

Supporting Information

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

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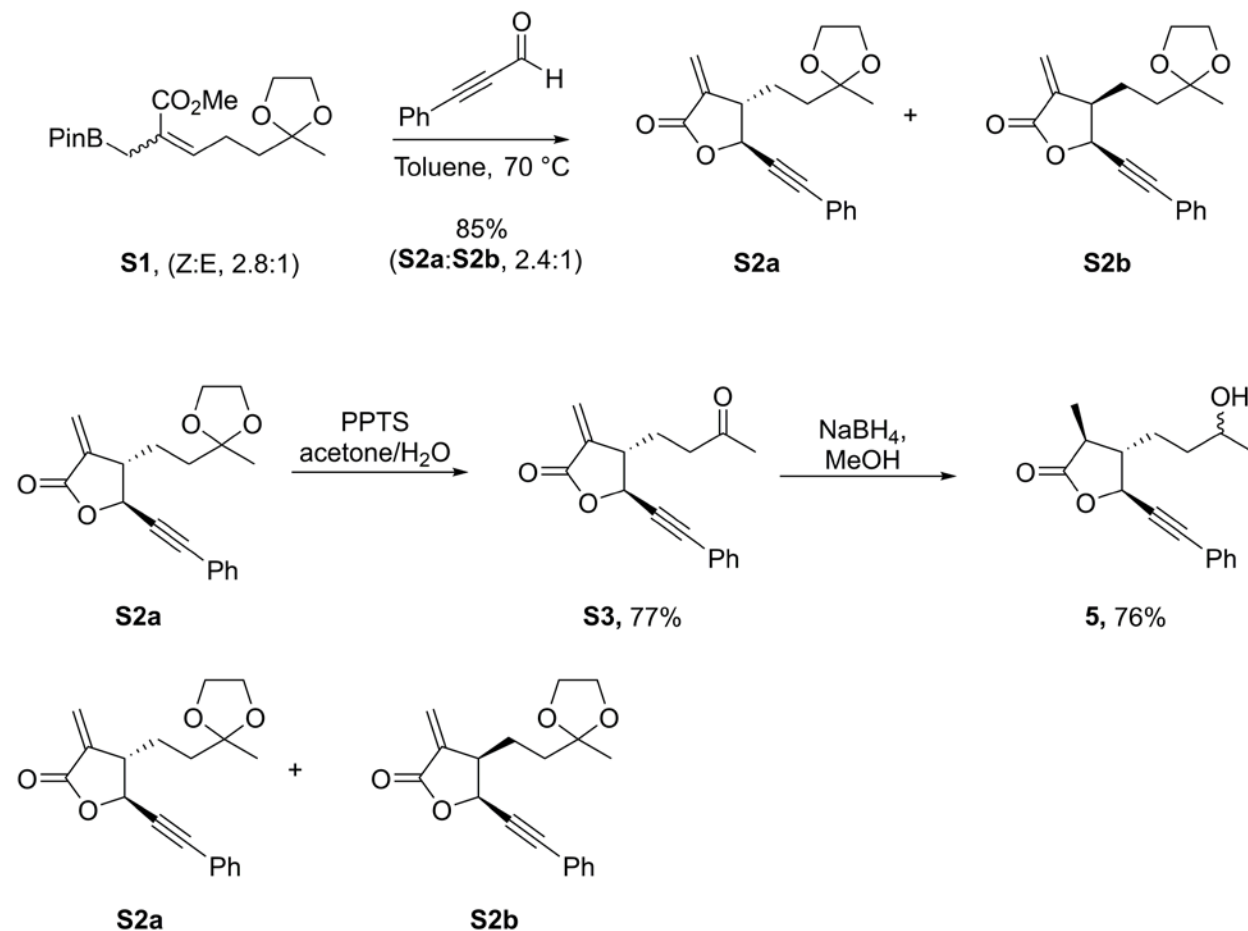
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General Methods

All commercially available compounds were used as received unless otherwise noted. Dichloromethane (DCM), diethyl ether (Et₂O), and tetrahydrofuran (THF) were purified by passing through alumina using a solvent purification system. Deuterated chloroform (CDCl₃) was stored over 4 Å molecular sieves. Boron trifluoride diethyl etherate (BF₃•OEt₂) was used as received or redistilled under a nitrogen atmosphere. Dicobalt octacarbonyl (Co₂(CO)₈) was used as purchased and was stored at -20 °C. 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 KMnO₄ stain. ¹H NMR and ¹³C 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, ¹H; 77.16 ppm, ¹³C). 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-phenylpropionaldehyde (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

¹H NMR *Trans*-lactone **S2a** (400 MHz, CDCl₃): 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, CDCl₃): 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.

¹³C NMR *Trans*-lactone **S2a** (100 MHz, CDCl₃): 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, CDCl₃): 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.

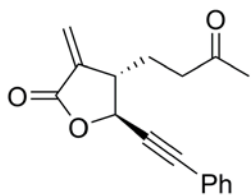
² Grillet, F.; Huang, C.; Brummond, K. M. *Org. Lett.* **2011**, *13*, 6304-6307.

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 C₁₉H₂₁O₄, 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 °C) 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

¹H NMR (400 MHz, CDCl₃)

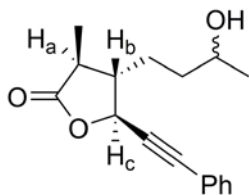
7.45-7.42 (m, 2H), 7.40-7.31 (m, 3H), 6.36 (d, $J = 2.8$ Hz, 1H), 5.69 (d, $J = 2.4$ Hz, 1H), 4.94 (d, $J = 5.6$ Hz, 1H), 3.24-3.19 (m, 1H), 2.74-2.64 (m, 2H), 2.17 (s, 3H), 2.16-2.07 (m, 1H), 1.94-1.84 (m, 1H) ppm;

^{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)
[$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{17}\text{H}_{17}\text{O}_3$, 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

¹H NMR (600 MHz, CDCl₃)

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

¹³C NMR (100 MHz, CDCl₃)

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.

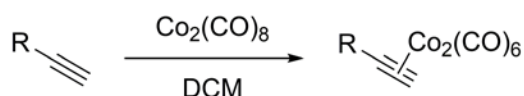
3430, 3059, 2969, 2934, 2360, 2232, 1780, 1491, 1456, 1331, 1166, 992, 759, 692
cm⁻¹;

HRMS (FTMS + p ESI Full ms)

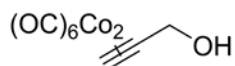
[M+H]⁺ calcd for C₁₇H₂₁O₃, 273.1485; found, 273.1468;

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 N₂ 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 (Co₂(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 $\text{Co}_2(\text{CO})_6$ -propargyl alcohol **4** in quantitative yield, as a dark red solid.

Data for **6a**

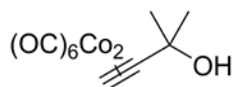
MP 48-52 °C

^1H 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 $\text{Co}_2(\text{CO})_6$ -2-methyl-3-butyn-2-ol **S4** in 92% yield, as a dark red solid.

Data for **S4**

MP 37.9-40.1 °C

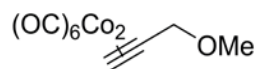
^1H NMR (300 MHz, CDCl_3)

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)

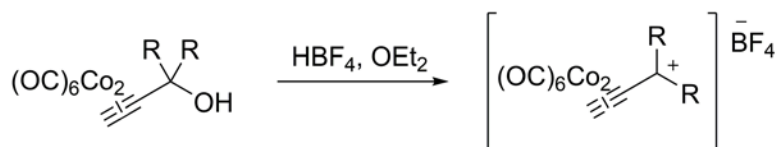


Dicobalt hexacarbonyl complexed methyl propargyl ether **6b.** Followed general procedure A: Dicobalt octacarbonyl (195 mg, 0.57 mmol, 1 equiv), dichloromethane (1.5 mL), methyl propargyl ether (40 mg, 0.57 mmol, 1 equiv), dissolved in dichloromethane (3.0 mL). The reaction was stirred for 1.5 h. The silica gel flash column chromatography was run with a gradient of 0-2.5% diethyl ether in hexanes to afford 94 mg of $\text{Co}_2(\text{CO})_6$ -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**

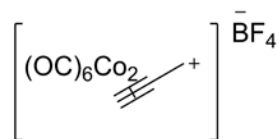
$^1\text{H NMR}$ (300 MHz, CDCl_3)
6.06 (s, 1H), 4.60 (s, 2H), 3.49 (s, 3H) ppm;

TLC R_f = 0.59 (10% diethyl ether in hexanes)
Silica gel, visible (red)

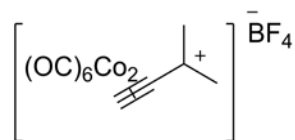


General Procedure B: Formation of propargylium tetrafluoroborate salts. A flame-dried 100 mL Schlenk flask equipped with a stir bar and septum was charged with either $\text{Co}_2(\text{CO})_6$ -propargyl alcohol **6a** or $\text{Co}_2(\text{CO})_6$ -2-methyl-3-butyn-2-ol (**S4**), dissolved in diethyl ether. The flask was cooled to $-10\text{ }^\circ\text{C}$ 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.



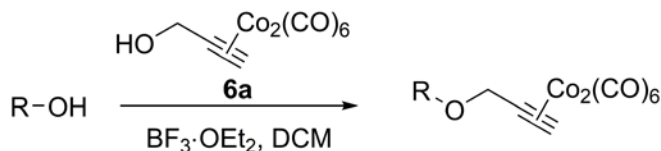
α -(Ethynyl)dicobalt hexacarbonyl carbonium tetrafluoroborate salt **6c.**⁴ Follows General procedure B: $\text{Co}_2(\text{CO})_6$ -propargyl alcohol **6a** (500 mg, 1.46 mmol, 1 equiv), diethyl ether (5 mL), tetrafluoroboric acid (356 mg of a 54% by weight soln in diethyl ether, 2.19 mmol, 1.5 equiv). 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: $\text{Co}_2(\text{CO})_6$ -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.

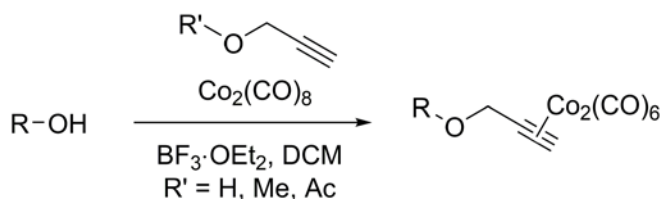
⁴ Conner, R. E.; Nicholas, K. M; *J. Organomet. Chem.*, **1977**, 125, C45-C48

General Procedure C: Nicholas Reaction Procedures



General Procedure C1: Use of pre-made dicobalt hexacarbonyl complexed alkyne. A

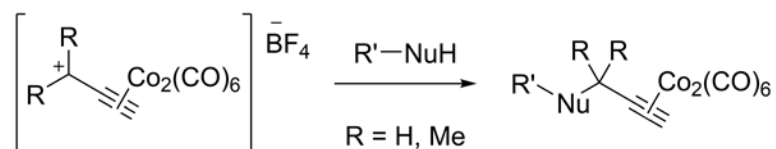
single-necked, round-bottomed flask equipped with a stir bar and a septum pierced with a needle was charged with dichloromethane (0.05 M), $\text{Co}_2(\text{CO})_6$ -propargyl alcohol **6a** or $\text{Co}_2(\text{CO})_6$ -methyl propargyl ether **6b** (2 equiv), and the nucleophilic species (1 equiv). 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

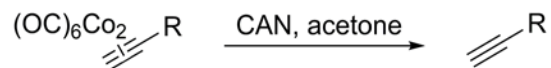
single necked, round-bottomed flask, equipped with a stir bar and a septum was charged with dicobalt octacarbonyl (2 equiv) in a N_2 filled glove box. The flask was transferred out of the glovebox and the septum was pierced with a nitrogen inlet needle. Either propargyl alcohol, methyl propargyl ether, 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.



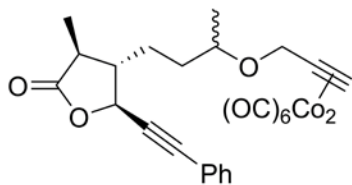
General Procedure C3: Reaction of tetrafluoroborate salts with nucleophiles. A single-necked, round-bottomed flask, equipped with a stir bar and a septum was charged with propargylium tetrafluoroborate salt **6c** or **12** (1.3 equiv) in a N₂ filled glove box. The flask was transferred out of the glove 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 $\text{Co}_2(\text{CO})_6$ -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:

$\text{Co}_2(\text{CO})_6$ -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 μ L, 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 μ L, 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

$^1\text{H NMR}$ (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

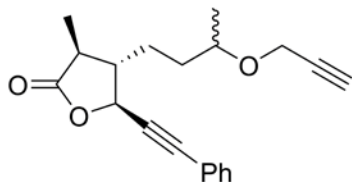
$^{13}\text{C NMR}$ (125 MHz, CDCl_3)
199.8 (6C), 177.9, 131.9 (2C), 129.2, 128.5 (2C), 121.9, 92.7, 87.9, 85.1, 75.2, 75.0*, 73.1, 71.3, 68.6, 51.2, 51.0*, 41.3, 34.5, 34.3*, 27.8, 27.5*, 19.3, 14.6, 14.4* ppm;

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 $\text{C}_{26}\text{H}_{22}\text{O}_9\text{Co}_2\text{Na}$, 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**

$^1\text{H NMR}$ (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;

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

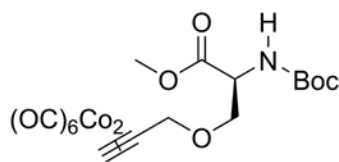
177.9, 131.93 (2C), 131.91 (2C)*, 129.2, 128.6 (2C), 121.8, 87.9, 85.0, 80.4, 74.1, 73.7, 73.1, 55.8, 55.7*, 51.0, 50.6*, 41.3, 34.2, 19.2, 14.7, 14.5* ppm;

* 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 C₂₀H₂₃O₃, 311.1642; found, 311.1631;

TLC R_f = 0.50 (30% ethyl acetate in hexanes)
Silica gel, UV active



Co₂(CO)₆-N-[(1,1-dimethylethoxy)carbonyl]-O-(prop-2-yn-1-yl)serine methyl ester (10a).

Method A. Follows general procedure C1: Co₂(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 μ L, 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

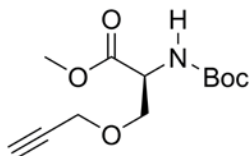
^1H NMR (500 MHz, CDCl_3):
6.02 (s, 1H), 5.35 (d, $J = 7.5$ Hz, 1H), 4.64 (s, 2H), 4.49 (broad d, $J = 8.5$ Hz, 1H), 4.07 (d, $J = 8.0$ Hz, 1H), 3.85 (d, $J = 7.0$ Hz, 1H), 3.74 (s, 3H), 1.45 (s, 9H) ppm;

^{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)
[$\text{M}+\text{Na}$] $^+$ calc'd for $\text{C}_{18}\text{H}_{19}\text{Co}_2\text{NO}_{11}\text{Na}$ 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**

¹H NMR (500 MHz, CDCl₃)

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;

¹³C NMR (125 MHz, CDCl₃)

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)

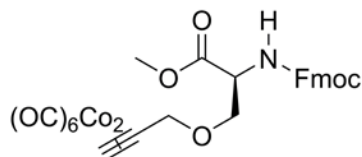
3429, 3297, 2951, 2922, 2851, 2341, 1748, 1717, 1501, 1462, 1440, 1388, 1366, 1300, 1248, 1210, 1165, 1108, 1062, 1024 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+Na]⁺ calc'd for C₁₂H₁₉NO₅Na 280.1155 m/z; found 280.1148 m/z;

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

Silica gel, potassium permanganate stain



Co₂(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 μ L, 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 μ L, 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 μ L, 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**

¹H NMR (500 MHz, CDCl₃):

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;

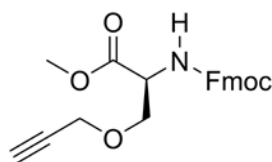
⁶ Zhang, F.; Zhang, W.; Zhang, Y.; Curran, D. P.; Liu, G. *J. Org. Chem.* **2009**, *74*, 2594-2597.

^{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)
[$\text{M}+\text{Na}$] $^+$ calc'd for $\text{C}_{28}\text{H}_{21}\text{Co}_2\text{NO}_{11}\text{Na}$ 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

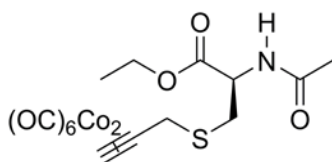
^1H NMR (500 MHz, CDCl_3):
7.77 (d, $J = 7.5$ Hz, 2H), 7.62 (app t, $J = 7.0$ Hz, 2H), 7.41 (app t, $J = 7.5$ Hz, 2H),
7.32 (app t, $J = 7.0$ Hz, 2H), 5.66 (d, $J = 8.0$ Hz, 1H), 4.47-4.35 (m, 1H), 4.41 (m,
2H), 4.25 (t, $J = 7.5$ Hz, 1H), 4.17 (s, 2H), 4.01 (dd, $J = 9.5, 3.0$ Hz, 1H), 3.81
(dd, $J = 9.5, 3.0$ 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 $\text{C}_{22}\text{H}_{21}\text{Co}_2\text{NO}_5$ 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**

^1H NMR (300 MHz, CDCl_3)

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

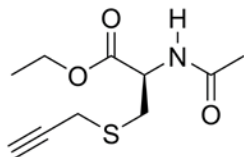
6.31 (d, $J = 4.5$ Hz, 1H), 6.14 (s, 1H), 4.87-4.85 (m, 1H), 4.25-4.23 (m, 2H), 4.02-3.91 (m, 2H), 3.19 (dd, $J = 13.5, 4.4$ Hz, 1H), 3.06 (dd, $J = 13.8, 4.7$ Hz, 1H), 2.03 (s, 3H), 1.30 (t, $J = 6.9$ Hz, 3H) ppm;

^{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)
[$\text{M}+\text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{15}\text{O}_9\text{NC}_2\text{NaS}$, 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

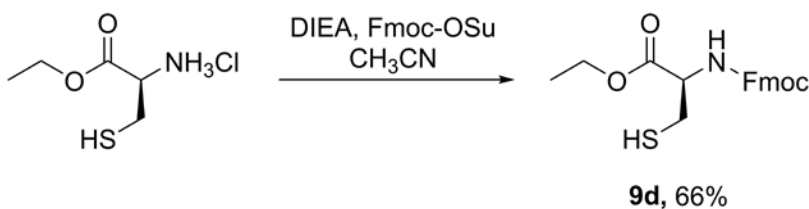
^1H NMR (500 MHz, CDCl_3)
6.42 (bs, 1H), 4.86 (dt, $J = 7.5, 5.0$ Hz, 1H), 4.25 (q, $J = 7.0$ Hz, 2H), 3.31 (dd, $J = 17.0, 2.5$ Hz, 1H), 3.25-3.21 (m, 2H), 3.14 (dd, $J = 14.3, 5.3$ Hz, 1H), 2.30 (t, $J = 2.5$ Hz, 1H), 2.08 (s, 3H), 1.31 (t, $J = 7.0$ Hz, 3H) ppm;

^{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 $\text{C}_{10}\text{H}_{16}\text{O}_3\text{NS}$, 230.0845; found, 230.0846;

TLC $R_f = 0.23$ (50% ethyl acetate in hexanes)
Silica gel, potassium permanganate



***N*-Fluorenylmoethoxycarbonyl-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

¹H NMR (400 MHz, CDCl₃)

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;

¹³C NMR (100 MHz, CDCl₃)

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)

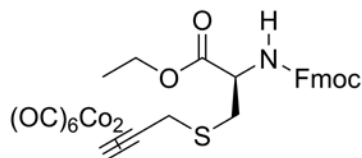
3337, 3065, 2981, 1723, 1513, 1450, 1339, 1204, 1035, 759, 741 cm⁻¹.

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₂₀H₂₂O₄NS, 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 μ L, 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: $\text{Co}_2(\text{CO})_6$ -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 μ L, 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**

^1H 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;

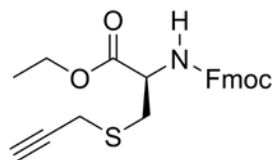
IR (thin film)
3345, 3070, 2923, 2094, 2053, 2023, 1726, 1507, 1450, 1339, 1204, 1052, 759, 741 cm^{-1} .

HRMS (FTMS + p ESI Full ms)

$[M+H]^+$ calcd for $C_{29}H_{24}O_{10}NC_2S$, 695.9779; found, 695.9745;

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

Silica gel, visible, UV



Ethyl *N*-(((9*H*-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**

1H 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)

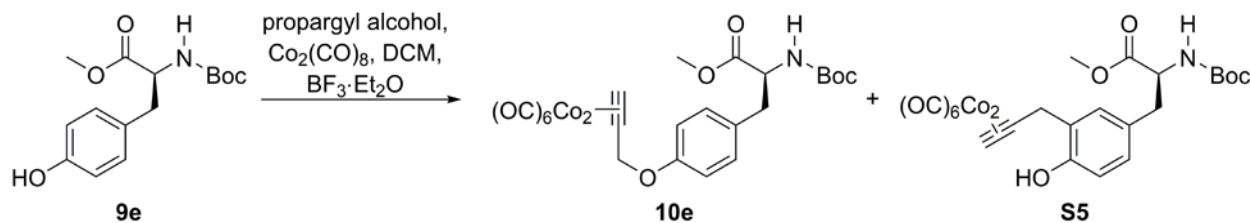
3291, 2924, 1723, 1517, 1450, 1339, 1210, 1051, 760, 741 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₂₃H₂₄O₄NS, 410.1421; found, 410.1424;

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

Silica gel, UV



Co₂(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 ¹H 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**

¹H NMR (500 MHz, CDCl₃):

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

¹³C NMR (125 MHz, CDCl₃):

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 C₂₄H₂₃Co₂NO₁₀Na 625.9878 m/z; found 625.9877 m/z;

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

Silica gel, visible, UV

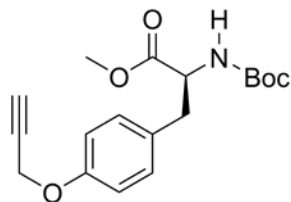
Data for **S5**

¹H NMR (400 MHz, CDCl₃)

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, 1H), 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**

^1H 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)

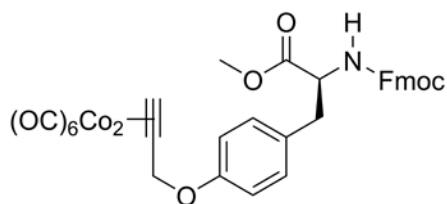
3368, 3288, 2977, 2956, 2929, 2868, 2121, 1743, 1712, 1611, 1586, 1506, 1438,
1392, 1366, 1218, 1165, 1113, 1058, 1027, 924, 825, 644 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calc'd for C₁₈H₂₄NO₅ 334.1649 m/z; found 334.1640 m/z

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

Silica gel, visible, UV



Co₂(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

¹H NMR (500 MHz, CDCl₃):

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;

¹³C NMR (125 MHz, CDCl₃):

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)

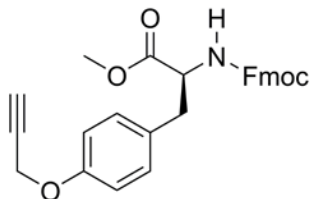
3429, 3066, 3033, 2951, 2923, 2896, 2852, 2096, 2054, 2022, 1724, 1610, 1583, 1510, 1449, 1347, 1242, 1213, 1177, 1033, 757, 741, 518, 494 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calc'd for C₃₄H₂₅Co₂NO₁₁ 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

¹H NMR (500 MHz, CDCl₃):

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;

¹³C NMR (125 MHz, CDCl₃):

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)

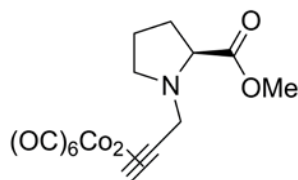
3286, 2956, 2923, 2852, 2049, 2022, 1723, 1608, 1511, 1462, 1449, 1377, 1344, 1259, 1240, 1218, 1177, 1108, 1051, 1026, 757, 741 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calc'd for C₂₈H₂₅NO₅ 456.1806 m/z; found 456.1793 m/z;

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

Silica gel, visible, UV



Co₂(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

¹H NMR (300 MHz, CDCl₃)

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;

¹³C NMR (100 MHz, CDCl₃)

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)

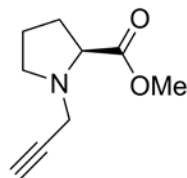
2955, 2798, 2093, 2020, 1736, 1551, 1437, 1356, 1278, 1199, 1173 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₁₅H₁₄O₈NC₂, 453.9378; found, 453.9361;

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

Silica gel, UV, potassium permanganate



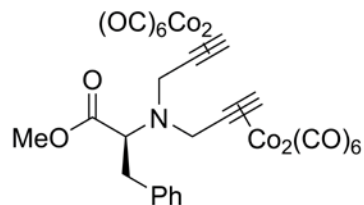
N-propargyl-L-proline methyl ester (11g). Followed general procedure D: Dicobalt hexacarbonyl complexed alkyne **10g** (20 mg, 0.044 mmol), acetone (5.0 mL), and ceric ammonium nitrate (97 mg, 0.18 mmol). After 1 h of stirring, **10g** remained, as evidenced by proton NMR. An additional amount of ceric 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 ^1H 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**

^1H 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 $\text{C}_9\text{H}_{14}\text{O}_2\text{N}$, 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 ether 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

¹H NMR (300 MHz, CDCl₃)

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;

¹³C NMR (100 MHz, CDCl₃)

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)

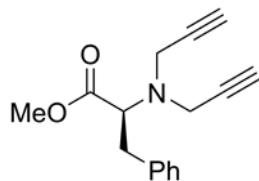
2093, 3052, 2017, 1735, 1425, 1200, 1165 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[*M*+*H*]⁺ calcd for C₂₈H₁₈O₁₄NC₀₄, 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

¹H NMR (400 MHz, CDCl₃)

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;

¹³C NMR (100 MHz, CDCl₃)

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)

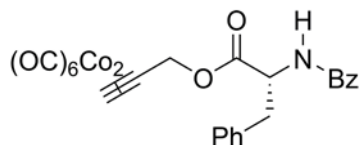
3250, 2991, 2914, 2813, 2344, 1714, 1478, 1421, 1347, 1199, 1153, 1112, 740, 692, 623 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₁₆H₁₈O₂N, 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 μ L, 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

¹H NMR (400 MHz, CDCl₃)

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;

¹³C NMR (125 MHz, CDCl₃)

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)

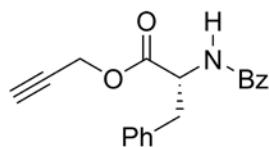
3031, 2925, 2097, 2056, 2025, 1746, 1647, 1531, 1487, 1178, 700 cm⁻¹.

HRMS (FTMS + p ESI Full ms)

[M+Na]⁺ calcd for C₂₅H₁₇O₉NCO₂, 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

¹H NMR (400 MHz, CDCl₃)

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

¹³C NMR (125 MHz, CDCl₃)

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)

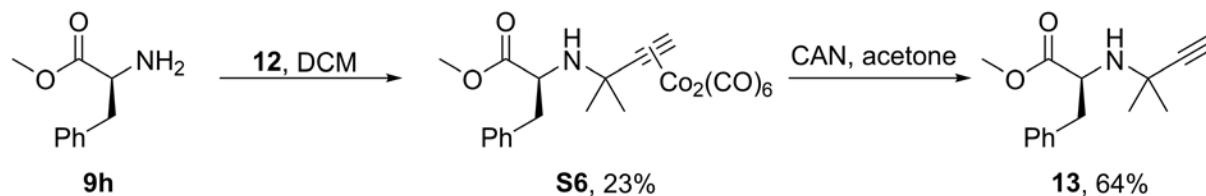
3396, 3277, 3070, 2920, 2851, 2131, 1762, 1647, 1521, 1488, 1205, 1171 cm⁻¹.

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₁₉H₁₇O₃N, 308.1281; found, 308.1282;

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

Silica gel, UV



Co₂(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 ether in hexanes) to afford 16 mg of cobalt complexed alkyne **S6** in 23% yield as a red oil.

Data for S6

¹H NMR (400 MHz, CDCl₃)

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;

¹³C NMR (100 MHz, CDCl₃)

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)

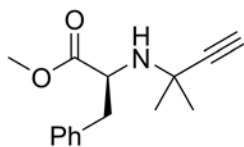
2973, 2927, 2092, 2050, 2019, 1739, 1455, 1194, 1172, 700 cm⁻¹;

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₂₁H₂₀O₈NC₂, 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: $\text{Co}_2(\text{CO})_6$ -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

^1H 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;

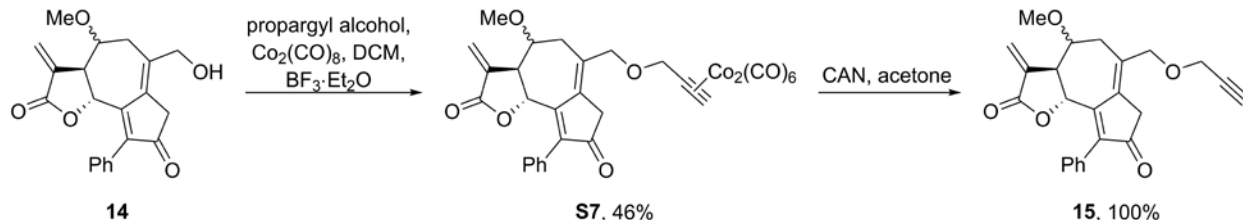
^{13}C NMR (100 MHz, CDCl_3)
176.3, 137.5, 129.6 (2C), 128.4 (2C), 126.8, 88.5, 70.1, 58.9, 51.7, 49.6, 41.4,
30.3, 29.6 ppm;

IR (thin film)
3286, 3027, 2977, 2929, 2368, 1736, 1458, 1438, 1196, 1171, 700 cm^{-1} ;

HRMS (FTMS + p ESI Full ms)
[$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2\text{N}$, 246.1489; found, 246.1478;

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

Silica gel, UV, potassium permanganate



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 μ L, 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

¹H NMR (400 MHz, CDCl₃)

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;

¹³C NMR (100 MHz, CDCl₃)

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.

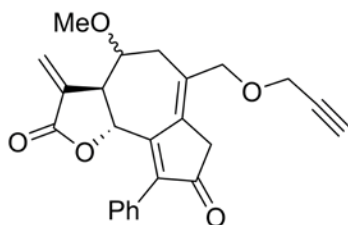
HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₃₀H₂₃O₁₁Co₂, 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

¹H NMR (400 MHz, CDCl₃)

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

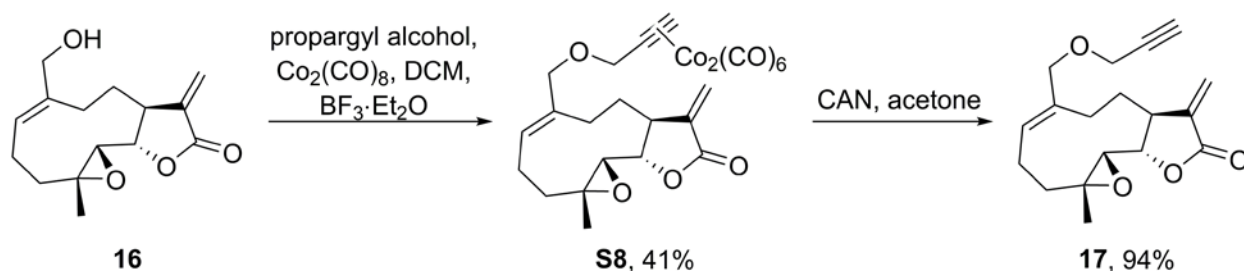
(FTMS + p ESI Full ms)

$[\text{M}+\text{H}]^+$ calcd for $\text{C}_{24}\text{H}_{23}\text{O}_5$, 391.1540; found, 391.1525;

TLC

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

Silica gel, UV, potassium permanganate



Co₂(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 μ L, 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 μ L, 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

¹H NMR (400 MHz, CDCl₃)

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)

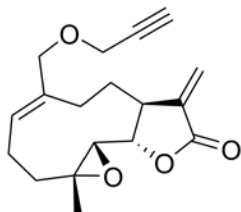
2931, 2360, 2095, 2053, 2023, 1770, 1262, 1138, 1075, 995 cm^{-1} ;

HRMS (FTMS + p ESI Full ms)

$[\text{M}+\text{H}]^+$ calcd for $\text{C}_{24}\text{H}_{23}\text{O}_{10}\text{Co}_2$, 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: $\text{Co}_2(\text{CO})_6$ -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

^1H 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} ;

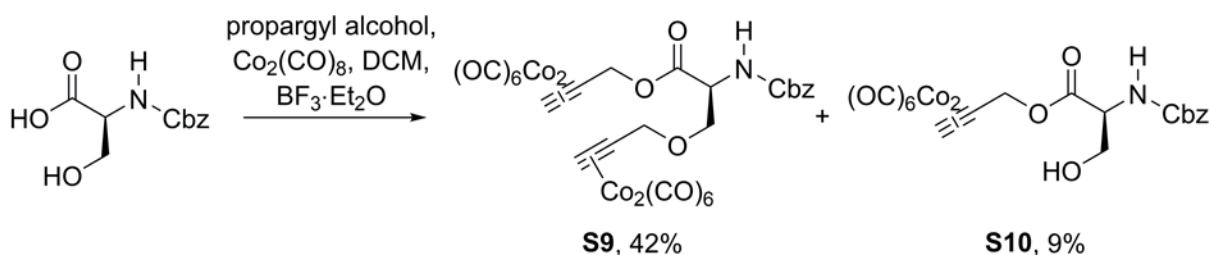
HRMS (FTMS + p ESI Full ms)

$[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{23}\text{O}_4$, 303.1591; found, 303.1595;

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

Silica gel, UV, potassium permanganate

Additional Examples



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

¹H NMR (400 MHz, CDCl₃)

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;

¹³C NMR (100 MHz, CDCl₃)

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 C₂₉H₁₇O₁₇NC₄Na, 909.7717; found, 909.7753;

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

Silica gel, visible, UV

Data for S10

¹H NMR (400 MHz, CDCl₃)

7.37-7.33 (m, 5H), 6.08 (s, 1H), 5.72-5.63 (m, 1H), 5.43 (d, *J* = 14.2 Hz, 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;

¹³C NMR (125 MHz, CDCl₃)

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)

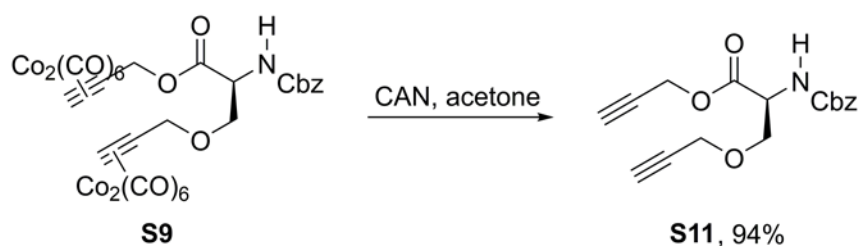
3439, 3091, 2933, 2360, 2098, 2057, 2026, 1725, 1521, 1456, 1333, 1191, 1063, 971, 698 cm^{-1} ;

HRMS (FTMS + p ESI Full ms)

$[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{20}\text{H}_{15}\text{O}_{11}\text{NCo}_2\text{Na}$, 585.9201; found, 585.9205;

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

Silica gel, visible, UV



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**

¹H NMR (400 MHz, CDCl₃)

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

¹³C NMR (100 MHz, CDCl₃)

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)

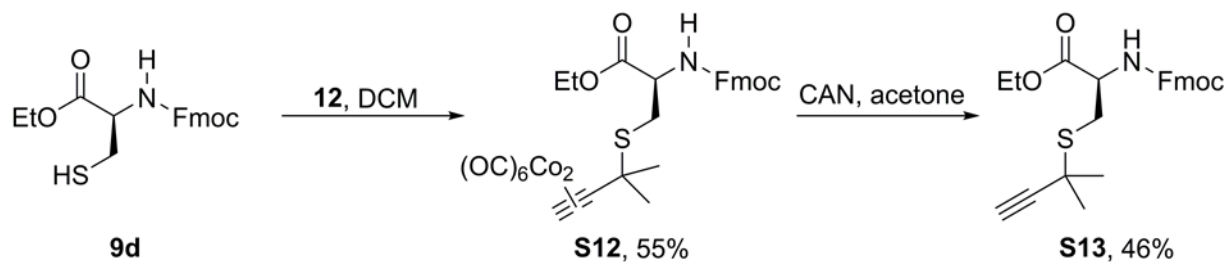
3288, 2915, 2850, 2129, 1750, 1715, 1515, 1456, 1342, 1260, 1027 cm⁻¹.

HRMS (FTMS + p ESI Full ms)

[M+H]⁺ calcd for C₁₇H₁₈O₅N, 316.1180; found, 316.1181;

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

Silica gel, UV, potassium permanganate



Co₂(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

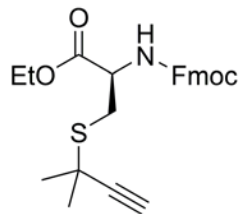
¹H NMR (400 MHz, CDCl₃)
7.77 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.41 (app t, *J* = 7.4 Hz, 2H),
7.32 (app t, *J* = 7.4 Hz, 2H), 6.23 (s, 1H), 5.59 (d, *J* = 7.6 Hz, 1H), 4.71-4.66 (m,
1H), 4.39 (d, *J* = 7.2 Hz, 2H), 4.27-4.21 (m, 3H), 3.19-3.10 (m, 2H), 1.621 (s,
3H), 1.616 (s, 3H), 1.30 (t, *J* = 7.2 Hz, 3H) ppm;

¹³C NMR (125 MHz, CDCl₃)
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+NH₄]⁺ calcd for C₃₁H₃₁O₁₀N₂SCo₂, 741.0358; found, 741.0379;

TLC R_f = 0.18 (20% diethyl ether in hexanes)
Silica gel, visible, UV



***N*-(((9*H*-fluoren-9-yl)methoxy)carbonyl)-*S*-(1,1-dimethyl-3-propynyl)-*L*-cysteine ethyl ester (**S13**). Followed general procedure D: $\text{Co}_2(\text{CO})_6$ -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**

^1H 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)

$[\text{M}+\text{H}]^+$ calcd for $\text{C}_{25}\text{H}_{28}\text{O}_4\text{NS}$, 438.1734; found, 438.1725;

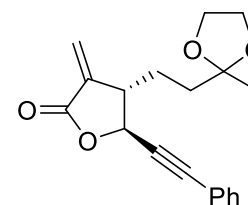
TLC $R_f = 0.23$ (30% diethyl ether in hexanes)

Silica gel, UV, potassium permanganate

SW07-024-B 1H 400



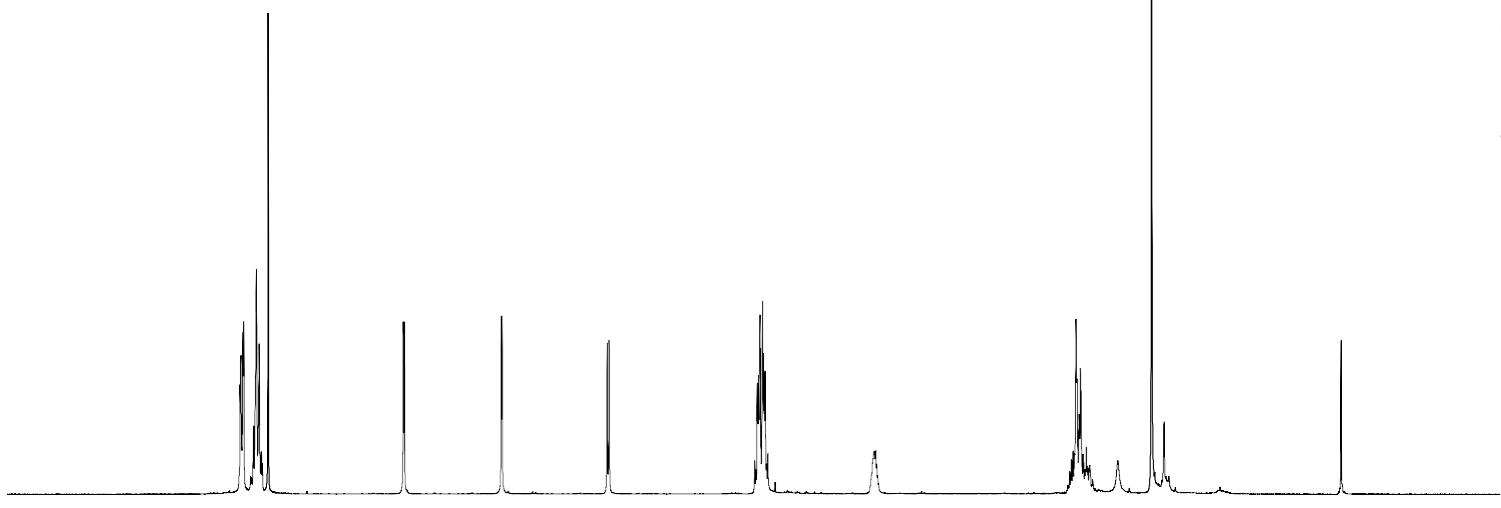
7.448
7.444
7.429
7.425
7.359
7.351
7.348
7.340
7.329
7.321
7.306
7.260
6.351
6.345
5.696
5.690
4.988
4.975
4.000
3.983
3.979
3.974
3.969
3.966
3.962
3.950
3.947
3.943
3.938
3.933
3.929
3.912
3.203
3.196
3.190
1.882
1.851
1.842
1.822
1.782
1.337



S2a

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



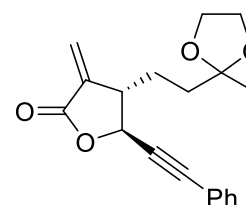
8 7 6 5 4 3 2 1 0 ppm

2.03
3.06
0.99
0.98
0.96
4.11
1.00
4.08
3.13

SW07-009-B 13C 400



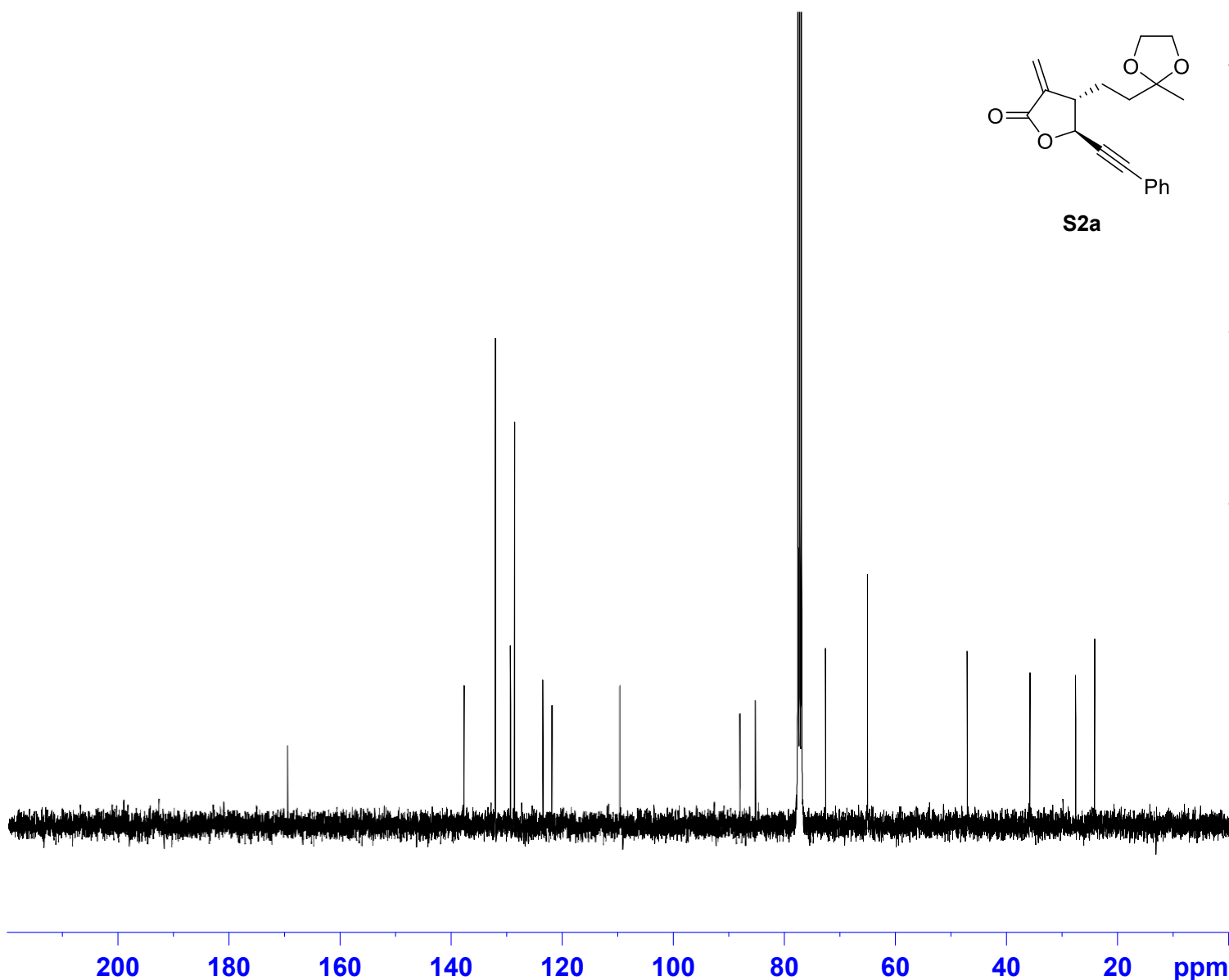
169.40
137.66
131.96
129.26
128.53
123.30
121.68
109.51
87.97
85.23
77.48
77.16
76.84
72.50
64.95
64.93
46.98
35.78
27.54
24.14



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 2.0000000 sec
D11 0.0300000 sec

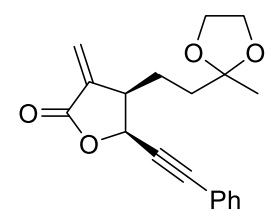
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SW07-024-C 1H 500

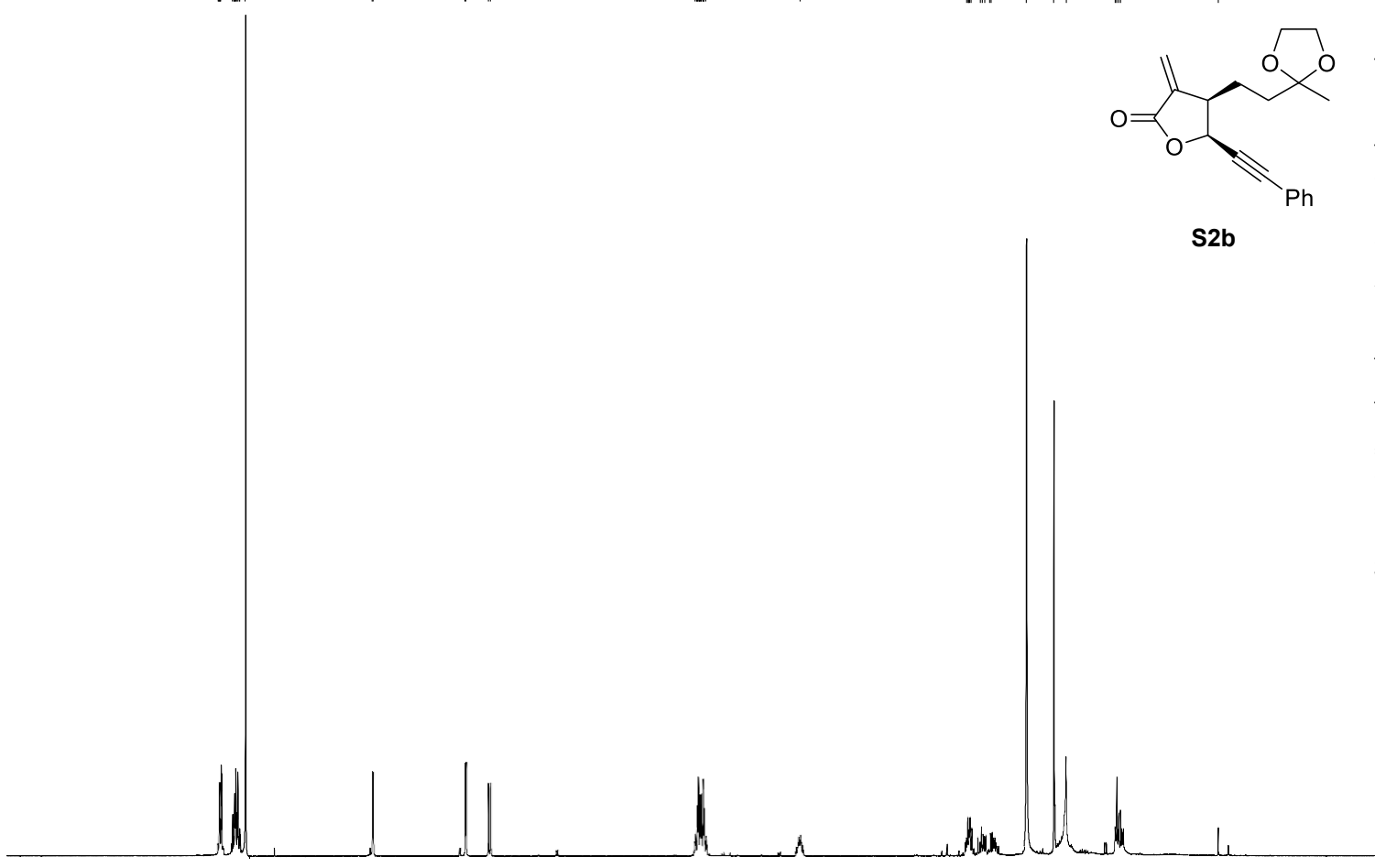


7.458
7.455
7.452
7.446
7.442
7.439
7.354
7.343
7.340
7.336
7.331
7.329
7.320
7.317
7.304
7.299
7.260
6.331
6.325
5.652
5.646
5.483
5.467
3.970
3.958
3.956
3.953
3.949
3.941
3.938
3.936
3.930
3.926
3.924
3.913
3.910
3.906
3.904
3.892
3.203
1.981
1.972
1.967
1.962
1.950
1.881
1.868
1.849
1.814
1.802
1.545
1.344
1.254
0.897
0.883
0.869
0.856
0.143



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



8 7 6 5 4 3 2 1 0 ppm

2.21 3.40
 0.99
 1.00 0.99
 4.40
 1.08
 1.99 1.31 1.32 8.27 3.40 3.34 2.59

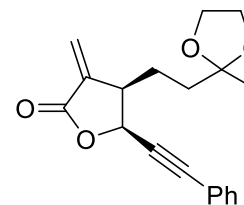
SW07-024-C 13C 500



— 169.724
— 137.694
— 132.025
— 129.266
— 128.528
— 122.543
— 121.726
— 109.731

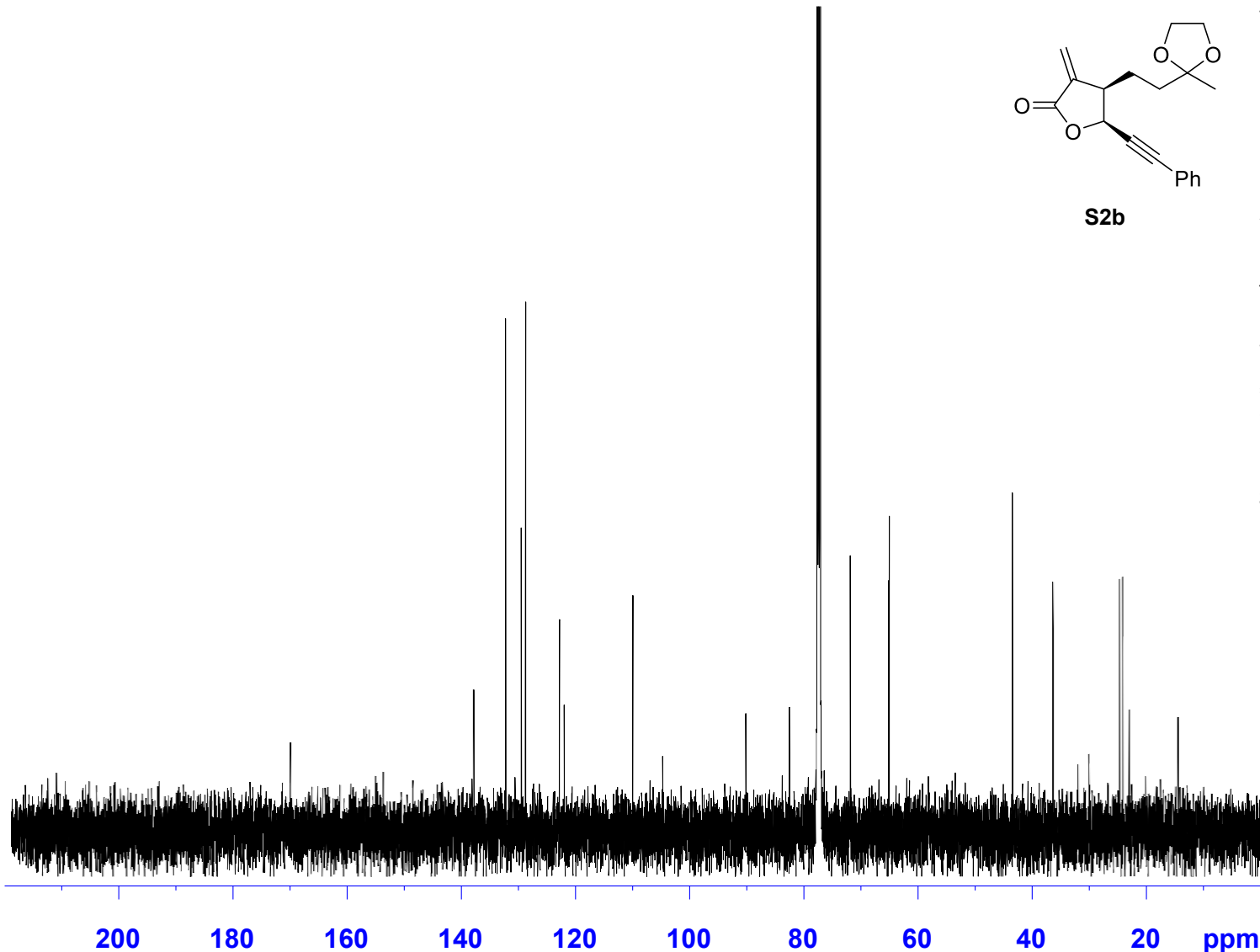
— 89.935
— 82.329
— 77.411
— 77.157
— 76.903
— 71.627
— 64.891
— 64.803

— 43.281
— 36.108
— 24.520
— 23.936
— 22.801
— 14.260



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

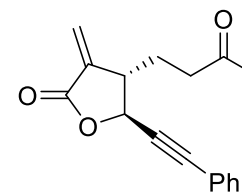
===== CHANNEL f1 =====
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653131 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SW07-033-A



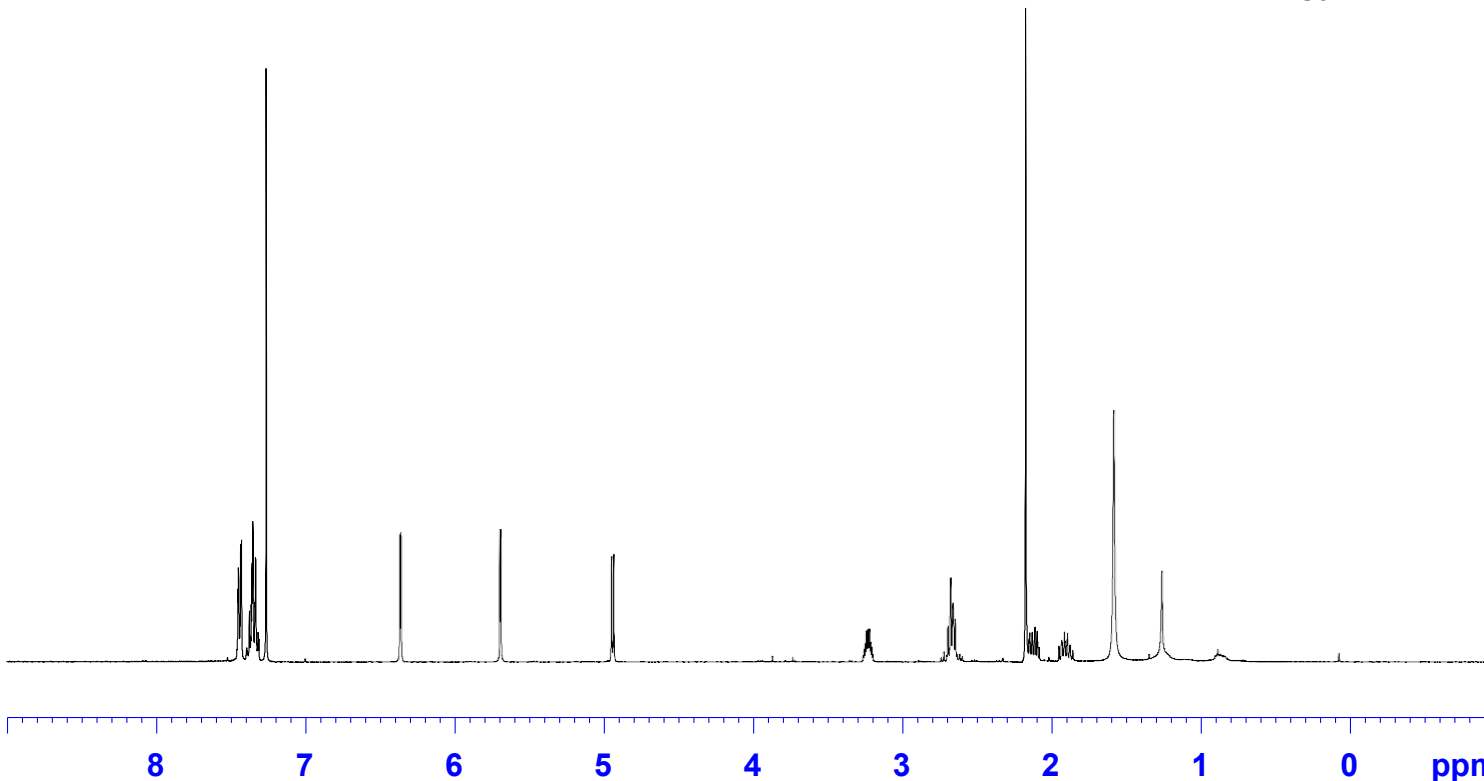
7.449
7.446
7.435
7.430
7.425
7.371
7.364
7.360
7.355
7.349
7.345
7.337
7.334
7.330
7.315
7.309
7.260
6.361
6.354
5.692
5.686
4.945
4.931
3.240
3.23 2
3.225
3.218
2.692
2.674
2.662
2.659
2.656
2.644
2.168
2.145
2.141
2.126
2.124
2.105
2.091
1.903
1.883
1.573
1.251



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



1.97
3.02
1.00
1.00
0.97
0.98
2.04
2.95
1.19
1.11
4.21
1.46
0.39

SW07-033-A 13C



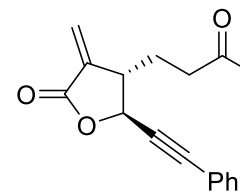
207.04

169.07

137.41
132.00
129.43
128.60
123.26
121.50

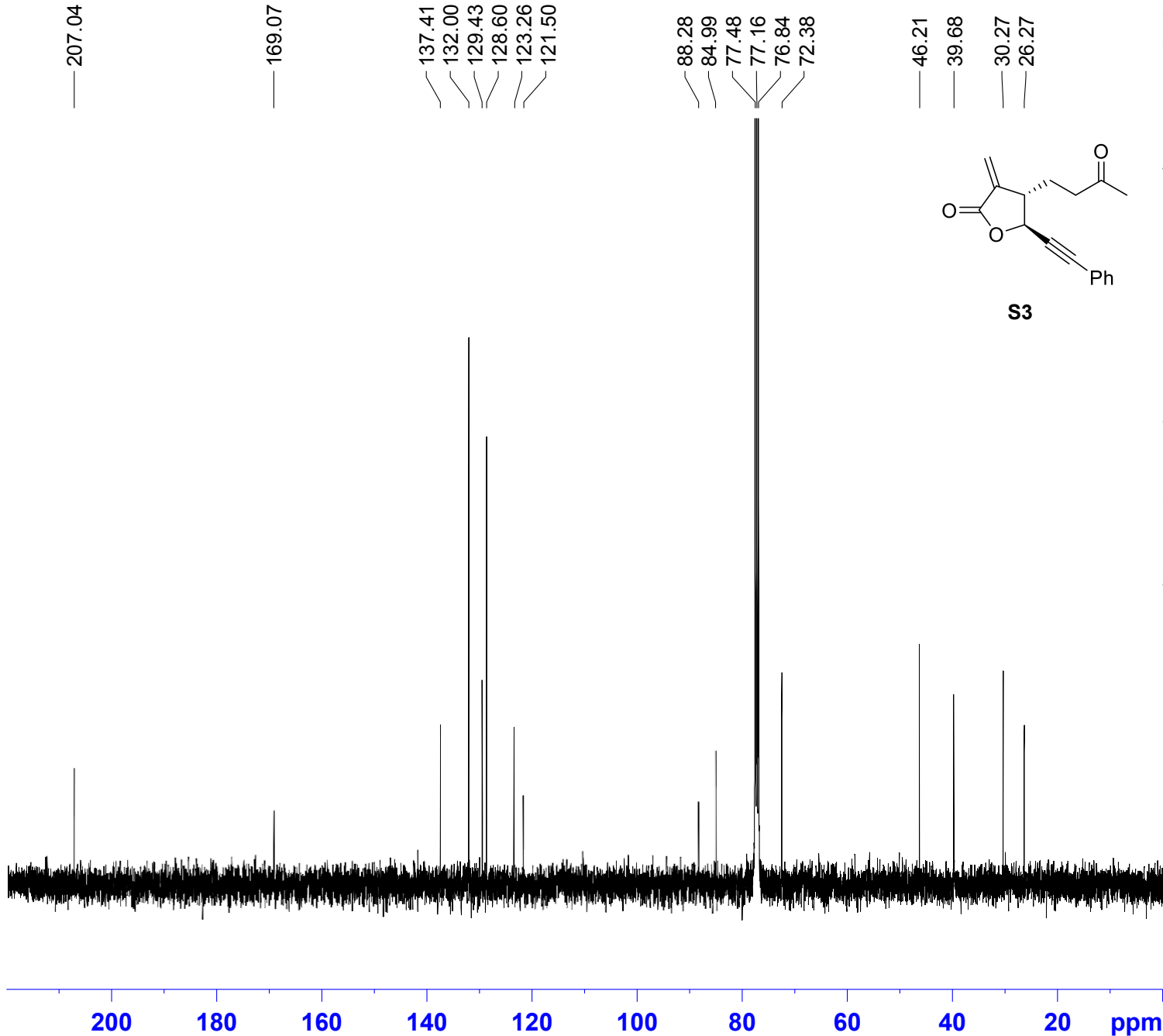
88.28
84.99
77.48
77.16
76.84
72.38

46.21
39.68
30.27
26.27

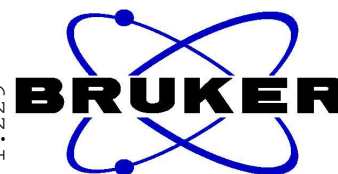


NAME SW07-033-A
EXPNO 11
PROCNO 1
Date_ 20150708
Time 4.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 95.5 K
D1 2.00000000 sec
D11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

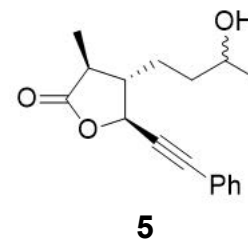


SW06-003-1H CDC13 600MHz



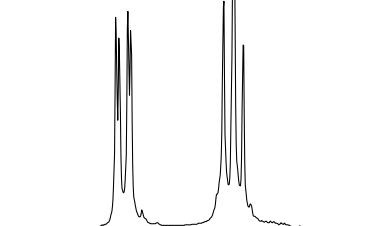
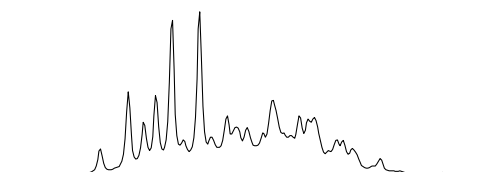
7.454
7.451
7.449
7.444
7.441
7.438
7.435
7.363
7.354
7.352
7.349
7.343
7.333
7.331
7.319
7.260
4.832
4.817
3.874
3.864
2.351
2.339
2.333
2.321
1.775
1.771
1.764
1.761
1.697
1.685
1.675
1.671
1.668
1.665
1.659
1.653
1.651
1.647
1.640
1.637
1.631
1.628
1.623
1.617
1.610
1.350
1.347
1.339
1.336
1.247
1.237
1.229

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 CDC13
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



2.362
2.351
2.344
2.339
2.333
2.328
2.321
2.317
2.310
2.306
2.302
2.295
2.287
2.284
2.280
2.277
2.274
2.265
2.262
2.259

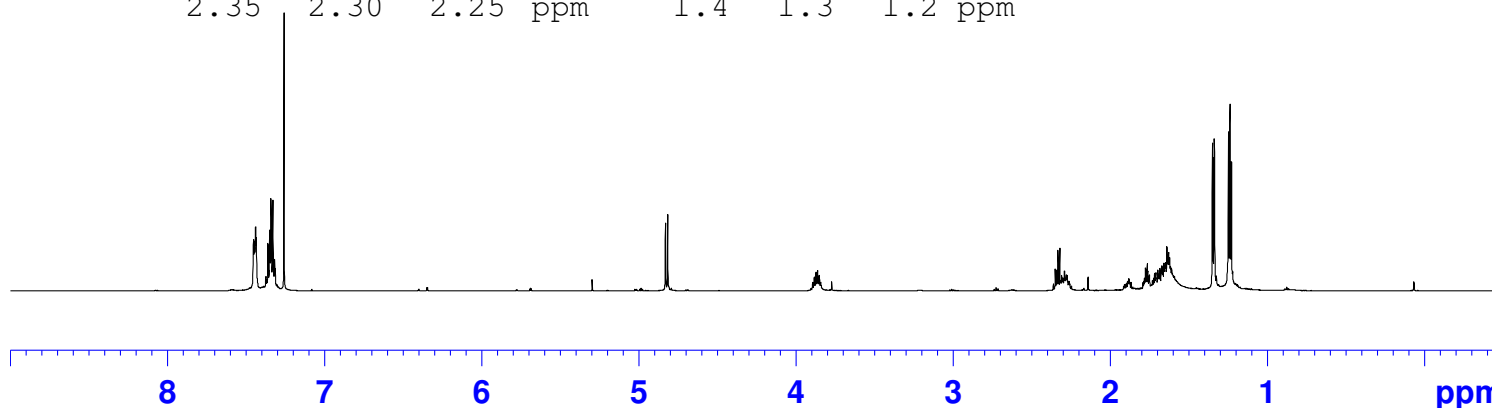
1.350
1.347
1.339
1.336
1.247
1.237
1.229



2.35 2.30 2.25 ppm

1.4 1.3 1.2 ppm

==== 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



2.22
3.28

0.90

1.05

2.00

0.64

5.78

3.00

3.92

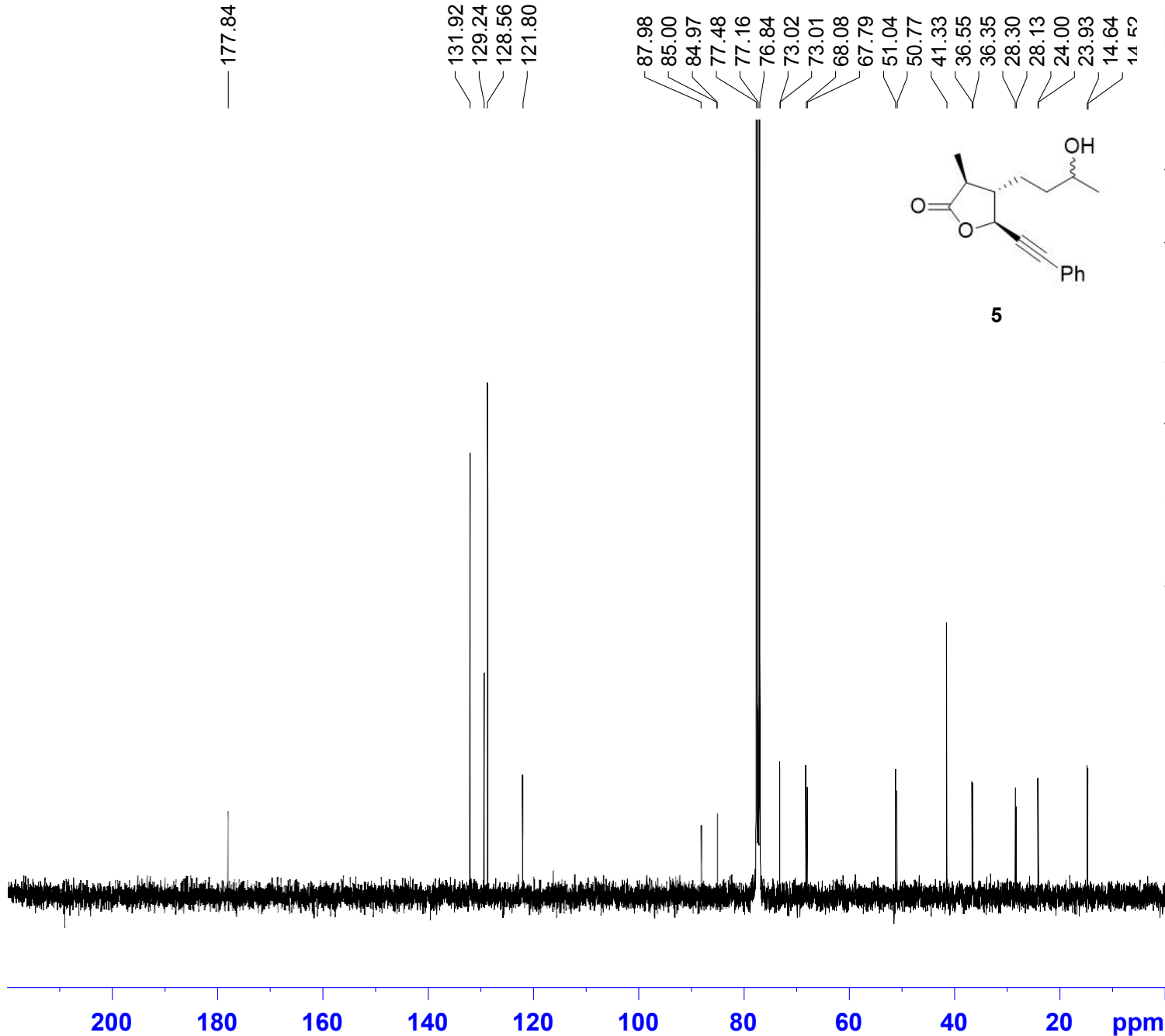
8 7 6 5 4 3 2 1 ppm

SW06-117-B 13C 400



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

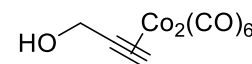


SW07-164-SM



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

1.811
1.791
1.770
1.529

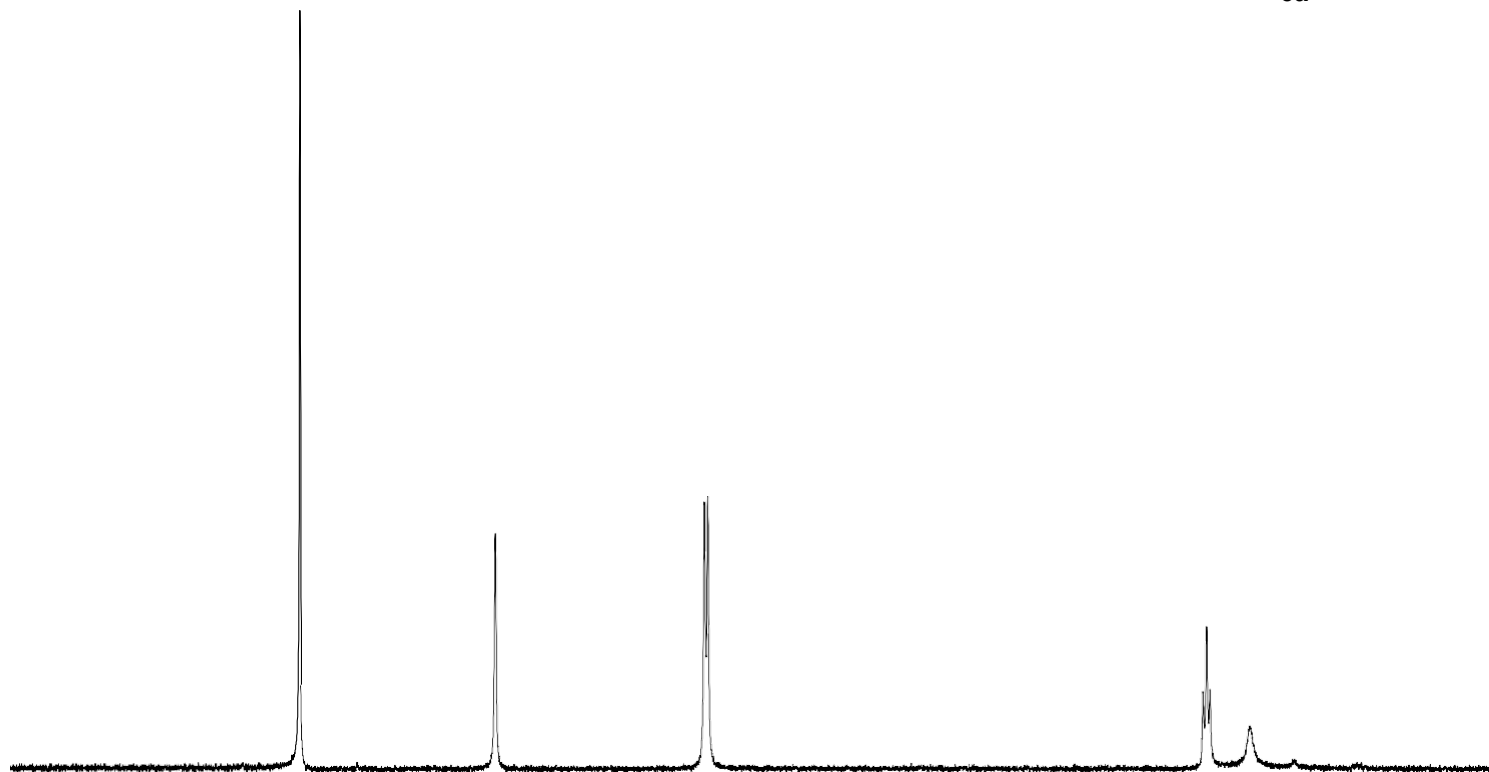


6a

7.260

6.077

4.817
4.797



8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

1.00

2.17

1.15

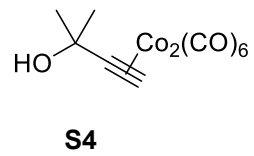
===== CHANNEL f1 =====
SFO1 300.2318540 MHz
NUC1 1H
P1 12.71 usec
SI 32768
SF 300.2300091 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

SW07-115-B 1H 300

7.260

6.034

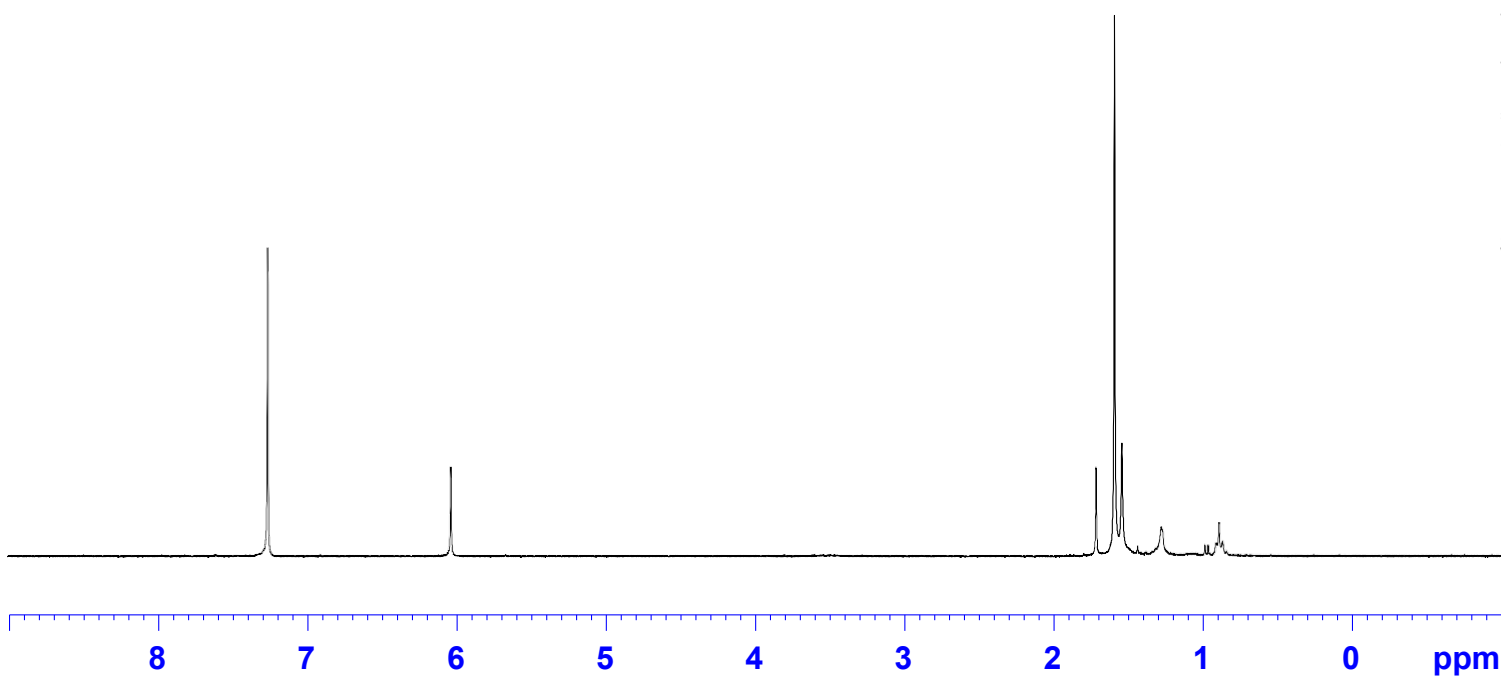
1.714
1.587
1.537
1.271
0.884



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	CDC13
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	

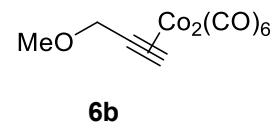


1.00
1.15
6.66
2.57
1.46
1.16

SW07-052-cr 1H 300

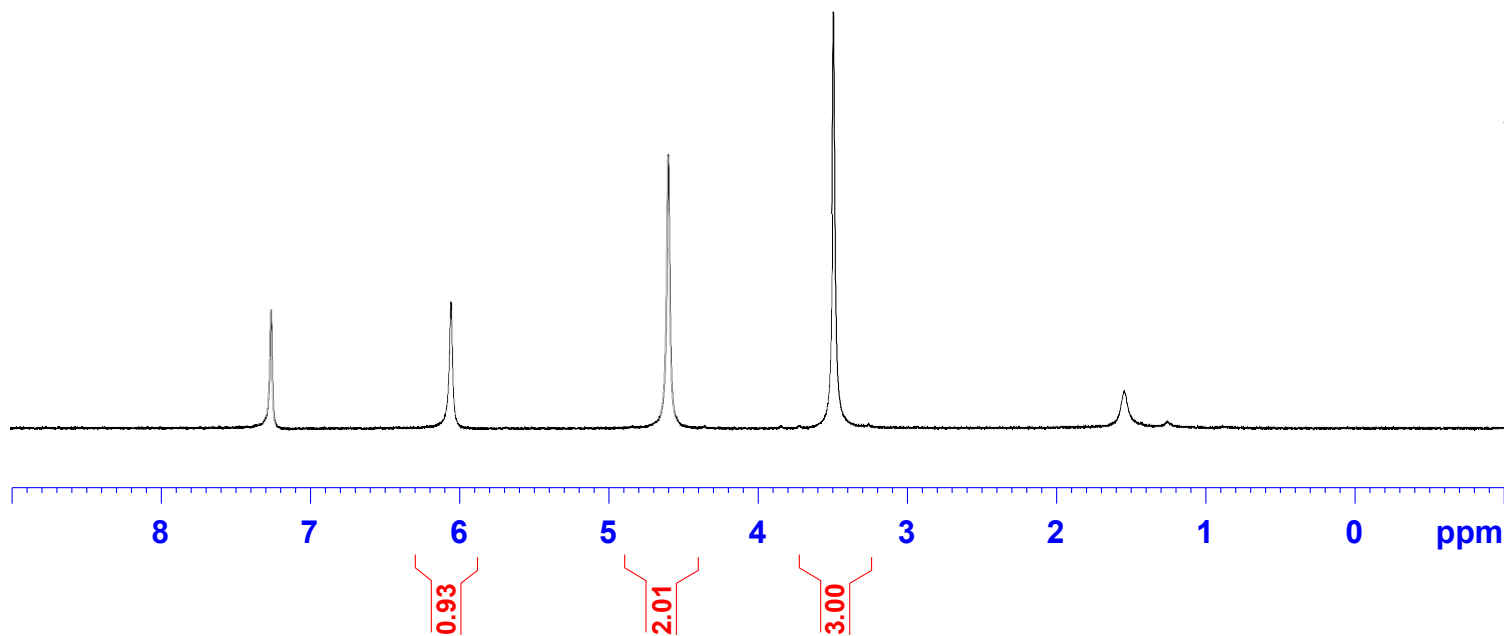


NAME SW07-052-cr
EXPNO 10
PROCNO 1
Date_ 20150728
Time 15.47
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 322
DW 80.800 usec
DE 6.50 usec
TE -930.4 K
D1 1.00000000 sec
TD0 1

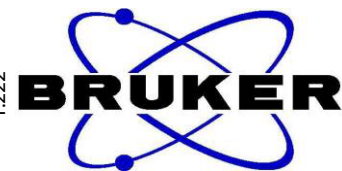


7.259
6.055
4.601
3.491
1.540

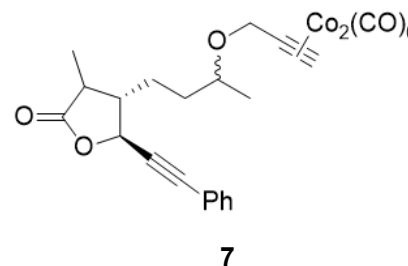
==== CHANNEL f1 =====
SFO1 300.2318540 MHz
NUC1 1H
P1 12.71 usec
SI 32768
SF 300.2300091 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



SW06-199-A 1H 500

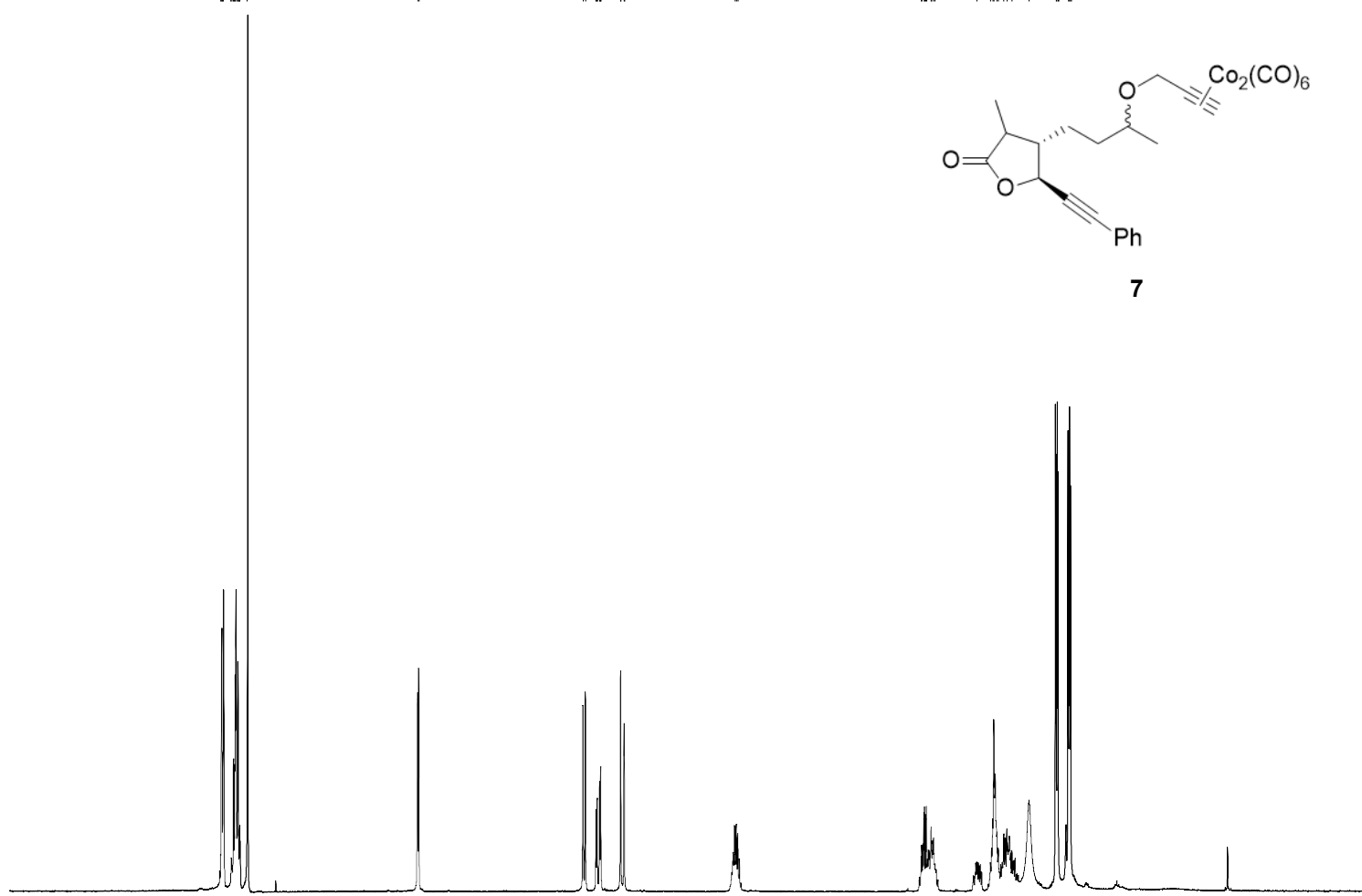


7.455
7.452
7.443
7.439
7.435
7.381
7.378
7.366
7.360
7.355
7.352
7.347
7.333
7.331
7.323
7.320
7.318
7.313
7.260
6.011
6.002
4.800
4.782
4.704
4.703
4.696
4.694
4.678
4.677
4.670
4.668
4.524
4.522
4.498
4.496
3.688
3.675
3.665
2.323
2.319
2.302
2.297
2.288
2.284
2.249
2.246
2.232
2.220
1.915
1.815
1.798
1.790
1.766
1.757
1.717
1.694
1.657
1.532
1.333
1.327
1.319
1.314
1.241
1.234
1.228
1.222



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



1.97
3.00

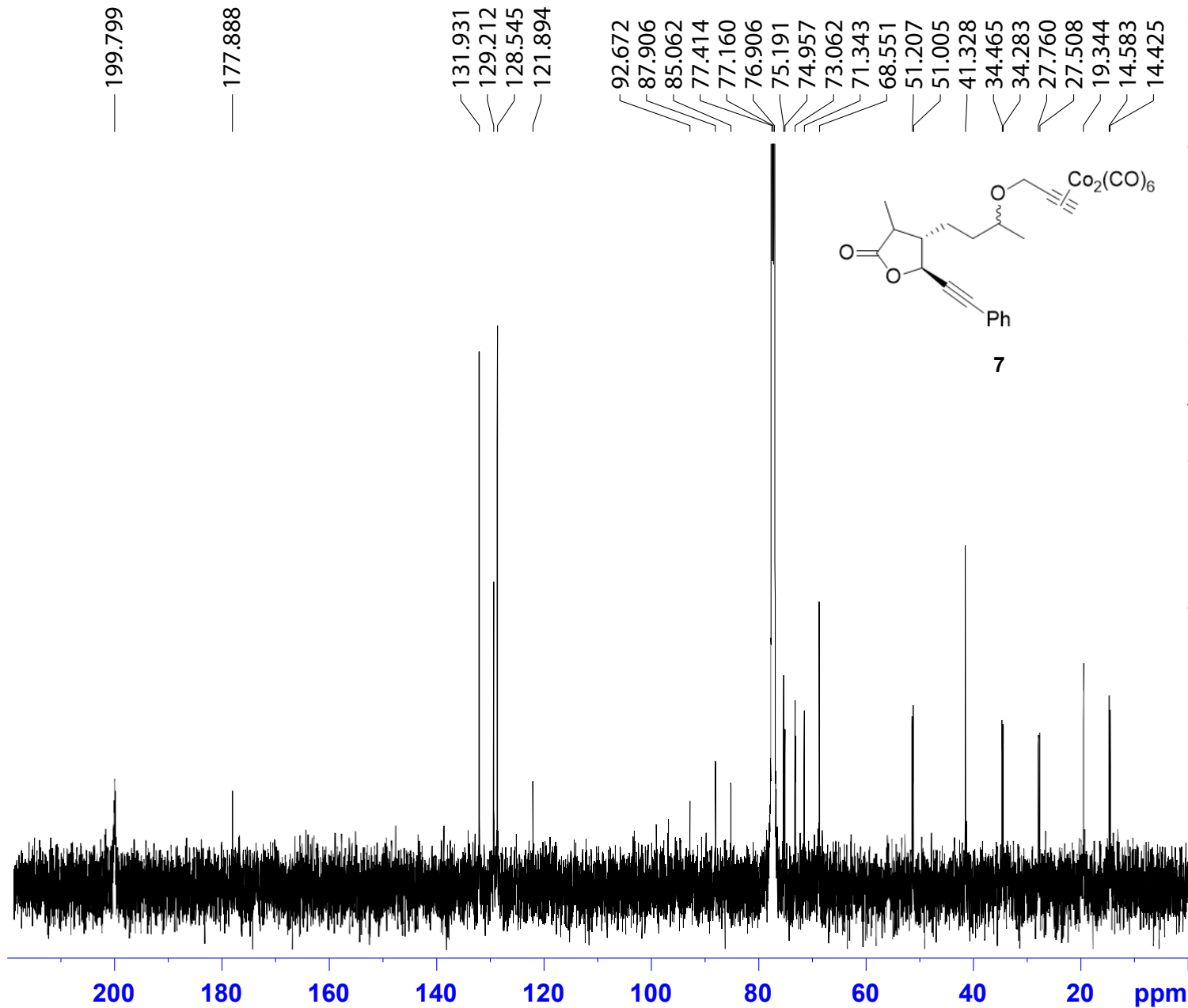
0.95

0.97
1.00
1.00

1.02

2.00
0.53
3.69
1.87
2.97
3.05

SW06-199-A 13C 500



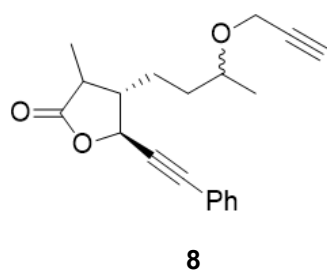
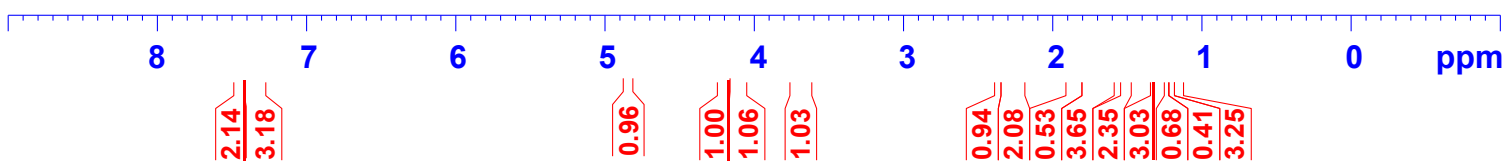
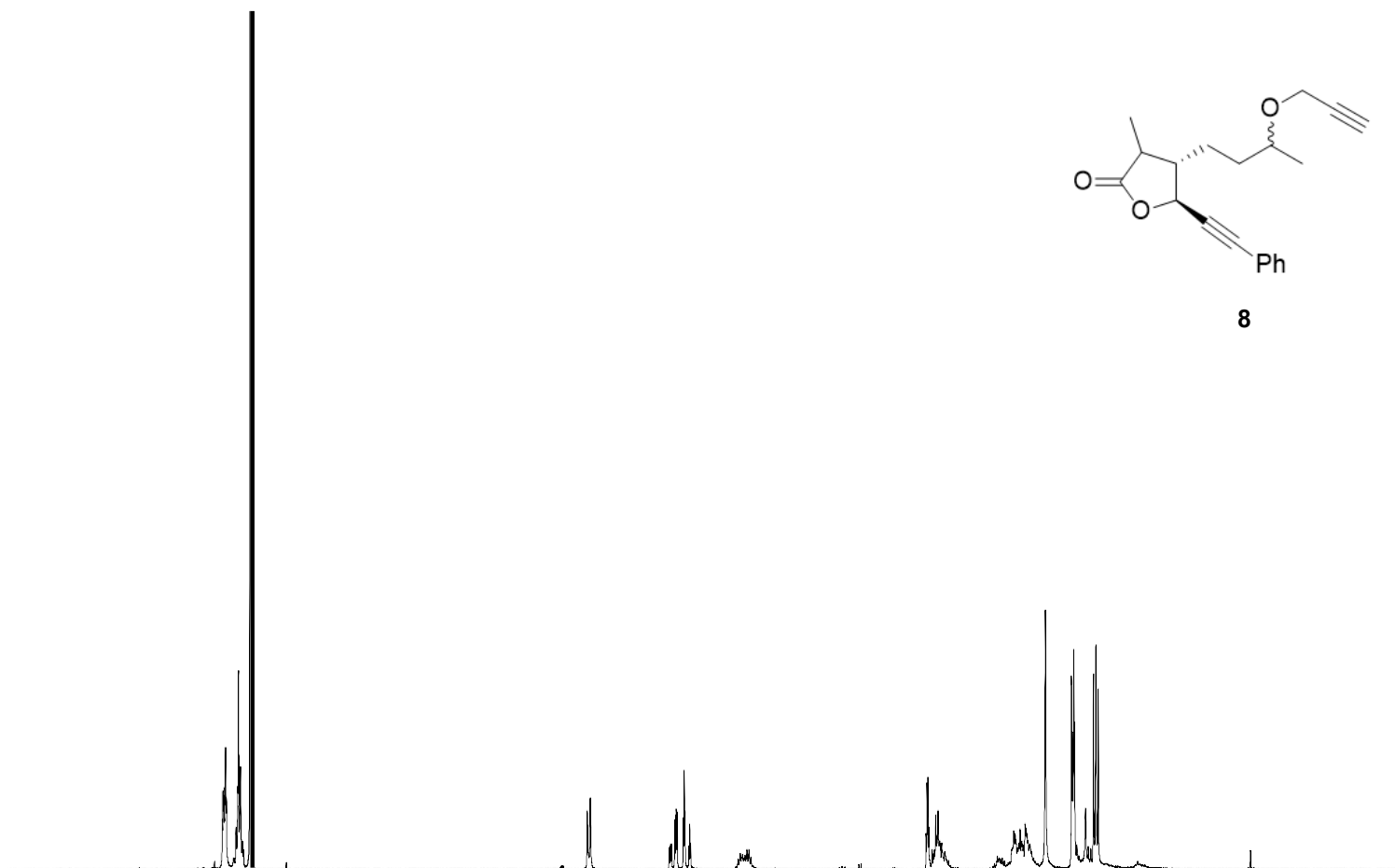
NAME SW06-199-A
EXPNO 20
PROCNO 1
Date_ 20150520
Time 0.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== 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



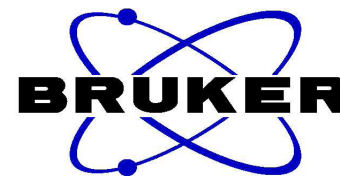
7.458
7.454
7.450
7.445
7.440
7.434
7.364
7.353
7.348
7.337
7.329
7.314
7.260
4.835
4.832
4.813
4.246
4.240
4.234
4.228
4.206
4.200
4.195
4.189
4.145
4.138
4.132
4.105
4.099
4.093
3.736
3.719
3.702
3.687
3.673
2.400
2.394
2.387
2.381
2.358
2.341
2.331
2.314
2.302
2.288
2.283
2.266
1.888
1.771
1.761
1.756
1.725
1.689
1.682
1.674
1.668
1.543
1.356
1.339
1.333
1.254
1.198
1.183
1.179
1.164



NAME SW06-123-A
 EXPNO 10
 PROCNO 1
 Date_ 20150 305
 Time 15.09
 INSTR UM spect
 PROBHD 5 mm PABBO BB-
 PULPR OG zg30
 TD 65536
 SOLVE NT CDC13
 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 1.0000000 sec

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

SW06-123-A 13C 400B

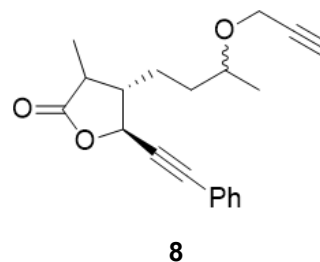


177.92

131.93
131.91
129.22
128.55
121.83

87.93
85.01
80.41
77.48
77.16
76.84
74.10
73.74
73.08
55.78
55.69
51.02
50.55
41.33
34.17

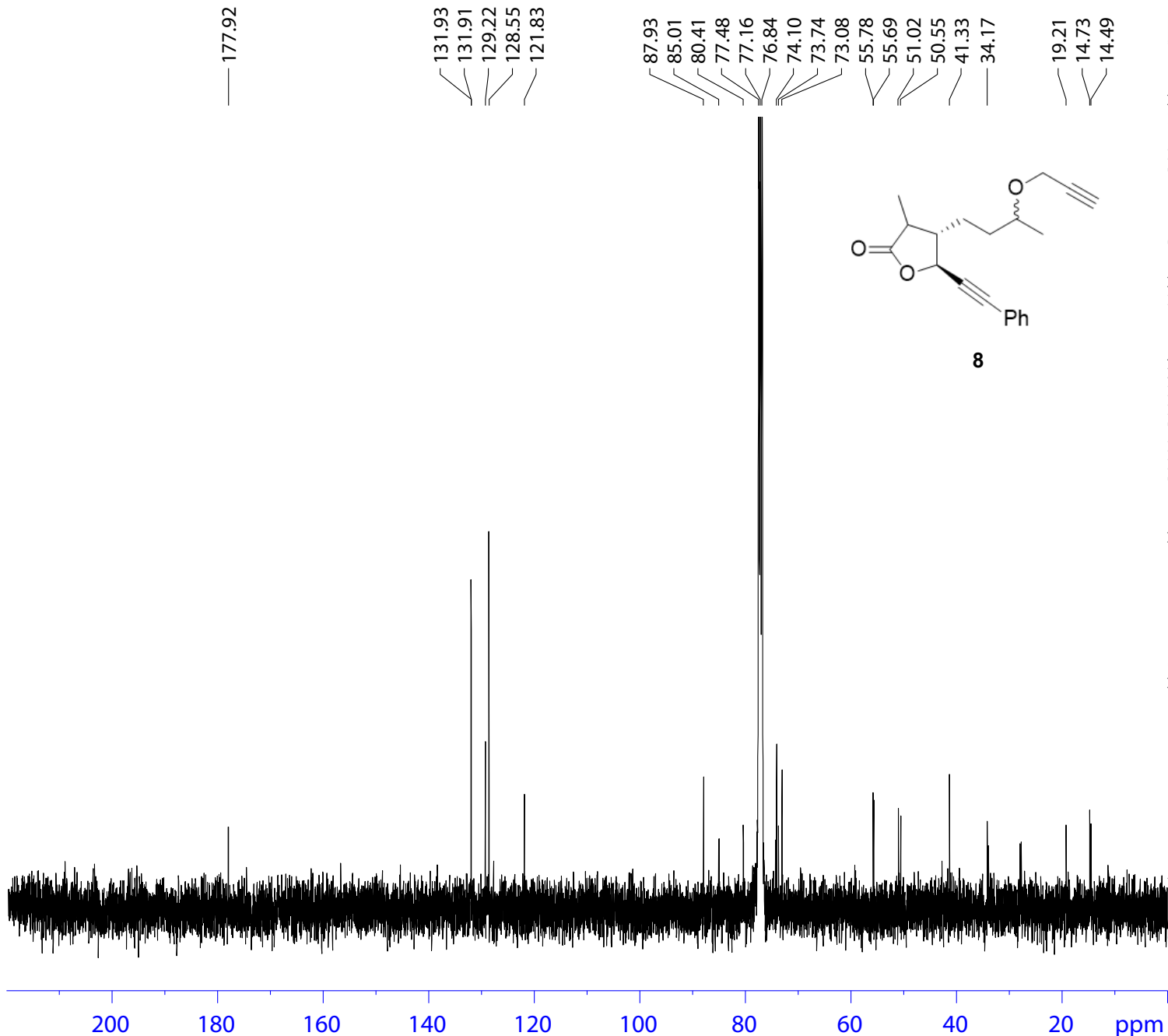
19.21
14.73
14.49

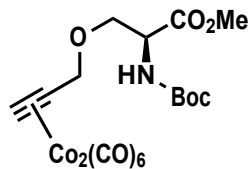


NAME SW06-123-A
EXPNO 2
PROCNO 1
Date_ 20150307
Time 14.50
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4394
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 45.2
DW 20.800 usec
DE 6.50 usec
TE 295.4 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1

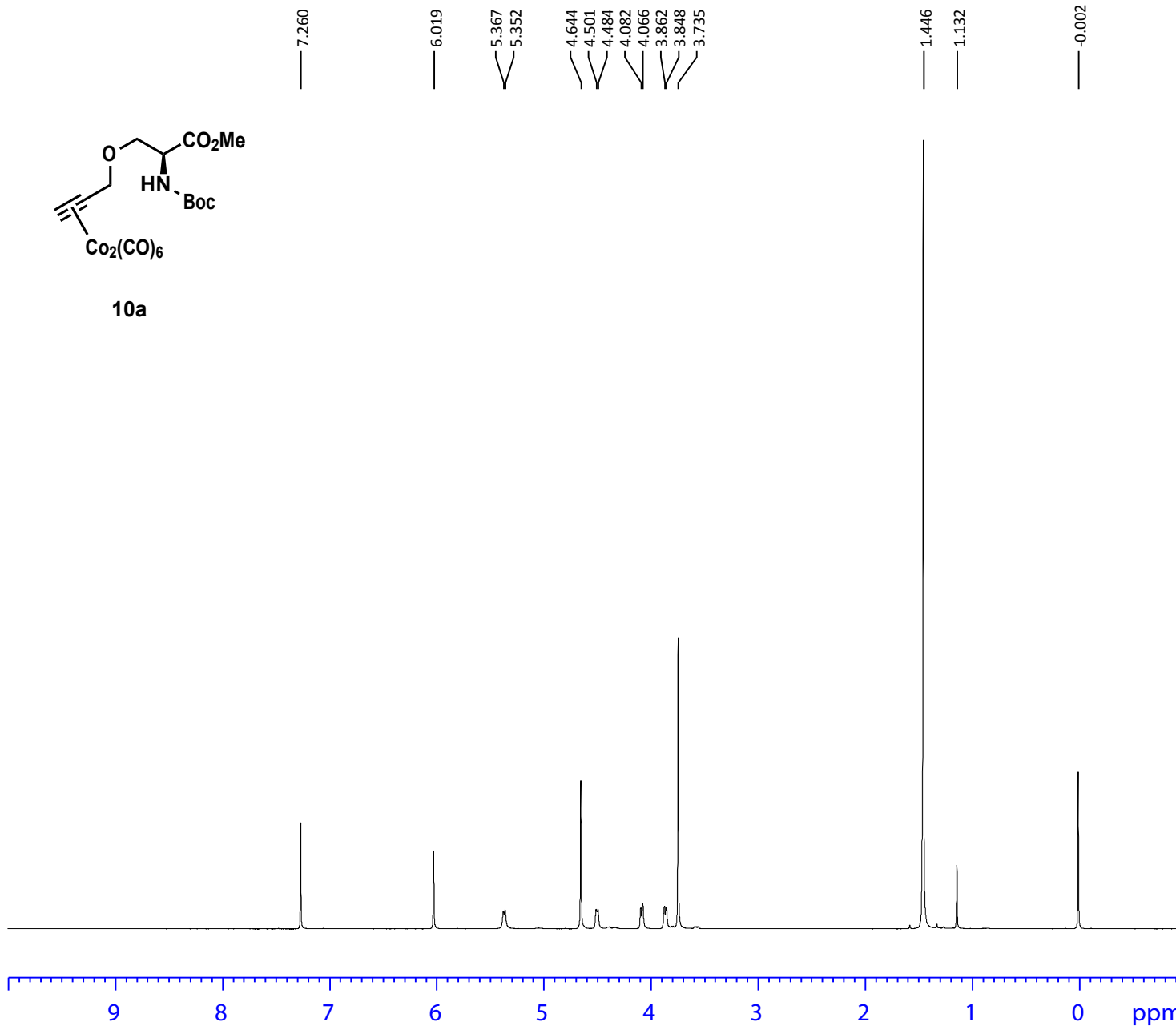
==== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -0.44 dB
PL1W 39.19395828 W
SFO1 100.6479773 MHz

==== 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





10a



7.260
6.019
5.367
5.352
4.644
4.501
4.484
4.082
4.066
3.862
3.848
3.735
1.446
1.132
-0.002



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 1.0000000 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

1.05
1.00
2.16
0.98
1.08
1.11
3.50
10.59



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 =====

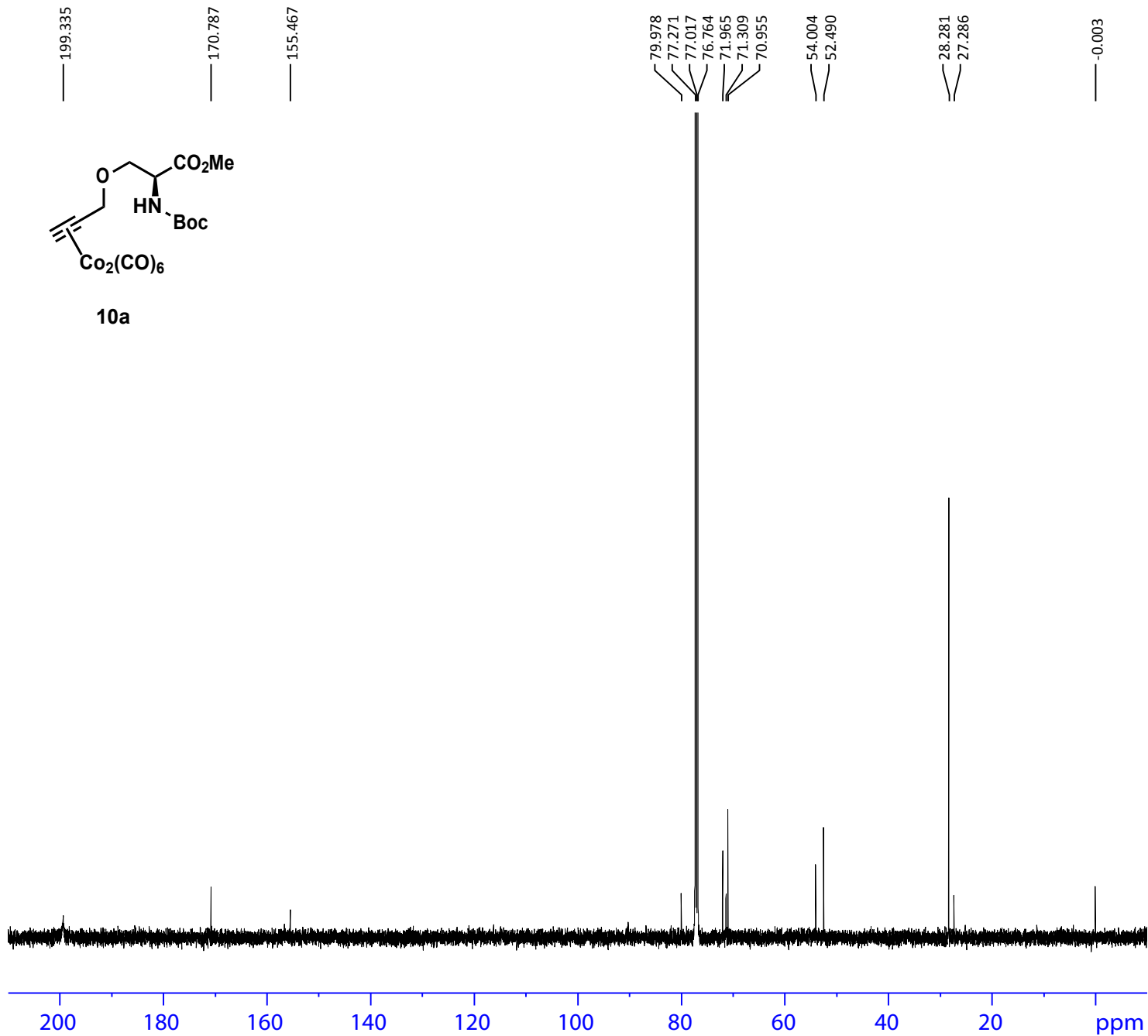
SFO1 125.7703643 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

===== 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

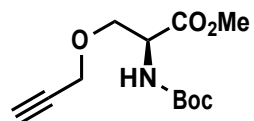
Date_ 20150423
Time 13.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 219998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.045455 Hz
AQ 10.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.0000000 sec
TD0 1

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

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

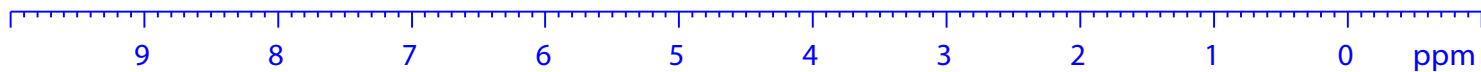
F2 - Processing parameters

SI 65536
SF 500.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7.260

5.362
5.346
4.461
4.455
4.451
4.444
4.143
4.138
3.958
3.952
3.939
3.933
3.769
3.759
3.750
3.743
2.441
2.436
2.431
1.444



1.00
1.03
2.24
1.05
4.69
1.14
10.61



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 =====

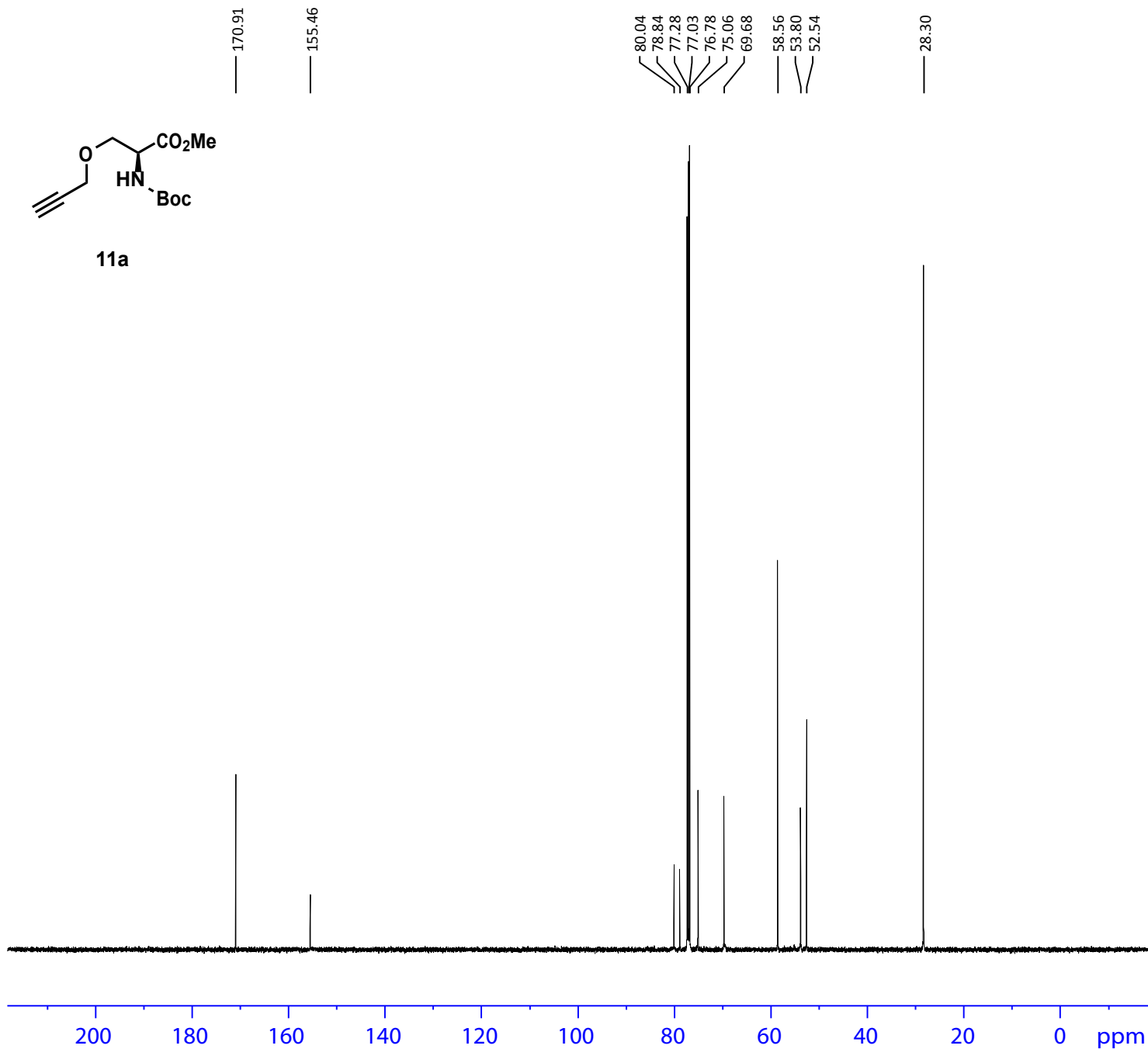
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

===== 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

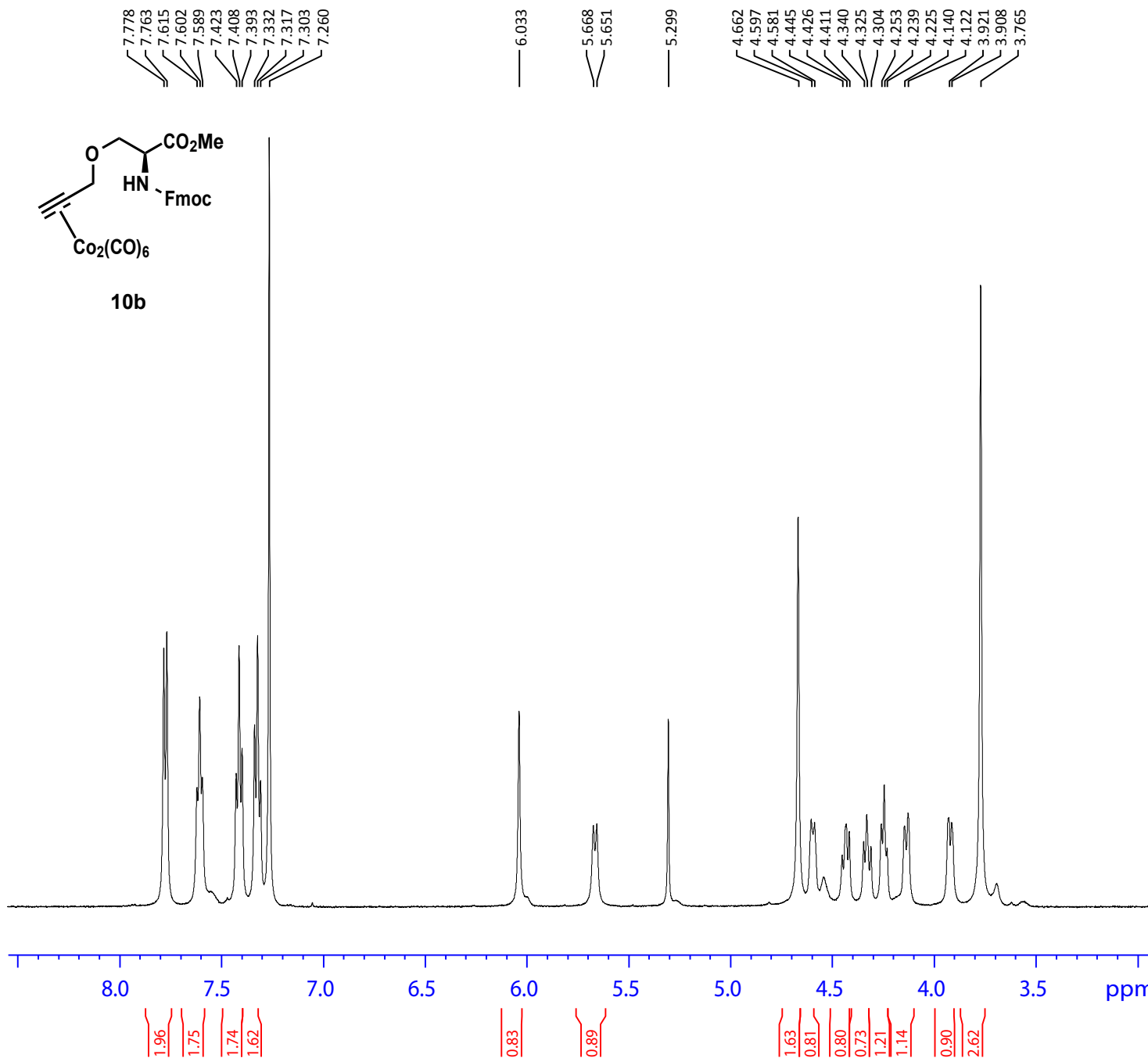
Date_ 20150511
Time 21.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 186.69
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

===== 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 =====

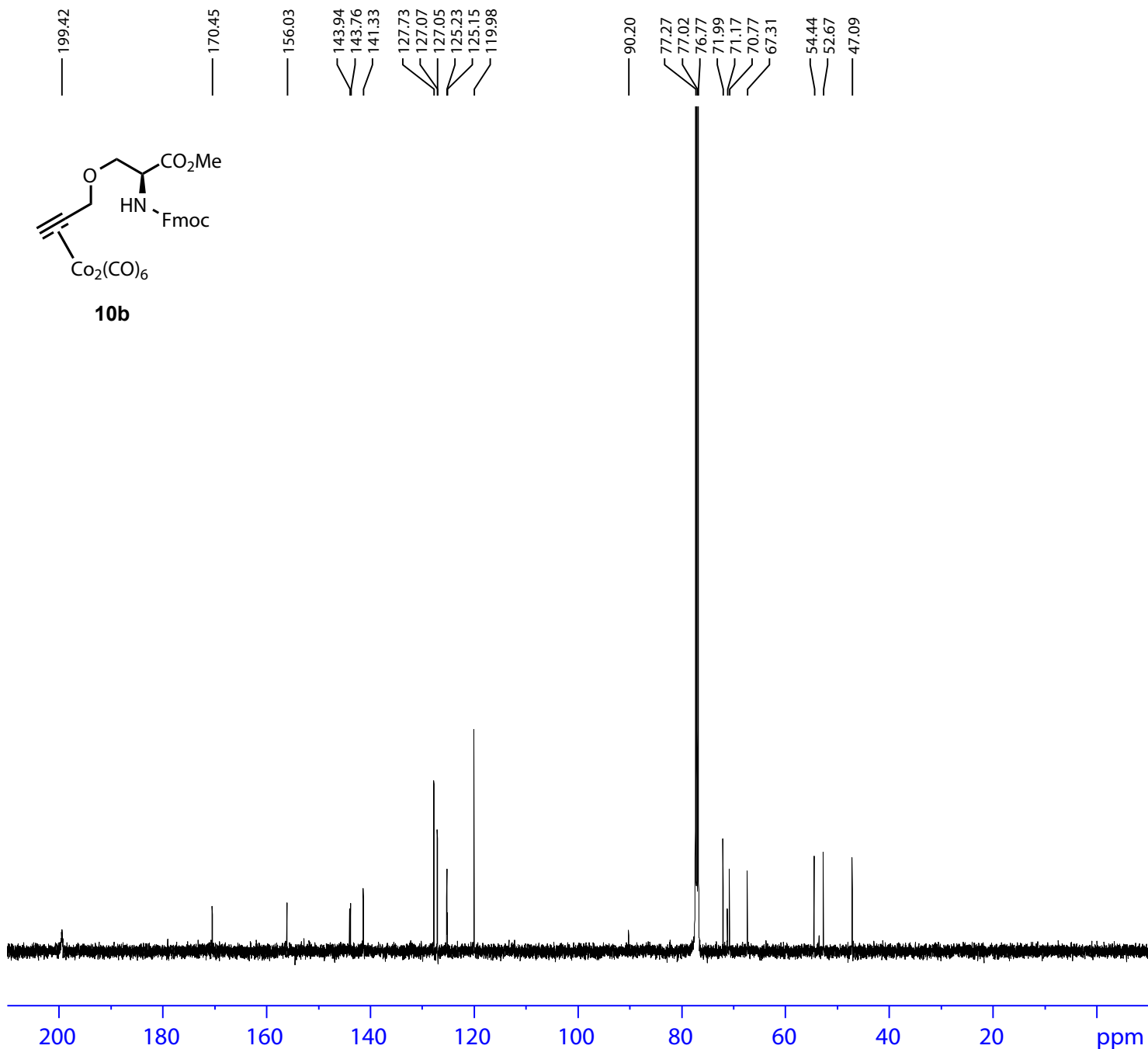
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

===== 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

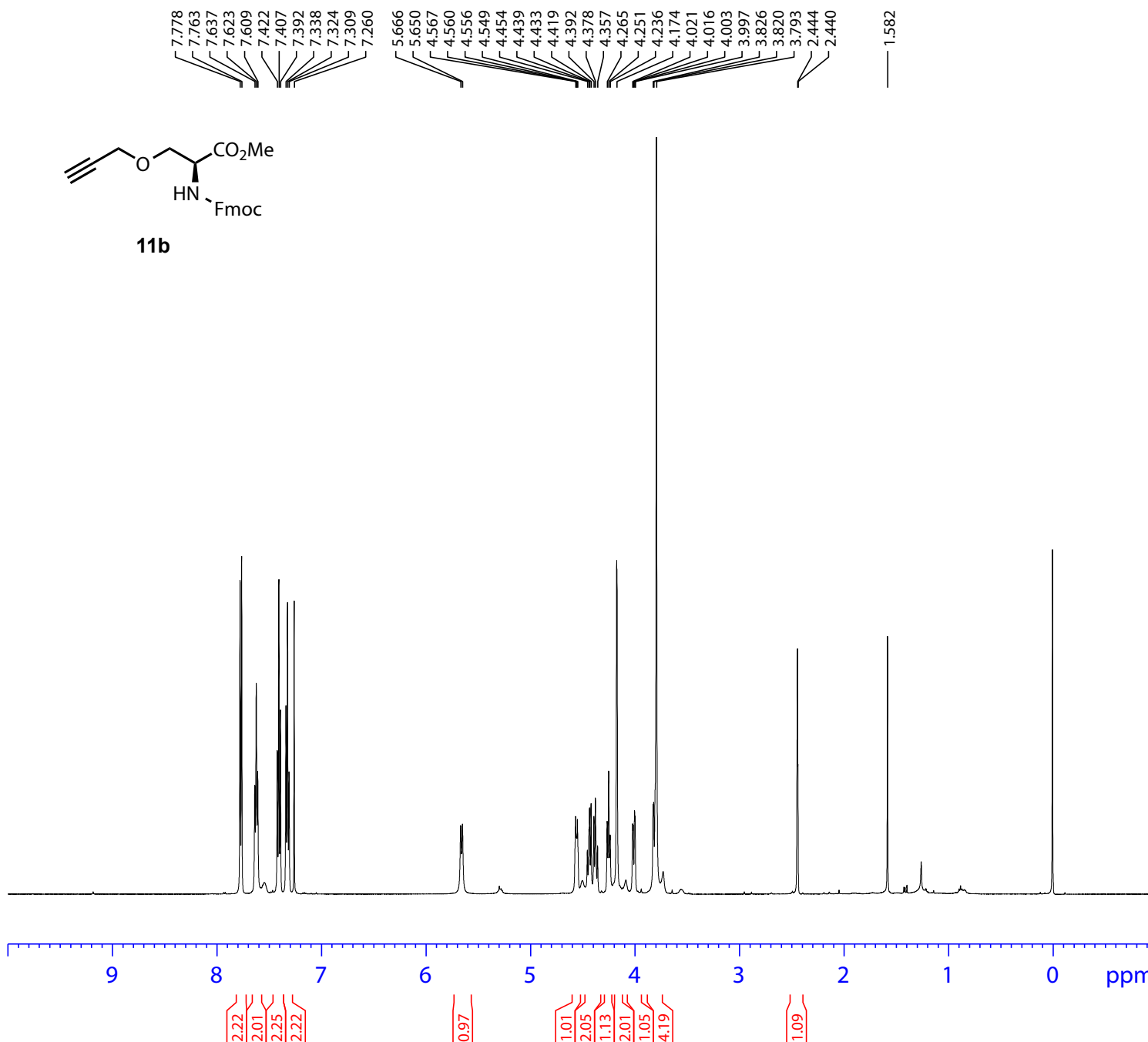
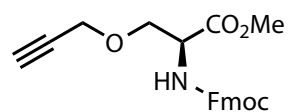
Date_ 20150727
Time 19.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 199998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.050000 Hz
AQ 9.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

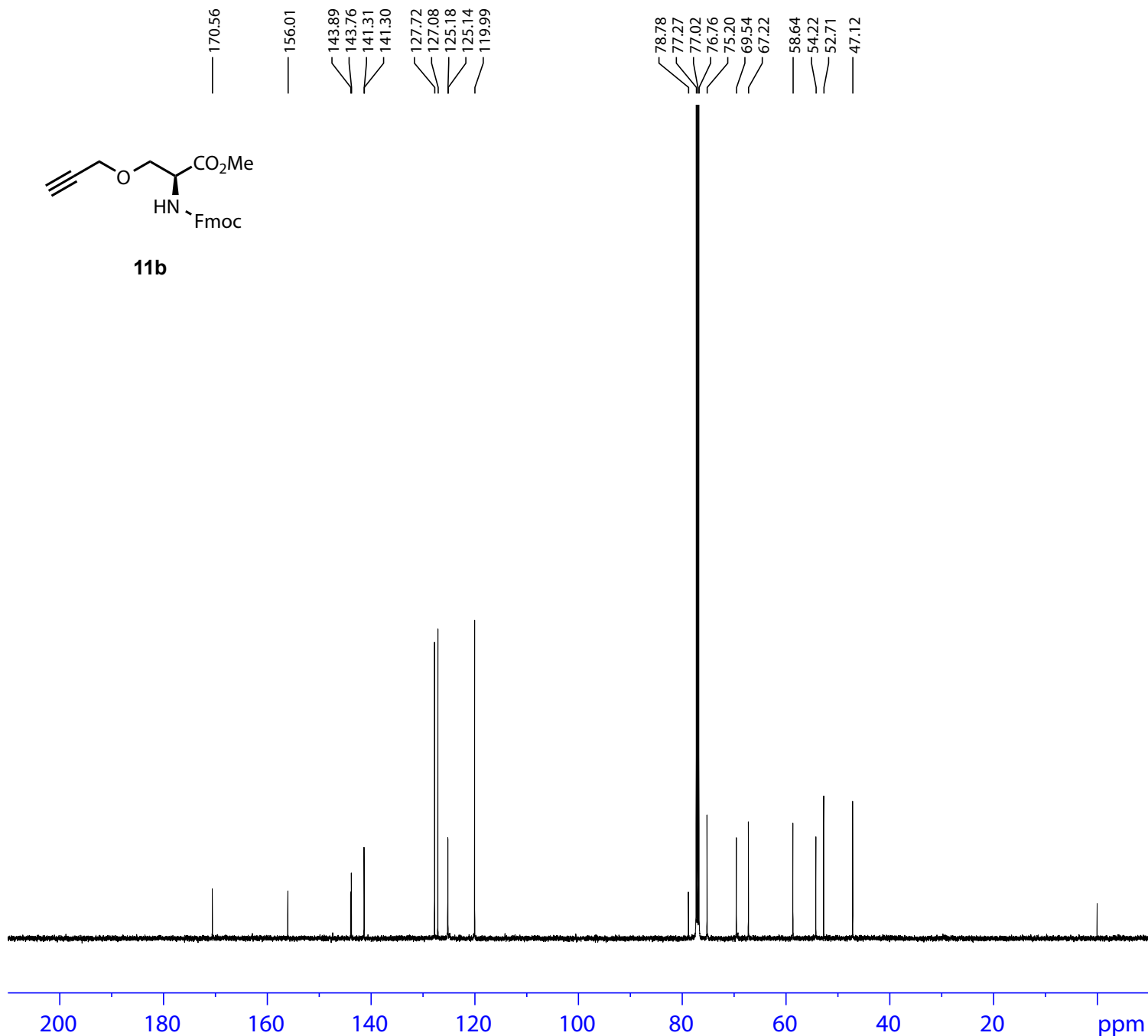
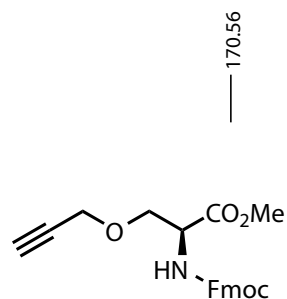
===== CHANNEL f1 =====

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

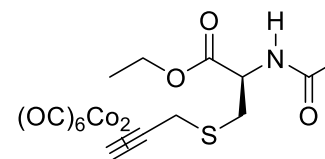
SI 32768
SF 125.7577906 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SW06-102-C 1H 300

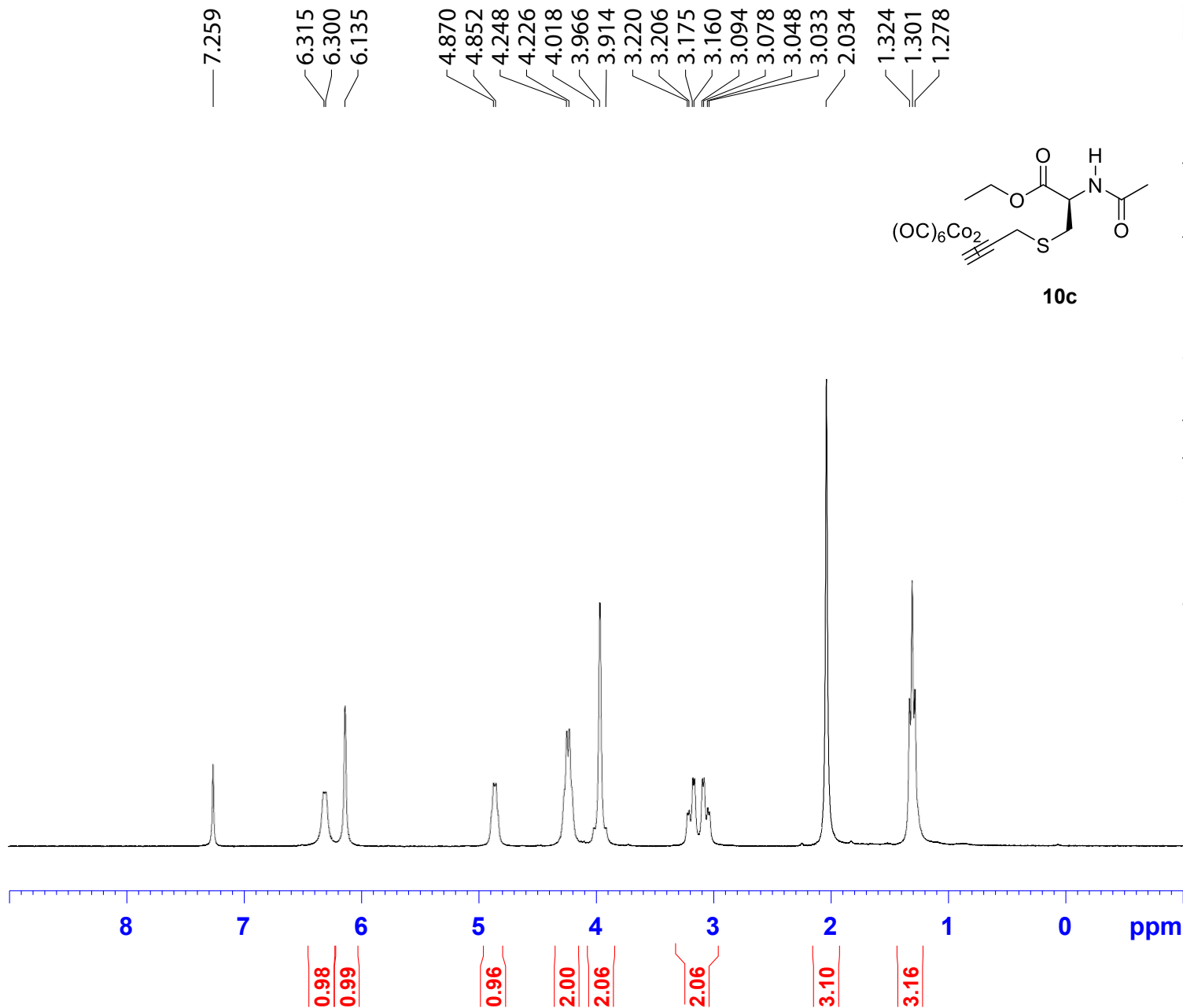


NAME SW06-102-C
EXPNO 10
PROCNO 1
Date_ 20150213
Time 16.20
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 128
DW 80.800 usec
DE 6.50 usec
TE -929.4 K
D1 1.00000000 sec
TD0 1

===== 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



10c

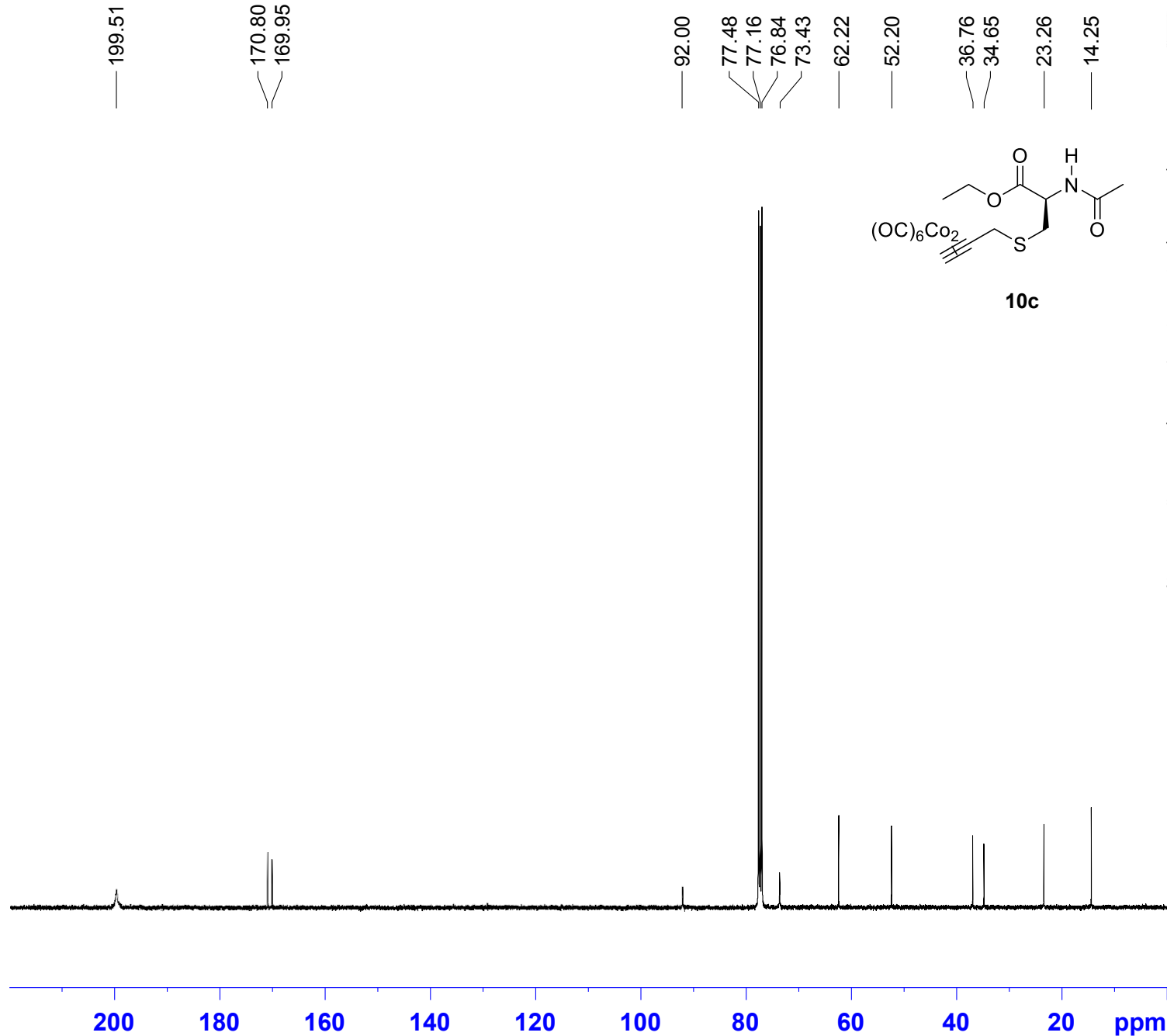
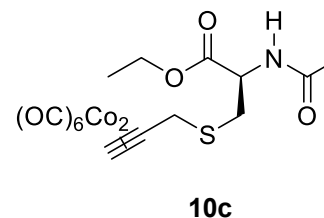


SW06-102-C 13C 400



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

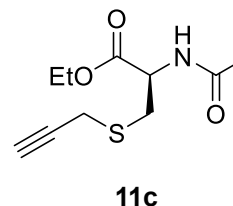
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



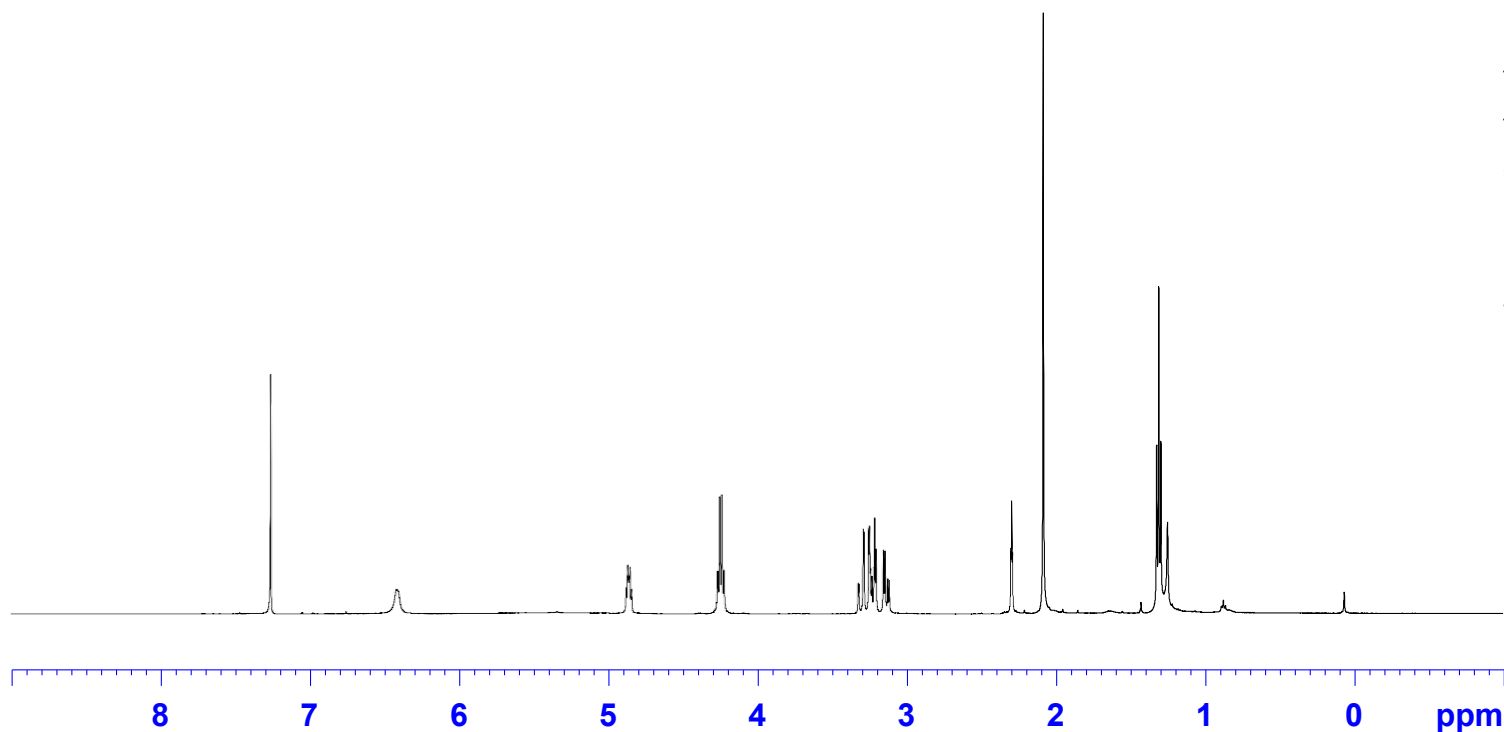
SW06-195-cr 1H 500



7.260
6.416
4.879
4.869
4.864
4.859
4.854
4.843
4.266
4.252
4.238
4.224
3.326
3.321
3.292
3.287
3.254
3.249
3.244
3.234
3.220
3.215
3.206
3.156
3.146
3.128
3.117
2.303
2.298
2.293
2.084
1.324
1.310
1.295
1.251
0.877



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



1.00
1.07
2.11
0.99
2.00
1.07
0.99
3.13
3.48

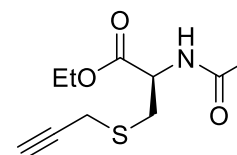
SW06-195-cr 1H 500



170.868
170.253

79.398
77.413
77.159
76.905
72.094
62.194
51.916

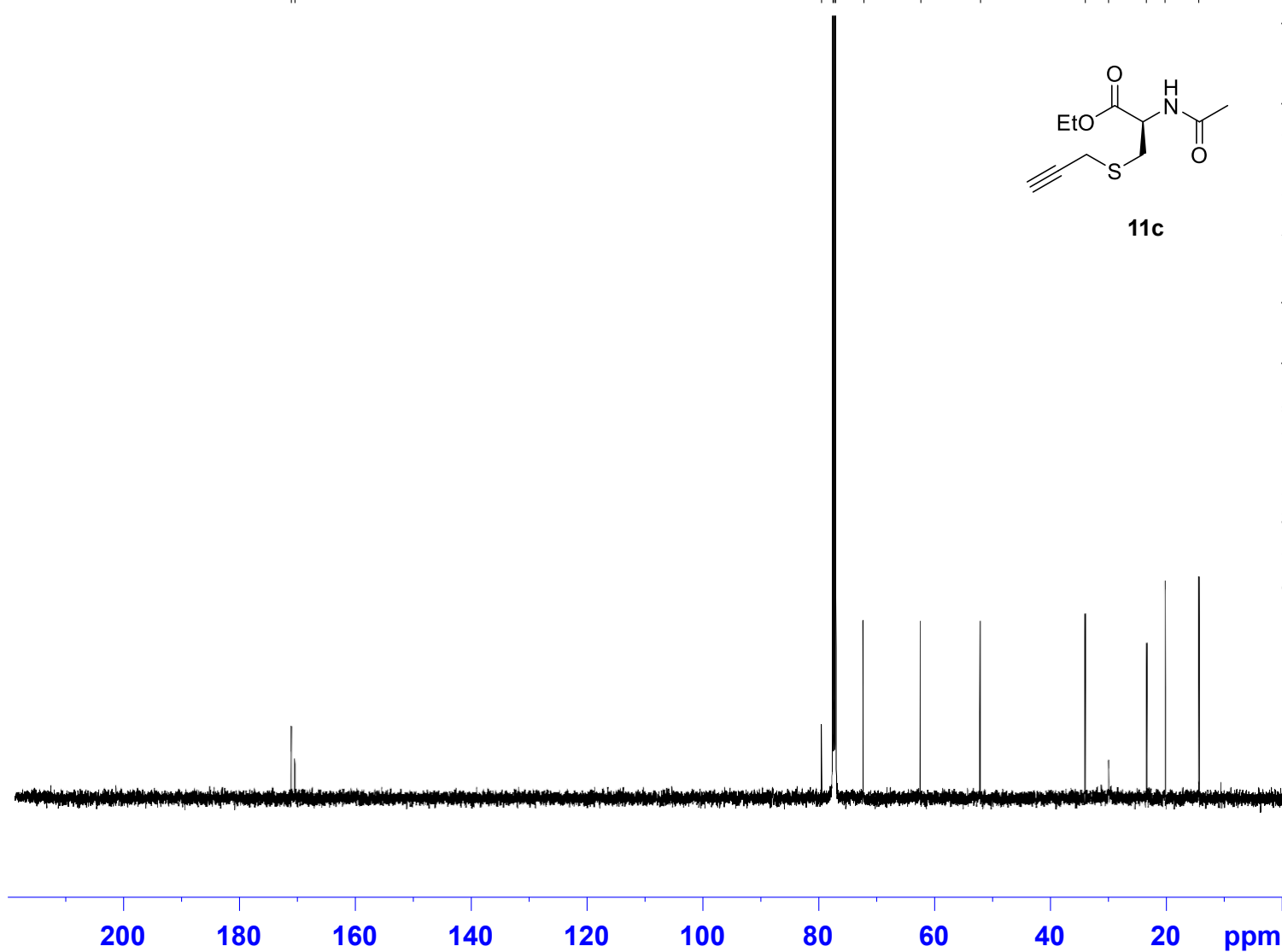
33.863
29.849
23.277
20.066
14.265



11c

NAME SW06-195-cr
EXPNO 11
PROCNO 1
Date_ 20150515
Time 0.46
INSTRUM spect
PROBHD 5 mm PABBOBB/
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

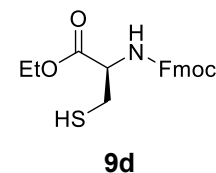
===== CHANNEL f1 =====
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653129 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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

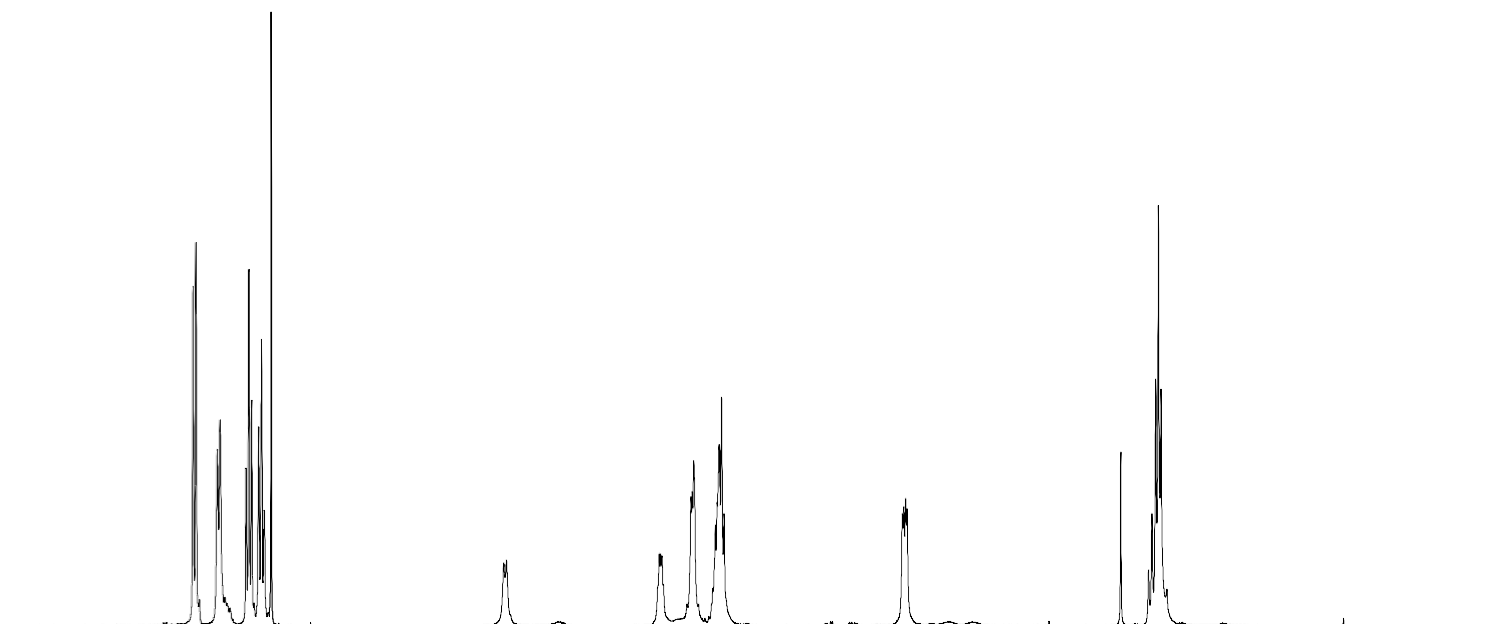


===== CHANNEL f1 =====
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7.784
7.765
7.622
7.604
7.431
7.412
7.394
7.350
7.347
7.343
7.332
7.328
7.325
7.313
7.309
7.306
7.260

5.701
5.683
4.659
4.650
4.641
4.631
4.473
4.447
4.438
4.428
4.396
4.300
4.283
4.271
4.258
4.242
4.225
3.030
3.020
3.008
2.998

1.566
1.380
1.358
1.332
1.314
1.296



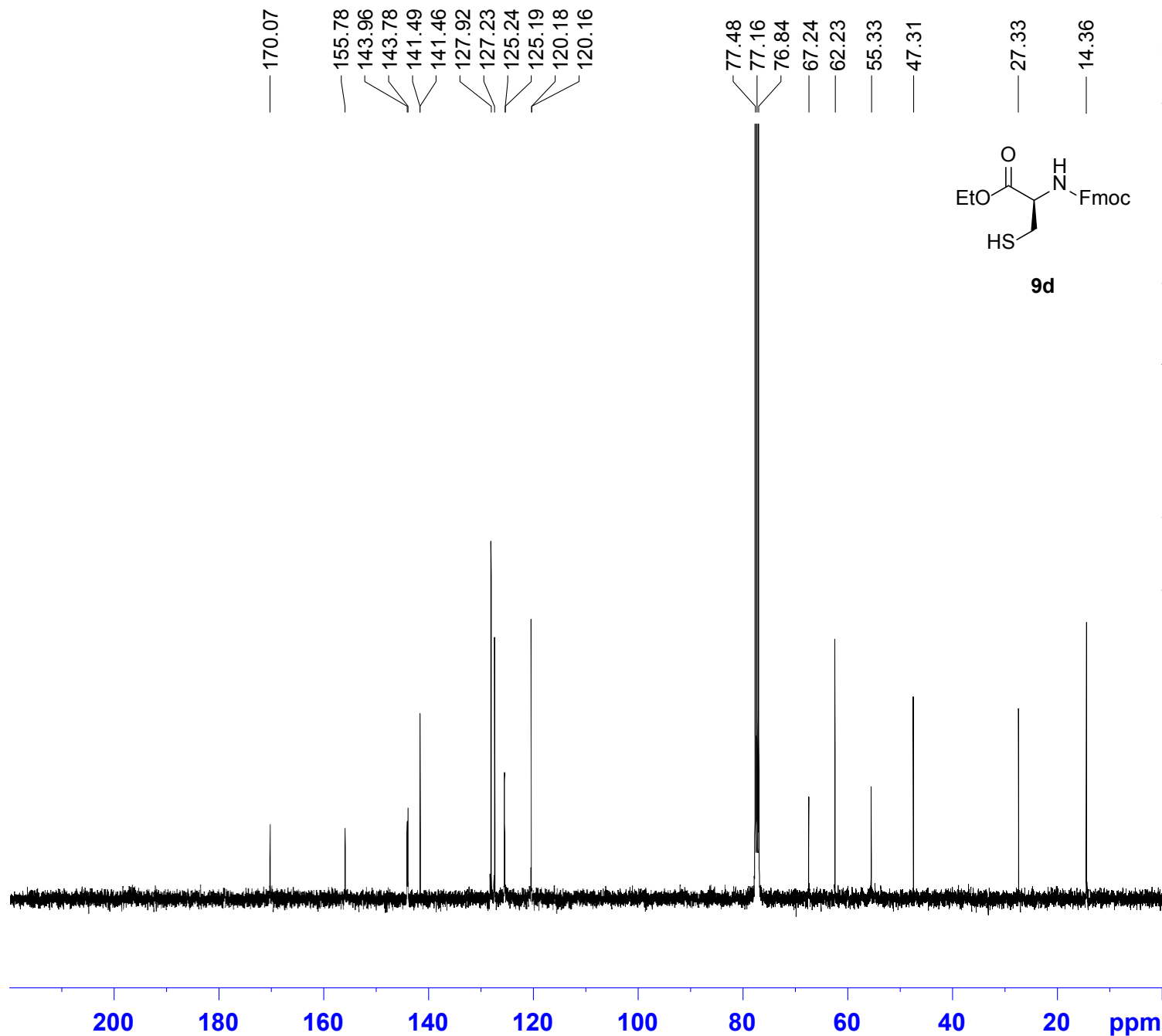
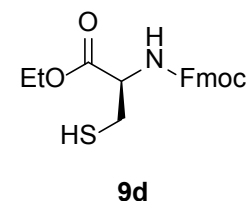
2.09
1.95
2.13
2.12
0.88
1.00
1.96
3.24
1.77
0.36
4.22

SW06-206-B 13C 400



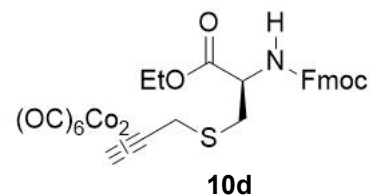
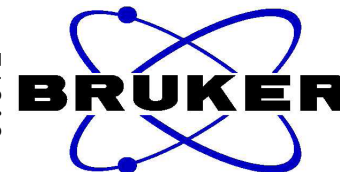
NAME SW06-206-B
EXPNO 21
PROCNO 1
Date_ 20150607
Time_ 23.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3072
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 161
DW 20.800 usec
DE 6.50 usec
TE 93.6 K
D1 2.00000000 sec
D11 0.03000000 sec

===== 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



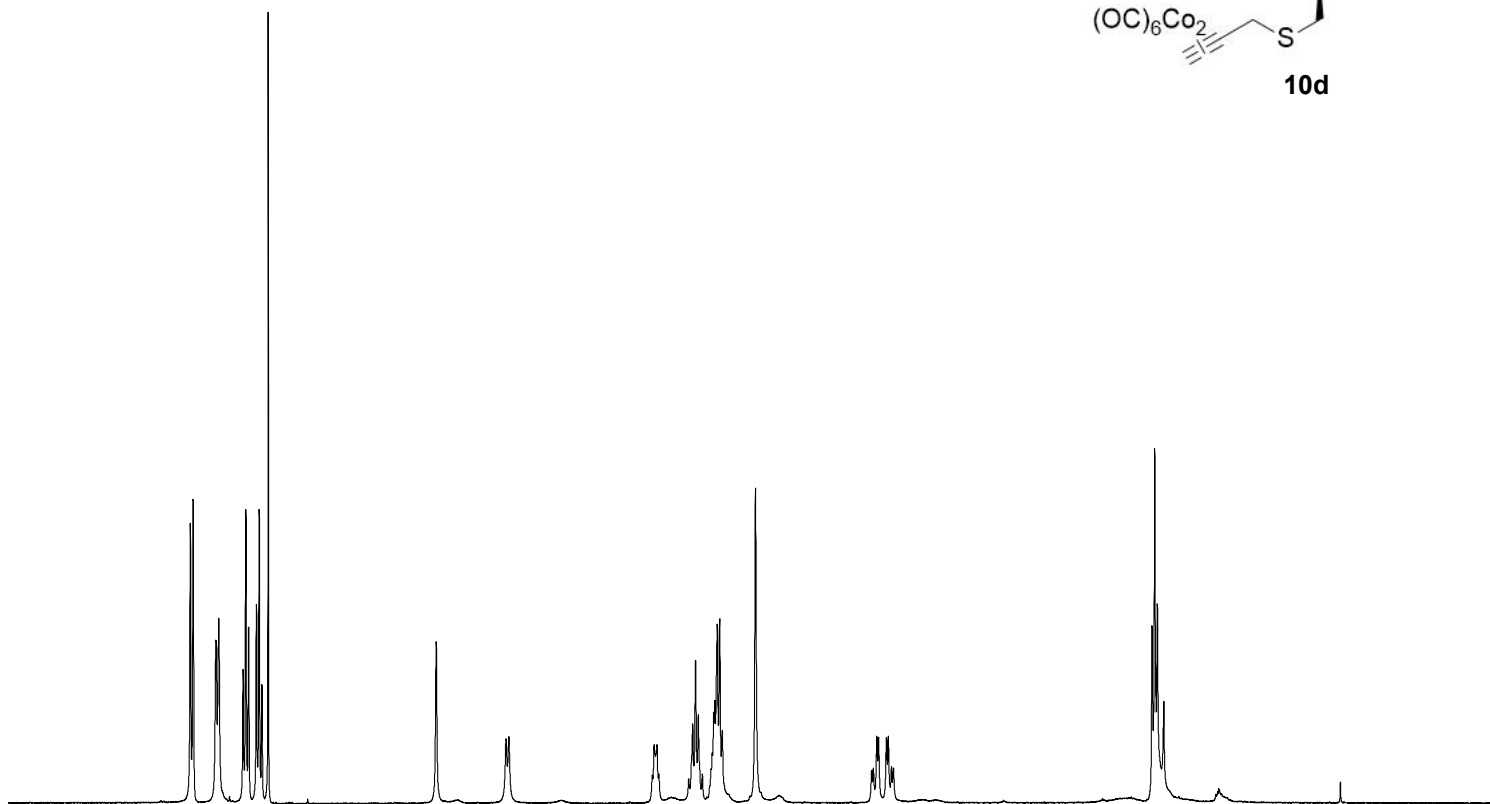
SW06-138-C 1H 400

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



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



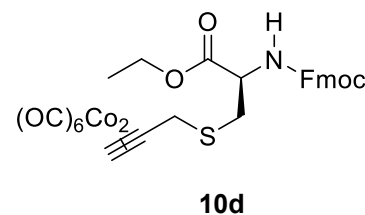
8 7 6 5 4 3 2 1 0 ppm

2.23
2.18
2.18
2.14
0.93
0.89
0.93
1.96
3.22
1.83
0.94
1.00
3.33
0.75

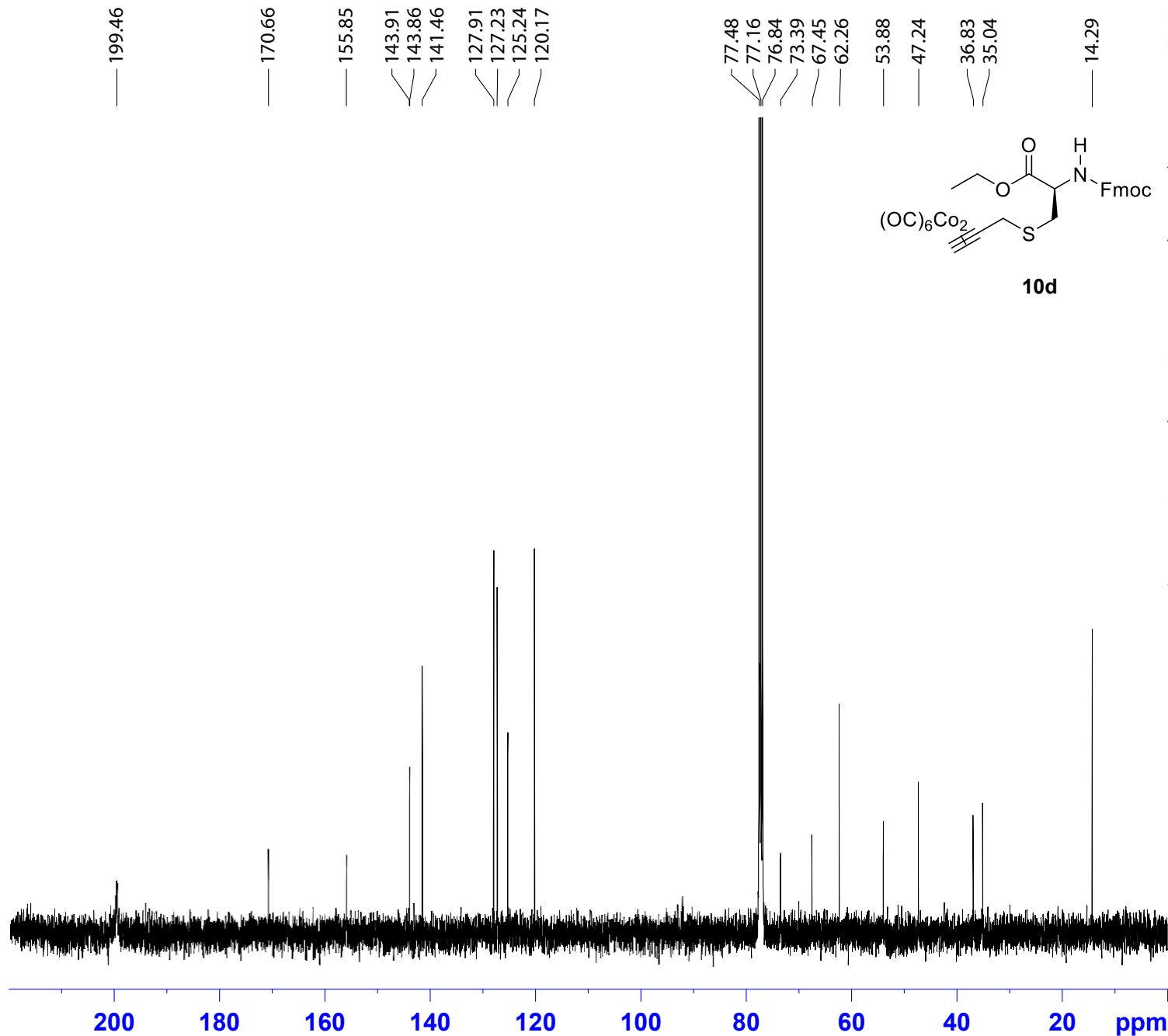
SW06-138-C 13C 400



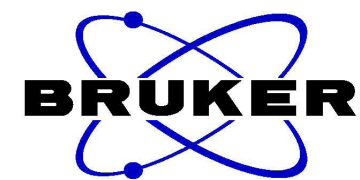
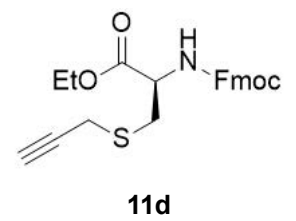
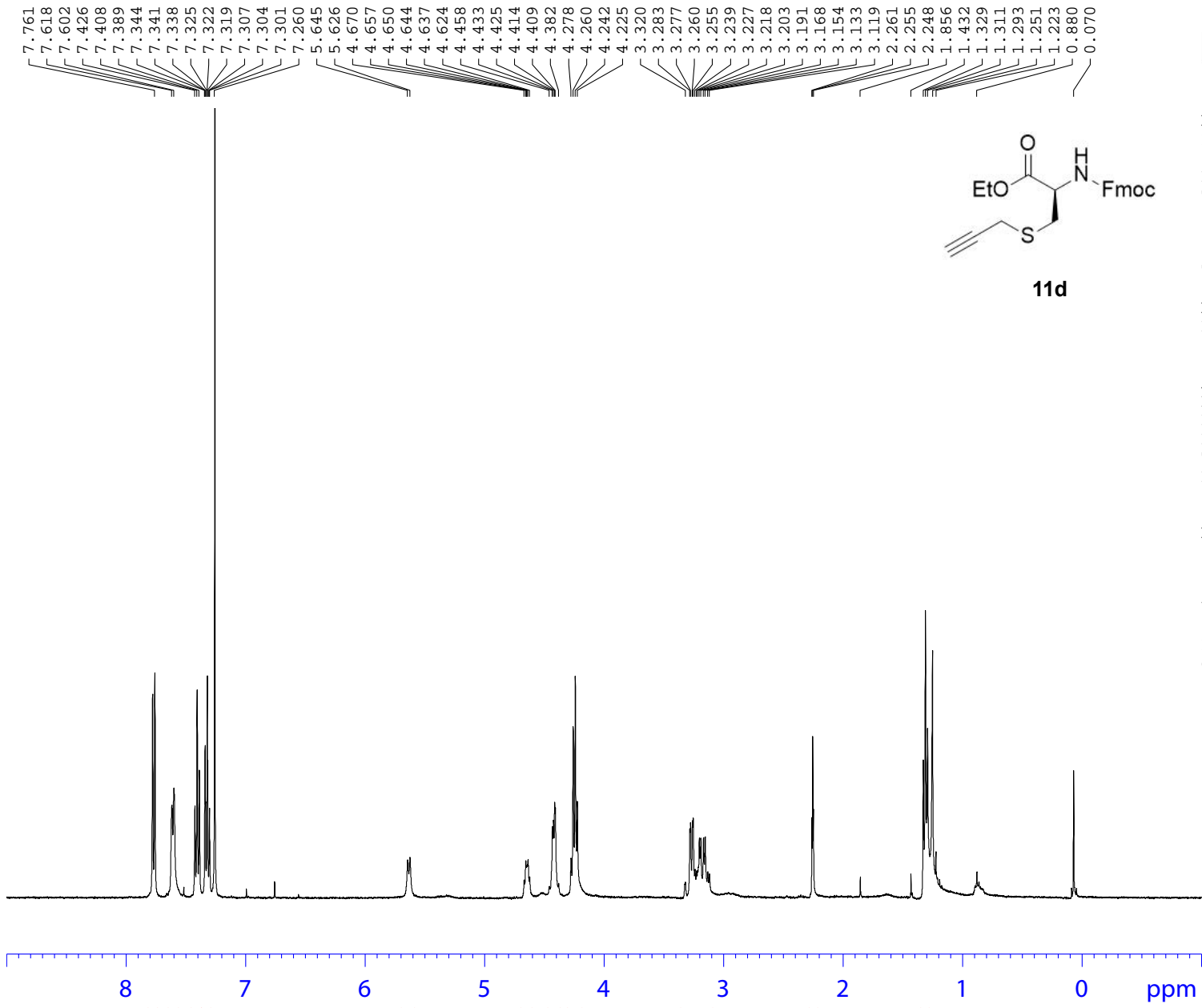
NAME SW06-138-C
EXPNO 11
PROCNO 1
Date_ 20150401
Time 1.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 96.9 K
D1 2.00000000 sec
D11 0.03000000 sec



===== 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



```

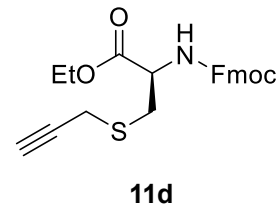
NAME          SW06-164-cr
EXPNO         10
PROCNO        1
Date_         20150415
Time          16.06
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            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

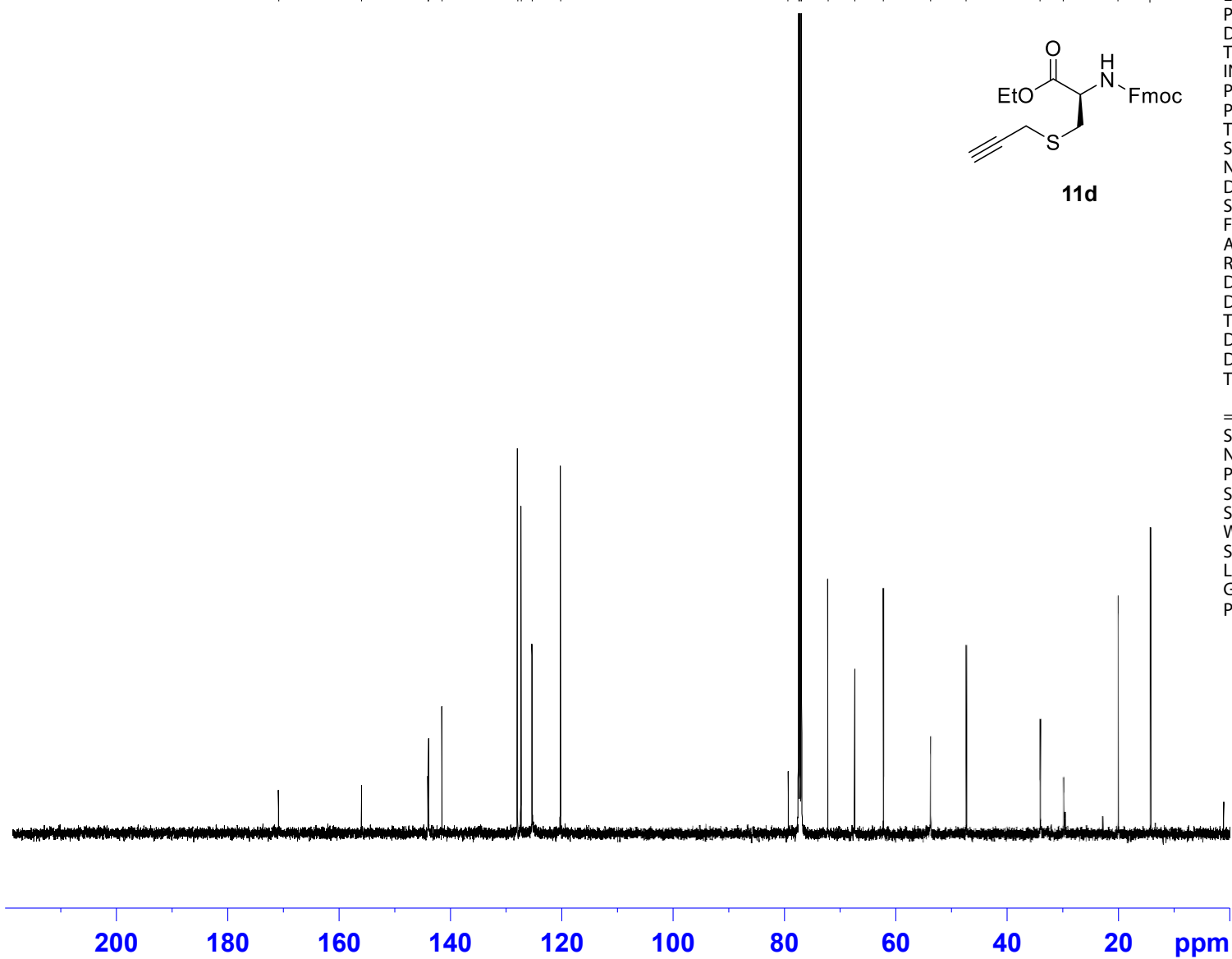


170.807
155.901
143.982
143.876
141.485
127.891
127.241
125.240
120.157
79.382
77.414
77.160
76.906
72.146
67.320
62.164
53.686
47.301
34.044
29.846
20.090
14.284



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

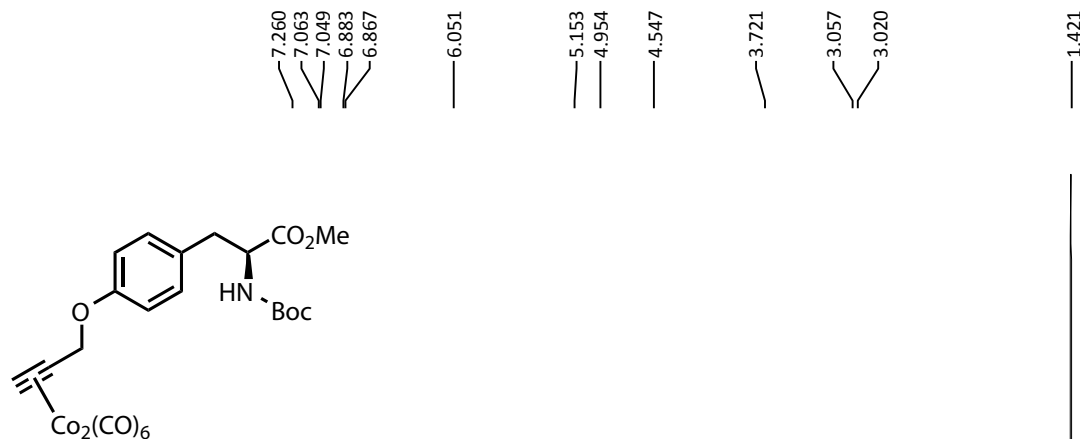
Date_ 20150512
Time 19.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 219998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.045455 Hz
AQ 10.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.0000000 sec
TD0 1

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

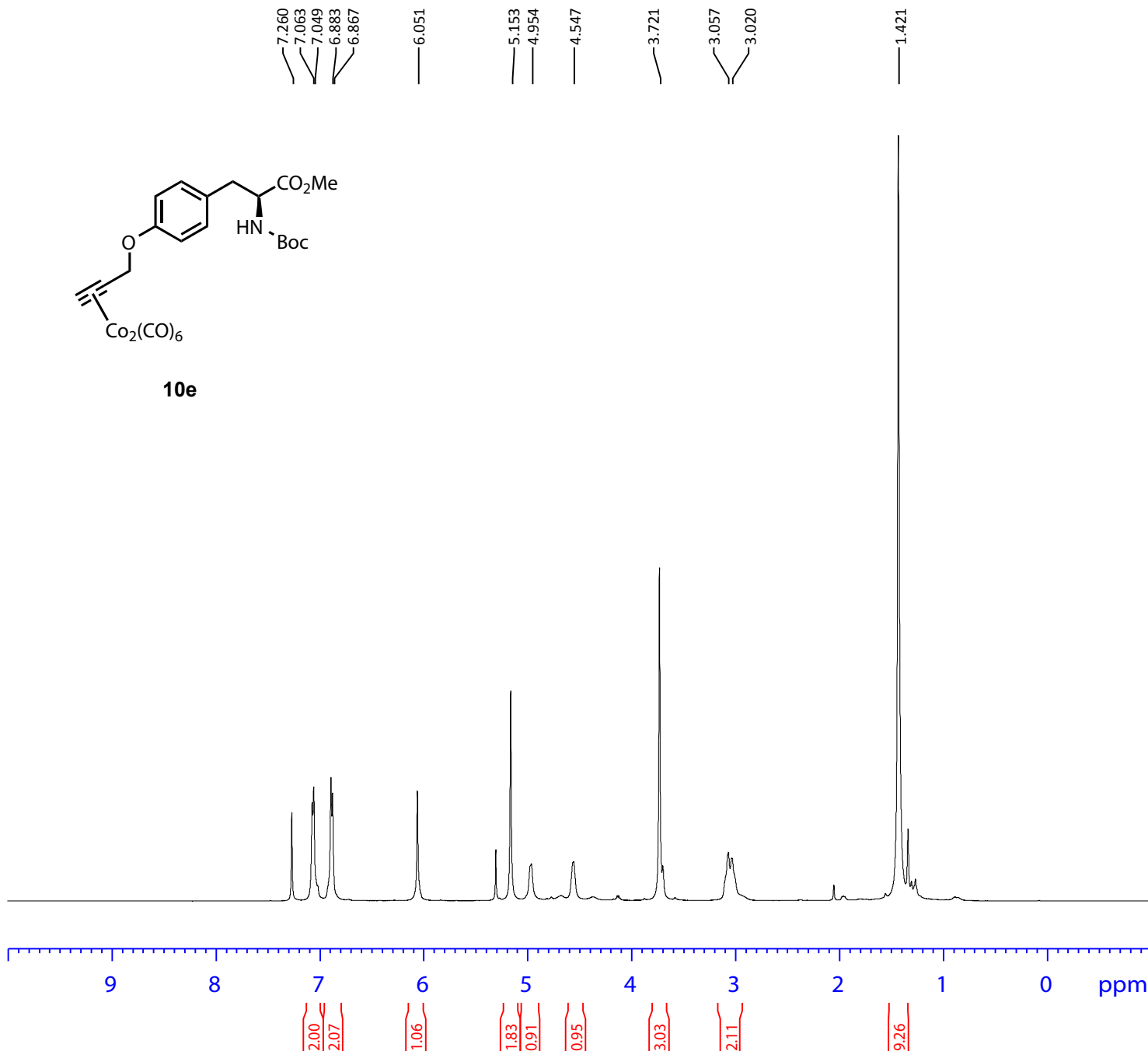
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters

SI 65536
SF 500.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

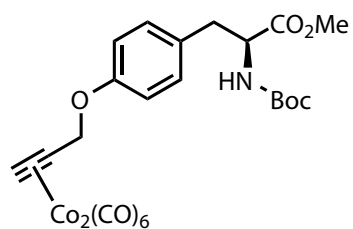


10e

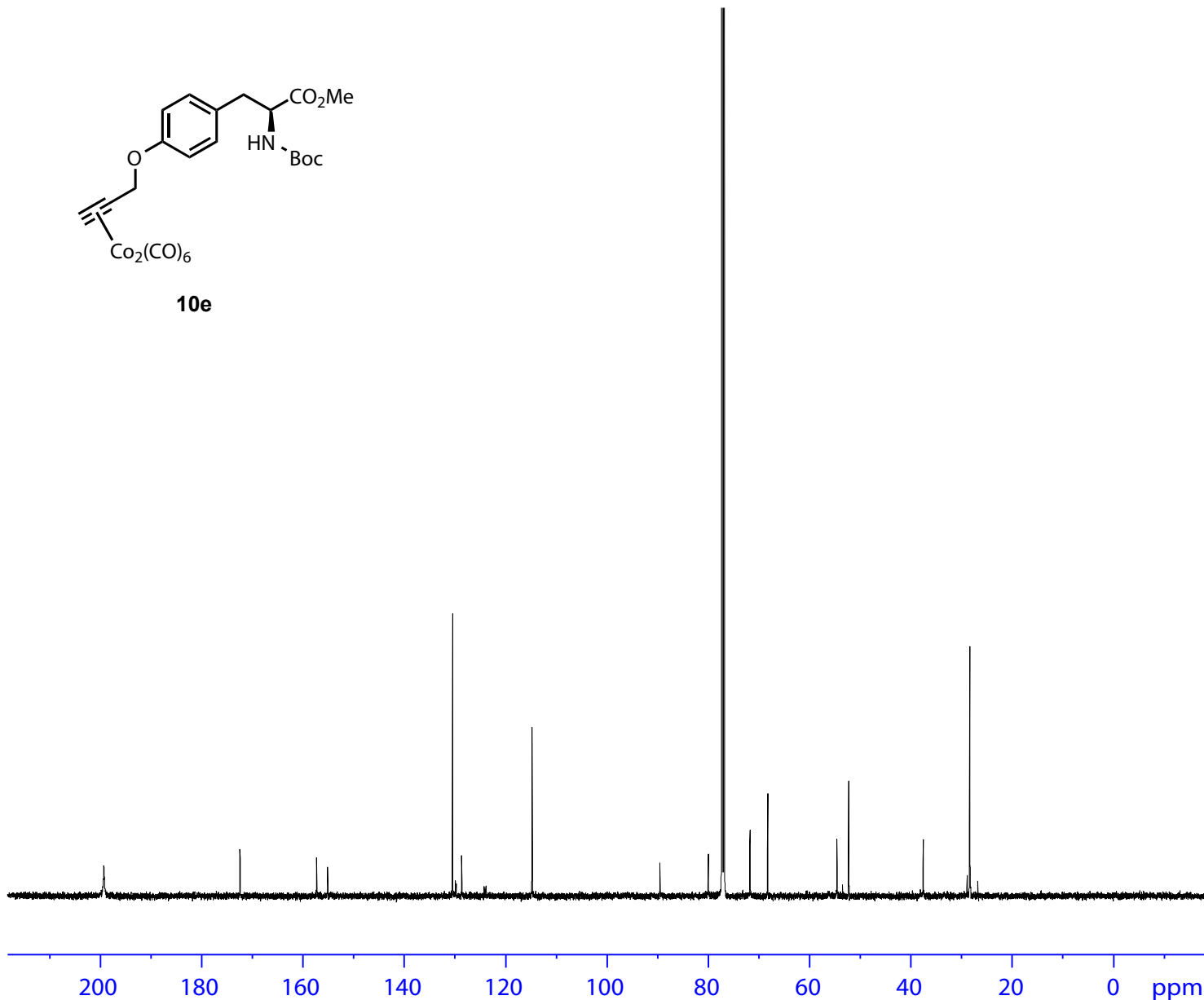




199.27
172.38
157.26
155.09
130.41
128.61
114.70
89.47
79.91
77.28
77.02
76.77
71.70
68.19
54.53
52.21
37.48
28.30



10e



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

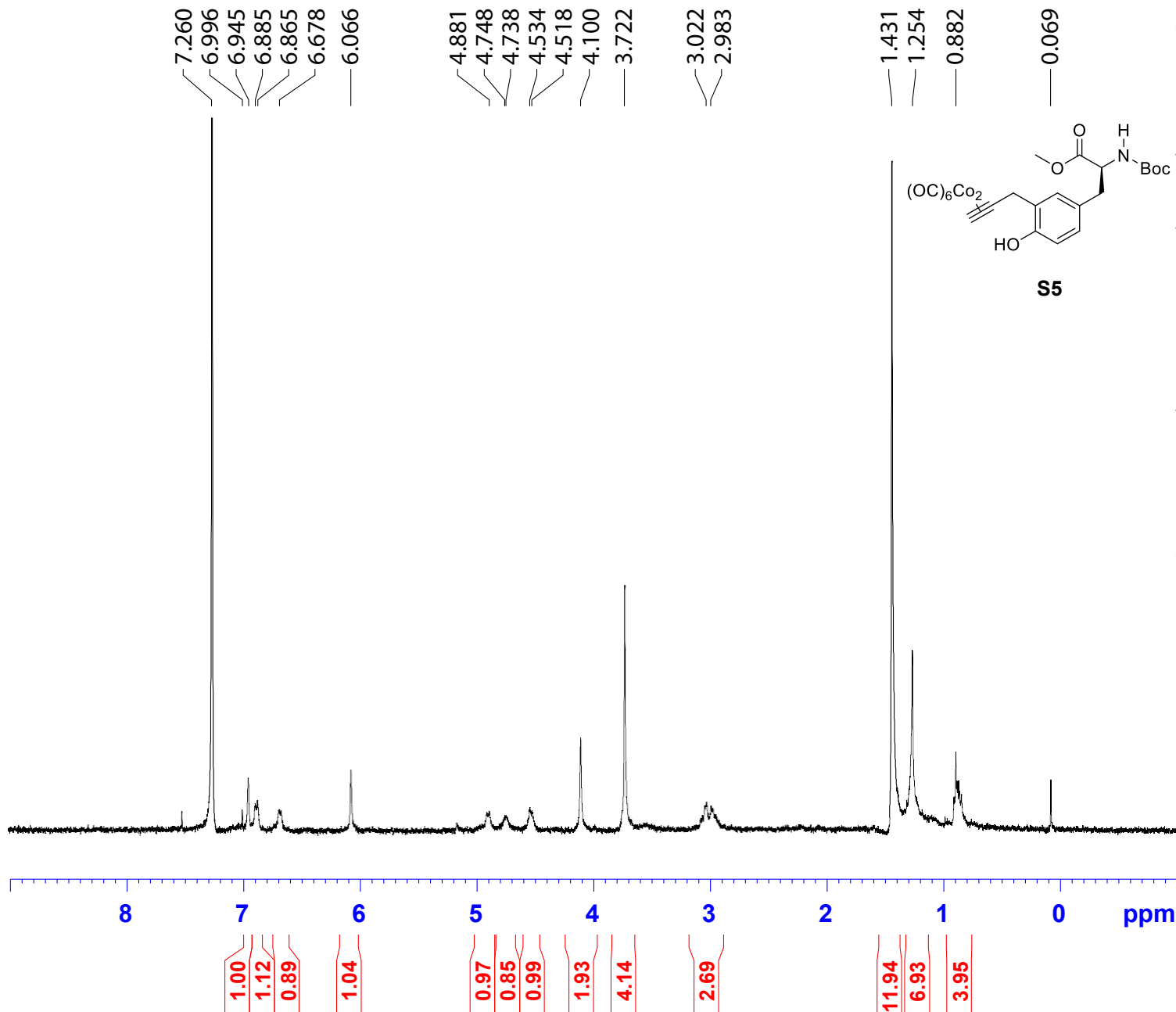
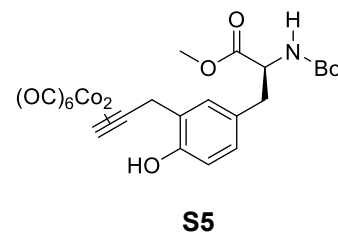
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SW06-166-D 1H 400



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

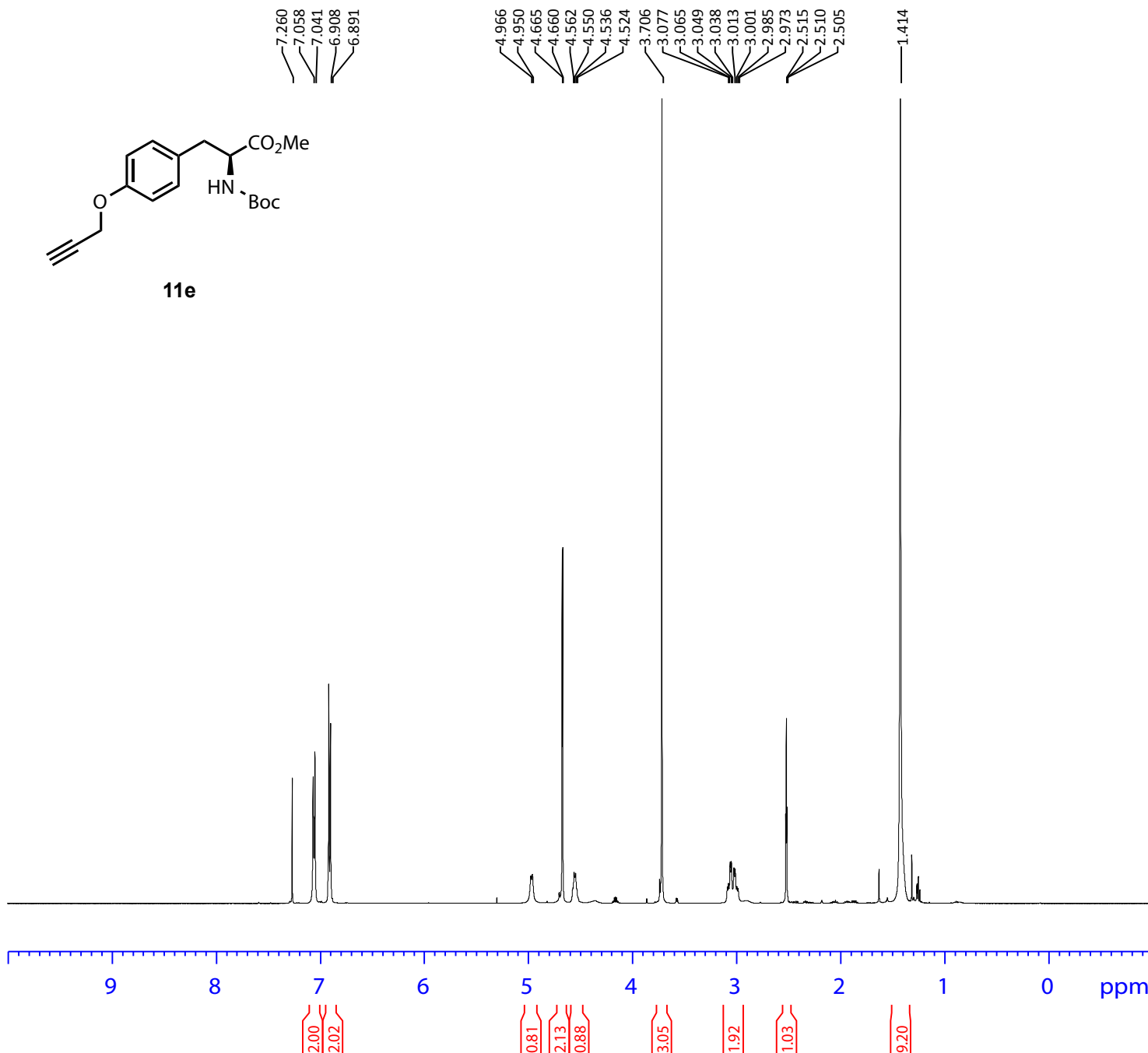
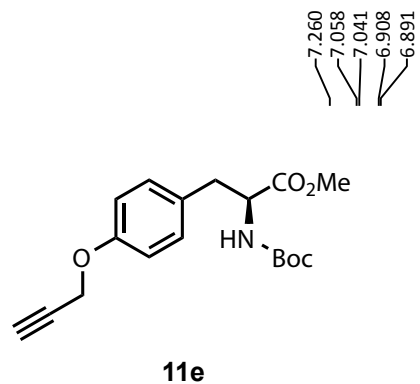
Date_ 20150414
Time 14.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 219998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.045455 Hz
AQ 10.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.0000000 sec
TD0 1

===== 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 =====

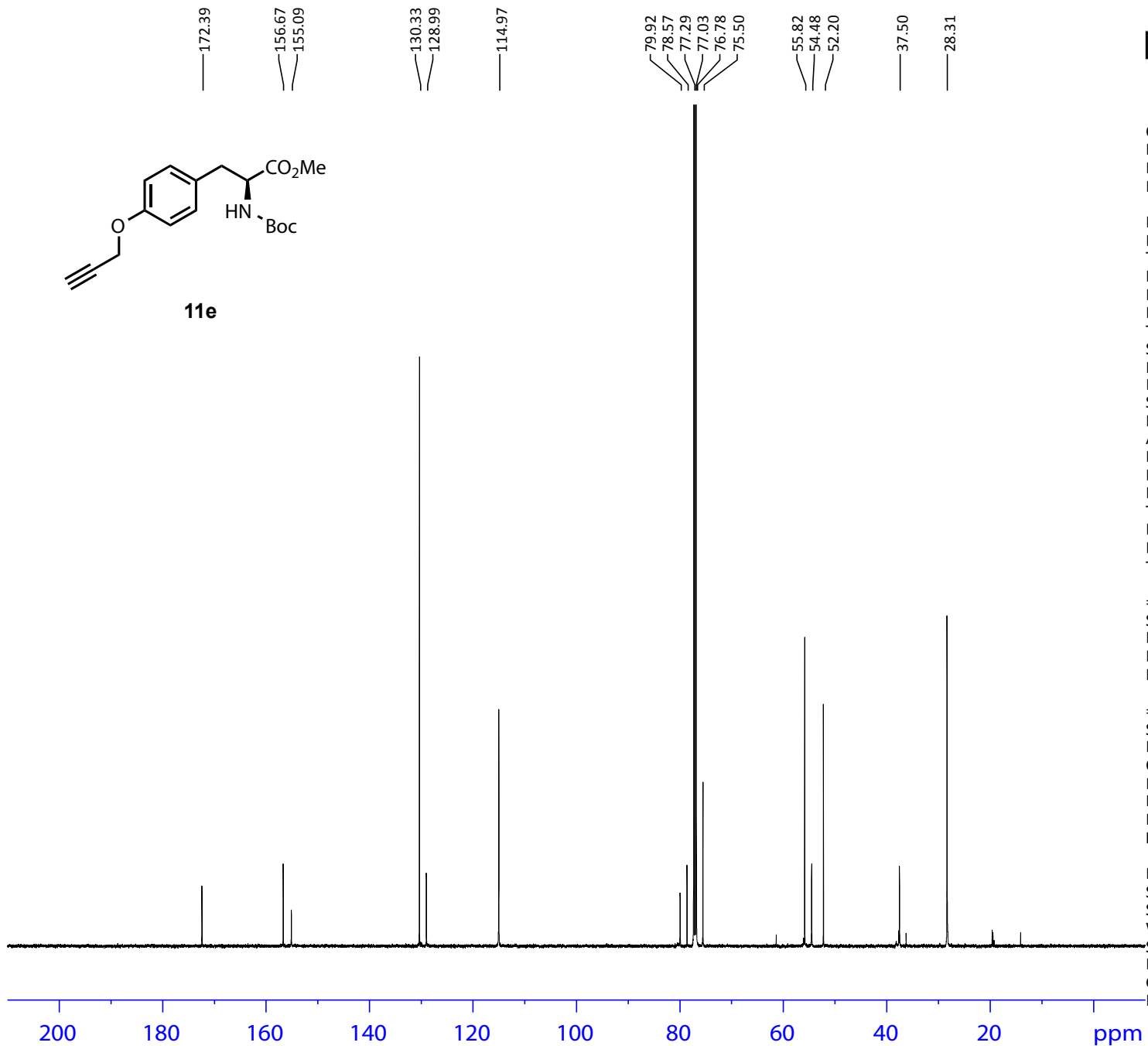
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

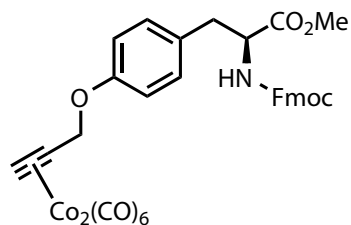
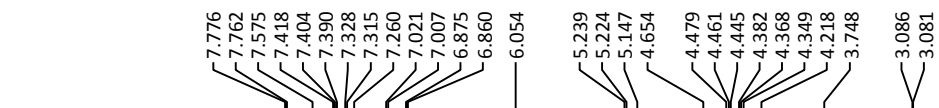
===== CHANNEL f2 =====

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

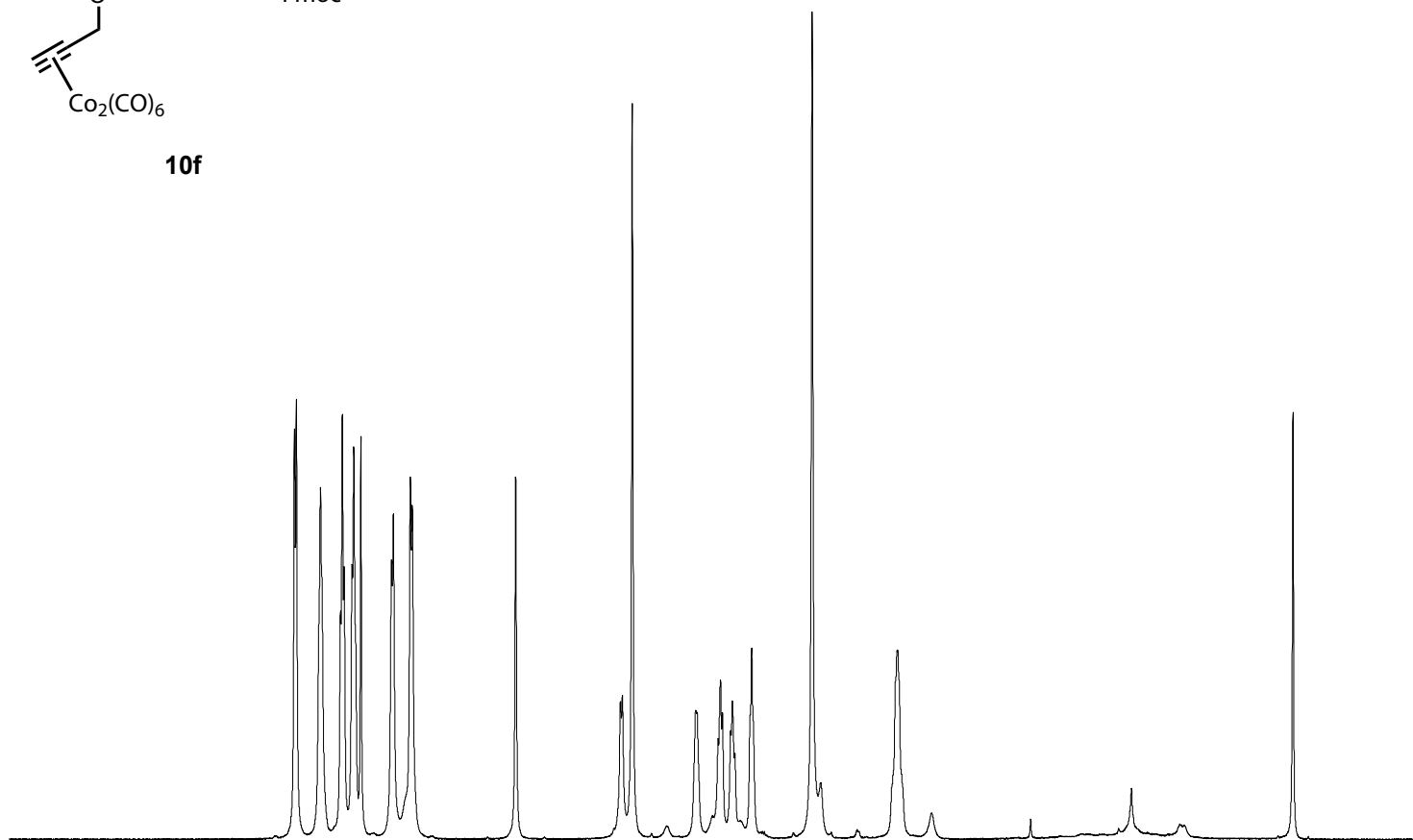
F2 - Processing parameters

SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





10f



2.00
2.04
4.00
1.78
2.24
0.98
0.93
1.95
0.89
2.20
1.09
3.04
1.73

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 =====

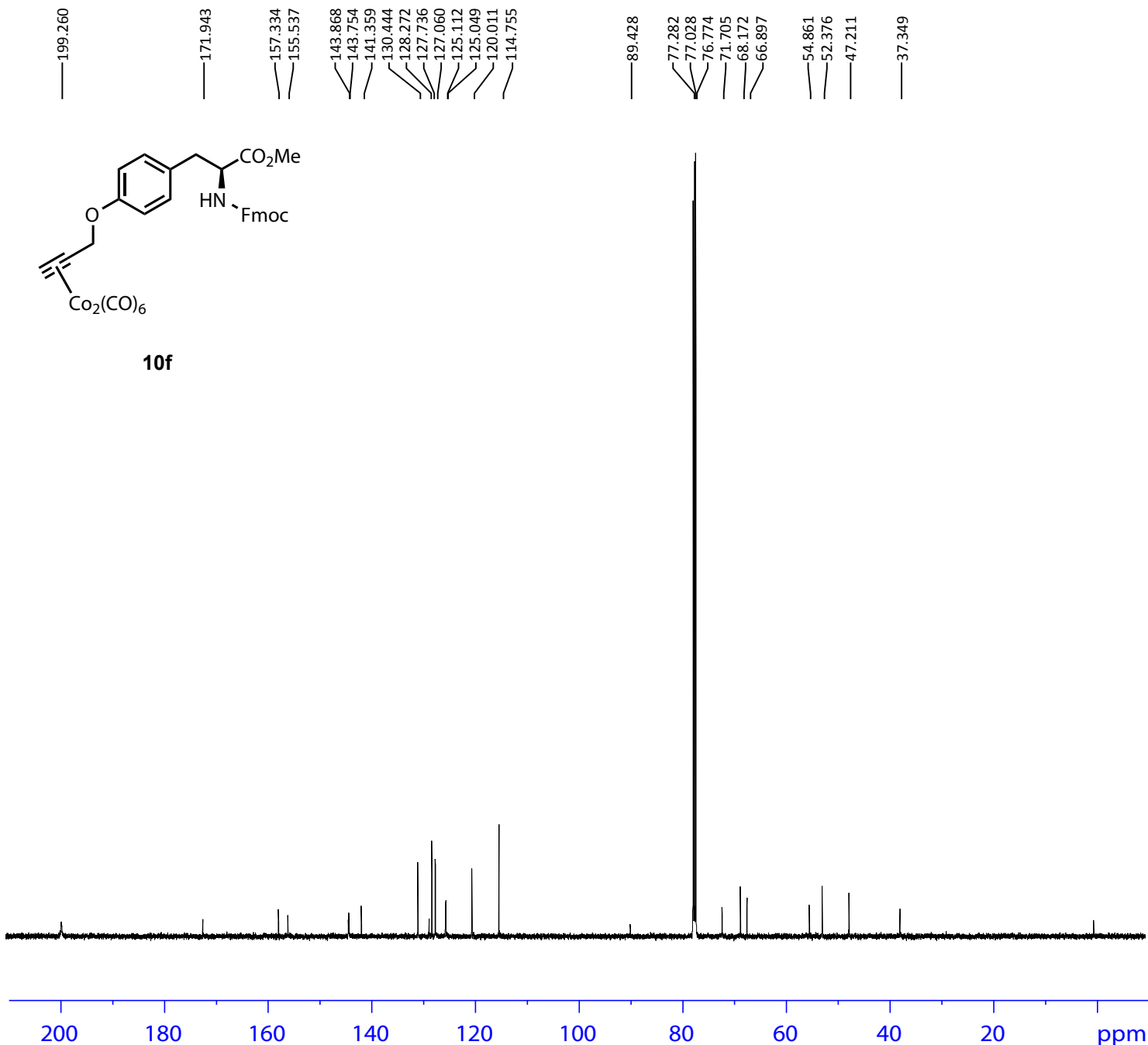
SFO1 125.7703643 MHz
NUC1 13C
P1 10.00 usec
PLW1 84.00000000 W

===== 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

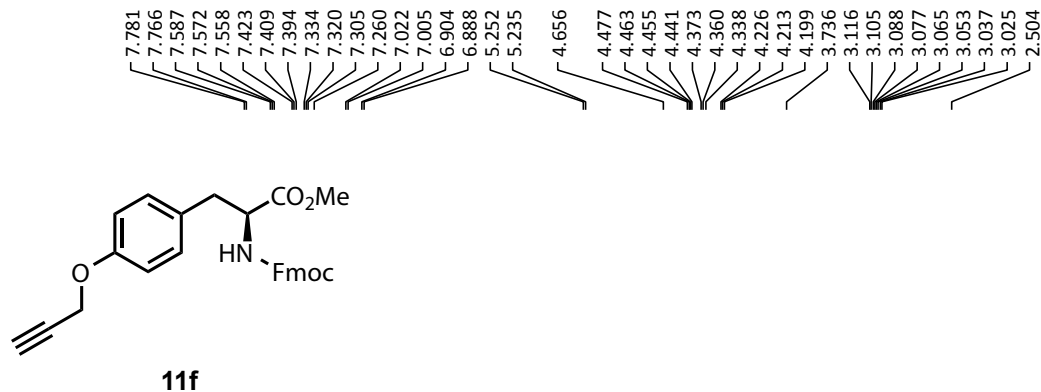
Date_ 20150728
Time 14.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 199998
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.050000 Hz
AQ 9.9998999 sec
RG 86.1
DW 50.000 usec
DE 13.29 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

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

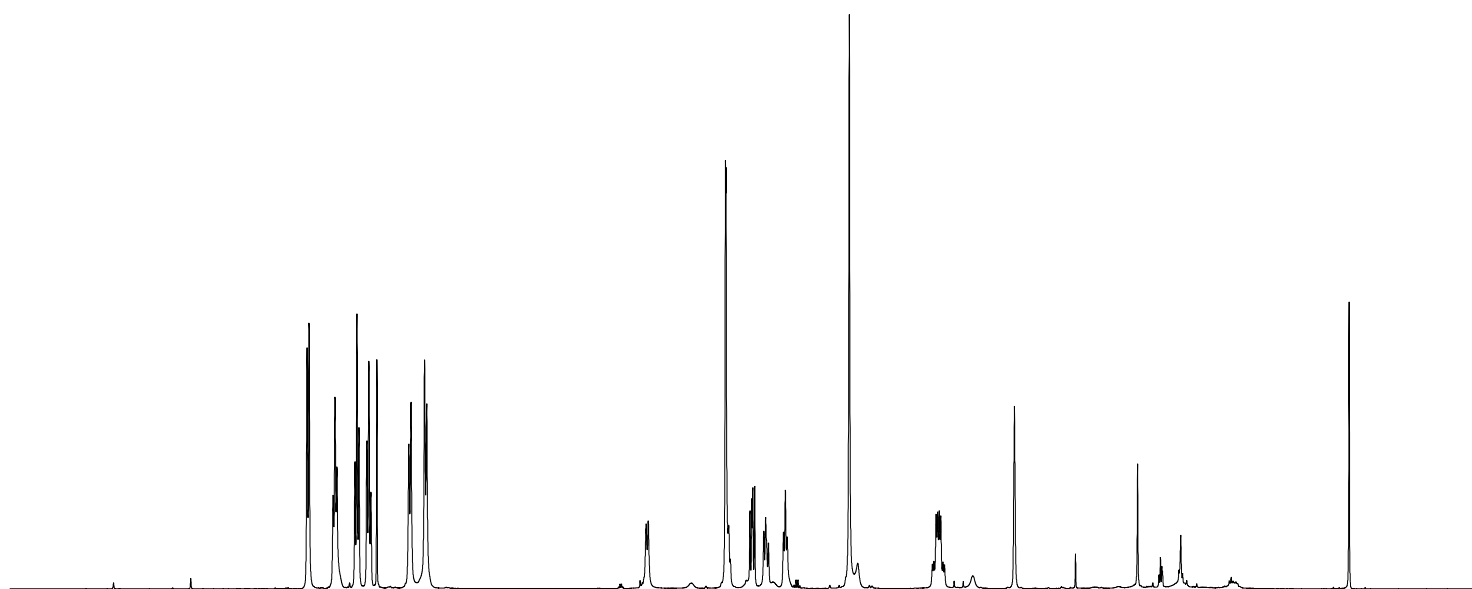
SFO1 500.1330885 MHz
NUC1 1H
P1 10.99 usec
PLW1 15.00000000 W

F2 - Processing parameters

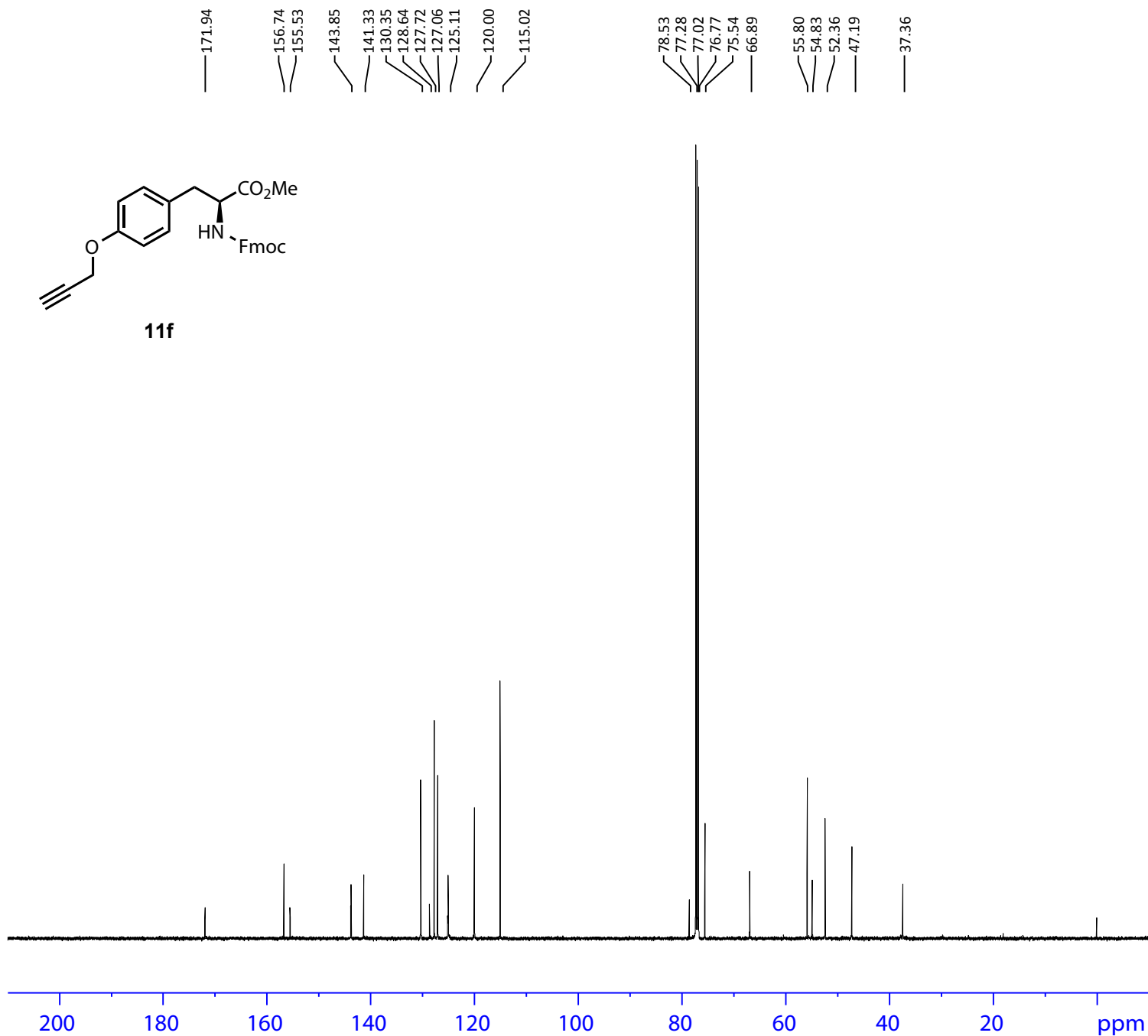
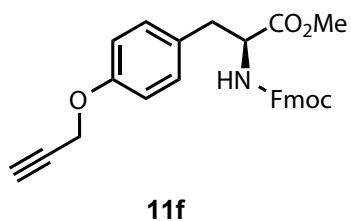
SI 65536
SF 500.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7.781
7.766
7.587
7.572
7.558
7.423
7.409
7.394
7.334
7.320
7.305
7.260
7.022
7.005
6.904
6.888
5.252
5.235
4.656
4.477
4.463
4.455
4.441
4.373
4.360
4.338
4.226
4.213
4.199
3.736
3.116
3.105
3.088
3.077
3.065
3.053
3.037
3.025
2.504



2.00
2.03
2.03
2.04
1.76
2.29
0.91
2.88
1.11
1.00
1.04
3.03
1.75
1.01



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

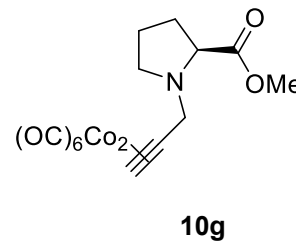
F2 - Processing parameters

SI 32768
SF 125.7577914 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SW07-060-C

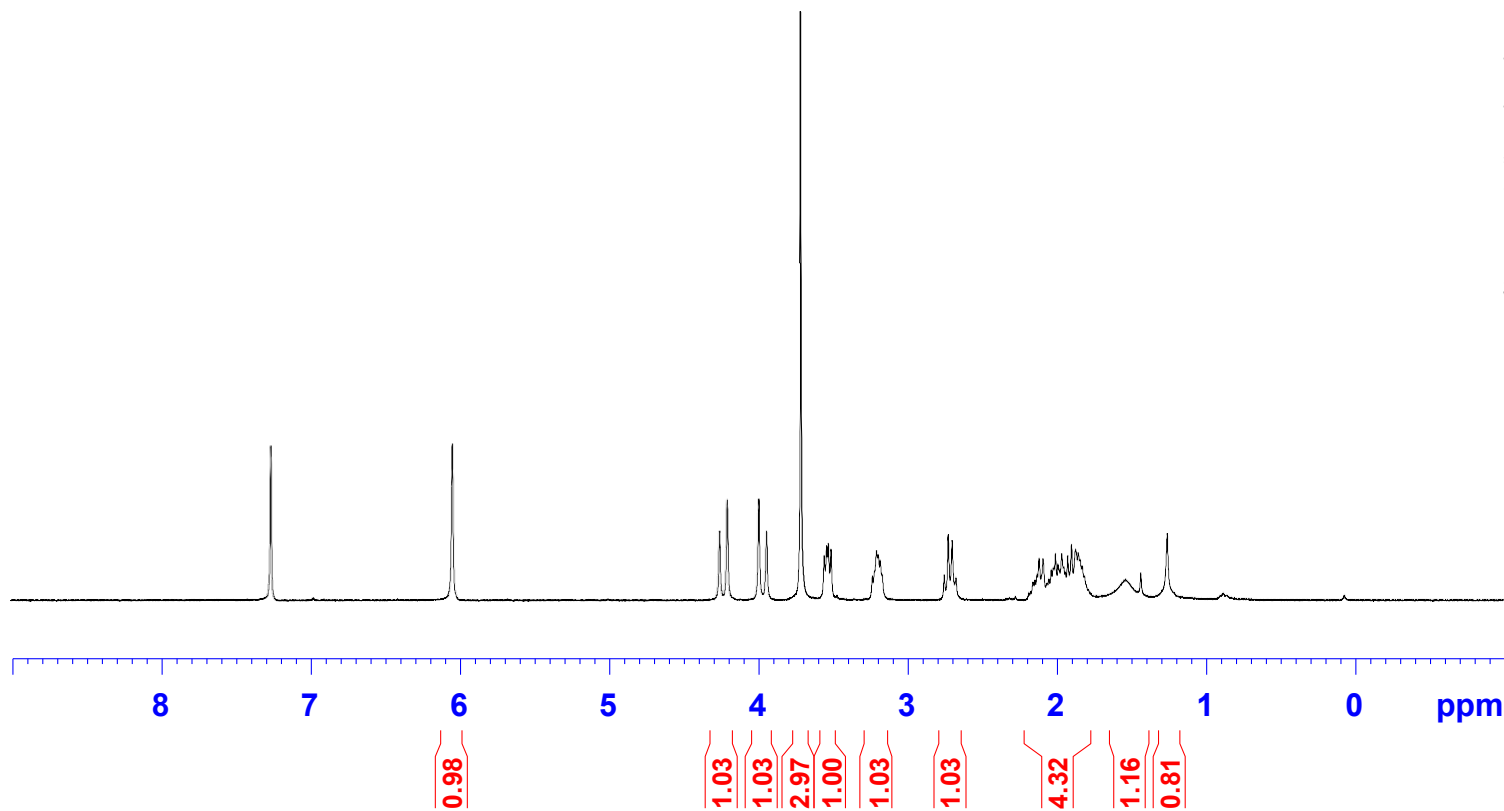


7.260
6.046
4.259
4.207
3.996
3.945
3.712
3.551
3.534
3.524
3.506
3.228
3.201
3.190
3.175
2.747
2.720
2.694
2.668
2.126
2.111
2.086
2.030
2.017
2.002
1.985
1.960
1.936
1.920
1.895
1.868
1.850
1.837
1.822
1.807



NAME SW07-060-C
EXPNO 10
PROCNO 1
Date_ 20150804
Time 18.03
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zg30
TD 32768
SOLVENT CDCI3
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

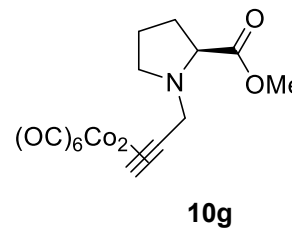
===== CHANNEL f1 =====
SFO1 300.2318540 MHz
NUC1 1H
P1 12.71 usec
SI 32768
SF 300.2300085 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



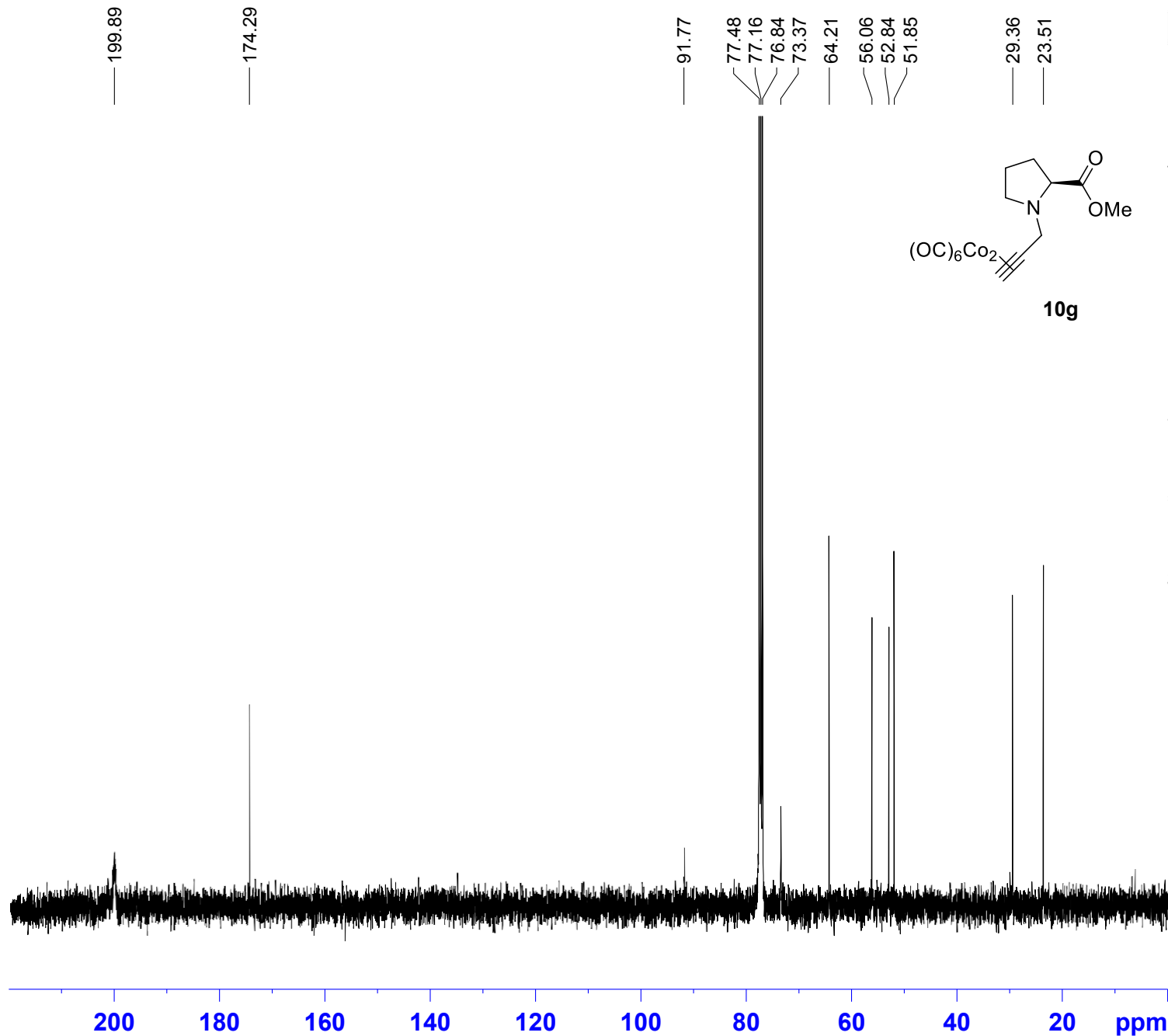
SW07-060-C 13C 400



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 2.0000000 sec
D11 0.0300000 sec



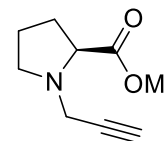
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SW07-074-cr



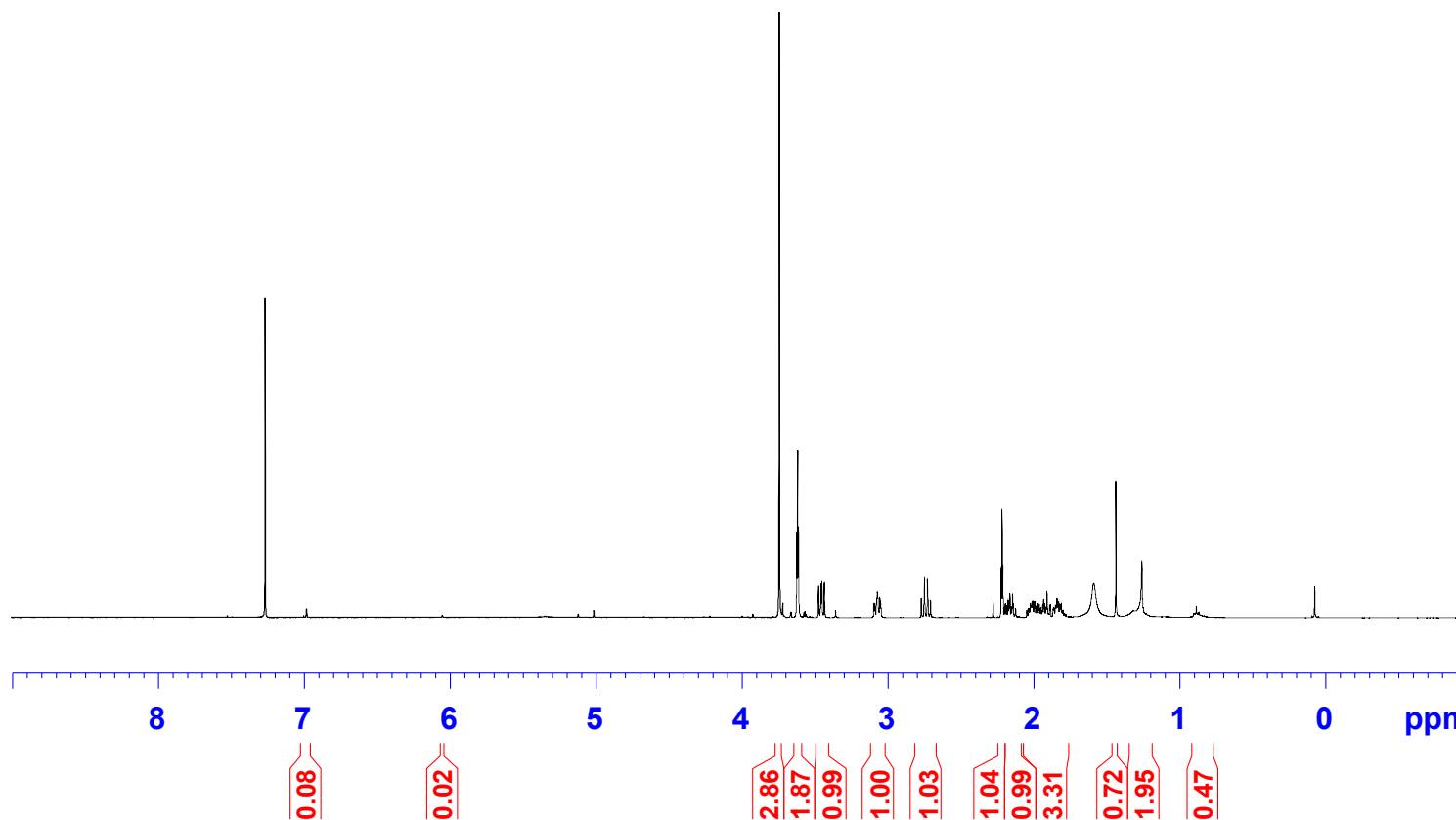
7.260
3.735
3.613
3.607
3.602
3.464
3.448
3.442
3.425
3.086
3.079
3.062
3.045
3.039
2.761
2.741
2.738
2.719
2.716
2.697
2.213
2.207
2.201
2.167
2.158
2.154
2.136
2.002
1.997
1.985
1.923
1.900
1.832
1.821
1.579



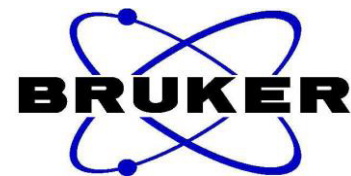
11g

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

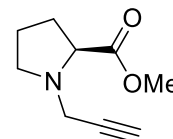
===== CHANN EL f1 =====
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.130099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



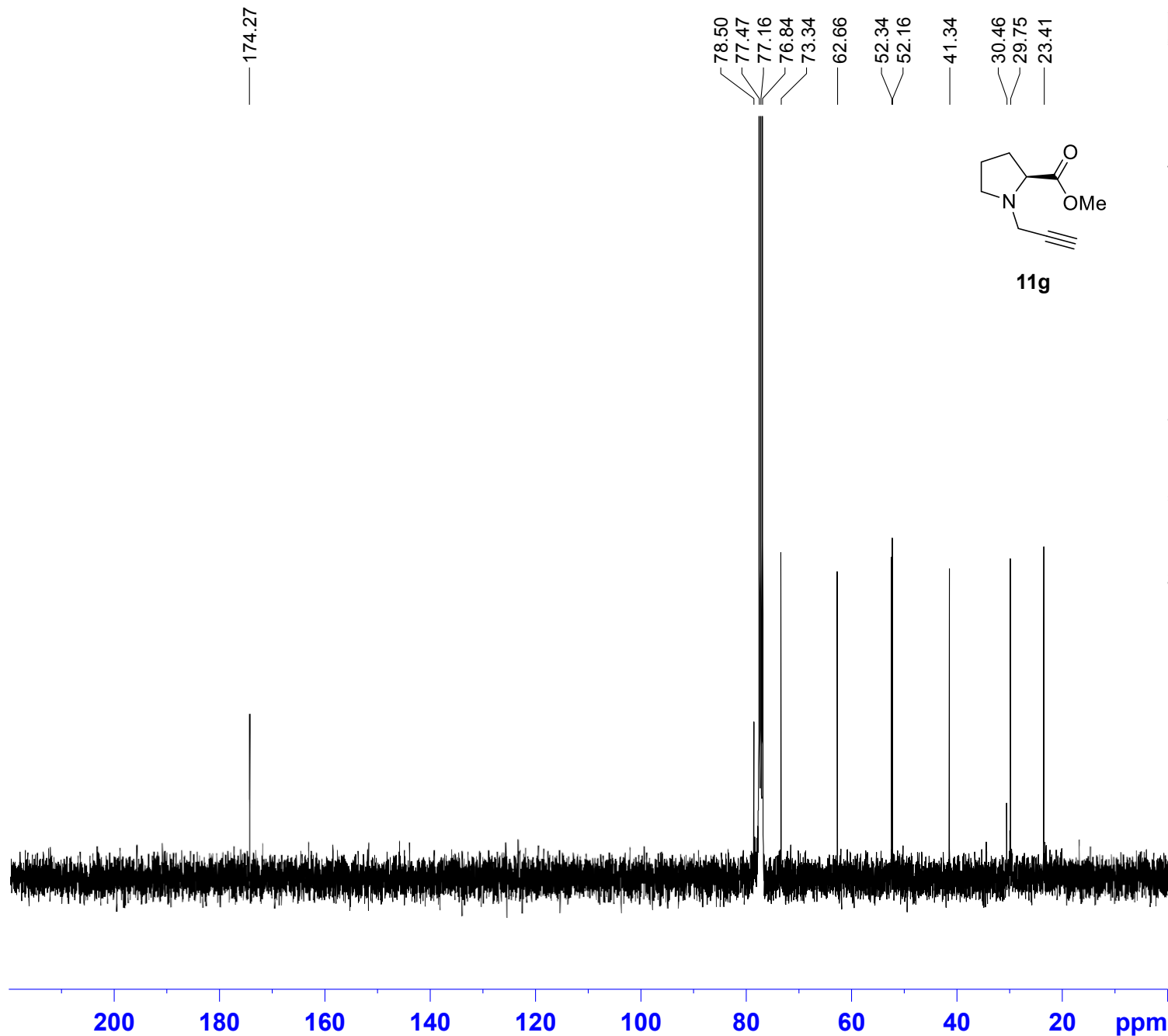
SW07-074-cr 13C 400



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 2.0000000 sec
D11 0.0300000 sec



11g



===== 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

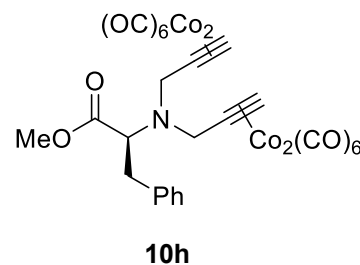
SW07-111-B 1H 400



7.308
7.291
7.272
7.260
7.244
7.226
7.181
7.163
6.100

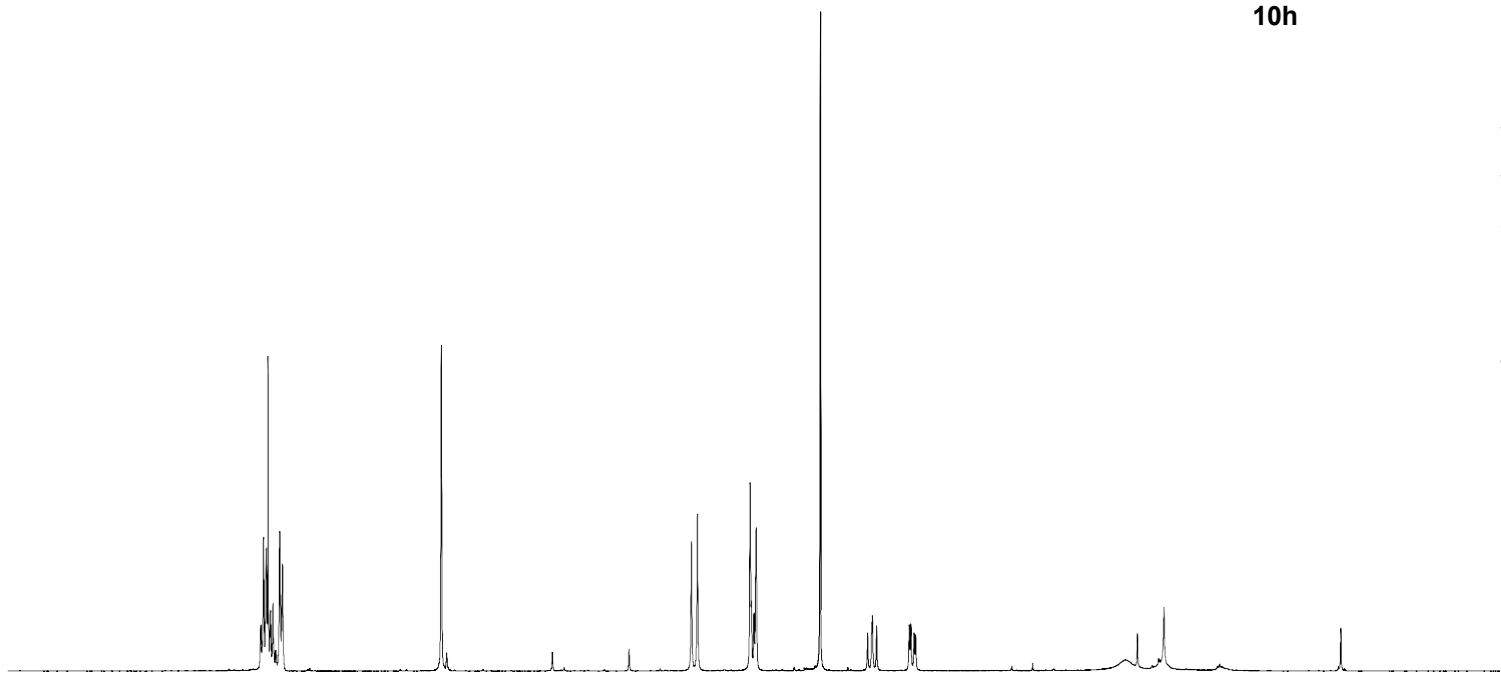
4.428
4.388
4.035
4.026
4.009
3.994
3.558
3.238
3.205
3.178
2.959
2.948
2.926
2.916

1.432
1.254
0.070



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



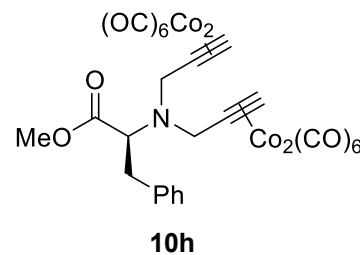
8 7 6 5 4 3 2 1 0 ppm

4.36
1.99
2.00
2.01
3.04
3.04
1.02
1.03

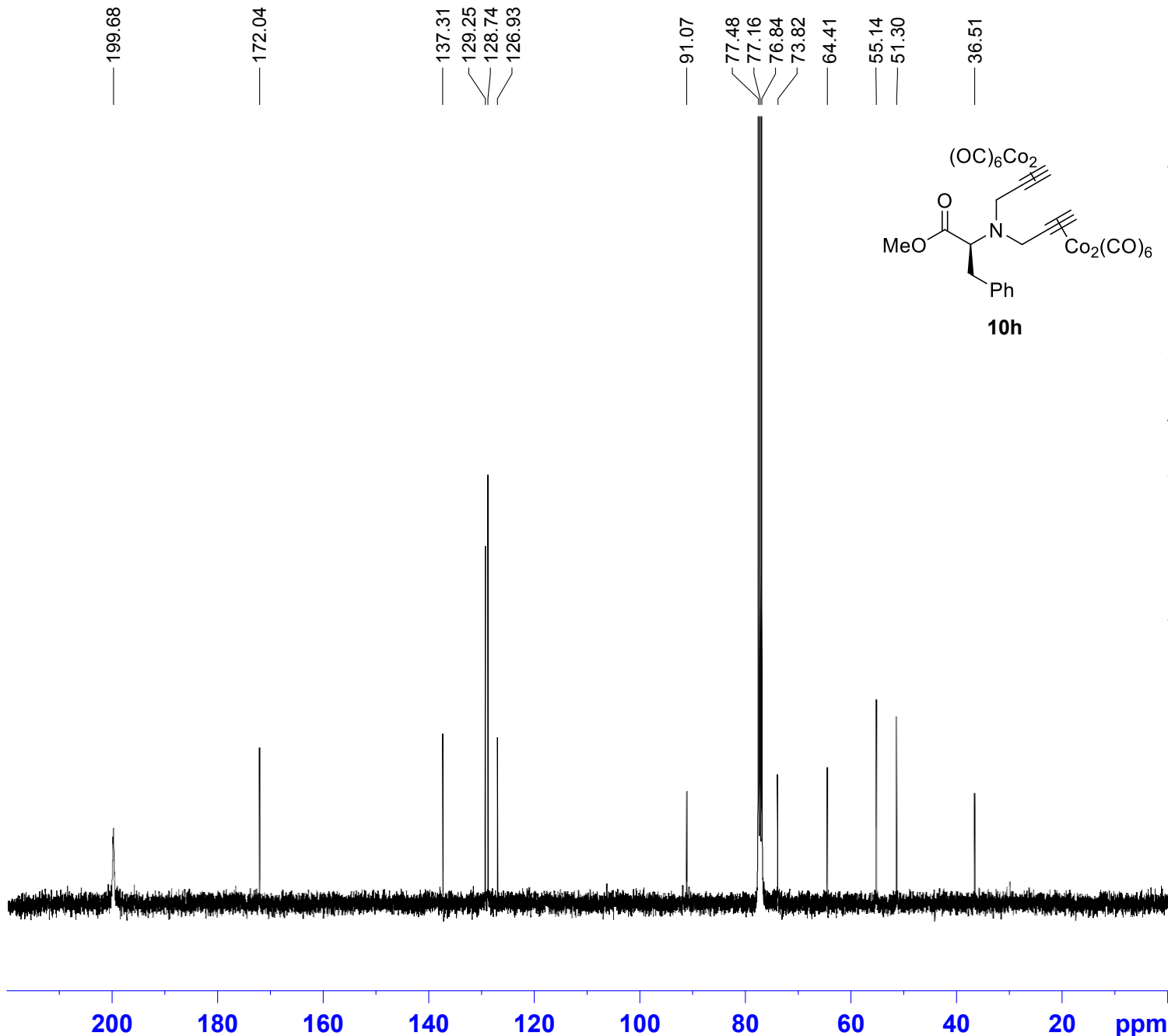
SW07-111-B 1H 400



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

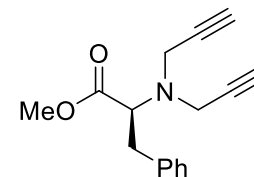


SW07-118-A



7.295
7.292
7.275
7.260
7.225
7.222
7.205
7.201
7.184

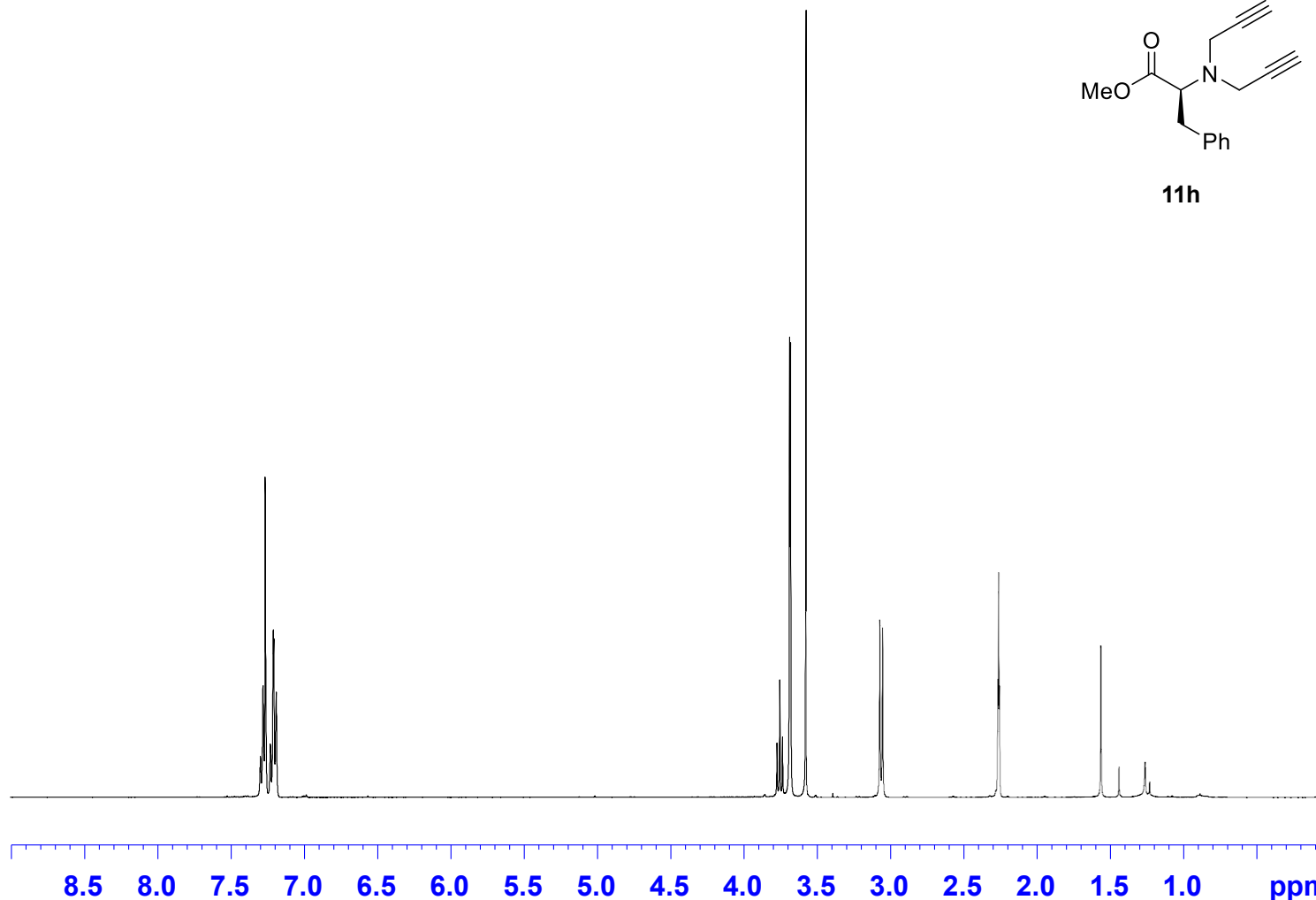
3.766
3.747
3.728
3.680
3.674
3.568
3.061
3.042
2.257
2.251
2.245



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



2.85
2.91

1.09
3.95
2.94

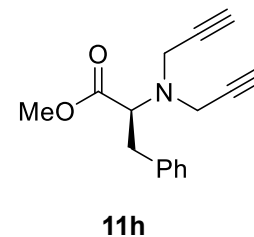
2.00

1.86

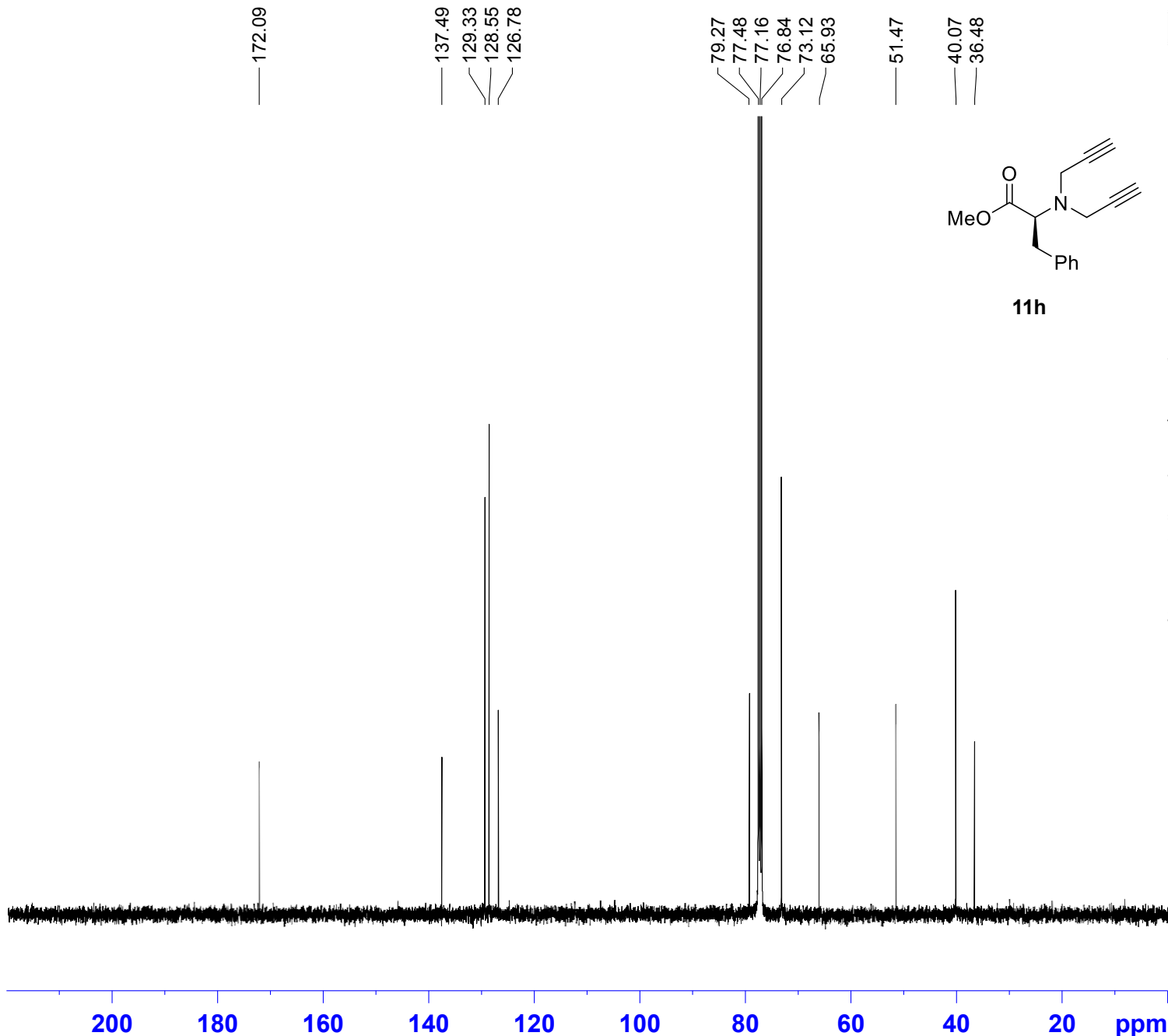
SW07-118-A 13C 400



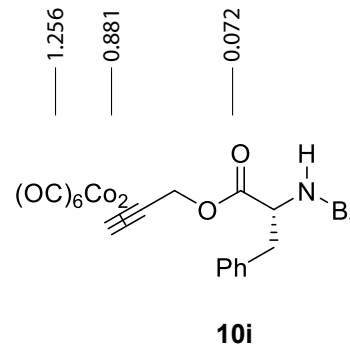
NAME SW07-118-A
EXPNO 11
PROCNO 1
Date_ 20151014
Time 23.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 97.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



===== 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

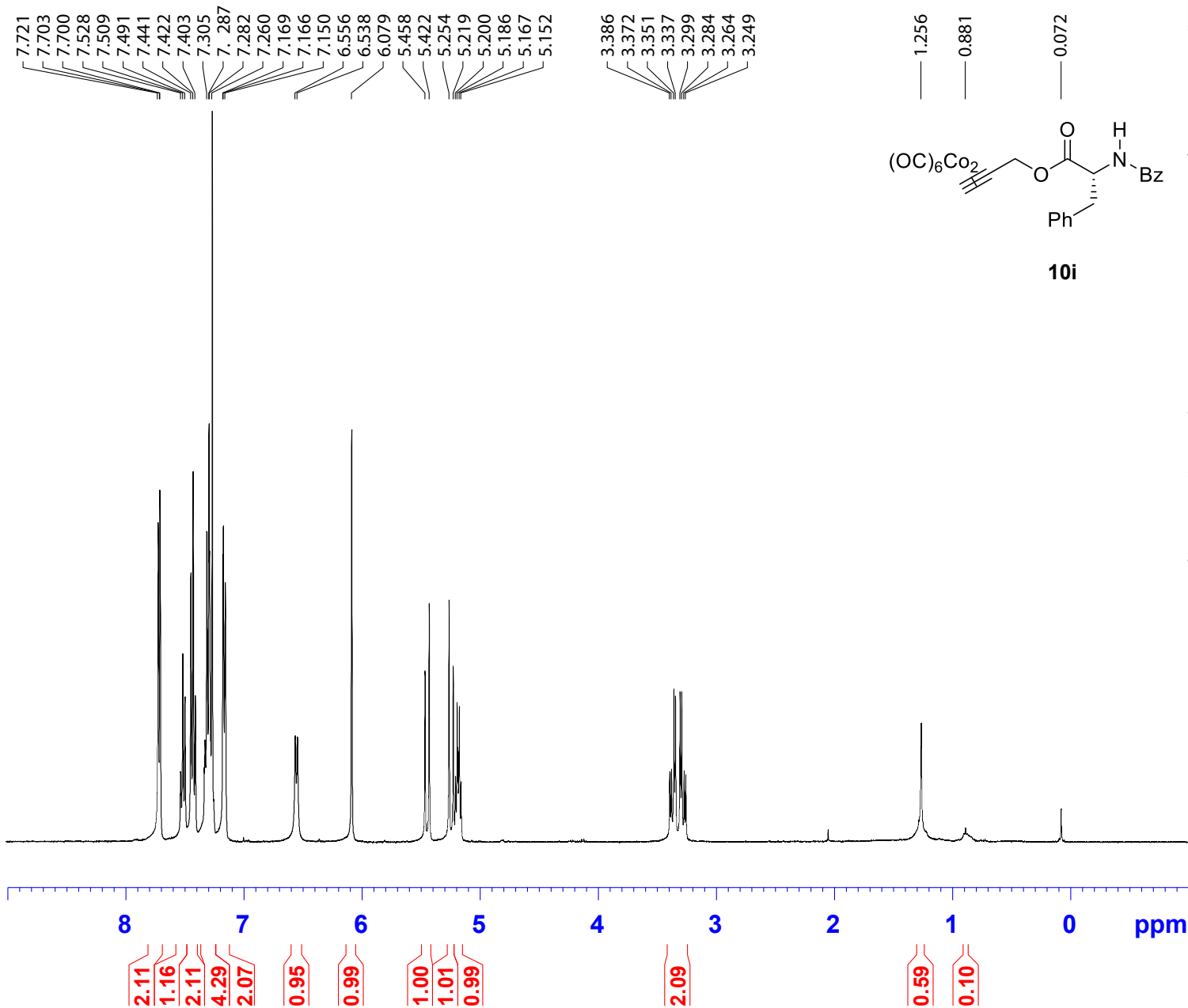


SW06-065-C 1H 400

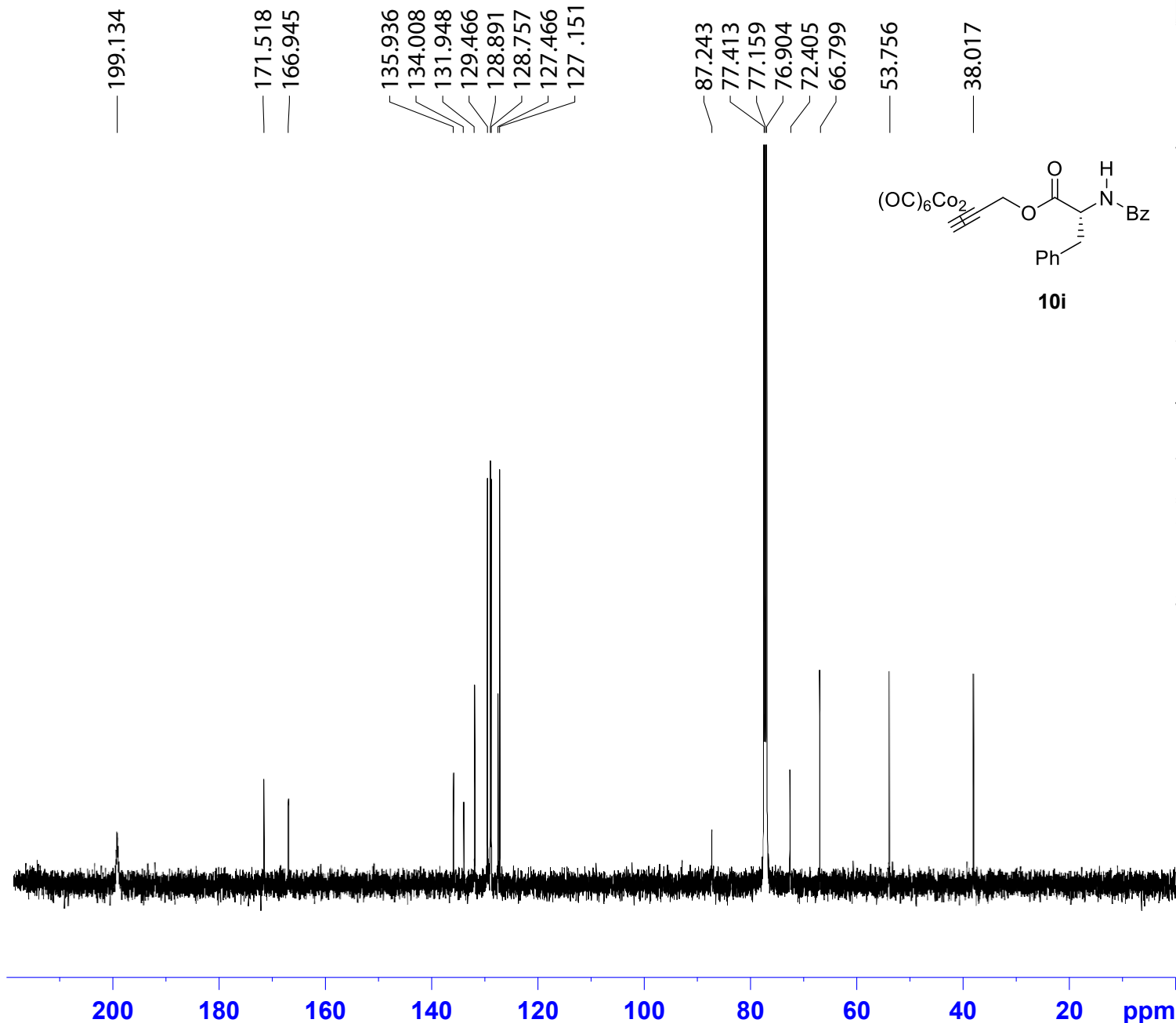


NAME SW06-065-C
EXPNO 10
PROCNO 1
Date_ 20150108
Time 18.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 96.1 K
D1 1.00000000 sec

===== 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



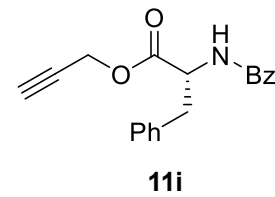
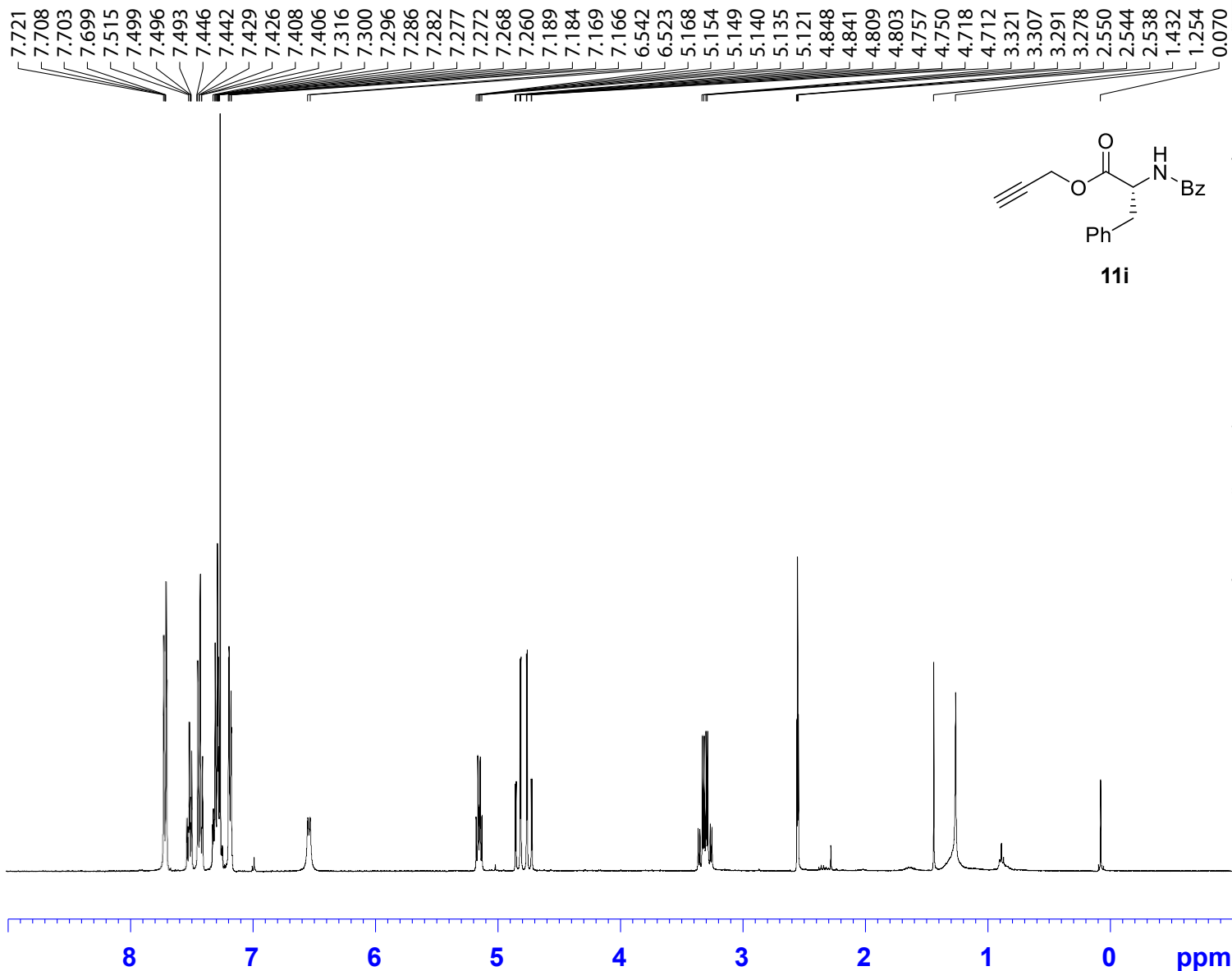
SW06-169-C 13C 500



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

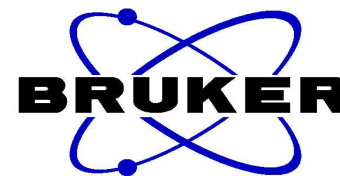
SW06-165-cr 1H 400



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



171.038
167.023

135.664
133.962
131.999
129.631
128.828
128.799
127.463
127.158

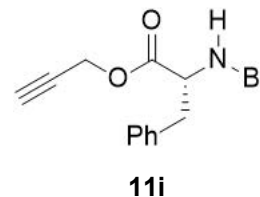
77.414
77.160
77.054
76.906
75.797

53.505
53.034

37.880

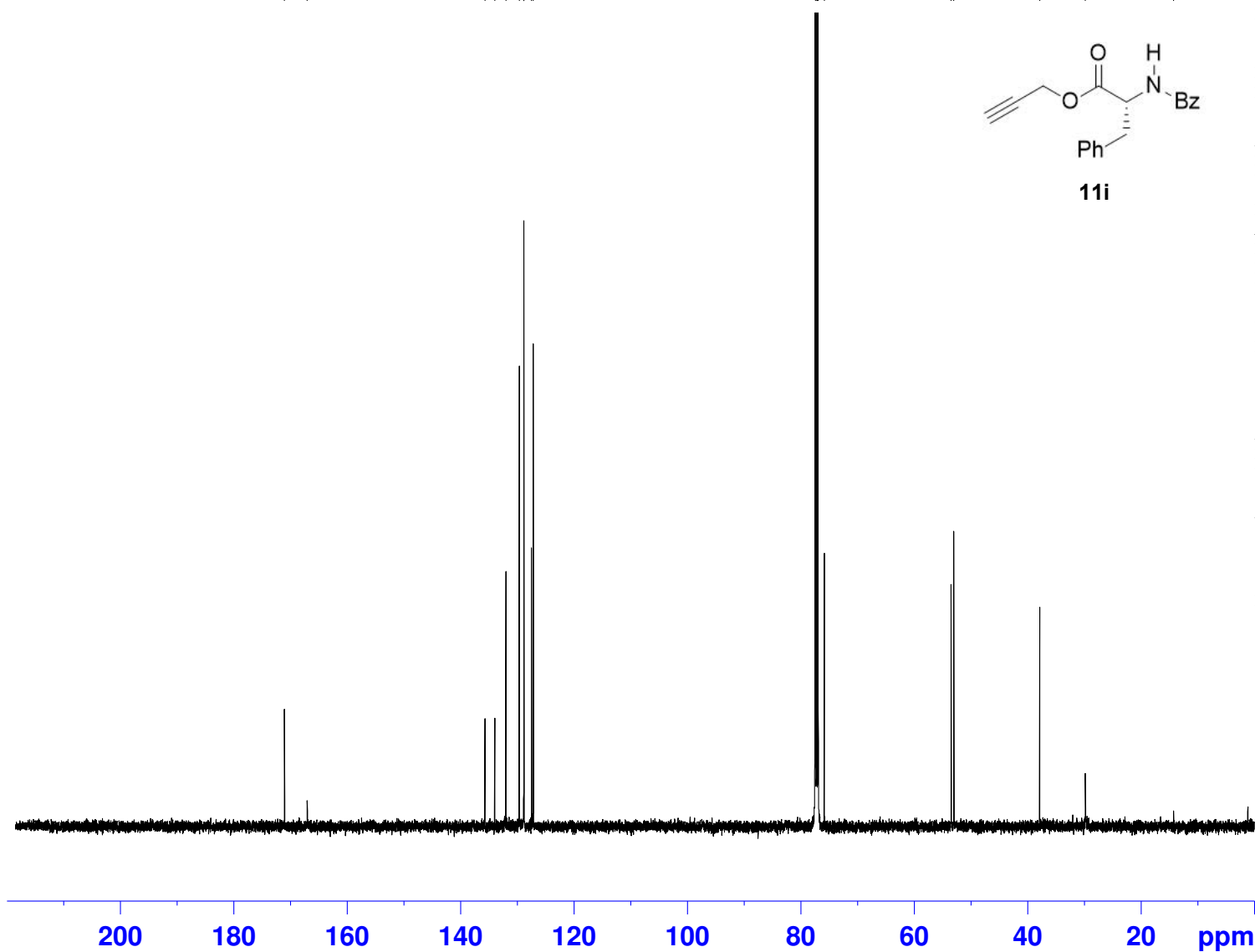
29.849

14.259



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 CDC13
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

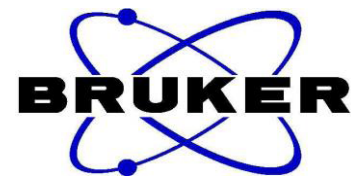
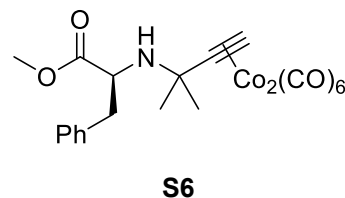


SW07-123-C 1H 400

7.292
7.275
7.260
7.231
7.214
7.190
7.172
— 5.994

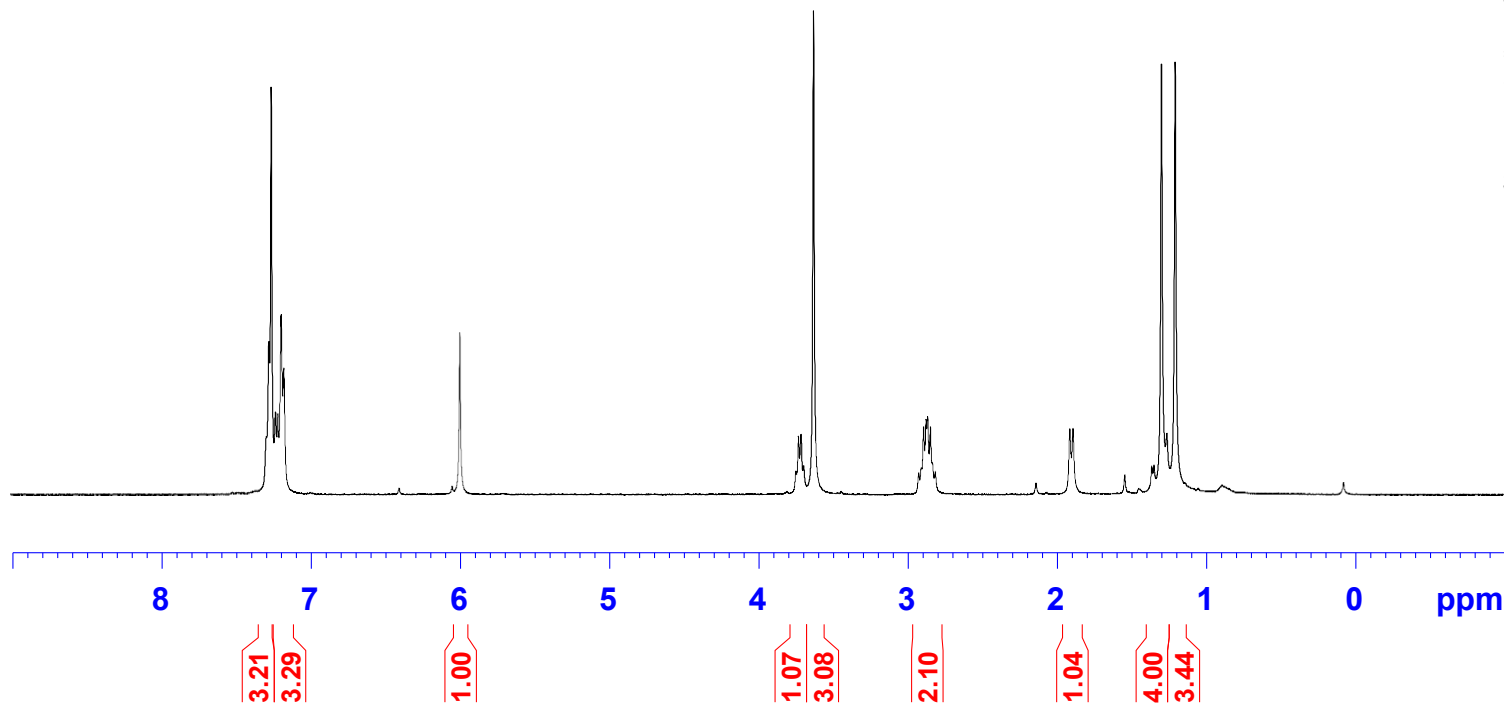
3.745
3.727
3.708
3.690
3.624
2.919
2.902
2.886
2.870
2.859
2.841
2.808
1.907
1.886

1.292
1.200



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

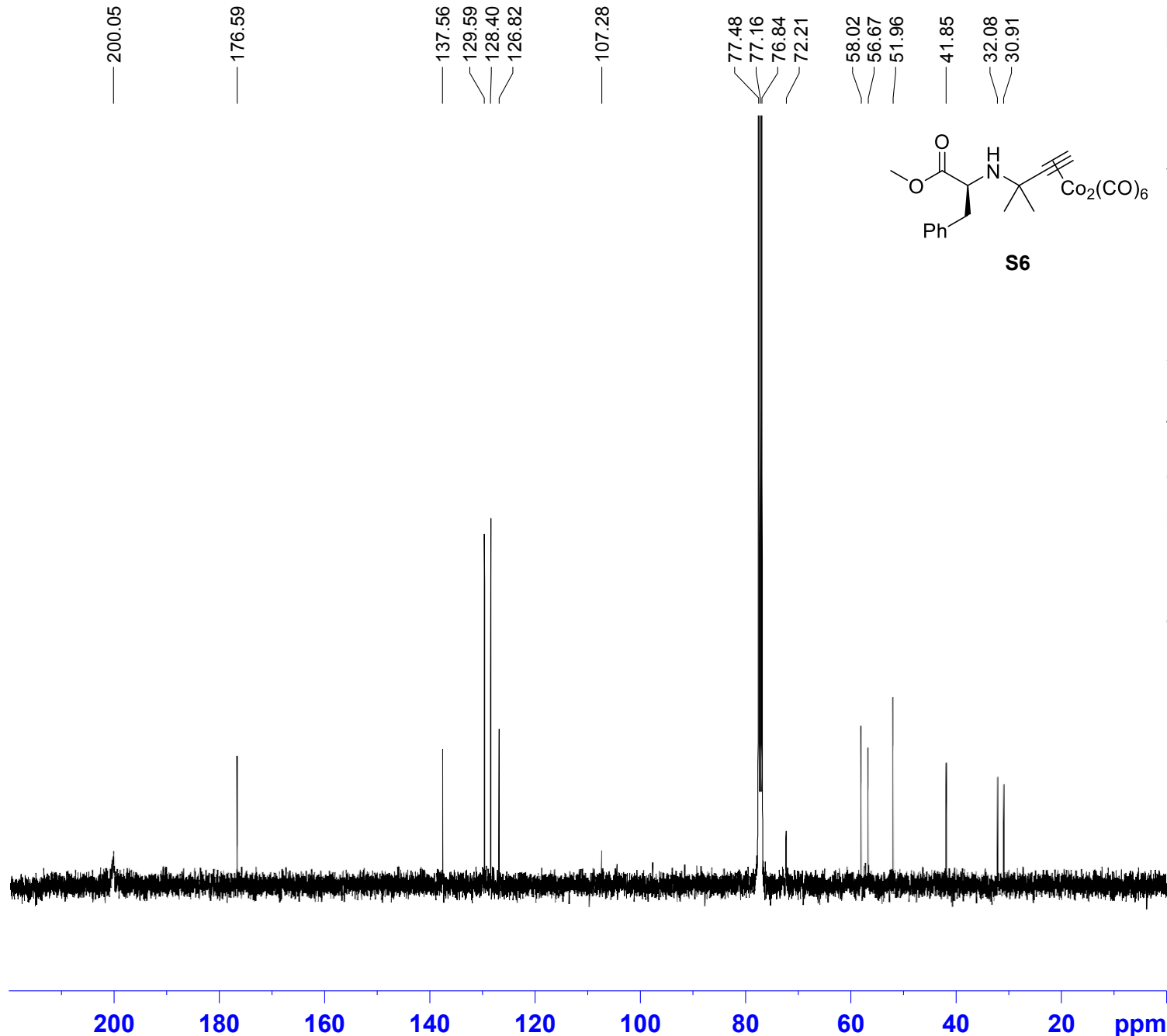
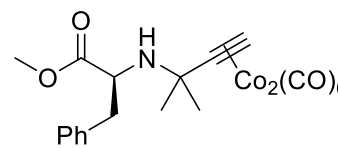


SW07-123-C 13C 400



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

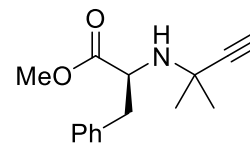
===== CHANNEL f1 =====
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SW07-131-A 1H 400

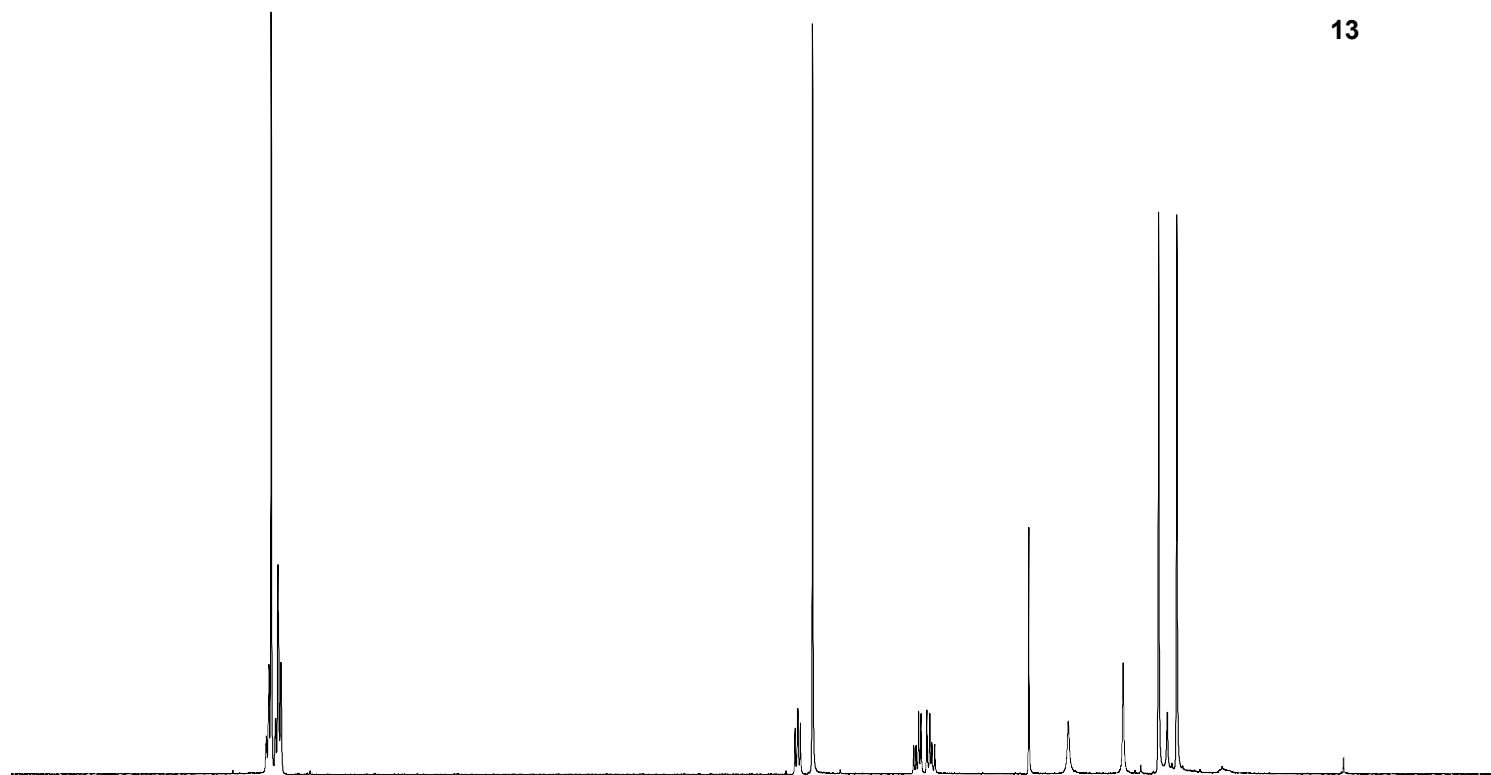
7.296
7.277
7.260
7.231
7.229
7.215
7.195

3.751
3.734
3.731
3.715
3.630
2.951
2.936
2.918
2.902
2.863
2.844
2.830
2.810
2.180
1.916
1.311
1.188



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 CDC13
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



8 7 6 5 4 3 2 1 0 ppm

4.44
2.96

1.00
3.07

1.03
1.08

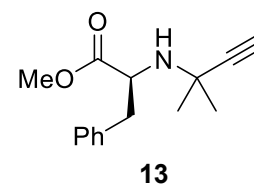
0.94
0.92

3.16
3.21

SW07-131-A 13C 400



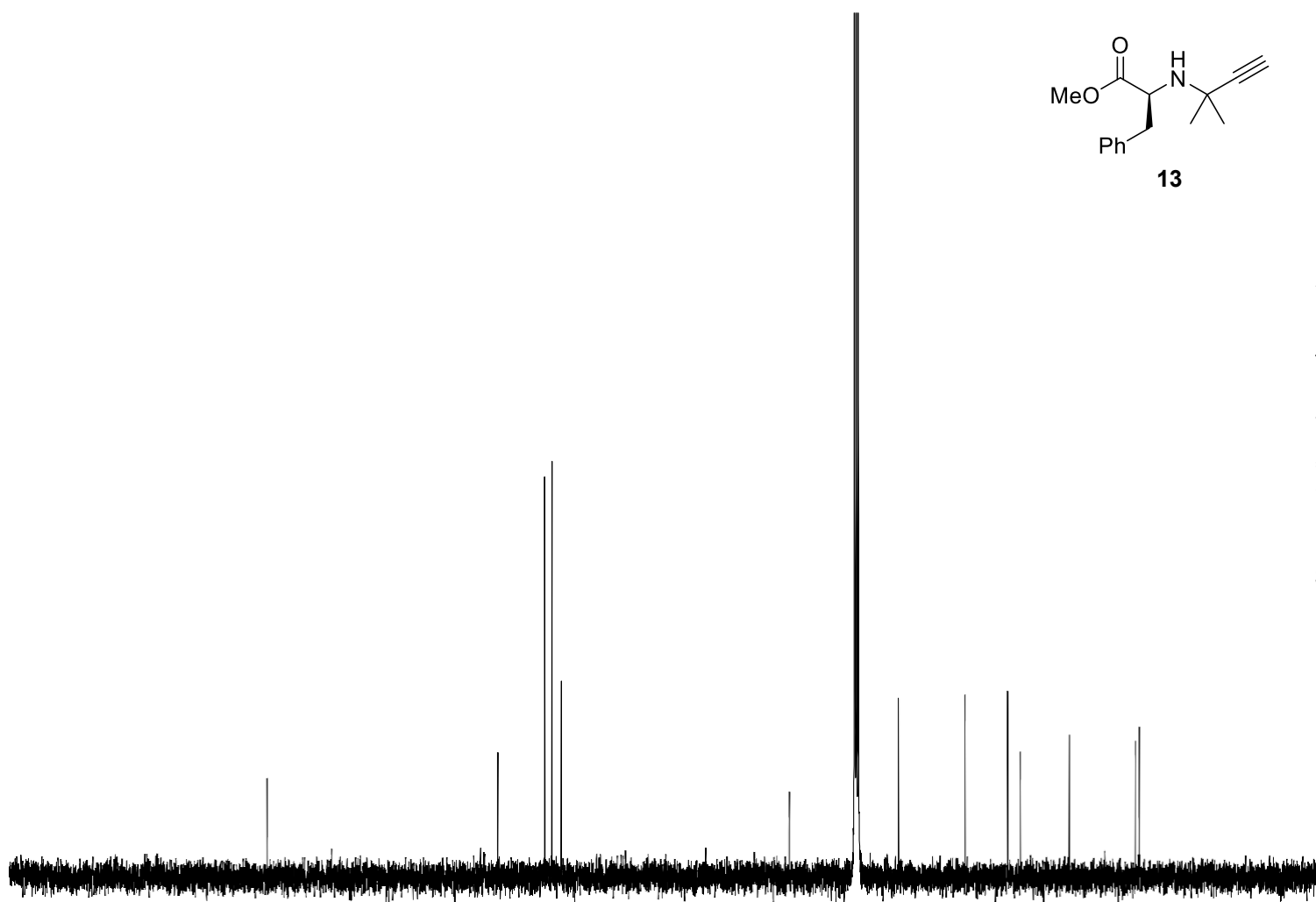
NAME SW07-131-A
EXPNO 11
PROCNO 1
Date_ 20151103
Time 23.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 97.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



176.27

137.52
129.60
128.37
126.79

88.51
77.48
77.16
76.84
70.06
58.87
51.73
49.59
41.37
30.28
29.63



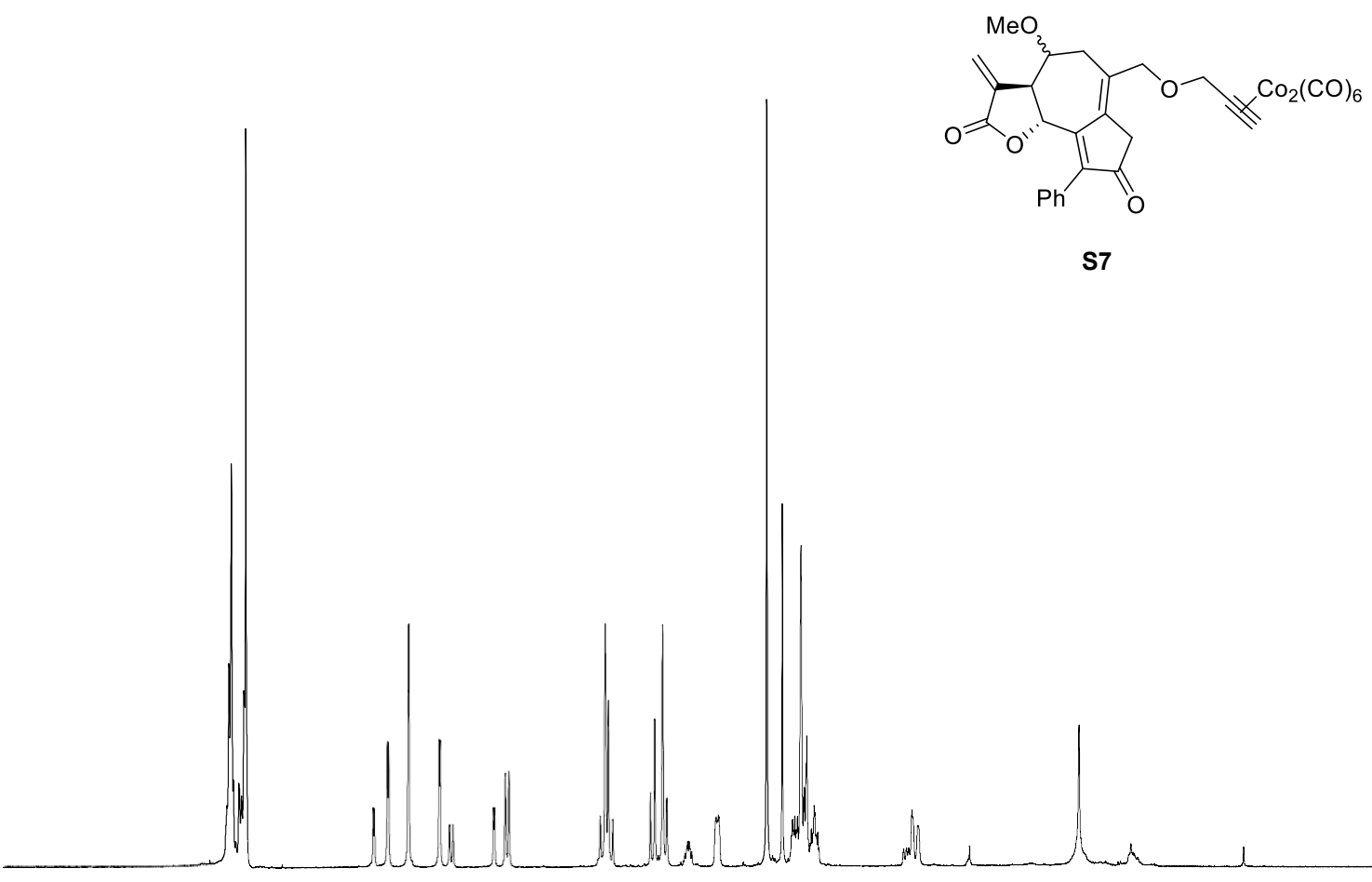
===== CHANNEL f1 =====
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 180 160 140 120 100 80 60 40 20 ppm

SW06-030-C 1H 400

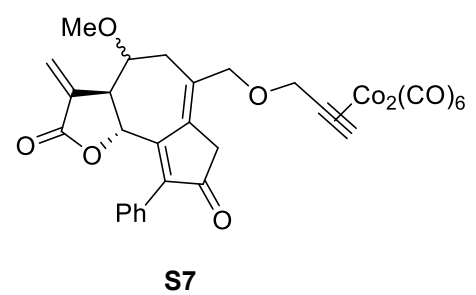


7.395
7.381
7.364
7.349
7.309
7.305
7.290
7.274
7.269
7.260
6.341
6.332
6.239
6.231
6.086
5.868
5.860
5.477
5.469
5.392
5.366
4.708
4.674
4.651
4.348
4.317
4.261
4.229
3.875
3.865
3.860
3.855
3.505
3.392
3.318
3.301
3.282
3.257
3.234
3.216
3.163
3.157
2.458
1.255



8 7 6 5 4 3 2 1 0 ppm

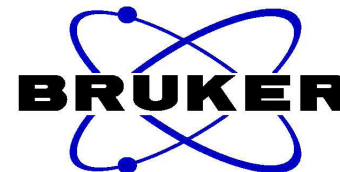
2.99
3.11
0.28
0.62
0.91
0.62
0.29
0.29
0.63
1.93
0.63
1.22
0.36
0.64
1.88
0.86
3.16
0.76
1.00



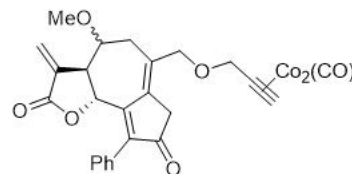
NAME SW06-030-C
EXPNO 20
PROCNO 1
Date_ 20141114
Time 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 94.6 K
D1 1.00000000 sec

===== 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



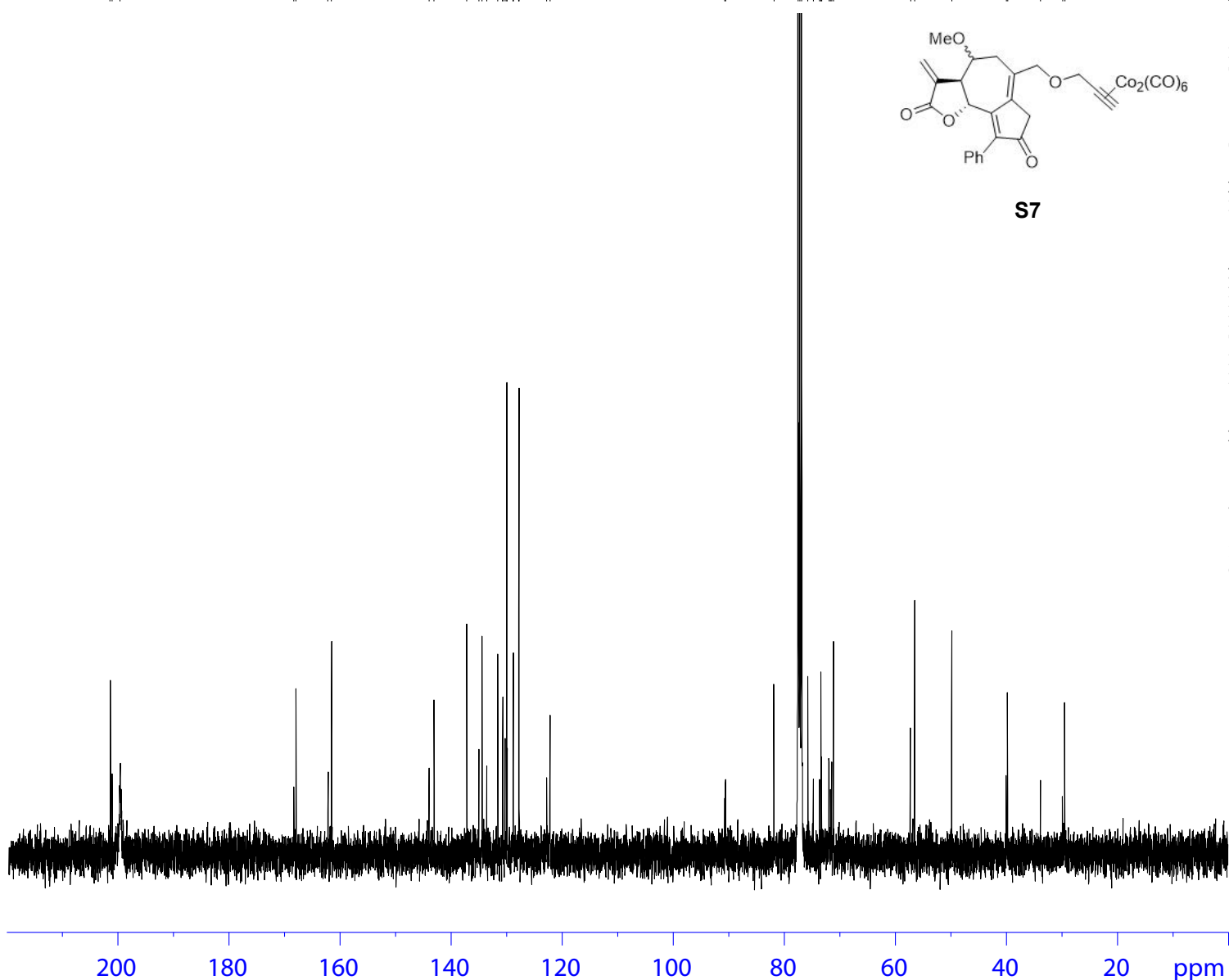
201.37
201.03
199.59
168.30
167.91
162.15
161.52
143.97
143.04
137.16
134.99
134.40
133.58
131.57
130.72
130.65
130.28
129.95
129.90
128.84
128.77
127.79
127.73
122.77
122.17
90.67
90.56
81.84
77.48
77.16
76.84
75.72
74.72
73.62
73.35
73.27
71.91
71.63
71.42
71.09
57.23
56.47
49.80
39.98
39.74
33.80
29.84
29.45



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

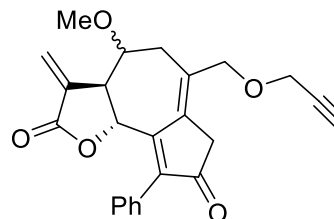
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```



SW06-039-cr 1H 400

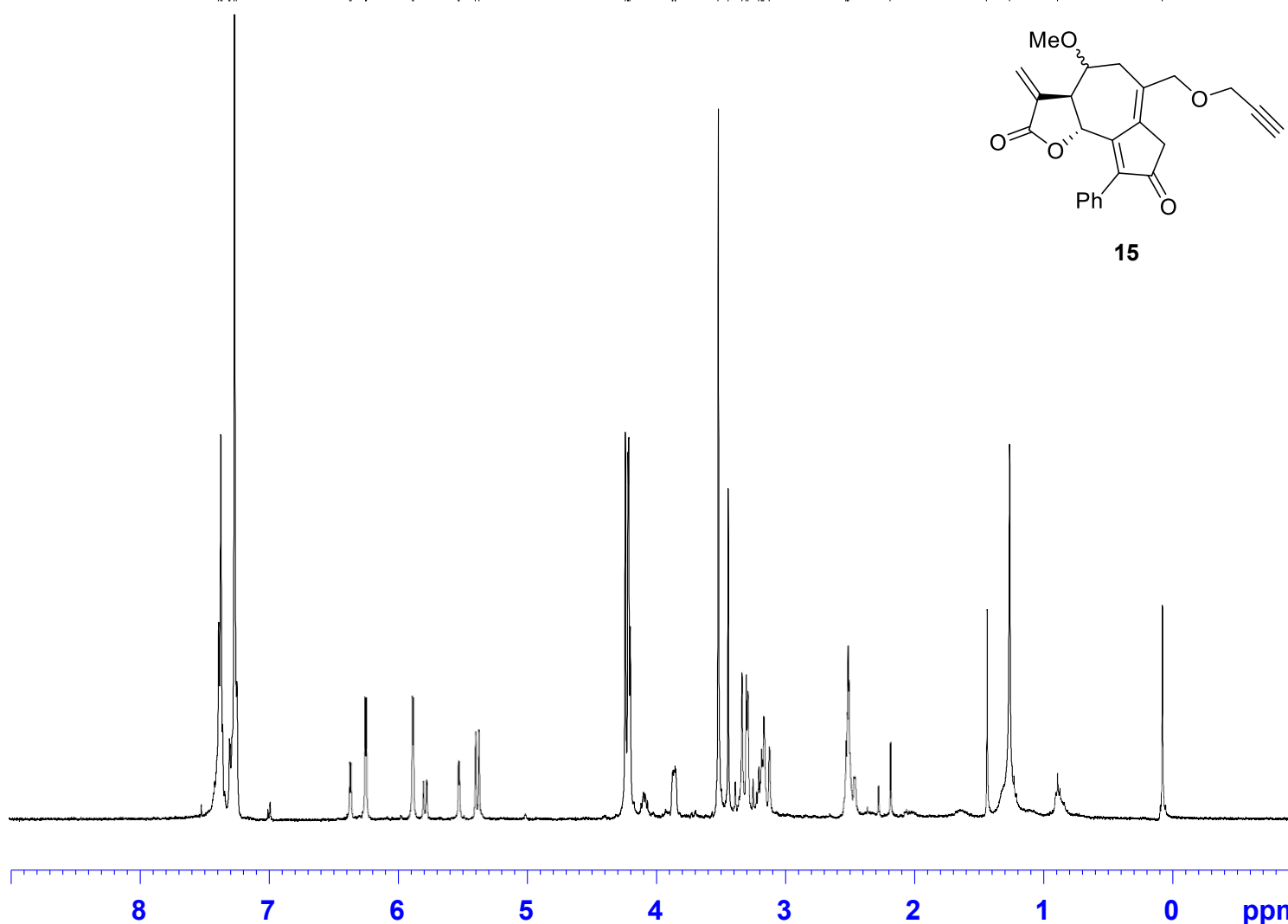


7.383
7.367
7.354
7.300
7.294
7.260
7.242
6.362
6.354
6.244
6.236
5.879
5.872
5.524
5.516
5.391
5.365
4.232
4.211
4.206
4.194
3.865
3.861
3.846
3.841
3.511
3.434
3.327
3.294
3.281
3.197
3.176
3.169
3.158
3.116
2.522
2.512
2.506
2.501
2.178
1.432
1.253
0.880
0.069



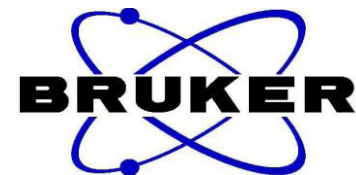
NAME SW06-039-cr
EXPNO 10
PROCNO 1
Date_ 20141117
Time 18.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCI3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 94.1 K
D1 1.00000000 sec

===== 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



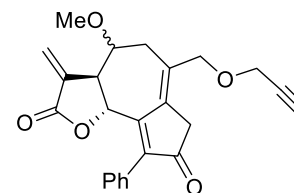
3.21
5.90
0.29
0.64
0.64
0.28
0.30
0.62
3.80
0.40
0.70
2.07
0.98
4.00
1.86
0.10
0.25
0.58
2.46
0.58

SW06-039-cr 13C
600MHz



201.51
201.25

168.29
167.93
161.87
161.38
144.15
143.22
137.17
135.83
135.43
133.60
130.91
130.73
130.65
129.93
129.89
129.68
128.86
128.78
127.80
127.75
122.85
122.23
81.79
79.48
79.34
77.37
77.16
76.95
75.70
75.55
75.41
74.75
73.75
71.93
71.50
67.59
57.83
57.42
57.19
56.46
49.83
49.72
40.02
39.65
34.21
29.97
29.85
29.70
29.67
23.99
22.85
14.27



15

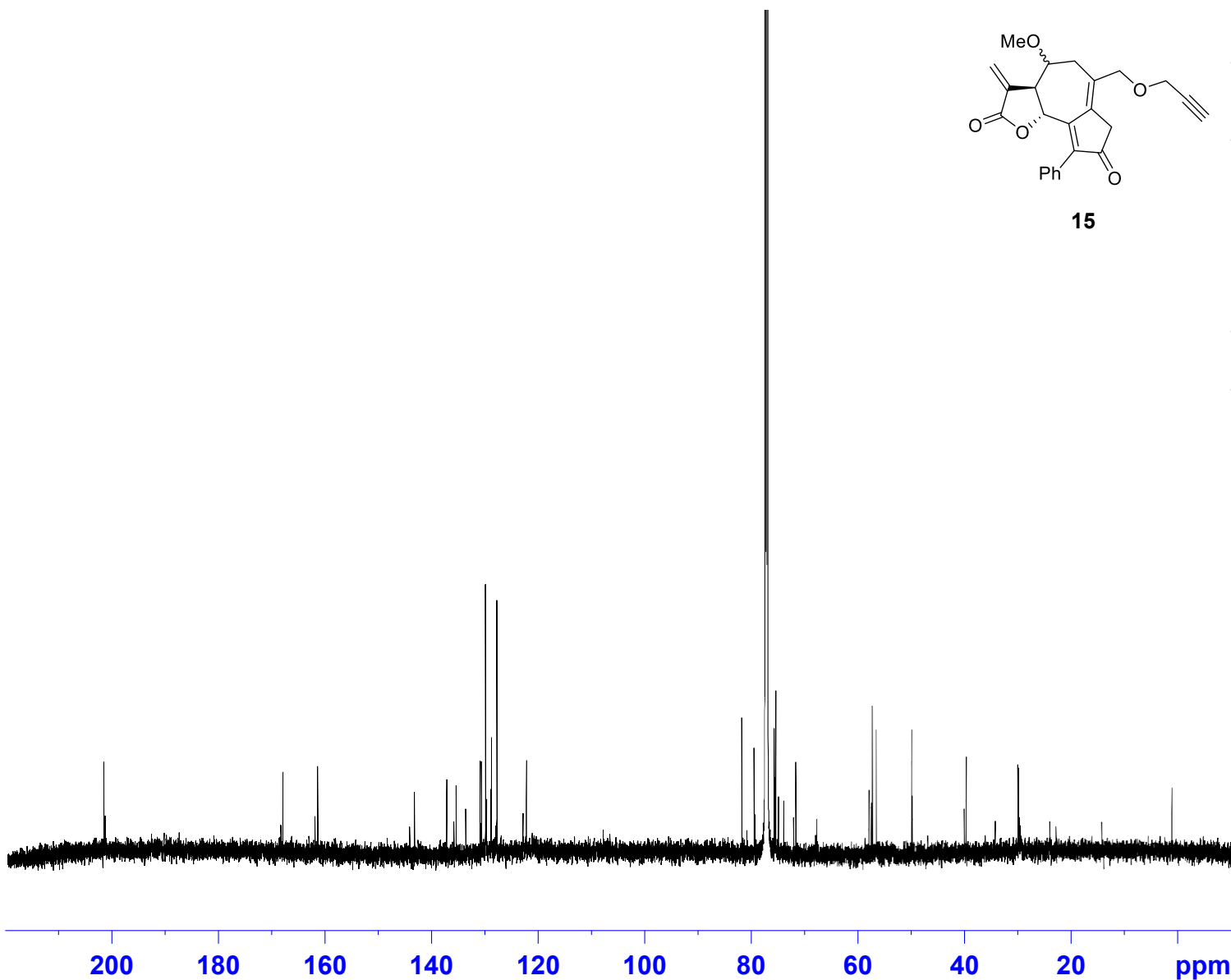
NAME SW06-039-cr dry
EXPNO 2
PROCNO 1
Date_ 20150623
Time 16.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 27093
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867usec
DE 6.50usec
TE 298.2 K
D1 2.00000000sec
D11 0.03000000sec
TD0 1

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

NUC1 13C
P1 11.50usec
PL1 0.00 dB
PL1W 97.46119690W
SFO1 151.0637542 MHz

===== CHANNEL f2=====

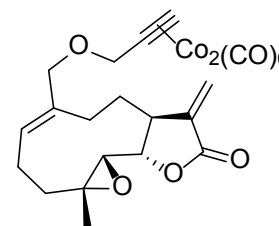
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00usec
PL2 -2.00 dB
PL12 14.19 dB
PL13 120.00 dB
PL2W 19.70630455W
PL12W 0.47381112W
PL13W 0.00000000W
SFO2 600.7124028 MHz
SI 32768
SF 151.0486269 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SW06-183-C 1H 400



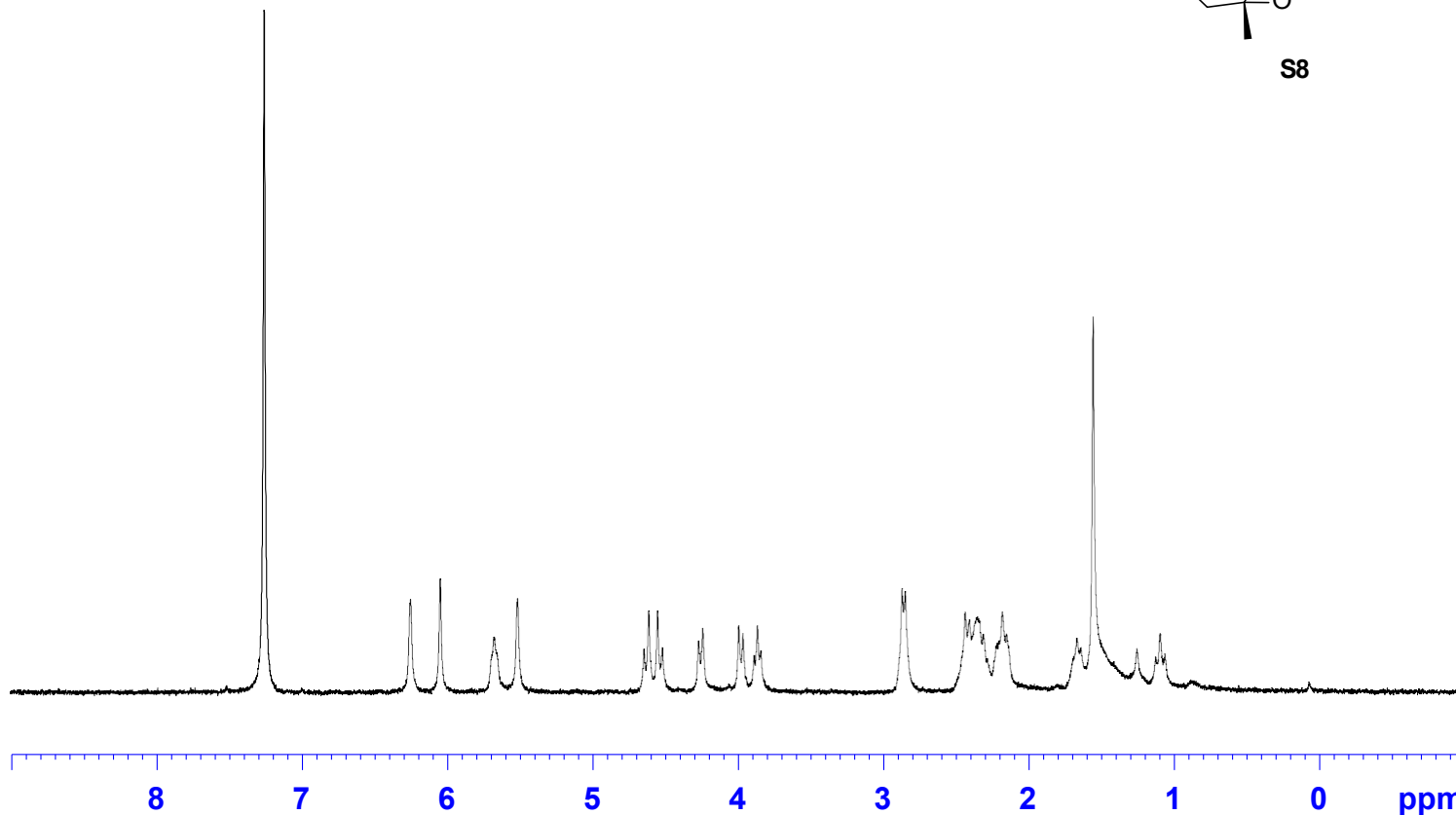
7.260
6.247
6.042
5.670
5.511
4.641
4.608
4.548
4.516
4.265
4.237
3.991
3.961
3.885
3.861
3.837
2.867
2.845
2.432
2.405
2.348
2.309
2.216
2.178
2.150
1.666
1.638
1.554
1.410
1.253
1.124
1.093
1.060



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 CDC13
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

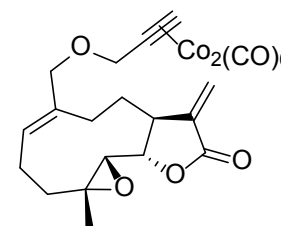


1.02
0.99
1.03
1.00
0.99
1.06
0.99
1.01
1.07
2.00
4.15
2.42
1.34
3.82
0.87
1.44

SW06-183-C 1H 400

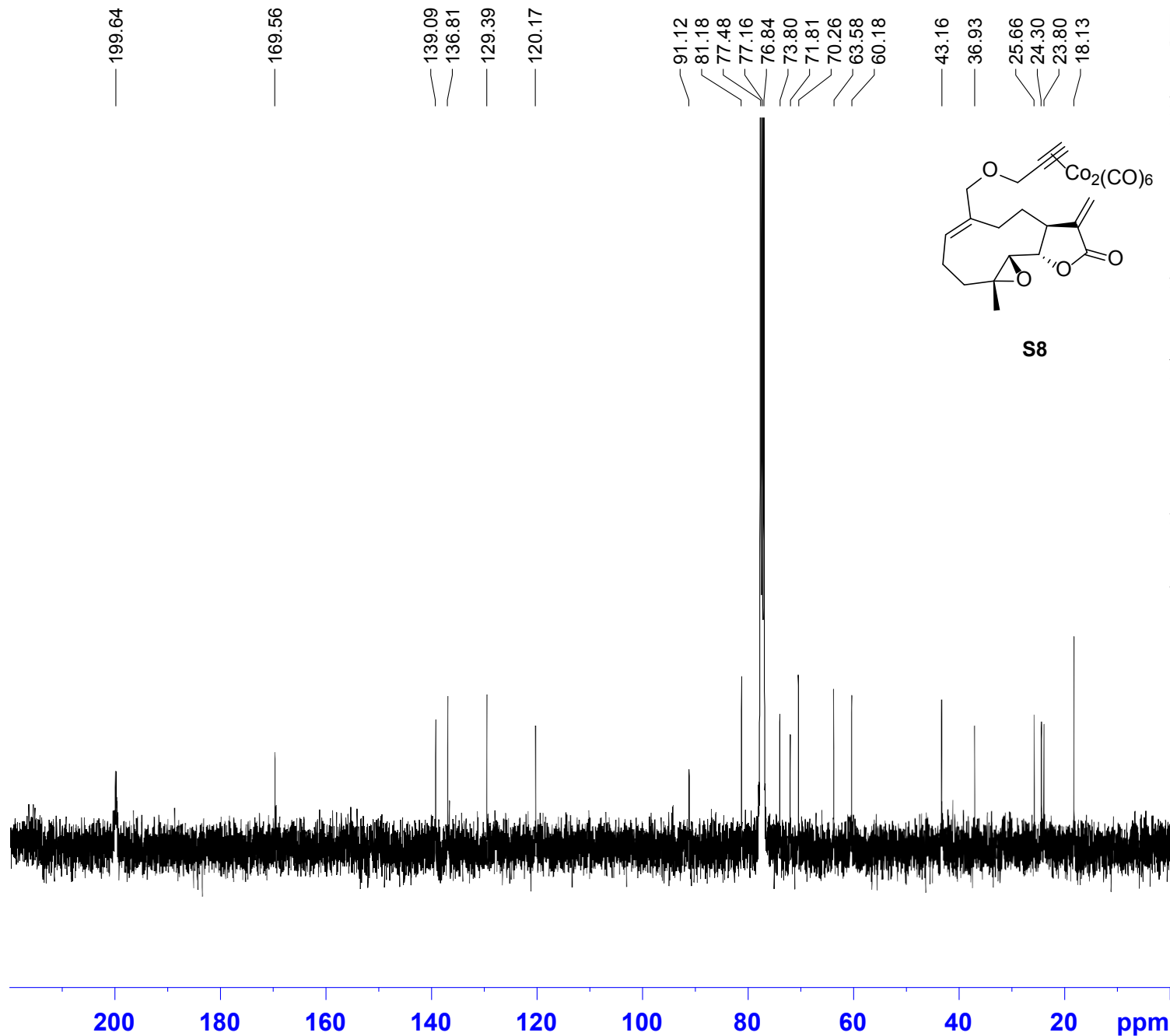


NAME SW06-183-C
EXPNO 11
PROCNO 1
Date_ 20150505
Time_ 7.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 95.3 K
D1 2.00000000 sec
D11 0.03000000 sec



S8

===== 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

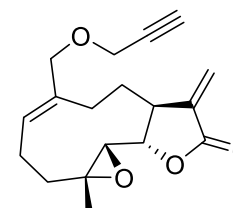


SW06-191-cr 1H 400

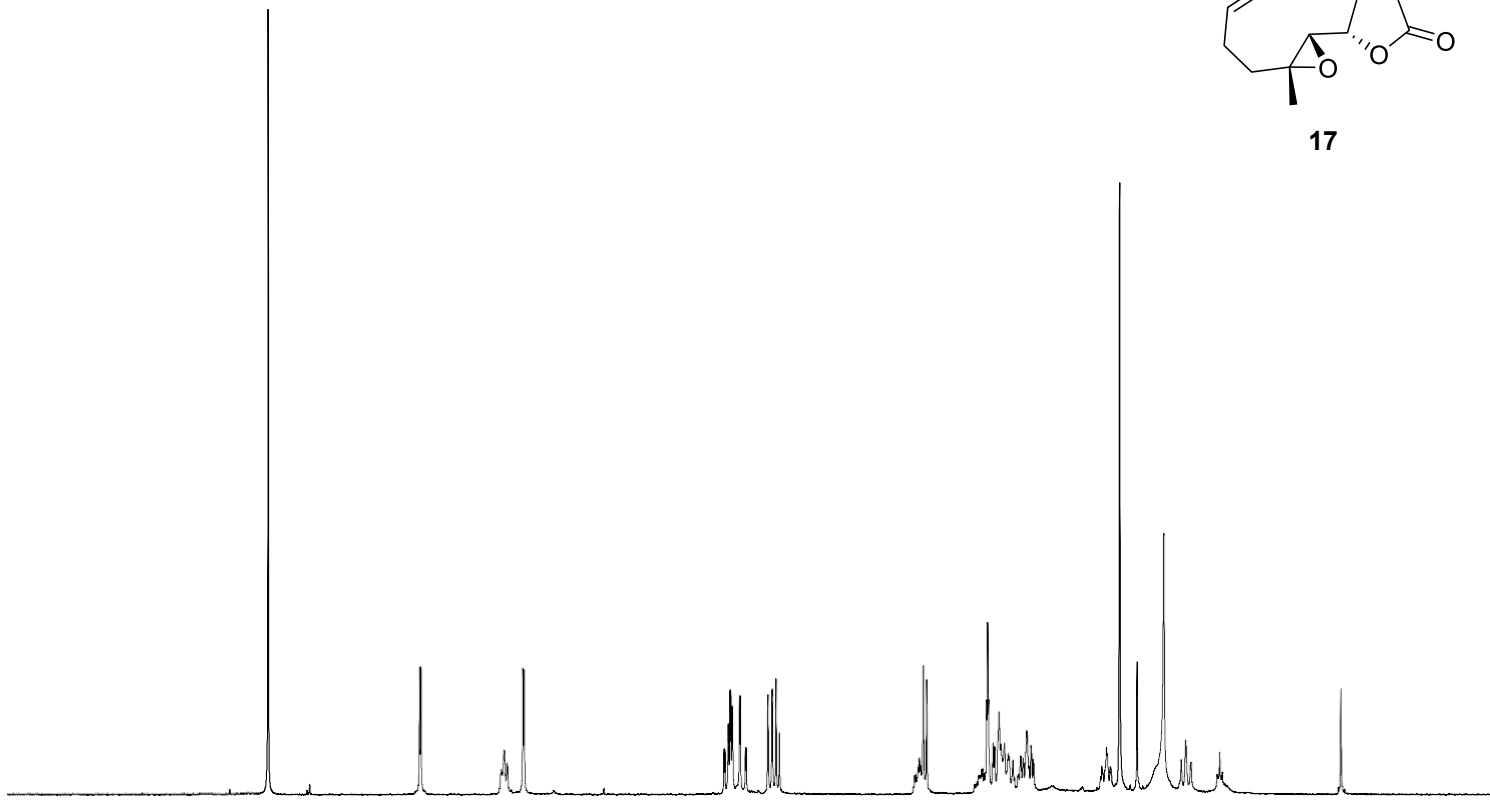


7.260
6.242
6.233
5.676
5.551
5.543
4.205
4.199
4.176
4.165
4.159
4.149
4.101
4.096
4.062
4.056
3.911
3.882
3.857
3.834
2.872
2.848
2.447
2.441
2.435
2.404
2.394
2.364
2.351
2.330
2.303
2.296
2.219
2.179
2.175
2.151
2.148
1.645
1.640
1.552
1.430
1.252
1.105
0.878

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



17



===== 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

8 7 6 5 4 3 2 1 0 ppm

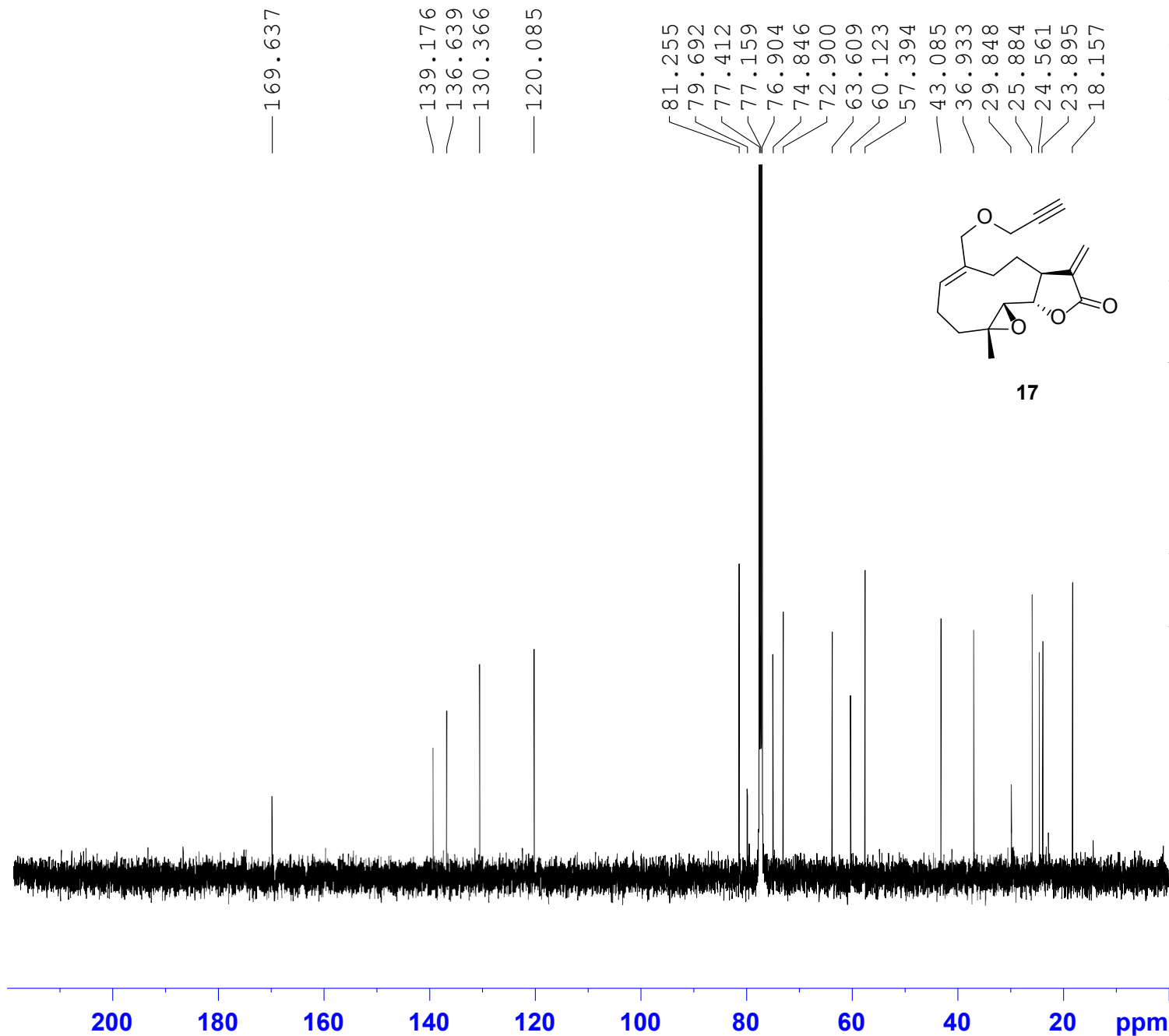
1.00
0.97
0.99
1.89
1.09
1.95
1.95
2.12
3.35
2.31
1.44
3.02
1.38

SW06-191-cr 13C 500



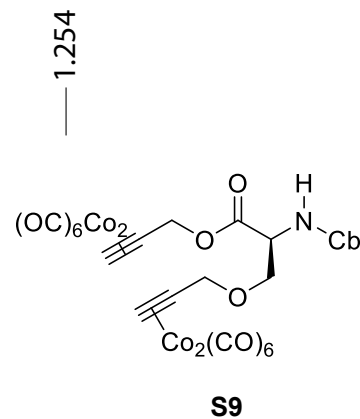
NAME SW06-191-cr
EXPNO 10
PROCNO 1
Date_ 20150513
Time_ 6.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== 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



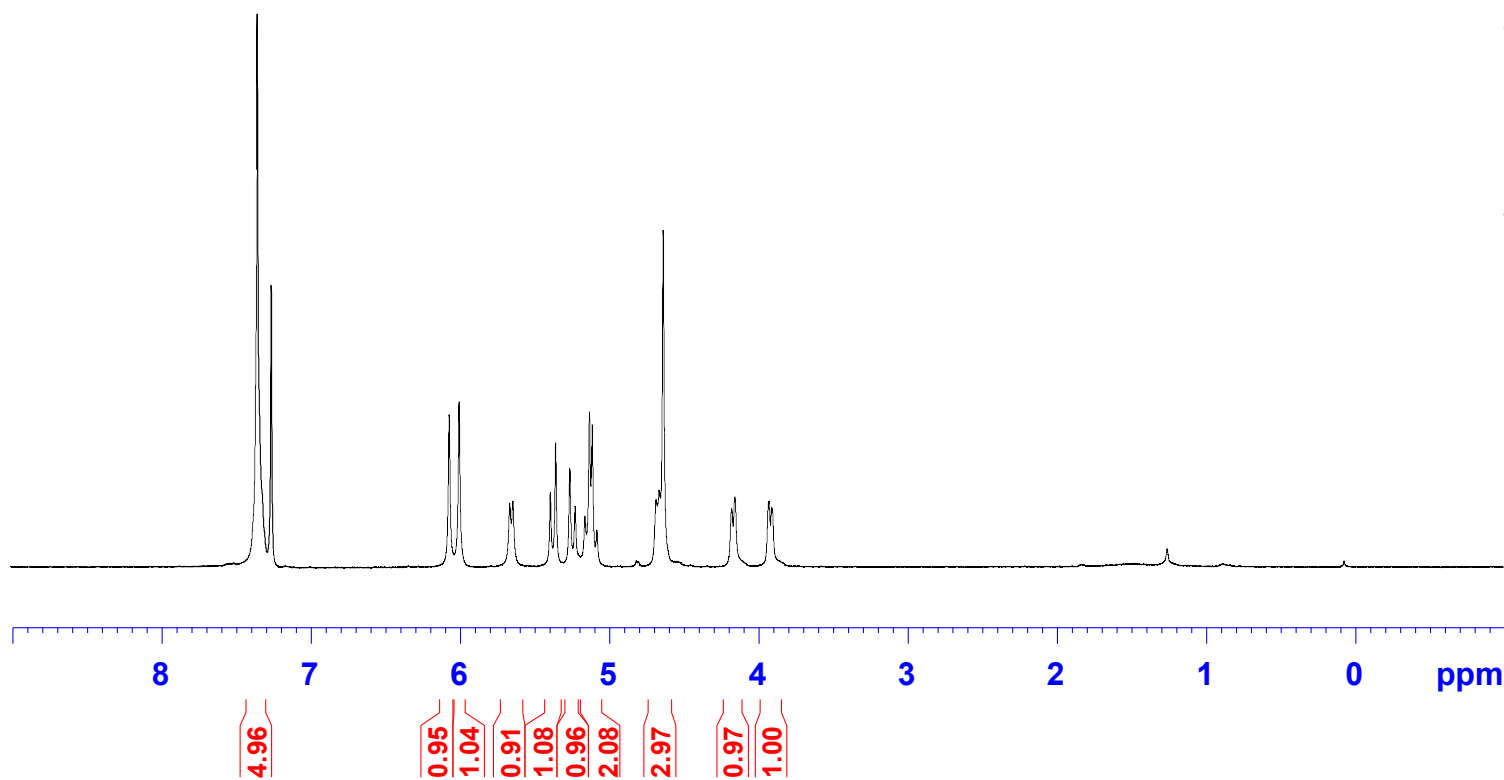
SW06-158-B 1H 400

7.354
7.260
6.066
5.999
5.660
5.639
5.388
5.352
5.258
5.222
5.157
5.126
5.107
5.076
4.680
4.658
4.632
4.173
4.152
3.923
3.905



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	CDC13
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

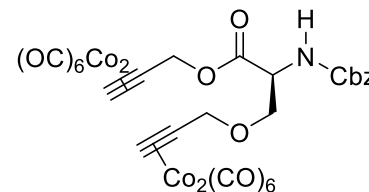


SW06-158-B 13C 400



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 2.0000000 sec
D11 0.0300000 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



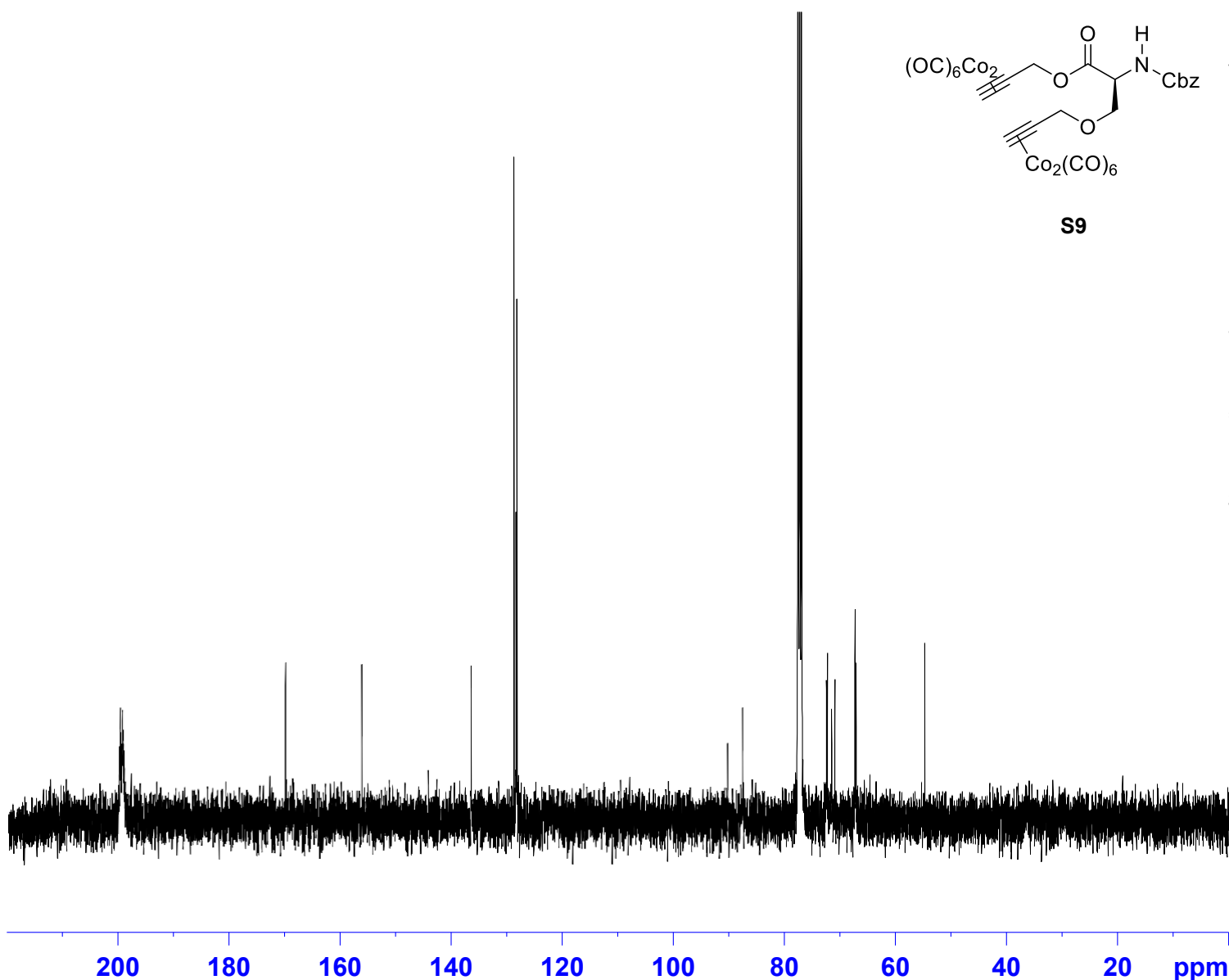
90.21
87.52
77.48
77.16
76.84
72.31
72.12
71.38
70.78
67.18
66.96
54.63

199.50
199.08

169.78

156.05

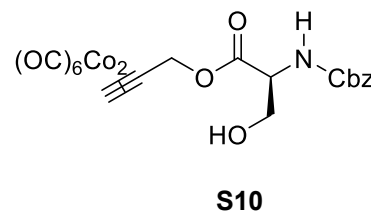
136.39
128.65
128.28
128.12



SW06-202-C 1H 400



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

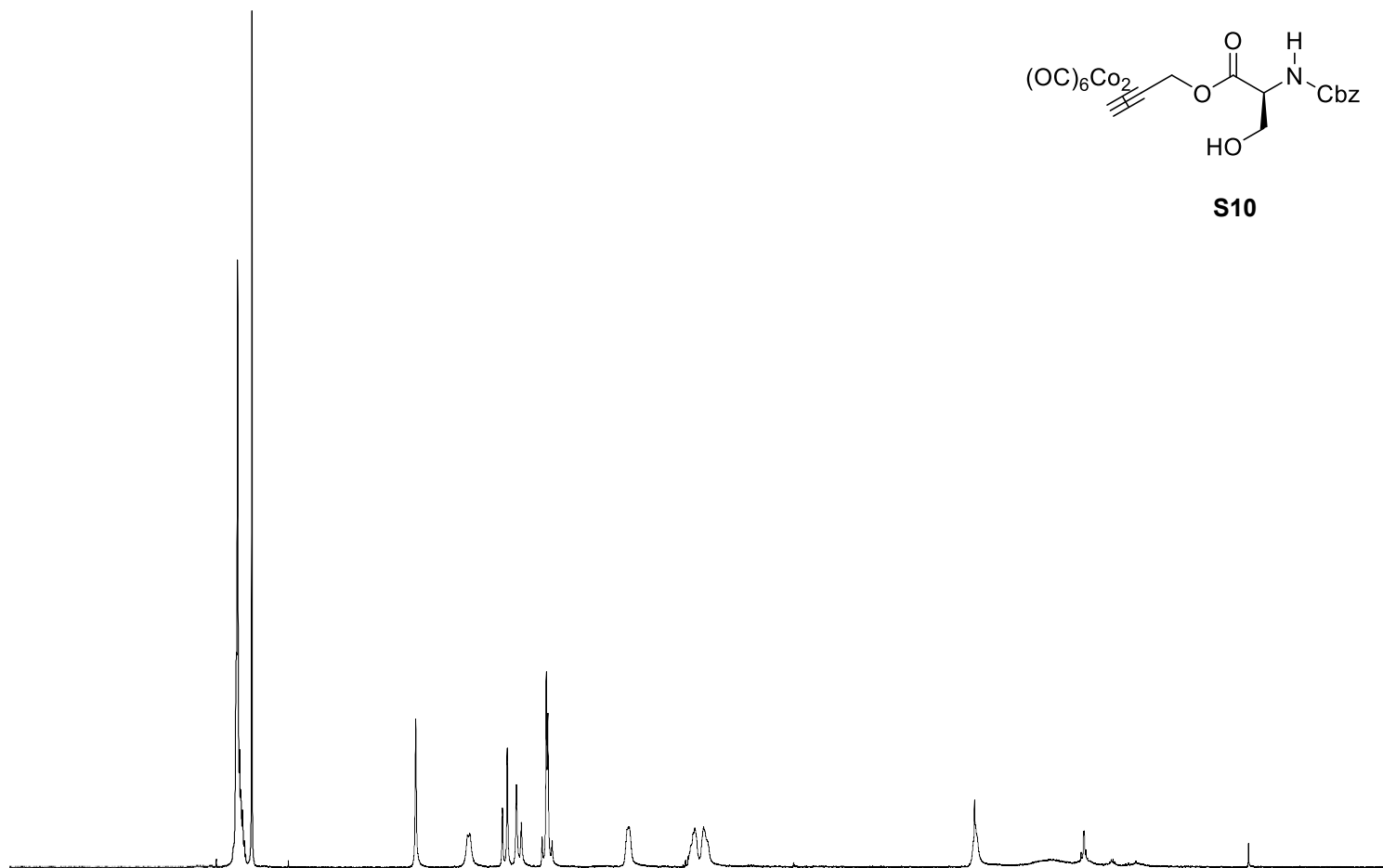


7.372
7.363
7.348
7.342
7.327
7.260
6.077
5.703
5.687
5.451
5.416
5.350
5.314
5.165
5.135
5.123
5.093
4.543
4.535
4.064
4.000

2.045

1.259
1.254

0.069

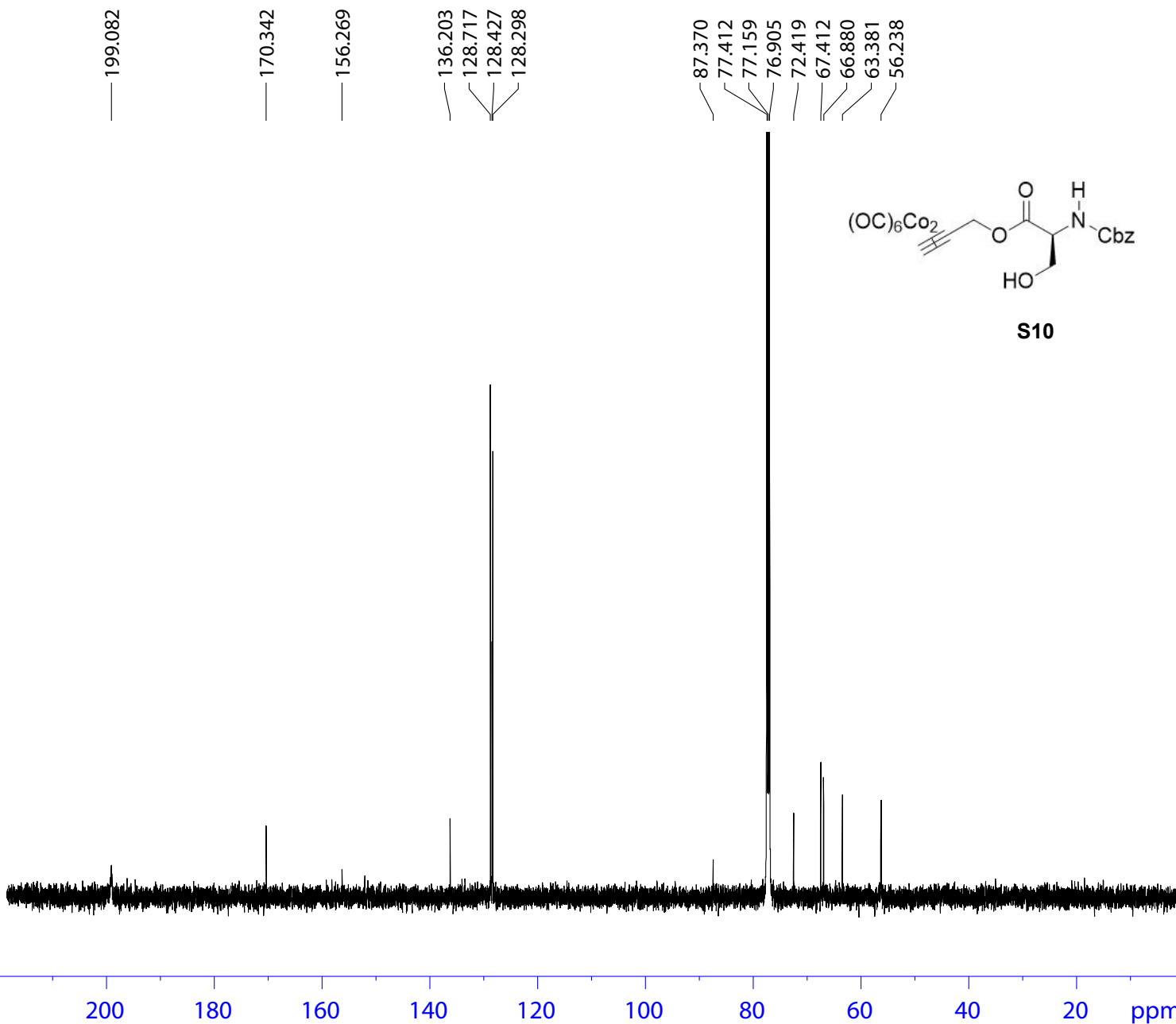
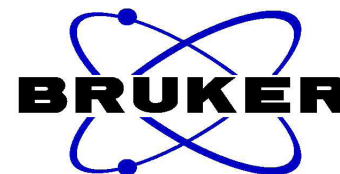


===== CHANNEL f1 =====
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

8 7 6 5 4 3 2 1 0 ppm

5.03
0.97
0.86
1.00
1.04
2.07
0.99
0.96
1.10
1.05
0.59

SW06-202-C 13C 500



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

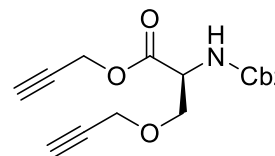
==== CHANNEL f1 =====
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653131 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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

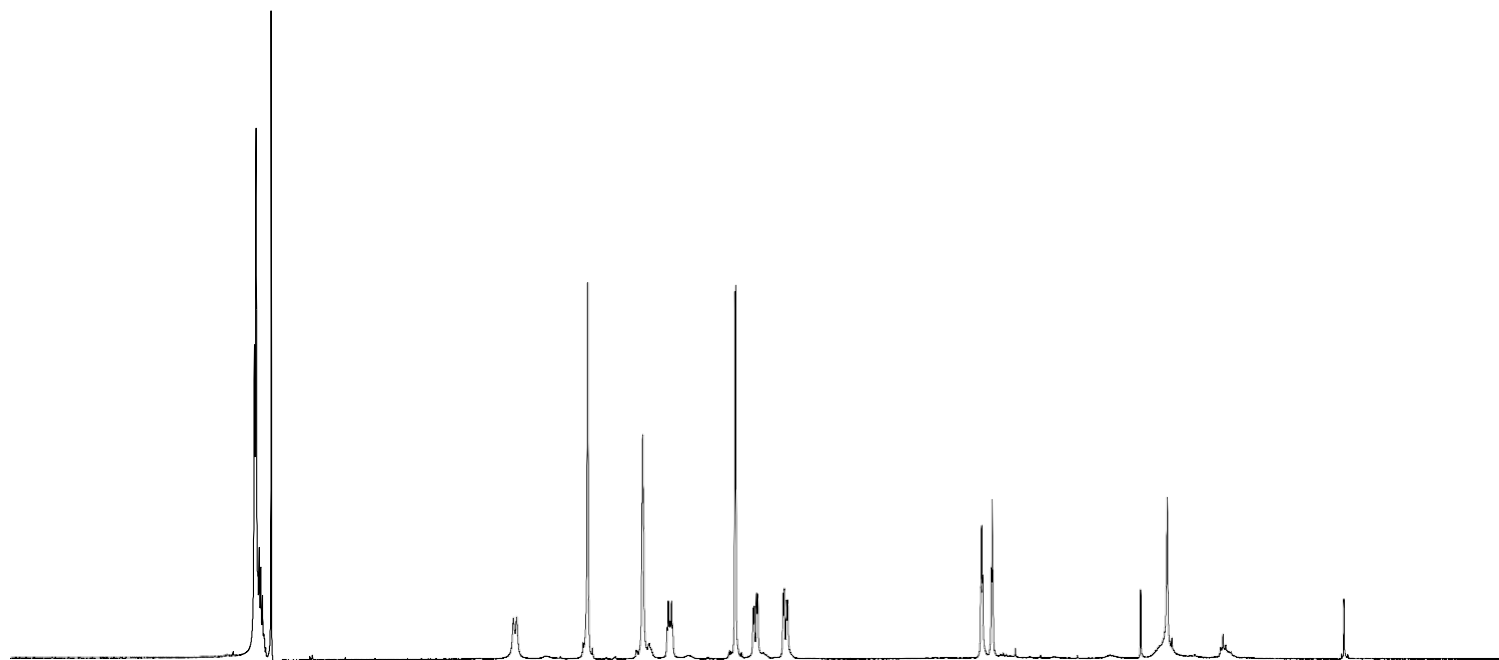


NAME SW06-196-cr
EXPNO 20
PROCNO 1
Date_ 20150515
Time 9.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec



S11

===== 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



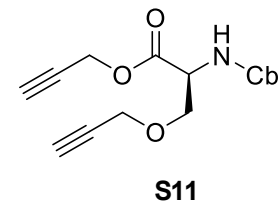
8 7 6 5 4 3 2 1 0 ppm

5.17
0.87
2.08
2.00
0.92
2.03
1.03
1.04
0.94
1.06
1.89
0.52

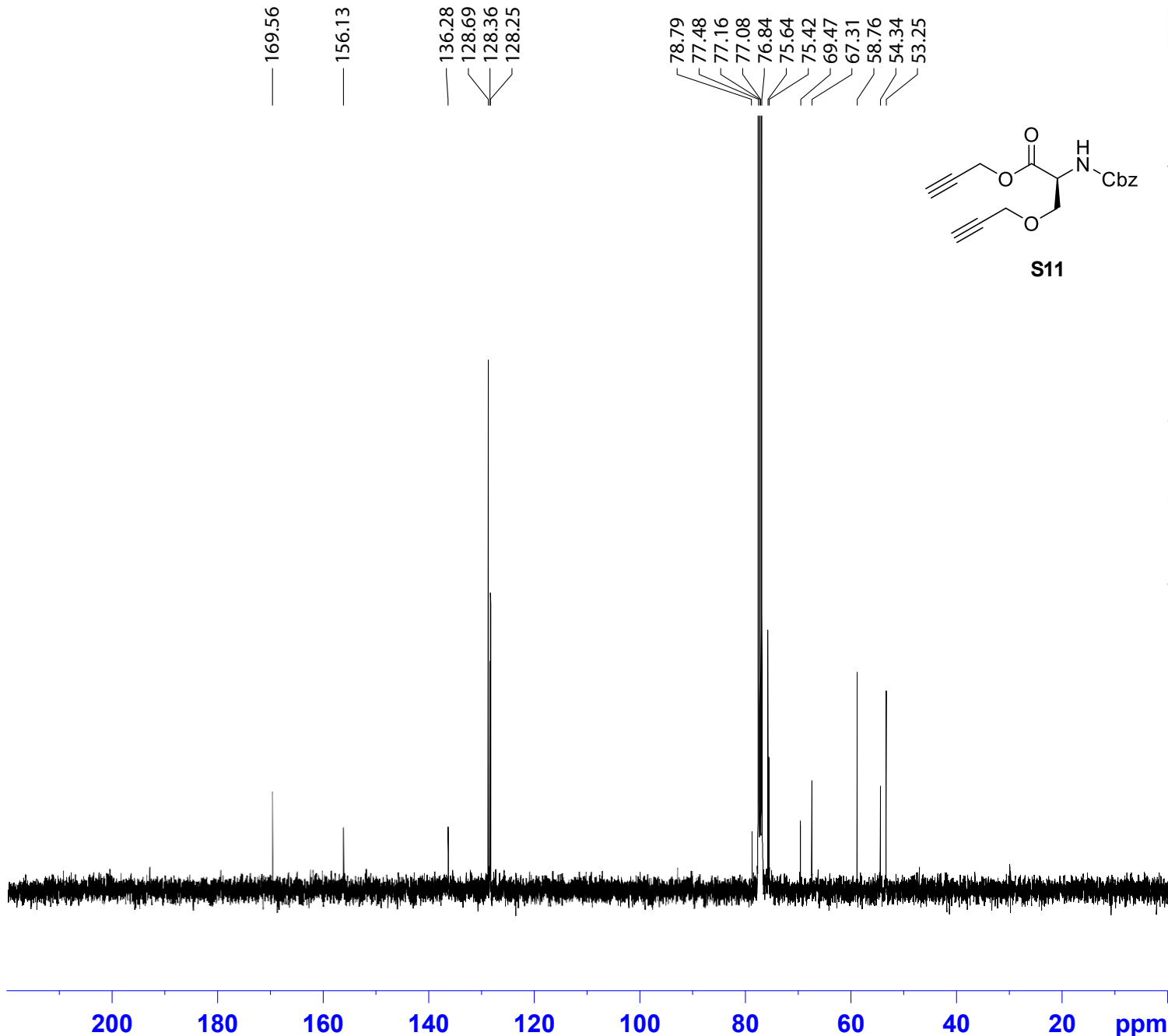
SW06-196-cr



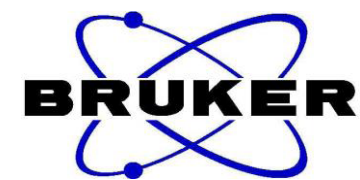
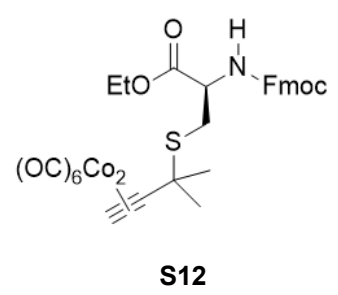
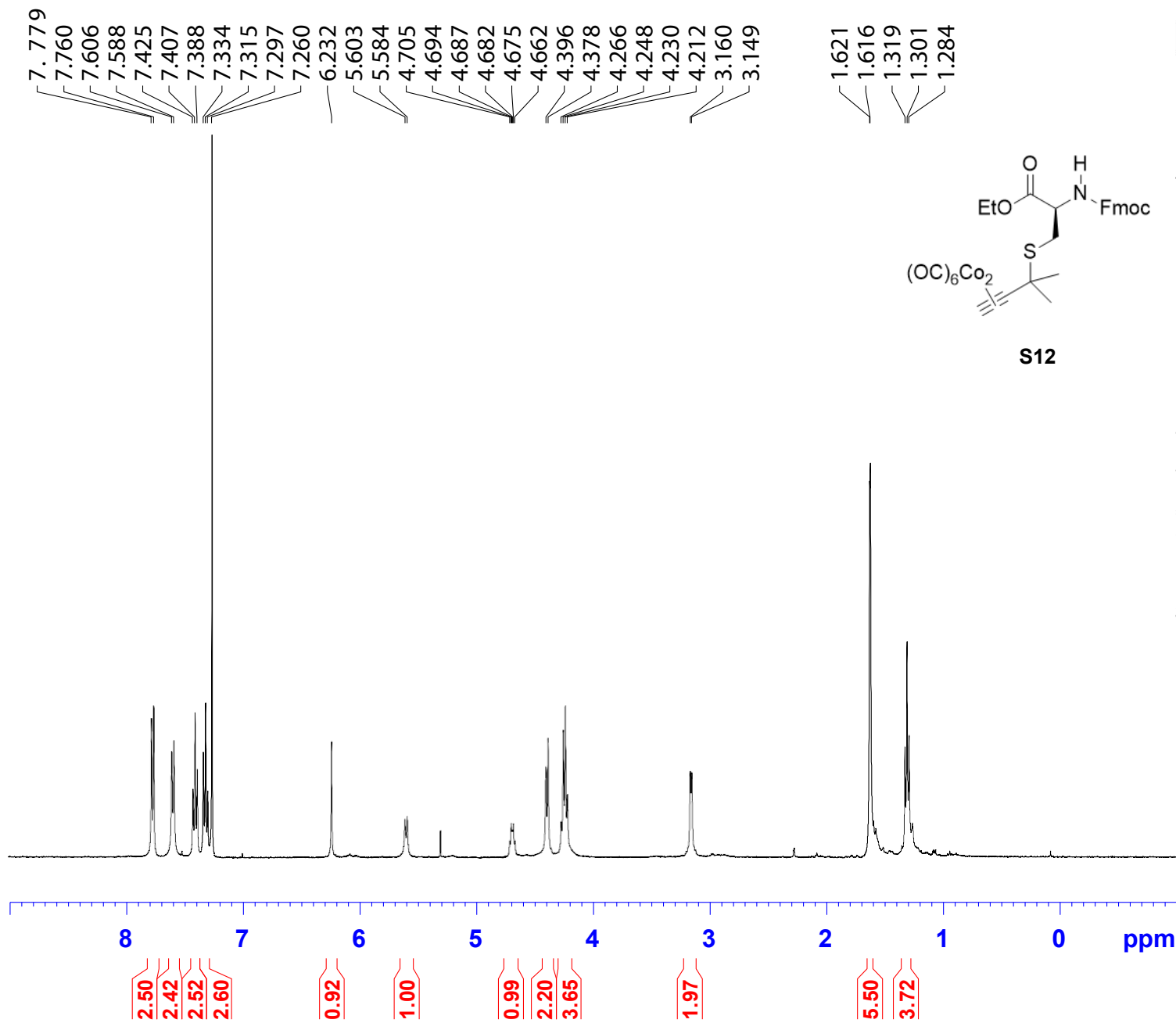
NAME SW06-196-cr
EXPNO 11
PROCNO 1
Date_ 20150515
Time 6.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 128
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.0000000 sec
D11 0.0300000 sec



===== 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



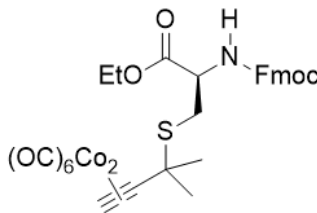
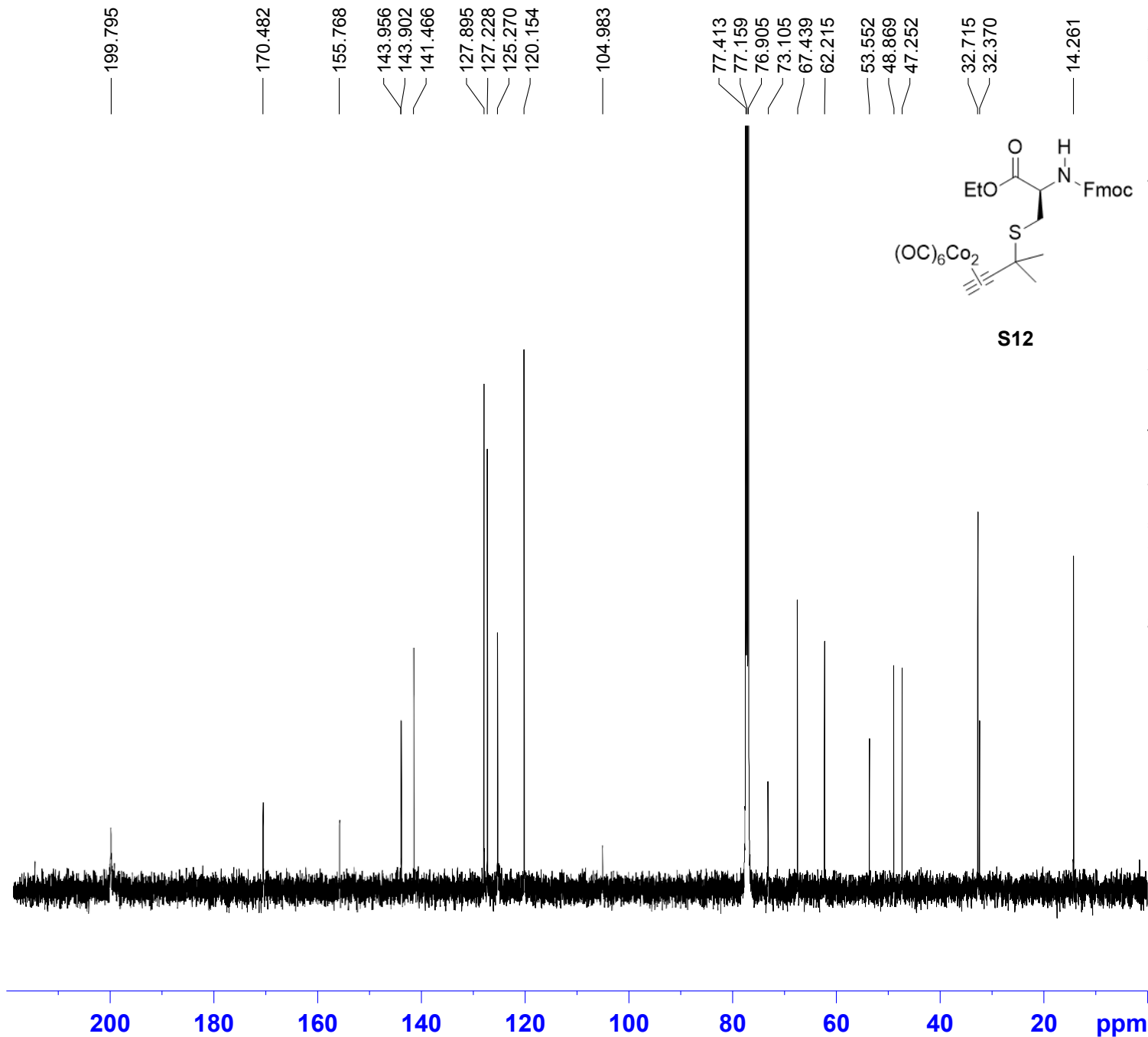
SW07-141-B



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

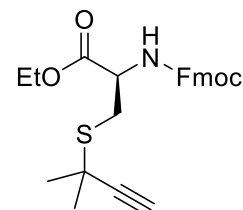
===== CHANNEL f1 =====
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653128 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SW07-147-A 1H 400

7.757
7.617
7.599
7.421
7.403
7.384
7.335
7.317
7.298
7.259
5.656
5.636
4.712
4.699
4.692
4.686
4.679
4.666
4.437
4.410
4.405
4.392
4.271
4.253
4.241
4.235
4.223
4.218
3.238
3.229
3.224
2.366
1.566
1.320
1.302
1.285

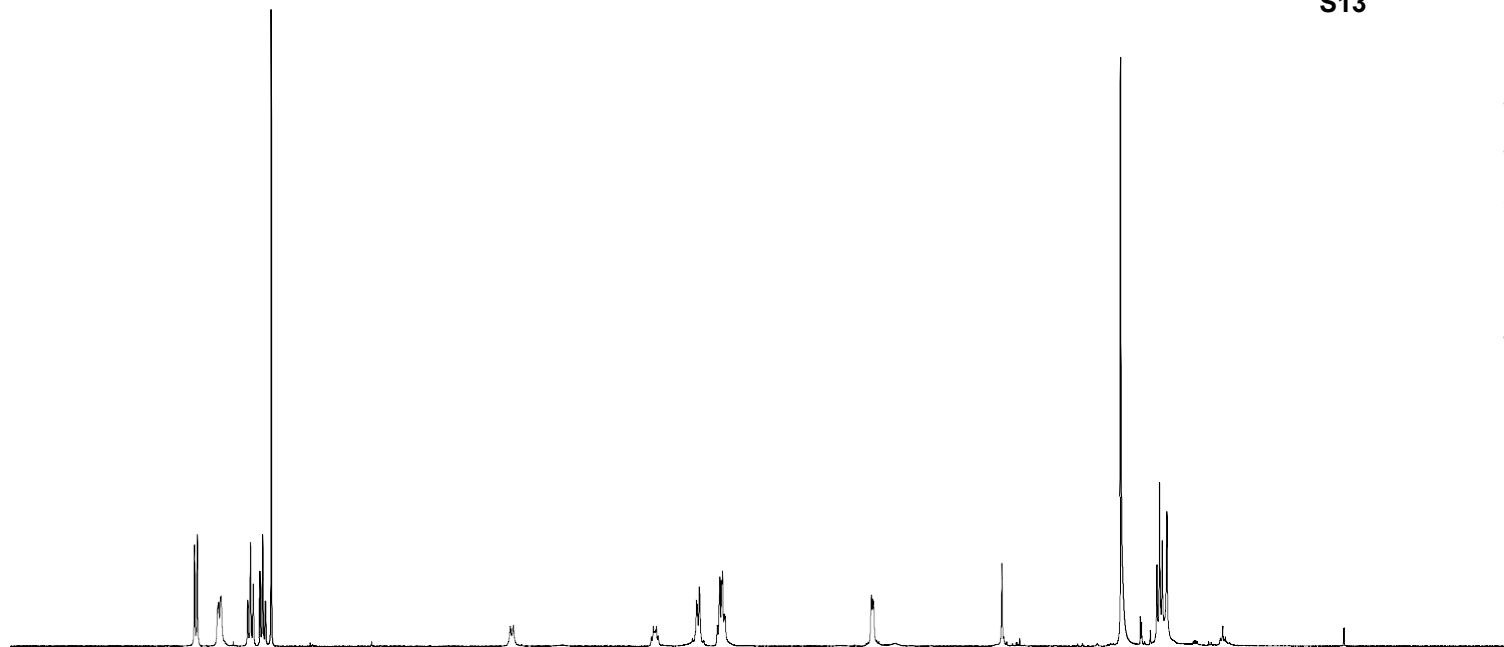


NAME SW07-147-A
EXPNO 10
PROCNO 1
Date_ 20151214
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 144
DW 62.400 usec
DE 6.50 usec
TE 96.5 K
D1 1.00000000 sec
TD0 1



S13

===== 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



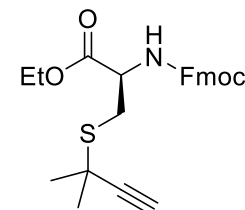
8 7 6 5 4 3 2 1 0 ppm

2.08
2.01
2.12
2.10
0.85
0.85
2.00
3.11
1.78
0.86
6.64
3.63

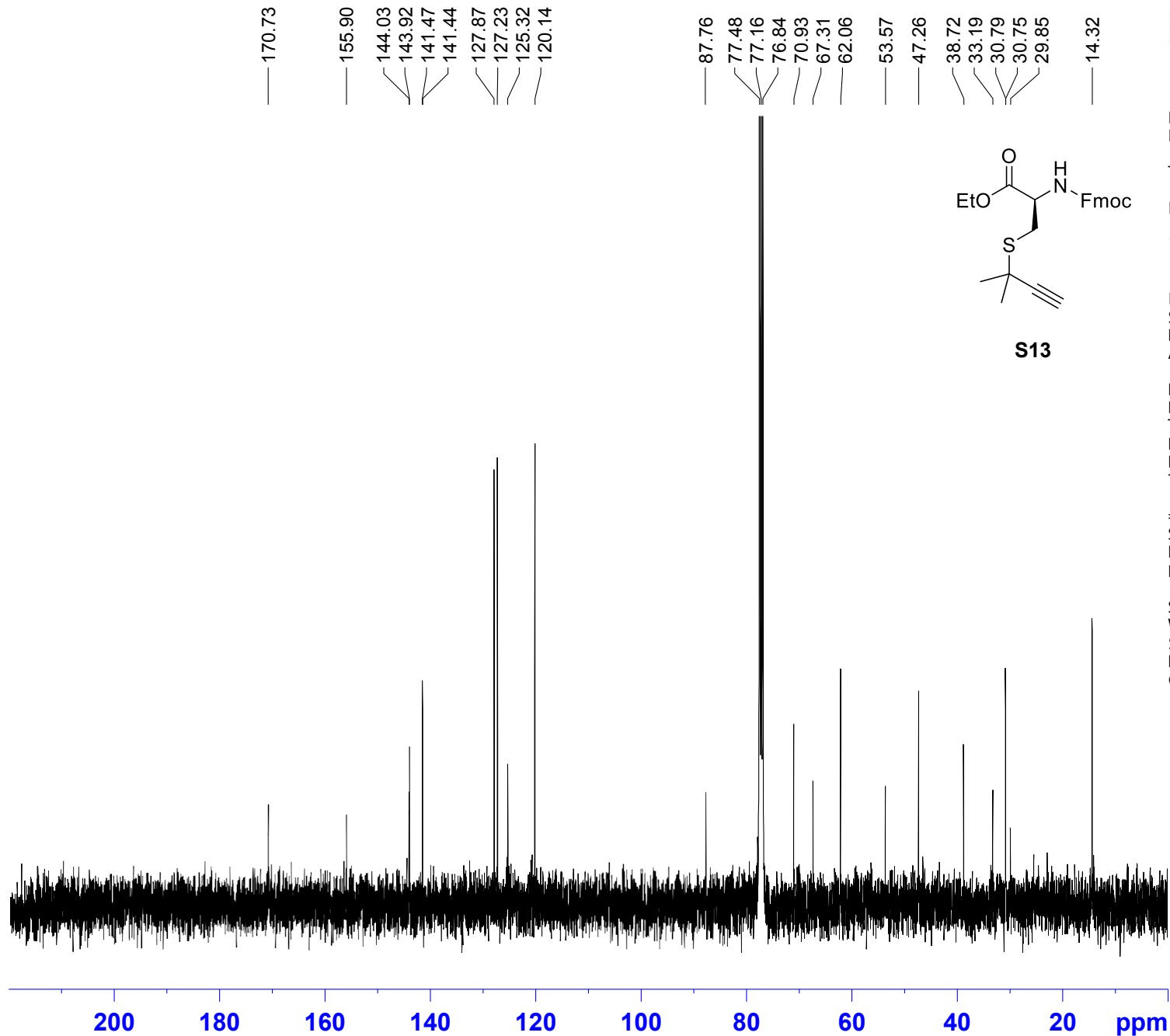
SW07-147-A 13C 400



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



S13



===== 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