boc-Gly-Gly-*rac*-Ala-O*t*-Bu**



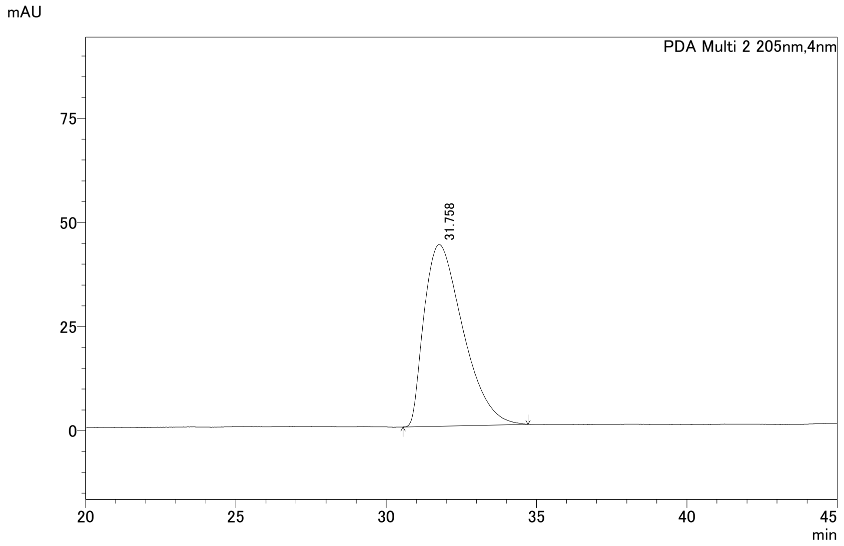
Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 26.596 | 6066032 | 49.908 |
| 2 | 31.497 | 6088485 | 50.092 |
| Total | | 12154517 | 100.000 |

26.596 min = Boc-Gly-Gly-D-Ala-O*t*-Bu; 31.497 min = Boc-Gly-Gly-L-Ala-O*t*-Bu
Conditions: 2-propanol/hexane = 7:93, *v* = 1.5 mL/min, λ = 205 nm, 40 °C
Chiral Column: IG-3 column from Daicel Chemical Ind., Ltd

**Boc-Gly-Gly-L-Ala-O*t*-Bu (6aa, Scheme 3)**

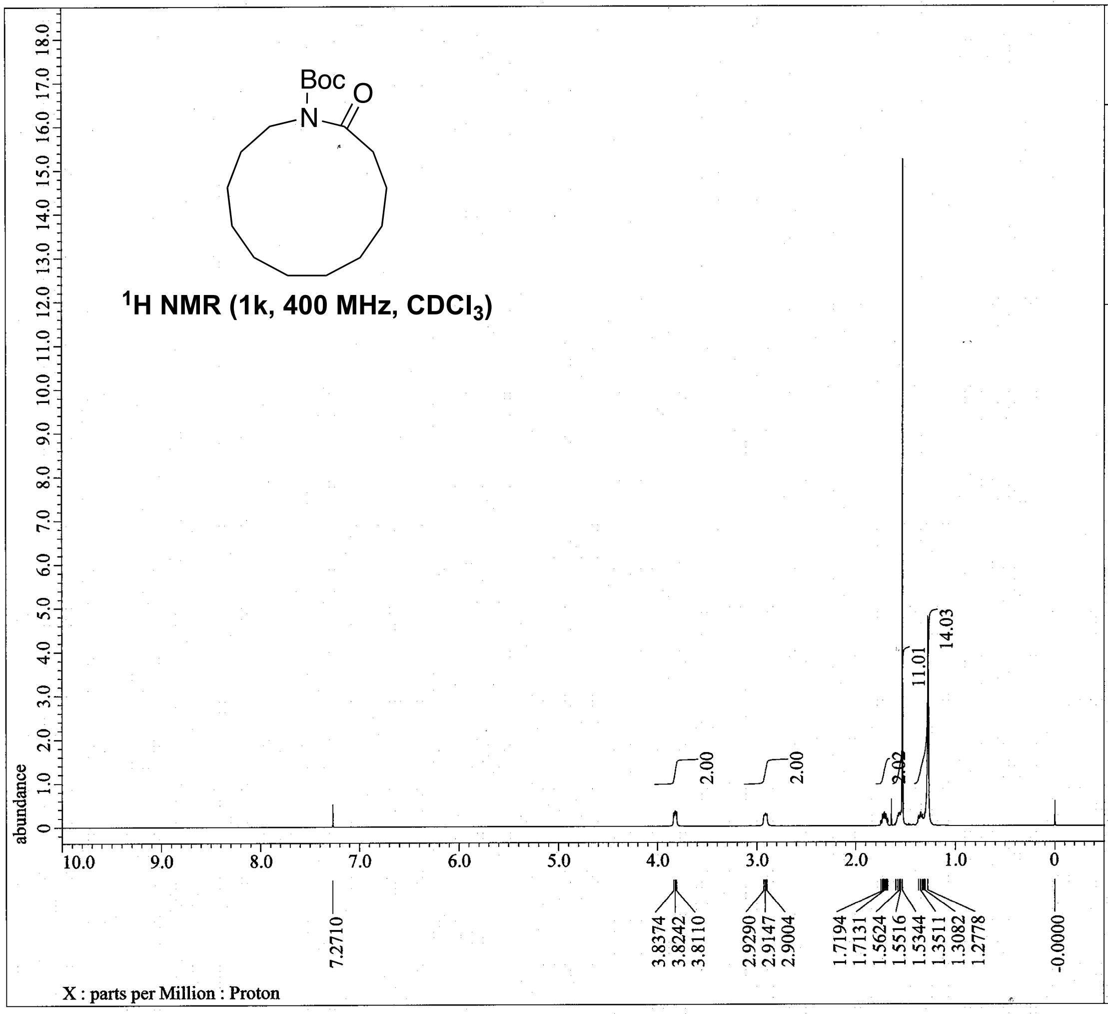


Peak Table

PDA Ch2 205nm

| Peak# | Ret. Time | Area | Area% |
|---|---|---|---|
| 1 | 31.758 | 3916898 | 100.000 |
| Total | | 3916898 | 100.000 |

# 13. NMR data



$^1$H NMR (1k, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-195_E1H-1-1.jdf
```

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Filename           = wm12-195_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-195
Solvent            = CHLOROFORM-D
Creation_Time      = 11-JAN-2022 11:40:23
Revision_Time      = 11-JAN-2022 11:42:44
Current_Time       = 11-JAN-2022 11:43:05

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 23.6[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
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X_Pulse            = 3.235[us]
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Tri_Mode           = Off
Dante_Presat       = FALSE
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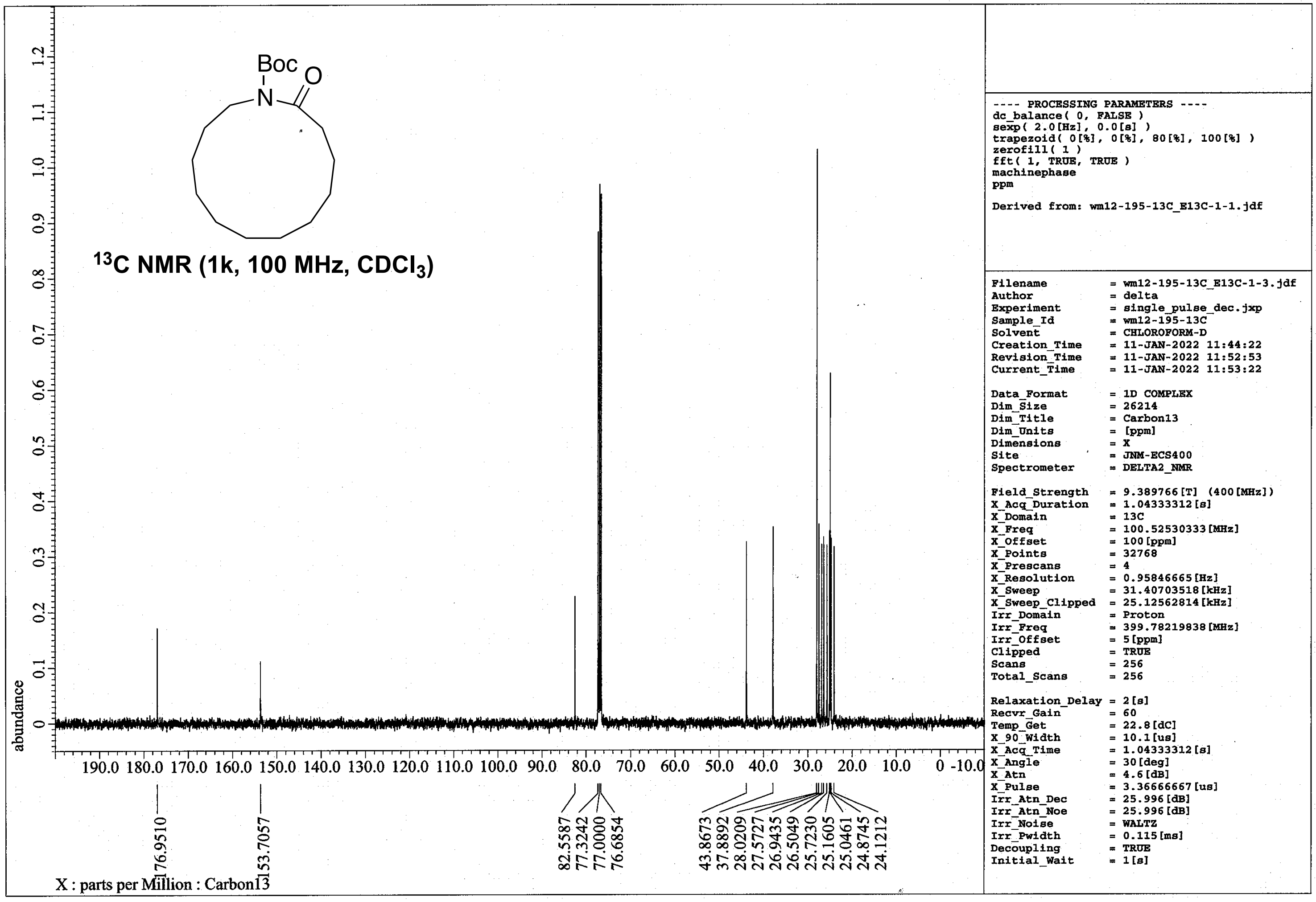
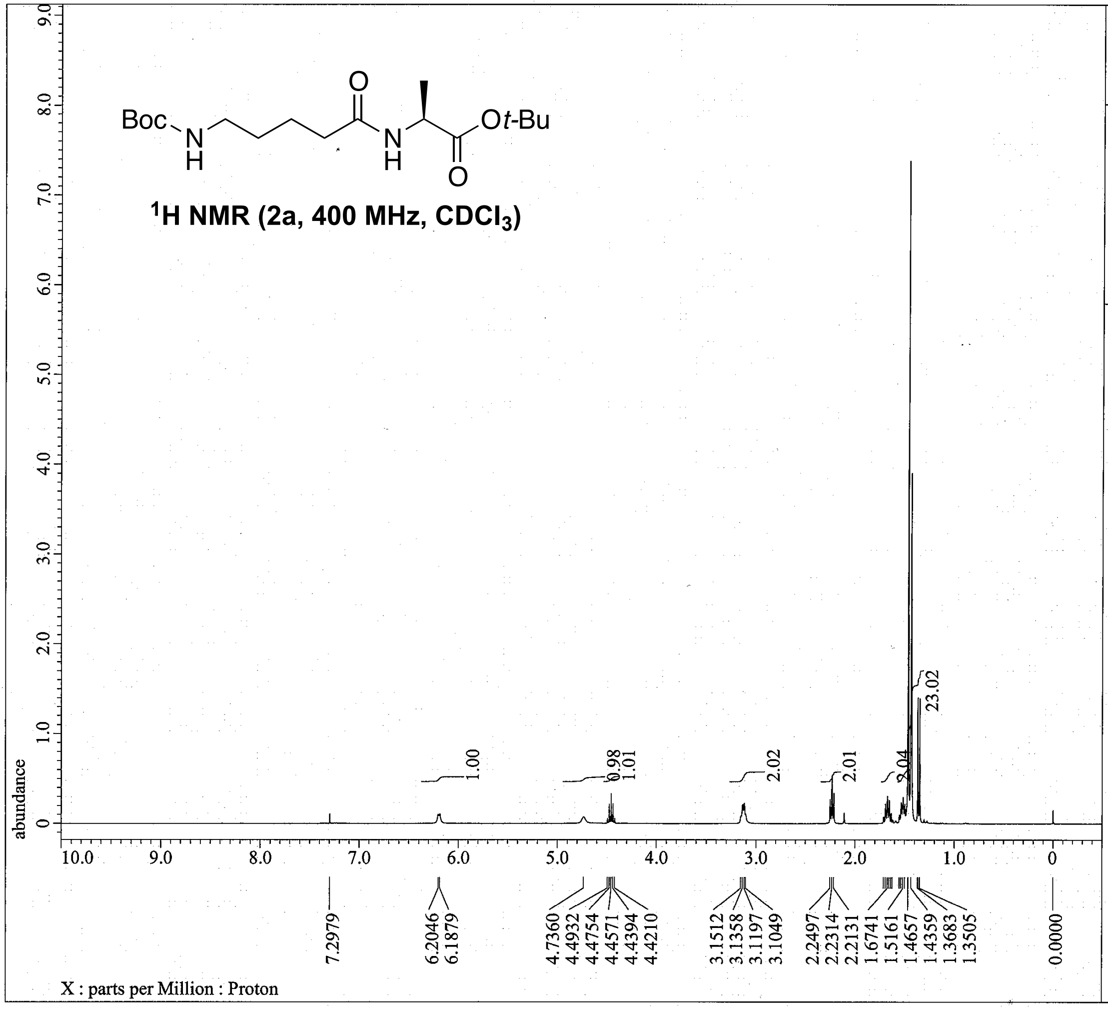


$^{13}C$ NMR (1k, 100 MHz, $CDCl_3$)

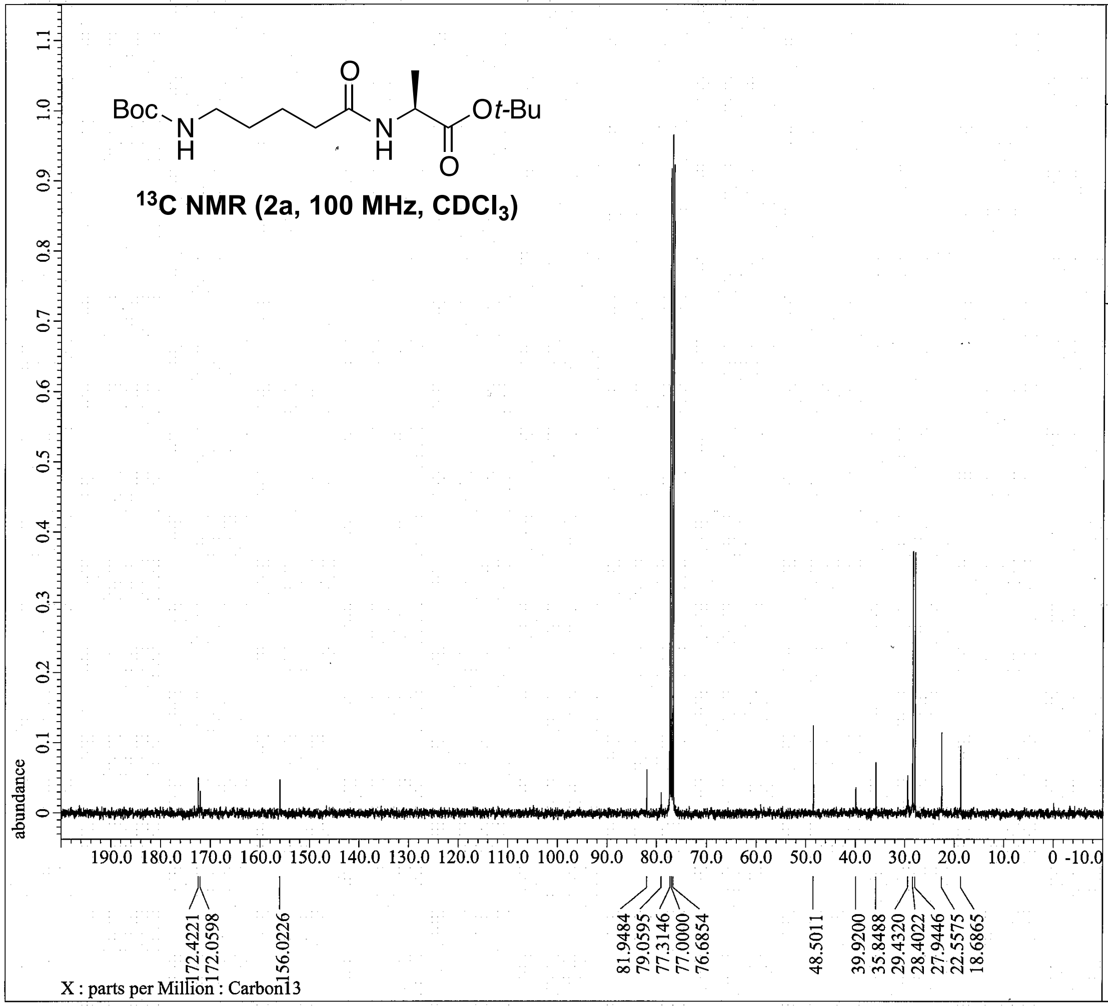
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-195-13C_E13C-1-1.jdf

Filename          = wm12-195-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-195-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 11-JAN-2022 11:44:22
Revision_Time     = 11-JAN-2022 11:52:53
Current_Time      = 11-JAN-2022 11:53:22

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (2a, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-142_E1H-1-1.jdf
```

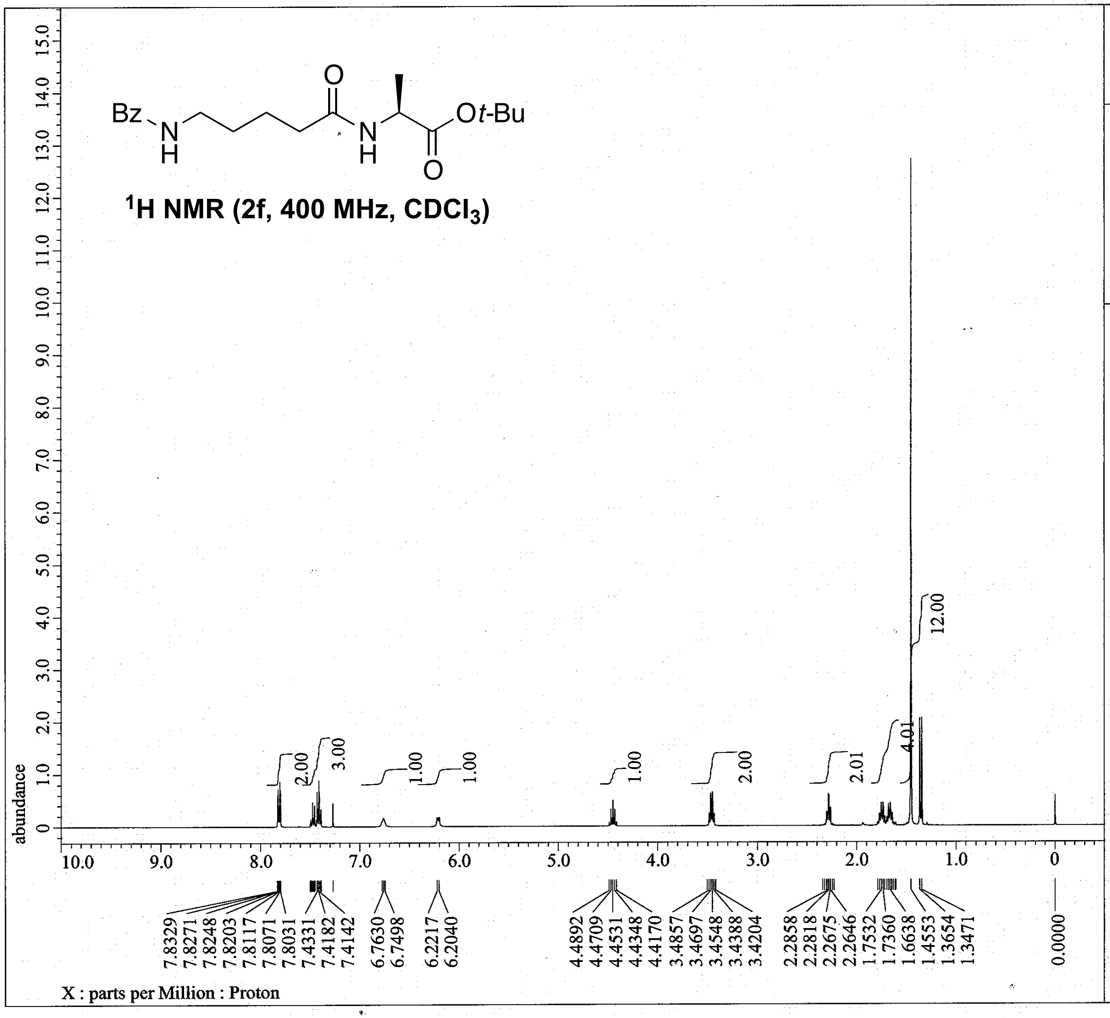
```
Filename           = wm11-142_E1H-1-6.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm11-142
Solvent            = CHLOROFORM-D
Creation_Time      = 24-MAR-2021 10:02:58
Revision_Time      = 24-MAR-2021 10:05:24
Current_Time       = 24-MAR-2021 10:05:46

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 20
Temp_Get           = 25[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



**$^{13}$C NMR (2a, 100 MHz, CDCl$_3$)**

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-142-1-13C_E13C-1-1.jdf
```

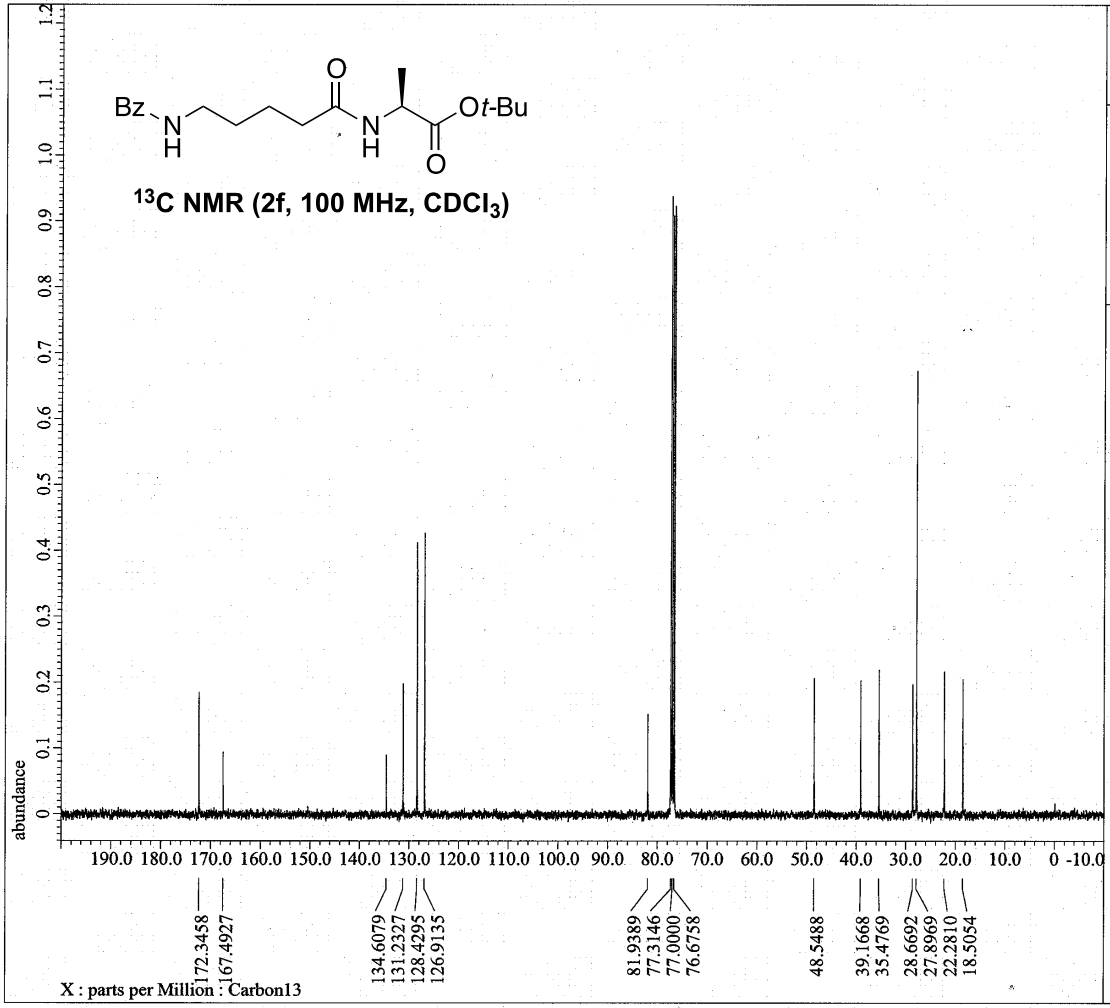
```
Filename          = wm11-142-1-13C_E13C-1-3.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm11-142-1-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 24-MAR-2021 10:28:19
Revision_Time     = 24-MAR-2021 10:50:45
Current_Time      = 24-MAR-2021 10:51:29

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.6[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (2f, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-23_E1H-1-1.jdf
```

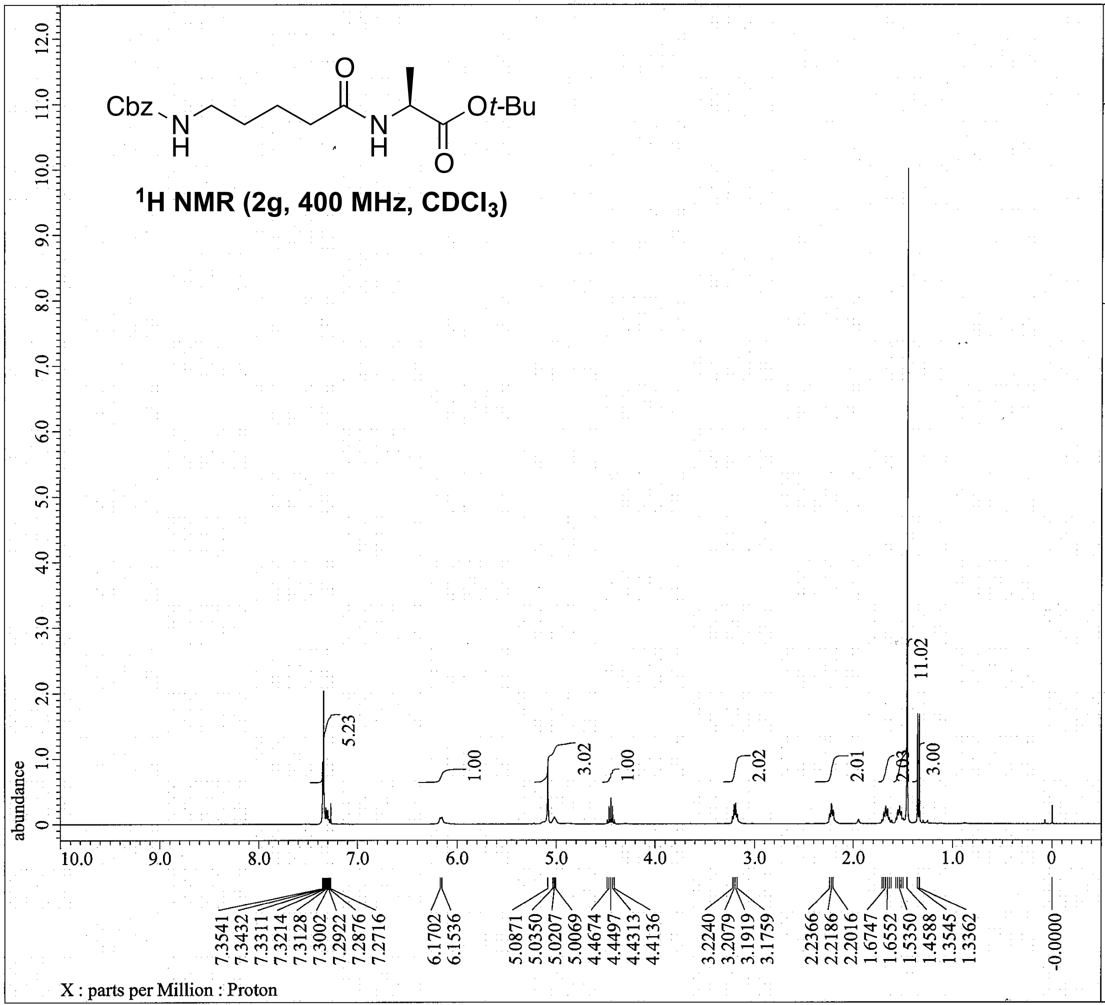
```
Filename            = wm13-23_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm13-23
Solvent             = CHLOROFORM-D
Creation_Time       = 24-FEB-2022 09:44:22
Revision_Time       = 24-FEB-2022 09:46:09
Current_Time        = 24-FEB-2022 09:46:25

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
Temp_Get            = 22.6[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (2f, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-23-13C_E13C-1-1.jdf
```

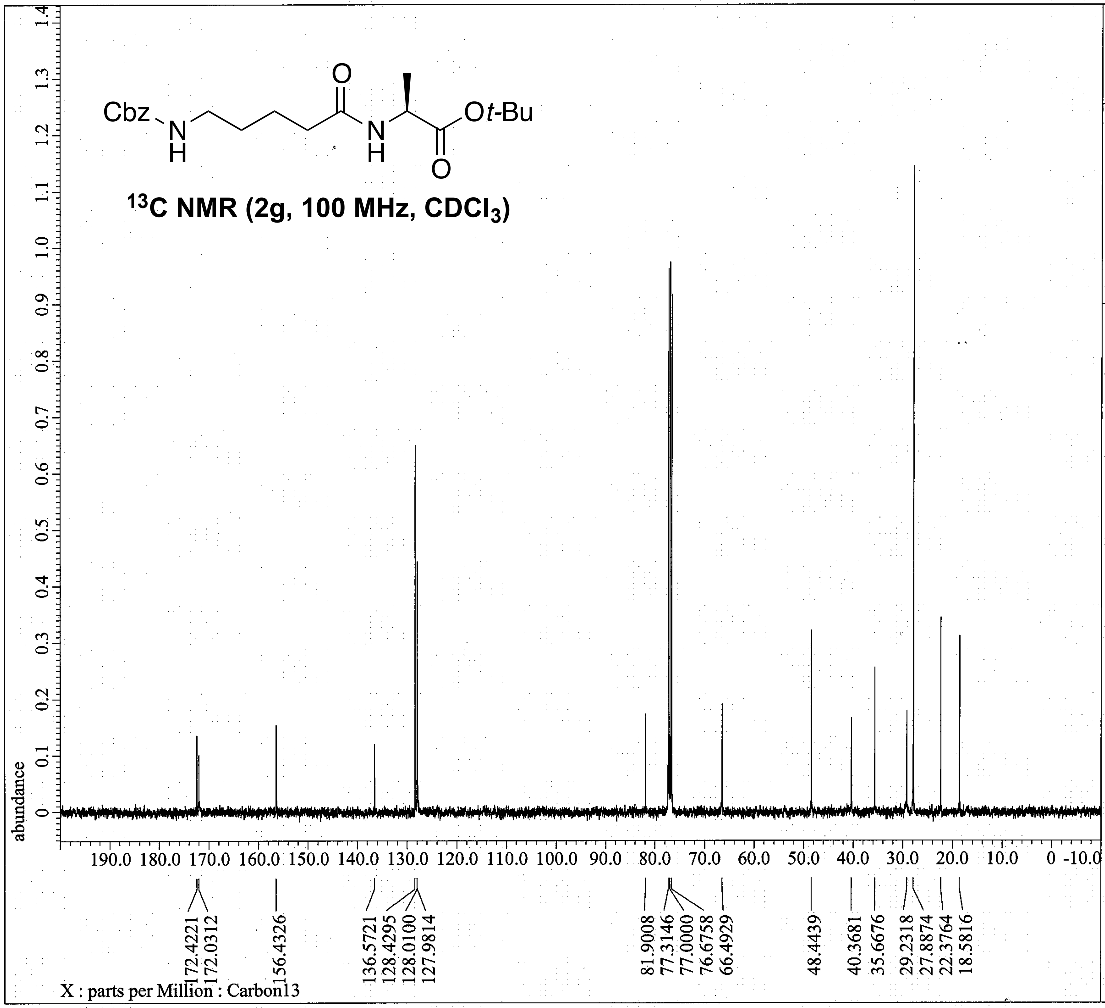
```
Filename            = wm13-23-13C_E13C-1-3.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm13-23-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 24-FEB-2022 09:46:29
Revision_Time       = 24-FEB-2022 10:07:56
Current_Time        = 24-FEB-2022 10:08:26

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = TRUE
Scans               = 512
Total_Scans         = 512

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.7[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (2g, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-172_E1H-1-1.jdf
```

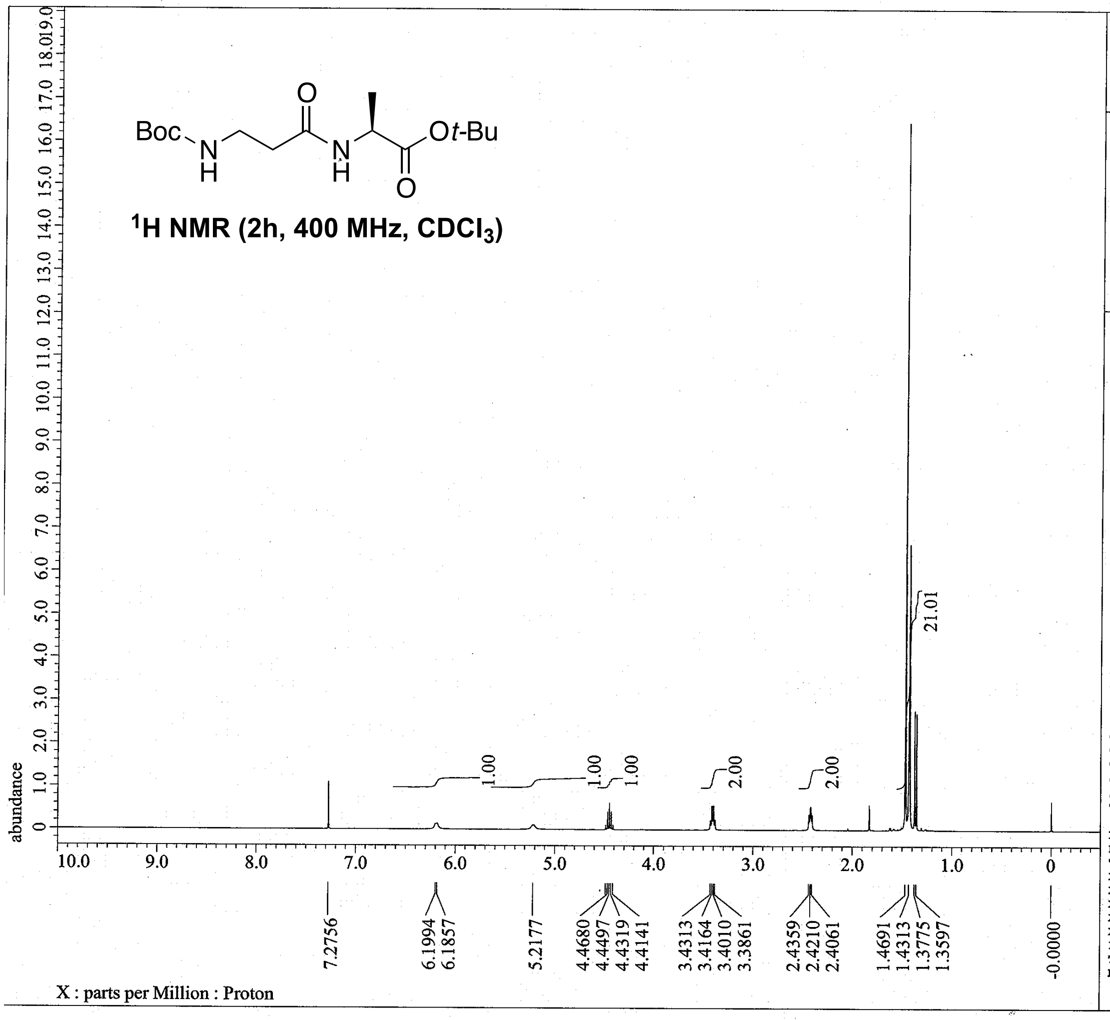
```
Filename            = wm11-172_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm11-172
Solvent             = CHLOROFORM-D
Creation_Time       = 23-APR-2021 08:14:53
Revision_Time       = 23-APR-2021 08:20:33
Current_Time        = 23-APR-2021 08:20:43

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 26
Temp_Get            = 22.5[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (2g, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-172-13C_E13C-1-1.jdf
```

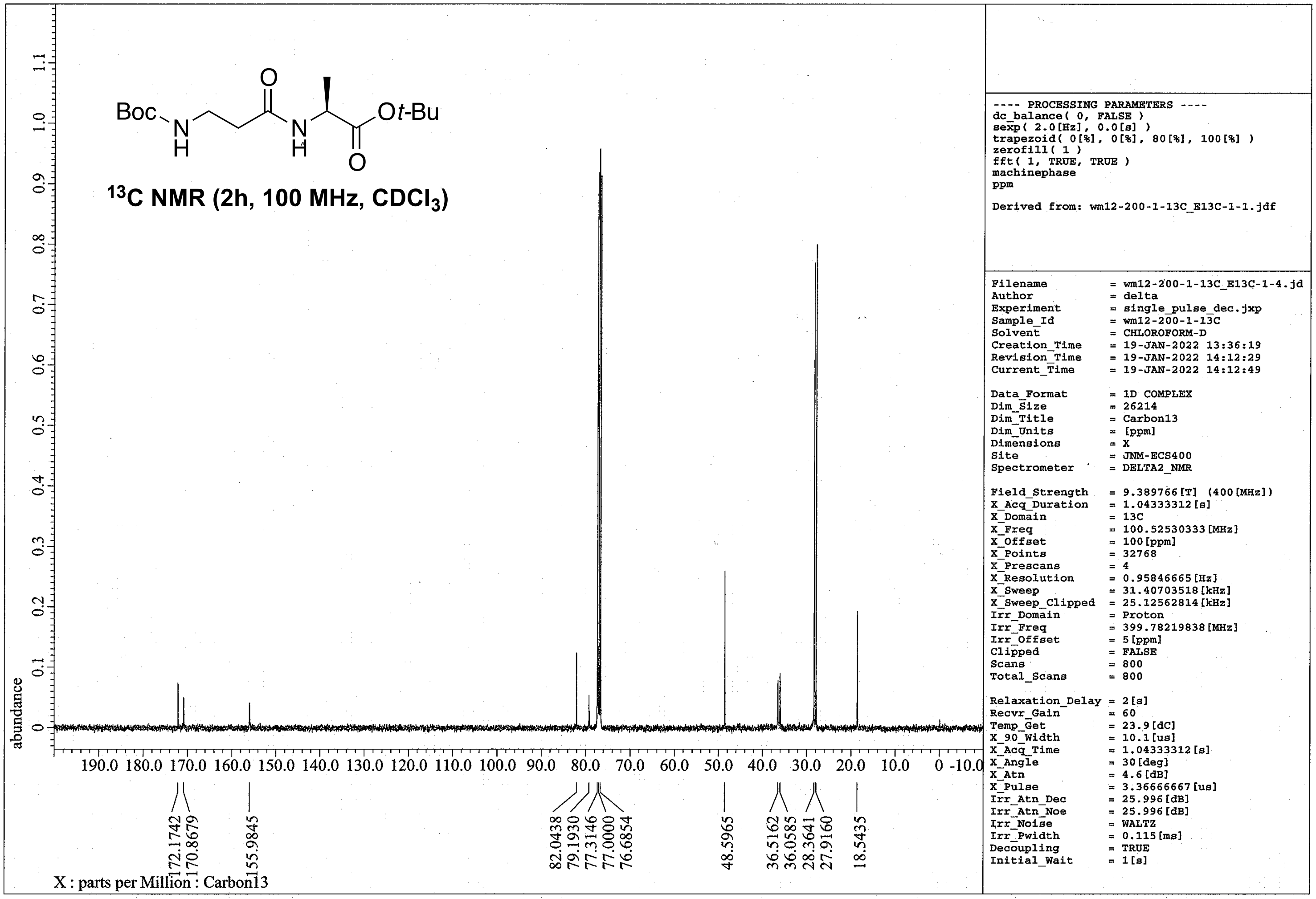
```
Filename           = wm11-172-13C_E13C-1-3.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm11-172-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 23-APR-2021 08:19:36
Revision_Time      = 23-APR-2021 08:30:13
Current_Time       = 23-APR-2021 08:30:31

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 281
Total_Scans        = 281

Relaxation_Delay = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.4[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (2h, 400 MHz, CDCl$_3$)

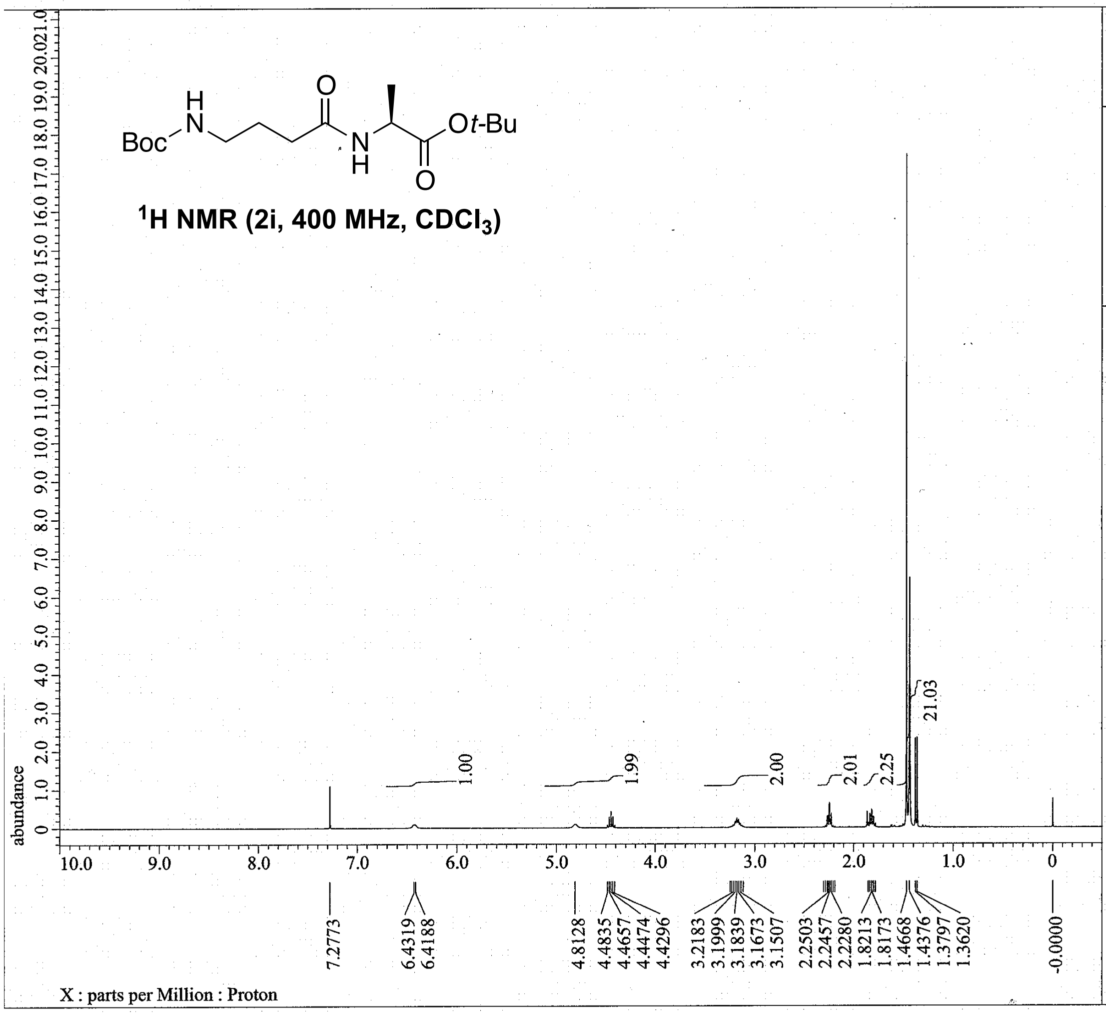
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-200-1_E1H-1-1.jdf
```

```
Filename          = wm12-200-1_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-200-1
Solvent           = CHLOROFORM-D
Creation_Time     = 19-JAN-2022 13:33:51
Revision_Time     = 19-JAN-2022 13:34:49
Current_Time      = 19-JAN-2022 13:35:09

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 24[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-200-1-13C_E13C-1-1.jdf
```

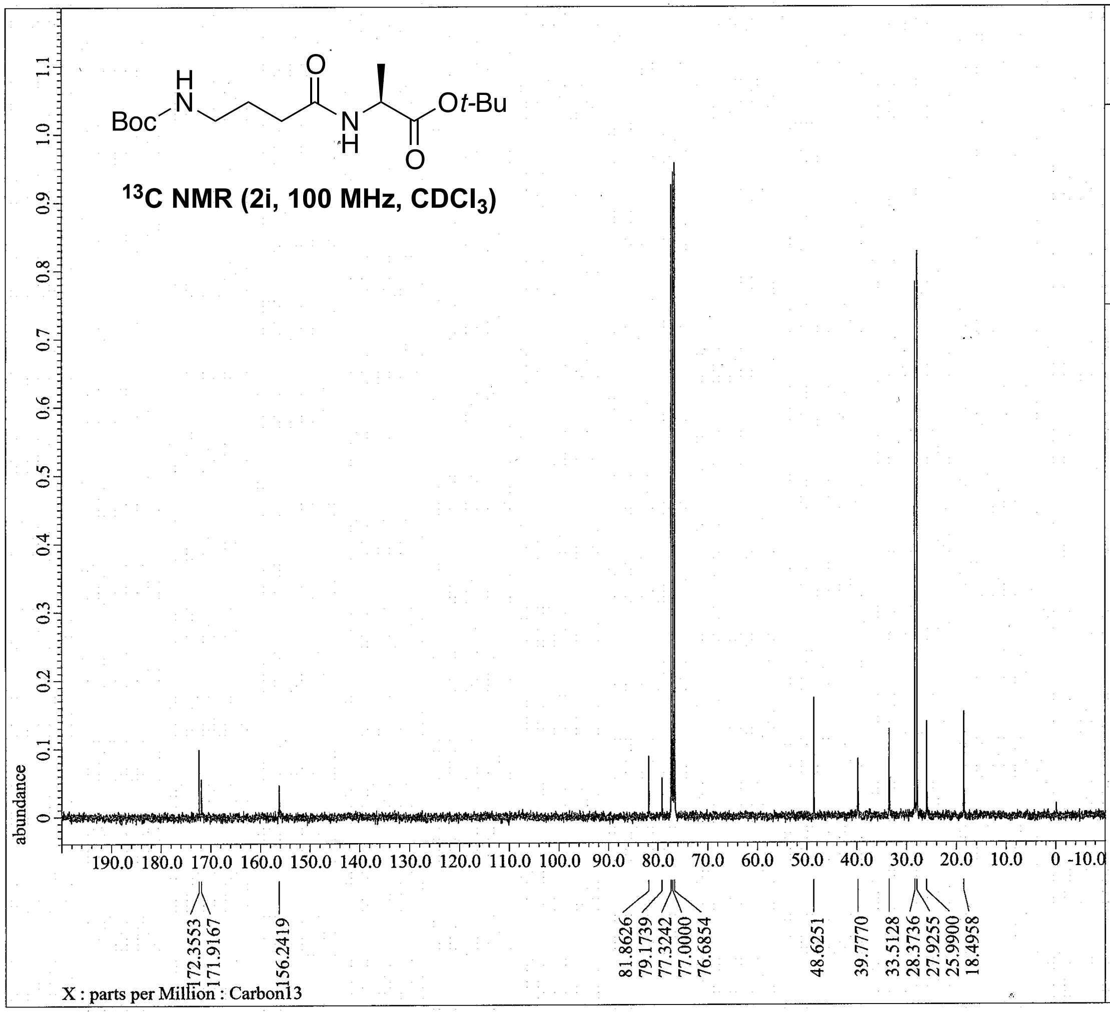
```
Filename           = wm12-200-1-13C_E13C-1-4.jd
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-200-1-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 19-JAN-2022 13:36:19
Revision_Time      = 19-JAN-2022 14:12:29
Current_Time       = 19-JAN-2022 14:12:49

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 800
Total_Scans        = 800

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.9[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (2i, 400 MHz, $CDCl_3$)

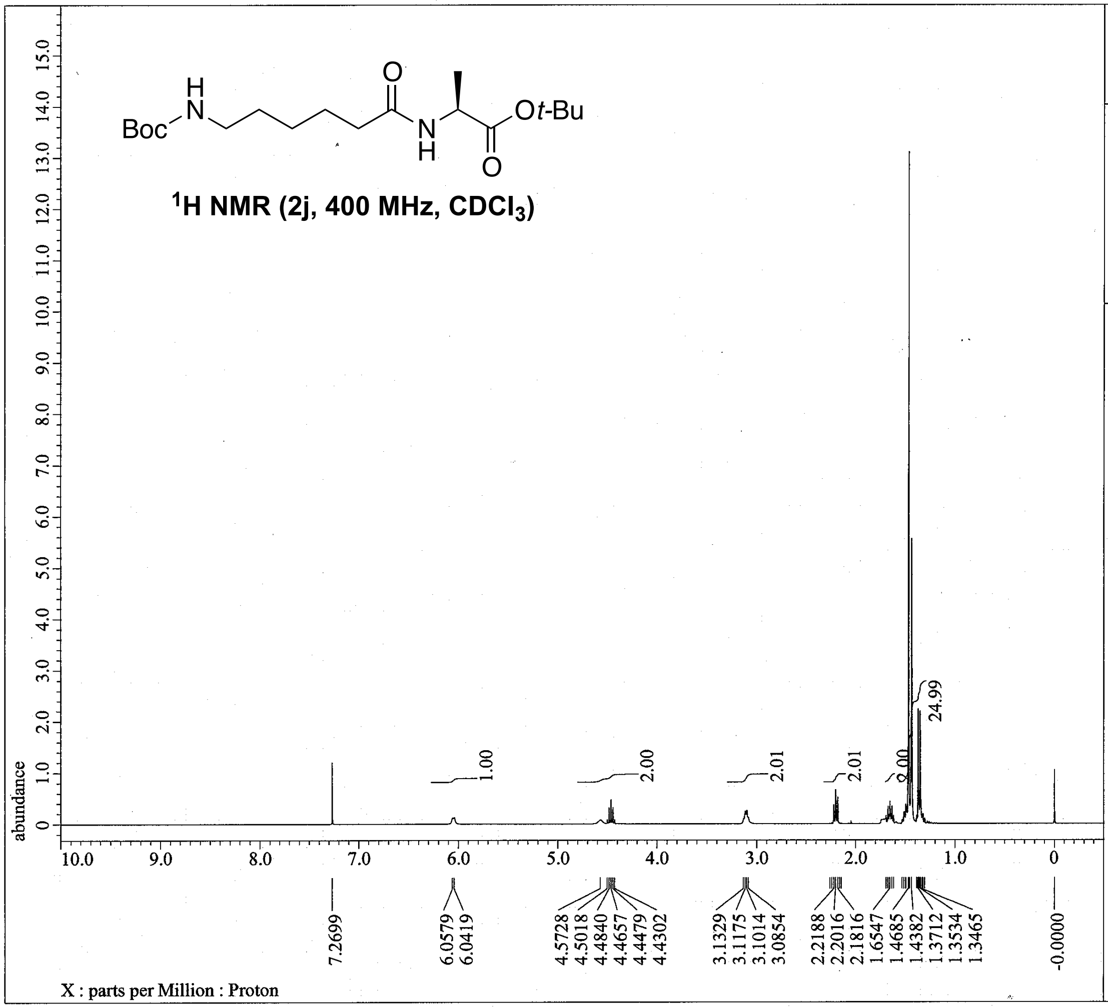
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-194-6_E1H-1-1.jdf
```

```
Filename          = wm12-194-6_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-194-6
Solvent           = CHLOROFORM-D
Creation_Time     =  7-JAN-2022 11:51:21
Revision_Time     =  7-JAN-2022 11:52:01
Current_Time      =  7-JAN-2022 11:52:13

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 48
Temp_Get          = 23.3[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-194-6-13C_E13C-1-1.jdf
```

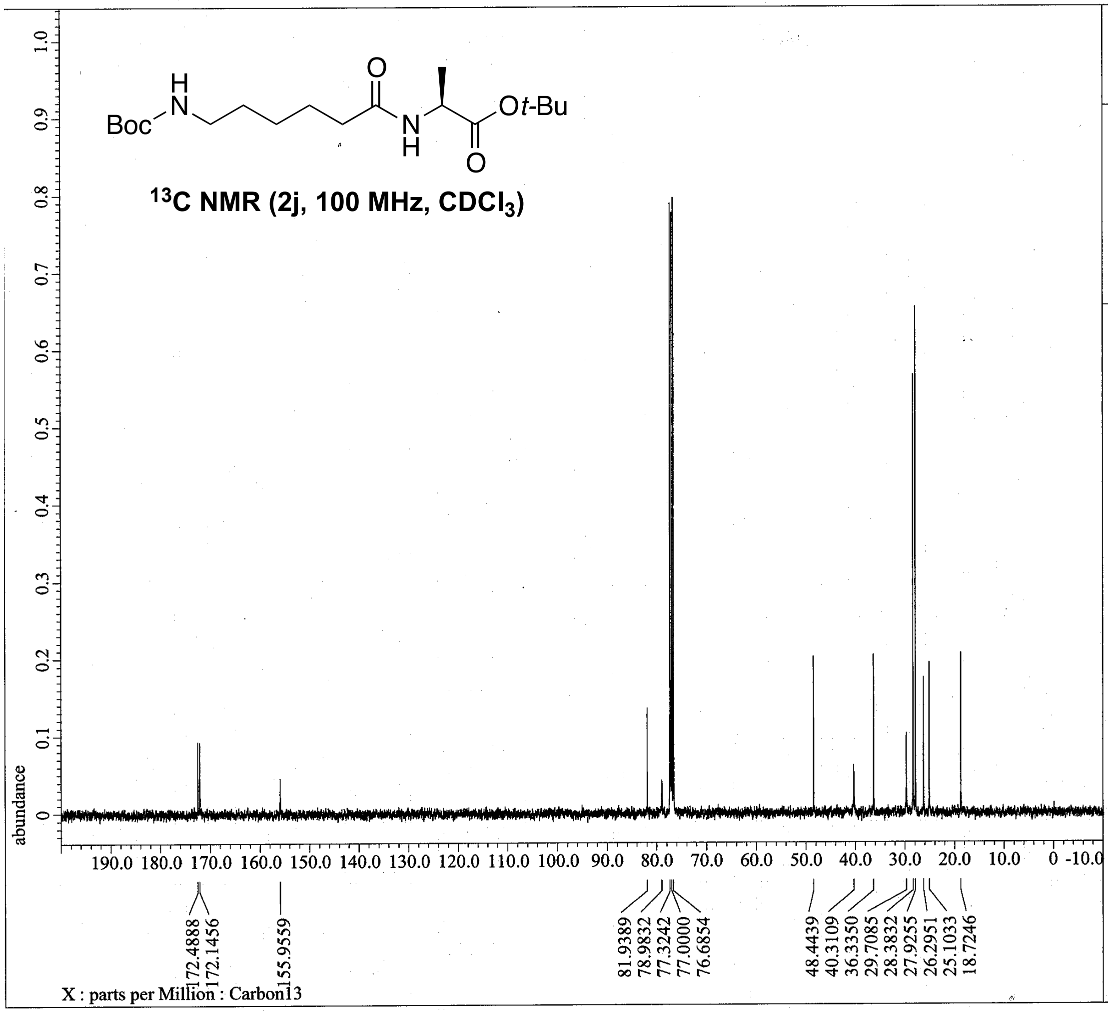
```
Filename          = wm12-194-6-13C_E13C-1-4.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-194-6-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  7-JAN-2022 12:14:24
Revision_Time     =  7-JAN-2022 12:36:07
Current_Time      =  7-JAN-2022 12:36:32

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.3[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (2j, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-191_E1H-2-1.jdf
```

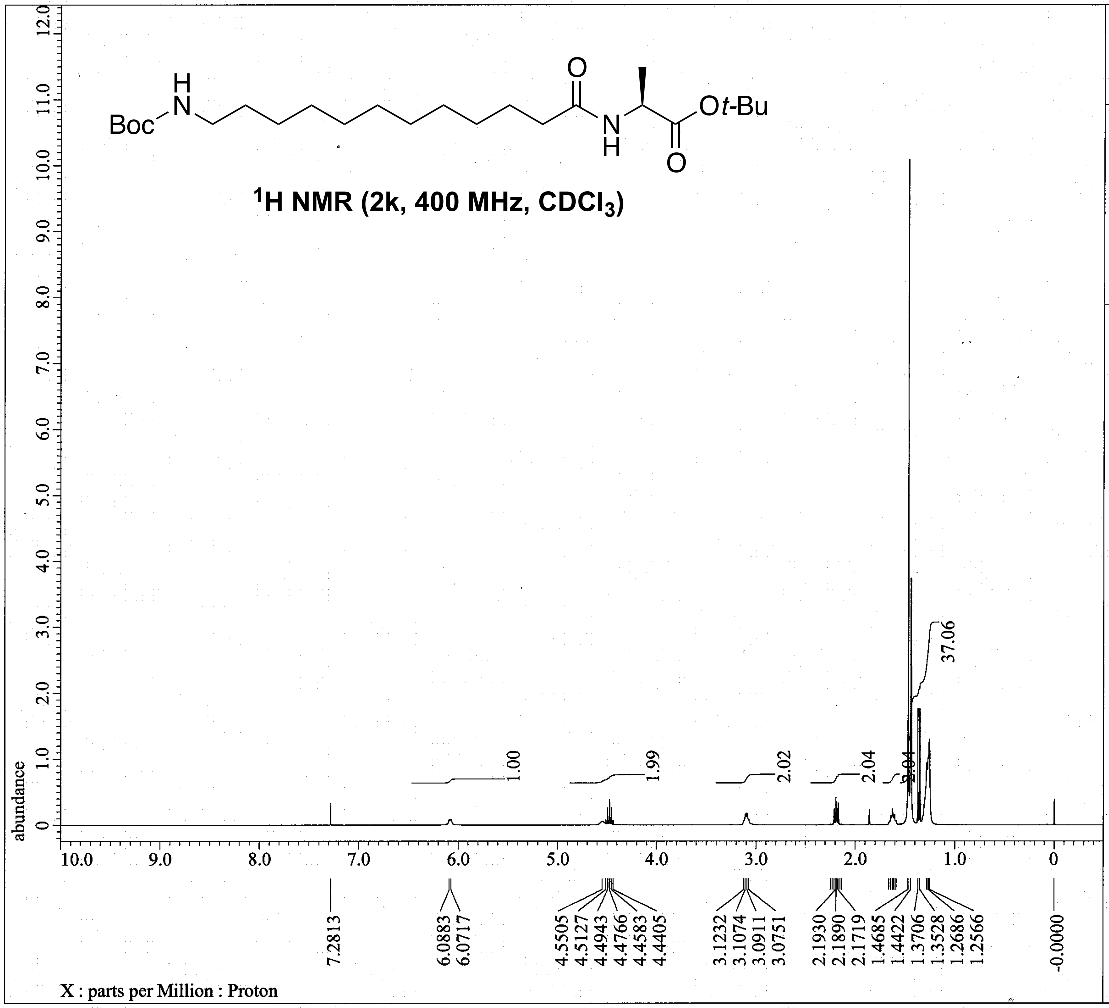
```
Filename           = wm12-191_E1H-2-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-191
Solvent            = CHLOROFORM-D
Creation_Time      = 12-JAN-2022 10:58:00
Revision_Time      = 12-JAN-2022 11:00:27
Current_Time       = 12-JAN-2022 11:00:37

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 34
Temp_Get           = 23.8[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (2j, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-191-13C_E13C-1-1.jdf
```

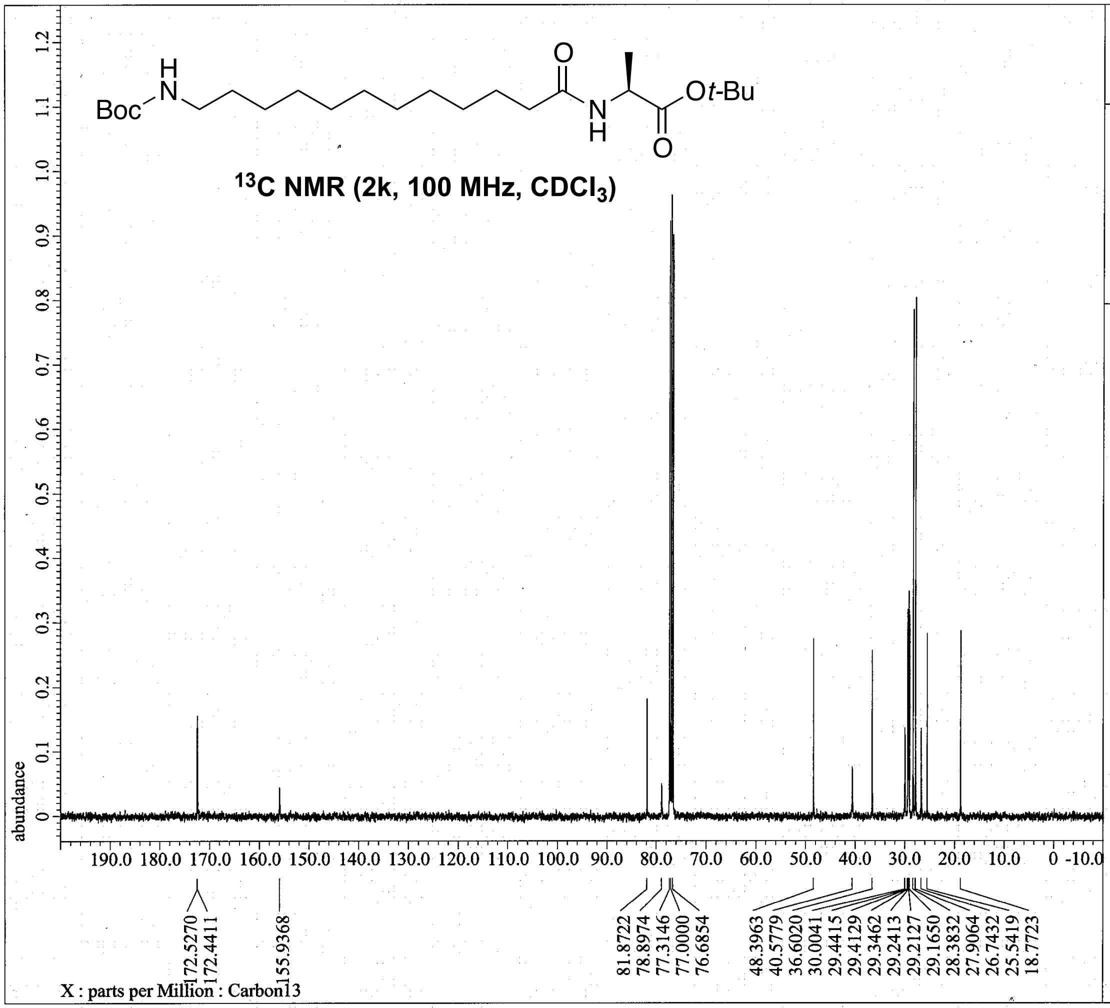
```
Filename          = wm12-191-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-191-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JAN-2022 10:23:03
Revision_Time     = 12-JAN-2022 10:46:41
Current_Time      = 12-JAN-2022 10:47:06

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.6[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (2k, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-198_E1H-1-1.jdf
```

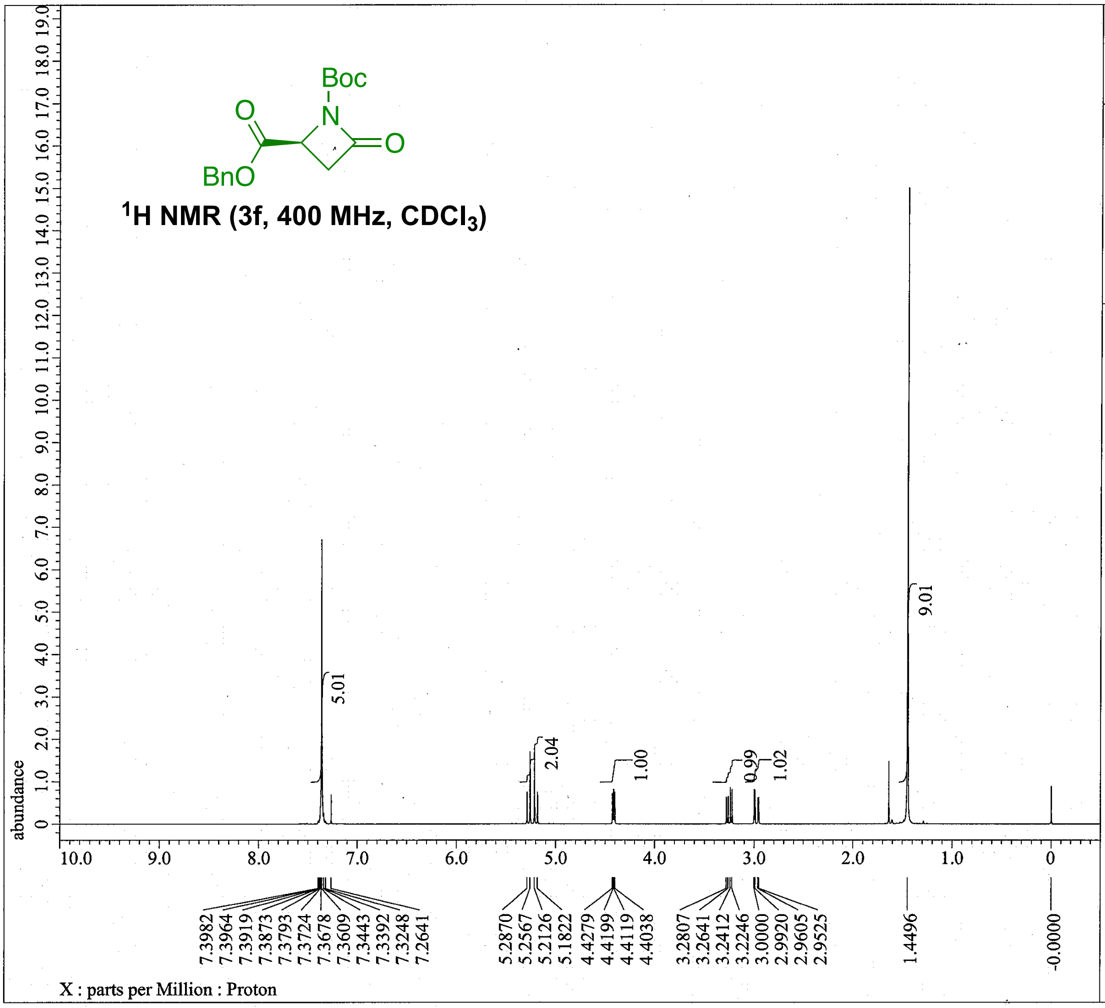
```
Filename           = wm12-198_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-128
Solvent            = CHLOROFORM-D
Creation_Time      = 13-JAN-2022 15:23:21
Revision_Time      = 13-JAN-2022 16:57:45
Current_Time       = 13-JAN-2022 16:58:00

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 24
Temp_Get           = 23.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (2k, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-128-13C_E13C-1-1.jdf
```

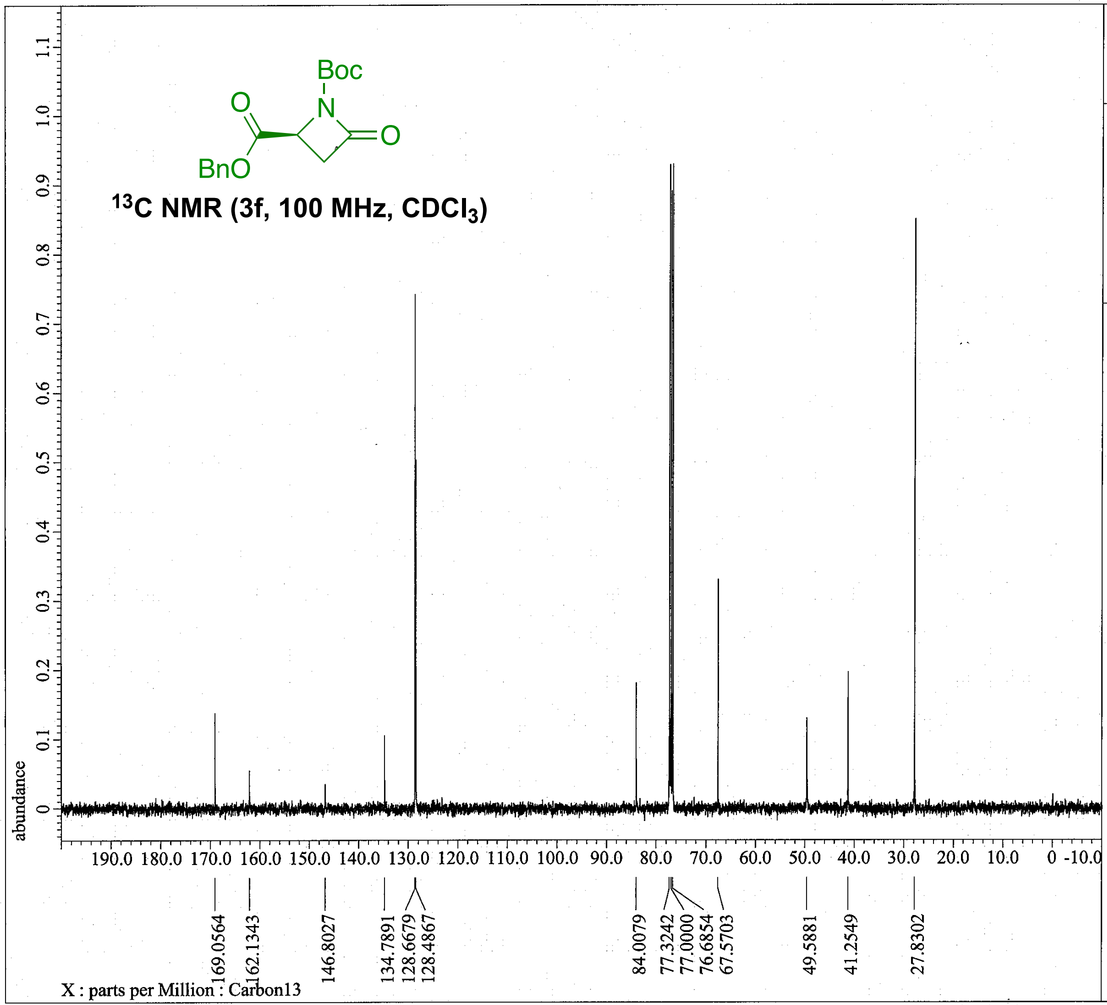
```
Filename          = wm12-128-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-128-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 13-JAN-2022 15:27:41
Revision_Time     = 13-JAN-2022 15:53:35
Current_Time      = 13-JAN-2022 15:54:13

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 600
Total_Scans       = 600

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.5[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (3f, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-44_E1H-1-1.jdf
```

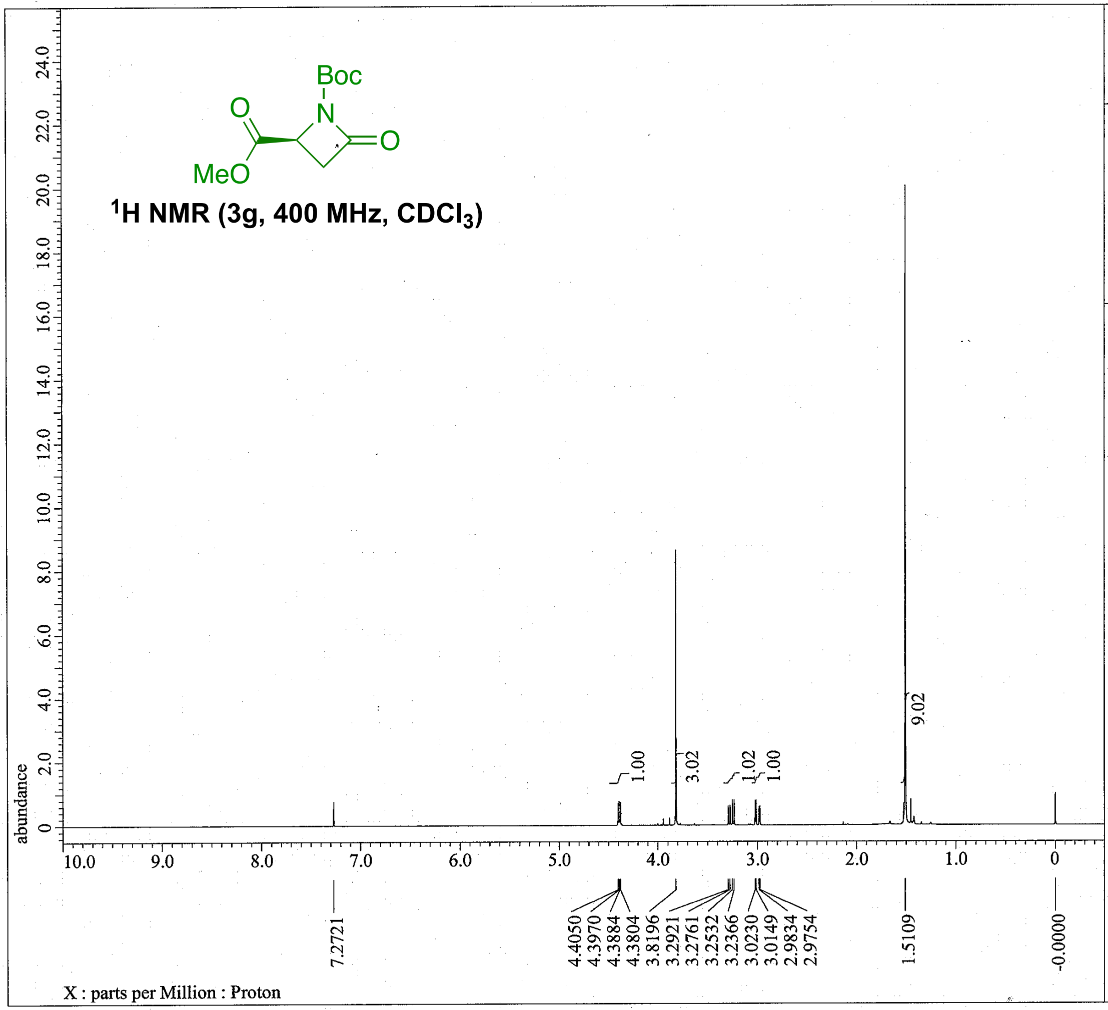
```
Filename            = wm12-44_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-44
Solvent             = CHLOROFORM-D
Creation_Time       =  8-JUL-2021 13:43:21
Revision_Time       =  8-JUL-2021 13:43:34
Current_Time        =  8-JUL-2021 13:43:54

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 32
Temp_Get            = 23.9[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (3f, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-44-13C_E13C-1-1.jdf
```

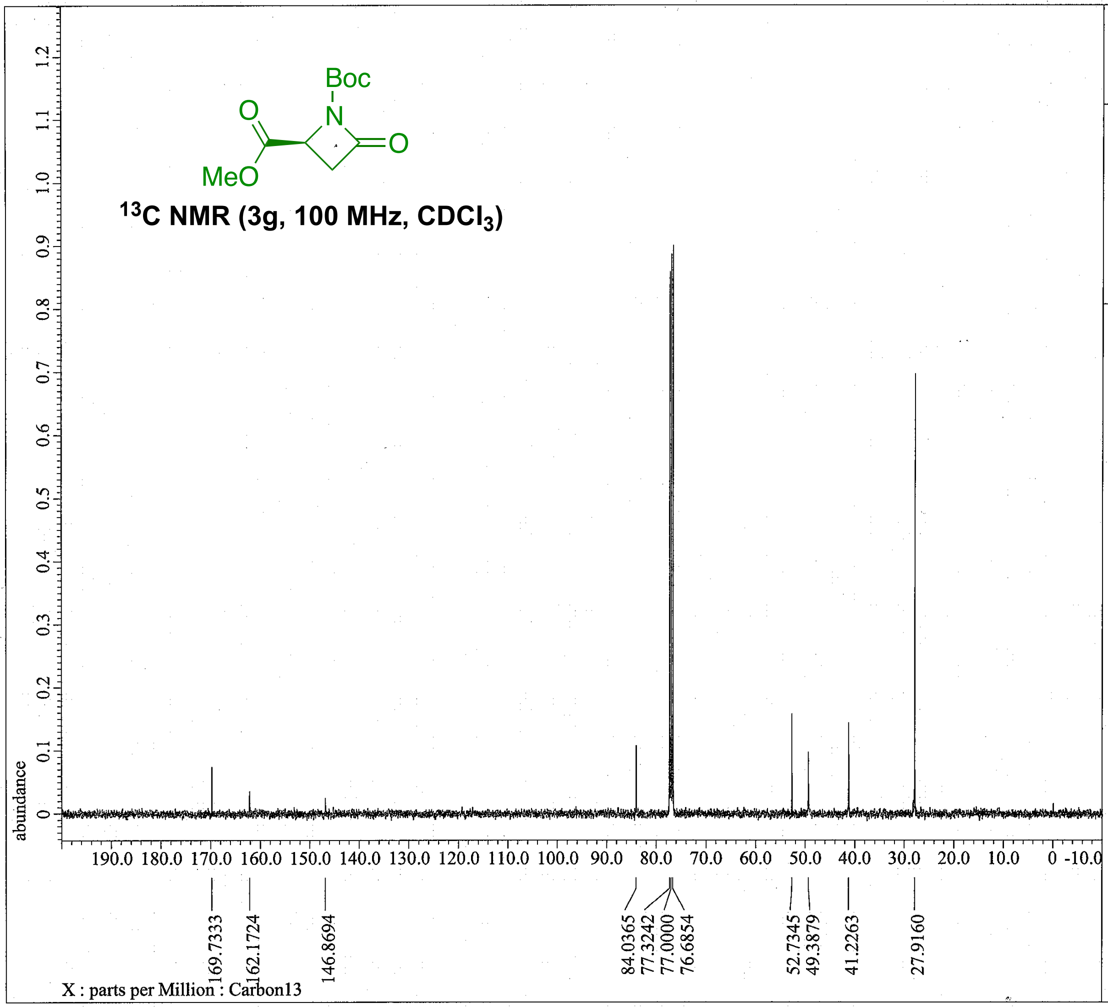
```
Filename          = wm12-44-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-44-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  8-JUL-2021 13:45:29
Revision_Time     =  8-JUL-2021 13:56:00
Current_Time      =  8-JUL-2021 13:57:01

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 281
Total_Scans       = 281

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.5[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (3g, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-88_E1H-1-1.jdf
```

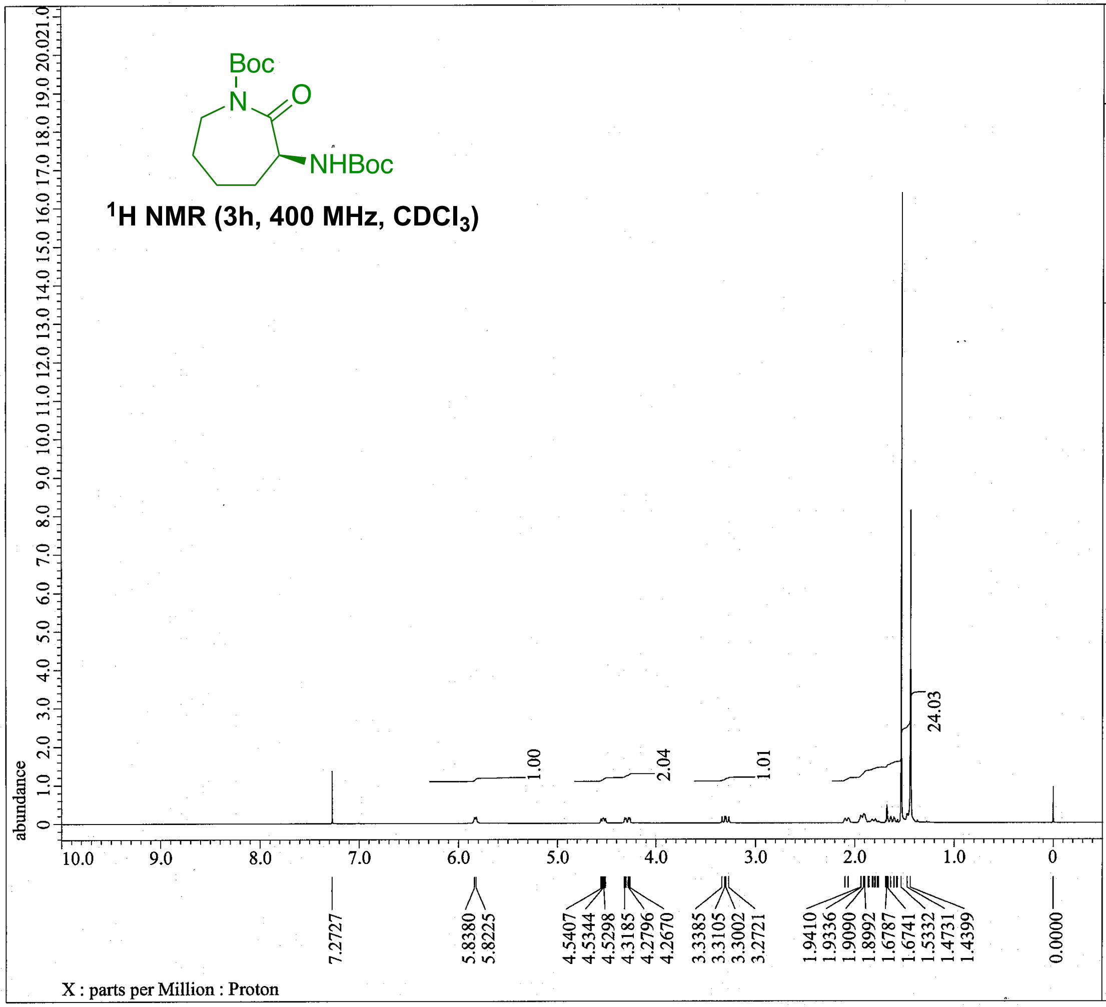
```
Filename          = wm12-88_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-88
Solvent           = CHLOROFORM-D
Creation_Time     = 10-SEP-2021 14:29:47
Revision_Time     = 10-SEP-2021 14:28:45
Current_Time      = 10-SEP-2021 14:28:55

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 5
Total_Scans       = 5

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 22.6[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



<sup></sup>$^{13}$C NMR (3g, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-88-13C_E13C-1-1.jdf
```

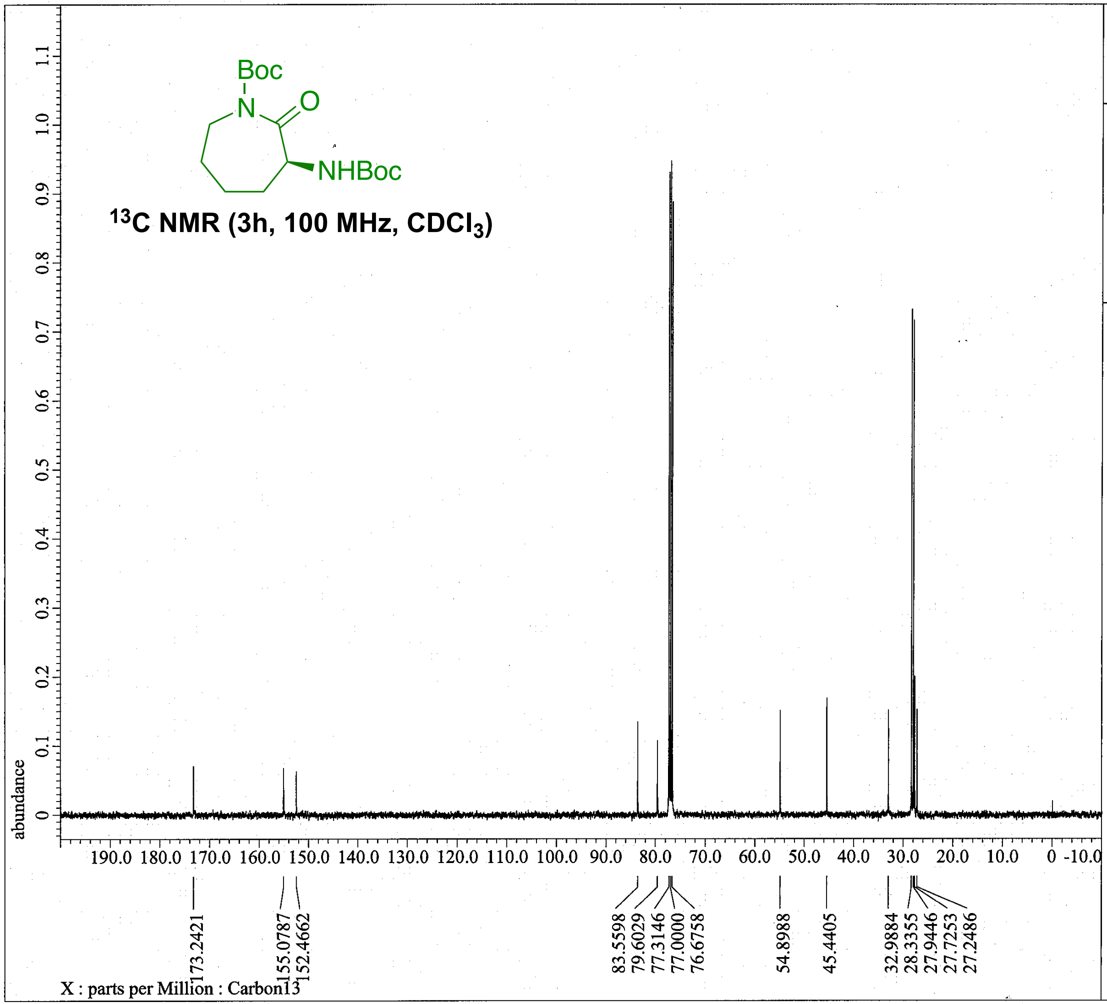
```
Filename           = wm12-88-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-88-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 10-SEP-2021 14:31:26
Revision_Time      = 10-SEP-2021 14:53:12
Current_Time       = 10-SEP-2021 14:53:27

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 512
Total_Scans        = 512

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 24.2[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (3h, 400 MHz, $CDCl_3$)

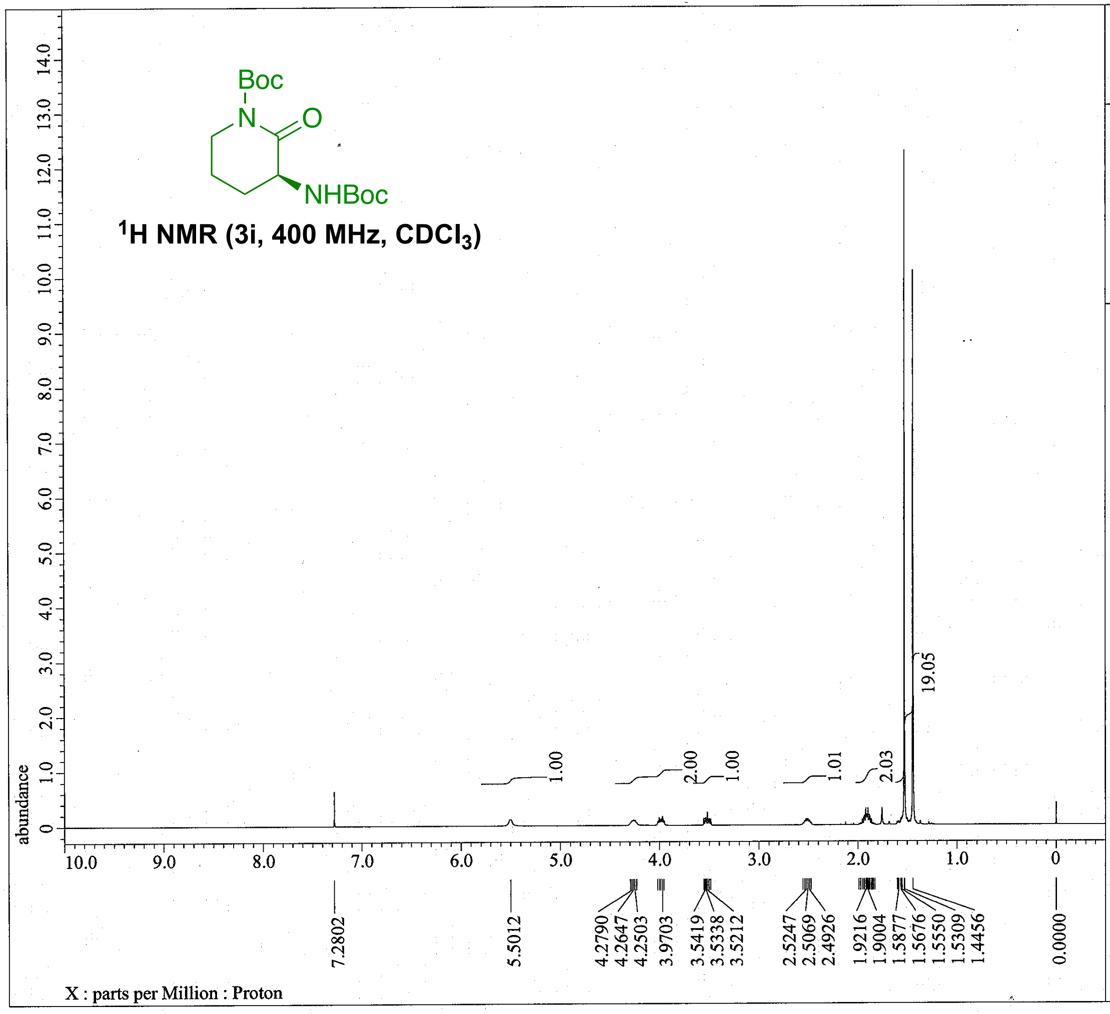
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-71_E1H-1-1.jdf
```

```
Filename           = wm12-71_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-71
Solvent            = CHLOROFORM-D
Creation_Time      =  6-AUG-2021 13:17:15
Revision_Time      =  6-AUG-2021 15:27:27
Current_Time       =  6-AUG-2021 15:27:38

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 30
Temp_Get           = 23.6[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-71-13C_E13C-1-1.jdf
```

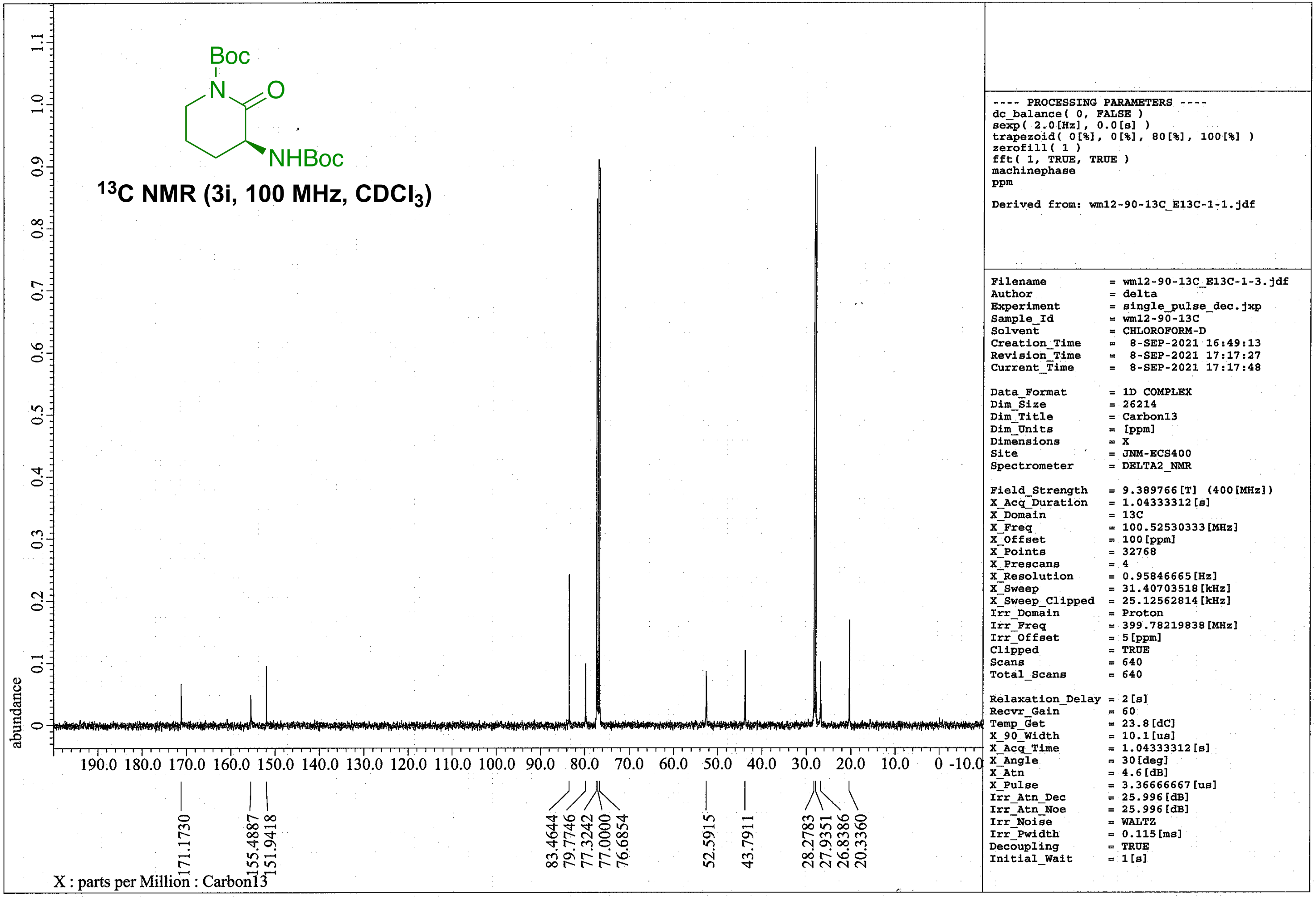
```
Filename           = wm12-71-13C_E13C-1-5.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-71-13C
Solvent            = CHLOROFORM-D
Creation_Time      =  6-AUG-2021 13:19:21
Revision_Time      =  6-AUG-2021 14:07:59
Current_Time       =  6-AUG-2021 14:08:20

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = TRUE
Scans              = 1024
Total_Scans        = 1024

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.6[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (3i, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-90_E1H-1-1.jdf
```

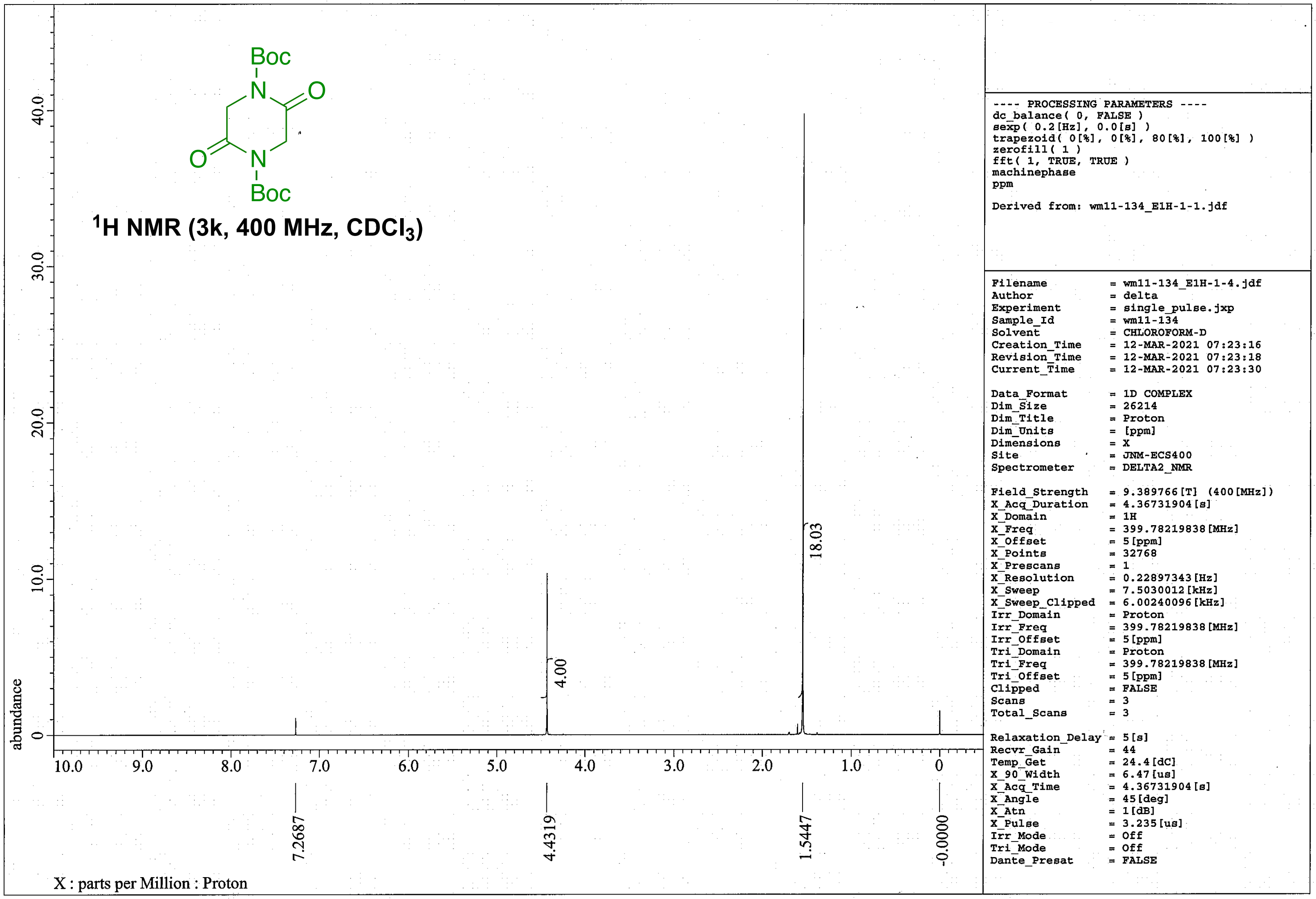
```
Filename           = wm12-90_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-90
Solvent            = CHLOROFORM-D
Creation_Time      =  8-SEP-2021 16:45:53
Revision_Time      =  8-SEP-2021 17:15:44
Current_Time       =  8-SEP-2021 17:15:58

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 23.9[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (3i, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-90-13C_E13C-1-1.jdf
```

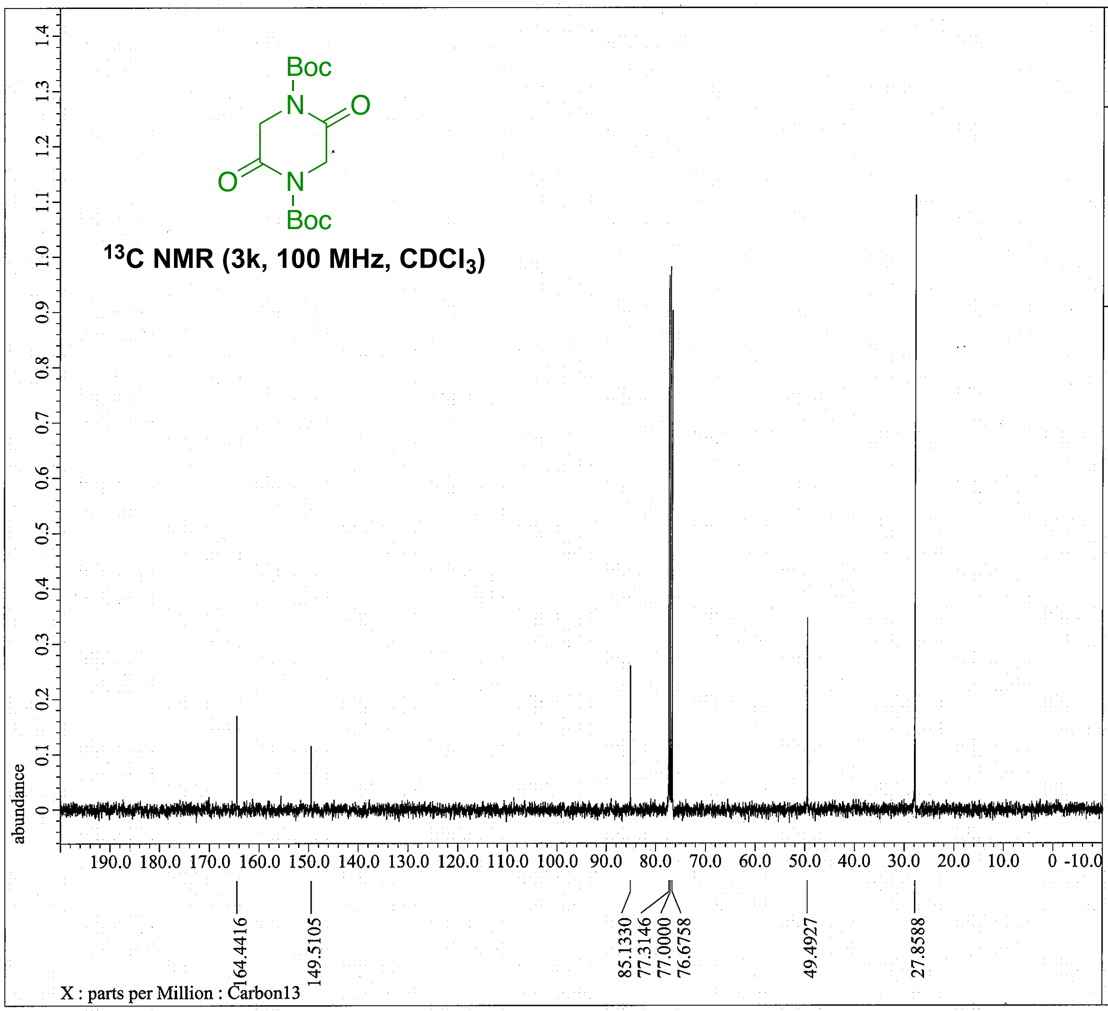
```
Filename          = wm12-90-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-90-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  8-SEP-2021 16:49:13
Revision_Time     =  8-SEP-2021 17:17:27
Current_Time      =  8-SEP-2021 17:17:48

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 640
Total_Scans       = 640

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1H$ NMR (3k, 400 MHz, $CDCl_3$)

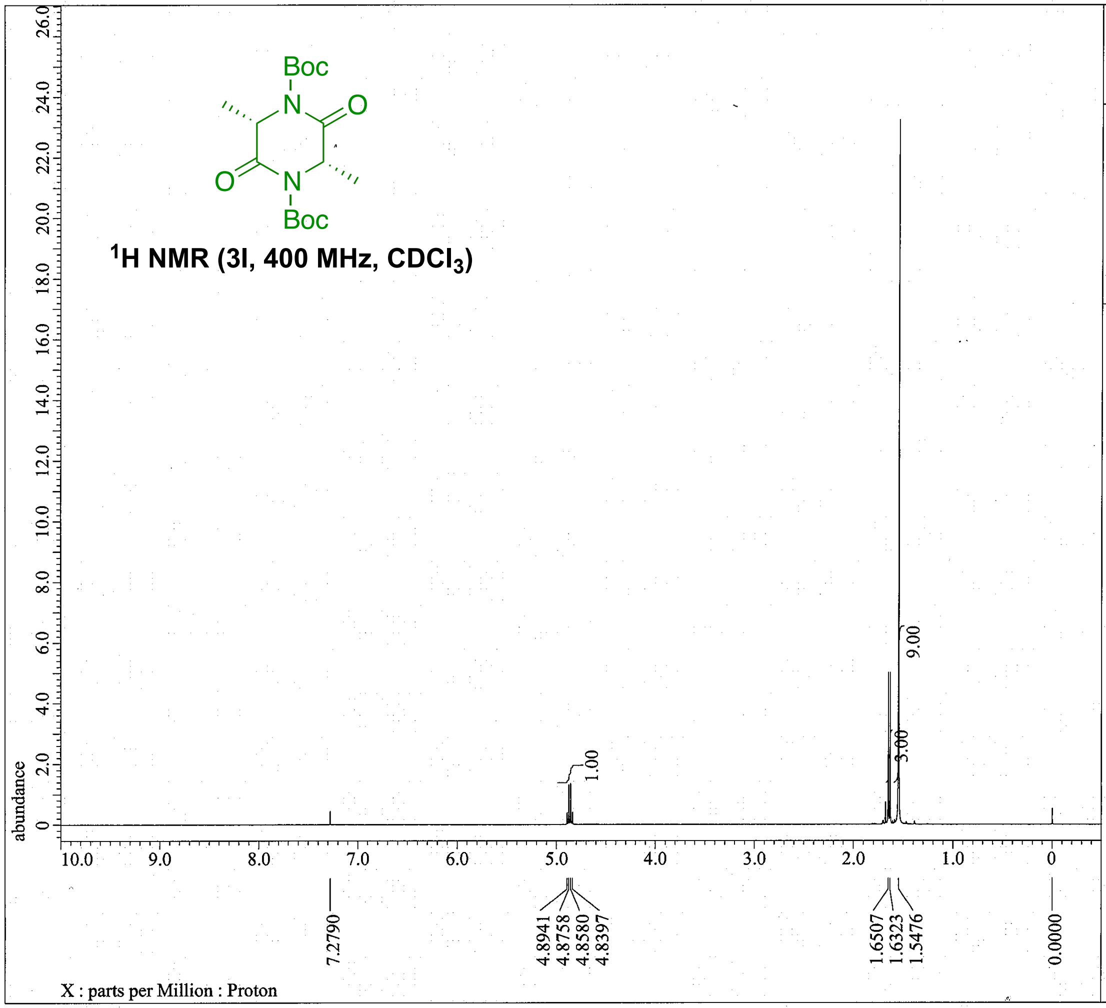
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-134_E1H-1-1.jdf
```

```
Filename            = wm11-134_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm11-134
Solvent             = CHLOROFORM-D
Creation_Time       = 12-MAR-2021 07:23:16
Revision_Time       = 12-MAR-2021 07:23:18
Current_Time        = 12-MAR-2021 07:23:30

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 3
Total_Scans         = 3

Relaxation_Delay    = 5[s]
Recvr_Gain          = 44
Temp_Get            = 24.4[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-134-13C_E13C-1-1.jdf
```

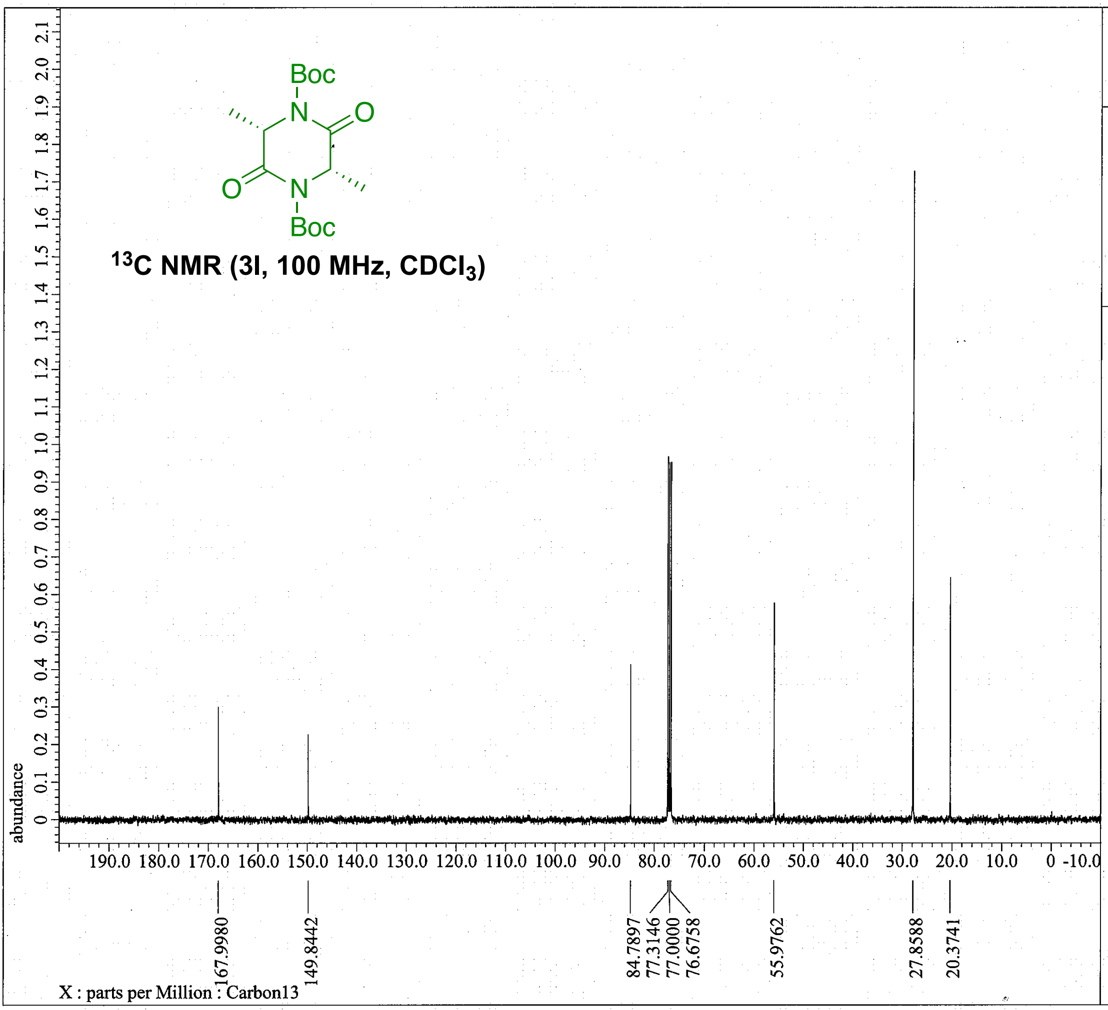
```
Filename            = wm11-134-13C_E13C-1-3.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm11-134-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 12-MAR-2021 07:26:22
Revision_Time       = 12-MAR-2021 07:29:42
Current_Time        = 12-MAR-2021 07:29:58

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 129
Total_Scans         = 129

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 24.7[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1H$ NMR (3l, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-181-1_E1H-1-1.jdf
```

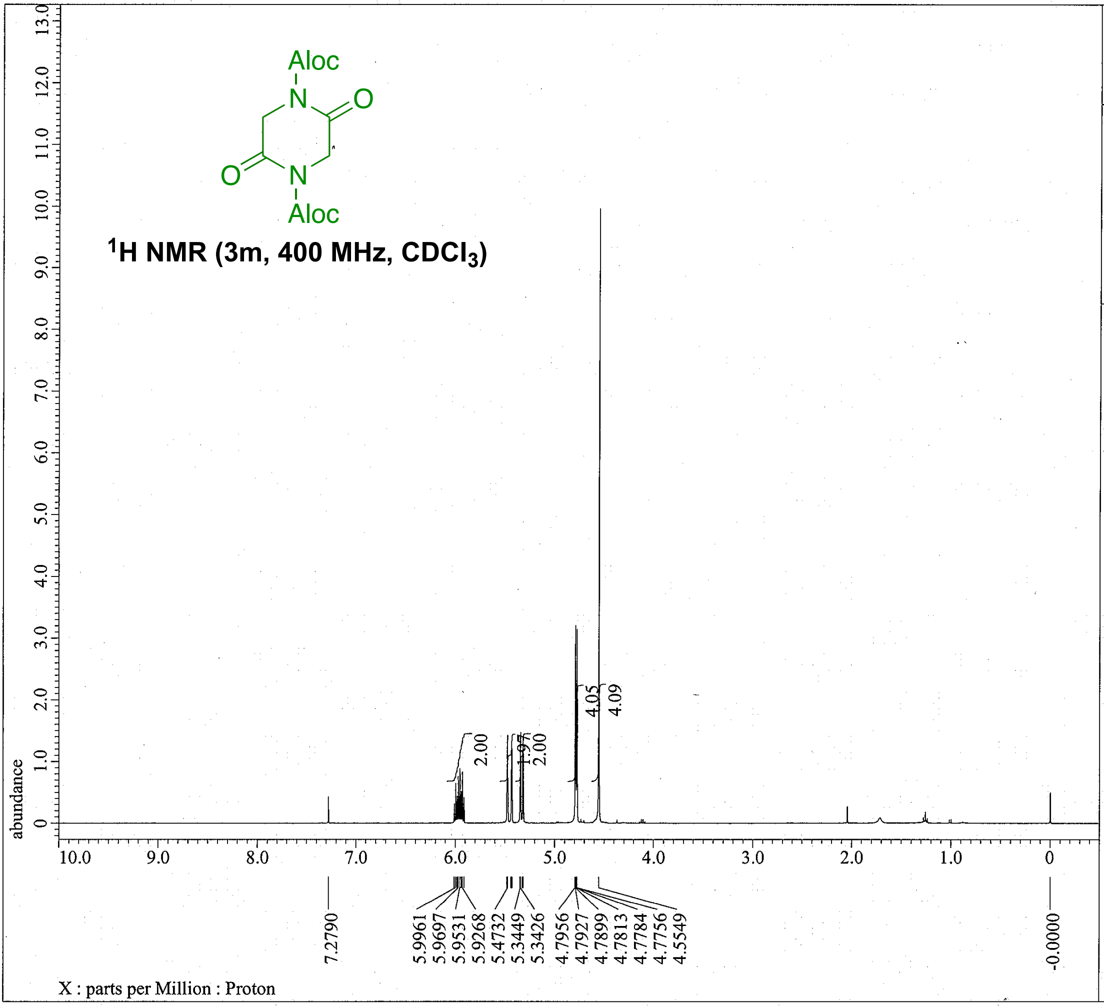
```
Filename          = wm11-181-1_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm11-181-1
Solvent           = CHLOROFORM-D
Creation_Time     = 28-APR-2021 15:31:01
Revision_Time     = 28-APR-2021 15:30:12
Current_Time      = 28-APR-2021 15:30:28

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 22.1[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (3l, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-181-1-13C_E13C-1-1.jdf
```

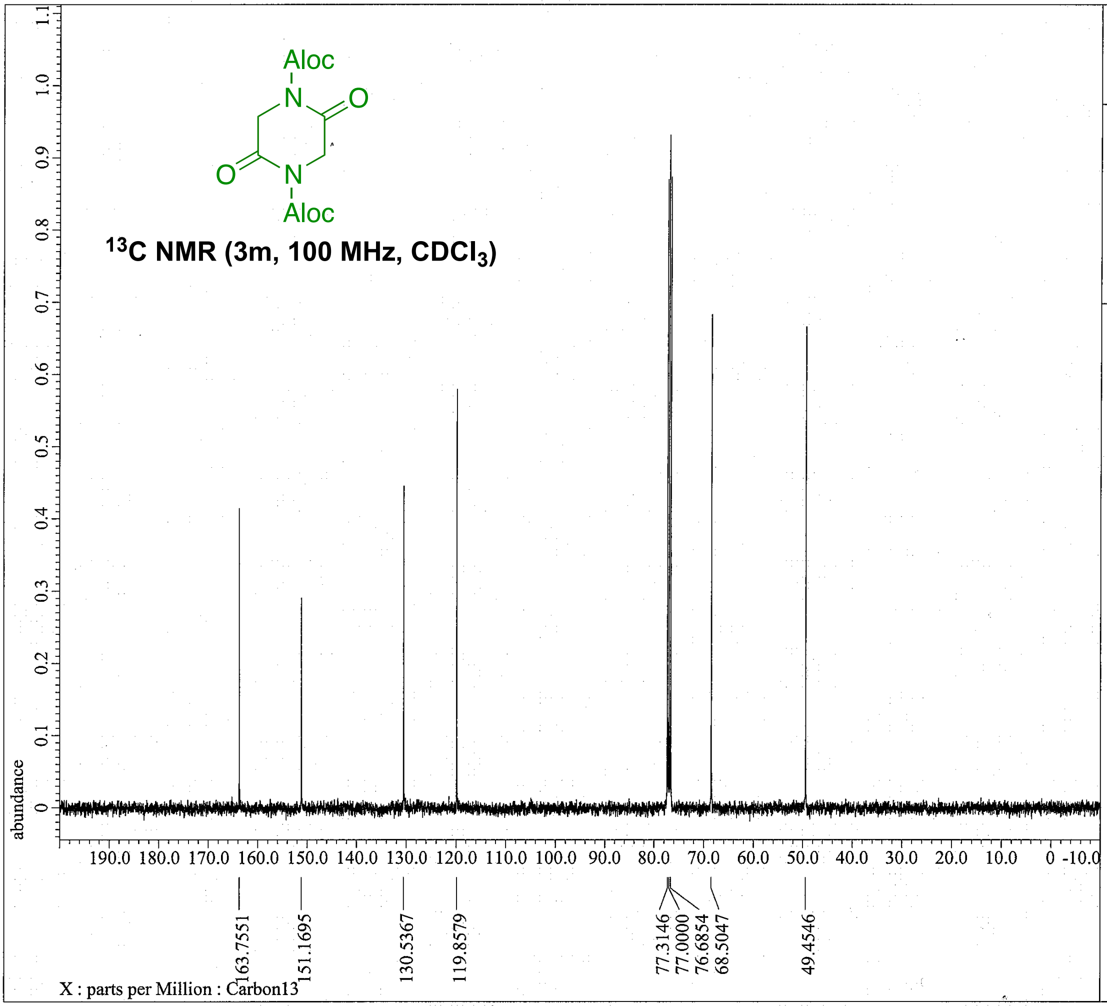
```
Filename           = wm11-181-1-13C_E13C-1-3.jd
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm11-181-1-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 28-APR-2021 15:35:14
Revision_Time      = 28-APR-2021 15:44:17
Current_Time       = 28-APR-2021 15:44:37

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 257
Total_Scans        = 257

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.2[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (3m, 400 MHz, $CDCl_3$)

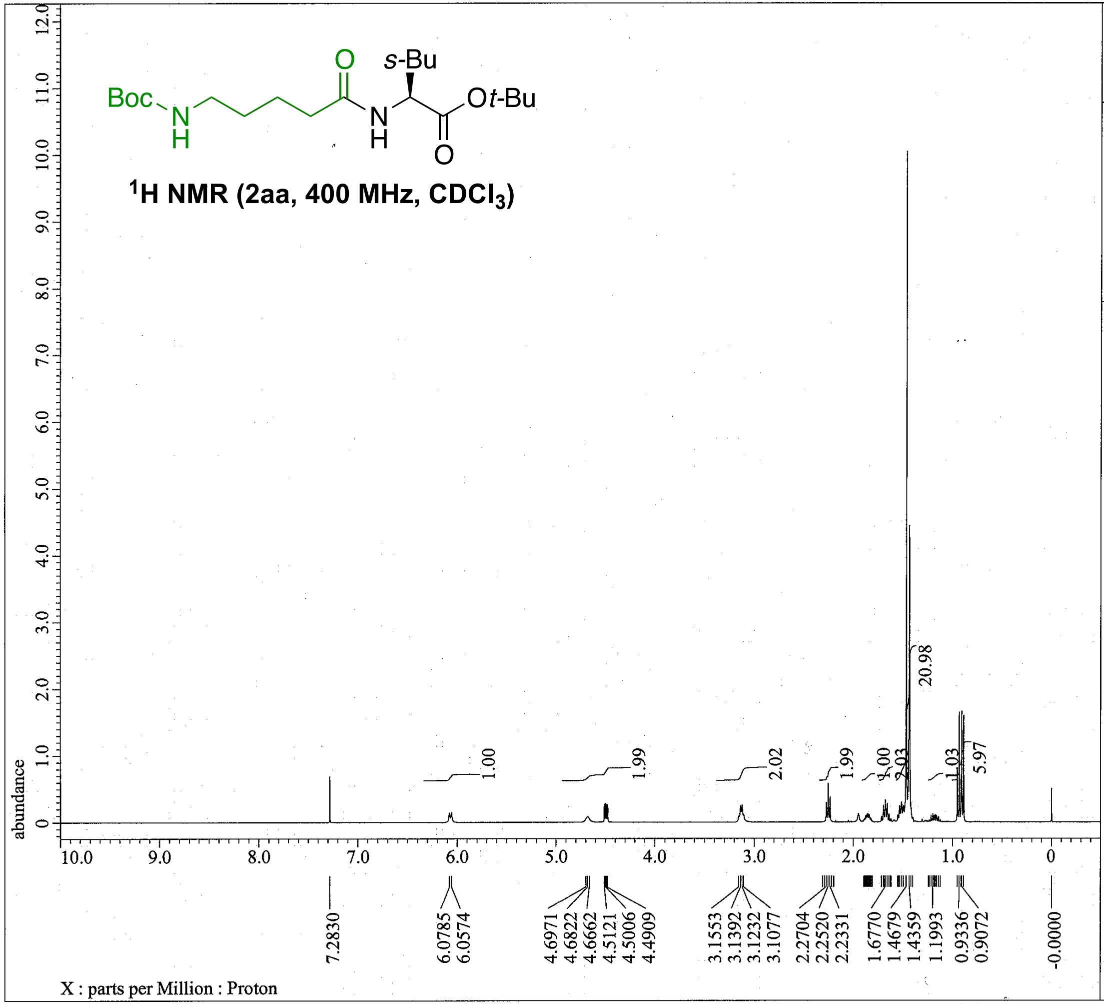
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-194_E1H-1-1.jdf
```

```
Filename           = wm11-194_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm11-194
Solvent            = CHLOROFORM-D
Creation_Time      = 24-MAY-2021 07:41:25
Revision_Time      = 24-MAY-2021 07:45:18
Current_Time       = 24-MAY-2021 07:45:36

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 30
Temp_Get           = 22.4[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-194-13C_E13C-1-1.jdf
```

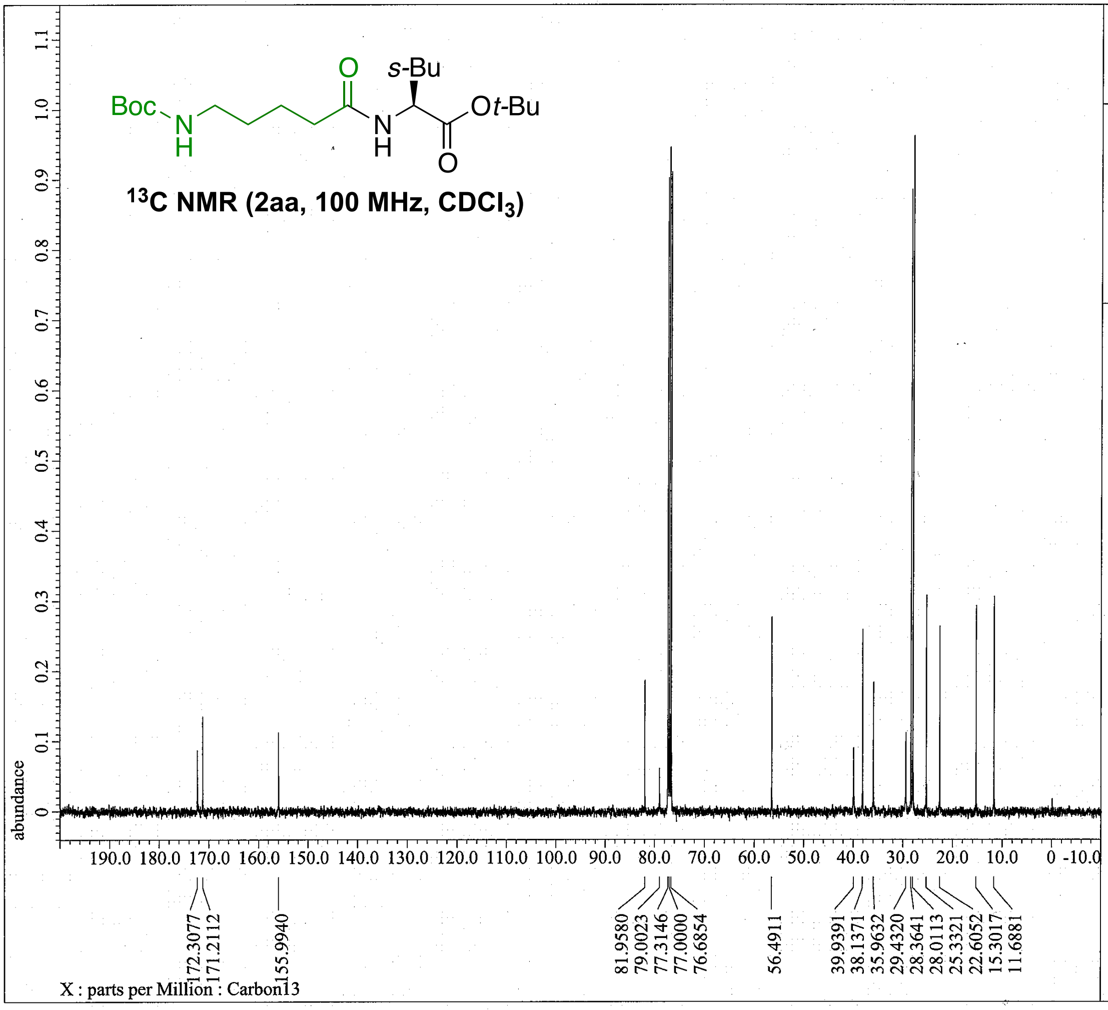
```
Filename         = wm11-194-13C_E13C-1-3.jdf
Author           = delta
Experiment       = single_pulse_dec.jxp
Sample_Id        = wm11-194-13C
Solvent          = CHLOROFORM-D
Creation_Time    = 24-MAY-2021 07:44:46
Revision_Time    = 24-MAY-2021 07:55:03
Current_Time     = 24-MAY-2021 07:55:22

Data_Format      = 1D COMPLEX
Dim_Size         = 26214
Dim_Title        = Carbon13
Dim_Units        = [ppm]
Dimensions       = X
Site             = JNM-ECS400
Spectrometer     = DELTA2_NMR

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 1.04333312[s]
X_Domain         = 13C
X_Freq           = 100.52530333[MHz]
X_Offset         = 100[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 0.95846665[Hz]
X_Sweep          = 31.40703518[kHz]
X_Sweep_Clipped  = 25.12562814[kHz]
Irr_Domain       = Proton
Irr_Freq         = 399.78219838[MHz]
Irr_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 257
Total_Scans      = 257

Relaxation_Delay = 2[s]
Recvr_Gain       = 60
Temp_Get         = 22.5[dC]
X_90_Width       = 10.1[us]
X_Acq_Time       = 1.04333312[s]
X_Angle          = 30[deg]
X_Atn            = 4.6[dB]
X_Pulse          = 3.36666667[us]
Irr_Atn_Dec      = 25.996[dB]
Irr_Atn_Noe      = 25.996[dB]
Irr_Noise        = WALTZ
Irr_Pwidth       = 0.115[ms]
Decoupling       = TRUE
Initial_Wait     = 1[s]
```



$^1$H NMR (2aa, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-13_E1H-1-1.jdf
```

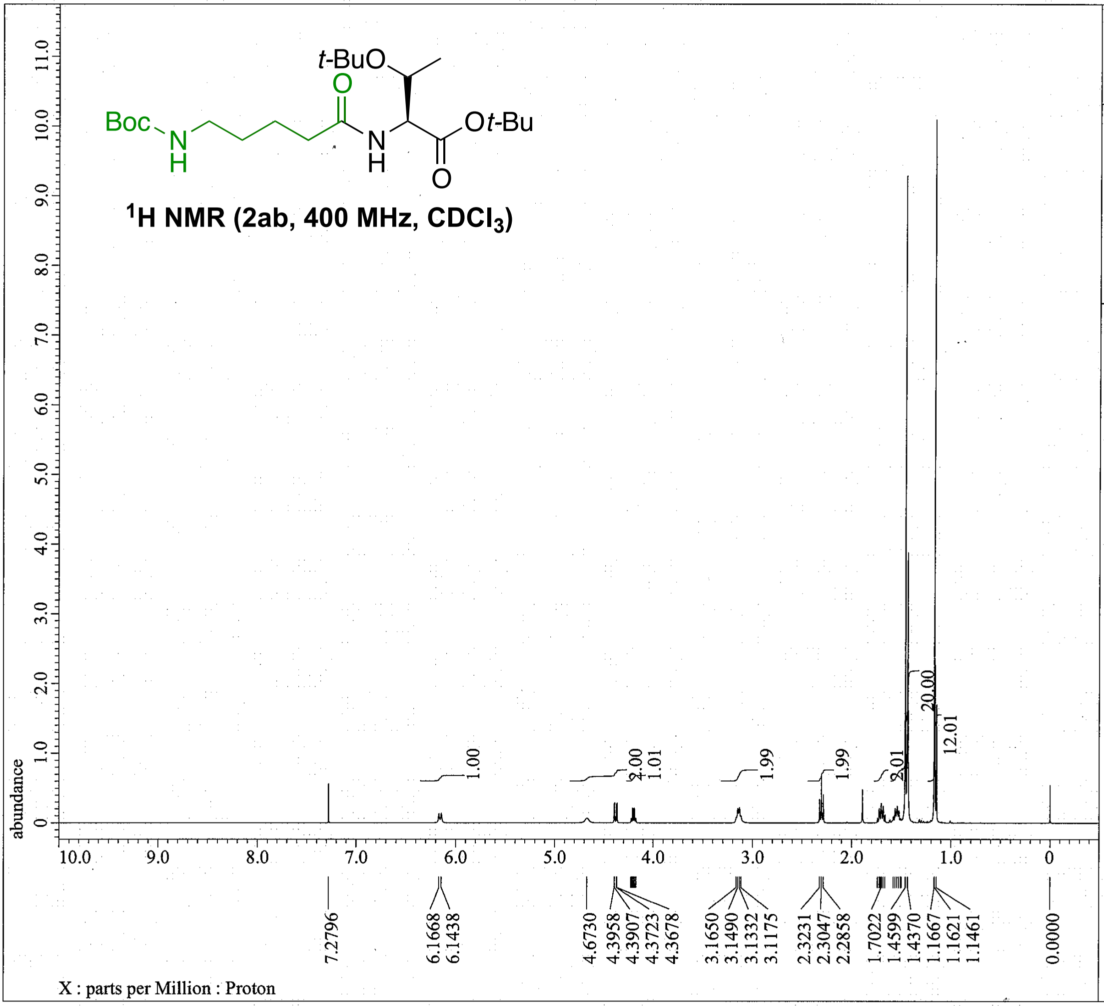
```
Filename           = wm12-13_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-13
Solvent            = CHLOROFORM-D
Creation_Time      = 11-JUN-2021 13:33:17
Revision_Time      = 11-JUN-2021 13:37:59
Current_Time       = 11-JUN-2021 13:38:10

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 22[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (2aa, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-13-13C_E13C-1-1.jdf
```

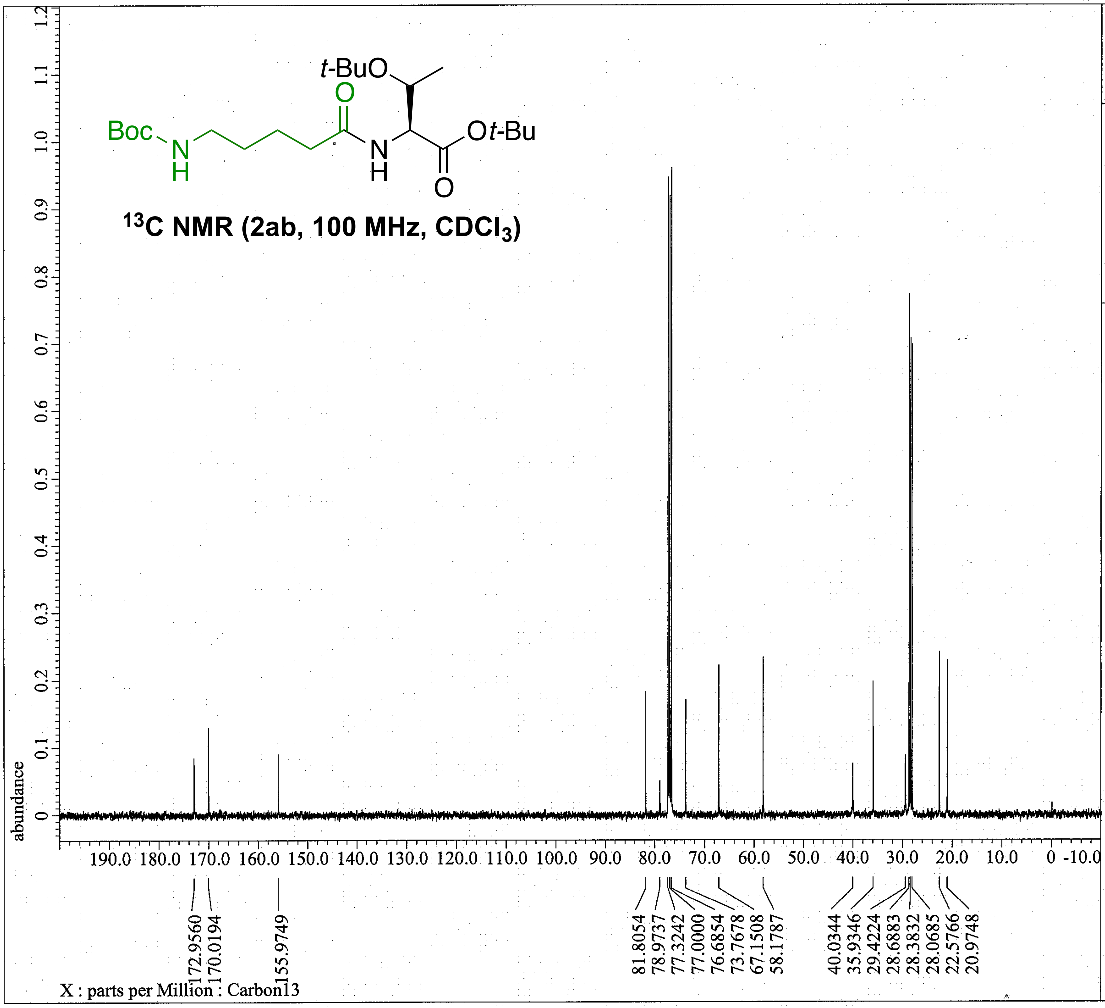
```
Filename          = wm12-13-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-13-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 11-JUN-2021 13:37:20
Revision_Time     = 11-JUN-2021 13:59:58
Current_Time      = 11-JUN-2021 14:00:21

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 513
Total_Scans       = 513

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.5[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (2ab, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-10-2_E1H-1-1.jdf
```

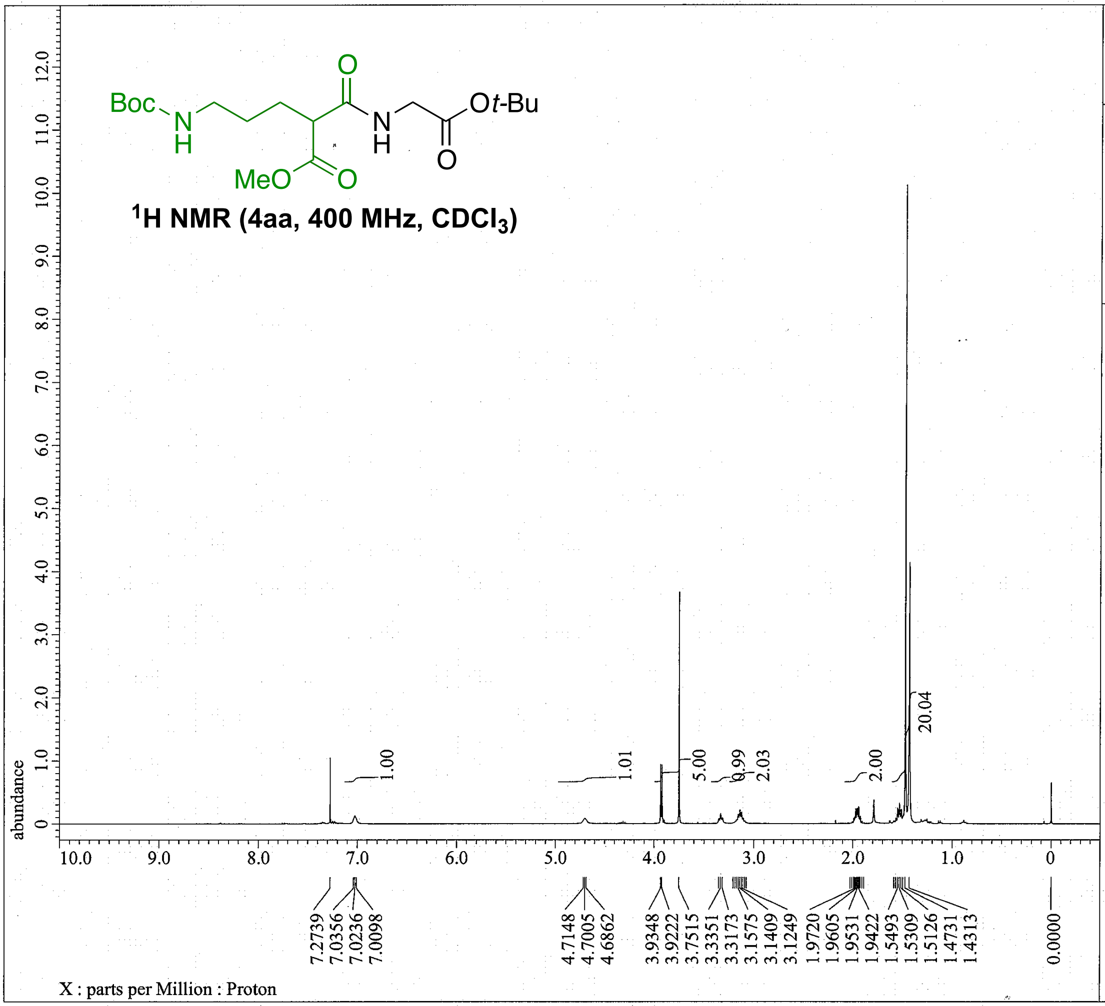
```
Filename           = wm12-10-2_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-10-2
Solvent            = CHLOROFORM-D
Creation_Time      = 10-JUN-2021 12:46:05
Revision_Time      = 10-JUN-2021 12:47:22
Current_Time       = 10-JUN-2021 12:47:34

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 23.3[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (2ab, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-10-2-13C_E13C-1-1.jdf
```

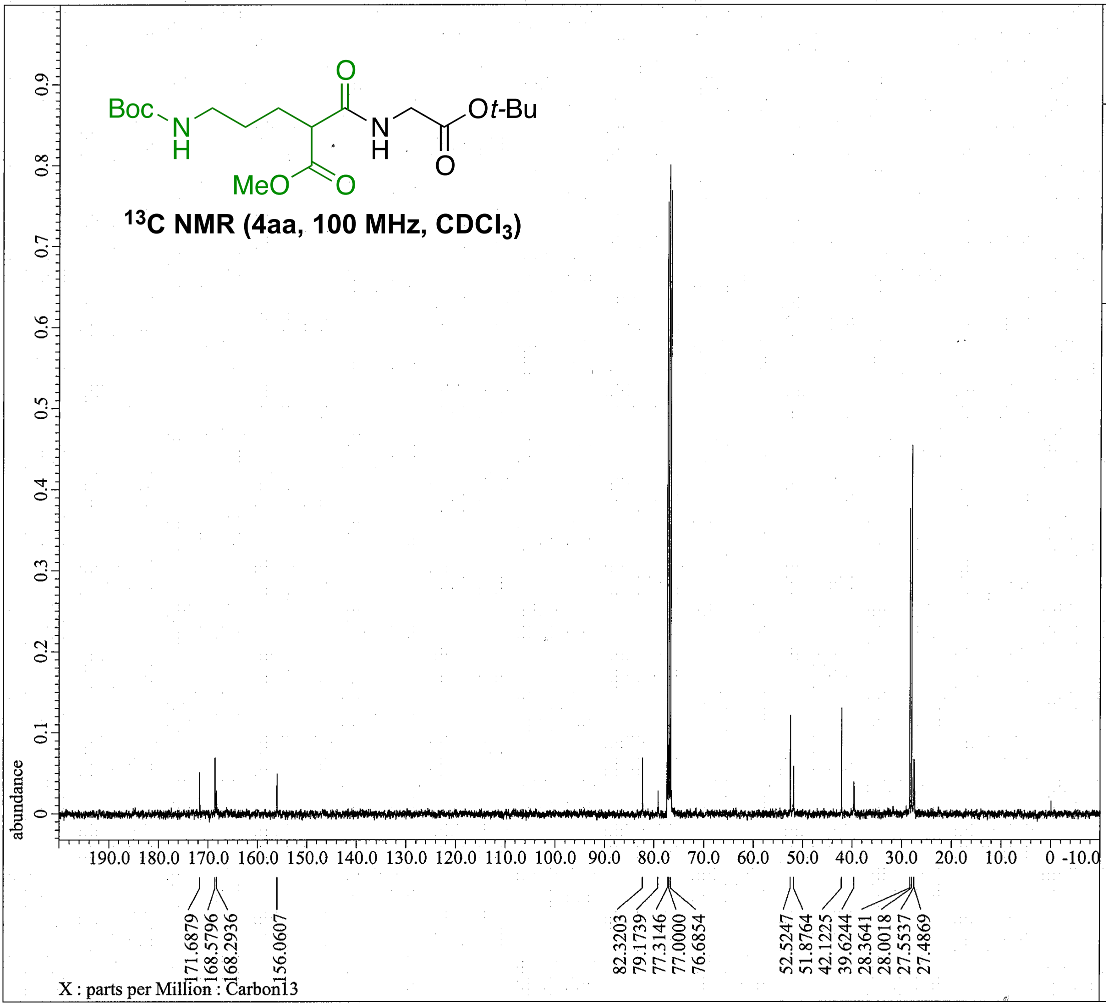
```
Filename          = wm12-10-2-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-10-2-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 10-JUN-2021 12:50:20
Revision_Time     = 10-JUN-2021 13:23:37
Current_Time      = 10-JUN-2021 13:23:56

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 721
Total_Scans       = 721

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.2[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (4aa, 400 MHz, CDCl$_3$)

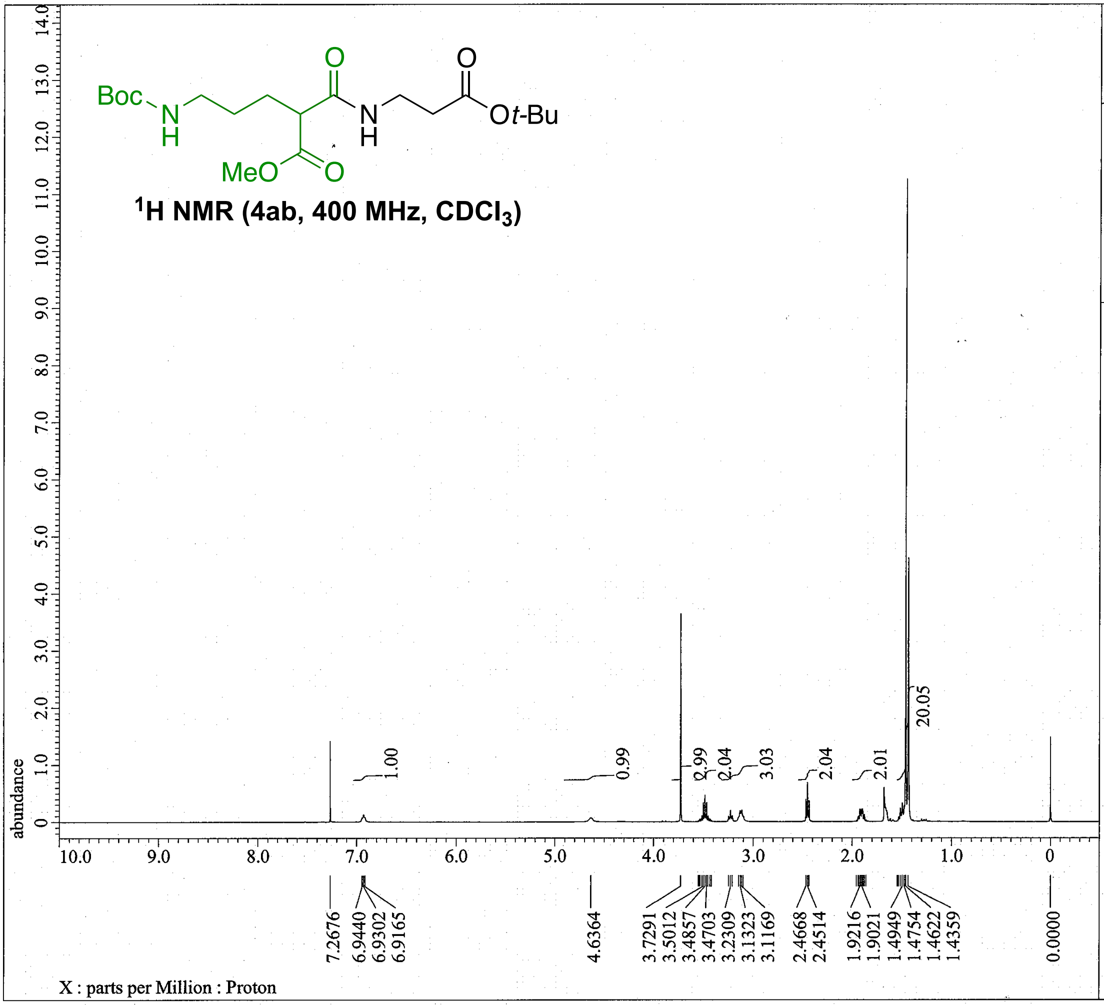
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-31-1_E1H-1-1.jdf
```

```
Filename           = wm12-31-1_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-31-1
Solvent            = CHLOROFORM-D
Creation_Time      = 25-JUN-2021 13:12:21
Revision_Time      = 25-JUN-2021 13:16:23
Current_Time       = 25-JUN-2021 13:16:39

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 30
Temp_Get           = 22.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```


$^{13}$C NMR (4aa, 100 MHz, CDCl$_3$)

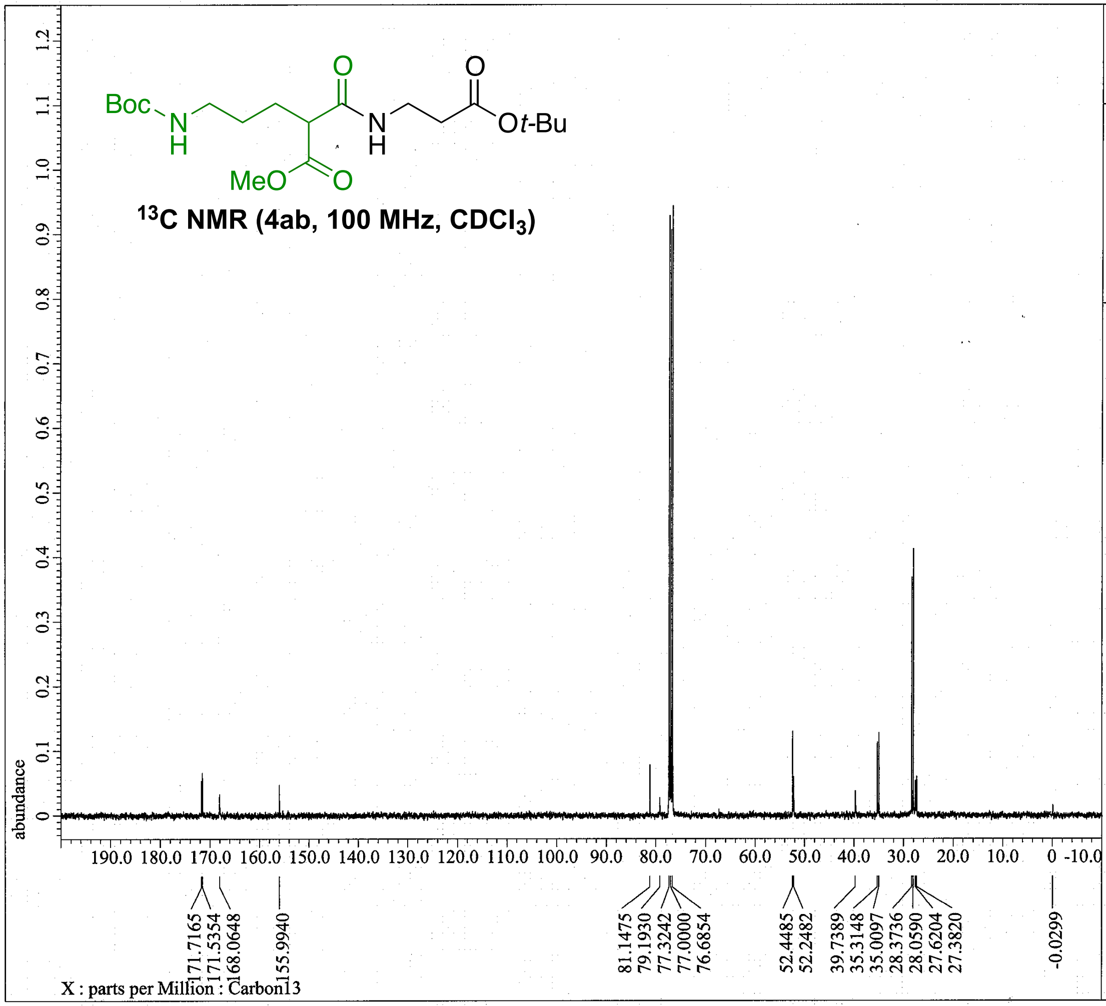
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-31-1-13C_E13C-1-1.jdf
```

```
Filename           = wm12-31-1-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-31-1-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 25-JUN-2021 13:14:29
Revision_Time      = 25-JUN-2021 13:51:42
Current_Time       = 25-JUN-2021 13:52:00

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 801
Total_Scans        = 801

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.4[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (4ab, 400 MHz, CDCl$_3$)

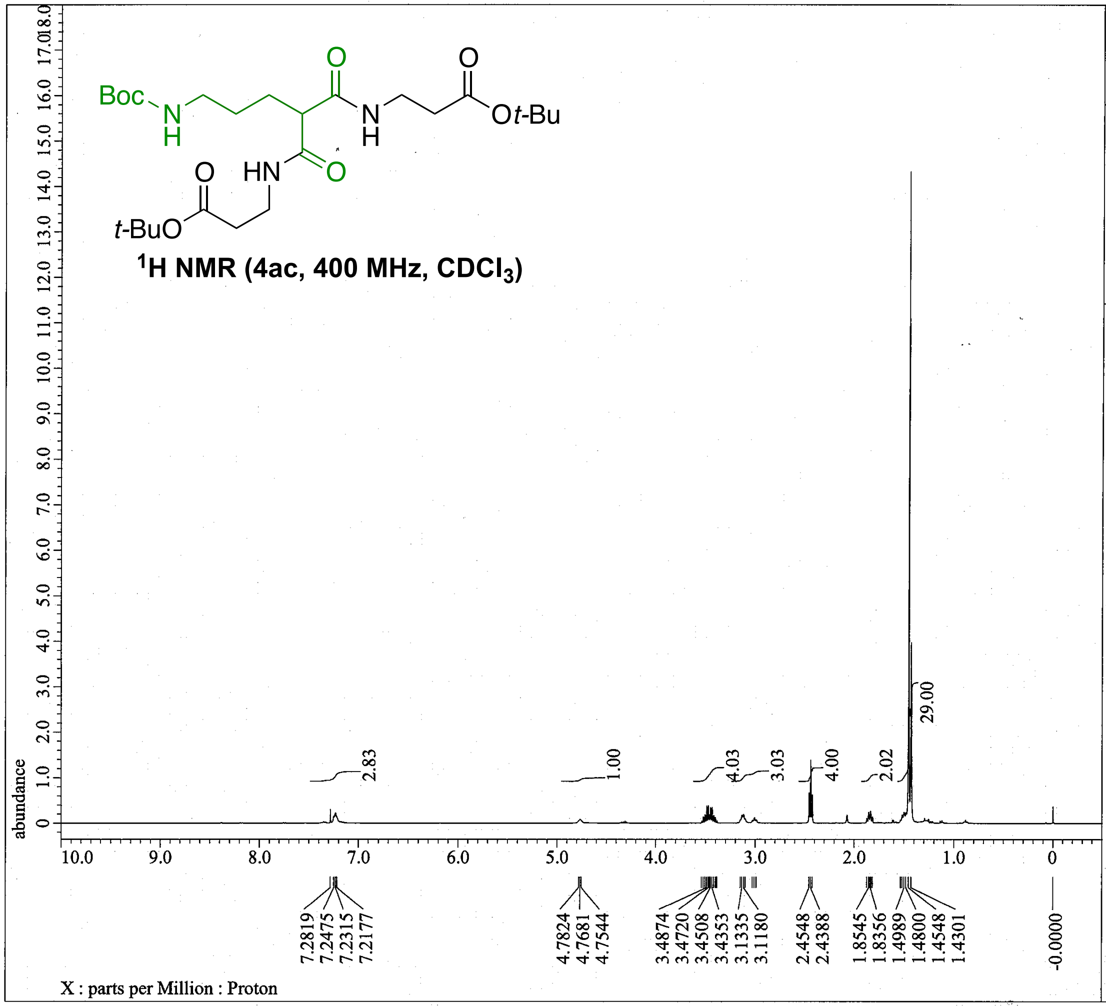
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-47-1_E1H-5-1.jdf

Filename           = wm12-47-1_E1H-5-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-47-1
Solvent            = CHLOROFORM-D
Creation_Time      = 10-JUL-2021 09:01:46
Revision_Time      = 10-JUL-2021 09:05:37
Current_Time       = 10-JUL-2021 09:05:52

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 38
Temp_Get           = 24[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (4ab, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-47-1-13C_E13C-1-1.jdf
```

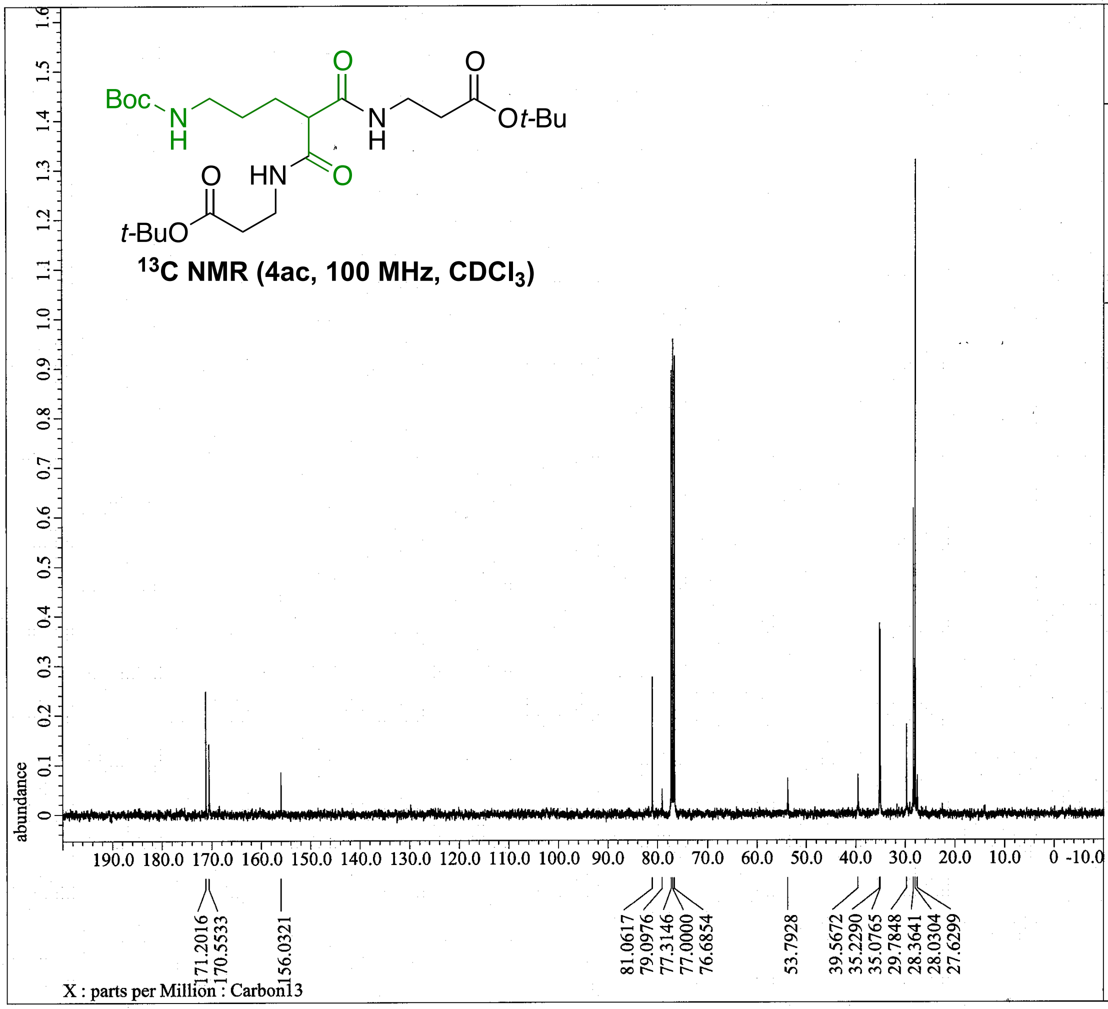
```
Filename            = wm12-47-1-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-47-1-13C
Solvent             = CHLOROFORM-D
Creation_Time       =  9-JUL-2021 16:28:55
Revision_Time       =  9-JUL-2021 17:17:21
Current_Time        =  9-JUL-2021 17:17:47

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 24[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (4ac, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-66-4_E1H-1-1.jdf
```

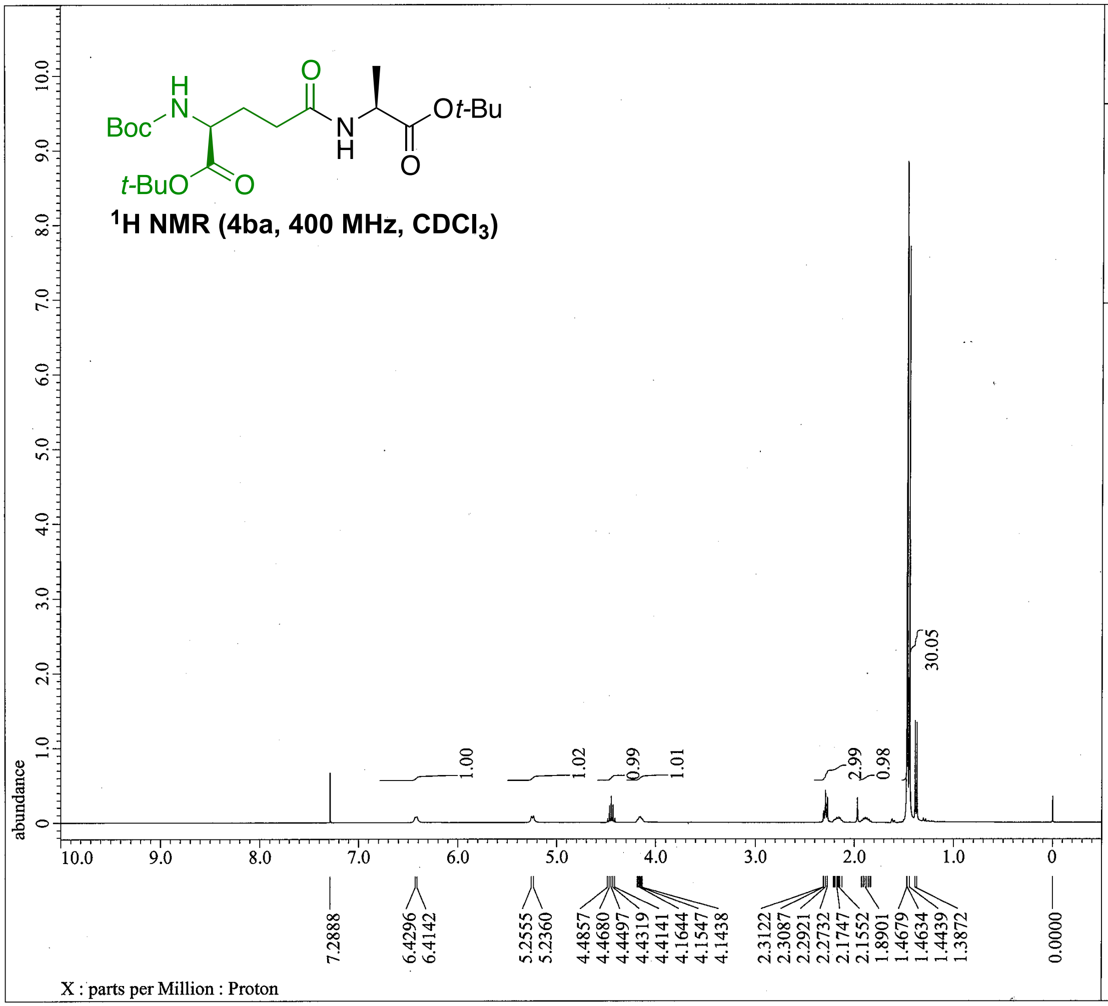
```
Filename          = wm12-66-4_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-66-4
Solvent           = CHLOROFORM-D
Creation_Time     = 29-JUL-2021 12:21:37
Revision_Time     = 29-JUL-2021 12:27:10
Current_Time      = 29-JUL-2021 12:27:25

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 23.7[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}C$ NMR (4ac, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-66-4-13C_E13C_copy2-1-1.jd
```

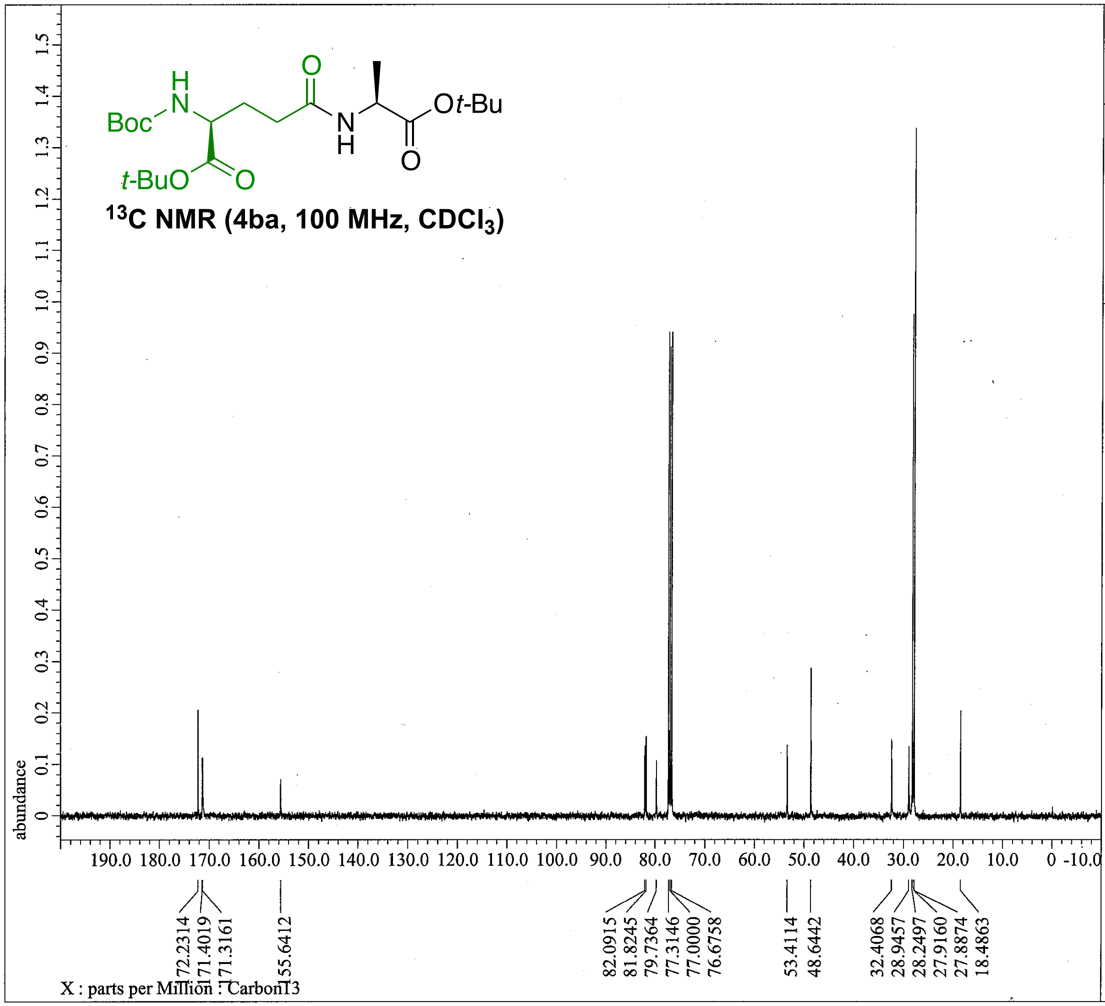
```
Filename            = wm12-66-4-13C_E13C_copy2-1
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-66-4-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 29-JUL-2021 12:23:46
Revision_Time       = 29-JUL-2021 12:33:19
Current_Time        = 29-JUL-2021 12:33:36

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 0[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Incomplete_Copy     = TRUE
Scans               = 256
Total_Scans         = 256

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 23.5[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
```



$^1$H NMR (4ba, 400 MHz, CDCl$_3$)

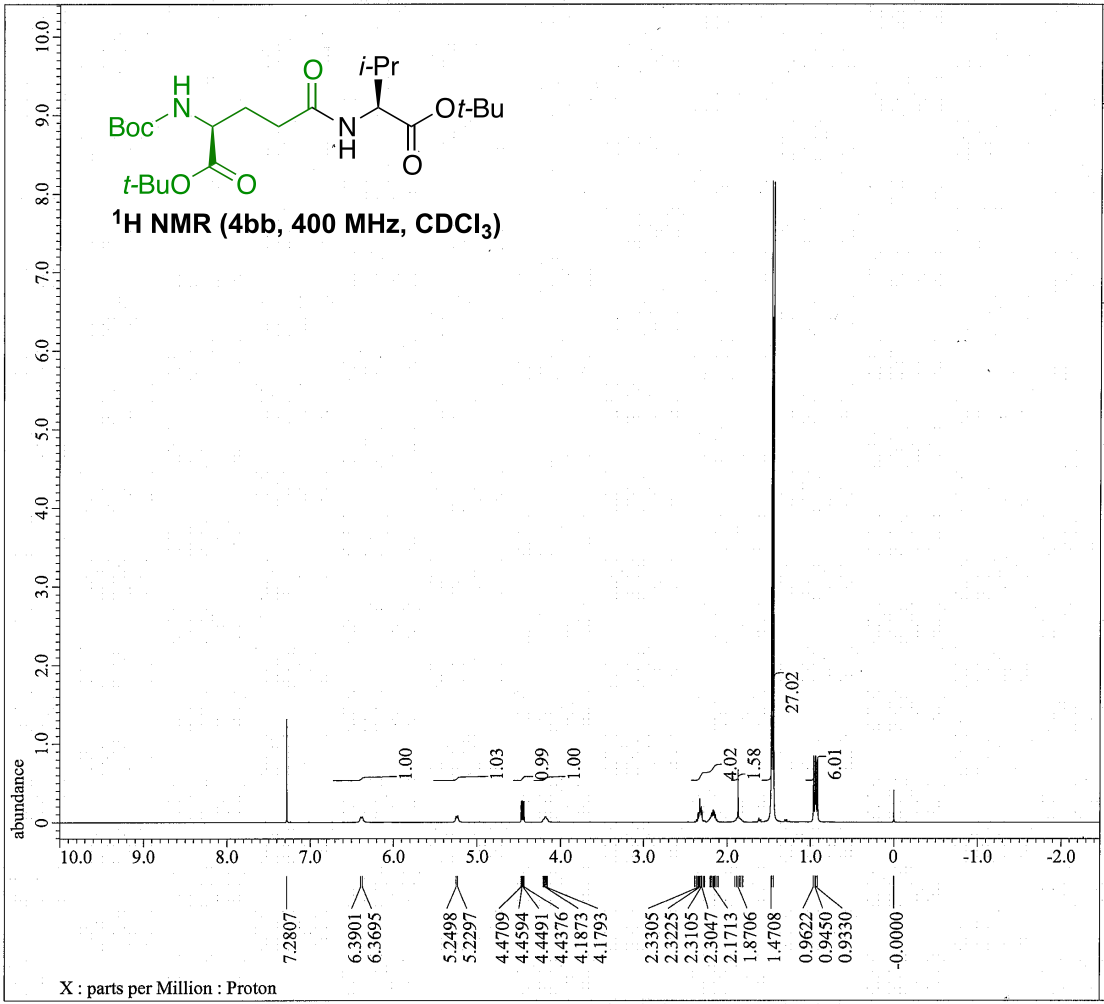
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-6_E1H-1-1.jdf
```

```
Filename          = wm12-6_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-6
Solvent           = CHLOROFORM-D
Creation_Time     =  7-JUN-2021 08:05:19
Revision_Time     =  7-JUN-2021 08:09:30
Current_Time      =  7-JUN-2021 08:10:00

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 22
Temp_Get          = 22.9[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (4ba, 100 MHz, CDCl$_3$)

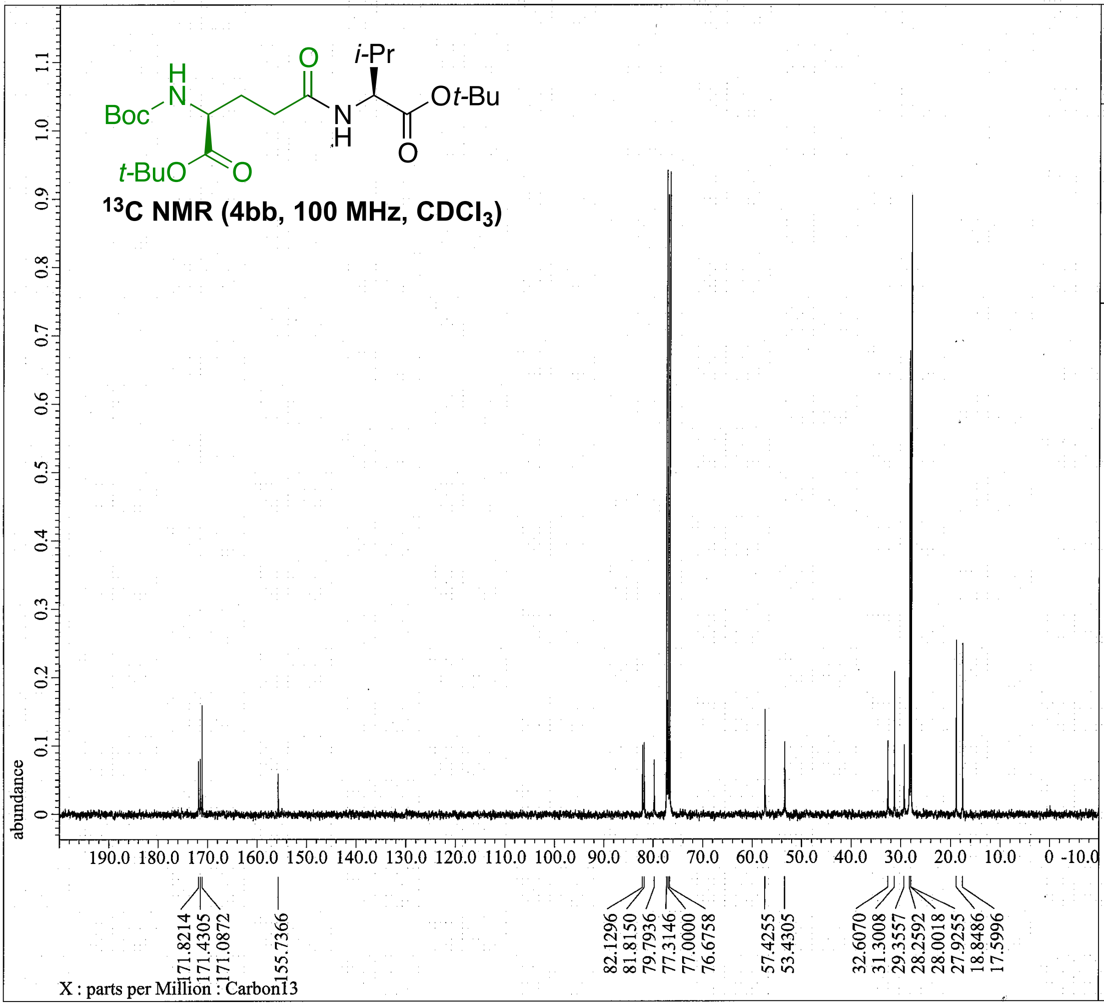
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-6-13C_E13C-1-1.jdf

Filename            = wm12-6-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-6-13C
Solvent             = CHLOROFORM-D
Creation_Time       =  7-JUN-2021 08:09:20
Revision_Time       =  7-JUN-2021 08:32:00
Current_Time        =  7-JUN-2021 08:32:39

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 513
Total_Scans         = 513

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.8[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (4bb, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-21-4_E1H-1-1.jdf
```

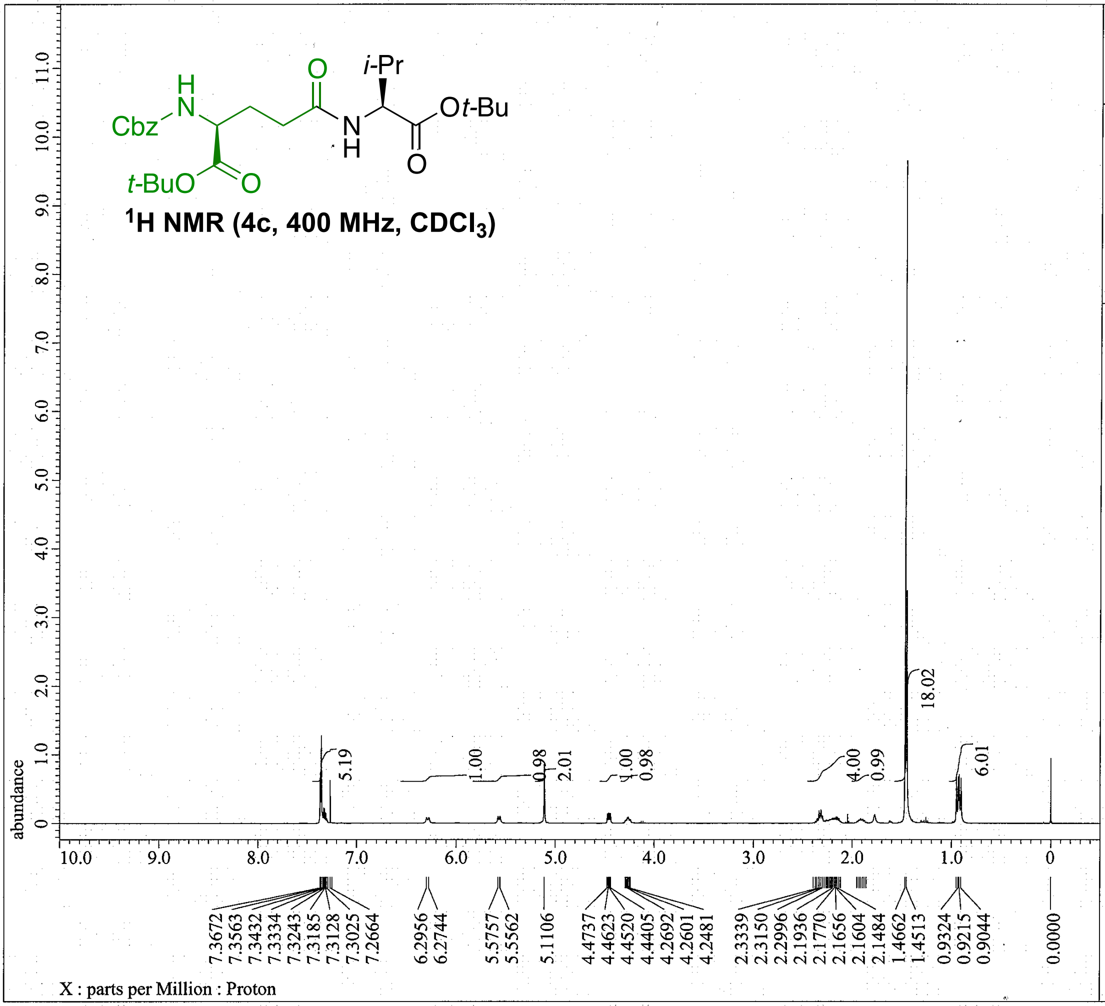
```
Filename          = wm12-21-4_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-21-4
Solvent           = CHLOROFORM-D
Creation_Time     = 26-JUN-2021 08:08:21
Revision_Time     = 26-JUN-2021 08:11:00
Current_Time      = 26-JUN-2021 08:11:16

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 24
Temp_Get          = 22.9[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (4bb, 100 MHz, CDCl$_3$)

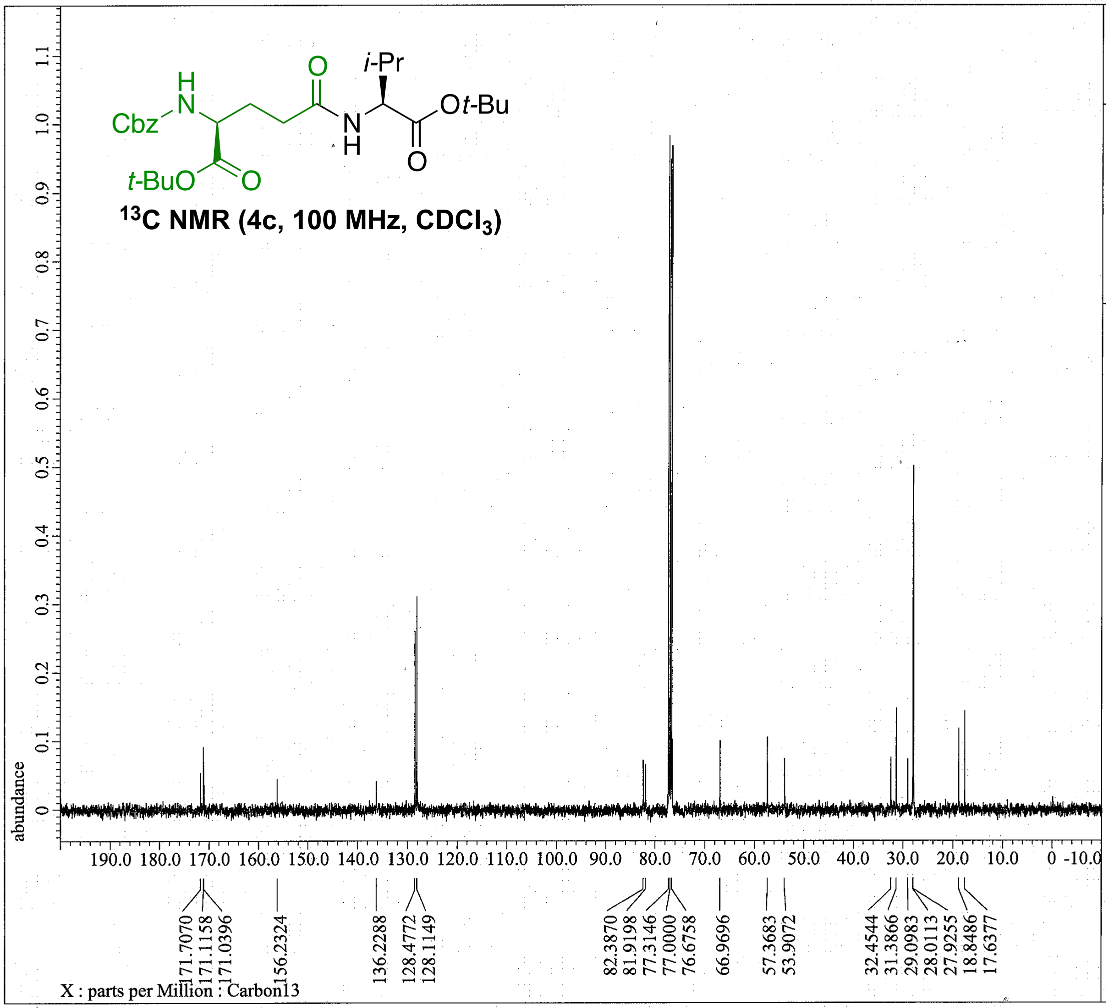
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-21-4-13C_E13C-1-1.jdf
```

```
Filename           = wm12-21-4-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-21-4-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 26-JUN-2021 08:13:10
Revision_Time      = 26-JUN-2021 08:46:43
Current_Time       = 26-JUN-2021 08:47:05

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 721
Total_Scans        = 721

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-20-1_E1H-1-1.jdf
```

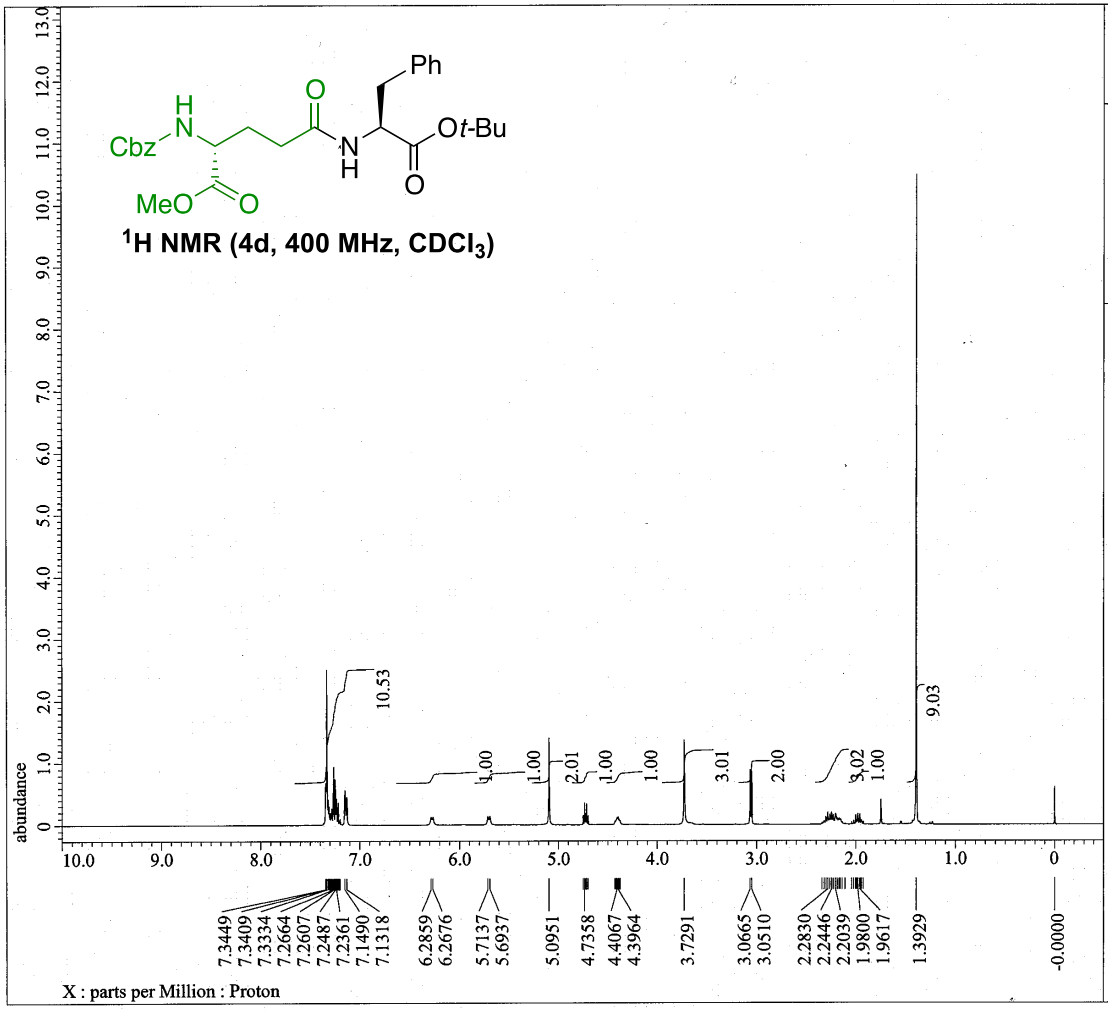
```
Filename             = wm12-20-1_E1H-1-5.jdf
Author               = delta
Experiment           = single_pulse.jxp
Sample_Id            = wm12-20-1
Solvent              = CHLOROFORM-D
Creation_Time        = 18-JUN-2021 13:46:18
Revision_Time        = 18-JUN-2021 13:50:03
Current_Time         = 18-JUN-2021 13:50:18

Data_Format          = 1D COMPLEX
Dim_Size             = 26214
Dim_Title            = Proton
Dim_Units            = [ppm]
Dimensions           = X
Site                 = JNM-ECS400
Spectrometer         = DELTA2_NMR

Field_Strength       = 9.389766[T] (400[MHz])
X_Acq_Duration       = 4.36731904[s]
X_Domain             = 1H
X_Freq               = 399.78219838[MHz]
X_Offset             = 5[ppm]
X_Points             = 32768
X_Prescans           = 1
X_Resolution         = 0.22897343[Hz]
X_Sweep              = 7.5030012[kHz]
X_Sweep_Clipped      = 6.00240096[kHz]
Irr_Domain           = Proton
Irr_Freq             = 399.78219838[MHz]
Irr_Offset           = 5[ppm]
Tri_Domain           = Proton
Tri_Freq             = 399.78219838[MHz]
Tri_Offset           = 5[ppm]
Clipped              = FALSE
Scans                = 8
Total_Scans          = 8

Relaxation_Delay     = 5[s]
Recvr_Gain           = 30
Temp_Get             = 22.5[dC]
X_90_Width           = 6.47[us]
X_Acq_Time           = 4.36731904[s]
X_Angle              = 45[deg]
X_Atn                = 1[dB]
X_Pulse              = 3.235[us]
Irr_Mode             = Off
Tri_Mode             = Off
Dante_Presat         = FALSE
```



$^{13}$C NMR (4c, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-20-13C_E13C_copy4-1-1.jdf
```

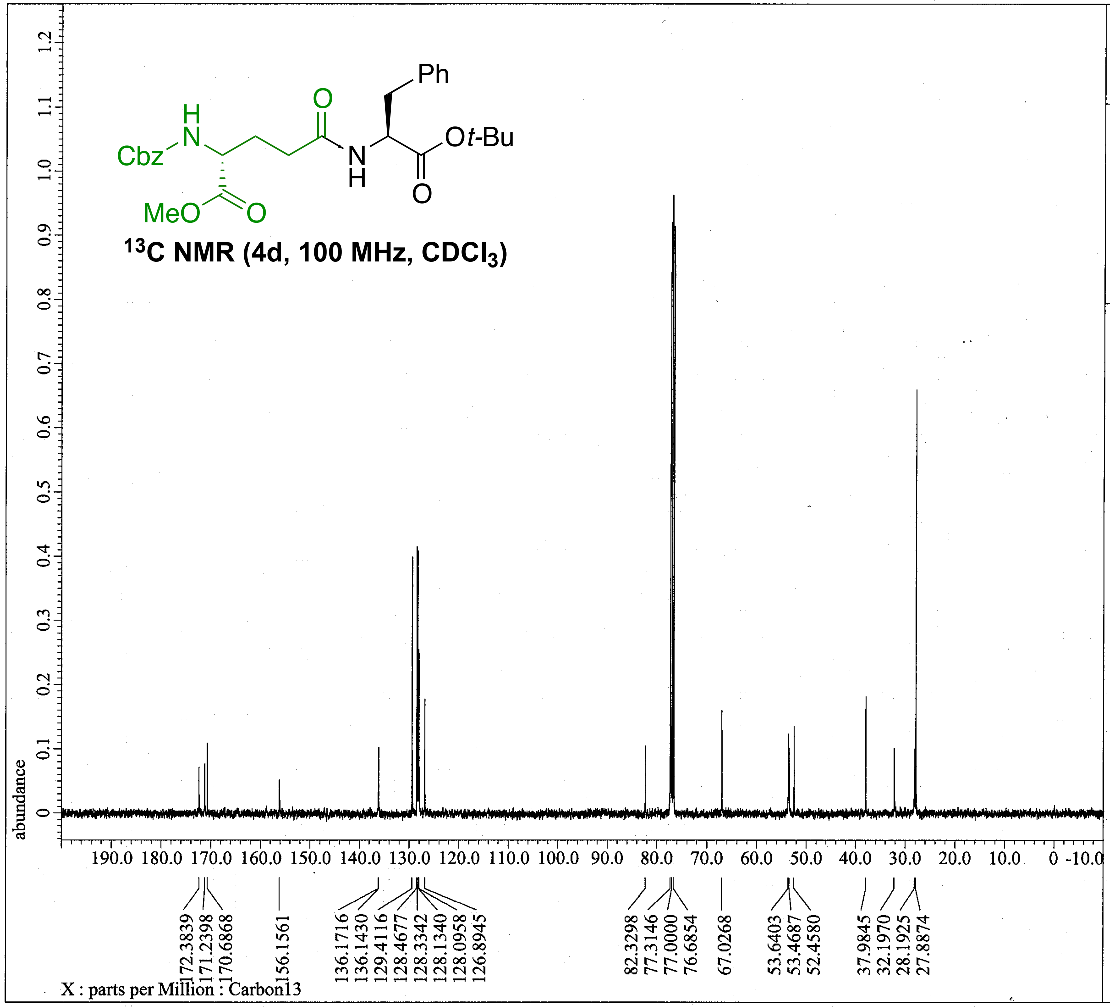
```
Filename           = wm12-20-13C_E13C_copy4-1-3
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-20-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 18-JUN-2021 13:51:24
Revision_Time      = 18-JUN-2021 14:00:52
Current_Time       = 18-JUN-2021 14:01:13

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 0[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Incomplete_Copy    = TRUE
Scans              = 257
Total_Scans        = 257

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.6[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
```



$^1$H NMR (4d, 400 MHz, CDCl$_3$)

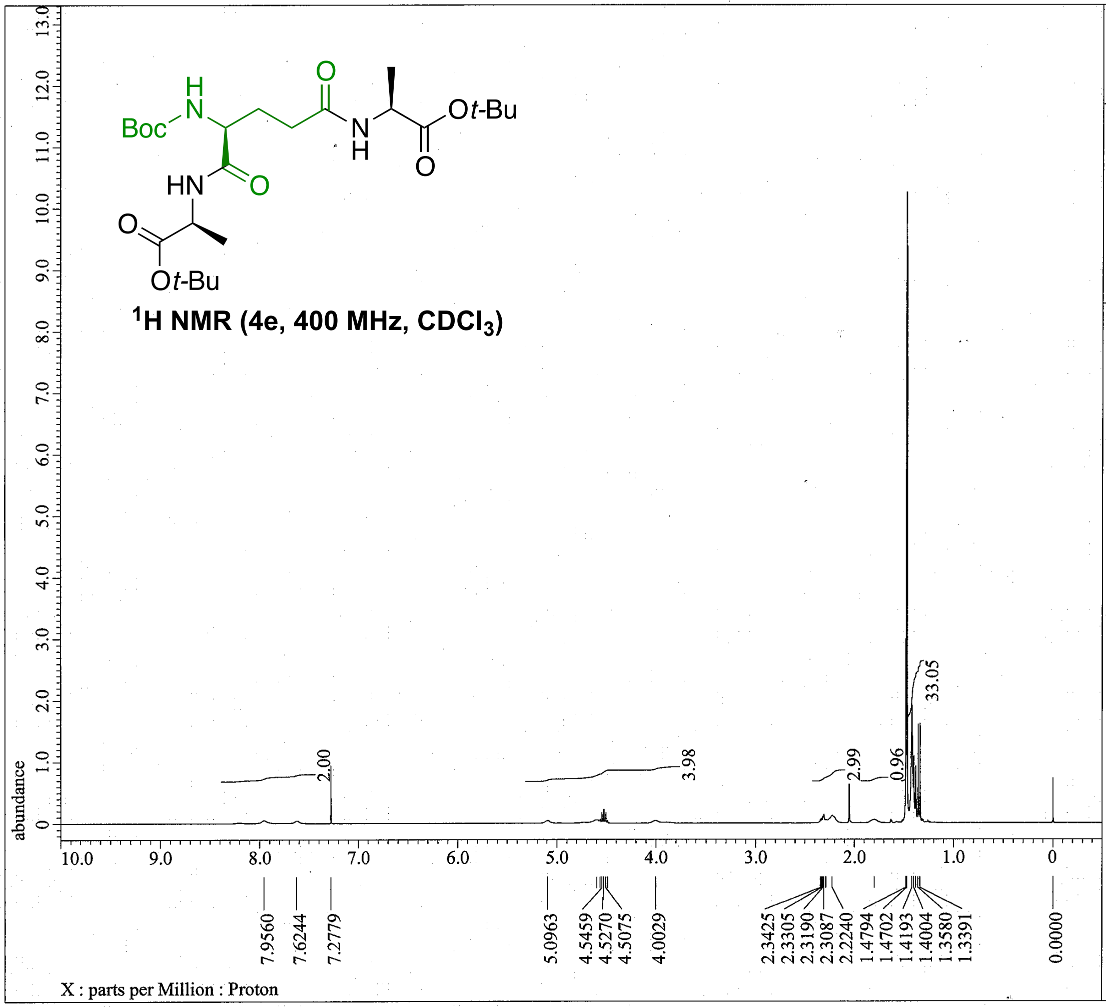
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-85-2_E1H-1-1.jdf
```

```
Filename          = wm12-85-2_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-85-2
Solvent           = CHLOROFORM-D
Creation_Time     = 30-OCT-2021 08:32:39
Revision_Time     = 30-OCT-2021 08:34:30
Current_Time      = 30-OCT-2021 08:34:40

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.8[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```


$^{13}$C NMR (4d, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-85-2-13C_E13C-1-1.jdf
```

```
Filename         = wm12-85-2-13C_E13C-1-4.jdf
Author           = delta
Experiment       = single_pulse_dec.jxp
Sample_Id        = wm12-85-2-13C
Solvent          = CHLOROFORM-D
Creation_Time    = 30-OCT-2021 08:34:46
Revision_Time    = 30-OCT-2021 08:57:01
Current_Time     = 30-OCT-2021 08:57:57

Data_Format      = 1D COMPLEX
Dim_Size         = 26214
Dim_Title        = Carbon13
Dim_Units        = [ppm]
Dimensions       = X
Site             = JNM-ECS400
Spectrometer     = DELTA2_NMR

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 1.04333312[s]
X_Domain         = 13C
X_Freq           = 100.52530333[MHz]
X_Offset         = 100[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 0.95846665[Hz]
X_Sweep          = 31.40703518[kHz]
X_Sweep_Clipped  = 25.12562814[kHz]
Irr_Domain       = Proton
Irr_Freq         = 399.78219838[MHz]
Irr_Offset       = 5[ppm]
Clipped          = TRUE
Scans            = 512
Total_Scans      = 512

Relaxation_Delay = 2[s]
Recvr_Gain       = 60
Temp_Get         = 24.7[dC]
X_90_Width       = 10.1[us]
X_Acq_Time       = 1.04333312[s]
X_Angle          = 30[deg]
X_Atn            = 4.6[dB]
X_Pulse          = 3.36666667[us]
Irr_Atn_Dec      = 25.996[dB]
Irr_Atn_Noe      = 25.996[dB]
Irr_Noise        = WALTZ
Irr_Pwidth       = 0.115[ms]
Decoupling       = TRUE
Initial_Wait     = 1[s]
```

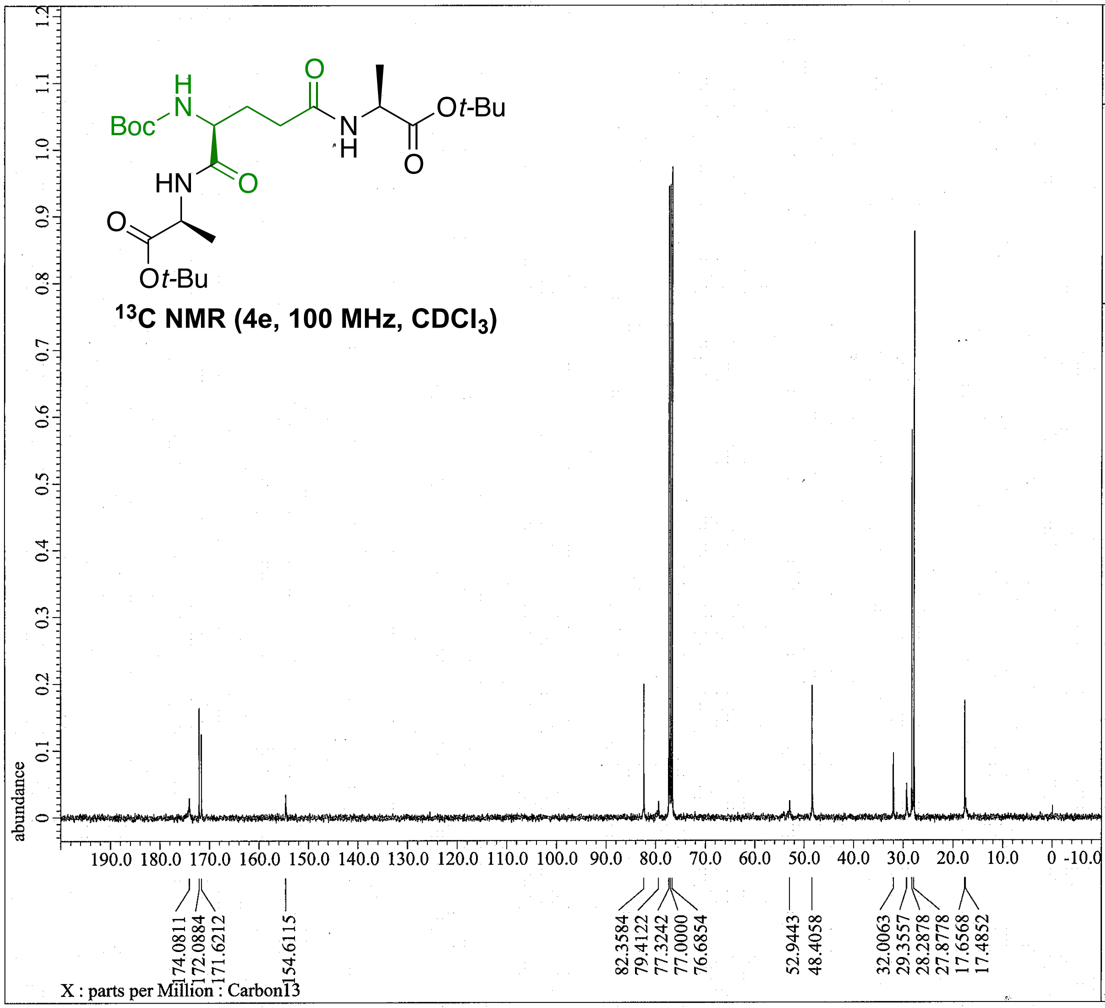


<sup></sup>

$^1$H NMR (4e, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-62_E1H-1-1.jdf
```

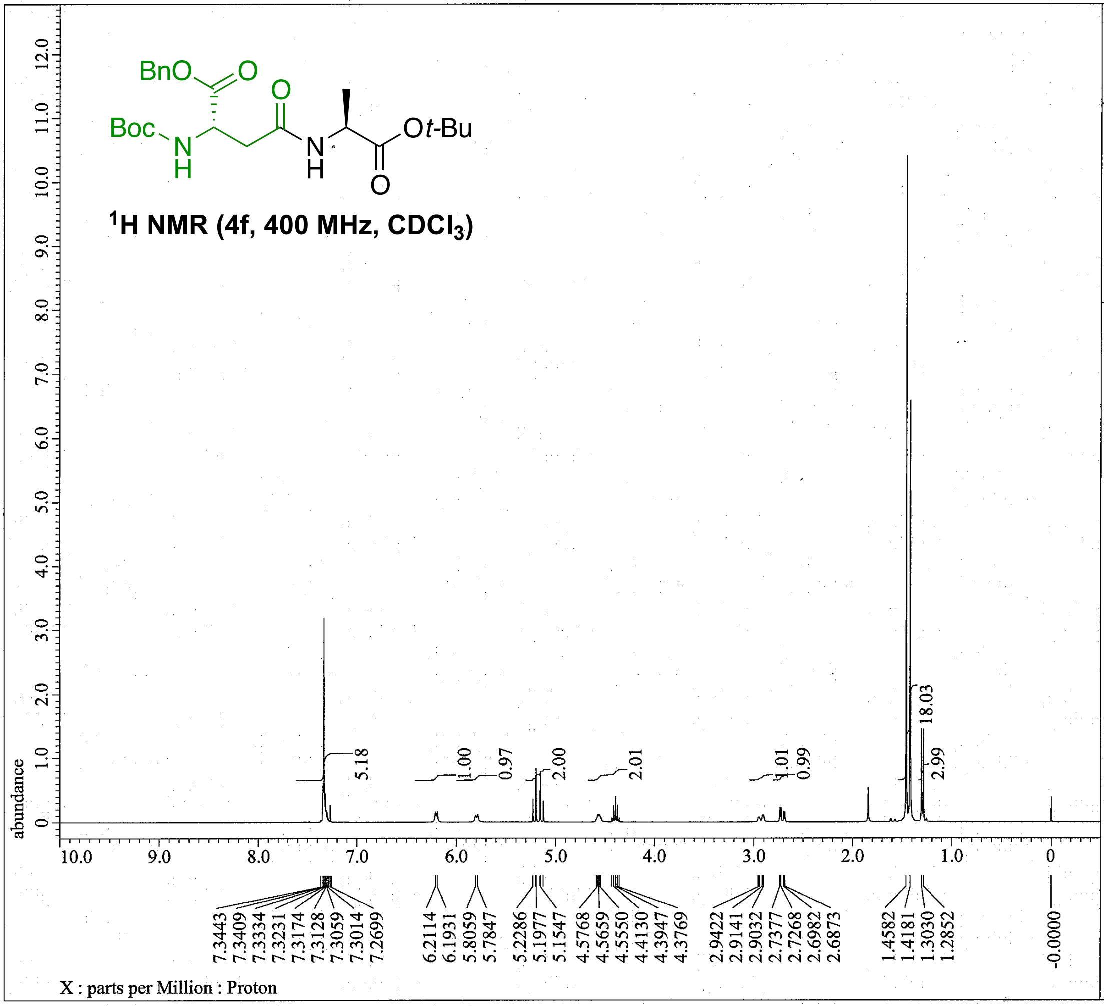
```
Filename           = wm12-62_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-62
Solvent            = CHLOROFORM-D
Creation_Time      = 26-JUL-2021 15:49:40
Revision_Time      = 26-JUL-2021 15:58:45
Current_Time       = 26-JUL-2021 15:58:51

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 28
Temp_Get           = 23.9[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (4e, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-62-13C_E13C-1-1.jdf
```

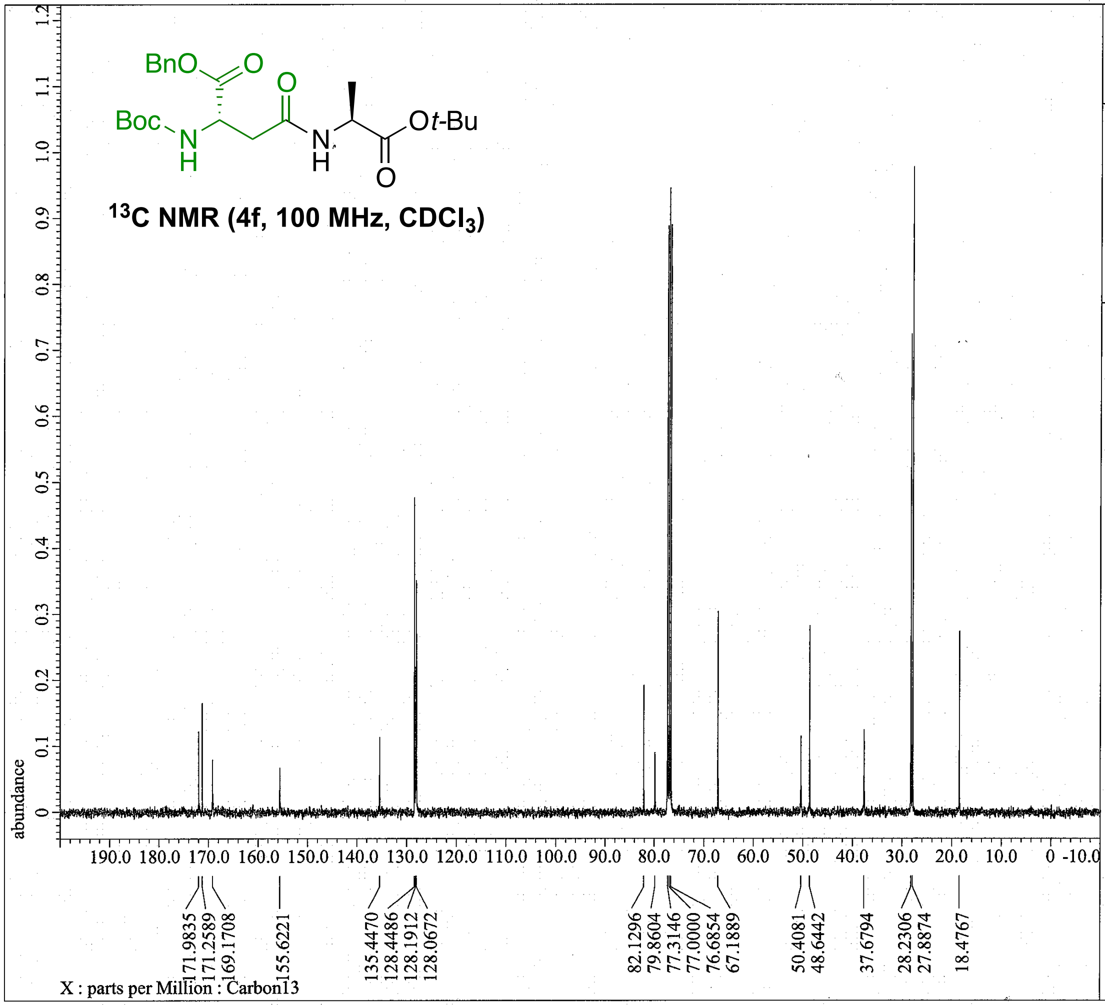
```
Filename          = wm12-62-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-62-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 26-JUL-2021 15:53:56
Revision_Time     = 26-JUL-2021 16:42:28
Current_Time      = 26-JUL-2021 16:42:46

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.7[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (4f, 400 MHz, CDCl$_3$)

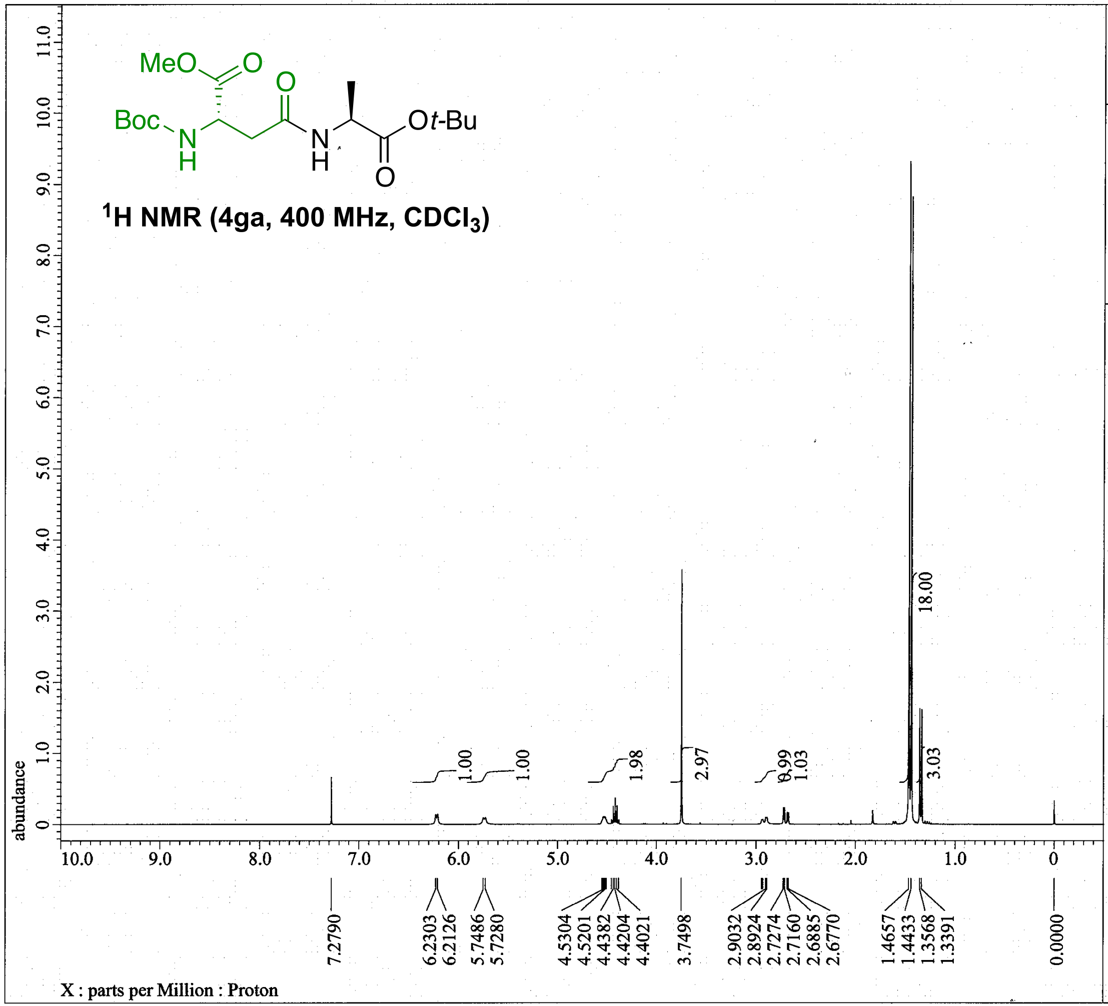
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-45_E1H-1-1.jdf
```

```
Filename            = wm12-45_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-45
Solvent             = CHLOROFORM-D
Creation_Time       =  7-JUL-2021 13:42:53
Revision_Time       =  7-JUL-2021 13:45:21
Current_Time        =  7-JUL-2021 13:45:37

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay = 5[s]
Recvr_Gain          = 26
Temp_Get            = 23.8[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-45-13C_E13C-1-1.jdf
```

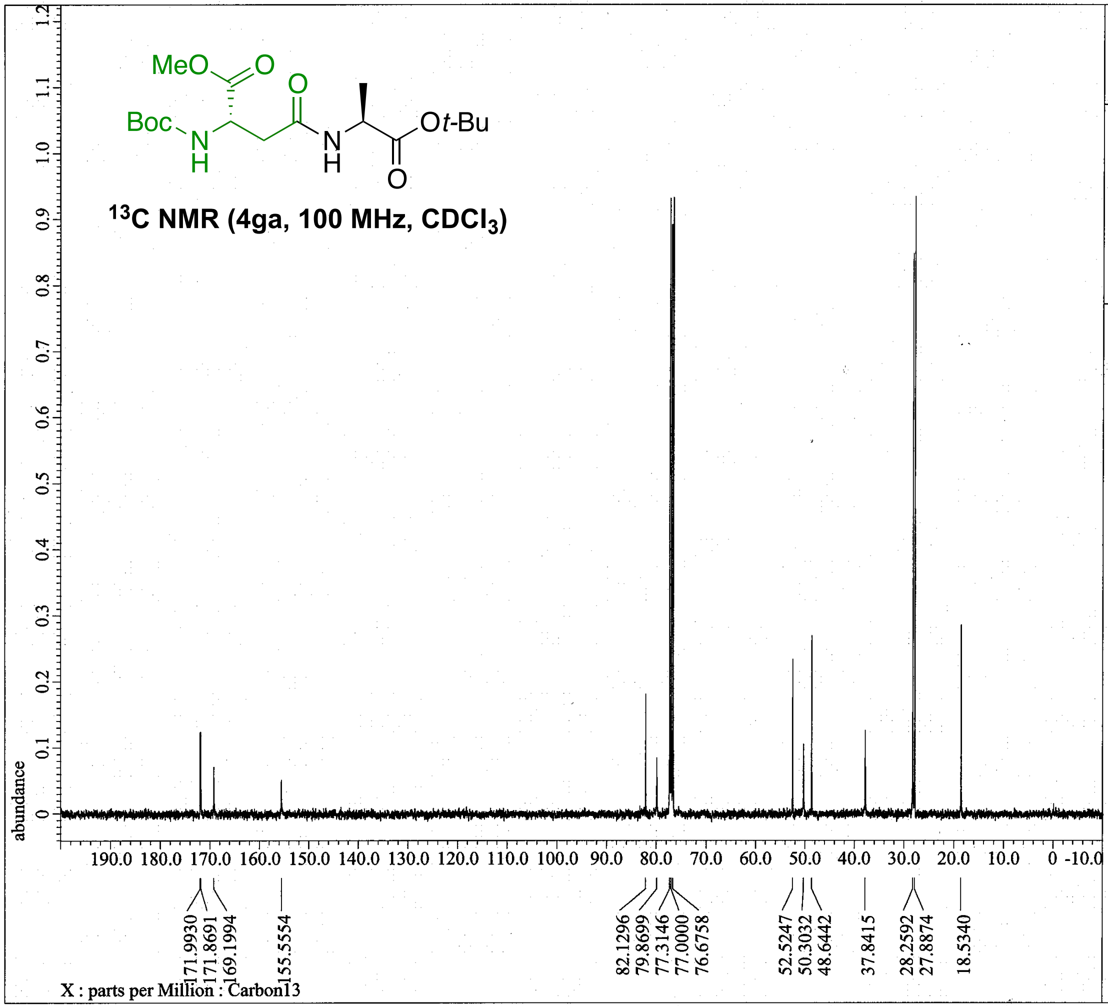
```
Filename          = wm12-45-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-45-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  7-JUL-2021 13:45:06
Revision_Time     =  7-JUL-2021 14:07:26
Current_Time      =  7-JUL-2021 14:07:48

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 513
Total_Scans       = 513

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (4ga, 400 MHz, CDCl$_3$)

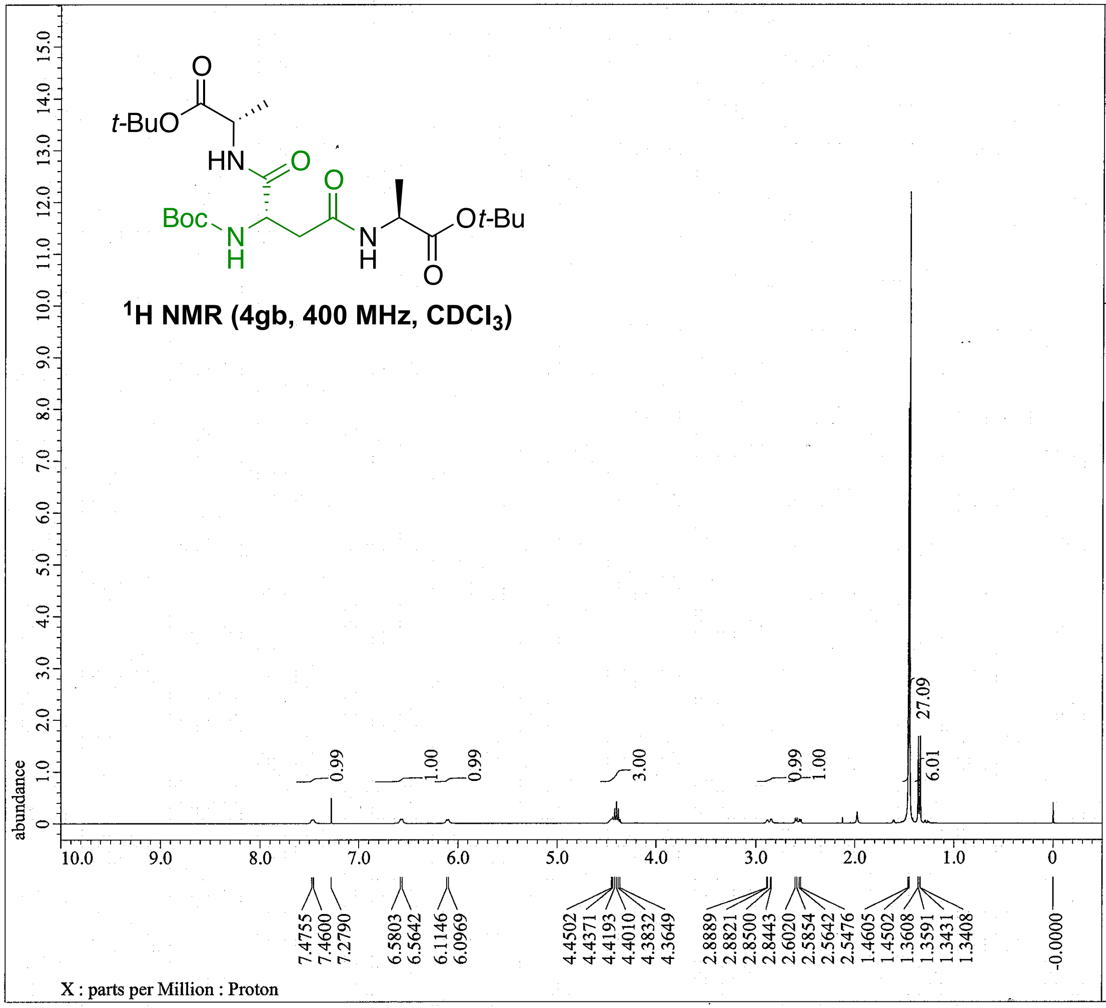
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-154-2_E1H-1-1.jdf
```

```
Filename           = wm12-154-2_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-154-2
Solvent            = CHLOROFORM-D
Creation_Time      = 12-NOV-2021 13:34:34
Revision_Time      = 12-NOV-2021 13:35:08
Current_Time       = 12-NOV-2021 13:35:29

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 24.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (4ga, 100 MHz, CDCl$_3$)

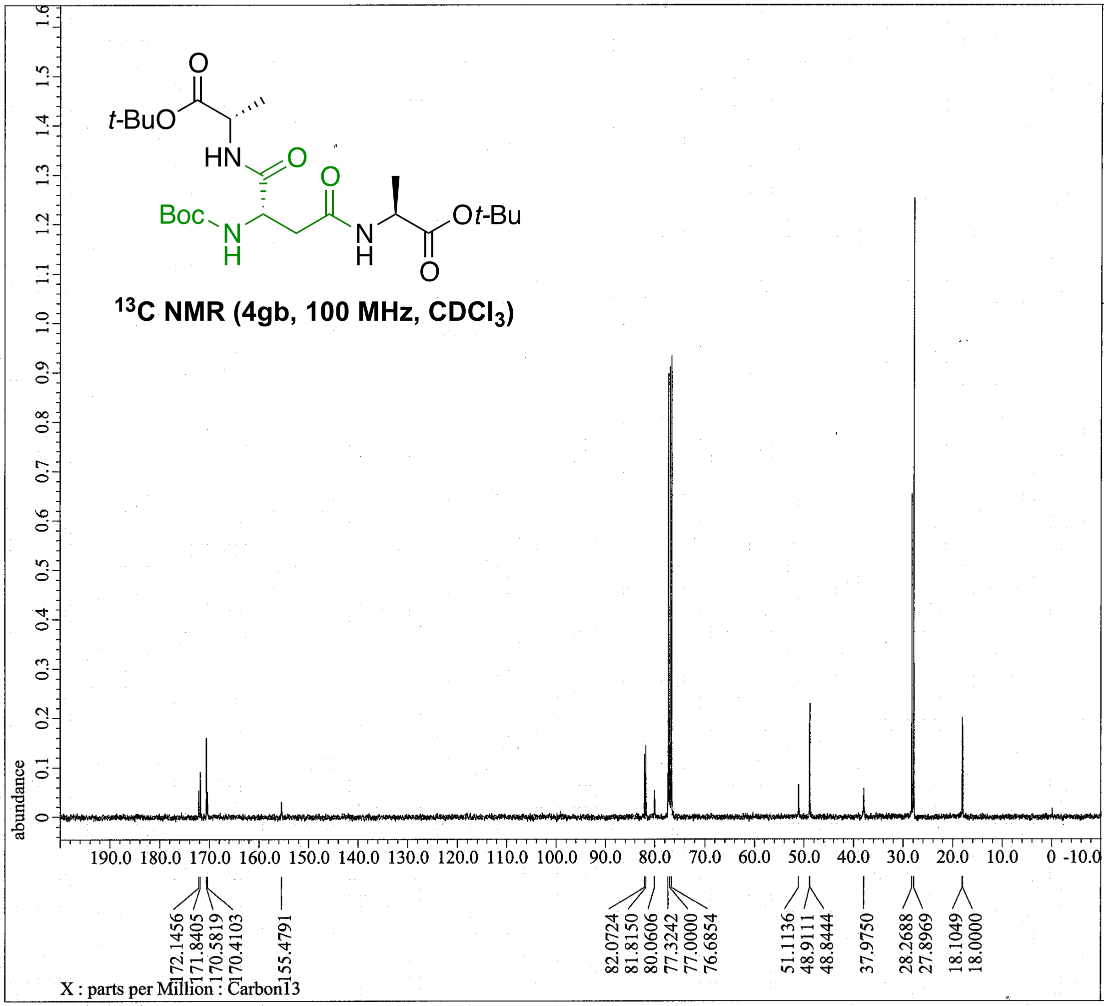
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-154-2-13C_E13C-1-1.jdf

Filename           = wm12-154-2-13C_E13C-1-4.jd
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-154-2-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 12-NOV-2021 13:36:42
Revision_Time      = 12-NOV-2021 13:58:36
Current_Time       = 12-NOV-2021 13:59:38

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = TRUE
Scans              = 512
Total_Scans        = 512

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.5[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (4gb, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-93_E1H-1-1.jdf
```

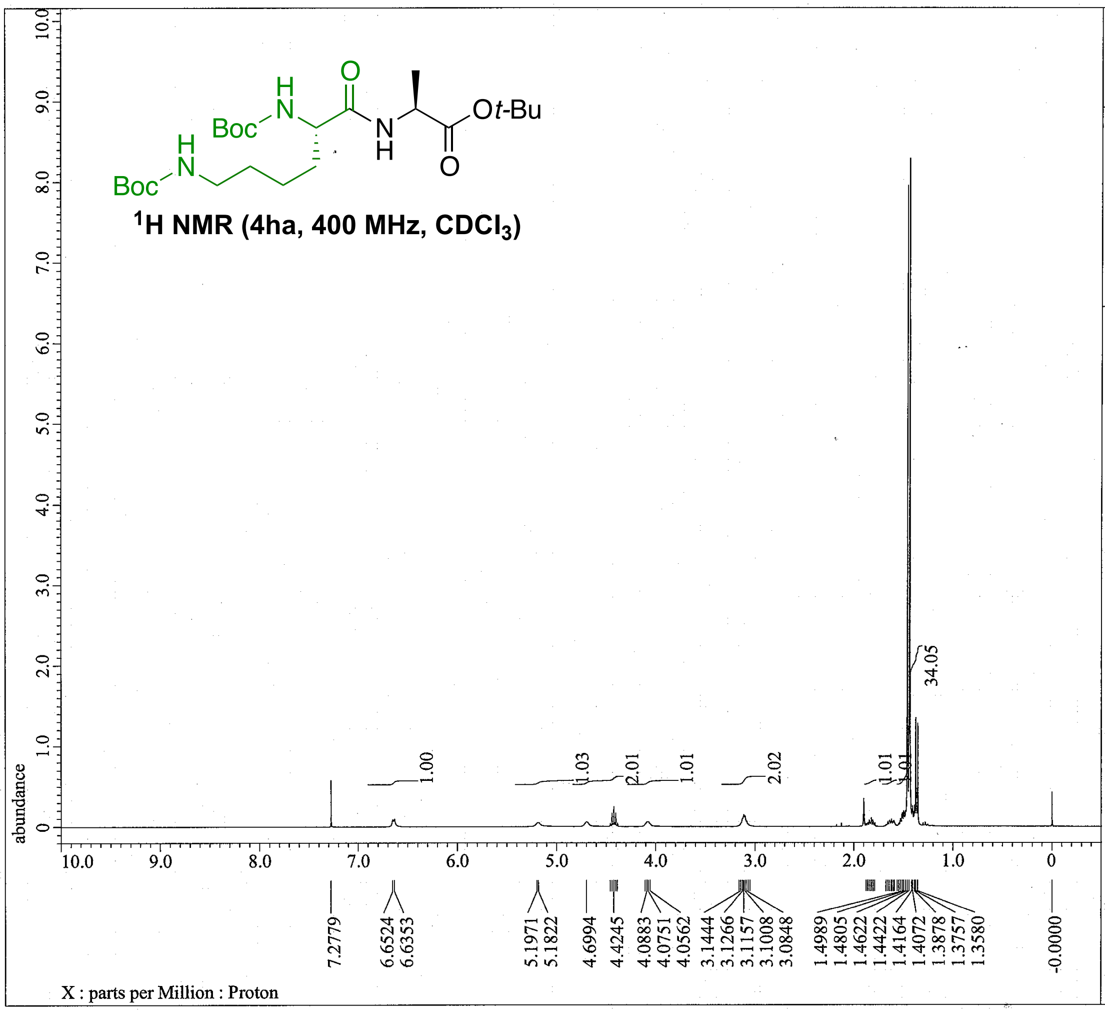
```
Filename          = wm12-93_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-93
Solvent           = CHLOROFORM-D
Creation_Time     = 13-SEP-2021 14:07:51
Revision_Time     = 13-SEP-2021 14:09:47
Current_Time      = 13-SEP-2021 14:10:05

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 24.1[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (4gb, 100 MHz, $CDCl_3$)

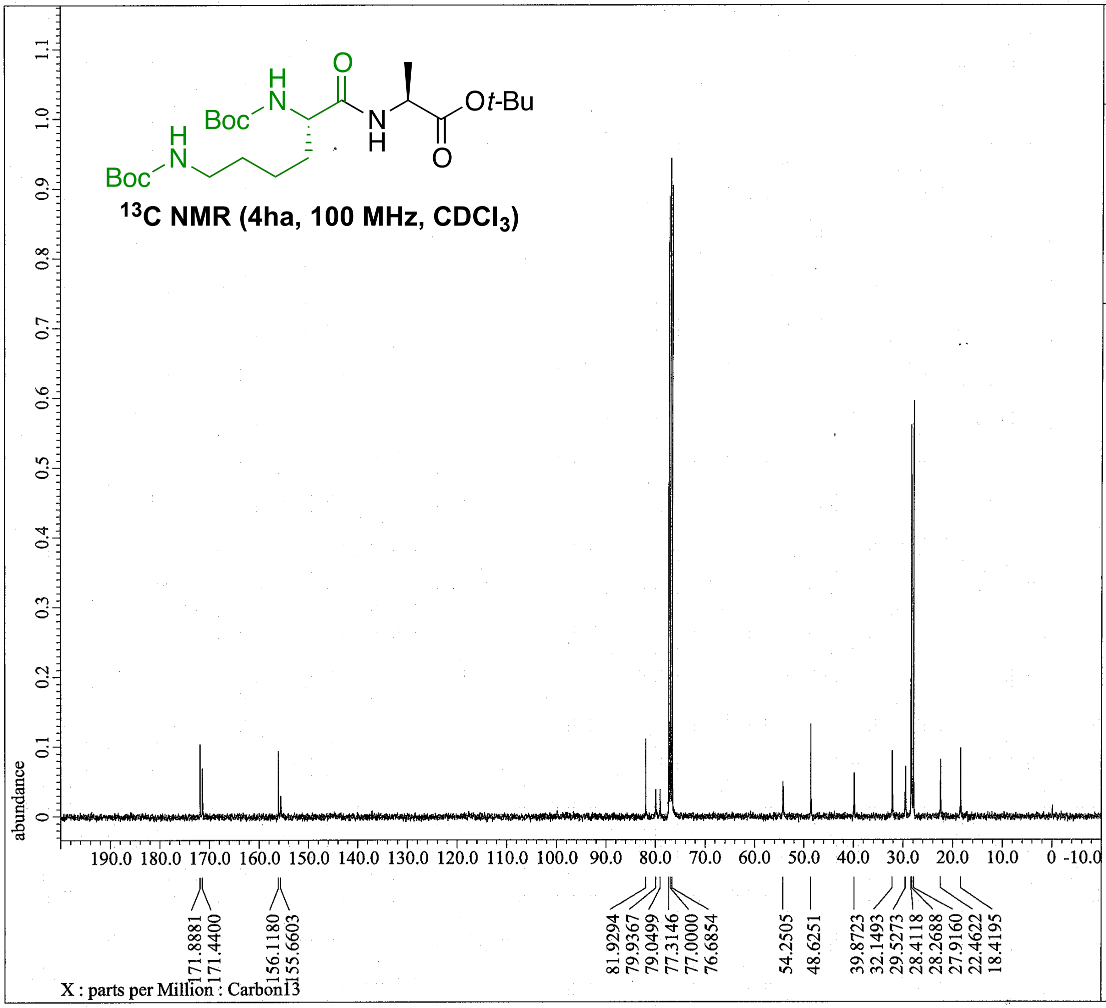
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-93-13C_E13C-1-1.jdf
```

```
Filename           = wm12-93-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-93-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 13-SEP-2021 14:09:58
Revision_Time      = 13-SEP-2021 14:43:45
Current_Time       = 13-SEP-2021 14:44:01

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 741
Total_Scans        = 741

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.3[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (4ha, 400 MHz, CDCl$_3$)

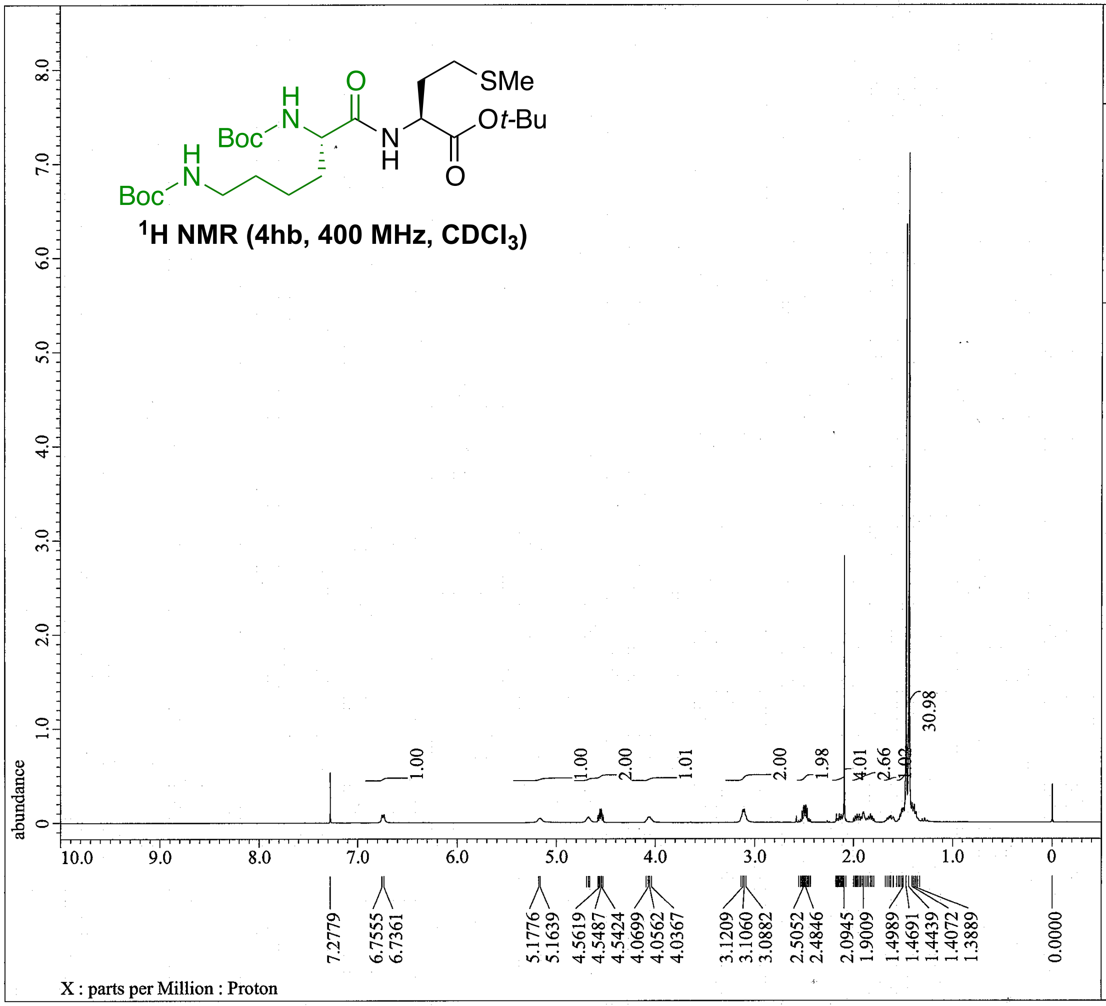
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-73-1_E1H-1-1.jdf

Filename          = wm12-73-1_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-73-1
Solvent           = CHLOROFORM-D
Creation_Time     = 25-AUG-2021 12:23:34
Revision_Time     = 25-AUG-2021 12:27:34
Current_Time      = 25-AUG-2021 12:27:46

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 24[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



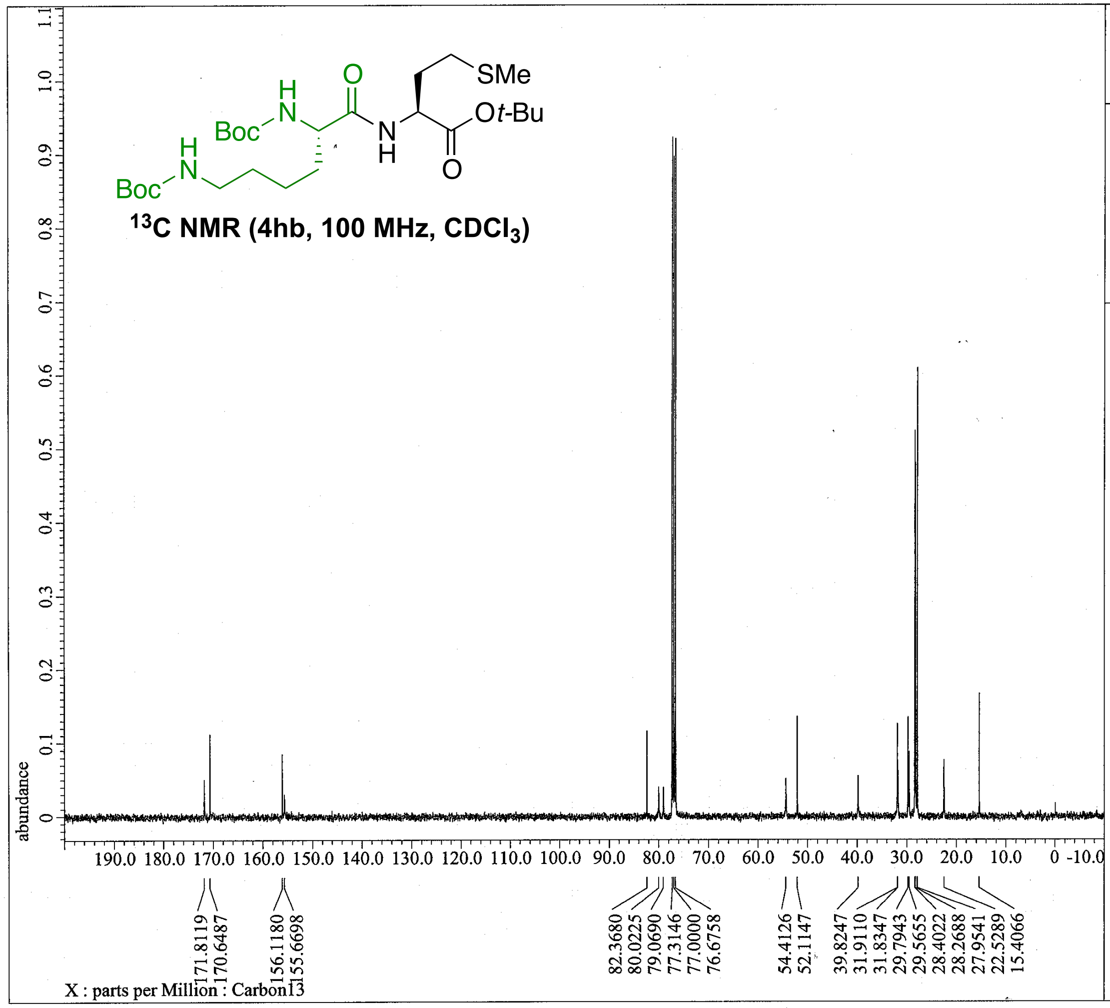
$^{13}$C NMR (4ha, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-73-1-13C_E13C-1-1.jdf
```

```
Filename            = wm12-73-1-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-73-1-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 25-AUG-2021 12:25:47
Revision_Time       = 25-AUG-2021 13:13:49
Current_Time        = 25-AUG-2021 13:14:10

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = TRUE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 23.7[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



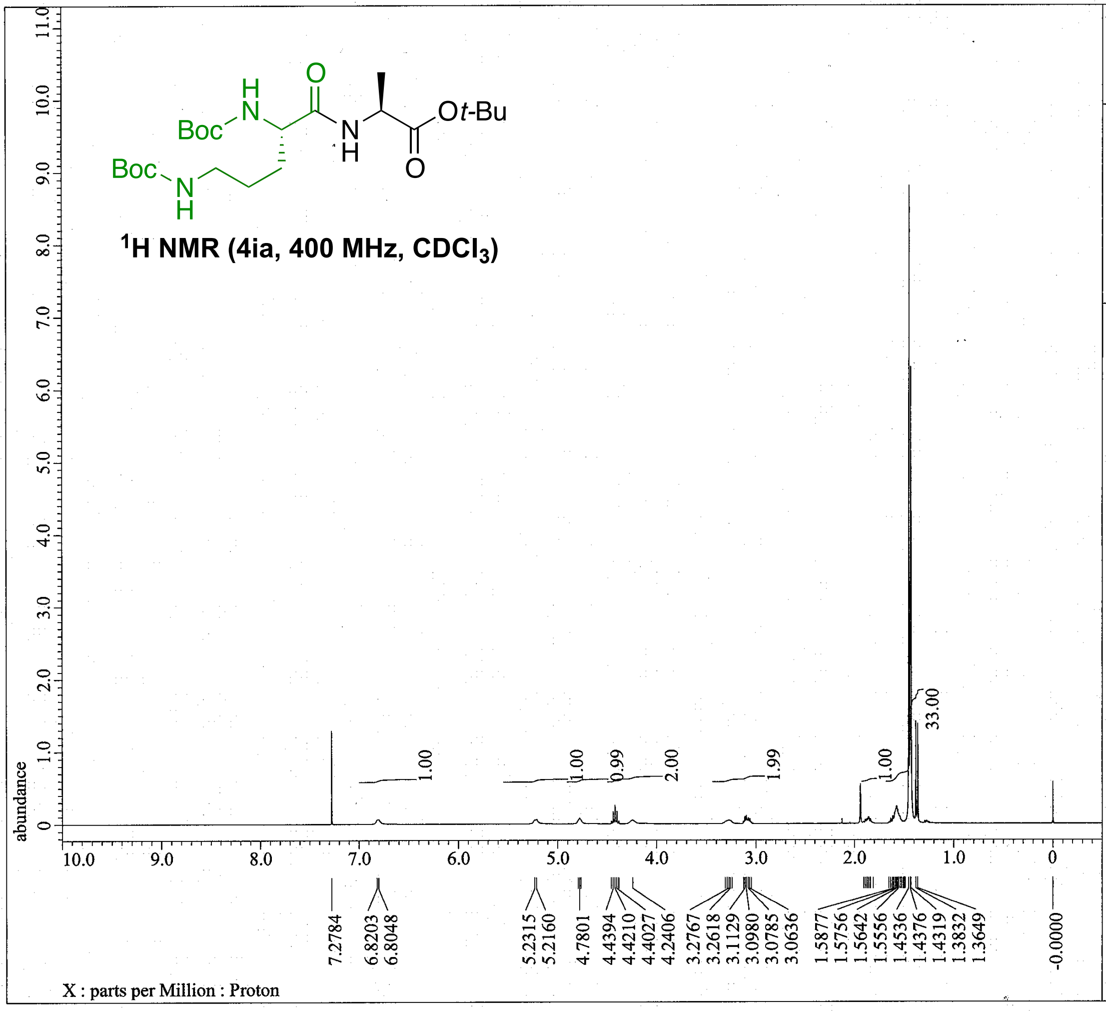
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-73-3_E1H-1-1.jdf

Filename           = wm12-73-3_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-73-3
Solvent            = CHLOROFORM-D
Creation_Time      = 26-AUG-2021 16:08:09
Revision_Time      = 26-AUG-2021 16:16:31
Current_Time       = 26-AUG-2021 16:16:42

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 26
Temp_Get           = 24.6[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}C$ NMR (4hb, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-73-3-13C_E13C-1-1.jdf
```

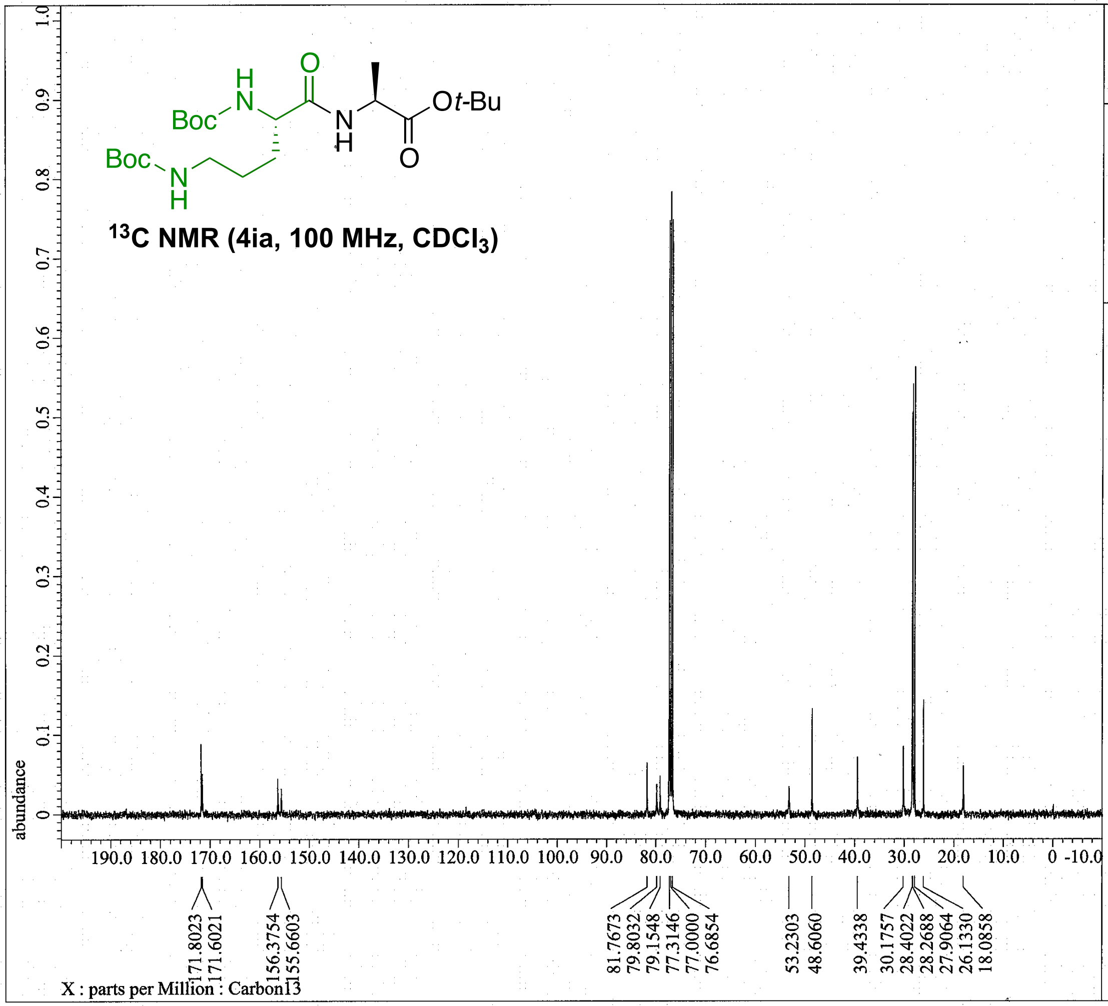
```
Filename            = wm12-73-3-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-73-3-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 26-AUG-2021 16:10:17
Revision_Time       = 26-AUG-2021 16:58:30
Current_Time        = 26-AUG-2021 16:58:49

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 24.1[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (4ia, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-92-1_E1H-1-1.jdf
```

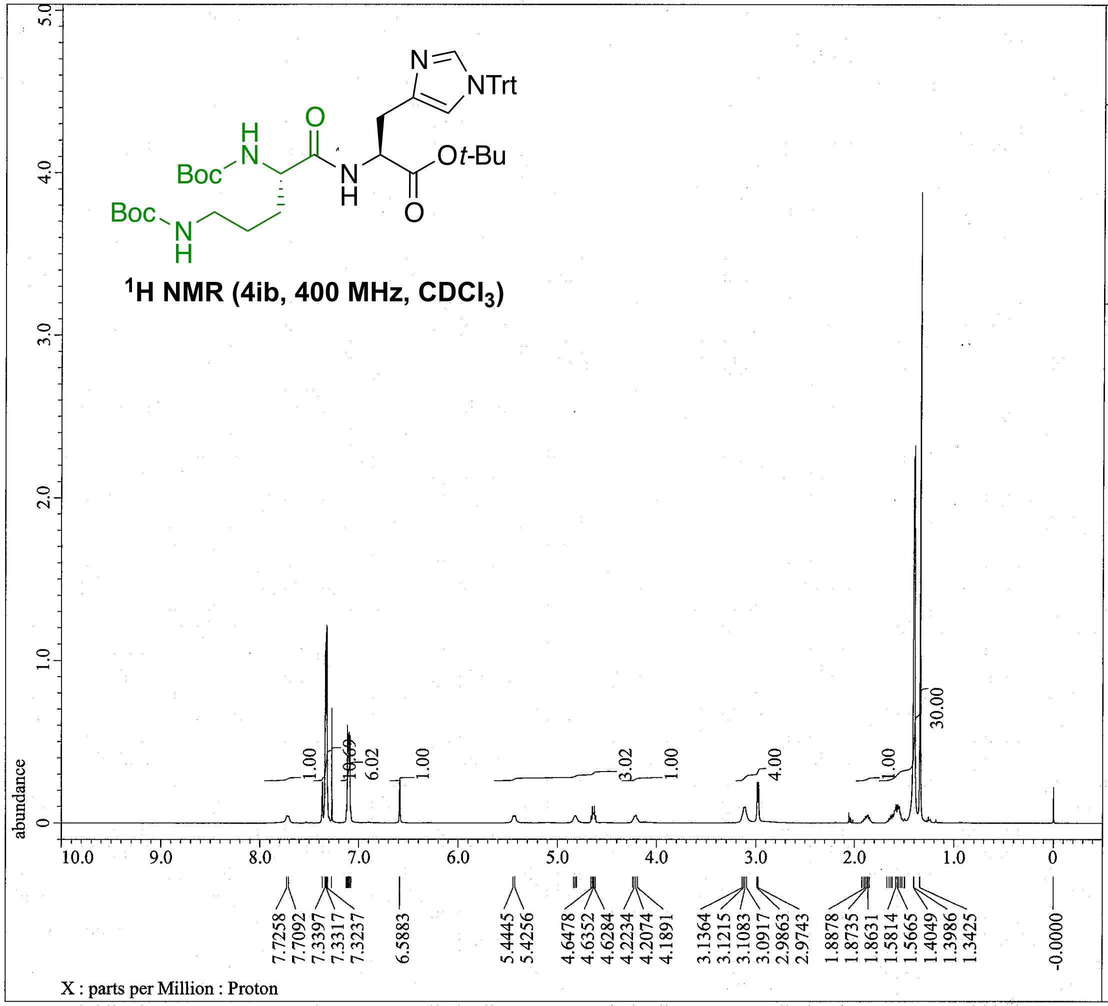
```
Filename           = wm12-92-1_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-92-1
Solvent            = CHLOROFORM-D
Creation_Time      = 12-SEP-2021 09:09:13
Revision_Time      = 12-SEP-2021 09:13:13
Current_Time       = 12-SEP-2021 09:13:43

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 23.8[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (4ia, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-92-1-13C_E13C-1-1.jdf
```

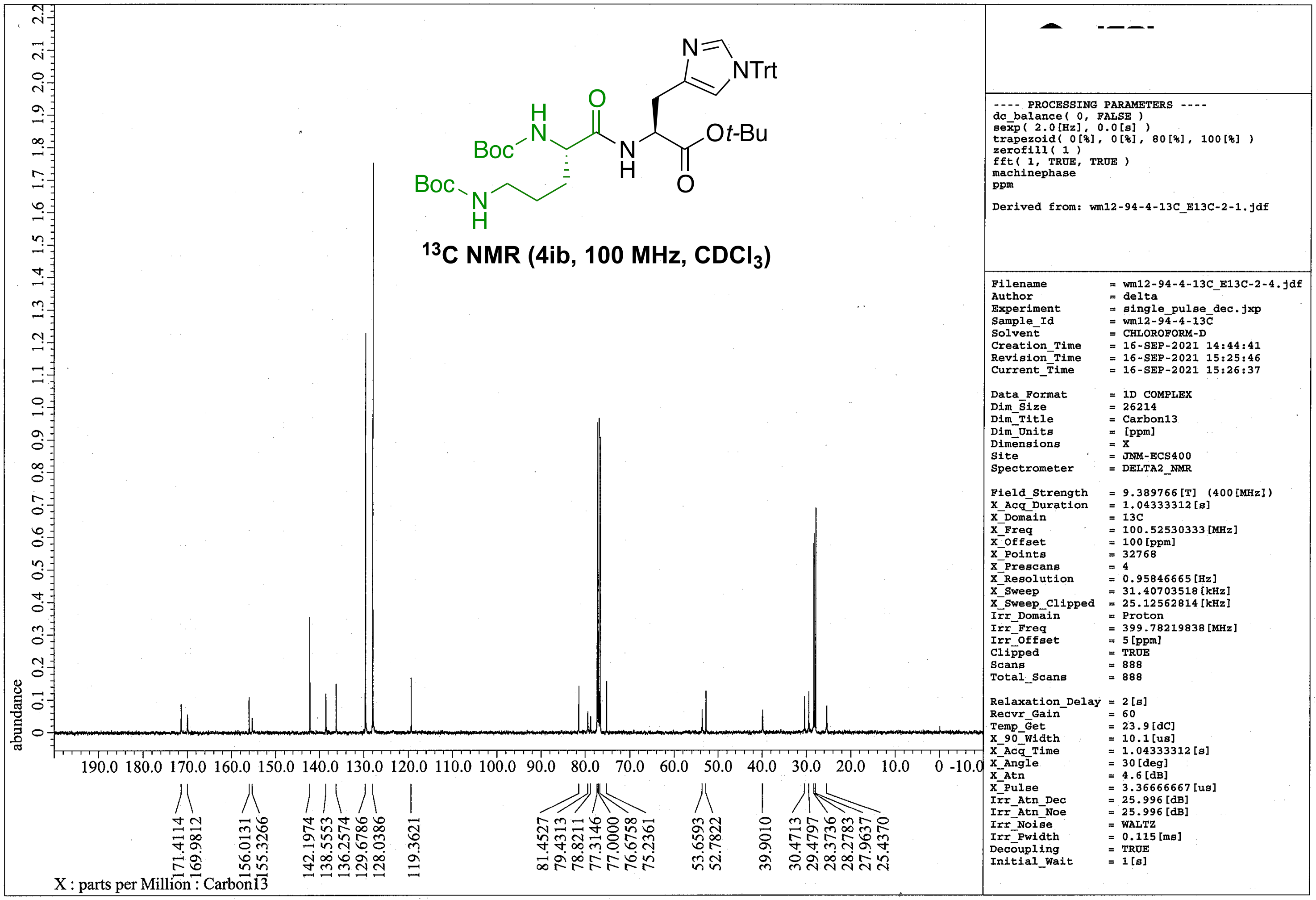
```
Filename            = wm12-92-1-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-92-1-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 12-SEP-2021 09:11:18
Revision_Time       = 12-SEP-2021 09:59:06
Current_Time        = 12-SEP-2021 09:59:25

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 24.1[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (4ib, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-94-4_E1H-2-1.jdf
```

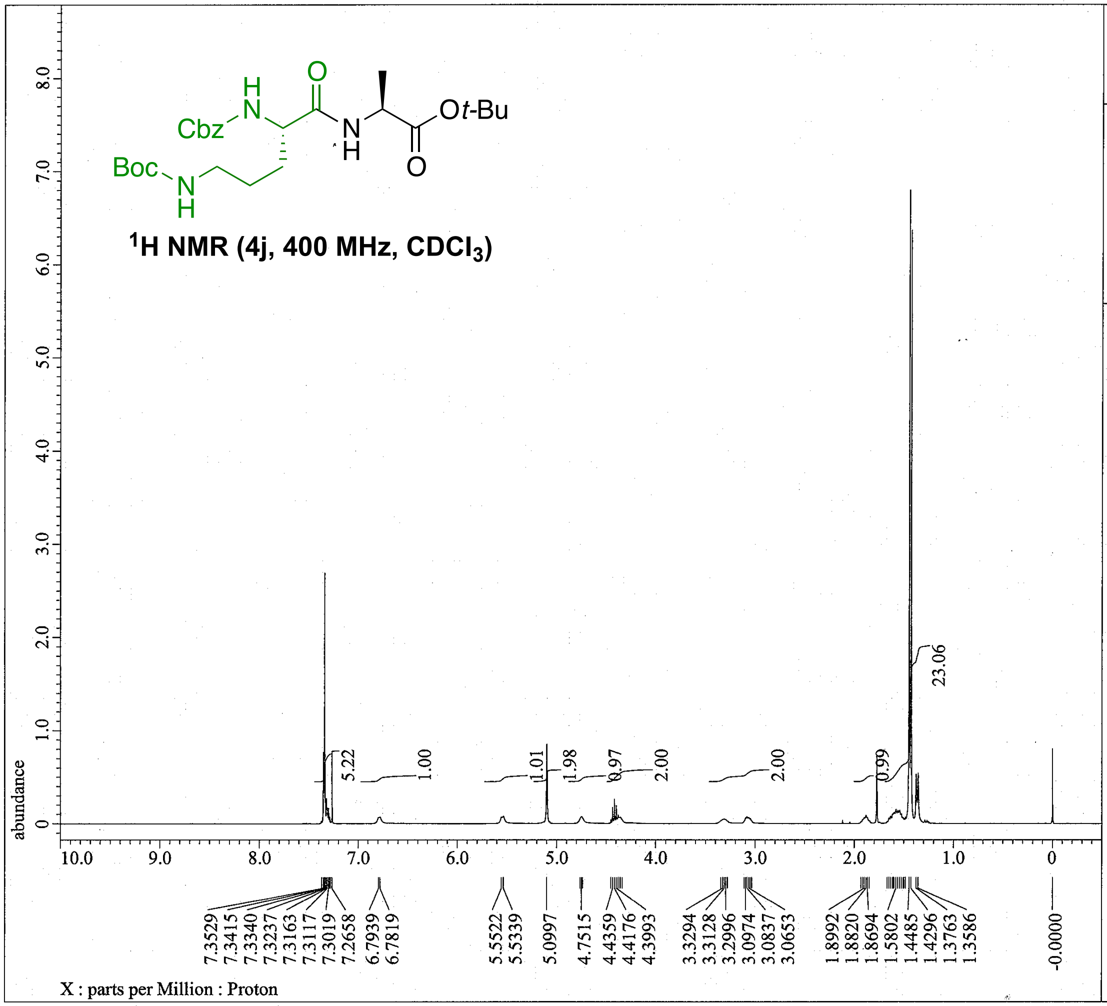
```
Filename          = wm12-94-4_E1H-2-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-94-4
Solvent           = CHLOROFORM-D
Creation_Time     = 16-SEP-2021 14:42:25
Revision_Time     = 16-SEP-2021 17:12:04
Current_Time      = 16-SEP-2021 17:12:25

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 22
Temp_Get          = 24[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (4ib, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-94-4-13C_E13C-2-1.jdf
```

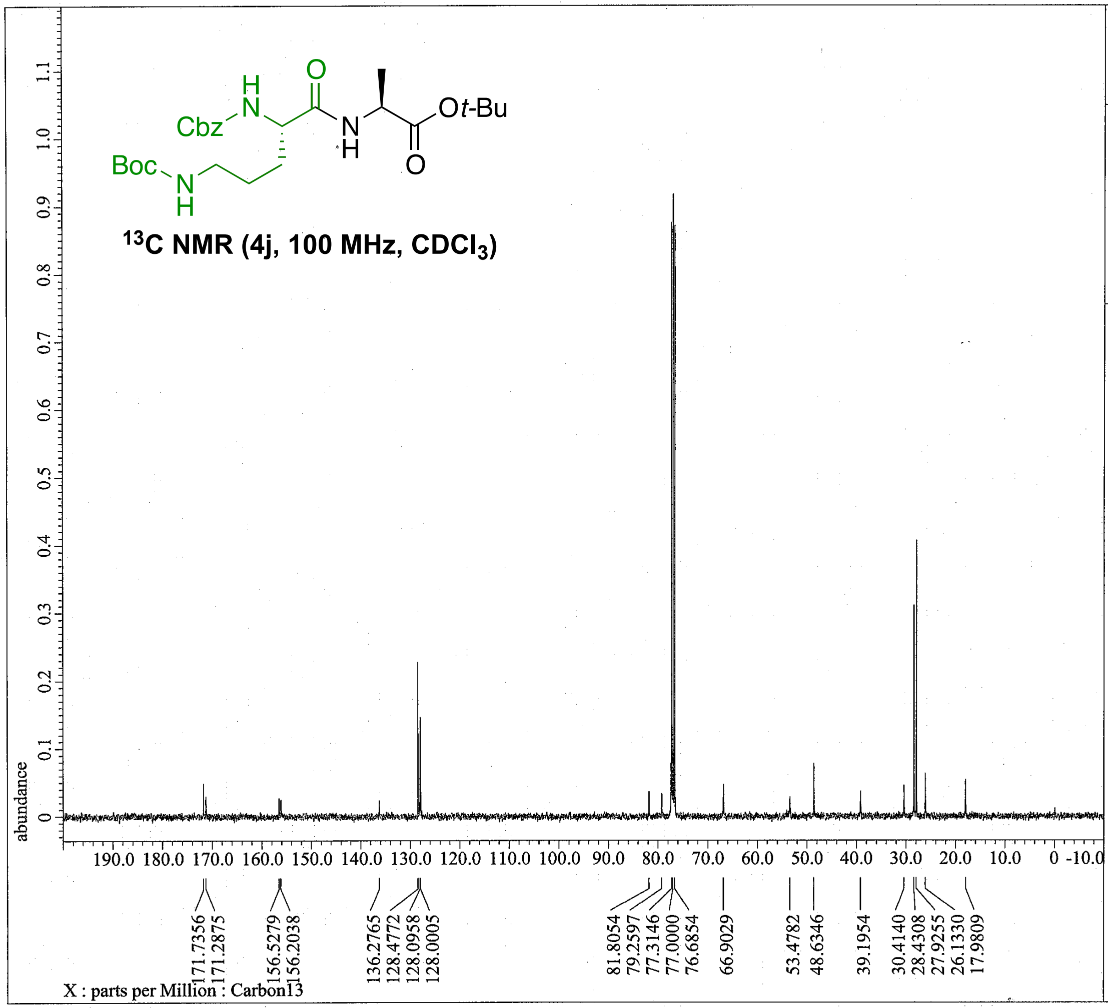
```
Filename           = wm12-94-4-13C_E13C-2-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-94-4-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 16-SEP-2021 14:44:41
Revision_Time      = 16-SEP-2021 15:25:46
Current_Time       = 16-SEP-2021 15:26:37

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = TRUE
Scans              = 888
Total_Scans        = 888

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.9[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (4j, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-89-5_E1H-1-1.jdf
```

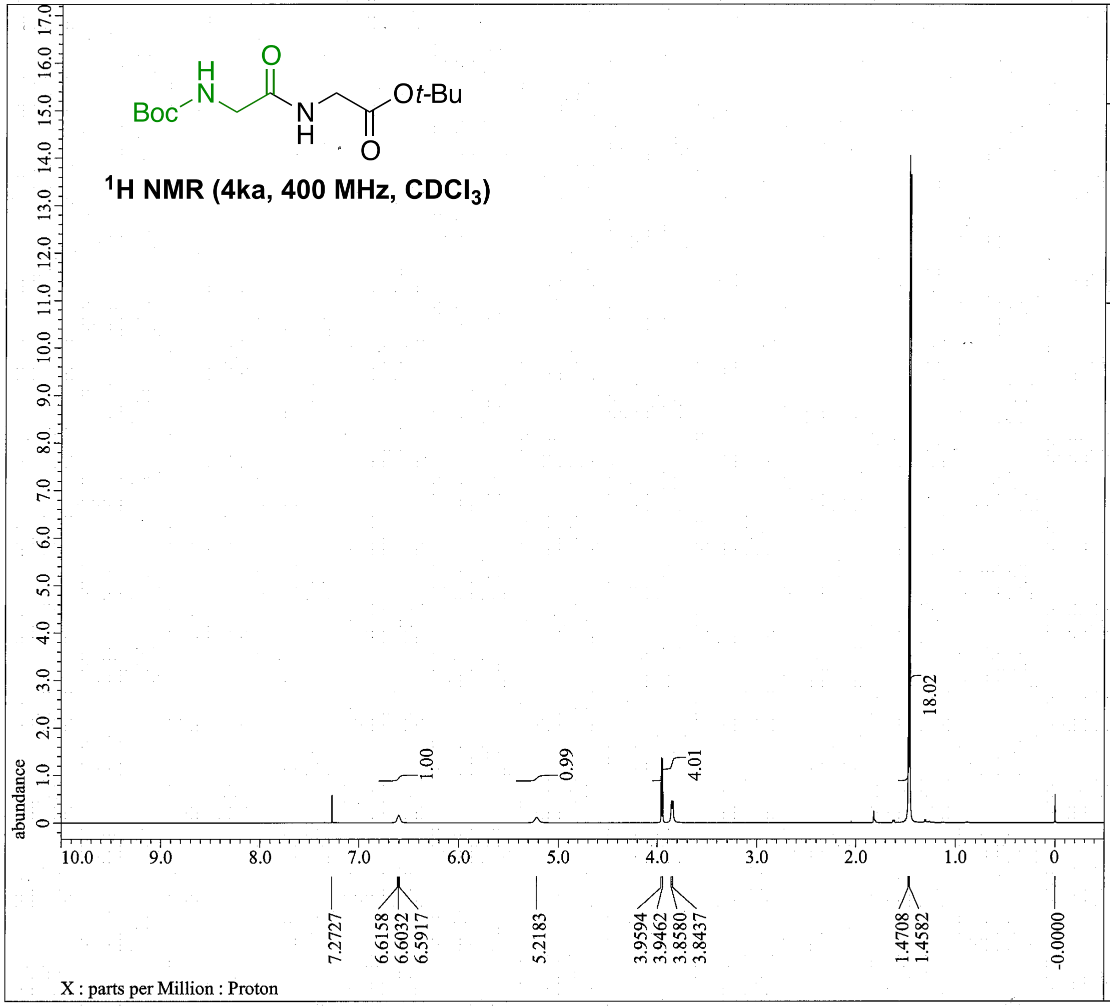
```
Filename          = wm12-89-5_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-89-5
Solvent           = CHLOROFORM-D
Creation_Time     = 10-SEP-2021 13:05:08
Revision_Time     = 10-SEP-2021 13:08:50
Current_Time      = 10-SEP-2021 13:08:59

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 24.1[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (4j, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-89-5-13C_E13C-1-1.jdf
```

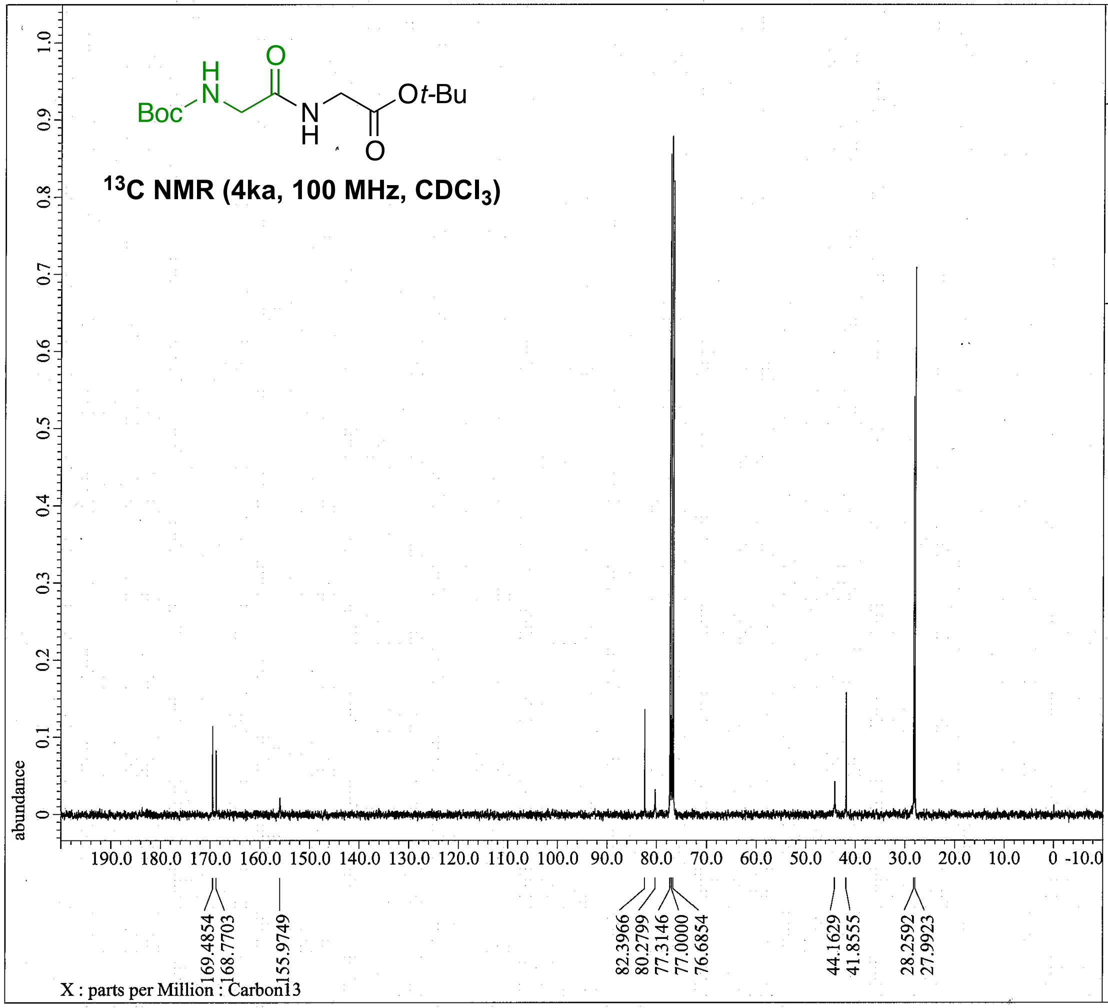
```
Filename           = wm12-89-5-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-89-5-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 10-SEP-2021 13:07:27
Revision_Time      = 10-SEP-2021 13:57:10
Current_Time       = 10-SEP-2021 13:57:30

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1024
Total_Scans        = 1024

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.8[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (4ka, 400 MHz, CDCl$_3$)

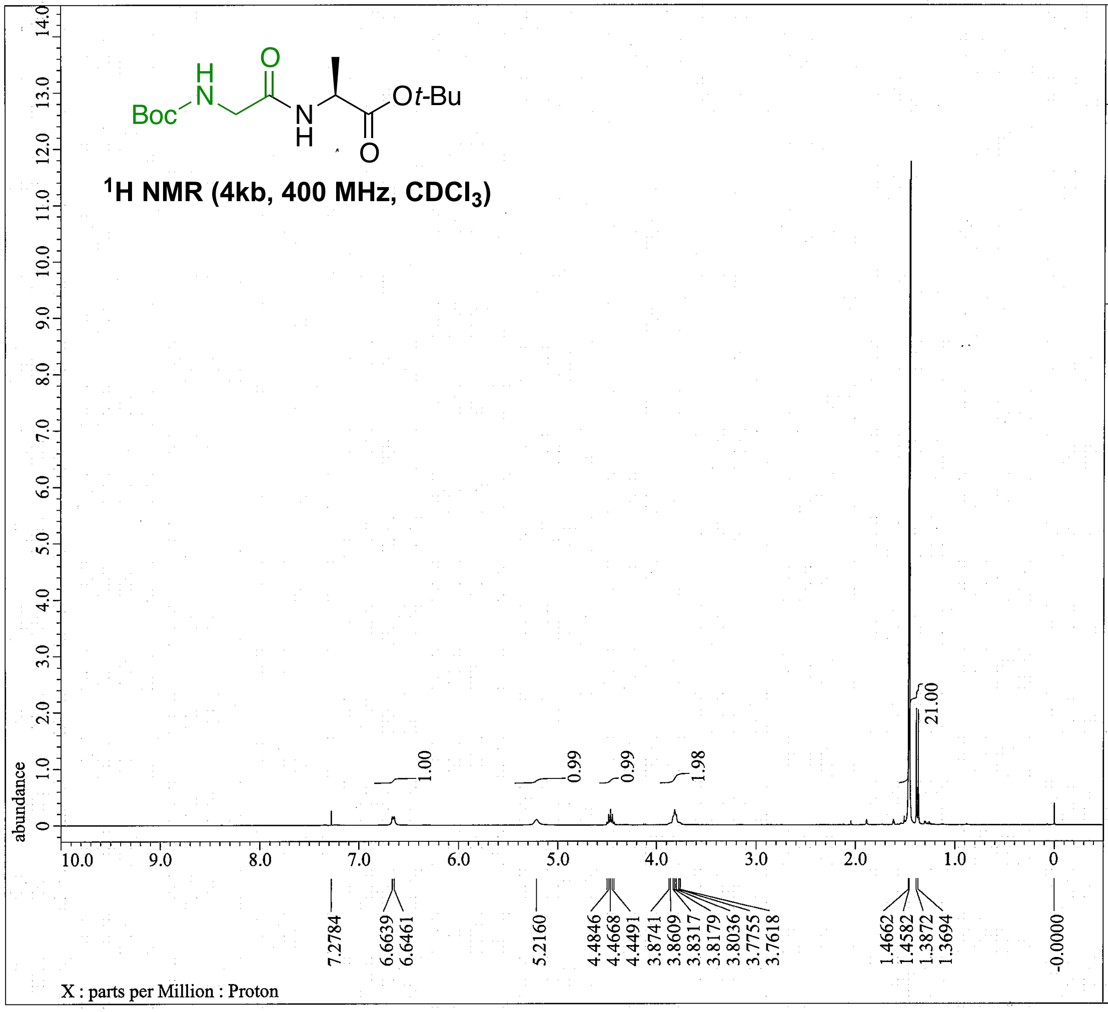
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-150-2_E1H-1-1.jdf
```

```
Filename          = wm11-150-2_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm11-150-2
Solvent           = CHLOROFORM-D
Creation_Time     =  1-APR-2021 14:12:13
Revision_Time     =  1-APR-2021 14:13:35
Current_Time      =  1-APR-2021 14:13:46

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.9[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```


$^{13}$C NMR (4ka, 100 MHz, CDCl$_3$)

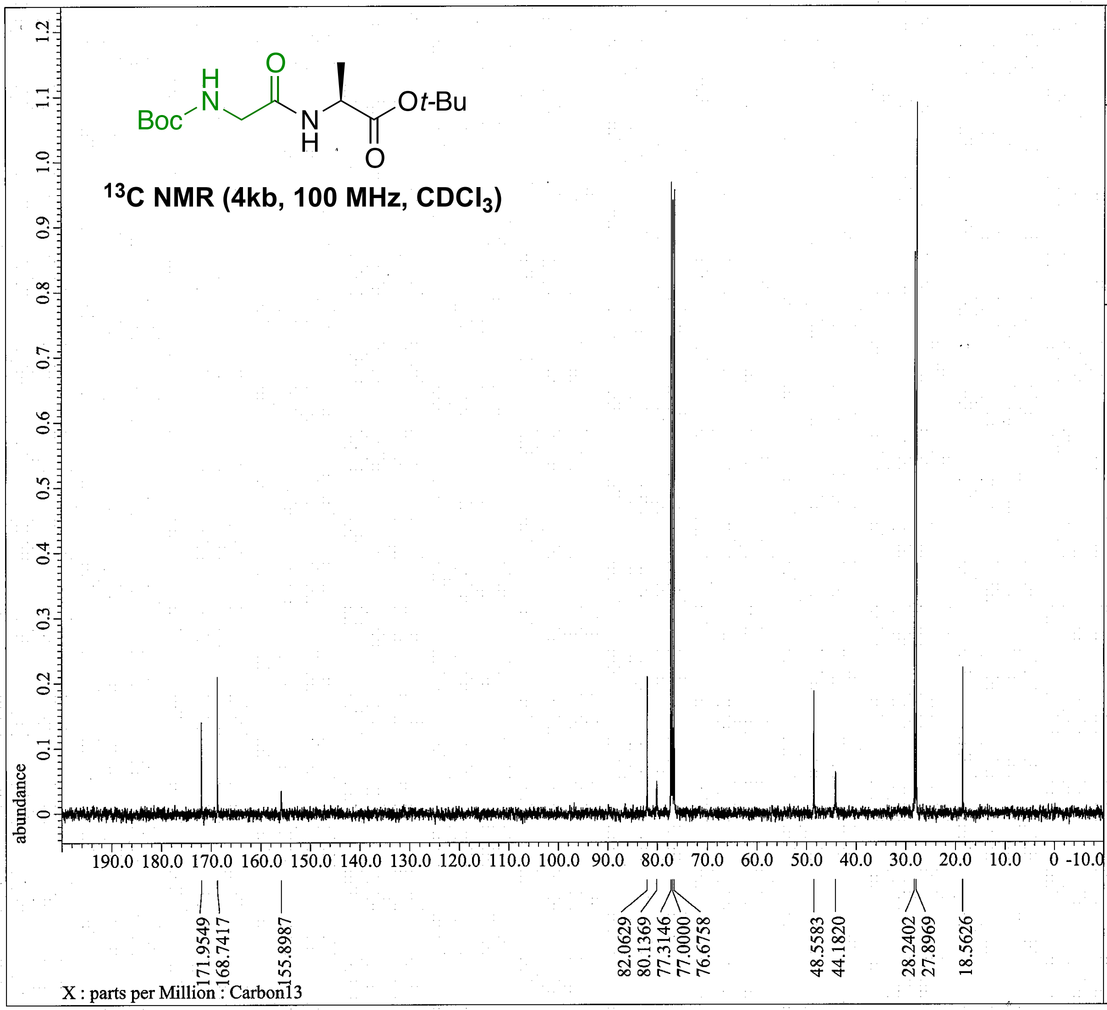
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-150-2-13C_E13C-1-1.jdf
```

```
Filename           = wm11-150-2-13C_E13C-1-3.jd
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm11-150-2-13C
Solvent            = CHLOROFORM-D
Creation_Time      =  1-APR-2021 14:19:18
Revision_Time      =  1-APR-2021 14:51:21
Current_Time       =  1-APR-2021 14:51:49

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 705
Total_Scans        = 705

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.5[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm136-2-2_E1H-2-1.jdf
```

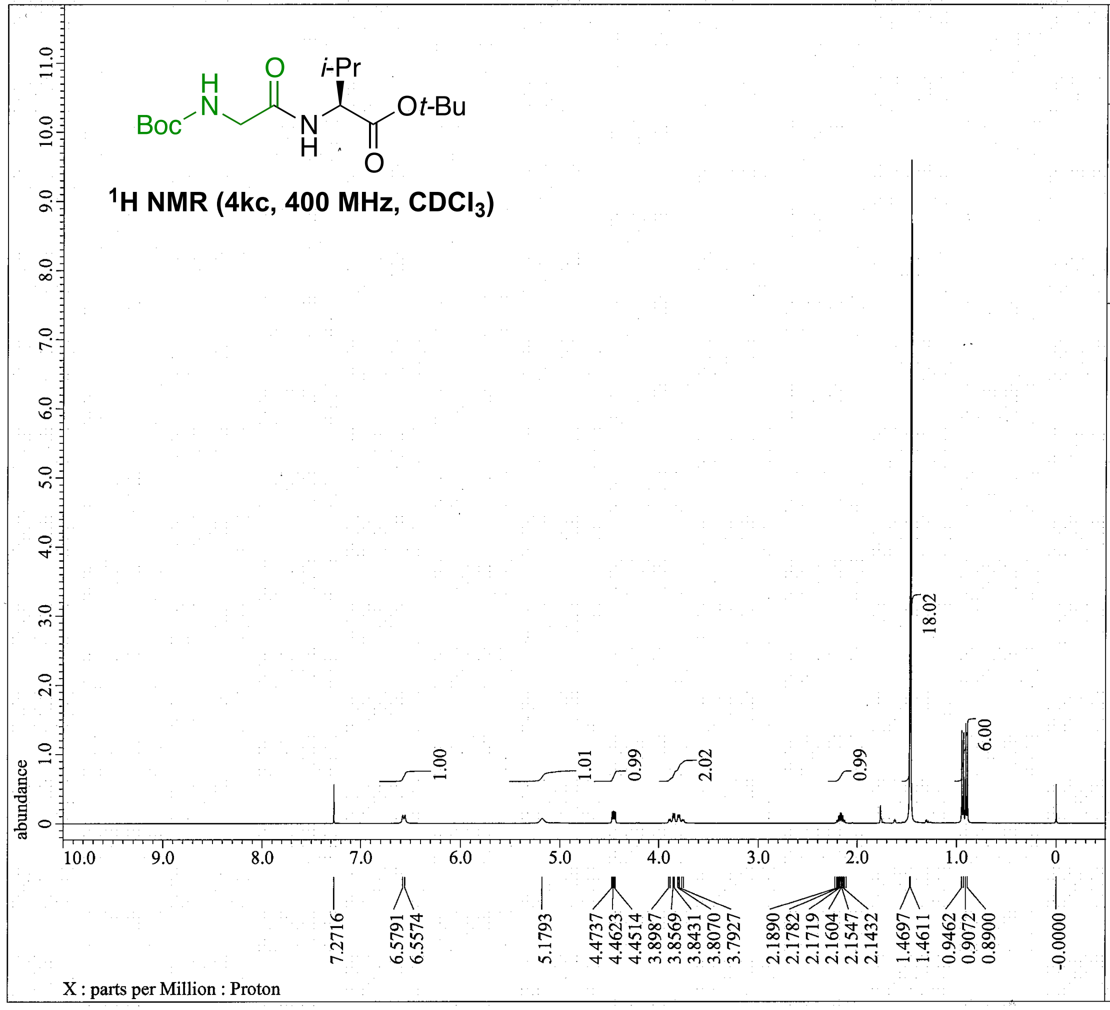
```
Filename           = wm136-2-2_E1H-2-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm136-2-2
Solvent            = CHLOROFORM-D
Creation_Time      = 15-MAR-2021 13:22:46
Revision_Time      = 15-MAR-2021 13:23:50
Current_Time       = 15-MAR-2021 13:24:09

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 24.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (4kb, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm136-2-2-13C_E13C-1-1.jdf
```

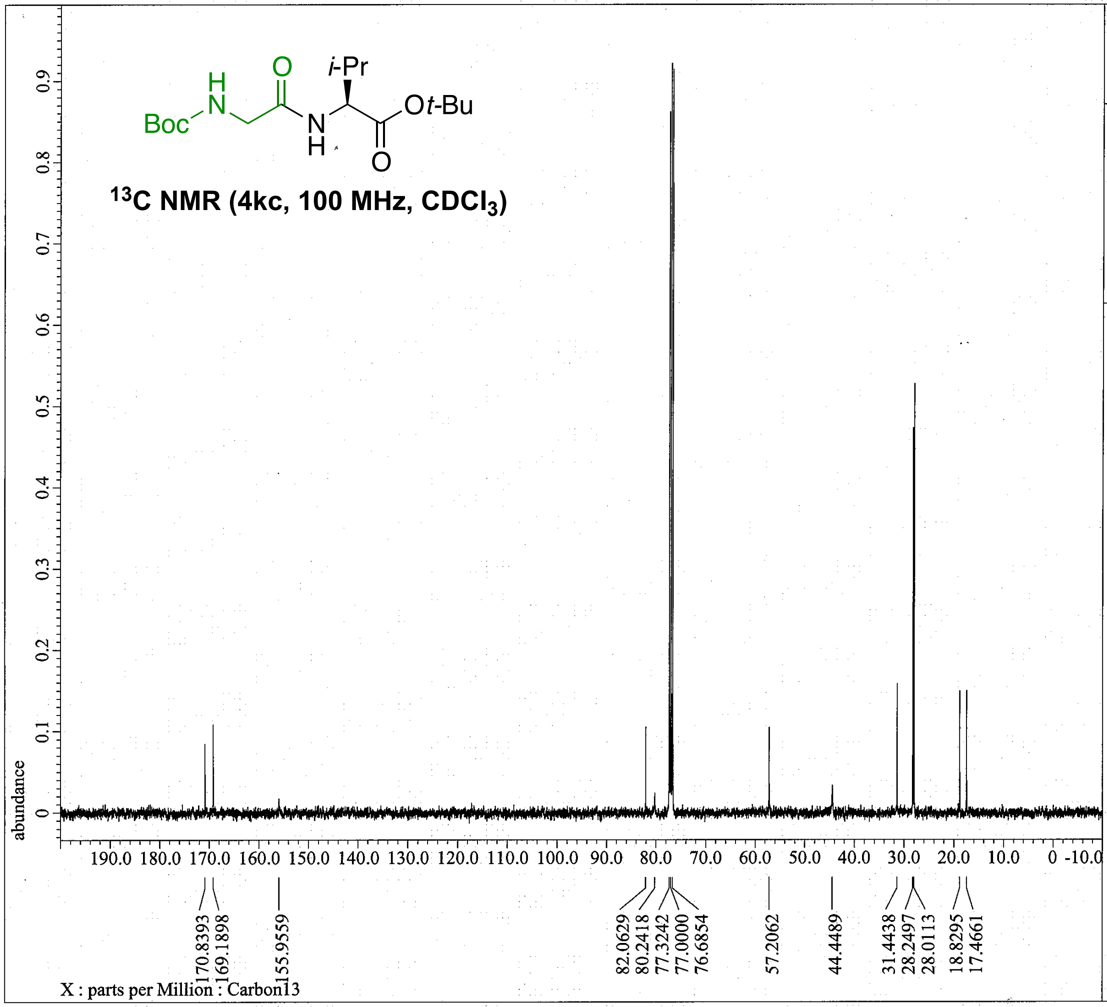
```
Filename           = wm136-2-2-13C_E13C-1-3.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm136-2-2-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 15-MAR-2021 13:27:04
Revision_Time      = 15-MAR-2021 13:36:43
Current_Time       = 15-MAR-2021 13:37:05

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = TRUE
Scans              = 256
Total_Scans        = 256

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.4[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1H$ NMR (4kc, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-149-2_E1H-1-1.jdf
```

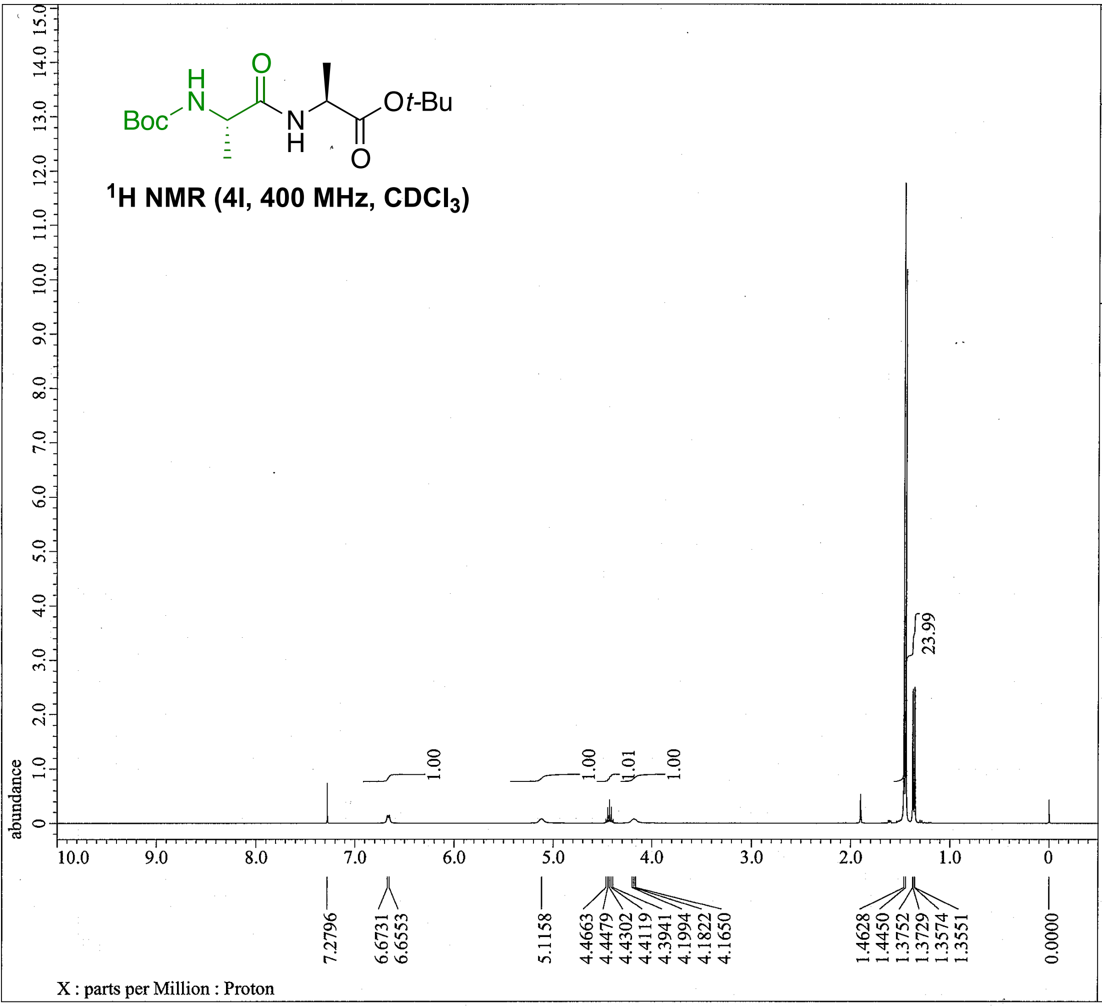
```
Filename            = wm11-149-2_E1H-1-5.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm11-149-2
Solvent             = CHLOROFORM-D
Creation_Time       = 30-MAR-2021 13:32:10
Revision_Time       = 30-MAR-2021 13:33:18
Current_Time        = 30-MAR-2021 13:33:30

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay = 5[s]
Recvr_Gain          = 30
Temp_Get            = 22.2[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (4kc, 100 MHz, CDCl$_3$)

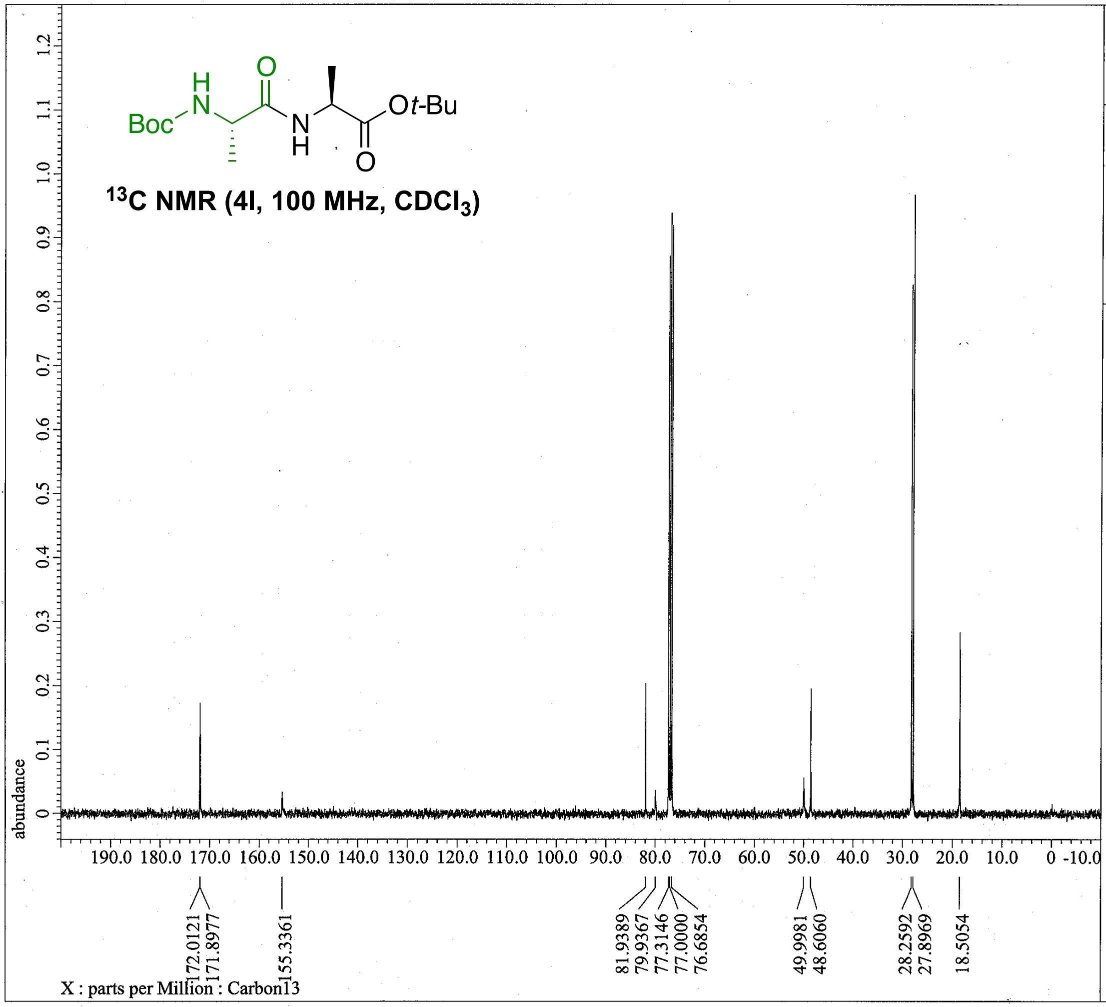
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-149-2-13C_E13C-1-1.jdf
```

```
Filename          = wm11-149-2-13C_E13C-1-6.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm11-149-2-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 30-MAR-2021 13:37:11
Revision_Time     = 30-MAR-2021 14:00:11
Current_Time      = 30-MAR-2021 14:00:45

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.9[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



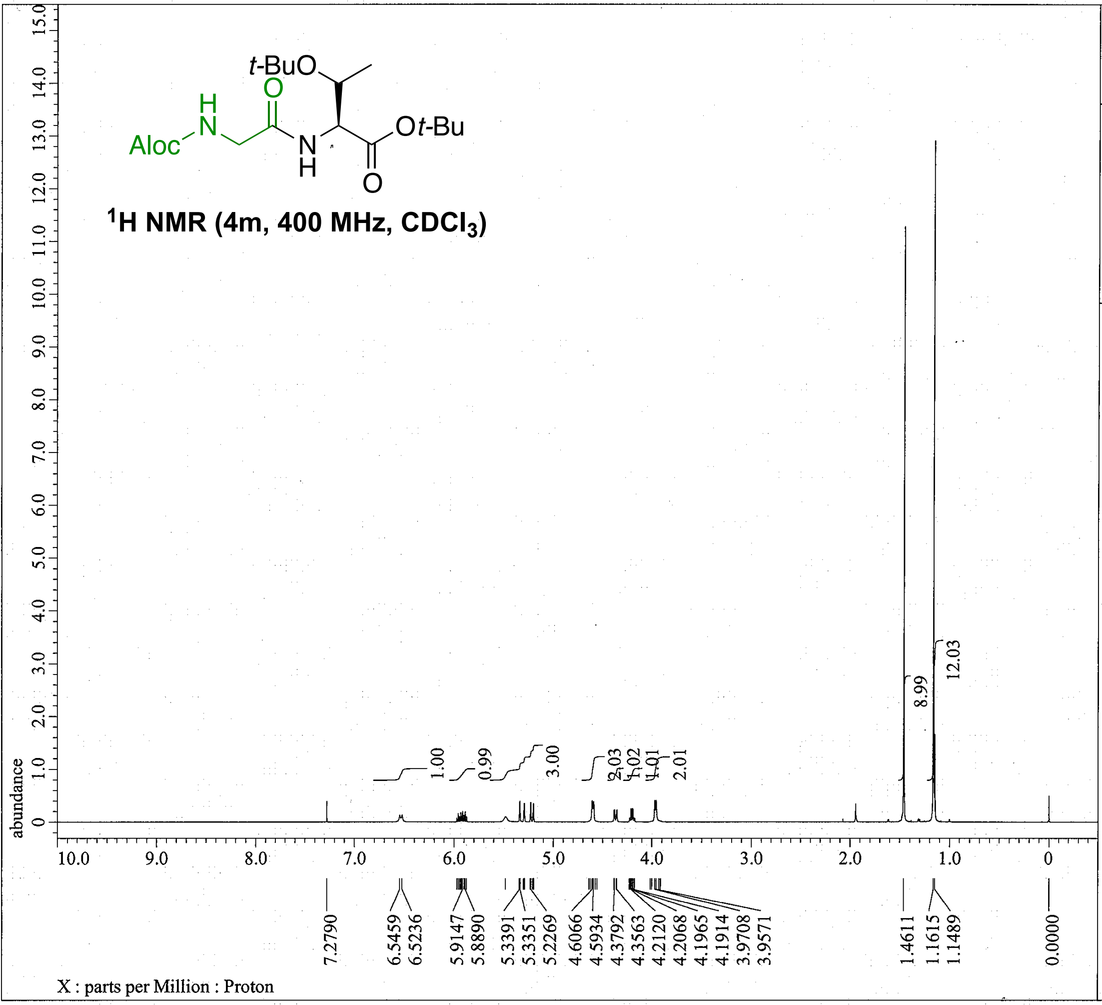
$^{1}$H NMR (4l, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-109-2_E1H-1-1.jdf

Filename          = wm12-109-2_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-109-2
Solvent           = CHLOROFORM-D
Creation_Time     =  1-OCT-2021 16:06:47
Revision_Time     =  1-OCT-2021 16:07:11
Current_Time      =  1-OCT-2021 16:07:30

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 23.6[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (4l, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-109-2-13C_E13C-1-1.jdf

Filename          = wm12-109-2-13C_E13C-1-4.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-109-2-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  1-OCT-2021 16:08:53
Revision_Time     =  1-OCT-2021 16:31:11
Current_Time      =  1-OCT-2021 16:31:36

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```

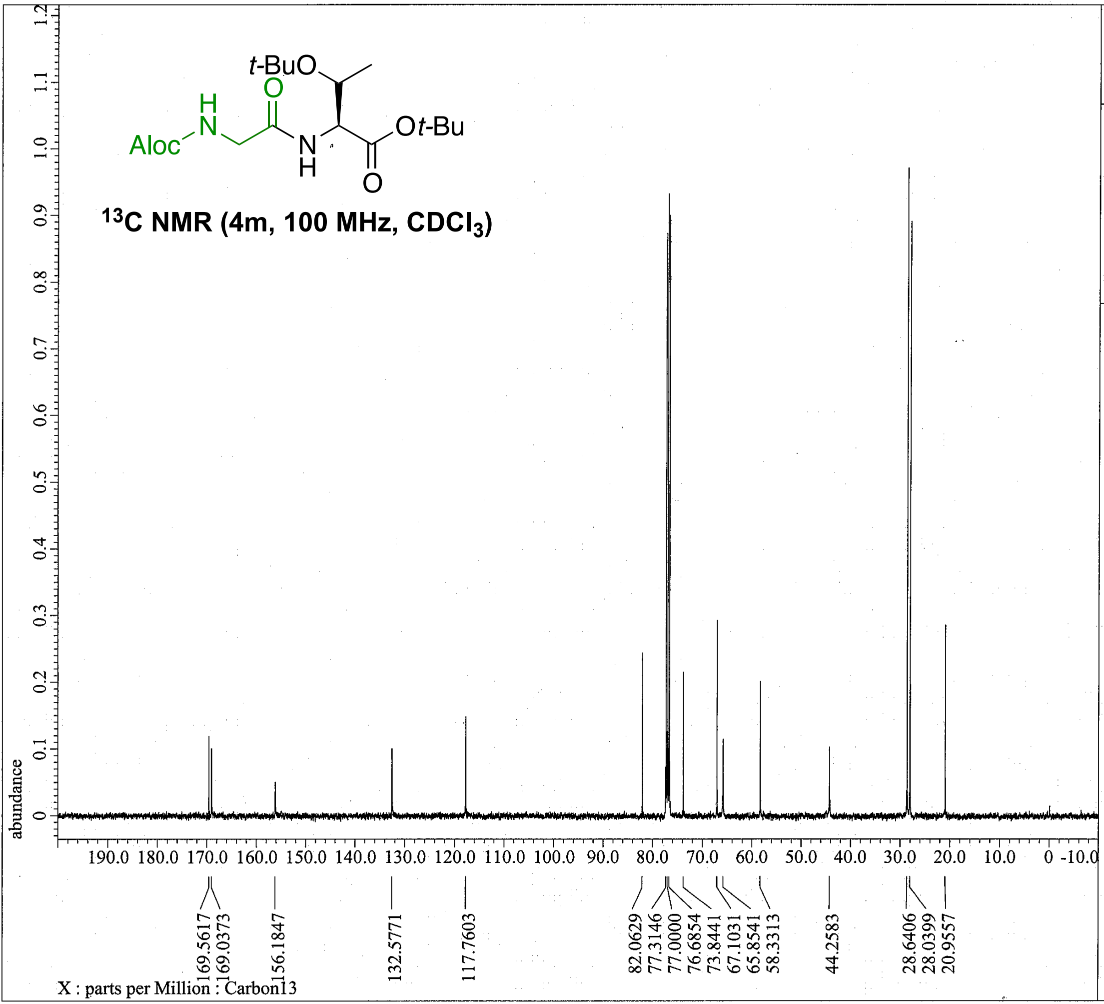


<sup></sup>

$^1$H NMR (4m, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-204_E1H-1-1.jdf
```

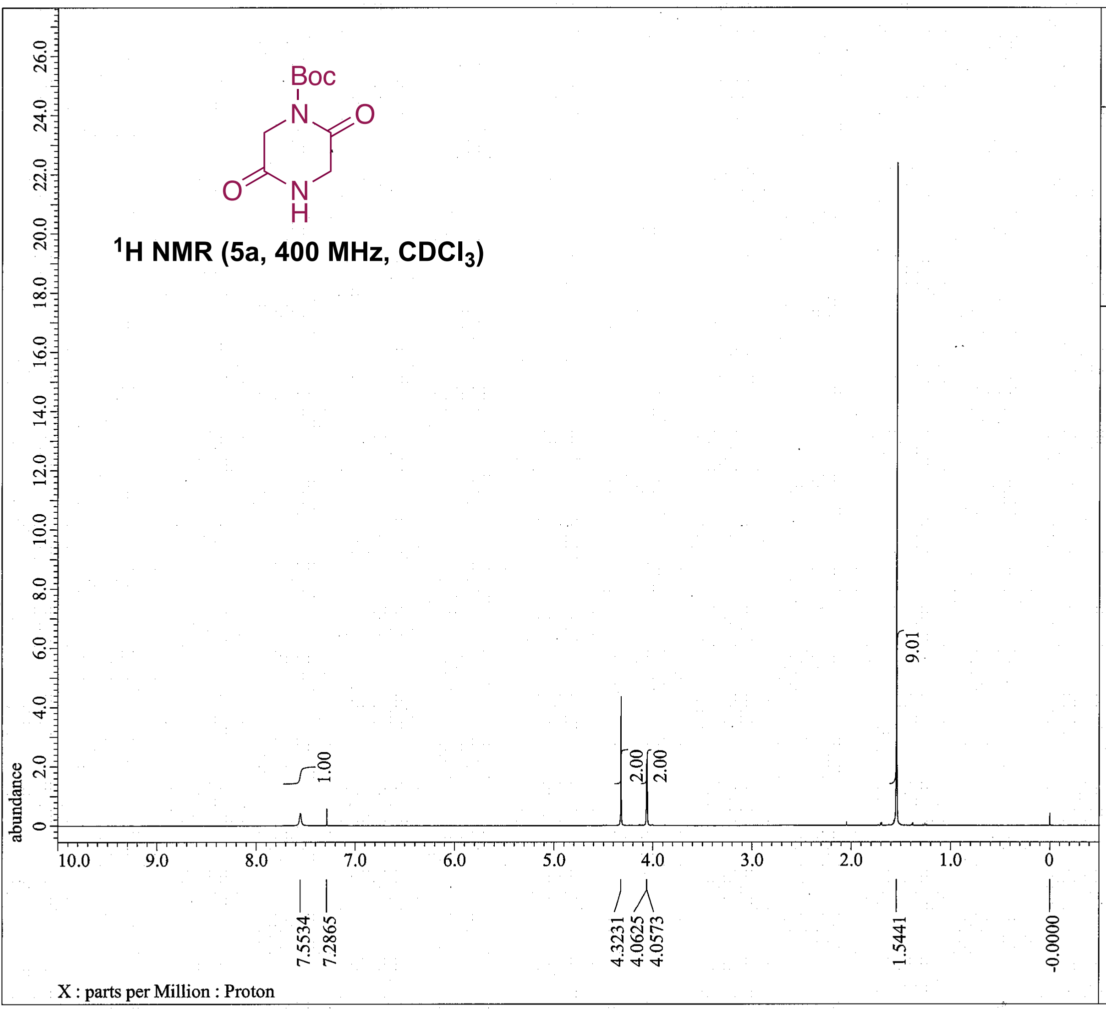
```
Filename          = wm11-204_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm11-204
Solvent           = CHLOROFORM-D
Creation_Time     =  4-JUN-2021 08:35:52
Revision_Time     =  4-JUN-2021 08:42:14
Current_Time      =  4-JUN-2021 08:42:43

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 22.8[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (4m, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-204-13C_E13C-1-1.jdf
```

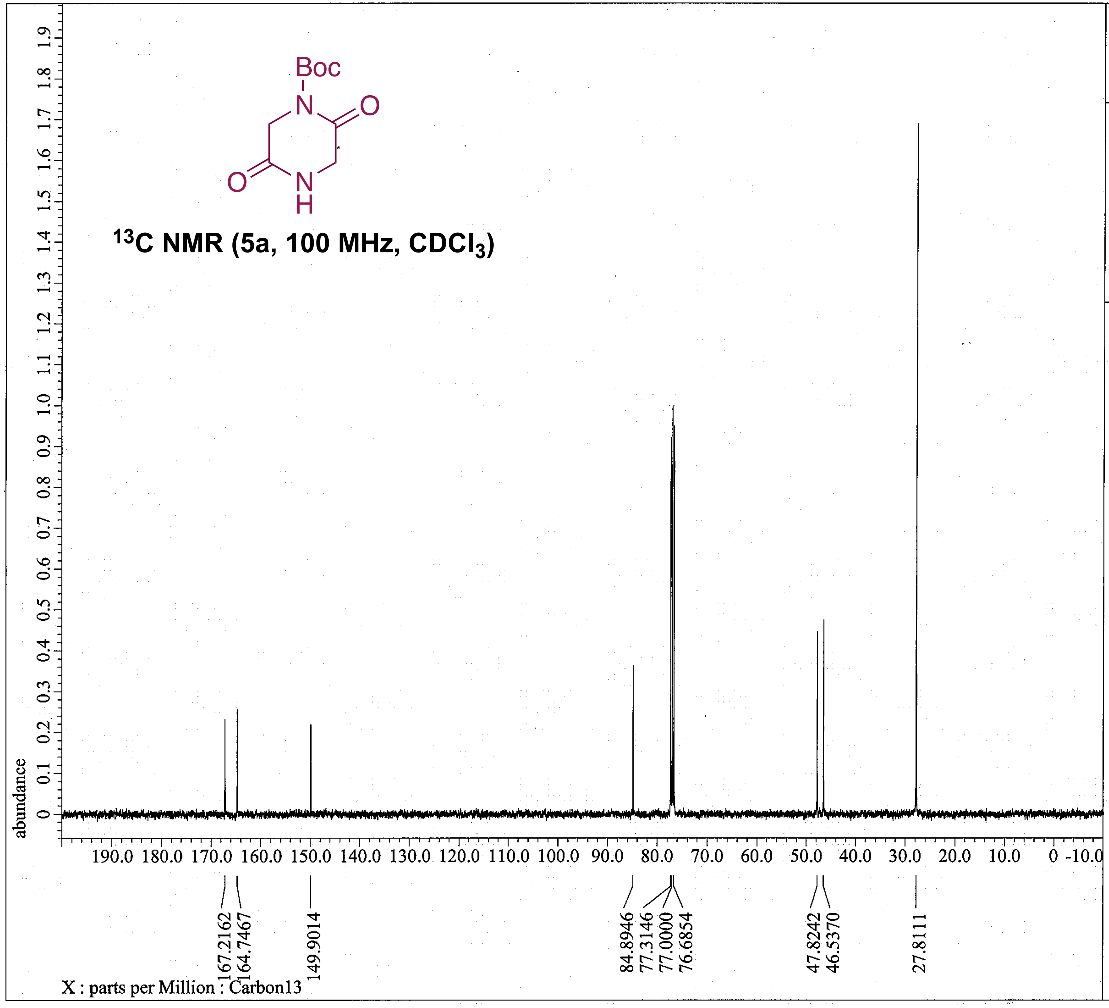
```
Filename          = wm11-204-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm11-204-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  4-JUN-2021 08:40:06
Revision_Time     =  4-JUN-2021 09:28:44
Current_Time      =  4-JUN-2021 09:28:57

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (5a, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-143-1-1_E1H-1-1.jdf
```

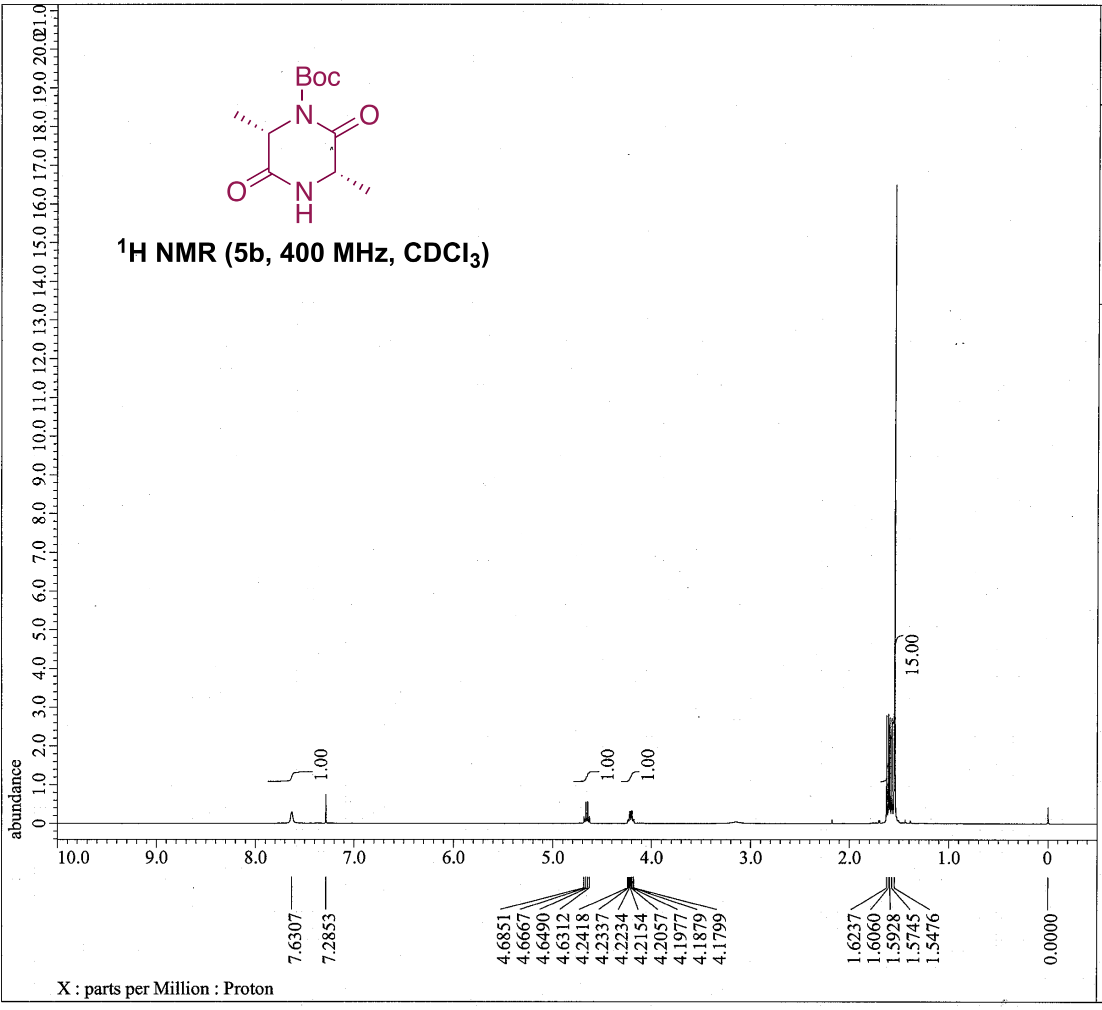
```
Filename           = wm11-143-1-1_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm11-143-1-1
Solvent            = CHLOROFORM-D
Creation_Time      = 25-MAR-2021 10:02:46
Revision_Time      = 25-MAR-2021 10:15:11
Current_Time       = 25-MAR-2021 10:15:23

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 28
Temp_Get           = 23.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (5a, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-143-1-1-13C_E13C-1-1.jdf
```

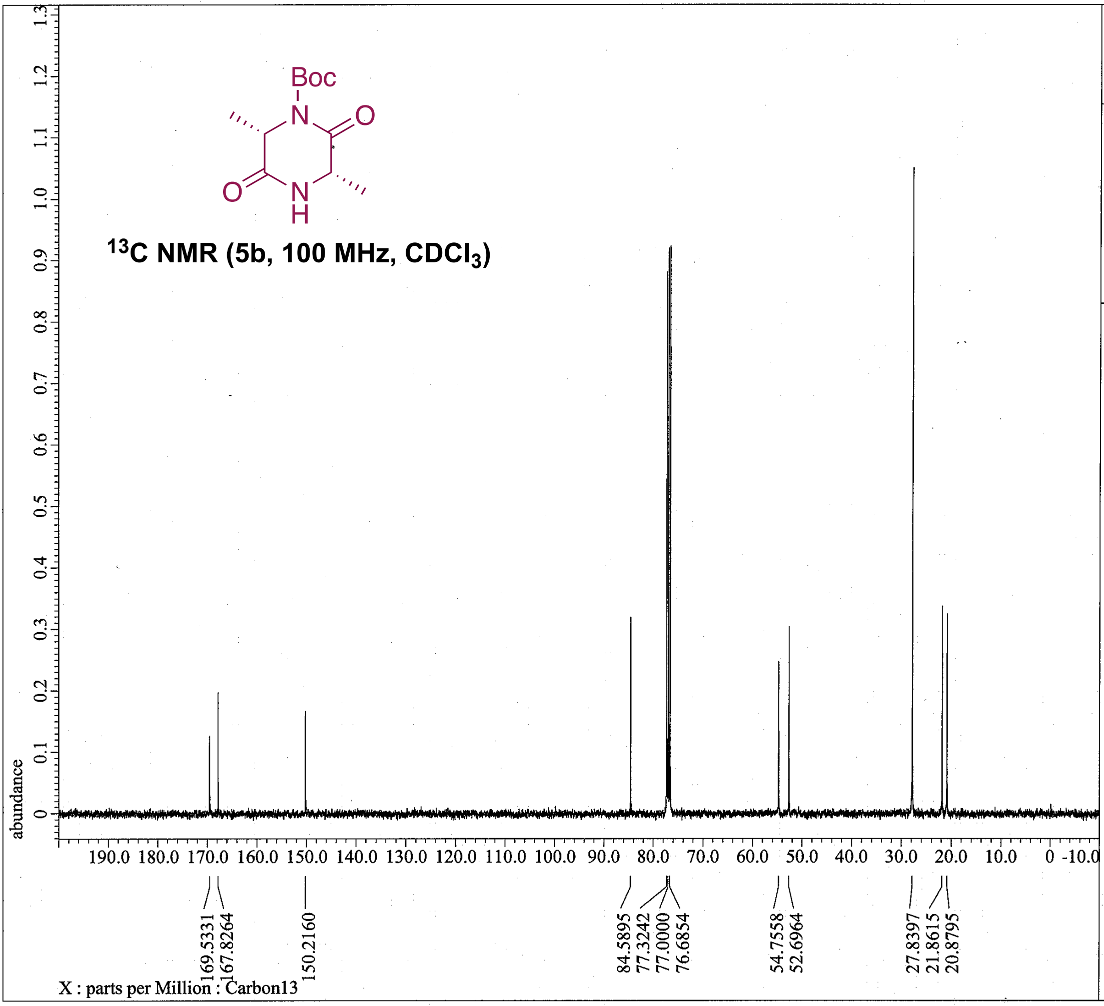
```
Filename           = wm11-143-1-1-13C_E13C-1-3.
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm11-143-1-1-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 25-MAR-2021 10:07:45
Revision_Time      = 25-MAR-2021 10:17:17
Current_Time       = 25-MAR-2021 10:17:45

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 257
Total_Scans        = 257

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.4[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



¹H NMR (5b, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-111_E1H-1-1.jdf
```

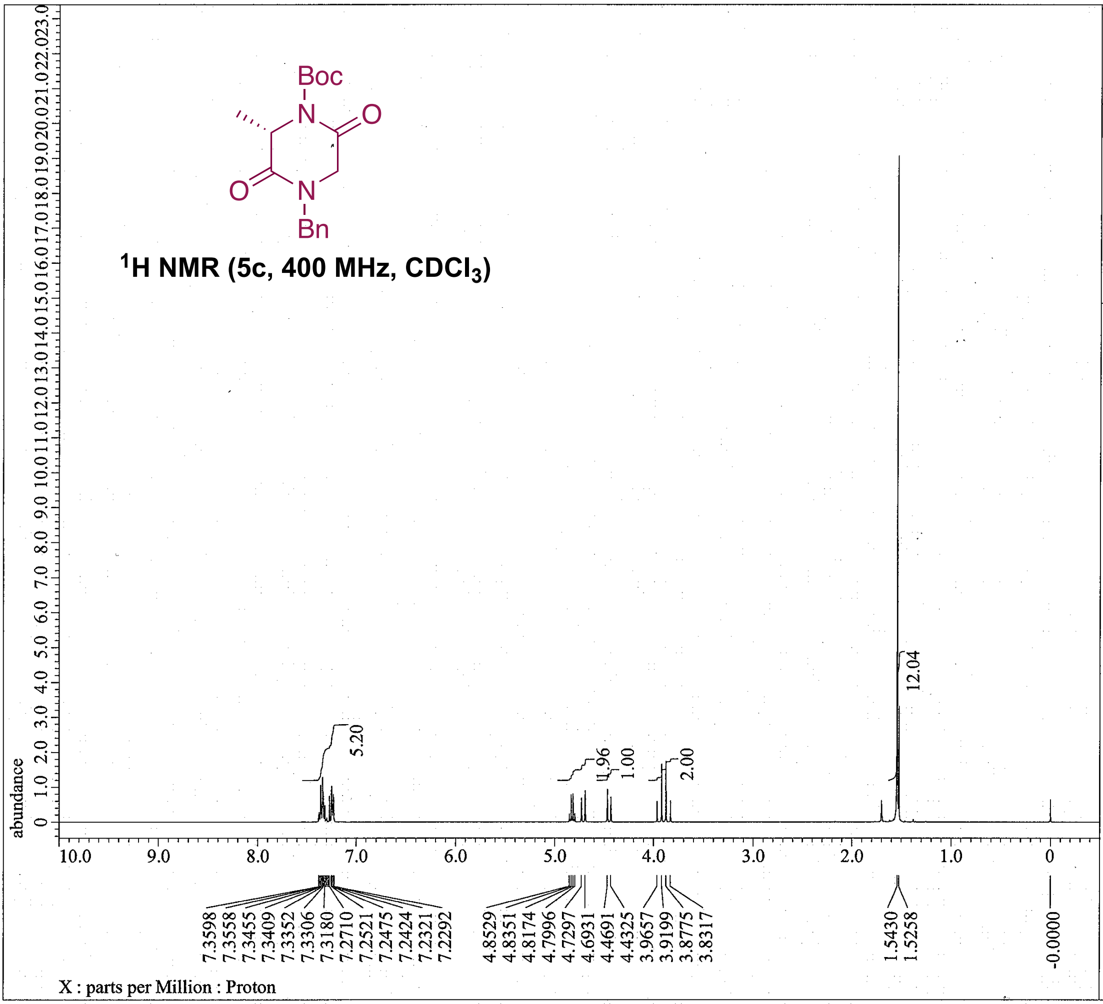
```
Filename           = wm12-111_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-111
Solvent            = CHLOROFORM-D
Creation_Time      =  1-OCT-2021 10:17:50
Revision_Time      =  1-OCT-2021 10:18:24
Current_Time       =  1-OCT-2021 10:18:40

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 28
Temp_Get           = 24.1[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (5b, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-111-13C_E13C-1-1.jdf
```

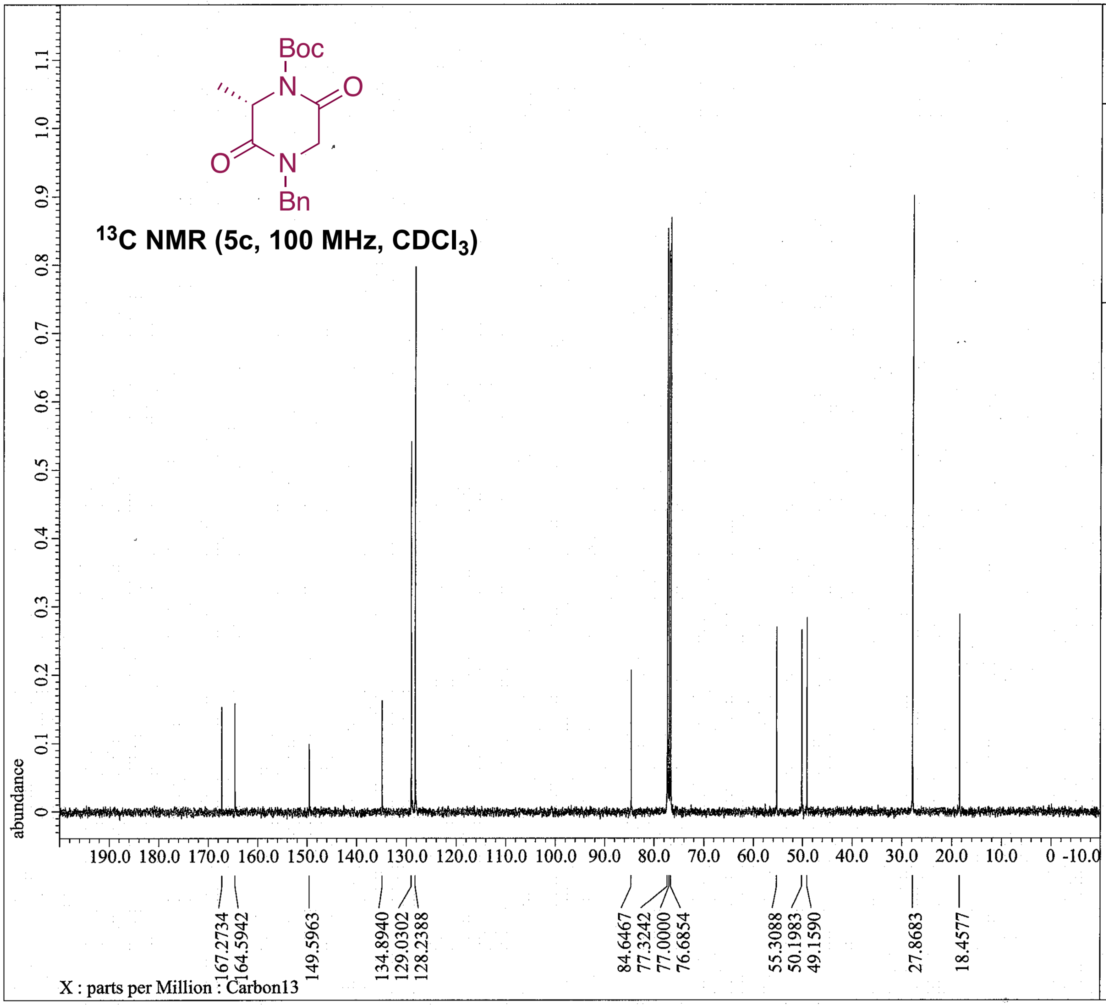
```
Filename           = wm12-111-13C_E13C-1-3.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-111-13C
Solvent            = CHLOROFORM-D
Creation_Time      =  1-OCT-2021 10:21:15
Revision_Time      =  1-OCT-2021 10:52:38
Current_Time       =  1-OCT-2021 10:52:54

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 512
Total_Scans        = 512

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 24[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (5c, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-78_E1H-1-1.jdf
```

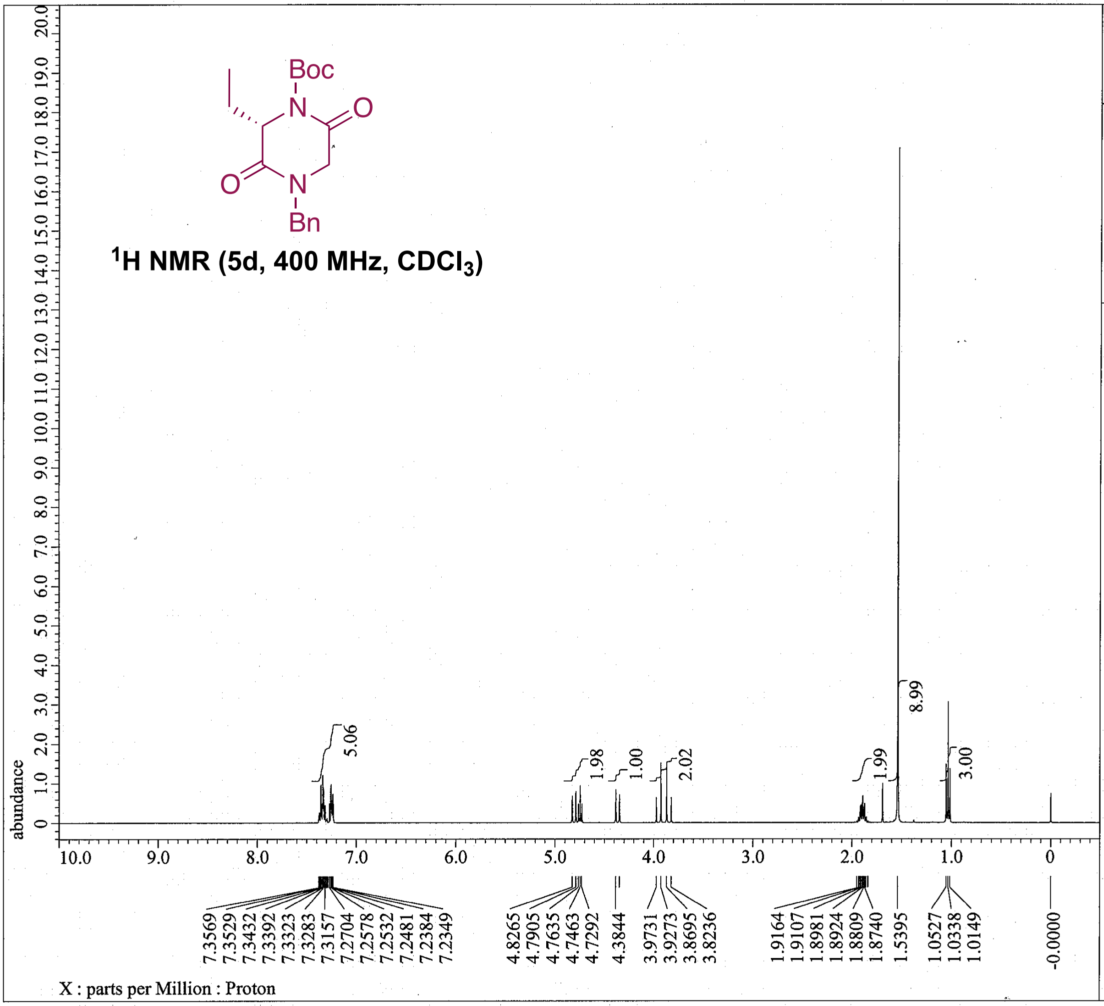
```
Filename            = wm12-78_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-78
Solvent             = CHLOROFORM-D
Creation_Time       = 20-AUG-2021 11:30:54
Revision_Time       = 20-AUG-2021 11:30:30
Current_Time        = 20-AUG-2021 11:30:41

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
Temp_Get            = 23.9[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (5c, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-78-13C_E13C-1-1.jdf
```

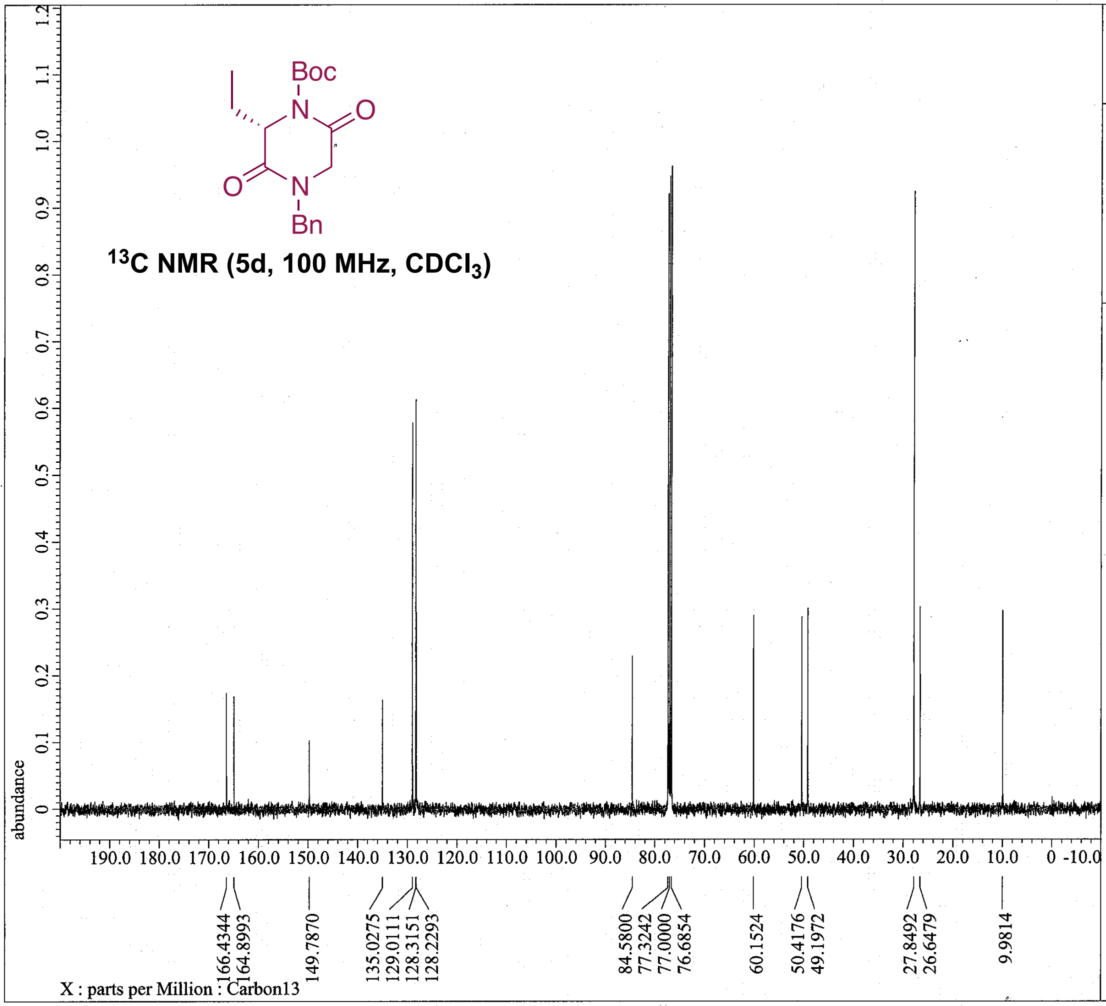
```
Filename            = wm12-78-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-78-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 20-AUG-2021 11:33:02
Revision_Time       = 20-AUG-2021 11:55:08
Current_Time        = 20-AUG-2021 11:55:29

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 513
Total_Scans         = 513

Relaxation_Delay = 2[s]
Recvr_Gain          = 60
Temp_Get            = 23.8[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (5d, 400 MHz, CDCl$_3$)

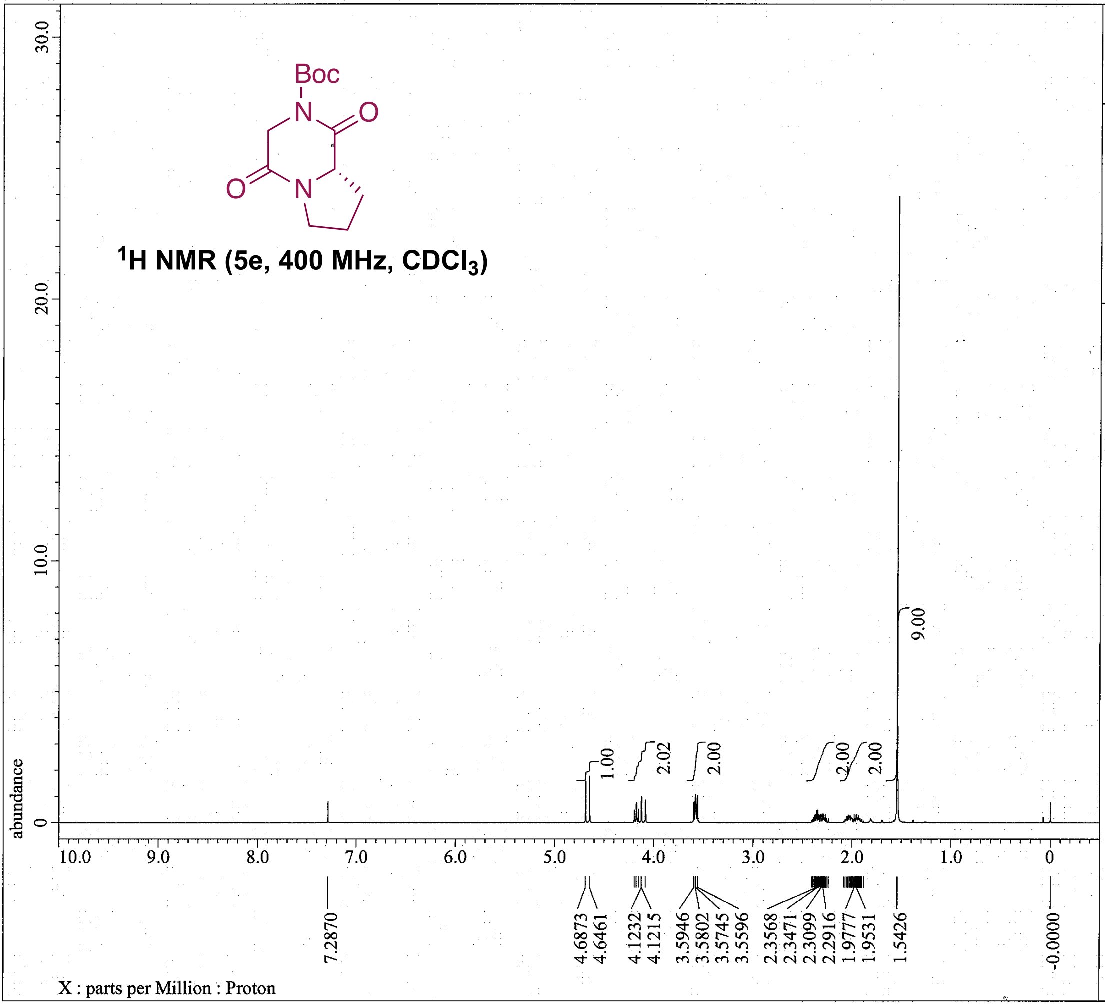
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-28_E1H-2-1.jdf
```

```
Filename            = wm12-28_E1H-2-5.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-28
Solvent             = CHLOROFORM-D
Creation_Time       = 28-JUN-2021 14:25:38
Revision_Time       = 28-JUN-2021 14:25:31
Current_Time        = 28-JUN-2021 14:25:43

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
Temp_Get            = 22.8[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



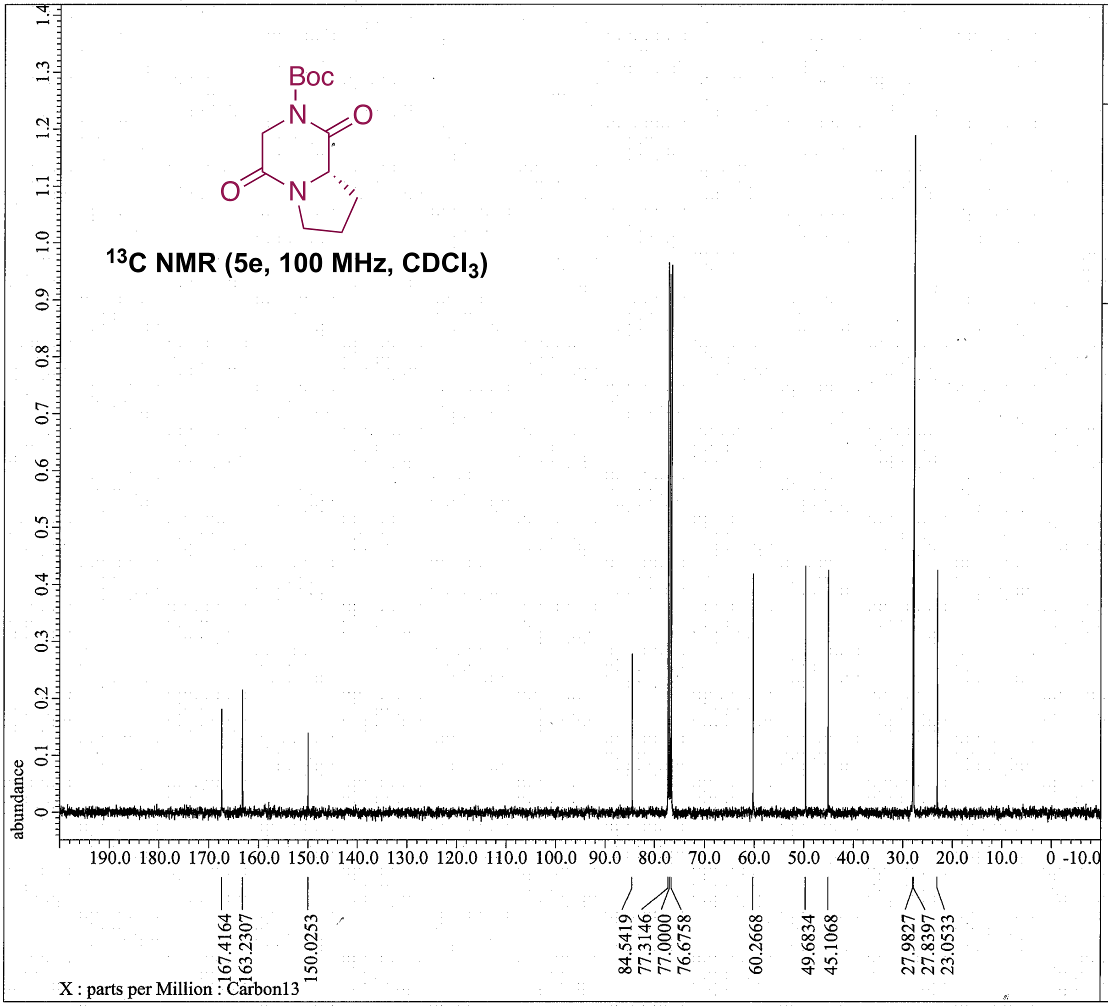
$^{13}$C NMR (5d, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-28-13C_E13C-1-1.jdf

Filename          = wm12-28-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-28-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 28-JUN-2021 14:27:48
Revision_Time     = 28-JUN-2021 14:37:14
Current_Time      = 28-JUN-2021 14:37:33

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 257
Total_Scans       = 257

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.9[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```


$^1$H NMR (5e, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-8_E1H-1-1.jdf
```

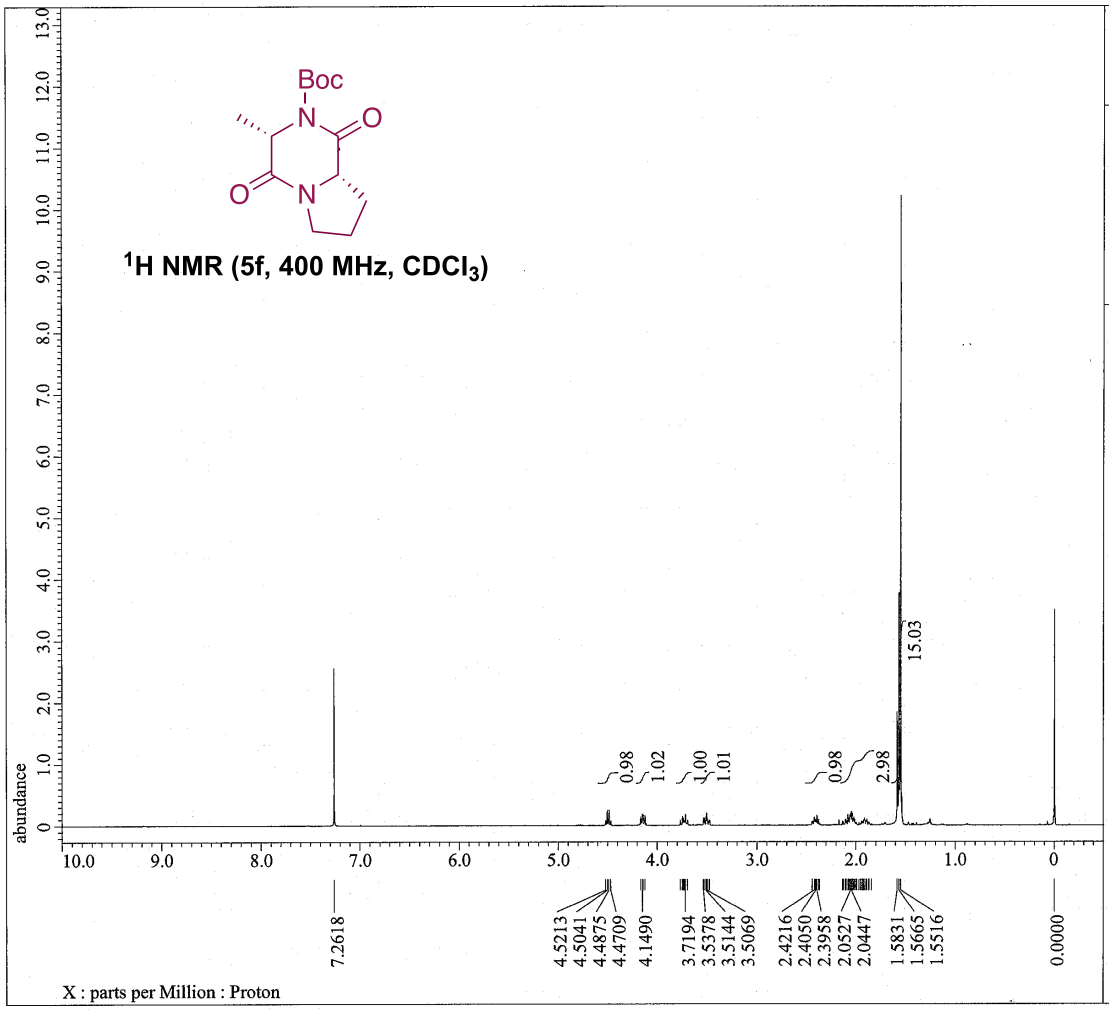
```
Filename          = wm12-8_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-8
Solvent           = CHLOROFORM-D
Creation_Time     =  9-JUN-2021 07:54:57
Revision_Time     =  9-JUN-2021 07:55:46
Current_Time      =  9-JUN-2021 07:56:00

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 22.9[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (5e, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-8-13C_E13C-1-1.jdf
```

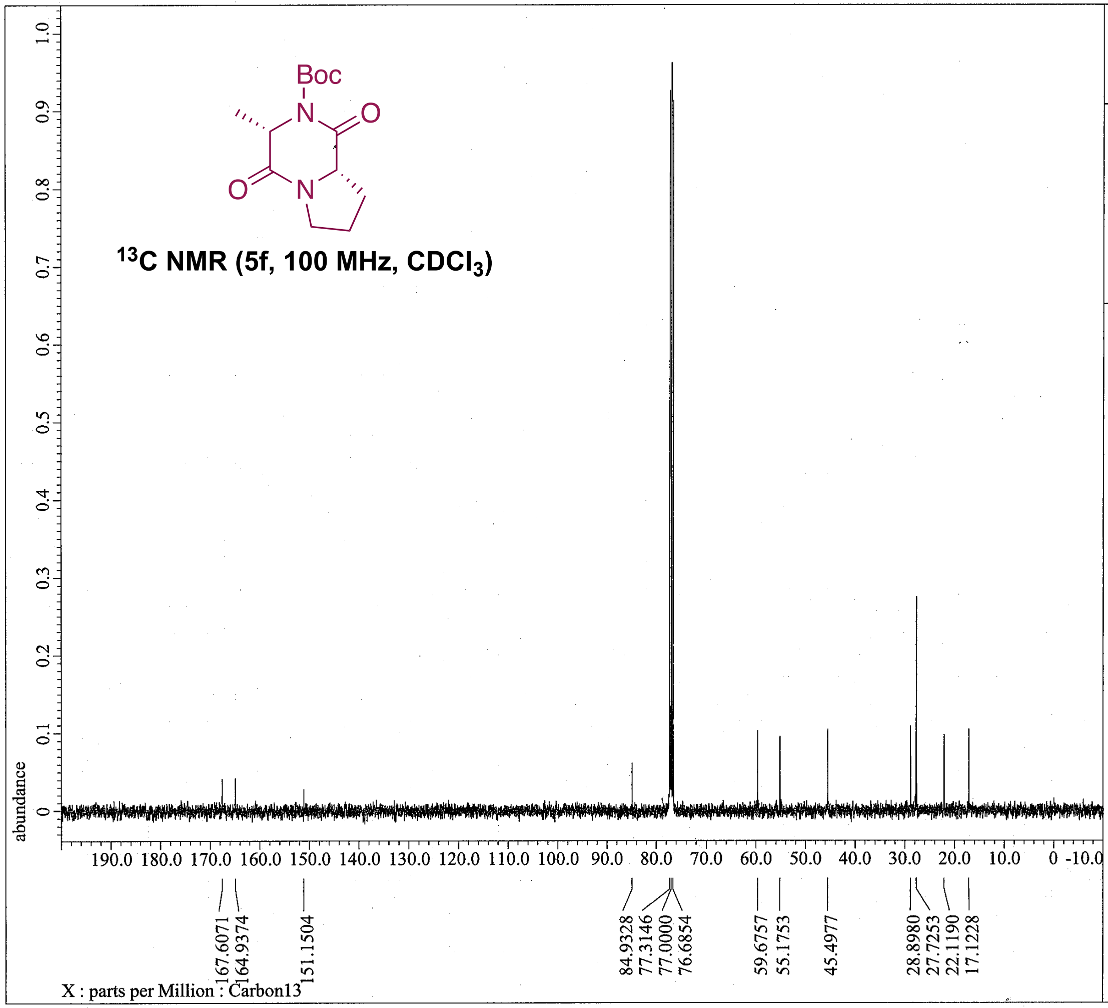
```
Filename           = wm12-8-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-8-13C
Solvent            = CHLOROFORM-D
Creation_Time      =  9-JUN-2021 08:00:01
Revision_Time      =  9-JUN-2021 08:09:44
Current_Time       =  9-JUN-2021 08:10:02

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 257
Total_Scans        = 257

Relaxation_Delay = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.9[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (5f, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-148_E1H-1-1.jdf
```

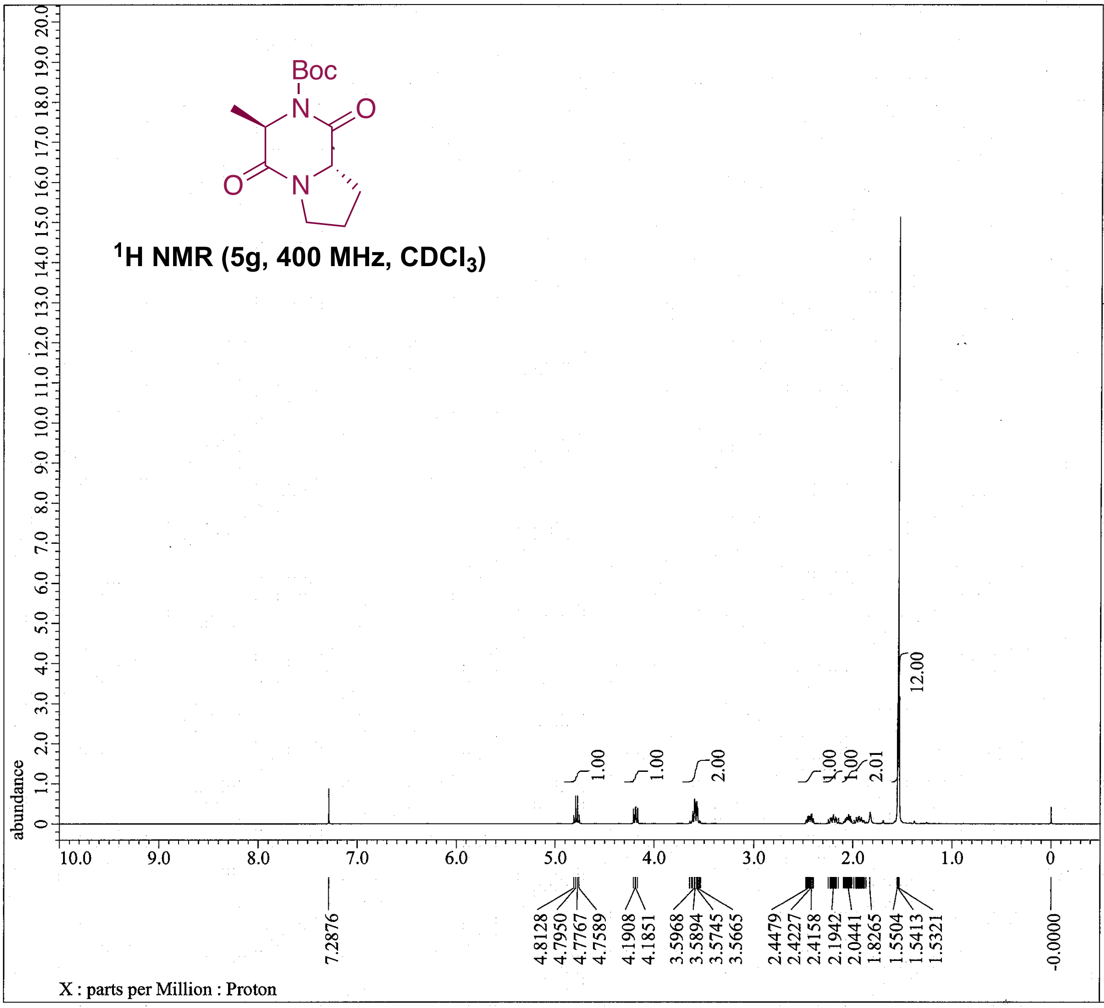
```
Filename          = wm12-148_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-148
Solvent           = CHLOROFORM-D
Creation_Time     =  8-NOV-2021 15:00:50
Revision_Time     =  8-NOV-2021 15:01:34
Current_Time      =  8-NOV-2021 15:01:53

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 24[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (5f, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-148_E13C_copy8-1-1.jdf
```

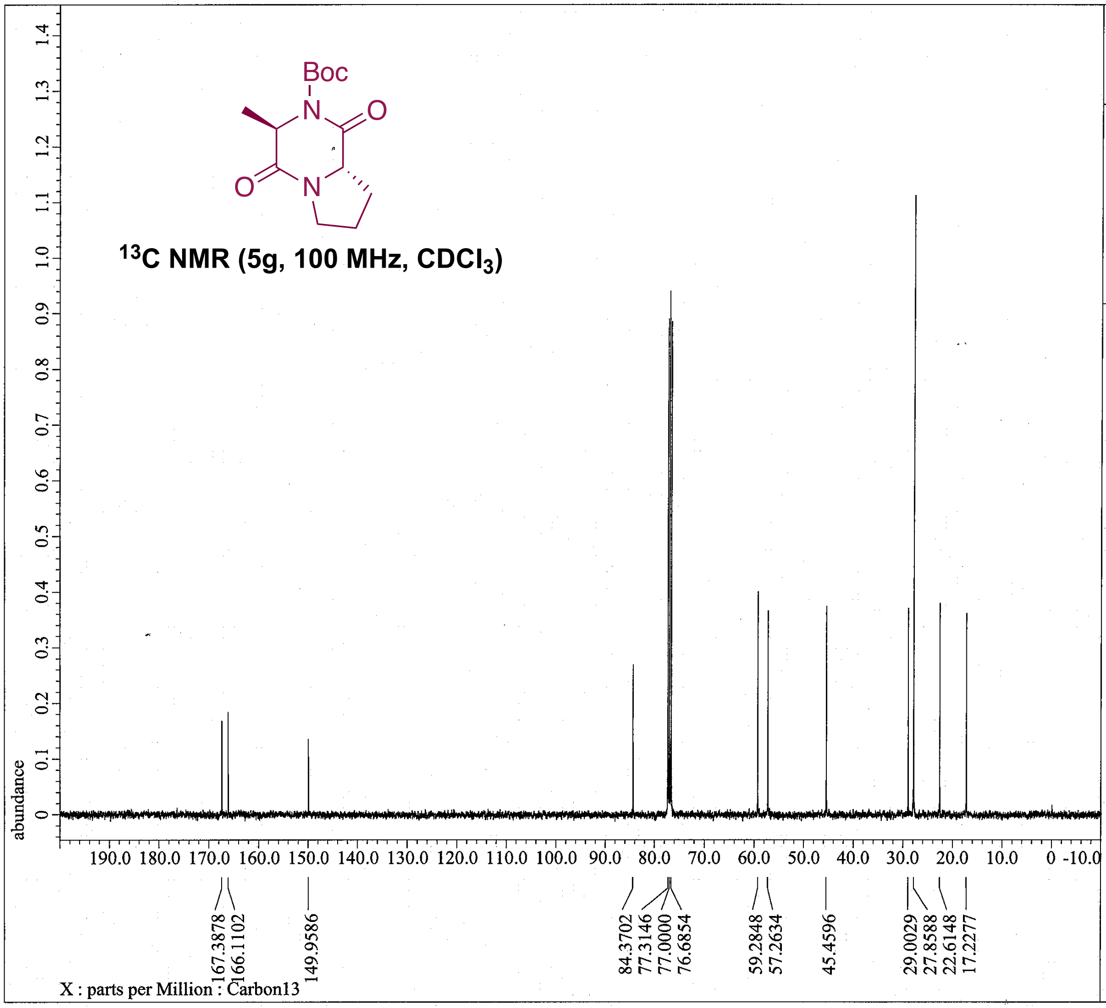
```
Filename           = wm12-148_E13C_copy8-1-3.jd
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-148
Solvent            = CHLOROFORM-D
Creation_Time      =  9-NOV-2021 09:55:41
Revision_Time      =  9-NOV-2021 10:04:23
Current_Time       =  9-NOV-2021 10:04:48

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 0[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Incomplete_Copy    = TRUE
Scans              = 256
Total_Scans        = 256

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.7[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
```



$^1$H NMR (5g, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-107-1-1_E1H-1-1.jdf
```

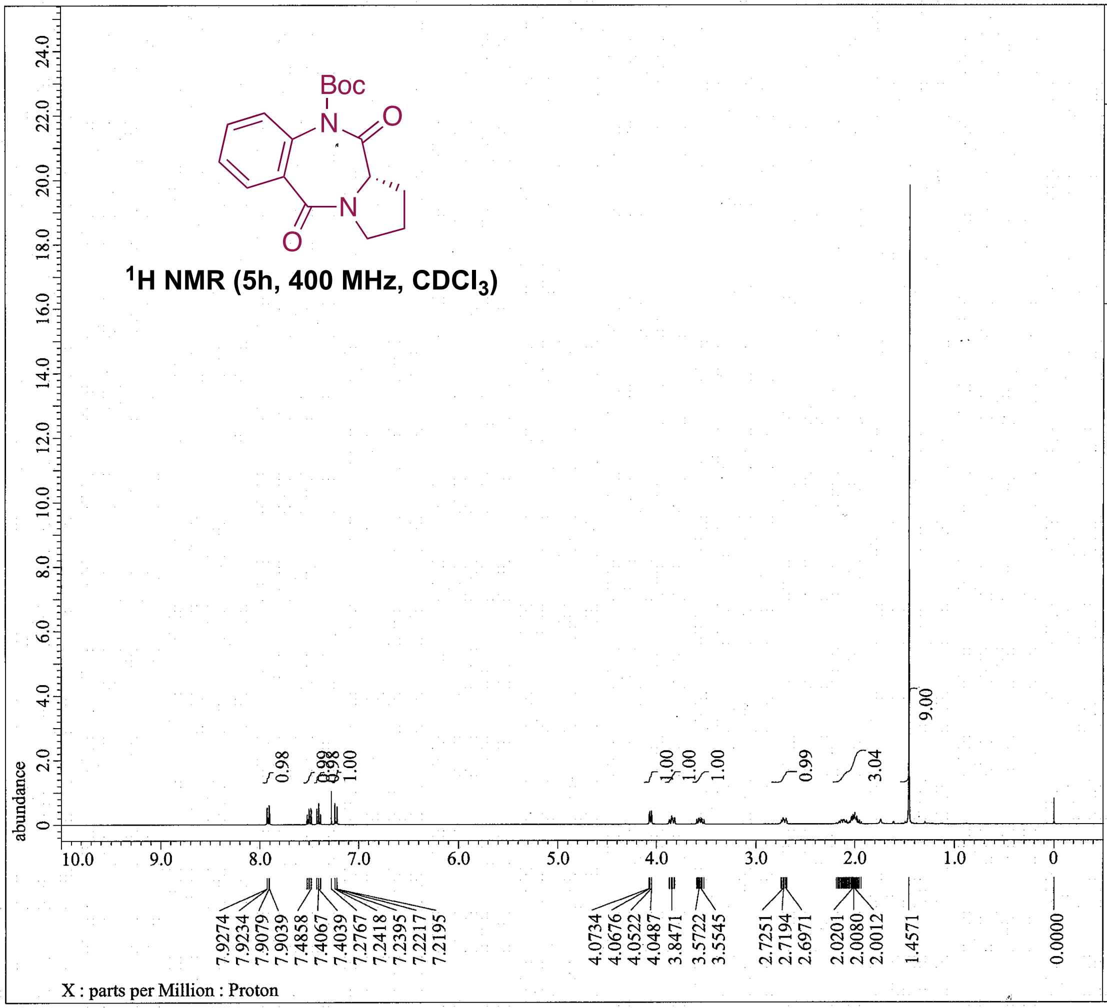
```
Filename           = wm12-107-1-1_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-107-1-1
Solvent            = CHLOROFORM-D
Creation_Time      = 29-SEP-2021 08:09:12
Revision_Time      = 29-SEP-2021 08:10:57
Current_Time       = 29-SEP-2021 08:11:14

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 28
Temp_Get           = 23.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (5g, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-107-1-1-13C_E13C-1-1.jdf
```

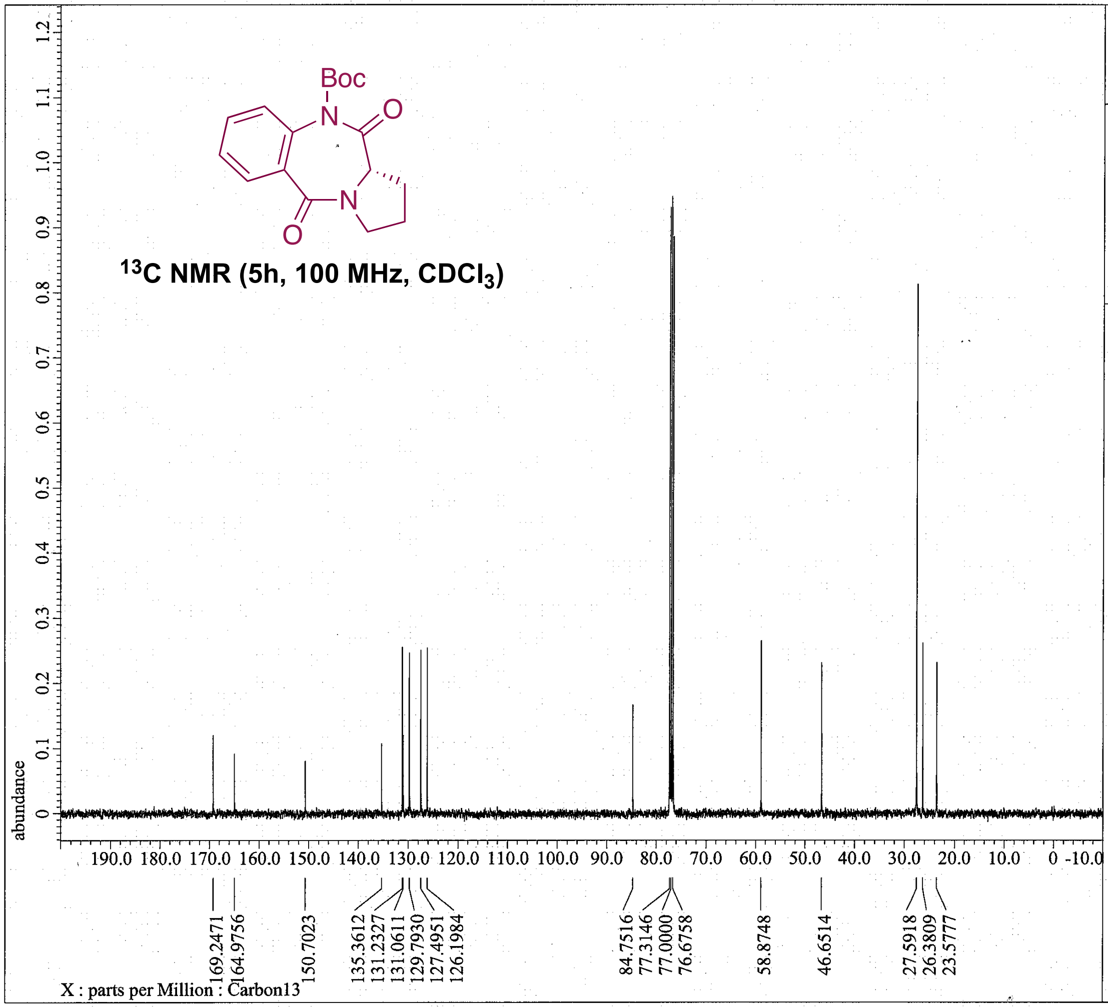
```
Filename          = wm12-107-1-1-13C_E13C-1-4.
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-107-1-1-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 29-SEP-2021 08:11:22
Revision_Time     = 29-SEP-2021 08:33:52
Current_Time      = 29-SEP-2021 08:34:14

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.5[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (5h, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-153_E1H-1-1.jdf
```

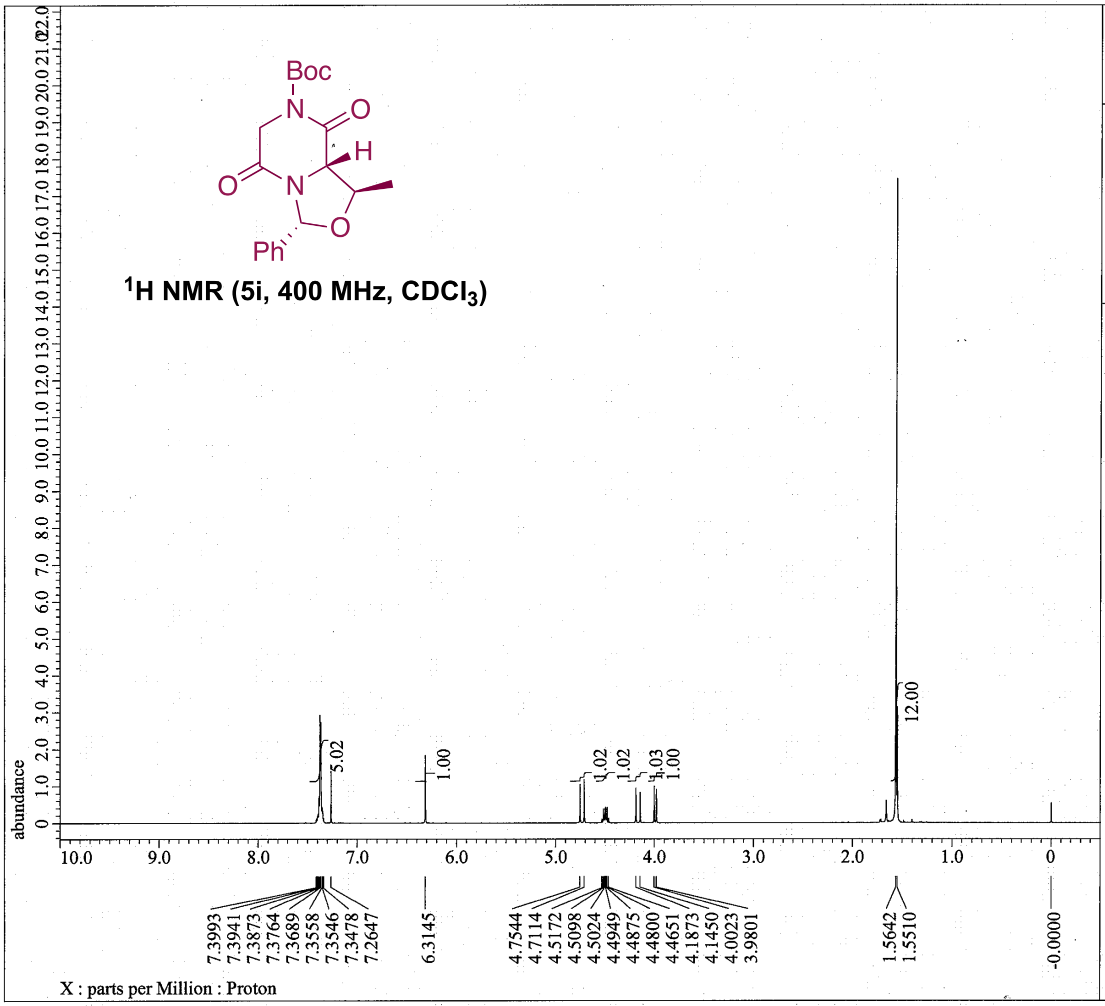
```
Filename           = wm12-153_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-153
Solvent            = CHLOROFORM-D
Creation_Time      = 15-NOV-2021 15:01:08
Revision_Time      = 15-NOV-2021 15:02:18
Current_Time       = 15-NOV-2021 15:02:31

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 30
Temp_Get           = 23.1[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (5h, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-153-13C_E13C-1-1.jdf
```

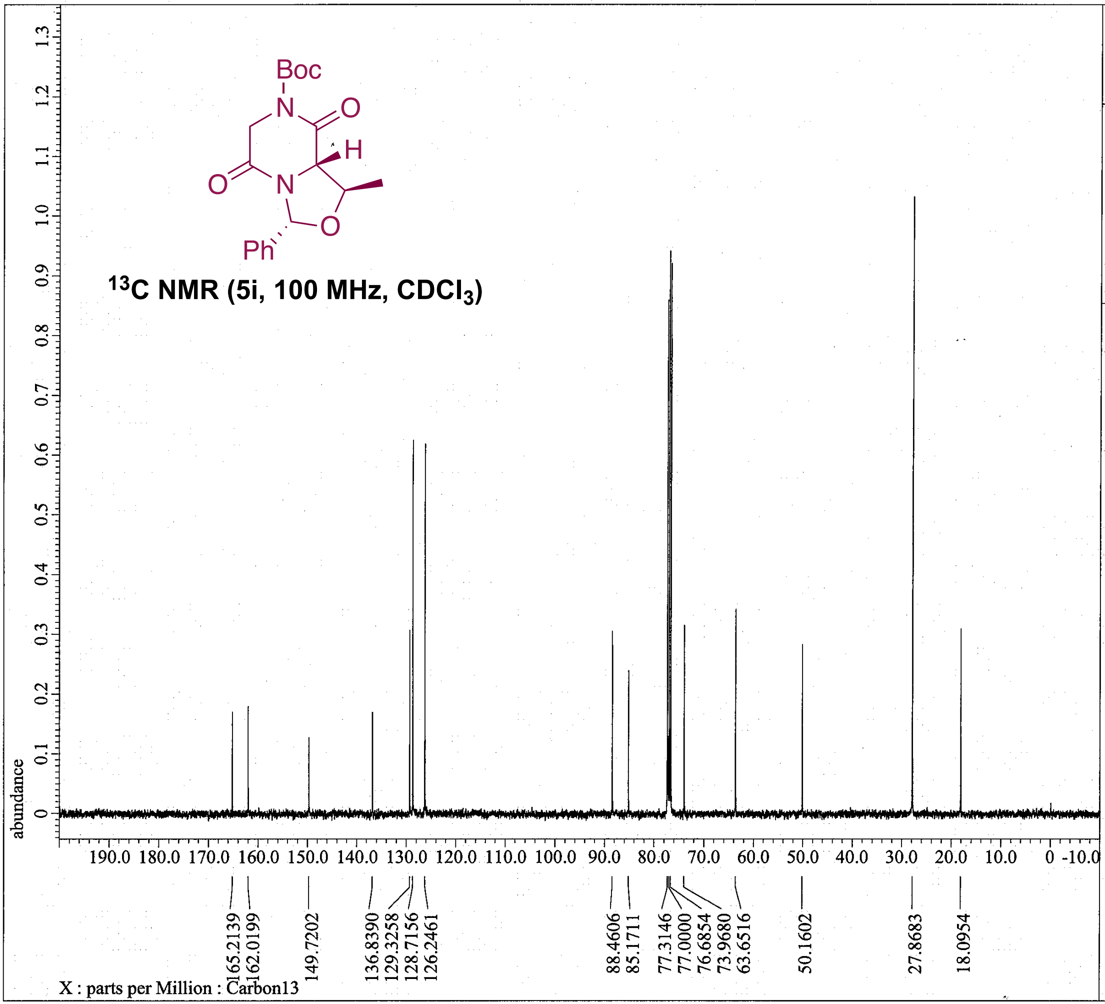
```
Filename          = wm12-153-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-153-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 15-NOV-2021 15:03:21
Revision_Time     = 15-NOV-2021 15:25:24
Current_Time      = 15-NOV-2021 15:25:45

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.3[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



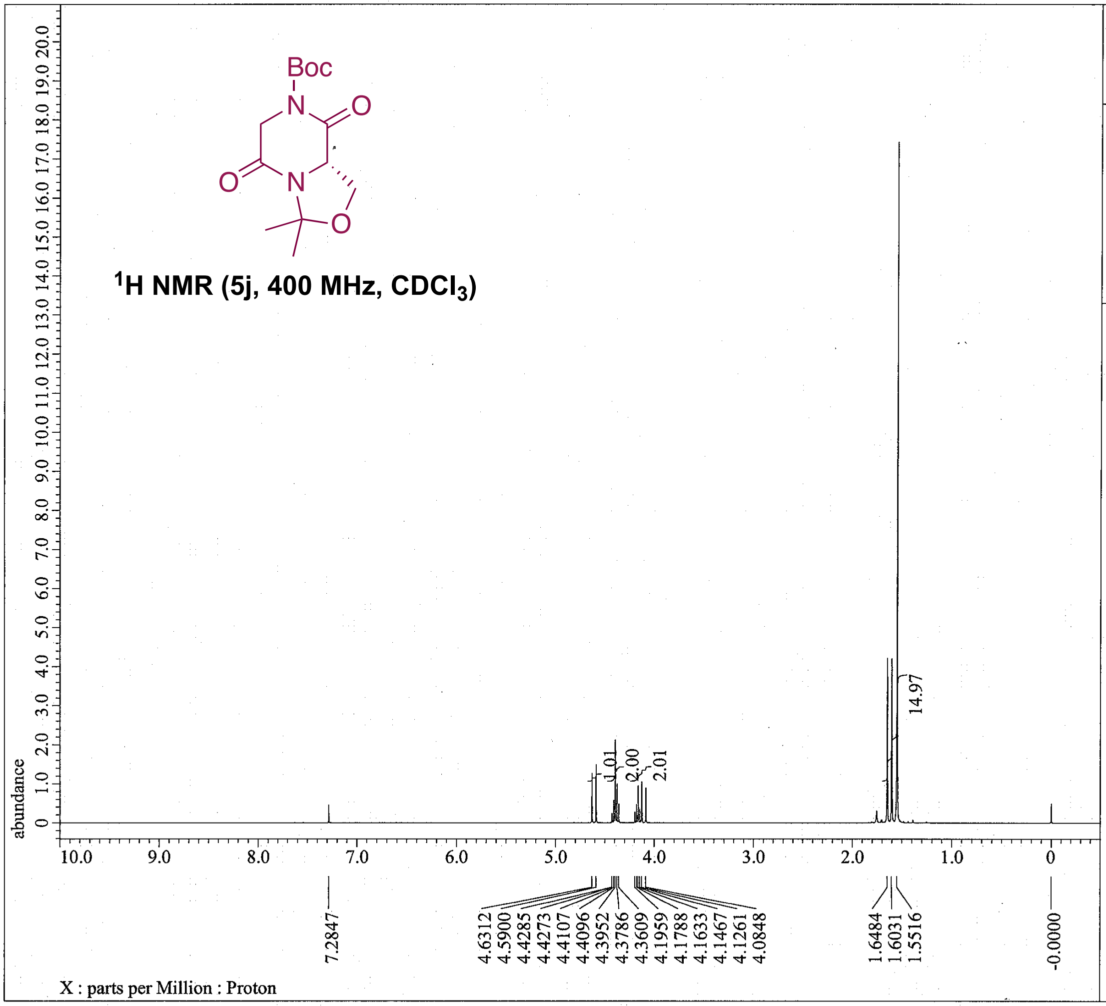
$^1$H NMR (5i, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-84_E1H-1-1.jdf
```

```
Filename          = wm12-84_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-84
Solvent           = CHLOROFORM-D
Creation_Time     =  2-SEP-2021 10:47:19
Revision_Time     =  2-SEP-2021 10:47:39
Current_Time      =  2-SEP-2021 10:47:50

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.7[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```

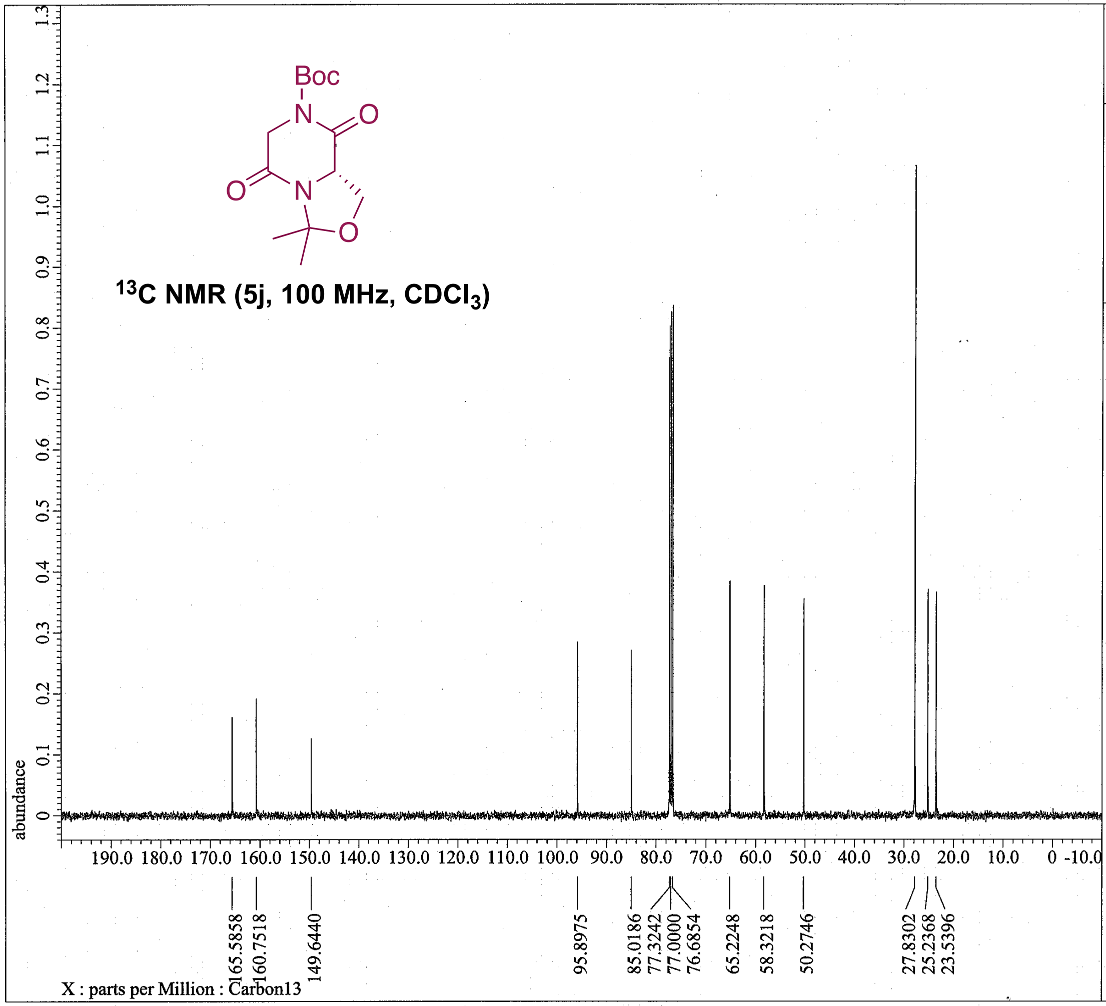


```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-84-13C_E13C-1-1.jdf

Filename            = wm12-84-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-84-13C
Solvent             = CHLOROFORM-D
Creation_Time       =  2-SEP-2021 10:49:30
Revision_Time       =  2-SEP-2021 11:11:32
Current_Time        =  2-SEP-2021 11:11:52

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = TRUE
Scans               = 513
Total_Scans         = 513

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 24[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```


$^1$H NMR (5j, 400 MHz, CDCl$_3$)

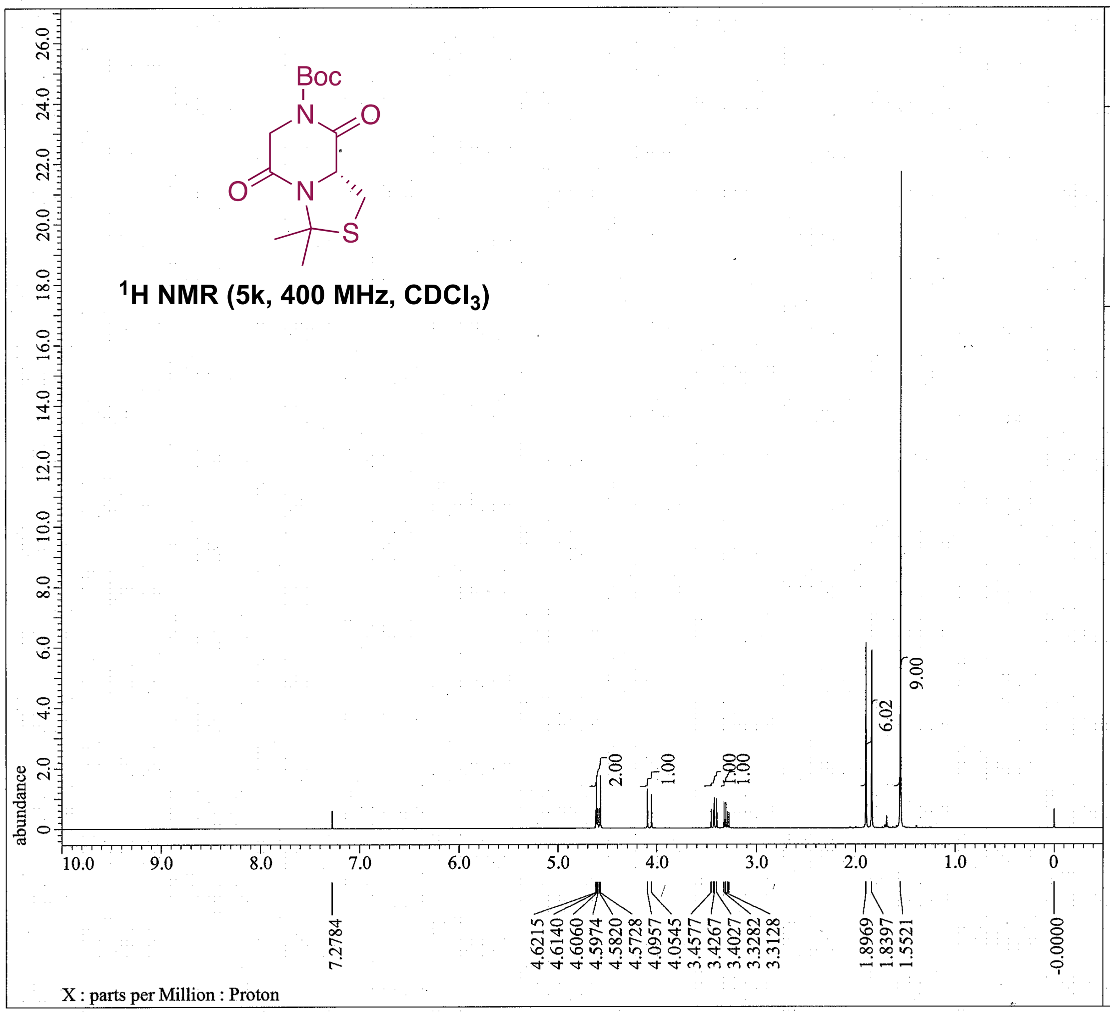
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-102_E1H-1-1.jdf
```

```
Filename          = wm12-102_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-102
Solvent           = CHLOROFORM-D
Creation_Time     = 24-SEP-2021 08:20:47
Revision_Time     = 24-SEP-2021 08:22:21
Current_Time      = 24-SEP-2021 08:22:34

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 23.3[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (5j, 100 MHz, CDCl$_3$)

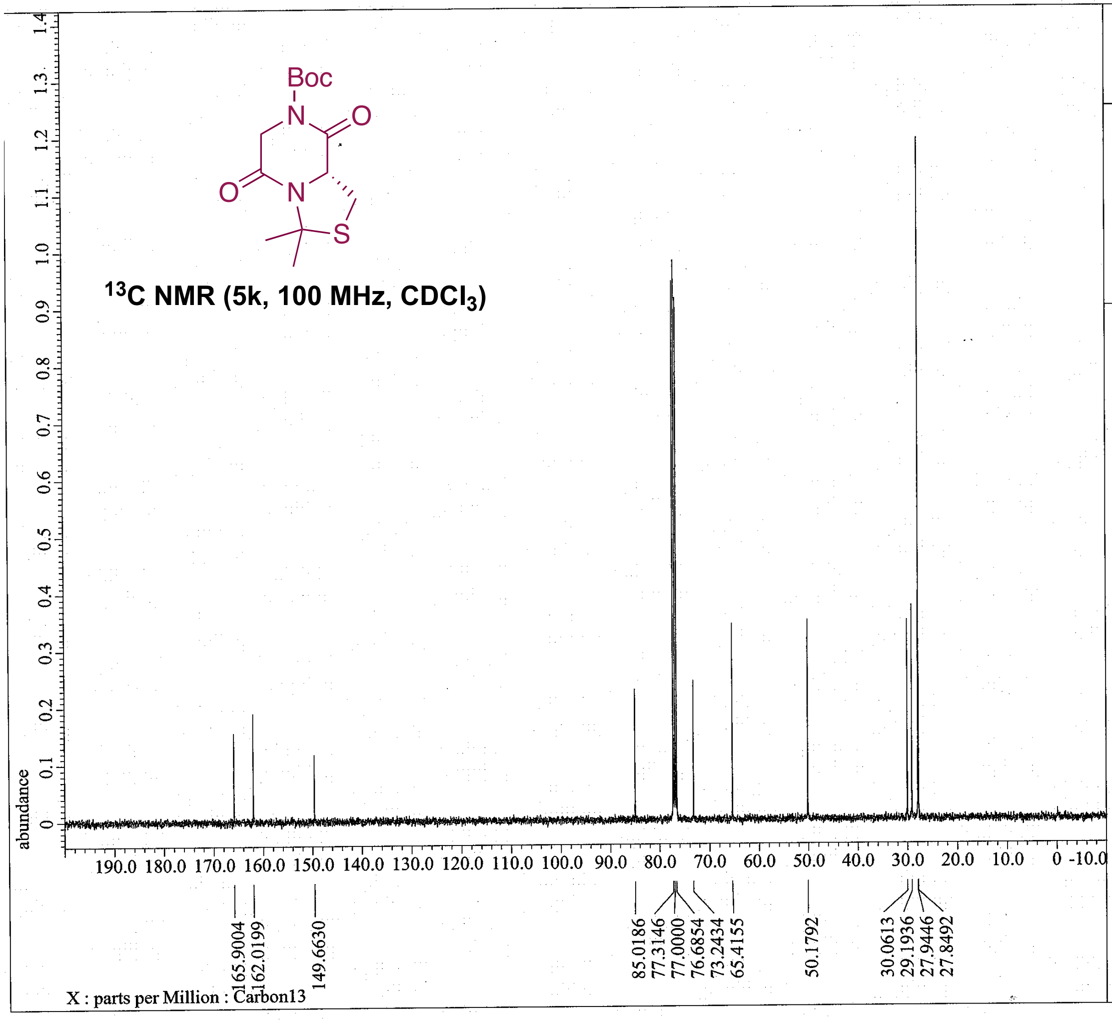
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-102-13C_E13C-1-1.jdf

Filename           = wm12-102-13C_E13C-1-3.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-102-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 24-SEP-2021 08:22:56
Revision_Time      = 24-SEP-2021 08:44:27
Current_Time       = 24-SEP-2021 08:44:44

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 512
Total_Scans        = 512

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.5[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (5k, 400 MHz, $CDCl_3$)

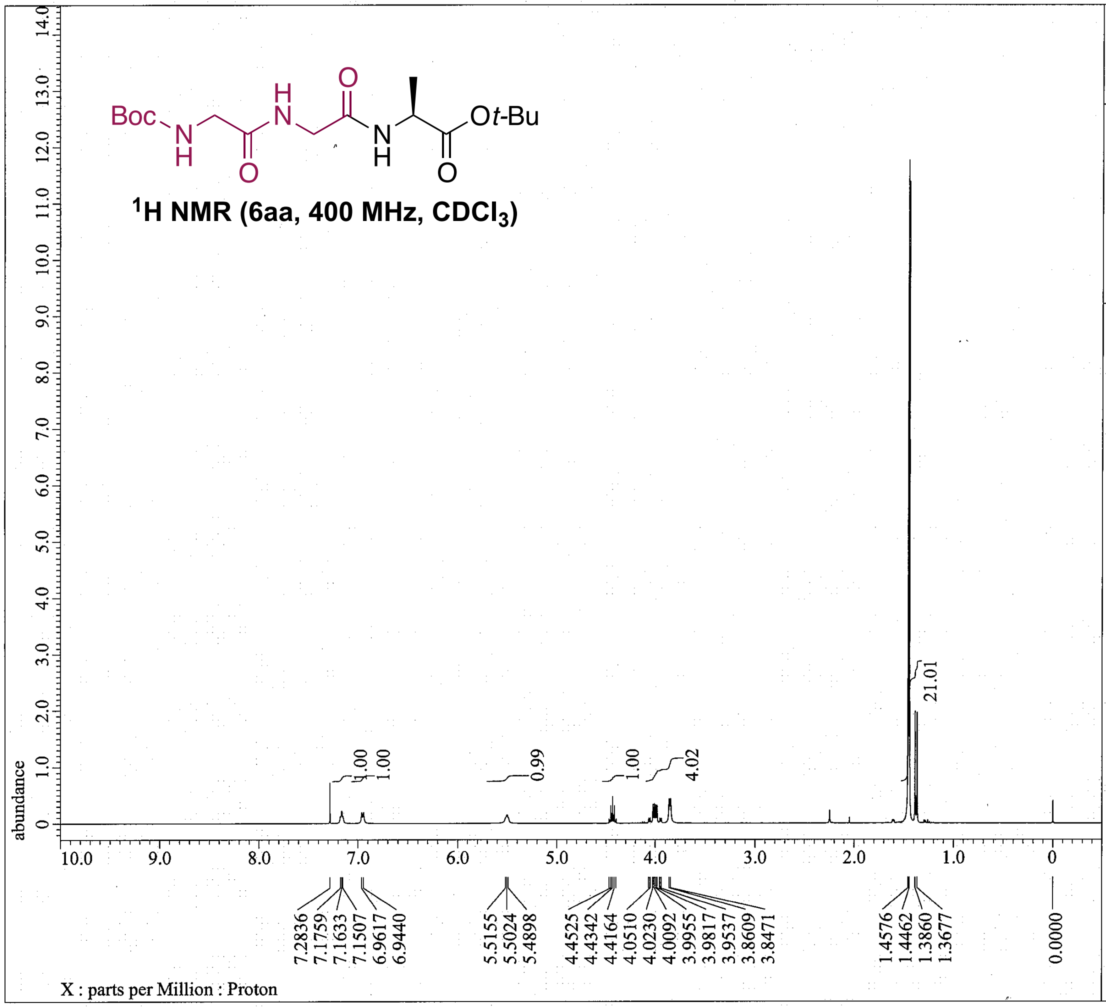
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-162_E1H-1-1.jdf
```

```
Filename           = wm12-162_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-162
Solvent            = CHLOROFORM-D
Creation_Time      = 29-NOV-2021 13:31:19
Revision_Time      = 29-NOV-2021 13:30:53
Current_Time       = 29-NOV-2021 13:31:11

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 30
Temp_Get           = 24.2[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (5k, 100 MHz, CDCl$_3$)

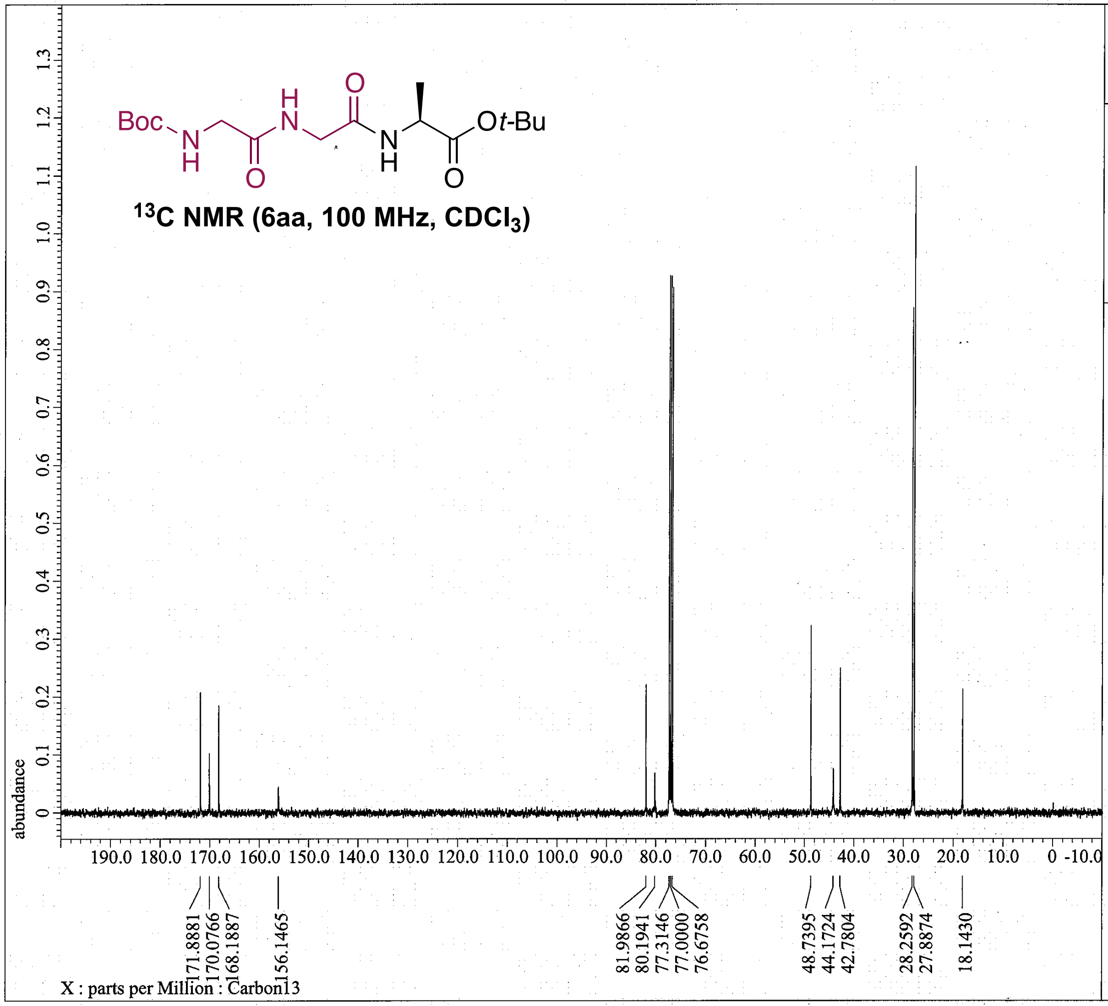
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-162-13C_E13C-1-1.jdf

Filename          = wm12-162-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-162-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 29-NOV-2021 13:33:29
Revision_Time     = 29-NOV-2021 13:55:19
Current_Time      = 29-NOV-2021 13:55:42

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 513
Total_Scans       = 513

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.6[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6aa, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-148_E1H-1-1.jdf
```

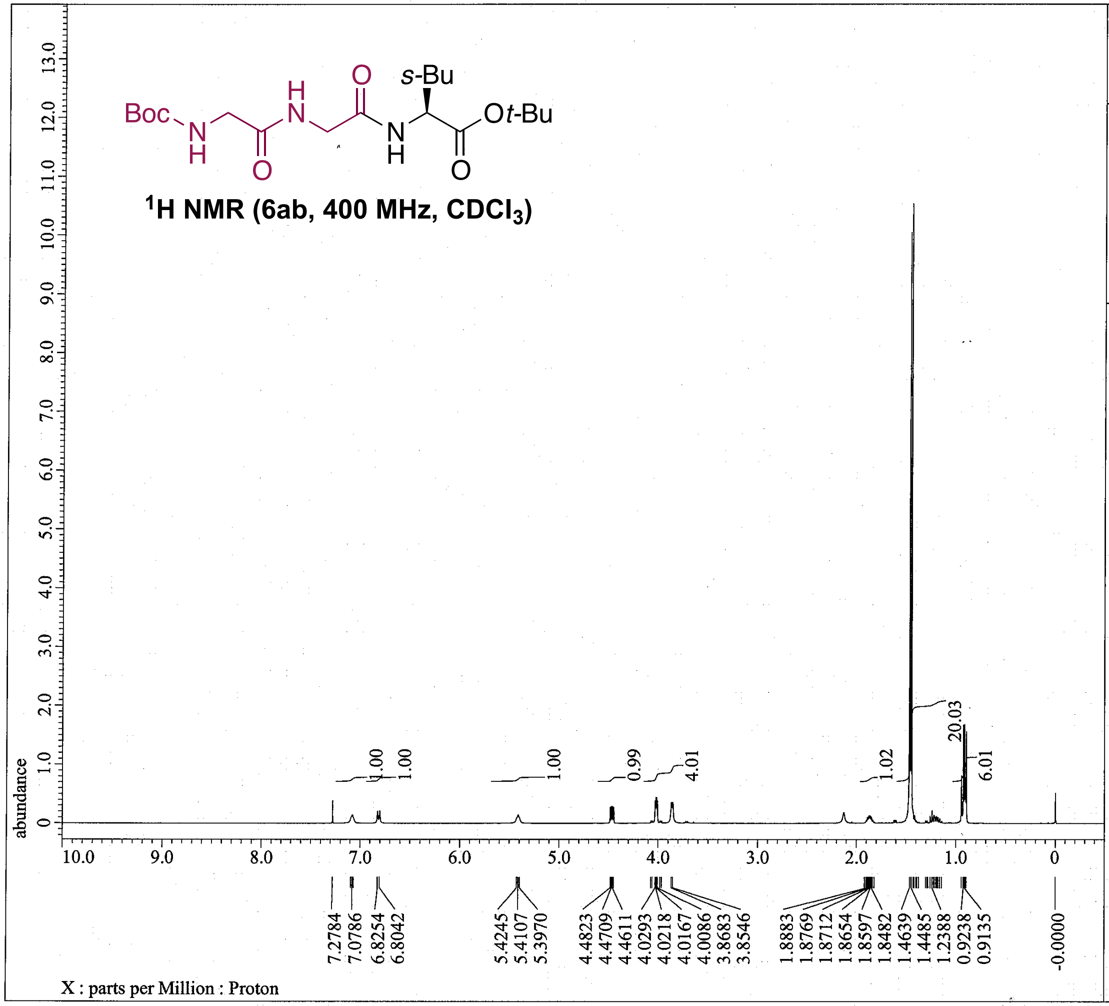
```
Filename           = wm11-148_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm11-148
Solvent            = CHLOROFORM-D
Creation_Time      = 29-MAR-2021 14:47:00
Revision_Time      = 29-MAR-2021 14:49:45
Current_Time       = 29-MAR-2021 14:49:57

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 24.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (6aa, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-148-13C_E13C-1-1.jdf
```

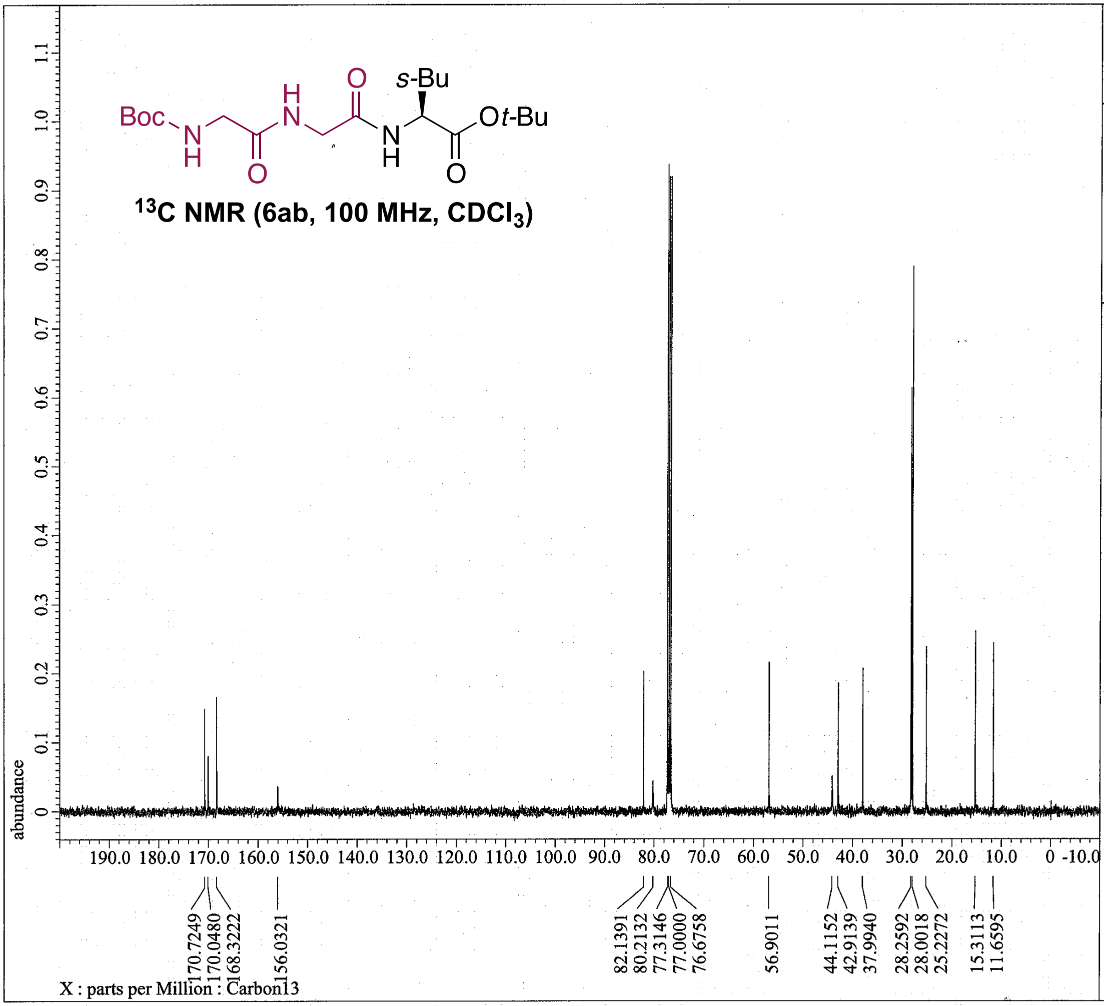
```
Filename             = wm11-148-13C_E13C-1-4.jdf
Author               = delta
Experiment           = single_pulse_dec.jxp
Sample_Id            = wm11-148-13C
Solvent              = CHLOROFORM-D
Creation_Time        = 29-MAR-2021 14:51:09
Revision_Time        = 29-MAR-2021 15:13:38
Current_Time         = 29-MAR-2021 15:14:06

Data_Format          = 1D COMPLEX
Dim_Size             = 26214
Dim_Title            = Carbon13
Dim_Units            = [ppm]
Dimensions           = X
Site                 = JNM-ECS400
Spectrometer         = DELTA2_NMR

Field_Strength       = 9.389766[T] (400[MHz])
X_Acq_Duration       = 1.04333312[s]
X_Domain             = 13C
X_Freq               = 100.52530333[MHz]
X_Offset             = 100[ppm]
X_Points             = 32768
X_Prescans           = 4
X_Resolution         = 0.95846665[Hz]
X_Sweep              = 31.40703518[kHz]
X_Sweep_Clipped      = 25.12562814[kHz]
Irr_Domain           = Proton
Irr_Freq             = 399.78219838[MHz]
Irr_Offset           = 5[ppm]
Clipped              = FALSE
Scans                = 513
Total_Scans          = 513

Relaxation_Delay     = 2[s]
Recvr_Gain           = 60
Temp_Get             = 23.9[dC]
X_90_Width           = 10.1[us]
X_Acq_Time           = 1.04333312[s]
X_Angle              = 30[deg]
X_Atn                = 4.6[dB]
X_Pulse              = 3.36666667[us]
Irr_Atn_Dec          = 25.996[dB]
Irr_Atn_Noe          = 25.996[dB]
Irr_Noise            = WALTZ
Irr_Pwidth           = 0.115[ms]
Decoupling           = TRUE
Initial_Wait         = 1[s]
```



$^1$H NMR (6ab, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-46_E1H-2-1.jdf
```

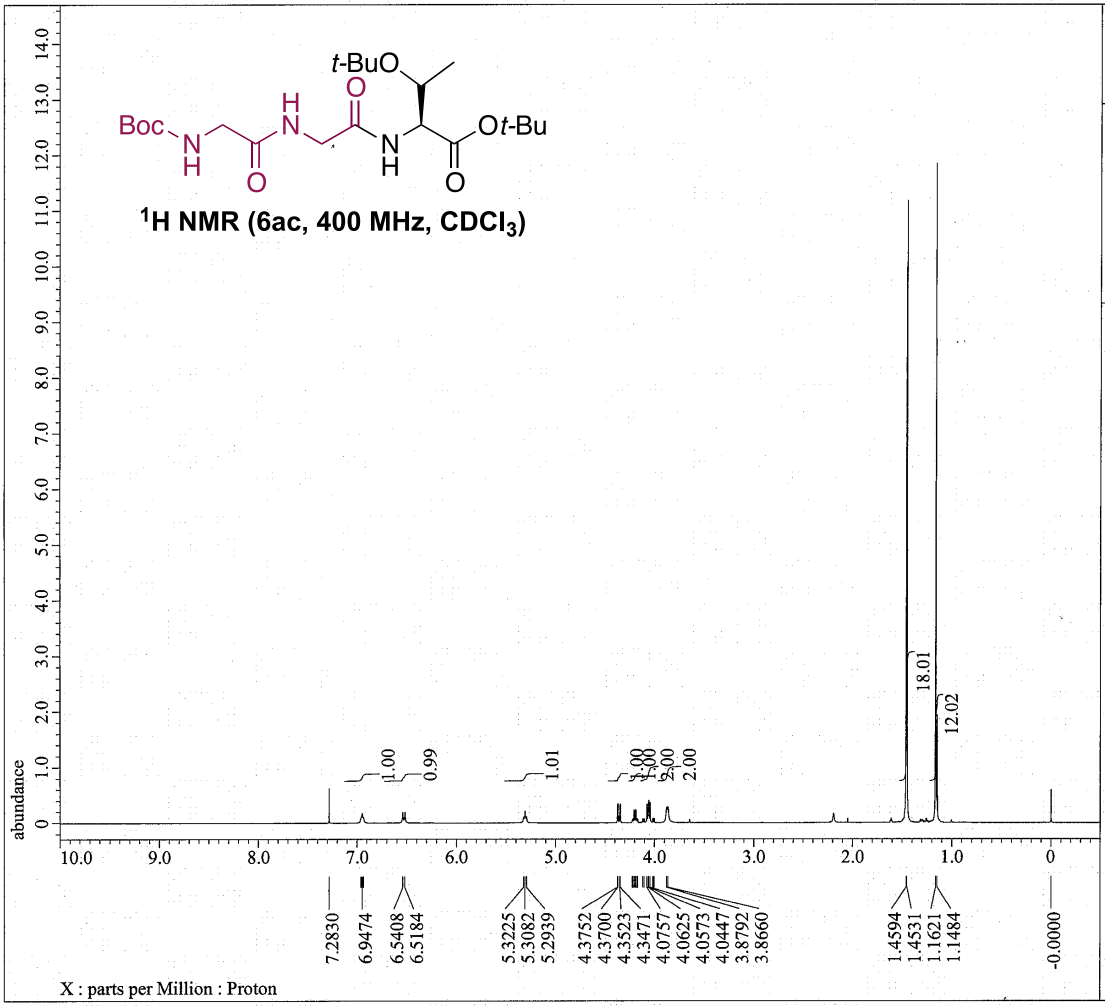
```
Filename           = wm12-46_E1H-2-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-46
Solvent            = CHLOROFORM-D
Creation_Time      =  8-JUL-2021 11:16:12
Revision_Time      =  8-JUL-2021 13:08:43
Current_Time       =  8-JUL-2021 13:09:14

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 28
Temp_Get         = 24.1[dC]
X_90_Width       = 6.47[us]
X_Acq_Time       = 4.36731904[s]
X_Angle          = 45[deg]
X_Atn            = 1[dB]
X_Pulse          = 3.235[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
```



$^{13}$C NMR (6ab, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-46-13C_E13C-2-1.jdf
```

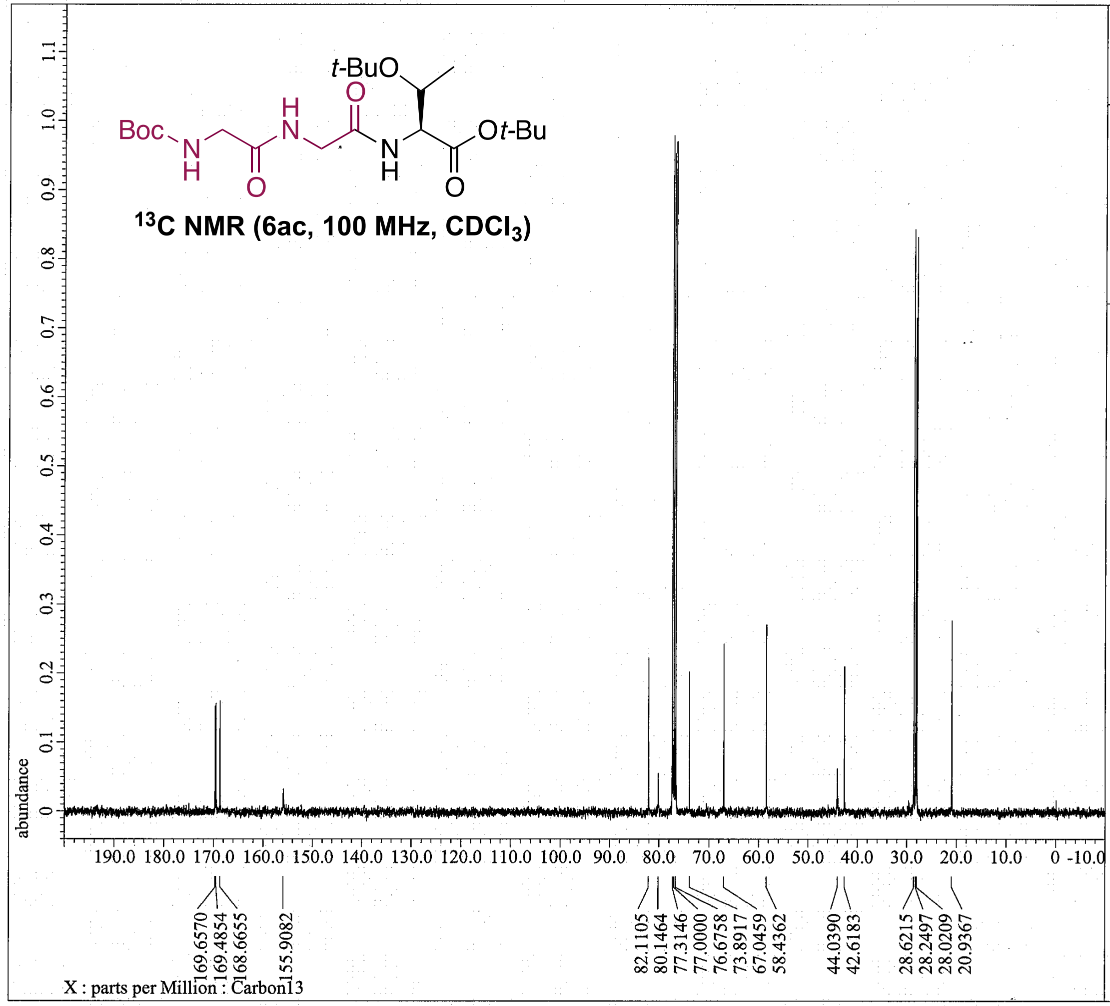
```
Filename            = wm12-46-13C_E13C-2-3.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-46-13C
Solvent             = CHLOROFORM-D
Creation_Time       =  8-JUL-2021 11:18:20
Revision_Time       =  8-JUL-2021 11:40:33
Current_Time        =  8-JUL-2021 11:40:50

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 513
Total_Scans         = 513

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 24[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (6ac, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-22_E1H-1-1.jdf
```

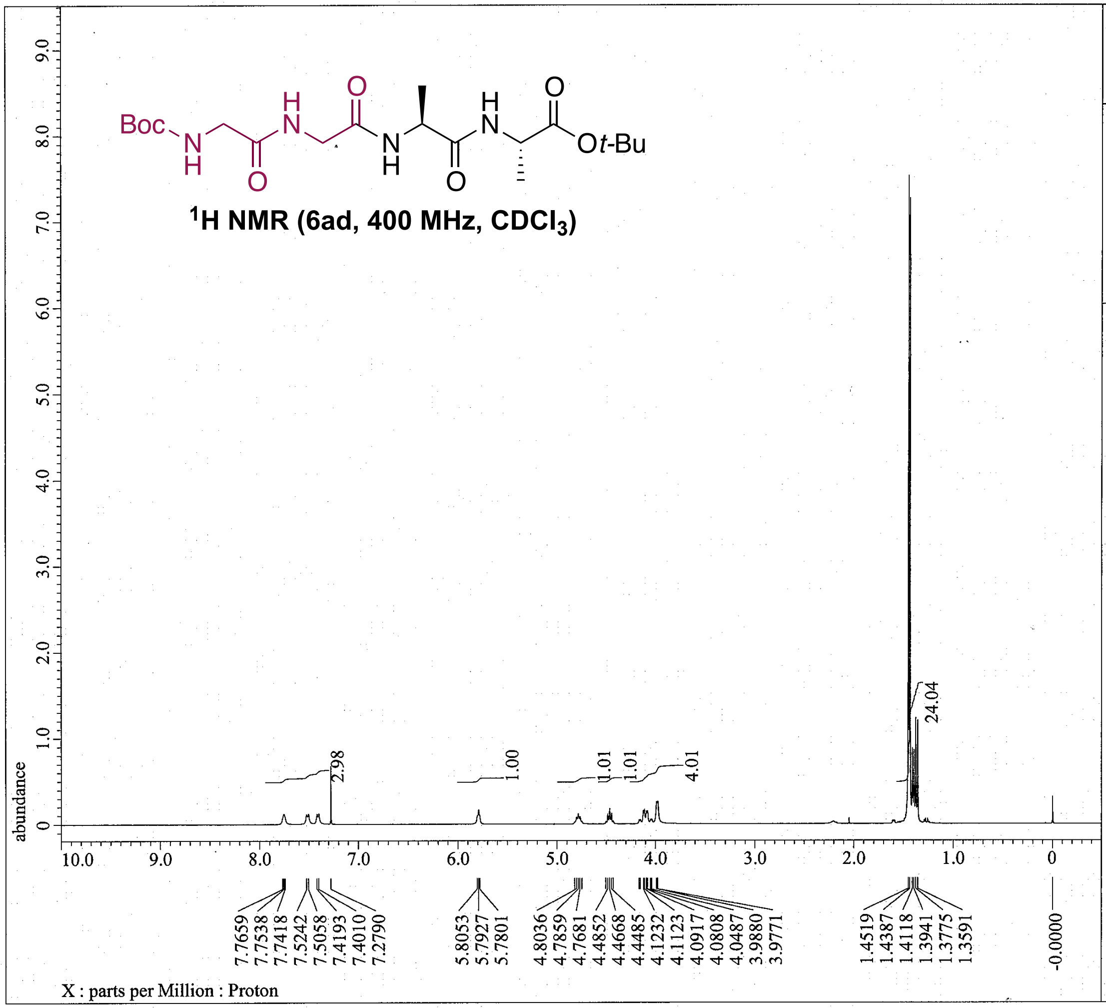
```
Filename            = wm12-22_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-22
Solvent             = CHLOROFORM-D
Creation_Time       = 17-JUN-2021 08:27:07
Revision_Time       = 17-JUN-2021 08:28:08
Current_Time        = 17-JUN-2021 08:28:18

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 26
Temp_Get            = 22.7[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (6ac, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-22-13C_E13C-1-1.jdf
```

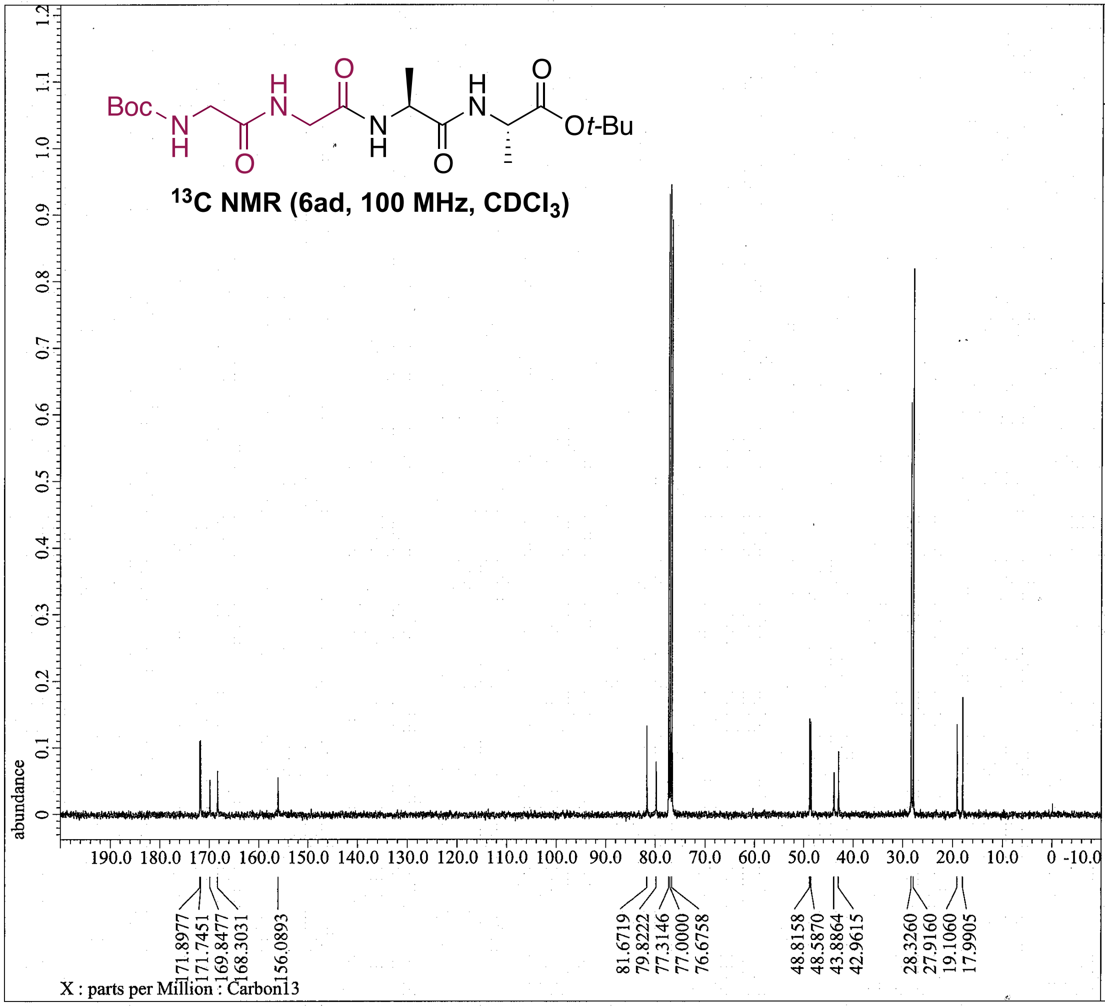
```
Filename          = wm12-22-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-22-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JUN-2021 08:31:23
Revision_Time     = 17-JUN-2021 08:54:03
Current_Time      = 17-JUN-2021 08:54:32

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 513
Total_Scans       = 513

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.6[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6ad, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-70-5_E1H-1-1.jdf
```

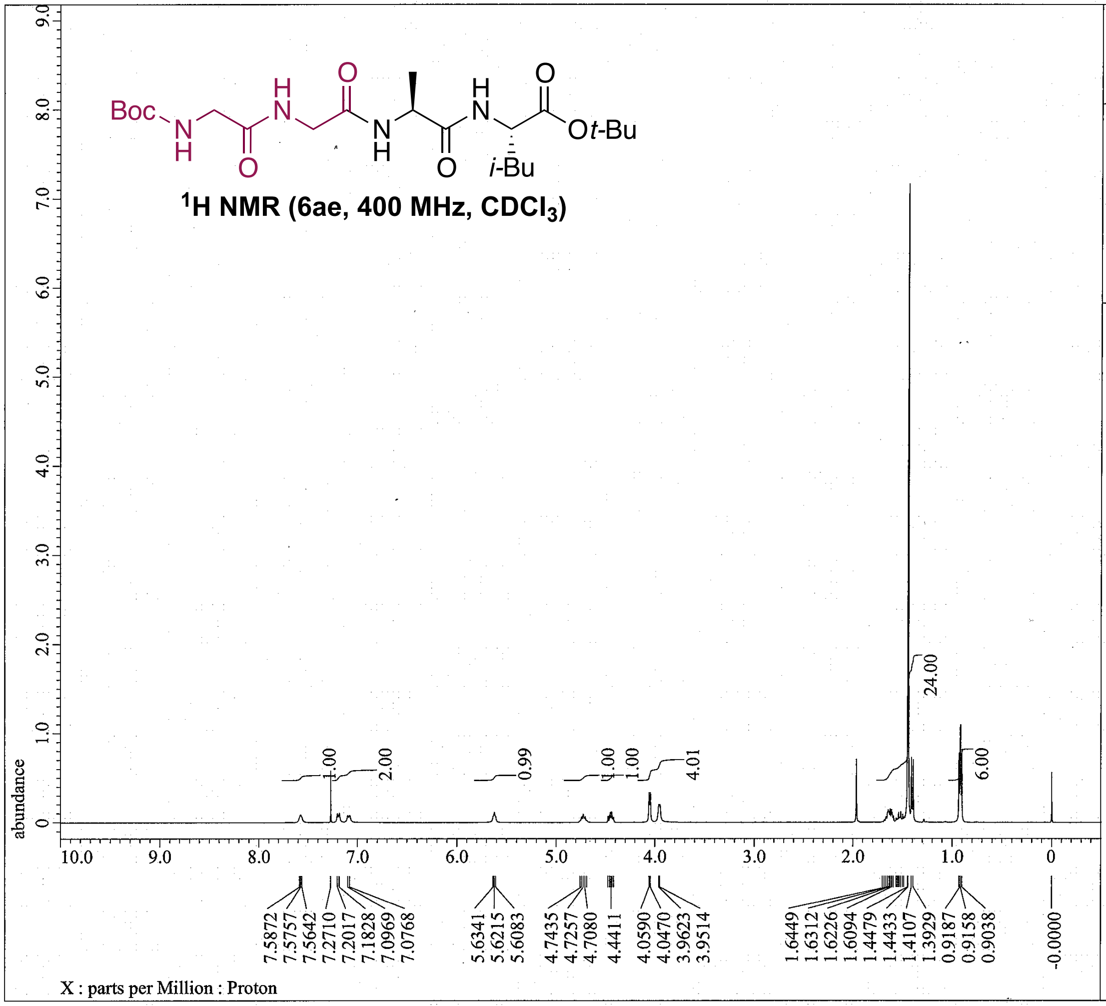
```
Filename           = wm12-70-5_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-70-5
Solvent            = CHLOROFORM-D
Creation_Time      =  4-AUG-2021 12:11:57
Revision_Time      =  4-AUG-2021 12:13:29
Current_Time       =  4-AUG-2021 12:13:43

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 26
Temp_Get           = 24.1[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}C$ NMR (6ad, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-70-5-13C_E13C-1-1.jdf
```

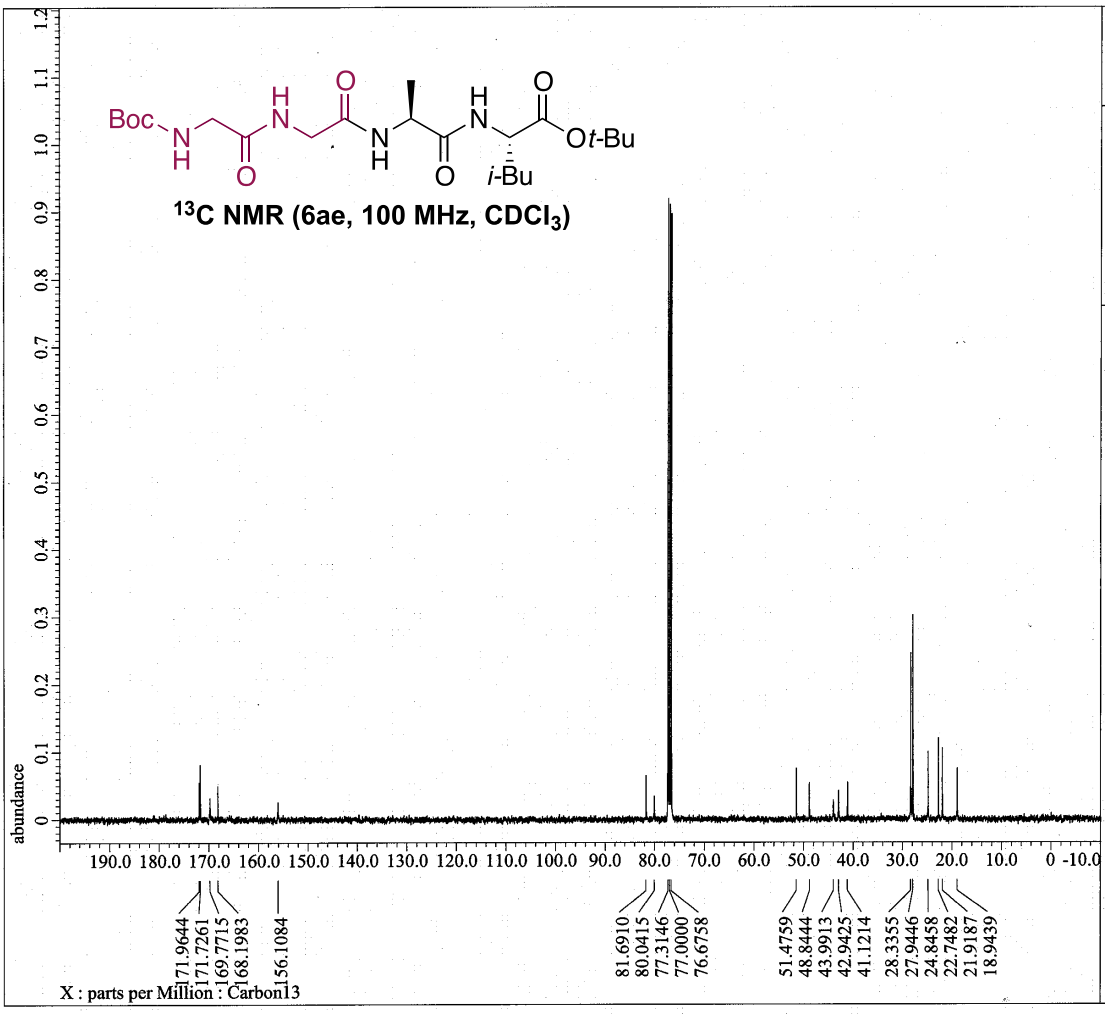
```
Filename          = wm12-70-5-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-70-5-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  4-AUG-2021 12:15:55
Revision_Time     =  4-AUG-2021 12:57:57
Current_Time      =  4-AUG-2021 12:58:18

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 901
Total_Scans       = 901

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6ae, 400 MHz, CDCl$_3$)

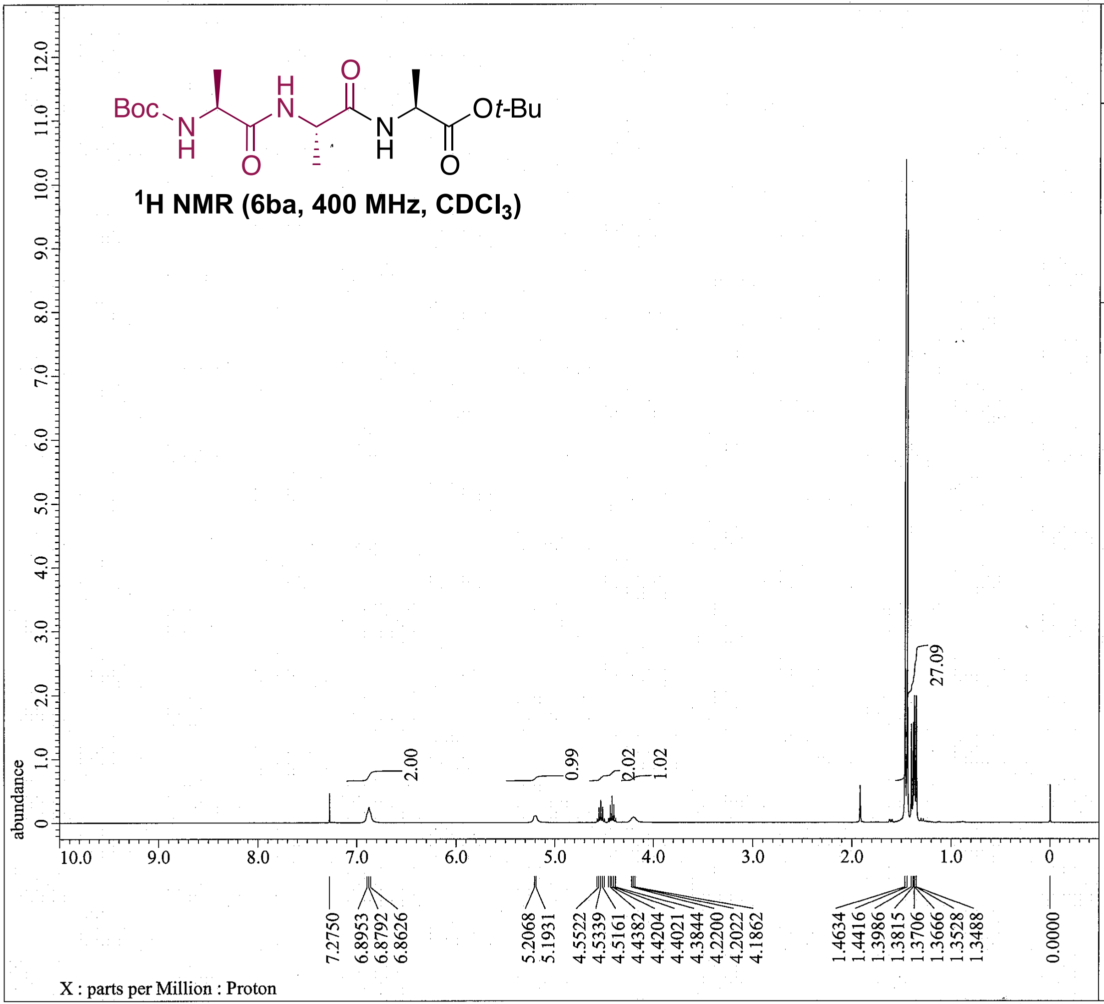
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-70-6_E1H-1-1.jdf
```

```
Filename          = wm12-70-6_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-70-6
Solvent           = CHLOROFORM-D
Creation_Time     =  5-AUG-2021 10:54:09
Revision_Time     =  5-AUG-2021 10:57:59
Current_Time      =  5-AUG-2021 10:58:09

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.7[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-70-6-13C_E13C-1-1.jdf
```

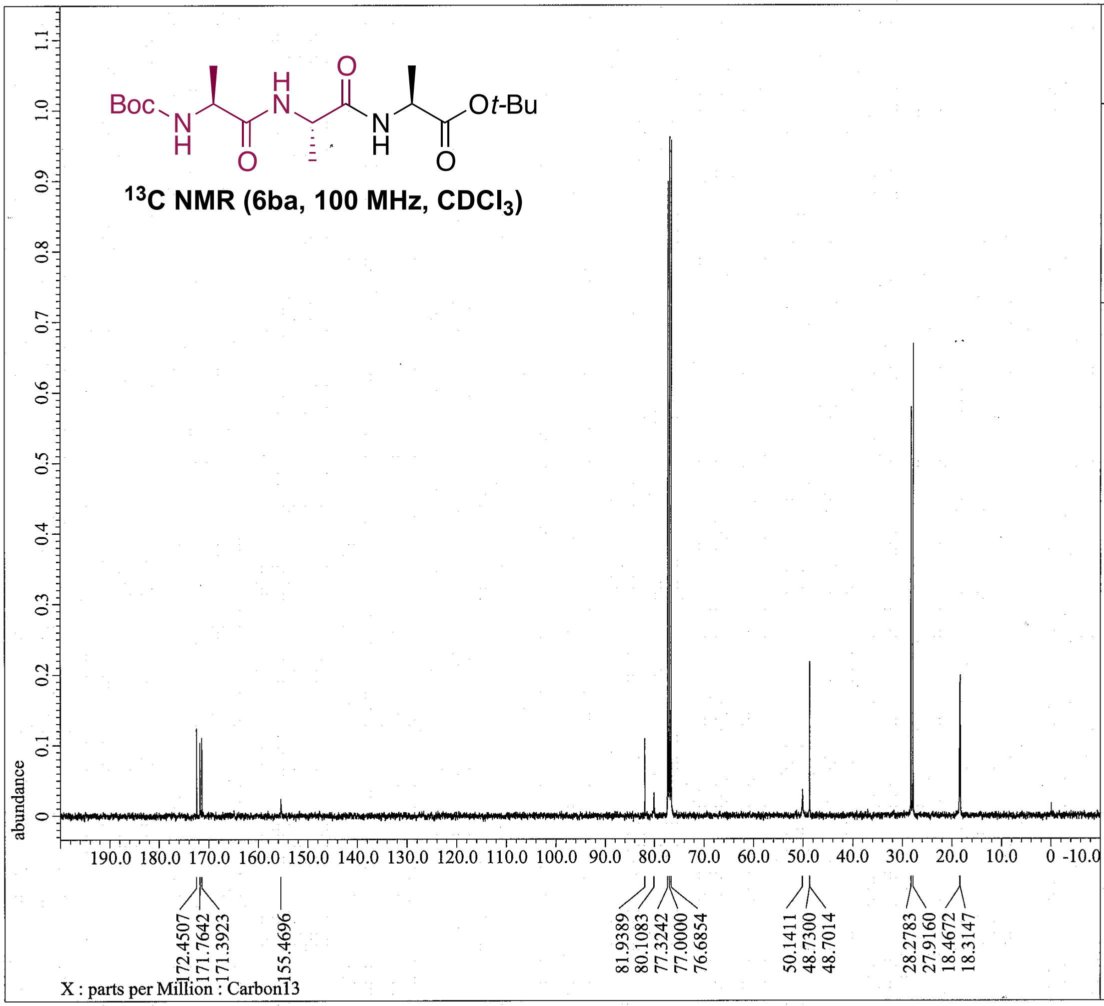
```
Filename          = wm12-70-6-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-70-6-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  5-AUG-2021 10:57:29
Revision_Time     =  5-AUG-2021 11:45:36
Current_Time      =  5-AUG-2021 11:46:00

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.5[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6ba, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-191_E1H-2-1.jdf
```

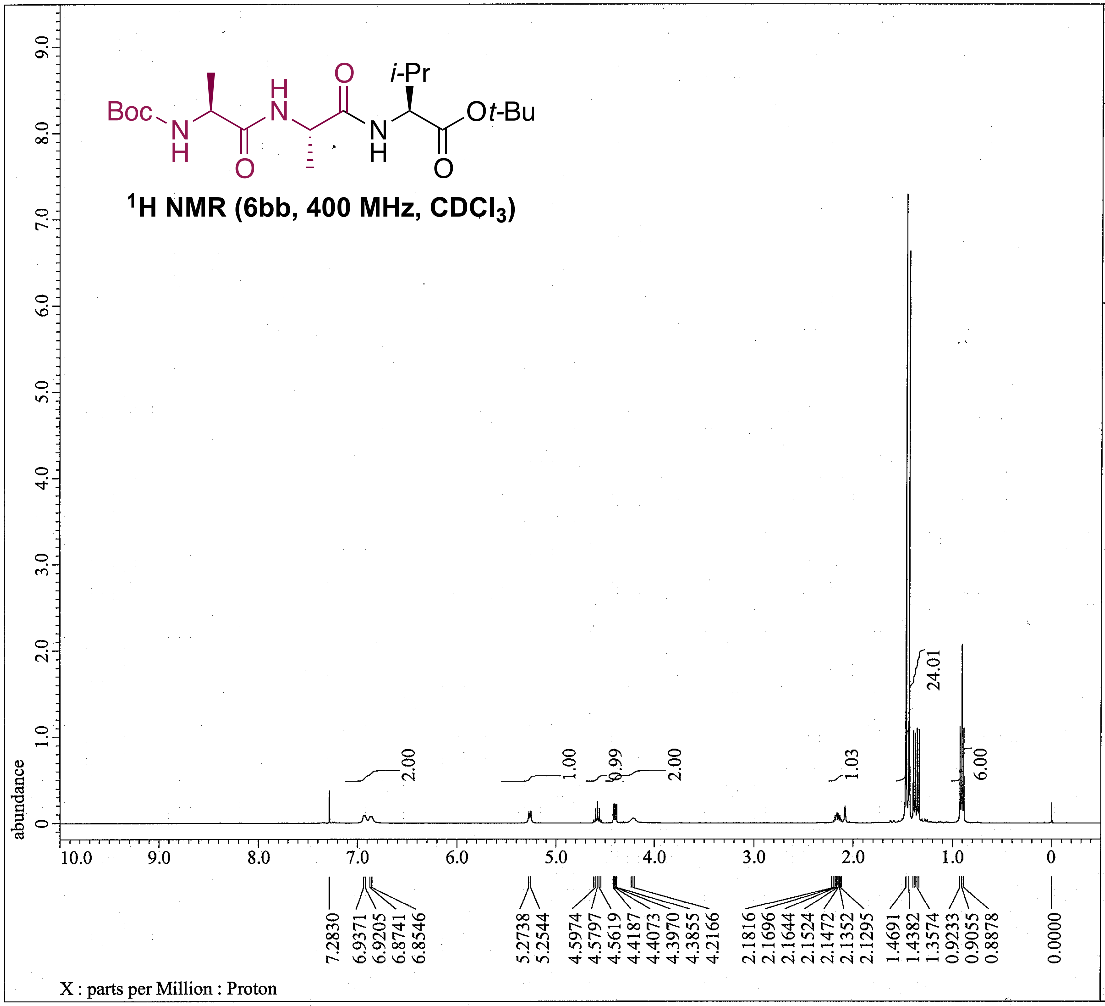
```
Filename          = wm11-191_E1H-2-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm11-191
Solvent           = CHLOROFORM-D
Creation_Time     = 19-MAY-2021 14:55:45
Revision_Time     = 19-MAY-2021 14:57:11
Current_Time      = 19-MAY-2021 14:57:24

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 22.7[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (6ba, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm11-191-13C_E13C-1-1.jdf
```

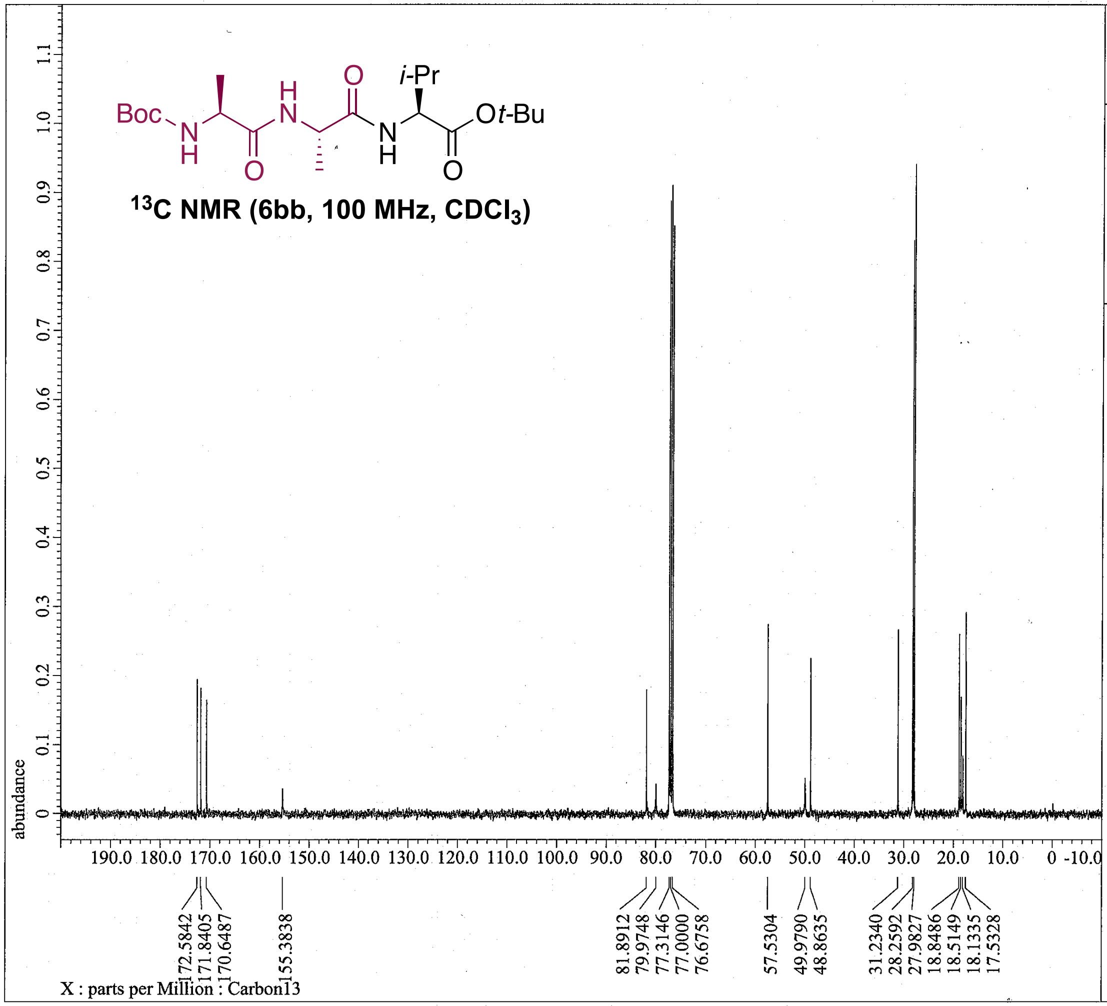
```
Filename          = wm11-191-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm11-191-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 19-MAY-2021 15:00:06
Revision_Time     = 19-MAY-2021 15:47:47
Current_Time      = 19-MAY-2021 15:48:09

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6bb, 400 MHz, CDCl$_3$)

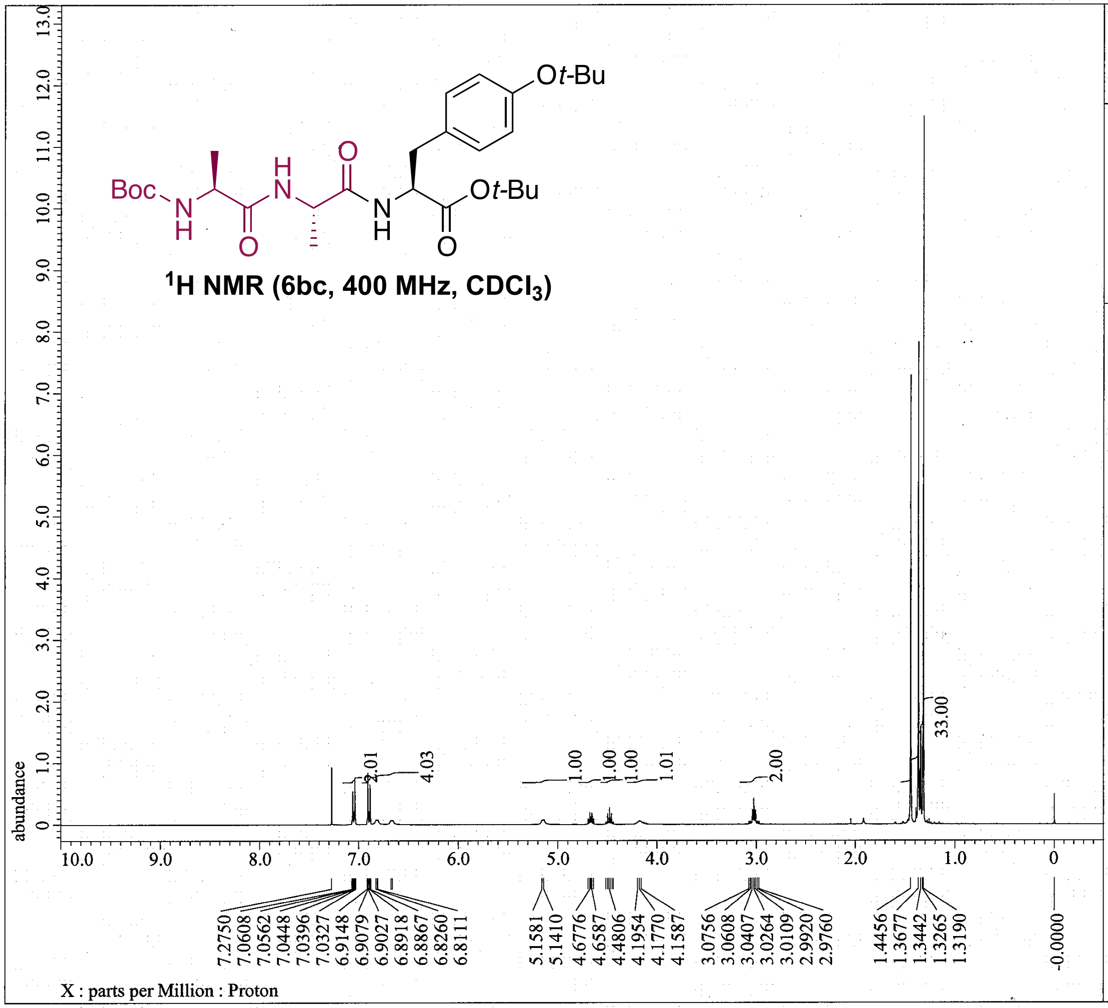
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-113-7_E1H-1-1.jdf
```

```
Filename            = wm12-113-7_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-113-7
Solvent             = CHLOROFORM-D
Creation_Time       =  8-OCT-2021 16:04:10
Revision_Time       =  8-OCT-2021 16:05:49
Current_Time        =  8-OCT-2021 16:06:14

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 24
Temp_Get            = 24[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



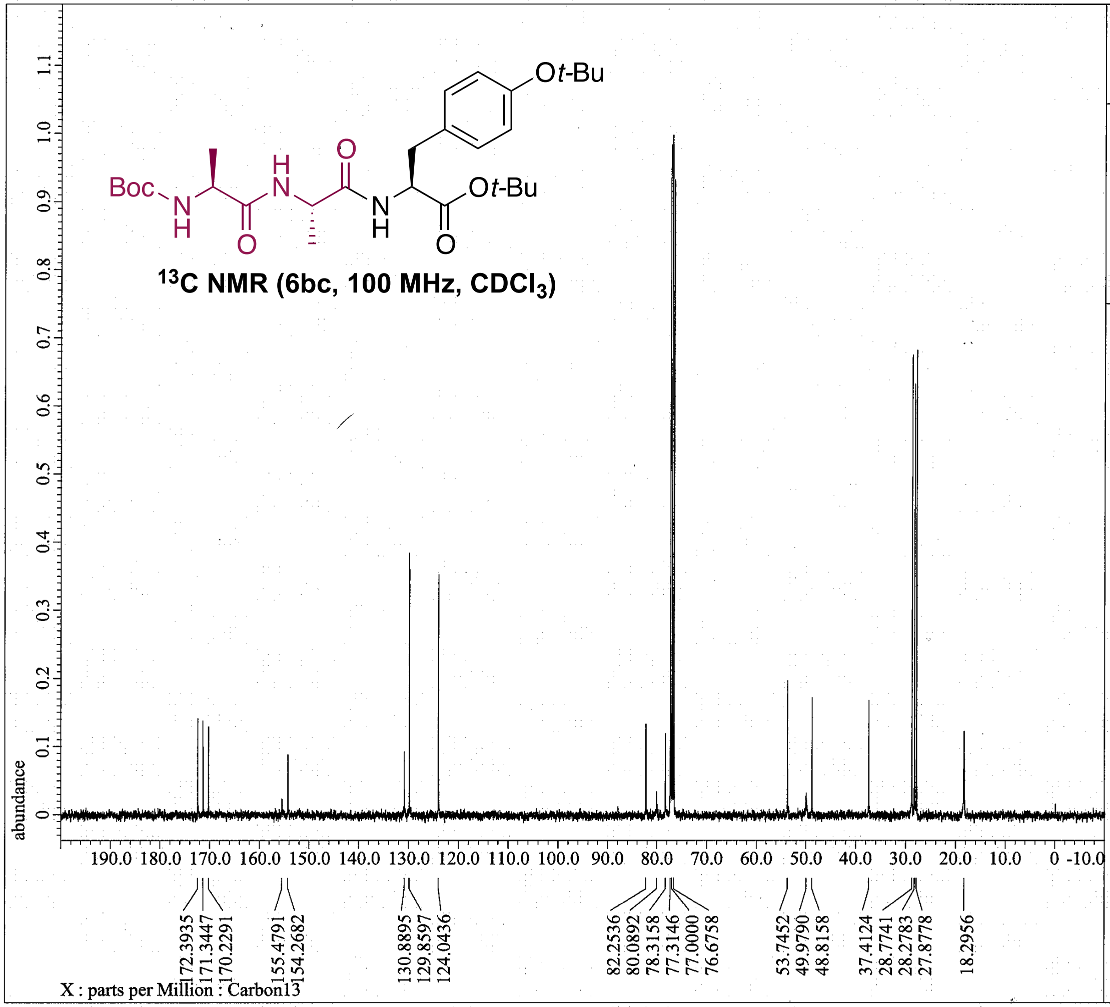
$^{13}$C NMR (6bb, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-113-7-13C_E13C-1-1.jdf

Filename          = wm12-113-7-13C_E13C-1-5.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-113-7-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  8-OCT-2021 16:06:16
Revision_Time     =  8-OCT-2021 16:33:32
Current_Time      =  8-OCT-2021 16:33:45

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 602
Total_Scans       = 602

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```


$^1$H NMR (6bc, 400 MHz, CDCl$_3$)

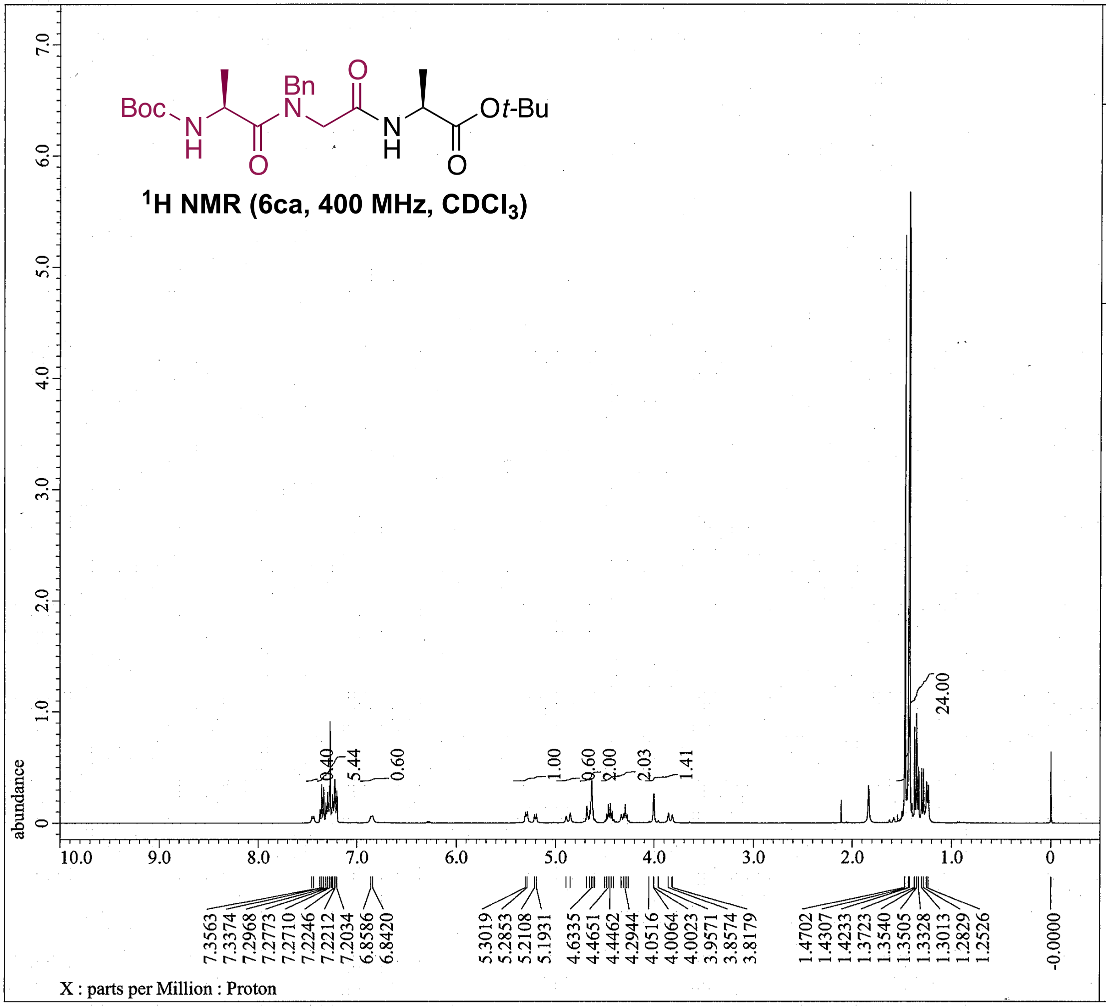
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-2-4_E1H-1-1.jdf
```

```
Filename           = wm13-2-4_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm13-2-4
Solvent            = CHLOROFORM-D
Creation_Time      = 31-JAN-2022 14:24:53
Revision_Time      = 31-JAN-2022 14:28:54
Current_Time       = 31-JAN-2022 14:29:05

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 26
Temp_Get           = 22.8[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```


$^{13}$C NMR (6bc, 100 MHz, CDCl$_3$)

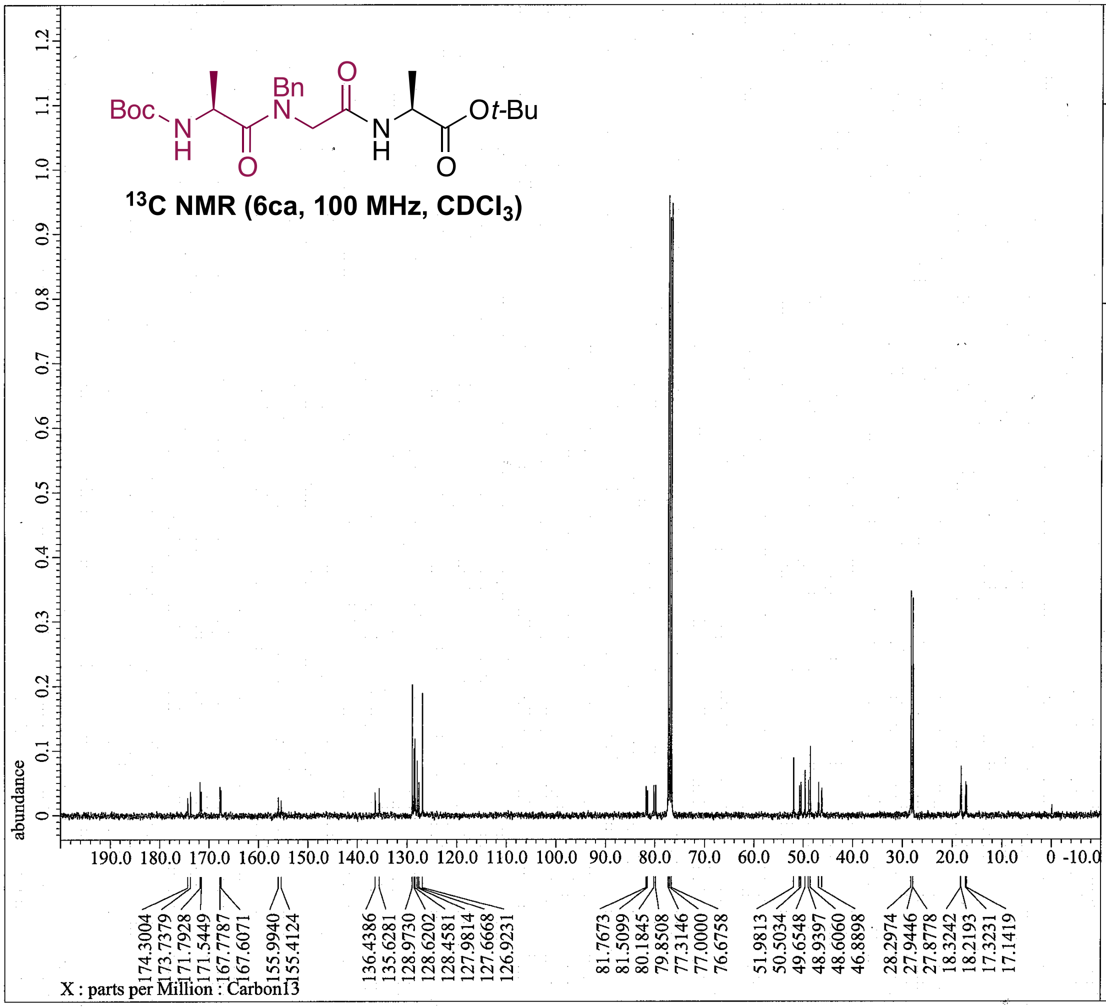
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-2-4-13C_E13C-1-1.jdf
```

```
Filename            = wm13-2-4-13C_E13C-1-4.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm13-2-4-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 31-JAN-2022 14:27:05
Revision_Time       = 31-JAN-2022 14:48:26
Current_Time        = 31-JAN-2022 14:48:50

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = TRUE
Scans               = 512
Total_Scans         = 512

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 23.9[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



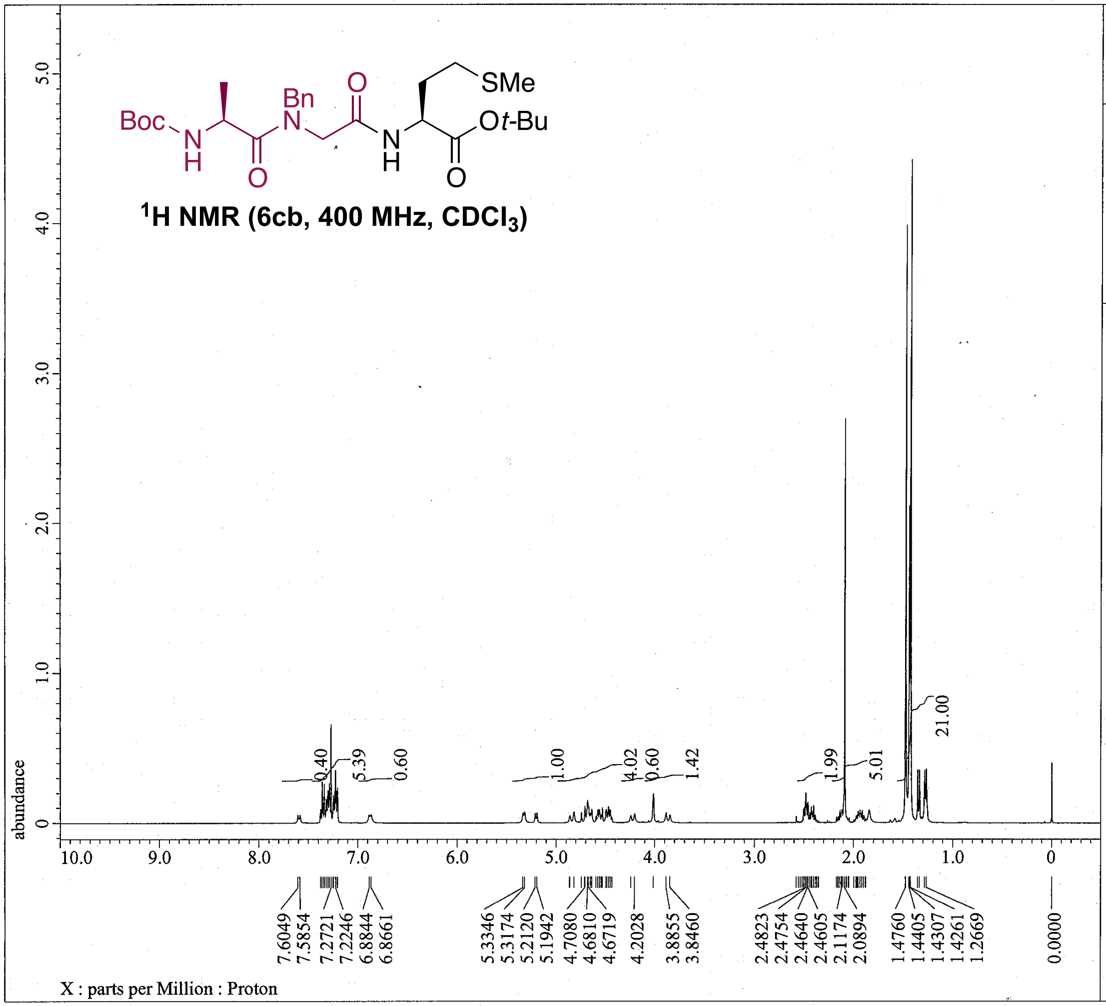
<sup></sup>$^1$H NMR (6ca, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-80-3_E1H-1-1.jdf

Filename          = wm12-80-3_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-80-3
Solvent           = CHLOROFORM-D
Creation_Time     = 26-AUG-2021 11:02:41
Revision_Time     = 26-AUG-2021 11:10:53
Current_Time      = 26-AUG-2021 11:11:06

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.9[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```


$^{13}$C NMR (6ca, 100 MHz, CDCl$_3$)

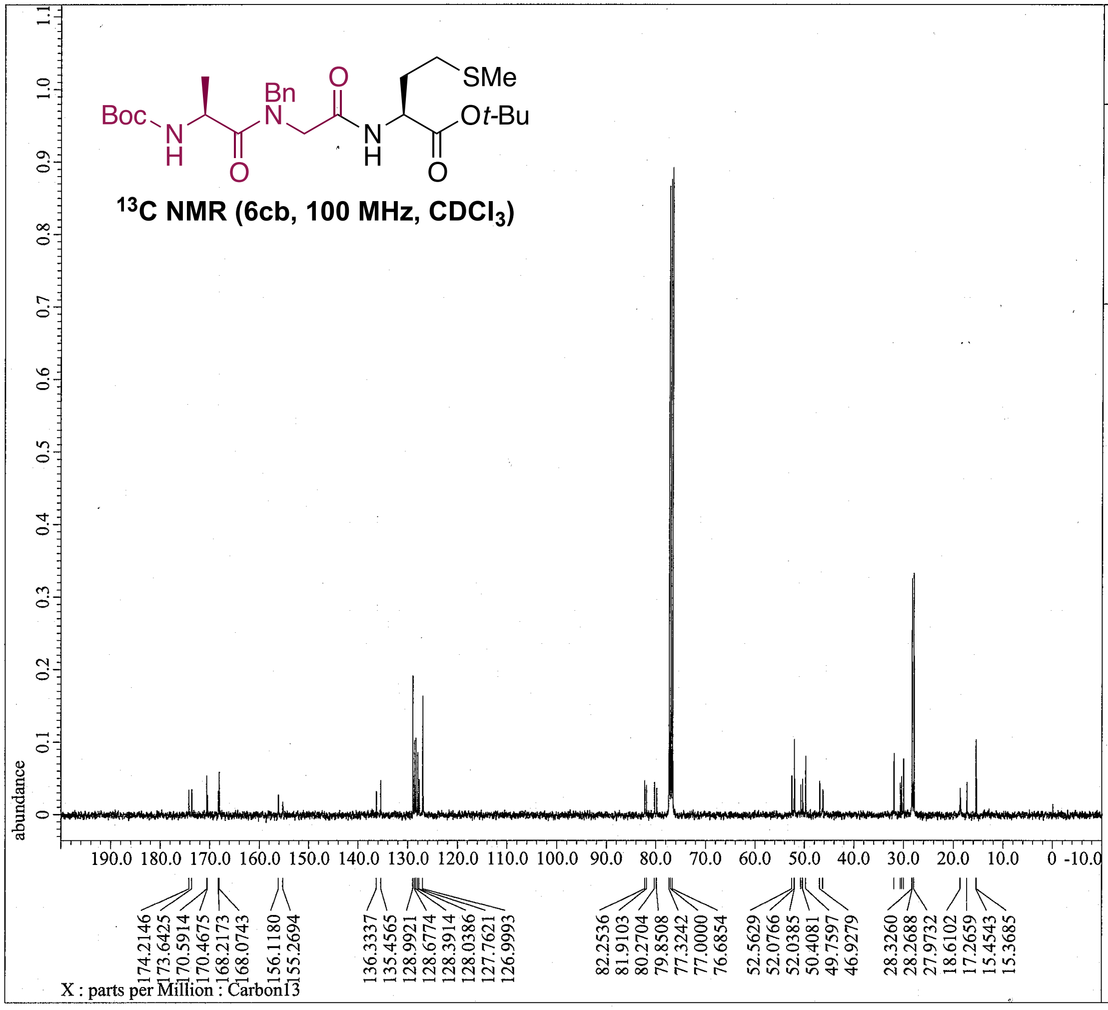
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-80-3-13C_E13C-1-1.jdf
```

```
Filename          = wm12-80-3-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-80-3-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 26-AUG-2021 11:04:50
Revision_Time     = 26-AUG-2021 11:53:07
Current_Time      = 26-AUG-2021 11:53:38

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.1[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6cb, 400 MHz, CDCl$_3$)

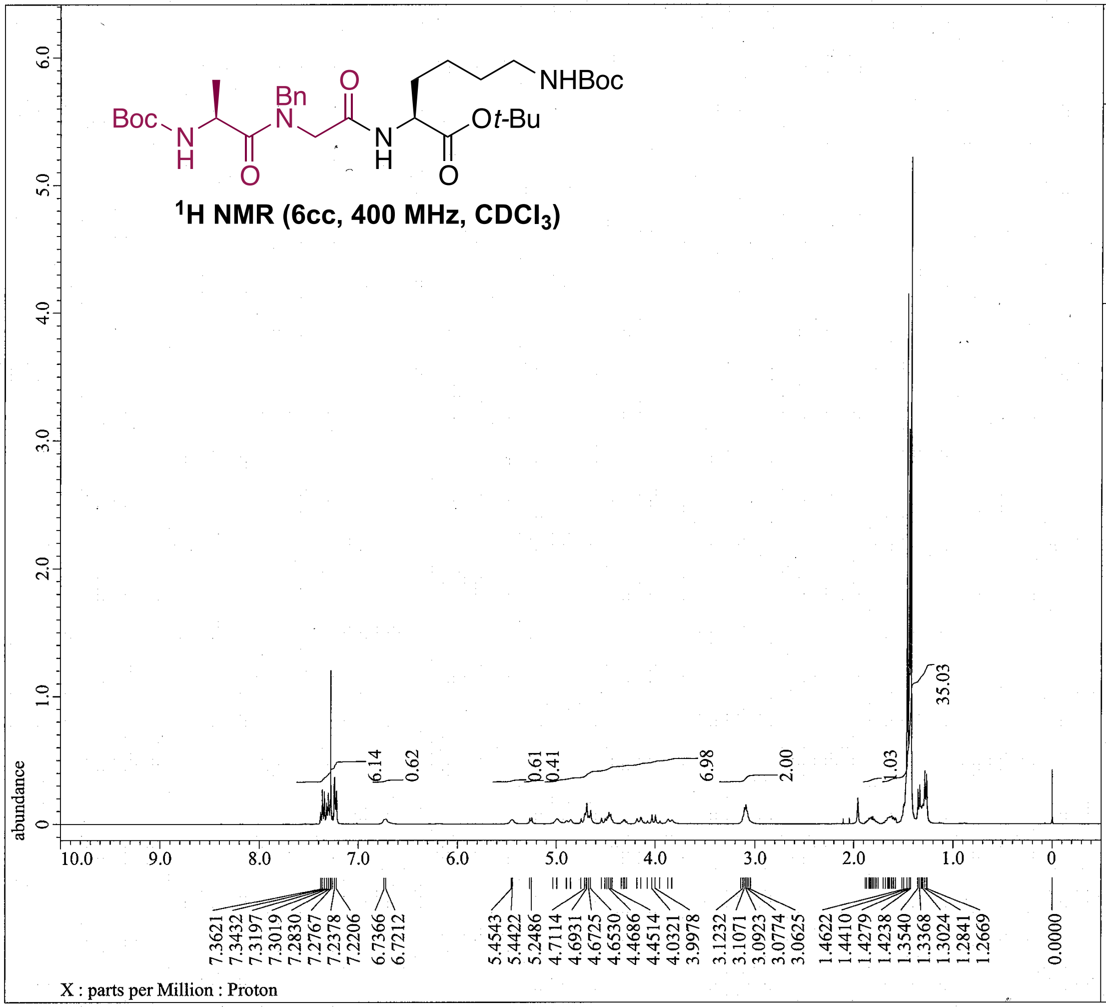
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-136-1_E1H-1-1.jdf

Filename           = wm12-136-1_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-136-1
Solvent            = CHLOROFORM-D
Creation_Time      = 25-OCT-2021 13:27:08
Revision_Time      = 25-OCT-2021 13:36:11
Current_Time       = 25-OCT-2021 13:37:14

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 28
Temp_Get           = 23.7[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (6cb, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-136-1-13C_E13C-1-1.jdf
```

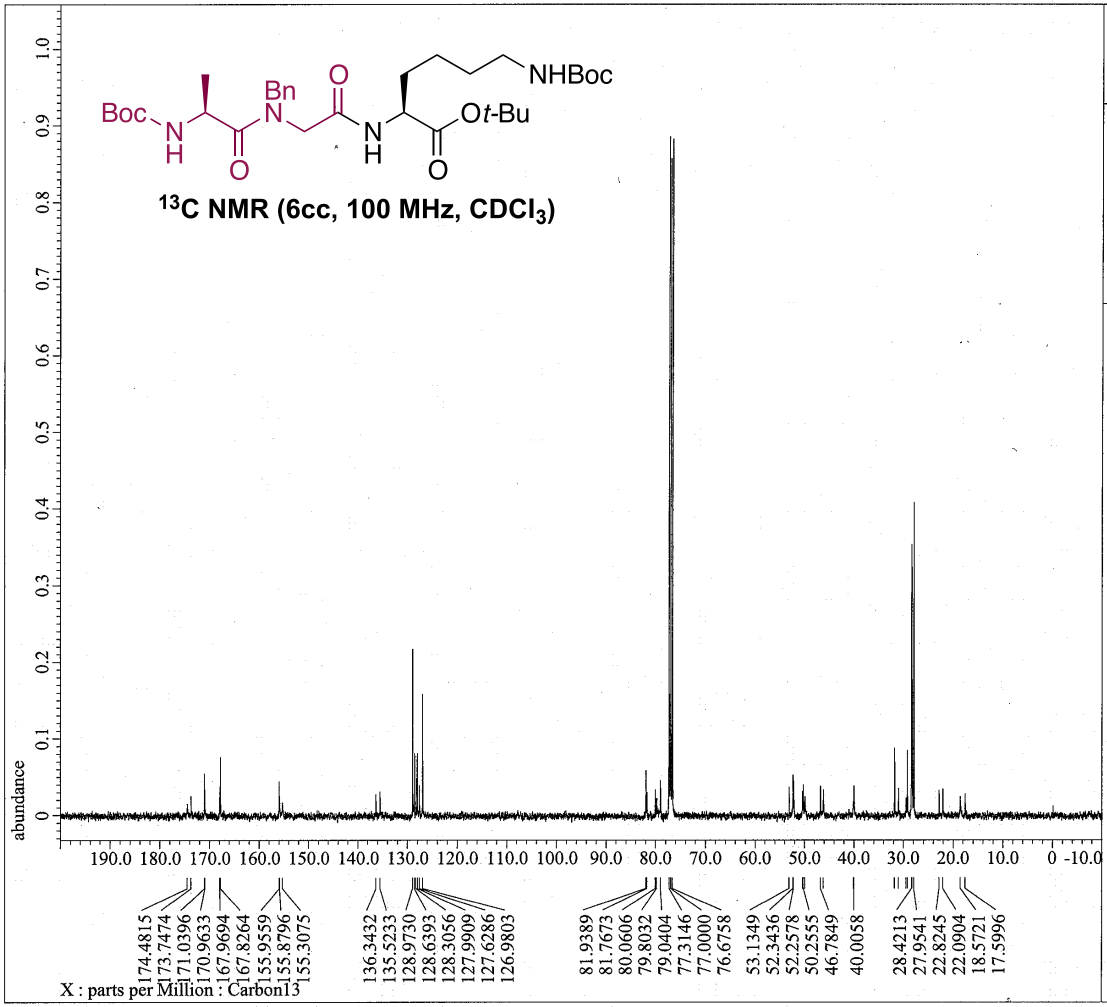
```
Filename           = wm12-136-1-13C_E13C-1-4.jd
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-136-1-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 25-OCT-2021 13:29:20
Revision_Time      = 25-OCT-2021 14:08:25
Current_Time       = 25-OCT-2021 14:08:44

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 800
Total_Scans        = 800

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.6[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (6cc, 400 MHz, CDCl$_3$)

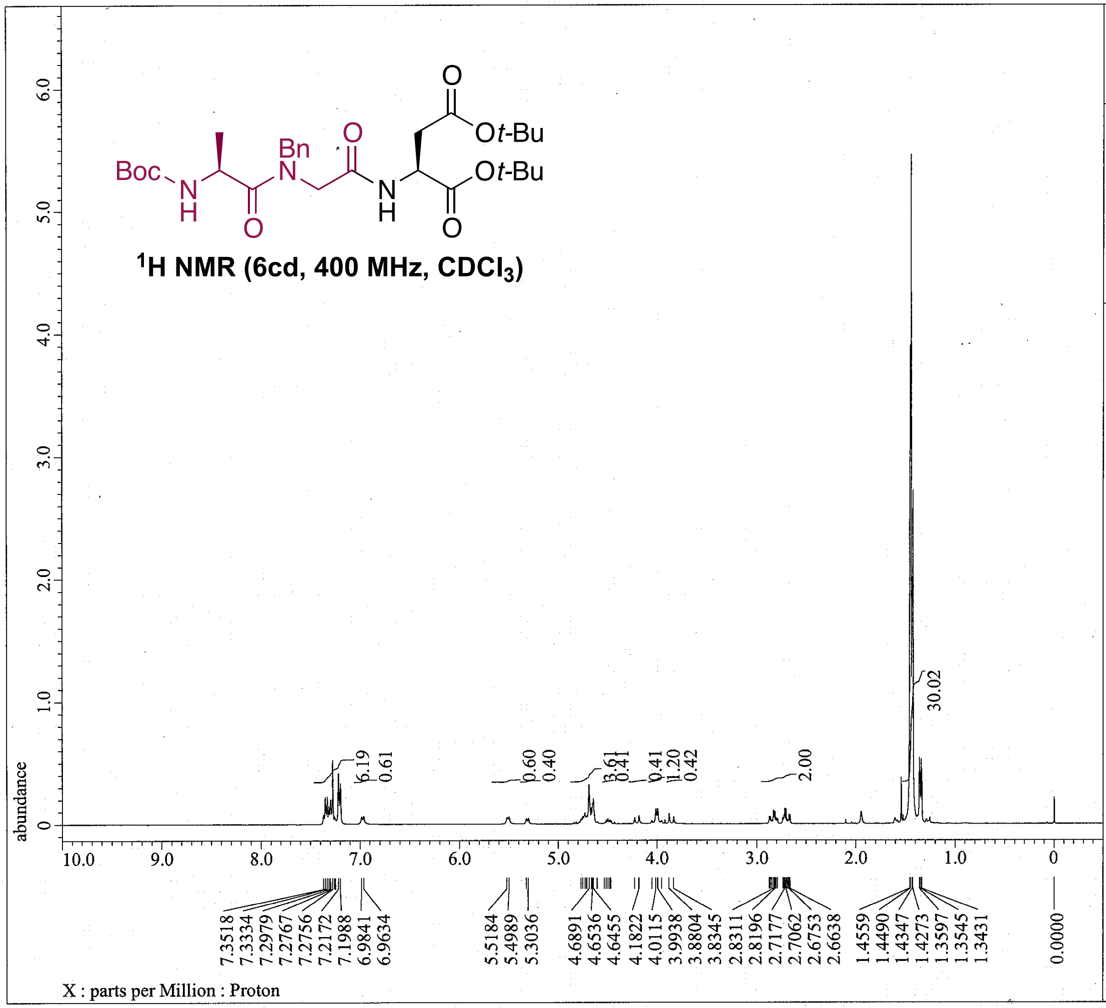
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-136-3_E1H-1-1.jdf
```

```
Filename          = wm12-136-3_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-136-3
Solvent           = CHLOROFORM-D
Creation_Time     = 25-OCT-2021 15:55:14
Revision_Time     = 25-OCT-2021 16:03:28
Current_Time      = 25-OCT-2021 16:03:44

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 24.2[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



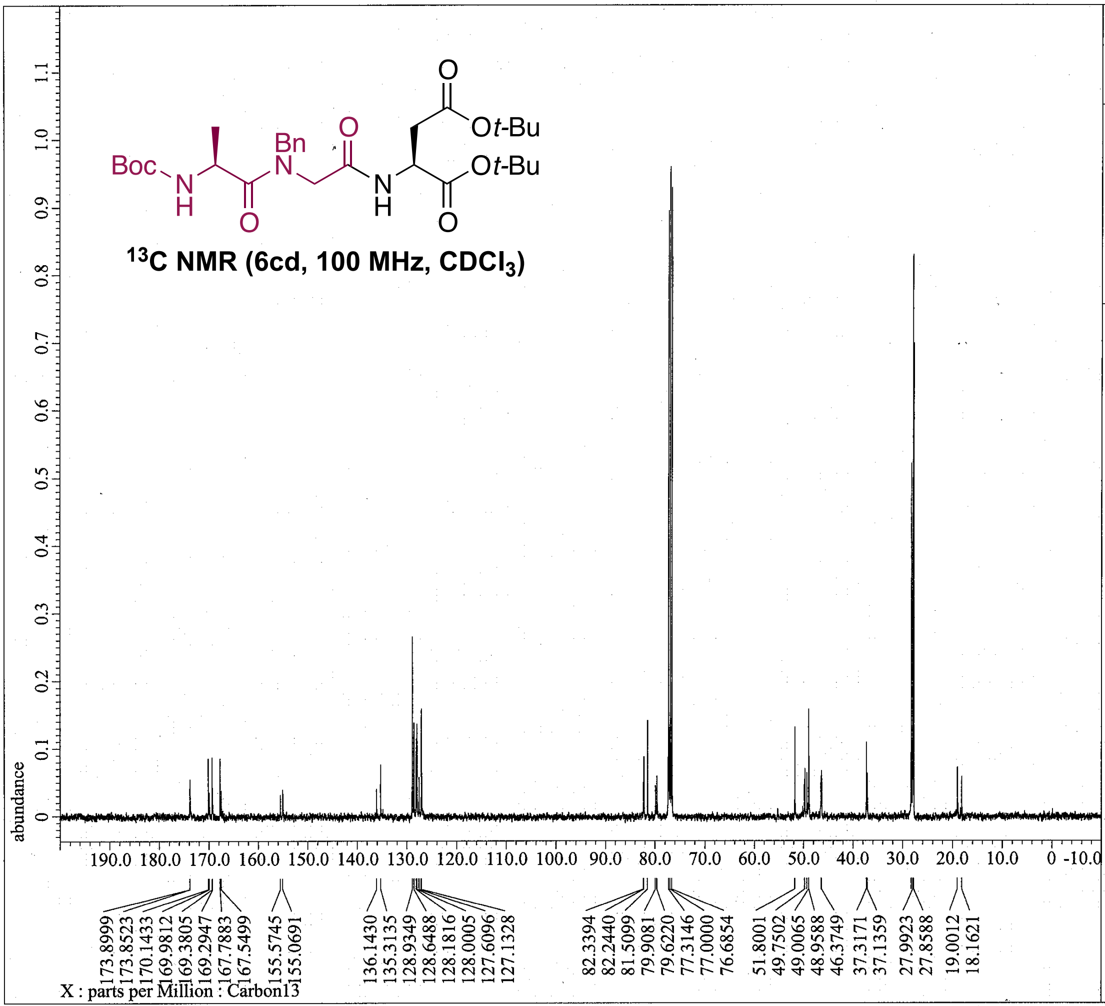
$^{13}$C NMR (6cc, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-136-3-13C_E13C-1-1.jdf

Filename          = wm12-136-3-13C_E13C-1-4.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-136-3-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 25-OCT-2021 15:57:23
Revision_Time     = 25-OCT-2021 16:45:54
Current_Time      = 25-OCT-2021 16:46:16

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.6[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```


**$^1$H NMR (6cd, 400 MHz, CDCl$_3$)**

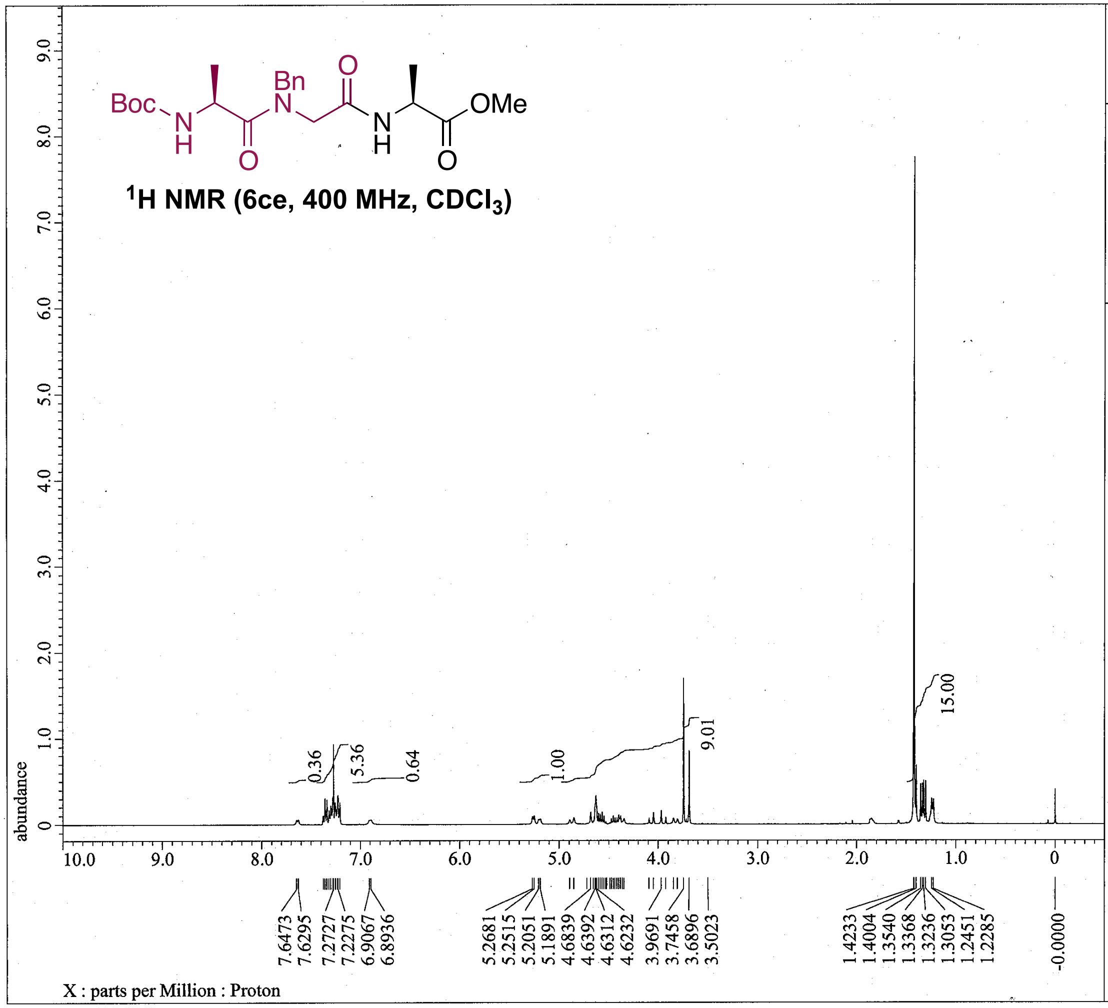
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-136-2_E1H-1-1.jdf
```

```
Filename           = wm12-136-2_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-136-2
Solvent            = CHLOROFORM-D
Creation_Time      = 26-OCT-2021 11:45:38
Revision_Time      = 26-OCT-2021 11:49:05
Current_Time       = 26-OCT-2021 11:49:16

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 24
Temp_Get           = 23.5[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-136-2-13C_E13C_copy1-1-1.j
```

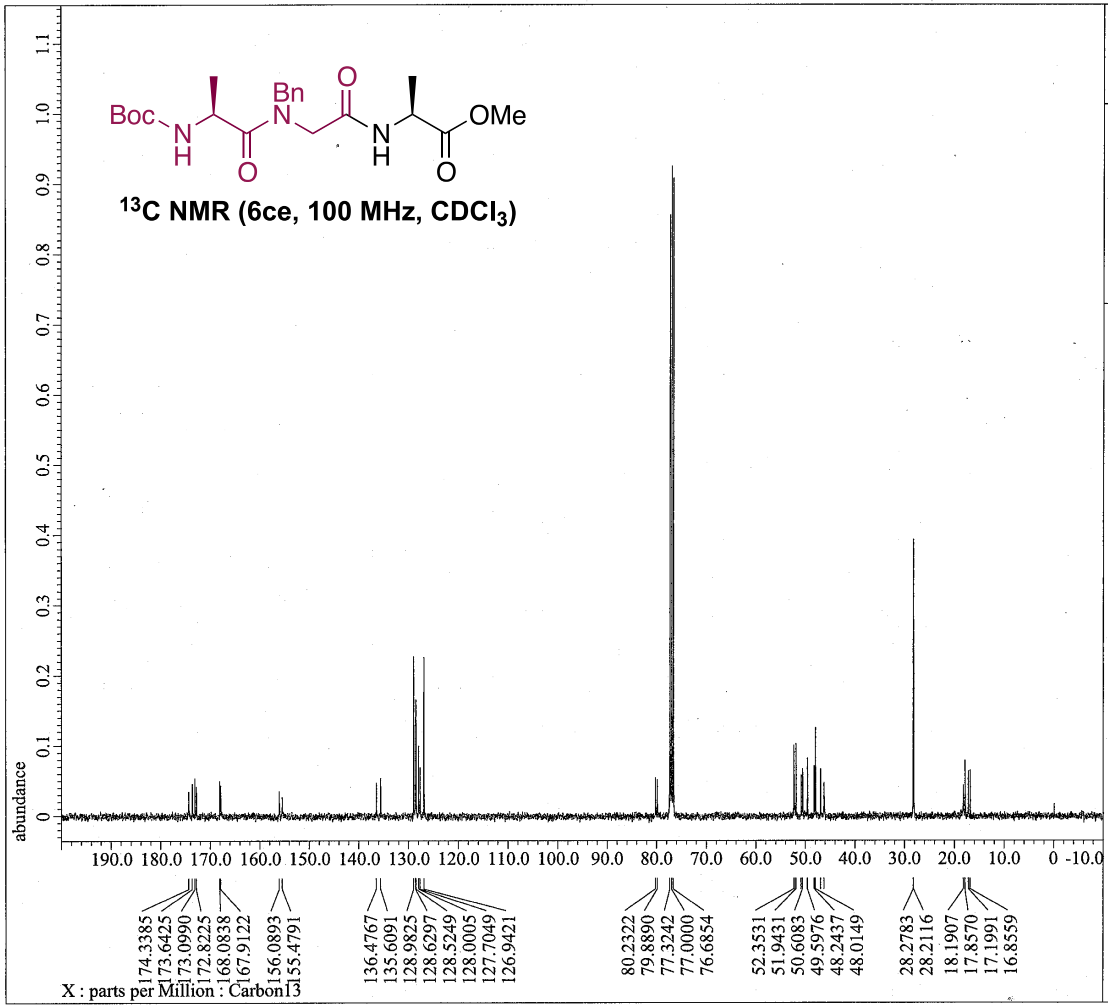
```
Filename           = wm12-136-2-13C_E13C_copy1-
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-136-2-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 26-OCT-2021 11:47:44
Revision_Time      = 26-OCT-2021 12:29:55
Current_Time       = 26-OCT-2021 12:30:23

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 0[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Incomplete_Copy    = TRUE
Scans              = 834
Total_Scans        = 834

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.6[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
```



**$^1$H NMR (6ce, 400 MHz, CDCl$_3$)**

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-132_E1H-1-1.jdf
```

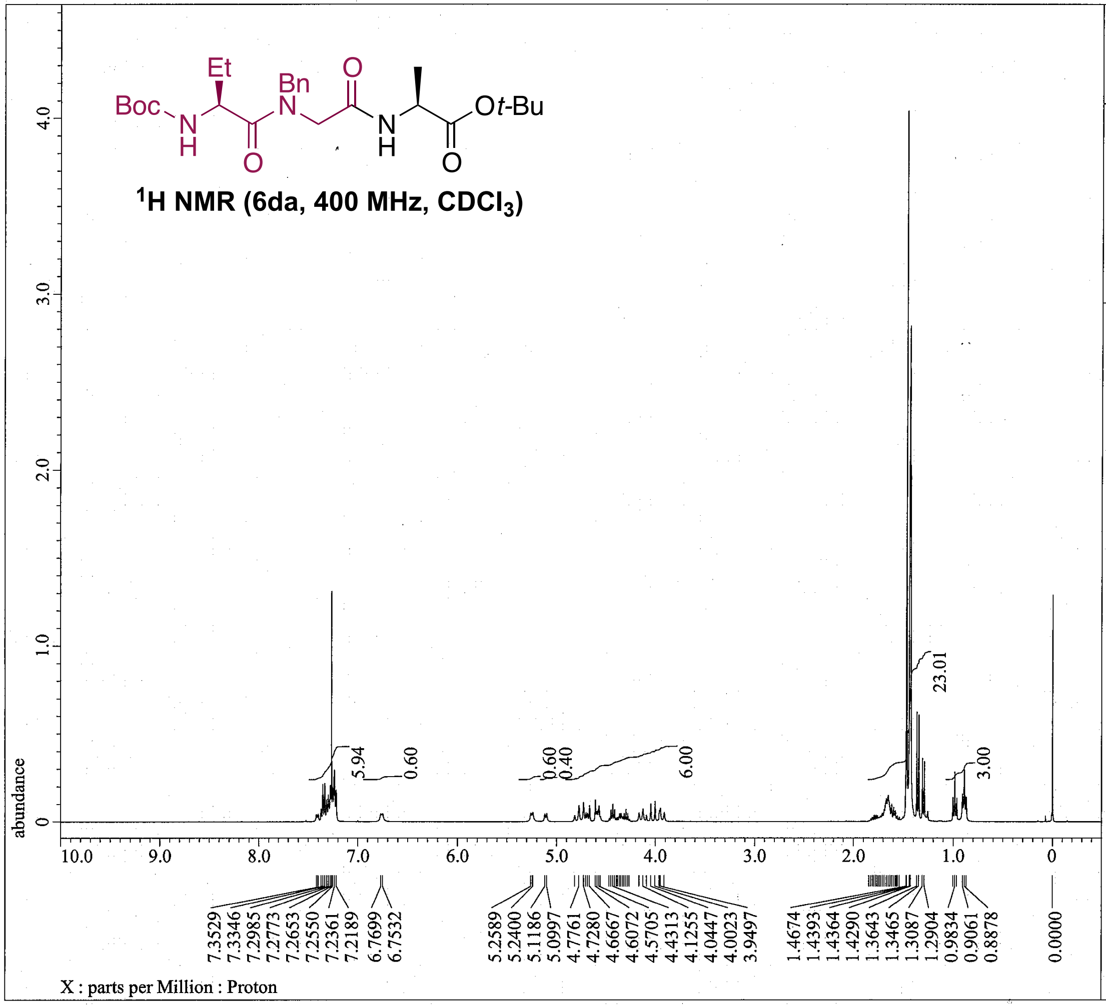
```
Filename          = wm12-132_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-132
Solvent           = CHLOROFORM-D
Creation_Time     = 22-OCT-2021 11:14:46
Revision_Time     = 22-OCT-2021 11:21:10
Current_Time      = 22-OCT-2021 11:21:22

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 24.5[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (6ce, 100 MHz, $CDCl_3$)

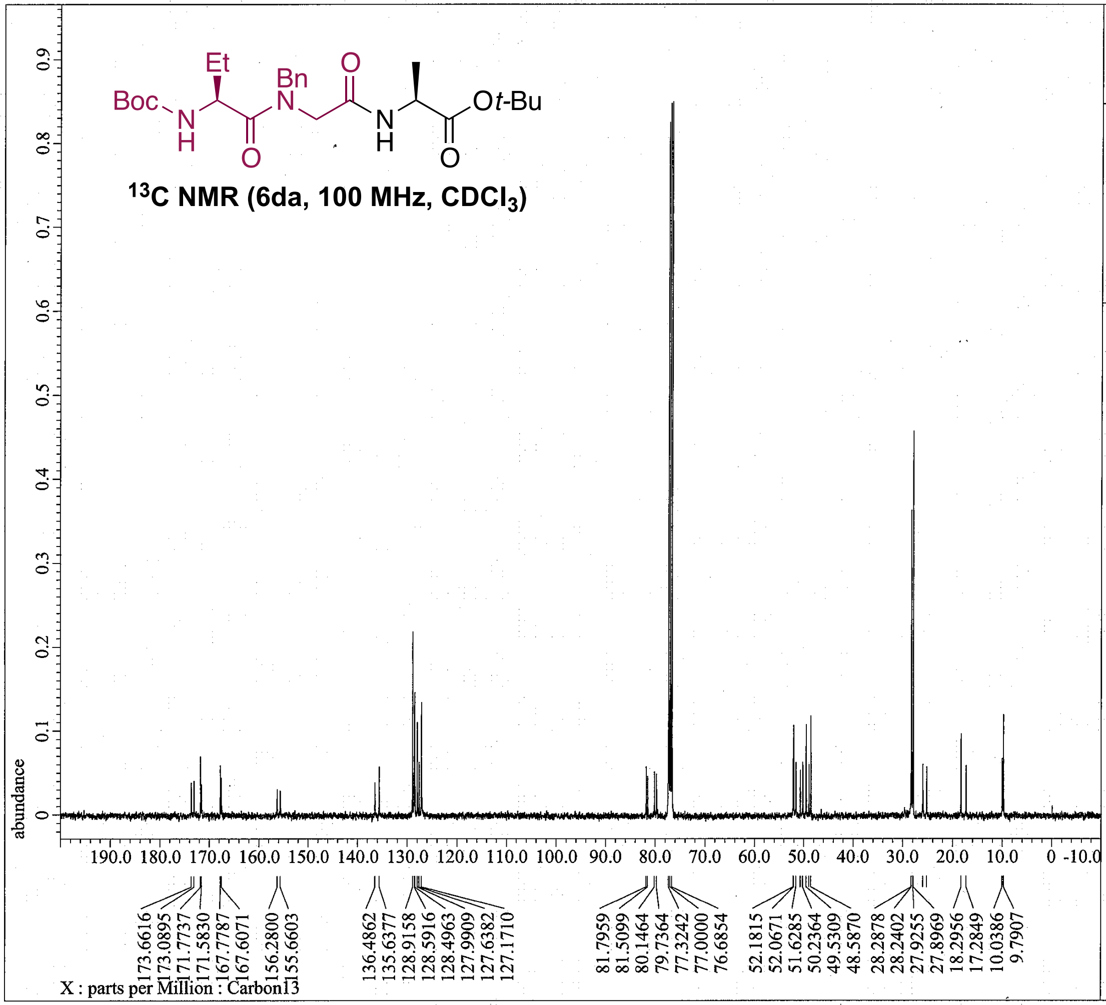
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-132-13C_E13C-1-1.jdf
```

```
Filename          = wm12-132-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-132-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 22-OCT-2021 11:17:35
Revision_Time     = 22-OCT-2021 12:00:39
Current_Time      = 22-OCT-2021 12:00:56

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 920.0
Total_Scans       = 920.0

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.1[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



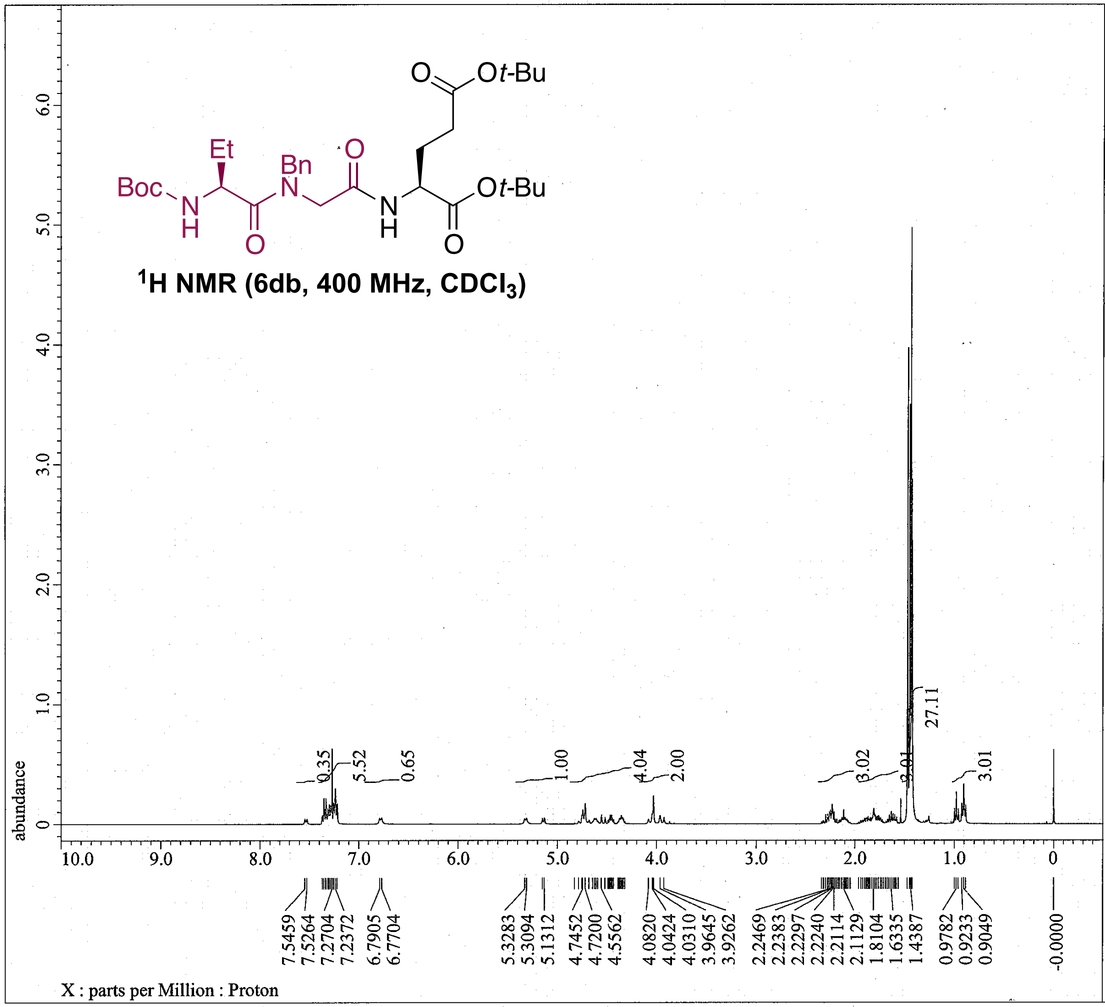
<sup></sup>$^{1}$H NMR (6da, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-30-_E1H-1-1.jdf

Filename           = wm12-30-_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-30-
Solvent            = CHLOROFORM-D
Creation_Time      = 28-JUN-2021 09:31:40
Revision_Time      = 28-JUN-2021 12:12:51
Current_Time       = 28-JUN-2021 12:13:20

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 36
Temp_Get           = 22.8[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```


$^{13}$C NMR (6da, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-30-13C_E13C-1-1.jdf
```

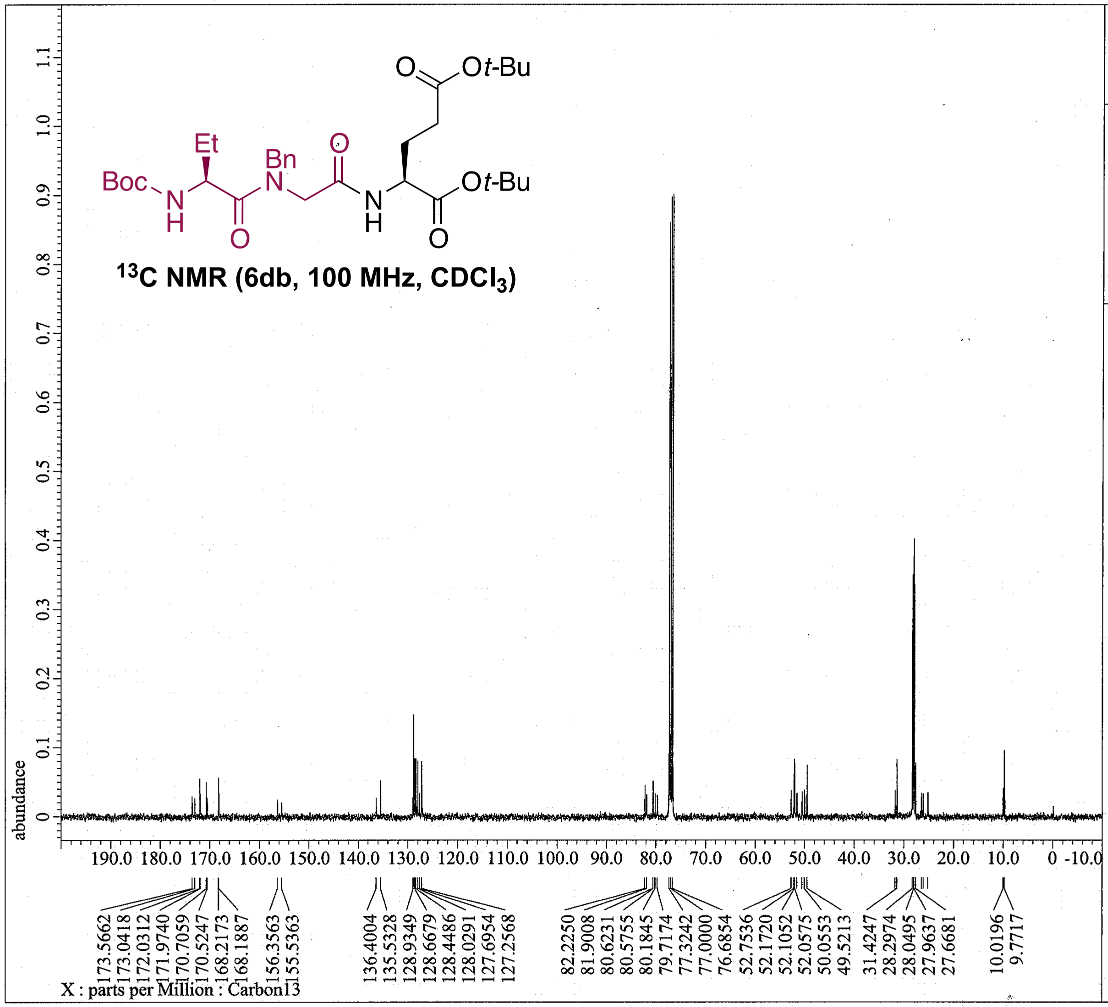
```
Filename          = wm12-30-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-30-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 28-JUN-2021 08:05:35
Revision_Time     = 28-JUN-2021 09:20:33
Current_Time      = 28-JUN-2021 09:21:19

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1521
Total_Scans       = 1521

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6db, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-139-6_E1H-1-1.jdf
```

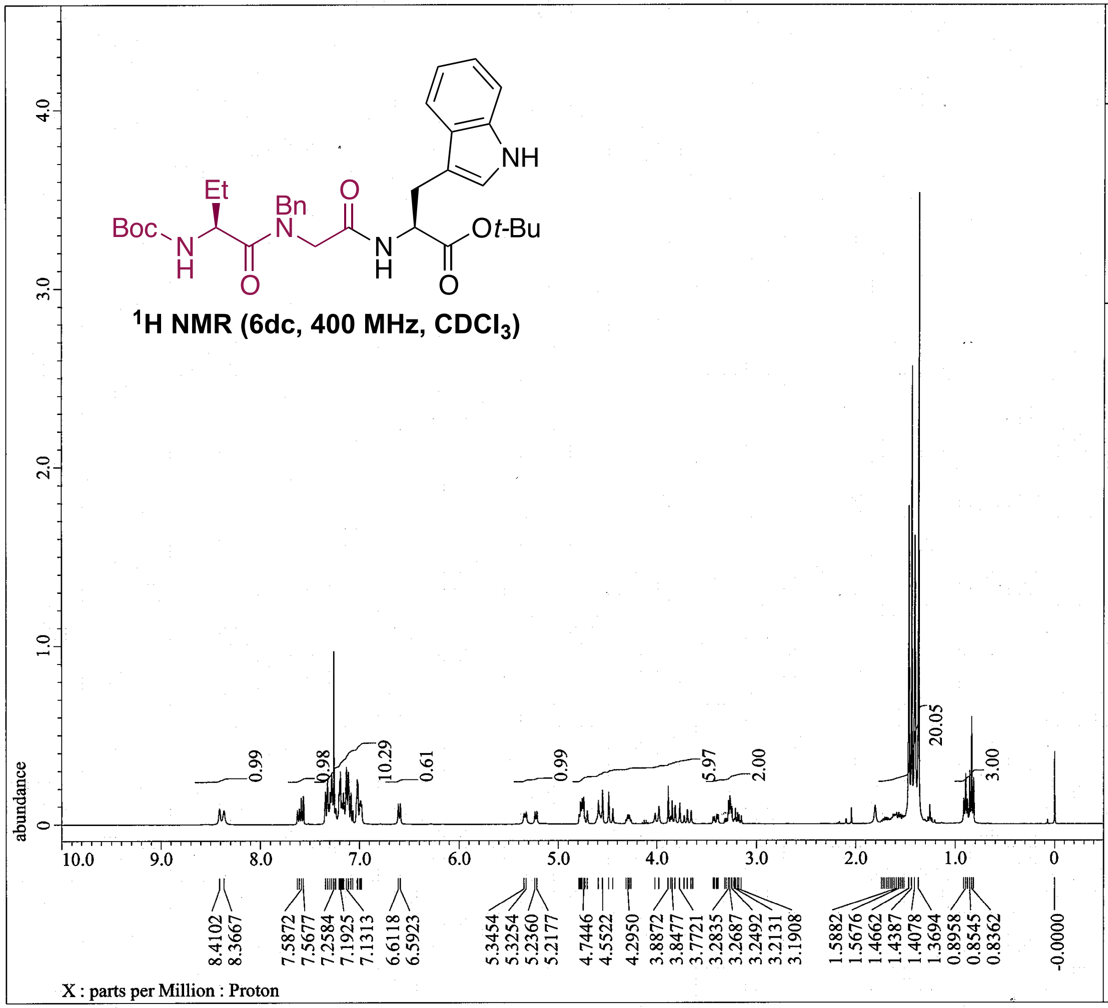
```
Filename            = wm12-139-6_E1H-1-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-139-6
Solvent             = CHLOROFORM-D
Creation_Time       = 29-OCT-2021 10:35:05
Revision_Time       = 29-OCT-2021 10:37:47
Current_Time        = 29-OCT-2021 10:38:00

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 28
Temp_Get            = 23.8[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (6db, 100 MHz, CDCl$_3$)

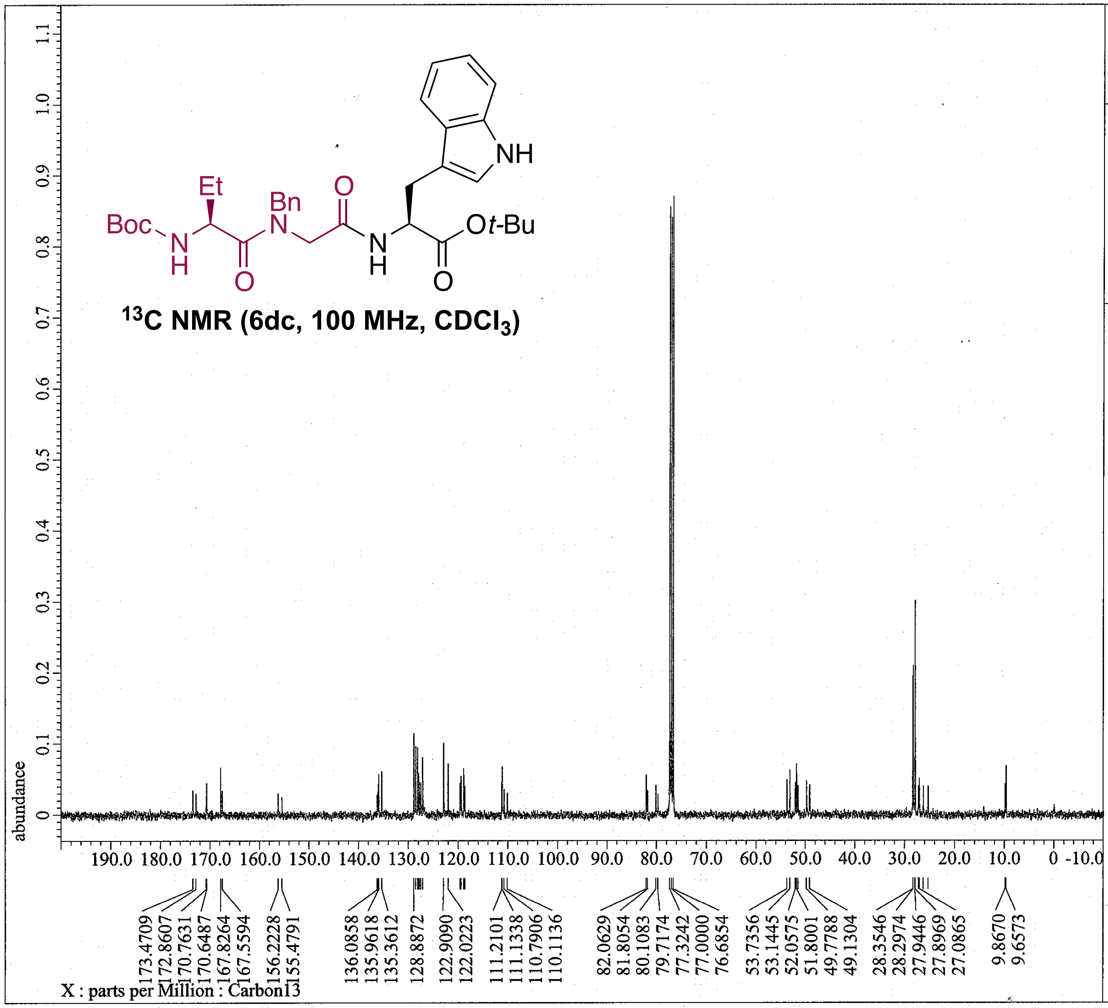
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-136-5-13C_E13C-1-1.jdf
```

```
Filename           = wm12-136-5-13C_E13C-1-4.jd
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-136-5-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 28-OCT-2021 13:26:48
Revision_Time      = 28-OCT-2021 14:18:29
Current_Time       = 28-OCT-2021 14:18:50

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = TRUE
Scans              = 1060
Total_Scans        = 1060

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.7[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (6dc, 400 MHz, CDCl$_3$)

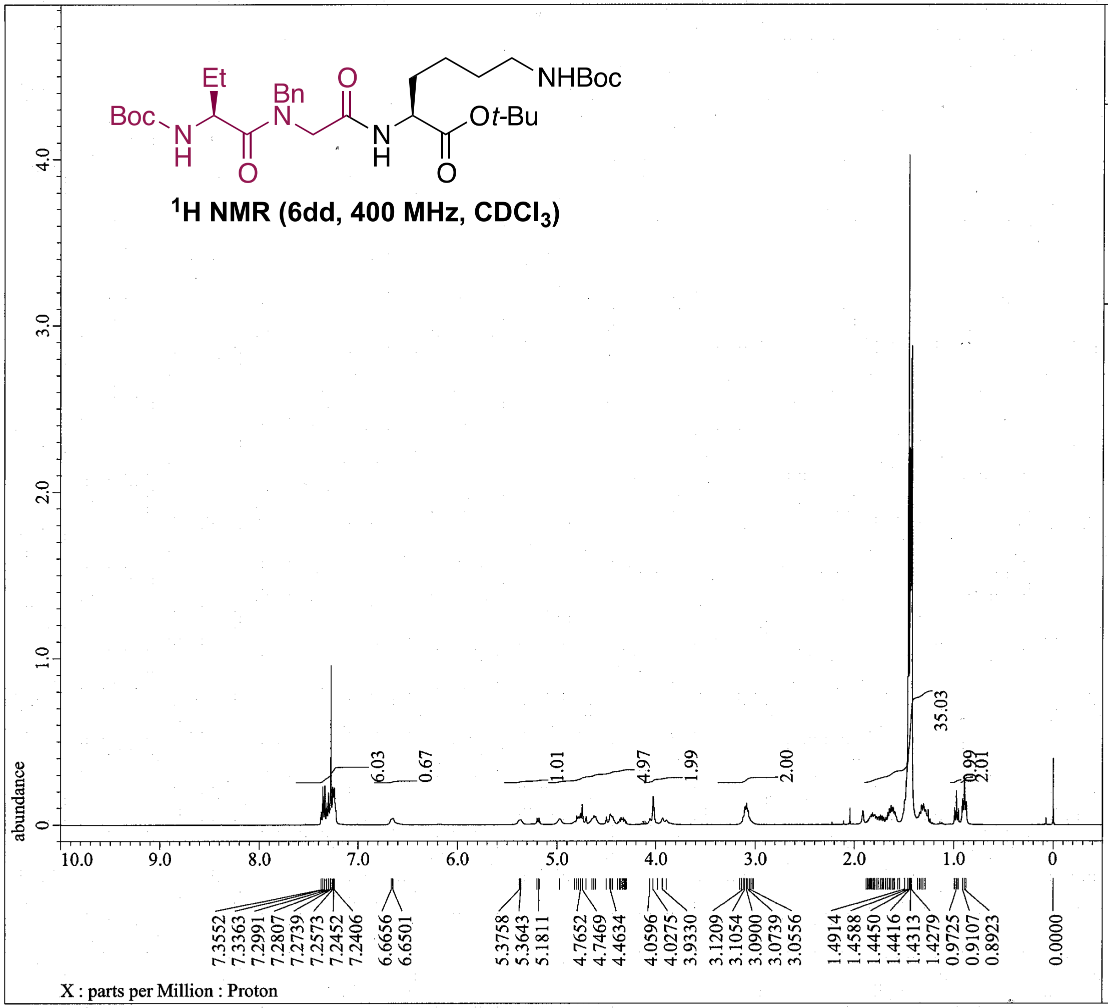
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-139-7_E1H-1-1.jdf

Filename          = wm12-139-7_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-139-7
Solvent           = CHLOROFORM-D
Creation_Time     = 29-OCT-2021 13:44:28
Revision_Time     = 29-OCT-2021 13:52:32
Current_Time      = 29-OCT-2021 13:52:47

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 24.1[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (6dc, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-139-3-13C_E13C-1-1.jdf
```

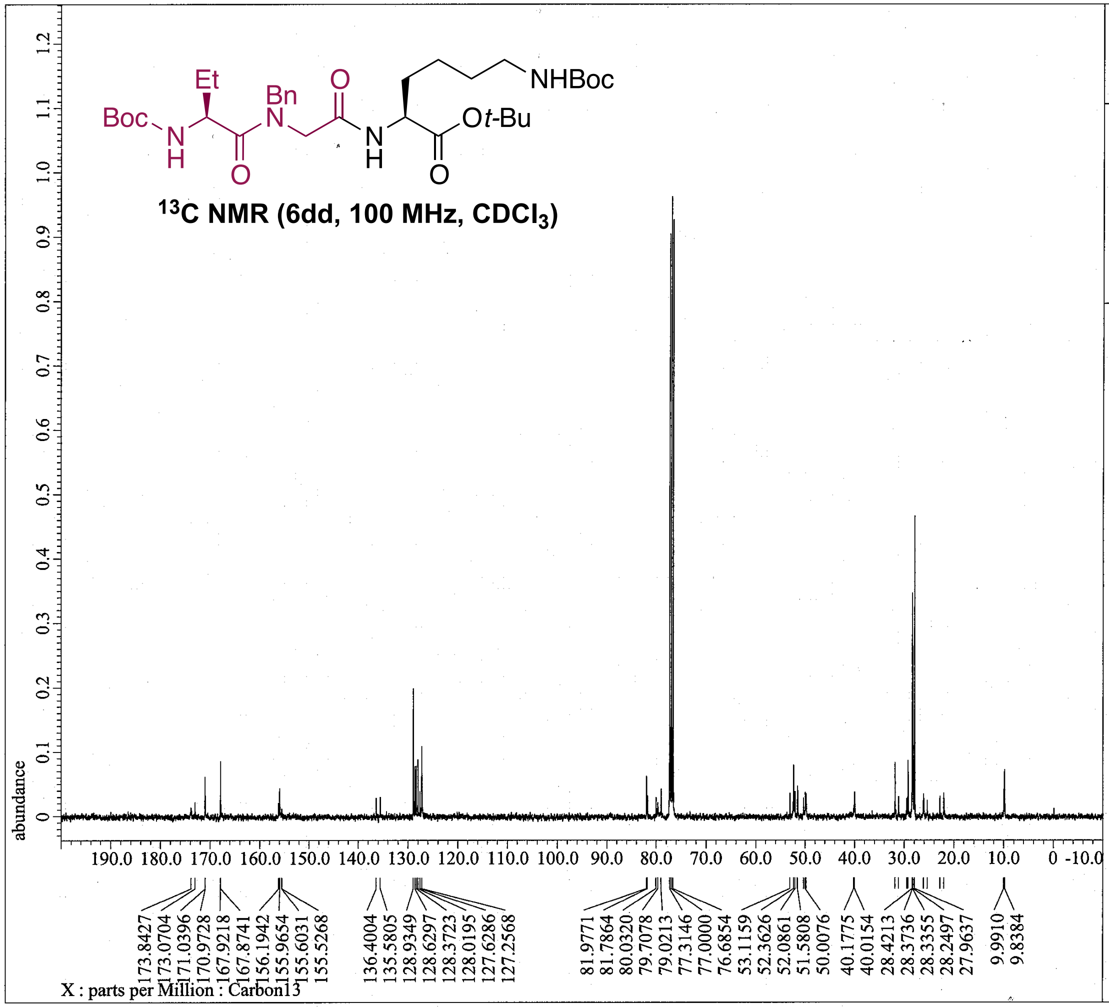
```
Filename            = wm12-139-3-13C_E13C-1-4.jd
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-139-3-13C
Solvent             = CHLOROFORM-D
Creation_Time       = 28-OCT-2021 15:55:15
Revision_Time       = 28-OCT-2021 16:28:59
Current_Time        = 28-OCT-2021 16:29:48

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = TRUE
Scans               = 720
Total_Scans         = 720

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 23.6[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (6dd, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-139-9_E1H-1-1.jdf
```

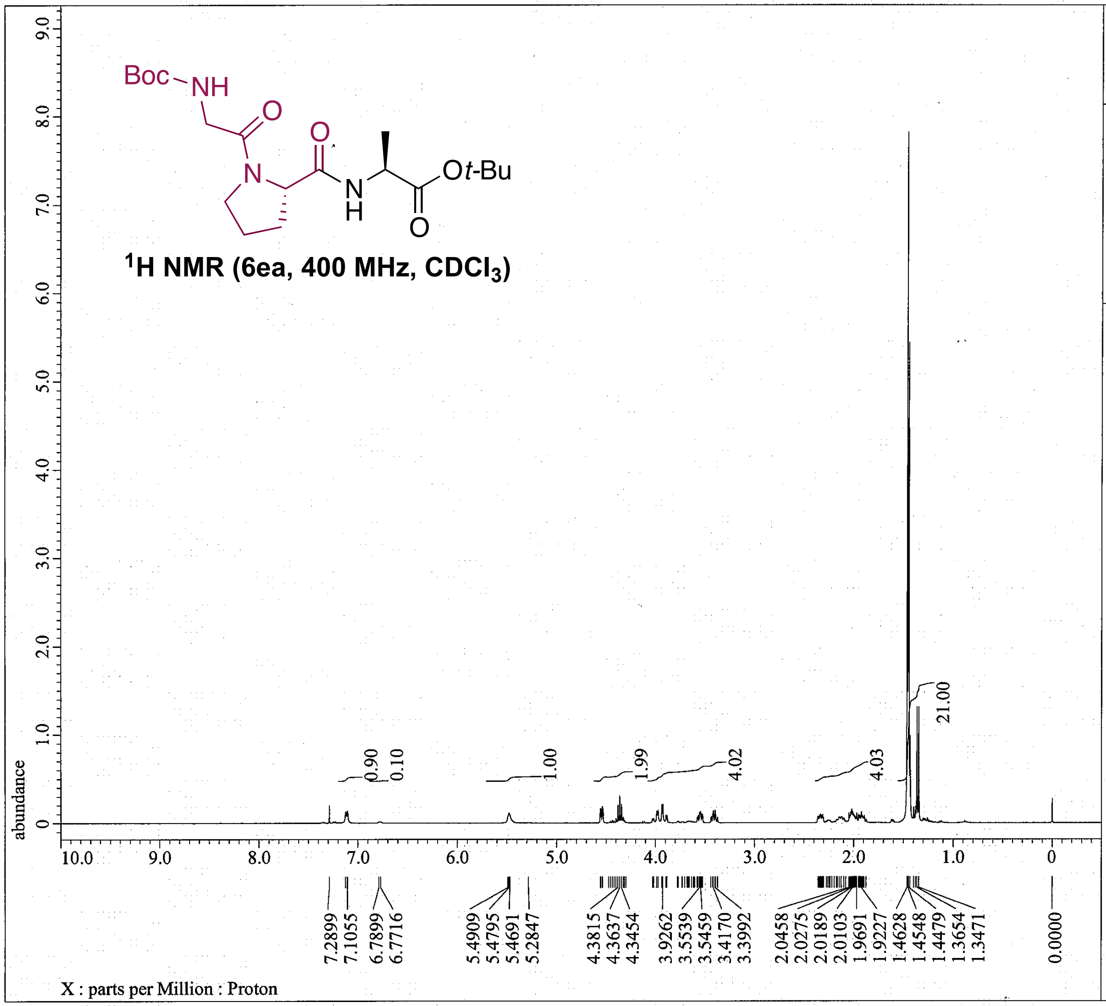
```
Filename           = wm12-139-9_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-139-9
Solvent            = CHLOROFORM-D
Creation_Time      = 30-OCT-2021 10:15:38
Revision_Time      = 30-OCT-2021 10:28:48
Current_Time       = 30-OCT-2021 10:29:01

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 24.8[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (6dd, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-139-9-13C_E13C-1-1.jdf
```

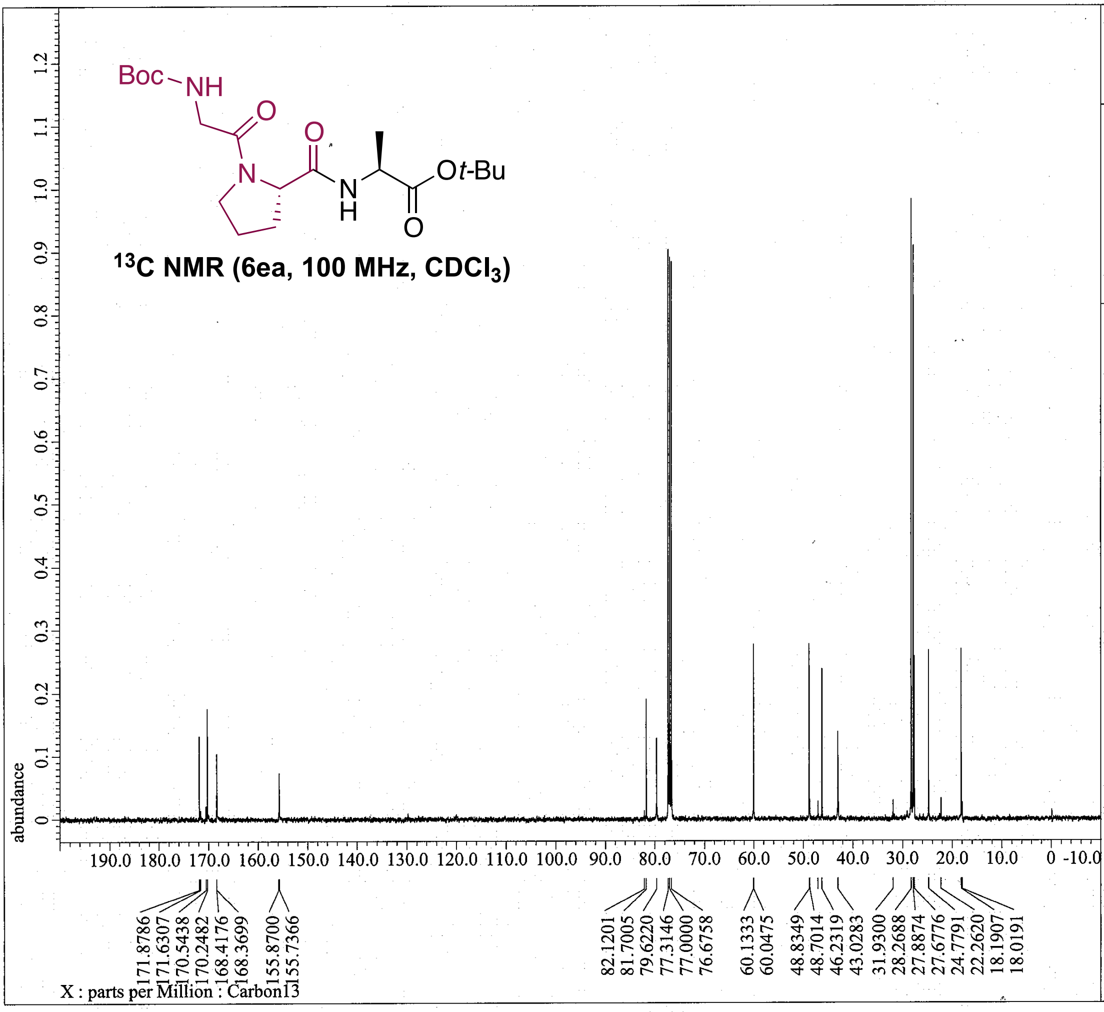
```
Filename          = wm12-139-9-13C_E13C-1-4.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-139-9-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 30-OCT-2021 10:17:44
Revision_Time     = 30-OCT-2021 11:07:42
Current_Time      = 30-OCT-2021 11:08:37

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6ea, 400 MHz, CDCl$_3$)

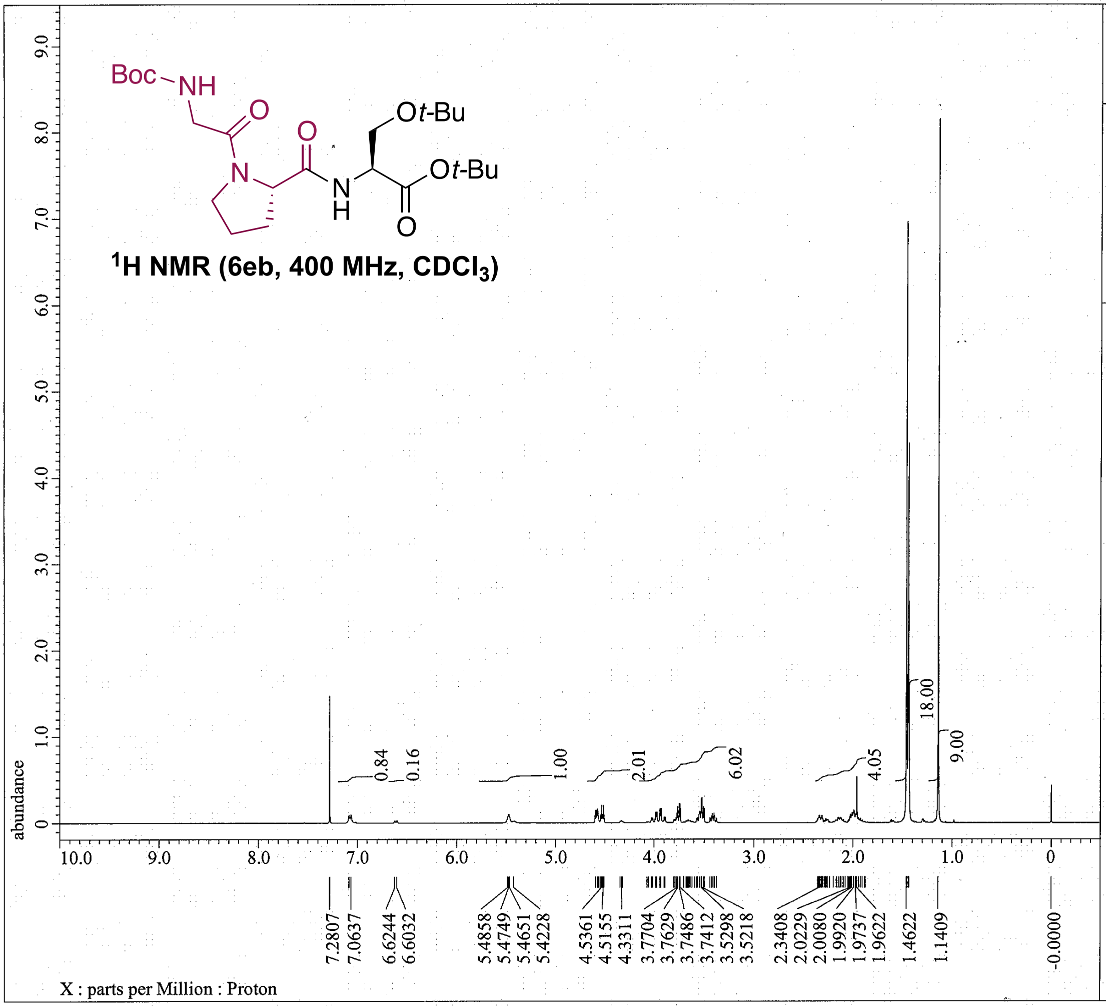
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-19_E1H-1-1.jdf
```

```
Filename          = wm12-19_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-19
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JUN-2021 08:02:30
Revision_Time     = 21-JUN-2021 08:11:52
Current_Time      = 21-JUN-2021 08:12:16

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 24
Temp_Get          = 22.8[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



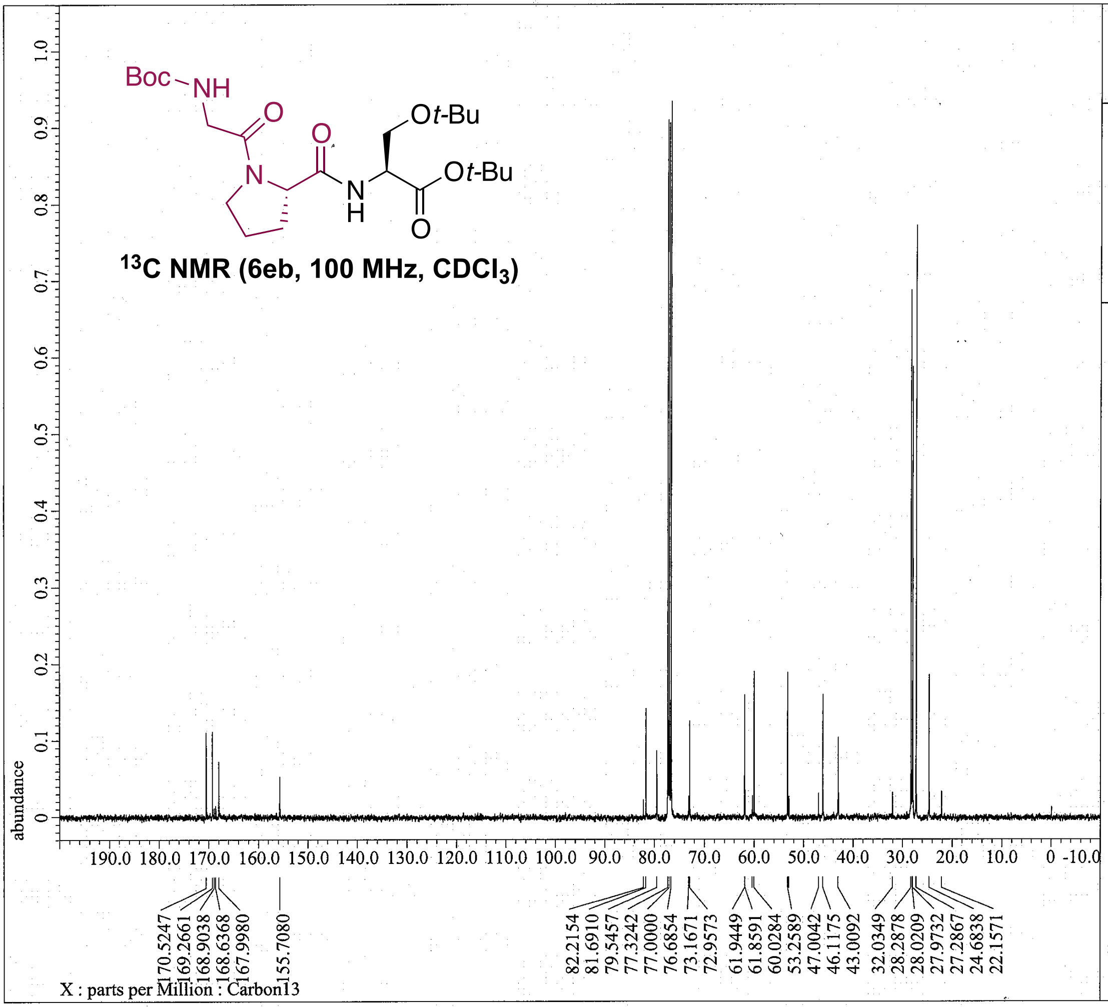
$^{13}$C NMR (6ea, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-19-13C_E13C-1-1.jdf

Filename           = wm12-19-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-19-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 21-JUN-2021 08:07:01
Revision_Time      = 21-JUN-2021 09:28:07
Current_Time       = 21-JUN-2021 09:28:30

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1621
Total_Scans        = 1621

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.8[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```


$^1$H NMR (6eb, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-24-2_E1H-1-1.jdf
```

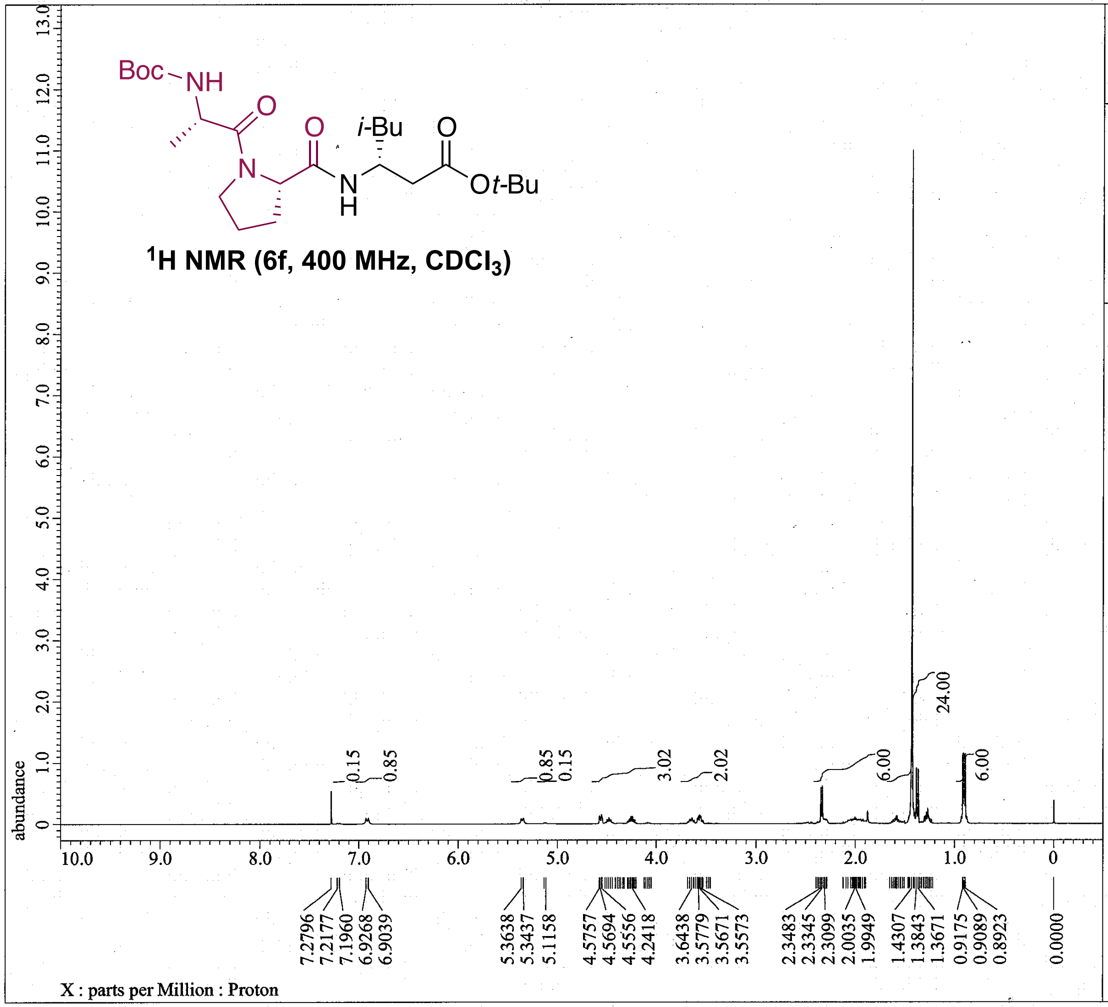
```
Filename           = wm12-24-2_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-24-2
Solvent            = CHLOROFORM-D
Creation_Time      = 23-JUN-2021 08:20:02
Revision_Time      = 23-JUN-2021 08:25:10
Current_Time       = 23-JUN-2021 08:25:23

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 22.9[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



## $^{13}$C NMR (6eb, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-24-2-C_E13C-1-1.jdf
```

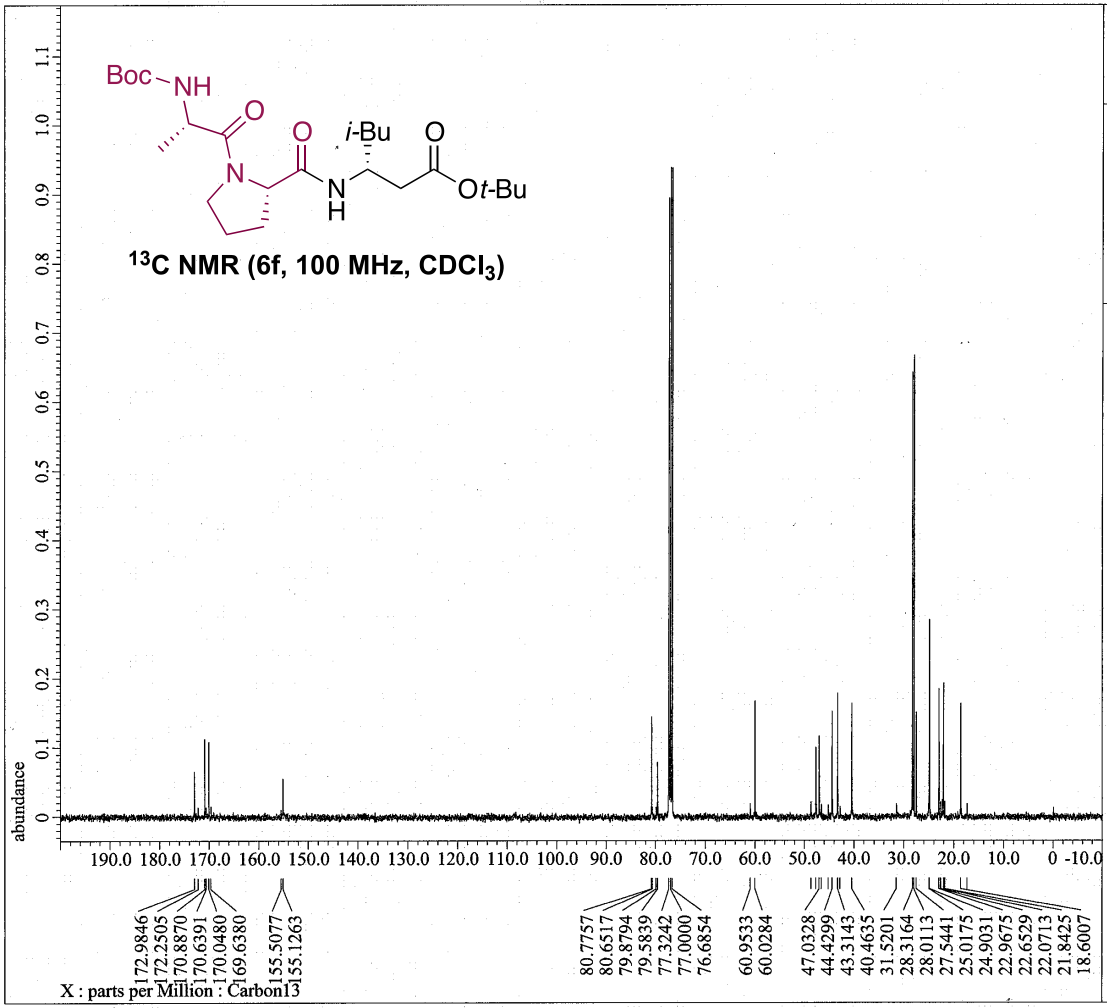
```
Filename           = wm12-24-2-C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-24-2-C
Solvent            = CHLOROFORM-D
Creation_Time      = 23-JUN-2021 08:24:15
Revision_Time      = 23-JUN-2021 09:37:20
Current_Time       = 23-JUN-2021 09:37:39

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1501
Total_Scans        = 1501

Relaxation_Delay = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.9[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^{1}$H NMR (6f, 400 MHz, CDCl$_3$)

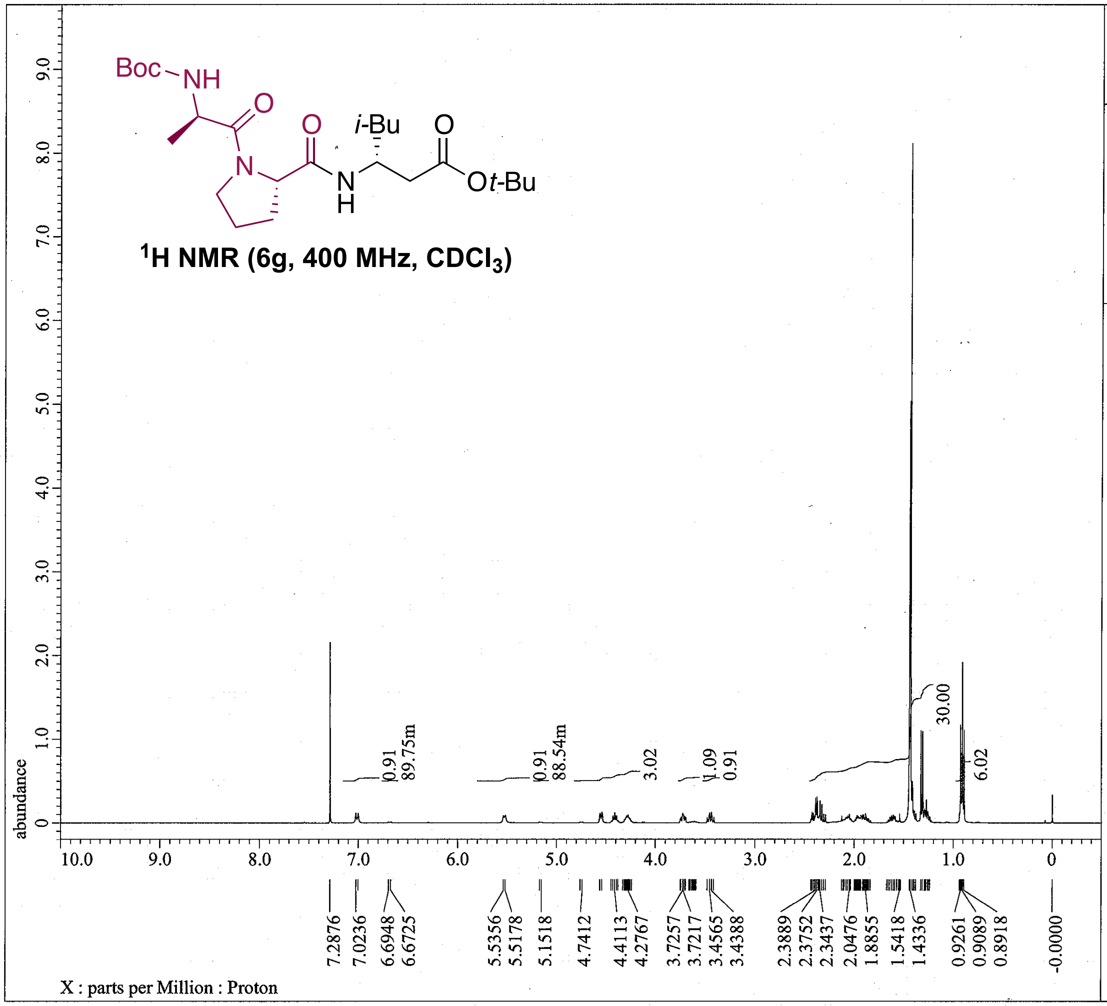
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-150_E1H-1-1.jdf
```

```
Filename             = wm12-150_E1H-1-4.jdf
Author               = delta
Experiment           = single_pulse.jxp
Sample_Id            = wm12-150
Solvent              = CHLOROFORM-D
Creation_Time        = 10-NOV-2021 13:58:15
Revision_Time        = 10-NOV-2021 15:35:27
Current_Time         = 10-NOV-2021 15:35:44

Data_Format          = 1D COMPLEX
Dim_Size             = 26214
Dim_Title            = Proton
Dim_Units            = [ppm]
Dimensions           = X
Site                 = JNM-ECS400
Spectrometer         = DELTA2_NMR

Field_Strength       = 9.389766[T] (400[MHz])
X_Acq_Duration       = 4.36731904[s]
X_Domain             = 1H
X_Freq               = 399.78219838[MHz]
X_Offset             = 5[ppm]
X_Points             = 32768
X_Prescans           = 1
X_Resolution         = 0.22897343[Hz]
X_Sweep              = 7.5030012[kHz]
X_Sweep_Clipped      = 6.00240096[kHz]
Irr_Domain           = Proton
Irr_Freq             = 399.78219838[MHz]
Irr_Offset           = 5[ppm]
Tri_Domain           = Proton
Tri_Freq             = 399.78219838[MHz]
Tri_Offset           = 5[ppm]
Clipped              = FALSE
Scans                = 8
Total_Scans          = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 26
Temp_Get         = 25[dC]
X_90_Width       = 6.47[us]
X_Acq_Time       = 4.36731904[s]
X_Angle          = 45[deg]
X_Atn            = 1[dB]
X_Pulse          = 3.235[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
```



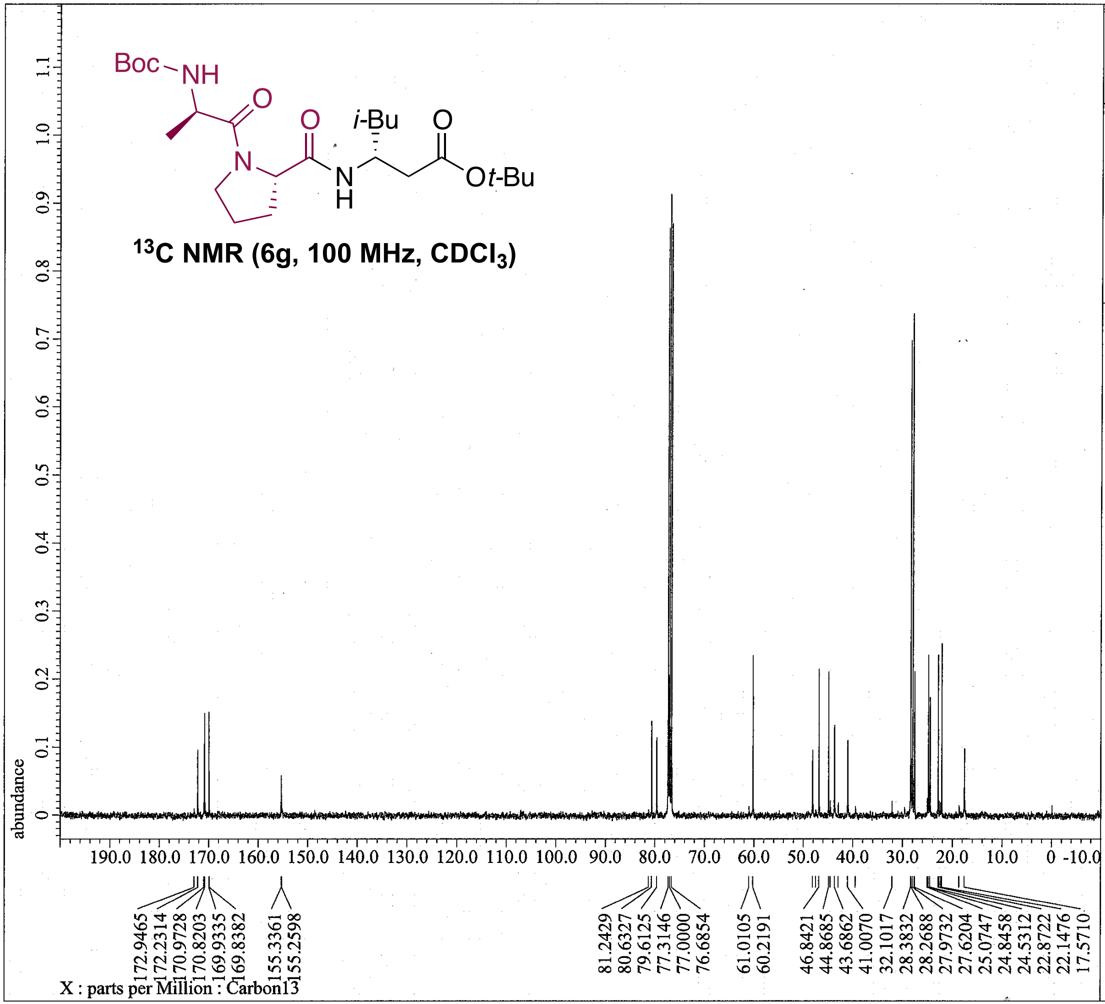
$^{13}$C NMR (6f, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-150-13C_E13C-1-1.jdf

Filename           = wm12-150-13C_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-150-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 10-NOV-2021 14:01:02
Revision_Time      = 10-NOV-2021 14:50:30
Current_Time       = 10-NOV-2021 14:51:37

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1024
Total_Scans        = 1024

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 24.7[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (6g, 400 MHz, CDCl$_3$)

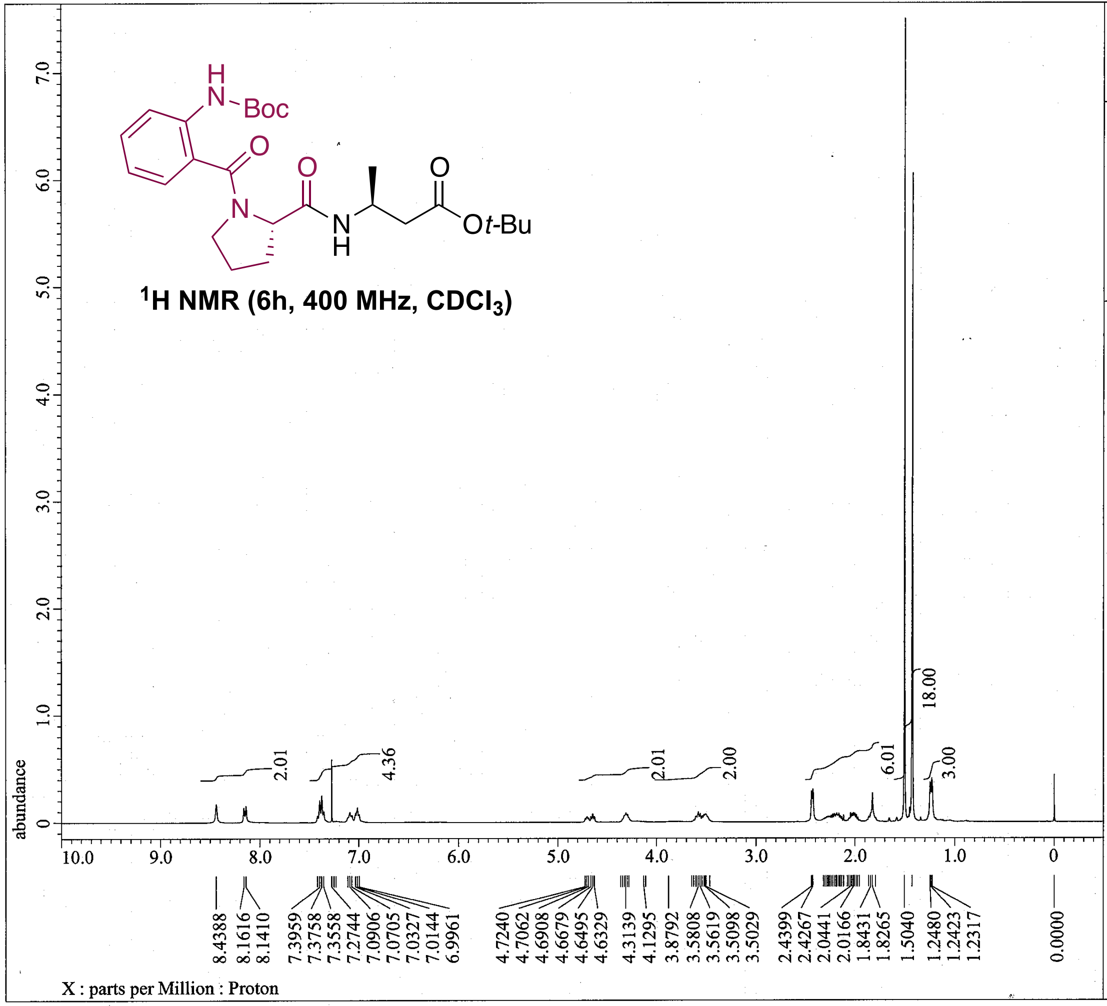
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-110-3_E1H-1-1.jdf

Filename            = wm12-110-3_E1H-1-5.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-110-3
Solvent             = CHLOROFORM-D
Creation_Time       =  2-OCT-2021 08:41:30
Revision_Time       =  2-OCT-2021 08:57:34
Current_Time        =  2-OCT-2021 08:57:46

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay = 5[s]
Recvr_Gain          = 24
Temp_Get            = 22.2[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (6g, 100 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-110-3-13C_E13C_copy3-1-1.j
```

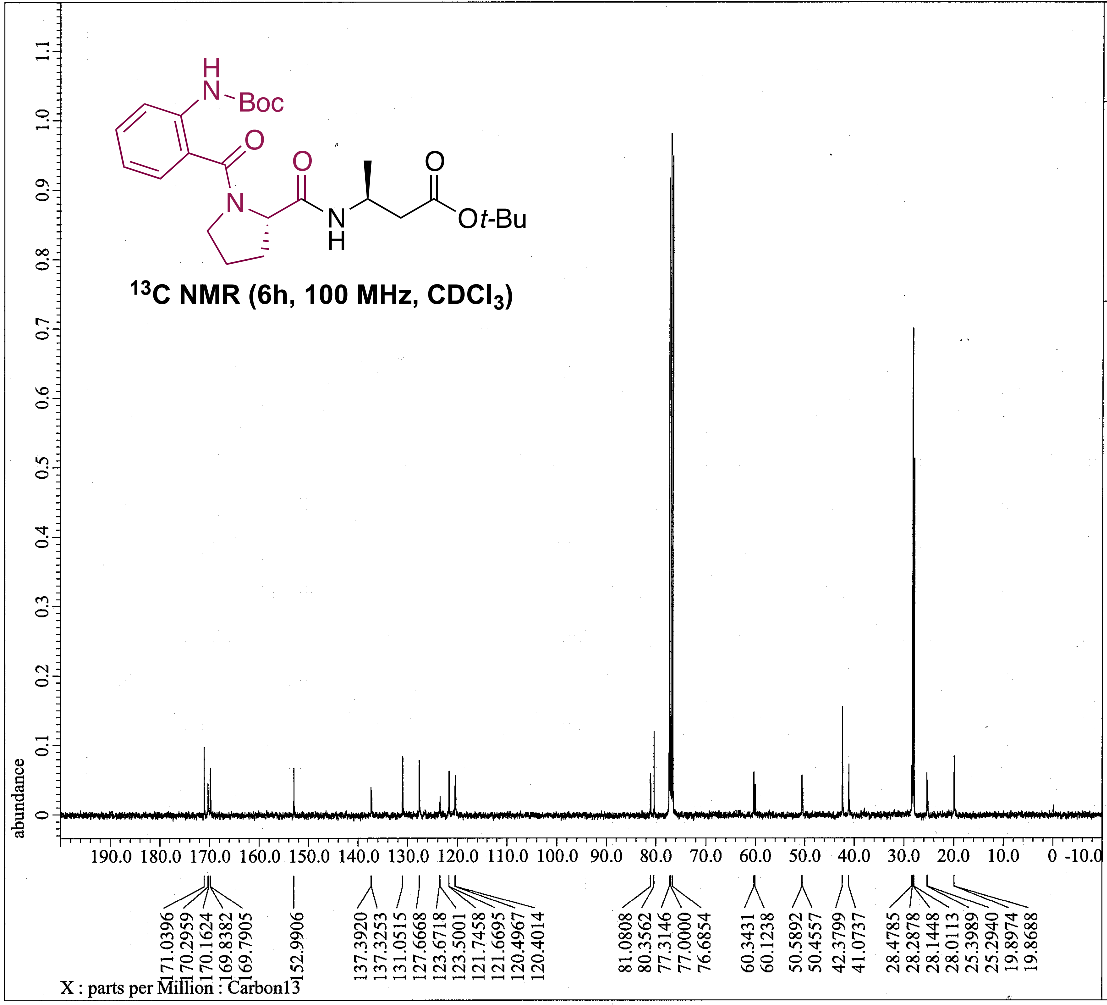
```
Filename           = wm12-110-3-13C_E13C_copy3-
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-110-3-13C
Solvent            = CHLOROFORM-D
Creation_Time      =  2-OCT-2021 08:43:37
Revision_Time      =  2-OCT-2021 09:34:09
Current_Time       =  2-OCT-2021 09:37:30

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 0[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Incomplete_Copy    = TRUE
Scans              = 1024
Total_Scans        = 1024

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 24[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
```



$^1$H NMR (6h, 400 MHz, $CDCl_3$)

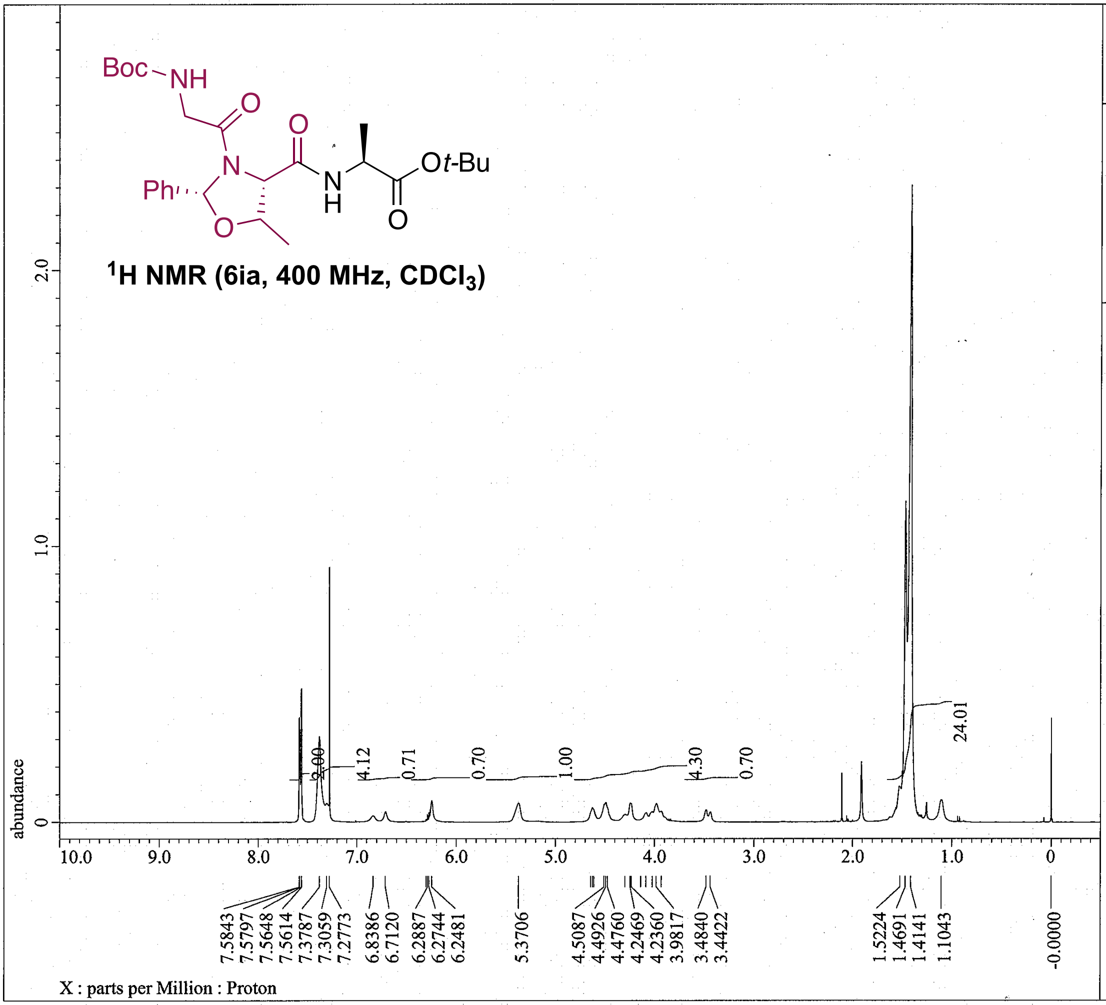
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-156-r.t._E1H-1-1.jdf
```

```
Filename          = wm12-156-r.t._E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-156-r.t.
Solvent           = CHLOROFORM-D
Creation_Time     = 18-NOV-2021 08:12:25
Revision_Time     = 18-NOV-2021 08:52:40
Current_Time      = 18-NOV-2021 08:52:48

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 24.4[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```


$^{13}$C NMR (6h, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-156-r.t.-13C_E13C-1-1.jdf
```

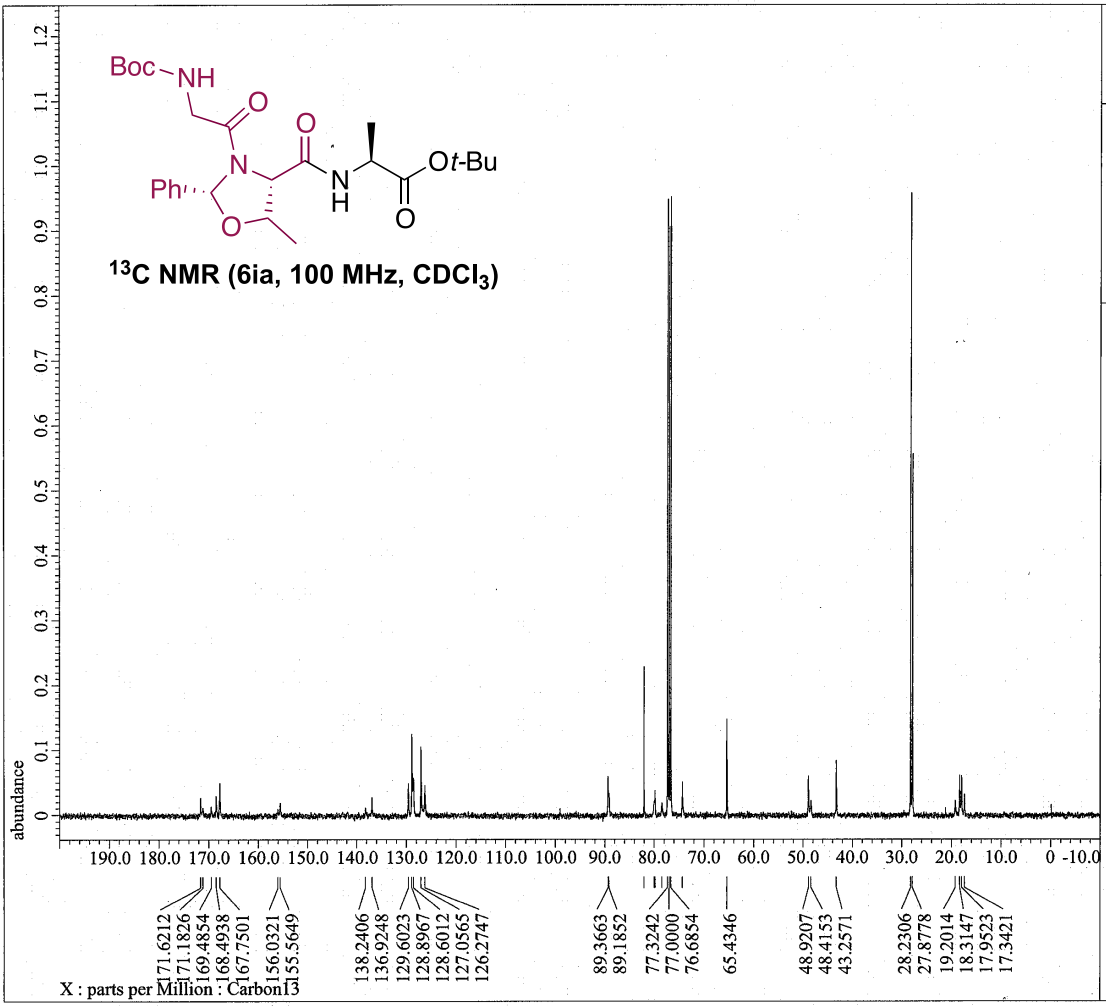
```
Filename          = wm12-156-r.t.-13C_E13C-1-4
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-156-r.t.-13C
Solvent           = CHLOROFORM-D
Creation_Time     = 18-NOV-2021 08:14:33
Revision_Time     = 18-NOV-2021 09:04:00
Current_Time      = 18-NOV-2021 09:04:16

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (6ia, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-92-2-H_E1H-1-1.jdf
```

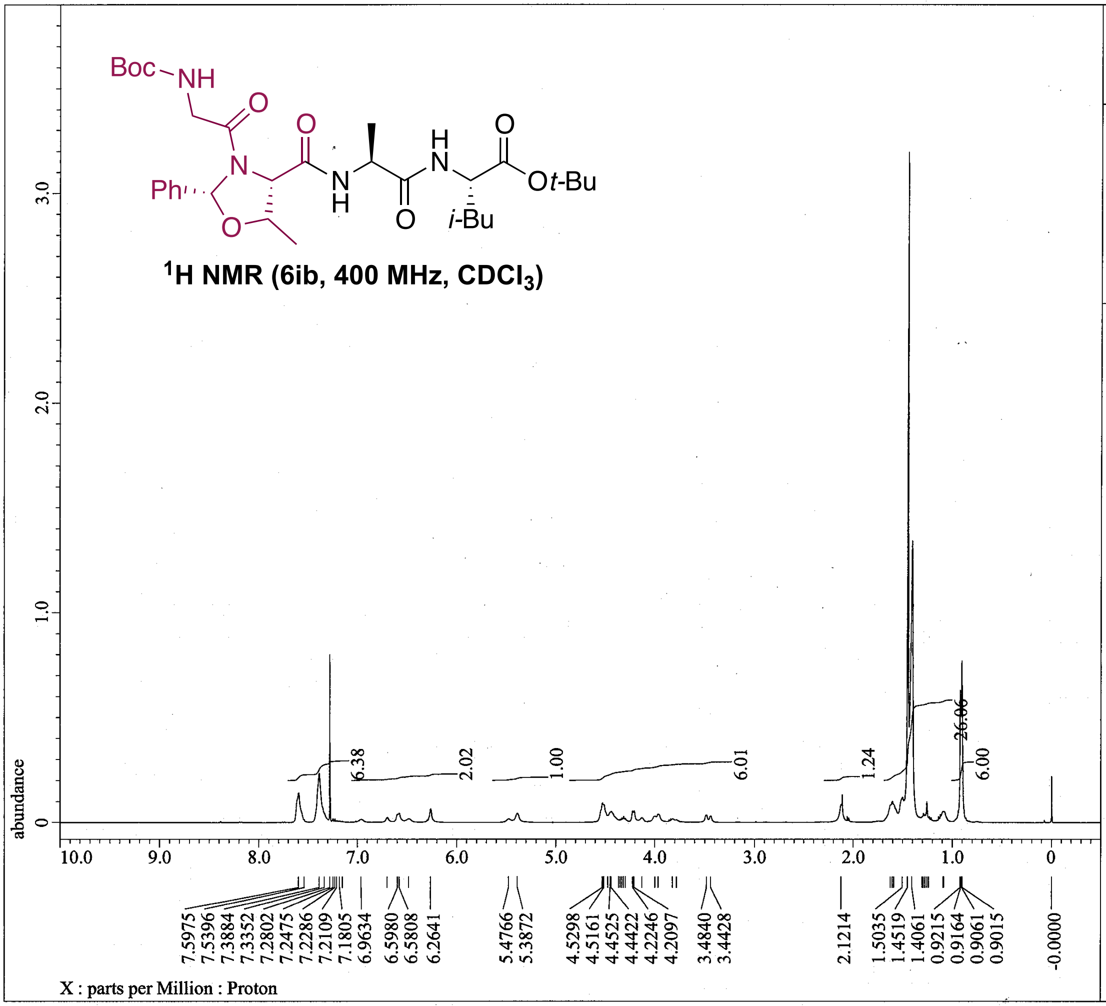
```
Filename            = wm12-92-2-H_E1H-1-5.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-92-2-H
Solvent             = CHLOROFORM-D
Creation_Time       = 12-SEP-2021 13:02:31
Revision_Time       = 12-SEP-2021 13:06:18
Current_Time        = 12-SEP-2021 13:06:29

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 24
Temp_Get            = 24[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (6ia, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-92-2_E13C_copy2-1-1.jdf
```

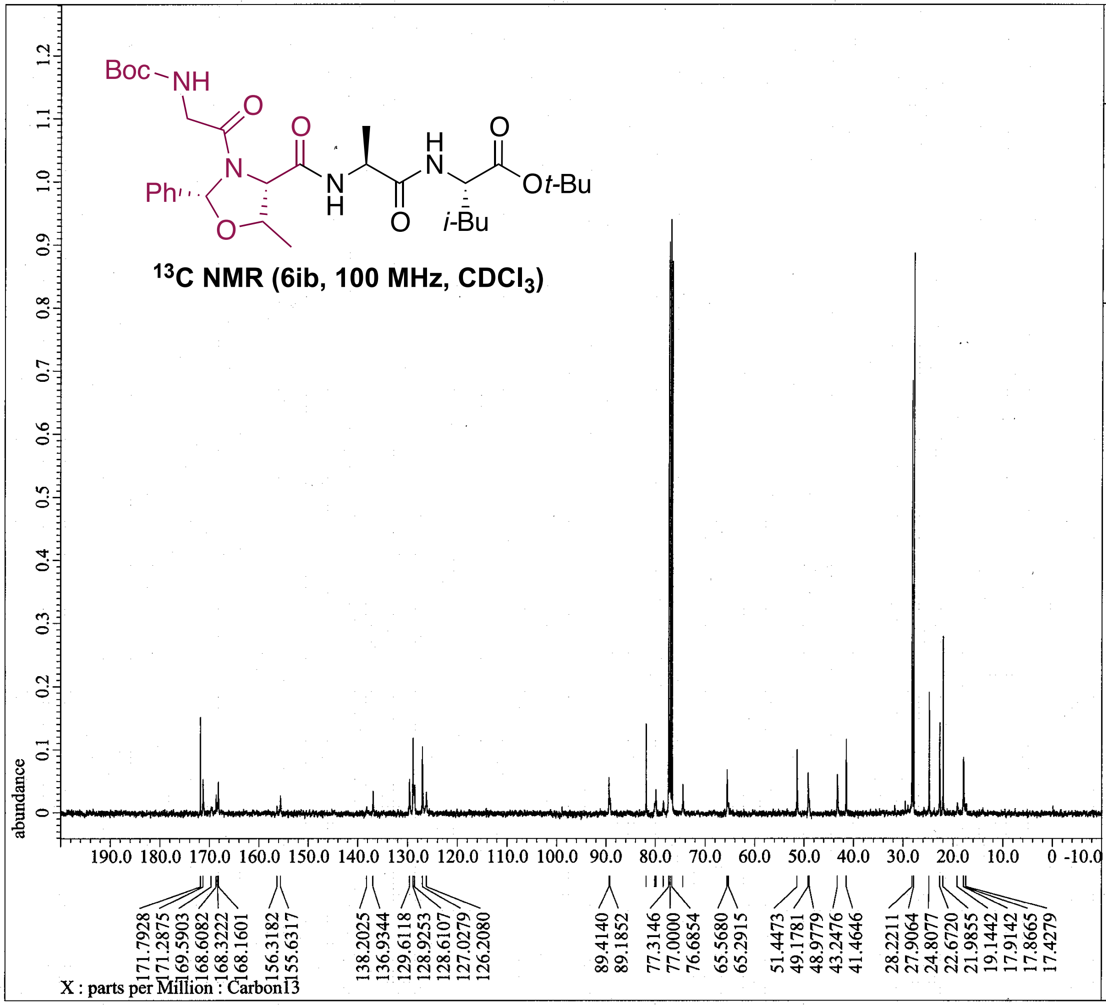
```
Filename            = wm12-92-2_E13C_copy2-1-4.j
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm12-92-2
Solvent             = CHLOROFORM-D
Creation_Time       = 12-SEP-2021 11:13:55
Revision_Time       = 12-SEP-2021 12:20:31
Current_Time        = 12-SEP-2021 12:20:53

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 0[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Incomplete_Copy     = TRUE
Scans               = 1366
Total_Scans         = 1366

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 24[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
```



$^1$H NMR (6ib, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-104_E1H-1-1.jdf
```

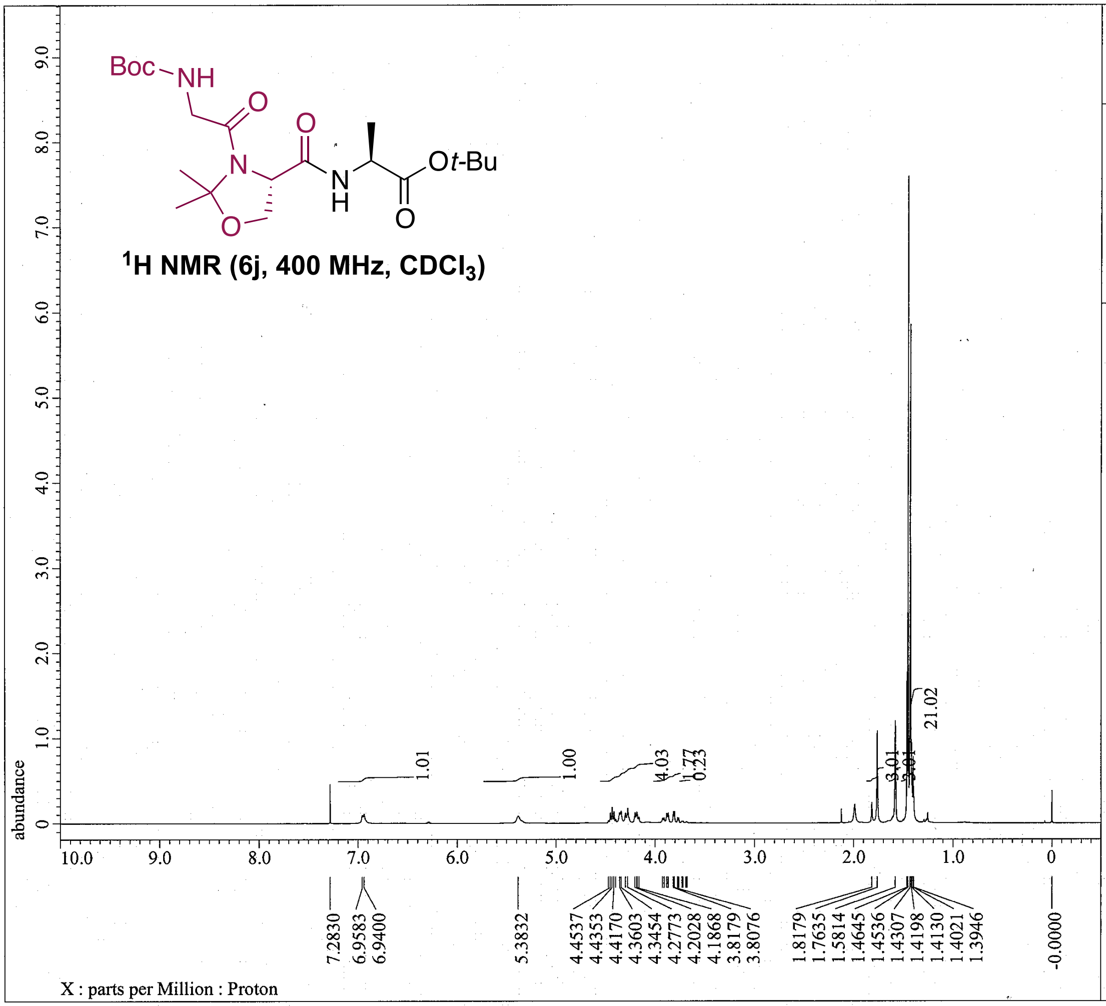
```
Filename          = wm12-104_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-104
Solvent           = CHLOROFORM-D
Creation_Time     = 27-SEP-2021 08:54:19
Revision_Time     = 27-SEP-2021 09:42:32
Current_Time      = 27-SEP-2021 09:42:45

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 22
Temp_Get          = 23.6[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}C$ NMR (6ib, 100 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-104_E13C-1-1.jdf
```

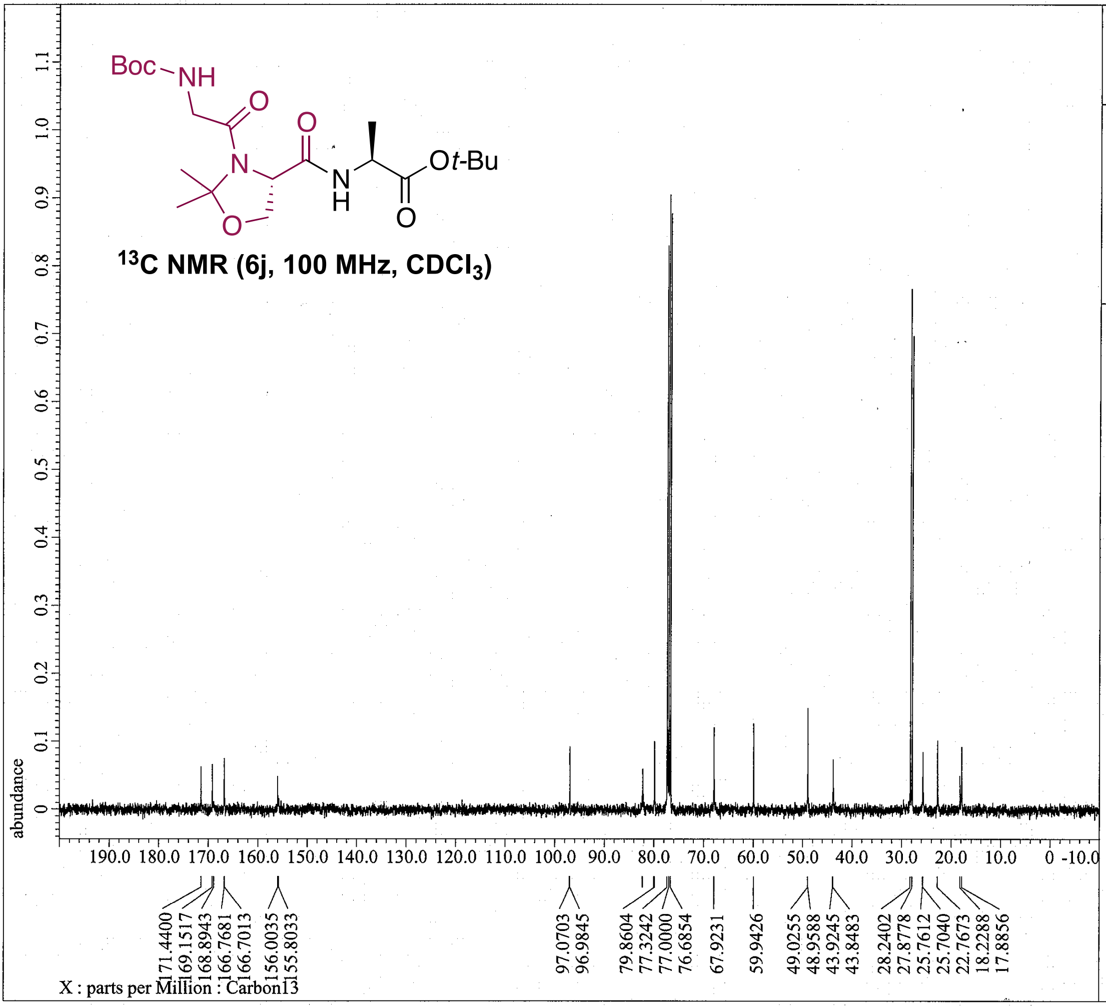
```
Filename           = wm12-104_E13C-1-4.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-104
Solvent            = CHLOROFORM-D
Creation_Time      = 27-SEP-2021 07:58:59
Revision_Time      = 27-SEP-2021 08:53:14
Current_Time       = 27-SEP-2021 08:53:32

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1024
Total_Scans        = 1024

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.7[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (6j, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-122-3_E1H-1-1.jdf
```

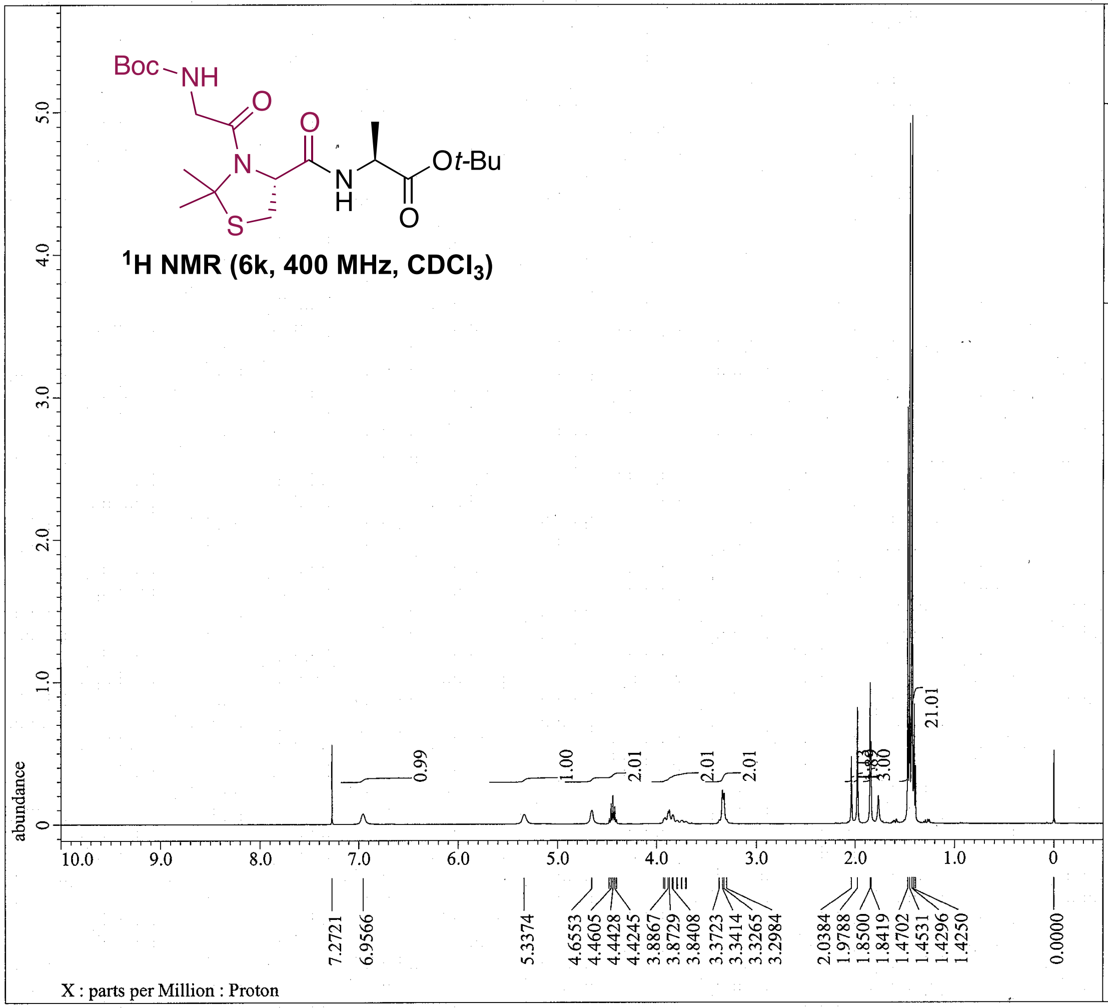
```
Filename           = wm12-122-3_E1H-1-4.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-122-3
Solvent            = CHLOROFORM-D
Creation_Time      = 13-OCT-2021 08:25:17
Revision_Time      = 13-OCT-2021 08:33:33
Current_Time       = 13-OCT-2021 08:33:46

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 26
Temp_Get           = 23.5[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (6j, 100 MHz, CDCl$_3$)

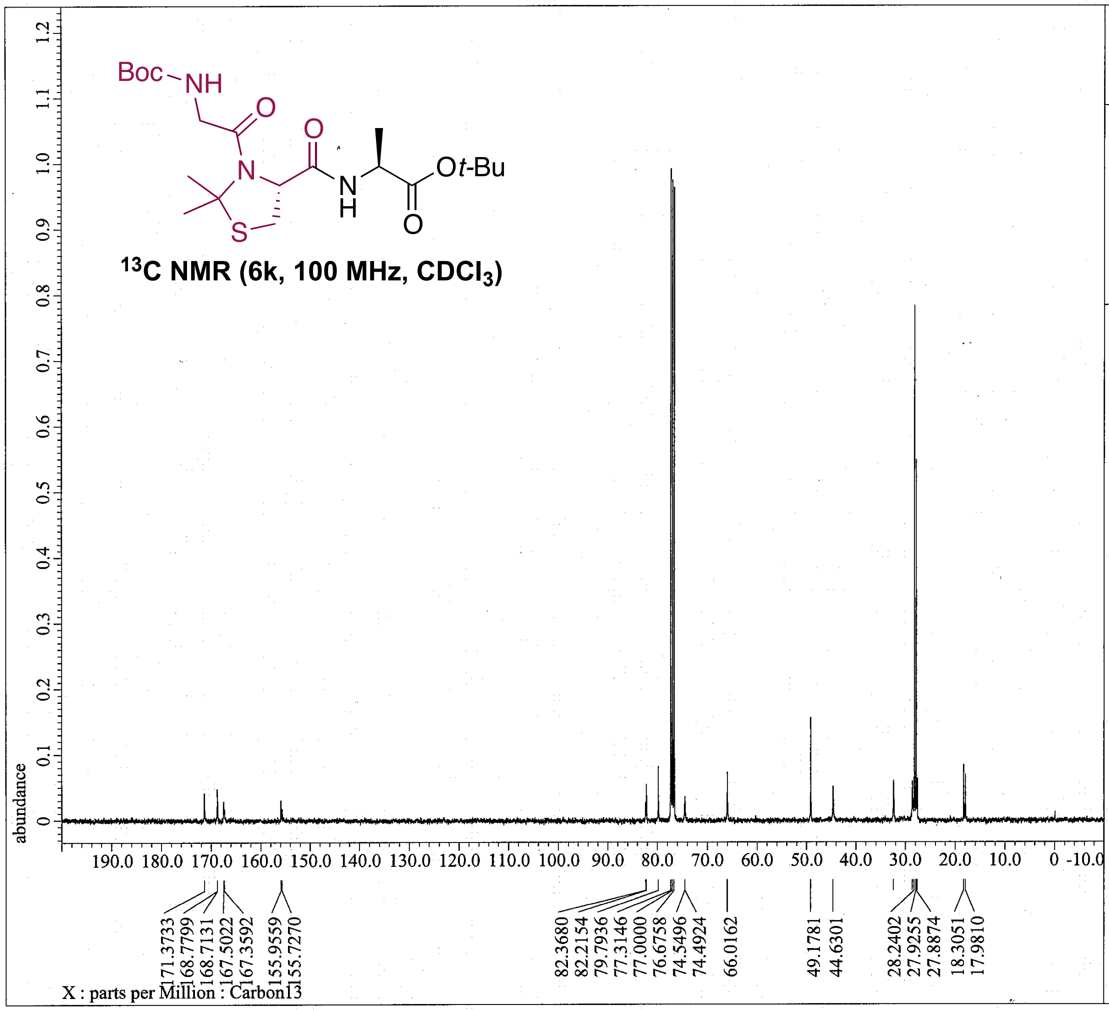
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-122-3-13C_E13C_copy1-1-1.j
```

```
Filename           = wm12-122-3-13C_E13C_copy1-
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm12-122-3-13C
Solvent            = CHLOROFORM-D
Creation_Time      = 13-OCT-2021 08:27:28
Revision_Time      = 13-OCT-2021 08:39:42
Current_Time       = 13-OCT-2021 08:40:40

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 0[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Incomplete_Copy    = TRUE
Scans              = 292
Total_Scans        = 292

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 23.8[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
```



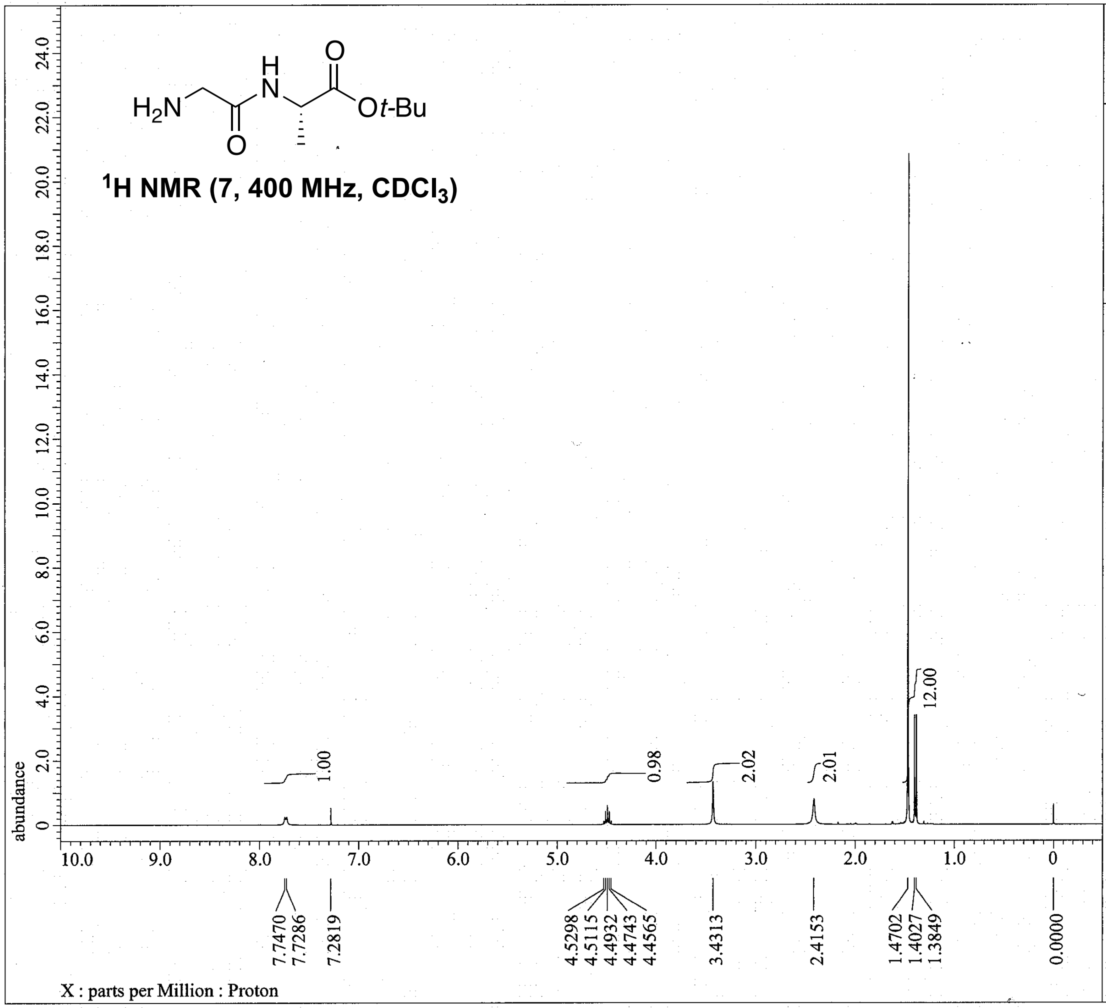
$^1$H NMR (6k, 400 MHz, CDCl$_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-166_E1H-1-1.jdf

Filename           = wm12-166_E1H-1-5.jdf
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = wm12-166
Solvent            = CHLOROFORM-D
Creation_Time      =  7-DEC-2021 15:08:01
Revision_Time      =  7-DEC-2021 15:14:18
Current_Time       =  7-DEC-2021 15:14:31

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 4.36731904[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 32768
X_Prescans         = 1
X_Resolution       = 0.22897343[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 30
Temp_Get           = 23.5[dC]
X_90_Width         = 6.47[us]
X_Acq_Time         = 4.36731904[s]
X_Angle            = 45[deg]
X_Atn              = 1[dB]
X_Pulse            = 3.235[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
```



$^{13}$C NMR (6k, 100 MHz, CDCl$_3$)

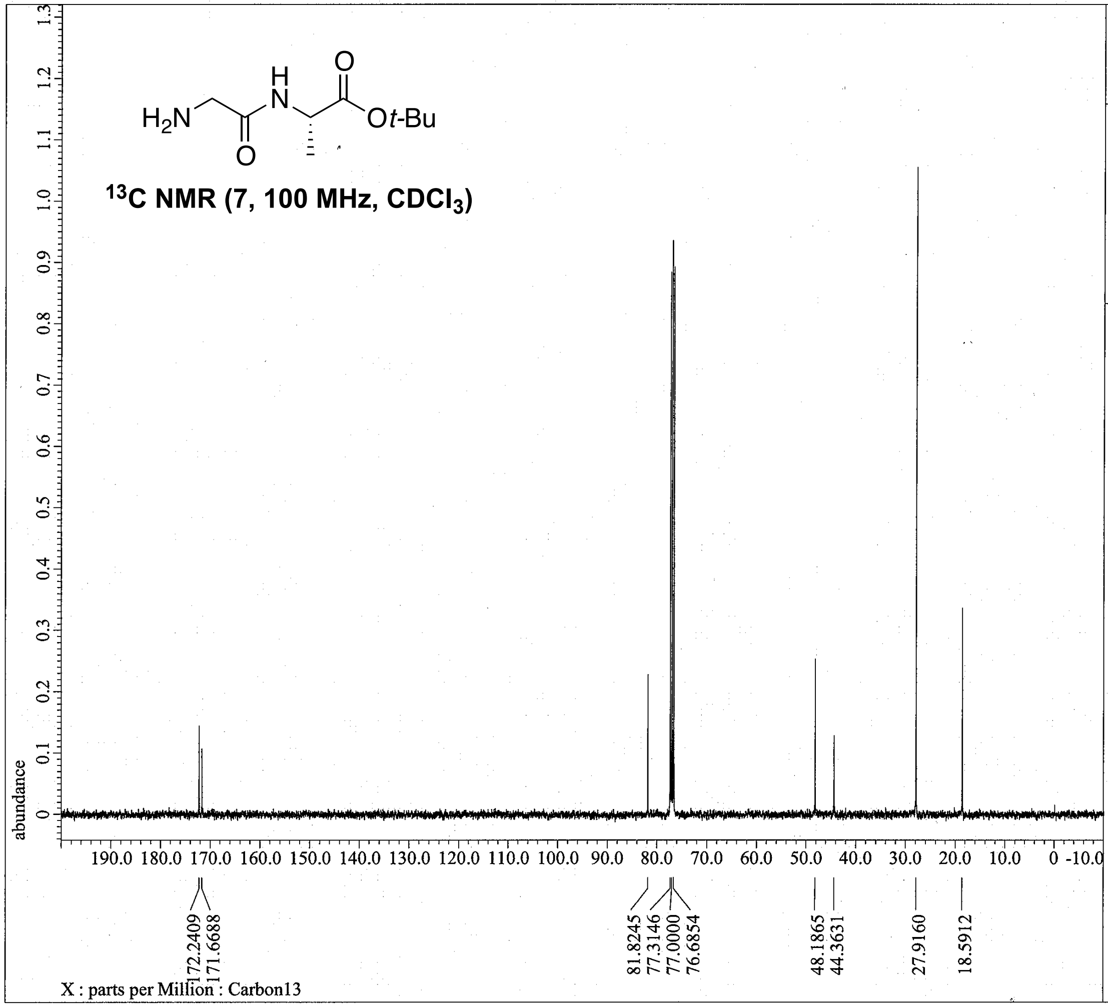
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-166-3-13C_E13C-1-1.jdf

Filename          = wm12-166-3-13C_E13C-1-4.jd
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-166-3-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  4-DEC-2021 08:55:13
Revision_Time     =  4-DEC-2021 10:14:03
Current_Time      =  4-DEC-2021 10:14:22

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1601
Total_Scans       = 1601

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (7, 400 MHz, $CDCl_3$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-172_E1H-1-1.jdf
```

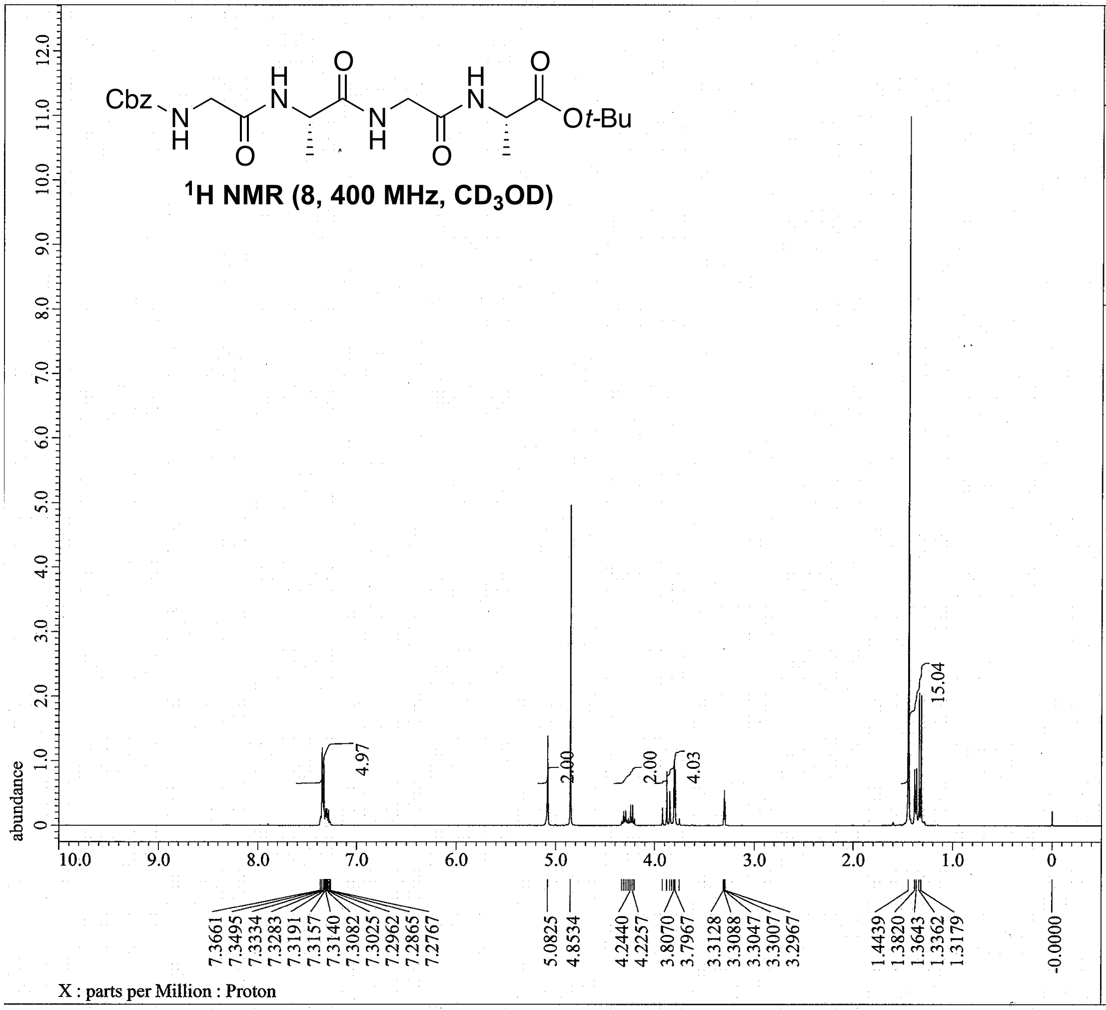
```
Filename          = wm12-172_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm12-172
Solvent           = CHLOROFORM-D
Creation_Time     =  6-DEC-2021 09:56:21
Revision_Time     =  6-DEC-2021 09:56:08
Current_Time      =  6-DEC-2021 09:56:22

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 24.8[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}C$ NMR (7, 100 MHz, $CDCl_3$)

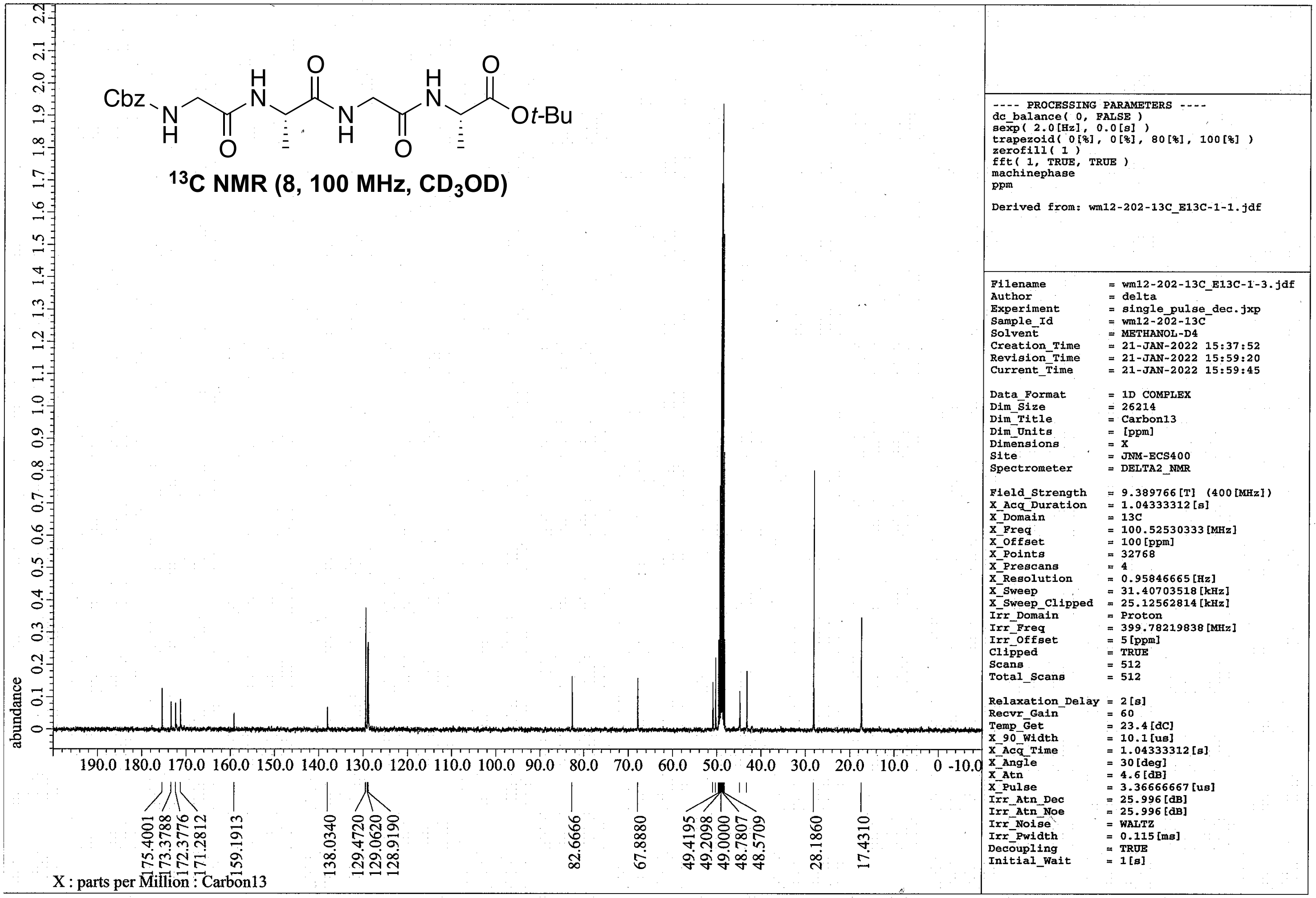
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-172-13C_E13C-1-1.jdf
```

```
Filename          = wm12-172-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-172-13C
Solvent           = CHLOROFORM-D
Creation_Time     =  6-DEC-2021 09:58:29
Revision_Time     =  6-DEC-2021 10:20:09
Current_Time      =  6-DEC-2021 10:20:24

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.8[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-202_E1H-1-1.jdf
```

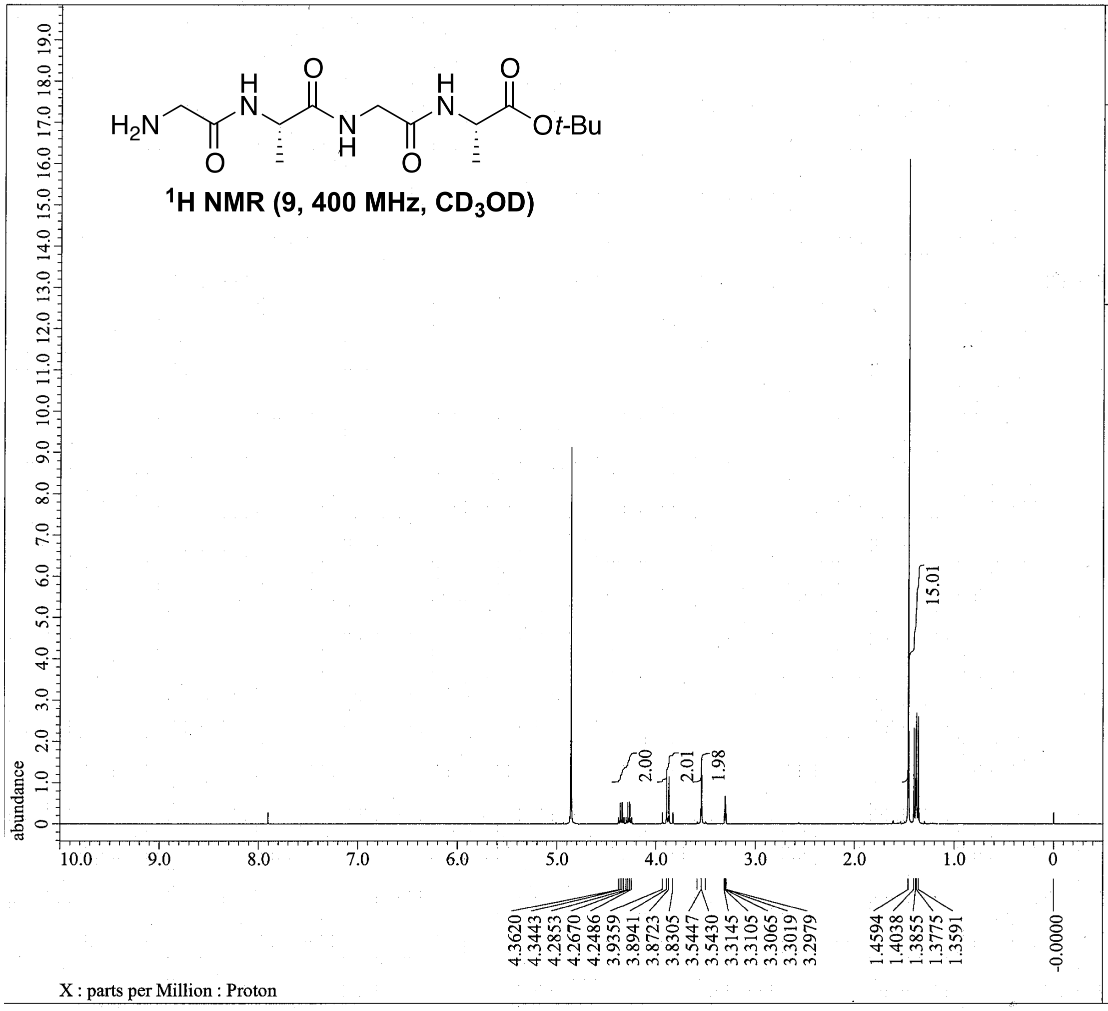
```
Filename            = wm12-202_E1H-1-5.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = wm12-202
Solvent             = METHANOL-D4
Creation_Time       = 21-JAN-2022 15:33:38
Revision_Time       = 21-JAN-2022 15:37:02
Current_Time        = 21-JAN-2022 15:37:17

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 4.36731904[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 32768
X_Prescans          = 1
X_Resolution        = 0.22897343[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 28
Temp_Get            = 22.9[dC]
X_90_Width          = 6.47[us]
X_Acq_Time          = 4.36731904[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 3.235[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
```



$^{13}$C NMR (8, 100 MHz, CD$_3$OD)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm12-202-13C_E13C-1-1.jdf
```

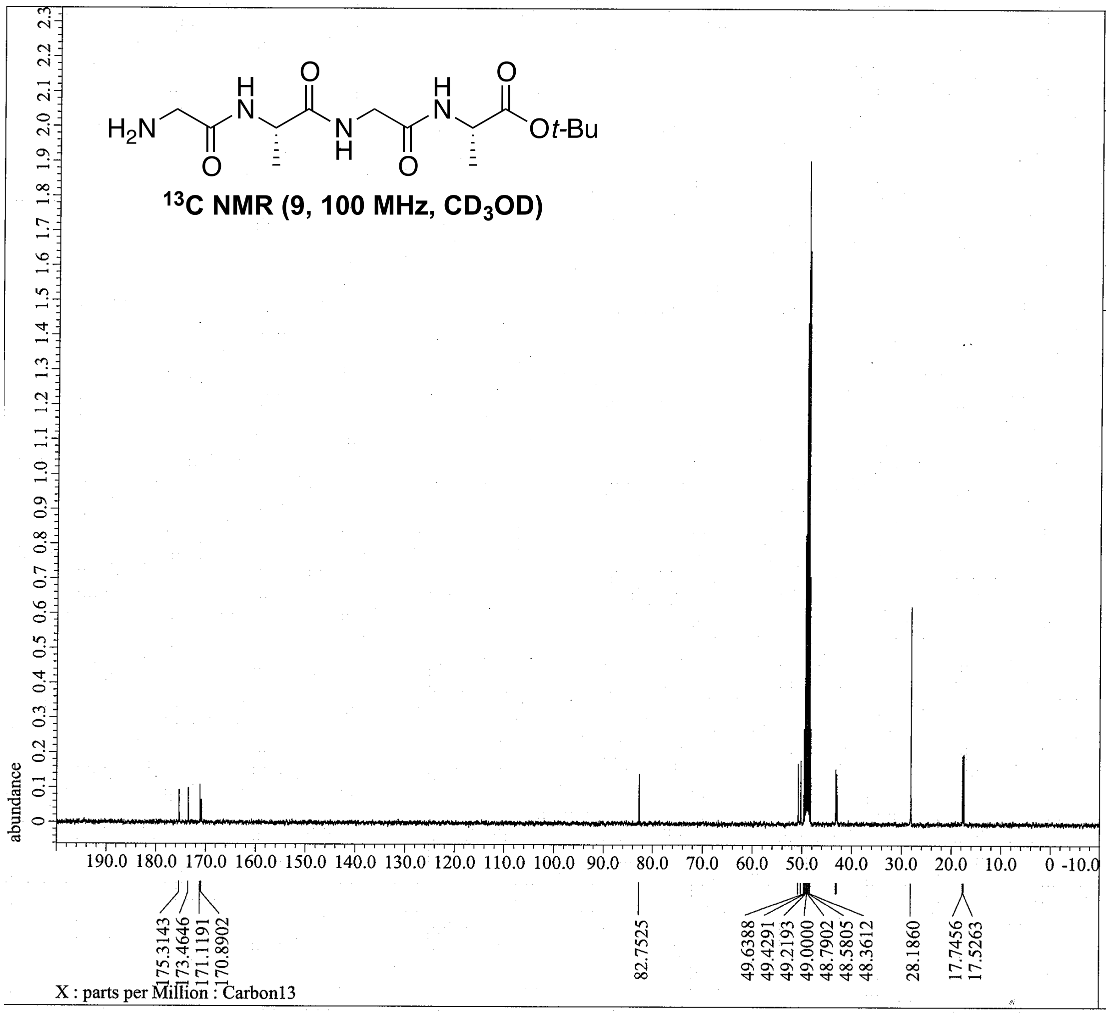
```
Filename          = wm12-202-13C_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm12-202-13C
Solvent           = METHANOL-D4
Creation_Time     = 21-JAN-2022 15:37:52
Revision_Time     = 21-JAN-2022 15:59:20
Current_Time      = 21-JAN-2022 15:59:45

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.4[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



$^1$H NMR (9, 400 MHz, CD$_3$OD)

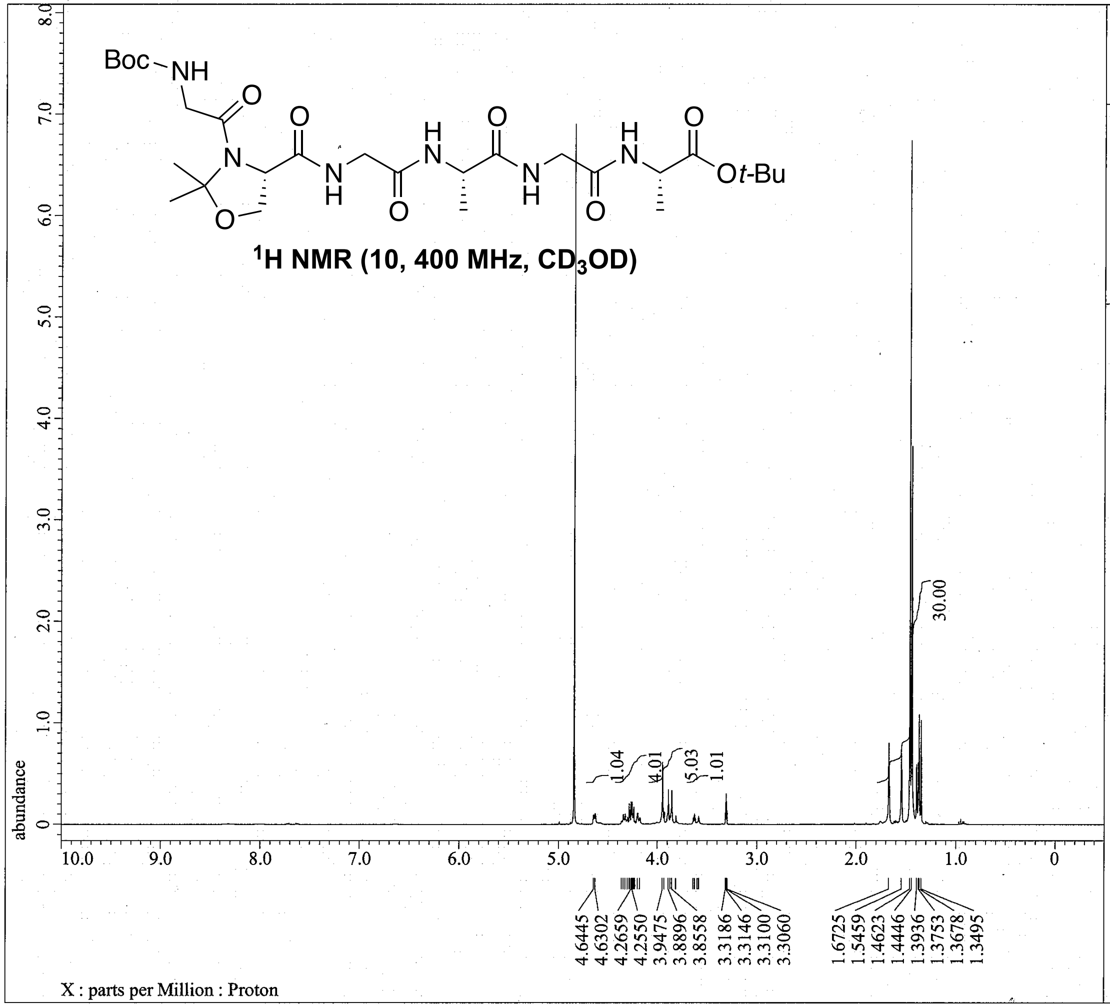
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-6_E1H-1-1.jdf
```

```
Filename          = wm13-6_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm13-6
Solvent           = METHANOL-D4
Creation_Time     =  2-FEB-2022 15:16:27
Revision_Time     =  2-FEB-2022 15:17:22
Current_Time      =  2-FEB-2022 15:17:41

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.8[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (9, 100 MHz, $CD_3OD$)

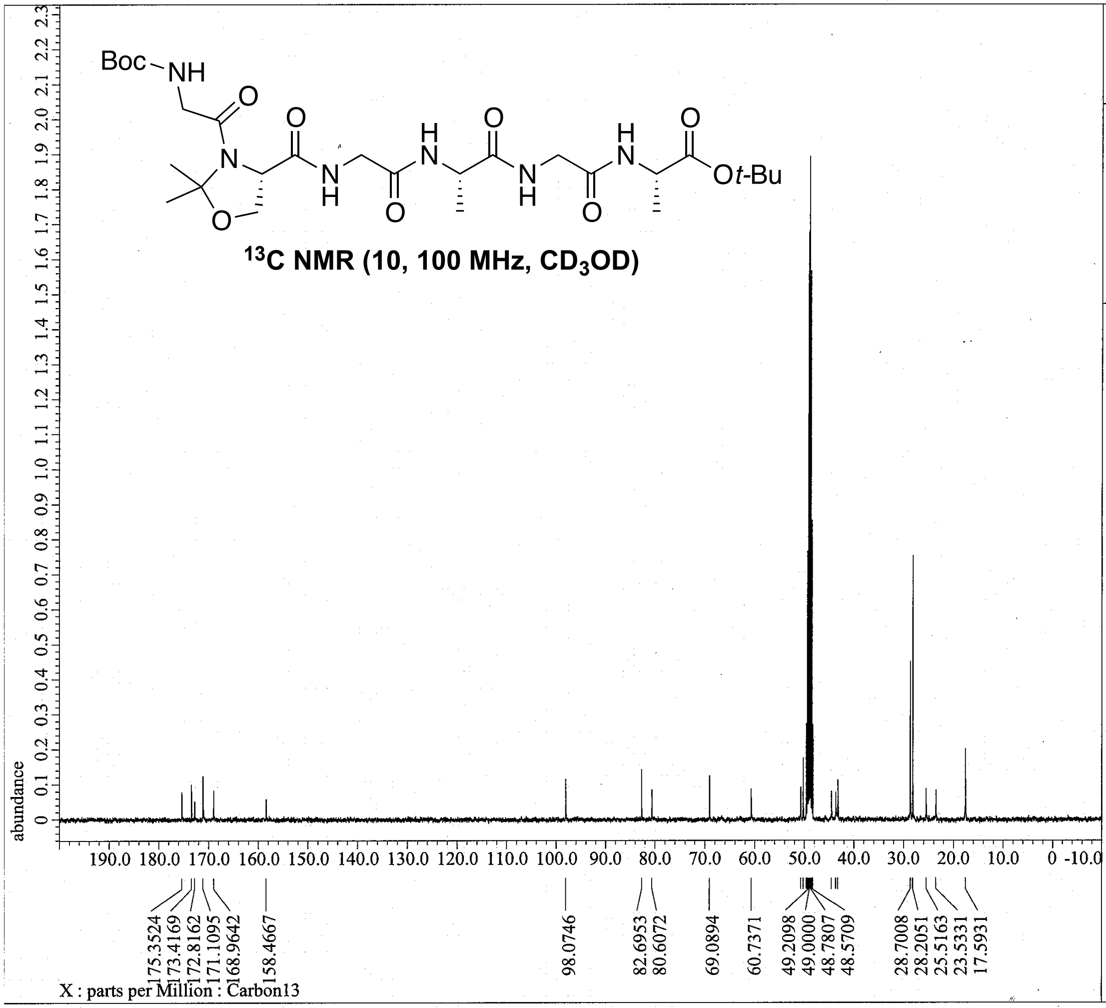
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-6-13C_E13C-1-1.jdf

Filename            = wm13-6-13C_E13C-1-3.jdf
Author              = delta
Experiment          = single_pulse_dec.jxp
Sample_Id           = wm13-6-13C
Solvent             = METHANOL-D4
Creation_Time       =  2-FEB-2022 15:18:37
Revision_Time       =  2-FEB-2022 15:40:07
Current_Time        =  2-FEB-2022 15:40:56

Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = TRUE
Scans               = 512
Total_Scans         = 512

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 23.8[dC]
X_90_Width          = 10.1[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.6[dB]
X_Pulse             = 3.36666667[us]
Irr_Atn_Dec         = 25.996[dB]
Irr_Atn_Noe         = 25.996[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
```



$^1$H NMR (10, 400 MHz, $CD_3OD$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-7-2_E1H-4-1.jdf
```

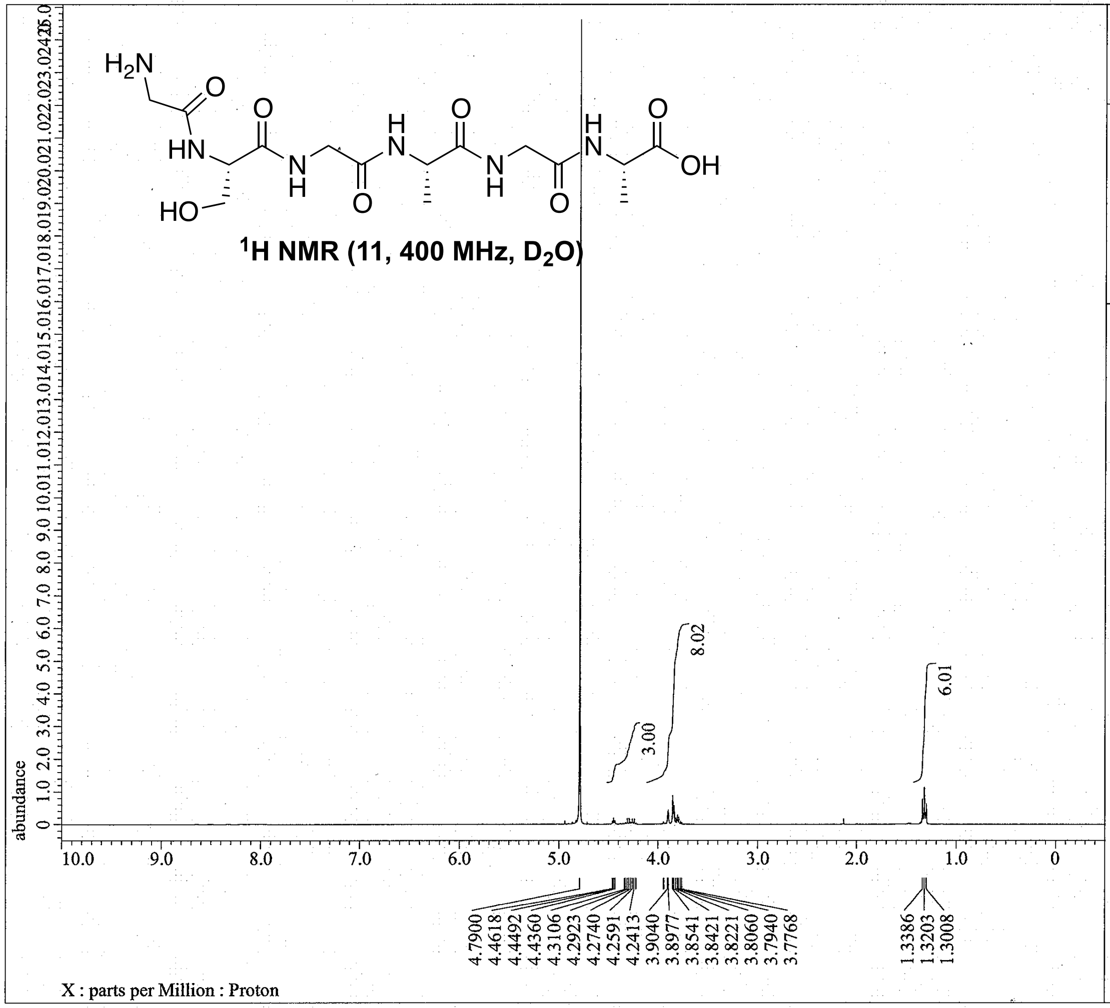
```
Filename          = wm13-7-2_E1H-4-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm13-7-2
Solvent           = METHANOL-D4
Creation_Time     =  5-FEB-2022 10:33:35
Revision_Time     =  5-FEB-2022 10:32:52
Current_Time      =  5-FEB-2022 10:33:10

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 24
Temp_Get          = 23.6[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (10, 100 MHz, CD$_3$OD)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-7-2-13C_E13C-4-1.jdf
```

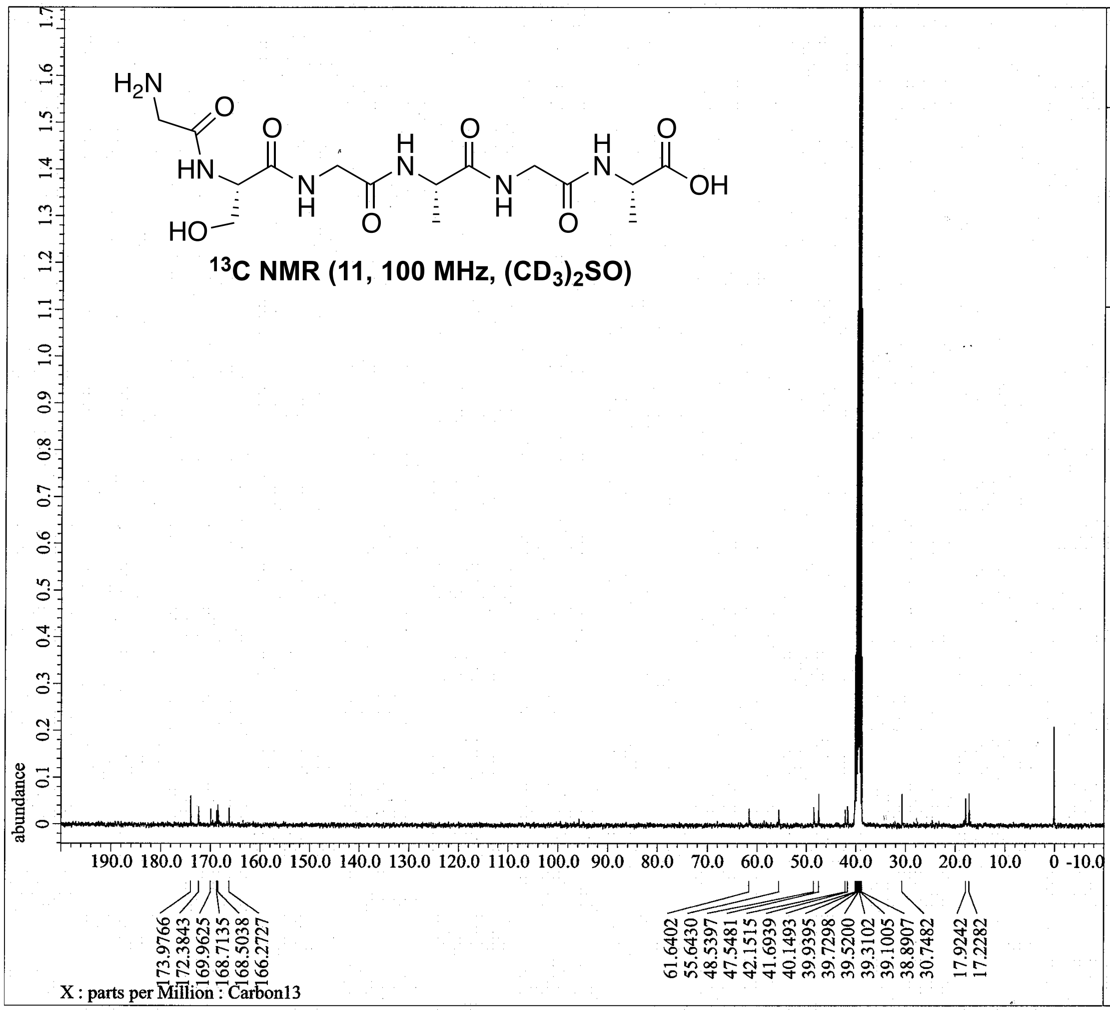
```
Filename           = wm13-7-2-13C_E13C-4-3.jdf
Author             = delta
Experiment         = single_pulse_dec.jxp
Sample_Id          = wm13-7-2-13C
Solvent            = METHANOL-D4
Creation_Time      =  5-FEB-2022 10:35:42
Revision_Time      =  5-FEB-2022 10:56:52
Current_Time       =  5-FEB-2022 10:57:26

Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 512
Total_Scans        = 512

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.7[dC]
X_90_Width         = 10.1[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.6[dB]
X_Pulse            = 3.36666667[us]
Irr_Atn_Dec        = 25.996[dB]
Irr_Atn_Noe        = 25.996[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
```



$^1$H NMR (11, 400 MHz, $D_2O$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-24-~~1-2~~2_E1H-1-1.jdf
```

```
Filename          = wm13-24-1-2_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wm13-24-1-2
Solvent           = D2O
Creation_Time     = 26-FEB-2022 10:43:27
Revision_Time     = 26-FEB-2022 10:43:01
Current_Time      = 26-FEB-2022 10:43:37

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 4.36731904[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22897343[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 23.1[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



$^{13}$C NMR (11, 100 MHz, $(CD_3)_2SO$)

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wm13-20-13C_E13C-1-1.jdf
```

```
Filename          = wm13-20-13C_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wm13-20-13C
Solvent           = DMSO-D6
Creation_Time     = 18-FEB-2022 11:43:00
Revision_Time     = 18-FEB-2022 12:30:36
Current_Time      = 18-FEB-2022 12:31:04

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.9[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```