Supporting Information for

Silicon-Based Hydrophobic Tags Applied in Liquid-Phase Peptide Synthesis: Protected DRGN-1 and Poly Alanine Chain Synthesis

An Wu,* Isai Ramakrishna, Tomohiro Hattori and Hisashi Yamamoto*

*Corresponding authors. Email: awuad@isc.chubu.ac.jp (A. W.)
hyamamoto@isc.chubu.ac.jp (H. Y.)

Molecular Catalyst Research Center, Chubu University,
1200 Matsumoto-cho, Kasugai, Aichi 487-8501, Japan

This PDF file includes:

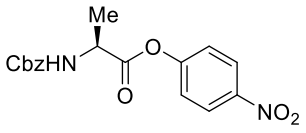
Table of Contents

I. General information

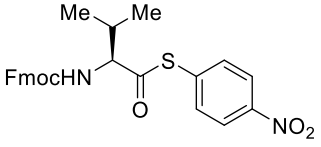
NMR spectra were recorded on a JEOL 400SS spectrometer operating at 400 MHz and 100 MHz for ^{1}H and ^{13}C acquisitions, respectively. Chemical shifts are reported in ppm with a solvent resonance as an internal standard (^{1}H NMR: tetramethylsilane, $CDCl_3$, $(CD_3)_2SO$, D_2O and CF_3CO_2D as internal standards, indicating 0, 7.26, 2.50, 4.79 and 11.50 ppm, respectively. ^{13}C NMR: $CDCl_3$, $(CD_3)_2SO$ and CF_3CO_2D as internal standards, indicating 77.0, 39.52 and 116.6 ppm, respectively). Data is reported as follows: s = singlet, br = broad, d = doublet, t = triplet, q = quartet, quin = quintet, sep = septet, m = multiplet; coupling constants in Hz; integration. FT-IR spectra were recorded with a Bruker ALPHA (Eco-ATR) spectrometer. MS spectra were recorded with a JEOL JMS-T100CS "AccuTOF CS" mass spectrometer with electrospray ionization time-of flight (ESI-TOF) for HRMS measurements. Ee and dr 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, IB-3, ID-3 and IE-3 columns (4.6 mm × 25 cm). Peptide purity was determined by reversed-phase high performance liquid chromatography (RP-HPLC) using an Agilent Technologies 1220 Infinity LC and ODS-HL column (5μm, 4.6 mm × 25 cm) from GL Siences Inc., XSelect CSH C18 column (5μm, 4.6 mm × 50 mm) from Waters. 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, THF and $CHCl_3$, were purchased from FUJIFILM Wako Pure Chemical Co. and Sigma-Aldrich Co. LLC. These solvents were used without further treatment. Amino acids and their derivatives were purchased from Sigma-Aldrich Co. LLC., Watanabe Chemical Ind., Ltd., Tokyo Chemical Industry Co., Ltd., Combi-Blocks, Inc., Chem-Impex Int'l Inc., and Fluorochem Ltd. Triethyl amine were purchased from FUJIFILM Wako Pure Chemical Co. Tris(triethylsilyl)silane, trichloro(phenyl)silane, chlorodiisobutyl(octadecyl)silane and Li were purchased from Sigma-Aldrich Co. LLC.. Chlorotrihexylsilane, pentafluorophenol, triflic acid, 5-oxohexanoic acid, 1-methylimidazole and diethyl 3-oxopentanedioate were purchased from Tokyo Chemical Industry Co., Ltd. Amberlyst™ A21 was purchased from Sigma-Aldrich Co. LLC.

II. Preparation of building blocks for peptide elongation

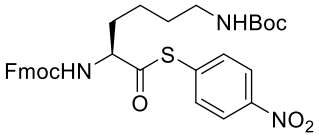
The following active amino acid esters and neutralized amino acids were used in this project.



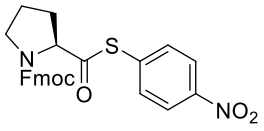
Cbz-Ala-ONp



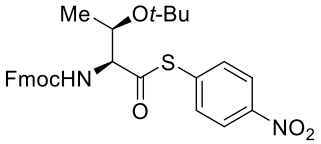
Fmoc-Val-SNp



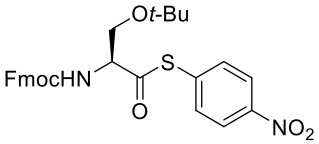
Fmoc-Lys(Boc)-SNp



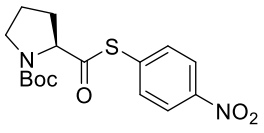
Fmoc-Pro-SNp



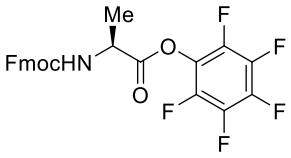
Fmoc-Thr(*t*-Bu)-SNp



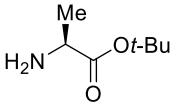
Fmoc-Ser(*t*-Bu)-SNp



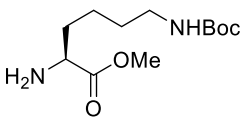
Boc-Pro-SNp



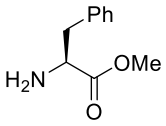
Fmoc-Ala-OPfp



H-Ala-O*t*-Bu

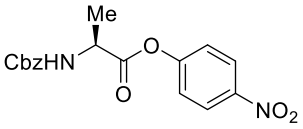


H-Lys(Boc)-OMe



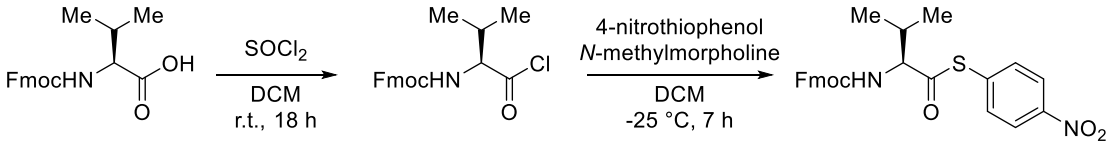
H-Phe-OMe

Synthesis of active amino acid esters.



Cbz-Ala-ONp

4-Nitrophenyl ((benzyloxy)carbonyl)-L-alaninate (Fmoc-Ala-ONp) was purchased from Chem-Impex Int'l Inc.



Fmoc-Val-OH **Fmoc-Val-Cl**, 99% **Fmoc-Val-SNp**, 93%

***S*-(4-Nitrophenyl) (*S*)-2-((((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)-3-methylbutanethioate (Fmoc-Val-SNp)** At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Val-OH** (3.39 g, 10.0 mmol, 1.0 equiv) was added dichloromethane (40 mL). The thionyl chloride (5.80 mL, 80.0 mmol, 8.0 equiv) was added. The resulting mixture was stirred under room temperature for 18 h. After completion, the reaction mixture was concentrated. The residue was dissolved in dichloromethane and the solvent was remove *in vacuo*. This step was repeated for another three times to remove the excess thionyl chloride. The product **Fmoc-Val-Cl** was obtained as a white solid in 99% yield (3.57 g).

At -25 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Val-Cl** (3.54 g, 9.9 mmol, 1.0 equiv) was added 4-nitrothiophenol (1.84 g, 11.9 mmol, 1.2 equiv) and dichloromethane (100 mL). The *N*-methylmorpholine (1.42 mL, 12.9 mmol, 1.3 equiv) was added over 10 min. The resulting mixture was stirred at -25 °C for 7 h. After completion, saturated Na_2CO_3 solution (70 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×50 mL). The combined organic layers were added 150 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×70 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 5:1) to afford the product **Fmoc-Val-SNp** as a pale yellow solid in 93% yield (4.40 g) with 99.8% ee.

Rf = 0.26 (hexanes/EtOAc = 5:1).

M.p. 123-125 °C.

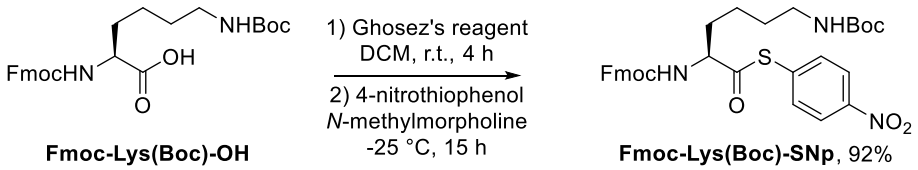
$[\alpha]_D^{27}$ = -24.44 (*c* 0.90, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, J = 8.8 Hz, 2H), 7.82 – 7.74 (m, 2H), 7.67 – 7.54 (m, 4H), 7.46 – 7.37 (m, 2H), 7.37 – 7.27 (m, 2H), 5.20 (d, J = 9.3 Hz, 1H), 4.65 – 4.55 (m, 1H), 4.54 – 4.43 (m, 2H), 4.27 (t, J = 6.6 Hz, 1H), 2.41 – 2.32 (m, 1H), 1.07 – 0.91 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 197.2, 156.1, 148.2, 143.6, 143.5, 141.4, 135.9, 135.0, 127.8, 127.1, 124.9, 123.9, 120.0, 67.2, 66.1, 47.2, 30.8, 19.3, 17.0.

IR (neat) 3328, 2965, 2930, 1703, 1599, 1518, 1449, 1342, 1249, 1223, 1107 cm^{-1}.

HRMS (ESI) Calcd for $C_{26}H_{24}N_2O_5SNa$ $[M+Na]^+$: 499.1304, Found: 499.1283.



Fmoc-Lys(Boc)-OH **Fmoc-Lys(Boc)-SNp**, 92%

***S*-(4-Nitrophenyl) (*S*)-2-((((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)-6-((*tert*-butoxycarbonyl)amino)hexanethioate (Fmoc-Lys(Boc)-SNp)** At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Lys(Boc)-OH** (468.6 mg, 1.0 mmol, 1.0 equiv) was added dichloromethane (10 mL). The Ghosez's reagent (145.5 μL, 1.1 mmol, 1.1 equiv) was added. The resulting mixture was stirred under room temperature. After 4 h, the reaction mixture was cooled to -25 °C. The 4-nitrothiophenol (186.2 mg, 1.2 mmol, 1.2 equiv) was added, followed by adding *N*-methylmorpholine (142.9 μL, 1.3 mmol, 1.3 equiv) over 10 min. The resulting mixture was stirred at -25 °C for 15 h. After completion, saturated Na_2CO_3 solution (10 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×30 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1 to 3:1). After the solvents were removed, the solid product was washed by diethyl ether and filtered to remove the byproduct of Ghosez's reagent to afford the pure product **Fmoc-Lys(Boc)-SNp** as a white solid in 92% yield (554.4 mg) with 98.7% ee.

Rf = 0.18 (hexanes/EtOAc = 3:1).

M.p. 154-155 °C.

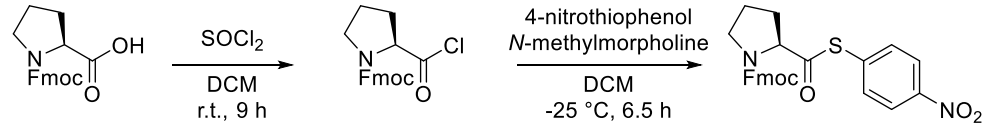
$[\alpha]_D^{27}$ = -30.00 (*c* 1.00, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, J = 8.6 Hz, 2H), 7.81 – 7.74 (m, 2H), 7.69 – 7.62 (m, 2H), 7.58 (d, J = 8.5 Hz, 2H), 7.45 – 7.35 (m, 2H), 7.35 – 7.27 (m, 2H), 5.65 (d, J = 8.1 Hz, 1H), 4.67 – 4.54 (m, 2H), 4.52 – 4.44 (m, 1H), 4.44 – 4.36 (m, 1H), 4.26 (t, J = 6.8 Hz, 1H), 3.26 – 3.04 (m, 2H), 2.01 – 1.88 (m, 1H), 1.88 – 1.74 (m, 1H), 1.54 – 1.40 (m, 13H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 197.8, 156.4, 156.0, 148.2, 143.65, 143.55, 141.3, 136.0, 135.0, 127.7, 127.1, 125.0, 123.9, 120.0, 79.4, 67.2, 61.3, 47.2, 39.4, 31.3, 29.7, 28.4, 22.2.

IR (neat) 3363, 3018, 2937, 1690, 1520, 1449,1343, 1244, 1231, 1215, 1163, 1105 cm^{-1}.

HRMS (ESI) Calcd for $C_{32}H_{35}N_3O_7SNa$ $[M+Na]^+$: 628.2093, Found: 628.2061.



Fmoc-Pro-OH **Fmoc-Pro-Cl**, >99% **Fmoc-Pro-SNp**, 93%

(9*H*-Fluoren-9-yl)methyl (*S*)-2-(((4-nitrophenyl)thio)carbonyl)pyrrolidine-1-carboxylate (Fmoc-Pro-SNp) At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Pro-OH** (1.69 g, 5.0 mmol, 1.0 equiv) was added dichloromethane (25 mL). The thionyl chloride (2.90 mL, 40.0 mmol, 8.0 equiv) was added. The resulting mixture was stirred under room temperature for 9 h. After completion, the reaction mixture was concentrated. The residue was dissolved in dichloromethane and the solvent was remove *in vacuo*. This step was repeated for another three times to remove the excess thionyl chloride. The product **Fmoc-Pro-Cl** was obtained as a white solid in >99% yield (1.78 g).

At -25 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Pro-Cl** (1.78 g, 5.0 mmol, 1.0 equiv) was added 4-nitrothiophenol (931.0 mg, 6.0 mmol, 1.2 equiv) and dichloromethane (50 mL). The *N*-methylmorpholine (714.7 μL, 6.5 mmol, 1.3 equiv) was added over 10 min. The resulting mixture was stirred at -25 °C for 6.5 h. After completion, saturated Na_2CO_3 solution (50 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×30 mL). The combined organic layers were added 100 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×50 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 8:1 to 4:1 to 2:1) to afford the product **Fmoc-Pro-SNp** as a yellow solid in 93% yield (2.21 g) with 99.7% ee.

Rf = 0.18 (hexanes/EtOAc = 5:1).

M.p. 122-124 °C.

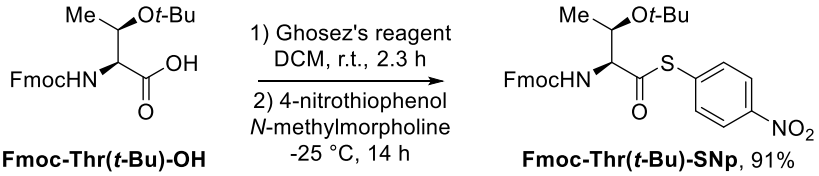
$[\alpha]_D^{27}$ = -56.71 (*c* 0.97, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 – 8.17 (m, 2H), 7.81 – 7.71 (m, 2H), 7.69 – 7.52 (m, 3H), 7.46 – 7.23 (m, 5H), 4.72 – 4.49 (m, 2H), 4.48 – 4.36 (m, 1H), 4.35 – 4.18 (m, 1H), 3.76 – 3.65 (m, 1H), 3.65 – 3.50 (m, 1H), 2.40 – 1.91 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 198.3, 197.9, 155.2, 154.5, 148.1(2C), 143.8, 143.7, 143.6, 141.3(2C), 136.2, 136.0, 134.9, 134.8, 127.7(2C), 127.0(2C), 125.04, 124.96, 124.84, 124.76, 123.8(2C), 120.0(2C), 67.7(2C), 66.6, 66.2, 47.4, 47.2(2C), 46.9, 31.7, 30.6, 24.2, 23.2.

IR (neat) 3019, 1698, 1598, 1578, 1518, 1409, 1340, 1215, 1176, 1107 cm^{-1}.

HRMS (ESI) Calcd for $C_{26}H_{22}N_2O_5SNa$ $[M+Na]^+$: 497.1147, Found: 497.1159.



Fmoc-Thr(*t*-Bu)-OH **Fmoc-Thr(*t*-Bu)-SNp**, 91%

***S*-(4-Nitrophenyl) (2*S*,3*R*)-2-((((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)-3-(tert-butoxy)butanethioate (Fmoc-Thr(*t*-Bu)-SNp)** At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Thr(*t*-Bu)-OH** (795.0 mg, 2.0 mmol, 1.0 equiv) was added dichloromethane (20 mL). The Ghosez's reagent (291.1 μL, 2.2 mmol, 1.1 equiv) was added. The resulting mixture was stirred under room temperature. After 2.3 h, the reaction mixture was cooled to -25 °C. The 4-nitrothiophenol (341.4 mg, 2.2 mmol, 1.1 equiv) was added, followed by adding *N*-methylmorpholine (285.9 μL, 2.6 mmol, 1.3 equiv) over 10 min. The resulting mixture was stirred at -25 °C for 14 h. After completion, saturated Na_2CO_3 solution (20 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were added 70 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×50 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 8:1 to 5:1 to 4:1). After the solvents were removed, the solid product was washed by hexane and filtered to remove the byproduct of Ghosez's reagent to afford the pure product **Fmoc-Thr(*t*-Bu)-SNp** as a white solid in 91% yield (968.0 mg) with 99.9:0.1 dr.

Rf = 0.35 (hexanes/EtOAc = 5:1).

M.p. 182-184 °C.

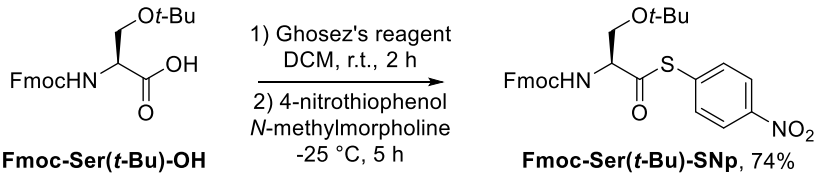
$[\alpha]_D^{27}$ = -52.53 (*c* 0.99, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, J = 8.8 Hz, 2H), 7.83 – 7.75 (m, 2H), 7.72 – 7.64 (m, 2H), 7.55 (d, J = 8.8 Hz, 2H), 7.46 – 7.37 (m, 2H), 7.37 – 7.29 (m, 2H), 5.83 (d, J = 9.2 Hz, 1H), 4.69 – 4.60 (m, 1H), 4.52 – 4.43 (m, 1H), 4.37 – 4.28 (m, 3H), 1.22 (d, J = 6.2 Hz, 3H), 1.15 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 198.6, 156.5, 148.2, 143.7, 143.6, 141.4, 136.5, 134.9, 127.7, 127.1, 125.0, 123.9, 120.0, 74.4, 67.3, 67.2, 66.8, 47.3, 28.6, 21.1.

IR (neat) 3347, 3020, 2976, 1720, 1522, 1497, 1344, 1214 cm^{-1}.

HRMS (ESI) Calcd for $C_{29}H_{30}N_2O_6SNa$ $[M+Na]^+$: 557.1722, Found: 557.1739.



Fmoc-Ser(*t*-Bu)-OH **Fmoc-Ser(*t*-Bu)-SNp**, 74%

***S*-(4-Nitrophenyl) (*S*)-2-((((9*H*-fluoren-9-yl)methoxy)carbonyl)amino)-3-(tert-butoxy)propanethioate (Fmoc-Ser(*t*-Bu)-SNp)** At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ser(*t*-Bu)-OH** (1.917 g, 5.0 mmol, 1.0 equiv) was added dichloromethane (50 mL). The Ghosez's reagent (727.6 μL, 5.5 mmol, 1.1 equiv) was added. The resulting mixture was stirred under room temperature. After 2 h, the reaction mixture was cooled to -25 °C. The 4-nitrothiophenol (853.4 mg, 5.5 mmol, 1.1 equiv) was added, followed by adding *N*-methylmorpholine (714.7 μL, 6.5 mmol, 1.3 equiv) over 10 min. The resulting mixture was stirred at -25 °C for 5 h. After completion, saturated Na_2CO_3 solution (50 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×30 mL). The combined organic layers were added 70 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×50 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 7:1 to 5:1 to 100% DCM to hexanes/EtOAc = 4:1). After the solvents were removed, the solid product was washed by hexane and filtered to remove the byproduct of Ghosez's reagent to afford the pure product **Fmoc-Ser(*t*-Bu)-SNp** as a white solid in 74% yield (1.93 g) with 94.9% ee.

Rf = 0.24 (hexanes/EtOAc = 5:1).

M.p. 146-148 °C.

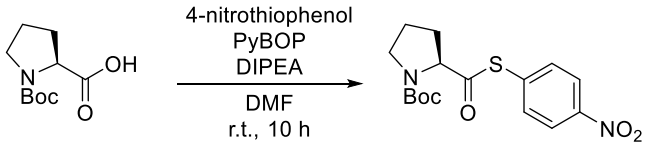
$[\alpha]_D^{23}$ = -42.16 (*c* 1.02, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, J = 8.8 Hz, 2H), 7.82 – 7.73 (m, 2H), 7.71 – 7.63 (m, 2H), 7.57 (d, J = 8.4 Hz, 2H), 7.47 – 7.37 (m, 2H), 7.37 – 7.27 (m, 2H), 5.83 (d, J = 8.9 Hz, 1H), 4.69 – 4.54 (m, 2H), 4.49 – 4.40 (m, 1H), 4.31 (t, J = 6.9 Hz, 1H), 3.97 (dd, J = 9.3, 2.6 Hz, 1H), 3.59 (dd, J = 9.2, 3.5 Hz, 1H), 1.20 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 197.3, 156.0, 148.2, 143.7, 143.6, 141.3, 136.4, 135.0, 127.8, 127.1, 125.1, 125.0, 123.9, 120.0, 73.9, 67.4, 61.7, 61.3, 47.2, 27.3.

IR (neat) 3353, 2974, 1703, 1600, 1578, 1518, 1499, 1477, 1449, 1342, 1313, 1274, 1215, 1192, 1106 cm^{-1}.

HRMS (ESI) Calcd for $C_{28}H_{28}N_2O_6SNa$ $[M+Na]^+$: 543.1566, Found: 543.1545.



Boc-Pro-OH **Boc-Pro-SNp**, 87%

***tert*-Butyl (*S*)-2-(((4-nitrophenyl)thio)carbonyl)pyrrolidine-1-carboxylate (Boc-Pro-SNp)**

At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co), **Boc-Pro-OH** (861.0 mg, 4.0 mmol, 1.0 equiv) and 4-nitrothiophenol (620.7 mg, 4.0 mmol, 1.0 equiv) was added *N*,*N*-dimethylformamide (14 mL). The resulting mixture was stirred under room temperature for 5 min. Then, benzotriazol-1-yloxytripyrrolidinophosphonium hexafluorophosphate (2.08 g, 4.0 mmol, 1.0 equiv) and *N*,*N*-diisopropylethylamine (714.7 μL, 6.5 mmol, 1.3 equiv) were added. After stirring under room temperature for 10 h, saturated Na_2CO_3 solution (20 mL) and EtOAc (20 mL) were added. The layers were separated. The aqueous layer was extracted with EtOAc (4×20 mL). The combined organic layers were washed with water for three times. The organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1) to afford the product **Boc-Pro-SNp** as a white solid in 87% yield (1.22 g) with 99.9% ee.

Rf = 0.29 (hexanes/EtOAc = 5:1).

M.p. 72-73 °C.

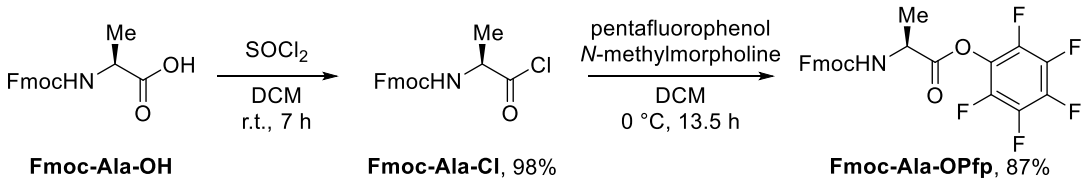
$[\alpha]_D^{23}$ = -111.02 (*c* 1.18, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 – 8.14 (m, 2H), 7.64 – 7.52 (m, 2H), 4.68 – 4.36 (m, 1H), 3.68 – 3.42 (m, 2H), 2.43 – 1.88 (m, 4H), 1.53 – 1.45 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 198.9, 198.5, 154.6, 153.6, 148.1, 148.0, 136.7, 136.5, 134.8, 134.7, 123.9, 123.7, 80.9, 80.7, 66.4, 66.1, 47.0, 46.7, 31.5, 30.6, 28.3(2C), 24.2, 23.5.

IR (neat) 3104, 2977, 2882, 1696, 1599, 1578, 1519, 1477, 1380, 1366, 1341, 1311, 1258, 1159, 1118, 1091 cm^{-1}.

HRMS (ESI) Calcd for $C_{16}H_{20}N_2O_5SNa$ $[M+Na]^+$: 375.0991, Found: 375.0994.



Fmoc-Ala-OH **Fmoc-Ala-Cl**, 98% **Fmoc-Ala-OPfp**, 87%

Perfluorophenyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OPfp) At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OH** (6.23 g, 20.0 mmol, 1.0 equiv) was added dichloromethane (100 mL). The thionyl chloride (11.6 mL, 160.0 mmol, 8.0 equiv) was added. The resulting mixture was stirred under room temperature for 7 h. After completion, the reaction mixture was concentrated. The residue was dissolved in dichloromethane and the solvent was remove *in vacuo*. This step was repeated for another three times to remove the excess thionyl chloride. The product **Fmoc-Ala-Cl** was obtained as a white solid in 98% yield (6.49 g).

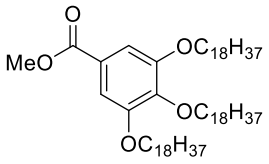
At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and pentafluorophenol (2.21 g, 12.0 mmol, 1.5 equiv) was added **Fmoc-Val-Cl** (2.64 g, 8.0 mmol, 1.0 equiv) and dichloromethane (80 mL). The *N*-methylmorpholine (1.32 mL, 12.0 mmol, 1.5 equiv) was added over 10 min. The resulting mixture was stirred at 0 ºC for 13.5 h. After completion, saturated Na_2CO_3 solution (80 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×50 mL). The combined organic layers were added 150 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×70 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc/DCM = 3:1:1) to afford the product **Fmoc-Ala-OPfp** as a white solid in 87% yield (3.34 g). It is a known compound. The characterization data match the reported data.[1]

Neutralization of amino acid HCl salts.

H-Ala-O*t*-Bu, **H-Lys(Boc)-OMe** and **H-Phe-OMe** were neutralized from the HCl salts with Amberlyst™ A21 according to the procedure in the literatures.[2, 6]

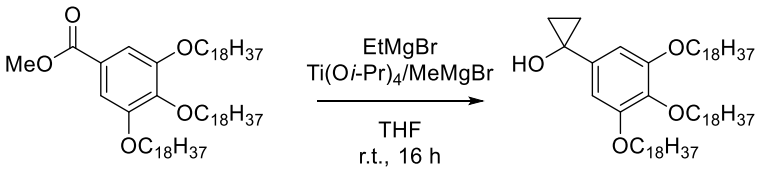
III. Synthesis of TAG2 and initial requirements test

Synthesis of TAG2.



S1

Methyl 3,4,5-tris(octadecyloxy)benzoate (S1) was prepared according to the procedure in the literature and the characterization data match the reported data.[3]



S1 **HOTAG2**, 44%

1-(3,4,5-Tris(octadecyloxy)phenyl)cyclopropan-1-ol (HOTAG2) At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co), 90 mL tetrahydrofuran and titanium isopropoxide (2.73 mL, 9.22 mmol, 1.0 equiv) was added methylmagnesium bromide solution (3.0 M in diethyl ether, 4.61 mL, 13.83 mmol, 1.5 equiv). After stirring for 5 min under room temperature, the mixture was cooled to 0 °C and the substrate **S1** (8.68 g, 9.22 mmol, 1.0 equiv) was added, followed by adding ethylmagnesium bromide solution (1.0 M in tetrahydrofuran, 23.05 mL, 23.05 mmol, 2.5 equiv) over 20 min. The reaction was stirred at room temperature for 16 h. Then, the mixture was cooled to 0 °C and 10% H_2SO_4 solution (40 mL) was added, and the layers were separated. The aqueous layer was extracted with EtOAc (3×50 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1) to afford the product **HOTAG2** as a white solid in 44% yield (3.77 g).

Rf = 0.44 (hexanes/EtOAc = 5:1).

M.p. 70-72 °C.

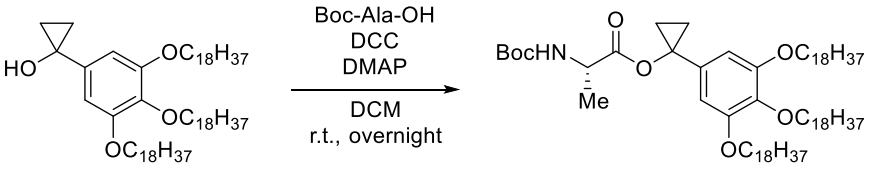
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.53 (s, 2H), 4.01 – 3.88 (m, 6H), 2.25 (s, 1H), 1.85 – 1.69 (m, 6H), 1.52 – 1.39 (m, 6H), 1.39 – 1.14 (m, 86H), 1.03 – 0.95 (m, 2H), 0.92 – 0.84 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.1, 139.2, 137.0, 103.7, 73.4, 69.2, 57.1, 31.9, 30.3, 29.72, 29.67, 29.5, 29.44, 29.37, 26.14, 26.11, 22.7, 17.3, 14.1.

IR (neat) 2916, 2849, 1587, 1505, 1463, 1214, 1120 cm^{-1}.

HRMS (ESI) Calcd for $C_{63}H_{118}O_4Na$ $[M+Na]^+$: 961.8928, Found: 961.8925.

Esterification test.



HOTAG2 **Boc-Ala-OTAG2**, 89%

1-(3,4,5-Tris(octadecyloxy)phenyl)cyclopropyl (*tert*-butoxycarbonyl)-L-alaninate (Boc-Ala-OTAG2) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG2** (281.9 mg, 0.30 mmol, 1.0 equiv) was added dichloromethane (1.5 mL). The Boc-Ala-OH (170.3 mg, 0.90 mmol, 3.0 equiv) was added, followed by adding DMAP (36.7 mg, 0.30 mmol, 1.0 equiv) and DCC (185.7 mg, 0.90 mmol, 3.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 26:1 to 20:1) to afford the product **Boc-Ala-OTAG2** as a white solid in 89% yield (295.4 mg).

Rf = 0.59 (hexanes/EtOAc = 5:1).

M.p. 46-48 ºC.

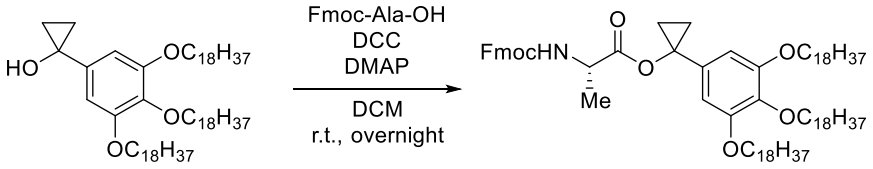
$[\alpha]_D^{23}$ = -8.74 (*c* 1.03, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.54 (s, 2H), 4.99 (d, J = 7.9 Hz, 1H), 4.34 – 4.16 (m, 1H), 4.01 – 3.87 (m, 6H), 1.83 – 1.66 (m, 6H), 1.51 – 1.09 (m, 106H), 0.92 – 0.84 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 155.0, 152.9, 137.6, 134.2, 105.4, 79.7, 73.3, 69.1, 61.2, 49.2, 31.9, 30.3, 29.71, 29.65, 29.44, 29.36, 28.3, 26.1, 22.7, 18.4, 14.6, 14.4, 14.1.

IR (neat) 2914, 2848, 1751, 1710, 1514, 1469, 1455, 1361, 1163, 1123 cm^{-1}.

HRMS (ESI) Calcd for $C_{71}H_{131}NO_7Na$ $[M+Na]^+$: 1132.9823, Found: 1132.9837.



HOTAG2 **Fmoc-Ala-OTAG2**, 65%

1-(3,4,5-Tris(octadecyloxy)phenyl)cyclopropyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG2) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG2** (281.9 mg, 0.30 mmol, 1.0 equiv) was added dichloromethane (1.5 mL). The Fmoc-Ala-OH (280.2 mg, 0.90 mmol, 3.0 equiv) was added, followed by adding DMAP (36.7 mg, 0.30 mmol, 1.0 equiv) and DCC (185.7 mg, 0.90 mmol, 3.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 25:1 to 20:1) to afford the product **Fmoc-Ala-OTAG2** as a pale yellow solid in 65% yield (241.7 mg).

Rf = 0.52 (hexanes/EtOAc = 5:1).

M.p. 77-78 °C.

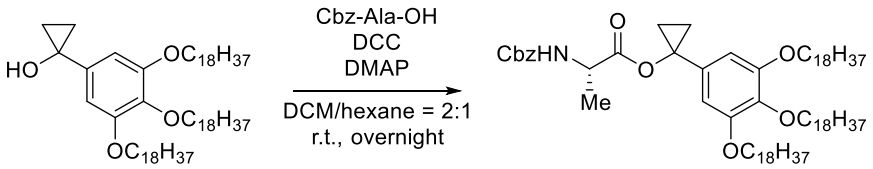
$[\alpha]_D^{23}$ = -2.04 (c 0.98, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, J = 7.6 Hz, 3H), 7.68 – 7.50 (m, 2H), 7.48 – 7.35 (m, 2H), 7.35 – 7.27 (m, 2H), 6.57 (s, 2H), 5.31 (d, J = 7.7 Hz, 1H), 4.44 – 4.29 (m, 3H), 4.21 (t, J = 7.2 Hz, 1H), 4.02 – 3.79 (m, 6H), 1.84 – 1.67 (m, 6H), 1.51 – 1.06 (m, 97H), 0.93 – 0.85 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 155.5, 152.9, 143.8, 143.7, 141.3, 137.8, 134.0, 127.7, 127.0, 125.0, 119.9, 105.6, 73.3, 69.1, 67.0, 61.6, 49.6, 47.1, 31.9, 30.3, 29.71, 29.66, 29.45, 29.36, 26.1, 22.7, 18.5, 14.5, 14.3, 14.1.

IR (neat) 3318, 2915, 2849, 1745, 1686, 1539, 1467, 1450, 1359, 1276, 1205, 1123 cm^{-1}.

HRMS (ESI) Calcd for $C_{81}H_{133}NO_7Na$ $[M+Na]^+$: 1254.9980, Found: 1255.0025.



HOTAG2 **Cbz-Ala-OTAG2**, 80%

1-(3,4,5-Tris(octadecyloxy)phenyl)cyclopropyl ((benzyloxy)carbonyl)-L-alaninate (Cbz-Ala-OTAG2) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG2** (114.1 mg, 0.12 mmol, 1.0 equiv) was added dichloromethane (1.2

mL) and hexane (0.6 mL). The Cbz-Ala-OH (81.3 mg, 0.36 mmol, 3.0 equiv) was added, followed by adding DMAP (17.8 mg, 0.15 mmol, 1.2 equiv) and DCC (75.2 mg, 0.36 mmol, 3.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 25:1 to 15:1 to 10:1) to afford the product **Cbz-Ala-OTAG2** as a white solid in 80% yield (110.7 mg).

Rf = 0.42 (hexanes/EtOAc = 5:1).

M.p. 67-68 °C.

$[\alpha]_D^{22}$ = -15.73 (*c* 0.89, $CHCl_3$).

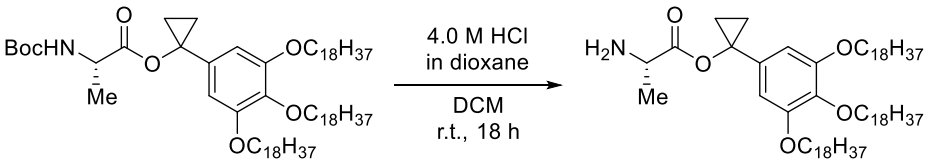
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.27 (m, 5H), 6.55 (s, 2H), 5.27 (d, J = 8.0 Hz, 1H), 5.16 – 5.03 (m, 2H), 4.42 – 4.27 (m, 1H), 3.99 – 3.88 (m, 6H), 1.84 – 1.68 (m, 6H), 1.51 – 1.09 (m, 97H), 0.93 – 0.84 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 155.5, 152.9, 137.7, 136.2, 134.0, 128.5, 128.1, 128.0, 105.5, 73.3, 69.1, 66.9, 61.5, 49.6, 31.9, 30.3, 29.71, 29.66, 29.44, 29.36, 26.1, 22.7, 18.5, 14.5, 14.3, 14.1.

IR (neat) 2923, 2852, 1748, 1719, 1586, 1509, 1468, 1424, 1360, 1214, 1119 cm^{-1}.

HRMS (ESI) Calcd for $C_{74}H_{129}NO_7Na$ $[M+Na]^+$: 1166.9667, Found: 1166.9668.

Tolerance test.



Boc-Ala-OTAG2 **H-Ala-OTAG2**, 63%

1-(3,4,5-Tris(octadecyloxy)phenyl)cyclopropyl L-alaninate (H-Ala-OTAG2) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Boc-Ala-OTAG2** (147.7 mg, 0.13 mmol, 1.0 equiv) was added dichloromethane (1.3 mL). The hydrochloric acid solution (4.0 M in dioxane, 0.16 mL, 0.65 mmol, 5.0 equiv) was added. The reaction was stirred at room temperature for 18 h. Then, 5 mL dichloromethane and 5 mL saturated $NaHCO_3$ solution were added, and the layers were separated. The aqueous layer was extracted with EtOAc (3×5 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography

(eluent: hexanes/EtOAc = 5:1 to 1:1) to afford the product **H-Ala-OTAG2** as a pale yellow solid in 63% yield (85.1 mg).

Rf = 0.09 (hexanes/EtOAc = 5:1).

M.p. 48-49 °C.

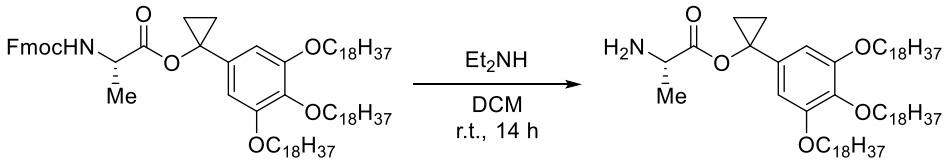
$[\alpha]_D^{20}$ = -7.48 (*c* 1.07, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.57 (s, 2H), 3.98 – 3.87 (m, 6H), 3.48 (q, J = 7.1 Hz, 1H), 1.83 – 1.67 (m, 6H), 1.48 – 1.39 (m, 6H), 1.38 – 1.10 (m, 93H), 0.92 – 0.84 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 152.9, 137.7, 134.4, 105.8, 73.4, 69.1, 60.9, 50.0, 31.9, 30.3, 29.7, 29.6, 29.44, 29.35, 26.1, 22.7, 20.5, 14.3, 14.1.

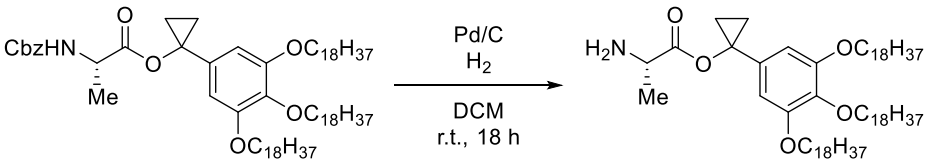
IR (neat) 2922, 2852, 1745, 1586, 1467, 1424, 1362, 1215, 1172, 1117 cm^{-1}.

HRMS (ESI) Calcd for $C_{66}H_{123}NO_5Na$ $[M+Na]^+$: 1032.9299, Found: 1032.9328.



Fmoc-Ala-OTAG2 **H-Ala-OTAG2**, 91%

1-(3,4,5-Tris(octadecyloxy)phenyl)cyclopropyl L-alaninate (H-Ala-OTAG2) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAG2** (123.3 mg, 0.10 mmol, 1.0 equiv) was added dichloromethane (1.0 mL). The diethylamine (51.7 μL, 0.50 mmol, 5.0 equiv) was added. The reaction was stirred at room temperature for 14 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1 to 1:1) to afford the product **H-Ala-OTAG2** as a pale yellow solid in 91% yield (91.6 mg).



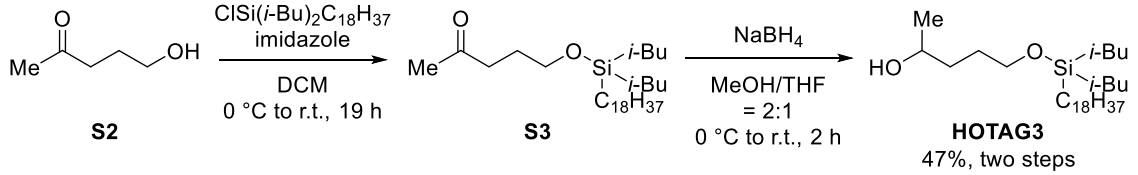
Cbz-Ala-OTAG2 **H-Ala-OTAG2**, 72%

1-(3,4,5-Tris(octadecyloxy)phenyl)cyclopropyl L-alaninate (H-Ala-OTAG2) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-OTAG2** (98.4 mg, 0.086 mmol, 1.0 equiv) was added dichloromethane (1.0 mL). The 10% Pd/C (18.3 mg, 0.017 mmol, 0.2 equiv) was added. Then evacuated and backfilled with hydrogen (this process was repeated in total of 5 times). The resulting mixture was stirred under

hydrogen atmosphere at room temperature for 18 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (5 mL) and the filtrate was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1 to 1:1) to afford the product **H-Ala-OTAG2** as a pale yellow solid in 72% yield (62.2 mg).

IV. Synthesis of TAG3 and initial requirements test

Synthesis of TAG3.



5-((Diisobutyl(octadecyl)silyl)oxy)pentan-2-ol (HOTAG3) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S2** (223.1 μL, 2.2 mmol, 1.1 equiv) was added dichloromethane (10 mL). The imidazole (204.2 mg, 3.0 mmol, 1.5 equiv) was added, followed by adding chlorodiisobutyl(octadecyl)silane (1.0 mL, 2.0 mmol, 1.0 equiv). The reaction was stirred at room temperature for 19 h. Then, water (15 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 30:1) to afford the product **S3** as a colorless oil with impurities. The product was put into next step without further purification.

At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above product was added methanol (6 mL) and tetrahydrofuran (3 mL). Cool the mixture to 0 ºC, followed by adding sodium borohydride (113.5 mg, 3.0 mmol, 1.5 equiv). The reaction was stirred at room temperature for 2 h. Then, 10 mL saturated NH_4Cl solution and 10 mL EtOAc were added, and the layers were separated. The aqueous layer was extracted with EtOAc (3×15 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 35:1 to 10:1) to afford the product **HOTAG3** as a colorless oil in 47% total yield (471.4 mg).

Rf = 0.48 (hexanes/EtOAc = 5:1).

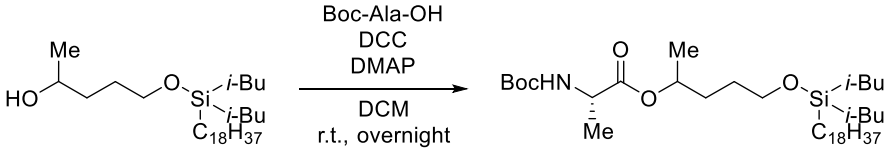
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 – 3.75 (m, 1H), 3.69 – 3.56 (m, 2H), 2.67 – 2.38 (m, 1H), 1.88 – 1.73 (m, 2H), 1.69 – 1.54 (m, 3H), 1.53 – 1.41 (m, 1H), 1.38 – 1.21 (m, 32H), 1.21 – 1.14 (m, 3H), 0.99 – 0.91 (m, 12H), 0.91 – 0.83 (m, 3H), 0.66 – 0.54 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 67.7, 62.9, 36.7, 33.7, 31.9, 29.7(8C), 29.65, 29.59, 29.4, 29.3(2C), 26.3, 24.4, 24.2, 23.4, 23.2, 22.7, 14.8, 14.1.

IR (neat) 3360, 2951, 2921, 2852, 1463, 1379, 1365, 1327, 1218, 1163, 1088, 1037 cm^{-1}.

HRMS (ESI) Calcd for $C_{31}H_{66}O_2SiNa$ $[M+Na]^+$: 521.4730, Found: 521.4717.

Esterification test.



HOTAG3 **Boc-Ala-OTAG3**, >99%

5-((Diisobutyl(octadecyl)silyl)oxy)pentan-2-yl (*tert*-butoxycarbonyl)-L-alaninate (Boc-Ala-OTAG3) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG3** (106.0 mg, 0.21 mmol, 1.0 equiv) was added dichloromethane (2.1 mL). The Boc-Ala-OH (120.5 mg, 0.64 mmol, 3.0 equiv) was added, followed by adding DMAP (31.1 mg, 0.25 mmol, 1.2 equiv) and DCC (131.5 mg, 0.64 mmol, 3.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1) to afford the product **Boc-Ala-OTAG3** as a colorless oil in >99% yield (143.5 mg).

Rf = 0.54 (hexanes/EtOAc = 5:1).

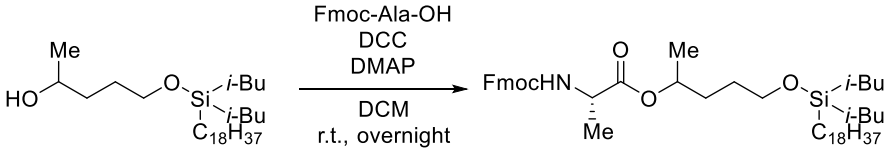
$[\alpha]_D^{27}$ = +11.11 (*c* 1.17, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.16 – 5.01 (m, 1H), 5.01 – 4.90 (m, 1H), 4.33 – 4.17 (m, 1H), 3.61 – 3.51 (m, 2H), 1.85 – 1.73 (m, 2H), 1.66 – 1.56 (m, 2H), 1.56 – 1.46 (m, 2H), 1.43 (s, 9H), 1.38 – 1.33 (m, 3H), 1.33 – 1.18 (m, 35H), 0.98 – 0.90 (m, 12H), 0.90 – 0.83 (m, 3H), 0.63 – 0.52 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 172.9(2C), 155.0(2C), 79.6(2C), 72.1, 72.0, 62.0(2C), 49.3(2C), 33.7(2C), 32.3, 32.2, 31.9(2C), 29.7(16C), 29.63(2C), 29.59(2C), 29.34(2C), 29.29(2C), 28.52, 28.47, 28.3(2C), 26.3(2C), 24.6(2C), 24.2(2C), 23.3(2C), 22.7(2C), 20.0, 19.8, 18.9, 18.8, 14.9(2C), 14.1(2C).

IR (neat) 2922, 2853, 1718, 1497, 1453, 1379, 1365, 1247, 1214, 1166 cm^{-1}.

HRMS (ESI) Calcd for $C_{39}H_{79}NO_5SiNa$ $[M+Na]^+$: 692.5625, Found: 692.5629.



HOTAG3 **Fmoc-Ala-OTAG3**, 85%

5-((Diisobutyl(octadecyl)silyl)oxy)pentan-2-yl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG3) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG3** (99.8 mg, 0.20 mmol, 1.0 equiv) was added dichloromethane (2.0 mL). The Fmoc-Ala-OH (186.8 mg, 0.6 mmol, 3.0 equiv) was added, followed by adding DMAP (29.3 mg, 0.24 mmol, 1.2 equiv) and DCC (123.8 mg, 0.6 mmol, 3.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 30:1 to 20:1 to 10:1) to afford the product **Fmoc-Ala-OTAG3** as a pale yellow oil in 85% yield (135.0 mg).

Rf = 0.44 (hexanes/EtOAc = 5:1).

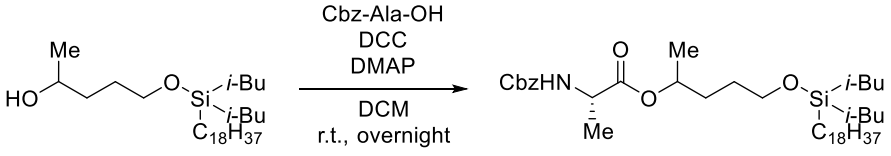
$[\alpha]_D^{26}$ = -5.21 (*c* 0.96, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.65 – 7.57 (m, 2H), 7.45 – 7.37 (m, 2H), 7.36 – 7.28 (m, 2H), 5.41 (d, *J* = 7.3 Hz, 1H), 5.05 – 4.93 (m, 1H), 4.47 – 4.29 (m, 3H), 4.28 – 4.19 (m, 1H), 3.63 – 3.52 (m, 2H), 1.89 – 1.74 (m, 2H), 1.70 – 1.47 (m, 4H), 1.47 – 1.39 (m, 3H), 1.35 – 1.21 (m, 35H), 0.99 – 0.92 (m, 12H), 0.92 – 0.85 (m, 3H), 0.66 – 0.54 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 172.7, 172.6, 155.5(2C), 143.9, 143.8, 141.3(2C), 127.7(2C), 127.0(2C), 125.1(2C), 119.9(2C), 72.42, 72.38, 66.9(2C), 62.0(2C), 49.8, 49.7, 47.1(2C), 33.8(2C), 32.3, 32.2, 31.9(2C), 29.7(16C), 29.64(2C), 29.61(2C), 29.35(2C), 29.30(2C), 28.53, 28.49, 26.4, 26.3, 24.5(2C), 24.2(2C), 23.3(2C), 22.7(2C), 20.0, 19.8, 18.9, 18.8, 14.9(2C), 14.1(2C).

IR (neat) 2951, 2922, 2852, 1725, 1506, 1450, 1380, 1331, 1247, 1210 cm^{-1}.

HRMS (ESI) Calcd for $C_{49}H_{81}NO_5SiNa$ $[M+Na]^+$: 814.5782, Found: 814.5741.



HOTAG3 **Cbz-Ala-OTAG3**, 98%

5-((Diisobutyl(octadecyl)silyl)oxy)pentan-2-yl ((benzyloxy)carbonyl)-L-alaninate (Cbz-Ala-OTAG3) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG3** (99.8 mg, 0.2 mmol, 1.0 equiv) was added dichloromethane (2.0 mL). The Cbz-Ala-OH (133.9 mg, 0.6 mmol, 3.0 equiv) was added, followed by adding DMAP (29.3 mg, 0.24 mmol, 1.2 equiv) and DCC (123.8 mg, 0.6 mmol, 3.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and

concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1) to afford the product **Cbz-Ala-OTAG3** as a colorless oil in 98% yield (137.9 mg).

Rf = 0.54 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{25}$ = -3.70 (*c* 1.08, $CHCl_3$).

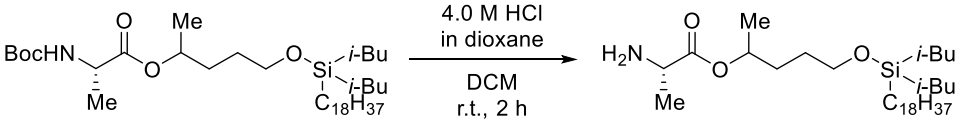
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 5H), 5.34 (d, *J* = 6.5 Hz, 1H), 5.14 – 5.09 (m, 2H), 5.01 – 4.90 (m, 1H), 4.40 – 4.28 (m, 1H), 3.61 – 3.52 (m, 2H), 1.88 – 1.73 (m, 2H), 1.66 – 1.46 (m, 4H), 1.44 – 1.37 (m, 3H), 1.35 – 1.20 (m, 35H), 0.98 – 0.92 (m, 12H), 0.92 – 0.84 (m, 3H), 0.65 – 0.54 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 172.6, 172.5, 155.5(2C), 136.3(2C), 128.5(2C), 128.11(2C), 128.07(2C), 72.35, 72.30, 66.8(2C), 62.0(2C), 49.8, 49.7, 33.8(2C), 32.3, 32.2, 31.9(2C), 29.7(16C), 29.64(2C), 29.60(2C), 29.35(2C), 29.30(2C), 28.51, 28.47, 26.3(2C), 24.6(2C), 24.2(2C), 23.3(2C), 22.7(2C), 19.9, 19.8, 18.9, 18.8, 14.9(2C), 14.1(2C).

IR (neat) 2950, 2922, 2853, 1726, 1455, 1331, 1247, 1212, 1184 cm^{-1}.

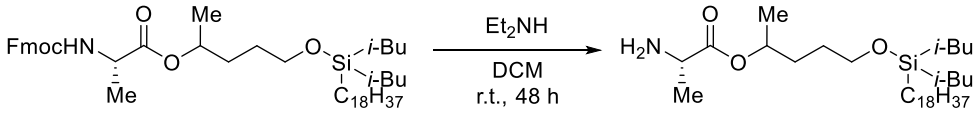
HRMS (ESI) Calcd for $C_{42}H_{77}NO_5SiNa$ $[M+Na]^+$: 726.5469, Found: 726.5460.

Tolerance test.



Boc-Ala-OTAG3 **H-Ala-OTAG3**, 0%

5-((Diisobutyl(octadecyl)silyl)oxy)pentan-2-yl L-alaninate (H-Ala-OTAG3) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Boc-Ala-OTAG3** (14.8 mg, 0.022 mmol, 1.0 equiv) was added dichloromethane (0.1 mL). The hydrochloric acid solution (4.0 M in dioxane, 0.04 mL, 0.16 mmol, 8.0 equiv) was added. The reaction was stirred at room temperature for 2 h. Then, 5 mL dichloromethane and 5 mL saturated $NaHCO_3$ solution were added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. No product **H-Ala-OTAG3** was detected from the crude ^{1}H NMR of the residue.



Fmoc-Ala-OTAG3 **H-Ala-OTAG3**, 76%

5-((Diisobutyl(octadecyl)silyl)oxy)pentan-2-yl L-alaninate (H-Ala-OTAG3) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAG3** (107.0 mg, 0.14 mmol, 1.0 equiv) was added dichloromethane (0.3 mL). The diethylamine (139.7 μL, 1.35 mmol, 10.0 equiv) was added. The reaction was stirred at room temperature for 48 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1 to 2:1) to afford the product **H-Ala-OTAG3** as a light grey oil in 76% yield (58.4 mg).

Rf = 0.22 (hexanes/EtOAc = 1:1).

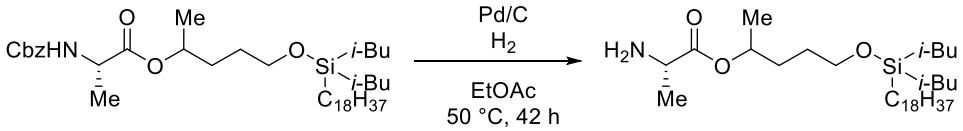
$[\alpha]_D^{27}$ = +26.81 (*c* 0.97, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.99 – 4.88 (m, 1H), 3.62 – 3.54 (m, 2H), 3.54 – 3.45 (m, 1H), 1.88 – 1.73 (m, 2H), 1.66 – 1.45 (m, 4H), 1.35 – 1.20 (m, 38H), 0.98 – 0.92 (m, 12H), 0.91 – 0.85 (m, 3H), 0.64 – 0.54 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 176.2(2C), 71.4(2C), 62.1(2C), 50.2, 50.1, 33.7(2C), 32.34, 32.27, 31.9(2C), 29.7(16C), 29.63(2C), 29.59(2C), 29.34(2C), 29.29(2C), 28.6(2C), 26.3(2C), 24.6(2C), 24.2(2C), 23.3(2C), 22.7(2C), 20.7, 20.6, 20.0, 19.9, 14.9(2C), 14.1(2C).

IR (neat) 2951, 2922, 2853, 1733, 1462, 1379, 1364, 1326, 1216, 1187, 1141, 1088 cm^{-1}.

HRMS (ESI) Calcd for $C_{34}H_{71}NO_3SiNa$ $[M+Na]^+$: 592.5101, Found: 592.5148.

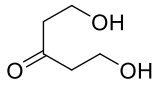


Cbz-Ala-OTAG3 **H-Ala-OTAG3**, 93%

5-((Diisobutyl(octadecyl)silyl)oxy)pentan-2-yl L-alaninate (H-Ala-OTAG3) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-OTAG3** (135.9 mg, 0.19 mmol, 1.0 equiv) was added EtOAc (2.0 mL). The 10% Pd/C (20.5 mg, 0.019 mmol, 0.1 equiv) was added. Then evacuated and backfilled with hydrogen (this process was repeated in total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 °C for 42 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (5 mL) and the filtrate was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 2:1) to afford the product **H-Ala-OTAG3** as a light grey oil in 93% yield (102.3 mg).

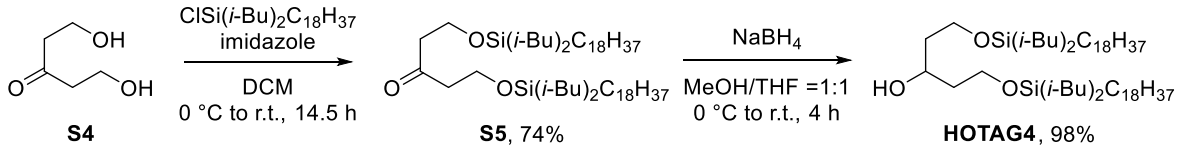
V. Synthesis of TAG4 and initial requirements test

Synthesis of TAG4.



S4

1,5-Dihydroxypentan-3-one (S4) was prepared from diethyl 3-oxopentanedioate in three steps according to the procedure in the literature and the characterization data match the reported data.[4]



19,19,27,27-Tetraisobutyl-20,26-dioxa-19,27-disilapentatetracontan-23-ol (HOTAG4) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S4** (945.1 mg, 8.0 mmol, 1.0 equiv) was added dichloromethane (40 mL). The imidazole (1.63 g, 24.0 mmol, 3.0 equiv) was added, followed by adding chlorodiisobutyl(octadecyl)silane (8.02 mL, 16.0 mmol, 2.0 equiv). The reaction was stirred at room temperature for 14.5 h. Then, water (40 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×40 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 80:1) to afford the product **S5** as a colorless oil in 74% yield (5.35 g).

At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S5** (5.35 g, 5.9 mmol, 1.0 equiv) was added methanol (25 mL) and tetrahydrofuran (25 mL). Cool the mixture to 0 °C, followed by adding sodium borohydride (446.1 mg, 11.8 mmol, 2.0 equiv). The reaction was stirred at room temperature for 4 h. Then, 40 mL saturated NH_4Cl solution and 40 mL EtOAc were added, and the layers were separated. The aqueous layer was extracted with EtOAc (3×40 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 70:1) to afford the product **HOTAG4** as a colorless oil in 98% yield (5.23 g).

Rf = 0.29 (hexanes/EtOAc = 20:1).

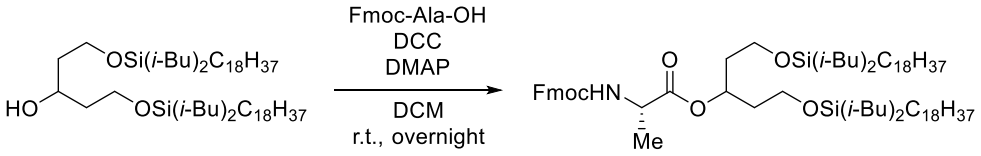
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.02 – 3.90 (m, 1H), 3.86 – 3.68 (m, 5H), 1.88 – 1.73 (m, 4H), 1.73 – 1.57 (m, 4H), 1.32 – 1.23 (m, 64H), 1.00 – 0.92 (m, 24H), 0.92 – 0.84 (m, 6H), 0.66 – 0.56 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 70.0, 61.1, 39.2, 33.8, 31.9, 29.72(8C), 29.68, 29.6, 29.4, 29.3, 26.4, 24.5, 24.2, 23.3, 22.7, 14.8, 14.1.

IR (neat) 2951, 2921, 2852, 1464, 1380, 1364, 1328, 1218, 1083 cm^{-1}.

HRMS (ESI) Calcd for $C_{57}H_{120}O_3Si_2Na$ $[M+Na]^+$: 931.8674, Found: 931.8642.

Esterification test.



HOTAG4 **Fmoc-Ala-OTAG4**, 83%

19,19,27,27-Tetraisobutyl-20,26-dioxa-19,27-disilapentatetracontan-23-yl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG4) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG4** (272.9 mg, 0.30 mmol, 1.0 equiv) was added dichloromethane (3.0 mL). The Fmoc-Ala-OH (233.5 mg, 0.75 mmol, 2.5 equiv) was added, followed by adding DMAP (44.0 mg, 0.36 mmol, 1.2 equiv) and DCC (154.8 mg, 0.75 mmol, 2.5 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 60:1 to 20:1) to afford the product **Fmoc-Ala-OTAG4** as a pale yellow oil in 83% yield (299.1 mg).

Rf = 0.23 (hexanes/EtOAc = 20:1).

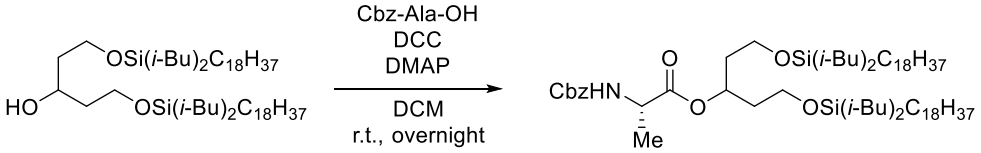
$[\alpha]_D^{24}$ = +6.31 (*c* 1.11, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.64 – 7.56 (m, 2H), 7.45 – 7.35 (m, 2H), 7.35 – 7.27 (m, 2H), 5.43 (d, *J* = 7.4 Hz, 1H), 5.21 – 5.08 (m, 1H), 4.44 – 4.31 (m, 3H), 4.28 – 4.17 (m, 1H), 3.67 – 3.52 (m, 4H), 1.87 – 1.75 (m, 8H), 1.43 (d, *J* = 7.1 Hz, 3H), 1.31 – 1.22 (m, 64H), 0.97 – 0.90 (m, 24H), 0.90 – 0.85 (m, 6H), 0.65 – 0.49 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 155.4, 143.9, 143.8, 141.3, 127.7, 127.0, 125.1, 119.9, 71.2, 67.0, 58.8, 49.7, 47.2, 37.2, 33.8, 31.9, 29.71(8C), 29.66(2C), 29.4, 29.3, 26.4, 24.5, 24.2, 23.3, 22.7, 19.0, 14.8, 14.1.

IR (neat) 2951, 2921, 2852, 1728, 1504, 1450, 1380, 1364, 1330, 1206, 1086 cm^{-1}.

HRMS (ESI) Calcd for $C_{75}H_{135}NO_6Si_2Na$ $[M+Na]^+$: 1224.9726, Found: 1224.9705.



HOTAG4 **Cbz-Ala-OTAG4**, 98%

19,19,27,27-Tetraisobutyl-20,26-dioxa-19,27-disilapentatetracontan-23-yl ((benzyloxy)carbonyl)-L-alaninate (Cbz-Ala-OTAG4) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **HOTAG4** (2.28 g, 2.5 mmol, 1.0 equiv) was added dichloromethane (25 mL). The Cbz-Ala-OH (1.40 g, 6.25 mmol, 2.5 equiv) was added, followed by adding DMAP (366.5 mg, 3.0 mmol, 1.2 equiv) and DCC (1.29 g, 6.25 mmol, 2.5 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (10 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 40:1 to 20:1) to afford the product **Cbz-Ala-OTAG4** as a colorless oil in 98% yield (2.73 g).

Rf = 0.20 (hexanes/EtOAc = 20:1).

$[\alpha]_D^{24}$ = +5.50 (*c* 1.09, $CHCl_3$).

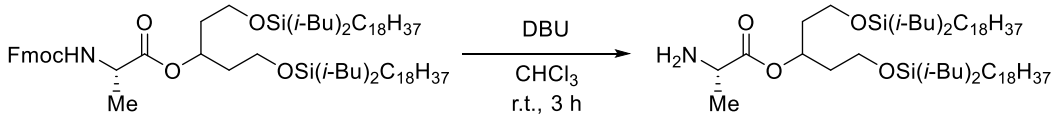
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.32 (m, 5H), 5.38 (d, *J* = 7.4 Hz, 1H), 5.17 – 5.07 (m, 3H), 4.39 – 4.29 (m, 1H), 3.65 – 3.54 (m, 4H), 1.85 – 1.71 (m, 8H), 1.41 (d, *J* = 7.0 Hz, 3H), 1.30 – 1.23 (m, 64H), 0.97 – 0.91 (m, 24H), 0.91 – 0.85 (m, 6H), 0.62 – 0.53 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 155.4, 136.3, 128.5, 128.11, 128.07, 71.1, 66.8, 58.8, 49.7, 37.2, 33.8, 31.9, 29.72(8C), 29.66(2C), 29.4, 29.3, 26.4, 24.5, 24.2, 23.3, 22.7, 19.0, 14.8, 14.1.

IR (neat) 2951, 2921, 2852, 1728, 1502, 1462, 1380, 1364, 1330, 1308, 1214, 1181 cm^{-1}.

HRMS (ESI) Calcd for $C_{68}H_{131}NO_6Si_2Na$ $[M+Na]^+$: 1136.9413, Found: 1136.9432.

Tolerance test.



Fmoc-Ala-OTAG4 **H-Ala-OTAG4**, 96%

19,19,27,27-Tetraisobutyl-20,26-dioxa-19,27-disilapentatetracontan-23-yl L-alaninate (H-Ala-OTAG4) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAG4** (132.2 mg, 0.11 mmol, 1.0 equiv) was added

chloroform (1.1 mL). The DBU (16.4 μL, 0.11 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 5:1) to afford the product **H-Ala-OTAG4** as a colorless oil in 96% yield (103.5 mg). Rf = 0.64 (hexanes/EtOAc = 2:1).

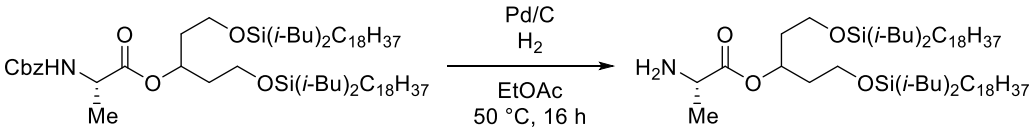
$[\alpha]_D^{24}$ = +2.91 (*c* 1.03, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.13 – 5.01 (m, 1H), 3.64 – 3.54 (m, 4H), 3.48 (q, *J* = 7.1 Hz, 1H), 1.87 – 1.72 (m, 8H), 1.35 – 1.19 (m, 67H), 0.97 – 0.91 (m, 24H), 0.91 – 0.84 (m, 6H), 0.63 – 0.53 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 70.1, 58.9, 50.2, 37.3, 33.8, 31.9, 29.7(8C), 29.6(2C), 29.4, 29.3, 26.3, 24.5, 24.2, 23.3, 22.7, 20.7, 14.8, 14.1.

IR (neat) 2952, 2921, 2852, 1736, 1626, 1463, 1380, 1364, 1327, 1217, 1185, 1088 cm^{-1}.

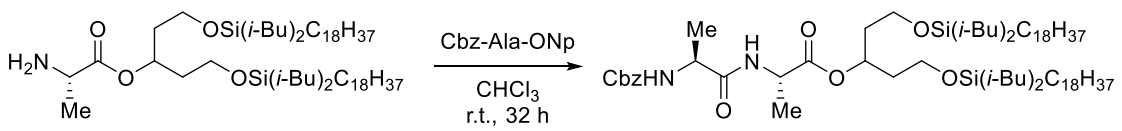
HRMS (ESI) Calcd for $C_{60}H_{125}NO_4Si_2Na$ $[M+Na]^+$: 1002.9045, Found: 1002.9028.



Cbz-Ala-OTAG4 **H-Ala-OTAG4**, 93%

19,19,27,27-Tetraisobutyl-20,26-dioxa-19,27-disilapentatetracontan-23-yl L-alaninate (H-Ala-OTAG4) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-OTAG4** (2.72 g, 2.44 mmol, 1.0 equiv) was added EtOAc (24 mL). The 10% Pd/C (259.7 mg, 0.244 mmol, 0.1 equiv) was added. Then evacuated and backfilled with hydrogen (this process was repeated a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 °C for 16 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (10 mL) and the filtrate was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 5:1) to afford the product **H-Ala-TAG4** as a colorless oil in 93% yield (2.24 g).

Elongation test (dipeptide synthesis).



H-Ala-OTAG4 **1**, 93%

Cbz-Ala-Ala-OTAG4 (1) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **H-Ala-OTAG4** (445.6 mg, 0.45 mmol, 1.0 equiv) was added chloroform (1.0 mL). The Cbz-Ala-ONp (312.8 mg, 0.9 mmol, 2.0 equiv) was added. The resulting mixture was stirred under room temperature for 32 h. After completion, the mixture was diluted with dichloromethane (5 mL). Saturated Na_2CO_3 solution (5 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were added 15 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 12.5:1 to 7:1) to afford the product **1** as a pale yellow oil in 93% yield (499.2 mg).

Rf = 0.38 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{24}$ = +3.00 (*c* 1.00, $CHCl_3$).

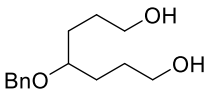
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 5H), 6.43 (d, *J* = 6.2 Hz, 1H), 5.38 – 5.25 (m, 1H), 5.14 – 5.07 (m, 3H), 4.55 – 4.46 (m, 1H), 4.30 – 4.18 (m, 1H), 3.64 – 3.53 (m, 4H), 1.88 – 1.72 (m, 8H), 1.42 – 1.36 (m, 6H), 1.33 – 1.21 (m, 64H), 0.97 – 0.91 (m, 24H), 0.91 – 0.85 (m, 6H), 0.62 – 0.53 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 171.4, 155.8, 136.2, 128.5, 128.1, 128.0, 71.3, 67.0, 58.7, 50.4, 48.3, 37.1, 33.8, 31.9, 29.7(8C), 29.6(2C), 29.35, 29.32, 26.3, 24.5, 24.2, 23.3, 22.7, 18.8, 18.5, 14.8, 14.1.

IR (neat) 3316, 2951, 2921, 2852, 1736, 1666, 1534, 1503, 1456, 1327, 1215, 1087 cm^{-1}.

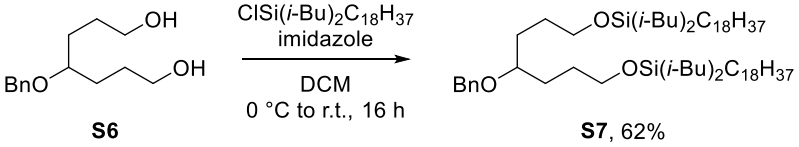
HRMS (ESI) Calcd for $C_{71}H_{136}N_2O_7Si_2Na$ $[M+Na]^+$: 1207.9784, Found: 1207.9770.

VI. Synthesis of TAG5 and H-Ala-OTAG5



S6

4-(Benzyloxy)heptane-1,7-diol (S6) was prepared according to the procedure in the literature and the characterization data match the reported data.[5]



S6 **S7**, 62%

24-(Benzyloxy)-19,19,29,29-tetraisobutyl-20,28-dioxa-19,29-disilaheptatetracontane (S7) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S6** (250.2 mg, 1.05 mmol, 1.0 equiv) was added dichloromethane (5 mL). The imidazole (204.2 g, 3.0 mmol, 2.9 equiv) was added, followed by adding chlorodiisobutyl(octadecyl)silane (1.00 mL, 2.0 mmol, 1.9 equiv). The reaction was stirred at room temperature for 16 h. Then, water (10 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 100:1) to afford the product **S7** as a colorless oil in 62% yield (635.1 mg).

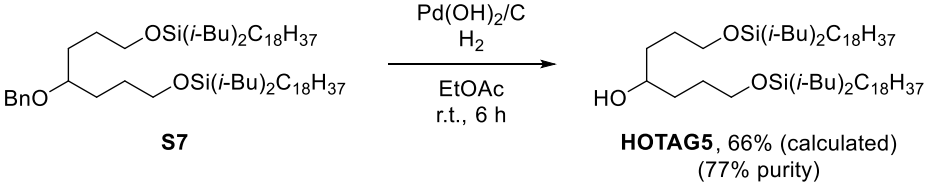
Rf = 0.41 (hexanes/EtOAc = 20:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.30 (m, 4H), 7.30 – 7.23 (m, 1H), 4.50 (s, 2H), 3.64 – 3.53 (m, 4H), 3.47 – 3.38 (m, 1H), 1.90 – 1.75 (m, 4H), 1.66 – 1.51 (m, 8H), 1.36 – 1.23 (m, 64H), 0.99 – 0.93 (m, 24H), 0.93 – 0.85 (m, 6H), 0.66 – 0.54 (m, 12H).

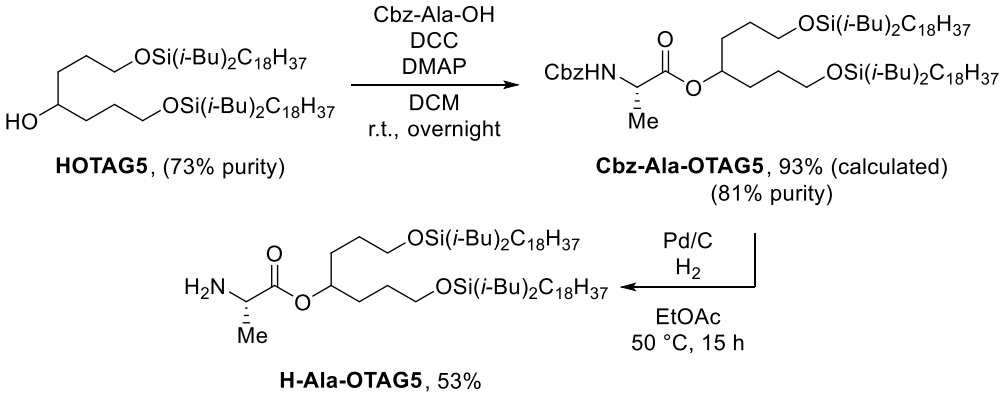
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 128.3, 127.7, 127.4, 78.7, 70.6, 62.7, 33.8, 31.9, 30.1, 29.72(8C), 29.67(2C), 29.4, 29.3, 28.6, 26.41, 26.38, 24.6, 24.3, 23.4, 22.7, 15.0, 14.1.

IR (neat) 2951, 2921, 2852, 1464, 1380, 1364, 1328, 1217, 1163, 1088 cm^{-1}.

HRMS (ESI) Calcd for $C_{66}H_{130}O_3Si_2Na$ $[M+Na]^+$: 1049.9456, Found: 1049.9450.



S7 **HOTAG5**, 66% (calculated) (77% purity)



19,19,29,29-Tetraisobutyl-20,28-dioxa-19,29-disilaheptatetracontan-24-yl L-alaninate (H-Ala-OTAG5) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S7** (123.4 mg, 0.12 mmol, 1.0 equiv) was added EtOAc (1.5 mL). The 20% $Pd(OH)_2/C$ (8.4 mg, 0.012 mmol, 0.1 equiv) was added. Then evacuated and backfilled with hydrogen (this process was repeated a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at room temperature for 6 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (5 mL) and the filtrate was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 40:1) to afford the product **HOTAG5** as a colorless oil in 66% yield (calculated yield, 95.7 mg, 77% purity). The product was put into next step without further purification.

At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG5** (73% purity, 181.5 mg, 0.14 mmol, 1.0 equiv) was added dichloromethane (1.4 mL). The Cbz-Ala-OH (78.1 g, 0.35 mmol, 2.5 equiv) was added, followed by adding DMAP (20.5 mg, 0.17 mmol, 1.2 equiv) and DCC (72.2 mg, 0.35 mmol, 2.5 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1) to afford the product **Cbz-Ala-OTAG5** as a colorless oil in 93% yield (calculated yield, 184.9 mg, 81% purity). The product was put into next step without further purification.

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-OTAG5** (81% purity, 148.0 mg, 0.104 mmol, 1.0 equiv) was added EtOAc (2.0 mL). The 10% Pd/C (10.6 mg, 0.01 mmol, 0.1 equiv) was added. Then evacuated and backfilled with hydrogen (this process was repeated in total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 °C for 15 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (5 mL) and the filtrate was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 5:1) to afford the product **H-Ala-OTAG5** as a pale yellow oil in 53% yield (56.1 mg).

Rf = 0.58 (hexanes/EtOAc = 2:1).

$[\alpha]_D^{24}$ = +4.17 (*c* 0.96, $CHCl_3$).

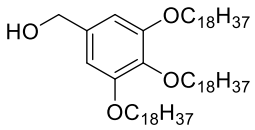
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.99 – 4.89 (m, 1H), 3.60 – 3.47 (m, 5H), 1.87 – 1.72 (m, 4H), 1.67 – 1.44 (m, 8H), 1.36 – 1.22 (m, 67H), 1.00 – 0.91 (m, 24H), 0.91 – 0.84 (m, 6H), 0.64 – 0.52 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.3, 74.5, 62.2, 50.2, 33.8, 31.9, 30.6, 29.70(8C), 29.66(2C), 29.4, 29.3, 28.5, 26.4, 26.3, 24.6, 24.2, 23.3, 22.7, 20.7, 14.9, 14.1.

IR (neat) 2951, 2921, 2852, 1734, 1463, 1380, 1364, 1327, 1217, 1185, 1089 cm^{-1}.

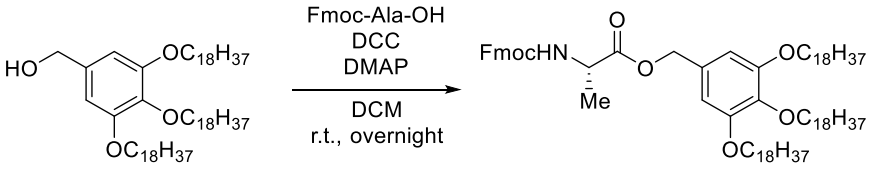
HRMS (ESI) Calcd for $C_{62}H_{129}NO_4Si_2Na$ $[M+Na]^+$: 1030.9358, Found: 1030.9355.

VII. Synthesis of H-Ala-OTAGTamiaki



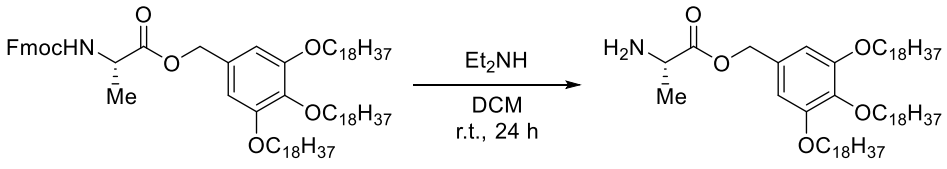
HOTAGTamiaki

(3,4,5-Tris(octadecyloxy)phenyl)methanol (HOTAGTamiaki) was prepared according to the procedure in the literature and the characterization data match the reported data.[3]



HOTAGTamiaki **Fmoc-Ala-OTAGTamiaki**, 43%

3,4,5-Tris(octadecyloxy)benzyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAGTamiaki) was prepared from **HOTAGTamiaki** in one step according to the procedure in the literature with a slight modification.[3] At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAGTamiaki** (764.8 mg, 0.84 mmol, 1.0 equiv) was added dichloromethane (8.5 mL). The Fmoc-Ala-OH (651.6 mg, 2.1 mmol, 2.5 equiv) was added, followed by adding DMAP (122.2 mg, 1.0 mmol, 1.2 equiv) and DCC (431.2 mg, 2.1 mmol, 2.5 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (10 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1 to 10:1) to afford the product **Fmoc-Ala-OTAGTamiaki** as a white solid in 43% yield (432.3 mg). It is a known compound. The characterization data match the reported data.[3]



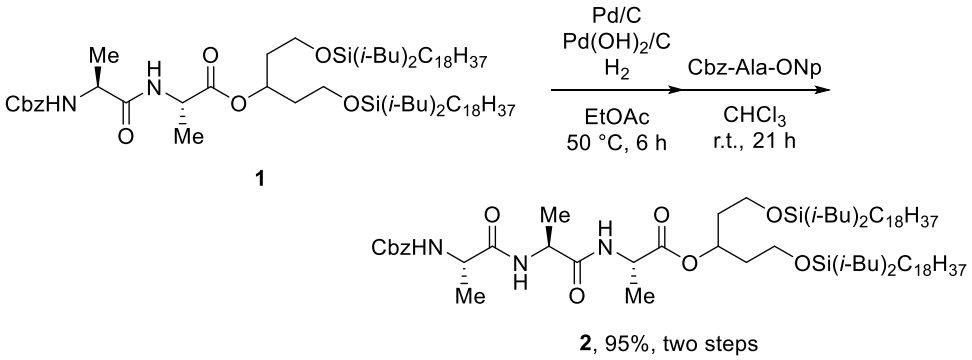
Fmoc-Ala-OTAGTamiaki **H-Ala-OTAGTamiaki**, 84%

3,4,5-Tris(octadecyloxy)benzyl L-alaninate (H-Ala-OTAGTamiaki) was prepared from **Fmoc-Ala-OTAGTamiaki** in one step according to the procedure in the literature with a slight modification.[3] At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAGTamiaki** (132.8 mg, 0.11 mmol, 1.0 equiv) was added dichloromethane (1.1 mL). The diethylamine (56.9 μL, 0.55 mmol, 5.0 equiv) was added. The

reaction was stirred at room temperature for 24 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1 to 1:1) to afford the product **H-Ala-OTAGTamiaki** as a white solid in 84% yield (90.7 mg). It is a known compound. The characterization data match the reported data.[3]

VIII. Comparison of short alanine chains with TAG4 and *t*-Bu as protecting groups

Short alanine chain with TAG4.



Cbz-Ala-Ala-Ala-OTAG4 (2) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **1** (749.0 mg, 0.63 mmol, 1.0 equiv) was added EtOAc (7 mL). The 10% Pd/C (67.2 mg, 0.063 mmol, 0.1 equiv) and 20% $Pd(OH)_2/C$ (33.6 mg, 0.063 mmol, 0.1 equiv) was added together. Then evacuated and backfilled with hydrogen (this process was repeated a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 ºC for 6 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (10 mL) and the filtrate was concentrated. The residue was put into next step without further purification.

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above residue was added chloroform (1.3 mL). The Cbz-Ala-ONp (434.9 mg, 1.26 mmol, 2.0 equiv) was added. The reaction was stirred under room temperature for 21 h. After completion, the mixture was diluted with dichloromethane (7 mL), followed by adding 2-aminoethanol (200 μL) and stirring under room temperature for 30 min to remove the excess Cbz-Ala-ONp. Saturated Na_2CO_3 solution (10 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were added 30 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1 to 2:1) to afford the product **2** as a pale yellow oil in 95% total yield (758.1 mg).

Rf = 0.74 (hexanes/EtOAc = 1:1).

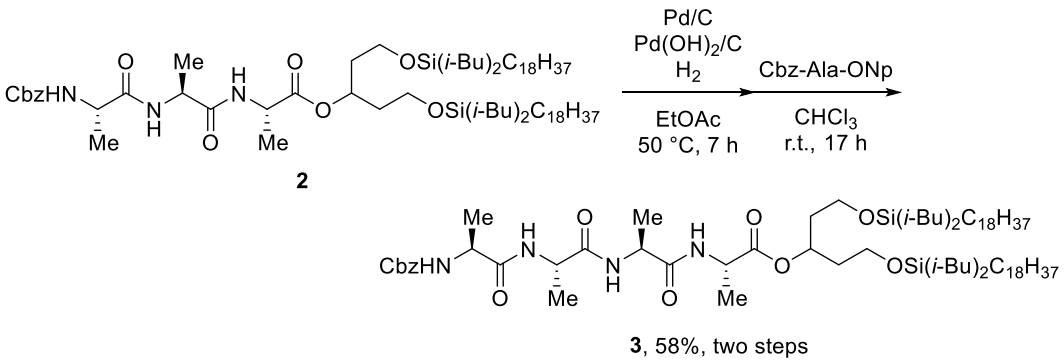
$[\alpha]_D^{24}$ = -2.54 (*c* 1.18, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 5H), 6.80 (d, J = 7.3 Hz, 1H), 6.72 (d, J = 7.2 Hz, 1H), 5.51 (d, J = 7.6 Hz, 1H), 5.15 – 5.04 (m, 3H), 4.56 – 4.43 (m, 2H), 4.36 – 4.22 (m, 1H), 3.65 – 3.51 (m, 4H), 1.91 – 1.71 (m, 8H), 1.43 – 1.35 (m, 9H), 1.35 – 1.23 (m, 64H), 0.99 – 0.90 (m, 24H), 0.90 – 0.83 (m, 6H), 0.63 – 0.51 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0(2C), 171.2, 155.9, 136.2, 128.5, 128.2, 128.0, 71.3, 67.0, 58.7, 50.5, 48.8, 48.3, 37.1, 33.8, 31.9, 29.7(8C), 29.6(2C), 29.34, 29.32, 26.3, 24.5, 24.2, 23.3, 22.7, 18.8, 18.48, 18.45, 14.8, 14.1.

IR (neat) 3286, 2952, 2922, 2853, 1737, 1705, 1640, 1520, 1454, 1379, 1364, 1328, 1216, 1162, 1088, 1045 cm^{-1}.

HRMS (ESI) Calcd for $C_{74}H_{141}N_3O_8Si_2Na$ $[M+Na]^+$: 1279.0155, Found: 1279.0139.



Cbz-Ala-Ala-Ala-Ala-OTAG4 (3) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **2** (251.4 mg, 0.2 mmol, 1.0 equiv) was added EtOAc (2 mL). The 10% Pd/C (21.3 mg, 0.02 mmol, 0.1 equiv) and 20% $Pd(OH)_2/C$ (10.6 mg, 0.02 mmol, 0.1 equiv) was added together. Then evacuated and backfilled with hydrogen (this process was repeated a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 ºC for 7 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (5 mL) and the filtrate was concentrated. The residue was put into next step without further purification.

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above residue was added chloroform (0.4 mL). The Cbz-Ala-ONp (137.7 mg, 0.4 mmol, 2.0 equiv) was added. The reaction was stirred under room temperature for 17 h. After completion, the mixture was diluted with dichloromethane (5 mL). Saturated Na_2CO_3 solution (5 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were added 15 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and

concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 3:1 to 1:1) to afford the product **3** as a pale yellow wax in 58% total yield (153.1 mg).

Rf = 0.26 (hexanes/EtOAc = 1:1).

M.p. 149-150 °C.

$[\alpha]_D^{24}$ = -11.34 (*c* 0.97, $CHCl_3$).

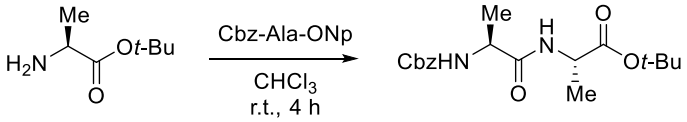
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.8 Hz, 1H), 7.44 – 7.36 (m, 1H), 7.36 – 7.26 (m, 6H), 6.09 (d, *J* = 7.7 Hz, 1H), 5.15 – 5.09 (m, 2H), 5.09 – 4.99 (m, 1H), 4.88 – 4.75 (m, 1H), 4.75 – 4.65 (m, 1H), 4.65 – 4.56 (m, 1H), 4.50 (p, *J* = 7.2 Hz, 1H), 3.64 – 3.49 (m, 4H), 1.86 – 1.71 (m, 8H), 1.47 – 1.19 (m, 76H), 0.97 – 0.90 (m, 24H), 0.90 – 0.83 (m, 6H), 0.63 – 0.51 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 172.0, 171.75, 171.66, 156.0, 136.3, 128.5, 128.1, 127.8, 71.2, 66.8, 58.8, 50.5, 49.0, 48.8, 48.2, 37.1, 33.8, 31.9, 29.7(8C), 29.6(2C), 29.3(2C), 26.3, 24.4, 24.2, 23.3, 22.7, 20.0, 19.7, 19.5, 18.4, 14.8, 14.1.

IR (neat) 3275, 2952, 2922, 2853, 1737, 1709, 1674, 1631, 1526, 1455, 1364, 1256, 1217, 1163, 1088 cm^{-1}.

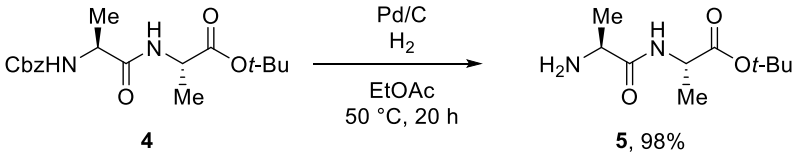
HRMS (ESI) Calcd for $C_{77}H_{146}N_4O_9Si_2Na$ $[M+Na]^+$: 1350.0526, Found: 1350.0530.

Short alanine chain with *t*-Bu.

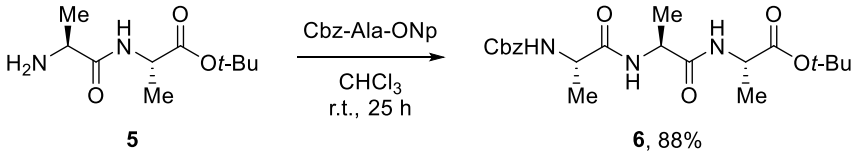


H-Ala-O*t*-Bu **4**, 98%

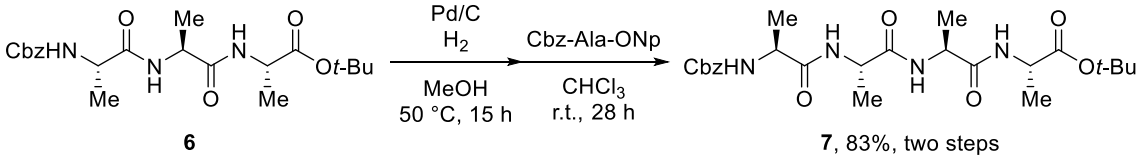
Cbz-Ala-Ala-O*t*-Bu (4) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **H-Ala-O*t*-Bu** (290.4 mg, 2.0 mmol, 1.0 equiv) was added chloroform (2.0 mL). The Cbz-Ala-ONp (1.38 g, 4.0 mmol, 2.0 equiv) was added. The resulting mixture was stirred under room temperature for 4 h. After completion, the mixture was diluted with dichloromethane (5 mL). Saturated Na_2CO_3 solution (10 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 4:1 to 2:1) to afford the product **4** as a yellow solid in 98% yield (684.1 mg). It is a known compound. The characterization data match the reported data.[6]



H-Ala-Ala-O*t*-Bu (5) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **7** (849.6 mg, 2.4 mmol, 1.0 equiv) was added EtOAc (24 mL). The 10% Pd/C (258.1 mg, 0.24 mmol, 0.1 equiv) was added. Then evacuated and backfilled with hydrogen (this process was repeated a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 ºC for 20 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (10 mL) and the filtrate was concentrated. The residue was purified by silica gel chromatography (eluent: DCM/MeOH = 10:1) to afford the product **4** as a pale yellow solid in 98% yield (512.0 mg). It is a known compound. The characterization data match the reported data.[6]



Cbz-Ala-Ala-Ala-O*t*-Bu (6) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **5** (351.6 mg, 1.6 mmol, 1.0 equiv) was added chloroform (3.2 mL). The Cbz-Ala-ONp (1.12 g, 3.2 mmol, 2.0 equiv) was added. The resulting mixture was stirred under room temperature for 25 h. After completion, the mixture was diluted with dichloromethane (5 mL). Saturated Na_2CO_3 solution (10 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to 1:2) to afford the product **6** as a white solid in 88% yield (601.3 mg). It is a known compound. The characterization data match the reported data.[6]



Cbz-Ala-Ala-Ala-Ala-O*t*-Bu (7) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **6** (171.4 mg, 0.4 mmol, 1.0 equiv) was added MeOH (4

mL). The 10% Pd/C (42.6 mg, 0.04 mmol, 0.1 equiv) was added. Then evacuated and backfilled with hydrogen (this process was repeated a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 °C for 15 h. After completion, the reaction mixture was filtrated through a short pad of celite, washed with EtOAc (5 mL) and the filtrate was concentrated. The residue was put into next step without further purification.

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above residue was added chloroform (1.0 mL). The Cbz-Ala-ONp (275.4 mg, 0.8 mmol, 2.0 equiv) was added. The reaction was stirred at room temperature for 28 h. After completion, the mixture was transferred onto SiO_2 column by a pipette. The reaction mixture was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 20:1) to afford the product **7** as a white solid in 83% total yield (166.9 mg).

Rf = 0.33 (DCM/MeOH = 10:1).

M.p. 239-240 °C.

$[\alpha]_D^{27}$ = -1.87 (*c* 1.07, $CHCl_3$).

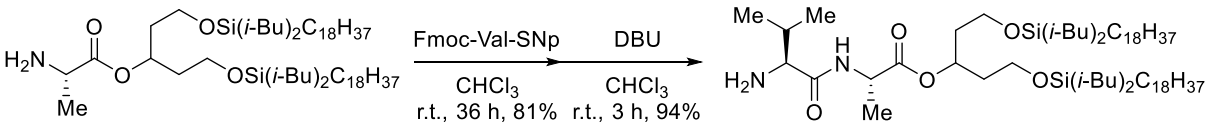
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 6H), 7.13 – 7.02 (m, 2H), 5.74 (d, *J* = 7.2 Hz, 1H), 5.11 (s, 2H), 4.74 – 4.65 (m, 1H), 4.62 – 4.54 (m, 1H), 4.49 – 4.37 (m, 2H), 1.44 (s, 9H), 1.43 – 1.32 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 172.1, 172.0, 171.9, 156.1, 136.6, 128.4, 127.9(2C), 81.6, 66.6, 50.4, 48.9, 48.8, 48.6, 27.9, 20.4, 20.3, 20.2, 18.4.

IR (neat) 3306, 2979, 2930, 1727, 1697, 1666, 1632, 1529, 1446, 1367, 1293, 1250, 1215, 1157, 1122 cm^{-1}.

HRMS (ESI) Calcd for $C_{24}H_{36}N_4O_7Na$ $[M+Na]^+$: 515.2482, Found: 515.2522.

IX. Synthesis of protected DRGN-1 with TAG4



H-Ala-OTAG4 **8**

H-Val-Ala-OTAG4 (8) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **H-Ala-OTAG4** (2.35 g, 2.40 mmol, 1.0 equiv) was added chloroform (4.8 mL). The Fmoc-Val-SNp (1.71 g, 3.60 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 36 h. After completion, the mixture was diluted with dichloromethane (20 mL). Saturated Na_2CO_3 solution (20 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 15:1 to 10:1) to afford the Fmoc protected peptide as a yellow oil in 81% yield (2.55 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (2.73 g, 2.10 mmol, 1.0 equiv) was added chloroform (21 mL). The DBU (313.4 μL, 2.10 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1 to 3:1 to 2:1 to 1:1) to afford the product **8** as a pale yellow oil in 93% yield (2.11 g).

Rf = 0.49 (hexanes/EtOAc = 1:1).

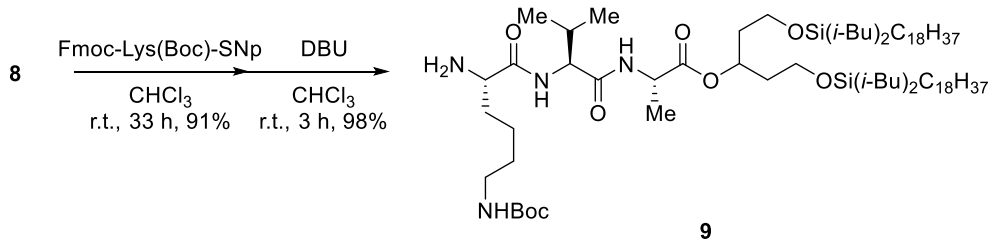
$[\alpha]_D^{23}$ = -13.72 (*c* 1.02, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.7 Hz, 1H), 5.10 (p, *J* = 5.9 Hz, 1H), 4.63 – 4.49 (m, 1H), 3.70 – 3.50 (m, 4H), 3.24 (d, *J* = 3.9 Hz, 1H), 2.37 – 2.21 (m, 1H), 1.91 – 1.71 (m, 8H), 1.40 (d, *J* = 7.0 Hz, 3H), 1.33 – 1.20 (m, 64H), 0.99 (d, *J* = 7.0 Hz, 3H), 0.96 – 0.90 (m, 24H), 0.90 – 0.85 (m, 6H), 0.83 (d, *J* = 6.9 Hz, 3H), 0.63 – 0.52 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 172.5, 71.0, 60.0, 58.8, 47.8, 37.2, 33.8, 31.9, 30.9, 29.70(8C), 29.66(2C), 29.4, 29.3, 26.4, 24.5, 24.2, 23.3, 22.7, 19.6, 18.8, 16.0, 14.9, 14.1.

IR (neat) 2952, 2921, 2852, 1740, 1677, 1504, 1463, 1380, 1364, 1328, 1217, 1162, 1087, 1039 cm^{-1}.

HRMS (ESI) Calcd for $C_{65}H_{134}N_2O_5Si_2Na$ $[M+Na]^+$: 1101.9729, Found: 1101.9703.



H-Lys(Boc)-Val-Ala-OTAG4 (9) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **8** (2.18 g, 2.02 mmol, 1.0 equiv) was added chloroform (6.1 mL). The Fmoc-Lys(Boc)-SNp (1.84 g, 3.03 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 33 h. After completion, the mixture was diluted with dichloromethane (20 mL), followed by adding 2-aminoethanol (500 μL) and stirring under room temperature for 15 min to remove the excess Fmoc-Lys(Boc)-SNp. Saturated Na_2CO_3 solution (20 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 6:1 to 3:1 to 2.5:1) to afford the Fmoc protected peptide as a yellow wax in 91% yield (2.83 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (2.71 g, 1.77 mmol, 1.0 equiv) was added chloroform (18 mL). The DBU (264.4 μL, 1.77 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to 1:2 to 100% EtOAc) to afford the product **9** as a pale yellow oil in 98% yield (2.26 g).

Rf = 0.11 (hexanes/EtOAc = 1:1).

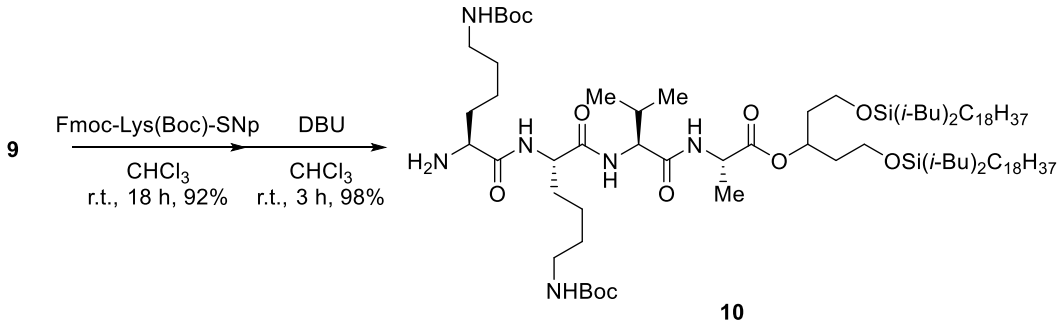
$[\alpha]_D^{23}$ = -4.76 (*c* 1.05, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 9.0 Hz, 1H), 6.41 (d, *J* = 7.2 Hz, 1H), 5.09 (p, *J* = 6.2 Hz, 1H), 4.63 – 4.43 (m, 2H), 4.20 (dd, *J* = 9.1, 6.4 Hz, 1H), 3.69 – 3.50 (m, 4H), 3.39 (dd, *J* = 8.0, 4.4 Hz, 1H), 3.19 – 3.03 (m, 2H), 2.22 – 2.06 (m, 1H), 1.92 – 1.71 (m, 9H), 1.61 – 1.45 (m, 3H), 1.46 – 1.35 (m, 14H), 1.35 – 1.18 (m, 64H), 0.99 – 0.90 (m, 30H), 0.90 – 0.82 (m, 6H), 0.62 – 0.52 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1, 172.1, 170.5, 156.0, 79.1, 71.3, 58.79, 58.75, 58.0, 55.1, 48.2, 40.1, 37.2, 34.6, 33.8, 31.9, 30.9, 29.9, 29.7(8C), 29.6(2C), 29.34, 29.32, 28.4, 26.4, 24.5, 24.2, 23.3, 22.9, 22.7, 19.3, 18.5, 18.0, 14.8, 14.1.

IR (neat) 3288, 2952, 2922, 2853, 1694, 1650, 1517, 1462, 1380, 1365, 1328, 1247, 1217, 1198, 1167, 1088, 1040 cm^{-1}.

HRMS (ESI) Calcd for $C_{76}H_{154}N_4O_8Si_2Na$ $[M+Na]^+$: 1330.1203, Found: 1330.1212.



H-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (10) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **9** (1.85 g, 1.41 mmol, 1.0 equiv) was added chloroform (4.3 mL). The Fmoc-Lys(Boc)-SNp (1.28 g, 2.12 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 18 h. After completion, the mixture was diluted with dichloromethane (15 mL). Saturated Na_2CO_3 solution (20 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 2:1 to DCM/EtOAc = 2:1) to afford the Fmoc protected peptide as a white solid in 92% yield (2.30 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (2.71 g, 1.54 mmol, 1.0 equiv) was added chloroform (16 mL). The DBU (230.1 μL, 1.54 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 10:1) to afford the product **10** as a pale yellow wax in 98% yield (2.32 g).

Rf = 0.25 (DCM/MeOH = 10:1).

M.p. 81-82 ºC.

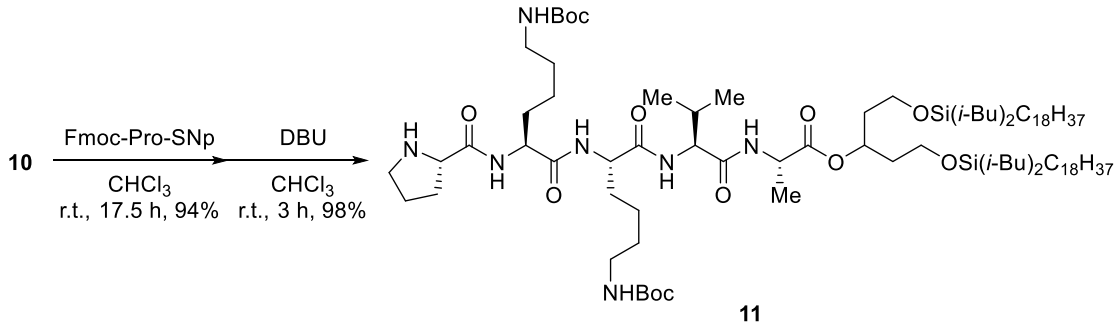
$[\alpha]_D^{23}$ = -8.70 (*c* 0.92, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.9 Hz, 1H), 6.79 (d, *J* = 8.5 Hz, 1H), 6.54 – 6.43 (m, 1H), 5.07 (p, *J* = 6.1 Hz, 1H), 4.89 – 4.76 (m, 1H), 4.76 – 4.61 (m, 1H), 4.57 – 4.44 (m, 1H), 4.44 – 4.35 (m, 1H), 4.22 (dd, *J* = 8.4, 6.2 Hz, 1H), 3.66 – 3.48 (m, 4H), 3.36 (dd, *J* = 7.9, 4.5 Hz, 1H), 3.19 – 2.99 (m, 4H), 2.20 – 2.05 (m, 1H), 1.92 – 1.72 (m, 10H), 1.69 – 1.57 (m, 2H), 1.54 – 1.45 (m, 4H), 1.45 – 1.33 (m, 25H), 1.33 – 1.18 (m, 64H), 0.98 – 0.88 (m, 30H), 0.89 – 0.82 (m, 6H), 0.64 – 0.49 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.5, 172.0, 171.8, 170.1, 156.1(2C), 79.02, 78.95, 71.3, 58.8, 58.7, 58.4, 54.9, 52.8, 48.1, 40.1(2C), 37.1, 34.5, 33.8, 31.9, 31.5, 30.9, 29.8, 29.7(8C), 29.6(2C), 29.5, 29.33, 29.30, 28.4(2C), 26.3, 24.5, 24.2, 23.3, 22.72(2C), 22.65, 19.1, 18.5, 17.9, 14.8, 14.1.

IR (neat) 3294, 2922, 2853, 1740, 1686, 1627, 1530, 1463, 1389, 1365, 1275, 1251, 1216, 1172, 1089, 1039 cm^{-1}.

HRMS (ESI) Calcd for $C_{87}H_{174}N_6O_{11}Si_2Na$ $[M+Na]^+$: 1558.2677, Found: 1558.2671.



H-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (11) At room tesmperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **10** (2.20 g, 1.41 mmol, 1.0 equiv) was added chloroform (4.2 mL). The Fmoc-Pro-SNp (996.5 mg, 2.10 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 17.5 h. After completion, the mixture was diluted with dichloromethane (15 mL). Saturated Na_2CO_3 solution (20 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc = 3:1 to 1:1 to 1:1.5 to DCM/acetone = 5:1) to afford the Fmoc protected peptide as a white solid in 94% yield (2.45 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (2.34 g, 1.26 mmol, 1.0 equiv) was added chloroform (13 mL). The DBU (188.3 μL, 1.26 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 10:1) to afford the product **11** as a yellow wax in 98% yield (2.01 g).

Rf = 0.33 (DCM/MeOH = 10:1).

M.p. 86-88 °C.

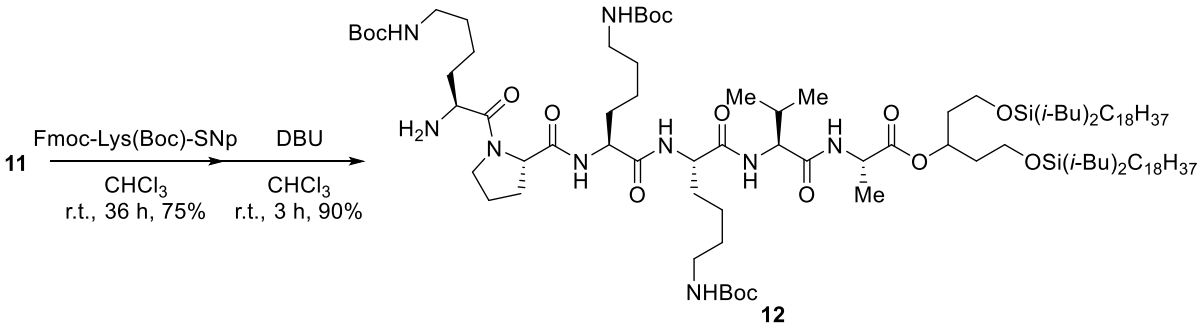
$[\alpha]_D^{23}$ = -21.51 (*c* 0.93, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.4 Hz, 1H), 7.63 – 7.45 (m, 1H), 7.23 – 7.02 (m, 1H), 6.70 (d, *J* = 6.3 Hz, 1H), 5.06 (p, *J* = 6.1 Hz, 1H), 4.99 – 4.87 (m, 1H), 4.88 – 4.72 (m, 1H), 4.66 – 4.29 (m, 4H), 3.71 (dd, *J* = 9.1, 5.2 Hz, 1H), 3.67 – 3.51 (m, 4H), 3.16 – 2.95 (m, 5H), 2.95 – 2.83 (m, 1H), 2.28 – 2.01 (m, 3H), 1.94 – 1.57 (m, 14H), 1.52 – 1.34 (m, 25H), 1.34 – 1.12 (m, 68H), 0.99 – 0.89 (m, 30H), 0.89 – 0.80 (m, 6H), 0.63 – 0.49 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$, lost three signals) δ 175.6, 172.0, 171.9, 171.4, 170.3, 156.1, 156.0, 78.9(2C), 71.1, 60.4, 58.9, 58.8, 58.0, 53.2, 52.3, 48.2, 47.2, 40.2(2C), 37.21, 37.16, 33.8, 32.21, 32.17, 31.9, 31.5, 30.8, 29.7(8C), 29.6(2C), 29.3(2C), 28.4(2C), 26.3, 26.2, 24.5, 24.2, 23.3, 22.7, 19.1, 18.2, 18.1, 14.8, 14.1.

IR (neat) 3278, 2922, 2854, 1740, 1687, 1631, 1526, 1463, 1391, 1365, 1275, 1250, 1216, 1171, 1089, 1039 cm^{-1}.

HRMS (ESI) Calcd for $C_{92}H_{181}N_7O_{12}Si_2Na$ $[M+Na]^+$: 1655.3204, Found: 1655.3175.



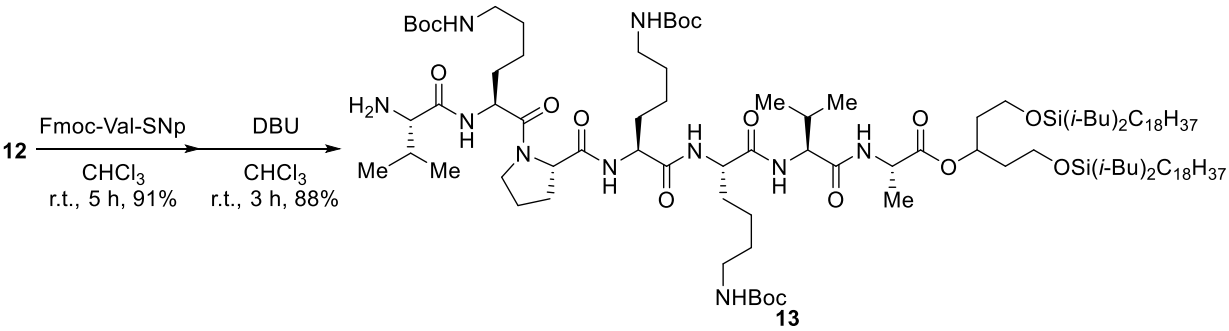
H-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (12) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **11** (1.89 g, 1.16 mmol, 1.0 equiv) was added chloroform (5.5 mL). The Fmoc-Lys(Boc)-SNp (1.05 g, 1.73 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 36 h. After completion, the mixture was diluted with dichloromethane (10 mL). Saturated Na_2CO_3 solution (15 mL)

was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were added 30 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc = 3:1 to 1:1.75 to DCM/acetone = 4:1 to 2:1) to afford the Fmoc protected peptide as a pale yellow solid in 75% yield (1.82 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (1.70 g, 0.82 mmol, 1.0 equiv) was added chloroform (8.2 mL). The DBU (122.5 μL, 0.82 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 8:1) to afford the product **12** as a pale yellow wax in 90% yield (1.38 g).

Rf = 0.28 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{103}H_{201}N_9O_{15}Si_2Na$ $[M+Na]^+$: 1883.4678, Found: 1883.4710.



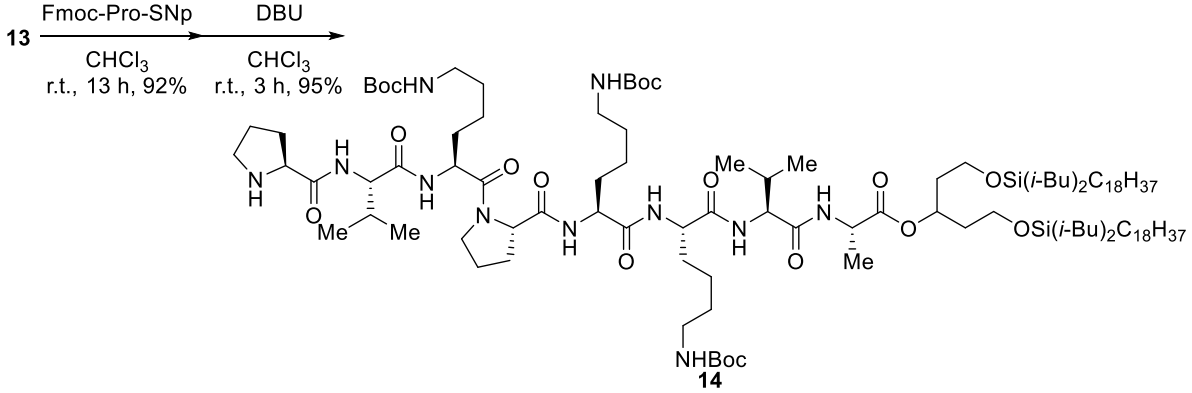
H-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (13) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **12** (1.18 g, 0.63 mmol, 1.0 equiv) was added chloroform (1.9 mL). The Fmoc-Val-SNp (452.7 g, 0.95 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 5 h. After completion, the mixture was diluted with dichloromethane (5 mL). Saturated Na_2CO_3 solution (10 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were added 20 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc =

3:1 to DCM/acetone = 3:1 to 2:1 to 1.5:1) to afford the Fmoc protected peptide as a light green solid in 91% yield (1.26 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (1.14 g, 0.52 mmol, 1.0 equiv) was added chloroform (5.2 mL). The DBU (77.7 μL, 0.52 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 7:1) to afford the product **13** as a white solid in 88% yield (1.38 g).

Rf = 0.39 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{108}H_{210}N_{10}O_{16}Si_2Na$ $[M+Na]^+$: 1982.5363, Found: 1982.5409.



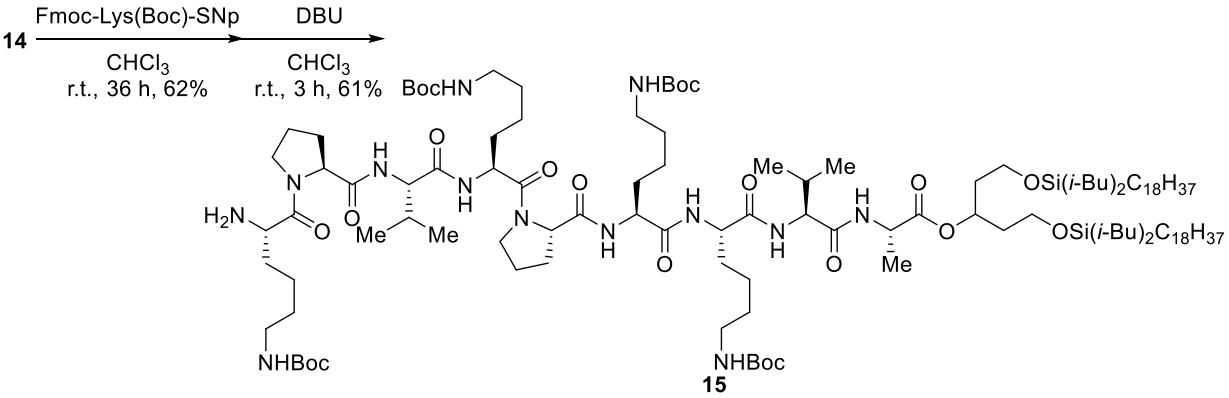
H-Pro-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (14) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **13** (849.9 mg, 0.43 mmol, 1.0 equiv) was added chloroform (1.3 mL). The Fmoc-Pro-SNp (308.5 g, 0.65 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 13 h. After completion, the mixture was diluted with dichloromethane (5 mL). Saturated Na_2CO_3 solution (5 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were added 15 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc = 3:1 to DCM/acetone = 3:1 to 1:1) to afford the Fmoc protected peptide as a white solid in 92% yield (899.9 mg).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (98.4 mg, 0.043 mmol, 1.0 equiv) was added chloroform (0.5 mL). The DBU (6.4 μL, 0.043 mmol, 1.0 equiv) was added. The resulting mixture was stirred

under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 7:1) to afford the product **14** as a white solid in 95% yield (84.0 mg).

Rf = 0.26 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{113}H_{217}N_{11}O_{17}Si_2Na$ $[M+Na]^+$: 2079.5890, Found: 2079.5894.

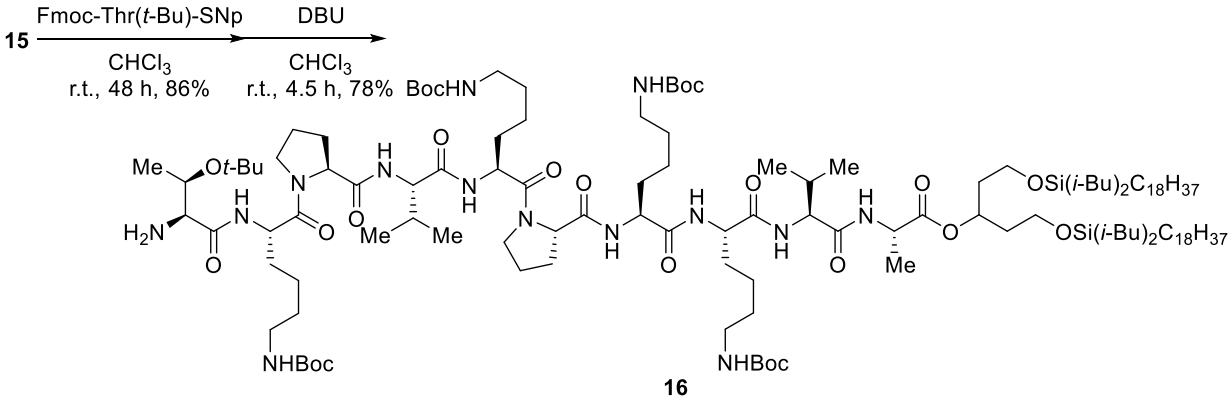


H-Lys(Boc)-Pro-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (15) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **14** (366.7 mg, 0.18 mmol, 1.0 equiv) was added chloroform (1.3 mL). The Fmoc-Lys(Boc)-SNp (161.7 mg, 0.27 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 36 h. After completion, the mixture was diluted with dichloromethane (3 mL). Saturated Na_2CO_3 solution (5 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were added 10 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc = 3:1 to DCM/acetone = 3:1 to 1:1) to afford the Fmoc protected peptide as a white solid in 62% yield (278.6 mg).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (257.6 mg, 0.10 mmol, 1.0 equiv) was added chloroform (1.0 mL). The DBU (15.3 μL, 0.10 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 7:1) to afford the product **15** as a white solid in 61% yield (142.6 mg).

Rf = 0.22 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{124}H_{237}N_{13}O_{20}Si_2Na$ $[M+Na]^+$: 2307.7364, Found: 2307.7350.

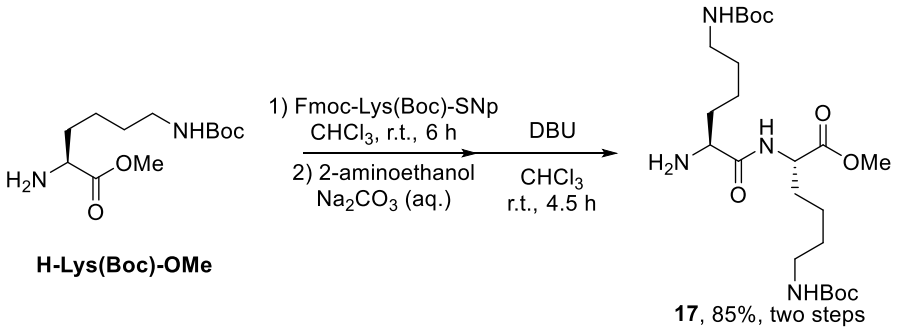


H-Thr(*t*-Bu)-Lys(Boc)-Pro-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (16) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **15** (129.4 mg, 0.057 mmol, 1.0 equiv) was added chloroform (0.3 mL). The Fmoc-Thr(*t*-Bu)-SNp (45.4 mg, 0.085 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 48 h. After completion, the mixture was diluted with dichloromethane (3 mL). Saturated Na_2CO_3 solution (5 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were added 10 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc = 3:1 to DCM/acetone = 3:1 to 1:1) to afford the Fmoc protected peptide as a white solid in 86% yield (130.2 mg).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (111.1 mg, 0.042 mmol, 1.0 equiv) was added chloroform (0.4 mL). The DBU (6.2 μL, 0.042 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 4.5 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 7:1) to afford the product **16** as a white solid in 78% yield (79.7 mg).

Rf = 0.21 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{132}H_{252}N_{14}O_{22}Si_2Na$ $[M+Na]^+$: 2464.8467, Found: 2464.8429.



H-Lys(Boc)-Lys(Boc)-OMe (17) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **H-Lys(Boc)-OMe** (781.0 mg, 3.0 mmol, 1.0 equiv) was added chloroform (6 mL). The Fmoc-Lys(Boc)-SNp (2.47 g, 4.1 mmol, 1.4 equiv) was added. The resulting mixture was stirred under room temperature for 6 h. After completion, the mixture was diluted with dichloromethane (20 mL), followed by adding 2-aminoethanol (535 μL) and stirring under room temperature for 15 min to remove the excess Fmoc-Lys(Boc)-SNp. Saturated Na_2CO_3 solution (20 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was put into next step without further purification.

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above residue was added chloroform (30.0 mL). The DBU (448.2 μL, 3.0 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 4.5 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 15:1 to 10:1) to afford the product **17** as a yellow oil in 85% total yield (1.24 g).

Rf = 0.45 (DCM/MeOH = 10:1).

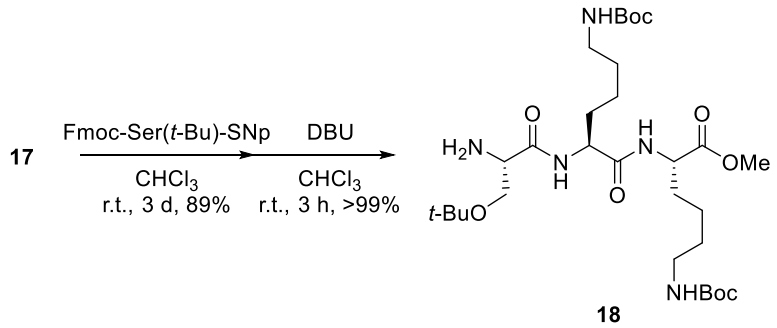
$[\alpha]_D^{24}$ = -7.29 (*c* 0.96, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, J = 8.4 Hz, 1H), 4.83 – 4.60 (m, 2H), 4.60 – 4.48 (m, 1H), 3.70 (s, 3H), 3.35 (dd, J = 7.8, 4.5 Hz, 1H), 3.16 – 2.97 (m, 4H), 1.92 – 1.74 (m, 2H), 1.73 – 1.61 (m, 1H), 1.59 – 1.22 (m, 27H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.9, 172.8, 156.03, 155.99, 79.0(2C), 54.9, 52.2, 51.5, 40.14, 40.09, 34.5, 32.0, 29.8, 29.5, 28.4(2C), 22.7, 22.5.

IR (neat) 3328, 2933, 1695, 1509, 1455, 1392, 1366, 1247, 1214, 1167 cm^{-1}.

HRMS (ESI) Calcd for $C_{23}H_{44}N_4O_7Na$ $[M+Na]^+$: 511.3108, Found: 511.3124.



H-Ser(*t*-Bu)-Lys(Boc)-Lys(Boc)-OMe (18) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **17** (1.14 g, 2.3 mmol, 1.0 equiv) was added chloroform (9.2 mL). The Fmoc-Ser(*t*-Bu)-SNp (1.82 g, 3.5 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 3 days. After completion, the mixture was diluted with dichloromethane (20 mL). Saturated Na_2CO_3 solution (20 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc = 3:1 to DCM/acetone = 5:1 to 3:1) to afford the Fmoc protected peptide as a pale yellow solid in 89% yield (1.76 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above Fmoc protected peptide (1.67 g, 1.96 mmol, 1.0 equiv) was added chloroform (20 mL). The DBU (292.1 μL, 1.96 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/MeOH = 15:1 to 10:1) to afford the product **18** as a colorless solid in >99% yield (1.23 g).

Rf = 0.45 (DCM/MeOH = 10:1).

M.p. 32-34 ºC.

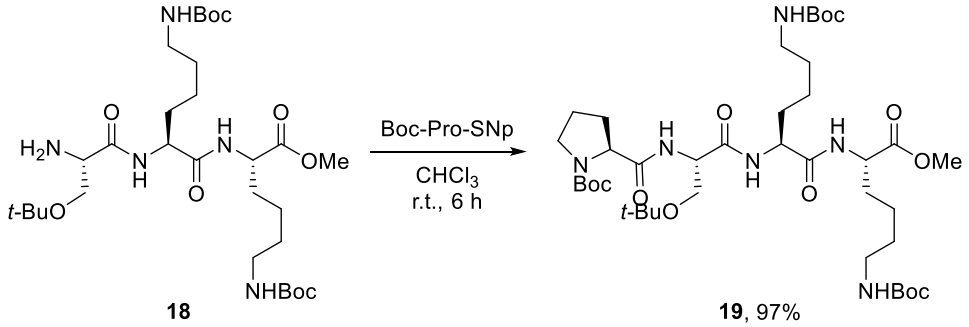
$[\alpha]_D^{24}$ = -20.18 (*c* 1.09, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.1 Hz, 1H), 6.88 (d, J = 7.8 Hz, 1H), 4.92 – 4.79 (m, 1H), 4.78 – 4.66 (m, 1H), 4.50 (td, J = 8.0, 4.9 Hz, 1H), 4.39 (td, J = 7.9, 5.9 Hz, 1H), 3.71 (s, 3H), 3.59 – 3.49 (m, 2H), 3.49 – 3.42 (m, 1H), 3.19 – 2.95 (m, 4H), 1.94 – 1.78 (m, 2H), 1.71 – 1.58 (m, 2H), 1.54 – 1.22 (m, 26H), 1.16 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$, lost one signal) δ 173.7, 172.6, 171.4, 156.1, 156.0, 79.0, 78.9, 73.3, 63.7, 55.1, 52.6, 52.3, 52.0, 40.0(2C), 31.5, 29.4(2C), 28.4(2C), 27.4, 22.5(2C).

IR (neat) 3310, 2975, 2933, 2867, 1743, 1690, 1651, 1514, 1455, 1391, 1365, 1248, 1168, 1083, 1012 cm^{-1}.

HRMS (ESI) Calcd for $C_{30}H_{57}N_5O_9Na$ $[M+Na]^+$: 654.4054, Found: 654.4023.



18 **19**, 97%

Boc-Pro-Ser(*t*-Bu)-Lys(Boc)-Lys(Boc)-OMe (19) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **18** (1.01 g, 1.59 mmol, 1.0 equiv) was added chloroform (3.2 mL). The Boc-Pro-SNp (841.8 mg, 2.39 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 6 h. After completion, the mixture was diluted with dichloromethane (15 mL). Saturated Na_2CO_3 solution (15 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×15 mL). The combined organic layers were added 50 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: DCM/EtOAc = 3:1 to DCM/acetone = 3:1 to 1.5:1) to afford the product **19** as a pale yellow solid in 97% yield (1.28 g).

Rf = 0.27 (DCM/acetone = 4:1).

M.p. 57-59 °C.

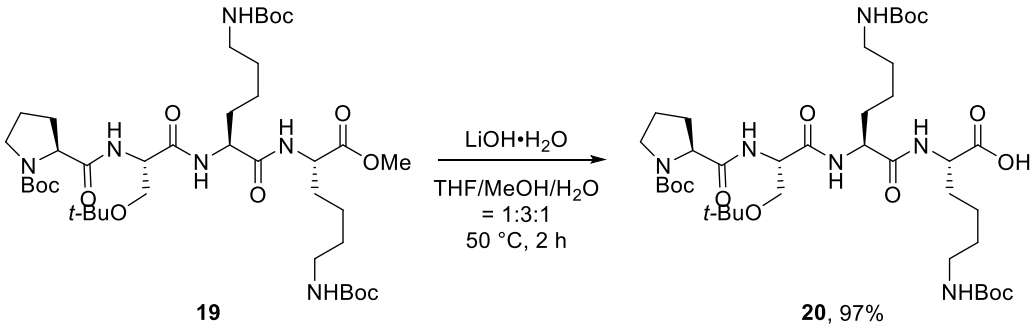
$[\alpha]_D^{25}$ = -36.36 (*c* 1.10, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, J = 8.6 Hz, 1H), 7.00 – 6.87 (m, 2H), 5.00 – 4.91 (m, 1H), 4.67 – 4.57 (m, 1H), 4.56 – 4.42 (m, 2H), 4.42 – 4.34 (m, 1H), 4.22 – 4.10 (m, 1H), 4.01 – 3.91 (m, 1H), 3.69 (s, 3H), 3.55 – 3.39 (m, 3H), 3.15 – 2.95 (m, 4H), 2.36 – 2.20 (m, 1H), 2.13 – 1.97 (m, 2H), 1.98 – 1.78 (m, 3H), 1.78 – 1.68 (m, 1H), 1.68 – 1.54 (m, 1H), 1.53 – 1.26 (m, 35H), 1.14 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 172.3, 171.5, 170.4, 156.0, 155.9, 155.8, 81.1, 78.8, 78.7, 73.6, 61.5, 60.6, 54.2, 53.1, 52.1(2C), 47.5, 40.2(2C), 31.3, 30.6, 30.0, 29.3(2C), 28.4, 28.35, 28.32, 27.4, 24.6, 23.1, 22.7.

IR (neat) 3321, 2976, 2932, 1744, 1669, 1515, 1455, 1392, 1365, 1248, 1164, 1095, 1041, 1017 cm^{-1}.

HRMS (ESI) Calcd for $C_{40}H_{72}N_6O_{12}Na$ $[M+Na]^+$: 851.5106, Found: 851.5083.



19 **20**, 97%

Boc-Pro-Ser(*t*-Bu)-Lys(Boc)-Lys(Boc)-OH (20) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **19** (165.8 mg, 0.2 mmol, 1.0 equiv) was added tetrahydrofuran (0.2 mL), methanol (0.6 mL) and water (0.2 mL). The lithium hydroxide monohydrate (8.4 mg, 0.2 mmol, 1.0 equiv) was added. The reaction was stirred under 50 ºC for 2 h. After completion, the reaction mixture was concentrated. Chloroform (5 mL) was added, followed by adding hydrochloric acid solution (2 N in water, 100 μL, 0.2 mmol, 1.0 equiv). Water (10 mL) was added, and the layers were separated. The aqueous layer was extracted with chloroform (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4 and filtered. The filtrate was concentrated to afford the product **20** as a pale yellow solid in 97% yield (158.8 mg). The product was pure enough without further purification.

Rf = 0.30 (DCM/MeOH = 10:1).

M.p. 82-83 ºC.

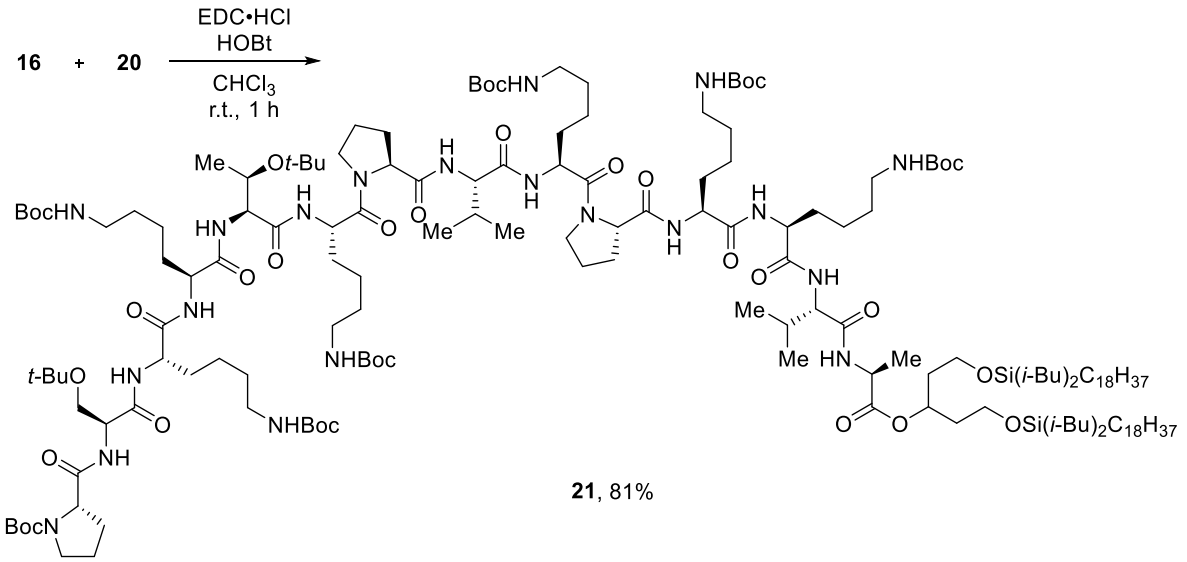
$[\alpha]_D^{25}$ = -28.97 (*c* 1.07, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.7 Hz, 1H), 7.12 (d, J = 7.6 Hz, 1H), 6.99 (d, J = 7.2 Hz, 1H), 5.13 – 4.92 (m, 1H), 4.85 – 4.70 (m, 1H), 4.58 – 4.30 (m, 3H), 4.28 – 4.15 (m, 1H), 3.98 – 3.85 (m, 1H), 3.57 – 3.35 (m, 3H), 3.18 – 2.94 (m, 4H), 2.37 – 2.20 (m, 1H), 2.12 – 1.97 (m, 2H), 1.96 – 1.82 (m, 3H), 1.82 – 1.72 (m, 1H), 1.71 – 1.60 (m, 1H), 1.54 – 1.28 (m, 35H), 1.14 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 173.3, 172.1, 170.8, 156.1, 155.9(2C), 81.2, 78.9, 78.7, 73.7, 61.4, 60.6, 54.3, 53.4(2C), 52.8, 47.6, 40.31, 40.28, 31.0, 30.0, 29.3(2C), 28.41, 28.39, 28.35, 27.4, 24.6, 23.1, 22.8.

IR (neat) 3315, 2976, 2934, 1667, 1516, 1455, 1392, 1365, 1248, 1163, 1095 cm^{-1}.

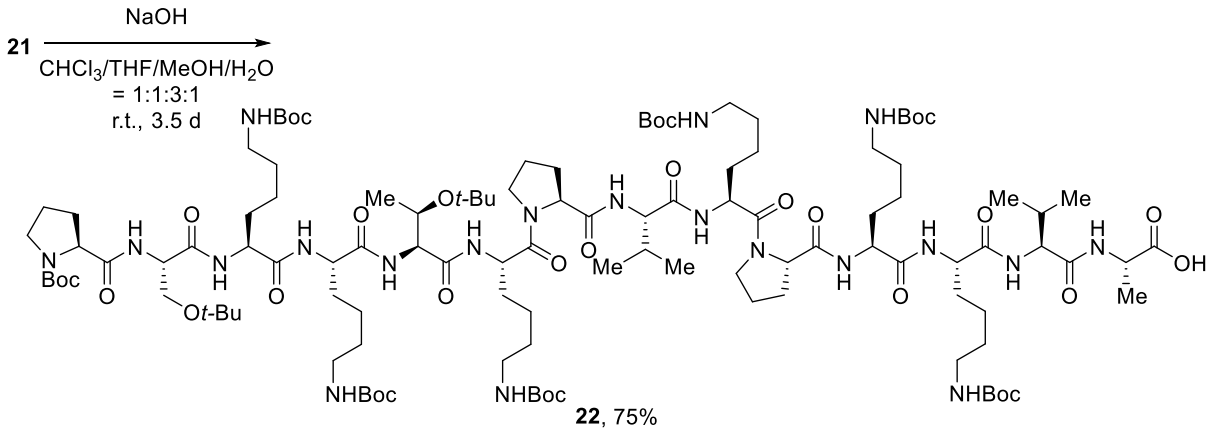
HRMS (ESI) Calcd for $C_{39}H_{70}N_6O_{12}Na$ $[M+Na]^+$: 837.4949, Found: 837.4917.



Boc-Pro-Ser(*t*-Bu)-Lys(Boc)-Lys(Boc)-Thr(*t*-Bu)-Lys(Boc)-Pro-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OTAG4 (21) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co), **16** (14.7 mg, 0.0060 mmol, 1.0 equiv), **20** (6.4 mg, 0.0078 mmol, 1.3 equiv), EDC·HCl (1.6 mg, 0.0084 mmol, 1.4 equiv) and HOBt (1.0 mg, 0.0072 mmol, 1.2 equiv) was added chloroform (0.12 mL). The resulting mixture was stirred under room temperature for 1 h. After completion, the mixture was diluted with chloroform (1 mL). Water (1 mL) was added, and the layers were separated. The aqueous layer was extracted with chloroform (3×1 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: $CHCl_3$/acetone = 2:1 to 1:1 to DCM/MeOH = 10:1) to afford the product **21** as a pale yellow solid in 81% yield (15.7 mg).

Rf = 0.46 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{171}H_{320}N_{20}O_{33}Si_2Na$ $[M+Na]^+$: 3261.3413, Found: 3263.2883.



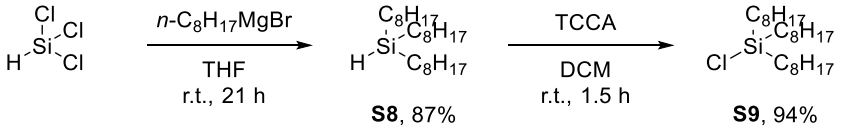
Boc-Pro-Ser(*t*-Bu)-Lys(Boc)-Lys(Boc)-Thr(*t*-Bu)-Lys(Boc)-Pro-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OH (22) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co), **21** (15.4 mg, 0.00475 mmol, 1.0 equiv) and sodium hydroxide (0.6 mg, 0.015 mmol, 3.2 equiv) was added chloroform (20 μL), tetrahydrofuran (20 μL), methanol (60 μL) and water (20 μL). The resulting mixture was stirred under room temperature for 3.5 days. After completion, the reaction mixture was concentrated. Chloroform (1 mL) was added, followed by adding hydrochloric acid solution (2 N in water, 16 μL, 0.032 mmol, 6.7 equiv). The mixture was then concentrated, followed by washing with hexane (3×1 mL). The hexane layers were collected carefully with pipette and concentrated to afford the **HOTAG4** as a colorless oil in 81% yield (3.5 mg). The remaining solid residue was dissolved in chloroform and filtered *via* PTFE syringe filter (0.22 μm) to remove the insoluble solids. The chloroform was removed to afford 8.4 mg product **22** as a white solid (75% yield). The purity of the product was 86% which was determined by RP-HPLC using a revised-phase column (ODS-HL, 4.6 mm × 25 cm).

Rf = 0.09 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{114}H_{202}N_{20}O_{31}Na$ $[M+Na]^+$: 2370.4743, Found: 2370.4721.

X. Synthesis of TAG6 and requirements test

Synthesis of TAG6.



Chlorotrioctylsilane (S9) At room temperature, under N_2, to a flame-dried flask equipped with a condenser and a magnetic stirring bar (Sm-Co) was added Mg (1.22 g, 51.0 mmol, 5.1 equiv) and tetrahydrofuran (60 mL), followed by adding 1-bromooctane (5.92 mL, 34.0 mmol, 3.4 equiv). The mixture boiled spontaneously. [***Note**: Using ice bath to slow down the reaction if necessary.*] After stirring for 10 min, the reaction was transferred to an oil bath to reflux for 2 h. Then, the oil bath was changed to an ice bath. Trichlorosilane (1.01 mL, 10.0 mmol, 1.0 equiv) was introduced dropwise into the reaction flask. The reaction was warmed to room temperature and stirred for 21 h. Then, water (40 mL) and saturated NH_4Cl solution (10 mL) were added, and the layers were separated. The aqueous layer was extracted with hexanes (3×50 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes) to afford the product **S8** as a colorless oil in 87% yield (3.21 g).

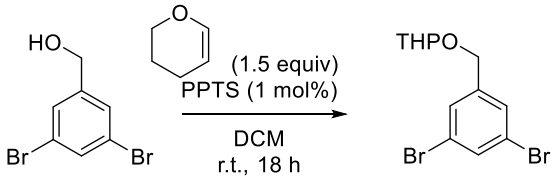
At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S8** (4.49 mL, 10.0 mmol, 1.0 equiv) was added dichloromethane (100 mL). The trichloroisocyanuric acid (790.2 mg, 3.4 mmol, 0.34 equiv) was added. The resulting mixture was stirred under room temperature for 1.5 h. After completion, the reaction mixture was concentrated. The residue was dissolved in 10 mL anhydrous hexane, filtrated through a short pad of celite and washed with anhydrous hexane (10 mL). The filtrate was concentrated to afford the product **S9** as a colorless oil in 94% yield (3.79 g).

^{1}H NMR (400 MHz, $CDCl_3$) δ 1.46 – 1.17 (m, 36H), 0.92 – 0.84 (m, 9H), 0.84 – 0.75 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 33.2, 31.9, 29.19, 29.17, 23.0, 22.7, 16.2, 14.1.

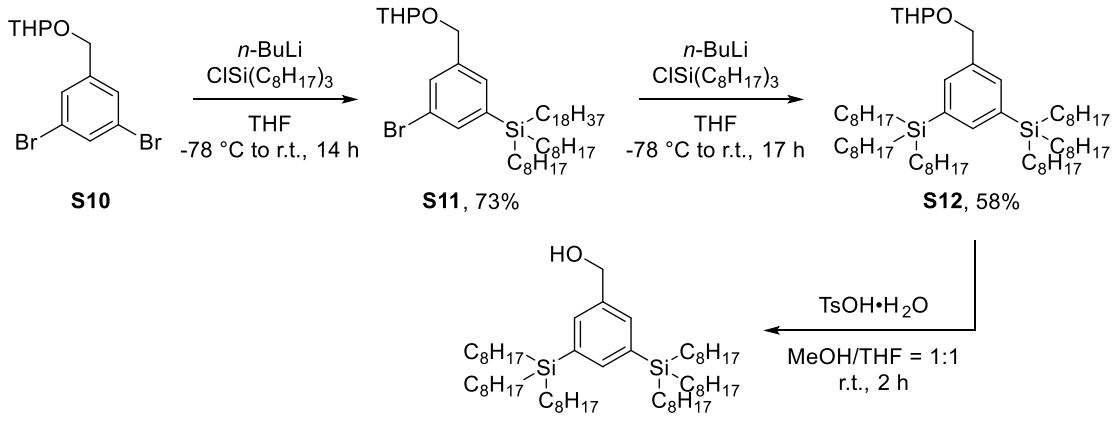
IR (neat) 2956, 2921, 2853, 1465, 1406, 1378, 1341, 1178, 1108, 1077, 1004 cm^{-1}.

HRMS (ESI) Calcd for $C_{24}H_{51}ClSiNa$ $[M+Na]^+$: 425.3346, Found: 425.3302.



S10, 95%

2-((3,5-Dibromobenzyl)oxy)tetrahydro-2*H*-pyran (S10) was prepared from 3,5-dibromobenzyl alcohol in one step according to the procedure in the literature with a slight modification.[7] At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co), 3,5-dibromobenzyl alcohol (2.66 g, 10.0 mmol, 1.0 equiv) and pyridinium p-toluenesulfonate (251.3 mg, 1.0 mmol, 0.1 equiv) was added dichloromethane (50 mL). The 3,4-dihydro-2*H*-pyran (1.37 mL, 15.0 mmol, 1.5 equiv) was added. The resulting mixture was stirred under room temperature for 18 h. After completion, saturated $NaHCO_3$ solution (50 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×40 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1) to afford the product **S10** as a pale yellow oil in 95% yield (3.32 g). It is a known compound. The characterization data match the reported data.[7]



S10 **S11**, 73% **S12**, 58%

HOTAG6, 95%

(3,5-Bis(trioctylsilyl)phenyl)methanol (HOTAG6) At -78 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S10** (1.40 g, 4.0 mmol, 1.0 equiv) was added tetrahydrofuran (24 mL). The *n*-butyllithium (1.57 M in hexanes, 2.7 mL, 4.2 mmol, 1.05 equiv) was added. The reaction was stirred at -78 °C for 1 h, followed by adding chlorotrioctylsilane (**S9**, 2.26 g, 5.6 mmol, 1.4 equiv). The reaction was warmed to room temperature and stirred for 14 h. After completion, the reaction mixture was concentrated. The

residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 75:1) to afford the product **S11** as a colorless oil in 73% yield (1.85 g).

At -78 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S13** (1.77 g, 2.8 mmol, 1.0 equiv) was added tetrahydrofuran (24 mL). The *n*-butyllithium (1.57 M in hexanes, 1.9 mL, 3.1 mmol, 1.1 equiv) was added. The reaction was stirred at -78 °C for 1 h, followed by adding chlorotrioctylsilane (**S9**, 1.51 g, 3.7 mmol, 1.35 equiv). The reaction was warmed to room temperature and stirred for 17 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 85:1) to afford the product **S12** as a colorless oil in 58% yield (1.54 g).

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S12** (1.54 g, 1.67 mmol, 1.0 equiv) was added methanol (8 mL) and tetrahydrofuran (8 mL). The *p*-toluenesulfonic acid monohydrate (31.8 mg, 0.167 mmol, 0.1 equiv) was added. The reaction was stirred under room temperature for 2 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 85:1) to afford the product **HOTAG6** as a colorless oil in 95% yield (1.33 g).

Rf = 0.31 (hexanes/EtOAc = 20:1).

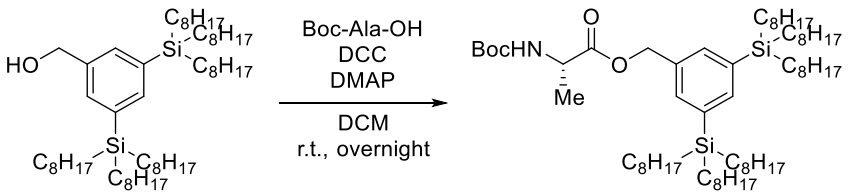
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (s, 1H), 7.43 (s, 2H), 4.68 (d, J = 6.0 Hz, 2H), 1.36 – 1.18 (m, 72H), 0.92 – 0.82 (m, 18H), 0.81 – 0.72 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.7, 138.7, 137.3, 133.2, 66.1, 33.8, 31.9, 29.32, 29.27, 23.9, 22.7, 14.1, 12.5.

IR (neat) 3364, 2955, 2919, 2851, 1463, 1411, 1377, 1205, 1175, 1108, 1016 cm^{-1}.

HRMS (ESI) Calcd for $C_{55}H_{108}OSi_2Na$ $[M+Na]^+$: 863.7836, Found: 863.7878.

Esterification test.



HOTAG6 **Boc-Ala-OTAG6**, 96%

3,5-Bis(trioctylsilyl)benzyl (*tert*-butoxycarbonyl)-L-alaninate (Boc-Ala-OTAG6) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and

HOTAG6 (67.3 mg, 0.08 mmol, 1.0 equiv) was added dichloromethane (1.0 mL). The Boc-Ala-OH (30.3 mg, 0.16 mmol, 2.0 equiv) was added, followed by adding DMAP (11.7 mg, 0.096 mmol, 1.2 equiv) and DCC (33.0 mg, 0.16 mmol, 2.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 40:1 to 20:1) to afford the product **Boc-Ala-OTAG6** as a pale yellow oil in 96% yield (77.8 mg).

Rf = 0.65 (hexanes/EtOAc = 5:1).

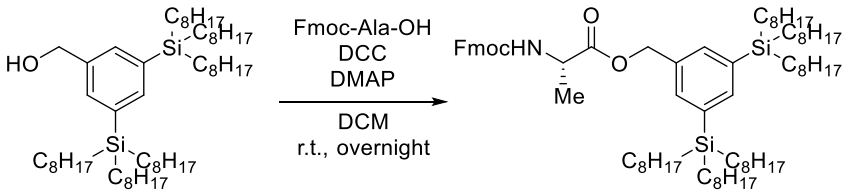
$[\alpha]_D^{25}$ = -14.03 (*c* 0.57, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (s, 1H), 7.38 (s, 2H), 5.24 (d, *J* = 12.3 Hz, 1H), 5.17 – 5.01 (m, 2H), 4.47 – 4.25 (m, 1H), 1.44 (s, 9H), 1.39 (d, *J* = 7.2 Hz, 3H), 1.35 – 1.19 (m, 72H), 0.92 – 0.83 (m, 18H), 0.81 – 0.72 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 155.0, 140.2, 137.4, 134.1, 133.4, 79.8, 67.7, 49.2, 33.8, 31.9, 29.31, 29.27, 28.3, 23.8, 22.7, 18.8, 14.1, 12.5.

IR (neat) 2957, 2922, 2853, 1711, 1500, 1456, 1367, 1341, 1215, 1163, 1055 cm^{-1}.

HRMS (ESI) Calcd for $C_{63}H_{121}NO_4Si_2Na$ $[M+Na]^+$: 1034.8732, Found: 1034.8707.



HOTAG6 **Fmoc-Ala-OTAG6**, 99%

3,5-Bis(trioctylsilyl)benzyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG6) At room temperature, to a flame-dried fask charged with magnetic stirring bar (Sm-Co) and **HOTAG6** (1.33 g, 1.58 mmol, 1.0 equiv) was added dichloromethane (16.0 mL). The Fmoc-Ala-OH (983.8 mg, 3.16 mmol, 2.0 equiv) was added, followed by adding DMAP (231.6 mg, 1.90 mmol, 1.2 equiv) and DCC (652.0 mg, 3.16 mmol, 2.0 equiv). The reaction was stirred at room temperature overnight. Then, filtered, washed with dichloromethane (10 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 40:1 to 20:1) to afford the product **Fmoc-Ala-OTAG6** as a colorless oil in 99% yield (1.78 g).

Rf = 0.20 (hexanes/EtOAc = 20:1).

$[\alpha]_D^{24}$ = +2.80 (*c* 1.07, $CHCl_3$).

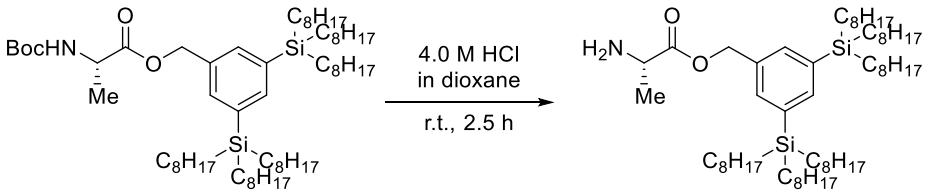
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 7.5 Hz, 2H), 7.66 – 7.55 (m, 3H), 7.45 – 7.36 (m, 4H), 7.36 – 7.28 (m, 2H), 5.43 (d, J = 7.7 Hz, 1H), 5.28 (d, J = 12.2 Hz, 1H), 5.13 (d, J = 12.2 Hz, 1H), 4.54 – 4.37 (m, 3H), 4.24 (t, J = 7.2 Hz, 1H), 1.46 (d, J = 7.1 Hz, 3H), 1.36 – 1.23 (m, 72H), 0.93 – 0.84 (m, 18H), 0.84 – 0.73 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 155.6, 143.9, 143.8, 141.3, 140.3, 137.5, 134.1, 133.3, 127.7, 127.0, 125.1, 120.0, 67.9, 67.0, 49.7, 47.1, 33.8, 31.9, 29.30, 29.27, 23.8, 22.7, 18.8, 14.1, 12.4.

IR (neat) 2955, 2920, 2852, 1728, 1505, 1451, 1336, 1201, 1173, 1106, 1074, 1054 cm^{-1}.

HRMS (ESI) Calcd for $C_{73}H_{123}NO_4Si_2Na$ $[M+Na]^+$: 1156.8888, Found: 1156.8890.

Tolerance test.



Boc-Ala-OTAG6 **H-Ala-OTAG6**, 88%

3,5-Bis(trioctylsilyl)benzyl L-alaninate (H-Ala-OTAG6) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Boc-Ala-OTAG6** (81.3 mg, 0.08 mmol, 1.0 equiv) was added hydrochloric acid solution (4.0 M in dioxane, 161 μL, 0.64 mmol, 8.0 equiv). The reaction was stirred at room temperature for 2.5 h. Then, 10 mL dichloromethane and 10 mL saturated Na_2CO_3 solution were added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 4:1) to afford the product **H-Ala-OTAG6** as a pale yellow oil in 88% yield (64.7 mg).

Rf = 0.61 (hexanes/EtOAc = 2:1).

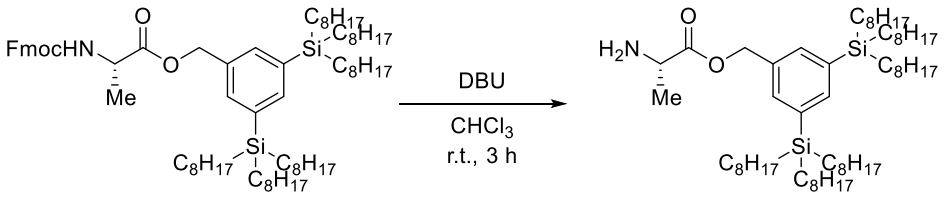
$[\alpha]_D^{27}$ = +33.33 (*c* 1.08, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (s, 1H), 7.38 (s, 2H), 5.24 – 5.07 (m, 2H), 3.67 – 3.49 (m, 1H), 1.36 (d, J = 7.0 Hz, 3H), 1.34 – 1.18 (m, 72H), 0.91 – 0.83 (m, 18H), 0.81 – 0.72 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 157.8, 140.1, 137.4, 134.0, 133.7, 67.2, 50.1, 33.8, 31.9, 29.31, 29.27, 23.9, 22.7, 20.6, 14.1, 12.5.

IR (neat) 2956, 2919, 2852, 1741, 1624, 1457, 1411, 1377, 1174, 1142, 1111, 1060 cm^{-1}.

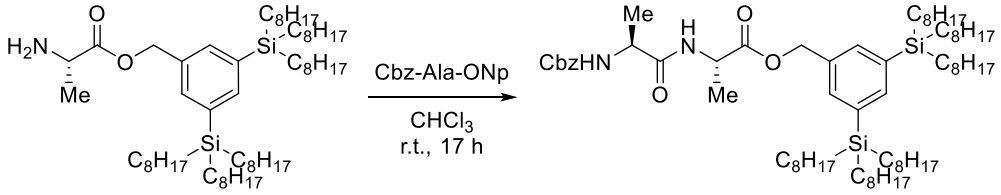
HRMS (ESI) Calcd for $C_{58}H_{113}NO_2Si_2Na$ $[M+Na]^+$: 934.8208, Found: 934.8256.



Fmoc-Ala-OTAG6 **H-Ala-OTAG6**, 97%

3,5-Bis(trioctylsilyl)benzyl L-alaninate (H-Ala-OTAG6) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAG6** (88.8 mg, 0.08 mmol, 1.0 equiv) was added chloroform (0.8 mL). The DBU (11.7 μL, 0.08 mmol, 1.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1 to 4:1) to afford the product **H-Ala-OTAG6** as a pale yellow oil in 97% yield (69.3 mg).

Elongation test (dipeptide synthesis).



H-Ala-OTAG6 **Cbz-Ala-Ala-OTAG6**, 85%

Cbz-Ala-Ala-OTAG6 At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **H-Ala-OTAG6** (136.8 mg, 0.15 mmol, 1.0 equiv) was added chloroform (0.3 mL). The Cbz-Ala-ONp (103.2 mg, 0.30 mmol, 2.0 equiv) was added. The resulting mixture was stirred under room temperature for 17 h. After completion, the mixture was diluted with dichloromethane (5 mL). Saturated Na_2CO_3 solution (5 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×5 mL). The combined organic layers were added 15 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 7:1 to 6.5:1) to afford the product **Cbz-Ala-Ala-OTAG6** as a pale yellow oil in 85% yield (143.1 mg).
Rf = 0.34 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{25}$ = -4.08 (*c* 0.98, $CHCl_3$).

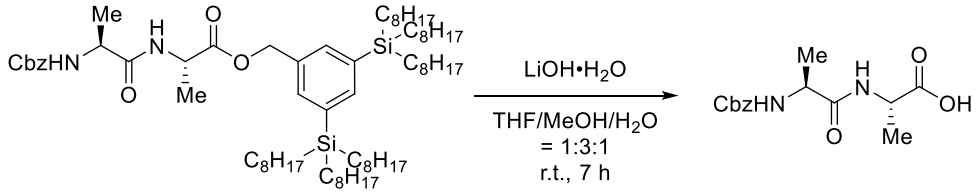
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (s, 1H), 7.38 (s, 2H), 7.37 – 7.26 (m, 5H), 6.67 – 6.39 (m, 1H), 5.39 (d, *J* = 6.1 Hz, 1H), 5.24 (d, *J* = 12.2 Hz, 1H), 5.17 – 5.05 (m, 3H), 4.70 – 4.57 (m, 1H), 4.36 – 4.13 (m, 1H), 1.44 – 1.36 (m, 6H), 1.35 – 1.17 (m, 72H), 0.92 – 0.83 (m, 18H), 0.82 – 0.73 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 171.7, 155.9, 140.3, 137.5, 136.2, 134.1, 133.2, 128.5, 128.2, 128.1, 67.9, 67.0, 50.4, 48.2, 33.8, 31.9, 29.30, 29.25, 23.8, 22.7, 18.7, 18.4, 14.1, 12.4.

IR (neat) 3312, 2956, 2921, 2852, 1739, 1667, 1510, 1455, 1378, 1340, 1215, 1145 cm^{-1}.

HRMS (ESI) Calcd for $C_{69}H_{124}N_2O_5Si_2Na$ $[M+Na]^+$: 1139.8946, Found: 1139.8899.

Cleavage test.



Cbz-Ala-Ala-OTAG6 **Cbz-Ala-Ala-OH**, 92%

Cbz-Ala-Ala-OH At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-Ala-OTAG6** (84.4 mg, 0.076 mmol, 1.0 equiv) was added tetrahydrofuran (75 μL), methanol (225 μL) and water (75 μL). The lithium hydroxide monohydrate (3.2 mg, 0.076 mmol, 1.0 equiv) was added. The reaction was stirred under room temperature for 7 h. After completion, the reaction mixture was concentrated. Chloroform (5 mL) was added, followed by adding hydrochloric acid solution (2 N in water, 45 μL, 0.09 mmol, 1.2 equiv). Saturated NaCl solution (5 mL) was added, and the layers were separated. The aqueous layer was extracted with chloroform (3×5 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was added hexanes to wash away the hydrophobic tag (3×5 mL). All the hexane phases were collected, and the mixture was concentrated to afford the **HOTAG6** as a colorless oil in 95% yield (60.3 mg). The remaining white solid was the product **Cbz-Ala-Ala-OH** in 92% yield (20.5 mg).

M.p. 143-145 °C.

$[\alpha]_D^{27}$ = +11.22 (*c* 0.98, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.26 (m, 5H), 6.97 – 6.82 (m, 1H), 5.61 (d, *J* = 7.2 Hz, 1H), 5.16 – 5.04 (m, 2H), 4.57 – 4.49 (m, 1H), 4.40 – 4.22 (m, 1H), 1.42 (d, *J* = 7.1 Hz, 3H), 1.36 (d, *J* = 7.0 Hz, 3H).

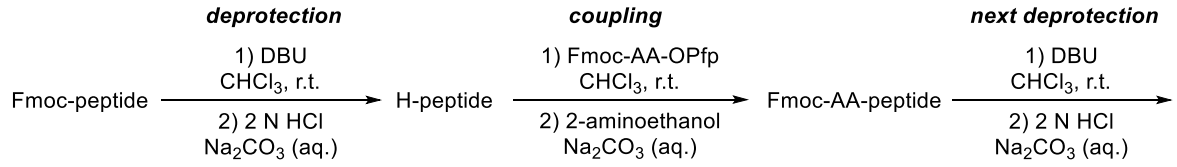
^{13}C NMR (100 MHz, $CDCl_3$) δ 175.5, 172.5, 156.2, 136.0, 128.6, 128.3, 128.1, 67.2, 50.4, 48.3, 18.4, 17.8.

IR (neat) 3300, 3086, 2978, 1730, 1645, 1534, 1453, 1400, 1332, 1255, 1138 cm^{-1}.

HRMS (ESI) Calcd for $C_{14}H_{18}N_2O_5Na$ $[M+Na]^+$: 317.1113, Found: 317.1114.

XI. Peptide elongation of alanine chain with TAG6

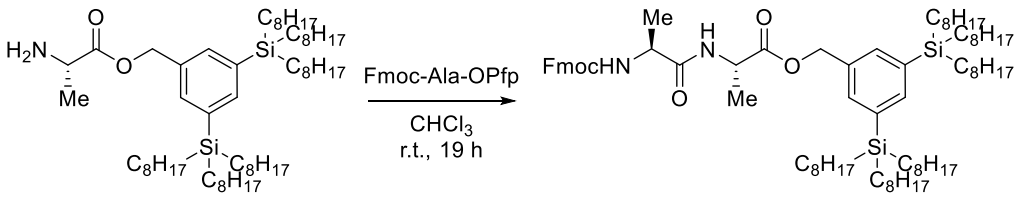
General procedures for Fmoc-deprotection and coupling reactions.



At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and Fmoc protected peptide (1.0 equiv) was added chloroform (0.1 M). The DBU (2.0 equiv) was added. The resulting mixture was stirred under room temperature for 0.5 h. After completion, hydrochloric acid solution (2 N in water, 1.0 equiv) was added and stirred for 1 min to remove DBU. Check the pH value of the mixture. More hydrochloric acid solution would be added to adjust the solution of pH 6~7 if necessary. The mixture was poured into a separation funnel charged with water (same volume with the reaction mixture) and shaken, followed by adding saturated Na_2CO_3 solution (same volume with the water). The layers were separated. The aqueous layer was extracted with dichloromethane for three times. The combined organic layers were added saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane for three times. The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. Check the crude ^1H NMR to confirm the removal of DBU. The residue was put into next Fmoc-coupling step without further purification.

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and above residue was added chloroform (0.3 to 0.5 M). The active pentafluorophenyl amino acid ester (Fmoc-AA-OPfp, 1.2 equiv) was added. The resulting mixture was stirred under room temperature. After completion, the mixture was diluted with dichloromethane, followed by adding 2-aminoethanol (1.65 equiv, 50 μL per 0.1 mmol unreacted Fmoc-AA-OPfp) and stirring under room temperature for 20 min to remove the excess Fmoc-AA-OPfp. Saturated Na_2CO_3 solution was added, and the layers were separated. The aqueous layer was extracted with dichloromethane for three times. The combined organic layers were added saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane for three times. The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was put into next Fmoc-deprotection step without further purification.

Elongation.



H-Ala-OTAG6 **23**, 92%

Fmoc-Ala-Ala-OTAG6 (23) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **H-Ala-OTAG6** (1.00 g, 1.10 mmol, 1.0 equiv) was added chloroform (2.2 mL). The Fmoc-Ala-OPfp (630.1 mg, 1.32 mmol, 1.2 equiv) was added. The resulting mixture was stirred under room temperature for 19 h. After completion, the mixture was diluted with dichloromethane (10 mL), followed by adding 2-aminoethanol (110 μL) and stirring under room temperature for 20 min to remove the excess Fmoc-Ala-OPfp. Saturated Na_2CO_3 solution (15 mL) was added, and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL). The combined organic layers were added 30 mL saturated Na_2CO_3 solution to wash again. The layers were separated, and the aqueous layer was extracted with dichloromethane (3×20 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 5:1) to afford the product **23** as a colorless oil in 92% yield (1.22 g).

Rf = 0.28 (hexanes/EtOAc = 5:1).

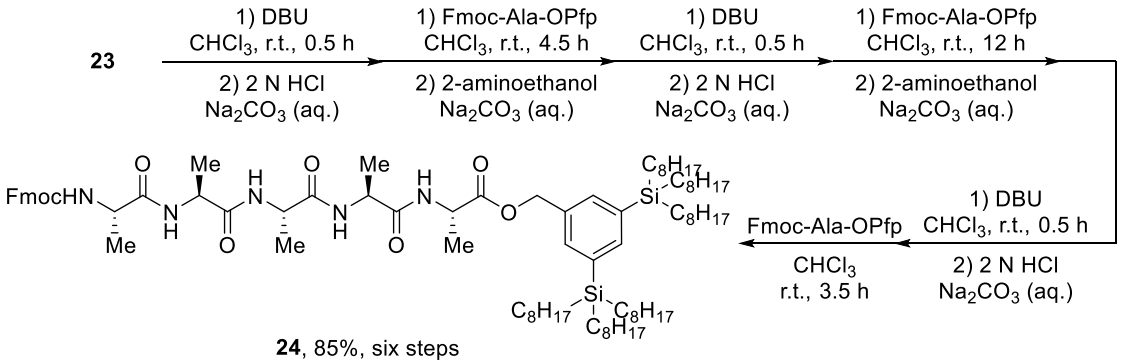
$[\alpha]_D^{24}$ = +2.41 (*c* 0.83, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.63 – 7.54 (m, 3H), 7.44 – 7.36 (m, 4H), 7.35 – 7.28 (m, 2H), 6.48 (d, *J* = 7.6 Hz, 1H), 5.42 (d, *J* = 6.9 Hz, 1H), 5.24 (d, *J* = 12.1 Hz, 1H), 5.10 (d, *J* = 12.1 Hz, 1H), 4.73 – 4.56 (m, 1H), 4.41 (d, *J* = 7.0 Hz, 2H), 4.33 – 4.16 (m, 2H), 1.46 – 1.38 (m, 6H), 1.35 – 1.20 (m, 72H), 0.92 – 0.83 (m, 18H), 0.82 – 0.73 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 171.6, 155.8, 143.8, 141.3, 140.3, 137.5, 134.1, 133.1, 127.7, 127.1, 125.1, 120.0, 68.0, 67.1, 50.4, 48.2, 47.1, 33.8, 31.9, 29.29, 29.26, 23.8, 22.7, 18.9, 18.4, 14.1, 12.4.

IR (neat) 3310, 2955, 2920, 2852, 1742, 1664, 1537, 1451, 1254, 1197, 1145, 1107 cm^{-1}.

HRMS (ESI) Calcd for $C_{76}H_{128}N_2O_5Si_2Na$ $[M+Na]^+$: 1227.9259, Found: 1227.9267.



Fmoc-Ala-Ala-Ala-Ala-Ala-OTAG6 (24) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and Fmoc-protected peptide **23** (1.22 g, 1.0 mmol, 1.0 equiv) was added chloroform (10 mL). Then, the mixture was followed the **General procedures for Fmoc-deprotection and coupling reactions** and repeated twice (three times in total). In the final coupling step, after the completion of the coupling reaction, the reaction mixture was concentrated without adding 2-aminoethanol. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc/DCM = 2:2:1 to DCM/EtOAc = 1:1 to DCM/MeOH = 10:1 to $CHCl_3$/MeOH = 10:1) to afford the product **24** as a pale yellow solid in 85% total yield (1.22 g).

Rf = 0.16 (DCM/EtOAc = 2:1).

M.p. 218-219 °C.

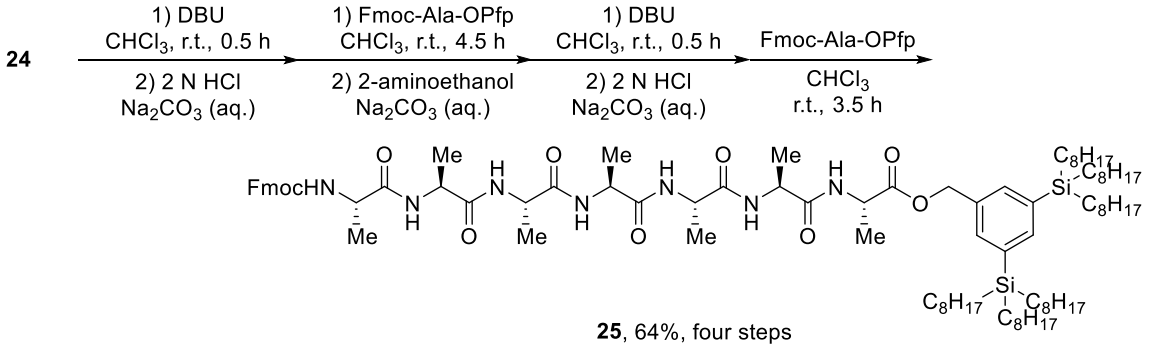
$[\alpha]_D^{25}$ = +19.78 (*c* 0.91, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 – 8.02 (m, 1H), 7.96 – 7.72 (m, 2H), 7.72 – 7.55 (m, 5H), 7.53 (s, 1H), 7.34 – 7.27 (m, 4H), 7.24 – 7.14 (m, 2H), 6.71 – 6.41 (m, 1H), 5.09 (d, *J* = 12.2 Hz, 1H), 5.03 – 4.72 (m, 4H), 4.73 – 4.53 (m, 2H), 4.52 – 4.38 (m, 1H), 4.38 – 4.26 (m, 1H), 4.26 – 4.16 (m, 1H), 1.53 – 1.37 (m, 12H), 1.37 – 1.15 (m, 75H), 0.93 – 0.79 (m, 18H), 0.79 – 0.56 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 172.5, 172.2(2C), 172.1, 156.4, 144.1, 143.8, 141.1, 140.1, 137.3, 133.9, 133.3, 127.5, 126.9, 125.4, 125.2, 119.7, 67.6, 67.3, 50.5, 49.0(2C), 48.9, 48.0, 47.0, 33.8, 31.9, 29.3, 29.2, 23.8, 22.7, 21.1, 21.0, 20.8, 20.4, 18.7, 14.1, 12.4.

IR (neat) 3279, 2955, 2920, 2852, 1740, 1708, 1679, 1656, 1628, 1520, 1451, 1374, 1252, 1216, 1167, 1135, 1106, 1053 cm^{-1}.

HRMS (ESI) Calcd for $C_{85}H_{143}N_5O_8Si_2Na$ $[M+Na]^+$: 1441.0373, Found: 1441.0377.

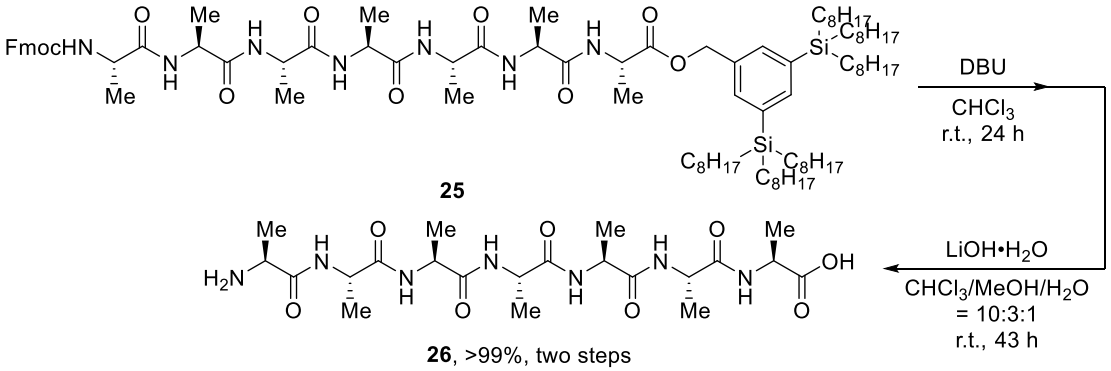


Fmoc-Ala-Ala-Ala-Ala-Ala-Ala-Ala-OTAG6 (25) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and Fmoc-protected peptide **24** (99.8 mg, 0.07 mmol, 1.0 equiv) was added chloroform (0.7 mL). Then, the mixture was followed the **General procedures for Fmoc-deprotection and coupling reactions** and repeated once (twice in total). In the final coupling step, after the completion of the coupling reaction, the reaction mixture was concentrated without adding 2-aminoethanol. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 1:1 to DCM/EtOAc = 1:1 to $CHCl_3$/acetone = 2:1 to 1:1 to DCM/MeOH = 10:1) to afford the product **25** as a pale yellow solid in 64% total yield (69.8 mg).

Rf = 0.30 ($CHCl_3$/acetone = 3:1).

HRMS (ESI) Calcd for $C_{91}H_{153}N_7O_{10}Si_2Na$ $[M+Na]^+$: 1583.1115, Found: 1583.1144.

Cleavage.



H-Ala-Ala-Ala-Ala-Ala-Ala-Ala-OH (26) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **25** (37.8 mg, 0.024 mmol, 1.0 equiv) was added chloroform (0.5 mL). The DBU (7.2 μL, 0.048 mmol, 2.0 equiv) was added. After stirring under room temperature for 2 h, 1 mL chloroform was added. The resulting mixture

was stirred under room temperature for another 22 h. After completion, the reaction mixture was concentrated. The residue was put into next step without further purification.

At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and above residue was added chloroform (0.5 mL), methanol (0.15 mL) and water (50 μL). The lithium hydroxide monohydrate (2.0 mg, 0.048 mmol, 2.0 equiv) was added. The reaction was stirred under room temperature for 43 h. After completion, the reaction mixture was concentrated. Chloroform (0.5 mL) was added, followed by adding hydrochloric acid solution (2 N in water, 25 μL, 0.05 mmol, 2.07 equiv). The mixture was filtered and the solid was washed by water (3×1 mL), followed by chloroform (3×1 mL). The solid was then dissolved in trifluoroacetic acid (TFA) and filtered to remove the insoluble solids. The TFA mixture was concentrated to afford 15.4 mg product **26** as a pale yellow solid (>99% total yield). The purity of the product was 77% which was determined by RP-HPLC using a revised-phase column (XSelect CSH C18, 4.6 mm × 50 mm).

$[\alpha]_D^{22}$ = -44.00 (*c* 1.00, CF_3CO_2H).

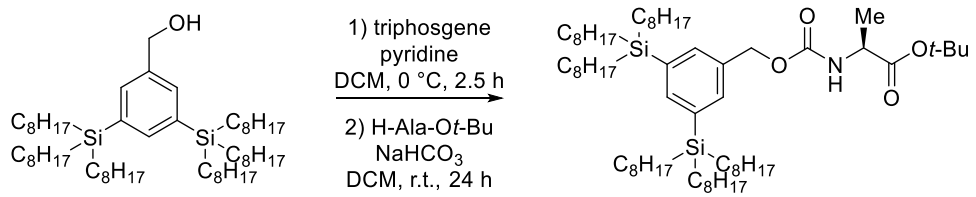
^{1}H NMR (400 MHz, CF_3CO_2D) δ 4.97 – 4.76 (m, 6H), 4.73 – 4.63 (m, 1H), 1.91 (d, *J* = 7.0 Hz, 3H), 1.78 (d, *J* = 7.2 Hz, 3H), 1.75 – 1.65 (m, 15H).

^{13}C NMR (100 MHz, CF_3CO_2D, lost one signal) δ 177.02, 176.96, 176.9, 176.8, 176.7, 176.3, 53.0, 52.5, 52.4(2C), 52.34, 52.28, 51.1, 18.35, 18.26, 18.2(2C), 18.1, 17.9, 17.6.

IR (neat) 3268, 3062, 2971, 1622, 1531, 1448, 1192, 1134, 1054 cm^{-1}.

HRMS (ESI) Calcd for $C_{21}H_{37}N_7O_8Na$ $[M+Na]^+$: 538.2601, Found: 538.2552.

XII. TAG6' at the N-terminal



HOTAG6 **TAG6'-Ala-O*t*-Bu**, 84%

***tert*-Butyl (((3,5-bis(trioctylsilyl)benzyl)oxy)carbonyl)-L-alaninate (TAG6'-Ala-O*t*-Bu)** At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and triphosgene (14.8 mg, 0.05 mmol, 0.5 equiv) was added dichloromethane (1 mL). Then, mixed **HOTAG6** (84.2 mg, 0.1 mmol, 1.0 equiv) and pyridine (8.1 μL, 0.1 mmol, 1.0 equiv) in dichloromethane (1 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. The resulting mixture was stirred for 2.5 h to form the chloroformate. At room temperature, under N_2, to another flame-dried flask charged with magnetic stirring bar (Sm-Co) and sodium bicarbonate (50.4 mg, 0.6 mmol, 6.0 equiv) was added dichloromethane (1 mL) and **H-Ala-O*t*-Bu** (29.0 mg, 0.2 mmol, 2.0 equiv). The resulting mixture was stirred for 30 min followed by cooling to 0 °C. The above chloroformate mixture was added into the reaction flask slowly. After stirring under room temperature for 24 h, the reaction mixture was filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 85:1 to 80:1) to afford the product **TAG6'-Ala-O*t*-Bu** as a colorless oil in 84% yield (85.4 mg).

Rf = 0.24 (hexanes/EtOAc = 20:1).

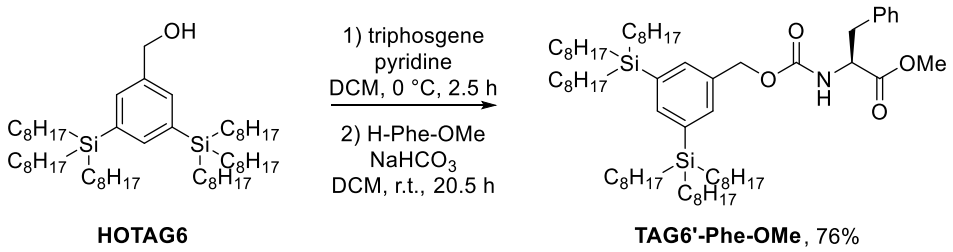
$[\alpha]_D^{24}$ = -28.71 (*c* 1.01, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (s, 1H), 7.41 (s, 2H), 5.32 (d, J = 7.7 Hz, 1H), 5.20 – 4.98 (m, 2H), 4.36 – 4.13 (m, 1H), 1.47 (s, 9H), 1.39 (d, J = 7.1 Hz, 3H), 1.36 – 1.17 (m, 72H), 0.92 – 0.83 (m, 18H), 0.81 – 0.72 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 155.6, 140.1, 137.2, 134.4, 134.1, 81.8, 67.6, 50.1, 33.8, 31.9, 29.32, 29.26, 27.9, 23.8, 22.7, 19.0, 14.1, 12.5.

IR (neat) 2956, 2920, 2852, 1725, 1501, 1457, 1410, 1377, 1368, 1338, 1314, 1217, 1158, 1068, 1051 cm^{-1}.

HRMS (ESI) Calcd for $C_{63}H_{121}NO_4Si_2Na$ $[M+Na]^+$: 1034.8732, Found: 1034.8706.



HOTAG6 **TAG6'-Phe-OMe**, 76%

Methyl (((3,5-bis(trioctylsilyl)benzyl)oxy)carbonyl)-L-phenylalaninate (TAG6'-Phe-OMe) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and triphosgene (29.7 mg, 0.1 mmol, 0.5 equiv) was added dichloromethane (2 mL). Then, mixed **HOTAG6** (168.3 mg, 0.2 mmol, 1.0 equiv) and pyridine (16.1 μL, 0.2 mmol, 1.0 equiv) in dichloromethane (2 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. The resulting mixture was stirred for 2.5 h to form the chloroformate. At room temperature, under N_2, to another flame-dried flask charged with magnetic stirring bar (Sm-Co) and sodium bicarbonate (100.8 mg, 1.2 mmol, 6.0 equiv) was added dichloromethane (2 mL) and **H-Phe-OMe** (71.7 mg, 0.4 mmol, 2.0 equiv). The resulting mixture was stirred for 30 min followed by cooling to 0 °C. The above chloroformate mixture was added into the reaction flask slowly. After stirring under room temperature for 20.5 h, the reaction mixture was filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 80:1 to 50:1 to 20:1) to afford the product **TAG6'-Phe-OMe** as a colorless oil in 76% yield (160.0 mg).

Rf = 0.21 (hexanes/EtOAc = 20:1).

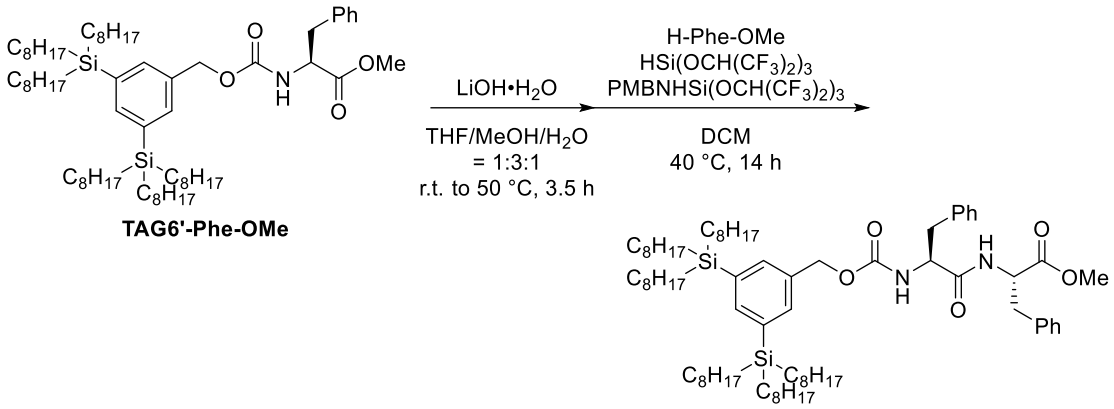
$[\alpha]_D^{25}$ = +25.74 (*c* 1.01, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.40 (s, 2H), 7.31 – 7.20 (m, 3H), 7.16 – 7.03 (m, 2H), 5.22 (d, *J* = 8.2 Hz, 1H), 5.15 – 5.03 (m, 2H), 4.76 – 4.64 (m, 1H), 3.72 (s, 3H), 3.22 – 3.02 (m, 2H), 1.38 – 1.19 (m, 72H), 0.95 – 0.84 (m, 18H), 0.83 – 0.71 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 155.7, 140.2, 137.3, 135.7, 134.5, 133.9, 129.2, 128.6, 127.1, 67.9, 54.8, 52.2, 38.3, 33.8, 31.9, 29.32, 29.26, 23.9, 22.7, 14.1, 12.5.

IR (neat) 2955, 2920, 2852, 1726, 1499, 1457, 1377, 1346, 1205, 1177, 1144 cm^{-1}.

HRMS (ESI) Calcd for $C_{66}H_{119}NO_4Si_2Na$ $[M+Na]^+$: 1068.8575, Found: 1068.8540.



TAG6'-Phe-Phe-OMe (27) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **TAG6'-Phe-OMe** (104.9 mg, 0.1 mmol, 1.0 equiv) was added tetrahydrofuran (0.1 mL), methanol (0.3 mL) and water (0.1 mL). The lithium hydroxide monohydrate (4.2 mg, 0.1 mmol, 1.0 equiv) was added. The reaction was stirred under room temperature for 1.5 h and then 50 °C for 2 h. After completion, the reaction mixture was concentrated. Chloroform (2 mL) was added, followed by adding hydrochloric acid solution (2 N in water, 50 μL, 0.1 mmol, 1.0 equiv). Water (2 mL) was added, and the layers were separated. The aqueous layer was extracted with chloroform (3×2 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was put into next step without further purification.

At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and above residue was added dichloromethane (0.1 mL), $HSi[OCH(CF_3)_2]_3$ (79.5 mg, 0.15 mol, 1.5 quiv), **H-Phe-OMe** (26.9 mg, 0.15 mmol, 1.5 equiv) and $PMBNHSi[OCH(CF_3)_2]_3$ (1.0 M in dichloromethane, 3.0 μL, 0.003 mmol, 0.03 quiv) in the glove box. [***Note**: HSi[OCH(CF₃)₂]₃ and PMBNHSi[OCH(CF₃)₂]₃ was prepared according to the procedure in the literature.* [8]] The vial was sealed and taken out of the glove box. The reaction was stirred under 40 °C for 14 h. After completion, the reaction mixture was transferred onto silica gel column by a pipette and purified by silica gel chromatography (eluent: hexanes/EtOAc = 7:1) to afford the product **27** as a colorless oil in 85% total yield (101.3 mg).

Rf = 0.34 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{22}$ = +20.20 (*c* 0.99, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (s, 1H), 7.41 (s, 2H), 7.33 – 7.10 (m, 8H), 7.05 – 6.92 (m, 2H), 6.20 (d, J = 7.3 Hz, 1H), 5.25 (d, J = 8.0 Hz, 1H), 5.18 – 4.94 (m, 2H), 4.86 – 4.74 (m, 1H), 4.52 – 4.35 (m, 1H), 3.68 (s, 3H), 3.22 – 2.92 (m, 4H), 1.42 – 1.16 (m, 72H), 0.97 – 0.84 (m, 18H), 0.84 – 0.68 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 170.3, 155.9, 140.2, 137.4, 136.2, 135.5, 134.4, 133.8, 129.3, 129.2, 128.7, 128.5, 127.1, 127.0, 68.0, 56.1, 53.3, 52.3, 38.3, 37.9, 33.8, 31.9, 29.32, 29.26, 23.8, 22.7, 14.1, 12.5.

IR (neat) 2955, 2920, 2852, 1746, 1711, 1664, 1498, 1456, 1377, 1213, 1177, 1144, 1109, 1078, 1031 cm^{-1}.

HRMS (ESI) Calcd for $C_{75}H_{128}N_2O_5Si_2Na$ $[M+Na]^+$: 1215.9259, Found: 1215.9275.

XIII. References

[1] Fraczyk, J.; Kaminski, Z. J.; Katarzynska, J.; Kolesinska, B. *Helv. Chim. Acta* **2018**, *101*, e1700187.

[2] Baker, E. L.; Yamano, M. M.; Zhou, Y.; Anthony, S. M.; Garg, N. K. *Nat. Commun.* **2016**, *7*, 11554.

[3] Tamiaki, H.; Obata, T.; Azefu, Y.; Toma, K. *Bull. Chem. Soc. Jpn.* **2001**, *74*, 733-738.

[4] Pan, S.; Jang, S.-Y.; Wang D.; Liew, S. S.; Li, Z.; Lee, J.-S.; Yao. S. Q. *Angew. Chem., Int. Ed.* **2017**, *56*, 11816-11821.

[5] Thota, K. K.; Trudell, M. L. *Synthesis* **2013**, *45*, 2280-2286.

[6] Muramatsu, W.; Hattori, T.; Yamamoto, H. *J. Am. Chem. Soc.* **2019**, *141*, 12288-12295.

[7] Henze, O.; Lehmann, U.; Schlüter, A. D. *Synthesis* **1999**, *31*, 683-687.

[8] Muramatsu, W.; Manthena, C.; Nakashima, E.; Yamamoto, H. *ACS Catal.* **2020**, *10*, 9594-9603.

Fmoc-DL-Val-SNp

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Sample ID :
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Vial# : 16
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Batch File : an.lcb
Report Format File : DEFAULT.lsr
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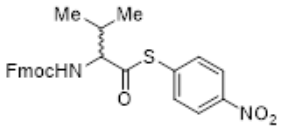
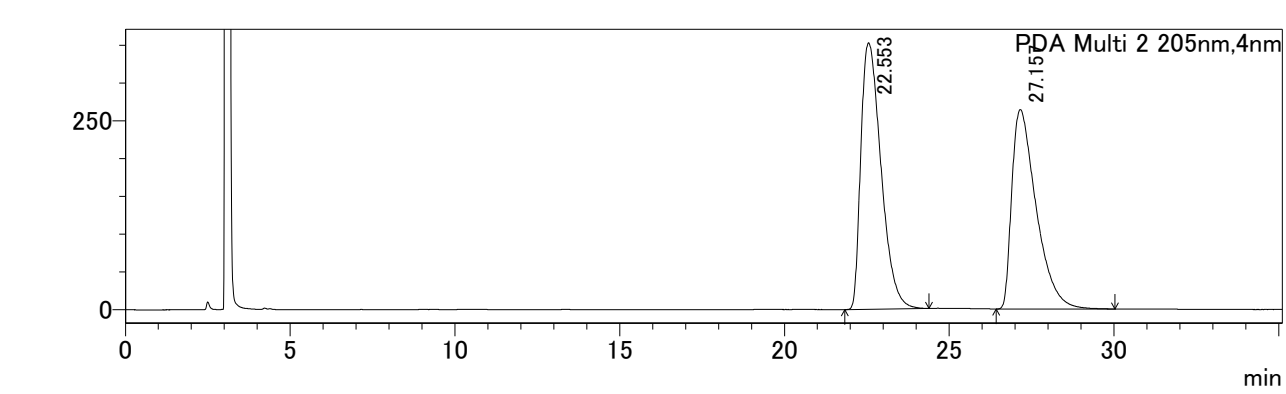
Date Processed : 2020/06/17 13:25:57





Peak Table

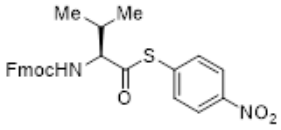
PDA Ch2 205nm

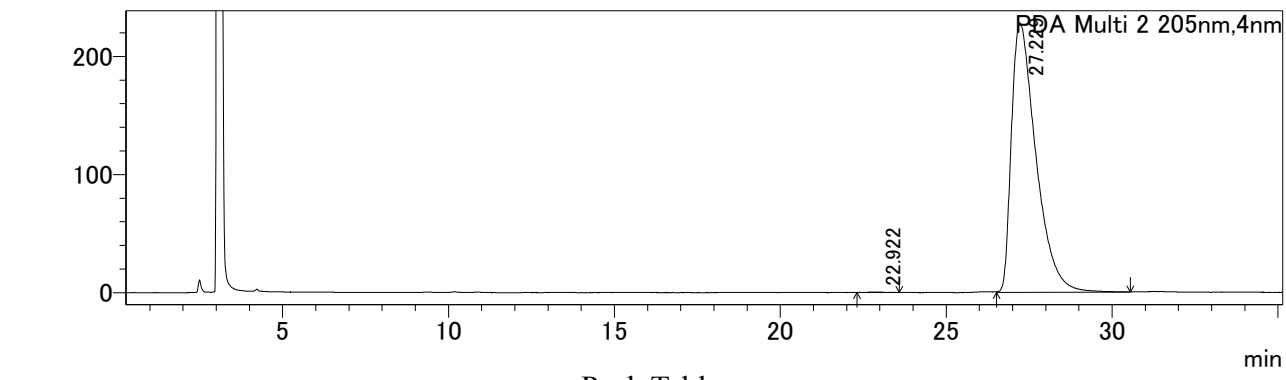
Peak#	Ret. Time	Area	Area%
1	22.553	15020245	52.506
2	27.157	13586219	47.494
Total		28606464	100.000

22.553 min = Fmoc-D-Val-SNp, 27.157 min = Fmoc-Val-SNp
Conditions: 2-propanol/hexane = 10:90, v = 1.5 mL/min, λ = 205 nm
Chiral Column: IE-3 column from Daicel Chemical Ind., Ltd.

Fmoc-Val-SNp

Acquired by : System Administrator
Sample Name :
Sample ID :
Tray# : 1
Vial# : 17
Injection Volume : 5
Data File : wuan-541.lcd
Method File : IE_90_10_1.5ml_40min.lcm
Batch File : an.lcb
Report Format File : DEFAULT.lsr
Date Acquired : 2020/06/17 13:24:03
Date Processed : 2020/06/17 14:40:21





Peak Table

PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
1	22.922	10522	0.090
2	27.229	11737898	99.910
Total		11748420	100.000

Fmoc-DL-Lys(Boc)-SNp

Acquired by : System Administrator
Sample Name :
Sample ID :
Tray# : 1
Vial# : 16
Injection Volume : 5
Data File : wuan-561+562-IB-90-10-1.0ml.lcd
Method File : IB_90_10_1ml_60min.lcm
Batch File : Test_85_15_1.0ml_60min.lcb
Report Format File : DEFAULT.lsr
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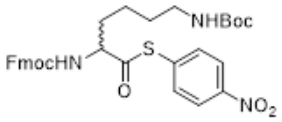
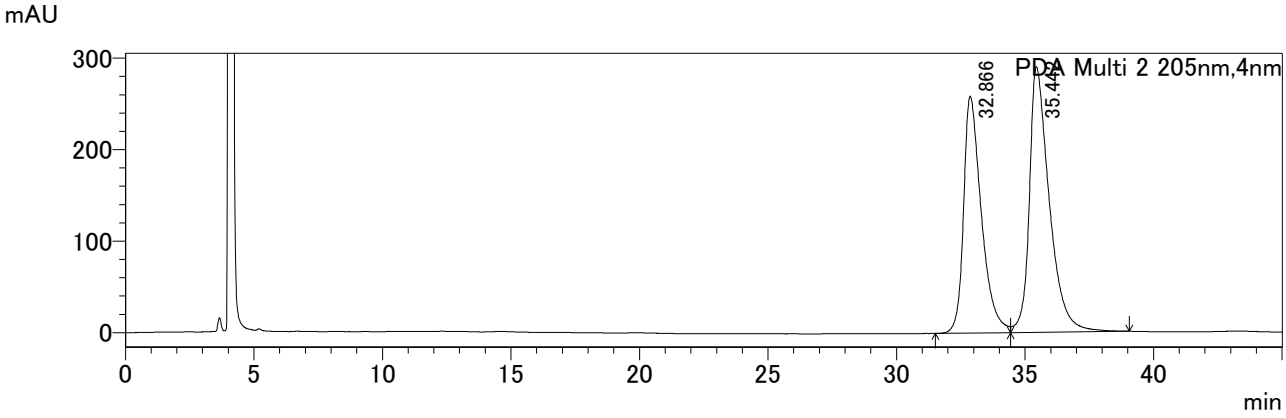
Date Processed : 2020/11/10 13:37:35





Peak Table

PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
1	32.866	12472865	44.336
2	35.442	15659956	55.664
Total		28132821	100.000

32.866 min = Fmoc-D-Lys(Boc)-SNp, 35.442 min = Fmoc-Lys(Boc)-SNp
Conditions: 2-propanol/hexane = 10:90, v = 1.0 mL/min, λ = 205 nm
Chiral Column: IB-3 column from Daicel Chemical Ind., Ltd.

Fmoc-Lys(Boc)-SNp

Acquired by : System Administrator
Sample Name :
Sample ID :
Tray# : 1
Vial# : 17
Injection Volume : 5
Data File : wuan-561-1-IB-90-10-1.0ml.lcd
Method File : IB_90_10_1ml_45min.lcm
Batch File : Test_85_15_1.0ml_60min.lcb
Report Format File : DEFAULT.lsr
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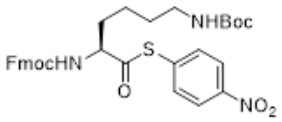
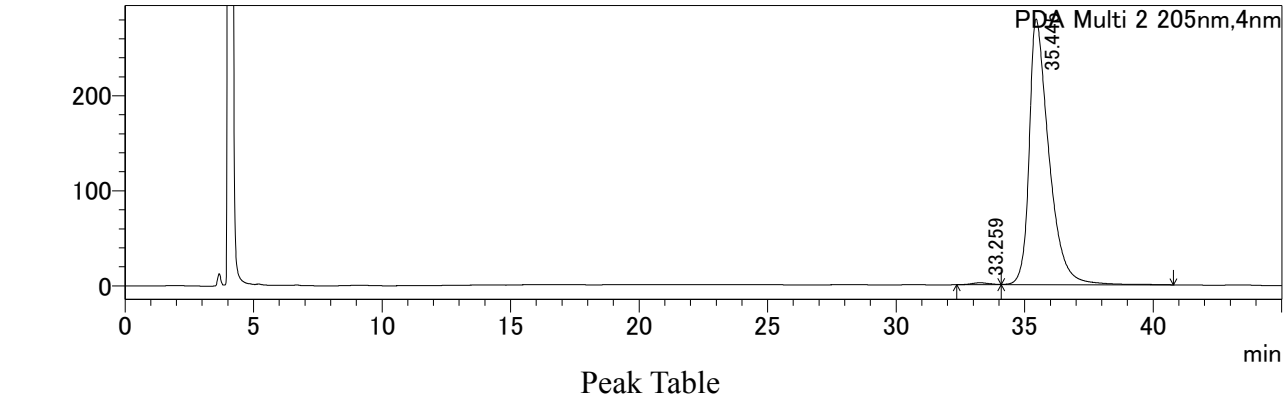
Date Processed : 2020/11/10 14:40:43





Peak Table

PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
1	33.259	104303	0.693
2	35.445	14946550	99.307
Total		15050852	100.000

Fmoc-DL-Pro-SNp

Acquired by	: System Administrator
Sample Name	:
Sample ID	:
Tray#	: 1
Vial#	: 16
Injection Volume	: 5
Data File	: wuan-589+741-IA-90-10-1.0ml.lcd
Method File	: IA_90_10_1.0ml_60min.lcm
Batch File	: Test_85_15_1.0ml_60min.lcb
Report Format File	: DEFAULT.lsr
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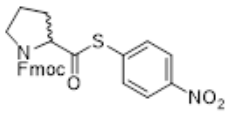

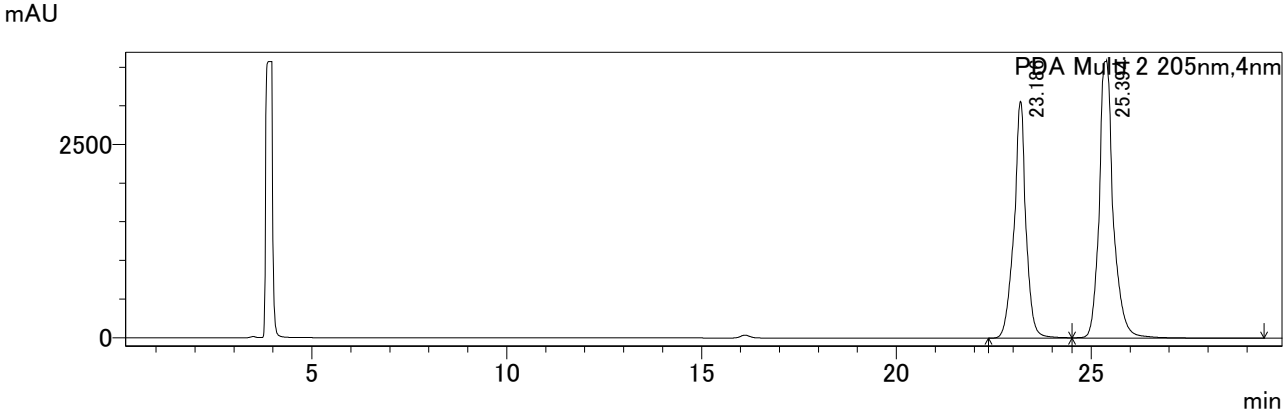

Peak Table

PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
1	23.186	65572332	42.029
2	25.394	90444606	57.971
Total		156016938	100.000

23.186 min = Fmoc-Pro-SNp, 25.394 min = Fmoc-D-Pro-SNp
Conditions: 2-propanol/hexane = 10:90, v = 1.0 mL/min, λ = 205 nm
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd

Fmoc-Pro-SNp

Acquired by	: System Administrator
Sample Name	:
Sample ID	:
Tray#	: 1
Vial#	: 17
Injection Volume	: 5
Data File	: wuan-589.lcd
Method File	: IA_90_10_1.0ml_30min.lcm
Batch File	: Test_85_15_1.0ml_60min.lcb
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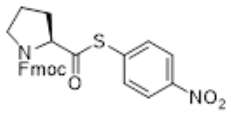

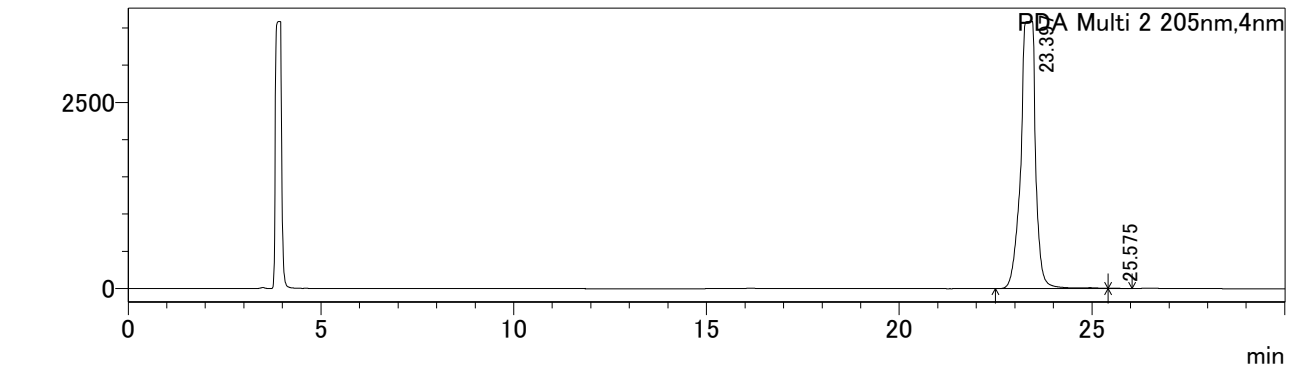

Peak Table

PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
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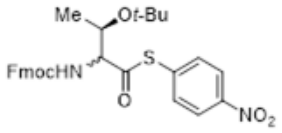
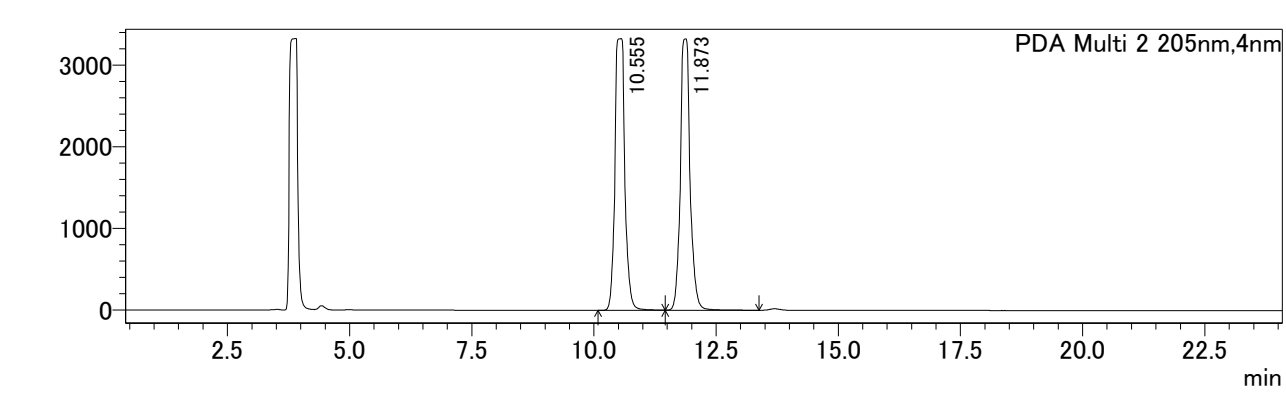
2	25.575	130305	0.135
Total		96453491	100.000

Fmoc-DL-Thr(*t*-Bu)-SNp

Acquired by : System Administrator
Sample Name :
Sample ID :
Tray# : 1
Vial# : 31
Injection Volume : 5
Data File : wuan-647.lcd
Method File : IA_85_15_1.0ml_60min.lcm
Batch File : An.lcb
Report Format File : DEFAULT.lsr
Date Acquired : 2021/10/27 12:09:36
Date Processed : 2021/10/27 13:40:32





Peak Table

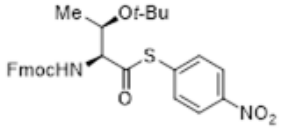
PDA Ch2 205nm

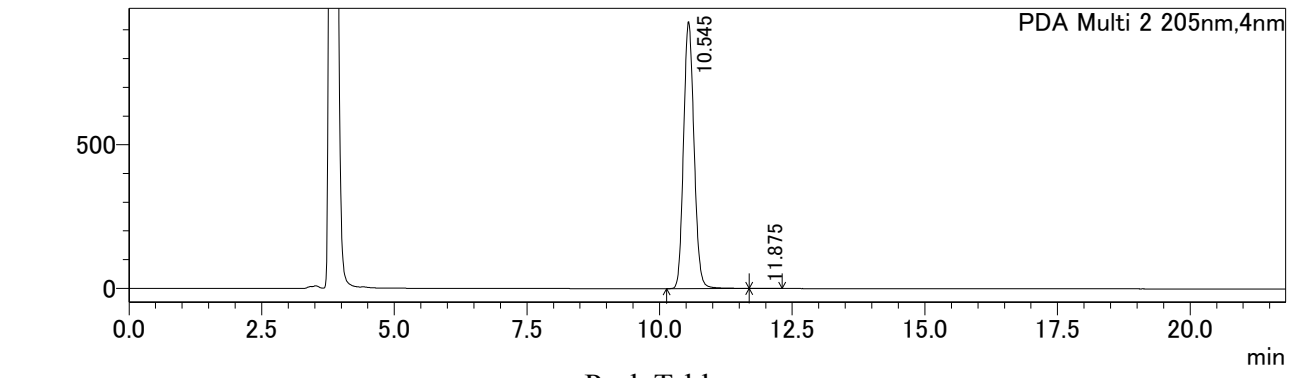
Peak#	Ret. Time	Area	Area%
1	10.555	47039310	50.452
2	11.873	46196212	49.548
Total		93235521	100.000

10.555 min = Fmoc-Thr(*t*-Bu)-SNp, 11.873 min = Fmoc-D-Thr(*t*-Bu)-SNp
Conditions: 2-propanol/hexane = 15:85, v = 1.0 mL/min, λ = 205 nm
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd.

Fmoc-Thr(*t*-Bu)-SNp

Acquired by : System Administrator
Sample Name :
Sample ID :
Tray# : 1
Vial# : 32
Injection Volume : 5
Data File : wuan-647A.lcd
Method File : IA_85_15_1.0ml_30min.lcm
Batch File : An.lcb
Report Format File : DEFAULT.lsr
Date Acquired : 2021/10/27 13:10:10
Date Processed : 2021/10/27 13:56:00





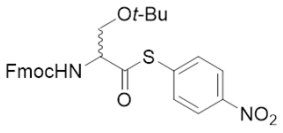
Peak Table

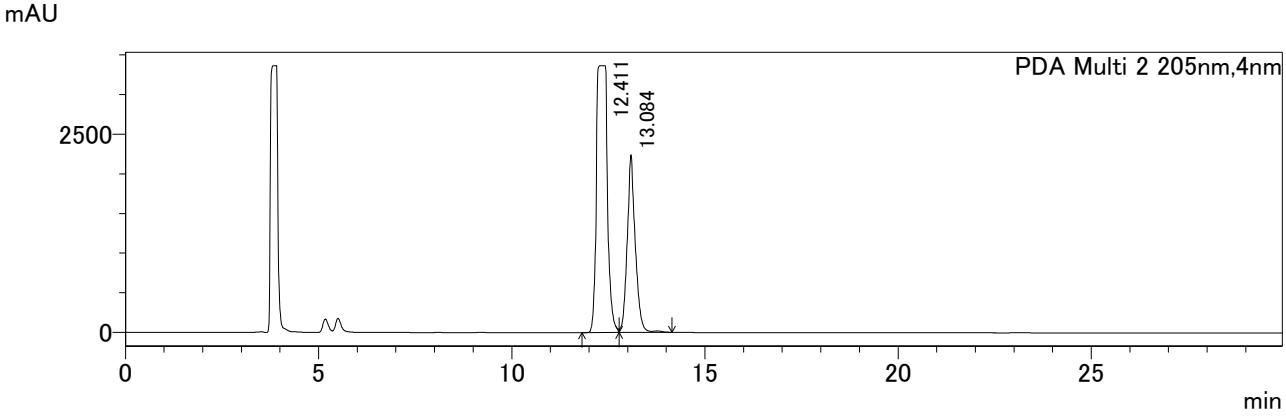
PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
1	10.545	12753482	99.883
2	11.875	14879	0.117
Total		12768361	100.000

Fmoc-DL-Ser(*t*-Bu)-SNp

Acquired by : System Administrator
Sample Name :
Sample ID :
Tray# : 1
Vial# : 16
Injection Volume : 5
Data File : wuan-664.lcd
Method File : IA_85_15_1.0ml_60min.lcm
Batch File : An.lcb
Report Format File : DEFAULT.lsr
Date Acquired : 2021/12/07 10:15:16
Date Processed : 2021/12/07 10:51:24





Peak Table

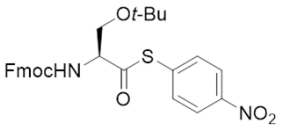
PDA Ch2 205nm

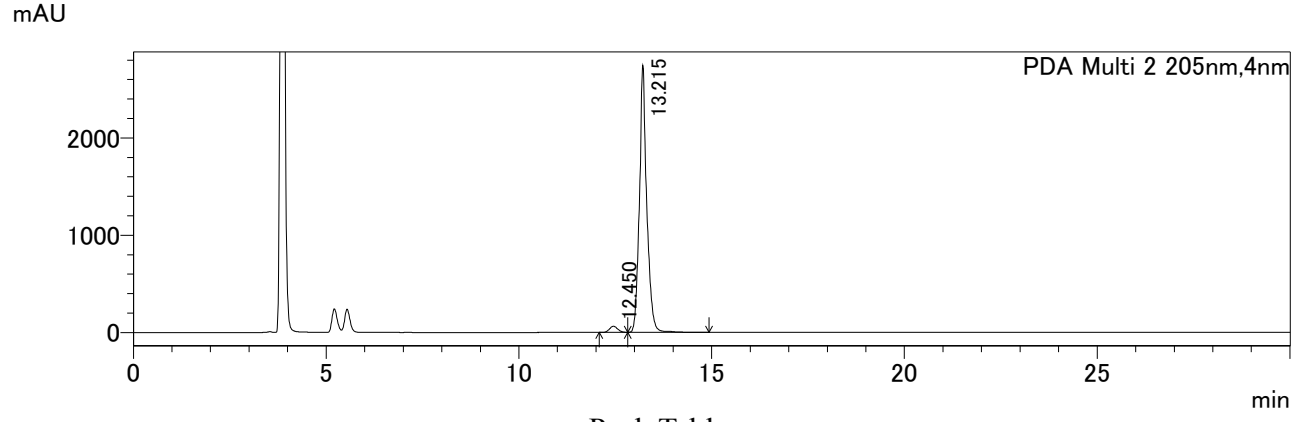
Peak#	Ret. Time	Area	Area%
1	12.411	66275099	67.031
2	13.084	32597020	32.969
Total		98872120	100.000

12.411 min = Fmoc-D-Ser(*t*-Bu)-SNp, 13.084 min = Fmoc-Ser(*t*-Bu)-SNp
Conditions: 2-propanol/hexane = 15:85, v = 1.0 mL/min, λ = 205 nm
Chiral Column: IA-3 column from Daicel Chemical Ind., Ltd.

Fmoc-Ser(*t*-Bu)-SNp

Acquired by : System Administrator
Sample Name :
Sample ID :
Tray# : 1
Vial# : 17
Injection Volume : 5
Data File : wuan-664C.lcd
Method File : IA_85_15_1.0ml_30min.lcm
Batch File : An.lcb
Report Format File : DEFAULT.lsr
Date Acquired : 2021/12/07 18:54:37
Date Processed : 2021/12/07 19:26:13





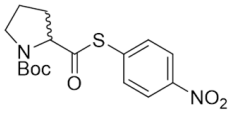
Peak Table

PDA Ch2 205nm

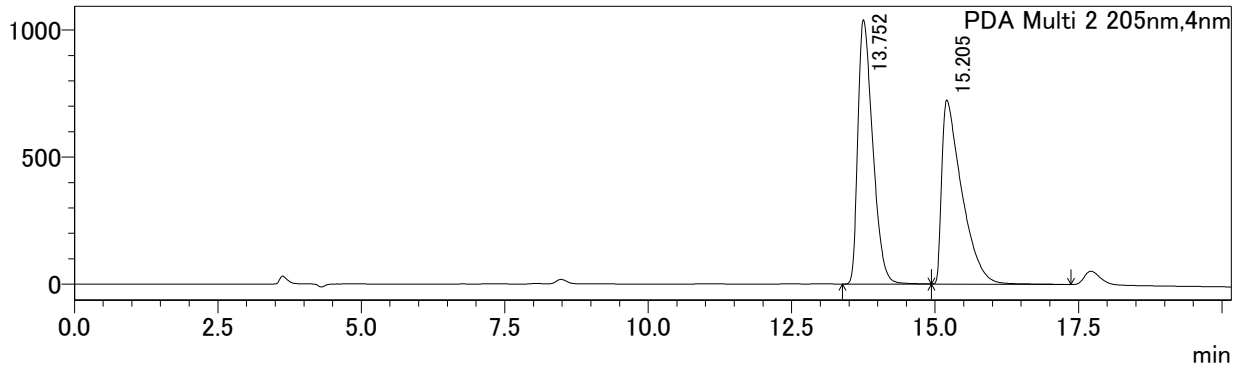
Peak#	Ret. Time	Area	Area%
1	12.450	953911	2.554
2	13.215	36397326	97.446
Total		37351236	100.000

Boc-DL-Pro-SNp

Acquired by	: System Administrator
Sample Name	:
Sample ID	:
Tray#	: 1
Vial#	: 46
Injection Volume	: 5
Data File	: wuan-677+680-ID-90-10-1.0ml.lcd
Method File	: ID_90_10_1ml_60min.lcm
Batch File	: Test - ƒRƒs[.lcb
Report Format File	: DEFAULT.lsr
Date Acquired	: 2020/10/01 18:28:42
Date Processed	: 2020/10/01 18:51:52



mAU



Peak Table

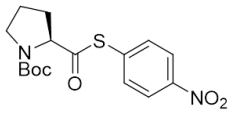
PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
1	13.752	18793050	51.496
2	15.205	17701222	48.504
Total		36494272	100.000

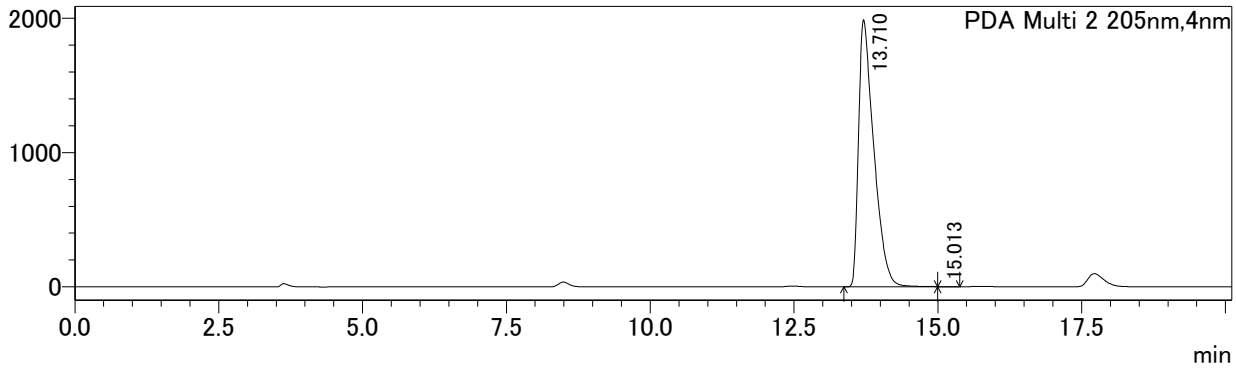
13.752 min = Boc-Pro-SNp, 15.205 min = Boc-D-Pro-SNp
Conditions: 2-propanol/hexane = 10:90, v = 1.0 mL/min, λ = 205 nm
Chiral Column: ID-3 column from Daicel Chemical Ind., Ltd.

Boc-Pro-SNp

Acquired by	: System Administrator
Sample Name	:
Sample ID	:
Tray#	: 1
Vial#	: 47
Injection Volume	: 5
Data File	: wuan-677-ID-90-10-1.0ml.lcd
Method File	: ID_90_10_1ml_60min.lcm
Batch File	: Test - ƒRƒs[.lcb
Report Format File	: DEFAULT.lsr
Date Acquired	: 2020/10/01 19:45:37
Date Processed	: 2020/10/01 20:08:33



mAU



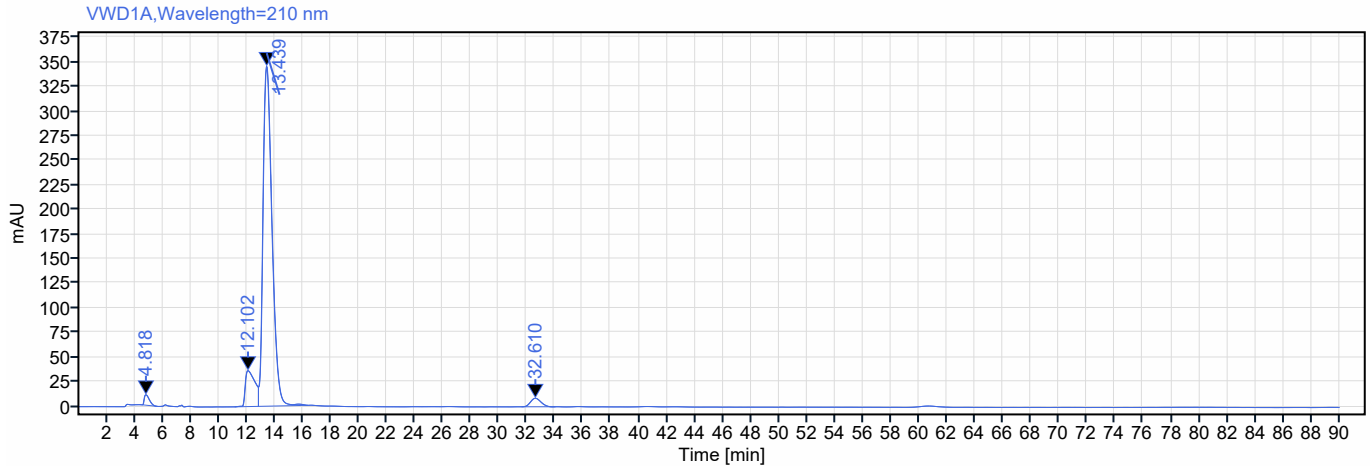
Peak Table

PDA Ch2 205nm

Peak#	Ret. Time	Area	Area%
1	13.710	36637963	99.971
2	15.013	10665	0.029
Total		36648628	100.000

Boc-Pro-Ser(t-Bu)-Lys(Boc)-Lys(Boc)-Thr(t-Bu)-Lys(Boc)-Pro-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OH (22)

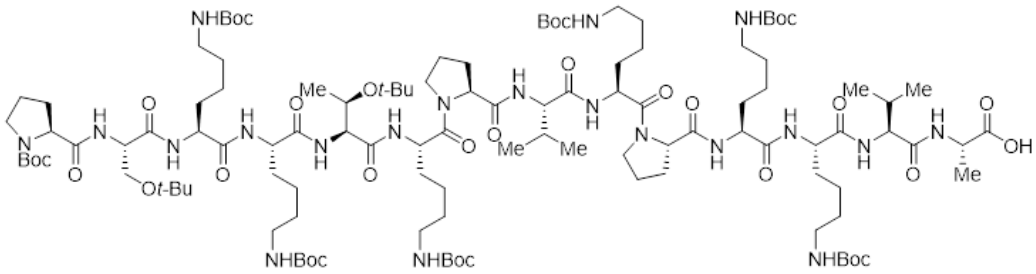
Data file:	wuan-1230-4-8.dx		
Sequence Name:	SingleSample	**Project Name:**	Yamamoto-Lab
Sample name:	wuan-1230-4	**Operator:**	SYSTEM
Instrument:	HPLC2	**Injection date:**	2022-01-12 17:33:46+09:00
Inj. volume:	0.000	**Location:**	
Acq. method:	ODS-HL_H2O85_MeCN15_0.5ml_rt_60min_210nm.amx	**Type:**	Sample
Processing method:	New method 1.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: VWD1A,Wavelength=210 nm

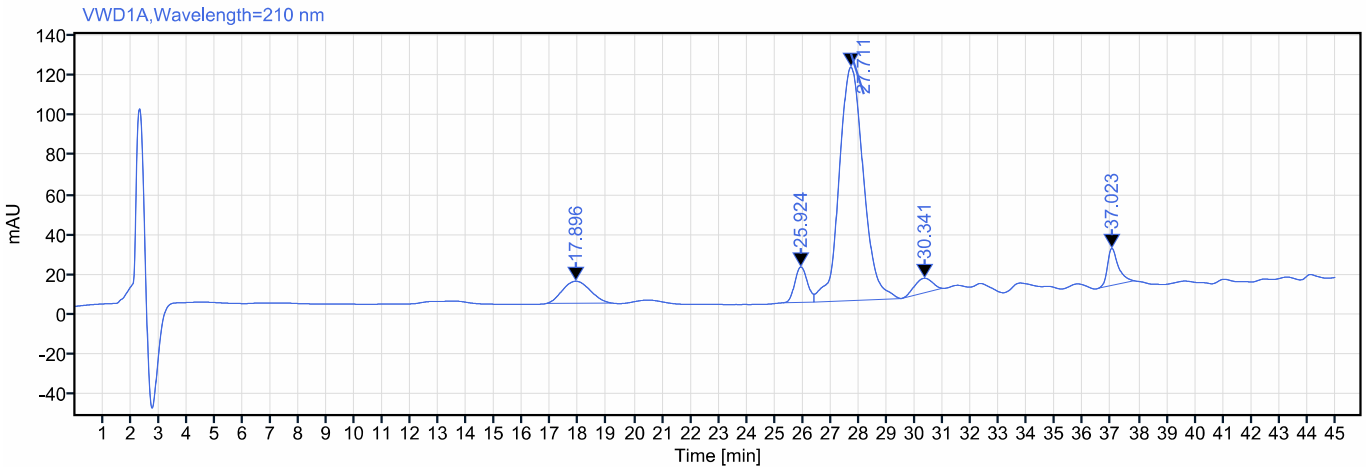
RT [min]	Type	Width [min]	Area	Height	Area%	Name
4.818	MM m	1.08	266.31	10.51	1.49	
12.102	BM m	1.69	1742.91	36.50	9.72	
13.439	MM m	3.54	15475.21	345.47	86.32	
32.610	MM m	2.41	444.29	8.66	2.48	
		Sum	**17928.74**			

13.439 min = Boc-Pro-Ser(t-Bu)-Lys(Boc)-Lys(Boc)-Thr(t-Bu)-Lys(Boc)-Pro-Val-Lys(Boc)-Pro-Lys(Boc)-Lys(Boc)-Val-Ala-OH (**22**)
Conditions: water/acetonitrile = 85:15, v = 0.5 mL/min, λ = 210 nm
Column: ODS-HL column from GL Siences Inc.



H-Ala-Ala-Ala-Ala-Ala-Ala-Ala-OH (26)

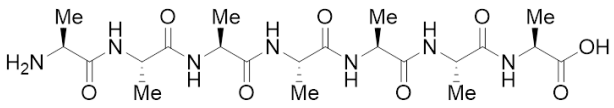
Data file:	wuan-1148-3-3.dx		
Sequence Name:	SingleSample	**Project Name:**	Yamamoto-Lab
Sample name:	wuan-1148-3	**Operator:**	SYSTEM
Instrument:	HPLC2	**Injection date:**	2022-05-31 16:19:46+09:00
Inj. volume:	0.000	**Location:**	
Acq. method:	C18_0.1%TFA-H2O100_0.5ml_40deg_50min_210nm.amx	**Type:**	Sample
Processing method:	New method 1.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		

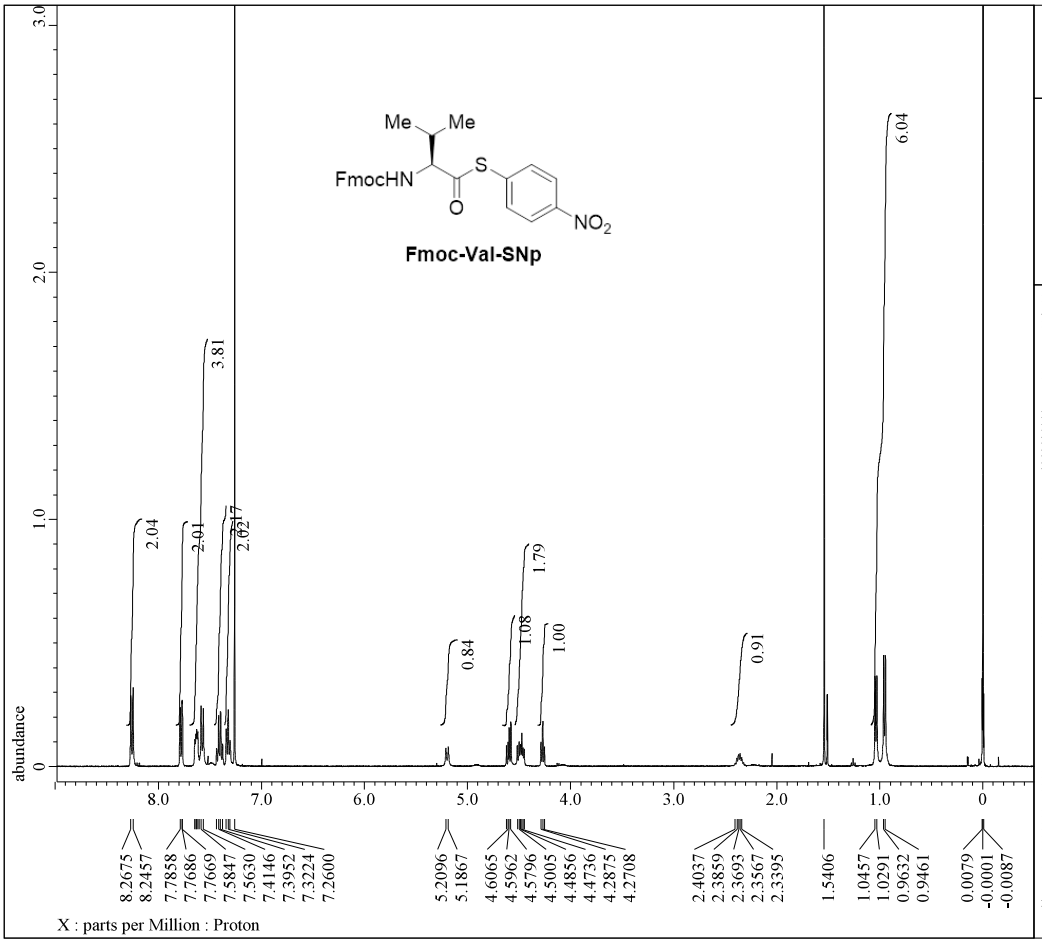


Signal: VWD1A,Wavelength=210 nm

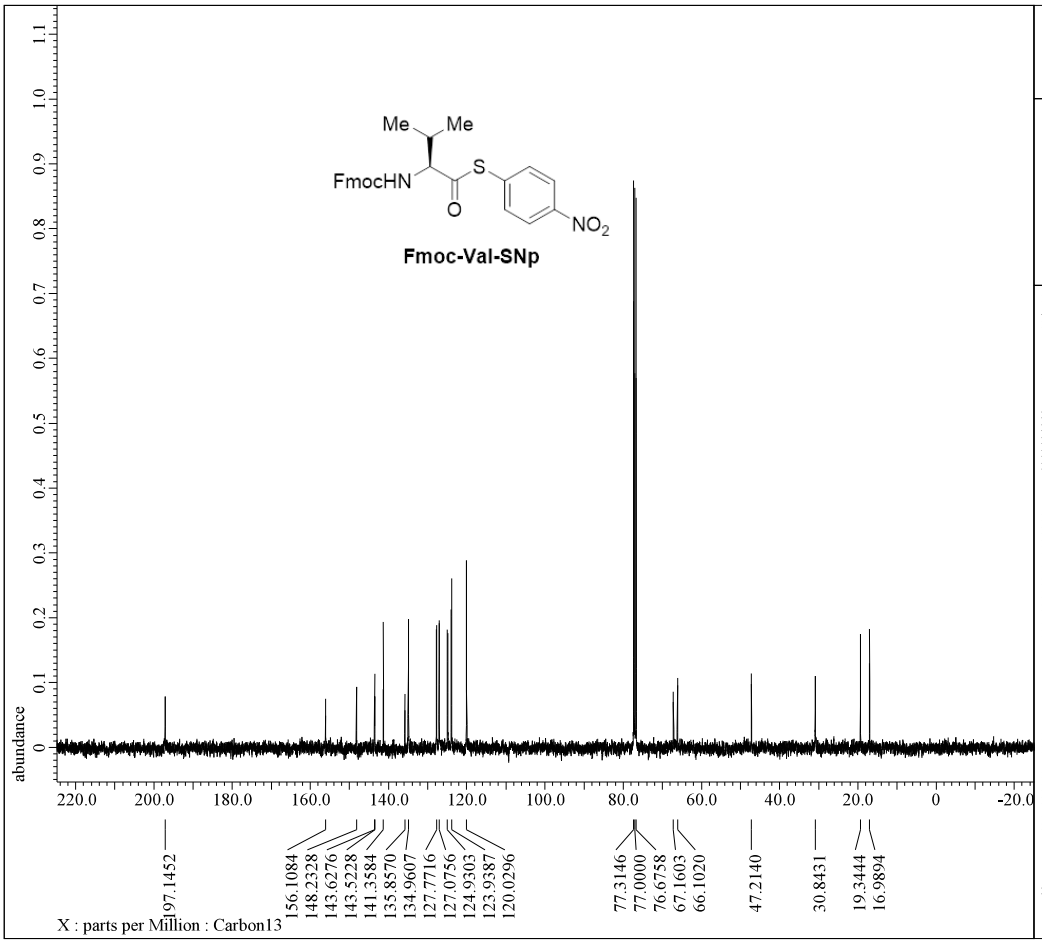
RT [min]	Type	Width [min]	Area	Height	Area%	Name
17.896	MM m	2.29	751.08	11.03	8.14	
25.924	MM m	1.12	559.95	17.83	6.07	
27.711	VB	3.10	7087.65	117.55	76.85	
30.341	MM m	1.47	319.39	7.22	3.46	
37.023	MM m	1.27	504.51	18.63	5.47	
		Sum	**9222.58**			

27.711 min = H-Ala-Ala-Ala-Ala-Ala-Ala-Ala-OH (**26**)
Conditions: 0.1% TFA in water/0.1% TFA in acetonitrile, gradient 100:0 (0-20 min), 100:0 to 90:10 (20-45 min)
v = 0.5 mL/min, λ = 210 nm
Column: XSelect CSH C18 column from Waters

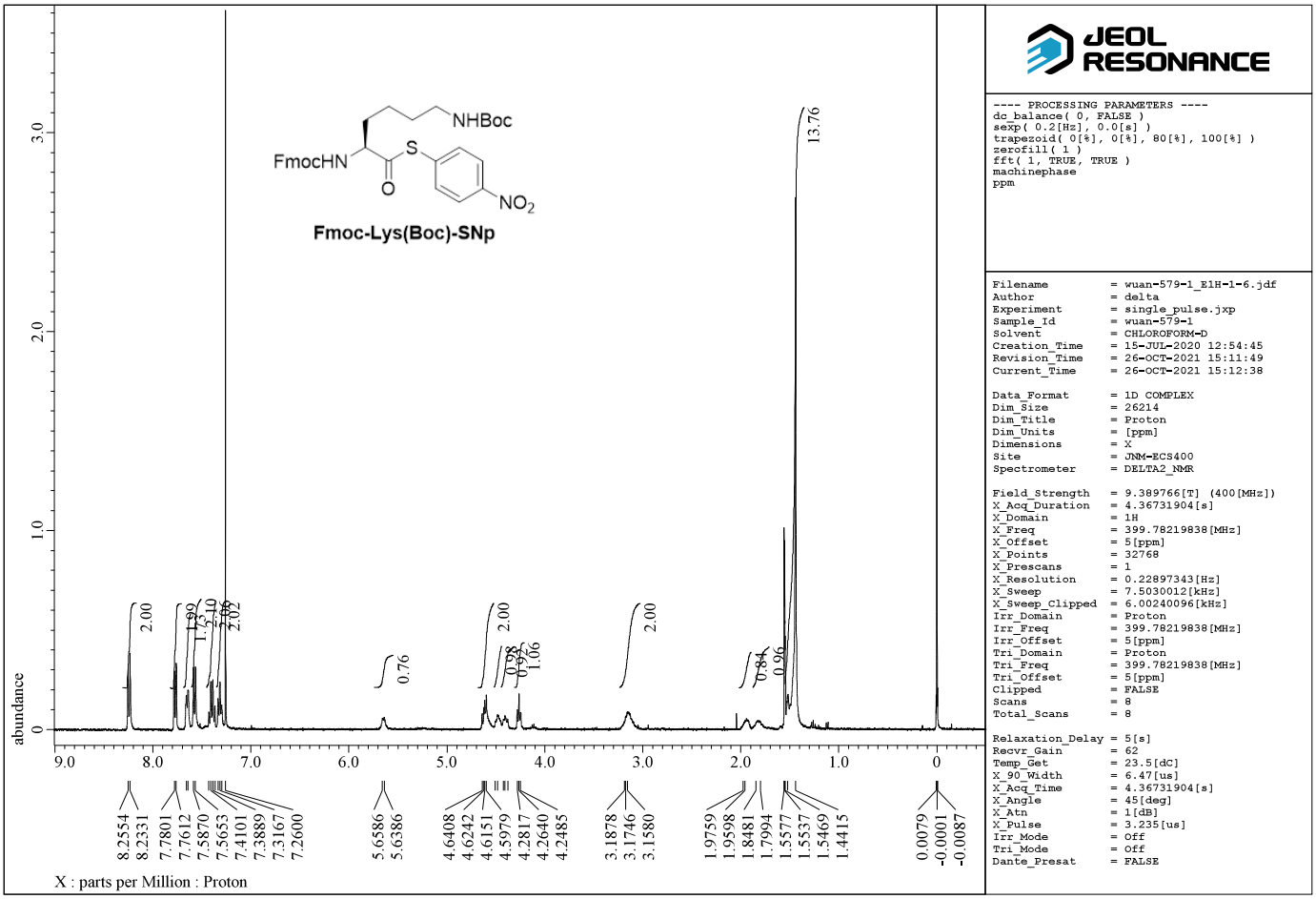





```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = wuan-577_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-577
Solvent           = CHLOROFORM-D
Creation_Time     = 14-JUL-2020 19:24:24
Revision_Time     = 26-OCT-2021 14:52:42
Current_Time      = 26-OCT-2021 14:53:40

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        = 50
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
```



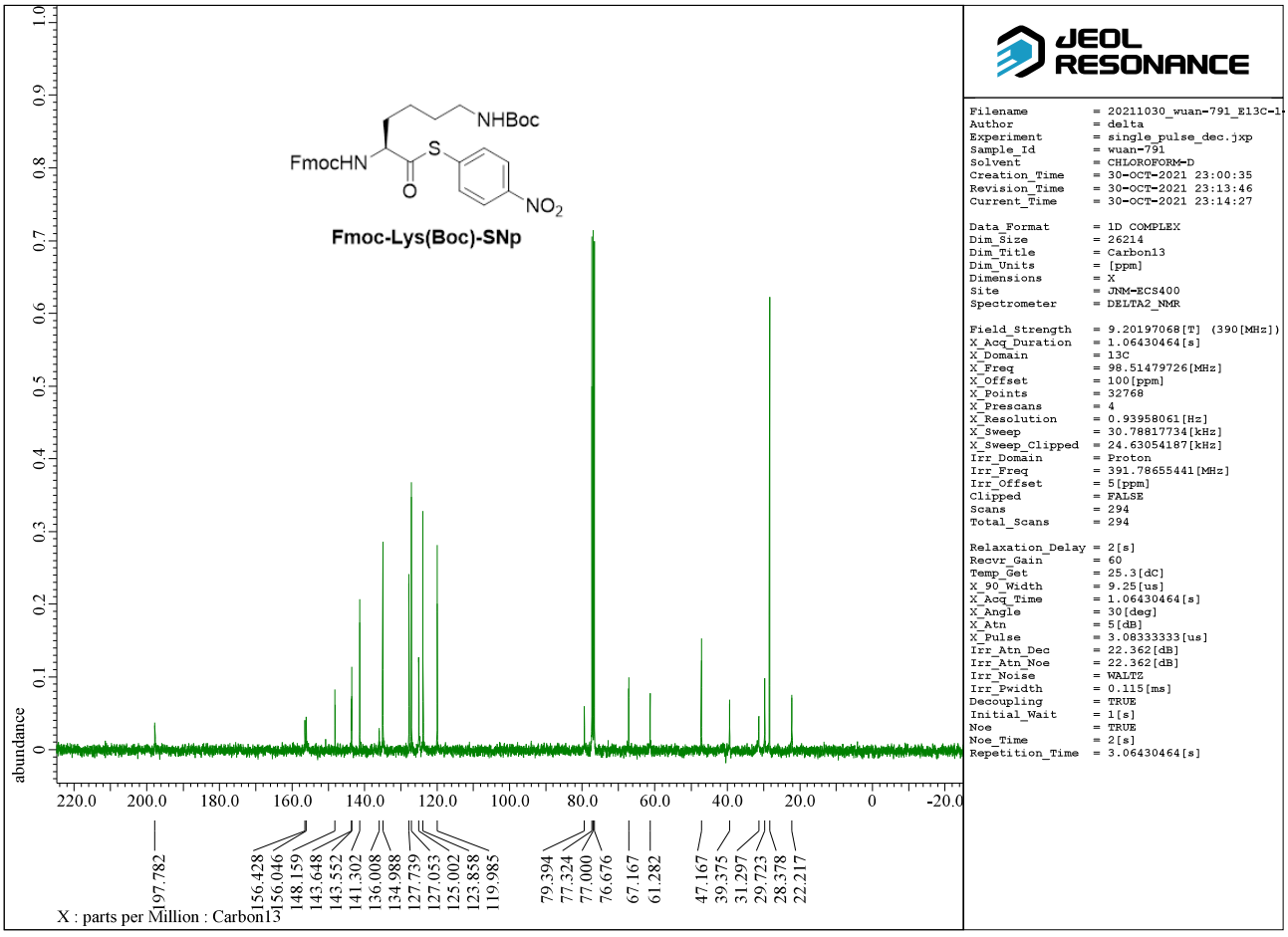

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-577_E13C-1-1.jdf

Filename          = wuan-577_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-577
Solvent           = CHLOROFORM-D
Creation_Time     = 26-OCT-2021 14:56:52
Revision_Time     = 26-OCT-2021 15:03:11
Current_Time      = 26-OCT-2021 15:03:26

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             = 165
Total_Scans       = 165

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]
```



JEOL RESONANCE

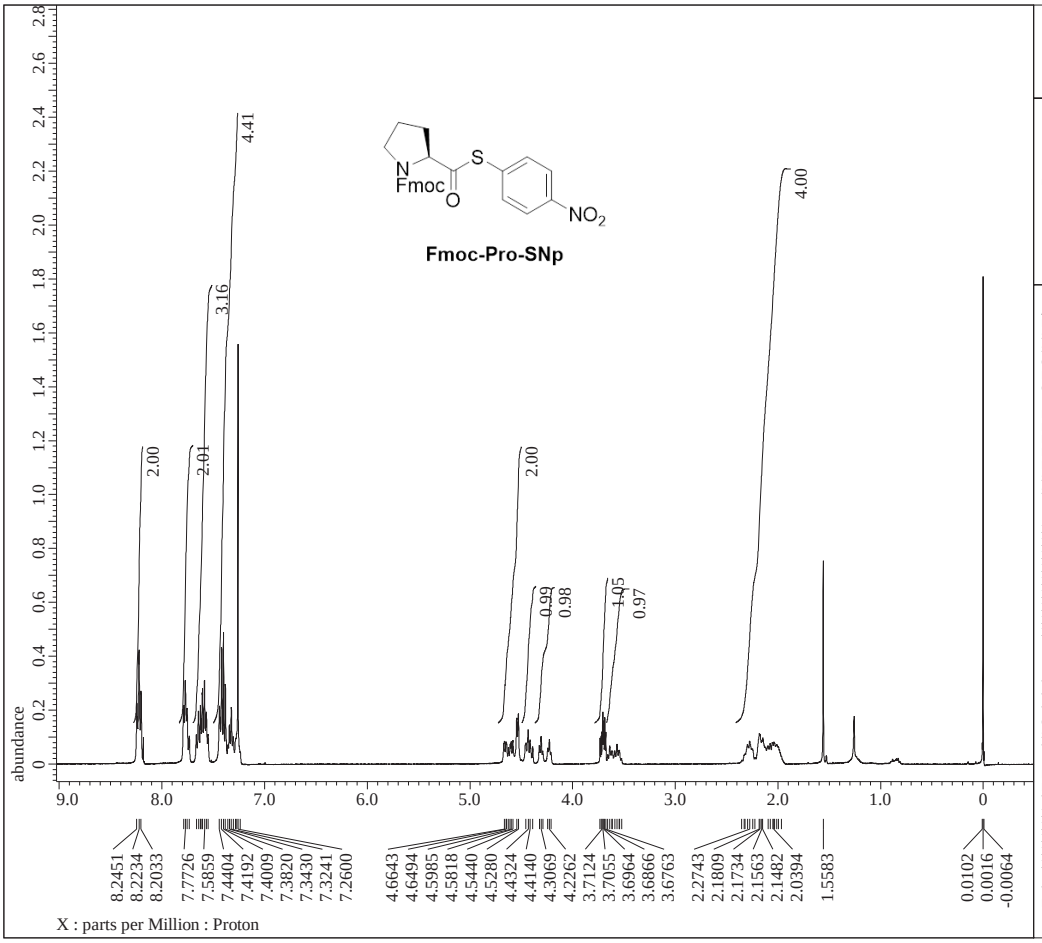
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= wuan-579-1_E1H-1-6.jdf
Author	= delta
Experiment	= single_pulse.jxp
Sample_Id	= wuan-579-1
Solvent	= CHLOROFORM-D
Creation_Time	= 15-JUL-2020 12:54:45
Revision_Time	= 26-OCT-2021 15:11:49
Current_Time	= 26-OCT-2021 15:12:38
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	= 62
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



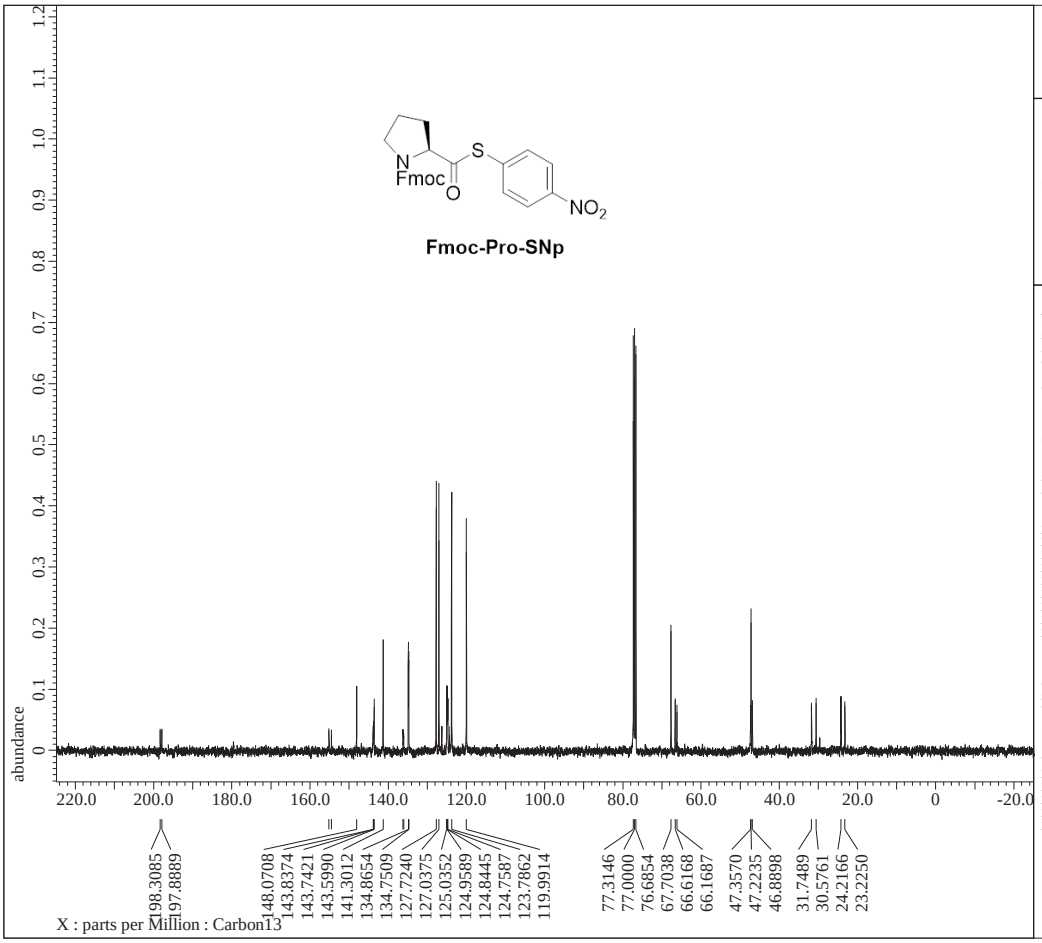
JEOL RESONANCE

Filename	= 20211030_wuan-791_E13C-1
Author	= delta
Experiment	= single_pulse_dec.jxp
Sample_Id	= wuan-791
Solvent	= CHLOROFORM-D
Creation_Time	= 30-OCT-2021 23:00:35
Revision_Time	= 30-OCT-2021 23:13:46
Current_Time	= 30-OCT-2021 23:14:27
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
Irr_Domain	= Proton
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 294
Total_Scans	= 294
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 25.3[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.08333333[us]
Irr_Atn_Dec	= 22.362[dB]
Irr_Atn_Noe	= 22.362[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.06430464[s]



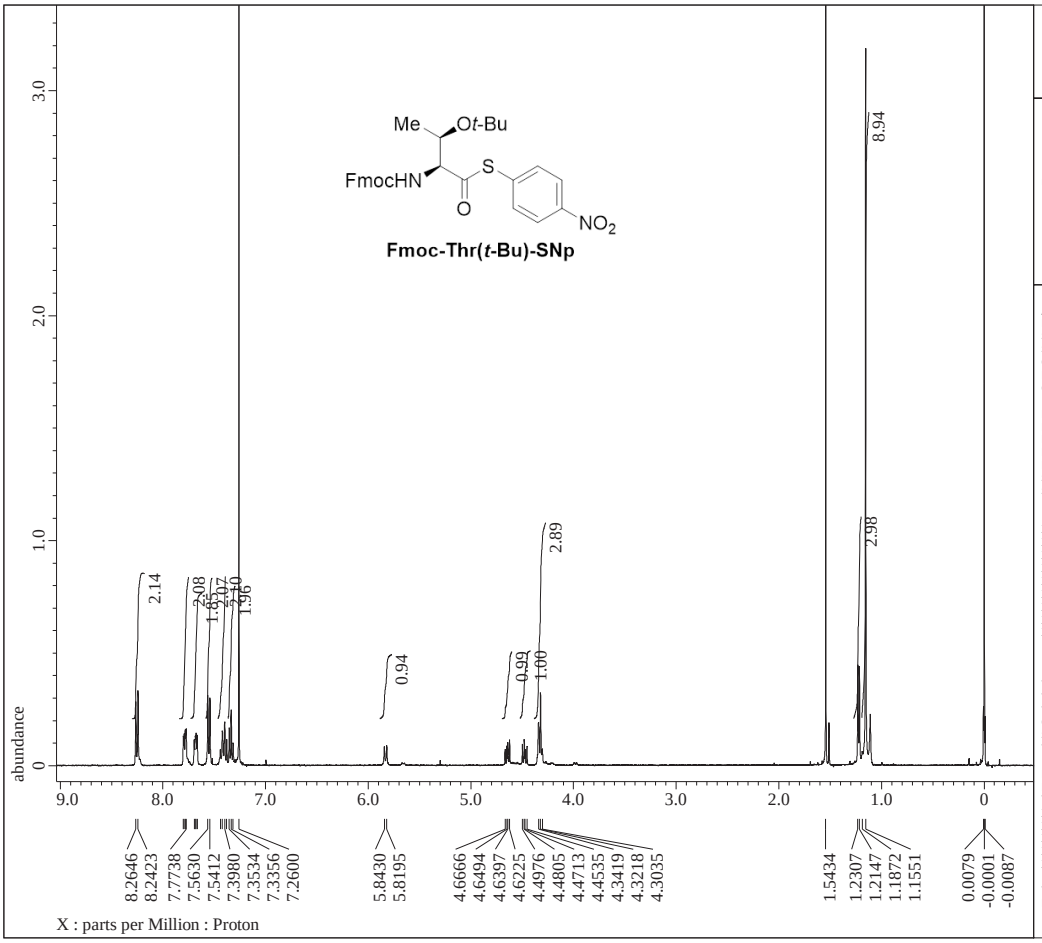

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-799_E1H-1-1.jdf
```

```
Filename          = wuan-799_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-799
Solvent           = CHLOROFORM-D
Creation_Time     = 26-OCT-2021 18:00:12
Revision_Time     = 26-OCT-2021 18:06:18
Current_Time      = 26-OCT-2021 18:07:09

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        = 42
Temp_Get          = 24.3[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
```



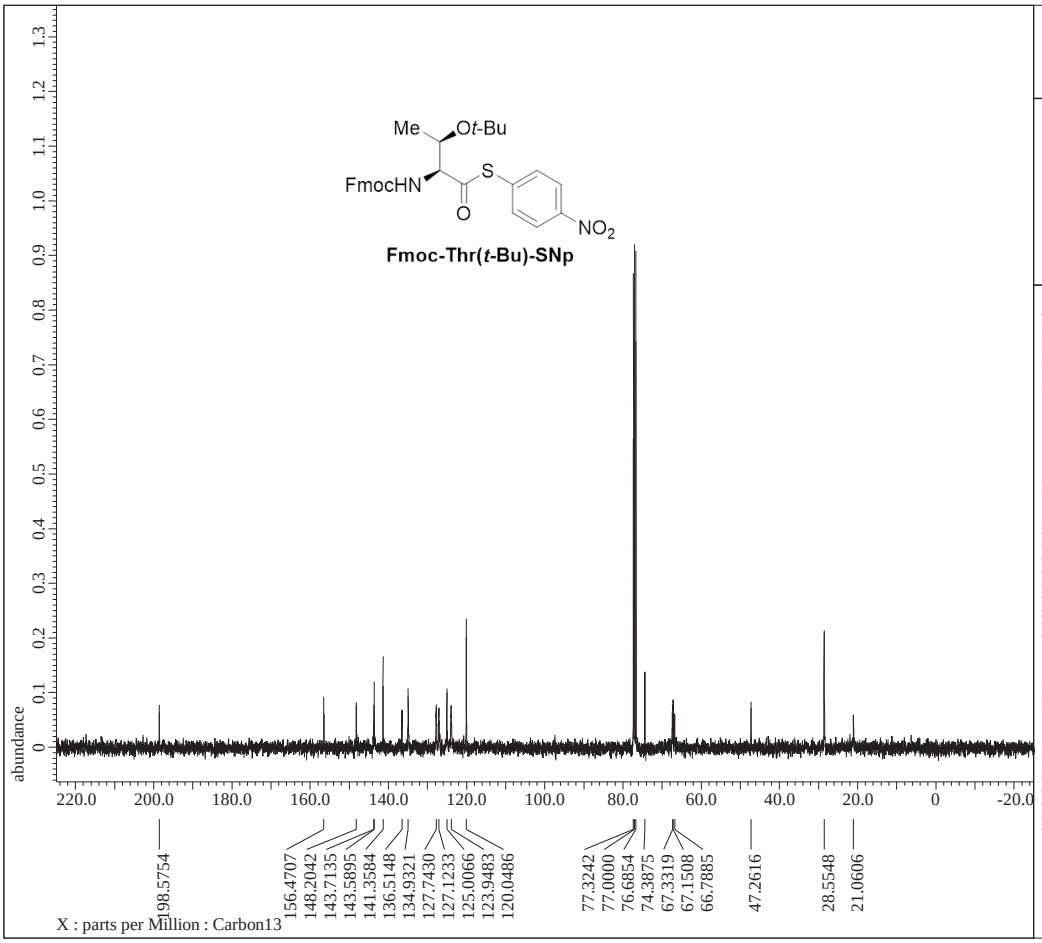

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-799_E13C-1-1.jdf
```

```
Filename          = wuan-799_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-799
Solvent           = CHLOROFORM-D
Creation_Time     = 26-OCT-2021 17:16:27
Revision_Time     = 26-OCT-2021 17:31:27
Current_Time      = 26-OCT-2021 17:32:21

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             = 322
Total_Scans       = 322

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]
```



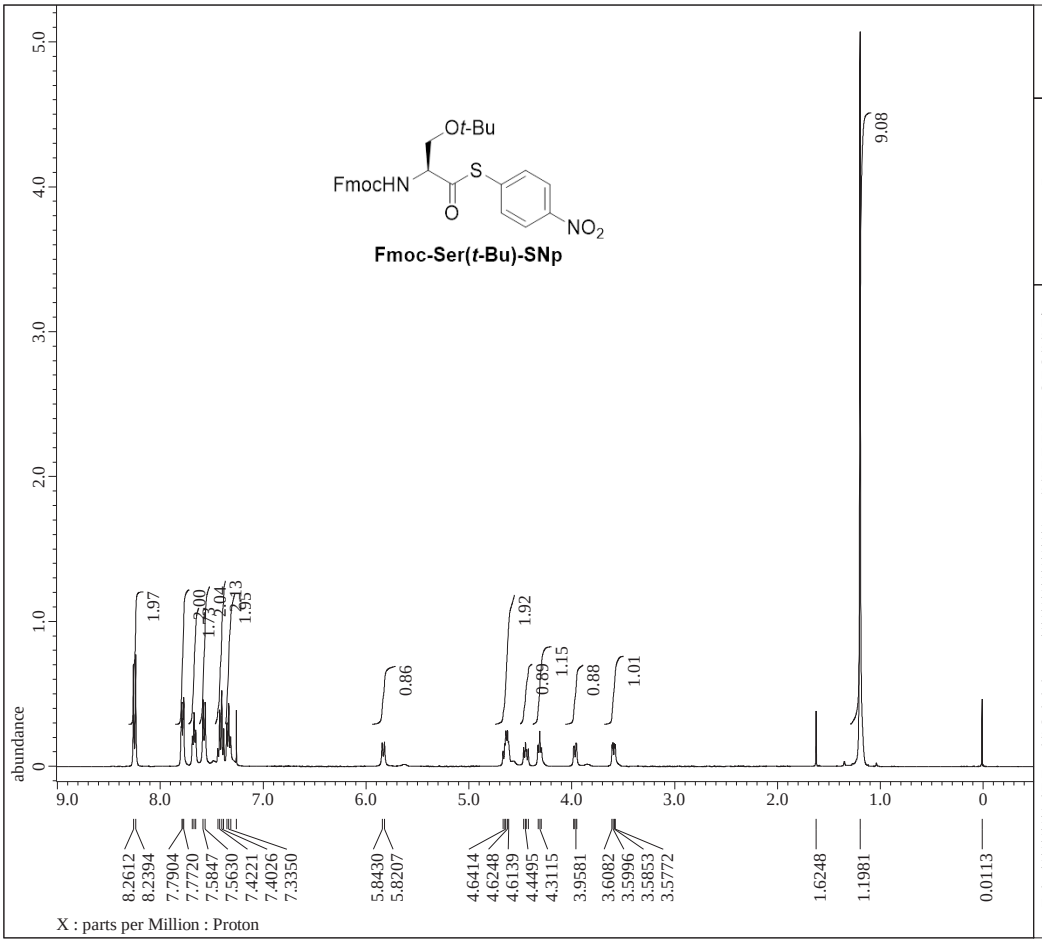

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-647_E1H-1-1.jdf
```

```
Filename          = wuan-647_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-647
Solvent           = CHLOROFORM-D
Creation_Time     = 10-SEP-2020 12:58:09
Revision_Time     = 26-OCT-2021 15:38:28
Current_Time      = 26-OCT-2021 15:38:56

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        = 50
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
```




```
---- 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: wuan-647_E13C-1-1.jdf
```

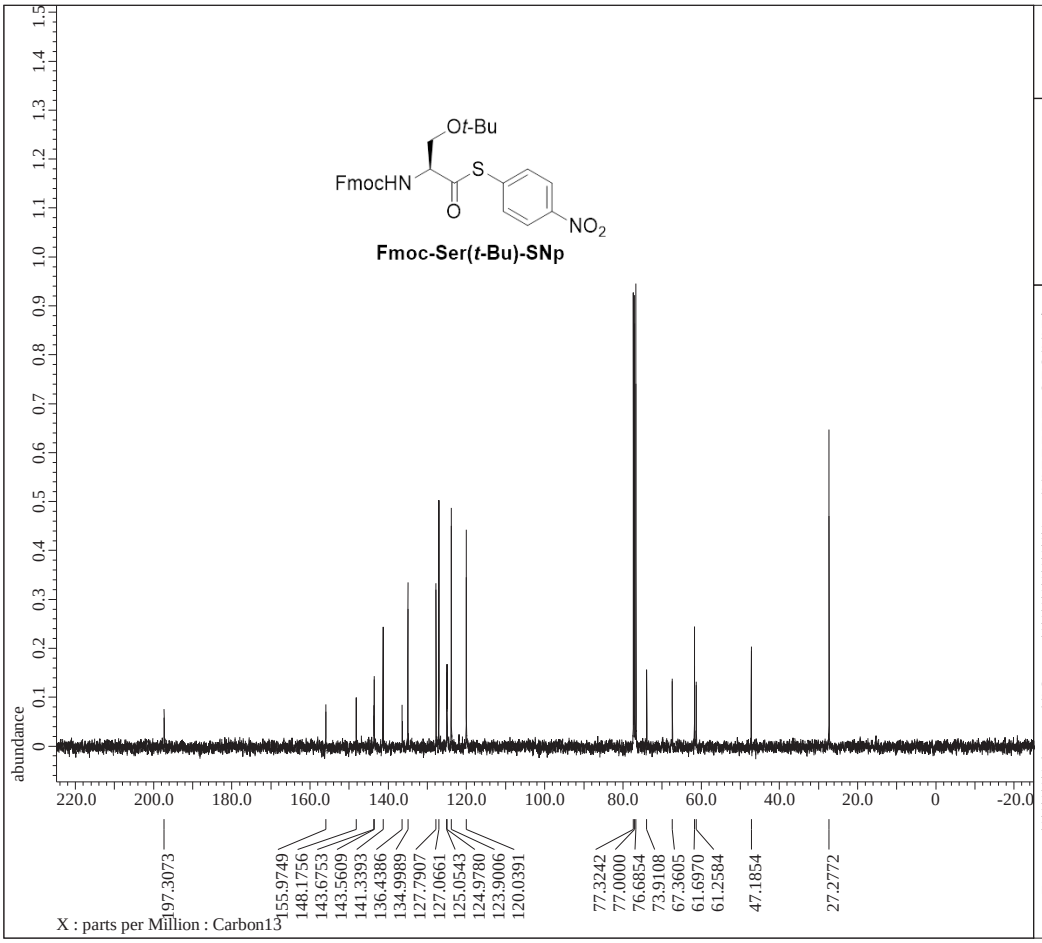
```
Filename          = wuan-647_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-647
Solvent           = CHLOROFORM-D
Creation_Time     = 26-OCT-2021 15:48:21
Revision_Time     = 26-OCT-2021 15:50:09
Current_Time      = 26-OCT-2021 15:50:49

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             = 103
Total_Scans       = 103

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]
```



JEOL RESONANCE

```
---- 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: wuan-664_E1H-1-1.jdf
```

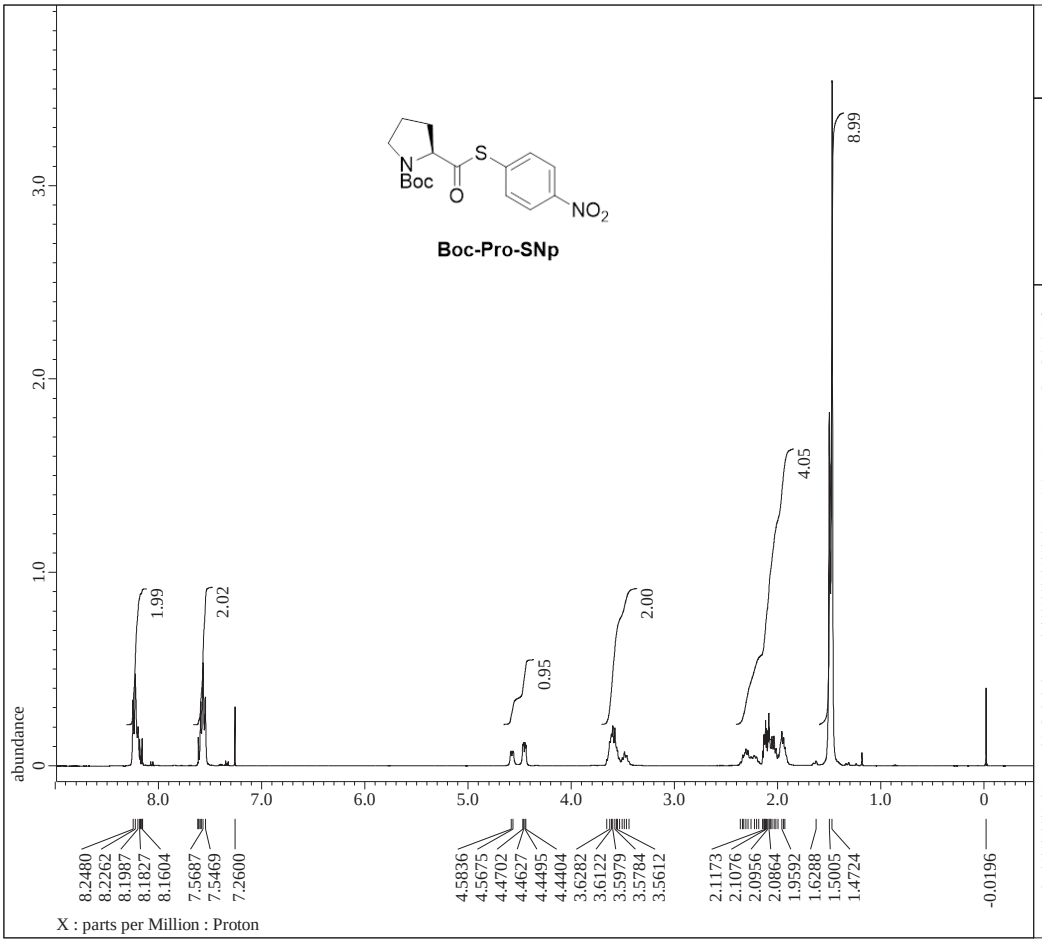
```
Filename          = wuan-664_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-664
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 19:00:24
Revision_Time     = 10-DEC-2021 19:03:38
Current_Time      = 10-DEC-2021 19:04:26

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
```



JEOL RESONANCE

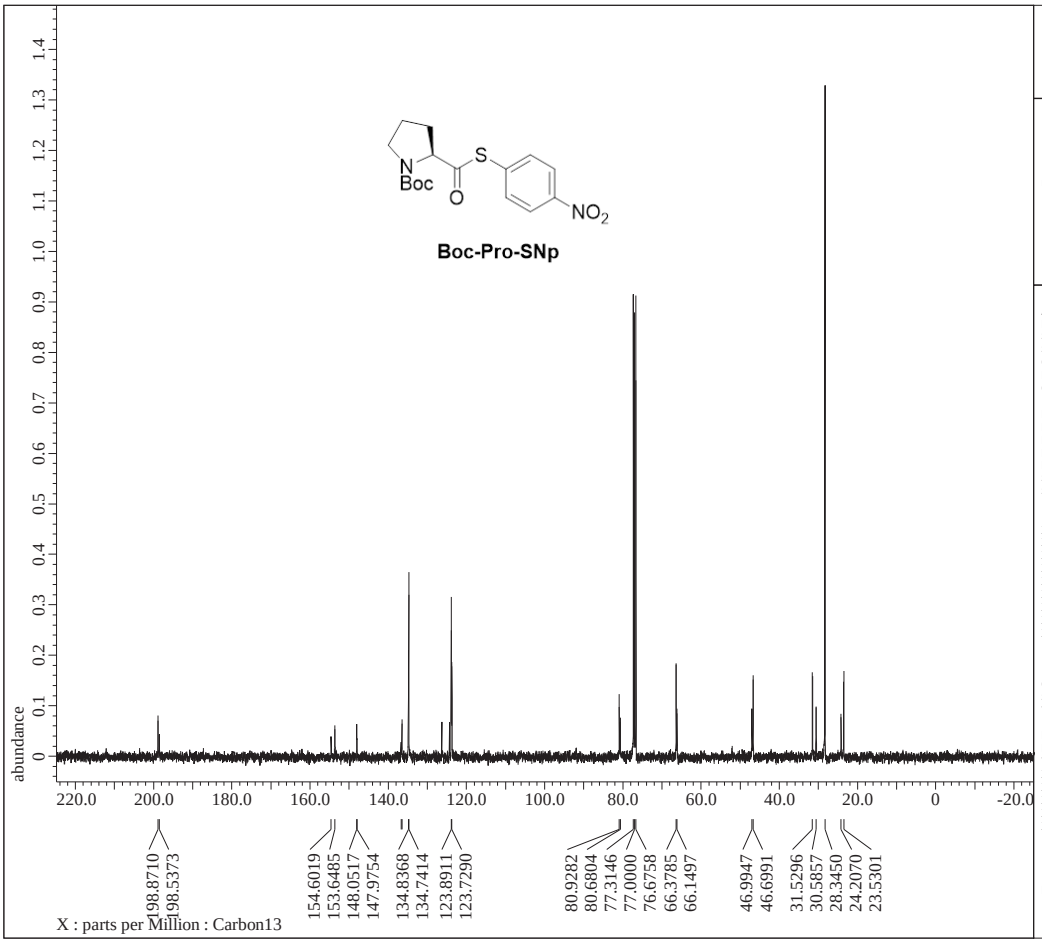
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-664_E13C-1-1.jdf
```

```
Filename          = wuan-664_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-664
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 19:05:06
Revision_Time     = 10-DEC-2021 19:06:26
Current_Time      = 10-DEC-2021 19:06:46

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             = 109
Total_Scans       = 109

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]
```



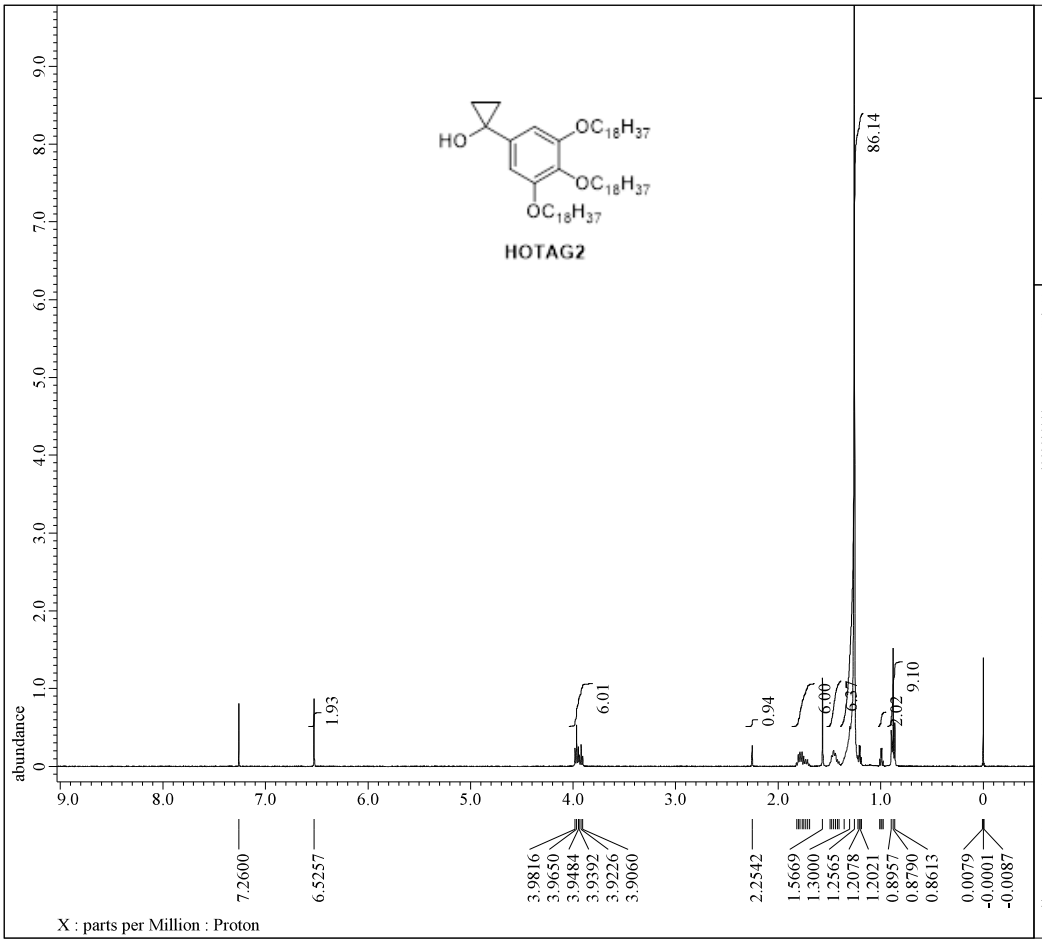

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-677_E1H-1-1.jdf
```

```
Filename          = wuan-677_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-677
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 18:04:52
Revision_Time     = 10-DEC-2021 18:06:38
Current_Time      = 10-DEC-2021 18:07:33

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
```




```
---- 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: wuan-677_E13C-1-1.jdf
```

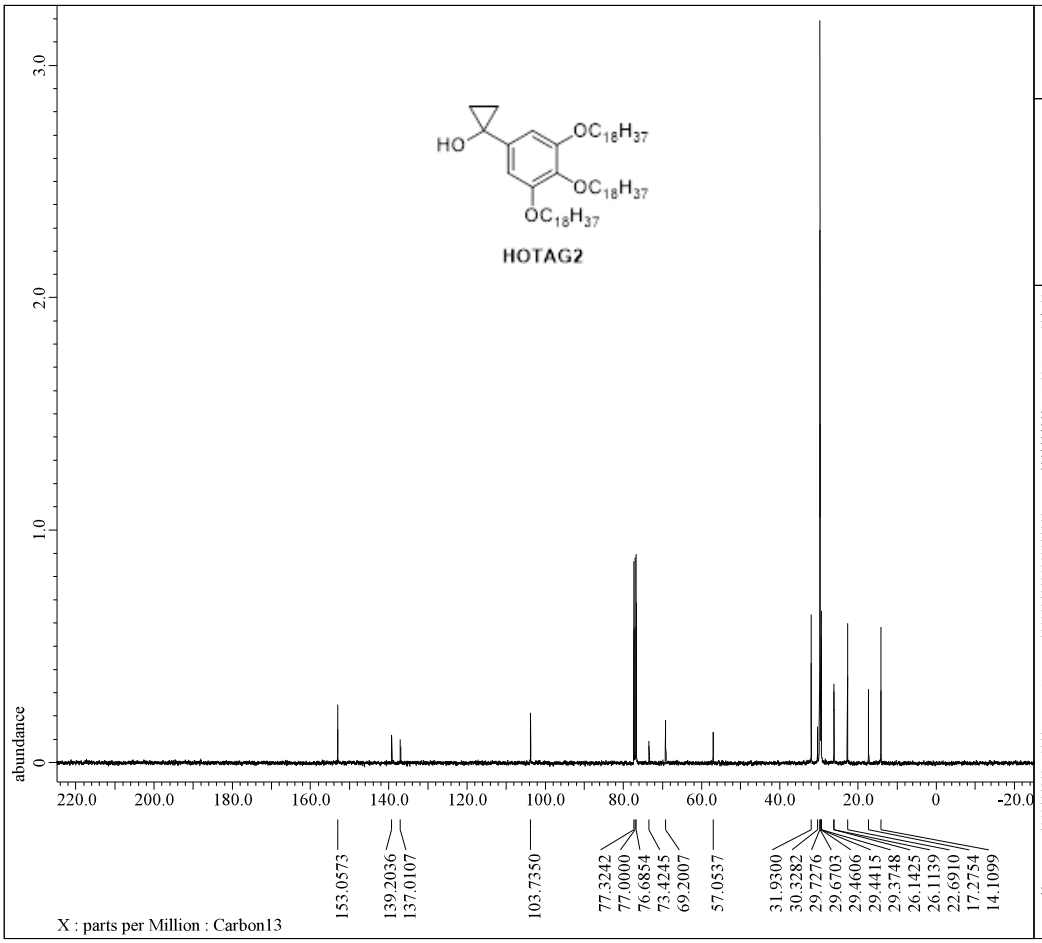
```
Filename          = wuan-677_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-677
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 18:09:34
Revision_Time     = 10-DEC-2021 18:17:01
Current_Time      = 10-DEC-2021 18:17:28

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             = 188
Total_Scans       = 188

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]
```



JEOL RESONANCE

```
---- 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: wuan-144-1_E1H-1-1.jdf
```

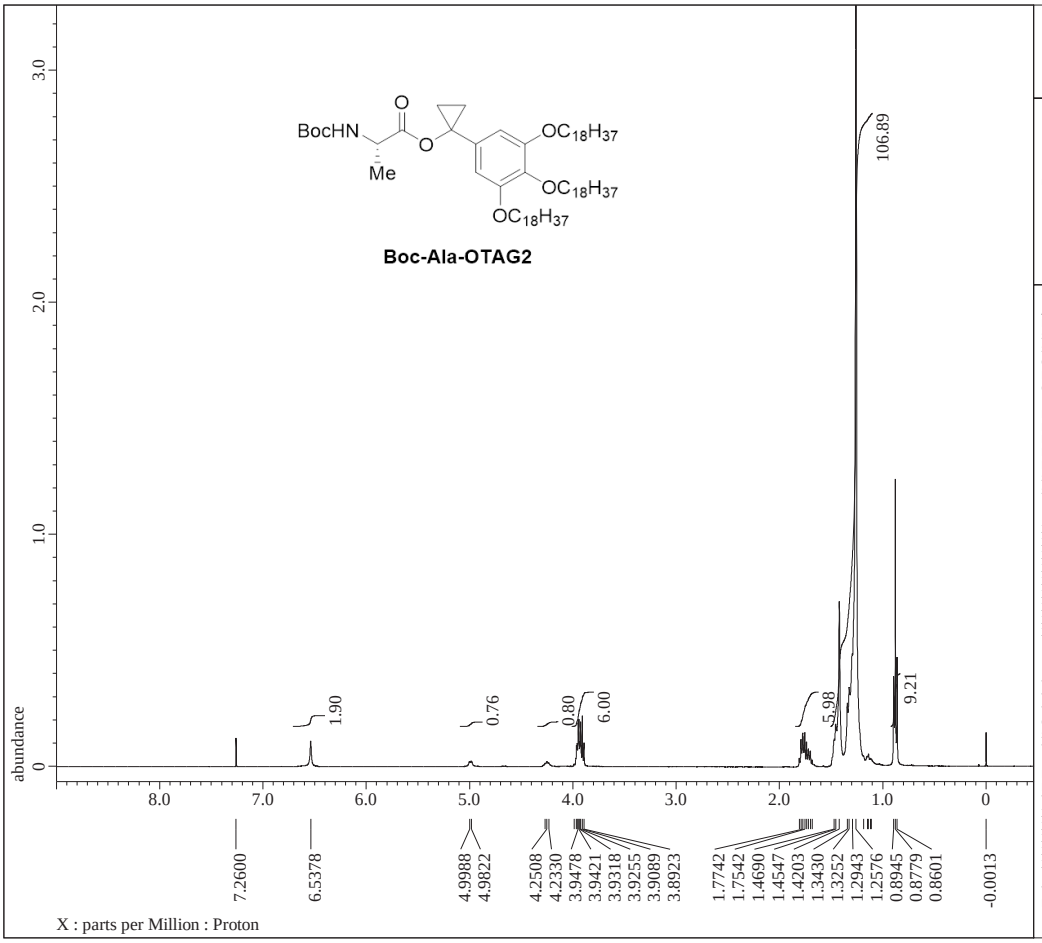
```
Filename          = wuan-144-1_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-144-1
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JUL-2019 20:34:37
Revision_Time     = 26-OCT-2021 17:23:14
Current_Time      = 26-OCT-2021 17:24:00

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        = 60
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
```



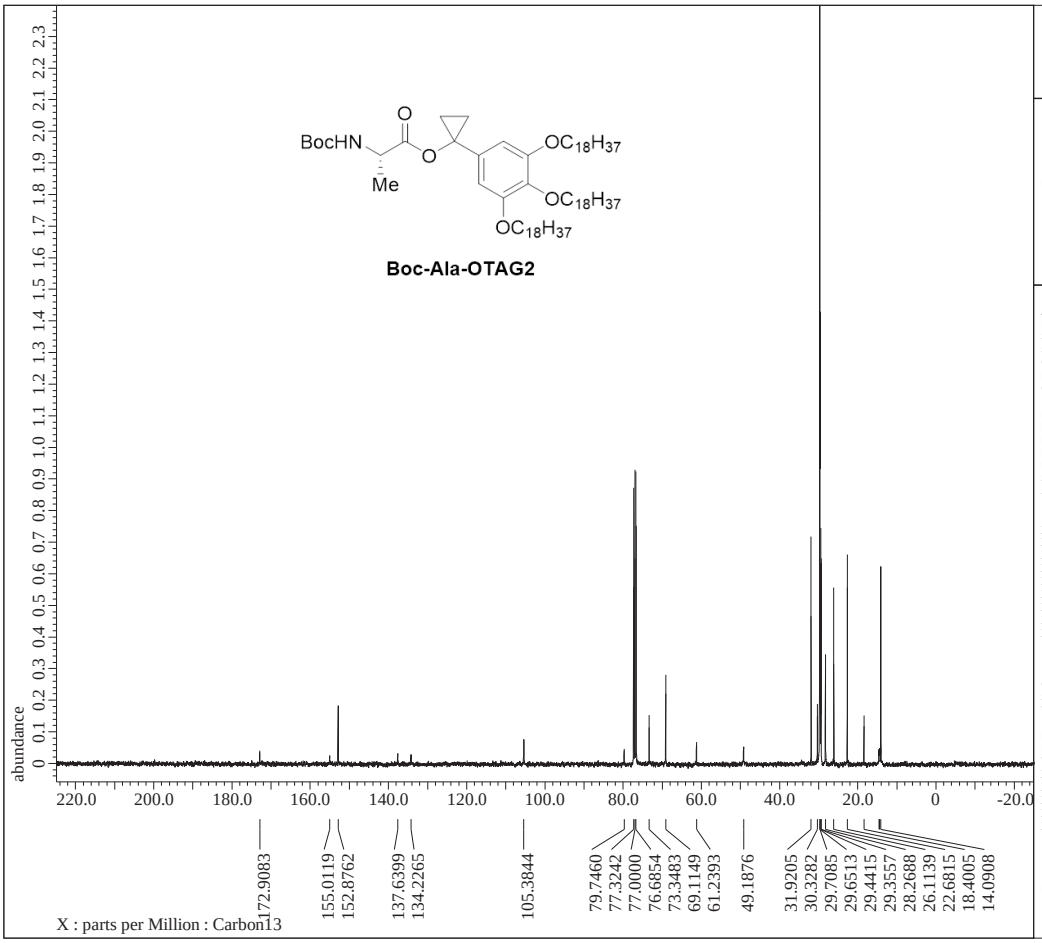
JEOL RESONANCE

```
---- 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: wuan-144-1_E13C-1-1.jdf
```

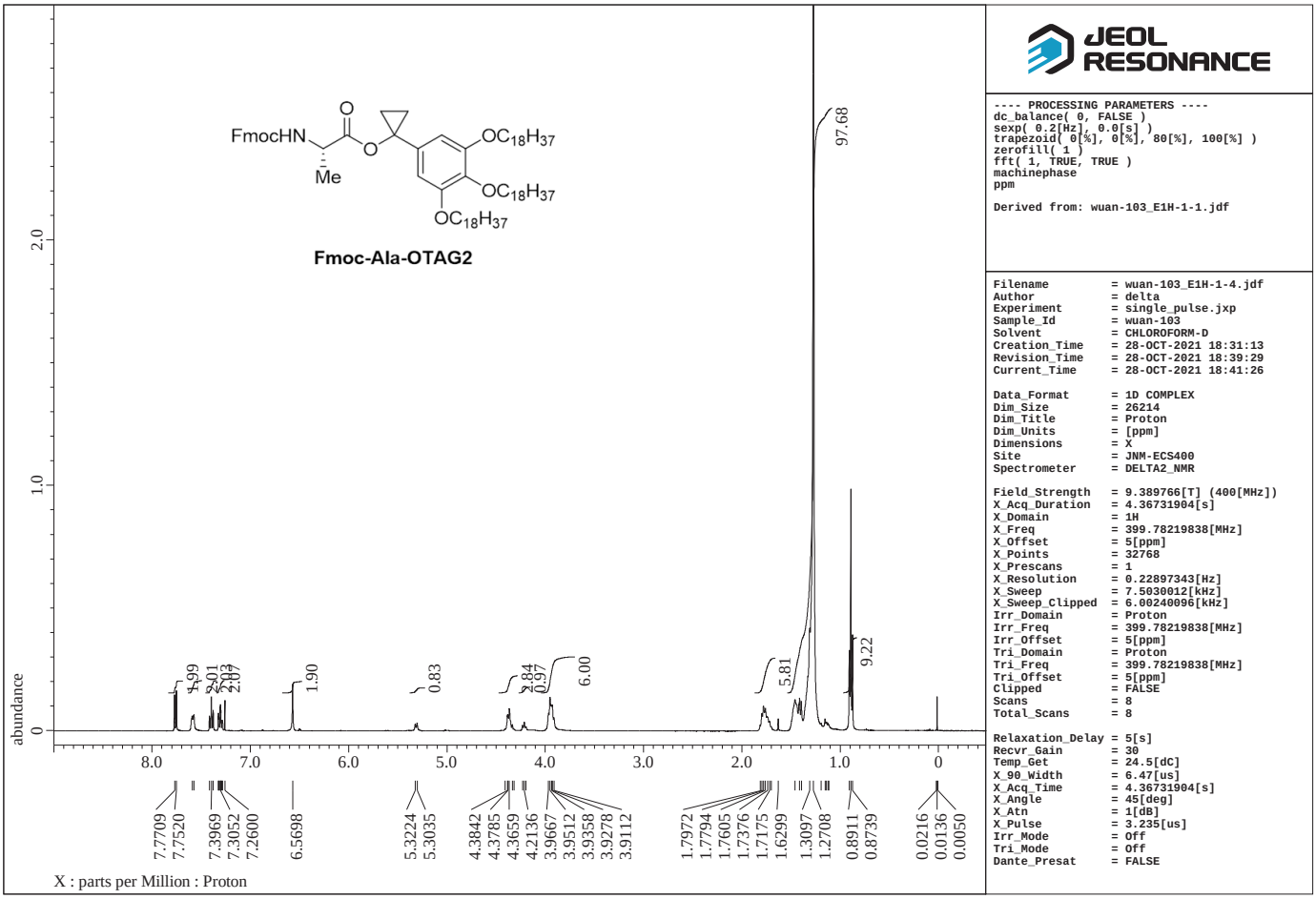
```
Filename          = wuan-144-1_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-144-1
Solvent           = CHLOROFORM-D
Creation_Time     = 26-OCT-2021 17:41:47
Revision_Time     = 26-OCT-2021 17:51:00
Current_Time      = 26-OCT-2021 17:51:16

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             = 231
Total_Scans       = 231

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: wuan-135_E1H-1-1.jdf

Filename          = wuan-135_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-135
Solvent           = CHLOROFORM-D
Creation_Time     = 28-OCT-2021 18:54:24
Revision_Time     = 28-OCT-2021 19:30:26
Current_Time      = 28-OCT-2021 19:31:18

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          = 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
```

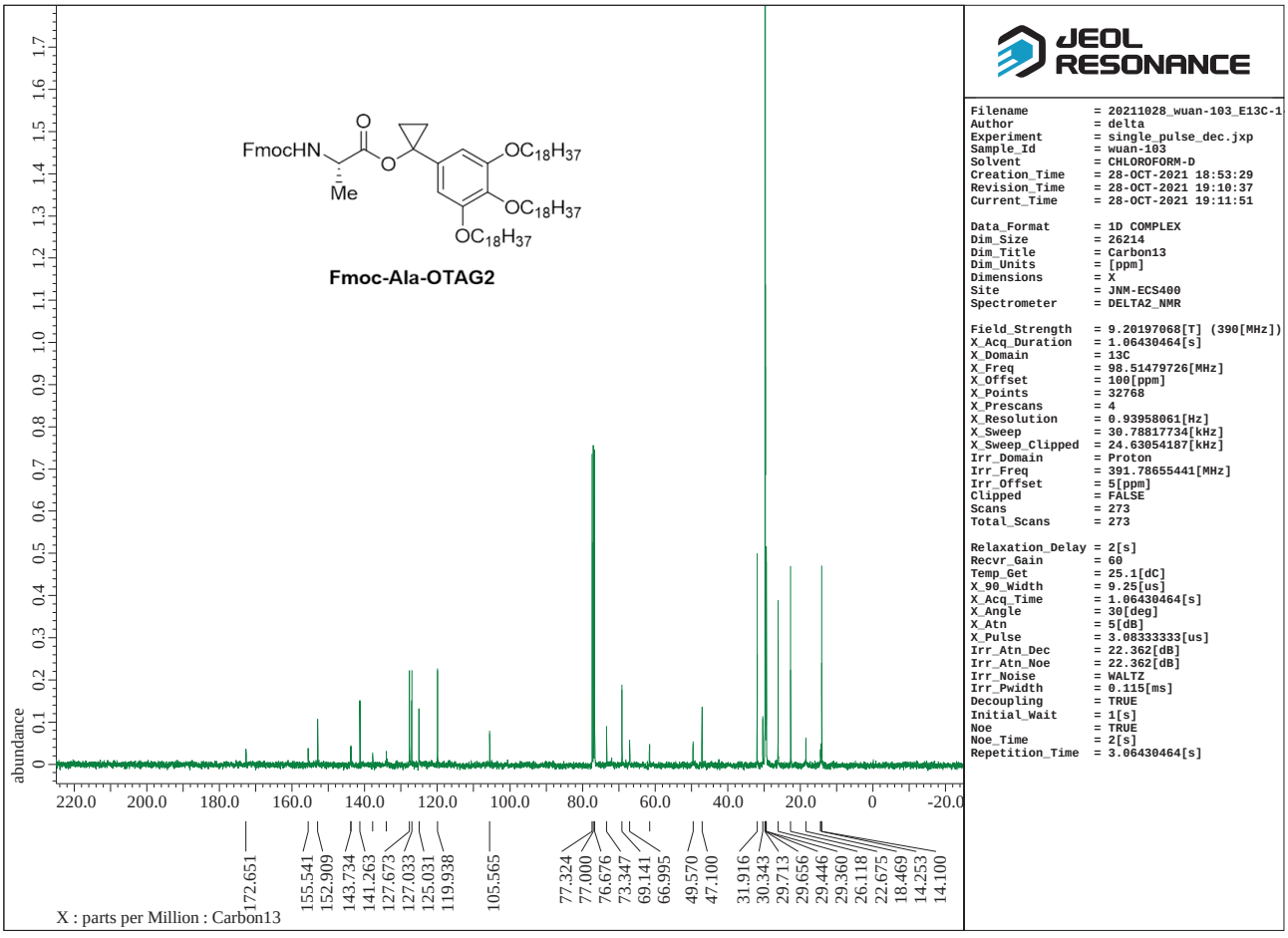



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-135_E13C-1-1.jdf

Filename          = wuan-135_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-135
Solvent           = CHLOROFORM-D
Creation_Time     = 28-OCT-2021 19:21:32
Revision_Time     = 28-OCT-2021 19:34:58
Current_Time      = 28-OCT-2021 19:36:04

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           = FALSE
Scans             = 347
Total_Scans       = 347

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.5[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]
```

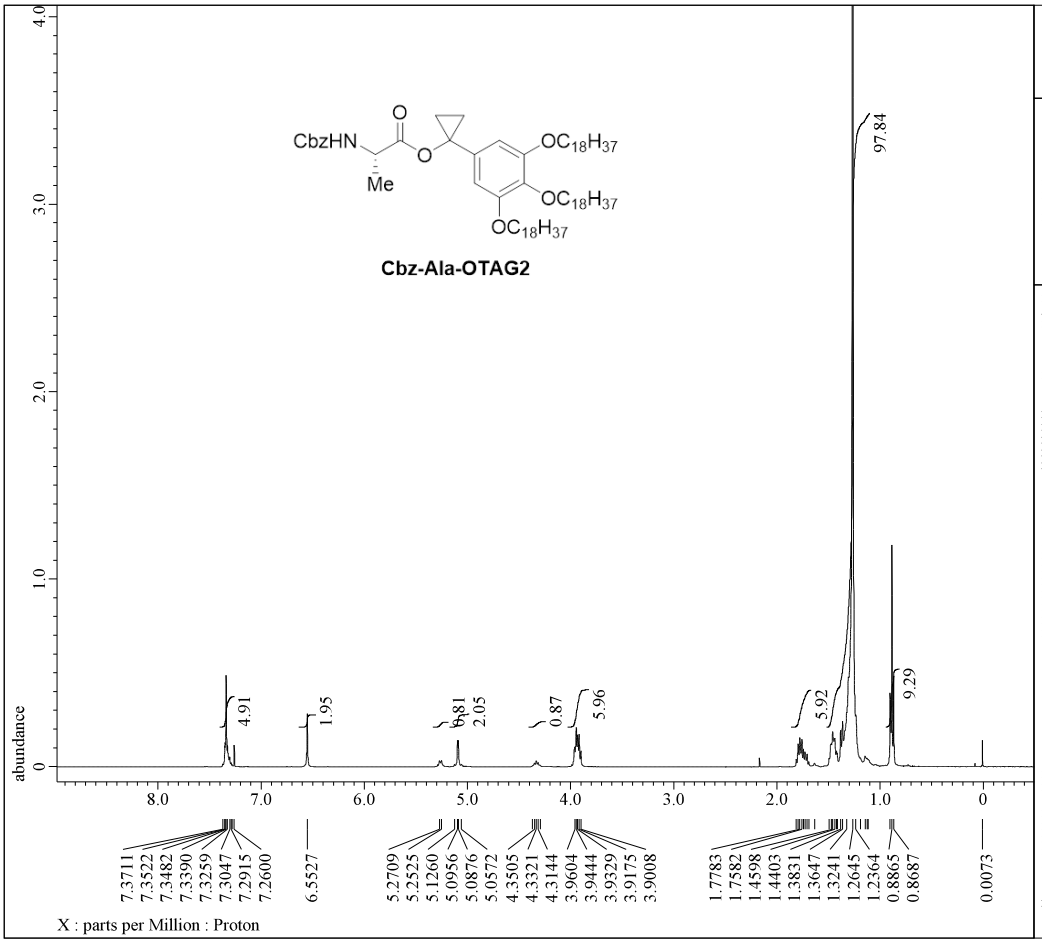



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-103_E1H-1-1.jdf
```

```
Filename          = wuan-103_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-103
Solvent           = CHLOROFORM-D
Creation_Time     = 28-OCT-2021 18:31:13
Revision_Time     = 28-OCT-2021 18:39:29
Current_Time      = 28-OCT-2021 18:41:26

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          = 24.5[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
```



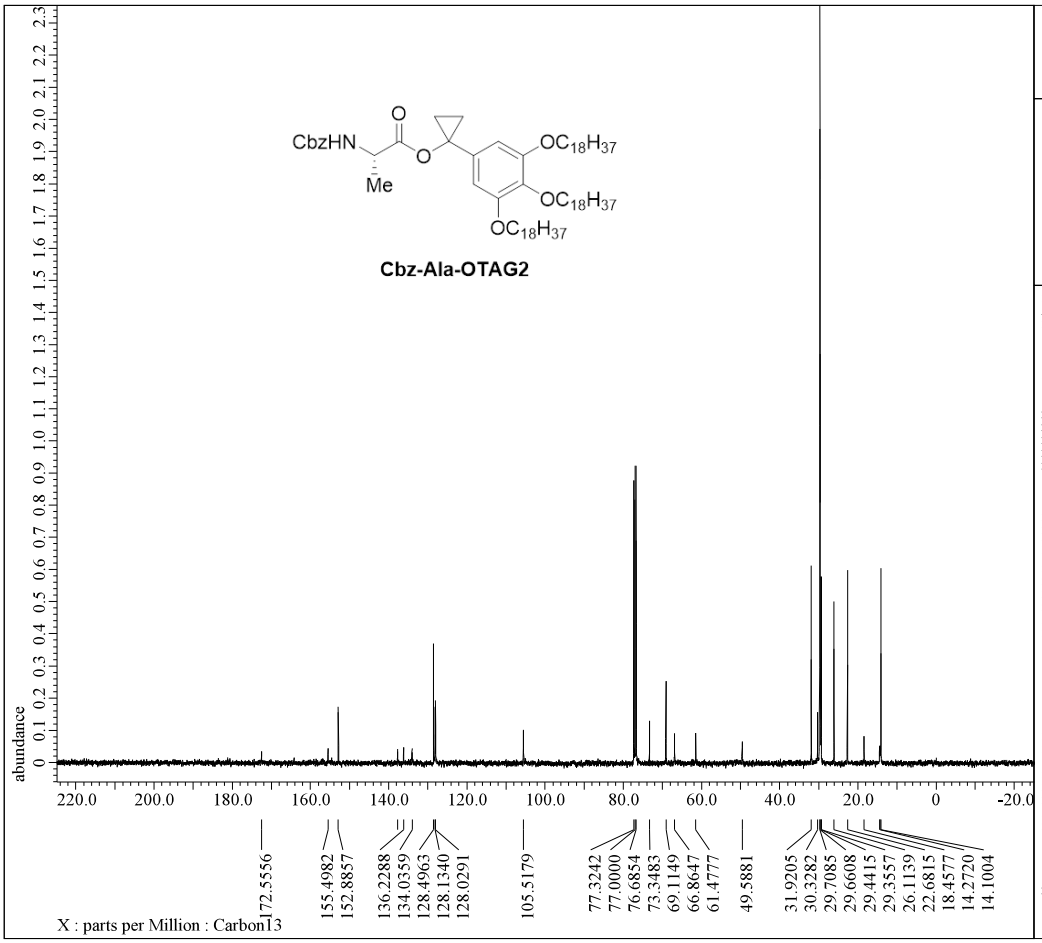

```
Filename          = 20211028_wuan-103_E13C-1
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-103
Solvent           = CHLOROFORM-D
Creation_Time     = 28-OCT-2021 18:53:29
Revision_Time     = 28-OCT-2021 19:10:37
Current_Time      = 28-OCT-2021 19:11:51

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 273
Total_Scans       = 273

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25.1[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.06430464[s]
```



JEOL RESONANCE

```
---- 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: wuan-1210_E1H-1-1.jdf
```

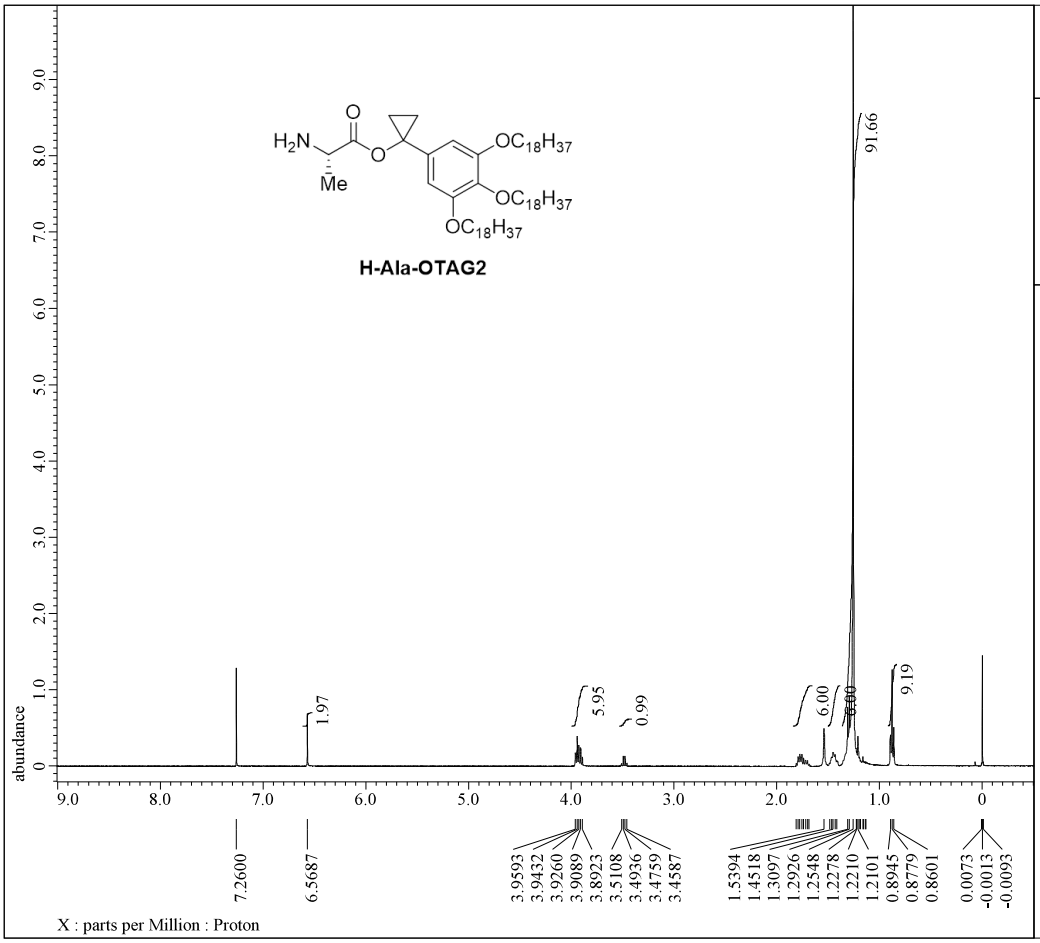
```
Filename          = wuan-1210_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1210
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 12:46:03
Revision_Time     = 12-NOV-2021 12:55:16
Current_Time      = 12-NOV-2021 12:56:16

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        = 18
Temp_Get          = 24.9[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
```



JEOL RESONANCE

```
---- 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: wuan-1210_E13C-1-1.jdf
```

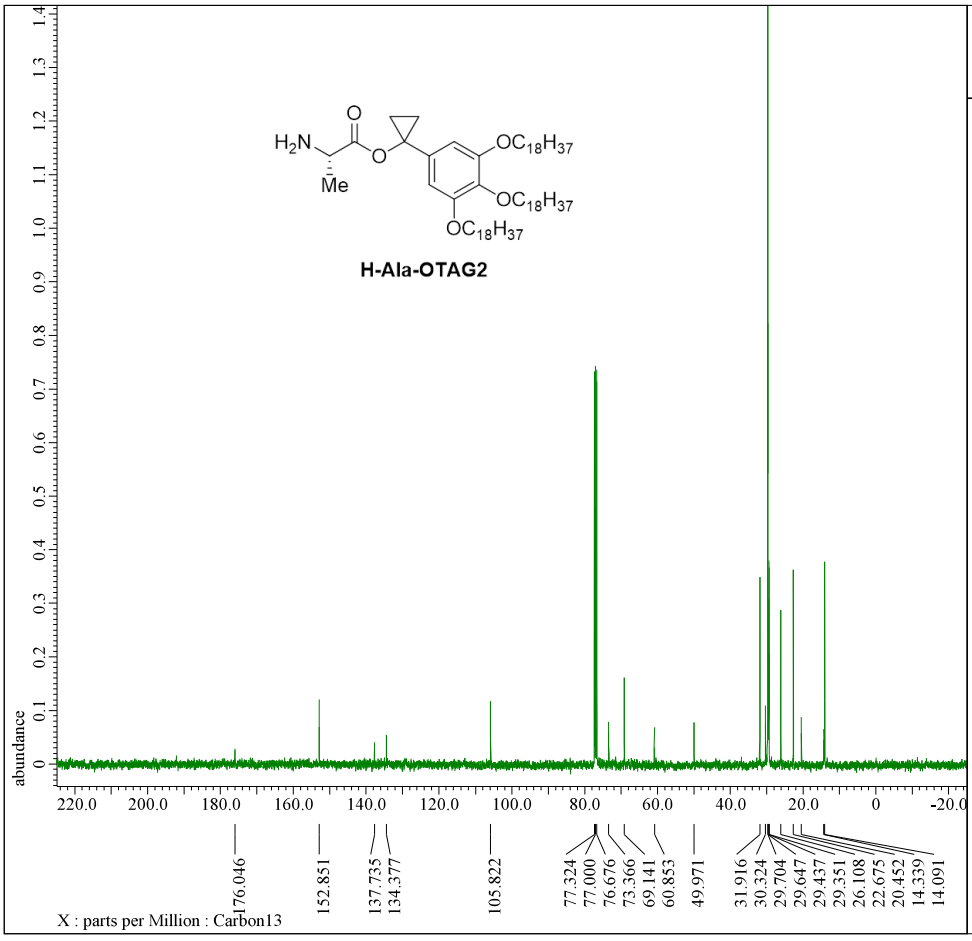
```
Filename          = wuan-1210_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1210
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 12:50:47
Revision_Time     = 12-NOV-2021 13:00:31
Current_Time      = 12-NOV-2021 13:01:04

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           = FALSE
Scans             = 246
Total_Scans       = 246

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]
```



JEOL RESONANCE

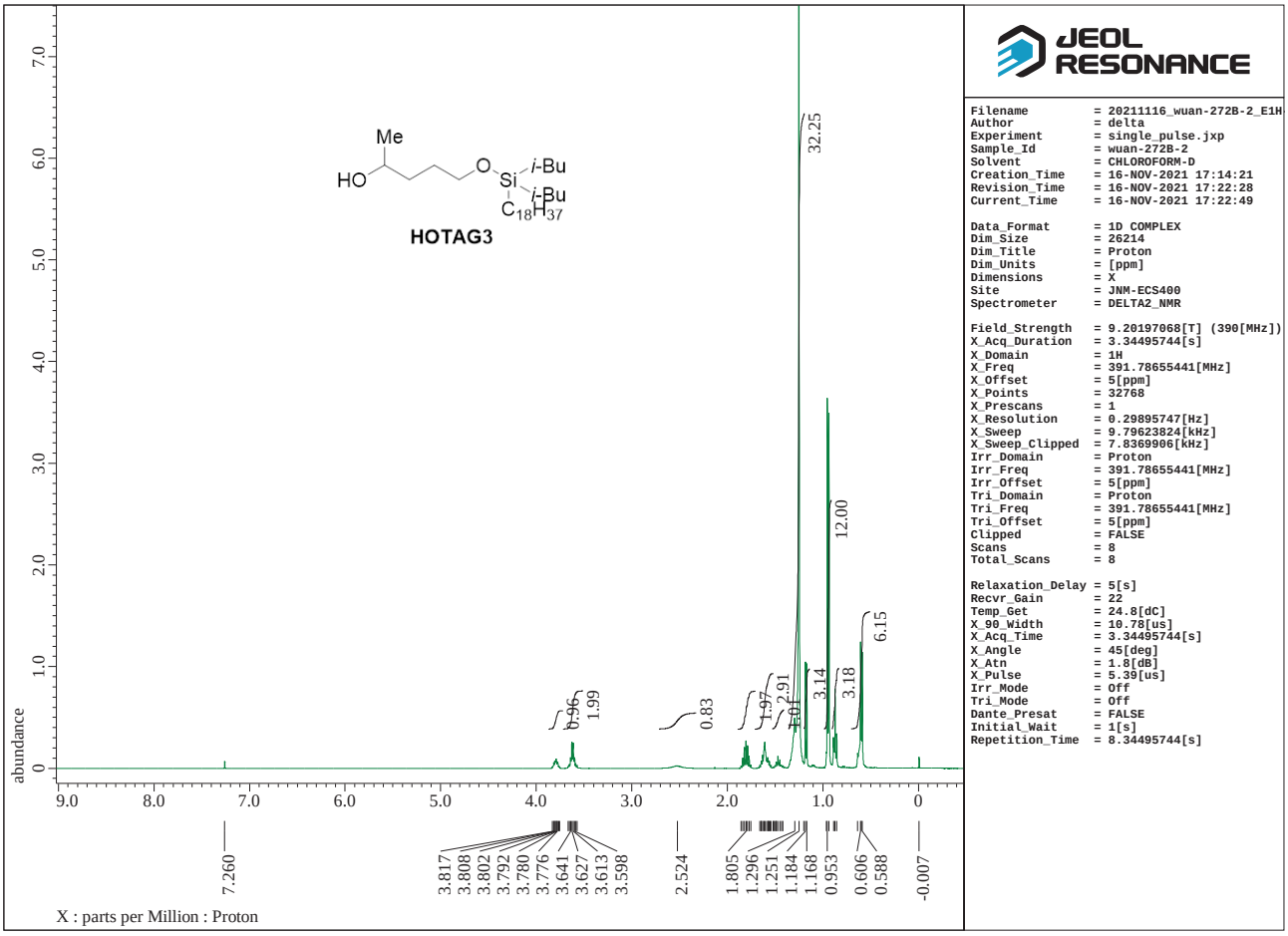
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= wuan-107_E1H-1-7.jdf
Author	= delta
Experiment	= single_pulse.jxp
Sample_Id	= wuan-107
Solvent	= CHLOROFORM-D
Creation_Time	= 7-JUN-2019 19:12:26
Revision_Time	= 12-NOV-2021 13:04:20
Current_Time	= 12-NOV-2021 13:05:16
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	= 60
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



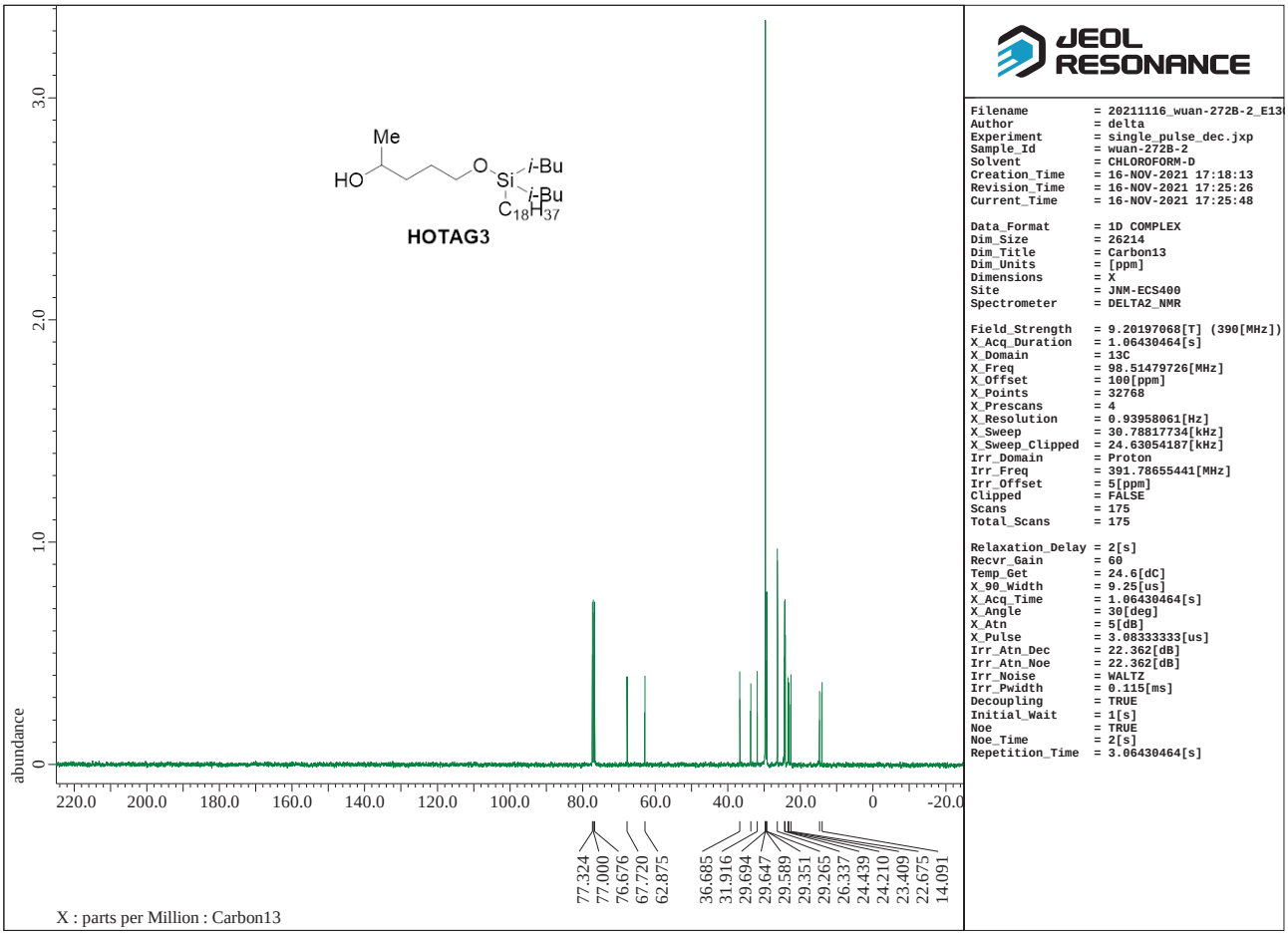
JEOL RESONANCE

Filename	= 20211030_wuan-115_E13C-1
Author	= delta
Experiment	= single_pulse_dec.jxp
Sample_Id	= wuan-115
Solvent	= CHLOROFORM-D
Creation_Time	= 30-OCT-2021 10:55:00
Revision_Time	= 30-OCT-2021 11:06:28
Current_Time	= 30-OCT-2021 11:09:52
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
Irr_Domain	= Proton
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 251.0
Total_Scans	= 251.0
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 25.2[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.08333333[us]
Irr_Atn_Dec	= 22.362[dB]
Irr_Atn_Noe	= 22.362[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.06430464[s]



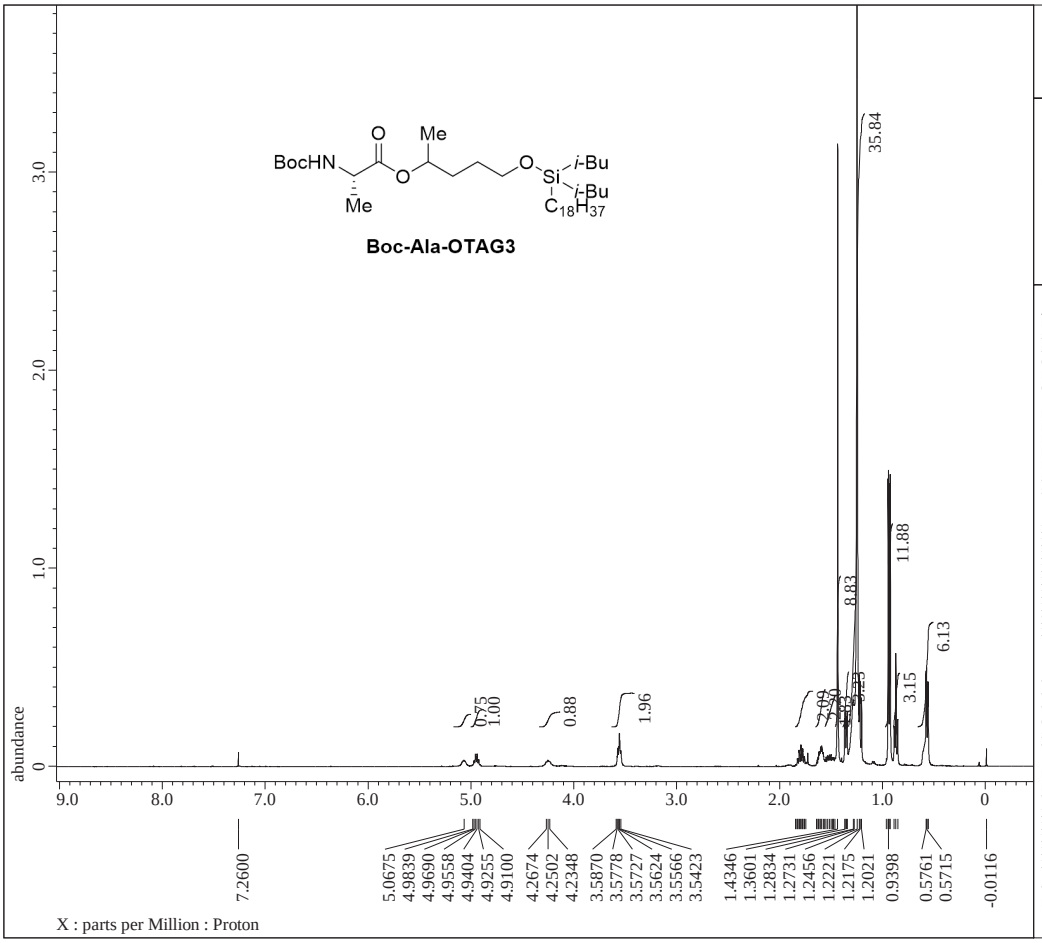
JEOL RESONANCE

```
Filename          = 20211116_wuan-272B-2_E1H
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-272B-2
Solvent           = CHLOROFORM-D
Creation_Time     = 16-NOV-2021 17:14:21
Revision_Time     = 16-NOV-2021 17:22:28
Current_Time      = 16-NOV-2021 17:22:49

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 3.34495744[s]
X_Domain          = 1H
X_Freq            = 391.78655441[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.29895747[Hz]
X_Sweep           = 9.79623824[kHz]
X_Sweep_Clipped   = 7.8369906[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 391.78655441[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 22
Temp_Get          = 24.8[dC]
X_90_Width        = 10.78[us]
X_Acq_Time        = 3.34495744[s]
X_Angle           = 45[deg]
X_Atn             = 1.8[dB]
X_Pulse           = 5.39[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 8.34495744[s]
```

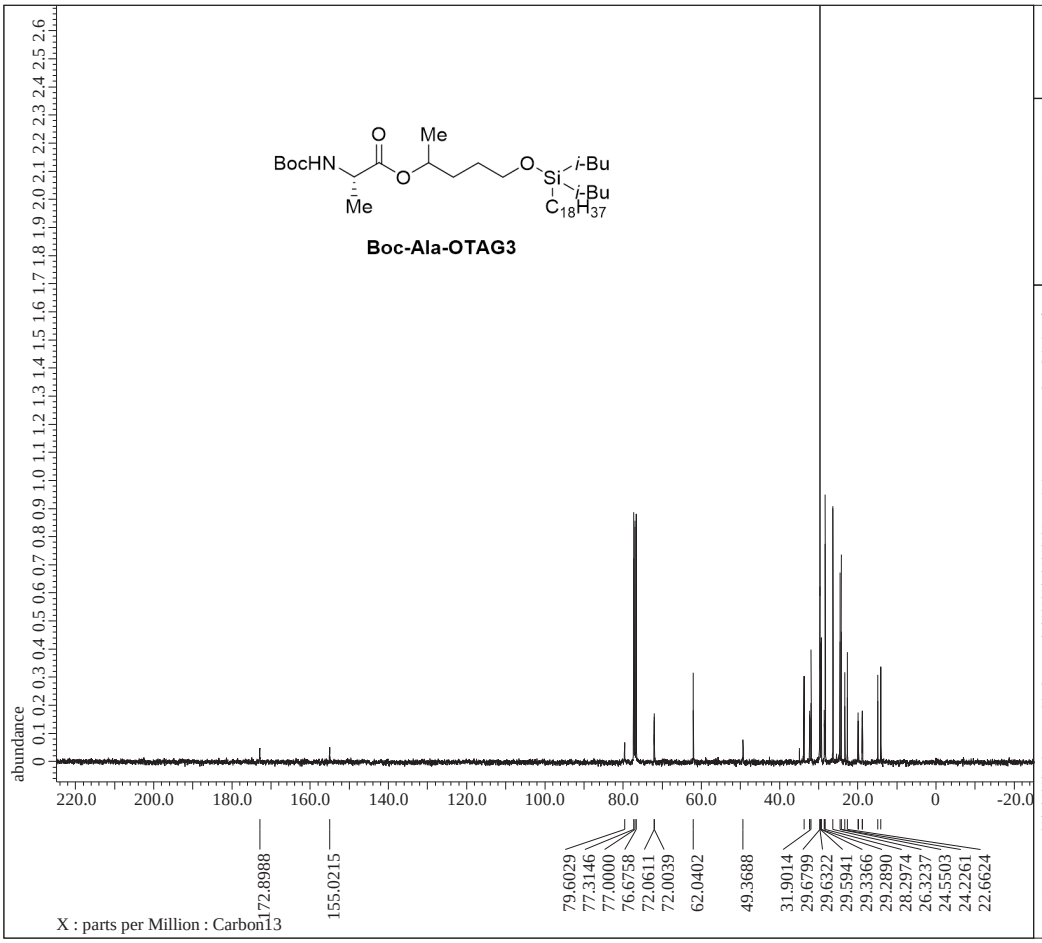


JEOL RESONANCE

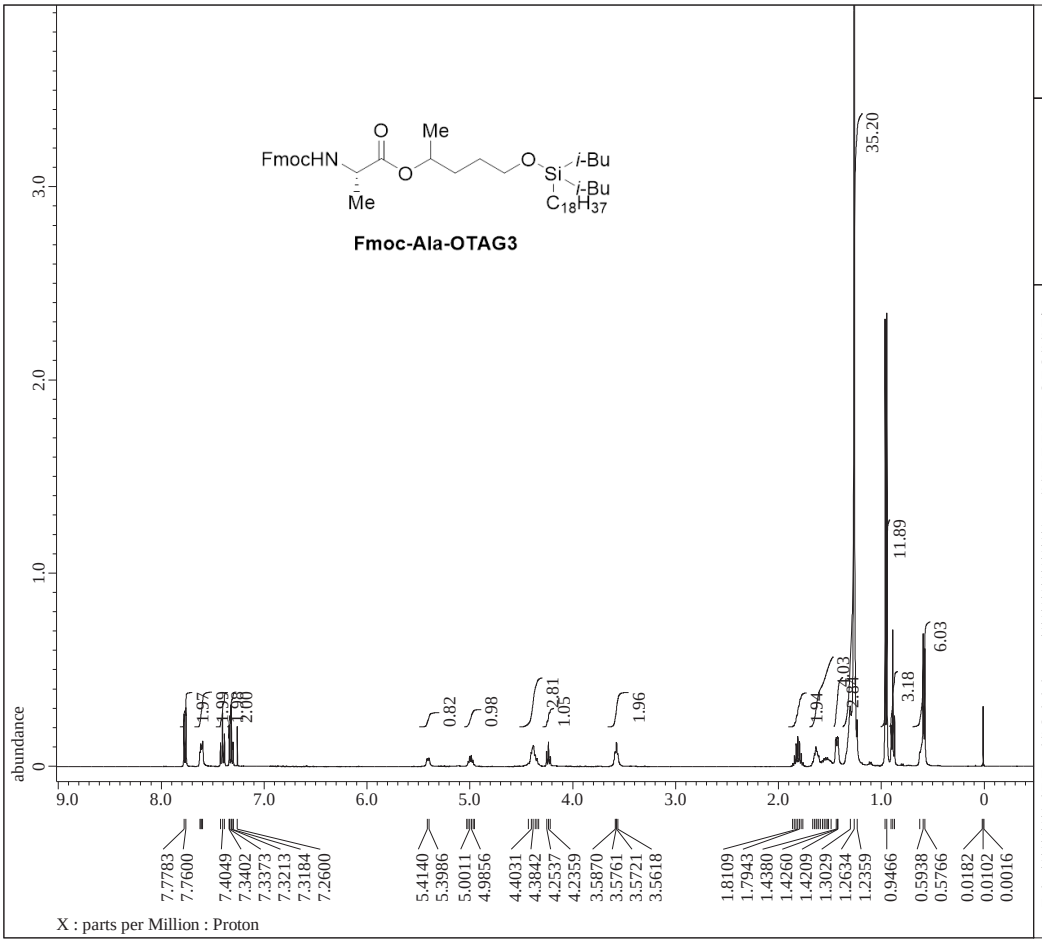
```
Filename          = 20211116_wuan-272B-2_E13
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-272B-2
Solvent           = CHLOROFORM-D
Creation_Time     = 16-NOV-2021 17:18:13
Revision_Time     = 16-NOV-2021 17:25:26
Current_Time      = 16-NOV-2021 17:25:48

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 175
Total_Scans       = 175

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.6[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.06430464[s]
```



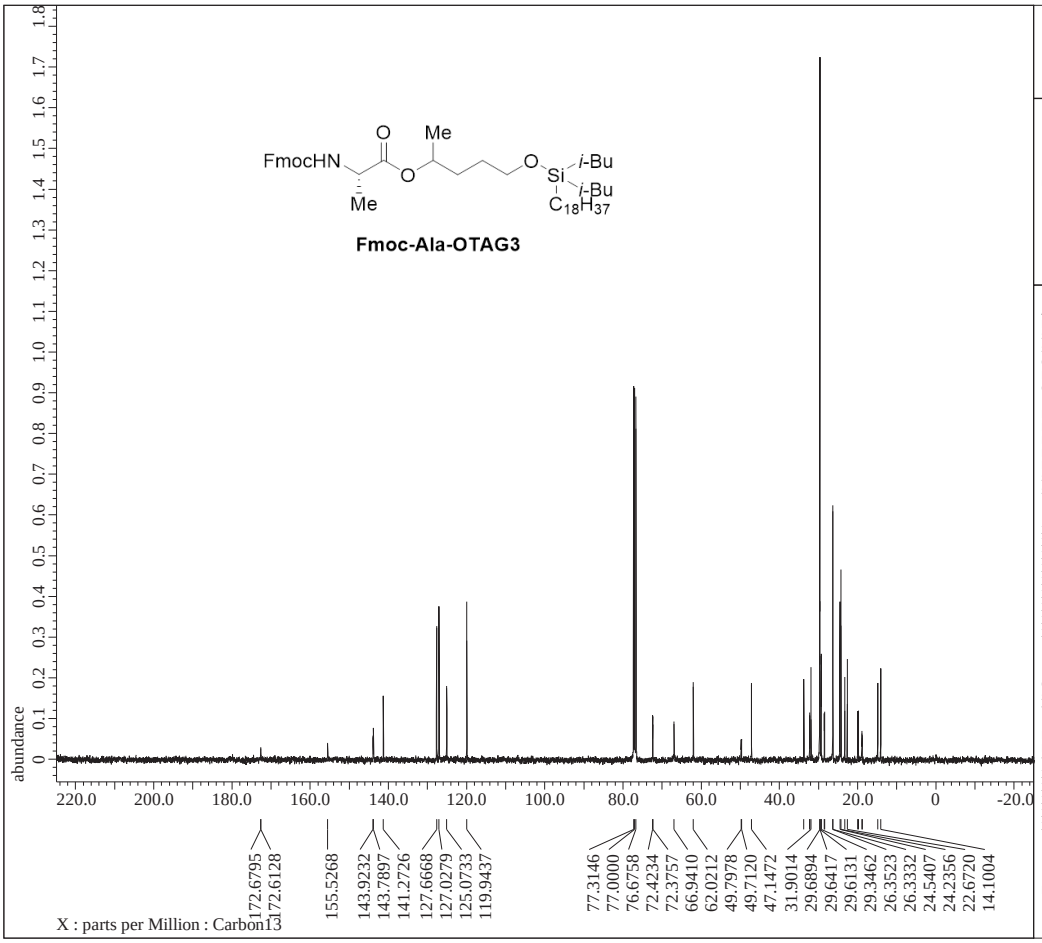

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-281_E1H-1-1.jdf

Filename          = wuan-281_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-281
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 15:11:47
Revision_Time     =  3-NOV-2021 15:17:20
Current_Time      =  3-NOV-2021 15:18:36

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        = 16
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
```



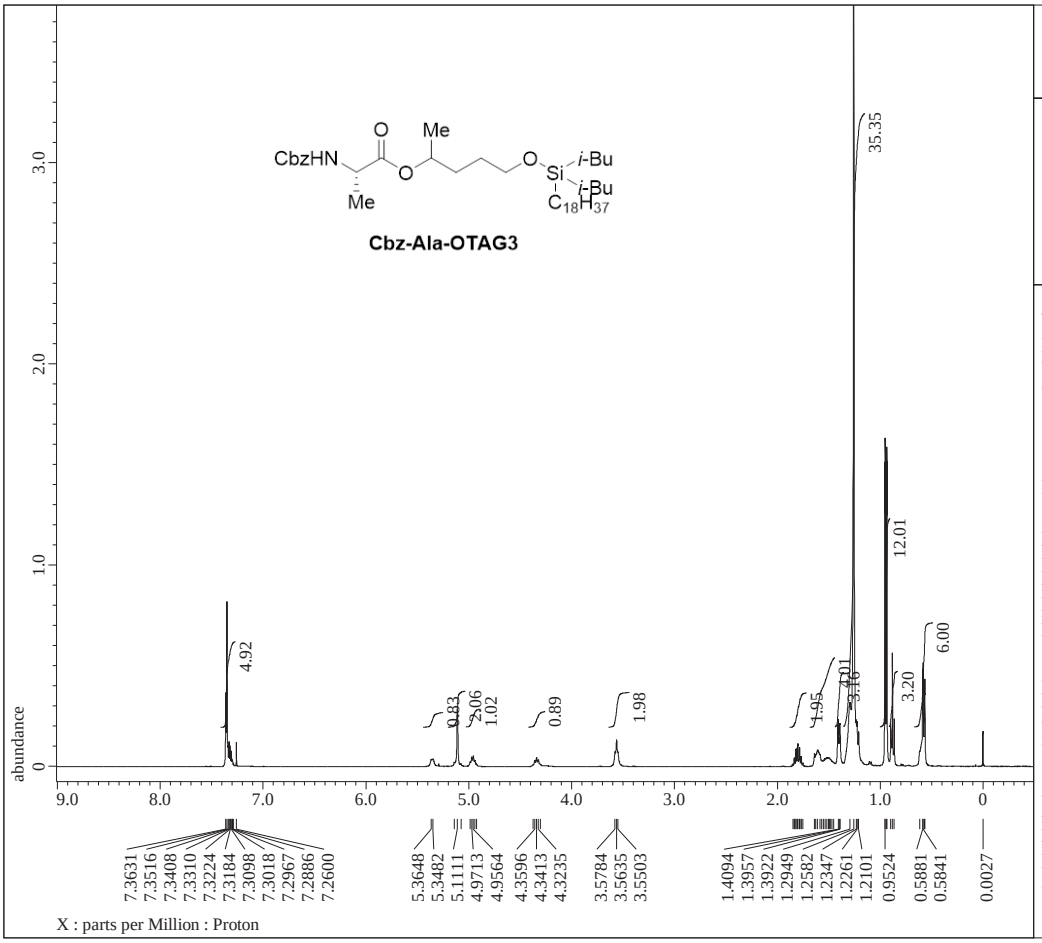

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-281_E13C-1-1.jdf

Filename          = wuan-281_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-281
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 15:15:43
Revision_Time     =  3-NOV-2021 15:30:57
Current_Time      =  3-NOV-2021 15:31:55

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             = 204
Total_Scans       = 204

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.3[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]
```



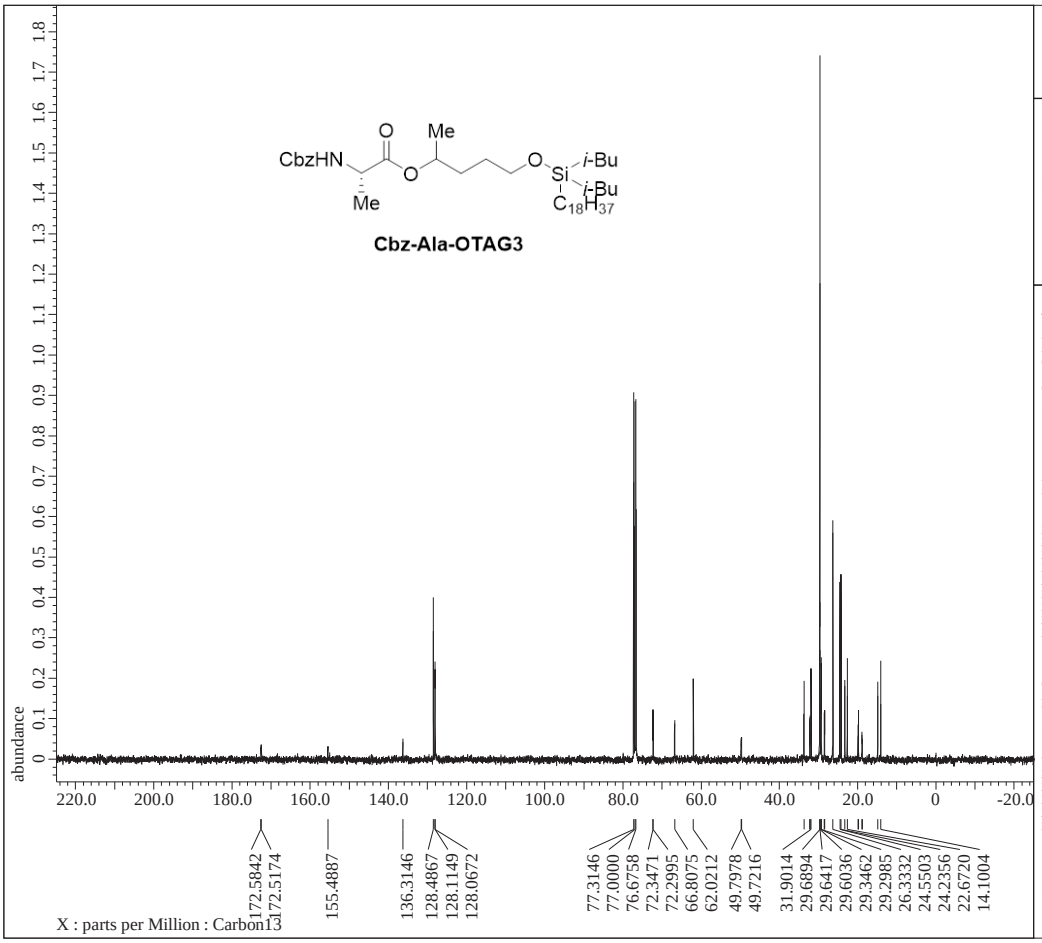

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-282B_E1H-1-1.jdf

Filename          = wuan-282B_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-282B
Solvent           = CHLOROFORM-D
Creation_Time     = 17-NOV-2021 13:43:38
Revision_Time     = 17-NOV-2021 13:45:28
Current_Time      = 17-NOV-2021 13:46:57

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        = 34
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
```



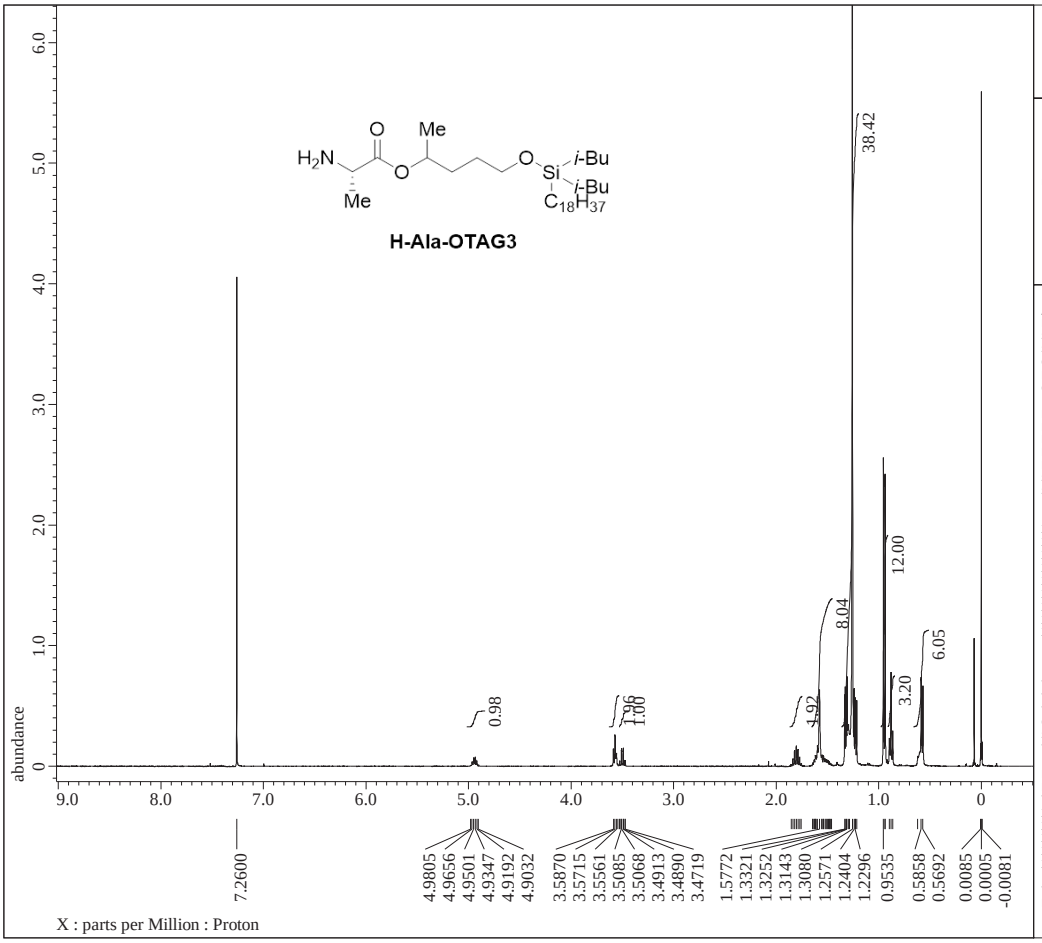

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-282B_E13C-1-1.jdf

Filename          = wuan-282B_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-282B
Solvent           = CHLOROFORM-D
Creation_Time     = 17-NOV-2021 13:47:16
Revision_Time     = 17-NOV-2021 13:59:58
Current_Time      = 17-NOV-2021 14:01:14

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             = 327
Total_Scans       = 327

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.3[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]
```




```
---- 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: wuan-273B_E1H-1-1.jdf

Filename          = wuan-273B_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-273B
Solvent           = CHLOROFORM-D
Creation_Time     = 17-NOV-2021 13:20:31
Revision_Time     = 17-NOV-2021 13:24:13
Current_Time      = 17-NOV-2021 13:25:07

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
```



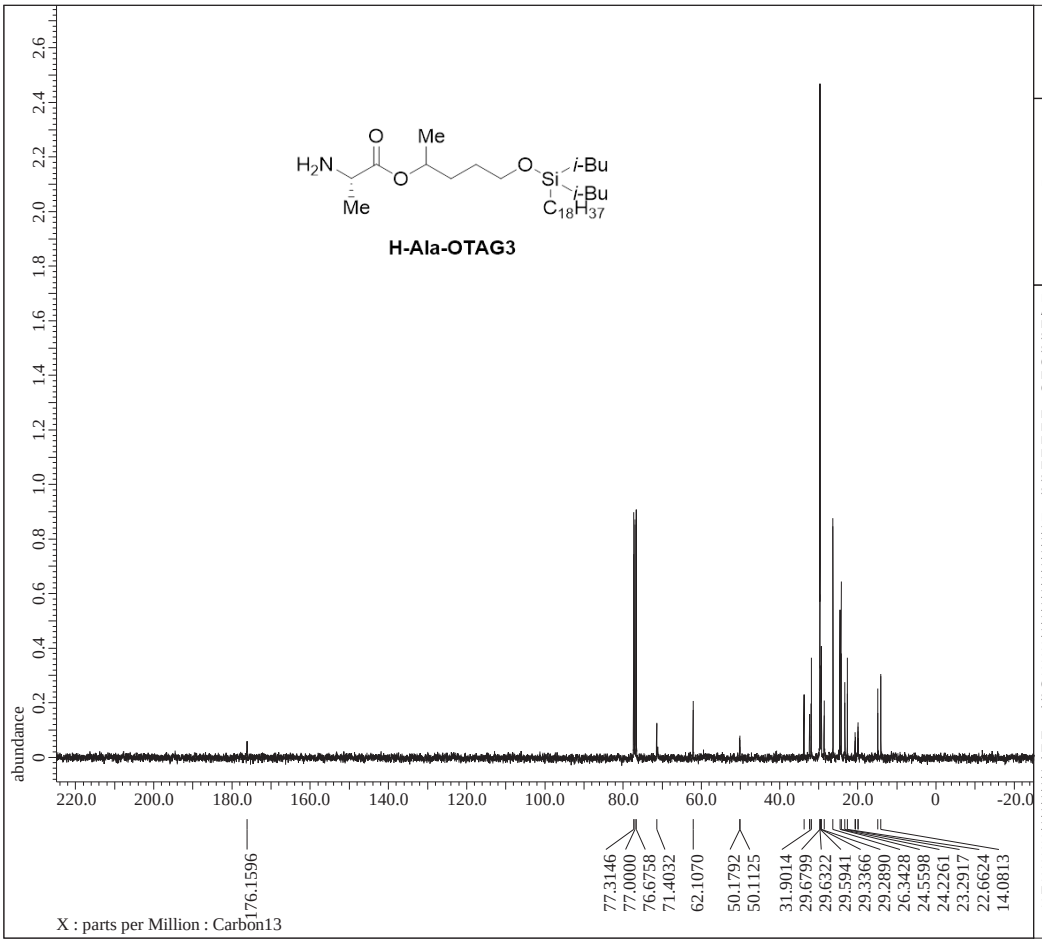

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-273B_E13C-1-1.jdf

Filename          = wuan-273B_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-273B
Solvent           = CHLOROFORM-D
Creation_Time     = 17-NOV-2021 13:24:27
Revision_Time     = 17-NOV-2021 13:34:43
Current_Time      = 17-NOV-2021 13:36:48

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             = 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
Initial_Wait      = 1[s]
```



JEOL RESONANCE

```
---- 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: wuan-275_E1H-1-1.jdf
```

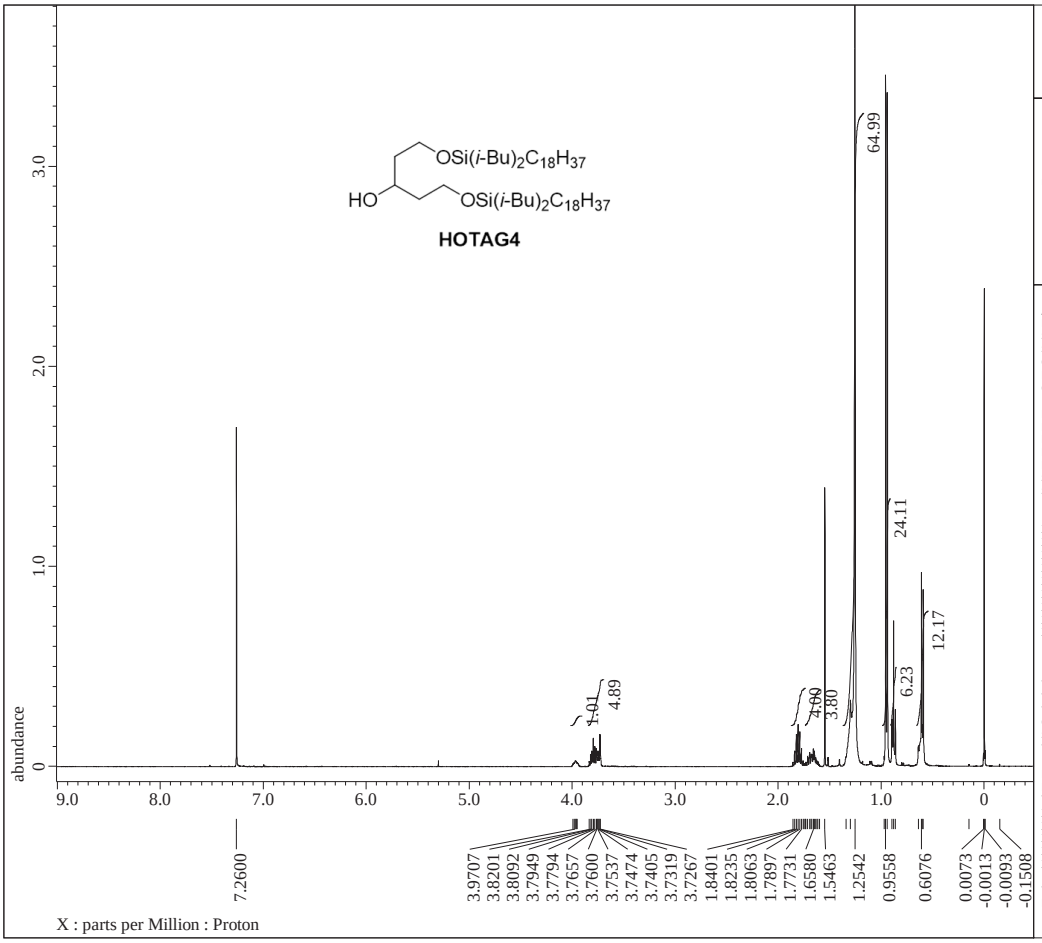
```
Filename          = wuan-275_E1H-1-3.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-275
Solvent           = CHLOROFORM-D
Creation_Time     = 14-DEC-2019 12:26:35
Revision_Time     =  3-NOV-2021 15:44:56
Current_Time      =  3-NOV-2021 15:45:35

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          = 26.3[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
```



JEOL RESONANCE

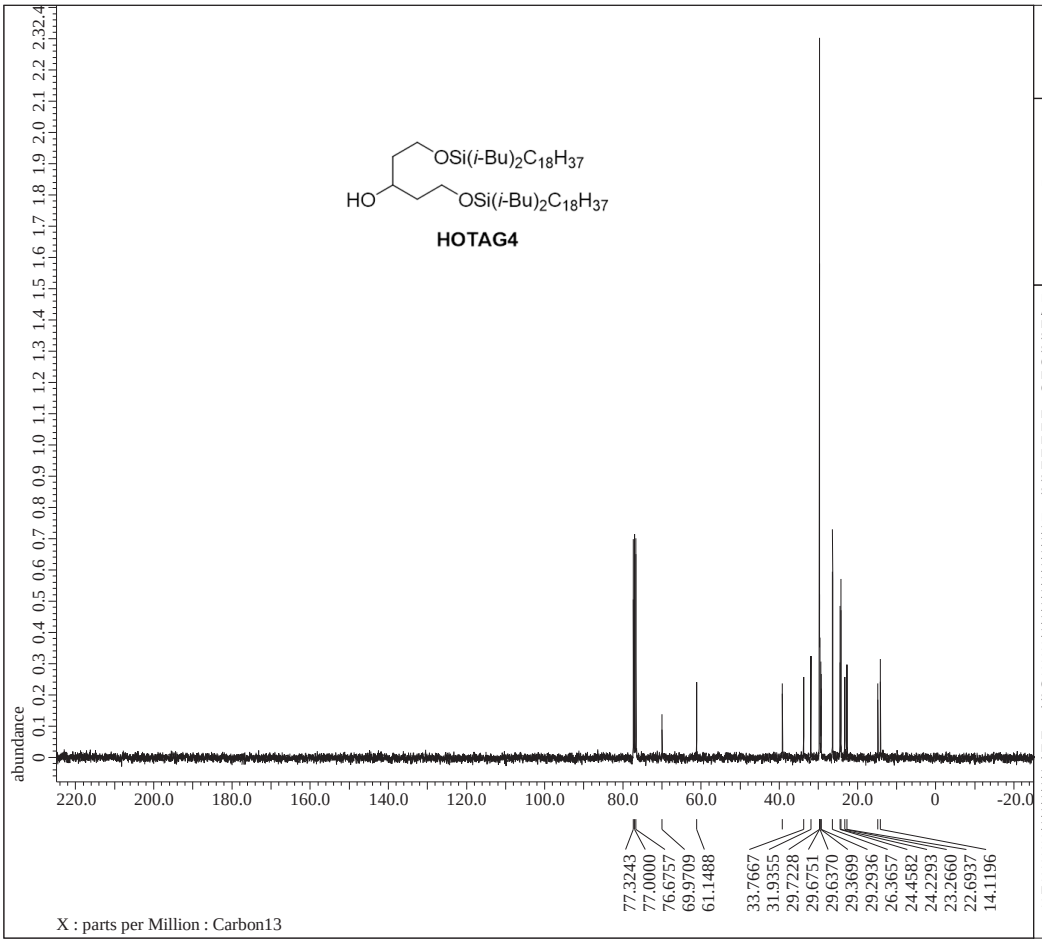
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-288-2_E13C-1-1.jdf
```

```
Filename          = wuan-288-2_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-288-2
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 15:35:55
Revision_Time     =  3-NOV-2021 15:38:36
Current_Time      =  3-NOV-2021 15:38:56

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             = 82
Total_Scans       = 82

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]
```



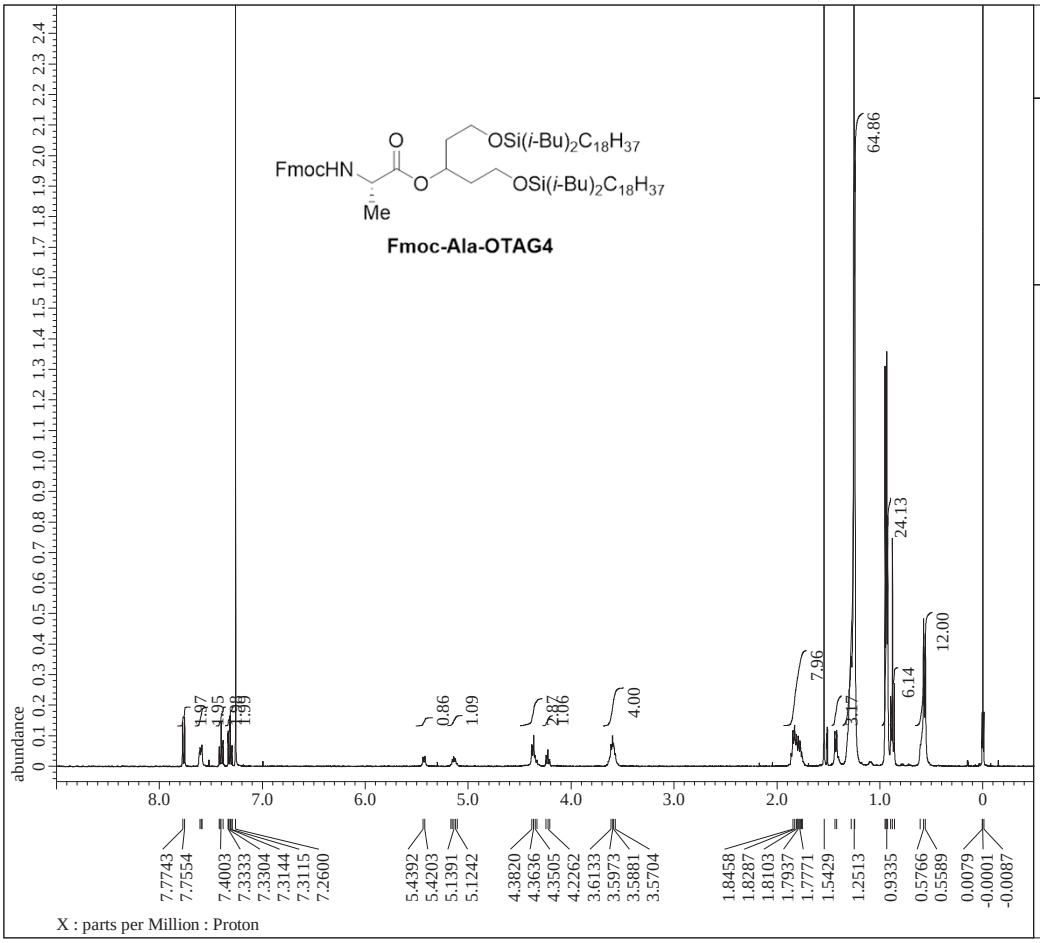

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-608B_E1H-1-1.jdf
```

```
Filename          = wuan-608B_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-608B
Solvent           = CHLOROFORM-D
Creation_Time     =  7-MAY-2021 18:03:08
Revision_Time     =  7-MAY-2021 18:16:36
Current_Time      =  7-MAY-2021 18:17:26

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        = 44
Temp_Get          = 22.3[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
```

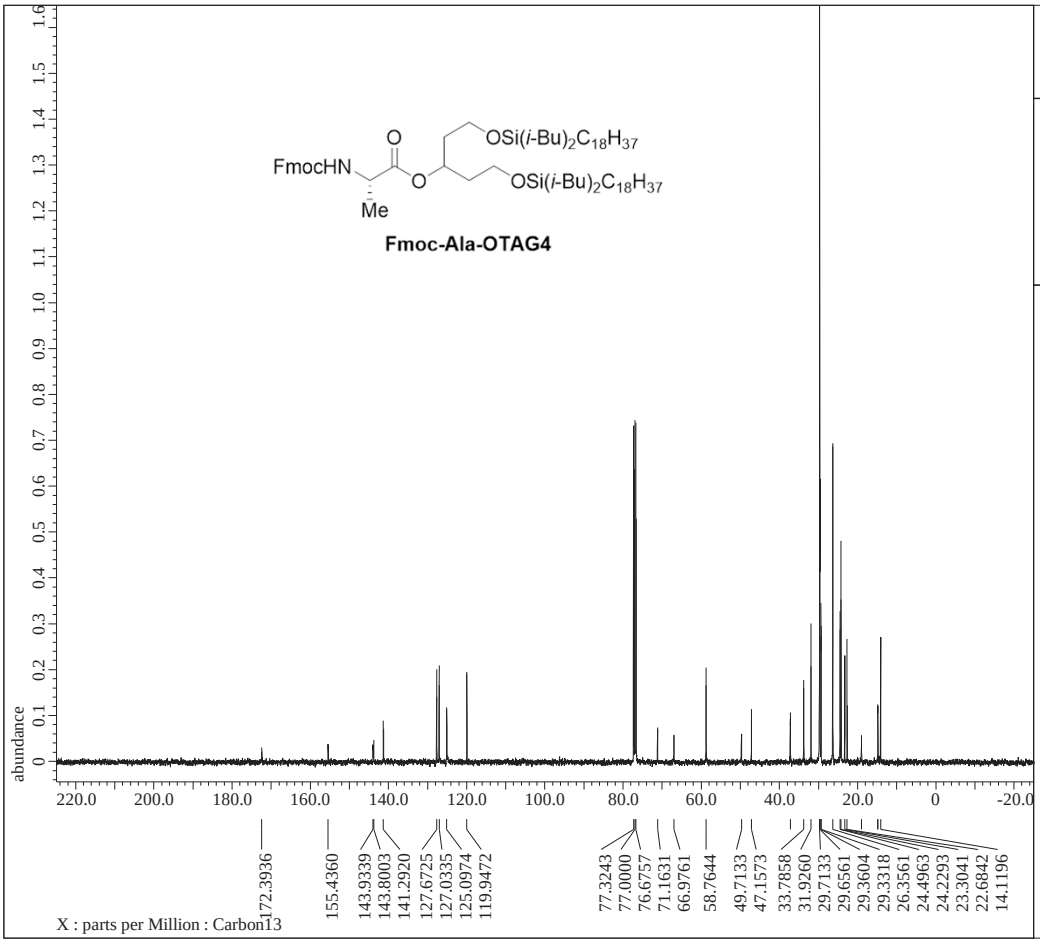



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-572_E13C-1-1.jdf
```

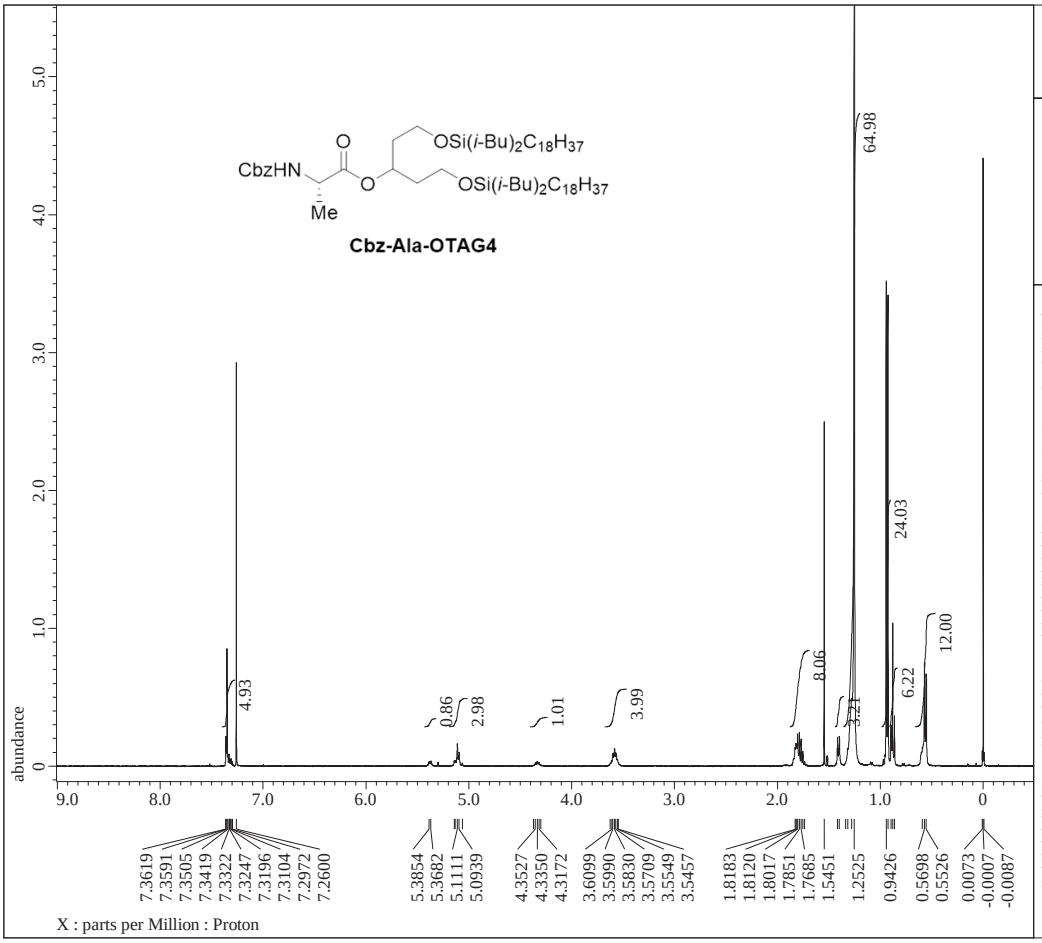
```
Filename          = wuan-572_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-572
Solvent           = CHLOROFORM-D
Creation_Time     =  7-MAY-2021 18:17:11
Revision_Time     =  7-MAY-2021 18:30:30
Current_Time      =  7-MAY-2021 18:31:41

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 80
Total_Scans       = 80

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.3[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[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

Filename          = wuan-574-2_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-574-2
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JUL-2020 12:38:36
Revision_Time     = 10-MAY-2021 15:44:36
Current_Time      = 10-MAY-2021 15:46:09

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        = 46
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
```

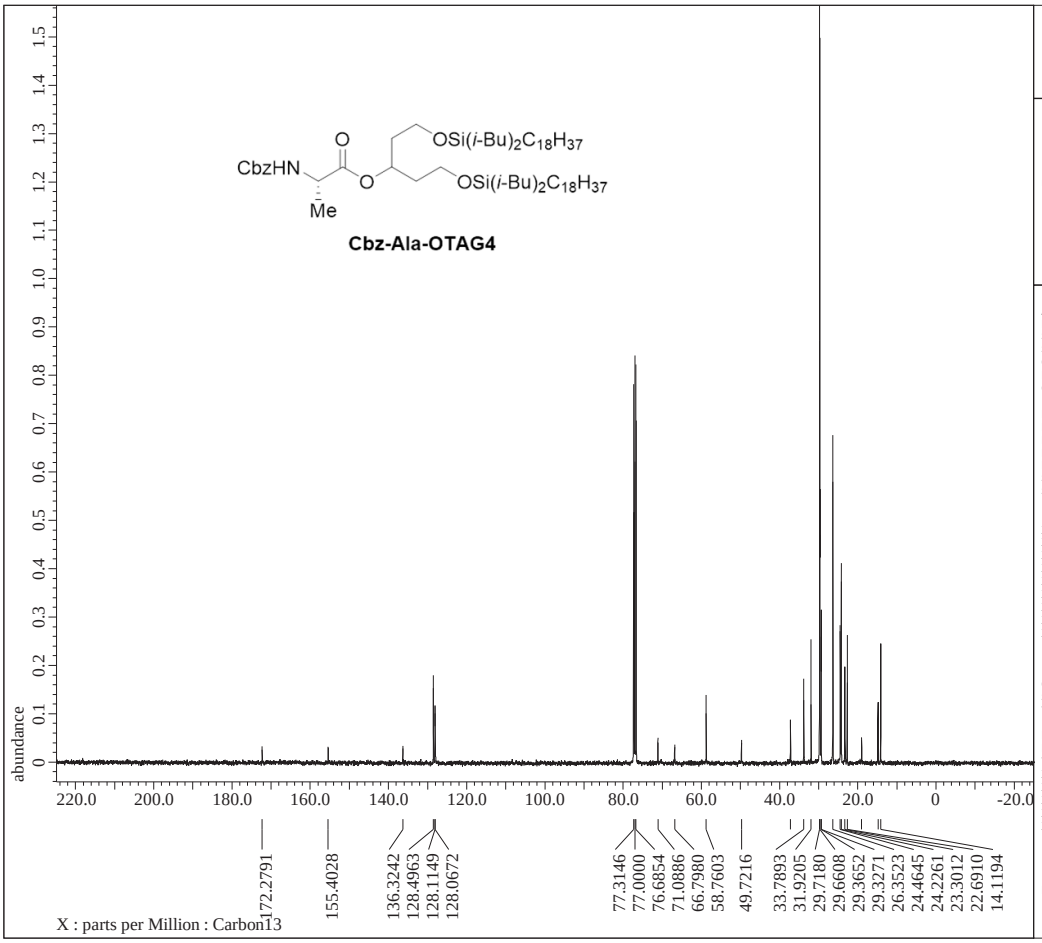



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-574-2_E13C-1-1.jdf

Filename          = wuan-574-2_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-574-2
Solvent           = CHLOROFORM-D
Creation_Time     = 10-MAY-2021 15:31:00
Revision_Time     = 10-MAY-2021 16:22:05
Current_Time      = 10-MAY-2021 16:22:39

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 460.0
Total_Scans       = 460.0

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.4[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



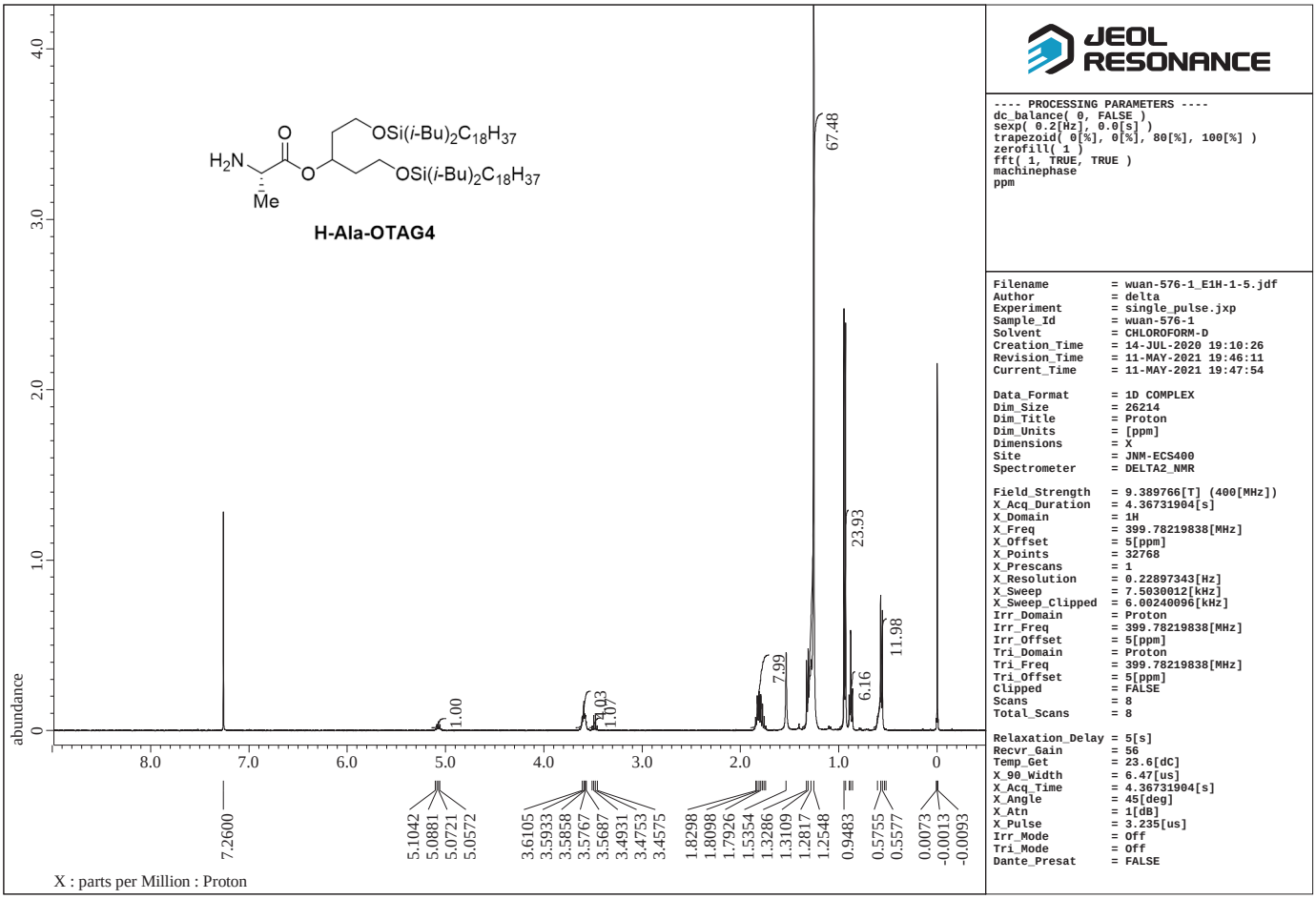

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-604B_E1H-1-1.jdf
```

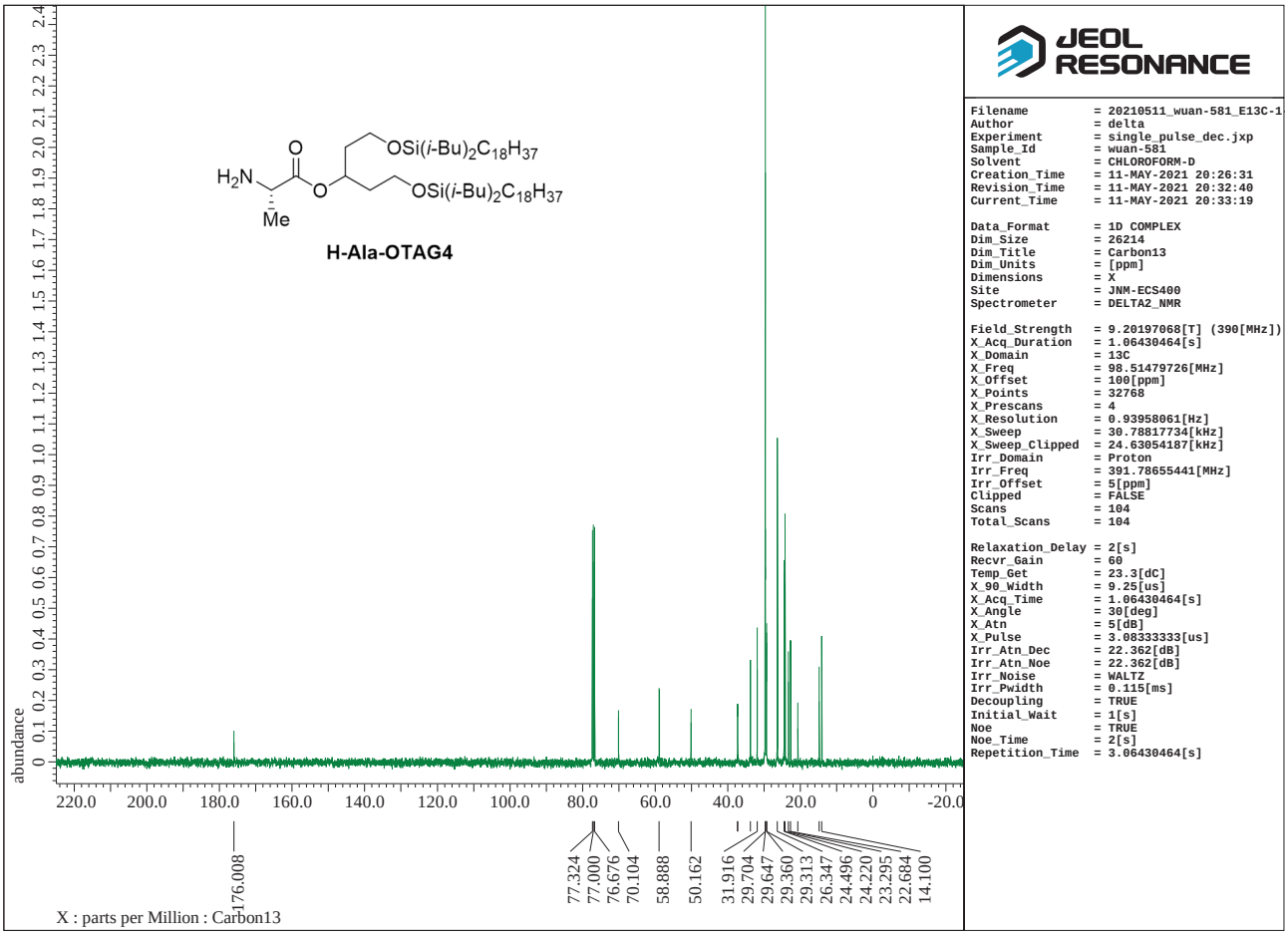
```
Filename          = wuan-604B_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-604B
Solvent           = CHLOROFORM-D
Creation_Time     = 11-MAY-2021 12:47:11
Revision_Time     = 11-MAY-2021 12:53:57
Current_Time      = 11-MAY-2021 12:55:16

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        = 58
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
```

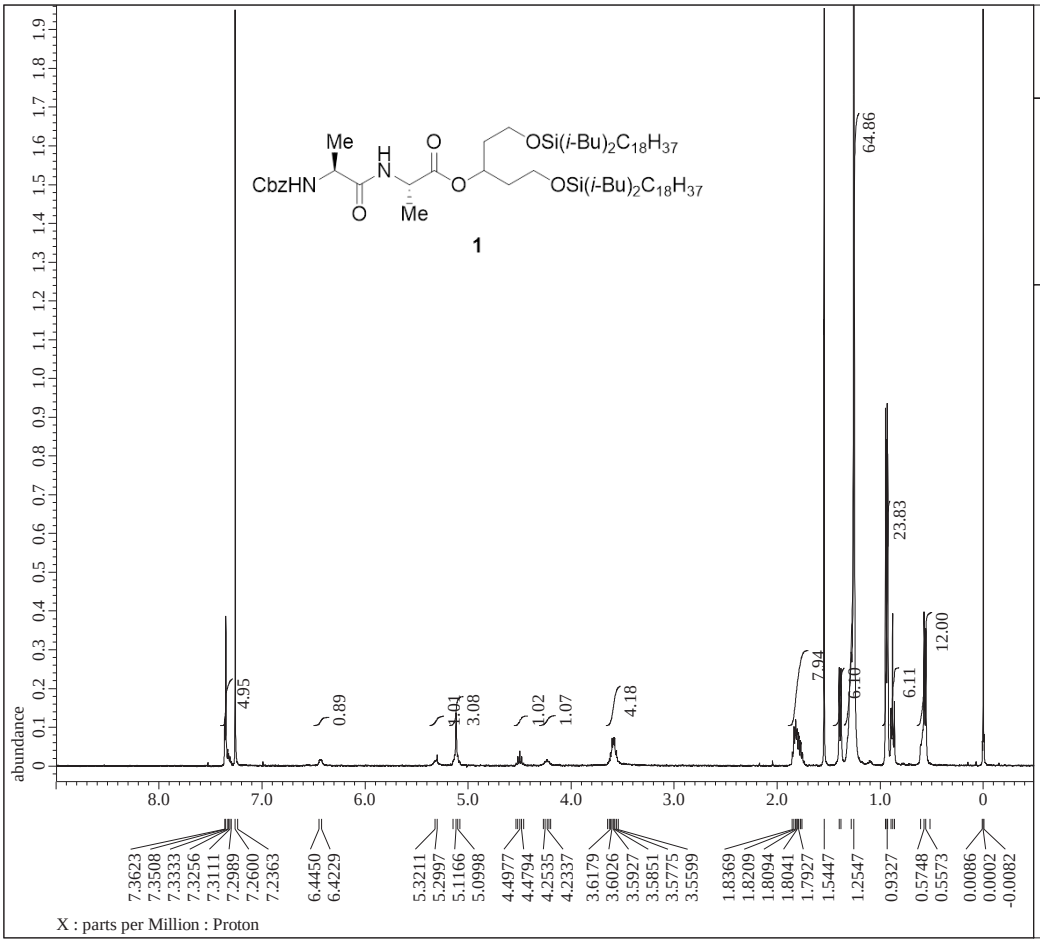



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-387_E13C-1-1.jdf
```

```
Filename          = wuan-387_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-387
Solvent           = CHLOROFORM-D
Creation_Time     = 10-MAY-2021 15:48:41
Revision_Time     = 10-MAY-2021 16:28:30
Current_Time      = 10-MAY-2021 16:31:15

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             = 870.0
Total_Scans       = 870.0

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20.8[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]
```


H-Ala-OTAG4
abundance
X : parts per Million : Proton
---- 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
Filename = wuan-576-1_E1H-1-5.jdf
Author = delta
Experiment = single_pulse.jxp
Sample_Id = wuan-576-1
Solvent = CHLOROFORM-D
Creation_Time = 14-JUL-2020 19:10:26
Revision_Time = 11-MAY-2021 19:46:11
Current_Time = 11-MAY-2021 19:47:54
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 = 56
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

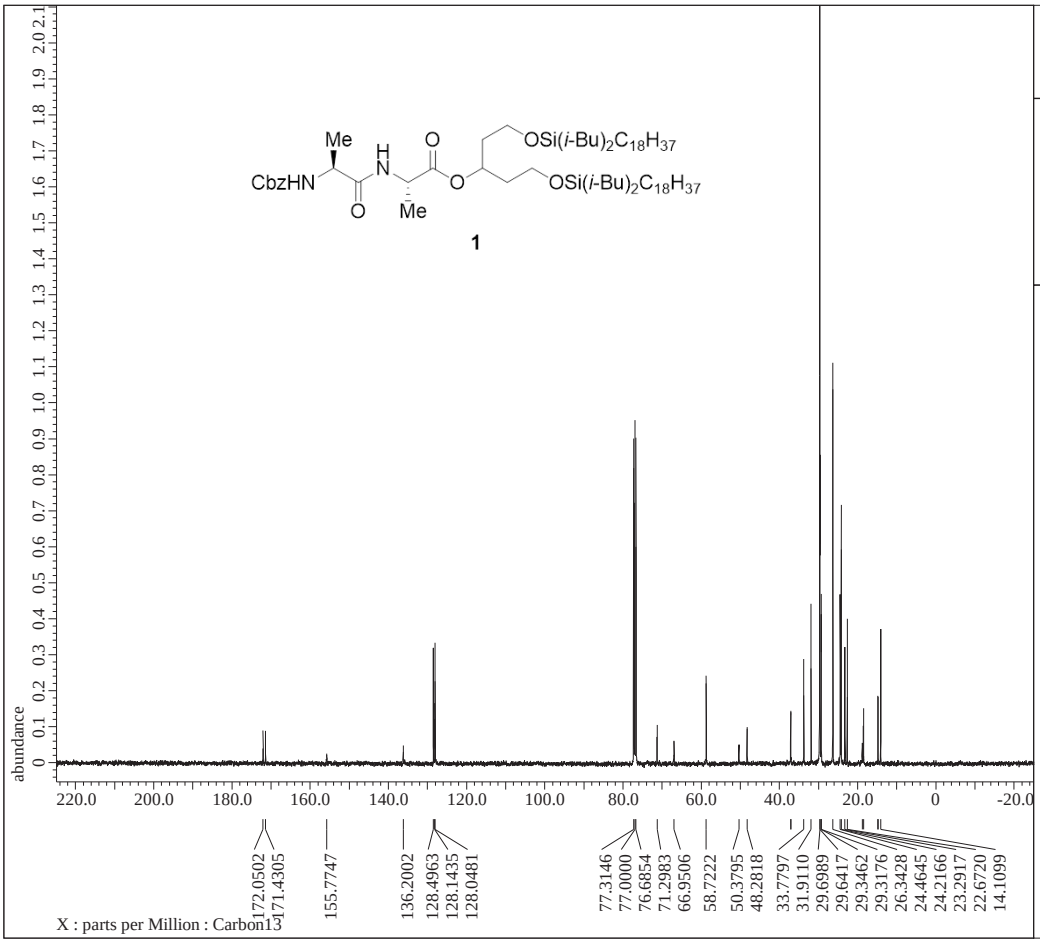

H-Ala-OTAG4
abundance
X : parts per Million : Carbon13
Filename = 20210511_wuan-581_E13C-1
Author = delta
Experiment = single_pulse_dec.jxp
Sample_Id = wuan-581
Solvent = CHLOROFORM-D
Creation_Time = 11-MAY-2021 20:26:31
Revision_Time = 11-MAY-2021 20:32:40
Current_Time = 11-MAY-2021 20:33:19
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
Irr_Domain = Proton
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 104
Total_Scans = 104
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.3[dC]
X_90_Width = 9.25[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 5[dB]
X_Pulse = 3.08333333[us]
Irr_Atn_Dec = 22.362[dB]
Irr_Atn_Noe = 22.362[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.06430464[s]



JEOL RESONANCE

```
---- 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
```

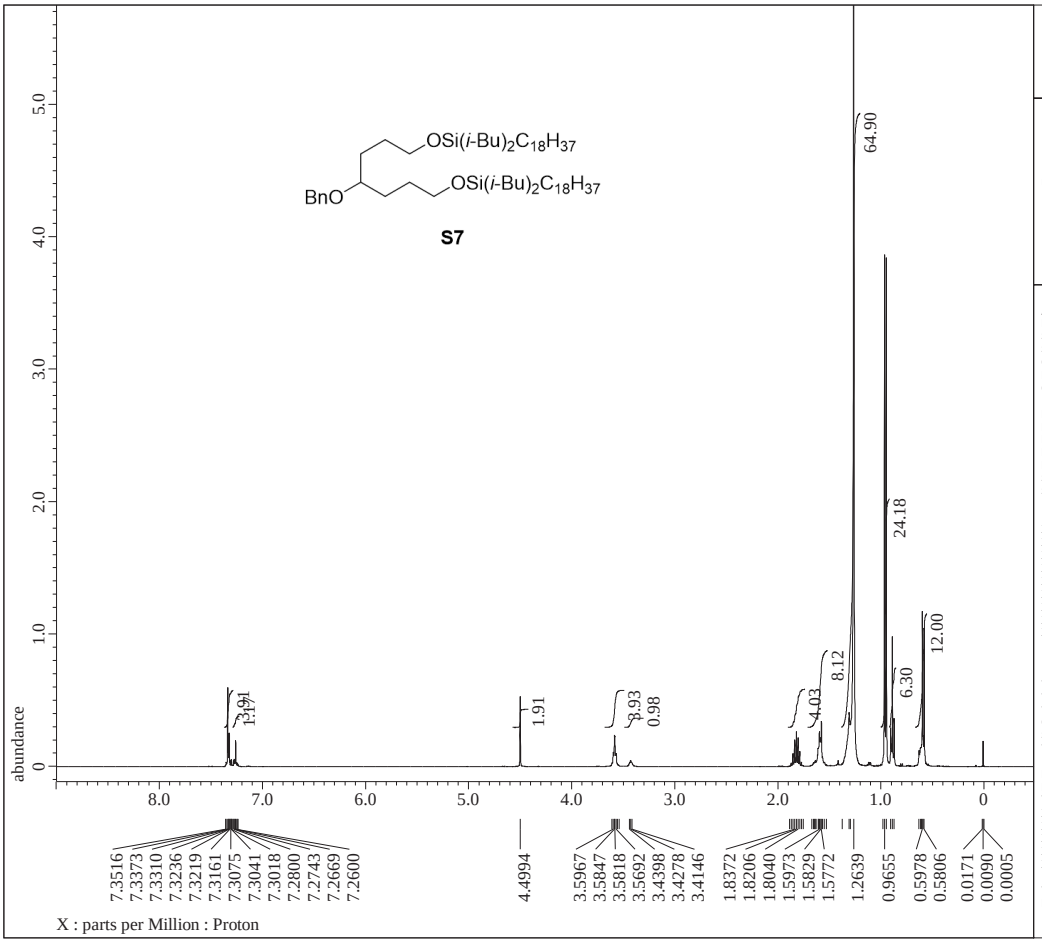
```
Filename          = wuan-451_E1H-1-8.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-451
Solvent           = CHLOROFORM-D
Creation_Time     = 17-APR-2020 11:06:48
Revision_Time     = 14-MAY-2021 17:25:43
Current_Time      = 14-MAY-2021 17:26:26

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 3.34495744[s]
X_Domain          = 1H
X_Freq            = 391.78655441[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.29895747[Hz]
X_Sweep           = 9.79623824[kHz]
X_Sweep_Clipped   = 7.8369906[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 391.78655441[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 24.7[dC]
X_90_Width        = 10.78[us]
X_Acq_Time        = 3.34495744[s]
X_Angle           = 45[deg]
X_Atn             = 1.8[dB]
X_Pulse           = 5.39[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



JEOL RESONANCE

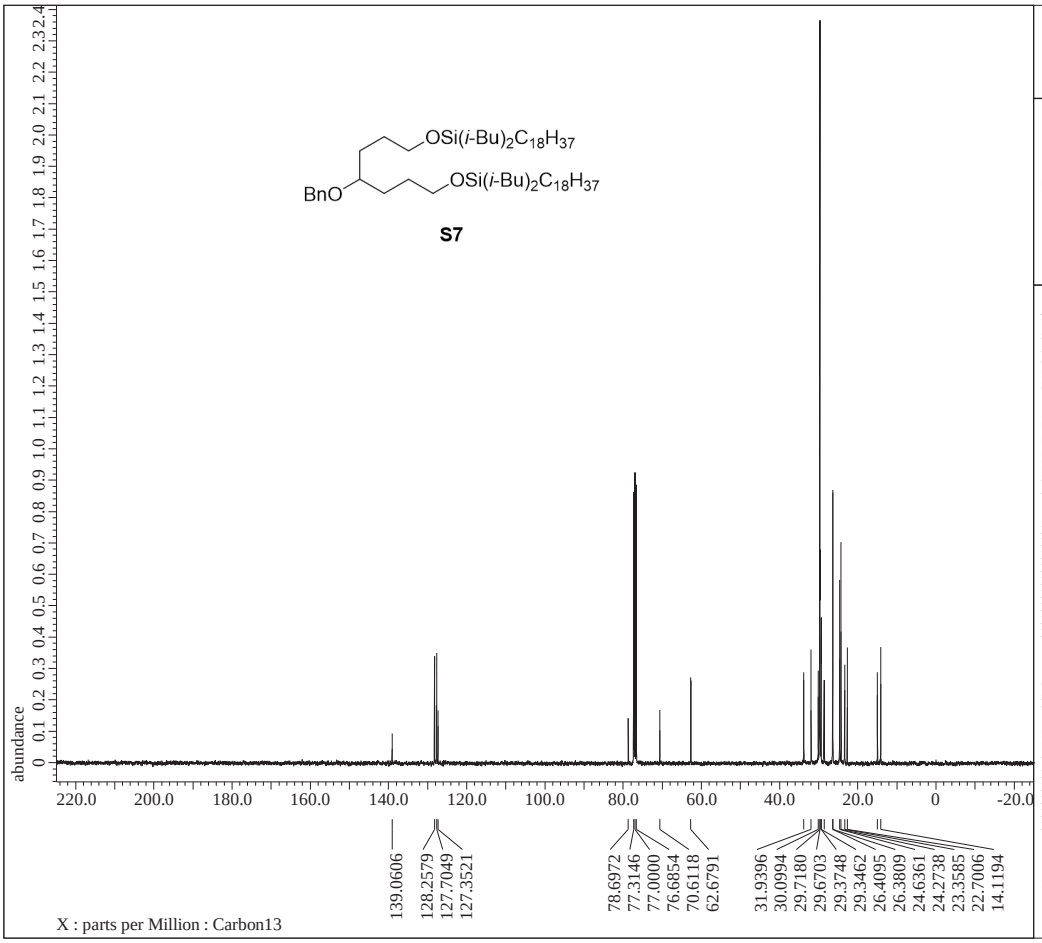
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-394_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-394
Solvent           = CHLOROFORM-D
Creation_Time     = 12-MAY-2021 15:20:52
Revision_Time     = 12-MAY-2021 15:46:37
Current_Time      = 12-MAY-2021 15:48:41

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             = 501
Total_Scans       = 501

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]
```



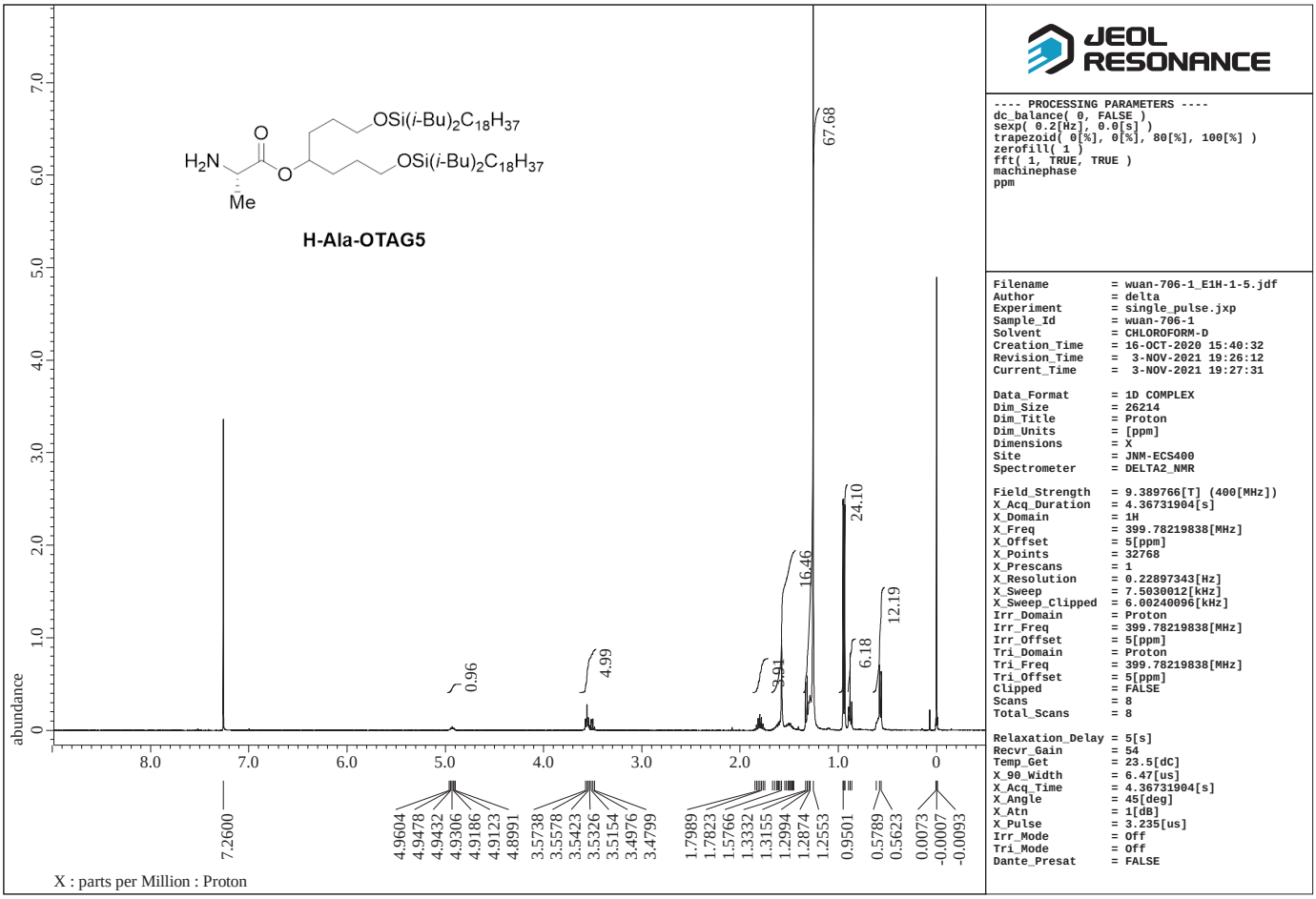

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-688_E1H-1-1.jdf
```

```
Filename          = wuan-688_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-688
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 19:20:22
Revision_Time     =  3-NOV-2021 19:39:57
Current_Time      =  3-NOV-2021 19:41:00

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        = 18
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
```




```
---- 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: wuan-688_E13C-1-1.jdf
```

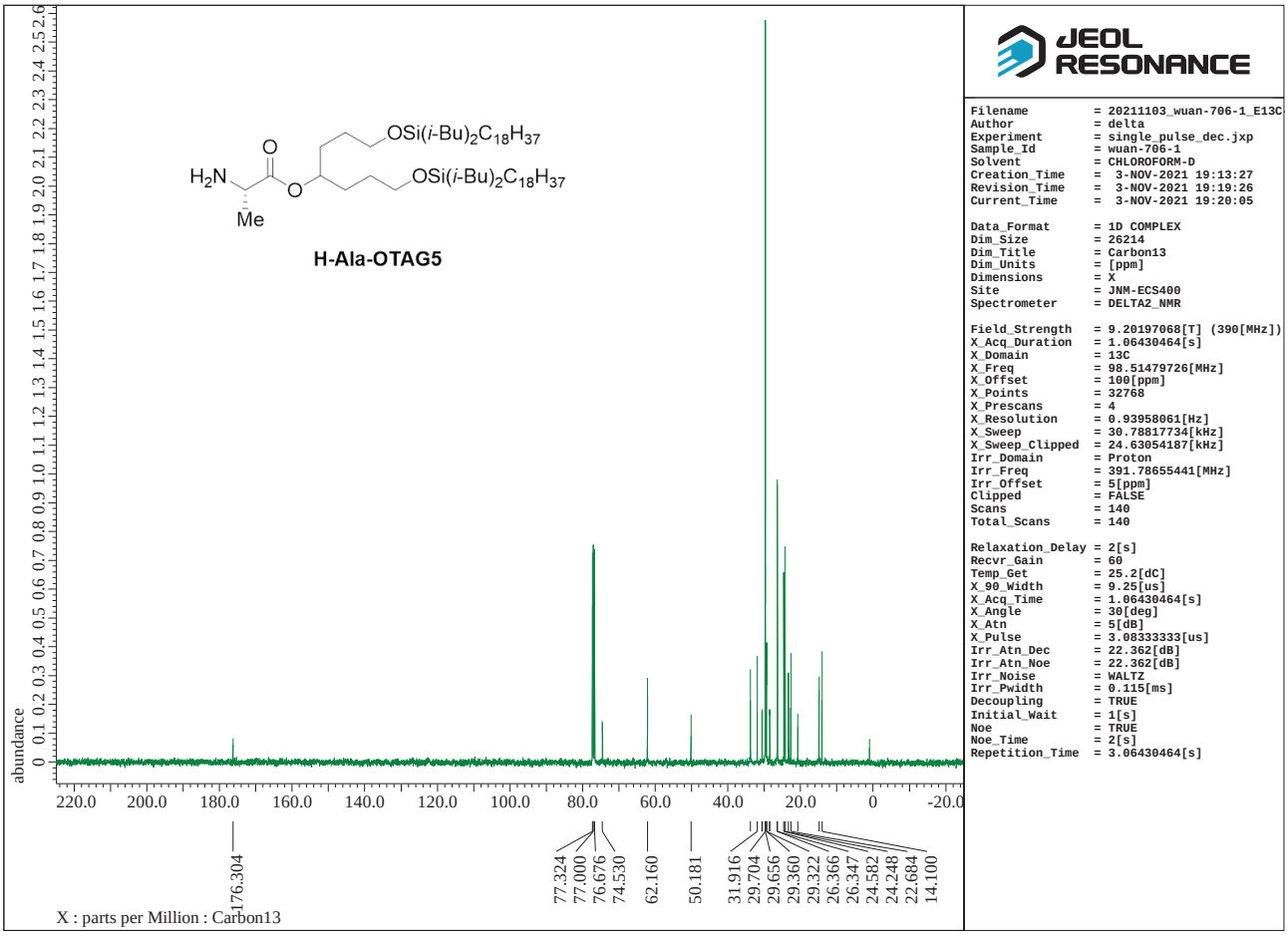
```
Filename          = wuan-688_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-688
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 19:23:55
Revision_Time     =  3-NOV-2021 19:43:24
Current_Time      =  3-NOV-2021 19:44:06

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             = 467
Total_Scans       = 467

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]
```



JEOL RESONANCE

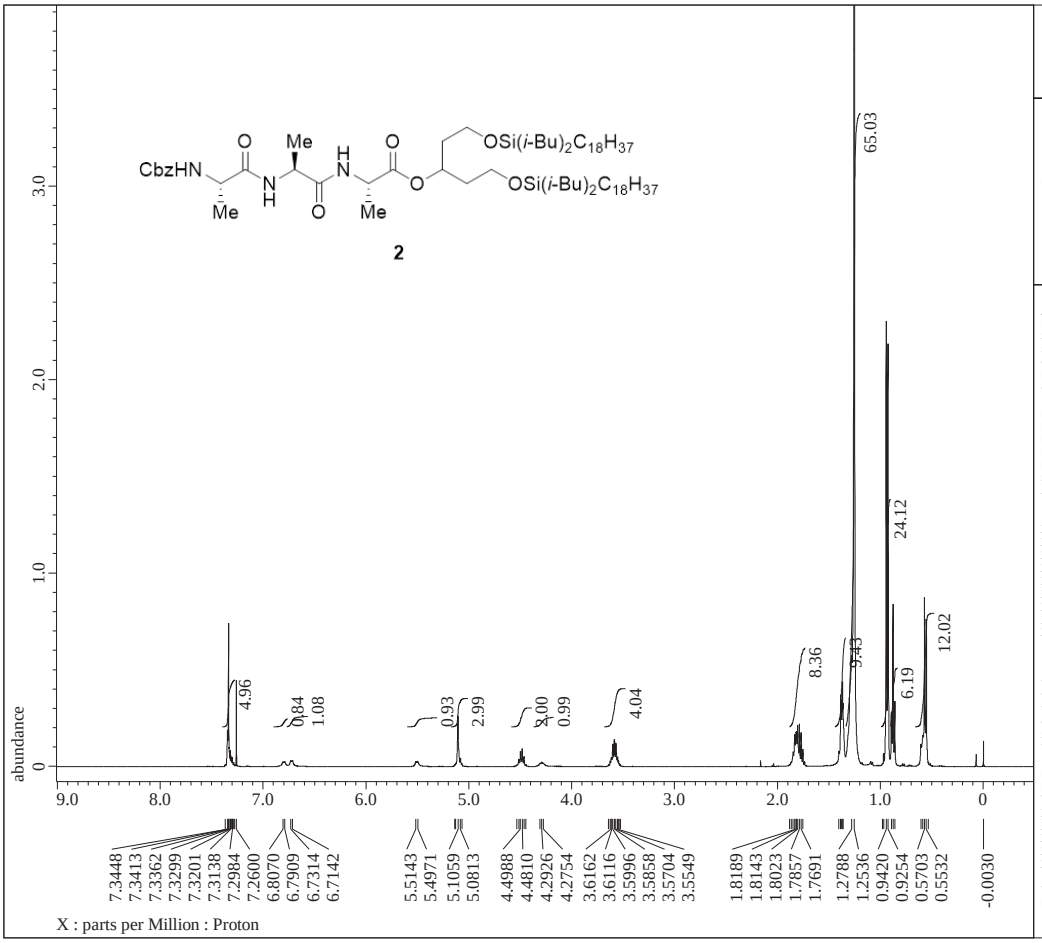
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-706-1_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-706-1
Solvent           = CHLOROFORM-D
Creation_Time     = 16-OCT-2020 15:40:32
Revision_Time     =  3-NOV-2021 19:26:12
Current_Time      =  3-NOV-2021 19:27:31

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        = 54
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
```



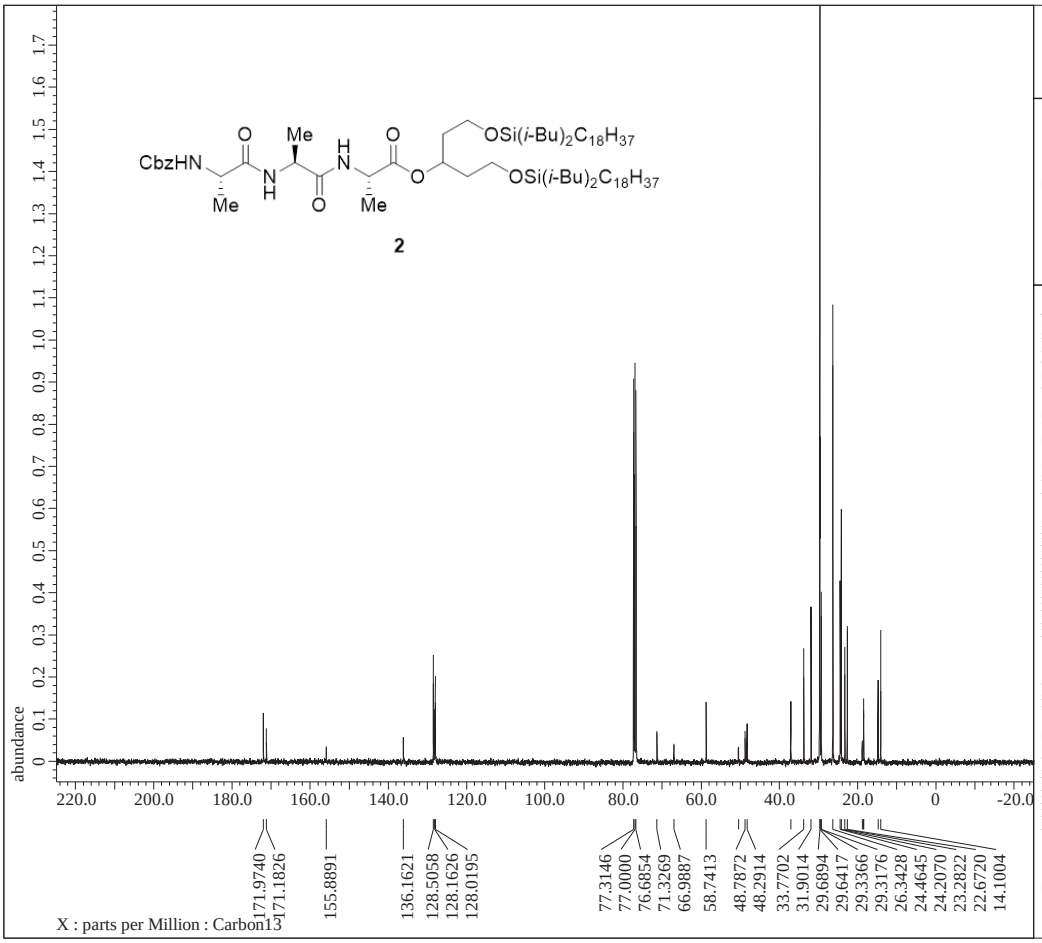
JEOL RESONANCE

```
Filename          = 20211103_wuan-706-1_E13C
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-706-1
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 19:13:27
Revision_Time     =  3-NOV-2021 19:19:26
Current_Time      =  3-NOV-2021 19:20:05

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 140
Total_Scans       = 140

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25.2[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.06430464[s]
```



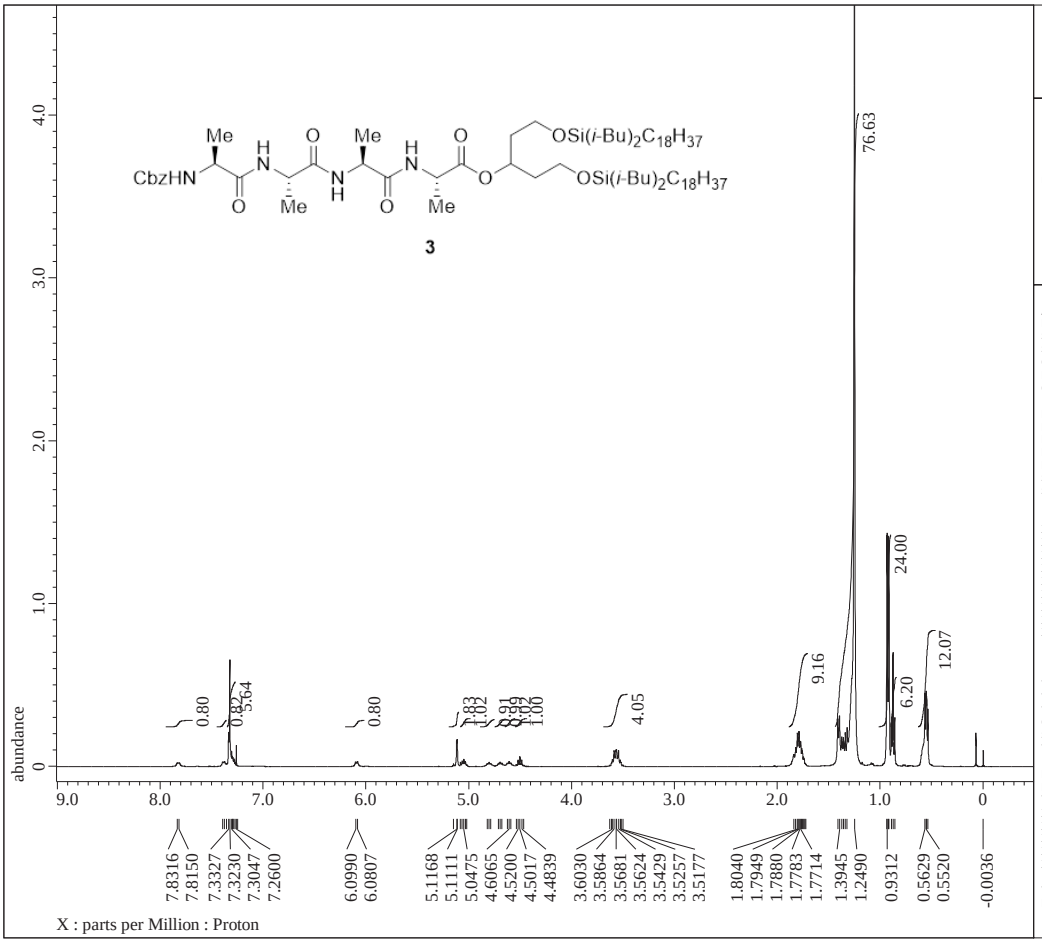

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-429B_E1H-1-1.jdf
```

```
Filename          = wuan-429B_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-429B
Solvent           = CHLOROFORM-D
Creation_Time     = 14-MAY-2021 17:10:25
Revision_Time     = 14-MAY-2021 17:17:56
Current_Time      = 14-MAY-2021 17:19:07

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          = 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
```

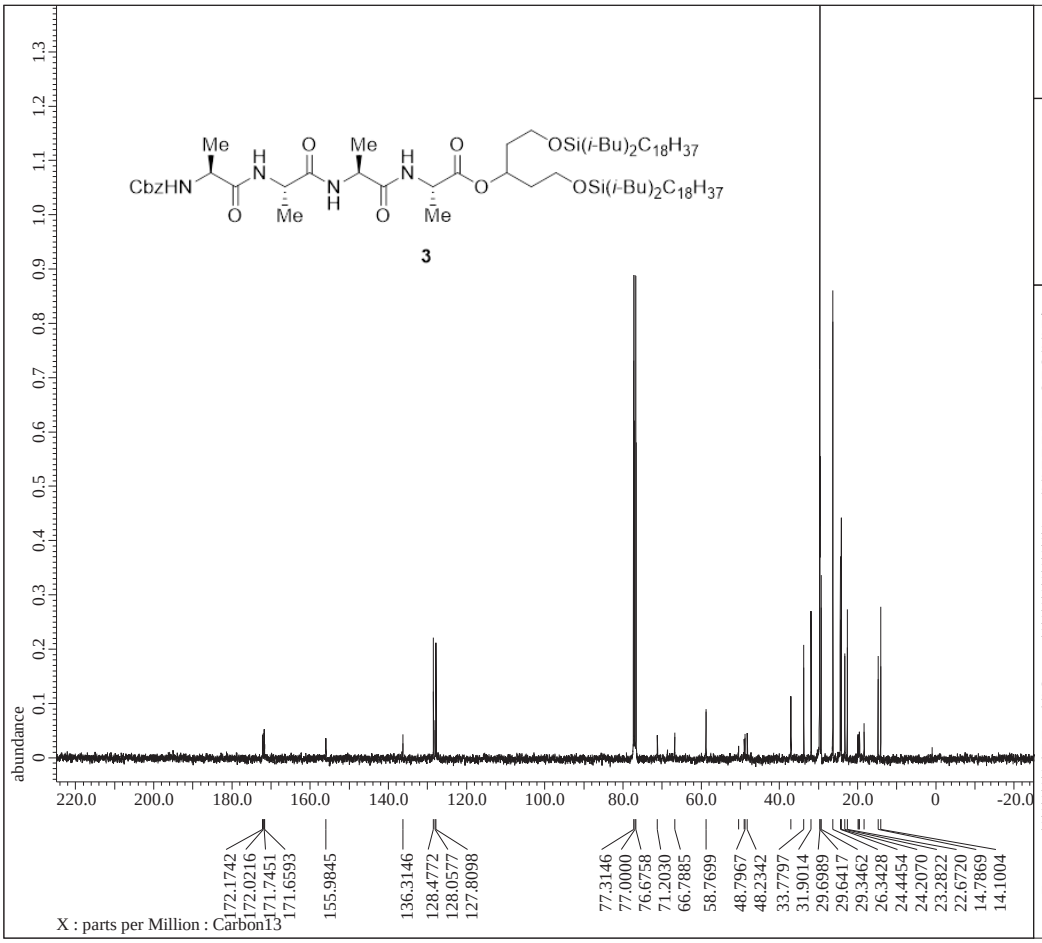



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-429B_E13C-1-1.jdf
```

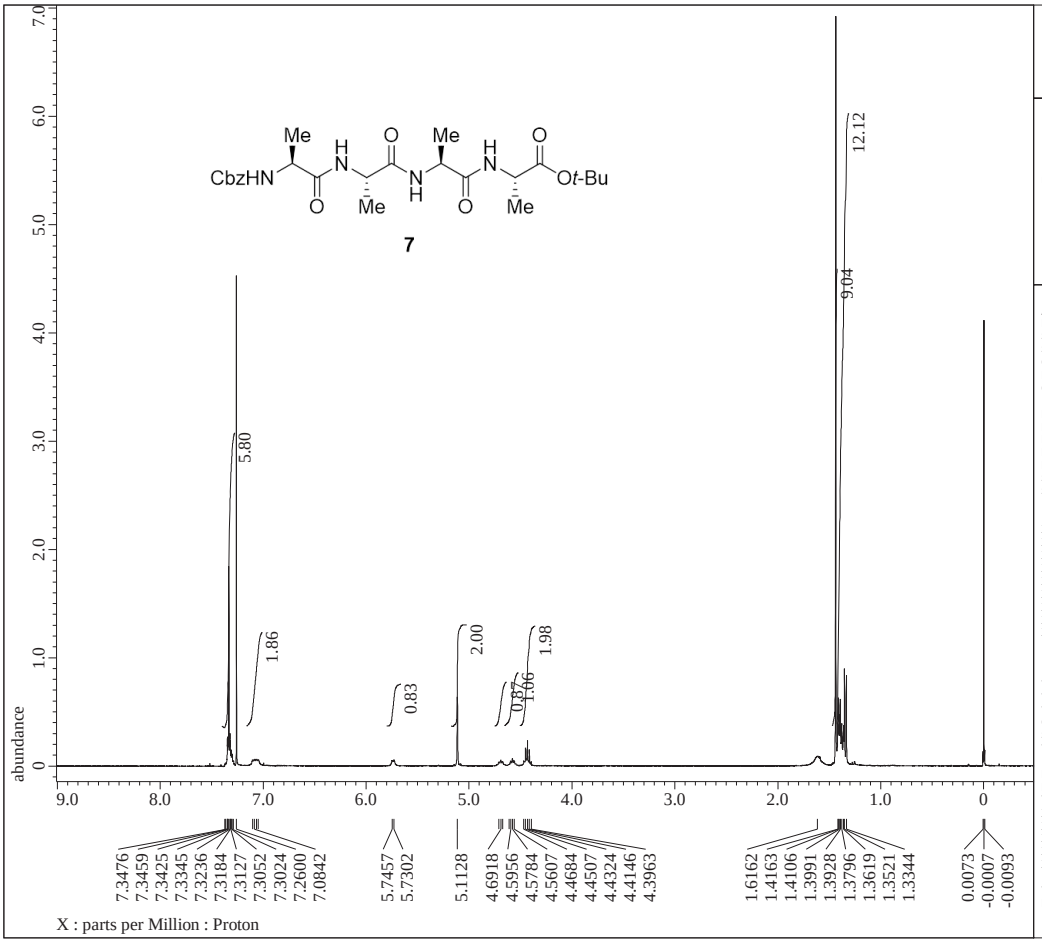
```
Filename          = wuan-429B_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-429B
Solvent           = CHLOROFORM-D
Creation_Time     = 14-MAY-2021 17:12:15
Revision_Time     = 14-MAY-2021 17:33:51
Current_Time      = 14-MAY-2021 17:34:18

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             = 503
Total_Scans       = 503

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]
```




```
---- 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

Filename          = wuan-434C_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-434C
Solvent           = CHLOROFORM-D
Creation_Time     = 20-MAY-2021 18:38:20
Revision_Time     =  4-NOV-2021 09:31:27
Current_Time      =  4-NOV-2021 09:32:18

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        = 20
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
```



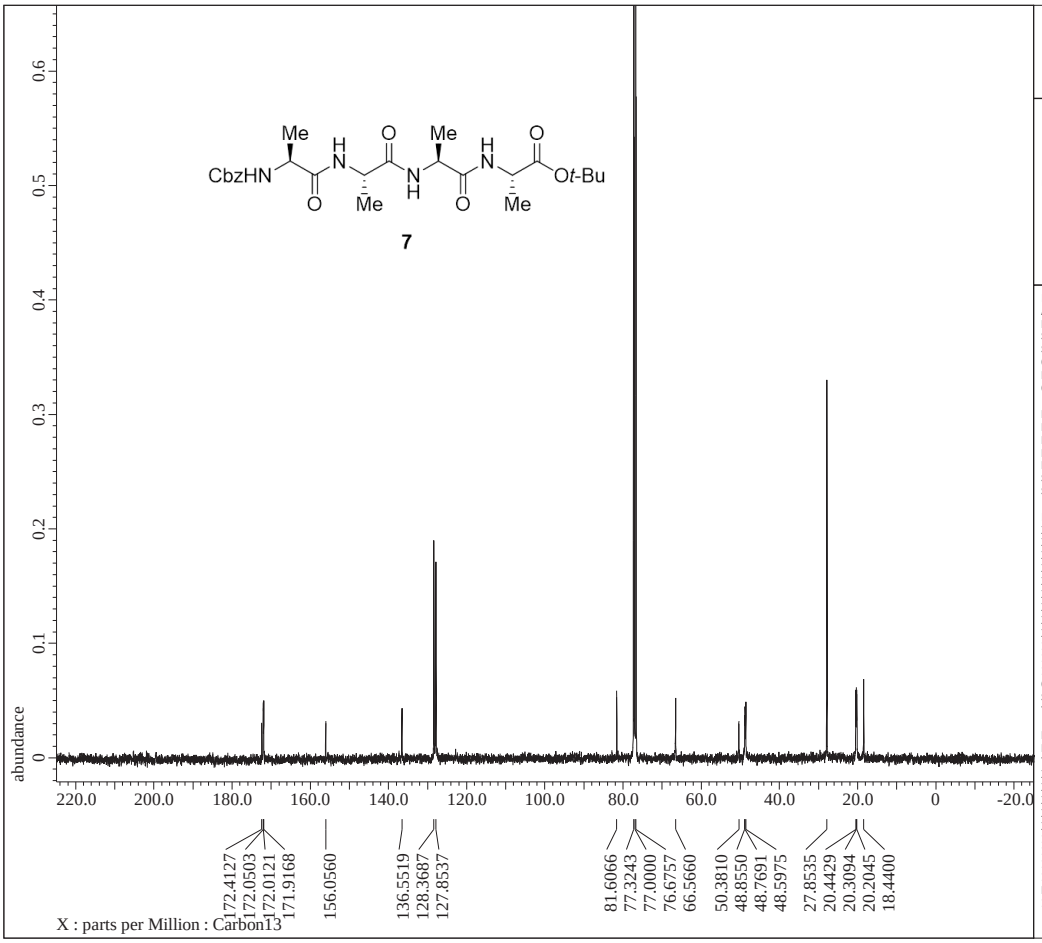

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-434C_E13C-1-1.jdf

Filename          = wuan-434C_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-434C
Solvent           = CHLOROFORM-D
Creation_Time     = 20-MAY-2021 18:40:10
Revision_Time     = 20-MAY-2021 18:52:36
Current_Time      = 20-MAY-2021 18:54:09

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             = 321
Total_Scans       = 321

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]
```



JEOL RESONANCE

```
---- 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
```

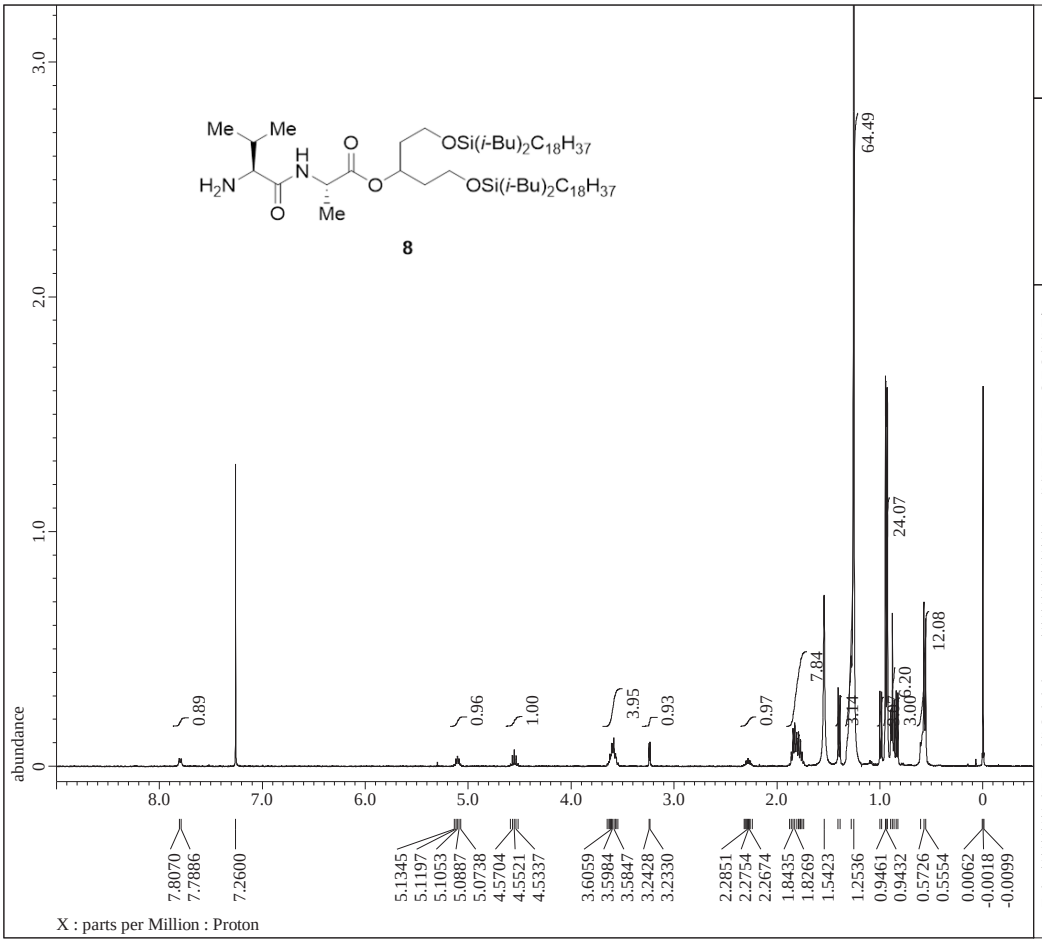
```
Filename          = wuan-412_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-412
Solvent           = CHLOROFORM-D
Creation_Time     = 28-MAR-2020 10:09:52
Revision_Time     = 19-MAY-2021 13:41:12
Current_Time      = 19-MAY-2021 13:41:55

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             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
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
```



JEOL RESONANCE

```
---- 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: wuan-412_E13C-1-1.jdf
```

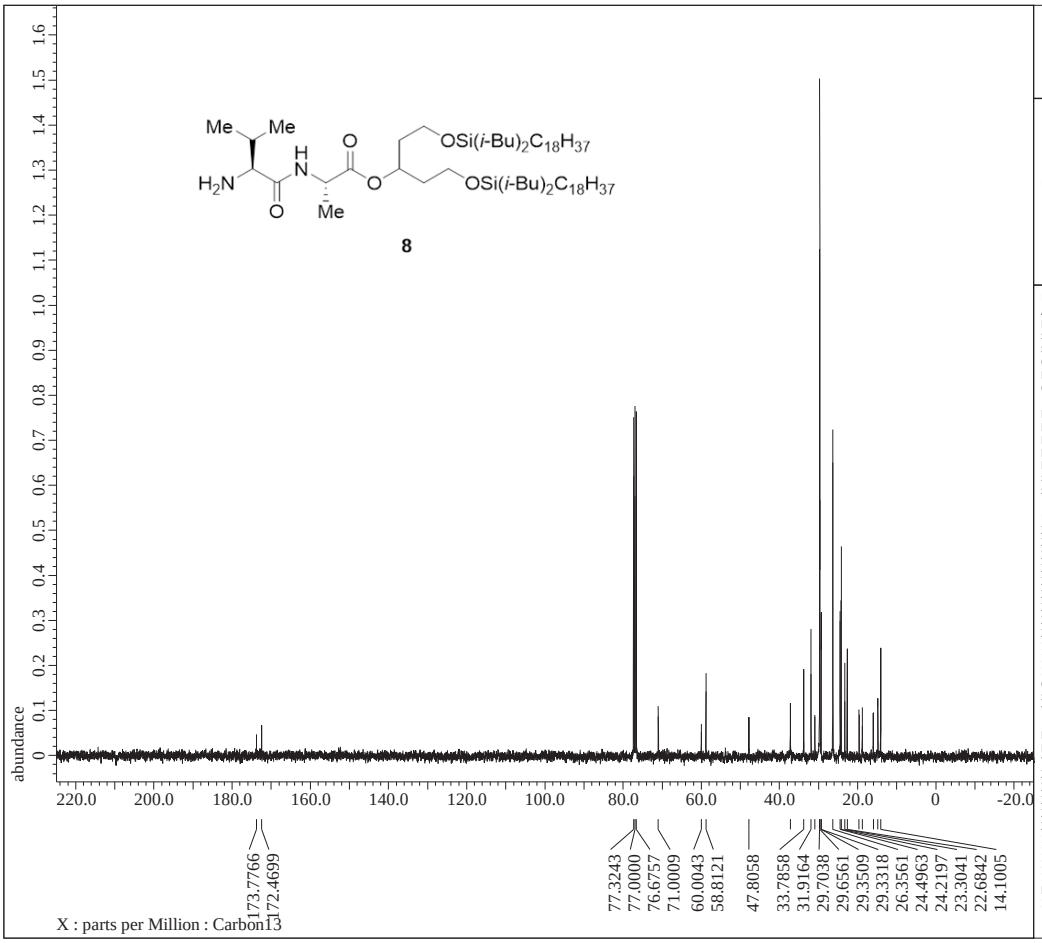
```
Filename          = wuan-412_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-412
Solvent           = CHLOROFORM-D
Creation_Time     = 19-MAY-2021 18:36:43
Revision_Time     = 19-MAY-2021 19:36:46
Current_Time      = 19-MAY-2021 19:37:08

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.7[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



JEOL RESONANCE

```
---- 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
```

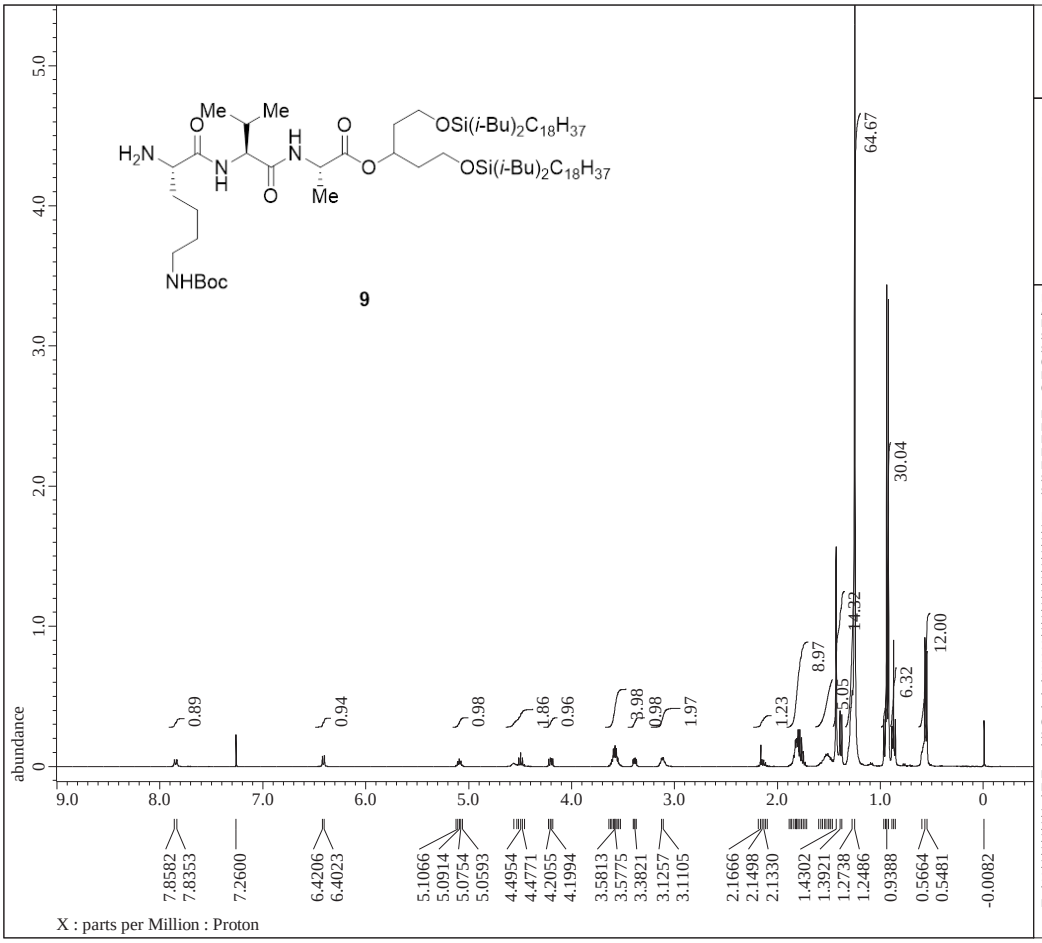
```
Filename          = wuan-558_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-558
Solvent           = CHLOROFORM-D
Creation_Time     = 30-JUN-2020 08:50:43
Revision_Time     = 10-DEC-2021 21:27:24
Current_Time      = 10-DEC-2021 21:28:24

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        = 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
```



JEOL RESONANCE

```
---- 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: wuan-558_E13C-1-1.jdf
```

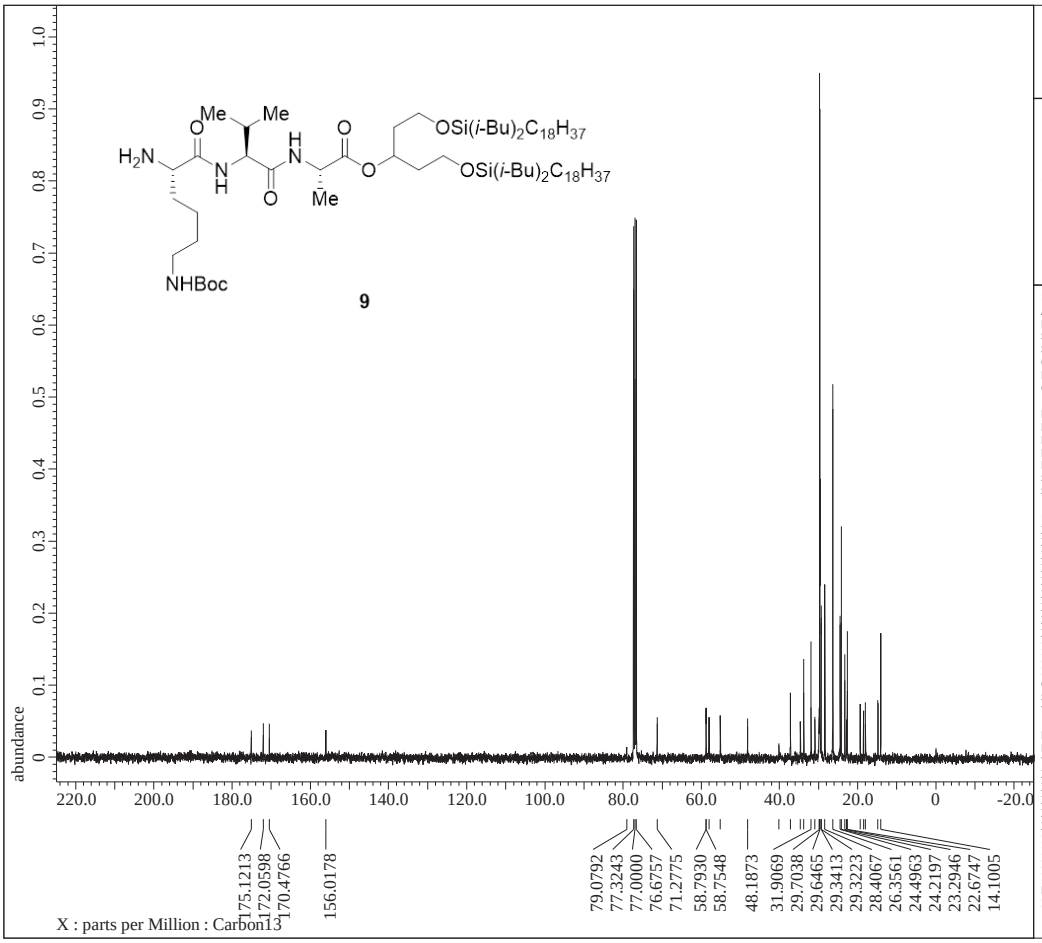
```
Filename          = wuan-558_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-558
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 21:19:17
Revision_Time     = 10-DEC-2021 21:31:24
Current_Time      = 10-DEC-2021 21:33:25

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 134
Total_Scans       = 134

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.7[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



JEOL RESONANCE

```
---- 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
```

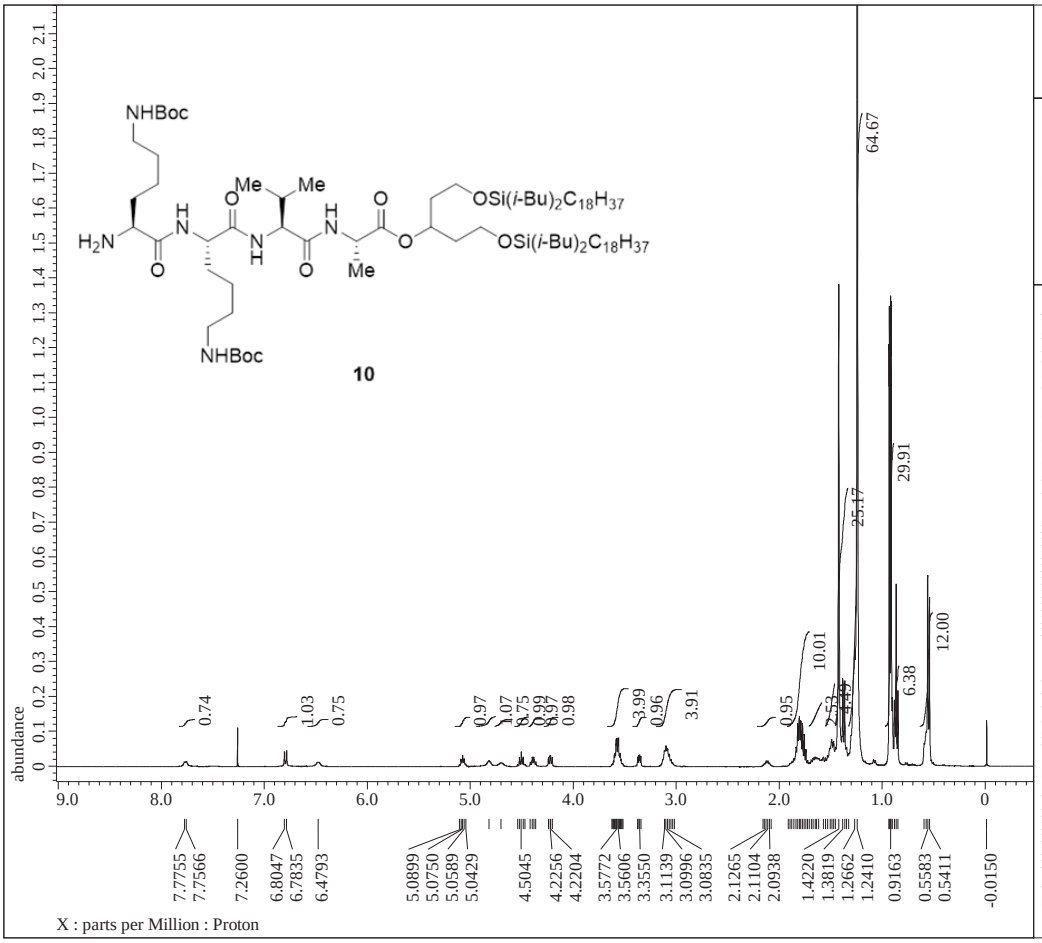
```
Filename          = wuan-617_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-617
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 21:42:26
Revision_Time     = 10-DEC-2021 22:02:11
Current_Time      = 10-DEC-2021 22:02:39

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 3.34495744[s]
X_Domain          = 1H
X_Freq            = 391.78655441[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.29895747[Hz]
X_Sweep           = 9.79623824[kHz]
X_Sweep_Clipped   = 7.8369906[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 391.78655441[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 24.6[dC]
X_90_Width        = 10.78[us]
X_Acq_Time        = 3.34495744[s]
X_Angle           = 45[deg]
X_Atn             = 1.8[dB]
X_Pulse           = 5.39[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



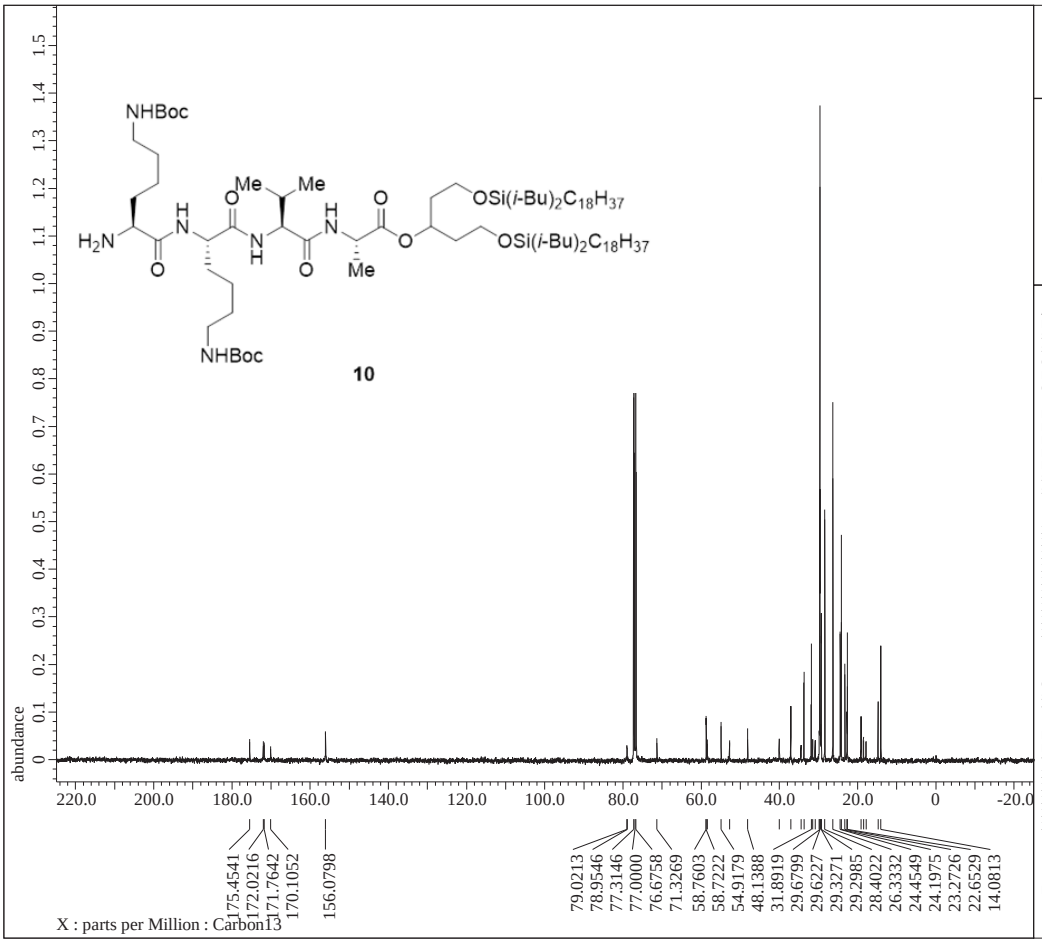
JEOL RESONANCE

```
---- 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
```

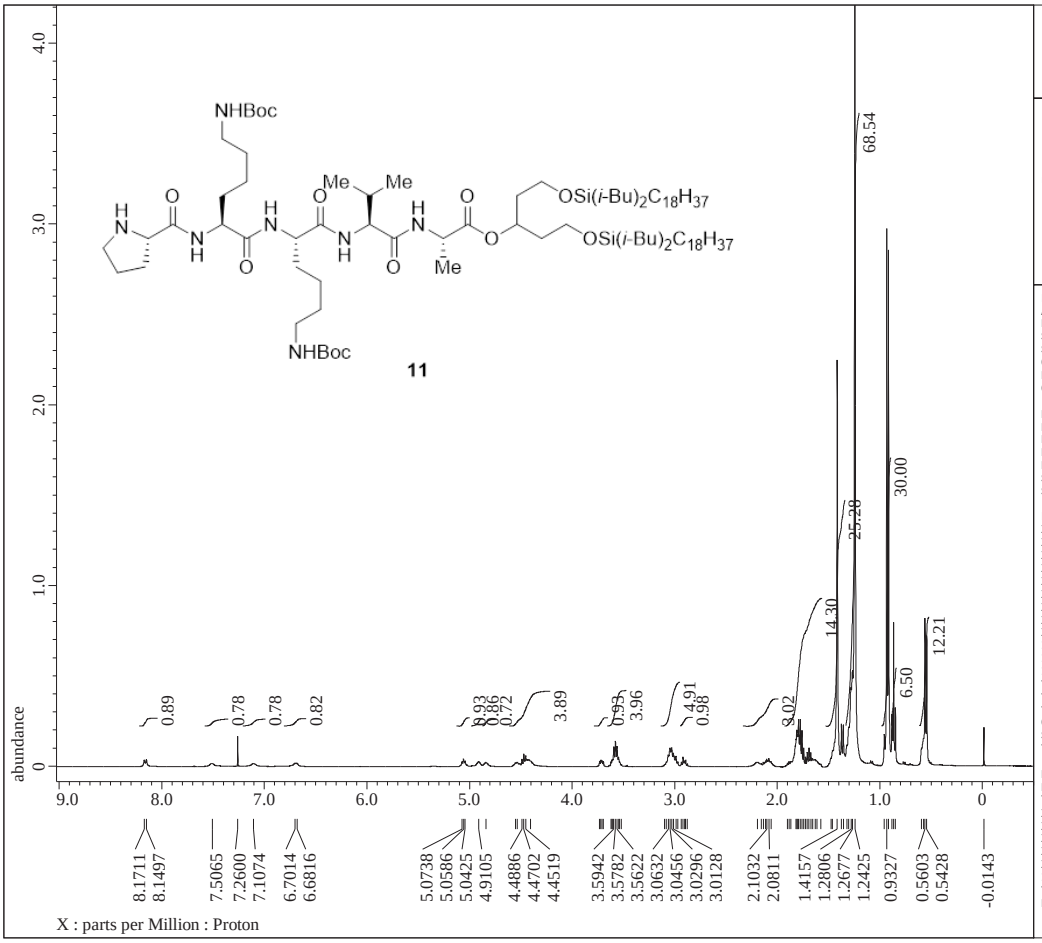
```
Filename          = wuan-617_E13C-1-6.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-617
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 21:45:41
Revision_Time     = 16-DEC-2021 15:04:27
Current_Time      = 16-DEC-2021 15:05:07

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 412
Total_Scans       = 412

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.4[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



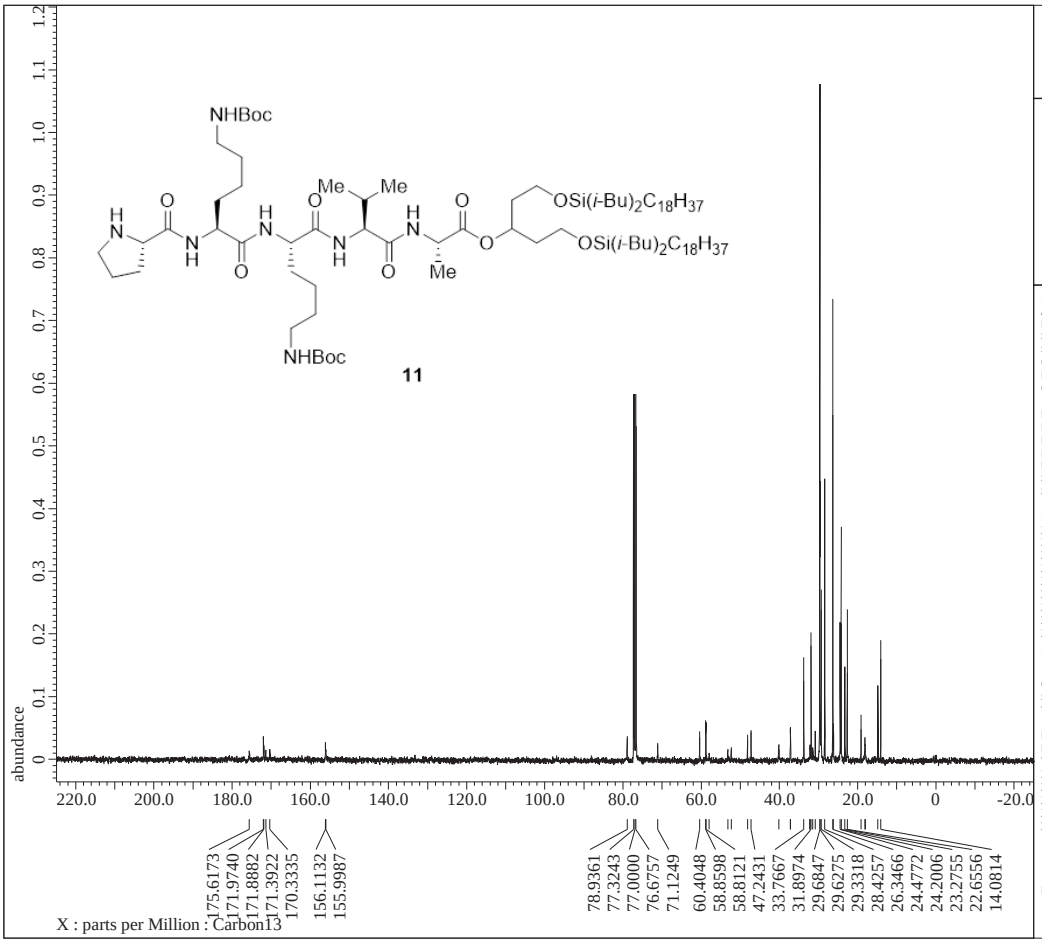

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-619_E1H-1-1.jdf

Filename          = wuan-619_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-619
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 22:11:27
Revision_Time     = 10-DEC-2021 22:36:49
Current_Time      = 10-DEC-2021 22:37:43

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        = 20
Temp_Get          = 23.4[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
```



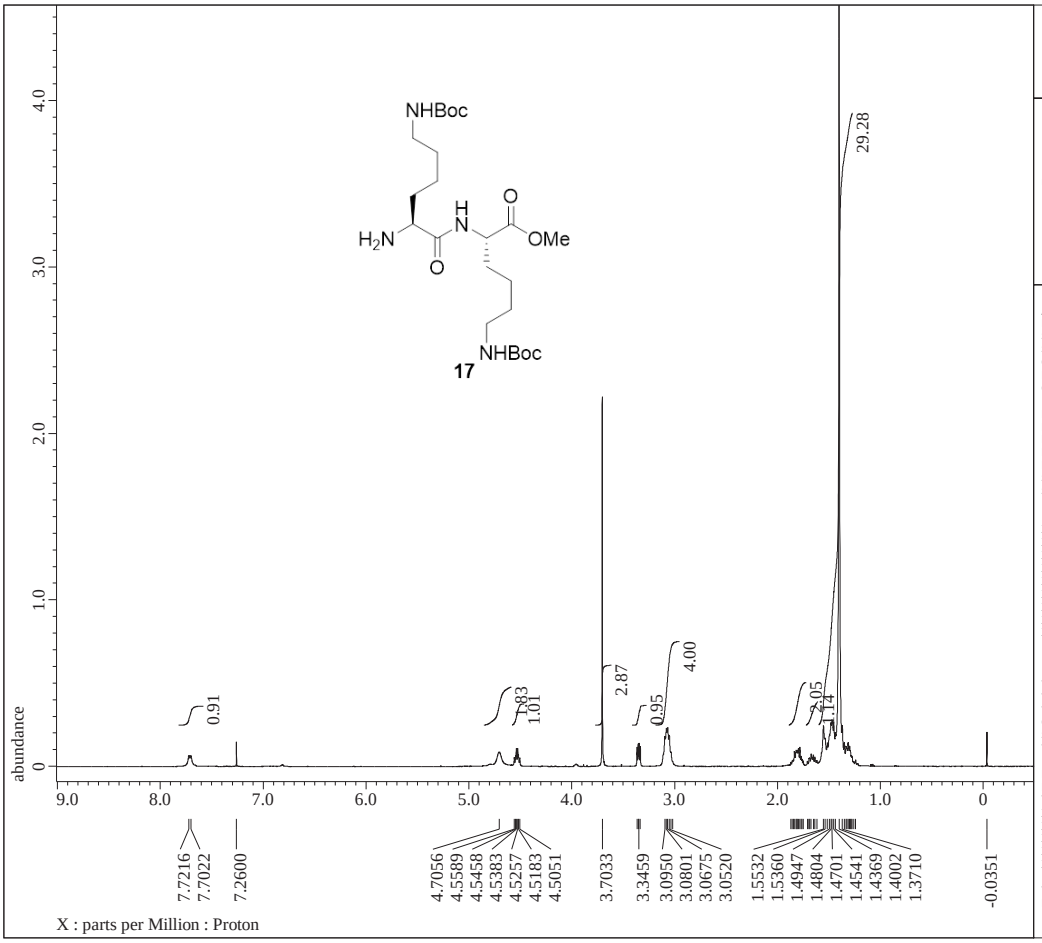

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = wuan-619_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-619
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 22:16:16
Revision_Time     = 16-DEC-2021 14:51:44
Current_Time      = 16-DEC-2021 15:02:03

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             = 792
Total_Scans       = 792

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]
```




```
---- 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

Filename          = wuan-622_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-622
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 22:11:57
Revision_Time     = 11-DEC-2021 11:38:22
Current_Time      = 11-DEC-2021 11:39:39

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 3.34495744[s]
X_Domain          = 1H
X_Freq            = 391.78655441[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.29895747[Hz]
X_Sweep           = 9.79623824[kHz]
X_Sweep_Clipped   = 7.8369906[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 391.78655441[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        = 10.78[us]
X_Acq_Time        = 3.34495744[s]
X_Angle           = 45[deg]
X_Atn             = 1.8[dB]
X_Pulse           = 5.39[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



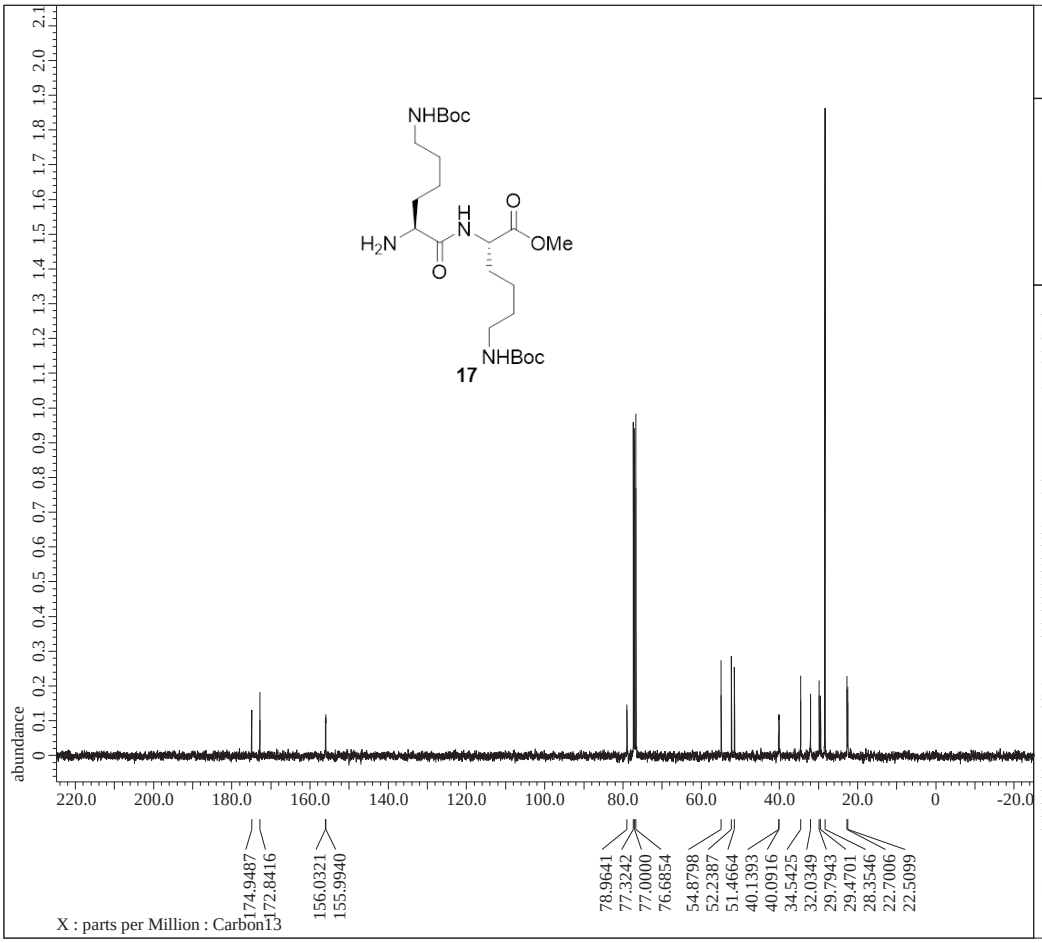

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = wuan-622_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-622
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 22:15:10
Revision_Time     = 11-DEC-2021 12:14:20
Current_Time      = 11-DEC-2021 14:15:13

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 810
Total_Scans       = 810

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.8[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



JEOL RESONANCE

```
---- 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: wuan-666_E1H-1-1.jdf
```

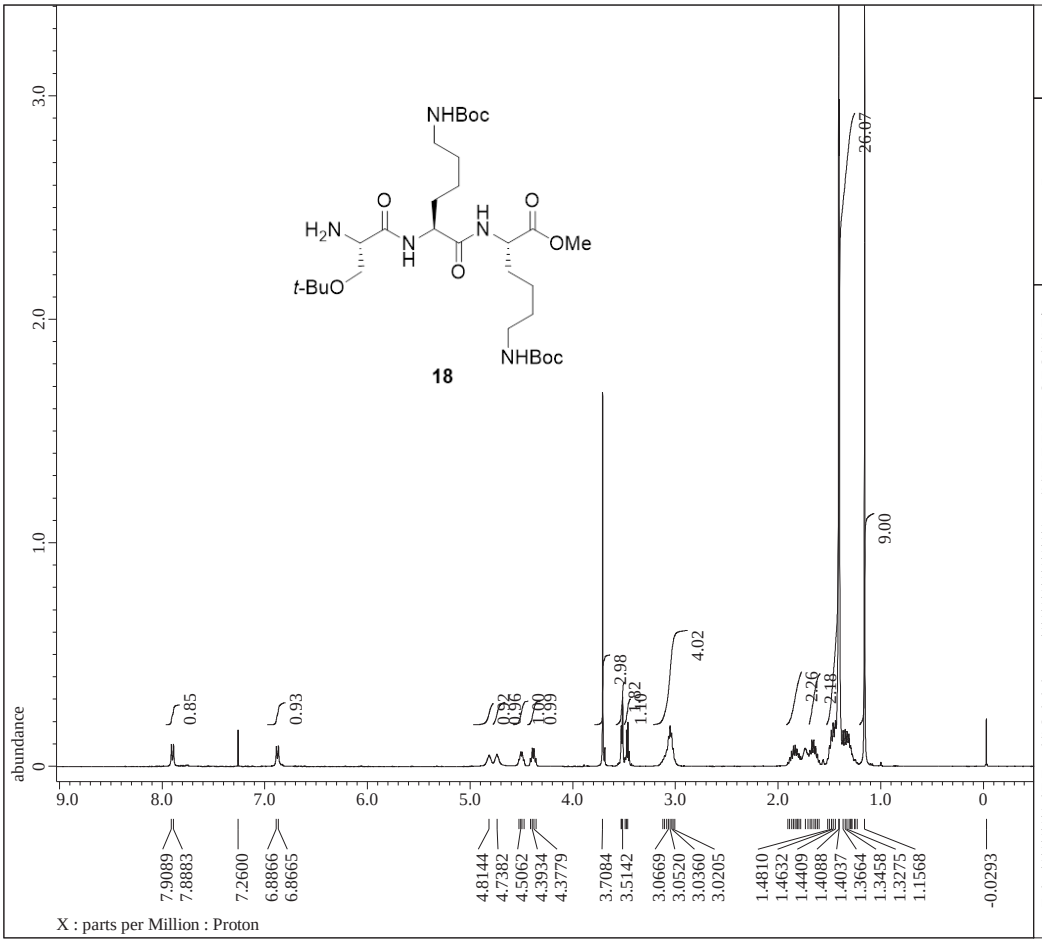
```
Filename          = wuan-666_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-666
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 18:25:41
Revision_Time     = 10-DEC-2021 18:28:35
Current_Time      = 10-DEC-2021 18:30:40

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        = 22
Temp_Get          = 23.4[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
```



JEOL RESONANCE

```
---- 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: wuan-666_E13C-1-1.jdf
```

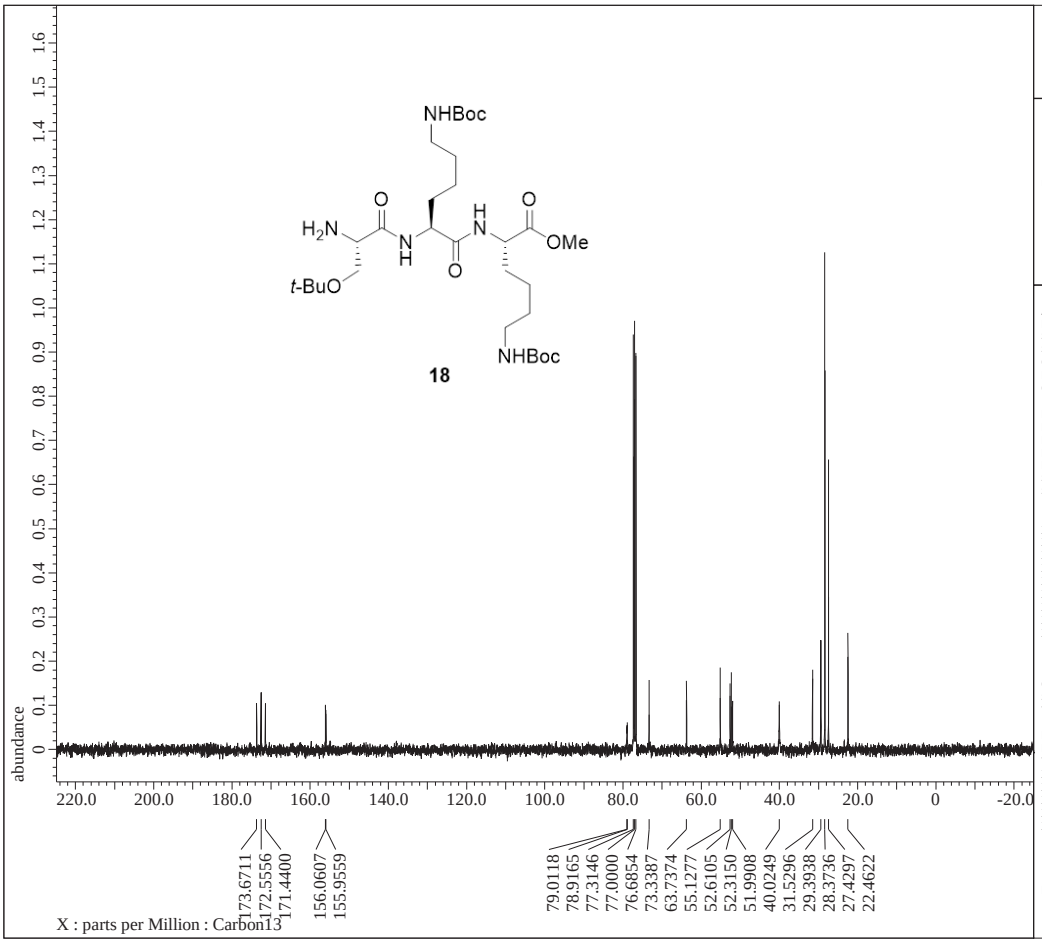
```
Filename          = wuan-666_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-666
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 18:30:22
Revision_Time     = 10-DEC-2021 18:35:41
Current_Time      = 10-DEC-2021 18:36:32

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           = FALSE
Scans             = 120
Total_Scans       = 120

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]
```



JEOL RESONANCE

```
---- 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: wuan-674_E1H-1-1.jdf
```

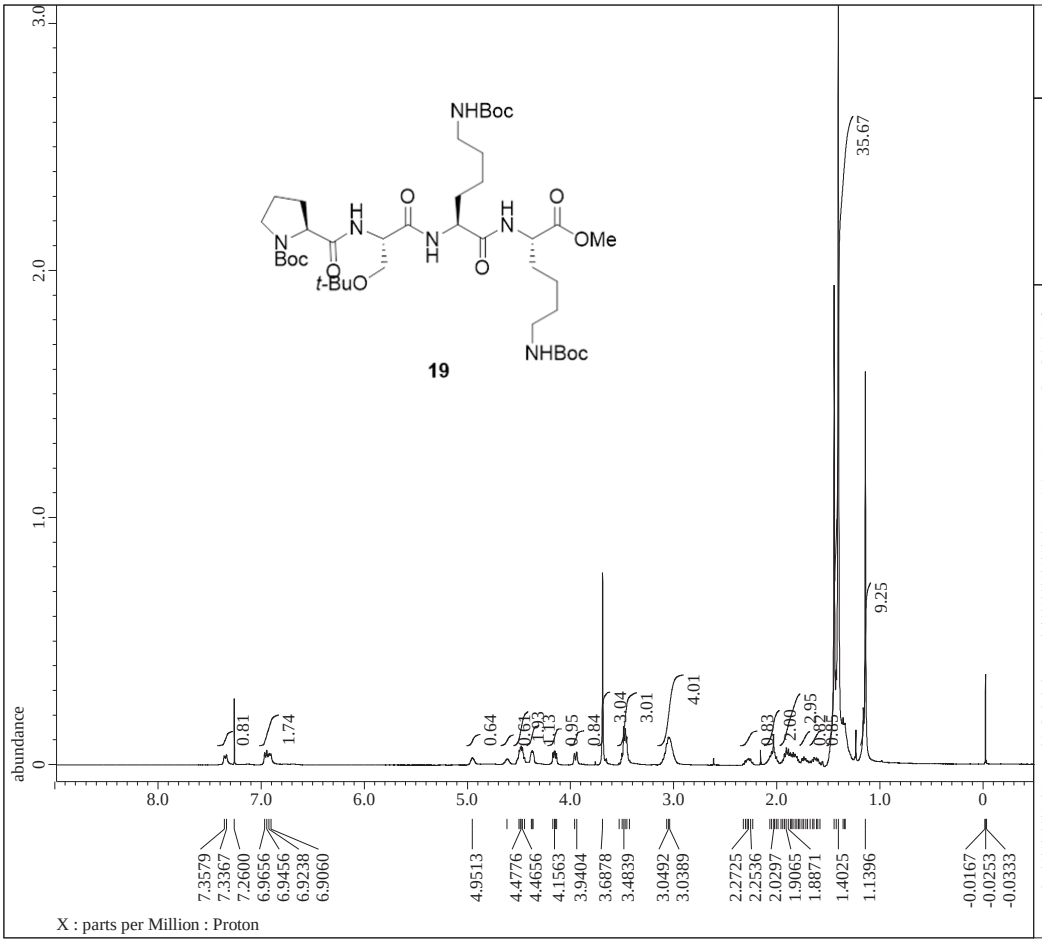
```
Filename          = wuan-674_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-674
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 18:43:16
Revision_Time     = 10-DEC-2021 18:48:13
Current_Time      = 10-DEC-2021 18:48:48

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        = 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
```



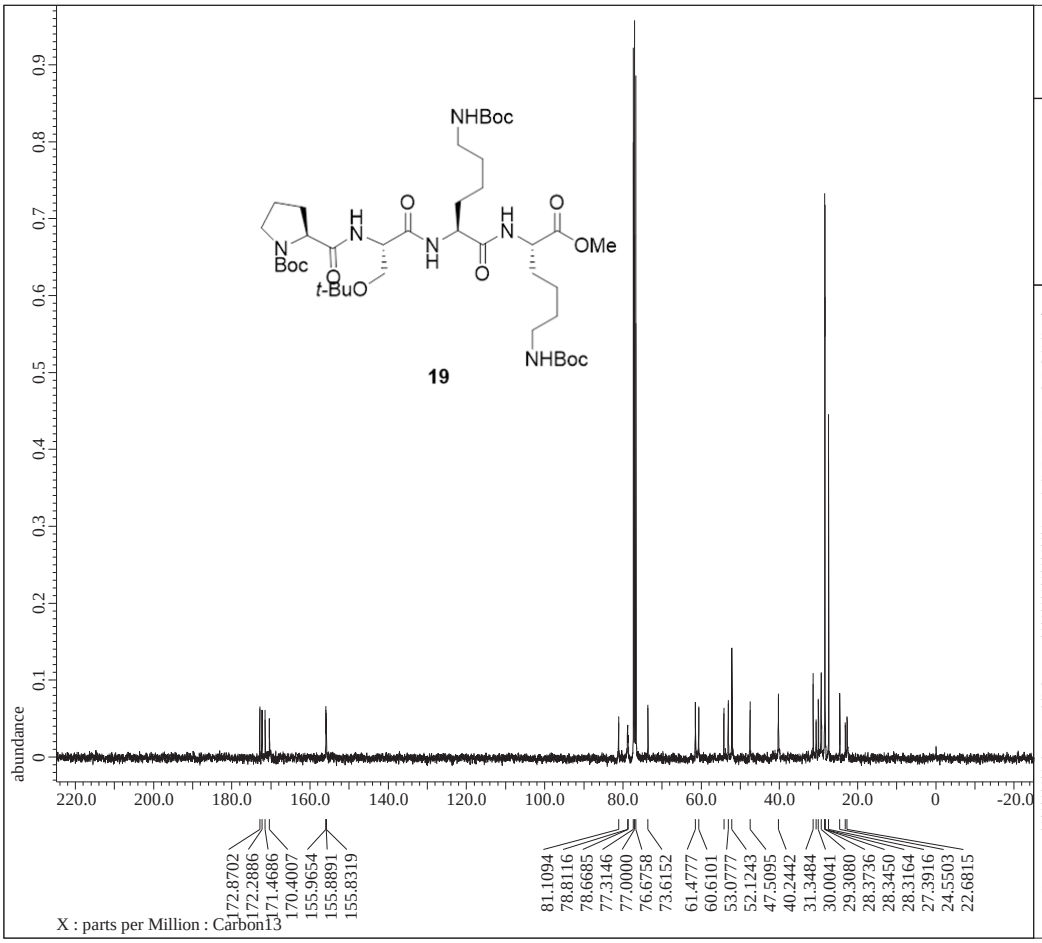
JEOL RESONANCE

```
---- 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: wuan-674_E13C-1-1.jdf
```

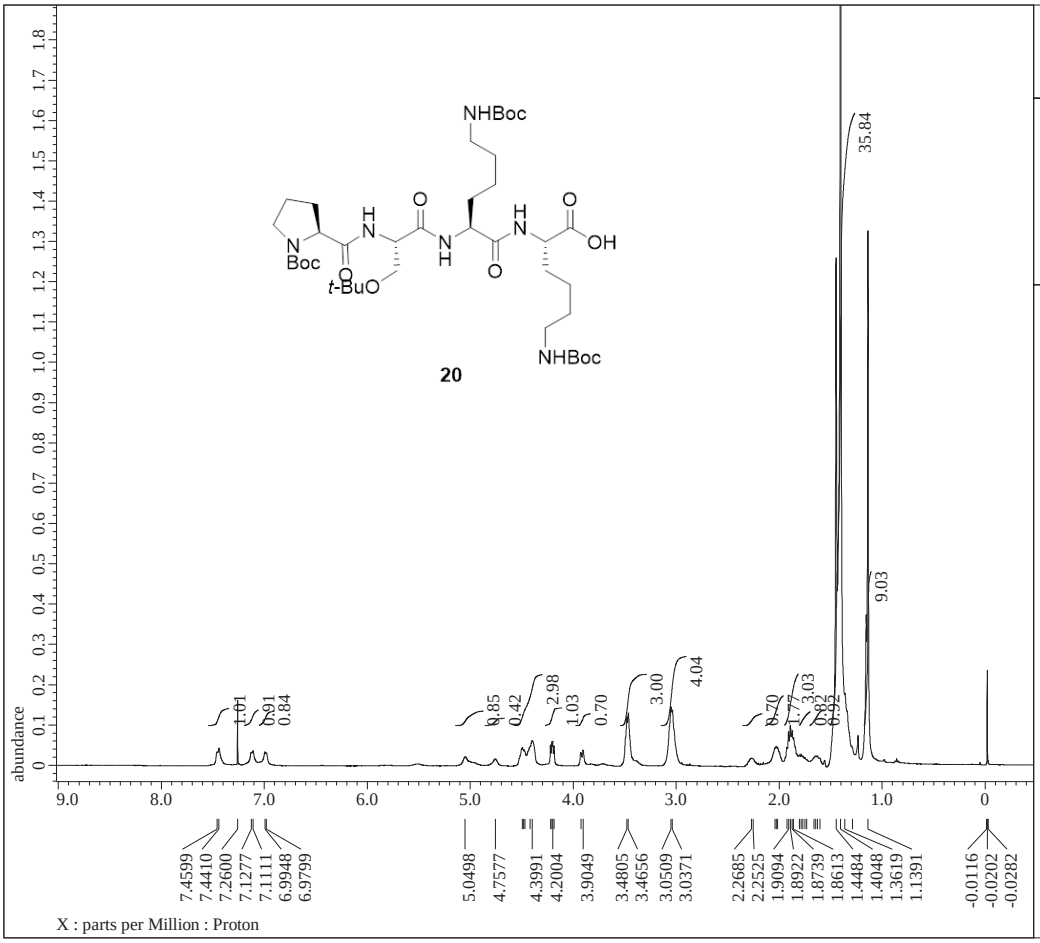
```
Filename          = wuan-674_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-674
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 18:47:54
Revision_Time     = 10-DEC-2021 18:52:20
Current_Time      = 10-DEC-2021 18:56:23

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    = 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             = 120
Total_Scans       = 120

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.6[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]
```



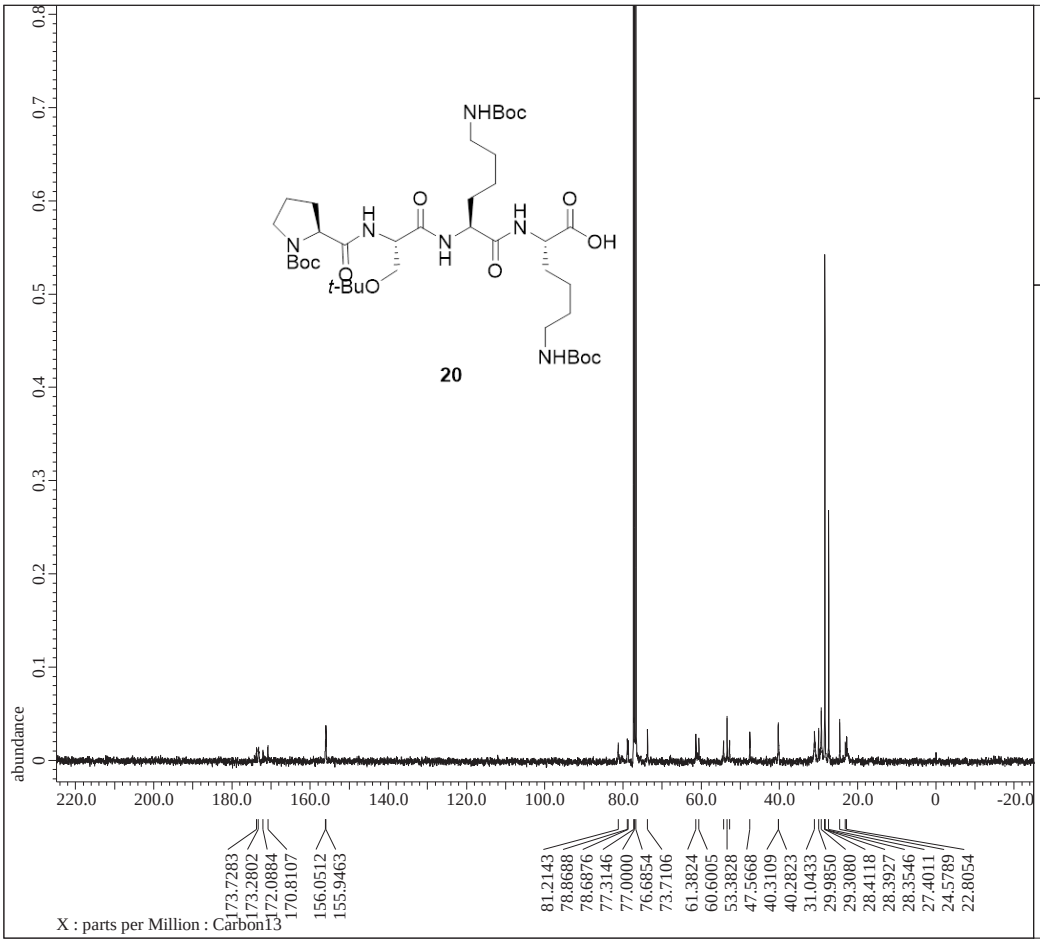

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = wuan-683_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-683
Solvent           = CHLOROFORM-D
Creation_Time     = 18-NOV-2021 14:32:22
Revision_Time     = 10-DEC-2021 21:03:27
Current_Time      = 10-DEC-2021 21:04:10

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        = 28
Temp_Get          = 23.2[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
```



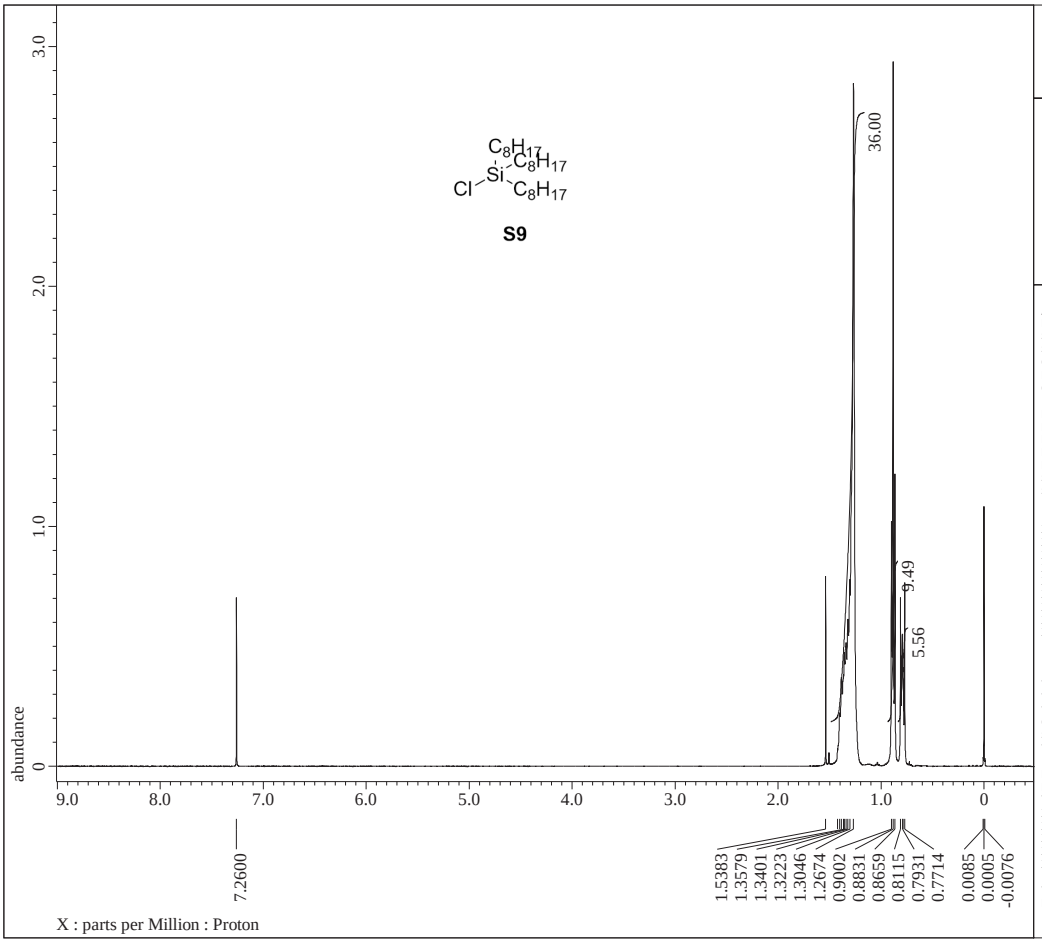

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-683_E13C-1-1.jdf

Filename          = wuan-683_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-683
Solvent           = CHLOROFORM-D
Creation_Time     = 18-NOV-2021 12:34:53
Revision_Time     = 18-NOV-2021 12:59:29
Current_Time      = 18-NOV-2021 13:00:59

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             = 560
Total_Scans       = 560

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]
```




```
---- 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: wuan-1217(crude)_E1H-1-1.jdf

Filename          = wuan-1217(crude)_E1H-1-4.j
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1217(crude)
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 20:52:05
Revision_Time     = 10-DEC-2021 20:59:49
Current_Time      = 10-DEC-2021 21:00:37

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        = 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
```



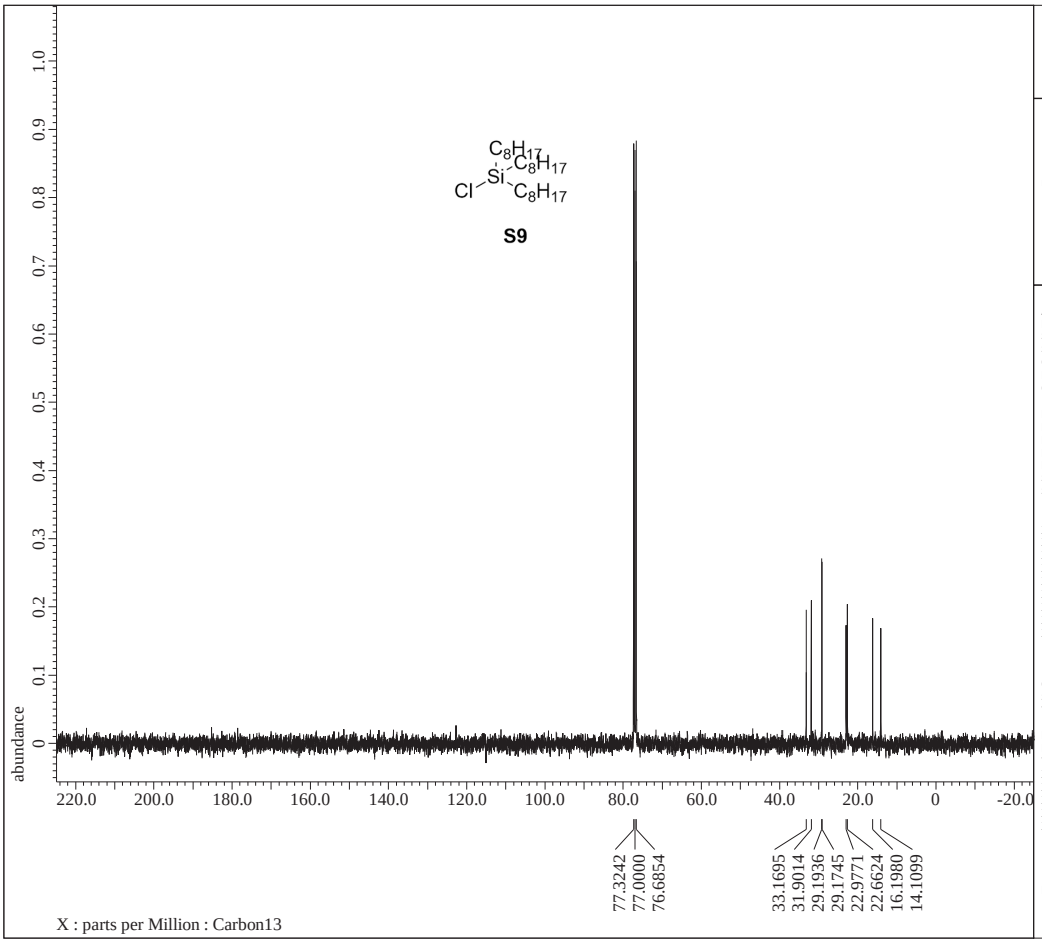

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = wuan-1217(crude)_E13C-1-5.
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1217(crude)
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2021 20:56:46
Revision_Time     = 10-DEC-2021 22:21:51
Current_Time      = 10-DEC-2021 22:22:26

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             = 1280
Total_Scans       = 1280

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]
```



JEOL RESONANCE

```
---- 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: wuan-1097_E1H-1-1.jdf
```

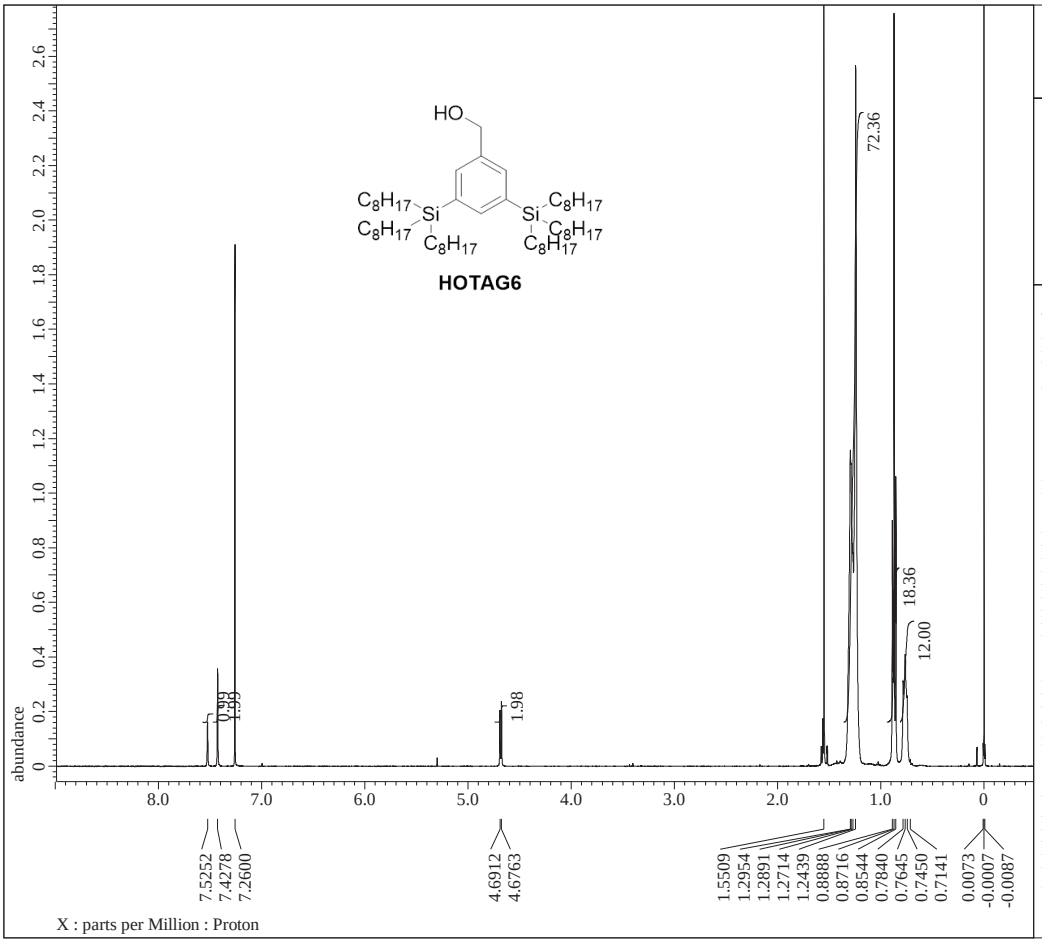
```
Filename          = wuan-1097_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1097
Solvent           = CHLOROFORM-D
Creation_Time     = 26-JUL-2021 18:50:00
Revision_Time     = 26-JUL-2021 18:52:25
Current_Time      = 26-JUL-2021 18:53:05

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        = 34
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
```



JEOL RESONANCE

```
---- 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: wuan-1097_E13C-1-1.jdf
```

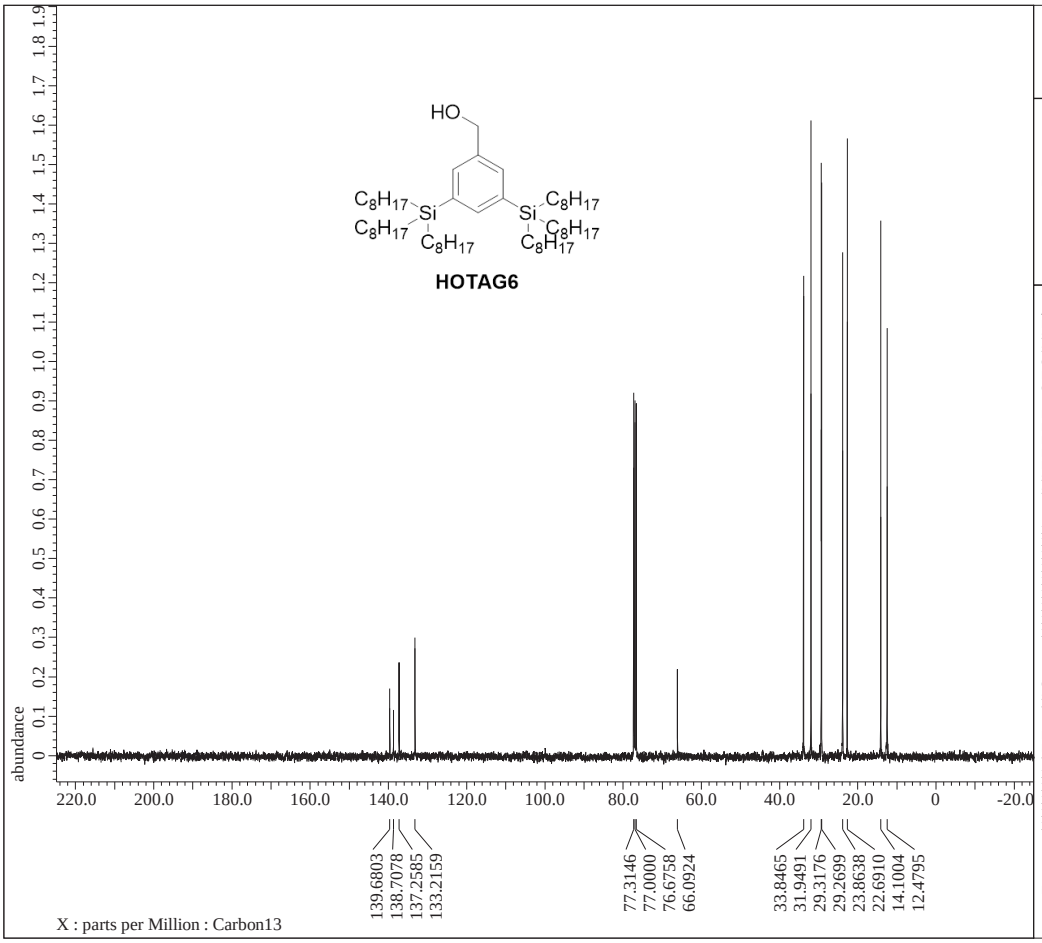
```
Filename          = wuan-1097_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1097
Solvent           = CHLOROFORM-D
Creation_Time     = 26-JUL-2021 18:51:58
Revision_Time     = 26-JUL-2021 18:54:34
Current_Time      = 26-JUL-2021 18:54:52

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           = FALSE
Scans             = 105
Total_Scans       = 105

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]
```



JEOL RESONANCE

```
---- 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: wuan-1047_E1H-2-1.jdf
```

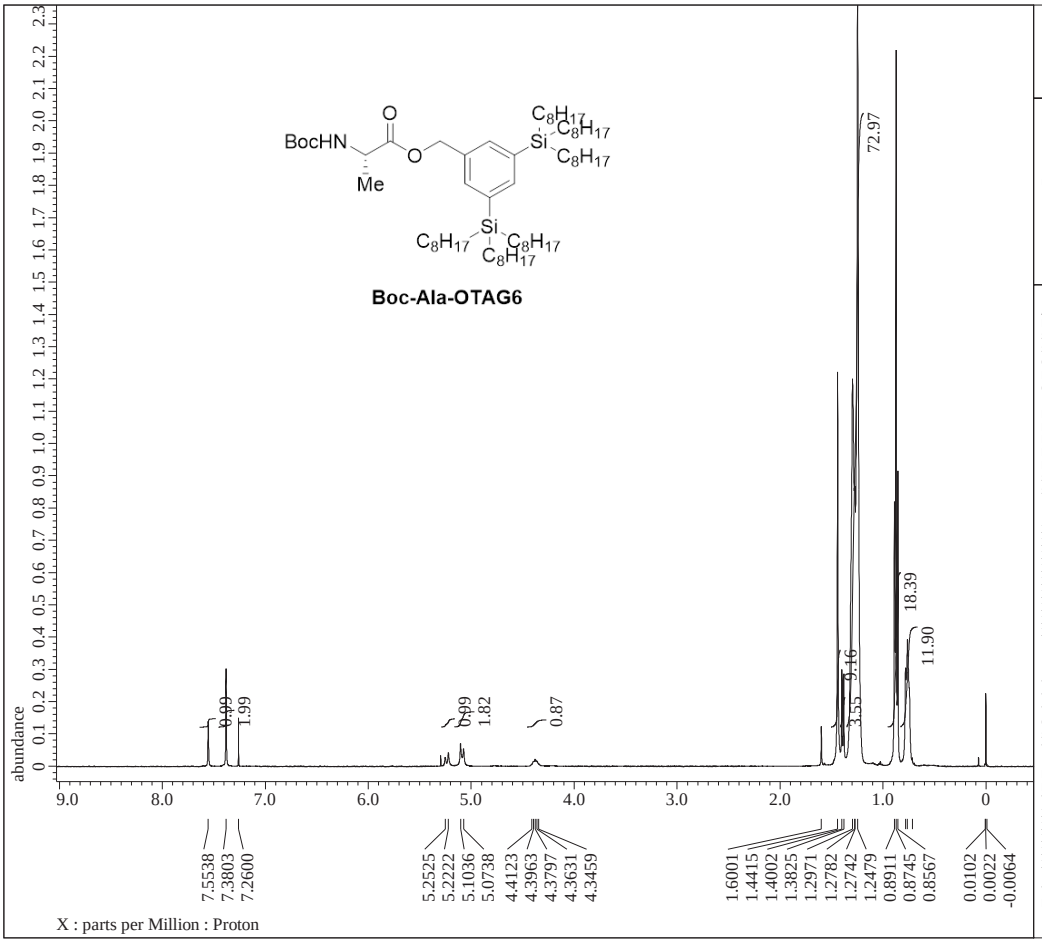
```
Filename          = wuan-1047_E1H-2-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1047
Solvent           = CHLOROFORM-D
Creation_Time     = 19-JUN-2021 13:37:47
Revision_Time     = 19-JUN-2021 13:42:08
Current_Time      = 19-JUN-2021 13:42:22

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        = 42
Temp_Get          = 22.3[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
```



JEOL RESONANCE

```
---- 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: wuan-1047_E13C-1-1.jdf
```

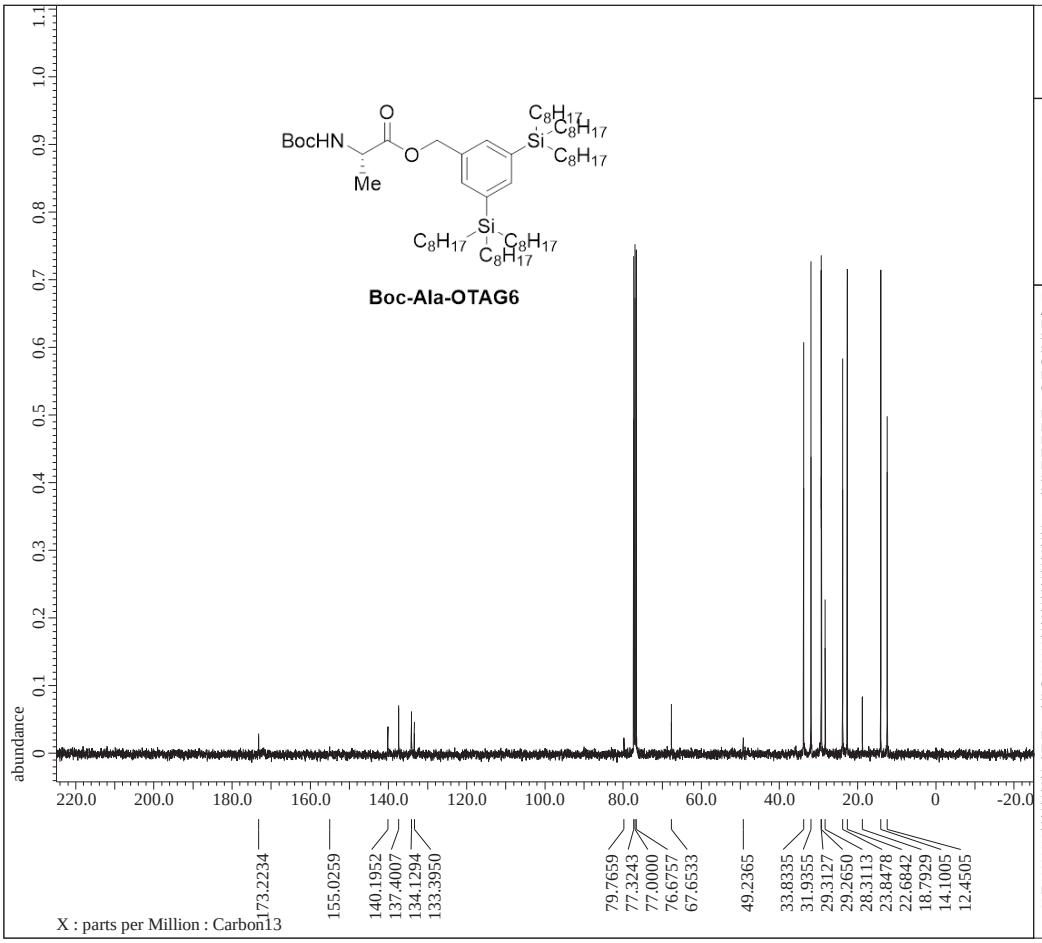
```
Filename          = wuan-1047_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1047
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JUN-2021 10:34:32
Revision_Time     = 21-JUN-2021 10:39:58
Current_Time      = 21-JUN-2021 10:40:11

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             = 157
Total_Scans       = 157

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]
```



JEOL RESONANCE

```
---- 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: wuan-1065_E1H-1-1.jdf
```

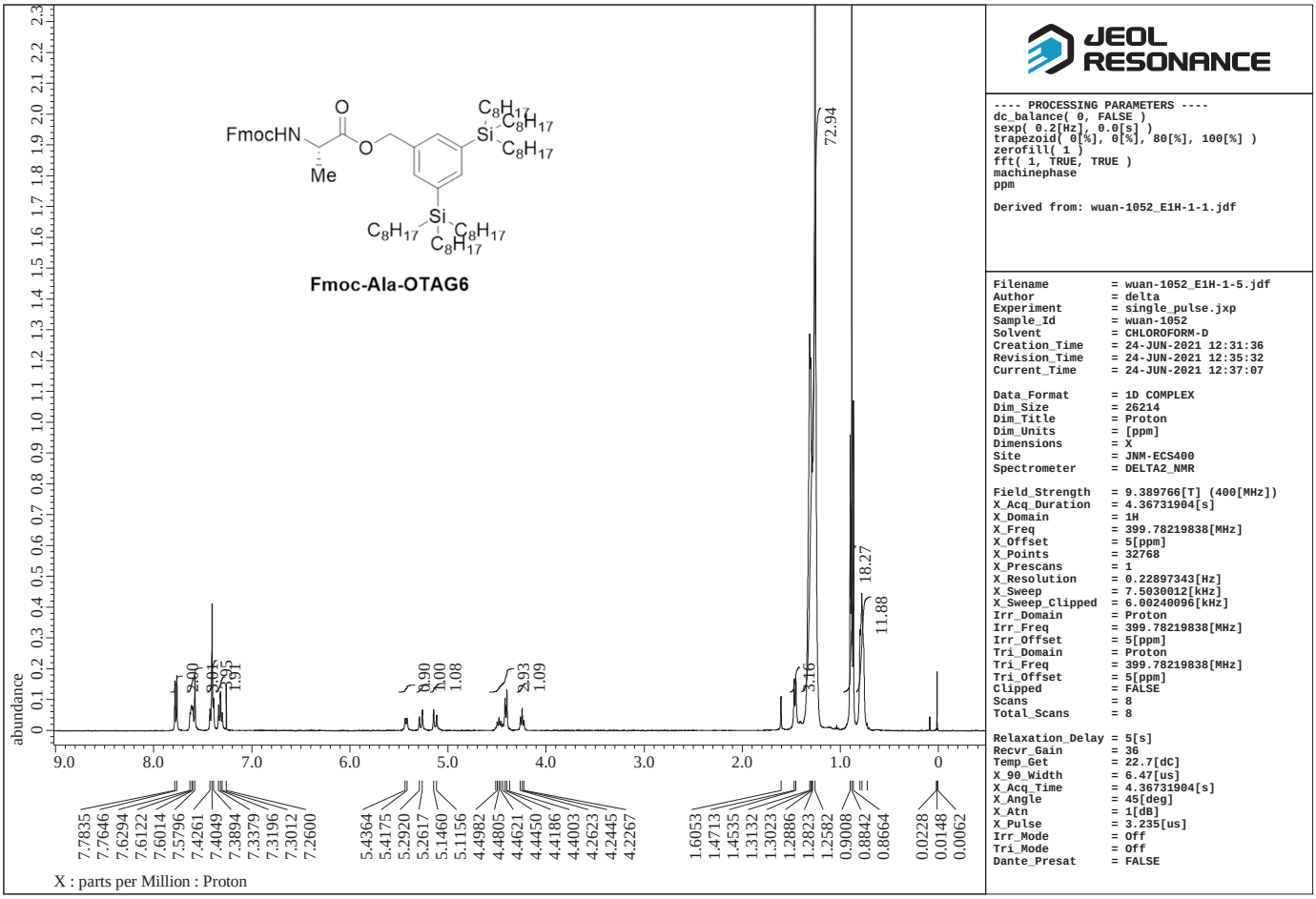
```
Filename          = wuan-1065_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1065
Solvent           = CHLOROFORM-D
Creation_Time     =  2-JUL-2021 10:51:23
Revision_Time     =  2-JUL-2021 11:00:50
Current_Time      =  2-JUL-2021 11:01:28

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        = 32
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
```



JEOL RESONANCE

```
---- 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
```

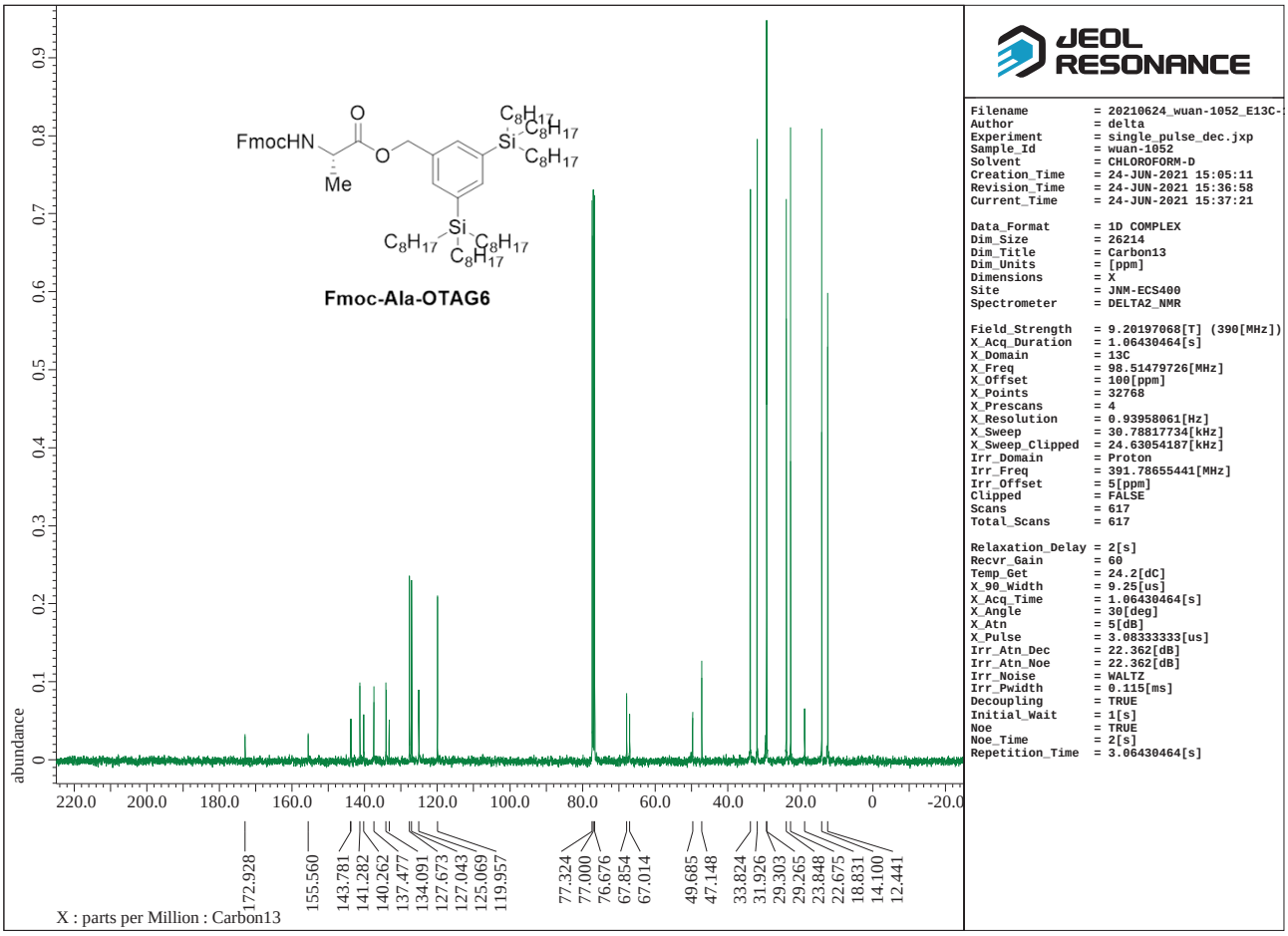
```
Filename          = wuan-1065_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1065
Solvent           = CHLOROFORM-D
Creation_Time     =  2-JUL-2021 10:59:30
Revision_Time     = 29-NOV-2021 17:33:23
Current_Time      = 29-NOV-2021 17:34:09

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 351
Total_Scans       = 351

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.6[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



JEOL RESONANCE

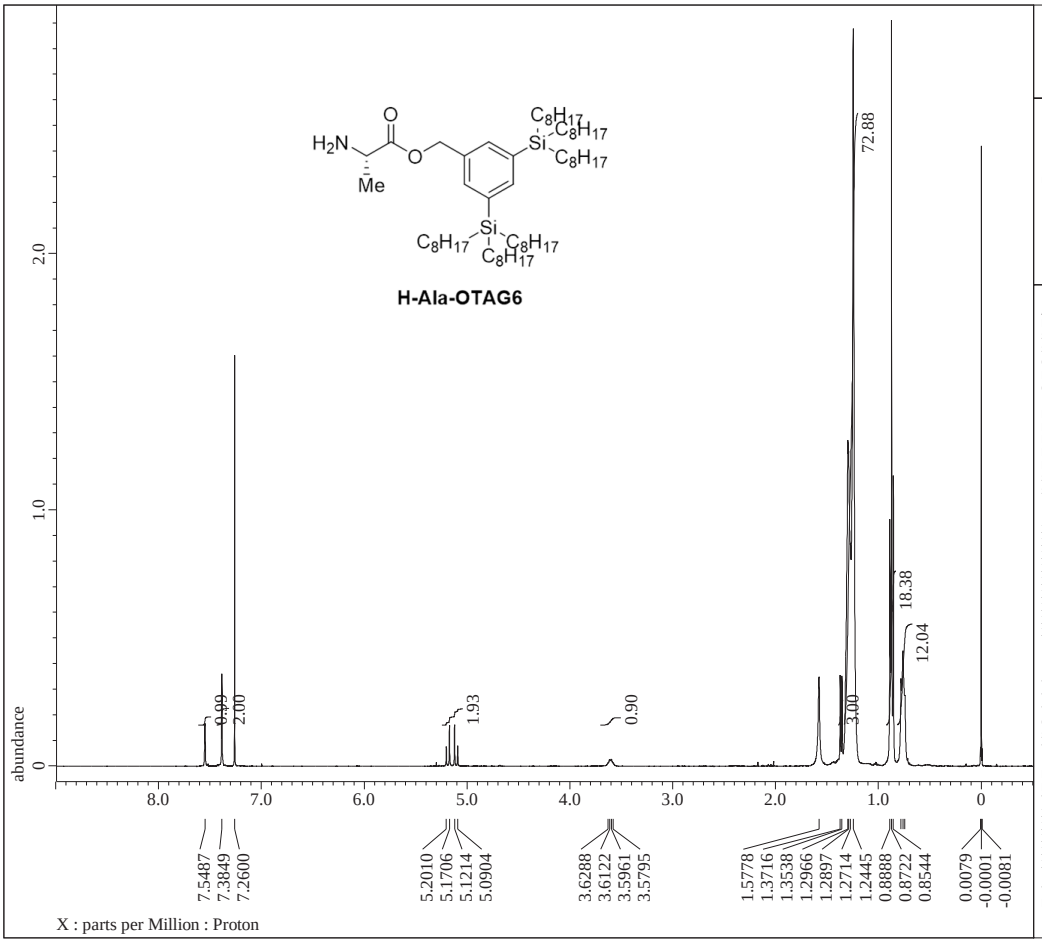
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-1052_E1H-1-1.jdf
```

```
Filename          = wuan-1052_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1052
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JUN-2021 12:31:36
Revision_Time     = 24-JUN-2021 12:35:32
Current_Time      = 24-JUN-2021 12:37:07

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          = 22.7[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
```



JEOL RESONANCE

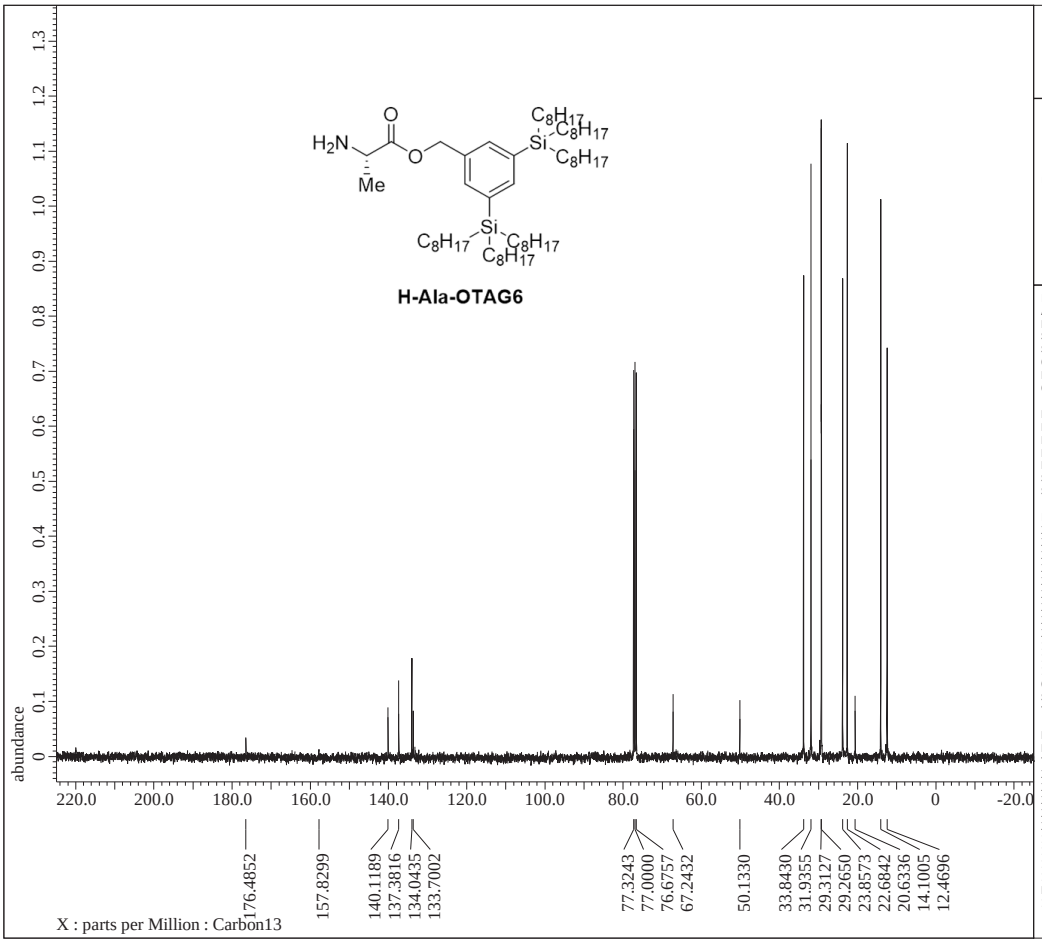
```
Filename          = 20210624_wuan-1052_E13C-1
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1052
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JUN-2021 15:05:11
Revision_Time     = 24-JUN-2021 15:36:58
Current_Time      = 24-JUN-2021 15:37:21

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 617
Total_Scans       = 617

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.2[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.06430464[s]
```



JEOL RESONANCE

```
---- 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
```

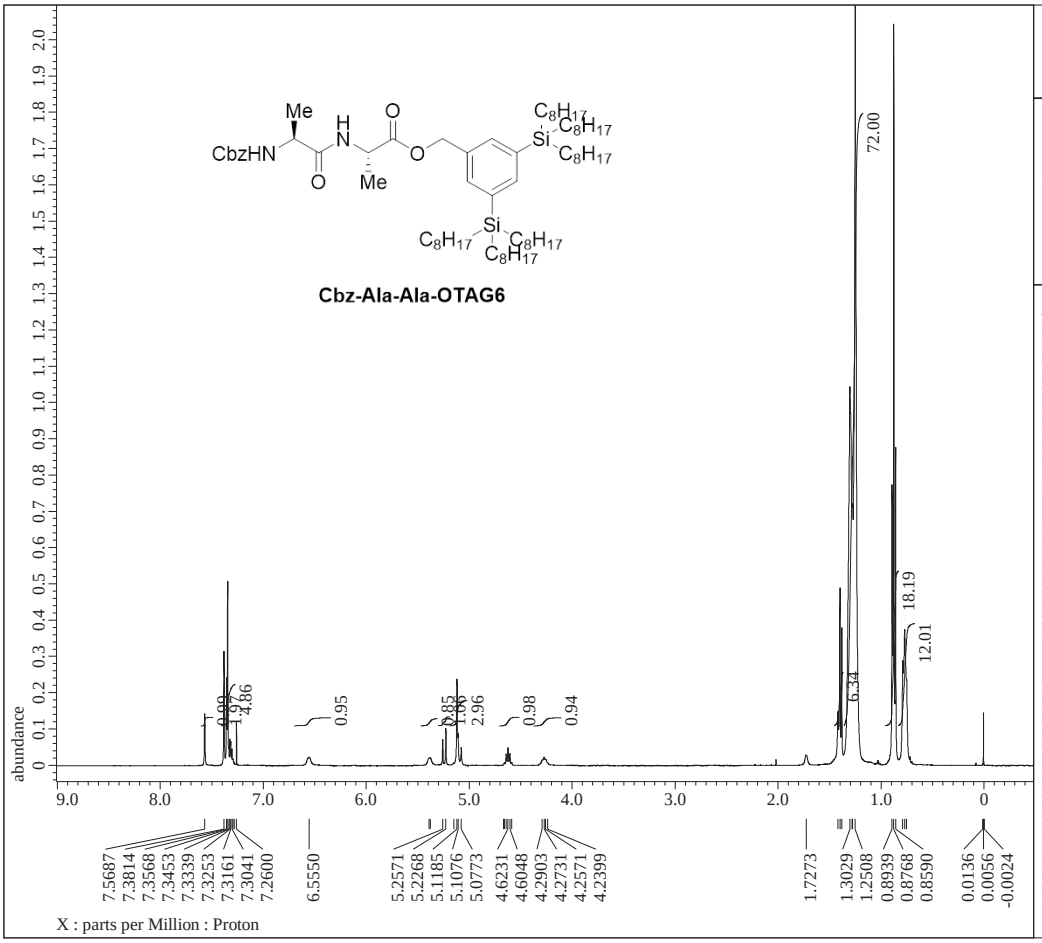
```
Filename          = wuan-1071_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1071
Solvent           = CHLOROFORM-D
Creation_Time     =  6-JUL-2021 15:43:26
Revision_Time     = 29-NOV-2021 17:36:14
Current_Time      = 29-NOV-2021 17:36:47

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        = 38
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
```



JEOL RESONANCE

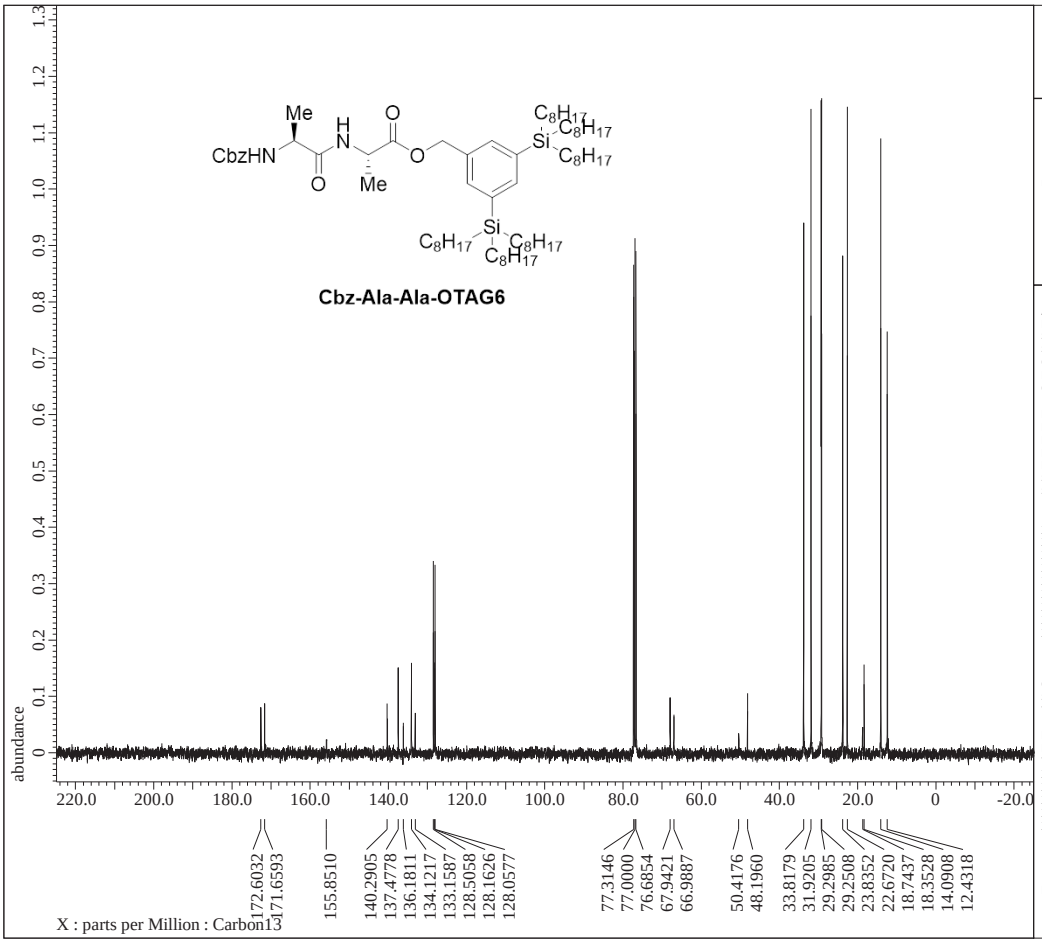
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1054_E13C-1-6.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1054
Solvent           = CHLOROFORM-D
Creation_Time     = 25-JUN-2021 12:08:22
Revision_Time     = 29-NOV-2021 17:38:26
Current_Time      = 29-NOV-2021 17:39:09

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 236
Total_Scans       = 236

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.7[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



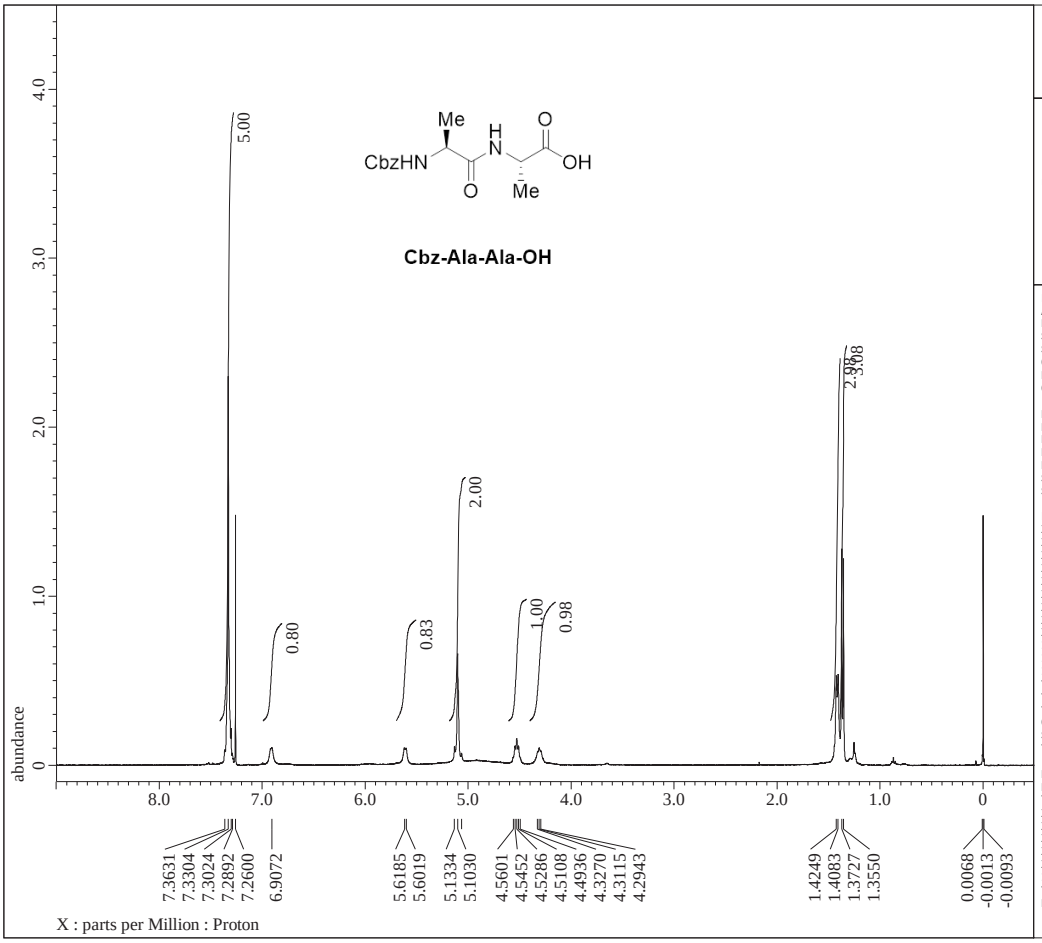

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-1072_E1H-1-1.jdf
```

```
Filename          = wuan-1072_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1072
Solvent           = CHLOROFORM-D
Creation_Time     =  7-JUL-2021 13:23:50
Revision_Time     =  7-JUL-2021 13:28:50
Current_Time      =  7-JUL-2021 13:29:12

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        = 18
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: wuan-1072_E13C-1-1.jdf
```

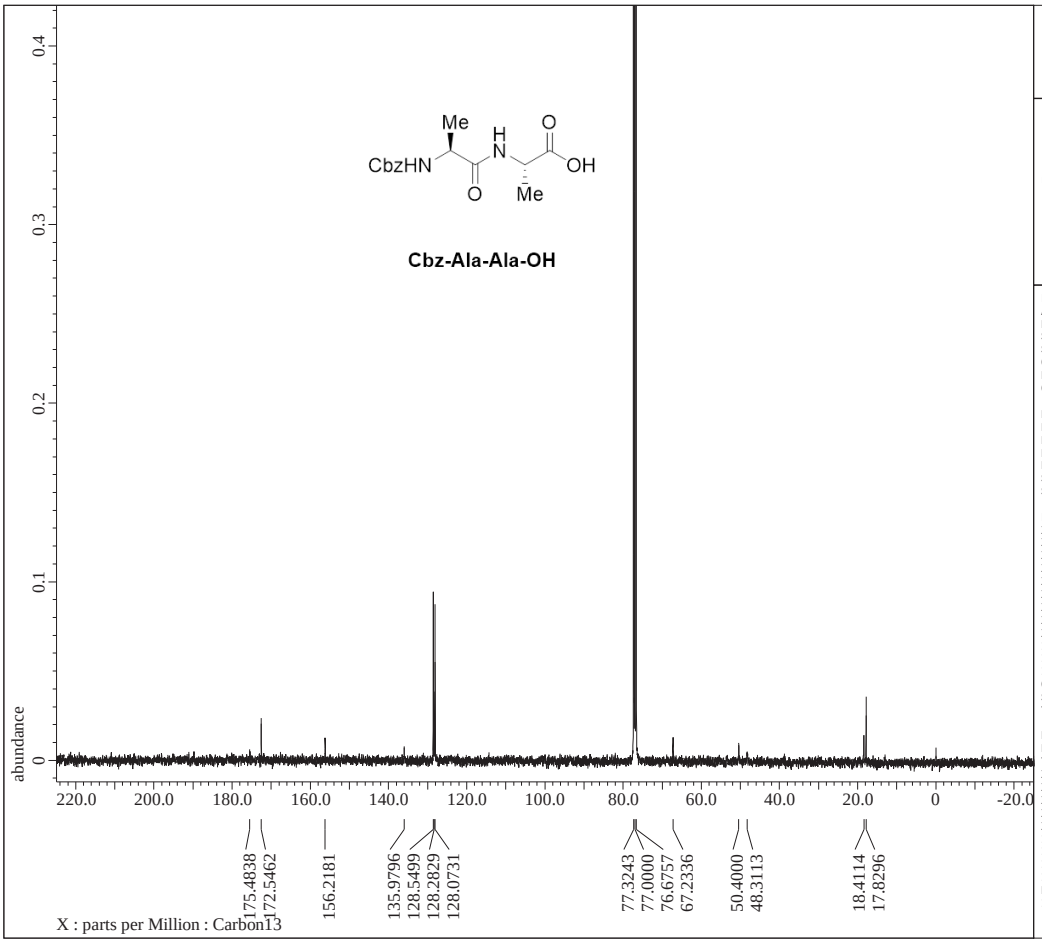
```
Filename          = wuan-1072_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1072
Solvent           = CHLOROFORM-D
Creation_Time     =  7-JUL-2021 13:25:40
Revision_Time     =  7-JUL-2021 15:35:01
Current_Time      =  7-JUL-2021 15:37:00

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             = 177
Total_Scans       = 177

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]
```



JEOL RESONANCE

```
---- 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: wuan-1075-1_E1H-1-1.jdf
```

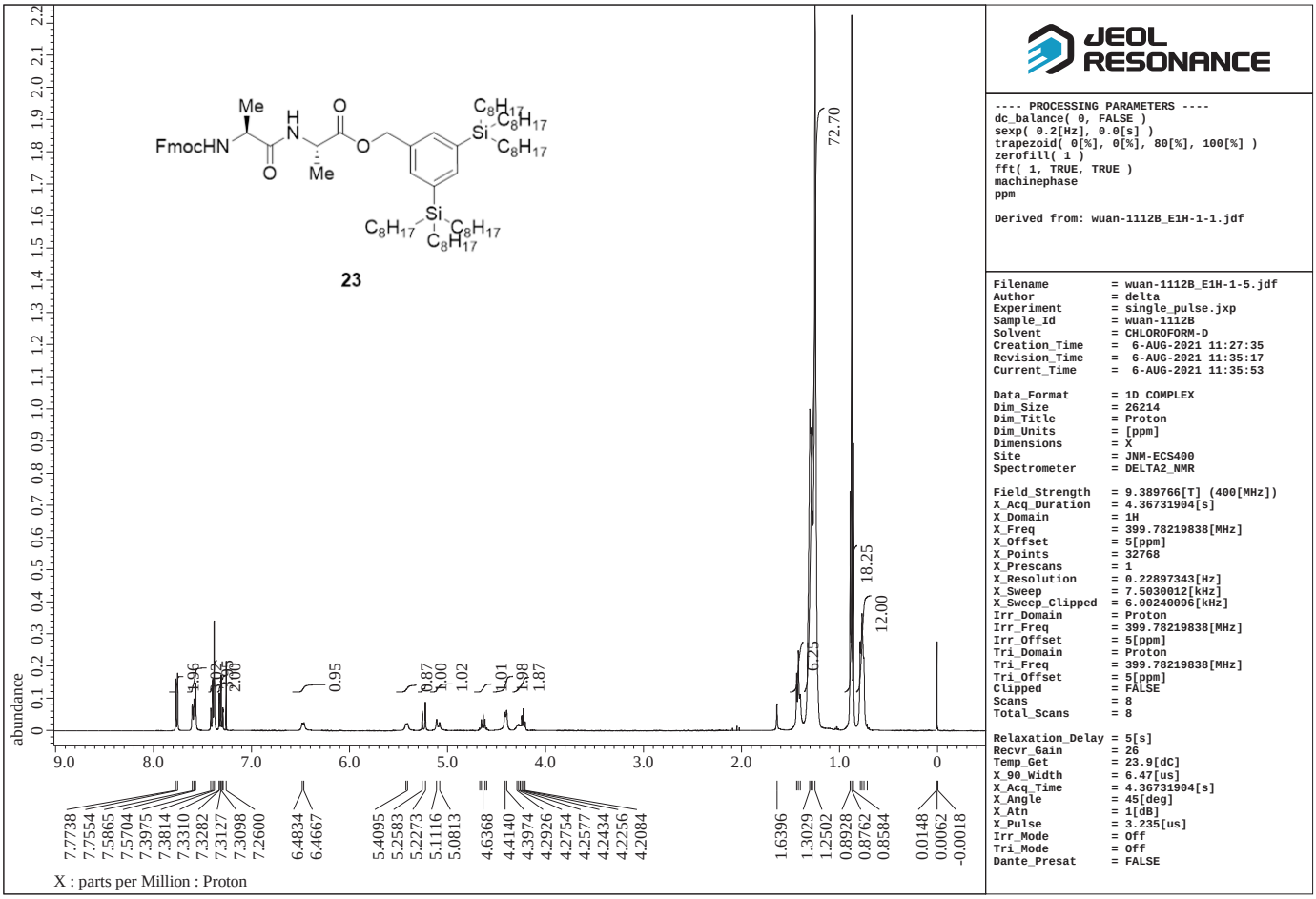
```
Filename          = wuan-1075-1_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1075-1
Solvent           = CHLOROFORM-D
Creation_Time     =  9-JUL-2021 14:52:50
Revision_Time     =  9-JUL-2021 14:55:05
Current_Time      =  9-JUL-2021 14:56:42

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        = 50
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
```



JEOL RESONANCE

```
---- 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
```

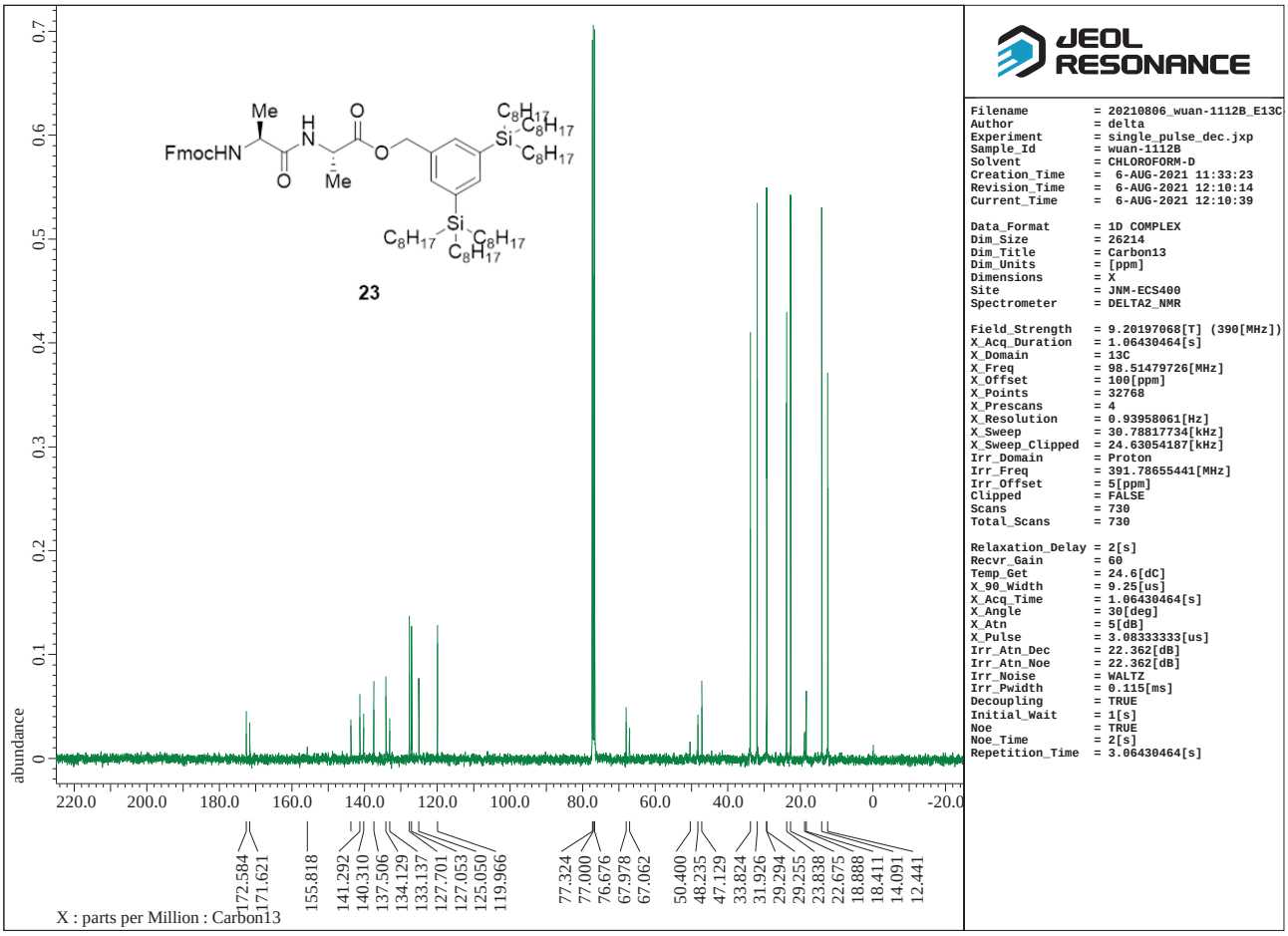
```
Filename          = wuan-1075-1_E13C-1-7.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1075-1
Solvent           = CHLOROFORM-D
Creation_Time     =  9-JUL-2021 18:03:35
Revision_Time     = 29-NOV-2021 17:40:58
Current_Time      = 29-NOV-2021 17:41:35

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2406
Total_Scans       = 2406

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.6[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```



JEOL RESONANCE

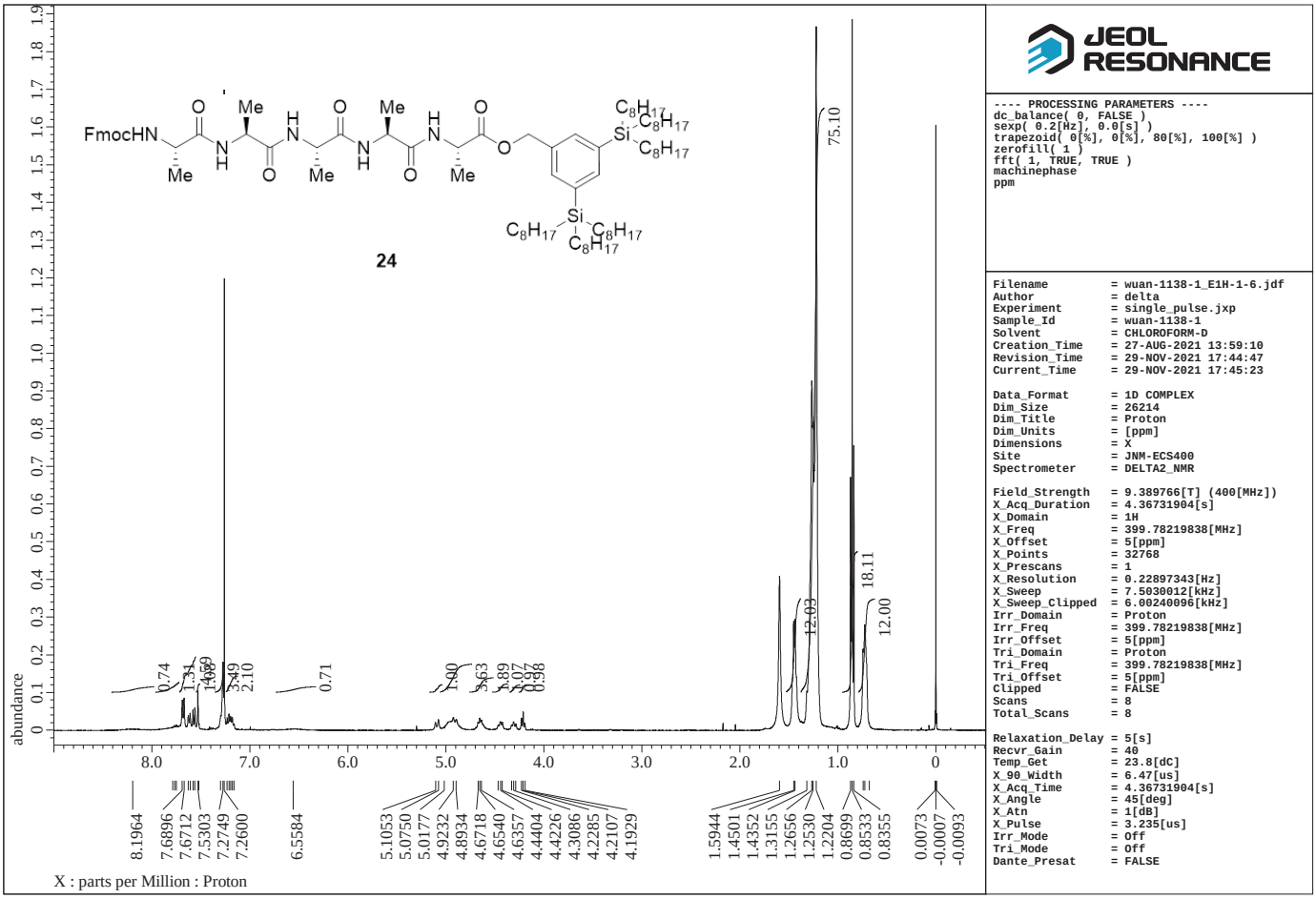
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-1112B_E1H-1-1.jdf
```

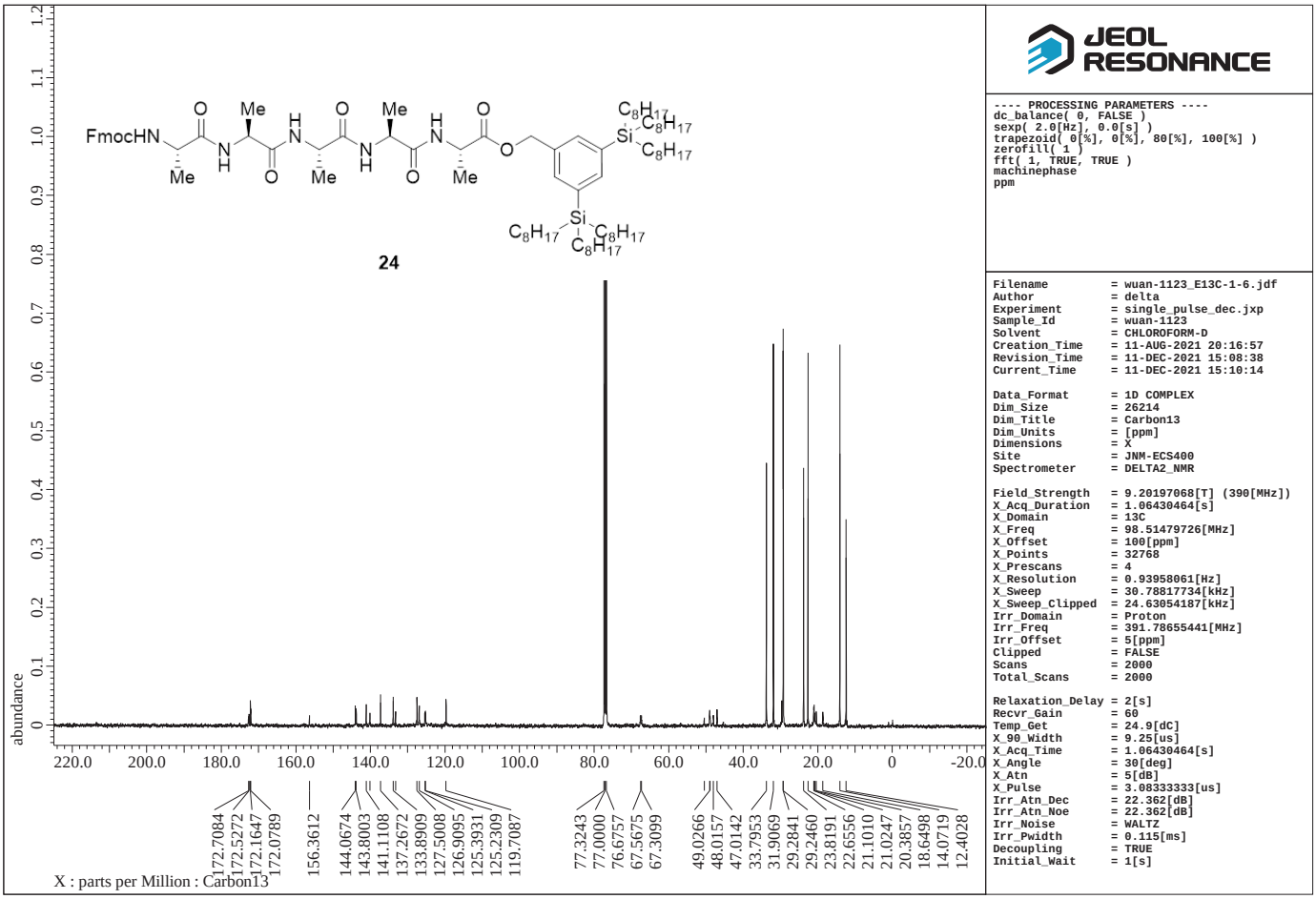
```
Filename          = wuan-1112B_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1112B
Solvent           = CHLOROFORM-D
Creation_Time     =  6-AUG-2021 11:27:35
Revision_Time     =  6-AUG-2021 11:35:17
Current_Time      =  6-AUG-2021 11:35:53

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        = 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
```

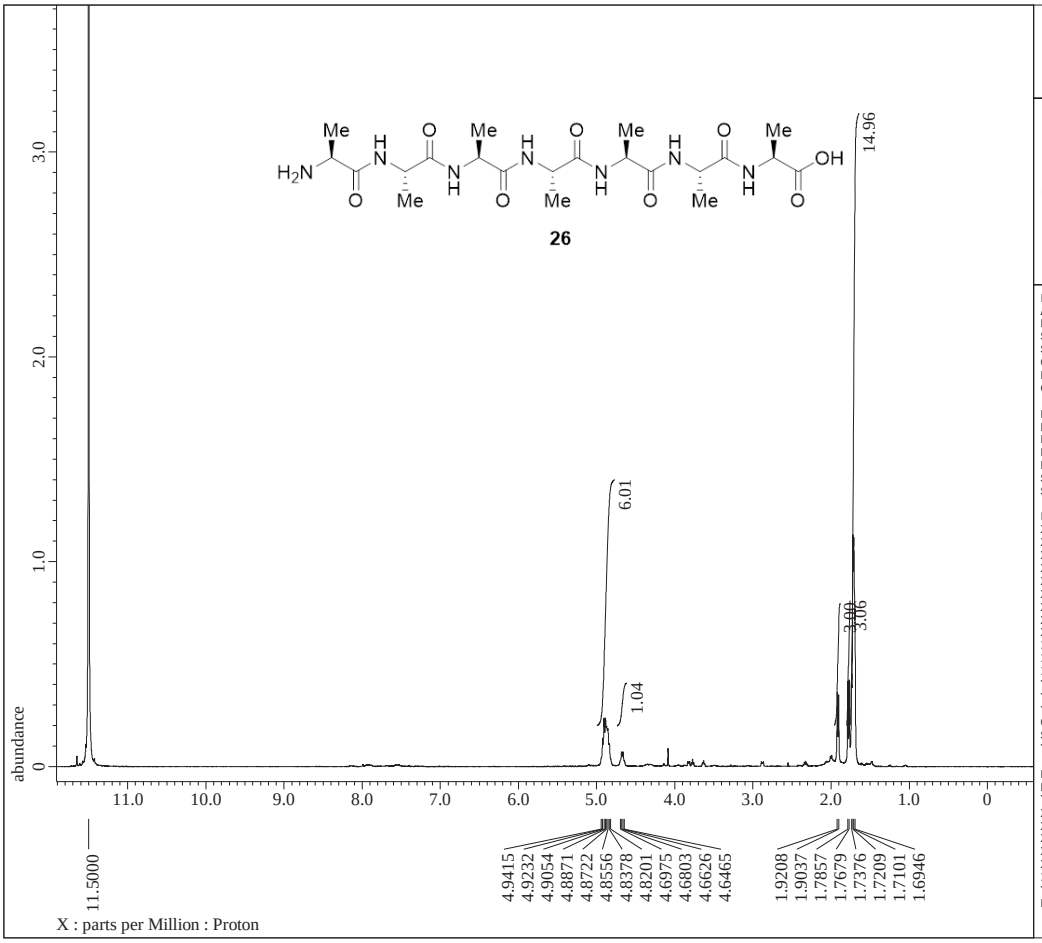


JEOL RESONANCE

```
Filename          = 20210806_wuan-1112B_E13C
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1112B
Solvent           = CHLOROFORM-D
Creation_Time     =  6-AUG-2021 11:33:23
Revision_Time     =  6-AUG-2021 12:10:14
Current_Time      =  6-AUG-2021 12:10:39

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 730
Total_Scans       = 730

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.6[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.06430464[s]
```


FmocHN
24
abundance
X : parts per Million : Proton
---- 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
Filename = wuan-1138-1_E1H-1-6.jdf
Author = delta
Experiment = single_pulse.jxp
Sample_Id = wuan-1138-1
Solvent = CHLOROFORM-D
Creation_Time = 27-AUG-2021 13:59:10
Revision_Time = 29-NOV-2021 17:44:47
Current_Time = 29-NOV-2021 17:45:23
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 = 40
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

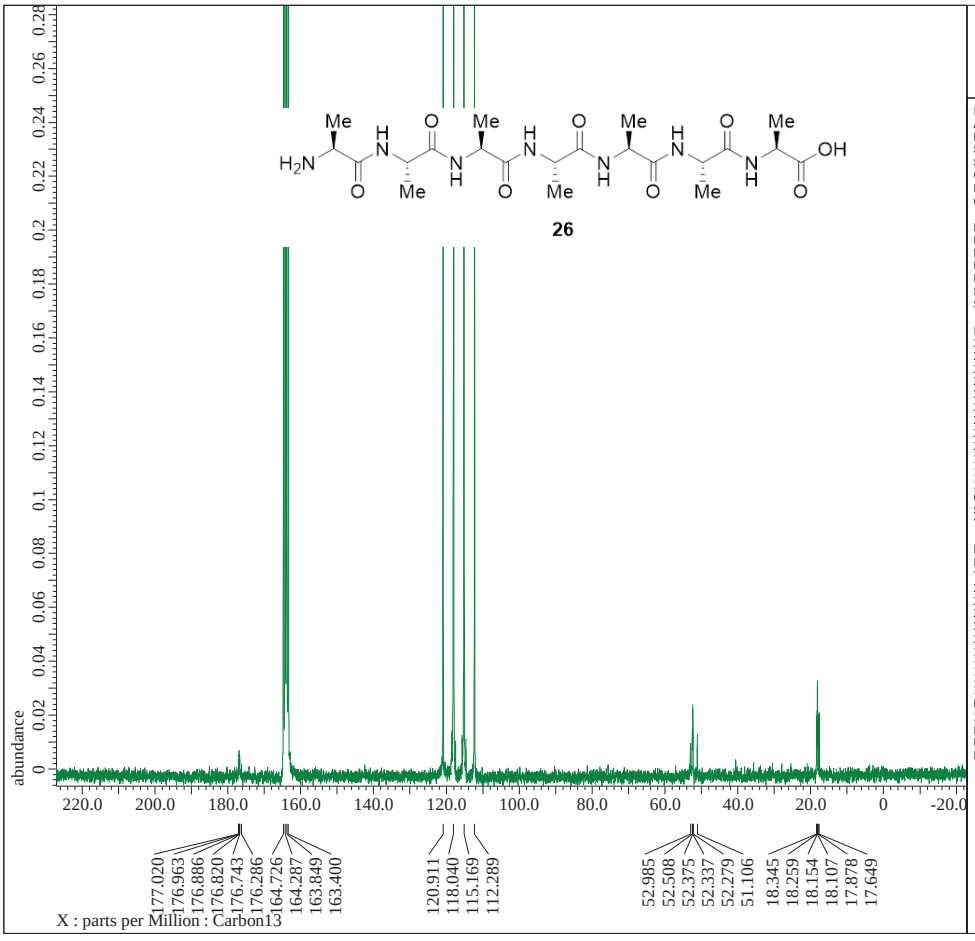

FmocHN
24
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = wuan-1123_E13C-1-6.jdf
Author = delta
Experiment = single_pulse_dec.jxp
Sample_Id = wuan-1123
Solvent = CHLOROFORM-D
Creation_Time = 11-AUG-2021 20:16:57
Revision_Time = 11-DEC-2021 15:08:38
Current_Time = 11-DEC-2021 15:10:14
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 1.06430464[s]
X_Domain = 13C
X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
Irr_Domain = Proton
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 2000
Total_Scans = 2000
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.9[dC]
X_90_Width = 9.25[us]
X_Acq_Time = 1.06430464[s]
X_Angle = 30[deg]
X_Atn = 5[dB]
X_Pulse = 3.08333333[us]
Irr_Atn_Dec = 22.362[dB]
Irr_Atn_Noe = 22.362[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]



JEOL RESONANCE

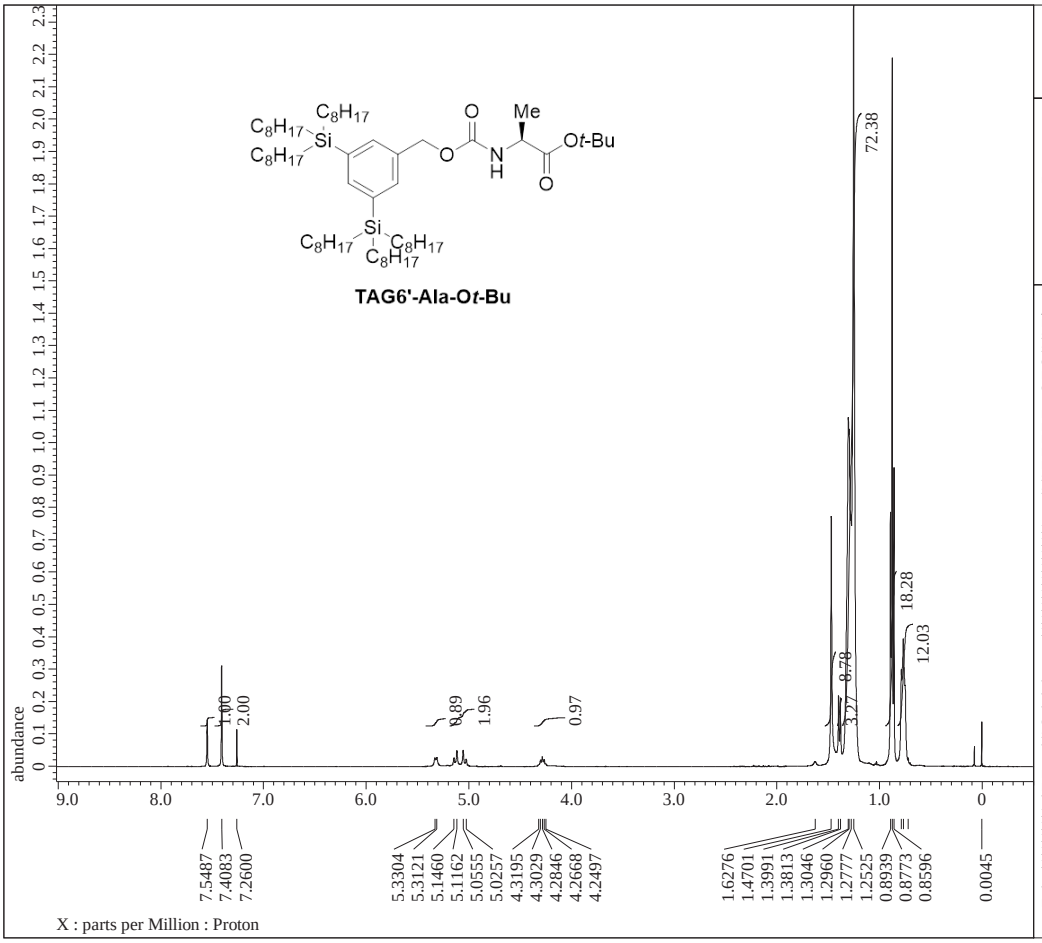
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-1148-3-TFA_E1H-2-1.jdf
```

```
Filename          = wuan-1148-3-TFA_E1H-2-4.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1148-3-TFA
Solvent           = TRIFLUOROACETIC_ACID-D
Creation_Time     = 10-SEP-2021 16:25:49
Revision_Time     = 10-SEP-2021 16:31:33
Current_Time      = 10-SEP-2021 16:34:39

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        = 40
Temp_Get          = 23.4[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
```



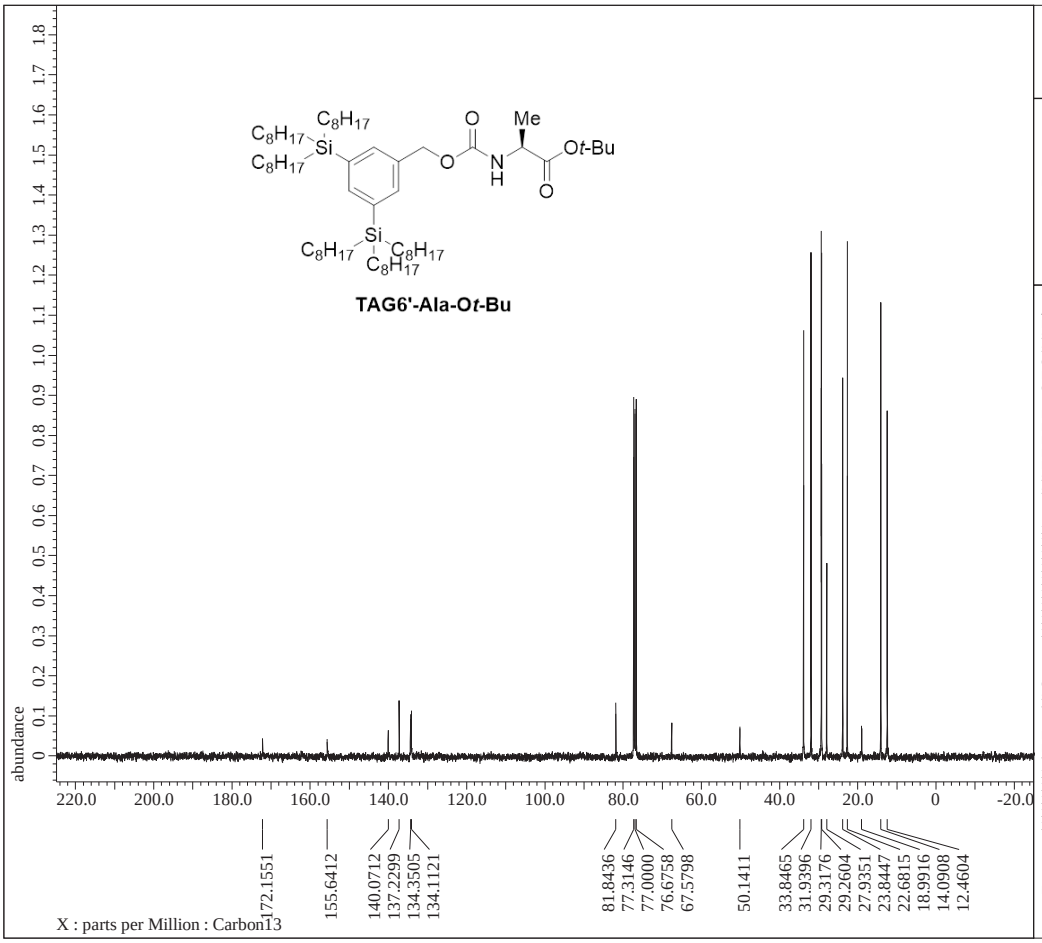
JEOL RESONANCE

```
Filename          = 20210911_wuan-1148-3-TFA
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1148-3-TFA
Solvent           = TRIFLUOROACETIC_ACID-D
Creation_Time     = 11-SEP-2021 09:20:23
Revision_Time     = 11-SEP-2021 11:59:46
Current_Time      = 11-SEP-2021 12:03:13

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

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
X_Sweep_Clipped   = 24.63054187[kHz]
Irr_Domain        = Proton
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 3056
Total_Scans       = 3056

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.9[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 5[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 22.362[dB]
Irr_Atn_Noe       = 22.362[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.06430464[s]
```



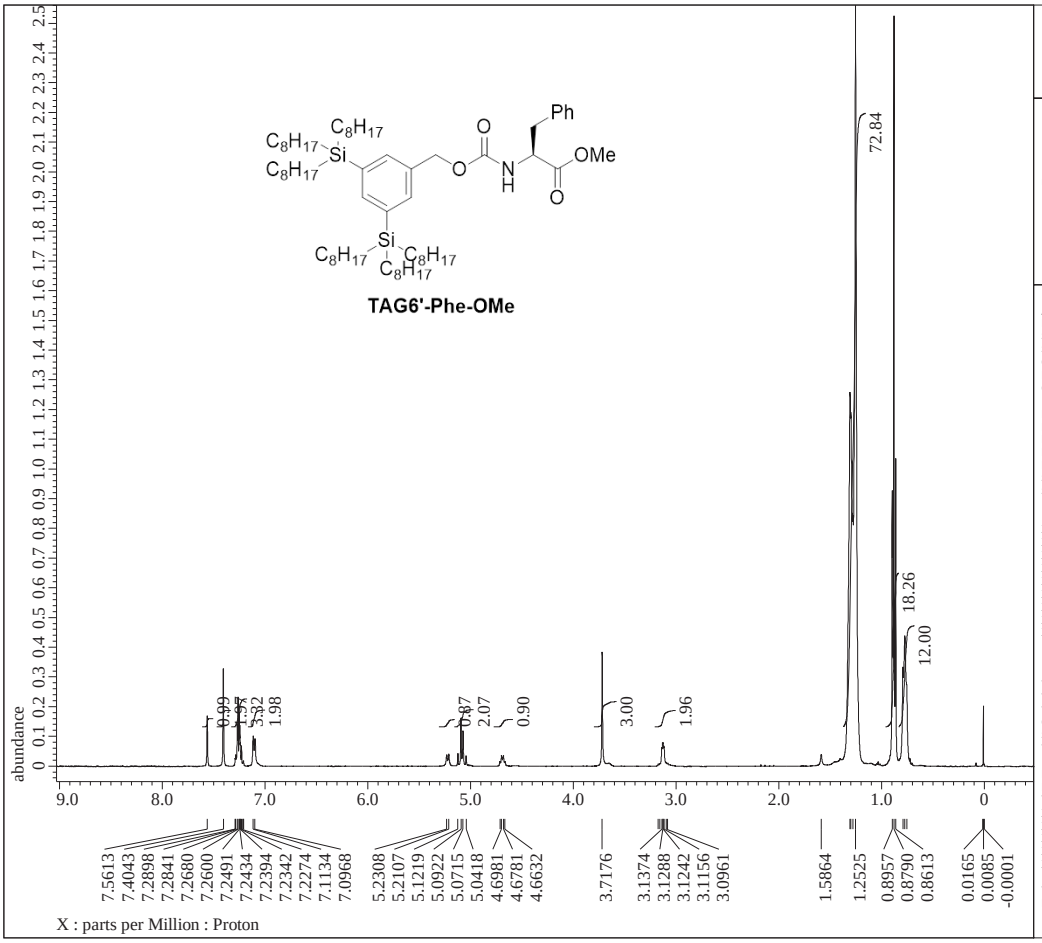

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1199_E1H-1-8.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1199
Solvent           = CHLOROFORM-D
Creation_Time     =  1-NOV-2021 11:07:56
Revision_Time     = 10-NOV-2021 11:13:10
Current_Time      = 10-NOV-2021 11:13:32

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        = 18
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
```




```
---- 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: wuan-1199_E13C-1-1.jdf
```

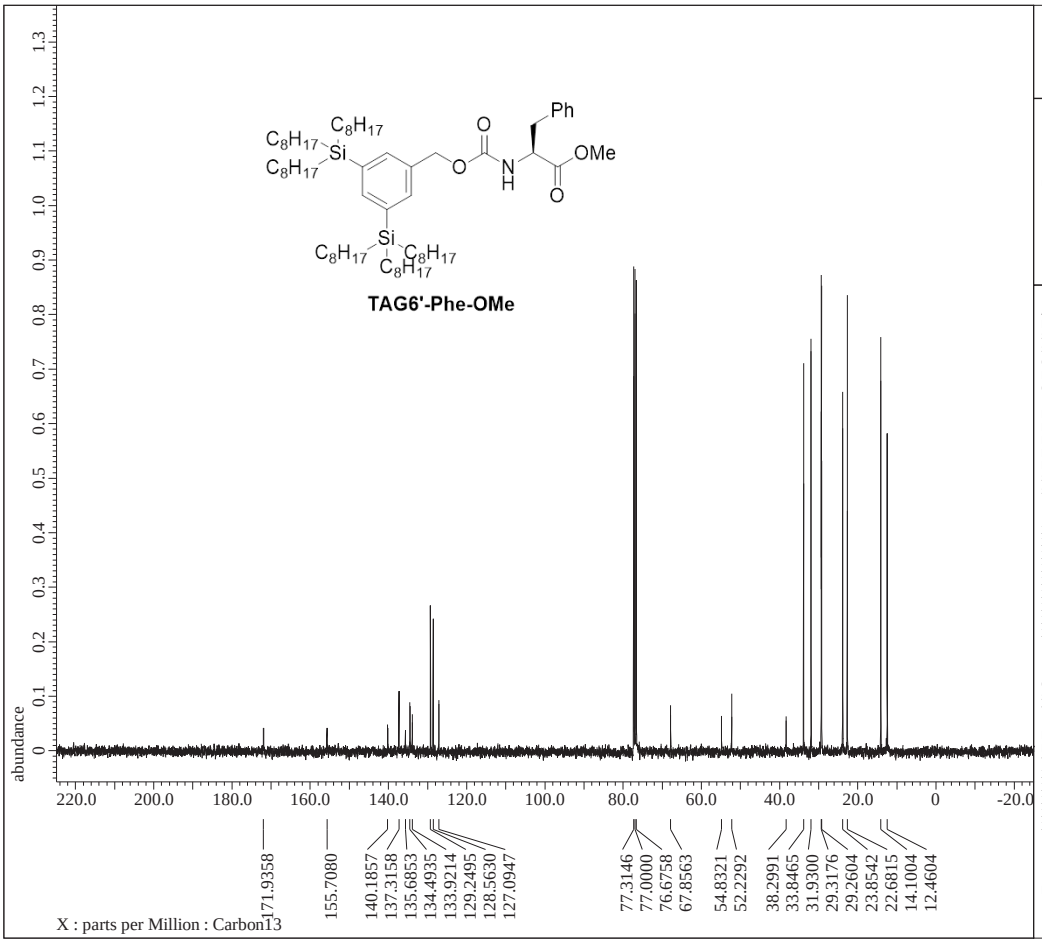
```
Filename          = wuan-1199_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1199
Solvent           = CHLOROFORM-D
Creation_Time     =  1-NOV-2021 11:11:31
Revision_Time     =  1-NOV-2021 11:19:24
Current_Time      =  1-NOV-2021 11:19:47

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             = 237
Total_Scans       = 237

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]
```



JEOL RESONANCE

```
---- 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: wuan-1205_E1H-1-1.jdf
```

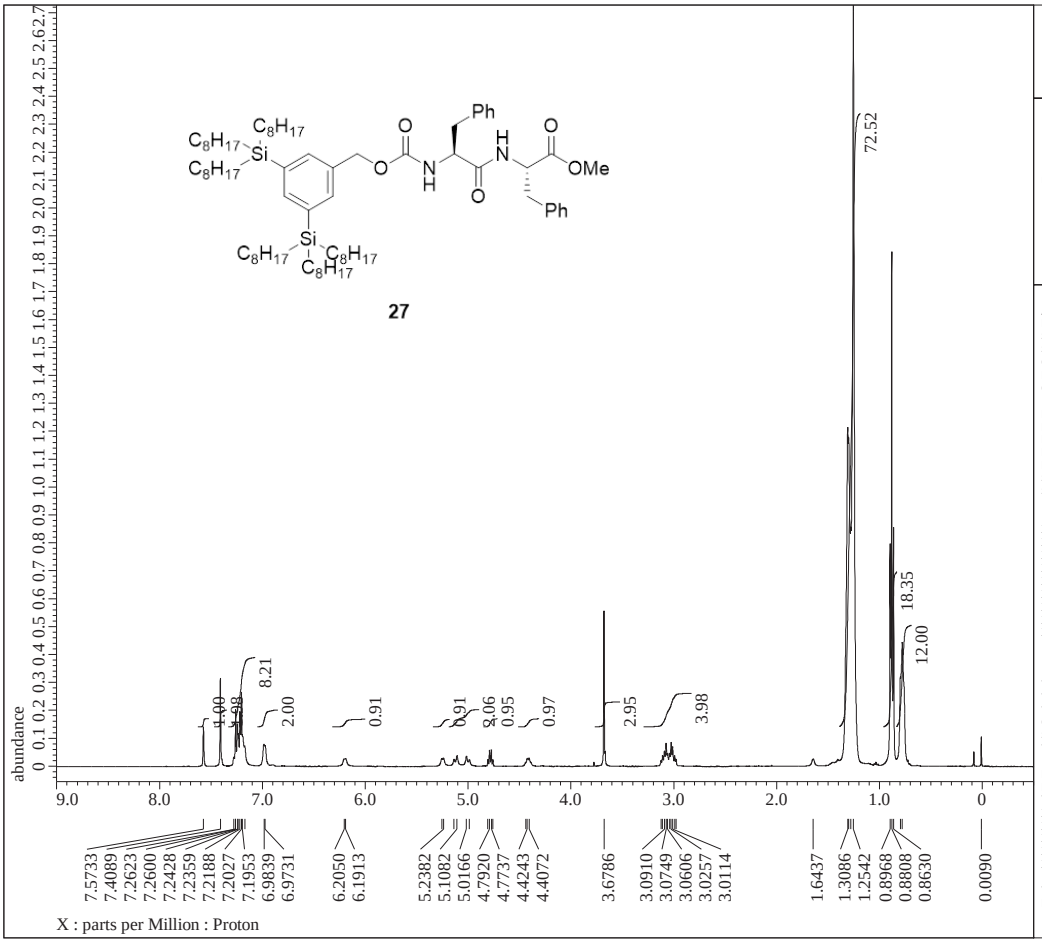
```
Filename          = wuan-1205_E1H-1-3.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1205
Solvent           = CHLOROFORM-D
Creation_Time     = 10-NOV-2021 11:06:30
Revision_Time     = 10-NOV-2021 11:10:45
Current_Time      = 10-NOV-2021 11:11:56

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          = 24.9[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
```



JEOL RESONANCE

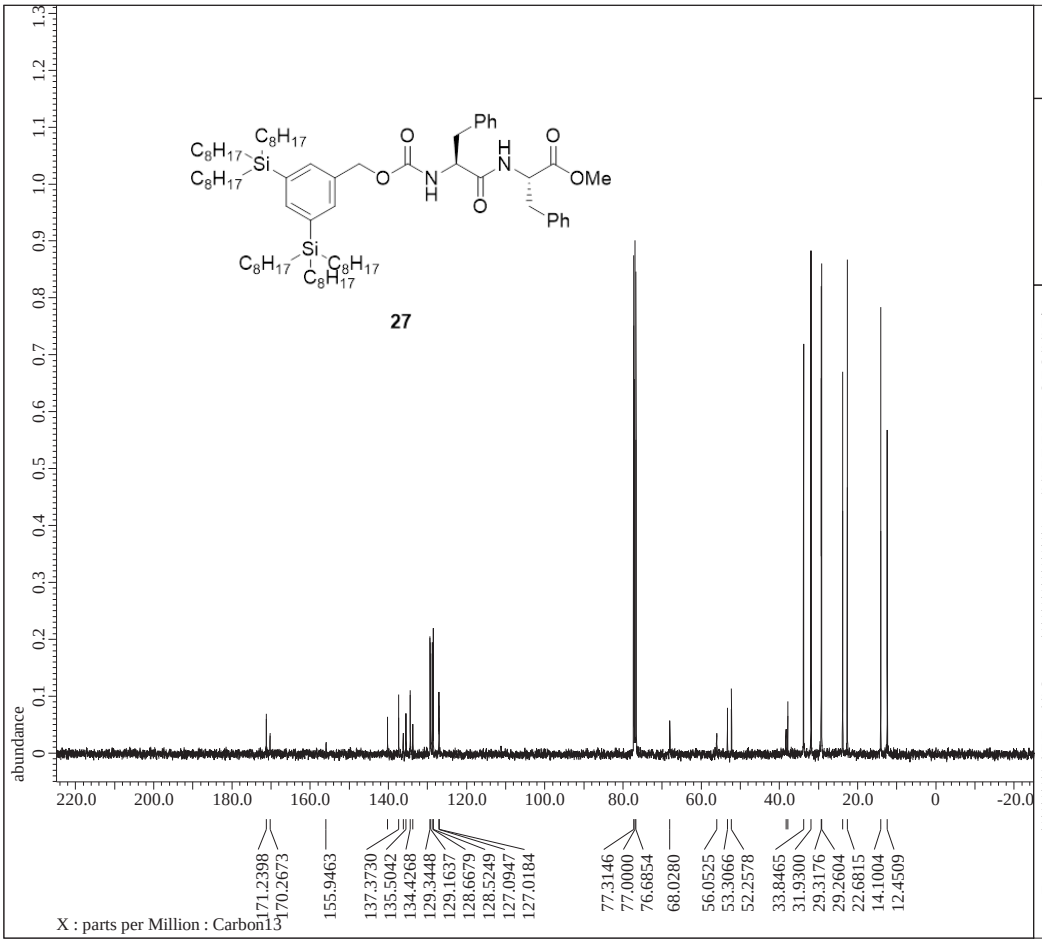
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: wuan-1205_E13C-1-1.jdf
```

```
Filename          = wuan-1205_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1205
Solvent           = CHLOROFORM-D
Creation_Time     = 10-NOV-2021 11:10:25
Revision_Time     = 10-NOV-2021 11:16:51
Current_Time      = 10-NOV-2021 11:17:31

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             = 212
Total_Scans       = 212

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]
```



JEOL RESONANCE

```
---- 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

Filename          = wuan-1208_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1208
Solvent           = CHLOROFORM-D
Creation_Time     = 11-NOV-2021 11:08:25
Revision_Time     = 11-NOV-2021 11:15:40
Current_Time      = 11-NOV-2021 11:16:41

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        = 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
```



JEOL RESONANCE

```
---- 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: wuan-1208_E13C-1-1.jdf

Filename          = wuan-1208_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1208
Solvent           = CHLOROFORM-D
Creation_Time     = 11-NOV-2021 11:12:25
Revision_Time     = 11-NOV-2021 11:23:39
Current_Time      = 11-NOV-2021 11:24:18

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             = 300
Total_Scans       = 300

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]
```