Supporting Information for

Super Silyl-Based Stable Protecting Groups for Both the C- and N-Terminals of Peptides: Applied as Effective Hydrophobic Tags in Liquid-Phase Peptide Synthesis

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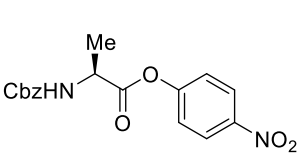
I. General information

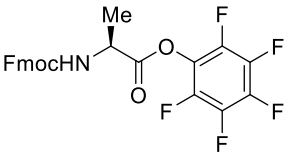
NMR spectra were recorded on a JEOL 400SS spectrometer operating at 400 MHz and 100 MHz for ^{1}H and ^{13}C acquisitions, respectively. Chemical shifts are reported in ppm with a solvent resonance as an internal standard (^{1}H NMR: tetramethylsilane, $CDCl_3$ and CF_3CO_2D as internal standards, indicating 0, 7.26 and 11.50 ppm, respectively. ^{13}C NMR: $CDCl_3$ and CF_3CO_2D as internal standards, indicating 77.0 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. 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 and Li were purchased from Sigma-Aldrich Co. LLC.. Chlorotrihexylsilane, pentafluorophenol, triflic acid, 5-oxohexanoic acid and 1-methylimidazole were purchased from Tokyo Chemical Industry Co., Ltd. Amberlyst^{TM} A21 was purchased from Sigma-Aldrich Co. LLC.

II. Preparation of building blocks for peptide elongation

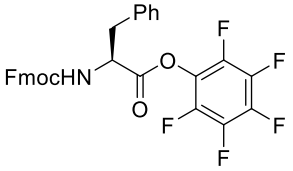
The following active amino acid esters and neutralized amino acids were used in this project.



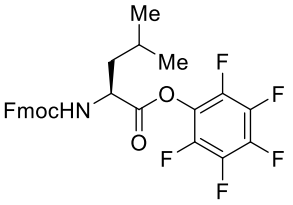
Cbz-Ala-ONp



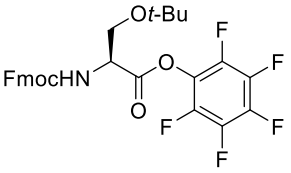
Fmoc-Ala-OPfp



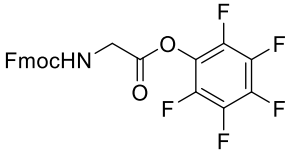
Fmoc-Phe-OPfp



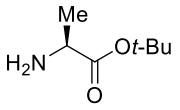
Fmoc-Leu-OPfp



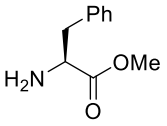
Fmoc-Ser(*t*-Bu)-OPfp



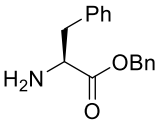
Fmoc-Gly-OPfp



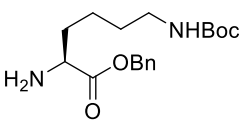
H-Ala-O*t*-Bu



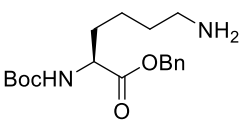
H-Phe-OMe



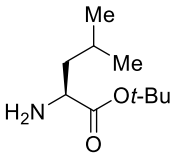
H-Phe-OBn



H-Lys(Boc)-OBn

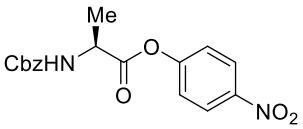


Boc-Lys-OBn



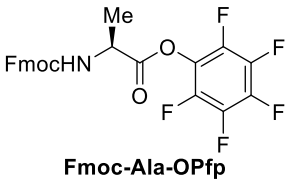
H-Leu-O*t*-Bu

Synthesis of active amino acid esters.



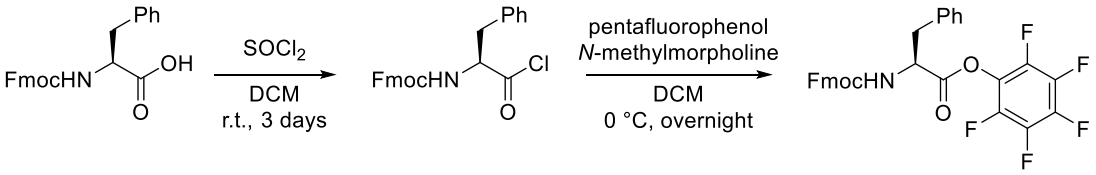
Cbz-Ala-ONp

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



Fmoc-Ala-OPfp

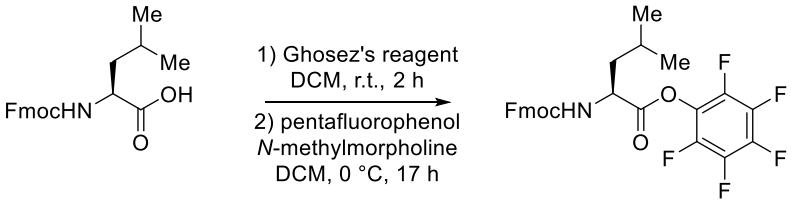
Perfluorophenyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OPfp) was prepared according to the procedure in the literatures.[1, 2].



Fmoc-Phe-OH **Fmoc-Phe-Cl**, 98% **Fmoc-Phe-OPfp**, 70%

Perfluorophenyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-phenylalaninate (Fmoc-Phe-OPfp) At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Phe-OH** (1.94 g, 5.0 mmol, 1.0 equiv) was added dichloromethane (25 mL). The thionyl chloride (2.9 mL, 40.0 mmol, 8.0 equiv) was added. The resulting mixture was stirred under room temperature for 3 days. 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-Phe-Cl** was obtained in 98% yield (1.99 g).

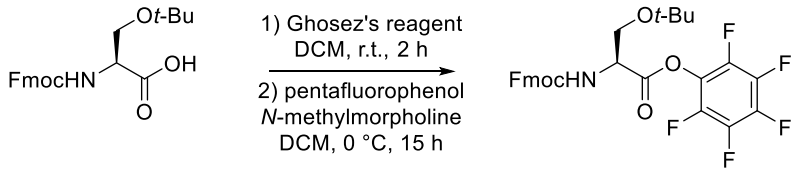
At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Phe-Cl** (4.06 g, 10.0 mmol, 1.0 equiv) was added pentafluorophenol (3.68 g, 20.0 mmol, 2.0 equiv) and dichloromethane (60 mL). The *N*-methylmorpholine (1.31 mL, 12.0 mmol, 1.2 equiv) was added dropwise. The resulting mixture was stirred overnight. After completion, saturated Na_2CO_3 solution (50 mL) was added. The layers were separated. 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/$CHCl_3$ = 4:1:0 to 1:1:0 to 1:3:1) to afford the product **Fmoc-Phe-OPfp** as a white solid in 70% yield (3.9 g). It is a known compound. The characterization data match the reported data.[3]



Fmoc-Leu-OH **Fmoc-Leu-OPfp**, 88%

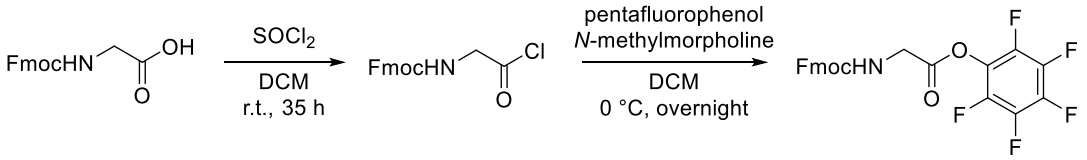
Perfluorophenyl (((9H-fluoren-9-yl)methoxy)carbonyl)-L-leucinate (Fmoc-Leu-OPfp) At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Leu-OH** (1.06 g, 3.0 mmol, 1.0 equiv) was added dichloromethane (30 mL). The Ghosez's reagent (436.6 μL, 3.3 mmol, 1.1 equiv) was added. The resulting mixture was stirred under room temperature. After 2 h, the reaction mixture was cooled to 0 °C. The pentafluorophenol (828.3 mg, 4.5 mmol, 1.5 equiv) was added, followed by adding *N*-

methylmorpholine (494.8 μL, 4.5 mmol, 1.5 equiv). The resulting mixture was stirred at 0 ºC for 17 h. After completion, saturated Na_2CO_3 solution (30 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 (3×70 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 = 5:1:1) to afford the product **Fmoc-Leu-OPfp** as a white solid in 88% yield (1.37 g). It is a known compound.[4]



Fmoc-Ser(*t*-Bu)-OH **Fmoc-Ser(*t*-Bu)-OPfp**, 94%

Perfluorophenyl N-(((9*H*-fluoren-9-yl)methoxy)carbonyl)-O-(tert-butyl)-L-serinate (Fmoc-Ser(*t*-Bu)-OPfp) At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ser(*t*-Bu)-OH** (1.92 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 0 ºC. The pentafluorophenol (1.38 g, 7.5 mmol, 1.5 equiv) was added, followed by adding *N*-methylmorpholine (824.6 μL, 7.5 mmol, 1.5 equiv). The resulting mixture was stirred at 0 ºC for 15 h. After completion, saturated Na_2CO_3 solution (30 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 (3×70 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 = 5:1:1) to afford the product **Fmoc-Ser(*t*-Bu)-OPfp** as a white solid in 94% yield (2.58 g). It is a known compound. The characterization data match the reported data.[3]



Fmoc-Gly-OH **Fmoc-Gly-Cl**, 97% **Fmoc-Gly-OPfp**, 60%

Perfluorophenyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)glycinate (Fmoc-Gly-OPfp) At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Gly-OH** (1.49 g, 5.0 mmol, 1.0 equiv, made into powder) was added dichloromethane (25 mL). The thionyl chloride (2.9 mL, 40.0 mmol, 8.0 equiv) was added. The resulting mixture was stirred under room temperature for 35 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-Gly-Cl** was obtained in 97% yield (1.53 g).

At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Fmoc-Gly-Cl** (3.16 g, 10.0 mmol, 1.0 equiv) was added pentafluorophenol (3.68 g, 20.0 mmol, 2.0 equiv) and dichloromethane (60 mL). The *N*-methylmorpholine (1.31 mL, 12.0 mmol, 1.2 equiv) was added dropwise. The resulting mixture was stirred overnight. After completion, saturated Na_2CO_3 solution (50 mL) was added. The layers were separated. 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/$CHCl_3$ = 4:1:0 to 1:1:0 to 1:3:1) to afford the product **Fmoc-Gly-OPfp** as a white solid in 60% yield (2.78 g). [***Note**: The purification by chromatography should be separated for 2~3 times because of easily solidification on the column.*] It is a known compound. The characterization data match the reported data.[5]

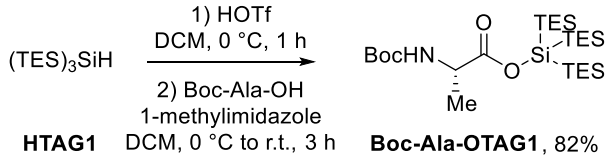
Neutralization of amino acid HCl salts.

H-Ala-O*t*-Bu, **H-Phe-OMe**, **H-Phe-OBn**, **H-Lys(Boc)-OBn** and **H-Leu-O*t*-Bu** were neutralized from the HCl salts with Amberlyst™ A21 according to the procedure in the literatures.[6, 7]

Boc-Lys-OBn was neutralized from the TsOH salts with Amberlyst™ A21 according to the procedure in the literatures.[6, 7]

III. Test of TAG1

Esterification test.



1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl (*tert*-butoxycarbonyl)-L-alaninate (Boc-Ala-OTAG1) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (15 mL) was added triflic acid (442.4 μL, 5.0 mmol, 1.0 equiv). The tris(triethylsilyl)silane (1.87 g, 5.0 mmol, 1.0 equiv) was added. The reaction was stirred for 1 h. Then, mixed Boc-Ala-OH (946.05 mg, 5.0 mmol, 1.0 equiv) and 1-methylimidazole (592.1 μL, 7.5 mmol, 1.5 equiv) in dichloromethane (3 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After stirring under room temperature for 3 h, the reaction mixture was diluted with hexanes, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 30:1) to afford the product **Boc-Ala-OTAG1** as a colorless oil in 82% yield (2.31 g).

Rf = 0.16 (hexanes/EtOAc = 20:1).

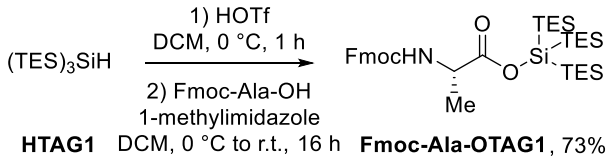
$[\alpha]_D^{25}$ = -0.94 (*c* 1.06, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.12 (d, J = 7.6 Hz, 1H), 4.26 – 4.13 (m, 1H), 1.42 (s, 9H), 1.32 (d, J = 7.2 Hz, 3H), 1.04 – 0.96 (m, 27H), 0.84 – 0.74 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 154.9, 79.4, 50.3, 28.3, 18.8, 8.5, 5.0.

IR (neat) 2952, 2908, 2875, 1702, 1496, 1454, 1366, 1345, 1214, 1163 cm^{-1}.

HRMS (ESI) Calcd for $C_{26}H_{59}NO_4Si_4Na$ $[M+Na]^+$: 584.3419, Found: 584.3382.



1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG1) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (3mL) was added triflic acid (88.5 μL, 1.0 mmol, 1.0 equiv). The tris(triethylsilyl)silane (422.7 μL, 1.0 mmol, 1.0 equiv) was added. The reaction was stirred for 1 h. Then, mixed Fmoc-Ala-OH (311.3 mg, 1.0 mmol, 1.0 equiv) and

1-methylimidazole (118.4 μL, 1.5 mmol, 1.5 equiv) in dichloromethane (1 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After stirring under room temperature for 16 h, the reaction mixture was diluted with hexanes, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 40:1 to 25:1) to afford the product **Fmoc-Ala-OTAG1** as a colorless oil in 73% yield (502.0 mg).

Rf = 0.11 (hexanes/EtOAc = 20:1).

$[\alpha]_D^{22}$ = -2.91 (*c* 1.03, $CHCl_3$).

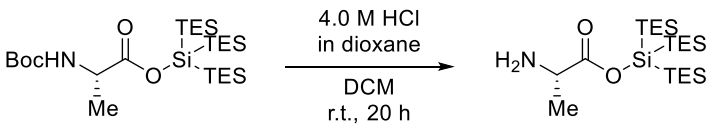
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 7.5 Hz, 2H), 7.66 – 7.57 (m, 2H), 7.45 – 7.36 (m, 2H), 7.36 – 7.28 (m, 2H), 5.47 (d, J = 7.3 Hz, 1H), 4.43 – 4.33 (m, 2H), 4.33 – 4.19 (m, 2H), 1.40 (d, J = 7.1 Hz, 3H), 1.07 – 0.99 (m, 27H), 0.87 – 0.78 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 155.4, 144.0, 143.8, 141.3, 127.6, 127.0, 125.2, 125.1, 119.9, 66.9, 50.8, 47.1, 18.9, 8.5, 5.0.

IR (neat) 2951, 2907, 2874, 1700, 1503, 1450, 1415, 1377, 1339, 1311, 1209, 1072 cm^{-1}.

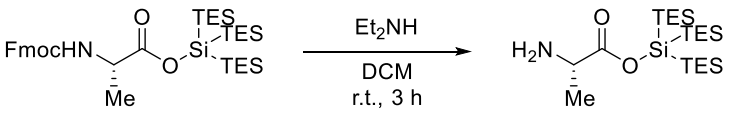
HRMS (ESI) Calcd for $C_{36}H_{61}NO_4Si_4Na$ $[M+Na]^+$: 706.3575, Found: 706.3613.

Tolerance test.



Boc-Ala-OTAG1 **H-Ala-OTAG1**, 0%

1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl L-alaninate (H-Ala-OTAG1) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Boc-Ala-OTAG1** (561.1 mg, 1.0 mmol, 1.0 equiv) was added hydrochloric acid solution (4.0 M in dioxane, 2.0 mL, 8.0 mmol, 8.0 equiv). The reaction was stirred at room temperature for 20 h. Then, 5 mL EtOAc and 10 mL saturated $NaHCO_3$ solution were added, and the layers were separated. The aqueous layer was extracted with EtOAc (3×10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated. No product **H-Ala-OTAG1** was detected from the crude ^{1}H NMR of the residue.



Fmoc-Ala-OTAG1 **H-Ala-OTAG1**, 33%

1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl L-alaninate (H-Ala-OTAG1) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAG1** (718.4 mg, 1.05 mmol, 1.0 equiv) was added dichloromethane (1.0 mL). The diethylamine (517.3 μL, 5.0 mmol, 4.8 equiv) was added. The reaction was stirred at 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 4:1) to afford the product **H-Ala-OTAG1** as a white solid in 33% yield (158.0 mg).

Rf = 0.16 (hexanes/EtOAc = 5:1).

M.p. 88-90 ºC.

$[\alpha]_D^{27}$ = +5.50 (*c* 1.09, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.41 (q, J = 7.1 Hz, 1H), 1.28 (d, J = 7.1 Hz, 3H), 1.08 – 0.96 (m, 27H), 0.86 – 0.74 (m, 18H).

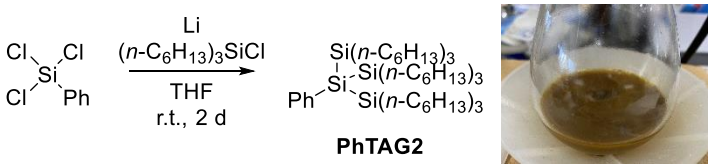
^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 51.3, 20.5, 8.5, 5.0.

IR (neat) 2951, 2908, 2874, 1702, 1458, 1415, 1376, 1234, 1198, 1142 cm^{-1}.

HRMS (ESI) Calcd for $C_{21}H_{51}NO_2Si_4Na$ $[M+Na]^+$: 484.2895, Found: 484.2922.

IV. Synthesis of TAG2 and test

Synthesis of TAG2.



1,1,1,3,3,3-Hexahexyl-2-phenyl-2-(trihexylsilyl)trisilane (PhTAG2) At room temperature, under N_2, to a flame-dried flask charged with Li (granular, 388.6 mg, 56.0 mmol, 8.0 equiv) was added tetrahydrofuran (50 mL). The trichloro(phenyl)silane (1.12 mL, 7.0 mmol, 1.0 equiv) and chlorotrihexylsilane (8.2 mL, 22.4 mmol, 3.2 equiv) were added together. The reaction was stirred at room temperature for 2~4 days. [***Note****: the mixture should turn dark brown within 24 h like the above figure. If not, more Li (1~2 equiv) should be added and ultrasonication (3~5 min, then stirred under r.t.) could also give an assistance. In a few cases, the colour did not change to dark brown, but the reaction still proceeded just with lower yields. The reaction was monitored by* ^{1}*H NMR.*] Then, the reaction mixture was poured into a separation funnel charged with 100 mL hexanes and 50 mL water. 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 **PhTAG2** as a colorless oil in 87% yield with impurities (5.84 g, ca. 60%~70% purity). [***Note****: The purification by chromatography should be repeated for several times to make the peaks of impurities in* ^{1}*H NMR (400 MHz,* $CDCl_3$*,* δ *0.70 – 0.45) as low as possible.*] The product was put into next steps without further purification. Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data and structure of installed compounds support it.

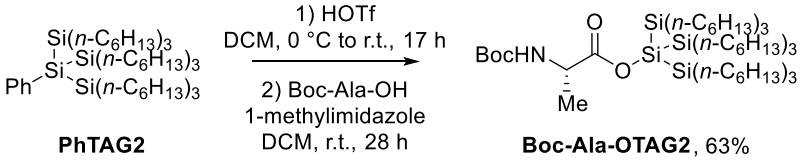
Rf = 0.88 (100% hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.40 (m, 2H), 7.21 – 7.12 (m, 3H), 1.35 – 1.19 (m, 72H), 0.93 – 0.83 (m, 27H), 0.80 – 0.71 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 137.2, 137.1, 127.1, 126.8, 33.9, 31.6, 25.1, 22.7, 15.2, 14.1.

IR (neat) 2956, 2919, 2871, 2853, 1465, 1377, 1182 cm^{-1}.

Esterification test.



1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl (*tert*-butoxycarbonyl)-L-alaninate (Boc-Ala-OTAG2) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (1.0 mL) was added **PhTAG2** (392.0 mg, 0.41 mmol, 1.36 equiv). The triflic acid (36.3 μL, 0.41 mmol, 1.36 equiv) was added. The reaction was stirred under room temperature for 17 h. Then, mixed Boc-Ala-OH (56.8 mg, 0.30 mmol, 1.0 equiv) and 1-methylimidazole (40.3 μL, 0.51 mmol, 1.7 equiv) in dichloromethane (0.5 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After 28 h, the reaction mixture was diluted with hexanes, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1) to afford the product **Boc-Ala-OTAG2** as a pale yellow oil in 63% yield (202.0 mg).

Rf = 0.43 (hexanes/EtOAc = 20:1).

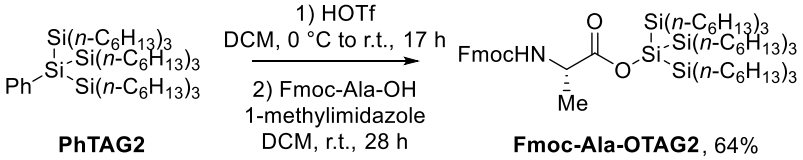
$[\alpha]_D^{26}$ = -5.00 (*c* 1.00, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.23 (d, J = 7.0 Hz, 1H), 4.28 – 4.09 (m, 1H), 1.44 (s, 9H), 1.37 – 1.24 (m, 75H), 0.93 – 0.85 (m, 27H), 0.78 – 0.48 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 154.8, 79.3, 50.4, 33.9, 31.6, 28.3, 25.0, 22.7, 19.0, 14.11, 14.05.

IR (neat) 2956, 2919, 2871, 2854, 1724, 1703, 1495, 1456, 1377, 1367, 1344, 1216, 1167 cm^{-1}.

HRMS (ESI) Calcd for $C_{62}H_{131}NO_4Si_4Na$ $[M+Na]^+$: 1088.9053, Found: 1088.9027.



1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG2) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (1.0 mL) was added **PhTAG2** (392.0 mg, 0.41 mmol, 1.36 equiv). The triflic acid (36.3 μL, 0.41 mmol, 1.36 equiv) was added. The reaction was stirred under room temperature for 17 h. Then, mixed Fmoc-Ala-OH (93.4 mg, 0.30 mmol, 1.0 equiv) and 1-methylimidazole (40.3 μL, 0.51 mmol, 1.7 equiv) in dichloromethane (0.5 mL) in a flame-dried vial. The mixture in the vial was added into the

reaction flask slowly. After 28 h, the reaction mixture was diluted with hexanes, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1) to afford the product **Fmoc-Ala-OTAG2** as a pale yellow oil in 64% yield (229.0 mg).

Rf = 0.37 (hexanes/EtOAc = 20:1).

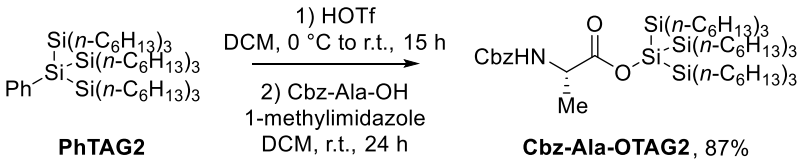
$[\alpha]_D^{24}$ = +0.97 (*c* 1.03, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 7.5 Hz, 2H), 7.67 – 7.55 (m, 2H), 7.45 – 7.37 (m, 2H), 7.35 – 7.28 (m, 2H), 5.57 (d, J = 6.7 Hz, 1H), 4.46 – 4.17 (m, 4H), 1.41 (d, J = 7.0 Hz, 3H), 1.39 – 1.21 (m, 72H), 0.95 – 0.85 (m, 27H), 0.83 – 0.53 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 155.3, 144.1, 143.9, 141.30, 141.26, 127.6, 127.0, 125.2, 125.1, 119.9, 66.9, 50.8, 47.2, 33.9, 31.6, 25.0, 22.7, 19.0, 14.11, 14.07.

IR (neat) 2956, 2920, 2854, 1732, 1704, 1503, 1451, 1378, 1339, 1209, 1185 cm^{-1}.

HRMS (ESI) Calcd for $C_{72}H_{133}NO_4Si_4Na$ $[M+Na]^+$: 1210.9209, Found: 1210.9189.



PhTAG2 **Cbz-Ala-OTAG2**, 87%

1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl ((benzyloxy)carbonyl)-L-alaninate (Cbz-Ala-OTAG2) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (3.0 mL) was added **PhTAG2** (669.2 mg, 0.70 mmol, 1.4 equiv). The triflic acid (61.9 μL, 0.70 mmol, 1.4 equiv) was added. The reaction was stirred under room temperature for 15 h. Then, mixed Cbz-Ala-OH (111.6 mg, 0.50 mmol, 1.0 equiv) and 1-methylimidazole (67.1 μL, 0.85 mmol, 1.7 equiv) in dichloromethane (1.0 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After 24 h, the reaction mixture was diluted with hexanes, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1) to afford the product **Cbz-Ala-OTAG2** as a colorless oil in 87% yield (476.3 mg).

Rf = 0.39 (hexanes/EtOAc = 20:1).

$[\alpha]_D^{24}$ = +6.55 (*c* 1.07, $CHCl_3$).

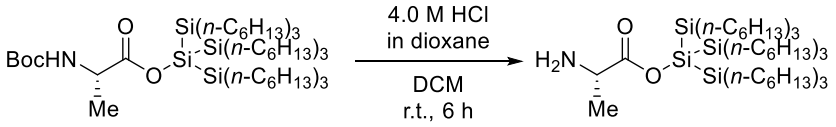
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 5.52 (d, J = 6.7 Hz, 1H), 5.20 – 4.96 (m, 2H), 4.30 – 4.15 (m, 1H), 1.38 (d, J = 7.0 Hz, 3H), 1.35 – 1.20 (m, 72H), 0.92 – 0.85 (m, 27H), 0.78 – 0.53 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 155.2, 136.5, 128.5, 128.02, 127.96, 66.6, 50.8, 33.9, 31.6, 25.0, 22.7, 19.0, 14.1, 14.0.

IR (neat) 2956, 2920, 2871, 2854, 1733, 1704, 1500, 1455, 1378, 1340, 1311, 1208, 1184 cm^{-1}.

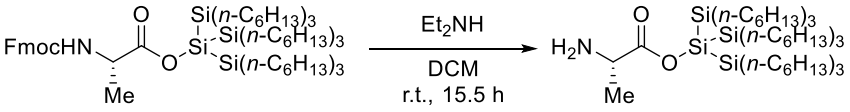
HRMS (ESI) Calcd for $C_{65}H_{129}NO_4Si_4Na$ $[M+Na]^+$: 1122.8896, Found: 1122.8908.

Tolerance test.



Boc-Ala-OTAG2 **H-Ala-OTAG2**, 0%

1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl 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** (53.4 mg, 0.05 mmol, 1.0 equiv) was added dichloromethane (0.3 mL). The hydrochloric acid solution (4.0 M in dioxane, 0.10 mL, 0.40 mmol, 8.0 equiv) was added. The reaction was stirred at room temperature for 6 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-OTAG2** was detected from the crude ^{1}H NMR of the residue.



Fmoc-Ala-OTAG2 **H-Ala-OTAG2**, 75%

1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl 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** (163.3 mg, 0.14 mmol, 1.0 equiv) was added dichloromethane (0.3 mL). The diethylamine (141.7 μL, 1.4 mmol, 10.0 equiv) was added. The reaction was stirred at room temperature for 15.5 h. After completion, 5 mL dichloromethane and 5 mL water 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. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 50:1 to 25:1) to afford the product **H-Ala-OTAG2** as a colorless oil in 75% yield (100.2 mg).

Rf = 0.19 (hexanes/EtOAc = 20:1).

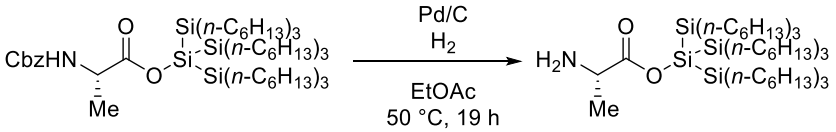
$[\alpha]_D^{27}$ = +10.78 (*c* 1.02, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.38 (q, *J* = 7.1 Hz, 1H), 1.38 – 1.19 (m, 75H), 0.93 – 0.84 (m, 27H), 0.79 – 0.52 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 51.4, 33.9, 31.6, 25.0, 22.7, 20.4, 14.1(2C).

IR (neat) 2956, 2919, 2871, 2853, 1703, 1457, 1411, 1377, 1186, 1100 cm^{-1}.

HRMS (ESI) Calcd for $C_{57}H_{123}NO_2Si_4Na$ $[M+Na]^+$: 988.8529, Found: 988.8535.

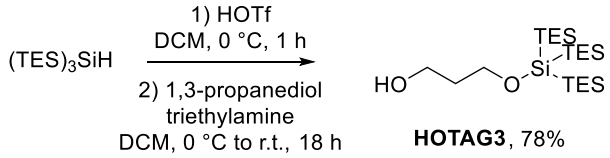


Cbz-Ala-OTAG2 **H-Ala-OTAG2**, 77%

1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl L-alaninate (H-Ala-OTAG2) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-OTAG2** (194.0 mg, 0.18 mmol, 1.0 equiv) was added EtOAc (2.0 mL). The 10% Pd/C (18.7 mg, 0.018 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 ºC for 19 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 25:1) to afford the product **H-Ala-OTAG2** as a colorless oil in 77% yield (131.5 mg).

V. Synthesis of TAG3 and test

Synthesis of TAG3.



3-((1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)propan-1-ol (HOTAG3) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (3 mL) was added triflic acid (88.5 μL, 1.0 mmol, 1.0 equiv). The tris(triethylsilyl)silane (422.7 μL, 1.0 mmol, 1.0 equiv) was added. The reaction was stirred for 1 h. Then, mixed 1,3-propanediol (144.9 μL, 2.0 mmol, 2.0 equiv) and triethylamine (418.1 μL, 3.0 mmol, 3.0 equiv) in dichloromethane (2 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After stirring under room temperature for 18 h, 5 mL dichloromethane and 10 mL saturated $NaHCO_3$ 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 = 30:1) to afford the product **HOTAG3** as a white solid in 78% yield (350.8 mg).

Rf = 0.50 (hexanes/EtOAc = 5:1).

M.p. 84-90 ºC.

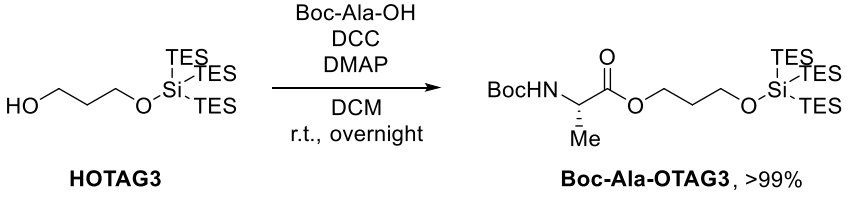
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.79 – 3.70 (m, 2H), 3.69 – 3.61 (m, 2H), 2.62 (t, J = 5.4 Hz, 1H), 1.77 – 1.66 (m, 2H), 1.09 – 0.99 (m, 27H), 0.84 – 0.72 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 68.7, 62.8, 34.3, 8.6, 5.3.

IR (neat) 2950, 2907, 2873, 1460, 1416, 1376, 1234 cm^{-1}.

HRMS (ESI) Calcd for $C_{21}H_{52}O_2Si_4Na$ $[M+Na]^+$: 471.2942, Found: 471.2922.

Esterification test.



3-((1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)propyl (*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** (134.7 mg, 0.30 mmol, 1.0 equiv) was added dichloromethane (3 mL). The Boc-Ala-OH (170.3 mg, 0.90 mmol, 3.0 equiv) was added, followed by adding DMAP (44.0 mg, 0.36 mmol, 1.2 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 = 30:1) to afford the product **Boc-Ala-OTAG3** as a light green oil in >99% yield (188.8 mg).

Rf = 0.49 (hexanes/EtOAc = 5:1).

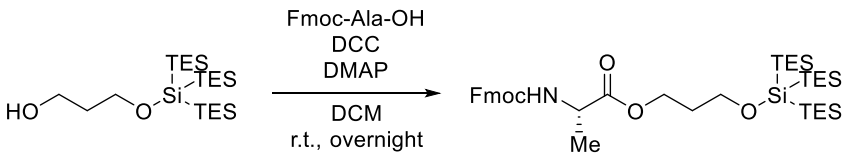
$[\alpha]_D^{27}$ = +1.02 (*c* 0.98, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.05 (d, *J* = 9.0 Hz, 1H), 4.34 – 4.23 (m, 1H), 4.22 – 4.13 (m, 2H), 3.46 (t, *J* = 6.0 Hz, 2H), 1.82 – 1.71 (m, 2H), 1.44 (s, 9H), 1.36 (d, *J* = 7.1 Hz, 3H), 1.07 – 0.98 (m, 27H), 0.81 – 0.70 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 155.0, 79.7, 64.1, 62.4, 49.2, 32.0, 28.3, 18.8, 8.6, 5.3.

IR (neat) 2952, 2908, 2874, 1719, 1498, 1456, 1366, 1237, 1164 cm^{-1}.

HRMS (ESI) Calcd for $C_{29}H_{65}NO_5Si_4Na$ $[M+Na]^+$: 642.3838, Found: 642.3794.



HOTAG3 **Fmoc-Ala-OTAG3**, 83%

3-((1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)propyl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG3) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG3** (154.8 mg, 0.35 mmol, 1.0 equiv) was added dichloromethane (3 mL). The Fmoc-Ala-OH (268.5 mg, 0.86 mmol, 2.5 equiv) was added, followed by adding DMAP (50.6 mg, 0.41 mmol, 1.2 equiv) and DCC (178.0 mg, 0.86 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 = 40:1 to 20:1 to 15:1) to afford the product **Fmoc-Ala-OTAG3** as a colorless oil in 83% yield (203.7 mg).

Rf = 0.47 (hexanes/EtOAc = 5:1).

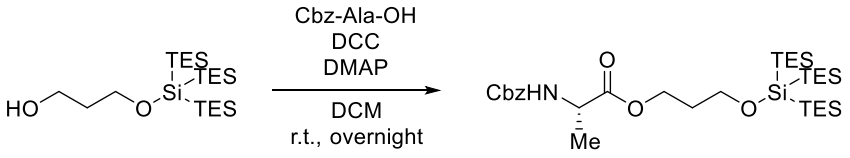
$[\alpha]_D^{27}$ = +14.95 (*c* 1.07, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 7.5 Hz, 2H), 7.64 – 7.57 (m, 2H), 7.46 – 7.37 (m, 2H), 7.37 – 7.29 (m, 2H), 5.40 (d, J = 7.7 Hz, 1H), 4.52 – 4.33 (m, 3H), 4.27 – 4.17 (m, 3H), 3.48 (t, J = 5.8 Hz, 2H), 1.84 – 1.73 (m, 2H), 1.43 (d, J = 7.1 Hz, 3H), 1.09 – 0.98 (m, 27H), 0.84 – 0.71 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.0, 155.5, 143.9, 143.8, 141.3, 127.7, 127.0, 125.1, 119.9, 66.9, 64.0, 62.6, 49.6, 47.1, 31.9, 18.8, 8.6, 5.3.

IR (neat) 3019, 2953, 2874, 1719, 1508, 1451, 1337, 1214 cm^{-1}.

HRMS (ESI) Calcd for $C_{39}H_{67}NO_5Si_4Na$ $[M+Na]^+$: 764.3994, Found: 764.4006.



HOTAG3 **Cbz-Ala-OTAG3**, 90%

3-((1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)propyl ((benzyloxy)carbonyl)-L-alaninate (Cbz-Ala-OTAG3) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG3** (157.1 mg, 0.35 mmol, 1.0 equiv) was added dichloromethane (3 mL). The Cbz-Ala-OH (195.3 mg, 0.88 mmol, 2.5 equiv) was added, followed by adding DMAP (51.3 mg, 0.42 mmol, 1.2 equiv) and DCC (180.5 mg, 0.88 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 = 30:1 to 15:1) to afford the product **Cbz-Ala-OTAG3** as a colorless oil in 90% yield (197.9 mg).

Rf = 0.47 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{27}$ = +8.41 (*c* 1.07, $CHCl_3$).

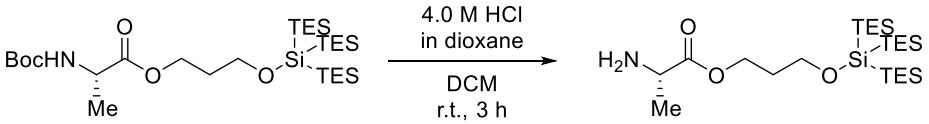
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 5H), 5.32 (d, J = 8.1 Hz, 1H), 5.17 – 5.08 (m, 2H), 4.40 – 4.31 (m, 1H), 4.18 (t, J = 6.4 Hz, 2H), 3.46 (t, J = 5.9 Hz, 2H), 1.80 – 1.68 (m, 2H), 1.40 (d, J = 7.1 Hz, 3H), 1.07 – 0.97 (m, 27H), 0.83 – 0.70 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 155.5, 136.3, 128.5, 128.11, 128.07, 66.8, 64.0, 62.6, 49.6, 31.9, 18.8, 8.6, 5.3.

IR (neat) 2951, 2907, 2874, 1726, 1505, 1455, 1338, 1308, 1203, 1177 cm^{-1}.

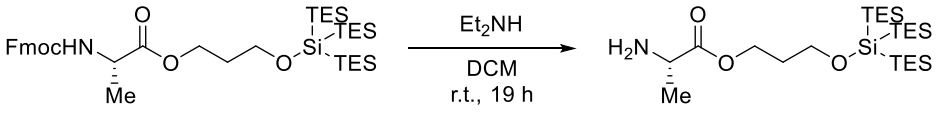
HRMS (ESI) Calcd for $C_{32}H_{63}NO_5Si_4Na$ $[M+Na]^+$: 676.3681, Found: 676.3661.

Tolerance test.



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

3-((1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)propyl 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** (12.4 mg, 0.02 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 3 h. After completion, the reaction mixture was concentrated. No product **H-Ala-OTAG3** was detected from the crude ^{1}H NMR of the residue.



Fmoc-Ala-OTAG3 **H-Ala-OTAG3**, 94%

3-((1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)propyl 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** (142.9 mg, 0.20 mmol, 1.0 equiv) was added dichloromethane (2.0 mL). The diethylamine (206.9 μL, 2.0 mmol, 10.0 equiv) was added. The reaction was stirred at room temperature for 19 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 2.5:1 to 2:1) to afford the product **H-Ala-OTAG3** as a colorless oil in 94% yield (92.5 mg).

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

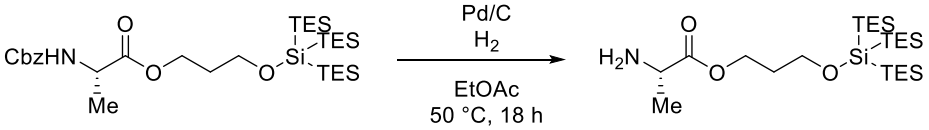
$[\alpha]_D^{27}$ = +22.45 (*c* 0.98, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 (t, *J* = 6.4 Hz, 2H), 3.55 – 3.43 (m, 3H), 1.82 – 1.71 (m, 2H), 1.32 (d, *J* = 7.0 Hz, 3H), 1.08 – 0.97 (m, 27H), 0.76 (qd, *J* = 7.8, 0.9 Hz, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 64.1, 61.9, 50.0, 32.0, 20.5, 8.6, 5.3.

IR (neat) 2951, 2908, 2874, 1738, 1459, 1416, 1376, 1234, 1182, 1141 cm^{-1}.

HRMS (ESI) Calcd for $C_{24}H_{57}NO_3Si_4Na$ $[M+Na]^+$: 542.3313, Found: 542.3283.

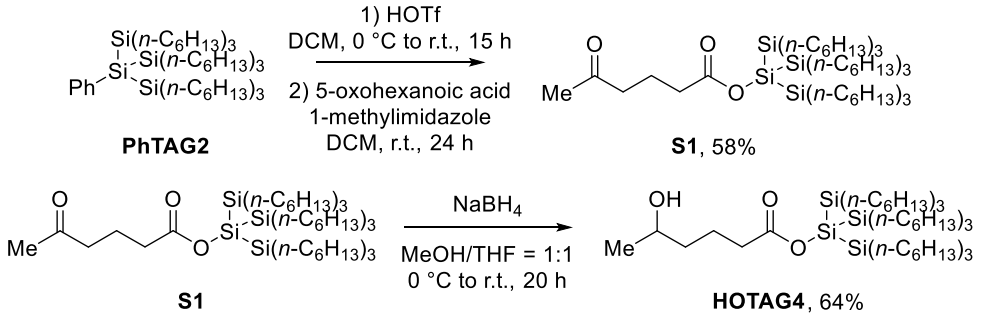


Cbz-Ala-OTAG3 **H-Ala-OTAG3**, 95%

3-((1,1,1,3,3,3-Hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)propyl L-alaninate (H-Ala-OTAG3) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-OTAG3** (125.2 mg, 0.20 mmol, 1.0 equiv) was added EtOAc (2.0 mL). The 10% Pd/C (21.3 mg, 0.02 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 ºC 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 = 2.5:1) to afford the product **H-Ala-OTAG3** as a colorless oil in 95% yield (93.8 mg).

VI. Synthesis of TAG4 and test

Synthesis of TAG4.



1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl 5-hydroxyhexanoate (HOTAG4) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **PhTAG2** (2.87g, 3.0 mmol, 1.5 equiv) was added dichloromethane (9 mL). The triflic acid (265.5 μL, 3.0 mmol, 1.5 equiv) was added. The reaction was stirred at room temperature for 15 h. Then, mixed 5-oxohexanoic acid (238.8 μL, 2.0 mmol, 1.0 equiv) and 1-methylimidazole (268.4 μL, 3.4 mmol, 1.7 equiv) in dichloromethane (4 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After stirring under room temperature for 24 h, the reaction mixture was diluted with hexanes, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 80:1) to afford the product **S1** as a colorless oil in 58% yield (1.16 g) with little impurity.

At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **S1** (1.16 g, 1.16 mmol, 1.0 equiv) was added methanol (5 mL) and tetrahydrofuran (5 mL). Cool the mixture to 0 °C, followed by adding sodium borohydride (87.6 mg, 2.3 mmol, 2.0 equiv). The reaction was stirred at room temperature for 20 h. Then, saturated NH_4Cl solution (10 mL) was added, and the layers were separated. The aqueous layer was extracted with EtOAc (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 = 50:1 to 25:1) to afford the product **HOTAG4** as a colorless oil in 64% yield (744.9 mg).

Rf = 0.38 (hexanes/EtOAc = 10:1).

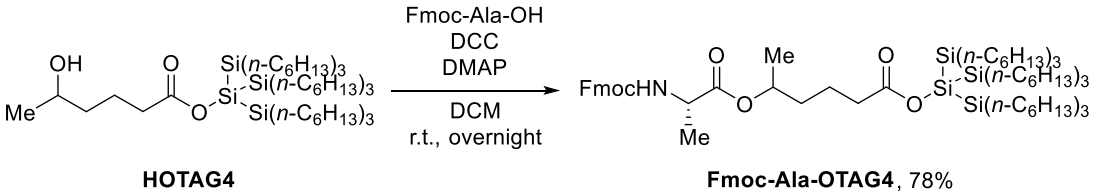
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.84 – 3.72 (m, 1H), 2.32 – 2.15 (m, 2H), 1.71 – 1.60 (m, 2H), 1.49 – 1.41 (m, 2H), 1.41 – 1.20 (m, 72H), 1.18 (d, J = 6.1 Hz, 3H), 0.95 – 0.81 (m, 27H), 0.78 – 0.53 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1, 67.5, 38.9, 35.8, 33.9, 31.7, 25.0, 23.4, 22.7, 21.3, 14.1(2C).

IR (neat) 2956, 2920, 2871, 2854, 1704, 1465, 1412, 1377, 1251, 1182 cm^{-1}.

HRMS (ESI) Calcd for $C_{60}H_{128}O_3Si_4Na$ $[M+Na]^+$: 1031.8838, Found: 1031.8876.

Esterification test.



1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl 5-(((((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alanyl)oxy)hexanoate (Fmoc-Ala-OTAG4) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG4** (151.5 mg, 0.15 mmol, 1.0 equiv) was added dichloromethane (1.5 mL). The Fmoc-Ala-OH (140.1 mg, 0.45 mmol, 3.0 equiv) was added, followed by adding DMAP (22.0 mg, 0.18 mmol, 1.2 equiv) and DCC (92.9 mg, 0.45 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 = 40:1 to 20:1) to afford the product **Fmoc-Ala-OTAG4** as a colorless oil in 78% yield (153.4 mg).

Rf = 0.57 (hexanes/EtOAc = 5:1).

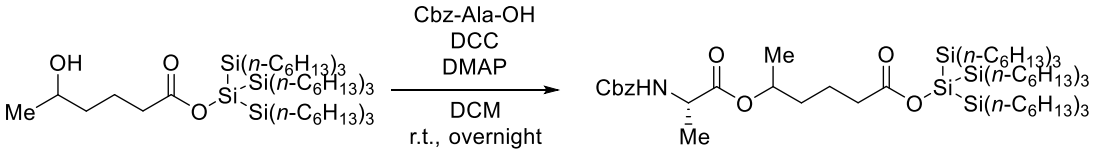
$[\alpha]_D^{21}$ = -9.43 (*c* 1.06, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 7.5 Hz, 2H), 7.68 – 7.54 (m, 2H), 7.45 – 7.35 (m, 2H), 7.35 – 7.27 (m, 2H), 5.49 – 5.35 (m, 1H), 5.03 – 4.89 (m, 1H), 4.52 – 4.31 (m, 3H), 4.29 – 4.18 (m, 1H), 2.36 – 2.10 (m, 2H), 1.72 – 1.55 (m, 4H), 1.51 – 1.21 (m, 78H), 0.99 – 0.84 (m, 27H), 0.83 – 0.52 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 174.5, 174.4, 172.6, 172.5, 155.55, 155.49, 143.9, 143.8, 141.3(2C), 127.7(2C), 127.0(2C), 125.1(2C), 119.9(2C), 72.1(2C), 67.0(2C), 49.74, 49.69, 47.2(2C), 35.6(2C), 35.4, 35.3, 33.9(2C), 31.6(2C), 25.0(2C), 23.1(2C), 22.7(2C), 21.04, 20.99, 18.92, 18.87, 14.1(4C).

IR (neat) 2955, 2920, 2871, 2854, 1727, 1704, 1451, 1378, 1334, 1252, 1205, 1182, 1132, 1100, 1071 cm^{-1}.

HRMS (ESI) Calcd for $C_{78}H_{143}NO_6Si_4Na$ $[M+Na]^+$: 1324.9890, Found: 1324.9890.



1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl 5-((((benzyloxy)carbonyl)-L-alanyl)oxy)hexanoate (Cbz-Ala-OTAG4) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG4** (394.5 mg, 0.39 mmol, 1.0 equiv) was added dichloromethane (4 mL). The Cbz-Ala-OH (217.7 mg, 0.98 mmol, 2.5 equiv) was added, followed by adding DMAP (57.2 mg, 0.47 mmol, 1.2 equiv) and DCC (201.2 mg, 0.98 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 = 40:1 to 20:1) to afford the product **Cbz-Ala-OTAG4** as a colorless oil in 94% yield (445.5 mg).

Rf = 0.57 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{20}$ = -7.45 (*c* 0.94, $CHCl_3$).

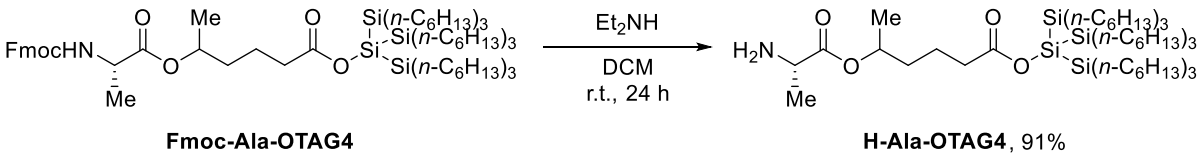
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.28 (m, 5H), 5.43 – 5.29 (m, 1H), 5.19 – 5.06 (m, 2H), 5.00 – 4.88 (m, 1H), 4.41 – 4.24 (m, 1H), 2.33 – 2.13 (m, 2H), 1.66 – 1.49 (m, 4H), 1.46 – 1.17 (m, 78H), 0.98 – 0.82 (m, 27H), 0.82 – 0.49 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 174.5, 174.4, 172.5, 172.4, 155.5(2C), 136.3(2C), 128.5(2C), 128.11(2C), 128.08(2C), 72.1, 72.0, 66.8(2C), 49.8(2C), 35.6(2C), 35.4, 35.3, 33.9(2C), 31.6(2C), 25.0(2C), 23.1(2C), 22.7(2C), 21.02, 20.97, 18.91, 18.85, 14.1(4C).

IR (neat) 2955, 2920, 2872, 2854, 1727, 1706, 1456, 1378, 1335, 1308, 1254, 1205, 1181, 1132, 1098, 1066 cm^{-1}.

HRMS (ESI) Calcd for $C_{71}H_{139}NO_6Si_4Na$ $[M+Na]^+$: 1236.9577, Found: 1236.9547.

Tolerance test.



1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl 5-((L-alanyl)oxy)hexanoate (H-Ala-OTAG4) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAG4** (124.9 mg, 0.096 mmol, 1.0 equiv) was added

dichloromethane (1 mL). The diethylamine (99.1 μL, 0.96 mmol, 10.0 equiv) was added. The resulting mixture was stirred under room temperature for 24 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1 to 5:1) to afford the product **H-Ala-OTAG4** as a pale yellow oil in 91% yield (94.5 mg).

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

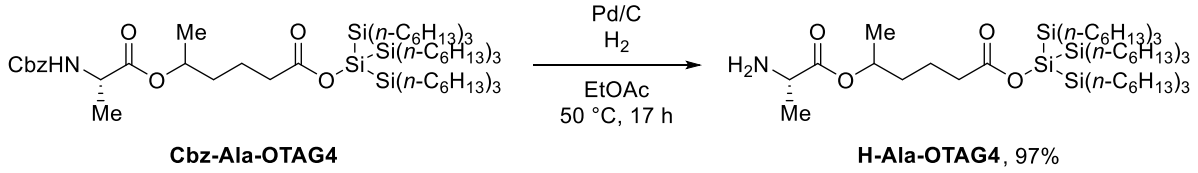
$[\alpha]_D^{24}$ = +4.76 (*c* 1.05, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.99 – 4.85 (m, 1H), 3.54 – 3.45 (m, 1H), 2.27 – 2.14 (m, 2H), 1.66 – 1.52 (m, 4H), 1.46 – 1.18 (m, 78H), 1.00 – 0.81 (m, 27H), 0.78 – 0.50 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 176.2, 176.1, 174.55, 174.51, 71.2, 71.1, 50.2, 50.1, 35.8, 35.7, 35.4(2C), 33.9(2C), 31.6(2C), 25.0(2C), 22.7(2C), 21.09, 21.06, 20.7, 20.6, 19.8, 19.7, 14.1(4C).

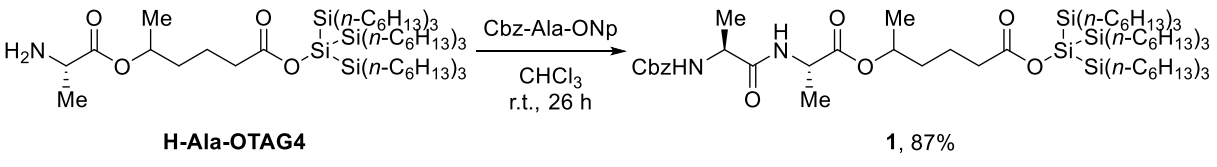
IR (neat) 2956, 2920, 2871, 2854, 1735, 1703, 1458, 1377, 1255, 1183, 1100 cm^{-1}.

HRMS (ESI) Calcd for $C_{63}H_{133}NO_4Si_4Na$ $[M+Na]^+$: 1102.9209, Found: 1102.9225.



1,1,1,3,3,3-Hexahexyl-2-(trihexylsilyl)trisilan-2-yl 5-((L-alanyl)oxy)hexanoate (H-Ala-OTAG4) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **Cbz-Ala-OTAG4** (442.5 mg, 0.36 mmol, 1.0 equiv) was added EtOAc (4 mL). The 10% Pd/C (38.7 mg, 0.036 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 17 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 = 3:1) to afford the product **H-Ala-TAG4** as a pale yellow oil in 97% yield (380.6 mg).

Elongation test (dipeptide synthesis).



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** (97.3 mg, 0.09 mmol, 1.0 equiv) was added chloroform (0.2 mL). The Cbz-Ala-ONp (62.0 mg, 0.18 mmol, 2.0 equiv) was added. The resulting mixture was stirred under room temperature for 26 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 87% yield (101.0 mg).

Rf = 0.27 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{24}$ = +1.04 (*c* 0.96, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 5H), 6.58 – 6.43 (m, 1H), 5.50 – 5.30 (m, 1H), 5.19 – 5.06 (m, 2H), 5.00 – 4.86 (m, 1H), 4.57 – 4.44 (m, 1H), 4.31 – 4.17 (m, 1H), 2.30 – 2.15 (m, 2H), 1.64 – 1.52 (m, 4H), 1.41 – 1.20 (m, 81H), 0.93 – 0.84 (m, 27H), 0.78 – 0.52 (m, 18H).

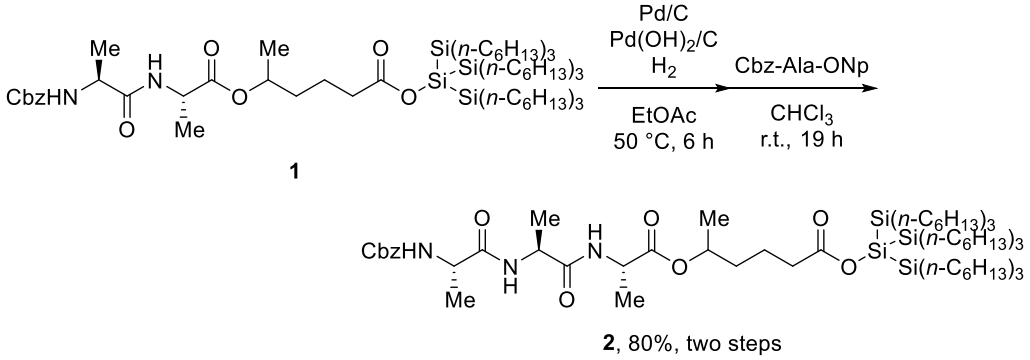
^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 174.5, 174.4, 172.23, 172.16, 171.6, 171.5, 155.8(2C), 136.2(2C), 128.5(2C), 128.2(2C), 128.0(2C), 72.3, 72.2, 67.0(2C), 50.4(2C), 48.32, 48.28, 35.6(2C), 35.4, 35.3, 33.9(2C), 31.6(2C), 25.0(2C), 23.1(2C), 22.7(2C), 21.01, 20.96, 18.7(2C), 18.42, 18.35, 14.1(4C).

IR (neat) 3302, 2956, 2920, 2871, 2854, 1735, 1703, 1665, 1532, 1455, 1254, 1184 cm^{-1}.

HRMS (ESI) Calcd for $C_{74}H_{144}N_2O_7Si_4Na$ $[M+Na]^+$: 1307.9948, Found: 1307.9936.

VII. 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** (135.1 mg, 0.105 mmol, 1.0 equiv) was added EtOAc (1 mL). The 10% Pd/C (11.2 mg, 0.0105 mmol, 0.1 equiv) and 20% $Pd(OH)_2/C$ (5.6 mg, 0.0105 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 (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 (0.2 mL). The Cbz-Ala-ONp (72.3 mg, 0.21 mmol, 2.0 equiv) was added. The reaction was stirred under room temperature for 19 h. After completion, the mixture was diluted with dichloromethane (4 mL), followed by adding 2-aminoethanol (55 μL) and stirring under room temperature for 30 min to remove the excess Cbz-Ala-ONp. 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 = 5:1 to 2:1) to afford the product **2** as a pale yellow wax in 80% total yield (113.7 mg).

Rf = 0.62 (hexanes/EtOAc = 1:1).

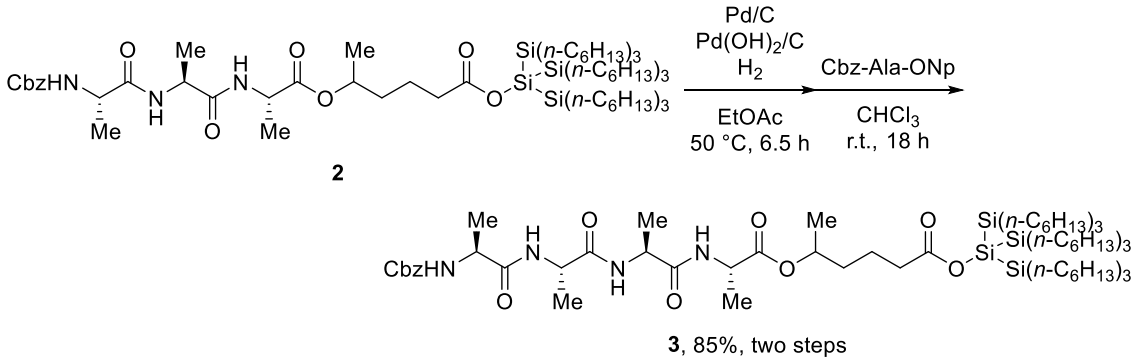
$[\alpha]_D^{23}$ = -9.26 (*c* 1.08, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 6.95 – 6.74 (m, 2H), 5.58 (d, J = 7.3 Hz, 1H), 5.17 – 5.06 (m, 2H), 5.00 – 4.83 (m, 1H), 4.60 – 4.42 (m, 2H), 4.40 – 4.21 (m, 1H), 2.35 – 2.09 (m, 2H), 1.62 – 1.48 (m, 4H), 1.40 – 1.16 (m, 84H), 0.93 – 0.83 (m, 27H), 0.78 – 0.51 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 174.5, 174.4, 172.2, 172.1(3C), 171.4, 171.3, 155.9(2C), 136.2(2C), 128.5(2C), 128.1(2C), 128.0(2C), 72.2, 72.1, 67.0(2C), 50.5(2C), 48.8(2C), 48.33, 48.26, 35.6(2C), 35.4, 35.3, 33.9(2C), 31.6(2C), 24.9(2C), 23.1(2C), 22.7(2C), 21.0, 20.9, 18.9(2C), 18.5(2C), 18.3, 18.2, 14.1(4C).

IR (neat) 3289, 2956, 2920, 2871, 2854, 1736, 1704, 1682, 1637, 1530, 1455, 1377, 1254, 1212, 1182, 1099, 1052 cm^{-1}.

HRMS (ESI) Calcd for $C_{77}H_{149}N_3O_8Si_4Na$ $[M+Na]^+$: 1379.0319, Found: 1379.0330.



Cbz-Ala-Ala-Ala-Ala-OTAG4 (3) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **2** (101.5 mg, 0.075 mmol, 1.0 equiv) was added EtOAc (1 mL). The 10% Pd/C (8.0 mg, 0.0075 mmol, 0.1 equiv) and 20% $Pd(OH)_2/C$ (4.0 mg, 0.0075 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 6.5 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 (0.2 mL). The Cbz-Ala-ONp (51.5 mg, 0.15 mmol, 2.0 equiv) was added. The reaction was stirred under room temperature for 18 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 = 3:1 to 1:1 to DCM/EtOAc = 1:1) to afford the product **3** as a light grey solid in 85% total yield (91.1 mg).

Rf = 0.16 (hexanes/EtOAc = 1:1).

M.p. 178-180 ºC.

$[\alpha]_D^{24}$ = -10.64 (*c* 0.94, $CHCl_3$).

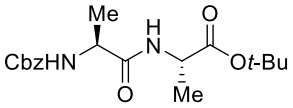
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.86 (m, 1H), 7.59 – 7.37 (m, 2H), 7.37 – 7.26 (m, 5H), 6.29 – 6.13 (m, 1H), 5.18 – 5.05 (m, 2H), 4.95 – 4.81 (m, 2H), 4.81 – 4.70 (m, 1H), 4.71 – 4.57 (m, 1H), 4.56 – 4.45 (m, 1H), 2.29 – 2.09 (m, 2H), 1.60 – 1.49 (m, 4H), 1.47 – 1.09 (m, 87H), 0.95 – 0.82 (m, 27H), 0.81 – 0.52 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$, two isomers) δ 174.5, 174.4, 172.2(4C), 171.8(4C), 156.0(2C), 136.4(2C), 128.5(2C), 128.0(2C), 127.8(2C), 72.0, 71.9, 66.7(2C), 50.4(2C), 49.0(2C), 48.8(2C), 48.24, 48.18, 35.6(2C), 35.4, 35.3, 33.9(2C), 31.6(2C), 25.0(2C), 23.1(2C), 22.7(2C), 21.01, 20.97, 20.1(2C), 19.9(2C), 19.7(2C), 18.2(2C), 14.1(4C).

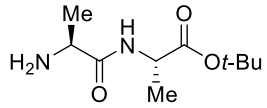
IR (neat) 3273, 2958, 2920, 2871, 2854, 1736, 1707, 1674, 1631, 1528, 1454, 1376, 1259, 1216, 1181, 1095, 1047, 1027 cm^{-1}.

HRMS (ESI) Calcd for $C_{80}H_{154}N_4O_9Si_4Na$ $[M+Na]^+$: 1450.0691, Found: 1450.0681.

Short alanine chain with *t*-Bu.



4



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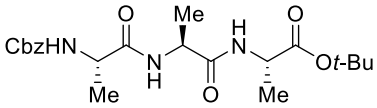

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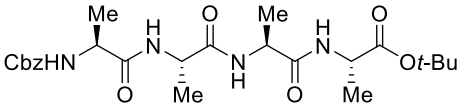
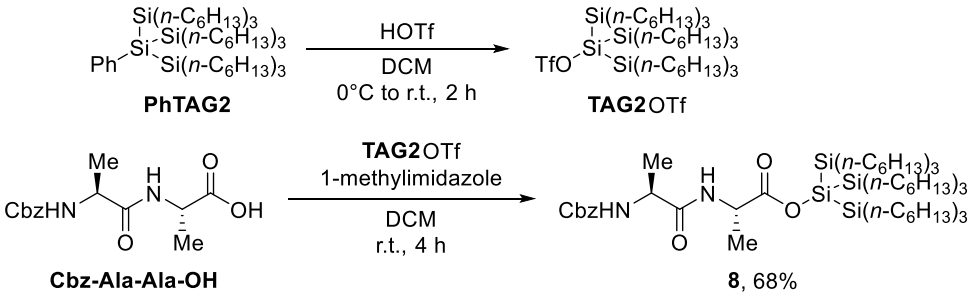


7

Cbz-Ala-Ala-O*t*-Bu (4), **H-Ala-Ala-O*t*-Bu (5)**, **Cbz-Ala-Ala-Ala-O*t*-Bu (6)** and **Cbz-Ala-Ala-Ala-Ala-O*t*-Bu (7)** were prepared according to the procedure in the literatures.[2]

VIII. Installing TAG2 at the C-terminal starting from dipeptide



Cbz-Ala-Ala-OTAG2 (8) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (60.7 μL, 0.69 mmol, 1.96 equiv). The **PhTAG2** (468.4 mg, 0.49 mmol, 1.4 equiv) was added. The reaction was stirred for 2 h. Then, mixed Cbz-Ala-Ala-OH (103.0 mg, 0.35 mmol, 1.0 equiv) and 1-methylimidazole (55.3 μL, 0.7 mmol, 2.0 equiv) in dichloromethane (3 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After stirring under room temperature for 4 h, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 20:1 to 10:1) to afford the product **8** as a colorless oil in 68% yield (227.4 mg).

Rf = 0.41 (hexanes/EtOAc = 5:1).

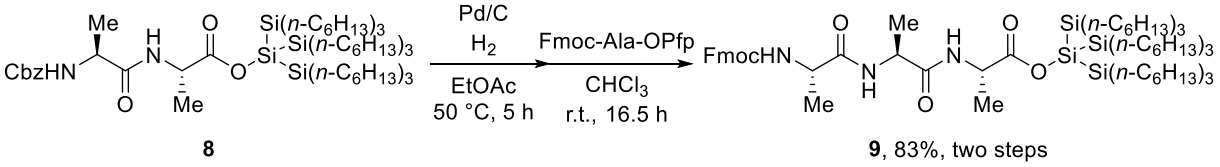
$[\alpha]_D^{22}$ = -11.70 (*c* 0.94, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 6.43 (d, *J* = 7.0 Hz, 1H), 5.45 (d, *J* = 7.6 Hz, 1H), 5.18 – 5.02 (m, 2H), 4.46 – 4.32 (m, 1H), 4.30 – 4.16 (m, 1H), 1.41 – 1.22 (m, 78H), 0.93 – 0.84 (m, 27H), 0.78 – 0.69 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 171.1, 155.6, 136.3, 128.5, 128.1, 128.0, 66.8, 50.4, 49.5, 33.9, 31.6, 25.0, 22.7, 19.3, 18.6, 14.1, 14.0.

IR (neat) 2956, 2920, 2871, 2854, 1713, 1669, 1502, 1455, 1377, 1352, 1306, 1209, 1185, 1147 cm^{-1}.

HRMS (ESI) Calcd for $C_{68}H_{134}N_2O_5Si_4Na$ $[M+Na]^+$: 1193.9268, Found: 1193.9241.



Fmoc-Ala-Ala-Ala-OTAG2 (9) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **8** (117.2 mg, 0.1 mmol, 1.0 equiv) was added EtOAc (2

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 a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 °C for 5 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 (0.2 mL). The Fmoc-Ala-OPfp (57.3 mg, 0.12 mmol, 1.2 equiv) was added. The reaction was stirred under room temperature for 16.5 h. After completion, the mixture was diluted with dichloromethane (4 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 = 5:1 to 3:1 to 2:1) to afford the product **9** as a pale white oil in 83% total yield (110.5 mg).

Rf = 0.62 (hexanes/EtOAc = 2:1).

$[\alpha]_D^{22}$ = -10.38 (*c* 1.06, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, J = 7.5 Hz, 2H), 7.65 – 7.55 (m, 2H), 7.44 – 7.36 (m, 2H), 7.36 – 7.26 (m, 2H), 6.59 (d, J = 7.2 Hz, 1H), 6.49 (d, J = 6.7 Hz, 1H), 5.44 (d, J = 7.7 Hz, 1H), 4.49 – 4.32 (m, 4H), 4.32 – 4.17 (m, 2H), 1.50 – 1.24 (m, 81H), 0.96 – 0.85 (m, 27H), 0.84 – 0.67 (m, 18H).

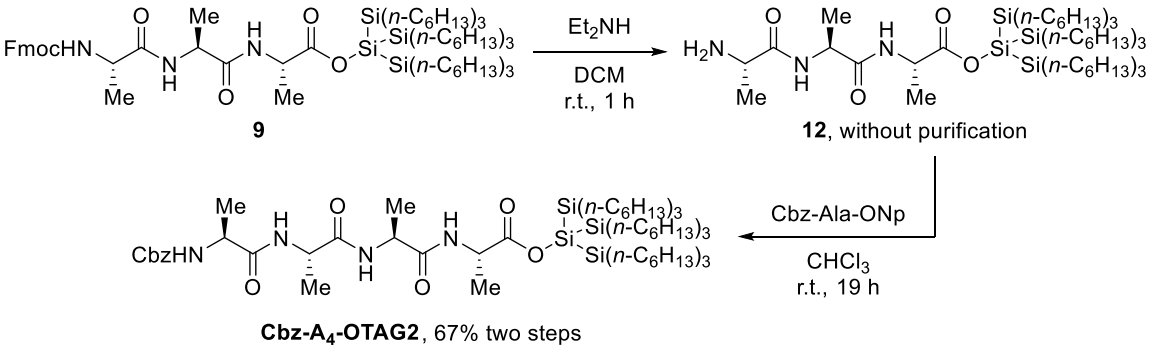
^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 171.6, 170.8, 155.7, 143.84, 143.76, 141.3, 127.7, 127.0, 125.1, 120.0, 67.0, 50.4, 49.5, 48.9, 47.1, 33.9, 31.6, 25.0, 22.7, 19.1, 18.9, 18.5, 14.1, 14.0.

IR (neat) 3302, 2956, 2920, 2871, 2854, 1707, 1644, 1521, 1451, 1411, 1377, 1335, 1306, 1210, 1167, 1100, 1076, 1043 cm^{-1}.

HRMS (ESI) Calcd for $C_{78}H_{143}N_3O_6Si_4Na$ $[M+Na]^+$: 1352.9952, Found: 1352.9982.

IX. Solubility comparison of the of the tagged peptides in CPME

Compounds preparation.



Cbz-A$_4$-OTAG2 At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **9** (43.5 mg, 0.033 mmol) was added dichloromethane (0.33 mL) and diethylamine (0.34 mL). The reaction was stirred at room temperature for 1 h. After completion, the reaction mixture was concentrated to remove the solvent and the excess diethylamine to afford the crude product **12**. It was put into the following coupling step without further purification.

At room temperature, to a flame-dried 6 mL vial charged with magnetic stirring bar (Sm-Co) and above residue was added chloroform (0.065 mL). The Cbz-Ala-ONp (22.5 mg, 0.065 mmol, 2.0 equiv) was added. The reaction was stirred under room temperature for 19 h. After completion, the mixture was diluted with dichloromethane (4 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 dried with anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 2:1 to 1:1) to afford the product **Cbz-A$_4$-OTAG2** as a pale white semi-oil in 67% total yield (28.9 mg).

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

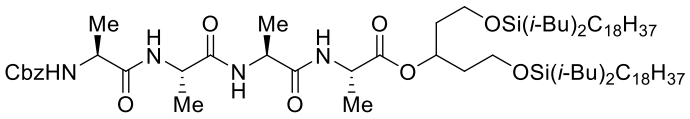
$[\alpha]_D^{24}$ = -22.88 (*c* 1.18, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.27 (m, 5H), 6.83 (d, *J* = 6.8 Hz, 1H), 6.71 (d, *J* = 7.3 Hz, 1H), 6.59 (d, *J* = 6.9 Hz, 1H), 5.44 (d, *J* = 7.5 Hz, 1H), 5.17 – 5.03 (m, 2H), 4.53 – 4.41 (m, 2H), 4.42 – 4.26 (m, 2H), 1.43 – 1.20 (m, 84H), 0.92 – 0.84 (m, 27H), 0.77 – 0.62 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 171.9, 171.3, 170.8, 155.9, 136.2, 128.5, 128.2, 128.1, 67.0, 50.5, 49.5, 48.91, 48.87, 33.9, 31.6, 25.0, 22.7, 19.02, 18.96 18.7, 18.5, 14.1, 14.0.

IR (neat) 3293, 2956, 2921, 2871, 2854, 1709, 1677, 1635, 1525, 1454, 1411, 1377, 1340, 1307, 1211, 1168, 1099, 1048 cm^{-1}.

HRMS (ESI) Calcd for $C_{74}H_{144}N_4O_7Si_4Na$ $[M+Na]^+$: 1336.0010, Found: 1336.0040.



Cbz-A_4-OTAGSimple

Cbz-A_4-OTAGSimple was prepared according to the procedure in the literatures.[2]

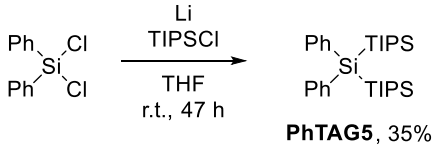
Solubility test.

At room temperature, to a 6 mL vial charged with **Cbz-A_4-OTAG2** (19.5 mg, 0.015 mmol) was added cyclopentyl methyl ether (CPME) 5 μL every time. When the total amount of added CMPE was 20 μL, the pale white semi-oil **Cbz-A_4-OTAG2** still could be seen. After adding another more 5 μL CMPE (total 25 μL), all the pale white semi-oil was disappear to form a colorless clean mixture. From these results, the solubility range of the **Cbz-A_4-OTAG2** in CPME under room temperature could be calculated as 780~975 mg/mL (0.593~0.742 M).

At room temperature, to a 6 mL vial charged with **Cbz-A_4-OTAGSimple** (31.4 mg, 0.024 mmol) was added cyclopentyl methyl ether (CPME) 10 μL every time. When the total amount of added CMPE was 60 μL, the pale yellow wax **Cbz-A_4-OTAGSimple** still could be seen. After adding another more 10 μL CMPE (total 70 μL), all the pale yellow wax was disappear to form a clean mixture. From these results, the solubility range of the **Cbz-A_4-OTAGSimple** in CPME under room temperature could be calculated as 449~523 mg/mL (0.338~0.394 M).

X. Super silyl tags at the N-terminal

Synthesis of TAG5, TAG6, TAG7.



PhTAG5, 35%

1,1,1,3,3,3-Hexaisopropyl-2,2-diphenyltrisilane (PhTAG5) At room temperature, under N_2, to a flame-dried flask charged with Li (granular, 555.2 mg, 80.0 mmol, 8.0 equiv) was added tetrahydrofuran (50 mL). The dichlorodiphenylsilane (2.08 mL, 10.0 mmol, 1.0 equiv) and triisopropylsilyl chloride (6.42 mL, 30.0 mmol, 3.0 equiv) were added together. The reaction was stirred at room temperature for 24 h. Then, another 1.0 equivalent triisopropylsilyl chloride (2.14 mL, 10.0 mmol) was added and stirred for another 23 h. Then, the reaction mixture was poured into a separation funnel charged with 100 mL hexanes and 50 mL water. 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 **PhTAG5** as a white solid in 35% yield (1.72 g). [***Note****: The purification by chromatography should be repeated if the product is not pure enough.*] Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data support it.

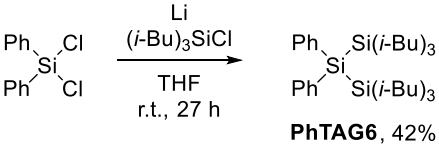
Rf = 0.58 (100% hexanes).

M.p. 110-115 ºC.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 – 7.66 (m, 4H), 7.32 – 7.26 (m, 6H), 1.38 (hept, J = 7.5 Hz, 6H), 1.00 (d, J = 7.5 Hz, 36H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8, 137.1, 127.9, 127.3, 20.1, 13.5.

IR (neat) 2945, 2864, 1463, 1427, 1381, 1364, 1236, 1215 cm^{-1}.



PhTAG6, 42%

1,1,1,3,3,3-Hexaisobutyl-2,2-diphenyltrisilane (PhTAG6) At room temperature, under N_2, to a flame-dried flask charged with Li (granular, 222.1 mg, 32.0 mmol, 8.0 equiv) was added tetrahydrofuran (20 mL). The dichlorodiphenylsilane (0.83 mL, 4.0 mmol, 1.0 equiv) and

chlorotriisobutylsilane (2.15 mL, 8.0 mmol, 2.0 equiv) were added together. The reaction was stirred at room temperature for 23 h. Then, another 0.8 equivalent chlorotriisobutylsilane (0.86 mL, 3.2 mmol) was added and stirred for another 4 h. Then, the reaction mixture was poured into a separation funnel charged with 50 mL hexanes and 30 mL water. The layers were separated. The aqueous layer was extracted with hexanes (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: 100% hexanes) to afford the product **PhTAG6** as a white solid in 42% yield (985.8 mg). [***Note**: The purification by chromatography should be repeated if the product is not pure enough.*] Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data and structure of installed compounds support it.

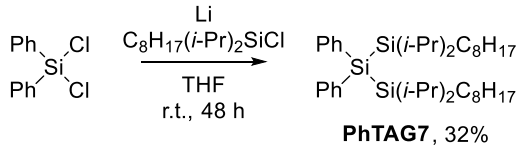
Rf = 0.53 (100% hexanes).

M.p. 58-61 ºC.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.46 (m, 4H), 7.32 – 7.26 (m, 6H), 1.80 – 1.65 (m, 6H), 0.93 – 0.78 (m, 48H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 137.2, 136.4, 127.9, 127.5, 26.8, 25.6, 25.5.

IR (neat) 2950, 2924, 2900, 2866, 1462, 1427, 1400, 1380, 1363, 1325, 1218, 1161 cm^{-1}.



PhTAG7, 32%

1,1,3,3-Tetraisopropyl-1,3-dioctyl-2,2-diphenyltrisilane (PhTAG7) At room temperature, under N_2, to a flame-dried flask charged with Li (granular, 555.2 mg, 80.0 mmol, 8.0 equiv) was added tetrahydrofuran (50 mL). The dichlorodiphenylsilane (2.08 mL, 10.0 mmol, 1.0 equiv) and chlorodiisopropyloctylsilane (6.61 mL, 22.0 mmol, 2.2 equiv) were added together. The reaction was stirred at room temperature for 24 h. Then, another 1.0 equivalent chlorodiisopropyloctylsilane (3.00 mL, 10.0 mmol) was added and stirred for another 24 h. Then, the reaction mixture was poured into a separation funnel charged with 100 mL hexanes and 50 mL water. 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 **PhTAG7** as a colorless oil in 32% yield (2.05 g). [***Note**: The purification by chromatography should be repeated if the product is not pure enough.*] Although molecular

ion peak was not located in the mass spectrum in ESI equipment, NMR data and structure of installed compounds support it.

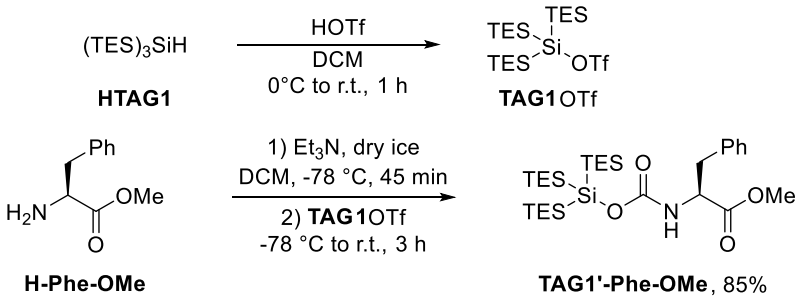
Rf = 0.62 (100% hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.60 (m, 4H), 7.35 – 7.26 (m, 6H), 1.34 – 1.18 (m, 28H), 1.06 – 0.81 (m, 34H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 136.8, 127.9, 127.4, 34.4, 31.9, 29.3, 29.2, 25.1, 22.7, 19.9, 19.7, 14.1, 13.4, 12.3.

IR (neat) 2922, 2862, 1461, 1427, 1380, 1364, 1300, 1238, 1175 cm^{-1}.

Synthesis of stable super silyl carbamates (TAG1', TAG2', TAG6', TAG7').



Methyl (((1,1,1,3,3,3-hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)carbonyl)-L-phenylalaninate (TAG1'-Phe-OMe) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (3 mL) was added triflic acid (88.5 μL, 1.0 mmol, 1.0 equiv). The tris(triethylsilyl)silane (422.7 μL, 1.0 mmol, 1.0 equiv) was added. The reaction was stirred at room temperature for 1 h to make **TAG1**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (7 mL) was added **H-Phe-OMe** (179.2 mg, 1.0 mmol, 1.0 equiv) and triethylamine (167.3 μL, 1.2 mmol, 1.2 equiv). The mixture was cooled to -78 °C. With a steam of N_2 over the solution, dry ice (1.54 g, 35.0 mmol, 35.0 equiv) was added. After stirring at this temperature for 45 min, **TAG1**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1 to 50:1 to 20:1) to afford the product **TAG1'-Phe-OMe** as a white solid in 85% yield (505.5 mg). Two rotamers of the carbamate were observed in a ratio of 4:1.

Rf = 0.15 (hexanes/EtOAc = 20:1).

M.p. 52-54 °C.

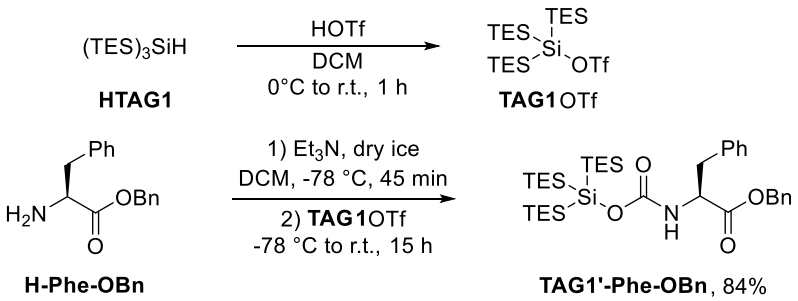
$[\alpha]_D^{19}$ = +26.73 (*c* 1.01, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$, major rotamer) δ 7.30 – 7.19 (m, 3H), 7.13 – 7.06 (m, 2H), 4.95 (d, J = 8.5 Hz, 1H), 4.63 (dt, J = 8.5, 5.9 Hz, 1H), 3.68 (s, 3H), 3.14 – 2.97 (m, 2H), 1.07 – 0.95 (m, 27H), 0.84 – 0.72 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 155.7, 136.0, 129.2, 128.4, 126.9, 54.9, 52.1, 38.3, 8.5, 5.0.

IR (neat) 2951, 2907, 2874, 1751, 1685, 1496, 1456, 1435, 1414, 1369, 1204, 1177, 1141 cm^{-1}.

HRMS (ESI) Calcd for $C_{29}H_{57}NO_4Si_4Na$ $[M+Na]^+$: 618.3262, Found: 618.3244.



Benzyl (((1,1,1,3,3,3-hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)carbonyl)-L-phenylalaninate (TAG1'-Phe-OBn) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (3 mL) was added triflic acid (88.5 μL, 1.0 mmol, 1.0 equiv). The tris(triethylsilyl)silane (422.7 μL, 1.0 mmol, 1.0 equiv) was added. The reaction was stirred at room temperature for 1 h to make **TAG1**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (7 mL) was added **H-Phe-OBn** (255.3 mg, 1.0 mmol, 1.0 equiv) and triethylamine (167.3 μL, 1.2 mmol, 1.2 equiv). The mixture was cooled to -78 °C. With a steam of N_2 over the solution, dry ice (1.54 g, 35.0 mmol, 35.0 equiv) was added. After stirring at this temperature for 45 min, **TAG1**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 15 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1 to 50:1 to 20:1) to afford the product **TAG1'-Phe-OBn** as a colorless oil in 84% yield (565.7 mg). Two rotamers of the carbamate were observed in a ratio of 4:1.

Rf = 0.16 (hexanes/EtOAc = 20:1).

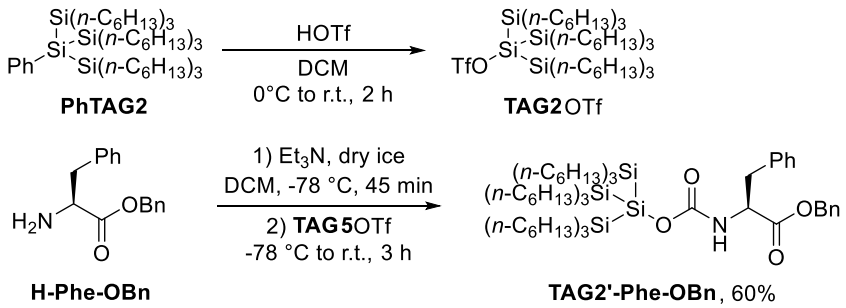
$[\alpha]_D^{22}$ = +40.74 (*c* 0.81, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$, major rotamer) δ 7.41 – 7.32 (m, 3H), 7.32 – 7.23 (m, 2H), 7.23 – 7.15 (m, 3H), 7.06 – 6.97 (m, 2H), 5.18 – 5.05 (m, 2H), 4.98 (d, J = 8.7 Hz, 1H), 4.69 (dt, J = 8.7, 5.9 Hz, 1H), 3.13 – 2.92 (m, 2H), 1.08 – 0.95 (m, 27H), 0.85 – 0.71 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 155.6, 135.8, 135.2, 129.3, 129.2, 128.5, 128.42, 128.37, 126.9, 67.0, 54.9, 38.4, 8.5, 5.0.

IR (neat) 2951, 2907, 2874, 1745, 1686, 1496, 1456, 1415, 1379, 1234, 1172 cm^{-1}.

HRMS (ESI) Calcd for $C_{35}H_{61}NO_4Si_4Na$ $[M+Na]^+$: 694.3575, Found: 694.3569.



Benzyl (((1,1,1,3,3,3-hexahexyl-2-(trihexylsilyl)trisilan-2-yl)oxy)carbonyl)-L-phenylalaninate (TAG2'-Phe-OBn) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (86.7 μL, 0.98 mmol, 1.96 equiv). The **PhTAG2** (669.2 mg, 0.7 mmol, 1.4 equiv) was added. The reaction was stirred at room temperature for 2 h to make **TAG2**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (4 mL) was added **H-Phe-OBn** (127.7 mg, 0.5 mmol, 1.0 equiv) and triethylamine (153.3 μL, 1.1 mmol, 2.2 equiv). The mixture was cooled to -78 °C. With a steam of N_2 over the solution, dry ice (0.77 g, 17.5 mmol, 35.0 equiv) was added. After stirring at this temperature for 45 min, **TAG2**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1) to afford the product **TAG2'-Phe-OBn** as a pale white oil in 60% yield (354.4 mg). Two rotamers of the carbamate were observed in a ratio of 3.2:1.

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

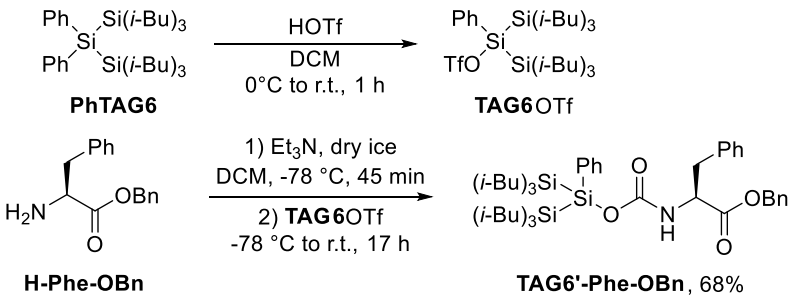
$[\alpha]_D^{23}$ = +37.38 (*c* 1.07, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$, major rotamer) δ 7.39 – 7.30 (m, 3H), 7.29 – 7.22 (m, 2H), 7.22 – 7.12 (m, 3H), 7.04 – 6.89 (m, 2H), 5.16 – 4.92 (m, 3H), 4.75 – 4.64 (m, 1H), 3.19 – 2.90 (m, 2H), 1.38 – 1.24 (m, 72H), 0.93 – 0.83 (m, 27H), 0.82 – 0.66 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 155.5, 135.8, 135.2, 129.4, 128.52, 128.48, 128.4, 128.3, 126.9, 66.9, 54.8, 38.7, 33.9, 31.6, 25.0, 22.7, 14.1, 14.0.

IR (neat) 2955, 2920, 2871, 2854, 1746, 1692, 1493, 1456, 1378, 1347, 1251, 1233, 1179 cm^{-1}.

HRMS (ESI) Calcd for $C_{71}H_{133}NO_4Si_4Na$ $[M+Na]^+$: 1198.9209, Found: 1198.9162.



Benzyl (((1,1,1,3,3,3-hexaisobutyl-2-phenyltrisilan-2-yl)oxy)carbonyl)-L-phenylalaninate (TAG6'-Phe-OBn) At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (60 μL, 0.68 mmol, 1.35 equiv). The **PhTAG6** (290.6 mg, 0.5 mmol, 1.0 equiv) was added. The reaction was stirred at room temperature for 1 h to make **TAG6**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (4 mL) was added **H-Phe-OBn** (127.7 mg, 0.5 mmol, 1.0 equiv) and triethylamine (97.6 μL, 0.7 mmol, 1.4 equiv). The mixture was cooled to -78 ºC. With a steam of N_2 over the solution, dry ice (0.77 g, 17.5 mmol, 35.0 equiv) was added. After stirring at this temperature for 45 min, **TAG6**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 17 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1 to 50:1 to 20:1) to afford the product **TAG6'-Phe-OBn** as a pale yellow oil in 68% yield (273.1 mg). Two rotamers of the carbamate were observed in a ratio of 4.2:1.

Rf = 0.17 (hexanes/EtOAc = 20:1).

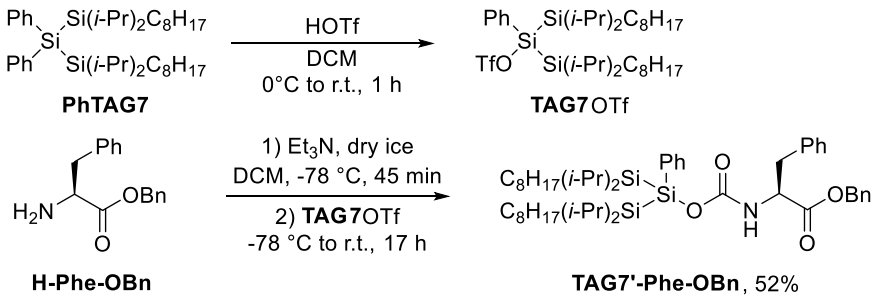
$[\alpha]_D^{24}$ = +36.52 (*c* 1.15, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$, major rotamer) δ 7.46 – 7.34 (m, 5H), 7.34 – 7.19 (m, 8H), 7.12 – 7.02 (m, 2H), 5.26 (d, *J* = 8.5 Hz, 1H), 5.14 (s, 2H), 4.78 (dt, *J* = 8.4, 5.7 Hz, 1H), 3.18 – 3.07 (m, 2H), 1.91 – 1.70 (m, 6H), 1.01 – 0.70 (m, 48H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 154.5, 137.9, 135.8, 135.2, 133.1, 129.4, 128.6, 128.5, 128.4, 128.1, 127.5, 127.0, 67.1, 54.9, 38.5, 26.8, 26.7, 25.5, 24.61, 24.57.

IR (neat) 2951, 2866, 1744, 1695, 1496, 1461, 1428, 1380, 1362, 1328, 1213, 1162 cm^{-1}.

HRMS (ESI) Calcd for $C_{47}H_{75}NO_4Si_3Na$ $[M+Na]^+$: 824.4902, Found: 824.4941.



Benzyl (((1,1,3,3-tetraisopropyl-1,3-dioctyl-2-phenyltrisilan-2-yl)oxy)carbonyl)-L-phenylalaninate (TAG7'-Phe-OBn) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (60 µL, 0.68 mmol, 1.35 equiv). The **PhTAG7** (318.6 mg, 0.5 mmol, 1.0 equiv) was added. The reaction was stirred at room temperature for 1 h to make **TAG7**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (4 mL) was added **H-Phe-OBn** (127.7 mg, 0.5 mmol, 1.0 equiv) and triethylamine (97.6 µL, 0.7 mmol, 1.4 equiv). The mixture was cooled to -78 °C. With a steam of N_2 over the solution, dry ice (0.77 g, 17.5 mmol, 35.0 equiv) was added. After stirring at this temperature for 45 min, **TAG7**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 17 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 80:1 to 50:1 to 20:1) to afford the product **TAG7'-Phe-OBn** as a pale white oil in 52% yield (222.8 mg). Two rotamers of the carbamate were observed in a ratio of 6:1.

Rf = 0.17 (hexanes/EtOAc = 20:1).

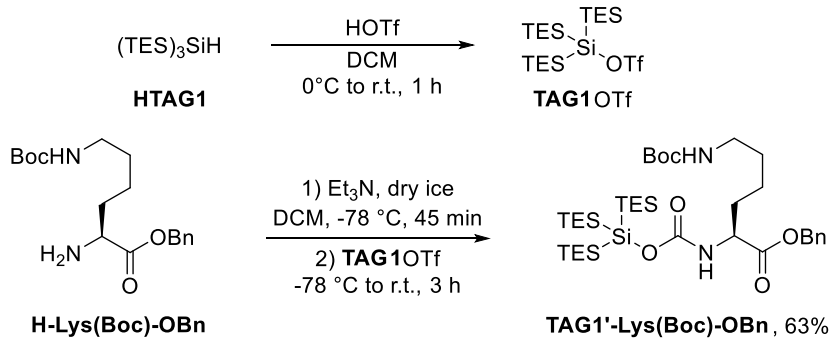
$[\alpha]_D^{24}$ = +38.46 (*c* 1.04, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$, major rotamer) δ 7.52 – 7.43 (m, 2H), 7.42 – 7.33 (m, 3H), 7.33 – 7.18 (m, 8H), 7.13 – 7.02 (m, 2H), 5.26 (d, *J* = 8.7 Hz, 1H), 5.21 – 5.08 (m, 2H), 4.78 (dt, *J* = 8.7, 5.9 Hz, 1H), 3.18 – 3.06 (m, 2H), 1.43 – 1.12 (m, 28H), 1.09 – 0.77 (m, 34H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 154.4, 138.7, 135.8, 135.2, 133.2, 129.4, 128.6, 128.5, 128.4, 128.0, 127.5, 127.0, 67.1, 54.9, 38.5, 34.4, 31.9, 29.3, 29.2, 25.0, 22.7, 19.6, 19.53, 19.47, 19.4, 14.1, 12.8, 12.6, 11.41, 11.36.

IR (neat) 2922, 2861, 1744, 1693, 1496, 1456, 1379, 1349, 1175 cm^{-1}.

HRMS (ESI) Calcd for $C_{51}H_{83}NO_4Si_3Na$ $[M+Na]^+$: 880.5528, Found: 880.5541.



Benzyl N^6-(tert-butoxycarbonyl)-N^2-(((1,1,1,3,3,3-hexaethyl-2-(triethylsilyl)trisilan-2-yl)oxy)carbonyl)-L-lysinate (TAG1'-Lys(Boc)-OBn) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (44.2 μL, 0.5 mmol, 1.0 equiv). The tris(triethylsilyl)silane (211.3 μL, 0.5 mmol, 1.0 equiv) was added. The reaction was stirred at room temperature for 1 h to make **TAG1**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (4 mL) was added **H-Lys(Boc)-OBn** (168.2 mg, 0.5 mmol, 1.0 equiv) and triethylamine (83.6 μL, 0.6 mmol, 1.2 equiv). The mixture was cooled to -78 °C. With a steam of N_2 over the solution, dry ice (770 mg, 17.5 mmol, 35.0 equiv) was added. After stirring at this temperature for 45 min, **TAG1**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 3 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 20:1 to 10:1 to 5:1) to afford the product **TAG1'-Lys(Boc)-OBn** as a colorless oil in 63% yield (238.5 mg).

Rf = 0.21 (hexanes/EtOAc = 5:1).

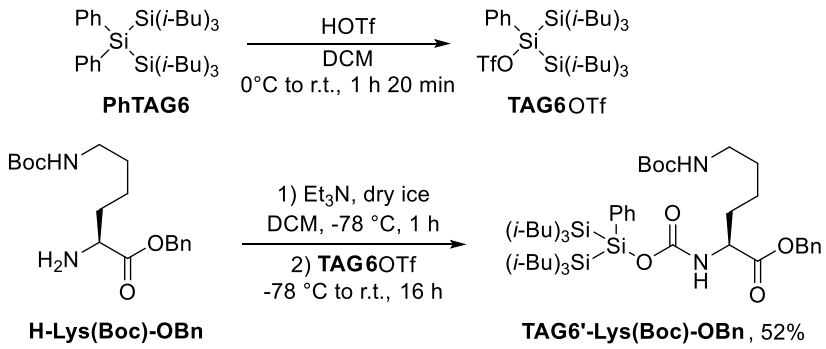
$[\alpha]_D^{22}$ = -14.73 (*c* 1.29, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 5H), 5.21 – 5.07 (m, 2H), 5.02 (d, J = 8.5 Hz, 1H), 4.49 – 4.32 (m, 2H), 3.06 – 2.98 (m, 2H), 1.86 – 1.75 (m, 1H), 1.67 – 1.55 (m, 1H), 1.50 – 1.34 (m, 11H), 1.32 – 1.13 (m, 2H), 1.04 – 0.95 (m, 27H), 0.85 – 0.73 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 156.0, 155.8, 135.4, 128.6, 128.4, 128.2, 79.0, 66.9, 53.9, 40.3, 32.3, 29.6, 28.4, 22.2, 8.5, 5.0.

IR (neat) 2951, 2908, 2874, 1689, 1499, 1456, 1365, 1244, 1169, 1001 cm^{-1}.

HRMS (ESI) Calcd for $C_{37}H_{72}N_2O_6Si_4Na$ $[M+Na]^+$: 775.4365, Found: 775.4376.



Benzyl N^6-(*tert*-butoxycarbonyl)-N^2-(((1,1,1,3,3,3-hexaisobutyl-2-phenyltrisilan-2-yl)oxy)carbonyl)-L-lysinate (TAG6'-Lys(Boc)-OBn) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (47.8 μL, 0.54 mmol, 1.35 equiv). The **PhTAG6** (232.5 mg, 0.4 mmol, 1.0 equiv) was added. The reaction was stirred at room temperature for 1 h 20 min to make **TAG6**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (4 mL) was added **H-Lys(Boc)-OBn** (mg, 0.52 mmol, 1.3 equiv) and triethylamine (83.6 μL, 0.6 mmol, 1.5 equiv). The mixture was cooled to -78 °C. With a steam of N_2 over the solution, dry ice (0.79 g, 18.0 mmol, 45.0 equiv) was added. After stirring at this temperature for 1 h, **TAG6**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 16 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 20:1 to 10:1 to 5:1) to afford the product **TAG6'-Lys(Boc)-OBn** as a colorless oil in 52% yield (185.2 mg).

Rf = 0.36 (hexanes/EtOAc = 5:1).

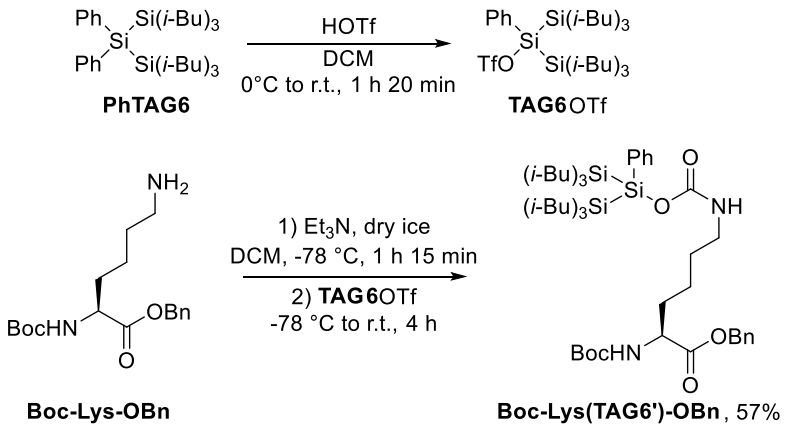
$[\alpha]_D^{24}$ = +4.92 (*c* 0.61, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 2H), 7.39 – 7.33 (m, 5H), 7.33 – 7.26 (m, 3H), 5.31 (d, *J* = 8.5 Hz, 1H), 5.21 – 5.13 (m, 2H), 4.51 – 4.34 (m, 2H), 3.17 – 2.92 (m, 2H), 1.90 – 1.69 (m, 8H), 1.50 – 1.24 (m, 13H), 0.96 – 0.73 (m, 48H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 155.9, 154.8, 137.9, 135.4, 133.2, 128.6, 128.5, 128.3, 128.1, 127.5, 79.1, 67.0, 53.9, 40.3, 32.6, 29.7, 28.4, 26.8, 26.7, 25.5, 24.6, 22.4.

IR (neat) 2952, 2929, 2866, 1698, 1501, 1462, 1428, 1380, 1364, 1327, 1250, 1216, 1171 cm^{-1}.

HRMS (ESI) Calcd for $C_{49}H_{86}N_2O_6Si_3Na$ $[M+Na]^+$: 905.5691, Found: 905.5716.



Benzyl N^2-(*tert*-butoxycarbonyl)-N^6-(((1,1,1,3,3,3-hexaisobutyl-2-phenyltrisilan-2-yl)oxy)carbonyl)-L-lysinate (Boc-Lys(TAG6')-OBn) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (71.7 μL, 0.81 mmol, 1.62 equiv). The **PhTAG6** (348.7 mg, 0.6 mmol, 1.2 equiv) was added. The reaction was stirred at room temperature for 1 h 20 min to make **TAG6**OTf solution. To another flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (4 mL) was added **Boc-Lys-OBn** (168.2 mg, 0.5 mmol, 1.0 equiv) and triethylamine (139.4 μL, 1.0 mmol, 2.0 equiv). The mixture was cooled to -78 °C. With a steam of N_2 over the solution, dry ice (0.77 g, 17.5 mmol, 35.0 equiv) was added. After stirring at this temperature for 1 h 15 min, **TAG6**OTf solution was transferred into the mixture. The mixture was allowed to warm to the room temperature slowly and stir for 4 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 20:1 to 10:1 to 5:1) to afford the product **Boc-Lys(TAG6')-OBn** as a colorless oil in 57% yield (253.4 mg).

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

$[\alpha]_D^{22}$ = -13.26 (*c* 0.98, $CHCl_3$).

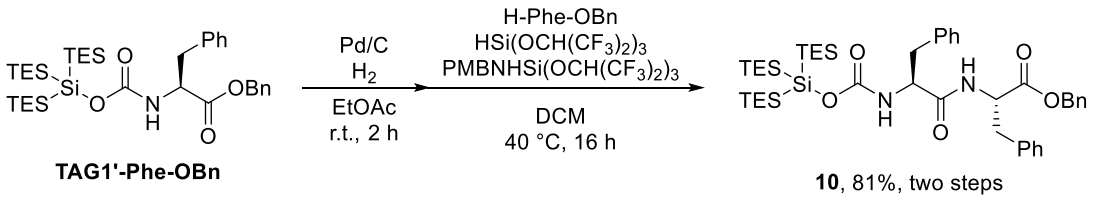
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.41 (m, 2H), 7.41 – 7.26 (m, 8H), 5.25 – 5.10 (m, 2H), 5.01 (d, *J* = 8.6 Hz, 1H), 4.84 – 4.69 (m, 1H), 4.44 – 4.26 (m, 1H), 3.21 – 3.07 (m, 2H), 1.88 – 1.73 (m, 7H), 1.71 – 1.58 (m, 1H), 1.58 – 1.28 (m, 13H), 0.93 – 0.71 (m, 48H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 156.1, 155.4, 138.3, 135.4, 133.2, 128.6, 128.4, 128.3, 128.0, 127.4, 79.9, 67.0, 53.3, 41.1, 32.5, 29.7, 28.3, 26.8, 26.7, 25.4, 24.6, 22.6.

IR (neat) 2951, 2866, 1689, 1500, 1460, 1427, 1392, 1380, 1364, 1326, 1250, 1217, 1160 cm^{-1}.

HRMS (ESI) Calcd for $C_{49}H_{86}N_2O_6Si_3Na$ $[M+Na]^+$: 905.5691, Found: 905.5675.

Elongation test of TAG1'.



TAG1'-Phe-Phe-OBn (10) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **TAG1'-Phe-OBn** (134.4 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) 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 2 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 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and above residue was added dichloromethane (0.4 mL), $HSi[OCH(CF_3)_2]_3$ (159.1 mg, 0.3 mol, 1.5 equiv), **H-Phe-OBn** (76.6 mg, 0.3 mmol, 1.5 equiv) and $PMBNHSi[OCH(CF_3)_2]_3$ (1.0 M in dichloromethane, 6.0 μL, 0.006 mmol, 0.03 equiv) in the glove box. [***Note**: $HSi[OCH(CF_3)_2]_3$ and $PMBNHSi[OCH(CF_3)_2]_3$ 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 ºC for 16 h. After completion, the reaction mixture was transferred onto silica gel column by a pipette and purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 5:1) to afford the product **10** as a pale yellow oil in 81% total yield (133.4 mg).

Rf = 0.47 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{23}$ = +37.50 (*c* 1.04, $CHCl_3$).

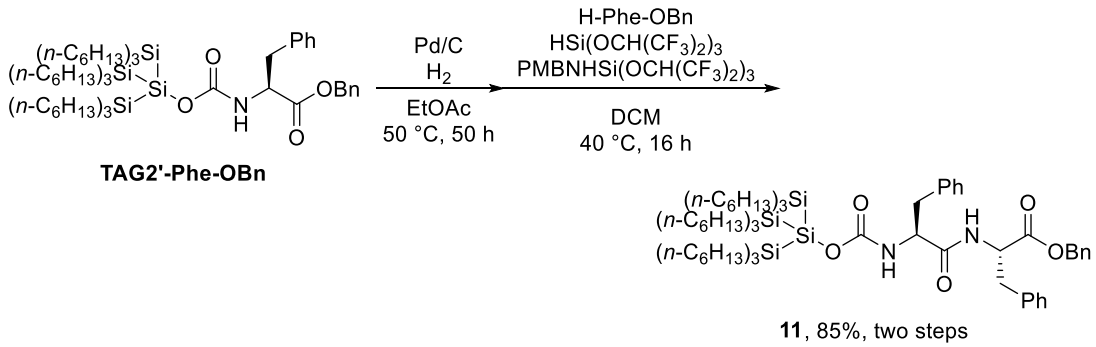
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 3H), 7.30 – 7.20 (m, 5H), 7.20 – 7.10 (m, 5H), 6.90 – 6.83 (m, 2H), 6.21 (d, *J* = 7.6 Hz, 1H), 5.07 (s, 2H), 4.90 (d, *J* = 8.4 Hz, 1H), 4.78 (dt, *J* = 7.6, 5.9 Hz, 1H), 4.39 – 4.29 (m, 1H), 3.07 – 2.88 (m, 4H), 1.04 – 0.94 (m, 27H), 0.83 – 0.72 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$, lost two signals) δ 170.52, 170.47, 156.0, 136.3, 135.4, 135.0, 129.3, 129.2, 128.6, 128.5, 128.4, 127.0, 126.9, 67.1, 56.2, 53.2, 38.3, 37.9, 8.6, 5.0.

IR (neat) 2951, 2908, 2873, 1740, 1661, 1495, 1455, 1416, 1379, 1286, 1212, 1182 cm^{-1}.

HRMS (ESI) Calcd for $C_{44}H_{70}N_2O_5Si_4Na$ $[M+Na]^+$: 841.4260, Found: 841.4214.

XI. Convergent synthesis of a pentapeptide



TAG2'-Phe-Phe-OBn (11) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **TAG2'-Phe-OBn** (117.7 mg, 0.1 mmol, 1.0 equiv) was added EtOAc (1 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 a total of 5 times). The resulting mixture was stirred under hydrogen atmosphere at 50 °C for 50 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 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and above residue was added dichloromethane (0.2 mL), $HSi[OCH(CF_3)_2]_3$ (79.5 mg, 0.15 mol, 1.5 equiv), **H-Phe-OBn** (38.3 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 equiv) in the glove box. [***Note**: $HSi[OCH(CF_3)_2]_3$ and $PMBNHSi[OCH(CF_3)_2]_3$ 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 16 h. After completion, the reaction mixture was transferred onto silica gel column by a pipette and purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1) to afford the product **11** as a white oil in 85% total yield (113.0 mg).

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

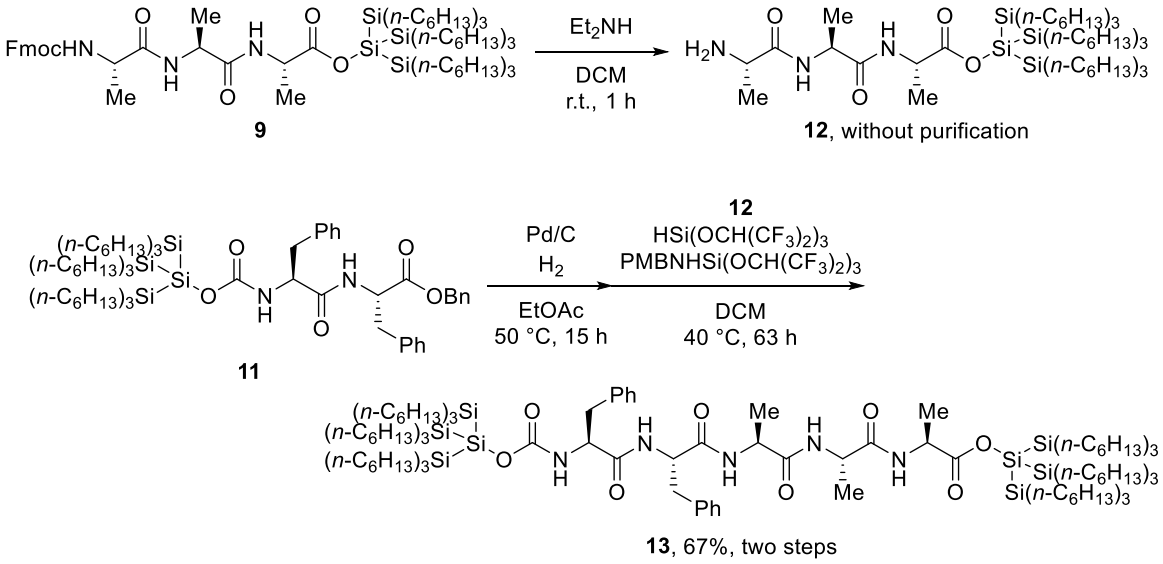
$[\alpha]_D^{23}$ = +37.38 (*c* 1.07, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 3H), 7.25 – 7.12 (m, 10H), 6.95 – 6.84 (m, 2H), 5.95 (d, *J* = 7.4 Hz, 1H), 5.13 (d, *J* = 8.2 Hz, 1H), 5.09 – 4.97 (m, 2H), 4.78 – 4.66 (m, 1H), 4.31 (td, *J* = 8.1, 5.3 Hz, 1H), 3.16 – 2.76 (m, 4H), 1.36 – 1.24 (m, 72H), 0.92 – 0.84 (m, 27H), 0.81 – 0.71 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$, lost three signals) δ 170.4, 170.3, 155.9, 136.4, 135.3, 135.0, 129.4, 129.2, 128.6, 128.5, 127.0, 126.9, 67.0, 56.4, 53.4, 39.1, 38.1, 33.9, 31.6, 25.0, 22.7, 14.1, 14.0.

IR (neat) 2955, 2920, 2871, 2853, 1743, 1678, 1482, 1466, 1456, 1377, 1350, 1256, 1186 cm^{-1}.

HRMS (ESI) Calcd for $C_{80}H_{142}N_2O_5Si_4Na$ $[M+Na]^+$: 1345.9894, Found: 1345.9903.



TAG2'-Phe-Phe-Ala-Ala-Ala-OTAG2 (13) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **9** (107.8 mg, 0.081 mmol) was added dichloromethane (0.8 mL) and diethylamine (0.8 mL). The reaction was stirred at room temperature for 1 h. After completion, the reaction mixture was concentrated to remove the solvent and the excess diethylamine to afford the crude product **12**. It was put into the following coupling step without further purification.

At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **11** (71.4 mg, 0.054 mmol, 1.0 equiv) was added EtOAc (1 mL). The 10% Pd/C (11.5 mg, 0.011 mmol, 0.2 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 coupling 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), $HSi[OCH(CF_3)_2]_3$ (42.9 mg, 0.081 mol, 1.5 equiv), all crude product **12** (dissolved in 0.1 mL dichloromethane, ca. 0.081 mmol, 1.5 equiv) and $PMBNHSi[OCH(CF_3)_2]_3$ (1.0 M in dichloromethane, 2.7 μL, 0.0027 mmol, 0.05 equiv) 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 63 h. After completion, the reaction mixture was transferred onto silica gel column by a pipette and purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 10:1 to 5:1) to afford the product **13** as a pale yellow oil in 67% total yield (83.7 mg). Two rotamers of the carbamate were observed in a ratio of 4:1.

Rf = 0.31 (hexanes/EtOAc = 5:1).

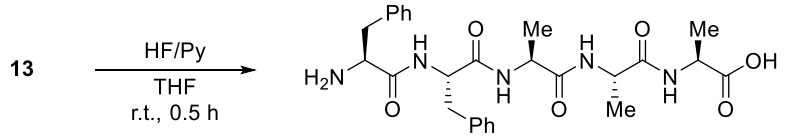
$[\alpha]_D^{24}$ = -15.97 (*c* 1.19, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$, major rotamer) δ 7.26 – 7.04 (m, 10H), 6.55 (d, *J* = 7.3 Hz, 1H), 6.47 (d, *J* = 7.0 Hz, 1H), 6.20 (d, *J* = 7.0 Hz, 1H), 6.07 (d, *J* = 7.2 Hz, 1H), 5.04 (d, *J* = 7.6 Hz, 1H), 4.50 – 4.20 (m, 5H), 3.05 – 2.85 (m, 4H), 1.48 – 1.21 (m, 153H), 0.98 – 0.83 (m, 54H), 0.83 – 0.49 (m, 36H).

^{13}C NMR (100 MHz, $CDCl_3$, lost two signals) δ 173.8, 170.9, 170.8, 170.7, 169.6, 156.1, 136.2, 136.0, 129.1, 128.7, 128.6, 127.1, 56.4, 54.7, 49.5, 48.9, 48.8, 38.7, 38.0, 33.93, 33.88, 31.63, 31.59, 25.0(2C), 22.73, 22.71, 18.7, 18.5, 18.1, 14.1(2C), 14.0(2C).

IR (neat) 2955, 2920, 2871, 2854, 1697, 1637, 1506, 1455, 1377, 1338, 1212, 1174 cm^{-1}.

HRMS (ESI) Calcd for $C_{136}H_{267}N_5O_8Si_8Na$ $[M+Na]^+$: 2345.8691, Found: 2345.8704.



14, >99%

H-Phe-Phe-Ala-Ala-Ala-OH (14) At room temperature, to a flame-dried 6 mL vial charged with magnetic stirring bar (Sm-Co) and **13** (69.8 mg, 0.03 mmol, 1.0 equiv) was added tetrahydrofuran (0.3 mL). The hydrogen fluoride pyridine solution (ca. 70%, 51.5 mg, ca. 1.8 mmol, ca. 60 equiv) was added. The reaction was stirred at room temperature for 0.5 h. After completion, the reaction mixture was concentrated. Dichloromethane (0.5 mL) was added and then removed which was repeated for three times to help remove most of the HF/Py. The residue was washed by hexanes (3×1 mL), followed by diethyl ether (3×1 mL) to remove the silyl fluoride and pyridine. The remaining solid residue was then dissolved in trifluoroacetic acid (TFA) and filtered *via* PTFE syringe filter (0.22 μm) to remove the insoluble solids. The TFA mixture was concentrated and the solid residue was wash again with diethyl ether (3×1 mL) to make sure all the pyridine was removed to afford the product **14** as a white solid in >99% yield (16.1 mg). The purity of the product was 95% which was determined by RP-HPLC using a revised-phase column (XSelect CSH C18, 4.6 mm × 50 mm).

$[\alpha]_D^{25}$ = -24.21 (*c* 1.57, CF_3CO_2H).

^{1}H NMR (400 MHz, CF_3CO_2D) δ 7.32 – 7.25 (m, 3H), 7.24 – 7.18 (m, 3H), 7.18 – 7.10 (m, 2H), 7.08 – 7.01 (m, 2H), 4.87 (t, J = 7.2 Hz, 1H), 4.73 – 4.63 (m, 1H), 4.63 – 4.52 (m, 3H), 3.28 – 3.16 (m, 2H), 3.02 (d, J = 7.3 Hz, 2H), 1.53 (d, J = 7.2 Hz, 3H), 1.49 (d, J = 7.0 Hz, 3H), 1.40 (d, J = 7.0 Hz, 3H).

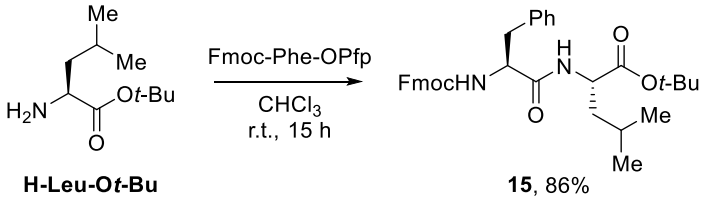
^{13}C NMR (100 MHz, CF_3CO_2D) δ 180.3, 176.7, 176.0, 173.9, 170.9, 136.0, 133.8, 131.6, 131.2, 131.03, 130.98, 130.0, 129.9, 58.1, 57.9, 52.2, 52.0, 51.1, 40.2, 39.1, 18.5(2C), 17.7.

IR (neat) 1671, 1633, 1521, 1454, 1372, 1200, 1139, 1057, 1031 cm^{-1}.

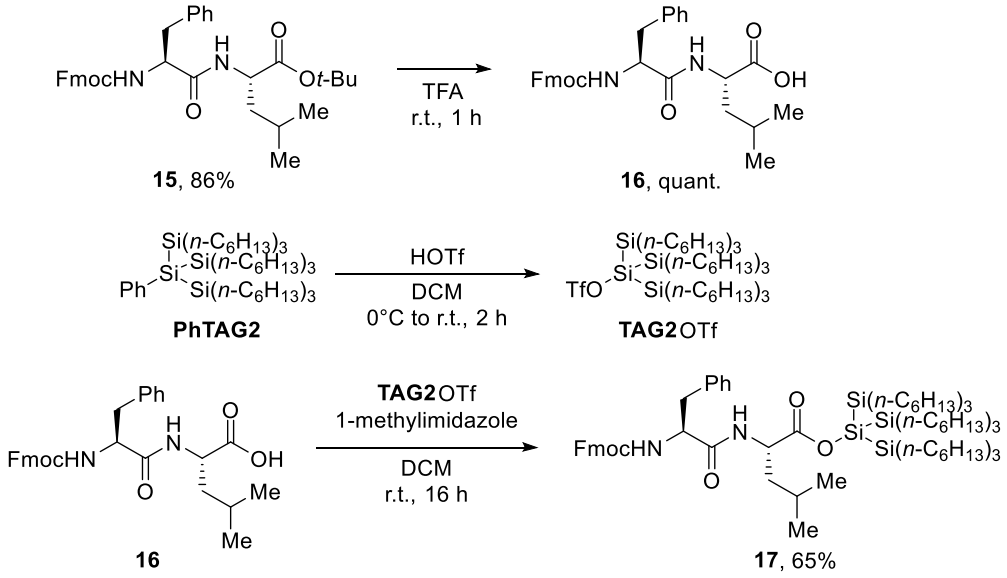
HRMS (ESI) Calcd for $C_{27}H_{35}N_5O_6Na$ $[M+Na]^+$: 548.2485, Found: 548.2525.

XII. Convergent synthesis of Nelipepimut-S with Fmoc-chemistry

Install TAG2 at the C-terminal.



Fmoc-Phe-Leu-O*t*-Bu (15) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and Fmoc-Phe-OPfp (830.2 mg, 1.5 mmol, 1.0 equiv) was added chloroform (1.5 mL). The **H-Leu-O*t*-Bu** (309.0 mg, 1.65 mmol, 1.1 equiv) was added. The resulting mixture was stirred under room temperature for 15 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 = 5:1 to 4:1) to afford the product **15** as a pale yellow solid in 86% yield (714.0 mg). It is a known compound. The characterization data match the reported data.[9]



Fmoc-Phe-Leu-OTAG2 (17) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **15** (167.0 mg, 0.3 mmol, 1.0 equiv) was added trifluoroacetic acid (1.5 mL). After stirring under room temperature for 1 h, the reaction mixture

was concentrated. The residue was co-evaporated several times with diethyl ether to remove all the trifluoroacetic acid to afford the product **16** in >99% yield (153.7 mg).

At 0 ºC, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dichloromethane (2 mL) was added triflic acid (55.7 μL, 0.63 mmol, 2.1 equiv). The **PhTAG2** (430.2 mg, 0.45 mmol, 1.5 equiv) was added. The reaction was stirred for 2 h. Then, mixed **16** (153.7 mg, 0.3 mmol, 1.0 equiv) and 1-methylimidazole (52.1 μL, 0.66 mmol, 2.2 equiv) in dichloromethane (3 mL) in a flame-dried vial. The mixture in the vial was added into the reaction flask slowly. After stirring under room temperature for 16 h, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes to hexanes/EtOAc = 20:1 to 15:1) to afford the product **17** as a colorless oil in 65% yield (269.7 mg).

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

$[\alpha]_D^{24}$ = -10.91 (*c* 1.10, $CHCl_3$).

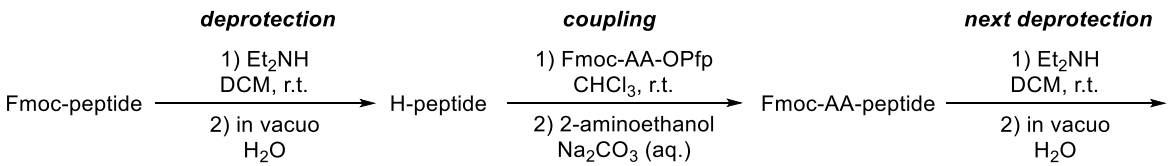
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.6 Hz, 2H), 7.59 – 7.51 (m, 2H), 7.46 – 7.38 (m, 2H), 7.37 – 7.17 (m, 7H), 6.13 (d, *J* = 8.3 Hz, 1H), 5.38 (d, *J* = 8.3 Hz, 1H), 4.60 – 4.37 (m, 3H), 4.34 – 4.23 (m, 1H), 4.18 (t, *J* = 7.0 Hz, 1H), 3.08 (d, *J* = 6.5 Hz, 2H), 1.64 – 1.52 (m, 2H), 1.43 – 1.20 (m, 73H), 0.97 – 0.83 (m, 33H), 0.82 – 0.55 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 170.0, 155.8, 143.83, 143.75, 141.3, 136.4, 129.5, 128.6, 127.7, 127.0, 126.9, 125.1, 125.0, 119.9, 67.0, 55.7, 51.8, 47.1, 42.4, 38.5, 33.9, 31.6, 25.0, 24.8, 23.2, 22.7, 21.5, 14.11, 14.06.

IR (neat) 2955, 2920, 2871, 2854, 1713, 1683, 1499, 1465, 1452, 1410, 1377, 1337, 1271, 1248, 1204, 1152 cm^{-1}.

HRMS (ESI) Calcd for $C_{84}H_{148}N_2O_5Si_4Na$ $[M+Na]^+$: 1400.0363, Found: 1400.0403.

General procedures for Fmoc-deprotection and coupling reactions.

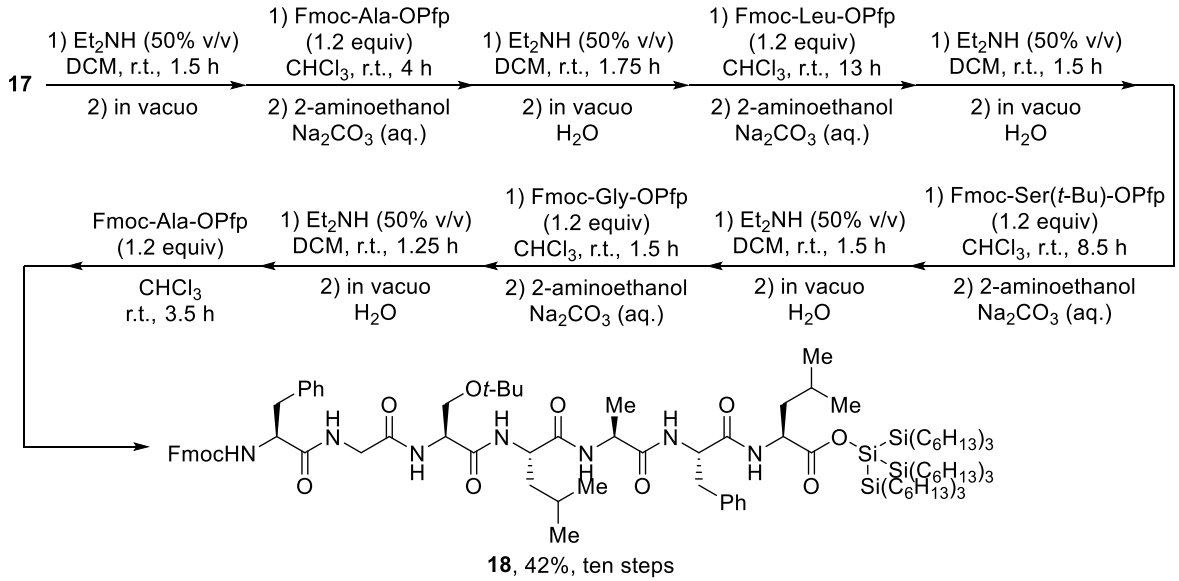


At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and Fmoc-protected peptide (1.0 equiv) was added diethylamine (50 to 100 equiv) and dichloromethane (same volume with diethylamine). The resulting mixture was stirred under room temperature for 1.25 to 1.75 h. After completion (monitored by TLC), the mixture was

concentrated to remove the diethylamine and then dissolved in dichloromethane, followed by adding water. The layers were separated. 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-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 (0.5 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 10 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 from the C-terminal.



18, 42%, ten steps

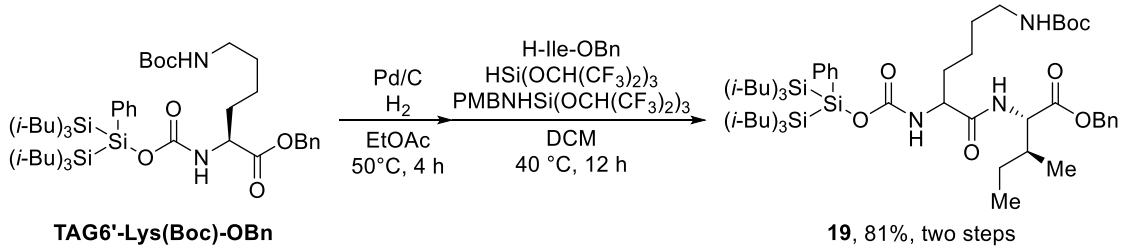
Fmoc-Phe-Gly-Ser(*t*-Bu)-Leu-Ala-Phe-Leu-OTAG2 (18) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and Fmoc-protected peptide **17** (202.5 mg, 0.147 mmol, 1.0 equiv) was added dichloromethane (0.75 mL) and diethylamine (0.75

mL). Then, the mixture was followed the **General procedures for Fmoc-deprotection and coupling reactions** and repeated for four times (five times in total) by changing the coupling active amino acid ester according to the sequence of the target peptide every time. 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 = 5:1 to 2:1 to 1:1 to 1:2) to afford the product **18** as a pale yellow solid in 42% total yield (118.2 mg).

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

HRMS (ESI) Calcd for $C_{111}H_{189}N_7O_{11}Si_4Na$ $[M+Na]^+$: 1931.3420, Found: 1931.3436.

Elongation from the N-terminal.



TAG6'-Lys(Boc)-OBn **19**, 81%, two steps

TAG6'-Lys(Boc)-Ile-OBn (19) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **TAG6'-Lys(Boc)-OBn** (150.1 mg, 0.17 mmol, 1.0 equiv) was added EtOAc (2 mL). The 10% Pd/C (18.1 mg, 0.017 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 4 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 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and above residue was added dichloromethane (0.34 mL), $HSi[OCH(CF_3)_2]_3$ (135.1 mg, 0.26 mol, 1.5 equiv), **H-Ile-OBn** (56.4 mg, 0.26 mmol, 1.5 equiv) and $PMBNHSi[OCH(CF_3)_2]_3$ (1.0 M in dichloromethane, 8.5 μL, 0.0085 mmol, 0.05 equiv) in the glove box. [***Note**: $HSi[OCH(CF_3)_2]_3$ and $PMBNHSi[OCH(CF_3)_2]_3$ 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 12 h. After completion, the reaction mixture was transferred onto silica gel column by a pipette and purified by silica gel chromatography (eluent: hexanes/EtOAc = 10:1 to 5:1) to afford the product **19** as a colorless oil in 81% total yield (137.9 mg).

Rf = 0.67 (hexanes/EtOAc = 2:1).

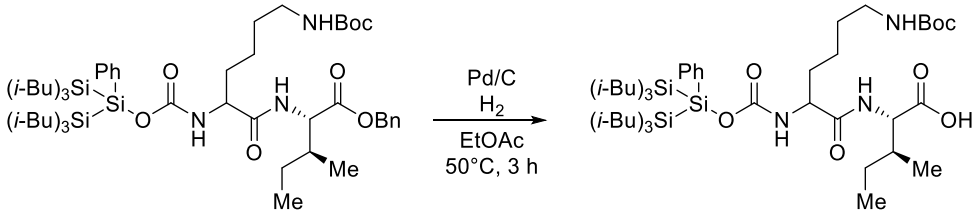
$[\alpha]_D^{24}$ = -0.87 (*c* 1.15, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.39 (m, 2H), 7.39 – 7.32 (m, 5H), 7.32 – 7.23 (m, 3H), 6.42 (d, J = 8.5 Hz, 1H), 5.36 (d, J = 8.4 Hz, 1H), 5.23 (d, J = 12.2 Hz, 1H), 5.12 (d, J = 12.2 Hz, 1H), 4.69 – 4.55 (m, 2H), 4.28 – 4.12 (m, 1H), 3.17 – 3.09 (m, 1H), 3.09 – 2.99 (m, 1H), 2.00 – 1.86 (m, 1H), 1.87 – 1.71 (m, 8H), 1.69 – 1.58 (m, 1H), 1.52 – 1.34 (m, 13H), 1.22 – 1.07 (m, 1H), 0.90 – 0.73 (m, 54H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 171.5, 156.0, 155.1, 137.8, 135.3, 133.1, 128.6, 128.45, 128.42, 128.1, 127.5, 79.0, 67.1, 56.6, 54.9, 40.0, 37.6, 32.5, 29.5, 28.4, 26.74, 26.68, 25.4, 25.0, 24.6, 24.5, 22.4, 15.6, 11.6.

IR (neat) 2951, 2866, 1676, 1498, 1461, 1428, 1380, 1364, 1326, 1248, 1215, 1163 cm^{-1}.

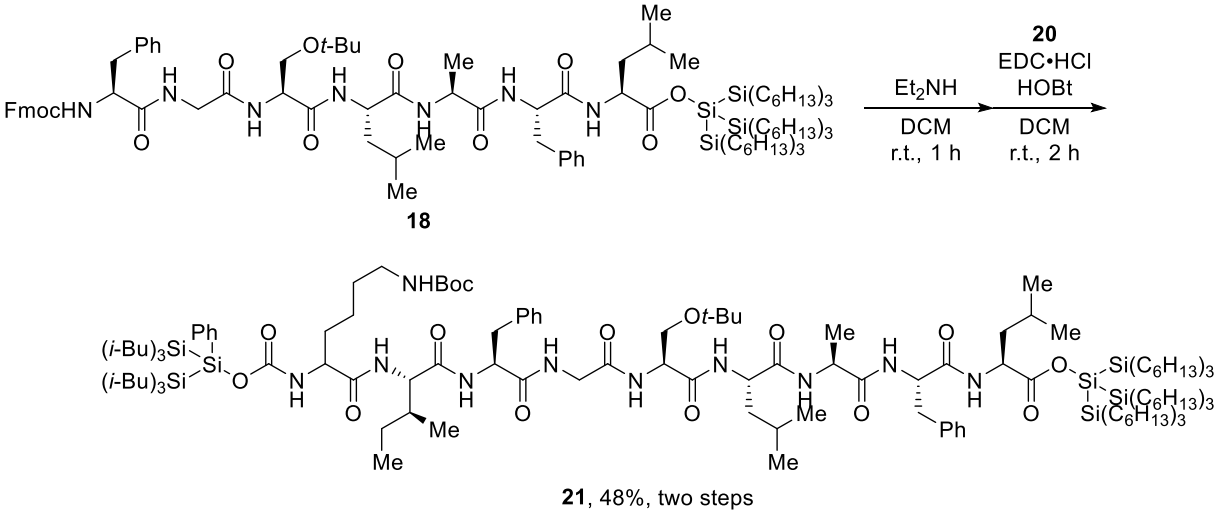
HRMS (ESI) Calcd for $C_{55}H_{97}N_3O_7Si_3Na$ $[M+Na]^+$: 1018.6532, Found: 1018.6535.



19 **20**, 96%

TAG6'-Lys(Boc)-Ile-OH (20) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **19** (94.9 mg, 0.095 mmol, 1.0 equiv) was added EtOAc (2 mL). The 10% Pd/C (20.3 mg, 0.019 mmol, 0.2 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 3 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.

Convergent connection.



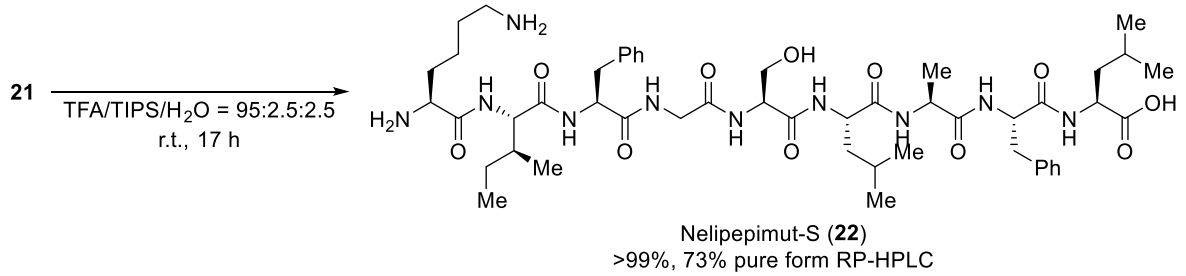
TAG6'-Lys(Boc)-Ile-Phe-Gly-Ser(*t*-Bu)-Leu-Ala-Phe-Leu-OTAG2 (21) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **18** (45.5 mg, 0.024 mmol) was added dichloromethane (0.24 mL) and diethylamine (0.25 mL). The reaction was stirred at room temperature for 1 h. After completion, the reaction mixture was concentrated to remove the solvent and the excess diethylamine. The residue was put into the next 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 dichloromethane (0.5 mL). The compound **20** (32.6 mg, 0.036 mmol, 1.5 equiv) was added followed by adding EDC·HCl (9.2 mg, 0.048 mmol, 2.0 equiv) and HOBt (3.2 mg, 0.024 mmol, 1.0 equiv). The resulting mixture was stirred under room temperature for 2 h. After completion, the reaction mixture was concentrated. The residue was purified by preparative thin layer chromatography (eluent: hexanes/EtOAc = 1:1) to afford the product **21** as a white solid in 48% total yield (29.4 mg).

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

HRMS (ESI) Calcd for $C_{144}H_{268}N_{10}O_{15}Si_7Na$ $[M+Na]^+$: 2596.8798, Found: 2596.8757.

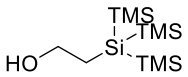
Cleavage.



H-Lys-Ile-Phe-Gly-Ser-Leu-Ala-Phe-Leu-OH (Nelipepimut-S, 22) At room temperature, to a flame-dried 6 mL vial charged with magnetic stirring bar (Sm-Co) and **21** (10.1 mg, 0.0039 mmol, 1.0 equiv) was added TFA/TIPS/H_2O (95:2.5:2.5 v/v/v, 0.078 mL). The reaction was stirred at room temperature for 17 h. After completion, the reaction mixture was concentrated. Diethyl ether (1 mL) was added and then removed which was repeated for three times. The remaining solid residue was then dissolved in trifluoroacetic acid (TFA) and filtered *via* PTFE syringe filter (0.22 μm) to remove the insoluble solids. The TFA mixture was concentrated and the solid residue was wash again with diethyl ether (3×1 mL) to afford the product Nelipepimut-S (**22**) as a white grey solid in >99% yield (4.4 mg). The purity of the product was 73% which was determined by RP-HPLC using a revised-phase column (XSelect CSH C18, 4.6 mm × 50 mm).

HRMS (ESI) Calcd for $C_{50}H_{78}N_{10}O_{11}Na$ $[M+Na]^+$: 1017.5749, Found: 1017.5711.

XIII. Synthesis of TAG8

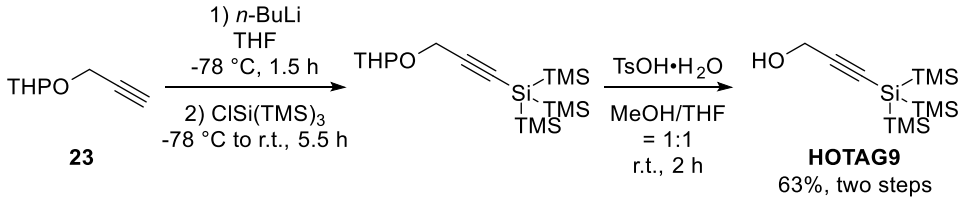


HO**TAG8**

2-(1,1,1,3,3,3-Hexamethyl-2-(trimethylsilyl)trisilan-2-yl)ethan-1-ol (HOTAG8) was prepared according to the procedure in the literatures.[10]

XIV. Synthesis of TAG9 and test

Synthesis of TAG9.



3-(1,1,1,3,3,3-Hexamethyl-2-(trimethylsilyl)trisilan-2-yl)prop-2-yn-1-ol (HOTAG9) At -78 ºC, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **23** (210.3 mg, 1.5 mmol, 1.5 equiv) was added tetrahydrofuran (8 mL). The *n*-butyllithium (1.55 M in hexanes, 0.97 mL, 1.5 mmol, 1.5 equiv) was added dropwise. The resulting mixture was stirred under -78 ºC for 1.5 h. Then, tris(trimethylsilyl)silyl chloride (283.1 mg, 1.0 mmol, 1.0 equiv) was added. The reaction mixture was warmed to room temperature slowly and stirred for 5.5 h. After completion, the reaction mixture was quenched with saturated NH_4Cl solution (10 mL), and the layers were separated. The aqueous layer was extracted with hexanes (3×10 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 methanol (5 mL) and tetrahydrofuran (5 mL). The *p*-toluenesulfonic acid monohydrate (19.0 mg, 0.1 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: hexanes/EtOAc = 30:1 to 20:1 to 15:1) to afford the product **HOTAG9** as a pale yellow oil in 63% total yield (190.5 mg).

Rf = 0.16 (hexanes/EtOAc = 20:1).

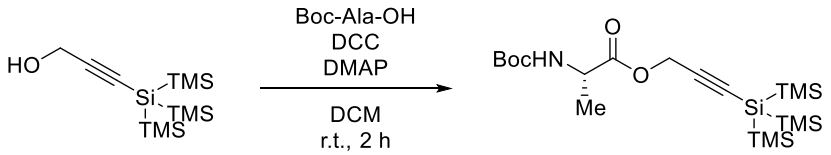
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.26 (s, 2H), 0.24 – 0.17 (m, 27H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 106.8, 84.2, 52.2, 0.3.

IR (neat) 2951, 2894, 1398, 1244, 1037 cm^{-1}.

HRMS (ESI) Calcd for $C_{12}H_{30}OSi_4Na$ $[M+Na]^+$: 325.1271, Found: 325.1248.

Esterification test.



HOTAG9 **Boc-Ala-OTAG9**, 96%

3-(1,1,1,3,3,3-Hexamethyl-2-(trimethylsilyl)trisilan-2-yl)prop-2-yn-1-yl (*tert*-butoxycarbonyl)-L-alaninate (Boc-Ala-OTAG9) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG9** (153.3 mg, 0.51 mmol, 1.0 equiv) was added dichloromethane (2.5 mL). The Boc-Ala-OH (191.6 mg, 1.01 mmol, 2.0 equiv) was added, followed by adding DMAP (74.2 mg, 0.61 mmol, 1.2 equiv) and DCC (209.0 mg, 1.01 mmol, 2.0 equiv). The reaction was stirred at room temperature for 2 h. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1 to 15:1) to afford the product **Boc-Ala-OTAG9** as a colorless oil in 96% yield (231.5 mg).

Rf = 0.58 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{23}$ = -27.10 (*c* 1.07, $CHCl_3$).

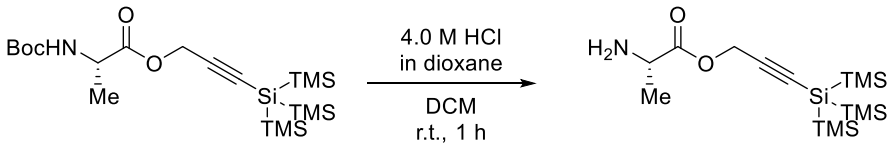
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.04 (d, J = 5.6 Hz, 1H), 4.82 (d, J = 15.6 Hz, 1H), 4.64 (d, J = 15.3 Hz, 1H), 4.43 – 4.23 (m, 1H), 1.43 (s, 9H), 1.38 (d, J = 7.2 Hz, 3H), 0.28 – 0.15 (m, 27H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 155.0, 101.3, 86.8, 79.8, 54.0, 49.1, 28.3, 18.6, 0.3.

IR (neat) 2951, 2894, 1748, 1709, 1502, 1451, 1367, 1340, 1308, 1245, 1159 cm^{-1}.

HRMS (ESI) Calcd for $C_{20}H_{43}NO_4Si_4Na$ $[M+Na]^+$: 496.2167, Found: 496.2167.

Tolerance test.



Boc-Ala-OTAG9 **H-Ala-OTAG9**, quant.

3-(1,1,1,3,3,3-Hexamethyl-2-(trimethylsilyl)trisilan-2-yl)prop-2-yn-1-yl L-alaninate (H-Ala-OTAG9) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Boc-Ala-OTAG9** (23.7 mg, 0.05 mmol, 1.0 equiv) was added dichloromethane (0.05 mL). The hydrochloric acid solution (4.0 M in dioxane, 0.125 mL, 0.5 mmol, 10.0 equiv) was added. The reaction was stirred at room temperature for 1 h. After completion, saturated Na_2CO_3 solution (5 mL) and dichloromethane (5 mL) was 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 to afford the product **H-Ala-OTAG9** as a pale yellow oil in quantitative yield (19.1 mg).

Rf = 0.06 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{23}$ = -47.50 (*c* 0.40, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.81 – 4.63 (m, 2H), 3.65 – 3.51 (m, 1H), 1.34 (d, J = 7.1 Hz, 3H), 0.23 – 0.16 (m, 27H).

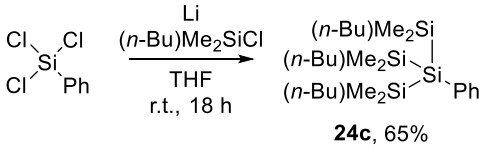
^{13}C NMR (100 MHz, $CDCl_3$) δ 175.8, 101.7, 86.4, 53.7, 50.0, 20.5, 0.2.

IR (neat) 2949, 2894, 1745, 1677, 1398, 1316, 1244, 1172, 1137, 1042 cm^{-1}.

HRMS (ESI) Calcd for $C_{15}H_{35}NO_2Si_4Na$ $[M+Na]^+$: 396.1643, Found: 396.1643.

XV. Development of TAG10 and test

Synthesis of 24c-24f.



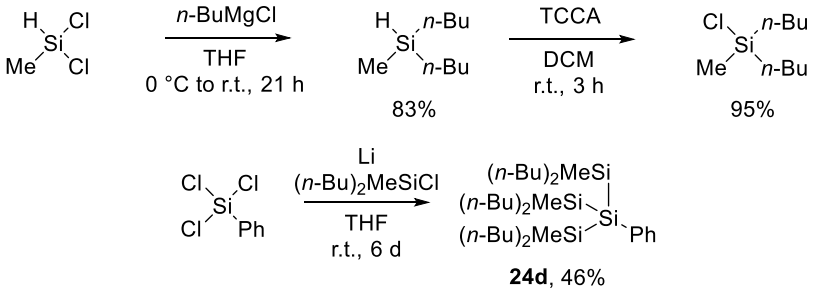
1,3-Dibutyl-2-(butyldimethylsilyl)-1,1,3,3-tetramethyl-2-phenyltrisilane (24c) At room temperature, under N_2, to a flame-dried flask charged with Li (granular, 222.1 mg, 32.0 mmol, 8.0 equiv) was added tetrahydrofuran (20 mL). The trichloro(phenyl)silane (0.641 mL, 4.0 mmol, 1.0 equiv) and butylchlorodimethylsilane (2.15 mL, 12.4 mmol, 3.1 equiv) were added together. The reaction was stirred at room temperature for 18 hours. Then, the reaction mixture was poured into a separation funnel charged with 50 mL hexanes and 50 mL water. 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 **24c** as a colorless oil in 65% yield (1.17 g). [***Note****: The purification by chromatography should be repeated if the product is not pure enough.*] Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data and structure of installed compounds support it.

Rf = 0.71 (100% hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.39 (m, 2H), 7.25 – 7.19 (m, 3H), 1.37 – 1.19 (m, 12H), 0.89 – 0.80 (m, 9H), 0.74 – 0.60 (m, 6H), 0.23 – 0.11 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.7, 135.9, 127.5, 127.2, 26.6, 26.5, 17.0, 13.7, -0.9.

IR (neat) 2955, 2921, 2871, 2857, 1463, 1427, 1243, 1078, 1024 cm^{-1}.



1,1,3,3-Tetrabutyl-2-(dibutyl(methyl)silyl)-1,3-dimethyl-2-phenyltrisilane (24d) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and

dichloromethylsilane (1.23 mL, 12 mmol, 1.0 equiv) was added tetrahydrofuran (60 mL). The *n*-BuMgCl (2.0 M in THF, 15 mL, 30 mmol, 2.5 equiv) was added dropwise. The resulting mixture was warmed to room temperature slowly and stirred for 21 h. After completion, the reaction mixture was quenched with saturated NH_4Cl solution (60 mL), and the layers were separated. The aqueous layer was extracted with hexanes (3×60 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and concentrated to afford the product dibutyl(methyl)silane as a colorless oil in 83% yield (1.58 g).

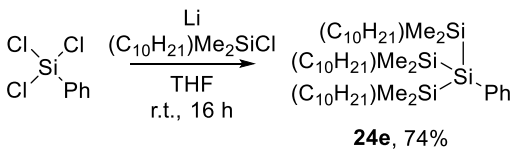
At room temperature, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and dibutyl(methyl)silane (1.58 g, 9.95 mmol, 1.0 equiv) was added dichloromethane (50 mL). The trichloroisocyanuric acid (786.3 mg, 3.38 mmol, 0.34 equiv) was added. The resulting mixture was stirred for 3 h. After completion, the reaction mixture was concentrated, and anhydrous hexanes (20 mL) were added. The mixture was filtered via Celite pad and washed by hexanes. The organic solution was concentrated to afford the product dibutylchloro(methyl)silane in 95% yield (1.83 g).

At room temperature, under N_2, to a flame-dried flask charged with Li (granular, 170.0 mg, 24.5 mmol, 8.0 equiv) was added tetrahydrofuran (15 mL). The trichloro(phenyl)silane (0.491 mL, 3.1 mmol, 1.0 equiv) and dibutylchloro(methyl)silane (1.83 g, 9.5 mmol, 3.1 equiv) were added together. The reaction was stirred at room temperature for 6 days. Then, the reaction mixture was poured into a separation funnel charged with 30 mL hexanes and 30 mL water. The layers were separated. The aqueous layer was extracted with hexanes (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: 100% hexanes) to afford the product **24d** as a colorless oil in 46% yield (818.7 mg). [***Note****: The purification by chromatography should be repeated if the product is not pure enough.*] Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data support it.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.39 (m, 2H), 7.24 – 7.16 (m, 3H), 1.36 – 1.16 (m, 24H), 0.93 – 0.78 (m, 18H), 0.78 – 0.58 (m, 12H), 0.22 – 0.16 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.9, 136.5, 127.3, 127.0, 27.0, 26.8, 16.0, 13.7, -2.9.

IR (neat) 2955, 2920, 2871, 2856, 1463, 1426, 1412, 1376, 1294, 1245, 1177 cm^{-1}.



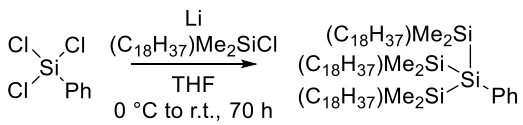
24e, 74%

1,3-Didecyl-2-(decyldimethylsilyl)-1,1,3,3-tetramethyl-2-phenyltrisilane (24e) At room temperature, under N_2, to a flame-dried flask charged with Li (granular, 222.1 mg, 32.0 mmol, 8.0 equiv) was added tetrahydrofuran (20 mL). The trichloro(phenyl)silane (0.641 mL, 4.0 mmol, 1.0 equiv) and decyldimethylchlorosilane (3.35 mL, 12.4 mmol, 3.1 equiv) were added together. The reaction was stirred at room temperature for 16 hours. Then, the reaction mixture was poured into a separation funnel charged with 50 mL hexanes and 50 mL water. 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 **24e** as a colorless oil in 74% yield (2.07 g). [***Note****: The purification by chromatography should be repeated if the product is not pure enough*] Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data and structure of installed compounds support it.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.38 (m, 2H), 7.25 – 7.18 (m, 3H), 1.34 – 1.20 (m, 48H), 0.92 – 0.84 (m, 9H), 0.74 – 0.58 (m, 6H), 0.25 – 0.11 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.7, 135.9, 127.5, 127.2, 33.8, 31.9, 29.7, 29.6, 29.4(2C), 24.3, 22.7, 17.4, 14.1, -0.8.

IR (neat) 2955, 2920, 2852, 1465, 1427, 1244, 1087, 1027 cm^{-1}.



24f, 59%

2-(Dimethyl(octadecyl)silyl)-1,1,3,3-tetramethyl-1,3-dioctadecyl-2-phenyltrisilane (24f) At 0 ºC, under N_2, to a flame-dried flask charged with Li (granular, 222.1 mg, 32.0 mmol, 8.0 equiv) was added tetrahydrofuran (20 mL). The trichloro(phenyl)silane (0.641 mL, 4.0 mmol, 1.0 equiv) and chloro(dimethyl)octadecylsilane (4.30 g, 12.4 mmol, 3.1 equiv) were added together. The reaction was stirred at 0 ºC for 4 h and then room temperature for 66 hours. Then, the reaction mixture was poured into a separation funnel charged with 50 mL hexanes and 50 mL water. 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 **24f** as a white wax in 59% yield (2.44 g). [***Note****: The purification by chromatography should be repeated if the product is not pure enough.*] Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data support it.

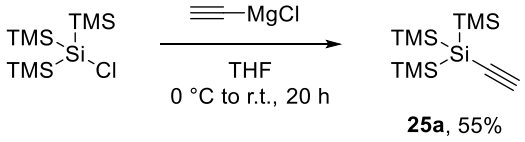
M.p. 25-27 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.39 (m, 2H), 7.25 – 7.17 (m, 3H), 1.33 – 1.19 (m, 96H), 0.92 – 0.84 (m, 9H), 0.75 – 0.56 (m, 6H), 0.26 – 0.11 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.7, 135.9, 127.5, 127.2, 33.8, 31.9, 29.74(8C), 29.69, 29.6, 29.4(2C), 24.3, 22.7, 17.4, 14.1, -0.8.

IR (neat) 2954, 2914, 2849, 1469, 1427, 1245, 1166 cm^{-1}.

Synthesis of 25a, 25c, 25e.



25a, 55%

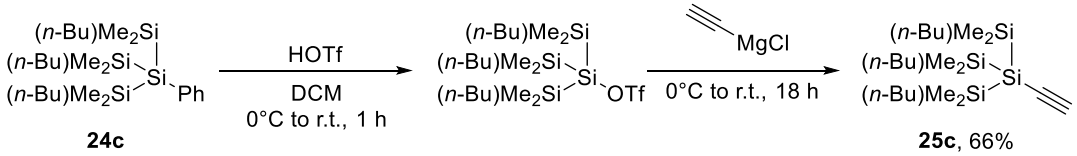
2-Ethynyl-1,1,1,3,3,3-hexamethyl-2-(trimethylsilyl)trisilane (25a) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and tris(trimethylsilyl)silyl chloride (141.6 mg, 0.5 mmol, 1.0 equiv) was added tetrahydrofuran (3 mL). The ethynylmagnesium chloride (0.5 M in THF, 1.5 mL, 0.75 mmol, 1.5 equiv) was added dropwise. The resulting mixture was stirred under room temperature for 20 h. After completion, the reaction mixture was quenched with saturated NH_4Cl solution (10 mL), and the layers were separated. The aqueous layer was extracted with hexanes (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: 100% hexanes) to afford the product **25a** as a colorless oil in 55% yield (75.4 mg). Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data support it.

Rf = 0.75 (100% hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 2.30 (s, 1H), 0.22 (s, 27H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 95.8, 84.0, 0.2.

IR (neat) 3299, 2950, 2894, 1398, 1244 cm^{-1}.



24c **25c**, 66%

1,3-Dibutyl-2-(butyldimethylsilyl)-2-ethynyl-1,1,3,3-tetramethyltrisilane (25c) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **24c** (225.5

mg, 0.5 mmol, 1.0 equiv) was added dichloromethane (2 mL). The triflic acid (57.5 μL, 0.65 mmol, 1.3 equiv) was added. The reaction was stirred at room temperature for 1 h. Then, cool to 0 °C, and the ethynylmagnesium chloride (0.5 M in THF, 2.0 mL, 1.0 mmol, 2.0 equiv) was added dropwise. The resulting mixture was stirred under room temperature for 18 h. After completion, the reaction mixture was quenched with saturated NH_4Cl solution (10 mL), and the layers were separated. The aqueous layer was extracted with hexanes (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: 100% hexanes) to afford the product **25c** as a colorless oil in 66% yield (132.4 mg).

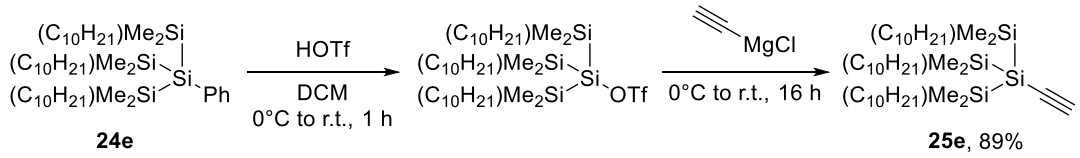
Rf = 0.82 (100% hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 2.31 (s, 1H), 1.41 – 1.27 (m, 12H), 0.93 – 0.86 (m, 9H), 0.75 – 0.66 (m, 6H), 0.22 – 0.16 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 96.2, 84.7, 26.6(2C), 16.7, 13.8, -1.6.

IR (neat) 3297, 2956, 2922, 2872, 1464, 1410, 1377, 1341, 1244, 1188, 1078, 1022 cm^{-1}.

HRMS (ESI) Calcd for $C_{20}H_{46}Si_4Na$ $[M+Na]^+$: 421.2574, Found: 421.2556.



24e **25e**, 89%

1,3-Didecyl-2-(decyldimethylsilyl)-2-ethynyl-1,1,3,3-tetramethyltrisilane (25e) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **24e** (351.7 mg, 0.5 mmol, 1.0 equiv) was added dichloromethane (2 mL). The triflic acid (57.5 μL, 0.65 mmol, 1.3 equiv) was added. The reaction was stirred at room temperature for 1 h. Then, cool to 0 °C, and the ethynylmagnesium chloride (0.5 M in THF, 2.0 mL, 1.0 mmol, 2.0 equiv) was added dropwise. The resulting mixture was stirred under room temperature for 16 h. After completion, the reaction mixture was concentrated. The residue was purified by silica gel chromatography (eluent: 100% hexanes) to afford the product **25e** as a colorless oil in 89% yield (289.1 mg). Although molecular ion peak was not located in the mass spectrum in ESI equipment, NMR data support it.

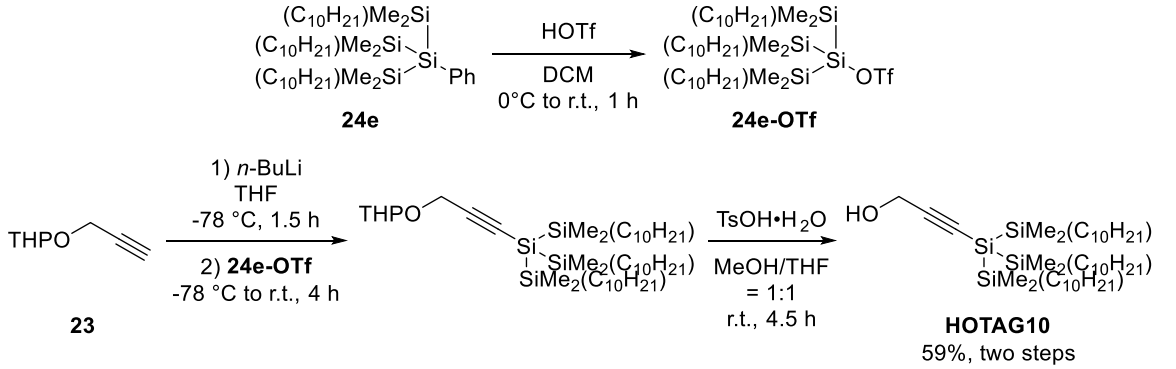
Rf = 0.88 (100% hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 2.30 (s, 1H), 1.40 – 1.20 (m, 48H), 0.92 – 0.84 (m, 9H), 0.74 – 0.65 (m, 6H), 0.25 – 0.12 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 96.2, 84.8, 33.8, 31.9, 29.70, 29.65, 29.42, 29.38, 24.5, 22.7, 17.1, 14.1, -1.6.

IR (neat) 3297, 2955, 2920, 2852, 1465, 1244 cm^{-1}.

Synthesis of TAG10.



3-(1,3-Didecyl-2-(decyldimethylsilyl)-1,1,3,3-tetramethyltrisilan-2-yl)prop-2-yn-1-ol (HOTAG10) At 0 °C, under N_2, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **24e** (703.5 mg, 1.0 mmol, 1.0 equiv) was added dichloromethane (4 mL). The triflic acid (115.0 μL, 1.3 mmol, 1.3 equiv) was added. The reaction was stirred at room temperature for 1 h to make **24e-OTf** solution.

At -78 °C, under N_2, to another flame-dried flask charged with magnetic stirring bar (Sm-Co) and **23** (252.3 mg, 1.8 mmol, 1.8 equiv) was added tetrahydrofuran (10 mL). The *n*-butyllithium (1.55 M in hexanes, 1.16 mL, 1.8 mmol, 1.8 equiv) was added dropwise. The resulting mixture was stirred under -78 °C for 1.5 h. Then, transfer the above **24e-OTf** solution into the reaction mixture slowly. The reaction mixture was warmed to room temperature slowly and stirred for 4 h. After completion, the reaction mixture was quenched with saturated NH_4Cl solution (10 mL), and the layers were separated. The aqueous layer was extracted with hexanes (3×10 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 methanol (5 mL) and tetrahydrofuran (5 mL). The *p*-toluenesulfonic acid monohydrate (19.0 mg, 0.1 mmol, 0.1 equiv) was added. The reaction 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 = 50:1) to afford the product **HOTAG10** as a colorless oil in 59% total yield (402.9 mg).

Rf = 0.32 (hexanes/EtOAc = 20:1).

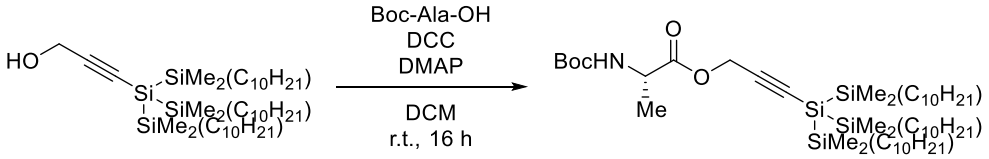
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.24 (d, J = 6.1 Hz, 2H), 1.37 – 1.24 (m, 48H), 0.92 – 0.84 (m, 9H), 0.73 – 0.64 (m, 6H), 0.21 – 0.12 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 107.0, 85.2, 52.3, 33.8, 31.9, 29.69, 29.65, 29.5, 29.4, 24.5, 22.7, 17.2, 14.1, -1.5.

IR (neat) 2955, 2920, 2852, 1465, 1410, 1378, 1243, 1037 cm^{-1}.

HRMS (ESI) Calcd for $C_{39}H_{84}OSi_4Na$ $[M+Na]^+$: 703.5497, Found: 703.5546.

Esterification test.



HOTAG10 **Boc-Ala-OTAG10**, 74%

3-(1,3-Didecyl-2-(decyldimethylsilyl)-1,1,3,3-tetramethyltrisilan-2-yl)prop-2-yn-1-yl (*tert*-butoxycarbonyl)-L-alaninate (Boc-Ala-OTAG10) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG10** (136.3 mg, 0.2 mmol, 1.0 equiv) was added dichloromethane (1.0 mL). The Boc-Ala-OH (75.7 mg, 0.4 mmol, 2.0 equiv) was added, followed by adding DMAP (29.3 mg, 0.24 mmol, 1.2 equiv) and DCC (82.5 mg, 0.4 mmol, 2.0 equiv). The reaction was stirred at room temperature for 16 h. 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-OTAG10** as a colorless oil in 74% yield (126.5 mg).

Rf = 0.69 (hexanes/EtOAc = 5:1).

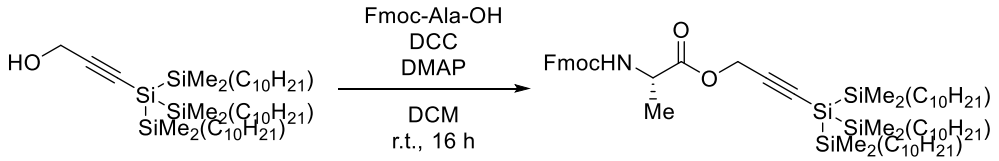
$[\alpha]_D^{23}$ = -34.94 (c 0.83, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.17 – 4.94 (m, 1H), 4.82 (d, J = 15.6 Hz, 1H), 4.62 (d, J = 15.5 Hz, 1H), 4.42 – 4.20 (m, 1H), 1.44 (s, 9H), 1.38 (d, J = 7.2 Hz, 3H), 1.36 – 1.19 (m, 48H), 0.96 – 0.78 (m, 9H), 0.75 – 0.59 (m, 6H), 0.26 – 0.08 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 155.0, 101.5, 87.6, 79.8, 54.1, 49.1, 33.8, 31.9, 29.69, 29.65, 29.42, 29.37, 28.3, 24.5, 22.7, 18.7, 17.1, 14.1, -1.5.

IR (neat) 2956, 2922, 2853, 1750, 1721, 1498, 1455, 1366, 1339, 1308, 1244, 1160 cm^{-1}.

HRMS (ESI) Calcd for $C_{47}H_{97}NO_4Si_4Na$ $[M+Na]^+$: 874.6392, Found: 874.6402.



3-(1,3-Didecyl-2-(decyldimethylsilyl)-1,1,3,3-tetramethyltrisilan-2-yl)prop-2-yn-1-yl (((9*H*-fluoren-9-yl)methoxy)carbonyl)-L-alaninate (Fmoc-Ala-OTAG10) At room temperature, to a 15 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **HOTAG10** (136.3 mg, 0.2 mmol, 1.0 equiv) was added dichloromethane (1.0 mL). The Fmoc-Ala-OH (124.5 mg, 0.4 mmol, 2.0 equiv) was added, followed by adding DMAP (29.3 mg, 0.24 mmol, 1.2 equiv) and DCC (82.5 mg, 0.4 mmol, 2.0 equiv). The reaction was stirred at room temperature for 16 h. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 20:1 to 15:1) to afford the product **Fmoc-Ala-OTAG10** as a pale yellow oil in 79% yield (153.4 mg).

Rf = 0.60 (hexanes/EtOAc = 5:1).

$[\alpha]_D^{23}$ = -27.35 (*c* 1.28, $CHCl_3$).

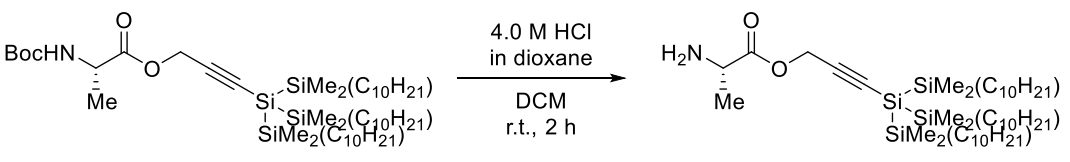
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.65 – 7.57 (m, 2H), 7.45 – 7.37 (m, 2H), 7.37 – 7.27 (m, 2H), 5.37 (d, *J* = 7.8 Hz, 1H), 4.85 (d, *J* = 15.6 Hz, 1H), 4.66 (d, *J* = 15.5 Hz, 1H), 4.49 – 4.36 (m, 3H), 4.23 (t, *J* = 7.0 Hz, 1H), 1.45 (d, *J* = 7.2 Hz, 3H), 1.37 – 1.18 (m, 48H), 0.93 – 0.84 (m, 9H), 0.73 – 0.64 (m, 6H), 0.26 – 0.11 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 155.5, 143.9, 143.8, 141.3, 127.7, 127.0, 125.1, 120.0, 101.4, 88.0, 67.0, 54.3, 49.6, 47.2, 33.8, 31.9, 29.69, 29.66, 29.42, 29.37, 24.5, 22.7, 18.7, 17.1, 14.1, -1.5.

IR (neat) 2921, 2852, 1726, 1506, 1450, 1379, 1335, 1306, 1243, 1195, 1167 cm^{-1}.

HRMS (ESI) Calcd for $C_{57}H_{99}NO_4Si_4Na$ $[M+Na]^+$: 996.6549, Found: 996.6551.

Tolerance test.



3-(1,3-Didecyl-2-(decyldimethylsilyl)-1,1,3,3-tetramethyltrisilan-2-yl)prop-2-yn-1-yl L-alaninate (H-Ala-OTAG10) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Boc-Ala-OTAG10** (77.8 mg, 0.091 mmol, 1.0 equiv) was added dichloromethane (0.090 mL). The hydrochloric acid solution (4.0 M in dioxane, 0.228 mL, 0.91 mmol, 10.0 equiv) was added. The reaction was stirred at room temperature for 2 h. After completion, saturated Na_2CO_3 solution (5 mL) and dichloromethane (5 mL) was 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 to afford the product **H-Ala-OTAG10** as a colorless oil in quantitative yield (69.8 mg).

Rf = 0.23 (hexanes/EtOAc = 5:1).

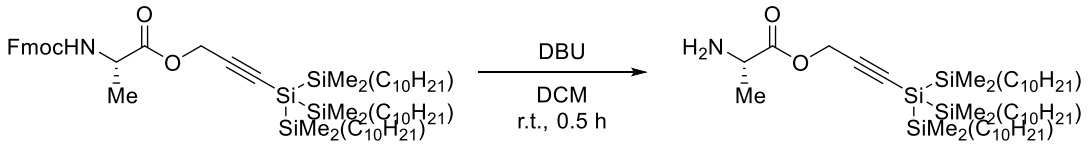
$[\alpha]_D^{24}$ = -18.75 (*c* 1.28, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.80 (d, *J* = 15.6 Hz, 1H), 4.65 (d, *J* = 15.6 Hz, 1H), 3.88 – 3.71 (m, 1H), 1.47 (d, *J* = 6.9 Hz, 3H), 1.37 – 1.23 (m, 48H), 0.94 – 0.83 (m, 9H), 0.71 – 0.62 (m, 6H), 0.16 (s, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 101.4, 87.9, 54.3, 49.6, 33.7, 31.9, 29.7, 29.6, 29.41, 29.36, 24.5, 22.7, 19.0, 17.1, 14.1, -1.6.

IR (neat) 2922, 2853, 1744, 1459, 1244, 1171, 1037 cm^{-1}.

HRMS (ESI) Calcd for $C_{42}H_{89}NO_2Si_4Na$ $[M+Na]^+$: 774.5868, Found: 774.5915.

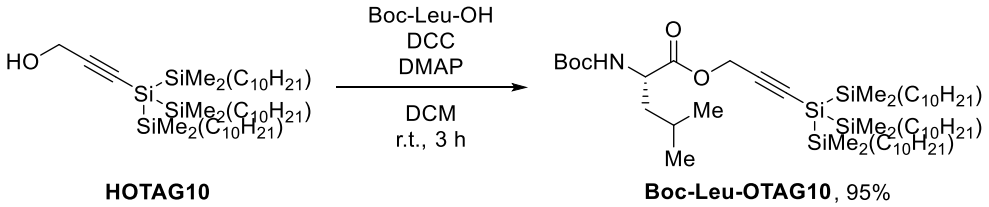


Boc-Ala-OTAG10 **H-Ala-OTAG10**, 87%

3-(1,3-Didecyl-2-(decyldimethylsilyl)-1,1,3,3-tetramethyltrisilan-2-yl)prop-2-yn-1-yl L-alaninate (H-Ala-OTAG10) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **Fmoc-Ala-OTAG10** (107.0 mg, 0.11 mmol, 1.0 equiv) was added dichloromethane (1.1 mL). The DBU (0.0328 mL, 0.22 mmol, 2.0 equiv) was added. The reaction was stirred at room temperature for 0.5 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-OTAG10** as a colorless oil in 87% yield (71.6 mg).

XVI. Synthesis of protected Nelipepimut-S with TAG10 in Boc-chemistry

First esterification step.



HOTAG10 **Boc-Leu-OTAG10**, 95%

3-(1,3-Didecyl-2-(decyldimethylsilyl)-1,1,3,3-tetramethyltrisilan-2-yl)prop-2-yn-1-yl (*tert*-butoxycarbonyl)-L-leucinate (Boc-Leu-OTAG10) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and **HOTAG10** (1.11 g, 1.63 mmol, 1.0 equiv) was added dichloromethane (8.0 mL). The Boc-Leu-OH (753.7 mg, 3.26 mmol, 2.0 equiv) was added, followed by adding DMAP (238.9 mg, 1.96 mmol, 1.2 equiv) and DCC (672.4 mg, 3.26 mmol, 2.0 equiv). The reaction was stirred at room temperature for 3 h. Then, filtered, washed with dichloromethane (5 mL) and concentrated. The residue was purified by silica gel chromatography (eluent: hexanes/EtOAc = 33:1) to afford the product **Boc-Leu-OTAG10** as a colorless oil in 95% yield (1.38 g).

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

$[\alpha]_D^{24}$ = -33.01 (*c* 1.03, $CHCl_3$).

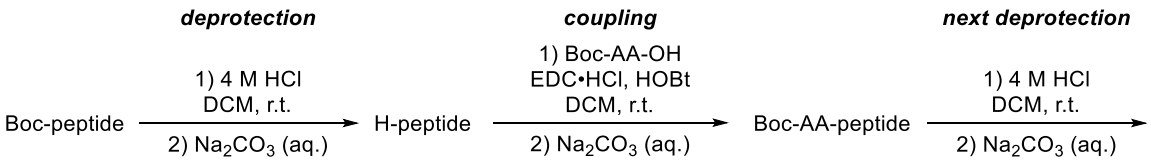
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.93 – 4.76 (m, 2H), 4.59 (d, J = 15.5 Hz, 1H), 4.44 – 4.27 (m, 1H), 1.81 – 1.66 (m, 1H), 1.64 – 1.58 (m, 1H), 1.54 – 1.40 (m, 10H), 1.36 – 1.17 (m, 48H), 0.98 – 0.92 (m, 6H), 0.92 – 0.84 (m, 9H), 0.71 – 0.63 (m, 6H), 0.22 – 0.12 (m, 18H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 155.3, 101.6, 87.4, 79.8, 53.9, 52.0, 42.0, 33.8, 31.9, 29.69, 29.66, 29.43, 29.37, 28.3, 24.8, 24.5, 22.8, 22.7, 21.9, 17.1, 14.1, -1.5.

IR (neat) 2956, 2921, 2853, 1750, 1721, 1500, 1466, 1367, 1333, 1244, 1158, 1121 cm^{-1}.

HRMS (ESI) Calcd for $C_{50}H_{103}NO_4Si_4Na$ $[M+Na]^+$: 916.6862, Found: 916.6907.

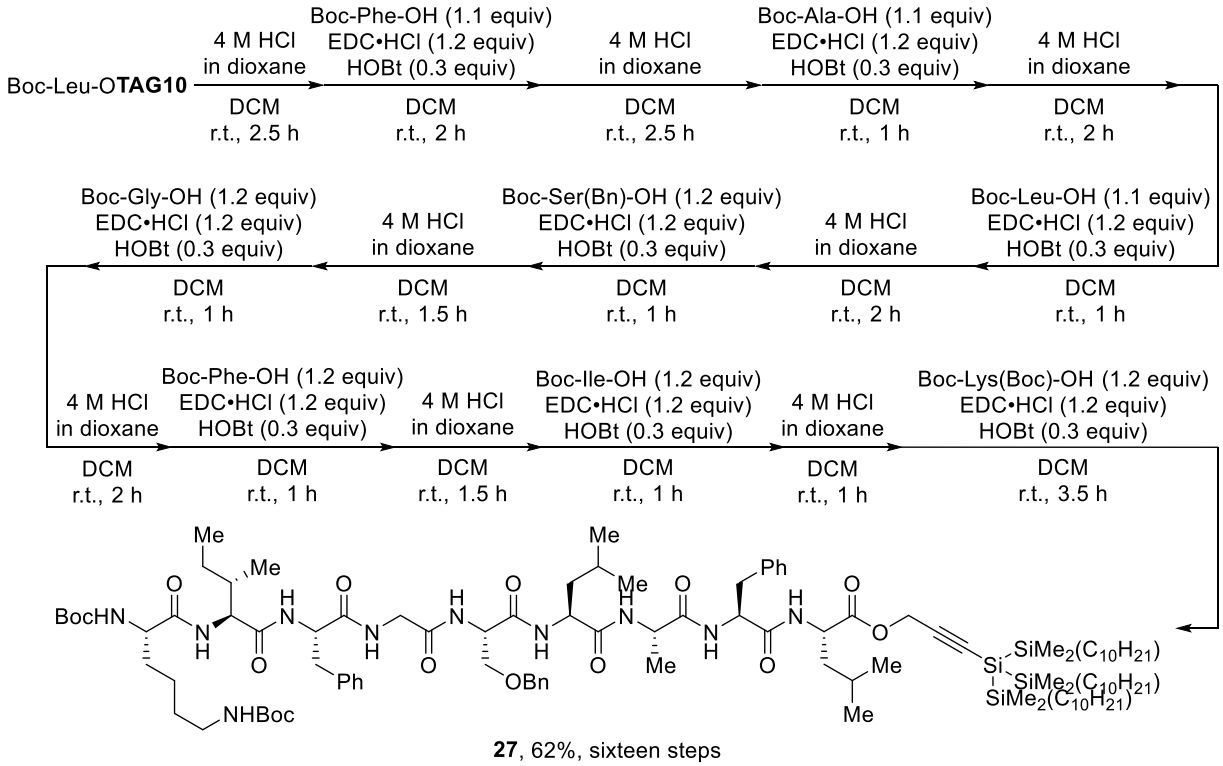
General procedures for Boc-deprotection and coupling reactions.



At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and Boc-protected peptide (1.0 equiv) was added dichloromethane (1.0 M). The hydrochloric acid solution (4.0 M in dioxane, 10.0 to 15.0 equiv) was added. The reaction was stirred at room temperature for 1 to 2 h (monitored by TLC). After completion, saturated Na_2CO_3 solution and dichloromethane was added, and the layers were separated. 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 Boc-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 dichloromethane (0.05 to 0.2 M). The Boc-protected amino acid (Boc-AA-OH, 1.1 to 1.2 equiv) was added followed by adding EDC·HCl (1.2 equiv) and HOBt (0.3 equiv). The resulting mixture was stirred under room temperature. After completion, the mixture was diluted with dichloromethane, followed by adding saturated Na_2CO_3 solution. The layers were separated. 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 Boc-deprotection step without further purification.

Elongation



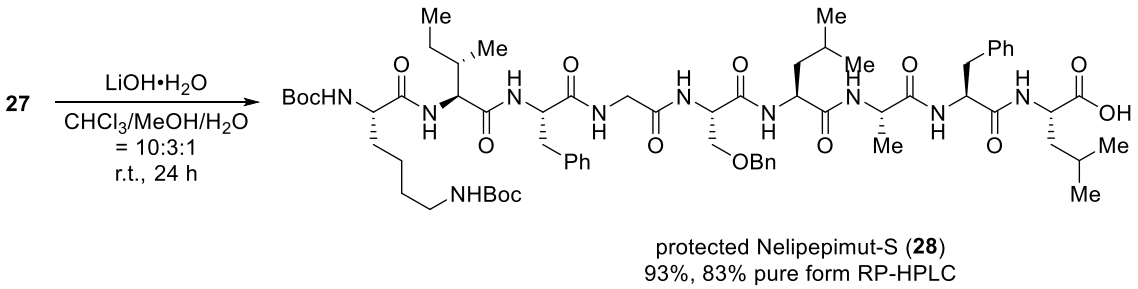
27, 62%, sixteen steps

Boc-Lys(Boc)-Ile-Phe-Gly-Ser(Bn)-Leu-Ala-Phe-Leu-OTAG10 (27) At room temperature, to a flame-dried flask charged with magnetic stirring bar (Sm-Co) and Boc-protected peptide **Boc-Leu-OTAG10** (134.2 mg, 0.15 mmol, 1.0 equiv) was added dichloromethane (0.15 mL). Then, the mixture was followed the **General procedures for Boc-deprotection and coupling reactions** and repeated for seven times (eight times in total) by changing the coupling amino acid according to the sequence of the target peptide every time. In the final coupling step, after the completion of the coupling reaction and finishing the work-up procedure, the residue was purified by silica gel chromatography (eluent: DCM/MeOH = 10:1) to afford the product **27** as a white solid in 62% total yield (181.2 mg).

Rf = 0.42 (DCM/MeOH = 10:1).

HRMS (ESI) Calcd for $C_{106}H_{182}N_{10}O_{15}Si_4Na$ $[M+Na]^+$: 1970.2761, Found: 1970.2796.

Cleavage.



Boc-Lys(Boc)-Ile-Phe-Gly-Ser(Bn)-Leu-Ala-Phe-Leu-OH (28) At room temperature, to a 6 mL flame-dried vial charged with magnetic stirring bar (Sm-Co) and **27** (39.0 mg, 0.02 mmol, 1.0 equiv) was added chloroform (0.2 mL), methanol (60 μL) and water (20 μL). The lithium hydroxide monohydrate (2.1 mg, 0.05 mmol, 2.5 equiv) was added. The reaction was stirred under room temperature for 24 h. After completion, the reaction mixture was concentrated. Dichloromethane (1.0 mL) was added, followed by adding hydrochloric acid solution (2 N in water, 60 μL, 0.12 mmol, 6.0 equiv). Additional 0.5 mL water was added and the layers was separated. The organic layer was concentrated and washed by hexanes (3×1 mL). The remaining white solid was the product **28** in 93% yield (23.9 mg). The purity of the product was 83% which was determined by RP-HPLC using a revised-phase column (ODS-HL, 4.6 mm × 25 cm).

HRMS (ESI) Calcd for $C_{67}H_{100}N_{10}O_{15}Na$ $[M+Na]^+$: 1307.7267, Found: 1307.7280.

XVII. References

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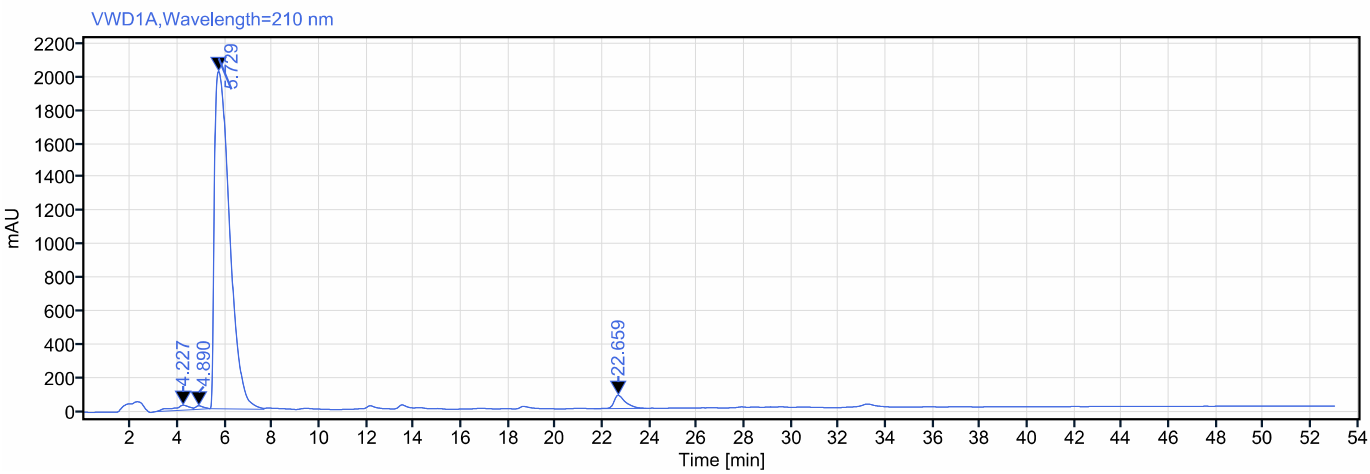
Single Injection Report

Agilent

Data file:	wuan-1352-1.dx		
Sequence Name:	SingleSample	**Project Name:**	Yamamoto-Lab
Sample name:	wuan-1352	**Operator:**	SYSTEM
Instrument:	HPLC2	**Injection date:**	2022-05-31 13:33:36+09:00
Inj. volume:	0.000	**Location:**	
Acq. method:	C18_0.1%TFA-H2O85_0.1%TFA-MeCN15_0.1%TFA-H2O57.5_0.1%TFA-MeCN42.5_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

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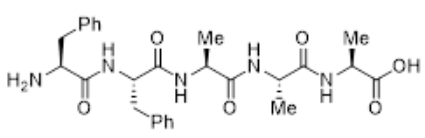
5.729	BV	2.30	89767.48	2015.19	95.17	
22.659	BB	2.21	2692.05	79.39	2.85	
		Sum	**94328.21**			

5.729 min = H-Phe-Phe-Ala-Ala-Ala-OH (**14**)
Conditions: 0.1% TFA in water/0.1% TFA in acetonitrile = 85:15 to 57.5:42.5, v = 0.5 mL/min, λ = 210 nm
Column: XSelect CSH C18 column from Waters



Single Injection Report

Agilent

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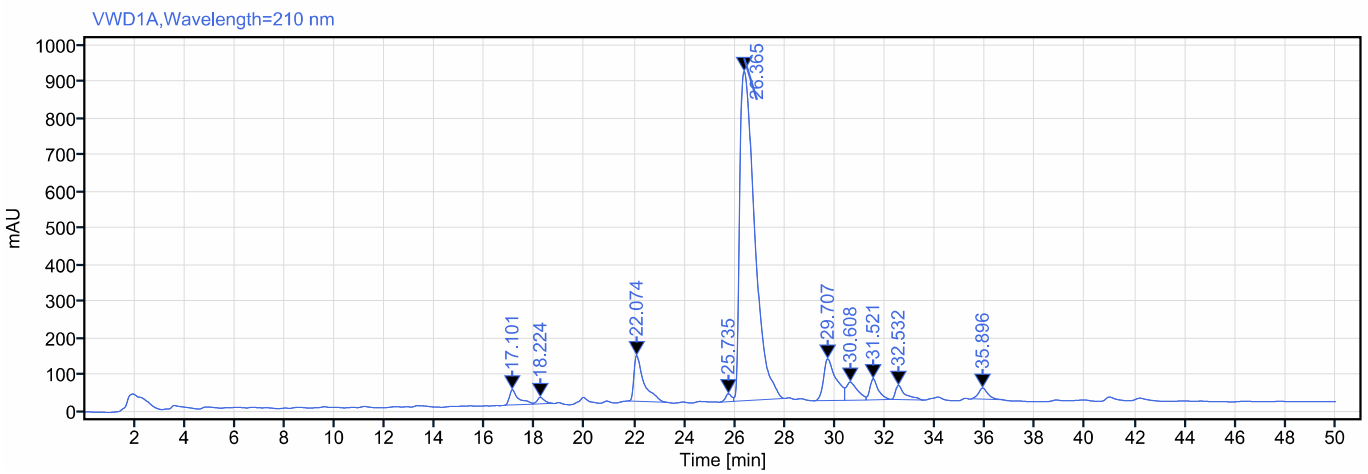
Sample name:	wuan-1439-1	**Operator:**	SYSTEM
Instrument:	HPLC2	**Injection date:**	2022-06-23 11:37:58+09:00
Inj. volume:	0.000	**Location:**	
Acq. method:	C18_0.1%TFA-H2O85_0.1%TFA-MeCN15_0.1%TFA-H2O57.5_0.1%TFA-MeCN42.5_0.5ml_40deg_50min_210nm-2.amx	**Type:**	Sample
Processing method:	New method 1.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: VWD1A,Wavelength=210 nm

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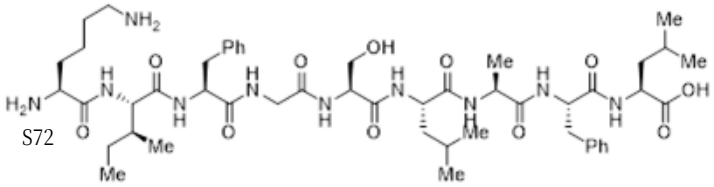
22.074	MM m	1.52	3432.19	124.56	6.70	
25.735	BM m	0.58	374.74	21.67	0.73	
26.365	MM m	2.03	37333.22	898.71	72.85	
29.707	BV	1.22	3942.60	113.67	7.69	
30.608	VV	0.86	1560.56	49.64	3.05	
31.521	VB	0.99	1327.11	57.53	2.59	
32.532	BB	1.31	1038.43	38.86	2.03	
35.896	MM m	1.24	804.32	30.36	1.57	
		Sum	**51244.82**			

26.365 min = H-Lys-Ile-Phe-Gly-Ser-Leu-Ala-Phe-Leu-OH (Nelipepimut-S, **22**)
Conditions: 0.1% TFA in water/0.1% TFA in acetonitrile = 85:15 to 57.5:42.5, v = 0.5 mL/min, λ = 210 nm
Column: XSelect CSH C18 column from Waters



Single Injection Report

Agilent

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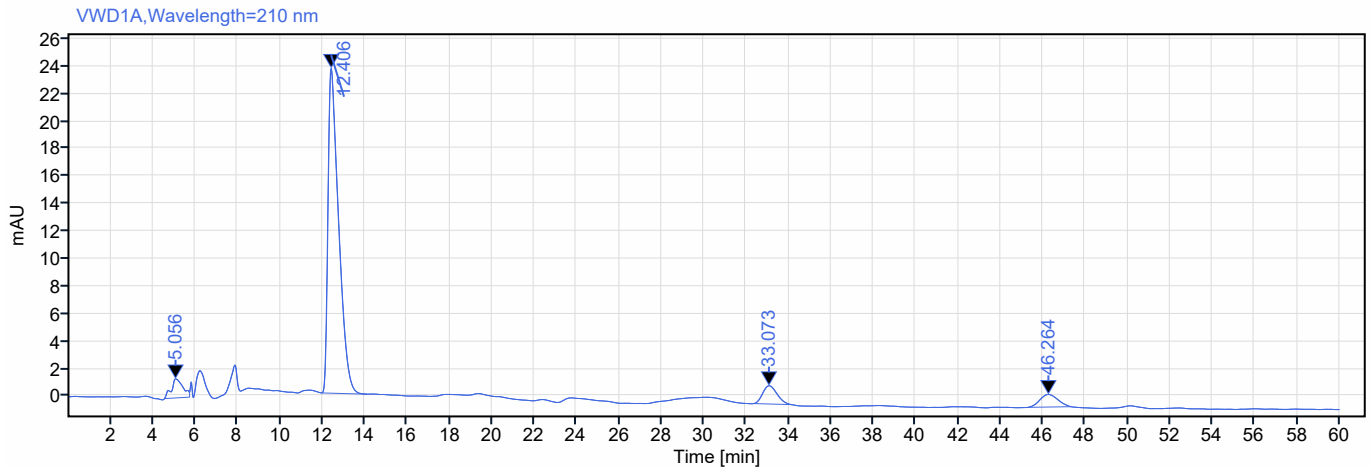
Inj. volume:	0.000	**Location:**	
Acq. method:	ODS-HL_H2O85_MeCN15_0.5ml_rt_60min_210nm.amx	**Type:**	Sample
Processing method:	New method 1.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: VWD1A,Wavelength=210 nm

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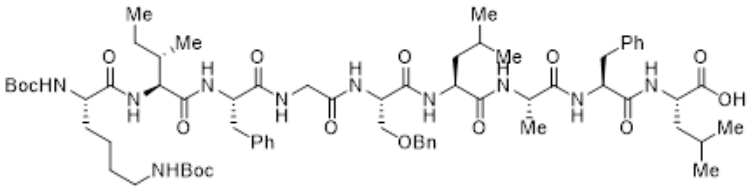
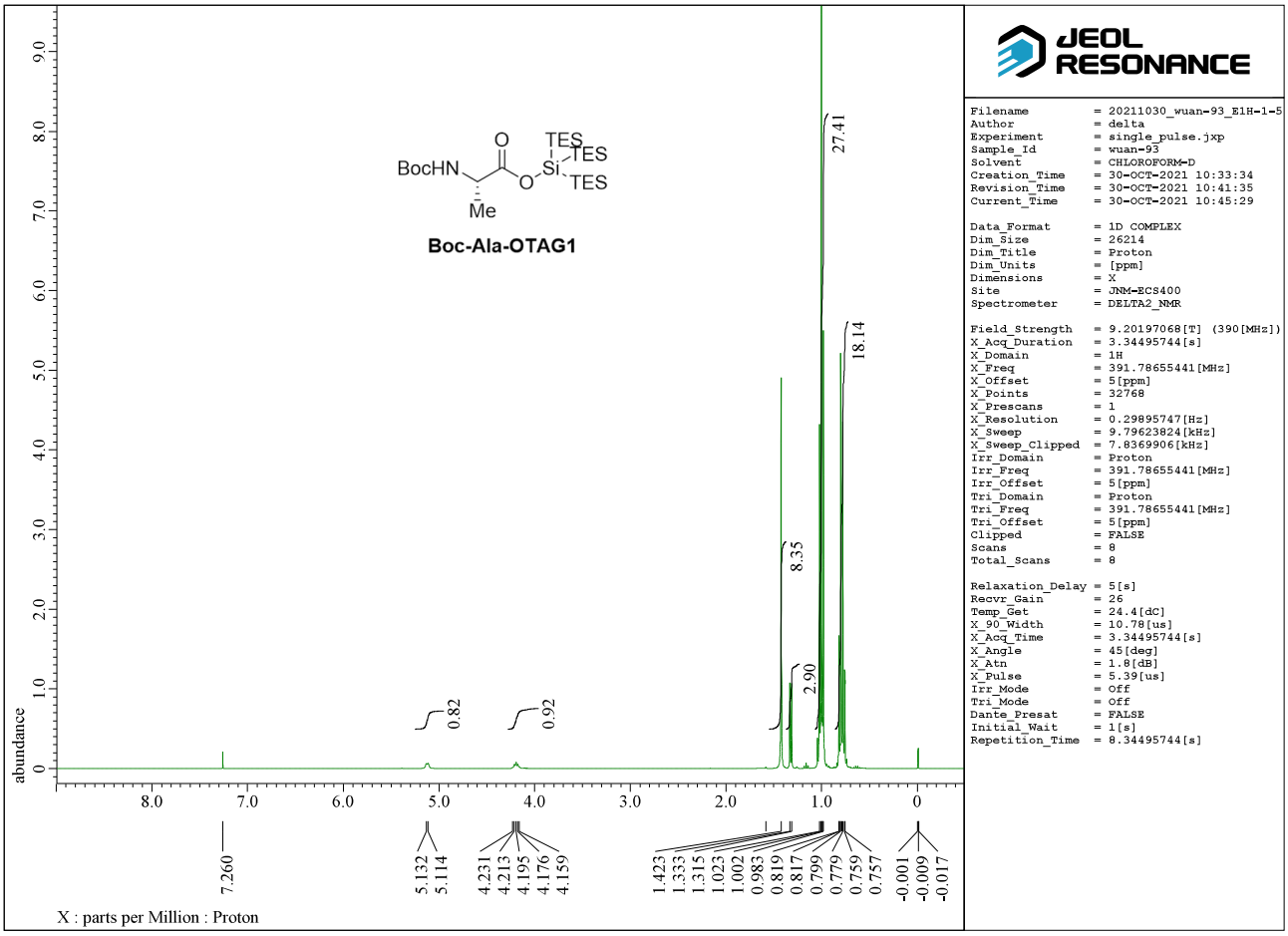
12.406	BB	2.14	822.53	23.61	82.98	
33.073	MM m	1.78	62.49	1.33	6.30	
46.264	MM m	2.03	53.66	0.91	5.41	
		Sum	**991.19**			

12.406 min = Boc-Lys(Boc)-Ile-Phe-Gly-Ser(Bn)-Leu-Ala-Phe-Leu-OH (**28**)
Conditions: water/acetonitrile = 85:15, v = 0.5 mL/min, λ = 210 nm
Column: ODS-HL column from GL Siences Inc.





JEOL RESONANCE

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Revision_Time     = 30-OCT-2021 10:41:35
Current_Time      = 30-OCT-2021 10:45:29

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Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

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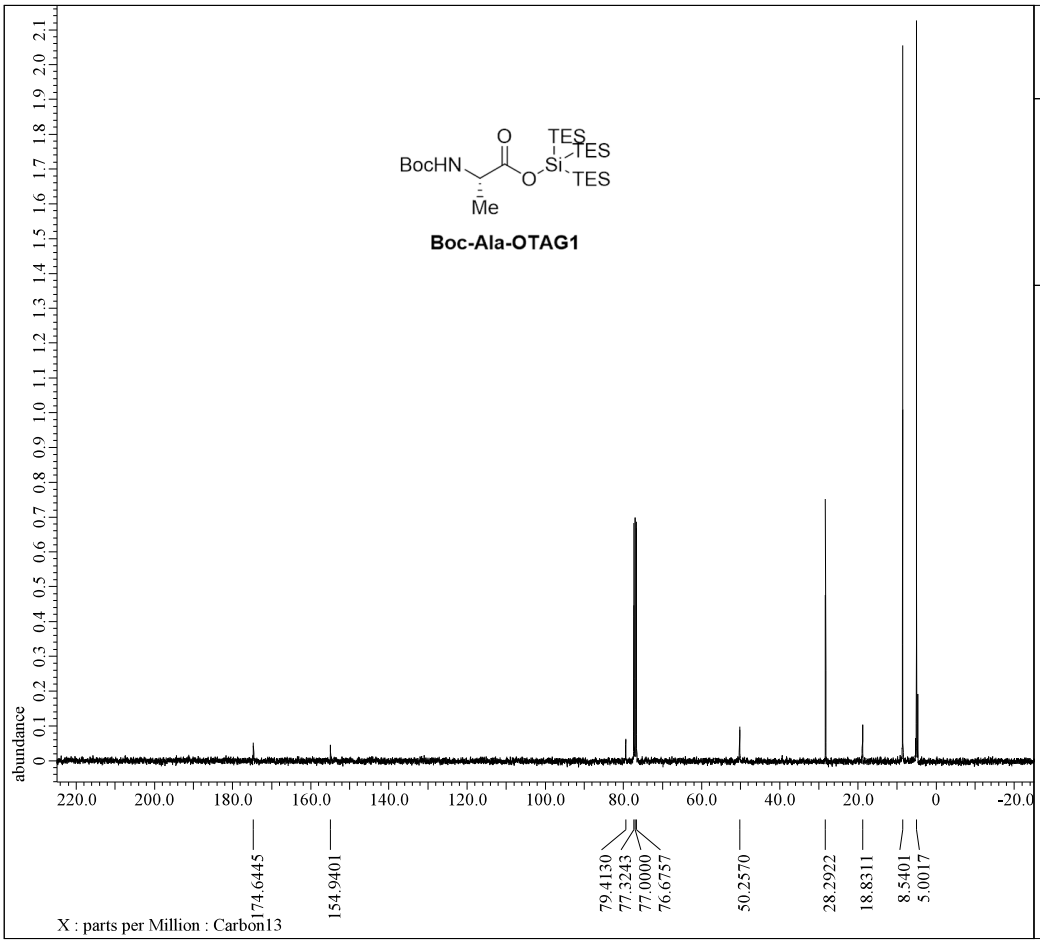

JEOL RESONANCE

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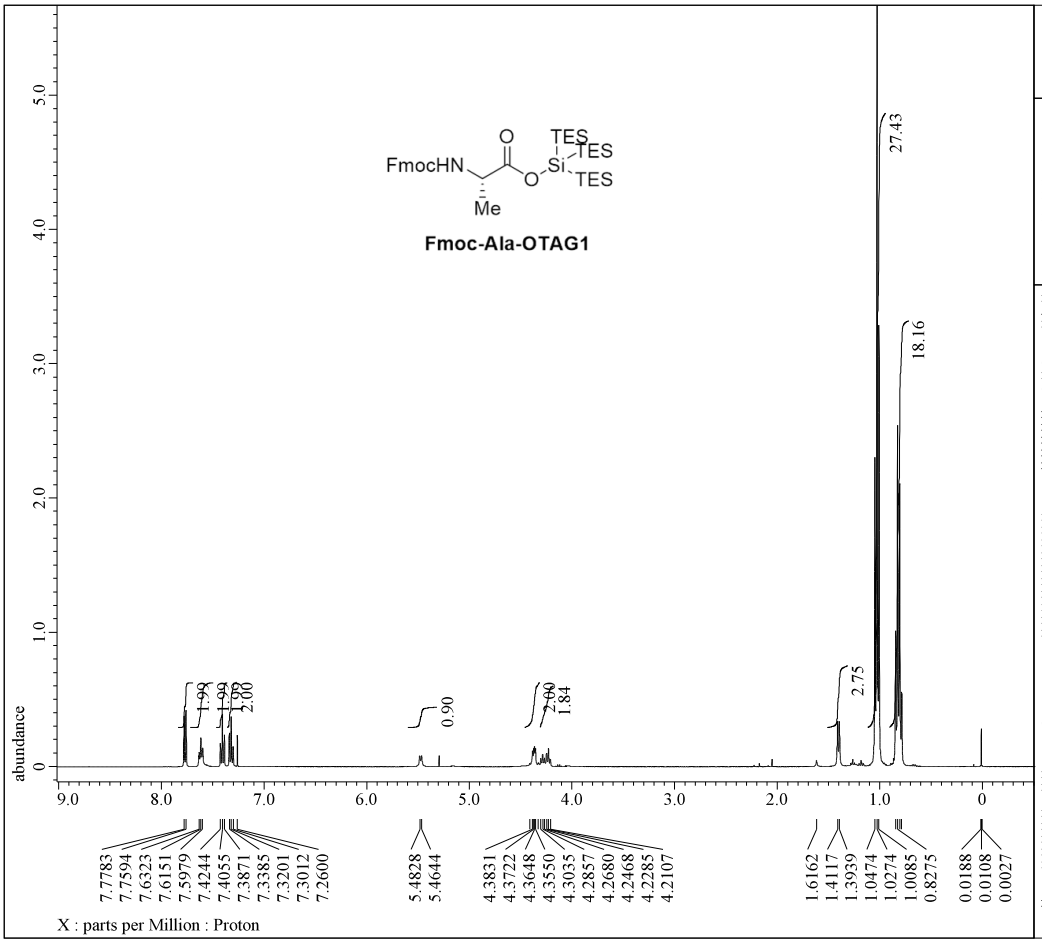
machinephase
ppm
```

```
Filename          = wuan-93_E13C-1-6.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-93
Solvent           = CHLOROFORM-D
Creation_Time     = 30-OCT-2021 10:36:51
Revision_Time     = 12-NOV-2021 12:38:32
Current_Time      = 12-NOV-2021 12:39:24

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

Field_Strength    = 9.20197068[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             = 186
Total_Scans       = 186

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]
```




```
---- 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

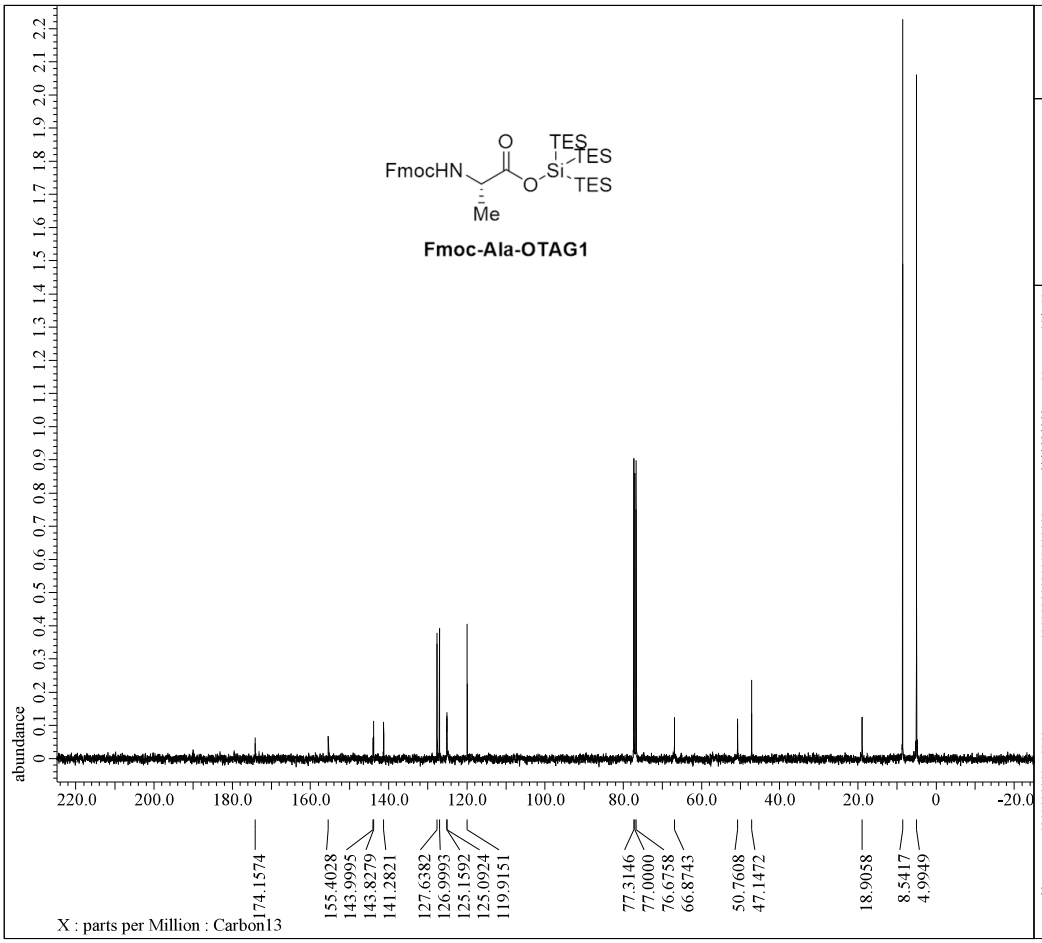
Derived from: wuan-1211-1_E1H-1-1.jdf
```

```
Filename          = wuan-1211-1_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1211-1
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 12:29:33
Revision_Time     = 12-NOV-2021 12:31:26
Current_Time      = 12-NOV-2021 12:32:29

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

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

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




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

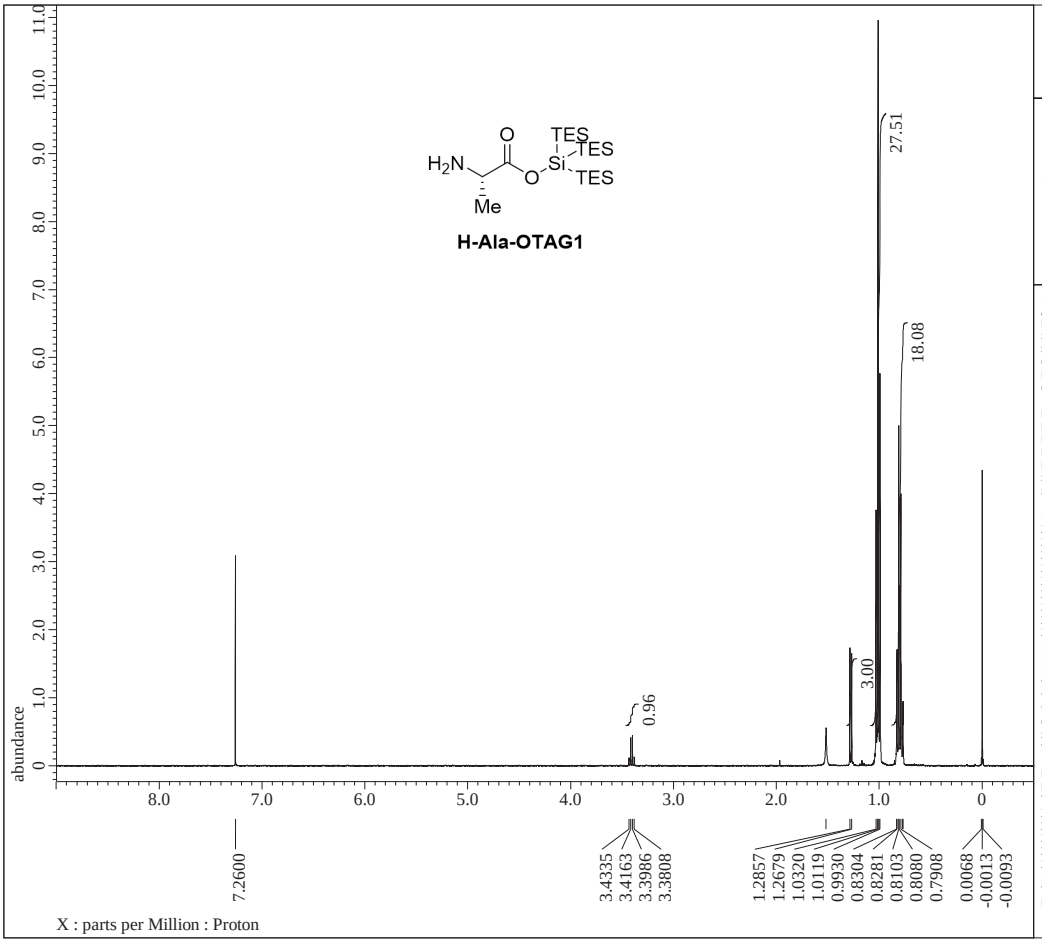
Derived from: wuan-1211-1_E13C-1-1.jdf
```

```
Filename          = wuan-1211-1_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1211-1
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 12:33:52
Revision_Time     = 12-NOV-2021 12:36:34
Current_Time      = 12-NOV-2021 12:37:19

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 108
Total_Scans       = 108

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]
```



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

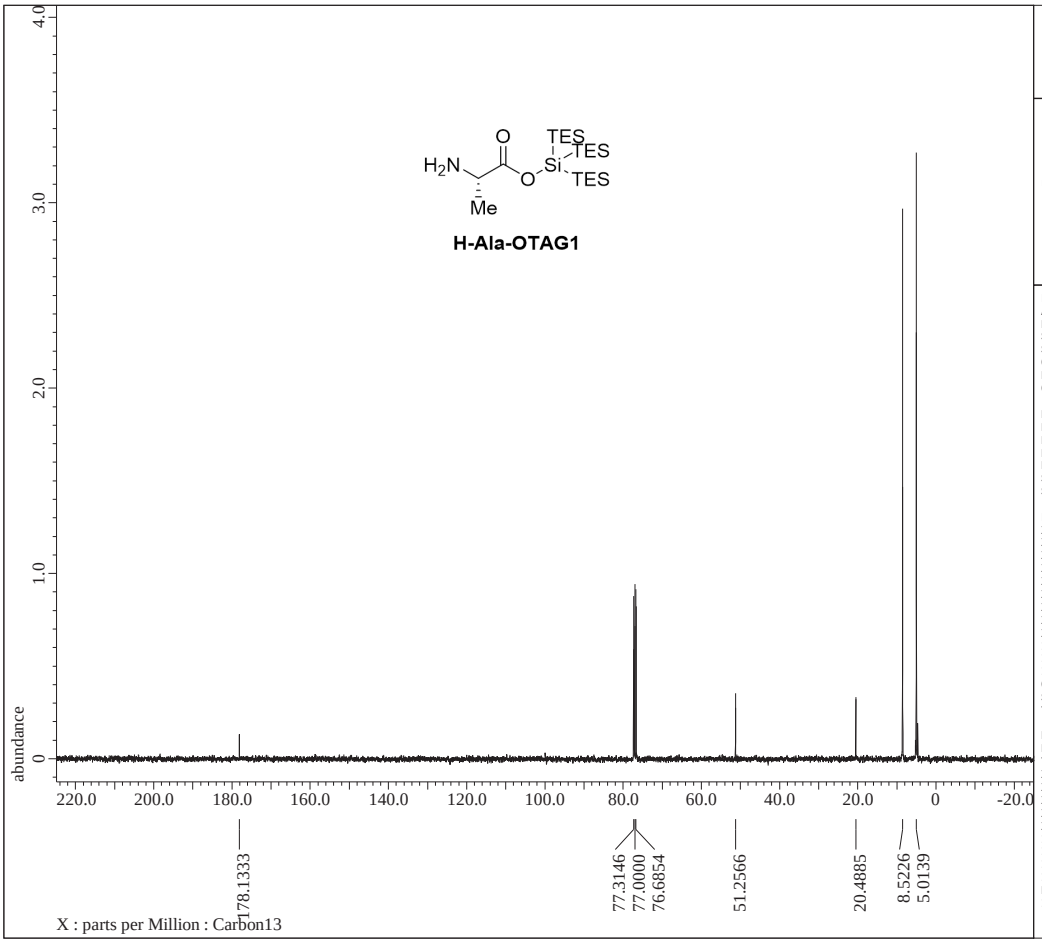
Derived from: wuan-266C-2_E1H-1-1.jdf
```

```
Filename          = wuan-266C-2_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-266C-2
Solvent           = CHLOROFORM-D
Creation_Time     =  9-DEC-2019 10:02:02
Revision_Time     = 30-OCT-2021 23:03:15
Current_Time      = 30-OCT-2021 23:03:34

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 64
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
```



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

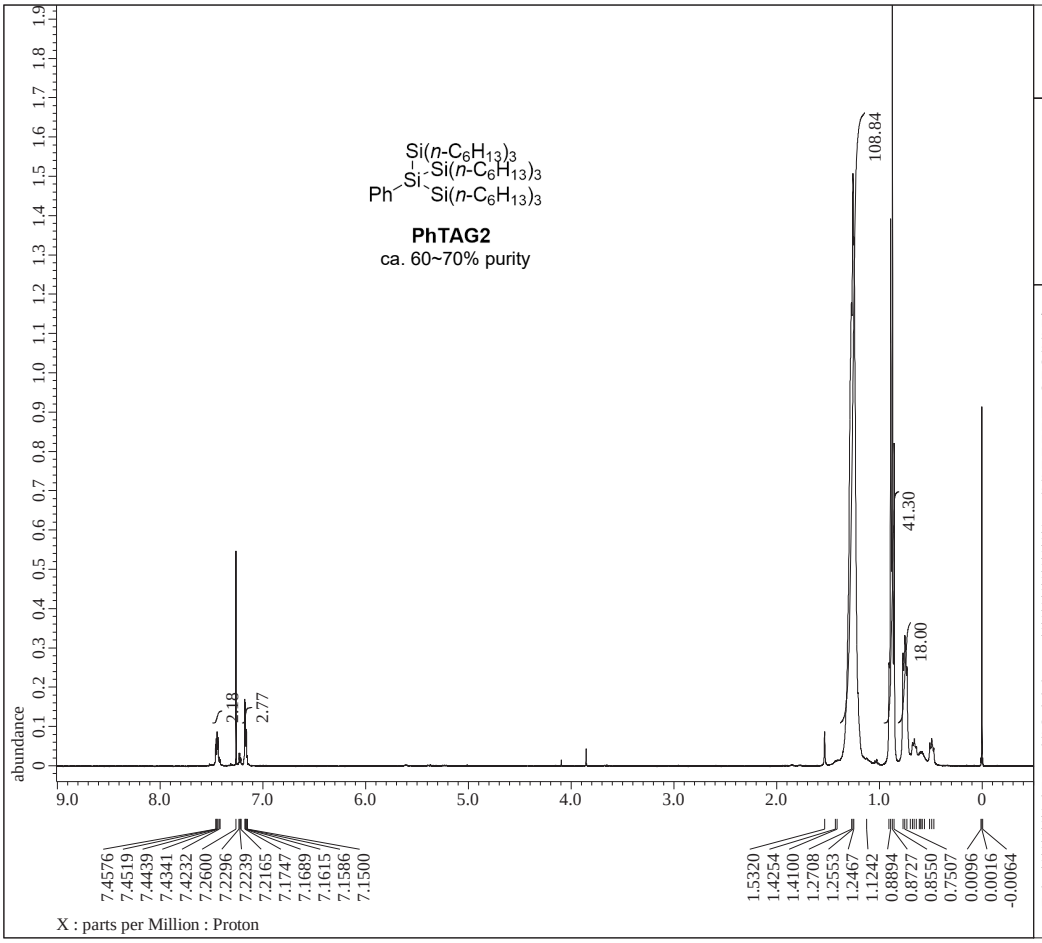
Derived from: wuan-266C-2_E13C-1-1.jdf
```

```
Filename          = wuan-266C-2_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-266C-2
Solvent           = CHLOROFORM-D
Creation_Time     = 30-OCT-2021 23:04:36
Revision_Time     = 30-OCT-2021 23:06:14
Current_Time      = 30-OCT-2021 23:06:30

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 82
Total_Scans       = 82

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

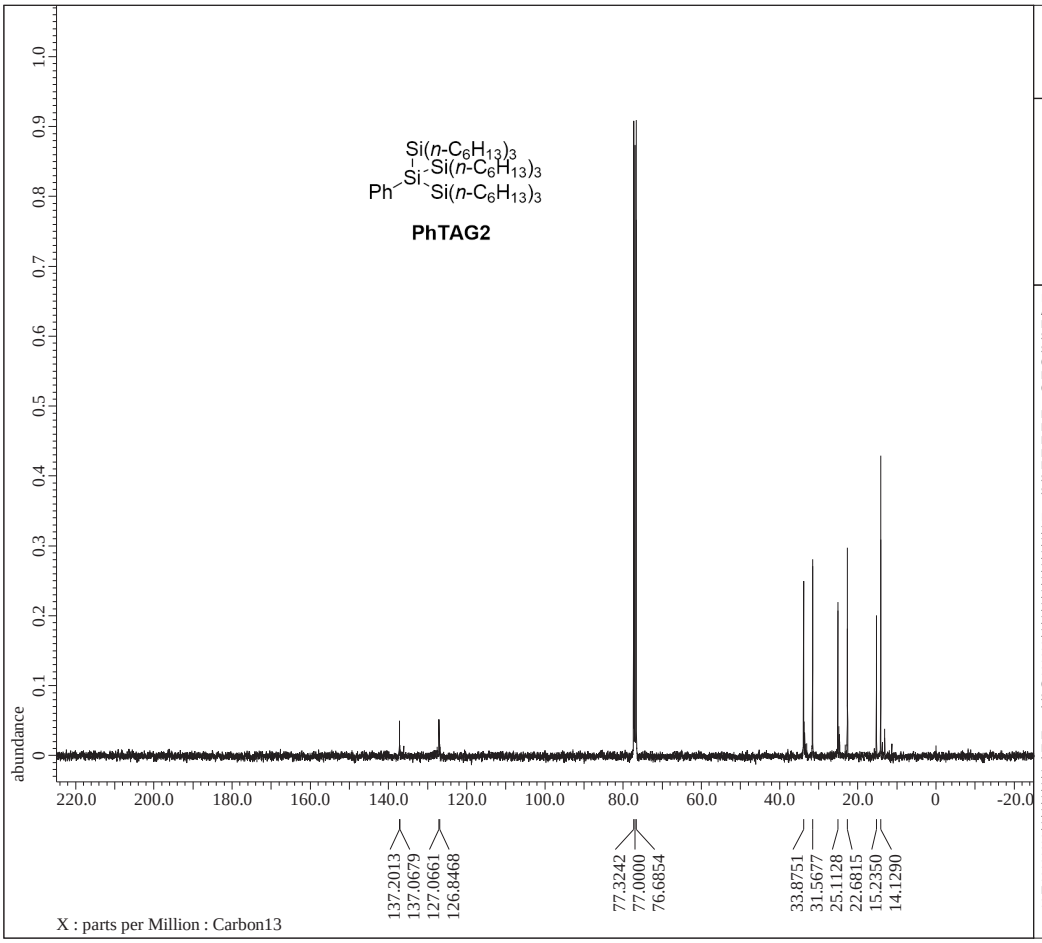
Derived from: wuan-213_E1H-1-1.jdf
```

```
Filename          = wuan-213_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-213
Solvent           = CHLOROFORM-D
Creation_Time     =  3-OCT-2019 14:27:16
Revision_Time     = 28-FEB-2022 15:01:58
Current_Time      = 28-FEB-2022 15:05:33

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        = 50
Temp_Get          = 23.3[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```




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

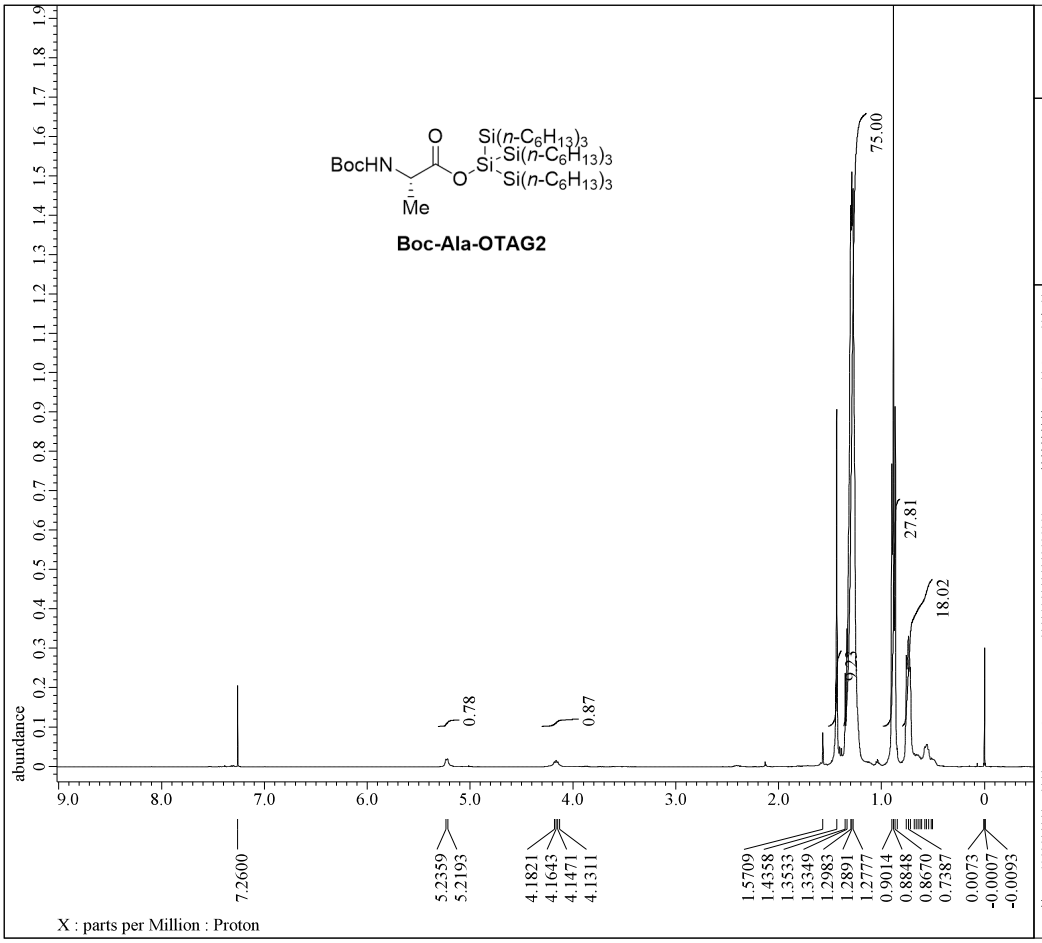
Derived from: wuan-1312-4_E13C-1-1.jdf
```

```
Filename          = wuan-1312-4_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1312-4
Solvent           = CHLOROFORM-D
Creation_Time     = 24-FEB-2022 12:49:01
Revision_Time     = 24-FEB-2022 13:13:58
Current_Time      = 24-FEB-2022 13:14:42

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 517
Total_Scans       = 517

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

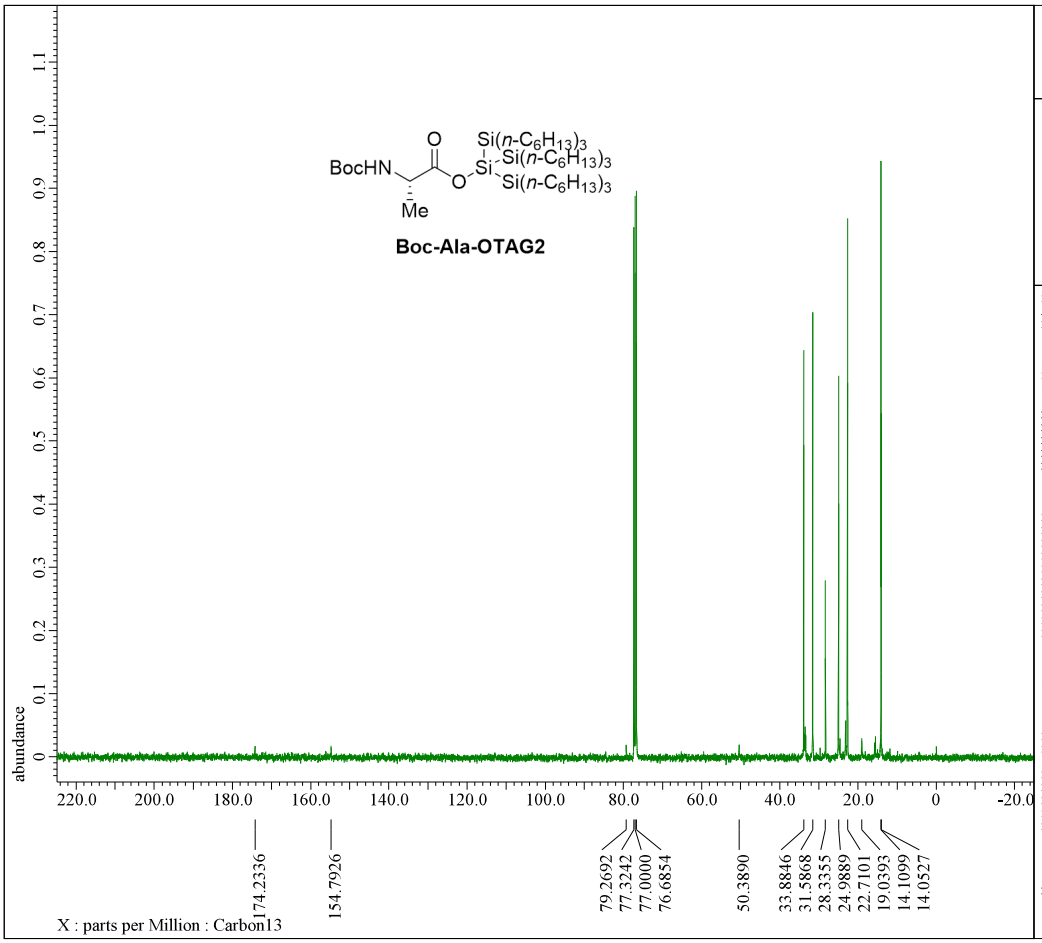


JEOL RESONANCE

```
---- 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-1215-2_E1H-1-1.jdf
```

Filename	= wuan-1215-2_E1H-1-5.jdf
Author	= delta
Experiment	= single_pulse.jxp
Sample_Id	= wuan-1215-2
Solvent	= CHLOROFORM-D
Creation_Time	= 17-NOV-2021 16:39:55
Revision_Time	= 17-NOV-2021 16:49:58
Current_Time	= 17-NOV-2021 16:50:18
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 4.36731904[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.22897343[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 22
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

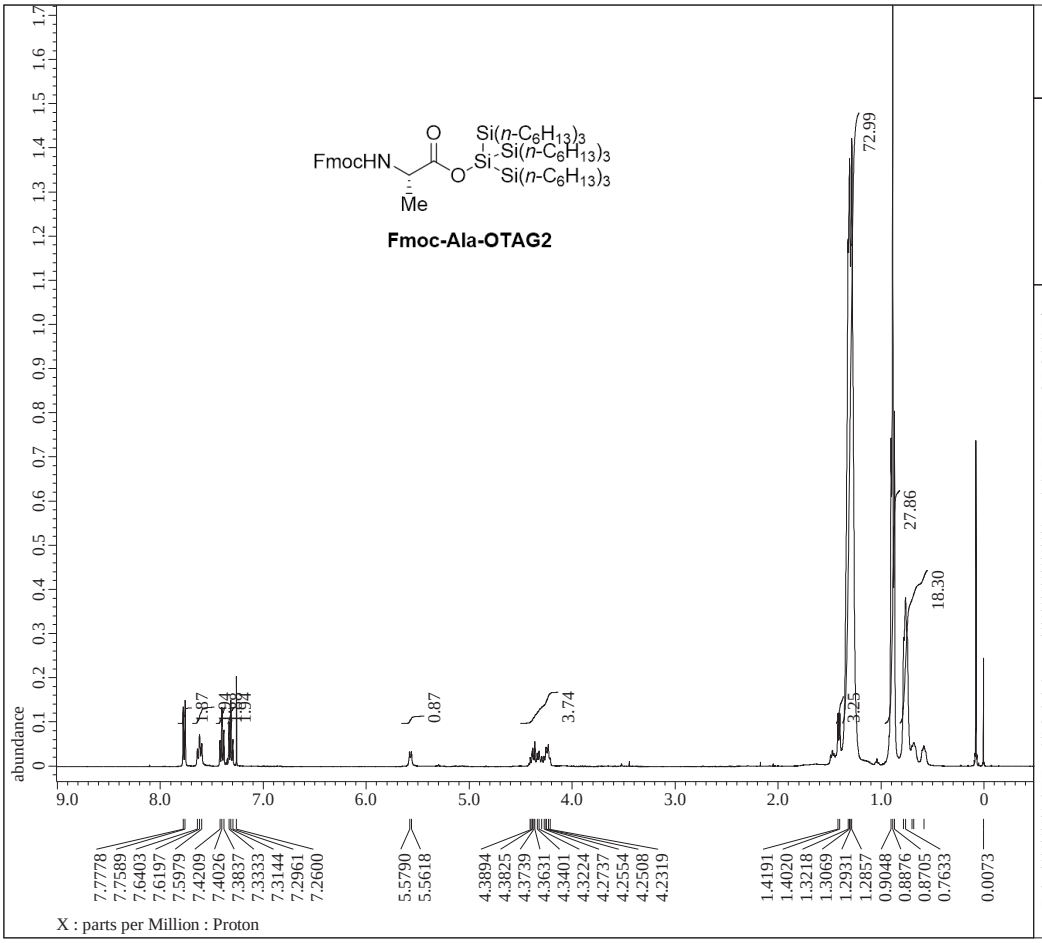


JEOL RESONANCE

```
---- 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-1215-2_E13C-1-1.jdf
```

Filename	= wuan-1215-2_E13C-1-4.jdf
Author	= delta
Experiment	= single_pulse_dec.jxp
Sample_Id	= wuan-1215-2
Solvent	= CHLOROFORM-D
Creation_Time	= 17-NOV-2021 16:44:57
Revision_Time	= 17-NOV-2021 17:12:32
Current_Time	= 17-NOV-2021 17:14:29
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 608
Total_Scans	= 608
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]



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

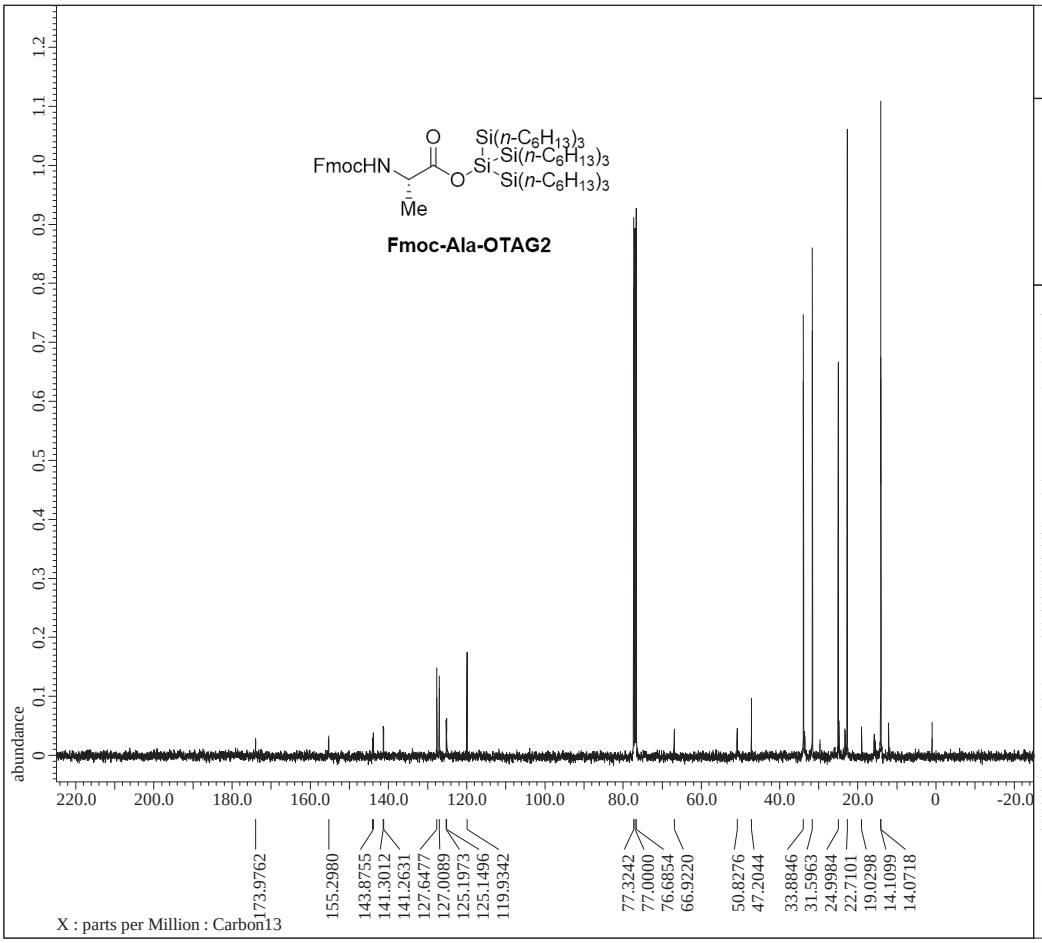
Derived from: wuan-233_E1H-1-1.jdf
```

```
Filename          = wuan-233_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-233
Solvent           = CHLOROFORM-D
Creation_Time     = 30-OCT-2021 23:32:44
Revision_Time     = 30-OCT-2021 23:42:17
Current_Time      = 30-OCT-2021 23:43:22

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        = 20
Temp_Get          = 24.7[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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

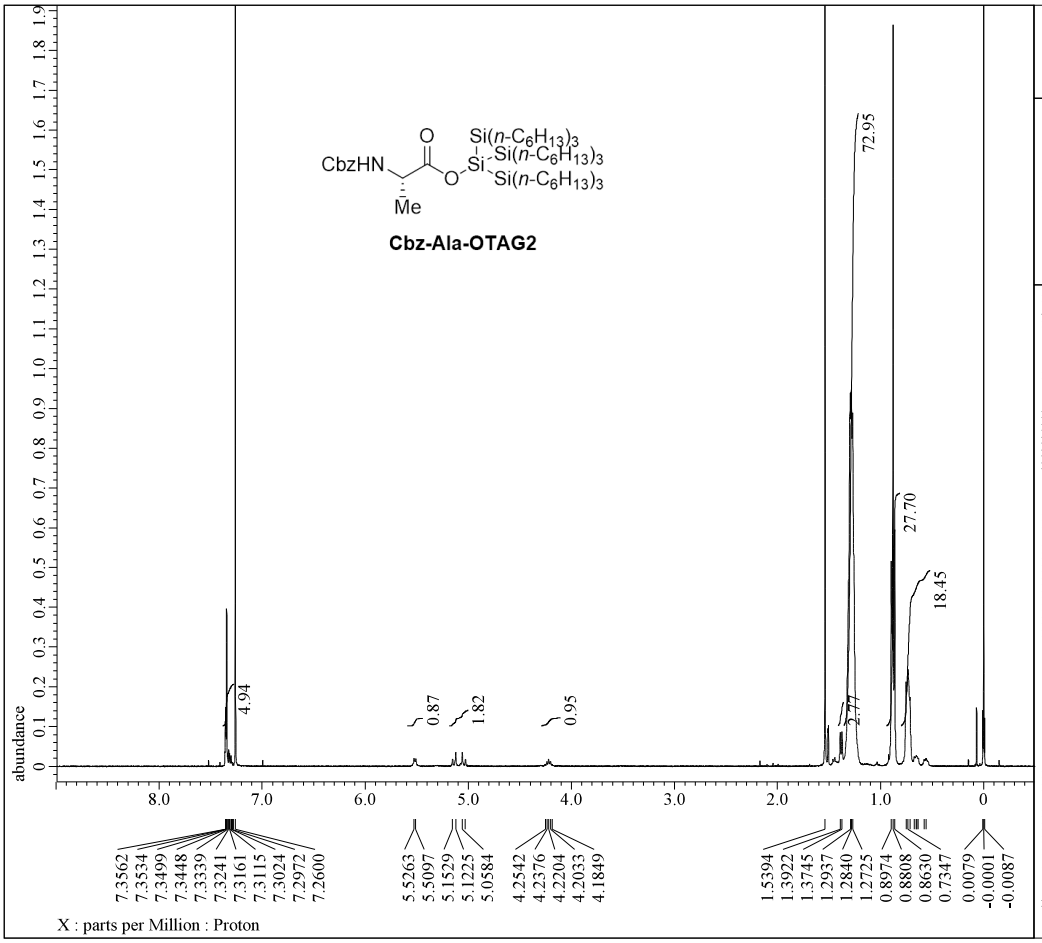
Derived from: wuan-233_E13C-1-1.jdf
```

```
Filename          = wuan-233_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-233
Solvent           = CHLOROFORM-D
Creation_Time     = 30-OCT-2021 23:36:24
Revision_Time     = 30-OCT-2021 23:52:29
Current_Time      = 30-OCT-2021 23:52:44

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 283
Total_Scans       = 283

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]
```



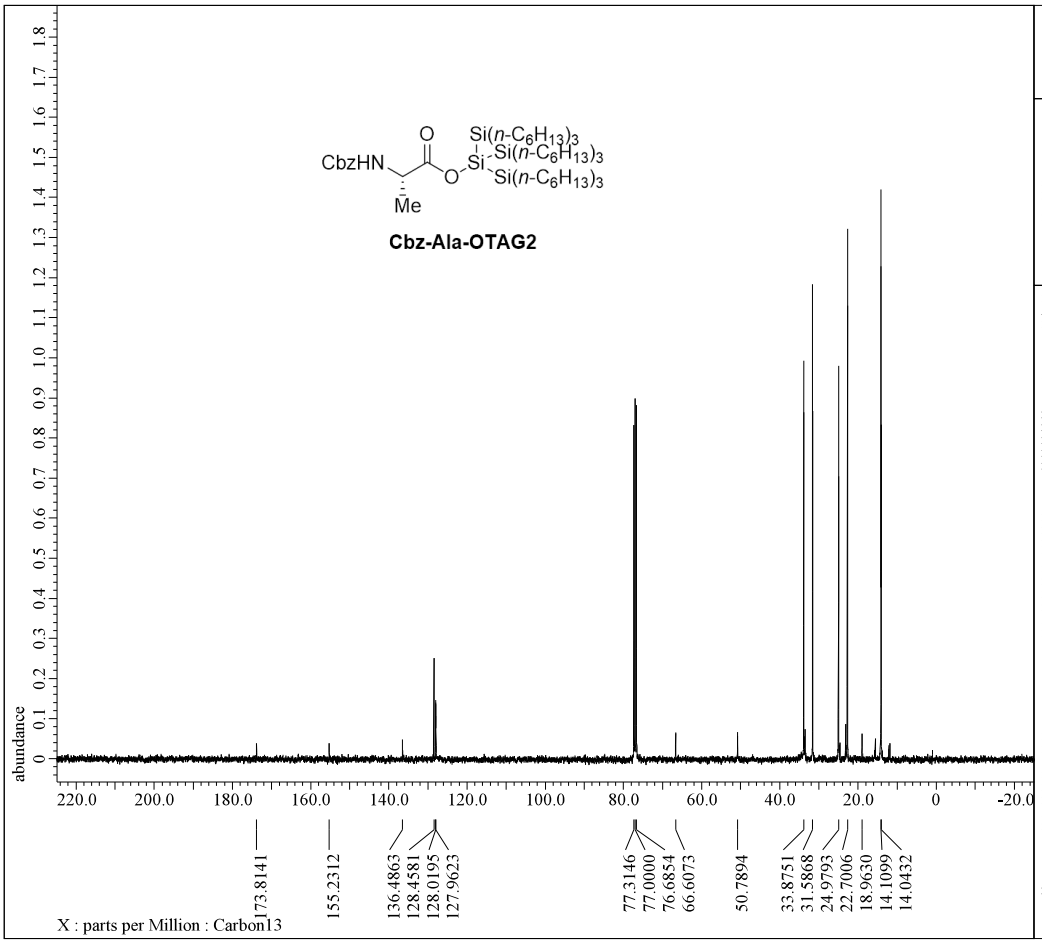

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-231_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-231
Solvent           = CHLOROFORM-D
Creation_Time     =  6-NOV-2019 18:51:51
Revision_Time     = 12-NOV-2021 13:10:19
Current_Time      = 12-NOV-2021 13:11:46

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
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

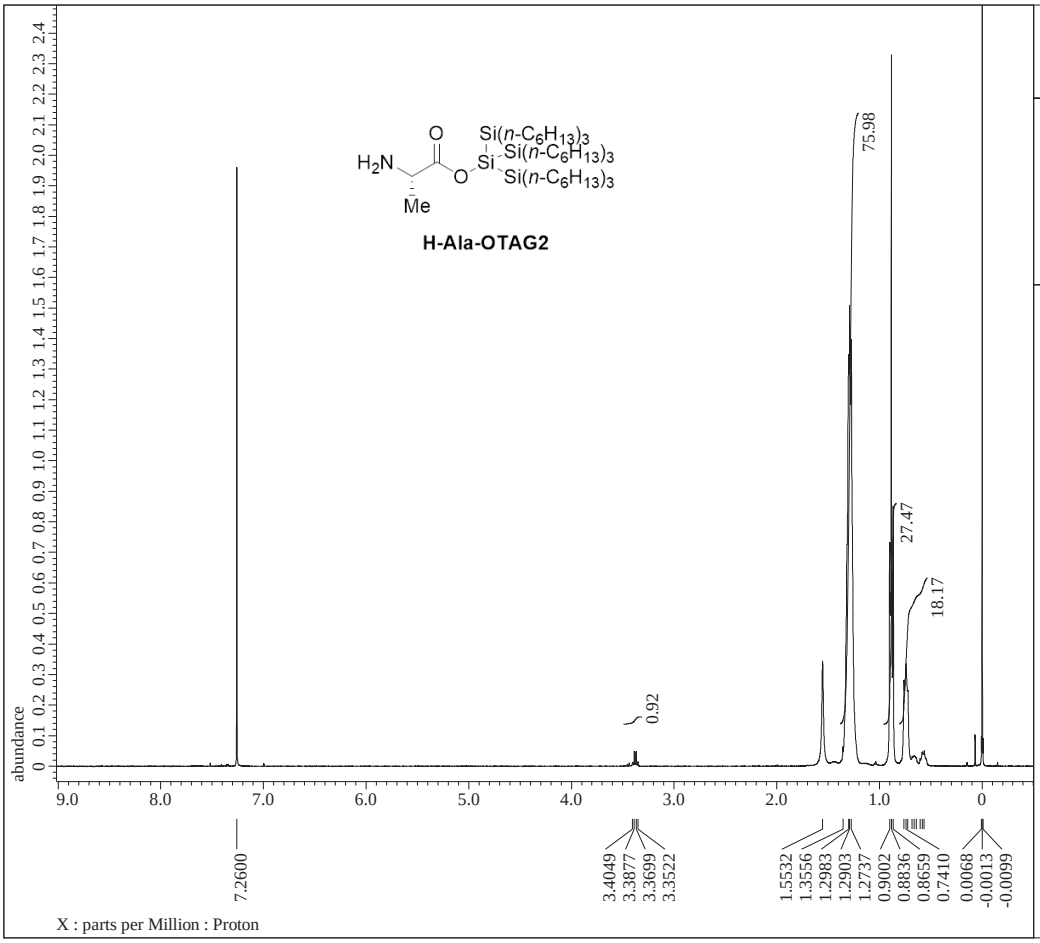
Derived from: wuan-403_E13C-1-1.jdf
```

```
Filename          = wuan-403_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-403
Solvent           = CHLOROFORM-D
Creation_Time     = 31-OCT-2021 00:01:18
Revision_Time     = 31-OCT-2021 00:14:00
Current_Time      = 31-OCT-2021 00:15:18

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             = 297
Total_Scans       = 297

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.4[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]
```



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

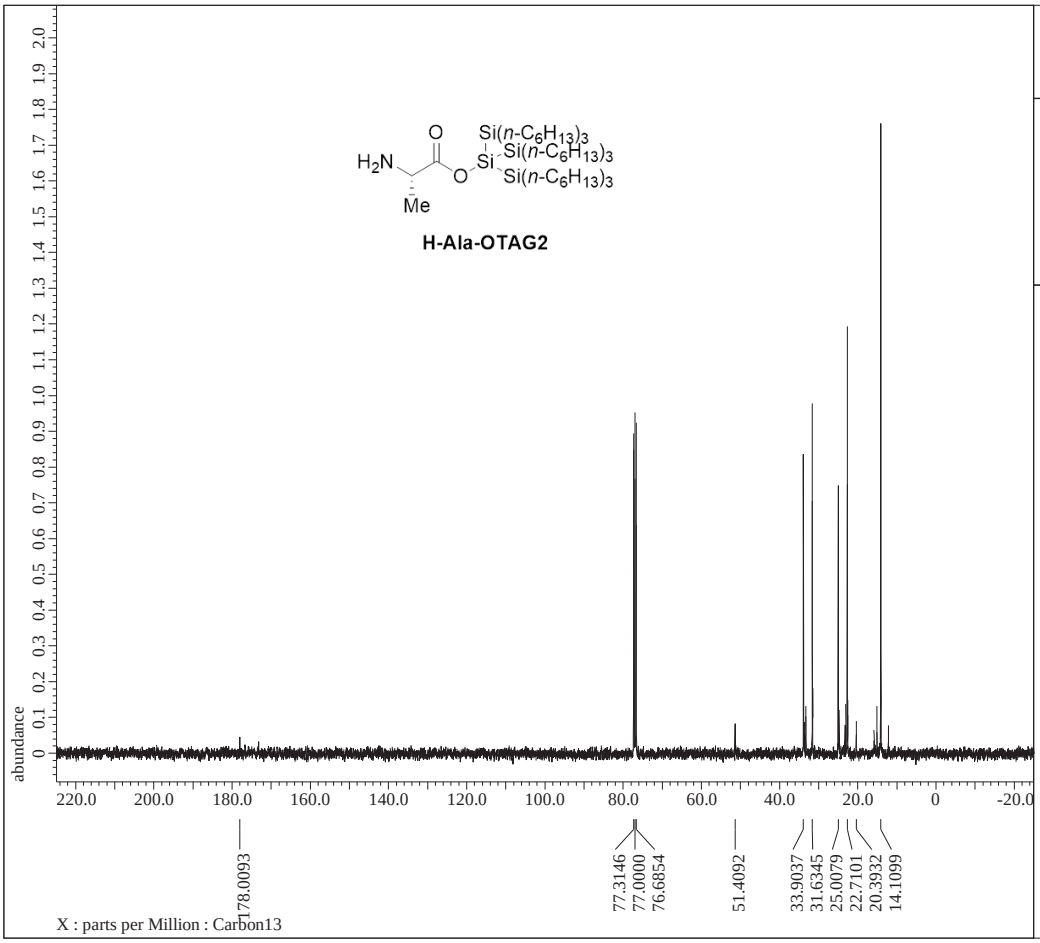
Derived from: wuan-224_E1H-1-1.jdf
```

```
Filename          = wuan-224_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-224
Solvent           = CHLOROFORM-D
Creation_Time     =  8-OCT-2019 18:49:38
Revision_Time     = 31-OCT-2021 00:22:13
Current_Time      = 31-OCT-2021 00:23:00

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        = 42
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

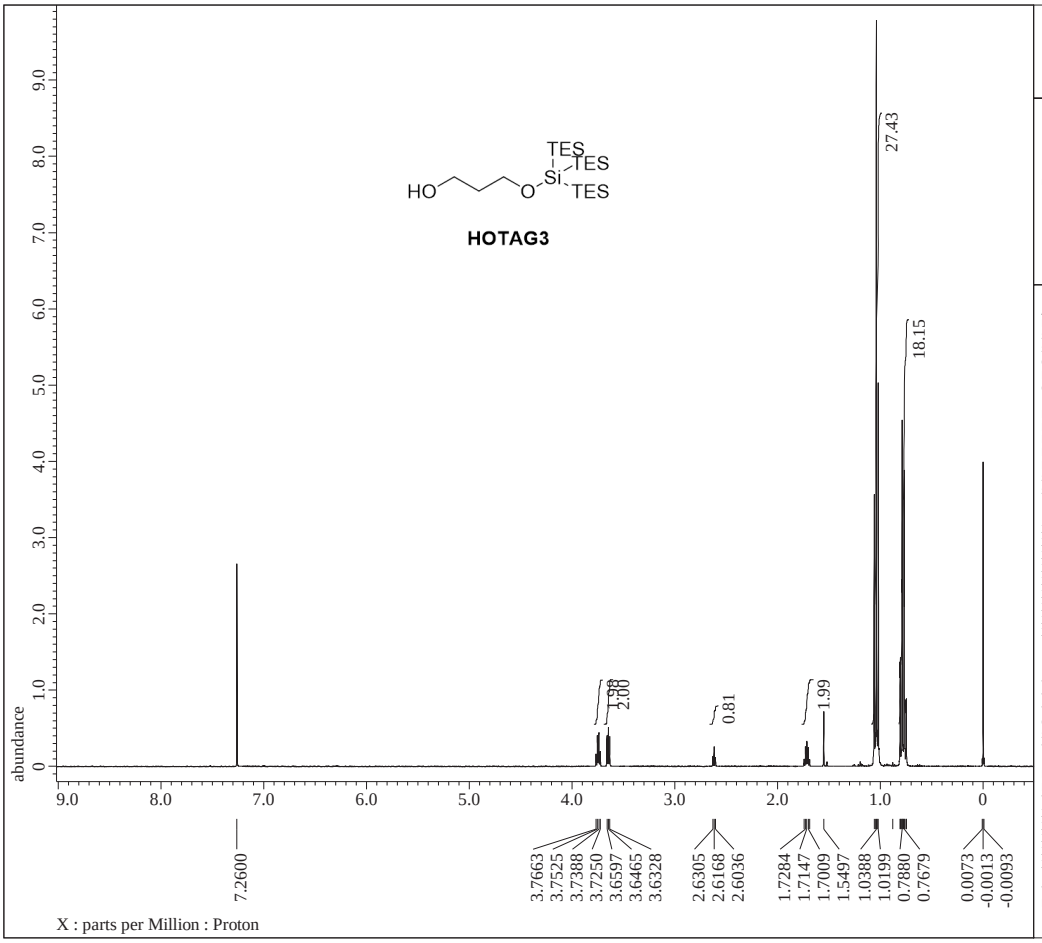
Derived from: wuan-235_E13C-1-1.jdf
```

```
Filename          = wuan-235_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-235
Solvent           = CHLOROFORM-D
Creation_Time     = 31-OCT-2021 00:26:10
Revision_Time     = 31-OCT-2021 00:28:19
Current_Time      = 31-OCT-2021 00:28:46

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             = 79
Total_Scans       = 79

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



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

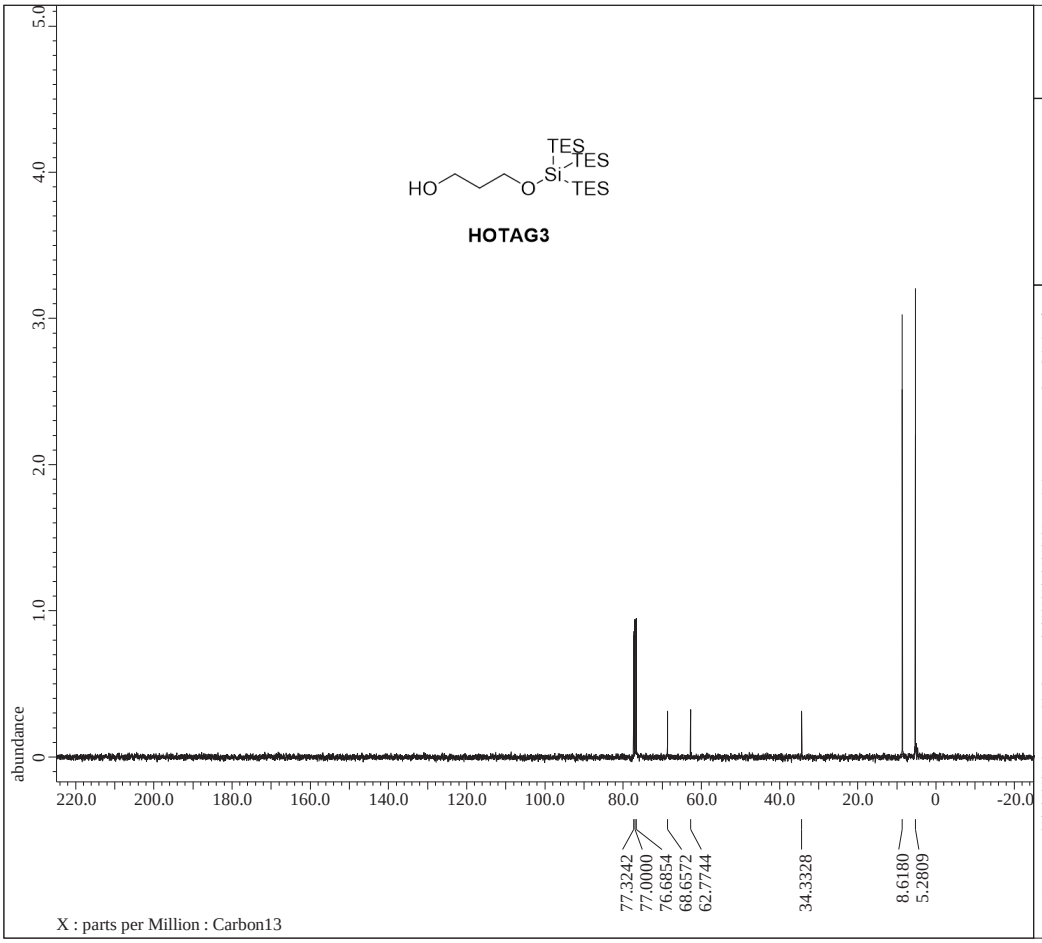
Derived from: wuan-330_E1H-1-1.jdf
```

```
Filename          = wuan-330_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-330
Solvent           = CHLOROFORM-D
Creation_Time     = 29-JAN-2020 19:31:50
Revision_Time     = 31-OCT-2021 00:35:23
Current_Time      = 31-OCT-2021 00:36:22

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        = 40
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
```



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

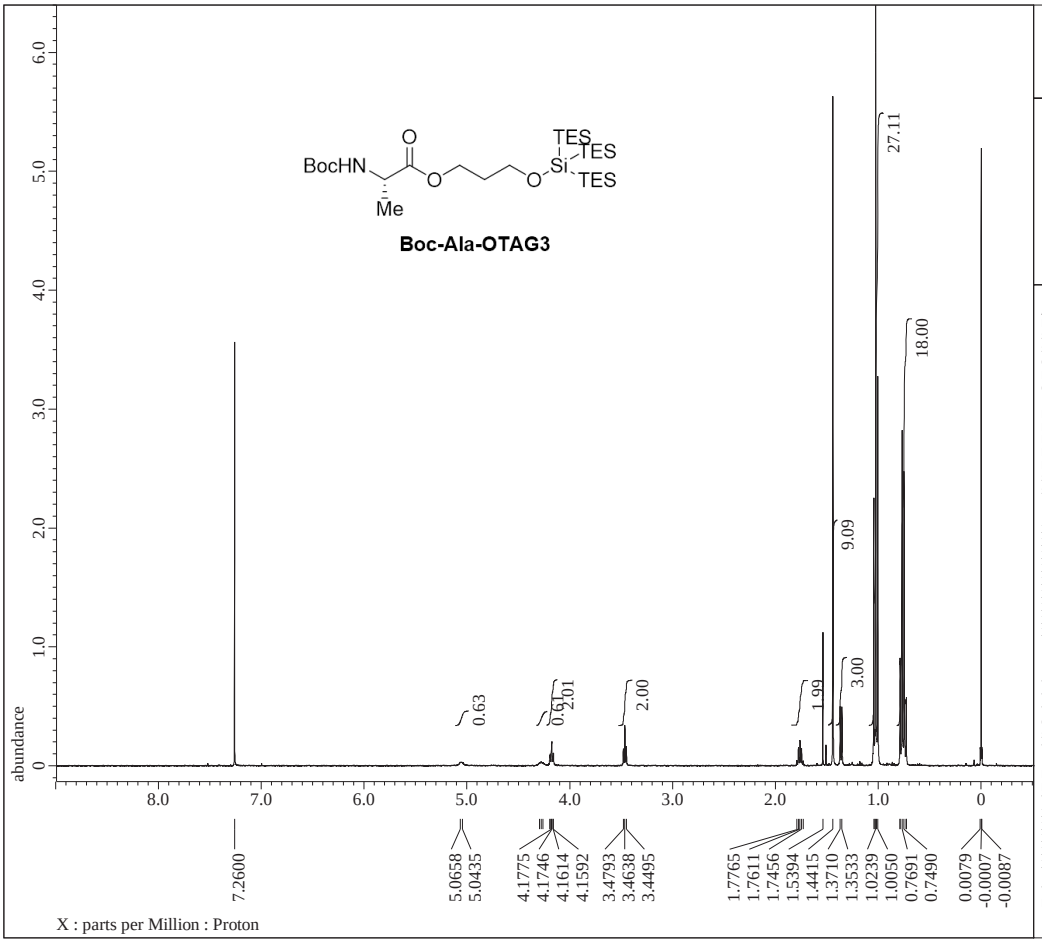
Derived from: wuan-348_E13C-1-1.jdf
```

```
Filename          = wuan-348_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-348
Solvent           = CHLOROFORM-D
Creation_Time     = 31-OCT-2021 00:40:32
Revision_Time     = 31-OCT-2021 00:38:39
Current_Time      = 31-OCT-2021 00:38:50

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 41
Total_Scans       = 41

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]
```



JEOL RESONANCE

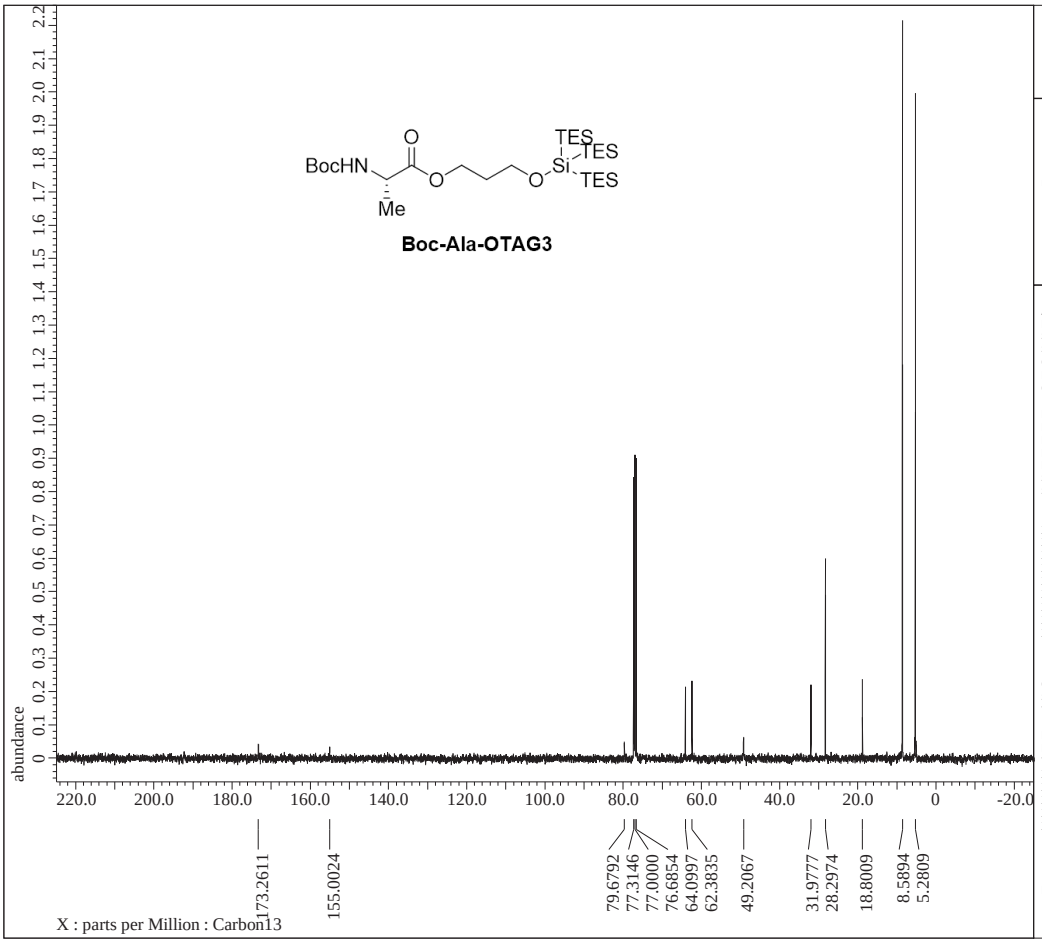
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = wuan-335_E1H-1-7.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-335
Solvent           = CHLOROFORM-D
Creation_Time     =  4-FEB-2020 11:56:18
Revision_Time     = 12-NOV-2021 13:14:13
Current_Time      = 12-NOV-2021 13:15:07

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        = 54
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

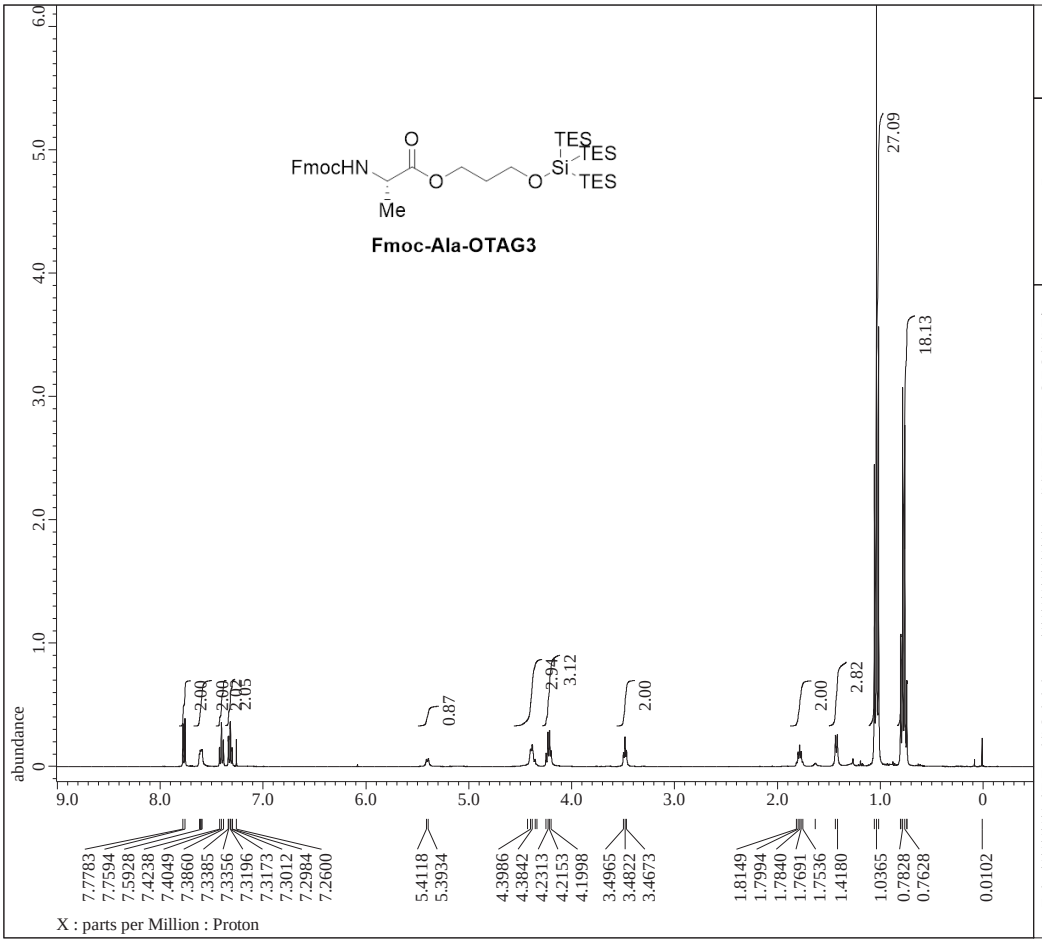
Derived from: wuan-335_E13C-1-1.jdf

Filename          = wuan-335_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-335
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 13:05:11
Revision_Time     =  3-NOV-2021 13:09:40
Current_Time      =  3-NOV-2021 13:09:53

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 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             = 134
Total_Scans       = 134

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




```
---- 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

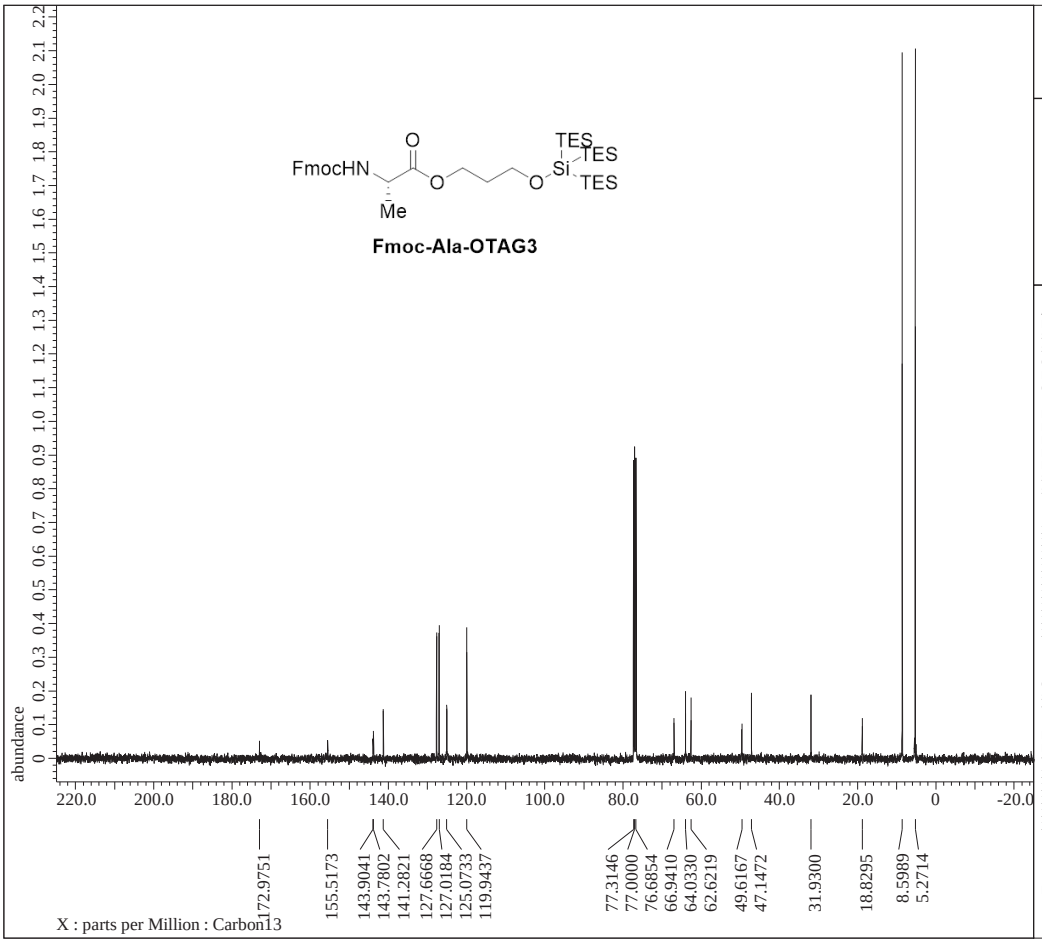
Derived from: wuan-347_E1H-1-1.jdf
```

```
Filename          = wuan-347_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-347
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 13:18:26
Revision_Time     =  3-NOV-2021 13:22:06
Current_Time      =  3-NOV-2021 13:22:56

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        = 22
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
```




```
---- 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

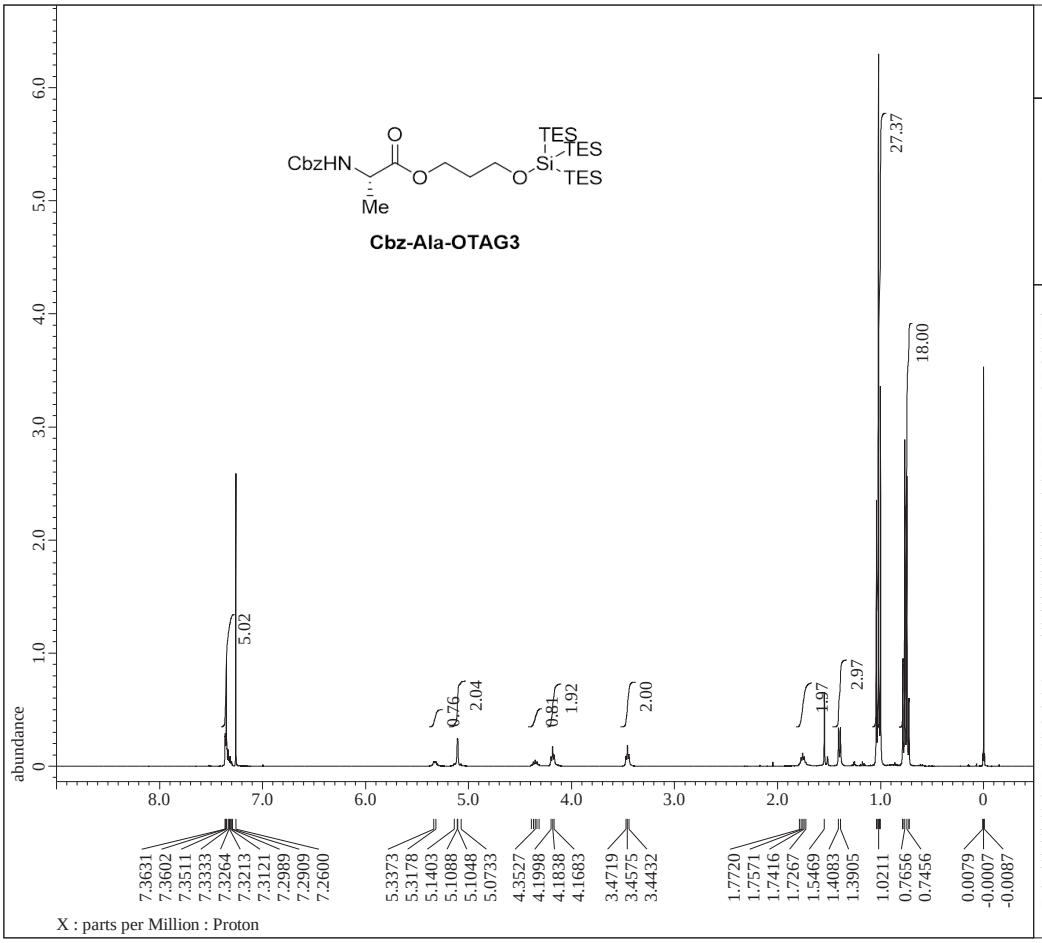
Derived from: wuan-347_E13C-1-1.jdf
```

```
Filename          = wuan-347_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-347
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 13:22:21
Revision_Time     =  3-NOV-2021 13:25:53
Current_Time      =  3-NOV-2021 13:26:57

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 118
Total_Scans       = 118

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]
```



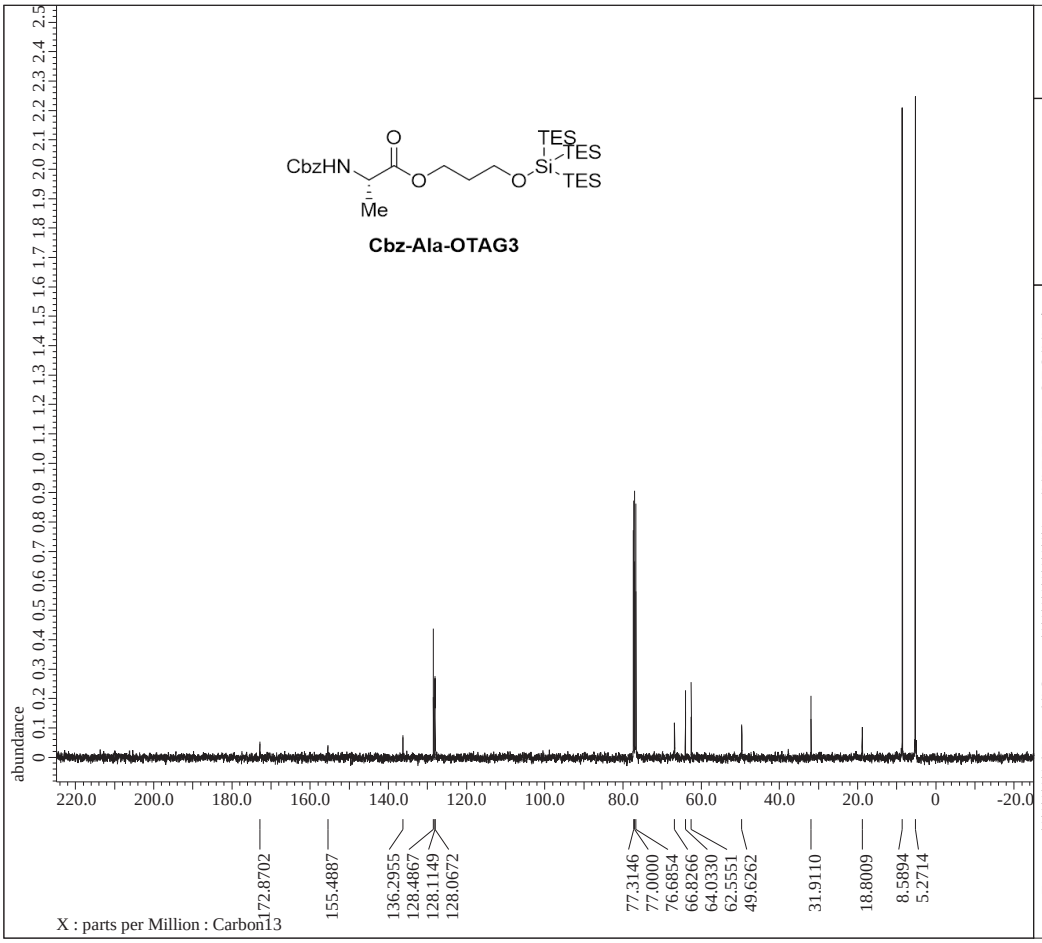

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = wuan-352_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-352
Solvent           = CHLOROFORM-D
Creation_Time     = 14-FEB-2020 14:31:53
Revision_Time     =  3-NOV-2021 13:04:29
Current_Time      =  3-NOV-2021 13:05:22

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          = 23[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

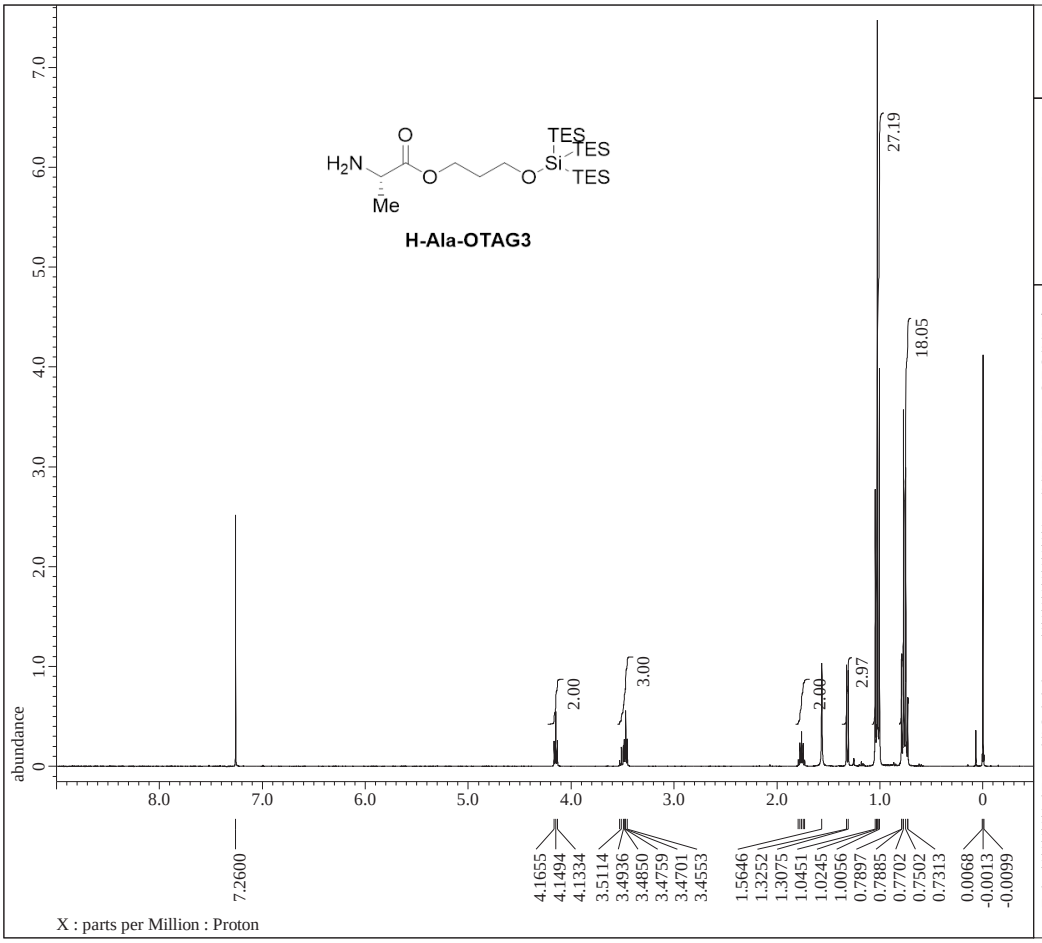
Derived from: wuan-352_E13C-1-1.jdf

Filename          = wuan-352_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-352
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 13:36:06
Revision_Time     =  3-NOV-2021 13:38:10
Current_Time      =  3-NOV-2021 13:38:39

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 89
Total_Scans       = 89

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



JEOL RESONANCE

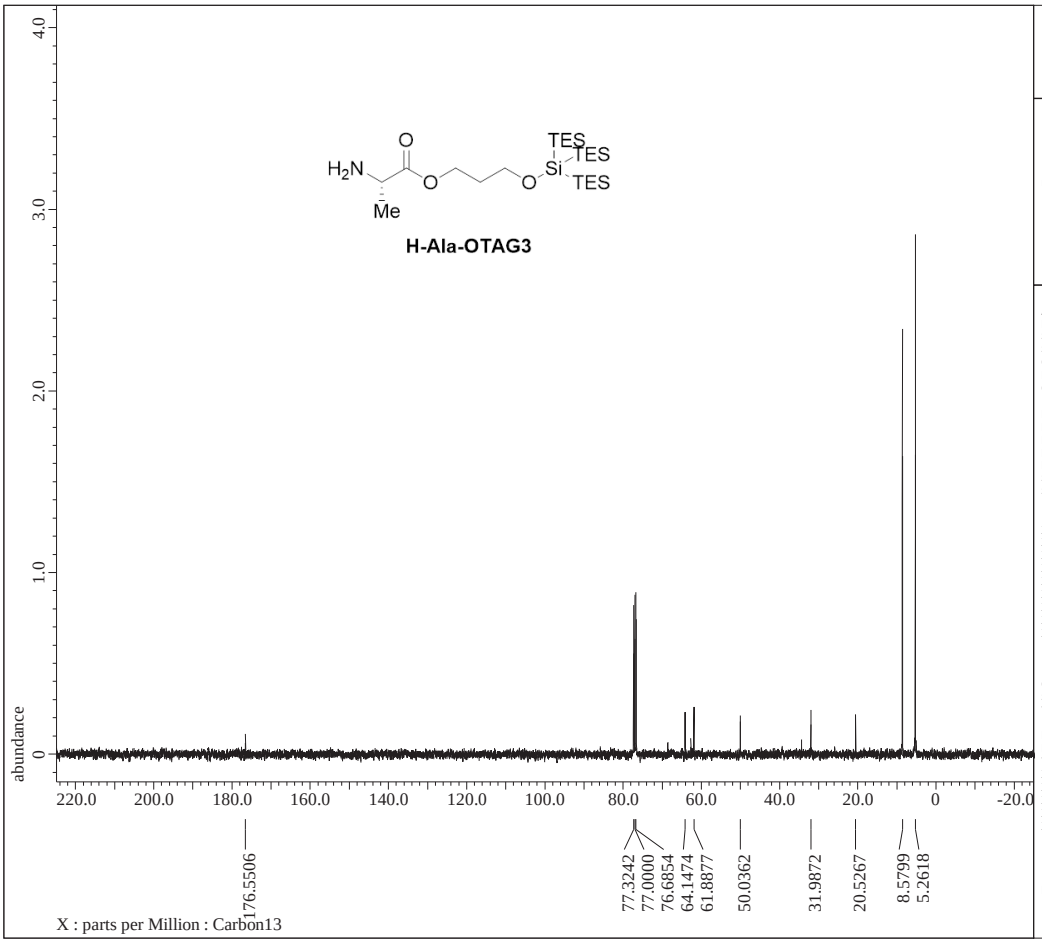
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-354_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-354
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2020 13:01:56
Revision_Time     =  3-NOV-2021 13:15:08
Current_Time      =  3-NOV-2021 13:16:09

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        = 44
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
```



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

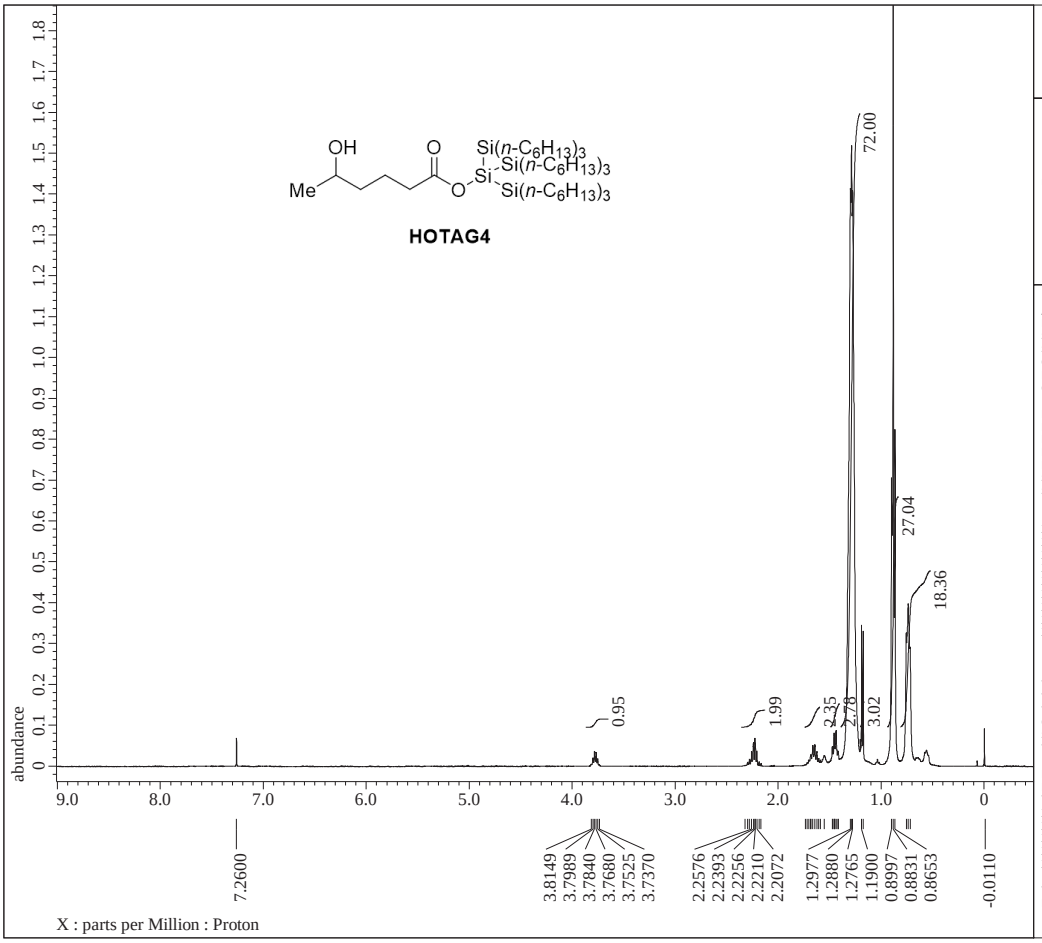
Derived from: wuan-354_E13C-1-1.jdf
```

```
Filename          = wuan-354_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-354
Solvent           = CHLOROFORM-D
Creation_Time     =  3-NOV-2021 13:48:27
Revision_Time     =  3-NOV-2021 13:47:35
Current_Time      =  3-NOV-2021 13:48:37

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 29
Total_Scans       = 29

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



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

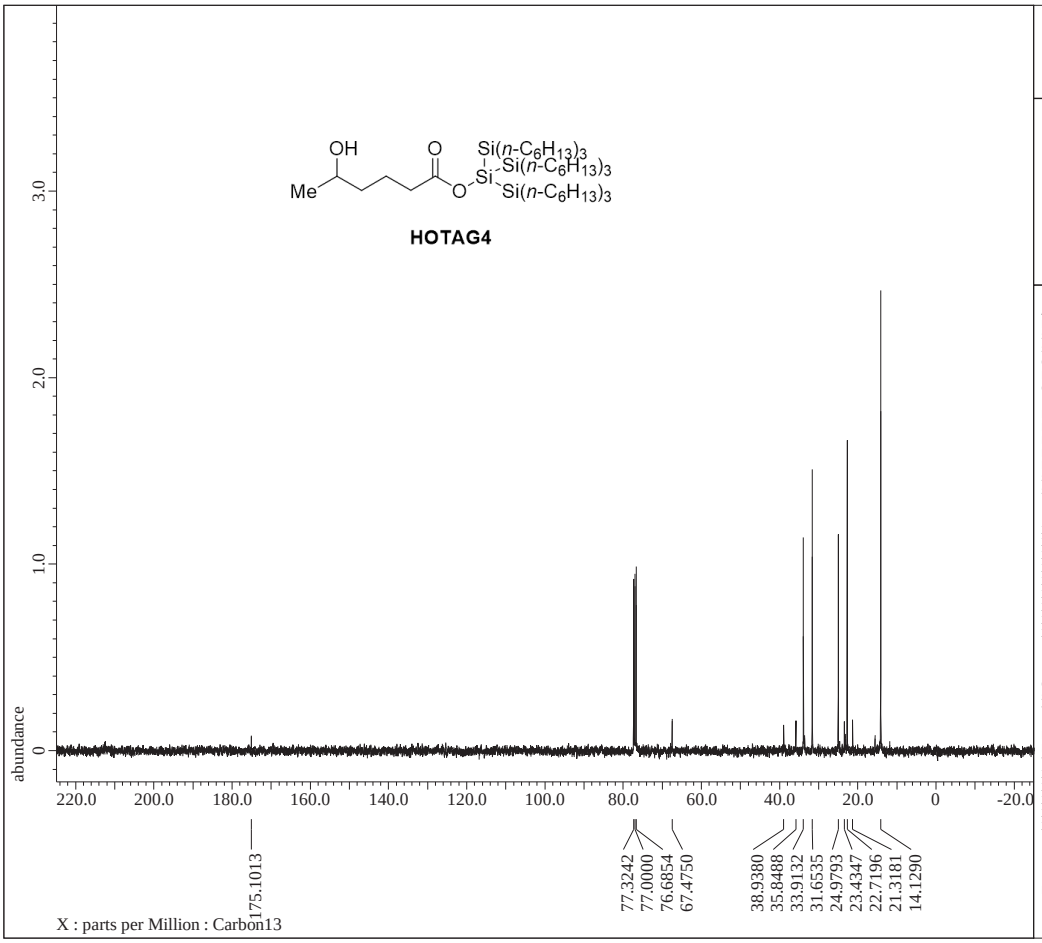
Derived from: wuan-382_E1H-1-1.jdf
```

```
Filename          = wuan-382_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-382
Solvent           = CHLOROFORM-D
Creation_Time     = 27-OCT-2021 12:53:48
Revision_Time     = 27-OCT-2021 13:20:33
Current_Time      = 27-OCT-2021 13:22:58

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 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
```



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

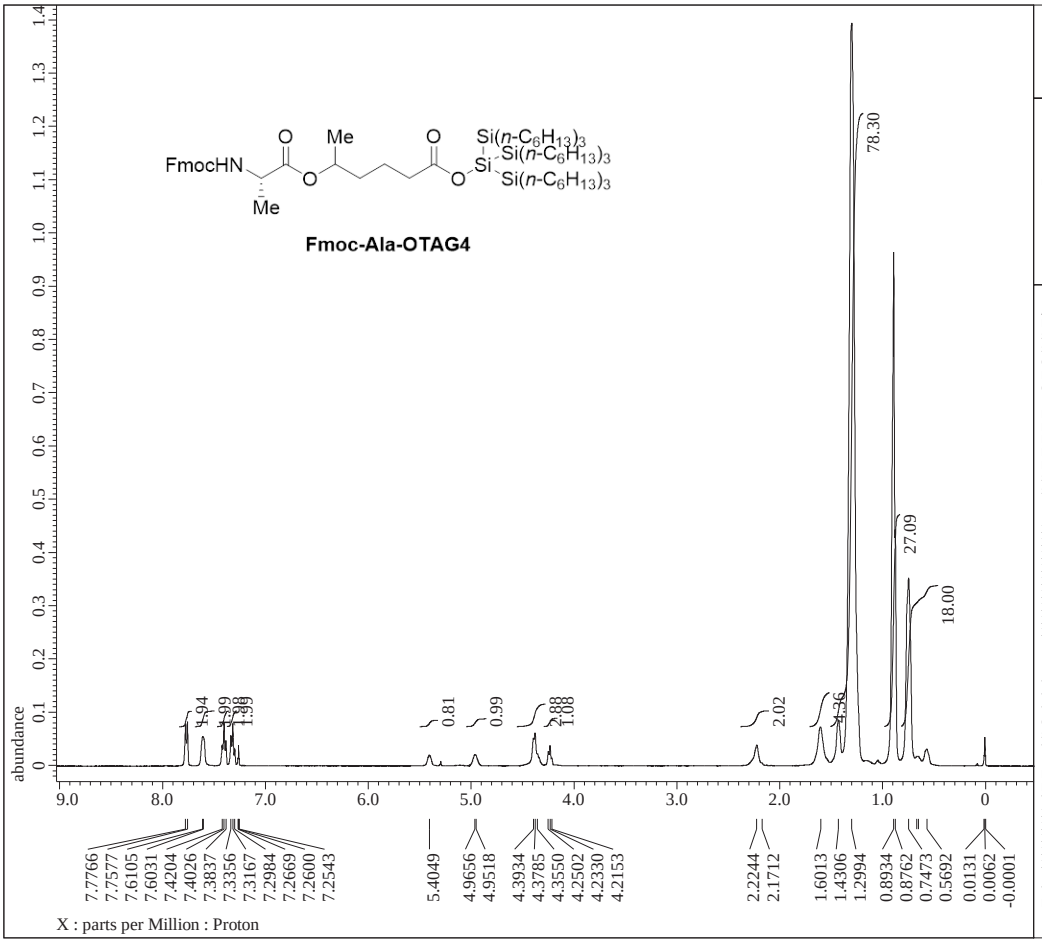
Derived from: wuan-382_E13C-2-1.jdf
```

```
Filename          = wuan-382_E13C-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-382
Solvent           = CHLOROFORM-D
Creation_Time     = 27-OCT-2021 13:11:37
Revision_Time     = 27-OCT-2021 13:10:47
Current_Time      = 27-OCT-2021 13:12:47

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             = 27
Total_Scans       = 27

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]
```



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

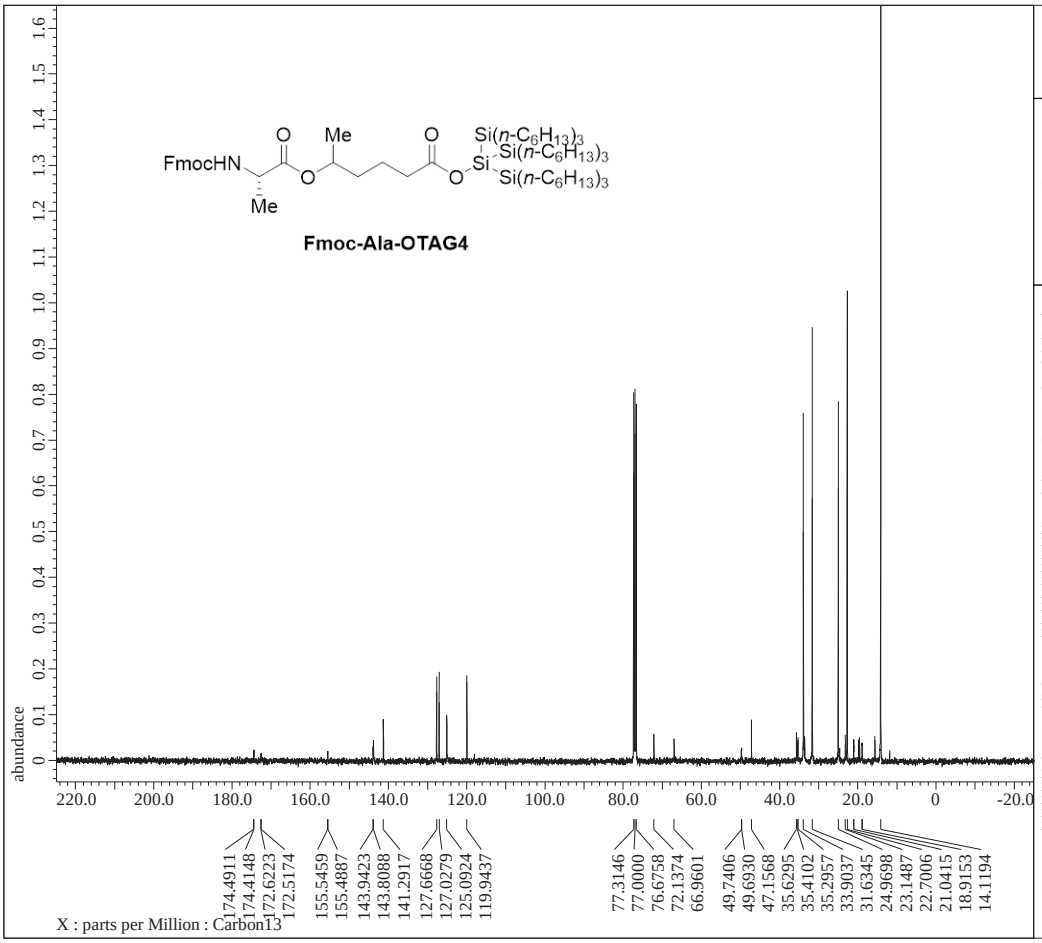
Derived from: wuan-1213_E1H-1-1.jdf
```

```
Filename          = wuan-1213_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1213
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 17:33:16
Revision_Time     = 12-NOV-2021 17:40:28
Current_Time      = 12-NOV-2021 17:41:07

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          = 23.6[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

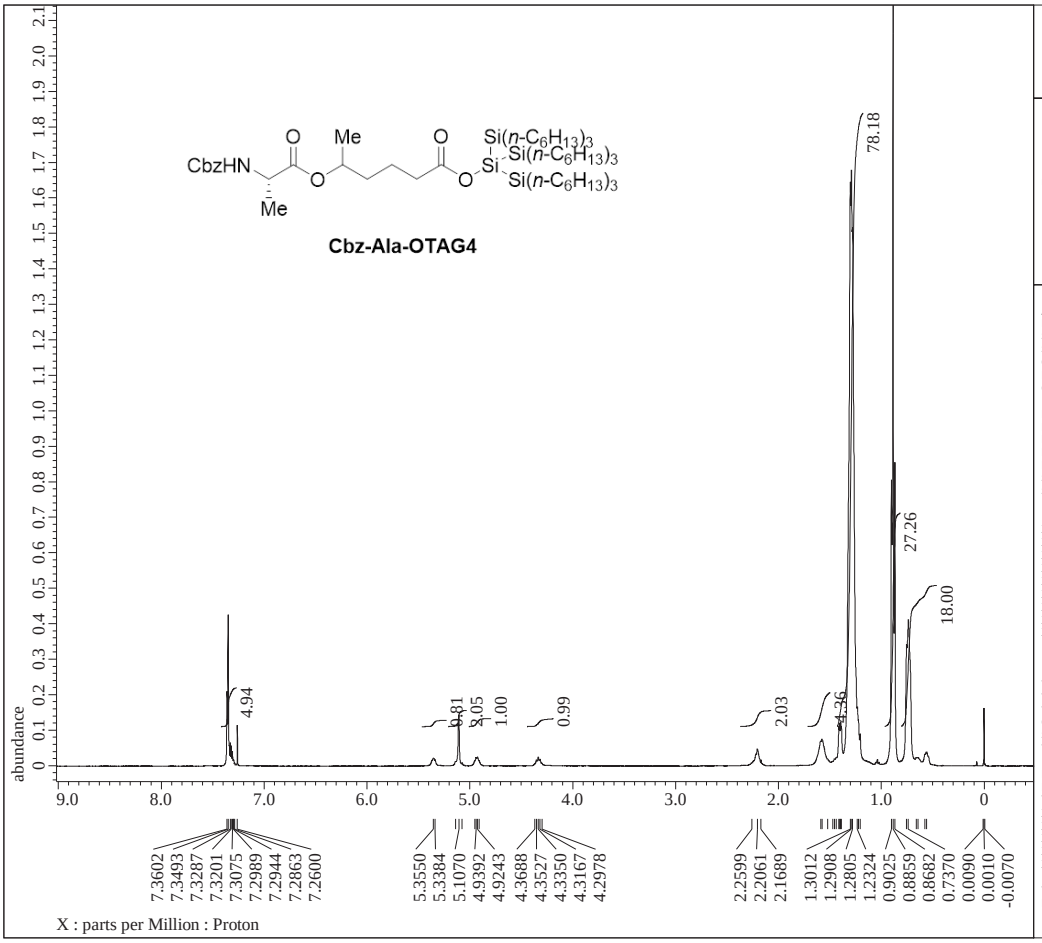
Derived from: wuan-1213_E13C-1-1.jdf
```

```
Filename          = wuan-1213_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1213
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 17:37:10
Revision_Time     = 12-NOV-2021 17:57:33
Current_Time      = 12-NOV-2021 17:59:32

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             = 436
Total_Scans       = 436

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

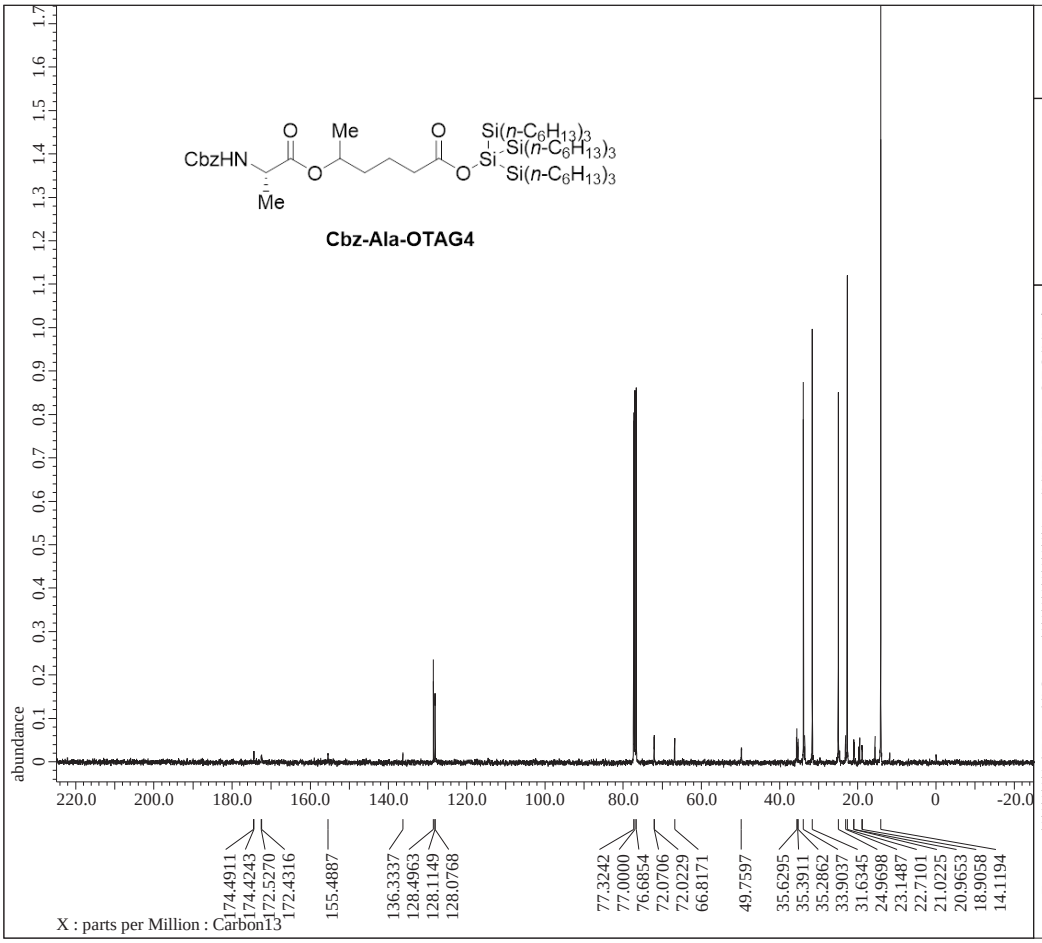
Derived from: wuan-1212_E1H-1-1.jdf
```

```
Filename          = wuan-1212_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1212
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 16:58:07
Revision_Time     = 12-NOV-2021 17:07:06
Current_Time      = 12-NOV-2021 17:08:08

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

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

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



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

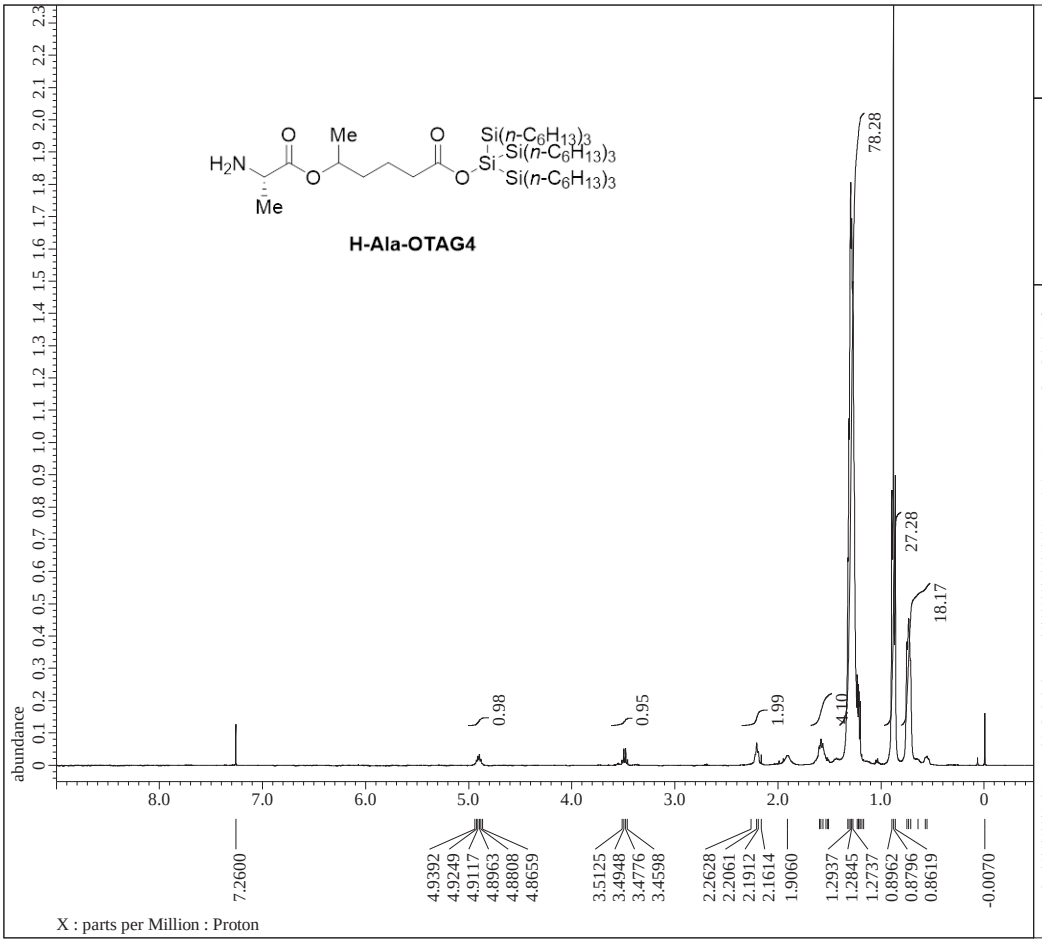
Derived from: wuan-1212_E13C-1-1.jdf
```

```
Filename          = wuan-1212_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1212
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2021 17:02:48
Revision_Time     = 12-NOV-2021 17:27:46
Current_Time      = 12-NOV-2021 17:32:03

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             = 480
Total_Scans       = 480

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



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

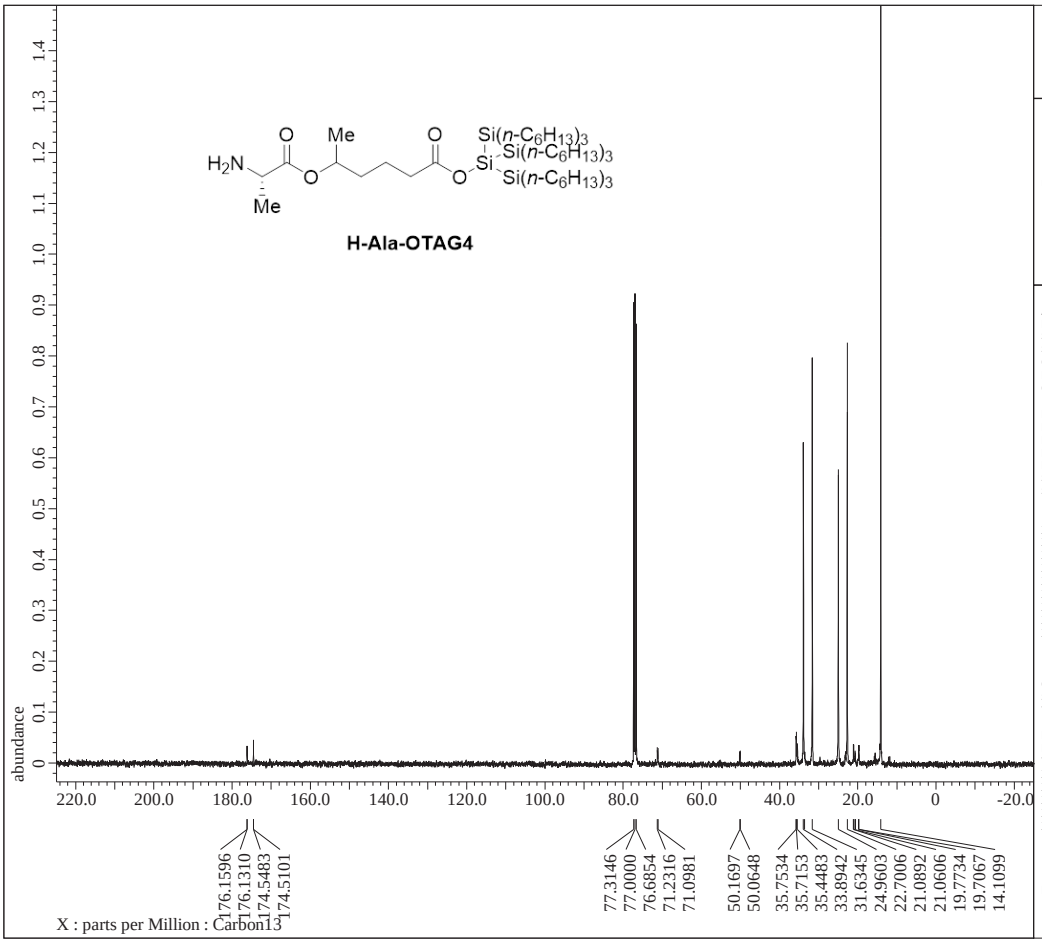
Derived from: wuan-300_E1H-1-1.jdf
```

```
Filename          = wuan-300_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-300
Solvent           = CHLOROFORM-D
Creation_Time     = 28-OCT-2021 10:02:51
Revision_Time     = 28-OCT-2021 10:08:15
Current_Time      = 28-OCT-2021 10:09:08

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          = 24.4[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

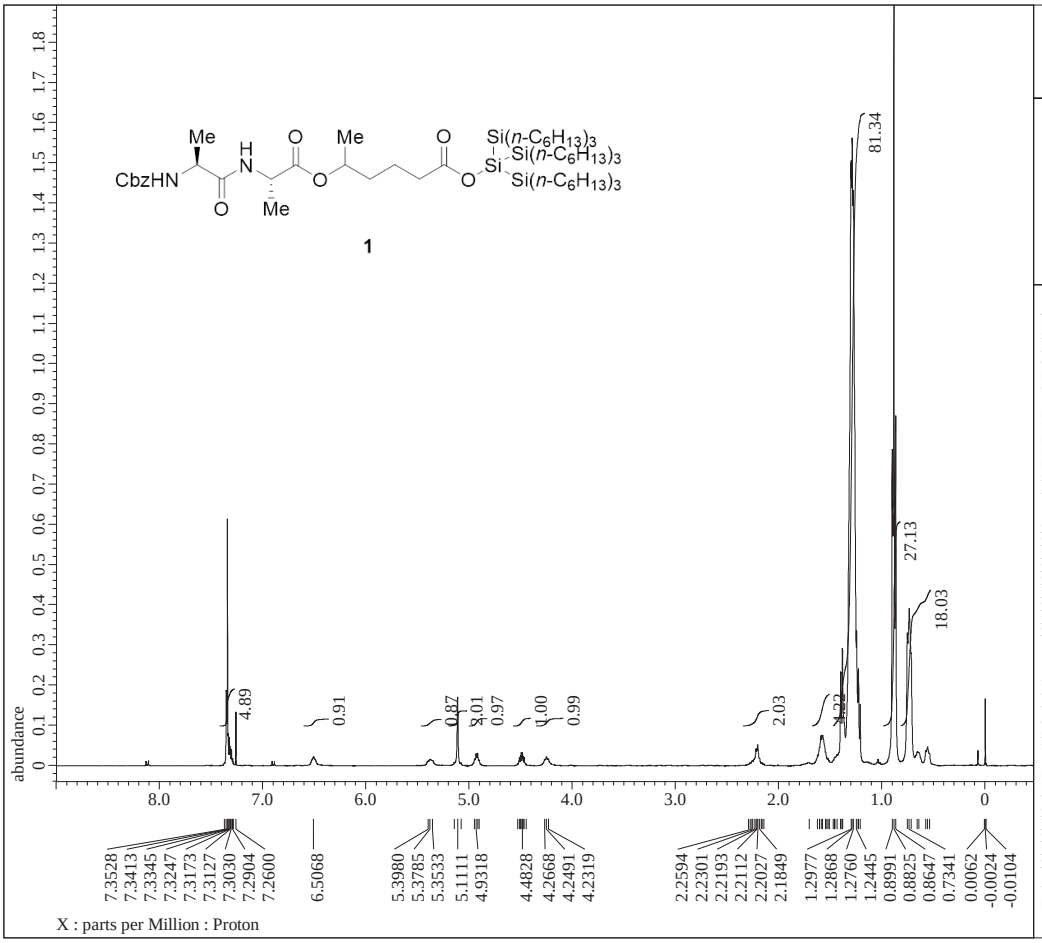
Derived from: wuan-300_E13C-1-1.jdf
```

```
Filename          = wuan-300_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-300
Solvent           = CHLOROFORM-D
Creation_Time     = 28-OCT-2021 10:04:47
Revision_Time     = 28-OCT-2021 10:34:52
Current_Time      = 28-OCT-2021 10:35:51

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

Field_Strength    = 9.389766[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             = 637
Total_Scans       = 637

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

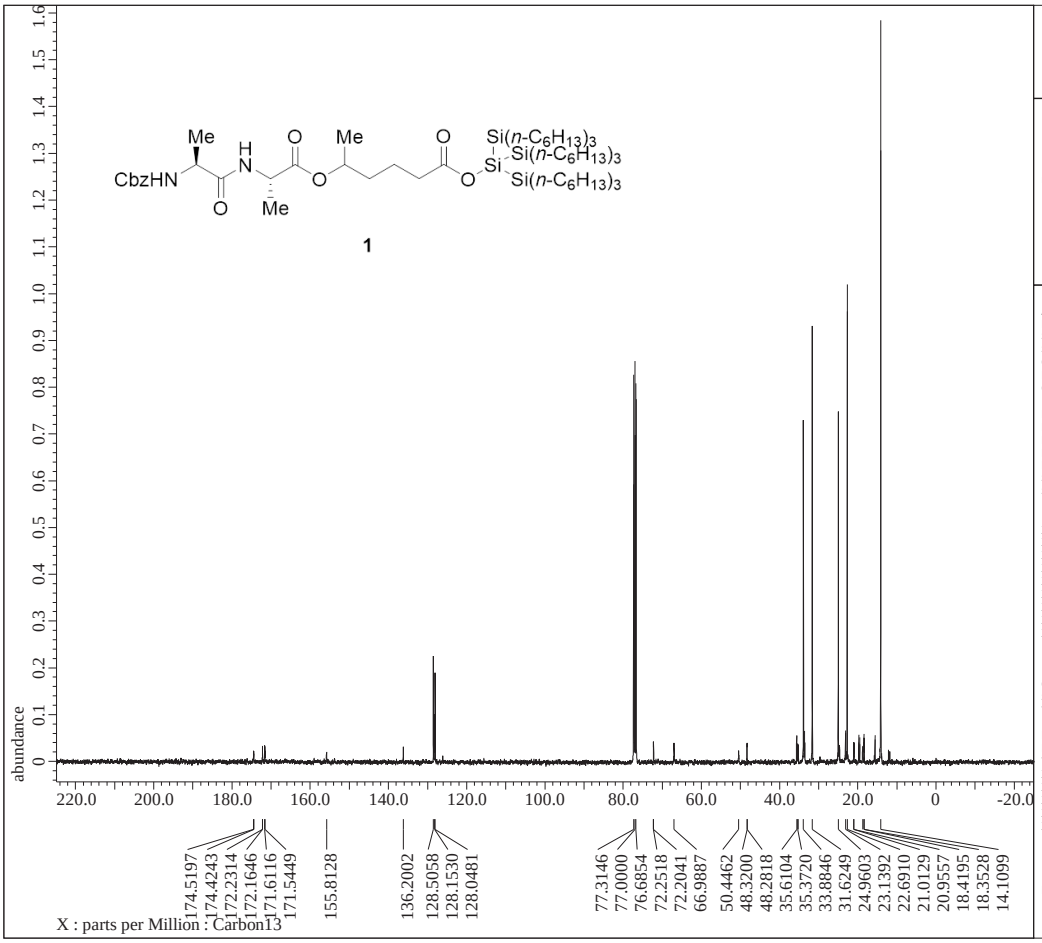
Derived from: wuan-407_E1H-1-1.jdf
```

```
Filename          = wuan-407_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-407
Solvent           = CHLOROFORM-D
Creation_Time     = 27-OCT-2021 18:25:36
Revision_Time     = 27-OCT-2021 18:49:05
Current_Time      = 27-OCT-2021 18:50:31

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        = 20
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

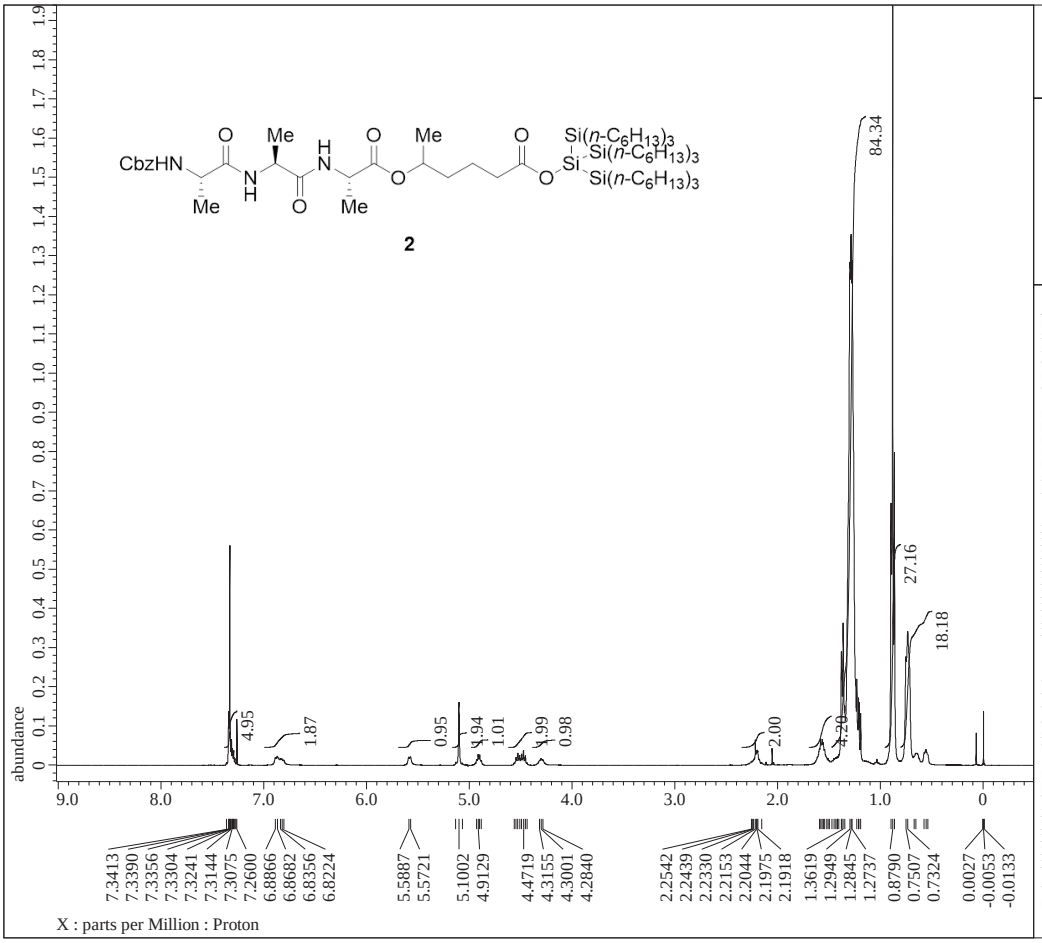
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-407_E13C-1-6.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-407
Solvent           = CHLOROFORM-D
Creation_Time     = 27-OCT-2021 18:27:25
Revision_Time     = 12-NOV-2021 17:30:21
Current_Time      = 12-NOV-2021 17:32:53

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 808
Total_Scans       = 808

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.1[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]
```



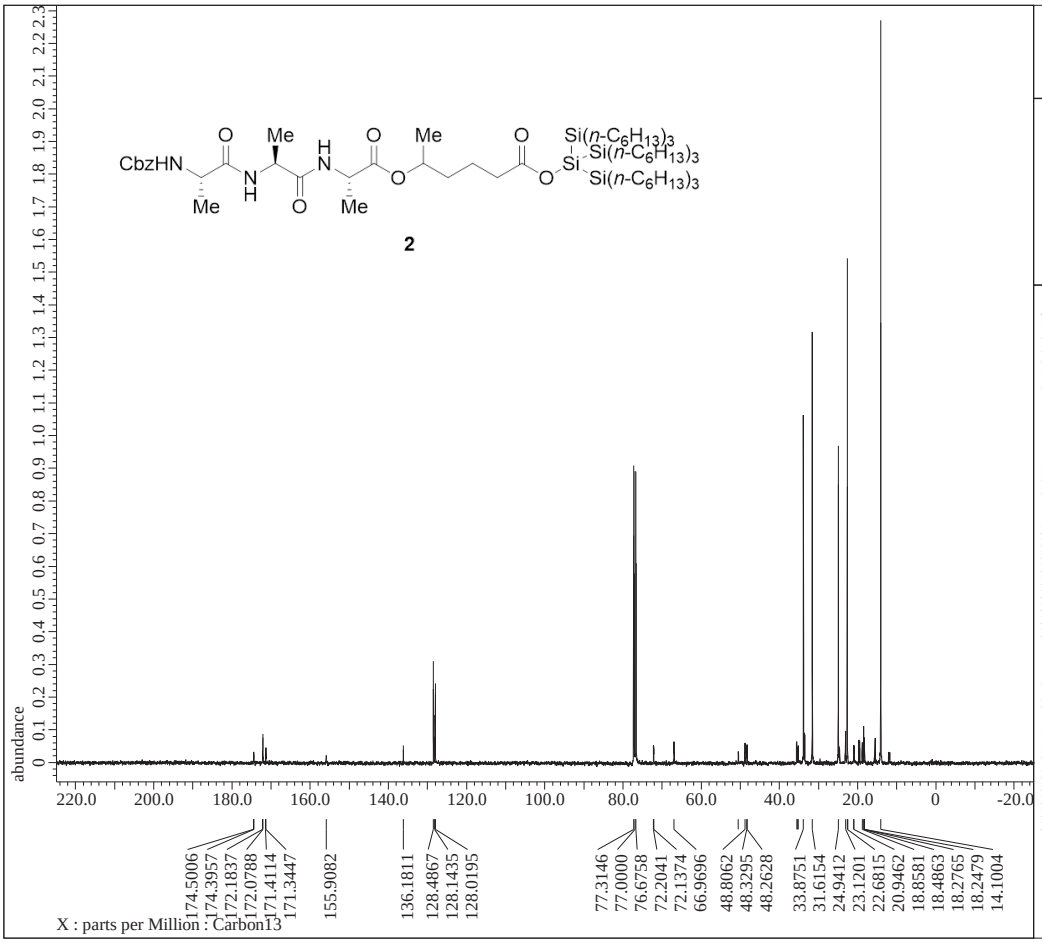

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-430B_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-430B
Solvent           = CHLOROFORM-D
Creation_Time     = 29-OCT-2021 15:03:52
Revision_Time     =  2-NOV-2021 14:12:21
Current_Time      =  2-NOV-2021 14:12:48

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        = 16
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
```



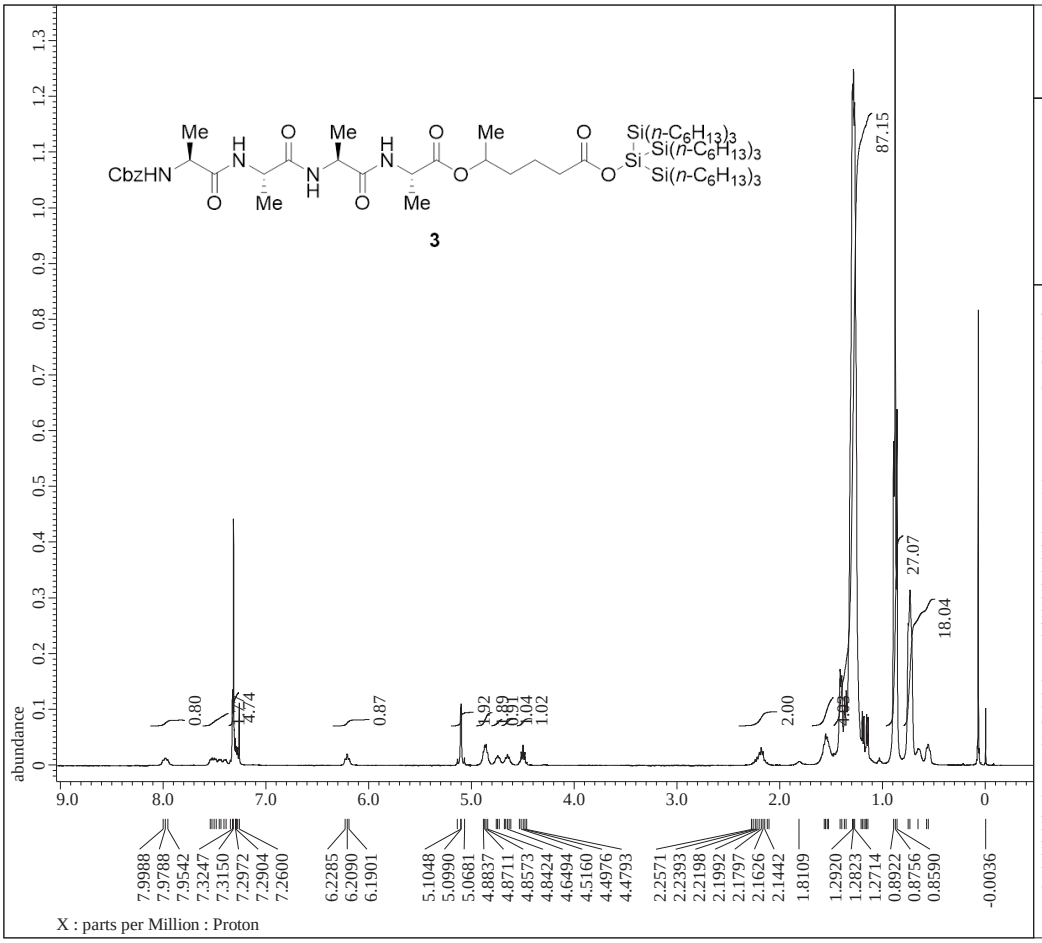

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-430B_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-430B
Solvent           = CHLOROFORM-D
Creation_Time     = 29-OCT-2021 15:07:33
Revision_Time     = 12-NOV-2021 17:46:24
Current_Time      = 12-NOV-2021 17:46:41

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             = 587
Total_Scans       = 587

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]
```



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

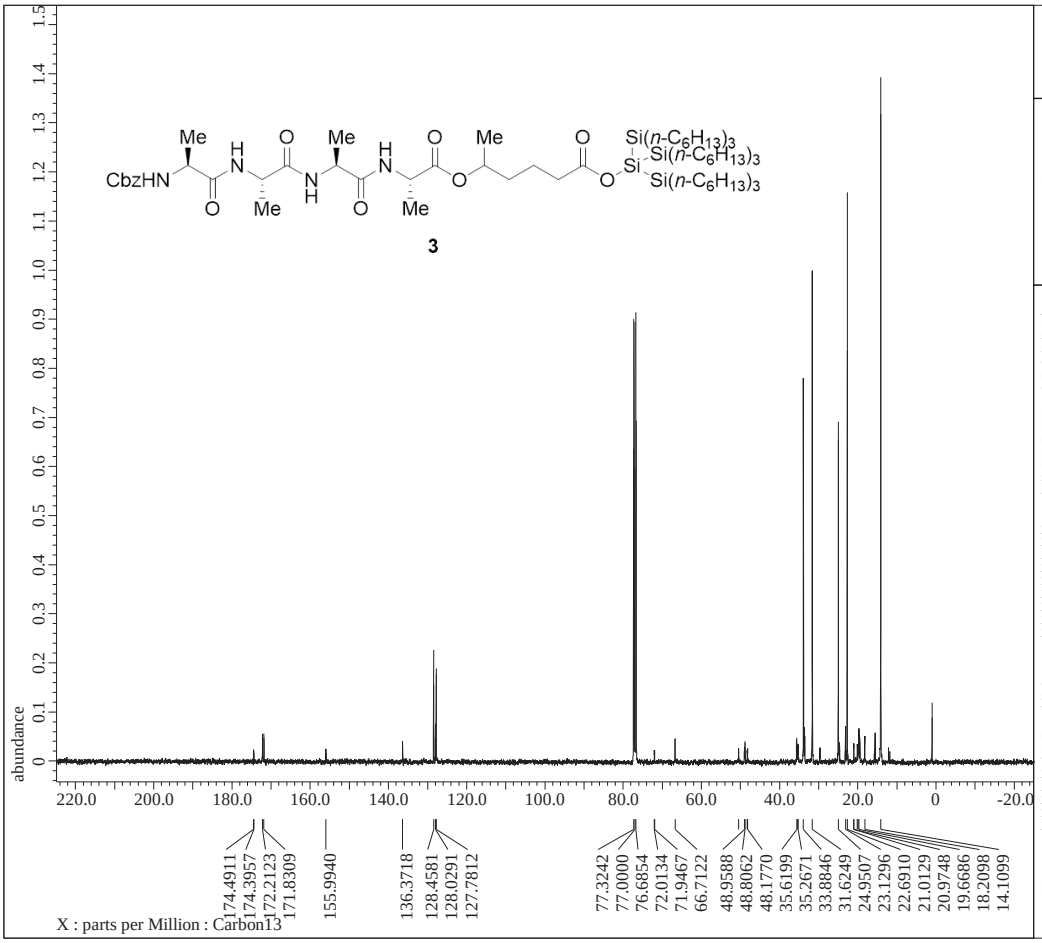
Derived from: wuan-1197_E1H-1-1.jdf
```

```
Filename          = wuan-1197_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1197
Solvent           = CHLOROFORM-D
Creation_Time     =  2-NOV-2021 14:02:33
Revision_Time     =  2-NOV-2021 14:09:06
Current_Time      =  2-NOV-2021 14:10:28

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        = 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
```



JEOL RESONANCE

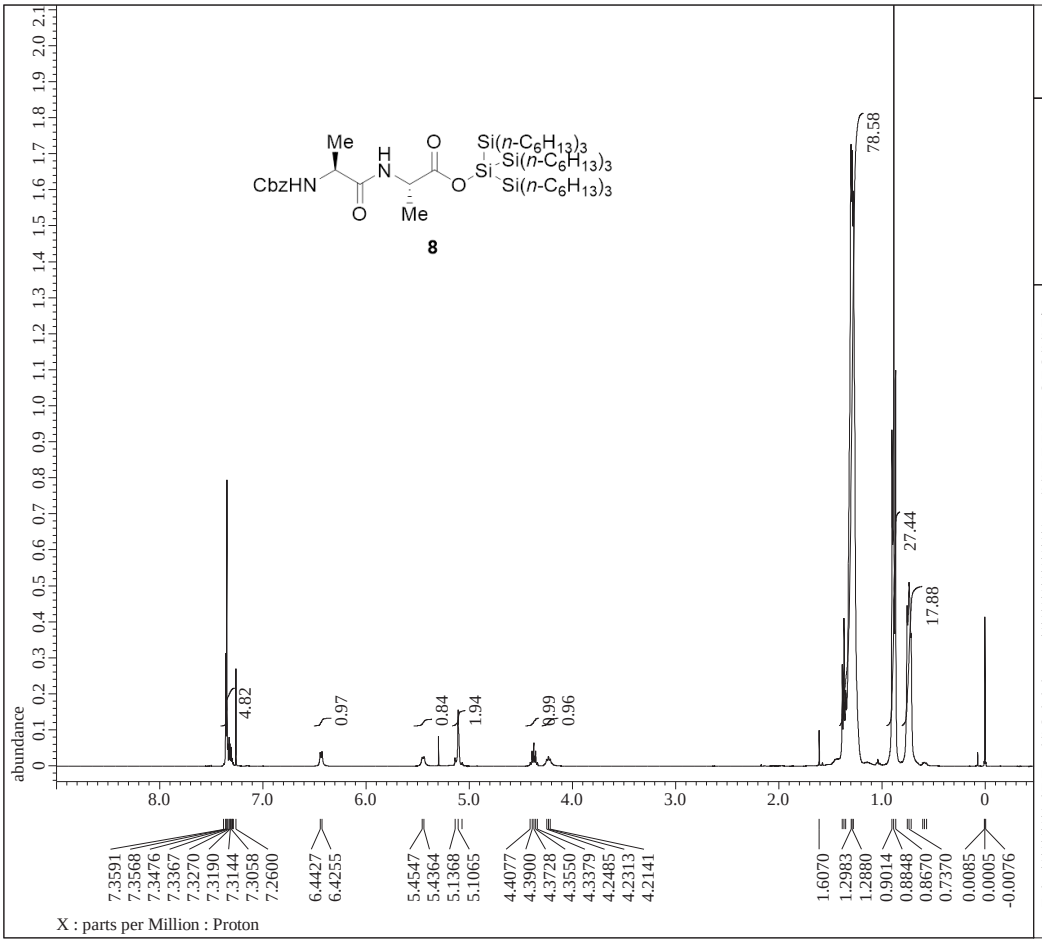
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1197_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1197
Solvent           = CHLOROFORM-D
Creation_Time     =  2-NOV-2021 14:06:25
Revision_Time     =  2-NOV-2021 14:47:50
Current_Time      =  2-NOV-2021 14:48:46

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             = 764
Total_Scans       = 764

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]
```



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

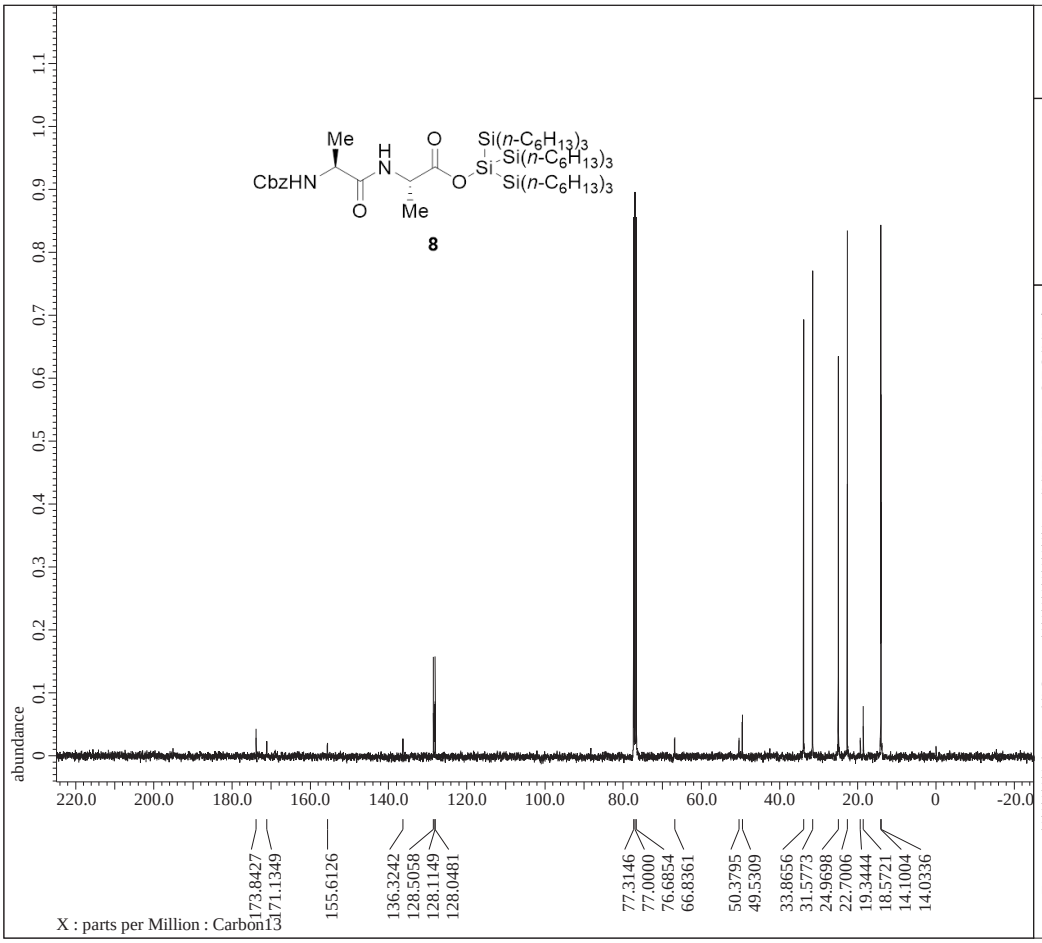
Derived from: wuan-1320_E1H-1-1.jdf
```

```
Filename          = wuan-1320_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1320
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAR-2022 10:10:50
Revision_Time     =  2-MAR-2022 10:14:32
Current_Time      =  2-MAR-2022 10:15:34

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

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

Relaxation_Delay = 5[s]
Recvr_Gain        = 24
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
```



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

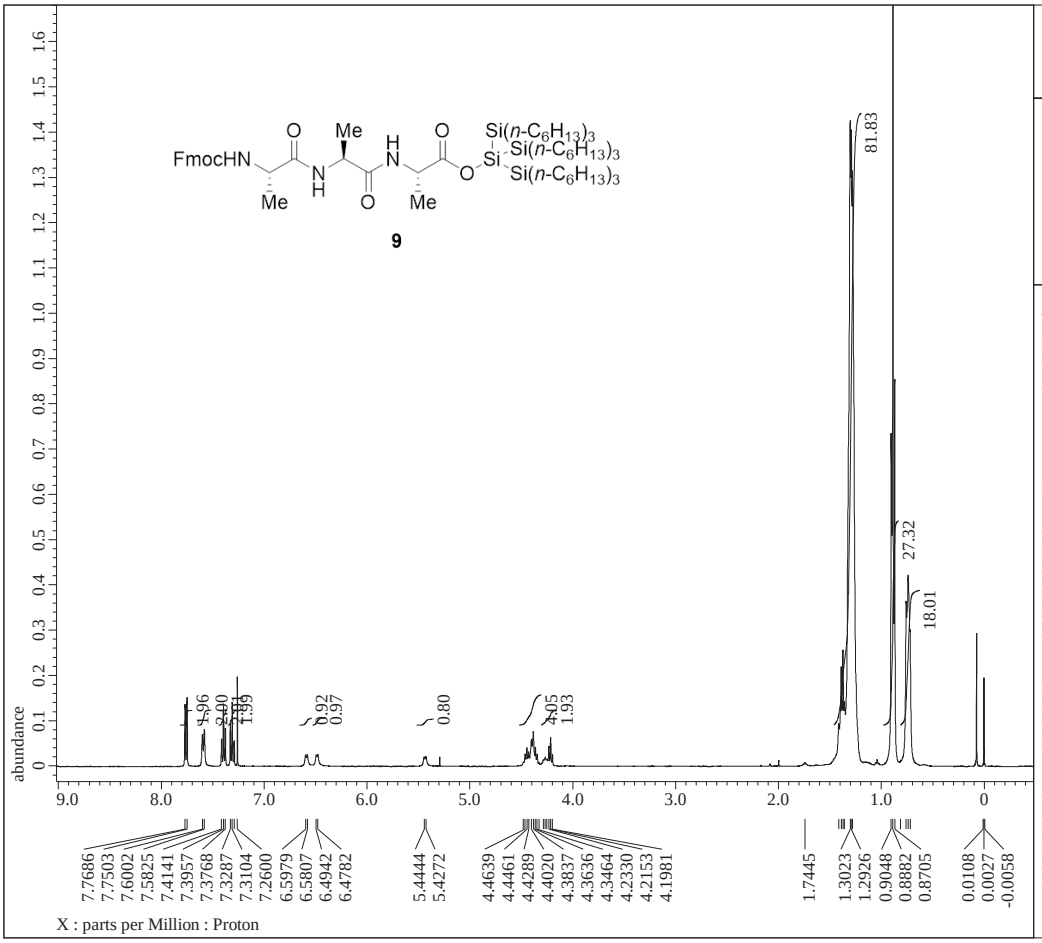
Derived from: wuan-1320_E13C-1-1.jdf
```

```
Filename          = wuan-1320_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1320
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAR-2022 10:14:44
Revision_Time     =  2-MAR-2022 10:34:03
Current_Time      =  2-MAR-2022 10:34:48

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             = 471
Total_Scans       = 471

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.4[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

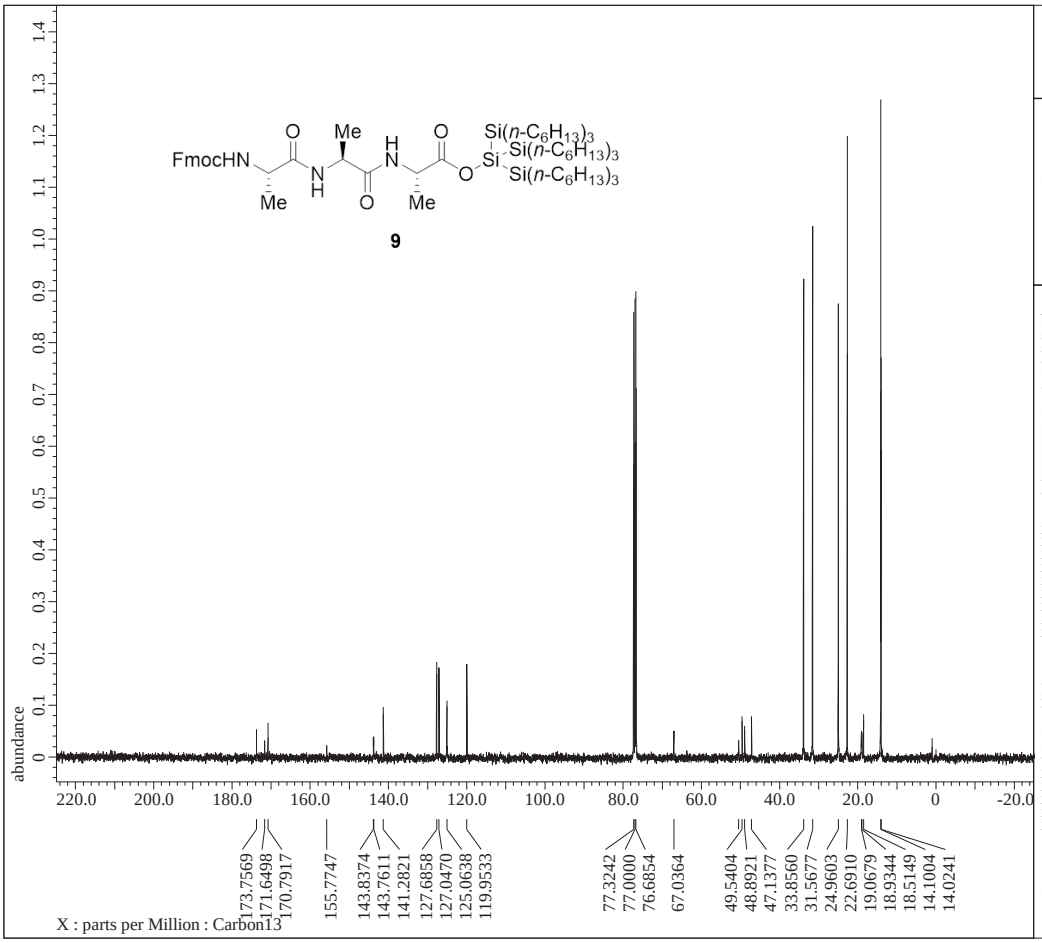
Derived from: wuan-1322_E1H-1-1.jdf

Filename          = wuan-1322_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1322
Solvent           = CHLOROFORM-D
Creation_Time     =  3-MAR-2022 10:43:59
Revision_Time     =  3-MAR-2022 10:46:14
Current_Time      =  3-MAR-2022 10:46:44

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 20
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
```




```
---- 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

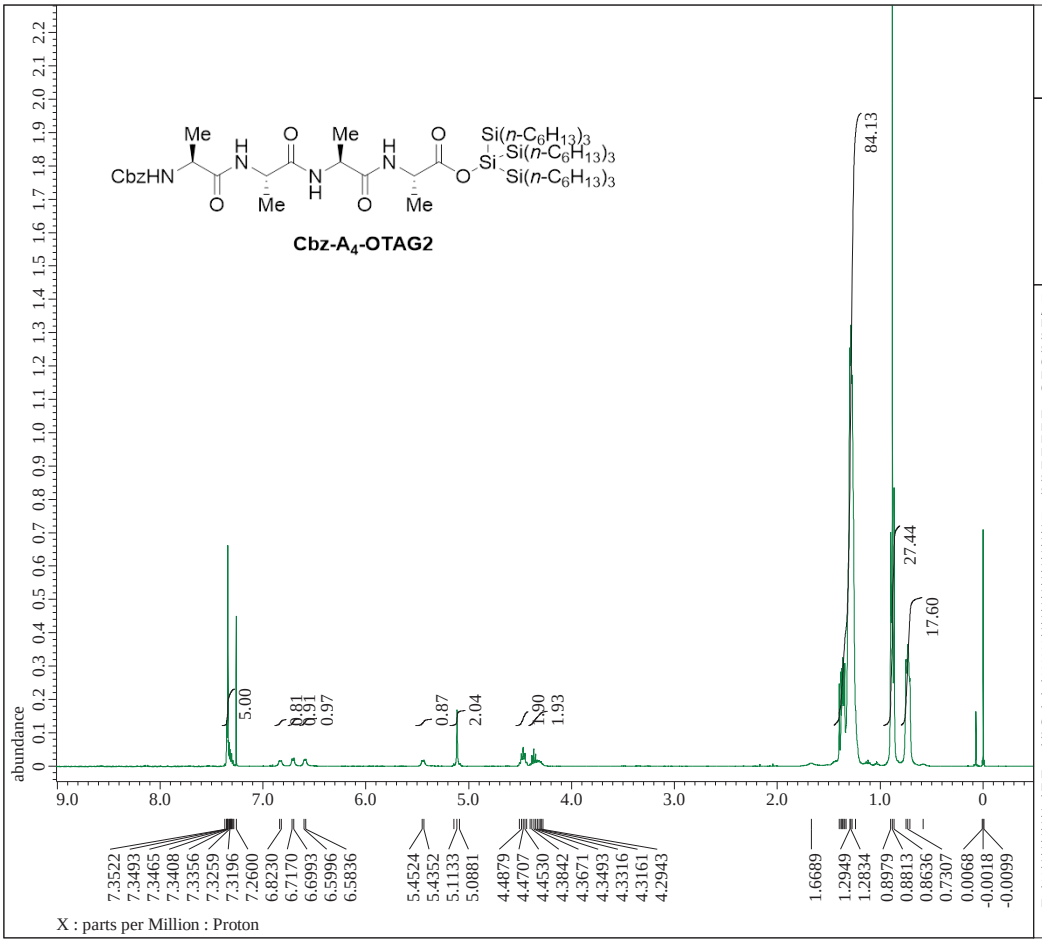
Derived from: wuan-1322_E13C-1-1.jdf

Filename          = wuan-1322_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1322
Solvent           = CHLOROFORM-D
Creation_Time     =  3-MAR-2022 10:48:31
Revision_Time     =  3-MAR-2022 10:59:54
Current_Time      =  3-MAR-2022 11:00:33

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 301
Total_Scans       = 301

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

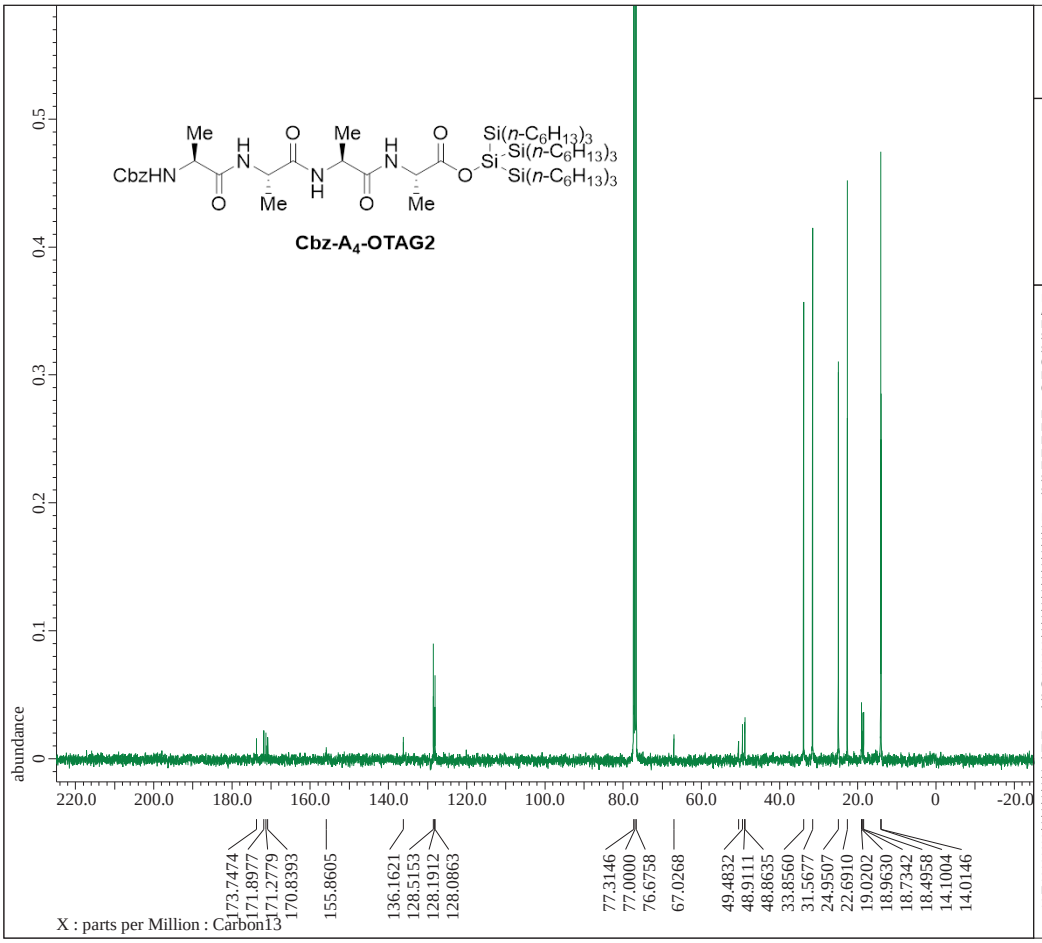
Derived from: wuan-1698-1_E1H-2-1.jdf

Filename          = wuan-1698-1_E1H-2-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1698-1
Solvent           = CHLOROFORM-D
Creation_Time     =  1-APR-2023 14:04:04
Revision_Time     =  1-APR-2023 14:03:27
Current_Time      =  1-APR-2023 14:04:08

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

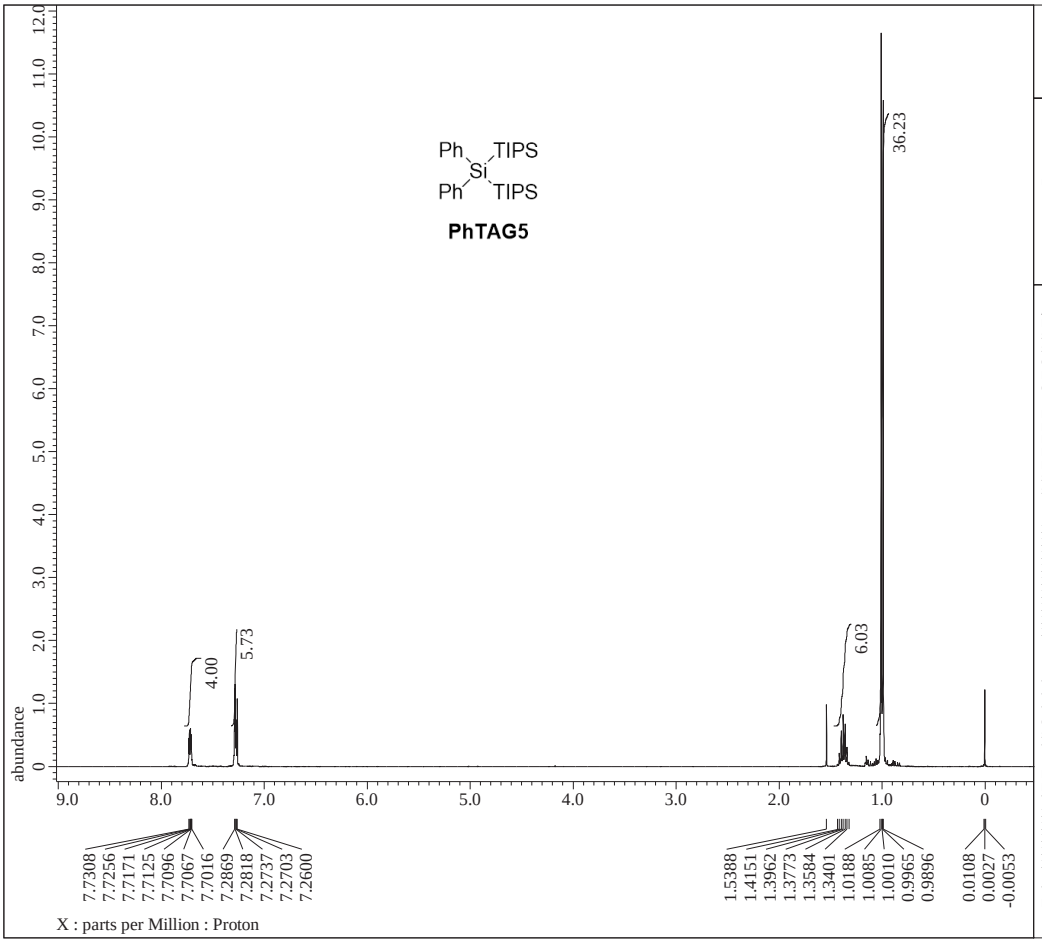
Derived from: wuan-1698-1_E13C-1-1.jdf

Filename          = wuan-1698-1_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1698-1
Solvent           = CHLOROFORM-D
Creation_Time     =  1-APR-2023 12:34:52
Revision_Time     =  1-APR-2023 13:18:58
Current_Time      =  1-APR-2023 13:19:33

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 989
Total_Scans       = 989

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



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

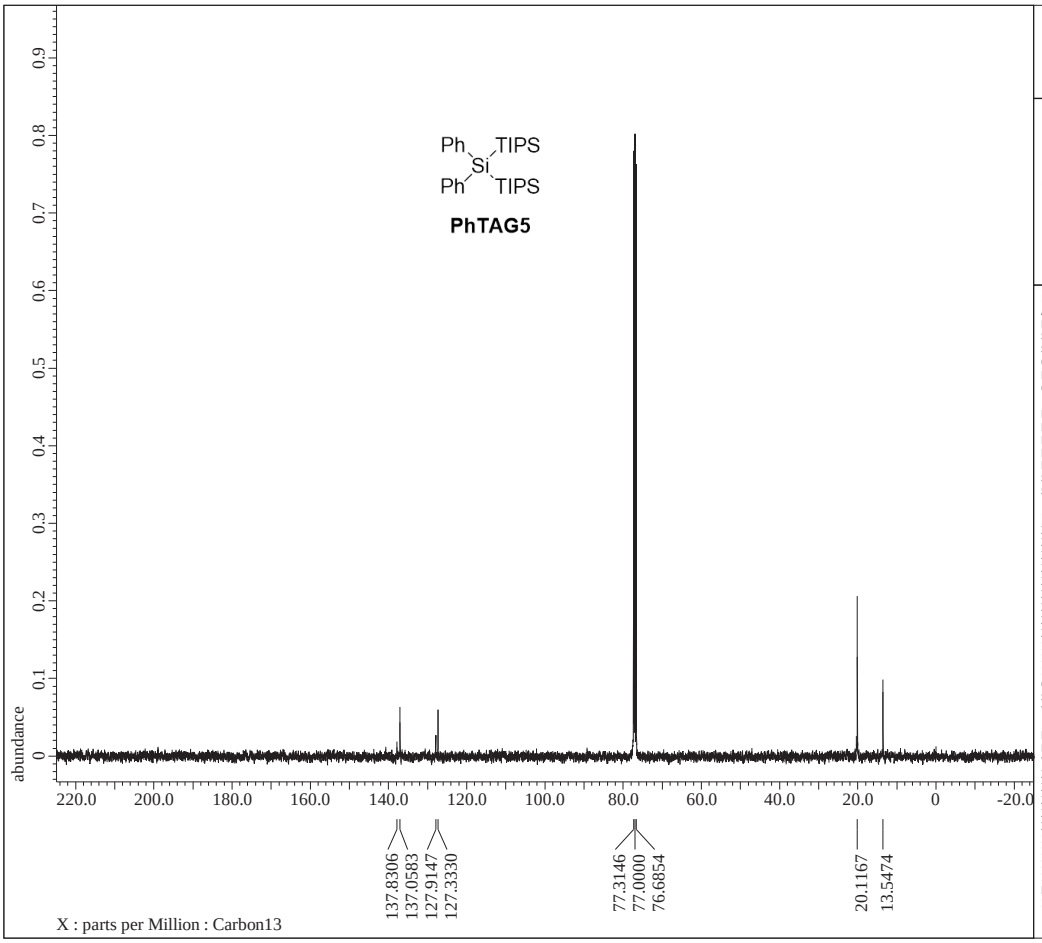
Derived from: wuan-105-1_E1H-1-1.jdf
```

```
Filename          = wuan-105-1_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-105-1
Solvent           = CHLOROFORM-D
Creation_Time     =  8-JUN-2019 13:14:23
Revision_Time     = 24-FEB-2022 13:29:00
Current_Time      = 24-FEB-2022 13:29:46

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
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
```



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

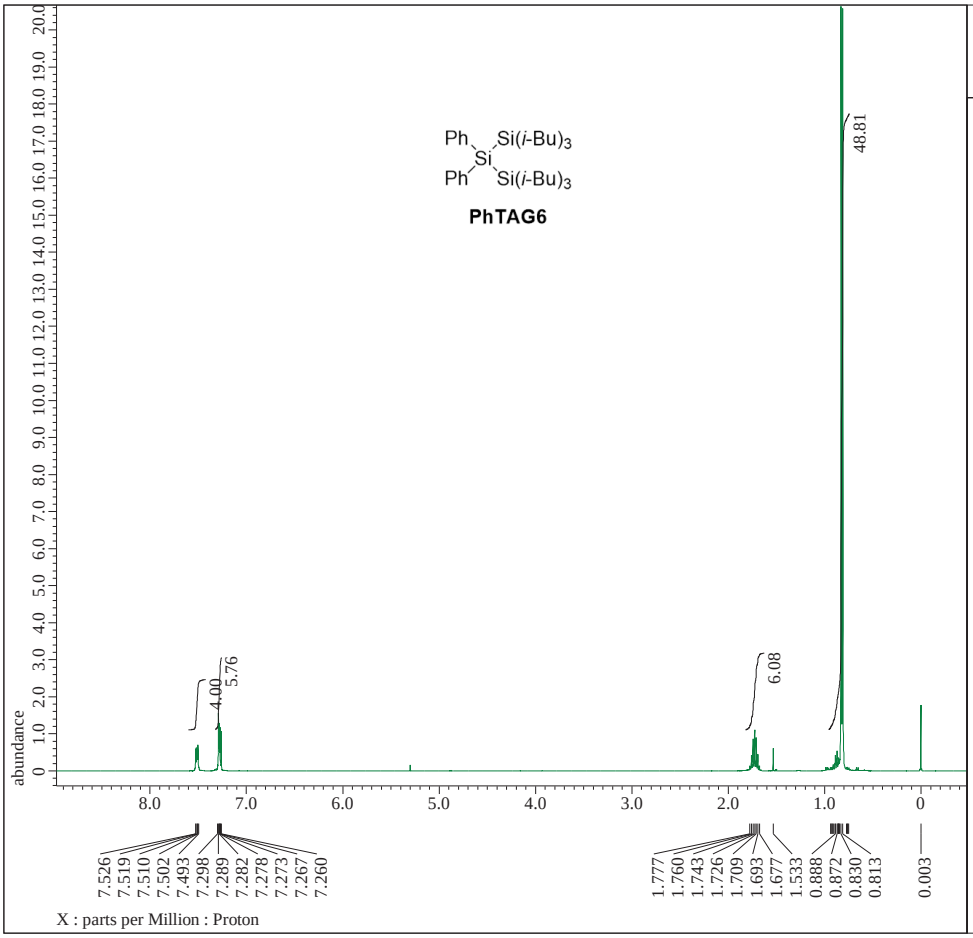
Derived from: wuan-1244-2_E13C-1-1.jdf
```

```
Filename          = wuan-1244-2_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1244-2
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2021 13:00:39
Revision_Time     = 22-DEC-2021 13:19:11
Current_Time      = 22-DEC-2021 13:19:32

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             = 435.0
Total_Scans       = 435.0

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



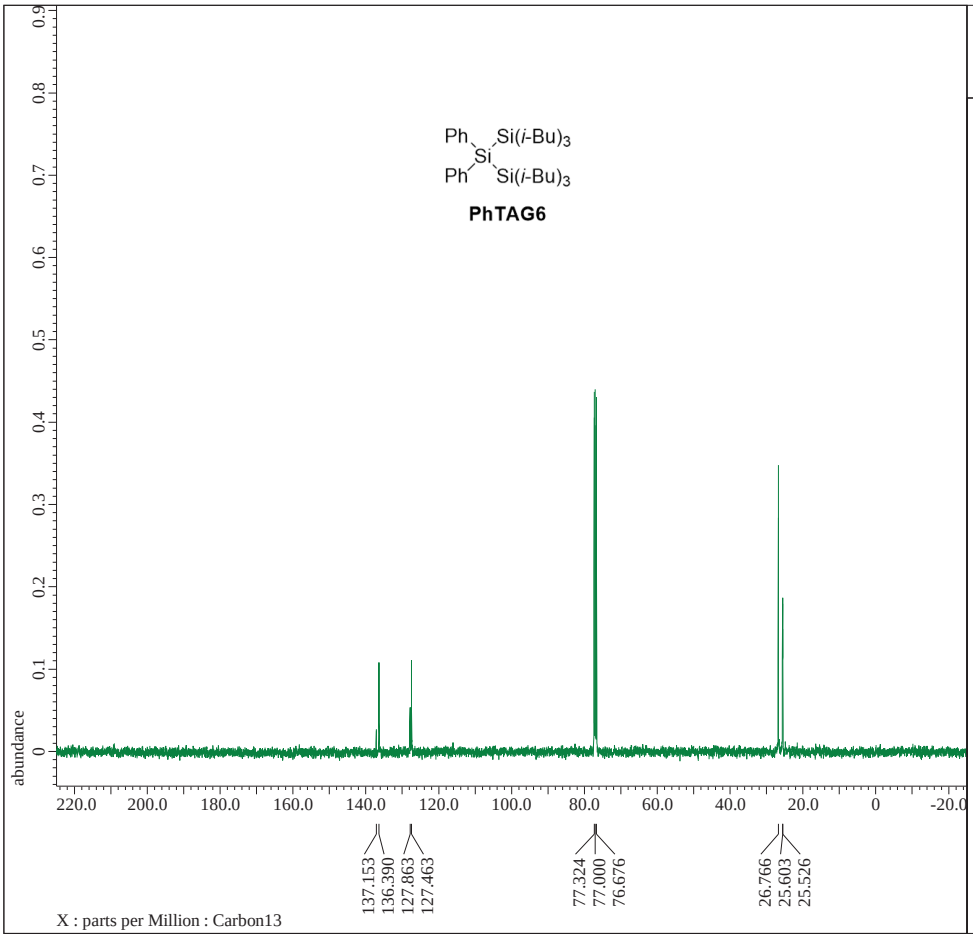
JEOL RESONANCE

```
Filename          = 20220117_wuan-1264_E1H-1
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1264
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2022 14:24:55
Revision_Time     = 17-JAN-2022 14:28:47
Current_Time      = 17-JAN-2022 14:29:28

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

Field_Strength    = 9.20197068[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        = 40
Temp_Get          = 23.7[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
Initial_Wait      = 1[s]
Repetition_Time   = 8.34495744[s]
```



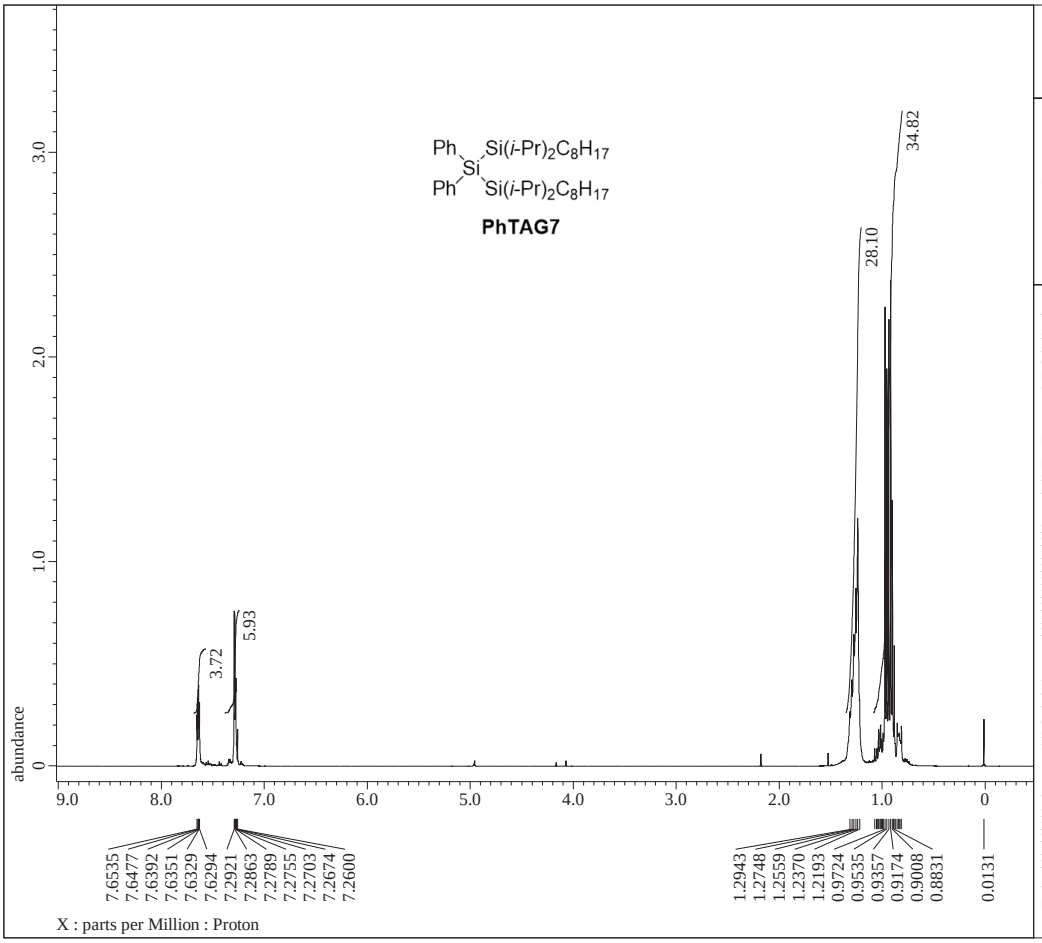
JEOL RESONANCE

```
Filename          = 20220117_wuan-1264_E13C-1
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1264
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2022 14:28:12
Revision_Time     = 17-JAN-2022 14:51:11
Current_Time      = 17-JAN-2022 14:51:40

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

Field_Strength    = 9.20197068[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             = 500
Total_Scans       = 500

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24[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

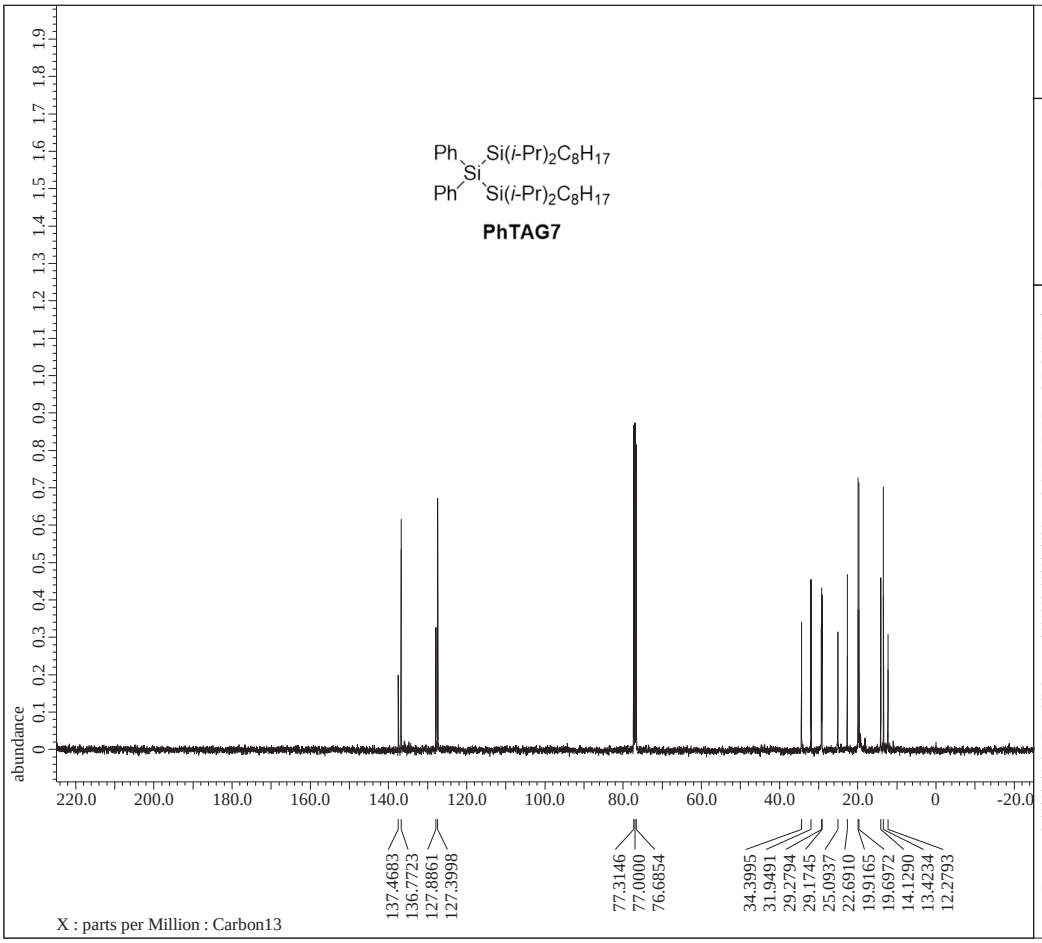
Derived from: wuan-1275_E1H-1-1.jdf
```

```
Filename          = wuan-1275_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1275
Solvent           = CHLOROFORM-D
Creation_Time     = 28-FEB-2022 14:38:55
Revision_Time     = 28-FEB-2022 14:56:38
Current_Time      = 28-FEB-2022 14:57:23

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        = 20
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

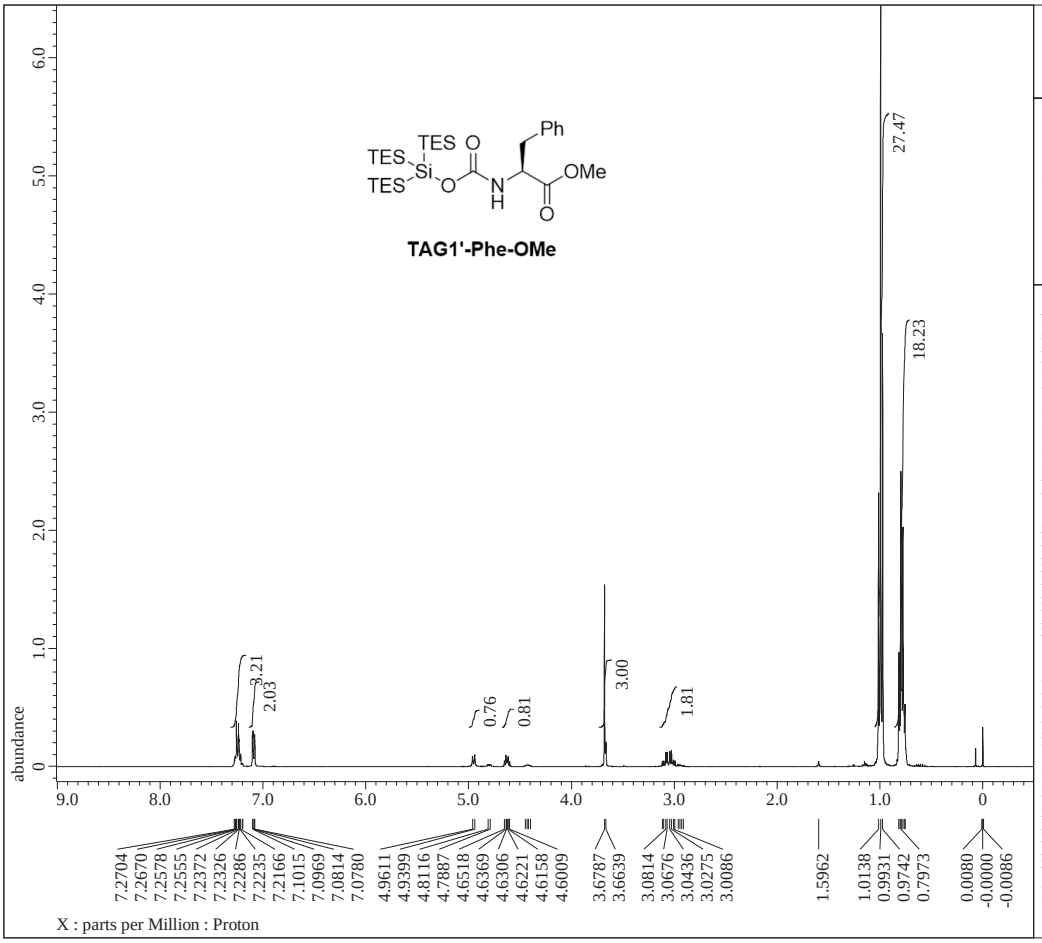
Derived from: wuan-1275_E13C-1-1.jdf
```

```
Filename          = wuan-1275_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1275
Solvent           = CHLOROFORM-D
Creation_Time     = 28-FEB-2022 14:42:49
Revision_Time     = 28-FEB-2022 14:47:44
Current_Time      = 28-FEB-2022 14:48:09

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 184
Total_Scans       = 184

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



JEOL RESONANCE

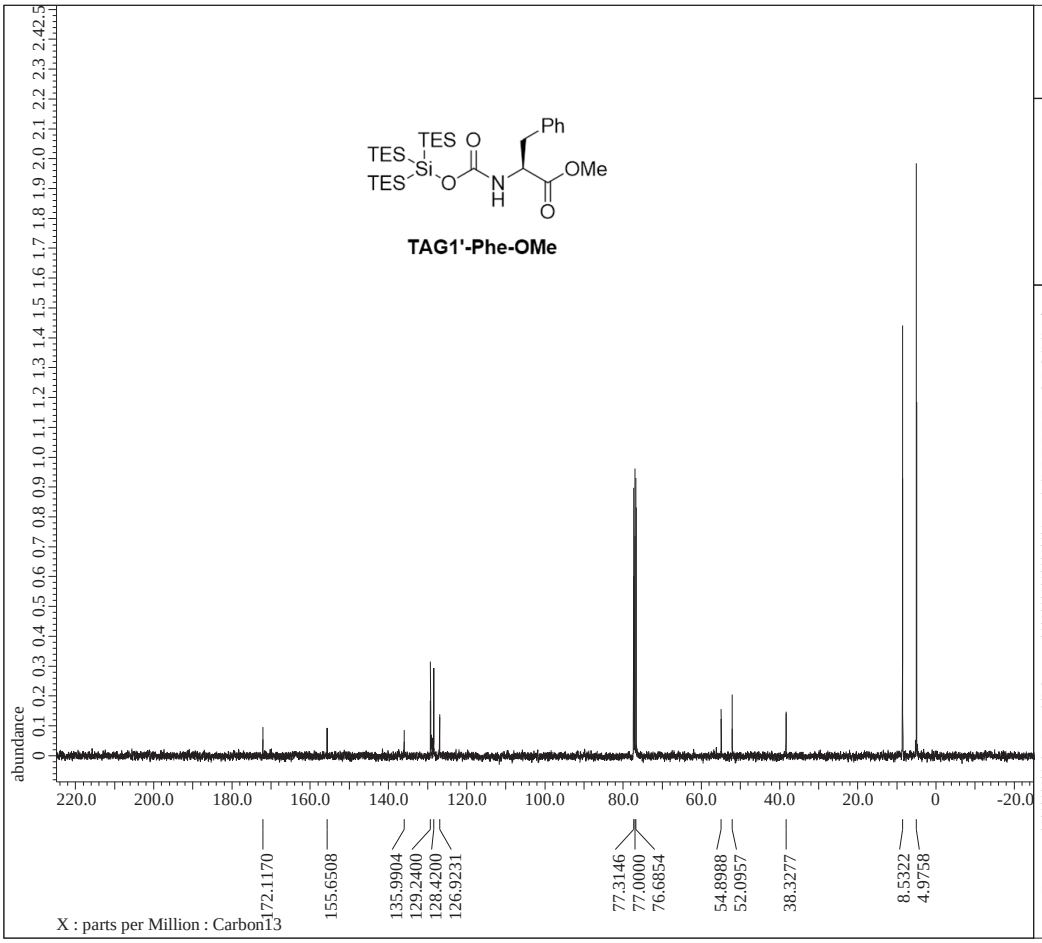
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1304_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1304
Solvent           = CHLOROFORM-D
Creation_Time     = 16-FEB-2022 16:32:01
Revision_Time     = 24-FEB-2022 13:35:47
Current_Time      = 24-FEB-2022 13:36:44

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 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
```



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

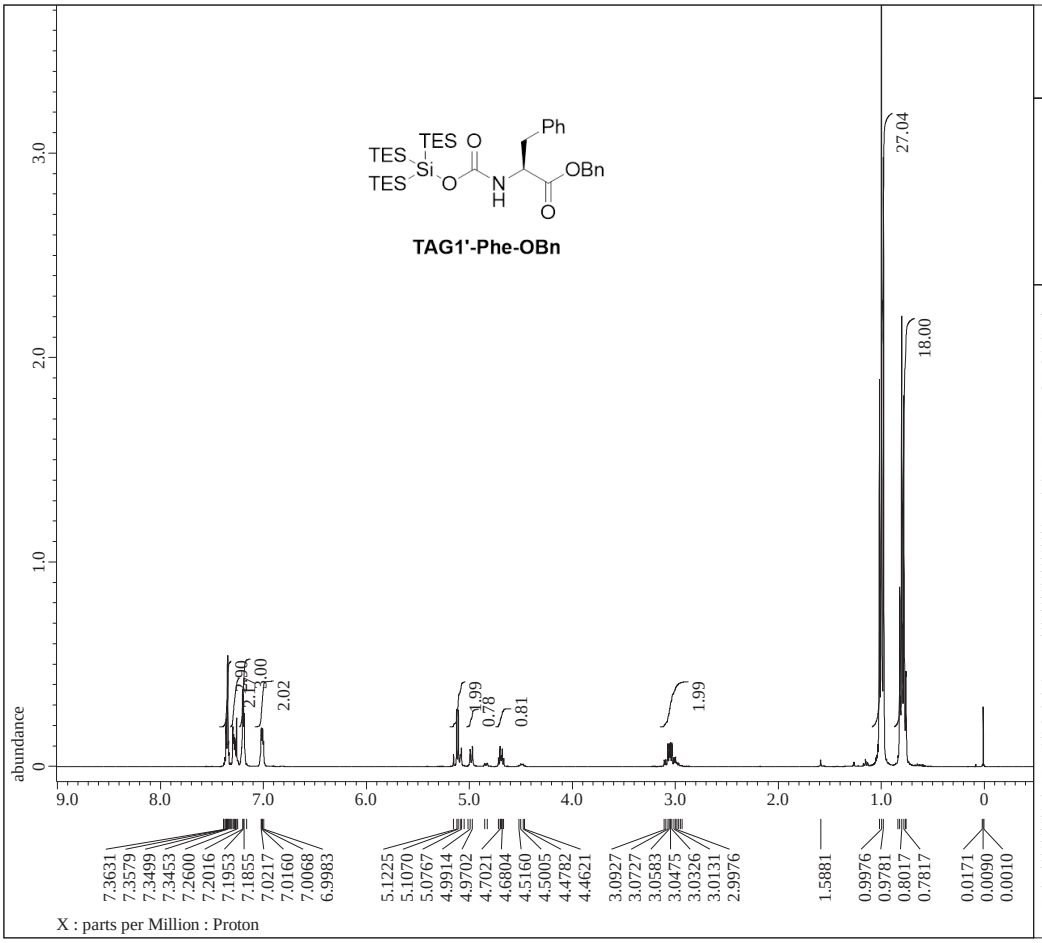
Derived from: wuan-1304_E13C-1-1.jdf
```

```
Filename          = wuan-1304_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1304
Solvent           = CHLOROFORM-D
Creation_Time     = 16-FEB-2022 16:36:36
Revision_Time     = 16-FEB-2022 16:37:36
Current_Time      = 16-FEB-2022 16:38:03

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             = 100
Total_Scans       = 100

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

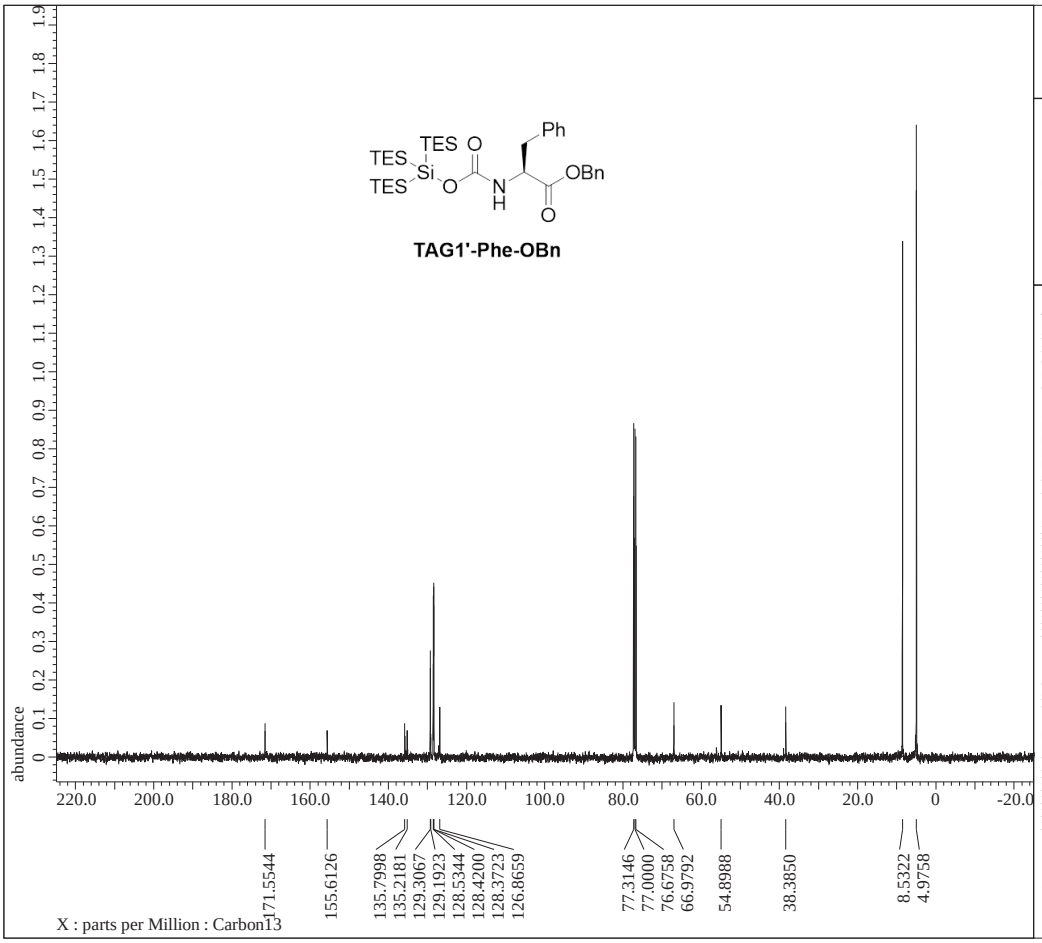
Derived from: wuan-1309_E1H-1-1.jdf
```

```
Filename          = wuan-1309_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1309
Solvent           = CHLOROFORM-D
Creation_Time     = 28-FEB-2022 12:33:56
Revision_Time     = 28-FEB-2022 12:34:50
Current_Time      = 28-FEB-2022 12:35:47

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



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

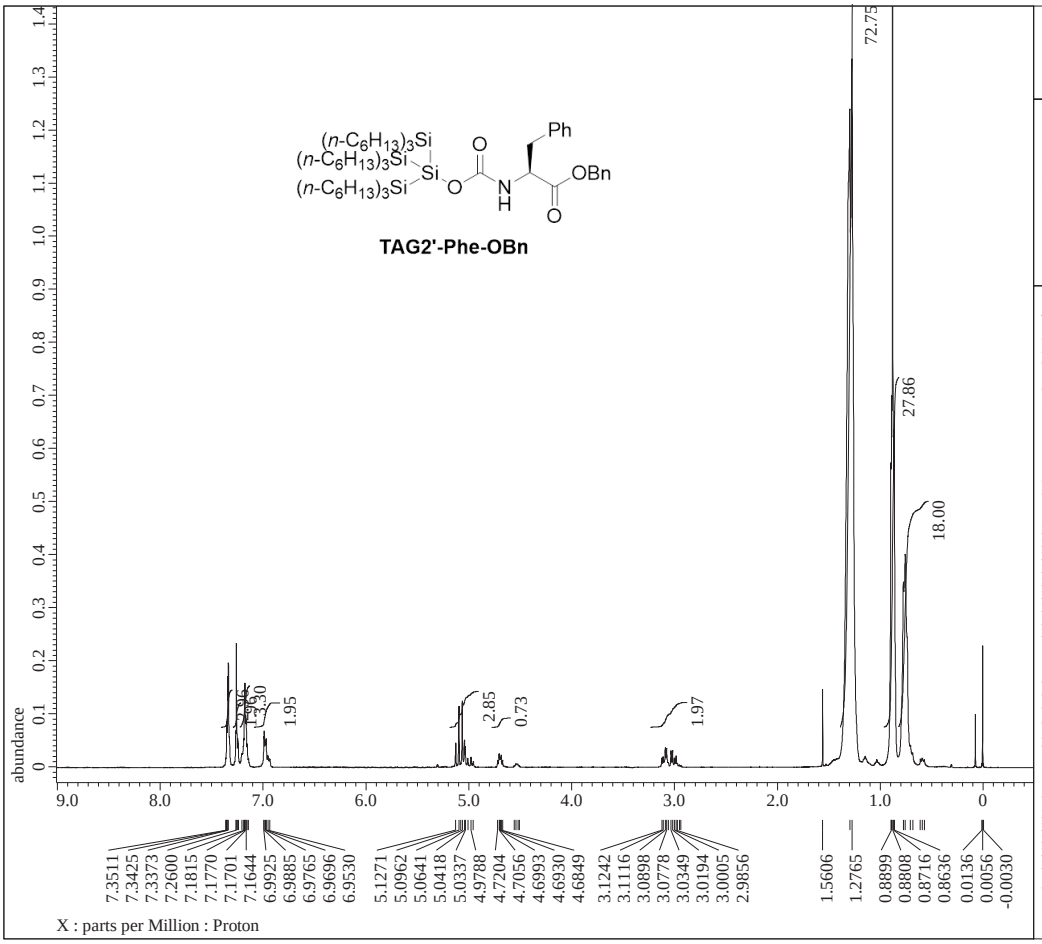
Derived from: wuan-1309_E13C-1-1.jdf
```

```
Filename          = wuan-1309_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1309
Solvent           = CHLOROFORM-D
Creation_Time     = 28-FEB-2022 12:38:07
Revision_Time     = 28-FEB-2022 12:42:50
Current_Time      = 28-FEB-2022 12:43:26

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             = 157
Total_Scans       = 157

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

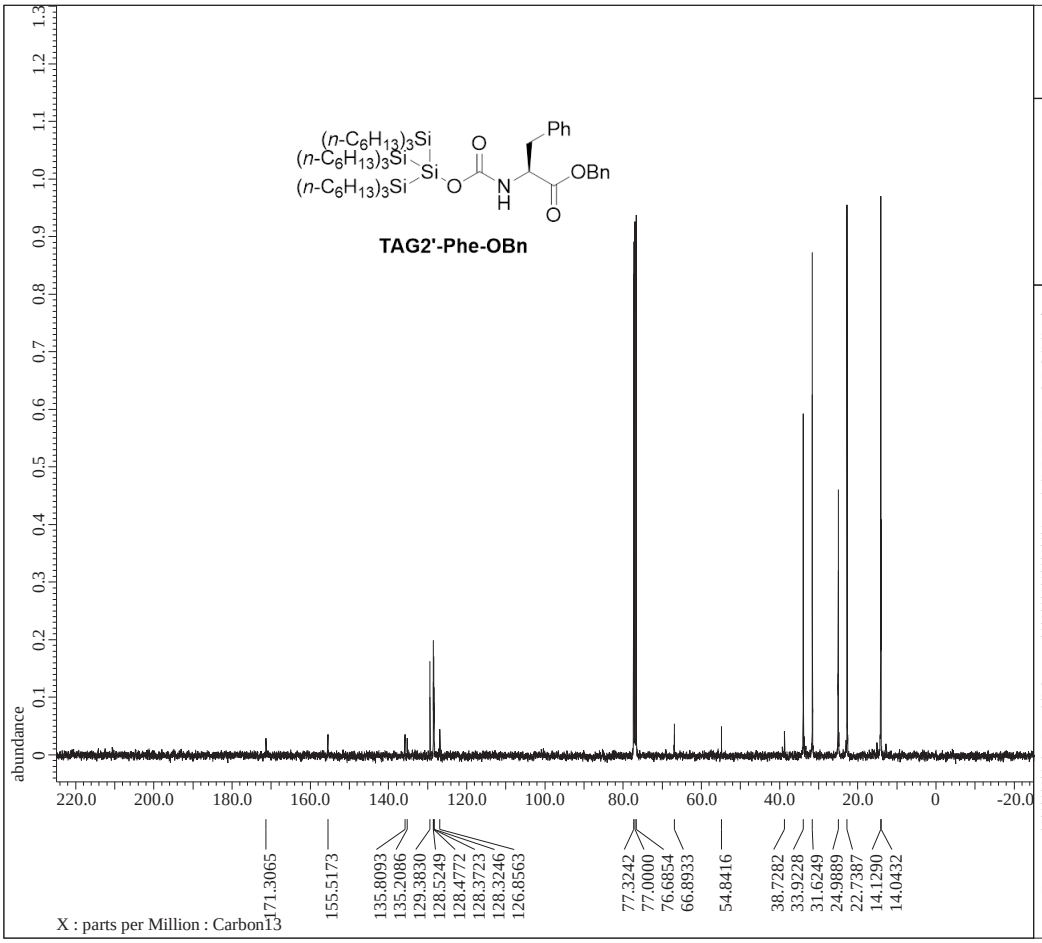
Derived from: wuan-1316_E1H-1-1.jdf

Filename          = wuan-1316_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1316
Solvent           = CHLOROFORM-D
Creation_Time     = 25-FEB-2022 12:33:35
Revision_Time     = 25-FEB-2022 12:49:06
Current_Time      = 25-FEB-2022 12:49:18

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

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

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




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

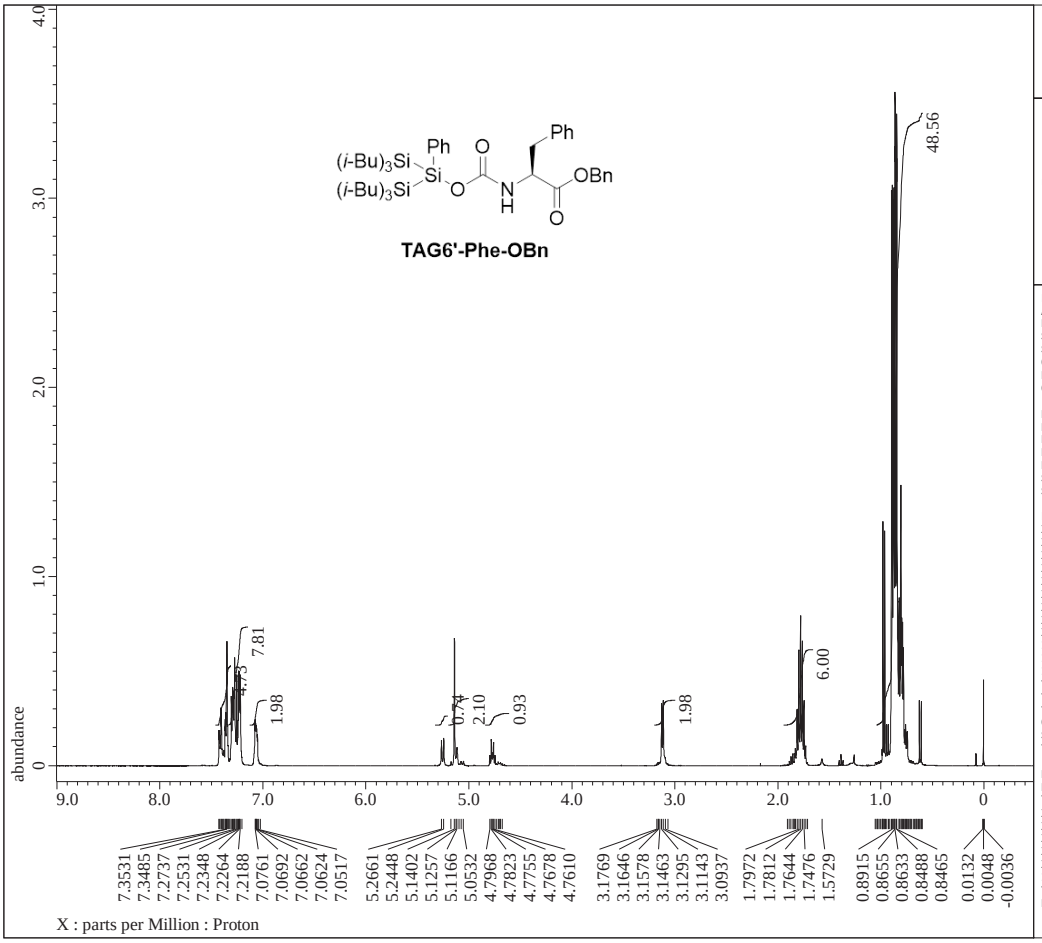
Derived from: wuan-1316_E13C-1-1.jdf

Filename          = wuan-1316_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1316
Solvent           = CHLOROFORM-D
Creation_Time     = 25-FEB-2022 12:38:12
Revision_Time     = 25-FEB-2022 12:55:10
Current_Time      = 25-FEB-2022 12:55:40

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 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             = 365
Total_Scans       = 365

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




```
---- 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

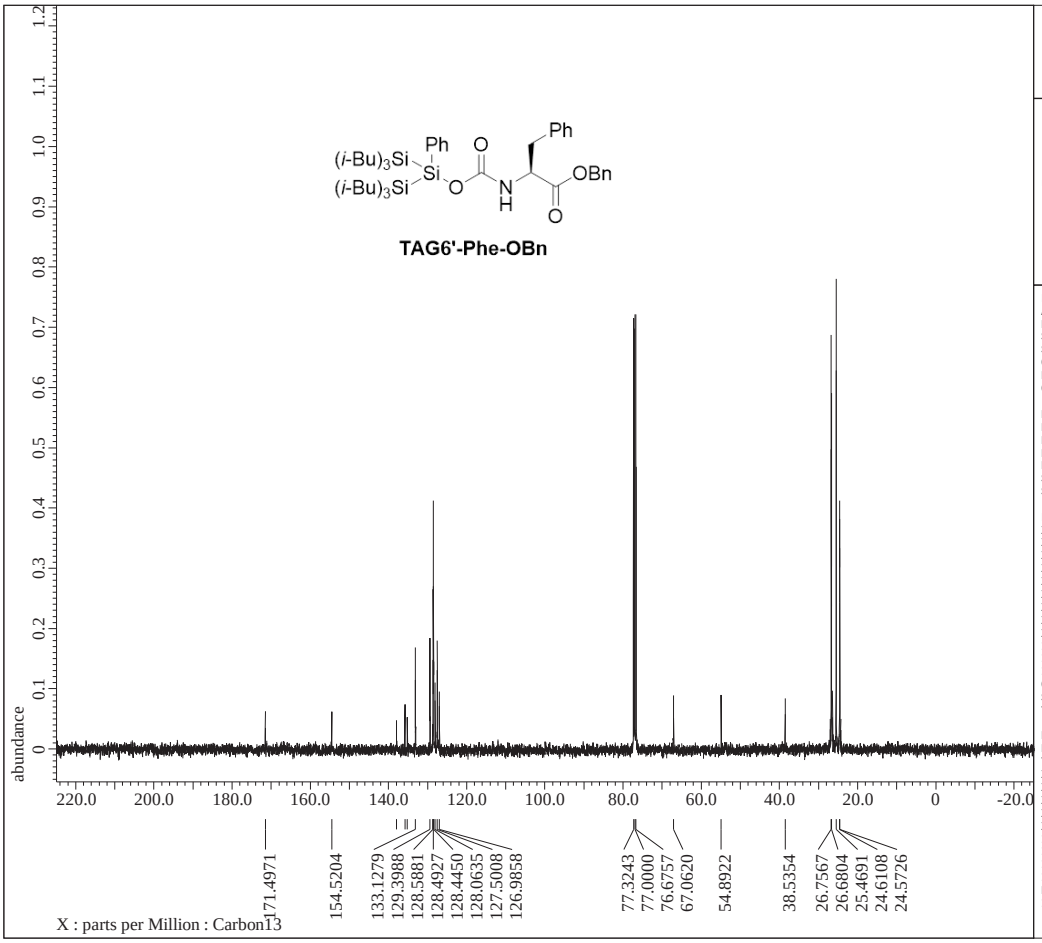
Derived from: wuan-1313_E1H-1-1.jdf

Filename          = wuan-1313_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1313
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2022 17:18:21
Revision_Time     = 22-FEB-2022 17:37:41
Current_Time      = 22-FEB-2022 17:38:07

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

Field_Strength    = 9.20197068[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        = 30
Temp_Get          = 23.9[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

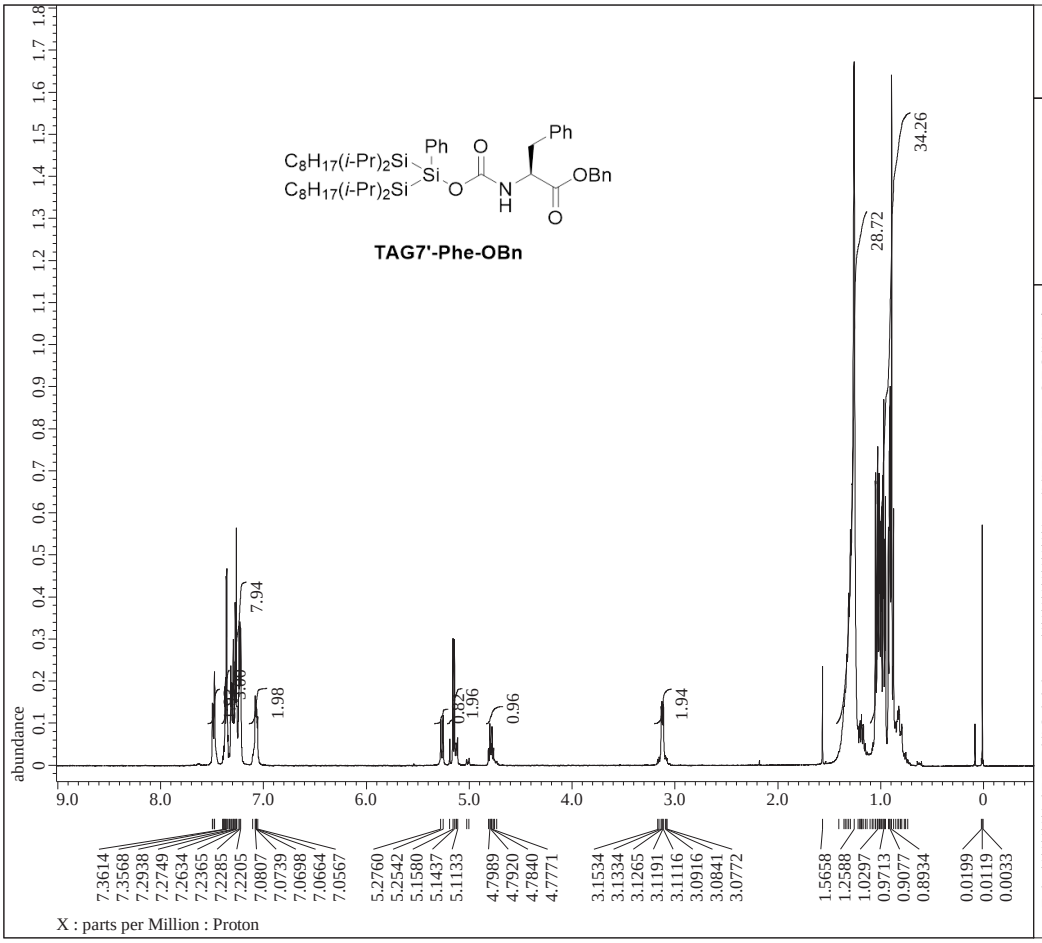
Derived from: wuan-1313_E13C-1-1.jdf

Filename          = wuan-1313_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1313
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2022 17:22:19
Revision_Time     = 22-FEB-2022 17:44:49
Current_Time      = 22-FEB-2022 17:46:04

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

Field_Strength    = 9.20197068[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             = 188
Total_Scans       = 188

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]
```




```
---- 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

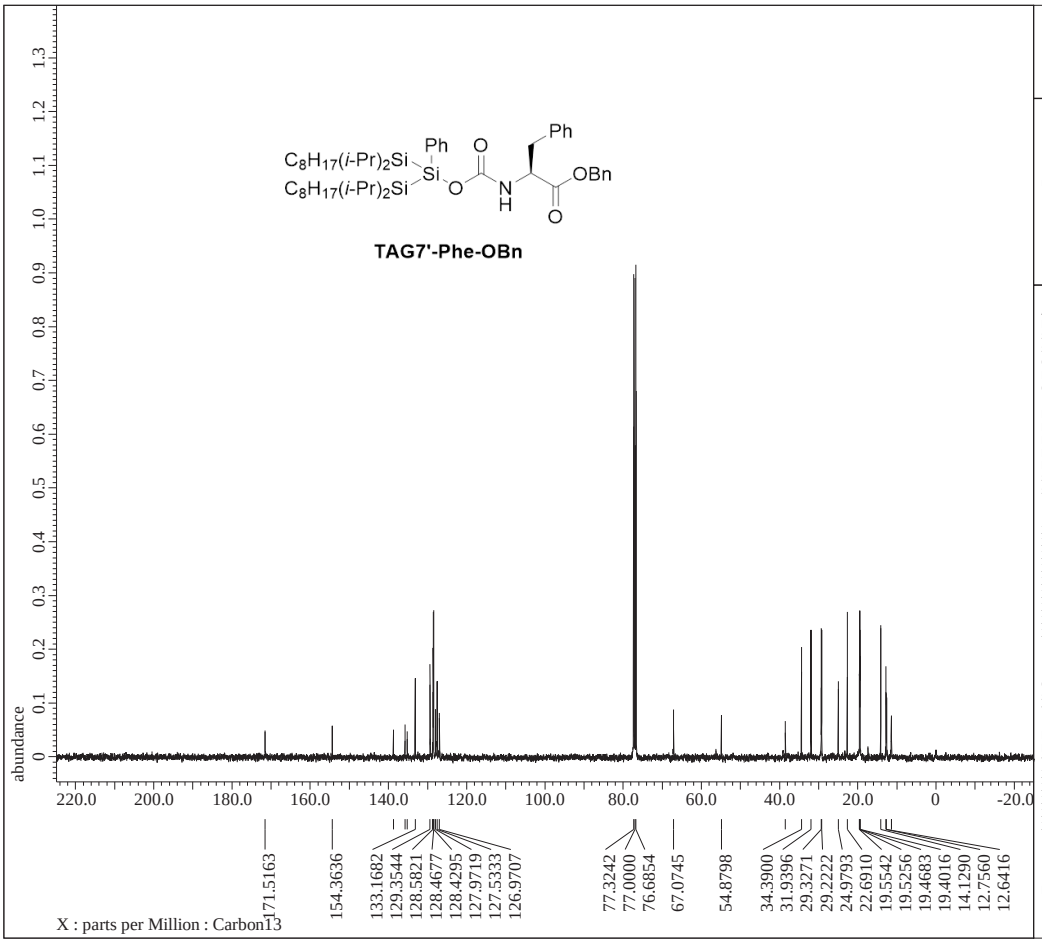
Derived from: wuan-1314_E1H-1-1.jdf

Filename          = wuan-1314_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1314
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2022 17:31:02
Revision_Time     = 22-FEB-2022 17:53:03
Current_Time      = 22-FEB-2022 17:54:22

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        = 40
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

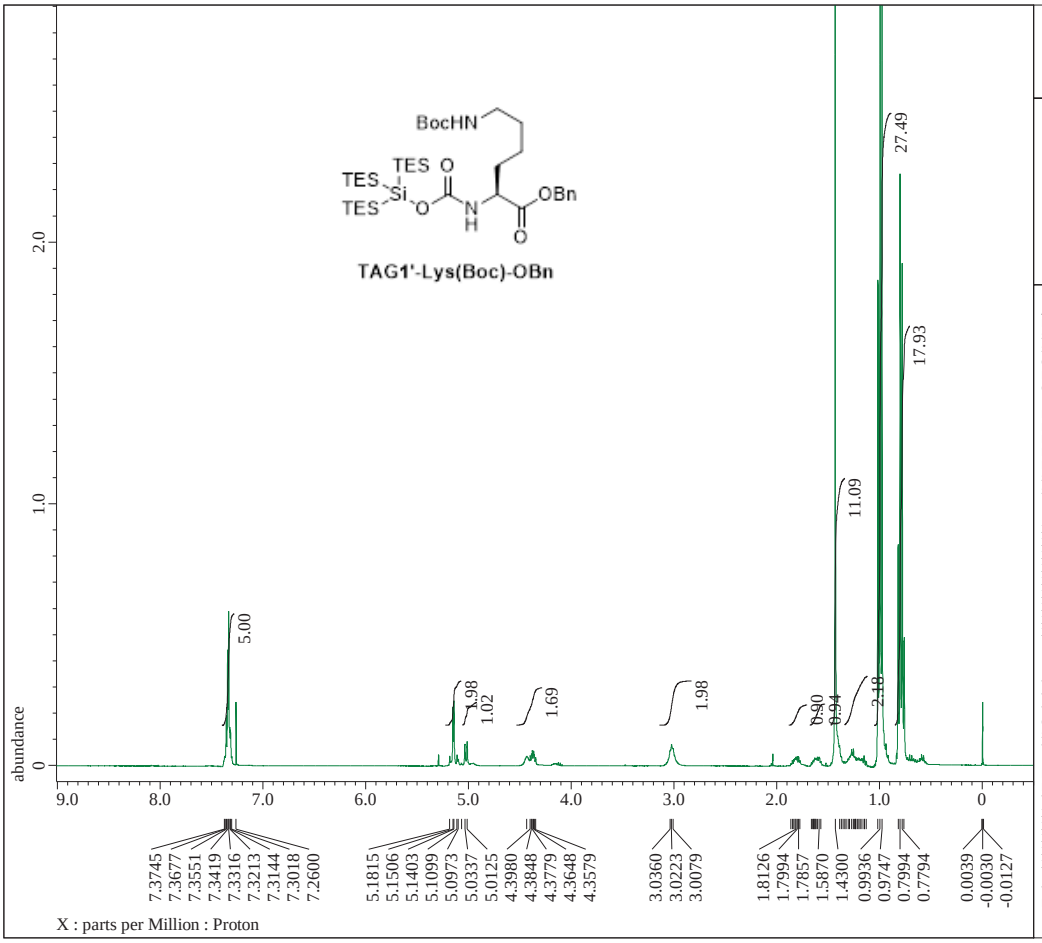
Derived from: wuan-1314_E13C-1-1.jdf

Filename          = wuan-1314_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1314
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2022 17:35:28
Revision_Time     = 22-FEB-2022 17:59:33
Current_Time      = 22-FEB-2022 17:59:54

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 491
Total_Scans       = 491

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

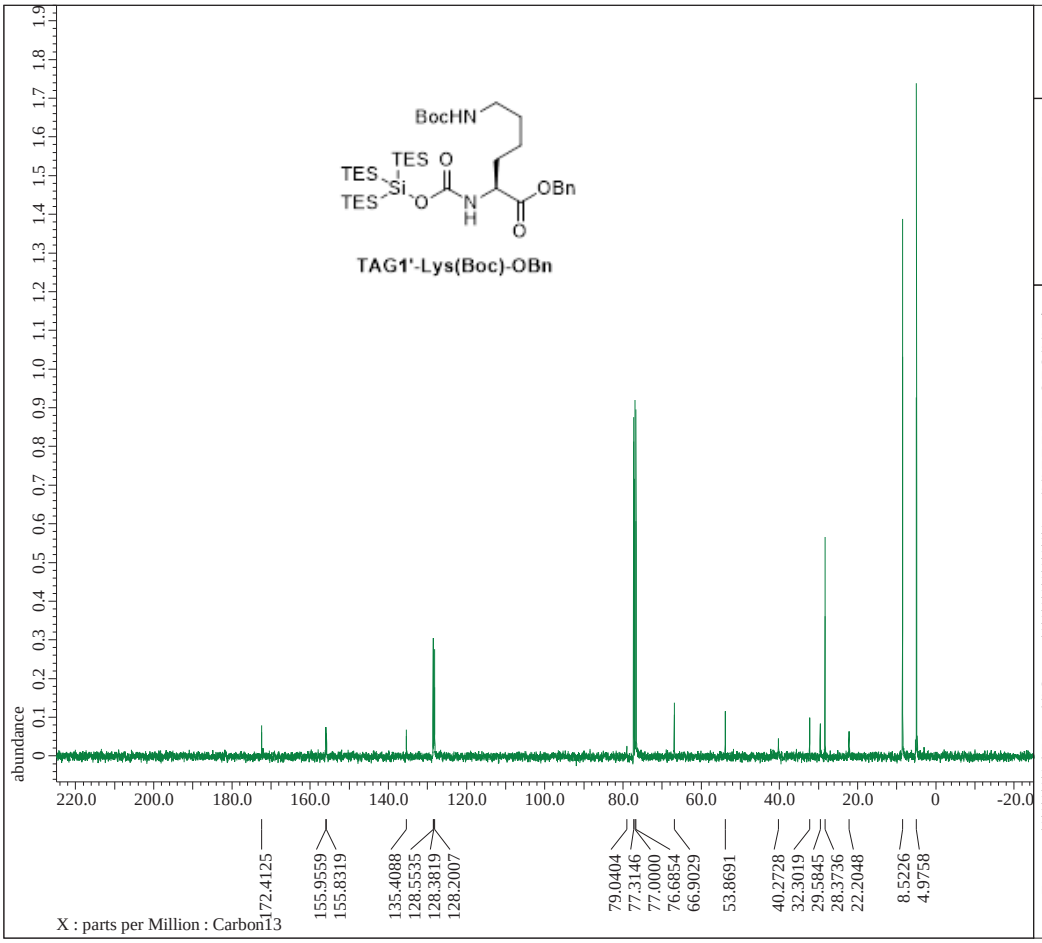
Derived from: wuan-1390_E1H-1-1.jdf
```

```
Filename          = wuan-1390_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1390
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 10:24:45
Revision_Time     = 17-JAN-2023 12:44:54
Current_Time      = 17-JAN-2023 12:46:45

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 24
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

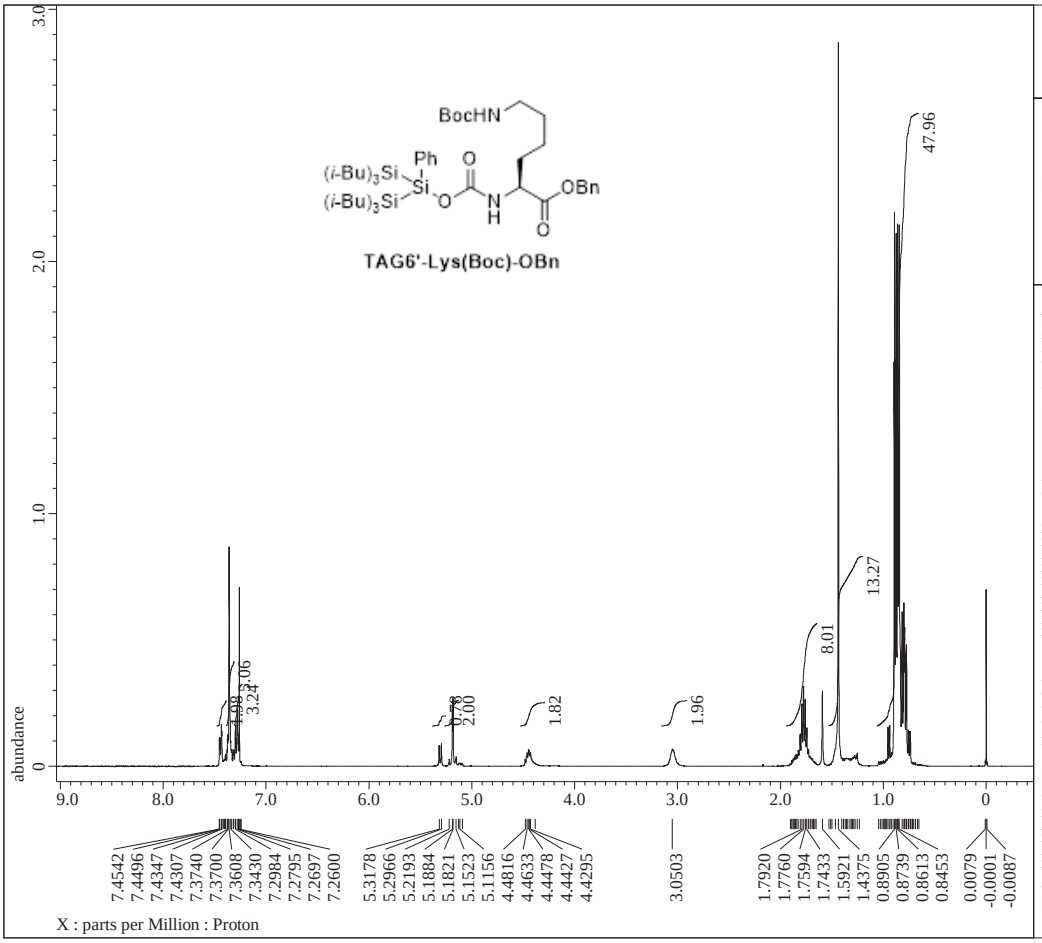
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1390_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1390
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 10:28:45
Revision_Time     = 17-JAN-2023 10:31:18
Current_Time      = 17-JAN-2023 12:50:00

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             = 143
Total_Scans       = 143

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



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

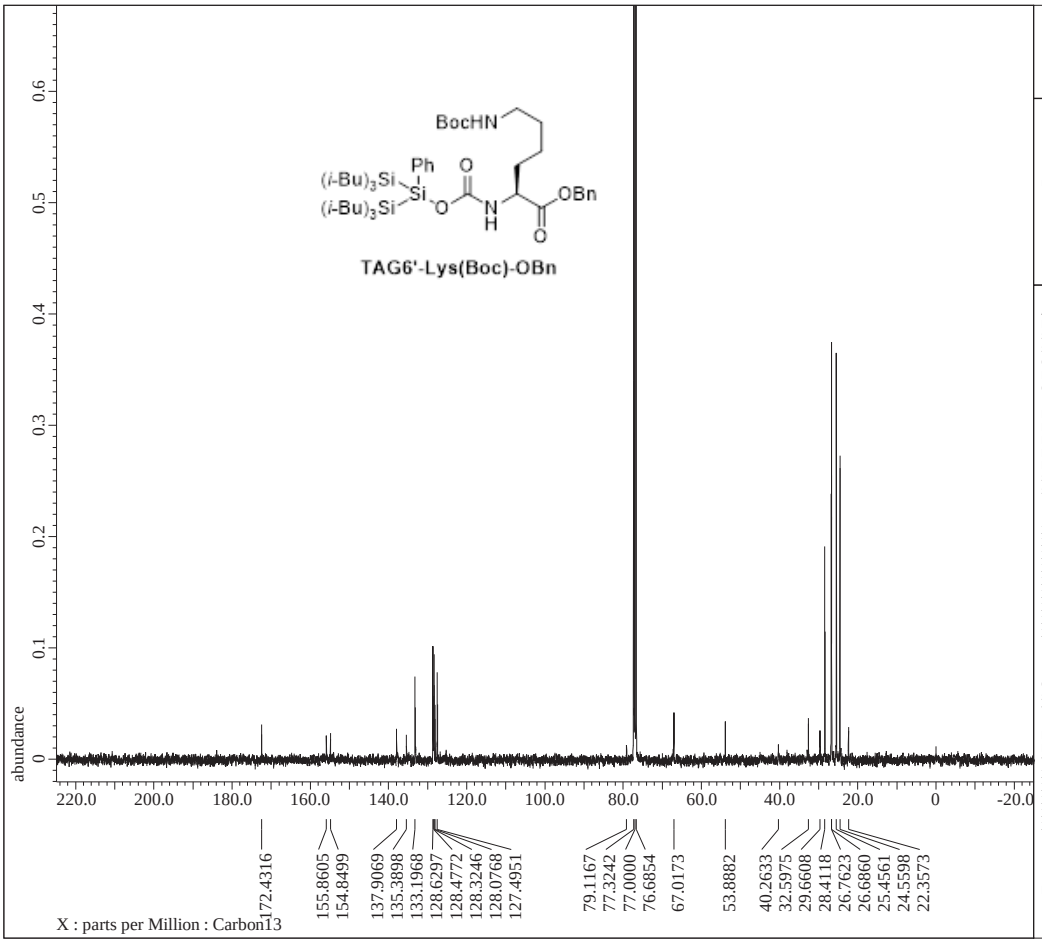
Derived from: wuan-1403_E1H-1-1.jdf
```

```
Filename          = wuan-1403_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1403
Solvent           = CHLOROFORM-D
Creation_Time     = 30-MAY-2022 13:08:27
Revision_Time     = 30-MAY-2022 13:16:19
Current_Time      = 30-MAY-2022 13:19:56

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        = 36
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
```



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

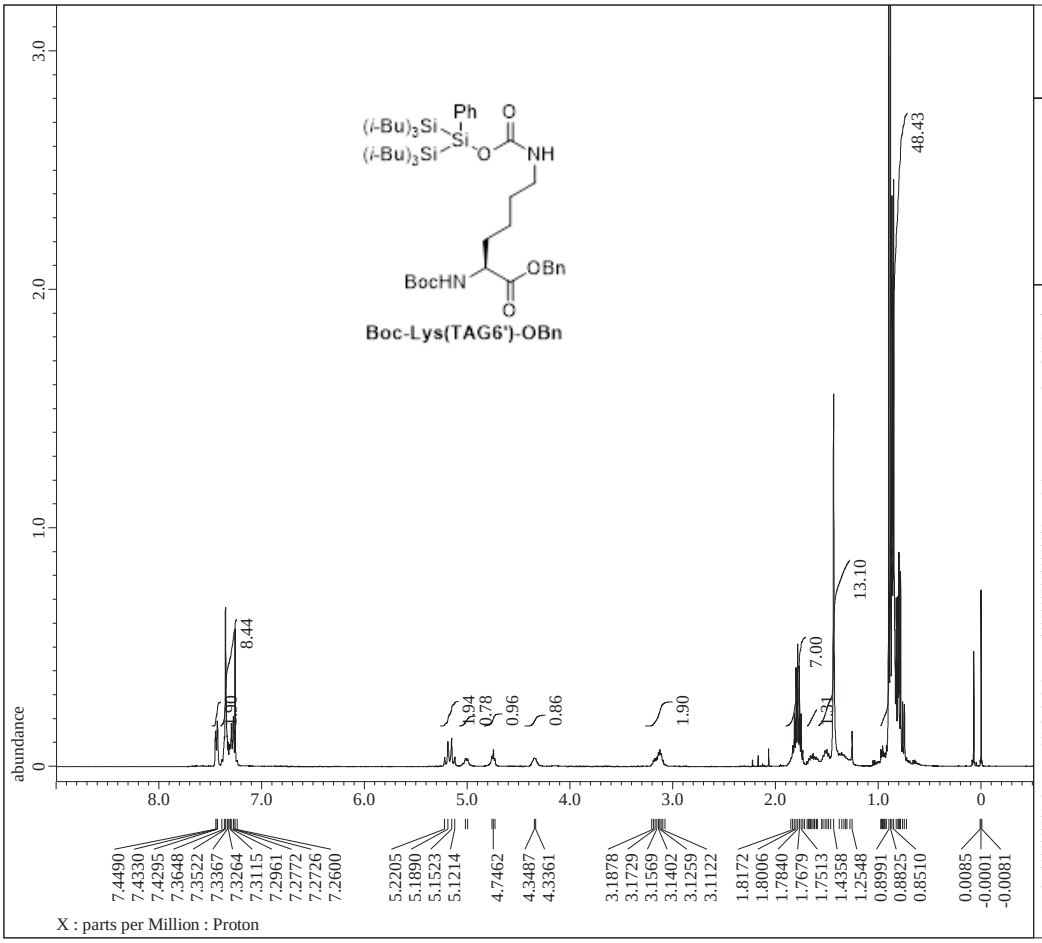
Derived from: wuan-1403_E13C-1-1.jdf
```

```
Filename          = wuan-1403_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1403
Solvent           = CHLOROFORM-D
Creation_Time     = 30-MAY-2022 13:12:51
Revision_Time     = 30-MAY-2022 13:55:22
Current_Time      = 30-MAY-2022 13:56:48

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             = 901
Total_Scans       = 901

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




```
---- 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

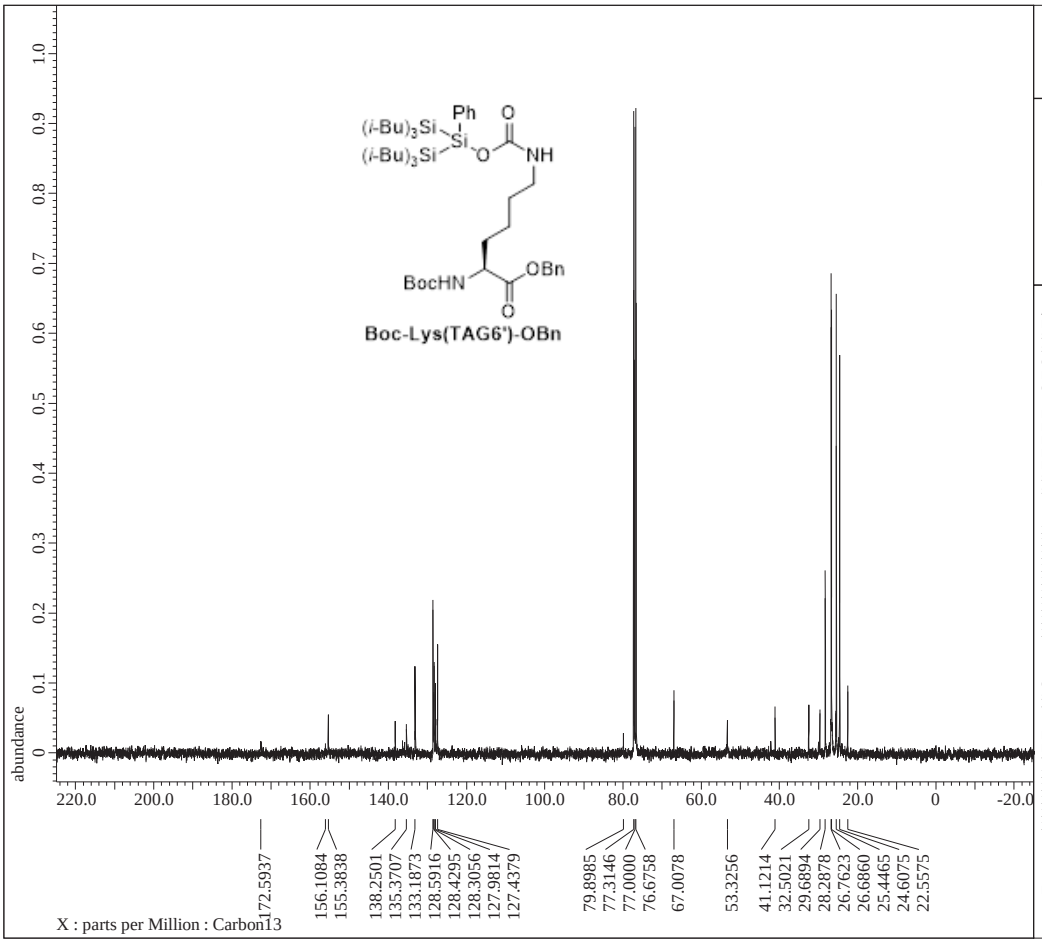
Derived from: wuan-1430_E1H-3-1.jdf

Filename          = wuan-1430_E1H-3-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1430
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JUN-2022 16:02:49
Revision_Time     = 17-JUN-2022 16:14:08
Current_Time      = 17-JUN-2022 16:14:24

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        = 38
Temp_Get          = 23.7[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```




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

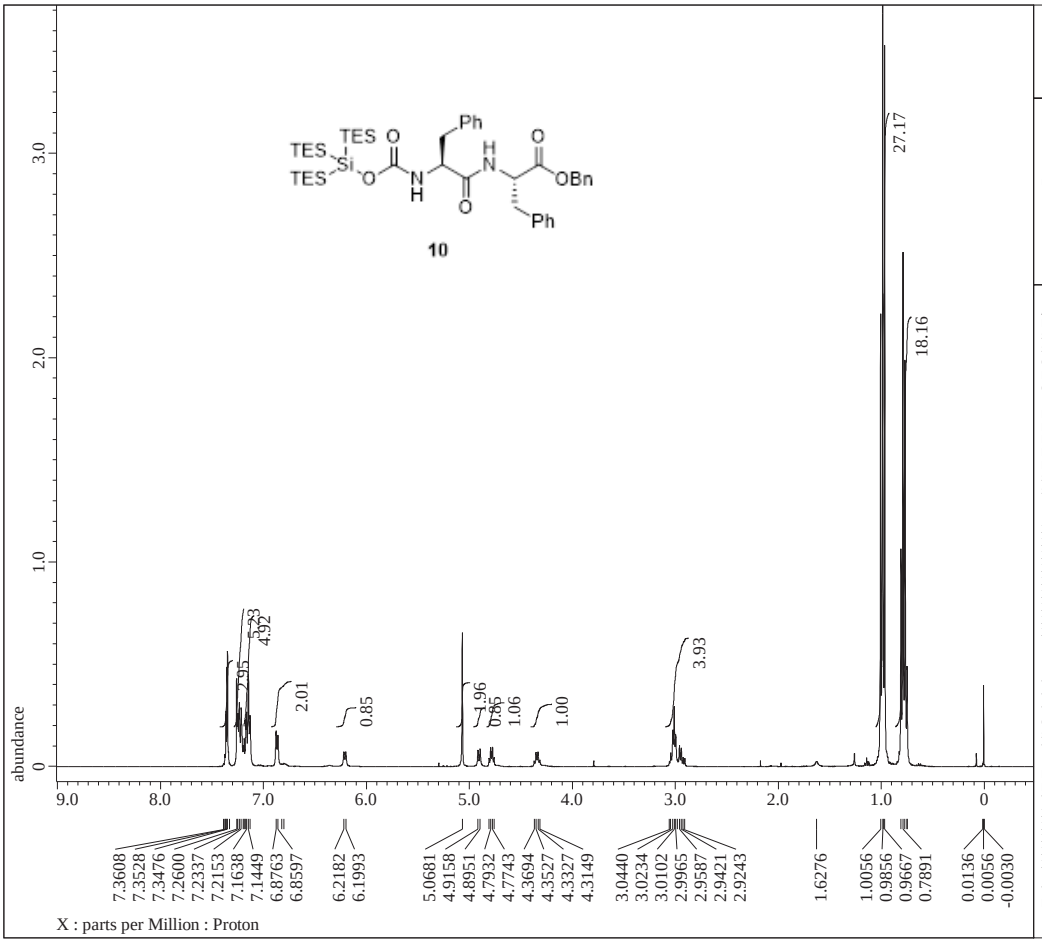
Derived from: wuan-1430_E13C-1-1.jdf

Filename          = wuan-1430_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1430
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JUN-2022 10:04:50
Revision_Time     = 17-JUN-2022 10:17:36
Current_Time      = 17-JUN-2022 10:18:10

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 317
Total_Scans       = 317

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

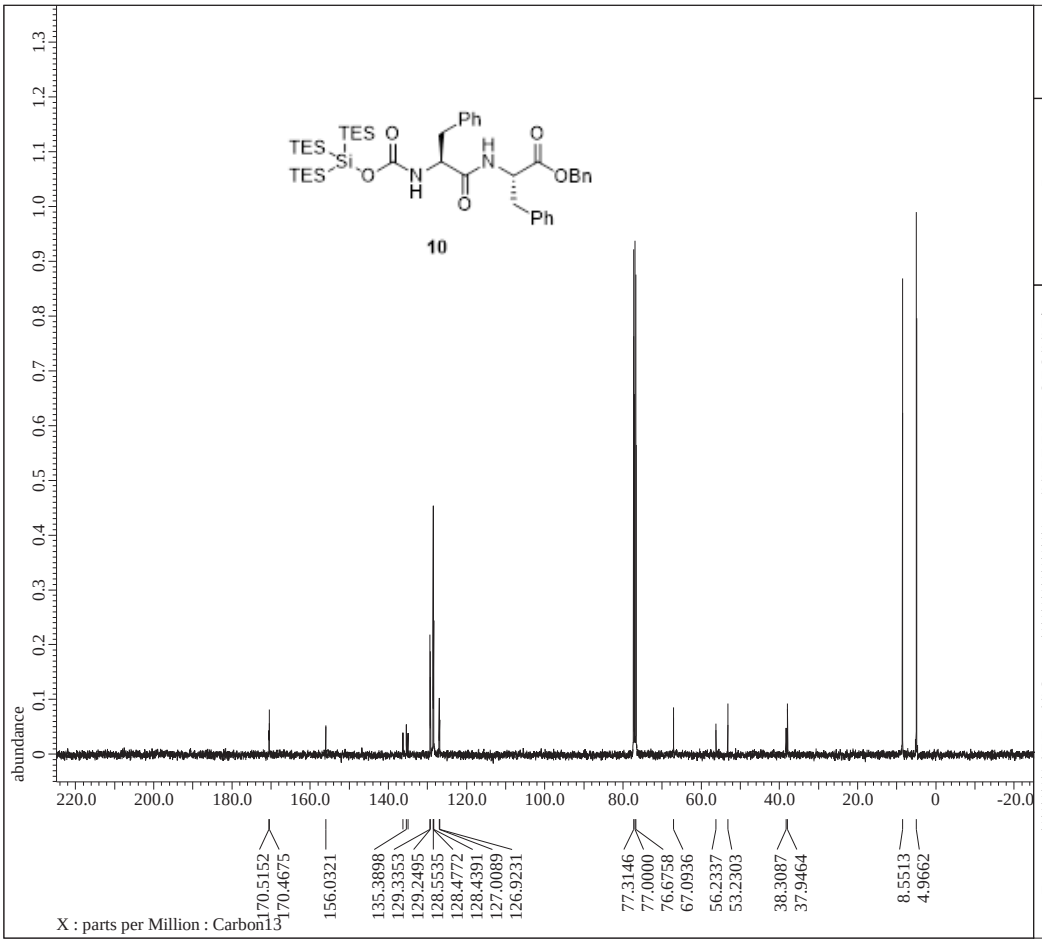
Derived from: wuan-1311_E1H-1-1.jdf
```

```
Filename          = wuan-1311_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1311
Solvent           = CHLOROFORM-D
Creation_Time     = 25-FEB-2022 13:02:56
Revision_Time     = 25-FEB-2022 13:08:04
Current_Time      = 25-FEB-2022 13:08:51

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 23.4[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

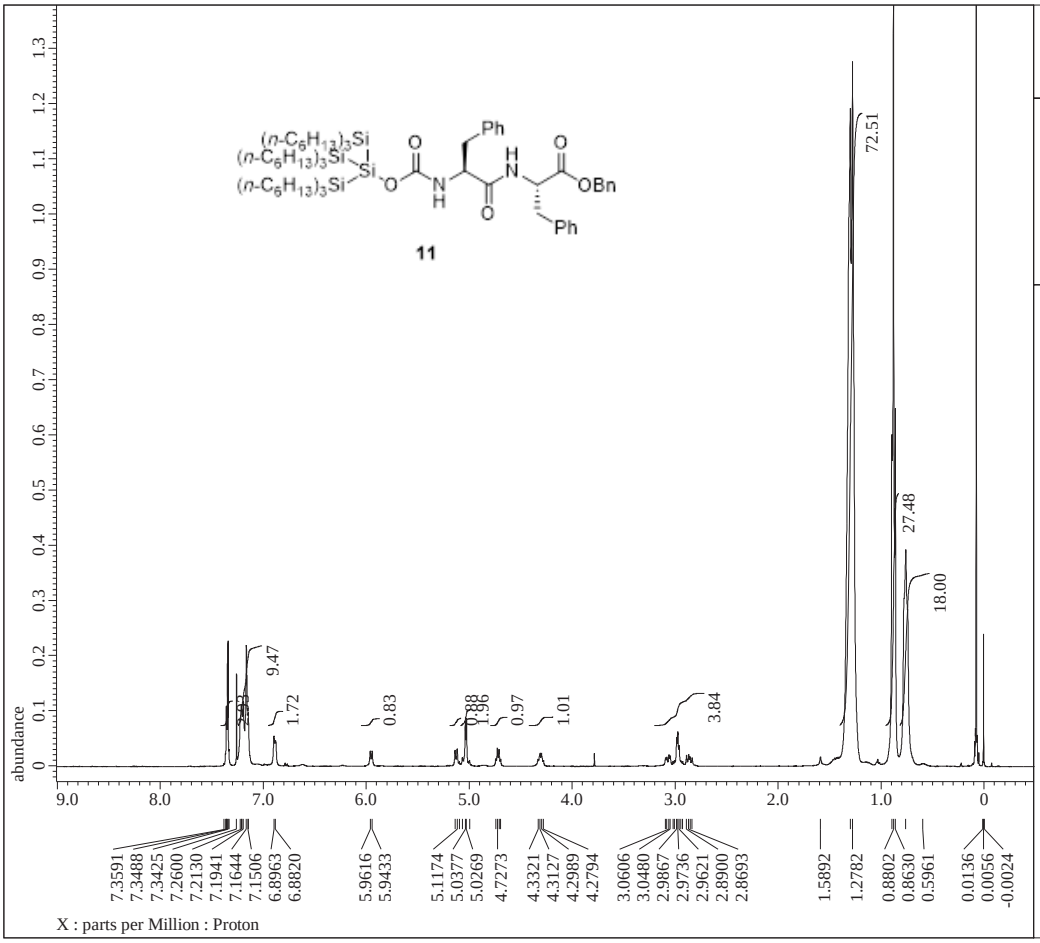
Derived from: wuan-1311_E13C-1-1.jdf
```

```
Filename          = wuan-1311_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1311
Solvent           = CHLOROFORM-D
Creation_Time     = 25-FEB-2022 13:07:39
Revision_Time     = 25-FEB-2022 13:21:07
Current_Time      = 25-FEB-2022 13:21:46

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             = 351
Total_Scans       = 351

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

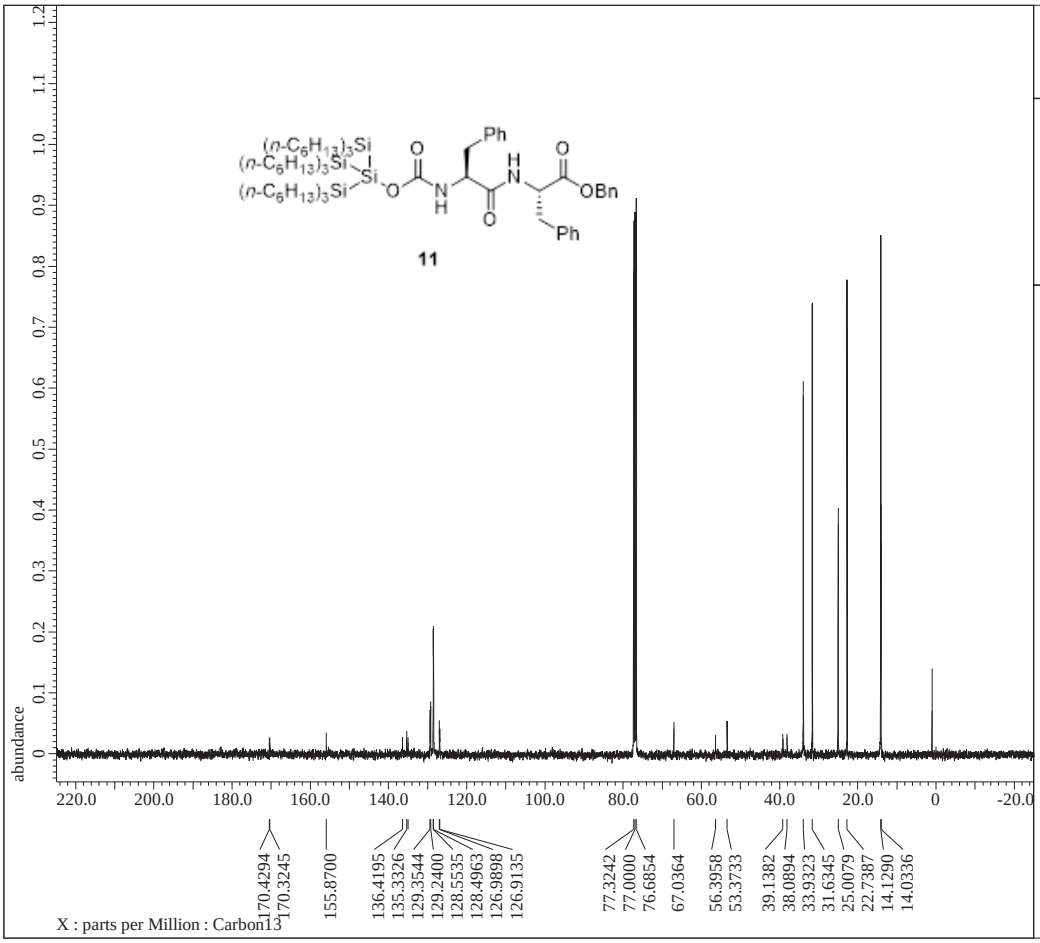
Derived from: wuan-1318_E1H-1-1.jdf
```

```
Filename          = wuan-1318_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1318
Solvent           = CHLOROFORM-D
Creation_Time     = 28-FEB-2022 12:52:41
Revision_Time     = 28-FEB-2022 12:57:03
Current_Time      = 28-FEB-2022 12:57:49

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

Field_Strength    = 9.389766[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          = 24.3[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-1318_E13C-1-1.jdf
```

```
Filename          = wuan-1318_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1318
Solvent           = CHLOROFORM-D
Creation_Time     = 28-FEB-2022 12:56:37
Revision_Time     = 28-FEB-2022 13:10:43
Current_Time      = 28-FEB-2022 13:11:23

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             = 368
Total_Scans       = 368

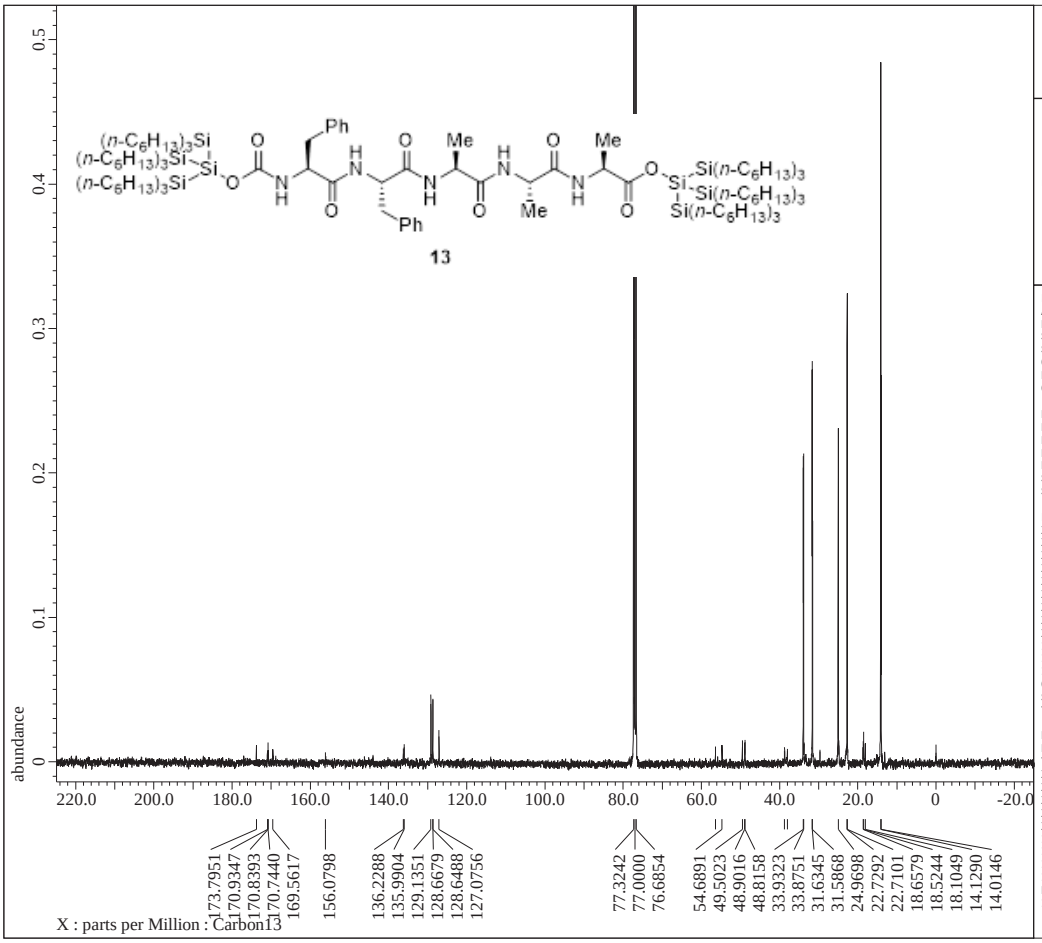
Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.5[dC]
X_90_Width        = 10.1[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4.6[dB]
X_Pulse           = 3.36666667[us]
Irr_Atn_Dec       = 25.996[dB]
Irr_Atn_Noe       = 25.996[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
```

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-1349-1_E1H-1-1.jdf
```

Filename	= wuan-1349-1_E1H-1-4.jdf
Author	= delta
Experiment	= single_pulse.jxp
Sample_Id	= wuan-1349-1
Solvent	= CHLOROFORM-D
Creation_Time	= 8-APR-2022 19:47:41
Revision_Time	= 8-APR-2022 19:49:01
Current_Time	= 8-APR-2022 19:49:44
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 4.36731904[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.22897343[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 26
Temp_Get	= 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

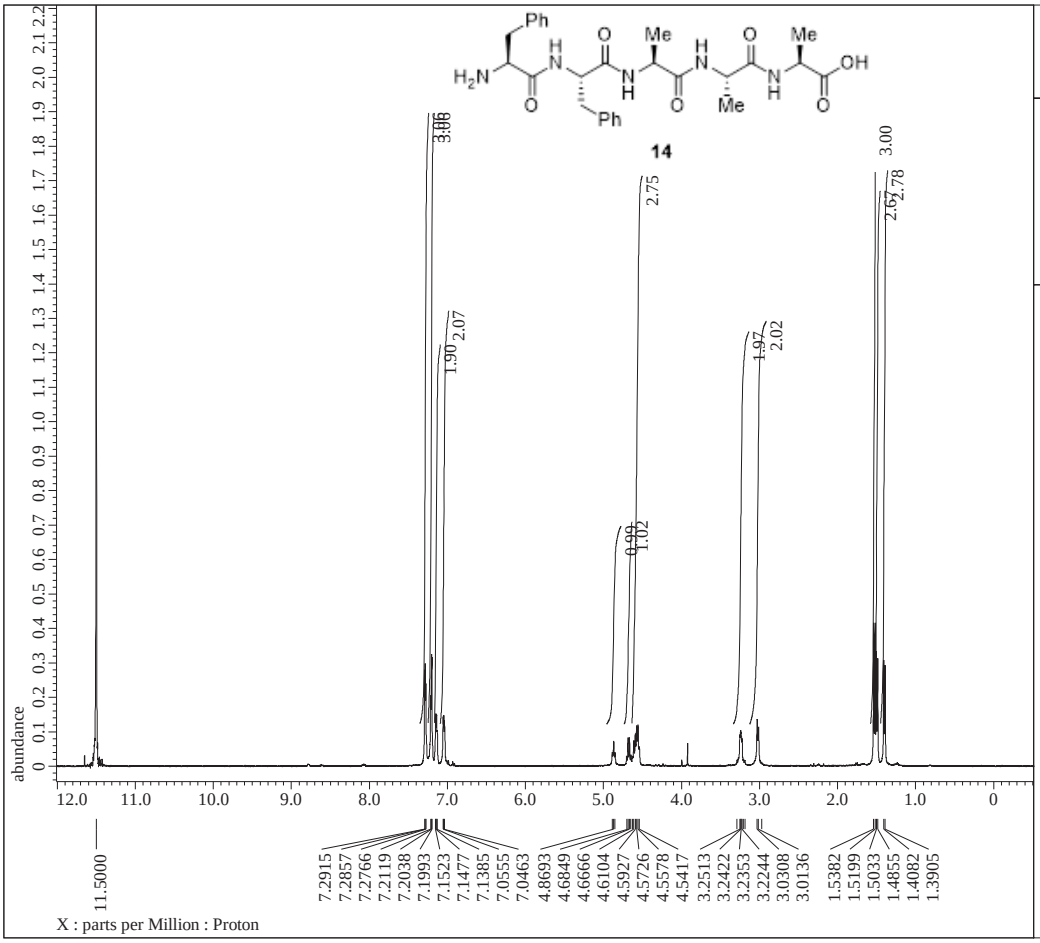


JEOL RESONANCE

```
---- 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-1349-1_E13C-1-1.jdf
```

Filename	= wuan-1349-1_E13C-1-4.jdf
Author	= delta
Experiment	= single_pulse_dec.jxp
Sample_Id	= wuan-1349-1
Solvent	= CHLOROFORM-D
Creation_Time	= 8-APR-2022 17:04:08
Revision_Time	= 8-APR-2022 19:17:56
Current_Time	= 8-APR-2022 19:18:39
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 2673
Total_Scans	= 2673
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]



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

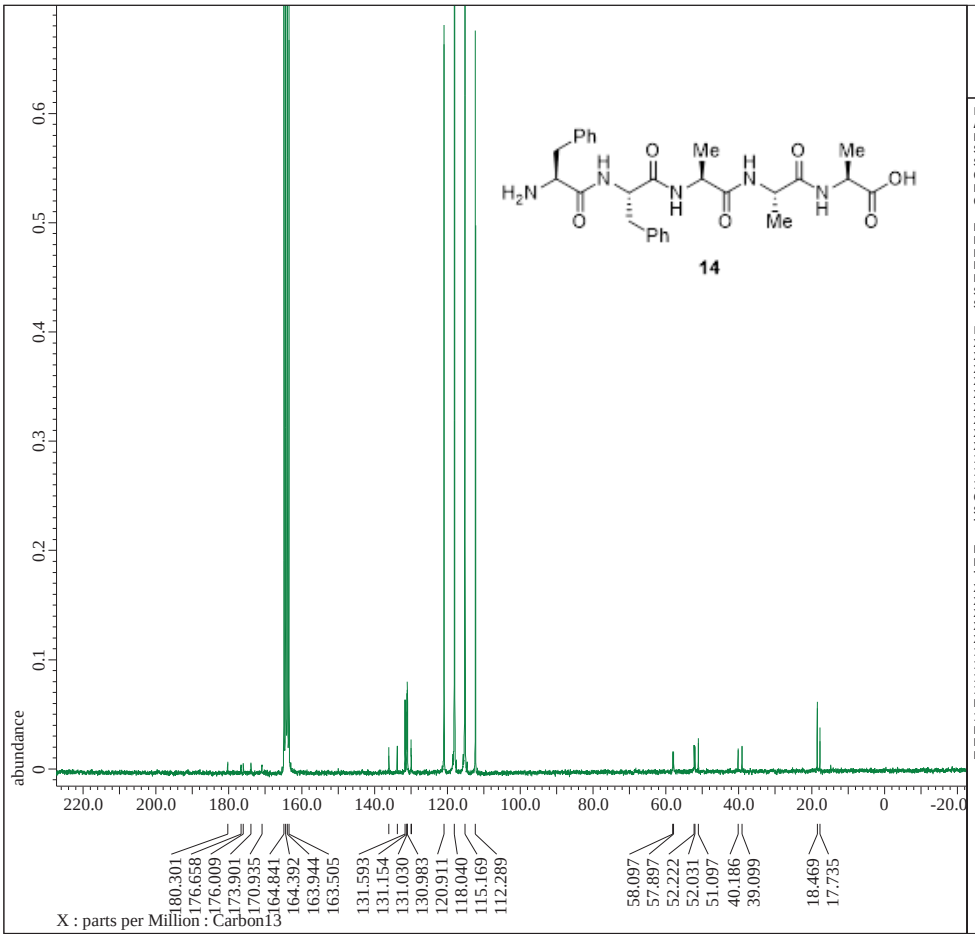
Derived from: wuan-1351-4_E1H-1-1.jdf
```

```
Filename          = wuan-1351-4_E1H-1-4.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1351-4
Solvent           = TRIFLUOROACETIC_ACID-D
Creation_Time     =  9-APR-2022 15:08:35
Revision_Time     =  9-APR-2022 15:11:20
Current_Time      =  9-APR-2022 15:11:45

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

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

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
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
```



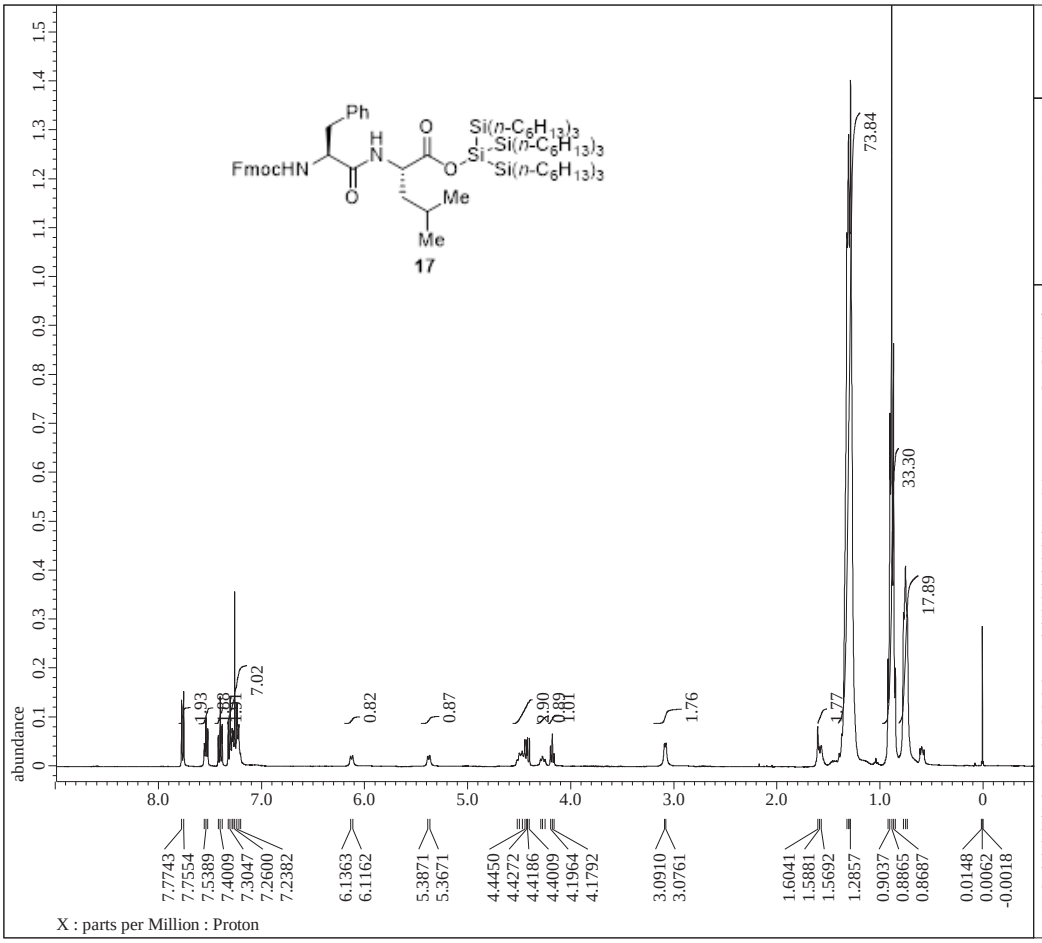
JEOL RESONANCE

```
Filename          = 20220411_wuan-1352_E13C-1
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1352
Solvent           = TRIFLUOROACETIC_ACID-D
Creation_Time     = 11-APR-2022 15:19:20
Revision_Time     = 11-APR-2022 18:43:46
Current_Time      = 11-APR-2022 18:46:03

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

Field_Strength    = 9.20197068[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             = 4000
Total_Scans       = 4000

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]
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

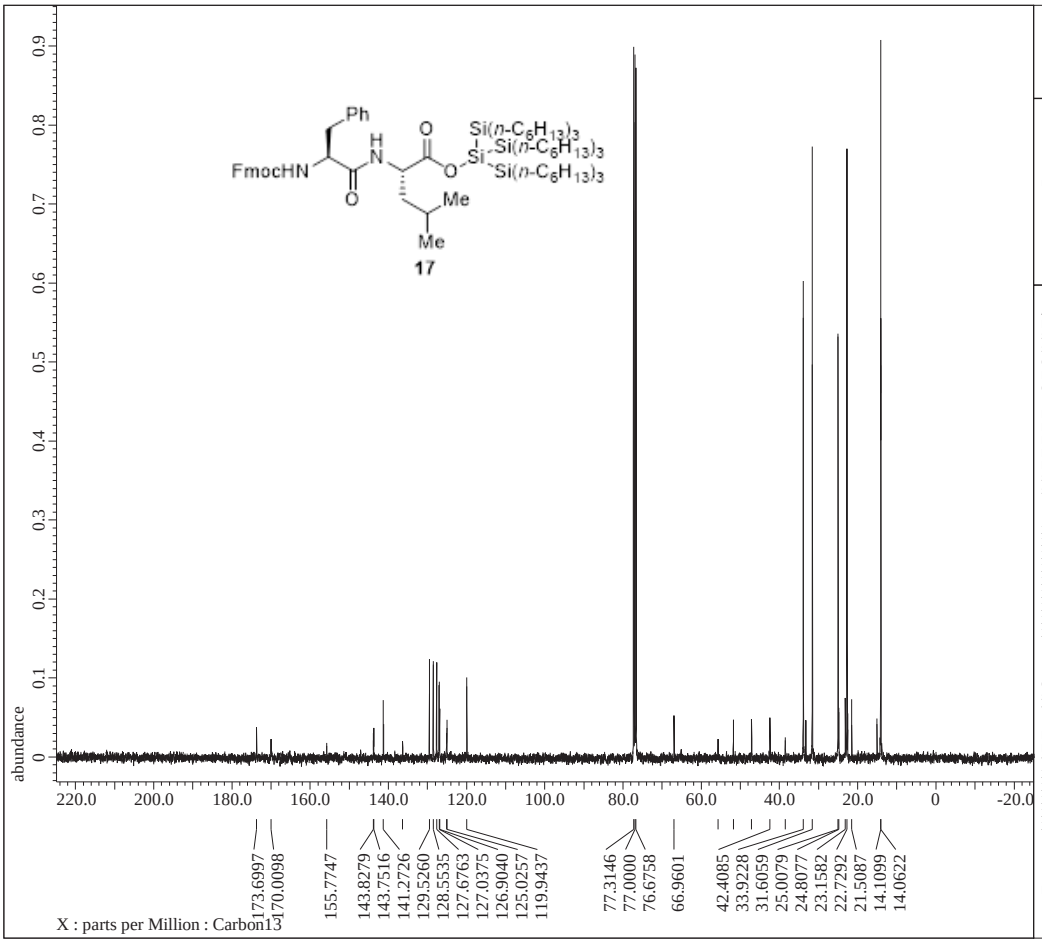
Derived from: wuan-1383_E1H-1-1.jdf
```

```
Filename          = wuan-1383_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1383
Solvent           = CHLOROFORM-D
Creation_Time     = 19-MAY-2022 15:11:57
Revision_Time     = 19-MAY-2022 15:15:03
Current_Time      = 19-MAY-2022 15:17:24

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        = 22
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

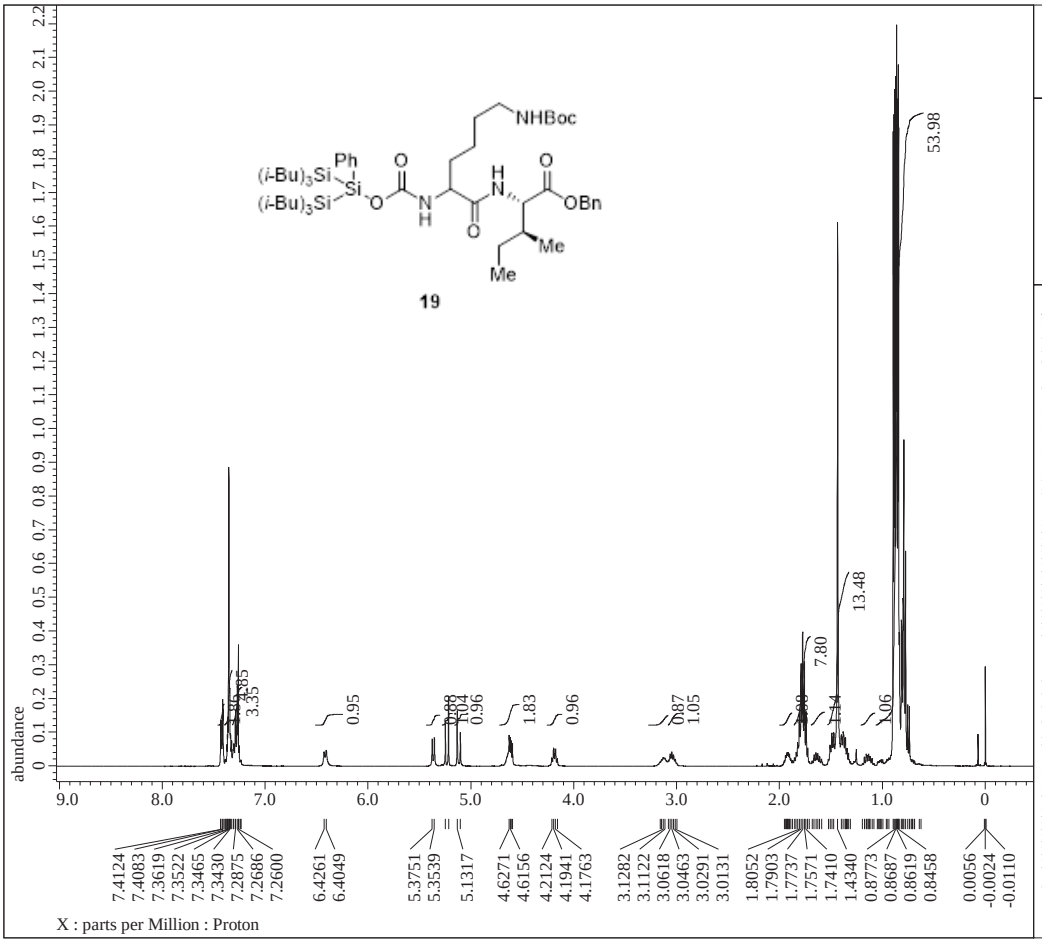
Derived from: wuan-1383_E13C-1-1.jdf
```

```
Filename          = wuan-1383_E13C-1-6.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1383
Solvent           = CHLOROFORM-D
Creation_Time     = 19-MAY-2022 15:15:53
Revision_Time     = 19-MAY-2022 15:37:43
Current_Time      = 19-MAY-2022 15:38:06

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             = 510.0
Total_Scans       = 510.0

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



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

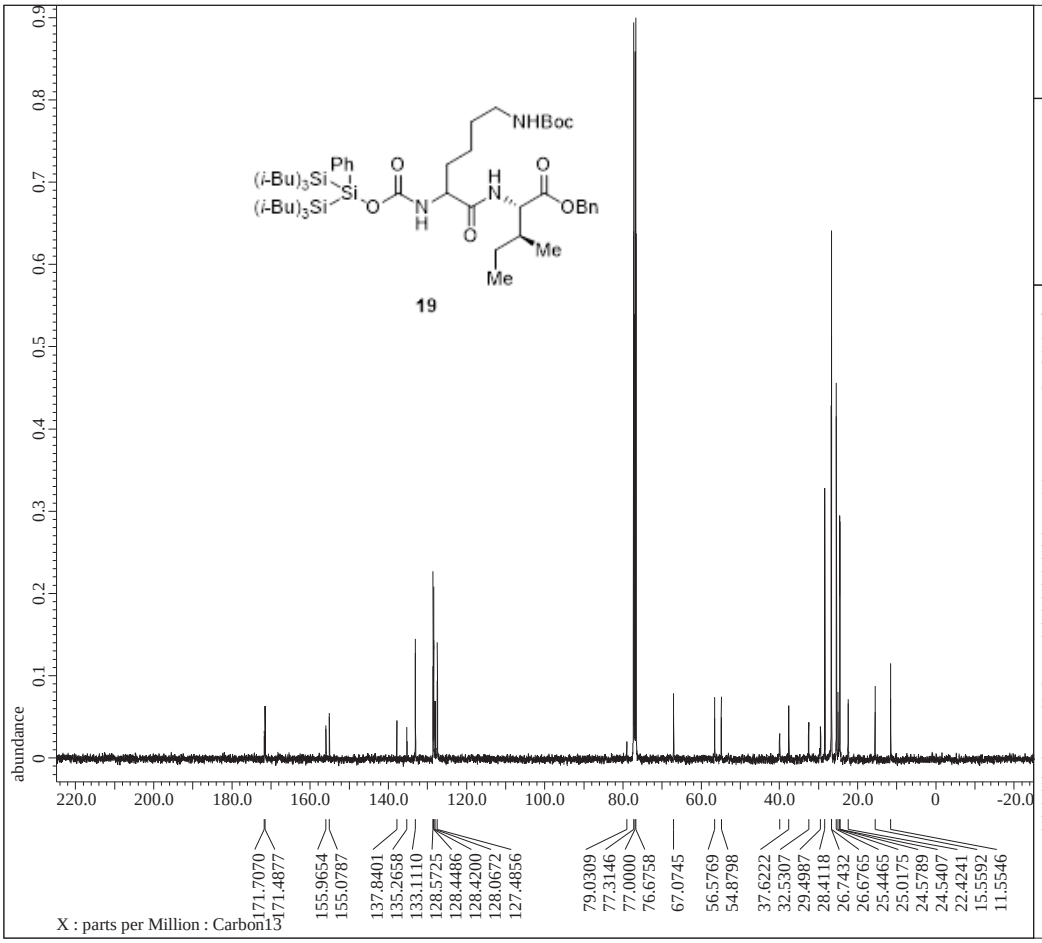
Derived from: wuan-1406_E1H-1-1.jdf
```

```
Filename          = wuan-1406_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1406
Solvent           = CHLOROFORM-D
Creation_Time     = 31-MAY-2022 11:10:33
Revision_Time     = 31-MAY-2022 11:24:06
Current_Time      = 31-MAY-2022 11:24:35

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        = 24
Temp_Get          = 23.6[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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

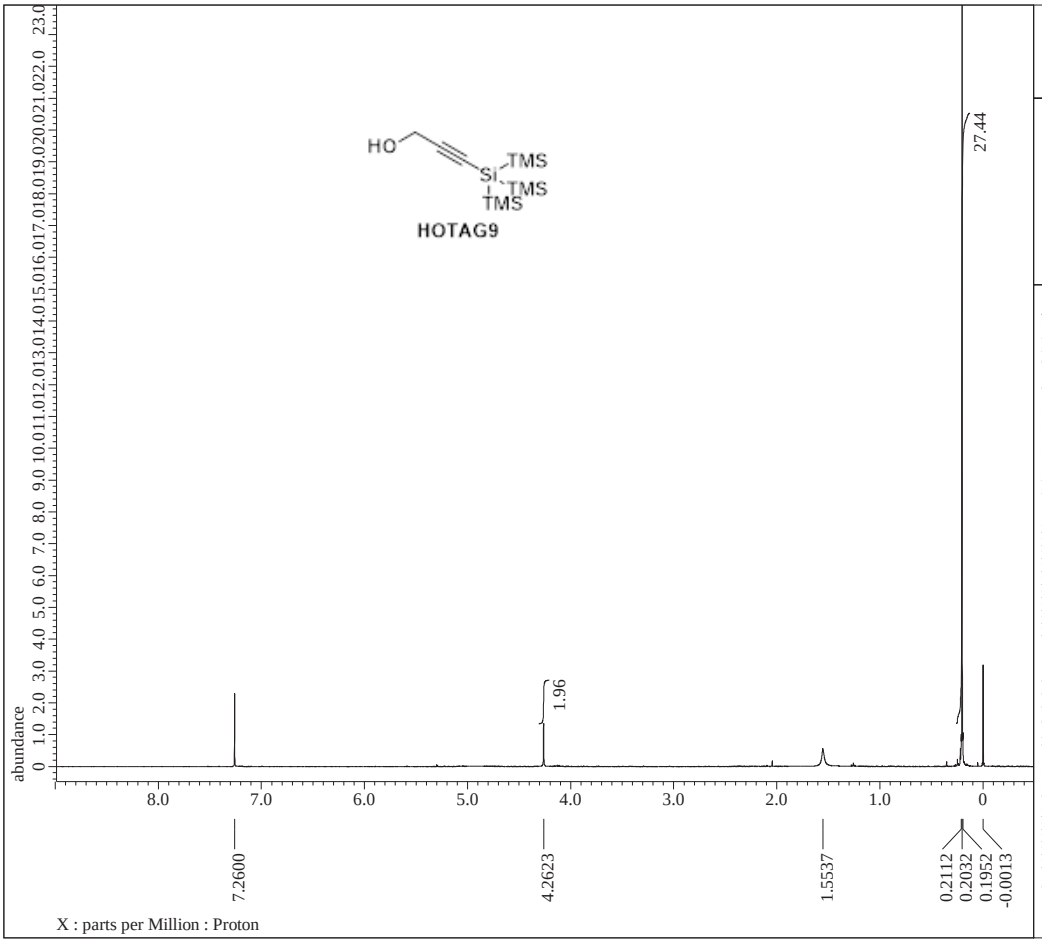
Derived from: wuan-1406_E13C-1-1.jdf
```

```
Filename          = wuan-1406_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1406
Solvent           = CHLOROFORM-D
Creation_Time     = 31-MAY-2022 11:15:14
Revision_Time     = 31-MAY-2022 11:49:37
Current_Time      = 31-MAY-2022 11:50:17

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             = 770
Total_Scans       = 770

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

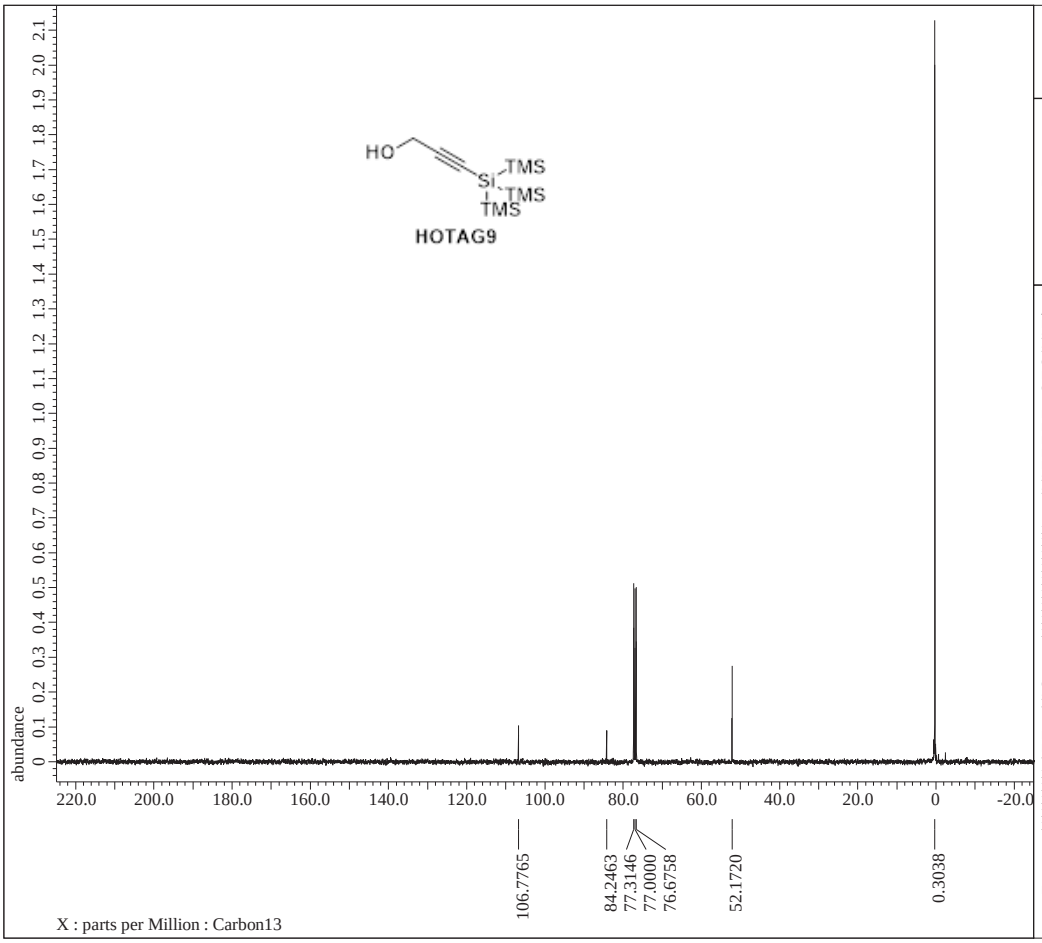
Derived from: wuan-1474_E1H-1-1.jdf
```

```
Filename          = wuan-1474_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1474
Solvent           = CHLOROFORM-D
Creation_Time     =  5-AUG-2022 13:32:49
Revision_Time     =  5-AUG-2022 13:33:12
Current_Time      =  5-AUG-2022 13:33:49

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

Field_Strength    = 9.389766[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          = 25[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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

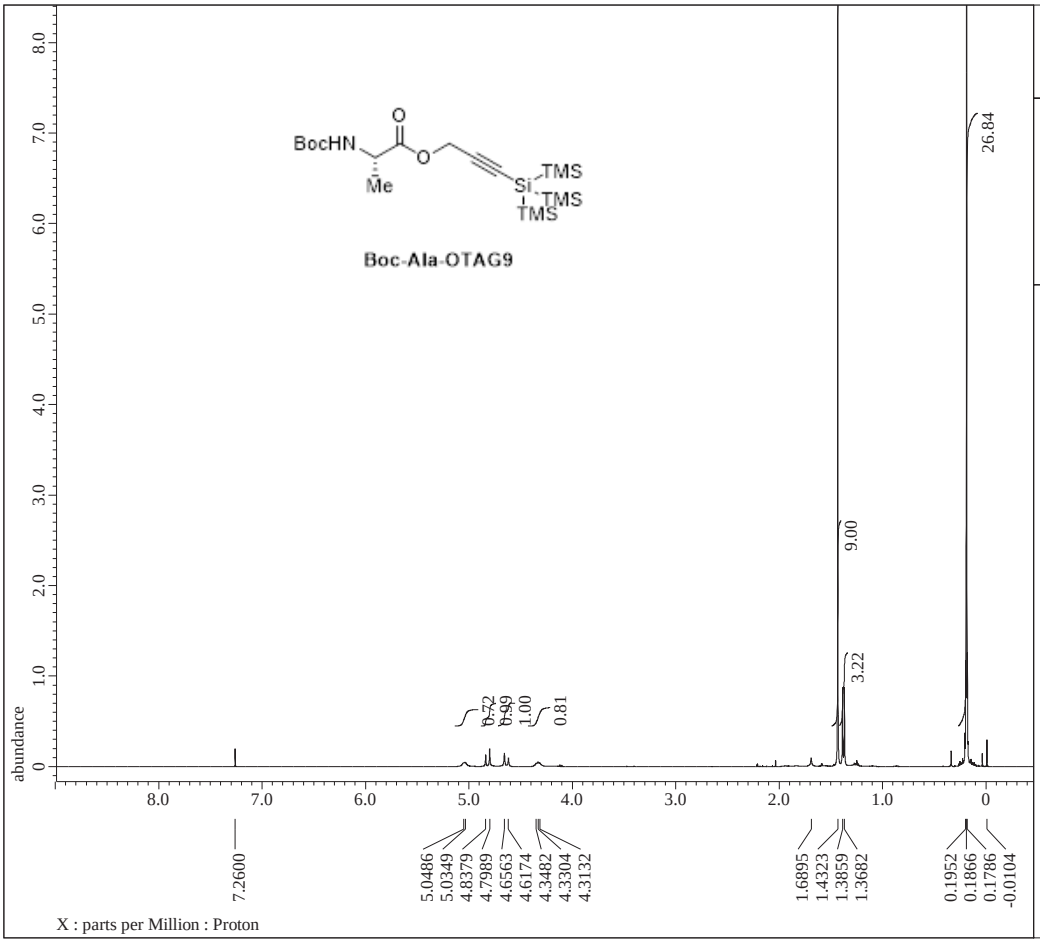
Derived from: wuan-1474_E13C-1-1.jdf
```

```
Filename          = wuan-1474_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1474
Solvent           = CHLOROFORM-D
Creation_Time     =  5-AUG-2022 15:43:48
Revision_Time     =  5-AUG-2022 15:57:27
Current_Time      =  5-AUG-2022 15:57:47

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             = 365
Total_Scans       = 365

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25[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

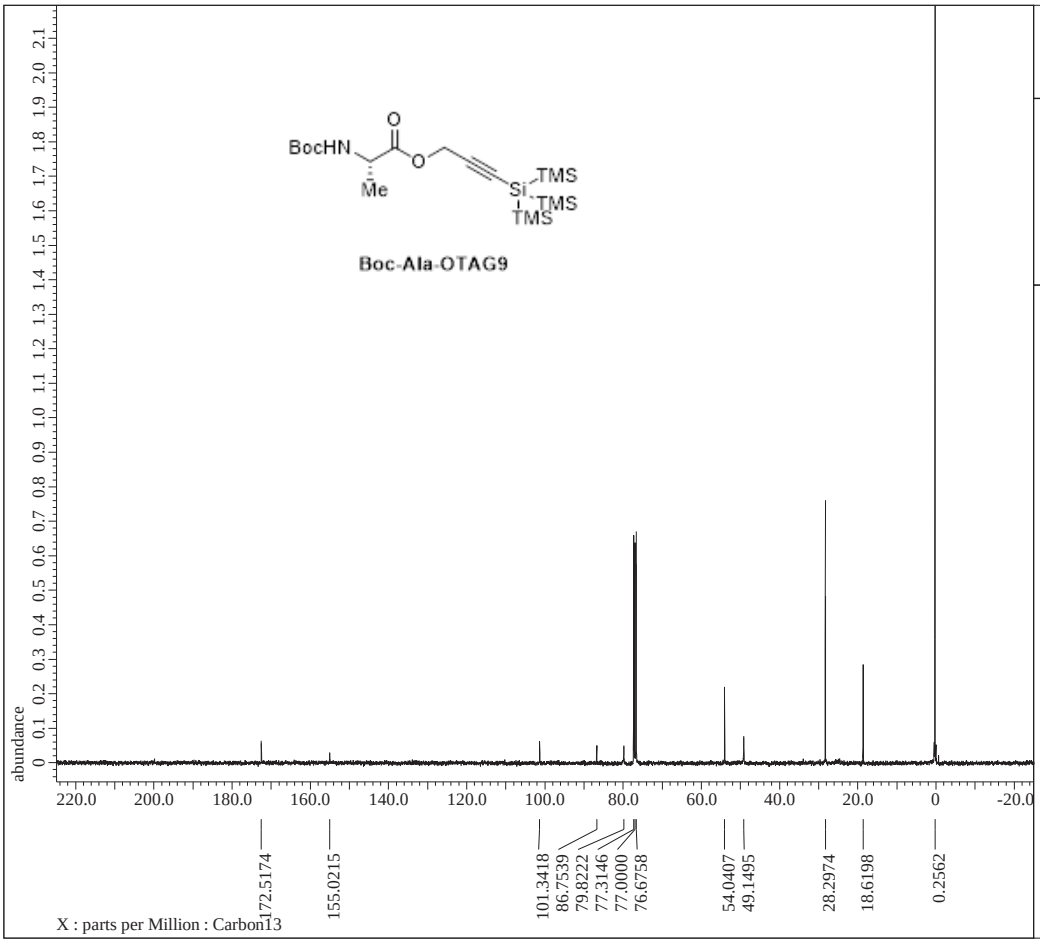
Derived from: wuan-1475_E1H-1-1.jdf
```

```
Filename          = wuan-1475_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1475
Solvent           = CHLOROFORM-D
Creation_Time     =  6-AUG-2022 10:42:25
Revision_Time     =  6-AUG-2022 11:05:47
Current_Time      =  6-AUG-2022 11:06:19

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
```




```
---- 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

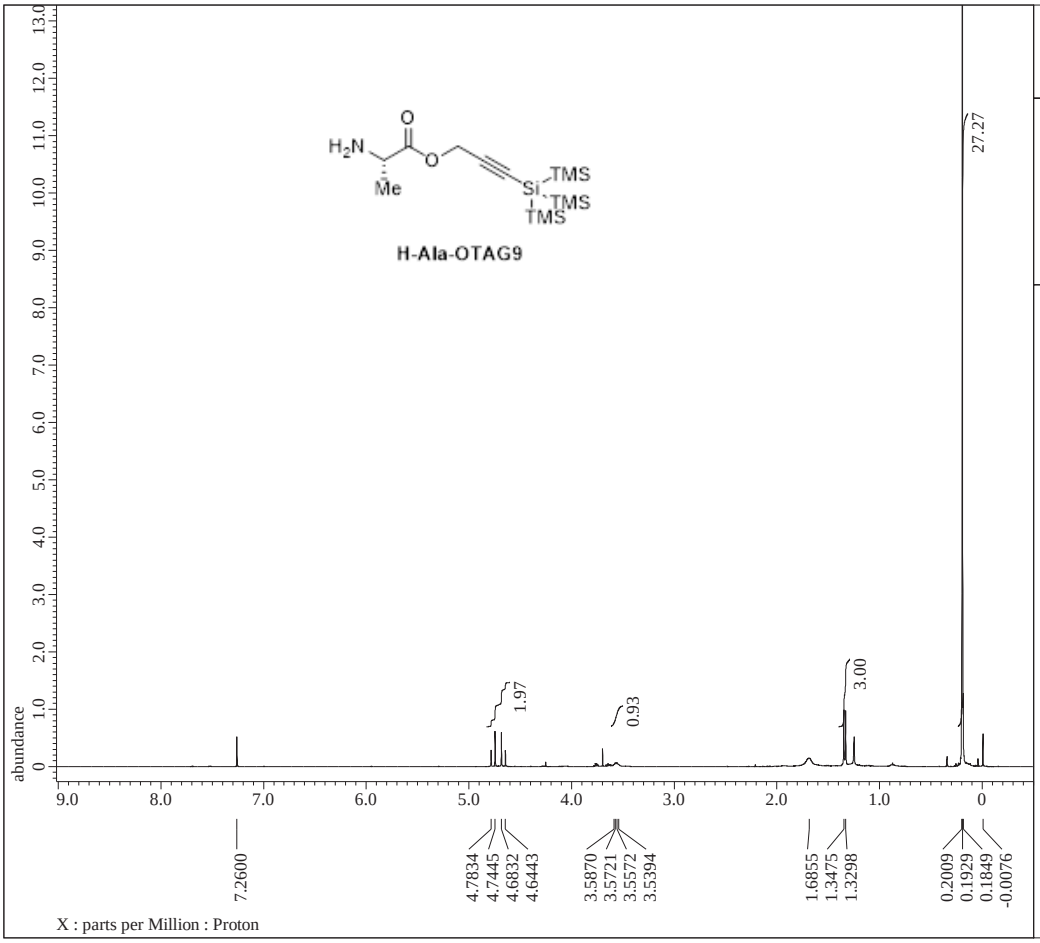
Derived from: wuan-1475_E13C-1-1.jdf
```

```
Filename          = wuan-1475_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1475
Solvent           = CHLOROFORM-D
Creation_Time     =  6-AUG-2022 10:46:20
Revision_Time     =  6-AUG-2022 11:08:30
Current_Time      =  6-AUG-2022 11:08:44

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 528
Total_Scans       = 528

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25[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

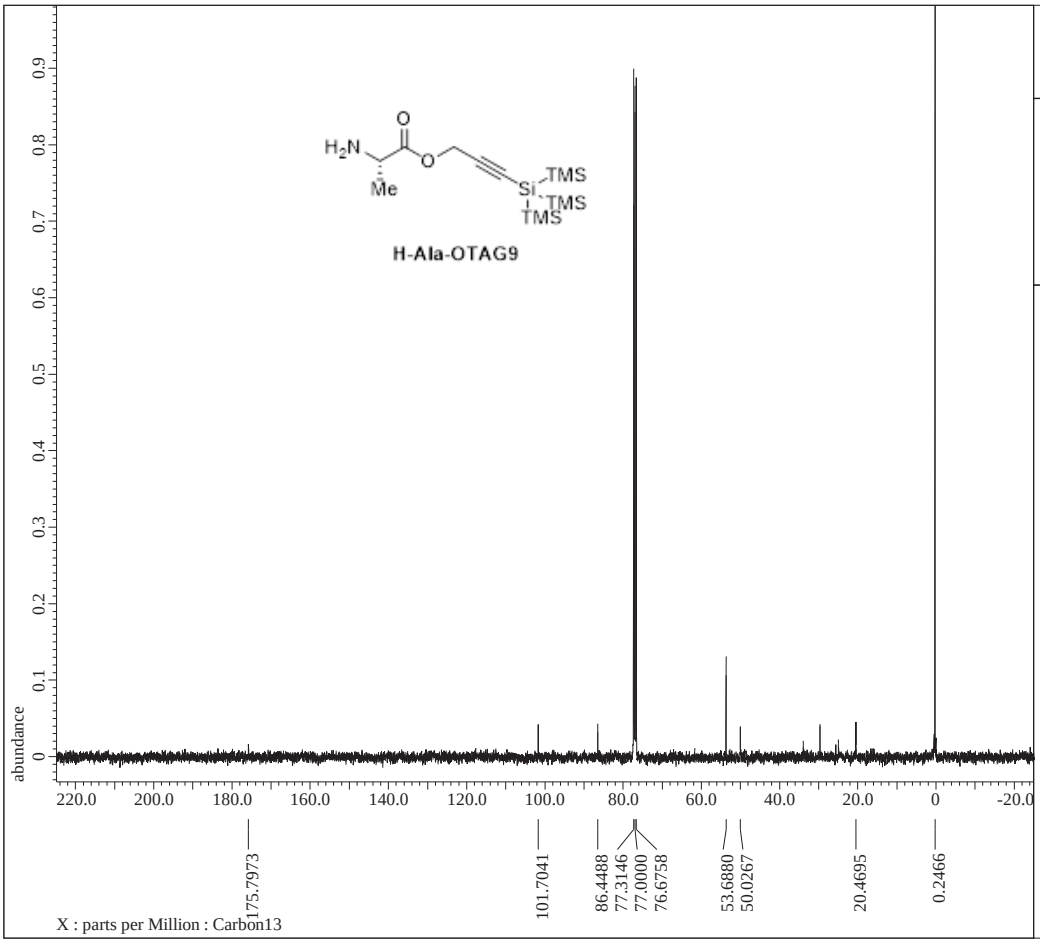
Derived from: wuan-1481_E1H-1-1.jdf
```

```
Filename          = wuan-1481_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1481
Solvent           = CHLOROFORM-D
Creation_Time     = 23-AUG-2022 13:45:26
Revision_Time     = 23-AUG-2022 14:03:02
Current_Time      = 23-AUG-2022 14:03:44

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
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
```



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

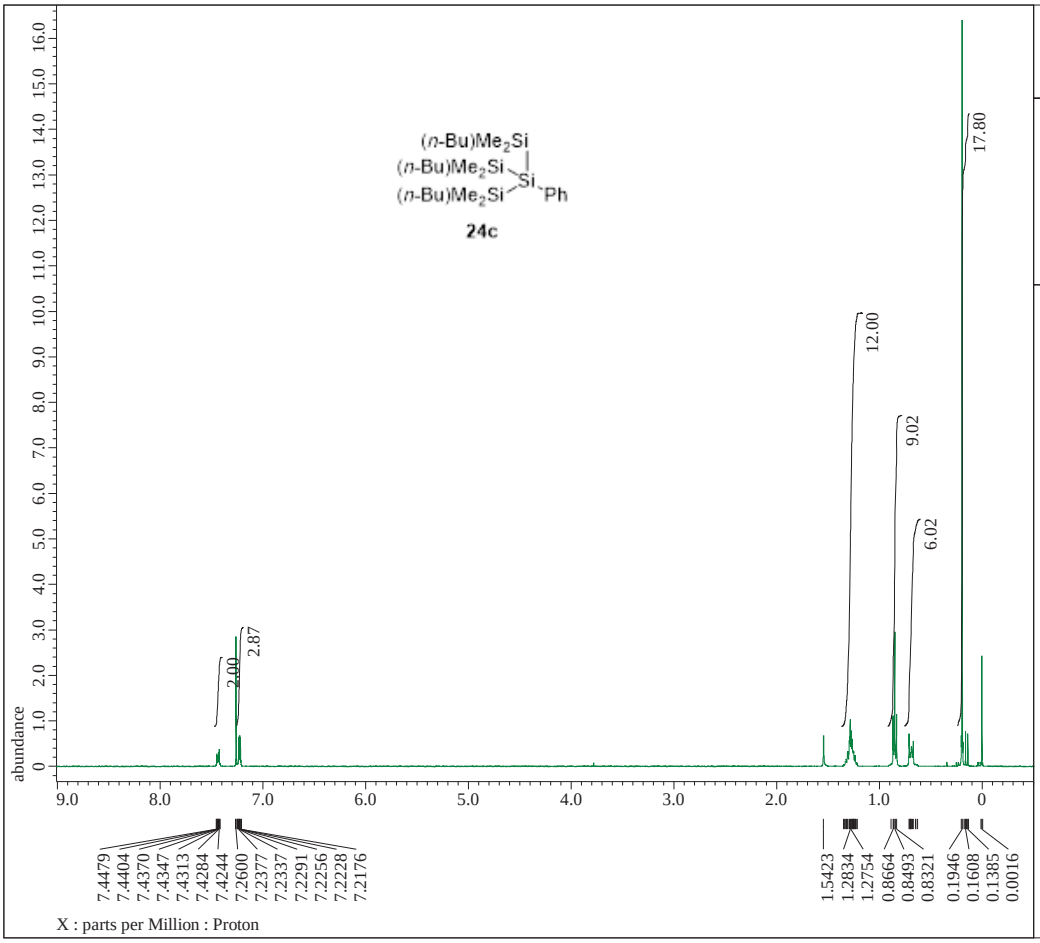
Derived from: wuan-1481_E13C-1-1.jdf
```

```
Filename          = wuan-1481_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1481
Solvent           = CHLOROFORM-D
Creation_Time     = 23-AUG-2022 13:51:39
Revision_Time     = 23-AUG-2022 14:06:31
Current_Time      = 23-AUG-2022 14:06:54

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 378
Total_Scans       = 378

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]
```



JEOL RESONANCE

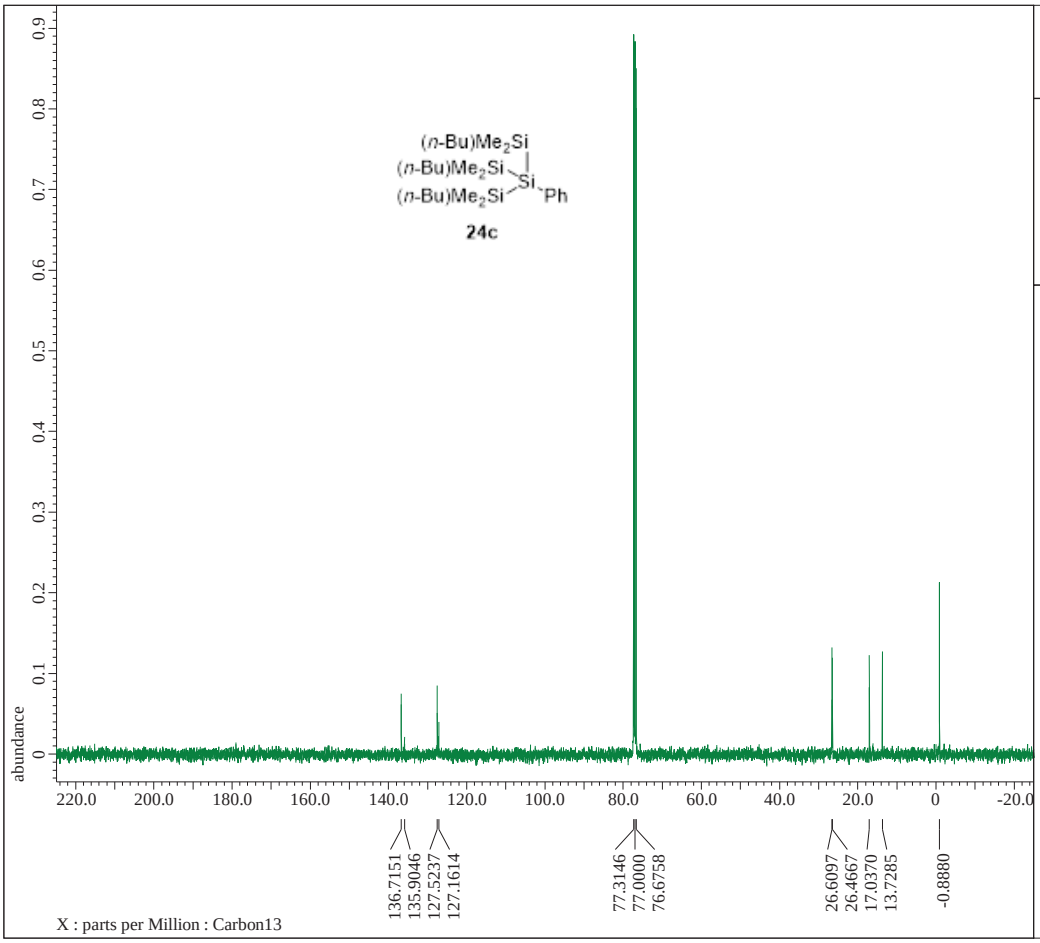
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1518_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1518
Solvent           = CHLOROFORM-D
Creation_Time     = 30-SEP-2022 14:16:46
Revision_Time     = 30-SEP-2022 14:16:23
Current_Time      = 17-JAN-2023 14:18:36

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        = 52
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
```



JEOL RESONANCE

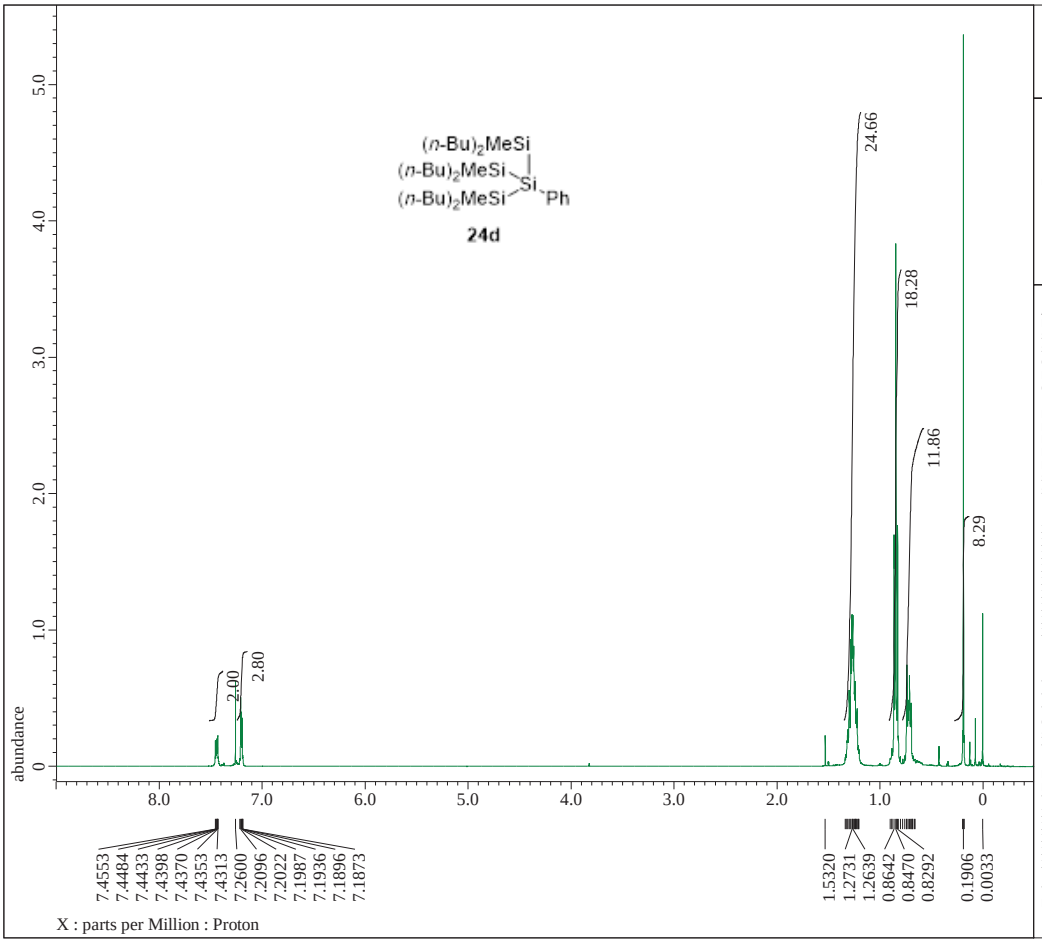
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1518_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1518
Solvent           = CHLOROFORM-D
Creation_Time     = 30-SEP-2022 14:30:00
Revision_Time     = 30-SEP-2022 14:40:21
Current_Time      = 17-JAN-2023 14:19:01

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 316
Total_Scans       = 316

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]
```



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

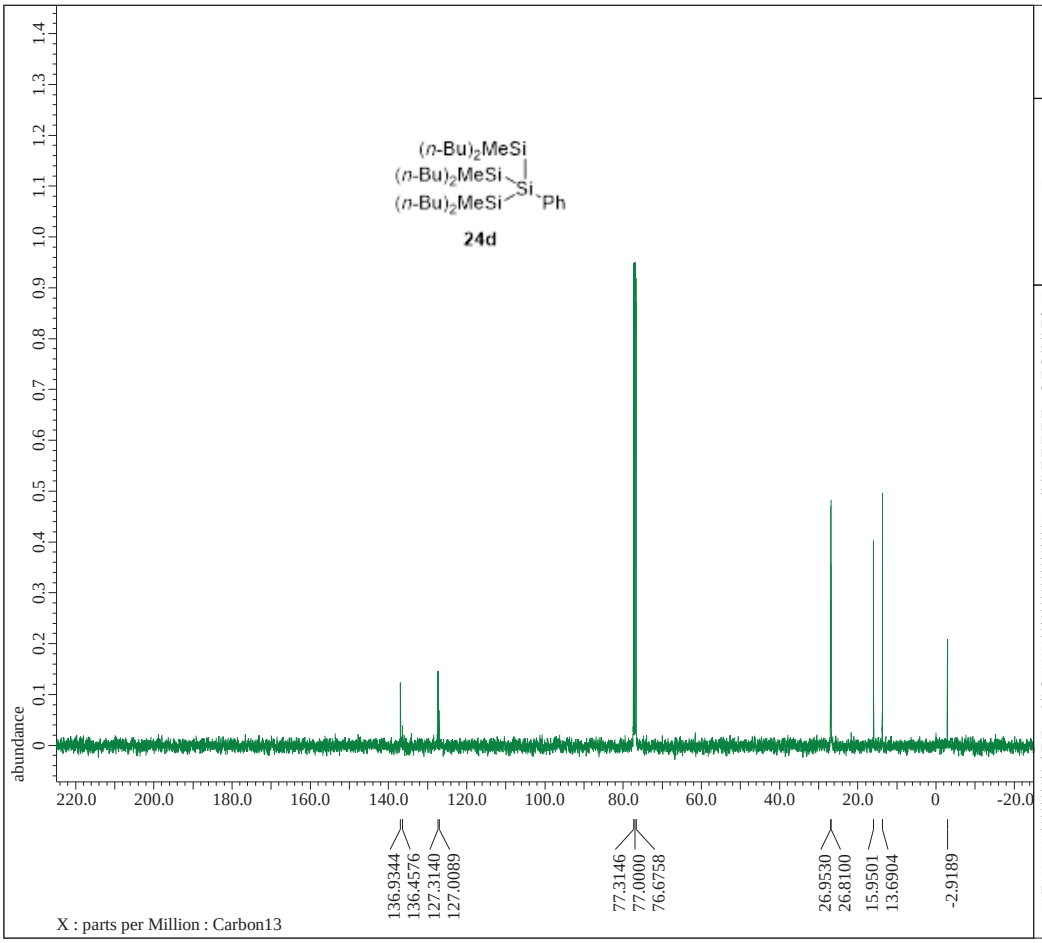
Derived from: wuan--1541_E1H-1-1.jdf
```

```
Filename          = wuan--1541_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan--1541
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 09:56:14
Revision_Time     = 17-JAN-2023 09:56:58
Current_Time      = 17-JAN-2023 09:57:36

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 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

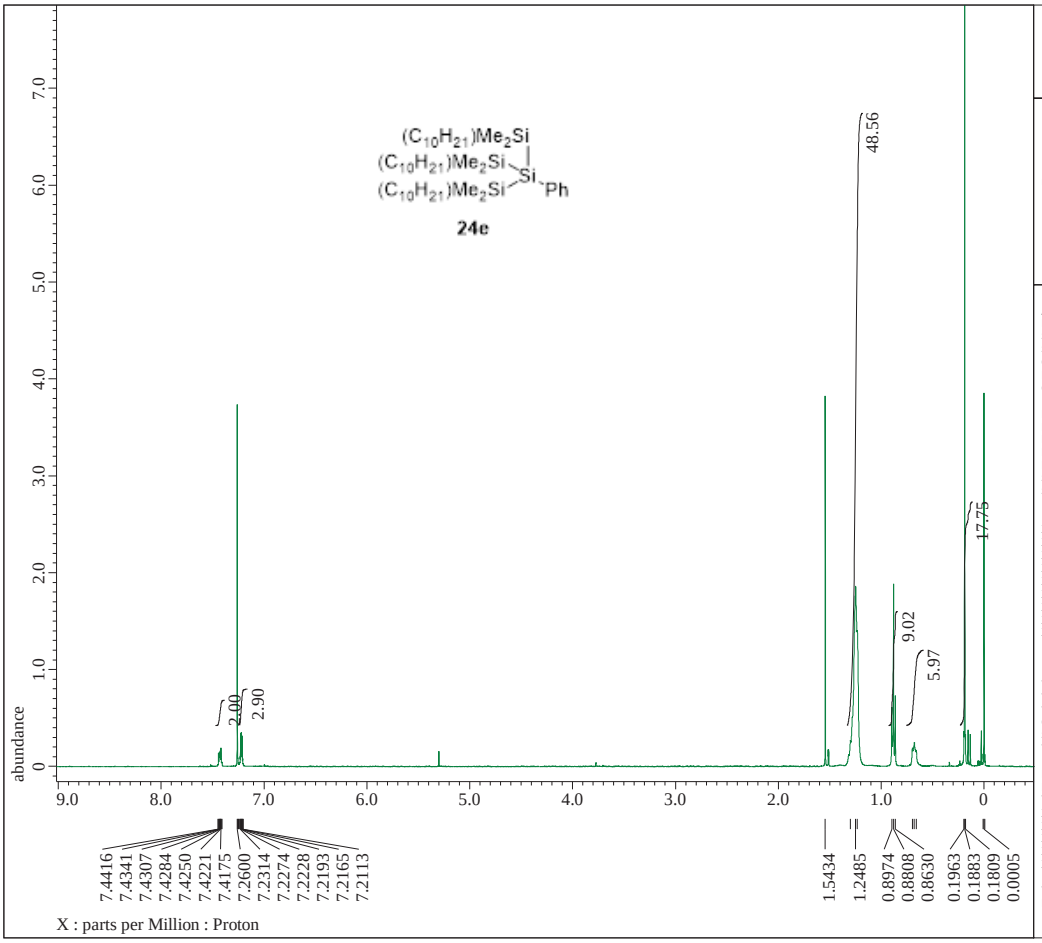
Derived from: wuan--1541_E13C-1-1.jdf
```

```
Filename          = wuan--1541_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan--1541
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 10:00:42
Revision_Time     = 17-JAN-2023 10:00:48
Current_Time      = 17-JAN-2023 10:01:20

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 99
Total_Scans       = 99

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



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

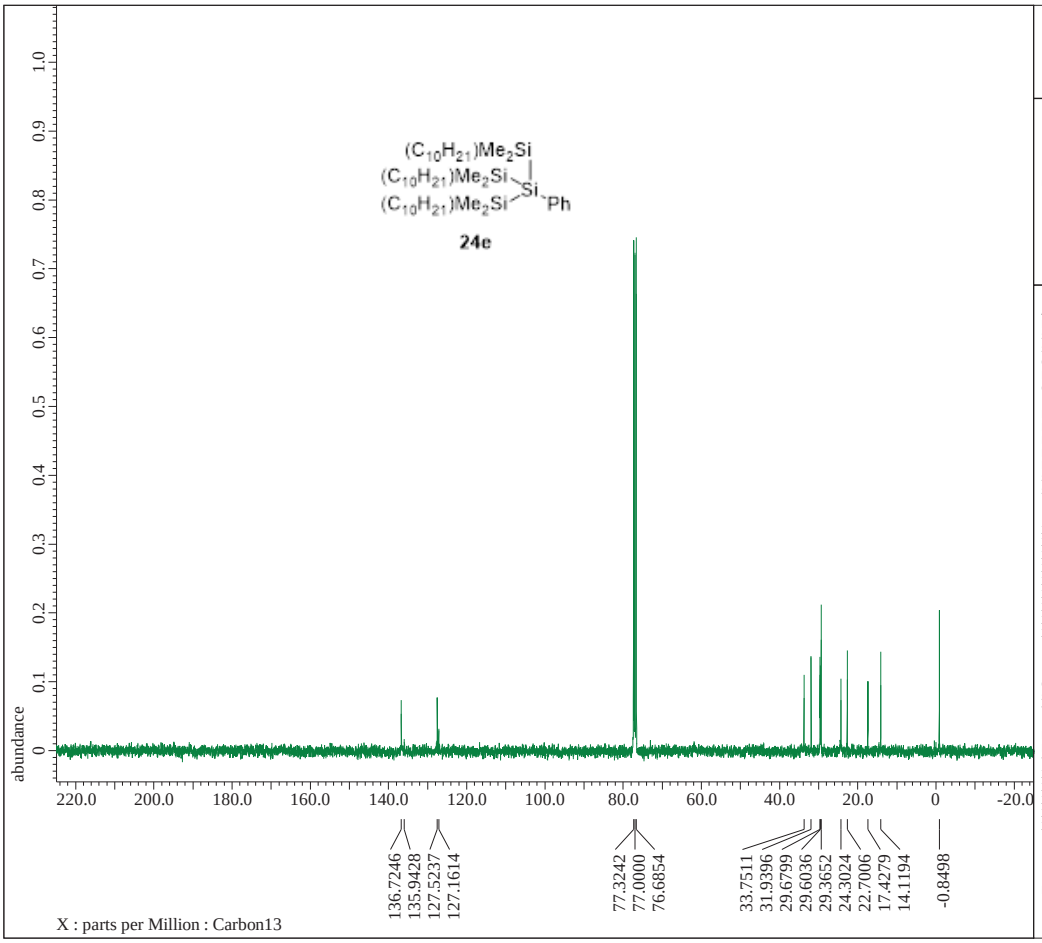
Derived from: wuan-1524_E1H-1-1.jdf
```

```
Filename          = wuan-1524_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1524
Solvent           = CHLOROFORM-D
Creation_Time     =  6-OCT-2022 16:30:54
Revision_Time     = 17-JAN-2023 13:04:04
Current_Time      = 17-JAN-2023 13:04:56

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        = 40
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
```



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

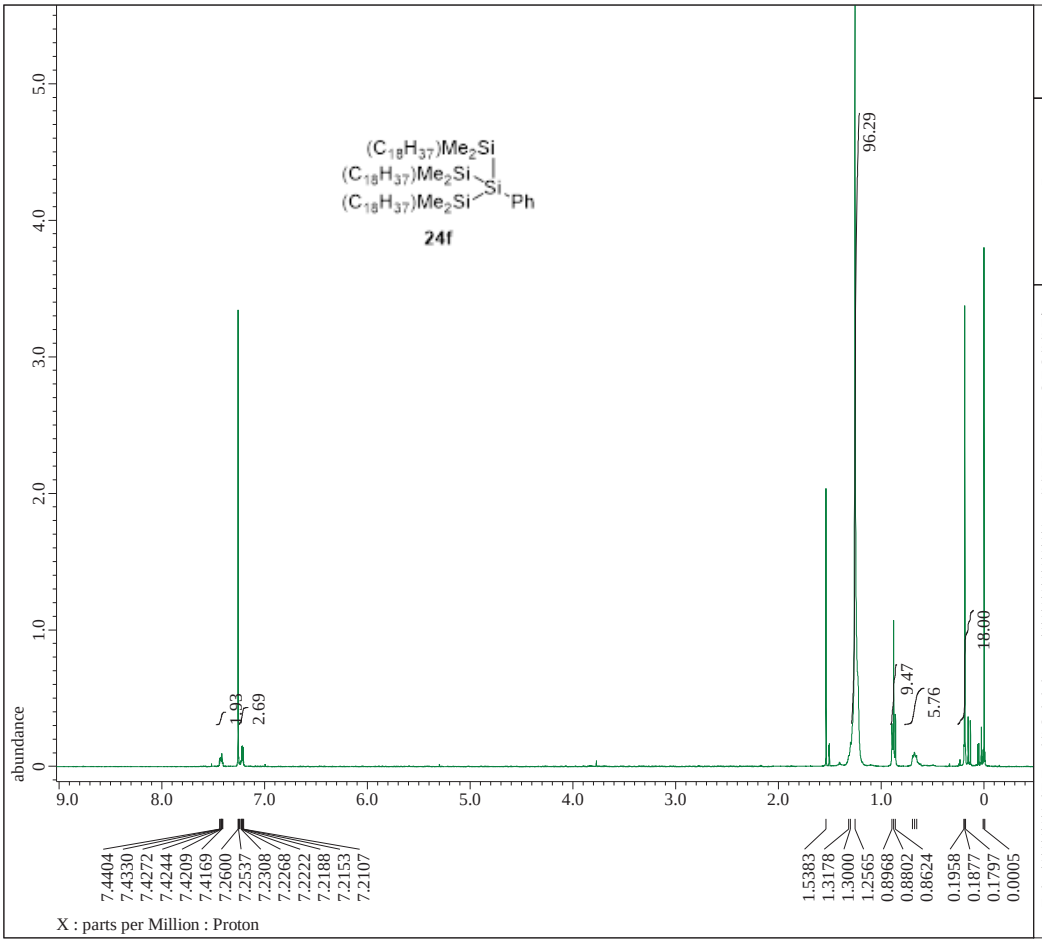
Derived from: wuan-1524_E13C-1-1.jdf
```

```
Filename          = wuan-1524_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1524
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 12:45:19
Revision_Time     = 17-JAN-2023 12:56:09
Current_Time      = 17-JAN-2023 12:56:49

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             = 274
Total_Scans       = 274

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

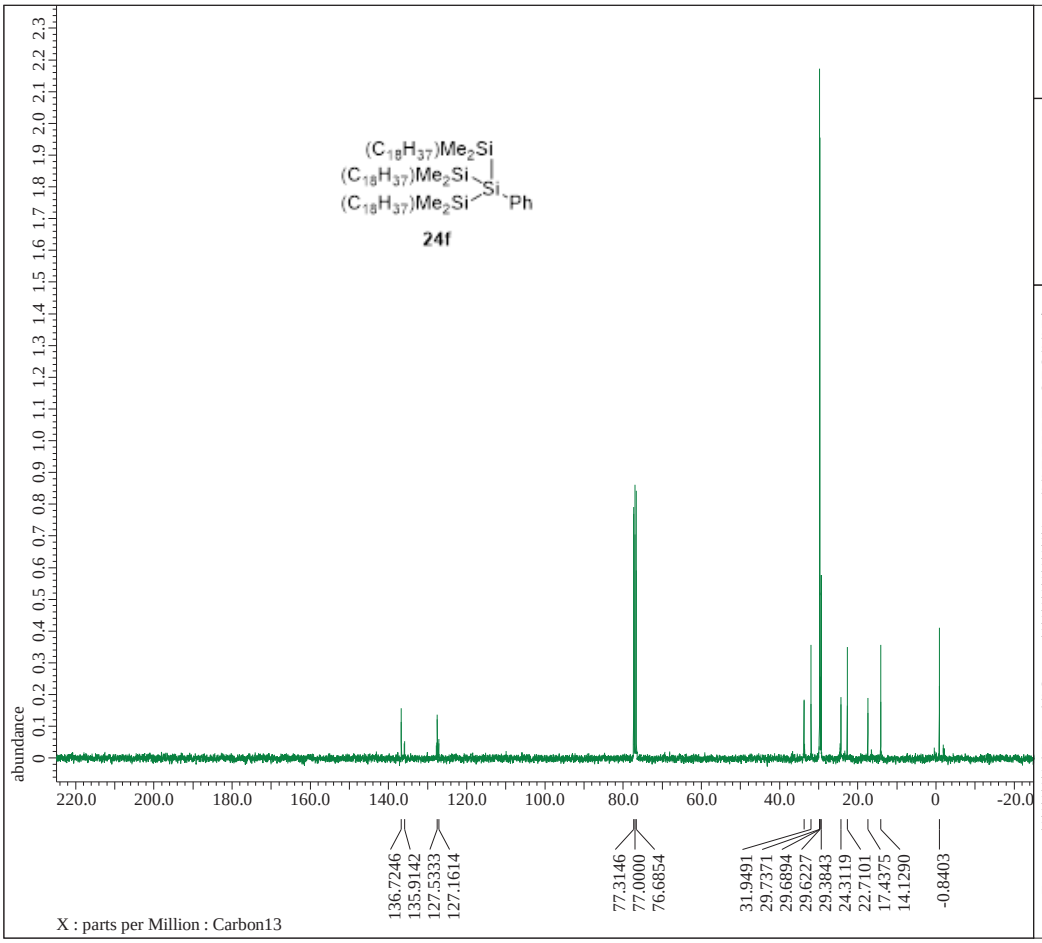
Derived from: wuan-1593_E1H-1-1.jdf
```

```
Filename          = wuan-1593_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1593
Solvent           = CHLOROFORM-D
Creation_Time     =  9-DEC-2022 12:36:12
Revision_Time     = 17-JAN-2023 13:35:13
Current_Time      = 17-JAN-2023 13:35:58

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
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

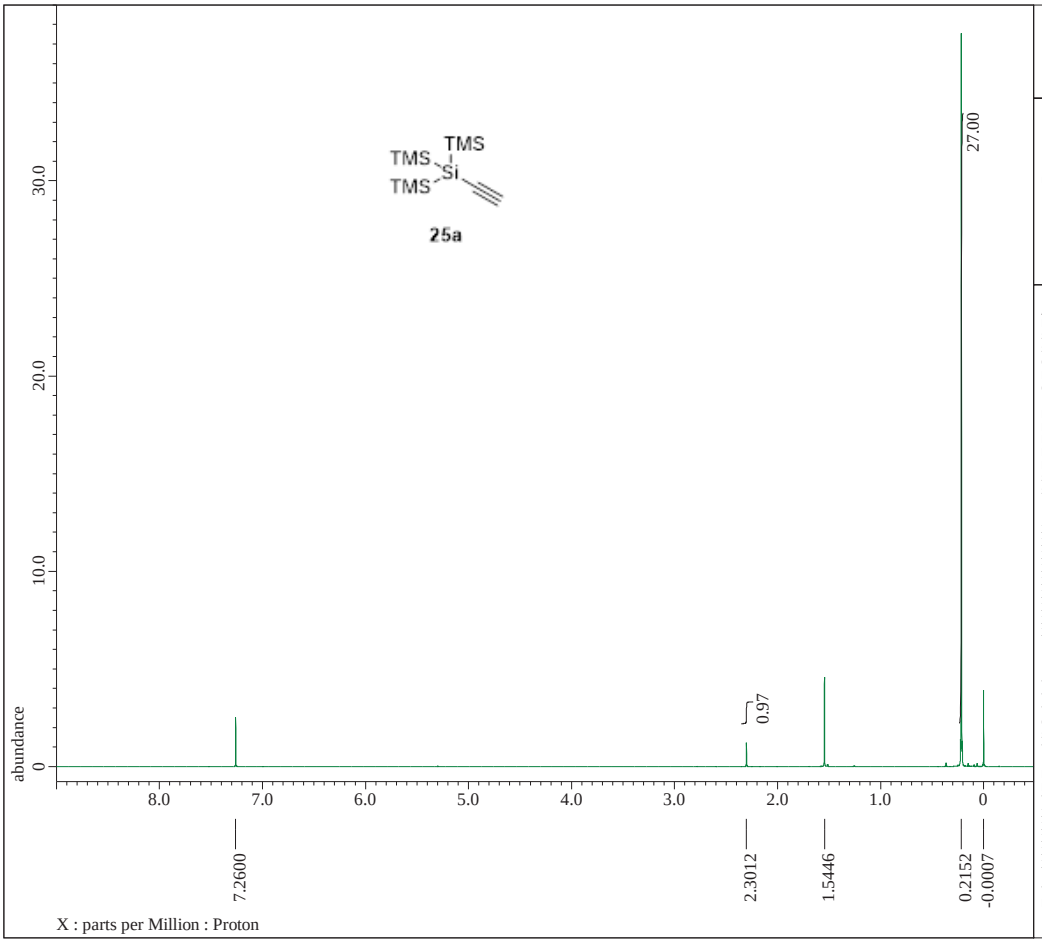
Derived from: wuan-1593_E13C-1-1.jdf
```

```
Filename          = wuan-1593_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1593
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 13:29:22
Revision_Time     = 17-JAN-2023 13:29:32
Current_Time      = 17-JAN-2023 13:29:57

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 118
Total_Scans       = 118

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]
```



JEOL RESONANCE

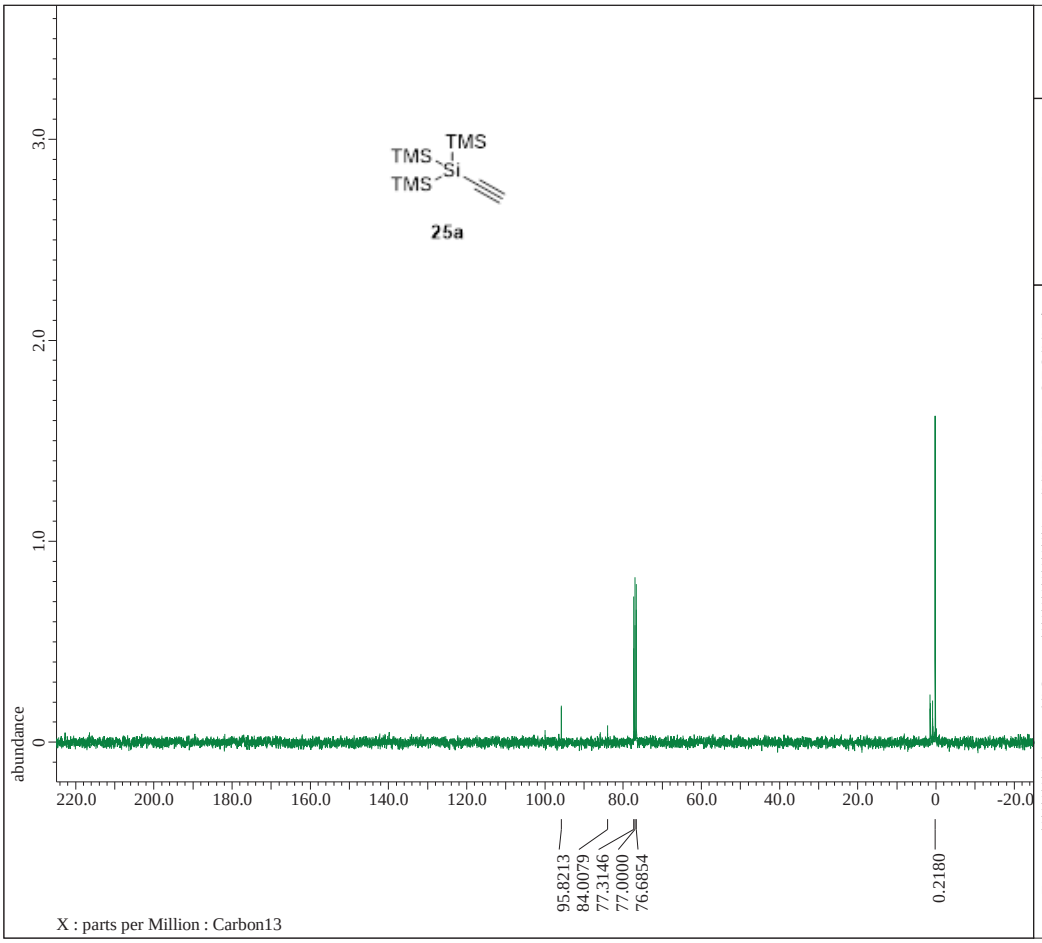
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1470_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1470
Solvent           = CHLOROFORM-D
Creation_Time     =  2-AUG-2022 12:53:24
Revision_Time     =  2-AUG-2022 12:55:39
Current_Time      = 17-JAN-2023 10:04:51

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
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
```



JEOL RESONANCE

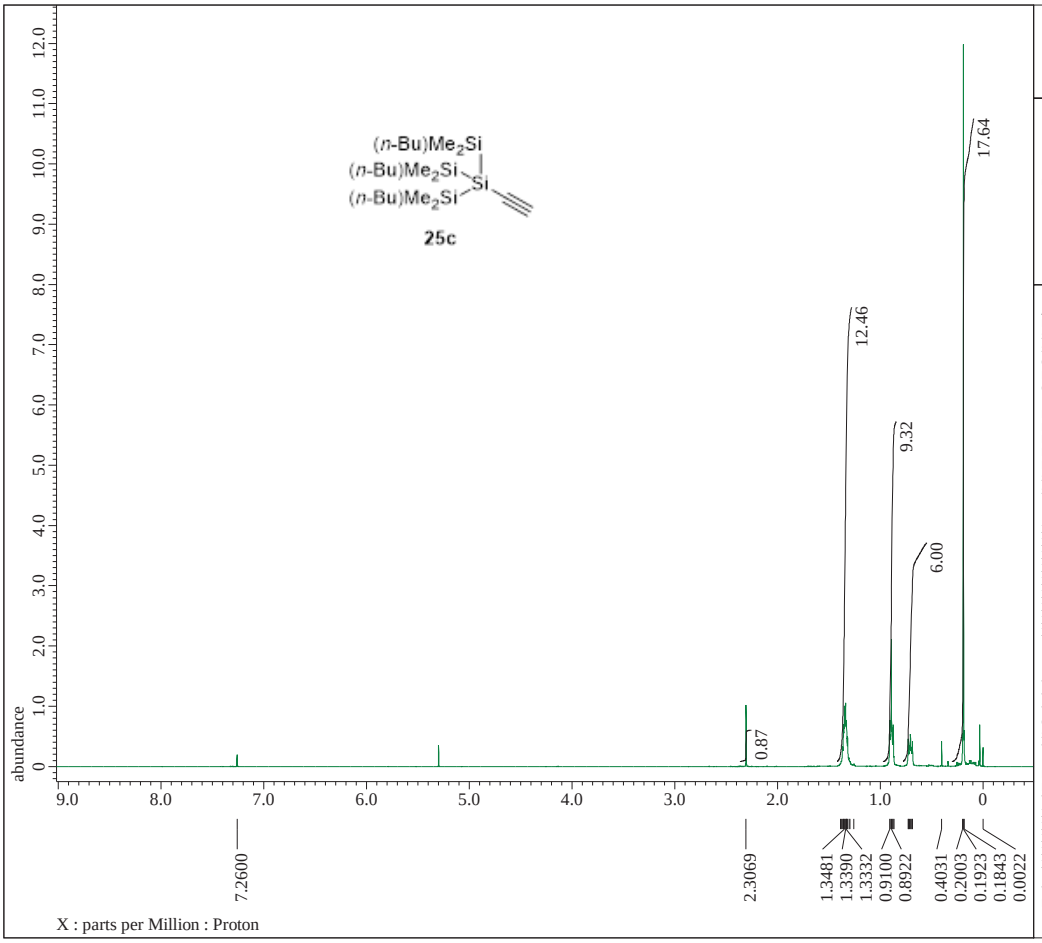
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1471_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1471
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 10:16:55
Revision_Time     = 17-JAN-2023 10:14:21
Current_Time      = 17-JAN-2023 12:30:53

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 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             = 26
Total_Scans       = 26

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




```
---- 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

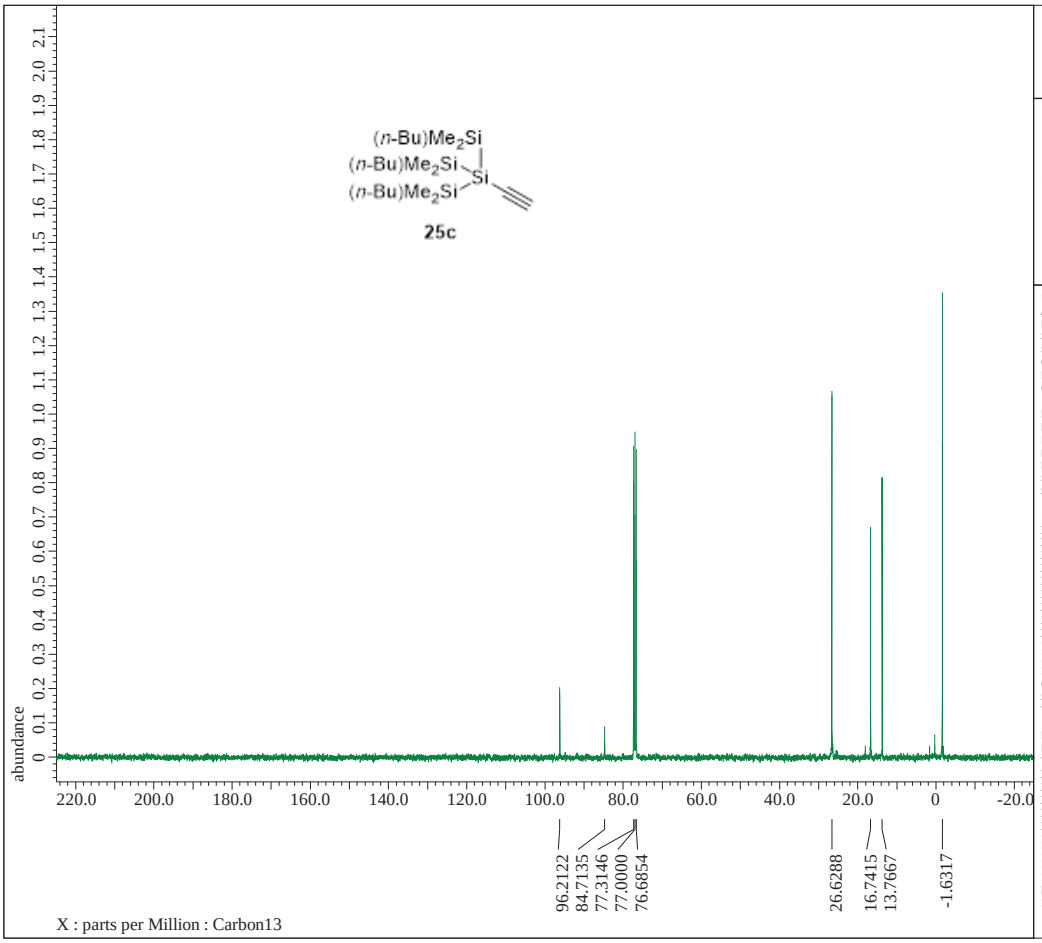
Derived from: wuan-1519_E1H-1-1.jdf
```

```
Filename          = wuan-1519_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan--1519
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 09:33:47
Revision_Time     = 17-JAN-2023 09:41:23
Current_Time      = 17-JAN-2023 09:41:55

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        = 22
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

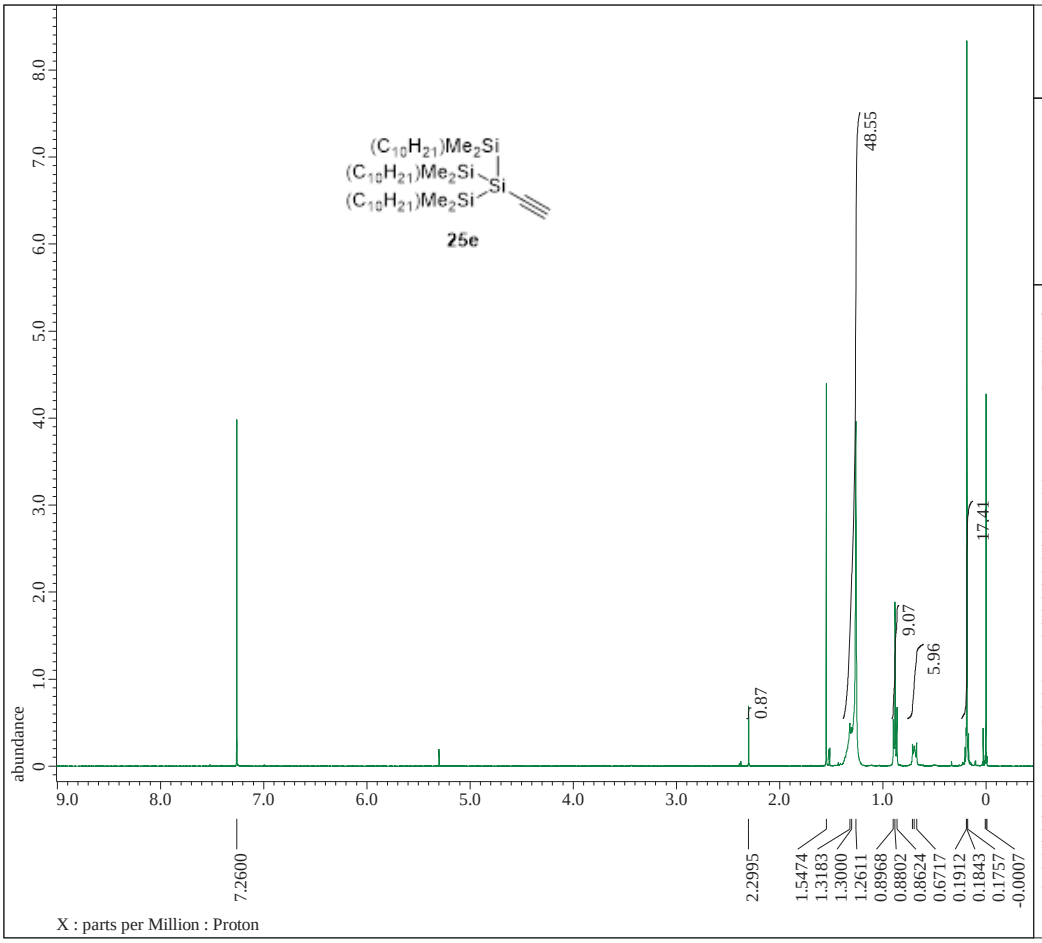
Derived from: wuan--1519_E13C-1-1.jdf
```

```
Filename          = wuan--1519_E13C-1-5.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan--1519
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 09:38:11
Revision_Time     = 17-JAN-2023 09:45:37
Current_Time      = 17-JAN-2023 09:46:28

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             = 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

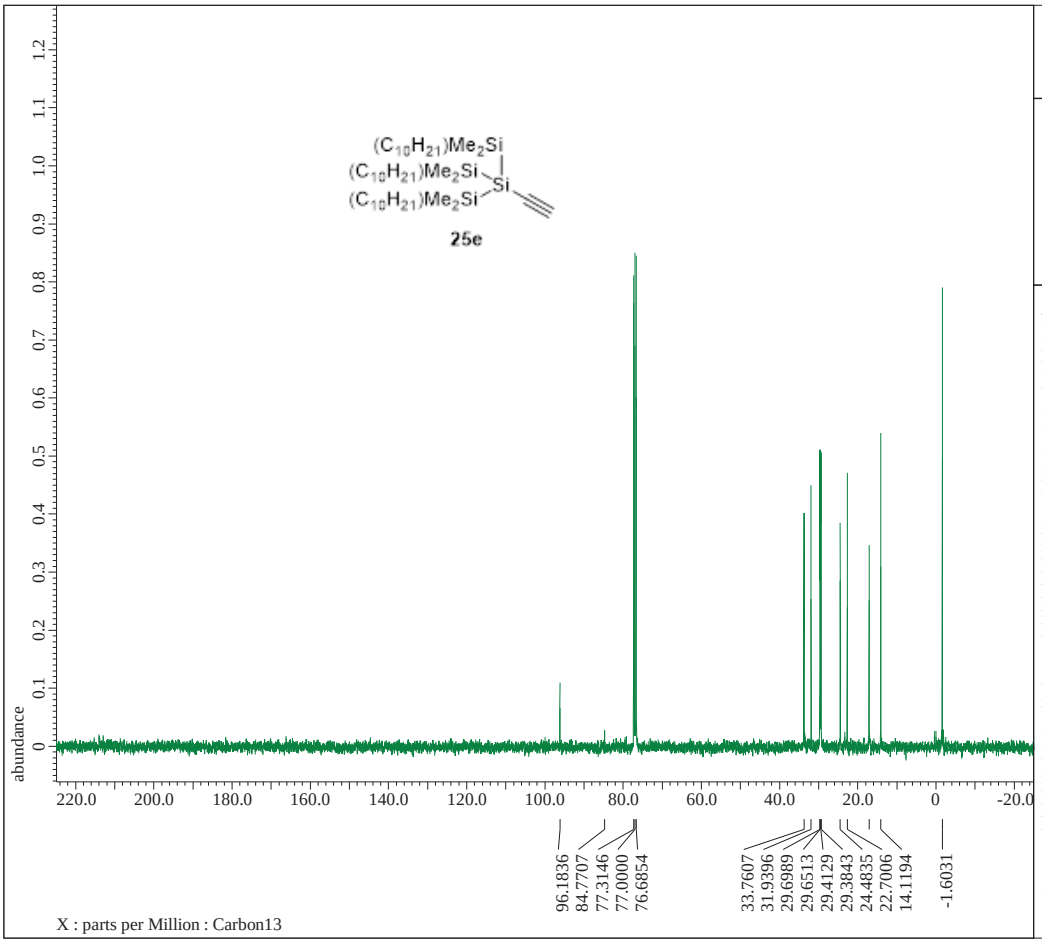
Derived from: wuan-1526_E1H-1-1.jdf
```

```
Filename          = wuan-1526_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1526
Solvent           = CHLOROFORM-D
Creation_Time     =  7-OCT-2022 13:13:49
Revision_Time     = 17-JAN-2023 13:14:31
Current_Time      = 17-JAN-2023 13:15:17

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 58
Temp_Get          = 23.3[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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

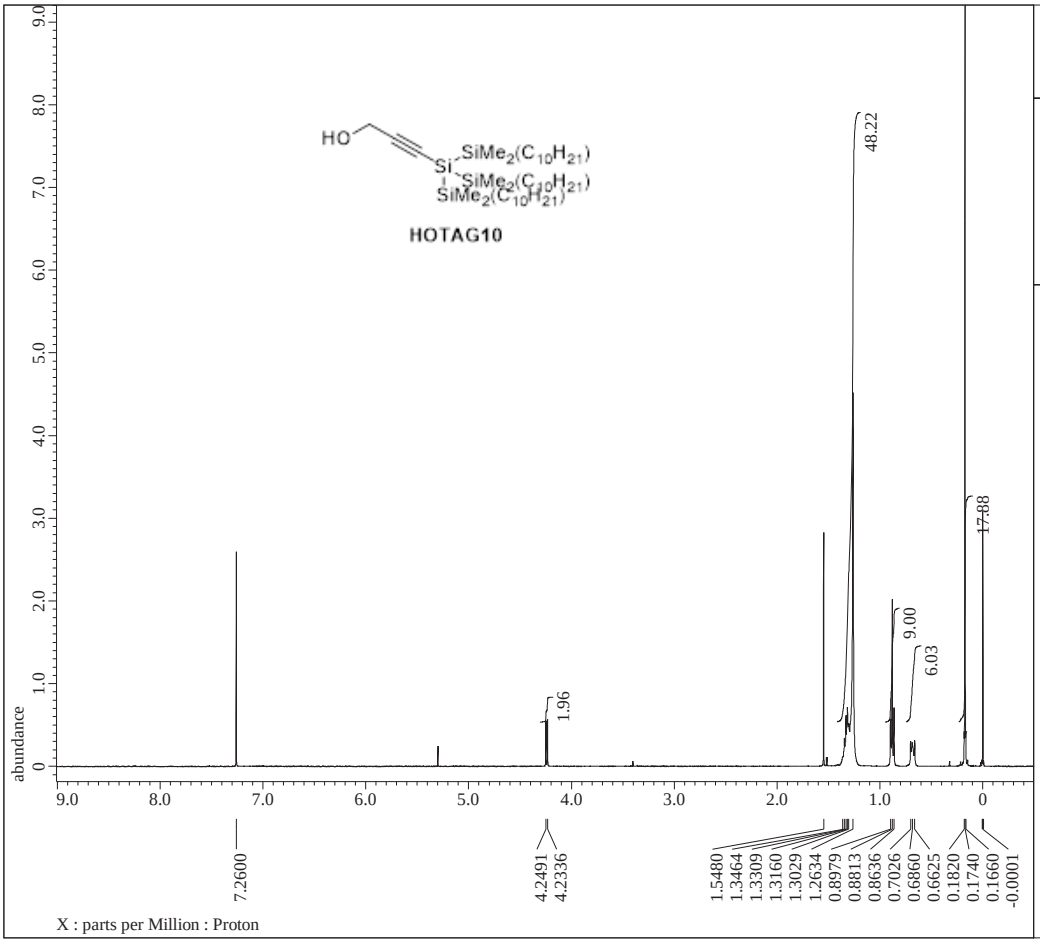
Derived from: wuan-1526_E13C-1-1.jdf
```

```
Filename          = wuan-1526_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1526
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2023 13:09:06
Revision_Time     = 17-JAN-2023 13:16:11
Current_Time      = 17-JAN-2023 13:17:20

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 189
Total_Scans       = 189

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.4[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]
```



JEOL RESONANCE

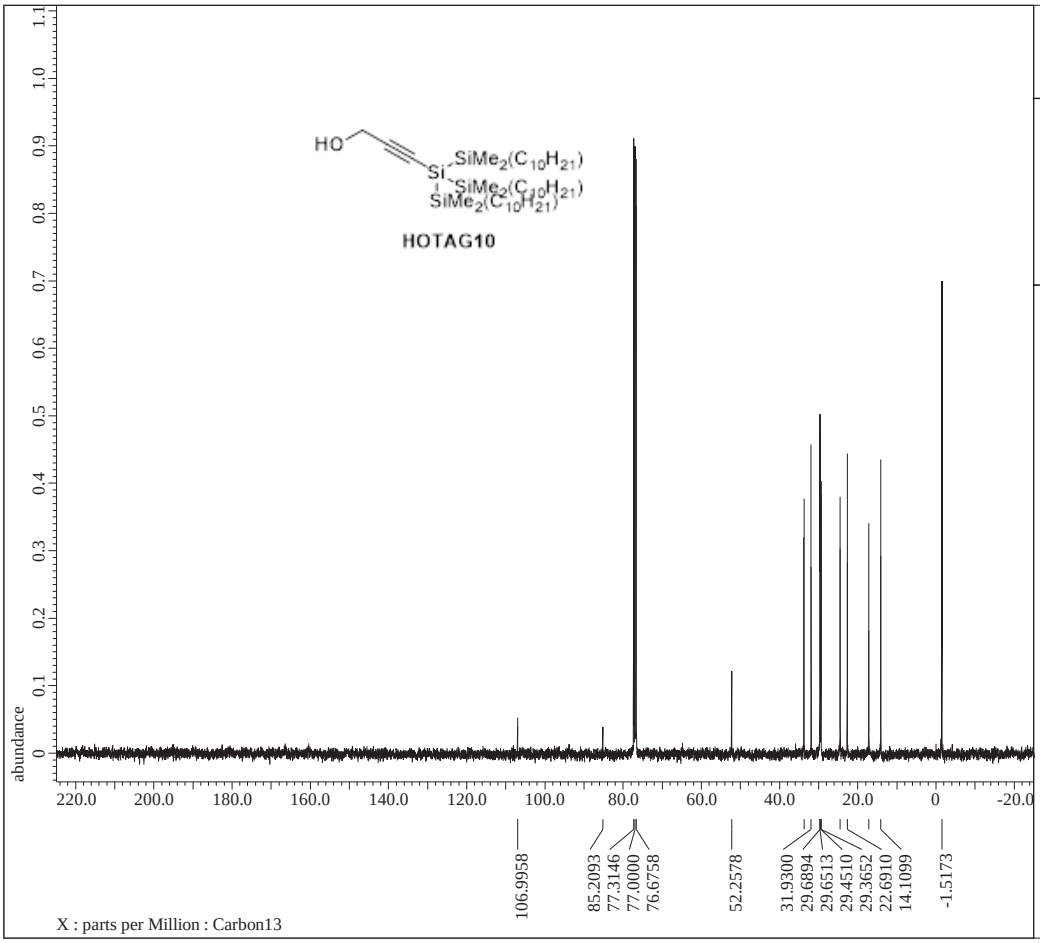
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = wuan-1530_E1H-1-6.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1530
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2022 16:19:03
Revision_Time     = 12-OCT-2022 16:22:35
Current_Time      = 13-OCT-2022 13:01:48

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        = 38
Temp_Get          = 25.1[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

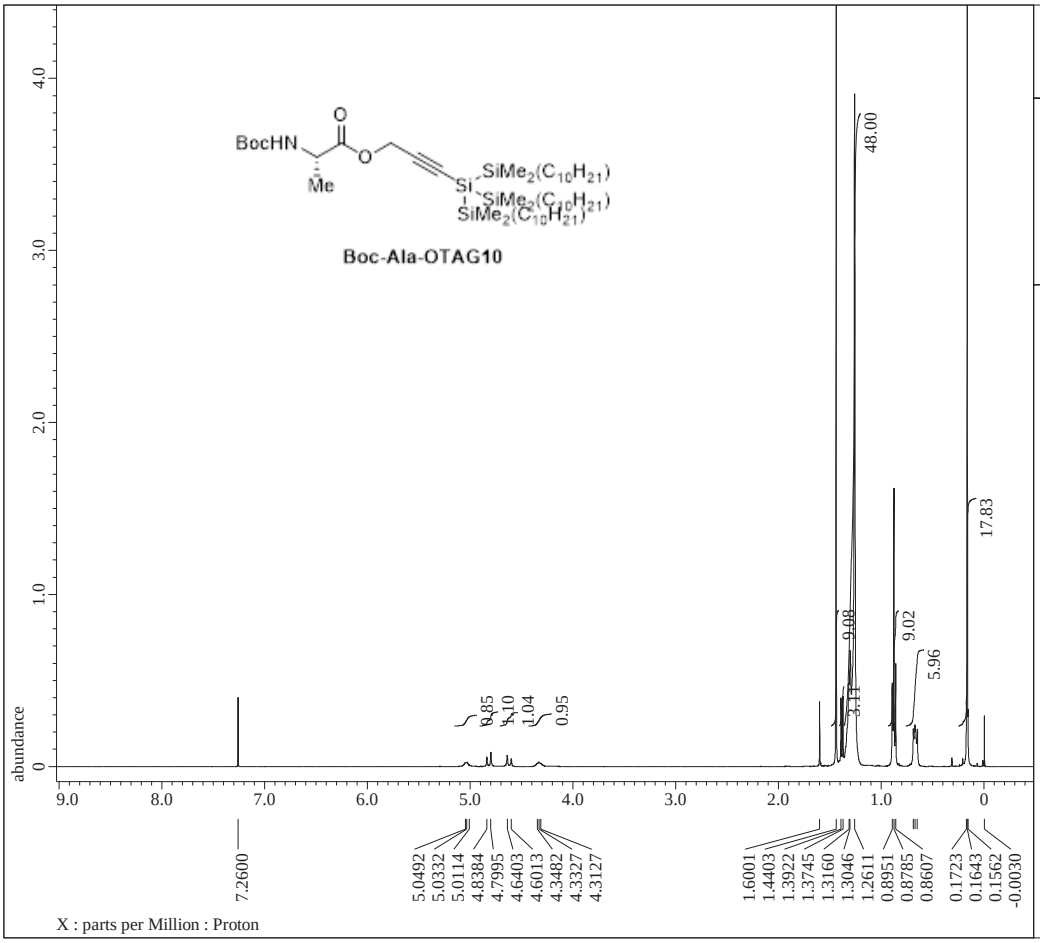
Derived from: wuan-1530_E13C-1-1.jdf
```

```
Filename          = wuan-1530_E13C-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1530
Solvent           = CHLOROFORM-D
Creation_Time     = 13-OCT-2022 13:04:00
Revision_Time     = 13-OCT-2022 13:12:01
Current_Time      = 13-OCT-2022 13:12:21

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             = 283
Total_Scans       = 283

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]
```



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

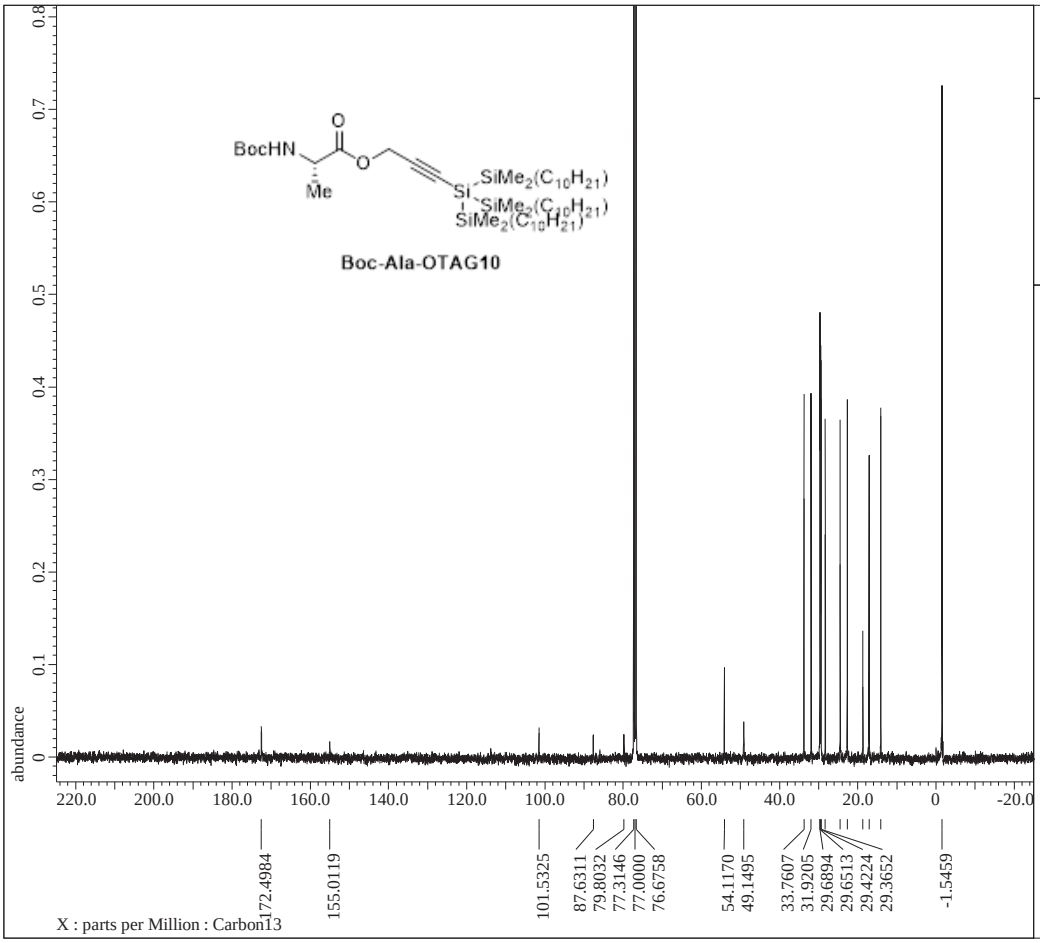
Derived from: wuan-1531-2_E1H-1-1.jdf
```

```
Filename          = wuan-1531-2_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1531-2
Solvent           = CHLOROFORM-D
Creation_Time     = 14-OCT-2022 13:46:06
Revision_Time     = 14-OCT-2022 13:48:08
Current_Time      = 14-OCT-2022 13:49:47

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

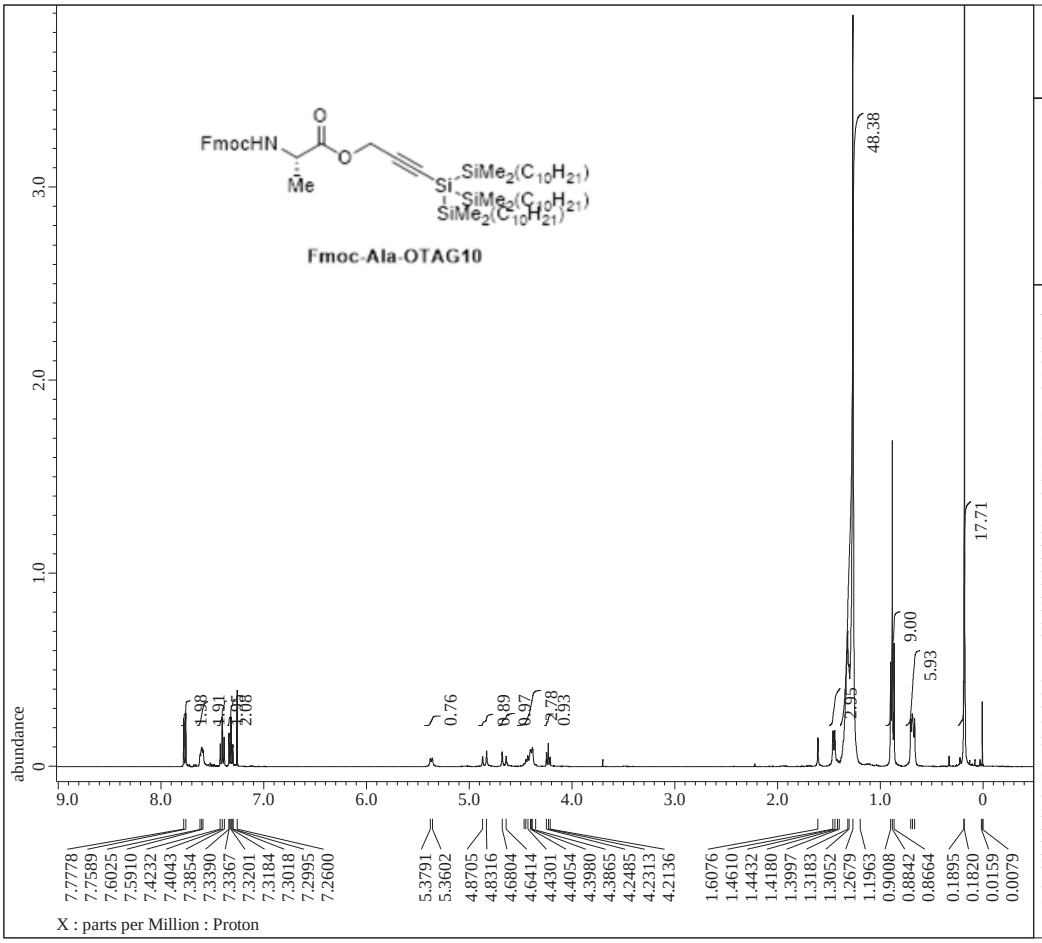
Derived from: wuan-1531-2_E13C-1-1.jdf
```

```
Filename          = wuan-1531-2_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1531-2
Solvent           = CHLOROFORM-D
Creation_Time     = 14-OCT-2022 13:49:54
Revision_Time     = 14-OCT-2022 14:16:08
Current_Time      = 14-OCT-2022 14:16:42

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 635
Total_Scans       = 635

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



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

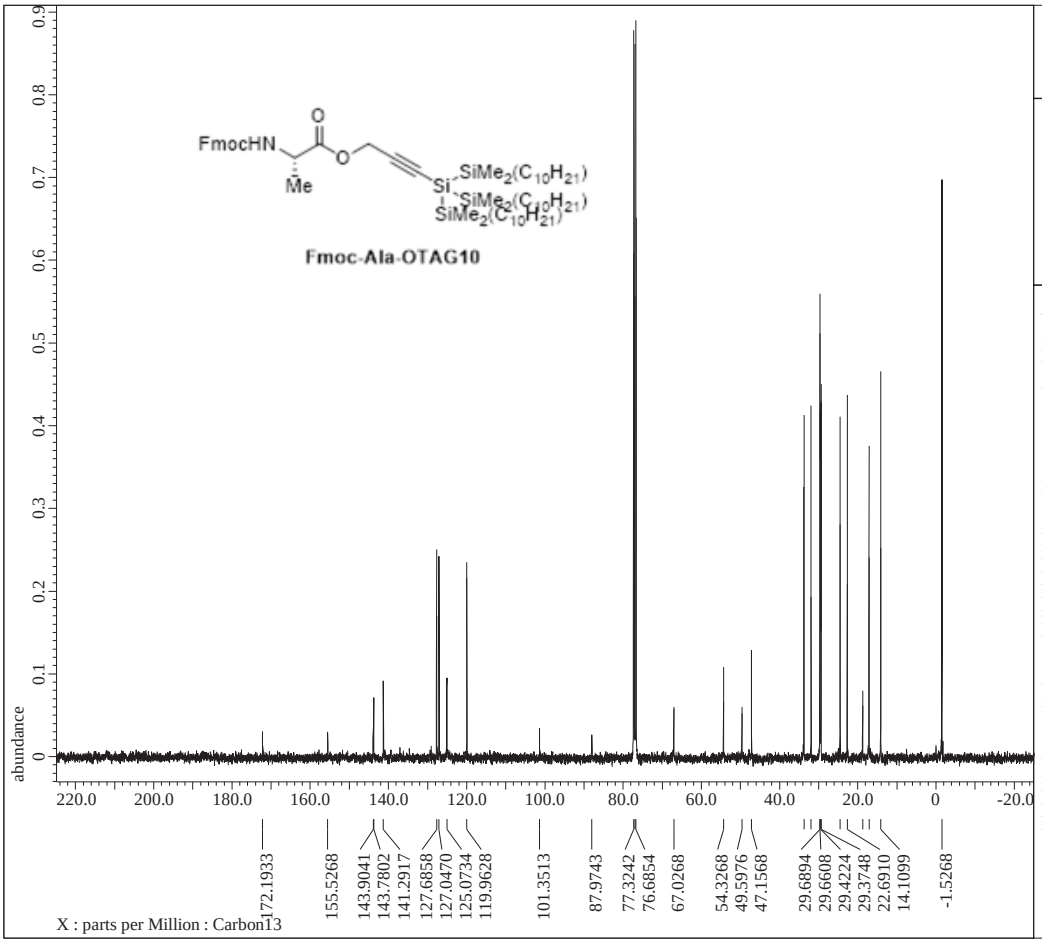
Derived from: wuan-1532_E1H-1-1.jdf
```

```
Filename          = wuan-1532_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1532
Solvent           = CHLOROFORM-D
Creation_Time     = 13-OCT-2022 15:22:43
Revision_Time     = 13-OCT-2022 15:26:27
Current_Time      = 13-OCT-2022 15:27:27

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 24.1[dC]
X_90_Width        = 6.47[us]
X_Acq_Time        = 4.36731904[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.235[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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

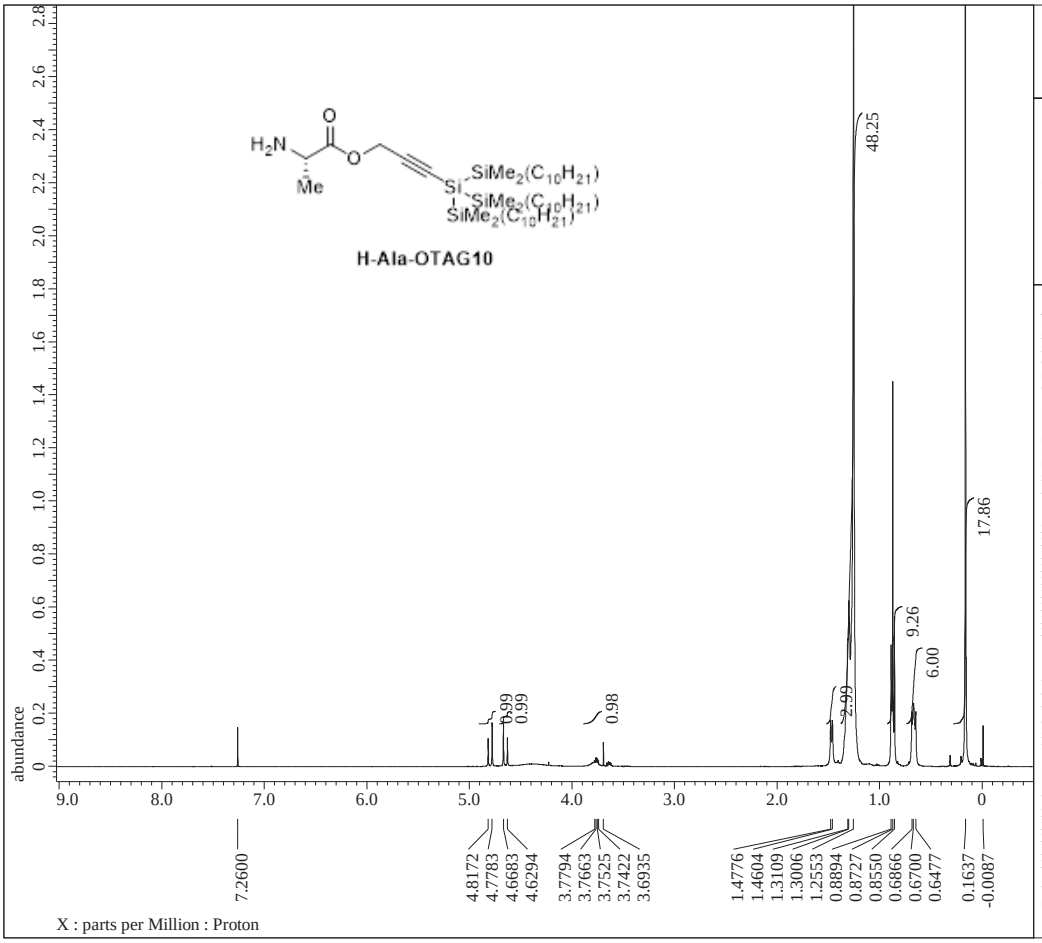
Derived from: wuan-1532_E13C-1-1.jdf
```

```
Filename          = wuan-1532_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1532
Solvent           = CHLOROFORM-D
Creation_Time     = 13-OCT-2022 15:26:39
Revision_Time     = 13-OCT-2022 15:51:47
Current_Time      = 13-OCT-2022 15:52:01

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 551
Total_Scans       = 551

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

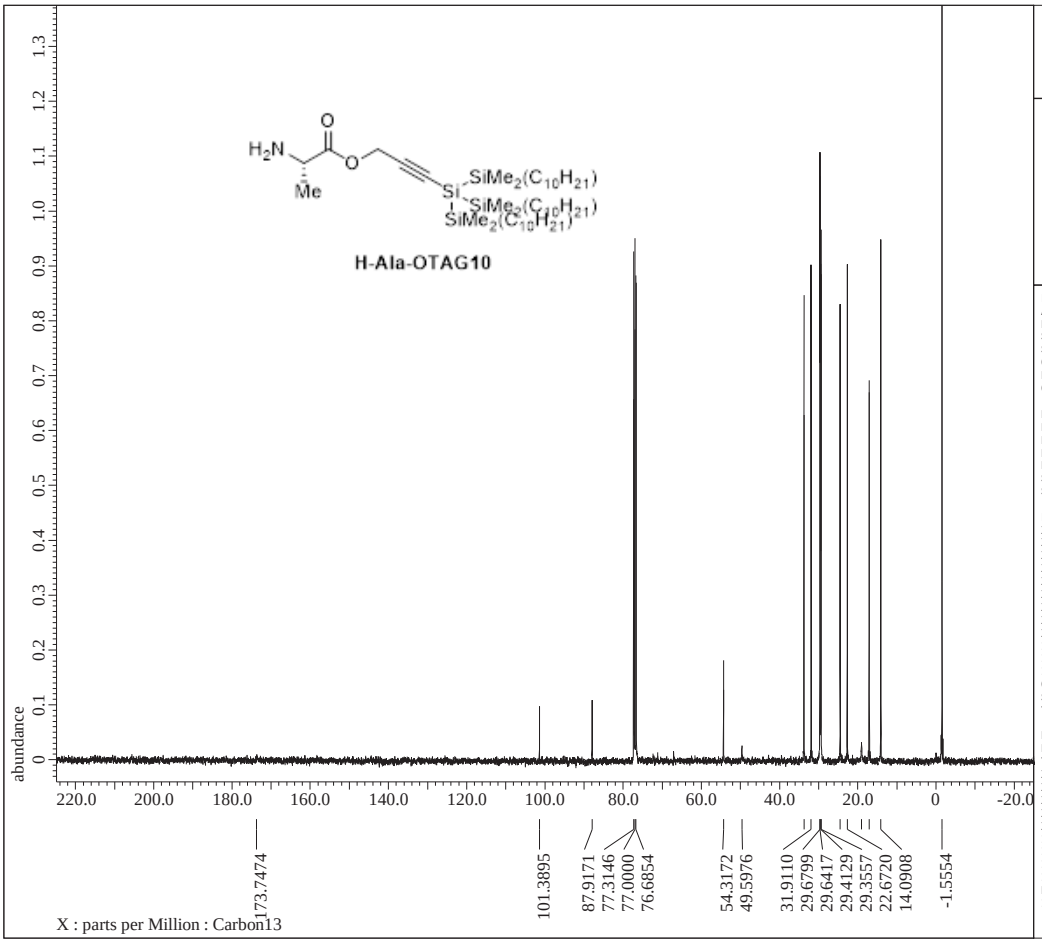
Derived from: wuan-1533B_E1H-1-1.jdf
```

```
Filename          = wuan-1533B_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1533B
Solvent           = CHLOROFORM-D
Creation_Time     = 17-OCT-2022 14:42:14
Revision_Time     = 17-OCT-2022 14:42:55
Current_Time      = 17-OCT-2022 14:43:35

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        = 16
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
```



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

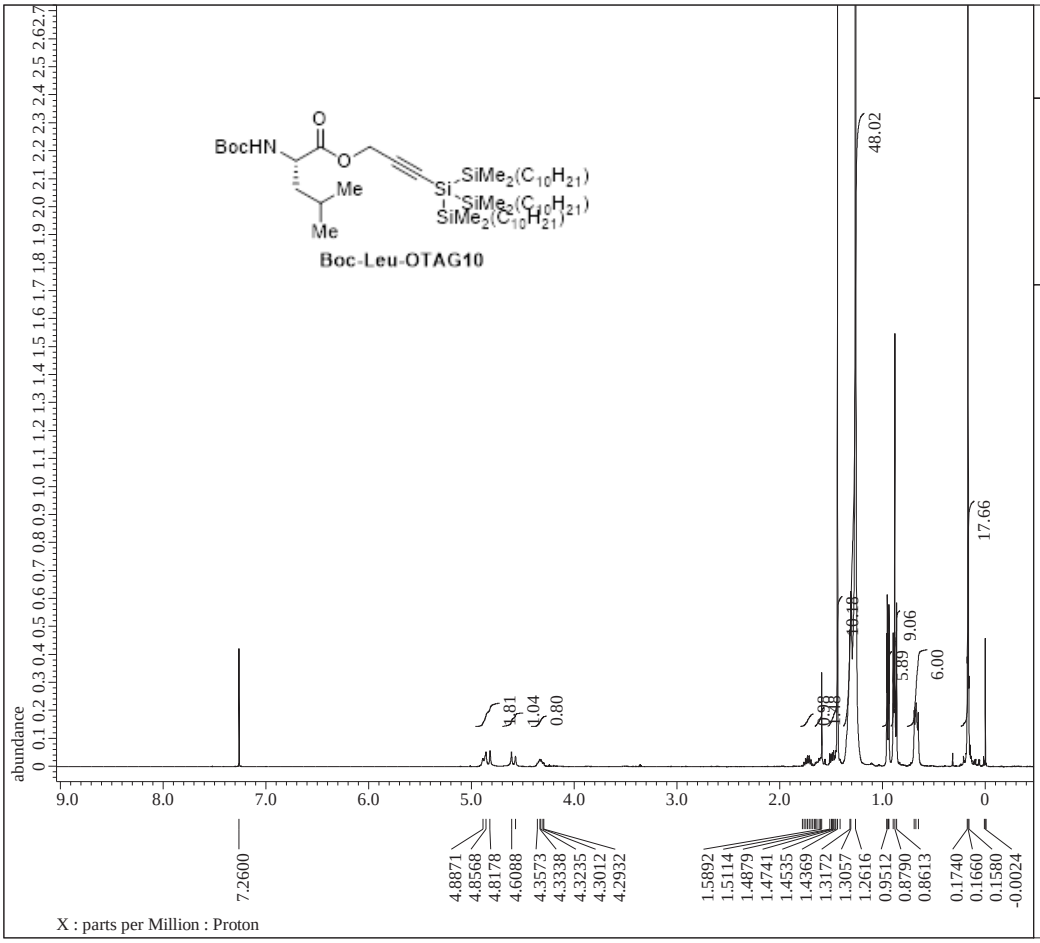
Derived from: wuan-1533B_E13C-1-1.jdf
```

```
Filename          = wuan-1533B_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1533B
Solvent           = CHLOROFORM-D
Creation_Time     = 17-OCT-2022 14:46:54
Revision_Time     = 17-OCT-2022 15:06:40
Current_Time      = 17-OCT-2022 15:07:01

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

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 502.0
Total_Scans       = 502.0

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



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

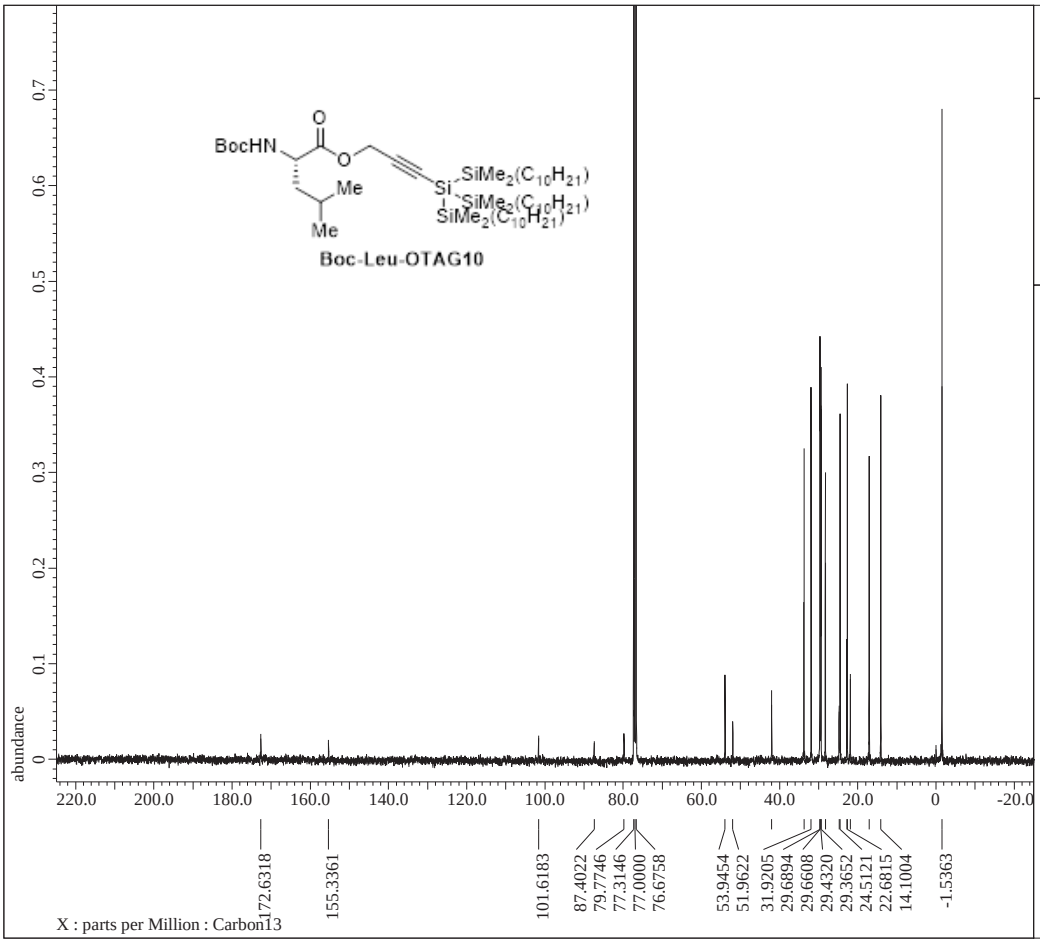
Derived from: wuan-1548_E1H-1-1.jdf
```

```
Filename          = wuan-1548_E1H-1-5.jdf
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = wuan-1548
Solvent           = CHLOROFORM-D
Creation_Time     =  1-NOV-2022 16:44:23
Revision_Time     =  1-NOV-2022 16:50:38
Current_Time      =  1-NOV-2022 16:52:27

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          = 24.2[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
```



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-1548_E13C-1-1.jdf
```

```
Filename          = wuan-1548_E13C-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = wuan-1548
Solvent           = CHLOROFORM-D
Creation_Time     =  1-NOV-2022 16:49:05
Revision_Time     =  1-NOV-2022 17:36:36
Current_Time      =  1-NOV-2022 17:37:48

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             = 1047
Total_Scans       = 1047

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]
```