Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Synthesis of allenynes via Pd-catalyzed coupling of 1,4-diyn-3-yl carbonates with boronic acids

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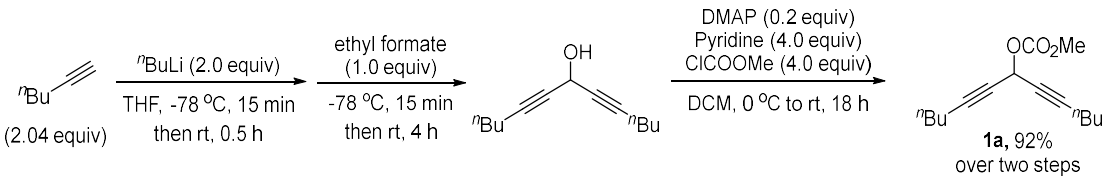
General Information. NMR spectra were taken with Bruker Avance III spectrometer (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR, 376 MHz for ^{19}F NMR) in $CDCl_3$. All ^{1}H NMR experiments were measured with tetramethylsilane (0 ppm) in $CDCl_3$ as the internal reference; ^{13}C NMR experiments were measured in relative to the signal of $CDCl_3$ (77.0 ppm); ^{19}F NMR experiments were measured in relative to the signal of $CFCl_3$ (0 ppm) in $CDCl_3$. All reactions were carried out in Schlenk tubes. ^{n}BuLi (2.5 M in hexane) was purchased from Infinity Scientific; $ClCO_2Me$ was purchased from Sinopharm Chemical Reagent Co., Ltd.; $Pd_2(dba)_3$•$CHCl_3$ was purchased from Strem Chemicals Inc.; $Pd(dba)_2$ was purchased from J&K; S-Phos was purchased from Bide Pharmatech Ltd.; Petroleum ether (b.p. 60-90ºC), ethyl acetate, and dichloromethane were purchased from Shanghai Titan Scientific Co., Ltd. THF and Dioxane were dried over sodium wire with benzophenone as the indicator and distilled freshly before use. All the temperatures are referred to the oil baths used. Recoveries of substrates were determined by ^{1}H NMR analysis using dibromomethane as the internal standard. 300-400 mesh silica gel purchased from Shanghai Heqi Glassware Co., Ltd. was used for column chromatography. The crude allenyne product was dissolved in petroleum ether and loaded to the silica gel column for the column chromatography purification. Due to the allenyne products are not very stable, the temperature of water bath should lower than 30 ºC during concentration and the whole work-up process should be as quick as possible.

Experimental details and analytical data

Synthesis of starting materials

(1) Preparation of trideca-5,8-diyn-7-yl methyl carbonate (1a, wj-2-021, wj-3-103)

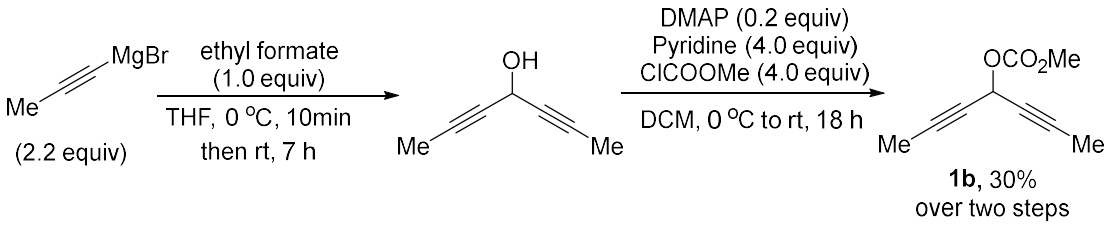


Typical Procedure I:[1] To a stirred solution of 1-hexyne (11.6 mL, d = 0.72 g/mL, 8.352 g, 102.0 mmol) in THF (60 mL) was added ^{n}BuLi (40.0 mL, 100.0 mmol, 2.5 M in hexane) dropwise over 15 min at -78 °C under Ar atmosphere. The resulting mixture was stirred for 30 min at room temperature and cooled to -78 °C. A solution of ethyl formate (4.0 mL, d = 0.921 g/mL, 3.684 g, 50.0 mmol) in THF (50 mL) was added dropwise over 15 min, and the resulting mixture was stirred at room temperature for 4 h as monitored by TLC. The reaction was quenched with water (30 mL) and extracted with ethyl acetate (30 mL x 3). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated. The crude alcohol product was directly used for next step without further purification.

Typical Procedure II:[2] To a stirred solution of DMAP (1.2224 g, 10.0 mmol) and the crude alcohol prepared above in DCM (100 mL) were added pyridine (16.1 mL, d = 0.983 g/mL, 15.8263 g, 200.0 mmol) and methyl chloroformate (15.5 mL, d = 1.22 g/mL, 18.91 g, 200.0 mmol) sequentially at 0 °C via an ice-water bath under Ar atmosphere. The resulting mixture was warmed up to room temperature, stirred at room temperature for 18 h as monitored by TLC, diluted with DCM (40 mL), washed sequentially with 1 M HCl (aq.) (50 mL x 3), and extracted with DCM (40 mL x 3). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated. The resulting residue was purified by column chromatography on silica gel to afford **1a** (11.4324 g, 92% over two steps) [eluent: petroleum ether / ethyl acetate = 40:1 (410 mL) to 30:1 (1230 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.92-5.87 (m, 1 H, CH), 3.82 (s, 3 H, CH_3), 2.24 (t, *J* = 6.8 Hz, 4 H,

2 x CH_2), 1.59-1.47 (m, 4 H, CH_2), 1.47-1.31 (m, 4 H, CH_2), 0.91 (t, J = 7.2 Hz, 6 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.5, 86.9, 74.1, 57.9, 55.0, 30.1, 21.8, 18.4, 13.5; **IR** (neat): v = 2958, 2934, 2265, 2239, 1751, 1441, 1320, 1309, 1247, 1170, 1123, 1045 cm^{-1}; **MS** (70 eV, EI) *m/z*: 250 (M^+, 13.35), 91 (100); **HRMS** (EI) calcd for $C_{15}H_{22}O_3$ [M^+]: 250.1563, found: 250.1566.

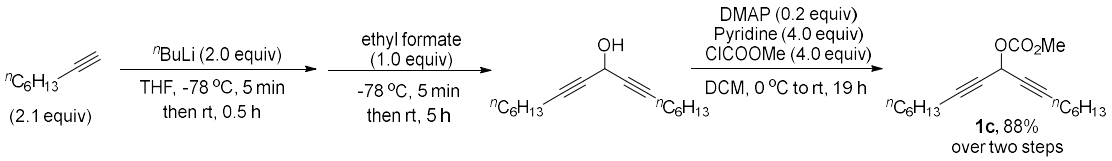
(2) Synthesis of hepta-2,5-diyn-4-yl methyl carbonate (1b, wj-3-104)



Step 1: To a stirred solution of ethyl formate (0.8 mL, d = 0.921 g/mL, 0.7368 g, 10.0 mmol) in THF (20 mL) was dropwise added 1-propynylmagnesium bromide (44 ml, 22.0 mmol, 0.5 M solution in THF) over 10 min at 0 °C via an ice-water bath under Ar atmosphere. The resulting mixture was warmed up to room temperature, stirred at room temperature for 7 h as monitored by TLC, quenched with water (220 mL), and extracted with ethyl acetate (30 mL x 3). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated. The crude alcohol product was directly used for next step without further purification.

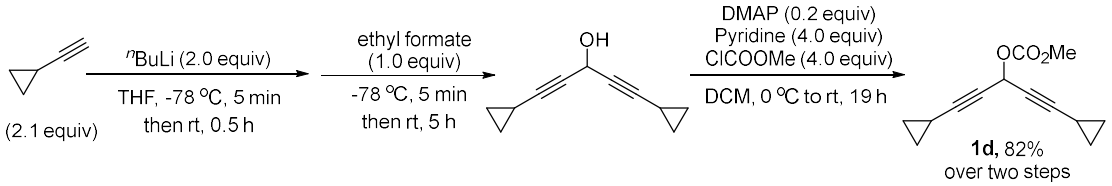
Step 2: Following **Typical Procedure II**, the reaction of the crude alcohol prepared above, DMAP (0.2443 mg, 2.0 mmol), pyridine (3.2 mL, d = 0.983 g/mL, 3.1456 g, 40.0 mmol), and $ClCO_2Me$ (3.1 mL, d = 1.22 g/mL, 3.782 g, 40.0 mmol) in DCM (30 mL) afforded **1b** (0.4980 g, 30% over two steps) [eluent: petroleum ether / ethyl acetate = 15:1 (480 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.90-5.82 (m, 1 H, CH), 3.82 (s, 3 H, CH_3), 1.89 (s, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.5, 82.7, 73.2, 57.8, 55.1, 3.6; **IR** (neat): v = 2962, 2239, 1752, 1441, 1315, 1249, 1172, 1119 cm^{-1}; **MS** (70 eV, EI) *m/z*: 151 ($(M-Me)^+$, 36.46), 91 (100); **HRMS** (EI) calcd for $C_8H_7O_3$ [$(M-Me)^+$]: 151.0390, found: 151.0390.

(3) Synthesis of hepta-2,5-diyn-4-yl methyl carbonate (1c, wj-3-081)



Following **Typical Procedure I**, the reaction of 1-octyne (3.1 mL, d = 0.747 g/mL, 2.3157 g, 21.0 mmol), THF (20 mL), ^{n}BuLi (8 mL, 20.0 mmol, 2.5 M in hexane), and ethyl formate (0.8 mL, d = 0.921 g/mL, 0.7368 g, 10.0 mmol) in THF (10 mL) afforded the crude alcohol; Then following **Typical Procedure II**, the reaction of the crude alcohol prepared above, DMAP (0.2447 g, 2.0 mmol), pyridine (3.2 mL, d = 0.983 g/mL, 3.1456 g, 40.0 mmol), and $ClCO_2Me$ (3.1 mL, d = 1.22 g/mL, 3.782 g, 40.0 mmol) in DCM (30 mL) afforded **1c** (2.6945 g, 88% over two steps) [eluent: petroleum ether / ethyl acetate = 50:1 (510 mL) to 40:1 (410 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.95-5.87 (m, 1 H, CH), 3.81 (s, 3 H, CH_3), 2.23 (t, *J* = 7.0 Hz, 4 H, 2 x CH_2), 1.52 (quint, *J* = 7.3 Hz, 4 H, 2 x CH_2), 1.43-1.20 (m, 12 H, 6 x CH_2), 0.89 (t, *J* = 6.8 Hz, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.5, 87.0, 74.1, 58.0, 55.0, 31.2, 28.4, 28.1, 22.5, 18.7, 14.0 cm^{-1}; **IR** (neat): *v* = 2932, 2858, 2237, 1752, 1441, 1320, 1250, 1169, 1124 cm^{-1}; **MS** (70 eV, EI) *m/z*: 306 (M^+, 3.17), 91(100); **HRMS** (EI) calcd for $C_{19}H_{30}O_3$ [M^+]: 306.2189, found: 306.2186.

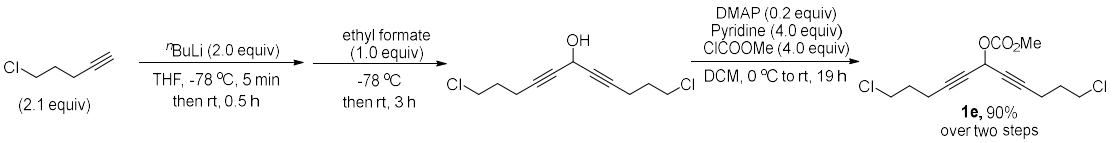
(4) Synthesis of 1,5-dicyclopropylpenta-1,4-diyn-3-yl methyl carbonate (1d, wj-3-082)



Following **Typical Procedure I**, the reaction of cyclopropylacethylene (1.8 mL, d = 0.78 g/mL, 1.404 g, 21.0 mmol), THF (20 mL), ^{n}BuLi (8 mL, 20.0 mmol, 2.5 M in hexane), and ethyl formate (0.8 mL, d = 0.921 g/mL, 0.7368 g, 10.0 mmol) in THF (10 mL) afforded the crude alcohol; Then following **Typical Procedure II**, the reaction of

the crude alcohol prepared above, DMAP (0.2450 mg, 2.0 mmol), pyridine (3.2 mL, d = 0.983 g/mL, 3.1456 g, 40.0 mmol), and $ClCO_2Me$ (3.1 mL, d = 1.22 g/mL, 3.782 g, 40.0 mmol) in DCM (30 mL) afforded **1d** (1.7747 g, 82% over two steps) [eluent: petroleum ether / ethyl acetate = 40:1 (410 mL) to 20:1 (420 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.89-5.82 (m, 1 H, CH), 3.81 (s, 3 H, CH_3), 1.37-1.21 (m, 2 H, 2 x CH), 0.88-0.67 (m, 8 H, 4 x CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.4, 89.9, 69.2, 58.0, 55.0, 8.2, -0.6 cm^{-1}; **IR** (neat): ν = 3013, 2251, 1748, 1441, 1363, 1306, 1248, 1153, 1027 cm^{-1}; **MS** (ESI) *m/z*: 241 ($M+Na^+$); **HRMS** (ESI) calcd for $C_{13}H_{14}O_3Na$ [$M+Na^+$]: 241.0835, found: 241.0834.

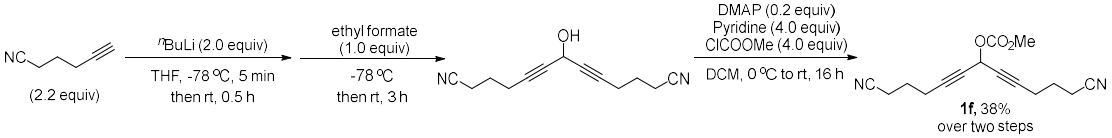
(5) Synthesis of 1,11-dichloroundeca-4,7-diyn-6-yl methyl carbonate (1e, wj-3-073, wj-3-154)



Following **Typical Procedure I**, the reaction of 5-chloro-1-pentyne (2.3 mL, d = 0.968 g/mL, 96%, 2.1373 g, 21.0 mmol), THF (20 mL), ^{n}BuLi (8 mL, 20.0 mmol, 2.5 M in hexane), and ethyl formate (0.8 mL, d = 0.921 g/mL, 0.7368 g, 10.0 mmol) in THF (10 mL) afforded the crude alcohol; Then following **Typical Procedure II**, the reaction of the crude alcohol prepared above, DMAP (0.2445 mg, 2.0 mmol), pyridine (3.2 mL, d = 0.983 g/mL, 3.1456 g, 40.0 mmol), and $ClCO_2Me$ (3.1 mL, d = 1.22 g/mL, 3.782 g, 40.0 mmol) in DCM (30 mL) afforded **1e** (2.6096 g, 90% over two steps) [eluent: petroleum ether / ethyl acetate = 30:1 (450 mL) to 10:1 (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.94-5.84 (m, 1 H, CH), 3.83 (s, 3 H, CH_3), 3.63 (t, J = 6.4 Hz, 4 H, 2 x CH_2), 2.52-2.38 (m, 4 H, 2 x CH_2), 1.99 (quint, J = 6.5 Hz, 4 H, 2 x CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.4, 85.1, 75.0, 57.6, 55.2, 43.4, 30.8, 16.2; **IR** (neat): ν = 2959, 2236, 1750, 1441, 1310, 1247, 1170, 1118 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 235 ([M($^{37}Cl_2$)-CO_2Me]$^+$, 4.17), 233 ([M($^{37}Cl^{35}Cl$)-CO_2Me]$^+$, 17.63), 231 ([M($^{35}Cl_2$)-CO_2Me]$^+$, 23.59), 115 (100); **HRMS** (EI) calcd for $C_{11}H_{13}{}^{35}Cl_2O$ [(M-

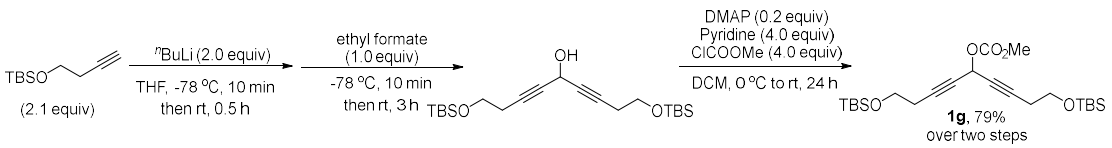
$CO_2Me)^+$]: 231.0338, found: 231.0335.

(6) Synthesis of 1,11-dicyanoundeca-4,7-diyn-6-yl methyl carbonate (1f, wj-3-094)



Following **Typical Procedure I**, the reaction of hex-5-ynenitrile (1.2 mL, d = 0.889 g/mL, 1.0668 g, 11.0 mmol), THF (10 mL), ^{n}BuLi (4 mL, 10.0 mmol, 2.5 M in hexane), and ethyl formate (0.4 mL, d = 0.921 g/mL, 0.3684 g, 5.0 mmol) in THF (5 mL) afforded the crude alcohol; Then following **Typical Procedure II**, the reaction of the crude alcohol prepared above, DMAP (0.1220 mg, 1.0 mmol), pyridine (1.6 mL, d = 0.983 g/mL, 1.5728 g, 20.0 mmol), and $ClCO_2Me$ (1.5 mL, d = 1.22 g/mL, 1.83 g, 19.4 mmol) in DCM (15 mL) afforded **1f** (0.5204 g, 38% over two steps) [eluent: petroleum ether / ethyl acetate = 2:1 (500 mL) to 1:1 (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.92-5.84 (m, 1 H, CH), 3.84 (s, 3 H, CH_3), 2.56-2.35 (m, 8 H, 4 x CH_2), 1.90 (quint, *J* = 7.0 Hz, 4 H, 2 x CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.2, 118.8, 84.1, 75.7, 57.2, 55.3, 24.0, 17.7, 16.1; **IR** (neat): *v* = 2959, 2246, 1751, 1441, 1312, 1250, 1167, 1120 cm^{-1}; **MS** (ESI) *m/z*: 295 ($M+Na^+$); **HRMS** (ESI) calcd for $C_{15}H_{16}O_3N_2Na$ [$M+Na^+$]: 295.1053, found: 295.1048.

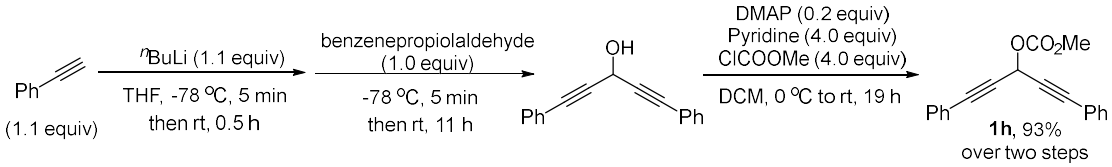
(7) Synthesis of 1,9-di((tert-butyldimethylsilyl)oxy)nona-3,6-diyn-5-yl methyl carbonate (1g, wj-3-080)



Following **Typical Procedure I**, the reaction of 4-tert-butyldimethylsilyloxylbut-1-yne (3.8742 g, 21.0 mmol), THF (20 mL), ^{n}BuLi (8 mL, 20.0 mmol, 2.5 M in hexane), and ethyl formate (0.8 mL, d = 0.921 g/mL, 0.7368 g, 10.0 mmol) in THF (10 mL) afforded the crude alcohol; Then following **Typical Procedure II**, the reaction of the

crude alcohol prepared above, DMAP (0.2450 mg, 2.0 mmol), pyridine (3.2 mL, d = 0.983 g/mL, 3.1456 g, 40.0 mmol), and $ClCO_2Me$ (3.1 mL, d = 1.22 g/mL, 3.782 g, 40.0 mmol) in DCM (30 mL) afforded **1g** (3.5589 g, 79% over two steps) [eluent: petroleum ether / ethyl acetate = 40:1 (410 mL) to 30/1 (450 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.94-5.88 (m, 1 H, CH), 3.81 (s, 3 H, CH_3), 3.73 (t, *J* = 7.0 Hz, 4 H, 2 x CH_2), 2.45 (t, *J* = 7.0 Hz, 4 H, 2 x CH_2), 0.89 (s, 18 H, 6 x CH_3), 0.07 (s, 12 H, 4 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.4, 84.0, 74.9, 61.3, 57.6, 55.1, 25.8, 23.1, 18.3, -5.4; **IR** (neat): *v* = 2953, 2929, 2856, 2240, 1754, 1472, 1442, 1320, 1250, 1104, 1057 cm^{-1}; **MS** (ESI) *m/z*: 477 ($M+Na^+$); **HRMS** (ESI) calcd for $C_{23}H_{42}O_5NaSi_2$ [$M+Na^+$]: 477.2463, found: 477.2462.

(8) Synthesis of 1,5-diphenylpenta-1,4-diyn-3-yl methyl carbonate (1h, wj-3-041)

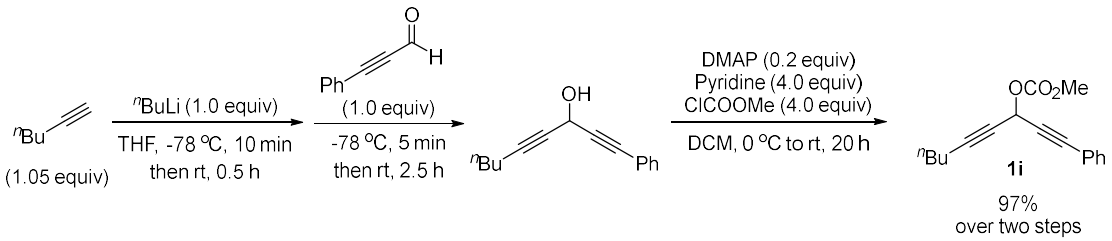


Typical Procedure III: To a stirred solution of phenylacetylene (2.5 mL, d = 0.93 g/mL, 2.325 g, 96% purity, 22.0 mmol) in THF (20 mL) was added *n*BuLi (8.8 mL, 22.0 mmol, 2.5 M in hexane) dropwise over 5 min at -78 °C under Ar atmosphere. The resulting mixture was stirred for 30 min at room temperature. Then the solution was cooled to -78 °C and 3-phenylpropiolaldehyde (2.4 mL, d = 1.064 g/mL, 2.5536 g, 19.6 mmol) was dropwise added over 5 min. The resulting mixture was stirred at room temperature for 11 h as monitored by TLC, quenched with water (10 mL), and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated. The crude alcohol product was directly used for next step without further purification.

Step 2: Following **Typical Procedure II**, the reaction of the crude alcohol prepared above, DMAP (0.4889 g, 4.0 mmol), pyridine (6.4 mL, d = 0.983 g/mL, 6.2912 g, 79.5 mmol), and $ClCO_2Me$ (6.2 mL, d = 1.22 g/mL, 7.564 g, 80.0 mmol) in DCM (60 mL)

afforded **1h** (5.3896 g, 93% over two steps) [eluent: petroleum ether / ethyl acetate = 20:1 (2000 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.51 (d, *J* = 7.2 Hz, 4 H, Ar-H), 7.40-7.27 (m, 6 H, Ar-H), 6.41 (s, 1 H, CH), 3.87 (s, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.4, 132.1, 129.2, 128.3, 121.5, 86.1, 82.1, 58.2, 55.4; **IR** (neat): *v* = 2239, 1751, 1490, 1441, 1318, 1242, 1095, 1045 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 290 (M^+, 22.1), 215 (100); **HRMS** calcd for $C_{19}H_{14}O_3$ [M^+]: 290.0937, found: 290.0937.

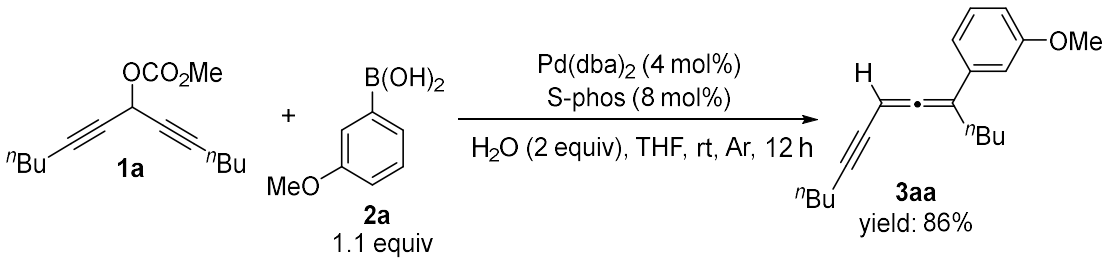
(9) Preparation of 1-phenylnona-1,4-diyn-3-yl methyl carbonate (1i, wj-2-062, wj-3-166)



Following **Typical Procedure III**, the reaction of 1-hexyne (2.4 mL, d = 0.715 g/mL, 1.7160 g, 20.9 mmol), THF (20 mL), ^{n}BuLi (8 mL, 20.0 mmol, 2.5 M in hexane), and benzenepropiolaldehyde (2.45 mL, d = 1.064 g/mL, 2.6068 g, 20.0 mmol) afforded the crude alcohol; Then following **Typical Procedure II**, the reaction of the crude alcohol prepared above, DMAP (0.4885 g, 4.0 mmol), pyridine (6.4 mL, d = 0.983 g/mL, 6.2912 g, 80.0 mmol), and $ClCO_2Me$ (6.2 mL, d = 1.22 g/mL, 7.5640 g, 80.0 mmol) in DCM (50 mL) afforded **1i** (5.2405 g, 97% over two steps)[eluent: petroleum ether / ethyl acetate = 30:1 (310 mL) to 20:1 (420 ml)]; oil: **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.53-7.42 (m, 2 H, Ar-H), 7.38-7.27 (m, 3 H, Ar-H), 6.19-6.10 (m, 1 H, CH), 3.84 (s, 3 H, CH_3), 2.27 (t, *J* = 7.0 Hz, 2 H, CH_2), 1.58-1.48 (m, 2 H, CH_2), 1.48-1.35 (m, 2 H, CH_2), 0.91 (t, *J* = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 154.6, 132.0, 129.0, 128.2, 121.7, 87.8, 85.4, 82.8, 73.6, 58.0, 55.1, 30.1, 21.9, 18.4, 13.5; **IR** (neat): *v* = 2958, 2934, 2872, 2237, 1751, 1491, 1441, 1319, 1245, 1149, 1096, 1008 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 271 (M^++1, 8.29), 270 (M^+, 45.15), 152 (100); **HRMS** (EI) calcd for $C_{17}H_{18}O_3$ [M^+]: 270.1250, found: 270.1251.

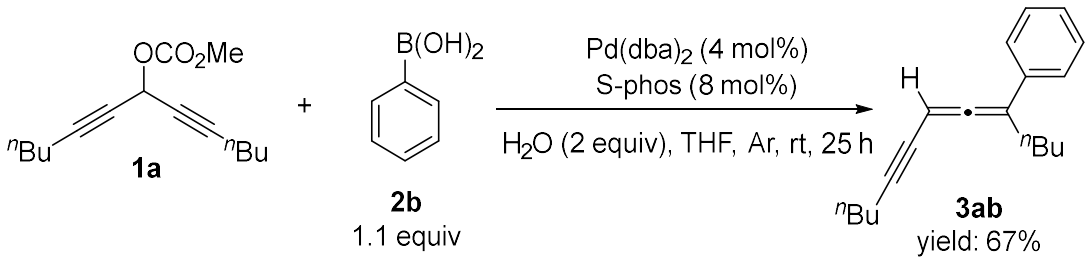
Synthesis of conjugated allenynes with S-phos

(1) Synthesis of 9-(3-methoxylphenyl)trideca-7,8-dien-5-yne (3aa, wj-2-063, wj-3-015)



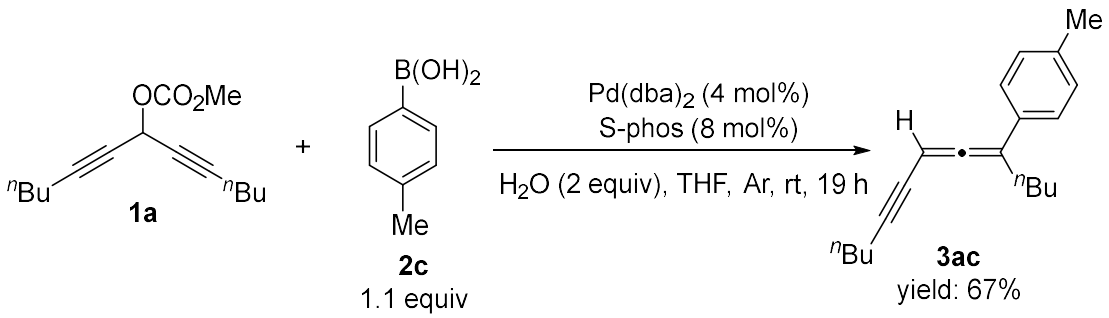
Typical Procedure IV: To a Schlenk tube were added 3-methoxyphenylboronic acid **2a** (167.1 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), and S-phos (32.7 mg, 0.08 mmol). After adding all of solid chemicals, the flask was degassed and refilled with Ar for three times. Then methyl carbonate **1a** (250.3 mg, 1.0 mmol)/THF (5 mL) and H_2O (2.0 mmol, 36 μL) were added sequentially. After that, the resulting mixture was stirred at room temperature for 12 h as monitored by TLC, diluted with ethyl acetate (5 mL), filtrated through a short column of silica gel (3 cm) eluted with ethyl acetate (20 mL), and evaporated. The resulting residue was purified by chromatography on silica gel to afford the product **3aa** (242.5 mg, 86%) [eluent: petroleum ether / dichloromethane = 5:1 (300 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.24 (t, J = 6.8 Hz, 1 H, Ar-H), 6.99 (d, J = 8.0 Hz, 1 H, Ar-H), 6.94 (s, 1 H, Ar-H), 6.78 (dd, J_1 = 8.2 Hz, J_2 = 1.8 Hz, 1 H, Ar-H), 5.74-5.68 (m, 1 H, CH), 3.81 (s, 3 H, CH_3), 2.52-2.37 (m, 2 H, CH_2), 2.30 (td, J_1 = 7.0 Hz, J_2 = 1.6 Hz, 2 H, CH_2), 1.59-1.48 (m, 4 H, 2 x CH_2), 1.48-1.36 (m, 4 H, 2 x CH_2), 0.98-0.85 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.1, 159.7, 137.2, 129.3, 118.9, 112.35, 112.32, 107.6, 91.1, 79.0, 72.8, 55.2, 30.8, 29.8, 29.7, 22.4, 22.0, 19.3, 13.9, 13.6; **IR** (neat): ν = 2956, 2930, 2861, 2216, 1935, 1600, 1581, 1487, 1463, 1431, 1380, 1322, 1287, 1268, 1226, 1198, 1166, 1104 cm^{-1}; **MS** (70 eV, EI) *m/z*: 282 (M^+, 26.07), 197 (100); **HRMS** (EI) calcd for $C_{20}H_{26}O$ [M^+]: 282.1978, found: 282.1980.

(2) Synthesis of 9-phenyltrideca-7,8-dien-5-yne (3ab, wj-3-044)



Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (134.0 mg, 1.1 mmol), $Pd(dba)_2$ (23.1 mg, 0.04 mmol), S-phos (32.7 mg, 0.08 mmol), methyl carbonate **1a** (250.2 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 µL) afforded the product **3ab** (170.1 mg, 67%) [eluent: petroleum ether (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.39 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.32 (t, *J* = 7.6 Hz, 2 H, Ar-H), 7.22 (t, *J* = 7.4 Hz, 1 H, Ar-H), 5.75-5.68 (m, 1 H, CH), 2.54-2.38 (m, 2 H, CH_2), 2.31 (td, J_1 = 6.9 Hz, J_2 = 1.2 Hz, 2 H, CH_2), 1.61-1.47 (m, 4 H, 2 x CH_2), 1.47-1.34 (m, 4 H, 2 x CH_2), 0.99-0.85 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.0, 135.6, 128.4, 127.1, 126.4, 107.6, 91.0, 79.0, 72.9, 30.8, 29.8, 29.6, 22.4, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2956, 2930, 2868, 2216, 1938, 1597, 1493, 1452, 1378, 1325, 1103, 1073, 1031 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 252 (M^+, 1.59), 167 (100); **HRMS** (EI) calcd for $C_{19}H_{24}$ [M^+]: 252.1873, found: 252.1872.

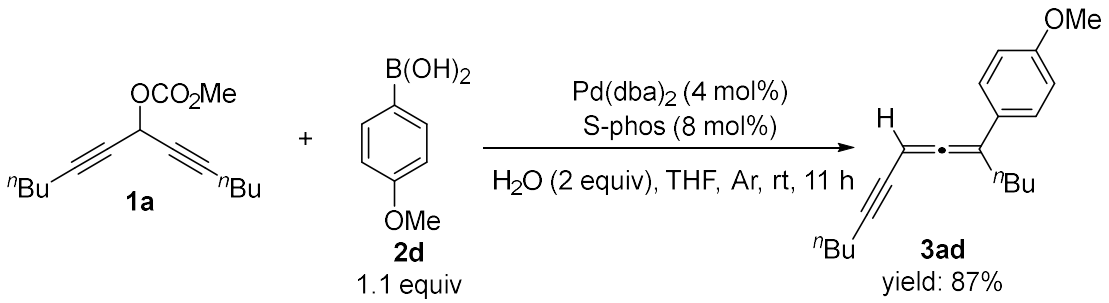
(3) Synthesis of 9-(4-methylphenyl)trideca-7,8-dien-5-yne (3ac, wj-3-043)



Following **Typical Procedure IV**, the reaction of *p*-tolylboronic acid **2c** (149.6 mg, 1.1 mmol), $Pd(dba)_2$ (23.1 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1a** (250.0 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 µL) afforded

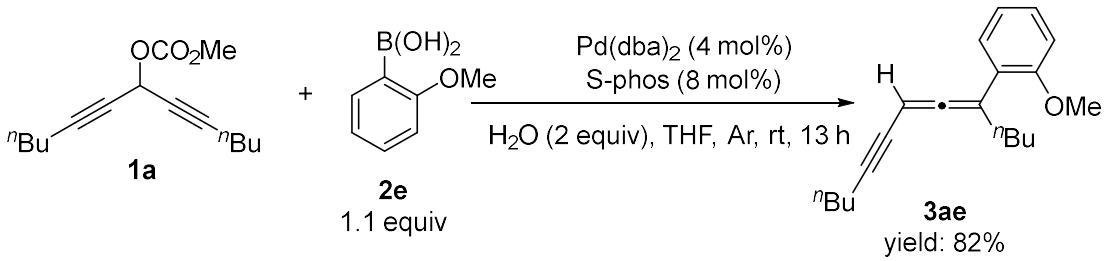
the product **3ac** (179.6 mg, 67%) [eluent: petroleum ether (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.28 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.13 (d, *J* = 7.6 Hz, 2 H, Ar-H), 5.73-5.66 (m, 1 H, CH), 2.51-2.37 (m, 2 H, CH_2), 2.36-2.24 (m, 5 H, CH_3 and CH_2), 1.59-1.47 (m, 4 H, 2 x CH_2), 1.47-1.34 (m, 4 H, 2 x CH_2), 0.98-0.86 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.9, 136.9, 132.6, 129.1, 126.3, 107.5, 90.8, 78.8, 73.1, 30.8, 29.8, 29.6, 22.4, 22.0, 21.1, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2957, 2929, 2860, 2215, 1934, 1511, 1462, 1378, 1326, 1185, 1110 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 266 (M^+, 4.86), 165 (100); **HRMS** (EI) calcd for $C_{20}H_{26}$ [M^+]: 266.2029, found: 266.2031.

(4) Synthesis of 9-(4-methoxylphenyl)trideca-7,8-dien-5-yne (3ad, wj-3-016)



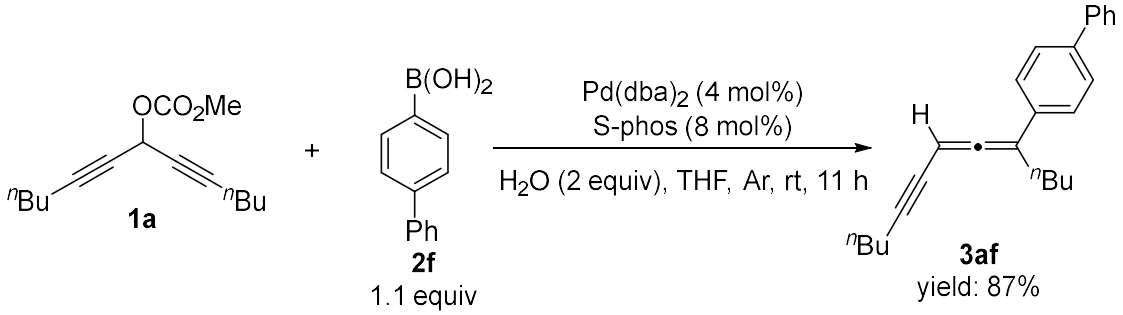
Following **Typical Procedure IV**, the reaction of 4-methoxyphenylboronic acid **2d** (167.3 mg, 1.1 mmol), $Pd(dba)_2$ (23.1 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1a** (250.0 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ad** (244.8 mg, 87%) [eluent: petroleum ether / dichloromethane = 5:1 (300 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.31 (d, *J* = 8.8 Hz, 2 H, Ar-H), 6.87 (d, *J* = 8.8 Hz, 2 H, Ar-H), 5.74-5.67 (m, 1 H, CH), 3.80 (s, 3 H, CH_3), 2.51-2.36 (m, 2 H, CH_2), 2.30 (td, J_1 = 7.0 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.59-1.47 (m, 4 H, 2 x CH_2), 1.47-1.36 (m, 4 H, 2 x CH_2), 0.99-0.86 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.7, 158.8, 127.8, 127.5, 113.9, 107.2, 90.6, 78.8, 73.2, 55.3, 30.8, 29.8, 29.7, 22.4, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2954, 2930, 2870, 2859, 2208, 1935, 1606, 1509, 1464, 1294, 1246, 1177, 1109, 1036 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 282 (M^+, 18.97), 165 (100); **HRMS** (EI) calcd for $C_{20}H_{26}O$ [M^+]: 282.1978, found: 282.1982.

(5) Synthesis of 9-(2-methoxylphenyl)trideca-7,8-dien-5-yne (3ae, wj-3-030)



Following **Typical Procedure IV**, the reaction of 2-methoxyphenylboronic acid **2e** (167.2 mg, 1.1 mmol), $Pd(dba)_2$ (23.1 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1a** (249.9 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ae** (230.4 mg, 82%) [eluent: petroleum ether / dichloromethane = 5:1 (300 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.21 (d, *J* = 7.6 Hz, 2 H, Ar-H), 6.92 (t, *J* = 7.4 Hz, 1 H, CH), 6.87 (d, *J* = 8.0 Hz, 1 H, Ar-H), 5.48 (t, *J* = 2.2 Hz, 1 H, CH), 3.82 (s, 3 H, CH_3), 2.49-2.38 (m, 2 H, CH_2), 2.31 (td, J_1 = 6.9 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.58-1.31 (m, 8 H, 4 x CH_2), 0.96-0.84 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.6, 156.9, 129.6, 128.5, 125.9, 120.6, 111.3, 105.1, 90.4, 75.7, 73.6, 55.6, 31.8, 30.9, 29.8, 22.3, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2956, 2931, 2870, 2216, 1938, 1595, 1580, 1490, 1461, 1434, 1284, 1051, 1028 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 282 (M^+, 5.13), 267 (100); **HRMS** (EI) calcd for $C_{20}H_{26}O$ [M^+]: 282.1978, found: 282.1983.

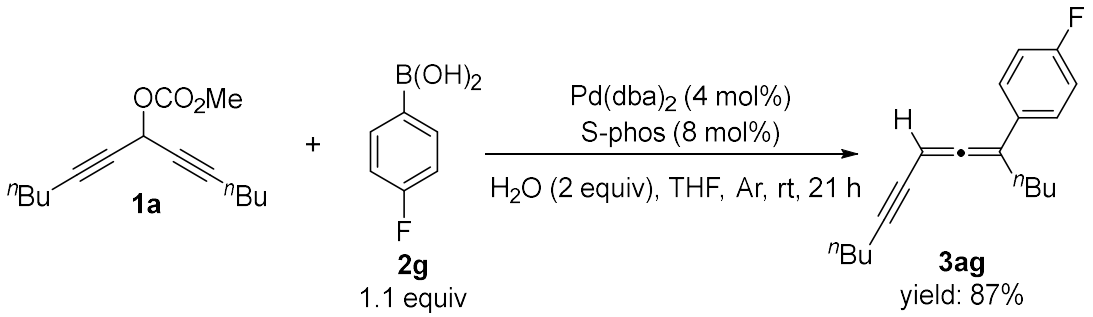
(6) Synthesis of 9-(4-phenylphenyl)trideca-7,8-dien-5-yne (3af, wj-3-017)



Following **Typical Procedure IV**, the reaction of 4-phenylphenylboronic acid **2f** (217.7 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.7 mg, 0.08 mmol),

methyl carbonate **1a** (250.1 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3af** (286.2 mg, 87%) [eluent: petroleum ether (800 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.63-7.53 (m, 4 H, Ar-H), 7.49-7.39 (m, 4 H, Ar-H), 7.34 (t, *J* = 7.4 Hz, 1 H, Ar-H), 5.76 (t, *J* = 2.4 Hz, 1 H, CH), 2.58-2.42 (m, 2 H, CH_2), 2.32 (td, J_1 = 7.0 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.63-1.48 (m, 4 H, 2 x CH_2), 1.48-1.36 (m, 4 H, 2 x CH_2), 1.00-0.88 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.3, 140.7, 139.9, 134.6, 128.8, 127.3, 127.1, 127.0, 126.8, 107.4, 91.2, 79.1, 72.8, 30.8, 29.8, 29.6, 22.4, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2956, 2930, 2870, 2215, 1933, 1600, 1486, 1462, 1265, 1109, 1006 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 328 (M^+, 11.46), 243 (100); **HRMS** (EI) calcd for $C_{25}H_{28}$ [M^+]: 328.2186, found: 328.2187.

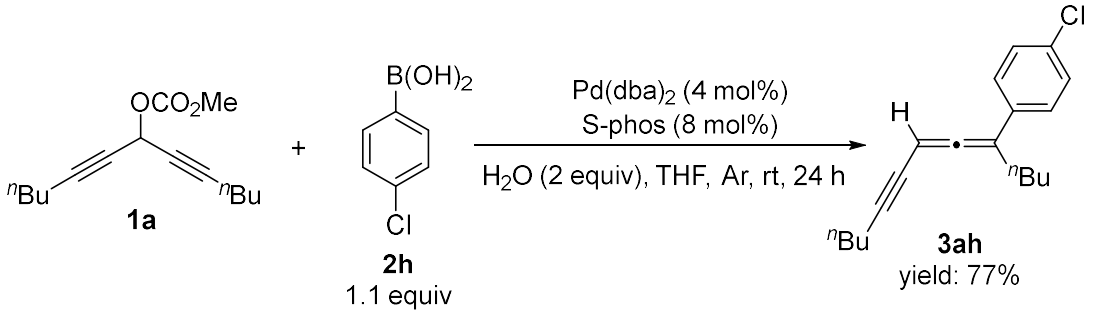
(7) Synthesis of 9-(4-fluorophenyl)trideca-7,8-dien-5-yne (3ag, wj-3-018)



Following **Typical Procedure IV**, the reaction of 4-fluorophenylboronic acid **2g** (154.0 mg, 1.1 mmol), $Pd(dba)_2$ (23.1 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1a** (250.0 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ag** (235.9 mg, 87%) [eluent: petroleum ether (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.41-7.29 (m, 2 H, Ar-H), 7.01 (t, *J* = 8.6 Hz, 2 H, Ar-H), 5.79-5.67 (m, 1 H, CH), 2.53-2.36 (m, 2 H, CH_2), 2.31 (td, J_1 = 7.0 Hz, J_2 = 1.6 Hz, 2 H, CH_2), 1.61-1.47 (m, 4 H, 2 x CH_2), 1.47-1.33 (m, 4 H, 2 x CH_2), 1.03-0.85 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.8 (d, *J* = 2.4 Hz), 162.0 (d, *J* = 244.9 Hz), 131.6 (d, *J* = 3.2 Hz), 127.9 (d, *J* = 7.9 Hz), 115.3 (d, *J* = 22.1 Hz), 106.8, 91.2, 79.2, 72.8, 30.8, 29.8, 29.7, 22.4, 22.0, 19.3, 13.9, 13.6; **^{19}F NMR** (376 MHz, $CDCl_3$): *δ* = -115.9; **IR** (neat): *v* = 2957, 2930, 2861, 2214, 1936, 1601, 1506, 1463,

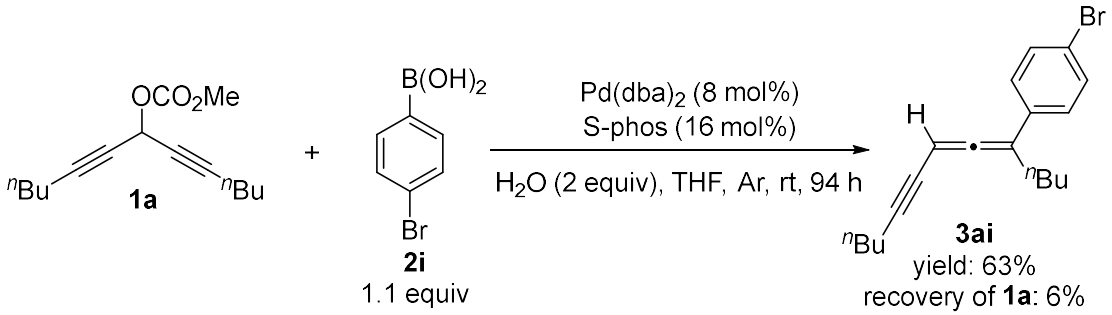
1230, 1159, 1098, 1014 cm^{-1}; **MS** (70 eV, EI) *m/z*: 270 (M^+, 2.75), 185 (100); **HRMS** (EI) calcd for $C_{19}H_{23}F$ [M^+]: 270.1778, found: 270.1782.

(8) Synthesis of 9-(4-chlorophenyl)trideca-7,8-dien-5-yne (3ah, wj-3-019)



Following **Typical Procedure IV**, the reaction of 4-chlorophenylboronic acid **2h** (172.1 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.7 mg, 0.08 mmol), methyl carbonate **1a** (250.2 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 µL) afforded the product **3ah** (220.9 mg, 77%) [eluent: petroleum ether (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.35-7.24 (m, 4 H, Ar-H), 5.78-5.69 (m, 1 H, CH), 2.50-2.35 (m, 2 H, CH_2), 2.31 (td, J_1 = 6.9 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.59-1.47 (m, 4 H, 2 x CH_2), 1.47-1.33 (m, 4 H, 2 x CH_2), 1.00-0.84 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.0, 134.2, 132.8, 128.5, 127.6, 106.9, 91.5, 79.5, 72.5, 30.8, 29.7, 29.5, 22.3, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2957, 2930, 2867, 2216, 1936, 1489, 1463, 1380, 1093, 1012 cm^{-1}; **MS** (70 eV, EI) *m/z*: 288 (M^+(^{37}Cl), 0.47), 286 (M^+(^{35}Cl), 1.43), 165 (100); **HRMS** (EI) calcd for $C_{19}H_{23}{}^{35}Cl$ [M^+]: 286.1483, found: 286.1483.

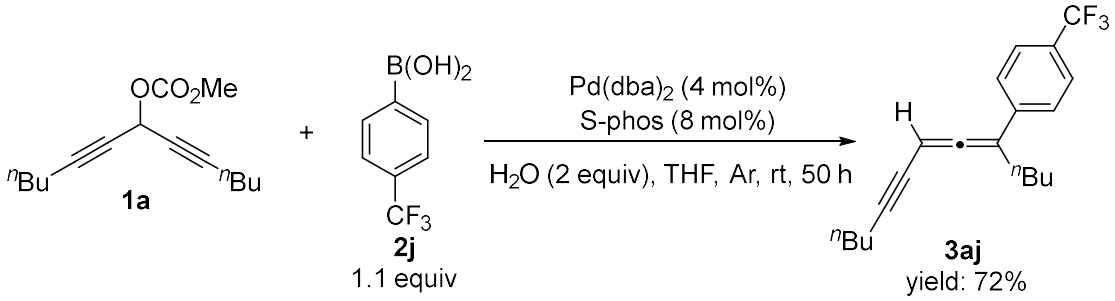
(9) Synthesis of 9-(4-bromophenyl)trideca-7,8-dien-5-yne (3ai, wj-3-028)



Following **Typical Procedure IV**, the reaction of 4-bromophenylboronic acid **2i**

(220.9 mg, 1.1 mmol), $Pd(dba)_2$ (46.1 mg, 0.08 mmol), S-phos (65.7 mg, 0.16 mmol), methyl carbonate **1a** (250.3 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ai** (209.0 mg, 63%) [eluent: petroleum ether (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.43 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.24 (d, *J* = 8.4 Hz, 2 H, Ar-H), 5.77-5.68 (m, 1 H, CH), 2.39-2.35 (m, 2 H, CH_2), 2.31 (td, J_1 = 7.1 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.61-1.47 (m, 4 H, 2 x CH_2), 1.47-1.33 (m, 4 H, 2 x CH_2), 1.00-0.85 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.0, 134.7, 131.5, 128.0, 121.0, 106.9, 91.6, 79.5, 72.5, 30.8, 29.6, 29.5, 22.3, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2956, 2930, 2860, 2215, 1936, 1485, 1463, 1380, 1264, 1102, 1073 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 332 (M^+(^{81}Br), 1.00), 330 (M^+(^{79}Br), 0.95), 165 (100); **HRMS** (EI) calcd for $C_{19}H_{23}{}^{79}Br$ [M^+]: 330.0978, found: 330.0977.

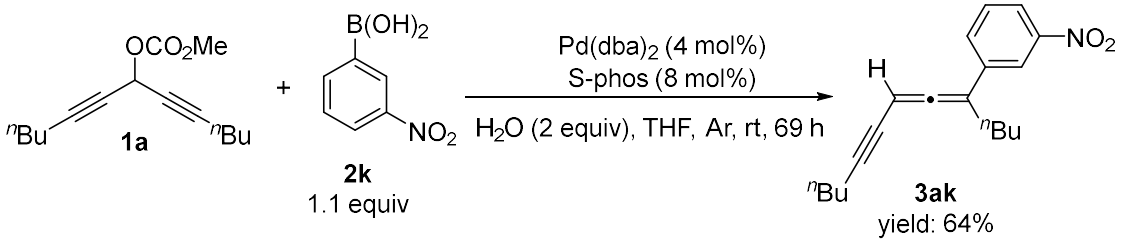
(10) Synthesis of 9-(4-trifluoromethylphenyl)trideca-7,8-dien-5-yne (3aj, wj-3-031)



Following **Typical Procedure IV**, the reaction of 4-trifluoromethylphenylboronic **2j** acid (209.1 mg, 1.1 mmol), $Pd(dba)_2$ (23.1 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1a** (250.6 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3aj** (234.0 mg, 72%) [eluent: petroleum ether (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.57 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.48 (d, *J* = 8.4 Hz, 2 H, Ar-H), 5.82-5.73 (m, 1 H, CH), 2.55-2.38 (m, 2 H, CH_2), 2.32 (td, J_1 = 6.9 Hz, J_2 = 1.6 Hz, 2 H, CH_2), 1.62-1.48 (m, 4 H, 2 x CH_2), 1.48-1.35 (m, 4 H, 2 x CH_2), 1.00-0.86 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.6, 139.6, 129.0 (q, *J* = 32.1 Hz), 126.6, 125.3 (q, *J* = 4.0 Hz), 124.2 (q, *J* = 270.2 Hz), 106.9, 92.0, 79.8, 72.2, 30.7, 29.7, 29.5, 22.3, 22.0, 19.3, 13.9, 13.6; **^{19}F NMR** (376 MHz, $CDCl_3$): *δ* = -63.0;

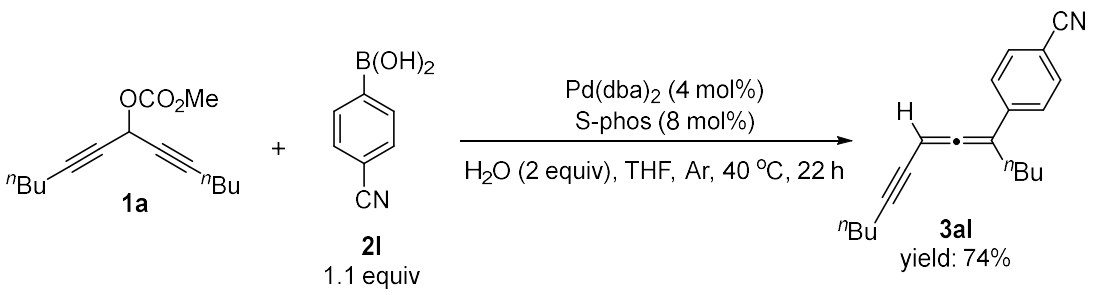
IR (neat): ν = 2959, 2932, 2862, 2218, 1937, 1615, 1464, 1323, 1165, 1124, 1068, 1015 cm^{-1}; **MS** (70 eV, EI) *m/z*: 320 (M^+, 1.71), 165 (100); **HRMS** (EI) calcd for $C_{20}H_{23}F_3$ [M^+]: 320.1746, found: 320.1753.

(11) Synthesis of 9-(3-nitrophenyl)trideca-7,8-dien-5-yne (3ak, wj-3-032)



Following **Typical Procedure IV**, the reaction of 3-nitrophenylboronic acid **2k** (183.7 mg, 1.1 mmol), $Pd(dba)_2$ (22.9 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1a** (250.5 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ak** (194.0 mg, 64%) [eluent: petroleum ether / dichloromethane = 5:1 (480 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 8.20 (s, 1 H, Ar-H), 8.08 (d, *J* = 8.0 Hz, 1 H, Ar-H), 7.72 (d, *J* = 7.6 Hz, 1 H, Ar-H), 7.49 (t, *J* = 8.0 Hz, 1 H, CH), 5.88-5.80 (m, 1 H, CH), 2.58-2.40 (m, 2 H, CH_2), 2.32 (td, J_1 = 7.0 Hz, J_2 = 1.6 Hz, 2 H, CH_2), 1.64-1.48 (m, 4 H, 2 x CH_2), 1.48-1.36 (m, 4 H, 2 x CH_2), 1.02-0.86 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.4, 148.6, 137.9, 132.5, 129.2, 121.9, 120.8, 106.4, 92.6, 80.6, 71.9, 30.7, 29.5, 29.4, 22.3, 22.0, 19.3, 13.9, 13.6; **IR** (neat): ν = 2958, 2930, 2870, 2218, 1934, 1527, 1465, 1346, 1097, 1079 cm^{-1}; **MS** (ESI) *m/z*: 298 ($M+H^+$); **HRMS** (ESI) calcd for $C_{19}H_{24}O_2N$ [$M+H^+$]: 298.1802, found: 298.1802.

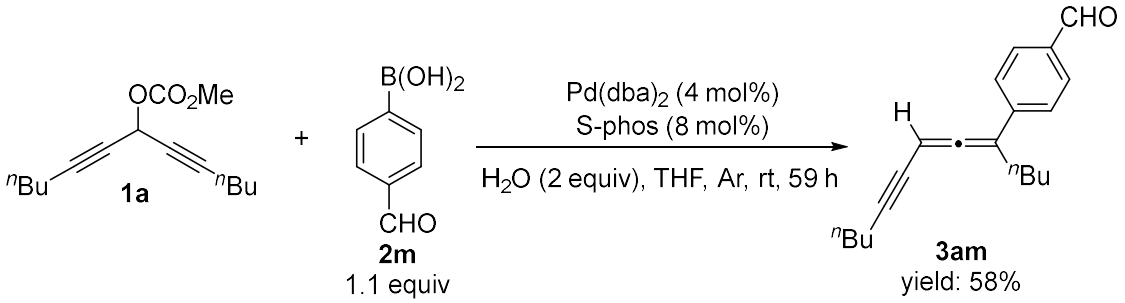
(12) Synthesis of 9-(4-cynaophenyl)trideca-7,8-dien-5-yne (3al, wj-3-110)



Following **Typical Procedure IV**, the reaction of 4-cynaophenylboronic acid **3al**

(170.0 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1a** (250.4 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3al** (204.1 mg, 74%) [eluent: petroleum ether / ethyl acetate = 50:1 (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.47 (d, *J* = 8.0 Hz, 2 H, Ar-H), 5.87-5.77 (m, 1 H, CH), 2.55-2.38 (m, 2 H, CH_2), 2.32 (t, *J* = 6.8 Hz, 2 H, CH_2), 1.61-1.48 (m, 4 H, 2 x CH_2), 1.48-1.33 (m, 4 H, 2 x CH_2), 1.00-0.84 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 214.1, 140.8, 132.2, 126.9, 119.0, 110.4, 106.9, 92.6, 80.3, 71.8, 30.7, 29.6, 29.2, 22.3, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2957, 2931, 2869, 2226, 1933, 1603, 1503, 1462, 1380, 1107, 1017 cm^{-1}; **MS** (DART) *m*/*z*: 278 ($M+H^+$); **HRMS** (DART) calcd for $C_{20}H_{24}N$ [$M+H^+$]: 278.1903, found: 278.1903.

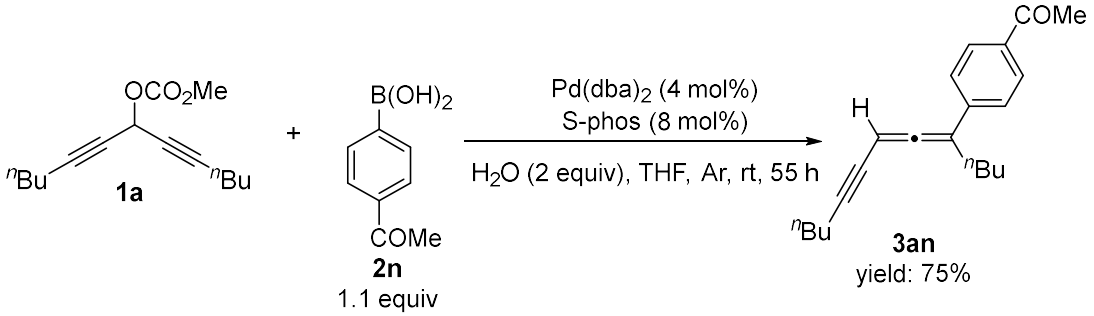
(13) Synthesis of 9-(4-formylphenyl)trideca-7,8-dien-5-yne (3am, wj-3-021)



Following **Typical Procedure IV**, the reaction of 4-formylphenylboronic acid **2m** (164.8 mg, 1.1 mmol), $Pd(dba)_2$ (22.9 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1a** (250.2 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3am** (163.3 mg, 58%) [eluent: petroleum ether / dichloromethane / ethyl ether = 50:1:1 (520 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 9.98 (s, 1 H, CHO), 7.83 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.54 (d, *J* = 8.0 Hz, 2 H, Ar-H), 5.88-5.77 (m, 1 H, CH), 2.57-2.41 (m, 2 H, CH_2), 2.32 (td, J_1 = 6.9 Hz, J_2 = 1.6 Hz, 2 H, CH_2), 1.62-1.48 (m, 4 H, 2 x CH_2), 1.48-1.36 (m, 4 H, 2 x CH_2), 1.02-0.84 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 214.3, 191.7, 142.3, 135.0, 129.8, 126.8, 107.3, 92.3, 80.0, 71.9, 30.7, 29.7, 29.4, 22.3, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2957, 2931, 2861,

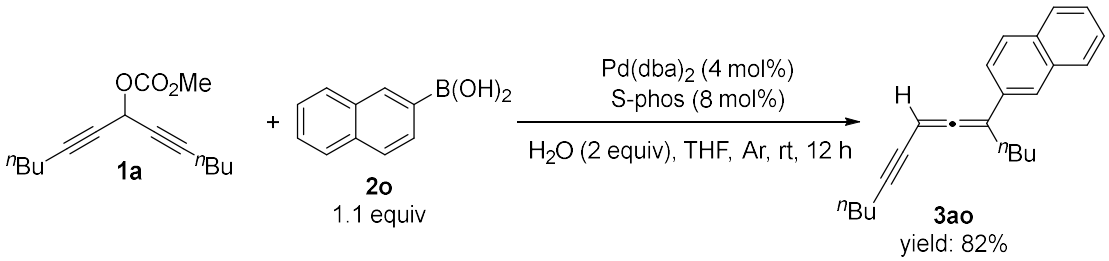
2213, 1933, 1699, 1601, 1463, 1381, 1306, 1212, 1169, 1106 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 280 (M^+, 3.56), 165 (100); **HRMS** (EI) calcd for $C_{20}H_{24}O$ [M^+]: 280.1822, found: 280.1822.

(14) Synthesis of 9-(4-acetylphenyl)trideca-7,8-dien-5-yne (3an, wj-3-022)



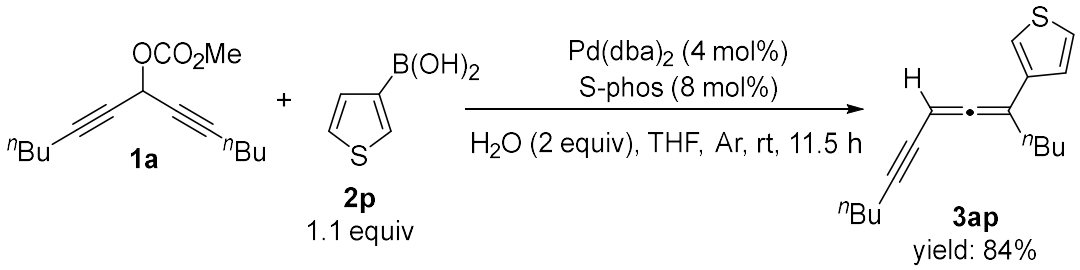
Following **Typical Procedure IV**, the reaction of 4-acetylphenylboronic acid **2n** (180.5 mg, 1.1 mmol), $Pd(dba)_2$ (22.9 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1a** (250.5 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3an** (220.8 mg, 75%) [eluent: petroleum ether / dichloromethane / ethyl ether = 25:1:1 (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.91 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.47 (d, *J* = 8.0 Hz, 2 H, Ar-H), 5.83-5.76 (m, 1 H, CH), 2.59 (s, 3 H, CH_3), 2.55-2.40 (m, 2 H, CH_2), 2.32 (td, J_1 = 6.9 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.61-1.48 (m, 4 H, 2 x CH_2), 1.48-1.35 (m, 4 H, 2 x CH_2), 1.01-0.86 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 214.0, 197.6, 140.8, 135.6, 128.5, 126.4, 107.3, 92.0, 79.7, 72.1, 30.7, 29.7, 29.4, 26.5, 22.3, 21.9, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2957, 2930, 2862, 2216, 1935, 1681, 1601, 1463, 1357, 1265, 1185, 1015 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 294 (M^+, 23.59), 252 (100); **HRMS** (EI) calcd for $C_{21}H_{26}O$ [M^+]: 294.1978, found: 294.1978.

(15) Synthesis of 9-(2-nathphyl)trideca-7,8-dien-5-yne (3ao, wj-3-026)



Following **Typical Procedure IV**, the reaction of 2-naphthylboronic acid **2o** (189.3 mg, 1.1 mmol), $Pd(dba)_2$ (22.9 mg, 0.04 mmol), S-phos (32.7 mg, 0.08 mmol), methyl carbonate **1a** (250.0 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ao** (246.6 mg, 82%) [eluent: petroleum ether / ethyl acetate = 300:1 (400 mL) to 100:1 (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.85-7.69 (m, 4 H, Ar-H), 7.56 (d, *J* = 8.8 Hz, 1 H, Ar-H), 7.50-7.37 (m, 2 H, Ar-H), 5.85-5.76 (m, 1 H, CH), 2.66-2.50 (m, 2 H, CH_2), 2.32 (td, J_1 = 7.0 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.66-1.35 (m, 8 H, 4 x CH_2), 1.01-0.85 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.8, 133.5, 133.0, 132.6, 128.0, 127.8, 127.5, 126.1, 125.8, 125.5, 124.2, 107.9, 91.3, 79.4, 72.8, 30.8, 29.8, 29.5, 22.4, 22.0, 19.3, 14.0, 13.6; **IR** (neat): *v* = 2956, 2929, 2869, 2212, 1933, 1597, 1504, 1463, 1266, 1103 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 302 (M^+, 11.96), 217 (100); **HRMS** (EI) calcd for $C_{23}H_{26}$ [M^+]: 302.2029, found: 302.2030.

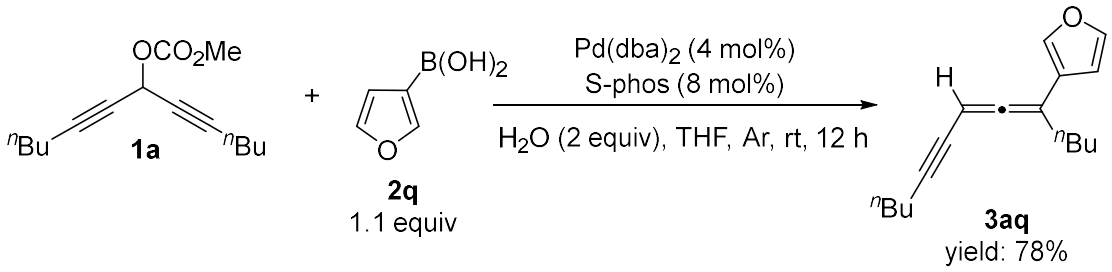
(16) Synthesis of 9-(3-thienyl)trideca-7,8-dien-5-yne (3ap, wj-3-025)



Following **Typical Procedure IV**, the reaction of 3-thiophenylboronic acid **2p** (140.9 mg, 1.1 mmol), $Pd(dba)_2$ (22.9 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1a** (250.5 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ap** (221.5 mg, 84%) [eluent: petroleum ether (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.29-7.20 (m, 1 H, Ar-H), 7.17-7.07 (m, 2 H, Ar-H), 5.69

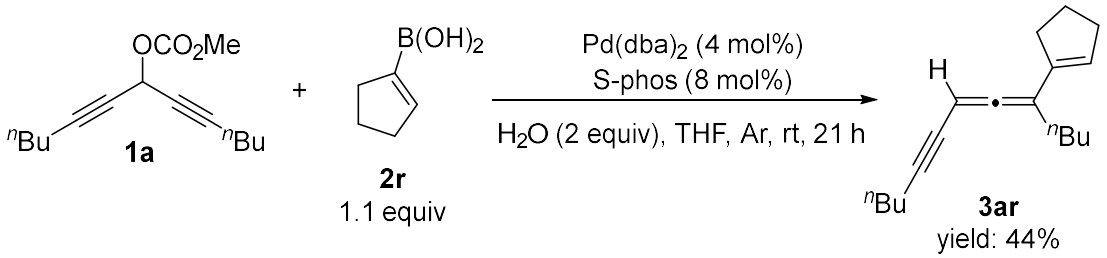
(t, J = 2.4 Hz, 1 H, CH), 2.50-2.35 (m, 2 H, CH_2), 2.30 (td, J_1 = 7.0 Hz, J_2 = 2.0 Hz, 2 H, CH_2), 1.62-1.47 (m, 4 H, 2 x CH_2), 1.47-1.33 (m, 4 H, 2 x CH_2), 1.00-0.85 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.2, 137.3, 127.0, 125.3, 119.8, 103.8, 91.0, 78.8, 72.9, 30.8, 30.3, 29.7, 22.4, 22.0. 19.2, 13.9, 13.6; **IR** (neat): v = 2956, 2928, 2869, 2215, 1936, 1461, 1377, 1326, 1232, 1080 cm^{-1}; **MS** (70 eV, EI) m/z: 258 (M^+, 6.23), 173 (100); **HRMS** (EI) calcd for $C_{17}H_{22}S$ [M^+]: 258.1437, found: 258.1438.

(17) Synthesis of 9-(3-furyl)trideca-7,8-dien-5-yne (3aq, wj-3-027)



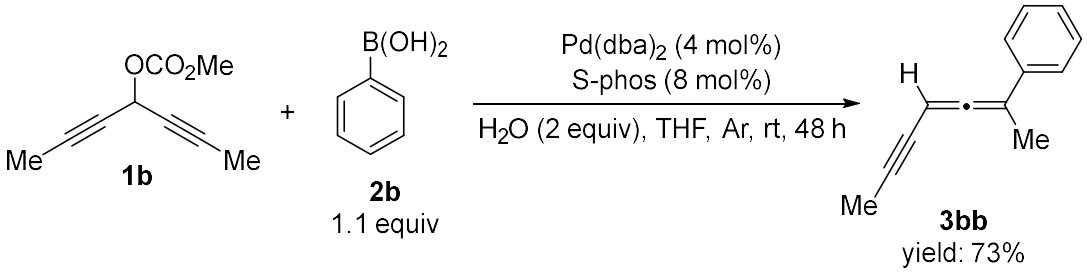
Following **Typical Procedure IV**, the reaction of 3-furanylboronic acid **2q** (128.3 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.7 mg, 0.08 mmol), methyl carbonate **1a** (249.9 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3aq** (189.1 mg, 78%) [eluent: petroleum ether / dichloromethane = 100:1 (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.42-7.36 (m, 2 H, Ar-H), 6.39 (s, 1 H, Ar-H), 5.70-5.62 (m, 1 H, CH), 2.37-2.22 (m, 4 H, 2 x CH_2), 1.59-1.47 (m, 4 H, 2 x CH_2), 1.47-1.34 (m, 4 H, 2 x CH_2), 0.98-0.87 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 211.9, 143.2, 138.6, 122.2, 109.4, 100.5, 90.9, 78.7, 72.9, 30.8, 30.1, 29.6, 22.3, 22.0, 19.2, 13.9, 13.6; **IR** (neat): v = 2957, 2930, 2871, 2216, 1936, 1463, 1160, 1072, 1036, 1011 cm^{-1}; **MS** (70 eV, EI) m/z: 242 (M^+, 11.05), 128 (100); **HRMS** (EI) calcd for $C_{17}H_{22}O$ [M^+]: 242.1665, found: 242.1667.

(18) Synthesis of 9-(1-cyclopentenyl)trideca-7,8-dien-5-yne (3ar, wj-3-036)



Following **Typical Procedure IV**, the reaction of cyclopent-1-en-1-ylboronic acid **2r** (123.2 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.7 mg, 0.08 mmol), methyl carbonate **1a** (250.1 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ar** (108.4 mg, 44%) [eluent: petroleum ether (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 5.71 (s, 1 H, CH), 5.54 (s, 1 H, Ar-H), 2.49-2.14 (m, 8 H, 4 x CH_2), 1.88 (quint, *J* = 7.5 Hz, 2 H, CH_2), 1.61-1.23 (m, 8 H, 4 x CH_2), 1.00-0.82 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 214.3, 138.9, 126.5, 105.6, 90.2, 77.6, 73.4, 33.9, 33.4, 30.9, 29.9, 29.5, 23.0, 22.5, 22.0, 19.3, 13.9, 13.6; **IR** (neat): *v* = 2955, 2927, 2860, 2216, 1930, 1720, 1463, 1378, 1326, 1296, 1255, 1037 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 242 (M^+, 2.18), 129 (100); **HRMS** (EI) calcd for $C_{18}H_{26}$ [M^+]: 242.2029, found: 242.2032.

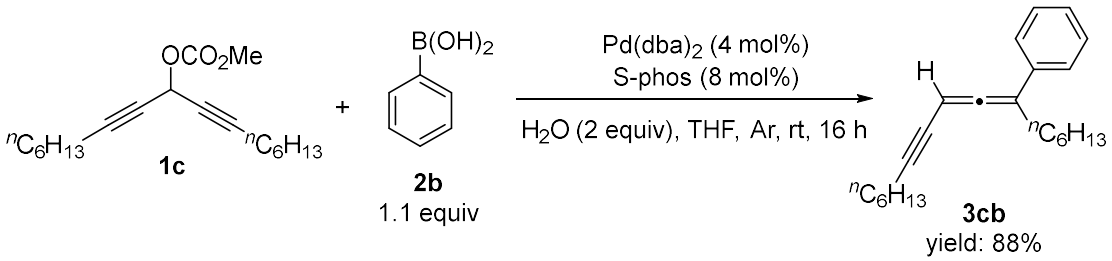
(19) Synthesis of 6-phenylhepta-4,5-dien-2-yne (3bb, wj-3-107)



Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (134.3 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1b** (166.1 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3bb** (123.2 mg, 73%) [eluent: petroleum ether (800 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.39 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.33 (t, *J* = 7.6 Hz, 2 H, Ar-H), 7.27-7.18 (m, 1 H, Ar-H), 5.70-5.62 (m, 1 H, CH), 2.13 (d, *J* = 2.4 Hz, 3 H, CH_3), 1.94

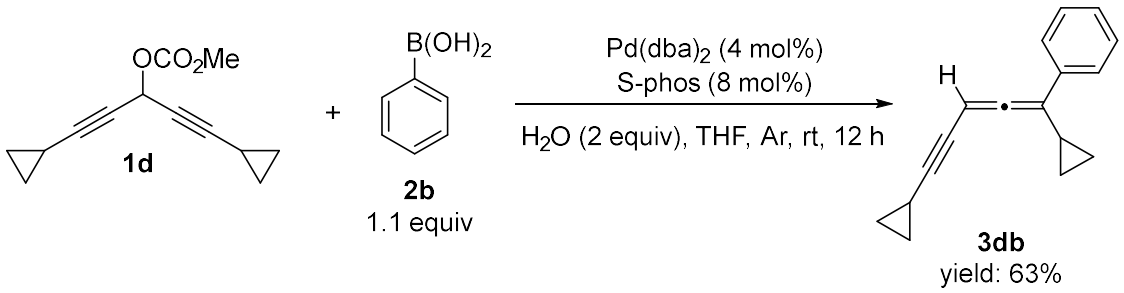
(d, J = 2.0 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.3, 135.7, 128.4, 127.2, 126.1, 102.6, 86.5, 77.8, 72.1, 16.7, 4.4; **IR** (neat): ν = 2971, 2915, 2220, 1940, 1750, 1597, 1493, 1442, 1371, 1260, 1067, 1026 cm^{-1}; **MS** (70 eV, EI) m/z: 168 (M^+, 60.44), 152 (100); **HRMS** (EI) calcd for $C_{13}H_{12}$ [M^+]: 168.0934, found: 168.0932.

(20) Synthesis of 11-phenylheptadeca-9,10-dien-7-yne (3cb, wj-3-085)



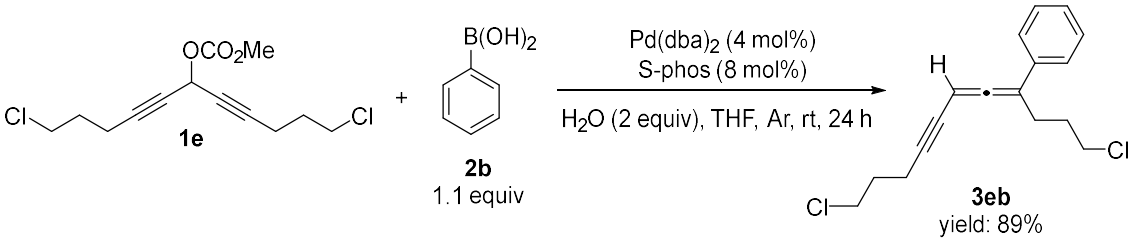
Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (134.2 mg, 1.1 mmol), $Pd(dba)_2$ (23.1 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1c** (306.6 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3cb** (281.1 mg, 88%) [eluent: petroleum ether (500 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.39 (d, J = 7.6 Hz, 2 H, Ar-H), 7.32 (t, J = 7.4 Hz, 2 H, Ar-H), 7.21 (t, J = 7.2 Hz, 1 H, Ar-H), 5.78-5.66 (m, 1 H, CH), 2.54-2.37 (m, 2 H, CH_2), 2.30 (t, J = 7.0 Hz, 2 H, CH_2), 1.64-1.46 (m, 4 H, 2 x CH_2), 1.46-1.18 (m, 12 H, 6 x CH_2), 0.96-0.79 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.1, 135.7, 128.4, 127.1, 126.4, 107.7, 91.1, 79.0, 72.9, 31.7, 31.3, 29.9, 29.0, 28.7, 28.6, 27.6, 22.7, 22.5, 19.6, 14.1, 14.0; **IR** (neat): ν = 2955, 2926, 2856, 2218, 1938, 1597, 1493, 1453, 1378, 1073, 1029 cm^{-1}; **MS** (70 eV, EI) m/z: 308 (M^+, 1.86), 167 (100); **HRMS** (EI) calcd for $C_{23}H_{32}$ [M^+]: 308.2499, found: 308.2502.

(21) Synthesis of 5-phenyl-1,5-dicyclopropylpenta-3,4-dien-1-yne (3db, wj-3-086)



Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (134.2 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1d** (218.1 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 µL) afforded the product **3db** (163.2 mg, 63%, purity: 85%) [eluent: petroleum ether (300 mL) to petroleum ether / ethyl acetate = 150:1 (300 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.55 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.34 (t, *J* = 7.6 Hz, 2 H, Ar-H), 7.27-7.20 (m, 1 H, Ar-H), 5.71 (s, 1 H, CH), 1.64-1.54 (m, 1 H, CH), 1.39-1.28 (m, 1 H, CH), 0.95-0.83 (m, 2 H, CH_2), 0.83-0.74 (m, 2 H, CH_2), 0.74-0.67 (m, 2 H, CH_2), 0.63-0.51 (m, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.8, 135.9, 128.4, 127.3, 126.6, 111.1, 93.8, 80.1, 68.0, 10.8, 8.4, 7.1, 6.9, 0.3; **IR** (neat): *v* = 3082, 3006, 2214, 1932, 1597, 1492, 1449, 1424, 1215, 1051, 1026 cm^{-1}; **MS** (70 eV, EI) *m/z*: 220 (M^+, 8.66), 191 (100); **HRMS** (EI) calcd for $C_{17}H_{16}$ [M^+]: 220.1247, found: 220.1250.

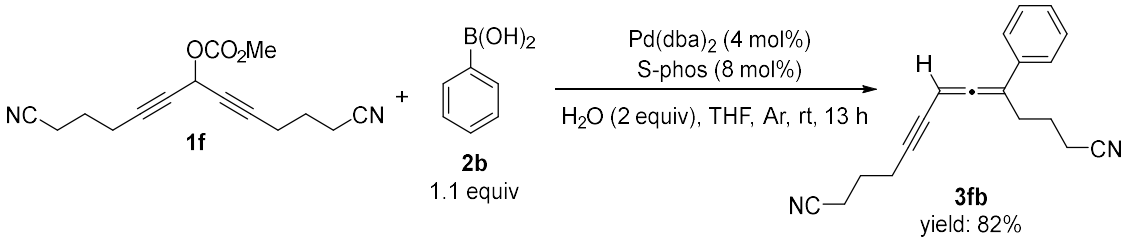
(22) Synthesis of 8-phenyl-1,11-dichloroundeca-6,7-dien-4-yne (3eb, wj-3-083)



Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (134.3 mg, 1.1 mmol), $Pd(dba)_2$ (22.9 mg, 0.04 mmol), S-phos (32.8 mg, 0.08 mmol), methyl carbonate **1e** (291.3 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 µL) afforded the product **3eb** (271.7 mg, 89%) [eluent: petroleum ether / ethyl acetate = 100:1 (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.45-7.30 (m, 4 H, Ar-H), 7.29-7.22 (m, 1 H, Ar-H), 5.83-5.69 (m, 1 H, CH), 3.76-3.59 (m, 4 H, 2 x CH_2), 2.73-2.57 (m, 2 H,

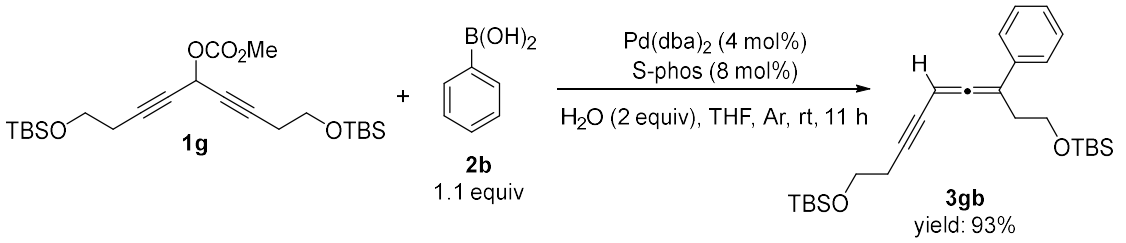
CH_2), 2.51 (t, J = 6.8 Hz, 2 H, CH_2), 2.12-1.92 (m, 4 H, 2 x CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.9, 134.9, 128.6, 127.5, 126.3, 106.7, 89.4, 79.6, 73.6, 44.4, 43.7, 31.3, 30.4, 26.9, 17.0; **IR** (neat): v = 2960, 2217, 1937, 1597, 1493, 1441, 1289, 1073, 1030 cm^{-1}; **MS** (ESI) m/z: 293 ($M(^{35}Cl_2)+H^+$); **HRMS** (ESI) calcd for $C_{17}H_{19}{}^{35}Cl_2$ [$M(^{35}Cl_2)+H^+$]: 293.0858, found: 293.0854.

(23) Synthesis of 5-phenyltrideca-5,6-dien-8-ynedinitrile (3fb, wj-3-106)



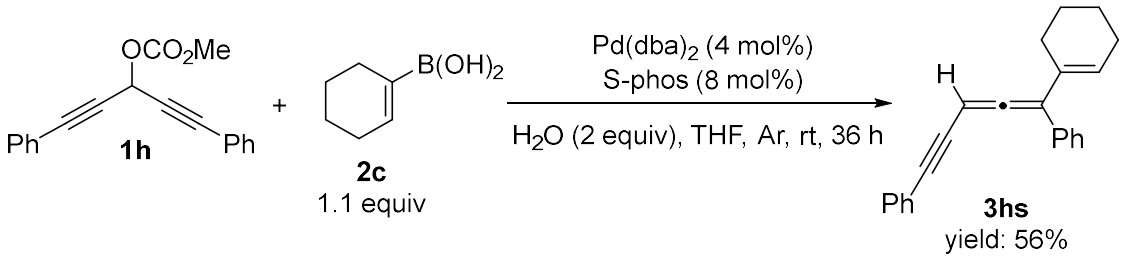
Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (134.0 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1f** (272.5 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3fb** (225.3 mg, 82%) [eluent: petroleum ether / ethyl ether / dichloromethane = 10:1:1 (320 mL) to 5:1:1 (700 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.42-7.32 (m, 4 H, Ar-H), 7.31-7.23 (m, 1 H, Ar-H), 5.83-5.74 (m, 1 H, CH), 2.73-2.58 (m, 2 H, CH_2), 2.56-2.39 (m, 6 H, 3 x CH_2), 2.02-1.83 (m, 4 H, 2 x CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.8, 134.3, 128.7, 127.8, 126.3, 119.4, 119.1, 106.4, 88.7, 79.7, 74.3, 28.5, 24.5, 23.4, 18.6, 16.5, 16.2; **IR** (neat): v = 2942, 2246, 1939, 1597, 1493, 1450, 1425, 1311, 1073, 1029 cm^{-1}; **MS** (70 eV, EI) m/z: 274 (M^+, 27.41), 220 (100); **HRMS** (EI) calcd for $C_{19}H_{18}N_2$ [M^+]: 274.1465, found: 274.1466.

(24) Synthesis of 7-phenylnona-5,6-dien-3-yn-1,9-diyl di(tert-butyldimethylsilyl) ether (3gb, wj-3-084)



Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (134.2 mg, 1.1 mmol), $Pd(dba)_2$ (23.0 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1g** (454.5 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3gb** (424.0 mg, 93%) [eluent: petroleum ether / ethyl ether / dichloromethane = 100:1:1 (500 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.39 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.32 (t, *J* = 7.4 Hz, 2 H, Ar-H), 7.22 (t, *J* = 7.2 Hz, 1 H, Ar-H), 5.75-5.64 (m, 1 H, CH), 3.81 (t, *J* = 7.2 Hz, 2 H, CH_2), 3.74 (t, *J* = 7.0 Hz, 2 H, CH_2), 2.75-2.65 (m, 2 H, CH_2), 2.57-2.48 (m, 2 H, CH_2), 0.89 (s, 18 H, 6 x CH_3), 0.07 (s, 6 H, 2 x CH_3), 0.04 (s, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.3, 135.1, 128.4, 127.3, 126.4, 104.7, 88.1, 78.8, 73.9, 61.9, 61.7, 33.3, 25.91, 25.88, 24.0, 18.33, 18.27, -5.28, -5.32; **IR** (neat): *v* = 2955, 2928, 2856, 2220, 1940, 1471, 1388, 1253, 1097, 1006 cm^{-1}; **MS** (70 eV, EI) *m/z*: 456 (M^+, 1.59), 73 (100); **HRMS** (EI) calcd for $C_{27}H_{44}O_2Si_2$ [M^+]: 456.2874, found: 456.2878.

(25) Synthesis of 5-(1-cyclohexenyl)-1,5-diphenylpenta-3,4-dien-1-yne (3hs, wj-3-056)

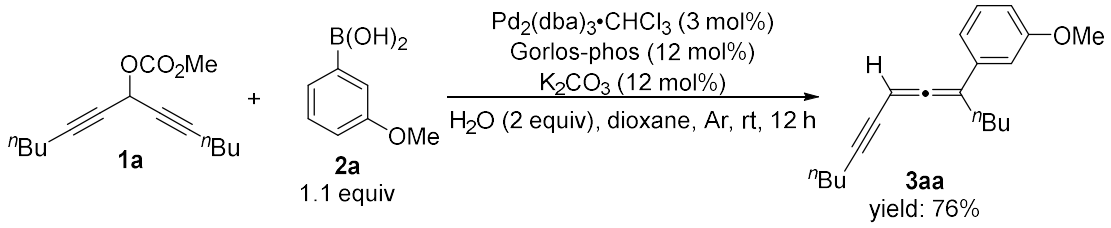


Following **Typical Procedure IV**: the reaction of cyclohex-1-en-1-ylboronic acid **2s** (138.5 mg, 1.1 mmol), $Pd(dba)_2$ (22.9 mg, 0.04 mmol), S-phos (32.9 mg, 0.08 mmol), methyl carbonate **1h** (290.5 mg, 1.0 mmol)/THF (5 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3hs** (165.3 mg, 56%) [eluent: petroleum ether / ethyl ether / dichloromethane = 300:1:1 (450 mL) to 200:1:1 (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.49-7.39 (m, 2 H, Ar-H), 7.38-7.24 (m, 8 H, Ar-H), 5.96 (s, 1 H, CH), 5.72-5.62 (m, 1 H, CH), 2.33-2.16 (m, 2 H, CH_2), 2.16-2.04 (m, 2 H, CH_2), 1.79-1.68 (m, 2 H, CH_2), 1.67-1.58 (m, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 214.2, 135.3,

131.9, 131.5, 129.3, 128.7, 128.3, 128.2, 128.0, 127.5, 123.5, 113.8, 89.8, 82.5, 78.4, 27.5, 26.0, 22.8, 22.1; **IR** (neat): ν = 3056, 2928, 2247, 1920, 1596, 1489, 1443, 1071, 1027 cm^{-1}; **MS** (70 eV, EI) *m/z*: 296 (M^+, 100); **HRMS** (EI) calcd for $C_{23}H_{20}$ [M^+]: 296.1560, found: 296.1562.

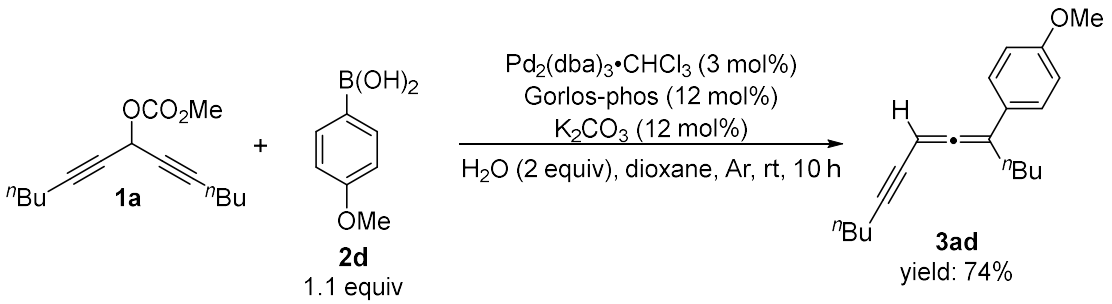
Synthesis of conjugated allenynes with Gorlos-phos:

(1) Synthesis of 9-(3-methoxylphenyl)trideca-7,8-dien-5-yne (3aa, wj-5-054)



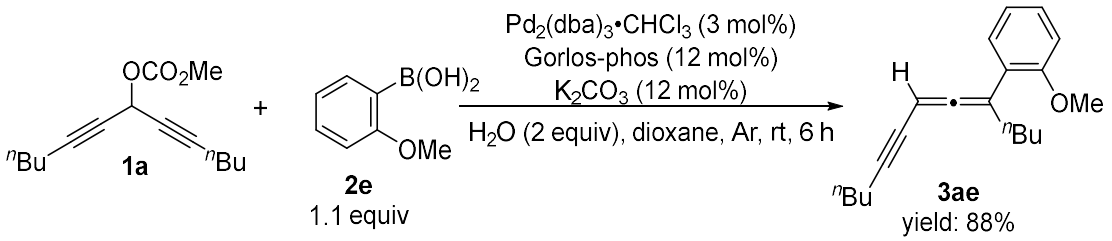
Typical Procedure V: To a Schlenk tube were added 3-methoxyphenylboronic acid **2a** (167.4 mg, 1.1 mmol), $Pd_2(dba)_3$•$CHCl_3$ (31.1 mg, 0.03 mmol), Gorlos-phos (57.3 mg, 0.12 mmol), and K_2CO_3 (16.6 mg, 0.12 mmol). After adding all of solid chemicals, the flask was degassed and refilled with Ar for three times. Then methyl carbonate **1a** (250.3 mg, 1.0 mmol)/dioxane (5 mL), and H_2O (2.0 mmol, 36 μL) were added sequentially. After that, the Ar gas line was closed and the resulting mixture was stirred at room temperature for 12 h as monitored by TLC, diluted with ethyl acetate (5 mL), filtrated through a short column of silica gel (3 cm) eluted with ethyl acetate (20 mL), and evaporated. The resulting residue was purified by chromatography on silica gel to afford the product **3aa** (215.1 mg, 76%) [eluent: petroleum ether / dichloromethane = 10:1 (550 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.24 (t, *J* = 8.0 Hz, 1 H, Ar-H), 6.99 (d, *J* = 7.6 Hz, 1 H, Ar-H), 6.94 (t, *J* = 2.0 Hz, 1 H, Ar-H), 6.77 (dd, J_1 = 8.0 Hz, J_2 = 2.2 Hz, 1 H, Ar-H), 5.71 (quint, *J* = 2.6 Hz, 1 H, CH), 3.81 (s, 3 H, CH_3), 2.52-2.37 (m, 2 H, CH_2), 2.30 (td, J_1 = 7.0 Hz, J_2 = 2.4 Hz, 2 H, CH_2), 1.59-1.47 (m, 4 H, 2 x CH_2), 1.47-1.35 (m, 4 H, 2 x CH_2), 0.98-0.86 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.1, 159.7, 137.3, 129.3, 119.0, 112.37, 112.35, 107.6, 91.1, 79.0, 72.8, 55.2, 30.8, 29.8, 29.7, 22.4, 22.0, 19.3, 13.9, 13.6.

(2) Synthesis of 9-(4-methoxylphenyl)trideca-7,8-dien-5-yne (3ad, wj-5-059)



Following **Typical Procedure V**, the reaction of 4-methoxyphenylboronic acid **2d** (172.4 mg, 1.1 mmol), $Pd_2(dba)_3$•$CHCl_3$ (31.0 mg, 0.03 mmol), Gorlos-phos (57.4 mg, 0.12 mmol), K_2CO_3 (16.6 mg, 0.12 mmol), methyl carbonate **1a** (250.0 mg, 1.0 mmol)/dioxane (5.0 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ad** (209.6 mg, 74%) [eluent: petroleum ether / dichloromethane = 6:1 (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.35-7.27 (m, 2 H, Ar-H), 6.90-6.82 (m, 2 H, Ar-H), 5.69 (quint, J = 2.6 Hz, 1 H, CH), 3.80 (s, 3 H, CH_3), 2.50-2.35 (m, 2 H, CH_2), 2.30 (td, J_1 = 7.0 Hz, J_2 = 2.4 Hz, 2 H, CH_2), 1.58-1.47 (m, 4 H, 2 x CH_2), 1.47-1.36 (m, 4 H, 2 x CH_2), 0.98-0.87 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.7, 158.8, 127.8, 127.5, 113.9, 107.2, 90.6, 78.8, 73.2, 55.3, 30.8, 29.8, 29.7, 22.4, 22.0, 19.3, 13.9, 13.6.

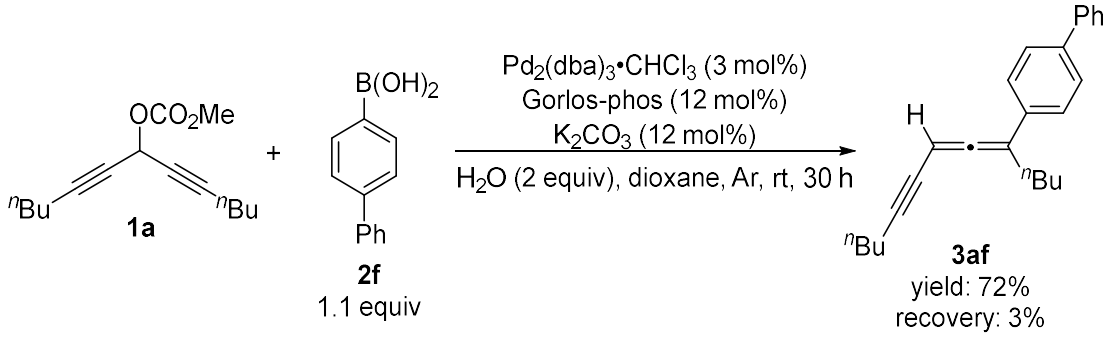
(3) Synthesis of 9-(2-methoxylphenyl)trideca-7,8-dien-5-yne (3ae, wj-5-072)



Following **Typical Procedure V**, the reaction of 2-methoxyphenylboronic acid **2e** (167.1 mg, 1.1 mmol), $Pd_2(dba)_3$•$CHCl_3$ (31.2 mg, 0.03 mmol), Gorlos-phos (57.5 mg, 0.12 mmol), K_2CO_3 (16.6 mg, 0.12 mmol), methyl carbonate **1a** (250.4 mg, 1.0 mmol)/dioxane (5.0 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3ae** (247.3 mg, 88%) [eluent: petroleum ether / dichloromethane = 10:1 (440 mL)]; oil; **^{1}H NMR**

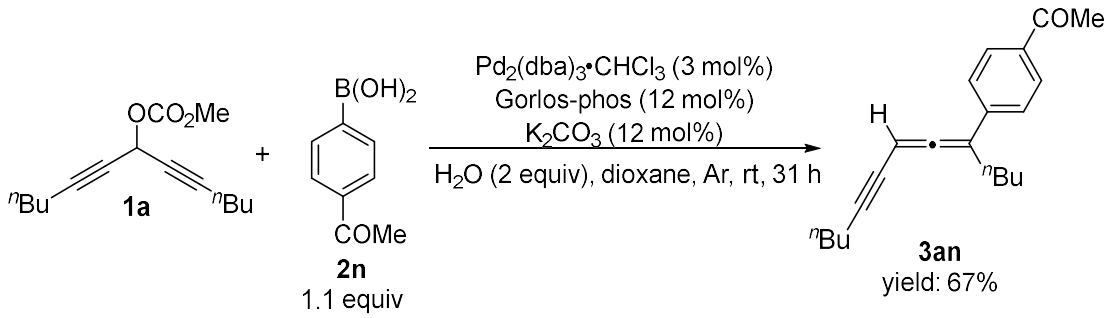
(400 MHz, $CDCl_3$): δ = 7.27-7.18 (m, 2 H, Ar-H), 6.92 (td, J_1 = 7.5 Hz, J_2 = 1.2 Hz, 1 H, CH), 6.87 (d, J = 8.0 Hz, 1 H, Ar-H), 5.48 (quint, J = 2.6 Hz, 1 H, CH), 3.82 (s, 3 H, CH_3), 2.49-2.39 (m, 2 H, CH_2), 2.31 (td, J_1 = 7.1 Hz, J_2 = 2.4 Hz, 2 H, CH_2), 1.58-1.31 (m, 8 H, 4 x CH_2), 0.98-0.84 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.6, 156.9, 129.6, 128.5, 126.0, 120.6, 111.3, 105.1, 90.4, 75.7, 73.7, 55.6, 31.8, 30.9, 29.8, 22.3, 22.0, 19.3, 13.9, 13.6.

(4) Synthesis of 9-(4-phenylphenyl)trideca-7,8-dien-5-yne (3af, wj-5-060)



Following **Typical Procedure V**, the reaction of 4-phenylphenylboronic acid **2f** (217.8 mg, 1.1 mmol), $Pd_2(dba)_3$•$CHCl_3$ (31.1 mg, 0.03 mmol), Gorlos-phos (57.4 mg, 0.12 mmol), K_2CO_3 (16.6 mg, 0.12 mmol), methyl carbonate **1a** (250.3 mg, 1.0 mmol)/dioxane (5.0 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3af** (235.4 mg, 72%) [eluent: petroleum ether: 700 mL]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.61-7.52 (m, 4 H, Ar-H), 7.49-7.38 (m, 4 H, Ar-H), 7.36-7.29 (m, 1 H, Ar-H), 5.75 (quint, J = 2.6 Hz, 1 H, CH), 2.57-2.41 (m, 2 H, CH_2), 2.30 (td, J_1 = 7.0 Hz, J_2 = 2.4 Hz, 2 H, CH_2), 1.64-1.35 (m, 8 H, 4 x CH_2), 0.99-0.87 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.3, 140.7, 139.9, 134.6, 128.8, 127.2, 127.1, 126.9, 126.8, 107.4, 91.2, 79.2, 72.9, 30.8, 29.8, 29.6, 22.4, 22.0, 19.3, 13.9, 13.6.

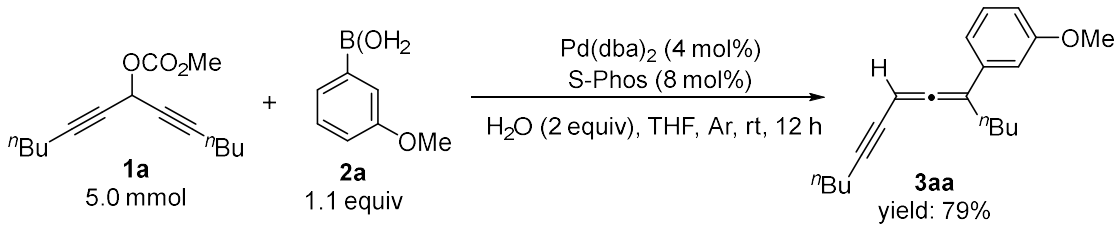
(5) Synthesis of 9-(4-acetylphenyl)trideca-7,8-dien-5-yne (3an, wj-5-073)



Following **Typical Procedure V**, the reaction of 4-acetylphenylboronic acid **2n** (180.4 mg, 1.1 mmol), $Pd_2(dba)_3$•$CHCl_3$ (31.1 mg, 0.03 mmol), Gorlos-phos (57.5 mg, 0.12 mmol), K_2CO_3 (16.6 mg, 0.12 mmol), methyl carbonate **1a** (250.3 mg, 1.0 mmol)/dioxane (5.0 mL), and H_2O (2.0 mmol, 36 μL) afforded the product **3an** (196.3 mg, 67%) [eluent: petroleum ether / dichloromethane / ethyl ether = 25:1:1 (500 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.91 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.47 (d, *J* = 8.4 Hz, 2 H, Ar-H), 5.79 (quint, *J* = 2.6 Hz, 1 H, CH), 2.59 (s, 3 H, CH_3), 2.55-2.41 (m, 2 H, CH_2), 2.32 (td, J_1 = 7.1 Hz, J_2 = 2.4 Hz, 2 H, CH_2), 1.60-1.48 (m, 4 H, 2 x CH_2), 1.48-1.36 (m, 4 H, 2 x CH_2), 1.00-0.88 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 214.0, 197.5, 140.8, 135.6, 128.5, 126.4, 107.3, 92.0, 79.7, 72.1, 30.7, 29.7, 29.4, 26.5, 22.3, 22.0, 19.3, 13.9, 13.6.

Gram scale reactions and transformations

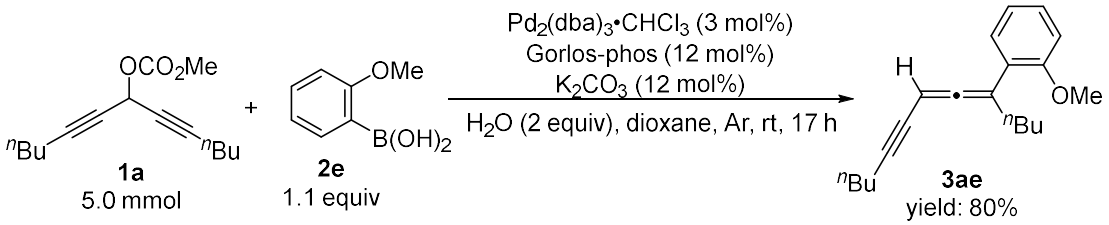
(1) Synthesis of 9-(3-methoxylphenyl)trideca-7,8-dien-5-yne (3aa, wj-3-100)



Following **Typical Procedure IV**, the reaction of 3-methoxyphenylboronic acid **2a** (836.0 mg, 5.5 mmol), $Pd(dba)_2$ (114.9 mg, 0.2 mmol), S-phos (164.1 mg, 0.4 mmol), methyl carbonate **1a** (1.2513 g, 5.0 mmol)/THF (25 mL), and H_2O (10 mmol, 180 μL) afforded the product **3aa** (1.1137 g, 79%) [eluent: petroleum ether / dichloromethane = 5:1 (480 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.24 (t, *J* = 8.0 Hz, 1 H, Ar-H), 6.99 (d, *J* = 7.6 Hz, 1 H, Ar-H), 6.94 (s, 1 H, Ar-H), 6.83-6.72 (m, 1 H, Ar-H), 5.71 (s,

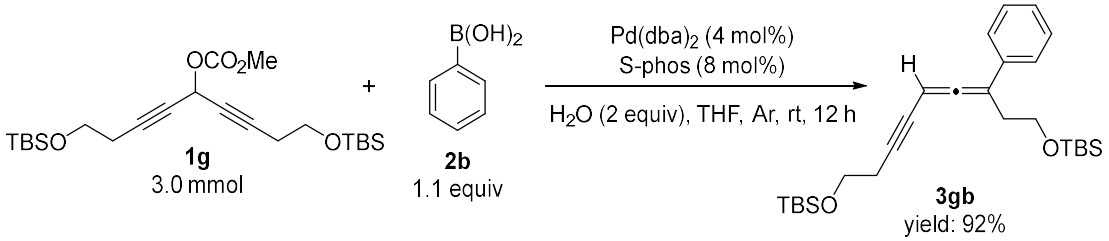
1 H, CH), 3.81 (s, 3 H, CH_3), 2.52-2.37 (m, 2 H, CH_2), 2.30 (t, J = 6.8 Hz, 2 H, CH_2), 1.62-1.47 (m, 4 H, 2 x CH_2), 1.47-1.35 (m, 4 H, 2 x CH_2), 1.00-0.84 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.1, 159.7, 137.3, 129.3, 119.0, 112.37, 112.35, 107.6, 91.1, 79.0, 72.8, 55.2, 30.8, 29.8, 29.7, 22.4, 22.0, 19.3, 13.9, 13.6.

(2) Synthesis of 9-(2-methoxylphenyl)trideca-7,8-dien-5-yne (3ae, wj-5-095)



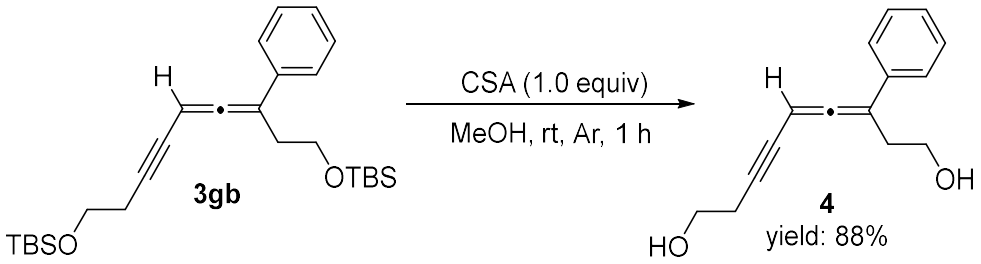
Following **Typical Procedure V**, the reaction of 2-methoxyphenylboronic acid (835.9 mg, 5.5 mmol), $Pd_2(dba)_3$•$CHCl_3$ (155.4 mg, 0.15 mmol), Gorlos-phos (287.1 mg, 0.6 mmol), K_2CO_3 (82.8 mg, 0.6 mmol), methyl carbonate **1a** (1.2517 g, 5.0 mmol)/dioxane (25.0 mL), and H_2O (10.0 mmol, 180 μL) afforded the product **3ae** (1.1289 g, 80%) [eluent: petroleum ether / dichloromethane = 10:1 (770 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.28-7.18 (m, 2 H, Ar-H), 6.92 (t, J = 7.6 Hz, 1 H, CH), 6.87 (d, J = 8.4 Hz, 1 H, Ar-H), 5.48 (quint, J = 2.5 Hz, 1 H, CH), 3.82 (s, 3 H, CH_3), 2.44 (td, J_1 = 7.8 Hz, J_2 = 2.8 Hz, 2 H, CH_2), 2.31 (td, J_1 = 6.9 Hz, J_2 = 2.4 Hz, 2 H, CH_2), 1.58-1.30 (m, 8 H, 4 x CH_2), 0.96-0.84 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 212.6, 156.9, 129.6, 128.5, 125.9, 120.6, 111.3, 105.1, 90.4, 75.7, 73.7, 55.6, 31.8, 30.9, 29.8, 22.3, 22.0, 19.3, 13.9, 13.6.

(3) Synthesis of 7-phenylnona-5,6-dien-3-yn-1,9-diyl di(*tert*-butyldimethylsilyl) ether (3gb, wj-3-138)



Following **Typical Procedure IV**, the reaction of phenylboronic acid **2b** (402.2 mg, 3.3 mmol), $Pd(dba)_2$ (69.0 mg, 0.12 mmol), S-phos (98.6 mg, 0.24 mmol), methyl carbonate (1.3647 g, 3.0 mmol)/THF (15.0 mL), and H_2O (6.0 mmol, 108 μL) afforded the product **3gb** (1.2604 g, 92%) [eluent: petroleum ether / ethyl ether / dichloromethane = 100:1:1 (900 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.38 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.32 (t, *J* = 7.6 Hz, 2 H, Ar-H), 7.22 (t, *J* = 7.2 Hz, 1 H, Ar-H), 5.70 (s, 1 H, CH), 3.81 (t, *J* = 7.0 Hz, 2 H, CH_2), 3.74 (t, *J* = 7.0 Hz, 2 H, CH_2), 2.75-2.65 (m, 2 H, CH_2), 2.52 (t, *J* = 7.0 Hz, 2 H, CH_2), 0.89 (s, 18 H, 6 x CH_3), 0.07 (s, 6 H, 2 x CH_3), 0.04 (s, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.4, 135.2, 128.4, 127.3, 126.4, 104.7, 88.1, 78.8, 73.9, 61.9, 61.7, 33.3, 25.91, 25.88, 24.0, 18.32, 18.27, -5.28, -5.31.

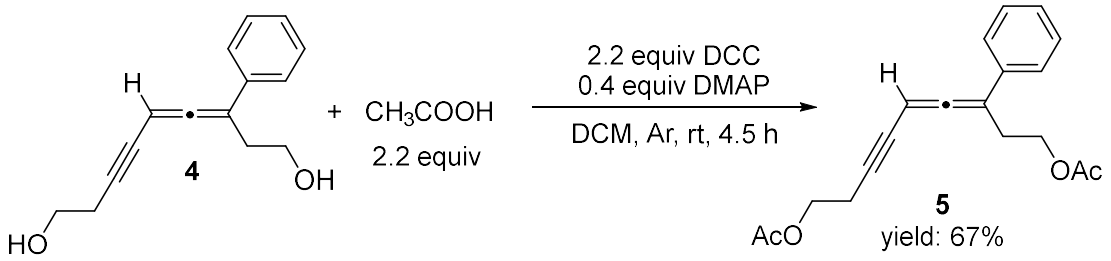
(4) Synthesis of 7-phenylnona-5,6-dien-3-yne-1,9-diol (4, wj-3-139)[3]



To a dried Schlenk tube were added **3gb** (685.4 mg, 1.5 mmol) and MeOH (7.5 mL) under Ar atmosphere. Then 10-camphorsulfonic acid (348.4 mg, 1.5 mmol) was added and the resulting mixture was stirred at room temperature for 1 h as monitored by TLC, diluted with Et_2O (3 mL), quenched with saturated $NaHCO_3$ (5 mL), extracted with Et_2O (3 x 5 mL), washed with brine, dried over Na_2SO_4, filtered, and concentrated. The resulting residue was purified by column chromatography on silica gel to afford the product diol **4** (0.3021 g, 88%) [eluent: petroleum ether / ethyl acetate = 1:1 (600 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.44-7.30 (m, 4 H, Ar-H), 7.30-7.20 (m, 1 H, Ar-H), 5.79 (s, 1 H, CH), 3.87 (t, *J* = 6.0 Hz, 2 H, CH_2), 3.73 (t, *J* = 6.0 Hz, 2 H, CH_2), 2.85-2.66 (m, 2 H, CH_2), 2.63-2.50 (m, 2 H, CH_2), 2.10-1.65 (m, 2 H, 2 x OH); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.1, 134.6, 128.6, 127.6, 126.4, 104.9, 88.2, 79.4, 74.4,

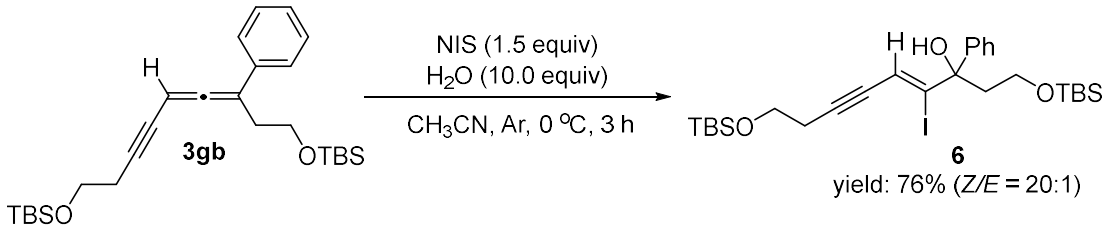
61.0, 60.8, 33.1, 23.9; **IR** (neat): ν = 3320, 2957, 2884, 2217, 1938, 1597, 1493, 1448, 1420, 1329, 1185, 1036 cm^{-1}; **MS** (70 eV, EI) *m*/*z*: 228 (M^+, 24.08), 152 (100); **HRMS** (EI) calcd for $C_{15}H_{16}O_2$ [M^+]: 228.1145, found: 228.1146.

(5) Synthesis of 7-phenylnona-5,6-dien-3-yne-1,9-diyl diacetate (5, wj-3-157)



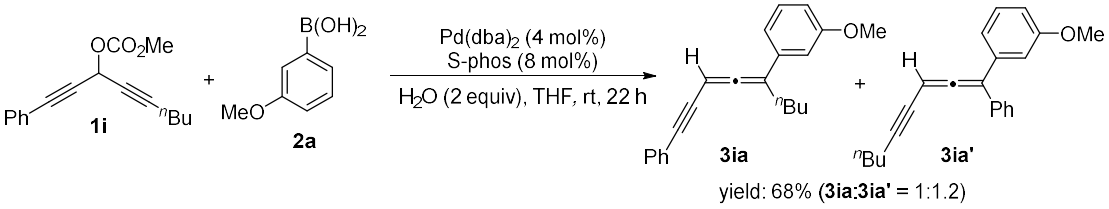
To a dried Schlenk tube were added DCC (91.0 mg, 0.44 mmol), DMAP (9.9 mg, 0.08 mol). Then the Schlenk tube was degassed and refilled with Ar for three times. Acetic acid (25 μL, d = 1.049 g/mL, 26.2 mg, 0.44 mmol) and diol **4** (45.5 mg, 0.2 mmol) in DCM (1.5 mL) were added sequentially. The resulting mixture was stirred at room temperature for 4.5 h as monitored by TLC, concentrated, diluted with cold ethyl acetate (10 mL), filtered through a short column silica gel (3 cm) eluted with cold ethyl acetate (10 mL), and concentrated. The resulting residue was purified by column chromatography on silica gel to afford the product **5** (41.5 mg, 67%) [eluent: petroleum ether / ethyl acetate = 6:1 (150 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.44-7.29 (m, 4 H, Ar-H), 7.29-7.22 (m, 1 H, Ar-H), 5.76 (s, 1 H, CH), 4.34-4.22 (m, 2 H, CH_2), 4.17 (t, *J* = 6.8 Hz, 2 H, CH_2), 2.90-2.72 (m, 2 H, CH_2), 2.65 (t, *J* = 6.4 Hz, 2 H, CH_2), 2.13-1.98 (m, 6 H, 2 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): δ = 213.1, 171.0, 170.7, 134.5, 128.6, 127.6, 126.3, 104.3, 86.9, 79.6, 74.1, 62.4, 62.2, 29.0, 20.9, 20.8, 20.0; **IR** (neat): ν = 2966, 2222, 1938, 1735, 1494, 1451, 1384, 1364, 1230, 1037 cm^{-1}; **MS** (ESI) *m*/*z*: 335 (M+Na^+); **HRMS** (ESI) calcd for $C_{19}H_{20}O_4Na$ [M+Na^+]: 335.1254, found: 335.1249.

(6) Synthesis of (*Z*)-4-iodo-1,9-di((*tert*-butyldimethylsilyl)oxy)-3-phenylnon-4-en-6-yn-3-ol (6, wj-4-017)[4]



To a Schleck tube were added **3gb** (91.5 mg, 0.2 mmol) and CH_3CN (2 mL) under Ar atmosphere. The resulting mixture was cooled to 0 °C via an ice-water bath followed by the sequential addition of NIS (69.0 mg, 0.3 mmol) and H_2O (36 μL, 2.0 mmol), stirred for 3 h as monitored by TLC, quenched with saturated sodium thiosulfate (2 mL), extracted with ethyl acetate (5 mL x 3), washed with brine, dried over Na_2SO_4, filtered, and concentrated. The resulting residue was purified by column chromatography on silica gel to afford the product **6** (91.5 mg, 76%, *Z*/*E* = 20:1) [eluent: petroleum ether / ethyl ether / dichloromethane = 50:1:1 (400 mL)]; oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.43 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.33 (t, *J* = 7.4 Hz, 2 H, Ar-H), 7.29-7.23 (m, 1 H, Ar-H), 6.73 (s, 1 H, CH), 5.50 (s, 1 H, OH), 3.94-3.74 (m, 4 H, 2 x CH_2), 2.70-2.62 (m, 1 H, one proton of CH_2), 2.59 (t, *J* = 7.0 Hz, 2 H, CH_2), 2.39-2.27 (m, 1 H, one proton of CH_2), 0.92-0.85 (m, 18 H, 6 x CH_3), 0.10-0.02 (m, 12 H, 4 x CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$): 143.4, 128.1, 127.5, 126.2, 126.1, 118.4, 93.6, 82.4, 81.3, 61.7, 61.0, 38.7, 25.9, 25.7, 24.2, 18.3, 18.0, -5.2, -5.7, -5.8; **IR** (neat): *v* = 3414, 2953, 2931, 2881, 2857, 2219, 1469, 1388, 1254, 1095, 1044 cm^{-1}; **MS** (ESI) *m/z* (%): 623 ($M+Na^+$); **HRMS** (ESI) calcd for $C_{27}H_{45}O_3INaSi_2$ [$M+Na^+$]: 623.1844, found: 623.1837.

Preliminary attempt on the non-symmetric 1,4-diyn-3-yl carbonate 1i (wj-7-142)



Following **Typical Procedure IV**, the reaction of 3-methoxyphenylboronic acid **2a** (33.5 mg, 0.22 mmol), $Pd(dba)_2$ (4.6 mg, 0.008 mmol), S-phos (6.6 mg, 0.016 mmol), methyl carbonate **1i** (53.9 mg, 0.2 mmol)/THF (1 mL), and H_2O (0.4 mmol, 7.2 μL)

afforded the product (41.1 mg, 68% (**3ia**:**3ia**' = 1:1.2)) [eluent: petroleum ether / dichloromethane = 20:1 (210 mL) to 10:1 (150 mL)]; oil; 32% NMR yield of **3ia** and 40% of **3ia**' (1:1.3) based on ^{1}H NMR analysis of the crude product.

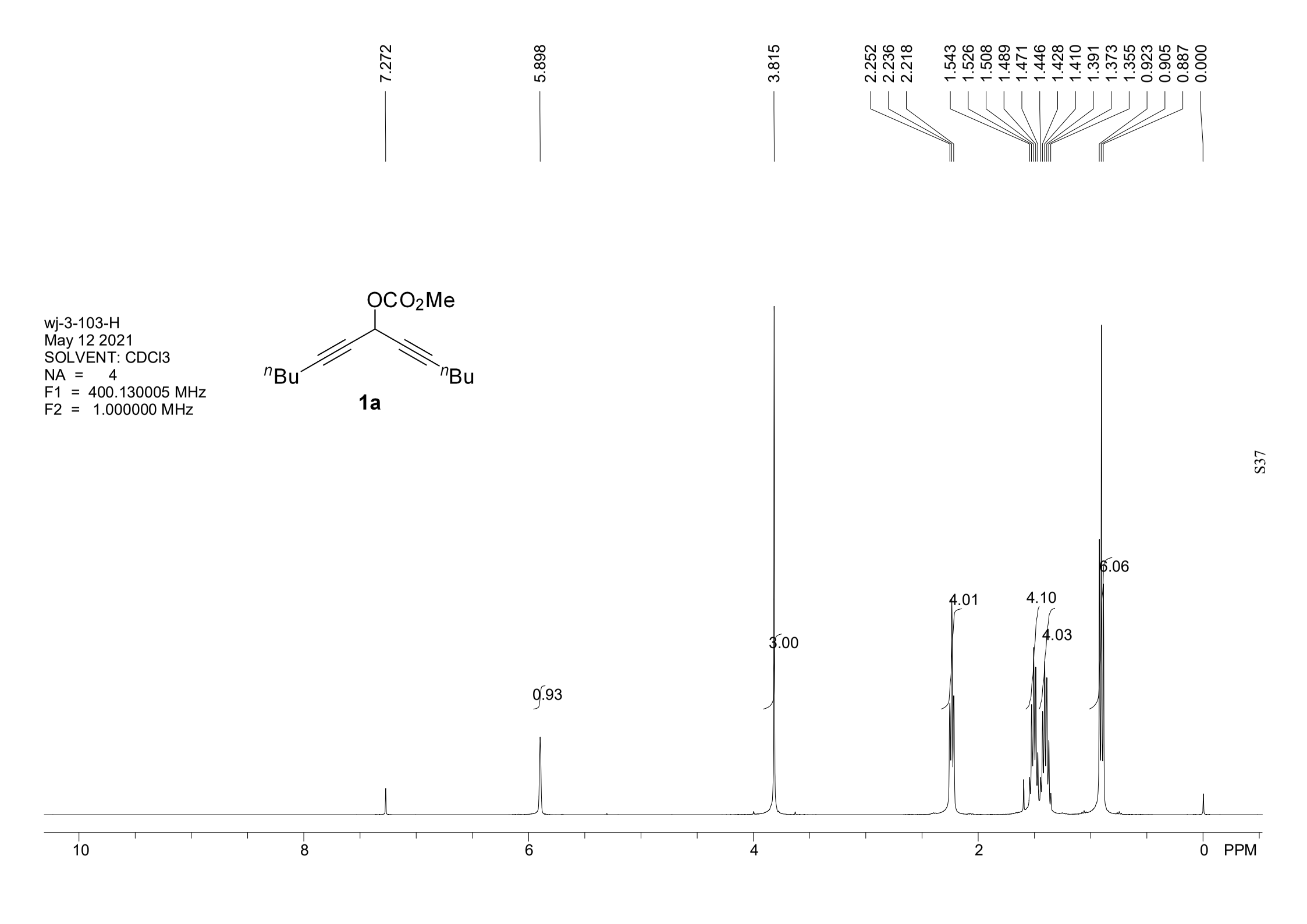
References:

1. Sawada, Y.; Furumi, S.; Takai, A.; Takeuchi, M.; Noguchi, K.; Tanaka, K. Rhodium-Catalyzed Enantioselective Synthesis, Crystal Structures, and Photophysical Properties of Helically Chiral 1,1'-Bitriphenylenes. *J. Am. Chem. Soc.* **2012**, *134*, 4080-4083.

2. Wu, P.; Jia, M.; Lin, W.; Ma, S. Matched Coupling of Propargylic Carbonates with Cyclopropanols. *Org. Lett*. **2018**, *20*, 554-557.

3. Umemiya, S.; Terada, M. Catalytic Enantioselective Allylation of Acetylenic Aldehydes by Chiral Phosphoric Acid/Transition Metal Cooperative Catalysis: Formal Synthesis of Fostriecin. *Org. Lett.* **2021**, *23*, 3767-3771.

4. Kong, W.; Guo, B.; Fu, C.; Ma, S. An Efficient Approach to 2-Bromoalken-3-ols by Regioselective Bromohydroxylation Reaction of Simple Allenes with NBS. *Eur. J. Org. Chem.* **2011**, 2278-2285.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3.815
2.252
2.236
2.218
1.543
1.526
1.508
1.489
1.471
1.446
1.428
1.410
1.391
1.373
1.355
0.923
0.905
0.887
0.000
wj-3-103-H
May 12 2021
SOLVENT: CDCl3
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F2 = 1.000000 MHz
OCO2Me
nBu
nBu
1a
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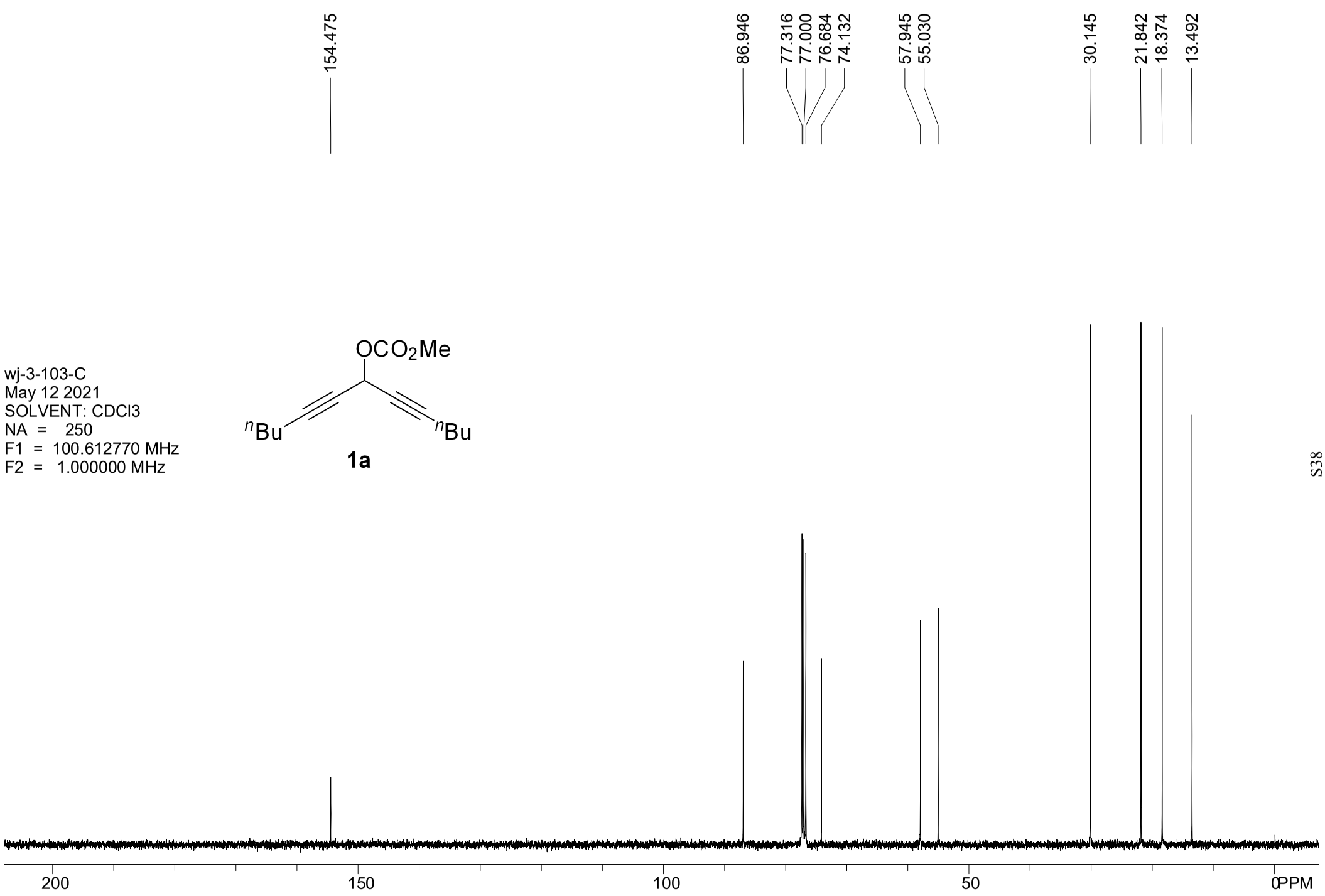
3.00
4.01
4.10
4.03
6.06
10
8
6
4
2
0
PPM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77.000
76.684
74.132
57.945
55.030
30.145
21.842
18.374
13.492
wj-3-103-C
May 12 2021
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F2 = 1.000000 MHz
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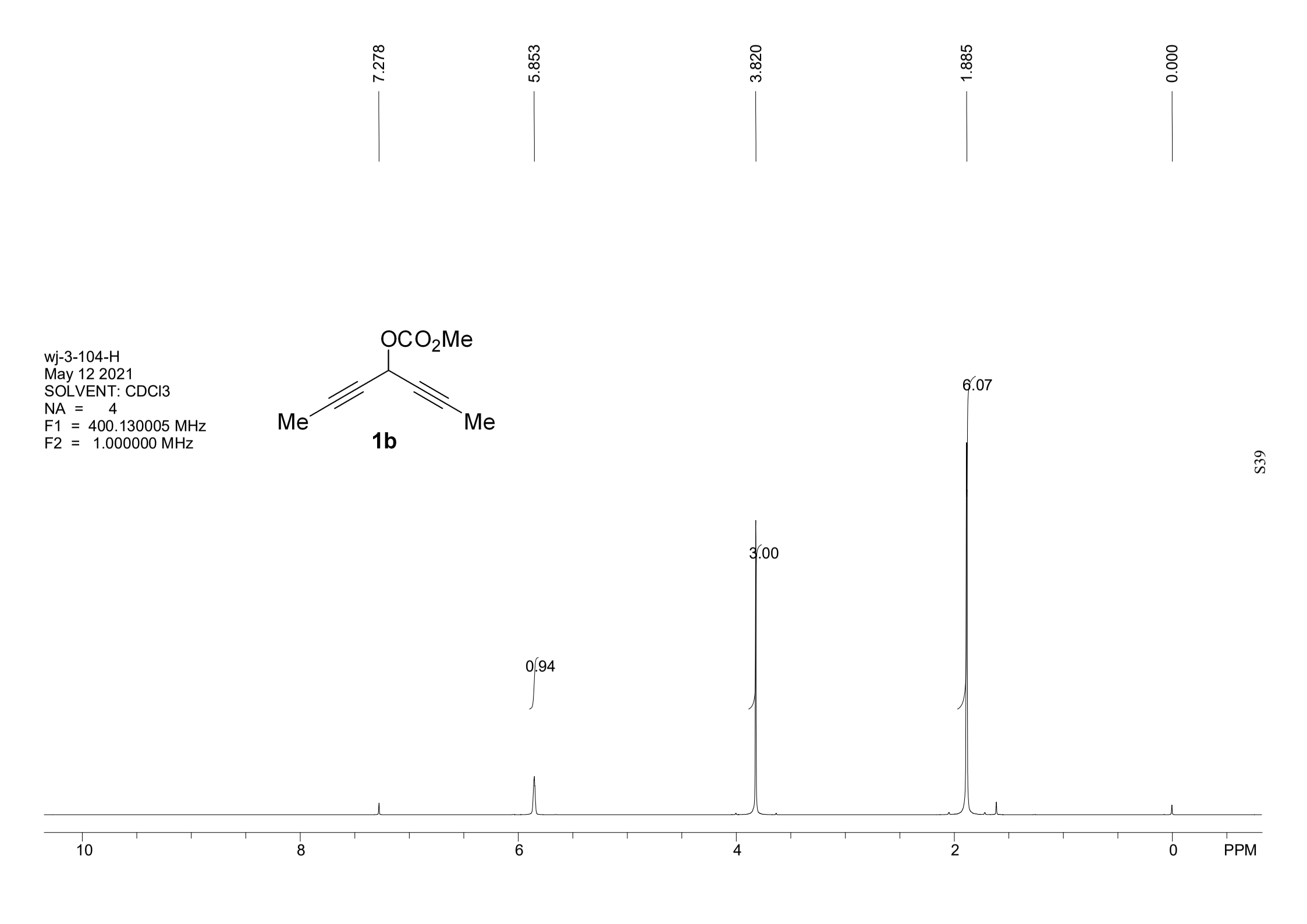
nBu
1a
200
150
100
50
0PPM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1.885
0.000
wj-3-104-H
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F2 = 1.000000 MHz
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1b
6.07
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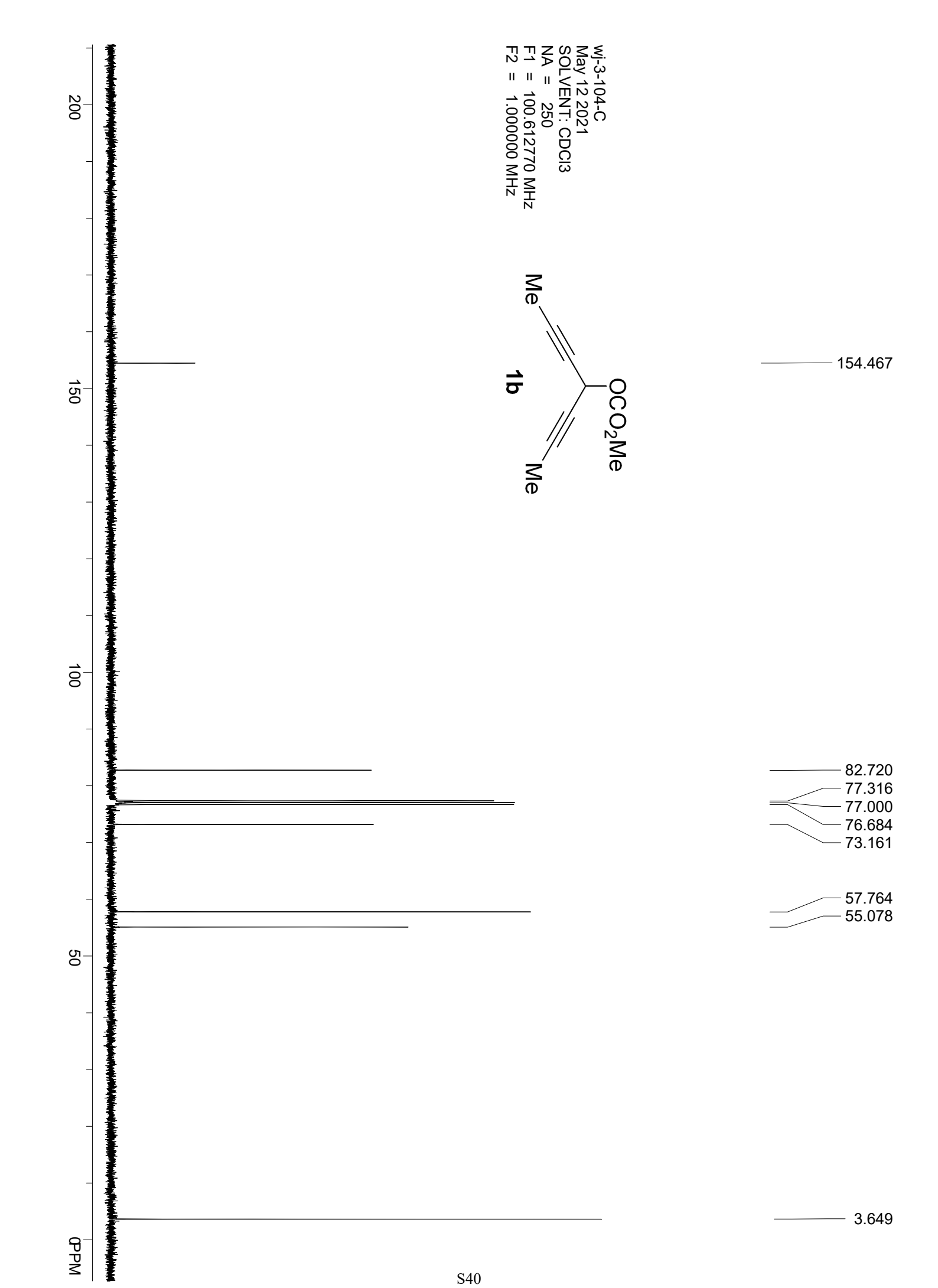
S39
10
8
6
4
2
0
PPM


wj-3-104-C
May 12 2021
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F2 = 1.000000 MHz
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Me
1b
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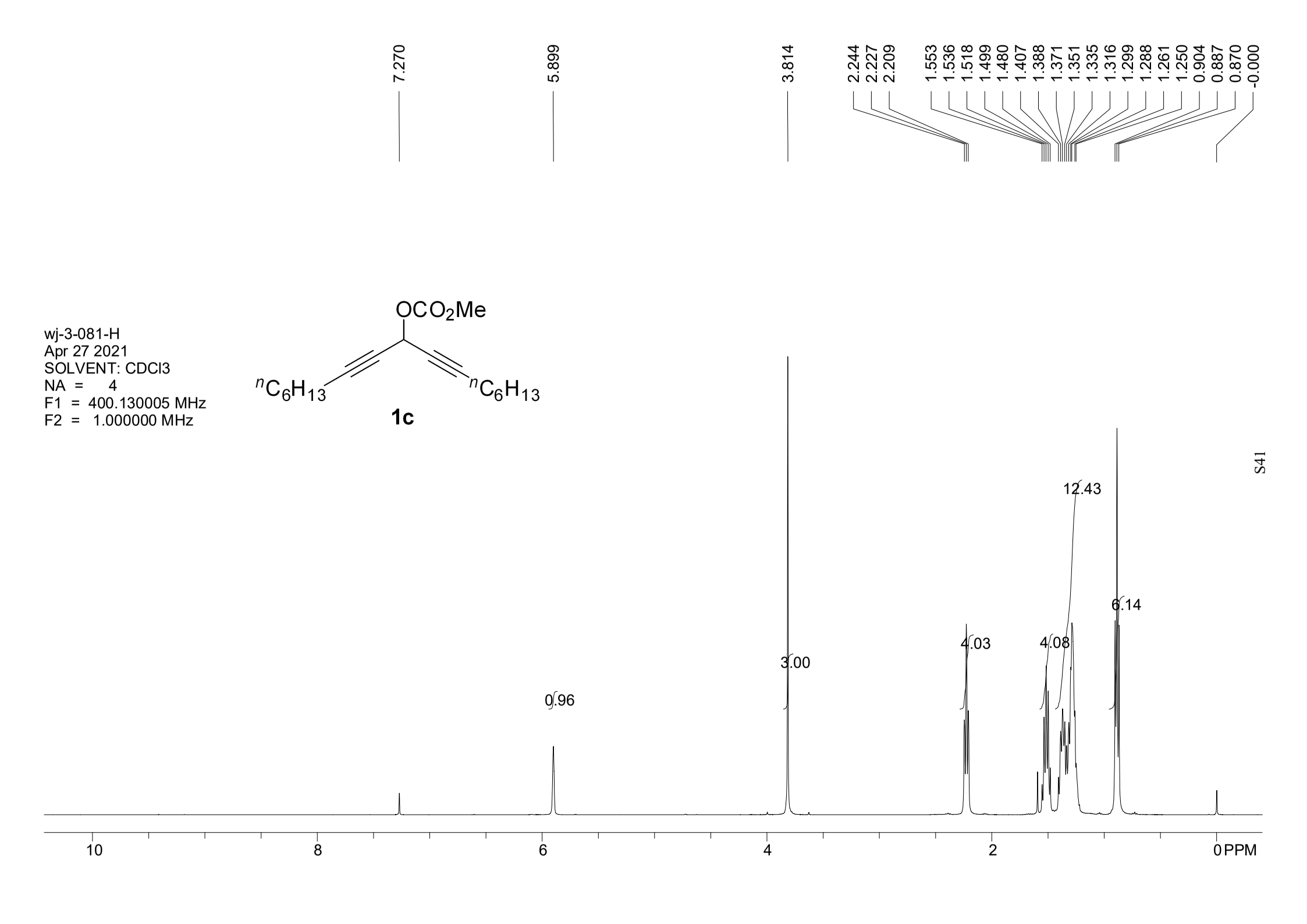
77.000
76.684
73.161
57.764
55.078
3.649
200
150
100
50
0PPM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3.814
2.244
2.227
2.209
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1.536
1.518
1.499
1.480
1.407
1.388
1.371
1.351
1.335
1.316
1.299
1.288
1.261
1.250
0.904
0.887
0.870
-0.000
wj-3-081-H
Apr 27 2021
SOLVENT: CDCl3
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F1 = 400.130005 MHz
F2 = 1.000000 MHz
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nC6H13
1c
S41
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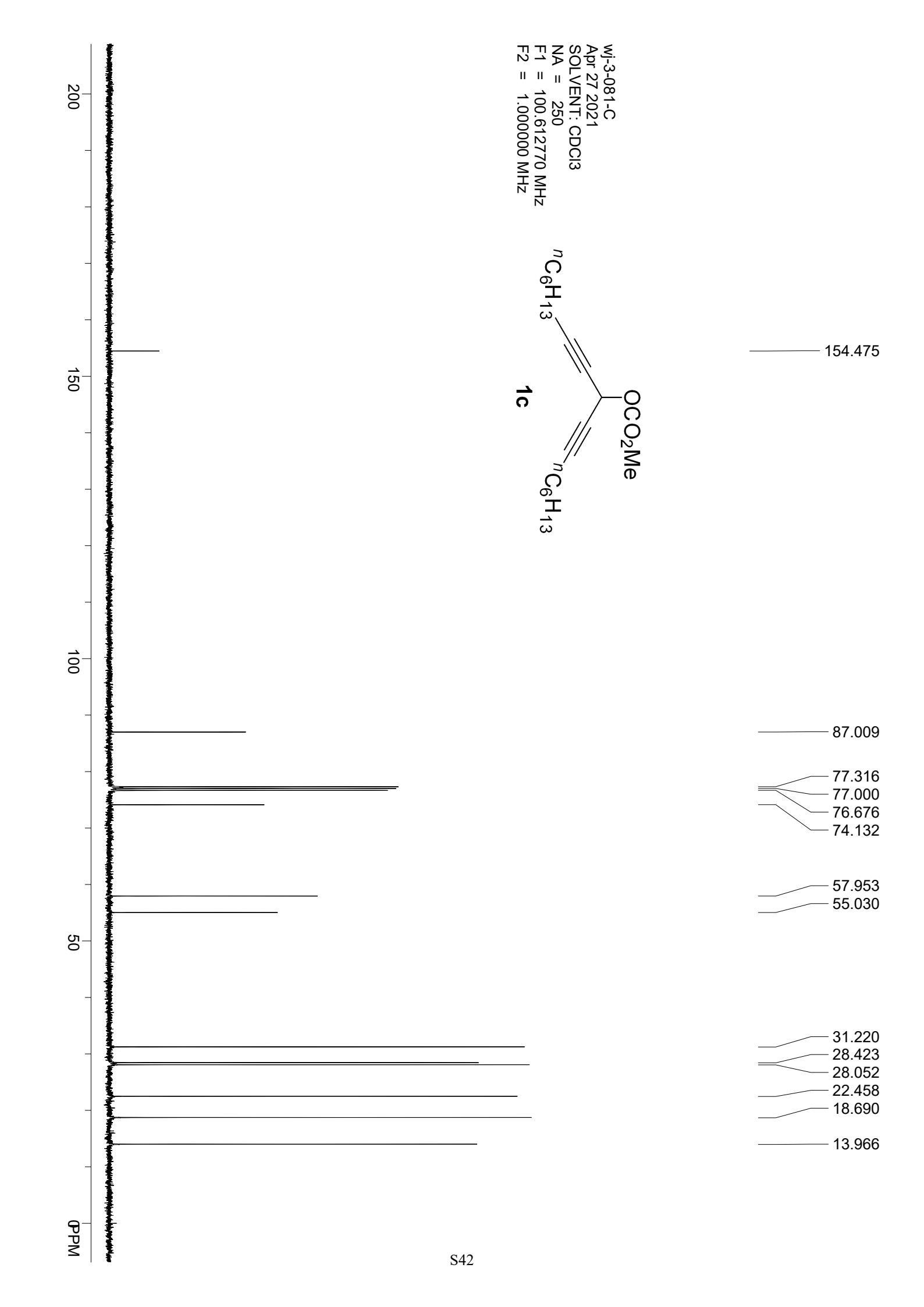
6.14
4.03
4.08
3.00
0.96
10
8
6
4
2
0PPM


wj-3-081-C
Apr 27 2021
SOLVENT: CDCl3
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F2 = 1.000000 MHz
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OCO2Me
$^{n}C_6H_{13}$
1c
154.475
87.009
77.316
77.000
76.676
74.132
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55.030
31.220
28.423
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22.458
18.690
13.966
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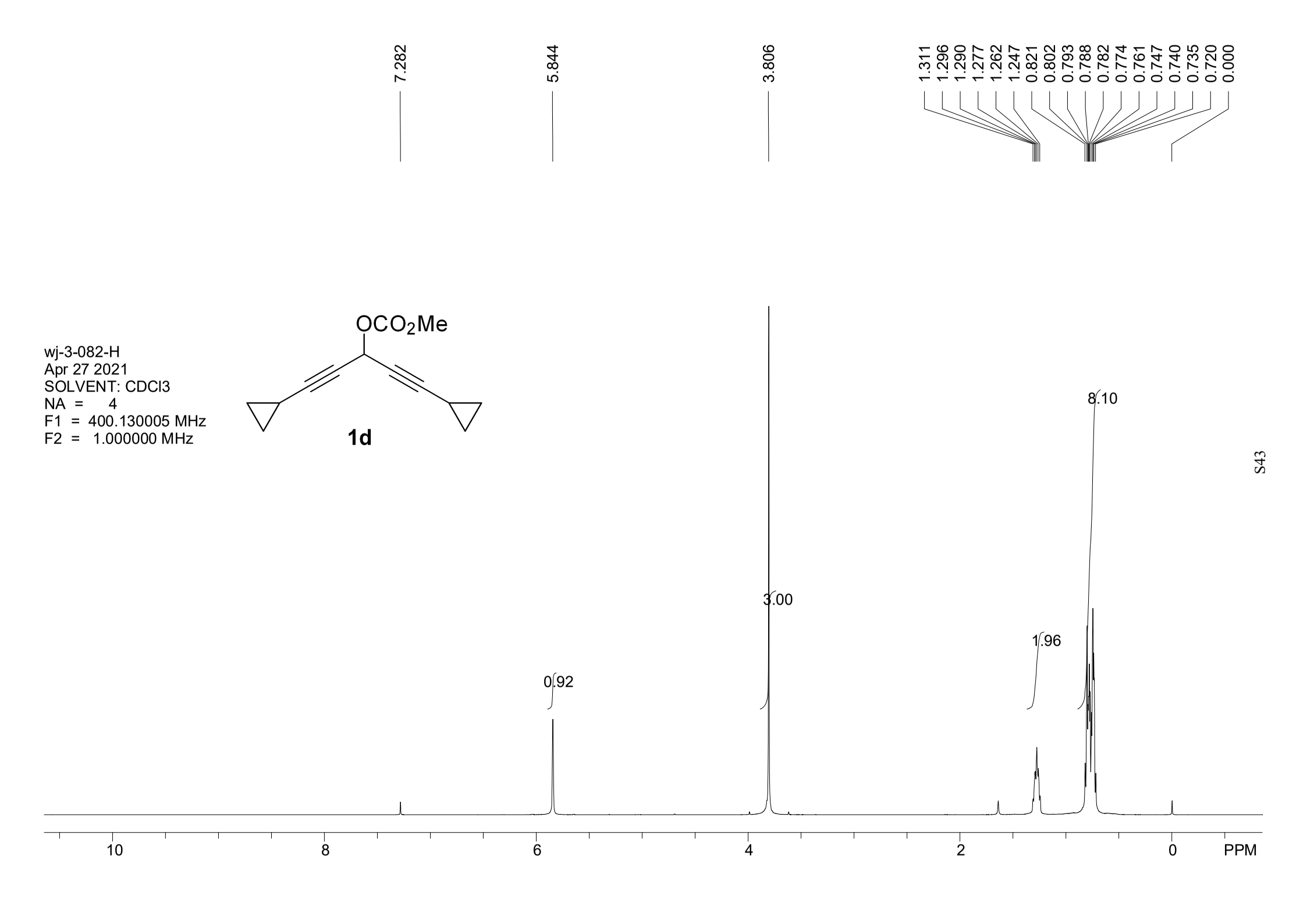
150
100
50
0
PPM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1.277
1.262
1.247
0.821
0.802
0.793
0.788
0.782
0.774
0.761
0.747
0.740
0.735
0.720
0.000
wj-3-082-H
Apr 27 2021
SOLVENT: CDCl3
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F2 = 1.000000 MHz
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S43
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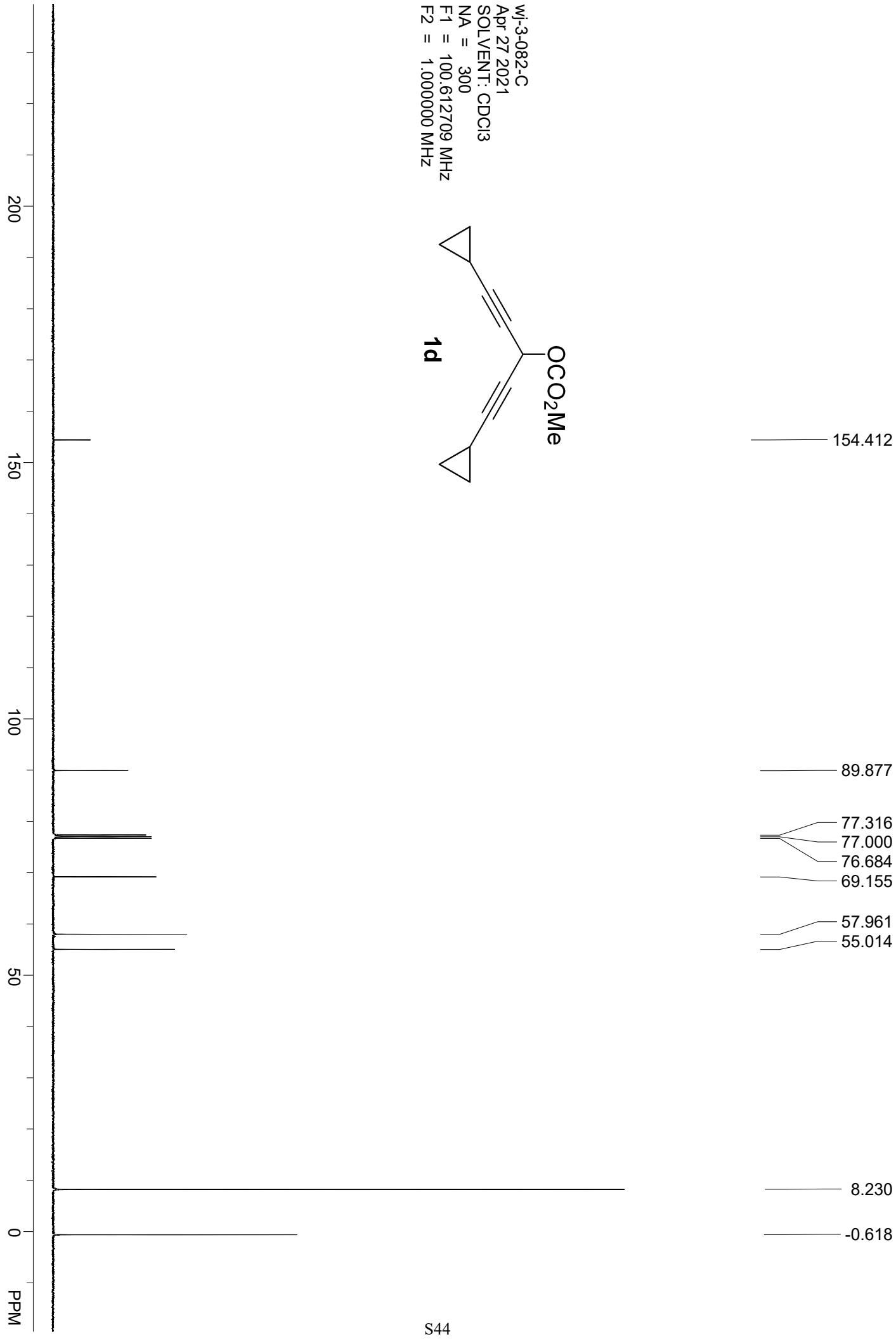
8
6
4
2
0
PPM


wj-3-082-C
Apr 27 2021
SOLVENT: CDCl3
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77.000
76.684
69.155
57.961
55.014
8.230
-0.618
200
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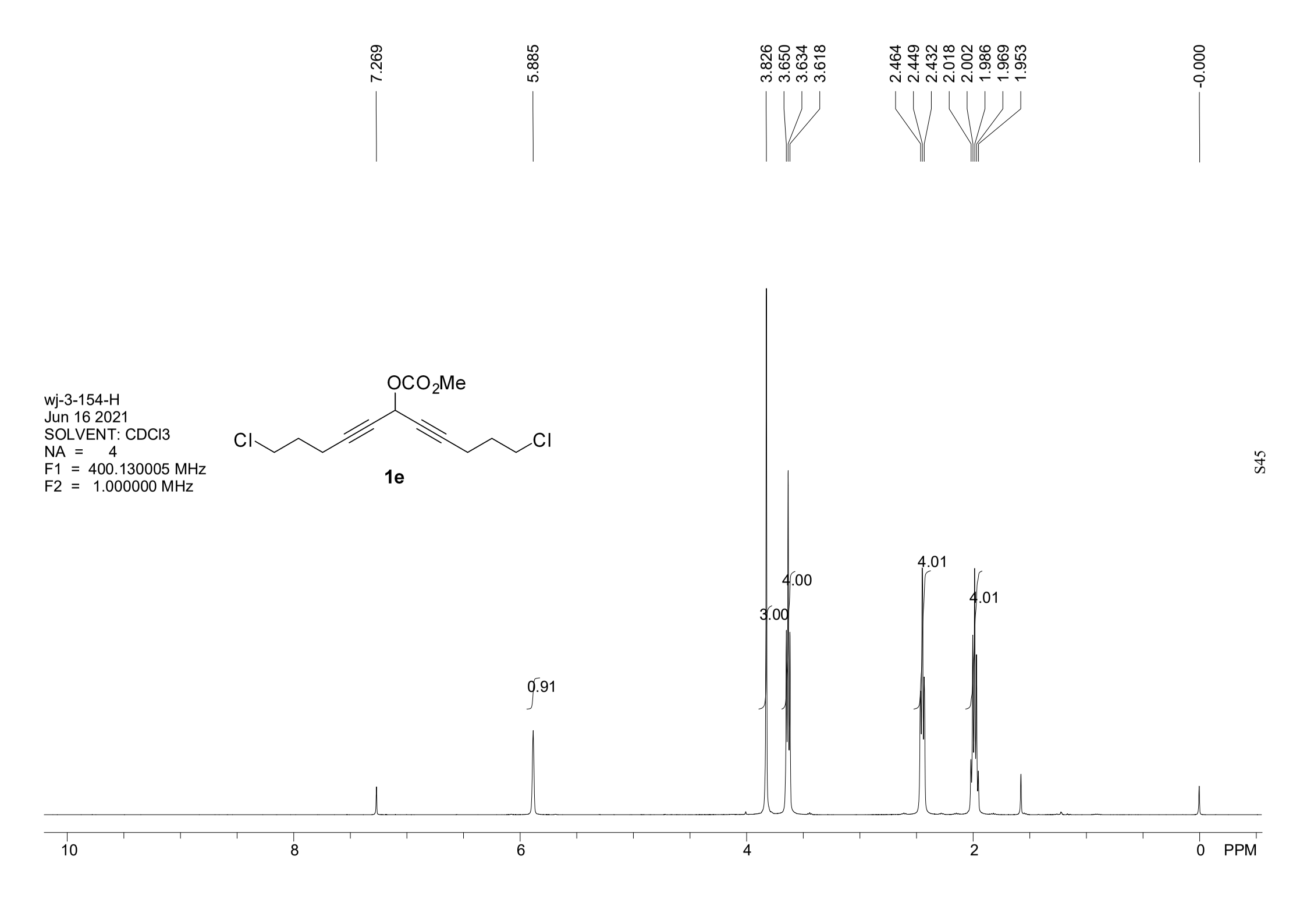
100
50
0
PPM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3.634
3.618
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2.449
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2.018
2.002
1.986
1.969
1.953
-0.000
wj-3-154-H
Jun 16 2021
SOLVENT: CDCl3
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F1 = 400.130005 MHz
F2 = 1.000000 MHz
OCO2Me
Cl
Cl
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4.00
4.01
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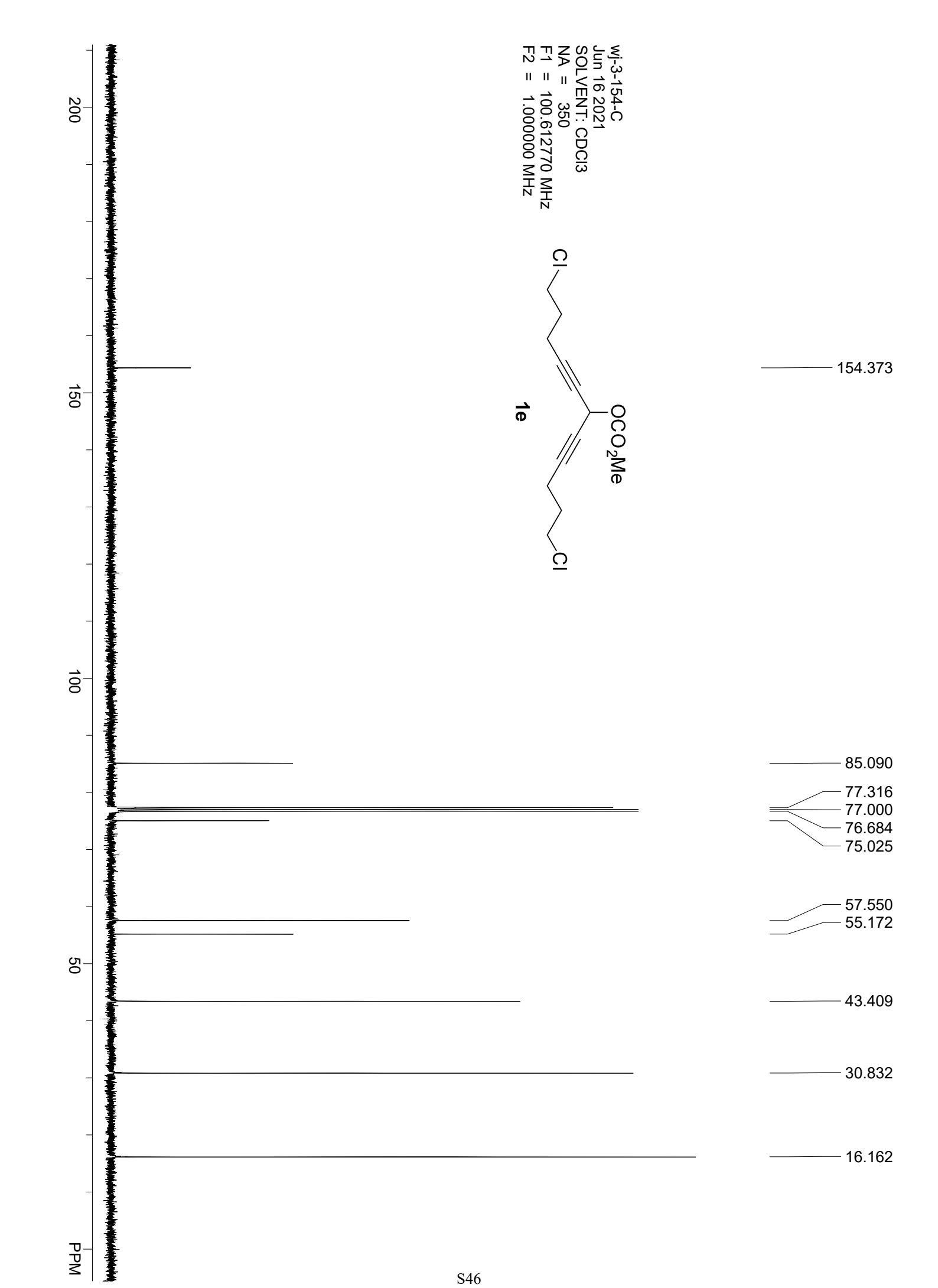
S45
10
8
6
4
2
0
PPM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Jun 16 2021
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Cl
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85.090
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75.025
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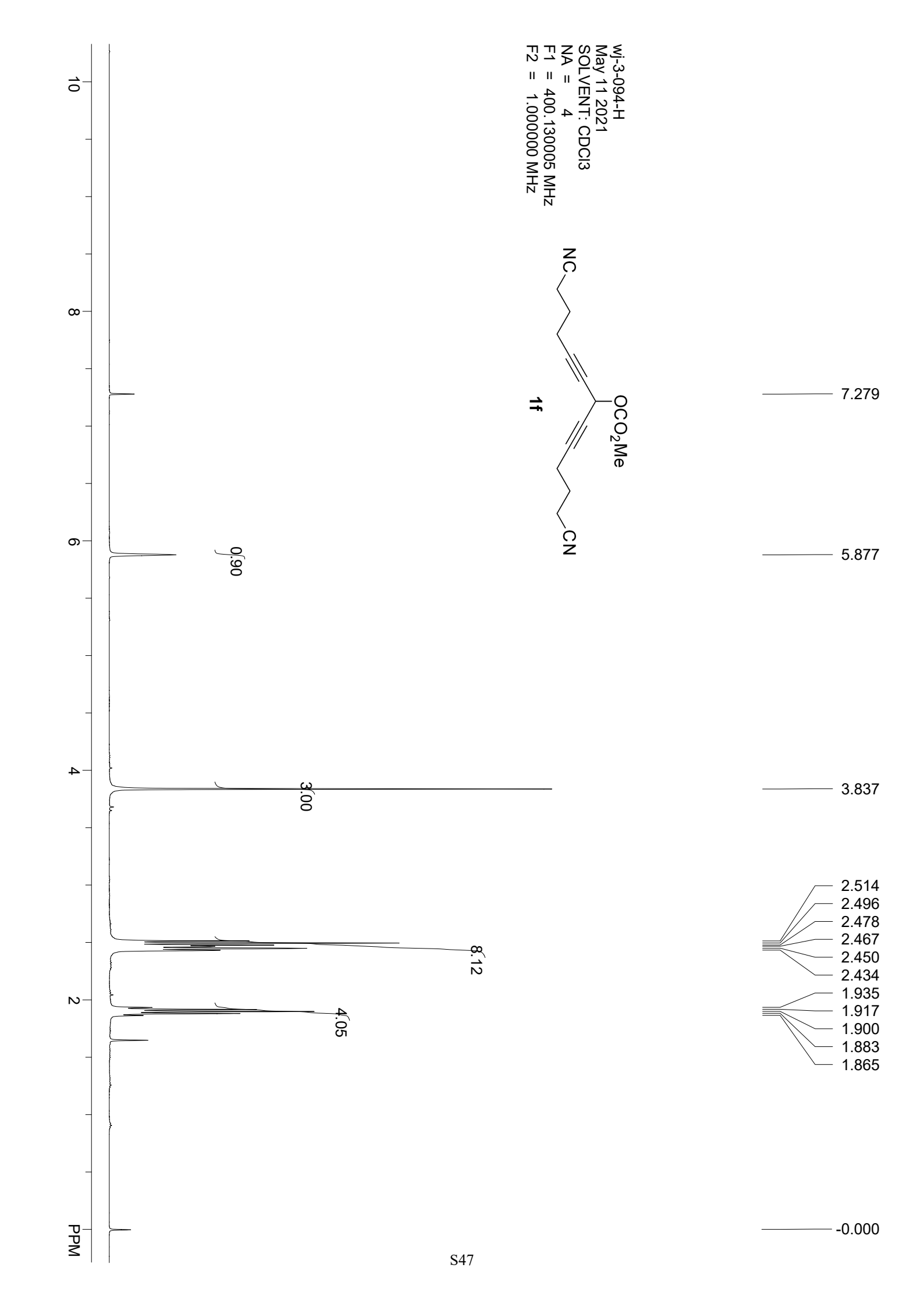
55.172
43.409
30.832
16.162
200
150
100
50
PPM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May 11 2021
SOLVENT: CDCl3
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2.496
2.478
2.467
2.450
2.434
1.935
1.917
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0.90
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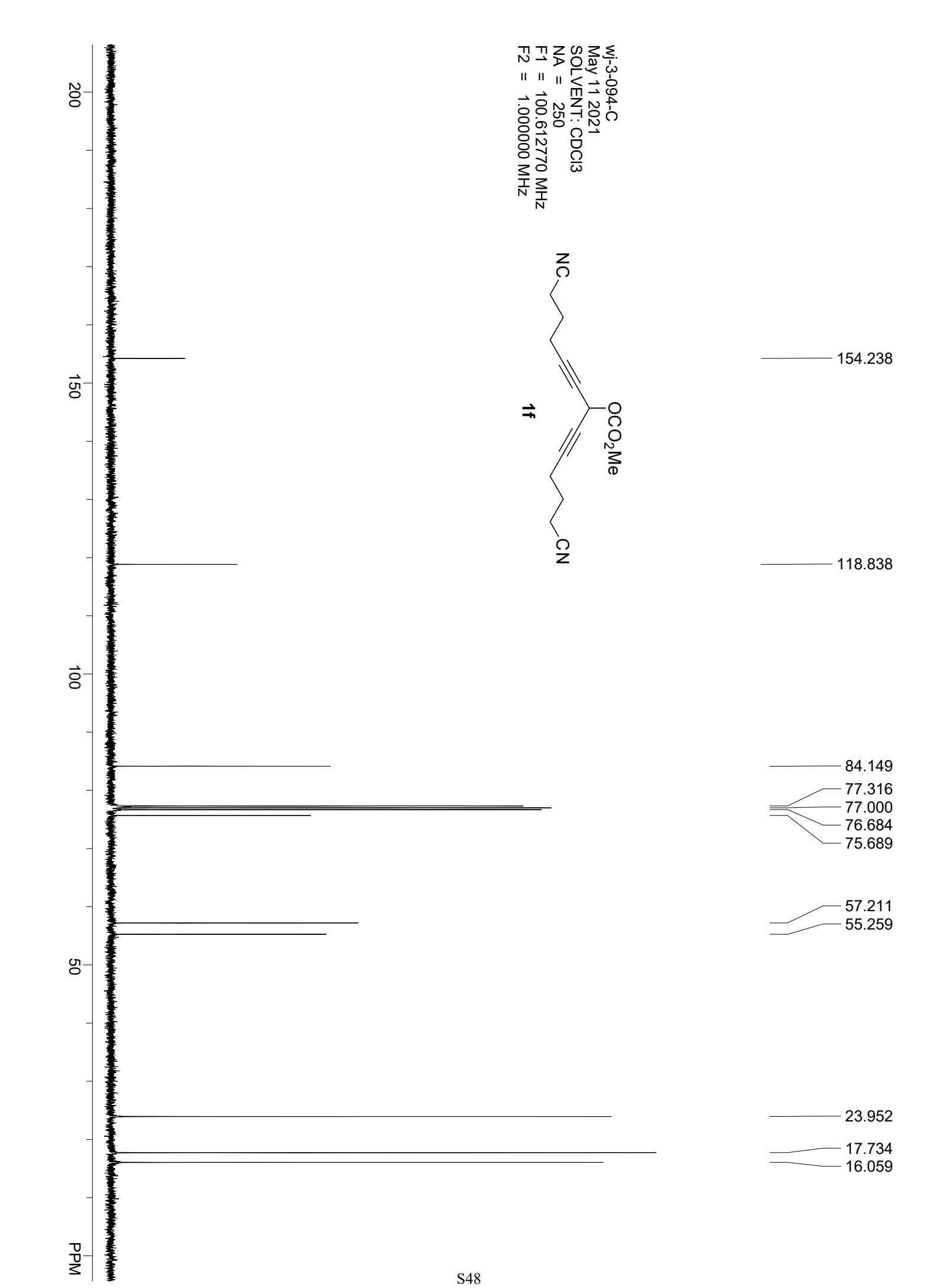
8.12
4.05
10
8
6
4
2
PPM


wj-3-094-C
May 11 2021
SOLVENT: CDCl3
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OCO2Me
CN
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77.000
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150
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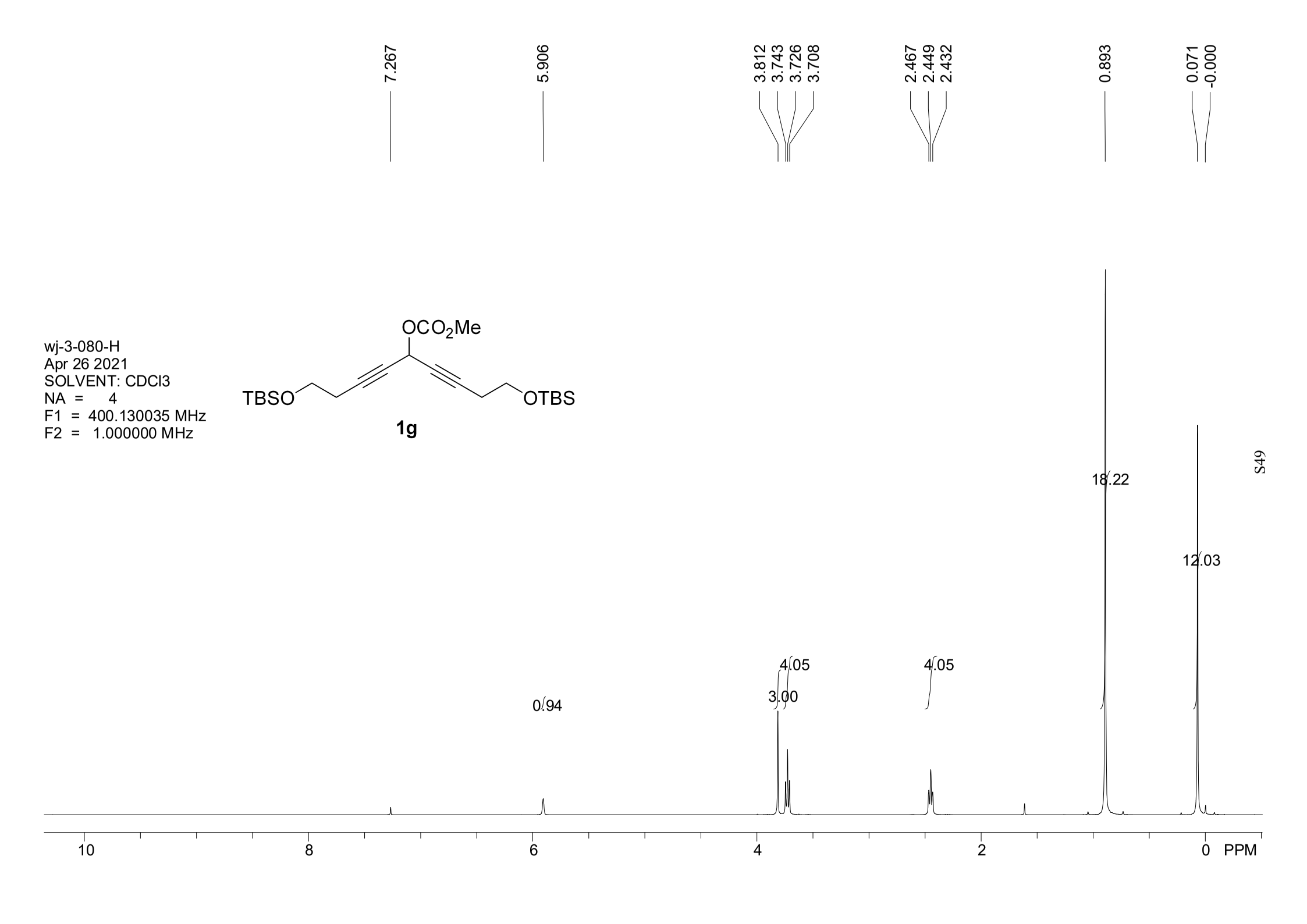
50
PPM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wj-3-080-H
Apr 26 2021
SOLVENT: CDCl3
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F2 = 1.000000 MHz
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TBSO
OTBS
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S49
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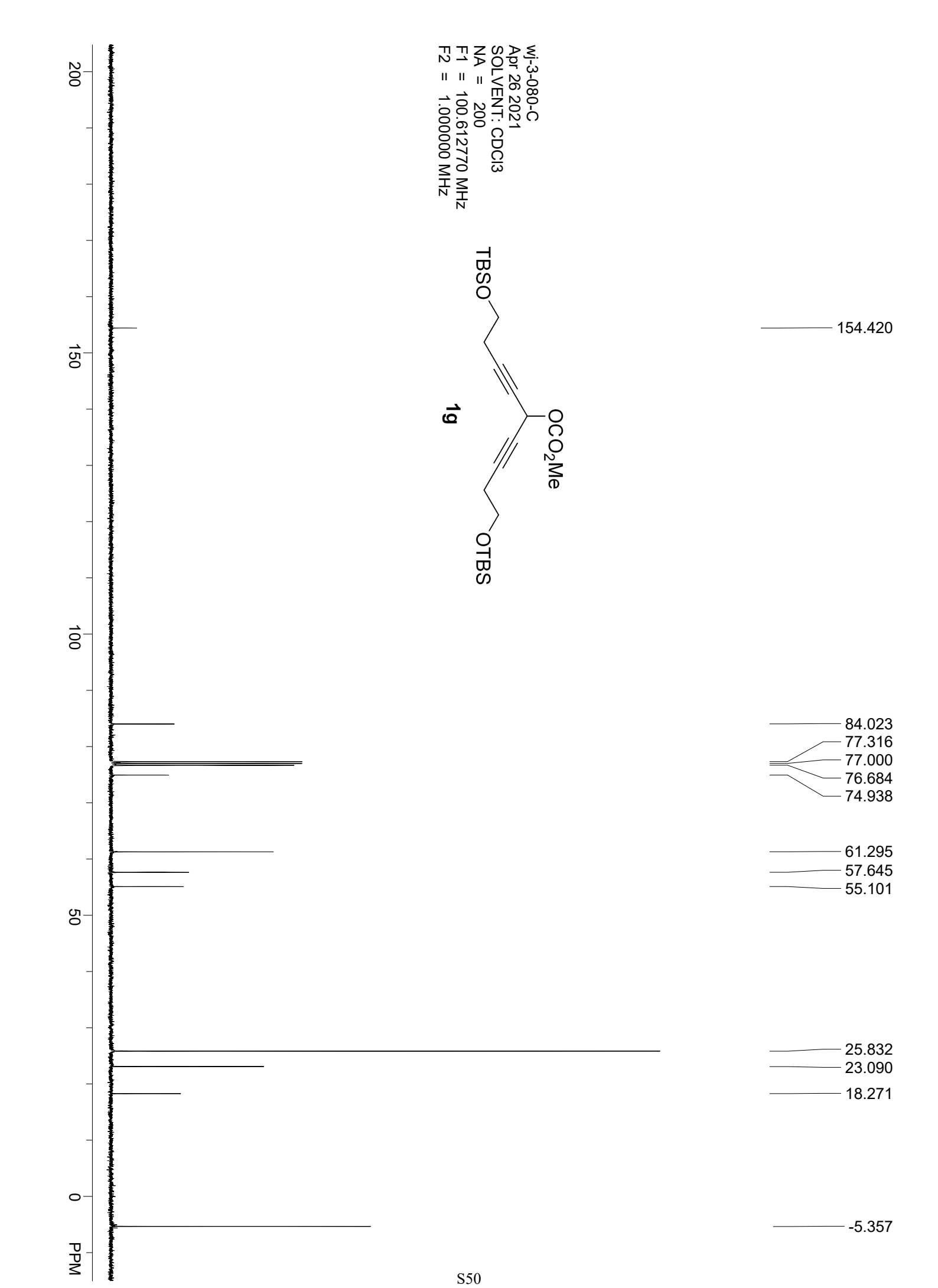
4
2
0
PPM


wj-3-080-C
Apr 26 2021
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OTBS
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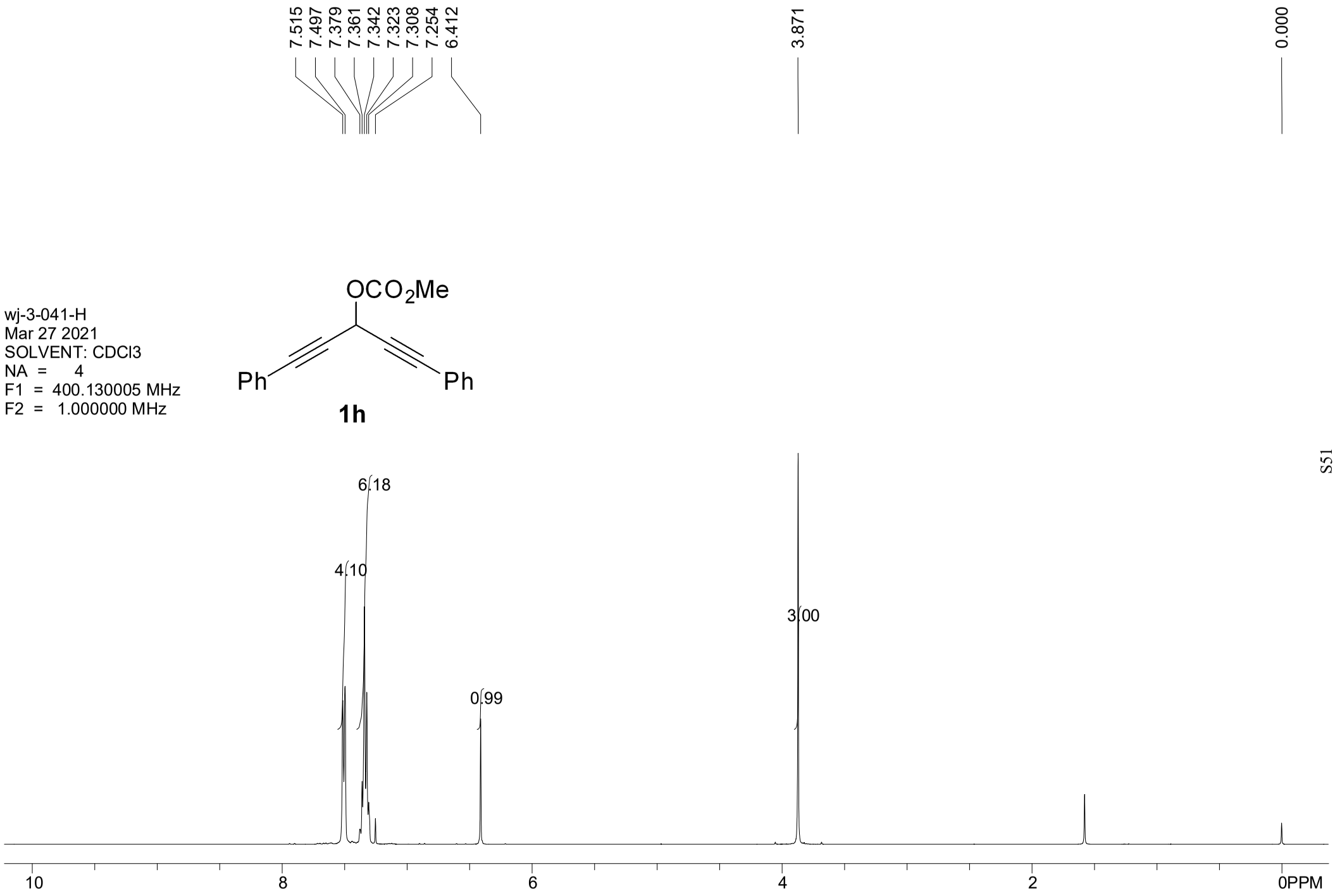
PPM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wj-3-041-H
Mar 27 2021
SOLVENT: CDCl3
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4
2
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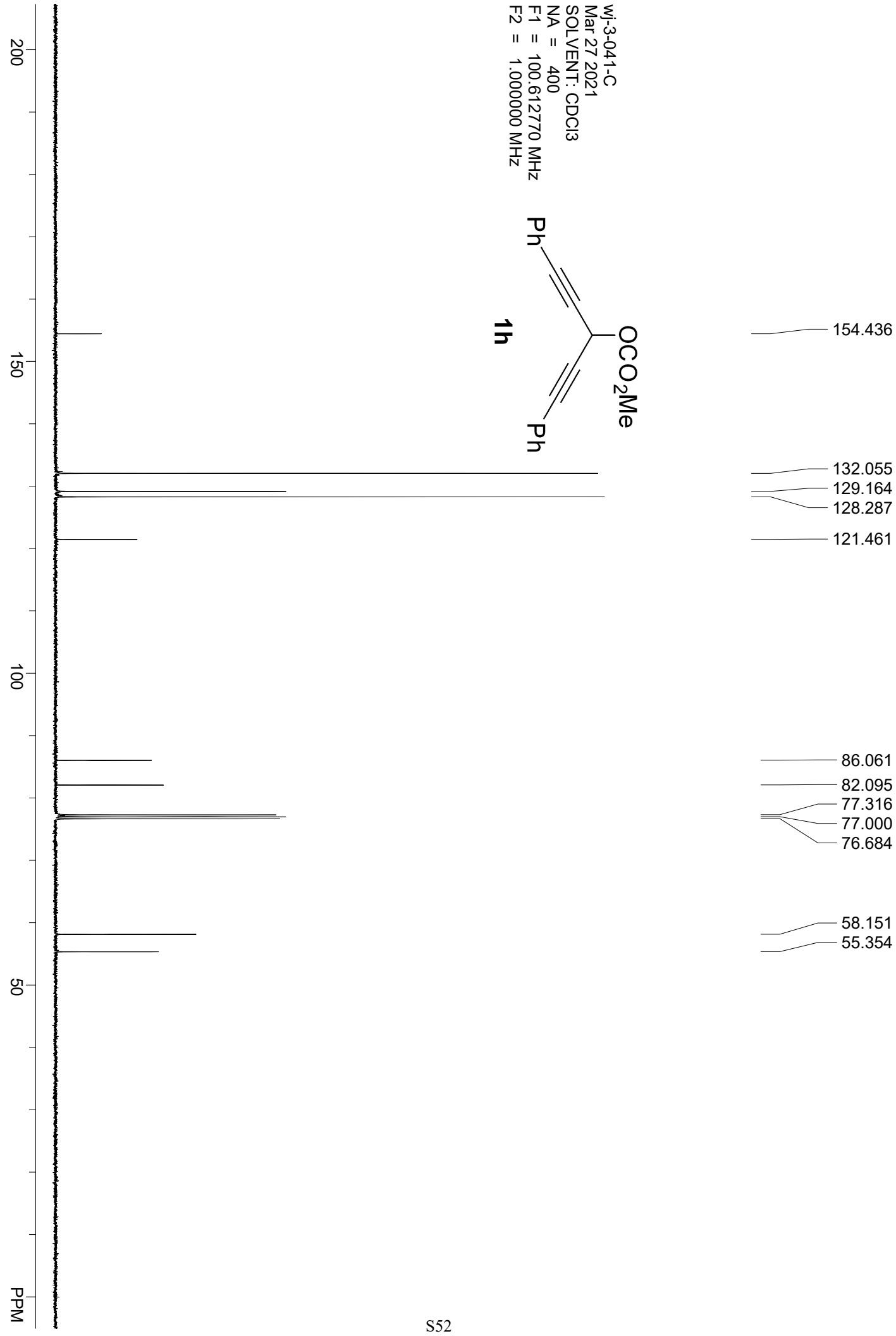
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Mar 27 2021
SOLVENT: CDCl3
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Ph
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77.000
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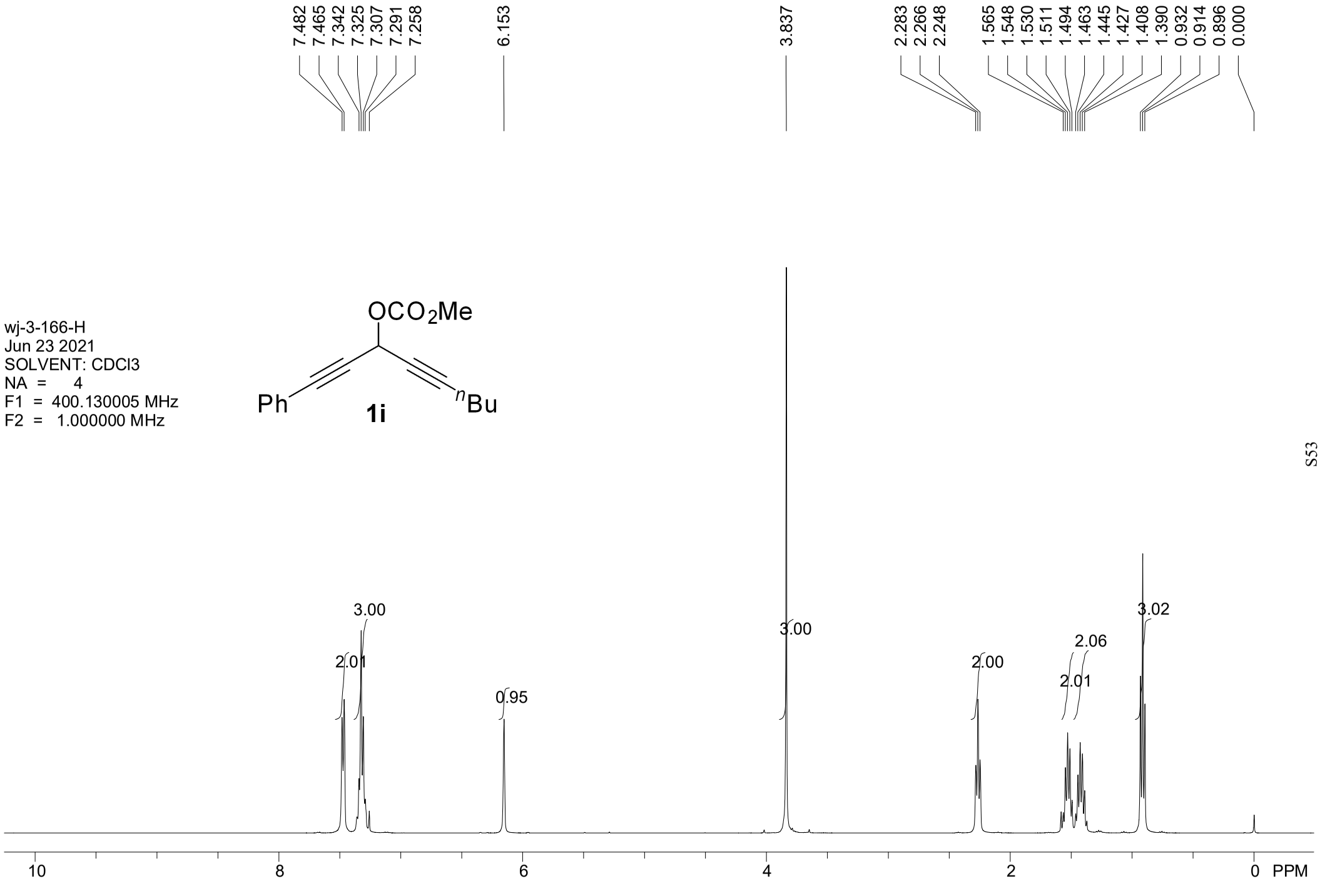
150
100
50
PPM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1.548
1.530
1.511
1.494
1.463
1.445
1.427
1.408
1.390
0.932
0.914
0.896
0.000
wj-3-166-H
Jun 23 2021
SOLVENT: CDCl3
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F2 = 1.000000 MHz
OCO2Me
Ph
nBu
1i
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2.01
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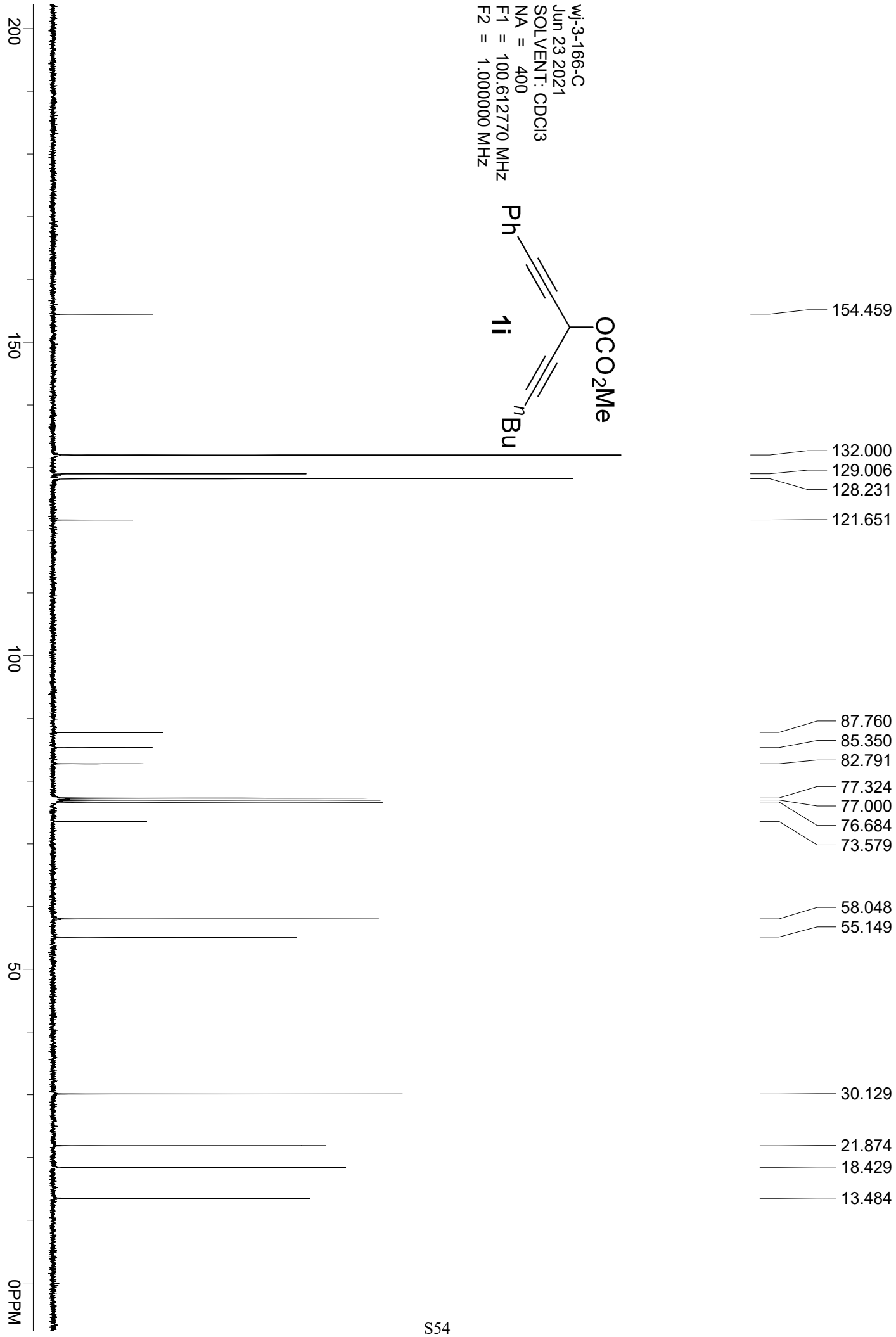
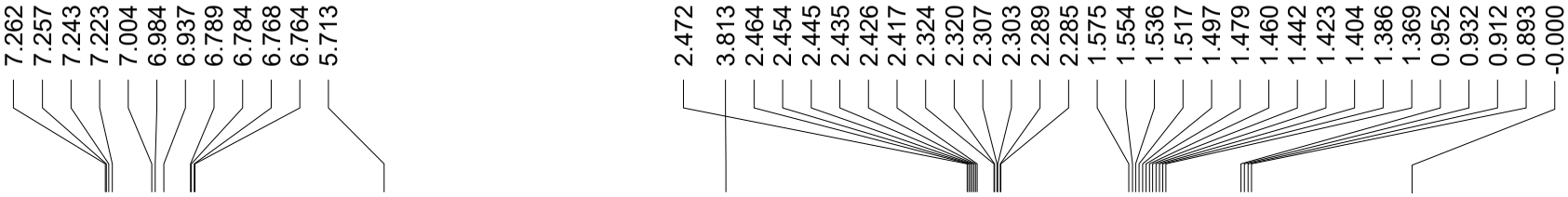
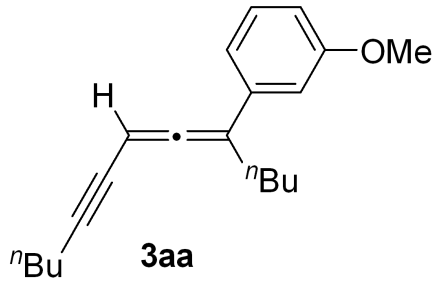
3.00
2.00
2.06
2.01
3.02
10
8
6
4
2
0
PPM


wj-3-166-C
Jun 23 2021
SOLVENT: CDCl3
NA = 400
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
OCO2Me
nBu
1i
154.459
132.000
129.006
128.231
121.651
87.760
85.350
82.791
77.324
77.000
76.684
73.579
58.048
55.149
30.129
21.874
18.429
13.484
200
150
100
50
0PPM

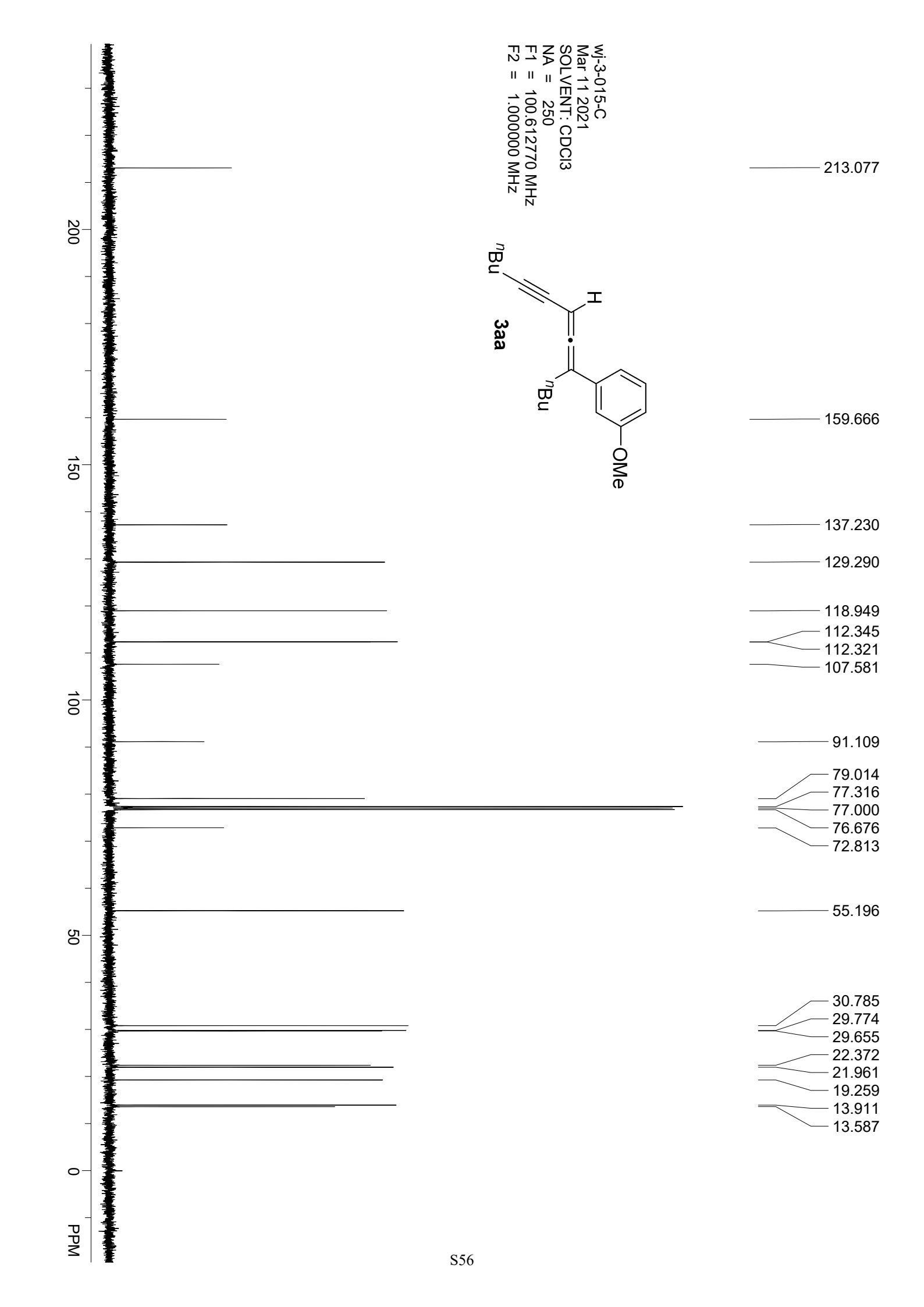


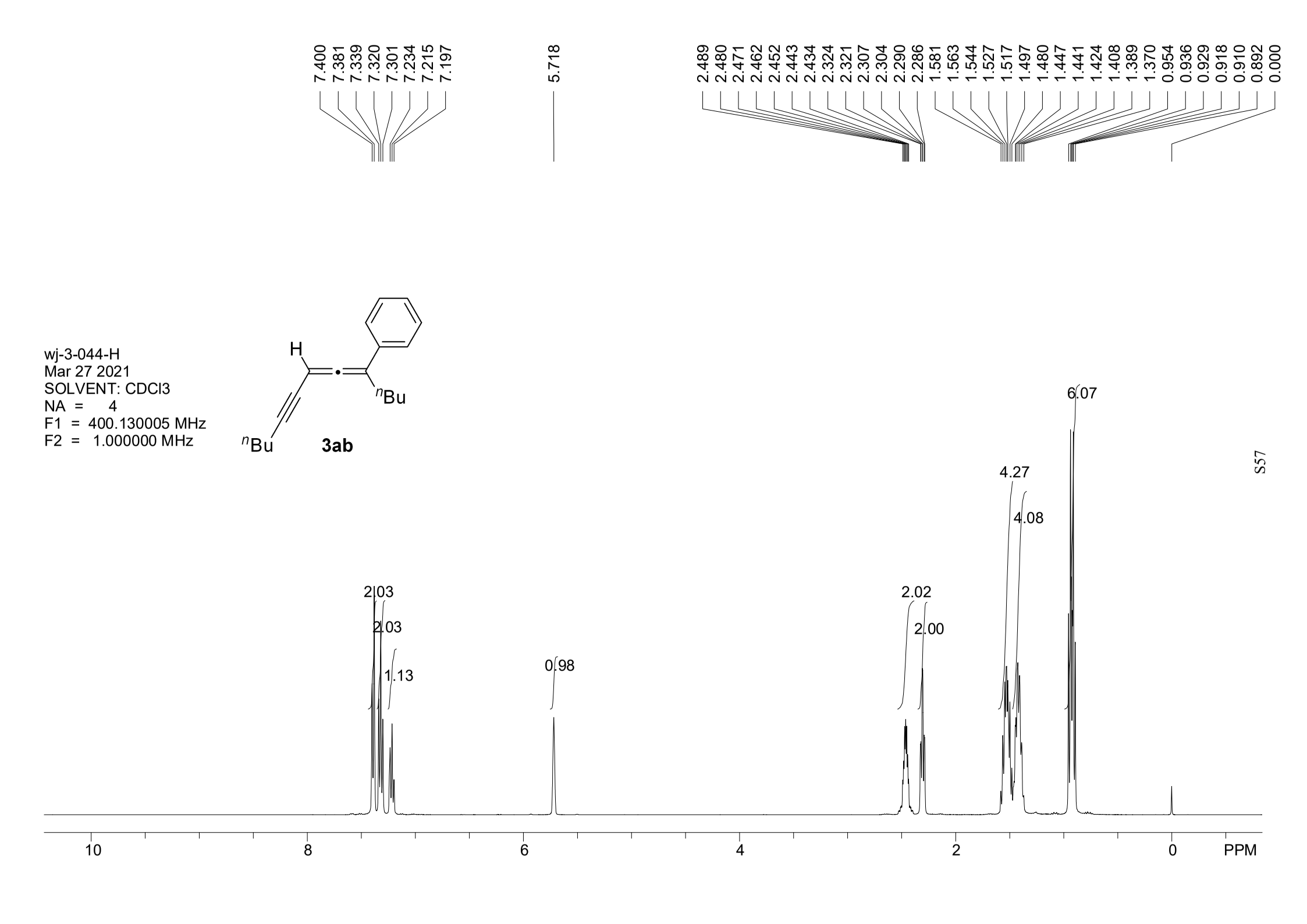
wj-3-015-H
Mar 11 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz

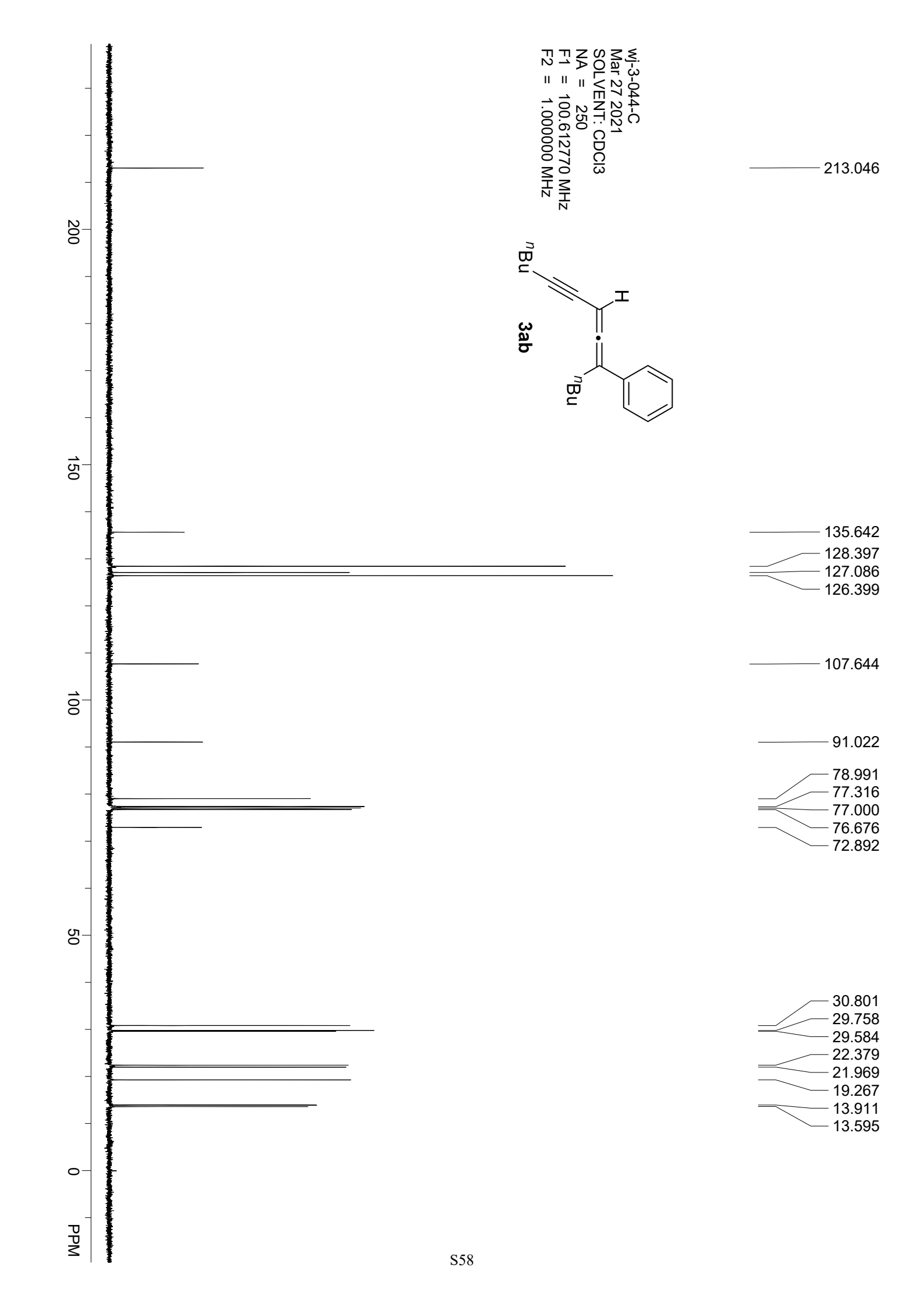


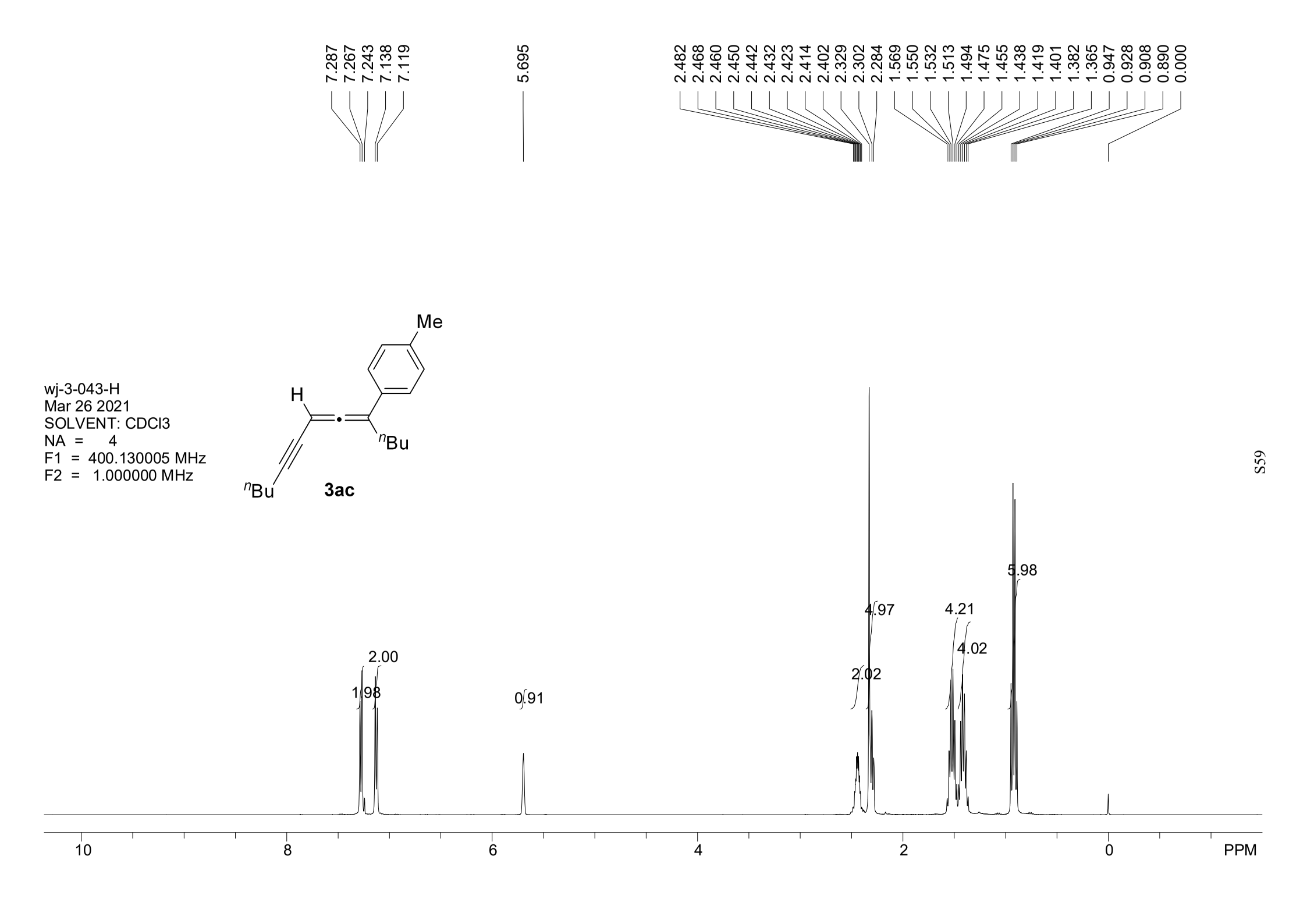
3aa

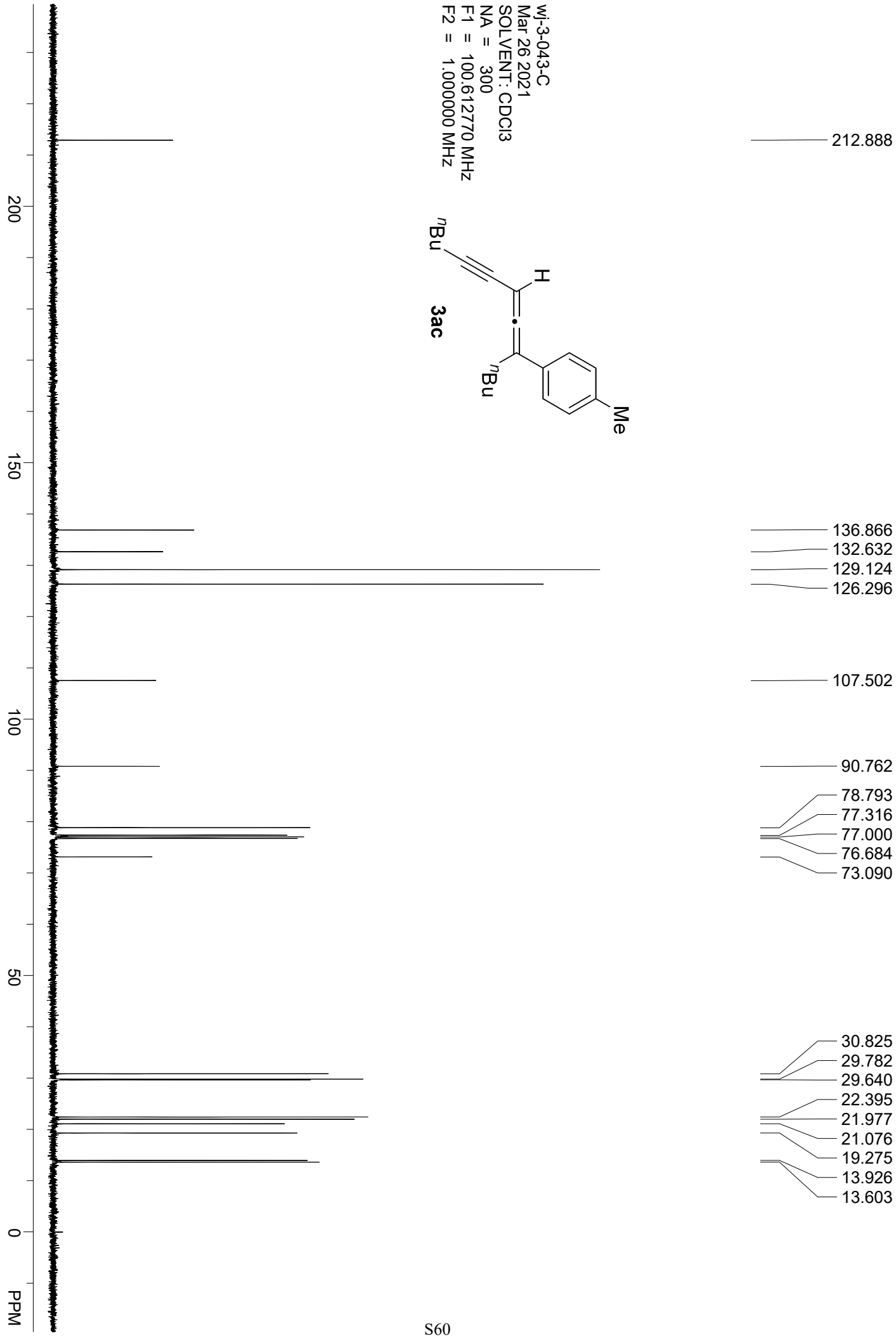
S55

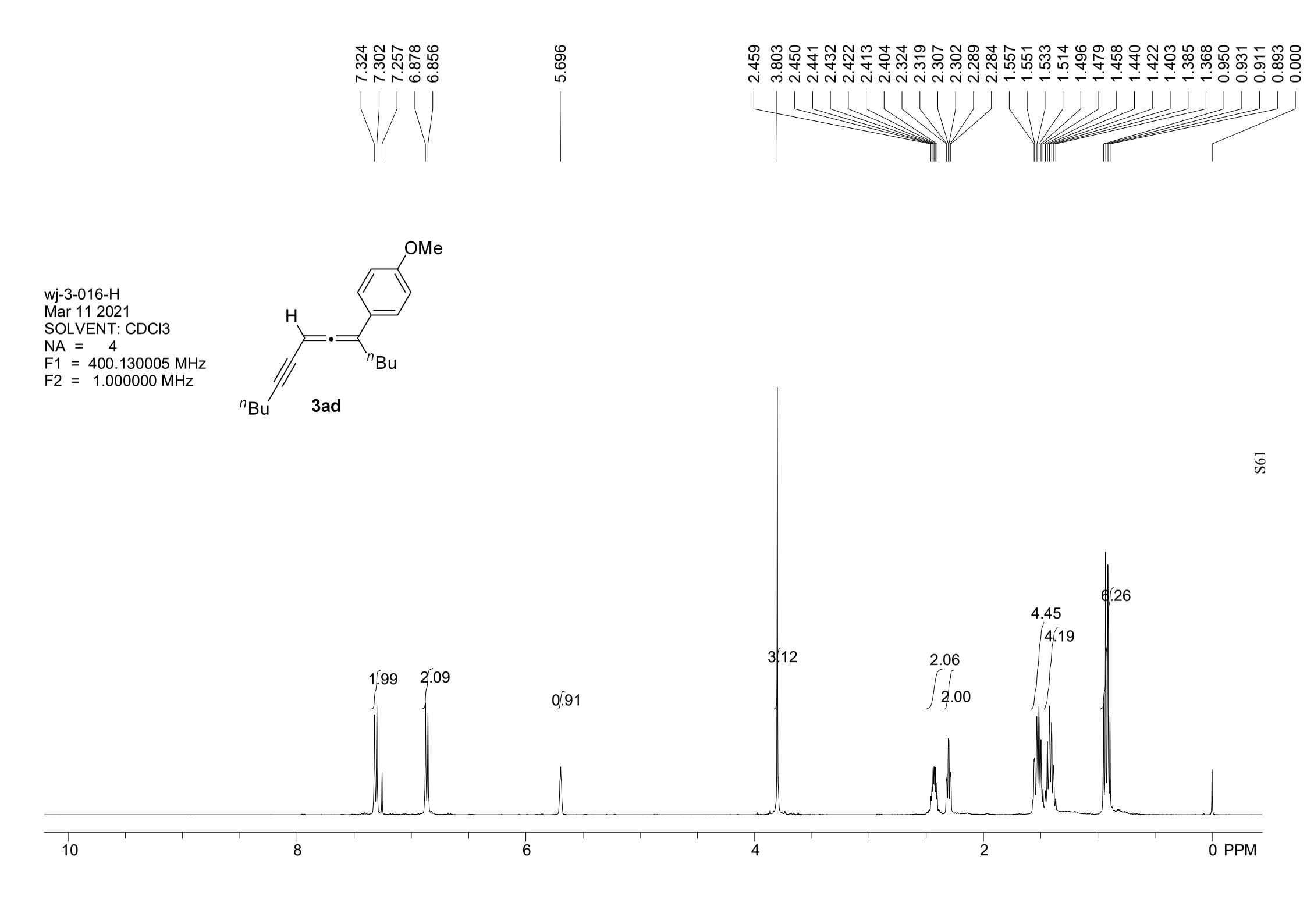

wj-3-015-C
Mar 11 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
OMe
3aa
213.077
159.666
137.230
129.290
118.949
112.345
112.321
107.581
91.109
79.014
77.316
77.000
76.676
72.813
55.196
30.785
29.774
29.655
22.372
21.961
19.259
13.911
13.587
200
150
100
50
0
PPM

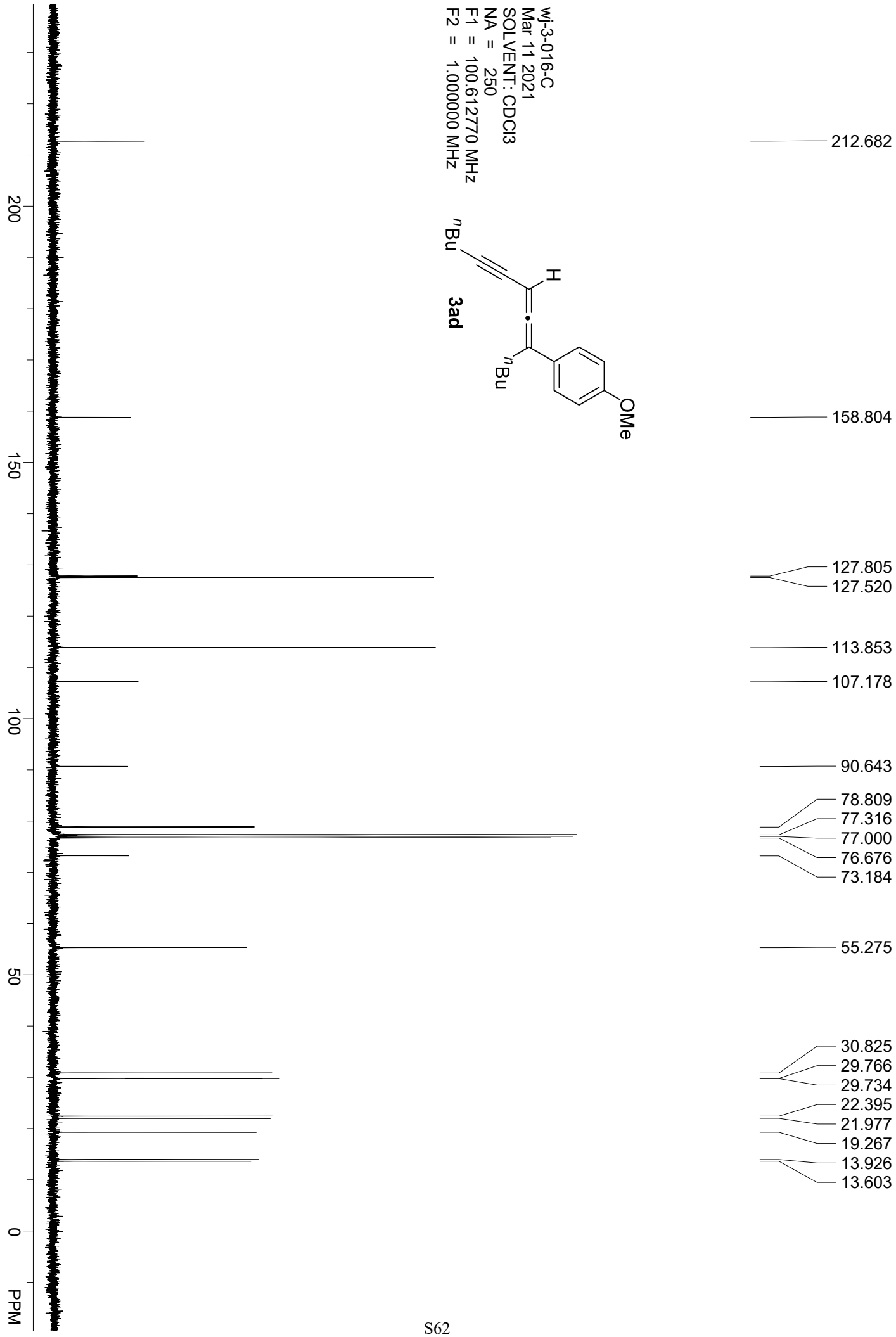

7.400
7.381
7.339
7.320
7.301
7.234
7.215
7.197
5.718
2.489
2.480
2.471
2.462
2.452
2.443
2.434
2.324
2.321
2.307
2.304
2.290
2.286
1.581
1.563
1.544
1.527
1.517
1.497
1.480
1.447
1.441
1.424
1.408
1.389
1.370
0.954
0.936
0.929
0.918
0.910
0.892
0.000
wj-3-044-H
Mar 27 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
nBu
nBu
3ab
2.03
2.03
1.13
0.98
2.02
2.00
4.27
4.08
6.07
S57
10
8
6
4
2
0
PPM

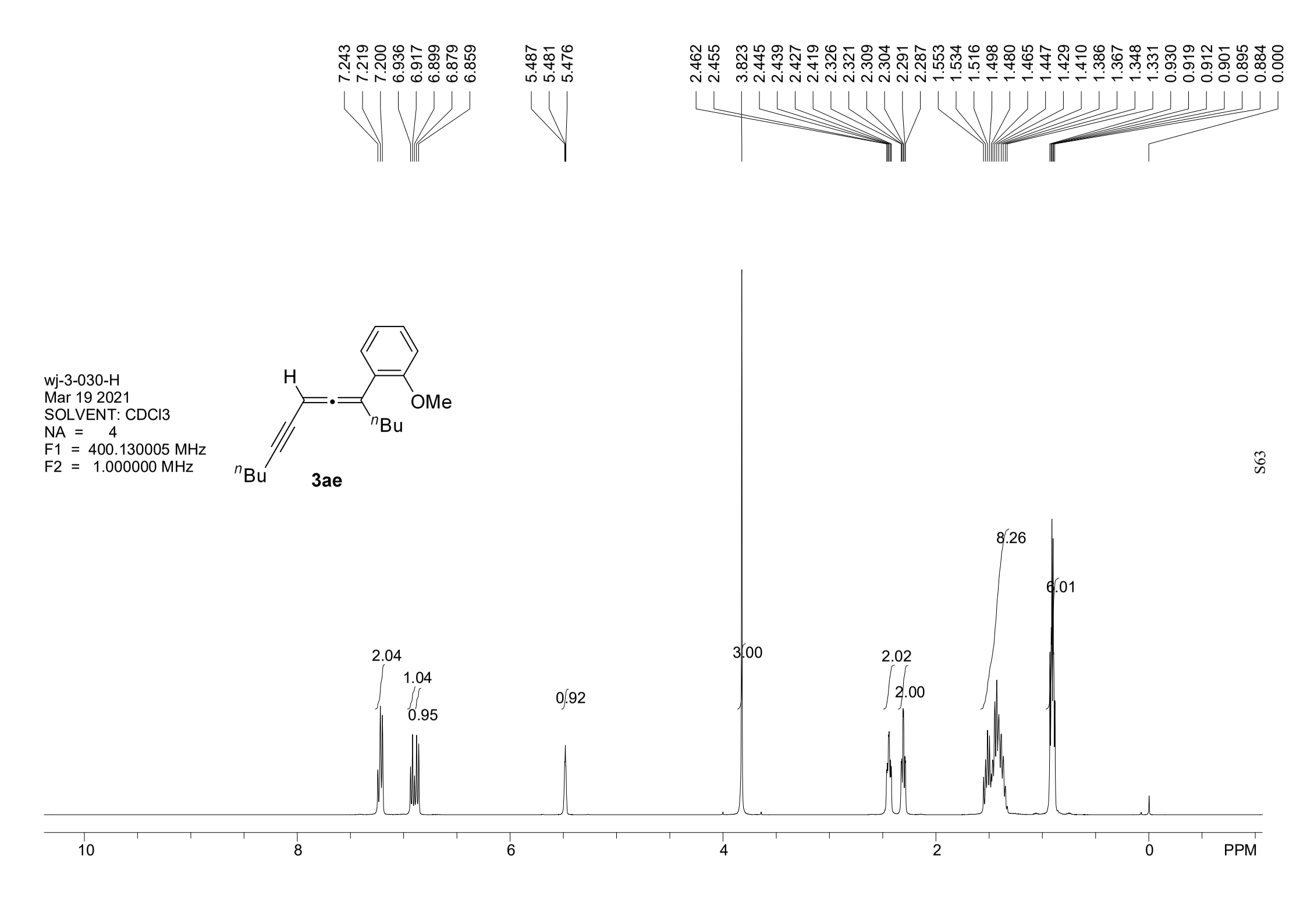

wj-3-044-C
Mar 27 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
3ab
213.046
135.642
128.397
127.086
126.399
107.644
91.022
78.991
77.316
77.000
76.676
72.892
30.801
29.758
29.584
22.379
21.969
19.267
13.911
13.595
200
150
100
50
0
PPM

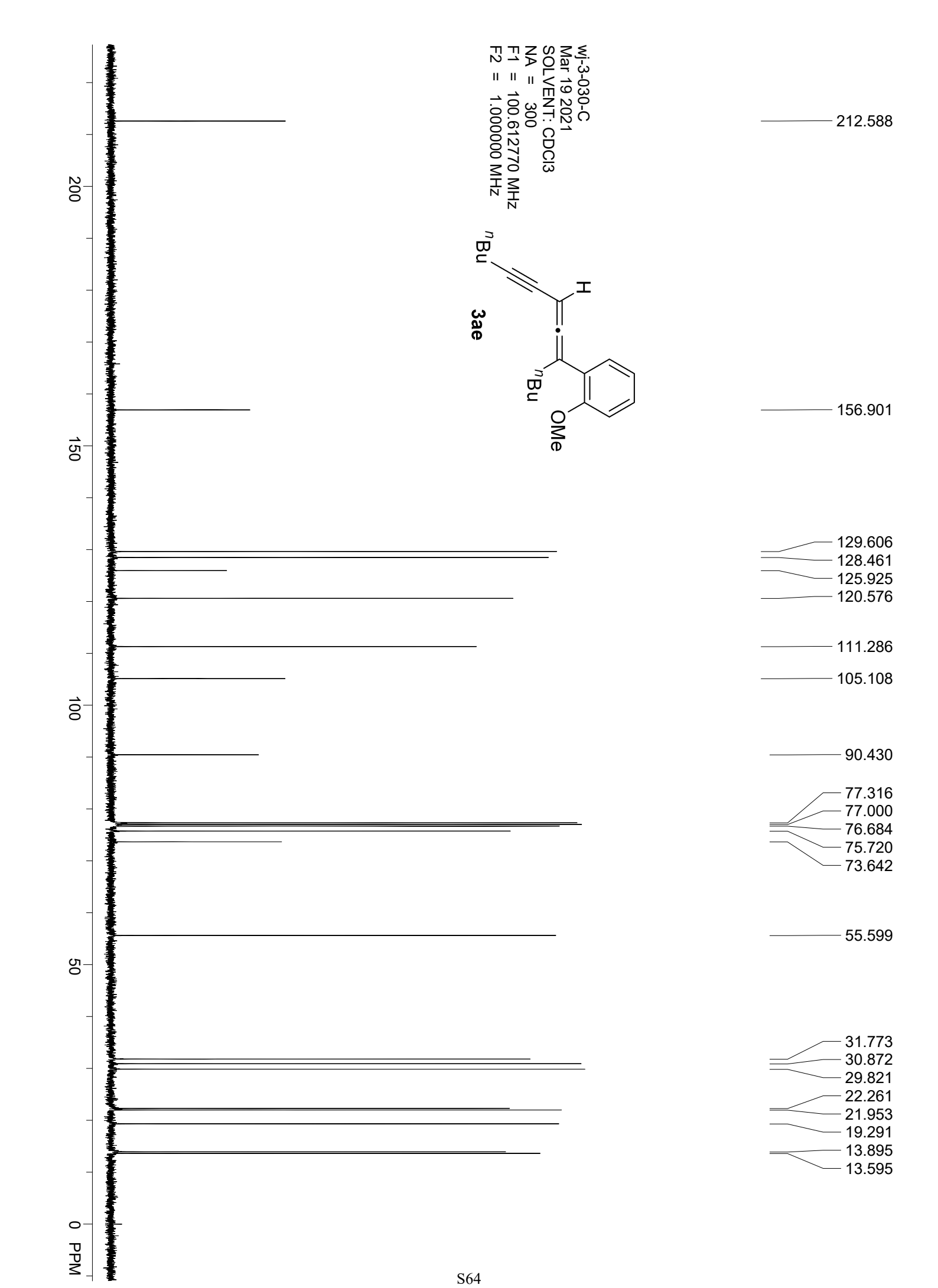

7.287
7.267
7.243
7.138
7.119
5.695
2.482
2.468
2.460
2.450
2.442
2.432
2.423
2.414
2.402
2.329
2.302
2.284
1.569
1.550
1.532
1.513
1.494
1.475
1.455
1.438
1.419
1.401
1.382
1.365
0.947
0.928
0.908
0.890
0.000
wj-3-043-H
Mar 26 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
Me
H
nBu
nBu
3ac
2.00
1.98
0.91
2.02
4.97
4.21
4.02
5.98
10
8
6
4
2
0
PPM

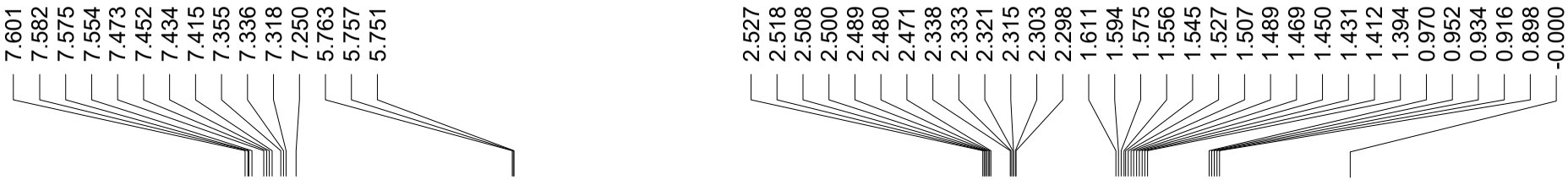

wj-3-043-C
Mar 26 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
Me
3ac
212.888
136.866
132.632
129.124
126.296
107.502
90.762
78.793
77.316
77.000
76.684
73.090
30.825
29.782
29.640
22.395
21.977
21.076
19.275
13.926
13.603
200
150
100
50
0
PPM

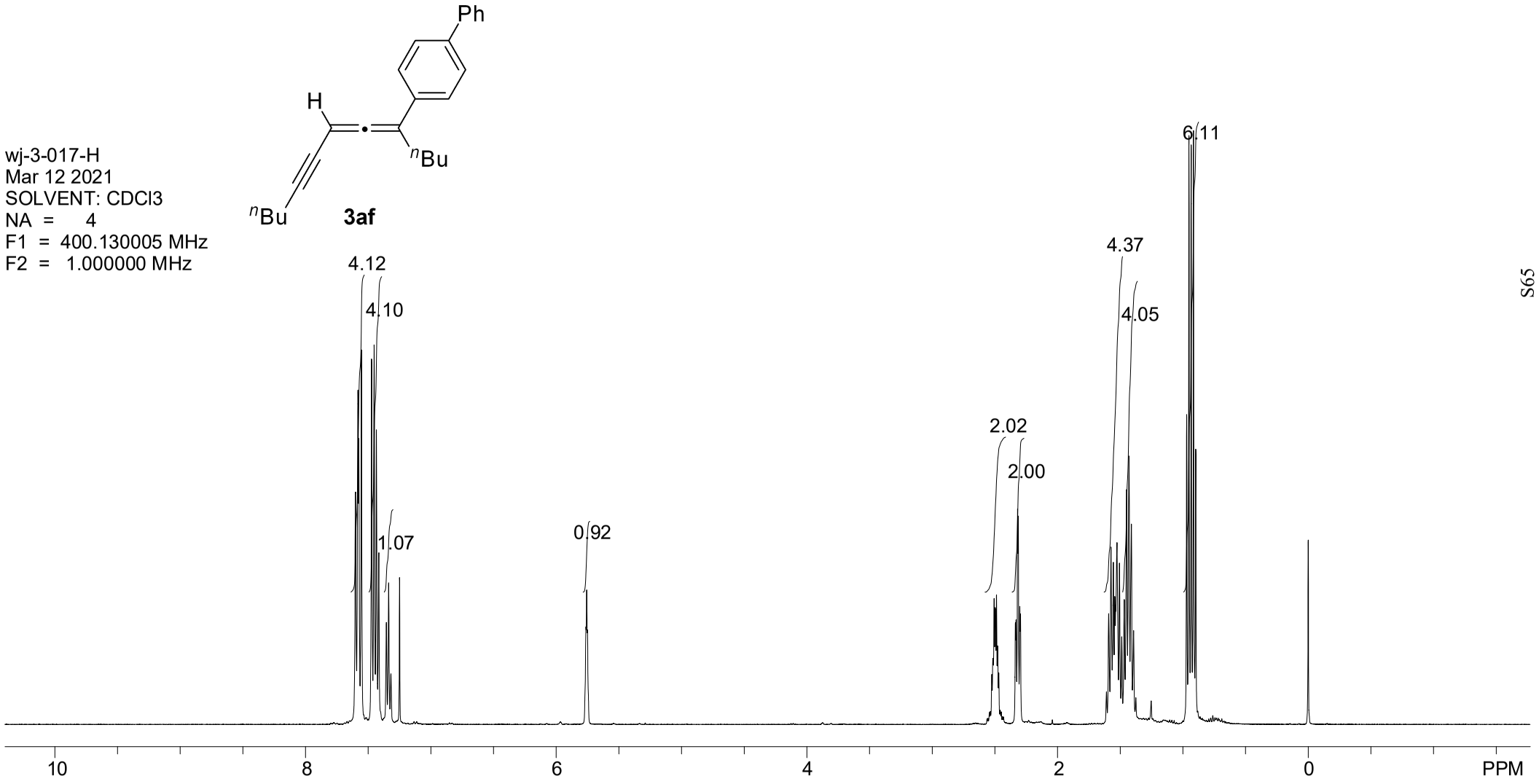

7.324
7.302
7.257
6.878
6.856
5.696
2.459
3.803
2.450
2.441
2.432
2.422
2.413
2.404
2.324
2.319
2.307
2.302
2.289
2.284
1.557
1.551
1.533
1.514
1.496
1.479
1.458
1.440
1.422
1.403
1.385
1.368
0.950
0.931
0.911
0.893
0.000
wj-3-016-H
Mar 11 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
OMe
H
nBu
nBu
3ad
S61
1.99
2.09
0.91
3.12
2.06
2.00
4.45
4.19
6.26
10
8
6
4
2
0 PPM

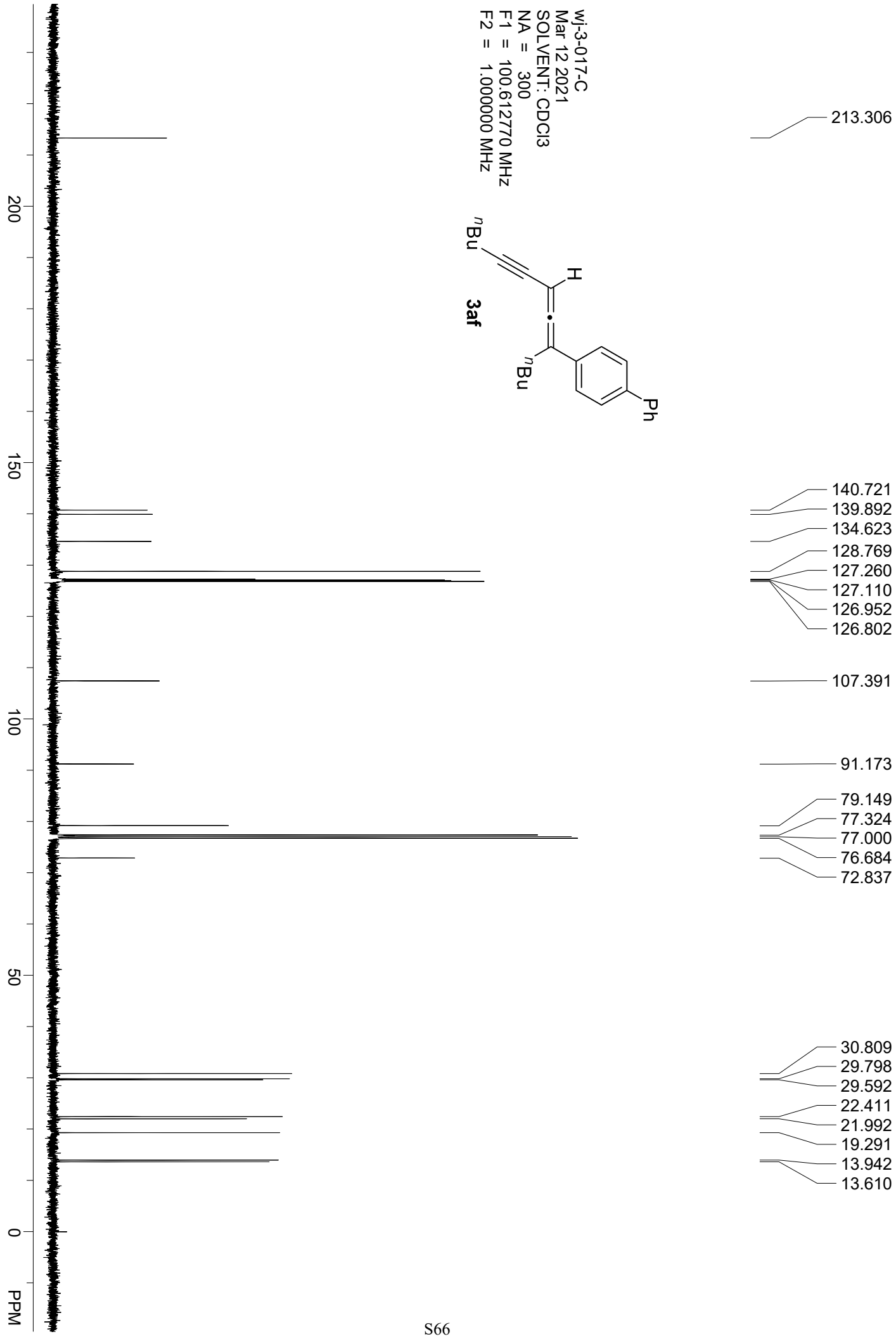

wj-3-016-C
Mar 11 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3ad
nBu
OMe
212.682
158.804
127.805
127.520
113.853
107.178
90.643
78.809
77.316
77.000
76.676
73.184
55.275
30.825
29.766
29.734
22.395
21.977
19.267
13.926
13.603
200
150
100
50
0
PPM

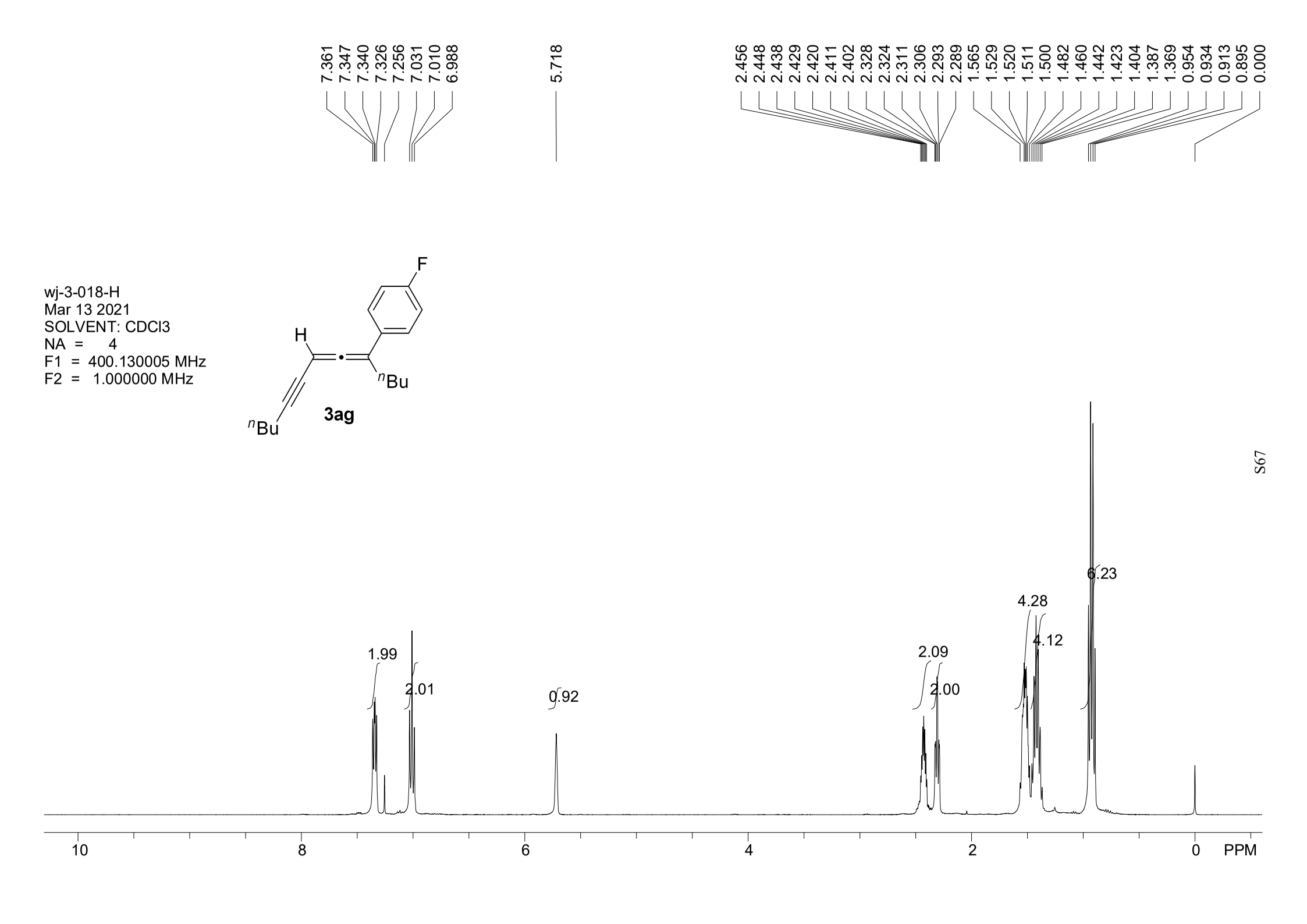

7.243
7.219
7.200
6.936
6.917
6.899
6.879
6.859
5.487
5.481
5.476
2.462
2.455
3.823
2.445
2.439
2.427
2.419
2.326
2.321
2.309
2.304
2.291
2.287
1.553
1.534
1.516
1.498
1.480
1.465
1.447
1.429
1.410
1.386
1.367
1.348
1.331
0.930
0.919
0.912
0.901
0.895
0.884
0.000
wj-3-030-H
Mar 19 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
OMe
nBu
nBu
3ae
S63
2.04
1.04
0.95
0.92
3.00
2.02
2.00
8.26
6.01
10
8
6
4
2
0
PPM

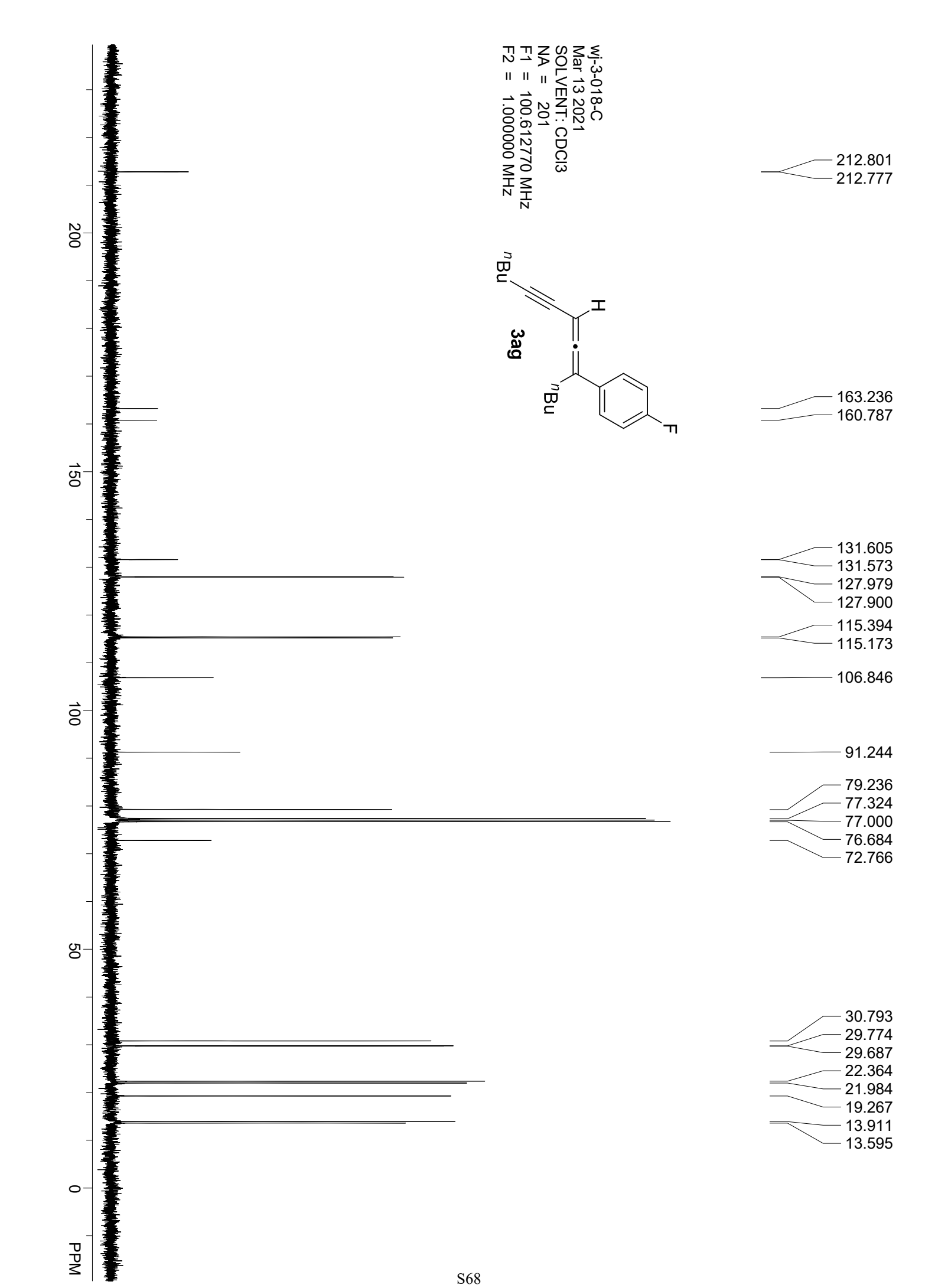

wj-3-030-C
Mar 19 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3ae
nBu
OMe
212.588
156.901
129.606
128.461
125.925
120.576
111.286
105.108
90.430
77.316
77.000
76.684
75.720
73.642
55.599
31.773
30.872
29.821
22.261
21.953
19.291
13.895
13.595
200
150
100
50
0 PPM


7.601
7.582
7.575
7.554
7.473
7.452
7.434
7.415
7.355
7.336
7.318
7.250
5.763
5.757
5.751
2.527
2.518
2.508
2.500
2.489
2.480
2.471
2.338
2.333
2.321
2.315
2.303
2.298
1.611
1.594
1.575
1.556
1.545
1.527
1.507
1.489
1.469
1.450
1.431
1.412
1.394
0.970
0.952
0.934
0.916
0.898
-0.000


wj-3-017-H
Mar 12 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
Ph
H
nBu
nBu
3af
4.12
4.10
1.07
0.92
2.02
2.00
4.37
4.05
6.11
10
8
6
4
2
0
PPM


wj-3-017-C
Mar 12 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
Ph
3af
213.306
140.721
139.892
134.623
128.769
127.260
127.110
126.952
126.802
107.391
91.173
79.149
77.324
77.000
76.684
72.837
30.809
29.798
29.592
22.411
21.992
19.291
13.942
13.610
200
150
100
50
0
PPM

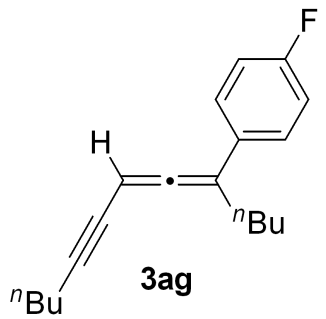

7.361
7.347
7.340
7.326
7.256
7.031
7.010
6.988
5.718
2.456
2.448
2.438
2.429
2.420
2.411
2.402
2.328
2.324
2.311
2.306
2.293
2.289
1.565
1.529
1.520
1.511
1.500
1.482
1.460
1.442
1.423
1.404
1.387
1.369
0.954
0.934
0.913
0.895
0.000
wj-3-018-H
Mar 13 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
F
H
nBu
nBu
3ag
1.99
2.01
0.92
2.09
2.00
4.28
4.12
6.23
10
8
6
4
2
0
PPM


wj-3-018-C
Mar 13 2021
SOLVENT: CDCl3
NA = 201
F1 = 100.612770 MHz
F2 = 1.000000 MHz
3ag
212.801
212.777
163.236
160.787
131.605
131.573
127.979
127.900
115.394
115.173
106.846
91.244
79.236
77.324
77.000
76.684
72.766
30.793
29.774
29.687
22.364
21.984
19.267
13.911
13.595
200
150
100
50
0
PPM

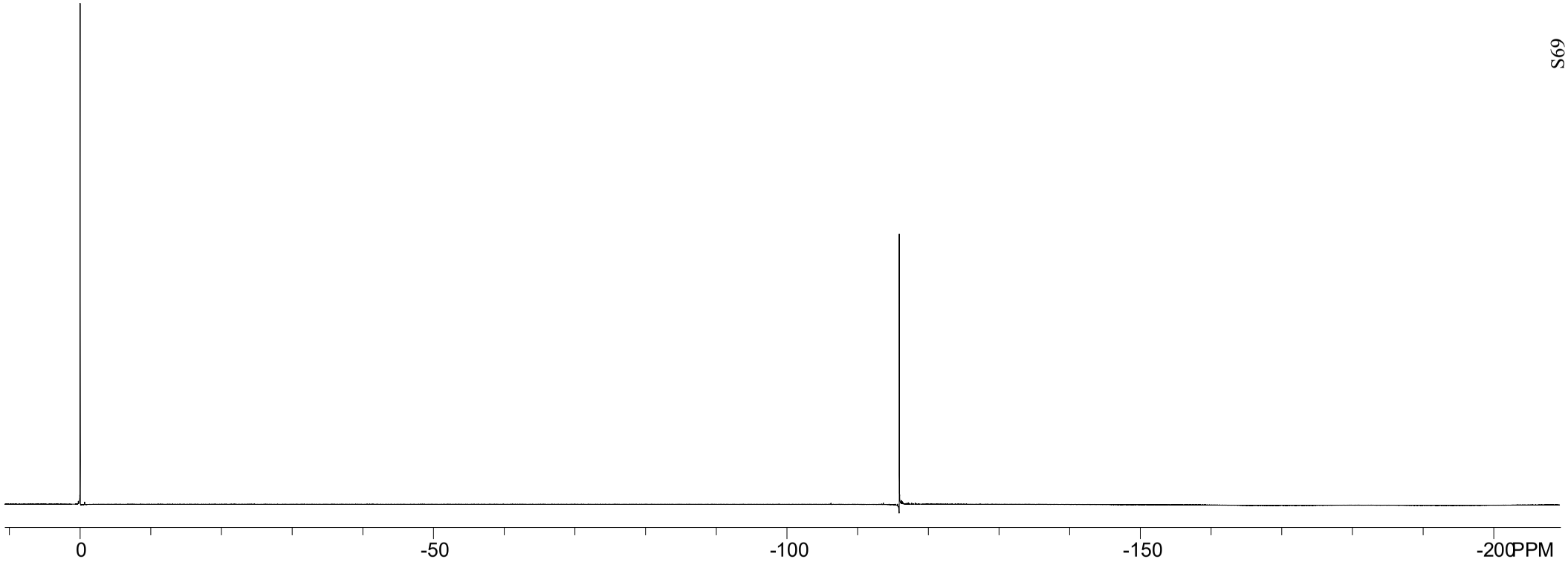
0.000

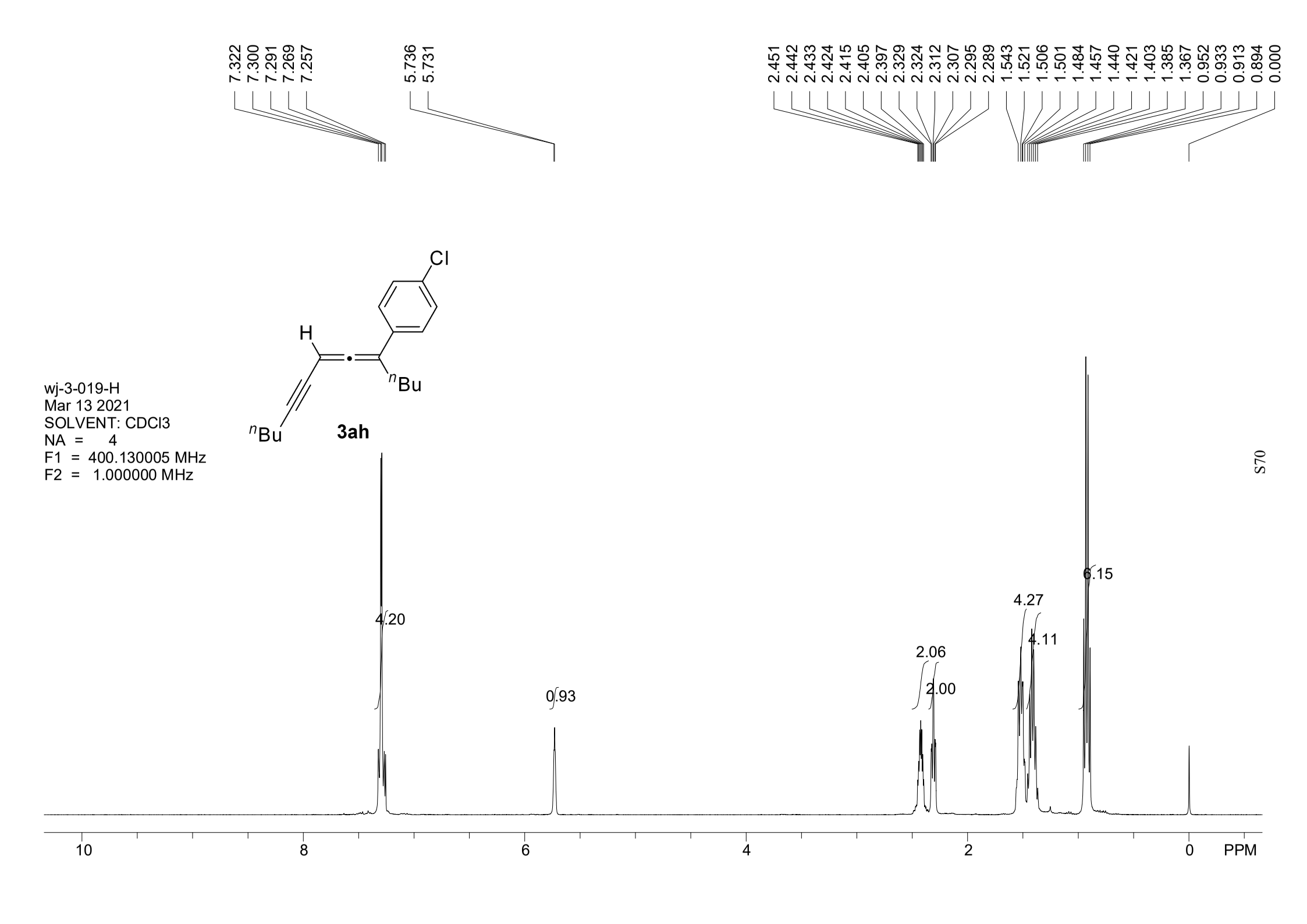
-115.941

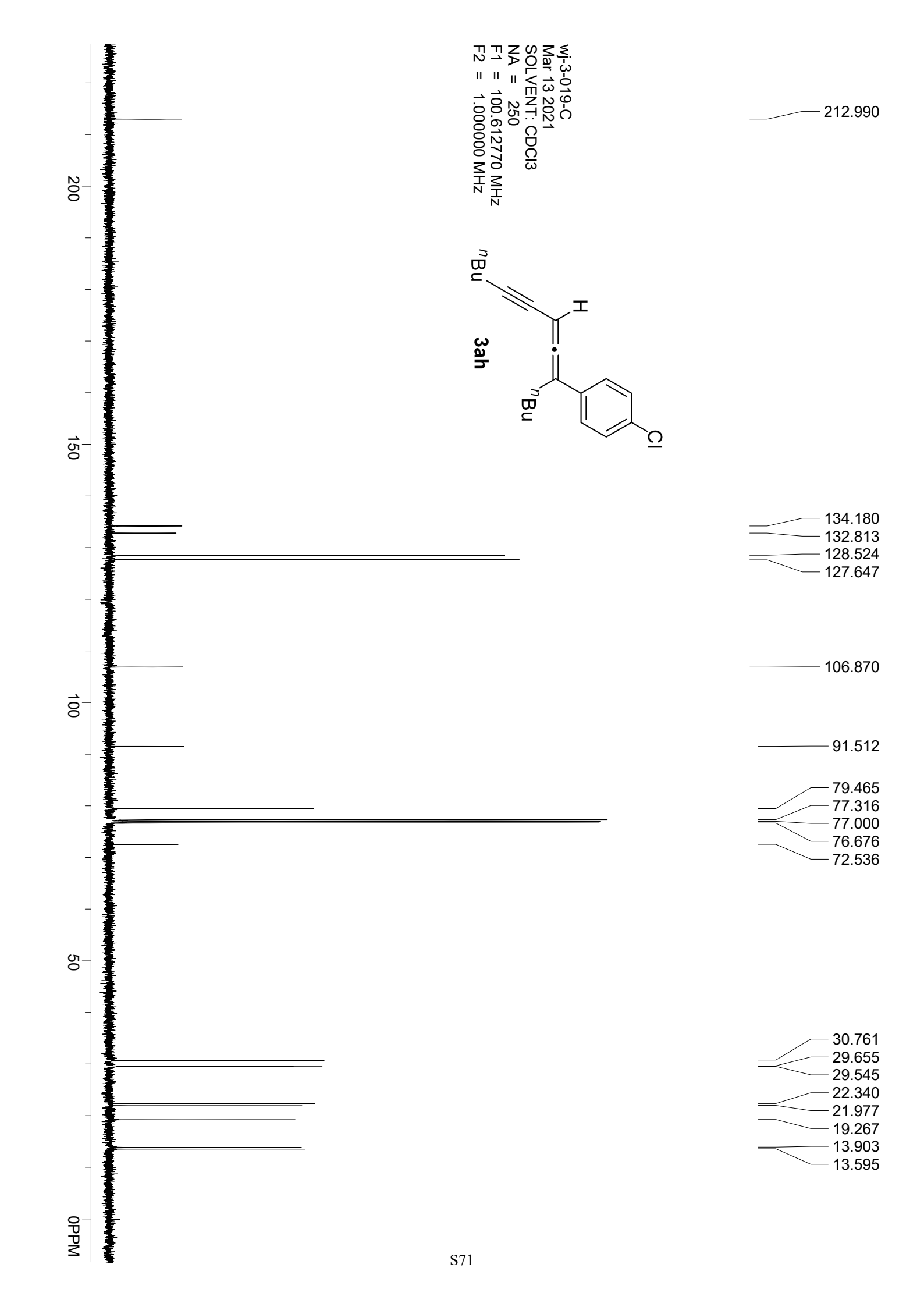
wj-3-018-F
Mar 13 2021
SOLVENT: CDCl3
NA = 16
F1 = 376.460724 MHz
F2 = 1.000000 MHz

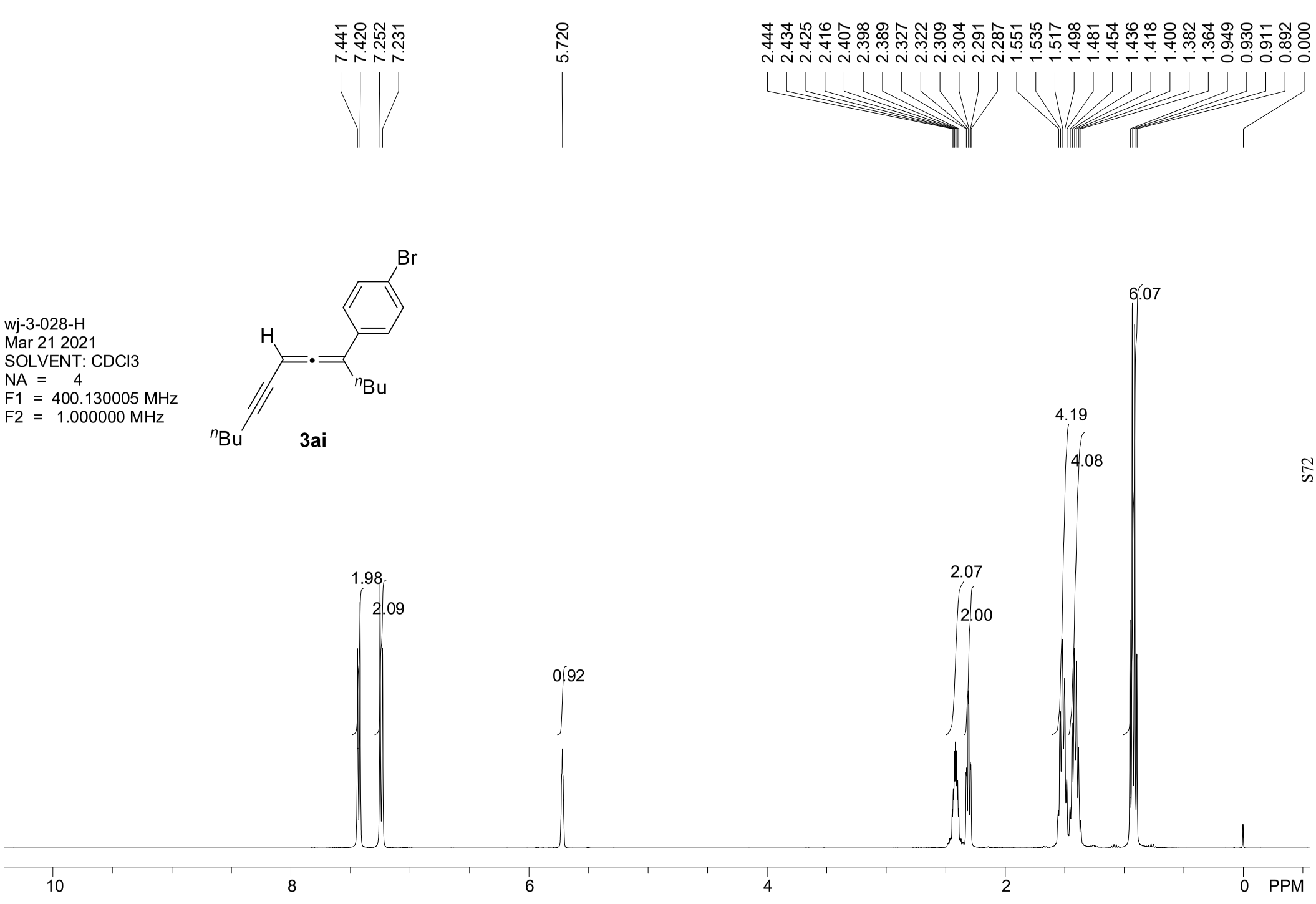


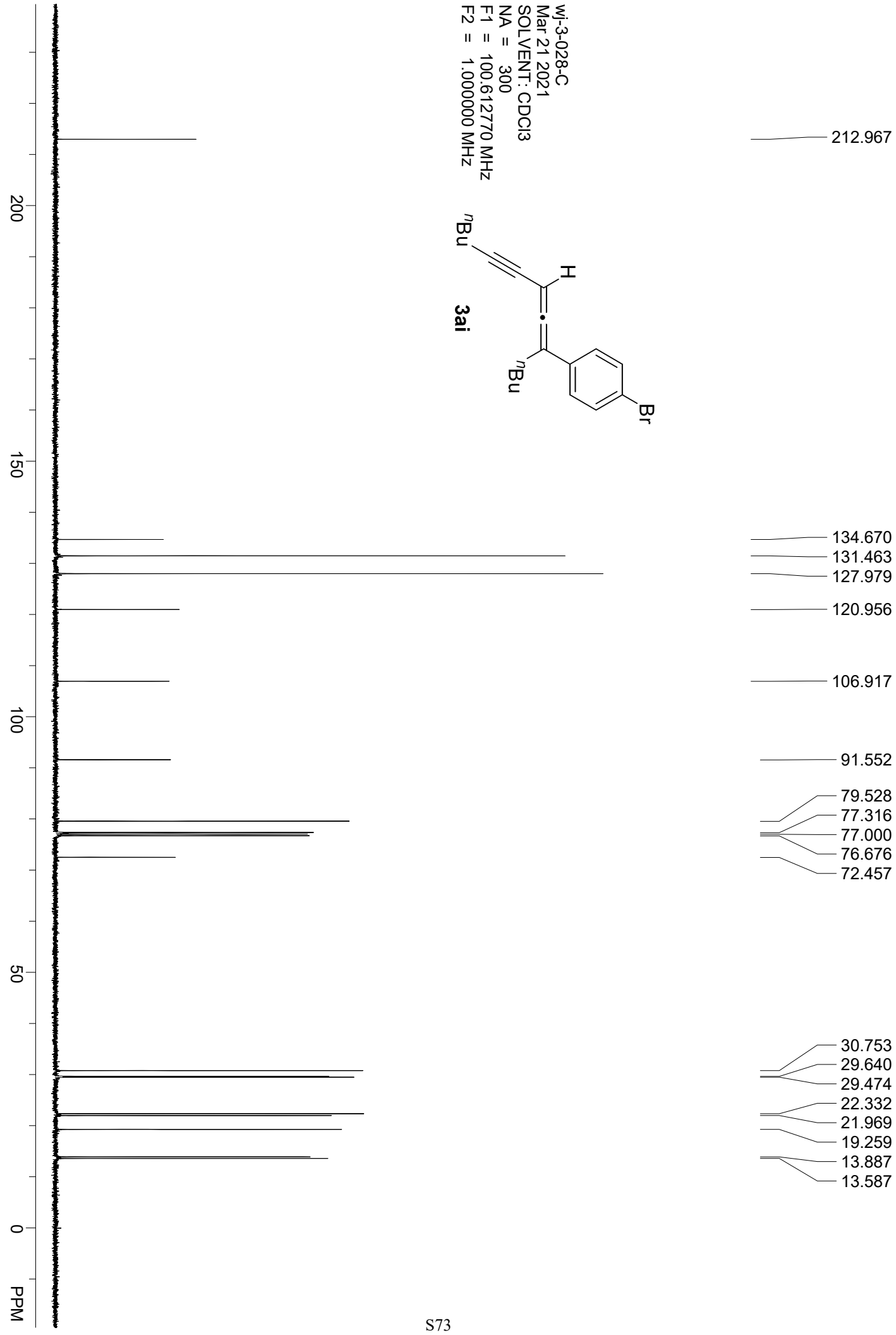
3ag

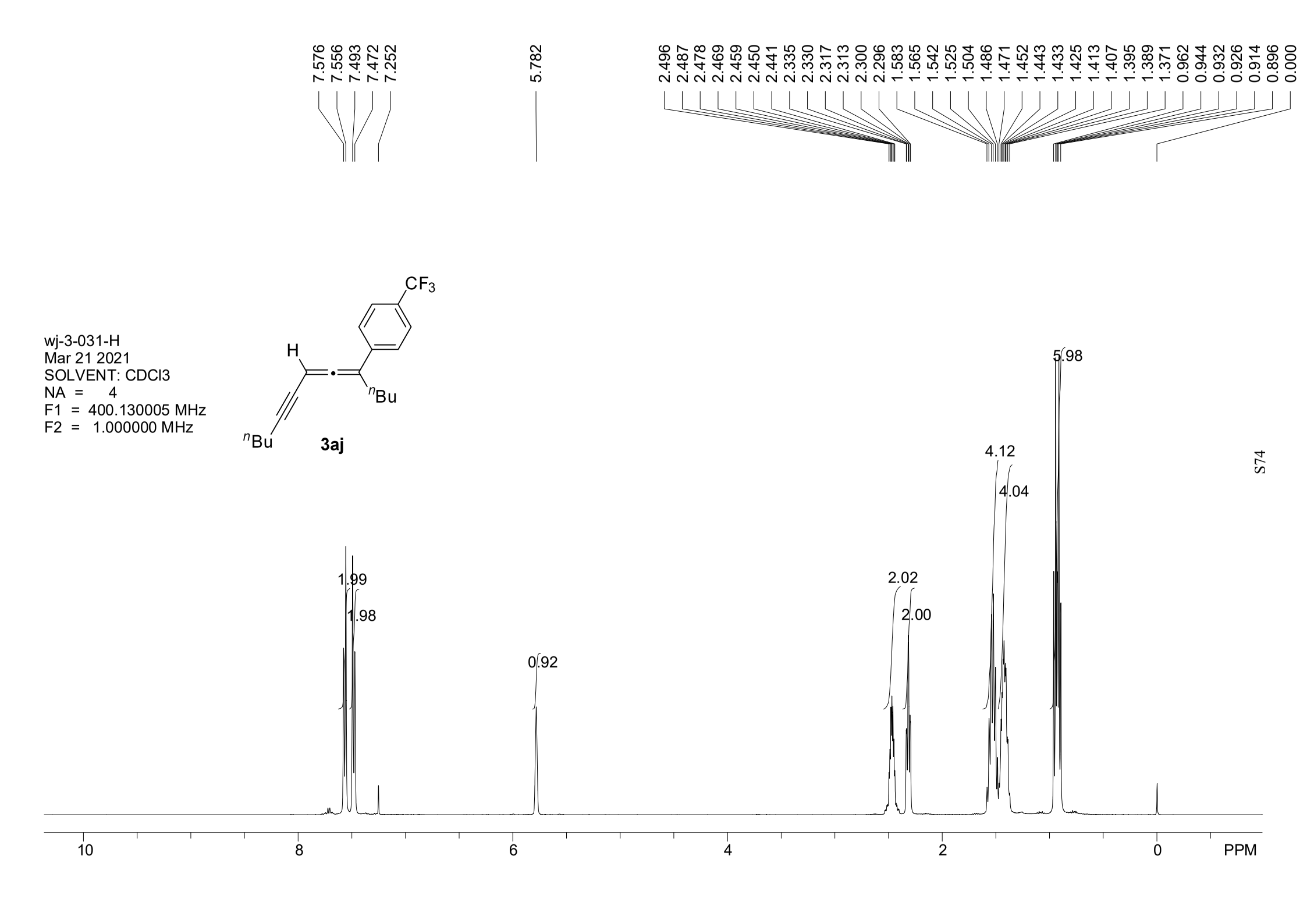


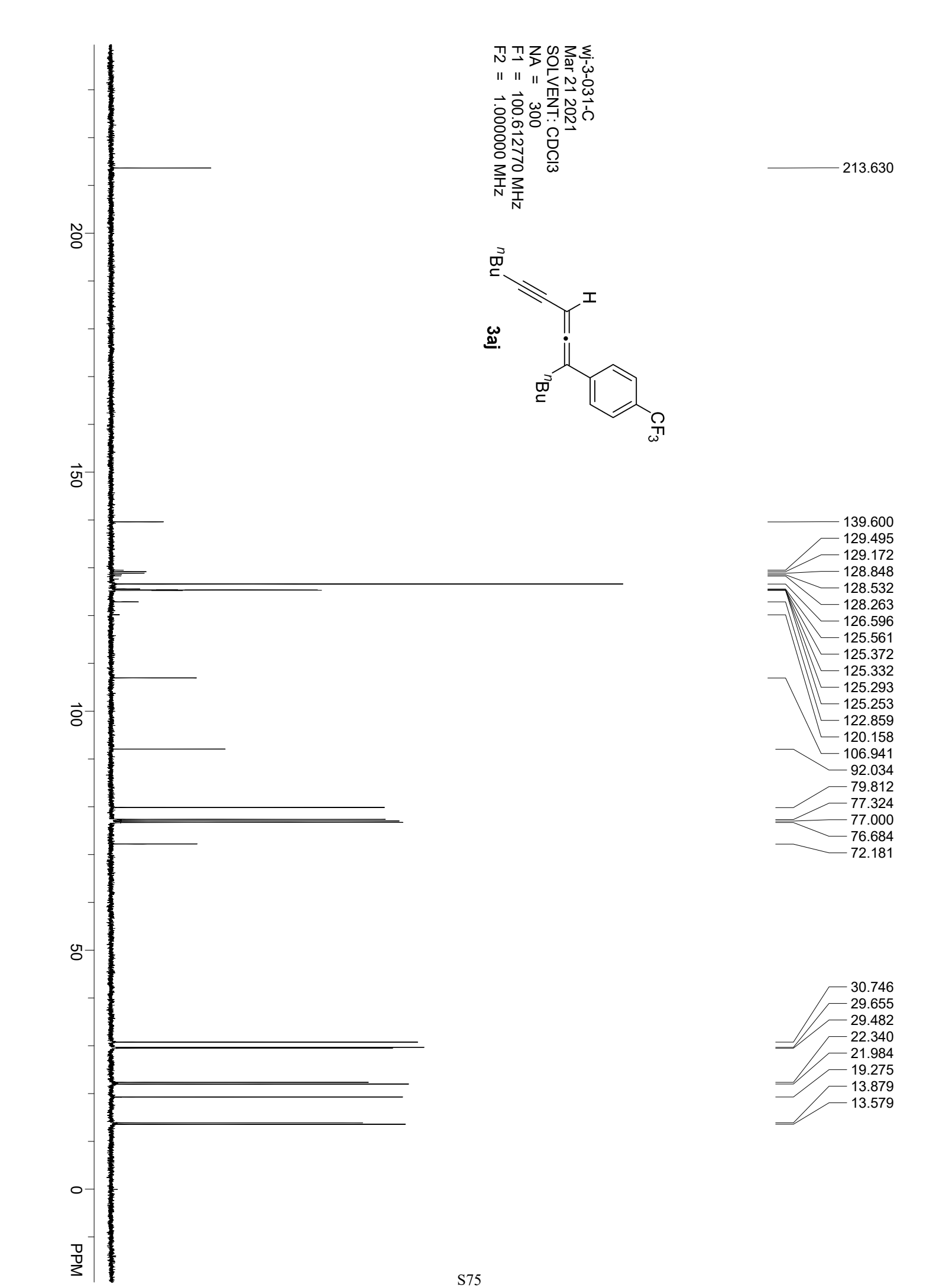

7.322
7.300
7.291
7.269
7.257
5.736
5.731
2.451
2.442
2.433
2.424
2.415
2.405
2.397
2.329
2.324
2.312
2.307
2.295
2.289
1.543
1.521
1.506
1.501
1.484
1.457
1.440
1.421
1.403
1.385
1.367
0.952
0.933
0.913
0.894
0.000
Cl
H
nBu
nBu
3ah
wj-3-019-H
Mar 13 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
S70
4.20
0.93
2.06
2.00
4.27
4.11
6.15
10
8
6
4
2
0
PPM

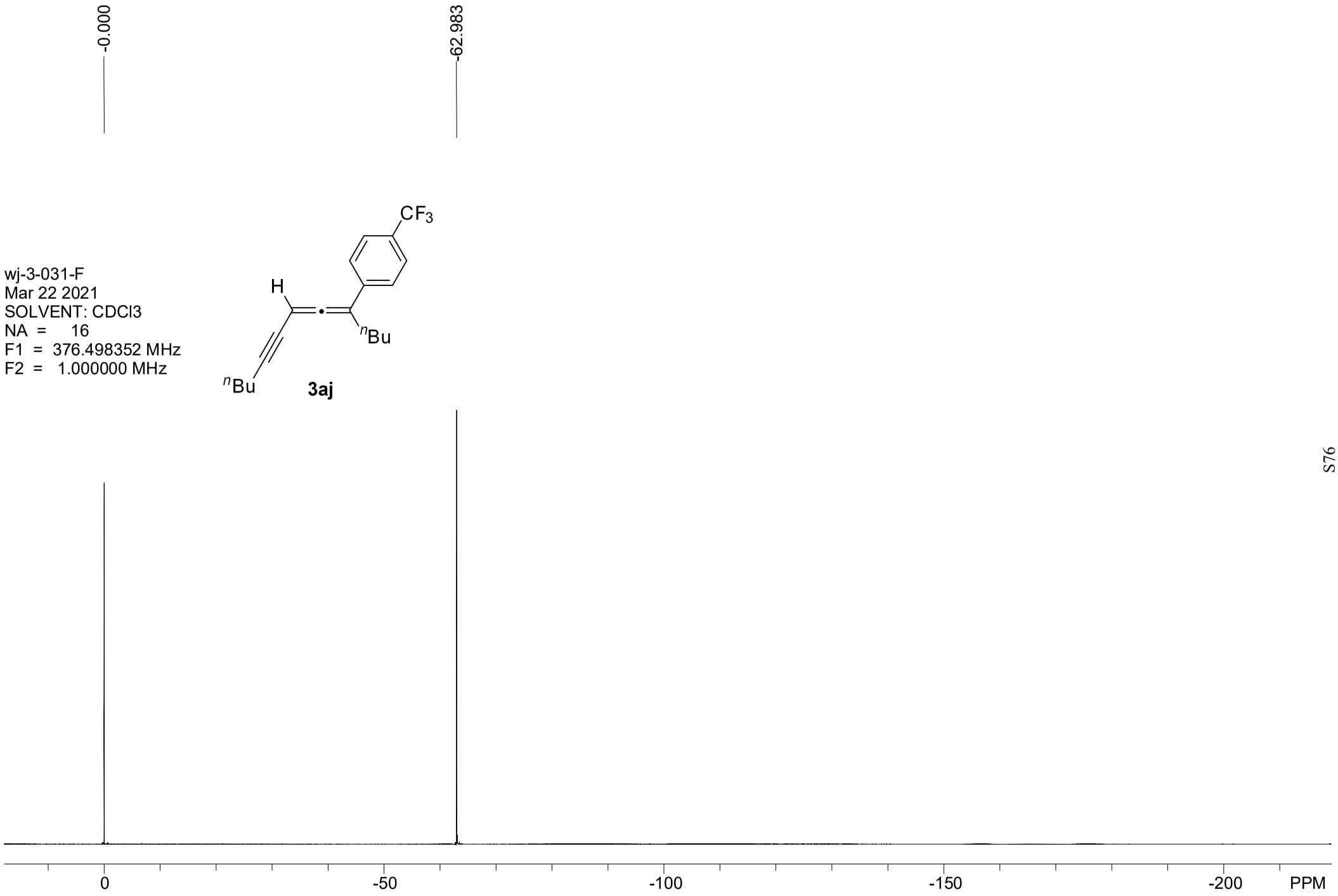

wj-3-019-C
Mar 13 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3ah
nBu
Cl
212.990
134.180
132.813
128.524
127.647
106.870
91.512
79.465
77.316
77.000
76.676
72.536
30.761
29.655
29.545
22.340
21.977
19.267
13.903
13.595
200
150
100
50
0PPM

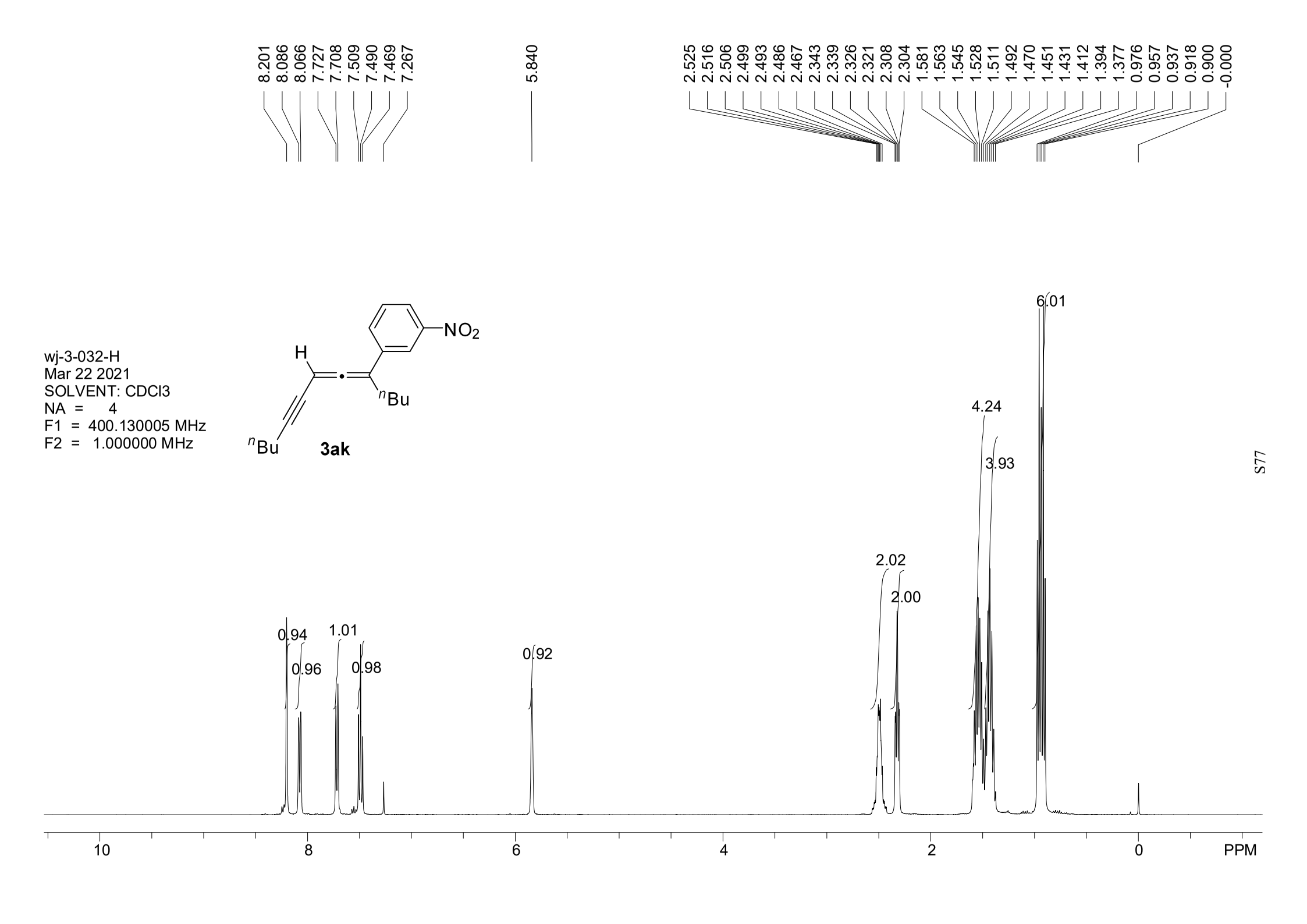

7.441
7.420
7.252
7.231
5.720
2.444
2.434
2.425
2.416
2.407
2.398
2.389
2.327
2.322
2.309
2.304
2.291
2.287
1.551
1.535
1.517
1.498
1.481
1.454
1.436
1.418
1.400
1.382
1.364
0.949
0.930
0.911
0.892
0.000
wj-3-028-H
Mar 21 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
Br
H
nBu
nBu
3ai
6.07
4.19
4.08
2.07
2.00
1.98
2.09
0.92
S72
10
8
6
4
2
0
PPM

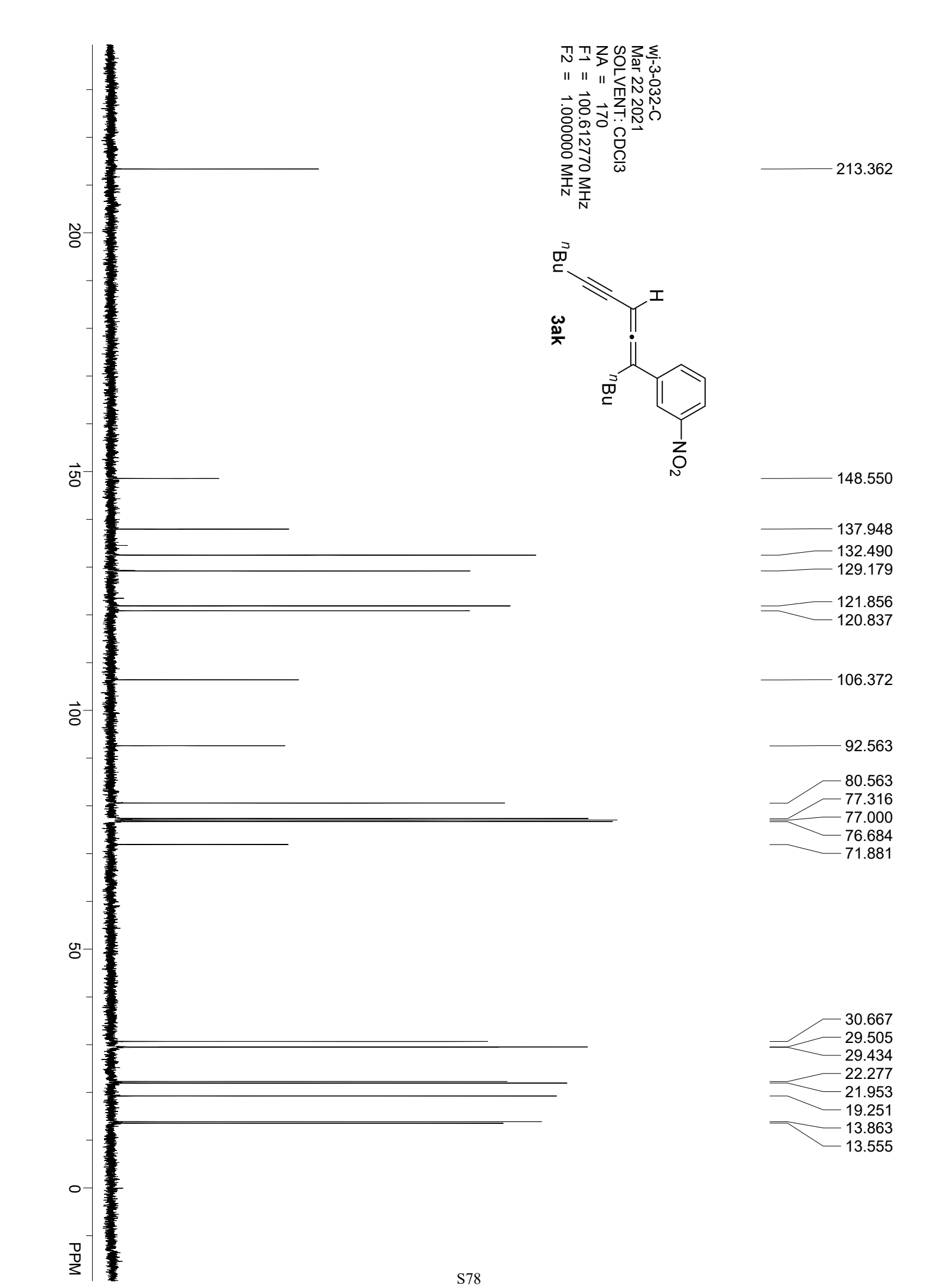

wj-3-028-C
Mar 21 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
Br
3ai
212.967
134.670
131.463
127.979
120.956
106.917
91.552
79.528
77.316
77.000
76.676
72.457
30.753
29.640
29.474
22.332
21.969
19.259
13.887
13.587
200
150
100
50
0
PPM

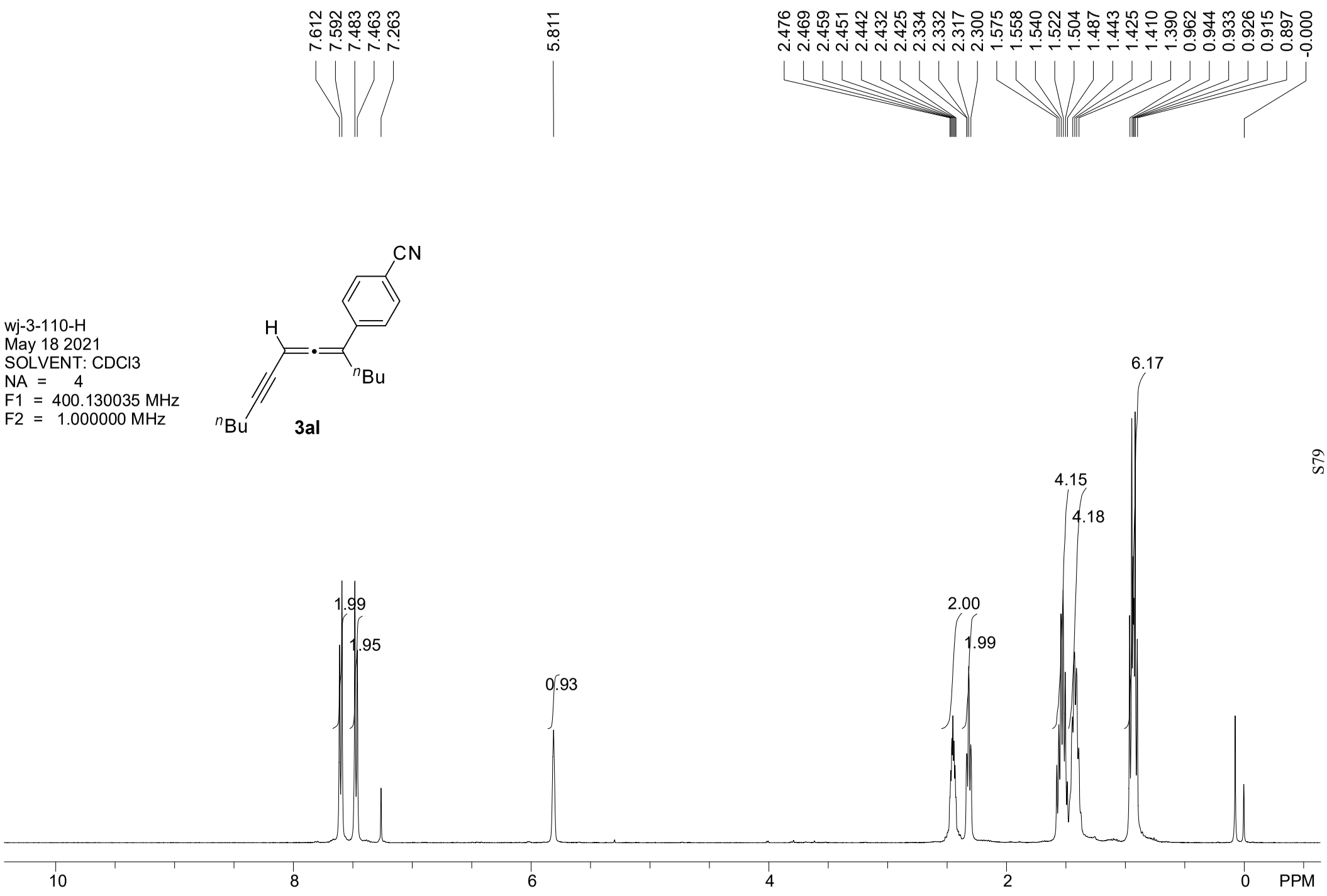

7.576
7.556
7.493
7.472
7.252
5.782
2.496
2.487
2.478
2.469
2.459
2.450
2.441
2.335
2.330
2.317
2.313
2.300
2.296
1.583
1.565
1.542
1.525
1.504
1.486
1.471
1.452
1.443
1.433
1.425
1.413
1.407
1.395
1.389
1.371
0.962
0.944
0.932
0.926
0.914
0.896
0.000
wj-3-031-H
Mar 21 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
CF3
H
nBu
nBu
3aj
S74
1.99
1.98
0.92
2.02
2.00
4.12
4.04
5.98
10
8
6
4
2
0
PPM

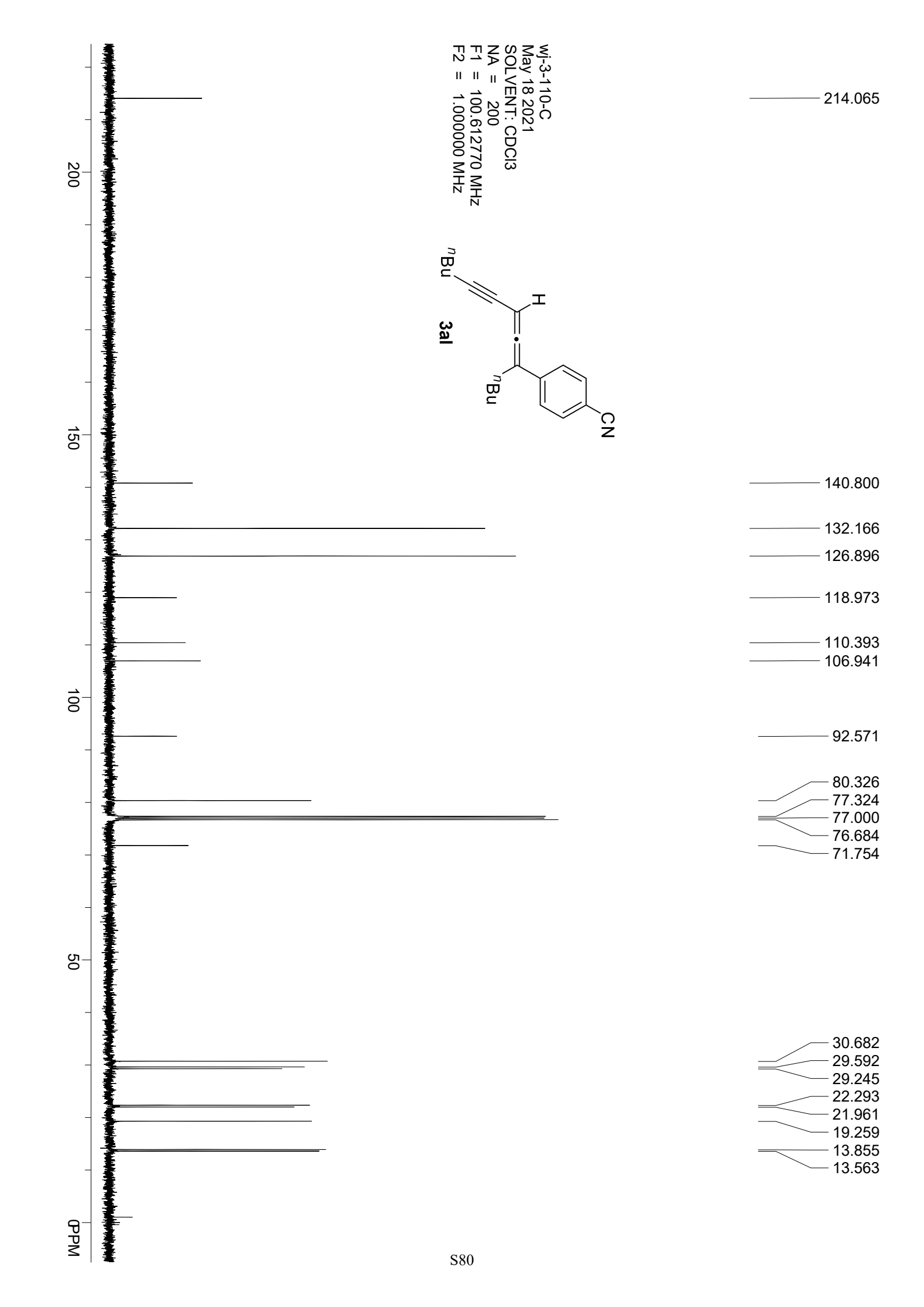

wj-3-031-C
Mar 21 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
CF3
3aj
213.630
139.600
129.495
129.172
128.848
128.532
128.263
126.596
125.561
125.372
125.332
125.293
125.253
122.859
120.158
106.941
92.034
79.812
77.324
77.000
76.684
72.181
30.746
29.655
29.482
22.340
21.984
19.275
13.879
13.579
200
150
100
50
0
PPM

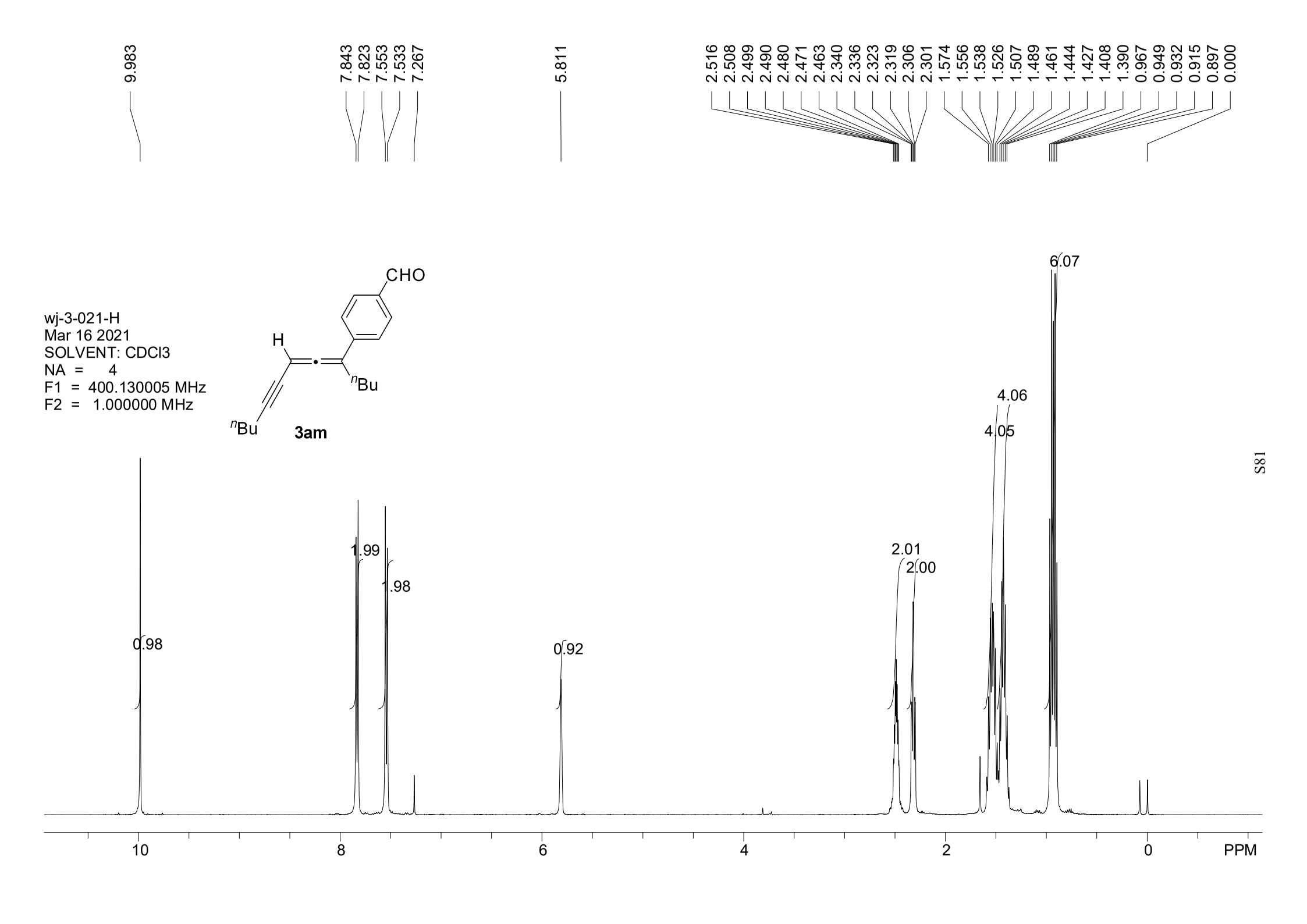

-0.000
-62.983
wj-3-031-F
Mar 22 2021
SOLVENT: CDCl3
NA = 16
F1 = 376.498352 MHz
F2 = 1.000000 MHz
CF3
H
nBu
nBu
3aj
0
-50
-100
-150
-200
PPM

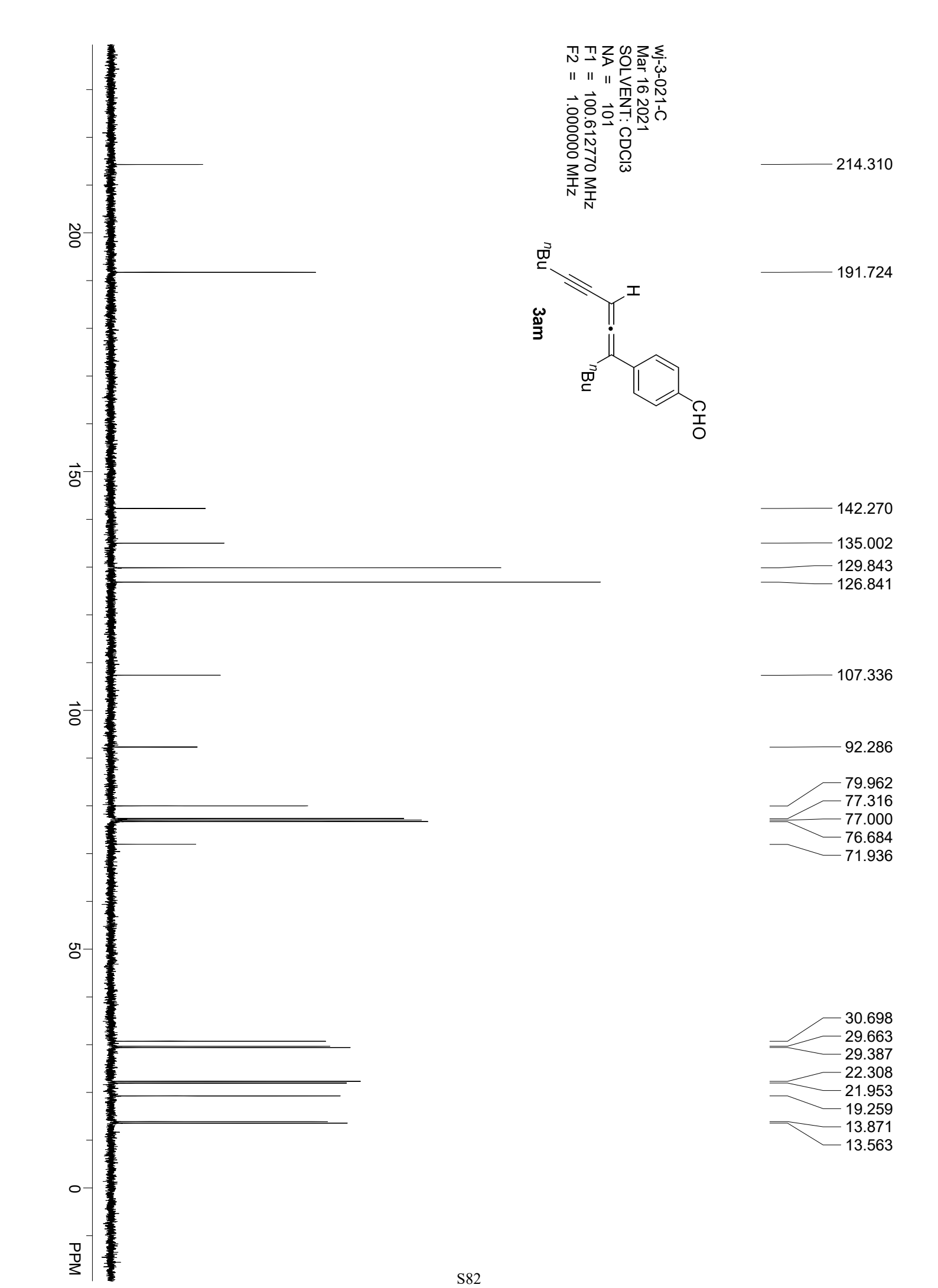

8.201
8.086
8.066
7.727
7.708
7.509
7.490
7.469
7.267
5.840
2.525
2.516
2.506
2.499
2.493
2.486
2.467
2.343
2.339
2.326
2.321
2.308
2.304
1.581
1.563
1.545
1.528
1.511
1.492
1.470
1.451
1.431
1.412
1.394
1.377
0.976
0.957
0.937
0.918
0.900
-0.000
wj-3-032-H
Mar 22 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
NO2
H
nBu
nBu
3ak
6.01
4.24
3.93
2.02
2.00
0.94
0.96
1.01
0.98
0.92
S77
10
8
6
4
2
0
PPM

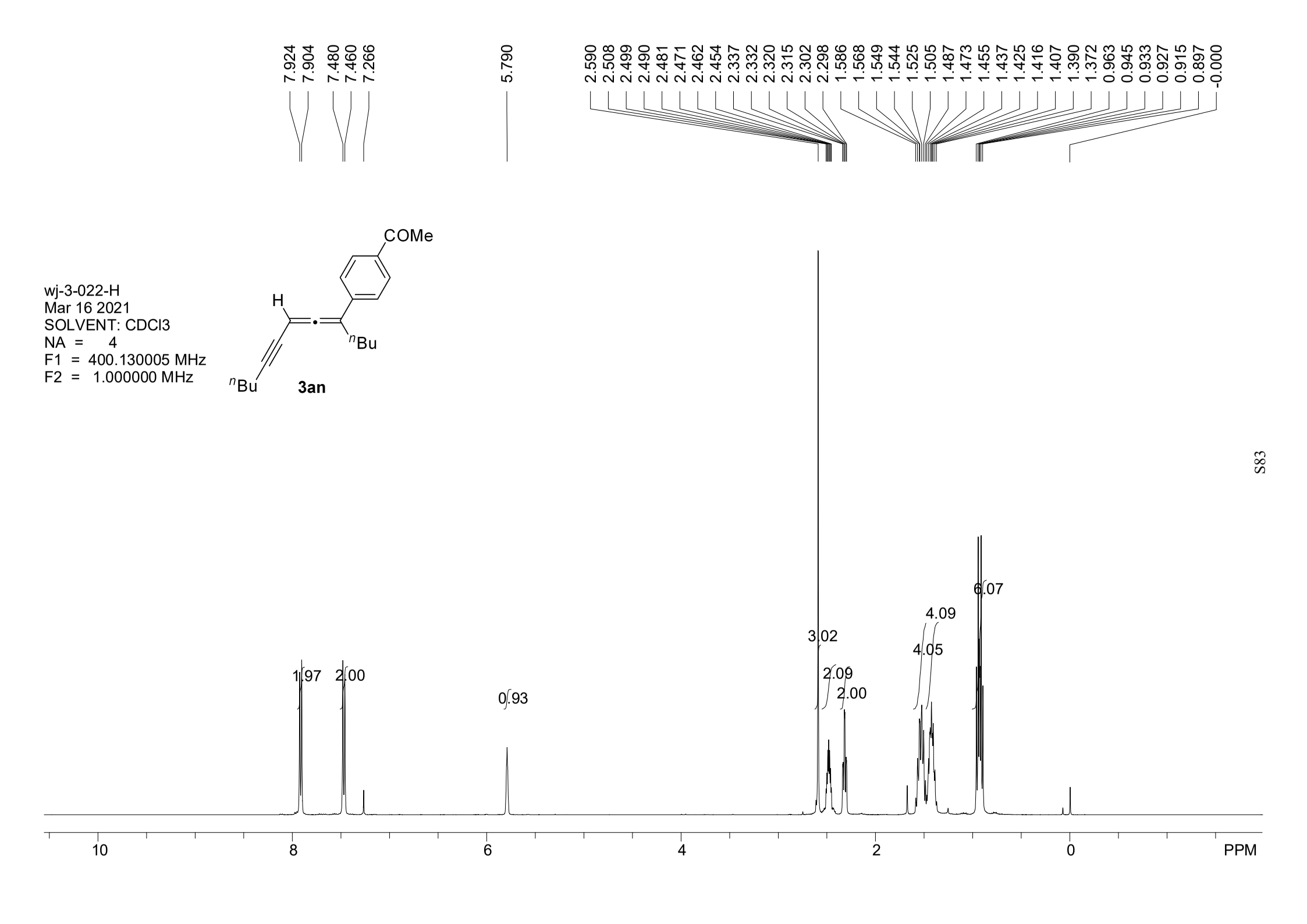

wj-3-032-C
Mar 22 2021
SOLVENT: CDCl3
NA = 170
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
NO2
3ak
213.362
148.550
137.948
132.490
129.179
121.856
120.837
106.372
92.563
80.563
77.316
77.000
76.684
71.881
30.667
29.505
29.434
22.277
21.953
19.251
13.863
13.555
200
150
100
50
0
PPM

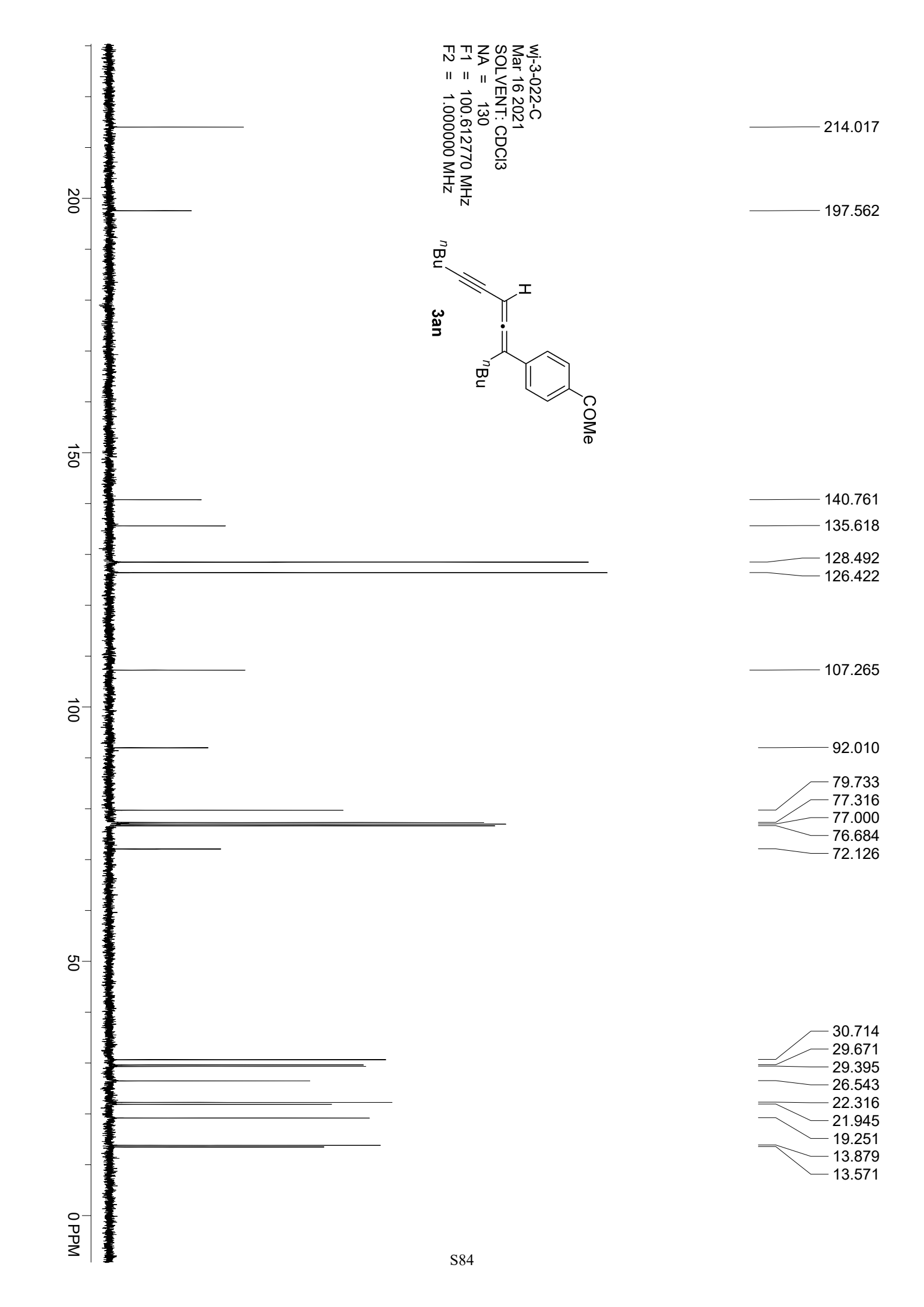

7.612
7.592
7.483
7.463
7.263
5.811
2.476
2.469
2.459
2.451
2.442
2.432
2.425
2.334
2.332
2.317
2.300
1.575
1.558
1.540
1.522
1.504
1.487
1.443
1.425
1.410
1.390
0.962
0.944
0.933
0.926
0.915
0.897
-0.000
wj-3-110-H
May 18 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
CN
H
nBu
nBu
3al
6.17
4.15
4.18
1.99
1.95
0.93
2.00
1.99
S79
10
8
6
4
2
0
PPM

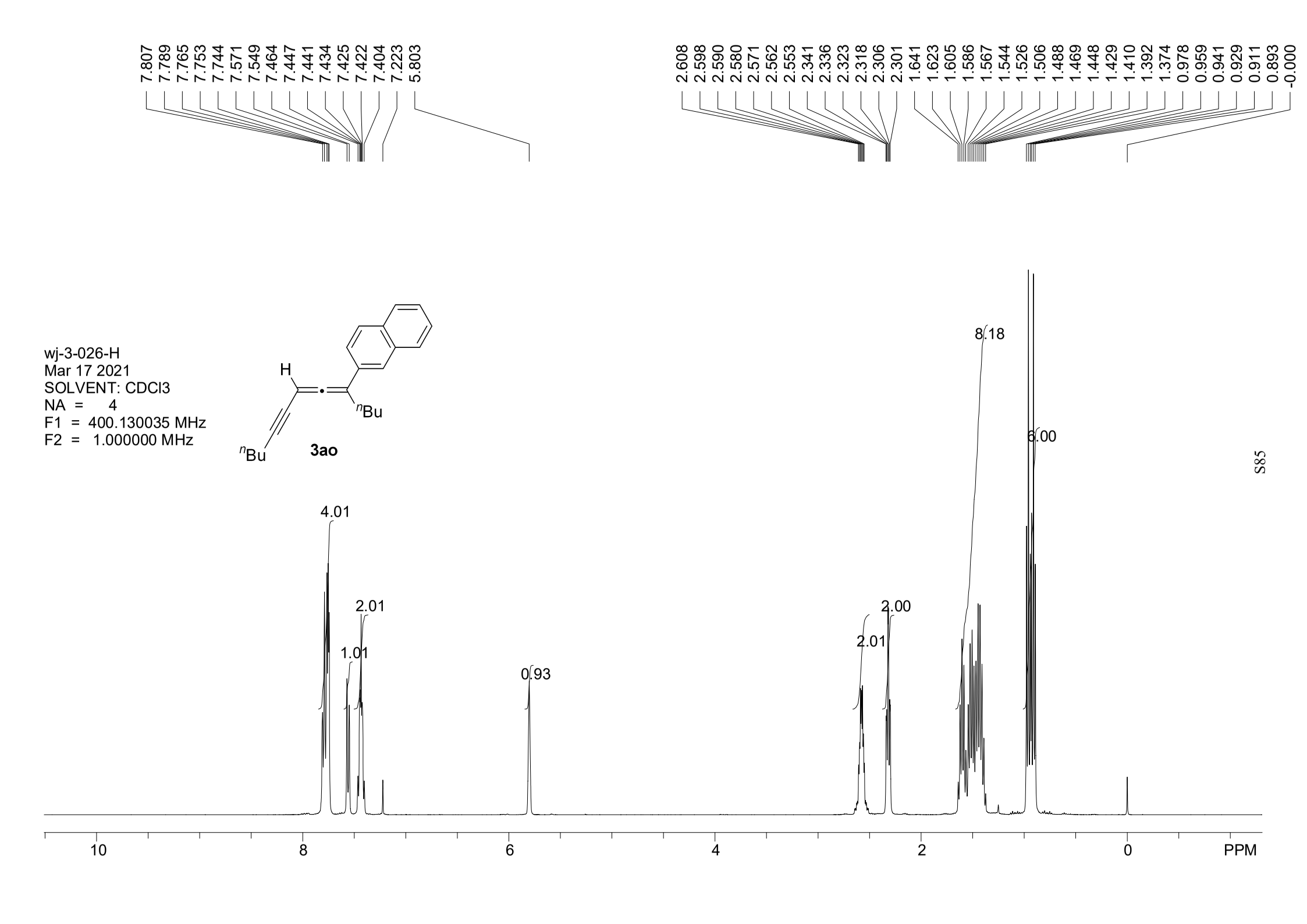

wj-3-110-C
May 18 2021
SOLVENT: CDCl3
NA = 200
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
CN
3al
214.065
140.800
132.166
126.896
118.973
110.393
106.941
92.571
80.326
77.324
77.000
76.684
71.754
30.682
29.592
29.245
22.293
21.961
19.259
13.855
13.563
200
150
100
50
0
PPM

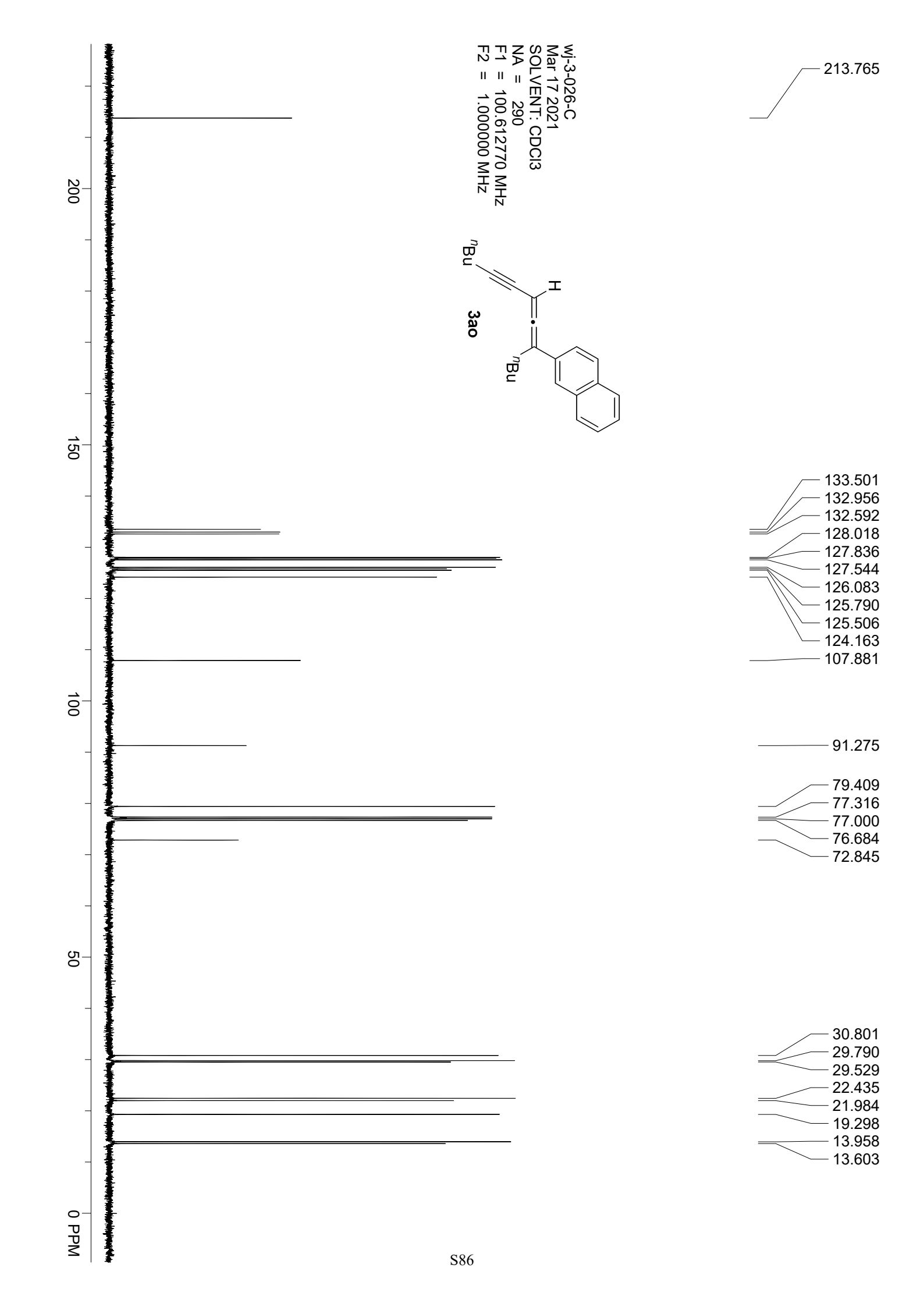

9.983
7.843
7.823
7.553
7.533
7.267
5.811
2.516
2.508
2.499
2.490
2.480
2.471
2.463
2.340
2.336
2.323
2.319
2.306
2.301
1.574
1.556
1.538
1.526
1.507
1.489
1.461
1.444
1.427
1.408
1.390
0.967
0.949
0.932
0.915
0.897
0.000
wj-3-021-H
Mar 16 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
CHO
H
nBu
nBu
3am
6.07
4.06
4.05
2.01
2.00
1.99
1.98
0.98
0.92
S81
10
8
6
4
2
0
PPM

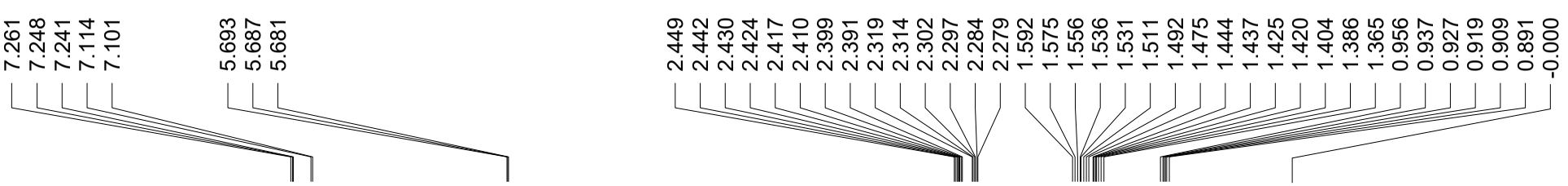

wj-3-021-C
Mar 16 2021
SOLVENT: CDCl3
NA = 101
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3am
nBu
CHO
214.310
191.724
142.270
135.002
129.843
126.841
107.336
92.286
79.962
77.316
77.000
76.684
71.936
30.698
29.663
29.387
22.308
21.953
19.259
13.871
13.563
200
150
100
50
0
PPM


7.924
7.904
7.480
7.460
7.266
5.790
2.590
2.508
2.499
2.490
2.481
2.471
2.462
2.454
2.337
2.332
2.320
2.315
2.302
2.298
1.586
1.568
1.549
1.544
1.525
1.505
1.487
1.473
1.455
1.437
1.425
1.416
1.407
1.390
1.372
0.963
0.945
0.933
0.927
0.915
0.897
-0.000
wj-3-022-H
Mar 16 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
COMe
H
nBu
nBu
3an
1.97
2.00
0.93
3.02
2.09
2.00
4.05
4.09
6.07
10
8
6
4
2
0
PPM

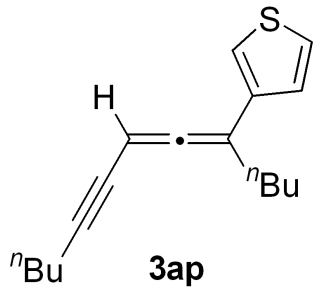

wj-3-022-C
Mar 16 2021
SOLVENT: CDCl3
NA = 130
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
COMe
3an
214.017
197.562
140.761
135.618
128.492
126.422
107.265
92.010
79.733
77.316
77.000
76.684
72.126
30.714
29.671
29.395
26.543
22.316
21.945
19.251
13.879
13.571
200
150
100
50
0 PPM


7.807
7.789
7.765
7.753
7.744
7.571
7.549
7.464
7.447
7.441
7.434
7.425
7.422
7.404
7.223
5.803
2.608
2.598
2.590
2.580
2.571
2.562
2.553
2.341
2.336
2.323
2.318
2.306
2.301
1.641
1.623
1.605
1.586
1.567
1.544
1.526
1.506
1.488
1.469
1.448
1.429
1.410
1.392
1.374
0.978
0.959
0.941
0.929
0.911
0.893
-0.000
wj-3-026-H
Mar 17 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
H
nBu
nBu
3ao
8.18
6.00
4.01
2.01
1.01
0.93
2.01
2.00
S85
10
8
6
4
2
0
PPM

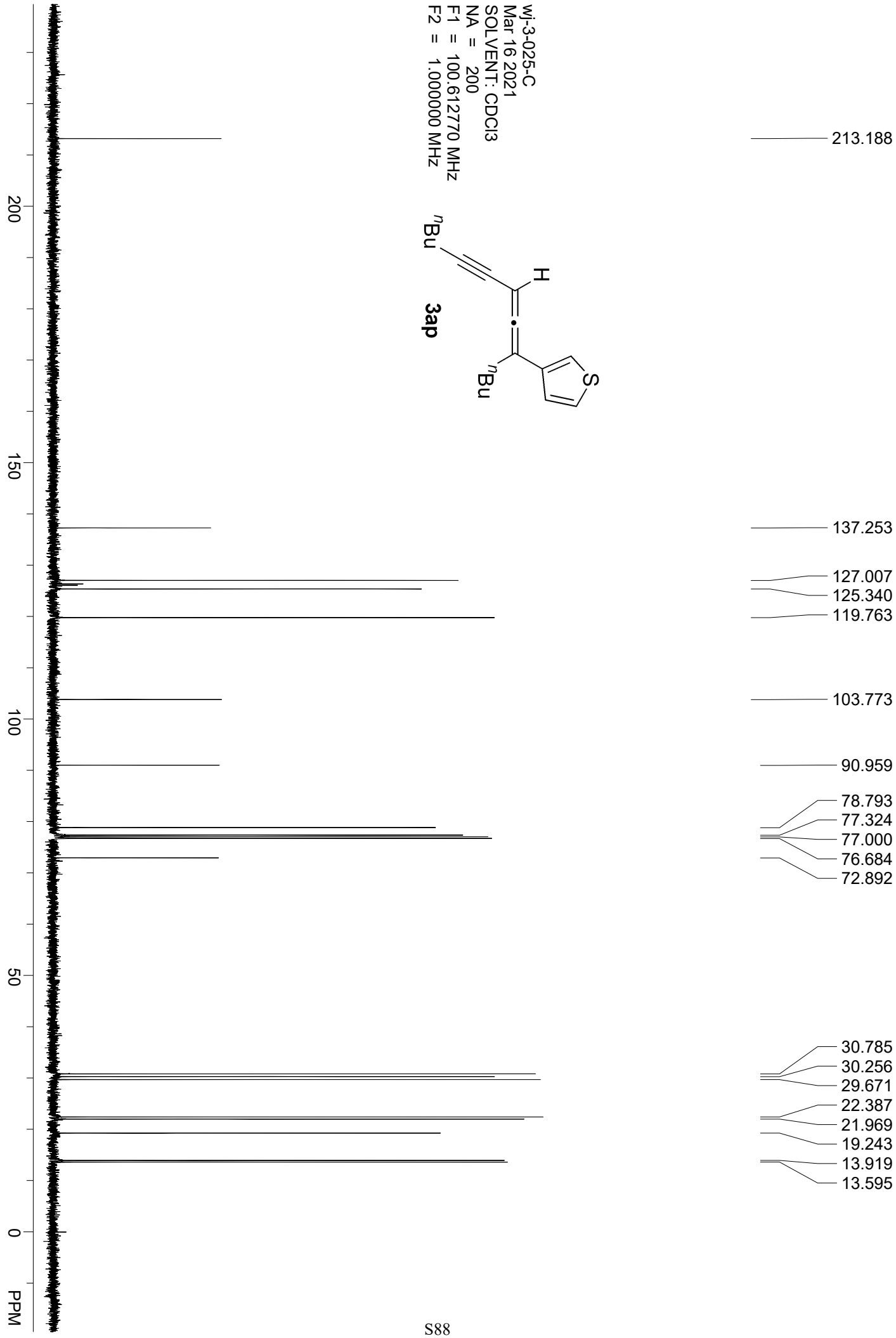

wj-3-026-C
Mar 17 2021
SOLVENT: CDCl3
NA = 290
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3ao
nBu
213.765
133.501
132.956
132.592
128.018
127.836
127.544
126.083
125.790
125.506
124.163
107.881
91.275
79.409
77.316
77.000
76.684
72.845
30.801
29.790
29.529
22.435
21.984
19.298
13.958
13.603
200
150
100
50
0 PPM

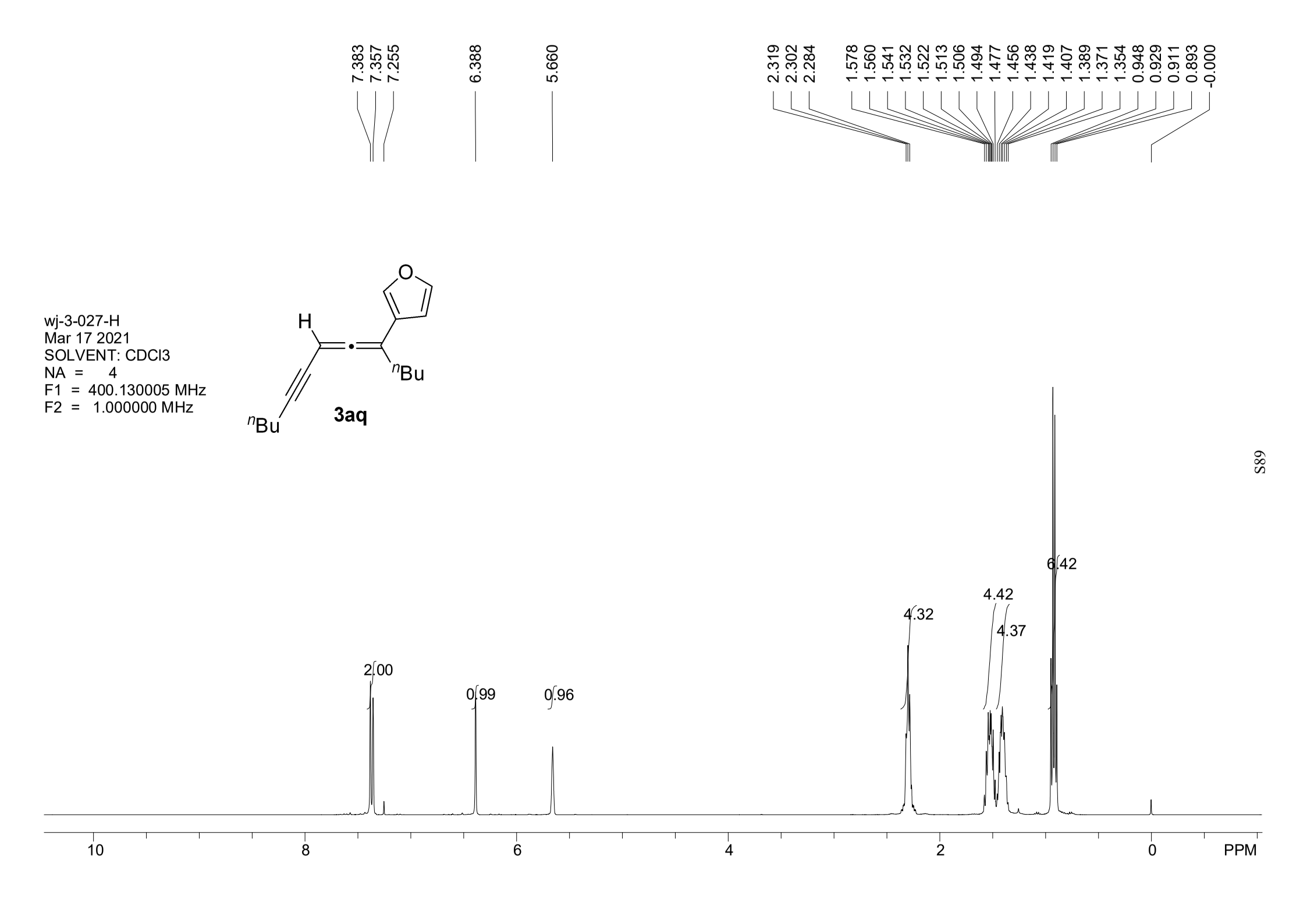


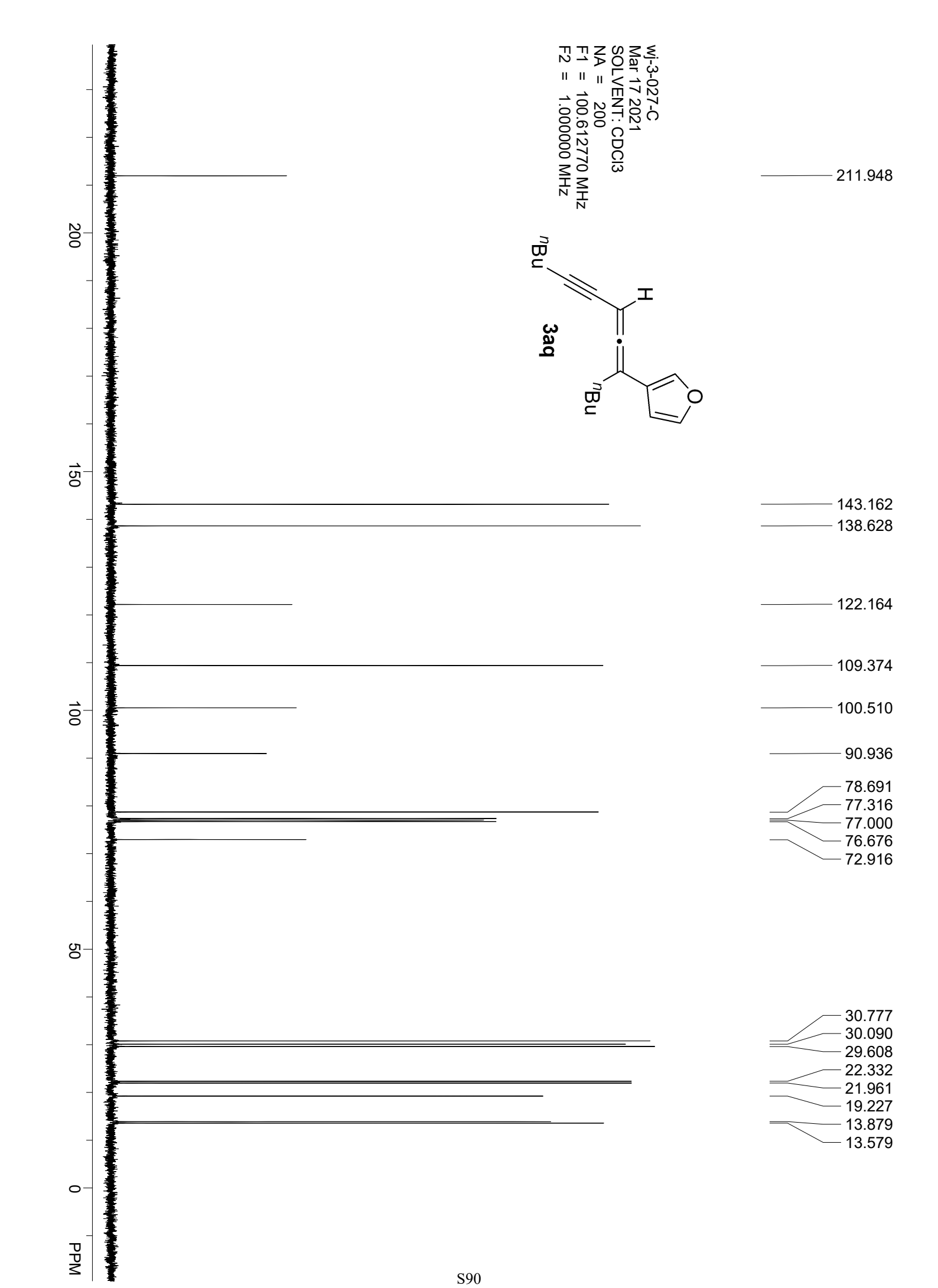
wj-3-025-H
Mar 16 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz

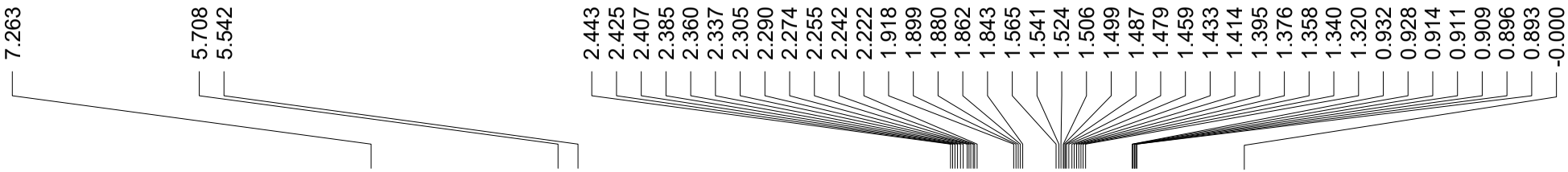


3ap

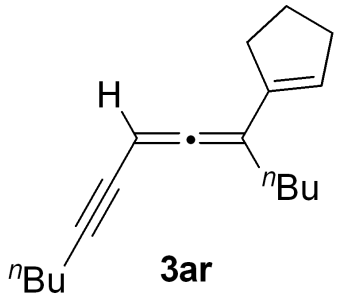

wj-3-025-C
Mar 16 2021
SOLVENT: CDCl3
NA = 200
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
S
3ap
213.188
137.253
127.007
125.340
119.763
103.773
90.959
78.793
77.324
77.000
76.684
72.892
30.785
30.256
29.671
22.387
21.969
19.243
13.919
13.595
200
150
100
50
0
PPM


7.383
7.357
7.255
6.388
5.660
2.319
2.302
2.284
1.578
1.560
1.541
1.532
1.522
1.513
1.506
1.494
1.477
1.456
1.438
1.419
1.407
1.389
1.371
1.354
0.948
0.929
0.911
0.893
-0.000
wj-3-027-H
Mar 17 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
O
H
nBu
nBu
3aq
2.00
0.99
0.96
4.32
4.42
4.37
6.42
10
8
6
4
2
0
PPM

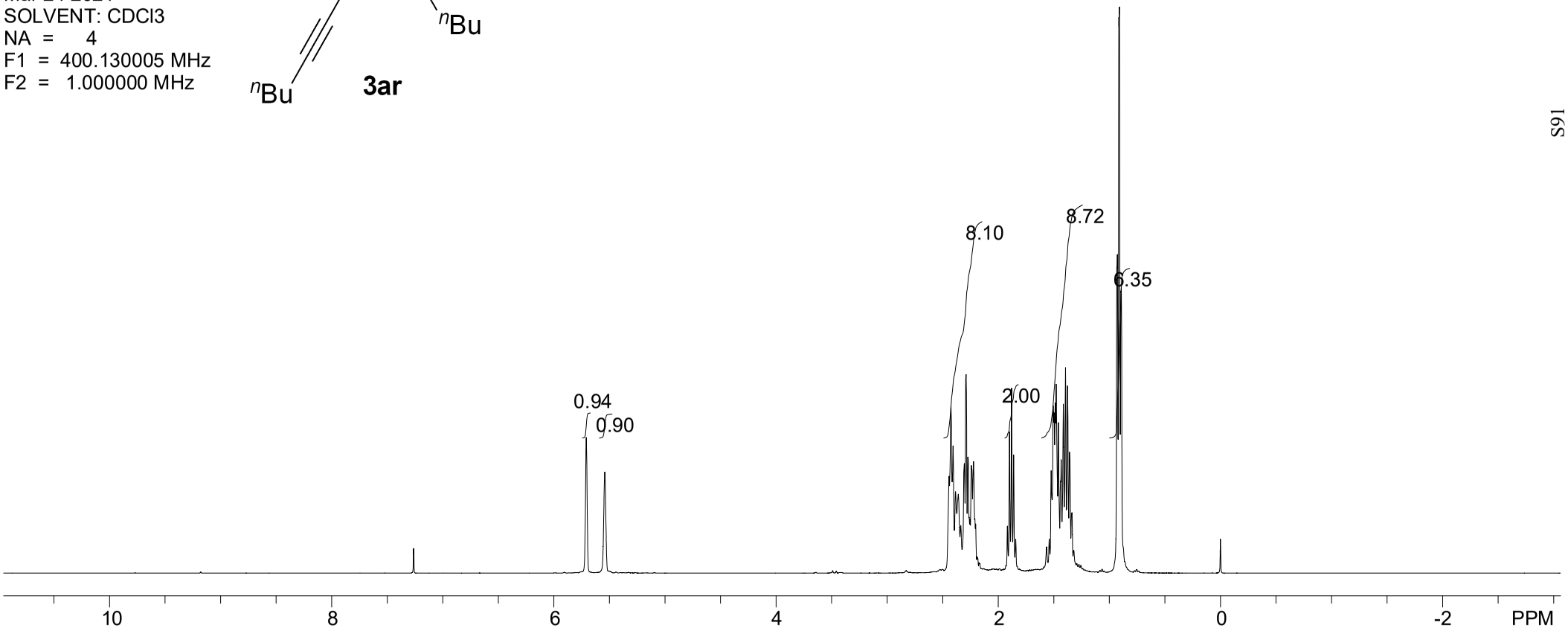

wj-3-027-C
Mar 17 2021
SOLVENT: CDCl3
NA = 200
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3aq
nBu
O
211.948
143.162
138.628
122.164
109.374
100.510
90.936
78.691
77.316
77.000
76.676
72.916
30.777
30.090
29.608
22.332
21.961
19.227
13.879
13.579
200
150
100
50
0
PPM

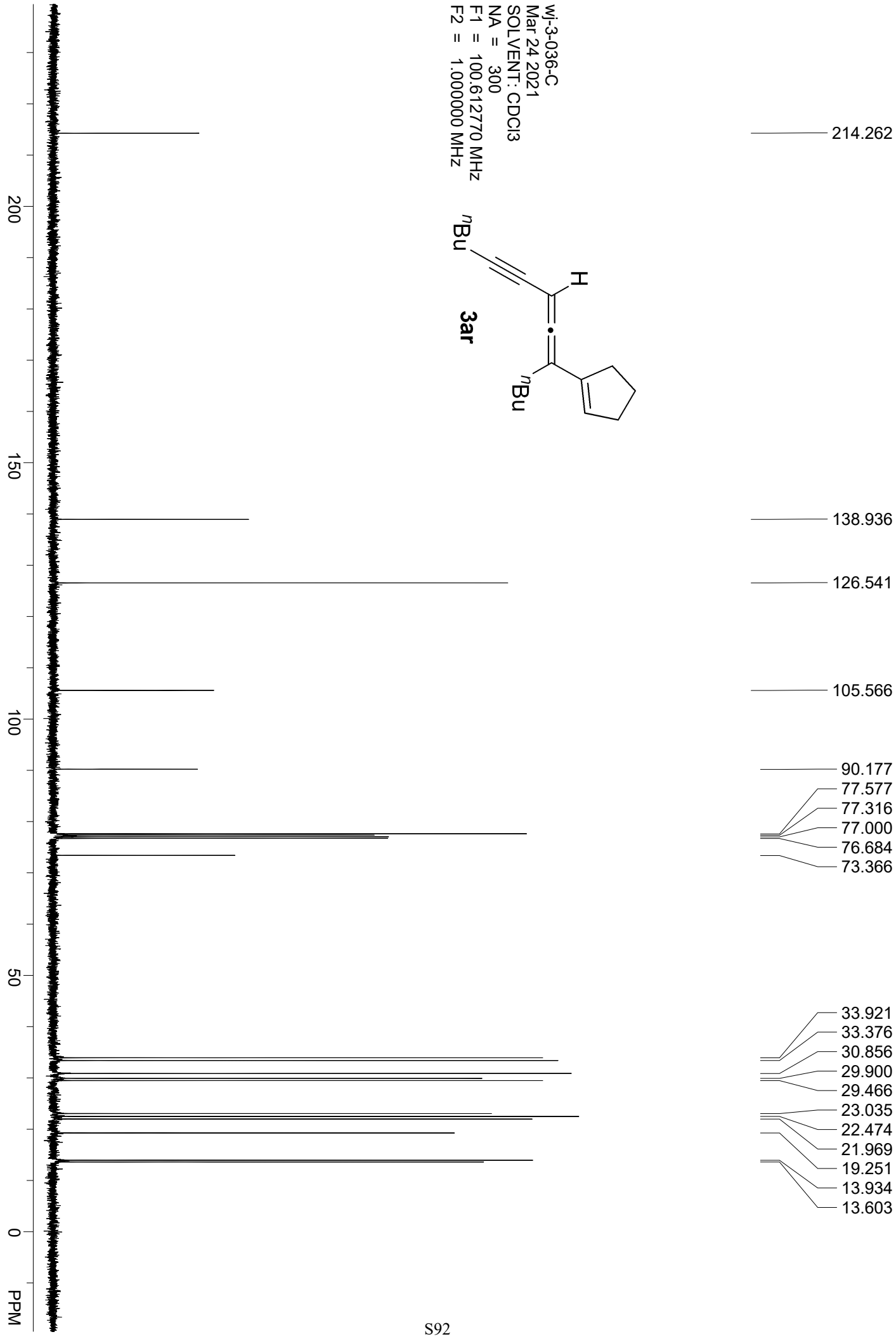


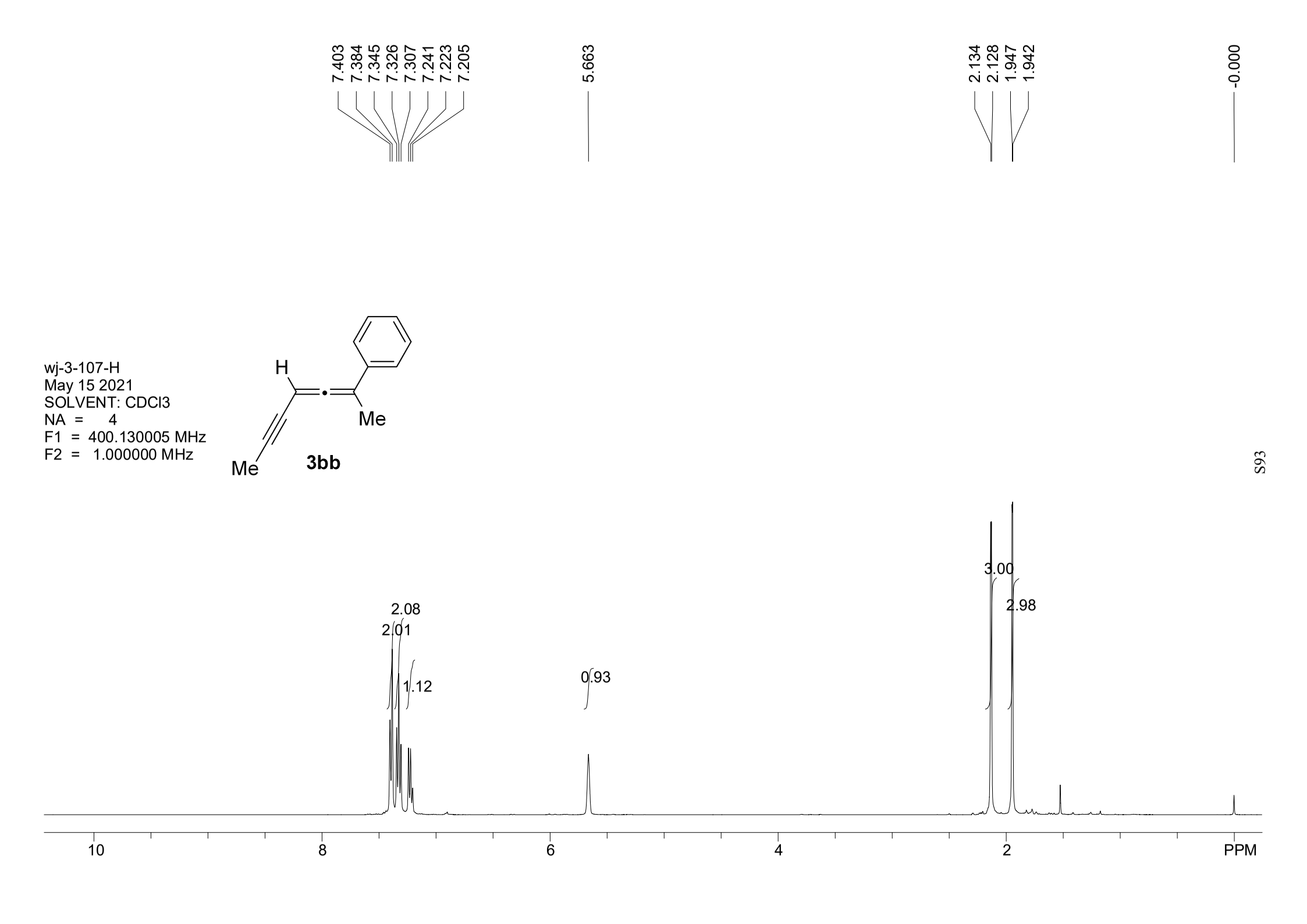
wj-3-036-H
Mar 24 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz

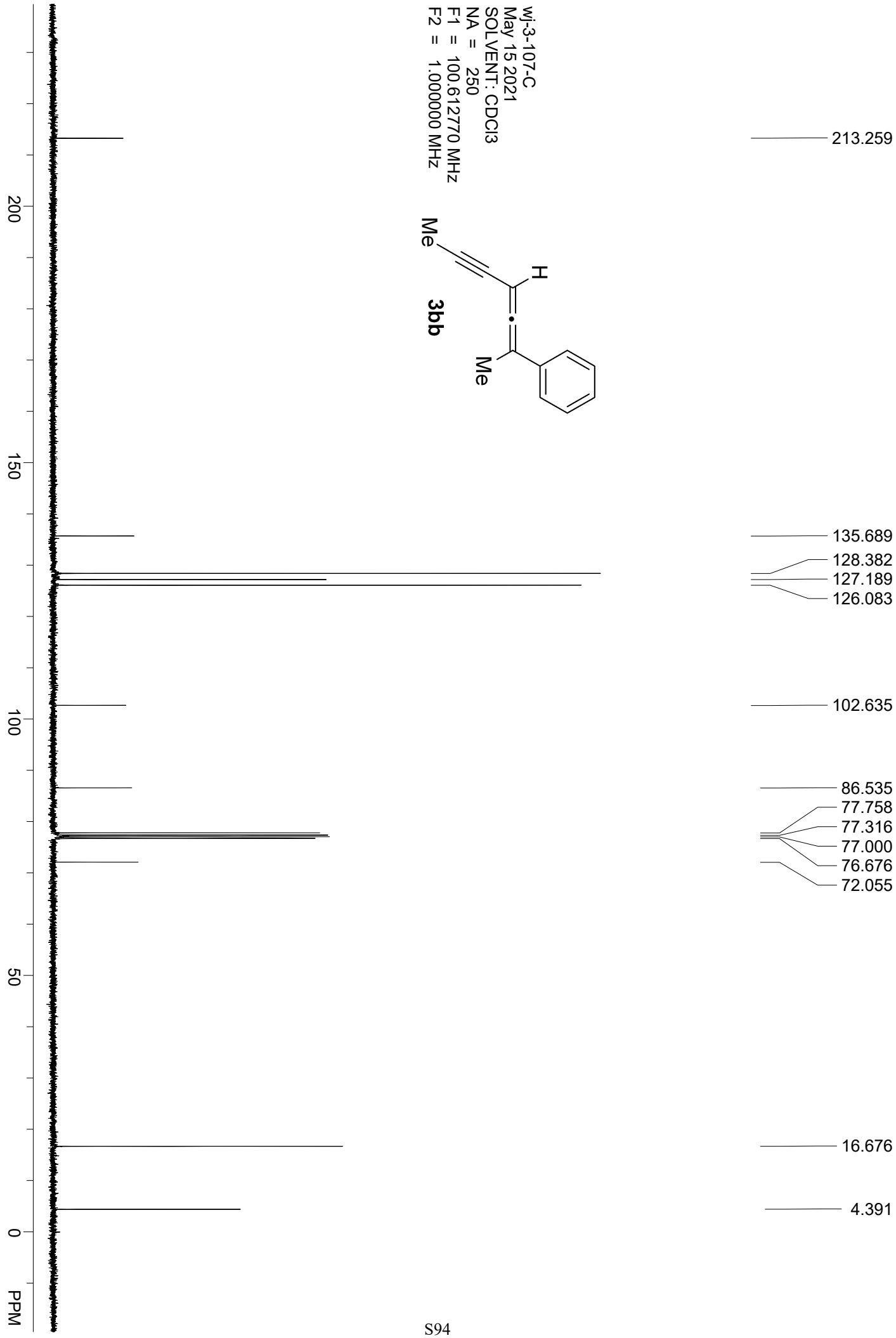


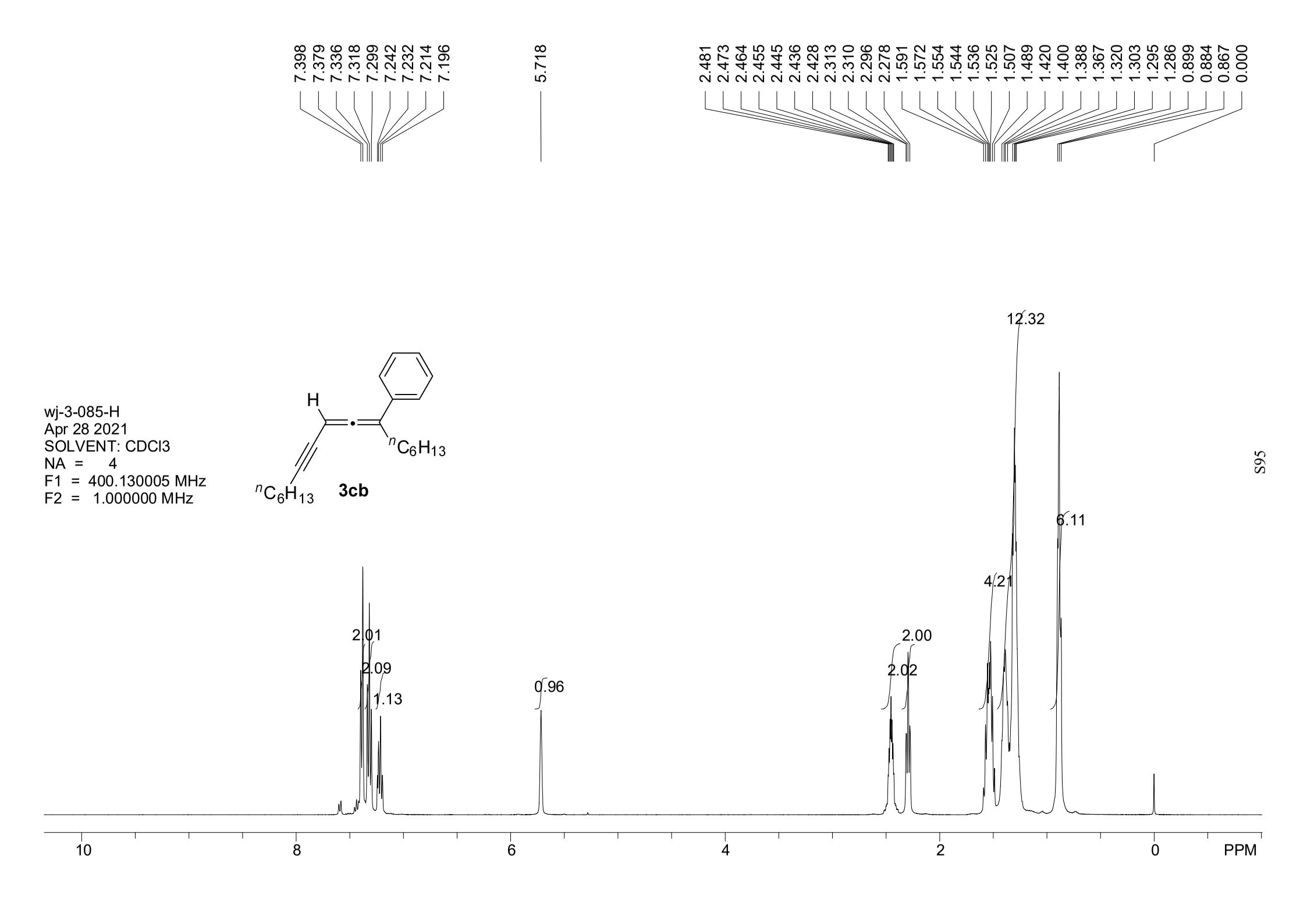
3ar

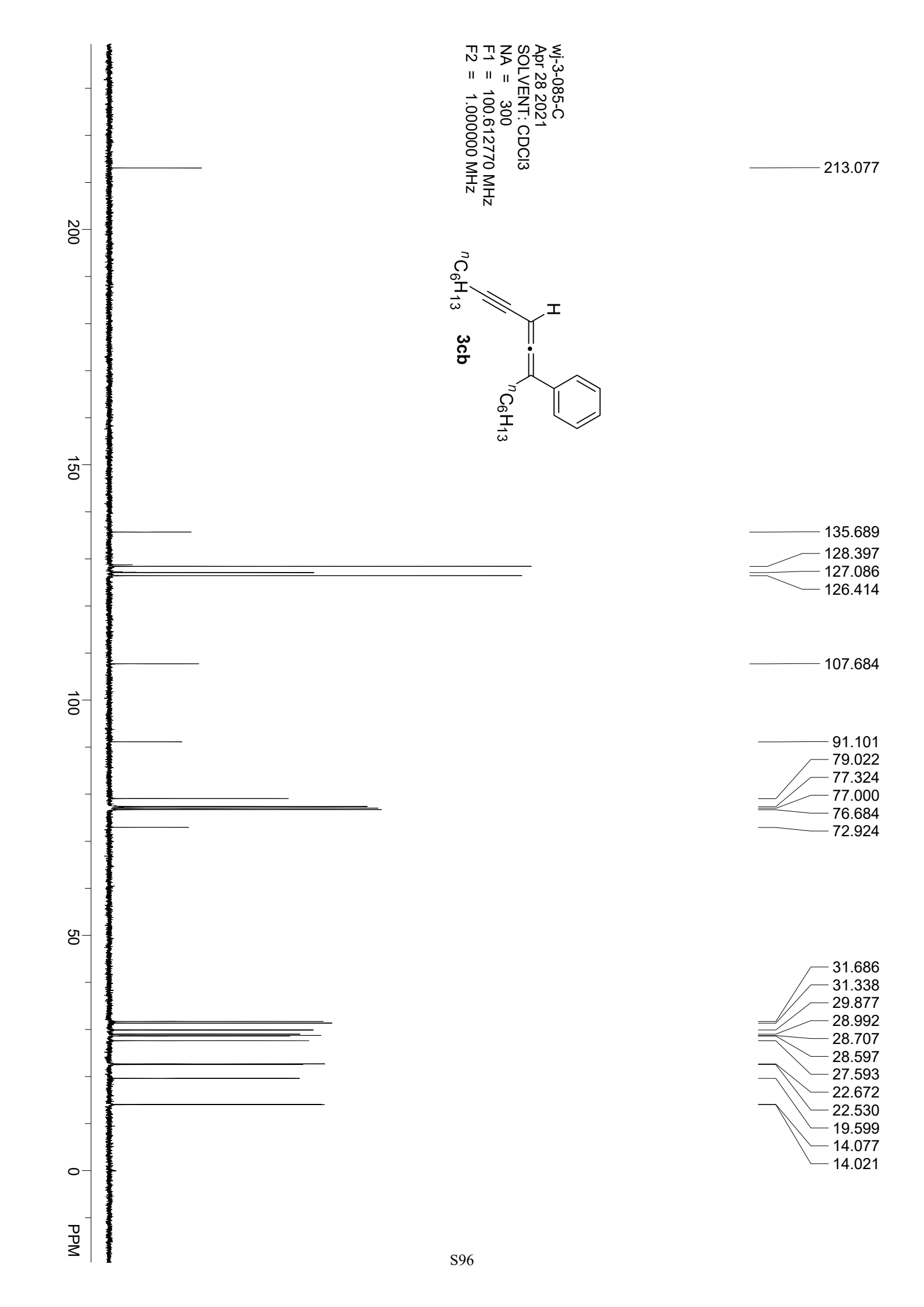


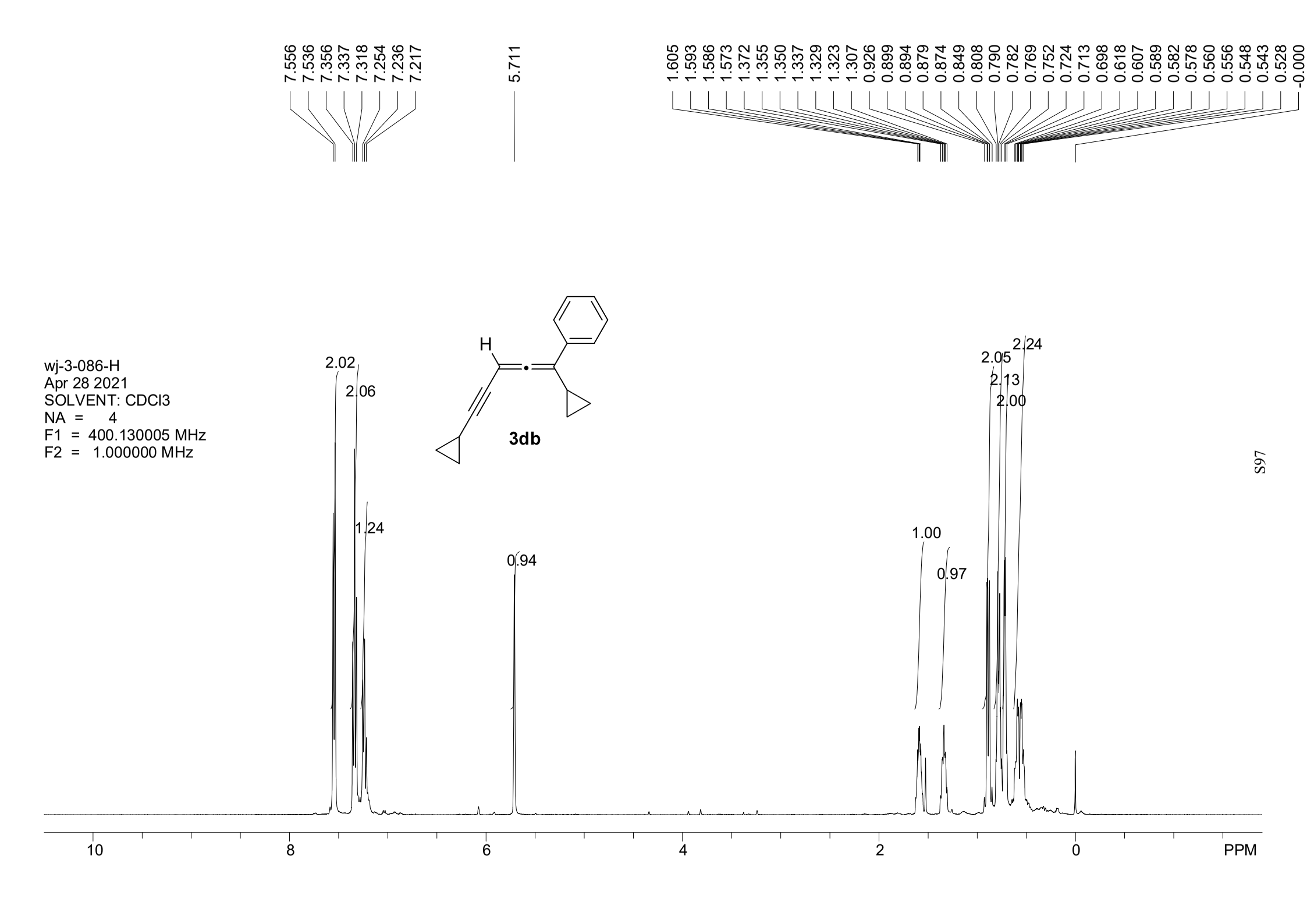

wj-3-036-C
Mar 24 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
3ar
214.262
138.936
126.541
105.566
90.177
77.577
77.316
77.000
76.684
73.366
33.921
33.376
30.856
29.900
29.466
23.035
22.474
21.969
19.251
13.934
13.603
200
150
100
50
0
PPM

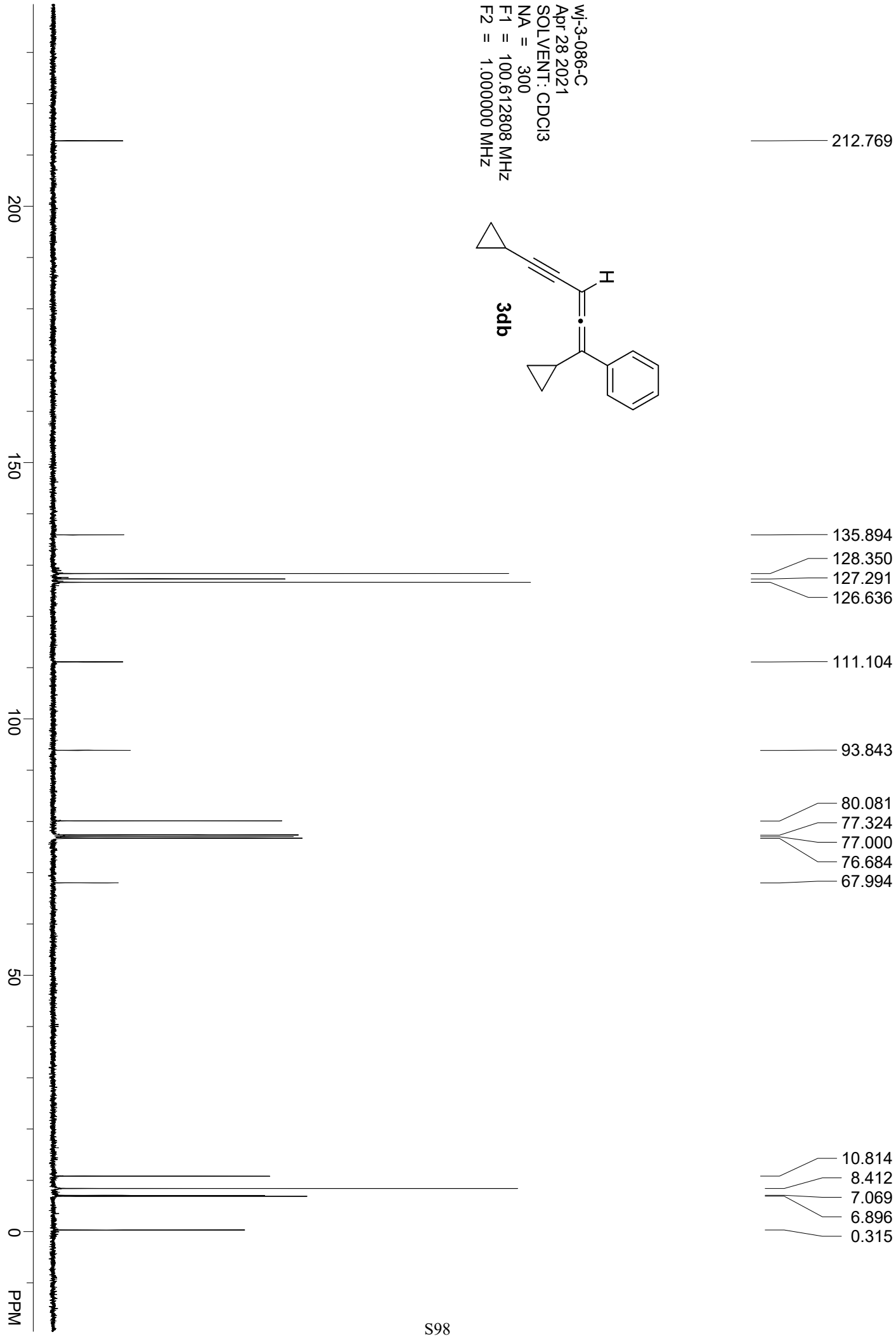

7.403
7.384
7.345
7.326
7.307
7.241
7.223
7.205
5.663
2.134
2.128
1.947
1.942
-0.000
wj-3-107-H
May 15 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
Me
Me
3bb
S93
2.08
2.01
1.12
0.93
3.00
2.98
10
8
6
4
2
PPM

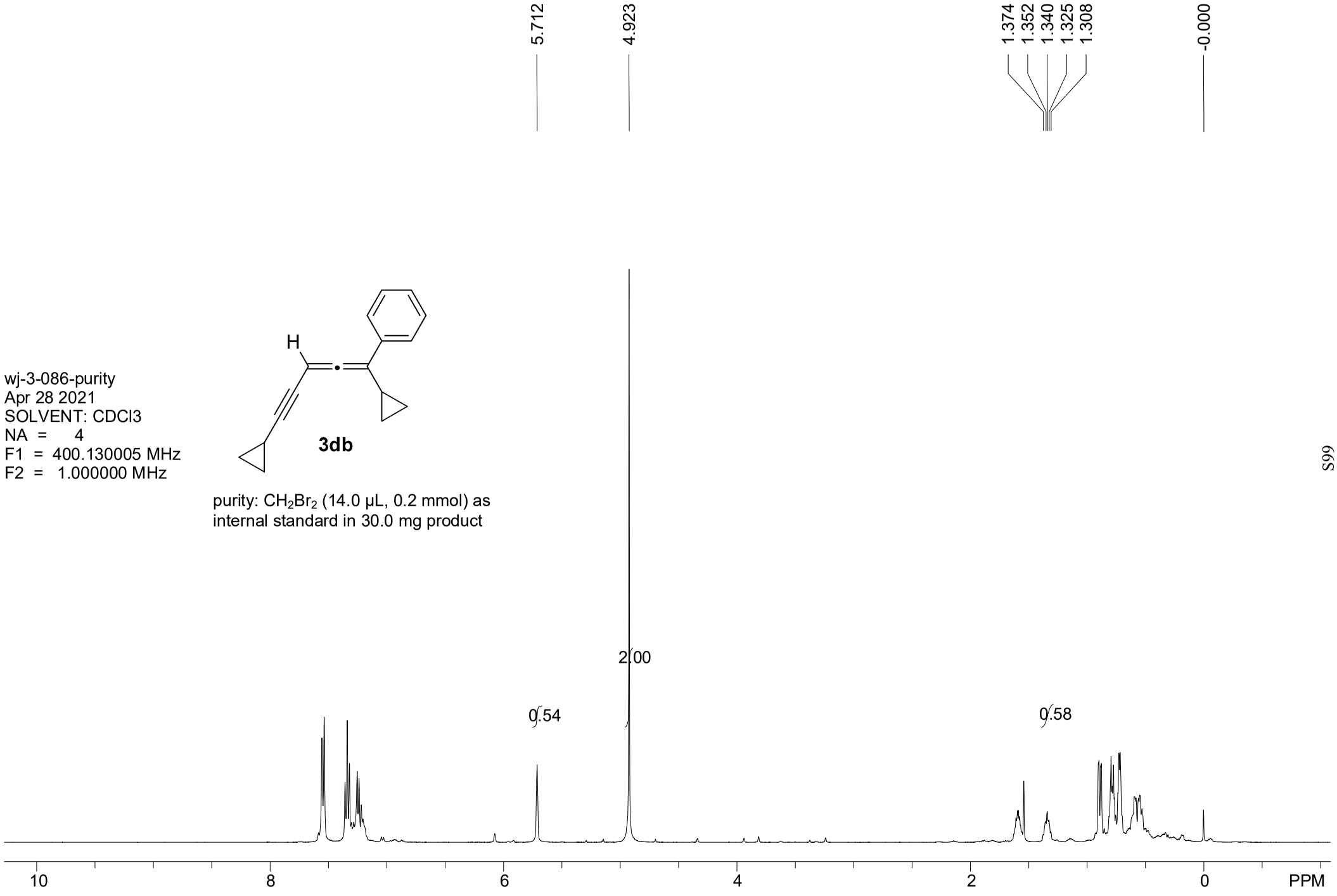

wj-3-107-C
May 15 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Me
H
3bb
Me
213.259
135.689
128.382
127.189
126.083
102.635
86.535
77.758
77.316
77.000
76.676
72.055
16.676
4.391
200
150
100
50
0
PPM

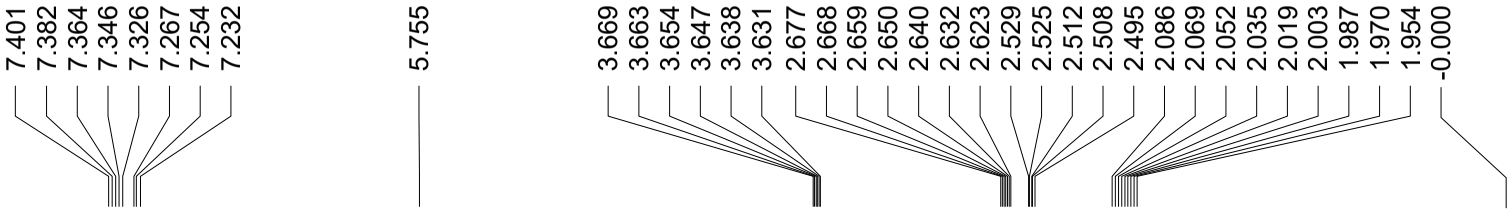

7.398
7.379
7.336
7.318
7.299
7.242
7.232
7.214
7.196
5.718
2.481
2.473
2.464
2.455
2.445
2.436
2.428
2.313
2.310
2.296
2.278
1.591
1.572
1.554
1.544
1.536
1.525
1.507
1.489
1.420
1.400
1.388
1.367
1.320
1.303
1.295
1.286
0.899
0.884
0.867
0.000
wj-3-085-H
Apr 28 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
nC6H13
nC6H13
3cb
12.32
6.11
4.21
2.01
2.09
1.13
0.96
2.00
2.02
10
8
6
4
2
0
PPM
S95


wj-3-085-C
Apr 28 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
$^{n}C_6H_{13}$
H
$^{n}C_6H_{13}$
3cb
213.077
135.689
128.397
127.086
126.414
107.684
91.101
79.022
77.324
77.000
76.684
72.924
31.686
31.338
29.877
28.992
28.707
28.597
27.593
22.672
22.530
19.599
14.077
14.021
200
150
100
50
0
PPM

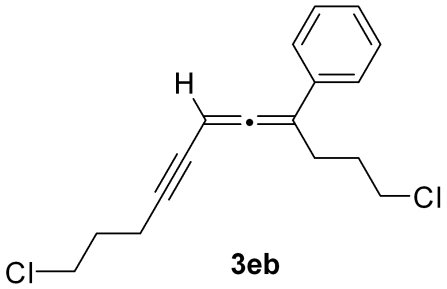

7.556
7.536
7.356
7.337
7.318
7.254
7.236
7.217
5.711
1.605
1.593
1.586
1.573
1.372
1.355
1.350
1.337
1.329
1.323
1.307
0.926
0.899
0.894
0.879
0.874
0.849
0.808
0.790
0.782
0.769
0.752
0.724
0.713
0.698
0.618
0.607
0.589
0.582
0.578
0.560
0.556
0.548
0.543
0.528
-0.000
wj-3-086-H
Apr 28 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
3db
2.02
2.06
1.24
0.94
1.00
0.97
2.05
2.13
2.00
2.24
S97
10
8
6
4
2
0
PPM


wj-3-086-C
Apr 28 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612808 MHz
F2 = 1.000000 MHz
3db
H
212.769
135.894
128.350
127.291
126.636
111.104
93.843
80.081
77.324
77.000
76.684
67.994
10.814
8.412
7.069
6.896
0.315
200
150
100
50
0
PPM


5.712
4.923
1.374
1.352
1.340
1.325
1.308
-0.000
wj-3-086-purity
Apr 28 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
3db
purity: CH2Br2 (14.0 µL, 0.2 mmol) as
internal standard in 30.0 mg product
2.00
0.54
0.58
10
8
6
4
2
0
PPM

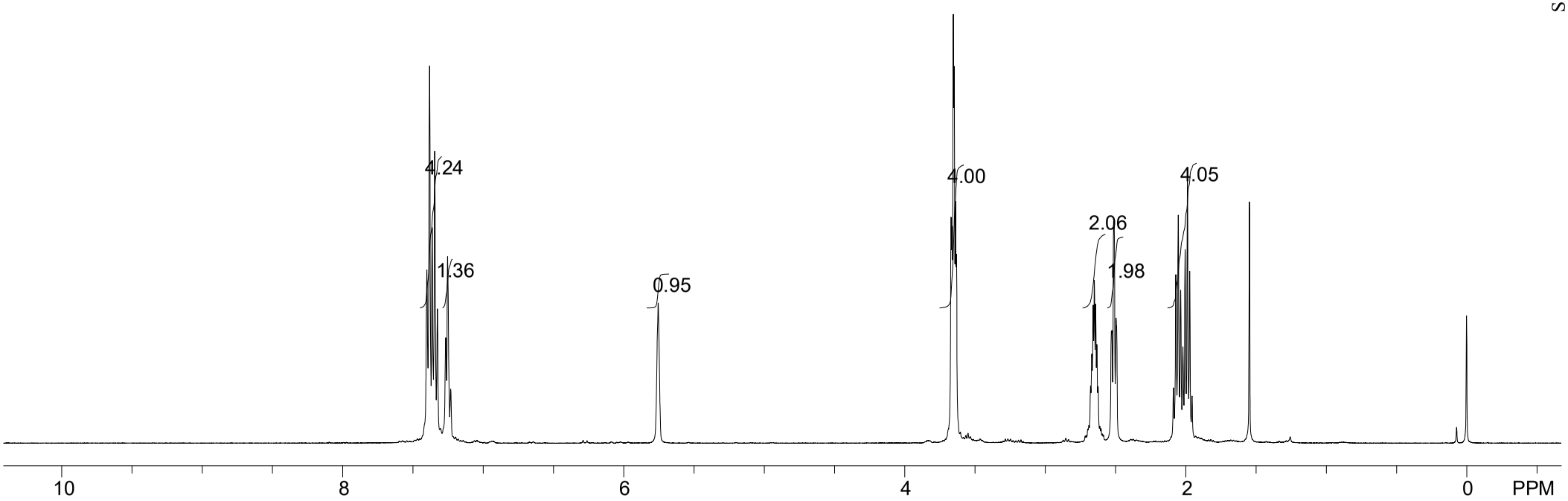


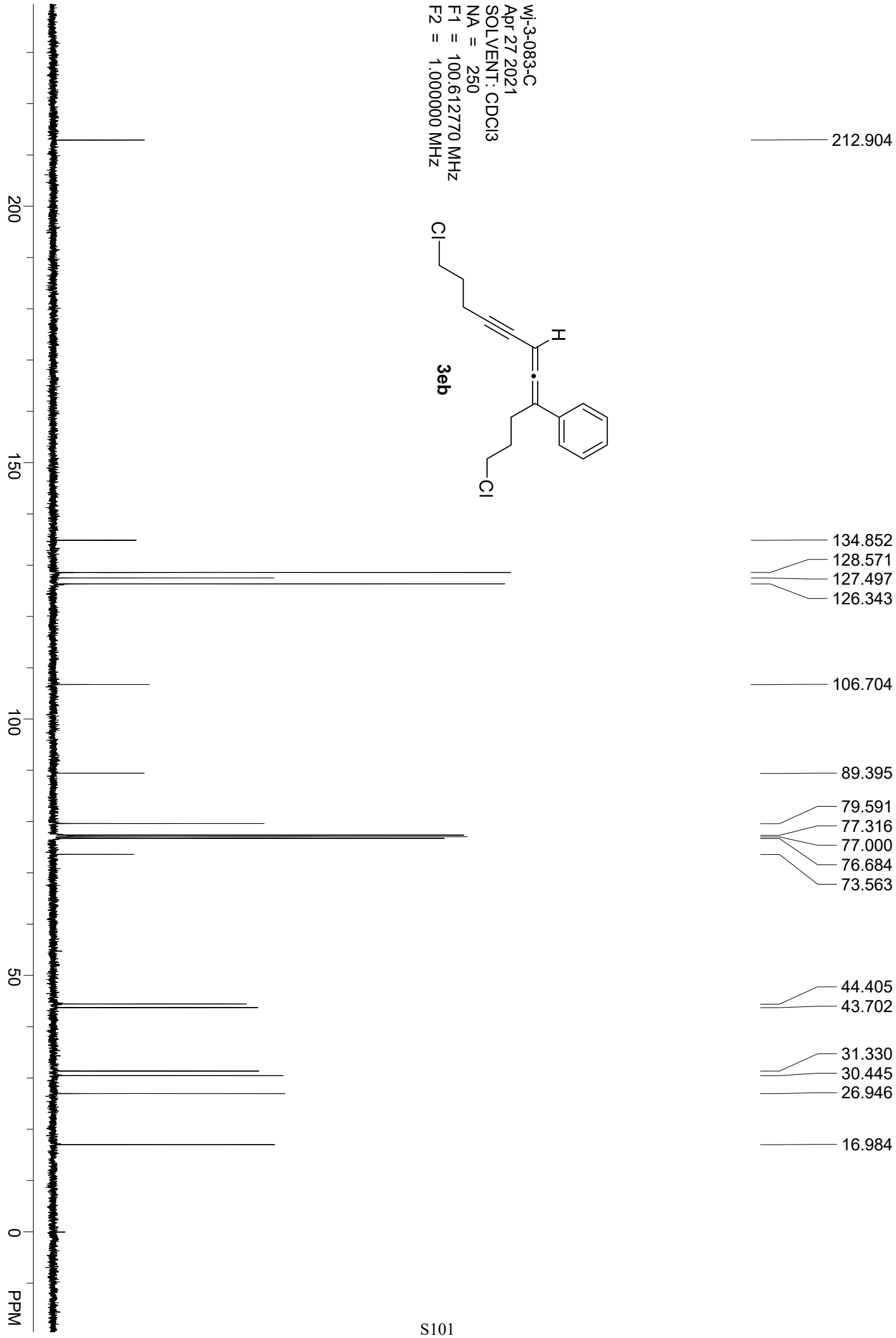
wj-3-083-H
Apr 27 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz

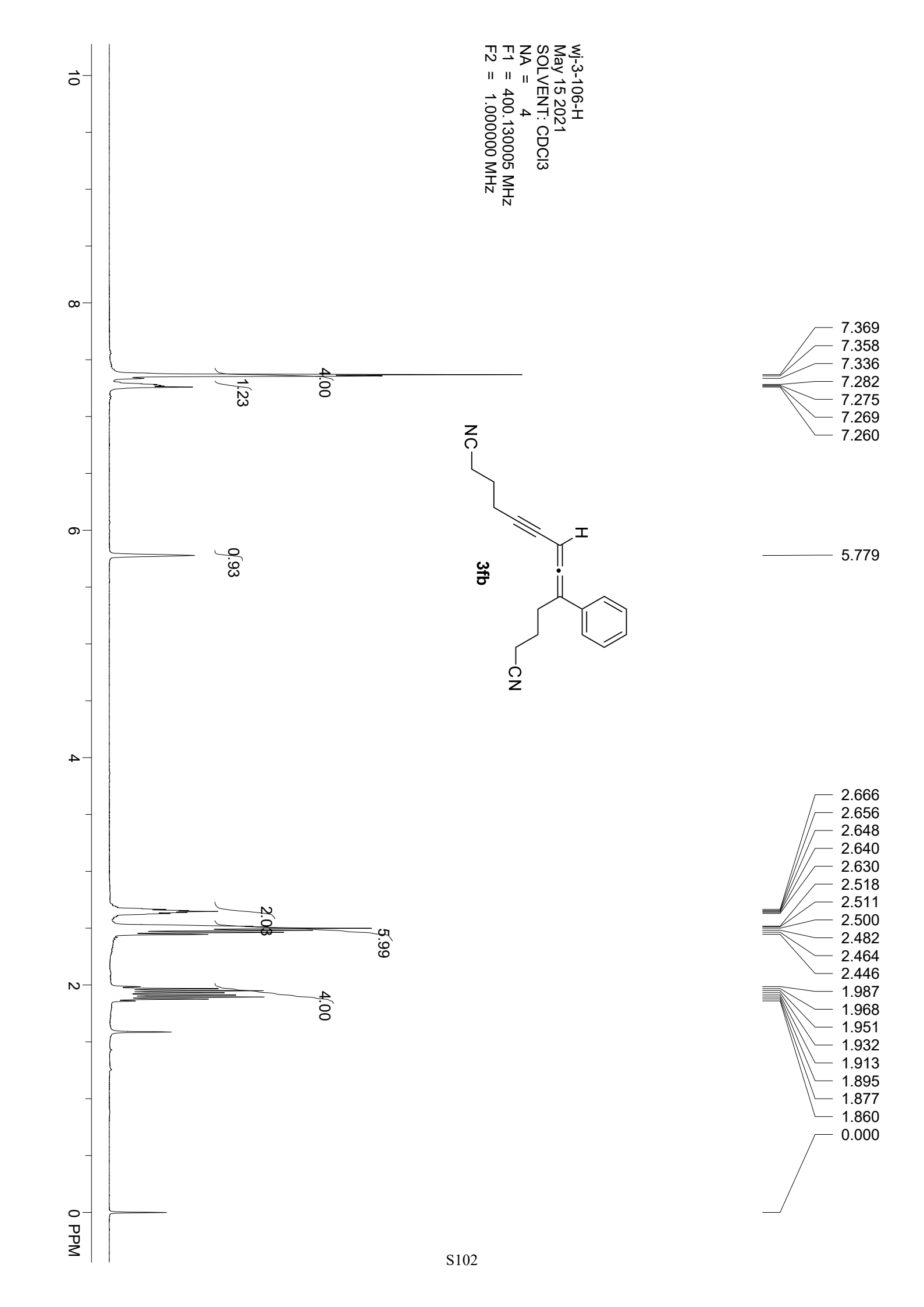


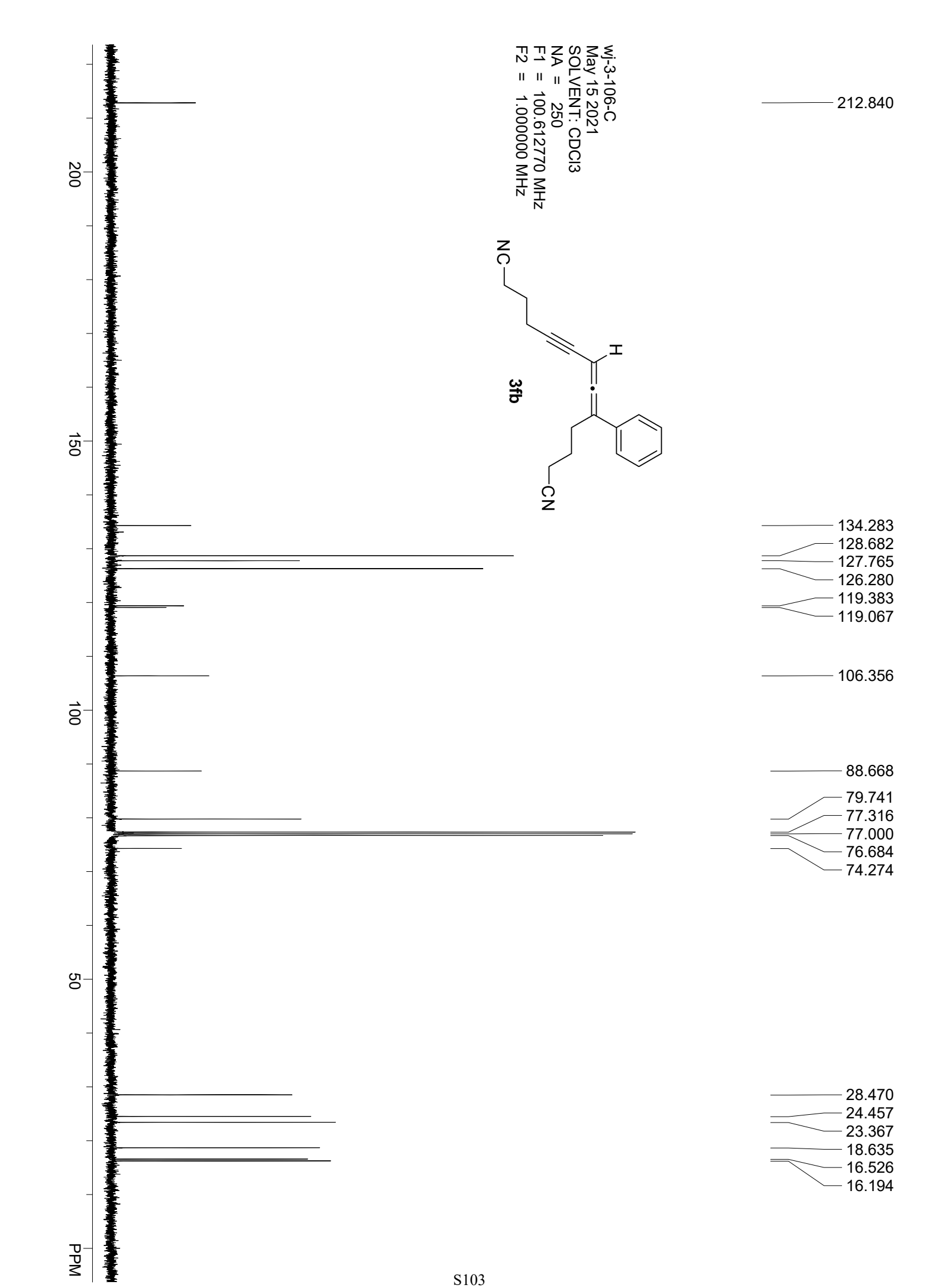
3eb

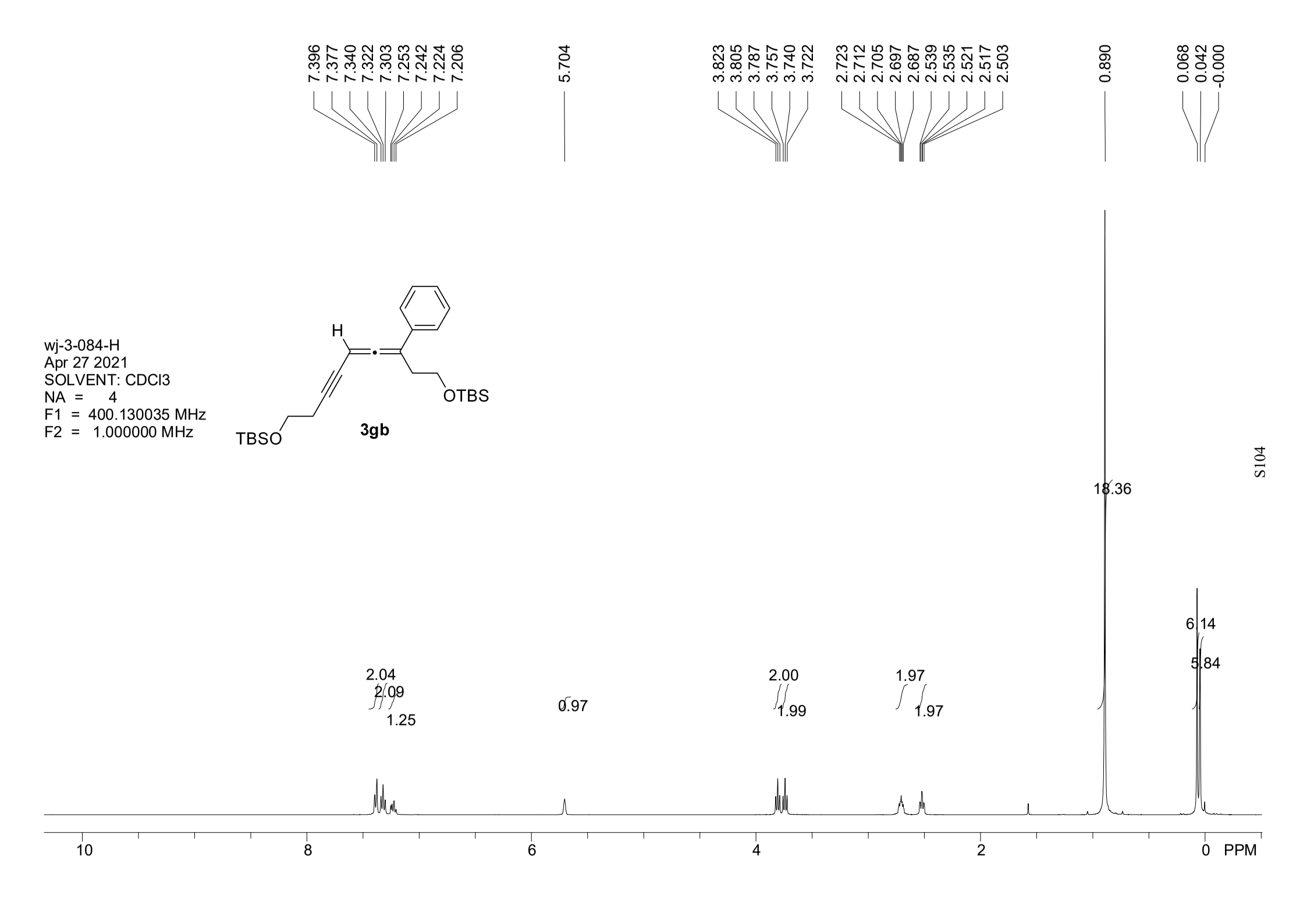
S100

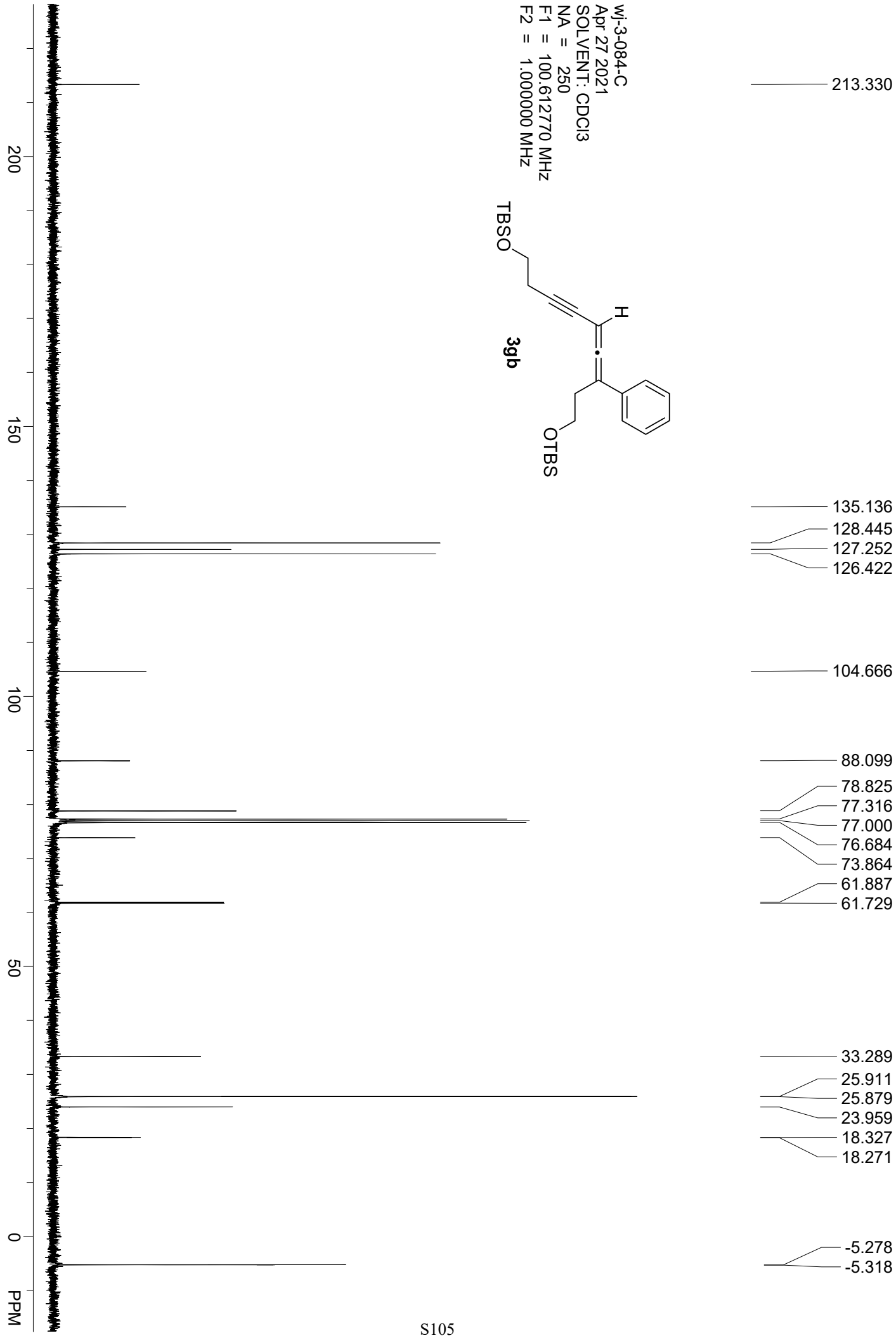


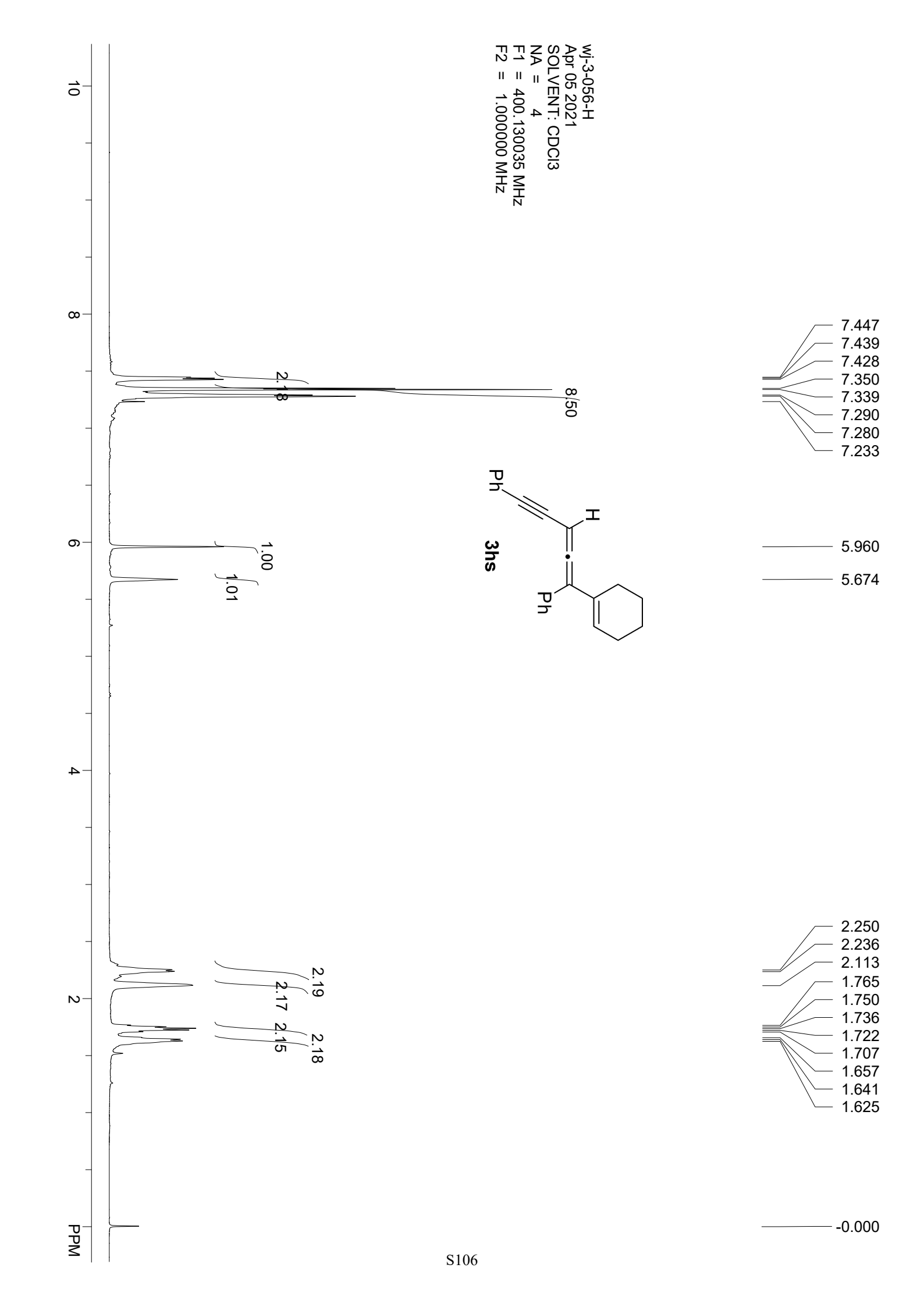

wj-3-083-C
Apr 27 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Cl
H
3eb
Cl
212.904
134.852
128.571
127.497
126.343
106.704
89.395
79.591
77.316
77.000
76.684
73.563
44.405
43.702
31.330
30.445
26.946
16.984
200
150
100
50
0
PPM

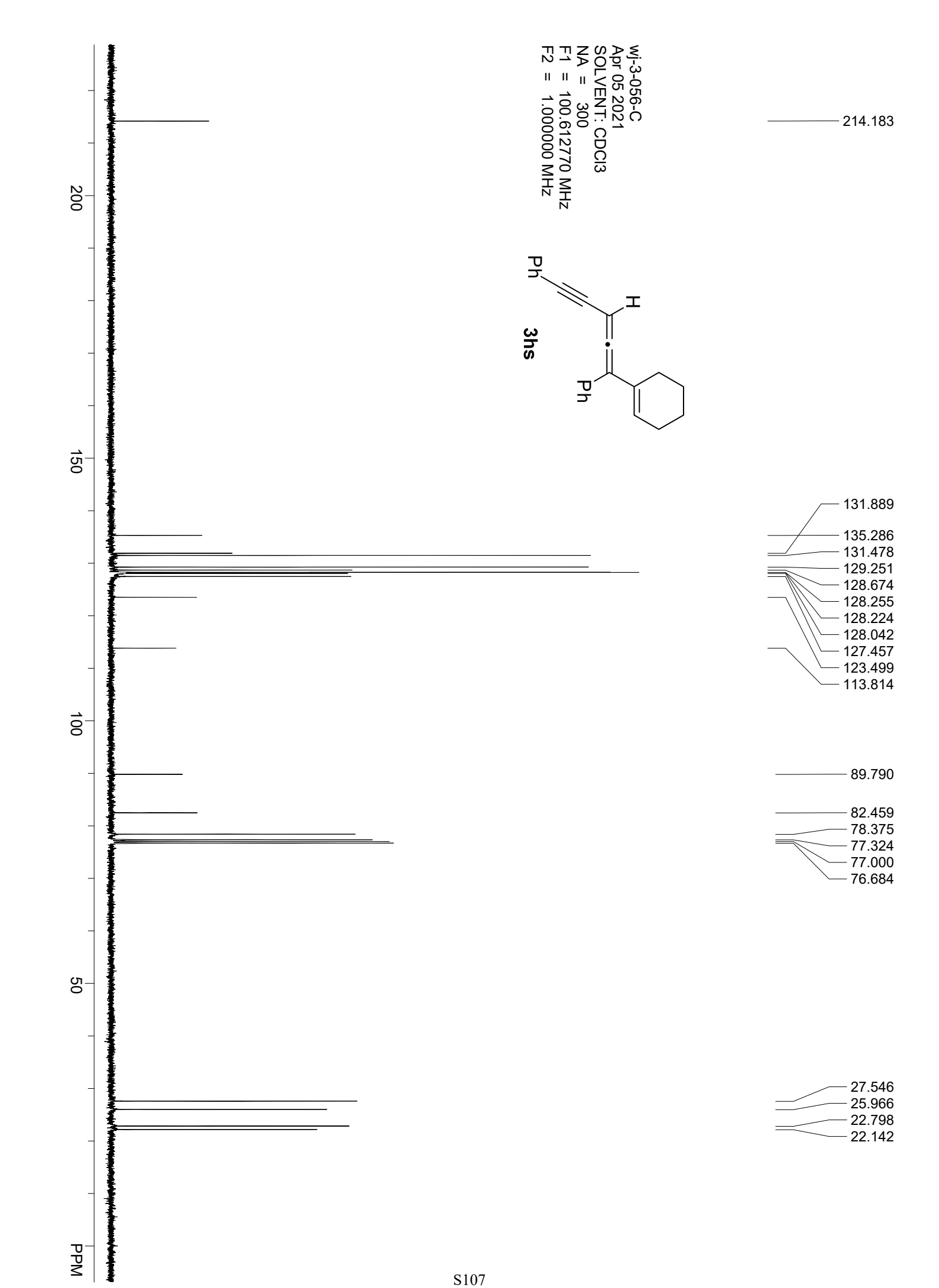

wj-3-106-H
May 15 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
NC
H
CN
3fb
7.369
7.358
7.336
7.282
7.275
7.269
7.260
5.779
2.666
2.656
2.648
2.640
2.630
2.518
2.511
2.500
2.482
2.464
2.446
1.987
1.968
1.951
1.932
1.913
1.895
1.877
1.860
0.000
4.00
1.23
0.93
2.03
5.99
4.00
10
8
6
4
2
0 PPM

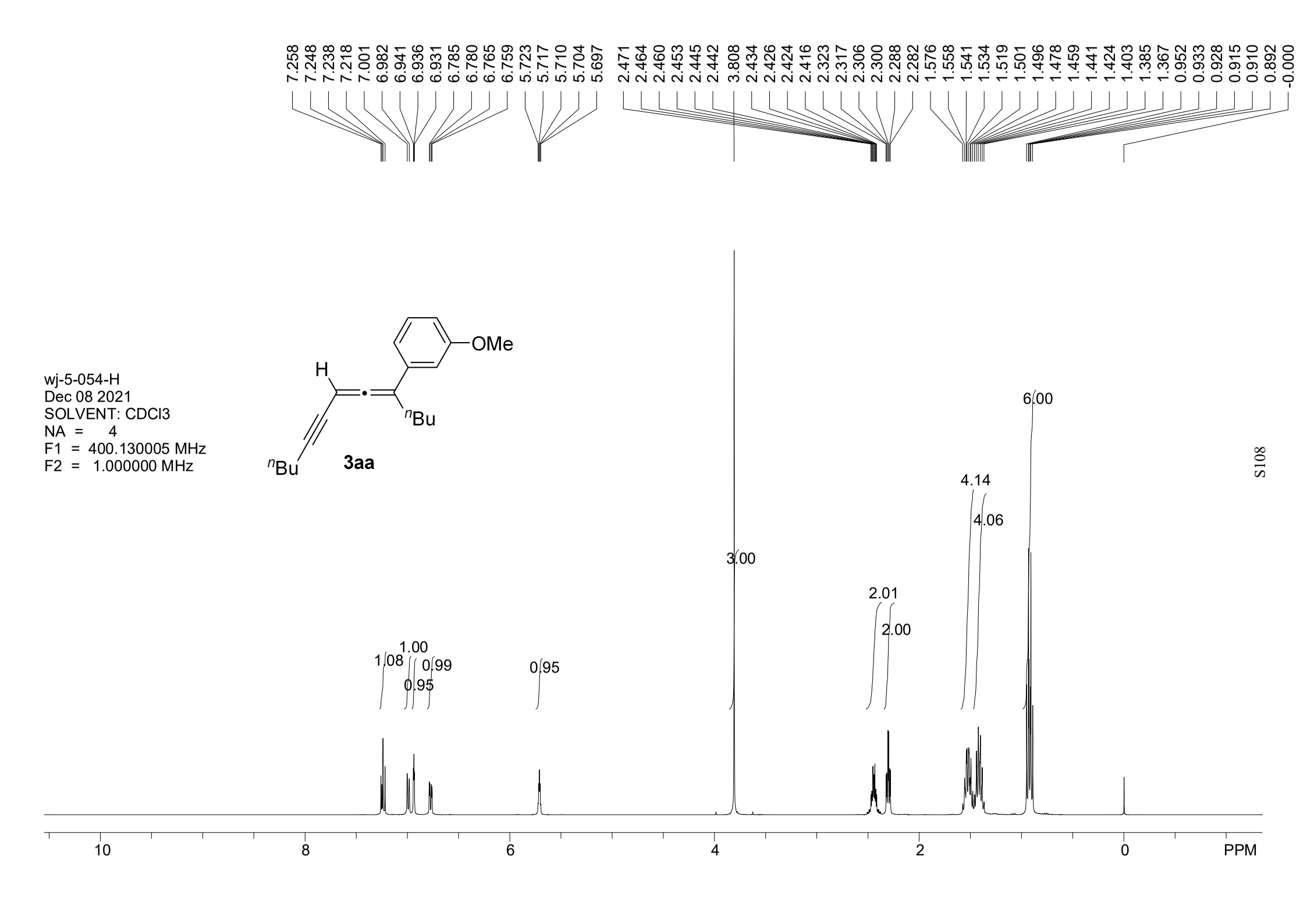

wj-3-106-C
May 15 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
NC
H
CN
3fb
212.840
134.283
128.682
127.765
126.280
119.383
119.067
106.356
88.668
79.741
77.316
77.000
76.684
74.274
28.470
24.457
23.367
18.635
16.526
16.194
200
150
100
50
PPM

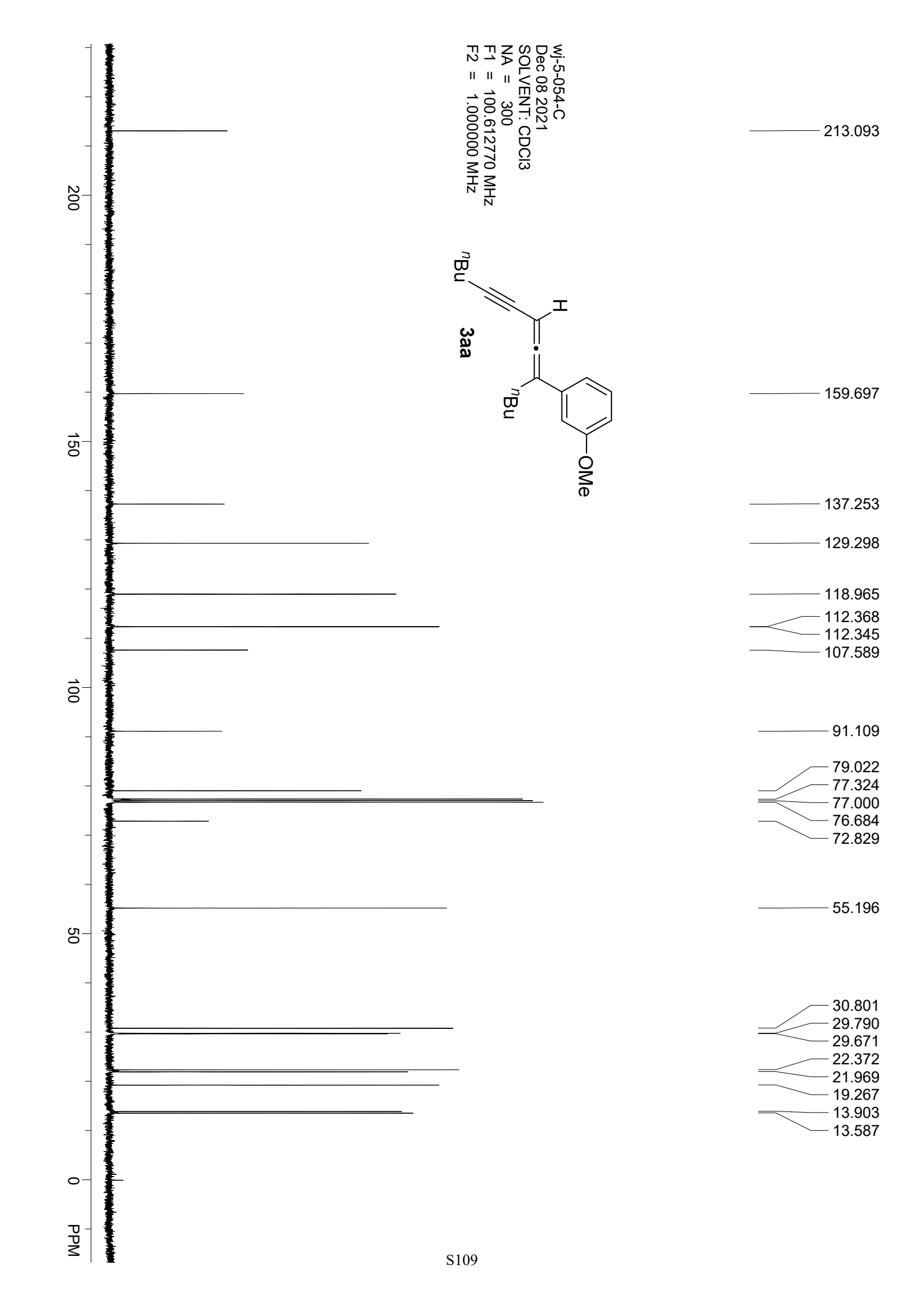

7.396
7.377
7.340
7.322
7.303
7.253
7.242
7.224
7.206
5.704
3.823
3.805
3.787
3.757
3.740
3.722
2.723
2.712
2.705
2.697
2.687
2.539
2.535
2.521
2.517
2.503
0.890
0.068
0.042
-0.000
wj-3-084-H
Apr 27 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
H
OTBS
TBSO
3gb
S104
18.36
6.14
5.84
2.04
2.09
1.25
0.97
2.00
1.99
1.97
1.97
10
8
6
4
2
0
PPM

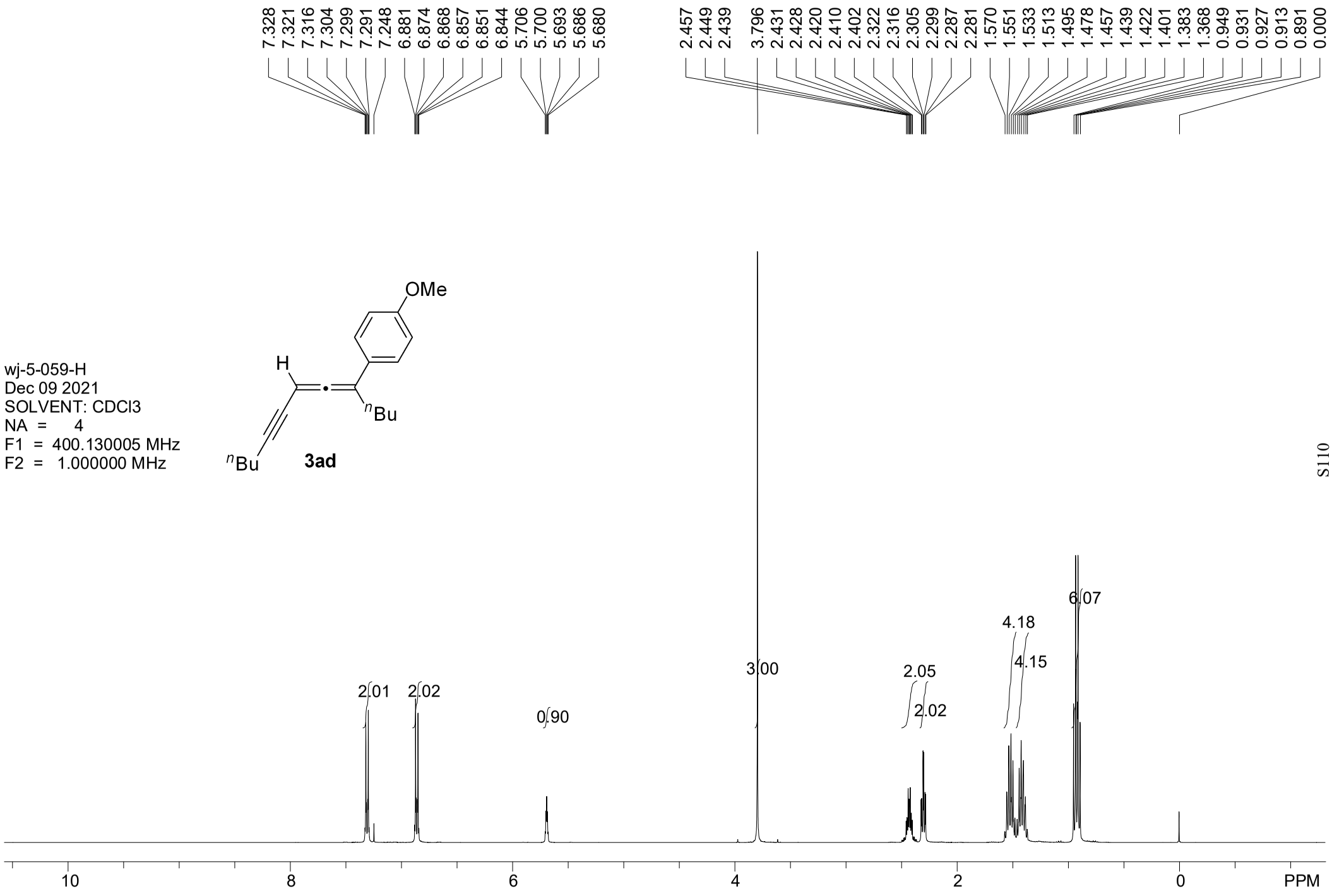

wj-3-084-C
Apr 27 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
TBSO
H
OTBS
3gb
213.330
135.136
128.445
127.252
126.422
104.666
88.099
78.825
77.316
77.000
76.684
73.864
61.887
61.729
33.289
25.911
25.879
23.959
18.327
18.271
-5.278
-5.318
200
150
100
50
0
PPM

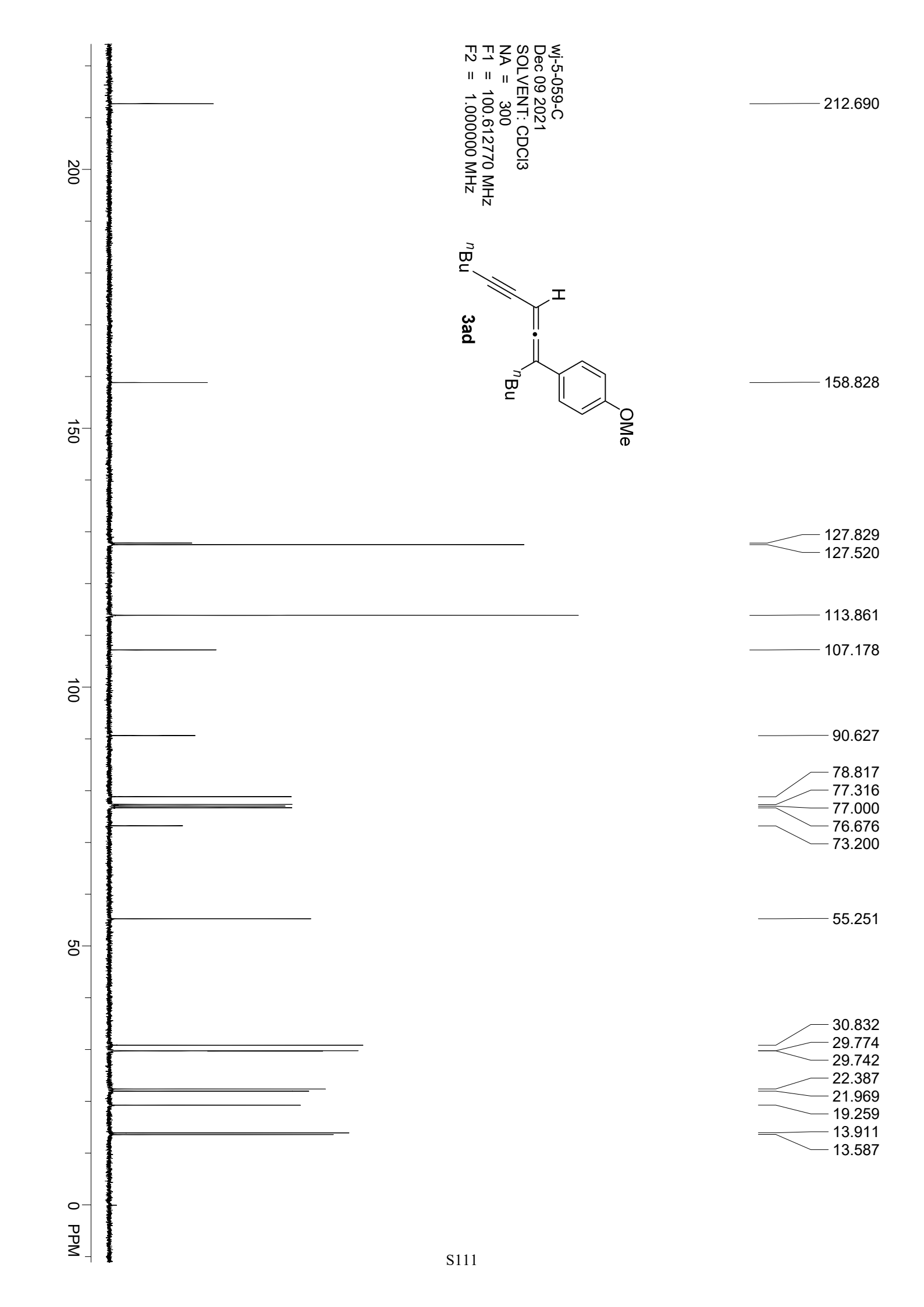

wj-3-056-H
Apr 05 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
7.447
7.439
7.428
7.350
7.339
7.290
7.280
7.233
5.960
5.674
2.250
2.236
2.113
1.765
1.750
1.736
1.722
1.707
1.657
1.641
1.625
-0.000
8.50
2.18
1.00
1.01
2.19
2.17
2.15
2.18
Ph
H
Ph
3hs
10
8
6
4
2
PPM

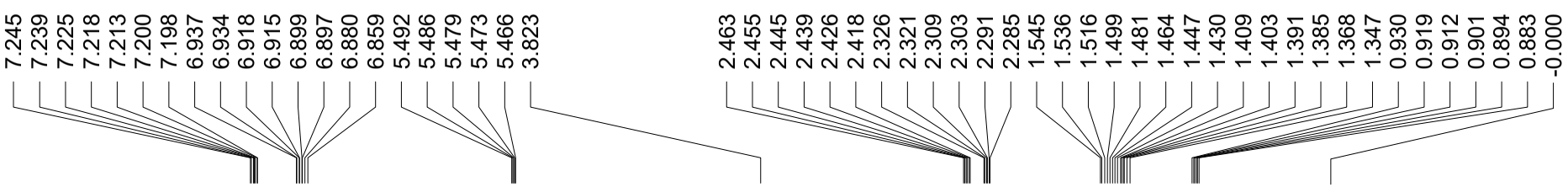

wj-3-056-C
Apr 05 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
H
Ph
3hs
214.183
131.889
135.286
131.478
129.251
128.674
128.255
128.224
128.042
127.457
123.499
113.814
89.790
82.459
78.375
77.324
77.000
76.684
27.546
25.966
22.798
22.142
200
150
100
50
PPM


7.258
7.248
7.238
7.218
7.001
6.982
6.941
6.936
6.931
6.785
6.780
6.765
6.759
5.723
5.717
5.710
5.704
5.697
2.471
2.464
2.460
2.453
2.445
2.442
3.808
2.434
2.426
2.424
2.416
2.323
2.317
2.306
2.300
2.288
2.282
1.576
1.558
1.541
1.534
1.519
1.501
1.496
1.478
1.459
1.441
1.424
1.403
1.385
1.367
0.952
0.933
0.928
0.915
0.910
0.892
-0.000
wj-5-054-H
Dec 08 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
OMe
H
nBu
nBu
3aa
S108
1.08
1.00
0.99
0.95
0.95
3.00
2.01
2.00
4.14
4.06
6.00
10
8
6
4
2
0
PPM

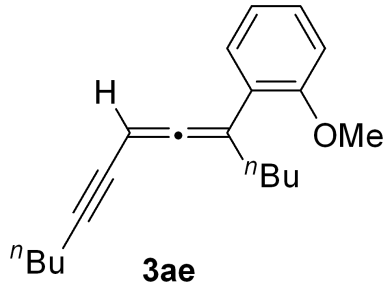

wj-5-054-C
Dec 08 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3aa
nBu
OMe
213.093
159.697
137.253
129.298
118.965
112.368
112.345
107.589
91.109
79.022
77.324
77.000
76.684
72.829
55.196
30.801
29.790
29.671
22.372
21.969
19.267
13.903
13.587
200
150
100
50
0
PPM


7.328
7.321
7.316
7.304
7.299
7.291
7.248
6.881
6.874
6.868
6.857
6.851
6.844
5.706
5.700
5.693
5.686
5.680
2.457
2.449
2.439
3.796
2.431
2.428
2.420
2.410
2.402
2.322
2.316
2.305
2.299
2.287
2.281
1.570
1.551
1.533
1.513
1.495
1.478
1.457
1.439
1.422
1.401
1.383
1.368
0.949
0.931
0.927
0.913
0.891
0.000
wj-5-059-H
Dec 09 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
OMe
H
nBu
nBu
3ad
2.01
2.02
0.90
3.00
2.05
2.02
4.18
4.15
6.07
10
8
6
4
2
0
PPM
S110

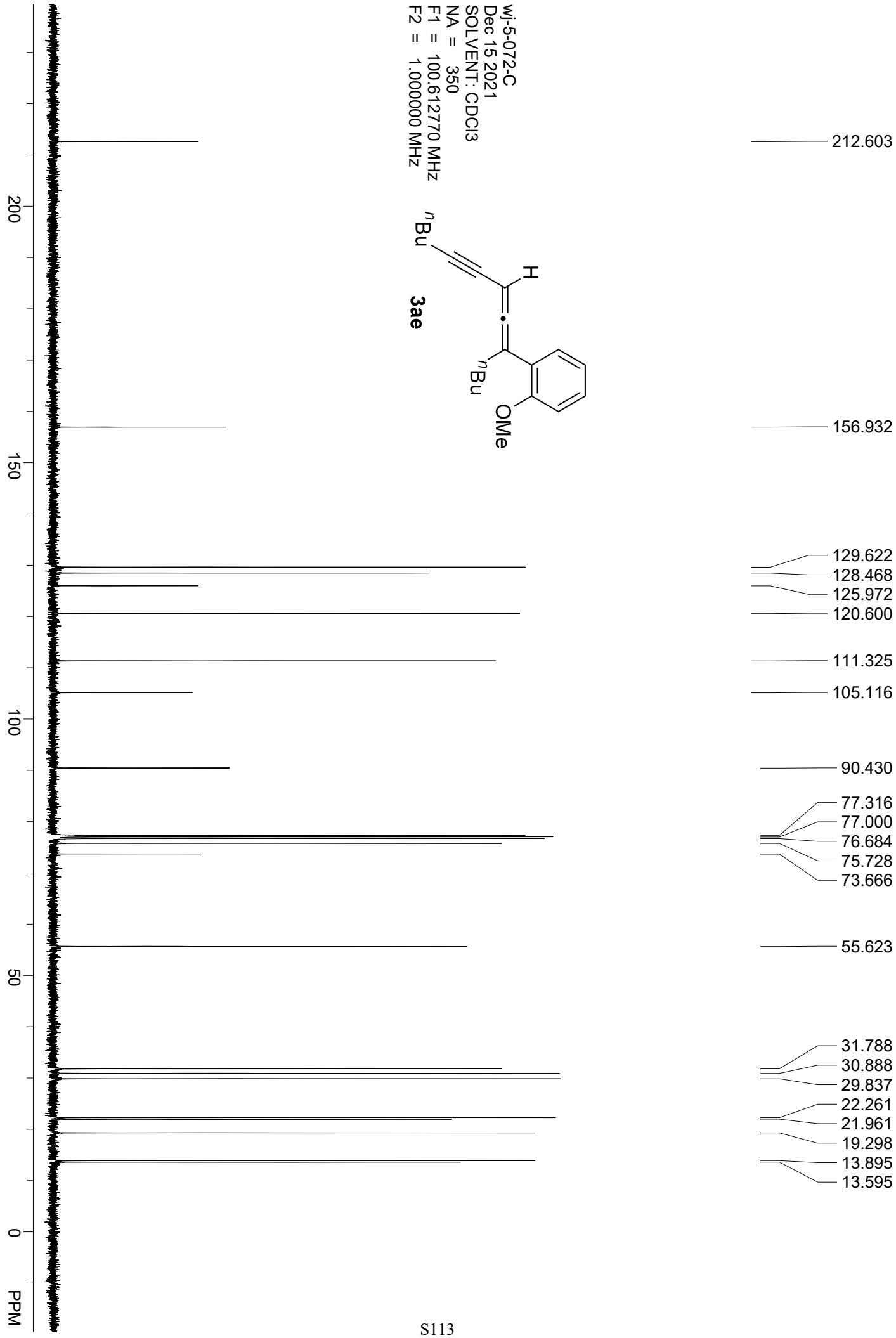

wj-5-059-C
Dec 09 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
OMe
3ad
212.690
158.828
127.829
127.520
113.861
107.178
90.627
78.817
77.316
77.000
76.676
73.200
55.251
30.832
29.774
29.742
22.387
21.969
19.259
13.911
13.587
200
150
100
50
0
PPM

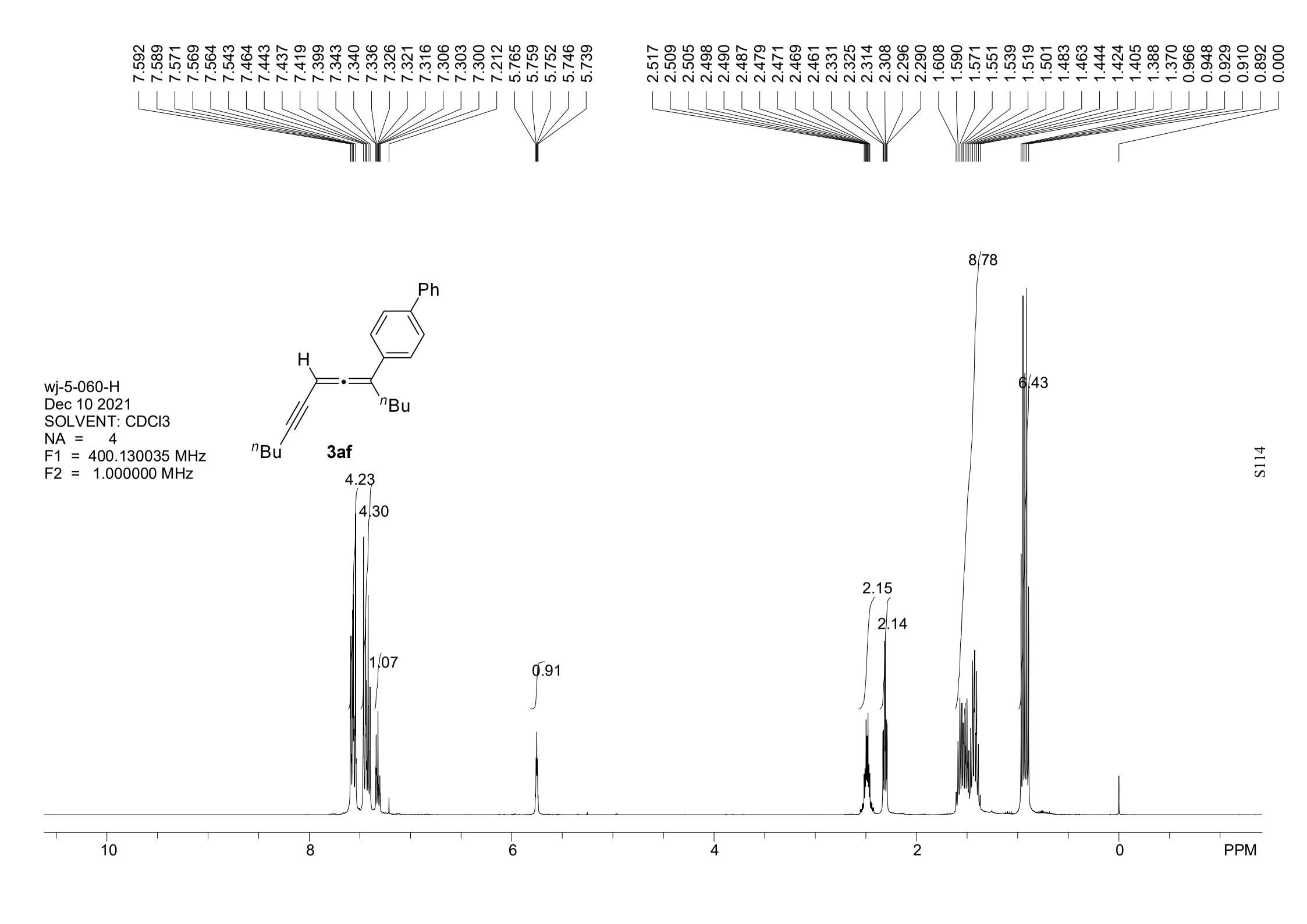


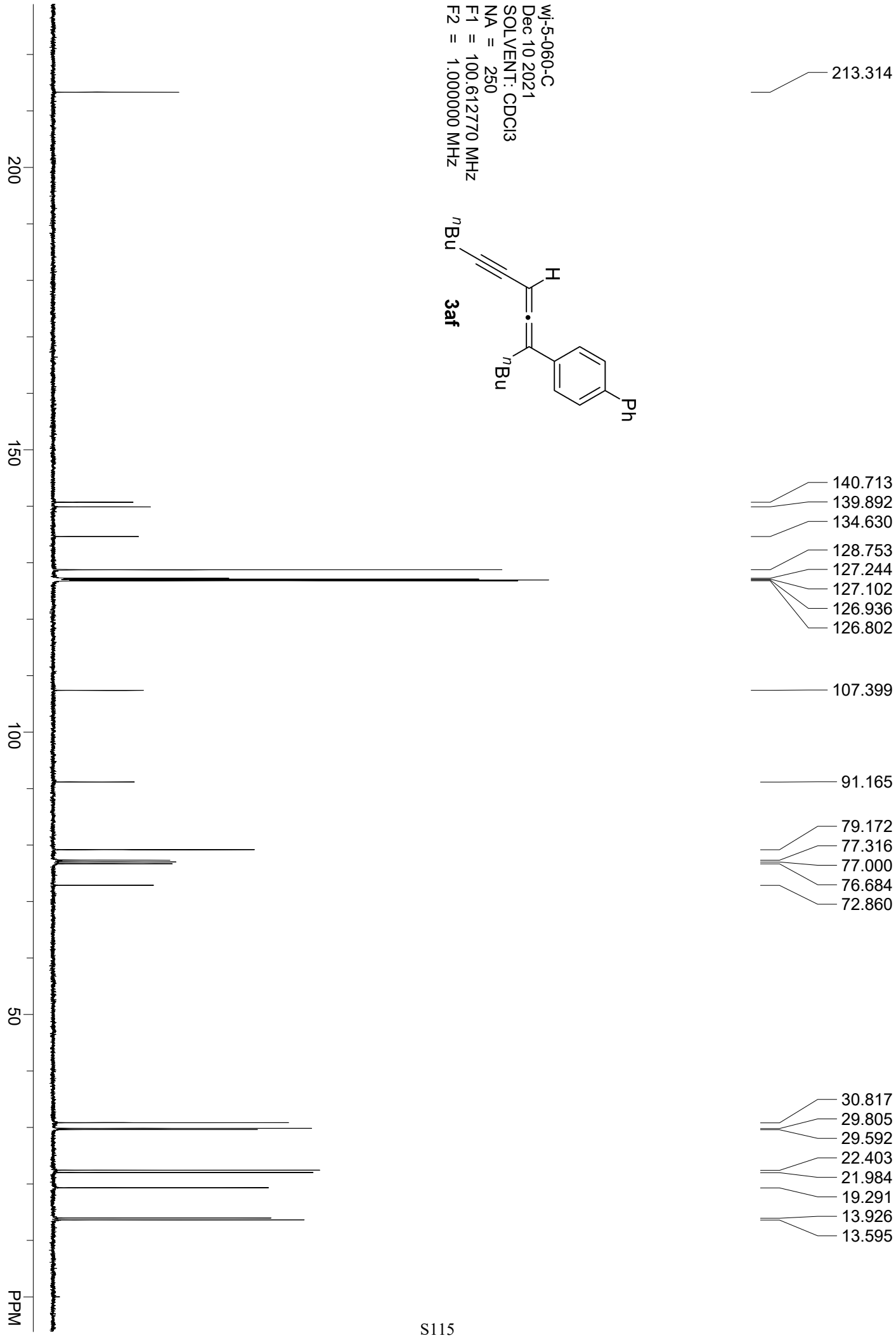
wj-5-072-H
Dec 15 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz

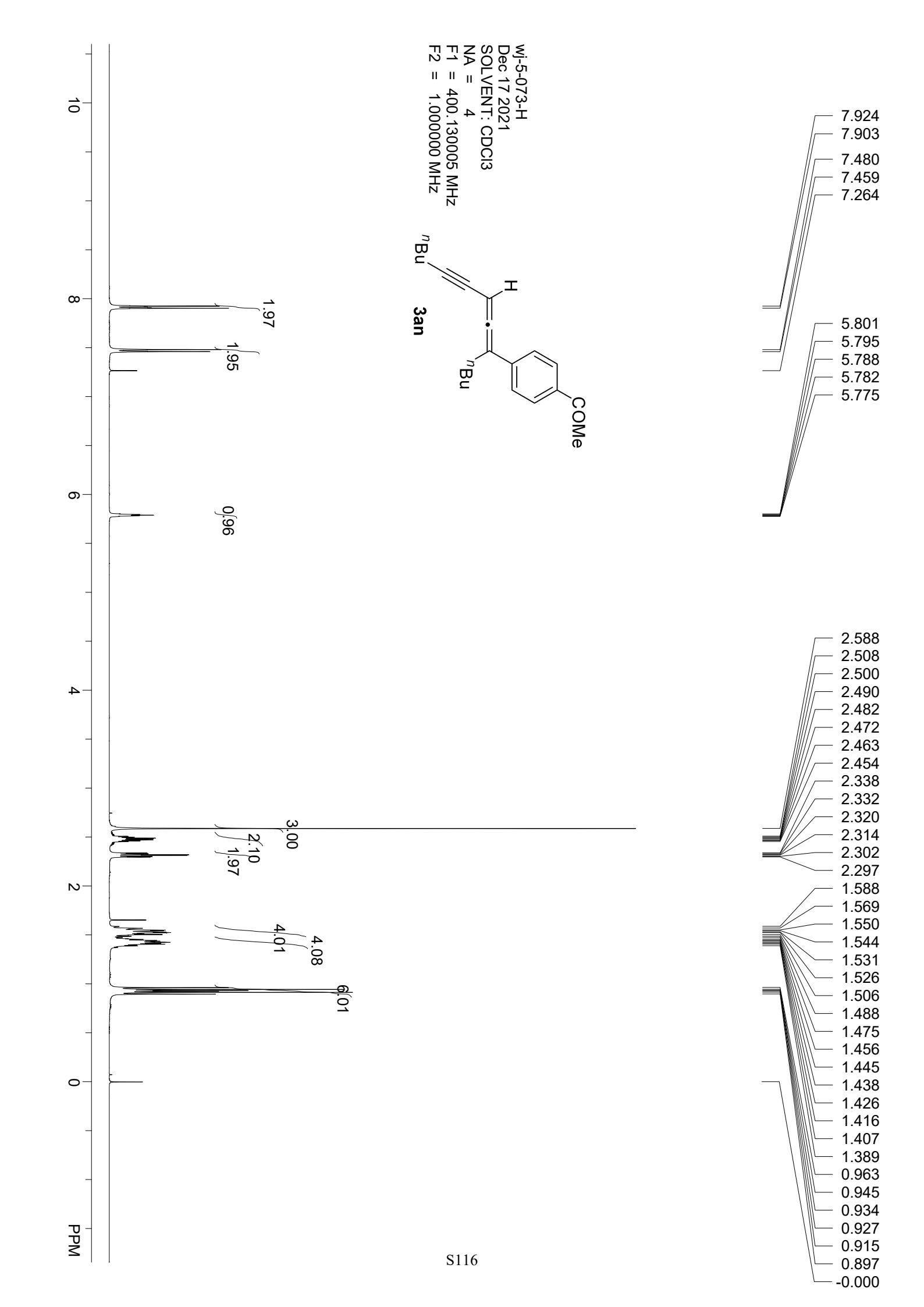


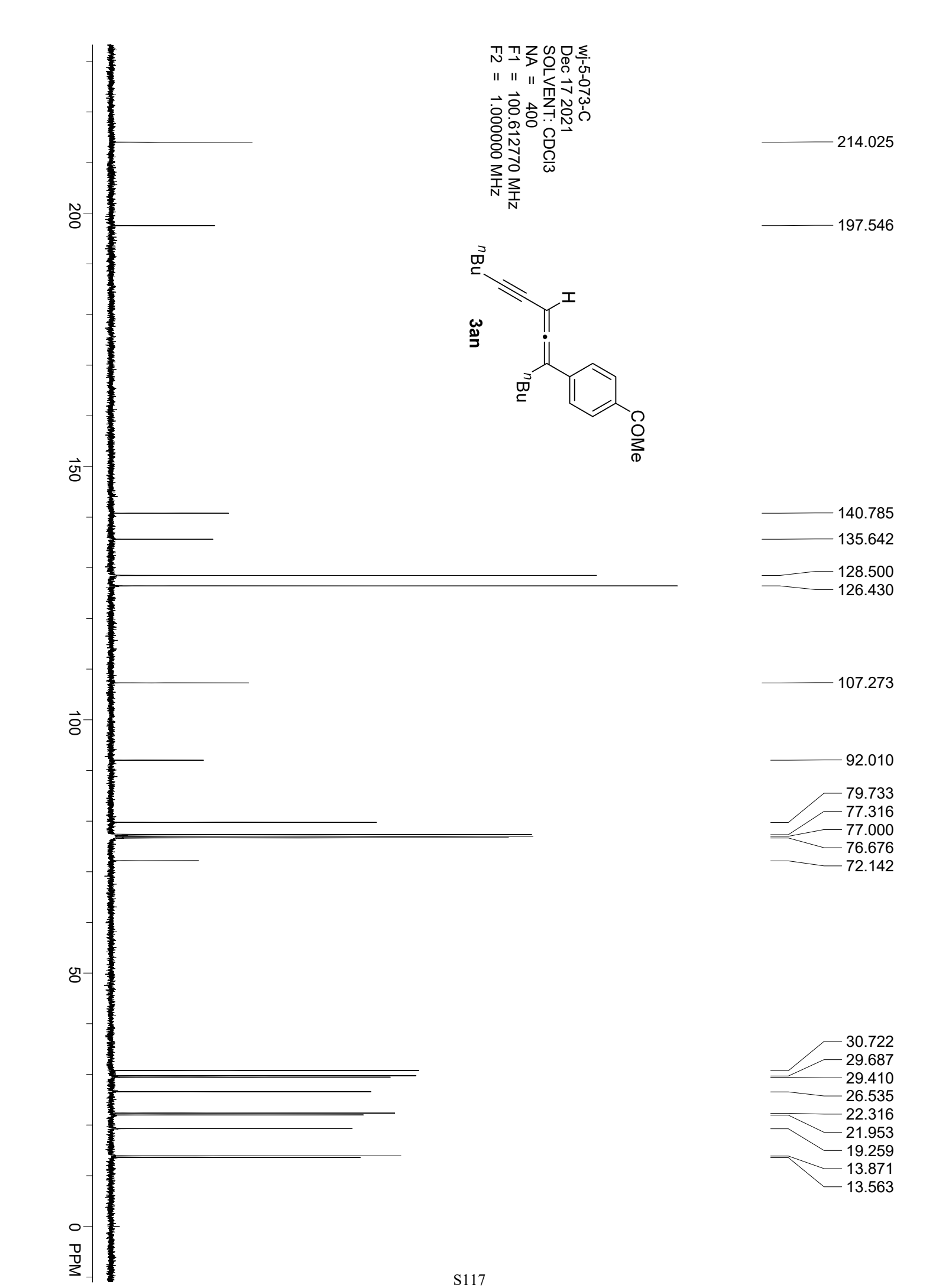
3ae

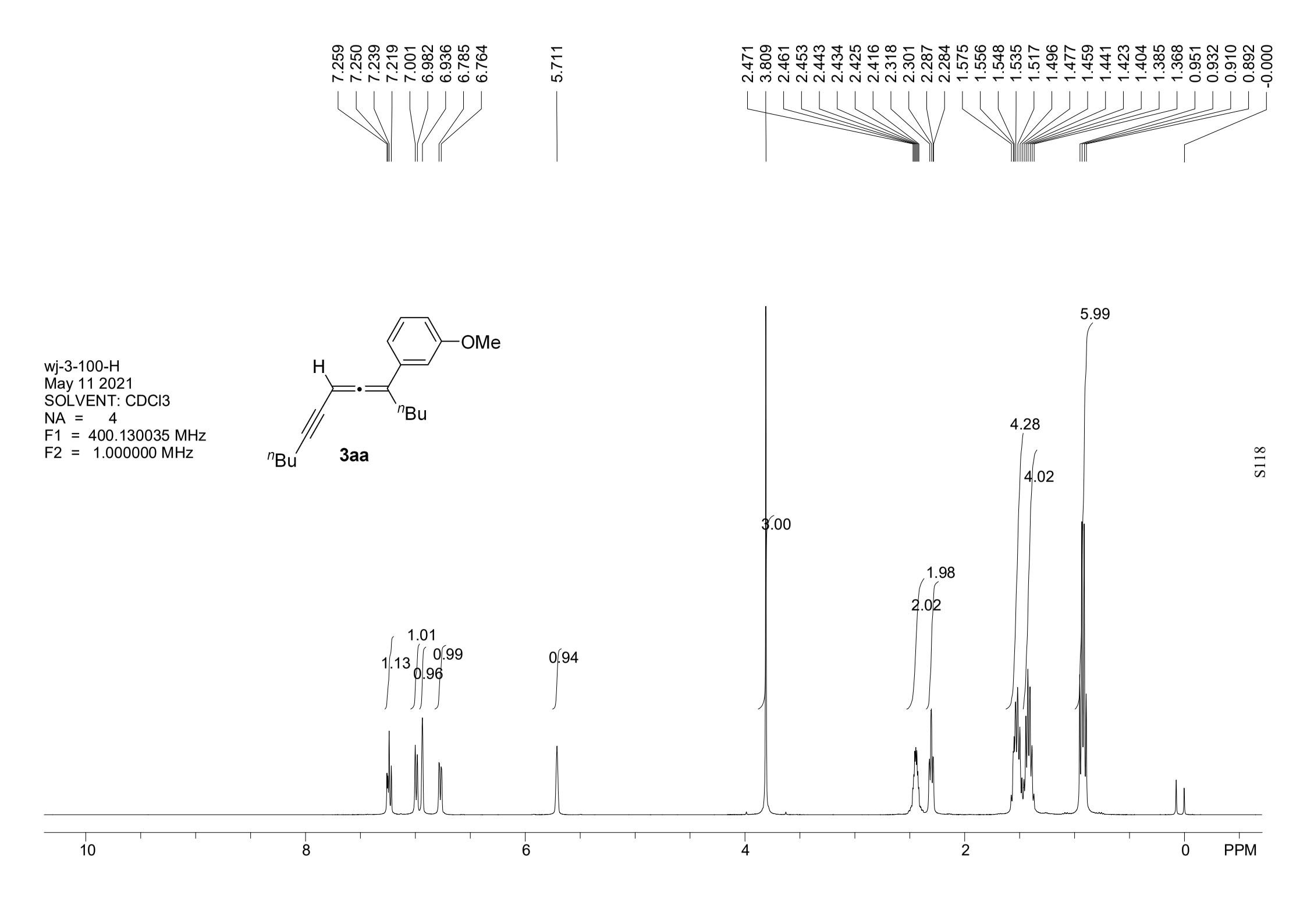

wj-5-072-C
Dec 15 2021
SOLVENT: CDCl3
NA = 350
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
OMe
3ae
212.603
156.932
129.622
128.468
125.972
120.600
111.325
105.116
90.430
77.316
77.000
76.684
75.728
73.666
55.623
31.788
30.888
29.837
22.261
21.961
19.298
13.895
13.595
200
150
100
50
0
PPM

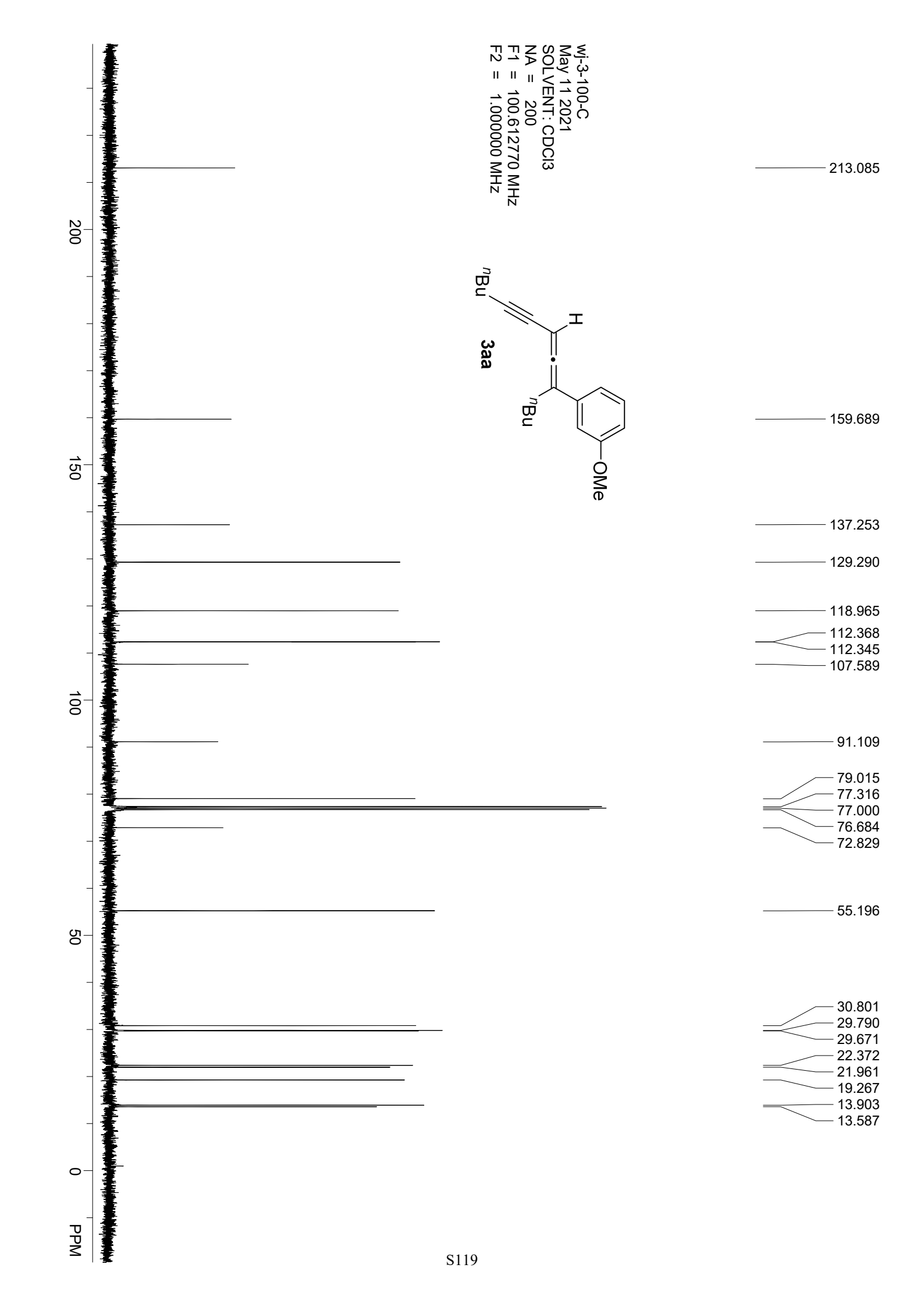

7.592
7.589
7.571
7.569
7.564
7.543
7.464
7.443
7.437
7.419
7.399
7.343
7.340
7.336
7.326
7.321
7.316
7.306
7.303
7.300
7.212
5.765
5.759
5.752
5.746
5.739
2.517
2.509
2.505
2.498
2.490
2.487
2.479
2.471
2.469
2.461
2.331
2.325
2.314
2.308
2.296
2.290
1.608
1.590
1.571
1.551
1.539
1.519
1.501
1.483
1.463
1.444
1.424
1.405
1.388
1.370
0.966
0.948
0.929
0.910
0.892
0.000
wj-5-060-H
Dec 10 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
Ph
H
nBu
nBu
3af
4.23
4.30
1.07
0.91
2.15
2.14
8.78
6.43
S114
10
8
6
4
2
0
PPM

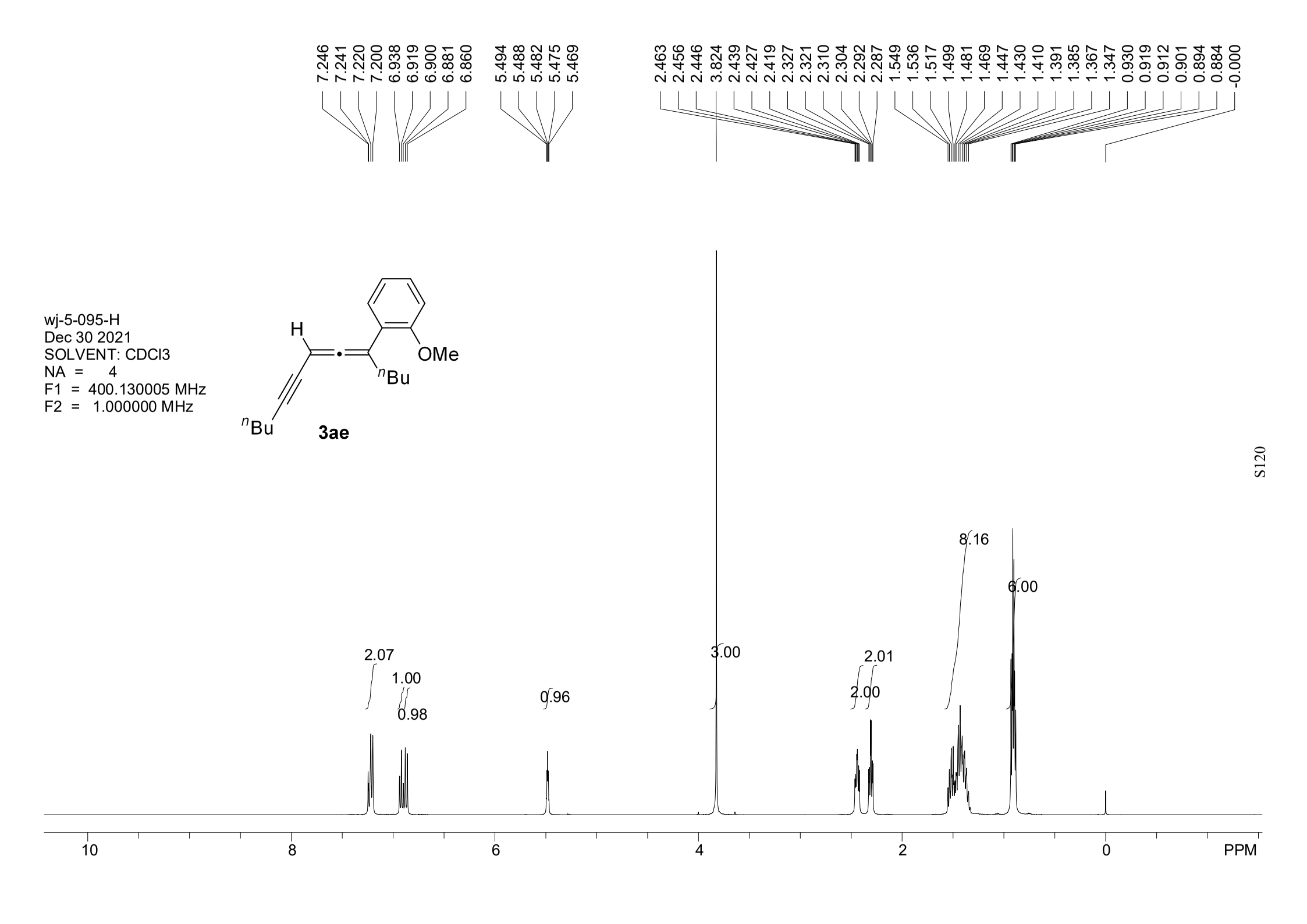

wj-5-060-C
Dec 10 2021
SOLVENT: CDCl3
NA = 250
F1 = 100.612770 MHz
F2 = 1.000000 MHz
3af
nBu
nBu
H
Ph
213.314
140.713
139.892
134.630
128.753
127.244
127.102
126.936
126.802
107.399
91.165
79.172
77.316
77.000
76.684
72.860
30.817
29.805
29.592
22.403
21.984
19.291
13.926
13.595
200
150
100
50
PPM

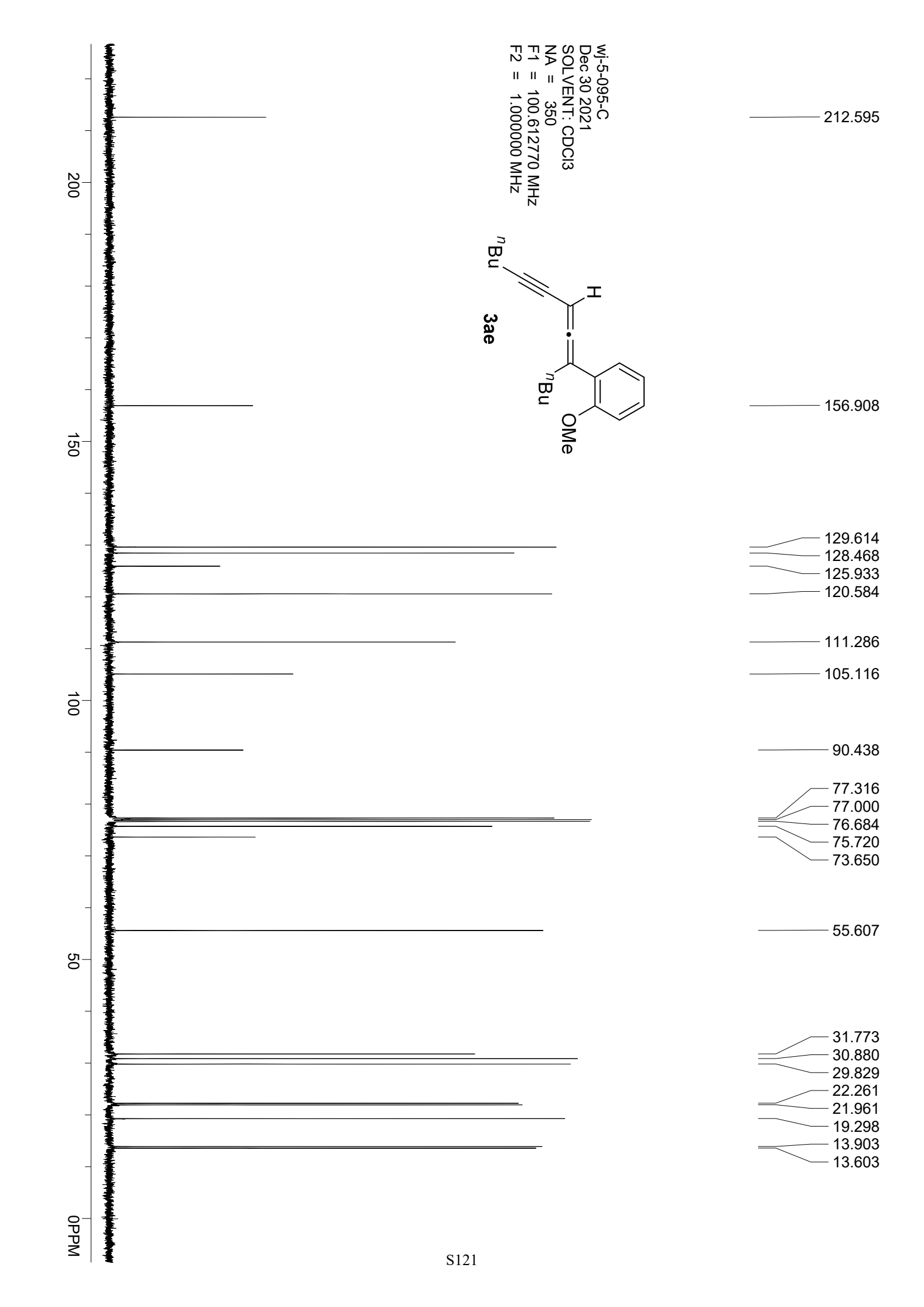

wj-5-073-H
Dec 17 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
nBu
H
nBu
COMe
3an
7.924
7.903
7.480
7.459
7.264
5.801
5.795
5.788
5.782
5.775
2.588
2.508
2.500
2.490
2.482
2.472
2.463
2.454
2.338
2.332
2.320
2.314
2.302
2.297
1.588
1.569
1.550
1.544
1.531
1.526
1.506
1.488
1.475
1.456
1.445
1.438
1.426
1.416
1.407
1.389
0.963
0.945
0.934
0.927
0.915
0.897
-0.000
1.97
1.95
0.96
3.00
2.10
1.97
4.01
4.08
6.01
10
8
6
4
2
0
PPM

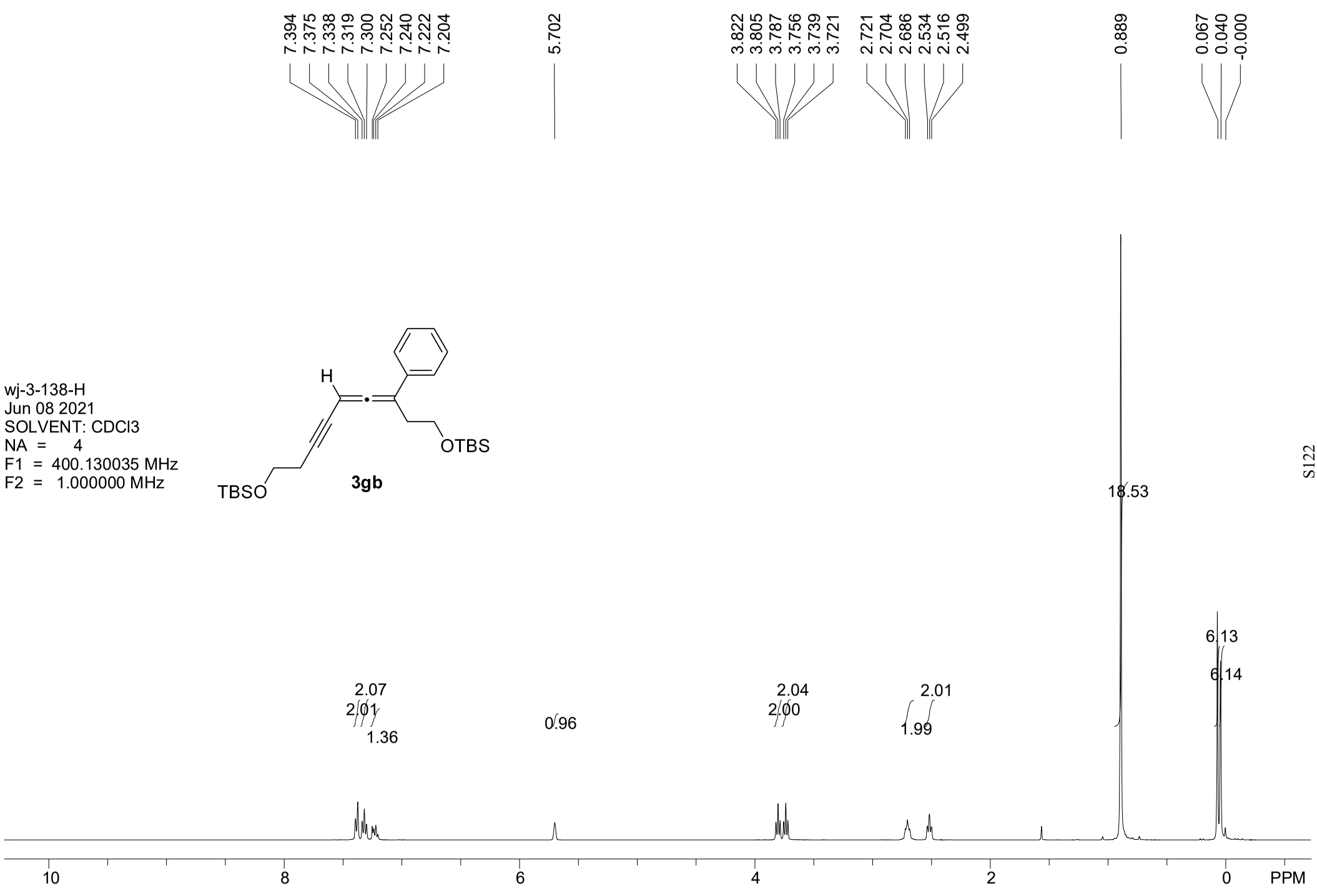

wj-5-073-C
Dec 17 2021
SOLVENT: CDCl3
NA = 400
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3an
nBu
COMe
214.025
197.546
140.785
135.642
128.500
126.430
107.273
92.010
79.733
77.316
77.000
76.676
72.142
30.722
29.687
29.410
26.535
22.316
21.953
19.259
13.871
13.563
200
150
100
50
0 PPM

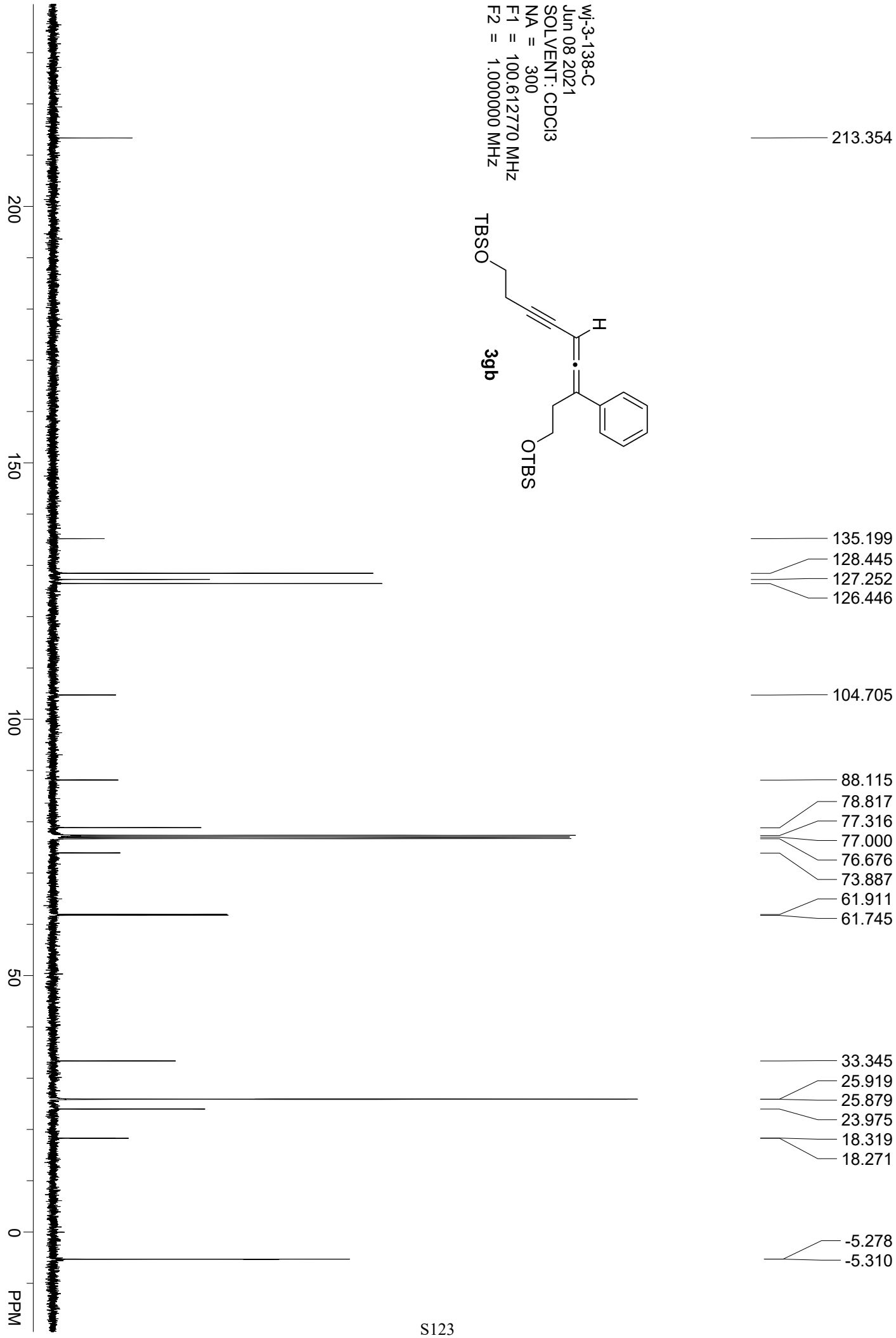

7.259
7.250
7.239
7.219
7.001
6.982
6.936
6.785
6.764
5.711
2.471
3.809
2.461
2.453
2.443
2.434
2.425
2.416
2.318
2.301
2.287
2.284
1.575
1.556
1.548
1.535
1.517
1.496
1.477
1.459
1.441
1.423
1.404
1.385
1.368
0.951
0.932
0.910
0.892
-0.000
wj-3-100-H
May 11 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
OMe
H
nBu
nBu
3aa
5.99
4.28
4.02
3.00
1.98
2.02
1.01
1.13
0.99
0.96
0.94
S118
10
8
6
4
2
0
PPM

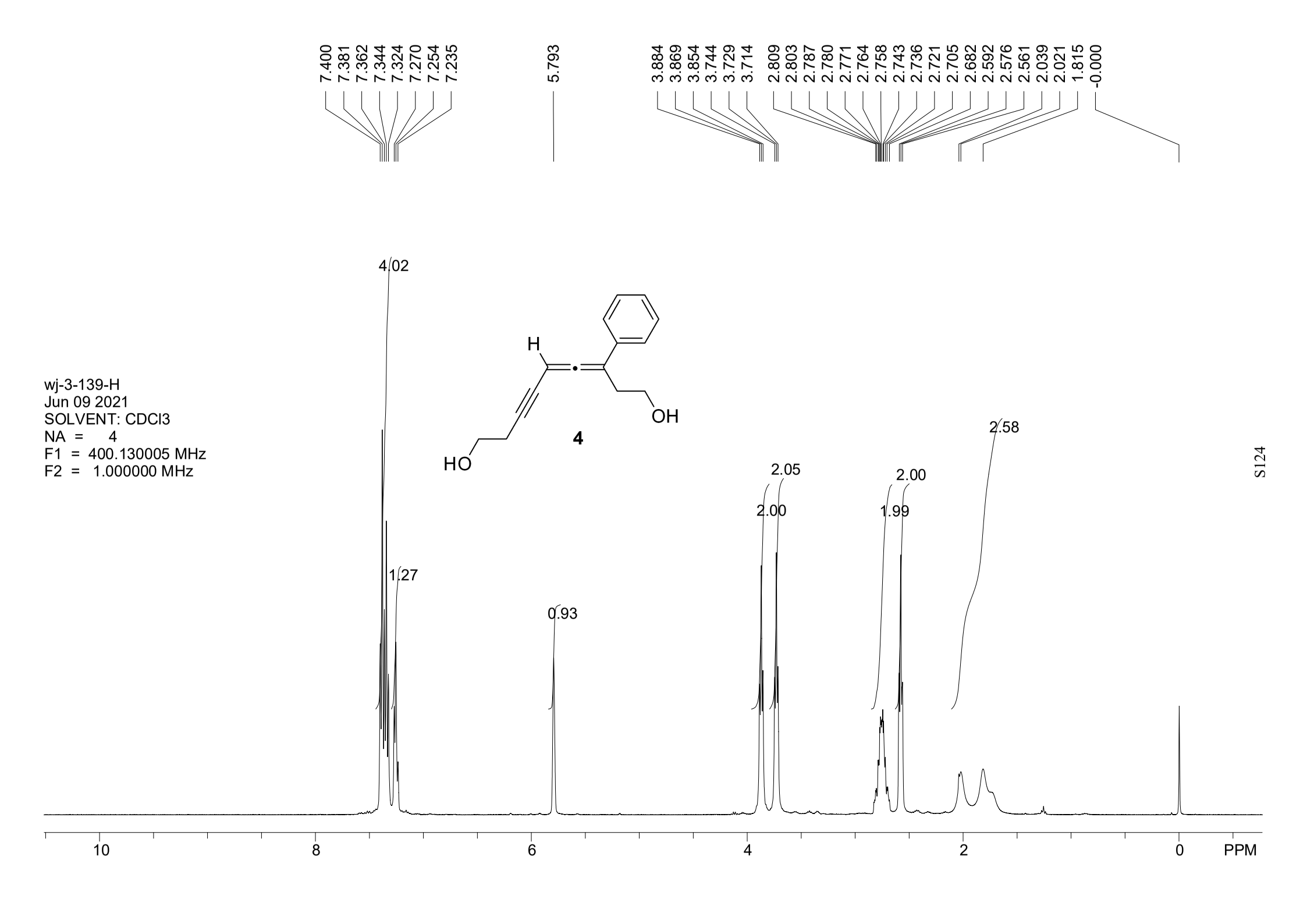

wj-3-100-C
May 11 2021
SOLVENT: CDCl3
NA = 200
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
nBu
OMe
3aa
213.085
159.689
137.253
129.290
118.965
112.368
112.345
107.589
91.109
79.015
77.316
77.000
76.684
72.829
55.196
30.801
29.790
29.671
22.372
21.961
19.267
13.903
13.587
200
150
100
50
0
PPM

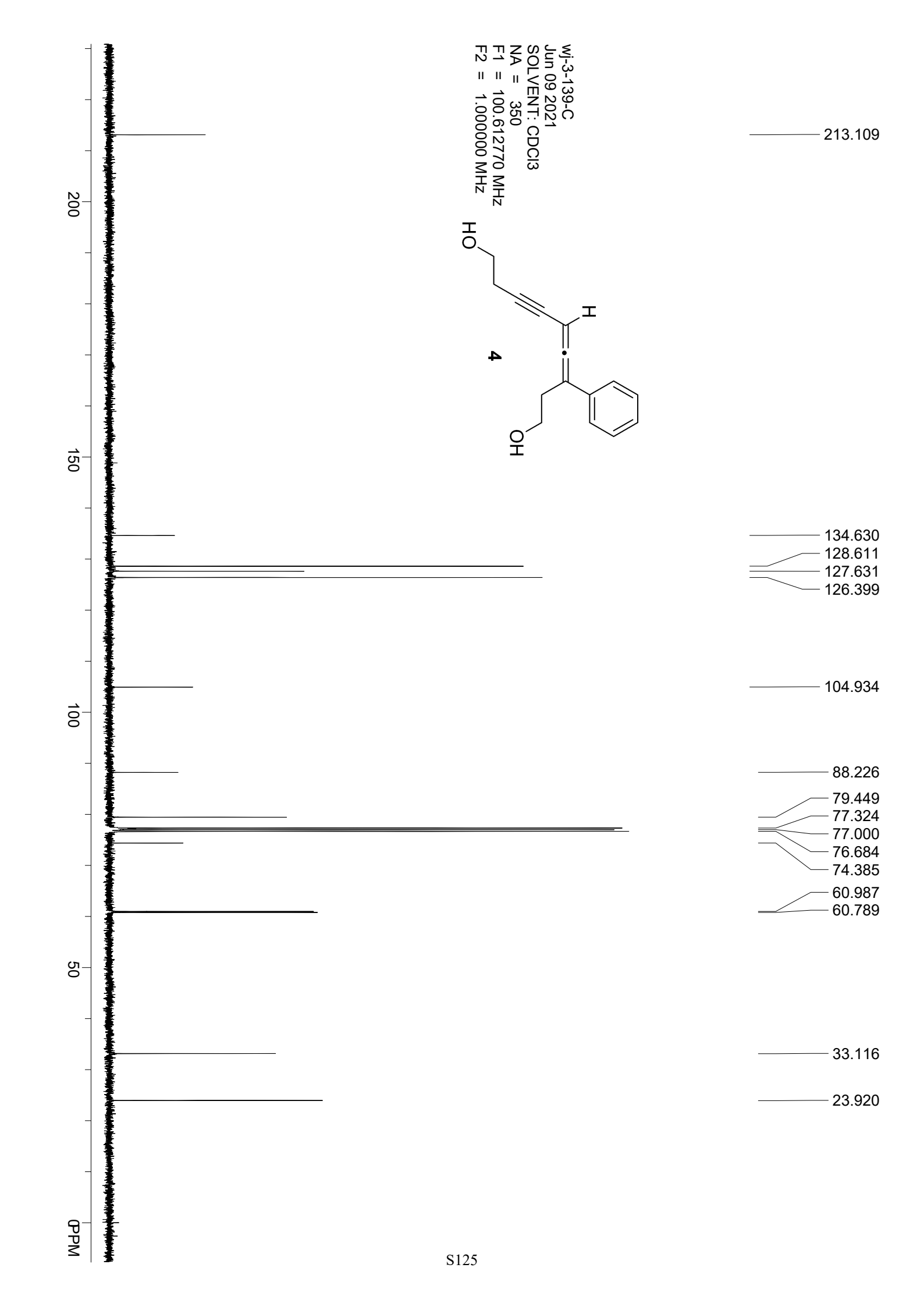

7.246
7.241
7.220
7.200
6.938
6.919
6.900
6.881
6.860
5.494
5.488
5.482
5.475
5.469
2.463
2.456
2.446
3.824
2.439
2.427
2.419
2.327
2.321
2.310
2.304
2.292
2.287
1.549
1.536
1.517
1.499
1.481
1.469
1.447
1.430
1.410
1.391
1.385
1.367
1.347
0.930
0.919
0.912
0.901
0.894
0.884
-0.000
wj-5-095-H
Dec 30 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
OMe
nBu
nBu
3ae
2.07
1.00
0.98
0.96
3.00
2.01
2.00
8.16
6.00
10
8
6
4
2
0
PPM

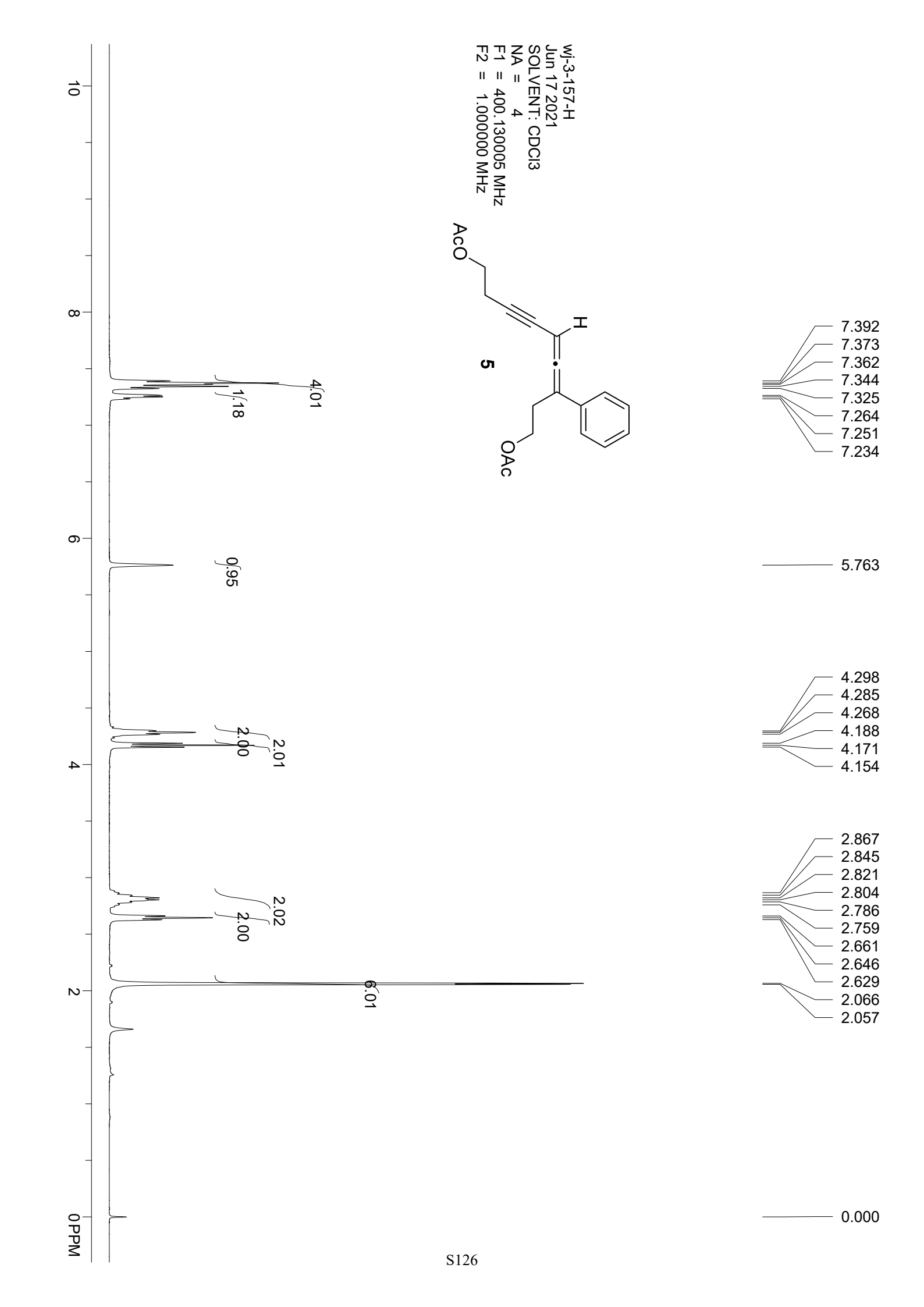

wj-5-095-C
Dec 30 2021
SOLVENT: CDCl3
NA = 350
F1 = 100.612770 MHz
F2 = 1.000000 MHz
nBu
H
3ae
nBu
OMe
212.595
156.908
129.614
128.468
125.933
120.584
111.286
105.116
90.438
77.316
77.000
76.684
75.720
73.650
55.607
31.773
30.880
29.829
22.261
21.961
19.298
13.903
13.603
200
150
100
50
0PPM

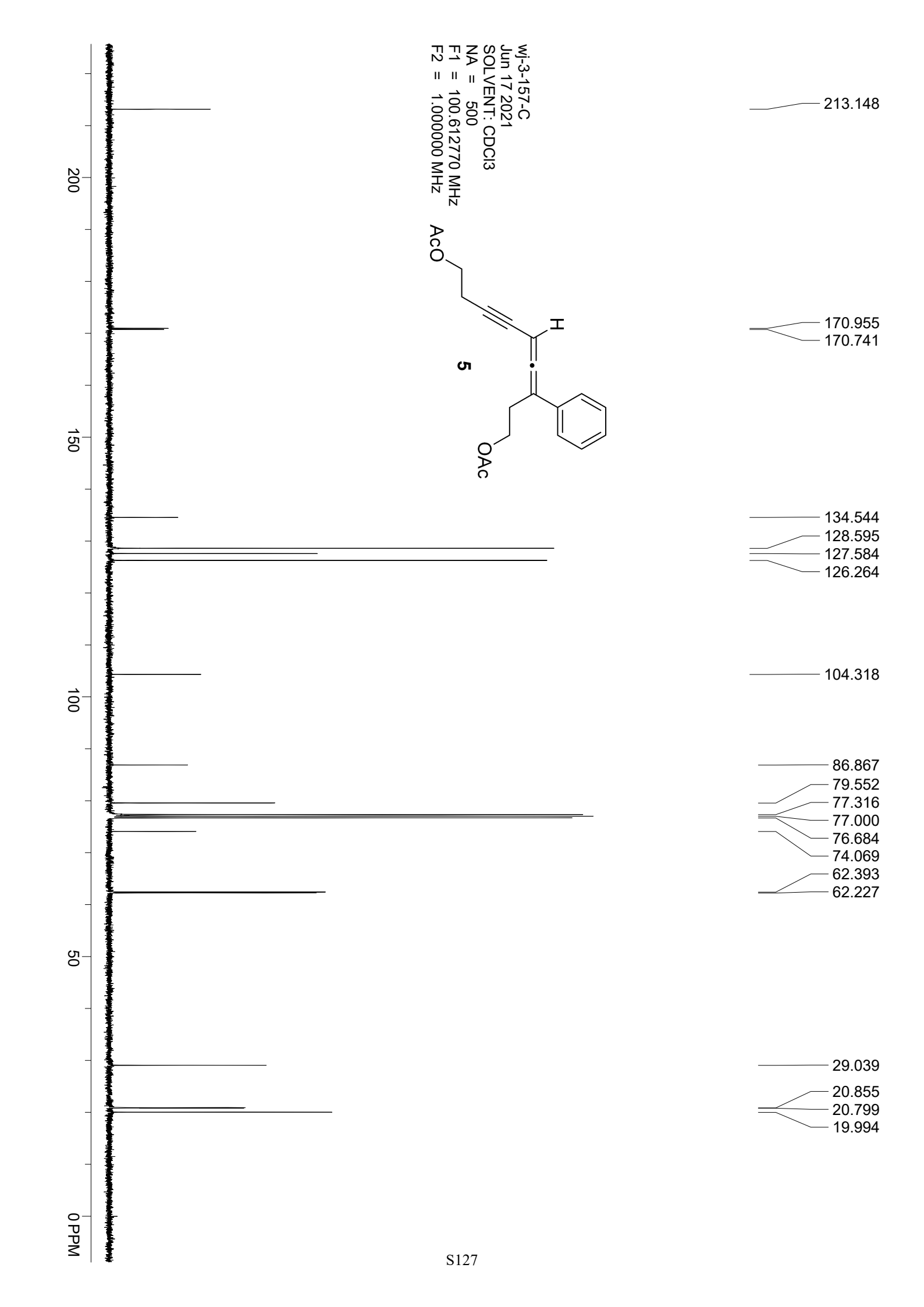

7.394
7.375
7.338
7.319
7.300
7.252
7.240
7.222
7.204
5.702
3.822
3.805
3.787
3.756
3.739
3.721
2.721
2.704
2.686
2.534
2.516
2.499
0.889
0.067
0.040
-0.000
wj-3-138-H
Jun 08 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
H
OTBS
TBSO
3gb
18.53
6.13
6.14
2.07
2.01
1.36
0.96
2.04
2.00
2.01
1.99
10
8
6
4
2
0
PPM

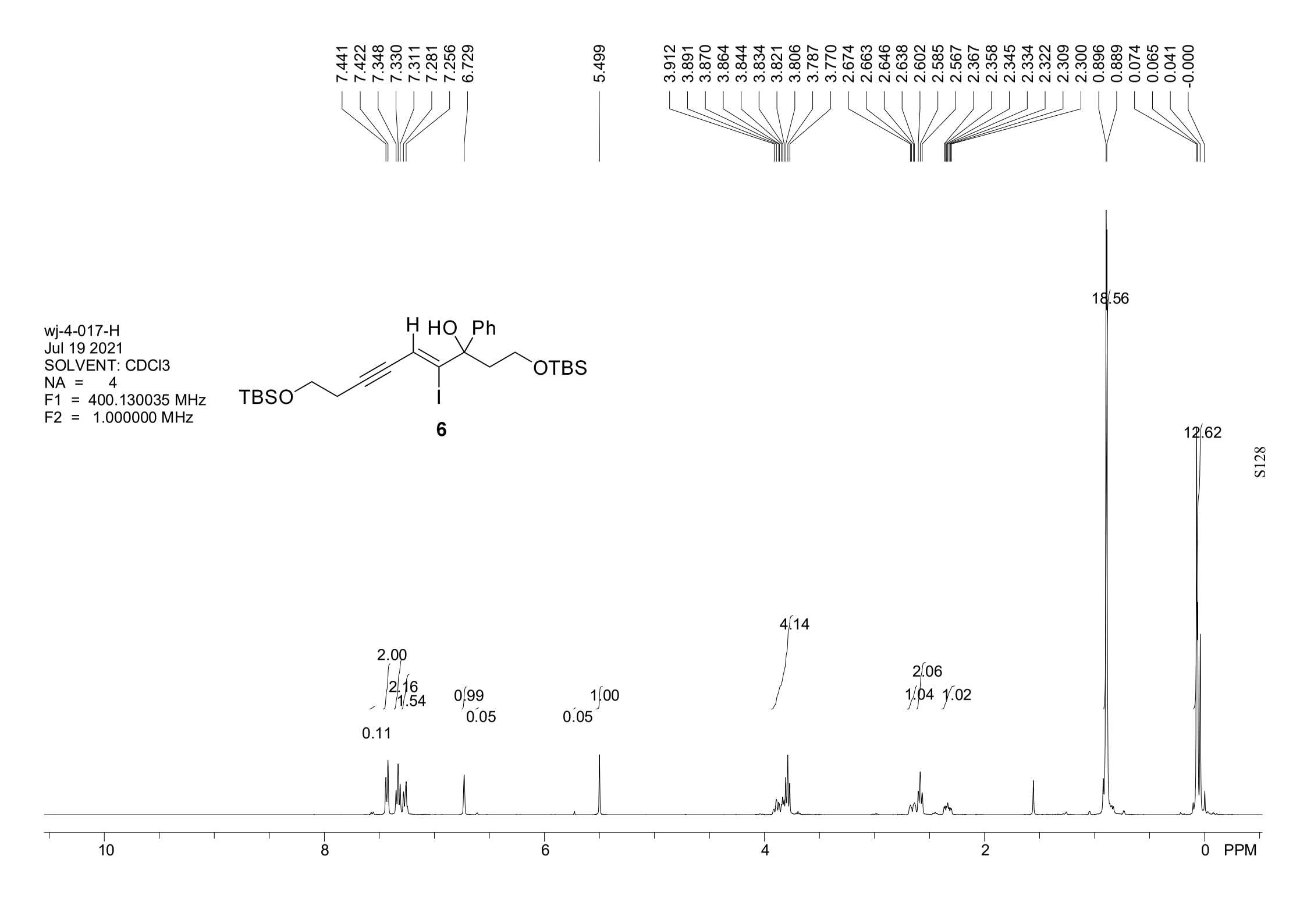

wj-3-138-C
Jun 08 2021
SOLVENT: CDCl3
NA = 300
F1 = 100.612770 MHz
F2 = 1.000000 MHz
TBSO
H
OTBS
3gb
213.354
135.199
128.445
127.252
126.446
104.705
88.115
78.817
77.316
77.000
76.676
73.887
61.911
61.745
33.345
25.919
25.879
23.975
18.319
18.271
-5.278
-5.310
200
150
100
50
0
PPM

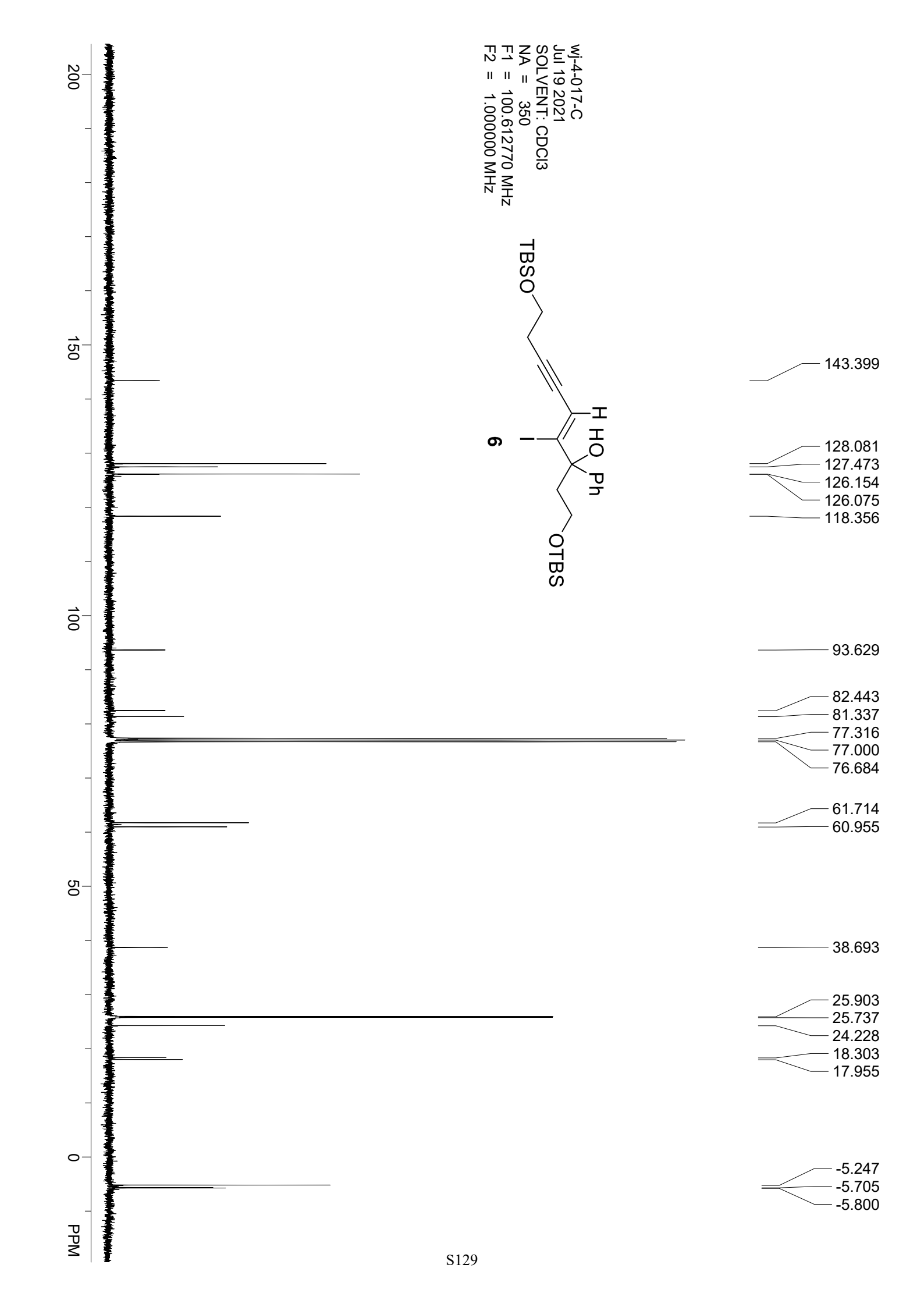

7.400
7.381
7.362
7.344
7.324
7.270
7.254
7.235
5.793
3.884
3.869
3.854
3.744
3.729
3.714
2.809
2.803
2.787
2.780
2.771
2.764
2.758
2.743
2.736
2.721
2.705
2.682
2.592
2.576
2.561
2.039
2.021
1.815
-0.000
wj-3-139-H
Jun 09 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
H
OH
HO
4
4.02
1.27
0.93
2.05
2.00
2.00
1.99
2.58
S124
10
8
6
4
2
0
PPM

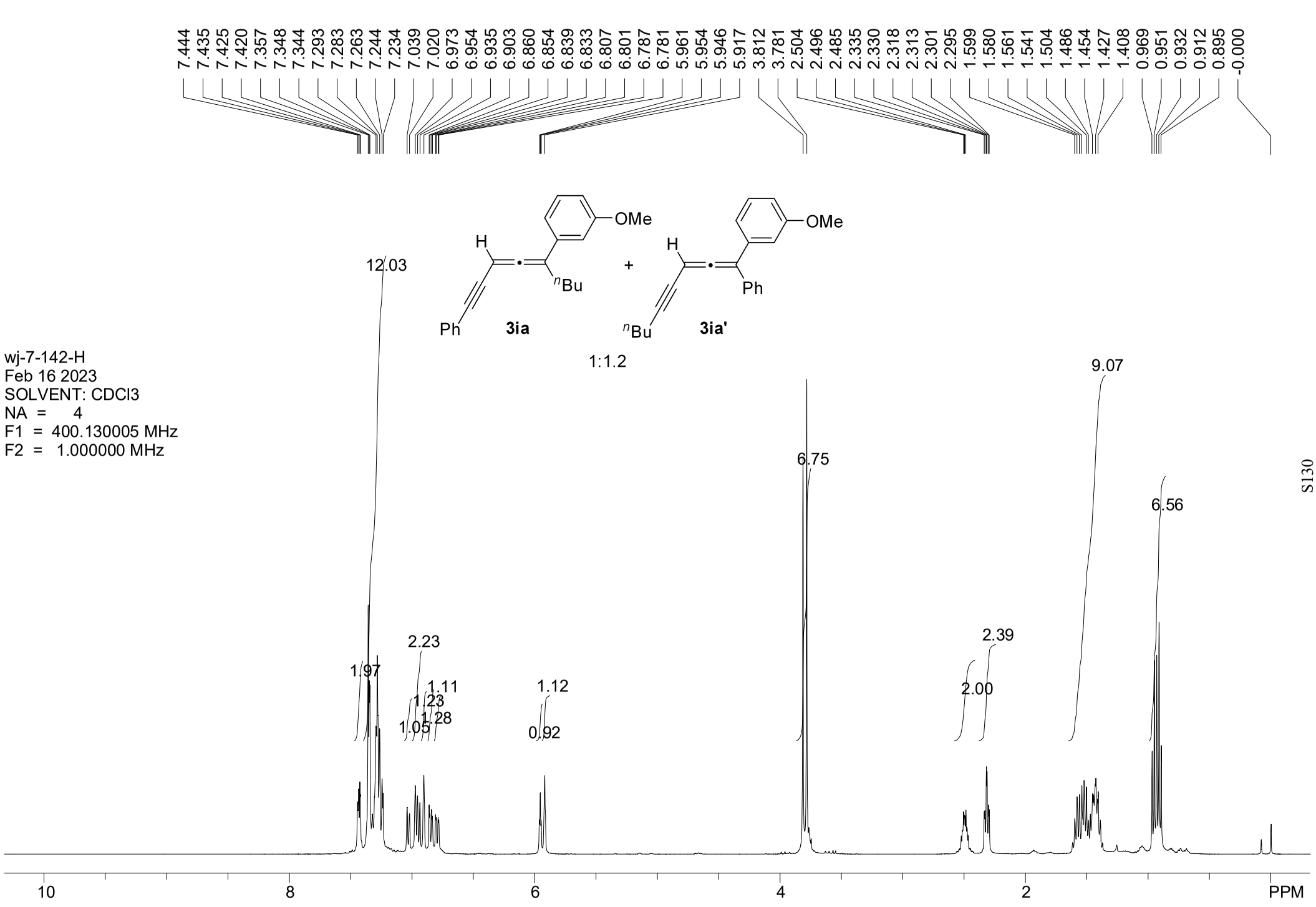

wj-3-139-C
Jun 09 2021
SOLVENT: CDCl3
NA = 350
F1 = 100.612770 MHz
F2 = 1.000000 MHz
HO
H
OH
4
213.109
134.630
128.611
127.631
126.399
104.934
88.226
79.449
77.324
77.000
76.684
74.385
60.987
60.789
33.116
23.920
200
150
100
50
0
PPM

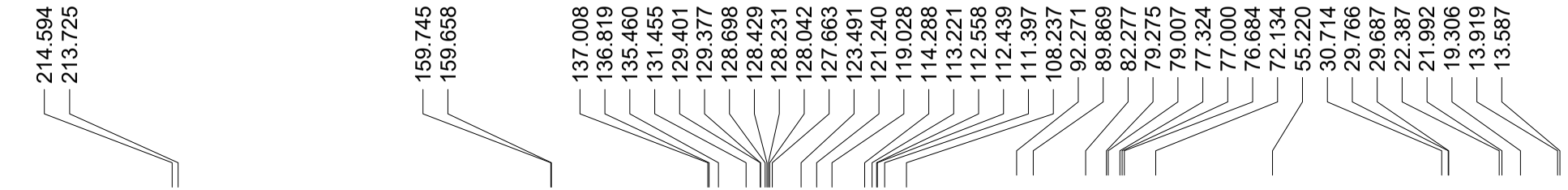

wj-3-157-H
Jun 17 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
AcO
H
5
OAc
7.392
7.373
7.362
7.344
7.325
7.264
7.251
7.234
5.763
4.298
4.285
4.268
4.188
4.171
4.154
2.867
2.845
2.821
2.804
2.786
2.759
2.661
2.646
2.629
2.066
2.057
0.000
4.01
1.18
0.95
2.00
2.01
2.00
2.02
6.01
10
8
6
4
2
0 PPM

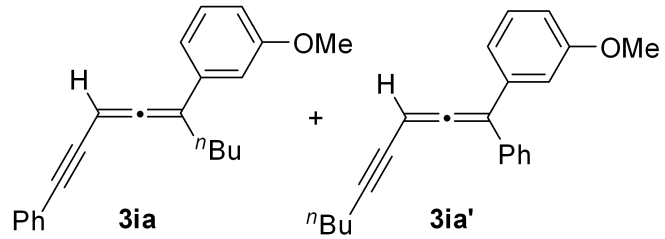

wj-3-157-C
Jun 17 2021
SOLVENT: CDCl3
NA = 500
F1 = 100.612770 MHz
F2 = 1.000000 MHz
AcO
H
5
OAc
213.148
170.955
170.741
134.544
128.595
127.584
126.264
104.318
86.867
79.552
77.316
77.000
76.684
74.069
62.393
62.227
29.039
20.855
20.799
19.994
200
150
100
50
0 PPM


7.441
7.422
7.348
7.330
7.311
7.281
7.256
6.729
5.499
3.912
3.891
3.870
3.864
3.844
3.834
3.821
3.806
3.787
3.770
2.674
2.663
2.646
2.638
2.602
2.585
2.567
2.367
2.358
2.345
2.334
2.322
2.309
2.300
0.896
0.889
0.074
0.065
0.041
-0.000
wj-4-017-H
Jul 19 2021
SOLVENT: CDCl3
NA = 4
F1 = 400.130035 MHz
F2 = 1.000000 MHz
H
HO
Ph
OTBS
TBSO
I
6
18.56
12.62
4.14
2.00
2.16
1.54
0.99
0.05
0.05
1.00
2.06
1.04
1.02
0.11
S128
10
8
6
4
2
0
PPM


wj-4-017-C
Jul 19 2021
SOLVENT: CDCl3
NA = 350
F1 = 100.612770 MHz
F2 = 1.000000 MHz
TBSO
H
HO
Ph
I
OTBS
6
143.399
128.081
127.473
126.154
126.075
118.356
93.629
82.443
81.337
77.316
77.000
76.684
61.714
60.955
38.693
25.903
25.737
24.228
18.303
17.955
-5.247
-5.705
-5.800
200
150
100
50
0
PPM


7.444
7.435
7.425
7.420
7.357
7.348
7.344
7.293
7.283
7.263
7.244
7.234
7.039
7.020
6.973
6.954
6.935
6.903
6.860
6.854
6.839
6.833
6.807
6.801
6.787
6.781
5.961
5.954
5.946
5.917
3.812
3.781
2.504
2.496
2.485
2.335
2.330
2.318
2.313
2.301
2.295
1.599
1.580
1.561
1.541
1.504
1.486
1.454
1.427
1.408
0.969
0.951
0.932
0.912
0.895
-0.000
OMe
H
nBu
Ph
3ia
+
OMe
H
Ph
nBu
3ia'
1:1.2
wj-7-142-H
Feb 16 2023
SOLVENT: CDCl3
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
12.03
2.23
1.97
1.11
1.23
1.28
1.05
1.12
0.92
6.75
2.39
2.00
9.07
6.56
S130
10
8
6
4
2
PPM





1:1.2

wj-7-142-C
Feb 16 2023
SOLVENT: CDCl3
NA = 350
F1 = 100.612770 MHz
F2 = 1.000000 MHz