Supporting Information

Alkyne Ligation Handles: Propargylation of Hydroxyl, Sulfhydryl, Amino, and Carboxyl Groups via the Nicholas Reaction

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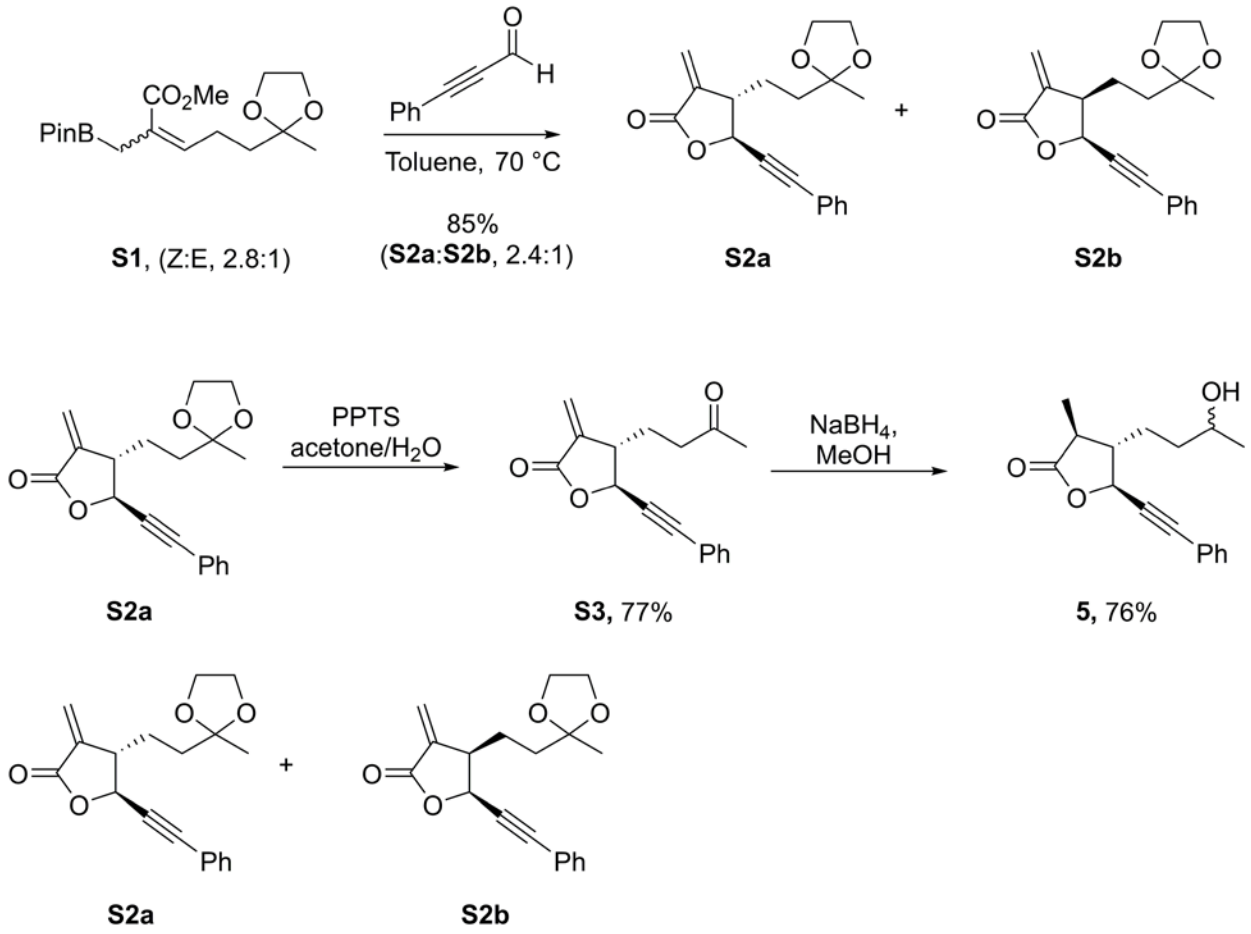
General Methods

All commercially available compounds were used as received unless otherwise noted. Dichloromethane (DCM), diethyl ether (Et_2O), and tetrahydrofuran (THF) were purified by passing through alumina using a solvent purification system. Deuterated chloroform ($CDCl_3$) was stored over 4 Å molecular sieves. Boron trifluoride diethyl etherate (BF_3•OEt_2) was used as received or redistilled under a nitrogen atmosphere. Dicobalt octacarbonyl ($Co_2(CO)_8$) was used as purchased and was stored at -20 ºC. All reactions were carried out using standard Schlenk line techniques unless otherwise noted. Purification of compounds via manual flash column chromatography or with a CombiFlash Rf200 instrument (Teledyne Isco) was performed using silica gel (40-63 μm particle size, 60 Å pore size) purchased from Sorbent Technologies or Silicycle. TLC analyses were performed on Silicycle SiliaPlate G silica gel glass plates (250 μm thickness) or EMD Chemicals Silica Gel 60 F254 glass plates (250 μm thickness) and visualized by UV irradiation (at 254 nm) and $KMnO_4$ stain. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker Avance 300 MHz, 400 MHz, 500 MHz, or 600 MHz. Spectra were referenced to residual chloroform with or without 0.05% v/v TMS (7.26 ppm, ^{1}H; 77.16 ppm, ^{13}C). Chemical shifts are reported in ppm, multiplicities are indicated by s (singlet), br s (broad singlet), d (doublet), t (triplet), q (quartet), p (pentet), and m (multiplet). Coupling constants, *J*, are reported in hertz (Hz). All NMR spectra were obtained at room temperature. IR spectra were obtained using a Nicolet Avatar E.S.P. 360 or a Perkin Elmer Spectrum 100 (NaCl plate) FT-IR. ESI mass spectrometry was performed on a Waters Q-TOF Ultima API, Micromass UK Limited or a Thermo Fisher Orbitrap Velos high resolution mass spectrometer.

Synthesis of alcohol 5



***Trans* and *cis*-4-(2-(2-methyl-1,3-dioxolan-2-yl)ethyl)-3-methylene-5-(phenylethynyl) dihydrofuran-2(3H)-one (S2a,b).** A 2 mL vial equipped with a stir bar and cap pierced with a nitrogen inlet needle was charged with a solution of *Z/E* (2.8: 1) allyl boronic ester **S1**[1] (43 mg, 0.13 mmol, 1 equiv) in toluene (0.7 mL) followed by a solution of 3-phenylpropioaldehyde (33 mg, 0.25 mmol, 2 equiv) in toluene (0.3 mL). The vial was lowered into a preheated oil bath (70 °C) and stirred for 6 d. The solution was cooled to RT and quenched by a 9:1 by volume solution of saturated ammonium chloride and ammonium hydroxide (7 mL). The contents were

[1] Allylboronic ester **S1** was previously synthesized in our group. Manuscript describing synthesis is in preparation.

transferred to a separatory funnel and extracted with ethyl acetate (3 x 8 mL). The combined organics were dried over magnesium sulfate, gravity filtered, and concentrated using reduced pressure rotary evaporation. The crude material was purified by silica gel flash column chromatography (gradient of 10-20% ethyl acetate in hexanes) to afford 18 mg of pure *trans*-lactone **S2a**, 9 mg of a mixture of *trans*- and *cis*-lactones **S2a,b** (1 : 1.3), and 6 mg of a mixture of *trans*- and *cis*-lactones **S2a,b** (1 : 3.0) in an overall 85% yield, as clear oils. *Cis*- and *trans*-configurations were determined by comparison to previous literature, where an X-ray was obtained for a *trans*-lactone compound.[2]

Data for **S2 a-b**

^{1}H NMR *Trans*-lactone **S2a** (400 MHz, $CDCl_3$): 7.45-7.36 (m, 2H), 7.35-7.31 (m, 3H), 6.35 (d, J = 2.4 Hz, 1H), 5.69 (d, J = 2.4 Hz, 1H), 4.98 (d, J = 5.2 Hz, 1H), 4.00-3.91 (m, 4H), 3.20-3.19 (m, 1H), 1.90-1.79 (m, 4H), 1.34 (s, 3H) ppm;

Cis-lactone **S2b** (500 MHz, $CDCl_3$): 7.46-7.44 (m, 2H), 7.35-7.32 (m, 3H), 6.33 (d, J = 3.0 Hz, 1H), 5.65 (d, J = 3.0 Hz, 1H), 5.48 (d, J = 8.0 Hz, 1H), 3.96-3.90 (m, 4H), 3.24-3.17 (m, 1H), 2.01-1.94 (m, 2H), 1.92-1.83 (m, 1H), 1.81-1.76 (m, 1H), 1.34 (s, 3H) ppm;

Impurities seen at 1.55, 1.25, 0.88 ppm.

^{13}C NMR *Trans*-lactone **S2a** (100 MHz, $CDCl_3$): 169.4, 137.7, 132.0 (2C), 129.3, 128.5 (2C), 123.3, 121.7, 109.5, 88.0, 85.2, 72.5, 65.0, 64.9, 47.0, 35.8, 27.5, 24.1 ppm;

Cis-lactone **S2b** (125 MHz, $CDCl_3$): 169.7, 137.7, 132.0 (2C), 129.3, 128.5 (2C), 122.5, 121.7, 109.7, 89.9, 82.3, 71.6, 64.9, 64.8, 43.3, 36.1, 24.5, 23.9 ppm;

Impurities seen at 104.5, 22.8, 14.3 ppm.

[2] Grillet, F.; Huang, C.; Brummond, K. M. *Org. Lett.* **2011**, *13*, 6304-6307.

IR (thin film)

2981, 2930, 2884, 2233, 1771, 1491, 1444, 1264, 1132, 1064, 759, 692 cm^{-1};

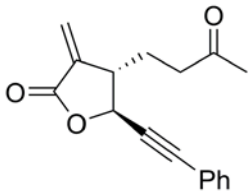
HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{19}H_{21}O_4$, 313.1434; found, 313.1433;

TLC *Trans*-lactone **S2a**: R_f = 0.25 (30% ethyl acetate in hexanes)

Cis-lactone **S2b**: R_f = 0.18 (30% ethyl acetate in hexanes)

Silica gel, UV



***Trans*-3-methylene-4-(3-oxobutyl)-5-(phenylethynyl)dihydrofuran-2(3H)-one (S3).** A 2 mL vial equipped with stir bar and cap pierced with a nitrogen inlet needle was charged with pyridinium *p*-toluenesulfonate (7 mg, 0.029 mmol, 0.5 equiv), followed by dioxolane *trans*-**S2a** (18 mg, 0.058 mmol, 1 equiv) dissolved in acetone (1 mL). The vial was lowered into a preheated oil bath (60 ºC) and stirred for 15 h. When the reaction was complete, as evidenced by TLC, the oil bath was removed and the vial cooled to rt. The reaction was diluted with ethyl acetate (4 mL). The contents were transferred to a separatory funnel, and washed with water (2 x 6 mL) followed by brine (6 mL). The organics were dried over magnesium sulfate, gravity filtered, and concentrated under reduced pressure rotary evaporation. The crude residue was purified by silica gel flash column chromatography (20% ethyl acetate in hexanes) to afford 12 mg of **S3** in 77% yield as a colorless oil.

Data for **S3**

^{1}H NMR (400 MHz, $CDCl_3$)

7.45-7.42 (m, 2H), 7.40-7.31 (m, 3H), 6.36 (d, *J* = 2.8 Hz, 1H), 5.69 (d, *J* = 2.4 Hz, 1H), 4.94 (d, *J* = 5.6 Hz, 1H), 3.24-3.19 (m, 1H), 2.74-2.64 (m, 2H), 2.17 (s, 3H), 2.16-2.07 (m, 1H), 1.94-1.84 (m, 1H) ppm;

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

207.0, 169.1, 137.4, 132.0 (2C), 129.4, 128.6 (2C), 123.3, 121.5, 88.3, 85.0, 72.4, 46.2, 39.7, 30.3, 26.3 ppm;

IR (thin film)

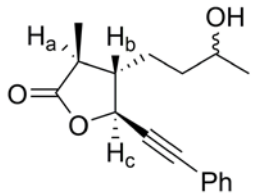
2921, 2852, 2233, 1769, 1714, 1491, 1444, 1408, 1364, 1268, 1130, 985, 759, 691 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{17}H_{17}O_3$, 269.1172; found, 269.1168;

TLC R_f = 0.22 (30% ethyl acetate in hexanes)

Silica gel, UV



4-(3-Hydroxybutyl)-3-methyl-5-(phenylethynyl)dihydrofuran-2(3H)-one (5). A 10 mL, single-necked, round-bottomed flask equipped with a stir bar and a septum pierced with a needle was charged with ketone **S3** (52 mg, 0.19 mmol, 1 equiv), dissolved in methanol (2.5 mL). The solution was cooled in an ice bath to 0 °C. Sodium borohydride (11 mg, 0.29 mmol, 1.5 equiv) was added in a single portion and the reaction was stirred at 0 °C for 2 h, until complete as evidenced by TLC. The ice bath was removed and the reaction was quenched by adding 5% acetic acid in water solution (6 mL). The reaction contents were transferred to a separatory

funnel; the aqueous layer was separated and extracted with diethyl ether (3 x 8 mL). The combined organic layers were washed with saturated sodium bicarbonate (10 mL) and brine (10 mL), dried over magnesium sulfate, gravity filtered, and concentrated using reduced pressure rotary evaporation. The crude material was purified by silica gel flash column chromatography (gradient of 30-40% ethyl acetate in hexanes) to afford 41 mg of alcohol **5** (76% yield) as a colorless oil. The product was obtained as a 1:1 mixture of diastereomers that were inseparable by column chromatography. Relative stereochemistry for the α-methyl group of the lactone was determined by J_{ab} (10.8 Hz). This coupling as well as J_{bc} (9.0 Hz) are consistent with a *trans,trans*-trisubstituted butyrolactone ring.[3]

Data for **5**

^{1}H NMR (600 MHz, $CDCl_3$)

7.47-7.42 (m, 2H), 7.38-7.30 (m, 3H), 4.82 (d, *J* = 9.0 Hz, 1H), 3.90-3.83 (m, 1H), 2.34 (dq, *J* = 10.8, 6.6 Hz, 1 H), 2.33-2.24 (m, 1H), 1.92-1.85 (m, 0.5H)*, 1.81-1.58 (m, 3.5 H), 1.35 (d, *J* = 6.6 Hz, 1.5H), 1.34 (d, *J* = 6.6 Hz, 1.5H)*, 1.24 (d, *J* = 7.2 Hz, 1.5H), 1.23 (d, *J* = 7.2 Hz, 1.5H)* ppm;

Trace impurities are observed at 6.4, 5.7, 5.0, 4.7, 3.0, 2.1 ppm

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

177.8, 131.9 (2C), 129.2, 128.6 (2C), 121.8, 88.0, 85.00, 84.97*, 73.02, 73.01*, 68.1, 67.8*, 51.0, 50.8*, 41.3, 36.6, 36.4*, 28.3, 28.1*, 24.0, 23.9*, 14.6, 14.5* ppm;

* Discernable signal for 1 of 2 diastereomers

IR (thin film)

[3] Jaime, C.; Ortuño, R. M.; Font, J.; *J. Org. Chem.* **1986**, 51, 3946-3951.

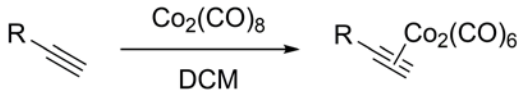
3430, 3059, 2969, 2934, 2360, 2232, 1780, 1491, 1456, 1331, 1166, 992, 759, 692 cm^{-1};

HRMS (FTMS + p ESI Full ms)

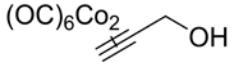
$[M+H]^+$ calcd for $C_{17}H_{21}O_3$, 273.1485; found, 273.1468;

TLC R_f = 0.24 (40% ethyl acetate in hexanes)

Silica gel, UV, potassium permanganate



General Procedure A: Coordination of alkyne to cobalt carbonyl complex. A single-necked, round-bottomed flask, equipped with a stir bar and a septum, was charged with dicobalt octacarbonyl (1 equiv) in a N_2 filled glove box. The flask was transferred out of the glove box and the septum was pierced with a nitrogen inlet needle. The flask was charged with dichloromethane, followed by the alkyne (1 equiv), dissolved in dichloromethane. The reaction stirred for 2 h, until evolution of CO gas, visible by small bubbles, was no longer observed. The contents were loaded directly onto a silica gel column for purification by flash column chromatography to afford the dicobalt hexacarbonyl complexed alkyne ($Co_2(CO)_6$-alkyne)



Dicobalt hexacarbonyl complexed propargyl alcohol 6a. Followed general procedure A: Dicobalt octacarbonyl (793 mg, 2.3 mmol, 1.3 equiv), dichloromethane (1.5 mL), propargyl alcohol (0.10 mL, 1.8 mmol, 1 equiv), dissolved in dichloromethane (2.5 mL). The reaction was stirred for 2 h. The silica gel flash column chromatography was run with a gradient of 10-20%

ethyl acetate in hexanes to afford 615 mg of $Co_2(CO)_6$-propargyl alcohol **4** in quantitative yield, as a dark red solid.

Data for **6a**

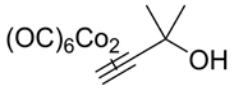
MP 48-52 °C

^{1}H NMR (300 MHz, $CDCl_3$)

6.08 (s, 1H), 4.81 (d, *J* = 6.0 Hz, 2H), 1.79 (t, *J* = 6.0 Hz, 1H) ppm;

TLC R_f = 0.28 (10% ethyl acetate in hexanes)

Silica gel, visible (red)



Dicobalt hexacarbonyl complexed 2-methyl-3-butyn-2-ol (S4). Followed general procedure A: Dicobalt octacarbonyl (1.37 g, 4.0 mmol, 1.0 equiv), dichloromethane (15 mL), 2-methyl-3-butyn-2-ol (344 mg, 4.0 mmol, 1 equiv), dissolved in dichloromethane (5 mL). The reaction was stirred for 2 h. The silica gel flash column chromatography was run with a gradient of 10-30% ethyl acetate in hexanes to afford 1.36 g of $Co_2(CO)_6$-2-methyl-3-butyn-2-ol **S4** in 92% yield, as a dark red solid.

Data for **S4**

MP 37.9-40.1 °C

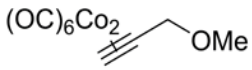
^{1}H NMR (300 MHz, $CDCl_3$)

6.03 (s, 1H), 1.71 (s, 1H), 1.59 (s, 6H) ppm;

Impurities observed at 1.54, 1.27, 0.88 ppm

TLC R_f = 0.26 (20% diethyl ether in hexanes)

Silica gel, visible (red)



Dicobalt hexacarbonyl complexed methyl propargyl ether 6b. Followed general procedure A: Dicobalt octacarbonyl (195 mg, 0.57 mmol, 1 equiv), dichloromethane (1.5 mL), methyl propargyl ether (40 mg, 0.57 mmol, 1 equiv), dissolved in dichloromethane (3.0 mL). The reaction was stirred for 1.5 h. The silica gel flash column chromatography was run with a gradient of 0-2.5% diethyl ether in hexanes to afford 94 mg of $Co_2(CO)_6$-methyl propargyl ether **6b** in 45% yield, as a dark red oil. Drying under high vacuum was not performed due to the volatility of the parent compound.

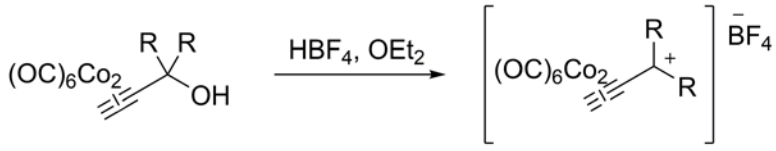
Data for **6b**

^{1}H NMR (300 MHz, $CDCl_3$)

6.06 (s, 1H), 4.60 (s, 2H), 3.49 (s, 3H) ppm;

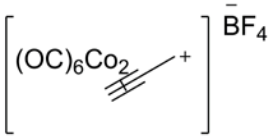
TLC R_f = 0.59 (10% diethyl ether in hexanes)

Silica gel, visible (red)

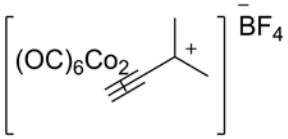


General Procedure B: Formation of propargylium tetrafluoroborate salts. A flame-dried 100 mL Schlenk flask equipped with a stir bar and septum was charged with either $Co_2(CO)_6$-propargyl alcohol **6a** or $Co_2(CO)_6$-2-methyl-3-butyn-2-ol (**S4**), dissolved in diethyl ether. The flask was cooled to -10 °C on an ice/acetone bath. Tetrafluoroboric acid (54% by weight soln in diethyl ether, 1.5 equiv) was added dropwise and the solution stirred for 2 h. Formation of a dark red precipitate was observed. The reaction was diluted with diethyl ether. The septum was replaced with a Schlenk filtration apparatus. The apparatus was inverted and partial vacuum was applied to separate the solid from the ether solution within the apparatus. The ether filtrate was

removed via syringe and the crystals were dried under vacuum. The apparatus was transferred to the nitrogen filled glove box, where the crystals were isolated and stored.



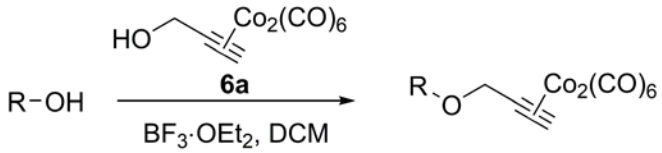
α-(Ethynyl)dicobalt hexacarbonyl carbonium tetrafluoroborate salt 6c.[4] Follows General procedure B: $Co_2(CO)_6$-propargyl alcohol **6a** (500 mg, 1.46 mmol, 1 equiv), diethyl ether (5 mL), tetrafluoroboric acid (356 mg of a 54% by weight soln in diethyl ether, 2.19 mmol, 1.5 equiv). The reaction was diluted with diethyl ether (20 mL) prior to filtration and drying which afforded 601 mg of salt **6c** in 60% yield as a red solid. Due to sensitivity to water and air, the salt was stored in the glove box and used within 24 h of isolation for best results.



α-(Dimethylethynyl)dicobalt hexacarbonyl carbonium tetrafluoroborate salt 12.[2] Follows General procedure B: $Co_2(CO)_6$-2-methyl-3-butyn-2-ol **S4** (774 mg, 2.09 mmol, 1 equiv), diethyl ether (10 mL), tetrafluoroboric acid (509 mg of a 54% by weight in diethyl ether, 3.14 mmol, 1.5 equiv). The reaction was diluted with diethyl ether (10 mL) prior to filtration and drying which afforded 558 mg of salt **12** in 61% yield as a red solid. Due to sensitivity to water and air, the salt was stored in the glove box and used within 24 h of isolation for best results.

[4] Conner, R. E.; Nicholas, K. M; *J. Organomet. Chem.*, **1977**, *125*, C45-C48

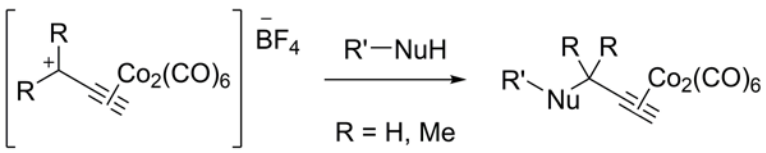
General Procedure C: Nicholas Reaction Procedures



General Procedure C1: Use of pre-made dicobalt hexacarbonyl complexed alkyne. A single-necked, round-bottomed flask equipped with a stir bar and a septum pierced with a needle was charged with dichloromethane (0.05 M), $Co_2(CO)_6$-propargyl alcohol **6a** or $Co_2(CO)_6$-methyl propargyl ether **6b** (2 equiv), and the nucleophilic species (1 equiv). The solution was cooled in an ice bath to 0 °C. Boron trifluoride diethyl etherate (2.5 equiv) was added dropwise and the reaction stirred until the nucleophile was fully consumed, or the reaction was no longer progressing, as determined by TLC. The reaction was quenched by the addition of saturated sodium bicarbonate. The mixture was transferred to a separatory funnel, the layers were separated and the aqueous layer was extracted with dichloromethane (3x). The combined organics were dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel flash column chromatography to afford the dicobalt hexacarbonyl complexed alkyne.

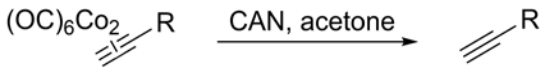
General Procedure C2: *In situ* formation of dicobalt hexacarbonyl complexed alkyne. A single necked, round-bottomed flask, equipped with a stir bar and a septum was charged with dicobalt octacarbonyl (2 equiv) in a N_2 filled glove box. The flask was transferred out of the glovebox and the septum was pierced with a nitrogen inlet needle. Either propargyl alcohol, methyl propargyl ether, or propargyl acetate (2 equiv), dissolved in dichloromethane (0.2 M),

was added and the reaction stirred for 1.5 h until evolution of CO gas was no longer observed. The solution was cooled to 0 °C in an ice bath. The nucleophilic species (1 equiv) was dissolved in dichloromethane (0.05 M overall) and added to the flask via syringe, followed by the dropwise addition of boron trifluoride diethyl etherate (2.5 equiv). The reaction stirred until the nucleophilic species was fully consumed, or the reaction was no longer progressing, as determined by TLC. The reaction was quenched by addition of saturated sodium bicarbonate. The mixture was transferred to a separatory funnel, the layers were separated, and the aqueous layer was extracted with dichloromethane (3x). The combined organic layers were dried over magnesium sulfate, filtered, and concentrated under reduced pressure rotary evaporation. The crude residue was purified by silica gel flash column chromatography to afford the dicobalt hexacarbonyl complexed alkyne.



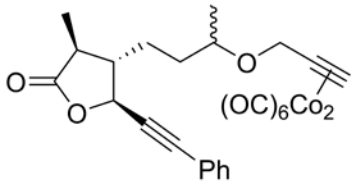
General Procedure C3: Reaction of tetrafluoroborate salts with nucleophiles. A single-necked, round-bottomed flask, equipped with a stir bar and a septum was charged with propargylium tetrafluoroborate salt **6c** or **12** (1.3 equiv) in a N_2 filled glove box. The flask was transferred out of the glove box and the septa was pierced with a nitrogen inlet needle. The flask was cooled to 0 °C in an ice and water bath. Dichloromethane was added followed by the amine nucleophile (1 equiv) dissolved in dichloromethane (0.05 M overall). The reaction stirred for 2 h or until complete as determined by TLC. The reaction was quenched by the addition of saturated sodium bicarbonate. The mixture was transferred to a separatory funnel; the layers were separated and the aqueous layer was extracted with dichloromethane (3x). The combined organics were dried over magnesium sulfate, filtered, and concentrated under reduced pressure

rotary evaporation. The crude residue was purified by silica gel flash column chromatography to afford the dicobalt hexacarbonyl complexed alkyne.



General Procedure D: Oxidative decomplexation of $Co_2(CO)_6$-alkynes.

A single-necked, round-bottomed flask, equipped with a stir bar and a septum pierced with a nitrogen inlet needle was charged with the dicobalt hexacarbonyl complexed alkyne (1 equiv), dissolved in acetone (0.01 M). The solution was cooled in an ice bath to 0° C. Ceric ammonium nitrate (5 equiv) was added to the flask in a single portion. The reaction stirred until complete as evidenced by TLC. The reaction was diluted with distilled water and diethyl ether. The mixture was transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with diethyl ether (3x). The combined organics were dried over magnesium sulfate, filtered, and concentrated under reduced pressure rotary evaporation. If necessary, the residue was purified by silica gel flash column chromatography to afford the alkyne.



Dicobalt hexacarbonyl complexed 3-methyl-5-(phenylethynyl)-4-(3-(prop-2-yn-1-yloxy)butyl)dihydrofuran-2(3H)-one (7). Method A: Follows general procedure C1: $Co_2(CO)_6$-propargyl alcohol **6a** (25 mg, 0.073 mmol, 2 equiv), alcohol **5** (10 mg, 0.037 mmol, 1 equiv), dichloromethane (0.75 mL), and boron trifluoride diethyl etherate (12 µL, 0.93 mmol, 2.5 equiv) The reaction stirred for 4 h. The crude residue was purified by silica gel flash column chromatography (gradient of 5-10% ethyl acetate/hexanes) to afford 12 mg of **7** in 55% yield as a

dark red/brown oil. **Method B:** Follows general procedure C2: propargyl alcohol (4 µL, 0.064 mmol, 2.2 equiv), dichloromethane (0.36 mL), dicobalt octacarbonyl (20 mg, 0.058 mmol, 2 equiv), alcohol **5** (8 mg, 0.029 mmol, 1 equiv), dissolved in dichloromethane (0.25 mL), and boron trifluoride diethyl etherate (9 µL, 0.073 mmol, 2.5 equiv). The reaction stirred for 3.5 h. The crude residue was purified by silica gel flash column chromatography (gradient of 5-10% ethyl acetate in hexanes) to afford 10 mg of **7** in 60% yield as a dark red/brown oil. The product was a 1:1 mixture of diastereomers that were inseparable by column chromatography.

Data for **7**

^{1}H NMR (500 MHz, $CDCl_3$)

7.46-7.44 (m, 2H), 7.37-7.33 (m, 3H), 6.01 (d, *J* = 4.5 Hz, 1 H), 4.79 (d, *J* = 9.0 Hz, 1H), 4.69 (dd, *J* = 13.0, 4.5 Hz, 1 H), 4.51 (d, *J* = 13.0, 1H), 3.71-3.64 (m, 1H), 2.34-2.19 (m, 2H), 1.95-1.87 (m, 0.5H)*, 1.80-1.61 (m, 3.5H), 1.33 (d, *J* = 6.8 Hz, 1.5H), 1.32 (d, *J* = 6.8 Hz, 1.5H)*, 1.24 (d, *J* = 6.3 Hz, 1.5H), 1.23 (d, *J* = 6.3 Hz, 1.5H)* ppm;

* Discernable signal for one of two diastereomers

^{13}C NMR (125 MHz, $CDCl_3$)

199.8 (6C), 177.9, 131.9 (2C), 129.2, 128.5 (2C), 121.9, 92.7, 87.9, 85.1, 75.2, 75.0*, 73.1, 71.3, 68.6, 51.2, 51.0*, 41.3, 34.5, 34.3*, 27.8, 27.5*, 19.3, 14.6, 14.4* ppm;

IR (thin film)

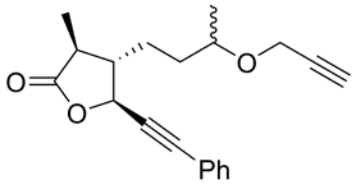
2971, 2934, 2094, 2052, 2022, 1784, 1491, 1456, 1377, 1327, 1164, 1086, 992, 758, 691 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calcd for $C_{26}H_{22}O_9Co_2Na$, 618.9820; found, 618.9807;

TLC R_f = 0.47 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



3-Methyl-5-(phenylethynyl)-4-(3-(prop-2-yn-1-yloxy)butyl)dihydrofuran-2(3H)-one (8). Follows general procedure D: cobalt complex **7** (15 mg, 0.025 mmol, 1 equiv), acetone (3.0 mL), and ceric ammonium nitrate (69 mg, 0.13 mmol, 5 equiv) The reaction stirred for 30 min. The crude residue was purified by silica gel flash column chromatography (15% ethyl acetate in hexanes) to afford 8 mg of alkyne **8** in 97% yield as a clear oil. The product was a 1:1 mixture of diastereomers that were inseparable by column chromatography.

Data for **8**

^{1}H NMR (400 MHz, $CDCl_3$)

7.46-7.43 (m, 2H), 7.36-7.33 (m, 3H), 4.83 (d, *J* = 8.8 Hz, 1H), 4.22 (dd, *J* = 15.6, 2.4 Hz, 0.5 H), 4.21 (dd, *J* = 15.6, 2.4 Hz, 0.5 H)*, 4.122 (dd, *J* = 15.6, 2.4 Hz, 0.5H), 4.115 (dd, *J* = 15.6, 2.4 Hz, 0.5H)*, 3.74-3.66 (m, 1H), 2.394 (t, *J* = 2.4 Hz, 0.5H), 2.387 (t, *J* = 2.4 Hz, 0.5H)*, 2.33-2.31 (m, 2H), 1.92-1.83 (m, 0.5H)*, 1.77-1.67 (m 3.5H), 1.35 (d, *J* = 6.8 Hz, 1.5H), 1.34 (d, *J* = 6.8 Hz, 1.5H)*, 1.19 (d, *J* = 6.0 Hz, 1.5H), 1.17 (d, *J* = 6.0 Hz, 1.5H)* ppm;

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

177.9, 131.93 (2C), 131.91 (2C)*, 129.2, 128.6 (2C), 121.8, 87.9, 85.0, 80.4, 74.1, 73.7, 73.1, 55.8, 55.7*, 51.0, 50.6*, 41.3, 34.2, 19.2, 14.7, 14.5* ppm;

* Discernable signal for one of two diastereomers

IR (thin film)

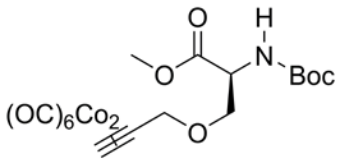
3291, 2924, 2853, 2232, 1780, 1491, 1457, 1166, 1076, 992, 759, 692 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{20}H_{23}O_3$, 311.1642; found, 311.1631;

TLC R_f = 0.50 (30% ethyl acetate in hexanes)

Silica gel, UV active



$Co_2(CO)_6$-*N*-[(1,1-dimethylethoxy)carbonyl]-*O*-(prop-2-yn-1-yl)serine methyl ester (**10a**).

Method A. Follows general procedure C1: $Co_2(CO)_6$-propargyl alcohol **6a** (505 mg, 1.48 mmol), *N*-Boc-L-serine methyl ester **9a** (150 mg, 0.74 mmol)[5], dichloromethane (3 mL), and boron trifluoride diethyl etherate (230 µL, 1.85 mmol) The reaction stirred for 1 h. The crude residue was purified by silica gel flash column chromatography (gradient of 0-25% ethyl acetate in hexanes) to afford 78 mg of **10a** in 20% yield as a dark red/brown oil.

Method B: Follows general procedure C2: Methyl propargyl ether (69.0 mg, 0.984 mmol), dichloromethane (5 mL), dicobalt octacarbonyl (337 mg, 0.984 mmol), *N*-Boc-L-serine methyl ester **9a** (100.0 mg, 0.492 mmol), dissolved in dichloromethane (5 mL), and boron trifluoride diethyl etherate (152 µL, 1.230 mmol). The crude residue was purified by silica gel flash column chromatography (gradient of 10-25% ethyl acetate in hexanes) to afford 252.8 mg of **10a** in 97% yield.

[5] Oh, J.; Lee, K. *Bioorg. Med. Chem.* **1999**, *7*, 2985-2990.

Method C: Follows general procedure C2: Propargyl acetate (96.5 mg, 0.984 mmol), dichloromethane (5 mL), dicobalt octacarbonyl (337 mg, 0.984 mmol), *N*-Boc-L-serine methyl ester **9a** (100.0 mg, 0.492 mmol), dissolved in dichloromethane (5 mL), and boron trifluoride diethyl etherate (150 μL, 1.230 mmol). The crude residue was purified by silica gel flash column chromatography (gradient of 10-25% ethyl acetate in hexanes) to afford 75.7 mg of **10a** in 29% yield.

Data for **10a**

^{1}H NMR (500 MHz, $CDCl_3$):

6.02 (s, 1H), 5.35 (d, *J* = 7.5 Hz, 1H), 4.64 (s, 2H), 4.49 (broad d, *J* = 8.5 Hz, 1H), 4.07 (d, *J* = 8.0 Hz, 1H), 3.85 (d, *J* = 7.0 Hz, 1H), 3.74 (s, 3H), 1.45 (s, 9H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$):

199.3 (6C), 170.8, 155.5, 80.0, 72.0, 71.3, 71.0, 54.0, 52.5, 28.3 (3C), 27.3 ppm;

IR (thin film)

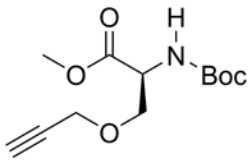
3451, 2978, 2874, 2096, 2054, 2022, 1750, 1718, 1500, 1454, 1438, 1391, 1367, 1347, 1298, 1248, 1207, 1165, 1110, 1062, 1021, 519, 494 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calc'd for $C_{18}H_{19}Co_2NO_{11}Na$ 565.9514 m/z; found 565.9500 m/z;

TLC R_f = 0.50 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



***N*-[(1,1-dimethylethoxy)carbonyl]-*O*-(prop-2-yn-1-yl)serine methyl ester (11a).** Follows general procedure D: cobalt complex **10a** (68 mg, 0.13 mmol, 1 equiv), acetone (5 mL), and

ceric ammonium nitrate (700 mg, 1.28 mmol, 10 equiv) The reaction stirred for 30 min. The crude residue was purified by silica gel flash column chromatography (gradient of 0-30% ethyl acetate in hexanes) to afford 28.3 mg of alkyne **11a** in 90% yield as a clear oil.

Data for **11a**

^{1}H NMR (500 MHz, $CDCl_3$)

5.35 (d, *J* = 8.0 Hz, 1H), 4.45 (dd, *J* = 5.0, 3.0 Hz, 1H), 4.14 (d, *J* = 2.5 Hz, 2H), 4.08-3.85 (m, 1H), 3.77 (s, 3H), 3.76 (m, 1H), 2.44 (t, *J* = 2.5 Hz, 1 H), 1.46 (s, 9H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

170.9, 155.5, 80.0, 78.8, 75.1, 69.7, 58.6, 53.8, 52.5, 28.3 (3C) ppm;

IR (thin film)

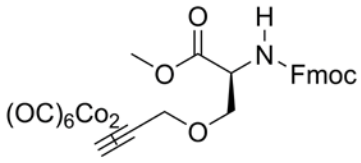
3429, 3297, 2951, 2922, 2851, 2341, 1748, 1717, 1501, 1462, 1440, 1388, 1366, 1300, 1248, 1210, 1165, 1108, 1062, 1024 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calc'd for $C_{12}H_{19}NO_5Na$ 280.1155 m/z; found 280.1148 m/z;

TLC R_f = 0.31 (20% ethyl acetate in hexanes)

Silica gel, potassium permanganate stain



$Co_2(CO)_6$-*N*-[(9H-fluoren-9-yl)methoxy)carbonyl]-*O*-(prop-2-yn-1-yl)serine methyl ester (10b). Method A. Follows general procedure C2: propargyl alcohol (65.7 mg, 1.17 mmol), dichloromethane (10 mL), dicobalt octacarbonyl (400 mg, 1.17 mmol), *N*-Fmoc-L-serine methyl

ester **9b** (200 mg, 0.59 mmol)[6] and boron trifluoride diethyl etherate (200 µL, 1.46 mmol). The reaction stirred for 1 h. The crude residue was purified by silica gel flash column chromatography (gradient of 10-30% ethyl acetate in hexanes) to afford 114 mg of **10b** in 29% yield as a dark red/brown sticky solid.

Method B: Follows general procedure C2: Methyl propargyl ether (24.1 mg, 0.34 mmol), dichloromethane (5 mL), dicobalt octacarbonyl (117.4 mg, 0.34 mmol), *N*-Fmoc-L-serine methyl ester **9b** (58.6 mg, 0.172 mmol), dissolved in dichloromethane (0.4 mL), and boron trifluoride diethyl etherate (50 µL, 0.429 mmol). The crude residue was purified by silica gel flash column chromatography (gradient of 10-50% ethyl acetate in hexanes) to afford 61.9 mg of **10b** in 54% yield.

Method C: Follows general procedure C2: Propargyl acetate (57.4 mg, 0.586 mmol), dichloromethane (5 mL), dicobalt octacarbonyl (200.3 mg, 0.586 mmol), *N*-Fmoc-L-serine methyl ester **9b** (100.0 mg, 0.293 mmol), dissolved in dichloromethane (5 mL), and boron trifluoride diethyl etherate (90 µL, 0.73 mmol). The crude residue was purified by silica gel flash column chromatography (gradient of 10-25% ethyl acetate in hexanes) to afford 45.6 mg of **10b** in 23% yield.

Data for **10b**

^{1}H NMR (500 MHz, $CDCl_3$):

7.77 (d, *J* = 7.5 Hz, 2H), 7.60 (app t, *J* = 6.5 Hz, 2H), 7.41 (app t, *J* = 7.5 Hz, 2H), 7.32 (app t, *J* = 7.5 Hz, 2H), 6.03 (s, 1H), 5.66 (d, *J* = 8.5 Hz, 1H), 4.66 (s, 2H), 4.59 (d, *J* = 8.0 Hz, 1H), 4.45-4.41 (m, 1H), 4.34-4.30 (m, 1H), 4.25-4.23 (m, 1H), 4.13 (d, *J* = 9.0 Hz, 1H), 3.91 (br d, *J* = 3.5 Hz, 1H), 3.77 (s, 3H) ppm;

[6] Zhang, F.; Zhang, W.; Zhang, Y.; Curran, D. P.; Liu, G. *J. Org. Chem.* **2009**, *74*, 2594-2597.

^{13}C NMR (125 MHz, $CDCl_3$)

199.4 (6C), 170.5, 156.0, 143.9, 143.8, 141.3 (2C), 127.7 (2C), 127.07, 127.05, 125.23, 125.15, 120.0 (2C), 90.2, 72.0, 71.2, 70.8, 67.3, 54.4, 52.7, 47.1 ppm;

IR (thin film)

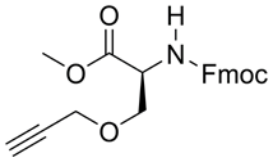
3450, 3069, 3038, 3016, 2954, 2879, 2830, 2095, 2054, 2022, 1728, 1510, 1450, 1338, 1297, 1243, 1208, 1108, 1085, 1059, 760, 741, 621, 519, 495 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calc'd for $C_{28}H_{21}Co_2NO_{11}Na$ 687.9657 m/z; found 687.9671 m/z;

TLC R_f = 0.41 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



***N*-[((9H-fluoren-9-yl)methoxy)carbonyl]-*O*-(prop-2-yn-1-yl)serine methyl ester (11b).**

Follows general procedure D: cobalt complex **10b** (41.1 mg, 0.06 mmol, 1 equiv), acetone (5 mL), and ceric ammonium nitrate (339 mg, 0.62 mmol). The reaction stirred for 30 min. The crude residue was purified by silica gel flash column chromatography (gradient of 0-40% ethyl acetate in hexanes) to afford 21 mg of alkyne **11b** in 90% yield as a clear oil.

Data for **11b**

^{1}H NMR (500 MHz, $CDCl_3$):

7.77 (d, *J* = 7.5 Hz, 2H), 7.62 (app t, *J* = 7.0 Hz, 2H), 7.41 (app t, *J* = 7.5 Hz, 2H), 7.32 (app t, *J* = 7.0 Hz, 2H), 5.66 (d, *J* = 8.0 Hz, 1H), 4.47-4.35 (m, 1H), 4.41 (m, 2H), 4.25 (t, *J* = 7.5 Hz, 1H), 4.17 (s, 2H), 4.01 (dd, *J* = 9.5, 3.0 Hz, 1H), 3.81 (dd, *J* = 9.5, 3.0 Hz, 1H), 3.80 (s, 3H), 2.44 (t, *J* = 2.0 Hz, 1H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$):

170.6, 156.0, 143.9, 143.8, 141.31, 141.30, 127.7 (2C), 127.1 (2C), 125.2, 125.1, 120.0 (2C), 78.8, 75.2, 69.5, 67.2, 58.6, 54.2, 52.7, 47.1 ppm;

IR (thin film)

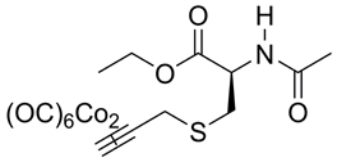
3287, 3060, 3038, 3016, 2952, 2890, 2115, 1745, 1717, 1515, 1476, 1465, 1450, 1341, 1298, 1240, 1210, 1106, 1084, 1059, 1032, 760, 741, 645, 621, 535 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calc'd for $C_{22}H_{21}Co_2NO_5$ 380.1493 m/z; found 380.1485 m/z;

TLC R_f = 0.22 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



Dicobalt octacarbonyl complexed *N*-acetyl-*S*-(prop-2-yn-1-yl)cysteine ethyl ester (10c). Followed general procedure C2: Dicobalt octacarbonyl (107 mg, 0.31 mmol), propargyl alcohol (18 mg, 0.314 mmol) dissolved in dichloromethane (1.5 mL), *N*-acetyl-L-cysteine ethyl ester (**9c**) (30 mg, 0.16 mmol)[7] in dichloromethane (1.5 mL), and borontrifluoride diethyl etherate (49 µL, 0.39 mmol). The reaction was stirred for 2 h. The crude residue was purified by silica gel flash column chromatography (gradient of 10-30% ethyl acetate in hexanes) to yield 70 mg of **10c** in 86% yield, as a red oil.

Data for **10c**

^{1}H NMR (300 MHz, $CDCl_3$)

[7] Aroyan, C. E.; Dermenci, A.; Miller, S. J. *J. Org. Chem* **2010**, *75*, 5784-5796.

6.31 (d, *J* = 4.5 Hz, 1H), 6.14 (s, 1H), 4.87-4.85 (m, 1H), 4.25-4.23 (m, 2H), 4.02-3.91 (m, 2H), 3.19 (dd, *J* = 13.5, 4.4 Hz, 1H), 3.06 (dd, *J* = 13.8, 4.7 Hz, 1H), 2.03 (s, 3H), 1.30 (t, *J* = 6.9 Hz, 3H) ppm;

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

199.5 (6 C), 170.8, 170.0, 92.0, 73.4, 62.2, 52.2, 36.8, 34.7, 23.3, 14.3 ppm;

IR (thin film)

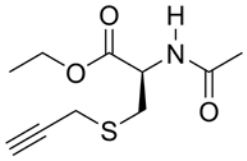
3295, 2984, 2093, 2052, 2020, 1742, 1655, 1543, 1374, 1208, 1032 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calcd for $C_{16}H_{15}O_9NCo_2NaS$, 537.9024; found, 537.9035;

TLC R_f = 0.55 (50% ethyl acetate in hexanes)

Silica gel, visible, UV



***N*-acetyl-*S*-(prop-2-yn-1-yl)cysteine ethyl ester (11c)**. Followed general procedure D: dicobalt hexacarbonyl complexed alkyne **10c** (23 mg, 0.045 mmol), acetone (4.0 mL), and ceric ammonium nitrate (99 mg, 0.18 mmol). The reaction was complete after 10 min of stirring. The work-up afforded 9 mg of alkyne **11c** in 83% yield as a colorless oil. Further purification was not performed.

Data for **11c**

^{1}H NMR (500 MHz, $CDCl_3$)

6.42 (bs, 1H), 4.86 (dt, *J* = 7.5, 5.0 Hz, 1H), 4.25 (q, *J* = 7.0 Hz, 2H), 3.31 (dd, *J* = 17.0, 2.5 Hz, 1H), 3.25-3.21 (m, 2H), 3.14 (dd, *J* = 14.3, 5.3 Hz, 1H), 2.30 (t, *J* = 2.5 Hz, 1H), 2.08 (s, 3H), 1.31 (t, *J* = 7.0 Hz, 3H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

170.9, 170.3, 79.4, 72.1, 62.2, 51.9, 33.9, 23.3, 20.0, 14.3 ppm;

Impurity present at 29.8 ppm

IR (thin film)

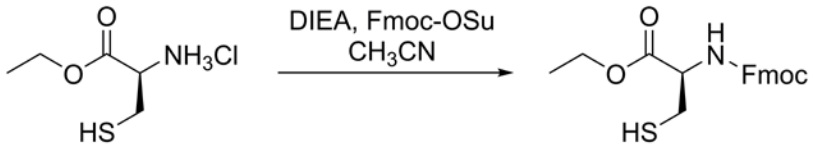
3287, 2919, 2850, 2361, 1739, 1660, 1539, 1374, 1213, 1028 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{10}H_{16}O_3NS$, 230.0845; found, 230.0846;

TLC R_f = 0.23 (50% ethyl acetate in hexanes)

Silica gel, potassium permanganate



9d, 66%

***N*-Fluorenylmoethyloxycarbonyl-L-cysteine ethyl ester (9d)**. A flame-dried, 15 mL, single-necked, round-bottomed flask equipped with a stir bar and a septum, pierced with an inlet needle was charged with L-cysteine ethyl ester hydrochloride salt (100 mg, 0.54 mmol, 1.1 equiv) and acetonitrile (2 mL). The solution was cooled in an ice bath to 0 °C. *N*,*N*-Diisopropylethylamine (35 µL, 0.49 mmol, 1 equiv) was added to the solution followed by *N*-(9-Fluorenyl-methoxycarbonyloxy) succinimide (FMoc-OSu) (165 mg, 0.49 mmol, 1 equiv). The reaction was slowly warmed to rt and stirred overnight (14 h). The solution was quenched by addition of saturated ammonium chloride. The flask contents were transferred to a separatory funnel; the aqueous layer was separated and extracted with ethyl acetate (3 x 10 mL). The combined organics were washed with saturated sodium bicarbonate (15 mL) and brine (15 mL), dried over magnesium sulfate, gravity filtered, and concentrated under reduced pressure rotary evaporation.

The crude material was purified by silica gel flash column chromatography (gradient of 10-20% ethyl acetate in hexanes) to afford 120 mg of **9d** in 66% yield as a white solid.

Data for **9d**

MP 119-121 °C

^{1}H NMR (400 MHz, $CDCl_3$)

7.77 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J* = 7.2 Hz, 2H), 7.41 (app t, *J* = 7.4 Hz, 2H), 7.34 (app tt, *J* = 7.4, 1.4 Hz, 2H), 5.69 (d, *J* = 7.2 Hz, 1H), 4.66-4.63 (m, 1H), 4.47-4.40 (m, 2H), 4.30-4.23 (m, 3H), 3.03-3.00 (m, 2H), 1.36 (t, *J* = 8.8 Hz, 1H), 1.31 (t, *J* = 7.2 Hz, 3H) ppm;

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

170.1, 155.8, 144.0, 143.8, 141.49, 141.46, 127.9 (2 C), 127.2 (2 C), 125.24, 125.19, 120.18, 120.16, 67.2, 62.2, 55.3, 47.3, 27.3, 14.4 ppm;

IR (thin film)

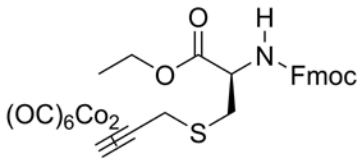
3337, 3065, 2981, 1723, 1513, 1450, 1339, 1204, 1035, 759, 741 cm^{-1}.

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{20}H_{22}O_4NS$, 372.1264; found, 372.1267;

TLC R_f = 0.28 (20% ethyl acetate in hexanes)

Silica gel, UV



Dicobalt hexacarbonyl complexed *N*-(((9H-fluoren-9-yl)methoxy)carbonyl)-S-(prop-2-yn-1-yl)-L-cysteine ethyl ester (10d). Method A: Followed general procedure C2: Dicobalt octacarbonyl (55 mg, 0.16 mmol), propargyl alcohol (9 mg, 0.16 mmol), dichloromethane (1.1

mL), *N*-Fmoc-L-cysteine ethyl ester (**9d**) (30 mg, 0.081 mmol) dissolved in dichloromethane (0.6 mL), and boron trifluoride diethyl etherate (25 µL, 0.20 mmol). The reaction was stirred for 45 min. The crude residue was purified by silica gel flash column chromatography (gradient of 5-20% ethyl acetate in hexanes) to afford 40 mg of **10d** in 71% yield, as a red oil.

Method B: Followed general procedure C1: $Co_2(CO)_6$-methyl propargyl ether **6b** (45 mg, 0.13 mmol), dichloromethane (0.6 mL), *N*-Fmoc-L-cysteine ethyl ester (**9d**) (24 mg, 0.063 mmol) dissolved in dichloromethane (0.6 mL), and boron trifluoride diethyl etherate (20 µL, 0.156 mmol). The reaction was stirred for 2 h. The crude residue was purified by silica gel flash column chromatography (gradient of 10-20% diethyl ether in hexanes) to afford 29 mg of **10d** in 67% yield.

Data for **10d**

^{1}H NMR (400 MHz, $CDCl_3$)

7.77 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 7.6 Hz, 2H), 7.41 (app t, *J* = 7.6 Hz, 2H), 7.32 (app t, *J* = 7.6 Hz, 2H), 6.13 (s, 1H), 5.66 (d, *J* = 7.6 Hz, 1H), 4.66 (dt, *J* = 7.6, 5.0 Hz, 1H), 4.44-4.35 (m, 2H), 4.28-4.22 (m, 3H), 3.99 (s, 2H), 3.19 (dd, *J* = 14.0, 4.8 Hz, 1H), 3.09 (dd, *J* = 14.0, 5.2 Hz, 1H), 1.32 (t, *J* = 7.2 Hz, 3H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

199.5 (6 C), 170.7, 155.9, 143.93, 143.86, 141.5 (2 C), 127.9 (2 C), 127.2 (2 C), 125.2 (2 C), 120.2 (2 C), 92.1, 73.4, 67.5, 62.3, 53.9, 47.3, 36.9, 35.1, 14.3 ppm;

IR (thin film)

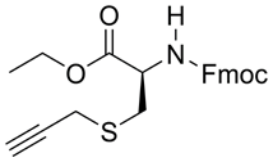
3345, 3070, 2923, 2094, 2053, 2023, 1726, 1507, 1450, 1339, 1204, 1052, 759, 741 cm^{-1}.

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{29}H_{24}O_{10}NCo_2S$, 695.9779; found, 695.9745;

TLC R_f = 0.36 (20% ethyl acetate in hexanes)

Silica gel, visibile, UV



Ethyl *N*-(((9H-fluoren-9-yl)methoxy)carbonyl)-S-(prop-2-yn-1-yl)-L-cysteinate (11d).

Followed general procedure D: Dicobalt hexacarbonyl complexed alkyne **10d** (22 mg, 0.032 mmol), acetone (3.5 mL), and ceric ammonium nitrate (69 mg, 0.13 mmol). The reaction was complete after 10 min of stirring. The work-up afforded 12 mg of alkyne **11d** in 92% yield as an off white oil. Further purification was not performed.

Data for **11d**

^{1}H NMR (400 MHz, $CDCl_3$)

7.77 (d, *J* = 7.4 Hz, 2H), 7.61 (d, *J* = 7.4 Hz, 2H), 7.41 (app t, *J* = 7.4 Hz, 2H), 7.32 (app tt, *J* = 7.4, 1.2 Hz, 2H), 5.64 (d, *J* = 7.6 Hz, 1H), 4.65 (dt, *J* = 8.0, 5.2 Hz, 1H), 4.46-4.38 (m, 2H), 4.28-4.23 (m, 3H), 3.32-3.22 (m, 2H), 3.22 (dd, *J* = 14.4, 4.8 Hz, 1H), 3.14 (dd, *J* = 14.0, 5.6 Hz, 1H), 2.26 (t, *J* = 2.6 Hz, 1H), 1.31 (t, *J* = 7.2 Hz, 3H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

170.8, 155.9, 144.0, 143.9, 141.5 (2 C), 127.9 (2 C), 127.2 (2 C), 125.2 (2 C), 120.2 (2 C), 79.4, 72.1, 67.3, 62.2, 53.7, 47.3, 34.0, 20.1, 14.3 ppm;

Impurity observed at 29.8 ppm.

IR (thin film)

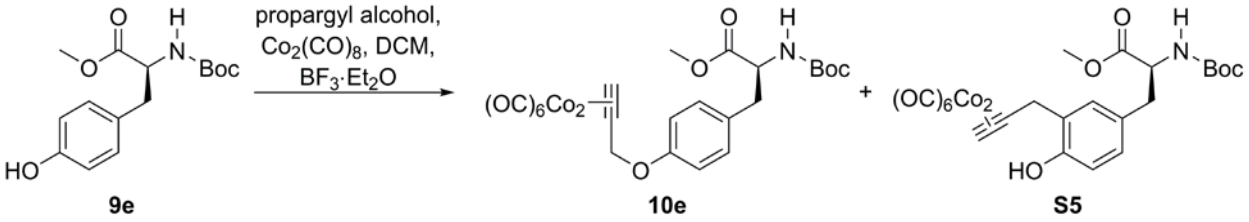
3291, 2924, 1723, 1517, 1450, 1339, 1210, 1051, 760, 741 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{23}H_{24}O_4NS$, 410.1421; found, 410.1424;

TLC R_f = 0.26 (20% ethyl acetate in hexanes)

Silica gel, UV



$Co_2(CO)_6$-*N*-[(1,1-dimethylethoxy)carbonyl]-*O*-(prop-2-yn-1-yl)-L-tyrosine methyl ester (10e). Method A: Follows general procedure C2: propargyl alcohol (75.9 mg, 1.35 mmol), dichloromethane (5 mL), dicobalt octacarbonyl (510 mg, 1.49 mmol), *N*-Boc-L-tyrosine methyl ester **9e** (200 mg, 0.68 mmol)[8] and boron trifluoride diethyl etherate (210 µL, 1.70 mmol). The reaction stirred for 1 h. Both **10e** and **S5** were observed by TLC. The crude residue was purified by silica gel flash column chromatography (gradient of 10-40% ethyl acetate in hexanes) to afford 187 mg of **10e** in 45% yield as a dark red/brown oil and no **S5** was isolated.

In a separate experiment, **10e** was afforded in 32% yield and byproduct **S5** was isolated in trace amounts. The ^{1}H NMR of this sample of **S5** is included in the spectra section. Further characterization was not obtained due to small amounts.

Method B: Follows general procedure C2: methyl propargyl ether (19 mg, 0.27 mmol), dichloromethane (2.0 mL), dicobalt octacarbonyl (93 mg, 0.27 mmol), *N*-Boc-L-tyrosine methyl ester **9e** (40.0 mg, 0.135 mmol) and boron trifluoride diethyl etherate (42 µL, 0.338 mmol). The

[8] Bulman Page, P.C.; Buckley, B. R.; Farah, M. M.; Blacker, A. J. *Eur. J. Org. Chem*. **2009**, 3413-3426.

reaction stirred for 3 h. Both **10e** and **S5** were observed by TLC. The crude residue was purified by silica gel flash column chromatography (gradient of 15-30% ethyl acetate in hexanes) to afford 19 mg of **10e** in 23% yield as a dark red/brown oil. Large amounts of baseline material was observed.

Data for **10e**

^{1}H NMR (500 MHz, $CDCl_3$):

7.05 (d, *J* = 7.0 Hz, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.05 (s, 1H), 5.15 (s, 2H), 4.95 (bs, 1H), 4.55 (bs, 1H), 3.72 (s, 3H), 3.03 (m, 2H), 1.42 (s, 9H) ppm

^{13}C NMR (125 MHz, $CDCl_3$):

199.3 (6 C), 172.4, 157.3, 155.1, 130.4 (2C), 128.6, 114.7 (2C), 89.5, 79.9, 71.7, 68.2, 54.5, 52.2, 37.5, 28.3 (3 C) ppm

Impurities seen at 129.7, 124.2, 28.8, 26.7

IR (thin film)

3445, 3368, 2979, 2956, 2929, 2097, 2056, 1746, 1716, 1612, 1585, 1510, 1445, 1392, 1367, 1244, 1216, 1172, 1111, 1059, 1018, 839, 779, 519, 497 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calc'd for $C_{24}H_{23}Co_2NO_{10}Na$ 625.9878 m/z; found 625.9877 m/z;

TLC R_f = 0.44 (20% ethyl acetate in hexanes)

Silica gel, visible, UV

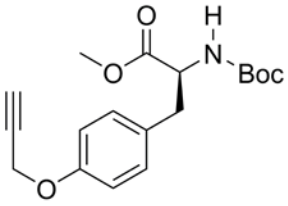
Data for **S5**

^{1}H NMR (400 MHz, $CDCl_3$)

6.95 (s, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.70-6.62 (m, 1H), 6.07 (s, 1H), 4.91-4.87 (m, 1 H), 4.79-4.71 (m, 1H), 4.58-4.49 (m, 1H), 4.10 (s, 1H), 3.72 (s, 3H), 3.08-2.92 (m, 2H), 1.43 (s, 9H) ppm;

TLC R_f = 0.26 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



***N*-[(1,1-dimethylethoxy)carbonyl]-*O*-(prop-2-yn-1-yl)-L-tyrosine methyl ester (11e).** Follows general procedure D: cobalt complex **10e** (186 mg, 0.31 mmol, 1 equiv), acetone (10 mL), and ceric ammonium nitrate (1.7 g, 3.1 mmol, 10 equiv) The reaction stirred for 30 min. The crude residue was purified by silica gel flash column chromatography (gradient of 0-50% ethyl acetate in hexanes) to afford 73.4 mg of alkyne **11e** in 75% yield as a clear oil.

Data for **11e**

^{1}H NMR (500 MHz, $CDCl_3$):

7.05 (d, *J* = 8.5 Hz, 2H), 6.90 (d, *J* = 8.5 Hz, 2H), 4.96 (br d, *J* = 8.0 Hz, 1H), 4.66 (d, *J* = 2.5 Hz, 2H), 4.59-4.49 (m, 1H), 3.71 (s, 3H), 3.08-2.97 (m, 2H), 2.51 (t, *J* = 2.0 Hz, 1H), 1.41 (s, 9H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$):

172.4, 156.7, 155.1, 130.3 (2C), 129.0, 115.0 (2C), 79.9, 78.6, 75.5, 55.8, 54.5, 52.2, 37.5, 28.3 (3C) ppm;

Impurities seen at 61.3, 37.7, 36.2, 19.6, 19.4, 14.1

IR (thin film)

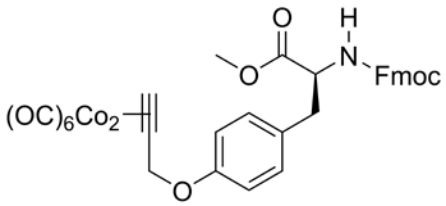
3368, 3288, 2977, 2956, 2929, 2868, 2121, 1743, 1712, 1611, 1586, 1506, 1438, 1392, 1366, 1218, 1165, 1113, 1058, 1027, 924, 825, 644 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calc'd for $C_{18}H_{24}NO_5$ 334.1649 m/z; found 334.1640 m/z

TLC R_f = 0.27 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



$Co_2(CO)_6$-*N*-[(9H-fluoren-9-yl)methoxy)carbonyl]-*O*-(prop-2-yn-1-yl)-L-tyrosine methyl ester (10f). Method A. Follows general procedure C2: propargyl alcohol (27.0 mg, 0.48 mmol), dichloromethane (5 mL), dicobalt octacarbonyl (164 mg, 0.48 mmol, 2 equiv), *N*-Fmoc-L-tyrosine methyl ester **9f** (100 mg, 0.24 mmol)[9] and boron trifluoride diethyl etherate (74 µL, 0.60 mmol). The reaction stirred for 1 h. The crude residue was purified by silica gel flash column chromatography (gradient of 10-40% ethyl acetate in hexanes) to afford 22.3 mg of **10f** in 6% yield as a dark red/brown sticky solid.

Method B: Follows general procedure C2: Methyl propargyl ether (16.8 mg, 0.240 mmol), dichloromethane (5 mL), dicobalt octacarbonyl (82.1 mg, 0.240 mmol), *N*-Fmoc-L-tyrosine methyl ester **9f** (50.0 mg, 0.120 mmol), dissolved in dichloromethane (5 mL), and boron trifluoride diethyl etherate (40 µL, 0.300 mmol). The crude residue was purified by silica gel flash column chromatography (gradient of 10-50% ethyl acetate in hexanes) to afford 64.9 mg of **10f** in 73% yield.

[9] Adamson, J.G.; Blaskovich, M.A.; Groenevelt, H.; Lajoie, G.A. *J. Am. Chem. Soc*. **1990**, *56*, 10, 3447-3449.

Data for **10f**

^{1}H NMR (500 MHz, $CDCl_3$):

7.78 (d, *J* = 7.5 Hz, 2H), 7.58 (s, 2H), 7.40-7.39 (m, 2H), 7.33-7.32 (m, 2H), 7.01 (d, *J* = 7.0 Hz, 2H), 6.87 (d, *J* = 7.5 Hz, 2H), 6.05 (s, 1H), 5.23 (d, *J* = 7.5 Hz, 1H), 5.15 (s, 2H), 4.65 (bs, 1H), 4.48-4.34 (m, 2H), 4.22 (bs, 1H), 3.75 (s, 3H), 3.08 (m, 2H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$):

199.3 (6C), 171.9, 157.3, 155.5, 143.9, 143.8, 141.4 (2C), 130.4 (2C), 128.3, 127.7 (2C), 127.1 (2C), 125.1, 125.0, 120.0 (2C), 114.8 (2C), 89.4, 71.7, 68.2, 66.9, 54.9, 52.4, 47.2, 37.3 ppm;

IR (thin film)

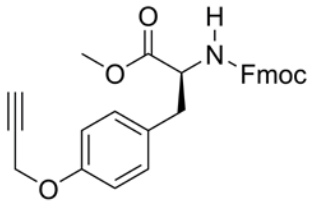
3429, 3066, 3033, 2951, 2923, 2896, 2852, 2096, 2054, 2022, 1724, 1610, 1583, 1510, 1449, 1347, 1242, 1213, 1177, 1033, 757, 741, 518, 494 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calc'd for $C_{34}H_{25}Co_2NO_{11}$ 742.0164 m/z; found 742.0170 m/z;

TLC R_f = 0.50 (30% ethyl acetate in hexanes)

Silica gel, visible, UV



***N*-[((9H-fluoren-9-yl)methoxy)carbonyl]-*O*-(prop-2-yn-1-yl)-L-tyrosine methyl ester (11f).**

Follows general procedure D: cobalt complex **10f** (46.4 mg, 0.063 mmol), acetone (2 mL), and ceric ammonium nitrate (34.3 mg, 0.630 mmol). The reaction stirred for 30 min. The crude

residue was purified by silica gel flash column chromatography (gradient of 0-50% ethyl acetate in hexanes) to afford 23.0 mg of alkyne **11f** in 81% yield as a white solid.

Data for **11f**

MP 94-95 °C

^{1}H NMR (500 MHz, $CDCl_3$):

7.77 (d, *J* = 7.5 Hz, 2H), 7.57 (app t, *J* = 7.0 Hz, 2H), 7.41 (app t, *J* = 7.0 Hz, 2H), 7.32 (app t, *J* = 7.0 Hz, 2H), 7.01 (d, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 8.0, 2H), 5.24 (d, *J* = 8.5 Hz, 1H), 4.66 (s, 2H), 4.46 (dd, *J* =11.0, 7.0 Hz, 1H), 4.37-4.34 (m, 1H), 4.21 (t, *J* = 6.5, 1H), 3.74 (s, 3H), 3.12-3.03 (m, 2H), 2.50 (s, 1H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$):

171.9, 156.8, 155.5, 143.9 (2C), 141.3 (2C), 130.4 (2C), 128.6, 127.7 (2C), 127.1 (2C), 125.1 (2C), 120.0 (2C), 115.0 (2C), 78.2, 75.5, 66.9, 55.8, 54.8, 52.4, 47.2, 37.4 ppm;

IR (thin film)

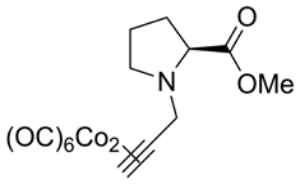
3286, 2956, 2923, 2852, 2049, 2022, 1723, 1608, 1511, 1462, 1449, 1377, 1344, 1259, 1240, 1218, 1177, 1108, 1051, 1026, 757, 741 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calc'd for $C_{28}H_{25}NO_5$ 456.1806 m/z; found 456.1793 m/z;

TLC R_f = 0.23 (30% ethyl acetate in hexanes)

Silica gel, visible, UV



$Co_2(CO)_6$-*N*-propargyl-L-proline methyl ester (10g). Follows general procedure C3: Tetrafluoroborate salt **6c** (140 mg, 0.340 mmol), dichloromethane (5 mL), L-proline methyl ester **9g** (57 mg, 0.442 mmol) dissolved in dichloromethane (1.8 mL). The reaction stirred for 1.5 h. The crude residue was purified by silica gel flash column chromatography (gradient of 5-10% diethyl ether in hexanes) to afford 75 mg of cobalt complexed alkyne **10g** in 46% yield as a red oil.

Data for **10g**

^{1}H NMR (300 MHz, $CDCl_3$)

6.05 (s, 1H), 4.23 (d, *J* = 15.6 Hz, 1H), 3.97 (d, *J* = 15.3 Hz, 1H), 3.71 (s, 3H), 3.53 (dd, *J* = 8.1, 5.1 Hz, 1H), 3.23-3.14 (m, 1H), 2.71 (q, *J* = 8.1 Hz, 1H), 2.13, 1.78 (m, 4H) ppm;

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

199.9 (6C), 174.3, 91.8, 73.4, 64.2, 56.1, 52.8, 51.9, 29.4, 23.5 ppm;

IR (thin film)

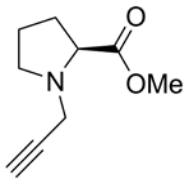
2955, 2798, 2093, 2020, 1736, 1551, 1437, 1356, 1278, 1199, 1173 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{15}H_{14}O_8NCo_2$, 453.9378; found, 453.9361;

TLC R_f = 0.37 (20% diethyl ether in hexanes)

Silica gel, UV, potassium permanganate



***N*-propargyl-L-proline methyl ester (11g).** Followed general procedure D: Dicobalt hexacarbonyl complexed alkyne **10g** (20 mg, 0.044 mmol), acetone (5.0 mL), and ceric ammonium nitrate (97 mg, 0.18 mmol). After 1 h of stirring, **10g** remained, as evidenced by proton NMR. An additional amount of ceric ammonium nitrate (10 mg, 0.018 mmol) was added and stirred for 20 min. The work-up afforded 5 mg of alkyne **11g** in 68% yield as a colorless oil. Further purification was not performed. Characterization via ^{1}H NMR, ^{13}C NMR, and HRMS was obtained, however, **11g** appears to be unstable leading to decomposition and poor reproducibility of these data.

Data for **11g**

^{1}H NMR (400 MHz, $CDCl_3$)

3.74 (s, 3H), 3.61 (app t, *J* = 2.4 Hz, 2H), 3.45 (dd, *J* = 8.8, 6.8 Hz, 1H), 3.09-3.04 (m, 1H), 2.73 (td, *J* = 8.8, 7.6 Hz, 1H), 2.21 (t, *J* = 2.4 Hz, 1H), 2.19-2.11 (m, 1H), 2.03-1.76 (m, 3H) ppm;

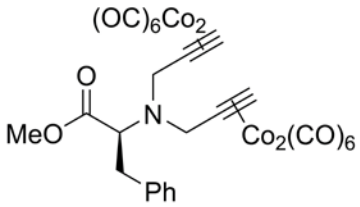
^{13}C NMR (100 MHz, $CDCl_3$)

174.3, 78.5, 73.3, 62.7, 52.3, 52.2, 41.3, 29.8, 23.4 ppm;

Impurity present at 30.5 ppm

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_9H_{14}O_2N$, 168.1019; found, 168.1013;



Bis(dicobalthexacarbonyl) complexed *N*,*N*-di(prop-2-ynyl)-L-phenylalanine methyl ester (**10h**). Follows general procedure C3: Tetrafluoroborate salt **6c** (46 mg, 0.11 mmol, 1 equiv), dichloromethane (2 mL), L-phenyl alanine methyl ester **9h** (20 mg, 0.11 mmol, 1 equiv) dissolved in dichloromethane (0.3 mL). The reaction stirred for 1.5 h. The crude residue was purified by silica gel flash column chromatography (gradient of 2-20% diethyl either in hexanes) to afford 46 mg of cobalt complexed dialkyne **10h** in 59% yield as a red oil. The yield was calculated using tetrafluoroborate salt **6c** as the limiting reagent.

Data for **10h**

^{1}H NMR (300 MHz, $CDCl_3$)

7.31-7.22 (m, 3H), 7.17 (d, *J* = 7.2 Hz, 2H), 6.10 (s, 2H), 4.41 (d, *J* = 15.9 Hz, 2H), 4.04-3.99 (m, 3H), 3.56 (s, 3H), 3.24-3.17 (m, 1H), 2.93 (dd, *J* = 13.0, 4.2 Hz, 1H) ppm;

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

199.7 (12C), 172.0, 137.3, 129.3 (2C), 128.7 (2C), 126.9, 91.1 (2C), 73.8 (2C), 64.4, 55.1 (2C), 51.3, 36.5 ppm;

IR (thin film)

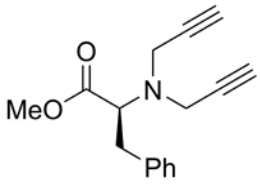
2093, 3052, 2017, 1735, 1425, 1200, 1165 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{28}H_{18}O_{14}NCo_4$, 827.8050; found, 827.8084;

TLC R_f = 0.48 (10% diethyl ether in hexanes)

Silica gel, UV, visible



***N,N*-di(prop-2-ynyl)-L-phenylalanine methyl ester (11h).** Followed general procedure D: Dicobalt hexacarbonyl complexed dialkyne **10h** (21 mg, 0.025 mmol, 1 equiv), acetone (4.0 mL), and ceric ammonium nitrate (111 mg, 0.20 mmol, 8 equiv). The reaction was complete after 20 min of stirring. The crude residue was purified using silica gel flash column chromatography (gradient of 15-30% diethyl ether in hexanes), which afforded 4 mg of alkyne **11h** in 56% yield as an oil.

Data for **11h**

^{1}H NMR (400 MHz, $CDCl_3$)

7.30-7.26 (m, 2H), 7.23-7.18 (m, 3H), 3.75 (t, *J* = 7.6 Hz, 1H), 3.68 (d, *J* = 2.4 Hz, 4H), 3.57 (s, 3H), 3.05 (d, *J* = 7.6 Hz, 2H), 2.25 (t, *J* = 2.4 Hz, 2H) ppm;

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

172.1, 137.5, 129.3 (2 C), 128.6 (2 C), 126.8, 79.3 (2 C), 73.1 (2 C), 65.9, 51.5, 40.1 (2 C), 36.5 ppm;

IR (thin film)

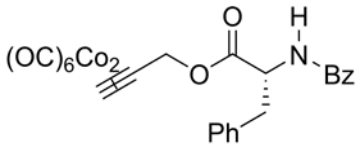
3250, 2991, 2914, 2813, 2344, 1714, 1478, 1421, 1347, 1199, 1153, 1112, 740, 692, 623 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{16}H_{18}O_2N$, 256.1332; found, 256.1335;

TLC R_f = 0.37 (20% diethyl ether in hexanes)

Silica gel, UV, potassium permanganate



Dicobalt hexacarbonyl prop-2-yn-1-yl benzoylphenylalaninate complex (10i). Follows general procedure C2: propargyl alcohol (11 mg, 0.20 mmol), dichloromethane (1.1 mL), dicobalt octacarbonyl (69 mg, 0.20 mmol), *N*-benzoyl-D-phenylalanine (**9i**) (27 mg, 0.10 mmol, 1 equiv), dissolved in dichloromethane (1.0 mL), and boron trifluoride diethyl etherate (32 µL, 0.26 mmol). The reaction stirred for 2 h. The crude residue was purified by silica gel flash column chromatography (10% ethyl acetate in hexanes) to afford 35 mg of **10i** in 60% yield as a dark red oil.

Data for **10i**

^{1}H NMR (400 MHz, $CDCl_3$)

7.72-7.70 (m, 1H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.31-7.28 (m, 3H), 7.17-7.15 (m, 2H), 6.55 (d, *J* = 7.2 Hz, 1H), 6.08 (s, 2H), 5.44 (d, *J* = 14.4 Hz, 1H), 5.24 (d, *J* = 14.0 Hz, 1H), 5.18 (dt, *J* = 7.6, 5.6 Hz, 1H), 3.36 (dd, *J* = 14.0, 5.6 Hz, 1H), 3.27 (dd, *J* = 14.0, 6.0 Hz, 1H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

199.1 (6C), 171.5, 166.9, 135.9, 134.0, 131.9, 129.5 (2C), 128.9 (2C), 128.8 (2C), 127.5, 127.2 (2C), 87.2, 72.4, 66.8, 53.8, 38.0 ppm;

IR (thin film)

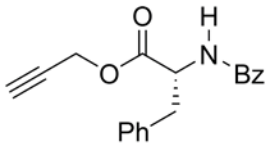
3031, 2925, 2097, 2056, 2025, 1746, 1647, 1531, 1487, 1178, 700 cm^{-1}.

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calcd for $C_{25}H_{17}O_9NCo_2$, 615.9460; found, 615.9453;

TLC R_f = 0.34 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



Prop-2-yn-1-yl benzoylphenylalaninate (11i). Followed general procedure D: Dicobalt hexacarbonyl complexed alkyne **10i** (20 mg, 0.034 mmol), acetone (2.5 mL), and ceric ammonium nitrate (75 mg, 0.138 mmol). The reaction was complete after 10 min of stirring. Reaction work up afforded 10 mg of pure alkyne **11i** as a white sticky solid in 90% yield. Purification by silica gel column was not performed.

Data for **11i**

^{1}H NMR (400 MHz, $CDCl_3$)

7.72-7.70 (m, 2H), 7.53-7.47 (m, 1H), 7.45-7.41 (m, 2H), 7.32-7.26 (m, 3H), 7.19-7.17 (m, 2H), 6.53 (d, *J* = 7.6 Hz, 1H), 5.14 (dt, *J* = 7.6, 5.6 Hz, 1H), 4.82 (dd, *J* = 15.6, 2.6 Hz, 1H), 4.73 (dd, *J* = 15.6, 2.6 Hz, 1H), 3.33 (dd, *J* = 13.8, 5.8 Hz, 1H), 3.27 (dd, *J* = 13.8, 5.4 Hz, 1H), 2.54 (t, *J* = 2.4 Hz, 1H) ppm;

Impurities seen at 1.43, 1.25, 0.88 ppm

^{13}C NMR (125 MHz, $CDCl_3$)

171.0, 167.0, 135.7, 134.0, 132.0, 129.6 (2C), 128.83 (2C), 128.80 (2C), 127.5, 127.2 (2C), 77.1, 75.8, 53.5, 53.0, 37.9 ppm;

Impurity seen at 29.8 ppm.

IR (thin film)

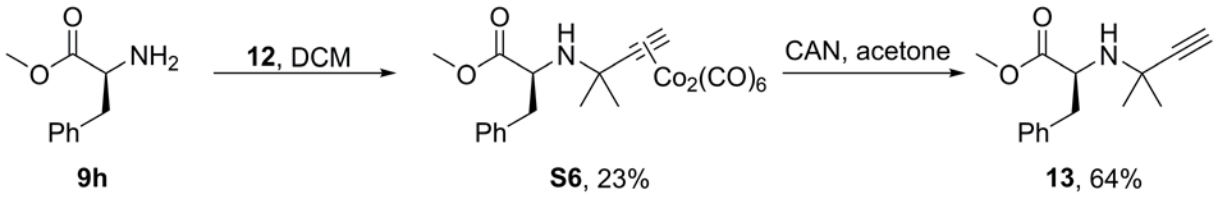
3396, 3277, 3070, 2920, 2851, 2131, 1762, 1647, 1521, 1488, 1205, 1171 cm^{-1}.

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{19}H_{17}O_3N$, 308.1281; found, 308.1282;

TLC R_f = 0.21 (20% ethyl acetate in hexanes)

Silica gel, UV



9h **S6**, 23% **13**, 64%

$Co_2(CO)_6$-*N*-(1,1-dimethyl-3-propynyl)-L-phenylalanine methyl ester (S6). Follows general procedure C3: Tetrafluoroborate salt **12** (77 mg, 0.18 mmol. 1.3 equiv), dichloromethane (2.5 mL), L-phenylalanine methyl ester **9h** (24 mg, 0.14 mmol, 1 equiv) dissolved in dichloromethane (0.5 mL). The reaction stirred for 40 min. The crude residue was purified by silica gel flash column chromatography (gradient of 5-20% diethyl either in hexanes) to afford 16 mg of cobalt complexed alkyne **S6** in 23% yield as a red oil.

Data for **S6**

^{1}H NMR (400 MHz, $CDCl_3$)

7.29-7.17 (m, 5 H), 5.99 (s, 1H), 3.72 (dt, *J* = 7.6, 3.2 Hz, 1H), 3.62 (s, 3H), 2.92-2.81 (m, 2H), 1.90 (d, *J* = 8.4 Hz, 1H), 1.29 (s, 3H), 1.20 (s, 3H) ppm;

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

200.1 (6C), 176.6, 137.6, 129.6 (2C), 128.4 (2C), 126.8, 107.3, 72.2, 58.0, 56.7, 52.0, 41.9, 32.1, 30.9 ppm;

IR (thin film)

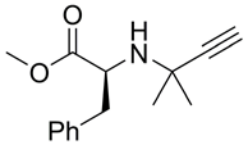
2973, 2927, 2092, 2050, 2019, 1739, 1455, 1194, 1172, 700 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{21}H_{20}O_8NCo_2$, 531.9847; found, 531.9848;

TLC R_f = 0.35 (10% diethyl ether in hexanes)

Silica gel, Visible, UV



***N*-(1,1-dimethyl-3-propynyl)-L-phenylalanine methyl ester (13).** Followed general procedure D: $Co_2(CO)_6$-alkyne **S6** (12 mg, 0.023 mmol), acetone (4.0 mL), and ceric ammonium nitrate (50 mg, 0.090 mmol). The reaction was complete after 20 min of stirring as indicated by consumption of **S6**, as evidenced by TLC. However, **13** was not visible by TLC until after the reaction work up. The crude residue was purified by silica gel flash column chromatography (10% diethyl ether in hexanes) which afforded 4 mg of alkyne **13** in 64% yield as an colorless oil.

Data for **13**

^{1}H NMR (400 MHz, $CDCl_3$)

7.30-7.26 (m, 2H), 7.23-7.19 (m, 3H), 3.73 (dd, *J* = 7.8, 6.4 Hz, 1H), 3.63 (s, 3H), 2.93 (dd, *J* =13.4, 6.4 Hz, 1H), 2.84 (dd, *J* = 13.4, 7.8 Hz, 1H), 2.18 (s, 1H), 1.92 (bs, 1H), 1.31(s, 3H), 1.19 (s, 3H) ppm;

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

176.3, 137.5, 129.6 (2C), 128.4 (2C), 126.8, 88.5, 70.1, 58.9, 51.7, 49.6, 41.4, 30.3, 29.6 ppm;

IR (thin film)

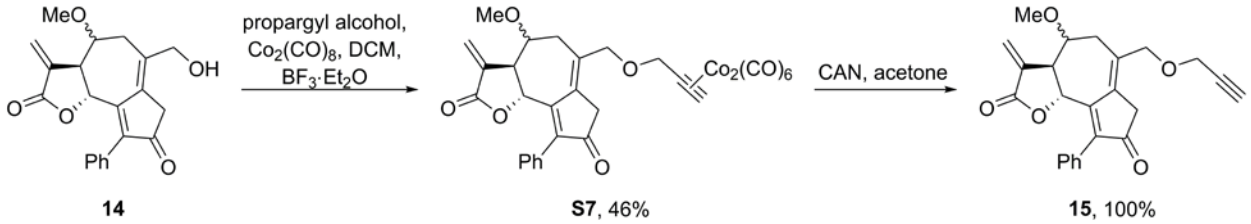
3286, 3027, 2977, 2929, 2368, 1736, 1458, 1438, 1196, 1171, 700 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{15}H_{20}O_2N$, 246.1489; found, 246.1478;

TLC R_f = 0.33 (20% diethyl ether in hexanes)

Silica gel, UV, potassium permanganate



14 **S7**, 46% **15**, 100%

Dicobalthexacarbonyl complexed 4-methoxy-3-methylene-9-phenyl-6-((prop-2-yn-1-yloxy)methyl)-3a,5,7,9b-tetrahydroazuleno[4,5-b]furan-2,8(3H,4H)-dione (S7). Follows general procedure C2: propargyl alcohol (5 mg, 0.091 mmol), dichloromethane (0.60 mL), dicobalt octacarbonyl (31 mg, 0.091 mmol), alcohol **14**[10] (16 mg, 0.045 mmol), dissolved in dichloromethane (0.40 mL), and boron trifluoride diethyl etherate (15 µL, 0.11 mmol). The reaction was monitored by TLC and stirred for 2 h. The crude residue was purified by silica gel flash column chromatography (gradient of 10-30% ethyl acetate in hexanes) to afford 14 mg of **S7** as a mixture of two diastereomers (2.1:1) in 46% yield as a dark red/brown oil.

Data for **S7**

^{1}H NMR (400 MHz, $CDCl_3$)

7.40-7.35 (m, 3 H), 7.31-7.26 (m, 2 H), 6.34 (d, *J* = 3.6 Hz, 1 H)**, 6.24 (d, *J* = 3.2 Hz, 1 H)*, 6.09 (s, 1 H), 5.86 (d, *J* = 3.2 Hz, 1 H)*, 5.78 (d, *J* = 9.6 Hz, 1 H)**, 5.47 (d, *J* = 3.2 Hz, 1 H)**, 5.38 (d, *J* = 10.4 Hz, 1 H)*, 4.671-4.61 (m, 2H), 4.33 (d, *J* = 12.4 Hz, 1 H)*, 4.25 (d, *J* = 12.8 Hz, 1 H)*, 4.26 (app s, 2 H)**, 4.10-4.05 (m, 1 H)**, 3.88-3.86 (m, 1 H)*, 3.51 (s, 3 H)*, 3.39 (s, 3H)**, 3.32-3.14 (m, 4 H), 2.52-2.41 (m, 1 H) ppm;

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

[10] Alcohol **14** was synthesized according to procedures previously reported and was used as a mixture of two diastereomers (2.4:1): *Org. Lett.* **2013**, 15, 2644-2647.

201.4*, 201.1**, 199.6 (6C) 168.3**, 167.9*, 162.2**, 161.5*, 144.0**, 143.0*, 137.2*, 135.0**, 134.4*, 133.6**, 131.6*, 130.72**, 130.65*, 130.3**, 130.0*, 129.9**, 128.84**, 128.77*, 127.8**, 127.7*, 122.8**, 122.2*, 90.7**, 90.6*, 81.8*, 75.7*, 74.7**, 73.6**, 73.4*, 73.3**, 71.9*, 71.6**, 71.4**, 71.1*, 57.2**, 56.5*, 49.8*, 40.0**, 39.7*, 33.8**, 29.8**, 29.5* ppm;

IR (thin film)

2927, 2829, 2372, 2093, 2051, 2022, 1773, 1702, 12.68, 1096, 1018, 697 cm^{-1}.

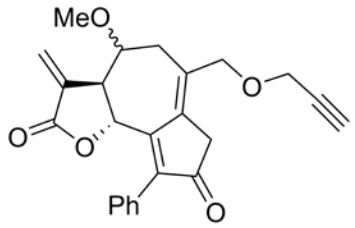
HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{30}H_{23}O_{11}Co_2$, 676.9899; found, 676.9903;

TLC R_f = 0.26*, 0.22** (30% ethyl acetate/hexanes)

Silica gel, Visible, UV

*Major diastereomer, **minor diastereomer



4-Methoxy-3-methylene-9-phenyl-6-((prop-2-yn-1-yloxy)methyl)-3a,5,7,9b-tetrahydroazuleno[4,5-b]furan-2,8(3H,4H)-dione (15). Follows general procedure D: cobalt complex **S7** (14 mg, 0.021 mmol, 1 equiv), acetone (1.5 mL), and ceric ammonium nitrate (68 mg, 0.12 mmol, 6 equiv). The reaction stirred for 15 min. The reaction afforded 9 mg of alkyne **15** as a mixture of two diastereomers (2.1:1) in quantitative yield as a colorless oil. The crude material was not purified further.

Data for **15**

^{1}H NMR (400 MHz, $CDCl_3$)

7.38-7.35 (m, 3 H), 7.30-7.24 (m, 2 H), 6.36 (d, *J* = 3.2 Hz, 1 H)**, 6.24 (d, *J* = 3.2 Hz, 1 H)*, 5.88 (d, *J* = 2.8 Hz, 1 H)*, 5.78 (d, *J* = 9.6 Hz, 1 H)**, 5.52 (d, *J* = 3.2 Hz, 1 H)**, 5.38 (d, *J* = 10.4 Hz, 1 H)*, 4.23-4.16 (m, 4 H), 4.09-4.06 (m, 1 H)**, 3.87-3.84 (m, 1 H)*, 3.51 (s, 3 H)*, 3.43 (s, 3 H)**, 3.38-3.12 (m, 4 H), 2.52-2.46 (m, 2 H) ppm;

Impurities observed at 2.27, 1.43, 1.25, 0.87 ppm.

^{13}C NMR (150 MHz, $CDCl_3$)

201.5*, 201.3**, 168.3**, 167.9*, 161.9**, 161.4*, 144.2**, 143.2*, 137.2*, 135.8**, 135.4*, 133.6**, 130.9*, 130.73**, 130.65*, 129.93 (2C)*, 129.89 (2C)**, 129.7**, 128.9**, 128.8*, 127.80 (2C)**, 127.75 (2C)*, 122.9**, 122.2*, 81.8*, 79.5*, 79.3*, 75.7*, 75.6**, 75.4*, 74.8**, 73.8**, 71.9**, 71.5*, 57.8**, 57.4**, 57.2*, 56.5*, 49.8*, 49.7**, 40.0**, 39.7*, 30.0*, 29.7** ppm;

Minor impurities observed: 67.6, 34.2, 29.9, 24.0, 22.9, 14.3 ppm

*Major diastereomer, **minor diastereomer

IR (thin film)

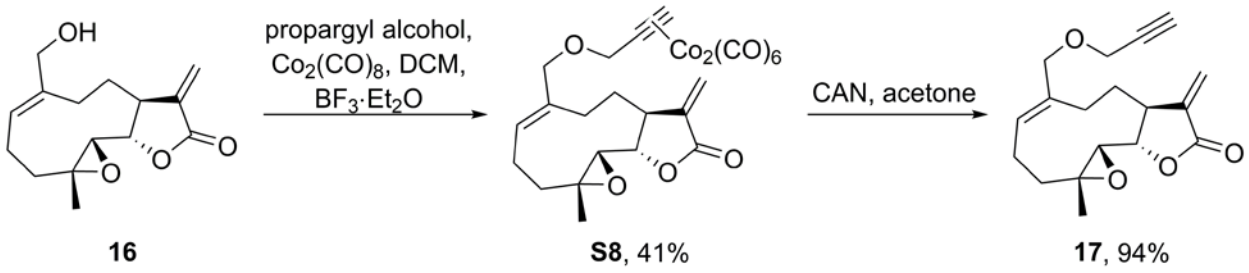
3279, 2933, 2852, 2115, 1769, 1703, 1492, 1445, 1269, 1134, 1095, 699 cm^{-1}.

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{24}H_{23}O_5$, 391.1540; found, 391.1525;

TLC R_f = 0.18 (40% ethyl acetate/hexanes)

Silica gel, UV, potassium permanganate



$Co_2(CO)_6$-*O*-(prop-2-ynyl)-MelB (S8). **Method A**: Follows general procedure C2: Propargyl alcohol (4 mg, 0.076 mmol), dichloromethane (0.5 mL), dicobalt octacarbonyl (26 mg, 0.076 mmol), Melampomagnolide B (MelB, **16**)[11] (10 mg, 0.038 mmol), dissolved in dichloromethane (0.3 mL), and boron trifluoride diethyl etherate (12 µL, 0.095 mmol). The reaction was quenched after 10 min of stirring despite a small amount of Mel B remaining in the reaction. The crude residue was purified by silica gel flash column chromatography (gradient of 10-20% ethyl acetate in hexanes) to afford 9 mg of **S8** in 41% yield as a dark red oil.

Method B: Follows general procedure C2: Methyl propargyl ether (8 mg, 0.11 mmol), dichloromethane (0.8 mL), dicobalt octacarbonyl (39 mg, 0.11 mmol), Mel B (**16**) (15 mg, 0.057 mmol), dissolved in dichloromethane (0.4 mL), and boron trifluoride diethyl etherate (18 µL, 0.14 mmol). The reaction was quenched after 40 min of stirring despite a small amount of Mel B remaining in the reaction. The crude residue was purified by silica gel flash column chromatography (gradient of 10-20% ethyl acetate in hexanes) to afford 13 mg of **S8** in 39% yield.

Data for **S8**

^{1}H NMR (400 MHz, $CDCl_3$)

6.25 (s, 1H), 6.04 (s, 1H), 5.67 (br s, 1H), 5.51 (s, 1H), 4.62 (d, *J* = 13.2 Hz, 1H), 4.53 (d, *J* = 12.8 Hz, 1H), 4.25 (d, *J* = 11.2 Hz, 1H), 3.98 (d, *J* = 12.0 Hz, 1H),

[11] El-Feraly, F.S. *Phytochemistry* **1984**, *23*, 2372-2374.

3.86 (t, *J* = 9.6 Hz, 1H), 2.87-2.85 (m, 2H), 2.43-2.31 (m, 4H), 2.22-2.15 (m, 2H), 1.67-1.64 (m, 1H), 1.55 (s, 3H), 1.12-1.06 (m, 1H) ppm;

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

199.6 (6C), 169.6, 139.1, 136.8, 129.4, 120.2, 91.1, 81.2, 73.8, 71.8, 70.3, 63.6, 60.2, 43.2, 36.9, 25.7, 24.3, 23.8, 18.1 ppm;

IR (thin film)

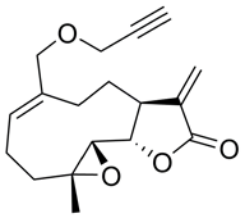
2931, 2360, 2095, 2053, 2023, 1770, 1262, 1138, 1075, 995 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{24}H_{23}O_{10}Co_2$, 588.9950; found, 588.9946;

TLC R_f = 0.19 (20% ethyl acetate in hexanes)

Silica gel, visible, UV



***O*-(prop-2-ynyl)-Mel B 17.** Followed general procedure B: $Co_2(CO)_6$-alkyne **S8** (9 mg, 0.015 mmol), acetone (1.5 mL), and ceric ammonium nitrate (34 mg, 0.061 mmol). The reaction was complete after 10 min of stirring. Reaction work up afforded 4 mg of pure alkyne **17** as a colorless oil in 94% yield. Further purification was not performed.

Data for **17**

^{1}H NMR (400 MHz, $CDCl_3$)

6.24 (d, *J* = 3.6 Hz, 1H), 5.70-5.66 (m, 1H), 5.55 (d, *J* = 3.2 Hz, 1H), 4.18 (dd, *J* = 16.0, 2.4 Hz, 1H), 4.16 (d, *J* = 10.8 Hz, 1H), 4.08 (dd, *J* = 16.0, 2.4 Hz, 1H), 3.90 (d, *J* = 11.6 Hz, 1H), 3.86 (t, *J* = 9.2 Hz, 1H), 2.96-2.82 (m, 1H), 2.86 (d, *J* = 9.6

Hz, 1H), 2.53-2.45 (m, 1H), 2.44 (t, *J* = 2.4 Hz, 1H), 2.40-2.27 (m, 3H), 2.22-2.14 (m, 2H), 1.68-1.62 (m, 1H), 1.55 (s, 3H), 1.14-1.07 (m, 1H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

169.6, 139.2, 136.6, 130.4, 120.1, 81.3, 79.7, 74.8, 72.9, 63.6, 60.1, 57.4, 43.1, 36.9, 25.9, 24.6, 23.9, 18.2 ppm;

Impurity observed at 29.8 ppm.

IR (thin film)

3274, 2921, 2850, 2112, 1764, 1261, 1138, 1073, 993, 815 cm^{-1};

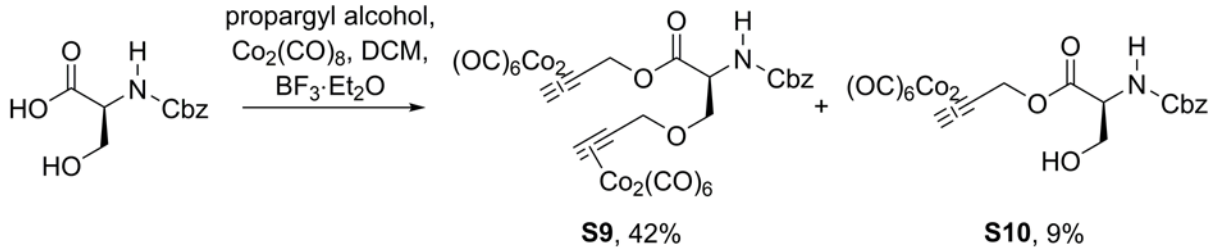
HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{18}H_{23}O_4$, 303.1591; found, 303.1595;

TLC R_f = 0.20 (30% ethyl acetate in hexanes)

Silica gel, UV, potassium permanganate

Additional Examples



S9, 42% **S10**, 9%

Dicobalt hexacarbonyl complexed prop-2-yn-1-yl N-((benzyloxy)carbonyl)-O-(prop-2-yn-1-yl)-L-serinate (S9). Follows general procedure C2: propargyl alcohol (23 mg, 0.42 mmol), dichloromethane (3.5 mL), dicobalt octacarbonyl (143 mg, 0.42 mmol), *N*-benzyloxycarbonyl-*L*-serine (50 mg, 0.21 mmol), additional dichloromethane (0.7 mL), and boron trifluoride diethyl etherate (65 µL, 0.52 mmol). The reaction stirred for 1.5 h. The crude residue was purified by

silica gel flash column chromatography (gradient of 5-30% ethyl acetate in hexanes) to afford 78 mg of **S9** in 42% yield as a dark red oil. 11 mg of **S10** (9% yield) was also isolated from the reaction.

Data for **S9**

^{1}H NMR (400 MHz, $CDCl_3$)

7.35 (s, 5H), 6.07 (s, 1H), 6.00 (s, 1H), 5.65 (d, *J* = 8.4 Hz, 1H), 5.37 (d, *J* = 14.4 Hz, 1H), 5.24 (d, *J* = 14.4 Hz, 1H), 5.14 (d, *J* = 12.4 Hz, 1H), 5.09 (d, *J* = 12.4 Hz, 1H), 4.68-4.65 (m, 1H), 4.63 (s, 2H), 4.16 (d, *J* = 8.4 Hz, 1H), 3.91 (d, *J* = 7.2 Hz, 1H), ppm;

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

199.5 (6C), 199.1 (6C), 169.8, 156.1, 136.4, 128.7 (2C), 128.3 (2C), 128.1, 90.2, 87.5, 72.3, 72.1, 71.4, 70.8, 67.2, 67.0, 54.6 ppm;

IR (thin film)

3452, 3093, 2934, 2097, 2055, 1024, 1729, 1507, 1332, 1195, 1112, 1065 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calcd for $C_{29}H_{17}O_{17}NCo_4Na$, 909.7717; found, 909.7753;

TLC R_f = 0.27 (10% ethyl acetate in hexanes)

Silica gel, visible, UV

Data for **S10**

^{1}H NMR (400 MHz, $CDCl_3$)

7.37-7.33 (m, 5H), 6.08 (s, 1H), 5.72-5.63 (m, 1H), 5.43 (d, *J* = 14.2 H, 1H), 5.33 (d, *J* = 14.2 Hz, 1H), 5.15 (d, *J* = 12.0 Hz, 1H), 5.11 (d, *J* = 12.0 Hz, 1H), 4.54 (br s, 1H), 4.11-3.92 (m, 2H), 2.05 (br s, 1H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

199.1 (6C), 170.3, 156.3, 136.2, 128.7 (2C), 128.4 (2C), 128.3, 87.4, 72.4, 67.4, 66.9, 63.4, 56.2 ppm;

IR (thin film)

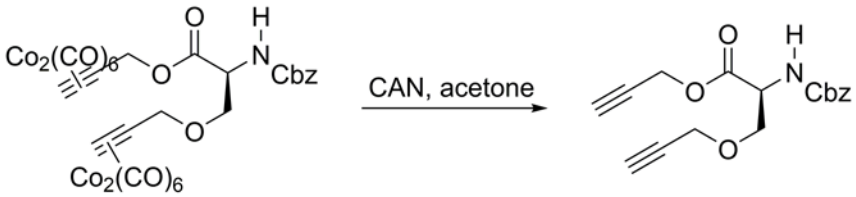
3439, 3091, 2933, 2360, 2098, 2057, 2026, 1725, 1521, 1456, 1333, 1191, 1063, 971, 698 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+Na]^+$ calcd for $C_{20}H_{15}O_{11}NCo_2Na$, 585.9201; found, 585.9205;

TLC R_f = 0.30 (30% ethyl acetate in hexanes)

Silica gel, visible, UV



S9 **S11**, 94%

Prop-2-yn-1-yl N-((benzyloxy)carbonyl)-O-(prop-2-yn-1-yl)-L-serinate (S11). A single-necked, 10 mL, round-bottomed flask, equipped with a stir bar and a septum pierced with a needle was charged with the dicobalt hexacarbonyl complexed dialkyne **S9** (35 mg, 0.04 mmol, 1 equiv), dissolved in acetone (4 mL). The solution was cooled to 0° C in an ice bath. Ceric ammonium nitrate (87 mg, 0.16 mmol, 4 equiv) was added to the flask in a single portion. After stirring for 10 min, the reaction was not complete as determined by TLC, and additional ceric ammonium nitrate (87 mg, 0.16 mmol, 4 equiv) was added. The reaction was stirred for 15 min. Upon completion, the reaction was diluted with distilled water (4 mL). The mixture was transferred to a separatory funnel. The aqueous layer was extracted with diethyl ether (3 x 6 mL). The combined organics were dried over magnesium sulfate, filtered, and concentrated under

reduced pressure rotary evaporation to afford 12 mg of **S11** in 94% yield as a colorless oil. Purification of the crude material was not performed.

Data for **S11**

^{1}H NMR (400 MHz, $CDCl_3$)

7.37-7.31 (m, 5H), 5.62 (d, *J* = 8.4 Hz, 1H), 5.17-5.10 (m, 2H), 4.81-4.72 (m, 2H), 4.58 (dt, *J* = 8.4, 2.8 Hz, 1H), 4.15-4.10 (m, 2H), 4.01 (dd, *J* = 9.2, 2.8 Hz, 1H), 3.81 (dd, *J* = 9.2, 2.8 Hz, 1H), 2.50, (t, *J* = 2.4 Hz, 1H), 2.43 (t, *J* = 2.4 Hz, 1H) ppm;

Impurities present at 1.43, 1.25, 0.88 ppm

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

169.6, 156.1, 136.3, 128.7 (2C), 128.4 (2C), 128.3, 78.8, 77.2, 75.6, 75.4, 69.5, 67.3, 58.8, 54.3, 53.3 ppm;

IR (thin film)

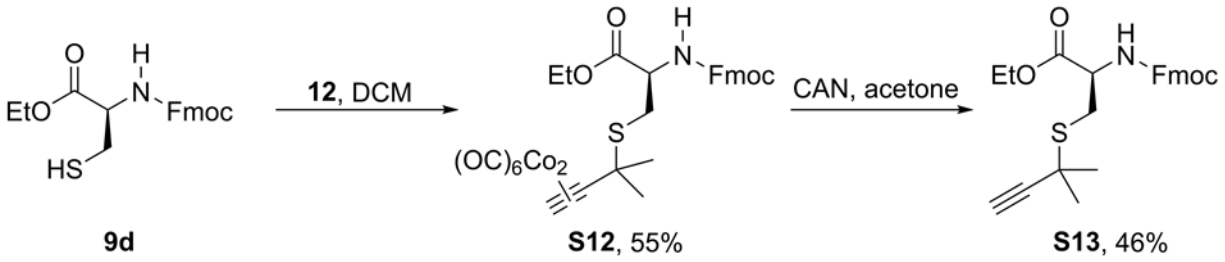
3288, 2915, 2850, 2129, 1750, 1715, 1515, 1456, 1342, 1260, 1027 cm^{-1}.

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{17}H_{18}O_5N$, 316.1180; found, 316.1181;

TLC R_f = 0.27 (30% ethyl acetate in hexanes)

Silica gel, UV, potassium permanganate



9d **S12**, 55% **S13**, 46%

$Co_2(CO)_6$-Ethyl *N*-(((9H-fluoren-9-yl)methoxy)carbonyl)-S-(1,1-dimethyl-3-propynyl)-L-cysteinate (S12). Follows general procedure C3: Tetrafluoroborate salt **12** (62 mg, 0.14 mmol, 1.3 equiv), dichloromethane (1.5 mL), *N*-(((9H-fluoren-9-yl)methoxy)carbonyl)-L-cysteine ethyl ester **9d** (42 mg, 0.11 mmol, 1 equiv) dissolved in dichloromethane (0.8 mL). The reaction stirred for 2 h. The crude residue was purified by silica gel flash column chromatography (gradient of 15-30% diethyl ether in hexanes) to afford 42 mg of cobalt complexed alkyne **S12** in 55% yield as a red oil.

Data for **S12**

^{1}H NMR (400 MHz, $CDCl_3$)

7.77 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.41 (app t, *J* = 7.4 Hz, 2H), 7.32 (app t, *J* = 7.4 Hz, 2H), 6.23 (s, 1H), 5.59 (d, *J* = 7.6 Hz, 1H), 4.71-4.66 (m, 1H), 4.39 (d, *J* = 7.2 Hz, 2H), 4.27-4.21 (m, 3H), 3.19-3.10 (m, 2H), 1.621 (s, 3H), 1.616 (s, 3H), 1.30 (t, *J* = 7.2 Hz, 3H) ppm;

^{13}C NMR (125 MHz, $CDCl_3$)

199.8 (6C), 170.5, 155.8, 144.0, 143.9, 141.5 (2C), 127.9 (2C), 127.2 (2C), 125.3 (2C), 120.2 (2C), 105.0, 73.1, 67.4, 62.2, 53.6, 48.9, 47.3, 32.7 (2C), 32.4, 14.3 ppm;

IR (thin film)

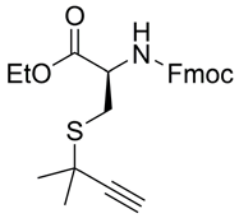
3338, 3070, 2979, 2092, 2053, 2022, 1725, 1510, 1451, 1200, 1052, 759, 740 cm^{-1};

HRMS (FTMS + p ESI Full ms)

$[M+NH_4]^+$ calcd for $C_{31}H_{31}O_{10}N_2SCo_2$, 741.0358; found, 741.0379;

TLC R_f = 0.18 (20% diethyl ether in hexanes)

Silica gel, visible, UV



***N*-(((9H-fluoren-9-yl)methoxy)carbonyl)-*S*-(1,1-dimethyl-3-propynyl)-L-cysteine ethyl ester (S13).** Followed general procedure D: $Co_2(CO)_6$-alkyne **S12** (47 mg, 0.069 mmol), acetone (6.9 mL), and ceric ammonium nitrate (152 mg, 0.28 mmol). The reaction was complete after 15 min of stirring. The crude residue was purified by silica gel flash column chromatography (20% diethyl ether in hexanes) which afforded 14 mg of alkyne **S13** in 46% yield as a colorless oil.

Data for **S13**

^{1}H NMR (400 MHz, $CDCl_3$)

7.77 (d, *J* = 7.6 Hz, 2H), 7.63-7.58 (m, 2H), 7.40 (app t, *J* = 7.4 Hz, 2H), 7.30 (app t, *J* = 7.4 Hz, 2H), 5.65 (d, *J* = 8.0 Hz, 1H), 4.69 (dt, *J* = 8.0, 5.2 Hz, 1H), 4.44-4.37 (m, 2H), 4.27-4.21 (m, 3H), 3.28-3.19 (m, 2H), 2.37 (s, 1H), 1.57 (s, 6H), 1.30 (t, *J* = 7.0 Hz, 3H) ppm;

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

170.7, 155.9, 144.0, 143.9, 141.5, 141.4, 127.9 (2C), 127.2 (2C), 125.3 (2C), 120.1 (2C), 87.8, 70.9, 67.3, 62.1, 53.6, 47.3, 38.7, 33.2, 30.79, 30.75, 14.3 ppm;

IR (thin film)

3292, 2976, 2925, 2365, 1719, 1509, 1449, 1339, 1208, 1051, 759, 740 cm^{-1};

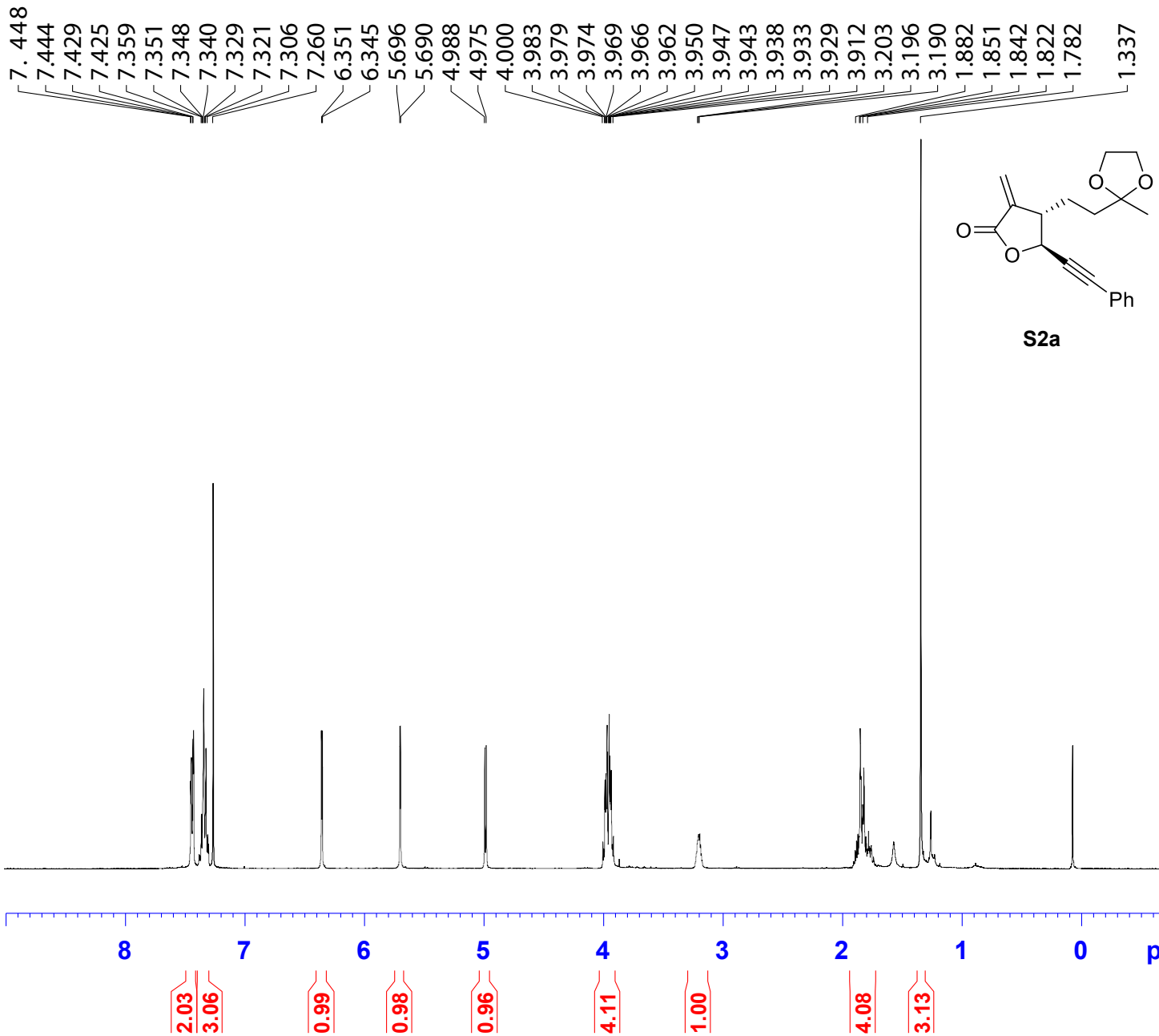
HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{25}H_{28}O_4NS$, 438.1734; found, 438.1725;

TLC R_f = 0.23 (30% diethyl ether in hexanes)

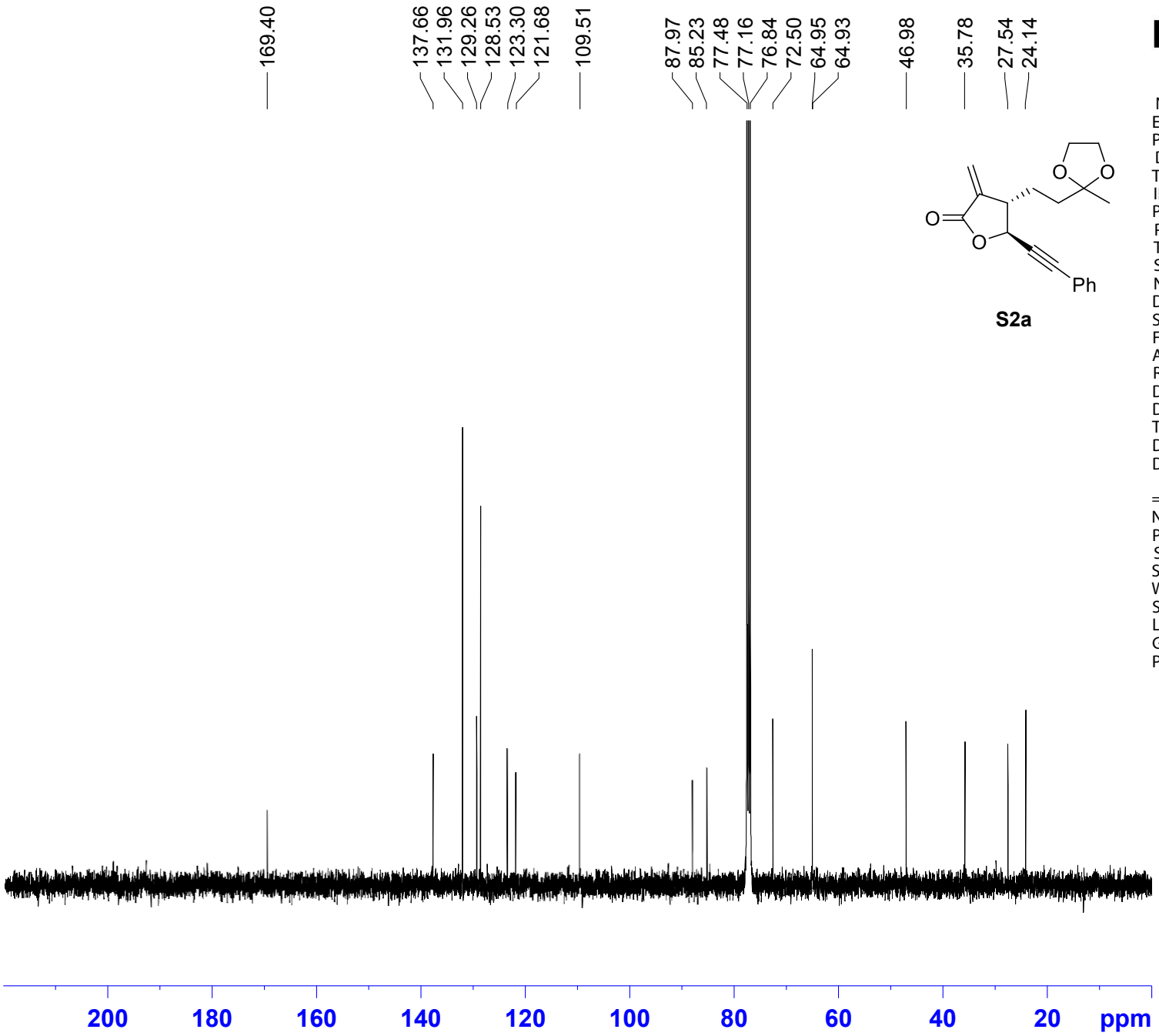
Silica gel, UV, potassium permanganate

SW07–024–B 1H 400



NAME	SW07–024–B	
EXPNO	10	
PROCNO	1	
Date_	20150625	
Time	8.39	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	128	
DW	60.800	usec
DE	6.50	usec
TE	94.7	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300101	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

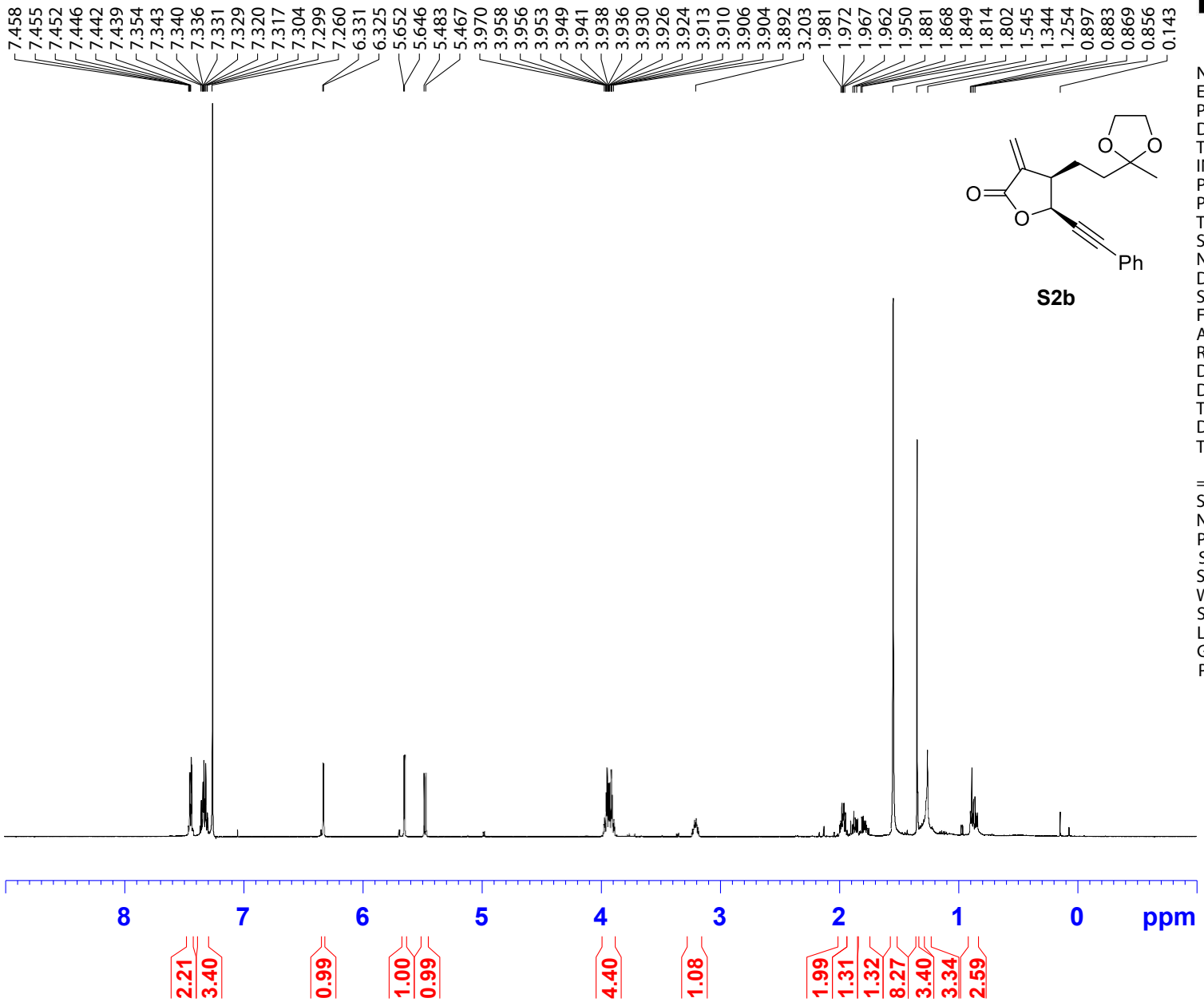
SW07–009–B 13C 400



S2a

NAME	SW07–009–B	
EXPNO	11	
PROCNO	1	
Date_	20150617	
Time	1.51	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3072	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	144	
DW	20.800	usec
DE	6.50	usec
TE	70.7	K
D1	2.00000000	sec
D11	0.03000000	sec
======== CHANNEL f1	========	
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127562	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

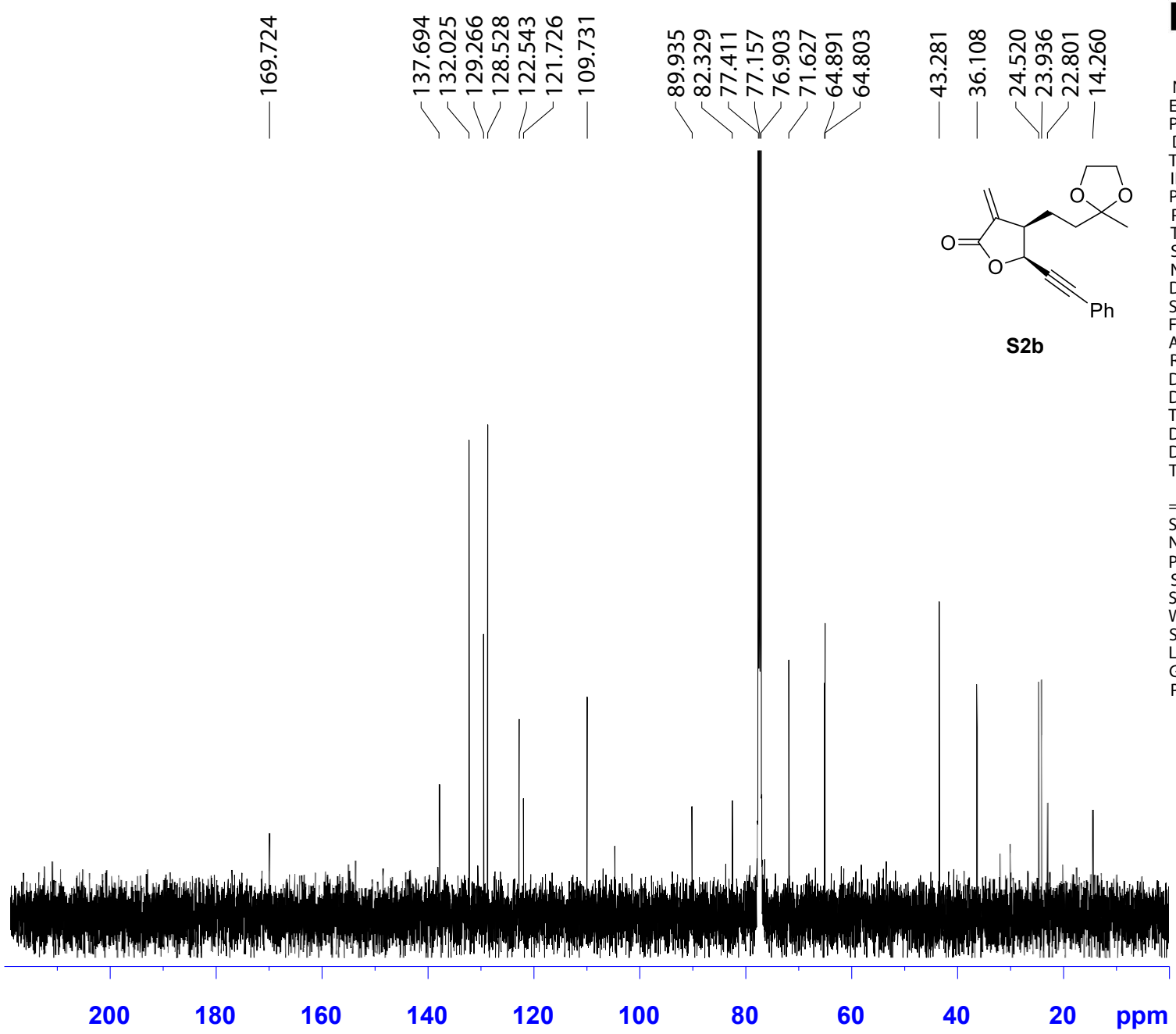
SW07–024–C 1H 500



S2b

NAME	SW07–024–C	
EXPNO	10	
PROCNO	1	
Date_	20150707	
Time	18.30	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10000.000	Hz
FIDRES	0.152588	Hz
AQ	3.2768500	sec
RG	203	
DW	50.000	usec
DE	6.50	usec
TE	298.2	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1========		
SFO1	500.1630887	MHz
NUC1	1H	
P1	11.45	usec
SI	65536	
SF	500.1600125	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

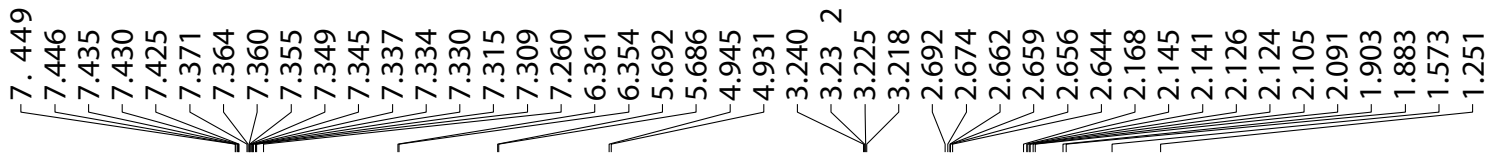
SW07–024–C 13C 500

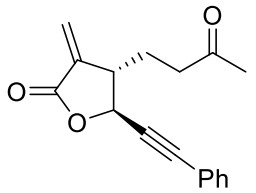


S2b

NAME	SW07–024–C	
EXPNO	11	
PROCNO	1	
Date_	20150707	
Time	23.48	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3072	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	298.3	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	125.7779086	MHz
NUC1	13C	
P1	10.50	usec
SI	32768	
SF	125.7653131	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

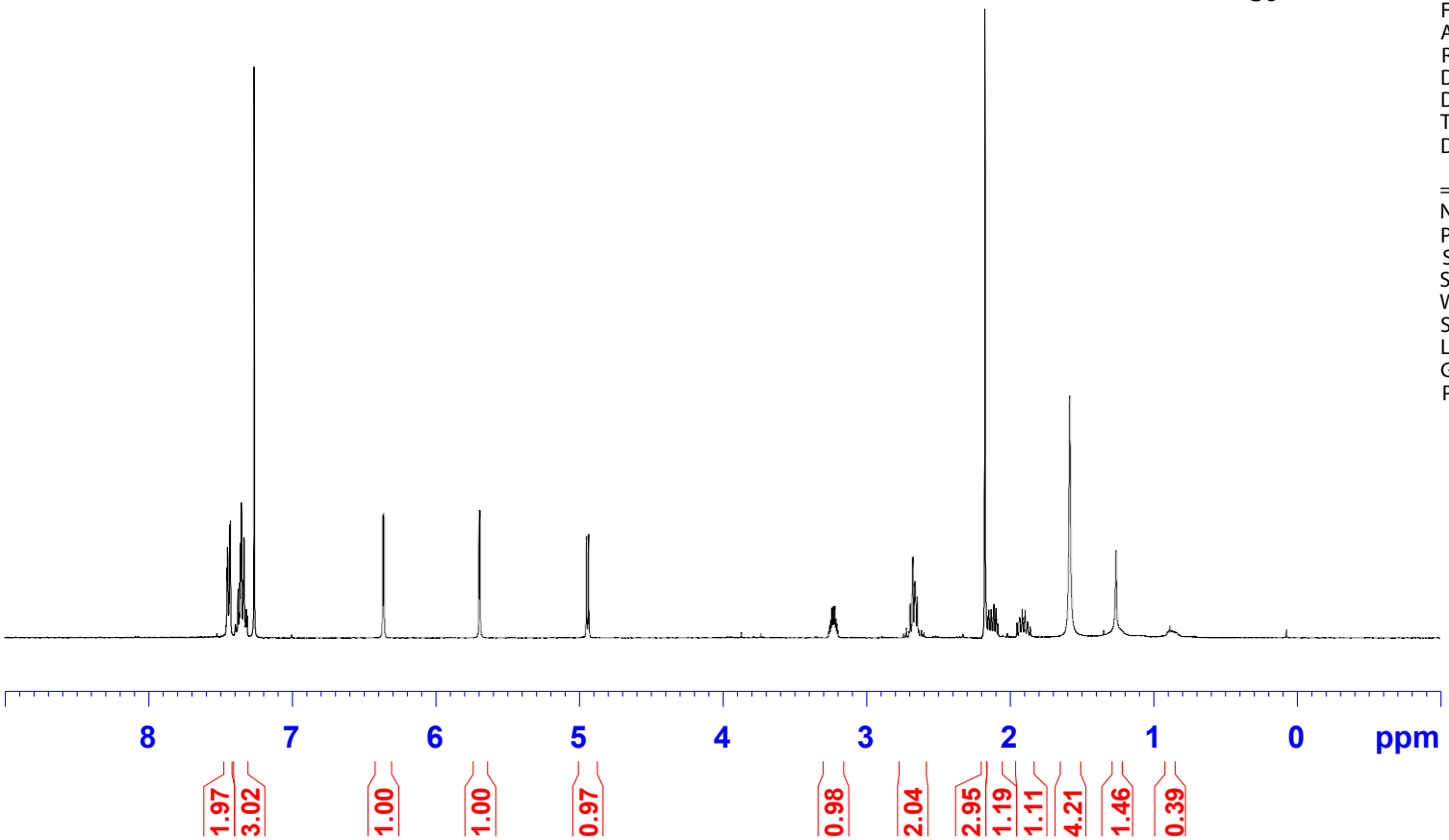
SW07–033–A



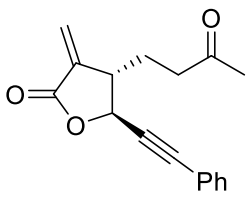


S3

NAME	SW07–033–A	
EXPNO	10	
PROCNO	1	
Date_	20150707	
Time	14.43	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	161	
DW	60.800	usec
DE	6.50	usec
TE	96.3	K
D1	1.00000 000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300104	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

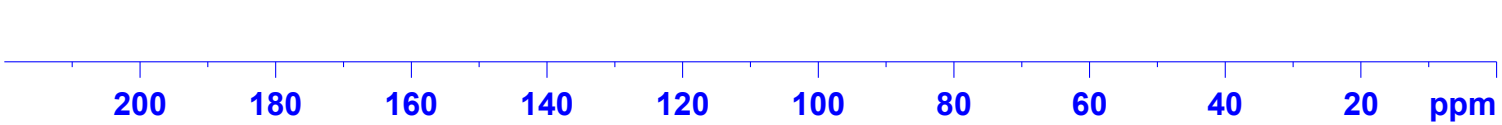


SW07–033–A 13C

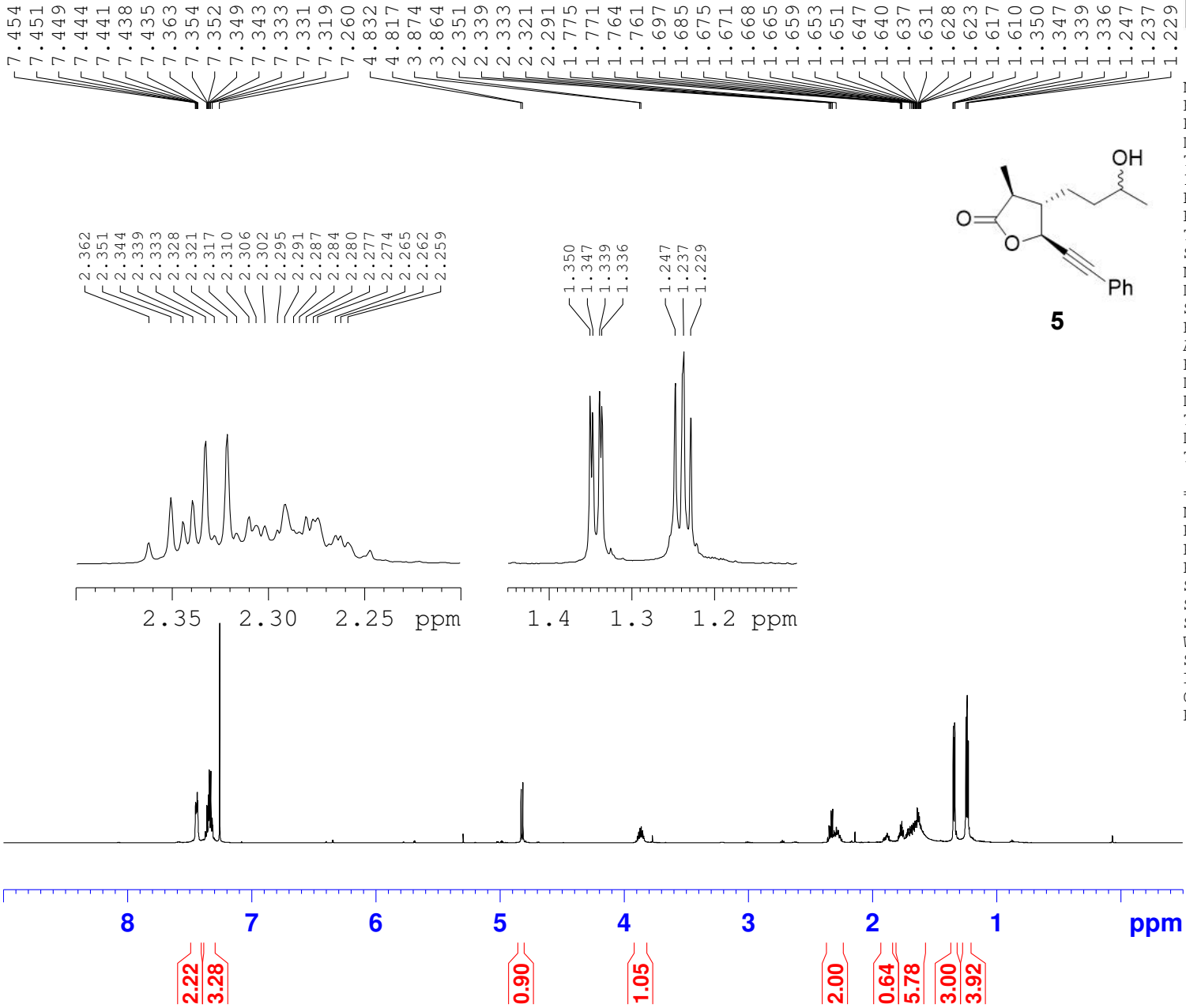


S3

NAME	SW07–033–A	
EXPNO	11	
PROCNO	1	
Date_	20150708	
Time	4.34	
INSTRUM	sp ect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	181	
DW	20.800	usec
DE	6.50	usec
TE	95.5	K
D1	2.00000000	sec
D11	0.03000000	sec
======== CHANNEL f1	========	
NUC1	13C	
P1	10. 00	usec
SI	32768	
SF	100.6127552	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	



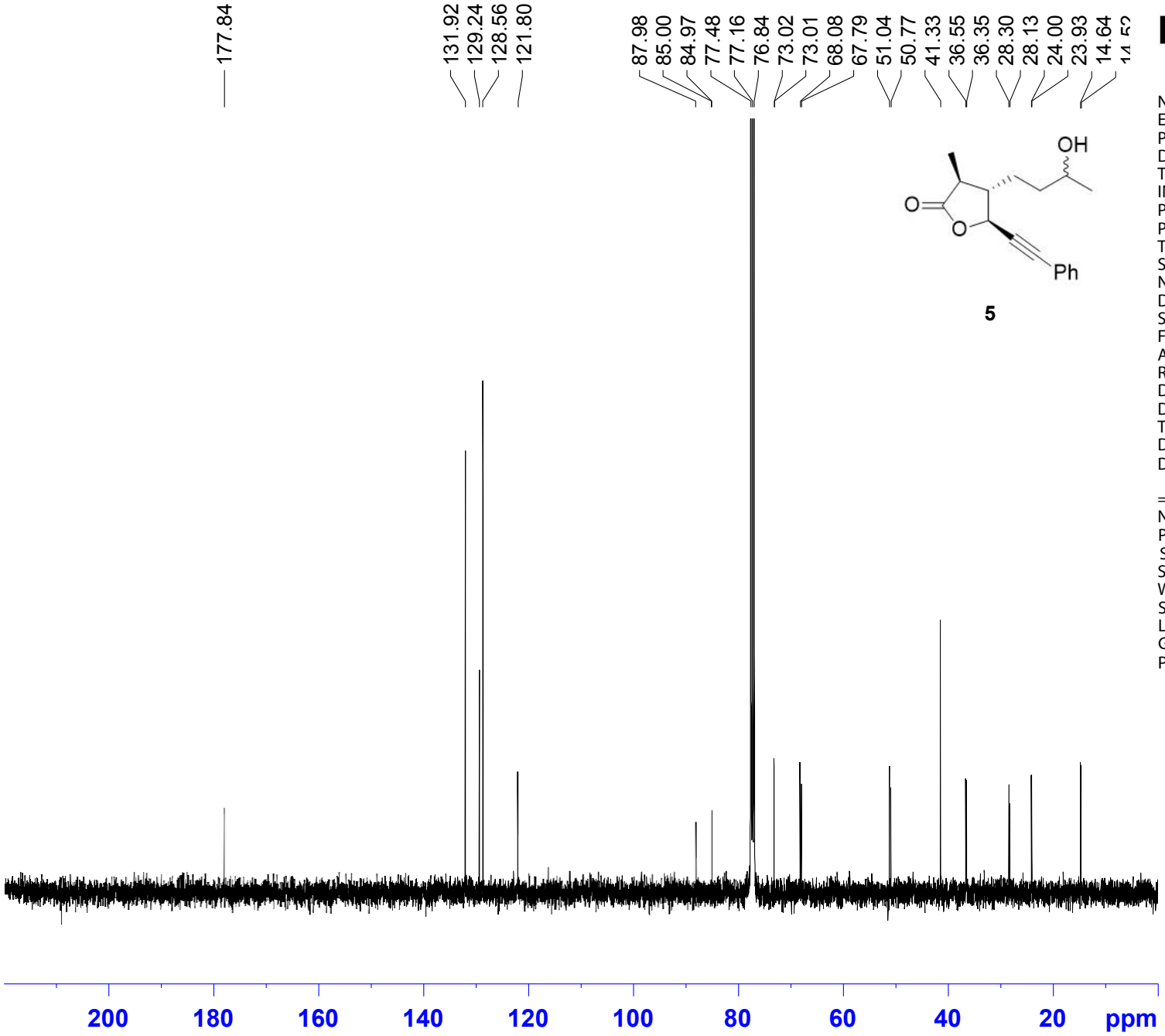
SW06-003-1H CDCl3 600MHz



5

NAME	SW06-003-1H
EXPNO	1
PROCNO	1
Date_	20160627
Time	14.14
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	161
DW	40.533 usec
DE	6.50 usec
TE	294.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.86 usec
PL1	-2.00 dB
PL1W	19.70630455 W
SFO1	600.7137096 MHz
SI	32768
SF	600.7100142 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

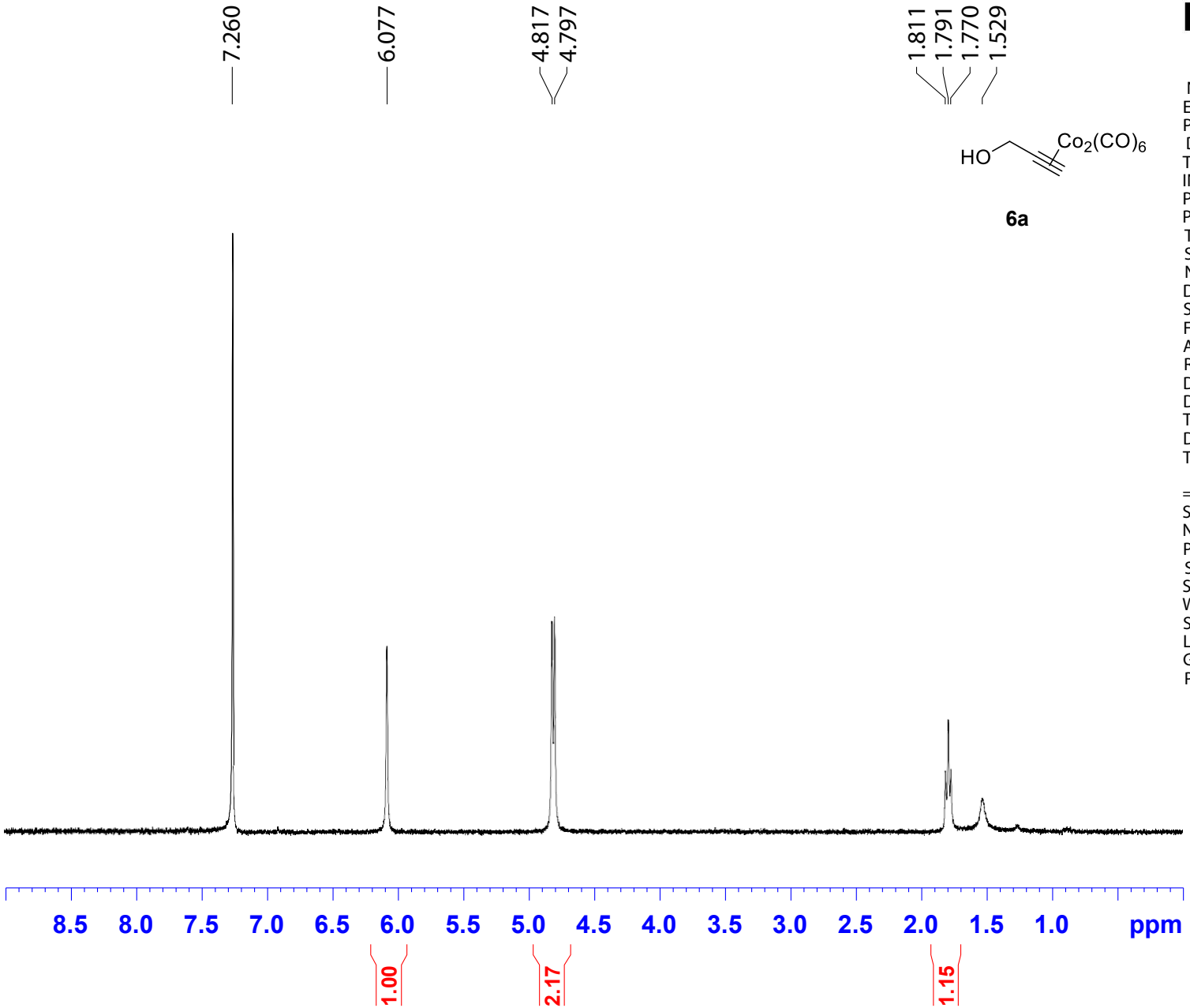
SW06–117–B 13C 400



5

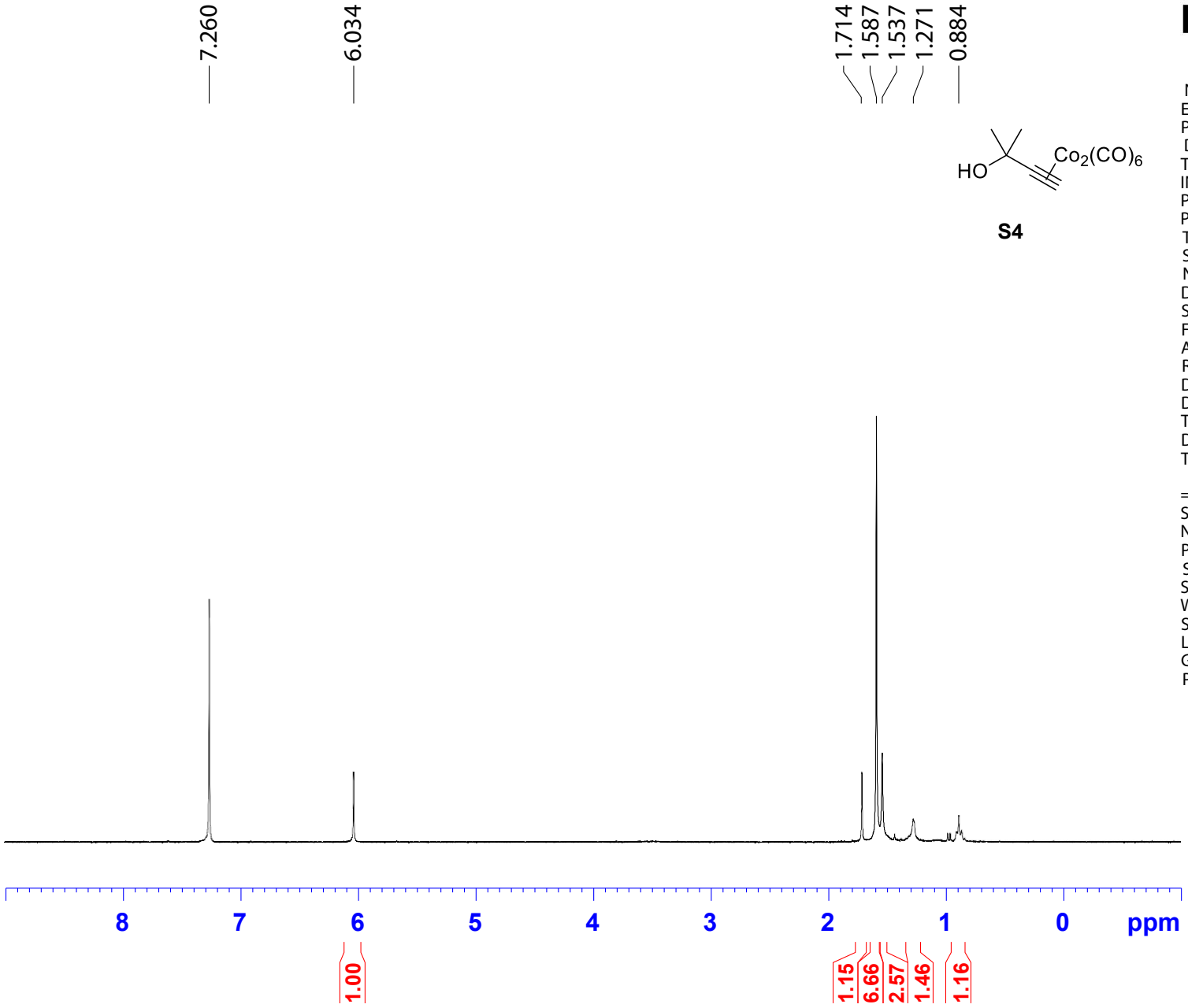
NAME	SW06–117–B
EXPNO	21
PROCNO	1
Date_	20150305
Time	0.33
INSTRUM	spect
PROBHD	5 mm PABBO BB–
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	94.8 K
D1	2.00000000 sec
D11	0.03000000 sec
======== CHANNEL f1========	
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127550 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

SW07–164–SM



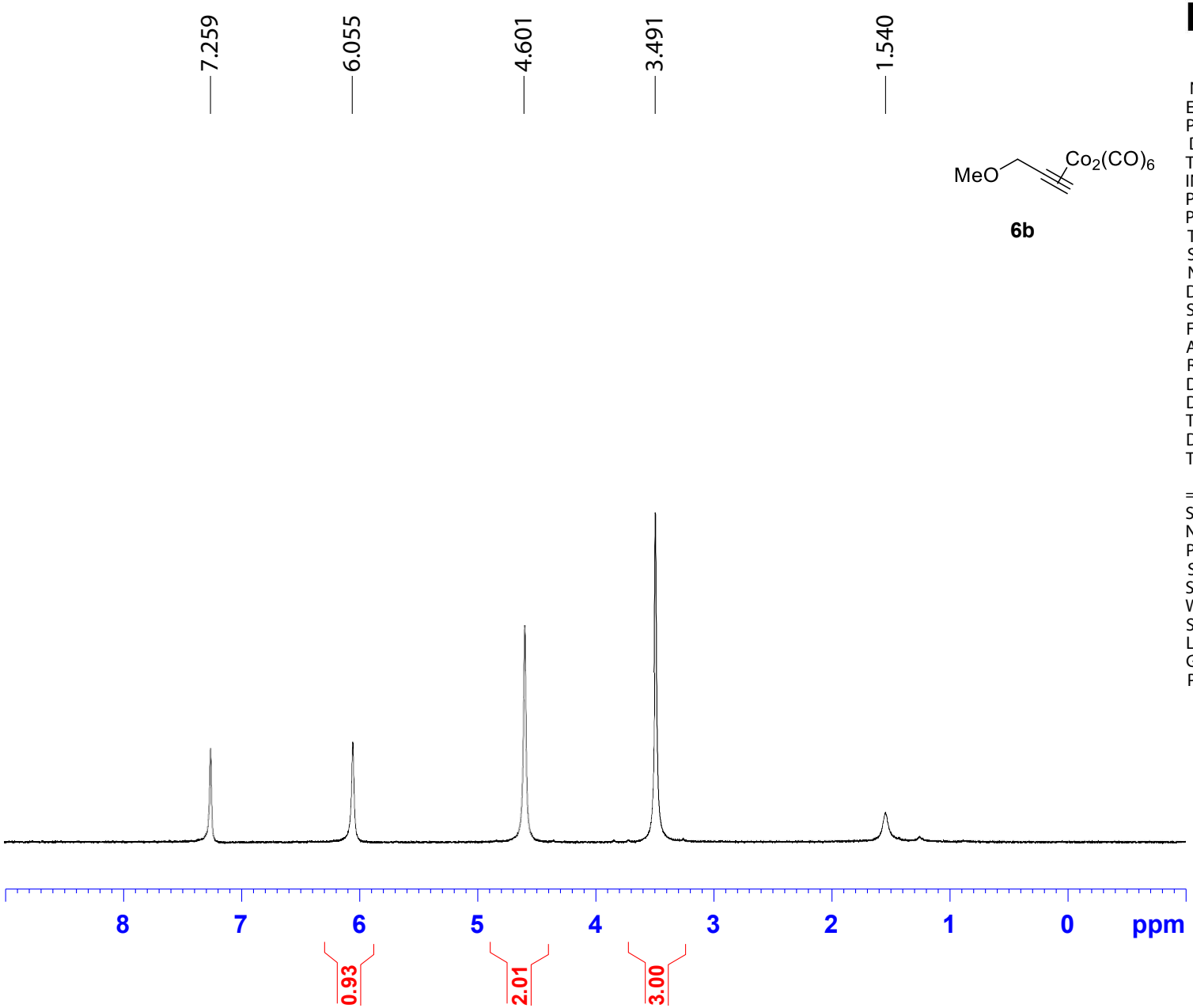
NAME	SW07–164–SM	
EXPNO	10	
PROCNO	1	
Date_	20160119	
Time	9.54	
INSTRUM	spect	
PROBHD 5 mm Q NP	1H/1	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	6188.119	Hz
FIDRES	0.188846	Hz
AQ	2.6477044	sec
RG	322	
DW	80.800	usec
DE	6.50	usec
TE	−925.8	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	300.2318540	MHz
NUC1	1H	
P1	12.71	usec
SI	32768	
SF	300.2300091	MHz
WDW	EM	
SSB	0	
LB	0.10	Hz
GB	0	
PC	1.00	

SW07–115–B 1H 300



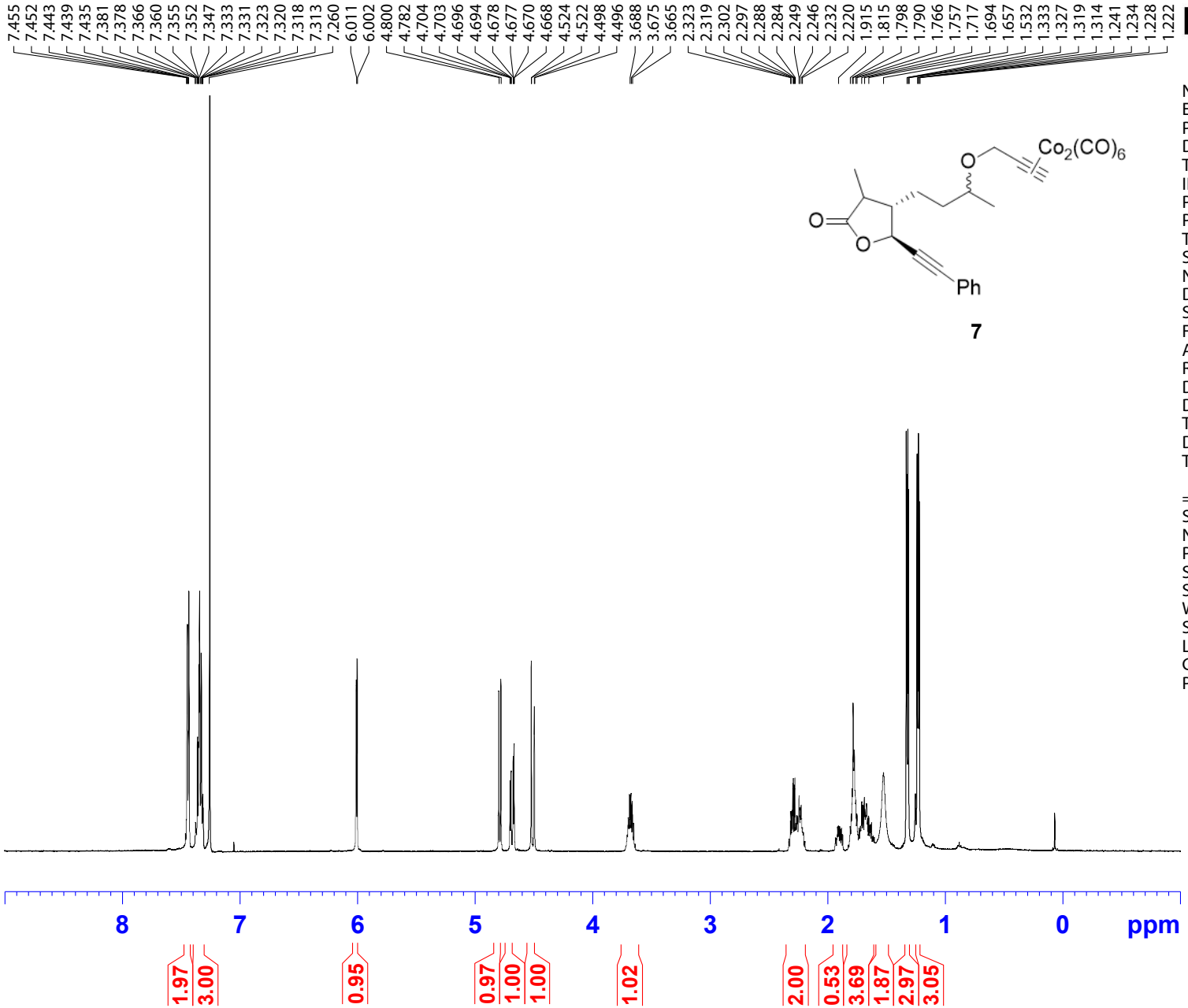
NAME	SW07–115–B	
EXPNO	10	
PROCNO	1	
Date_	20151008	
Time	14.36	
INSTRUM	spect	
PROBHD 5 mm QNP 1H/1		
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	6188.119	Hz
FIDRES	0.188846	Hz
AQ	2.6477044	sec
RG	322	
DW	80.800	usec
DE	6.50	usec
TE	−922.4	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1========		
SFO1	300.2318540	MHz
NUC1	1H	
P1	12.71	usec
SI	32768	
SF	300.2300088	MHz
WDW	EM	
SSB	0	
LB	0.10	Hz
GB	0	
PC	1.00	

SW07–052–cr 1H 300



NAME	SW07–052–cr	
EXPNO	10	
PROCNO	1	
Date_	20150728	
Time	15.47	
INSTRUM	spect	
PROBHD 5 mm QNP	1H/1	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	6188.119	Hz
FIDRES	0.188846	Hz
AQ	2.6477044	sec
RG	322	
DW	80.800	usec
DE	6.50	usec
TE	−930.4	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	300.2318540	MHz
NUC1	1H	
P1	12.71	usec
SI	32768	
SF	300.2300091	MHz
WDW	EM	
SSB	0	
LB	0.10	Hz
GB	0	
PC	1.00	

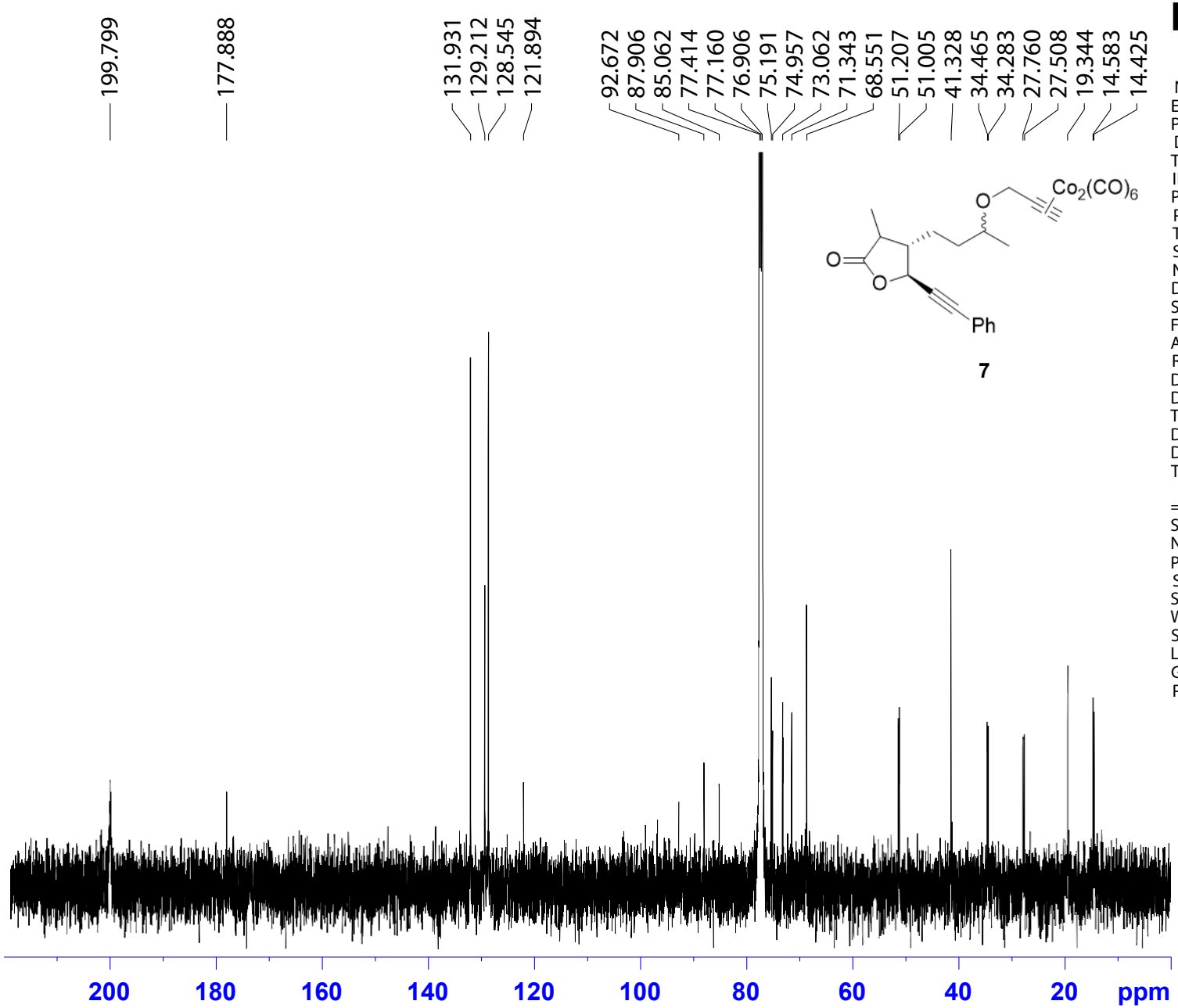
SW06–199–A 1H 500



7

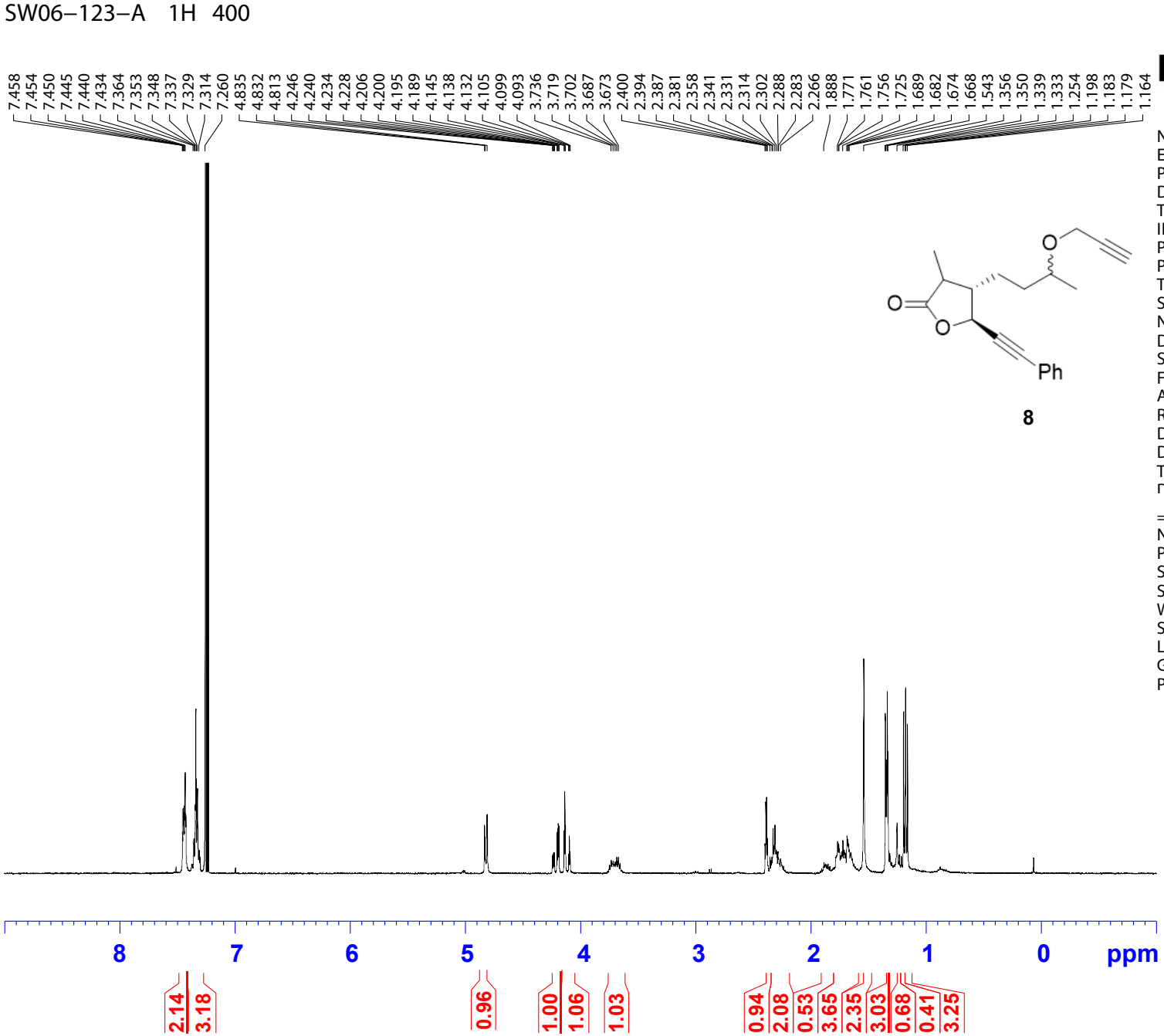
NAME	SW06–199–A	
EXPNO	10	
PROCNO	1	
Date_	20150518	
Time	12.47	
INSTRUM	spect	
PROBHD	5 mn PABBO BB/	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10000.000	Hz
FIDRES	0.152588	Hz
AQ	3.2768500	sec
RG	203	
DW	50.000	usec
DE	6.50	usec
TE	298.2	K
D1	1.00000000	sec
TD0	1	
========	CHANNEL f1	========
SFO1	500.1630887	MHz
NUC1	1H	
P1	11.45	usec
SI	65536	
SF	500.1600119	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW06–199–A 13C 500



NAME	SW06–199–A	
EXPNO	20	
PROCNO	1	
Date_	20150520	
Time	0.43	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	4096	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	298.5	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	125.7779086	MHz
NUC1	13C	
P1	10.50	usec
SI	32768	
SF	125.7653126	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

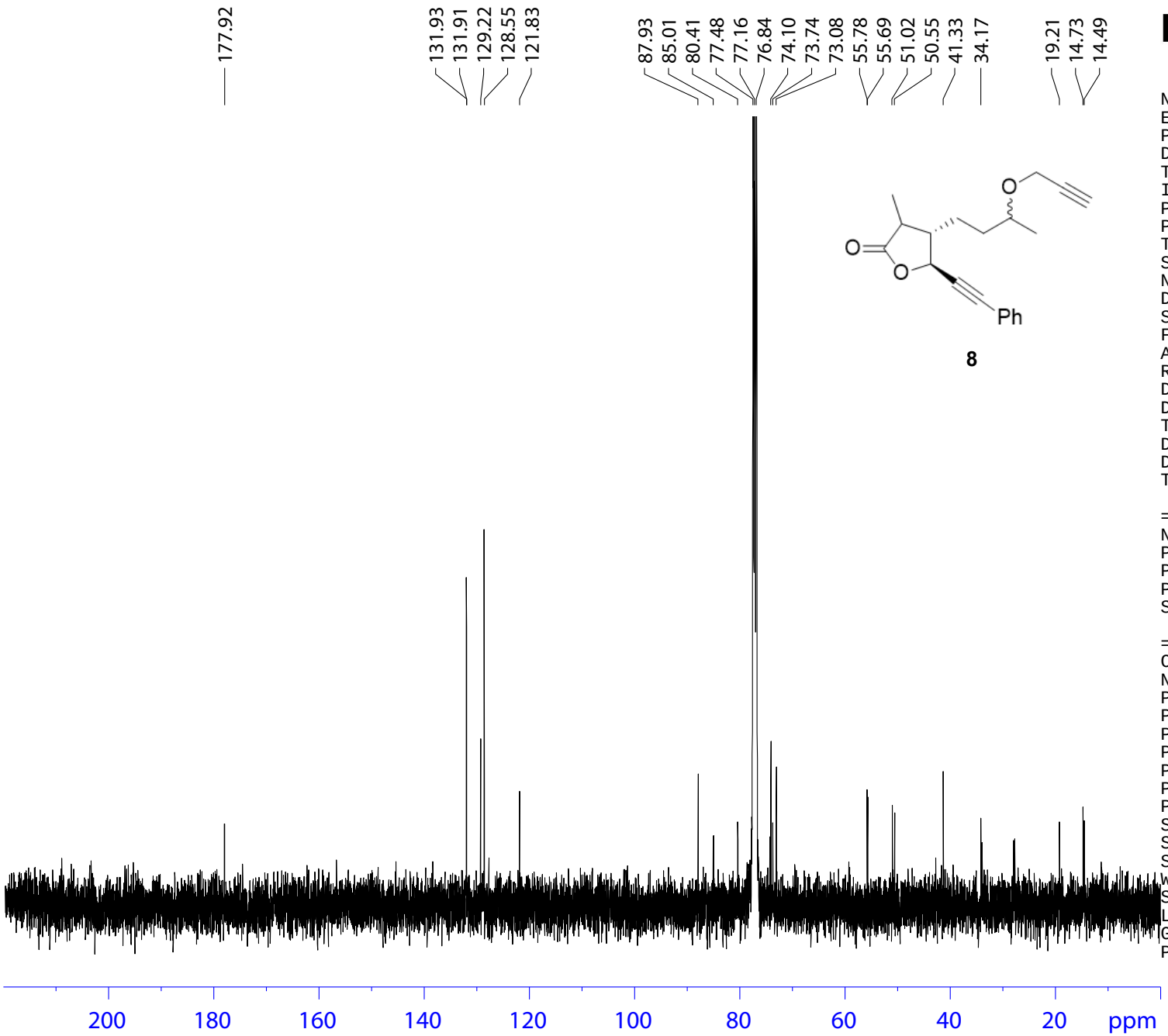
SW06–123–A 1H 400



BRUKER

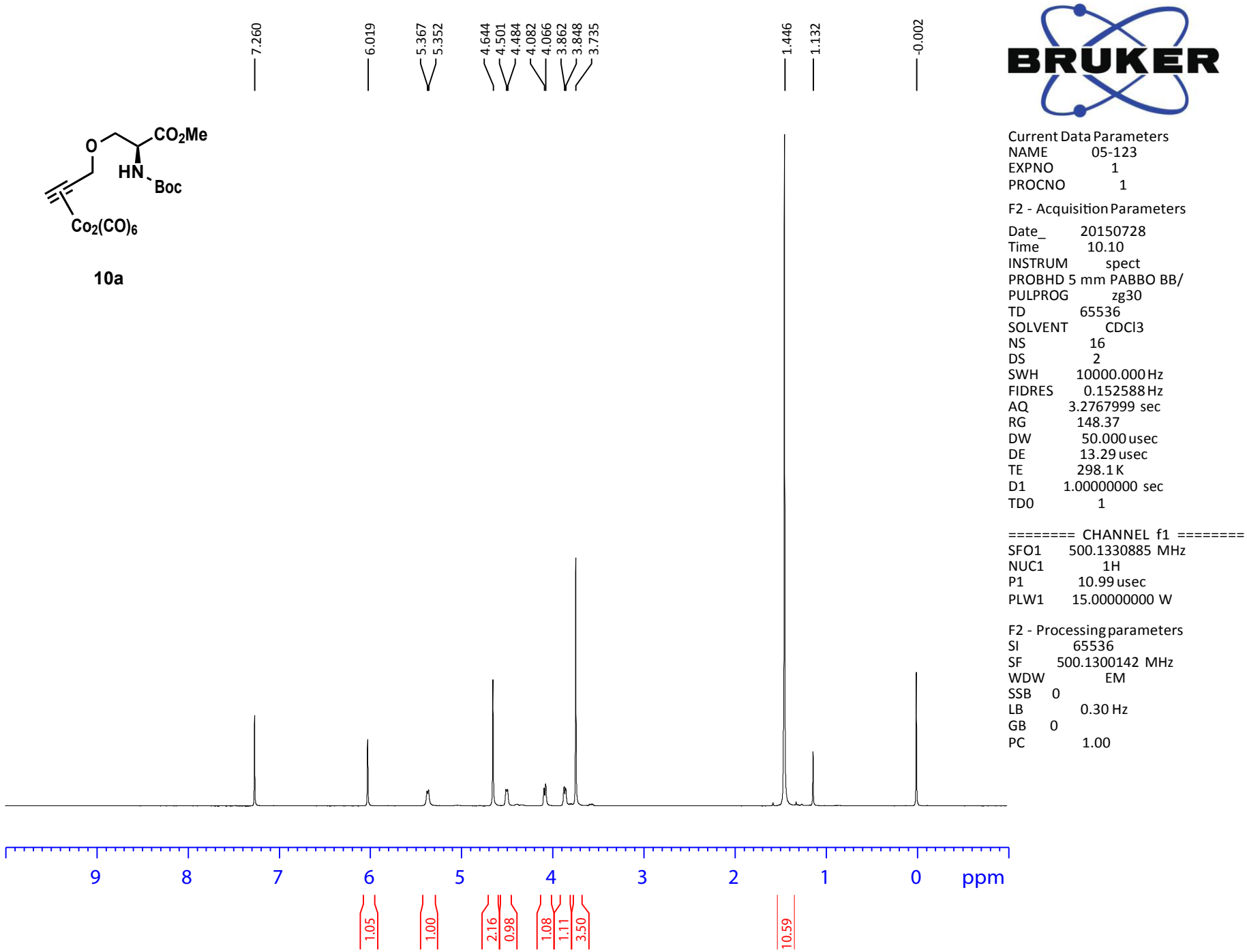
NAME	SW06–123–A	
EXPNO	10	
PROCNO	1	
Date_	20150305	
Time	15.09	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	161	
DW	60.800	usec
DE	6.50	usec
TE	95.9	K
D1	1.000 00000	sec
======== CHANNEL f1 ========		
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1 3001 06	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW06–123–A 13C 400B



8

NAME	SW06–123–A
EXPNO	2
PROCNO	1
Date_	20150307
Time	14.50
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4394
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	45.2
DW	20.800 usec
DE	6.50 usec
TE	295.4 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	–0.44 dB
PL1W	39.19395828 W
SFO1	100.6479773 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	–3.90 dB
PL12	15.81 dB
PL13	120.00 dB
PL2W	21.64248466 W
PL12W	0.23137002 W
PL13W	0.00000000 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6379009 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



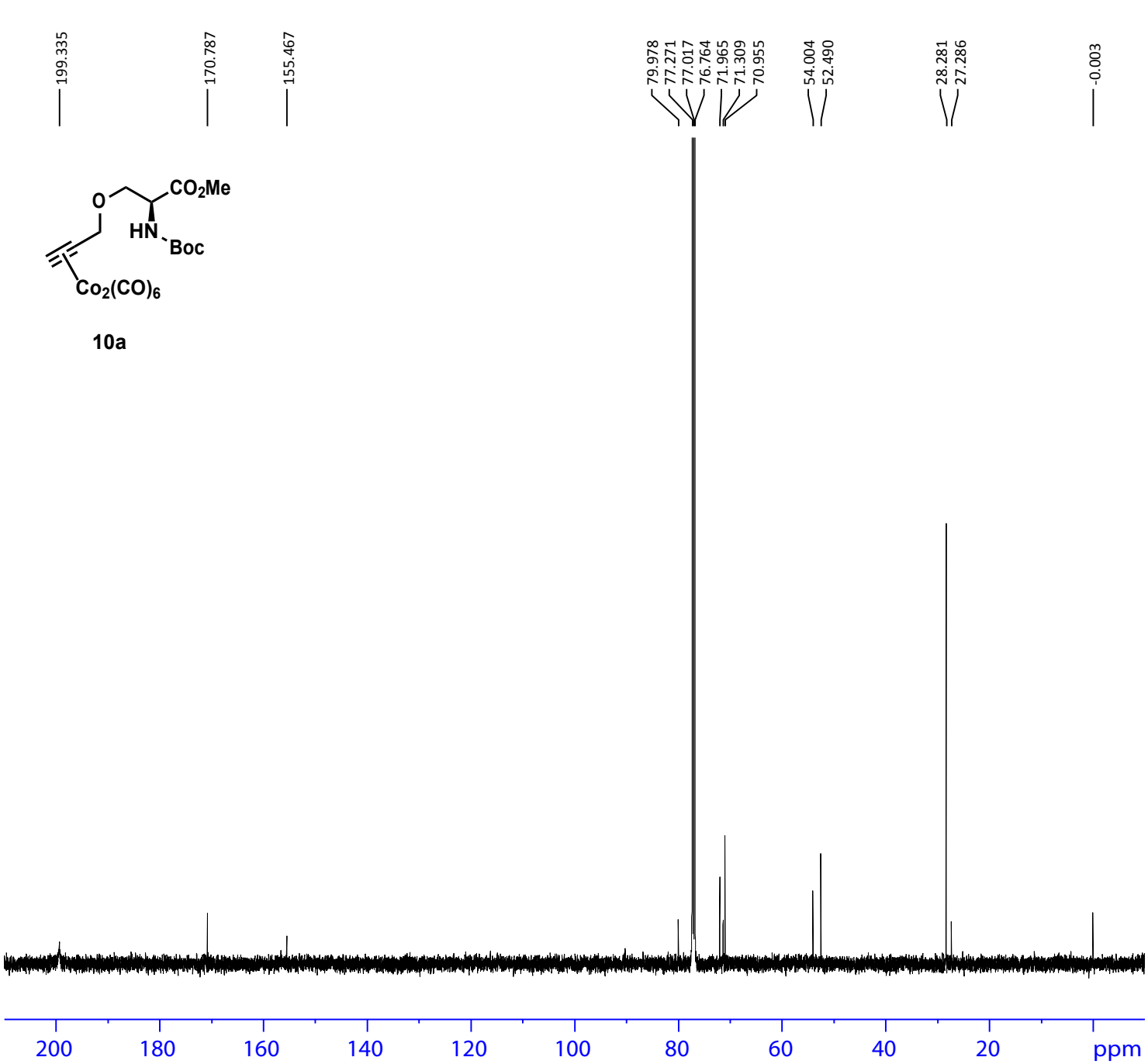
Current Data Parameters
NAME 05-123
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20150728
Time 10.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 148.37
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

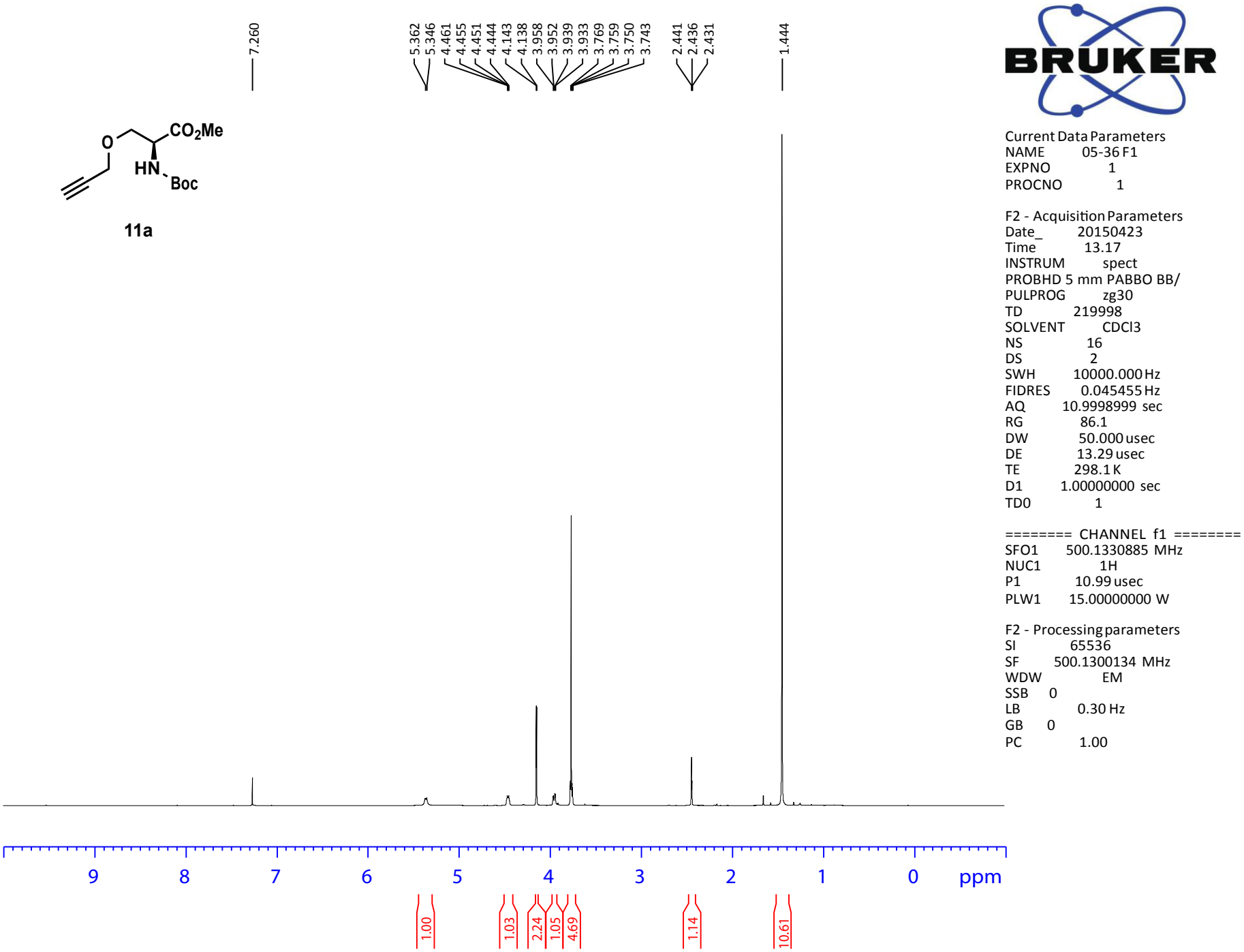
Current Data Parameters
NAME 05-123
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150728
Time 10.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703643 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

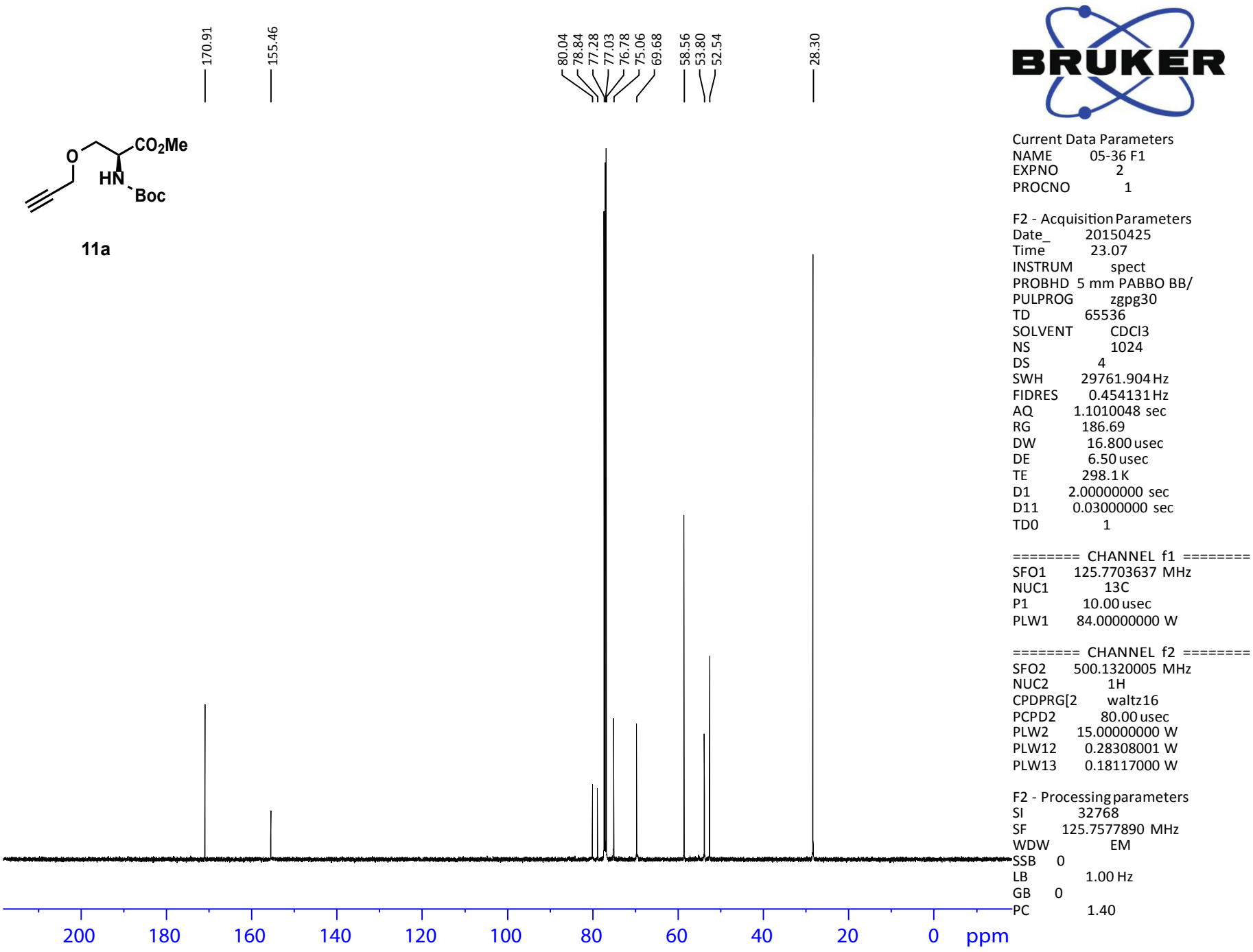


Current Data Parameters
NAME 05-36 F1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150423
Time 13.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 219998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.045455 Hz
AQ 10.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



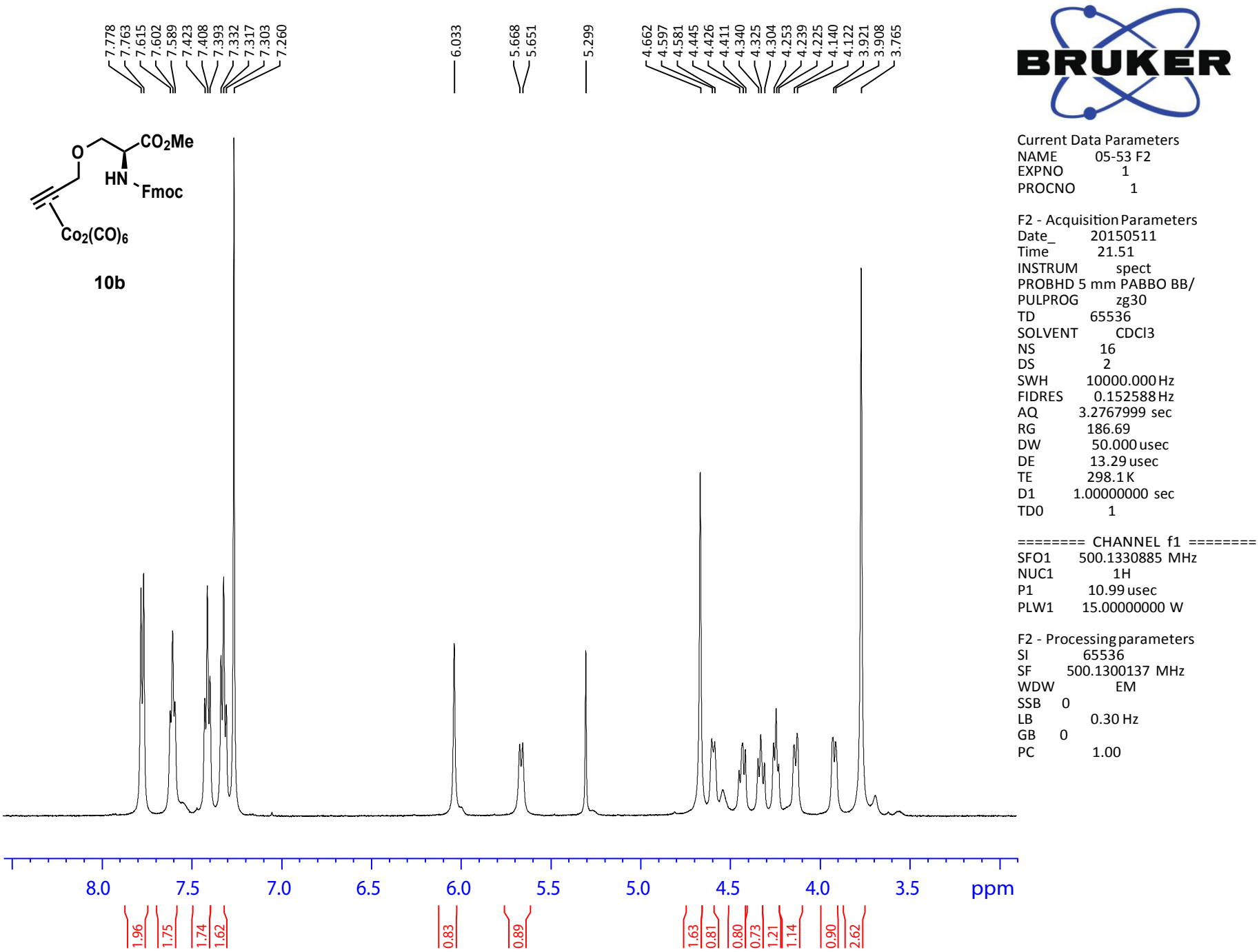
Current Data Parameters
NAME 05-36 F1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150425
Time 23.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

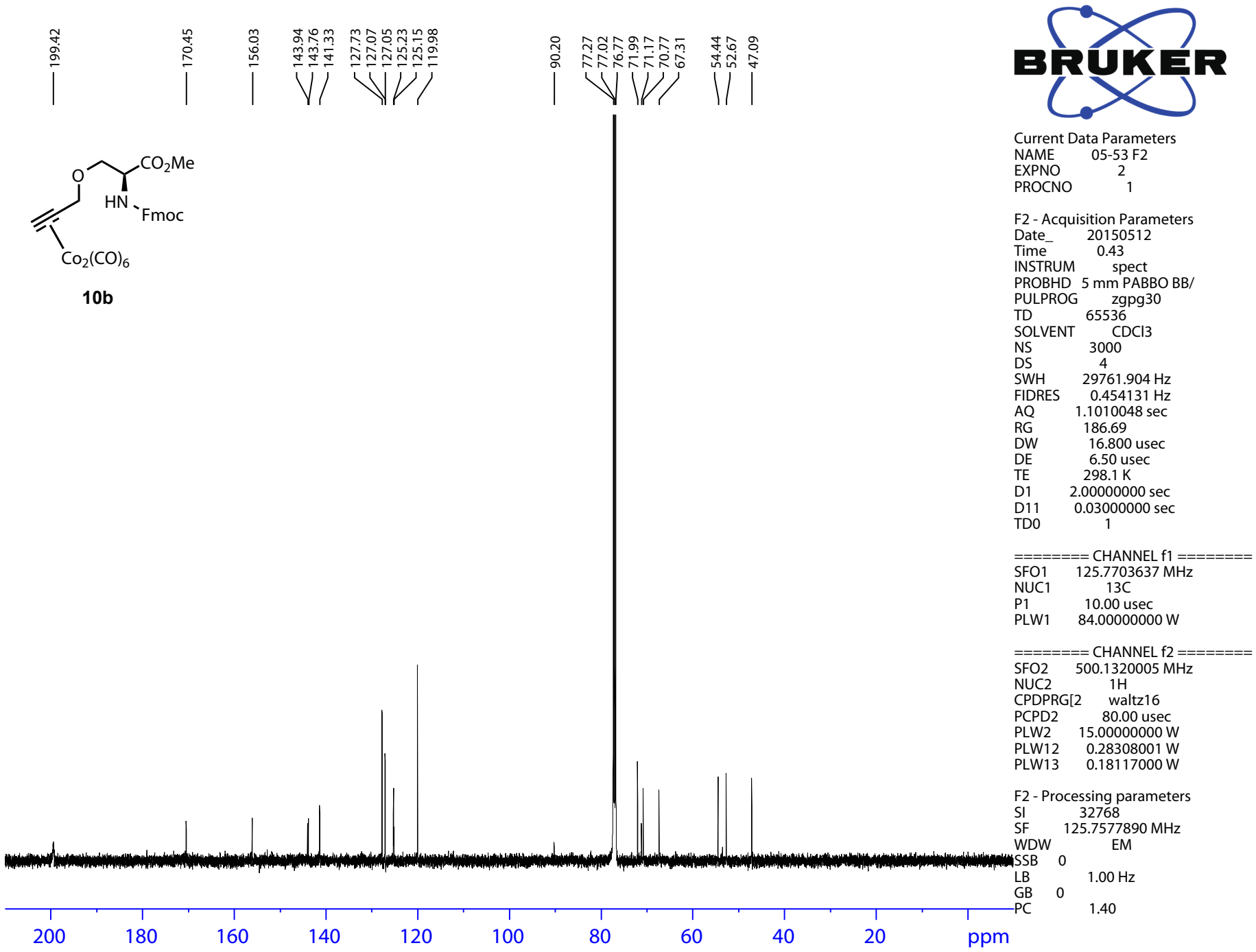


Current Data Parameters
NAME 05-53 F2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150511
Time 21.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 186.69
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300137 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



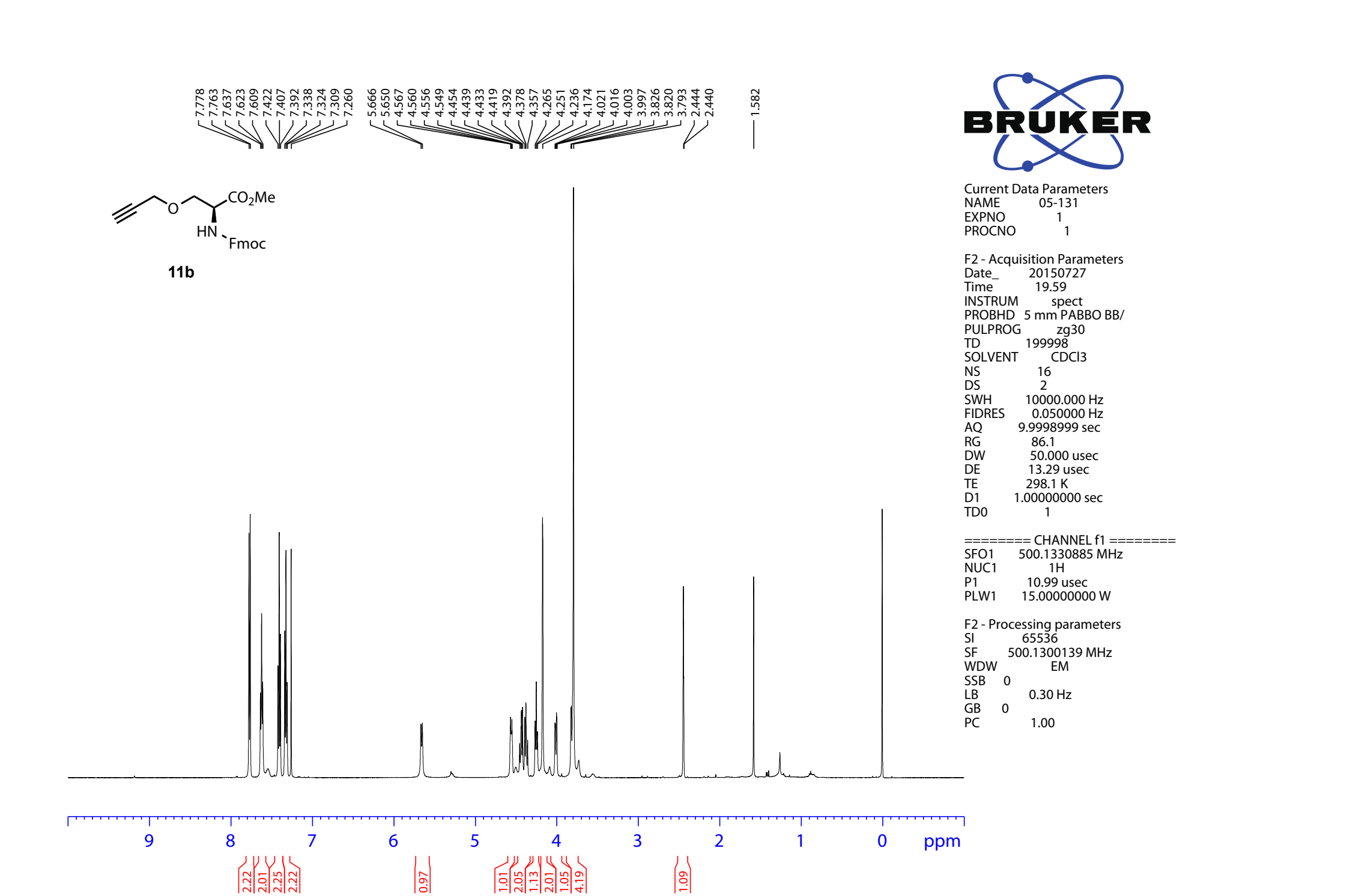
Current Data Parameters
NAME 05-53 F2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150512
Time 0.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

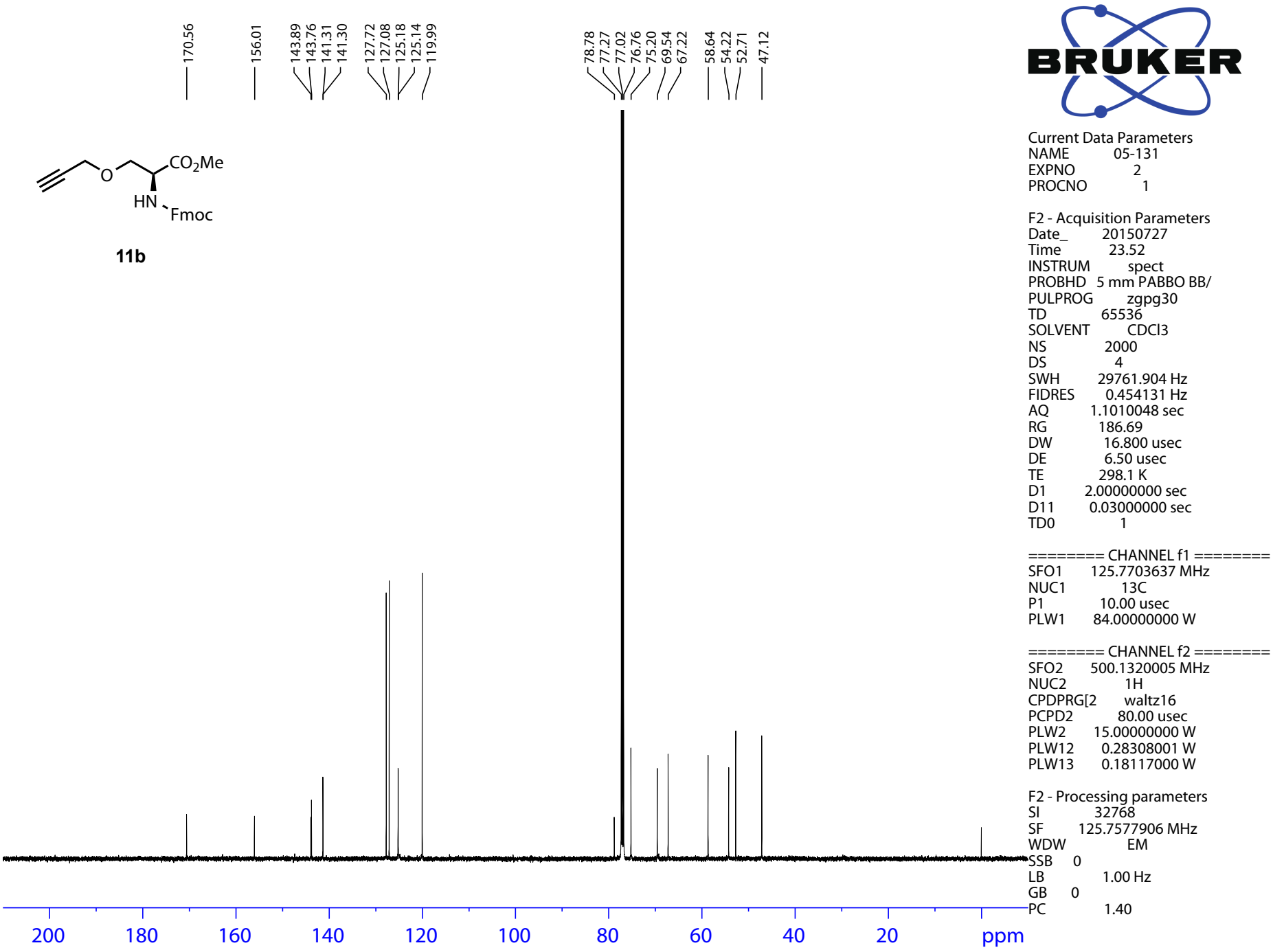


Current Data Parameters
NAME 05-131
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150727
Time 19.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 199998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.050000 Hz
AQ 9.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 05-131
EXPNO 2
PROCNO 1

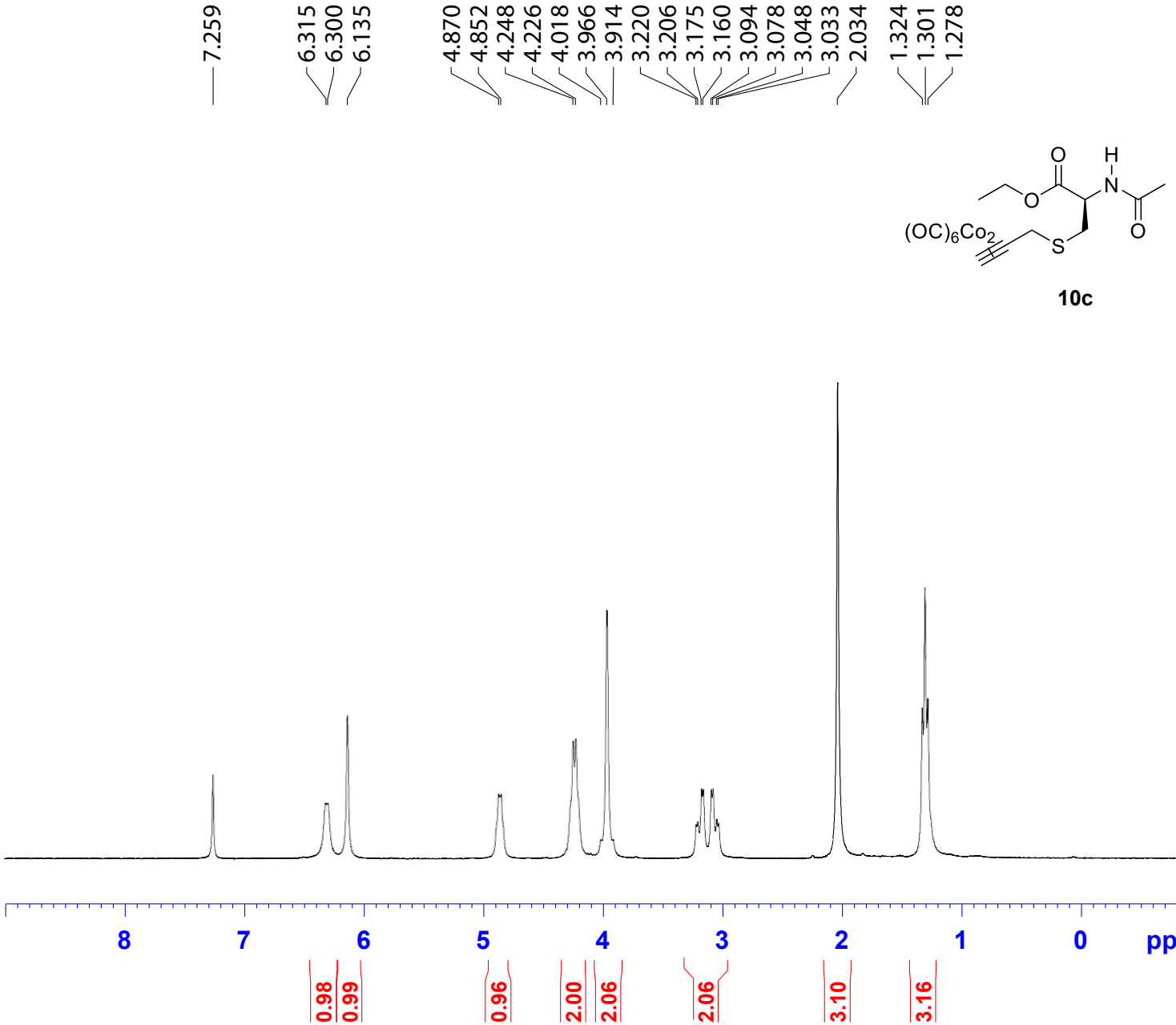
F2 - Acquisition Parameters
Date_ 20150727
Time 23.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

F2 - Processing parameters
SI 32768
SF 125.7577906 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

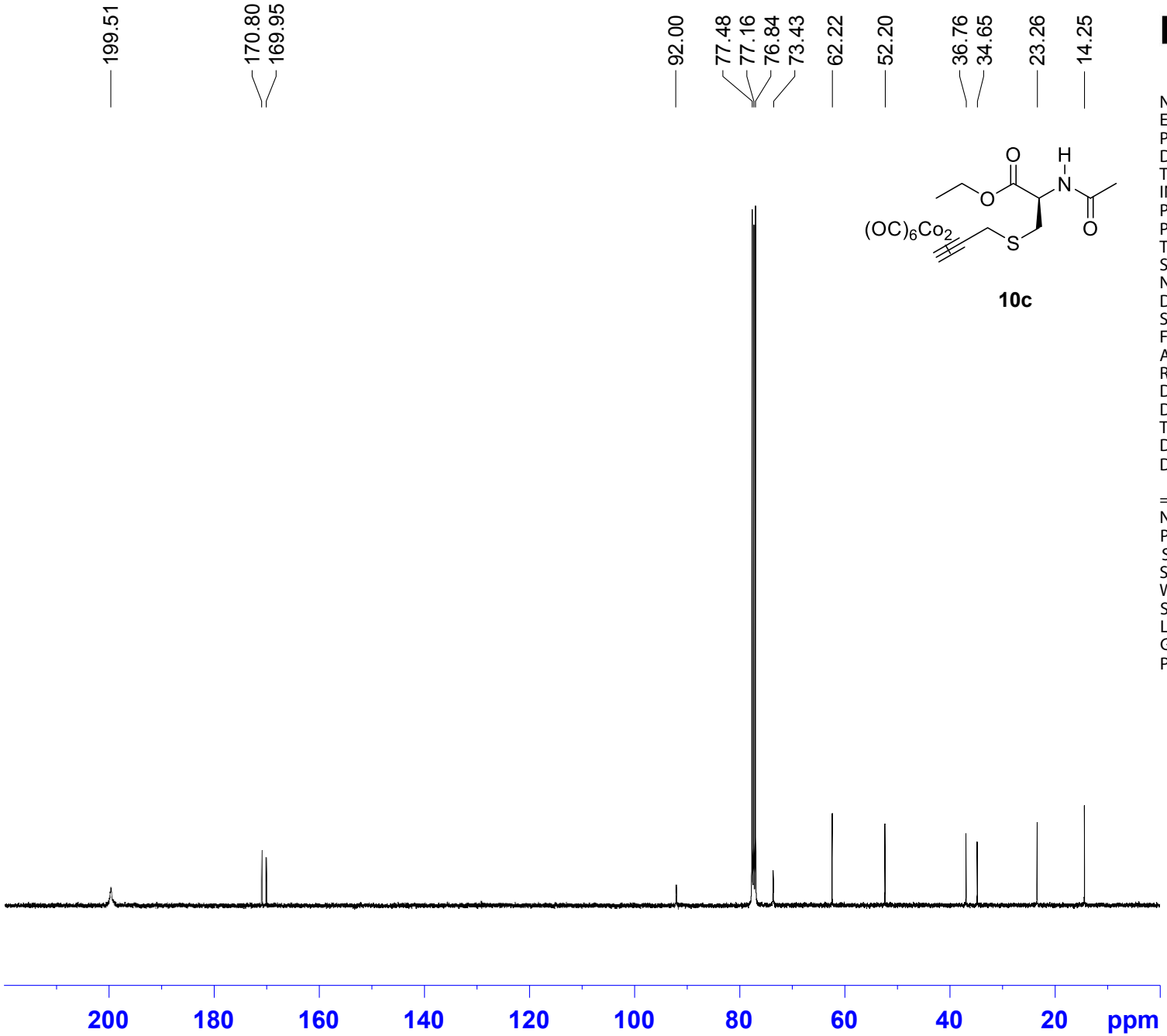
SW06–102–C 1H 300



10c

NAME	SW06–102–C	
EXPNO	10	
PROCNO	1	
Date_	20150213	
Time	16.20	
INSTRUM	spect	
PROBHD	5 mm QNP 1H/1	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	6188.119	Hz
FIDRES	0.188846	Hz
AQ	2.6477044	sec
RG	128	
DW	80.800	usec
DE	6.50	usec
TE	−929.4	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1========		
SFO1	300.2318540	MHz
NUC1	1H	
P1	12.71	usec
SI	32768	
SF	300.2300093	MHz
WDW	EM	
SSB	0	
LB	0.10	Hz
GB	0	
PC	1.00	

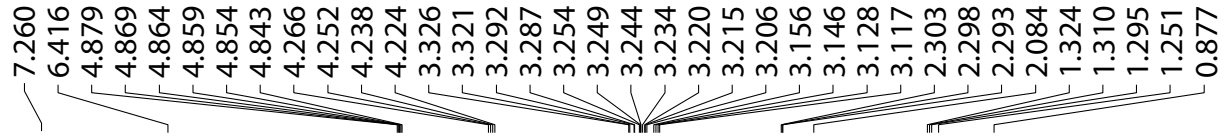
SW06–102–C 13C 400

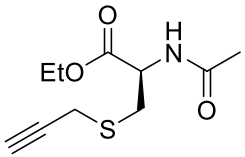


10c

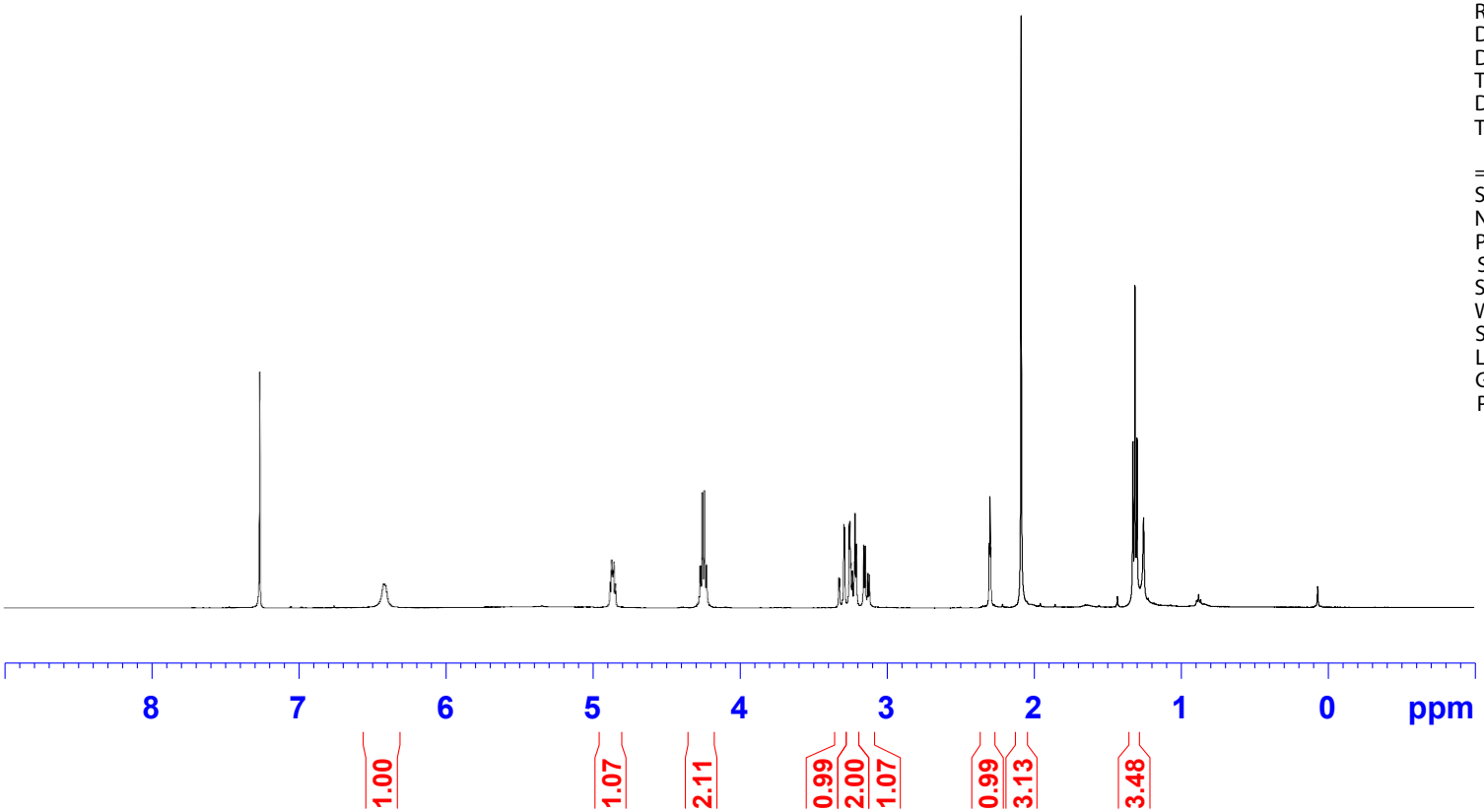
NAME	SW06–102–C
EXPNO	10
PROCNO	1
Date_	20150214
Time	5.45
INSTRUM	spect
PROBHD	5 mm PABBO BB–
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	97.6 K
D1	2.00000000 sec
D11	0.03000000 sec
======== CHANNEL f1========	
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127552 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

SW06–195–cr 1H 500





11c

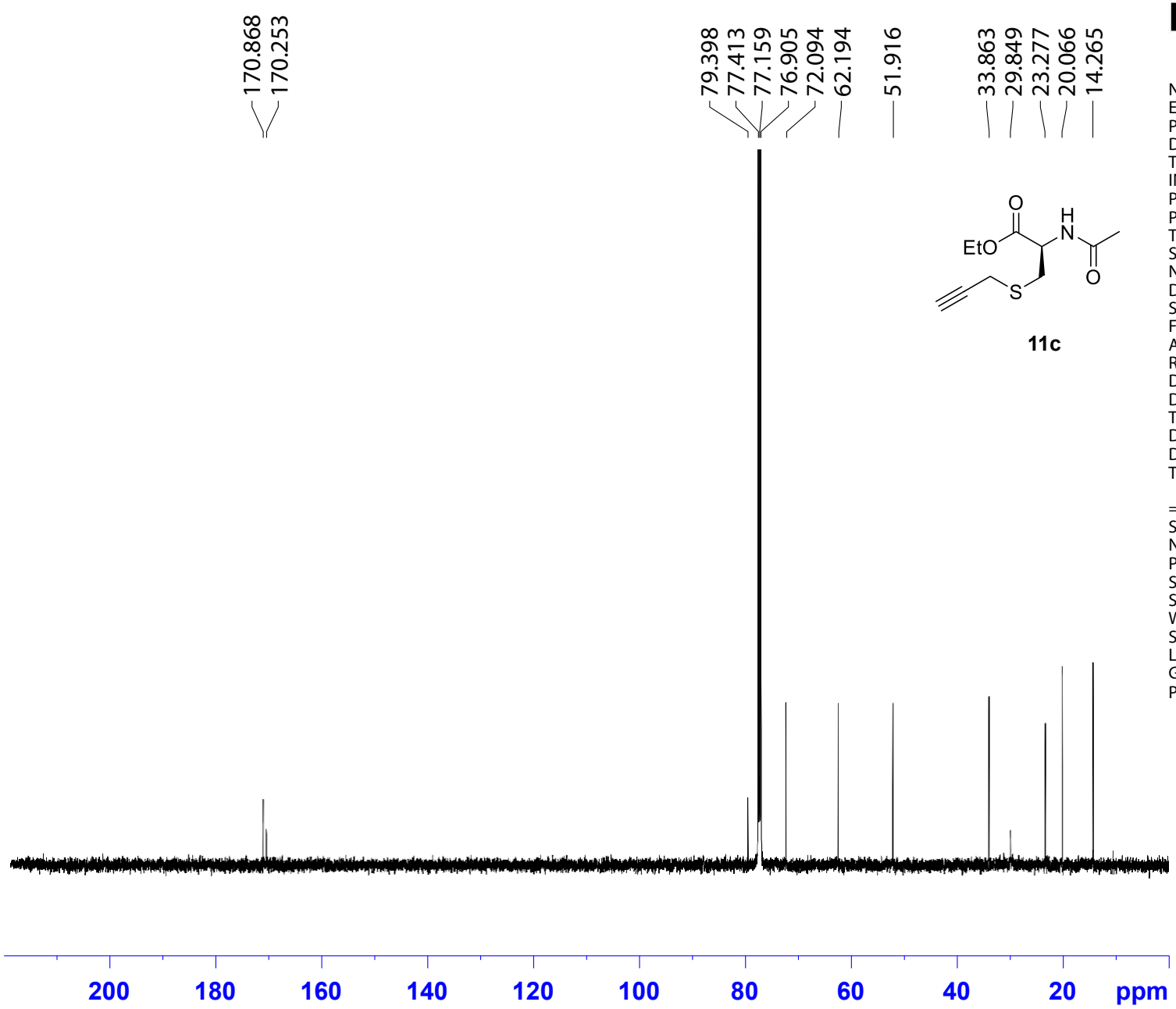


NAME	SW06–195–cr	
EXPNO	10	
PROCNO	1	
Date_	20150513	
Time	17.30	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10000.000	Hz
FIDRES	0.152588	Hz
AQ	3.2768500	sec
RG	203	
DW	50.000	usec
DE	6.50	usec
TE	298.2	K
D1	1.00000000	sec
TD0	1	

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

SFO1	500.1630887	MHz
NUC1	1H	
P1	11.45	usec
SI	65536	
SF	500.1600124	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

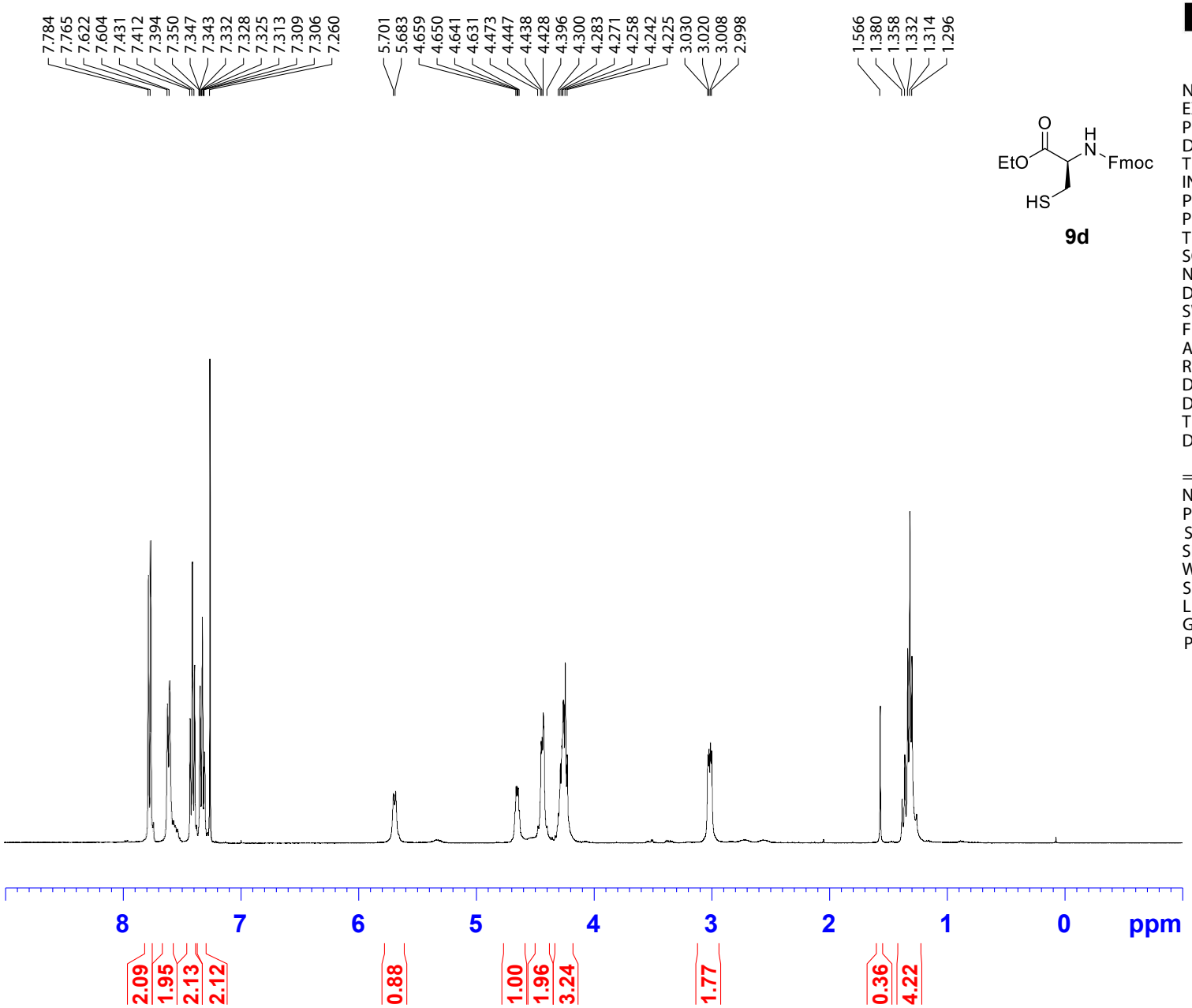
SW06–195–cr 1H 500



11c

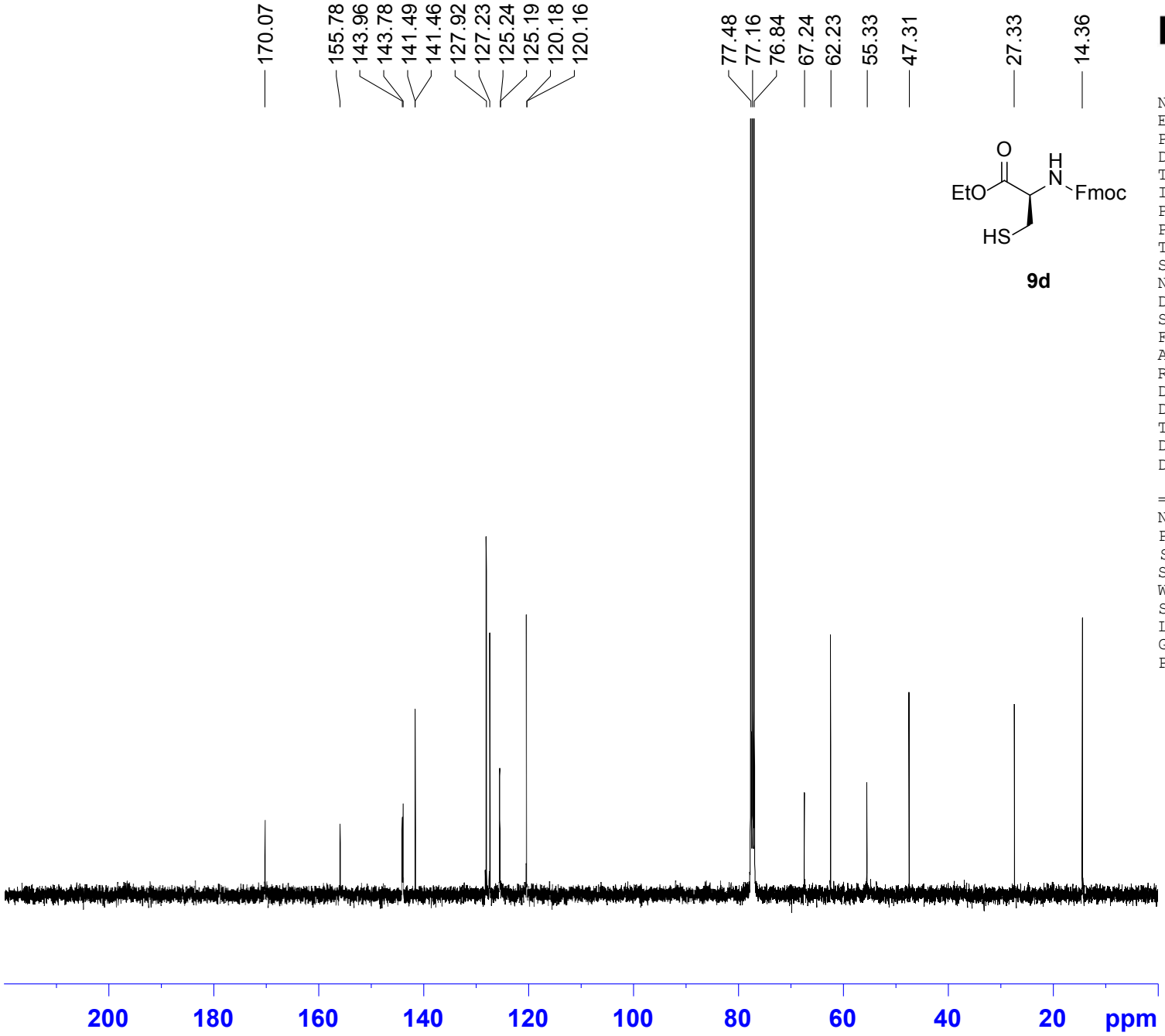
NAME	SW06–195–cr	
EXPNO	11	
PROCNO	1	
Date_	20150515	
Time	0.46	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	298.6	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1========		
SFO1	125.7779086	MHz
NUC1	13C	
P1	10.50	usec
SI	32768	
SF	125.7653129	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

SW06–206–B



NAME	SW06–206–B
EXPNO	20
PROCNO	1
Date_	20150606
Time	14.33
INSTRUM	spect
PROBHD	5 mm PABBO BB–
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	114
DW	60.800 usec
DE	6.50 usec
TE	97.1 K
D1	1.00000000 sec
======== CHANNEL f1========	
NUC1	1H
P1	13.75 usec
SI	65536
SF	400.1300102 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

SW06-206-B 13C 400

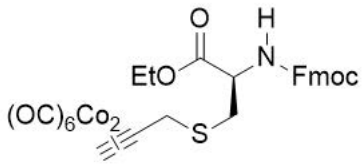


9d

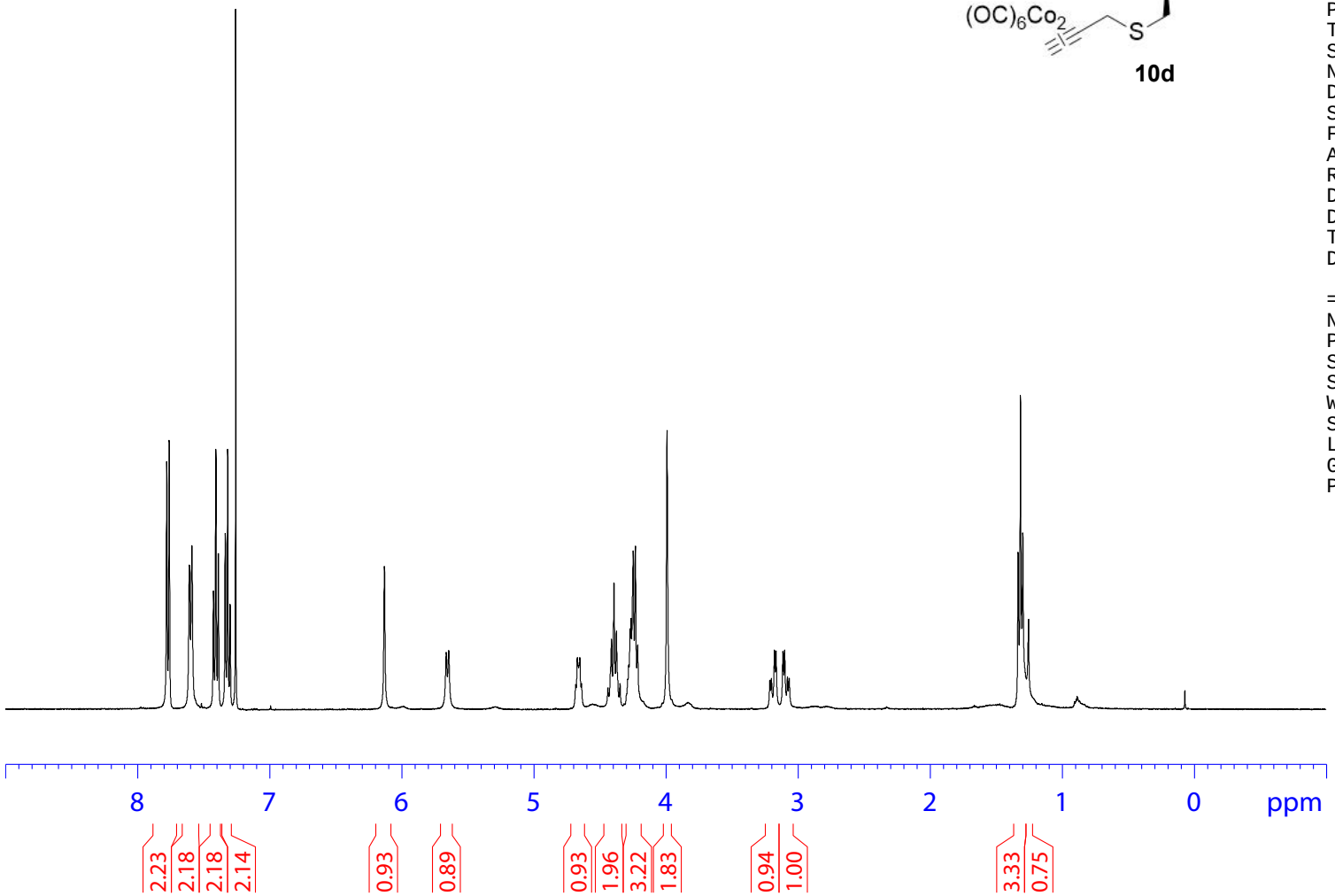
NAME	SW06-206-B
EXPNO	21
PROCNO	1
Date_	20150607
Time	23.17
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	161
DW	20.800 usec
DE	6.50 usec
TE	93.6 K
D1	2.00000000 sec
D11	0.03000000 sec
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127562 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

SW06–138–C 1H 400

7.782 7.763 7.610 7.591 7.428 7.410 7.391 7.339 7.321 7.302 7.260 6.134 5.666 5.647 4.685 4.673 4.654 4.641 4.441 4.415 4.395 4.376 4.350 4.283 4.274 4.266 4.250 4.233 4.216 3.993 3.215 3.203 3.180 3.168 3.116 3.103 3.081 3.068 1.335 1.317 1.299 1.256 0.888 0.072

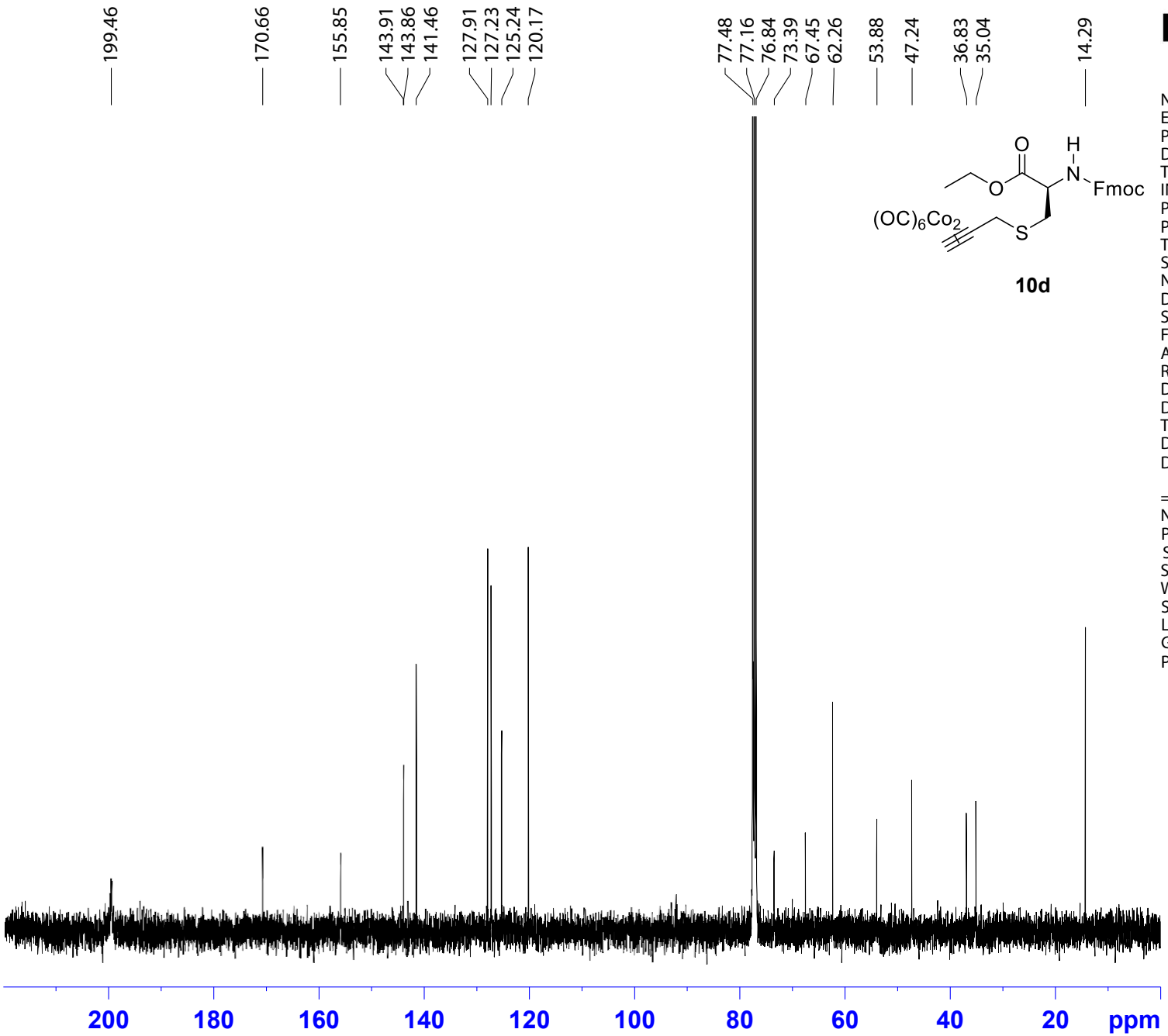


10d




```
NAME                SW06-138-C
EXPNO                       10
PROCNO                       1
Date_                 20150331
Time                     18.10
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                        128
DW                     60.800 usec
DE                      6.50 usec
TE                      91.6 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.75 usec
SI                      65536
SF              400.1300104 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                     1.00
```

SW06–138–C 13C 400



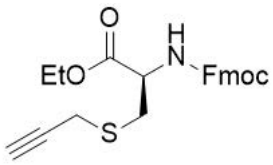
10d

NAME	SW06–138–C	
EXPNO	11	
PROCNO	1	
Date_	20150401	
Time	1.15	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	96.9	K
D1	2.00000000	sec
D11	0.03000000	sec

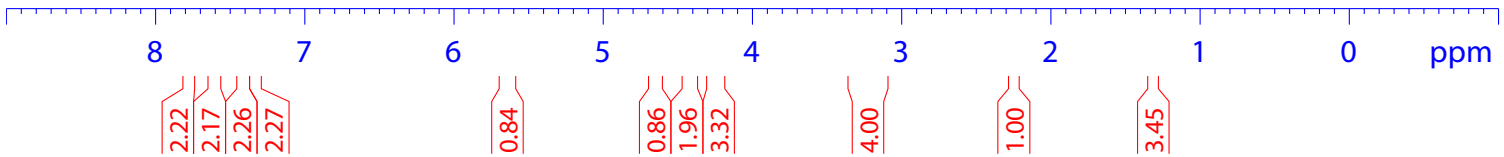
======== CHANNEL f1	========	
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127549	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

SW06–164–cr 1H 400

7.761 7.618 7.602 7.426 7.408 7.389 7.344 7.341 7.338 7.325 7.322 7.319 7.307 7.304 7.301 7.260 5.645 5.626 4.670 4.657 4.650 4.644 4.637 4.624 4.458 4.433 4.425 4.414 4.409 4.382 4.278 4.260 4.242 4.225 3.320 3.283 3.277 3.260 3.255 3.239 3.227 3.218 3.203 3.191 3.168 3.154 3.133 3.119 2.261 2.255 2.248 1.856 1.432 1.329 1.311 1.293 1.251 1.223 0.880 0.070



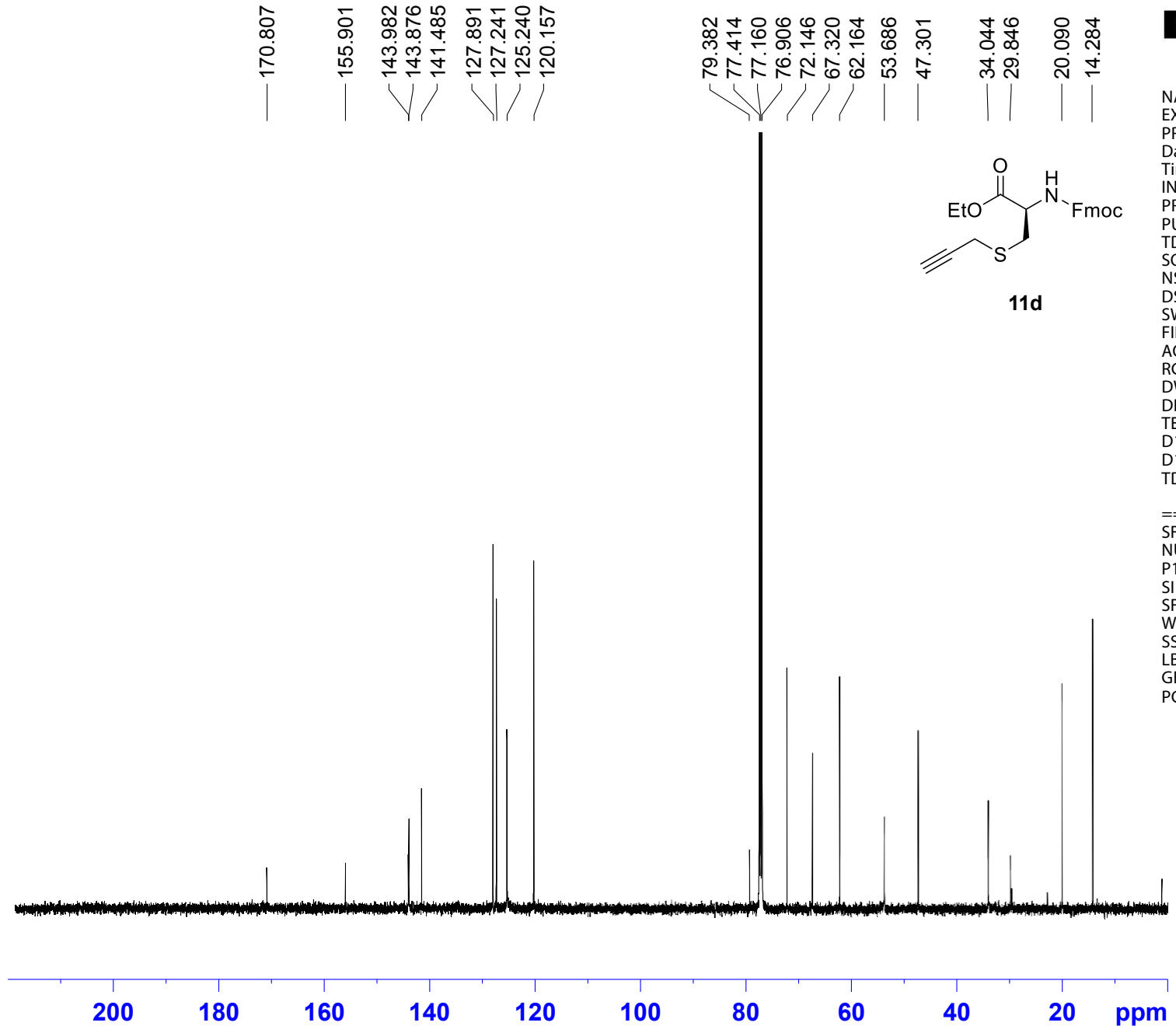
11d



2.22 2.17 2.26 2.27 0.84 0.86 1.96 3.32 4.00 1.00 3.45

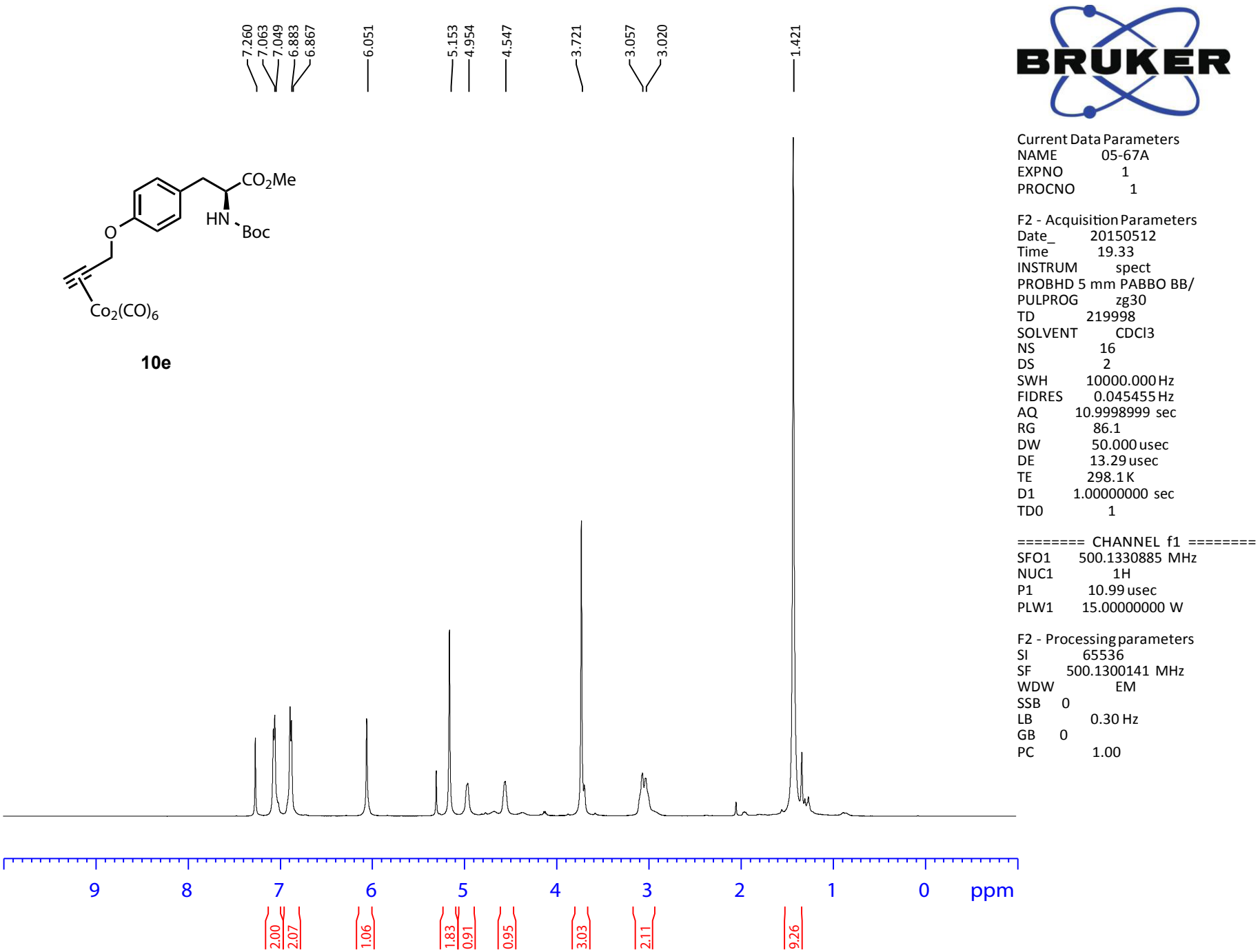
```
NAME            SW06-164-cr
EXPNO                    10
PROCNO                    1
Date_              20150415
Time                  16.06
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      161
DW                 60.800 usec
DE                   6.50 usec
TE                    90.5 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                  13.75 usec
SI                    65536
SF            400.1300100 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                    1.00
```

SW06–164 cr 13 C 500



11d

NAME	SW06–164 cr	
EXPNO	11	
PROCNO	1	
Date_	20150512	
Time	6.40	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	298.6	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	125.7779086	MHz
NUC1	13C	
P1	10.50	usec
SI	32768	
SF	125.7653142	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

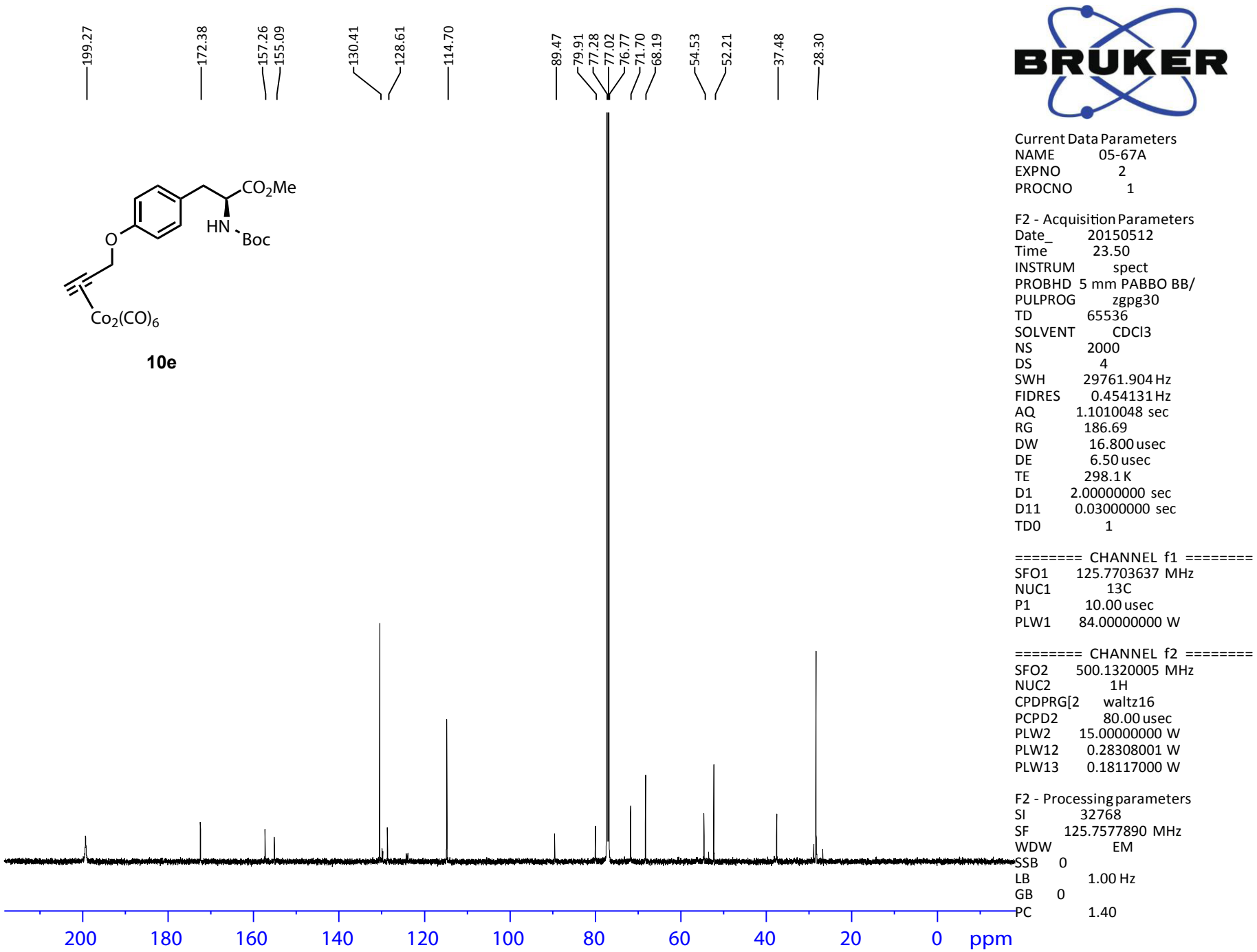


Current Data Parameters
NAME 05-67A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150512
Time 19.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 219998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.045455 Hz
AQ 10.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 05-67A
EXPNO 2
PROCNO 1

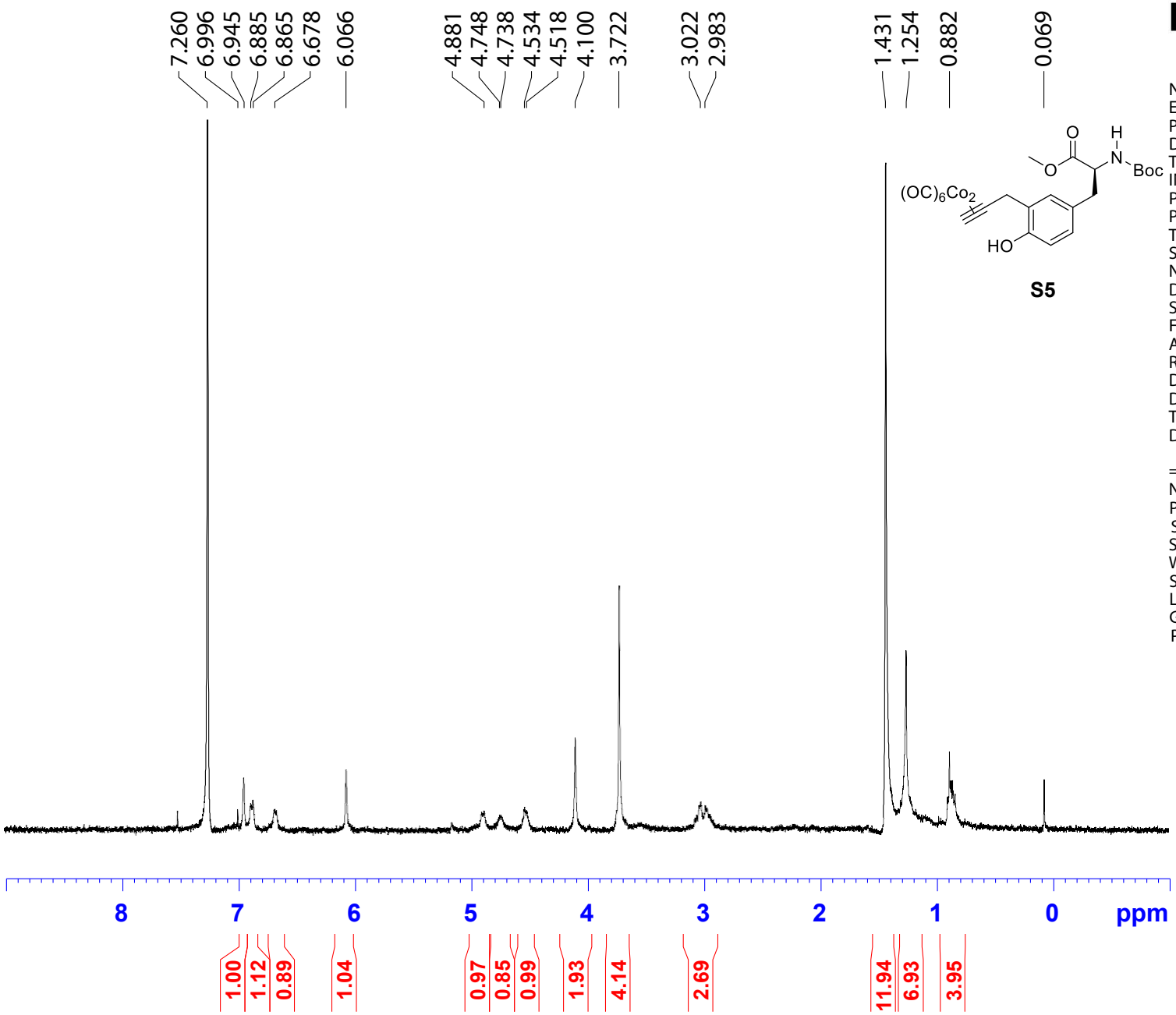
F2 - Acquisition Parameters
Date_ 20150512
Time 23.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

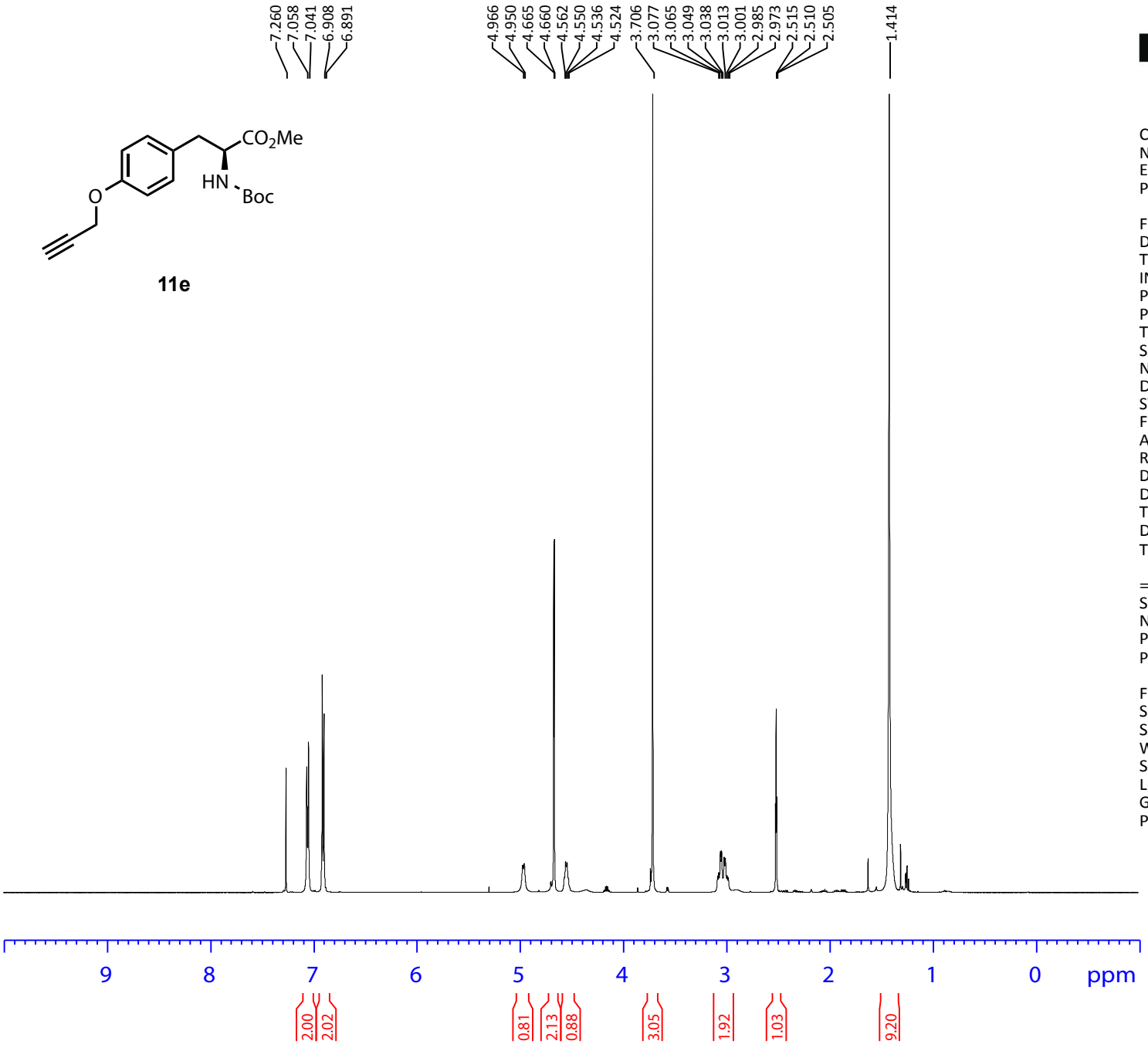
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SW06–166–D 1H 400



S5

NAME	SW06–166–D	
EXPNO	10	
PROCNO	1	
Date_	20150421	
Time	17.09	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	181	
DW	60.800	usec
DE	6.50	usec
TE	94.1	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300096	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

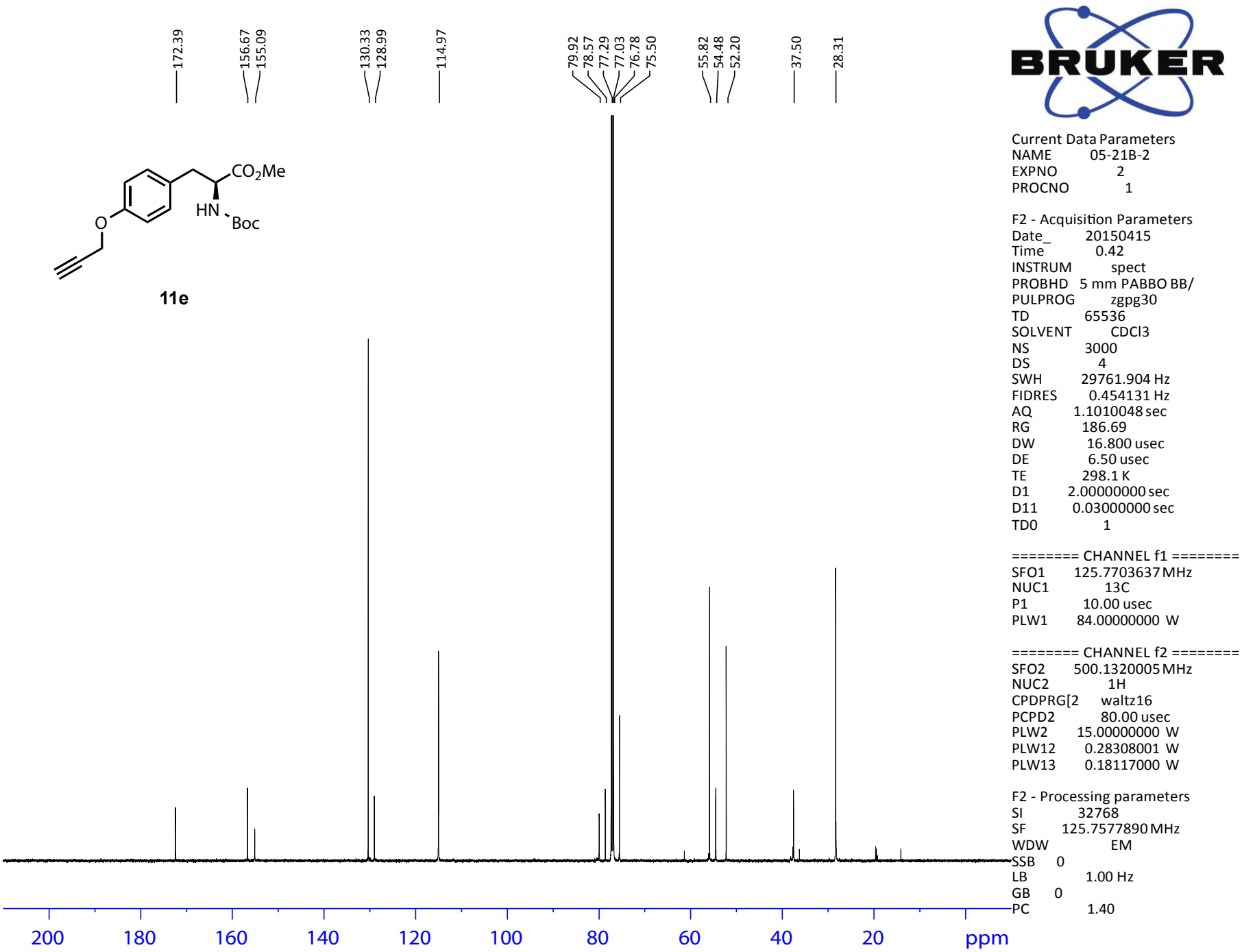


Current Data Parameters
NAME 05-21B-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150414
Time 14.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 219998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.045455 Hz
AQ 10.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300136 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



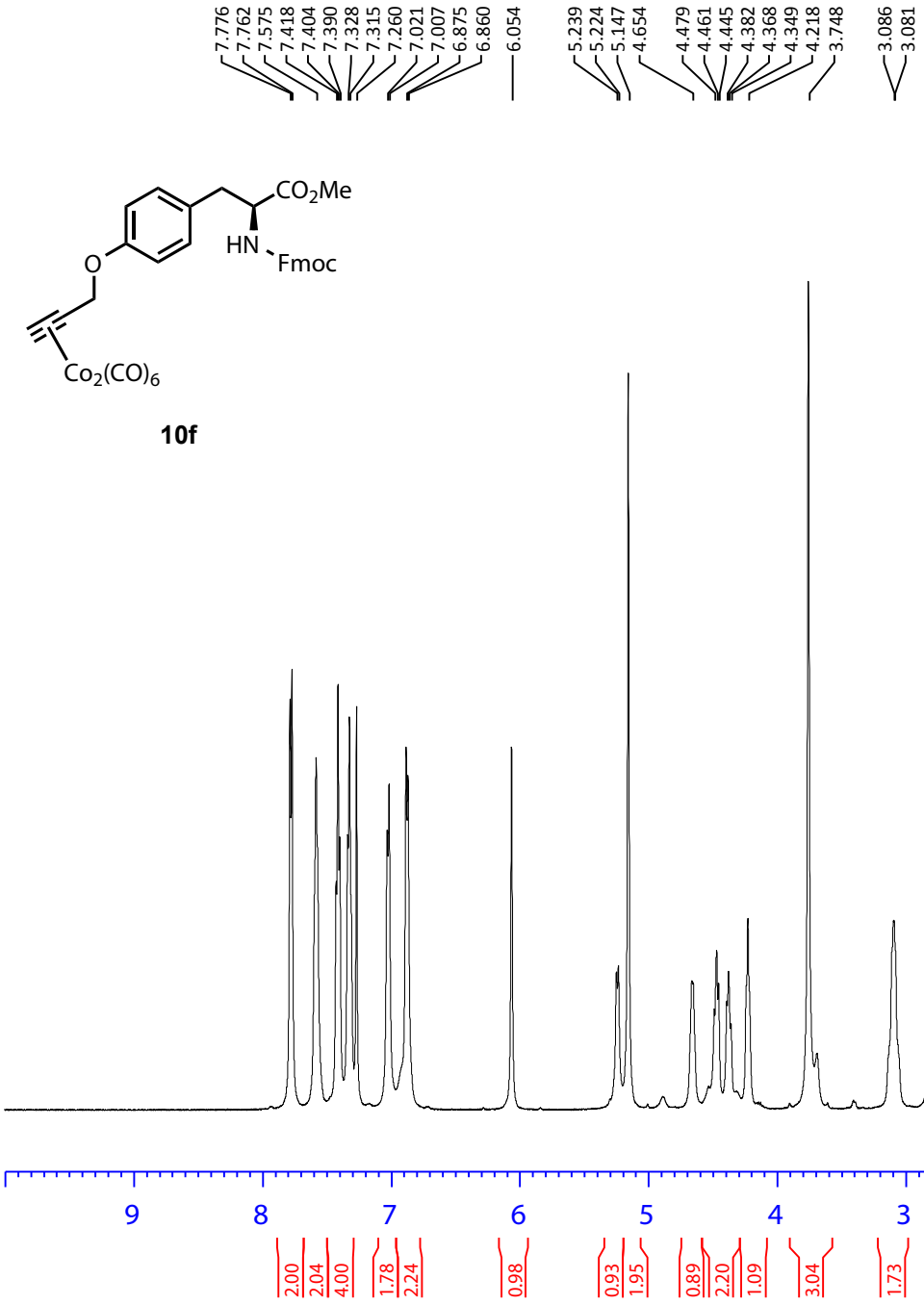
Current Data Parameters
NAME 05-21B-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150415
Time 0.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

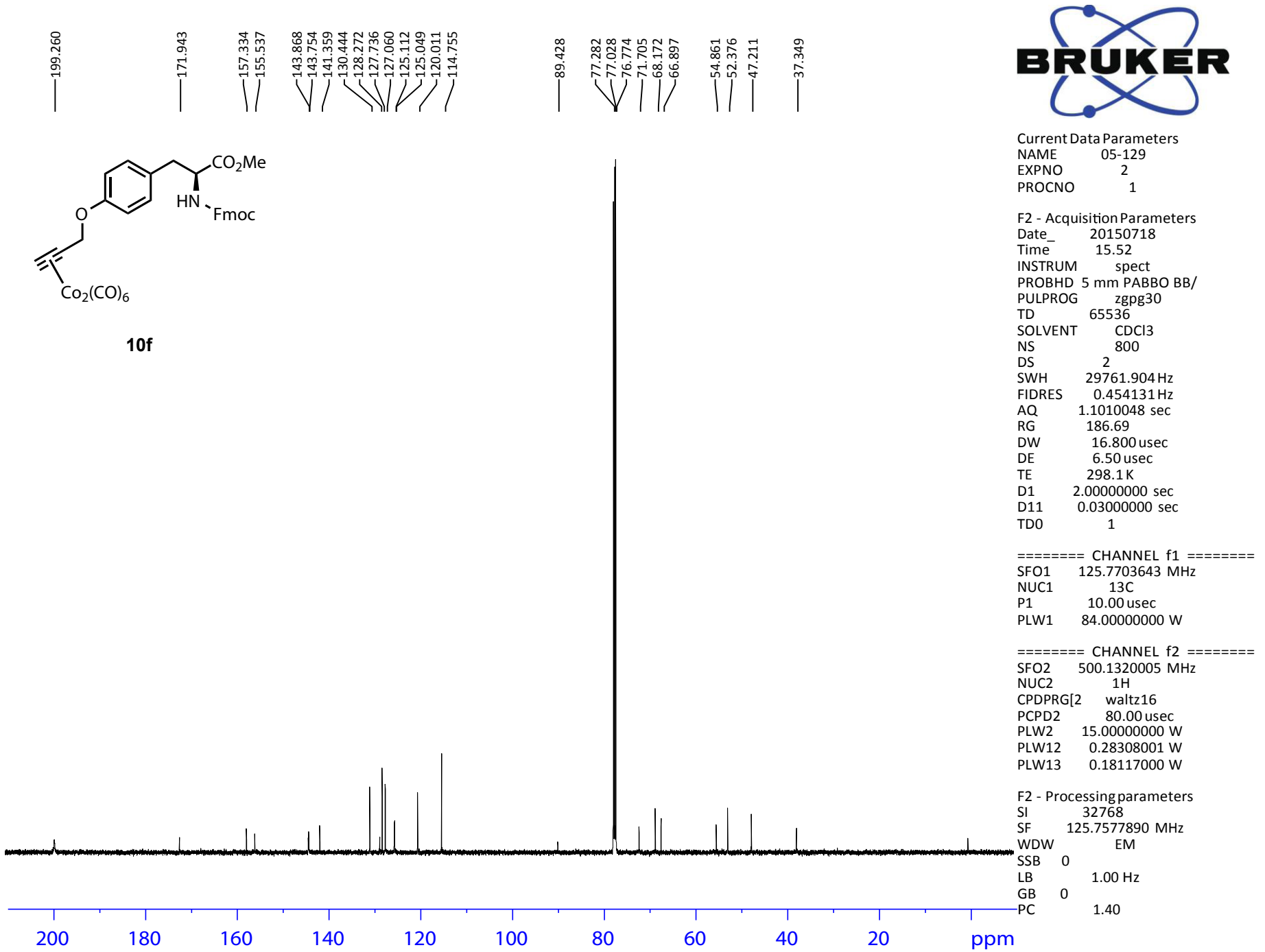


Current Data Parameters
NAME 05-129
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150718
Time 14.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 148.37
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300152 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



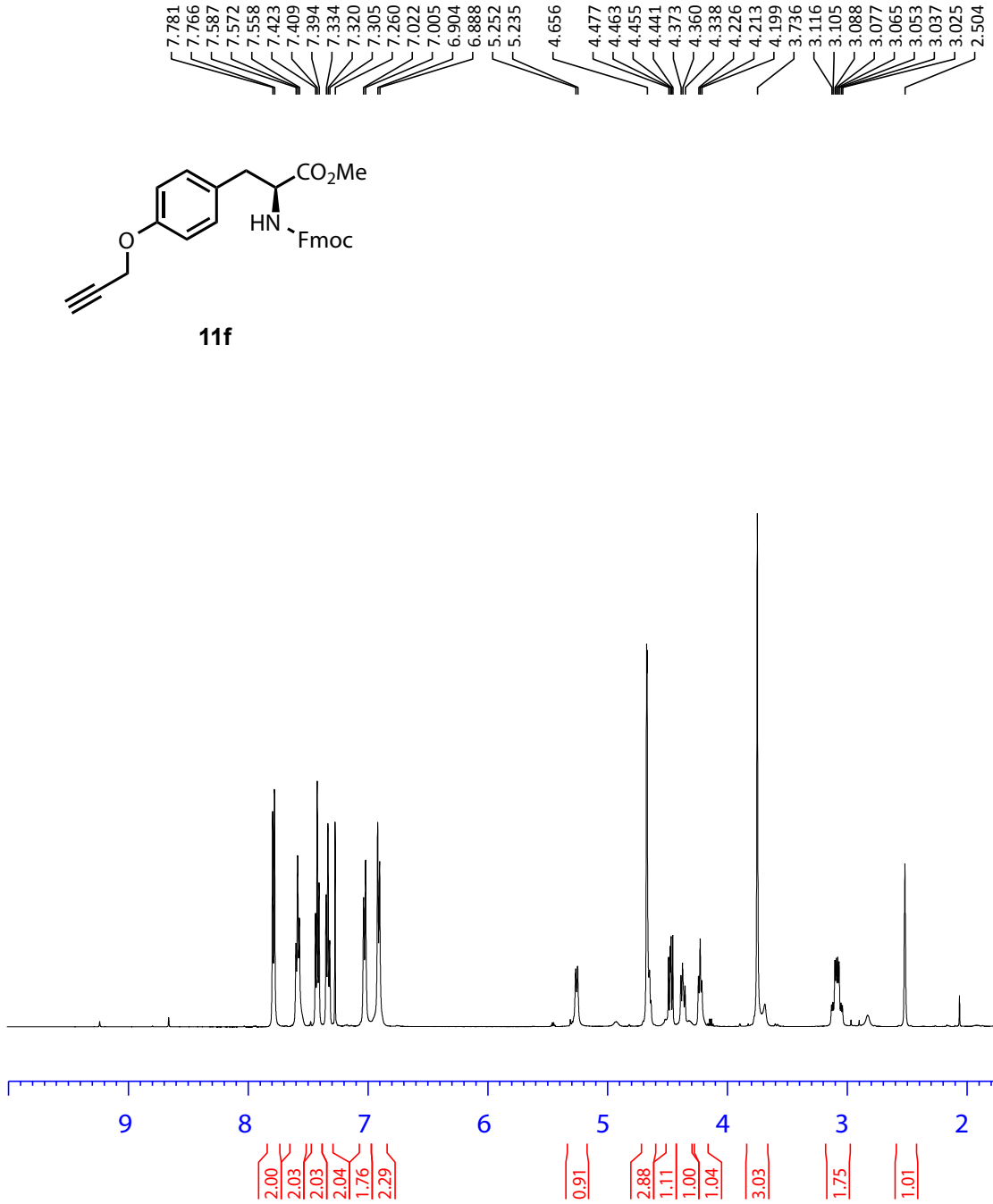
Current Data Parameters
NAME 05-129
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150718
Time 15.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703643 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

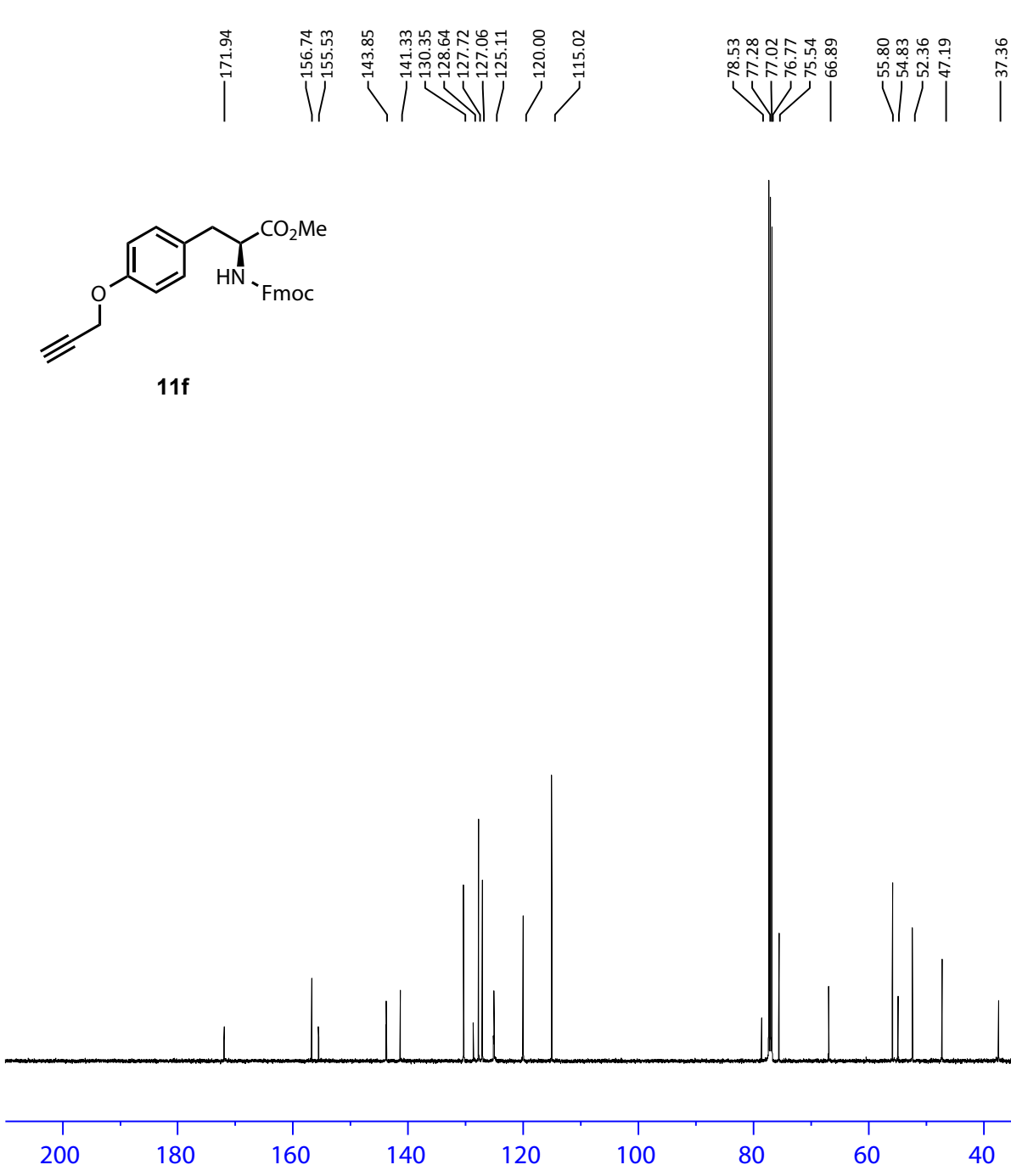


Current Data Parameters
NAME 05-133
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150728
Time 14.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 199998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.050000 Hz
AQ 9.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 05-133
EXPNO 2
PROCNO 1

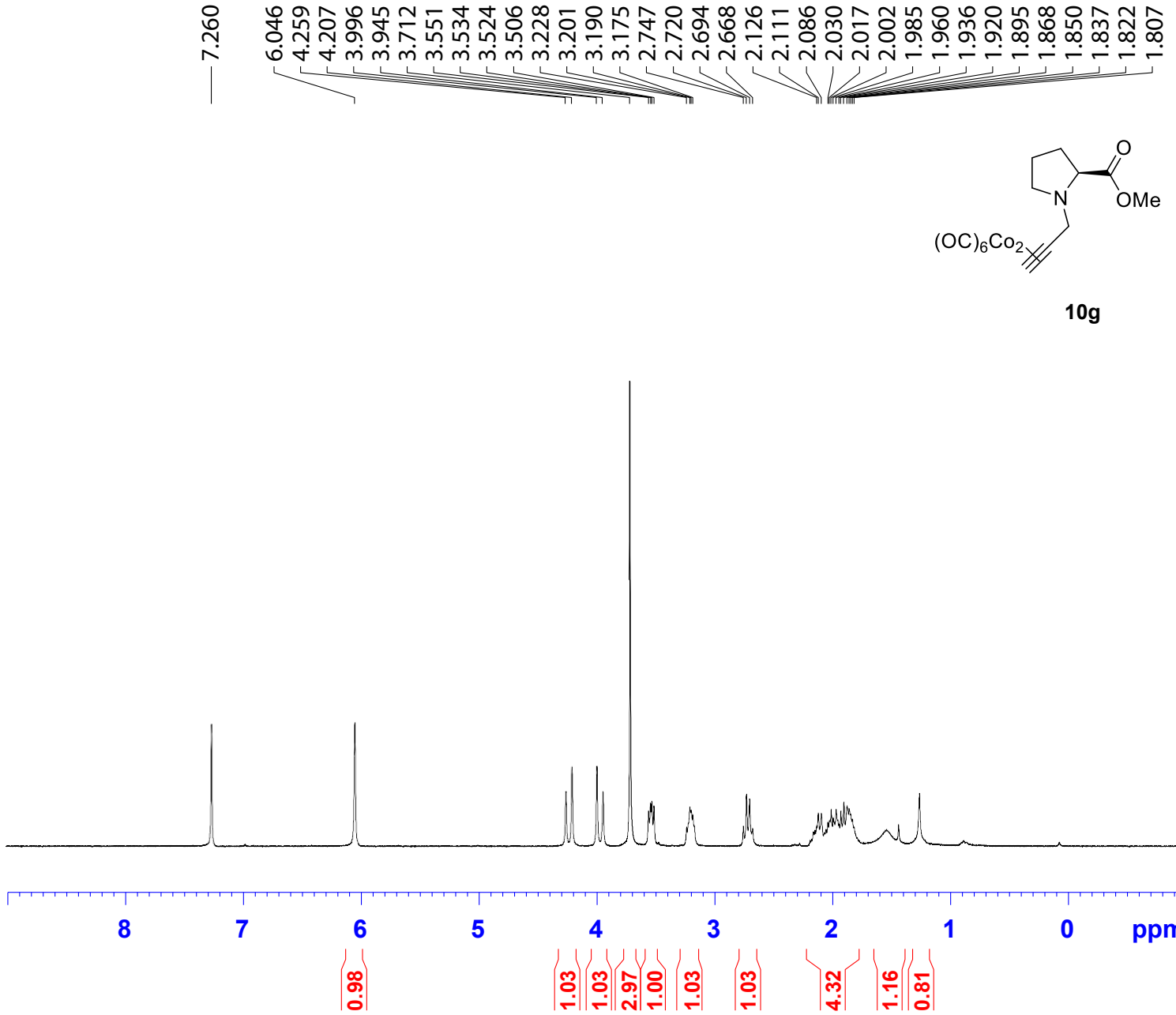
F2 - Acquisition Parameters
Date_ 20150728
Time 23.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 186.69
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.00000000 W
PLW12 0.28308001 W
PLW13 0.18117000 W

F2 - Processing parameters
SI 32768
SF 125.7577914 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

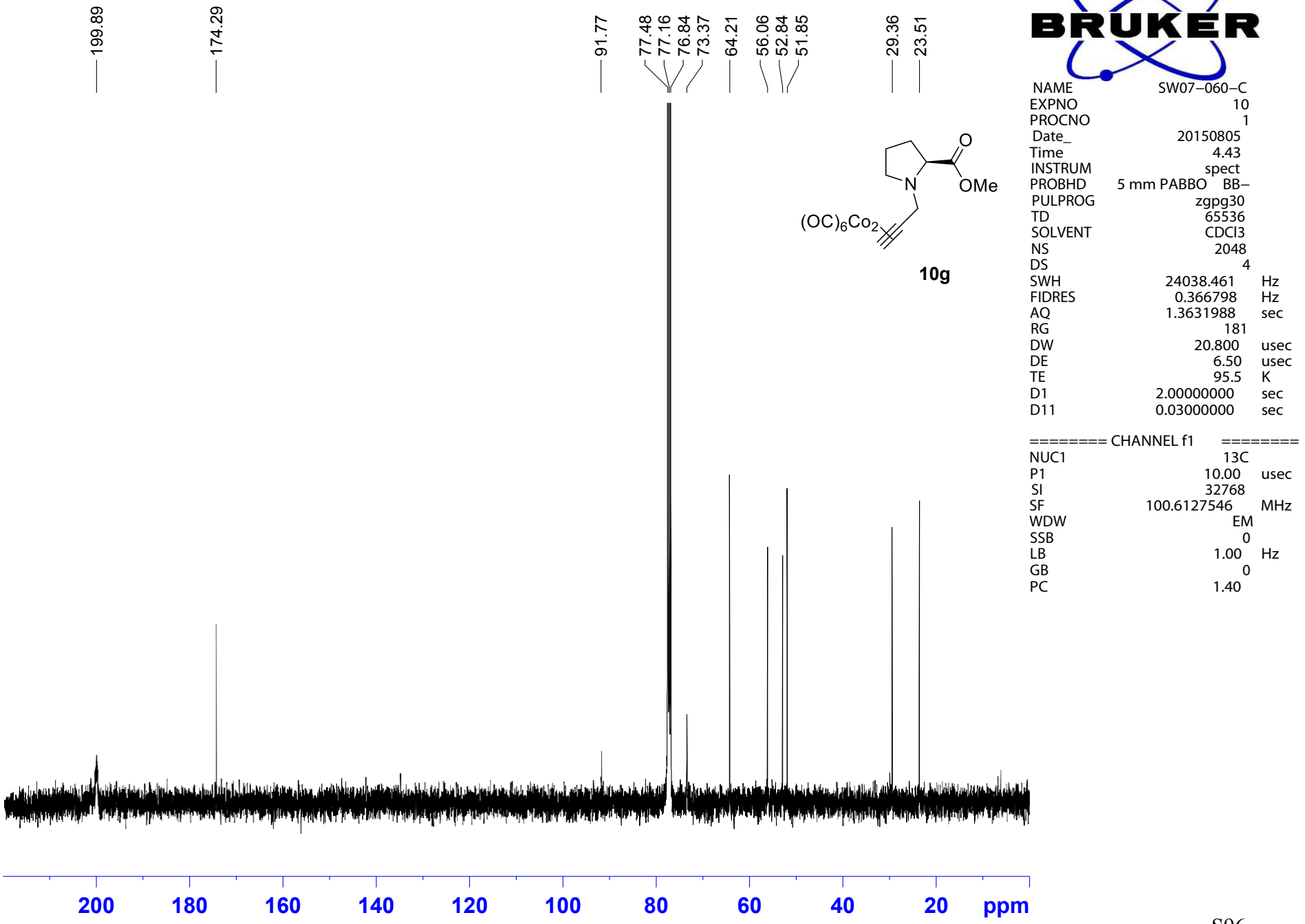
SW07–060–C



10g

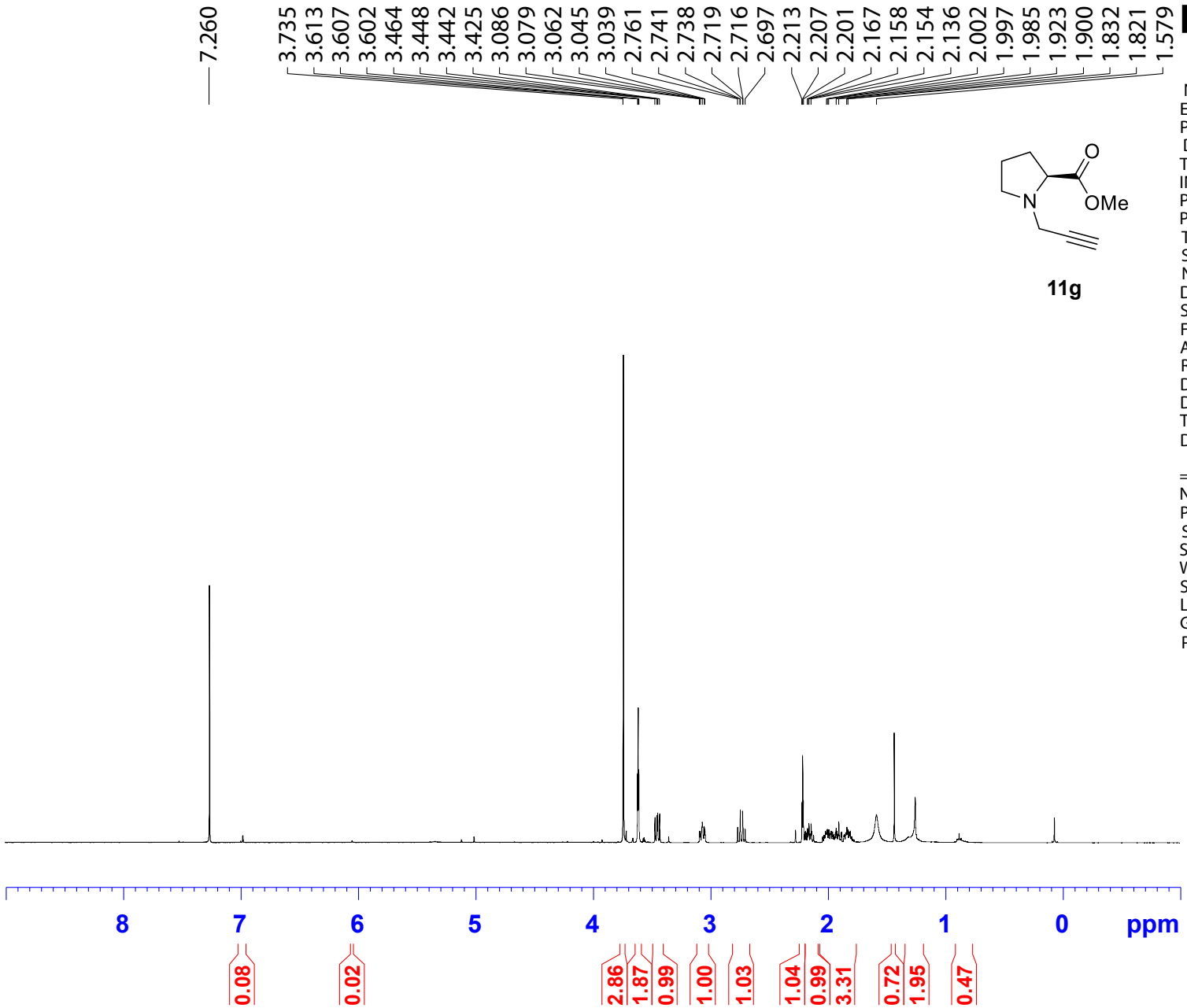
NAME	SW07–060–C	
EXPNO	10	
PROCNO	1	
Date_	20150804	
Time	18.03	
INSTRUM	spect	
PROBHD 5 mm QNP	1H/1	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	6188.1 19	Hz
FIDRES	0.188846	Hz
AQ	2.6477044	sec
RG	322	
DW	80.800	usec
DE	6.50	usec
TE	−922.2	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	300.2318540	MHz
NUC1	1H	
P1	12.71	usec
SI	32768	
SF	300.2300085	MHz
WDW	EM	
SSB	0	
LB	0.10	Hz
GB	0	
PC	1.00	

SW07−060−C 13C 400



NAME	SW07−060−C	
EXPNO	10	
PROCNO	1	
Date_	20150805	
Time	4.43	
INSTRUM	spect	
PROBHD	5 mm PABBO BB−	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	181	
DW	20.800	usec
DE	6.50	usec
TE	95.5	K
D1	2.00000000	sec
D11	0.03000000	sec
======== CHANNEL f1	========	
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127546	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

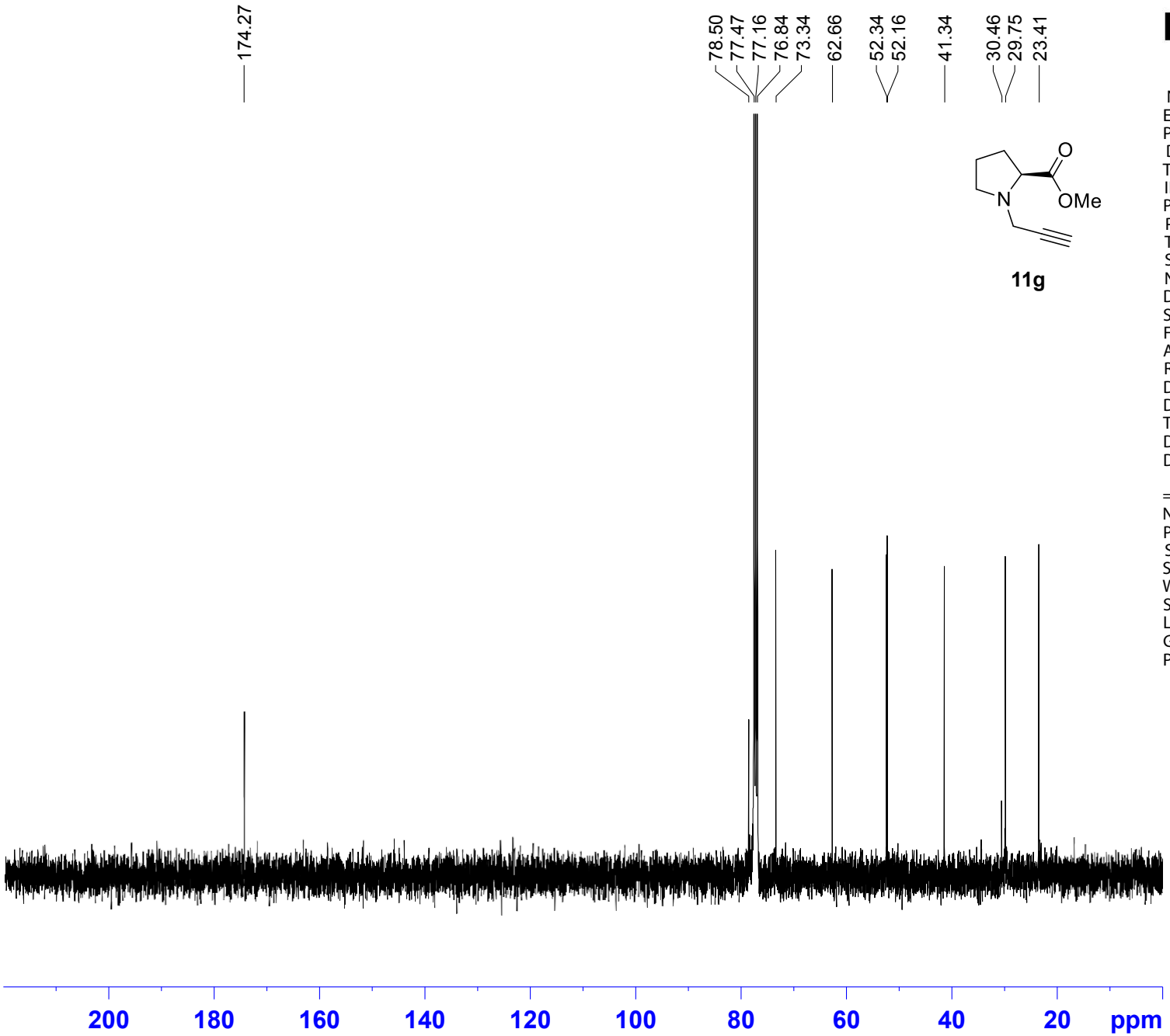
SW07–074–cr



11g

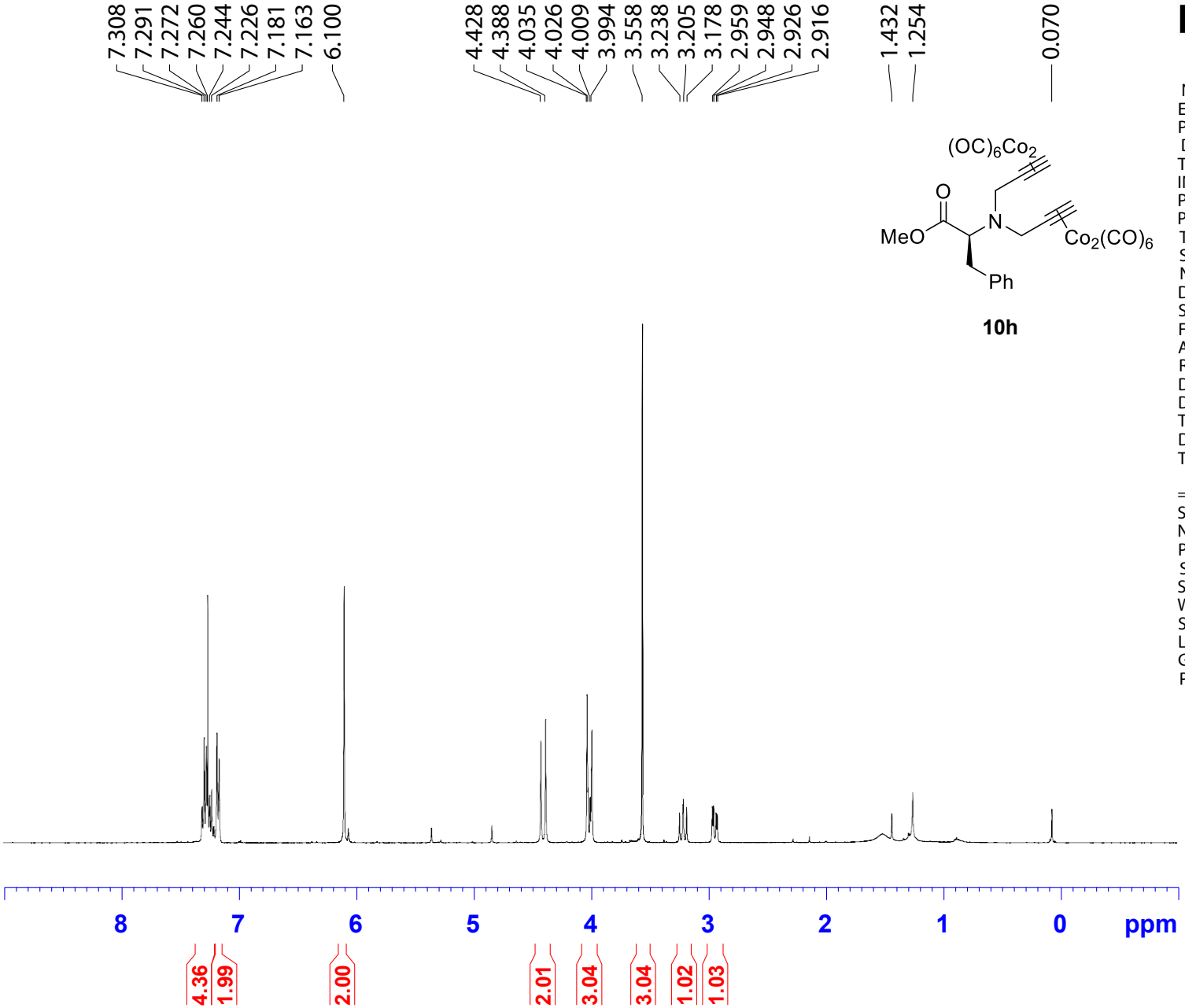
NAME	SW07–074–cr	
EXPNO	10	
PROCNO	1	
Date_	20150814	
Time	17.03	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	144	
DW	60.800	usec
DE	6.50	usec
TE	93.5	K
D1	1.00000000	sec
======== CHANN EL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300099	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW07–074–cr 13C 400



NAME	SW07–074–cr	
EXPNO	11	
PROCNO	1	
Date_	20150816	
Time	1.33	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	144	
DW	20.800	usec
DE	6.50	usec
TE	95.0	K
D1	2.00000000	sec
D11	0.03000000	sec
======== CHANNEL f1	========	
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100 .6127555	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

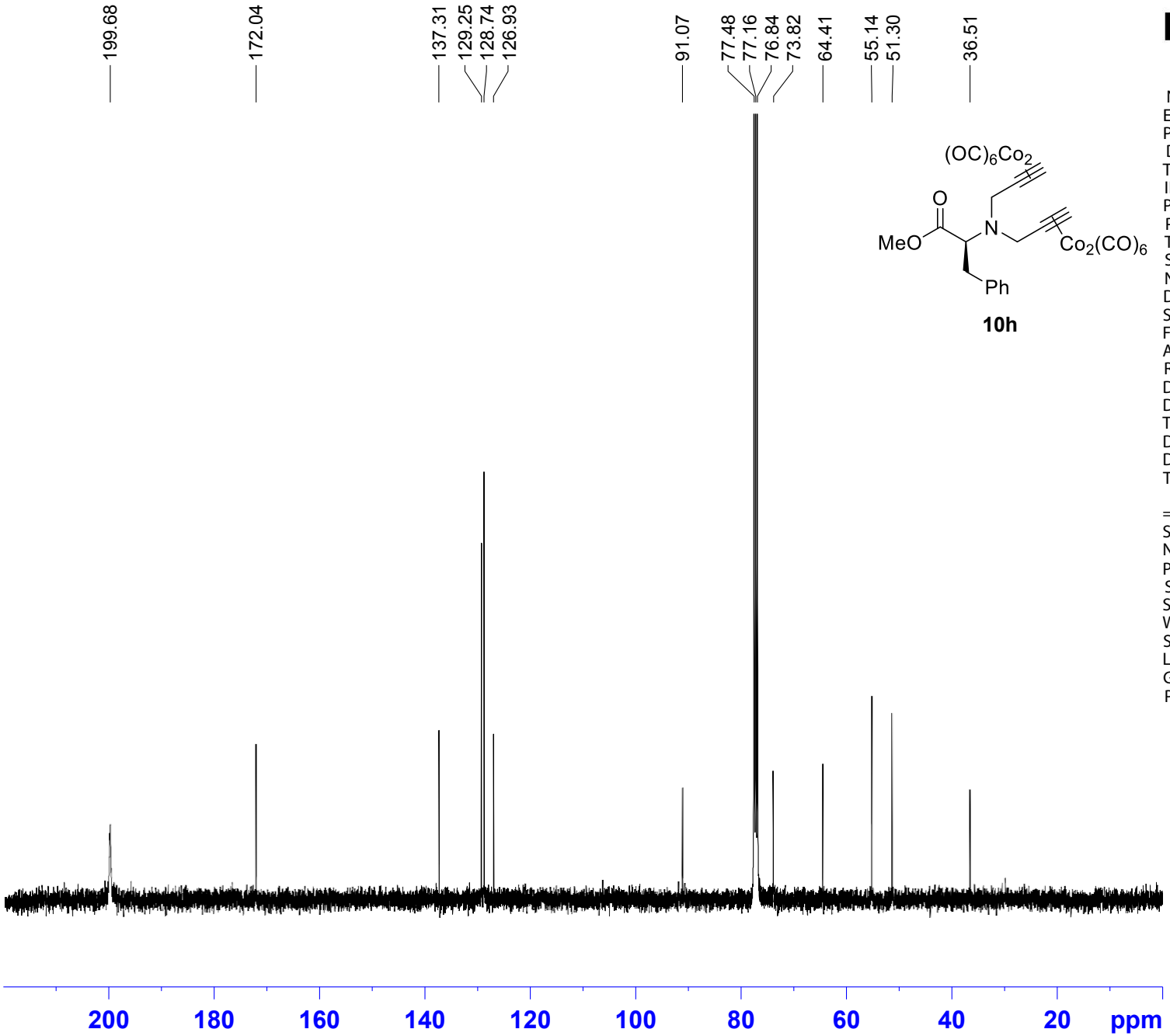
SW07–111–B 1H 400



10h

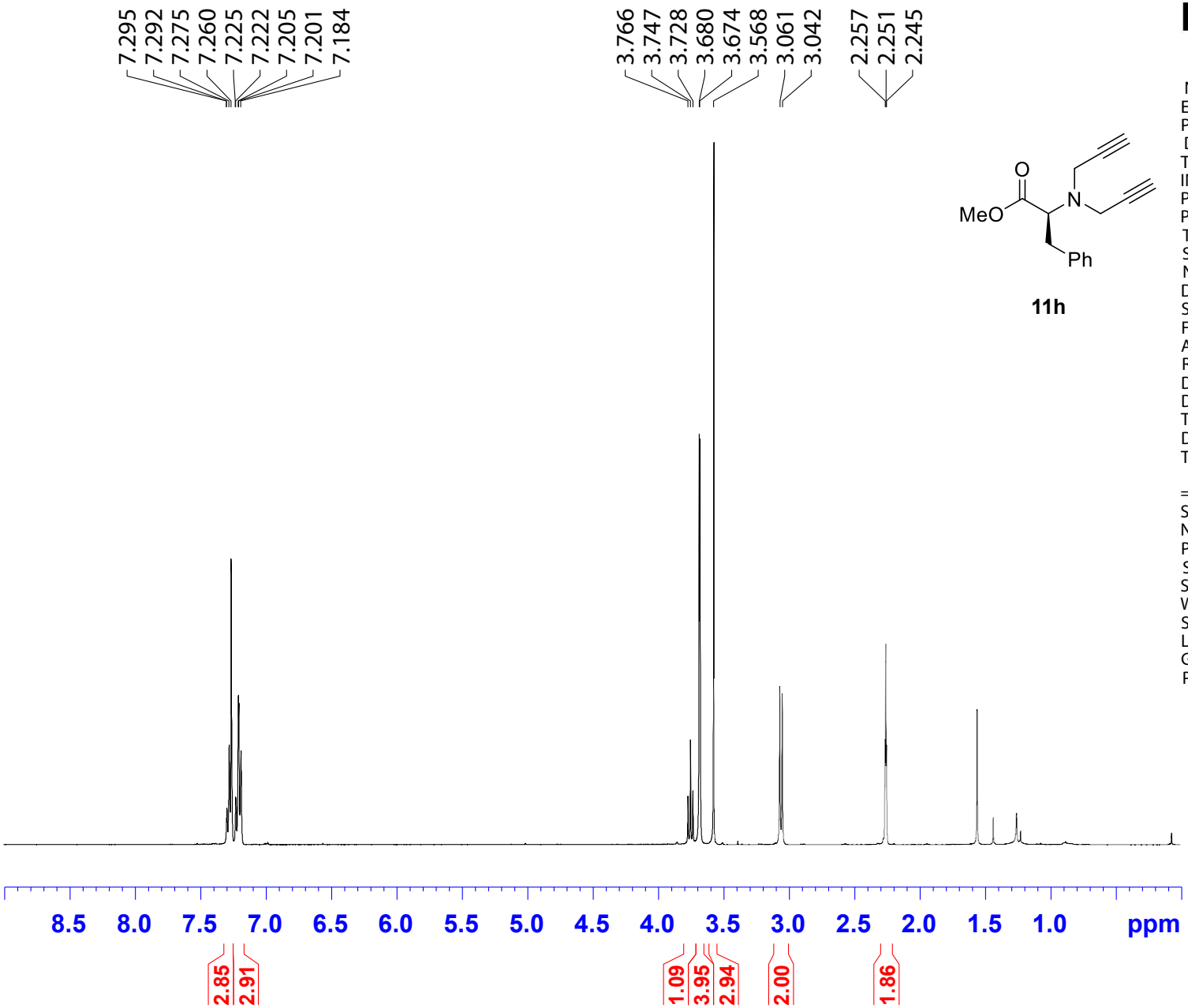
NAME	SW07–111–B	
EXPNO	10	
PROCNO	1	
Date_	20151013	
Time	14.03	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894966	sec
RG	144	
DW	62.400	usec
DE	6.50	usec
TE	96.7	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	400.1324710	MHz
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300107	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW07–111–B 1H 400



NAME	SW07–111–B	
EXPNO	11	
PROCNO	1	
Date_	20151014	
Time	3.34	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3072	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	97.8	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	100.6228293	MHz
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127541	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

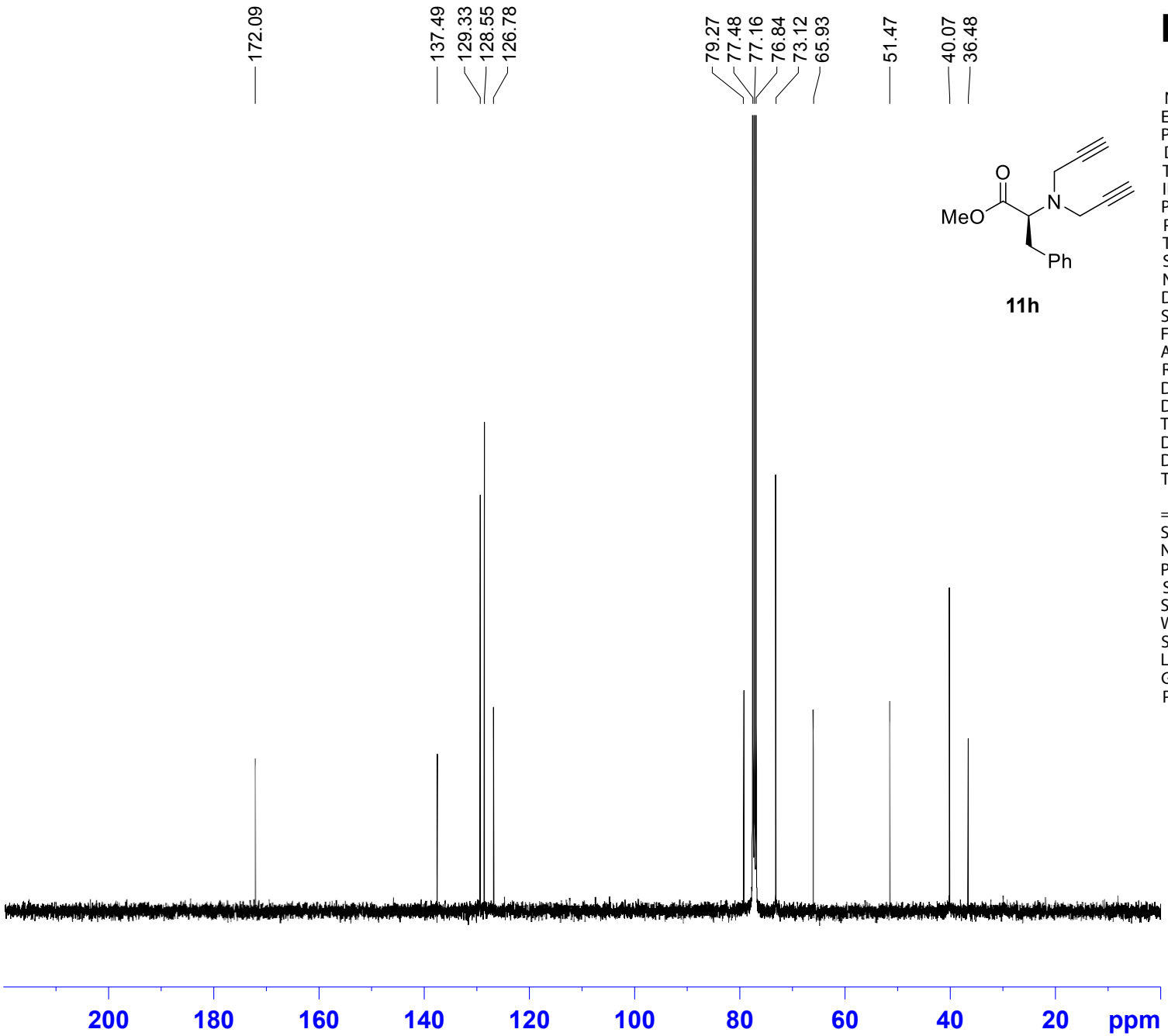
SW07–118–A



11h

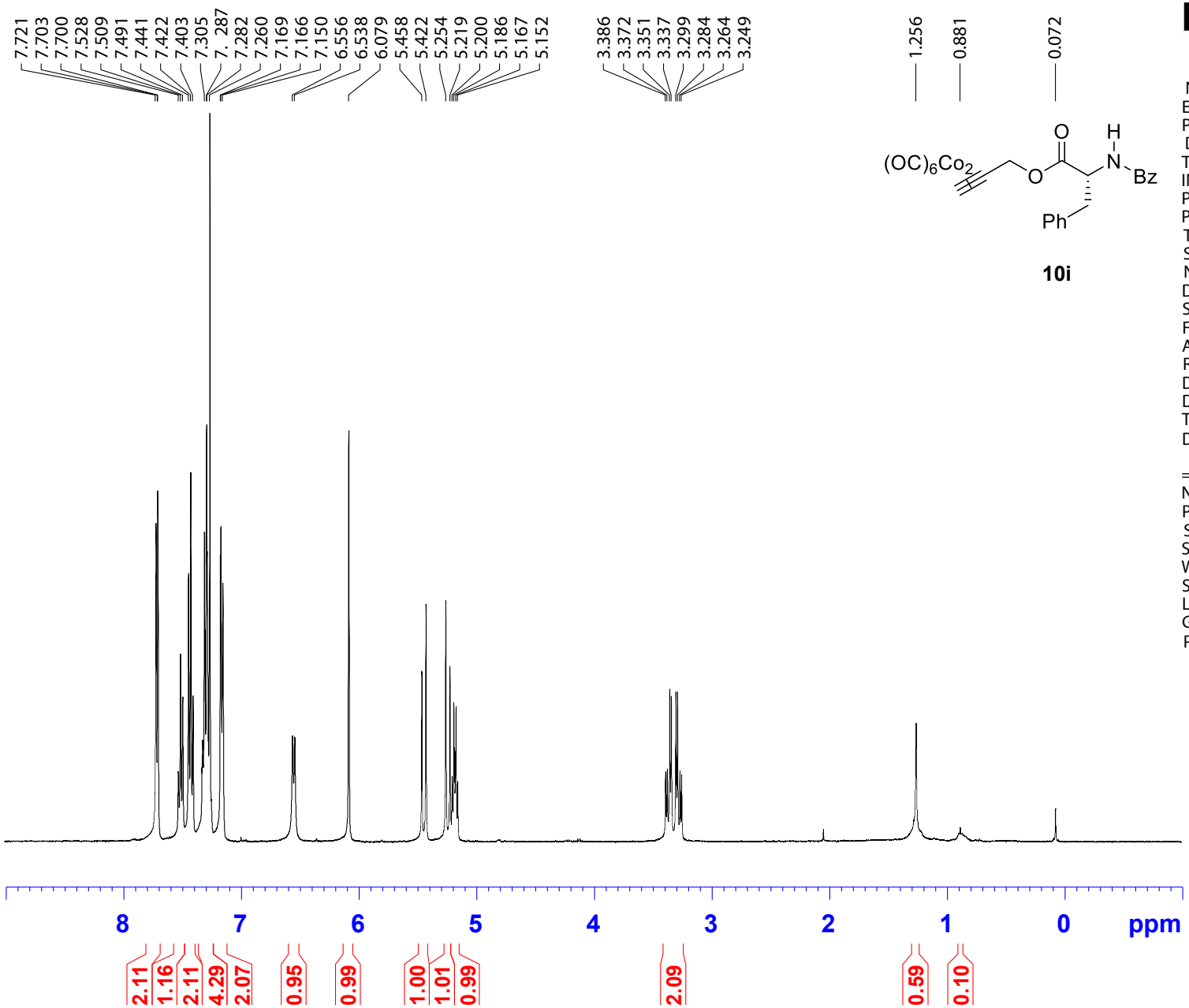
NAME	SW07–118–A	
EXPNO	10	
PROCNO	1	
Date_	20151014	
Time	18.07	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894966	sec
RG	128	
DW	62.400	usec
DE	6.50	usec
TE	96.8	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	400.1324710	MHz
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300106	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW07–118–A 13C 400



NAME	SW07–118–A	
EXPNO	11	
PROCNO	1	
Date_	20151014	
Time	23.00	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3072	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	181	
DW	20.800	usec
DE	6.50	usec
TE	97.9	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	100.6228293	MHz
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127546	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

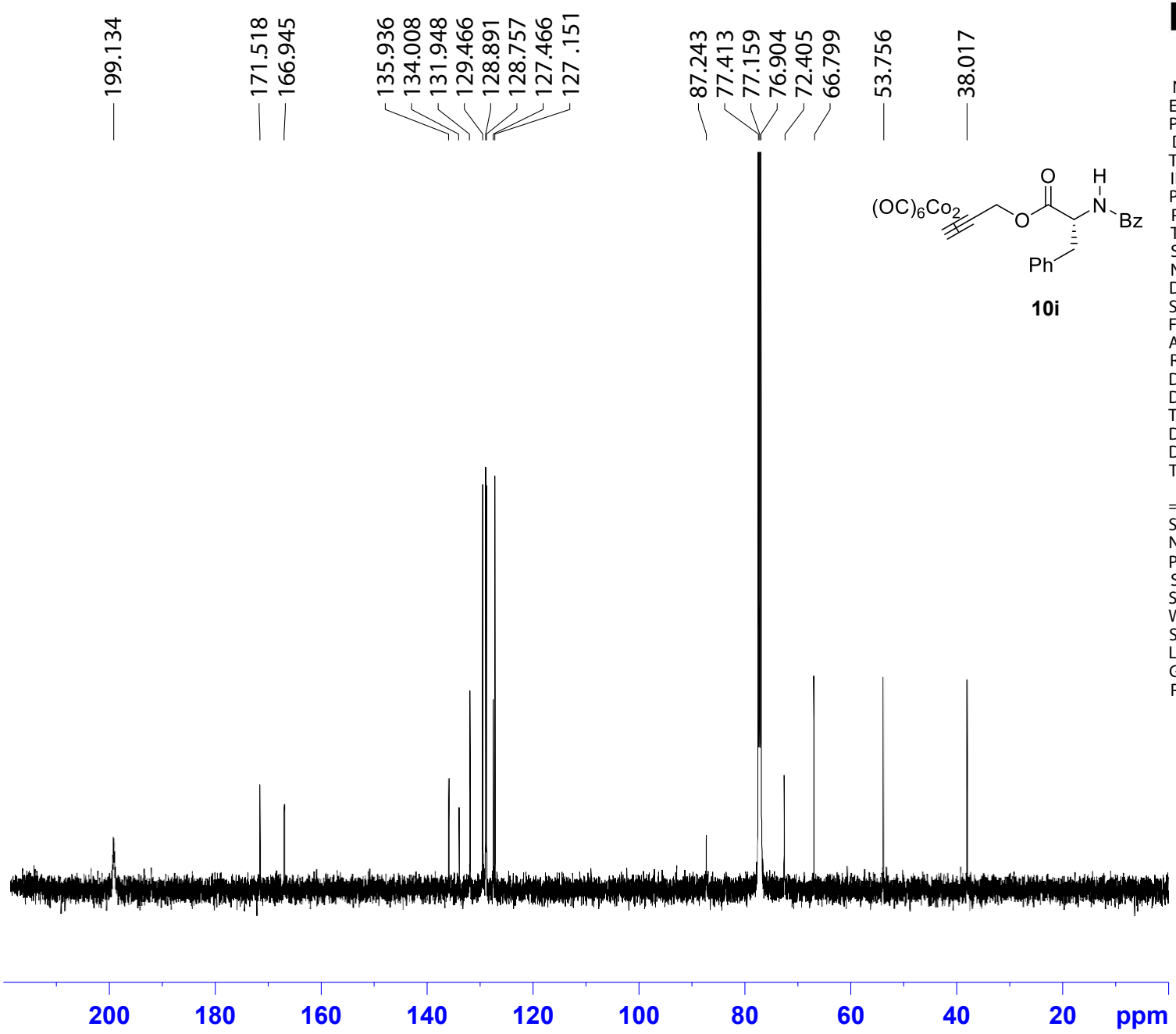
SW06–065–C 1H 400



10i

NAME	SW06–065–C	
EXPNO	10	
PROCNO	1	
Date_	20150108	
Time	18.42	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	128	
DW	60.800	usec
DE	6.50	usec
TE	96.1	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300108	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

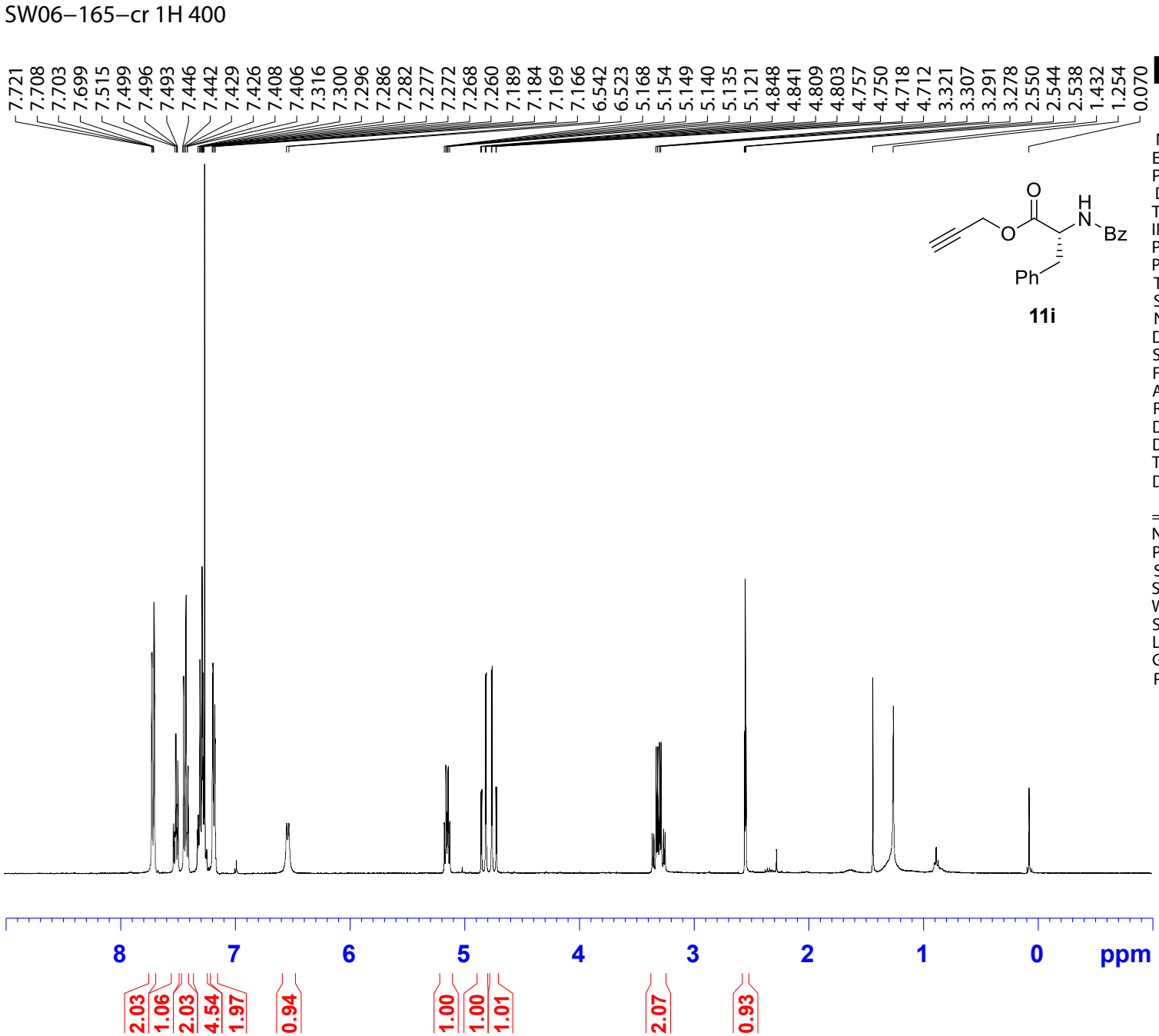
SW06–169–C 13C 500



10i

NAME	SW06–169–C	
EXPNO	10	
PROCNO	1	
Date_	20150425	
Time	2.37	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	299.2	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	125.7779086	MHz
NUC1	13C	
P1	10.50	usec
SI	32768	
SF	125.7653129	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

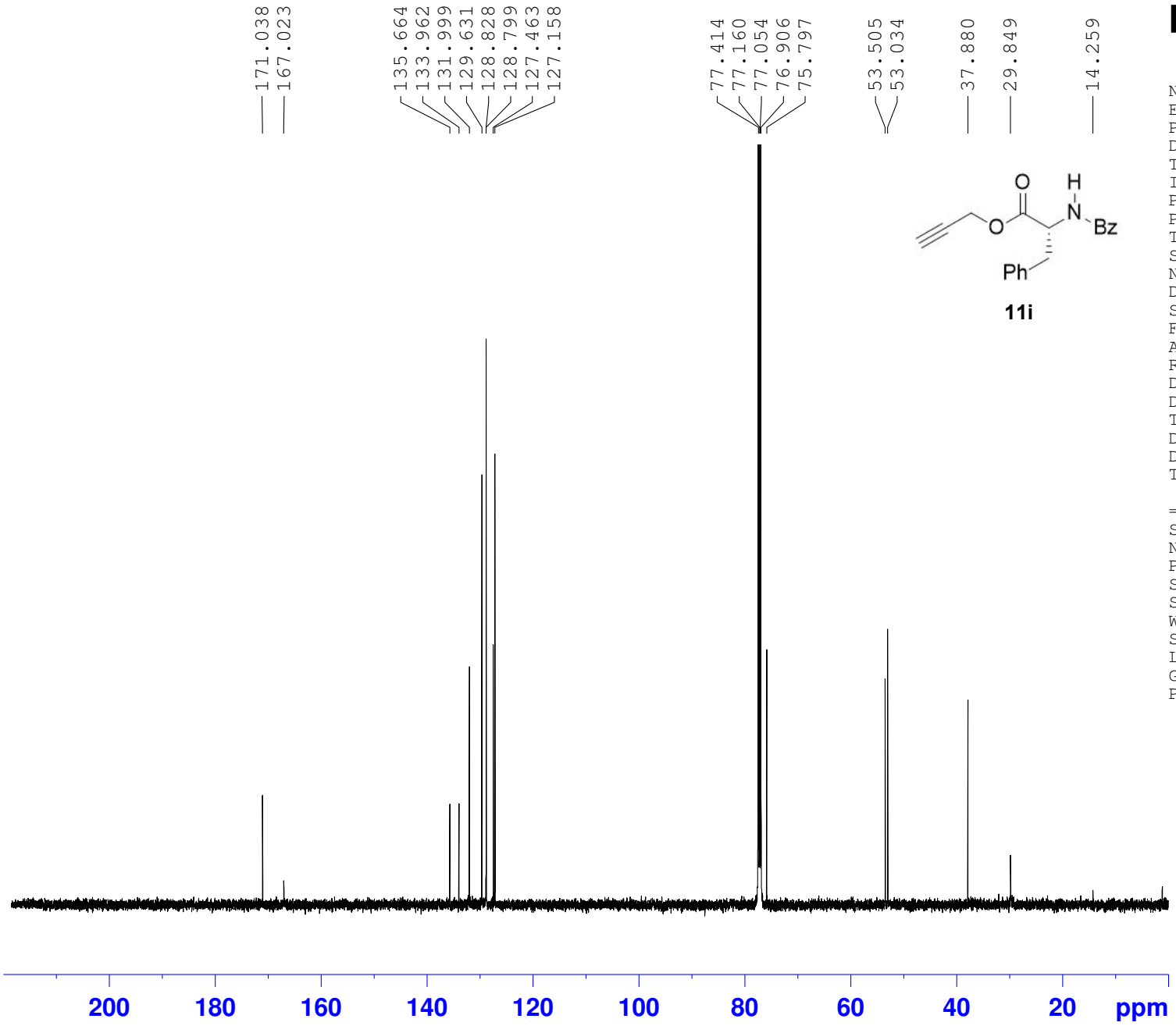
SW06–165–cr 1H 400



11i

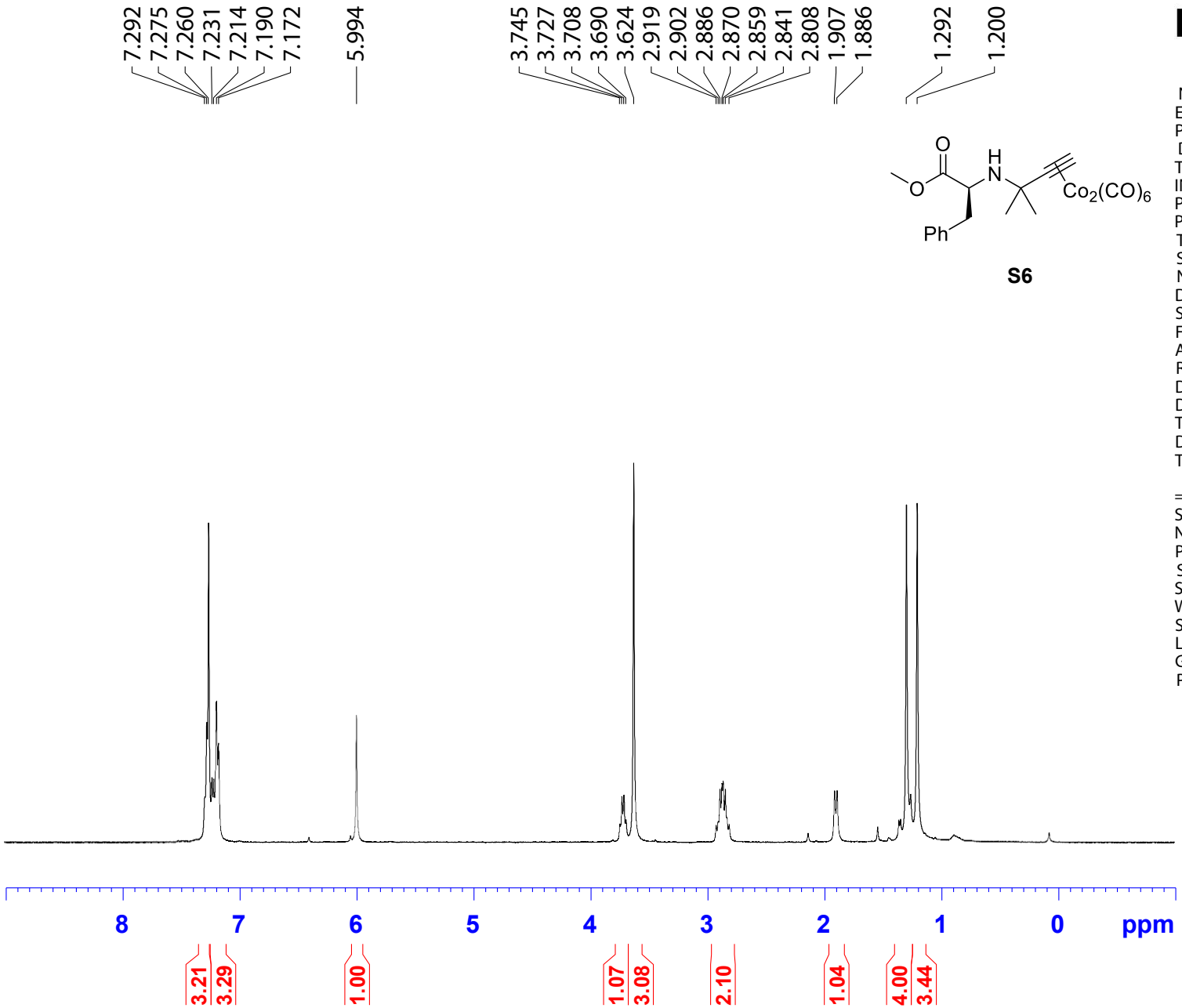
NAME	SW06–165–cr	
EXPNO	10	
PROCNO	1	
Date_	2015 0415	
Time	16.11	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	144	
DW	60.800	usec
DE	6.50	usec
TE	90.6	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300098	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW06-165-cr 1H 500



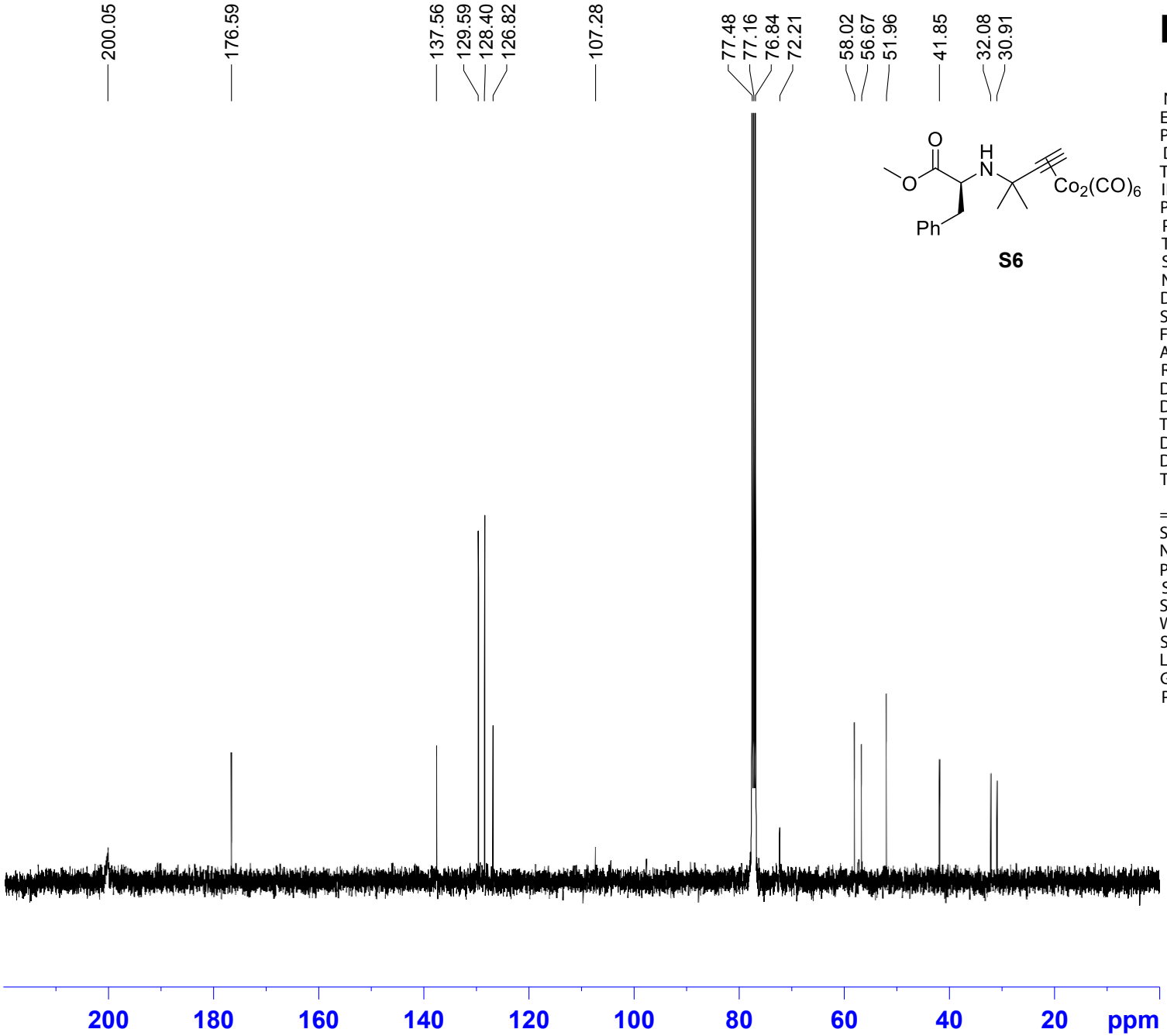
NAME	SW06-165-cr
EXPNO	11
PROCNO	1
Date_	20150502
Time	0.44
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	2
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7779086 MHz
NUC1	13C
P1	10.50 usec
SI	32768
SF	125.7653137 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

SW07–123–C 1H 400



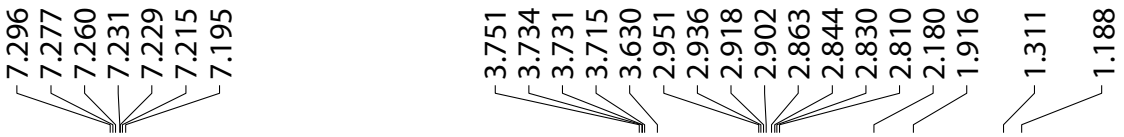
NAME	SW07–123–C	
EXPNO	10	
PROCNO	1	
Date_	20151021	
Time	16.09	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894966	sec
RG	144	
DW	62.400	usec
DE	6.50	usec
TE	93.4	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	400.1324710	MHz
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300091	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

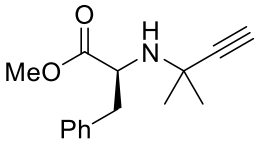
SW07−123−C 13C 400



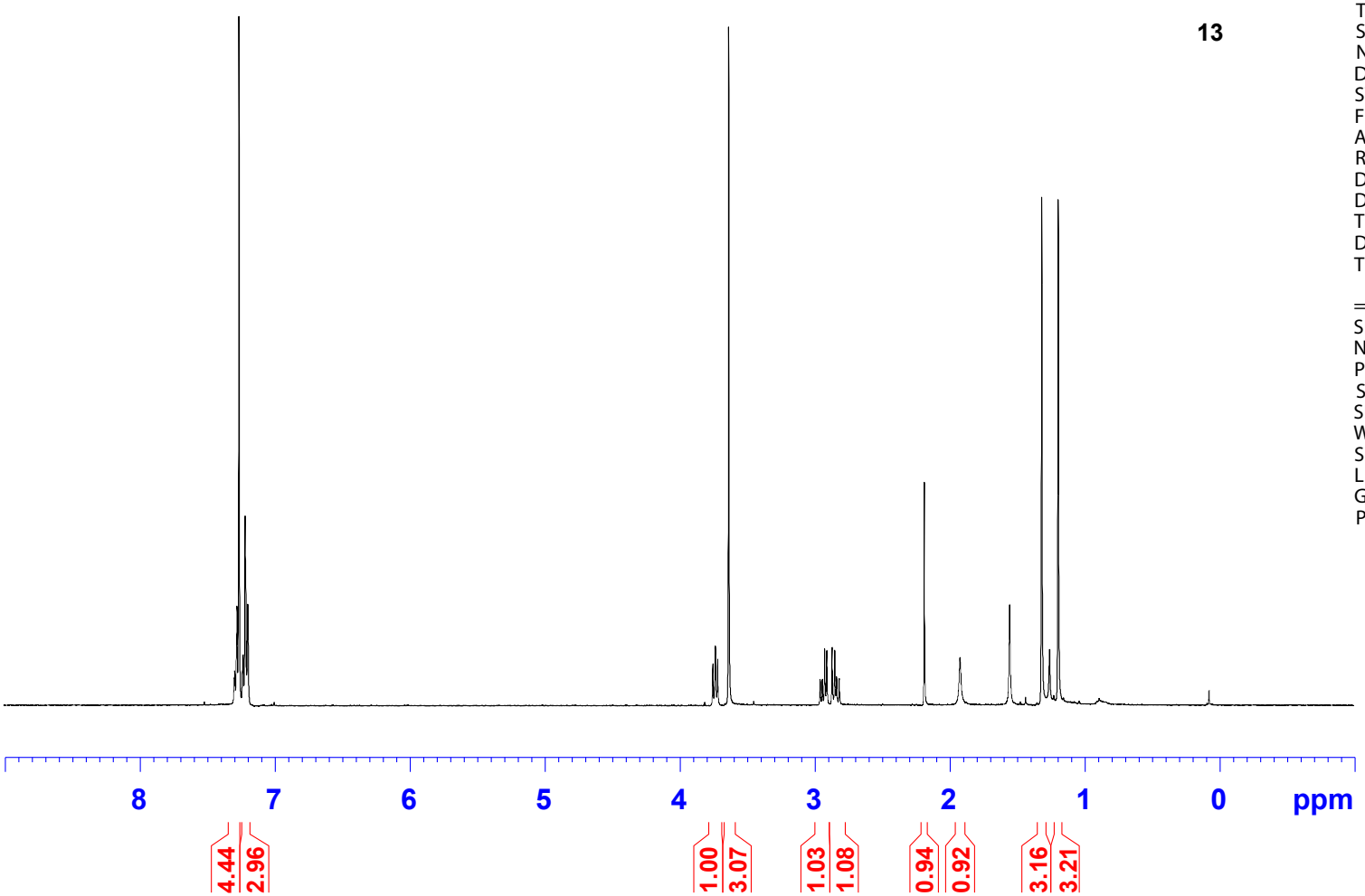
NAME	SW07−123−C	
EXPNO	11	
PROCNO	1	
Date_	20151021	
Time	23.01	
INSTRUM	spect	
PROBHD	5 mm PABBO BB−	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3072	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	181	
DW	20.800	usec
DE	6.50	usec
TE	95.9	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	100.6228293	MHz
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127540	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

SW07−131−A 1H 400



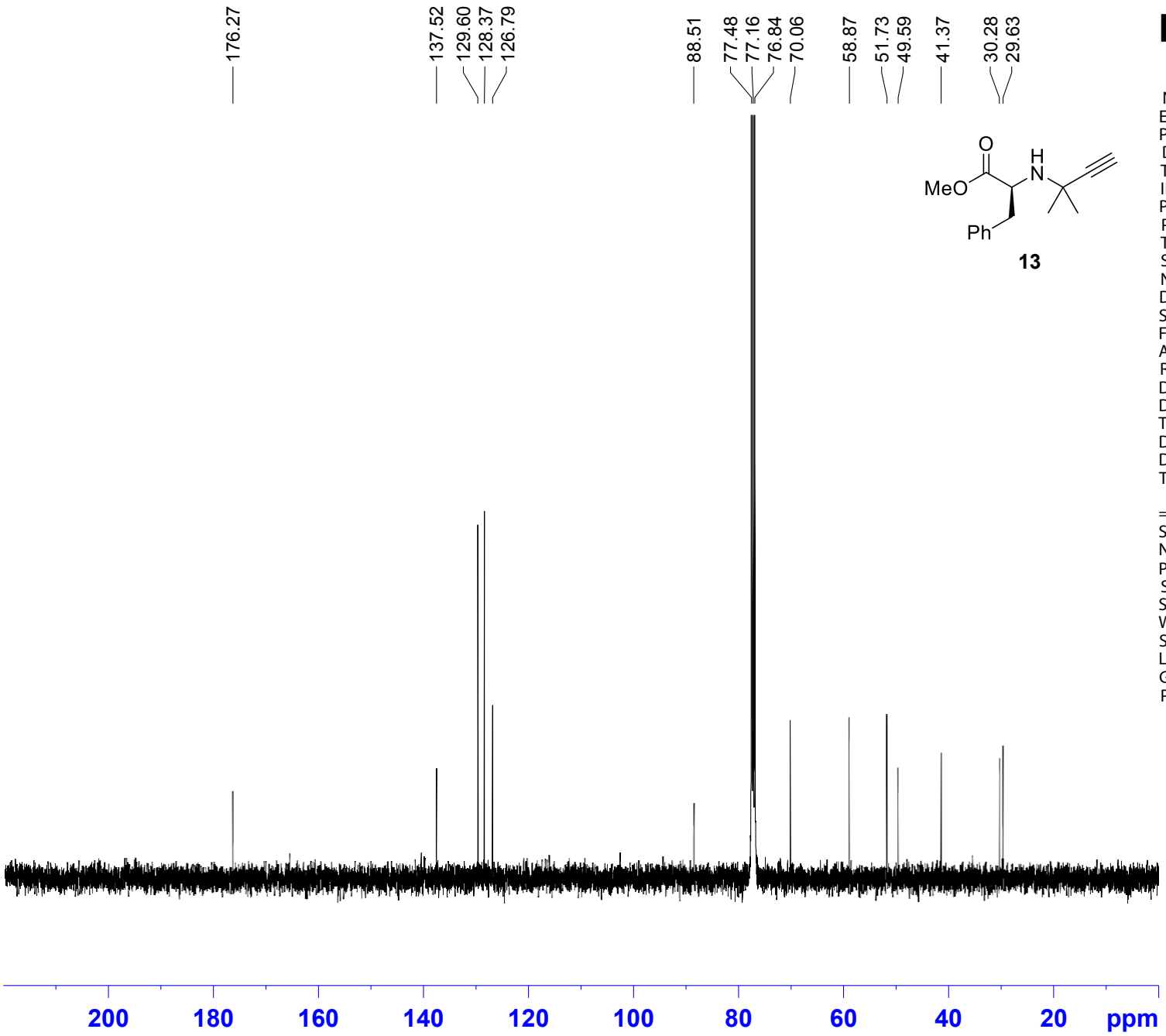


13



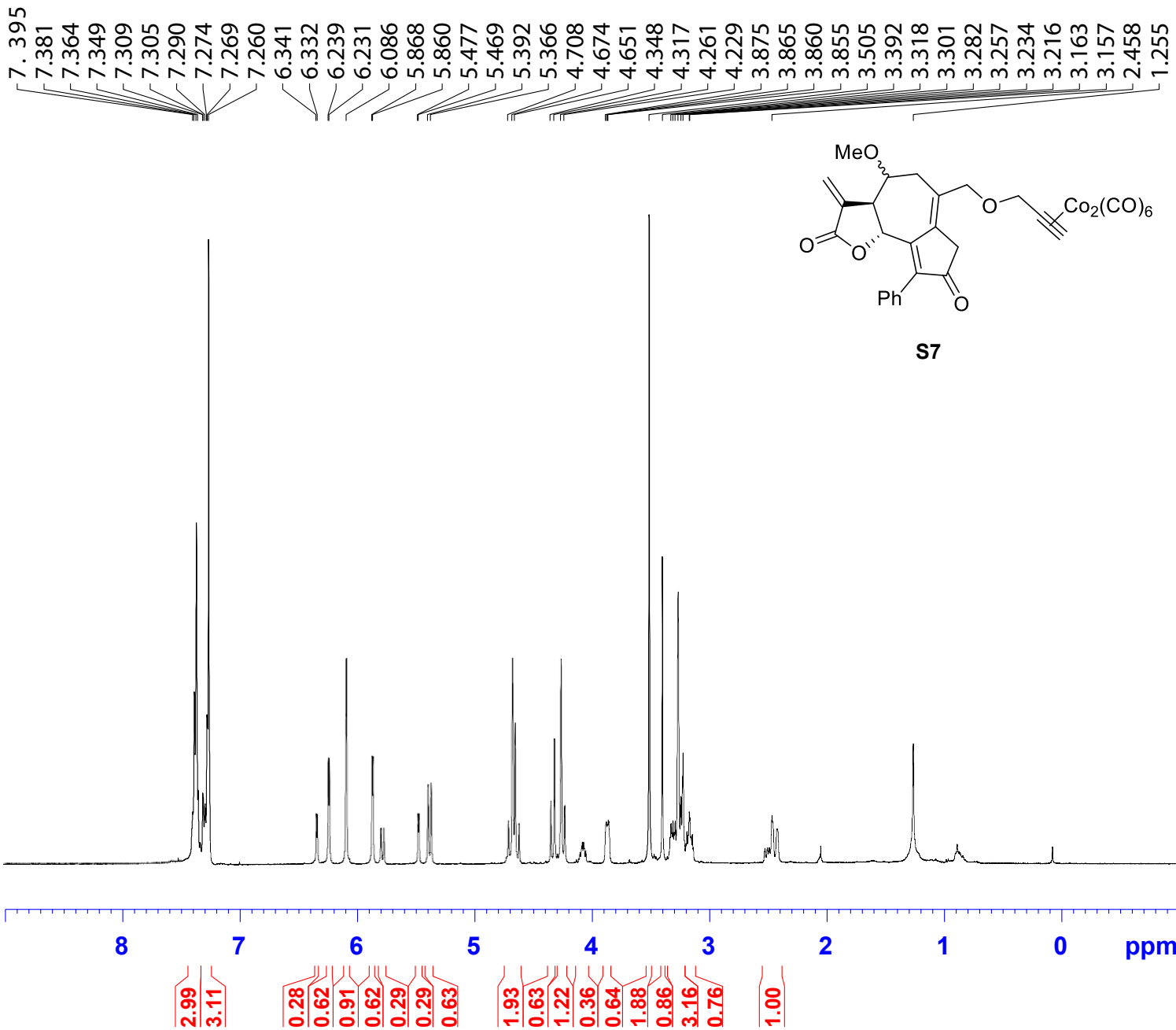
NAME	SW07−131−A	
EXPNO	10	
PROCNO	1	
Date_	20151103	
Time	17.21	
INSTRUM	spect	
PROBHD 5 mm PABBO BB−		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894966	sec
RG	144	
DW	62.400	usec
DE	6.50	usec
TE	91.8	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	400.1324710	MHz
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300105	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW07–131–A 13C 400



NAME	SW07–131–A	
EXPNO	11	
PROCNO	1	
Date_	20151103	
Time	23.00	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3072	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	97.2	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	100.6228293	MHz
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127542	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

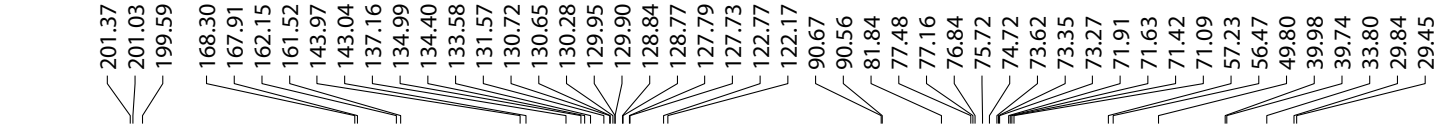
SW06–030–C 1H 400

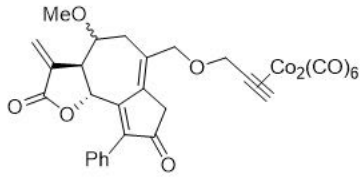


S7

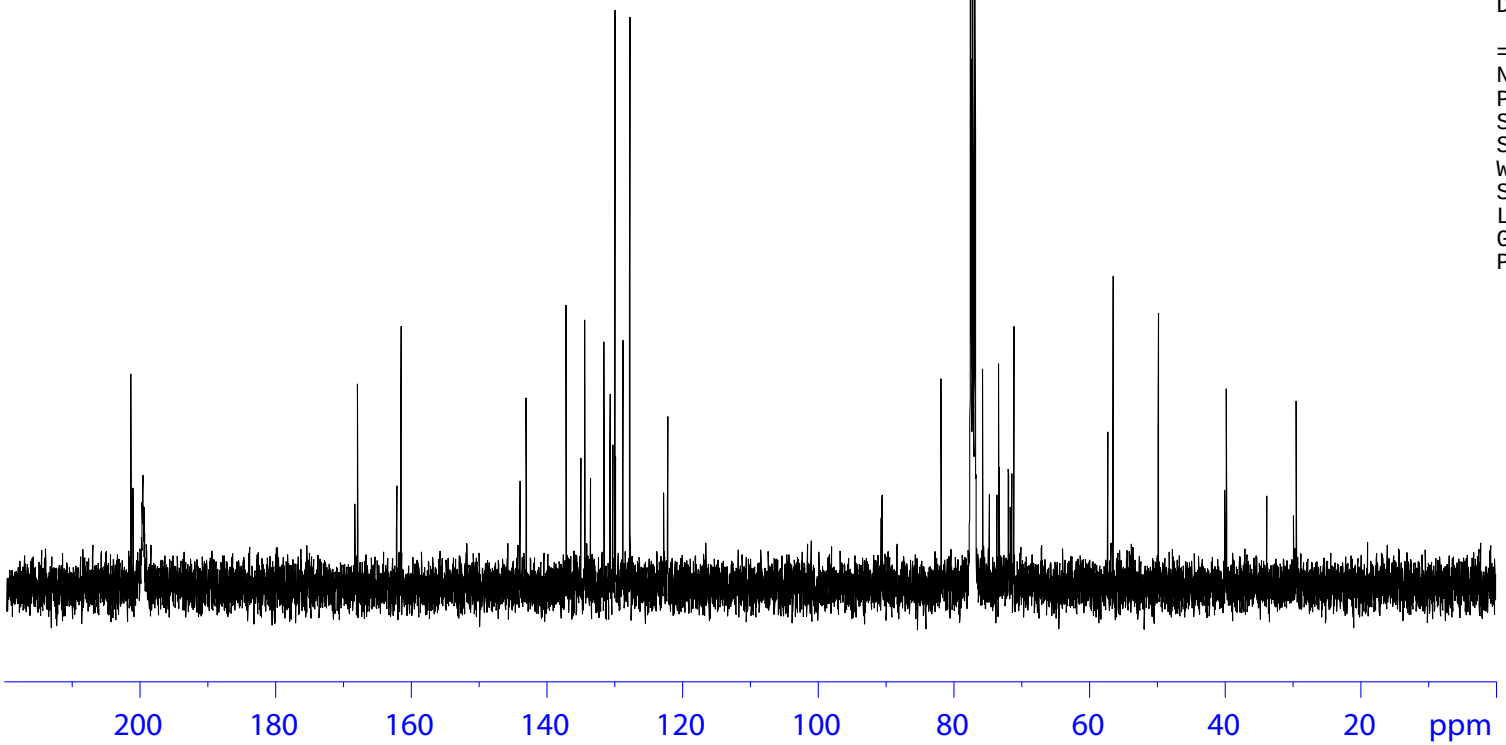
NAME	SW06–030–C	
EXPNO	20	
PROCNO	1	
Date_	20141114	
Time	17.01	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	114	
DW	60.800	usec
DE	6.50	usec
TE	94.6	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300105	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW06–030–C 13C 400



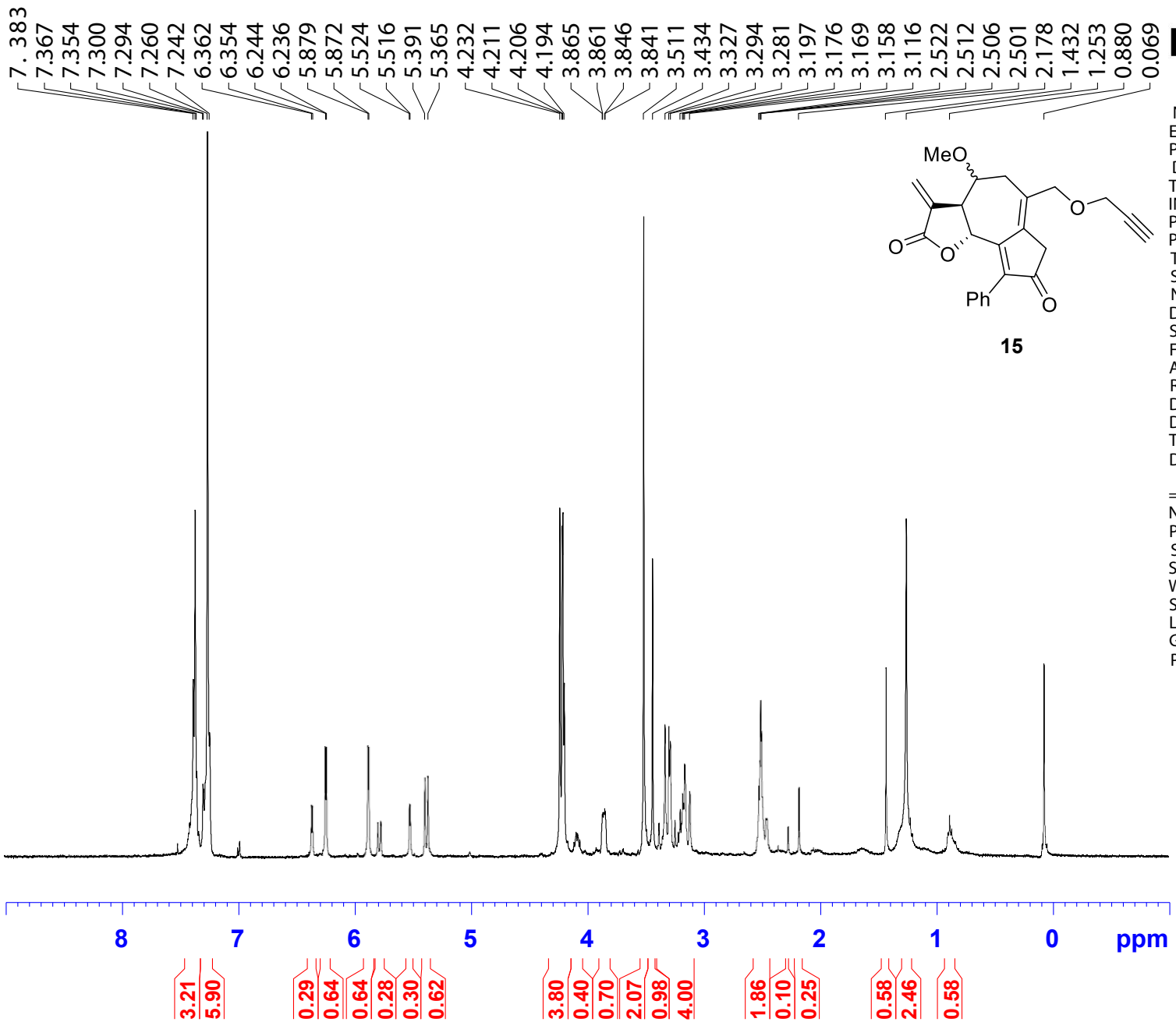


S7



NAME	SW06–030–C
EXPNO	21
PROCNO	1
Date_	20141115
Time	6.09
INSTRUM	spect
PROBHD	5 mm PABBO BB–
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2200
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	93.3 K
D1	2.00000000 sec
D11	0.03000000 sec
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127547 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

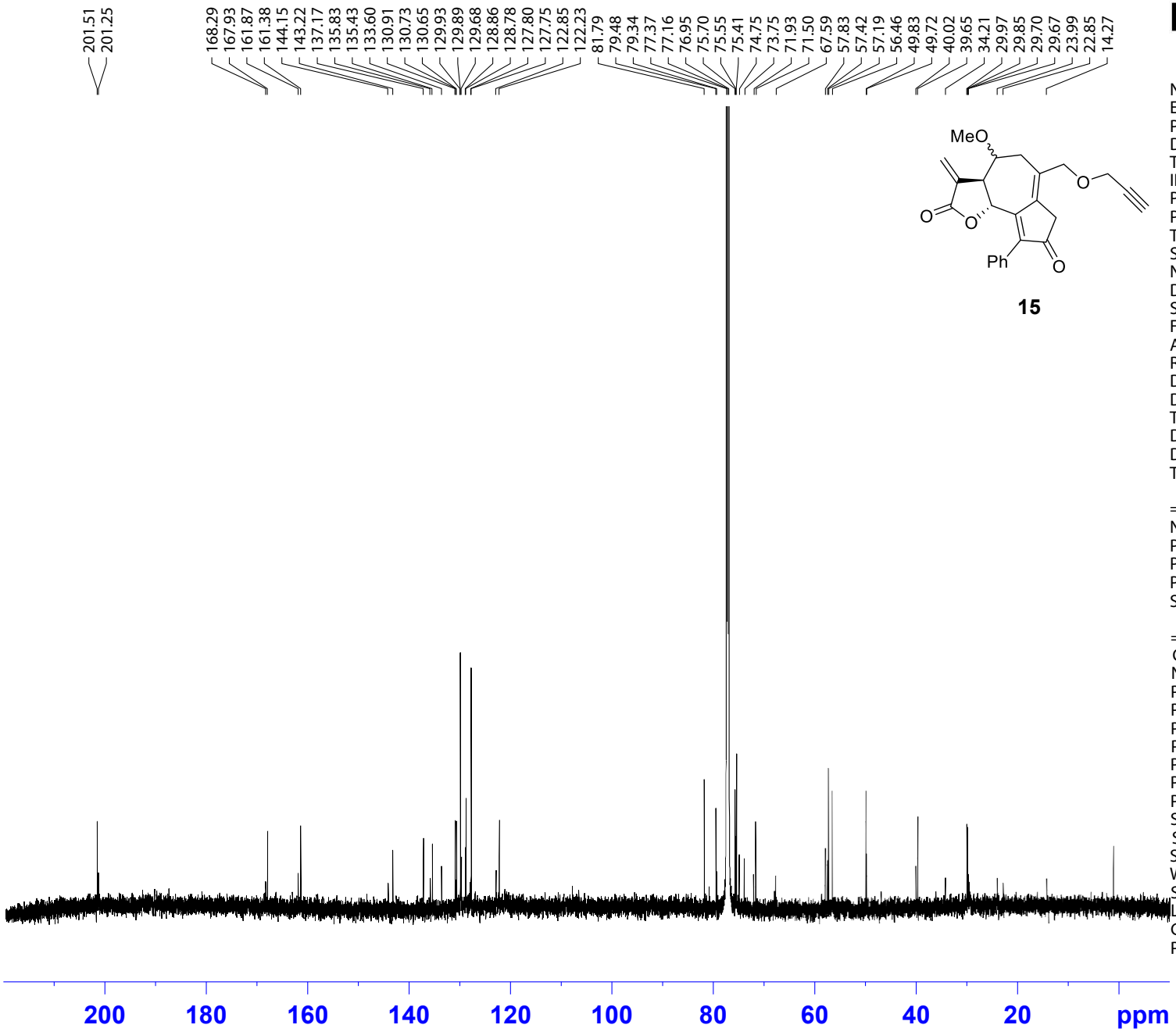
SW06–039–cr 1H 400



15

NAME	SW06–039–cr	
EXPNO	10	
PROCNO	1	
Date_	20141117	
Time	18.53	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	161	
DW	60.800	usec
DE	6.50	usec
TE	94.1	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300101	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW06–039–cr 13C
600MHz

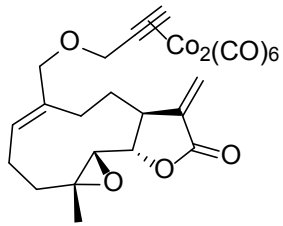


15

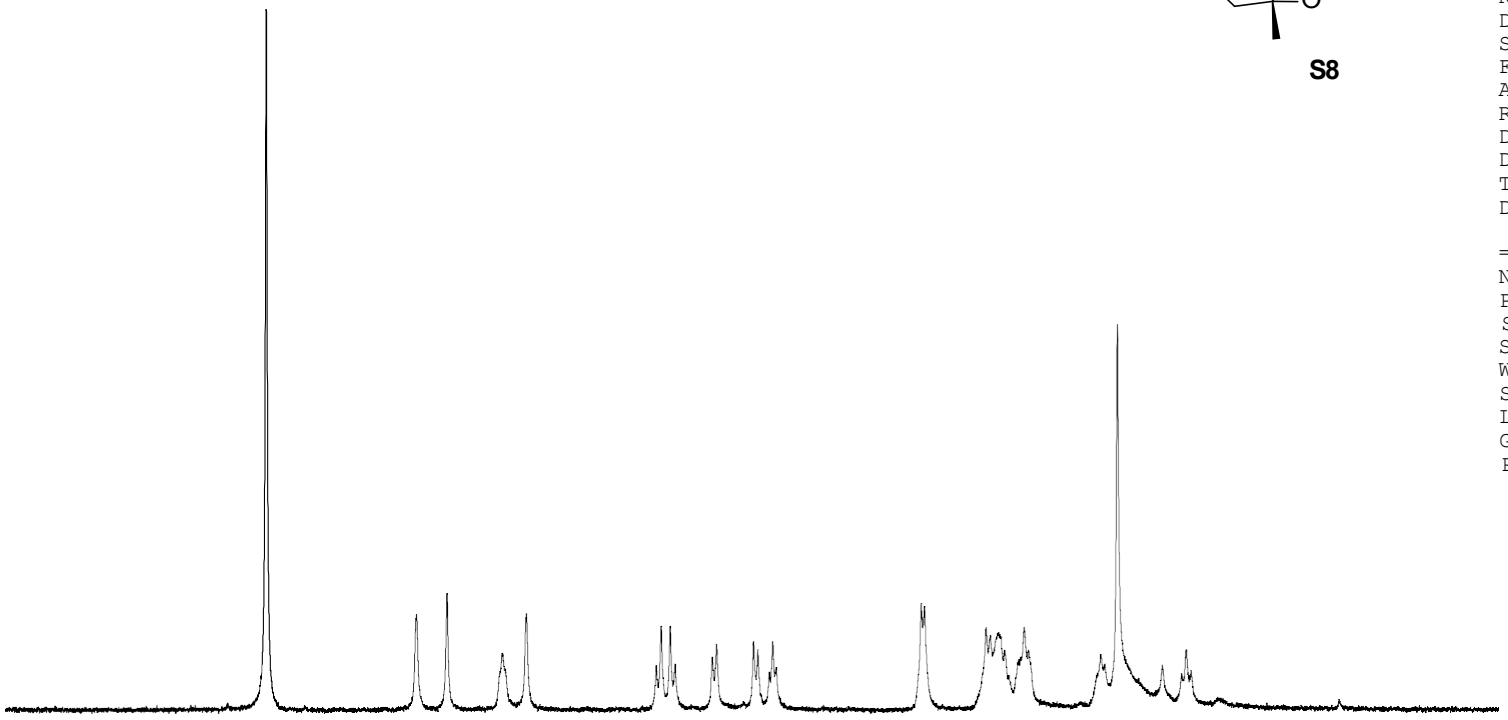
NAME	SW06–039–cr dry	
EXPNO	2	
PROCNO	1	
Date_	20150623	
Time	16.32	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	27093	
DS	4	
SWH	36057.691	Hz
FIDRES	0.550197	Hz
AQ	0.9088159	sec
RG	203	
DW	13.867	usec
DE	6.50	usec
TE	298.2	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1========		
NUC1	13C	
P1	11.50	usec
PL1	0.00	dB
PL1W	97.46119690	W
SFO1	151.0637542	MHz
======== CHANNEL f2========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	70.00	usec
PL2	−2.00	dB
PL12	14.19	dB
PL13	120.00	dB
PL2W	19.70630455	W
PL12W	0.47381112	W
PL13W	0.00000000	W
SFO2	600.7124028	MHz
SI	32768	
SF	151.0486269	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

SW06-183-C 1H 400

7.260
6.247
6.042
5.670
5.511
4.641
4.608
4.548
4.516
4.265
4.237
3.991
3.961
3.885
3.861
3.837
2.867
2.845
2.432
2.405
2.348
2.309
2.216
2.178
2.150
1.666
1.638
1.554
1.410
1.253
1.124
1.093
1.060

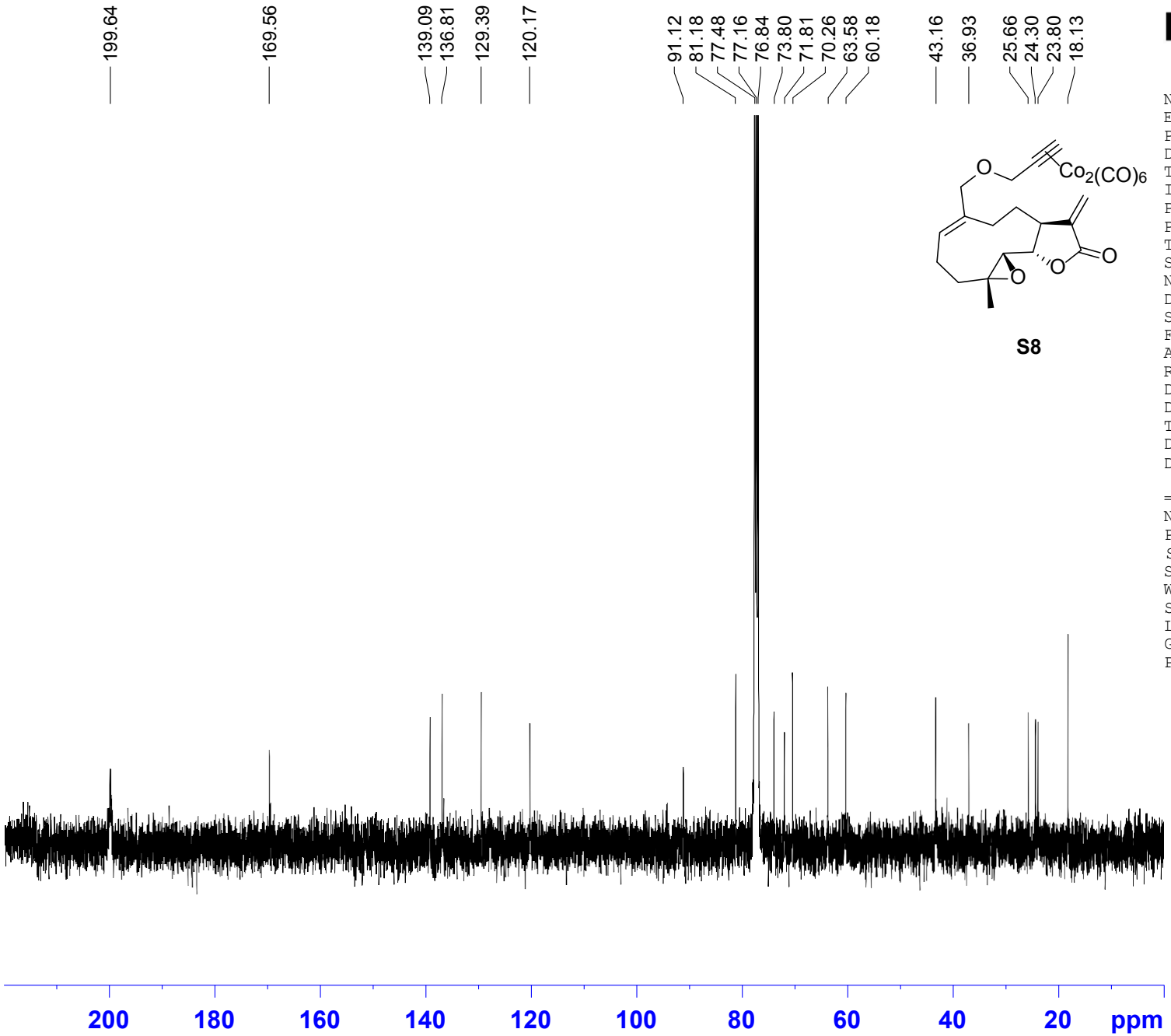


S8



NAME	SW06-183-C
EXPNO	20
PROCNO	1
Date_	20150507
Time	12.52
INSTRUM	spect
PROBHD 5 mm PABBO BB-	
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	181
DW	60.800 usec
DE	6.50 usec
TE	90.6 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	13.75 usec
SI	65536
SF	400.1300102 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

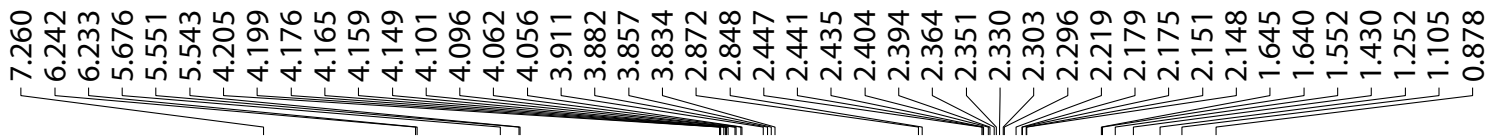
SW06-183-C 1H 400

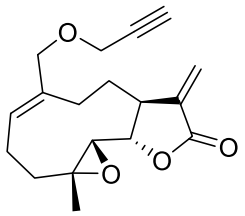


S8

```
NAME              SW06-183-C
EXPNO                     11
PROCNO                     1
Date_               20150505
Time                    7.27
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     2048
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                      203
DW                 20.800 usec
DE                  6.50 usec
TE                  95.3 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                 10.00 usec
SI                      32768
SF           100.6127547 MHz
WDW                       EM
SSB                        0
LB                  1.00 Hz
GB                         0
PC                      1.40
```

SW06–191–cr 1H 400

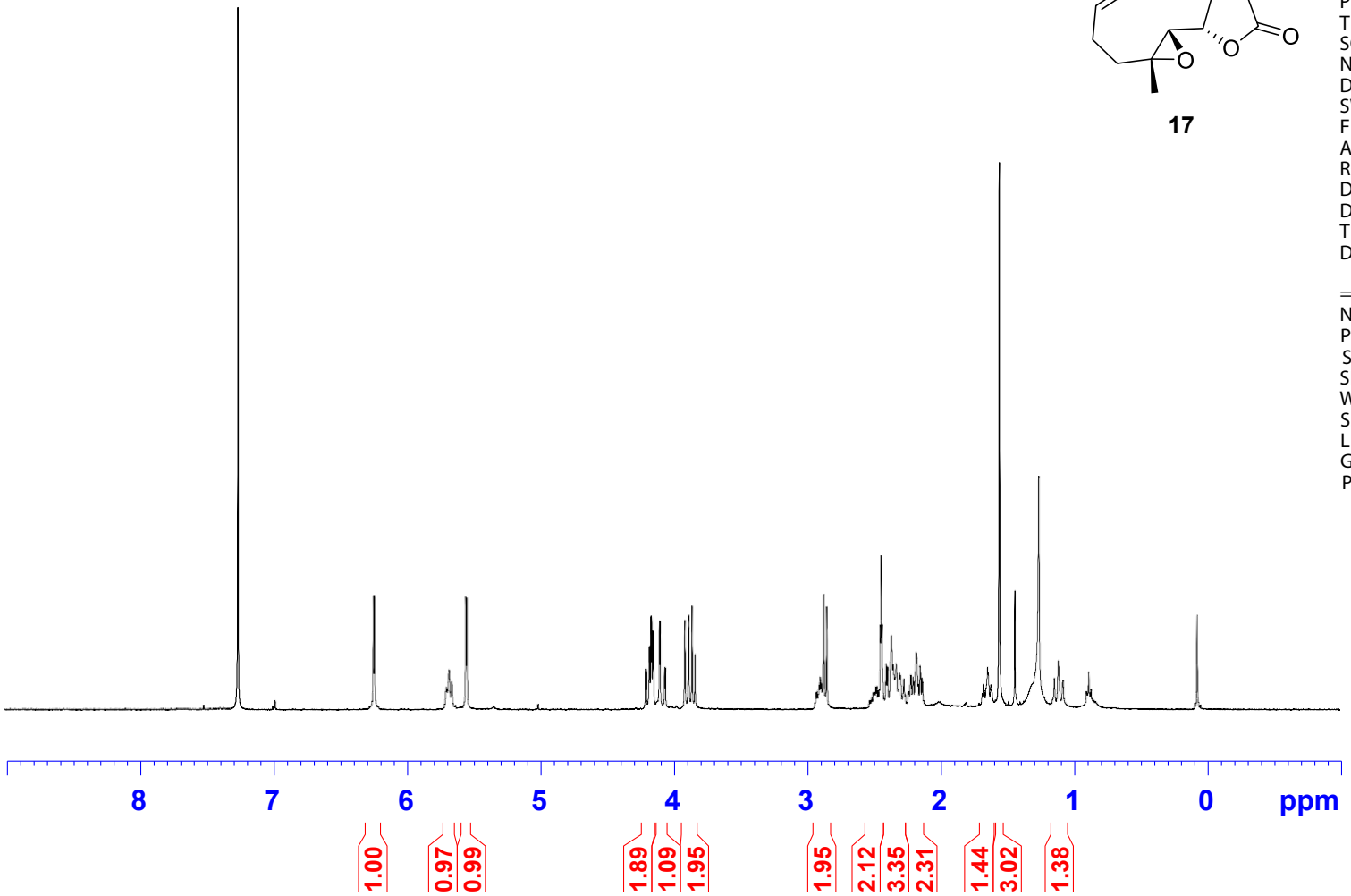




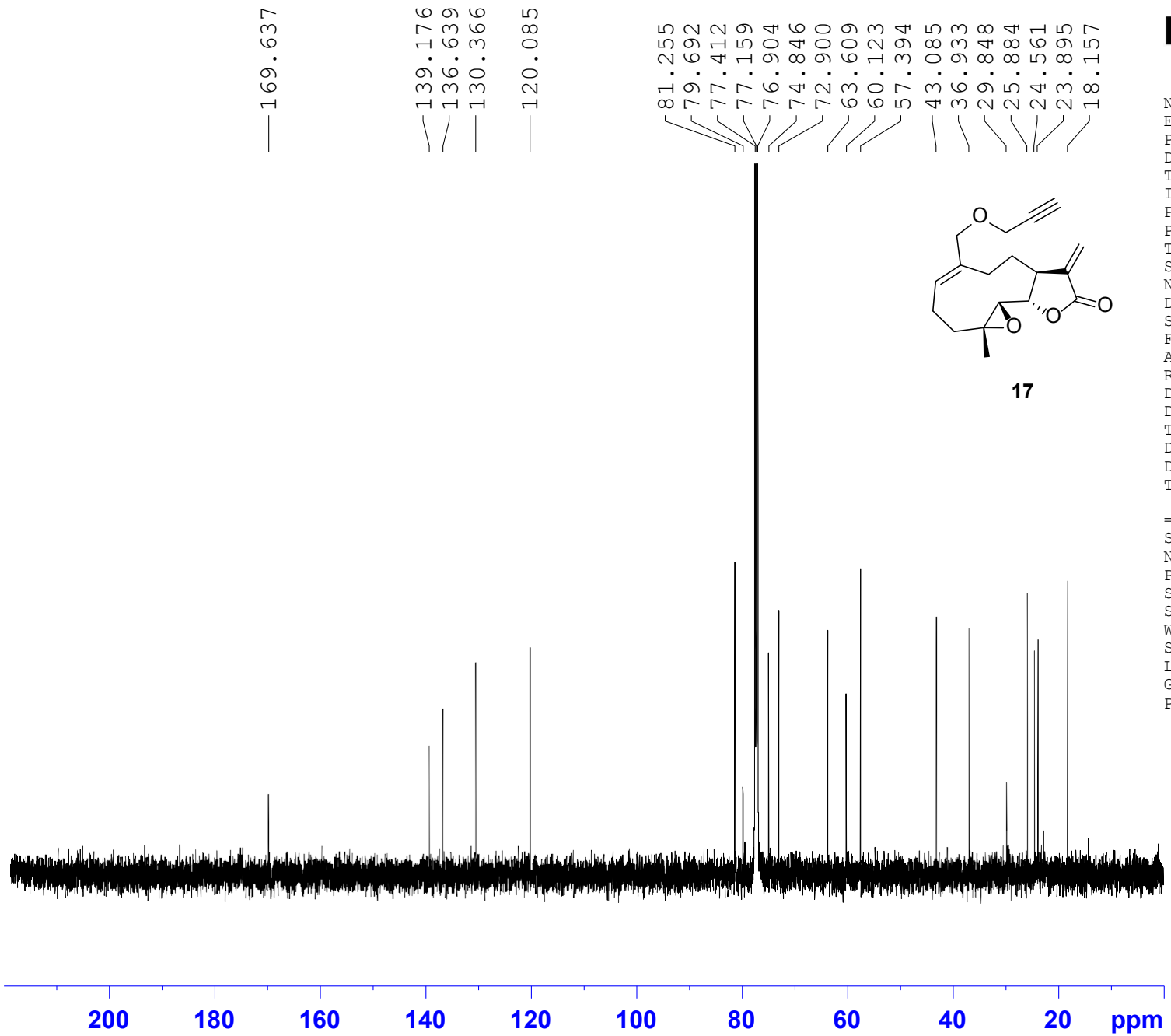
17

NAME SW06–191–cr
EXPNO 10
PROCNO 1
Date_ 20150512
Time 17.34
INSTRUM spect
PROBHD 5 mm PABBO BB–
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 3049.3 K
D1 1.00000000 sec

======== CHANNEL f1========
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



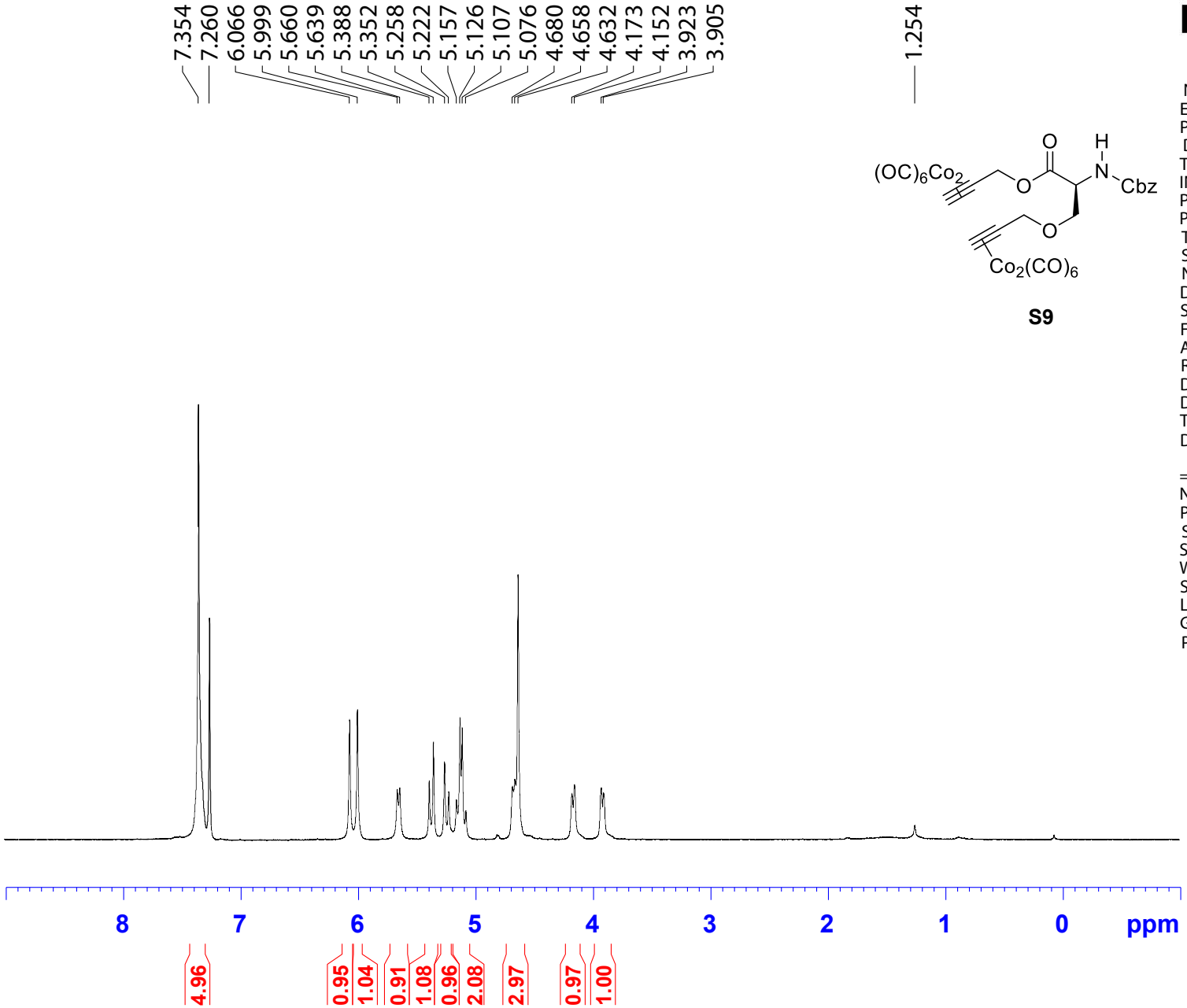
SW06-191-cr 13C 500



17

NAME	SW06-191-cr
EXPNO	10
PROCNO	1
Date_	20150513
Time	6.46
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	2
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7779086 MHz
NUC1	13C
P1	10.50 usec
SI	32768
SF	125.7653131 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

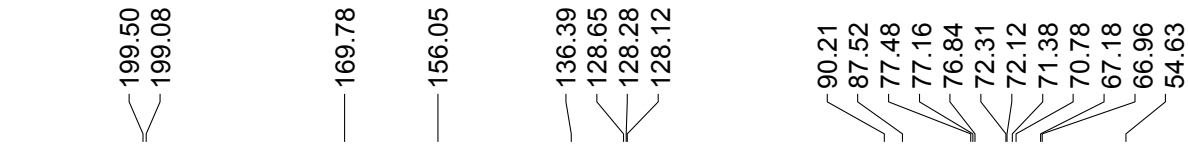
SW06–158–B 1H 400

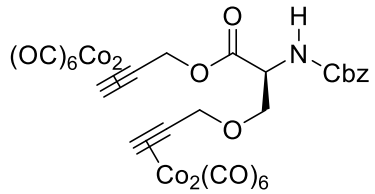


S9

NAME	SW06–158–B	
EXPNO	10	
PROCNO	1	
Date_	20150414	
Time	17.59	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	144	
DW	60.800	usec
DE	6.50	usec
TE	94.7	K
D1	1.000000 00	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300100	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

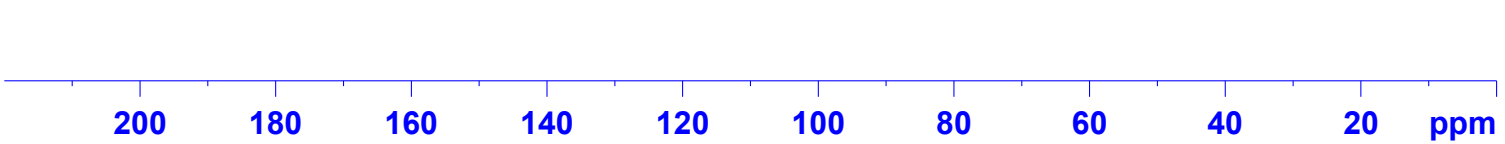
SW06–158–B 13C 400



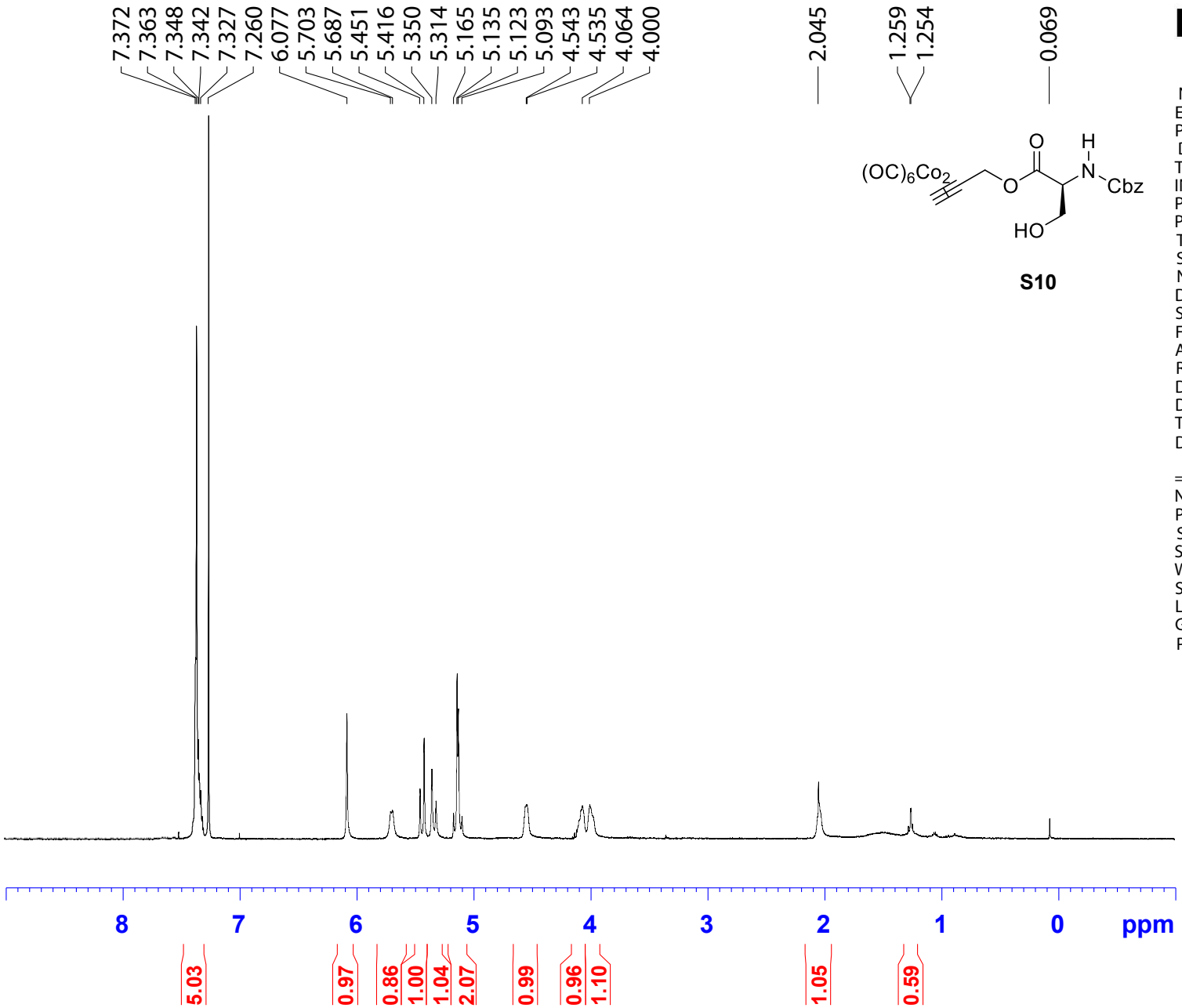


S9

NAME	SW06–158–B	
EXPNO	20	
PROCNO	1	
Date_	20150415	
Time	23.28	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	92.7	K
D1	2.00000000	sec
D11	0.03000000	sec
======== CHANNEL f1	========	
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127549	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	



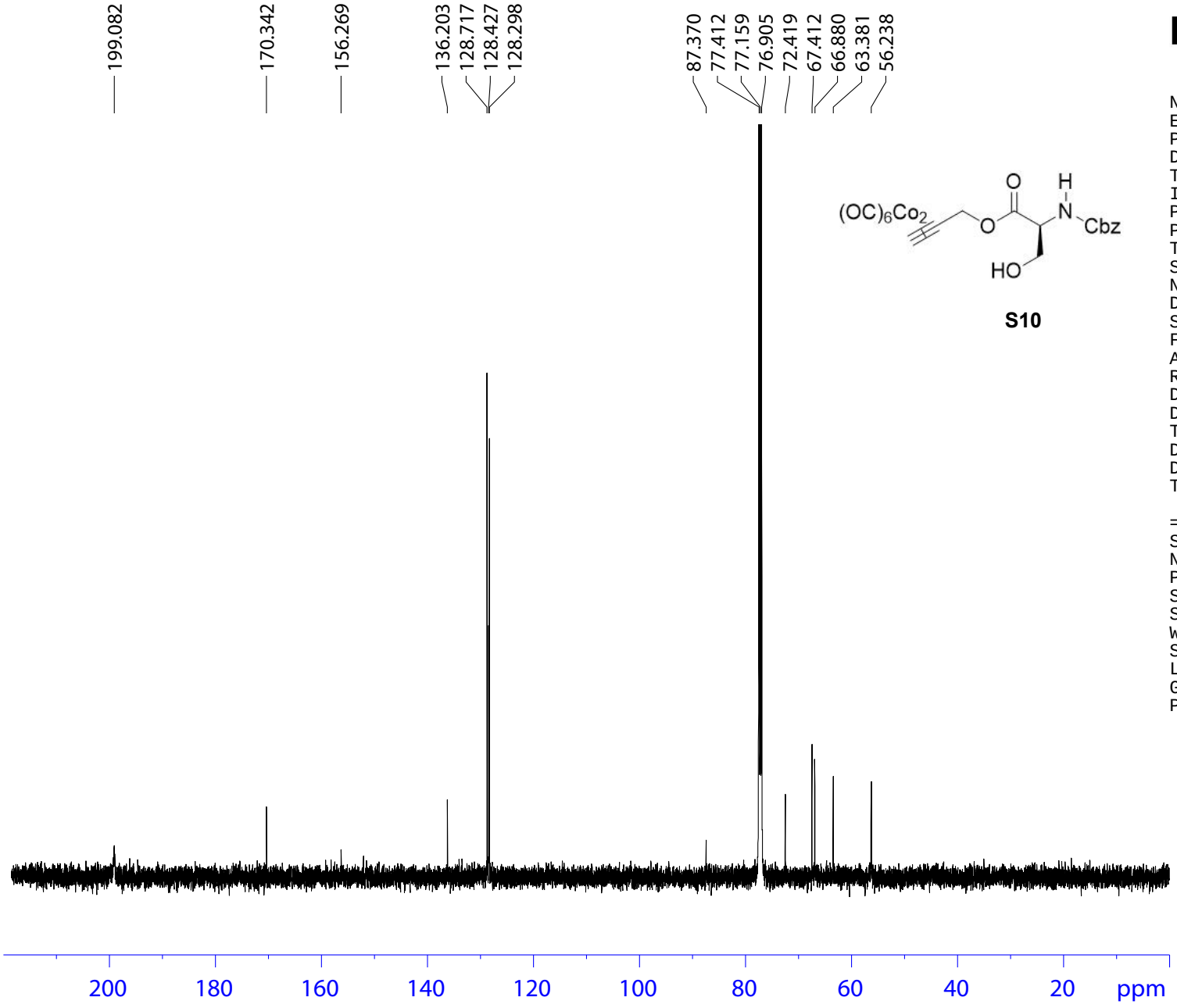
SW06–202–C 1H 400



S10

NAME	SW06–202–C	
EXPNO	10	
PROCNO	1	
Date_	20150522	
Time	13.34	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	161	
DW	60.800	usec
DE	6.50	usec
TE	94.3	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300099	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW06–202–C 13C 500

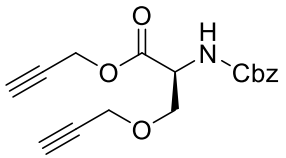


S10

```
NAME                SW06–202–C
EXPNO                       10
PROCNO                       1
Date_                 20150526
Time                      0.37
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        3072
DS                           2
SWH                29761.904 Hz
FIDRES              0.454131 Hz
AQ                  1.1010548 sec
RG                         203
DW                    16.800 usec
DE                     6.50 usec
TE                     298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             125.7779086 MHz
NUC1                       13C
P1                    10.50 usec
SI                        32768
SF               125.7653131 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.40
```

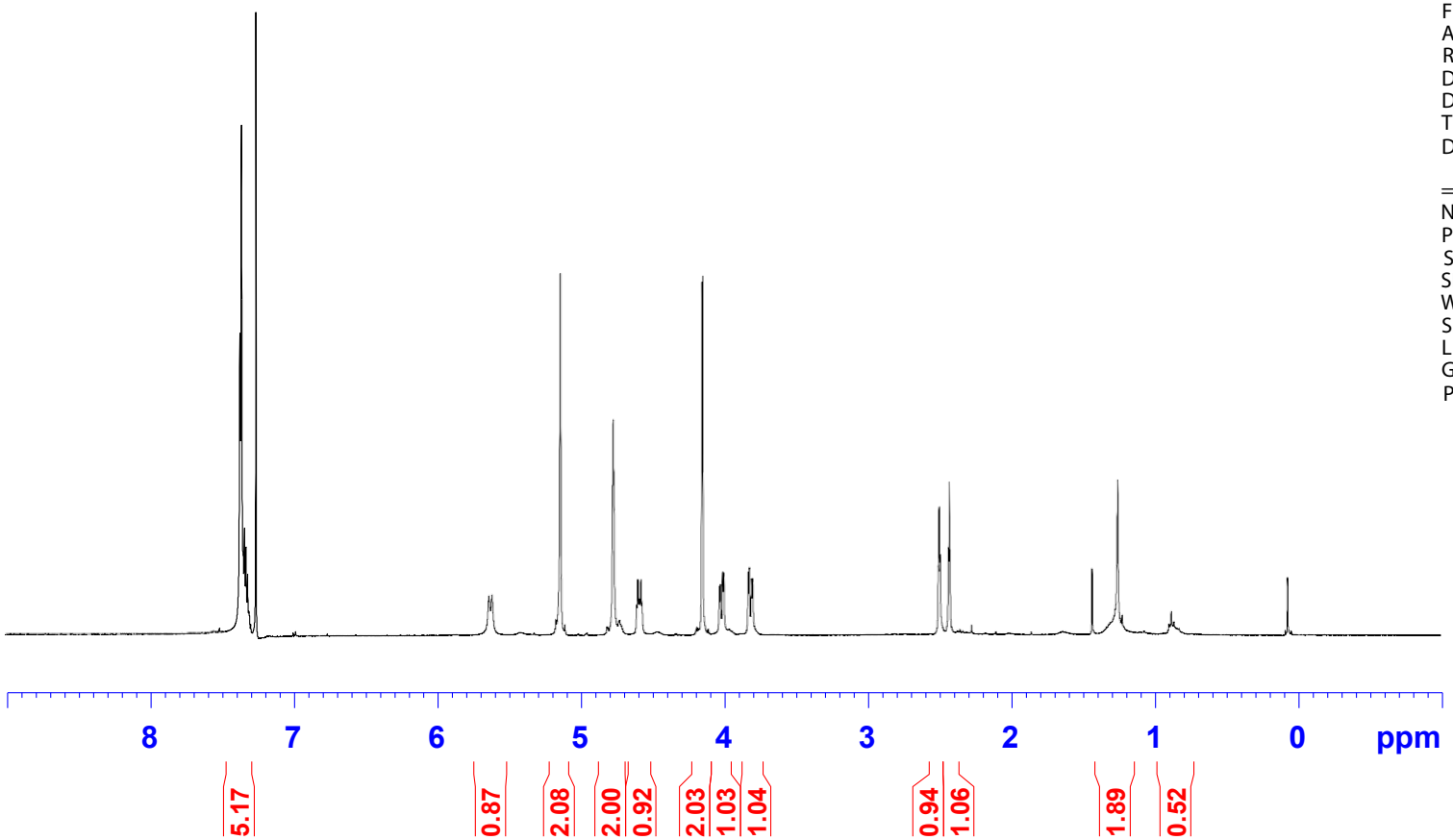
SW06–196–cr 1H 400

7.372
7.361
7.349
7.339
7.330
7.318
7.260
5.632
5.611
5.166
5.135
5.103
4.811
4.804
4.766
4.728
4.722
4.602
4.595
4.587
4.581
4.573
4.566
4.149
4.144
4.027
4.020
4.004
3.996
3.826
3.819
3.803
3.795
2.502
2.496
2.490
2.432
2.426
2.421
1.432
1.253
1.222
0.880
0.070

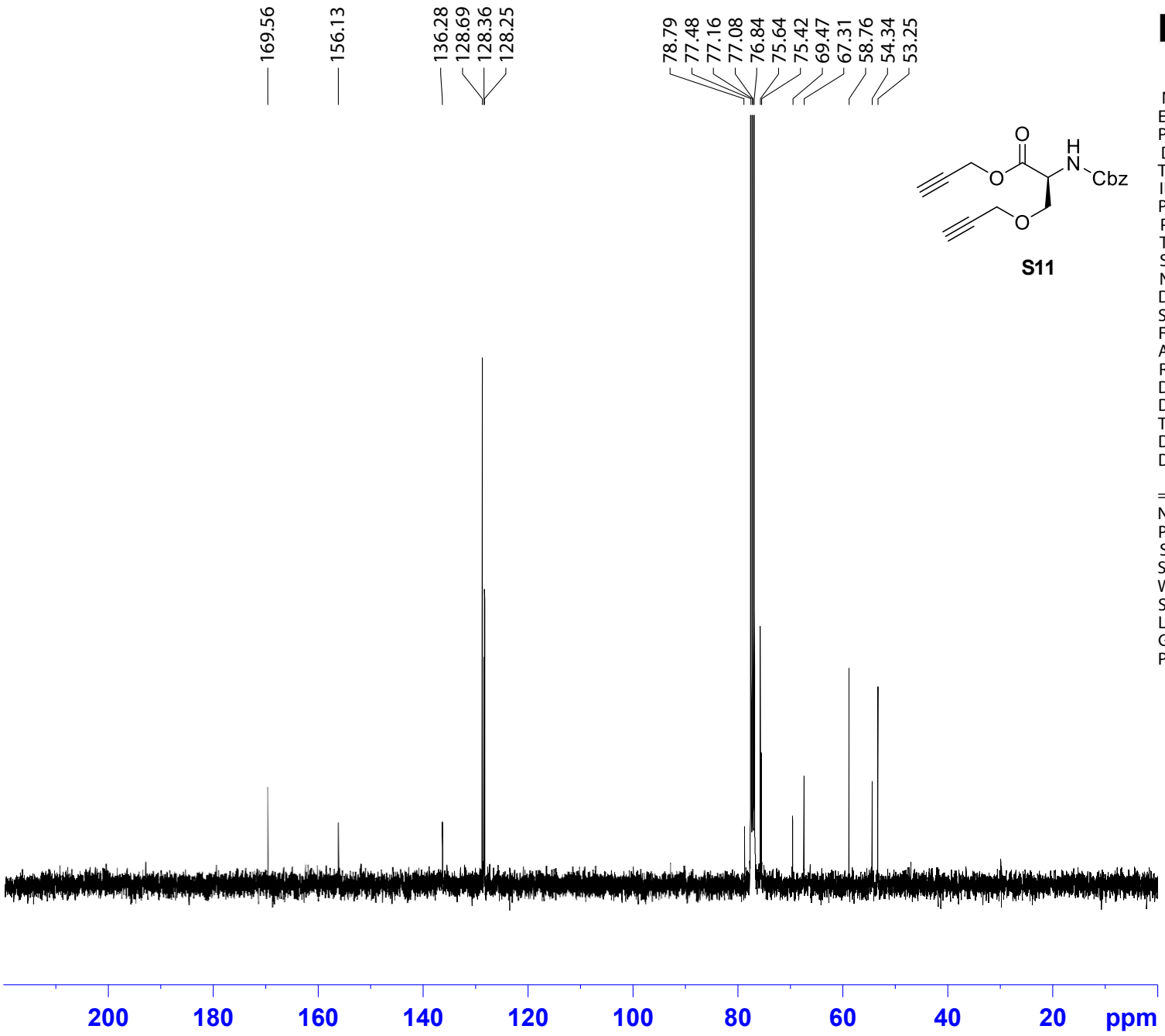


S11

NAME	SW06–196–cr	
EXPNO	20	
PROCNO	1	
Date_	20150515	
Time	9.03	
INSTRUM	spect	
PROBHD 5 mm PABBO BB–		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	144	
DW	60.800	usec
DE	6.50	usec
TE	296.8	K
D1	1.00000000	sec
======== CHANNEL f1	========	
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300103	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

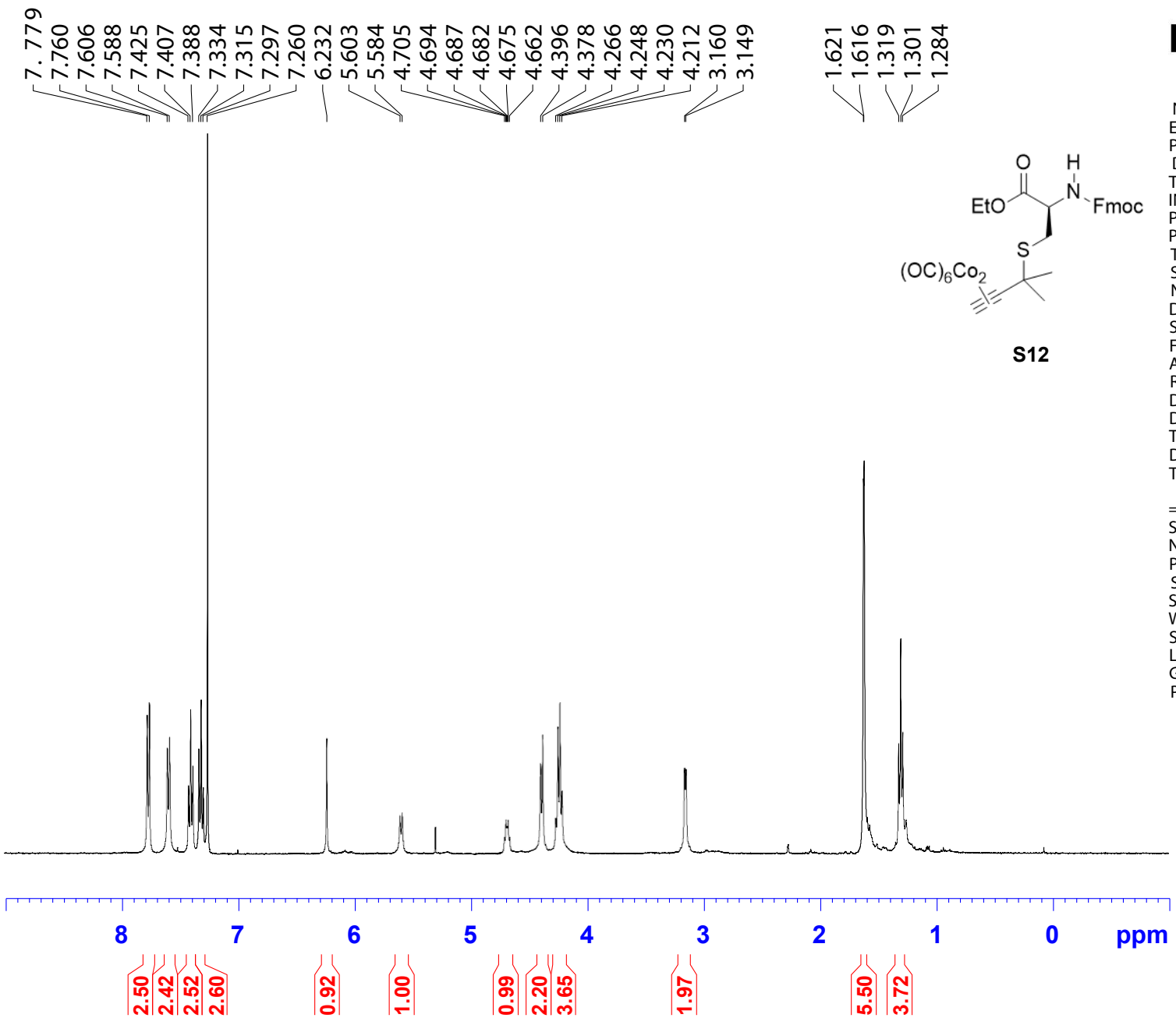


SW06–196–cr



NAME	SW06–196–cr	
EXPNO	11	
PROCNO	1	
Date_	20150515	
Time	6.32	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	2048	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	128	
DW	20.800	usec
DE	6.50	usec
TE	297.2	K
D1	2.00000000	sec
D11	0.03000000	sec
======== CHANNEL f1	========	
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127550	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

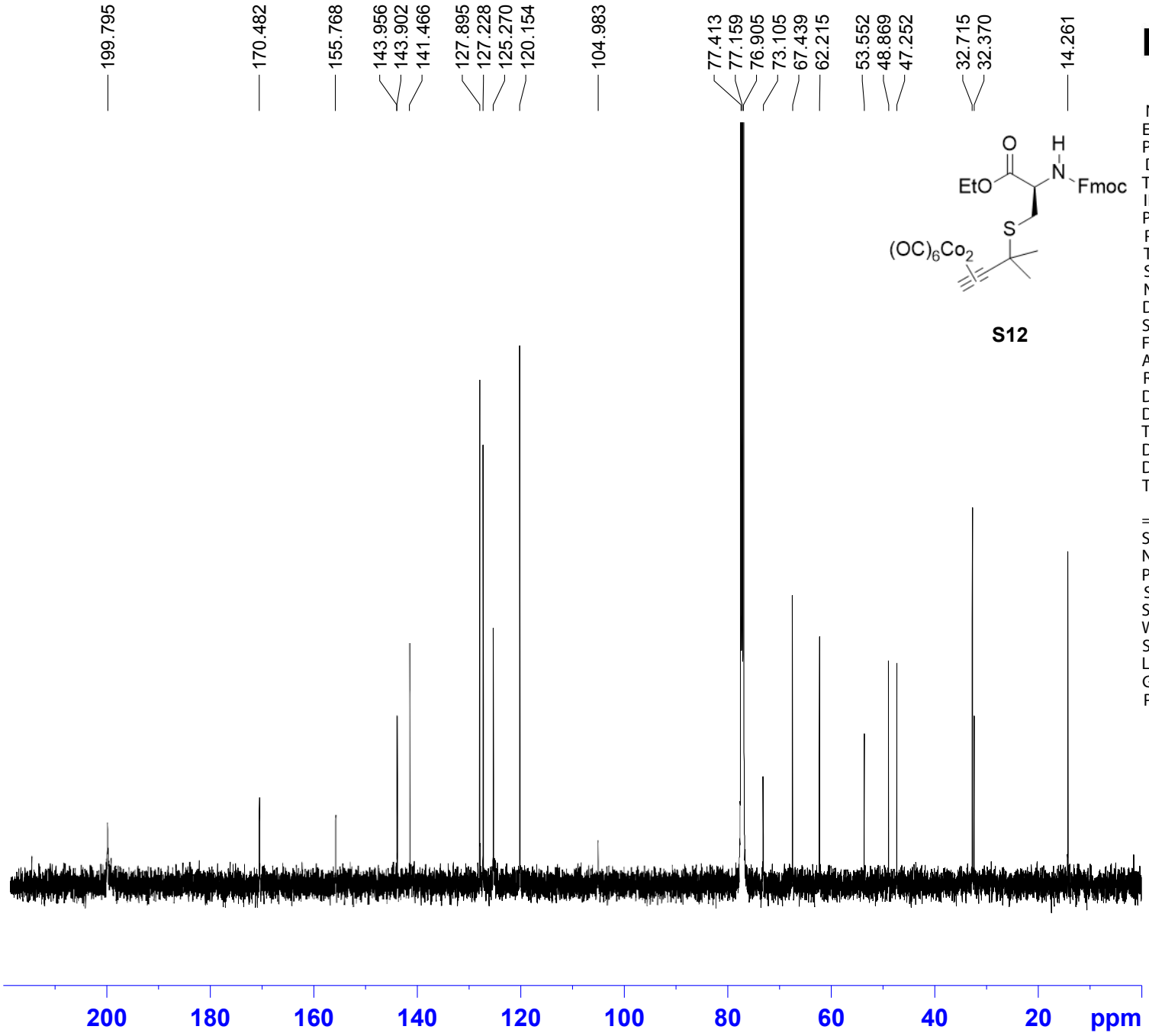
SW07−141−B



S12

NAME	SW07−141−B	
EXPNO	20	
PROCNO	1	
Date_	20151210	
Time	14.12	
INSTRUM	spect	
PROBHD 5 mm PABBO BB−		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894966	sec
RG	144	
DW	62.400	usec
DE	6.50	usec
TE	96.4	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	400.1324710	MHz
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300096	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

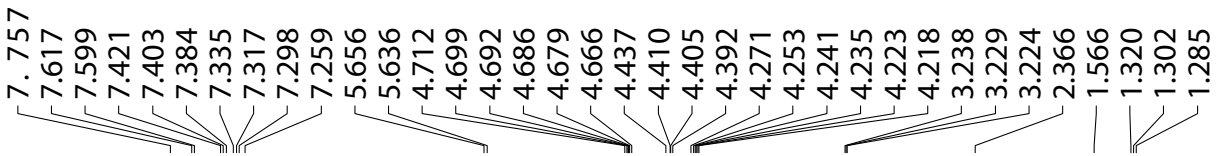
SW07−141−B 13C 500

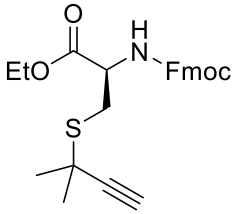


S12

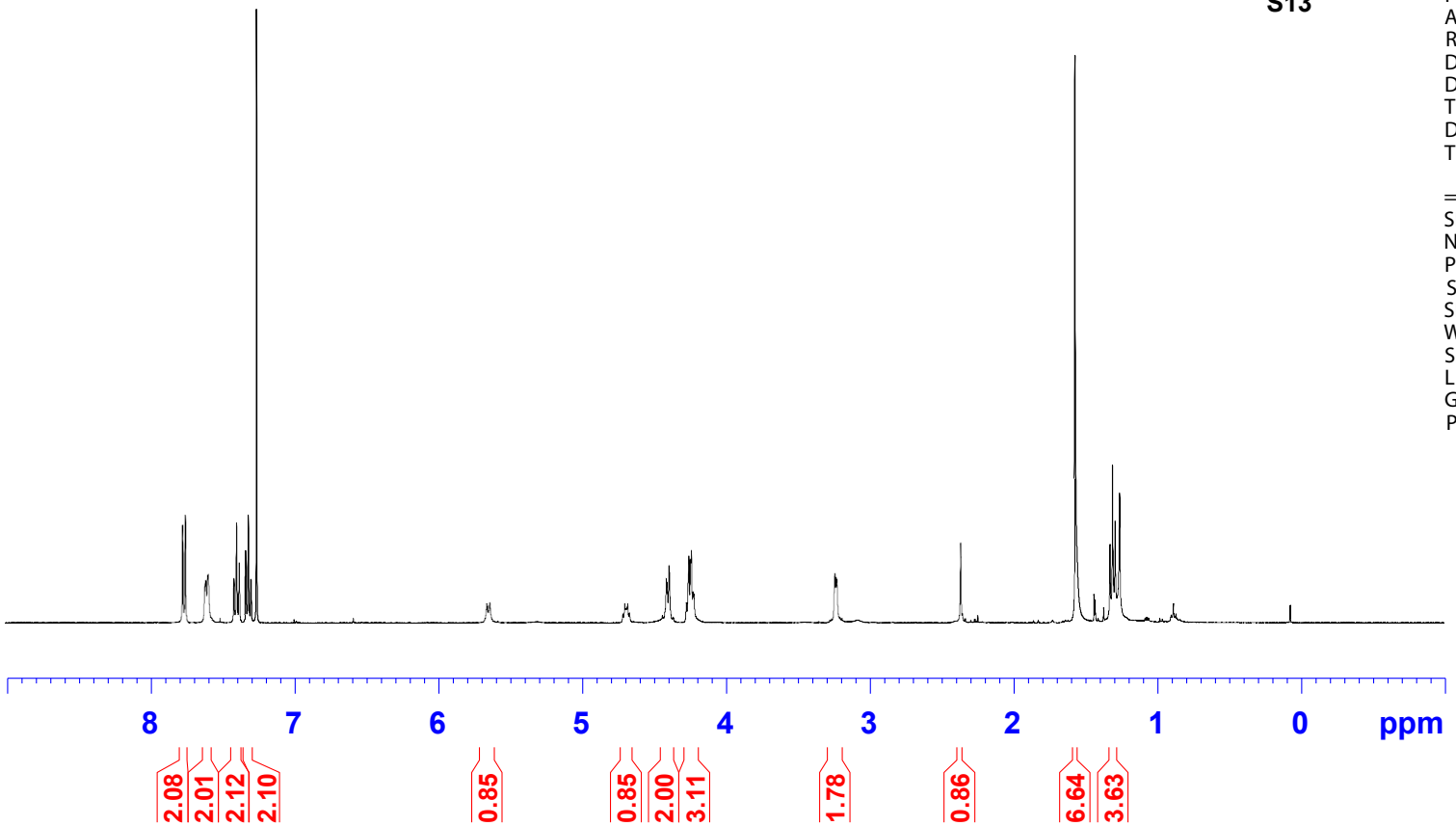
NAME	SW07−141−B	
EXPNO	10	
PROCNO	1	
Date_	20151212	
Time	1.36	
INSTRUM	spect	
PROBHD	5 mm PABBO BB/	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	5120	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	298.7	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	125.7779086	MHz
NUC1	13C	
P1	10.50	usec
SI	32768	
SF	125.7653128	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

SW07−147−A 1H 400



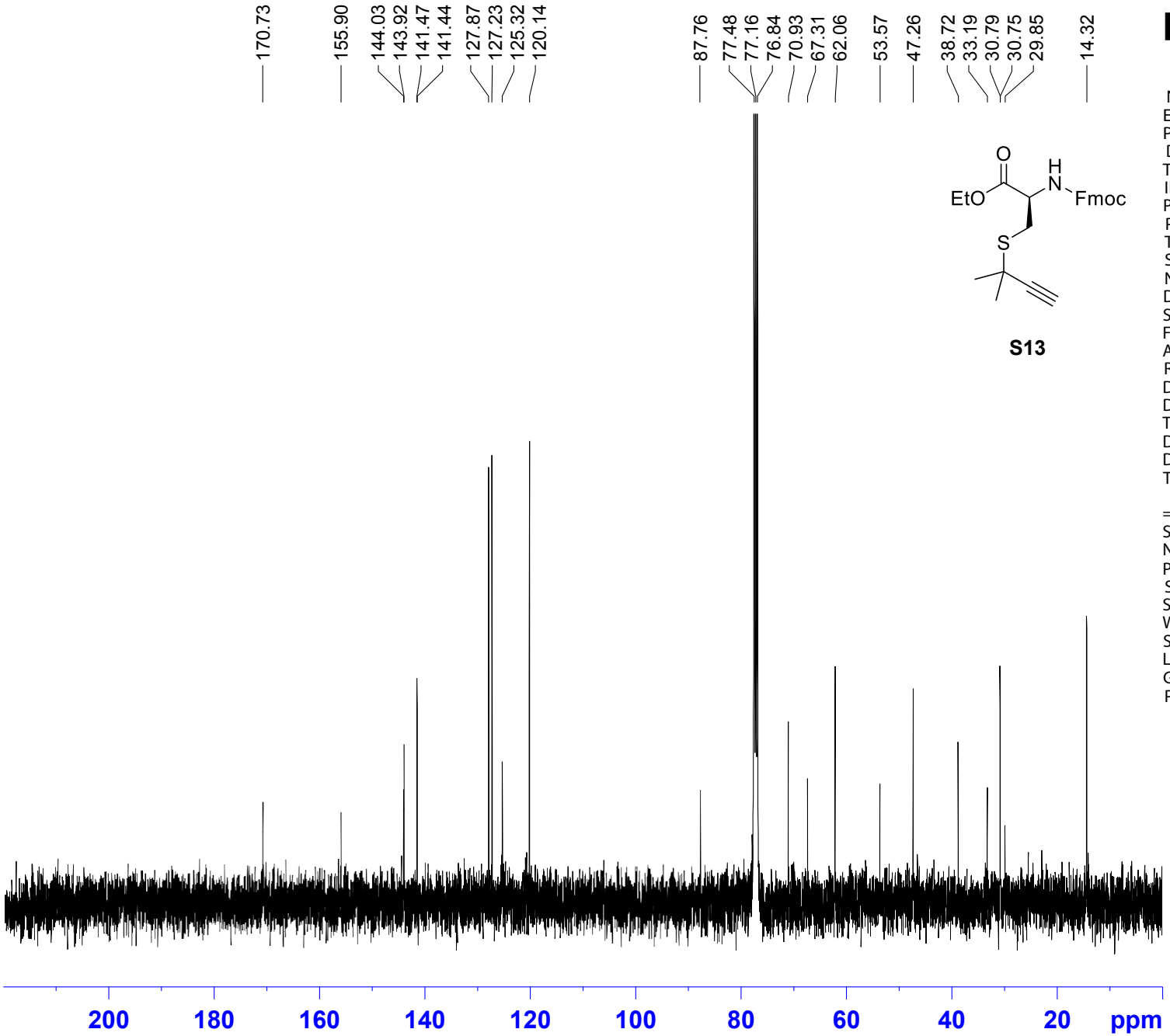


S13



NAME	SW07−147−A	
EXPNO	10	
PROCNO	1	
Date_	20151214	
Time	13.46	
INSTRUM	spect	
PROBHD 5 mm PABBO BB−		
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894966	sec
RG	144	
DW	62.400	usec
DE	6.50	usec
TE	96.5	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	400.1324710	MHz
NUC1	1H	
P1	13.75	usec
SI	65536	
SF	400.1300108	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

SW07–147–A 13C 400



S13

NAME	SW07–147–A	
EXPNO	11	
PROCNO	1	
Date_	20151214	
Time	22.59	
INSTRUM	spect	
PROBHD	5 mm PABBO BB–	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	3072	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	161	
DW	20.800	usec
DE	6.50	usec
TE	96.4	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1	========	
SFO1	100.6228293	MHz
NUC1	13C	
P1	10.00	usec
SI	32768	
SF	100.6127543	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	