Enantioselective Alkynylation of Trifluoromethyl Ketones Catalyzed By Cation-Binding Salen Nickel Complexes.

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Materials and Methods

Unless otherwise stated, all reactions were carried out under air atmosphere. Reaction progress was monitored by thin-layer chromatography (TLC) or Agilent 1290 U HPLC-MS. TLC was performed using E. Merck silica gel 60 F254 precoated glass plates (0.25 mm) and visualized by UV fluorescence quenching, *p*-anisaldehyde, or $KMnO_4$ staining. Silicycle Silia*Flash*® P60 Academic Silica gel (particle size 40–63 nm) or 230-400 Mesh 60 Å Silica Gel (Merck Inc.). was used for flash chromatography. All alkynylation reactions were performed in 10 ml vial sealed with a screw cap. At the Gwangju Institute of Science and Technology (GIST) all NMR spectra was recorded on a JEOL spectrometer, operating at 400 MHz or 300 MHz for ^{1}H NMR and at 100 MHz or 75 MHz for ^{13}C NMR. At the California Institute of Technology (Caltech), ^{1}H NMR spectra were recorded on Bruker 400 MHz or Varian Mercury 300 MHz spectrometers. ^{13}C NMR spectra were recorded on Bruker 400 MHz spectrometer (101 MHz). ^{19}F NMR spectra were recorded on Varian Mercury 300 MHz spectrometer (282 MHz). Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm) (multiplicity, coupling constant

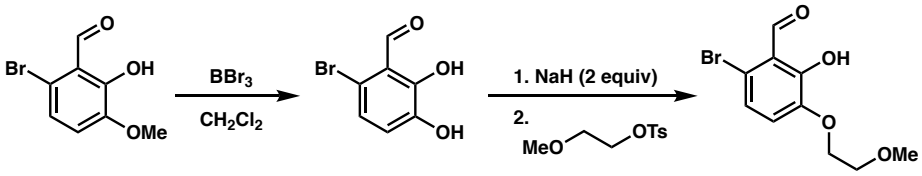
(Hz), integration). Multiplicities are reported as follows: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, sept = septuplet, m = multiplet, br s = broad singlet, br d = broad doublet, app = apparent. Data for ^{13}C NMR are reported in terms of chemical shifts (δ ppm). All chemical shifts for ^{1}H and ^{13}C NMR were referenced to residual signals from $CDCl_3$ (^{1}H) 7.26 ppm and (^{13}C) 77.16 ppm. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-700 MStation mass spectrometer. At the GIST, Infrared (IR) spectra were obtained on a Nicolet iS10 FT-IR spectrometer with an ATR unit and recorded in wave numbers (cm^{-1}). At Caltech, the IR spectra were obtained using Perkin Elmer Spectrum BXII spectrometer or Nicolet 6700 FTIR spectrometer using thin films deposited on NaCl plates and reported in frequency of absorption (cm^{-1}) High-performance liquid chromatography (HPLC) was performed on an Agilent 1260 Infinity Series machine equipped with a variable wavelength detector and Daicel Chiralpak I Series columns (0.46 cm $_X$ 25 cm). Analytical SFC was performed with a Mettler SFC supercritical CO_2 analytical chromatography system utilizing Chiralpak (AD-H, AS-H or IC) or Chiralcel (OD-H, OJ-H, or OB-H) columns (4.6 mm x 25 cm) obtained from Daicel Chemical Industries, Ltd. High resolution mass spectra (HRMS) were obtained from Agilent 6200 Series TOF with an Agilent G1978A Multimode source in electrospray ionization (ESI+), atmospheric pressure chemical ionization (APCI+), or mixed ionization mode (MM: ESI-APCI+), or obtained from Caltech mass spectrometry laboratory. Specific optical rotations were measured with a Jasco P-2000 polarimeter operating on the sodium D-line (589 nm), using a 100 mm path-length cell and are reported as: $[\alpha]_D^T$ (concentration in 10 mg/1 mL, solvent). Yields refer to isolated yield of analytically pure material, unless otherwise noted.

All chemicals were purchased from Aldrich, Acros, TCI, or Alfa-Aesar Chemical Co. and used as received unless otherwise noted. At GIST, anhydrous tetrahydrofuran (THF), diethyl ether (Et_2O) and dichloromethane (CH_2Cl_2) were dried using J.C. Meyer solvent purification system. Hexane was distilled from calcium hydride (CaH_2). At Caltech, solvents were dried by passage through an activated alumina column under argon. Unless specified, all the other chemicals were purchased from Sigma-Aldrich Co., Acros Organics, TCI, Alfa Aesar, and Strem Chemicals Inc. and were used as received without further purification.

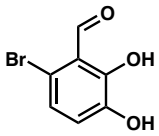
Synthesis of Salen-Crown Ether Ligands

L2 and **L4** are both known compounds, and were synthesized according to previously reported procedures.[1,2]

General Procedure for Aldehyde Synthesis



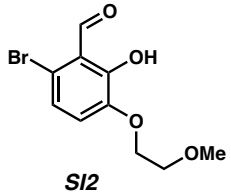
Spectroscopic Data for Ligand Intermediates



SI1

6-bromo-2,3-dihydroxybenzaldehyde (SI1): To a flame-dried round bottom flask under argon was added 6-bromo-2-hydroxy-3-methoxybenzaldehyde (5.74 g, 24.8 mmol, 1.0 equiv) and methylene chloride (50 mL). The solution was cooled to –78 °C and boron tribromide (7.1 mL, 74.5 mmol, 3.0 equiv) in methylene chloride (68 mL) was added dropwise. The reaction was warmed to room temperature and stirred for 5 h. Upon reaction completion the crude reaction mixture was poured into ice water and stirred for 1 h. The aqueous layer was extracted with methylene chloride three times. The combined extracts were washed with water and dried with sodium sulfate, and concentrated by rotary evaporator. The crude solid was then rinsed with hexanes to afford 6-bromo-2,3-dihydroxybenzaldehyde (**SI1**) as an orange solid (4.58 g, 21.1 mmol, 85% yield). ^{1}H

NMR (300 MHz, $CDCl_3$) δ 12.24 – 12.06 (m, 1H), 10.27 (s, 1H), 7.17 – 6.95 (m, 2H), 5.65 (s, 1H). All characterization data match those reported.[3]

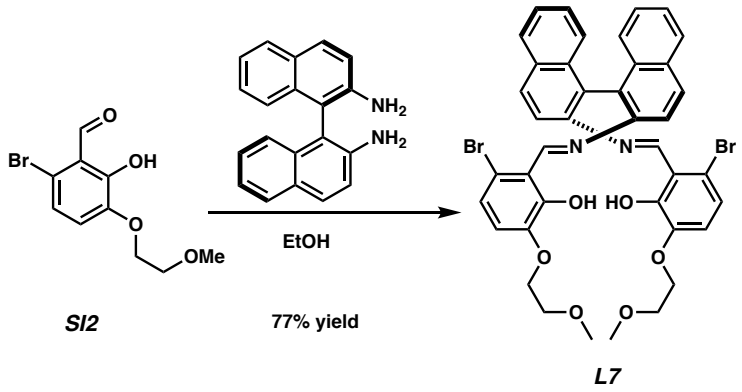


SI2

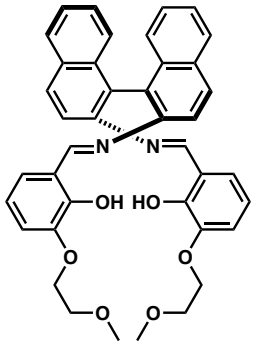
6-bromo-2-hydroxy-3-(2-methoxyethoxy)benzaldehyde (SI2): Sodium hydride (1.17 g, 48.75 mmol, 2.3 equiv) was added to a flame-dried flask under argon. This flask placed in an ice bath, and a solution of 6-bromo-2,3-dihydroxybenzaldehyde (SI1, 4.6 g, 21.2 mmol, 1.0 equiv) in DMSO (42 mL) was added dropwise. The resulting solution was stirred for 3 hours and then 2-methoxyethyl 4-methylbenzenesulfonate[4] (5.37 g, 23.22 mmol, 1.1 equiv) in DMSO (4.2 mL) was added dropwise. The reaction was stirred for 24 hours at room temperature. The reaction was quenched with water and the pH was checked and adjusted to pH = 7. The aqueous layer was extracted with methylene chloride three times and the combined organic extracts were washed with 1M HCl, dried with Na_2SO_4, and then concentrated. The crude reaction mixture was filtered through a pad of silica and concentrated to afford 6-bromo-2-hydroxy-3-(2-methoxyethoxy)benzaldehyde as an yellow solid (3.38 g, 12.3 mmol, 58% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 12.25 (s, 1H), 10.29 (s, 1H), 7.07 (d, J = 8.6 Hz, 1H), 6.99 (d, J = 8.6 Hz, 1H), 4.23 – 4.15 (m, 2H), 3.82 – 3.75 (m, 2H), 3.45 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.5, 155.3, 147.8, 123.6, 121.3, 117.6, 71.0, 69.4, 59.4; IR (neat) 2984, 2942, 2933, 2932, 2750, 1686, 1641, 1579, 1467, 1454, 1438, 1388, 1370, 1332, 1317, 1285, 1274, 1250, 1211, 1202, 1127, 1102, 1081, 898, 861, 822, 789, 749, 676; HRMS $C_{10}H_{12}BrO_4$ $(M+H)^+$: 274.9913, Found: 274.9921.

The corresponding aldehydes for other ligands tested were synthesized according to the procedure described above.

Synthesis of Salen Crown Ether Ligands: Imine Formation



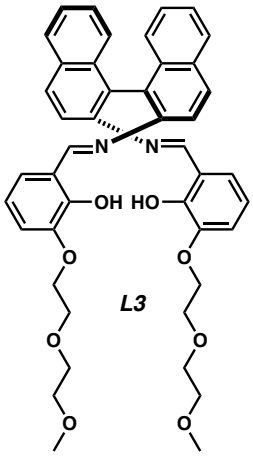
6,6'-((1E,1'E)-(((R)-[1,1'-binaphthalene]-2,2'-diyl)bis(azanylylidene))bis(methanylylidene))bis(5-bromo-2-(2-methoxyethoxy)phenol)(L7): 6-bromo-2-hydroxy-3-(2-methoxyethoxy)benzaldehyde (2.38 g, 8.67 mmol, 2.0 equiv) and (*R*)-(+)-1,1′-Binaphthyl-2,2′-diamine (1.7 g, 4.33 mmol, 1.0 equiv) were combined in EtOH (14 mL). The reaction mixture was heated to 120 °C and stirred for 6 h. Upon completion the reaction was cooled to room temperature and subsequently filtered. The filtered solid were washed with EtOH, concentrated under reduced pressure, affording scarlet solids (2.69 g, 3.36 mmol, 77% yield); $[\alpha]_D^{20}$ = –301.6 (c 0.652, CH_2Cl_2); ^{1}H NMR (400 MHz, C_6D_6) δ 13.84 (s, 1H), 9.08 (s, 1H), 7.65 (d, 12 Hz, 1H), 7.56 (d, 8 Hz, 1H), 7.30 (d, 12 Hz, 1H), 7.25 (d, 8 Hz, 1H), 7.03 (t, 8 Hz, 1H), 6.87 (t, 8 Hz, 1H), 6.54 (d, 8 Hz, 1H), 6.12 (d, 8Hz, 1H), 3.55 (t, 6 Hz, 2H), 3.21 (t, 4 Hz, 2H), 3.01 (s, 3H); ^{13}C NMR (75 MHz, C_6D_6) δ 162.8, 155.0, 147.8, 143.6, 133.5, 132.8, 130.3, 129.4, 128.4, 127.2, 126.5, 126.1, 121.7, 118.2, 117.6, 117.0, 116.8, 70.7, 68.8, 58.5; IR (neat) 2925, 2876, 2817, 1600, 1586, 1440, 1338, 1246, 1224.50, 1125, 1085, 871, 814, 790, 752; HRMS (EI) Calcd. for $C_{40}H_{34}Br_2N_2O_6$: 796.0784, Found: 796.0782.



L1

6,6'-((1E,1'E)-(((R)-[1,1'-binaphthalene]-2,2'-diyl)bis(azanylylidene))bis(methanylylidene))bis(2-(2-methoxyethoxy)phenol)(L1):

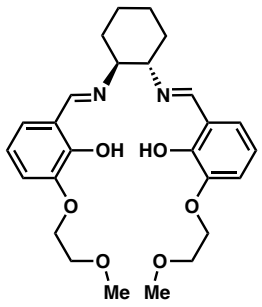
2-hydroxy-3-(2-methoxyethoxy)benzaldehyde (365 mg, 1.86 mmol, 2.0 equiv) and (*R*)-(+)-1,1′-binaphthyl-2,2′-diamine (264.5 mg, 0.930 mmol, 1.0 equiv) were combined in EtOH (7 mL). The reaction mixture was stirred at room temperature for 48 h. The suspension was filtered. The filtered solid were washed with EtOH, concentrated under reduced pressure, affording orange solids (556.8 mg, 0.869 mmol, 94% yield); $[\alpha]_D^{20}$ = – 371.3 (c = 0.6, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.33 (s, 2H), 8.05, 8.02 (d, *J* = 12 Hz, 2H), 8.55 (s, 2H), 7.93, 7.91 (d, *J* = 8 Hz, 2H), 7.54, 7.51 (d, *J* = 12 Hz, 2H), 7.40 – 7.44 (t, *J* = 8 Hz, 2H), 7.22 – 7.24 (d, *J* = 8 Hz, 2H), 7.19, 7.17 (d, *J* = 8 Hz, 2H), 6.89, 6.88 (d, *J* = 4 Hz, 2H), 6.80, 6.79 (d, *J* = 4 Hz, 2H), 6.66 – 6.70 (t, *J* = 8 Hz, 2H), 4.03 – 4.06 (m, 4H), 3.65 – 3.68 (t, *J* = 6 Hz, 4H), 3.39 (s, 6H); HRMS (EI) Calcd. for $C_{40}H_{36}N_2O_6$: 640.2573, Found: 640.2573.



L3

6,6'-((1E,1'E)-(((R)-[1,1'-binaphthalene]-2,2'-diyl)bis(azanylylidene))bis(methanylylidene))bis(2-(2-(2-methoxyethoxy)ethoxy)phenol)(L3):

2-hydroxy-3-(2-(2-methoxyethoxy)ethoxy)benzaldehyde (191.3 mg, 0.797 mmol, 2.0 equiv) and (*R*)-(+)-1,1′-binaphthyl-2,2′-diamine (96.4 mg, 0.339 mmol, 1.0 equiv) were combined in EtOH (3 mL). The reaction mixture was stirred at room temperature for 48 h. The suspension was filtered. The filtered solid were washed with EtOH, and concentrated under reduced pressure to afford an orange solid (196.5 mg, 0.2697 mmol, 80% yield); $[\alpha]_D^{20}$ = –352.1 (c 0.518, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.28 (s, 2H), 8.03 (d, *J* = 8 Hz, 2H), 7.92 (d, *J* = 8 Hz, 2H), 7.52 (d, *J* = 8 Hz, 2H), 7.44-7.40 (m, 2H), 7.26-7.22 (m, 2H), 7.18 (d, *J* = 8 Hz, 2H), 6.88 (d, *J* = 8 Hz, 2H), 6.79 (d, *J* = 8 Hz, 2H), 6.70 – 6.66 (m, 2H), 4.09 – 4.06 (m, 4H), 3.79 – 3.76 (m, 4H), 3.67 – 3.65 (m, 4H), 3.53 – 3.51 (m, 4H), 3.37 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.0, 151.7, 147.2, 144.4, 133.3, 132.6, 130.2, 128.4, 127.0, 126.6, 125.8, 124.9, 119.7, 118.2, 118.0, 117.8, 72.0, 70.7, 69.7, 69.1, 59.1; IR (neat) 3269, 3046, 2955, 2362, 1603, 1574, 1506, 1470, 1429, 1389, 1359, 1306, 1261, 1194, 1143, 1087, 969, 856, 815, 744, 700; HRMS (EI) Calcd. for $C_{44}H_{44}N_2O_8$: 728.3098, Found: 728.3096.

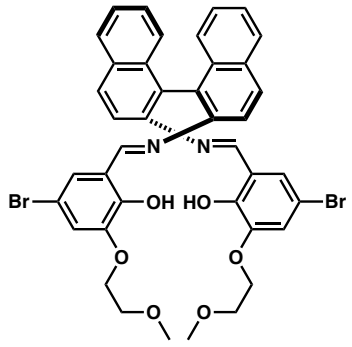


L5

6,6'-((1E,1'E)-(((1R,2R)-cyclohexane-1,2-diyl)bis(azanylylidene))bis(methanylylidene))bis(2-(2-methoxyethoxy)phenol)(L5):

2-hydroxy-3-(2-methoxyethoxy)benzaldehyde (193 mg, 0.986 mmol, 2.0 equiv) and (1*R*,2*R*)-cyclohexane-1,2-diamine (56.3 mg, 0.493 mmol, 1.0 equiv) were combined in EtOH (8 mL). The reaction mixture was stirred at room temperature for 48 h. The solvent was removed under reduced pressure. The residue was purified by column

chromatography on silica-gel (*n*-hexane/EtOAc = 2/1, 1/2, 1/4), affording an oil (195 mg, 0.414 mmol, 84% yield): $[\alpha]_D^{20}$ = +120.0 (c = 0.5, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 13.83 (s, 2H), 8.21 (s, 2H), 6.88 – 6.90 (m, 2H), 6.77 – 6.80 (m, 2H), 6.67 – 6.71 (t, *J* = 6 Hz, 2H), 4.13 – 4.16 (t, *J* = 4.5 Hz, 4H), 3.76 – 3.78 (t, *J* = 3 Hz, 4H), 3.43 (s, 6H), 1.84 –1.93 (m, 4H), 1.66 –1.69 (d, *J* = 9 Hz, 2H), 1.42 – 1.47 (m, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.8, 152.2, 147.4, 124.0, 118.7, 117.9, 116.6, 72.5, 71.2, 68.6, 59.3, 33.1, 24.1; IR (neat) 3315, 2929, 2856, 1625, 1578, 1477, 1450, 1376, 1338, 1289, 1233, 1022; HRMS (EI) Calcd. for $C_{26}H_{34}N_2O_6$: 470.2417, Found: 470.2425.

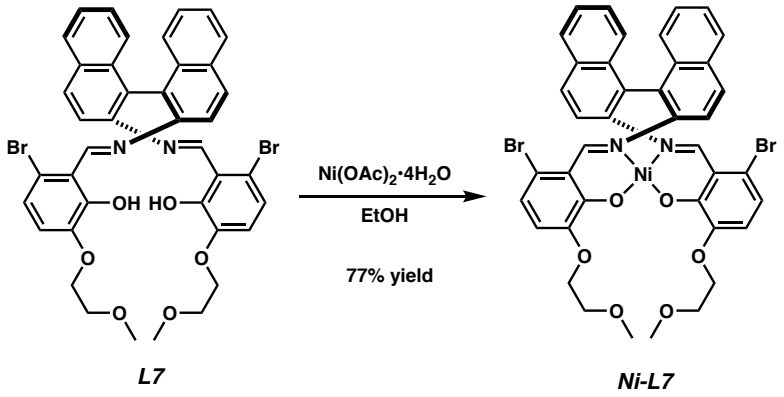


L6

6,6'-((1E,1'E)-(((R)-[1,1'-binaphthalene]-2,2'-diyl)bis(azanylylidene))bis(methanylylidene))bis(4-bromo-2-(2-methoxyethoxy)phenol)(L6): 5-bromo-2-hydroxy-3-(2-(2-ethoxyethoxy)ethoxy)benzaldehyde (80 mg, 0.291 mmol) and (*R*)-(+)-1,1′-Binaphthyl-2,2′-diamine (41.232 mg, 0.145 mmol) were combined in EtOH (10 mL). The reaction mixture was stirred at room temperature for 48 h. The suspension was filtered. The filtered solid were washed with EtOH, concentrated under reduced pressure, affording scarlet solids (100 mg, 0.125 mmol, 86% yield); $[\alpha]_D^{20}$ = –298.5 (c 0.5, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.32 (s, 2H), 8.45 (s, 2H), 8.02 (d, 12 Hz, 2H), 7.91 (d, 8 Hz, 2H), 7.48 (d, 12 Hz, 2H), 7.41 (m, 2H), 7.21 (m, 2H), 7.13 (d, 8 Hz, 2H), 6.96 (d, 4 Hz, 2H), 6.91 (d, 4 Hz, 2H), 4.01 (m, 4H), 3.65 (t, 4Hz, 4H), .38 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.6, 150.9, 148.2, 143.7, 133.2, 132.8, 130.4, 126.2, 120.4, 120.2, 117.5, 109.5, 77.5, 77.2, 76.9, 70.8, 69.1, 59.2; IR (neat) 2875, 2360, 2342, 1605, 1569, 1450,

1398, 1363, 1332, 1250, 1199, 1125, 1089, 1026, 972, 900, 817, 751; HRMS (EI) Calcd. for $C_{40}H_{34}Br_2N_2O_6$: 796.0784, Found: 796.0784.

Complexation Procedure (Ni/L synthesis)



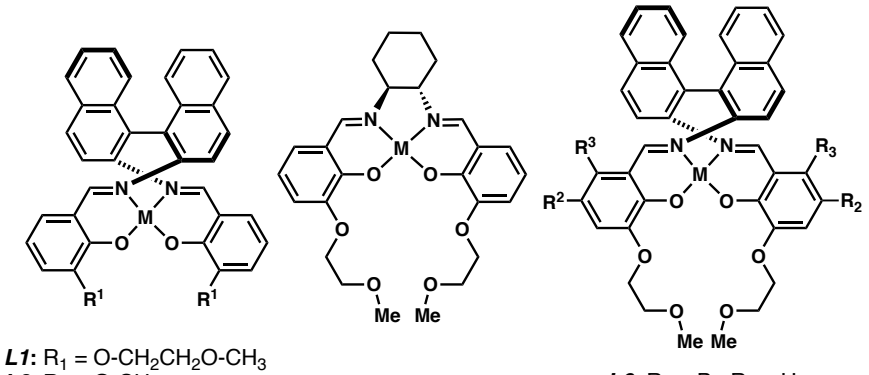
L7 *Ni-L7*

Ni-Salen Complex (Ni-L7): $Ni(OAc)_2$•$4H_2O$ (1.17 g, 4.7 mmol, 1.4 equiv) and **L7** (2.69 g, 3.36 mmol, 1.0 equiv**)** were combined in EtOH and heated to 100 °C for 12 h. While still hot, the crude mixture was transferred into a 20 mL vial, rinsing with a small amount of additional EtOH. The vial was placed in the freezer for 12h. The crude mixture was then centrifuged down, and the EtOH was decanted. The mother liquor was then reduced via rotary evaporator, and the crude reaction mixture was allowed to rest in the freezer, and centrifuged a second time. The combined precipitates were washed with hexanes, and dried under vacuum to afford a yellow solid (2.2 g, 2.58 mmol, 77% yield).

All other metal complexes were prepared using this identical procedure.

HRMS for the Catalysts with Nickel

The normal spectral region could not be determined by ^{1}H NMR, since Ni(II)-salen complexes **1d**, **2**, **3a-c**, **4** and **5** are paramagnetic. High-resolution mass spectra (HRMS) shows desired Ni(II)-salen complexes bearing one nickel atom. Similar effects for Ni(II)-salen complexes have been reported before.[5,6]



L1: R_1 = O-CH_2CH_2O-CH_3
L2: R_1 = O-CH_3
L3: R_1 = O-$[CH_2CH_2O]_2$-CH_3
L4: R_1 = *t*-Bu

L5

L6: R_2 = Br, R_3 = H
L7: R_2 = H, R_3 = Br

L1-Ni: HRMS (EI) Calcd. $C_{40}H_{34}N_2NiO_6$: 696.1770, Found: 696.1769

L2-Ni: HRMS (EI) Calcd. $C_{36}H_{26}N_2NiO_4$: 608.1246, Found: 608.1243

L3-Ni: HRMS (EI) Calcd. $C_{44}H_{42}N_2NiO_8$: 784.2295, Found: 784.2294

L4-Ni: HRMS (EI) Calcd. $C_{42}H_{38}N_2NiO_2$: 660.2287, Found: 660.2286

L5-Ni: HRMS (EI) Calcd. $C_{26}H_{32}N_2NiO_6$: 526.1614, Found: 526.1614

L6-Ni: HRMS (EI) Calcd. $C_{40}H_{33}Br_2N_2NiO_6$: 853.0059, Found: 853.0068

L7-Ni: HRMS (EI) Calcd. $C_{40}H_{32}Br_2N_2NiO_6$: 851.9980, Found: 851.9981

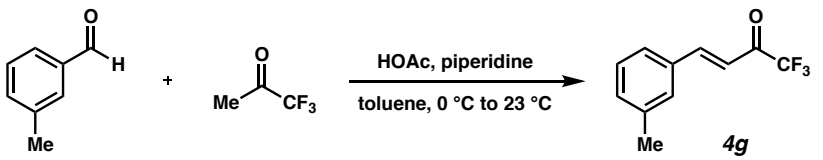
Synthesis of Trifluoromethylketones:

Aryl trifluoromethylketone **1h** was prepared according to a previously reported procedure.[7] All other aryl trifluoromethylketones were purchased from Alfa Aesar, Sigma-Aldrich, or TCI and used without further purification.

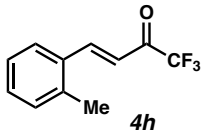
Synthesis of Vinyl Trifluoromethylketones:

Previously reported methods were used to prepare **4a**[8], **4b**[8], **4c**[9], **4d**[8], **4e**[8], **4i**[10], **4j**[10], **4k**[9], and **4n**[10].

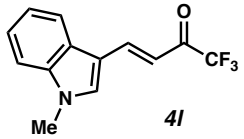
General Procedure for the Synthesis of Vinyl Trifluoromethylketones:



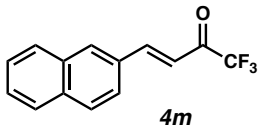
(*E*)-1,1,1-trifluoro-4-(*m*-tolyl)but-3-en-2-one (4g): 3-methylbenzaldehyde (601 mg, 5.0 mmol, 1.0 equiv), piperidine (493 μL, 5.0 mmol, 1.0 equiv) and acetic acid (429 μL, 7.5 mmol, 1.5 equiv) were all combined in dry toluene (5 mL) in a flame-dried flask under argon. The resultant solution was cooled to 0 °C, and then trifluoroacetone (1.8 mL, 20 mmol, 4 equiv) in toluene (5 mL) was added slowly. The reaction was stirred at 0 °C for 2 hours and then allowed to stir at room temperature for 24 h. The reaction was quenched with saturated ammonium chloride solution and the aqueous layer was extracted three times with ethyl acetate. The combined organic extracts were washed with water, and dried with sodium sulfate**.** Product **4g** purified by column chromatography (5% EtOAc in hexanes) to provide a colorless oil (415 mg, 38% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 15.9 Hz, 1H), 7.48 – 7.42 (m, 2H), 7.37 – 7.29 (m, 2H), 7.01 (dq, *J* = 16.0, 1.0 Hz, 1H), 2.41 (t, *J* = 0.7 Hz, 3H). All characterization data match those reported.[11]



(*E*)-1,1,1-trifluoro-4-(*o*-tolyl)but-3-en-2-one (4h): Product **4h** purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (107 mg, 50% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31 (dt, *J* = 15.9, 0.6 Hz, 1H), 7.74 – 7.64 (m, 1H), 7.38 (td, *J* = 7.3, 1.4 Hz, 1H), 7.26 (m, 2H), 6.96 (dq, *J* = 15.8, 0.9 Hz, 1H), 2.50 (s, 3H). All characterization data match those reported.[11]

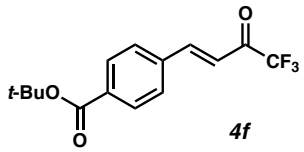


(*E*)-1,1,1-trifluoro-4-(1-methyl-1*H*-indol-3-yl)but-3-en-2-one (4l): Product **4l** purified by column chromatography (20% EtOAc in hexanes) to provide a yellow oil (192 mg, 15% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (dd, *J* = 15.5, 0.7 Hz, 1H), 7.97 – 7.91 (m, 1H), 7.58 (s, 1H), 7.44 – 7.32 (m, 3H), 6.97 (dt, *J* = 15.6, 1.0 Hz, 1H), 3.88 (s, 3H). All characterization data match those reported.[12]



4m

(*E*)-1,1,1-trifluoro-4-(naphthalen-2-yl)but-3-en-2-one (4m): Product **4m** purified by column chromatography (8% EtOAc in hexanes) to provide a light yellow oil (500 mg, 40% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 – 8.05 (m, 2H), 7.95 – 7.86 (m, 3H), 7.75 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.64 – 7.52 (m, 2H), 7.13 (dq, *J* = 16.0, 0.9 Hz, 1H). All characterization data match those reported.[13]



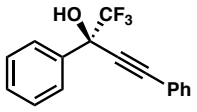
4f

***tert*-butyl (*E*)-4-(4,4,4-trifluoro-3-oxobut-1-en-1-yl)benzoate (4f):** Product **4f** was prepared using the general procedure and purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (687 mg, 2.3 mmol, 46% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.1 Hz, 2H), 7.97 (d, *J* = 16.0 Hz, 1H), 7.68 (d, *J* = 8.1 Hz, 2H), 7.07 (dt, *J* = 16.0, 1.0 Hz, 1H), 1.61 (d, *J* = 0.9 Hz, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 148.8, 136.9, 135.2, 130.3, 129.0, 118.5, 82.0, 28.30; ^{19}F NMR (282 MHz, $CDCl_3$) δ –77.66; IR (Neat Film, NaCl) 3051, 2995, 1709, 1613, 1568, 1417, 1392, 1368, 1305, 1265, 1189, 1141, 1063, 994, 896, 844, 772, 733, 705, 683 cm^{-1}.

Procedure for Enantioselective Alkynylation of Aryl Trifluoromethyl Ketones

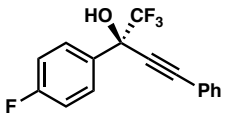
To a stirred solution of catalyst (0.012 mmol, 0.05 equiv) in THF (0.5 mL), alkyne (0.968 mmol, 4 equiv), 4 Å molecular sieve (377 mg), and KO*t*Bu (0.484 mmol, 0.2 equiv) were added at room temperature slowly. After the solution had been stirred at

room temperature for 30 min to give a dark yellow mixture, ketone (0.242 mmol, 1.0 equiv) was added dropwise to a solution. After the resulting mixture was stirred at room temperature for 24 h, saturated ammonium chloride (2mL) was added to quench the reaction. The solution was extracted with ethyl acetate (3×15 mL). The combined organic layers were washed with brine, dried over anhydrous magnesium sulfate, and concentrated by rotary evaporation. The residue was purified by flash column chromatography ethyl acetate/hexane. Enantiomeric excesses were determined by HPLC on chiral stationary phase (Daicel Chiralpak IB or ID column (0.46 cm × 25 cm)).



3aa

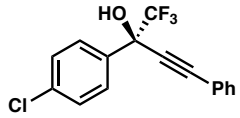
1,1,1-trifluoro-2,4-diphenylbut-3-yn-2-ol (3aa) : Product **3aa** was prepared using the general procedure to provide a pale yellow oil (93% yield, 93% ee); $[\alpha]_D^{24}$ = +26.8 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 11.081 min, major t_R = 9.556 min. All characterization data match those reported. [14]



3ba

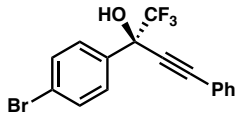
1,1,1-trifluoro-2-(4-fluorophenyl)-4-phenylbut-3-yn-2-ol (3ba):

Product **3ba** was prepared using the general procedure to provide a pale yellow oil (93% yield, 90% ee); $[\alpha]_D^{24}$ = +25.2 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak ID column; Hexane/i-PrOH = 99:1; flow rate 0.5 mL/min, 254 nm wave length UV; minor t_R = 11.809 min, major t_R = 11.014 min. All characterization data match those reported.[15]



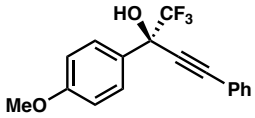
3ca

2-(4-chlorophenyl)-1,1,1-trifluoro-4-phenylbut-3-yn-2-ol (3ca): Product **3ca** was prepared using the general procedure to provide a pale yellow oil (94% yield, 89% ee); $[\alpha]_D^{24}$ = +17.8 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak ID column; Hexane/i-PrOH = 99:1; flow rate 0.5 mL/min, 254 nm wave length UV; minor t_R = 12.818 min, major t_R = 11.254 min. All characterization data match those reported.[14]



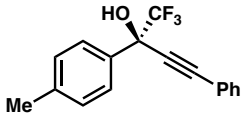
3da

2-(4-bromophenyl)-1,1,1-trifluoro-4-phenylbut-3-yn-2-ol (3da): Product **3da** was prepared using the general procedure to provide a pale yellow oil (97% yield, 89% ee); $[\alpha]_D^{24}$ = +14.2 (c = 0.8, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak ID column; Hexane/i-PrOH = 99:1; flow rate 0.5 mL/min, 254 nm wave length UV; minor t_R = 14.004 min, major t_R = 11.924 min. All characterization data match those reported.[15]



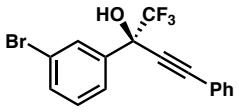
3ea

(*R*)-1,1,1-trifluoro-2-(4-methoxyphenyl)-4-phenylbut-3-yn-2-ol (3ea): Product **3ea** was prepared using the general procedure to provide a pale yellow oil (86% yield, 97% ee); $[\alpha]_D^{24}$ = +22.7 (c = 0.6, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 12.240 min, major t_R = 22.694 min. All characterization data match those reported.[14]



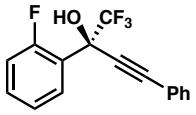
3fa

1,1,1-trifluoro-4-phenyl-2-(p-tolyl)but-3-yn-2-ol (3fa): Product **3fa** was prepared using the general procedure to provide a pale yellow oil (86% yield, 97% ee); $[\alpha]_D^{24}$ = +22.4 (c = 0.6, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak ID column; Hexane/i-PrOH = 99:1; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 8.663 min, major t_R = 7.225 min. All characterization data match those reported.[14]



3ga

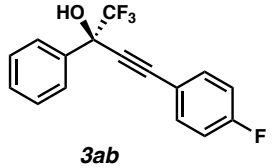
2-(3-bromophenyl)-1,1,1-trifluoro-4-phenylbut-3-yn-2-ol (3ga) : Product **3ga** was prepared using the general procedure to provide a pale yellow oil (92% yield, 85% ee); $[\alpha]_D^{24}$ = +23.0 (c = 0.7, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.74 (d, 8 Hz, 1H), 7.53 (m, 3H), 7.35 (m, 3H), 7.29 (t, 8 Hz, 1H), 3.25 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.6, 132.8, 132.2, 130.44, 129.9, 128.6, 126.1, 125.2, 122.5, 121.4, 120.7, 88.7, 83.8, 73.1, 72.7; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.08; IR (Neat) 3547, 3066, 2233, 1594, 1570, 1490, 1473, 1444, 1423, 1348, 1246, 1168, 1111, 1074, 1012, 997, 937, 884, 782, 755, 735, 708, 687; HRMS (EI) Calcd. for $C_{16}H_{10}BrF_3O$: 353.9867, Found: 353.9871. The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak ID column; Hexane/i-PrOH = 99:1; flow rate 0.5 mL/min, 254 nm wave length UV; minor t_R = 12.137 min, major t_R = 11.398 min.



3ha

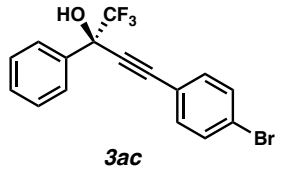
1,1,1-trifluoro-2-(2-fluorophenyl)-4-phenylbut-3-yn-2-ol (3ha): Product **3ha** was prepared using the general procedure to provide a pale yellow oil (70% yield, 87% ee);

$[\alpha]_D^{24}$ = +10.6 (c = 0.2, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (m, 1H), 7.53 (m, 2H), 7.32 (m, 4H), 7.11(m, 2H), 3.55 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.4, 158.9, 132.2, 131.8, 131.7, 129.9, 129.6, 128.5, 124.3, 124.2, 121.0, 117.0, 116.7, 88.2, 83.1, 72.4, 71.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.24, –110.69; IR (neat) 3576, 2927, 2235, 1655, 1613, 1585, 1489, 1453, 1377, 1249, 1228, 1174, 1153, 1120, 1000, 1010, 922, 823, 755, 740, 711, 689; HRMS (EI) Calcd. for $C_{16}H_{10}F_4O$: 294.0668, Found: 294.0670; The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak ID column; Hexane/i-PrOH = 99:1; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 7.523 min, major t_R = 6.467 min.



3ab

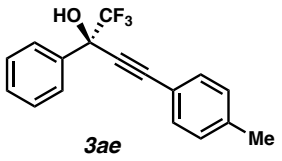
(*R*)-1,1,1-trifluoro-2-phenyl-4-(p-tolyl)but-3-yn-2-ol (3ab) : Product **3ab** was prepared using the general procedure to provide a pale yellow oil (98% yield, 91% ee); $[\alpha]_D^{24}$ = +18.0 (c = 0.2, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 12.079 min, major t_R = 8.448 min. All characterization data match those reported.[15]



3ac

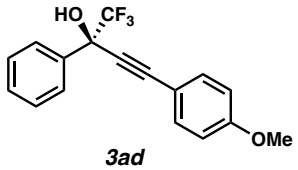
(*R*)-4-(4-bromophenyl)-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol (3ac): Product **3ac** was prepared using general procedure to provide a pale yellow oil (90% yield, 91% ee); $[\alpha]_D^{24}$ = +29.9 (c = 0.7, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (m, 2H), 7.38(m, 7H), 3.19(s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.1, 133.6, 131.9, 129.7, 128.4, 127.2, 125.3, 124.2, 121.5, 119.9, 87.1, 85.6, 73.7, 73.3; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.06; IR (neat) 3566, 2926, 2360, 2234, 1605, 1587, 1452, 1394, 1361, 1248, 1166, 1116, 1098, 1064, 1012, 932, 906, 822, 761, 729, 696, 668; HRMS (EI) Calcd. for

$C_{16}H_{10}BrF_3O$: 353.9867, Found: 353.9868; The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 13.405 min, major t_R = 9.004 min.



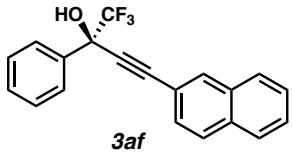
3ae

(*R*)-1,1,1-trifluoro-2-phenyl-4-(p-tolyl)but-3-yn-2-ol (3ae) : Product **3ae** was prepared using general procedure to provide a pale yellow oil (94% yield, 90% ee); $[\alpha]_D^{24}$ = +26.5 (c = 0.6, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 10.213 min, major t_R = 7.287 min. All characterization data match those reported.[15]



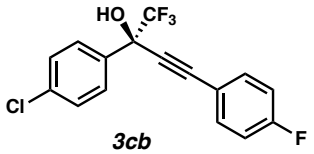
3ad

(*R*)-1,1,1-trifluoro-4-(4-methoxyphenyl)-2-phenylbut-3-yn-2-ol (3ad): Product **3ad** was prepared using general procedure to provide a pale yellow oil (99% yield, 93% ee); $[\alpha]_D^{24}$ = +26.7 (c = 0.2, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (m, 2H), 7.41 (m, 5H), 6.86 (m, 2H), 3.83 (s, 3H), 3.12 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.6, 135.6, 133.7, 129.5, 128.3, 127.3, 114.2, 113.0, 88.3, 83.3, 73.7, 73.2, 55.4, 29.7; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.19; IR (neat) 3428, 2923, 2230, 1605, 1570, 1510, 1451, 1359, 1294, 1248, 1172, 1108, 1065, 1016, 933, 907, 832, 764, 706; HRMS (EI) Calcd. for $C_{17}H_{13}F_3O_2$: 306.0868, Found: 306.0869; The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 17.337 min, major t_R = 12.175 min.



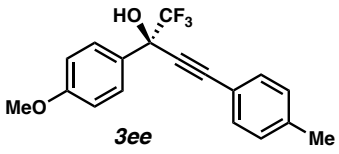
3af

(*R*)-1,1,1-trifluoro-4-(naphthalen-2-yl)-2-phenylbut-3-yn-2-ol (3af): Product **3af** was prepared using general procedure to provide a pale orange solid (89% yield, 89% ee); $[\alpha]_D^{24}$ = +2.59 (c = 0.6, CH_2Cl_2); ^{1}H NMR (300 MHz, CDCl3) δ 8.09 (s, 1H), 7.81 (m, 5H), 7.47 (m, 6H), 3.23 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.5, 133.5, 132.9, 132.7, 129.7, 128.4, 128.3, 128.2, 128.0, 127.9, 127.4, 127.3, 127.0, 125.5, 121.7, 118.3, 88.6, 84.8, 73.8, 73.4, 29.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.01; IR (neat) 3528, 3061, 2924, 2853, 2360, 2228, 1595, 1501, 1488, 1450, 1360, 1226, 1168, 1097, 1063, 1005, 906, 868, 822, 767, 751, 700, 663; HRMS (EI) Calcd. for $C_{20}H_{13}F_3O$: 326.0918, Found: 326.0918; The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 16.764 min, major t_R = 11.734 min.



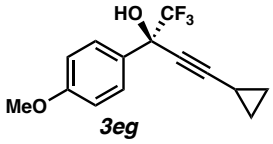
3cb

(*R*)-2-(4-chlorophenyl)-1,1,1-trifluoro-4-(4-fluorophenyl)but-3-yn-2-ol (3cb) : Product **3cb** was prepared using general procedure to provide a pale yellow oil (92% yield, 89% ee); $[\alpha]_D^{24}$ = +14.6 (c = 0.7, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, 9 Hz, 2H), 7.49 (m, 2H), 7.40 (m, 2H), 7.03 (m, 2H), 3.10 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.1, 161.7, 135.9, 134.3, 134.2, 133.8, 128.7, 128.6, 125.2, 121.4, 116.9, 116.2, 115.9, 87.4, 83.8, 73.3, 72.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.27, –108.15; IR (neat) 3453, 2928, 2235, 1706, 1652, 1601, 1507, 1491, 1406, 1359, 1233, 1184, 1121, 1093, 1010, 949, 917, 765, 730, 717, 694; HRMS (EI) Calcd. for $C_{16}H_9ClF_4O$: 328.0278, Found: 328.0278; The enantiomeric excess was determined by HPLC through chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 9.181 min, major t_R = 7.976 min.



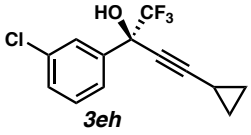
3ee

(*R*)-1,1,1-trifluoro-2-(4-methoxyphenyl)-4-(p-tolyl)but-3-yn-2-ol (3ee): Product **3ee** was prepared using general procedure to provide a pale yellow oil (92% yield, 96% ee); $[\alpha]_D^{24}$ = +17.5 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 98:2; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 28.064 min, major t_R = 9.684 min. All characterization data match those reported.[15]



3eg

(*R*)-4-cyclopropyl-1,1,1-trifluoro-2-(4-methoxyphenyl)but-3-yn-2-ol (3eg): Product **3eg** was prepared using general procedure to provide a pale yellow solid (93% yield, 96% ee); $[\alpha]_D^{24}$ = +3.84 (c = 0.3, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 (d, 9 Hz, 2H), 6.89 (m, 2H), 3.82 (s, 3H), 2.93 (s, 1H), 1.26 (m, 1H), 0.76 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.4, 128.6, 127.9, 125.4, 121.6, 113.5, 92.5, 71.3, 55.4, 8.5; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.69; IR (neat) 2994, 2931, 2828, 1605, 1573, 1505, 1458, 1431, 1396, 1345, 1249, 1204, 1078, 970, 923, 818, 781, 733, 713, 687; HRMS (EI) Calcd. for $C_{14}H_{13}F_3O_2$: 270.0868, Found: 270.0868; The enantiomeric excess was determined by HPLC through chiral HPLC analysis: Daicel Chiralpak ID column; Hexane/i-PrOH = 99:1; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 12.873 min, major t_R = 11.879 min.

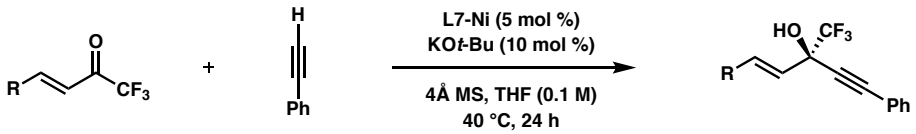


3eh

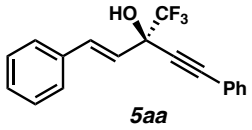
(*R*)-2-(3-chlorophenyl)-4-cyclopropyl-1,1,1-trifluorobut-3-yn-2-ol (3eh): Product **3eh** was prepared using general procedure to provide a pale yellow oil (95% yield, 80% ee); $[\alpha]_D^{24}$ = +2.4 (c = 0.3, CH_2Cl_2); **^{1}**H NMR (300 MHz, $CDCl_3$) δ 7.70 (s, 1H), 7.58 (m, 1H), 7.30 (m, 2H), 2.95 (s, 1H), 1.32 (m, 1H), 0.78 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.7, 134.2, 129.6, 129.4, 127.6, 125.5, 93.2, 72.7, 72.1, 70.6, 29.8, 8.6, 0.6 ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.46; IR (neat) 3458, 3016, 2441, 1597, 1578, 1475, 1428, 1364, 1261, 1165, 1106, 1076, 1027, 943, 925, 884, 814, 787, 721, 688; HRMS (EI) Calcd. for

$C_{13}H_{10}ClF_3O$: 274.0323, Found: 274.0372; The enantiomeric excess was determined by chiral HPLC analysis: Daicel Chiralpak IB column; Hexane/i-PrOH = 99:1; flow rate 1 mL/min, 254 nm wave length UV; minor t_R = 9.858 min, major t_R = 9.287 min.

Procedure for Enantioselective Alkynylation of Vinyl Trifluoromethyl Ketones

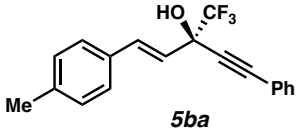


n = number of reactions. All reactions were set-up in a N_2-filled glovebox. To a vial containing **L7-Ni** (8.6n mg, 0.01n mmol, 0.05 equiv) was added KO*t*-Bu (2.24n mg, 0.02 n mmol, 0.1 equiv) in THF (1.6n mL). The resulting solution was stirred until the solids were fully dissolved. To a new 4 dram vial was added 4 Å MS (32 mg) and phenylacetylene (88 μL, 0.8 mmol, 4.0 equiv). The **L7-Ni** + KO*t*-Bu solution (1.6 mL) was then added to this vial, and the solution was stirred for 30 min. The vinyl trifluoromethylketone (0.2 mmol, 1.0 equiv) in THF (0.8 mL) was then added and the reaction was stirred at 40 °C for 24 h. The reaction was then quenched with sat. NH_4Cl solution and the aqueous layer was extracted three times with ethyl acetate. The combined organic extracts were dried with sodium sulfate and concentrated by rotary evaporator. The crude oil was then purified by column chromatography to afford the desired product.



(*R*,*E*)-1,5-diphenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5aa). Product **5aa** was purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (51.8 mg, 86% yield); 90% ee, $[\alpha]_D^{25}$ +11.8 (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.48 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.45 – 7.27 (m, 6H), 7.21 (d, *J* = 15.8 Hz, 1H), 6.35 (d, *J* = 15.8 Hz, 1H), 2.91 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.8, 135.3, 132.2, 129.7, 129.0, 128.9, 128.6, 127.4, 123.6 (d, *J* = 285.3 Hz),

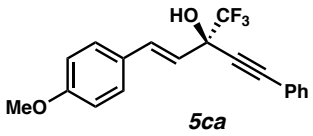
122.4, 121.1, 88.7, 82.7, 72.4 (q, J = 32.9 Hz);^{19}F NMR (282 MHz, $CDCl_3$) δ 80.72 IR (Neat Film, NaCl) 3412, 3030, 2924, 1491, 1445, 1249, 1187, 1130, 1056, 966, 753, 690 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{18}H_{12}F_3$ $[M\text{-}OH]^+$: 285.0886 found 285.0883; SFC Conditions: 15% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 254 nm, t_R (min): major = 3.89, minor = 4.43.



5ba

(*R*,*E*)-5-phenyl-1-(*p*-tolyl)-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ba):

Product **(5ba)** was prepared using general procedure and purified by column chromatography (10% EtOAc in hexanes) to provide a colorless oil (62.2 mg, 98% yield); 92% ee, $[\alpha]_D^{25}$ +8.5 (*c* 0.99, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, J = 8.0, 1.6 Hz, 2H), 7.46 – 7.32 (m, 5H), 7.23 – 7.15 (m, 3H), 6.31 (d, J = 15.8 Hz, 1H), 2.96 (s, 1H), 2.38 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.1, 135.7, 132.5, 132.2, 129.63, 129.55, 128.6, 127.3, 123.6 (q, J = 285.3 Hz), 121.3, 121.1, 88.7, 82.8, 72.5 (q, J = 32.8 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.72; IR (Neat Film, NaCl) 3412, 2924, 1654, 1515, 1491, 1444, 1361, 1249, 1186, 1131, 1054, 968, 797, 756, 727, 689 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{19}H_{14}F_3$ $[M\text{-}OH]^+$: 299.1042 found 299.1041; SFC Conditions: 6% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 254 nm, t_R (min): major = 14.12, minor = 15.02.

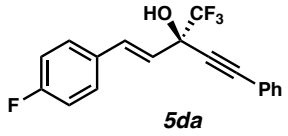


5ca

(*R*,*E*)-1-(4-methoxyphenyl)-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ca):

Product **(5ca)** was prepared using general procedure and purified by column chromatography (10% EtOAc in hexanes) to provide a colorless oil (63.9 mg, 98% yield); 92% ee, $[\alpha]_D^{25}$ +7.7 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$ δ 7.58 – 7.50 (m, 2H), 7.46 – 7.32 (m, 5H), 7.15 (d, J = 15.8 Hz, 1H), 6.94 – 6.85 (m, 2H), 6.20 (dd, J = 15.8, 0.7 Hz, 1H), 3.83 (s, 3H), 2.92 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.3, 135.3, 132.2, 129.6, 128.7, 128.6, 128.0, 125.0, 122.2, 121.2, 120.1, 114.3, 88.6, 82.9,

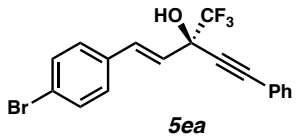
72.5 (q, 32.8 Hz), 55.5; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.73; IR (Neat Film, NaCl) 3411, 2936, 2840, 1654, 1608, 1513, 1466, 1444, 1422, 1250, 1176, 1132, 1106, 1059, 967, 850, 824, 803, 757, 728, 690 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{19}H_{14}F_3O$ [M-OH]$^+$: 315.0991 found 315.0993; SFC Conditions: 15% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 254 nm, t_R (min): major = 5.17, minor = 5.42.



5da

(*R*, *E*)-1-(4-fluorophenyl)-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5da):

Product **5da** was prepared using general procedure and purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (61.7 mg, 92% yield); 91% ee, $[\alpha]_D^{25}$ +12.0 (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, *J* = 8.1, 1.6 Hz, 2H), 7.45 (dd, *J* = 8.7, 5.3 Hz, 2H), 7.42 – 7.34 (m, 3H), 7.16 (d, *J* = 15.8 Hz, 1H), 7.06 (t, *J* = 8.7 Hz, 2H), 6.25 (d, *J* = 15.8 Hz, 1H), 2.92 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.4, 161.9, 134.6, 132.2, 131.5 (q, *J* = 3.3 Hz), 129.7, 129.1 (d, *J* = 8.2 Hz) 128.6, 125.0, 122.2, 121.0, 116.0, 115.8, 88.8, 82.7, 72.3 (q, *J* = 32.9 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.76, –112.35; IR (Neat Film, NaCl) 3401, 3056, 2927, 1602, 1510, 1492, 1444, 1362, 1234, 1187, 1159, 1130, 1094, 1055, 967, 854, 826, 808, 757, 728, 690 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{18}H_{11}F_4$ [M-OH]$^+$: 303.0791 found 303.0794; SFC Conditions: 20% IPA, 2.5 mL/min, Chiralpak AD-H column, λ = 254 nm, t_R (min): major = 7.83, minor = 10.52.

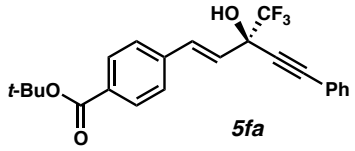


5ea

(*R*, *E*)-1-(4-bromophenyl)-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ea):

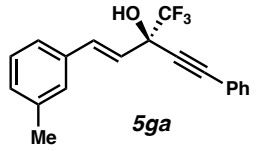
Product **5ea** was prepared using general procedure and purified by column chromatography (15% Et_2O in hexanes) to provide a colorless oil (70.8 mg, 93% yield); 90% ee, $[\alpha]_D^{25}$ –0.6 (*c* 0.98, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.46 (m, 4H), 7.45 – 7.31 (m, 5H), 7.13 (d, *J* = 15.8 Hz, 1H), 6.32 (d, *J* = 15.8 Hz, 1H), 2.93 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 134.5, 134.2, 132.2, 132.0, 129.8, 128.9, 128.6, 123.5 (q,

J = 285.4 Hz), 123.1, 123.0, 120.9, 88.9, 82.5, 72.3 (q, J = 33.0 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.71; IR (Neat Film, NaCl) 3400, 1489, 1248, 1187, 1130, 1056, 1010, 967, 816, 756, 690 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{18}H_{11}BrF_3O$ $[M\text{-}OH]^+$: 362.9991 found 362.9984; SFC Conditions: 8% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 254 nm, t_R (min): major = 12.79, minor = 13.56.



5fa

***tert*-butyl(*R*,*E*)-4-(3-hydroxy-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-1-yl)benzoate (5fa)** Product **5fa** was prepared using general procedure and purified by column chromatography (15% EtOAc in hexanes) to provide a colorless oil (65.9 mg, 82% yield); 89% ee, $[\alpha]_D^{25}$ –10.4 (*c* 0.95, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 8.4 Hz, 2H), 7.58 – 7.47 (m, 4H), 7.45 – 7.32 (m, 3H), 7.22 (d, J = 15.8 Hz, 1H), 6.41 (d, J = 15.8 Hz, 1H), 2.95 (s, 1H), 1.60 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 139.3, 134.6, 132.2, 132.0, 130.0, 129.7, 128.6, 127.1, 125.0, 124.8, 123.5 (q, J = 285.5 Hz), 88.7, 82.6, 81.6, 72.2 (q, J = 32.9 Hz), 28.3; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.66 IR (Neat Film, NaCl) 3402, 2979, 1711, 1691, 1608, 1478, 1492, 1445, 1394, 1370, 1317, 1299,1250, 1184, 1127, 1070, 1018, 972, 846, 757, 691, 613 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{23}H_{20}F_3O_2$ $[M\text{-}OH]^+$: 385.1410 found 385.1409; SFC Conditions: 10% IPA, 2.5 mL/min, Chiralpak IC column, λ = 210 nm, t_R (min): minor = 3.47, major = 4.28.

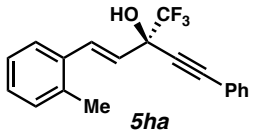


5ga

(*R*,*E*)-5-phenyl-1-(*m*-tolyl)-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ga)

Product **5ga** was prepared using general procedure and purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (61.2 mg, 97% yield); 90% ee, $[\alpha]_D^{25}$ +8.5 (*c* 0.88, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, J = 8.0, 1.6 Hz, 2H), 7.43 – 7.31 (m, 3H), 7.30 – 7.21 (m, 3H), 7.17 (d, J = 15.8 Hz, 1H), 7.13 (d, J =

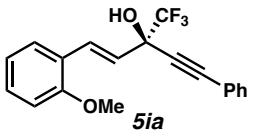
6.8 Hz, 1H), 6.32 (d, *J* = 15.8 Hz, 1H), 2.93 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.5, 135.9, 135.2, 132.2, 129.8, 129.7, 128.8, 128.6, 128.0, 126.1 (q, *J* = 229.9 Hz), 124.6, 122.2, 121.1, 88.7, 82.8, 72.5 (q, *J* = 32.8 Hz), 21.5; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.7; IR (Neat Film, NaCl) 3407, 2924, 1490, 1444, 1379, 1252, 1186, 1130, 1055, 1000, 966, 918, 844, 778, 756, 726, 689, 629 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{19}H_{14}F_3$ $[M\text{-}OH]^+$: 299.1042 found 299.1045; SFC Conditions: 15% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 254 nm, t_R (min): major = 3.83, minor = 4.20.



5ha

(*R*,*E*)-5-phenyl-1-(*o*-tolyl)-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ha)

Product **5ha** was prepared using general procedure and purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (57.8 mg, 92% yield); 86% ee, $[\alpha]_D^{25}$ +15.4 (*c* 0.87, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.46 (m, 4H), 7.45 – 7.33 (m, 3H), 7.25 – 7.16 (m, 3H), 6.24 (d, *J* = 15.7 Hz, 1H), 2.93 (s, 1H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.4, 134.5, 133.8, 132.2, 130.6, 129.7, 128.8, 128.6, 126.4, 126.3, 123.7, 123.6 (q, *J* = 285.3 Hz), 121.1, 88.7, 82.9, 72.6 (q, *J* = 32.8 Hz), 19.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.73; IR (Neat Film, NaCl) 3411, 3061, 2926, 1600, 1490, 1462, 1444, 1381, 1261, 1248, 1185, 1133, 1098, 1058, 1000, 967, 817, 753, 690, 628 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{19}H_{14}F_3$ $[M\text{-}OH]^+$: 299.1042 found 299.1043; SFC Conditions: 15% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 254 nm, t_R (min): major = 3.58, minor = 4.33.

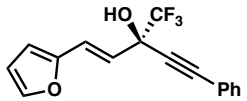


5ia

(*E*)-1-(2-methoxyphenyl)-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ia):

Product **5ia** was prepared using general procedure and purified by column chromatography (12% EtOAc in hexanes) to provide a colorless oil (59.6 mg, 92% yield); 86% ee, $[\alpha]_D^{25}$ +19.5 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.50 (m, 3H), 7.48 (d, *J* = 7.7 Hz, 1H), 7.43 – 7.33 (m, 3H), 7.33 – 7.27 (m, 1H), 6.96 (t, *J* =

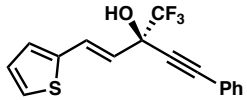
7.5 Hz, 1H), 6.91 (d, *J* = 8.3 Hz, 1H), 6.43 (d, *J* = 15.9 Hz, 1H), 3.88 (s, 3H), 2.88 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.5, 132.2, 131.0, 130.1, 129.6, 128.6, 128.1, 123.6 (q, *J* = 285.2 Hz), 124.2, 123.0, 121.3, 120.8, 111.4, 111.2, 88.6, 83.0, 72.8 (q, *J* = 32.7 Hz), 55.7; ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.66; IR (Neat Film, NaCl) 3429, 2940, 2360, 2237, 1599, 1490, 1465, 1248, 1185, 1136, 1103, 1048, 971, 754, 690 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{19}H_{14}F_3O$ $[M\text{-}OH]^+$: 315.09860 found 315.09993; SFC Conditions: 15% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 254 nm, t_R (min): major = 4.95, minor = 5.67.



5ja

(*R*,*E*)-1-(4-bromophenyl)-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ja):

Product **5ja** was prepared using general procedure and purified by column chromatography (15% Et_2O in hexanes) to provide a colorless oil (52 mg, 89% yield); 90% ee, $[\alpha]_D^{25}$ –7.42 (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.2 Hz, 2H), 7.45 – 7.32 (m, 4H), 6.99 (d, *J* = 15.6 Hz, 1H), 6.42 (m, 2H), 6.29 (d, *J* = 15.6 Hz, 1H), 2.85 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.1, 143.3, 132.2, 129.7, 128.6, 123.5 (q, *J* = 285.3 Hz), 123.5, 121.0, 120.6, 111.8, 111.2, 88.6, 82.6, 72.2 (q, *J* = 33.1 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.78; IR (Neat Film, NaCl) 3429, 3060, 2926, 1661, 1600, 1564, 1491, 1445, 1400, 1300, 1266, 1249, 1188, 1154, 1127, 1056, 1016, 1000, 960, 928, 884, 844, 804, 757, 742, 728, 690, 673, 654, 612 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{16}H_{10}F_3O$ $[M\text{-}OH]^+$: 275.0678 found 275.0668; SFC Conditions: 15% IPA, 2.5 mL/min, Chiralpak OJ-H column, λ = 210 nm, t_R (min): major = 3.12, minor = 3.86.

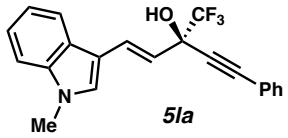


5ka

(*R*,*E*)-5-phenyl-1-(thiophen-2-yl)-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ka):

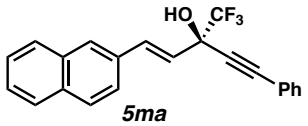
Product **5ka** was prepared using general procedure and purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (51.8 mg, 84% yield); 90% ee, $[\alpha]_D^{25}$ +25.8 (*c* 0.88, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, *J* = 8.0,

1.5 Hz, 2H), 7.45 – 7.30 (m, 5H), 7.28 (dd, *J* = 4.8, 1.5 Hz, 1H), 7.21 (d, *J* = 15.7 Hz, 1H), 6.20 (d, *J* = 15.7 Hz, 1H), 2.94 (s, 1H);[13]C NMR (101 MHz, $CDCl_3$) δ 137.9, 132.2, 129.8, 129.7, 128.6, 126.7, 125.2, 125.0, 122.1, 121.0, 88.7, 82.7, 72.4 (q, *J* = 32.9 Hz); [19]F NMR (282 MHz, $CDCl_3$) δ –80.78; IR (Neat Film, NaCl) 3406, 2924, 1656, 1491, 1444, 1358, 1308, 1249, 1186, 1126, 1054, 1000, 964, 868, 775, 757, 725, 690, 606 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{16}H_{10}F_3S$ $[M\text{-}OH]^+$: 291.045 found 291.045; SFC Conditions: 15% IPA, 2.5 mL/min, Chiralpak OD-H column, λ = 210 nm, t_R (min): major = 4.20, minor = 4.50.



5la

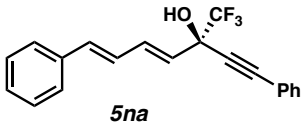
(*R*,*E*)-1-(1-methyl-1*H*-indol-3-yl)-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5la): Product **5la** was prepared using general procedure and purified by column chromatography (30% EtOAc in hexanes) to provide a yellow oil (32.2 mg, 63% yield); 96% ee, $[\alpha]_D^{25}$ +4.2 (*c* 0.95 $CHCl_3$); [1]H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.8 Hz, 1H), 7.48 (d, *J* = 8.1 Hz, 2H), 7.30 (dd, *J* = 11.5, 4.2 Hz, 4H), 7.26 – 7.19 (m, 2H), 7.18 – 7.12 (m, 2H), 6.22 (d, *J* = 15.8 Hz, 1H), 3.71 (s, 3H); [13]C NMR (101 MHz, $CDCl_3$) δ 137.8, 132.2, 130.3, 129.5, 128.8, 128.6, 126.1, 122.6, 121.4, 120.6, 120.3, 117.6, 111.9, 88.6, 83.3, 73.13 (q, *J* = 32.8 Hz), 33.1, 29.9; [19]F NMR (282 MHz, $CDCl_3$) δ –80.83; IR (Neat Film, NaCl) 3382, 2922, 1651, 1535, 1491, 1444, 1378, 1333, 1255, 1184, 1125, 1060, 960, 787, 758, 741, 691, 645 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{21}H_{15}F_3N$ $[M\text{-}OH]^+$: 338.1151 found 338.1151; SFC Conditions: 30% IPA, 2.5 mL/min, Chiralpak AD-H column, λ = 254 nm, t_R (min): major = 6.29, minor = 7.80.



5ma

(*R*, *E*)-1-(naphthalen-2-yl)-5-phenyl-3-(trifluoromethyl)pent-1-en-4-yn-3-ol (5ma): Product **5ma** was prepared using general procedure and purified by column chromatography (10% EtOAc in hexanes) to provide a colorless oil (67.7 mg, 96%

yield); 92% ee, $[\alpha]_D^{25}$ +13.8 (*c* 1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.80 (m, 4H), 7.66 (dd, *J* = 8.7, 1.6 Hz, 1H), 7.58 (d, *J* = 6.3 Hz, 2H), 7.50 (d, *J* = 9.4 Hz, 2H), 7.46 – 7.34 (m, 4H), 6.47 (d, *J* = 15.8 Hz, 1H), 2.98 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.9, 133.7, 133.5, 132.7, 132.2, 129.7, 128.62, 128.60, 128.3, 128.2, 127.9, 126.7, 126.6, 123.2 (q, *J* = 110.0 Hz), 88.8, 82.8, 72.5 (q, *J* = 32.9 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.62; IR (Neat Film, NaCl) 3400, 3057, 1652, 1491, 1444, 1361, 1252, 1186, 1126, 1054, 965, 894, 843, 810 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{18}H_{11}BrF_3O$ $[M\text{-}OH]^+$: 335.1042 found 335.1043; SFC Conditions: 8% IPA, 2.5 mL/min, Chiralpak IC column, λ = 210 nm, t_R (min): major = 6.56, minor = 7.11.



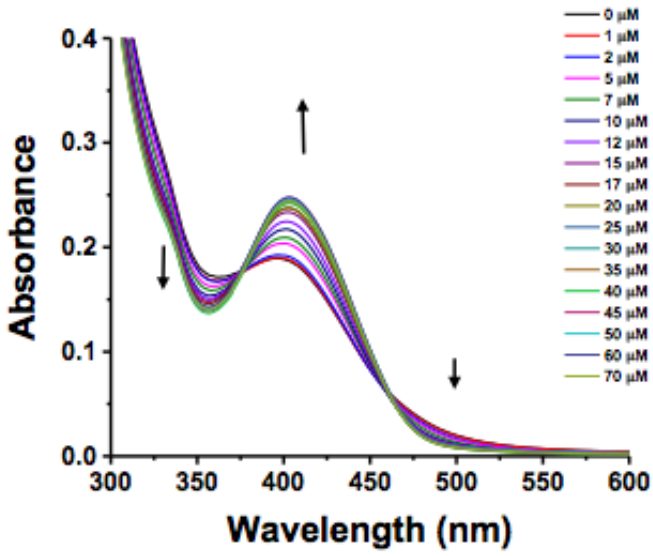
5na

(*R*,4*E*,6*E*)-1,7-diphenyl-3-(trifluoromethyl)hepta-4,6-dien-1-yn-3-ol (5na): Product **5na** was prepared using general procedure and purified by column chromatography (8% EtOAc in hexanes) to provide a colorless oil (28.1 mg, 43% yield); 91% ee, $[\alpha]_D^{25}$ +11.3 (*c* 0.67, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.47 – 7.26 (m, 8H), 6.99 (dd, *J* = 14.9, 10.4 Hz, 1H), 6.86 (dd, *J* = 15.4, 10.5 Hz, 1H), 6.74 (d, *J* = 15.5 Hz, 1H), 5.95 (d, *J* = 14.9 Hz, 1H), 2.85 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.6, 136.5, 135.9, 132.2, 129.7, 128.9, 128.6, 128.4, 126.9, 126.7, 125.6, 123.5 (q, *J* = 285.3 Hz), 121.1, 88.6, 82.7, 72.3 (q, *J* = 33.0 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –80.78; IR (Neat Film, NaCl) 3396, 3027, 2922, 2850, 1644, 1491, 1447, 1253, 1186, 1127, 1051, 990, 974, 828, 756, 726, 690 cm^{-1}; HRMS (MM) *m/z* calc'd for $C_{20}H_{14}F_3$ $[M\text{-}OH]^+$: 311.1042 found 311.1037; SFC Conditions: 35% IPA, 2.5 mL/min, Chiralpak AD-H column, λ = 254 nm, t_R (min): major = 3.3, minor =4.4.

UV-Vis Data for Metal Titration

A sample solution containing **L7-Ni** (20 M) and different amounts of KO*t*-Bu (0, 1, 2, 5, 7, 10, 12, 15, 17, 20, 25, 30, 35, 40, 45, 50, 60, and 70 M) prepared in dry THF,

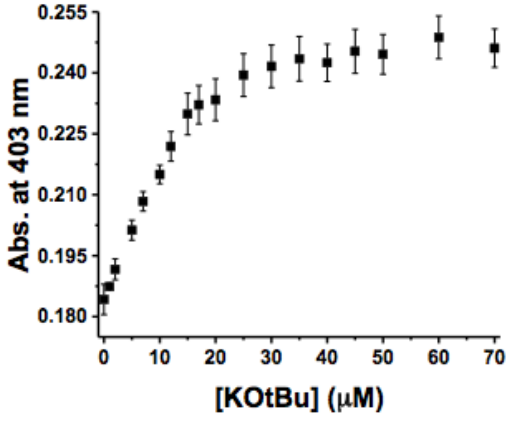
respectively. After incubation for 30 min at room temperature, UV-Vis spectra were recorded at 25 °C and each measurement was repeated thrice.



Isobestic point 1. 376 nm 2. 460 nm

Job plot

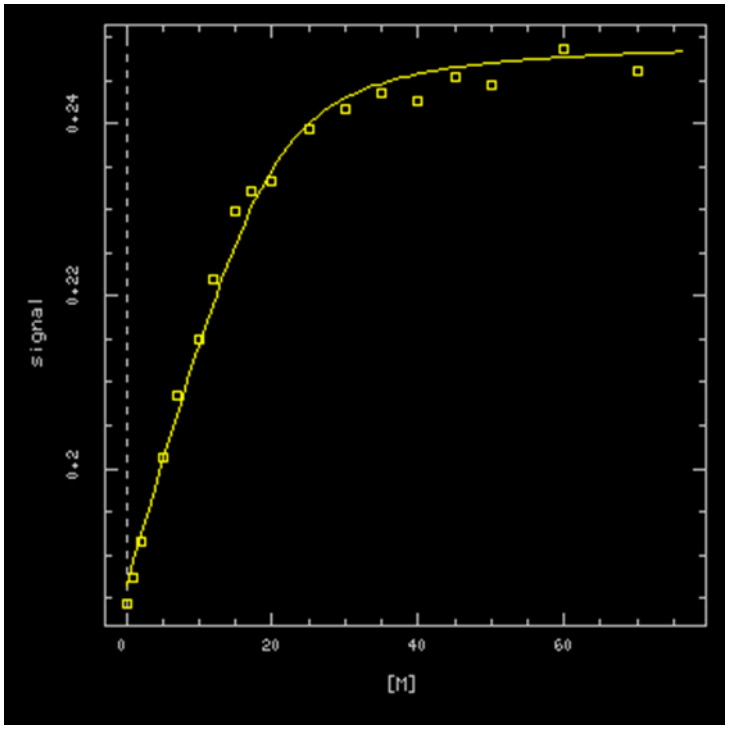
A solutions of **L7-Ni** (40 M) in THF were mixed with solutions of KOtBu in THF (40 M) at varying ratios. After incubation for 30 min at room temperature, UV-Vis spectra were recorded at 25 °C and each measurement was repeated thrice.



Association constant for the binding of L7-Ni and K+

The program DynaFit was used for non-linear regression fitting of the titration data based on absorbance changes observed at 403 nm. From the result of Job plot, the fitting of titration data were performed as a 1:1 binding mode. The DynaFit scripts for the binding models used are provided below.

```
[task]
  task = fit
  data = equilibria
[mechanism]
  L + M <==> LM : K1 association
[constants]
  K1 = 0.5?
[concentrations]
  L = 20
[responses]
  L = 0.0092?
  LM = 0.0125
[data]
  variable M
  file C:\./Users/skang/Desktop/dynafit4-win/DynaFit4/input/titration.txt
[output]
  directory C:\./Users/skang/Desktop/dynafit4-win/DynaFit4/output
[end]
```



The association constant for the binding of **L7-Ni** and K+ ion. $K_a = 6.6 \times 10^5\ M^{-1}$.

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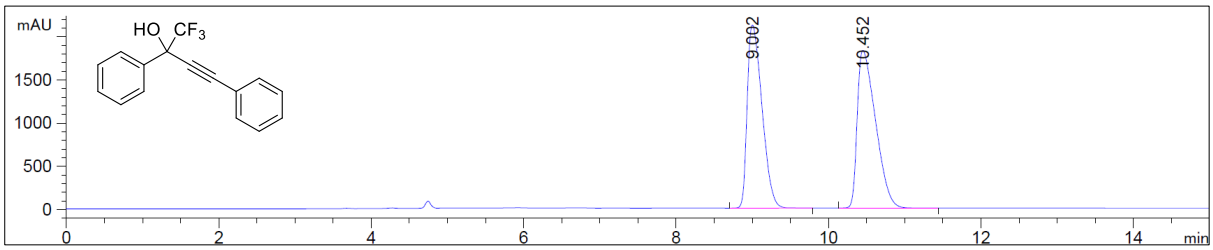
HPLC and SFC data for Trifluoromethyl Alcohol Products:

Racemic propargylic alcohols were prepared according to reported literature protocols.[13,14]

HPLC data for Aryl Trifluoromethyl Ketones:

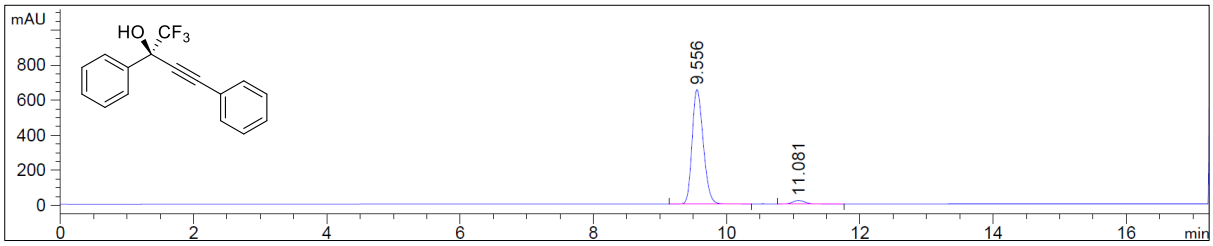
Racemic 3aa



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.002	BV	0.2129	2.85165e4	2119.92139	48.4080
2	10.452	VB	0.2642	3.03921e4	1825.36560	51.5920
Totals :				5.89086e4	3945.28699	

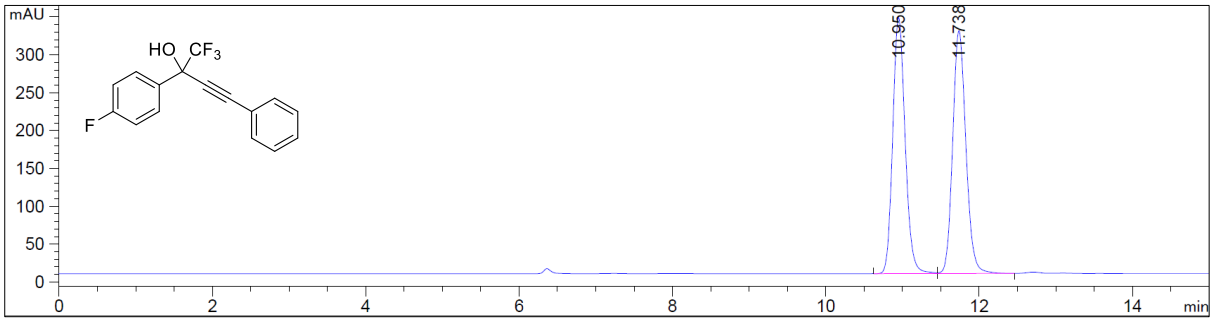
Enantioenriched 3aa



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.556	BB	0.1813	7575.20215	653.19965	96.6815
2	11.081	BB	0.1965	260.01358	20.42832	3.3185
Totals :				7835.21573	673.62797	

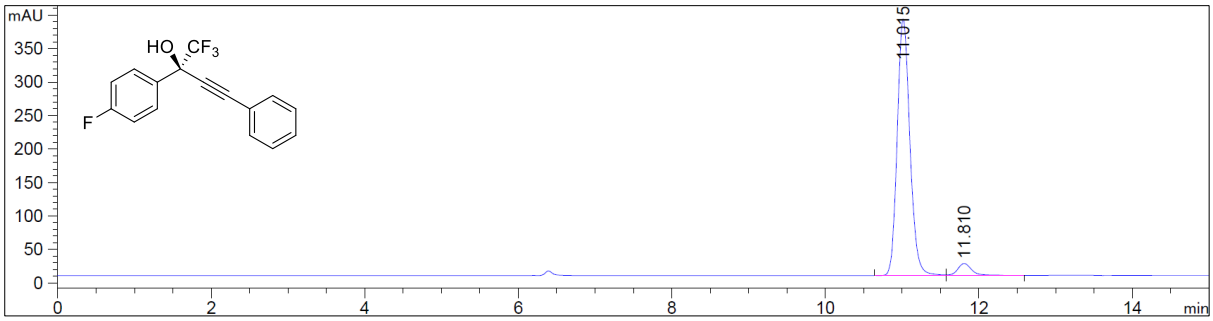
Racemic 3ba



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.950	BV	0.1752	3802.66162	338.24939	49.7561
2	11.738	VB	0.1838	3839.94897	320.52200	50.2439
Totals :				7642.61060	658.77139	

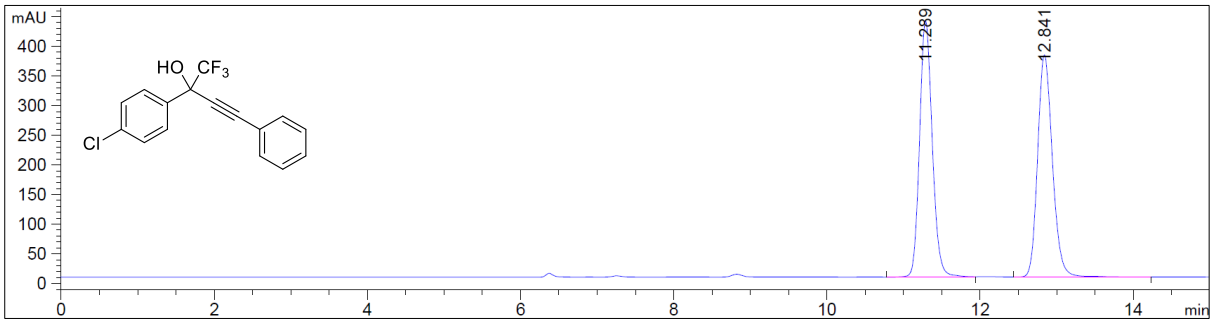
Enantioenriched 3ba



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.015	BV	0.1778	4391.09912	382.97543	95.0508
2	11.810	VB	0.1966	228.63799	17.94620	4.9492
Totals :				4619.73711	400.92163	

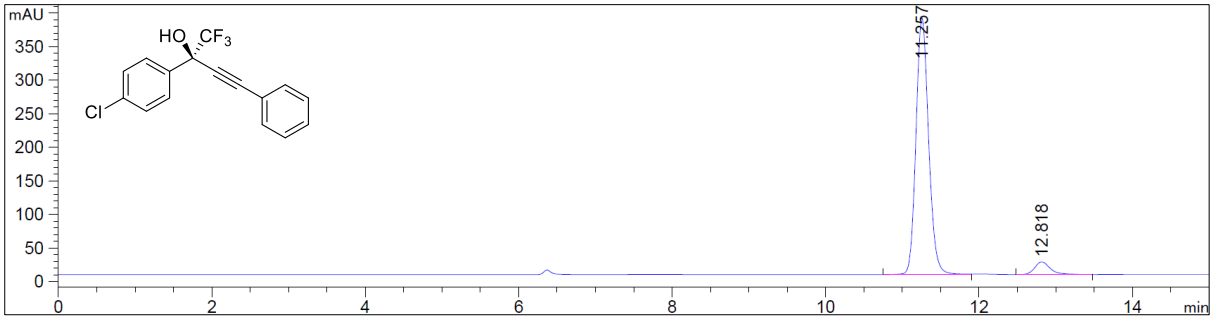
Racemic 3ca



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.289	BV	0.1786	4981.56006	431.86307	49.7393
2	12.841	VB	0.2088	5033.77783	374.57556	50.2607
Totals :				1.00153e4	806.43863	

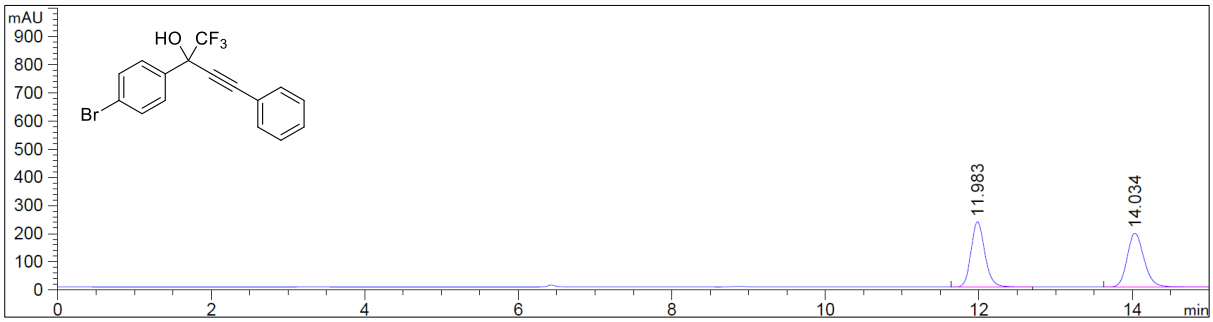
Enantioenriched 3ca



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.257	BV	0.1776	4376.18311	382.06796	94.2957
2	12.818	BV	0.2167	264.73257	18.74982	5.7043
Totals :				4640.91568	400.81779	

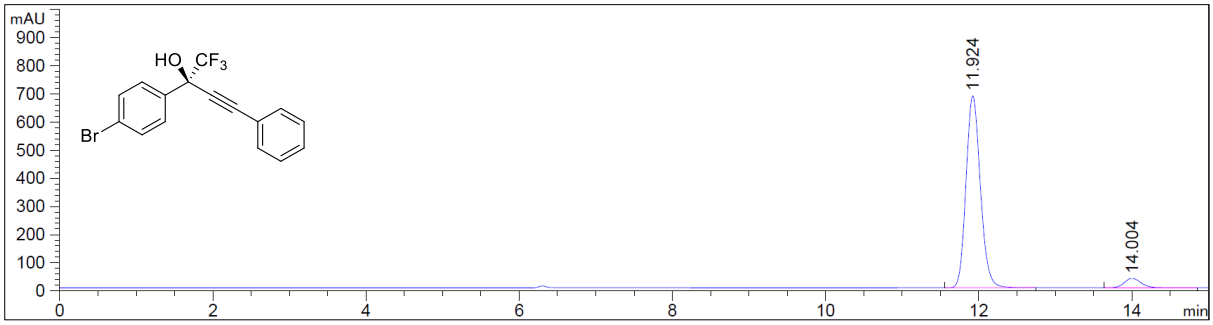
Racemic 3da



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.983	BB	0.1939	2885.78491	230.71150	49.9867
2	14.034	VB	0.2364	2887.32275	190.96884	50.0133
Totals :				5773.10767	421.68034	

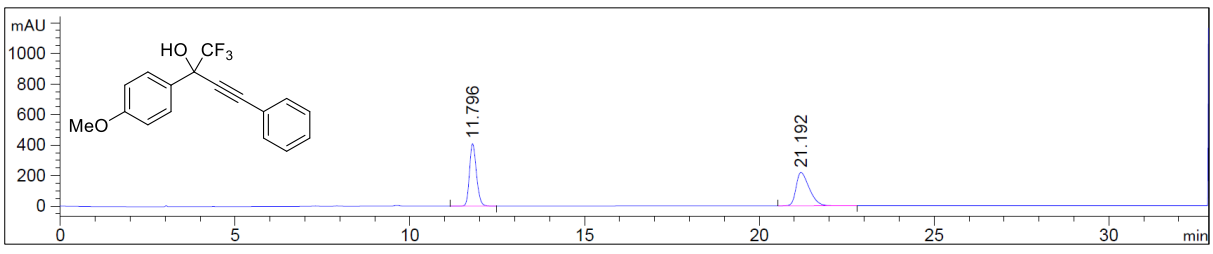
Enantioenriched 3da



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.924	BB	0.2017	8857.81738	681.41754	94.5631
2	14.004	BB	0.2339	509.27783	33.40222	5.4369
Totals :				9367.09521	714.81976	

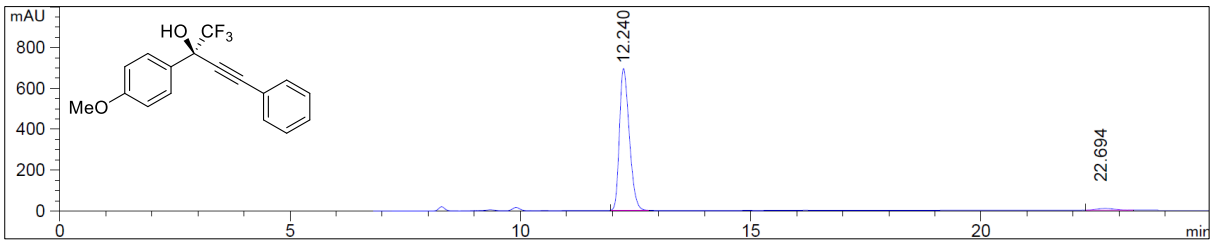
Racemic 3ea



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.796	BB	0.2126	5615.20752	408.01105	49.6589
2	21.192	BB	0.3985	5692.34326	218.12773	50.3411
Totals :				1.13076e4	626.13878	

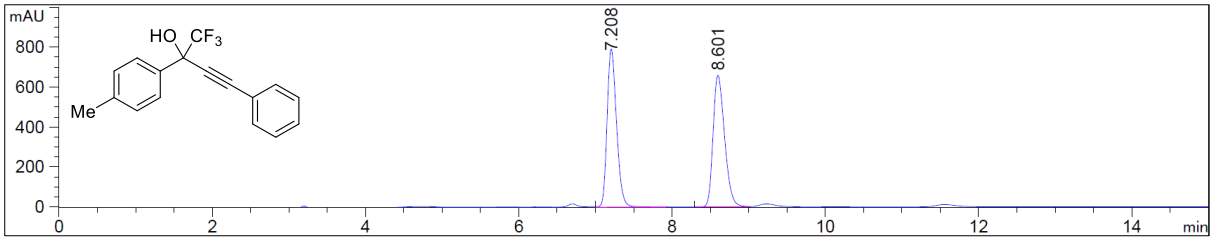
Enantioenriched 3ea



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.240	MM T	0.2478	1.03469e4	695.88904	97.9445
2	22.694	MM T	0.4294	217.14453	8.42797	2.0555
Totals :				1.05640e4	704.31701	

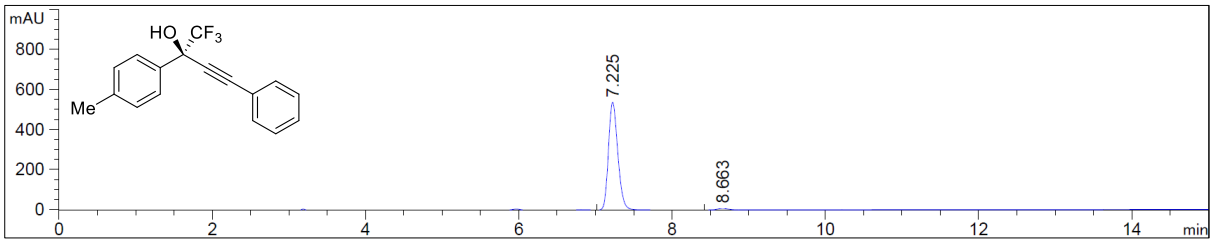
Racemic 3fa



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.208	VB	0.1318	6763.79443	790.41278	49.6057
2	8.601	BV	0.1660	6871.33252	657.30743	50.3943
Totals :				1.36351e4	1447.72021	

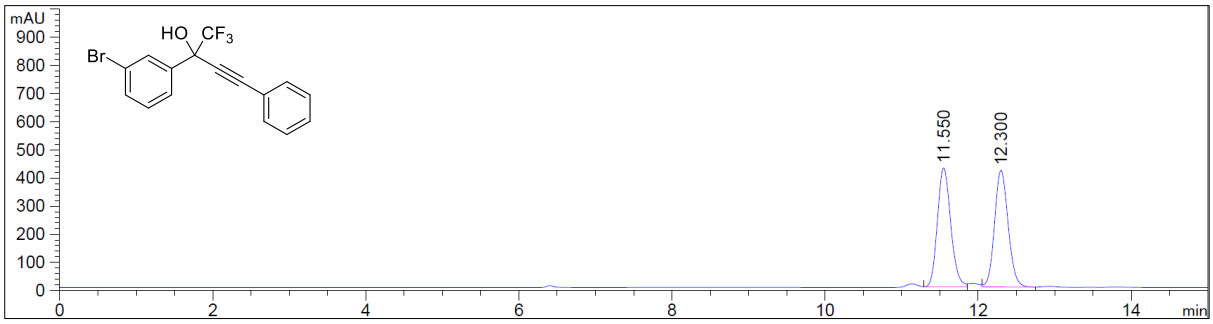
Enantioenriched 3fa



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.225	VB	0.1334	4580.36963	537.67596	98.4503
2	8.663	BB	0.1504	72.09988	7.35334	1.5497
Totals :				4652.46951	545.02931	

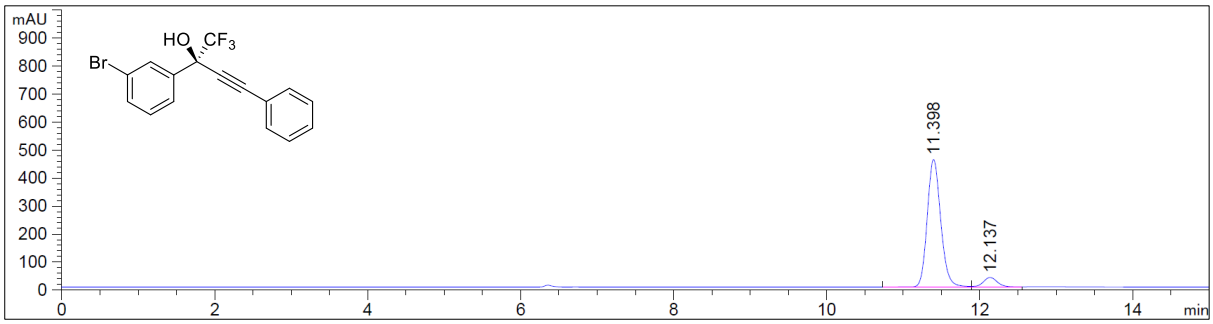
Racemic 3ga



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.550	BV	0.1889	5098.31445	422.24817	49.9791
2	12.300	VB	0.1898	5102.57080	413.86041	50.0209
Totals :				1.02009e4	836.10858	

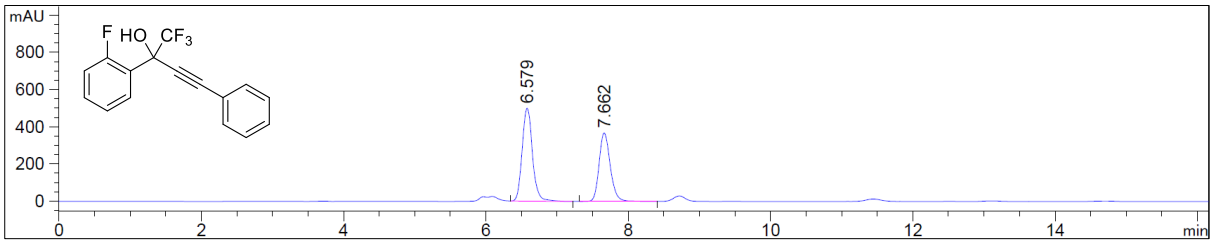
Enantioenriched 3ga



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.398	BV	0.1909	5573.68750	455.13403	92.6746
2	12.137	VV	0.1960	440.57138	34.26094	7.3254
Totals :				6014.25888	489.39498	

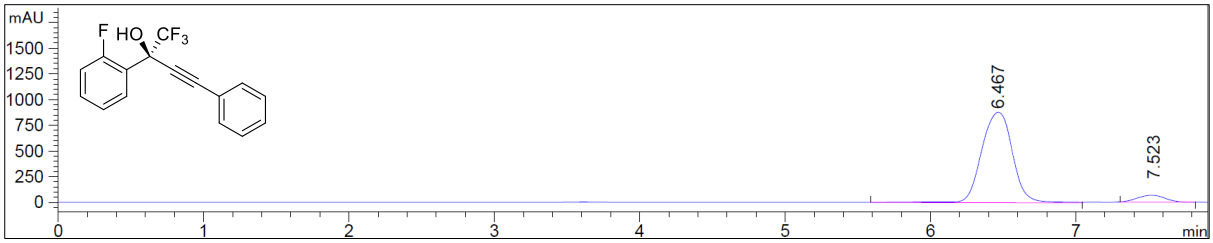
Racemic 3ha



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.579	BB	0.1575	5030.50830	499.48083	55.6302
2	7.662	BV	0.1671	4012.25366	368.32654	44.3698
Totals :				9042.76196	867.80737	

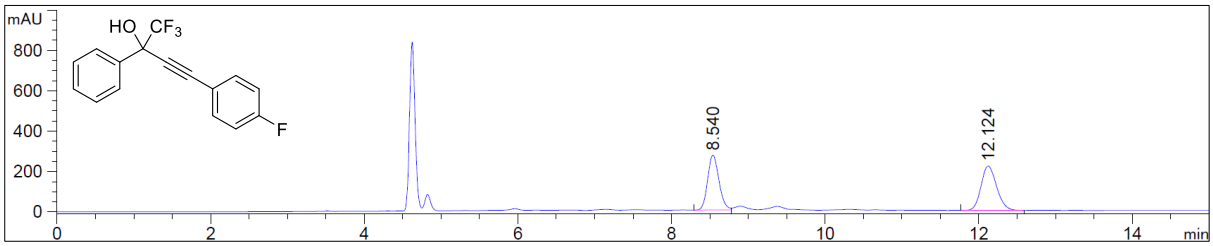
Enantioenriched 3ha



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.467	MM T	0.2475	1.30589e4	879.53400	93.6053
2	7.523	MM T	0.2215	892.12994	67.11796	6.3947
Totals :				1.39510e4	946.65195	

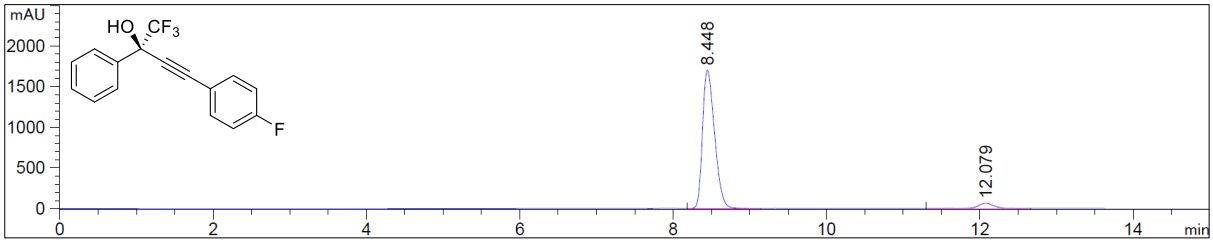
Racemic 3ab



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.540	BV	0.1593	2771.67334	271.11499	47.2416
2	12.124	BV	0.2162	3095.34009	219.89032	52.7584
Totals :				5867.01343	491.00531	

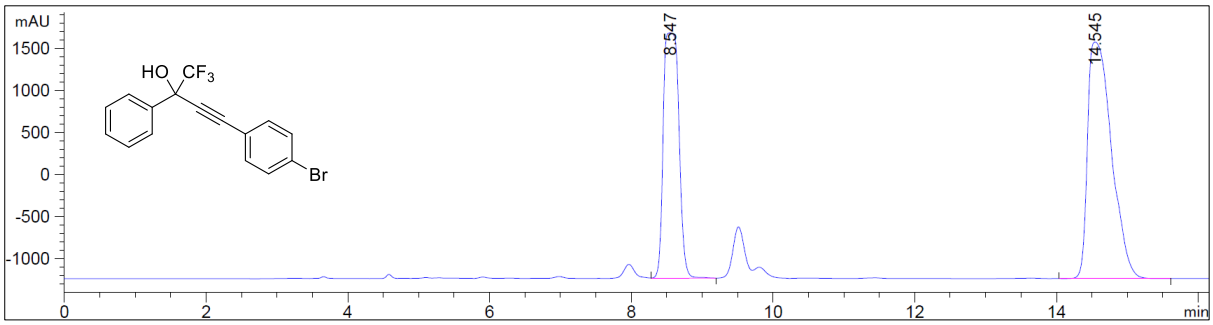
Enantioenriched 3ab



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.448	VV	0.1713	1.85722e4	1701.68274	95.4708
2	12.079	VV	0.2142	881.07227	63.35938	4.5292
Totals :				1.94533e4	1765.04211	

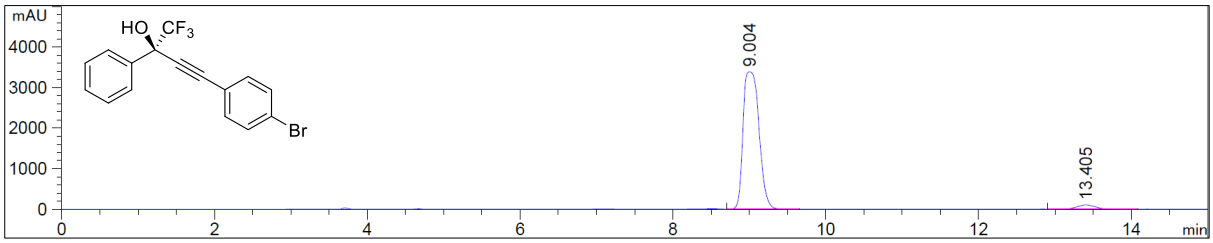
Racemic 3ac



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.547	VB	0.2459	4.44456e4	2914.54517	40.3203
2	14.545	BB	0.3686	6.57857e4	2811.18628	59.6797
Totals :				1.10231e5	5725.73145	

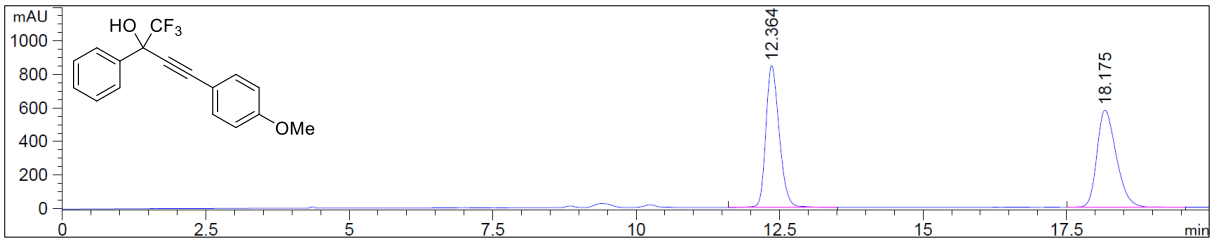
Enantioenriched 3ac



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.004	VV	0.2305	4.88785e4	3385.17822	96.8035
2	13.405	BB	0.2407	1614.00610	104.20264	3.1965
Totals :				5.04925e4	3489.38087	

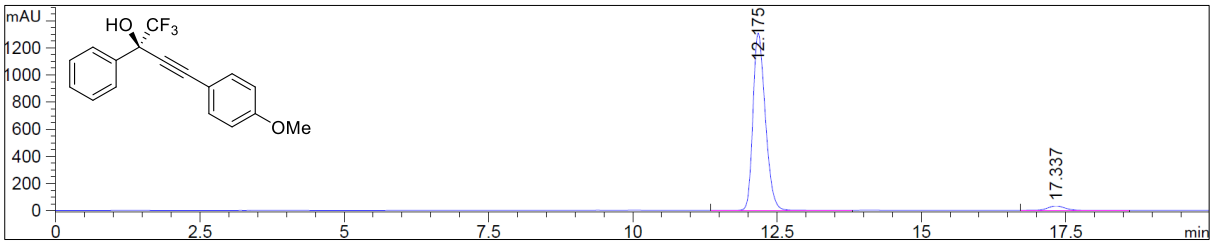
Racemic 3ad



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.364	BV	0.2492	1.37259e4	846.13507	50.3008
2	18.175	BB	0.3607	1.35618e4	580.18530	49.6992
Totals :				2.72877e4	1426.32037	

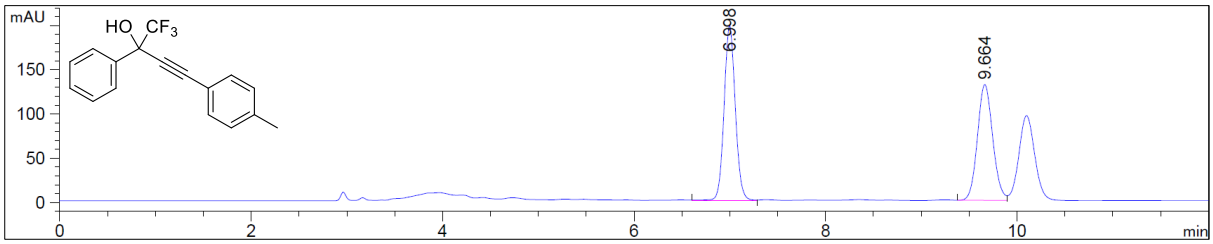
Enantioenriched 3ad



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.175	BV	0.2292	1.93857e4	1306.10767	96.6746
2	17.337	BB	0.3178	666.82703	32.70996	3.3254
Totals :				2.00525e4	1338.81763	

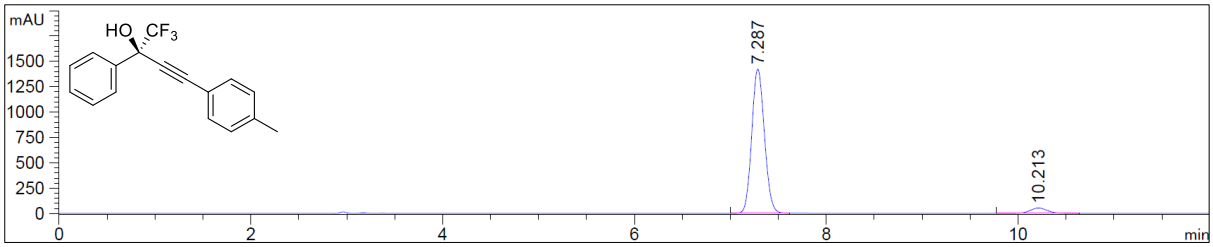
Racemic 3ae



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.998	VV	0.1267	1602.97388	197.45538	52.4931
2	9.664	BV	0.1732	1450.71130	130.96457	47.5069
Totals :				3053.68518	328.41995	

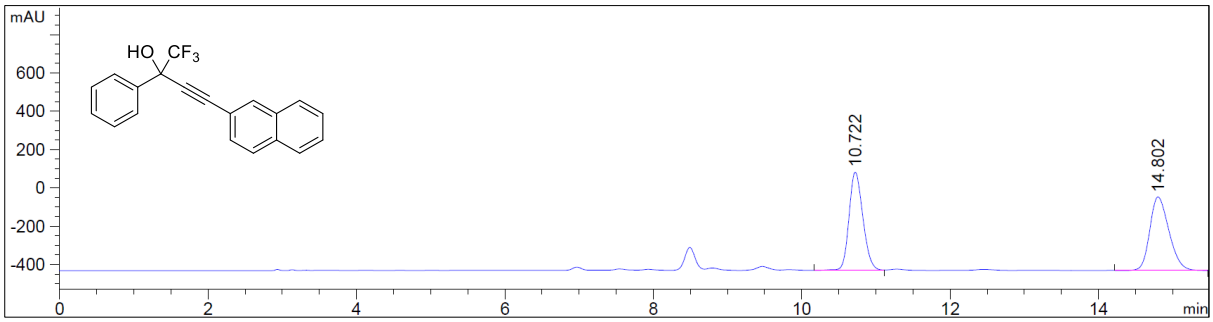
Enantioenriched 3ae



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.287	BV	0.1452	1.30561e4	1420.86133	95.2771
2	10.213	VV	0.1892	647.19513	52.72830	4.7229
Totals :				1.37033e4	1473.58963	

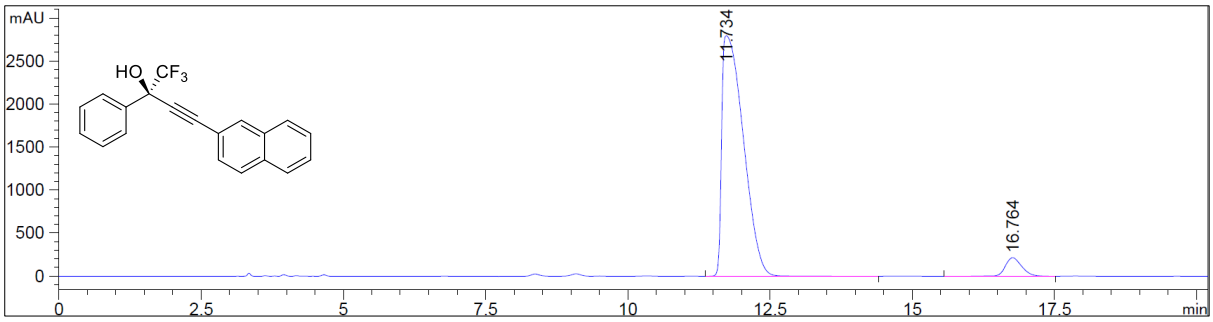
Racemic 3af



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.722	BV	0.2016	6669.84424	513.04938	49.2508
2	14.802	BBA	0.2769	6872.76123	383.90787	50.7492
Totals :				1.35426e4	896.95724	

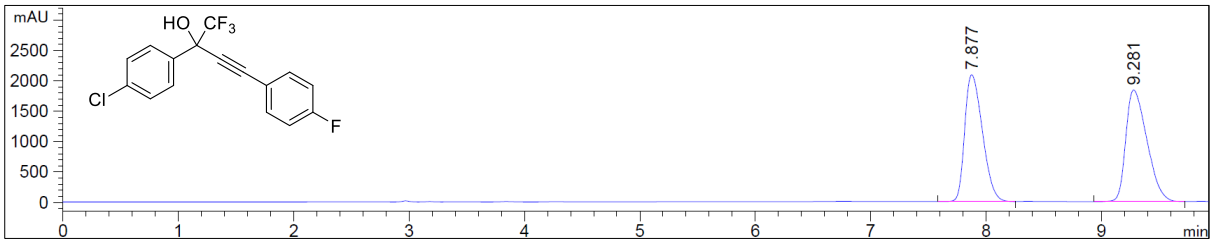
Enantioenriched 3af



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.734	BV	0.4197	7.37367e4	2795.73999	94.5055
2	16.764	BV	0.3041	4287.04248	215.23865	5.4945
Totals :				7.80238e4	3010.97864	

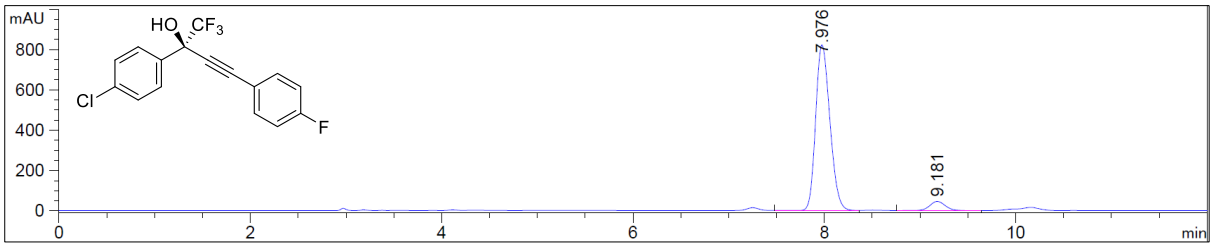
Racemic 3cb



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.877	VV	0.1687	2.23440e4	2090.93481	48.8914
2	9.281	BB	0.2003	2.33573e4	1837.32886	51.1086
Totals :				4.57013e4	3928.26367	

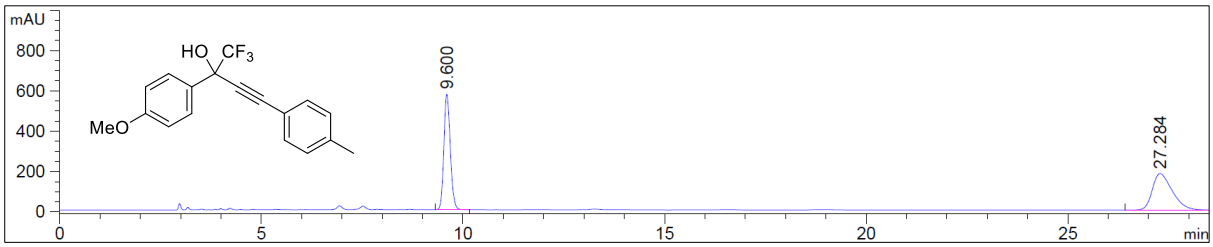
Enantioenriched 3cb



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.976	VV	0.1624	8487.25586	822.15076	94.3225
2	9.181	BV	0.1736	510.86423	45.28932	5.6775
Totals :				8998.12009	867.44008	

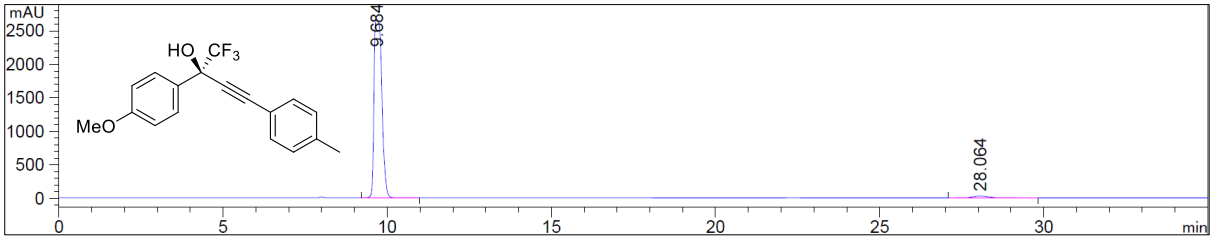
Racemic 3ee



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.600	VV	0.1712	6343.68701	572.83759	50.7212
2	27.284	BBA	0.5220	6163.29248	180.65030	49.2788
Totals :				1.25070e4	753.48788	

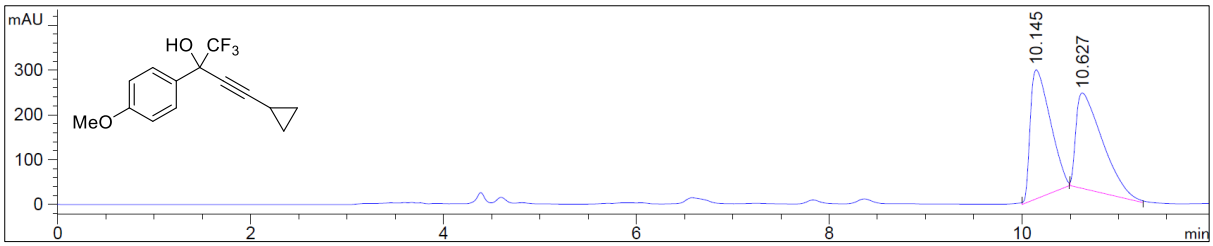
Enantioenriched 3ee



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.684	VV	0.2616	4.36613e4	2658.75806	97.9380
2	28.064	BB	0.5130	919.27368	27.71325	2.0620
Totals :				4.45806e4	2686.47131	

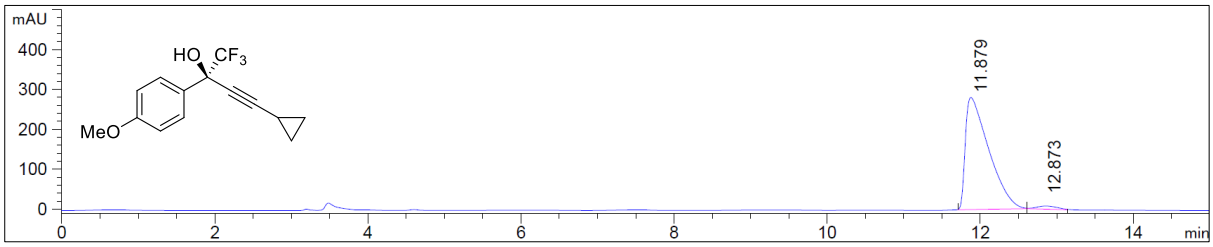
Racemic 3eg



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.145	MM T	0.2390	4132.77051	288.21365	50.1379
2	10.627	MM T	0.3213	4110.03418	213.22989	49.8621
Totals :				8242.80469	501.44354	

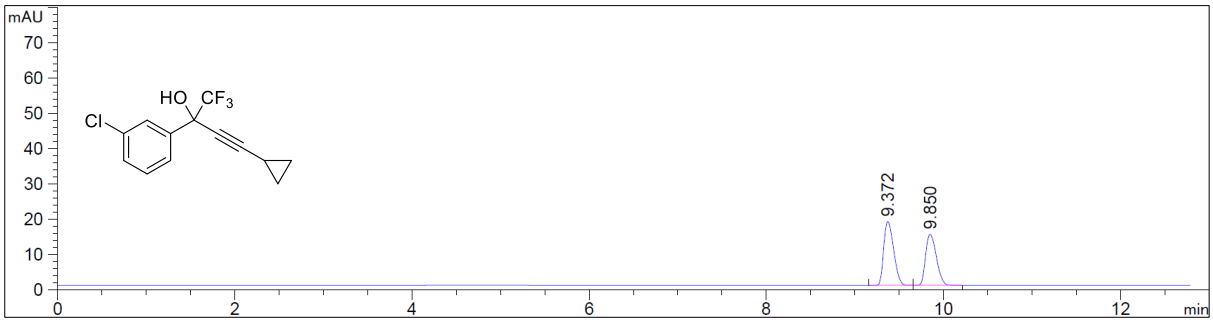
Enantioenriched 3eg



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.879	MM T	0.3534	5950.80176	280.63693	97.6302
2	12.873	MM T	0.3043	144.44800	7.91217	2.3698

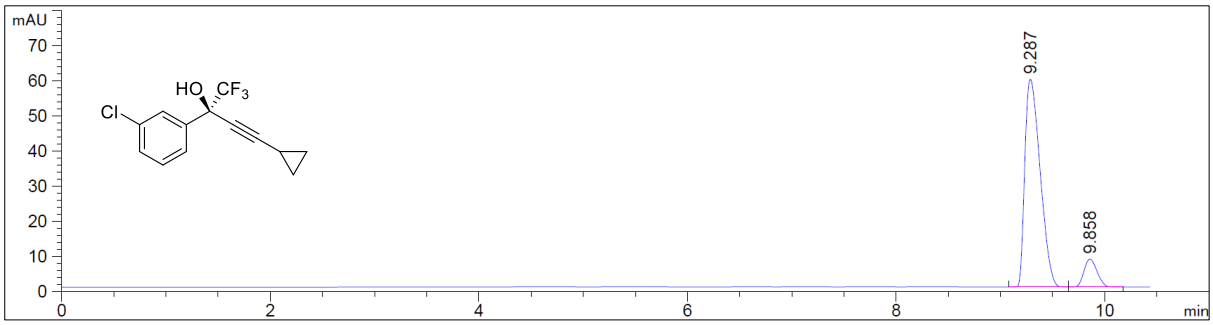
Racemic 3eh



Signal 1: MWD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.372	BB	0.1246	140.50111	18.09298	53.5707
2	9.850	BB	0.1342	121.77124	14.46669	46.4293
Totals :				262.27235	32.55967	

Enantioenriched 3eh

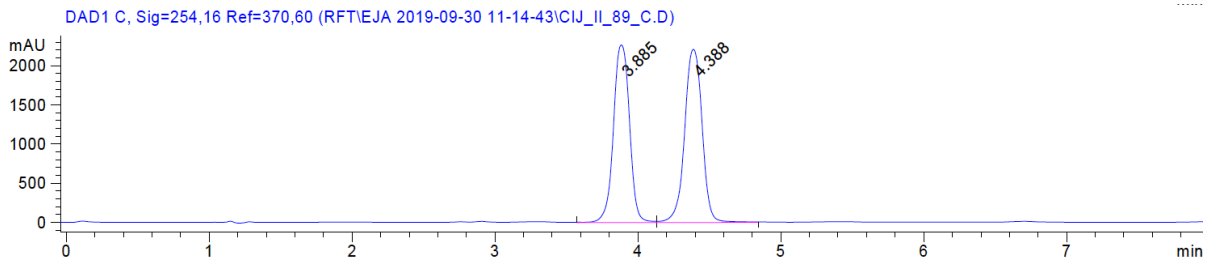


Signal 1: MWD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.287	BB	0.1629	603.86737	59.24526	89.7577
2	9.858	BBA	0.1350	68.90726	7.96194	10.2423
Totals :				672.77464	67.20720	

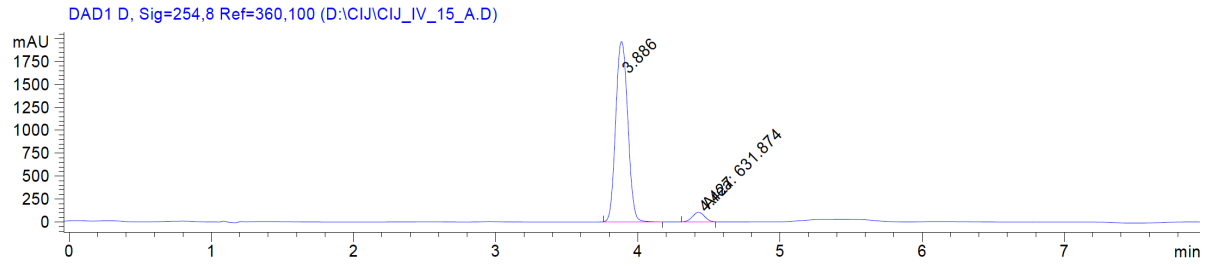
SFC data for Vinyl Trifluoromethyl Ketone Products:

Racemic 5aa



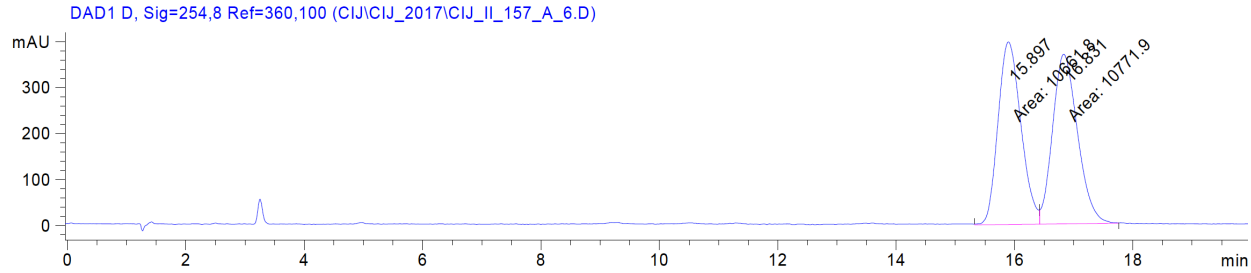
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.885	VV	0.1234	1.77626e4	2267.70264	48.5024
2	4.388	VB	0.1356	1.88595e4	2208.62769	51.4976

Enantioenriched 5aa



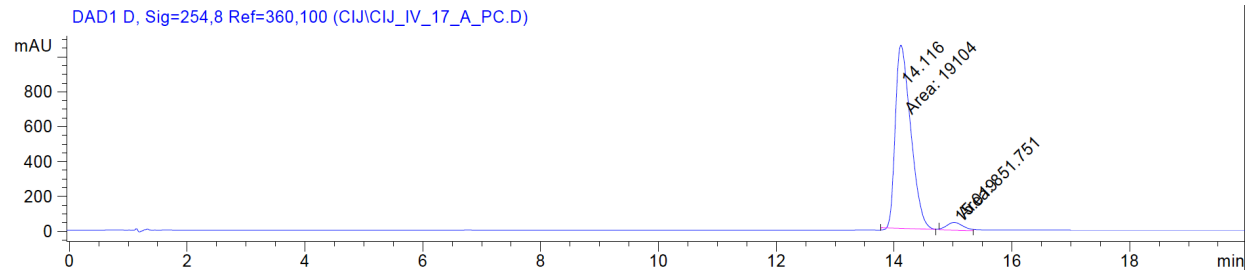
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.886	BB	0.0932	1.16488e4	1963.02271	94.8547
2	4.427	MM	0.0985	631.87439	106.89740	5.1453

Racemic 5ba



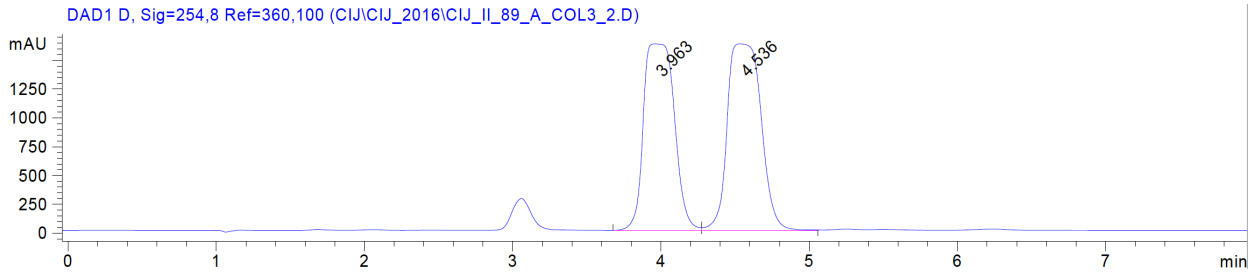
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.897	MF	0.4483	1.06618e4	396.33667	49.7431
2	16.831	FM	0.4871	1.07719e4	368.60919	50.2569

Enantioenriched 5ba



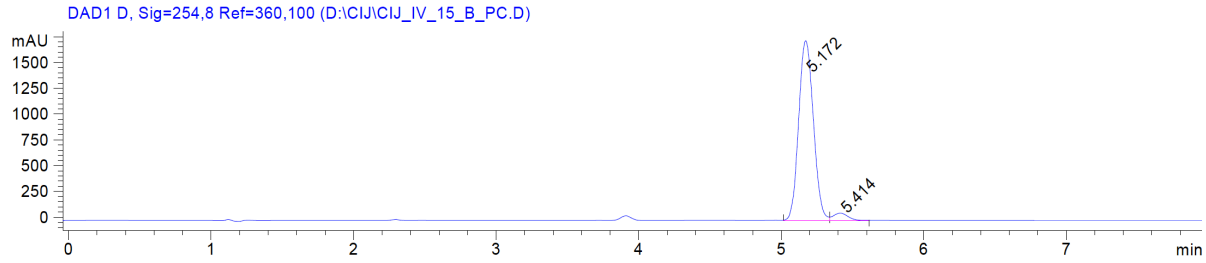
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.116	MM	0.3030	1.91040e4	1050.90735	95.7318
2	15.019	MM	0.3204	851.75140	44.30221	4.2682

Racemic 5ca



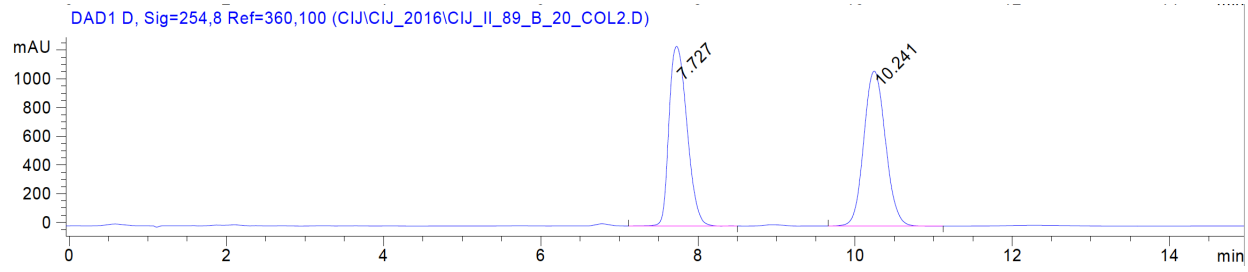
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.963	BV	0.2325	2.33956e4	1620.28918	47.7740
2	4.536	VV	0.2561	2.55758e4	1620.41089	52.2260

Enantioenriched 5ca



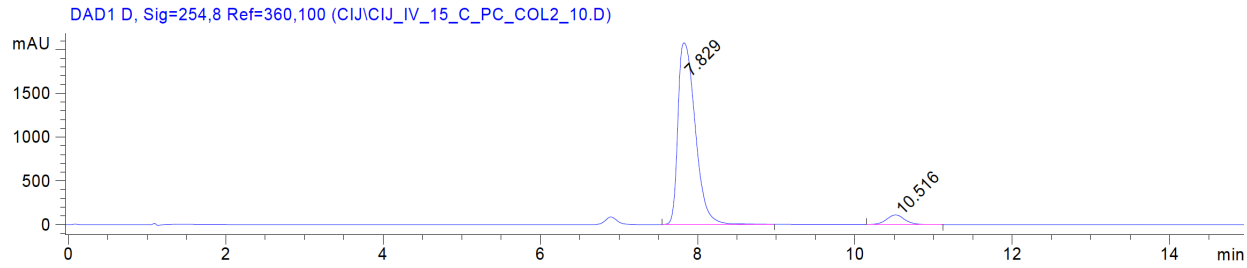
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.172	BV	0.1119	1.25042e4	1738.45972	96.0937
2	5.414	VB	0.1121	508.31110	70.54485	3.9063

Racemic 5da



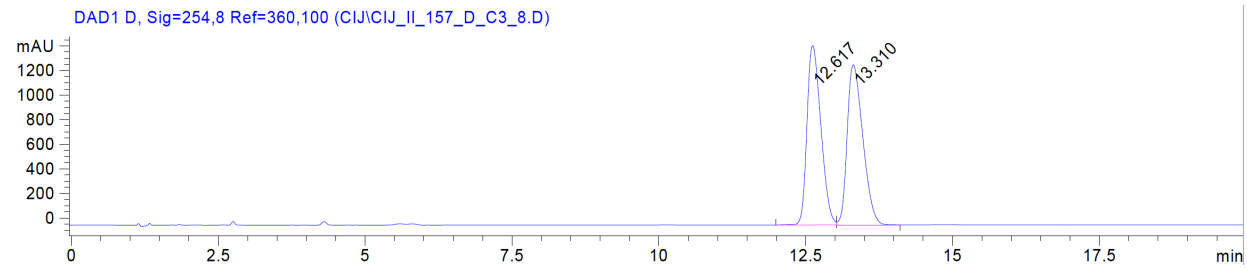
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.727	VB	0.2539	1.99510e4	1252.39722	49.0524
2	10.241	BB	0.3000	2.07219e4	1079.43774	50.9476

Enantioenriched 5da



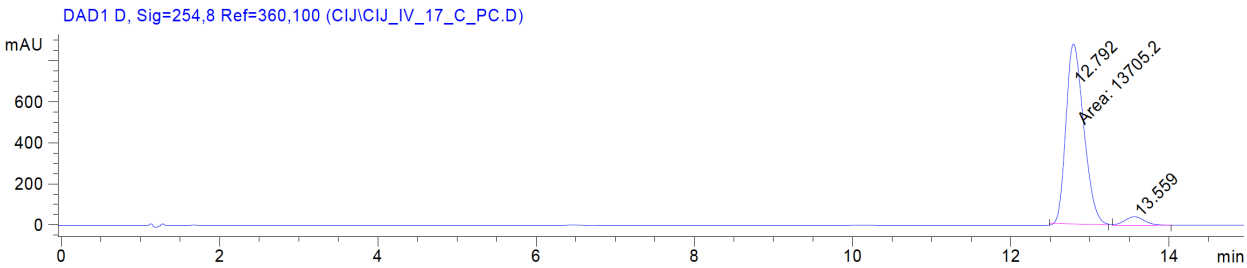
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.829	BB	0.2428	3.18149e4	2075.78711	94.7337
2	10.516	BB	0.2476	1768.60828	110.00776	5.2663

Racemic 5ea



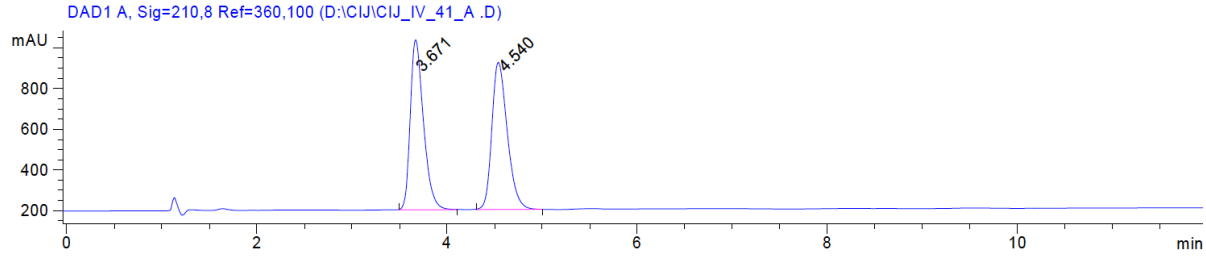
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.617	BV	0.2555	2.39572e4	1459.49976	49.8502
2	13.310	VB	0.2898	2.41012e4	1303.09802	50.1498

Enantioenriched 5ea



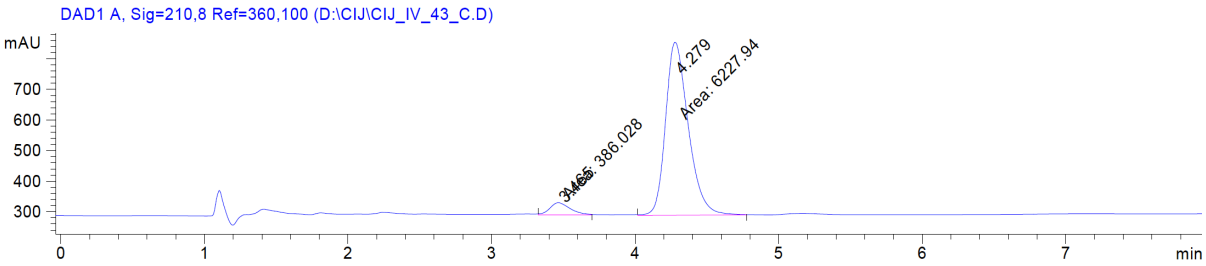
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.792	MM	0.2605	1.37052e4	876.92377	95.0351
2	13.559	VB	0.2631	715.99207	41.95095	4.9649

Racemic 5fa



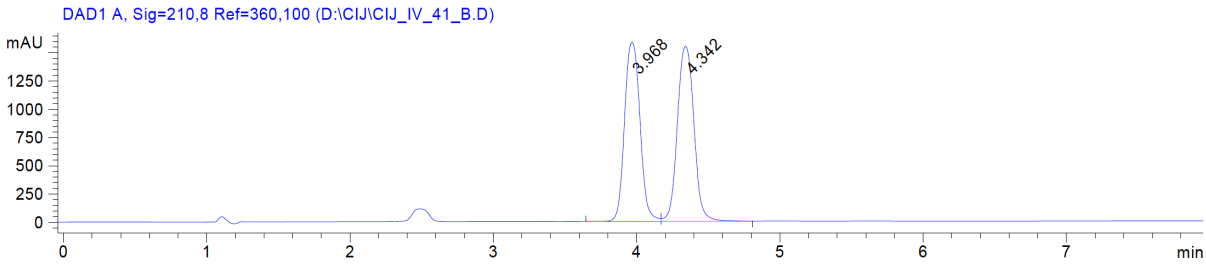
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.671	BB	0.1490	8086.00391	834.70844	49.7844
2	4.540	BB	0.1717	8156.03906	722.36987	50.2156

Enantioenriched 5fa



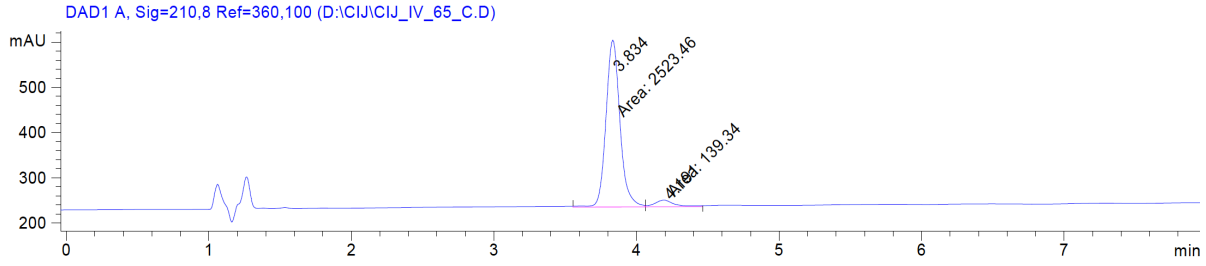
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.465	MM	0.1624	386.02798	39.61908	5.8366
2	4.279	MM	0.1831	6227.94189	566.79132	94.1634

Racemic 5ga



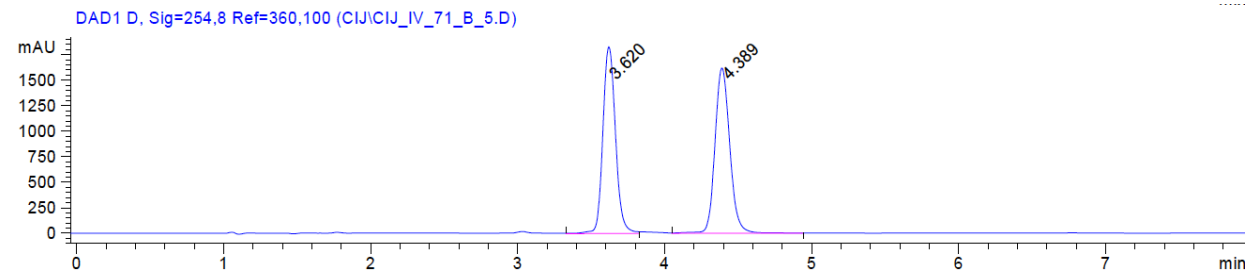
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.968	VV	0.1195	1.18848e4	1585.30981	48.8014
2	4.342	VV	0.1260	1.24686e4	1547.48511	51.1986

Enantioenriched 5ga



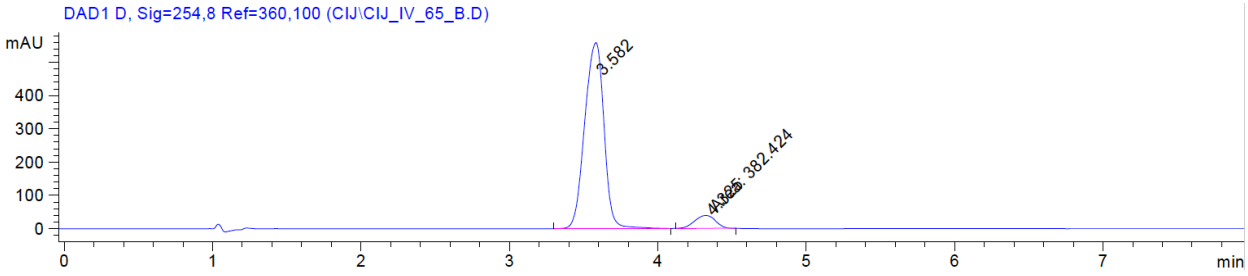
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.834	MF	0.1139	2523.45898	369.41278	94.7672
2	4.191	FM	0.1556	139.33974	14.92964	5.2328

Racemic 5ha



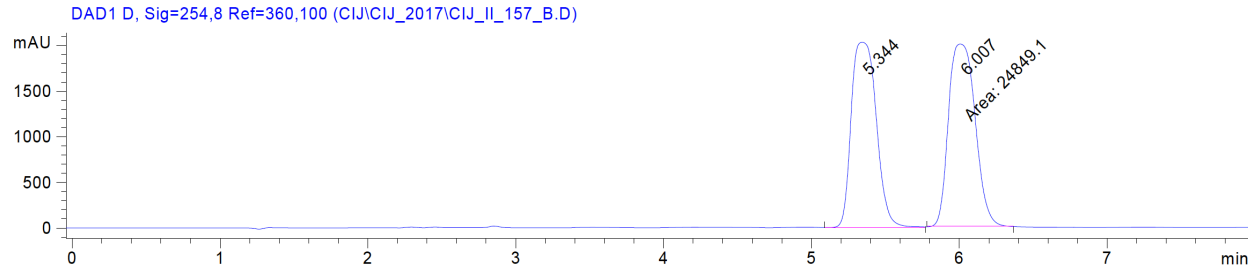
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.620	VV	0.0938	1.09643e4	1831.39258	48.9468
2	4.389	VV	0.1102	1.14361e4	1623.07410	51.0532

Enantioenriched 5ha



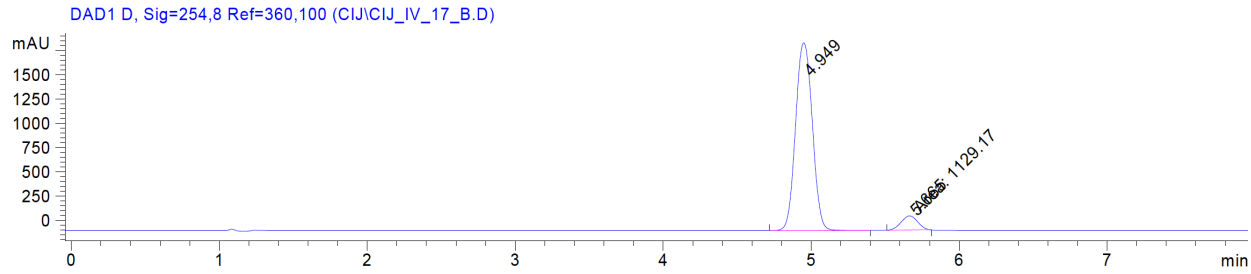
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.582	BB	0.1456	5064.83740	559.75128	92.9795
2	4.325	MM	0.1644	382.42398	38.77777	7.0205

Racemic 5ia



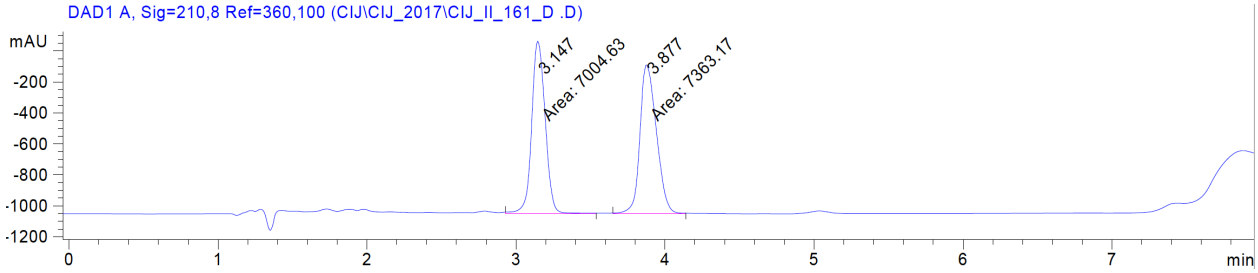
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.344	VV	0.1902	2.40919e4	2033.53210	49.2264
2	6.007	MM	0.2077	2.48491e4	1993.68933	50.7736

Enantioenriched 5ia



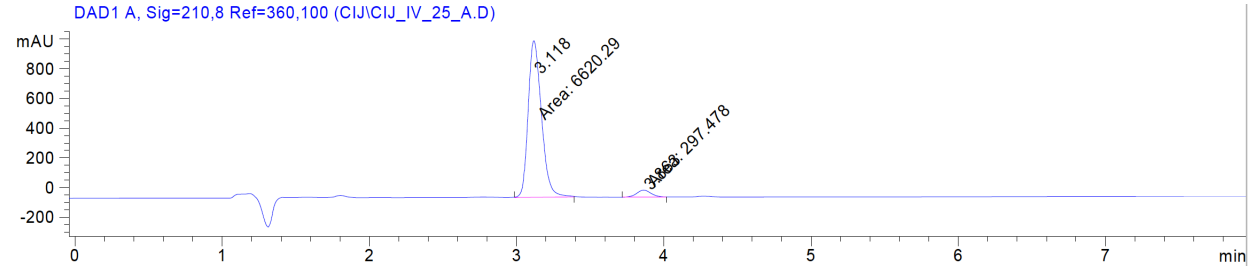
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.949	BB	0.1256	1.51929e4	1935.24060	93.0819
2	5.665	MM	0.1293	1129.16772	145.50713	6.9181

Racemic 5ja



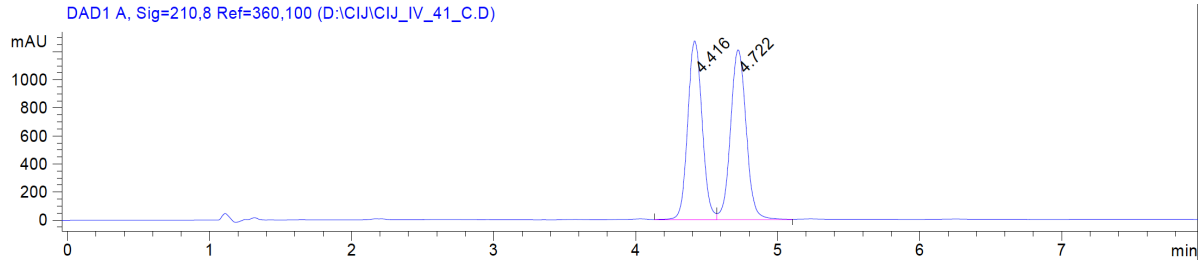
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.147	MM	0.1045	7004.62793	1117.27197	48.7523
2	3.877	MM	0.1277	7363.17383	961.19696	51.2477

Enantioenriched 5ja



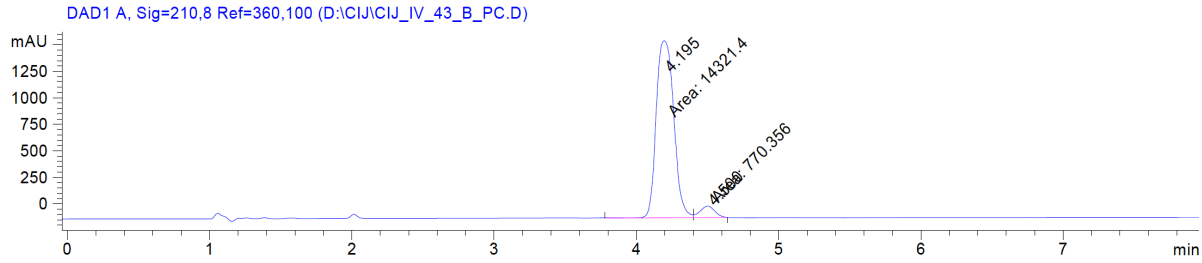
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.118	MM	0.1040	6620.28564	1060.52246	95.6998
2	3.863	MM	0.1079	297.47842	45.96951	4.3002

Racemic 5ka



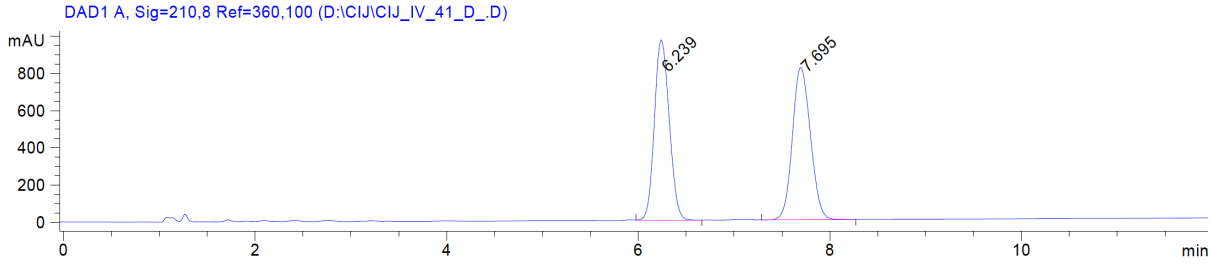
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.416	VV	0.1109	9051.17480	1273.78198	49.3289
2	4.722	VV	0.1177	9297.46680	1209.88831	50.6711

Enantioenriched 5ka



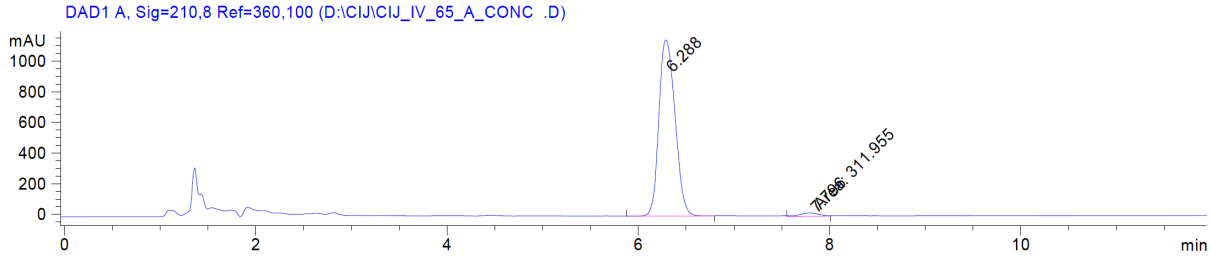
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.195	MF	0.1430	1.43214e4	1668.58252	94.8955
2	4.500	FM	0.1187	770.35632	108.20892	5.1045

Racemic 5la



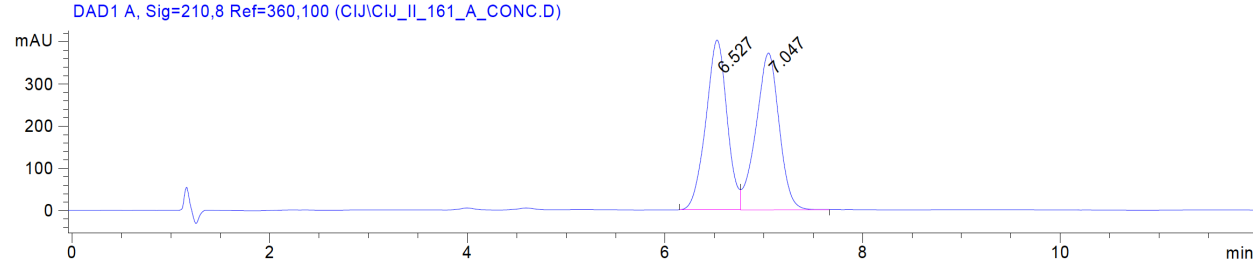
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.239	VB	0.1726	1.05089e4	968.62952	49.4613
2	7.695	VB	0.2052	1.07378e4	817.57489	50.5387

Enantioenriched 5la



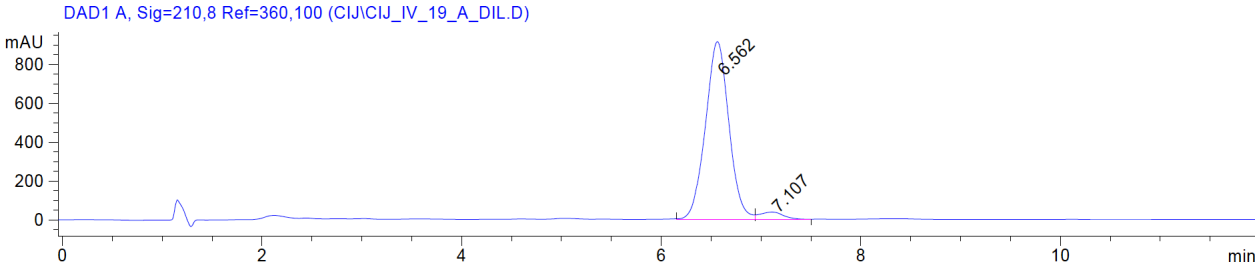
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.288	BB	0.1869	1.34793e4	1149.03198	97.7380
2	7.796	MM	0.2344	311.95532	22.18306	2.2620

Racemic 5ma



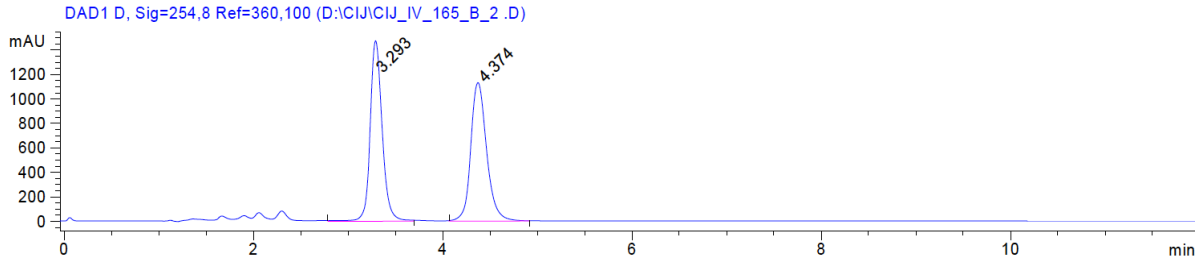
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.527	BV	0.2267	6022.24268	402.25772	49.7994
2	7.047	VB	0.2426	6070.75537	371.66992	50.2006

Enantioenriched 5ma



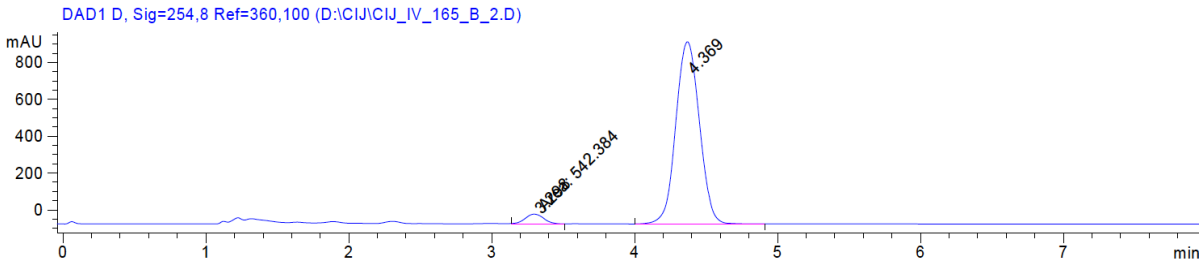
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.562	BV	0.2463	1.49132e4	914.24634	96.2033
2	7.107	VB	0.2379	588.55762	37.34389	3.7967

Racemic 5na



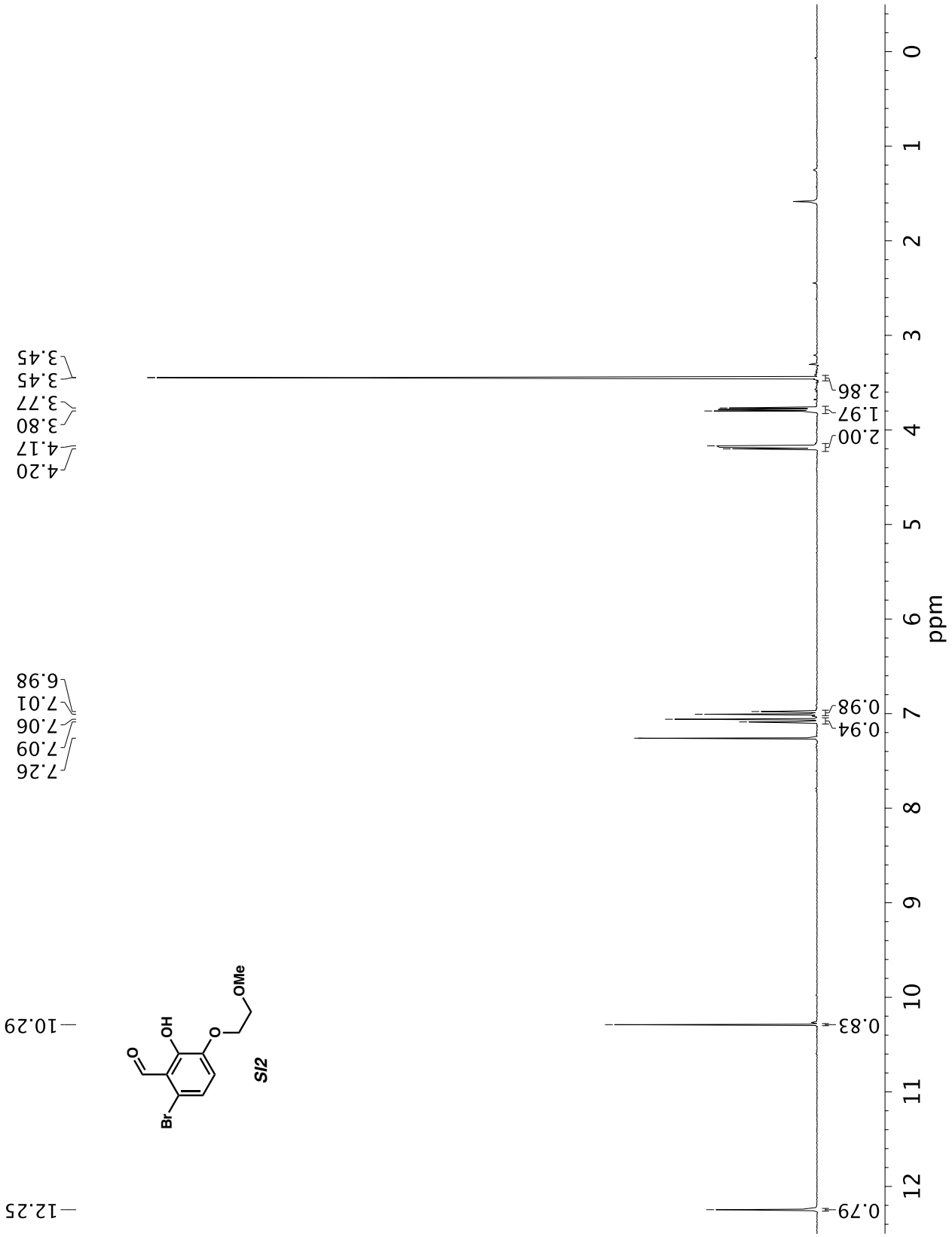
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.293	BB	0.1487	1.21569e4	1258.85498	47.9847
2	4.374	BB	0.1971	1.31781e4	1059.61987	52.0153

Enantioenriched 5na

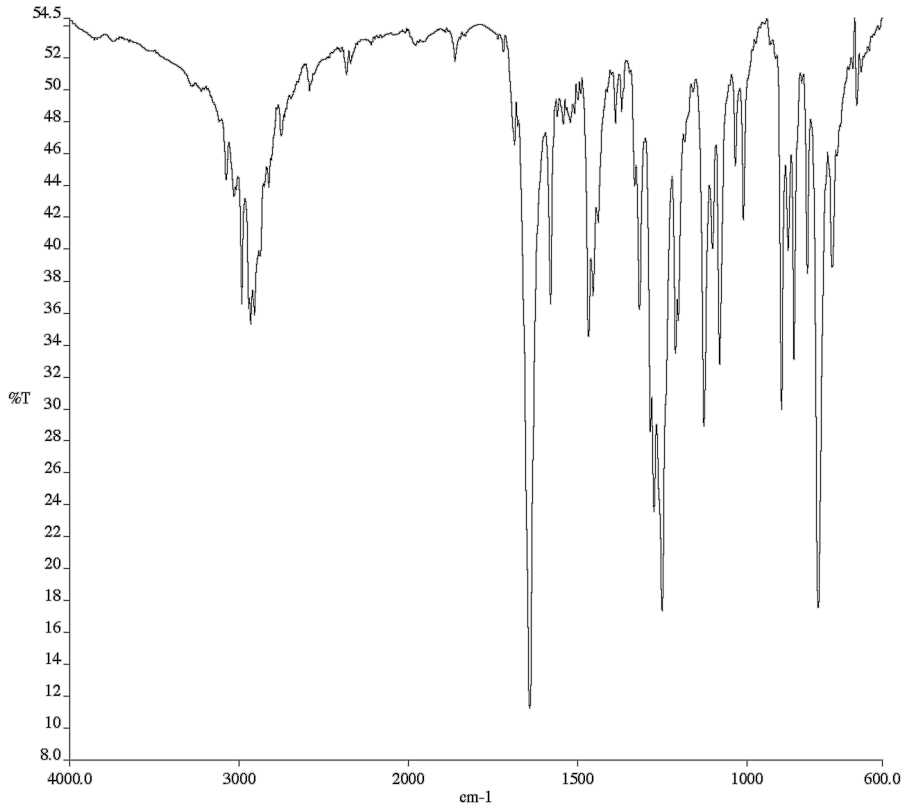


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.298	MM	0.1606	542.38373	56.29001	4.5637
2	4.369	BB	0.1819	1.13423e4	988.30817	95.4363

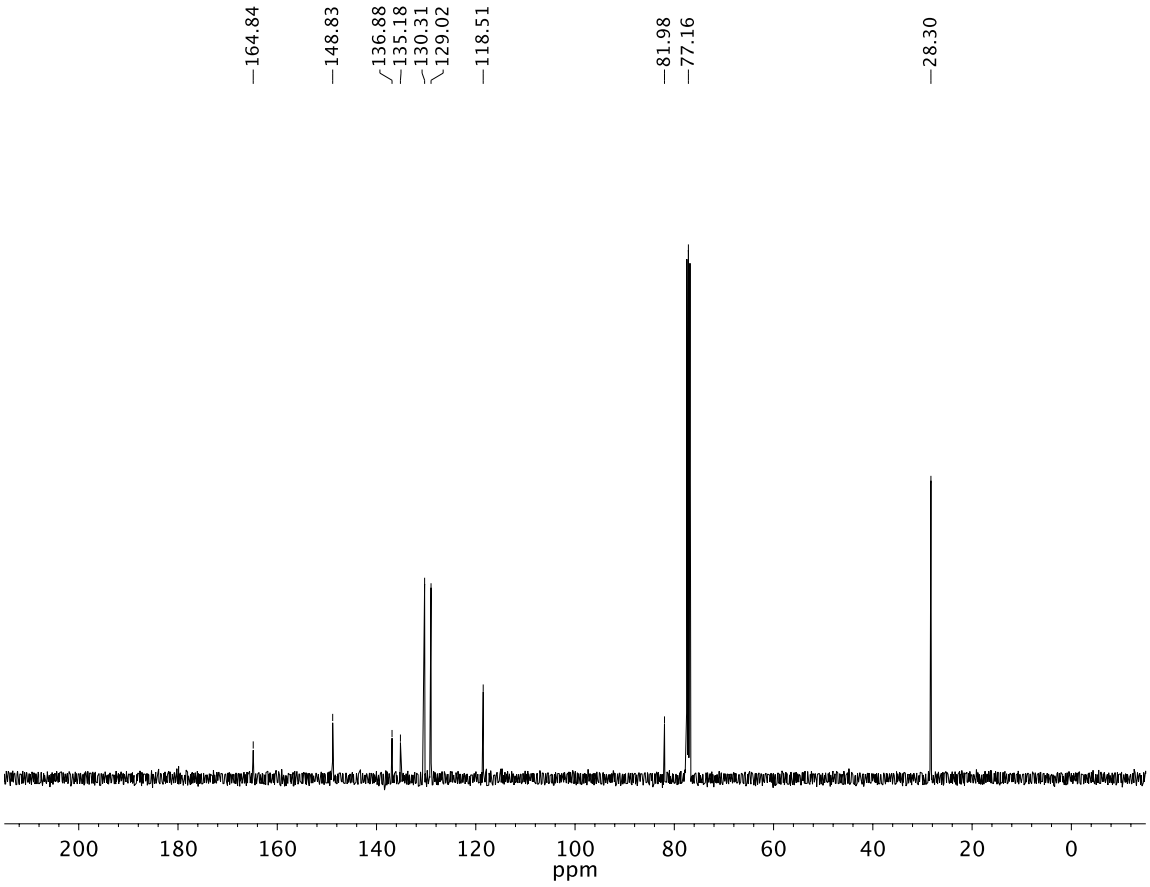
^{1}H, ^{13}C, ^{19}F NMR Spectra for Ligands Synthesized:



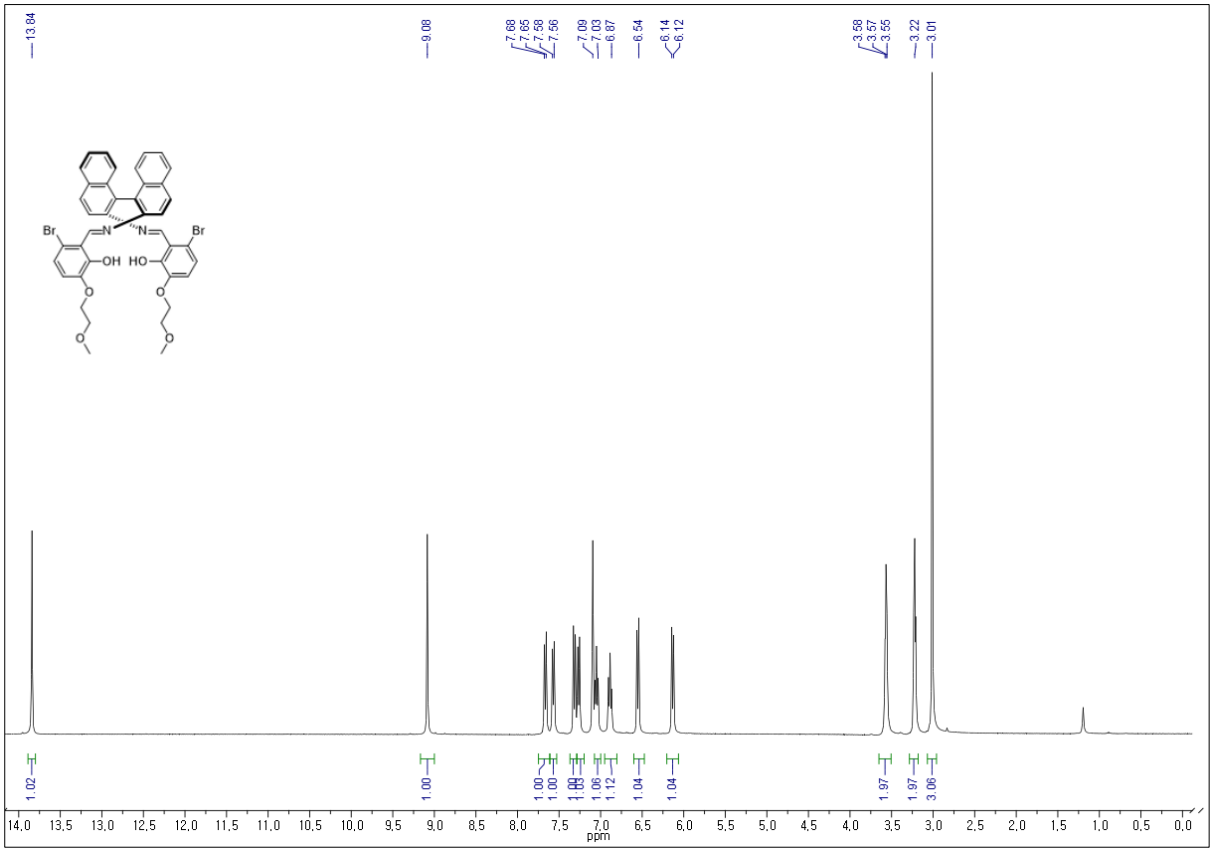
^{1}H NMR (300 MHz, $CDCl_3$) of compound **SI2**

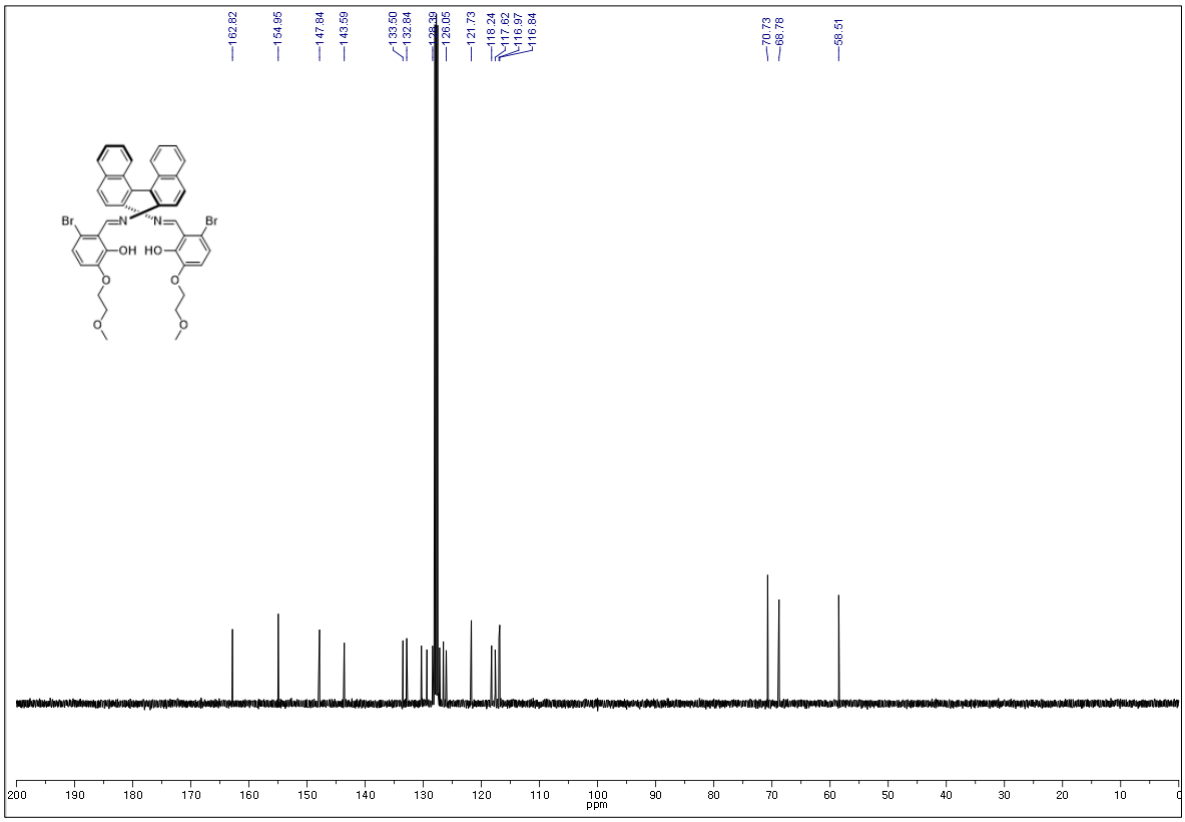


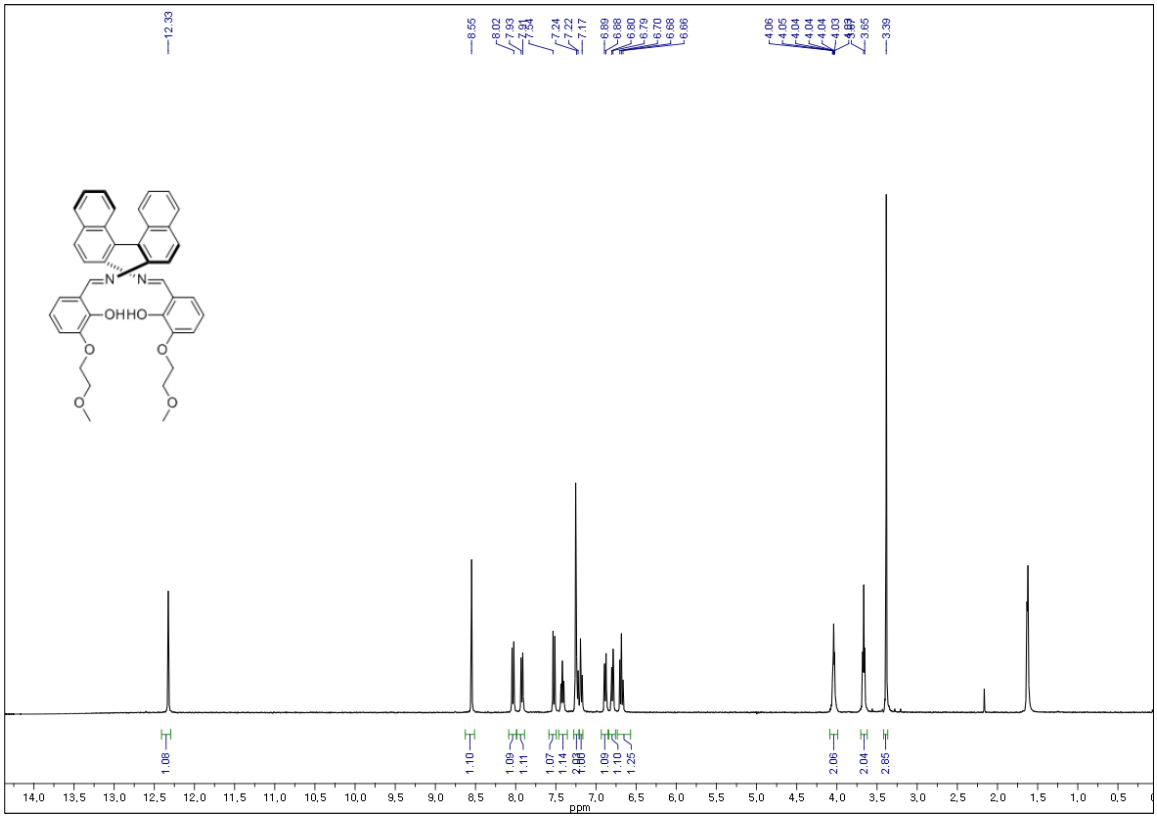
Infrared spectrum (Thin Film, NaCl) of compound **SI2**.

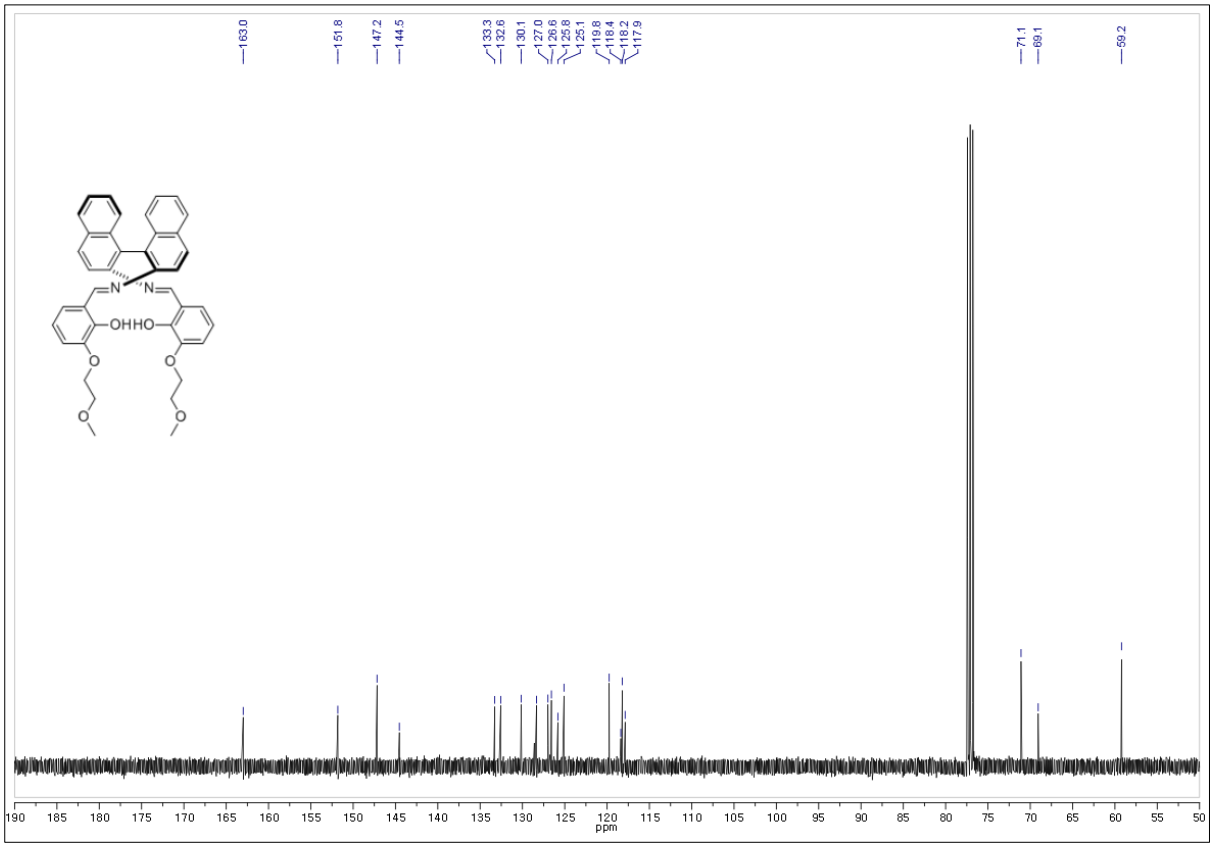


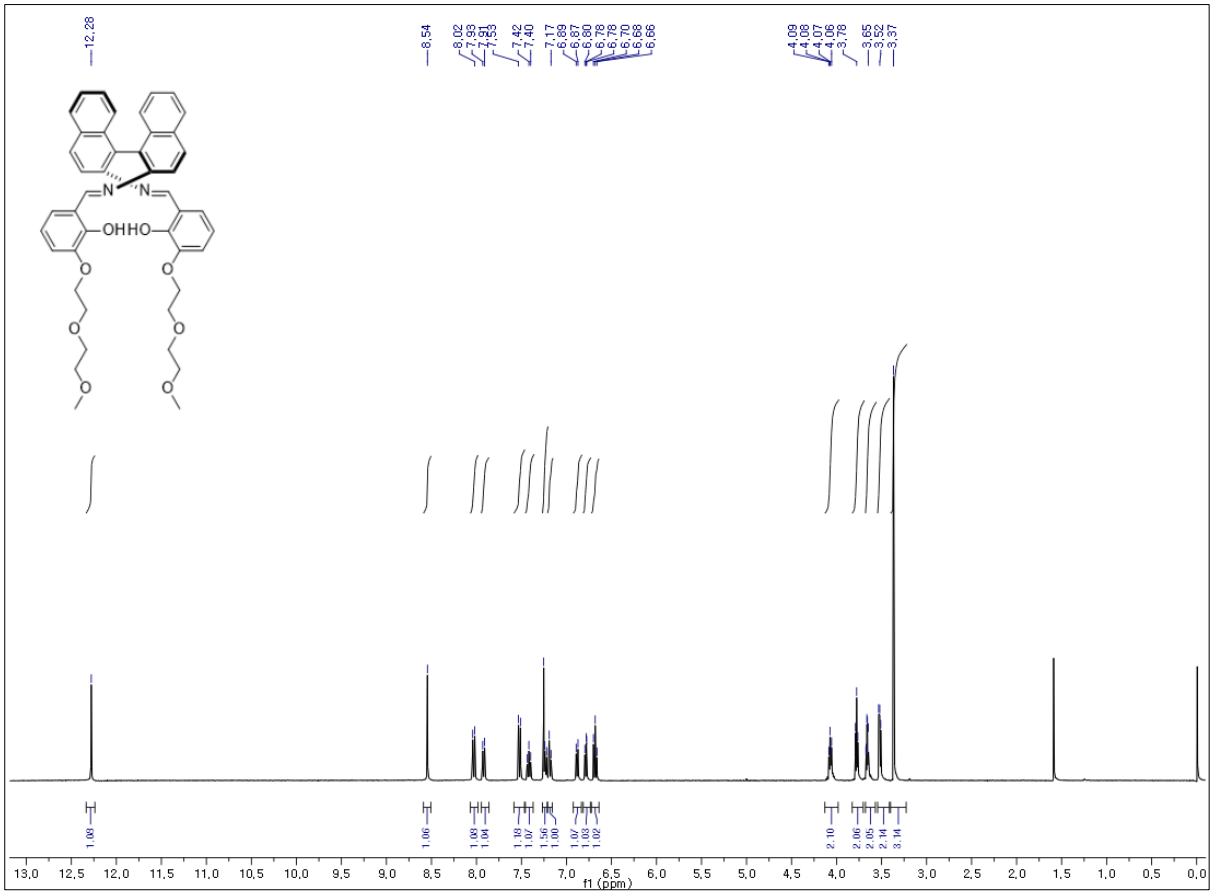
^{13}C NMR (101 MHz, $CDCl_3$) of compound **SI2**

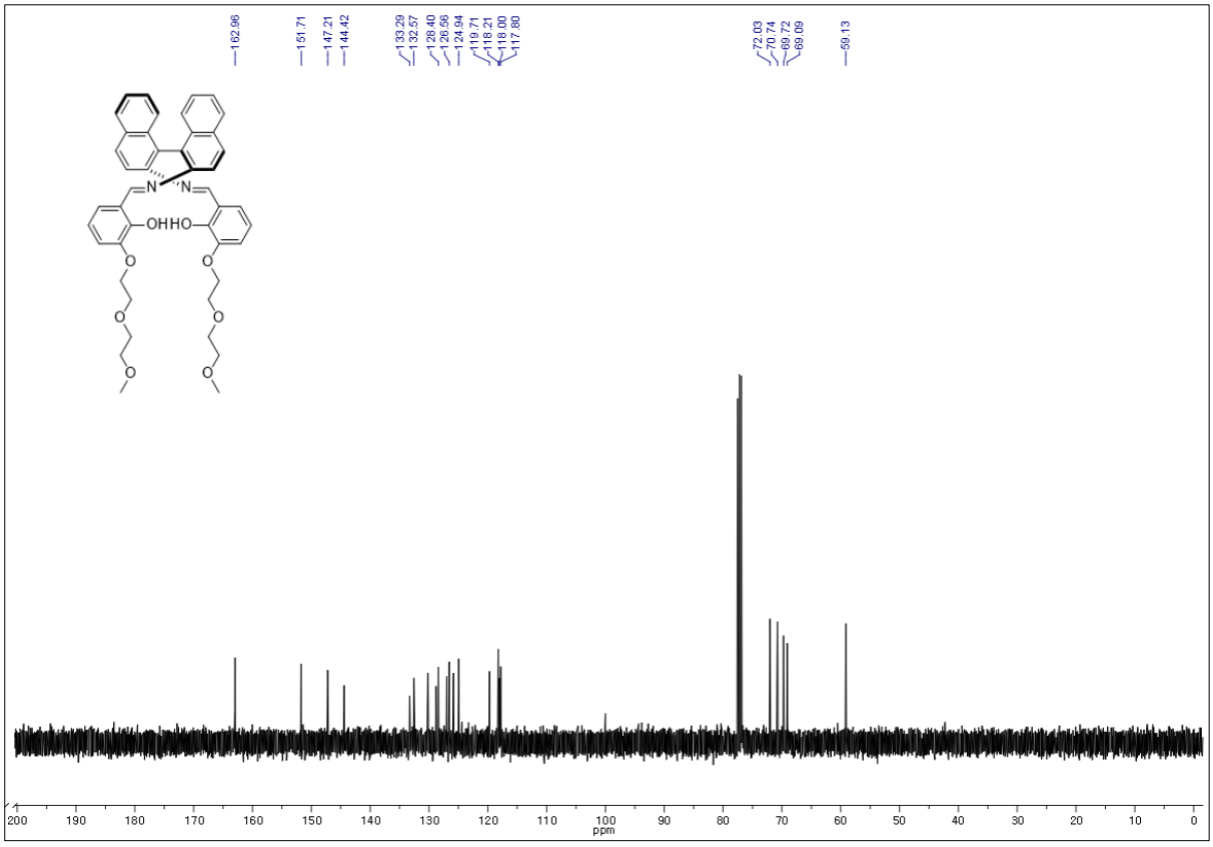

13.84
9.08
7.68
7.65
7.58
7.56
7.09
7.03
6.87
6.54
6.14
6.12
3.58
3.57
3.55
3.22
3.01
Br
N
N
Br
OH
HO
O
O
O
O
1.02
1.00
1.00
1.06
1.12
1.04
1.04
1.97
1.97
3.06
ppm

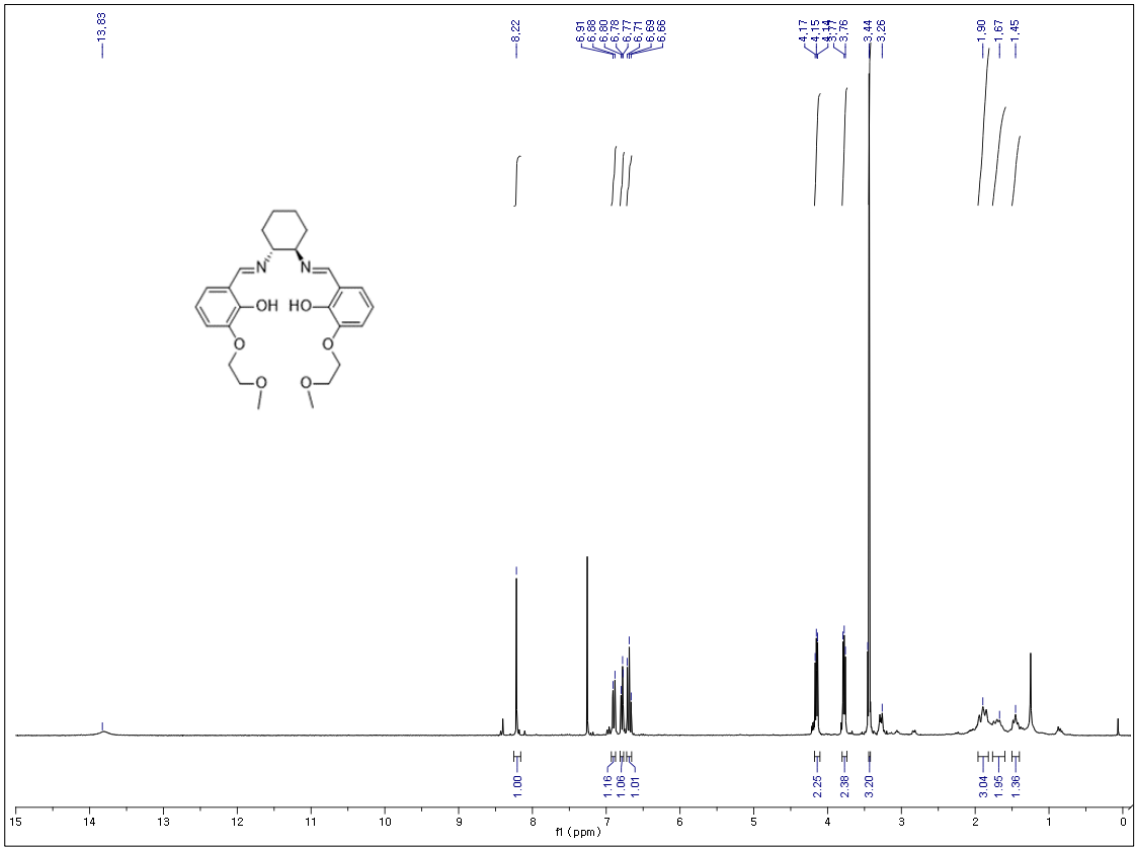

162.82
154.95
147.84
143.59
133.50
132.84
126.05
121.73
118.24
117.62
116.97
116.84
70.73
68.78
58.51
Br
N
N
Br
OH
HO
O
O
O
O
ppm

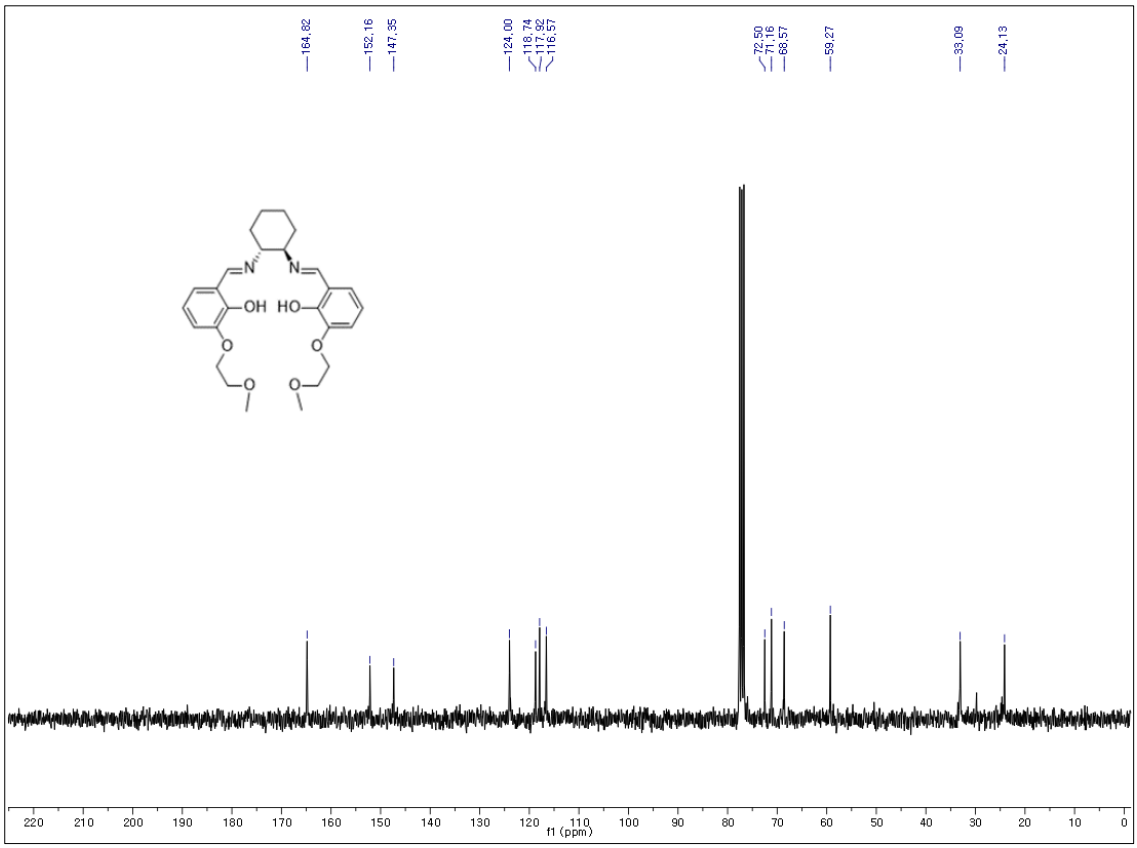

12.33
8.55
8.02
7.93
7.91
7.24
7.22
7.17
6.89
6.88
6.80
6.79
6.70
6.68
6.66
4.06
4.05
4.04
4.04
4.04
4.03
3.65
3.39
OHHO
1.08
1.10
1.09
1.11
1.07
1.14
1.09
1.10
1.25
2.06
2.04
2.85
ppm

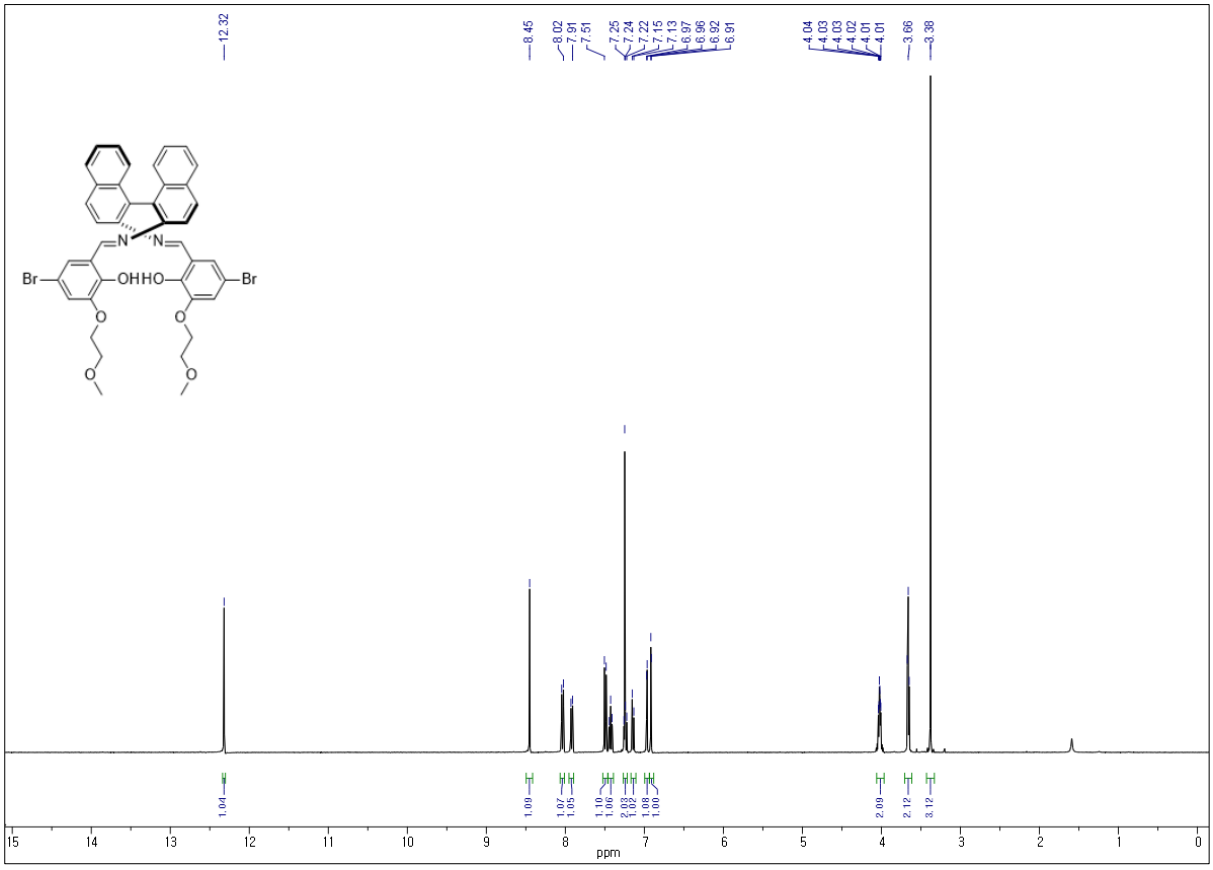

163.0
151.8
147.2
144.5
133.3
132.6
130.1
127.0
126.6
125.8
125.1
119.8
118.4
118.2
117.9
71.1
69.1
69.2
OHHO
ppm

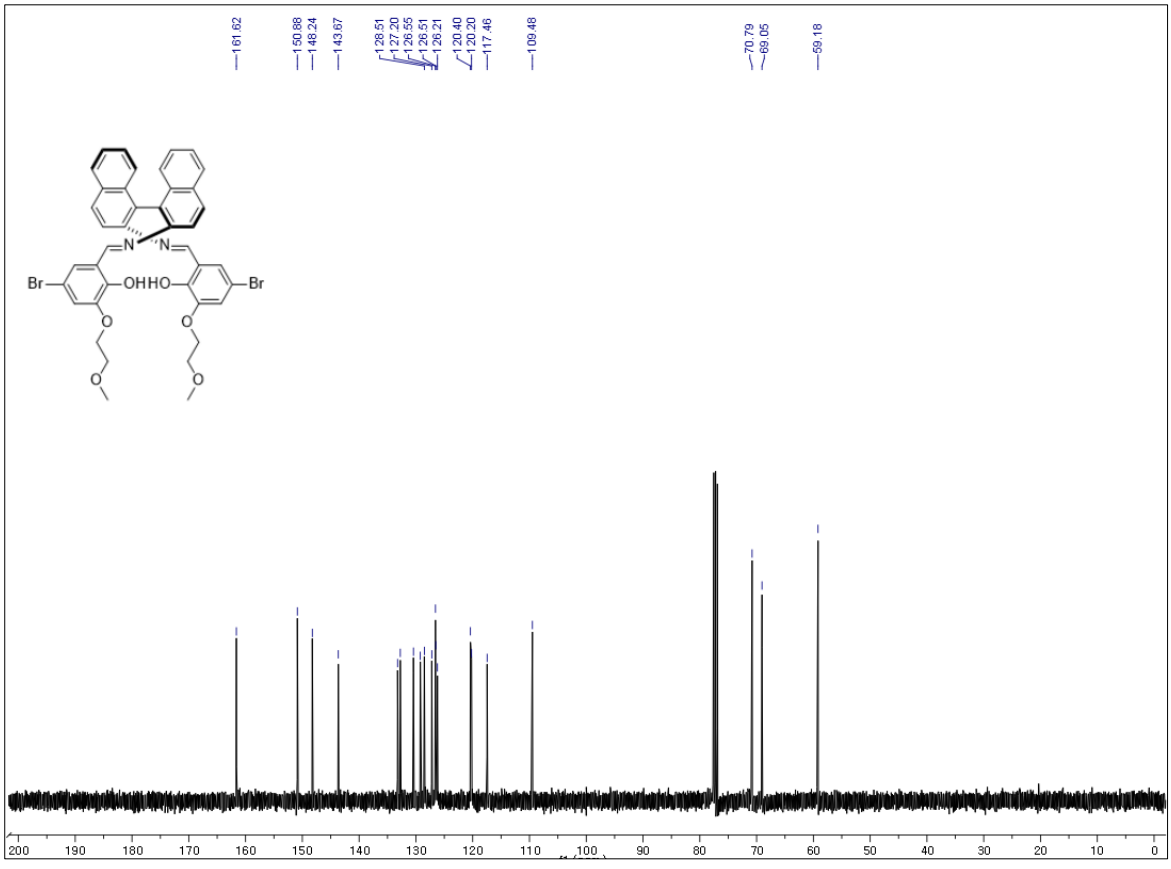

12.28
8.54
8.02
7.93
7.81
7.53
7.42
7.40
7.17
6.89
6.87
6.80
6.78
6.78
6.70
6.68
6.66
4.09
4.08
4.07
4.06
3.78
3.65
3.52
3.37
OHHO
f1 (ppm)

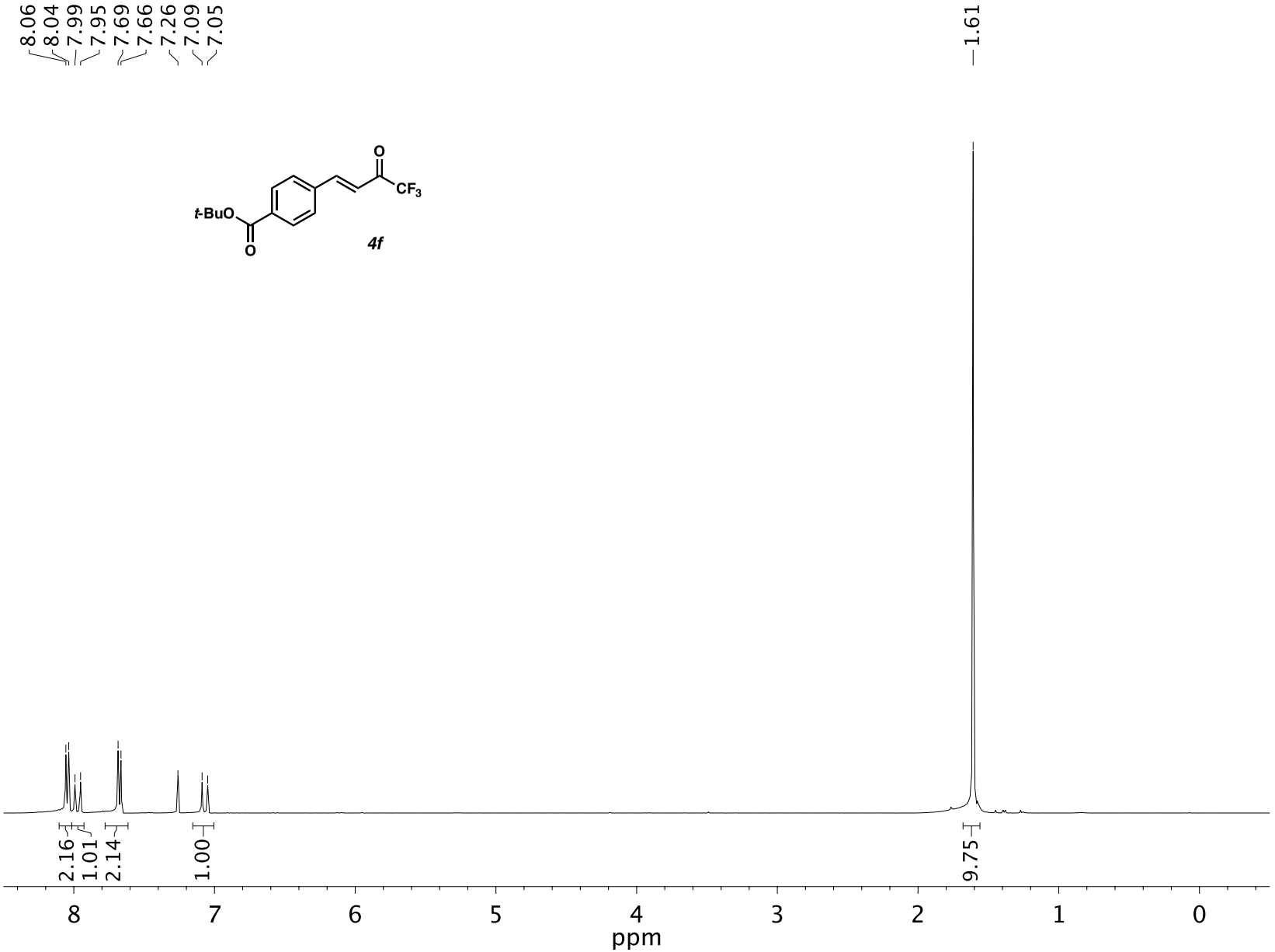

162.96
151.71
147.21
144.42
133.29
132.57
128.40
126.56
124.94
119.71
118.21
118.00
117.80
72.03
70.74
69.72
69.09
59.13
OHHO
ppm


13.83
8.22
6.91
6.88
6.80
6.78
6.77
6.71
6.69
6.66
4.17
4.15
4.14
3.77
3.76
3.44
3.26
1.90
1.67
1.45
OH
HO
1.00
1.16
1.06
1.01
2.25
2.38
3.20
3.04
1.95
1.36
f1 (ppm)

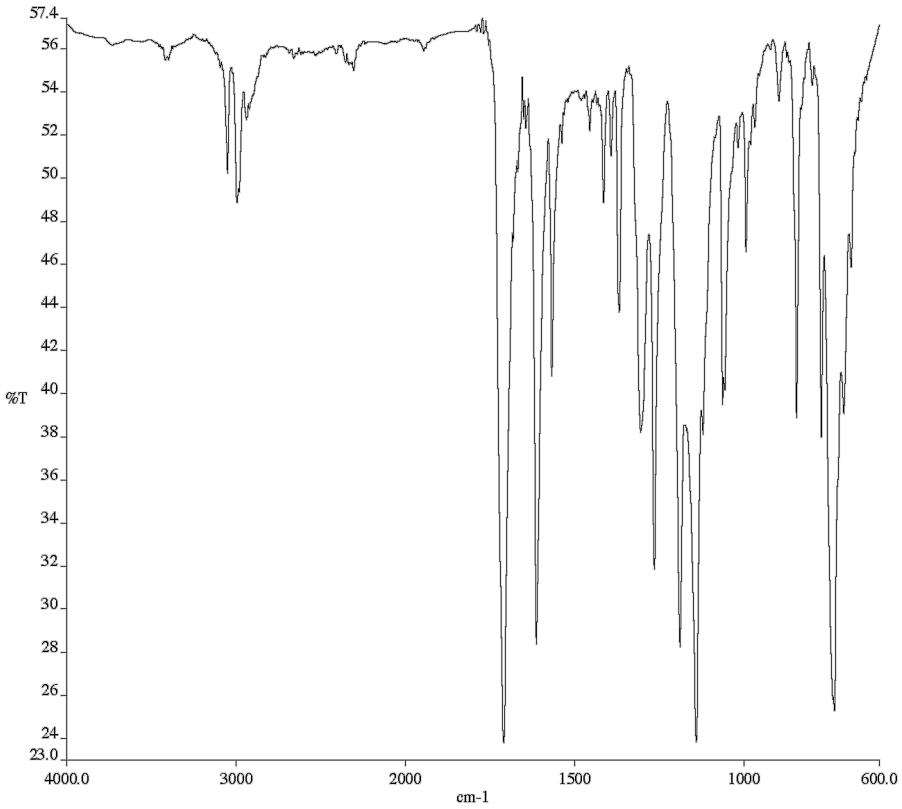

164.82
152.16
147.35
124.00
118.74
117.92
116.57
72.50
71.16
68.57
59.27
33.09
24.13
OH
HO
f1 (ppm)


12.32
8.45
8.02
7.91
7.51
7.25
7.24
7.22
7.15
7.13
6.97
6.96
6.92
6.91
4.04
4.03
4.03
4.02
4.01
4.01
3.66
3.38
Br
OHHO
Br
ppm

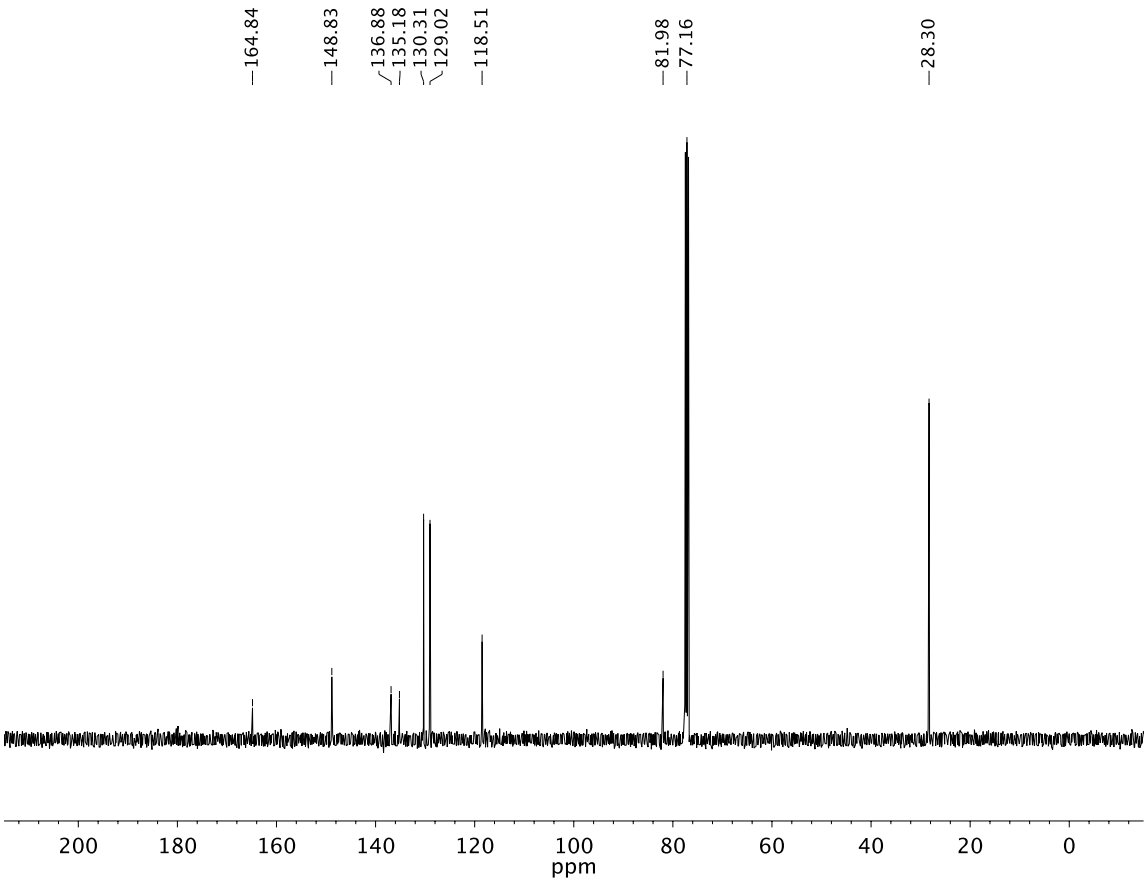

161.62
150.88
148.24
143.67
128.51
127.30
126.55
126.51
126.21
120.40
120.20
117.46
109.48
70.79
69.05
59.18
Br
OHHO
Br



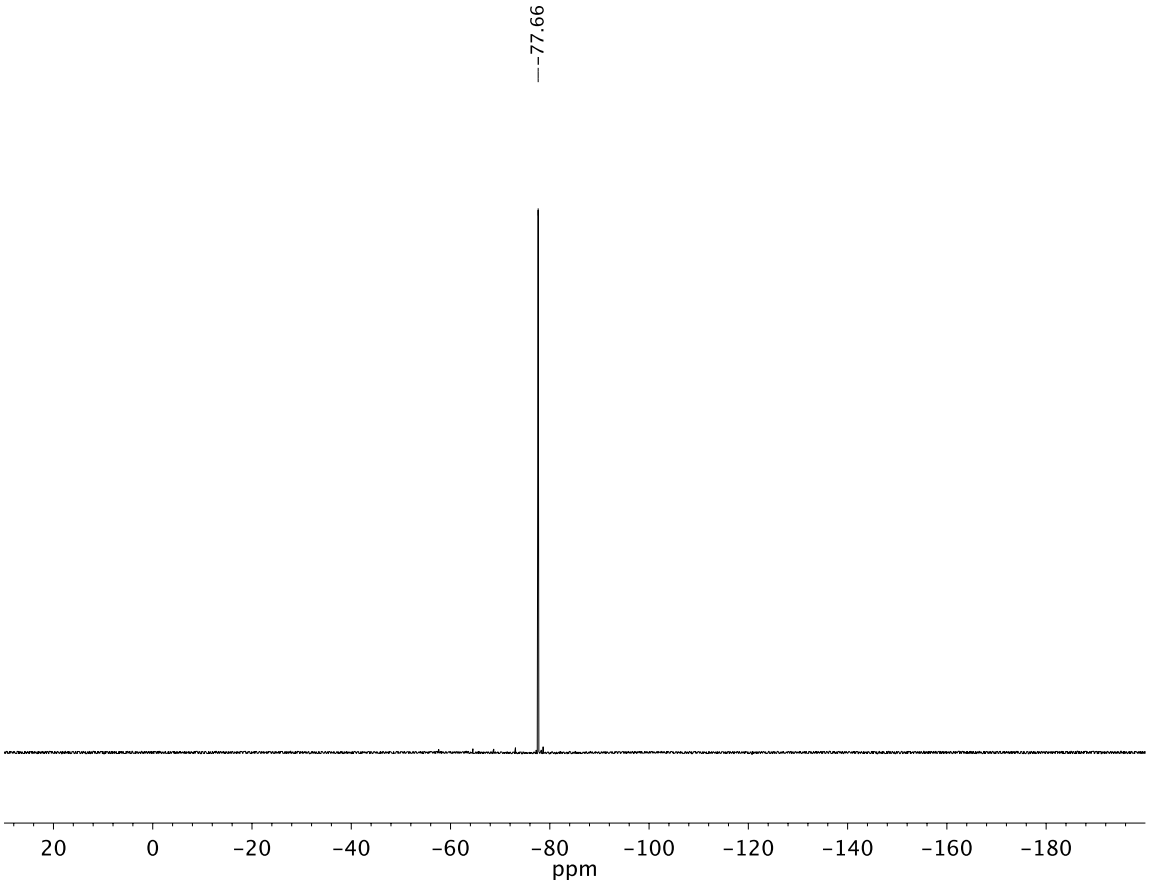
^{1}H NMR (400 MHz, $CDCl_3$) of compound **4f**



Infrared spectrum (Thin Film, NaCl) of compound **4f**.

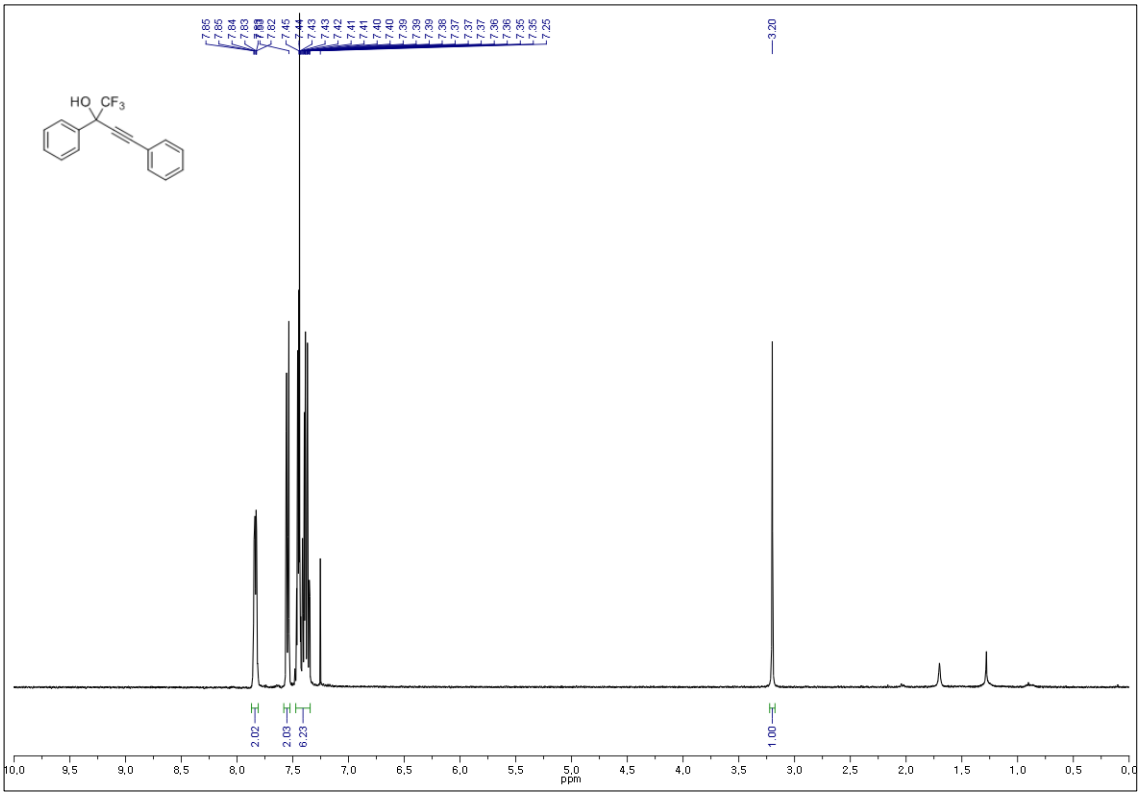


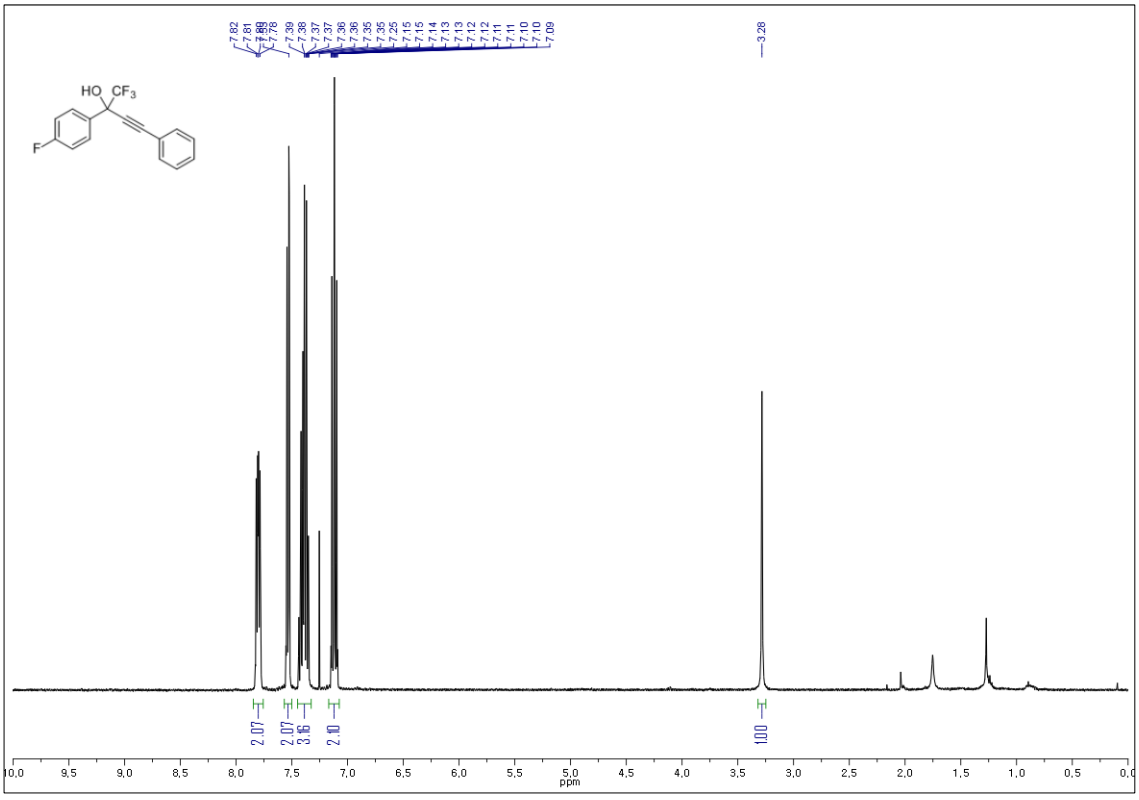
^{13}C NMR (101 MHz, $CDCl_3$) of compound **4f**

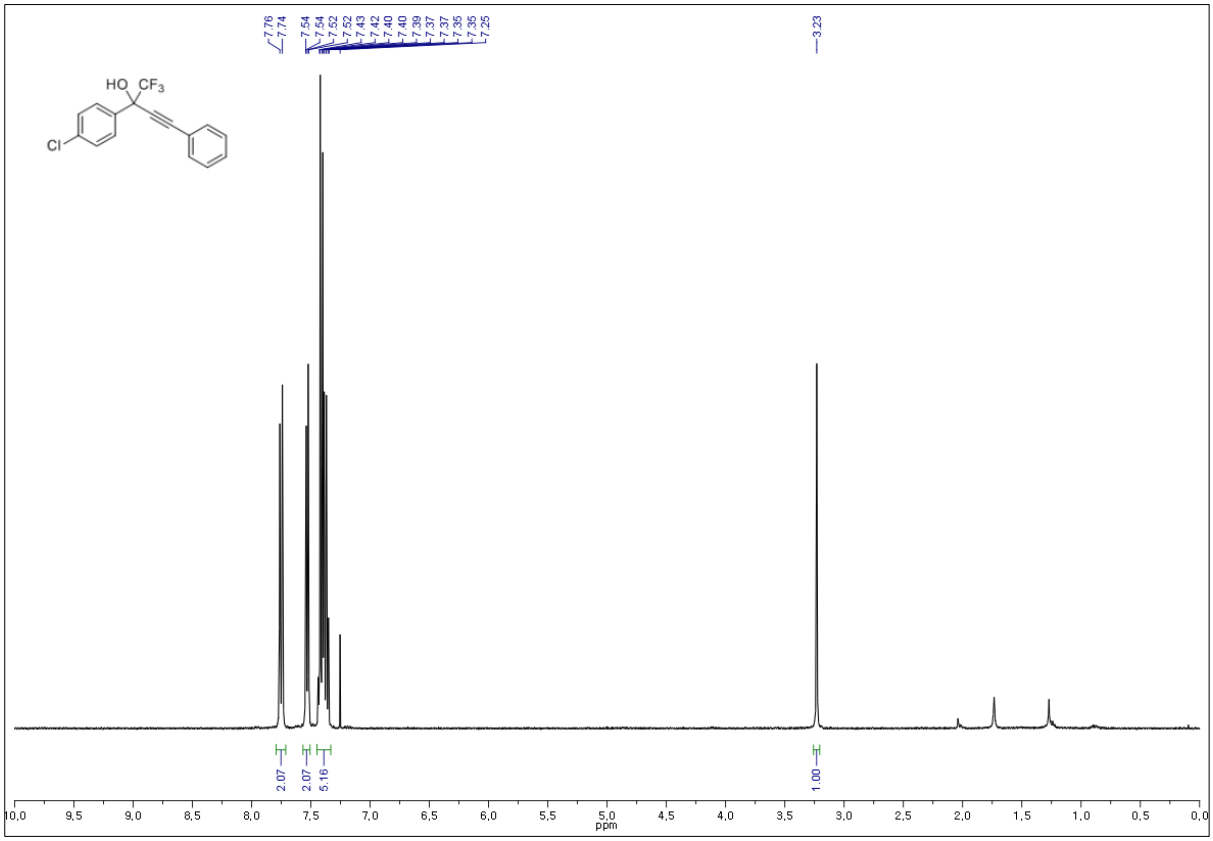


^{19}F NMR (282 MHz, $CDCl_3$) of compound **4f**

NMR and IR Data for Trifluoromethyl Products



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CF3
Cl
7.76
7.74
7.54
7.54
7.52
7.52
7.43
7.42
7.40
7.40
7.39
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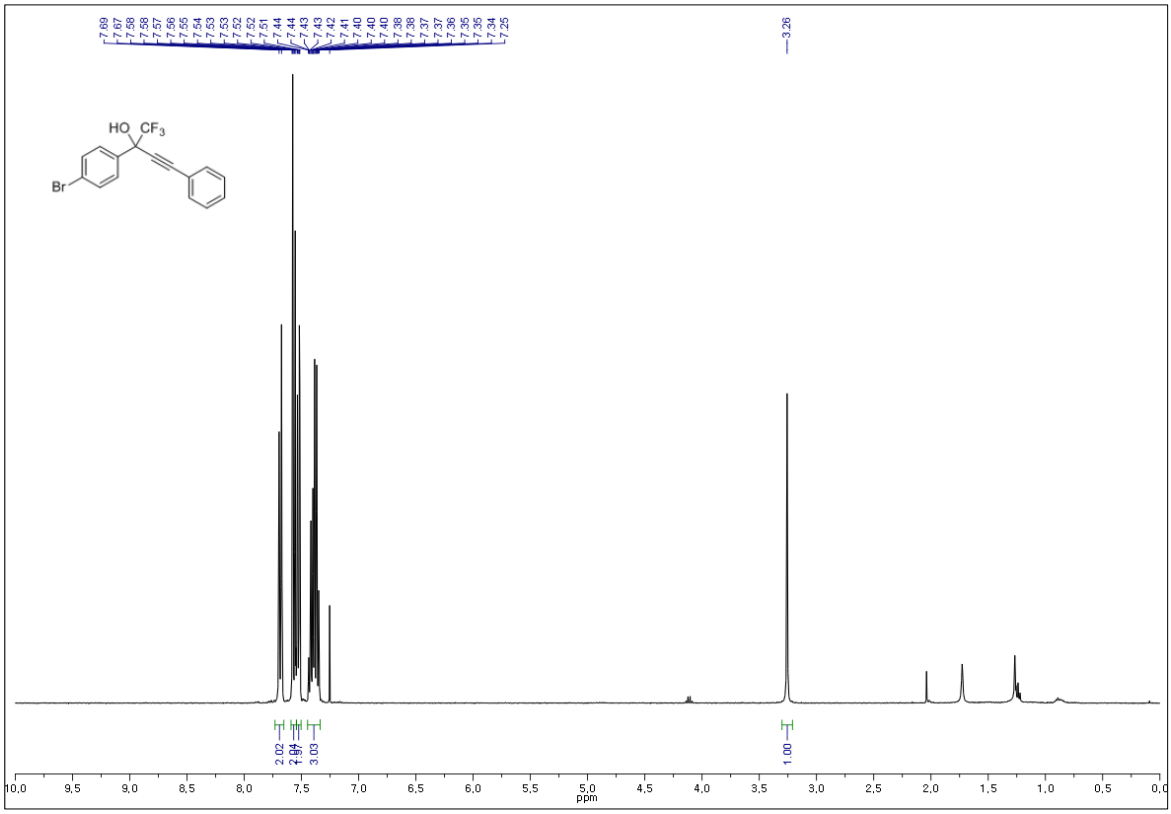
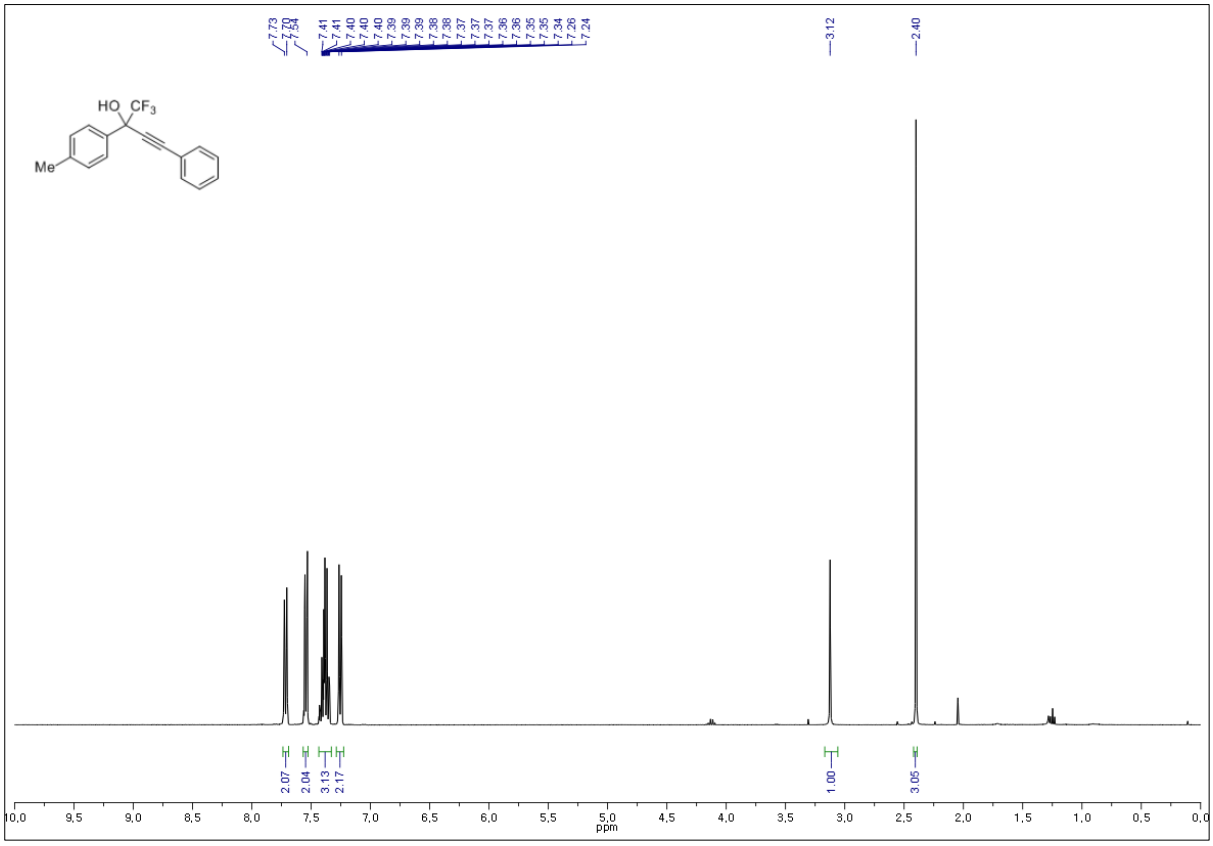
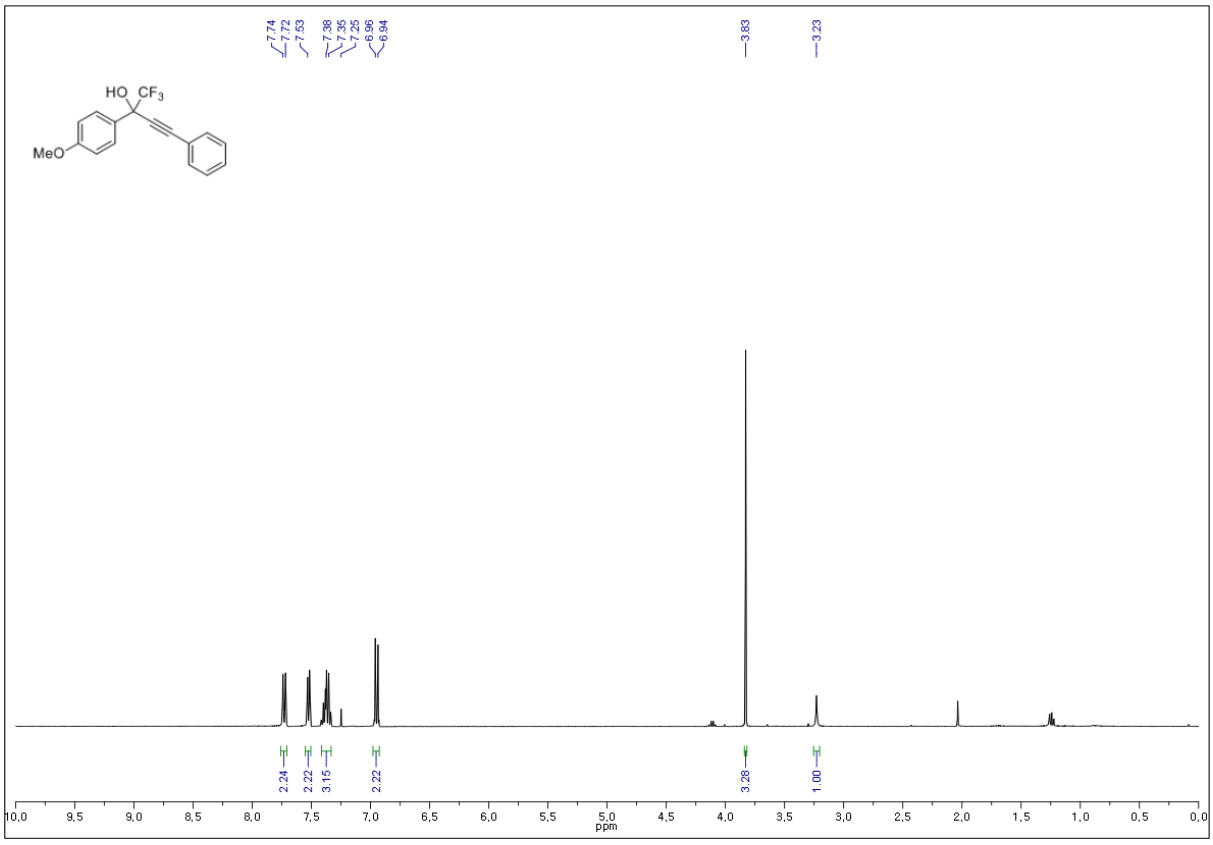
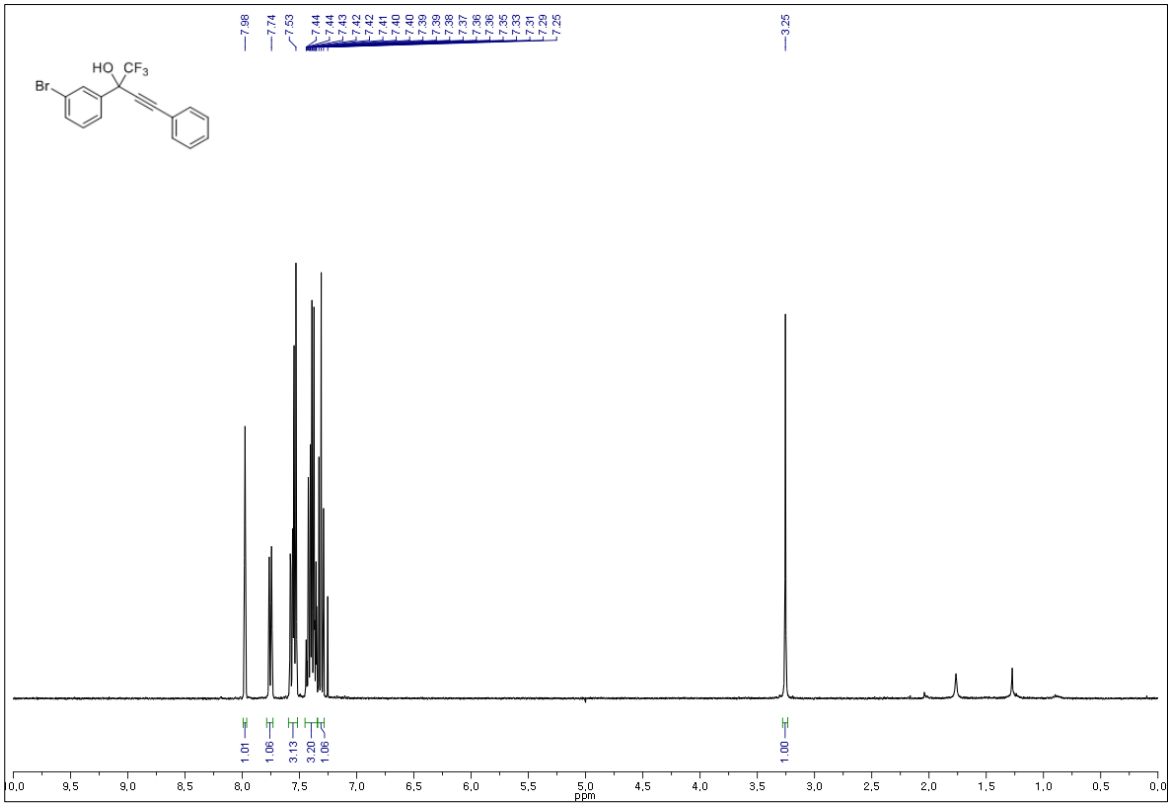
7.37
7.35
7.35
7.25
3.23
2.07
2.07
5.16
1.00
ppm


HO
CF3
Br
7.69
7.67
7.58
7.58
7.57
7.57
7.56
7.55
7.54
7.53
7.53
7.52
7.52
7.51
7.44
7.44
7.43
7.43
7.42
7.41
7.40
7.40
7.40
7.38
7.38
7.37
7.37
7.36
7.35
7.35
7.34
7.25
3.26
2.02
2.01
2.01
3.03
1.00
ppm


HO
CF3
Me
7.73
7.72
7.54
7.41
7.41
7.40
7.40
7.40
7.39
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.35
7.35
7.34
7.26
7.24
3.12
2.40
2.07
2.04
3.13
2.17
1.00
3.05
ppm


HO
CF3
MeO
7.74
7.72
7.53
7.38
7.35
7.25
6.96
6.94
3.83
3.23
2.24
2.22
3.15
2.22
3.28
1.00
ppm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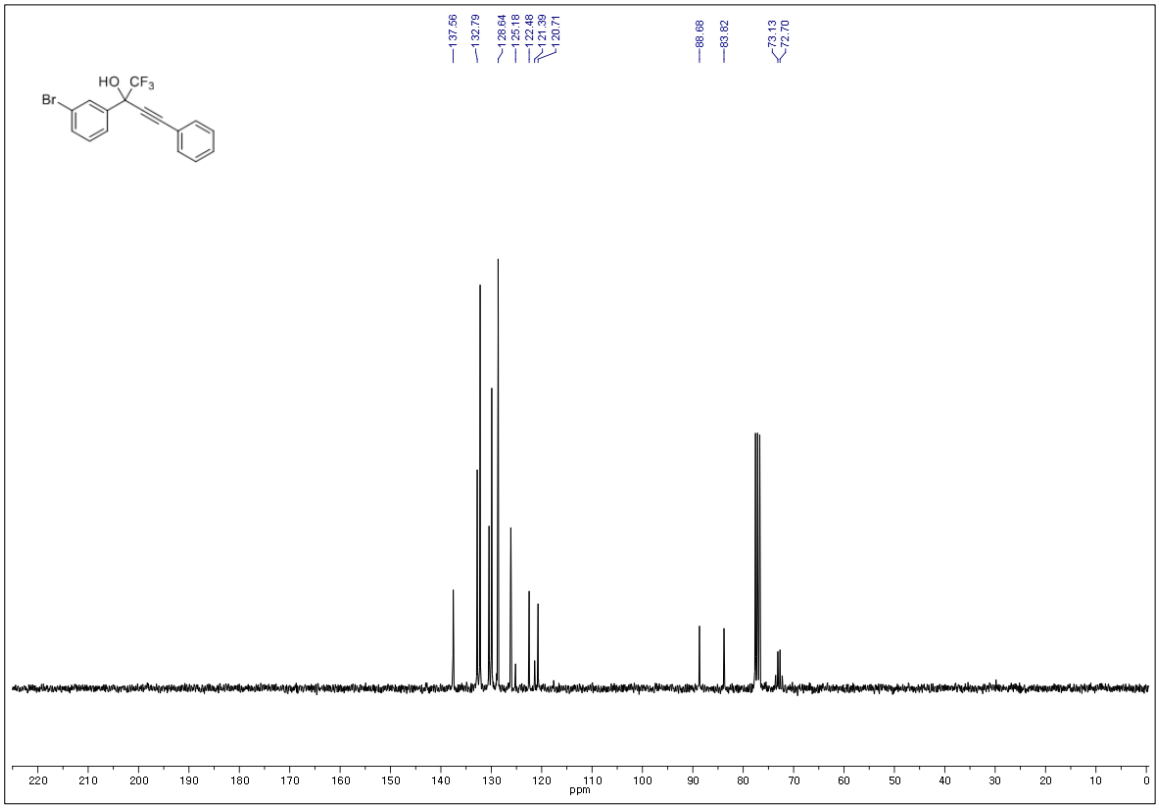
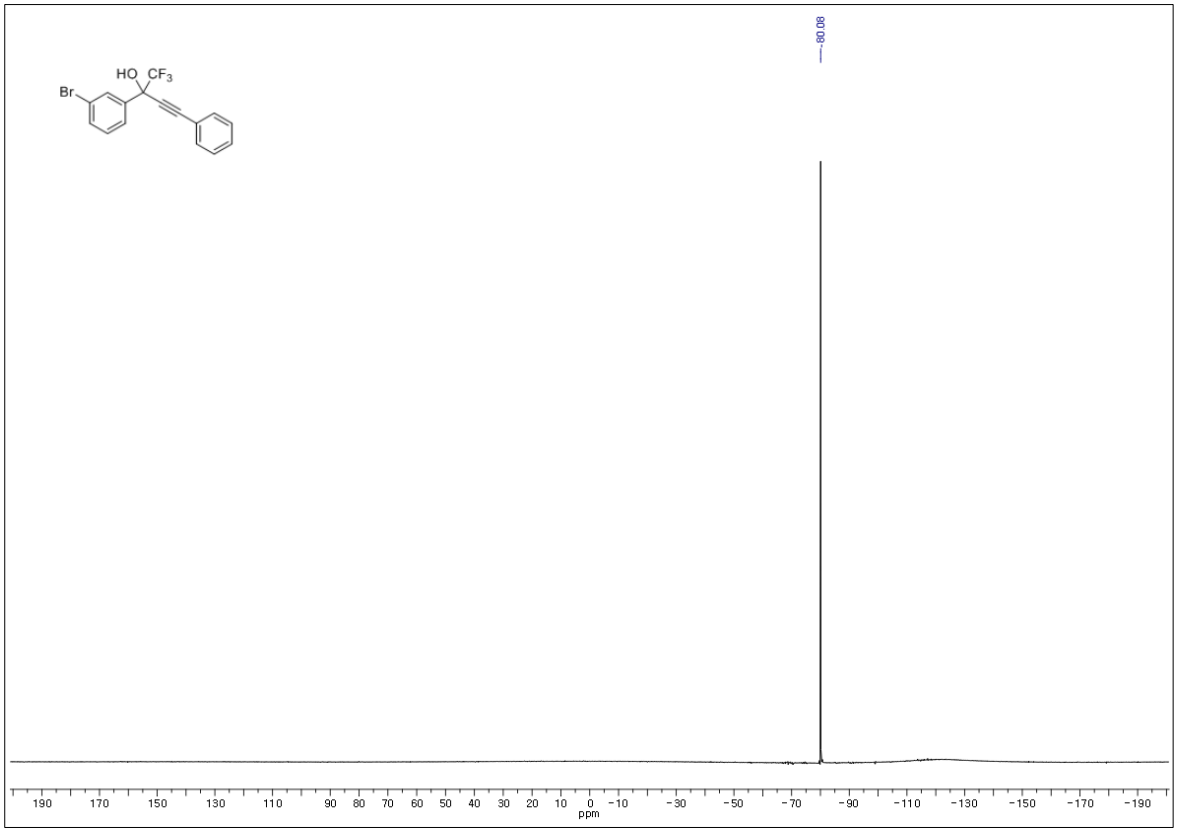
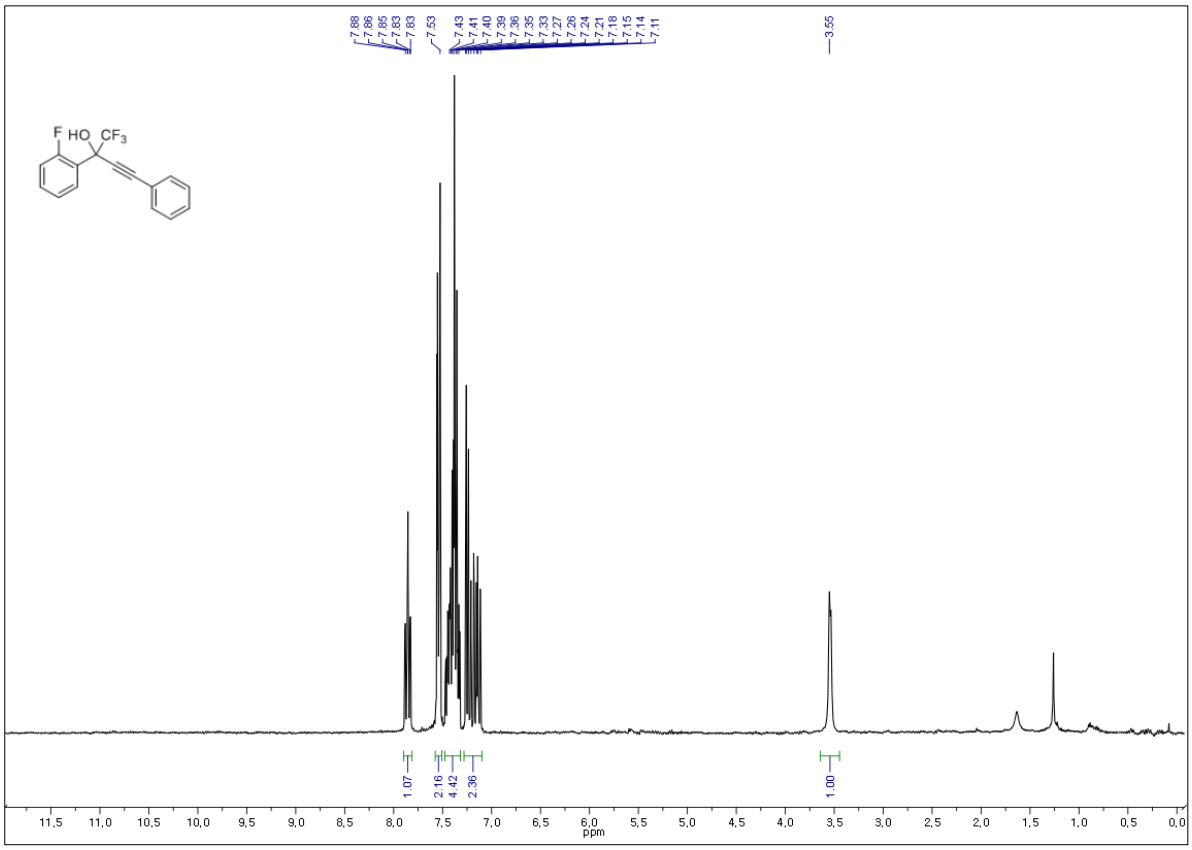
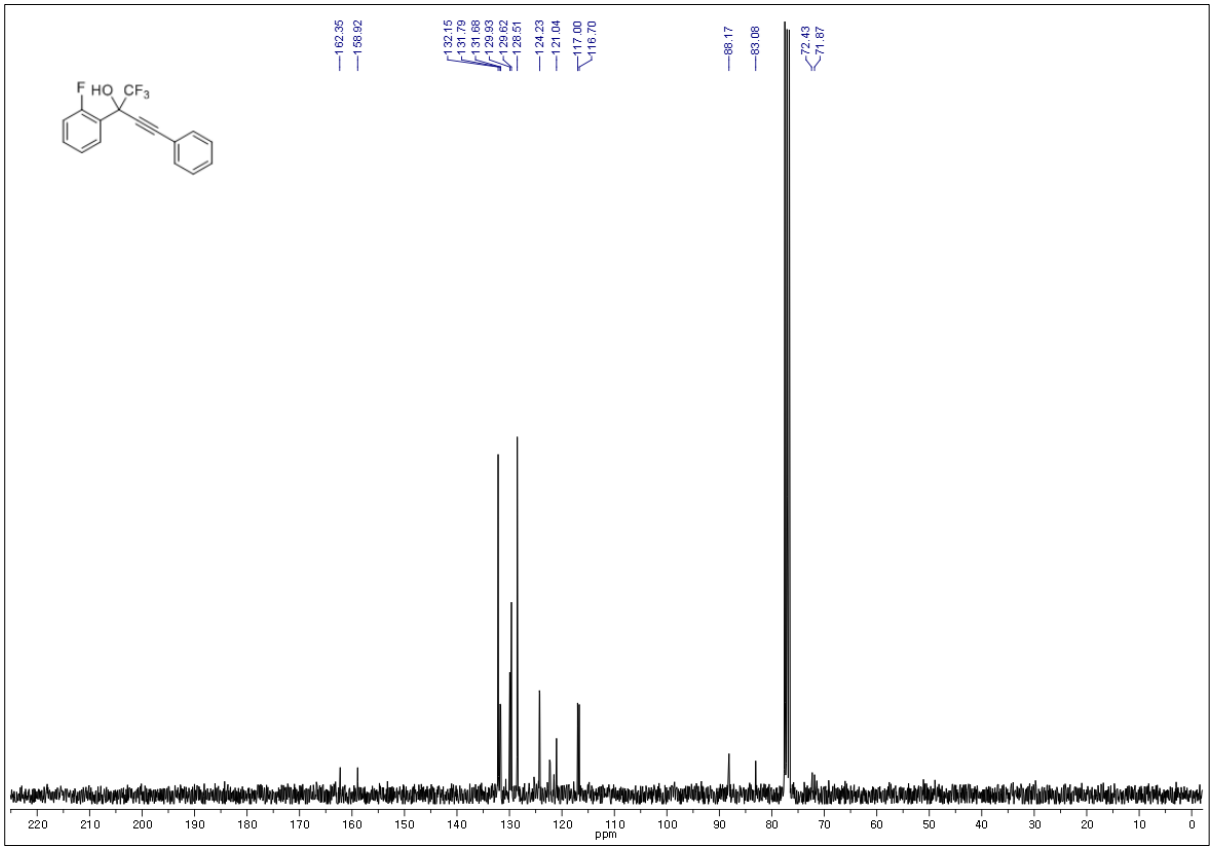
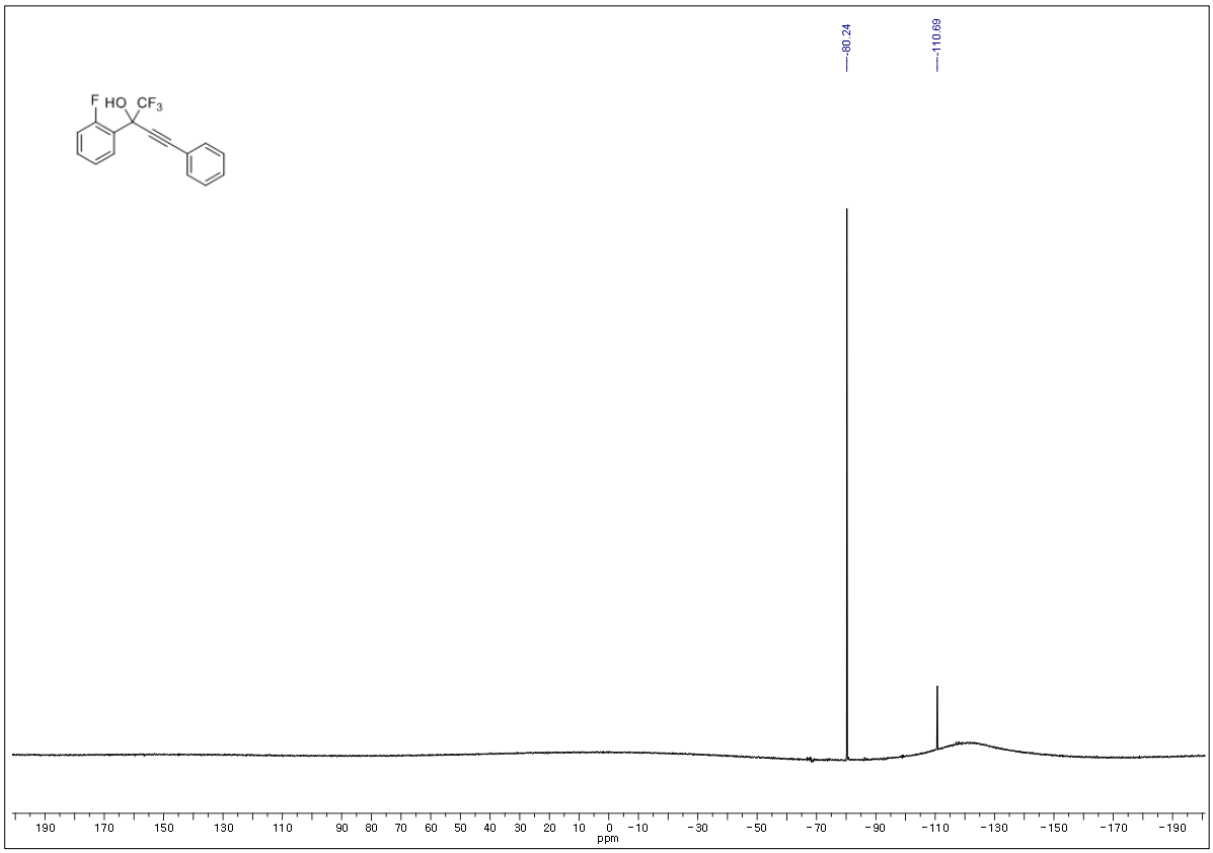
7.74
7.53
7.44
7.44
7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.39
7.38
7.37
7.36
7.35
7.33
7.31
7.29
7.25
3.25
HO
CF3
Br
1.01
1.06
3.13
3.20
1.06
1.00
ppm

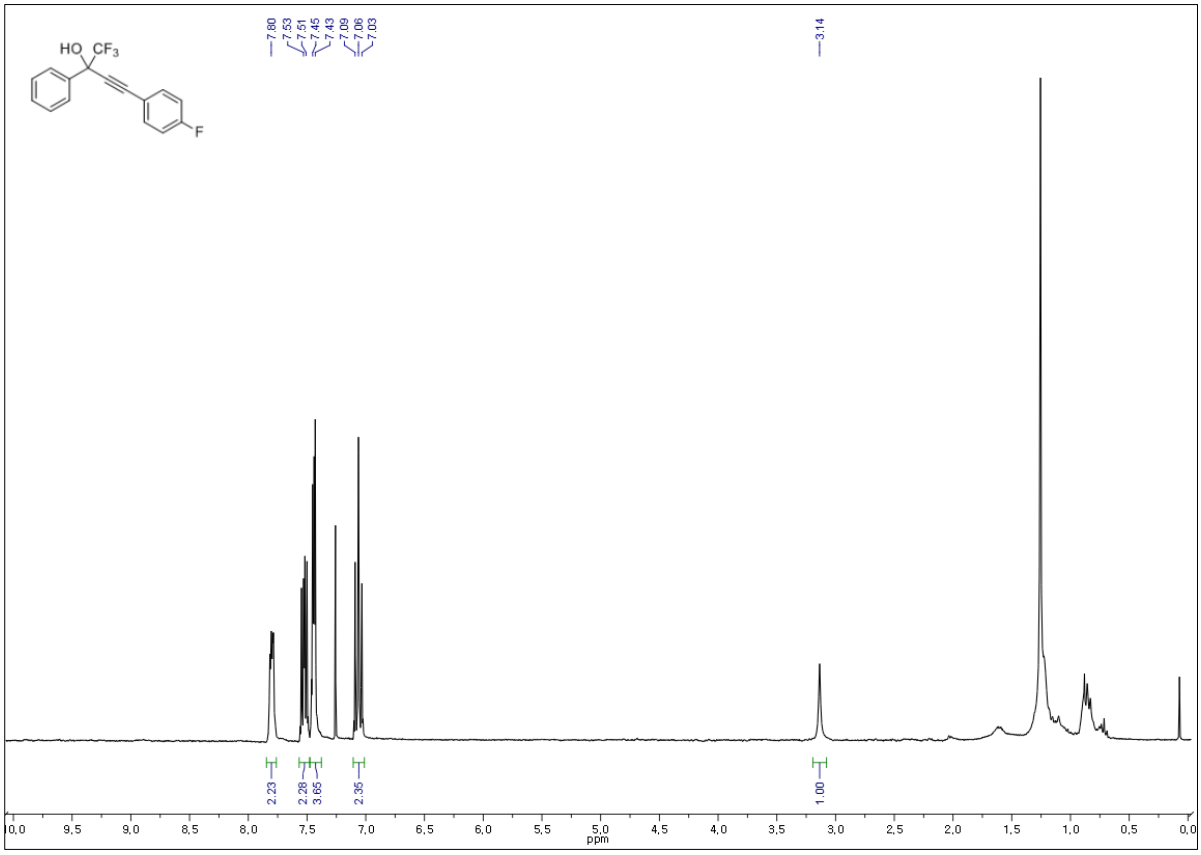

137.56
132.79
128.64
125.18
122.48
121.39
120.71
88.68
83.82
73.13
72.70
HO
CF3
Br
ppm

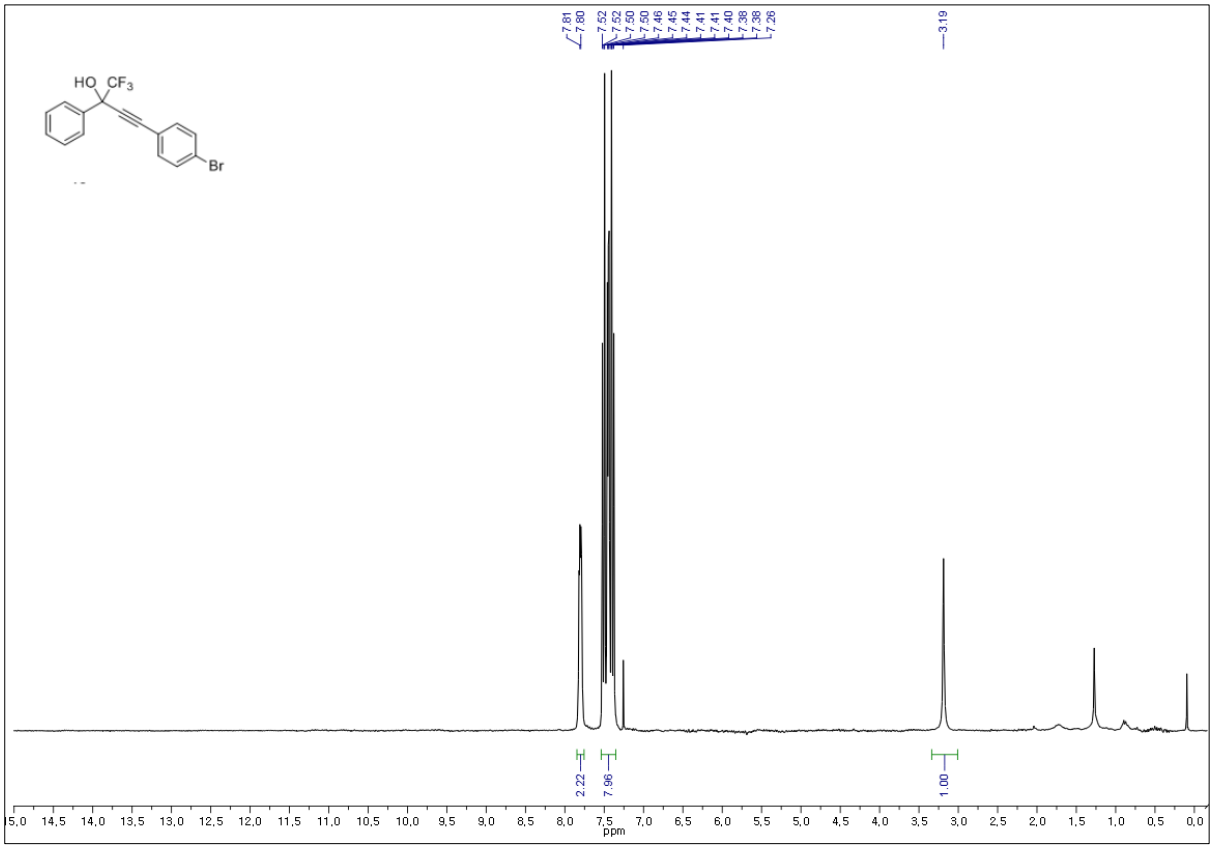

HO CF3
Br
-80.08
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190
ppm

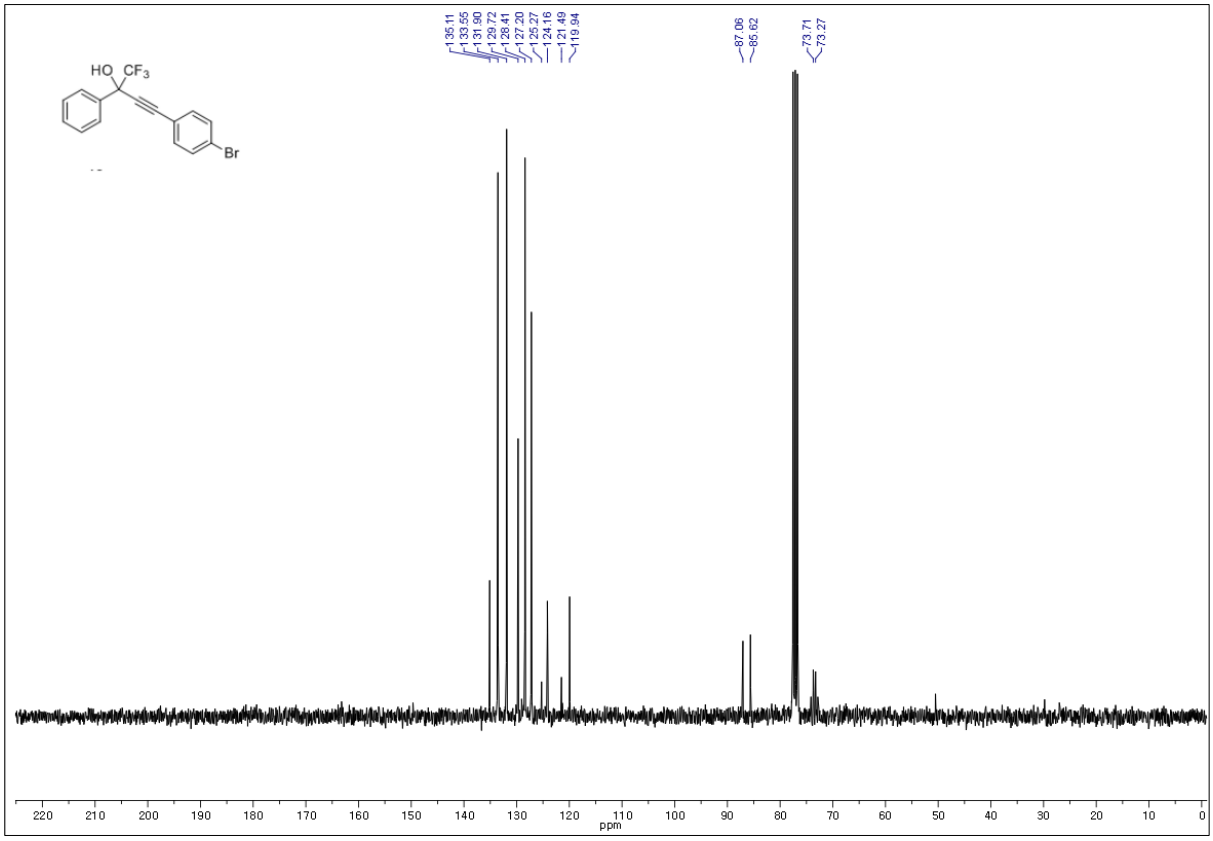

7.88
7.86
7.85
7.83
7.83
7.53
7.43
7.41
7.40
7.39
7.36
7.35
7.33
7.27
7.26
7.24
7.21
7.18
7.15
7.14
7.11
3.55
F HO CF3
1.07
2.16
4.42
2.36
1.00
11,5 11,0 10,5 10,0 9,5 9,0 8,5 8,0 7,5 7,0 6,5 6,0 5,5 5,0 4,5 4,0 3,5 3,0 2,5 2,0 1,5 1,0 0,5 0,0
ppm

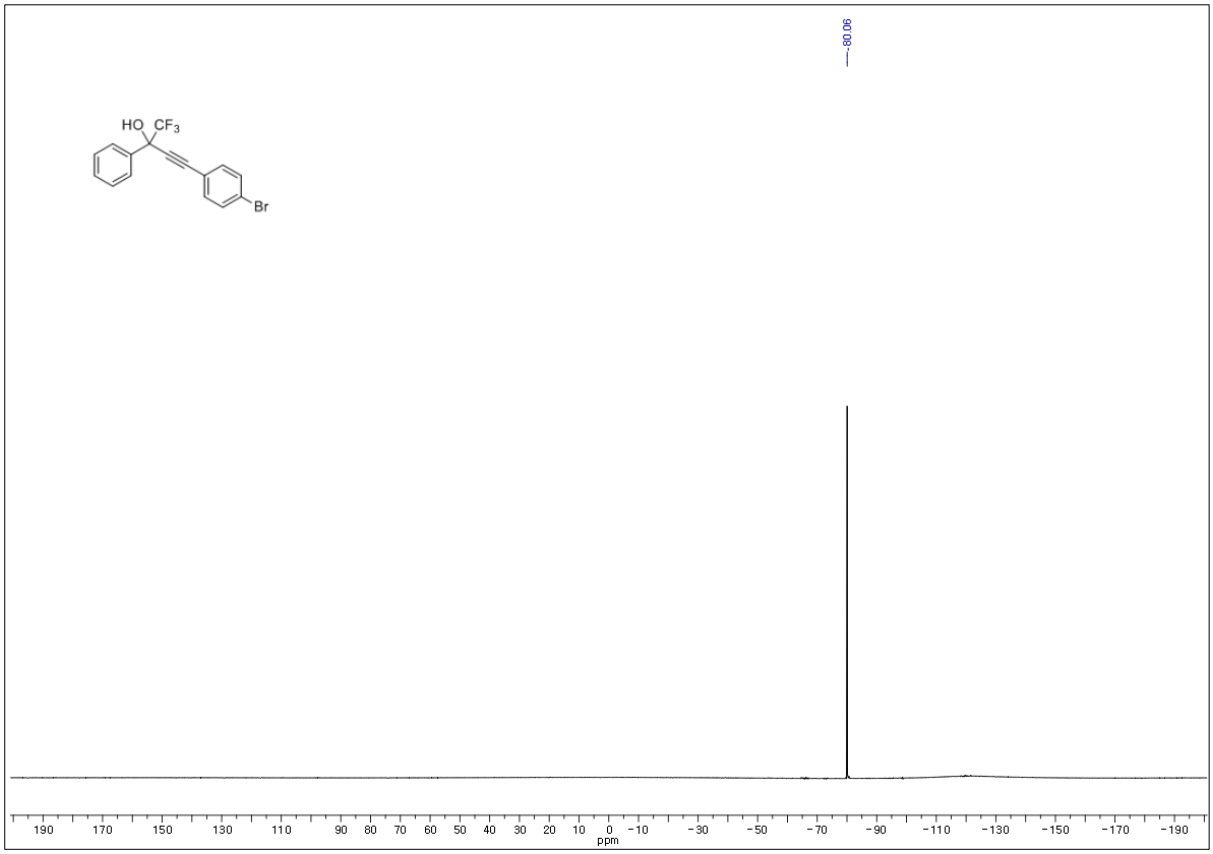

F HO CF3
162.35
158.92
132.15
131.79
131.68
129.93
129.62
128.51
124.23
121.04
117.00
116.70
88.17
83.08
72.43
71.87
ppm

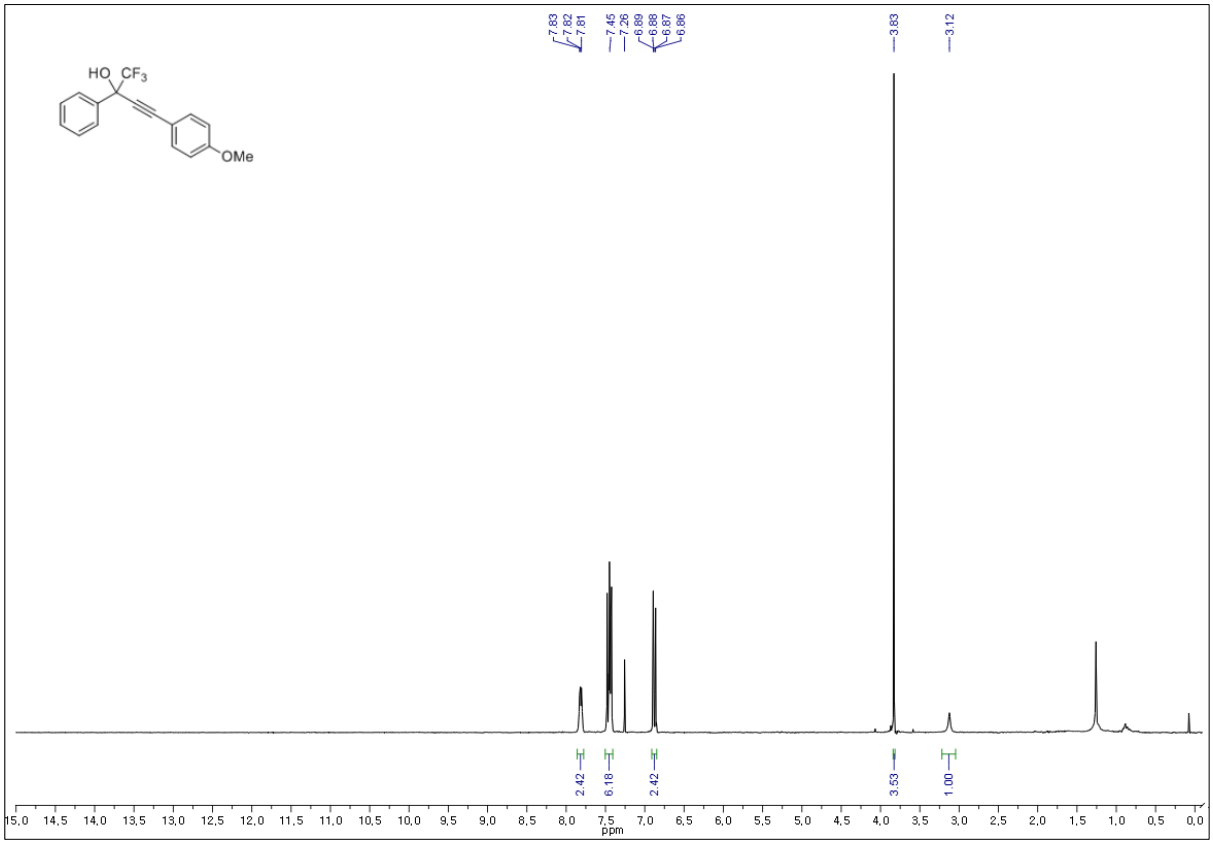

F HO CF3
-80.24
-110.69
ppm

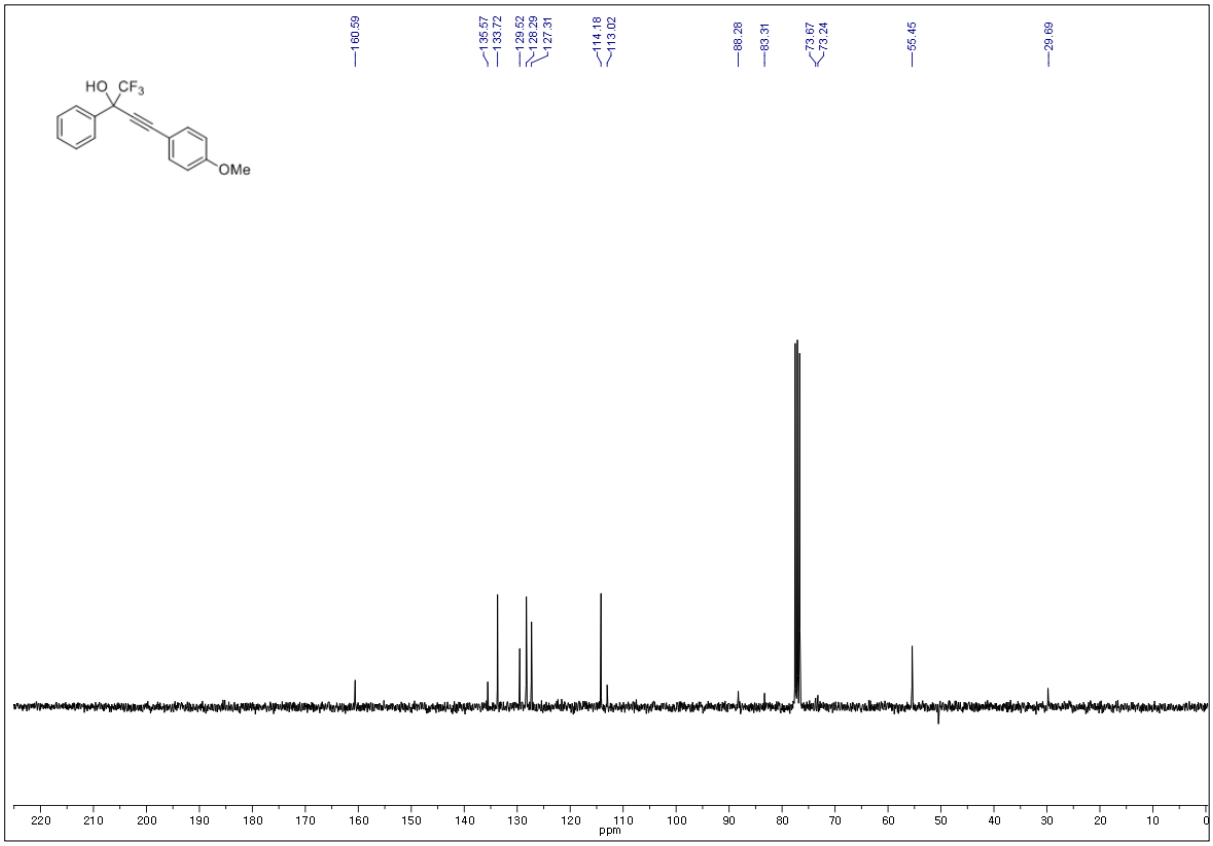

HO
CF3
F
7.80
7.53
7.51
7.45
7.43
7.09
7.06
7.03
3.14
2.23
2.28
3.65
2.35
1.00
ppm

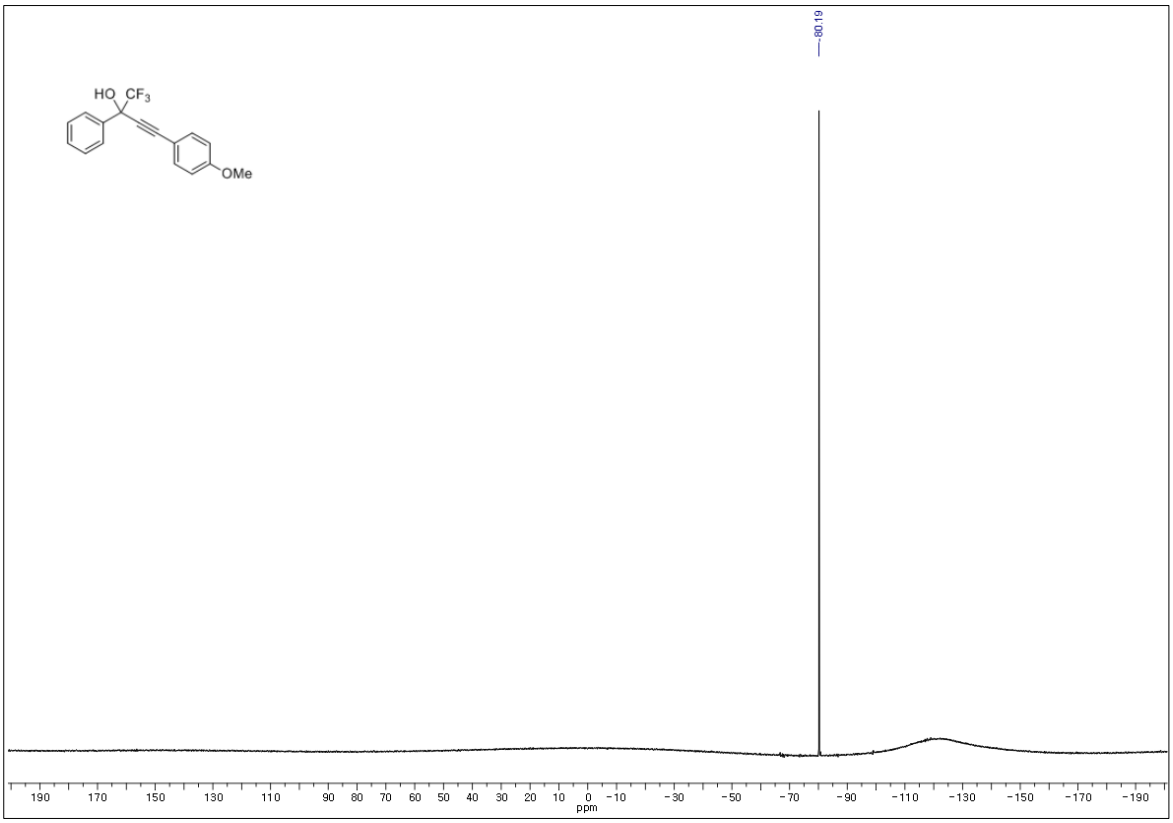

HO
CF3
Br
7.81
7.80
7.52
7.52
7.50
7.50
7.46
7.45
7.44
7.41
7.41
7.40
7.38
7.38
7.26
3.19
2.22
7.96
1.00
ppm

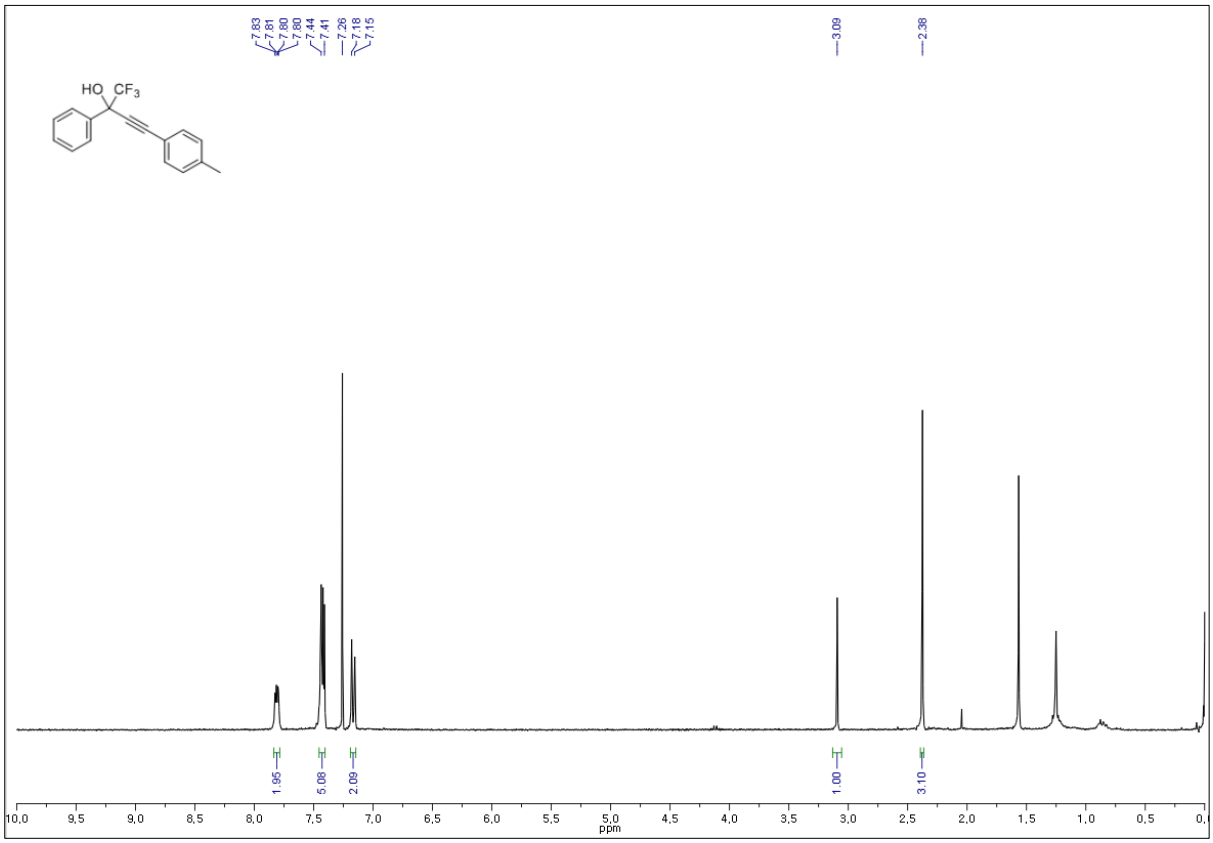

135.11
133.55
131.90
129.72
128.41
127.20
125.27
124.16
121.49
119.94
87.06
85.62
73.71
73.27
HO
CF3
Br
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

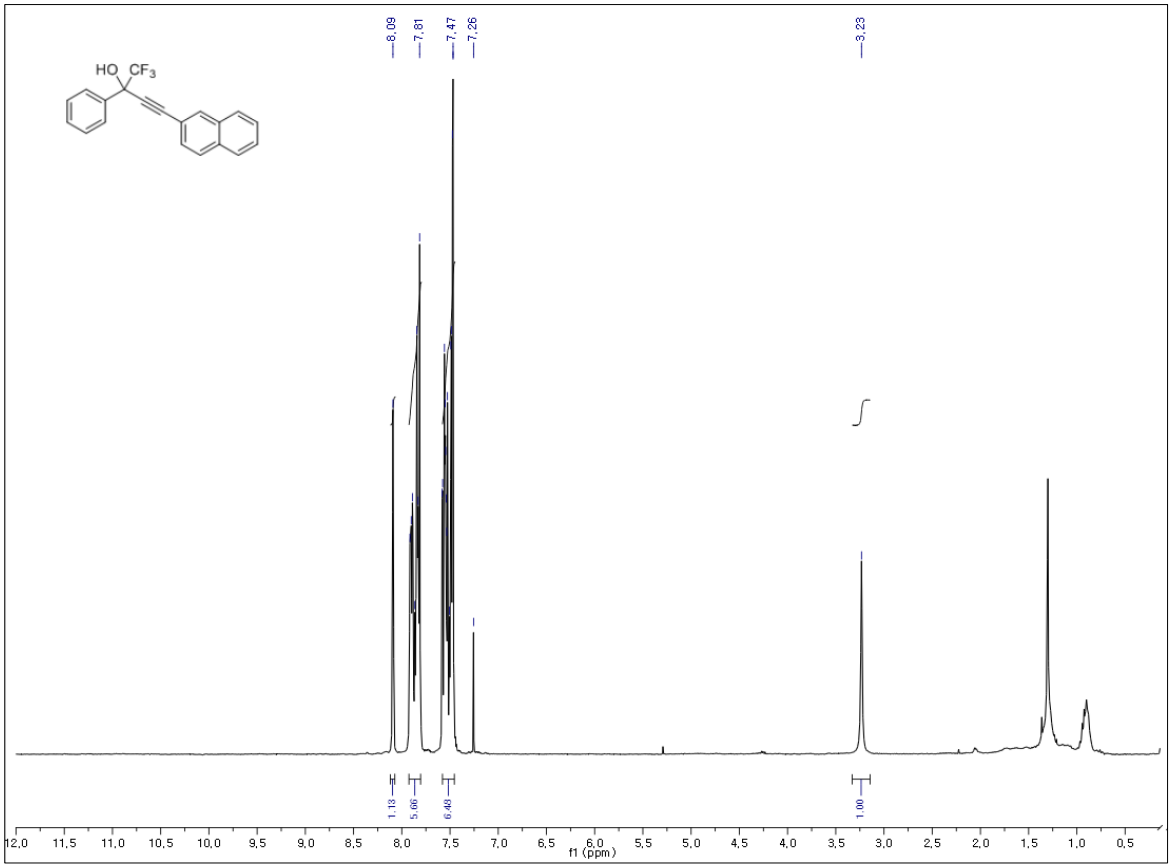

80.06
HO
CF3
Br
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
ppm


HO CF3
OMe
7.83
7.82
7.81
7.45
7.26
6.89
6.88
6.87
6.86
3.83
3.12
2.42
6.18
2.42
3.53
1.00
ppm


HO CF3
OMe
160.59
135.57
133.72
129.52
128.29
127.31
114.18
113.02
88.28
83.31
73.67
73.24
55.45
29.69
ppm


HO
CF3
OMe
-80.19
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190
ppm


HO
CF3
7.83
7.81
7.80
7.80
7.44
7.41
7.26
7.18
7.15
3.09
2.38
1.95
5.08
2.09
1.00
3.10
10,0 9,5 9,0 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 0,5
ppm


HO
CF3
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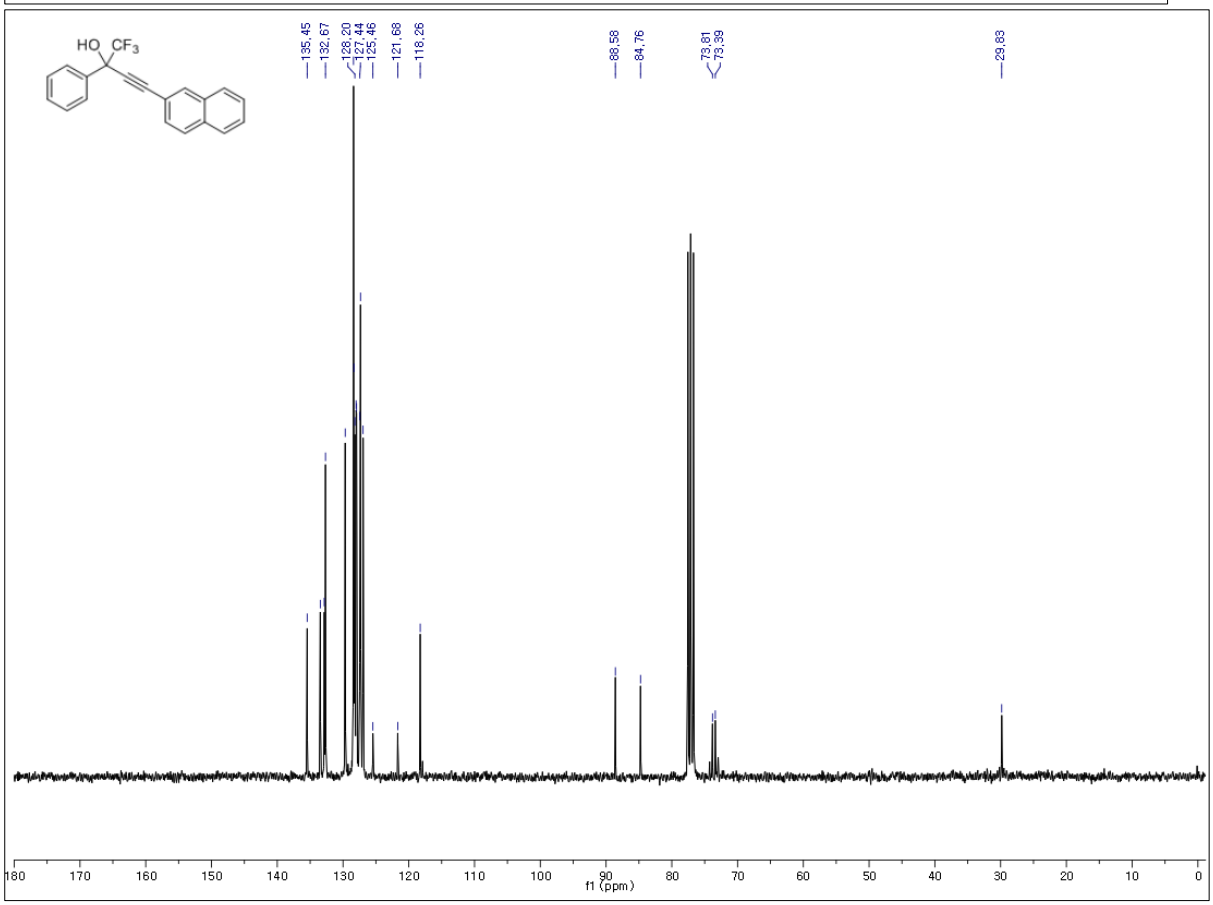
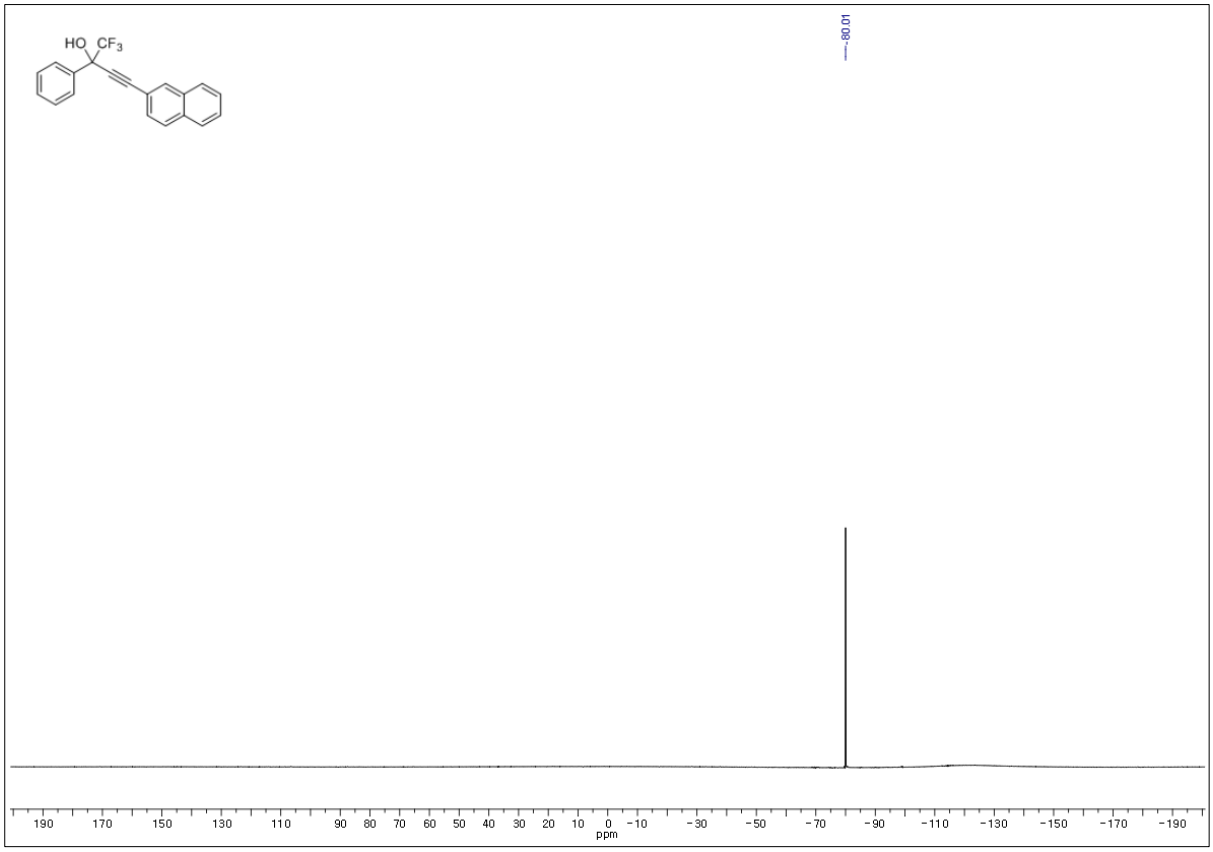
7.81
7.47
7.26
3.23
1.13
5.66
6.49
1.00
f1 (ppm)


HO
CF3
135.45
132.67
128.20
127.44
125.46
121.68
118.26
88.58
84.76
73.81
73.39
29.83
f1 (ppm)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CF3
-80.01
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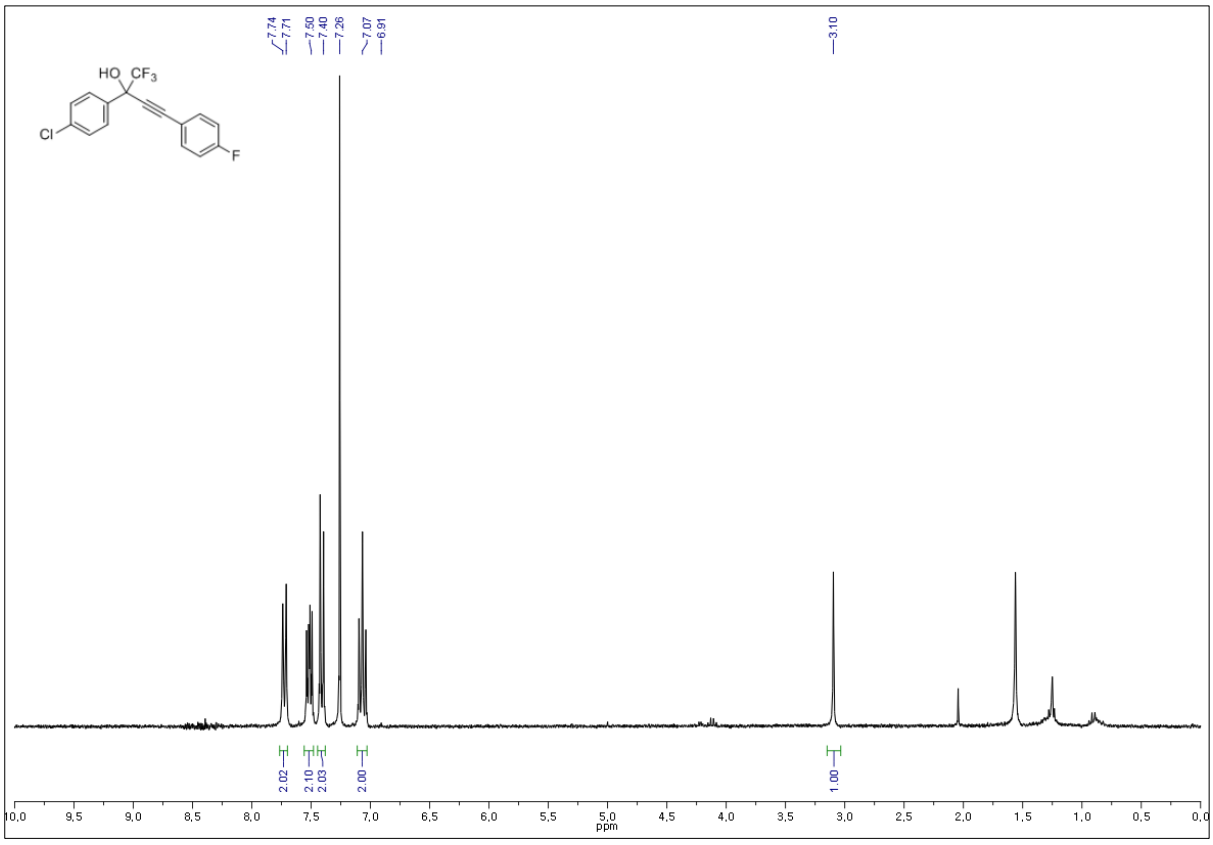
ppm


HO
CF3
Cl
F
7.74
7.71
7.50
7.40
7.26
7.07
6.91
3.10
2.02
2.10
2.03
2.00
1.00
10,0 9,5 9,0 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 0,5 0,0
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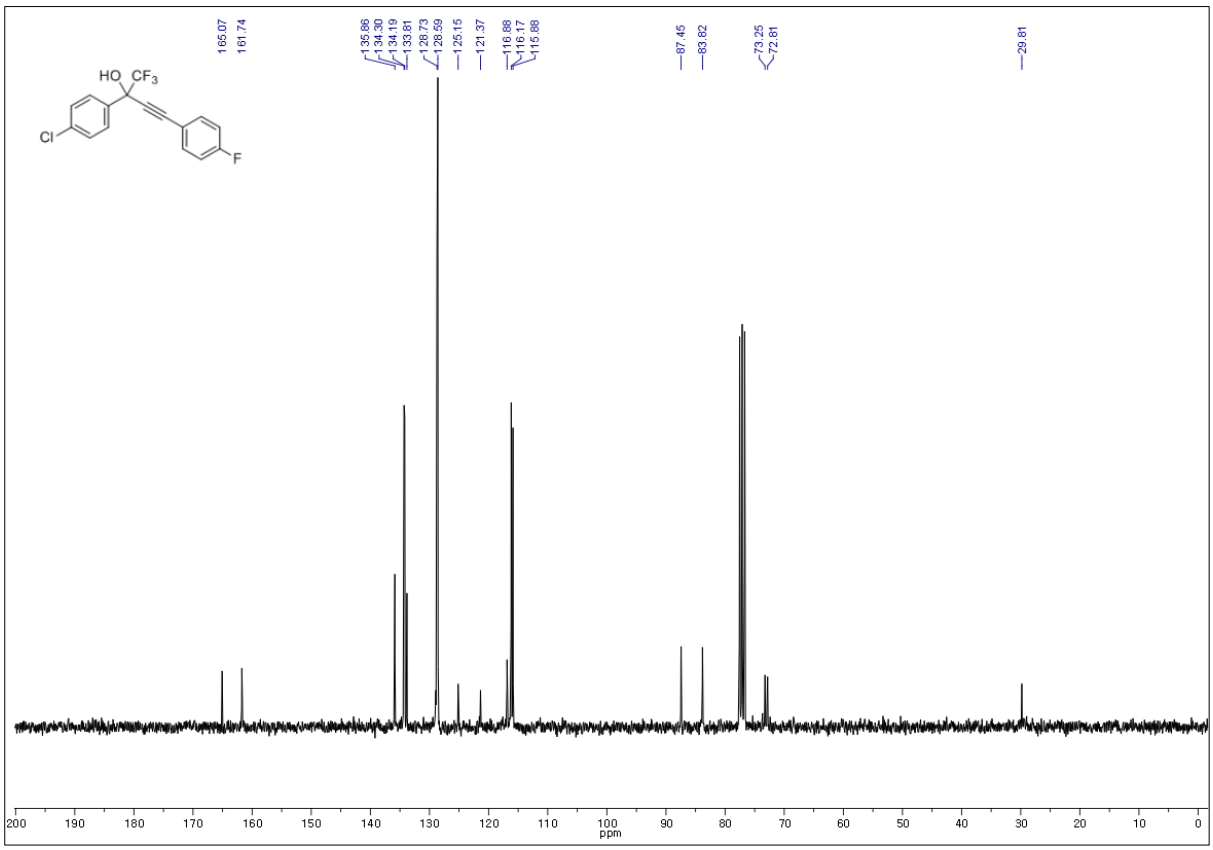
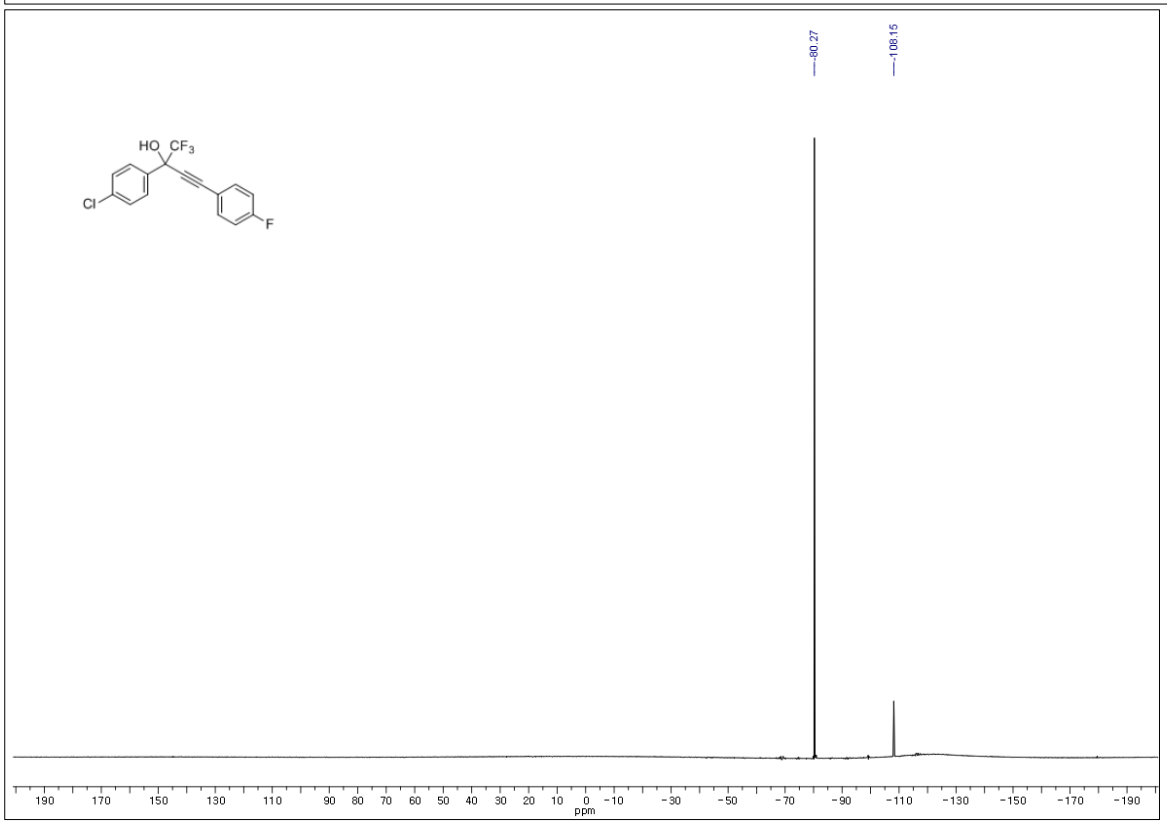

165.07
161.74
135.86
134.30
134.19
133.81
128.73
128.59
125.15
121.37
116.88
116.17
115.88
87.45
83.82
73.25
72.81
29.81
HO
CF3
Cl
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


-80.27
-108.15
HO
CF3
Cl
F
190
170
150
130
110
90
80
70
60
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-50
-70
-90
-110
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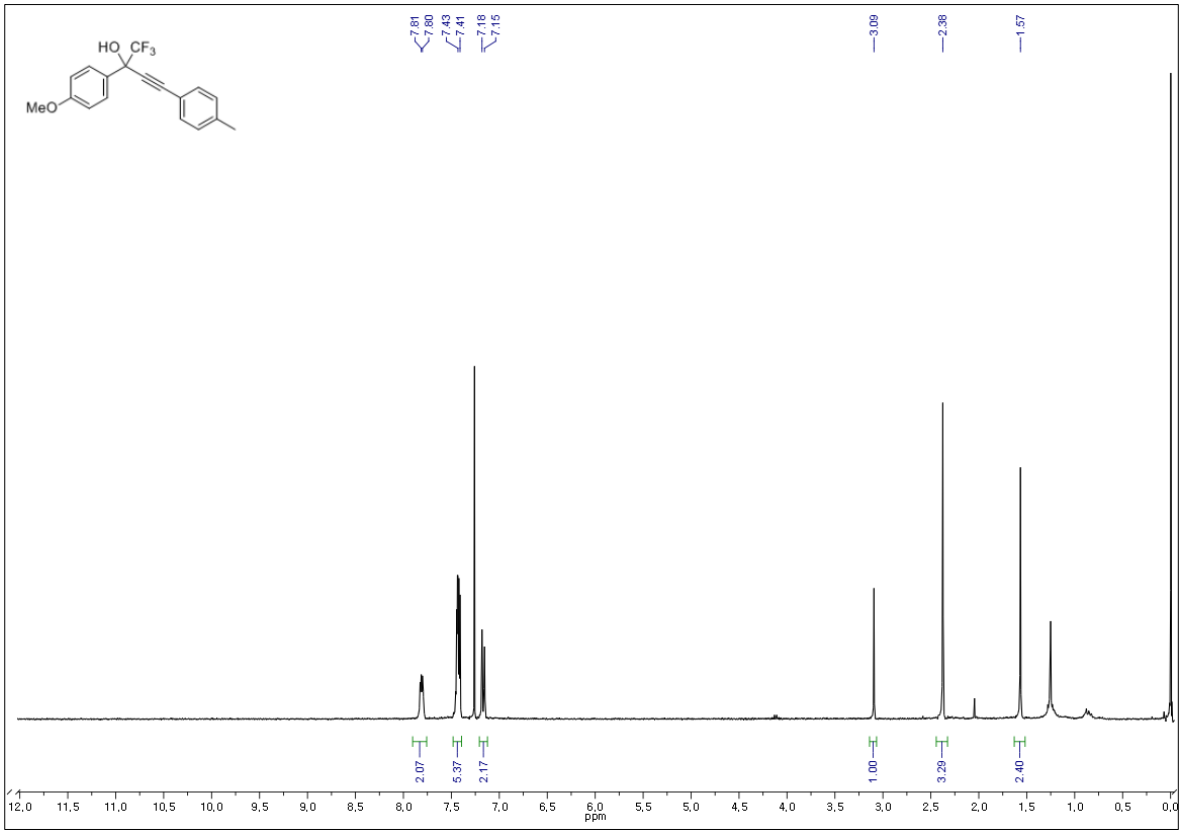
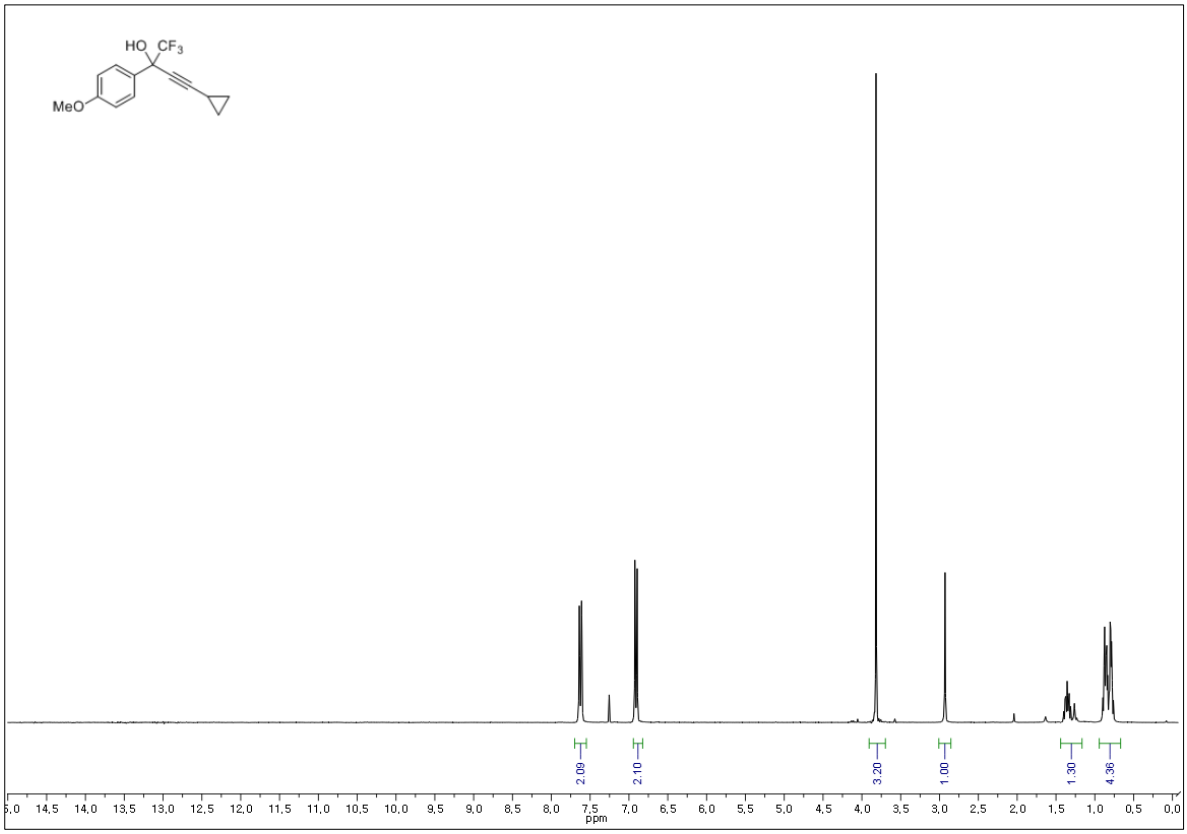
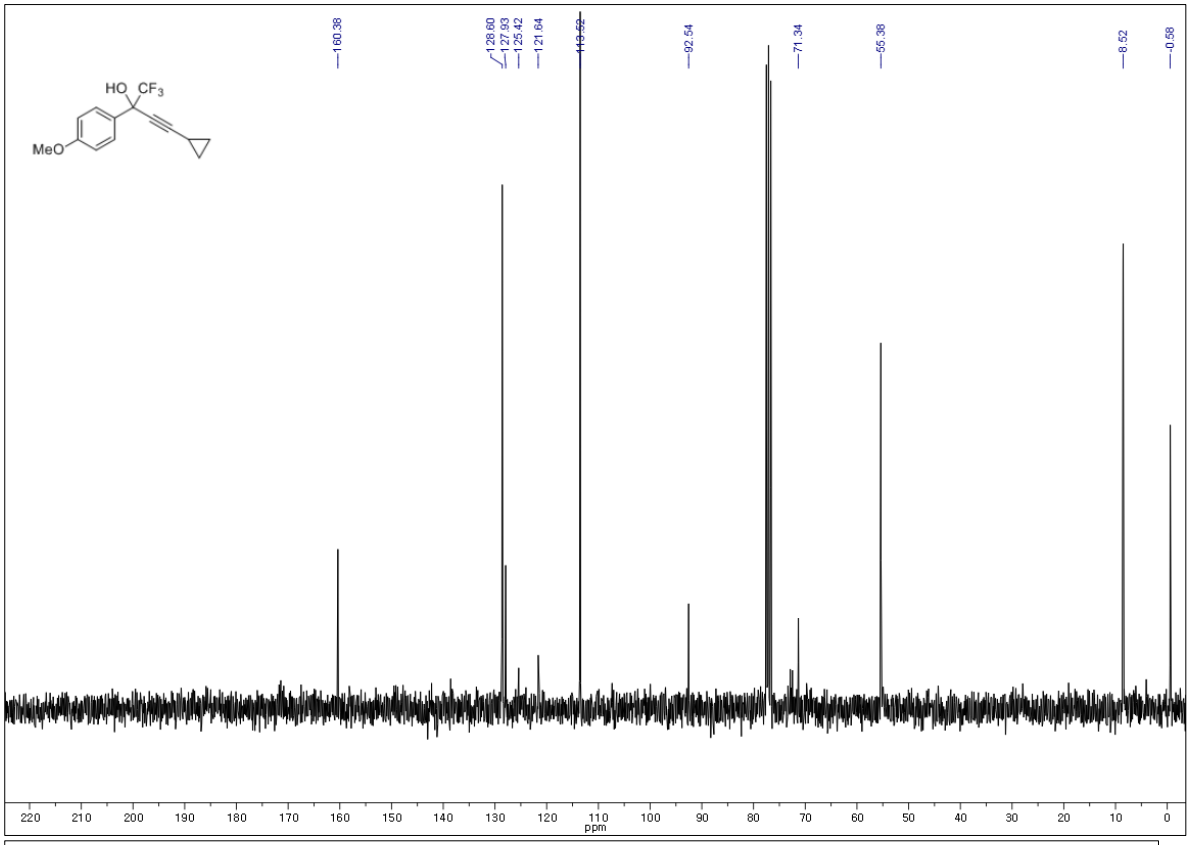
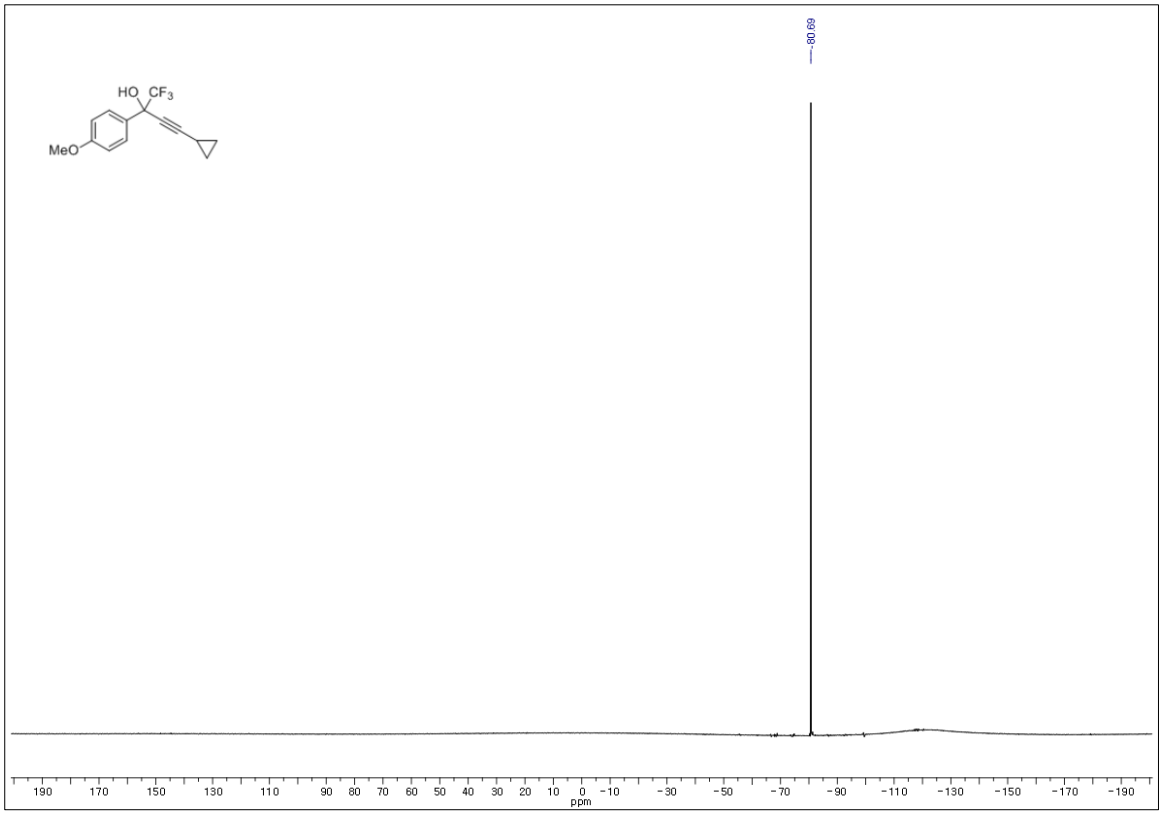
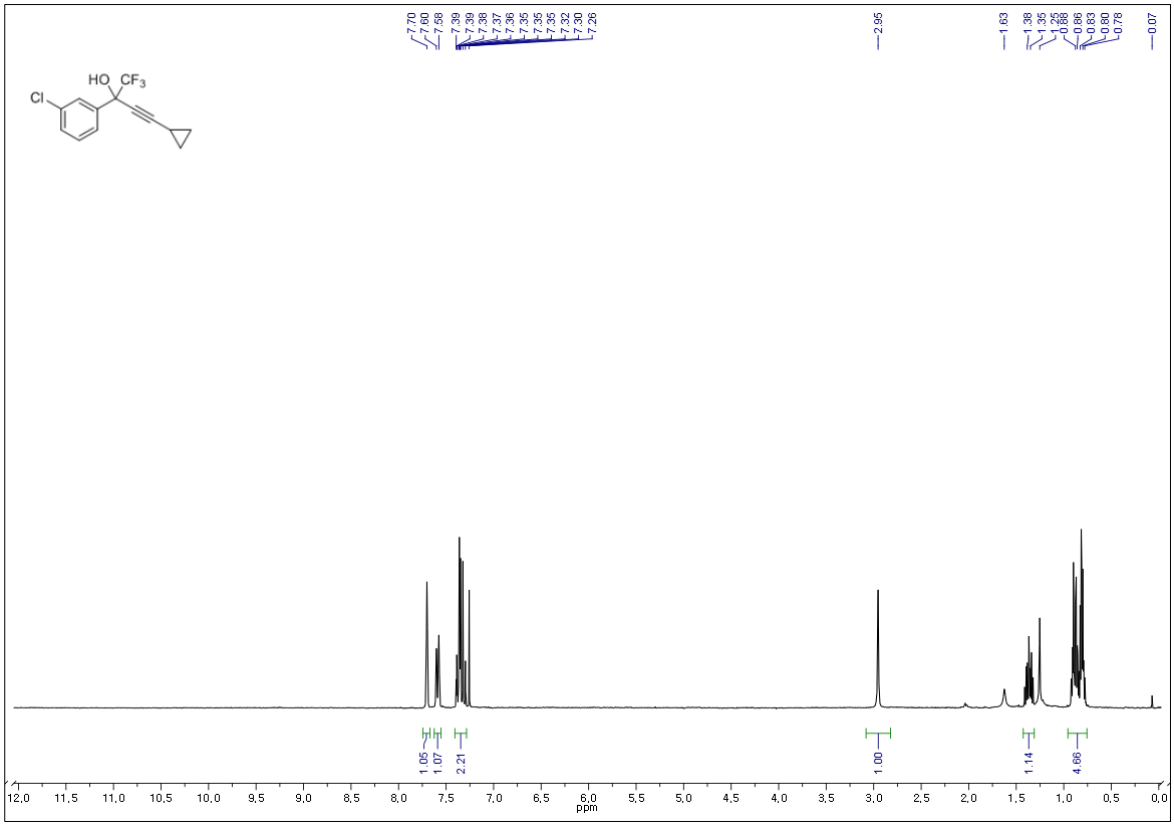
ppm

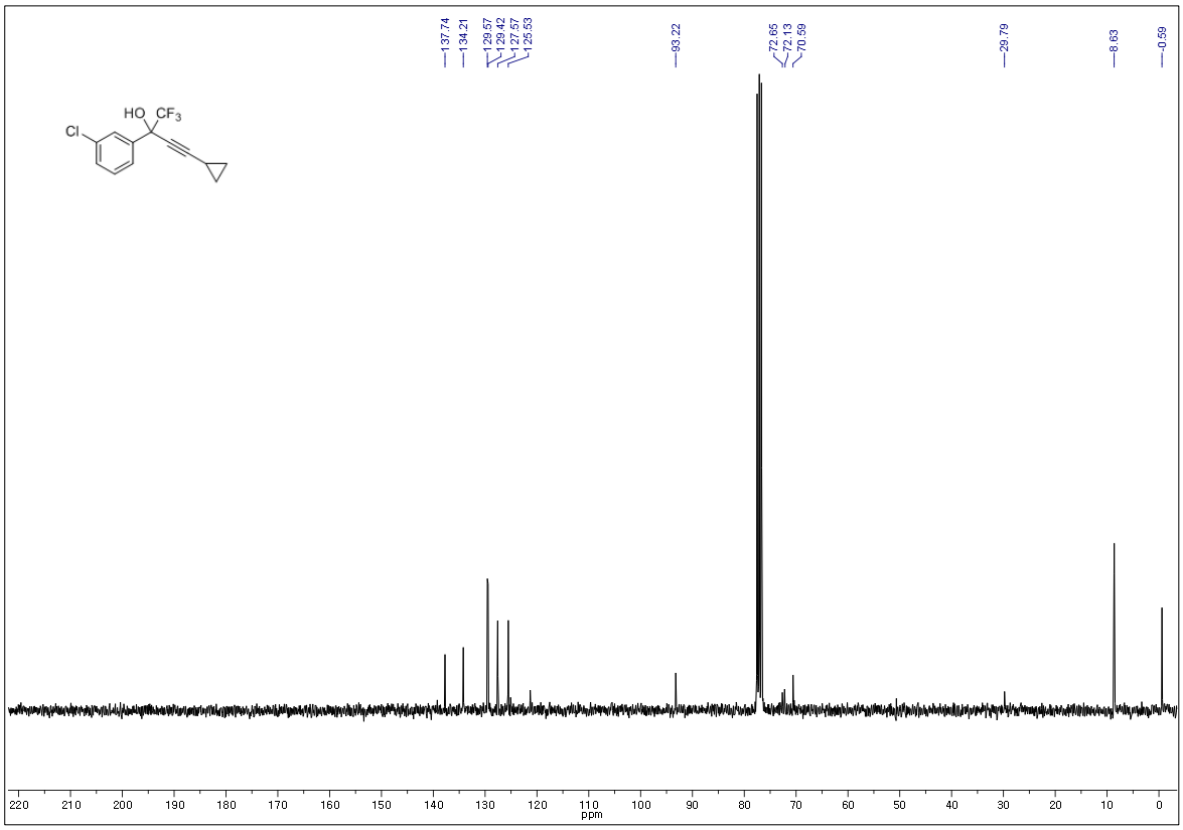

HO
CF3
MeO
7.81
7.80
7.43
7.41
7.18
7.15
3.09
2.38
1.57
2.07
5.37
2.17
1.00
3.29
2.40
ppm

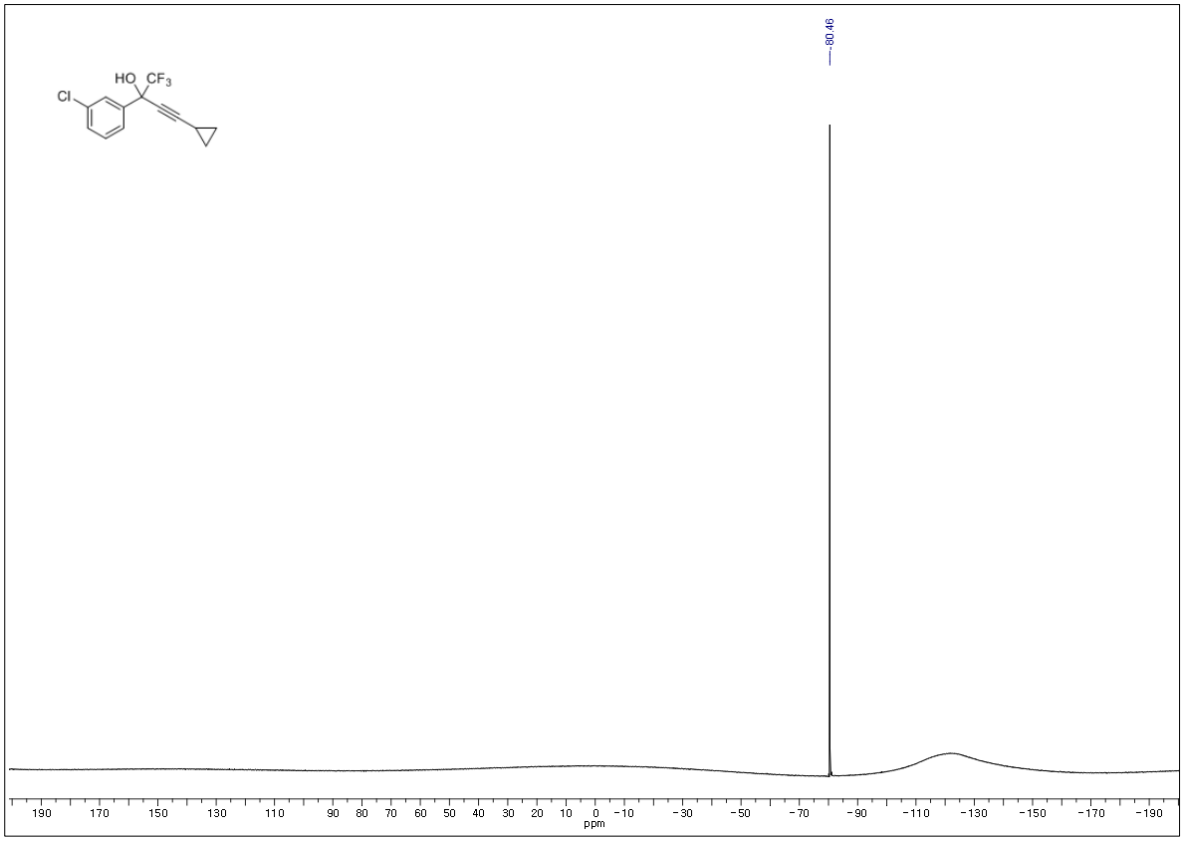

HO
CF3
MeO
2.09
2.10
3.20
1.00
1.30
4.36
ppm


160.38
128.60
127.93
125.42
121.64
113.52
92.54
71.34
55.38
6.52
-0.58
HO
CF3
MeO
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


-80.69
HO
CF3
MeO
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
ppm


HO
CF3
Cl
7.70
7.60
7.58
7.39
7.39
7.38
7.37
7.36
7.35
7.35
7.35
7.32
7.30
7.26
2.95
1.63
1.38
1.35
0.86
0.83
0.80
0.78
0.07
1.05
1.07
2.21
1.00
1.14
4.66
ppm


HO
CF3
Cl
137.74
134.21
129.57
129.42
127.57
125.53
93.22
72.65
72.13
70.59
29.79
8.63
-0.59
ppm


HO
CF3
Cl
-80.46
190
170
150
130
110
90
80
70
60
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-10
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-50
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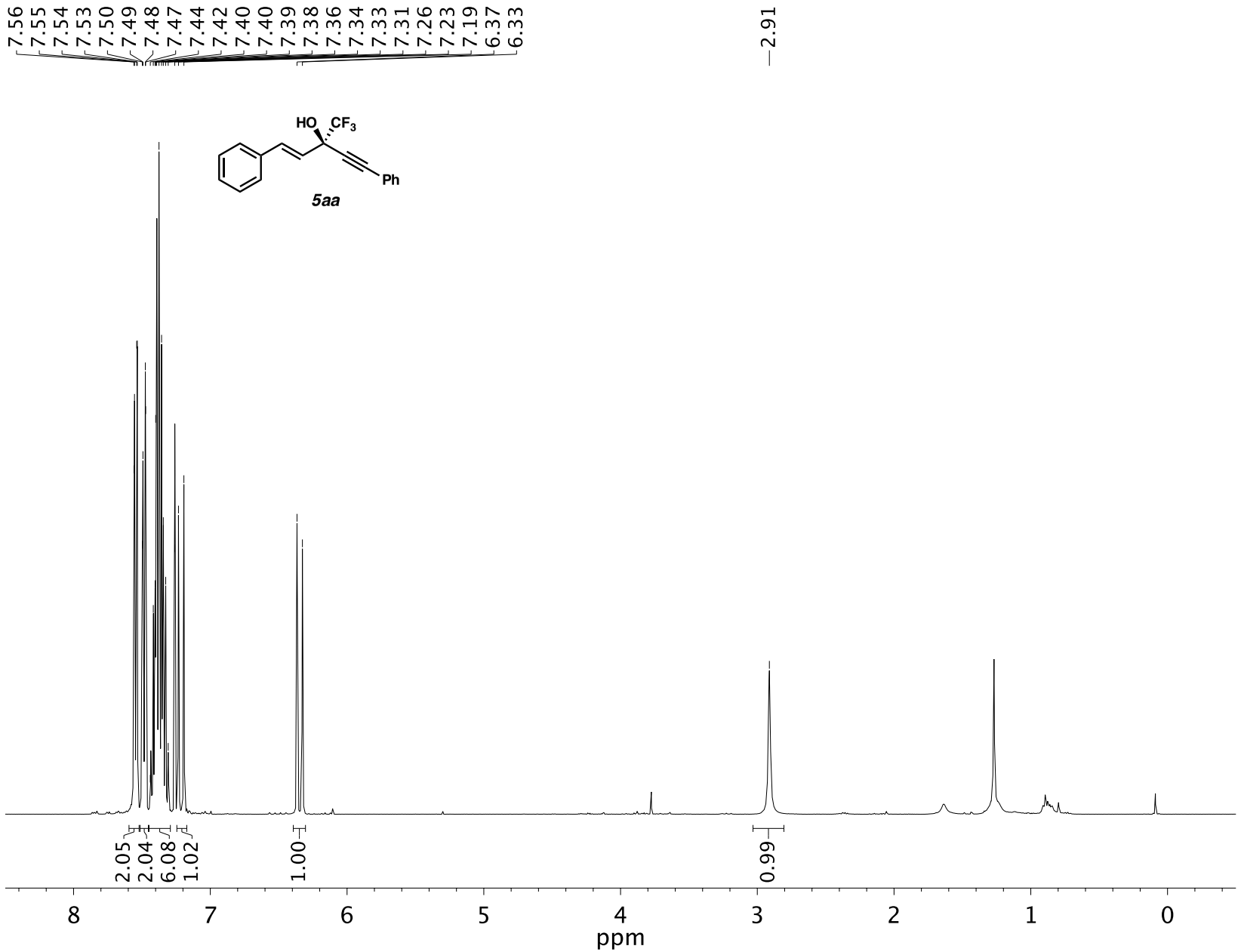
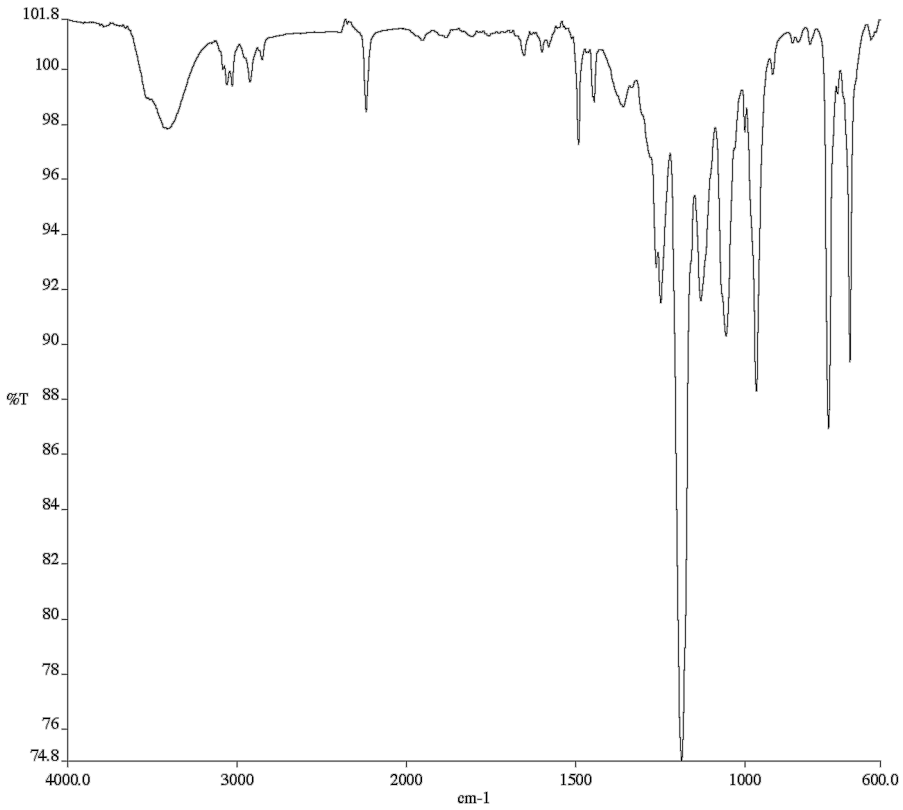
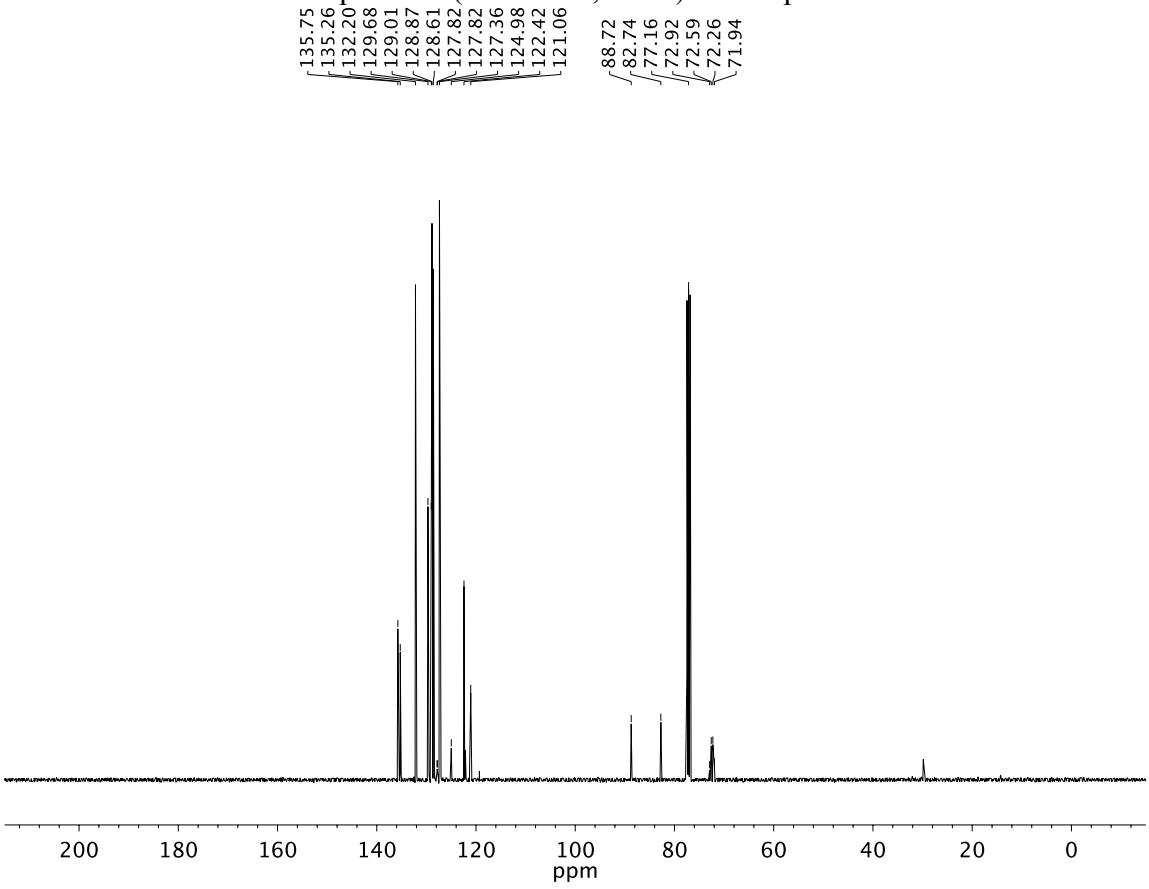
-90
-110
-130
-150
-170
-190
ppm



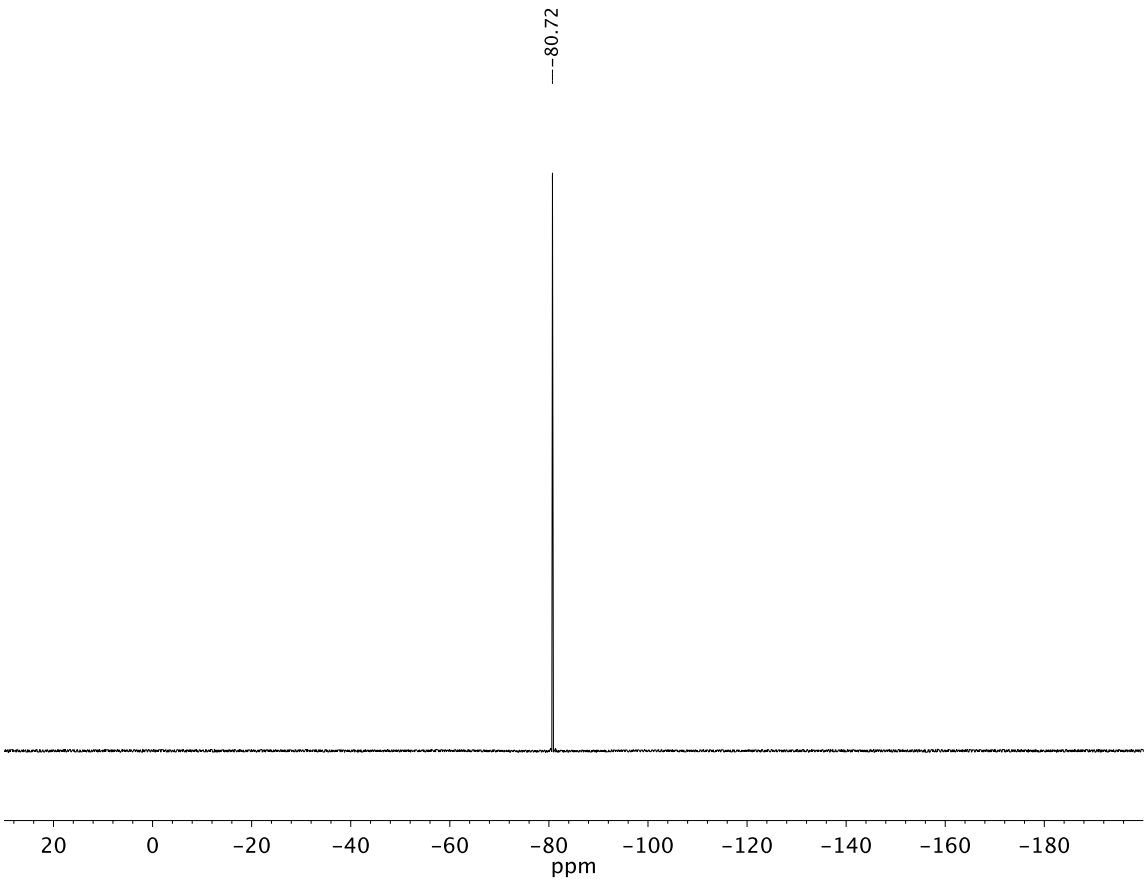
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5aa**



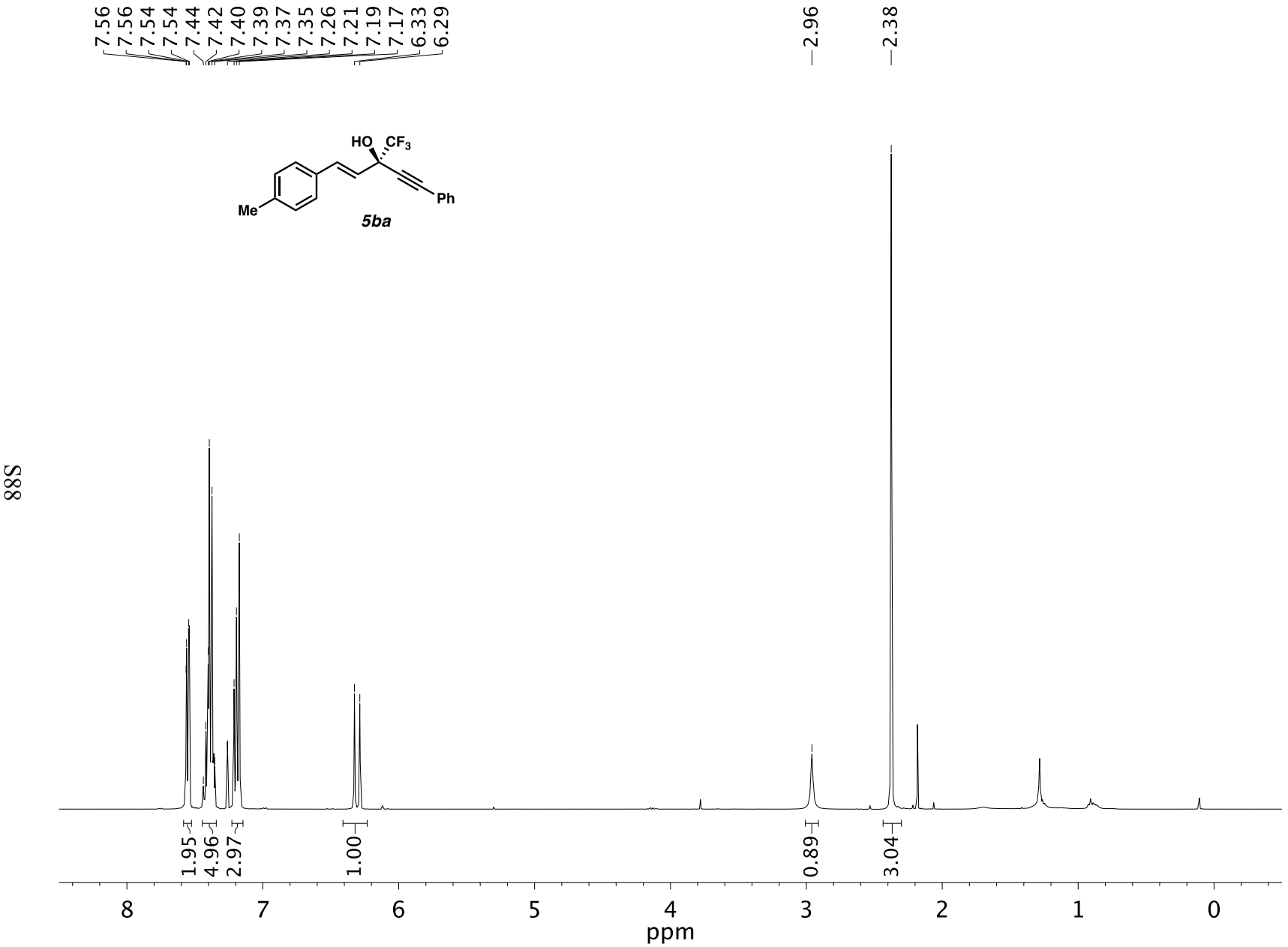
Infrared spectrum (Thin Film, NaCl) of compound **5aa**.



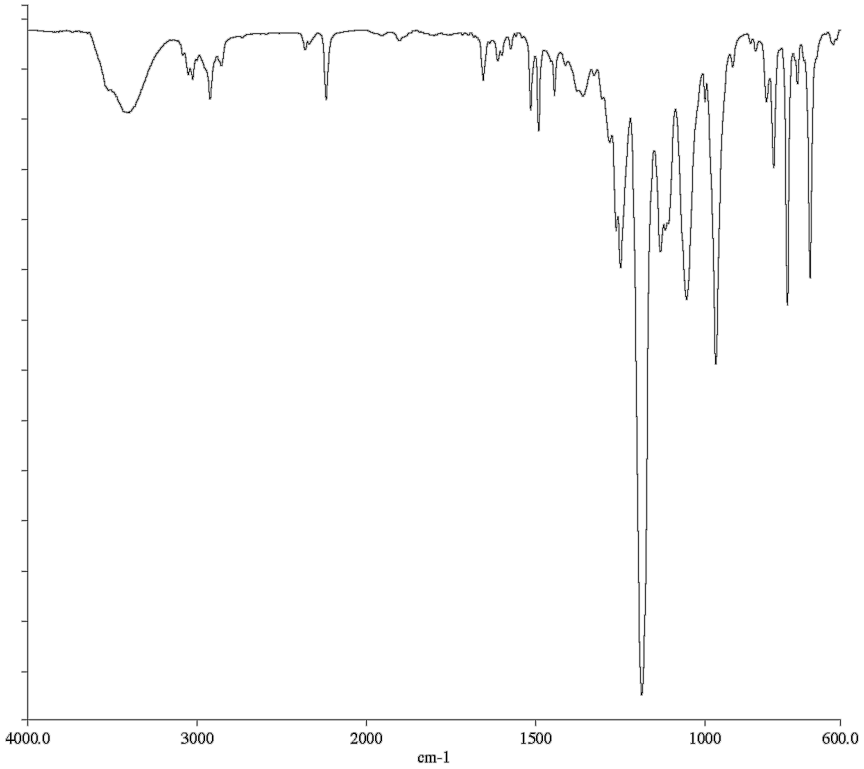
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5aa**



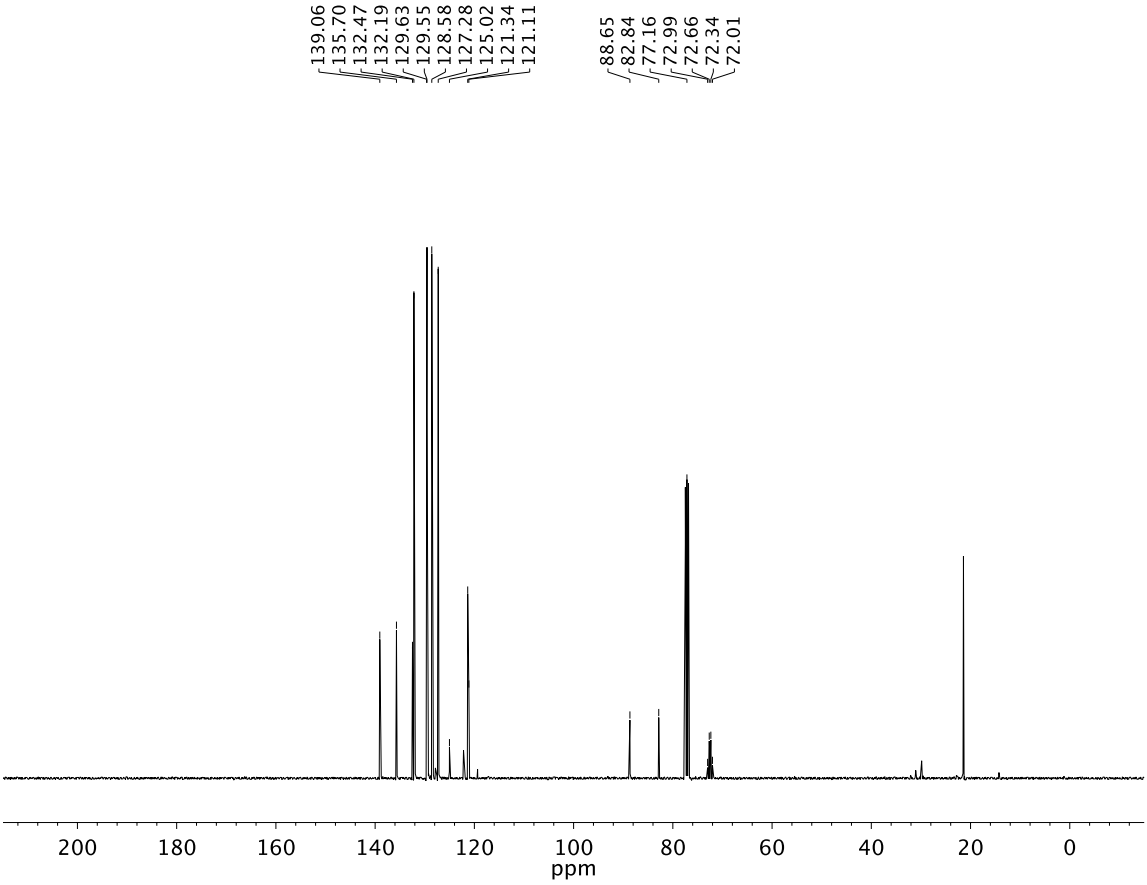
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5aa**



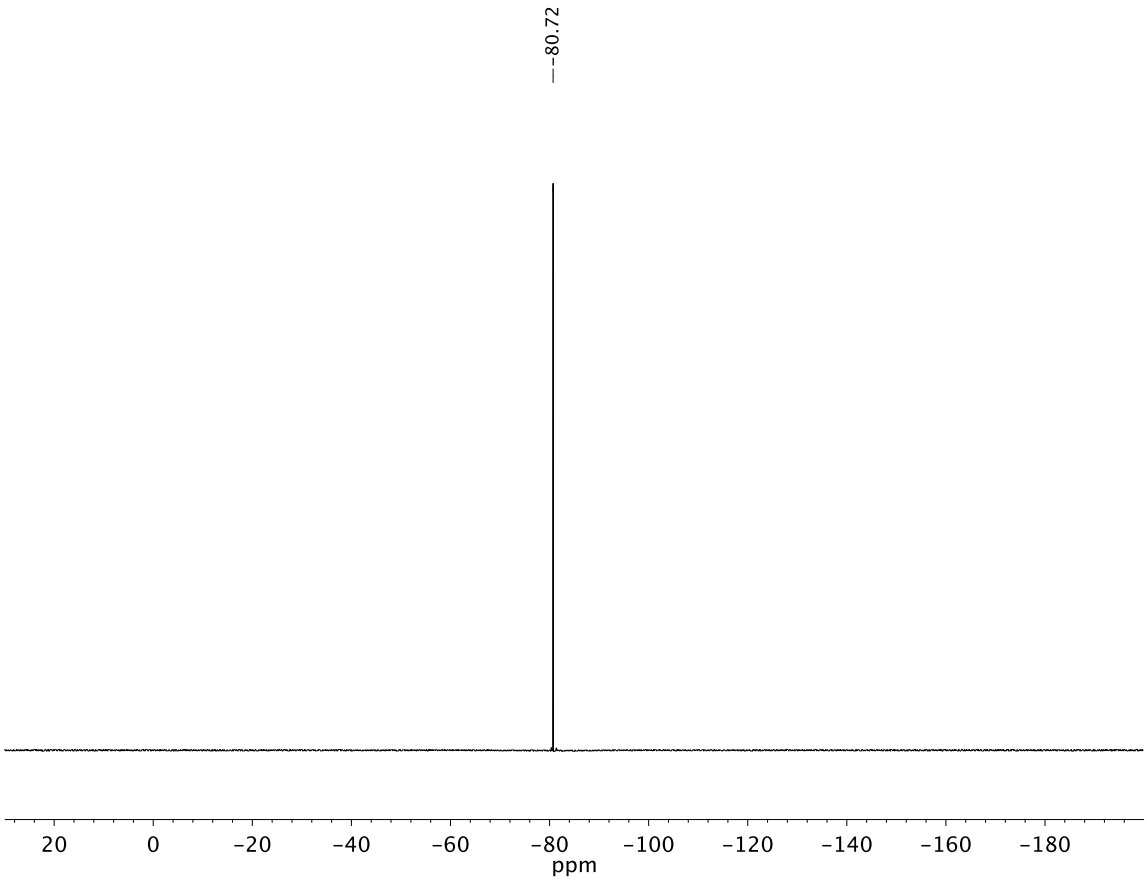
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ba**



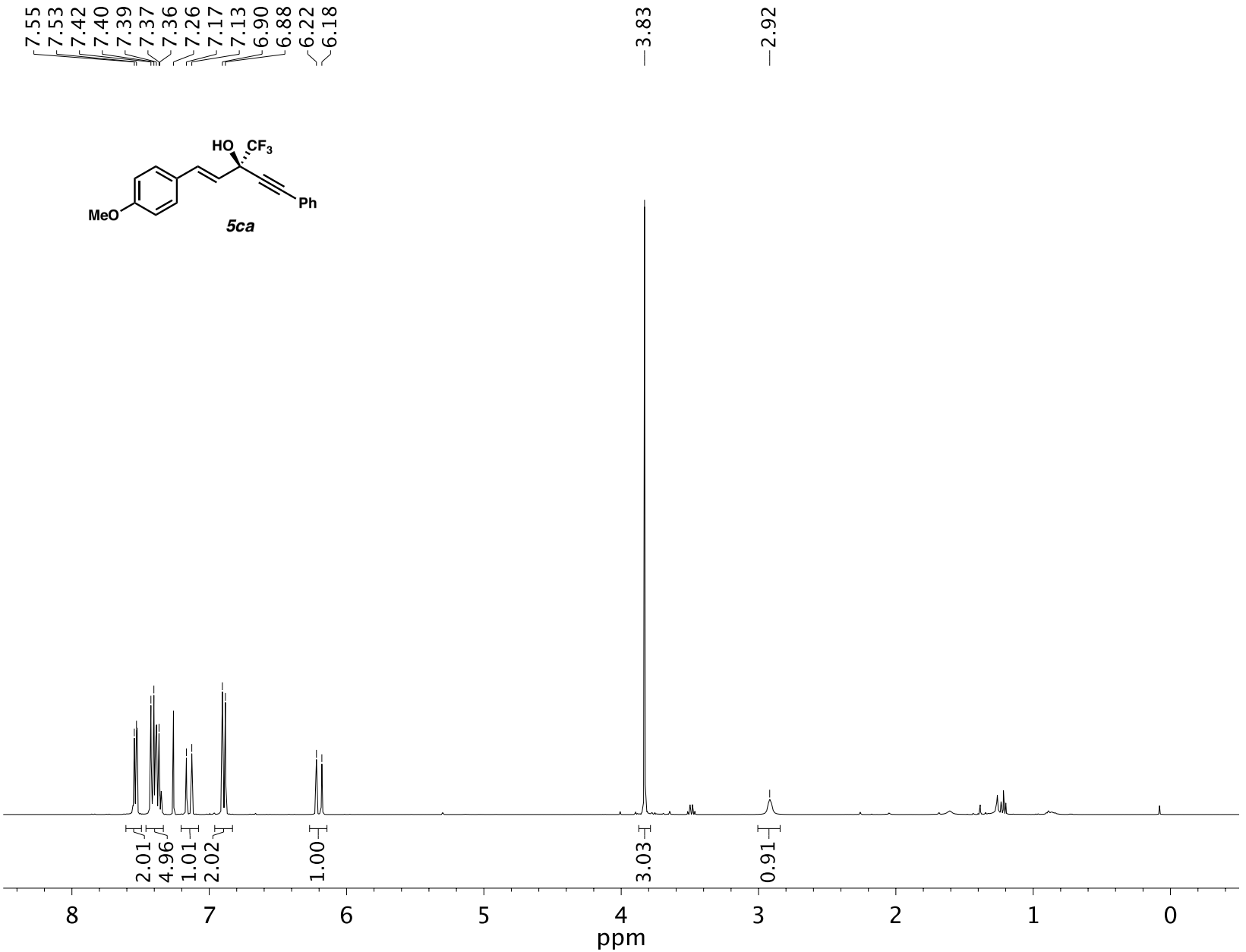
Infrared spectrum (Thin Film, NaCl) of compound **5ba.**



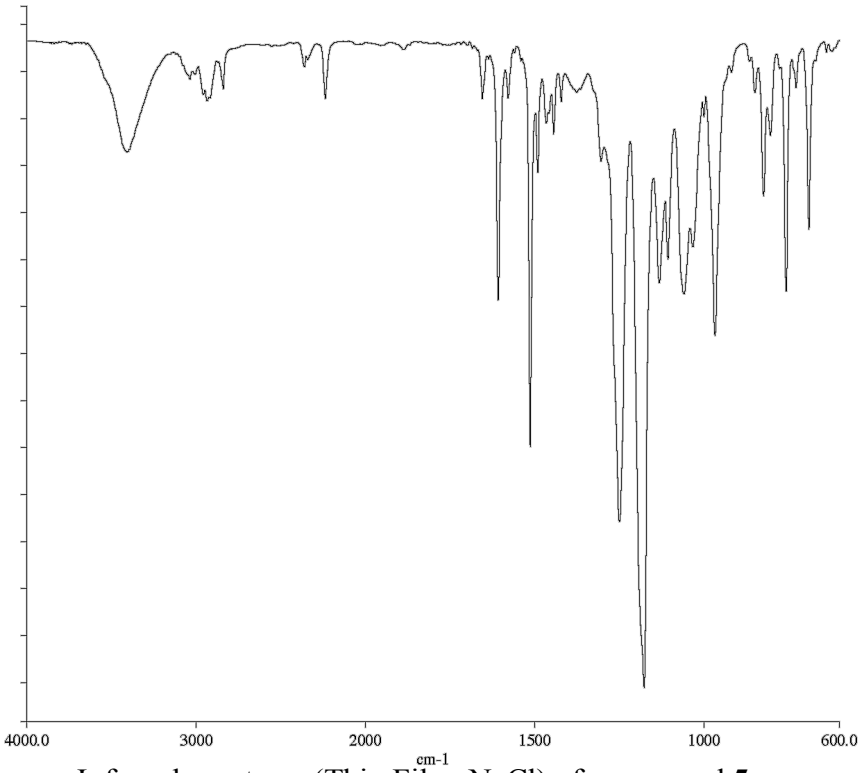
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ba**



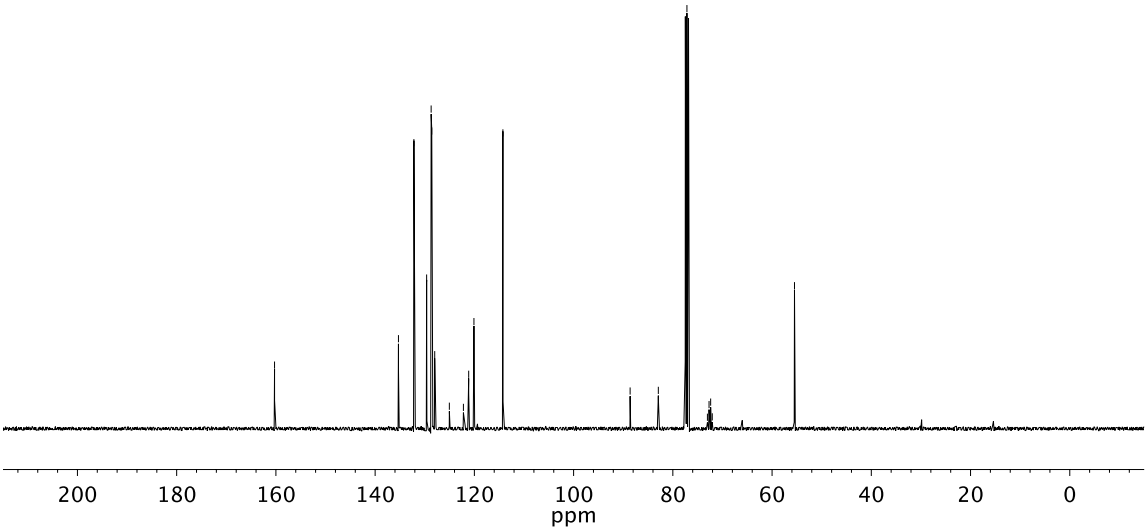
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ba**



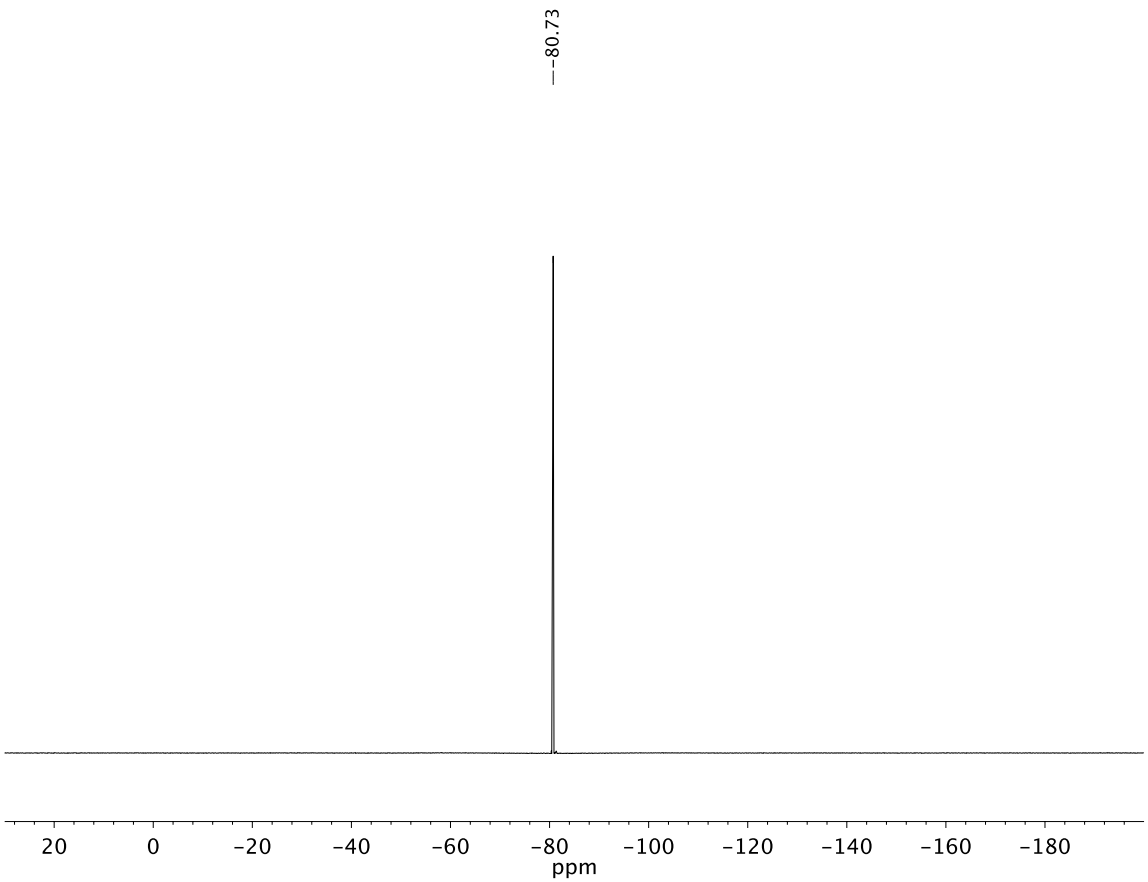
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ca**



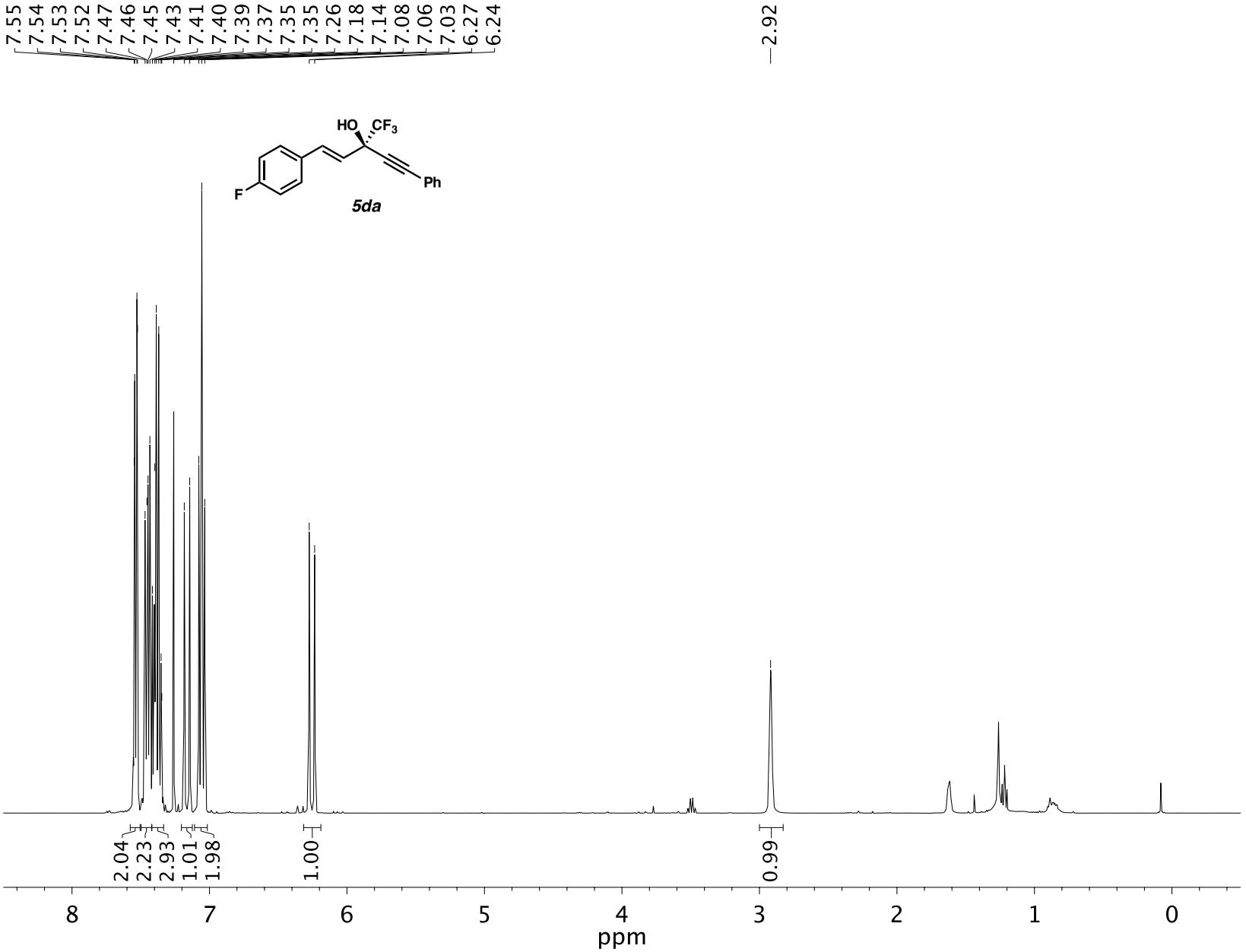
Infrared spectrum (Thin Film, NaCl) of compound **5ca**.



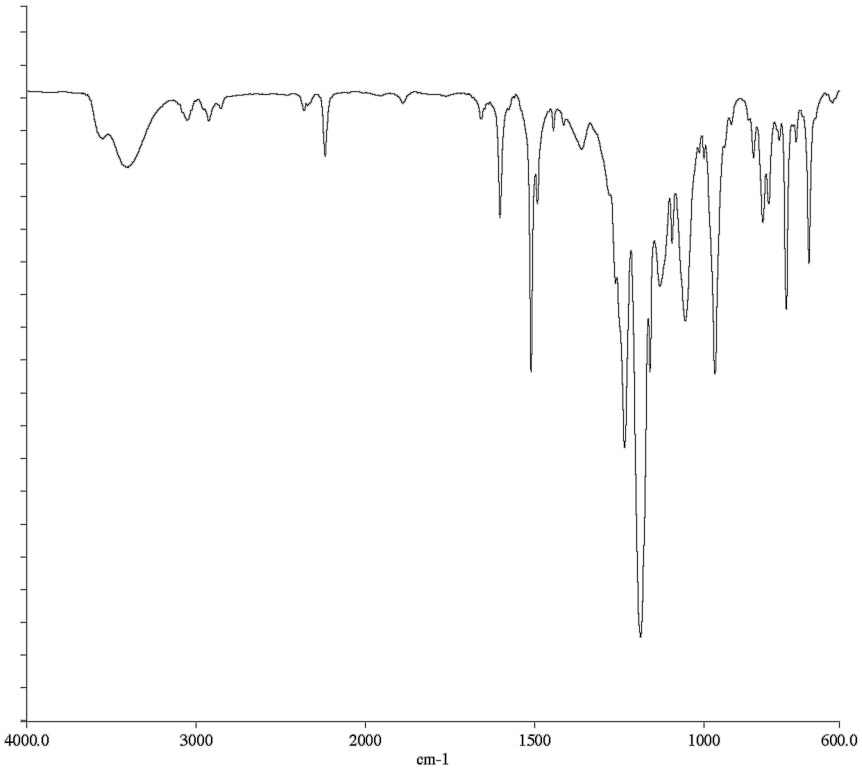
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ca**



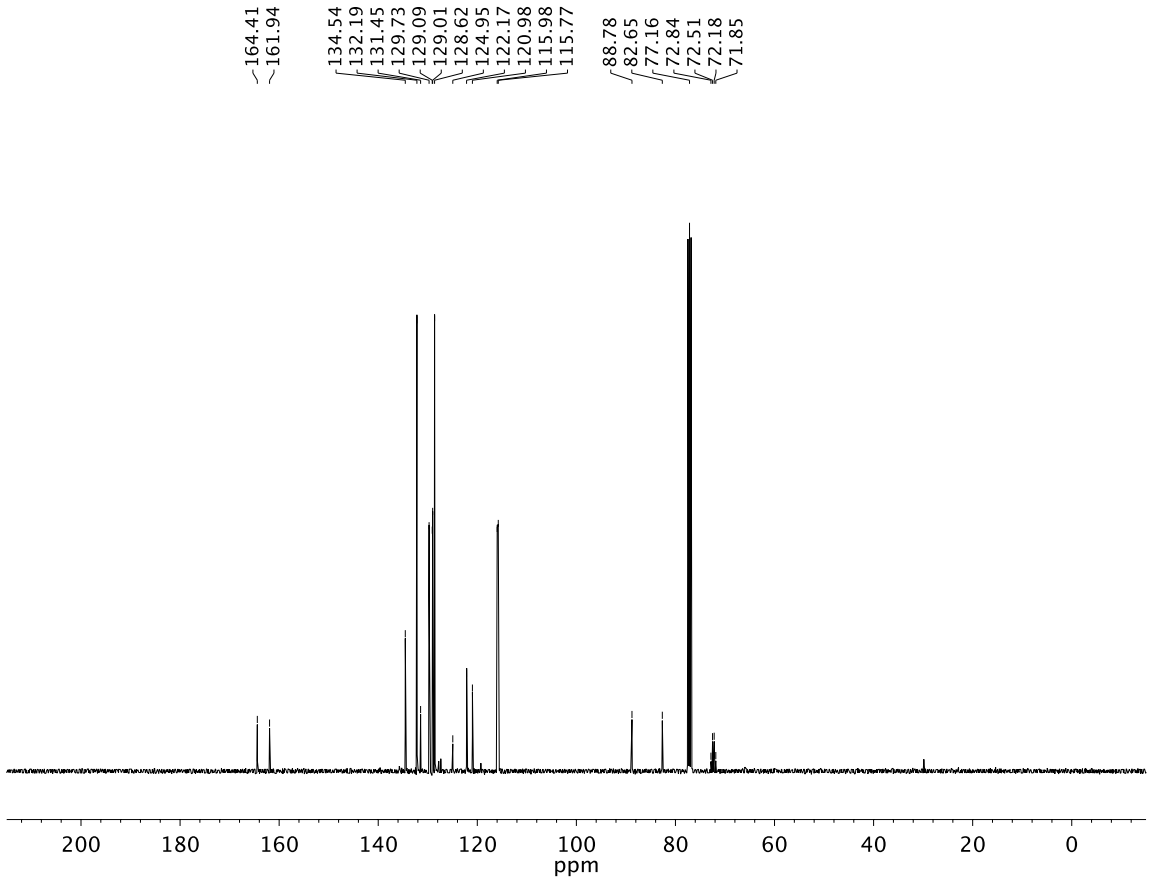
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ca**



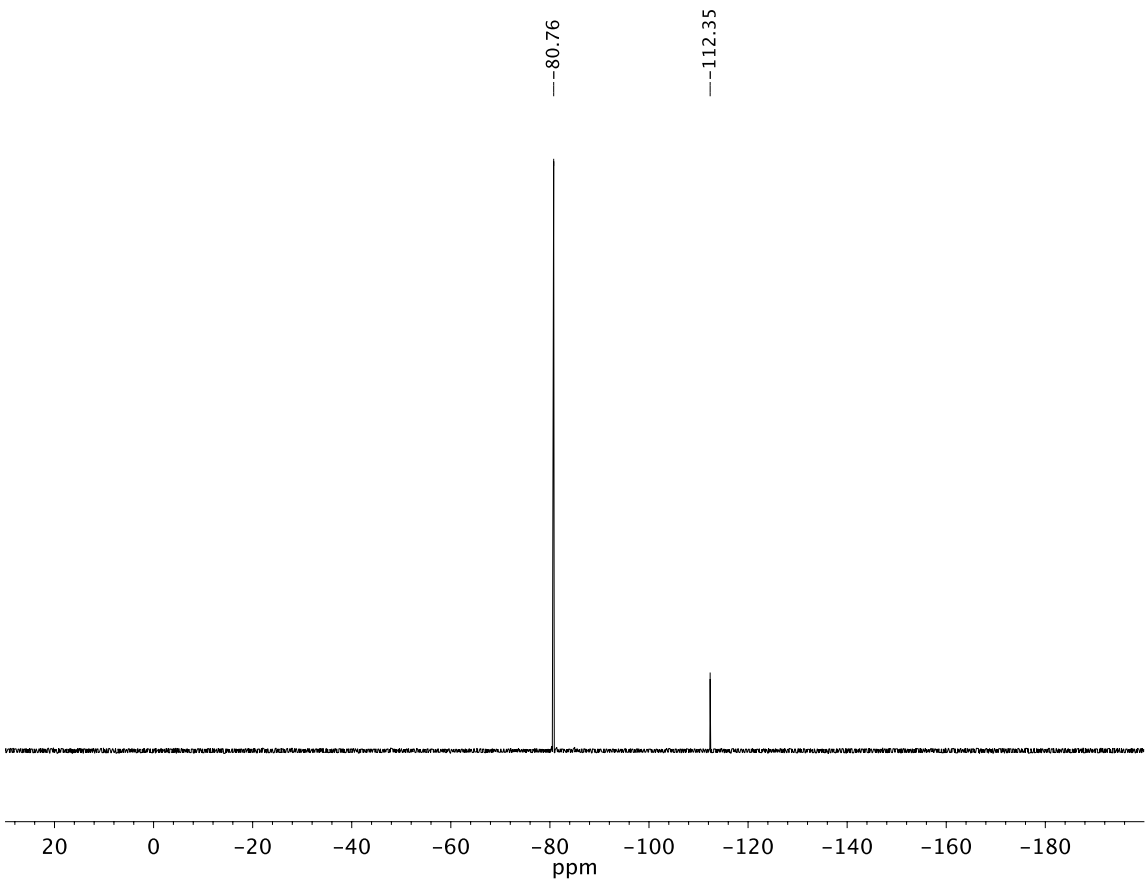
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5da**



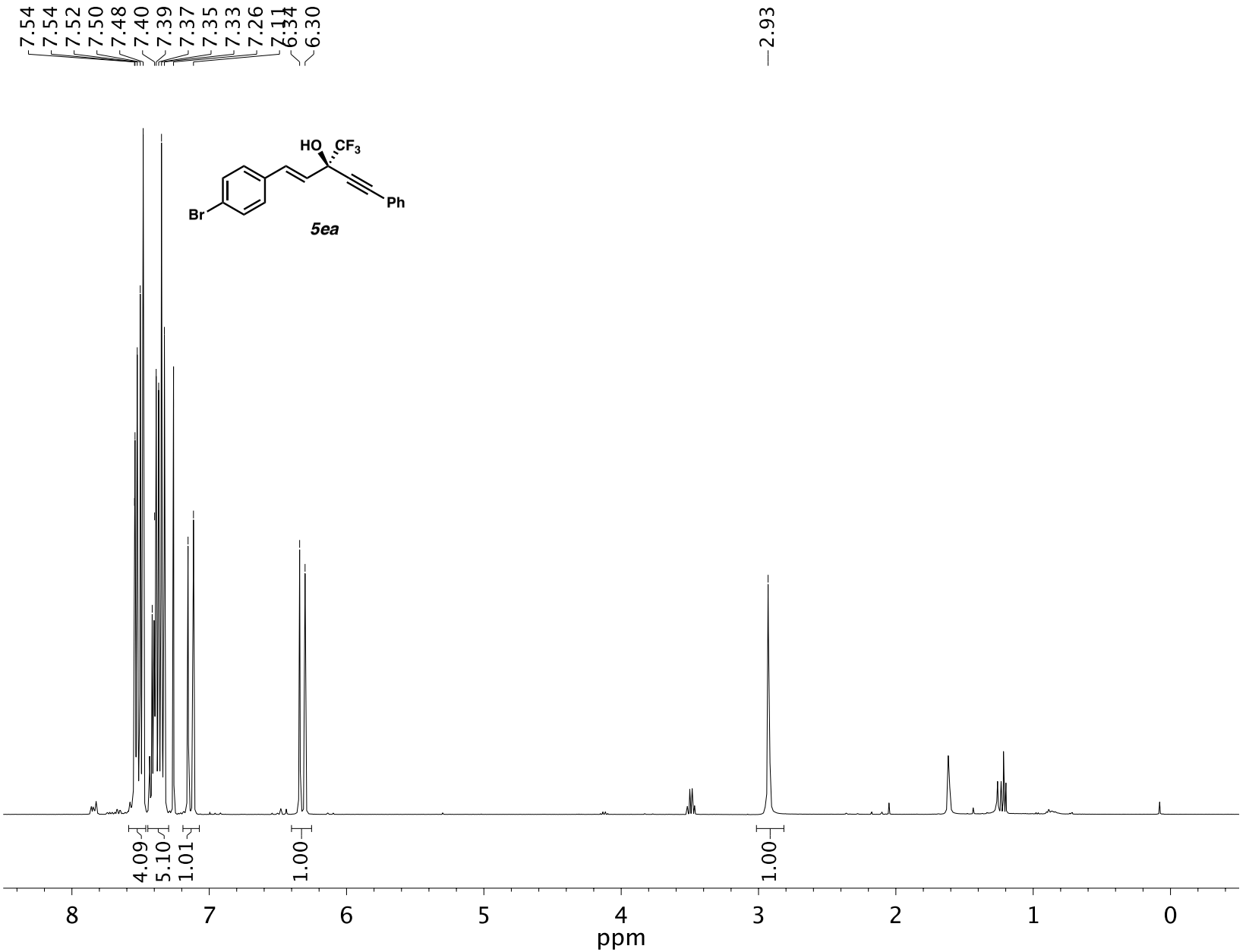
Infrared spectrum (Thin Film, NaCl) of compound **5da**



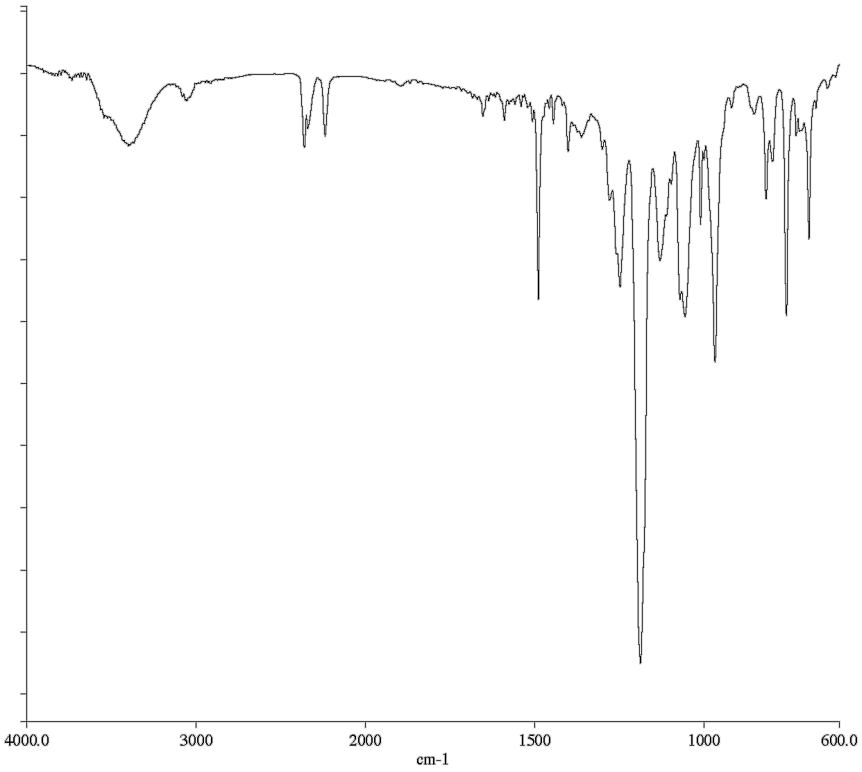
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5da**



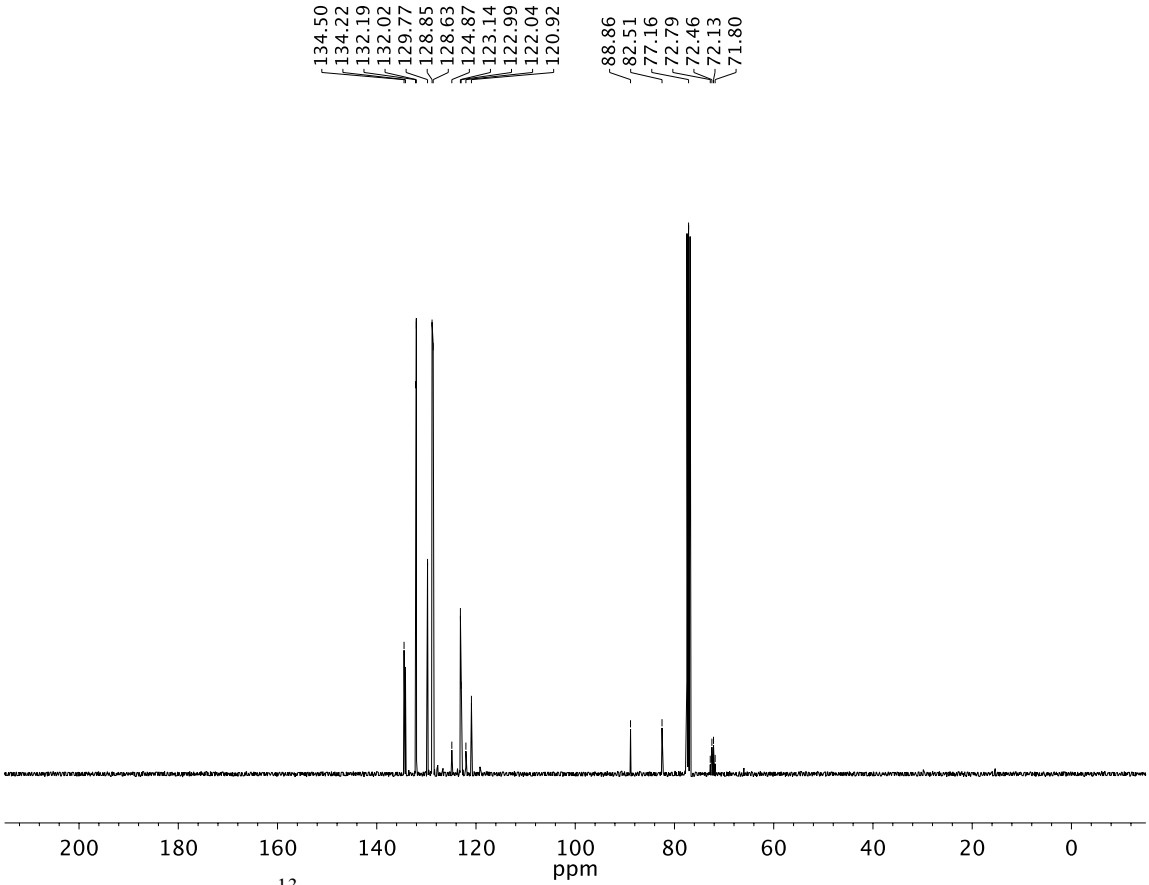
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5da**



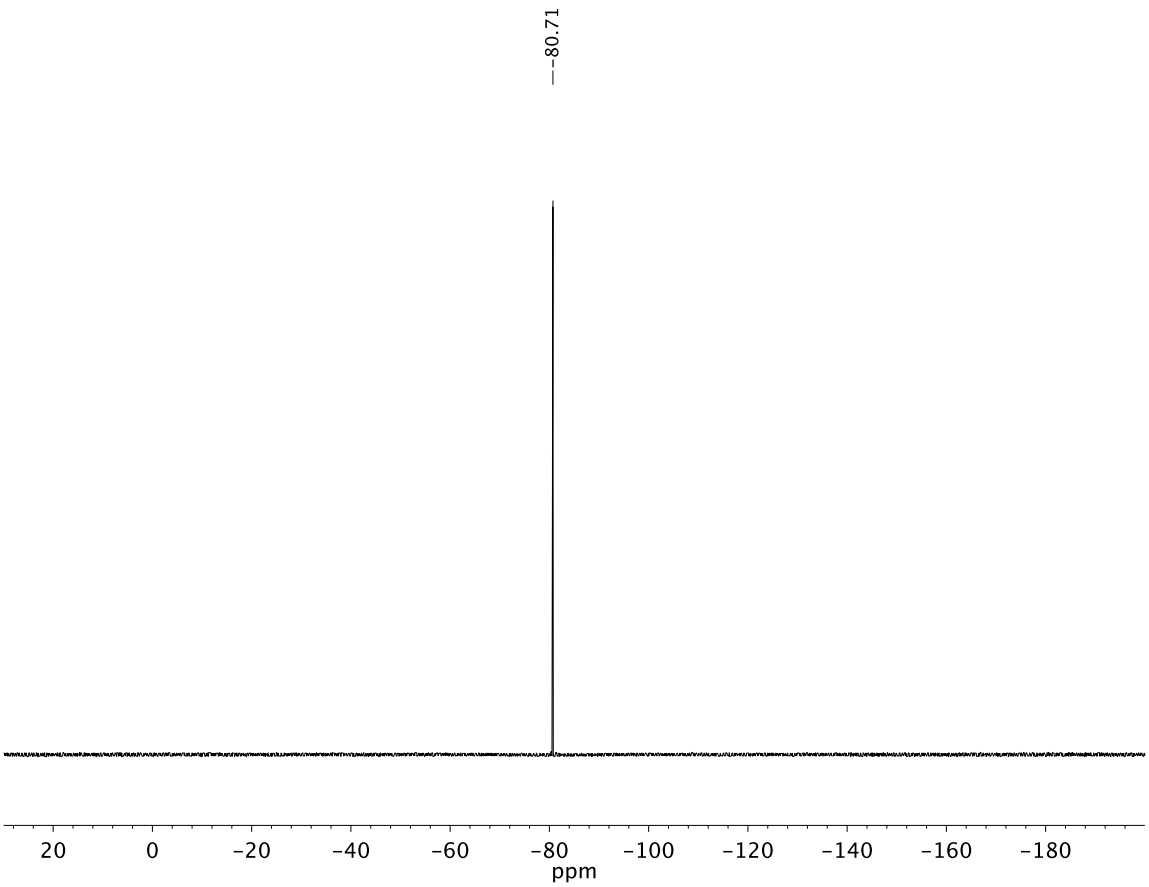
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ea**



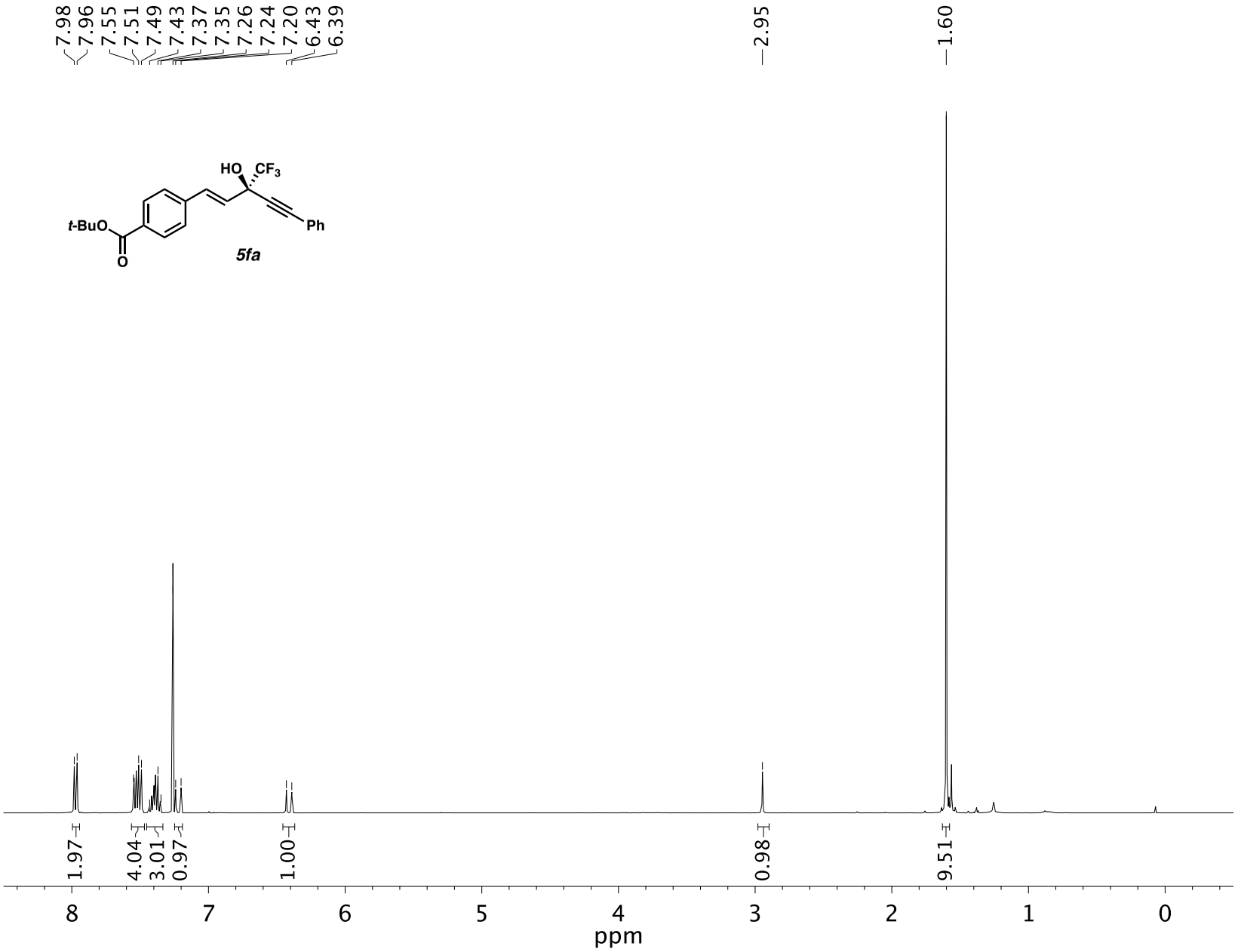
Infrared spectrum (Thin Film, NaCl) of compound **5ea**



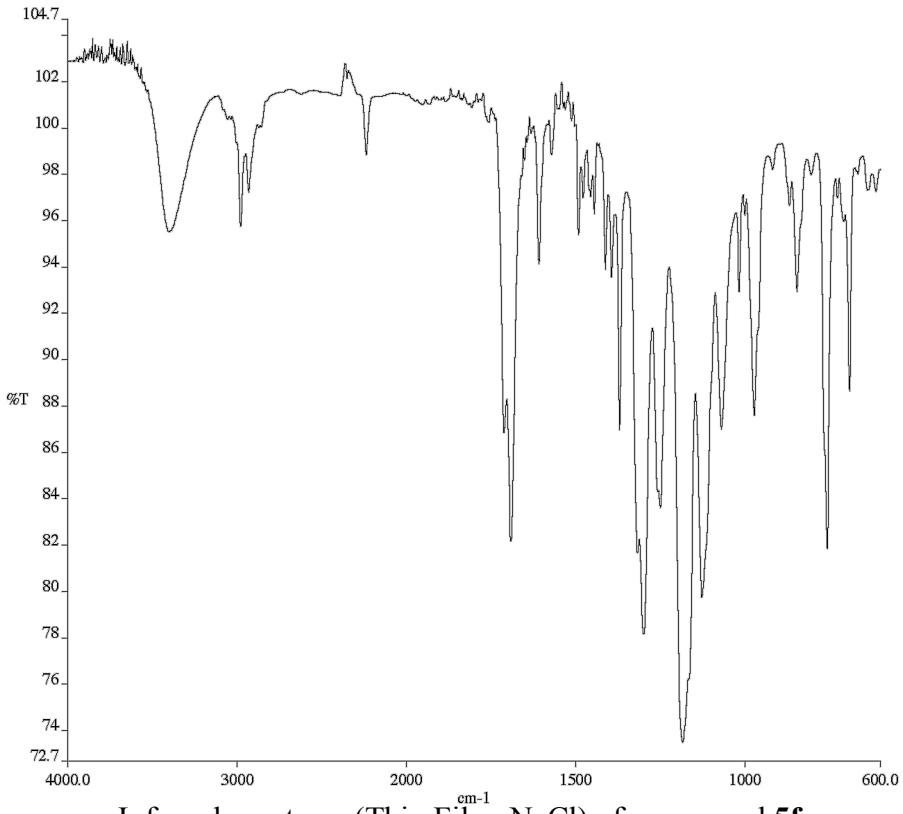
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ea**



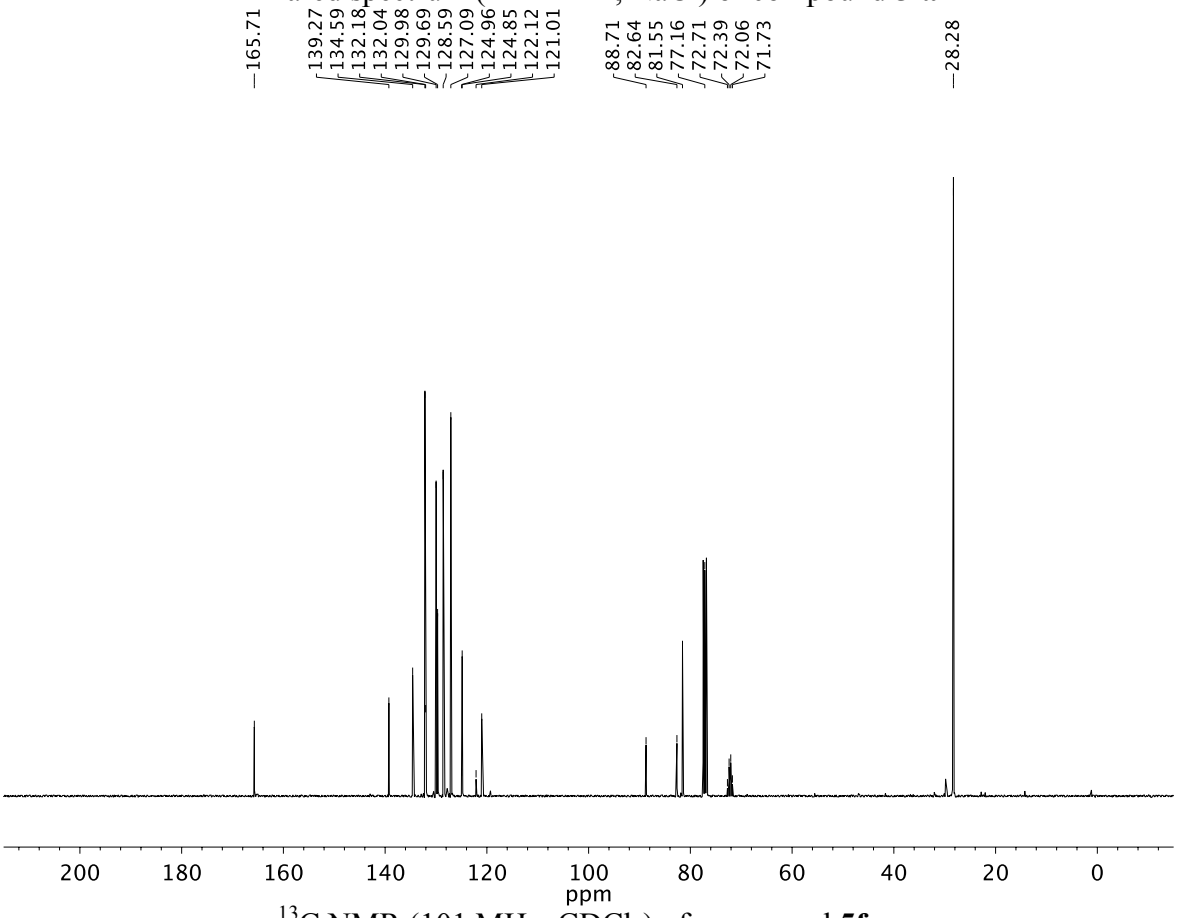
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ea**



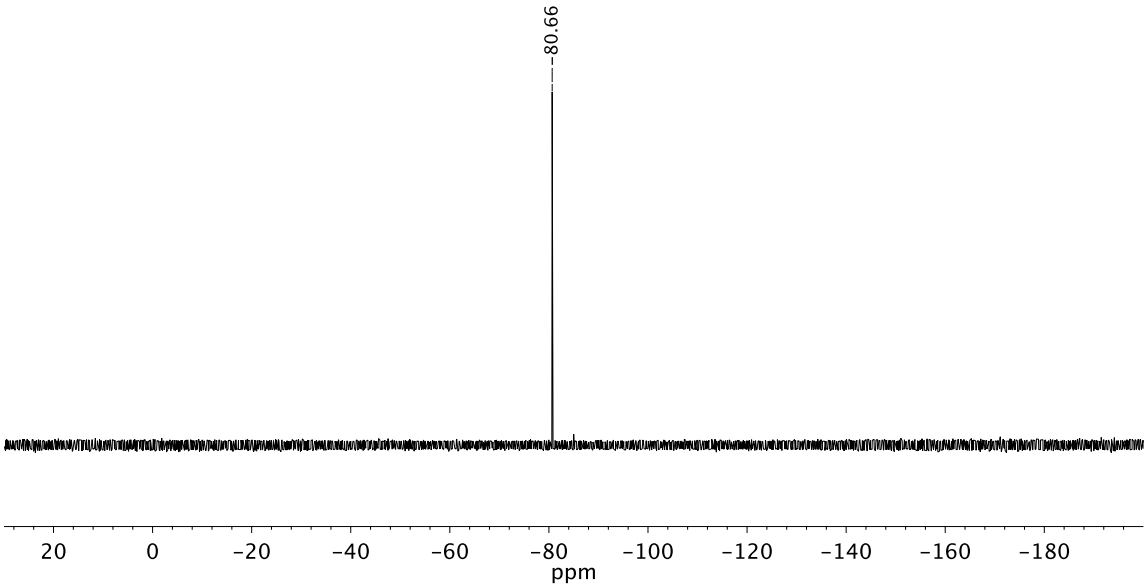
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5fa**



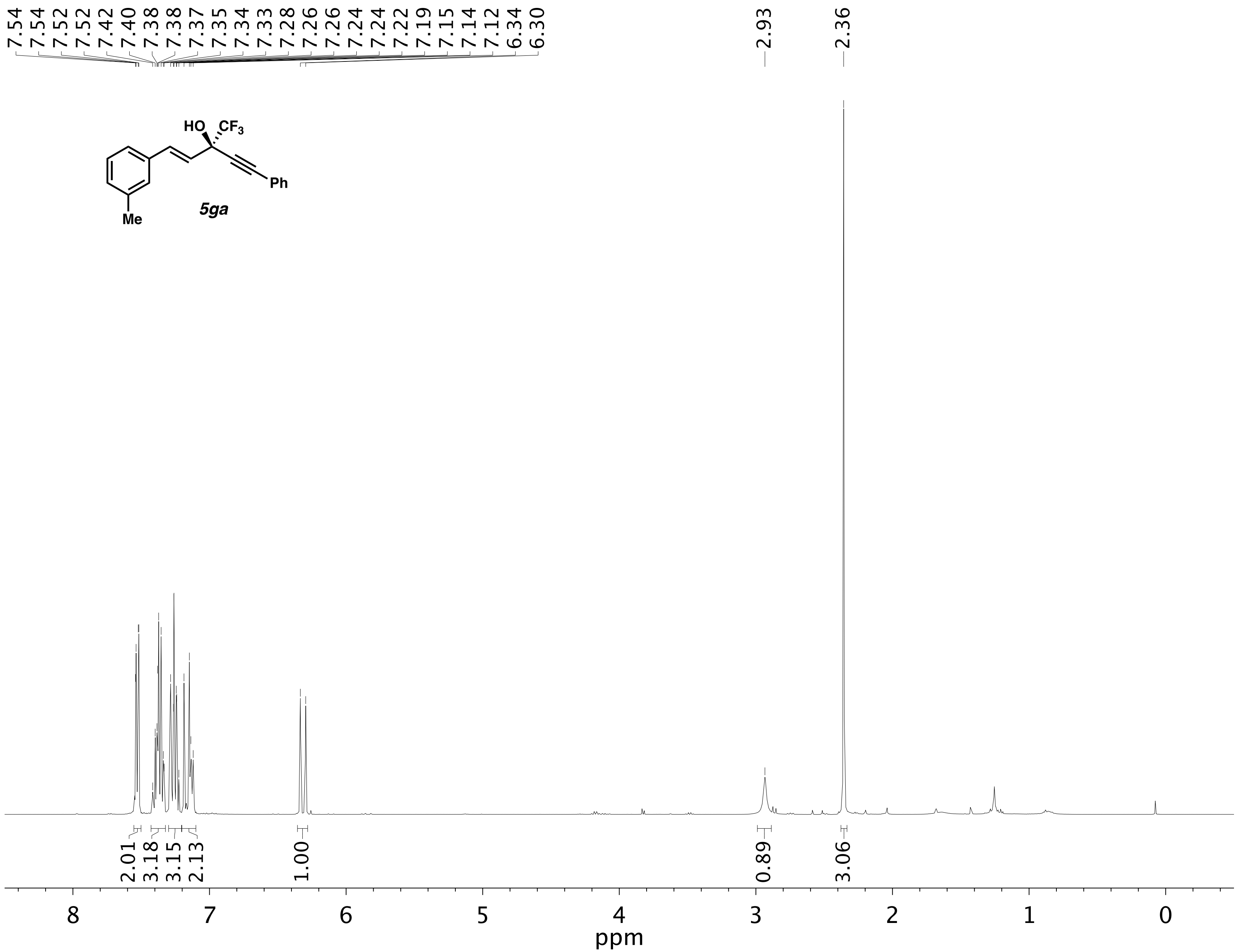
Infrared spectrum (Thin Film, NaCl) of compound **5fa**



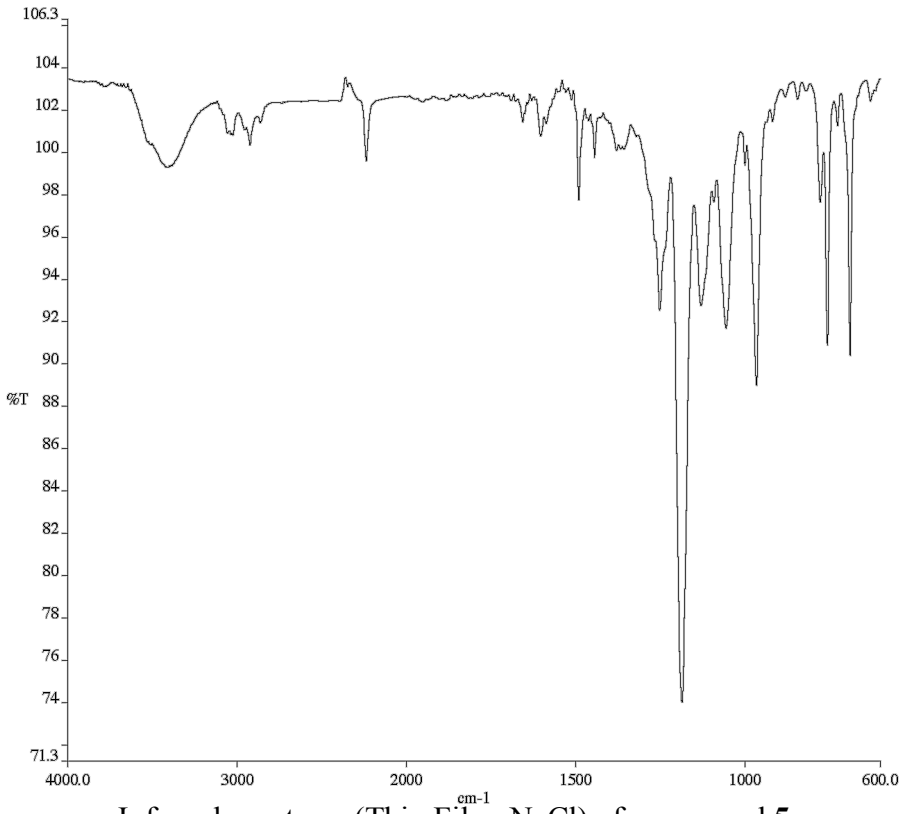
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5fa**



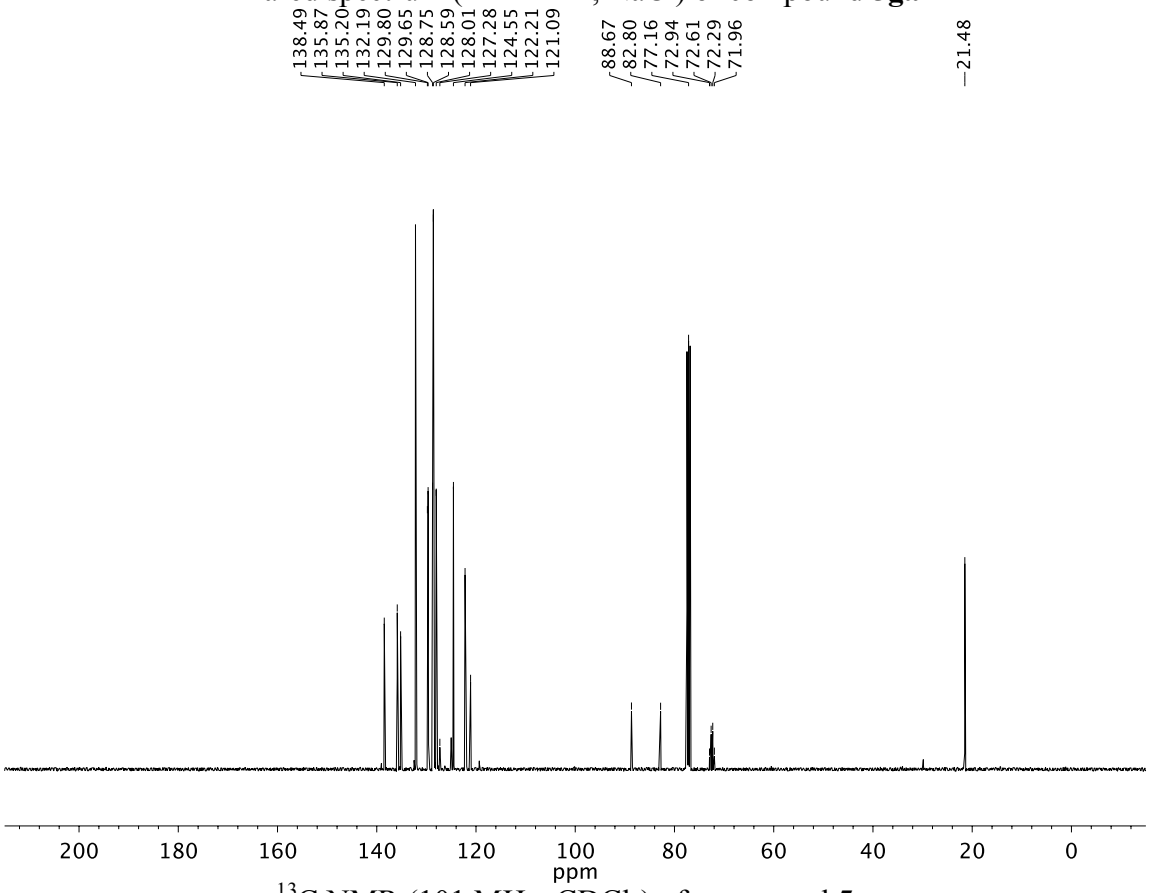
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5fa**



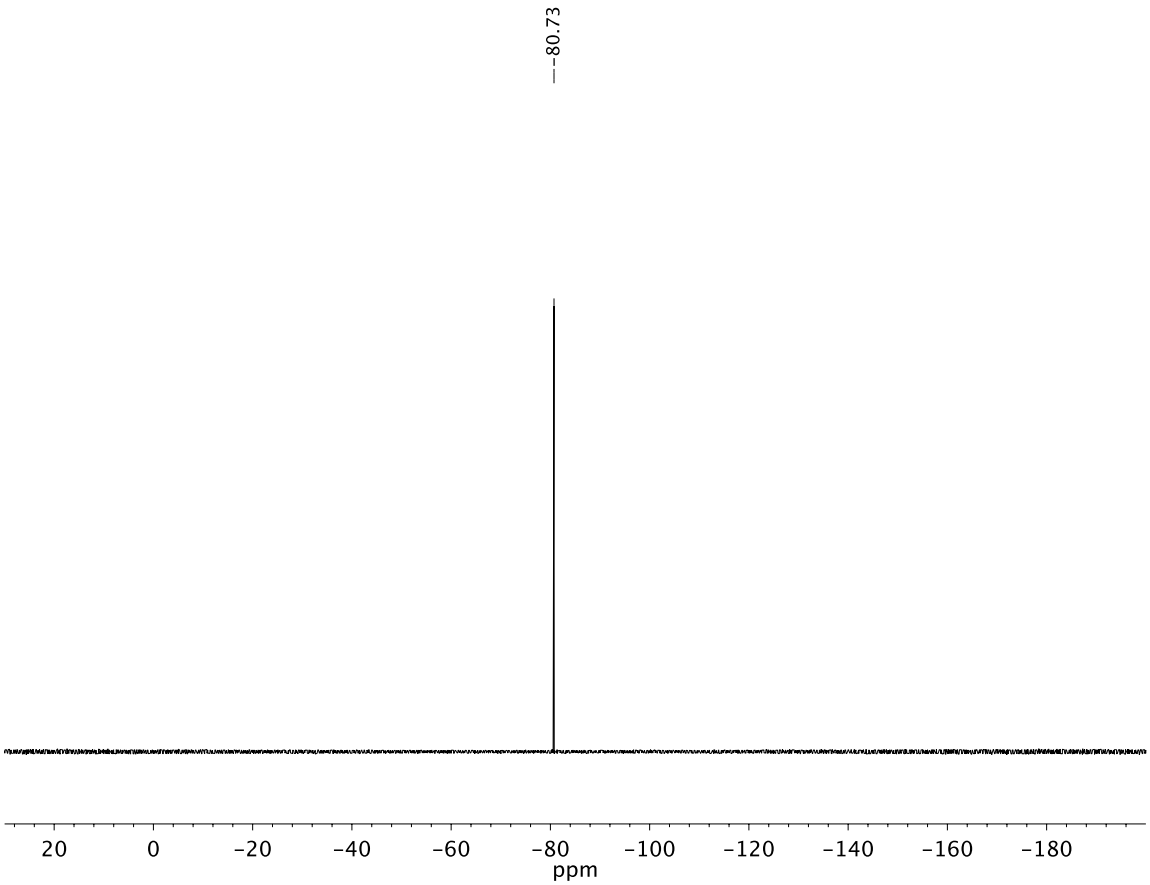
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ga**



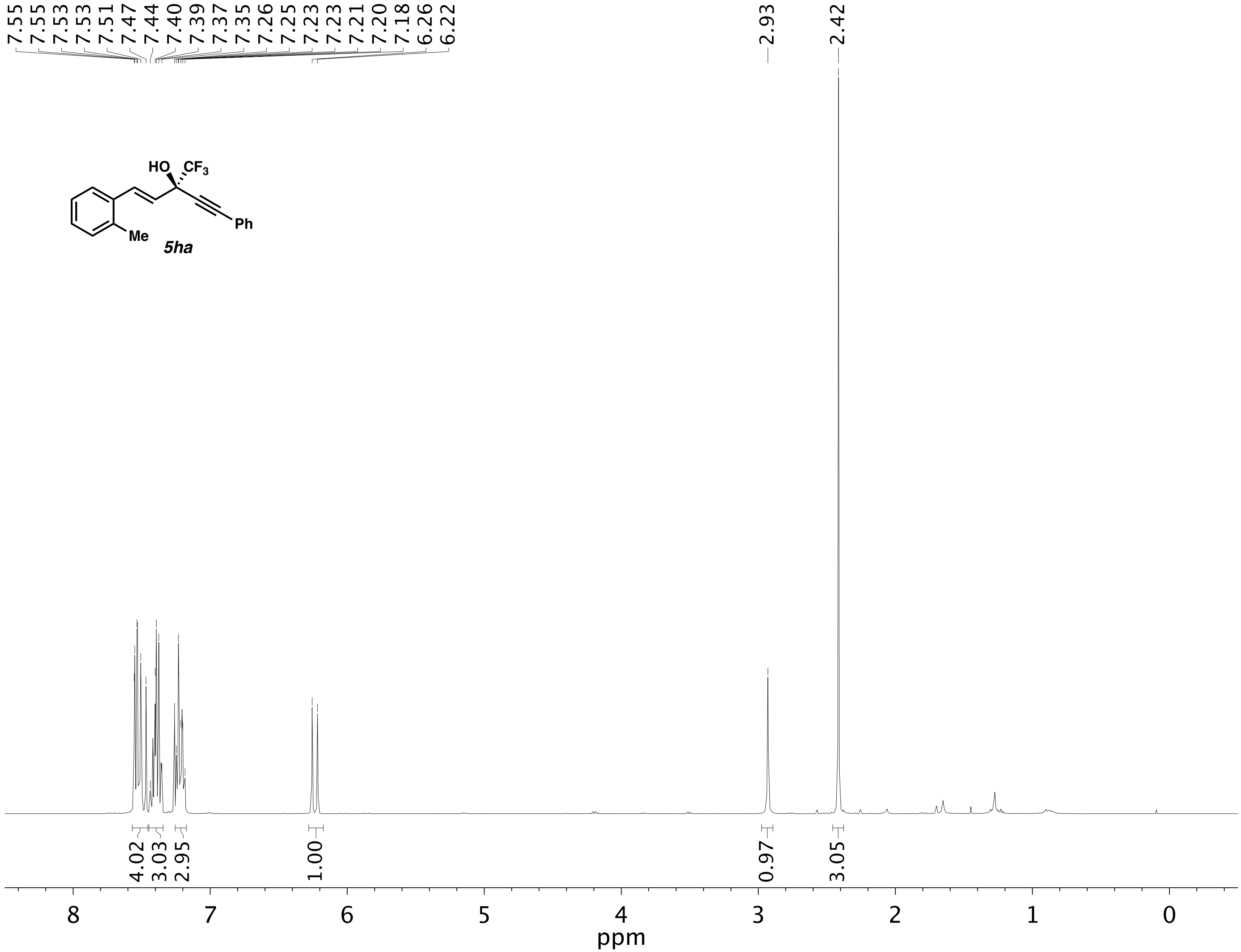
Infrared spectrum (Thin Film, NaCl) of compound **5ga**



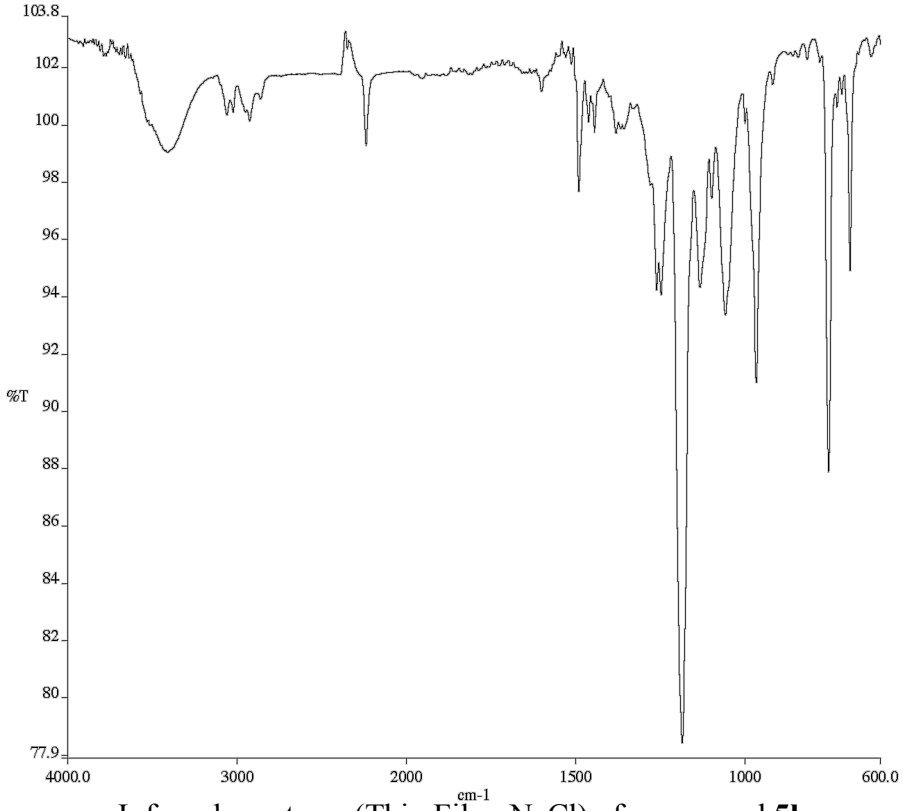
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ga**



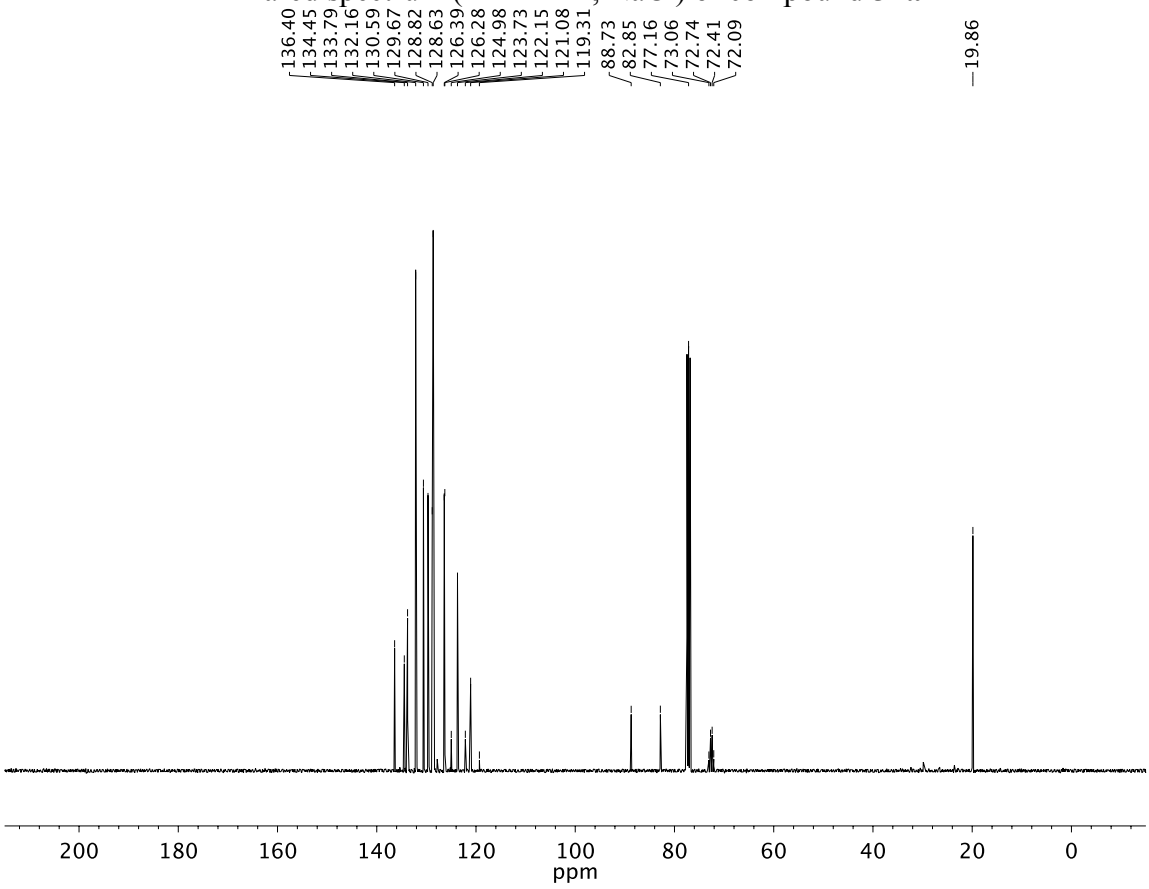
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ga**



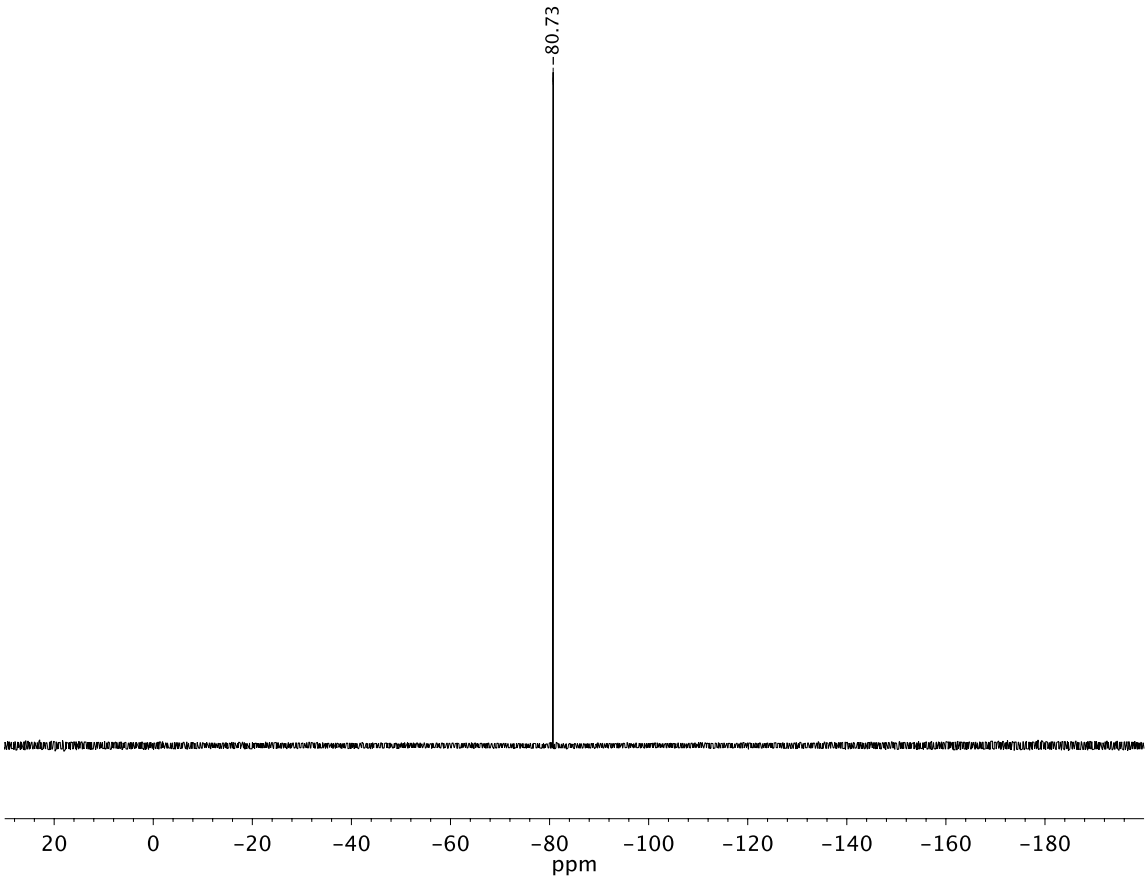
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ha**



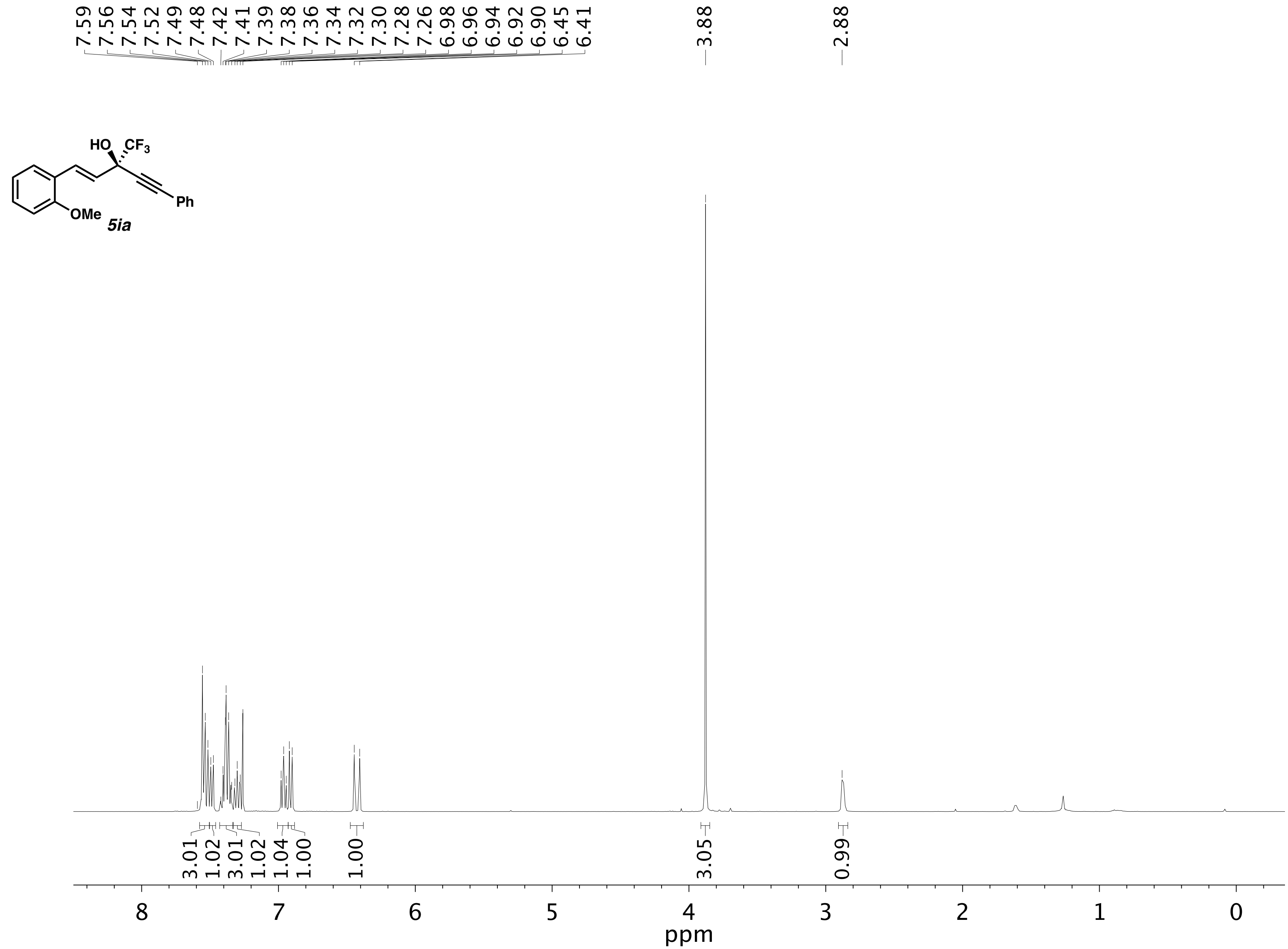
Infrared spectrum (Thin Film, NaCl) of compound **5ha**



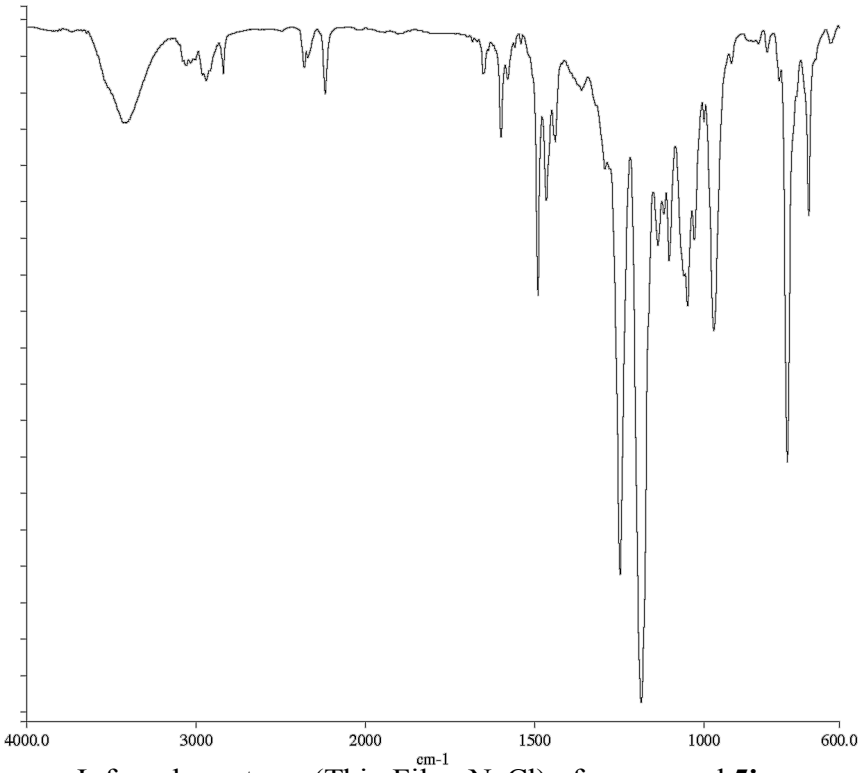
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ha**



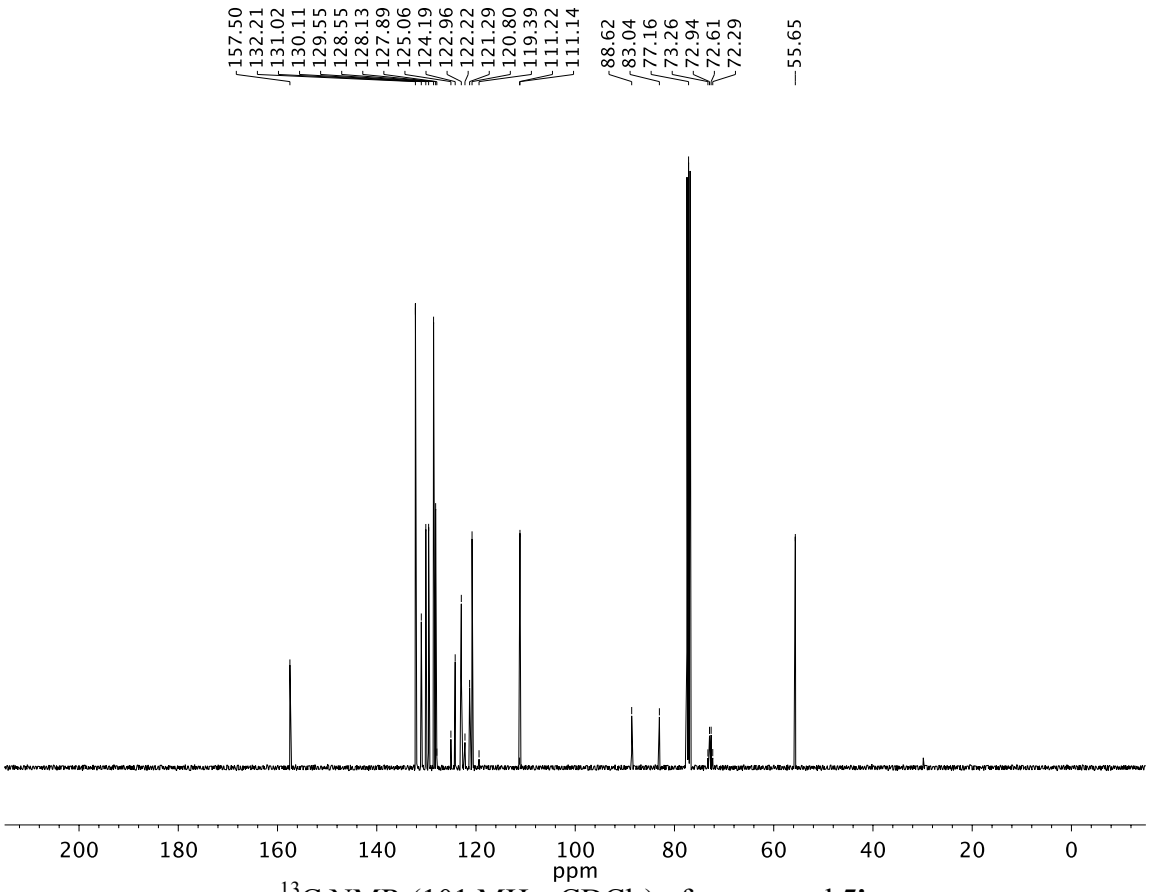
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ha**



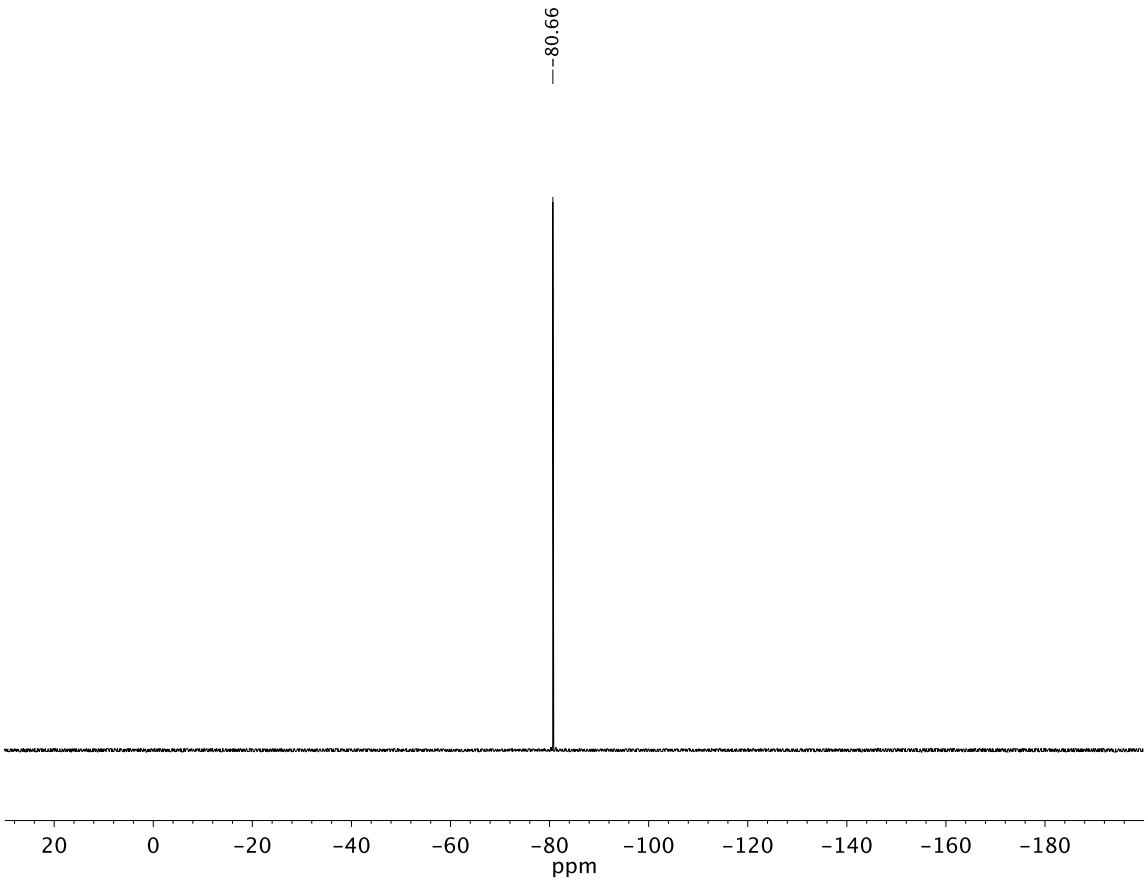
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ia**



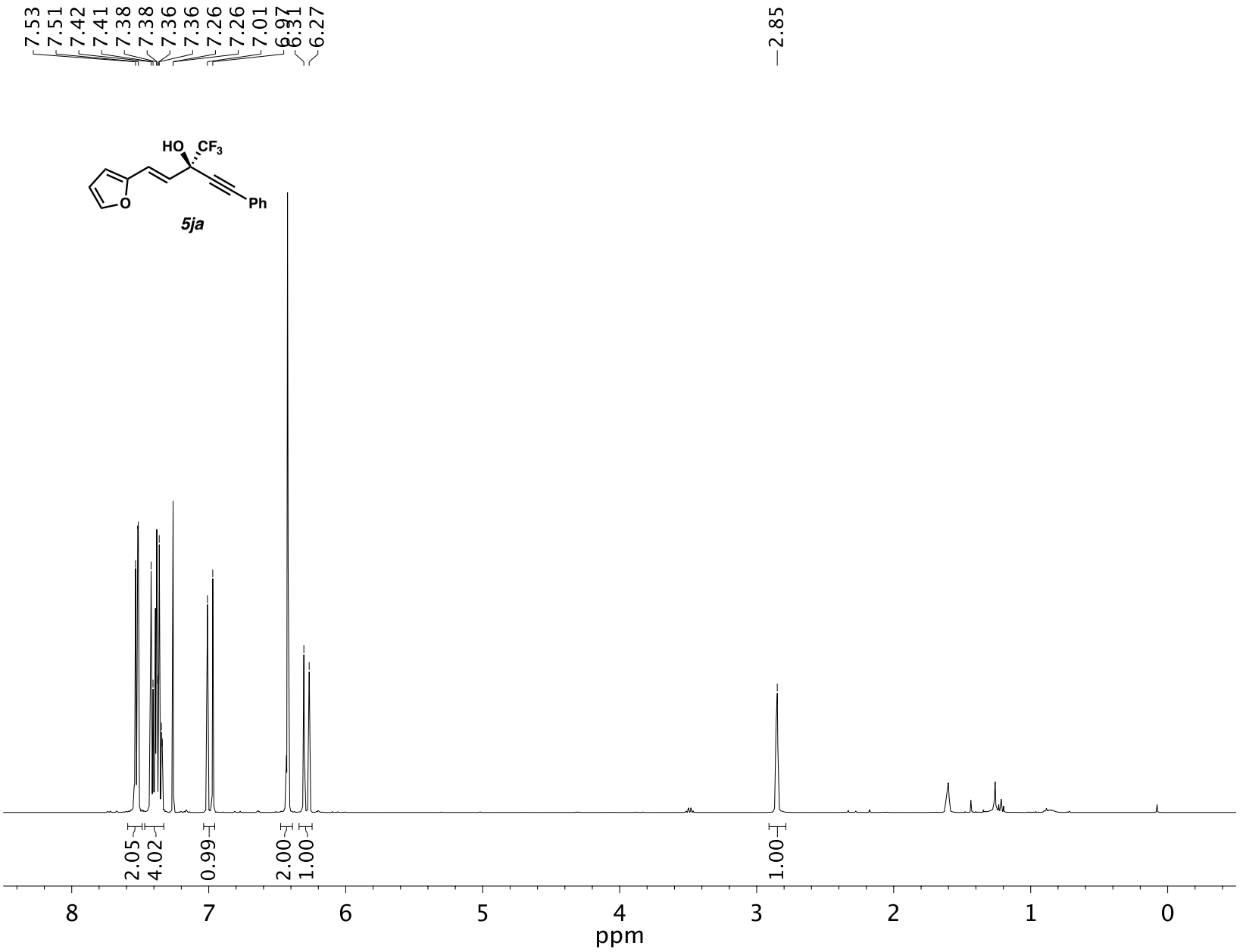
Infrared spectrum (Thin Film, NaCl) of compound **5ia**



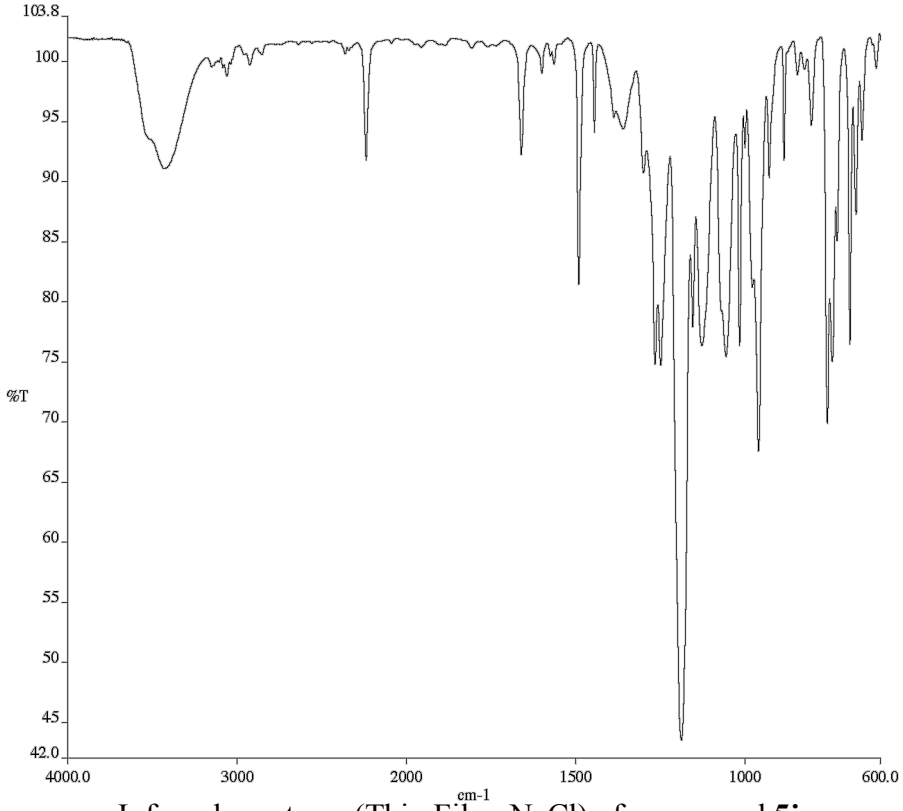
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ia**



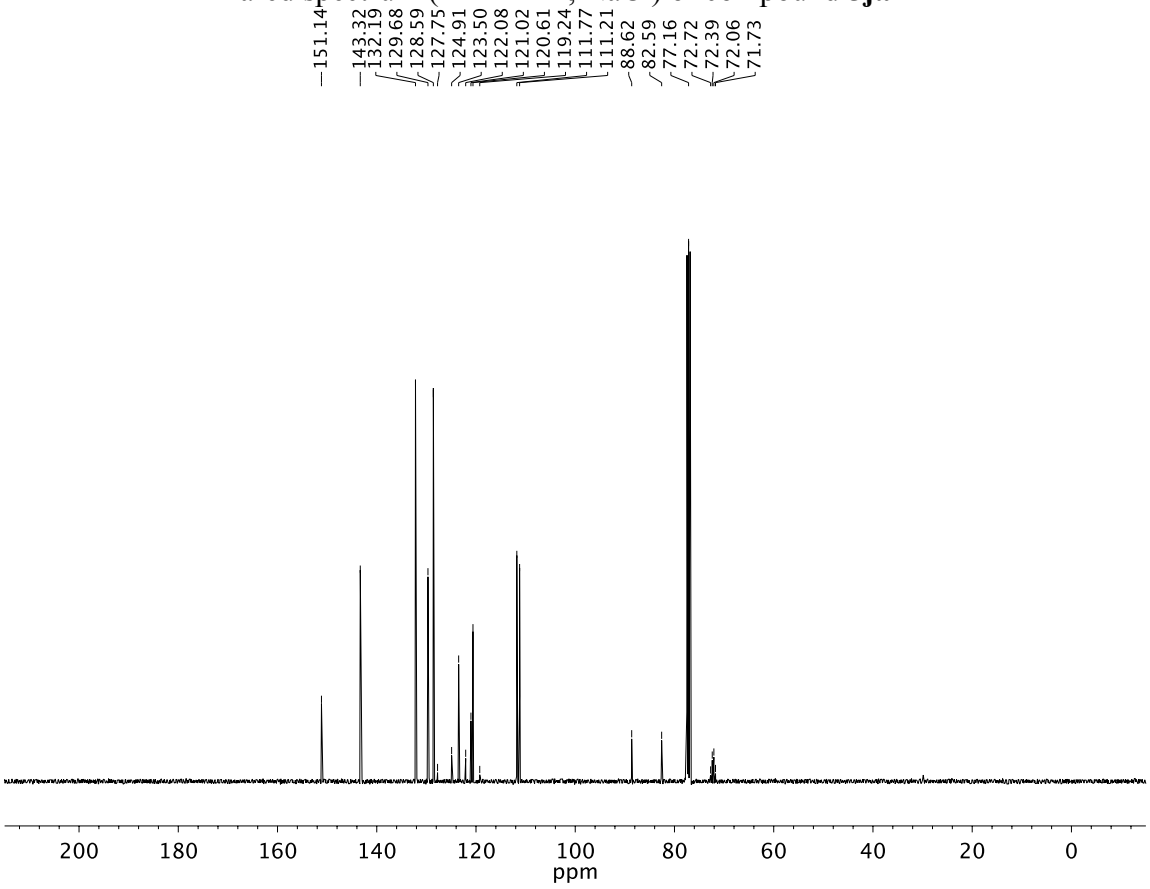
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ia**



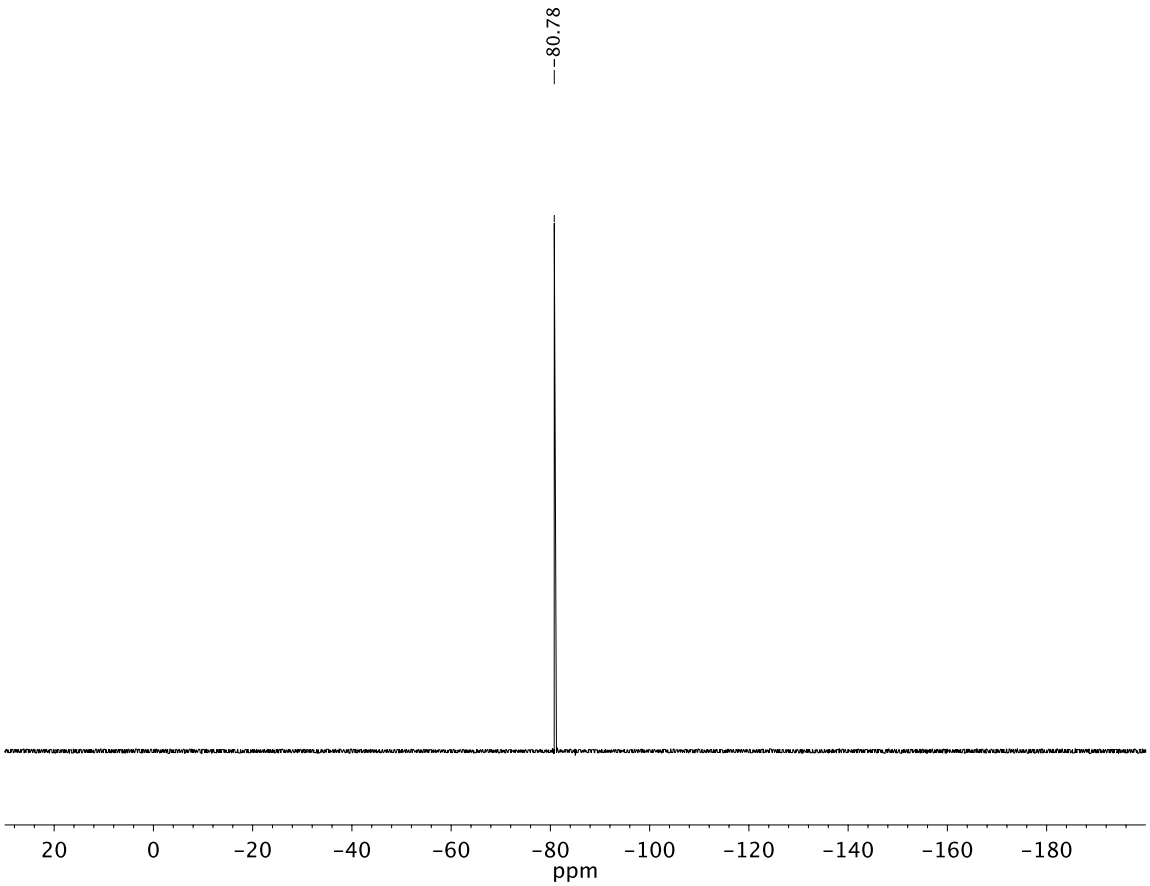
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ja**



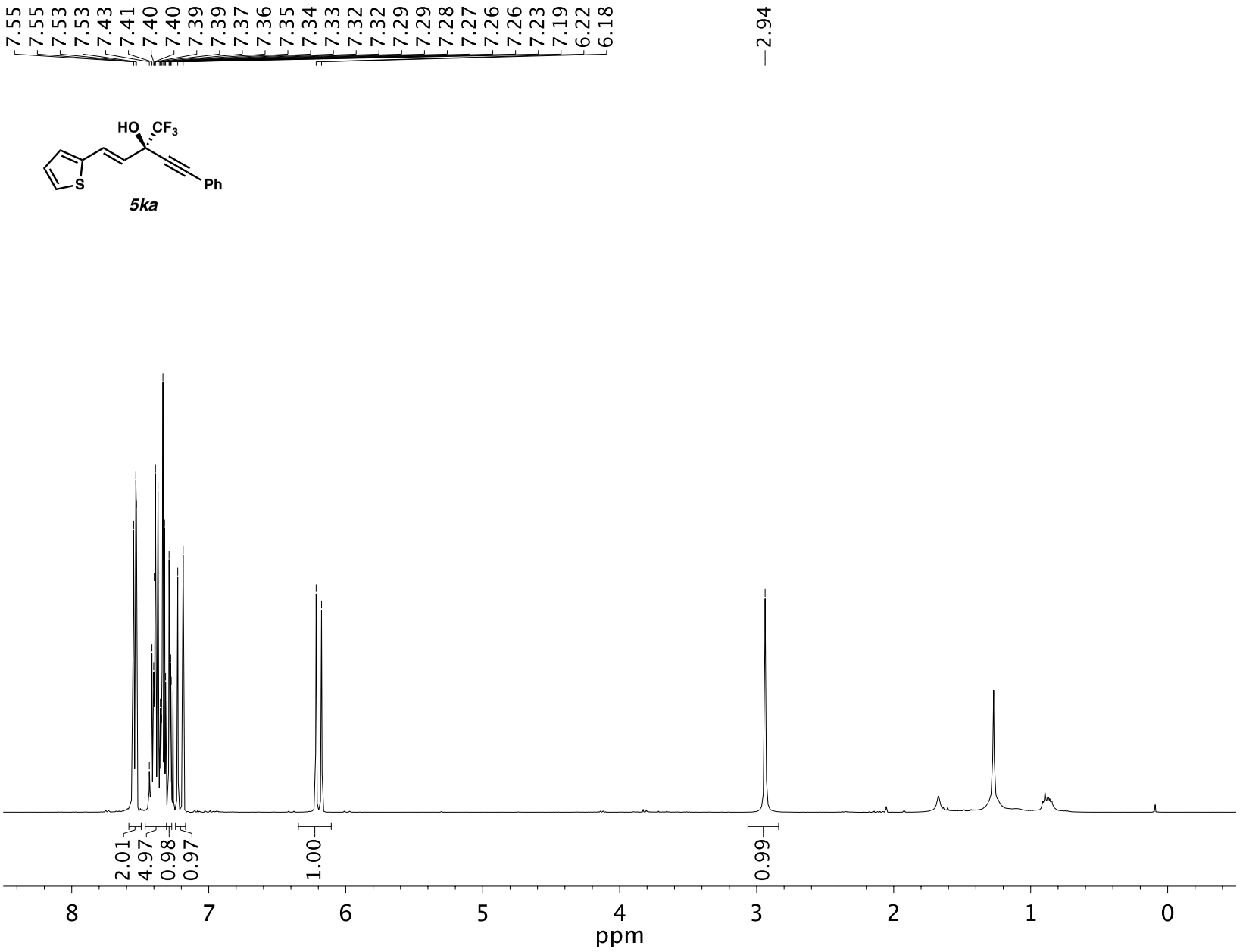
Infrared spectrum (Thin Film, NaCl) of compound **5ja**



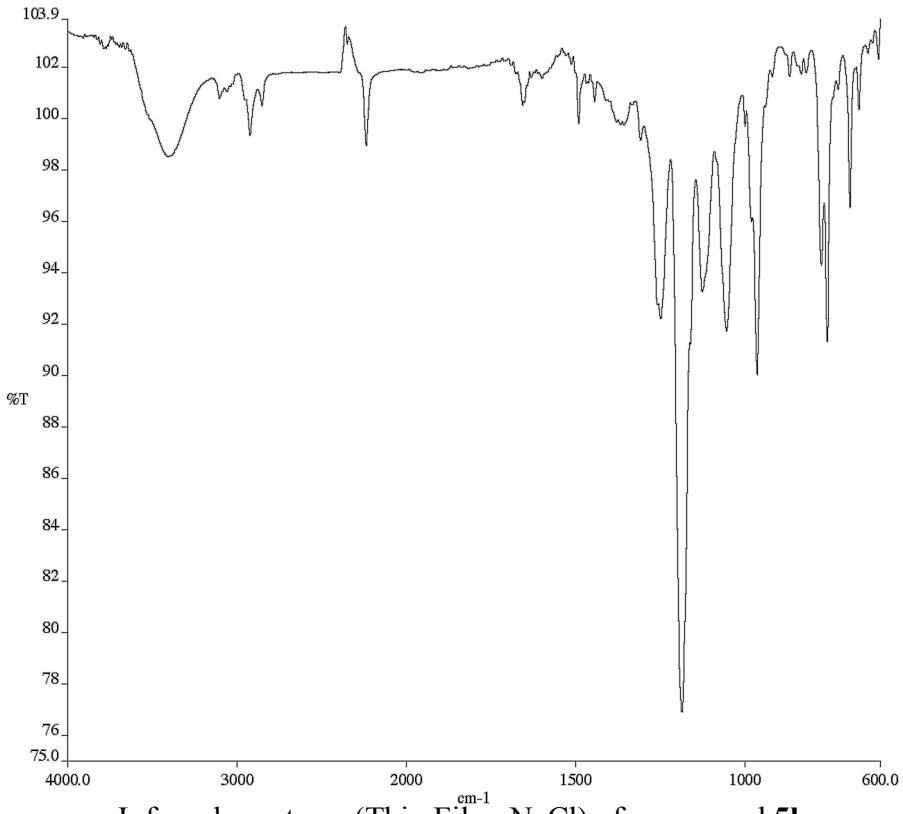
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ja**



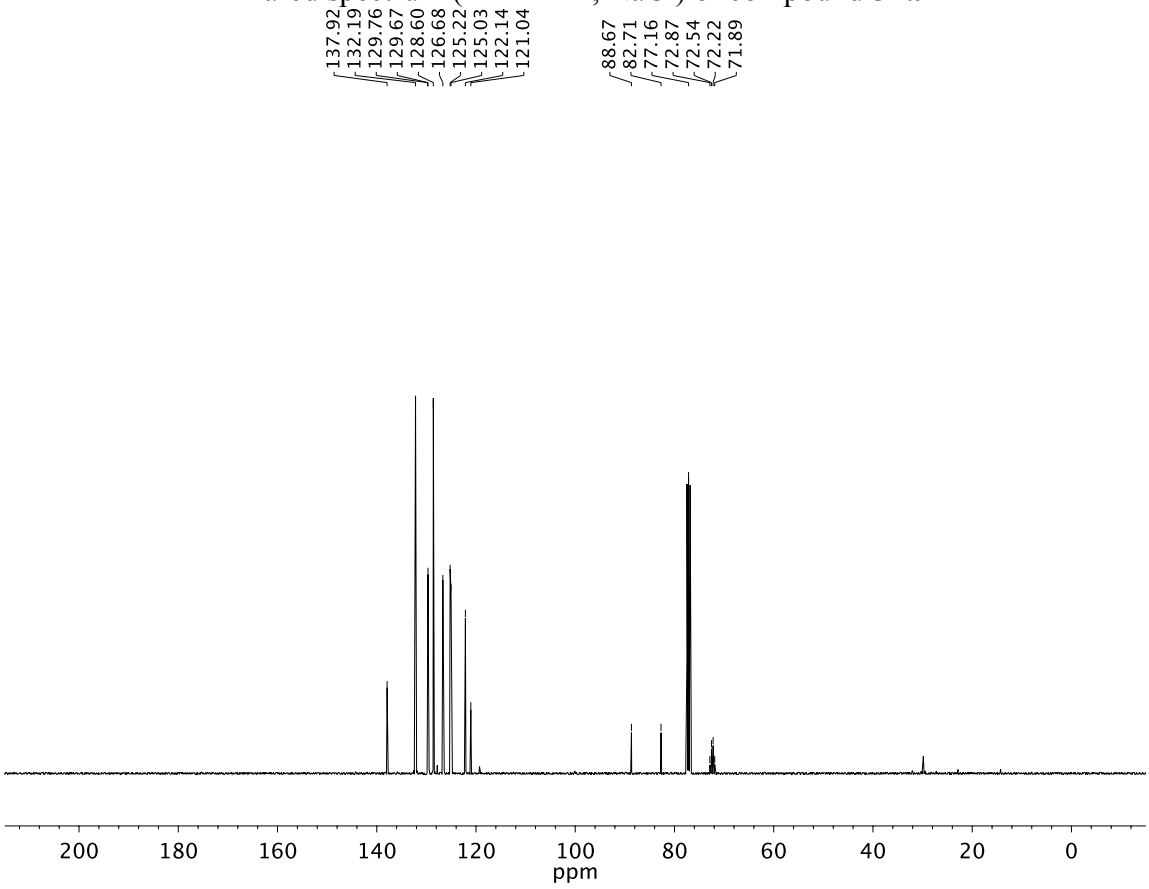
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ja**



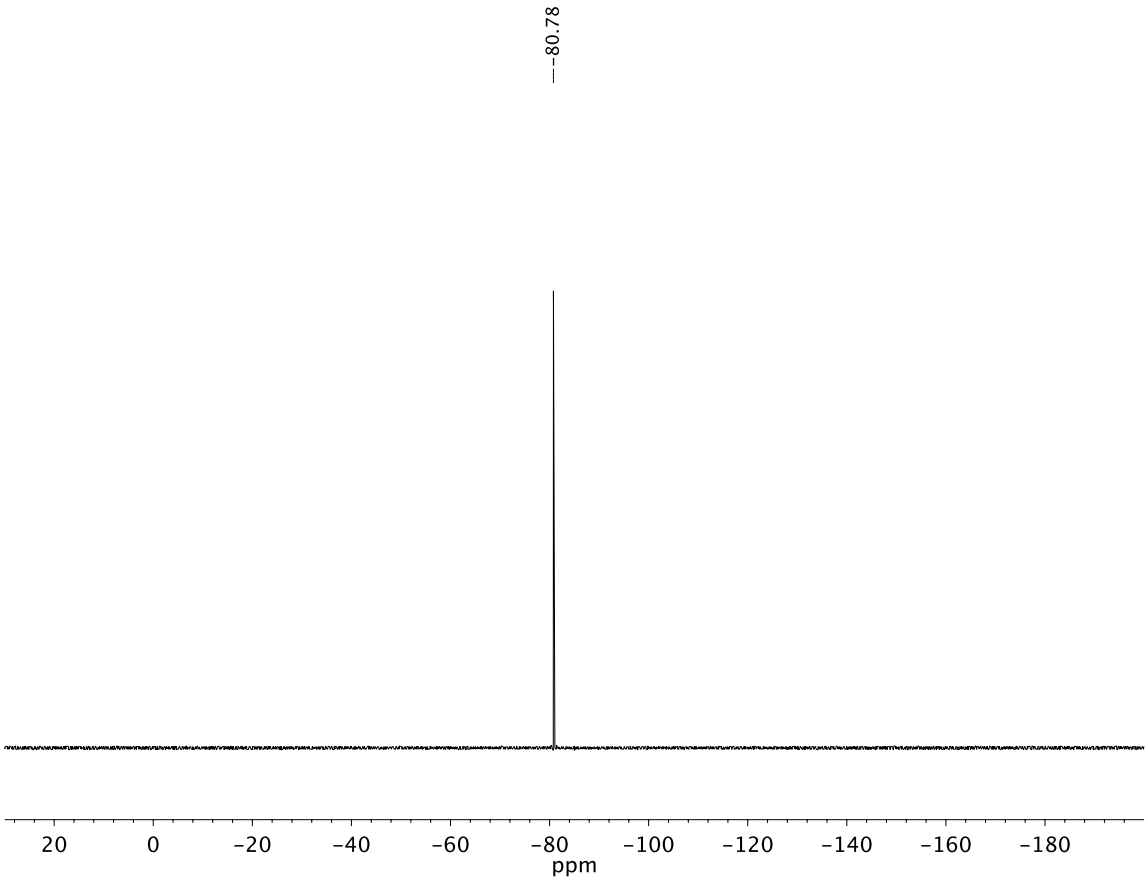
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ka**



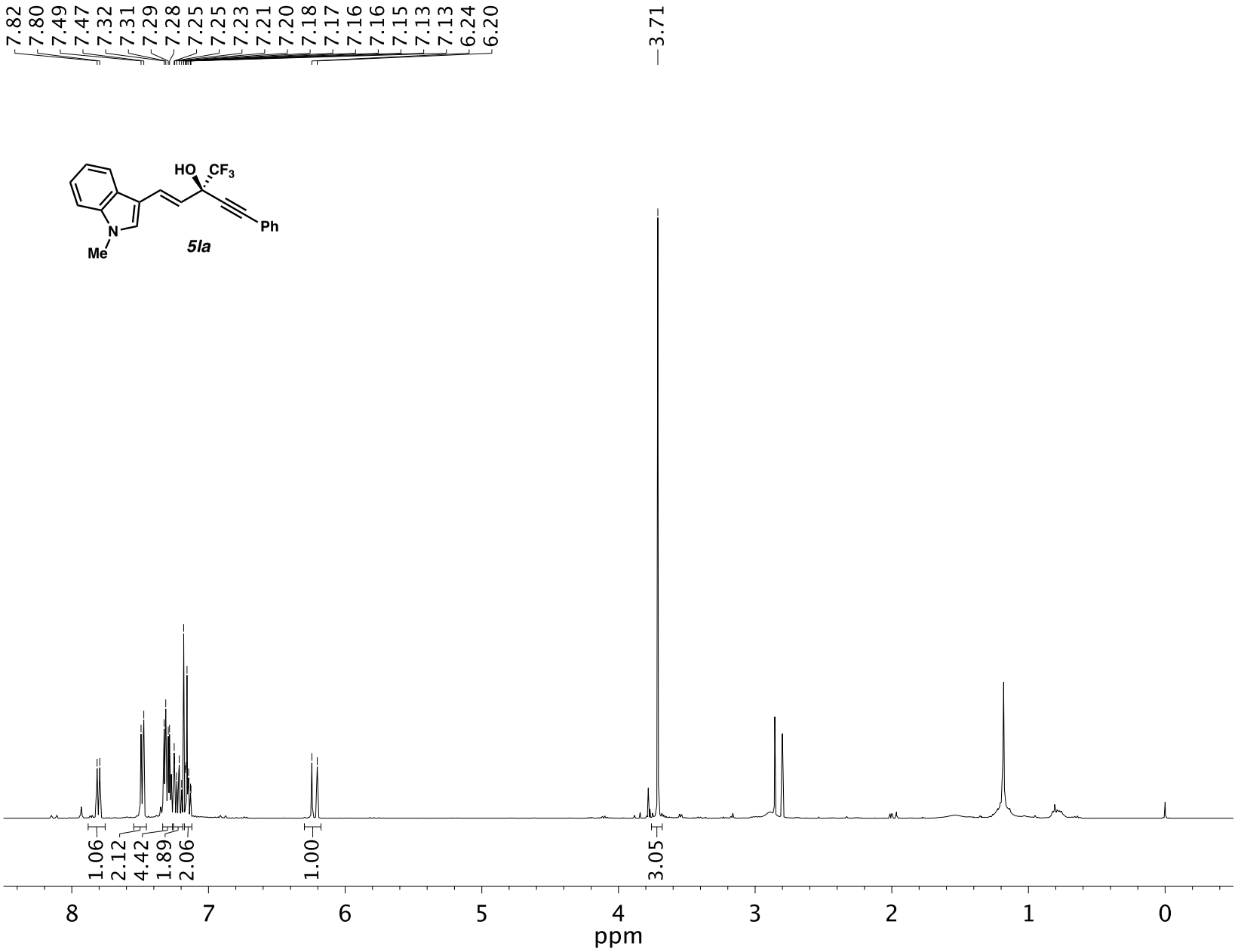
Infrared spectrum (Thin Film, NaCl) of compound **5ka**



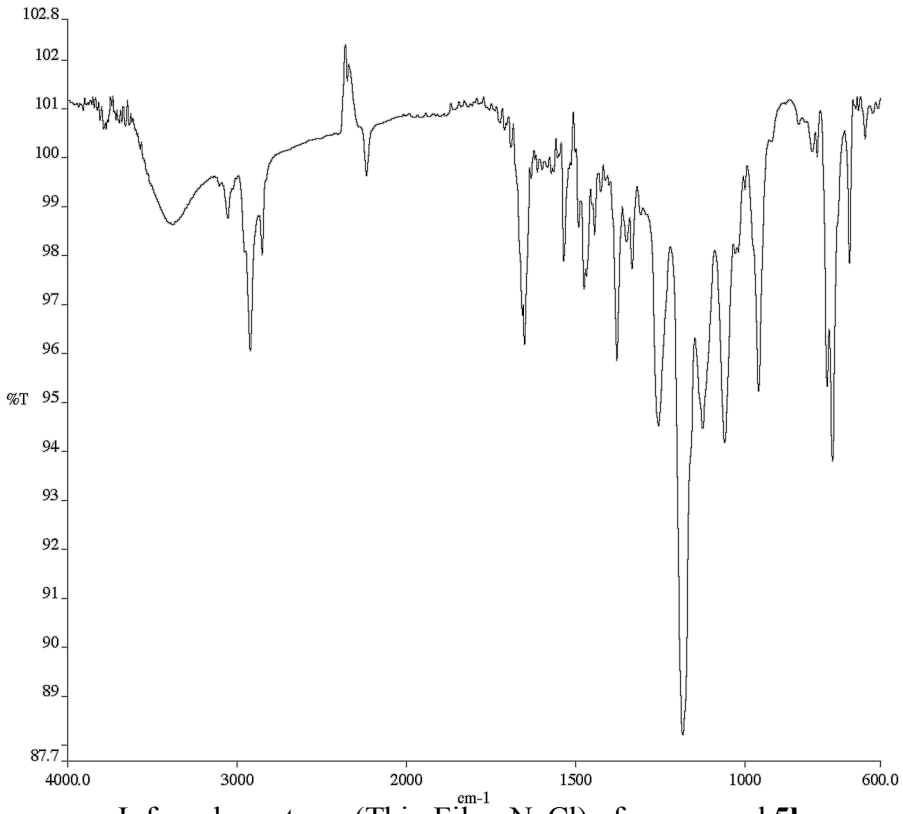
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ka**



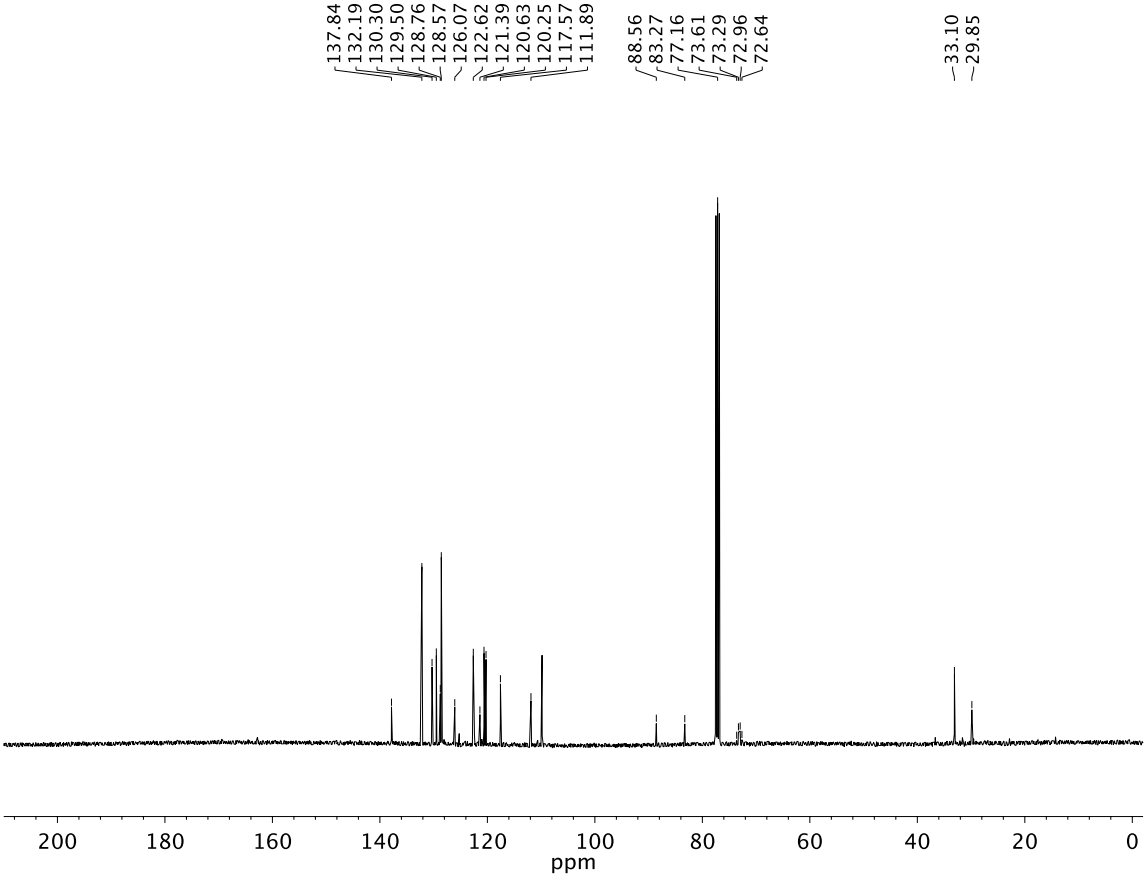
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ka**



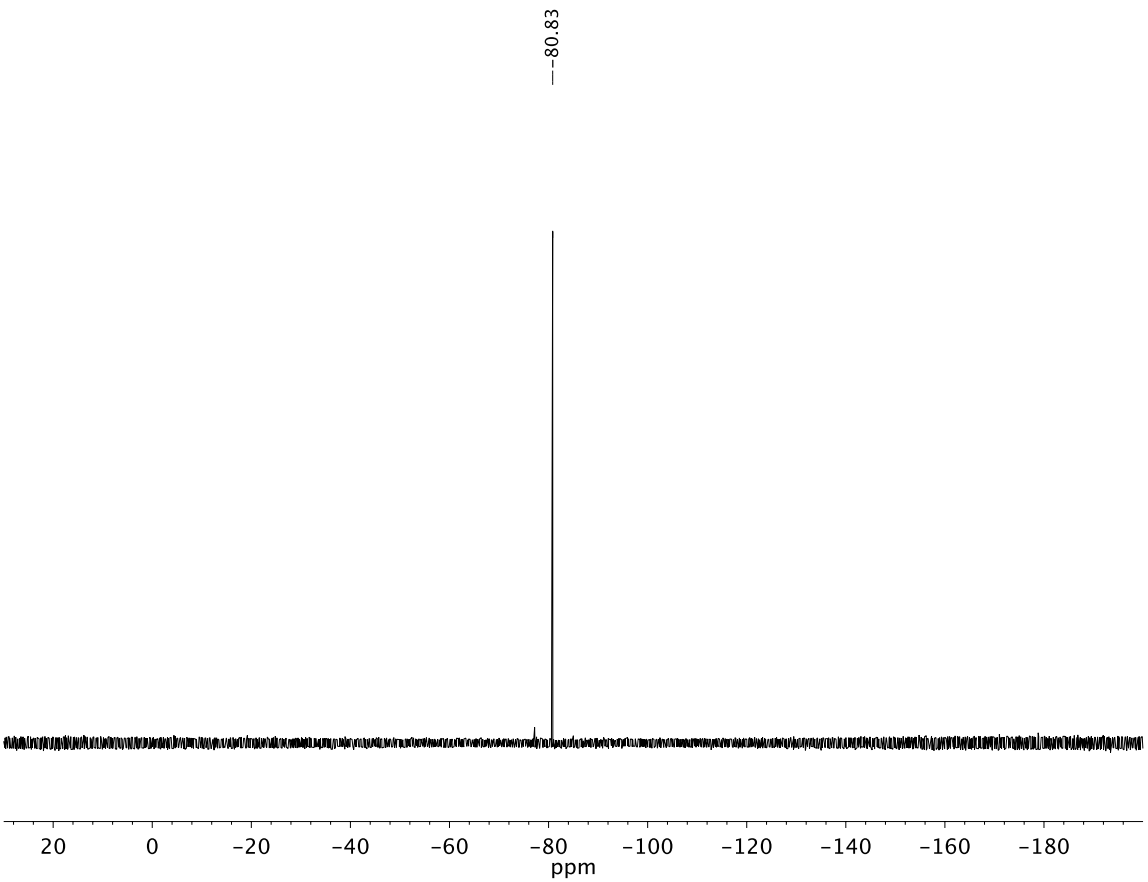
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5la**



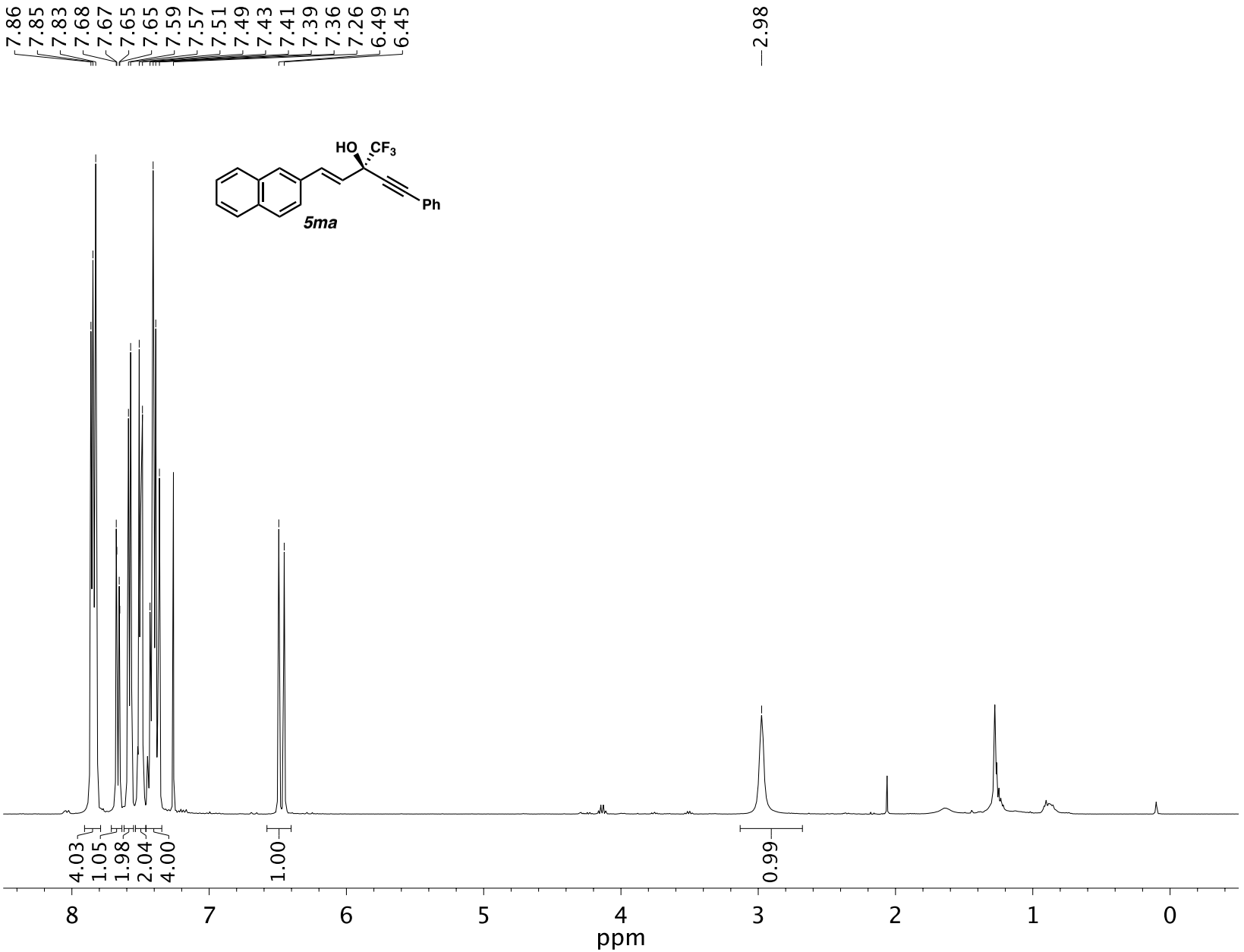
Infrared spectrum (Thin Film, NaCl) of compound **5la**



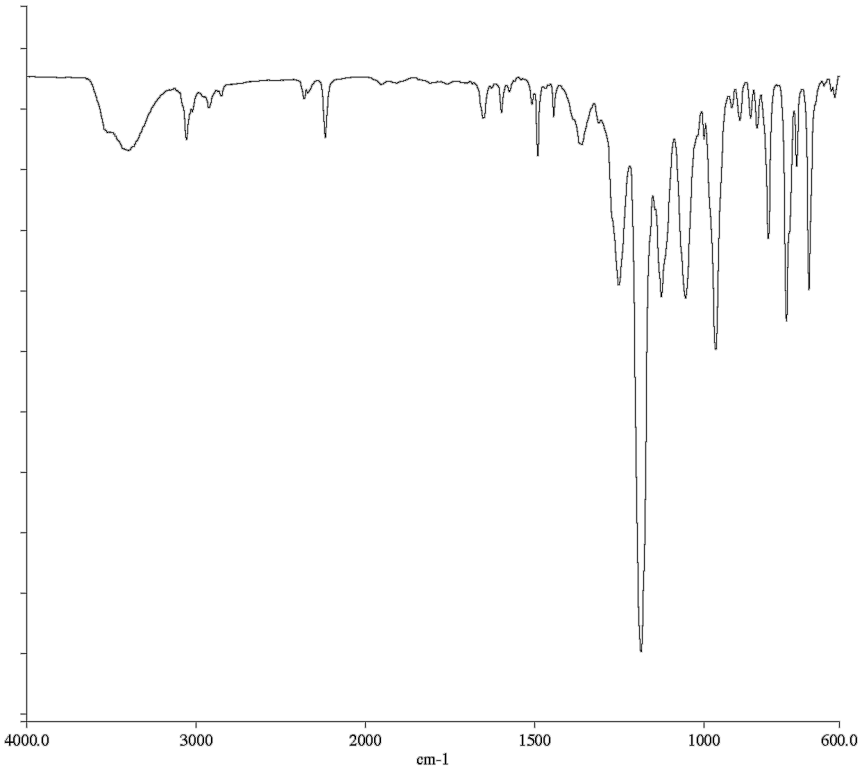
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5la**



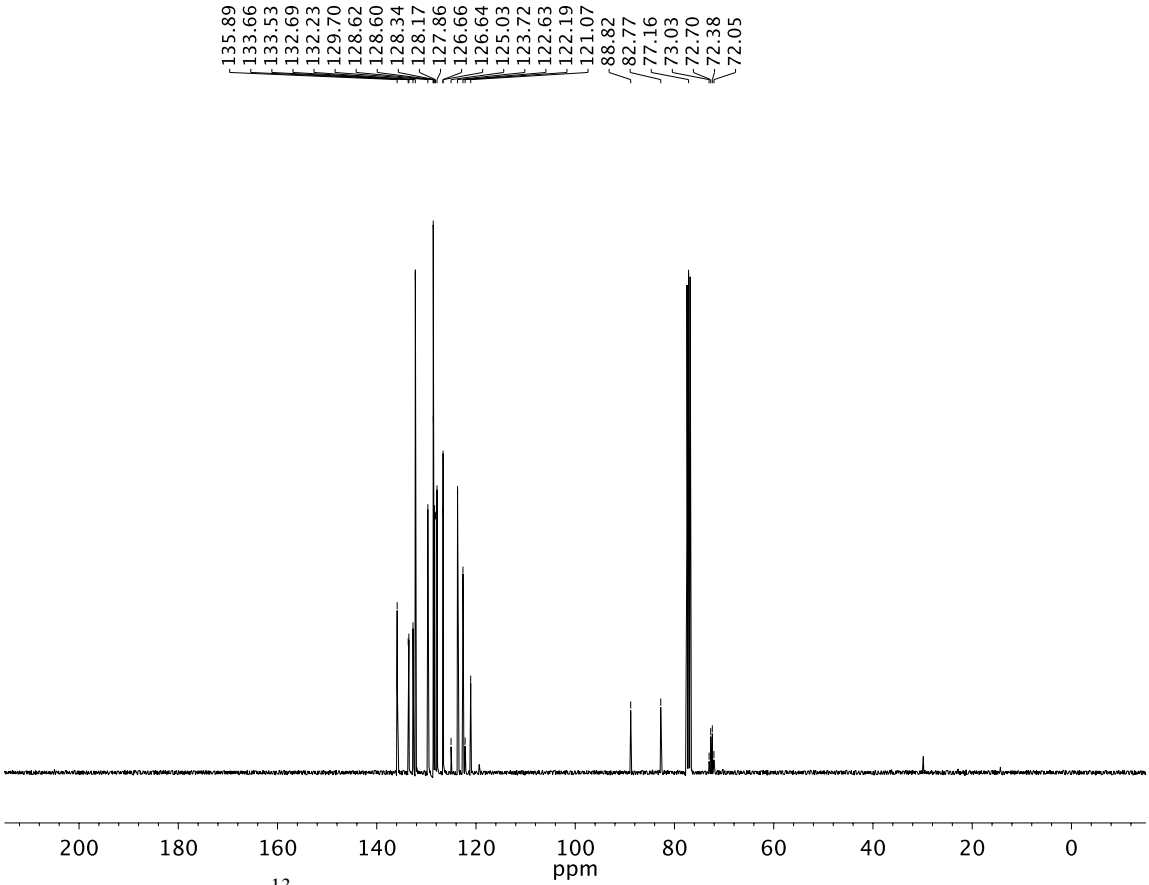
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5la**



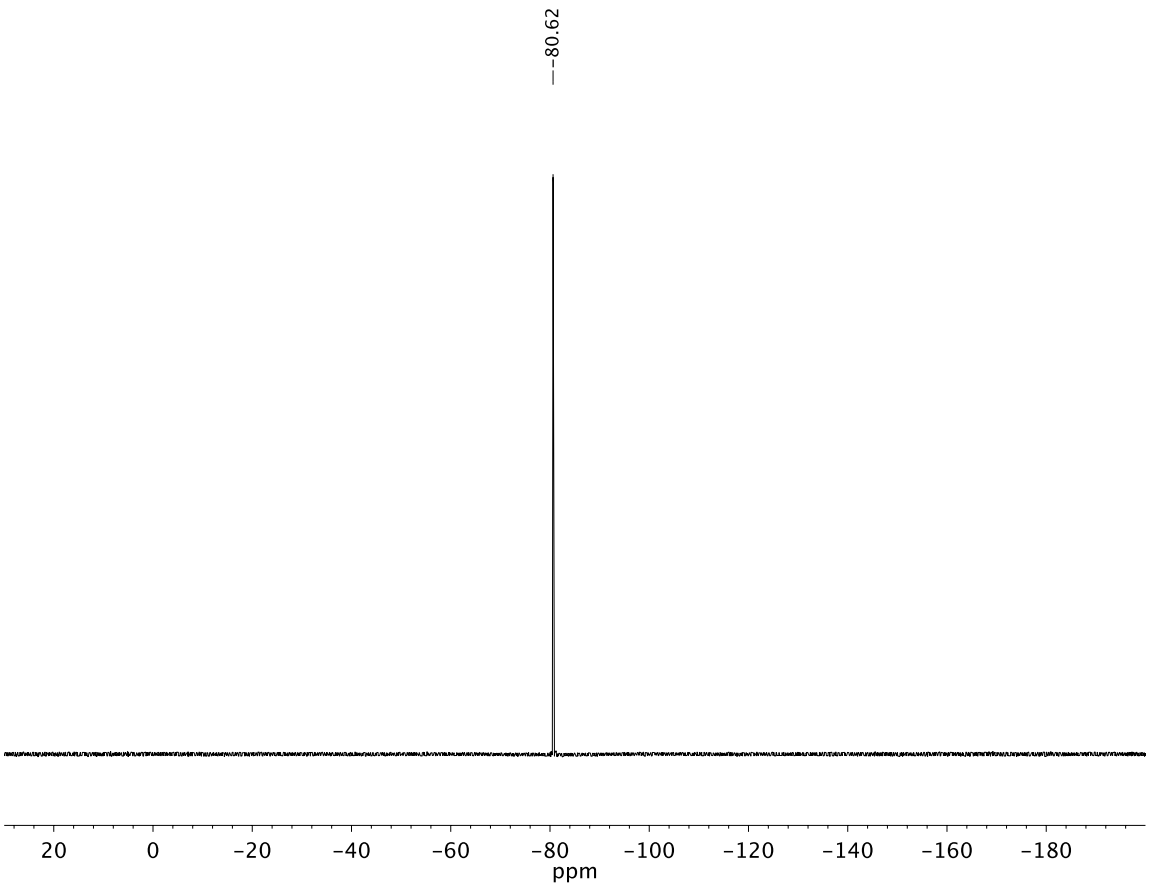
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5ma**



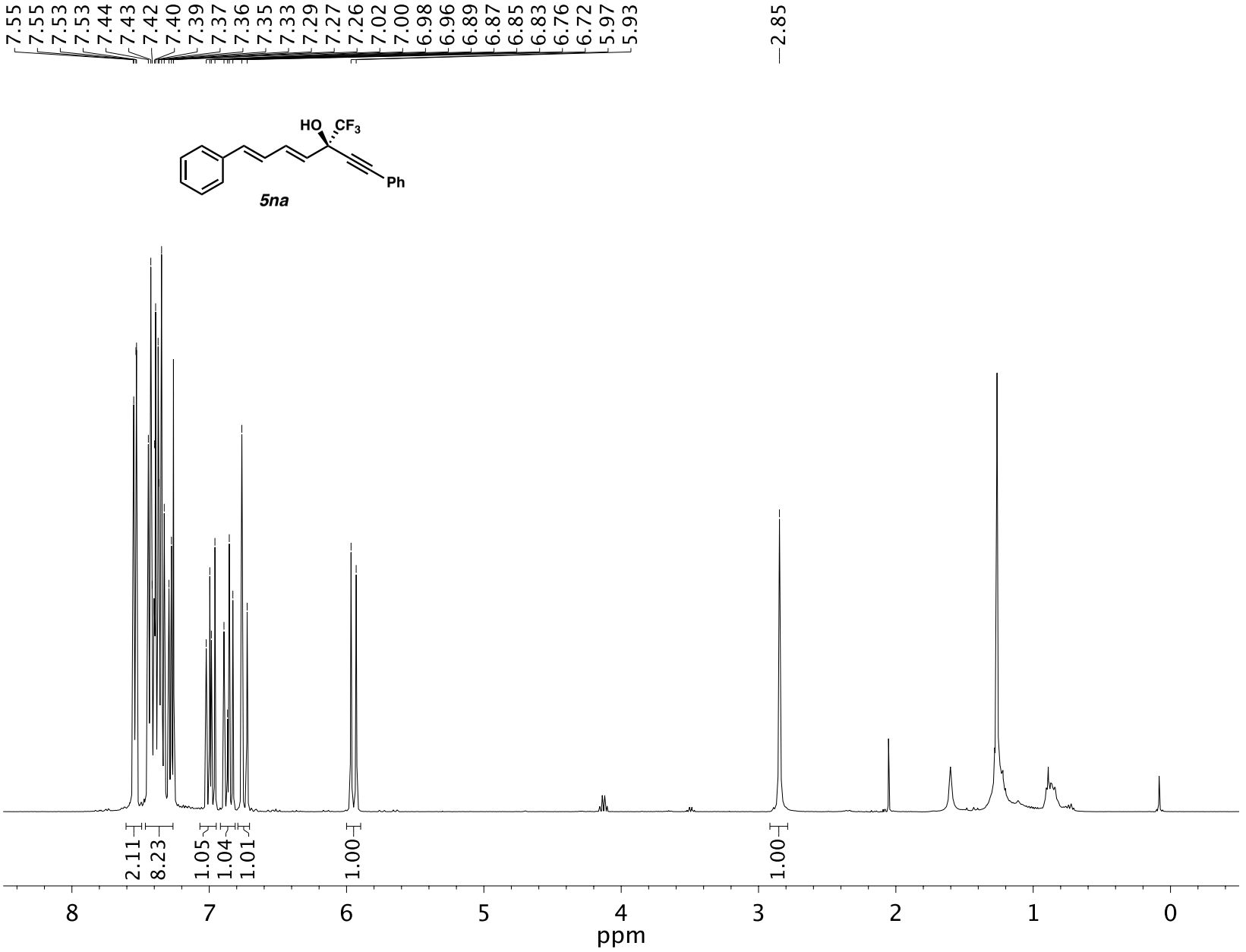
Infrared spectrum (Thin Film, NaCl) of compound **5ma**.



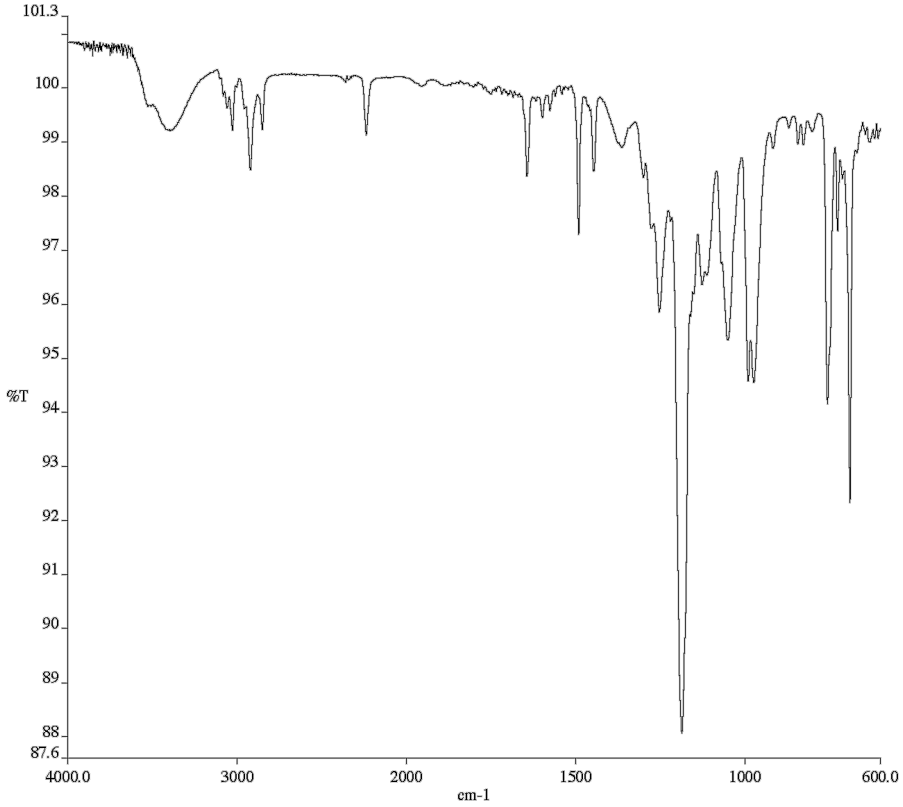
^{13}C NMR (101 MHz, $CDCl_3$) of compound **5ma**



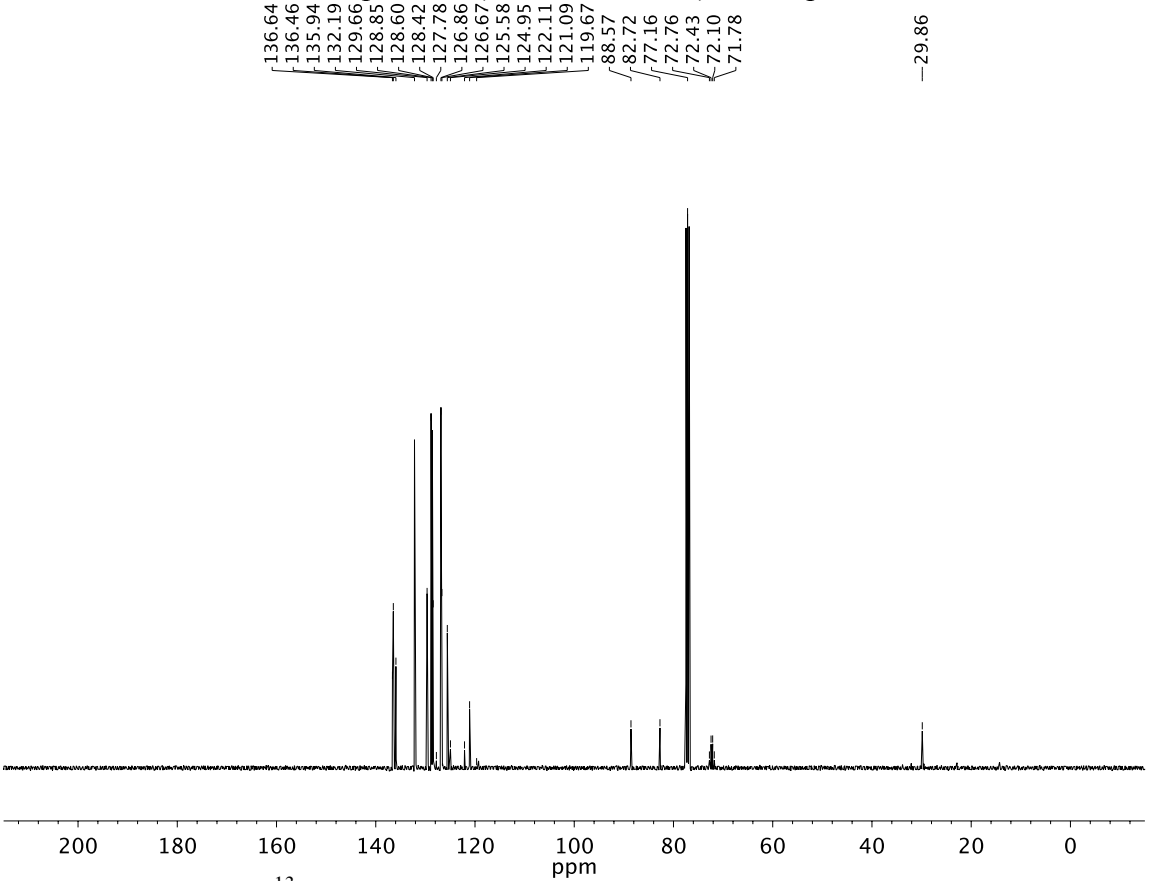
^{19}F NMR (282 MHz, $CDCl_3$) of compound **5ma**



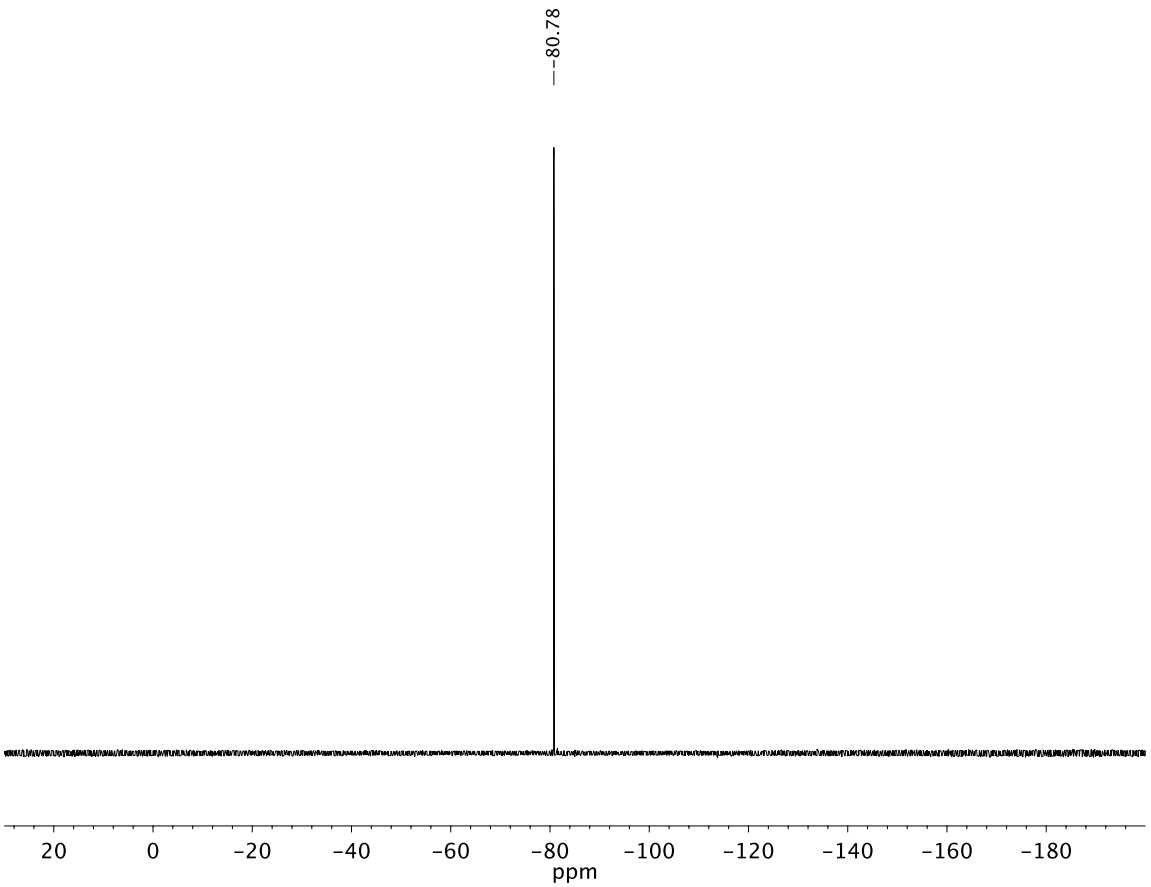
^{1}H NMR (400 MHz, $CDCl_3$) of compound **5na**



Infrared spectrum (Thin Film, NaCl) of compound **5na**

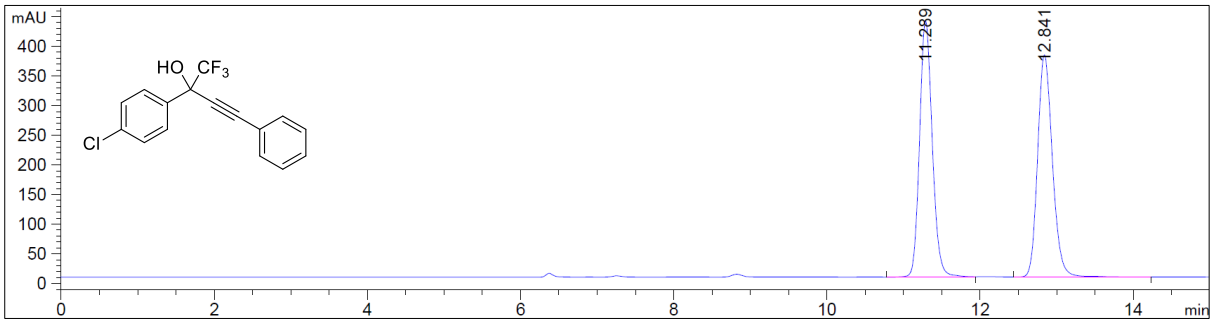


^{13}C NMR (101 MHz, $CDCl_3$) of compound **5na**



^{19}F NMR (282 MHz, $CDCl_3$) of compound **5na**

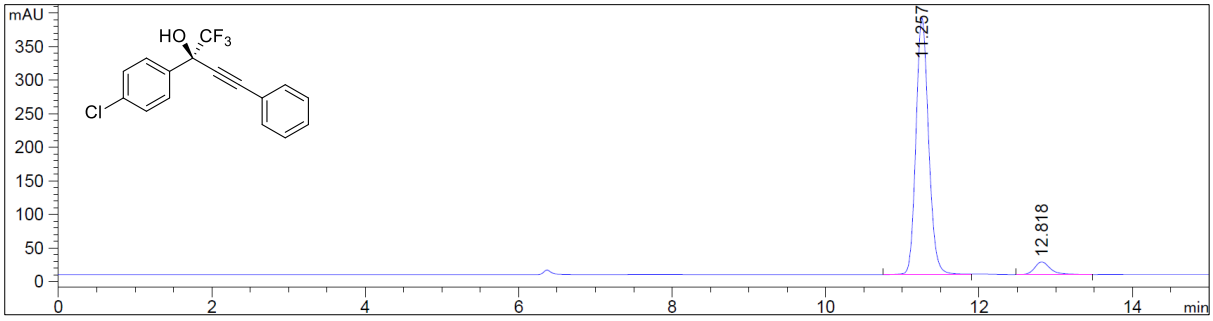
Racemic 3ca



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.289	BV	0.1786	4981.56006	431.86307	49.7393
2	12.841	VB	0.2088	5033.77783	374.57556	50.2607
Totals :				1.00153e4	806.43863	

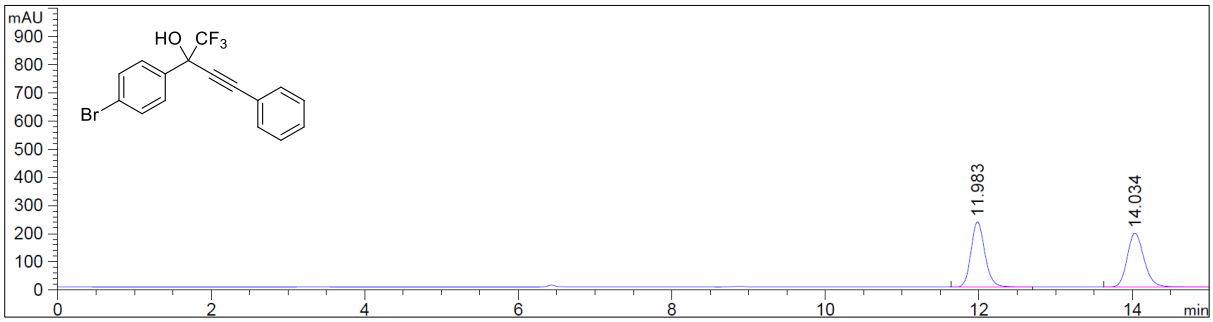
Enantioenriched 3ca



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.257	BV	0.1776	4376.18311	382.06796	94.2957
2	12.818	BV	0.2167	264.73257	18.74982	5.7043
Totals :				4640.91568	400.81779	

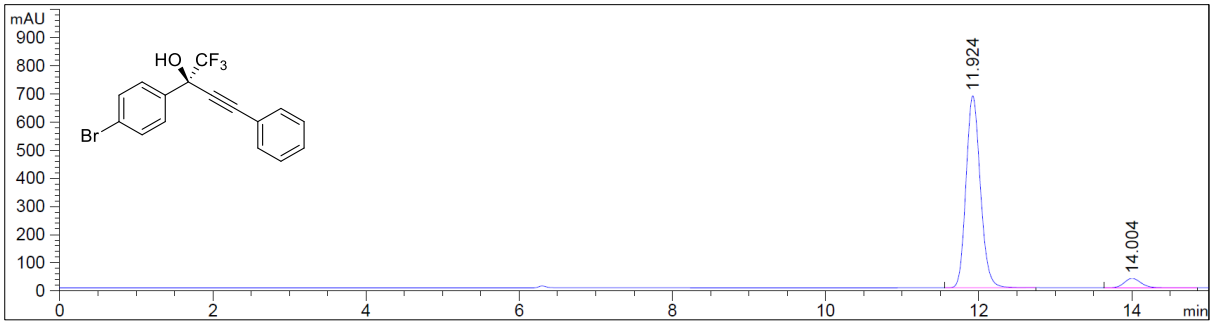
Racemic 3da



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.983	BB	0.1939	2885.78491	230.71150	49.9867
2	14.034	VB	0.2364	2887.32275	190.96884	50.0133
Totals :				5773.10767	421.68034	

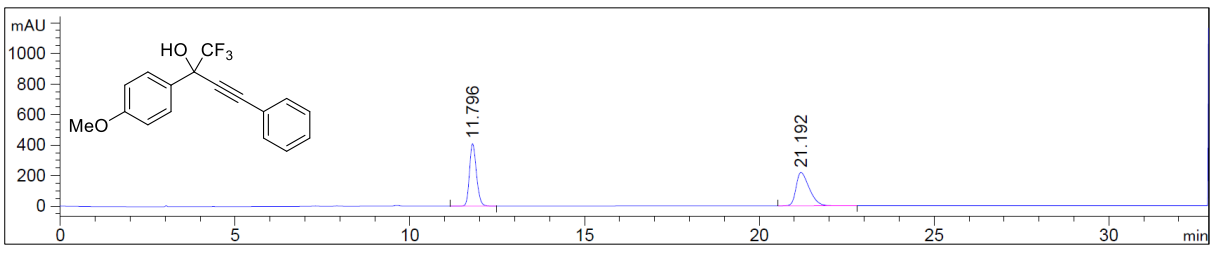
Enantioenriched 3da



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.924	BB	0.2017	8857.81738	681.41754	94.5631
2	14.004	BB	0.2339	509.27783	33.40222	5.4369
Totals :				9367.09521	714.81976	

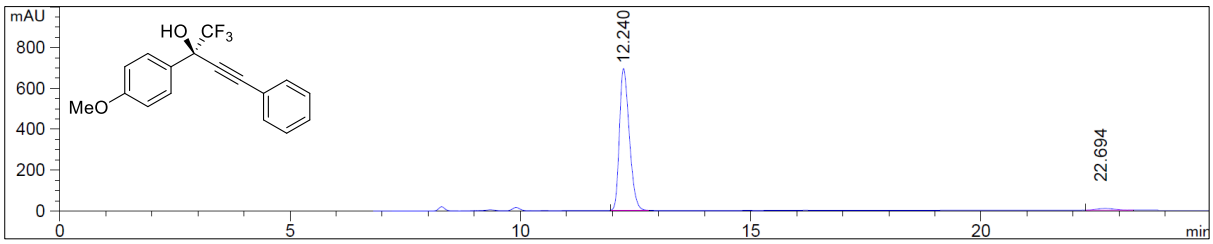
Racemic 3ea



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.796	BB	0.2126	5615.20752	408.01105	49.6589
2	21.192	BB	0.3985	5692.34326	218.12773	50.3411
Totals :				1.13076e4	626.13878	

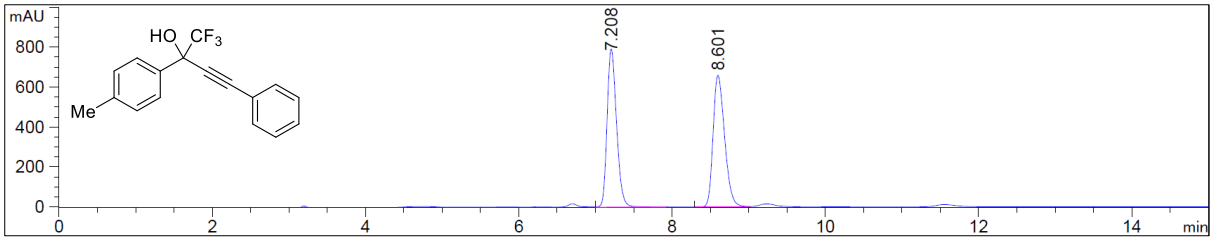
Enantioenriched 3ea



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.240	MM T	0.2478	1.03469e4	695.88904	97.9445
2	22.694	MM T	0.4294	217.14453	8.42797	2.0555
Totals :				1.05640e4	704.31701	

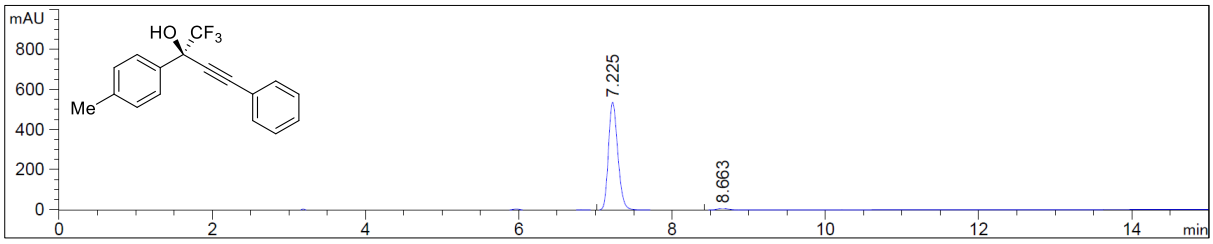
Racemic 3fa



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.208	VB	0.1318	6763.79443	790.41278	49.6057
2	8.601	BV	0.1660	6871.33252	657.30743	50.3943
Totals :				1.36351e4	1447.72021	

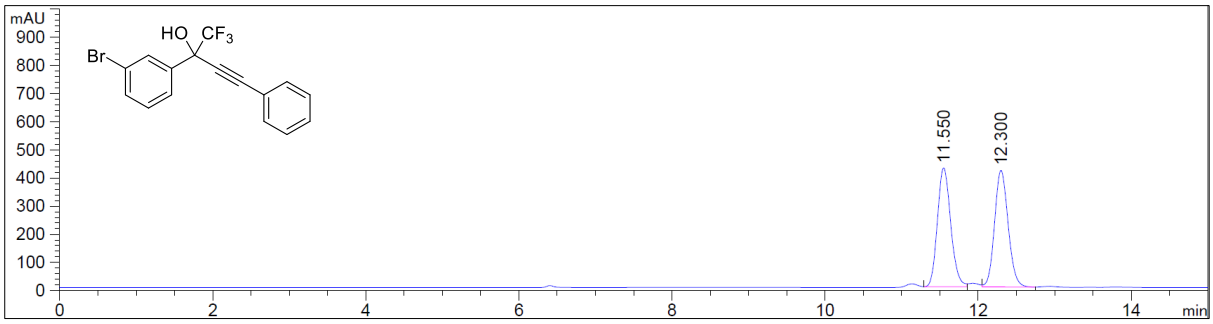
Enantioenriched 3fa



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.225	VB	0.1334	4580.36963	537.67596	98.4503
2	8.663	BB	0.1504	72.09988	7.35334	1.5497
Totals :				4652.46951	545.02931	

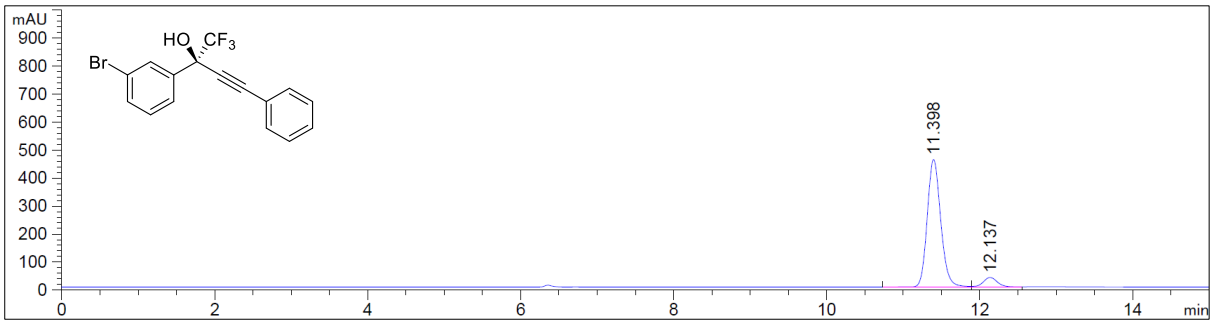
Racemic 3ga



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.550	BV	0.1889	5098.31445	422.24817	49.9791
2	12.300	VB	0.1898	5102.57080	413.86041	50.0209
Totals :				1.02009e4	836.10858	

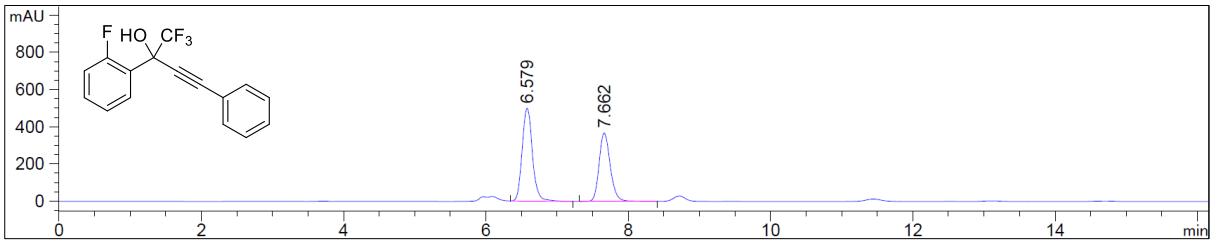
Enantioenriched 3ga



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.398	BV	0.1909	5573.68750	455.13403	92.6746
2	12.137	VV	0.1960	440.57138	34.26094	7.3254
Totals :				6014.25888	489.39498	

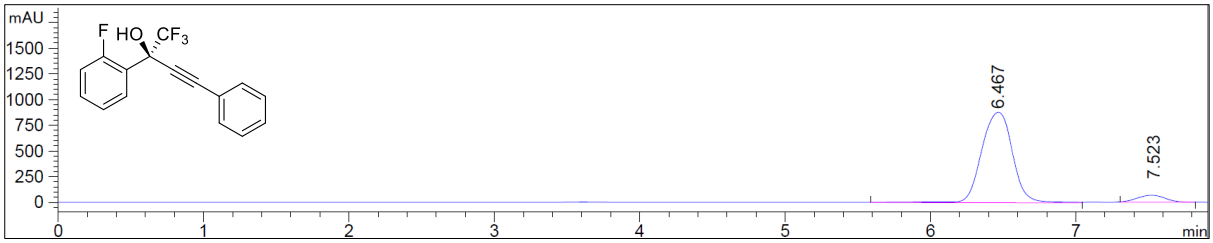
Racemic 3ha



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.579	BB	0.1575	5030.50830	499.48083	55.6302
2	7.662	BV	0.1671	4012.25366	368.32654	44.3698
Totals :				9042.76196	867.80737	

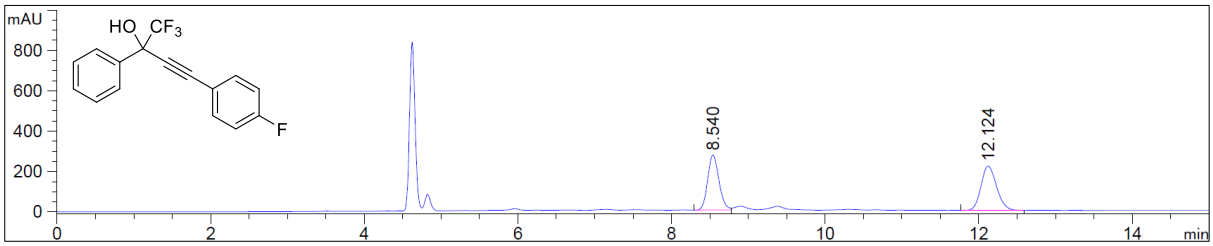
Enantioenriched 3ha



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.467	MM T	0.2475	1.30589e4	879.53400	93.6053
2	7.523	MM T	0.2215	892.12994	67.11796	6.3947
Totals :				1.39510e4	946.65195	

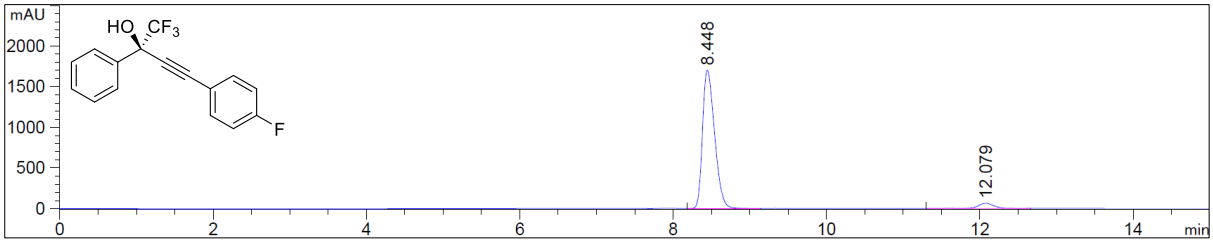
Racemic 3ab



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.540	BV	0.1593	2771.67334	271.11499	47.2416
2	12.124	BV	0.2162	3095.34009	219.89032	52.7584
Totals :				5867.01343	491.00531	

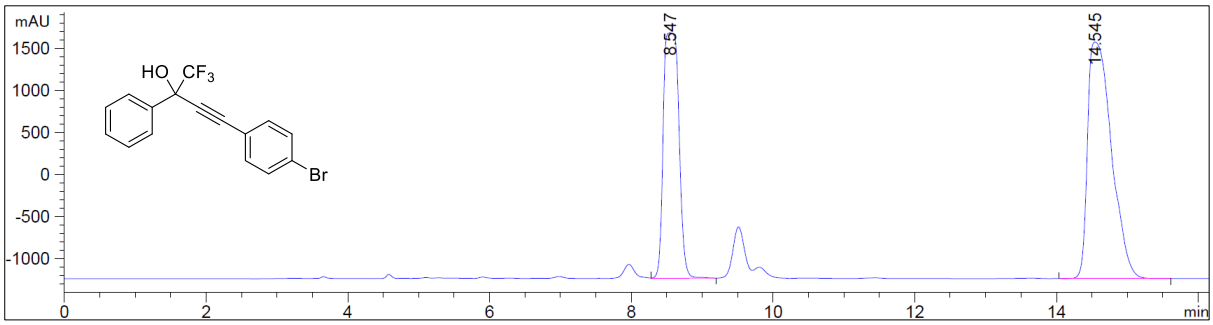
Enantioenriched 3ab



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.448	VV	0.1713	1.85722e4	1701.68274	95.4708
2	12.079	VV	0.2142	881.07227	63.35938	4.5292
Totals :				1.94533e4	1765.04211	

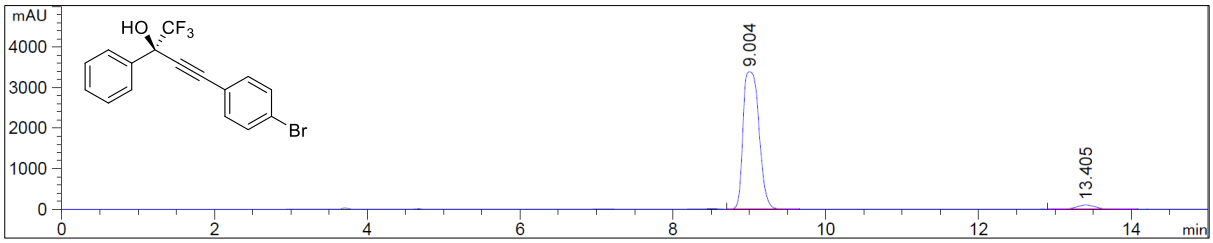
Racemic 3ac



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.547	VB	0.2459	4.44456e4	2914.54517	40.3203
2	14.545	BB	0.3686	6.57857e4	2811.18628	59.6797
Totals :				1.10231e5	5725.73145	

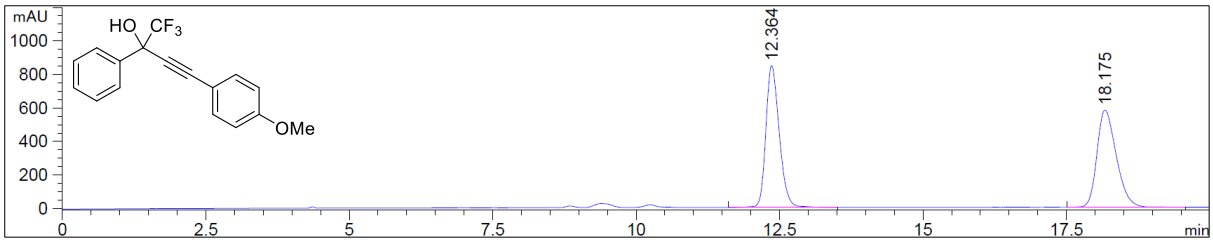
Enantioenriched 3ac



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.004	VV	0.2305	4.88785e4	3385.17822	96.8035
2	13.405	BB	0.2407	1614.00610	104.20264	3.1965
Totals :				5.04925e4	3489.38087	

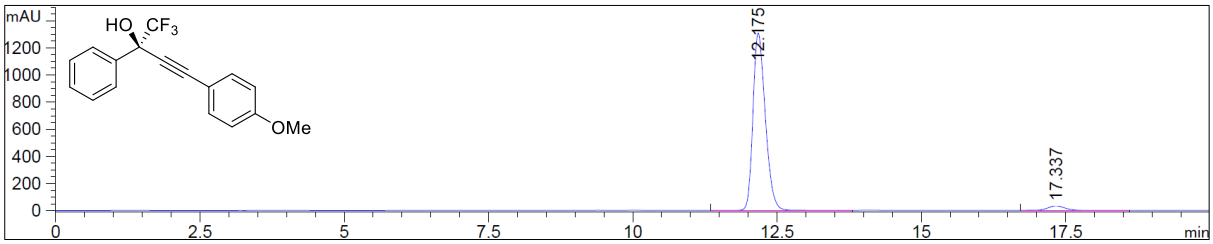
Racemic 3ad



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.364	BV	0.2492	1.37259e4	846.13507	50.3008
2	18.175	BB	0.3607	1.35618e4	580.18530	49.6992
Totals :				2.72877e4	1426.32037	

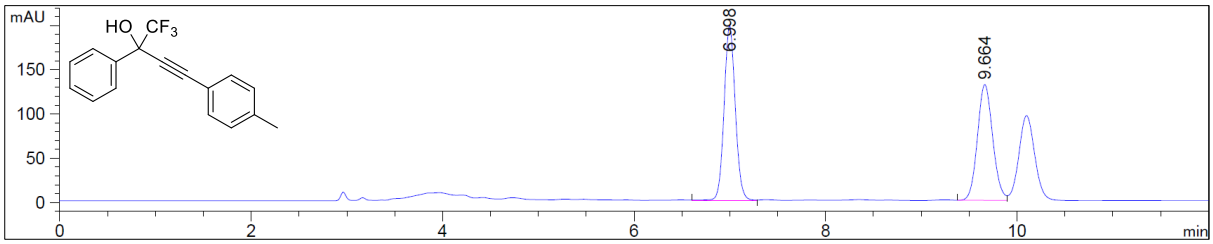
Enantioenriched 3ad



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.175	BV	0.2292	1.93857e4	1306.10767	96.6746
2	17.337	BB	0.3178	666.82703	32.70996	3.3254
Totals :				2.00525e4	1338.81763	

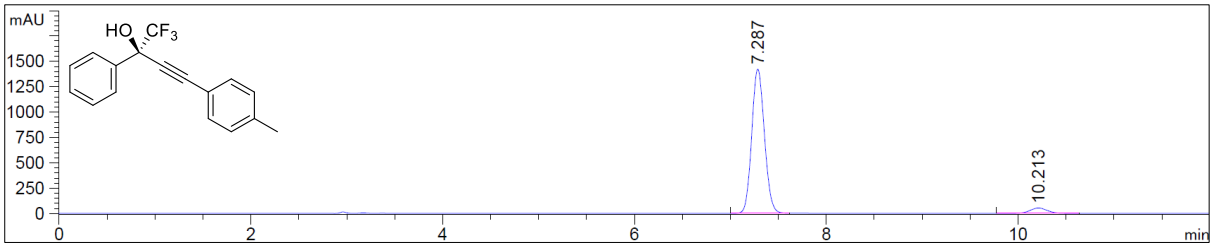
Racemic 3ae



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.998	VV	0.1267	1602.97388	197.45538	52.4931
2	9.664	BV	0.1732	1450.71130	130.96457	47.5069
Totals :				3053.68518	328.41995	

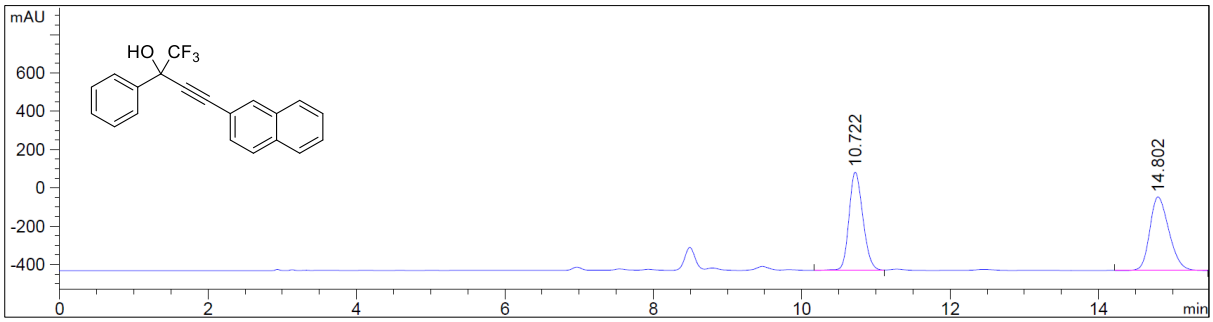
Enantioenriched 3ae



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.287	BV	0.1452	1.30561e4	1420.86133	95.2771
2	10.213	VV	0.1892	647.19513	52.72830	4.7229
Totals :				1.37033e4	1473.58963	

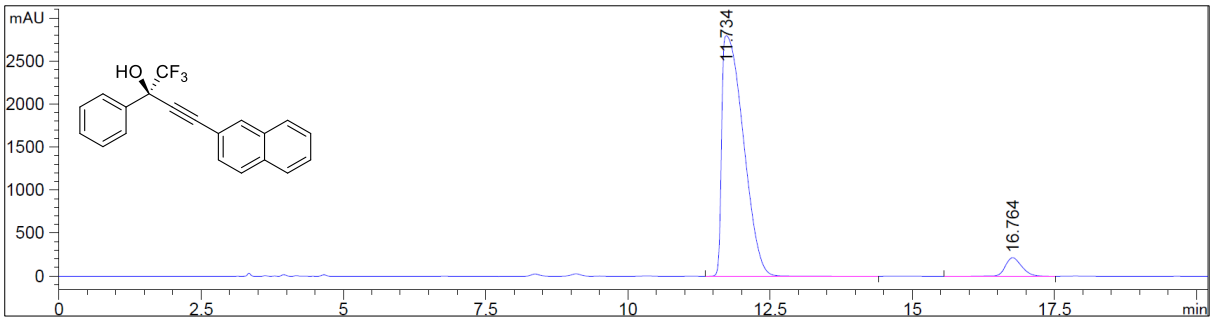
Racemic 3af



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.722	BV	0.2016	6669.84424	513.04938	49.2508
2	14.802	BBA	0.2769	6872.76123	383.90787	50.7492
Totals :				1.35426e4	896.95724	

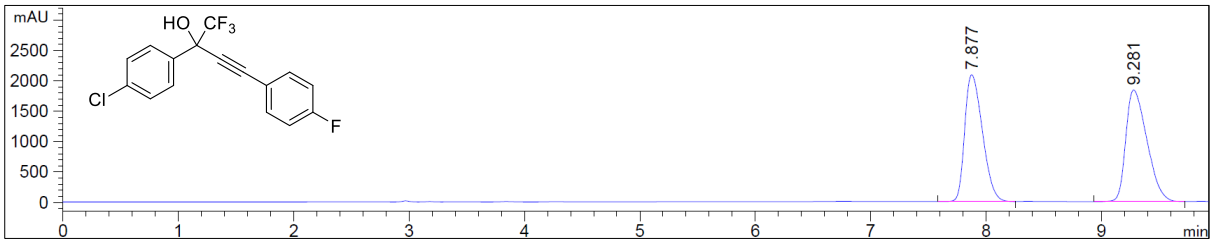
Enantioenriched 3af



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.734	BV	0.4197	7.37367e4	2795.73999	94.5055
2	16.764	BV	0.3041	4287.04248	215.23865	5.4945
Totals :				7.80238e4	3010.97864	

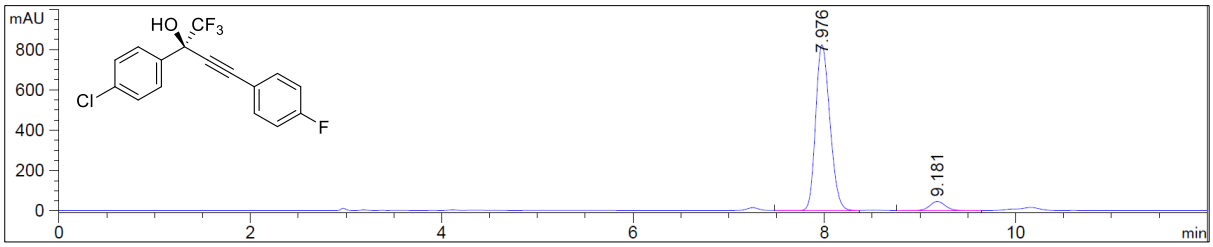
Racemic 3cb



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.877	VV	0.1687	2.23440e4	2090.93481	48.8914
2	9.281	BB	0.2003	2.33573e4	1837.32886	51.1086
Totals :				4.57013e4	3928.26367	

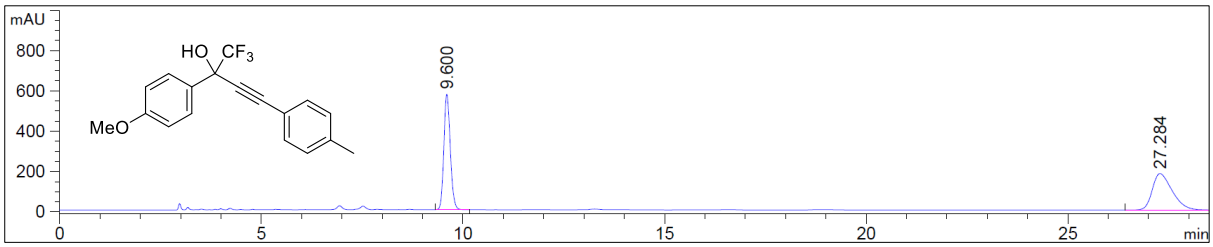
Enantioenriched 3cb



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.976	VV	0.1624	8487.25586	822.15076	94.3225
2	9.181	BV	0.1736	510.86423	45.28932	5.6775
Totals :				8998.12009	867.44008	

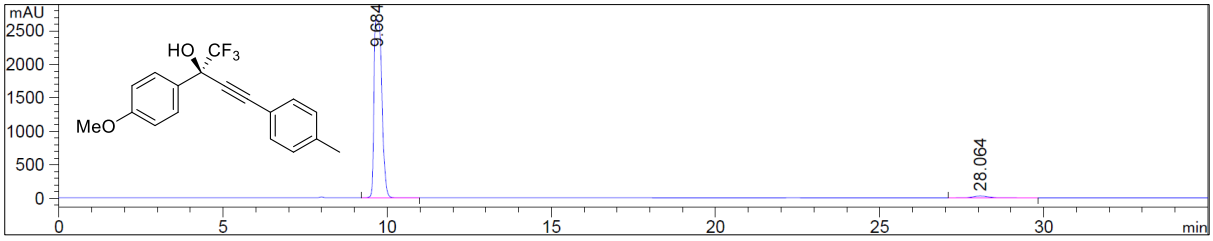
Racemic 3ee



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.600	VV	0.1712	6343.68701	572.83759	50.7212
2	27.284	BBA	0.5220	6163.29248	180.65030	49.2788
Totals :				1.25070e4	753.48788	

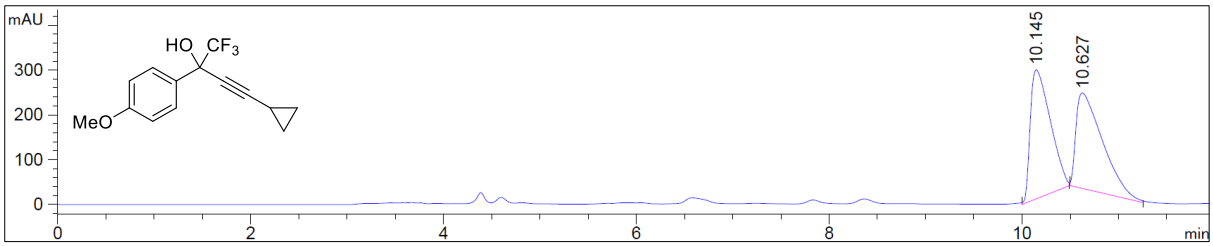
Enantioenriched 3ee



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.684	VV	0.2616	4.36613e4	2658.75806	97.9380
2	28.064	BB	0.5130	919.27368	27.71325	2.0620
Totals :				4.45806e4	2686.47131	

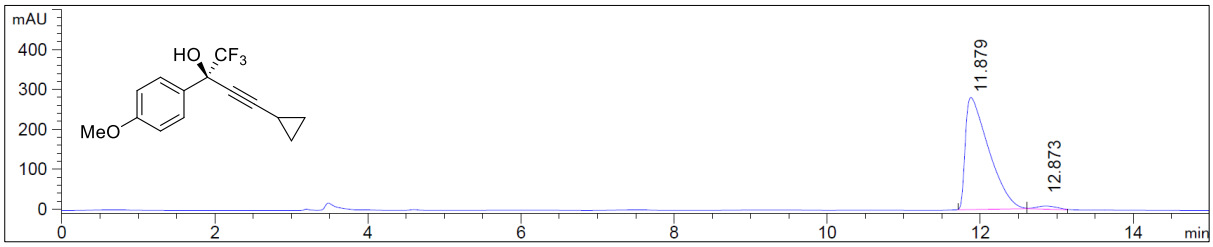
Racemic 3eg



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.145	MM T	0.2390	4132.77051	288.21365	50.1379
2	10.627	MM T	0.3213	4110.03418	213.22989	49.8621
Totals :				8242.80469	501.44354	

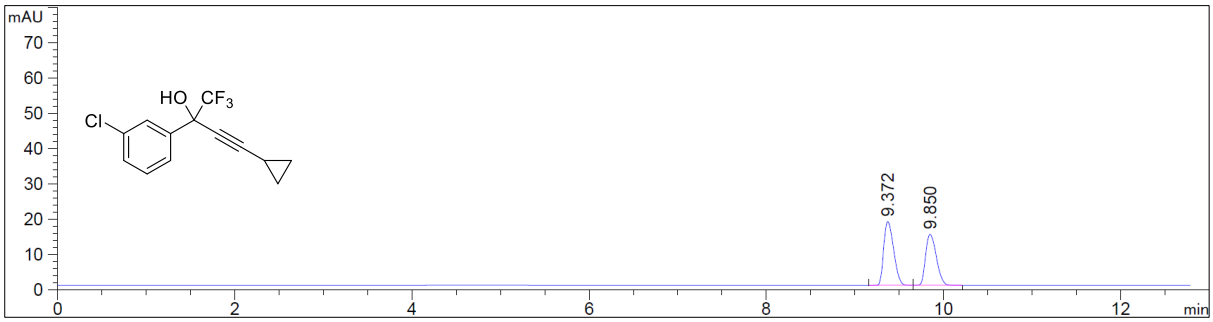
Enantioenriched 3eg



Signal 2: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.879	MM T	0.3534	5950.80176	280.63693	97.6302
2	12.873	MM T	0.3043	144.44800	7.91217	2.3698

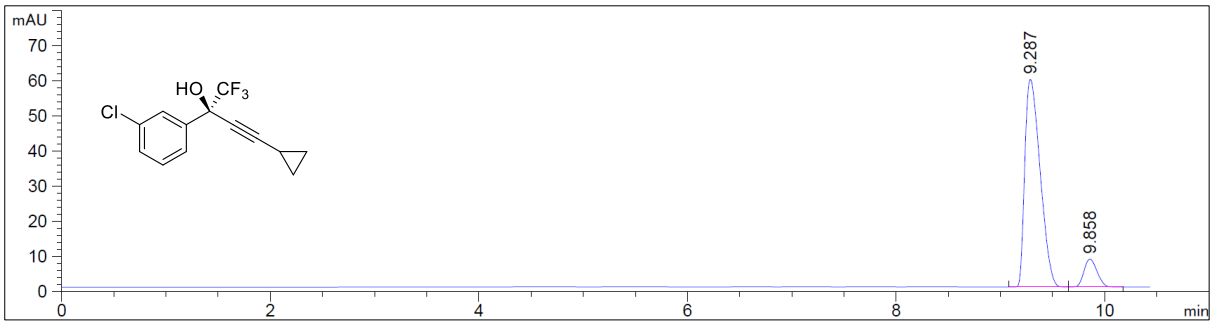
Racemic 3eh



Signal 1: MWD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.372	BB	0.1246	140.50111	18.09298	53.5707
2	9.850	BB	0.1342	121.77124	14.46669	46.4293
Totals :				262.27235	32.55967	

Enantioenriched 3eh

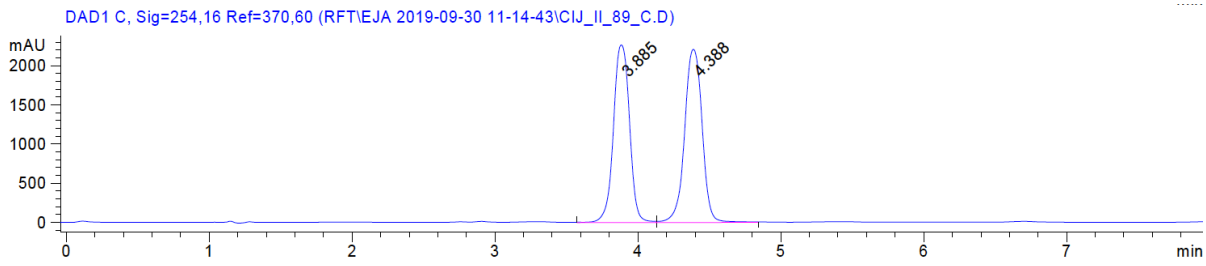


Signal 1: MWD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.287	BB	0.1629	603.86737	59.24526	89.7577
2	9.858	BBA	0.1350	68.90726	7.96194	10.2423
Totals :				672.77464	67.20720	

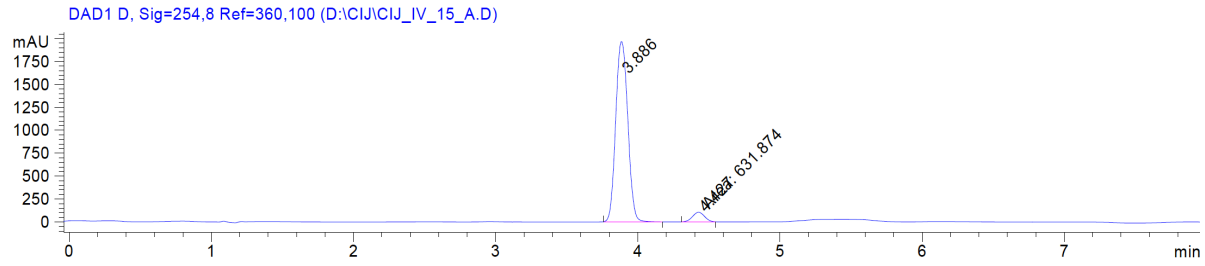
SFC data for Vinyl Trifluoromethyl Ketone Products:

Racemic 5aa



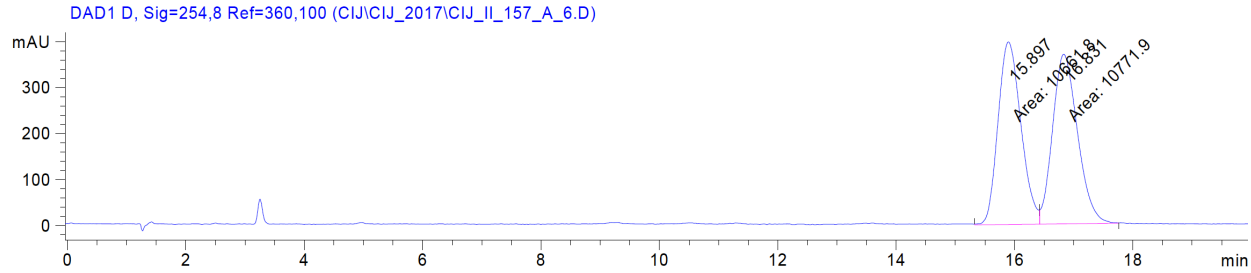
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.885	VV	0.1234	1.77626e4	2267.70264	48.5024
2	4.388	VB	0.1356	1.88595e4	2208.62769	51.4976

Enantioenriched 5aa



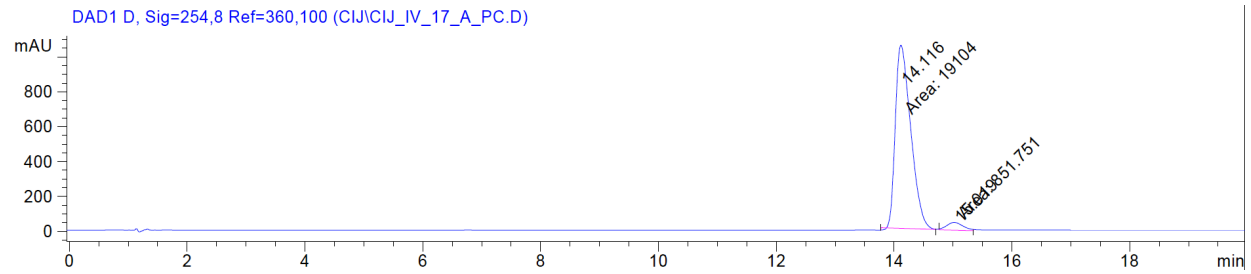
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.886	BB	0.0932	1.16488e4	1963.02271	94.8547
2	4.427	MM	0.0985	631.87439	106.89740	5.1453

Racemic 5ba



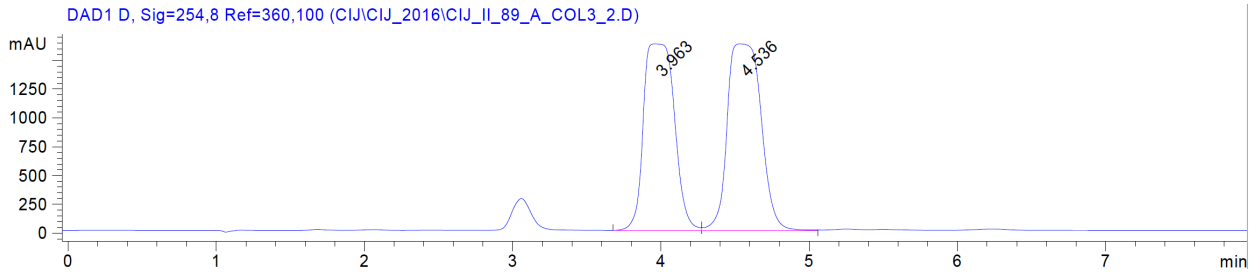
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.897	MF	0.4483	1.06618e4	396.33667	49.7431
2	16.831	FM	0.4871	1.07719e4	368.60919	50.2569

Enantioenriched 5ba



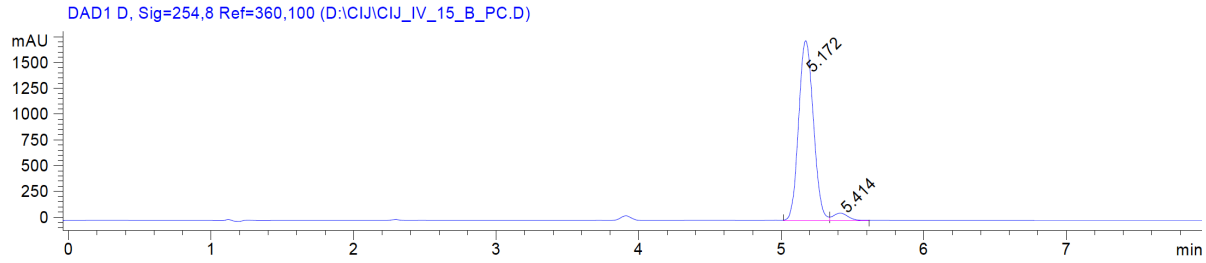
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.116	MM	0.3030	1.91040e4	1050.90735	95.7318
2	15.019	MM	0.3204	851.75140	44.30221	4.2682

Racemic 5ca



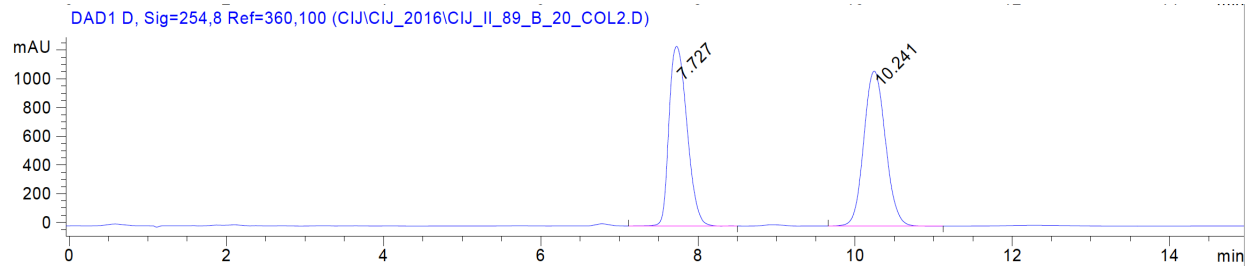
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.963	BV	0.2325	2.33956e4	1620.28918	47.7740
2	4.536	VV	0.2561	2.55758e4	1620.41089	52.2260

Enantioenriched 5ca



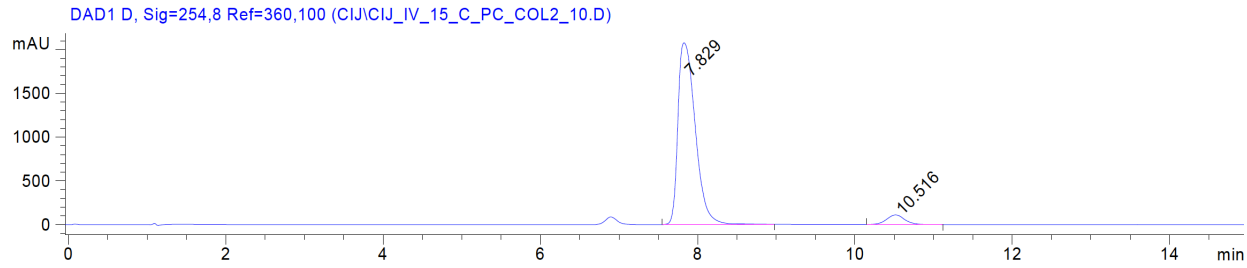
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.172	BV	0.1119	1.25042e4	1738.45972	96.0937
2	5.414	VB	0.1121	508.31110	70.54485	3.9063

Racemic 5da



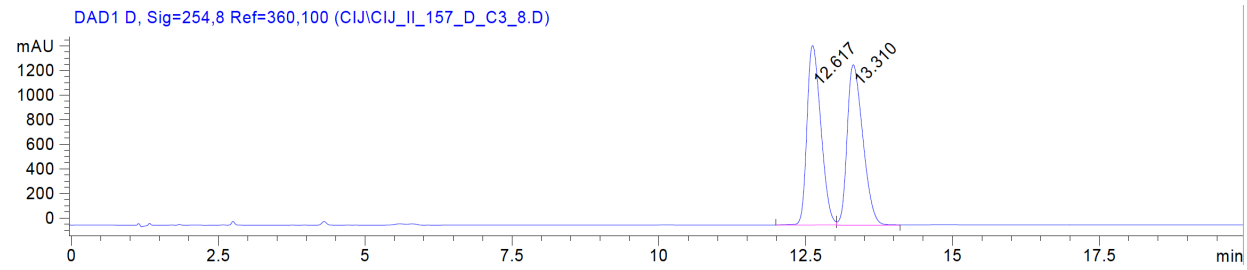
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.727	VB	0.2539	1.99510e4	1252.39722	49.0524
2	10.241	BB	0.3000	2.07219e4	1079.43774	50.9476

Enantioenriched 5da



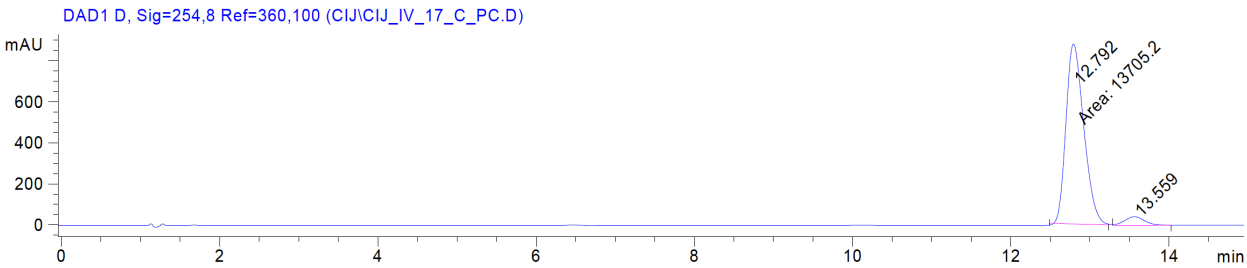
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.829	BB	0.2428	3.18149e4	2075.78711	94.7337
2	10.516	BB	0.2476	1768.60828	110.00776	5.2663

Racemic 5ea



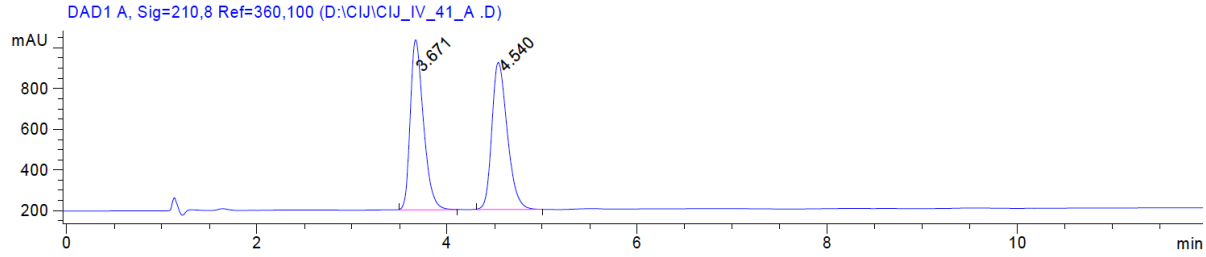
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.617	BV	0.2555	2.39572e4	1459.49976	49.8502
2	13.310	VB	0.2898	2.41012e4	1303.09802	50.1498

Enantioenriched 5ea



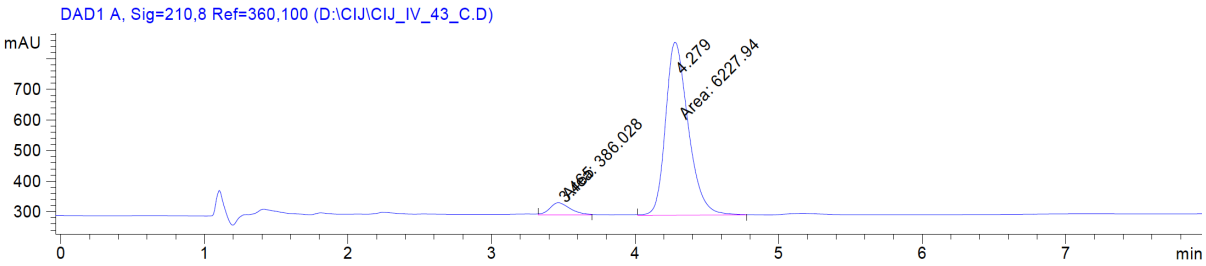
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.792	MM	0.2605	1.37052e4	876.92377	95.0351
2	13.559	VB	0.2631	715.99207	41.95095	4.9649

Racemic 5fa



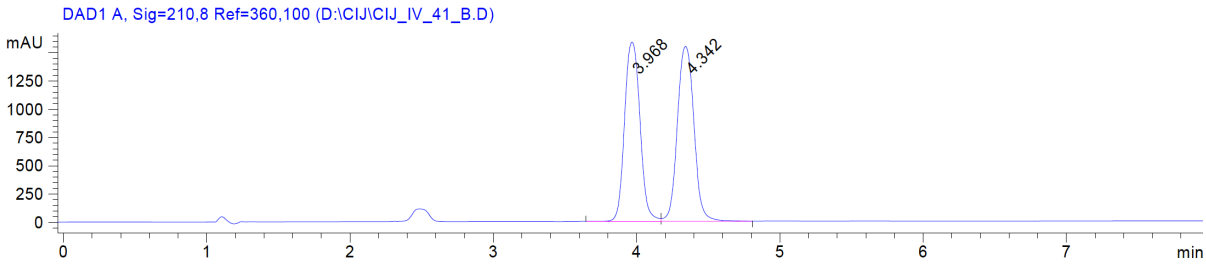
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.671	BB	0.1490	8086.00391	834.70844	49.7844
2	4.540	BB	0.1717	8156.03906	722.36987	50.2156

Enantioenriched 5fa



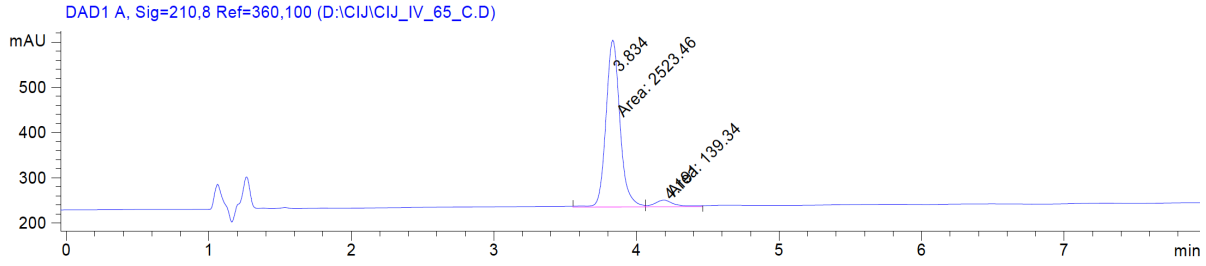
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.465	MM	0.1624	386.02798	39.61908	5.8366
2	4.279	MM	0.1831	6227.94189	566.79132	94.1634

Racemic 5ga



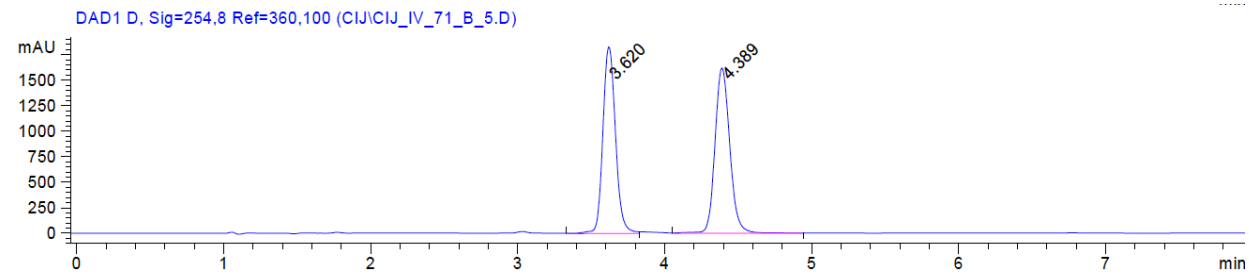
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.968	VV	0.1195	1.18848e4	1585.30981	48.8014
2	4.342	VV	0.1260	1.24686e4	1547.48511	51.1986

Enantioenriched 5ga



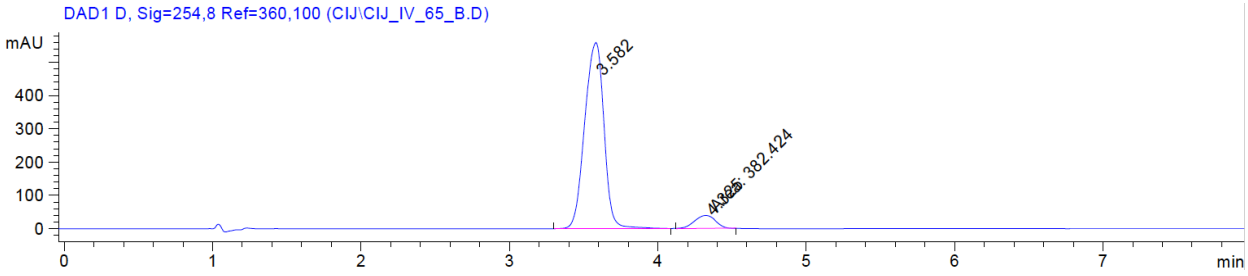
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.834	MF	0.1139	2523.45898	369.41278	94.7672
2	4.191	FM	0.1556	139.33974	14.92964	5.2328

Racemic 5ha



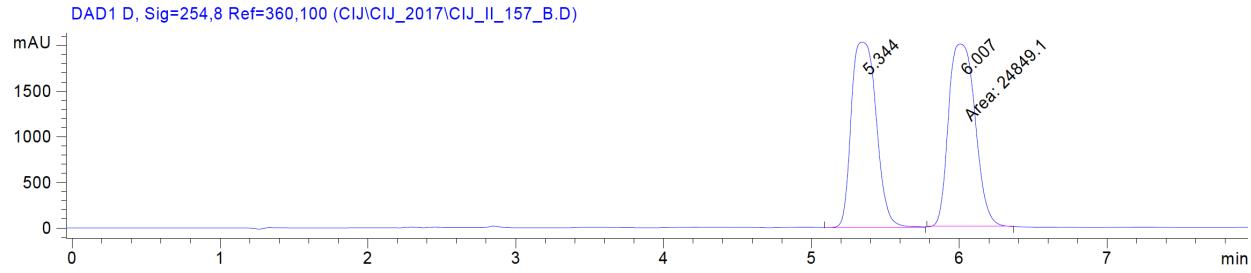
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.620	VV	0.0938	1.09643e4	1831.39258	48.9468
2	4.389	VV	0.1102	1.14361e4	1623.07410	51.0532

Enantioenriched 5ha



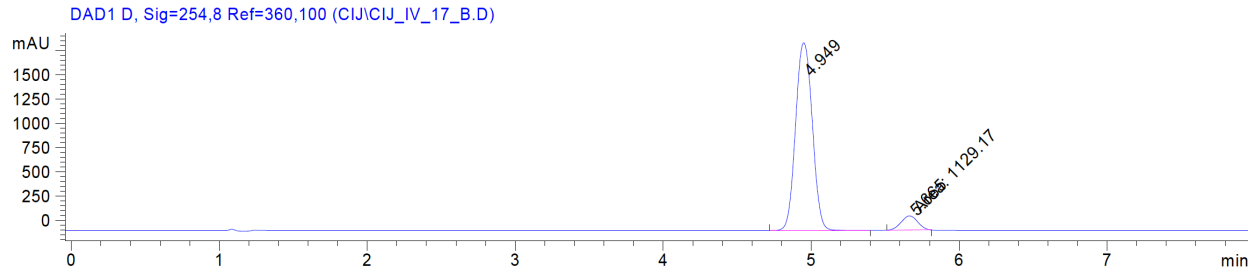
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.582	BB	0.1456	5064.83740	559.75128	92.9795
2	4.325	MM	0.1644	382.42398	38.77777	7.0205

Racemic 5ia



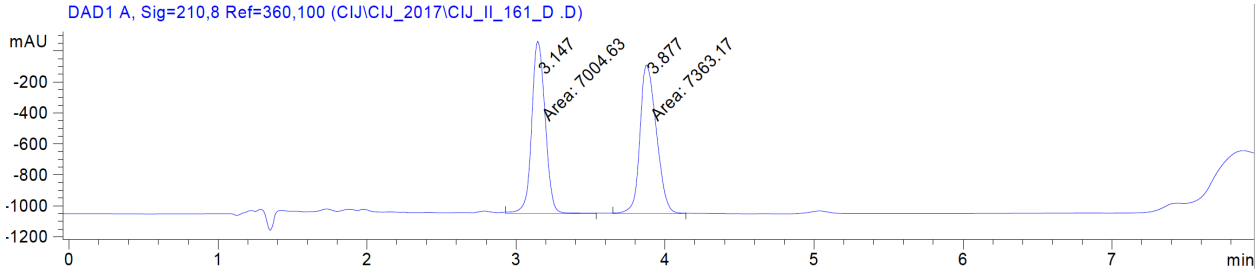
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.344	VV	0.1902	2.40919e4	2033.53210	49.2264
2	6.007	MM	0.2077	2.48491e4	1993.68933	50.7736

Enantioenriched 5ia



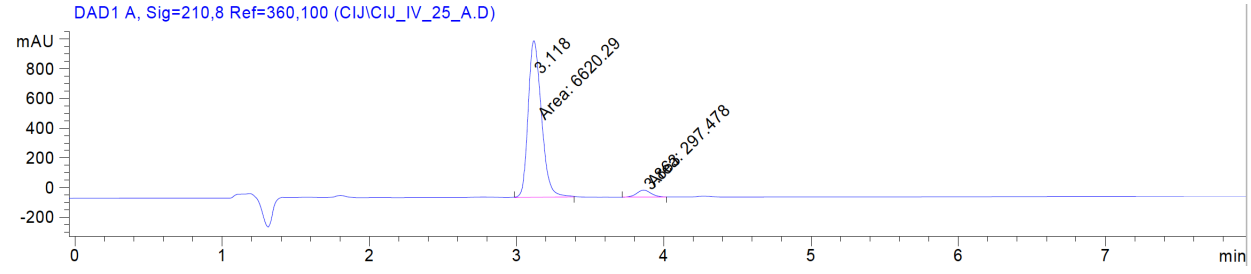
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.949	BB	0.1256	1.51929e4	1935.24060	93.0819
2	5.665	MM	0.1293	1129.16772	145.50713	6.9181

Racemic 5ja



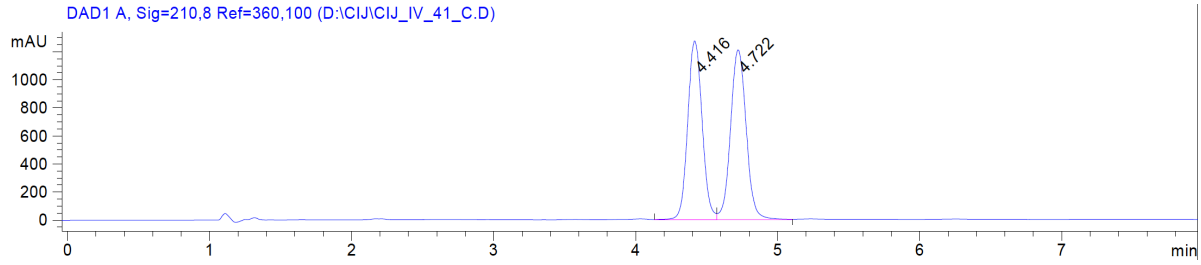
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.147	MM	0.1045	7004.62793	1117.27197	48.7523
2	3.877	MM	0.1277	7363.17383	961.19696	51.2477

Enantioenriched 5ja



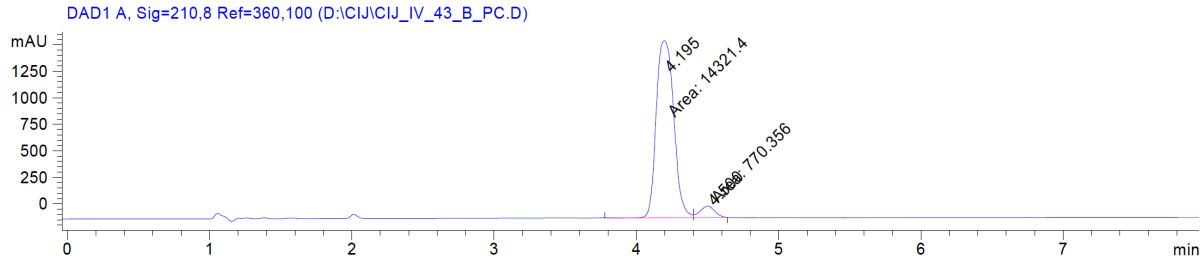
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.118	MM	0.1040	6620.28564	1060.52246	95.6998
2	3.863	MM	0.1079	297.47842	45.96951	4.3002

Racemic 5ka



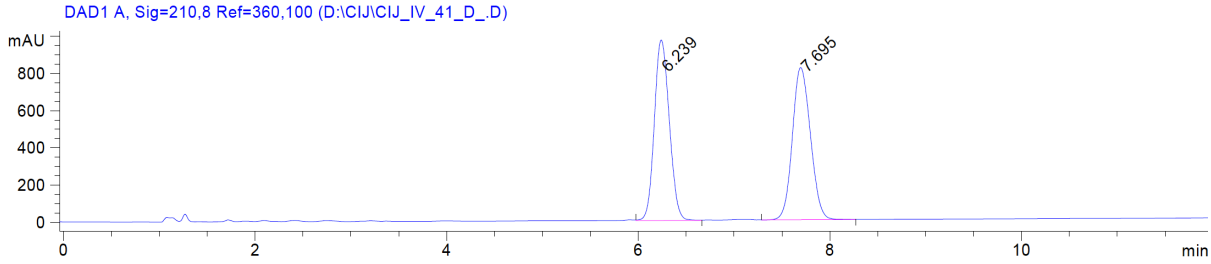
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.416	VV	0.1109	9051.17480	1273.78198	49.3289
2	4.722	VV	0.1177	9297.46680	1209.88831	50.6711

Enantioenriched 5ka



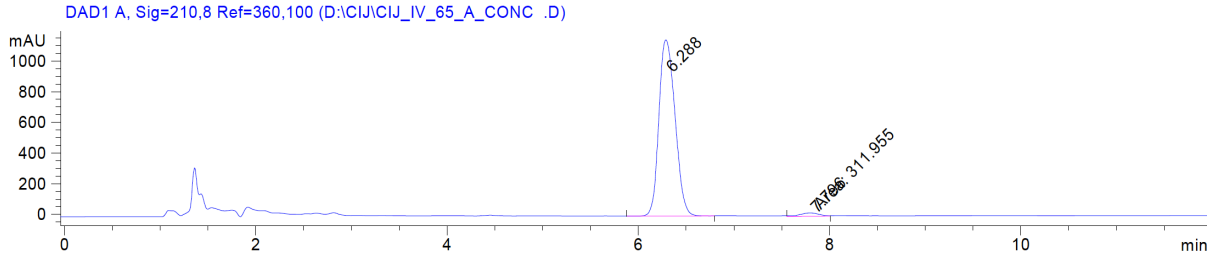
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.195	MF	0.1430	1.43214e4	1668.58252	94.8955
2	4.500	FM	0.1187	770.35632	108.20892	5.1045

Racemic 5la



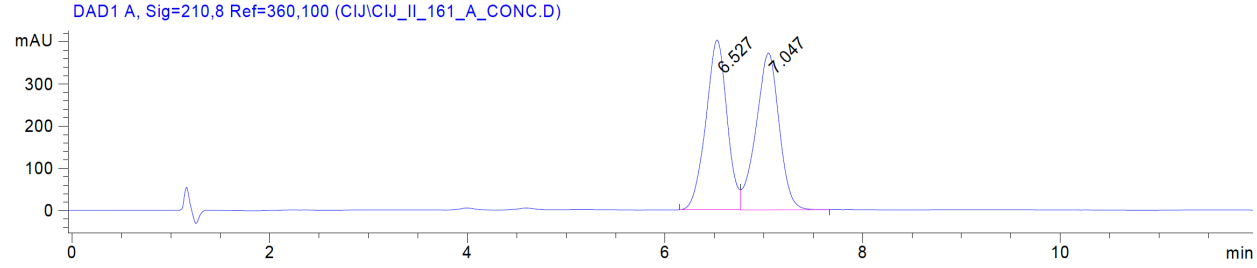
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.239	VB	0.1726	1.05089e4	968.62952	49.4613
2	7.695	VB	0.2052	1.07378e4	817.57489	50.5387

Enantioenriched 5la



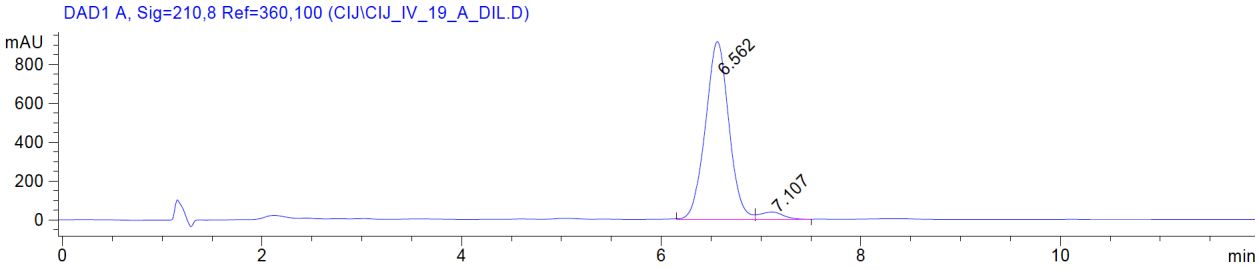
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.288	BB	0.1869	1.34793e4	1149.03198	97.7380
2	7.796	MM	0.2344	311.95532	22.18306	2.2620

Racemic 5ma



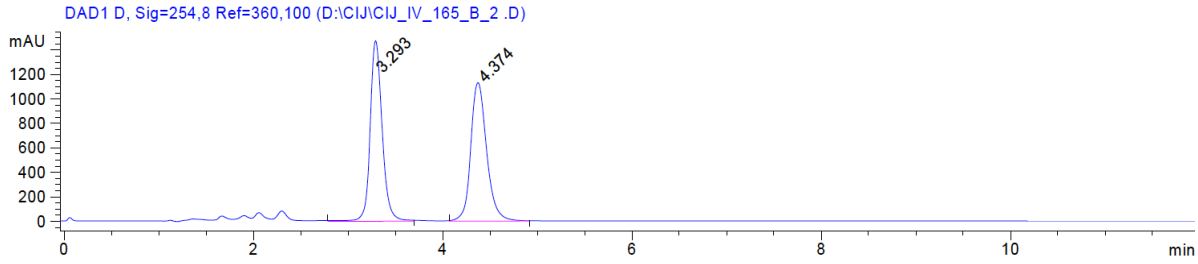
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.527	BV	0.2267	6022.24268	402.25772	49.7994
2	7.047	VB	0.2426	6070.75537	371.66992	50.2006

Enantioenriched 5ma



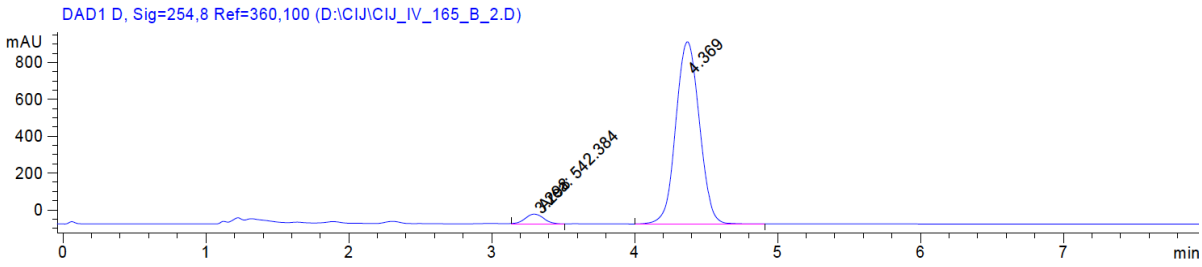
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.562	BV	0.2463	1.49132e4	914.24634	96.2033
2	7.107	VB	0.2379	588.55762	37.34389	3.7967

Racemic 5na



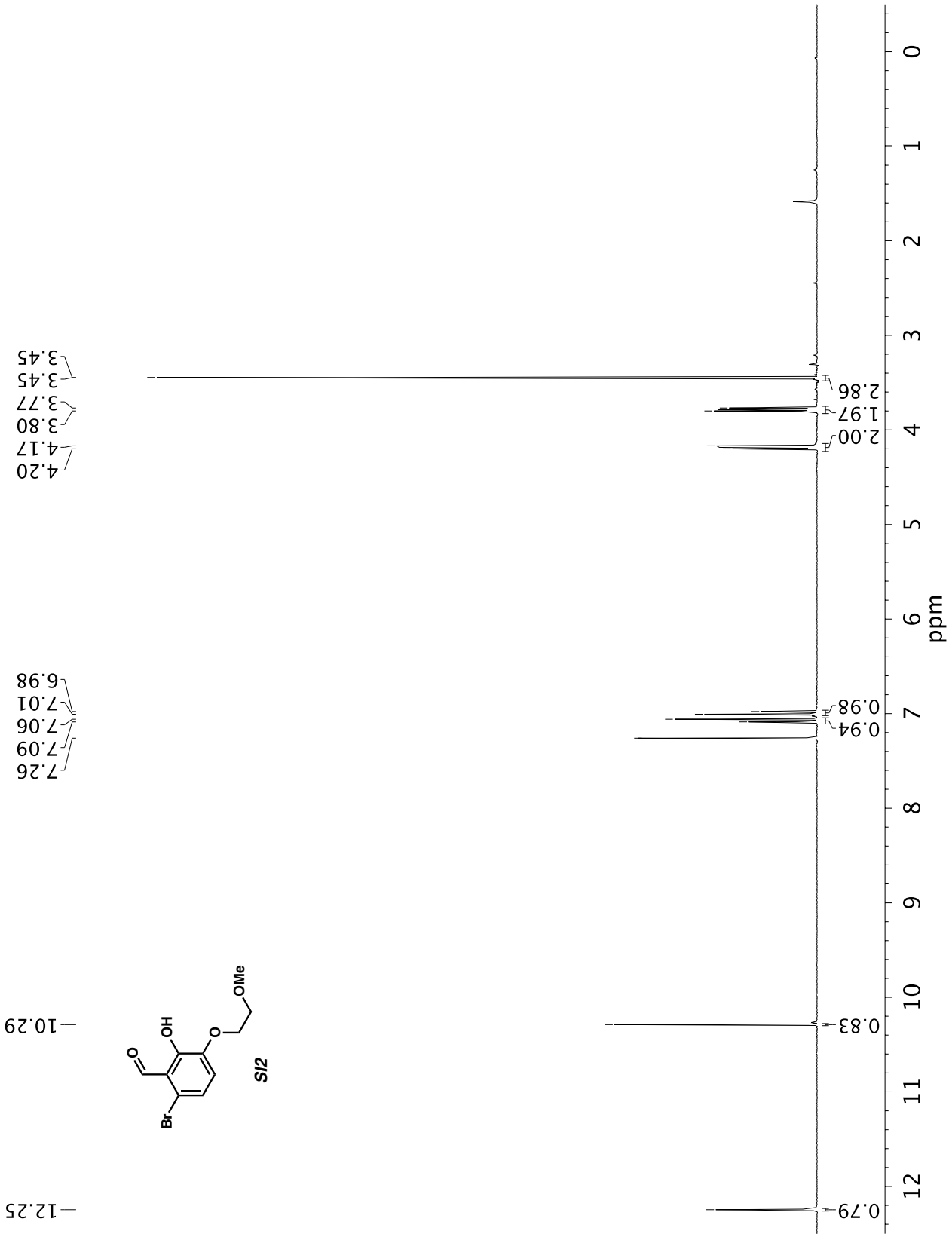
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.293	BB	0.1487	1.21569e4	1258.85498	47.9847
2	4.374	BB	0.1971	1.31781e4	1059.61987	52.0153

Enantioenriched 5na

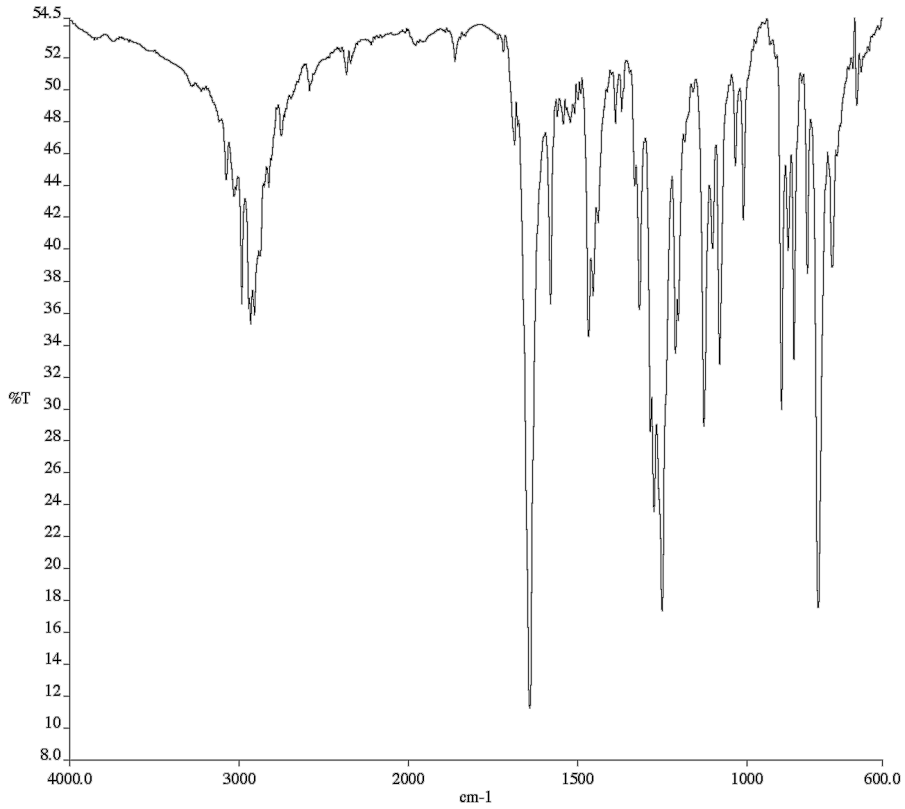


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.298	MM	0.1606	542.38373	56.29001	4.5637
2	4.369	BB	0.1819	1.13423e4	988.30817	95.4363

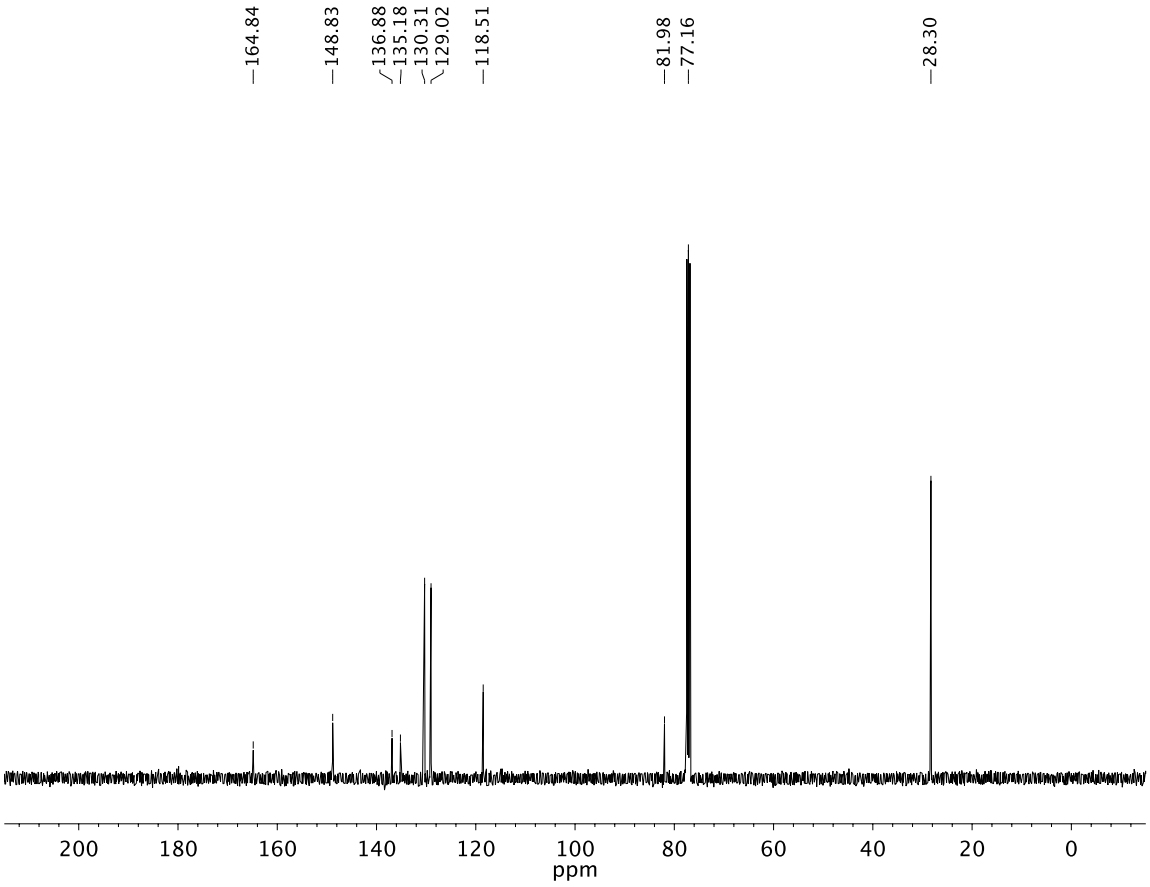
^{1}H, ^{13}C, ^{19}F NMR Spectra for Ligands Synthesized:



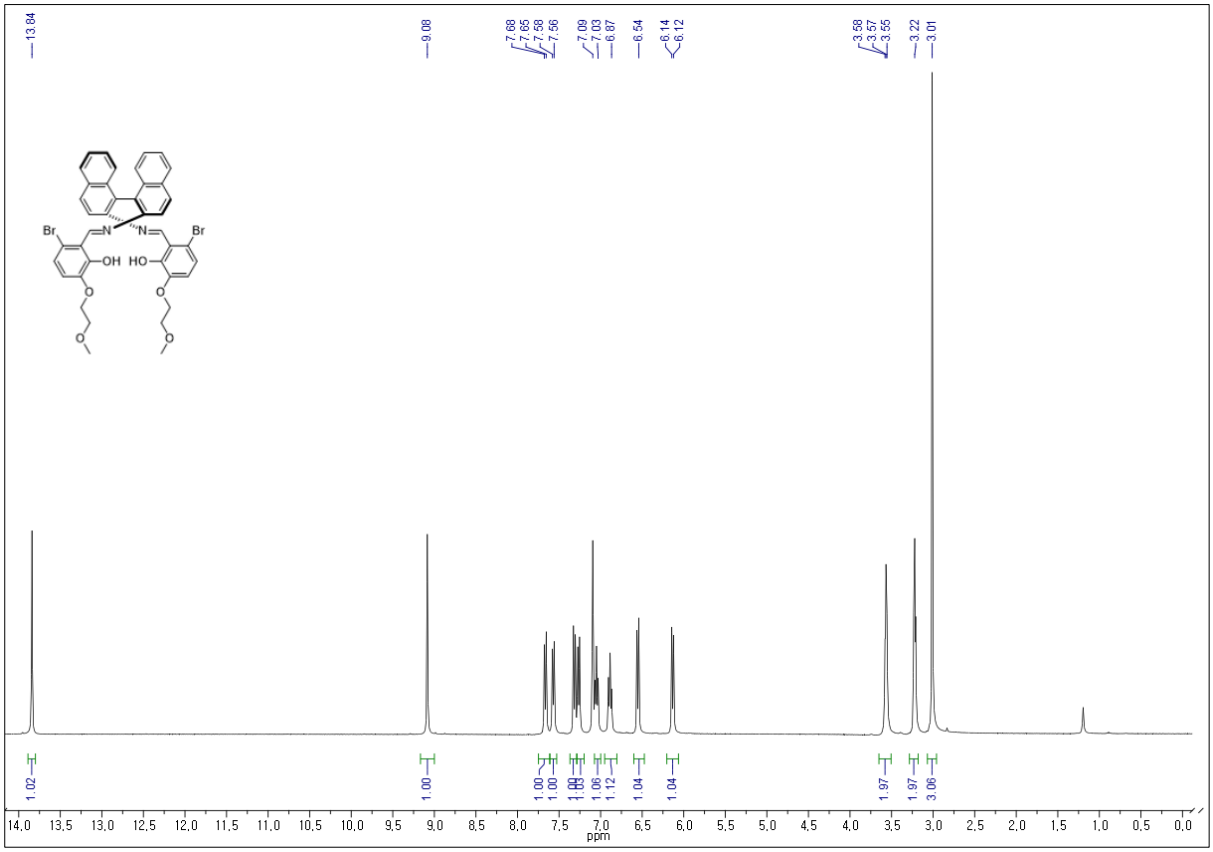
^{1}H NMR (300 MHz, $CDCl_3$) of compound **SI2**



Infrared spectrum (Thin Film, NaCl) of compound **SI2**.



^{13}C NMR (101 MHz, $CDCl_3$) of compound **SI2**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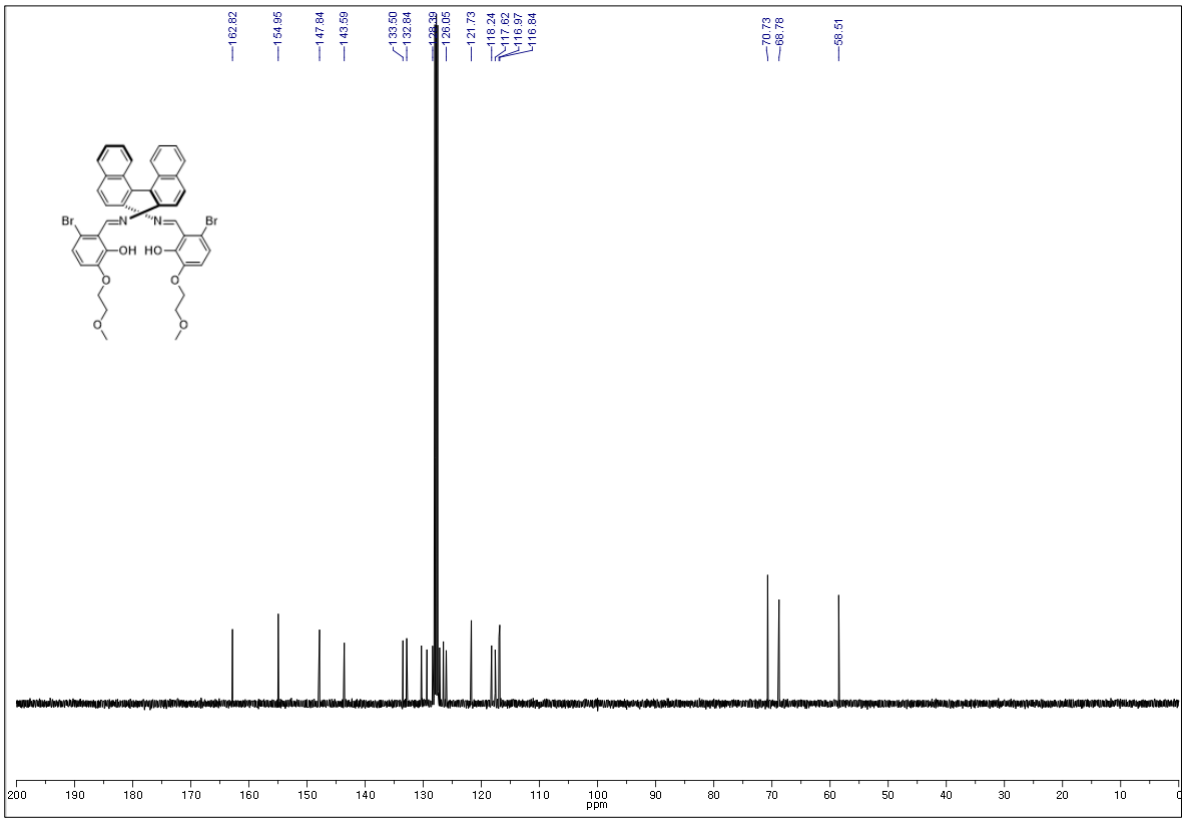
7.65
7.58
7.56
7.09
7.03
6.87
6.54
6.14
6.12
3.58
3.57
3.55
3.22
3.01
Br
N
N
Br
OH
HO
O
O
O
O
1.02
1.00
1.00
1.06
1.12
1.04
1.04
1.97
1.97
3.06
ppm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68.78
58.51
Br
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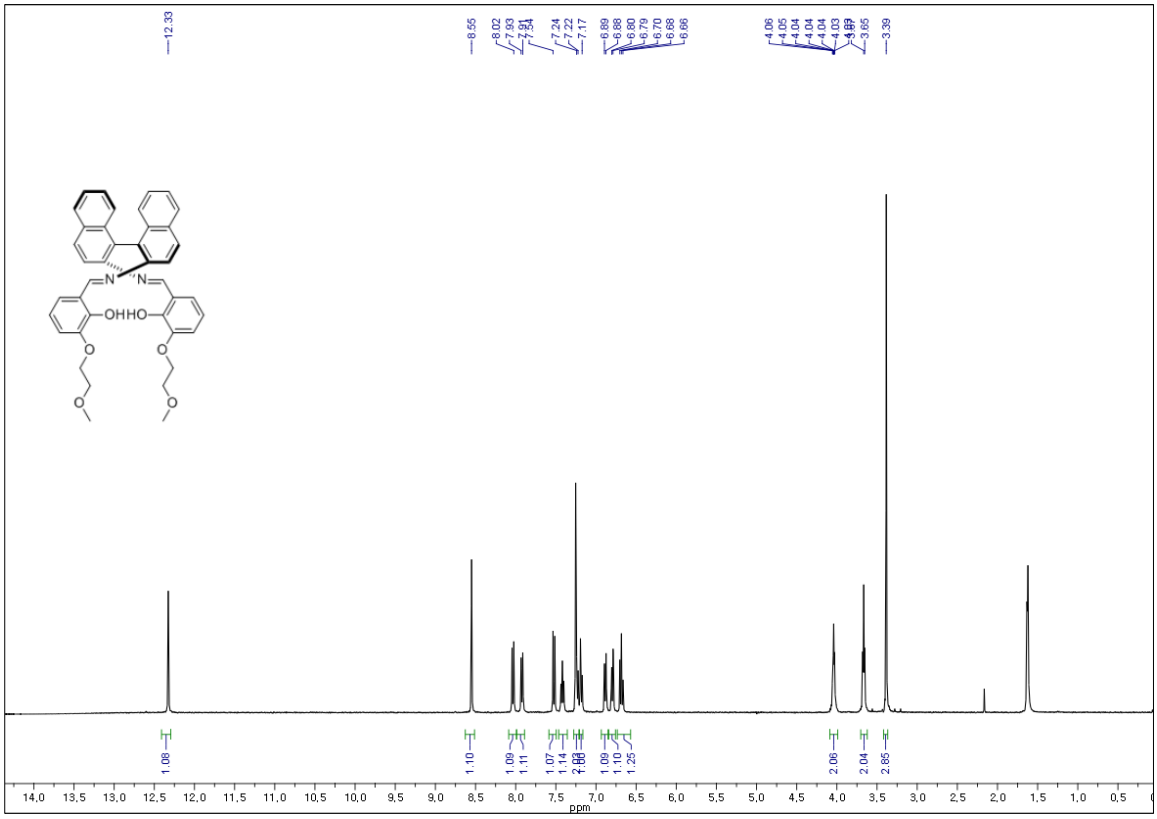
N
Br
OH
HO
O
O
O
O
ppm


12.33
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6.88
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6.68
6.66
4.06
4.05
4.04
4.04
4.04
4.03
3.65
3.39
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1.11
1.07
1.14
1.09
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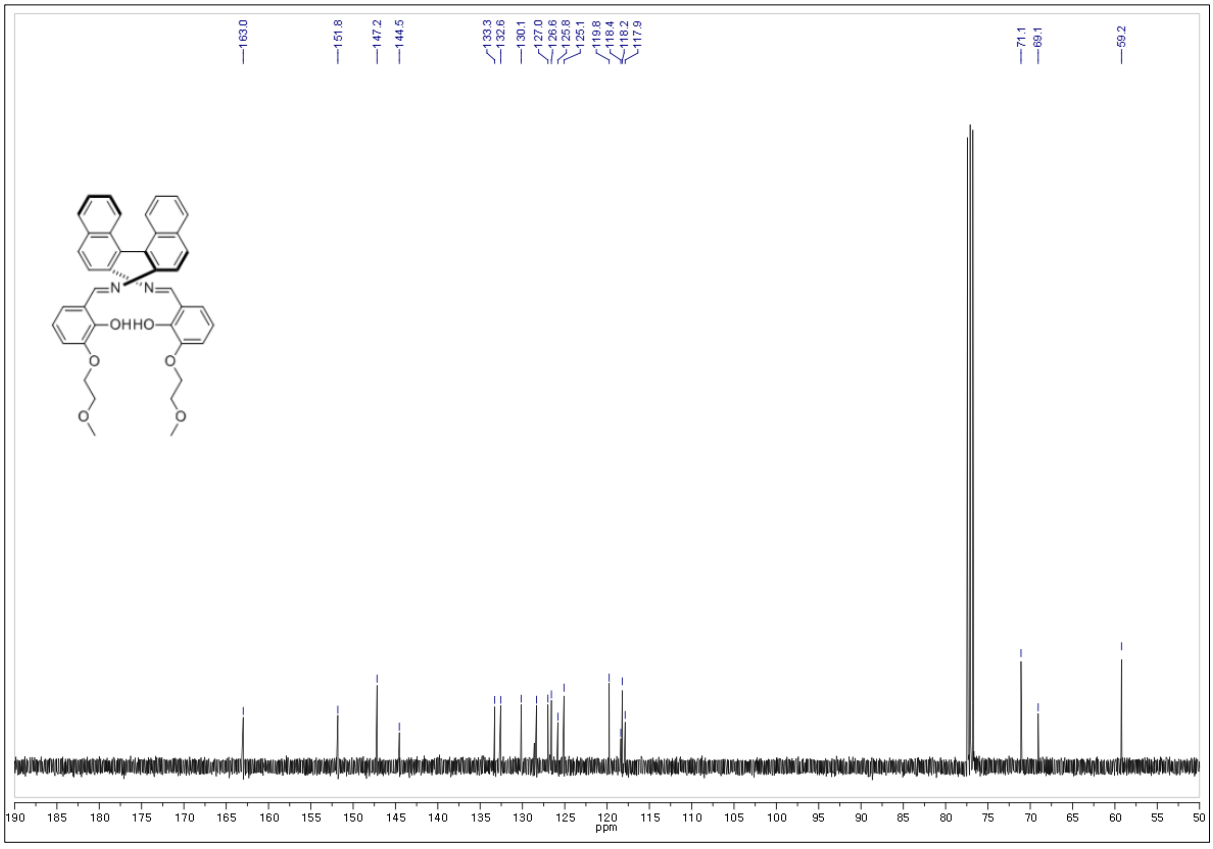
1.25
2.06
2.04
2.85
ppm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133.3
132.6
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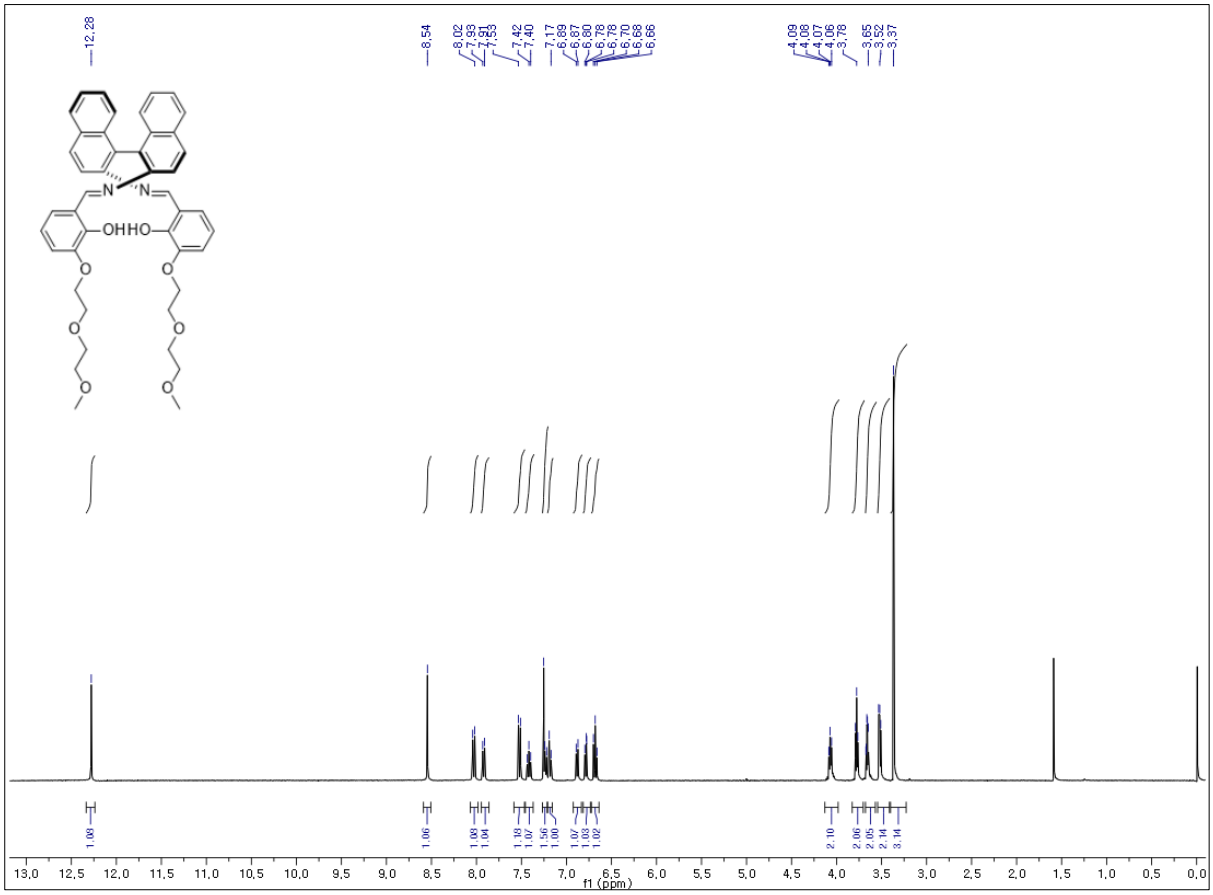
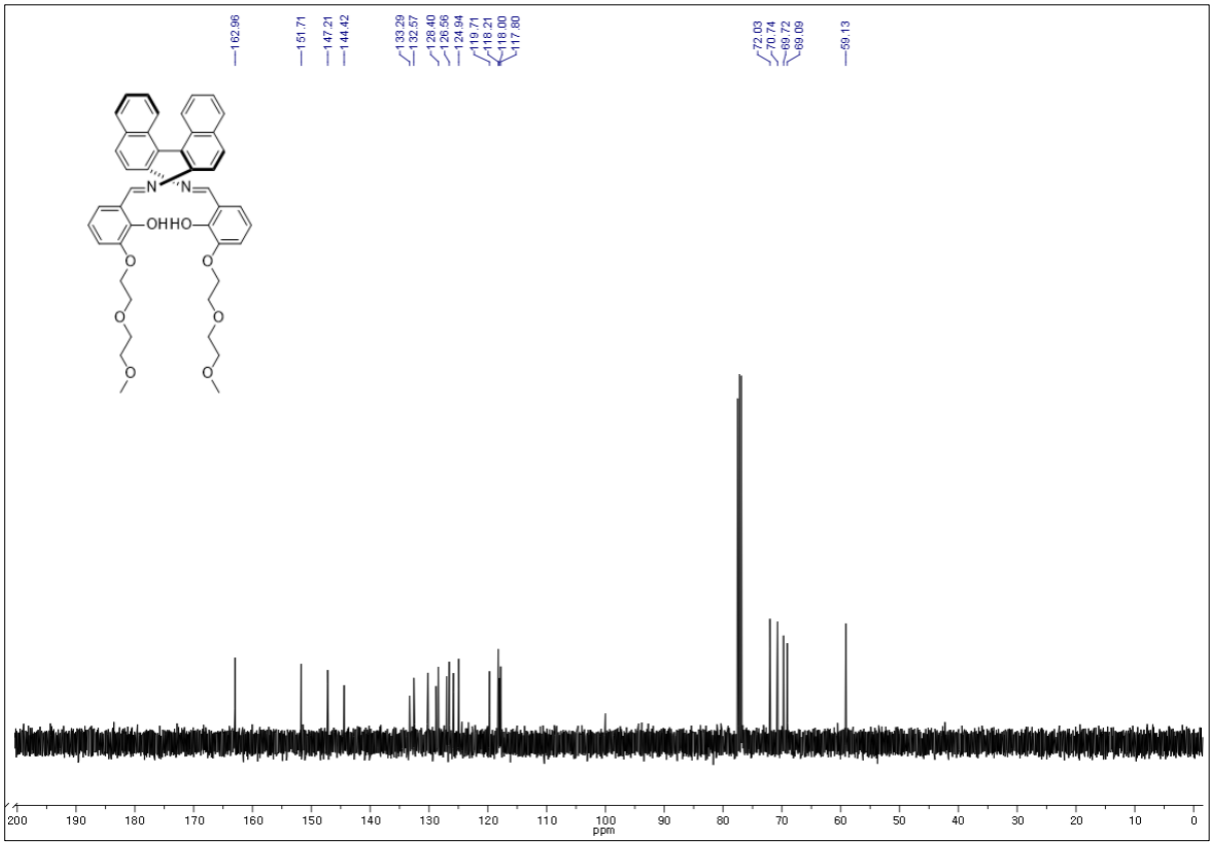
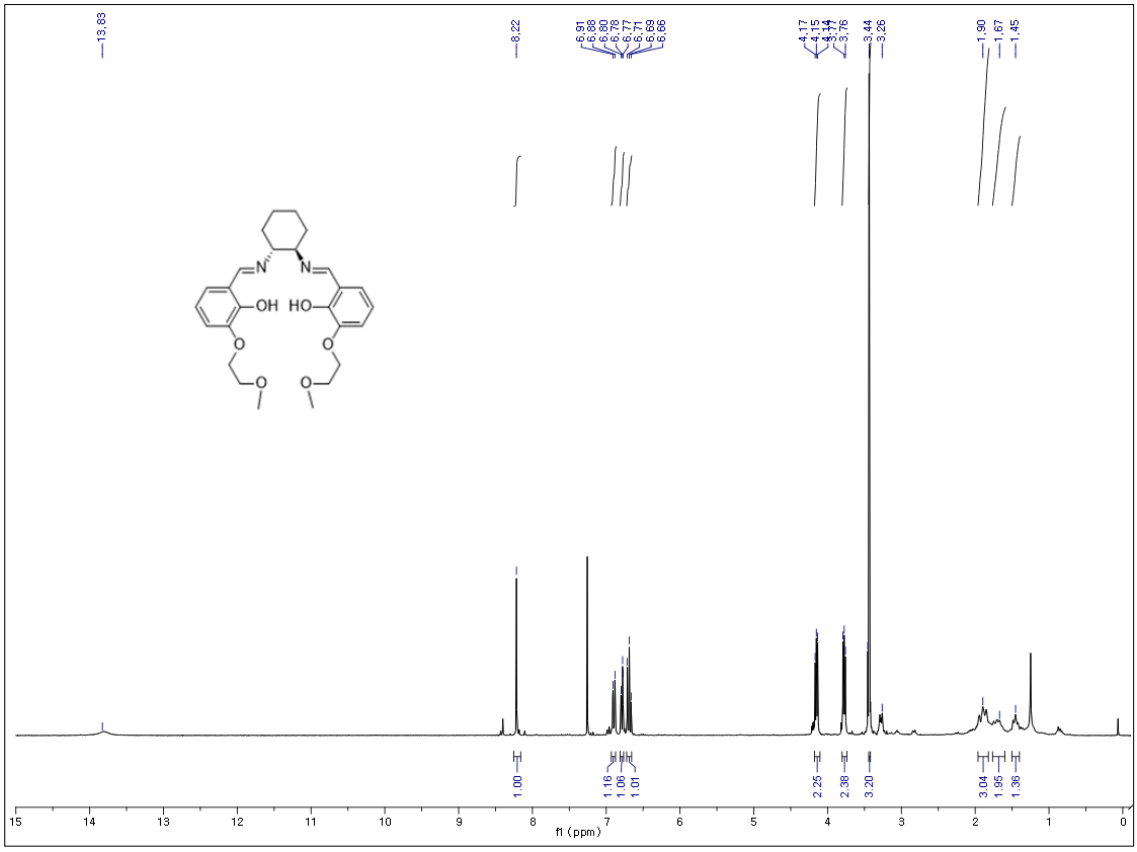
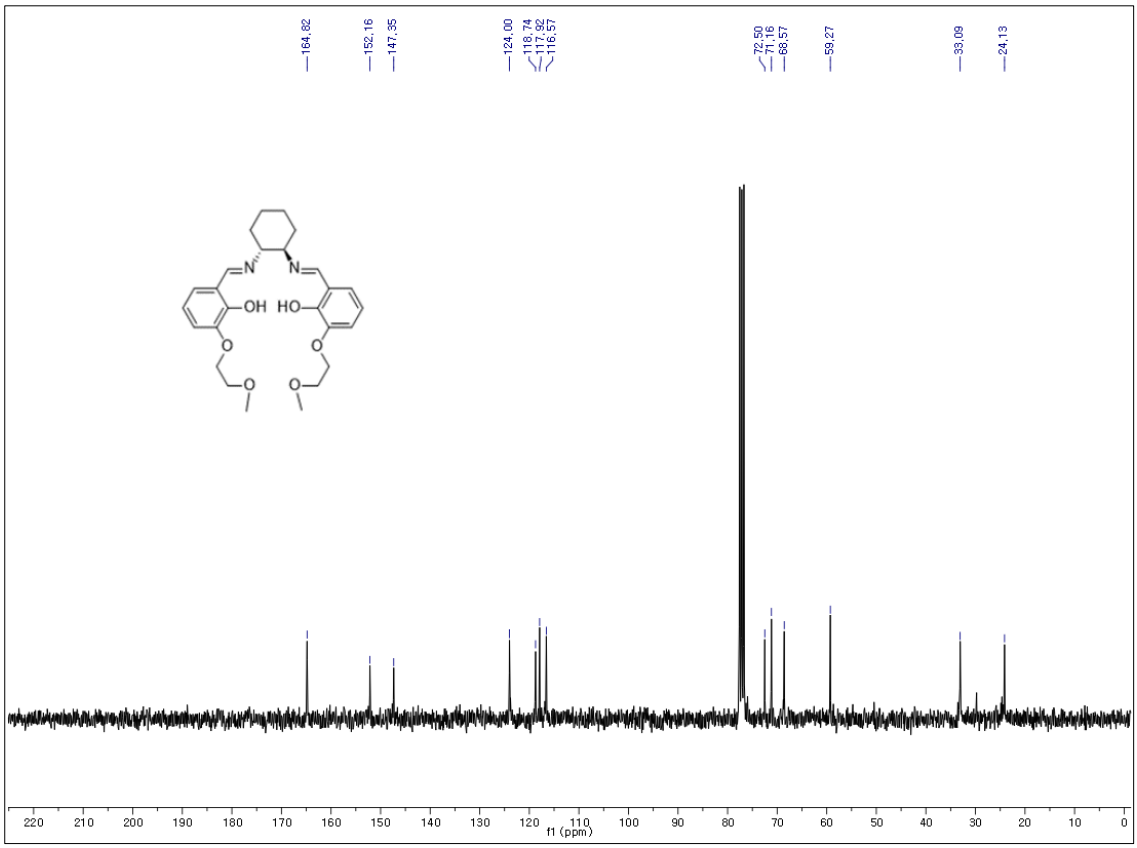
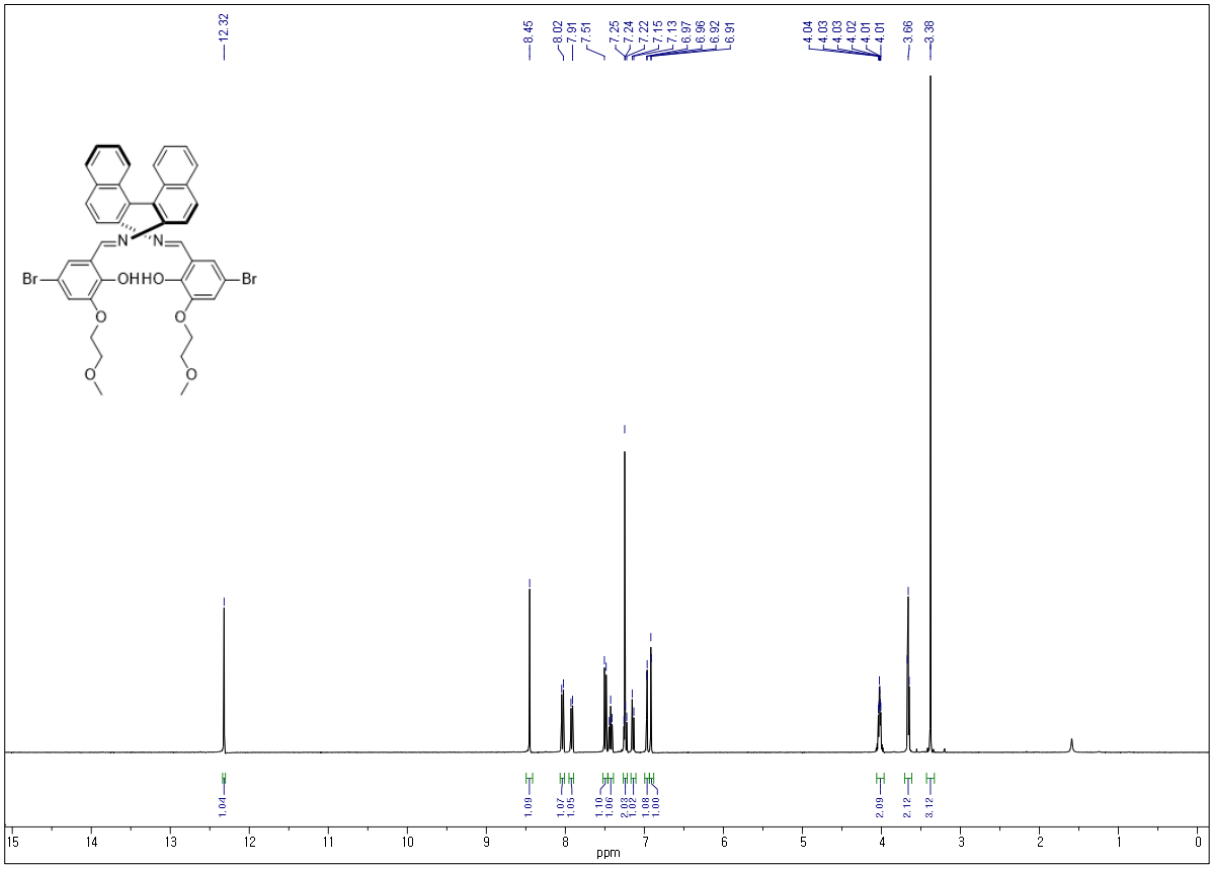
125.1
119.8
118.4
118.2
117.9
71.1
69.1
69.2
OHHO
ppm

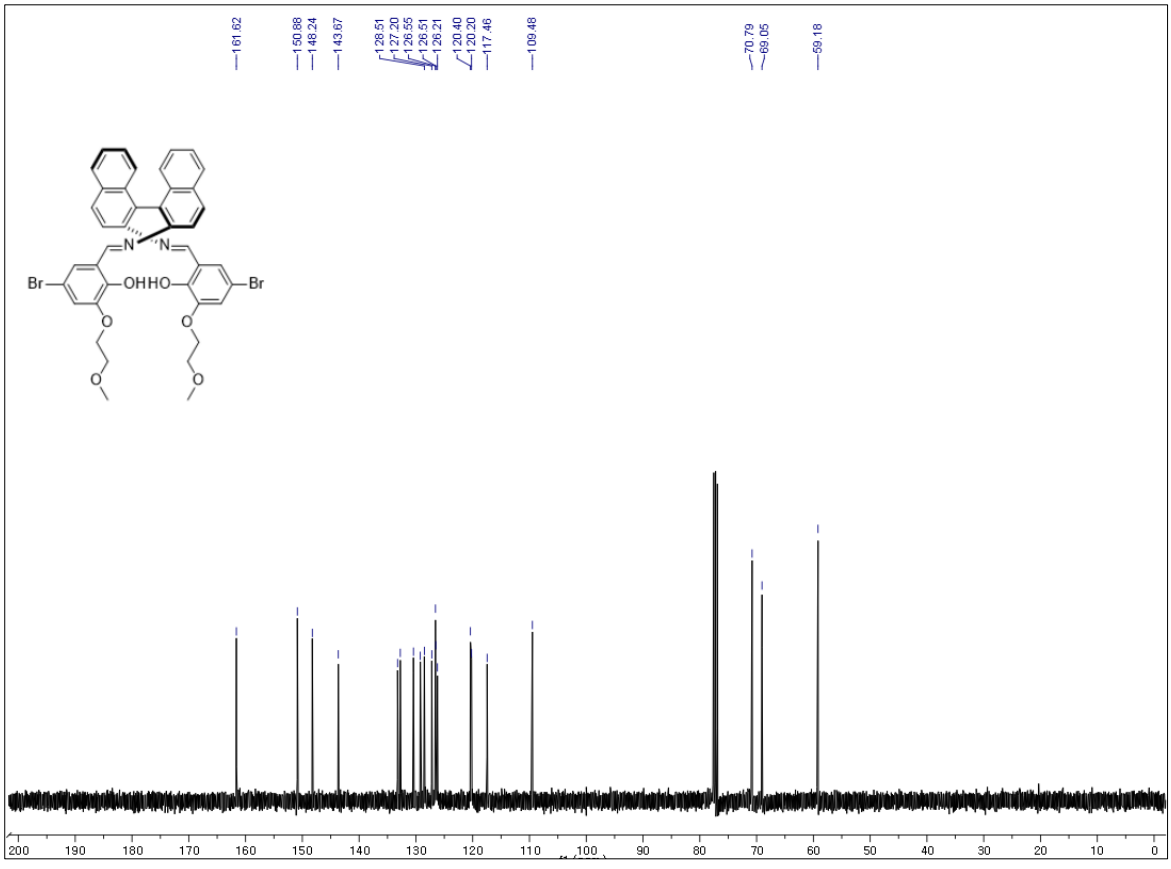

12.28
8.54
8.02
7.93
7.81
7.53
7.42
7.40
7.17
6.89
6.87
6.80
6.78
6.78
6.70
6.68
6.66
4.09
4.08
4.07
4.06
3.78
3.65
3.52
3.37
OHHO
f1 (ppm)

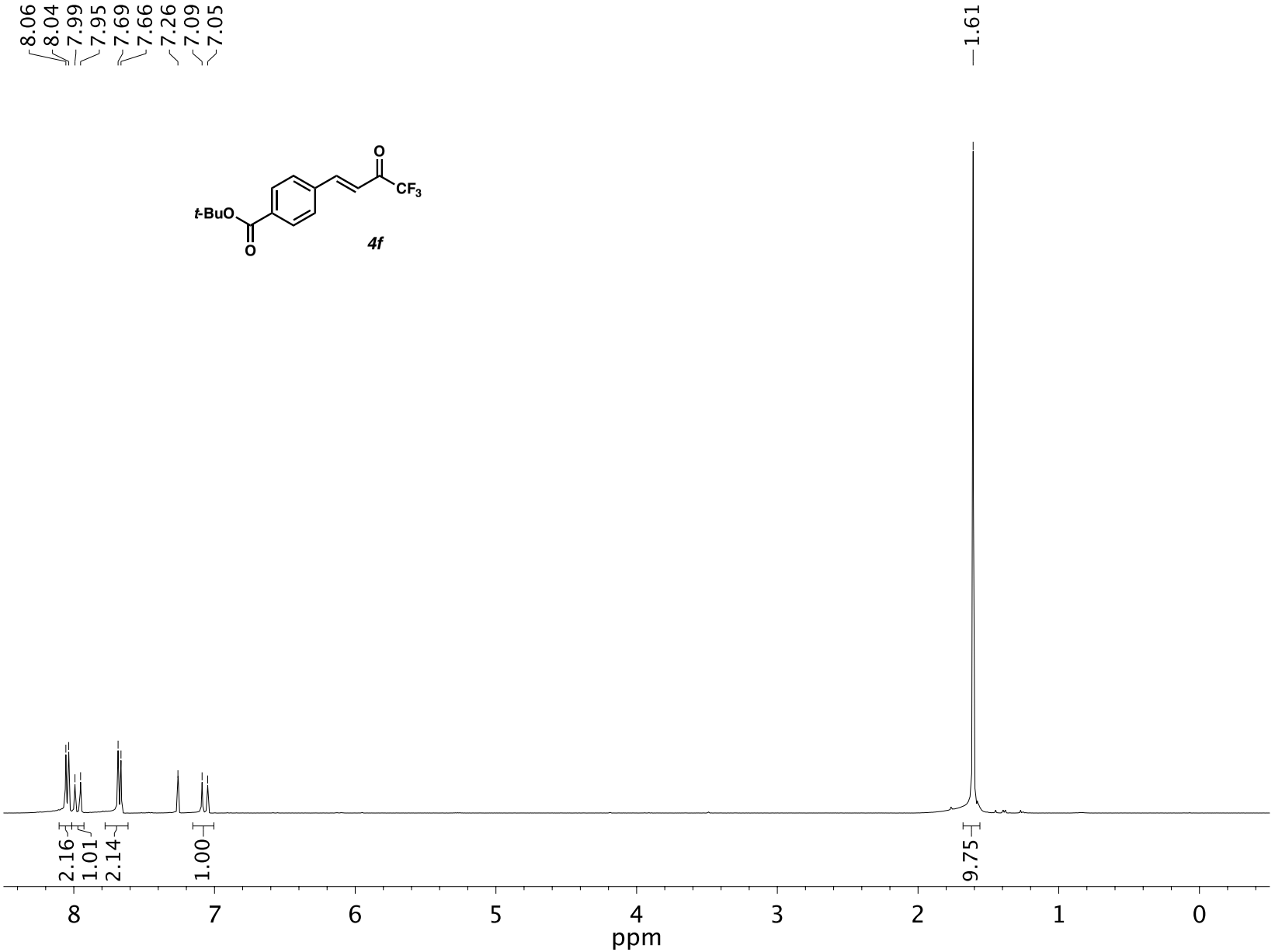

162.96
151.71
147.21
144.42
133.29
132.57
128.40
126.56
124.94
119.71
118.21
118.00
117.80
72.03
70.74
69.72
69.09
59.13
OHHO
ppm


13.83
8.22
6.91
6.88
6.80
6.78
6.77
6.71
6.69
6.66
4.17
4.15
4.14
3.77
3.76
3.44
3.26
1.90
1.67
1.45
OH
HO
1.00
1.16
1.06
1.01
2.25
2.38
3.20
3.04
1.95
1.36
f1 (ppm)

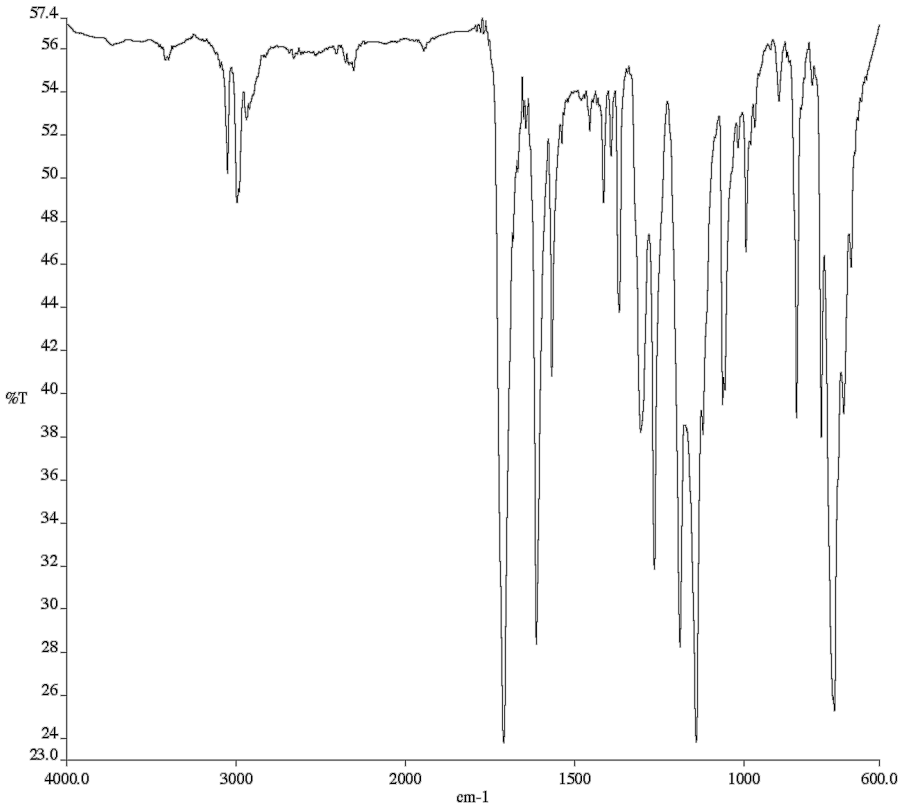

164.82
152.16
147.35
124.00
118.74
117.92
116.57
72.50
71.16
68.57
59.27
33.09
24.13
OH
HO
f1 (ppm)


12.32
8.45
8.02
7.91
7.51
7.25
7.24
7.22
7.15
7.13
6.97
6.96
6.92
6.91
4.04
4.03
4.03
4.02
4.01
4.01
3.66
3.38
Br
OHHO
Br
ppm

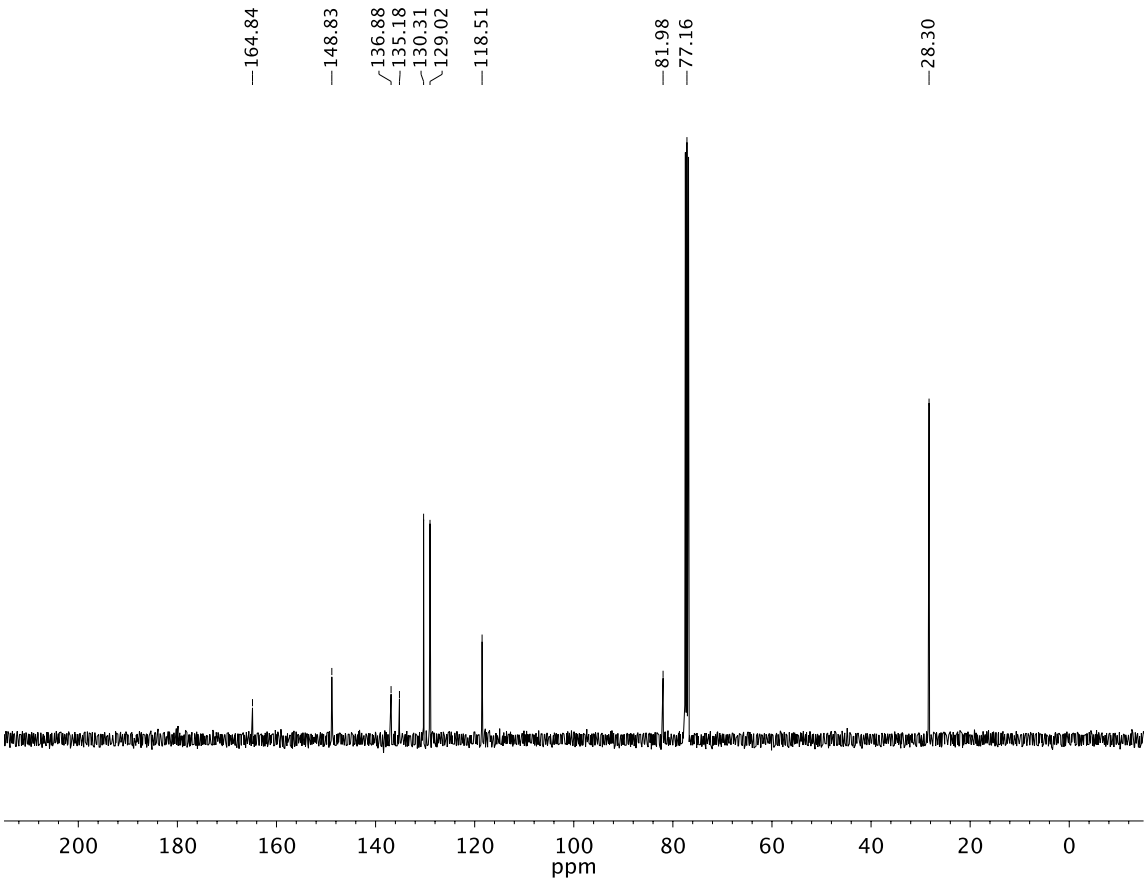

161.62
150.88
148.24
143.67
128.51
127.30
126.55
126.51
126.21
120.40
120.20
117.46
109.48
70.79
69.05
59.18
Br
OHHO
Br



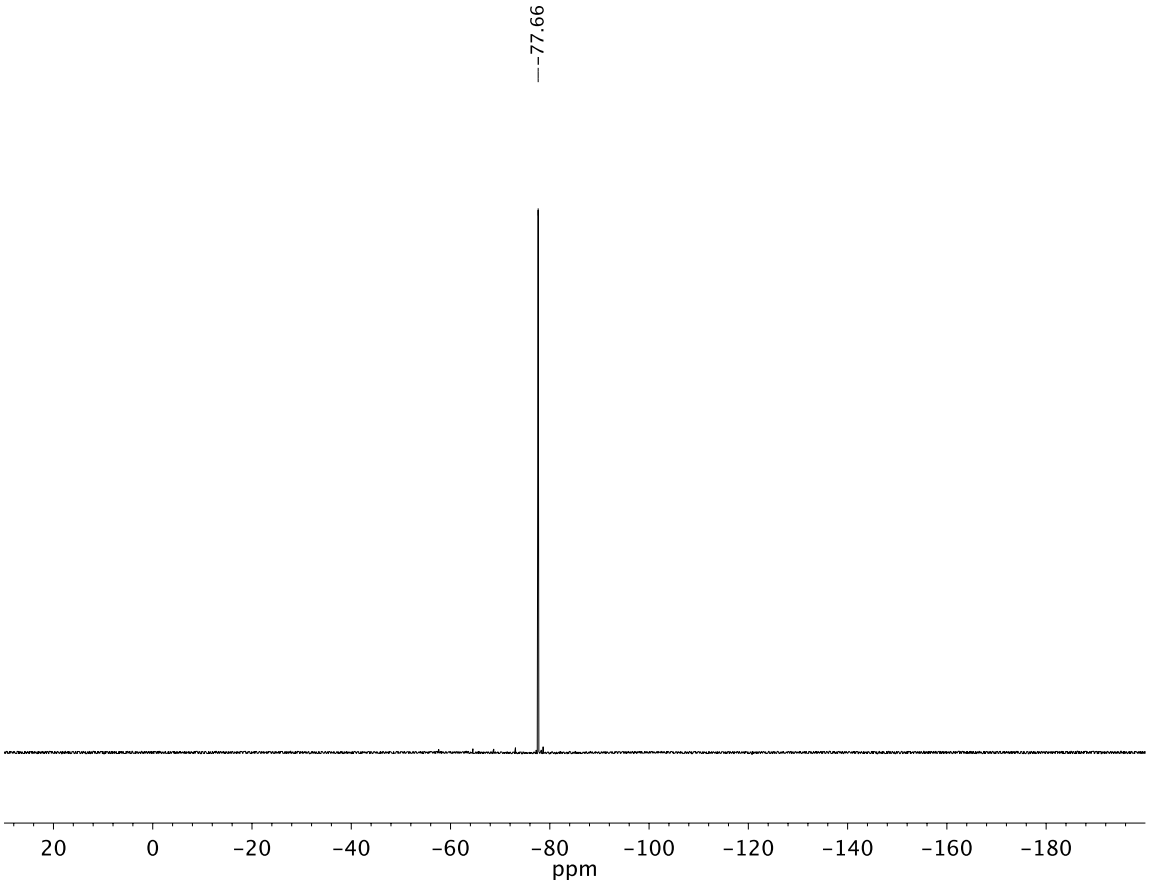
^{1}H NMR (400 MHz, $CDCl_3$) of compound **4f**



Infrared spectrum (Thin Film, NaCl) of compound **4f**.

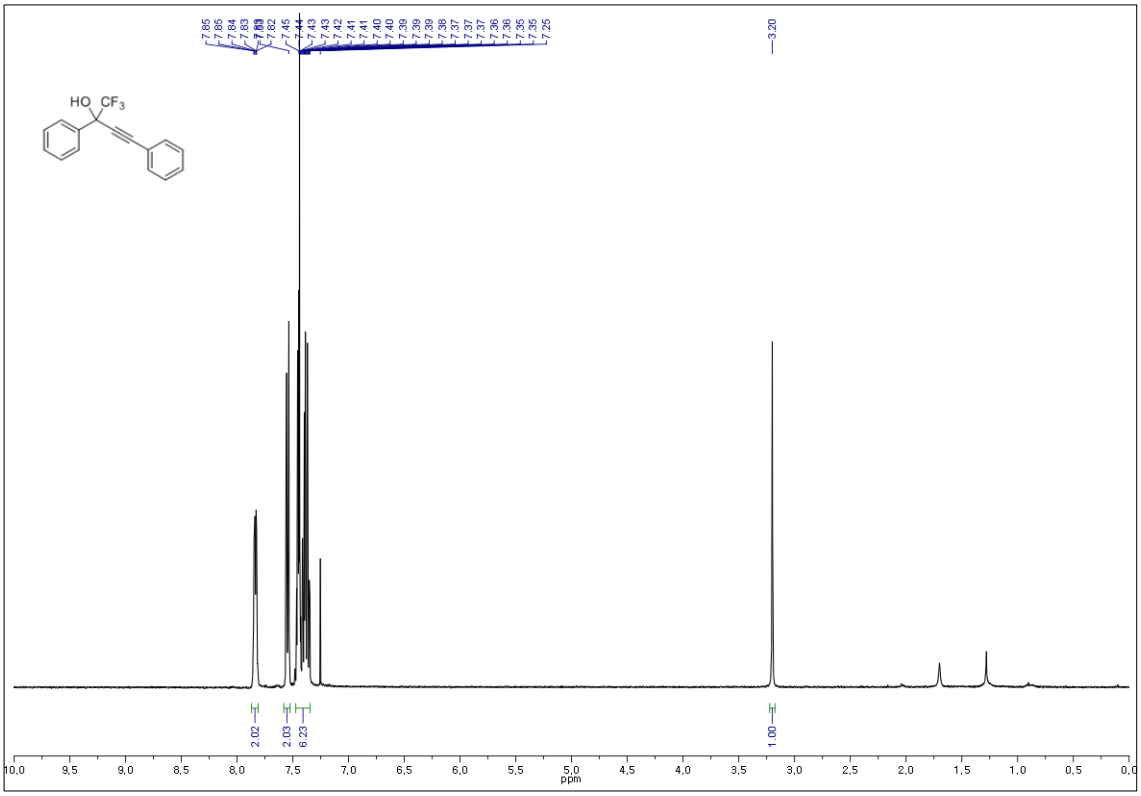


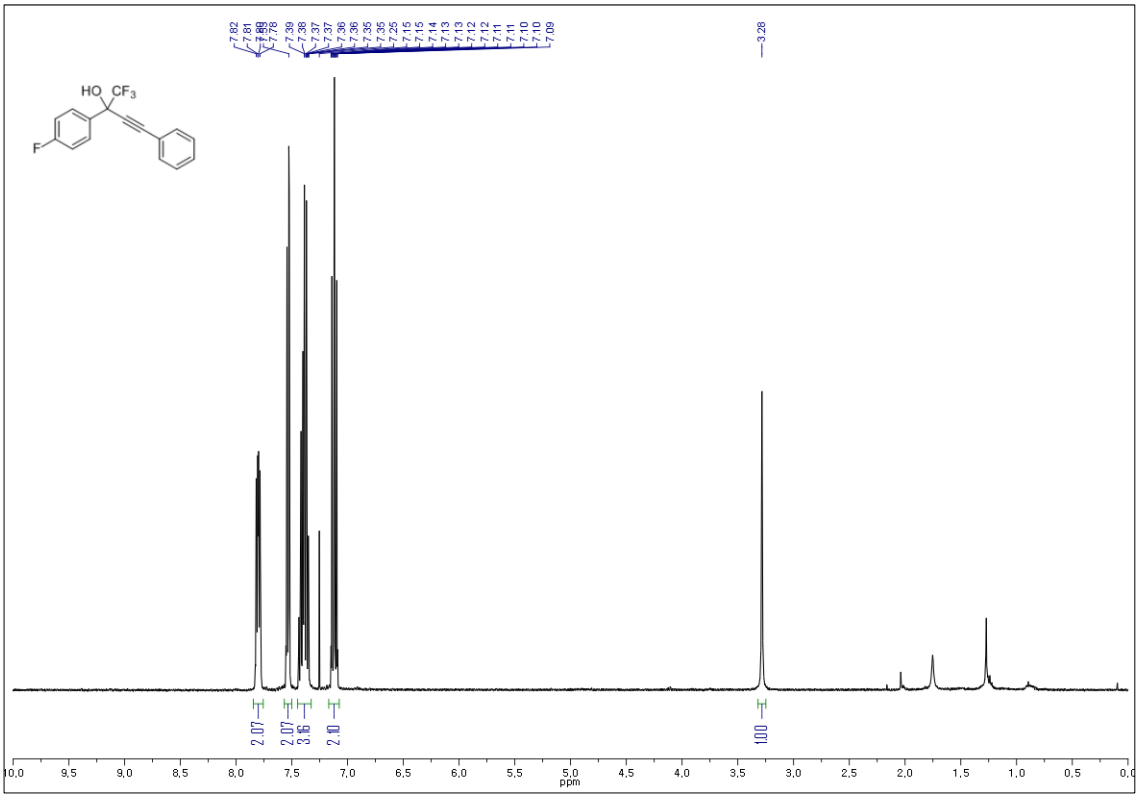
^{13}C NMR (101 MHz, $CDCl_3$) of compound **4f**

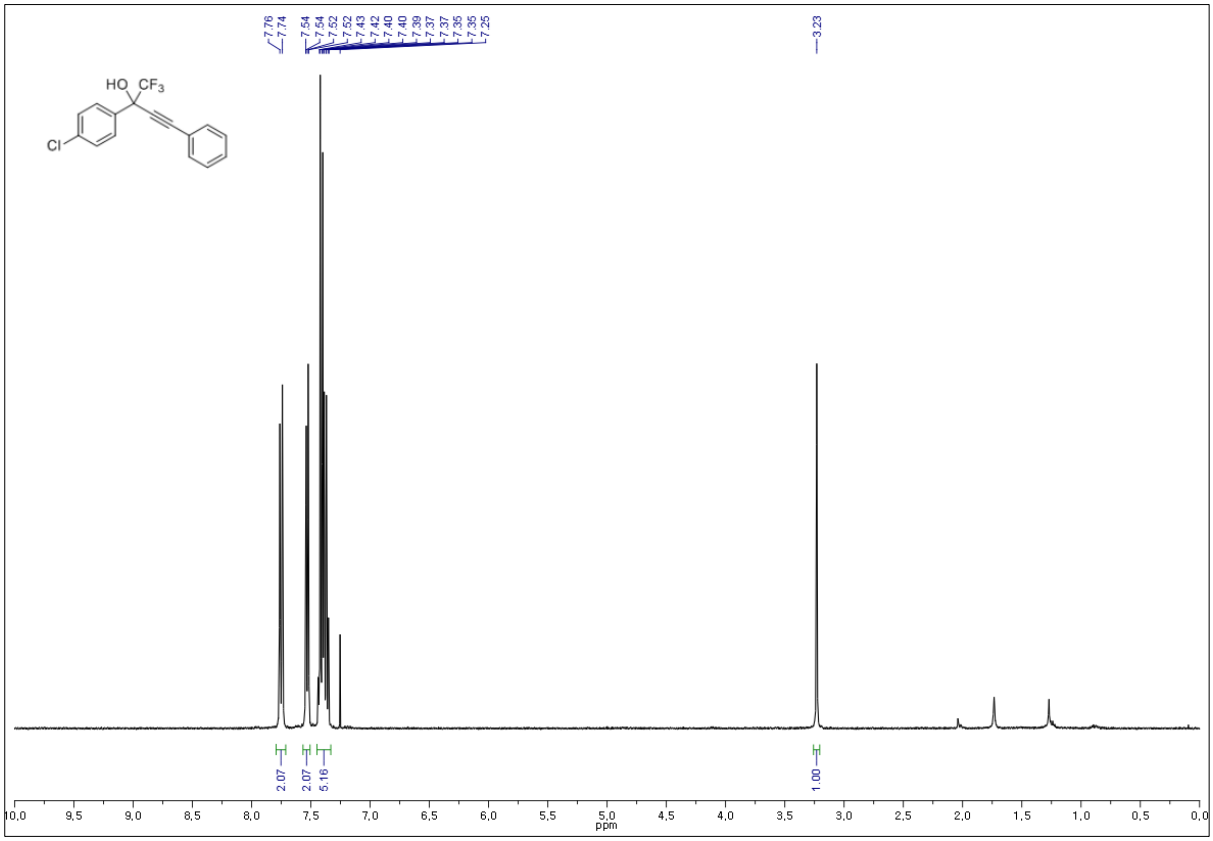


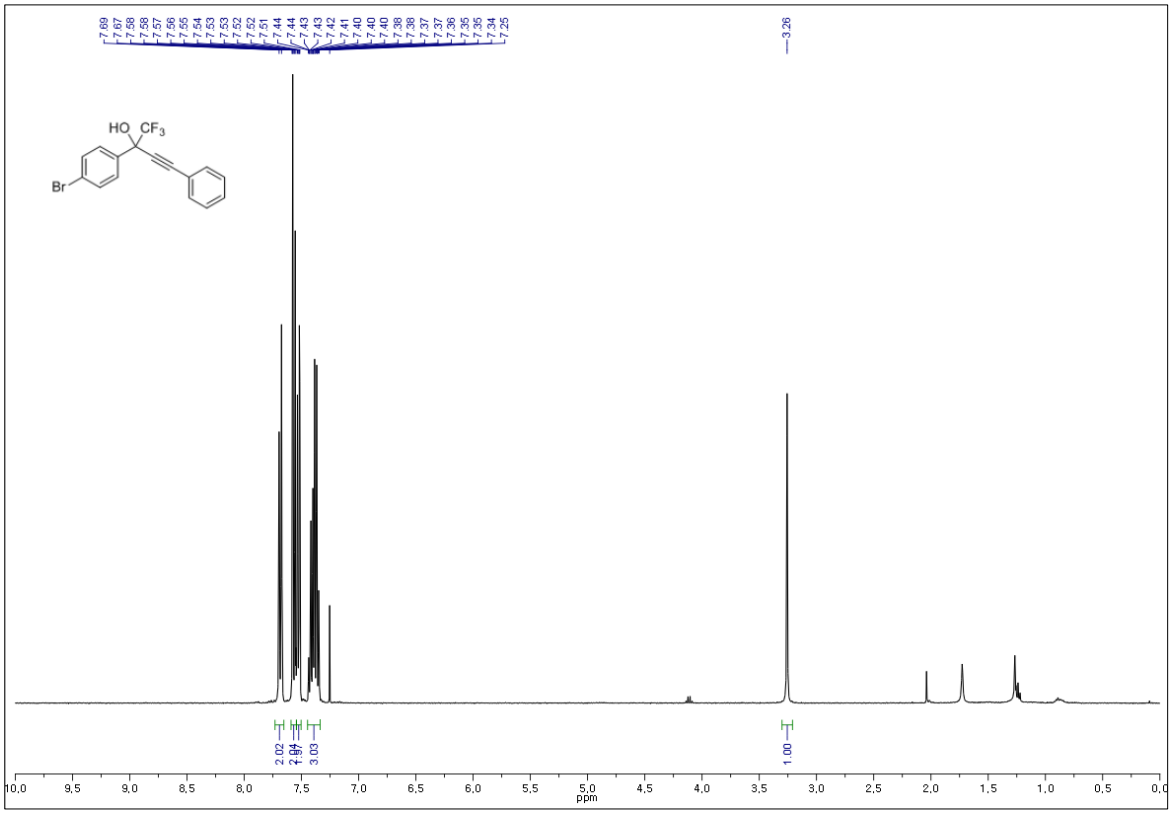
^{19}F NMR (282 MHz, $CDCl_3$) of compound **4f**

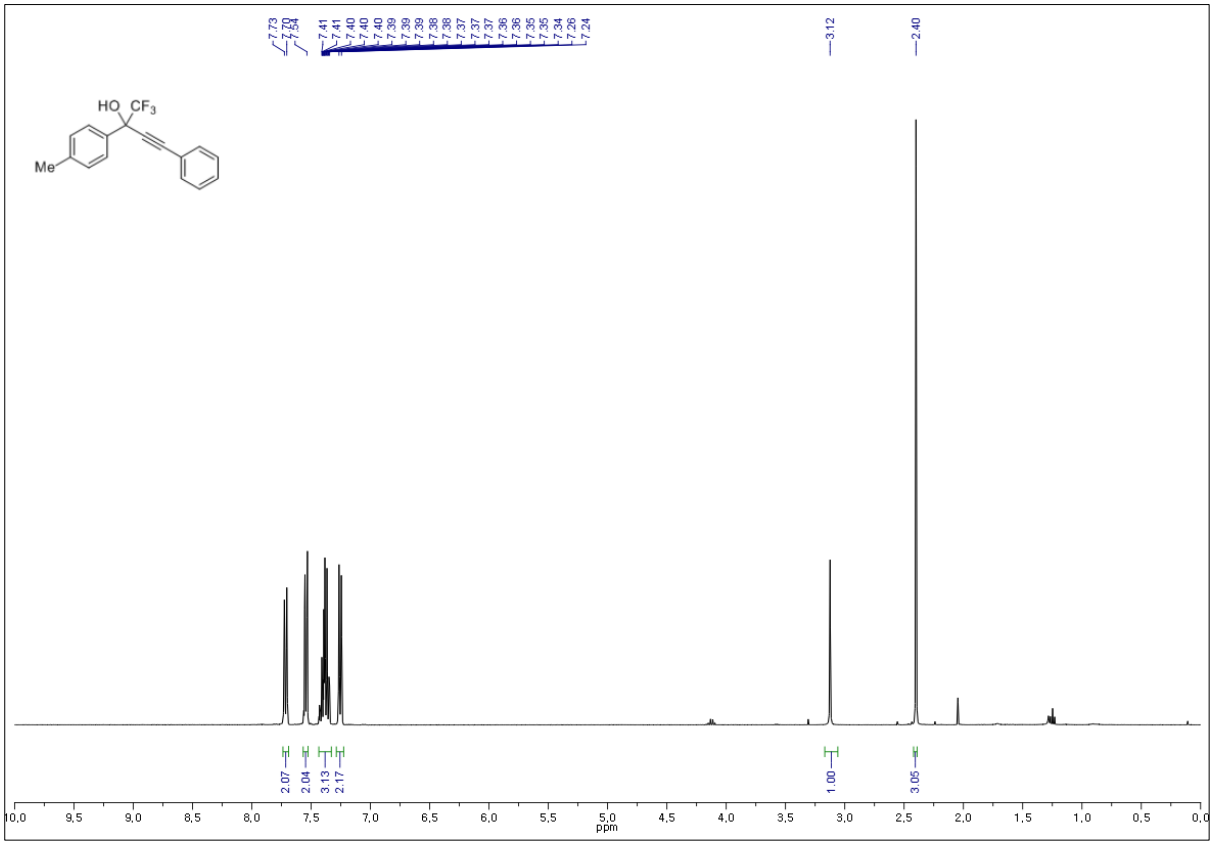
NMR and IR Data for Trifluoromethyl Products

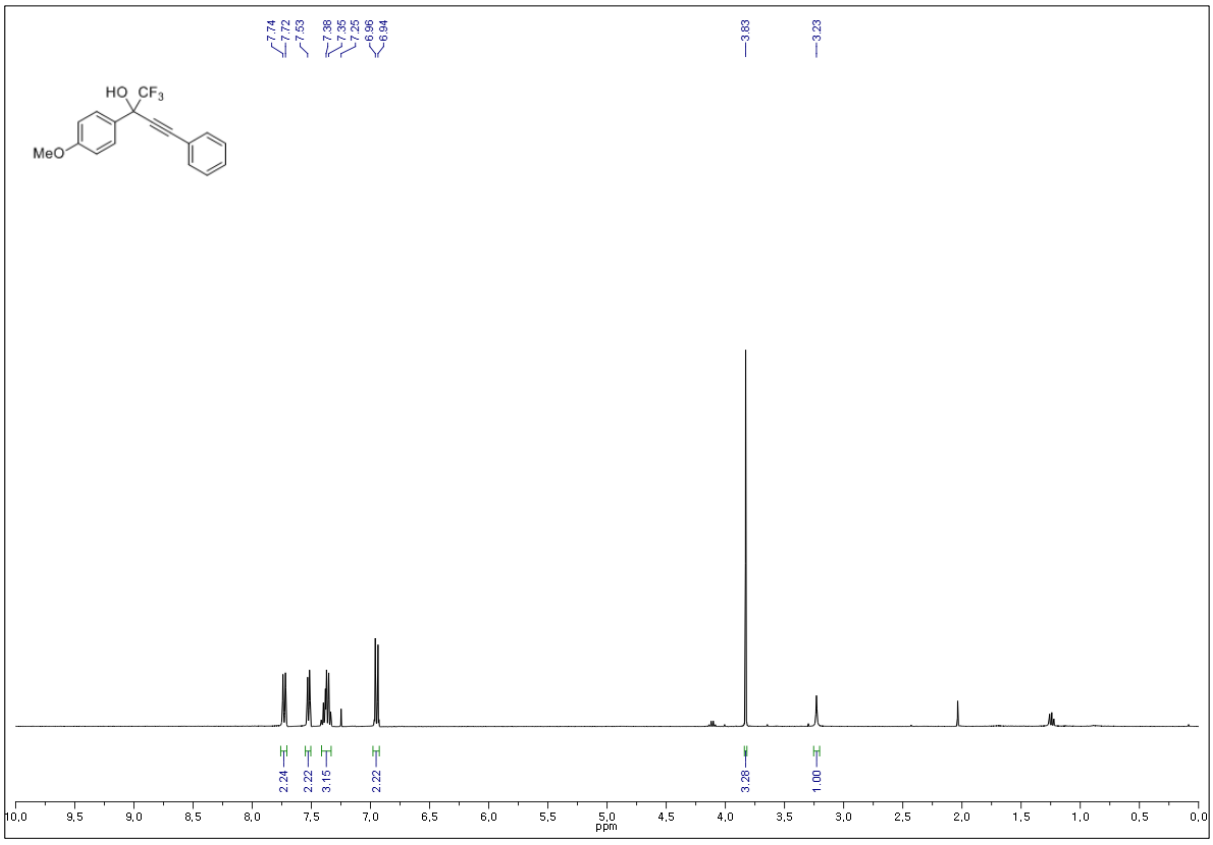


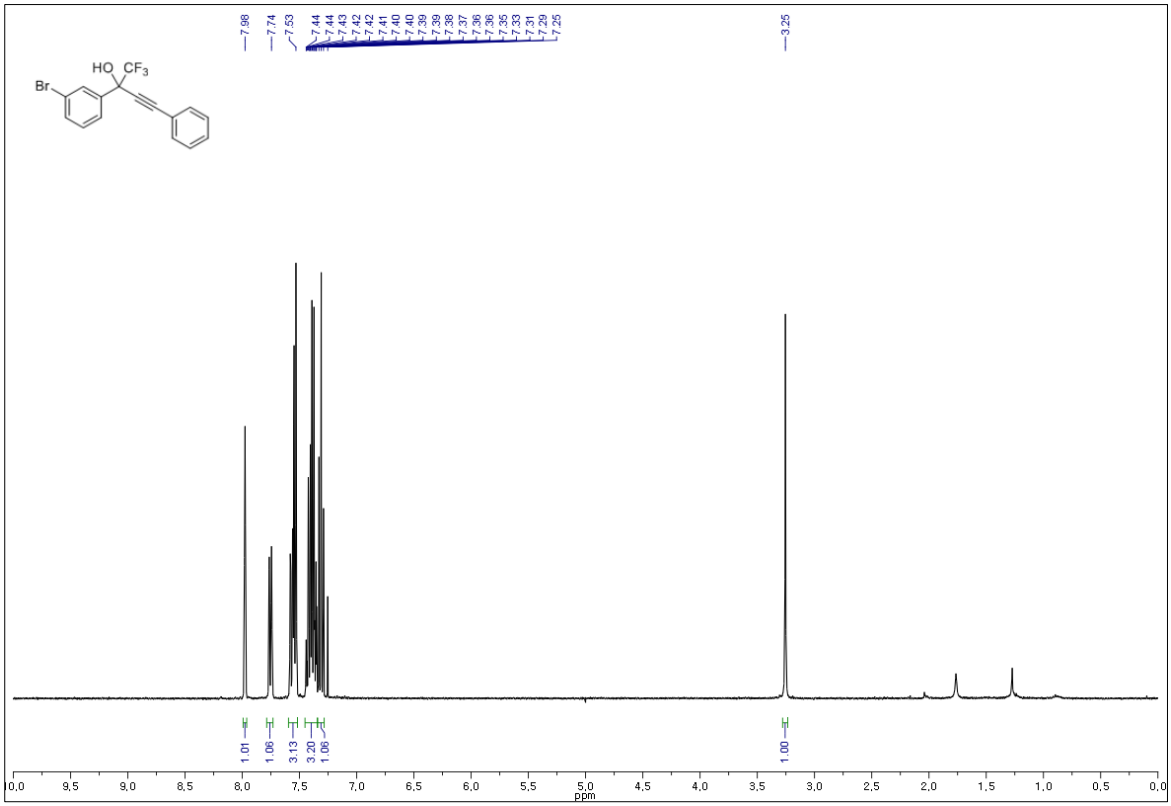


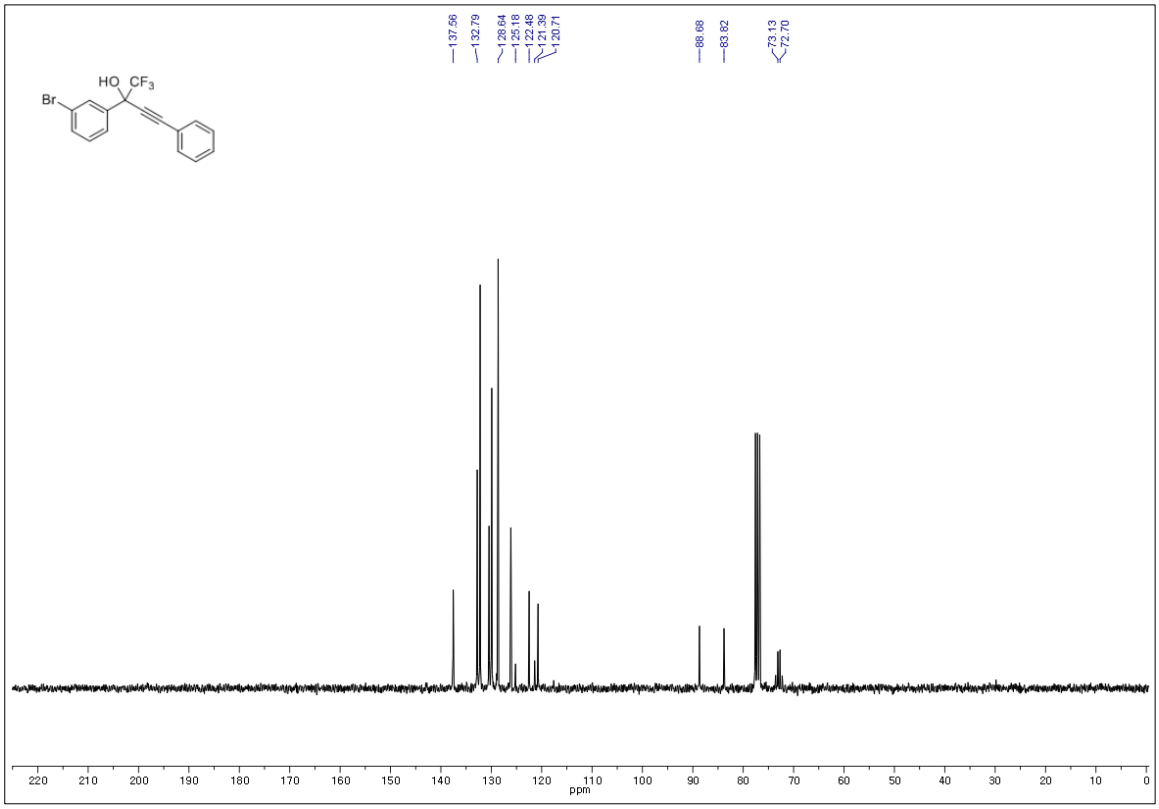

HO
CF3
Cl
7.76
7.74
7.54
7.54
7.52
7.52
7.43
7.42
7.40
7.40
7.39
7.37
7.37
7.35
7.35
7.25
3.23
2.07
2.07
5.16
1.00
ppm

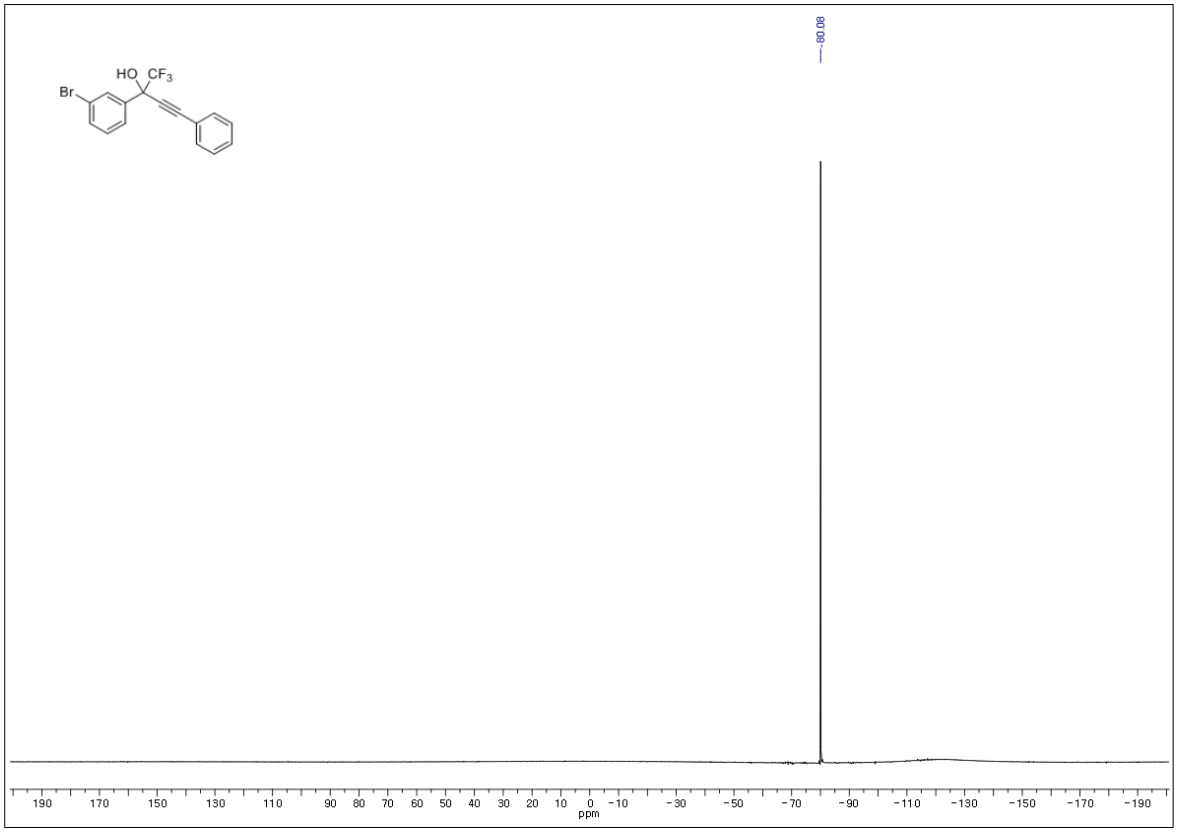

HO
CF3
Br
7.69
7.67
7.58
7.58
7.57
7.57
7.56
7.55
7.54
7.53
7.53
7.52
7.52
7.51
7.44
7.44
7.43
7.43
7.42
7.41
7.40
7.40
7.40
7.38
7.38
7.37
7.37
7.36
7.35
7.35
7.34
7.25
3.26
2.02
2.01
2.01
3.03
1.00
ppm

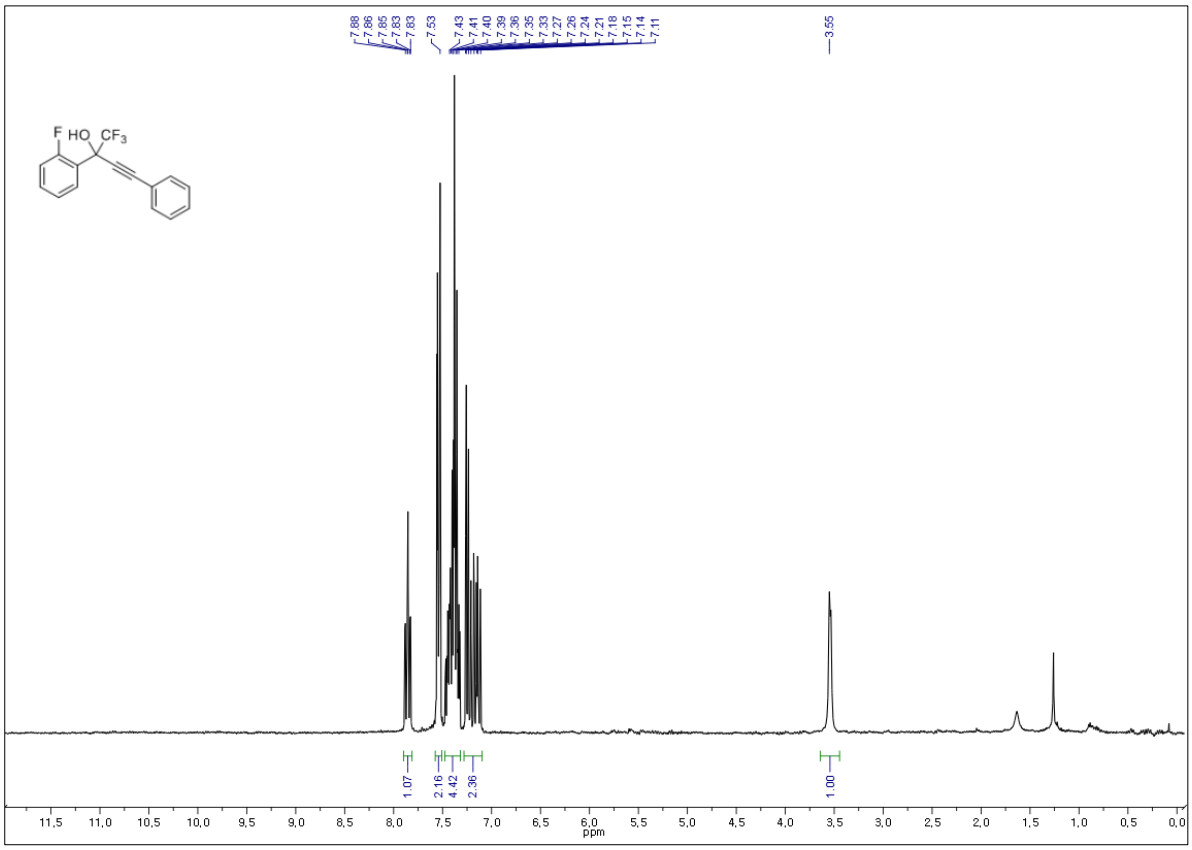

HO
CF3
Me
7.73
7.72
7.54
7.41
7.41
7.40
7.40
7.40
7.39
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.35
7.35
7.34
7.26
7.24
3.12
2.40
2.07
2.04
3.13
2.17
1.00
3.05
ppm

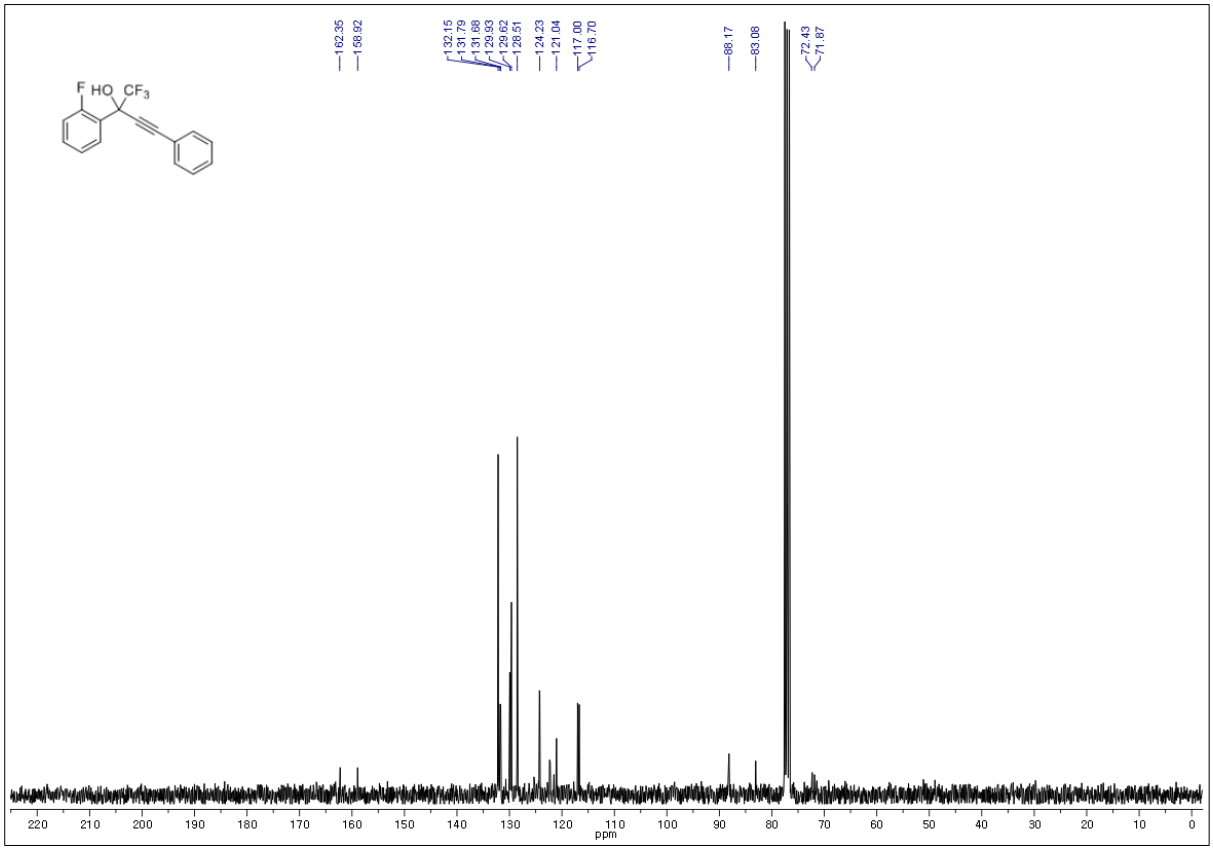

HO
CF3
MeO
7.74
7.72
7.53
7.38
7.35
7.25
6.96
6.94
3.83
3.23
2.24
2.22
3.15
2.22
3.28
1.00
ppm

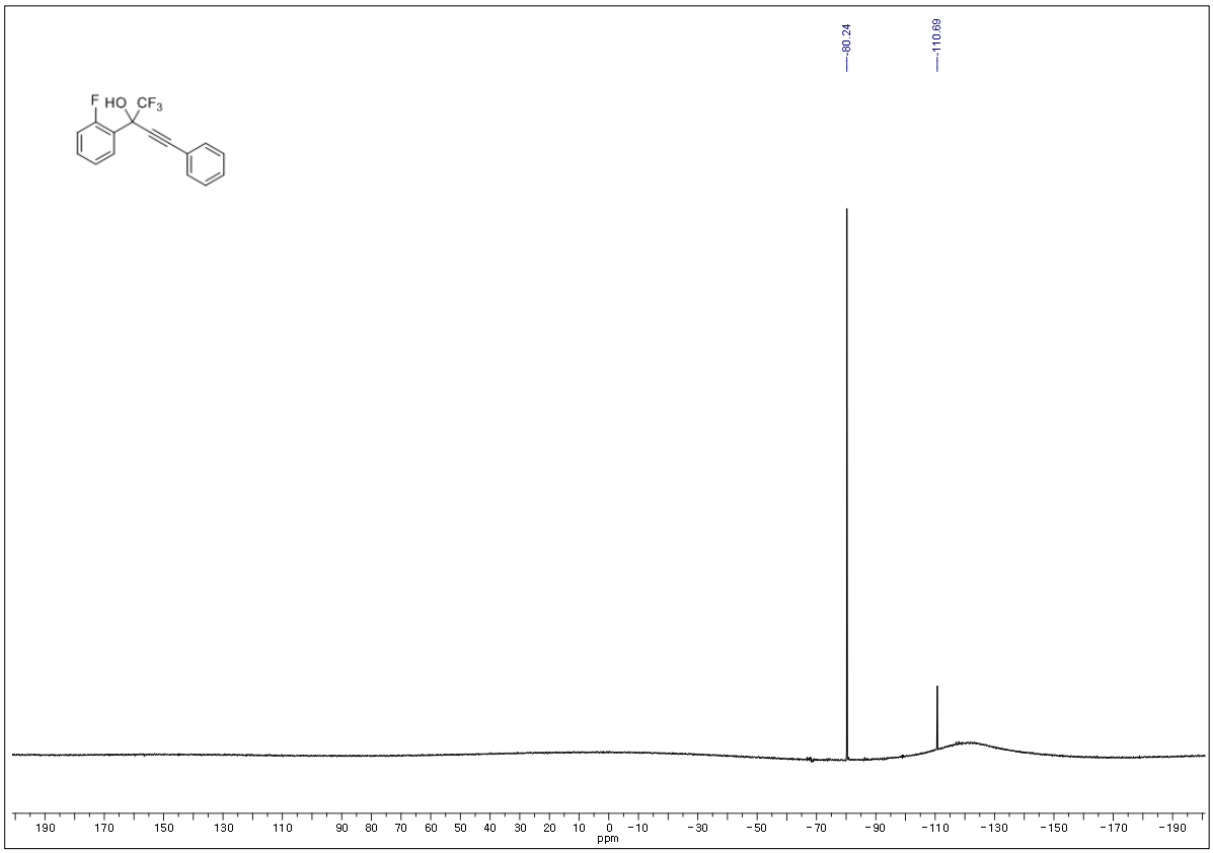

7.98
7.74
7.53
7.44
7.44
7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.39
7.38
7.37
7.36
7.35
7.33
7.31
7.29
7.25
3.25
HO
CF3
Br
1.01
1.06
3.13
3.20
1.06
1.00
ppm

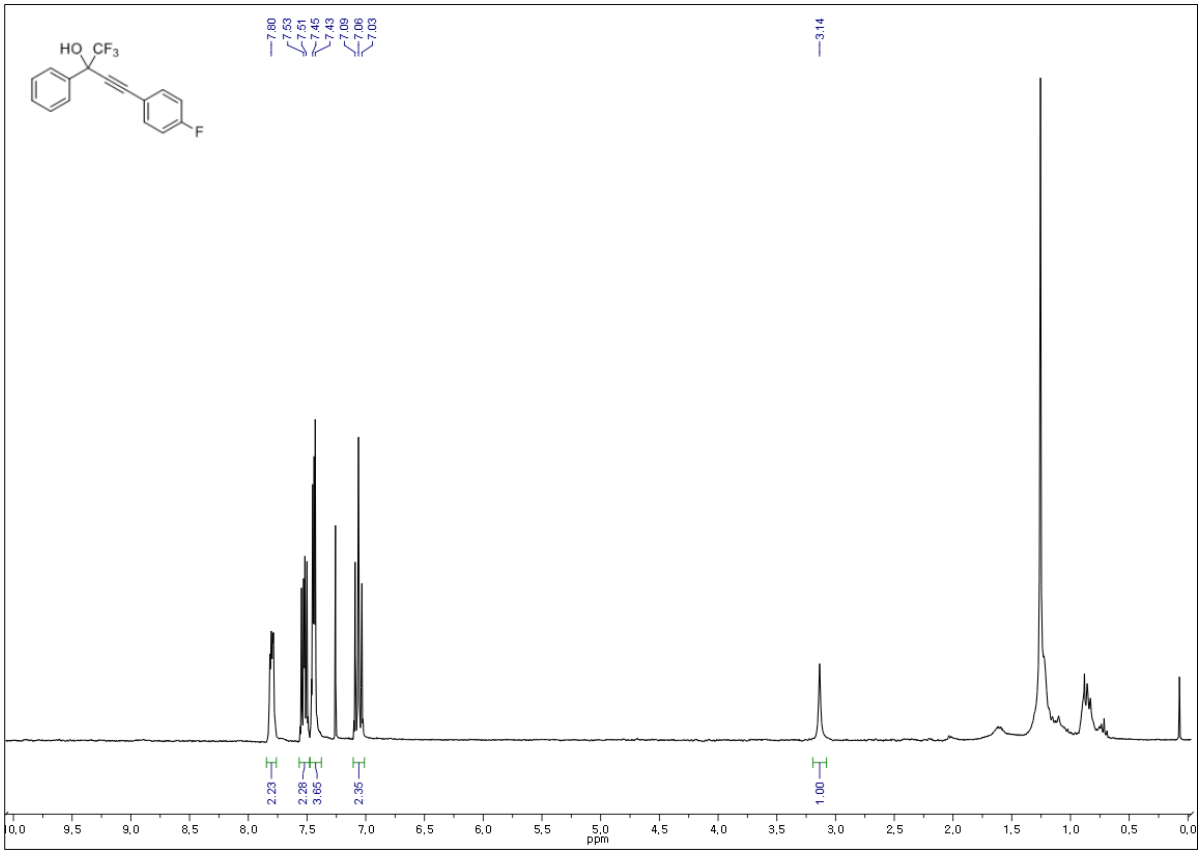

137.56
132.79
128.64
125.18
122.48
121.39
120.71
88.68
83.82
73.13
72.70
HO
CF3
Br
ppm

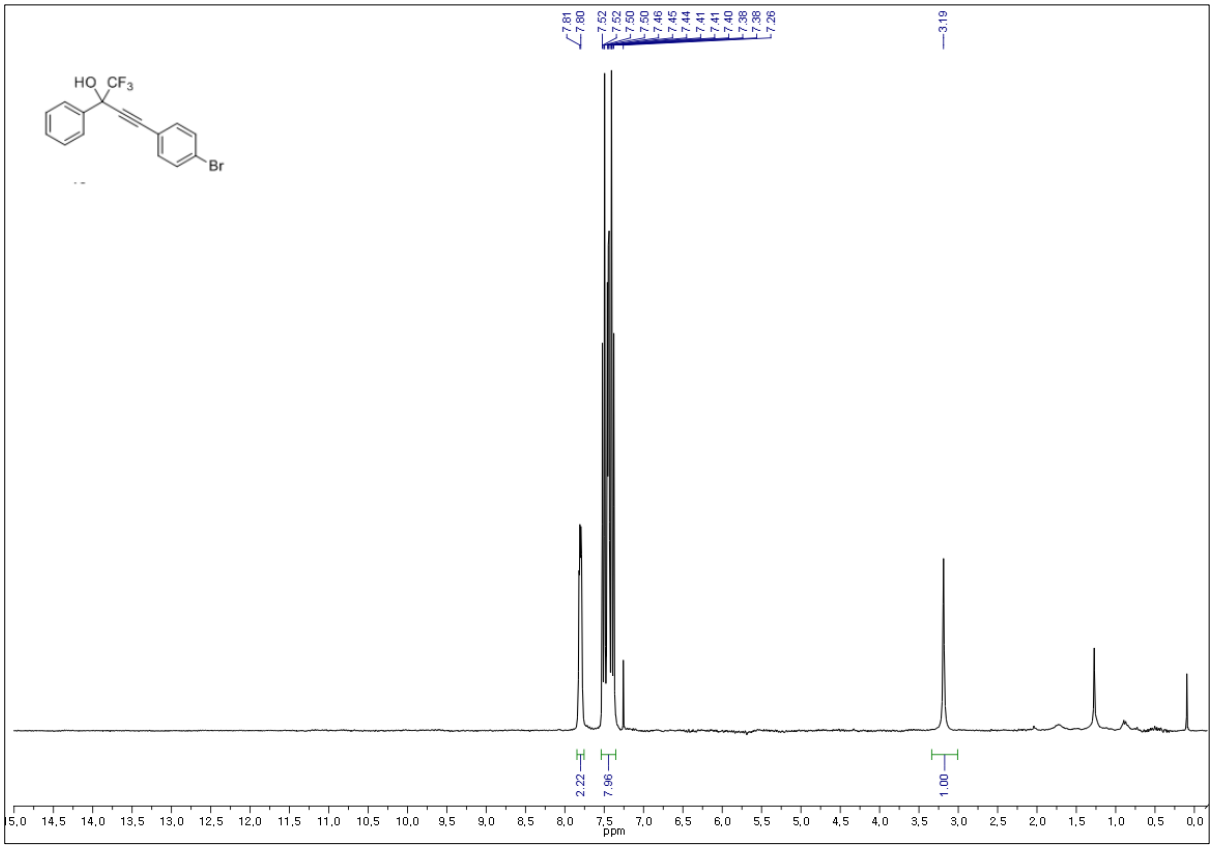

HO CF3
Br
-80.08
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190
ppm

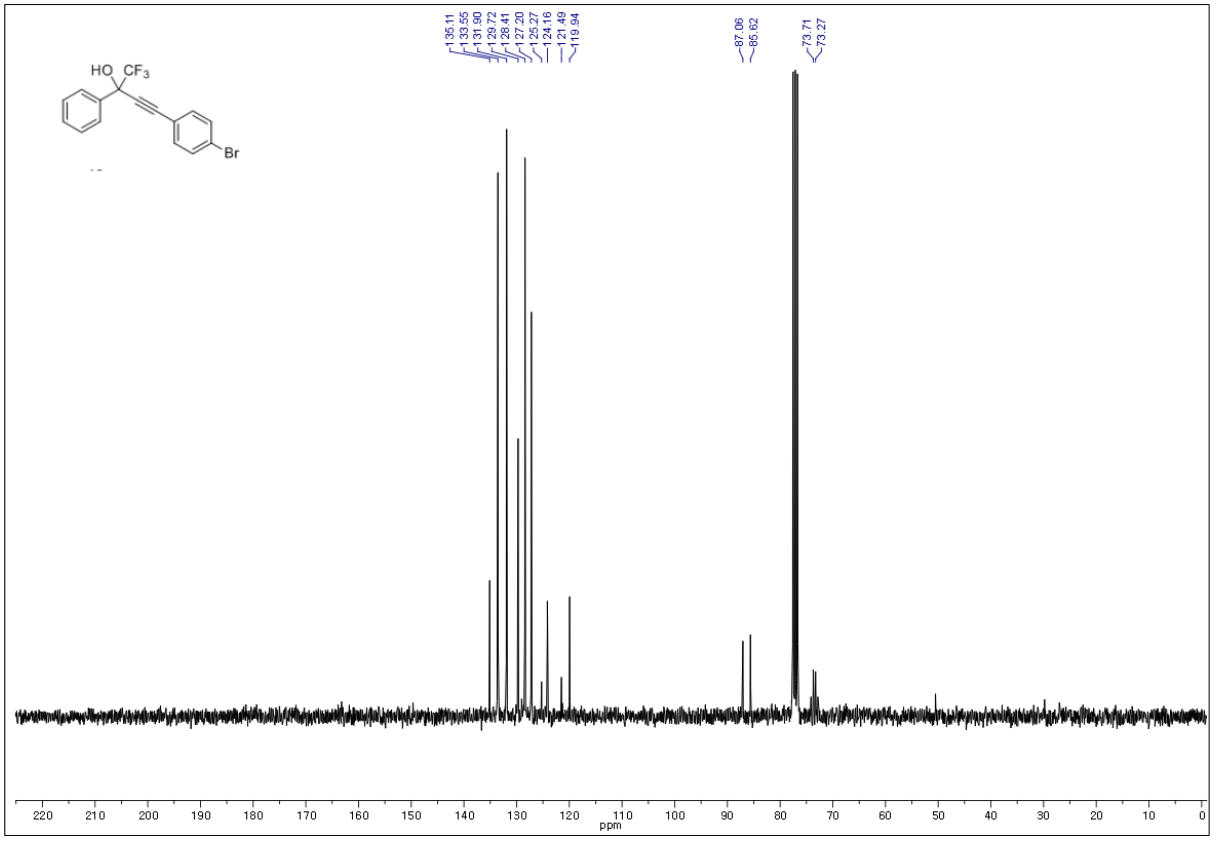

7.88
7.86
7.85
7.83
7.83
7.53
7.43
7.41
7.40
7.39
7.36
7.35
7.33
7.27
7.26
7.24
7.21
7.18
7.15
7.14
7.11
3.55
F HO CF3
1.07
2.16
4.42
2.36
1.00
11,5 11,0 10,5 10,0 9,5 9,0 8,5 8,0 7,5 7,0 6,5 6,0 5,5 5,0 4,5 4,0 3,5 3,0 2,5 2,0 1,5 1,0 0,5 0,0
ppm

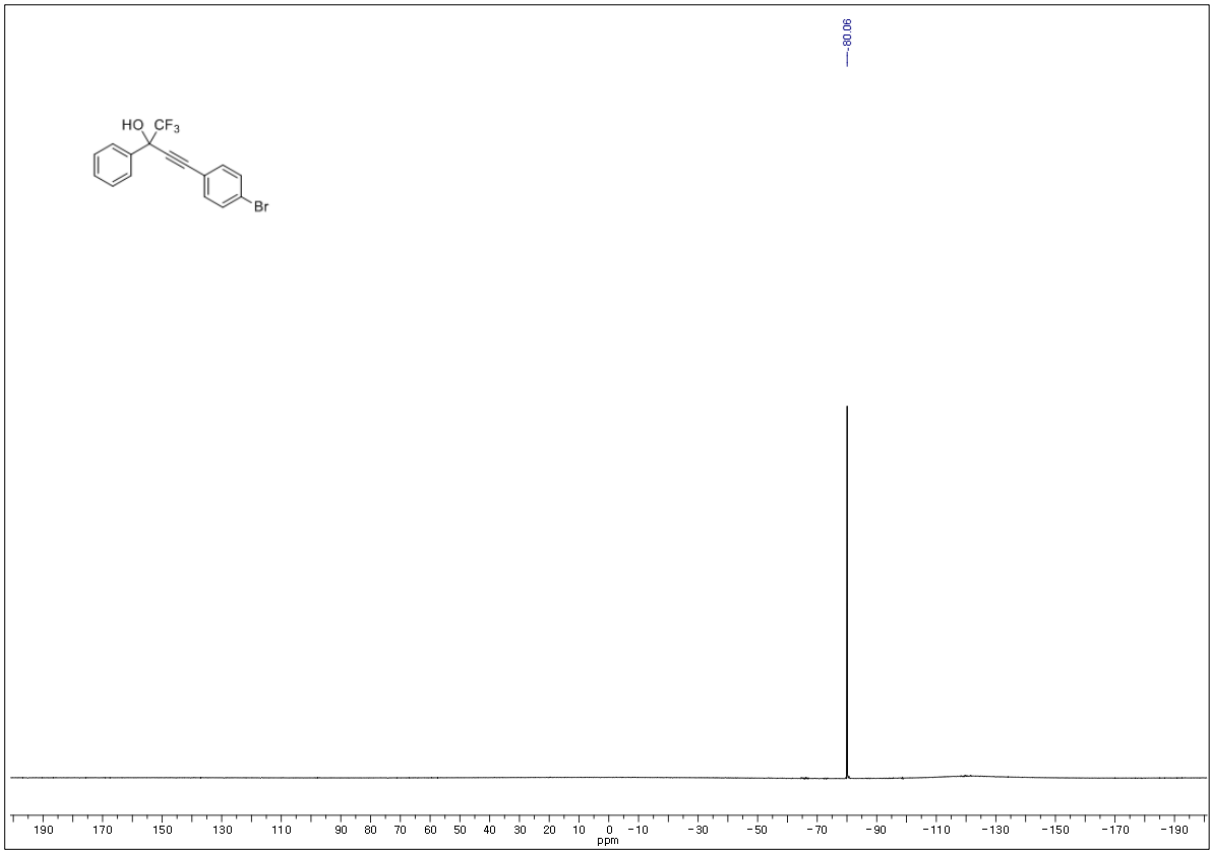

F HO CF3
162.35
158.92
132.15
131.79
131.68
129.93
129.62
128.51
124.23
121.04
117.00
116.70
88.17
83.08
72.43
71.87
ppm

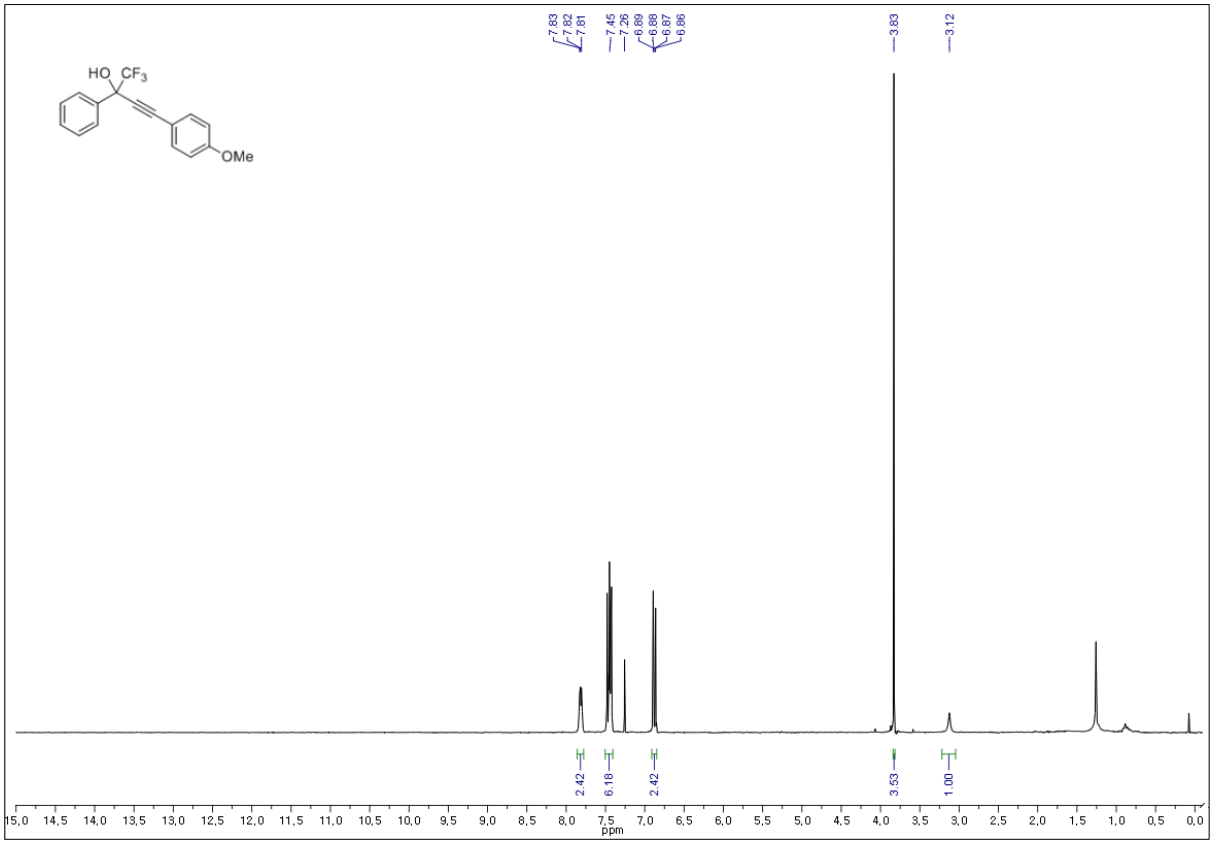

F HO CF3
-80.24
-110.69
ppm

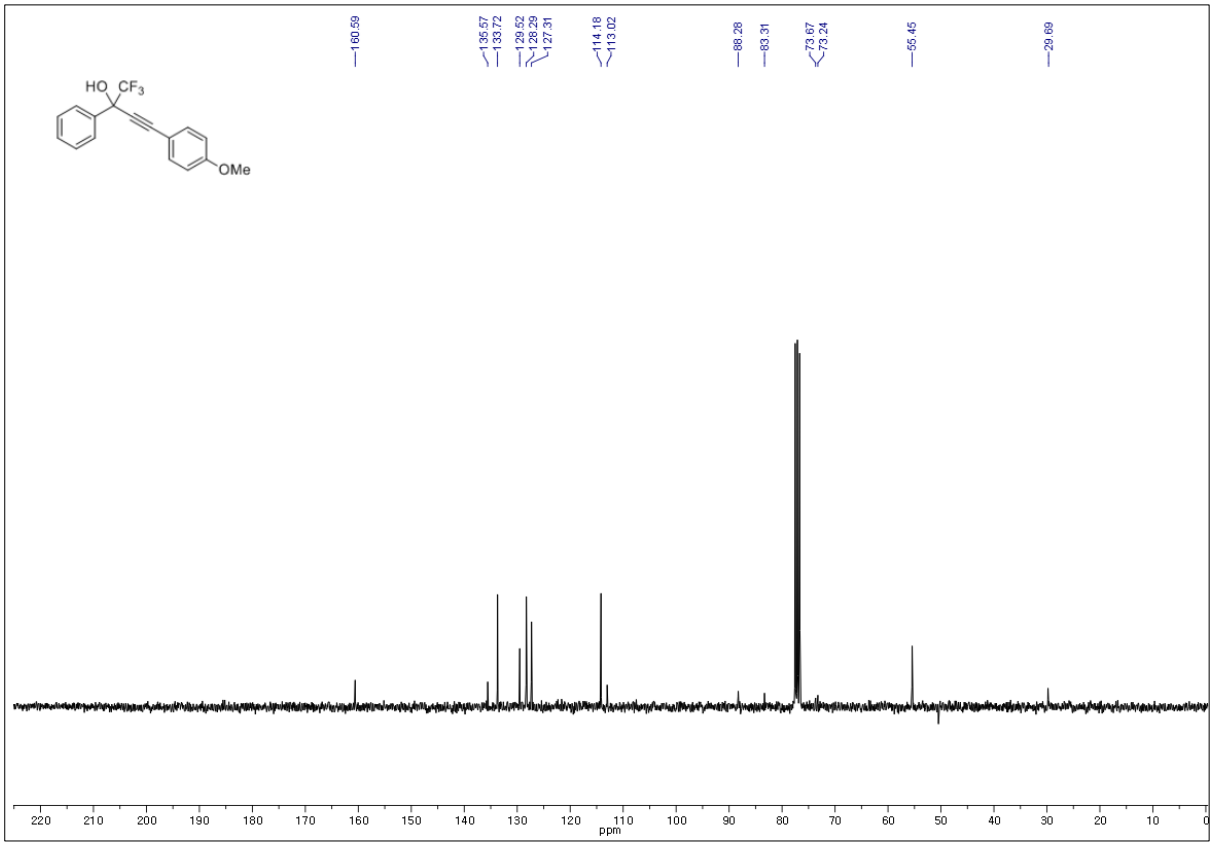

HO
CF3
F
7.80
7.53
7.51
7.45
7.43
7.09
7.06
7.03
3.14
2.23
2.28
3.65
2.35
1.00
ppm

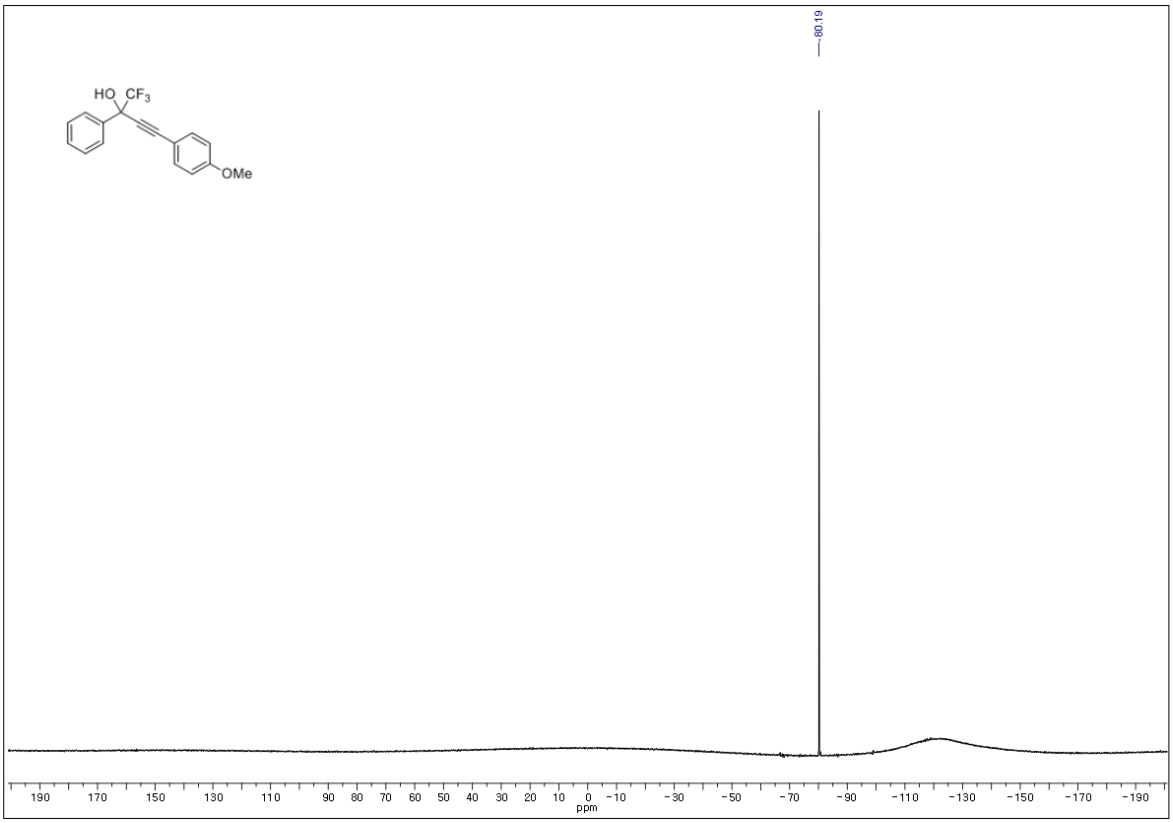

HO
CF3
Br
7.81
7.80
7.52
7.52
7.50
7.50
7.46
7.45
7.44
7.41
7.41
7.40
7.38
7.38
7.26
3.19
2.22
7.96
1.00
ppm

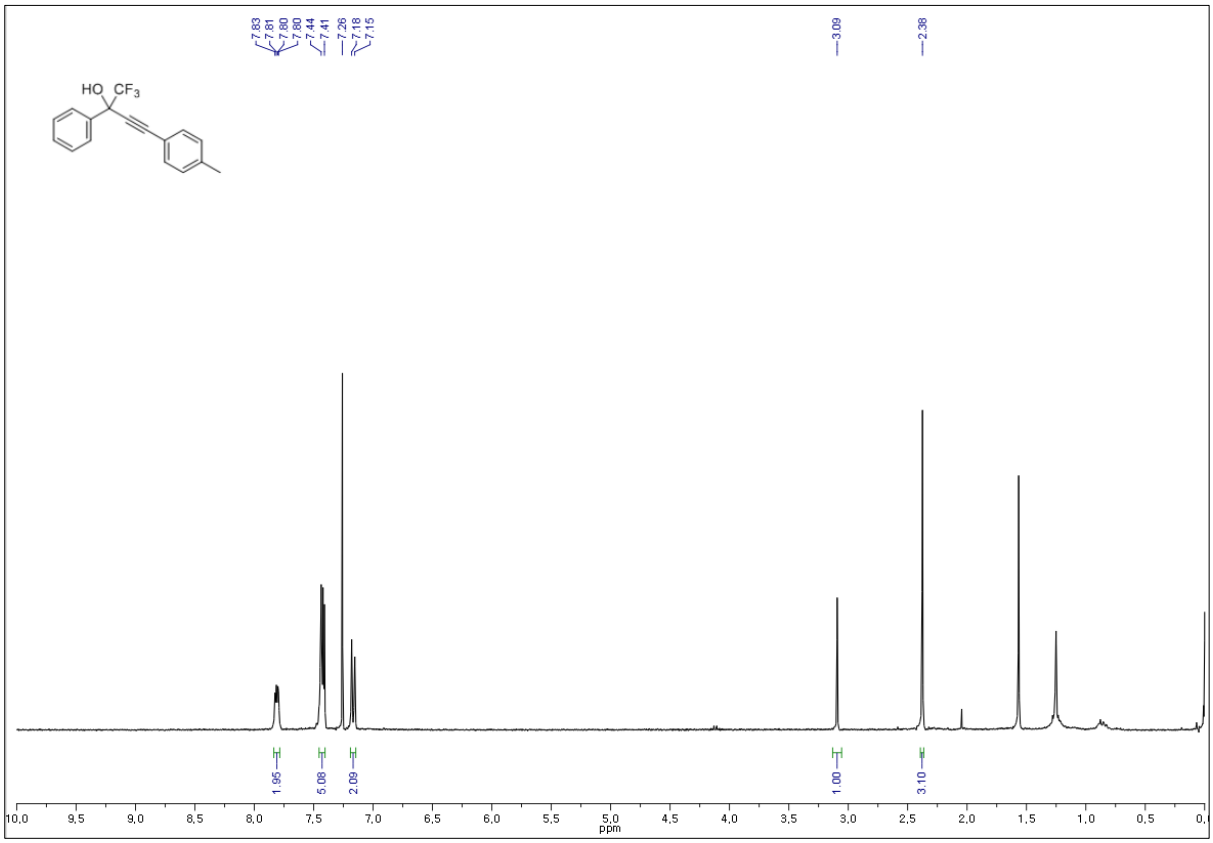

135.11
133.55
131.90
129.72
128.41
127.20
125.27
124.16
121.49
119.94
87.06
85.62
73.71
73.27
HO
CF3
Br
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

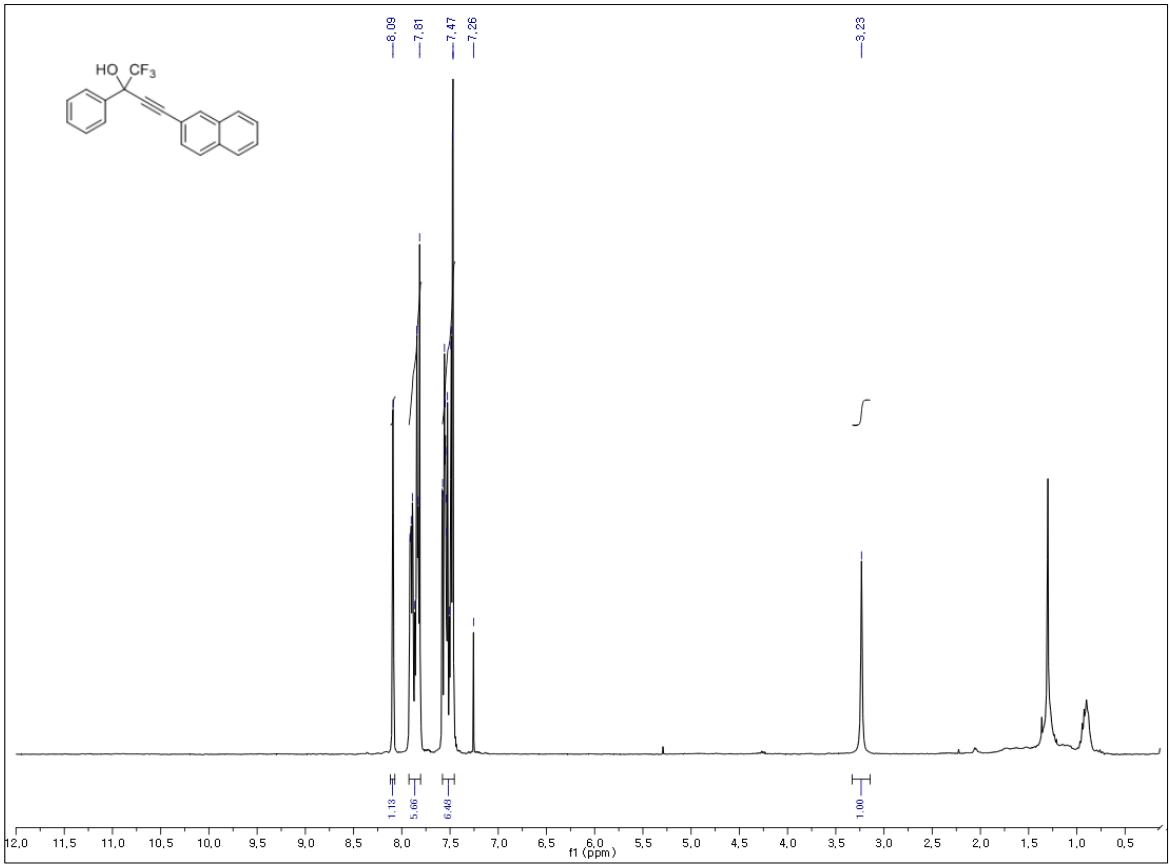

80.06
HO
CF3
Br
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
ppm

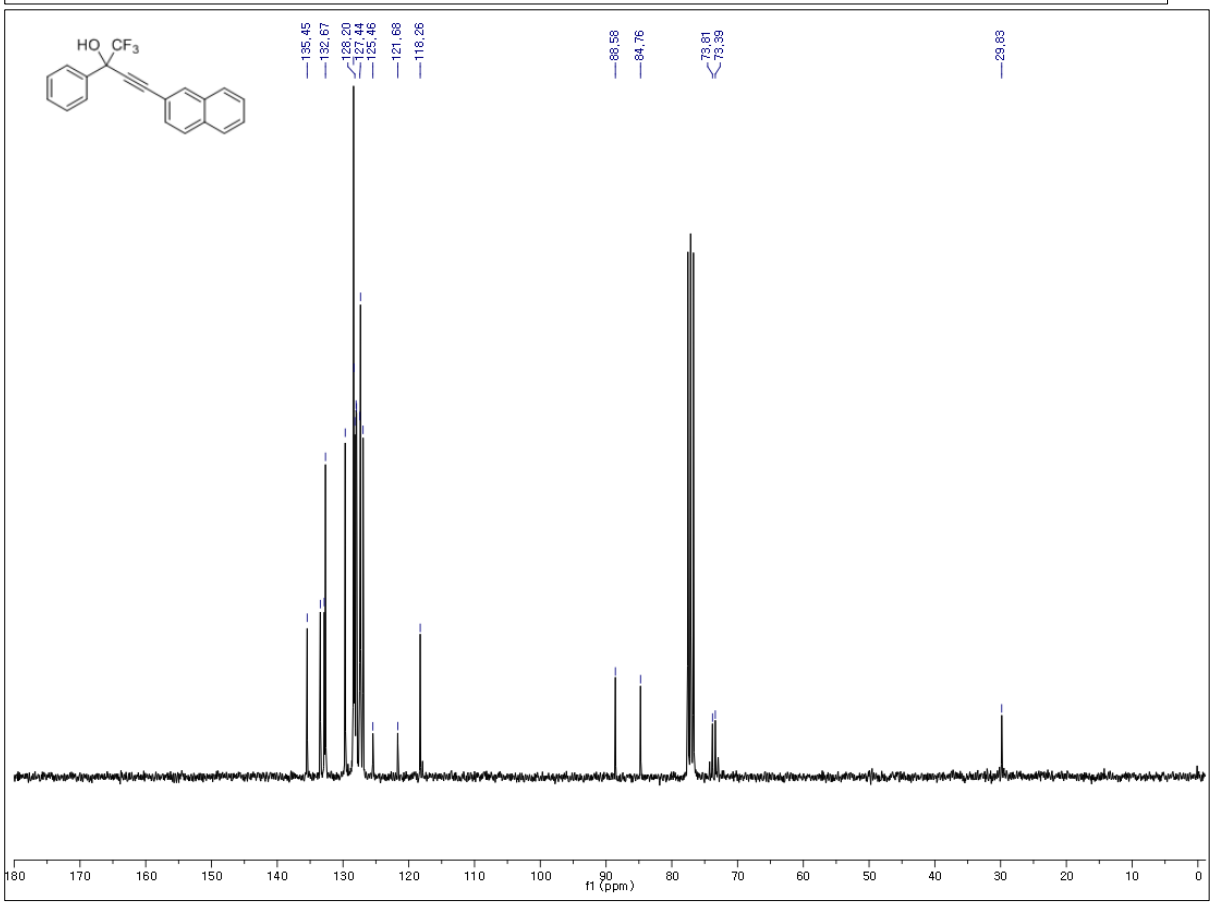

HO CF3
OMe
7.83
7.82
7.81
7.45
7.26
6.89
6.88
6.87
6.86
3.83
3.12
2.42
6.18
2.42
3.53
1.00
ppm

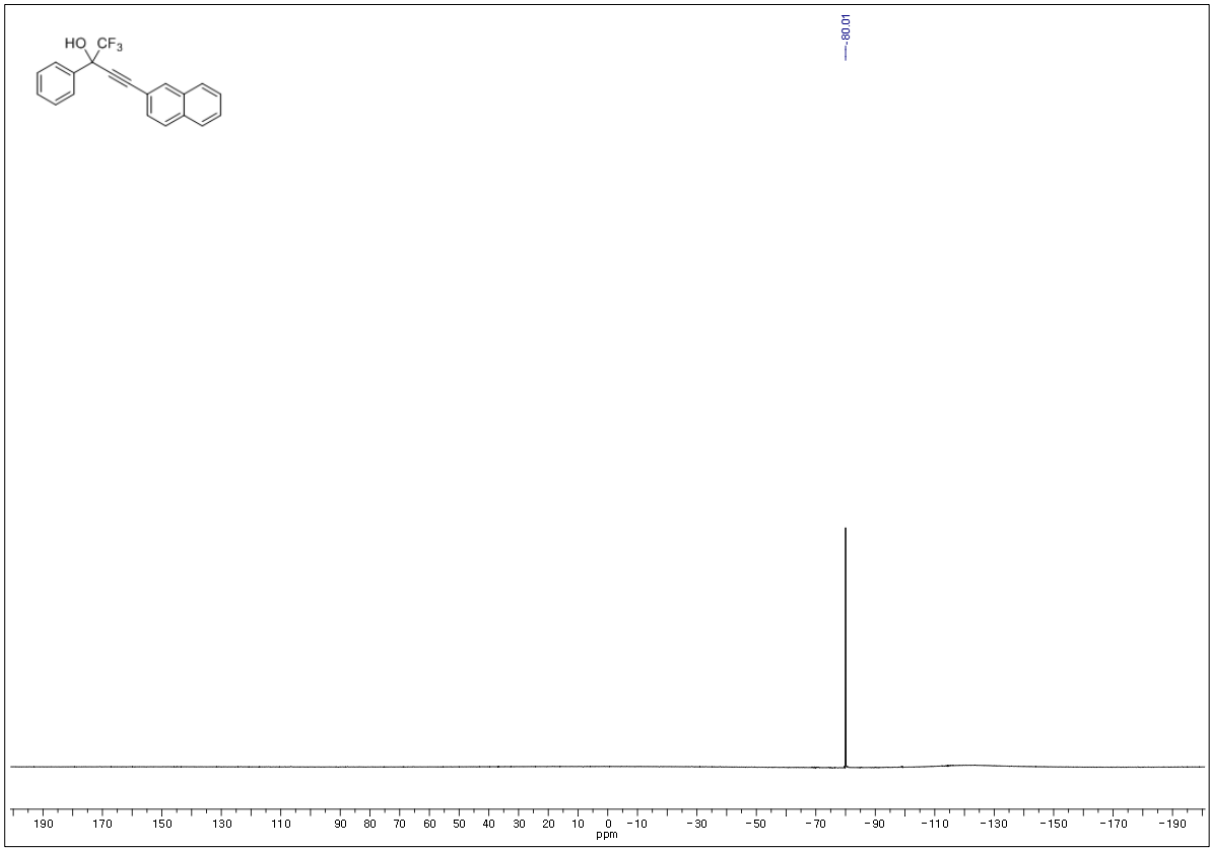

HO CF3
OMe
160.59
135.57
133.72
129.52
128.29
127.31
114.18
113.02
88.28
83.31
73.67
73.24
55.45
29.69
ppm

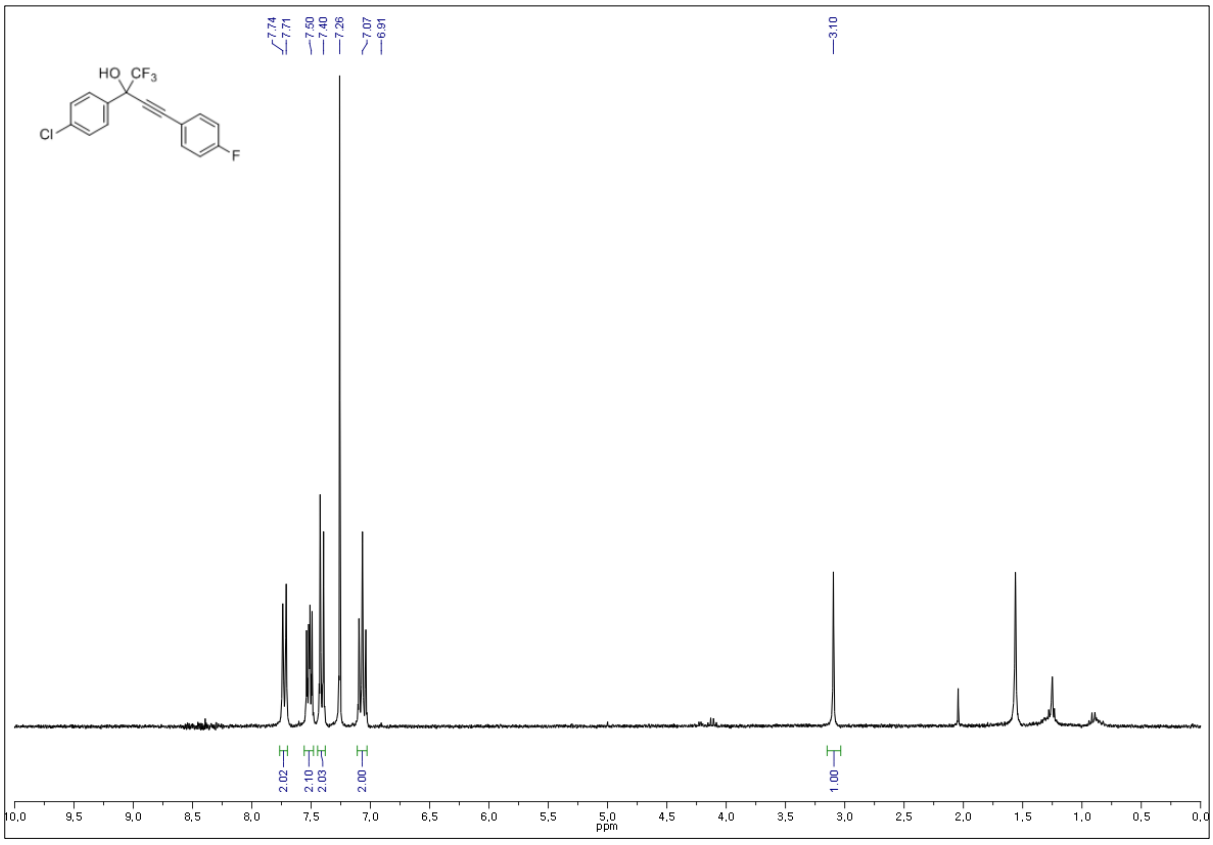

HO
CF3
OMe
-80.19
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190
ppm

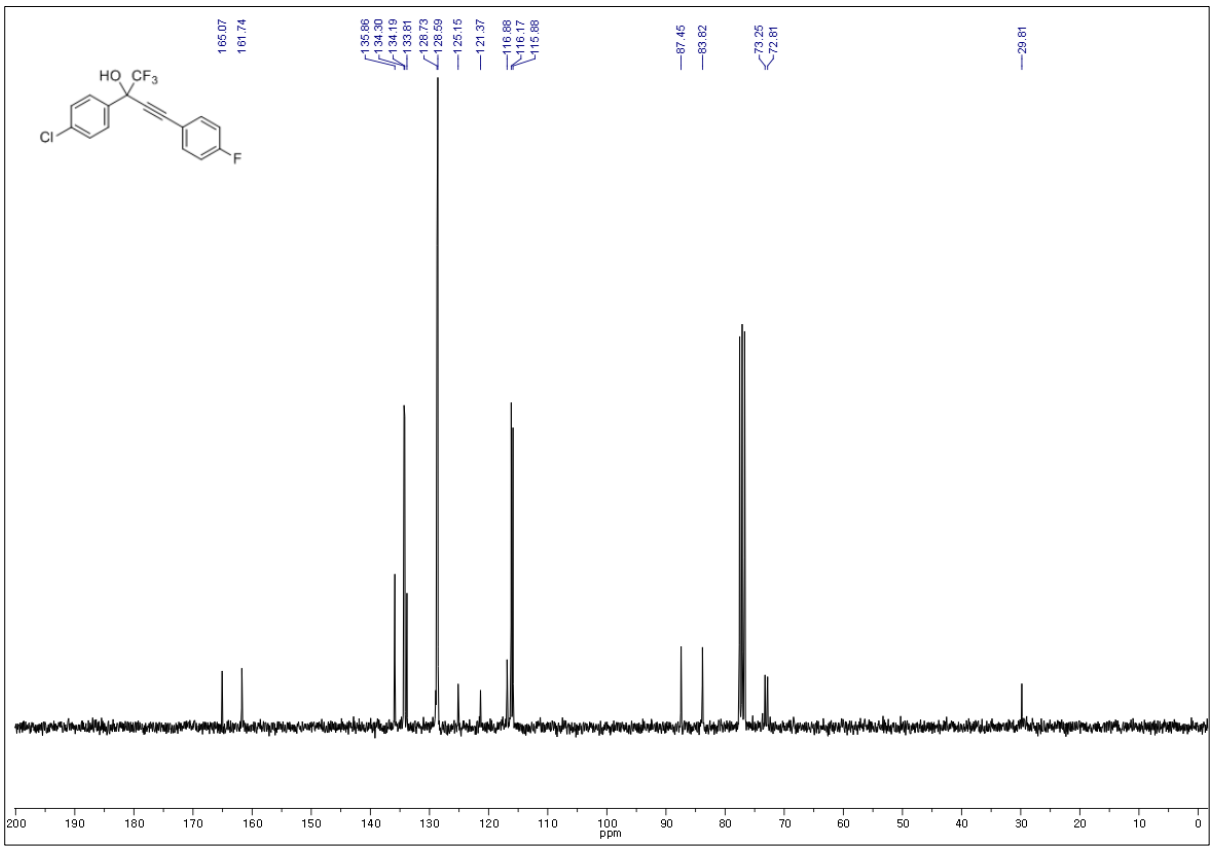

HO
CF3
7.83
7.81
7.80
7.80
7.44
7.41
7.26
7.18
7.15
3.09
2.38
1.95
5.08
2.09
1.00
3.10
10,0 9,5 9,0 8,5 8,0 7,5 7,0 6,5 6,0 5,5 5,0 4,5 4,0 3,5 3,0 2,5 2,0 1,5 1,0 0,5
ppm

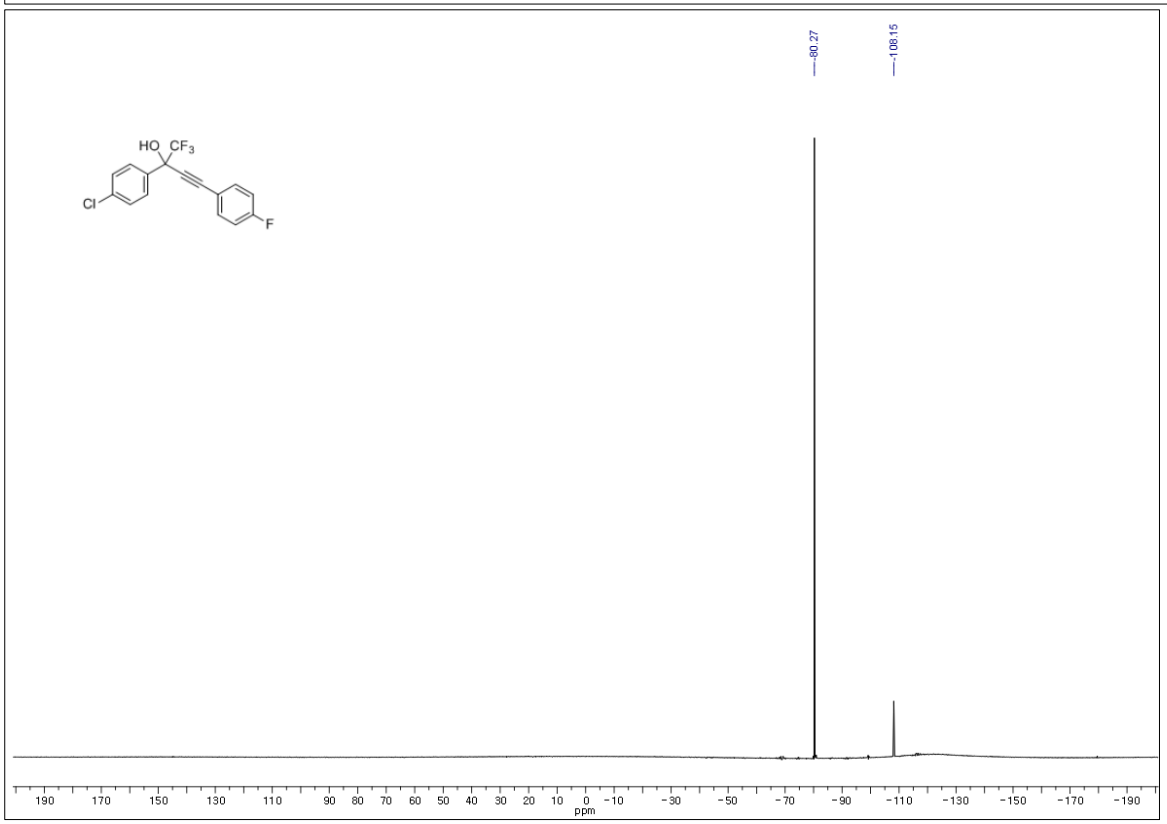

HO
CF3
8.09
7.81
7.47
7.26
3.23
1.13
5.66
6.49
1.00
f1 (ppm)

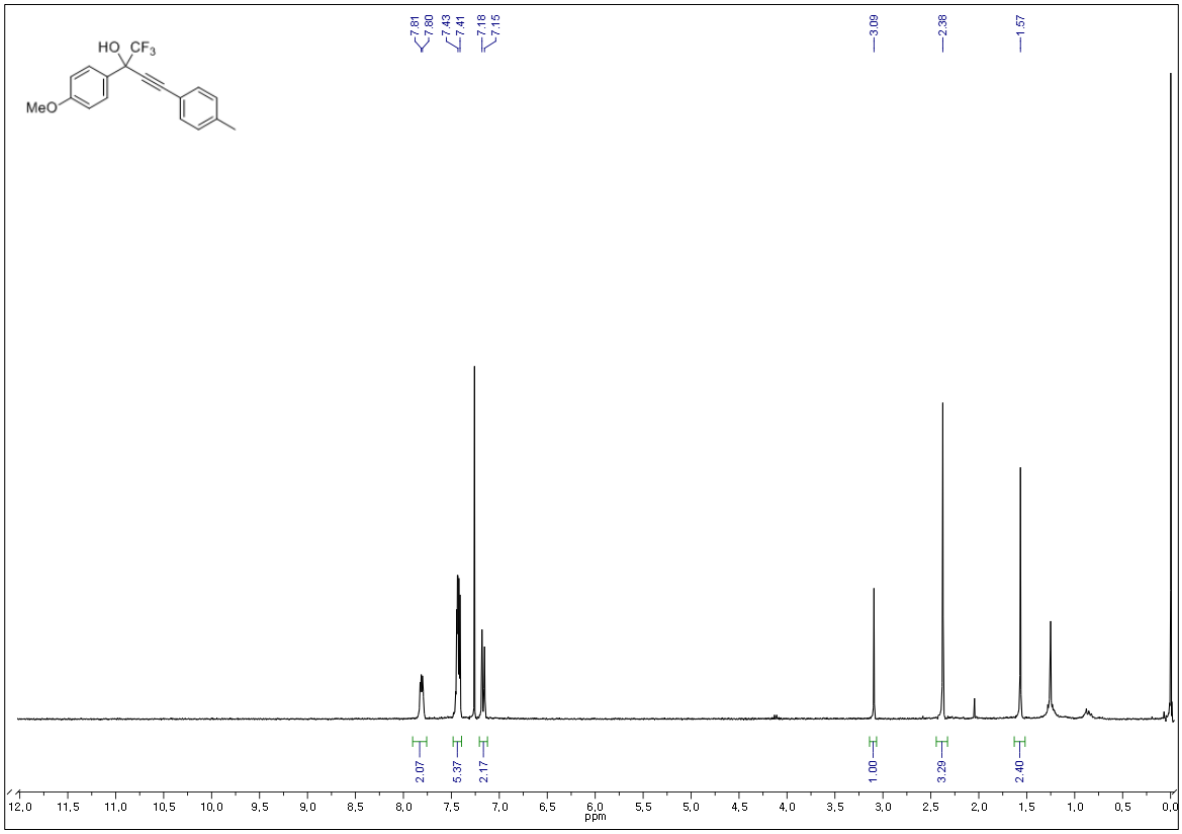

HO
CF3
135.45
132.67
128.20
127.44
125.46
121.68
118.26
88.58
84.76
73.81
73.39
29.83
f1 (ppm)

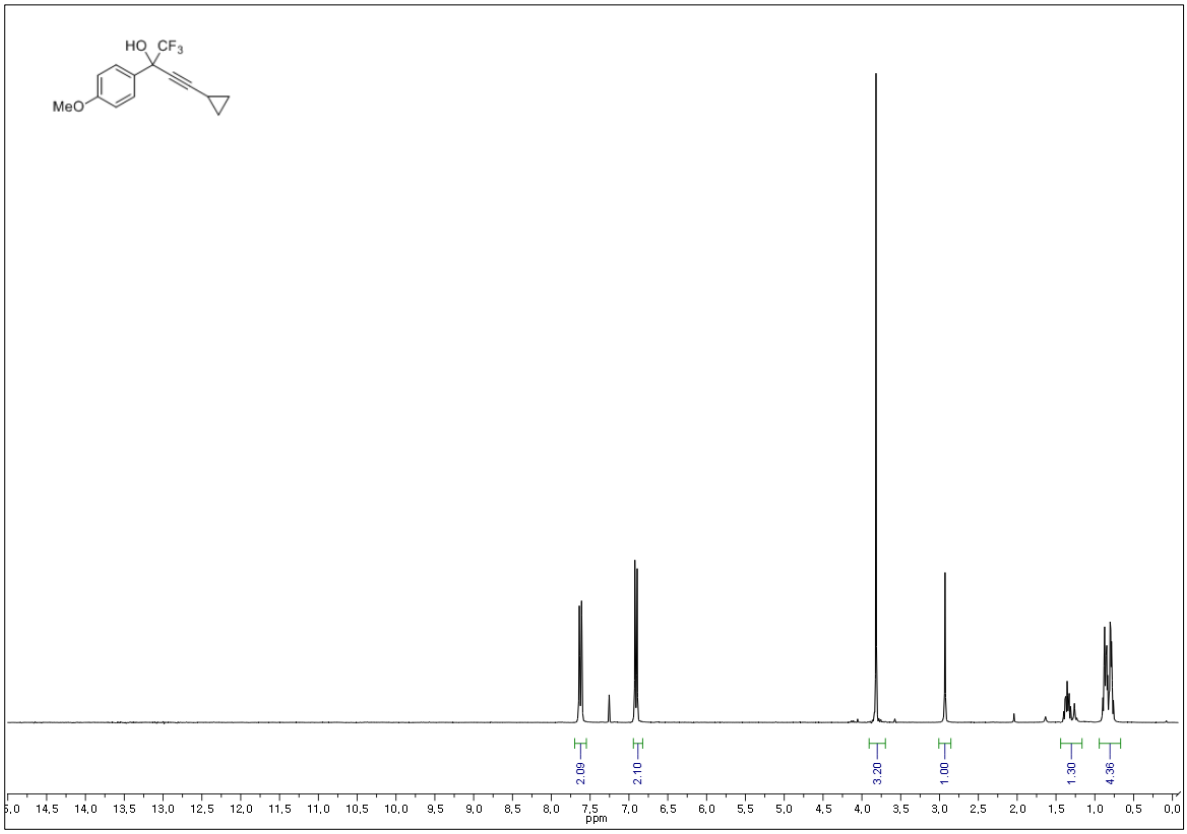

HO
CF3
-80.01
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190
ppm

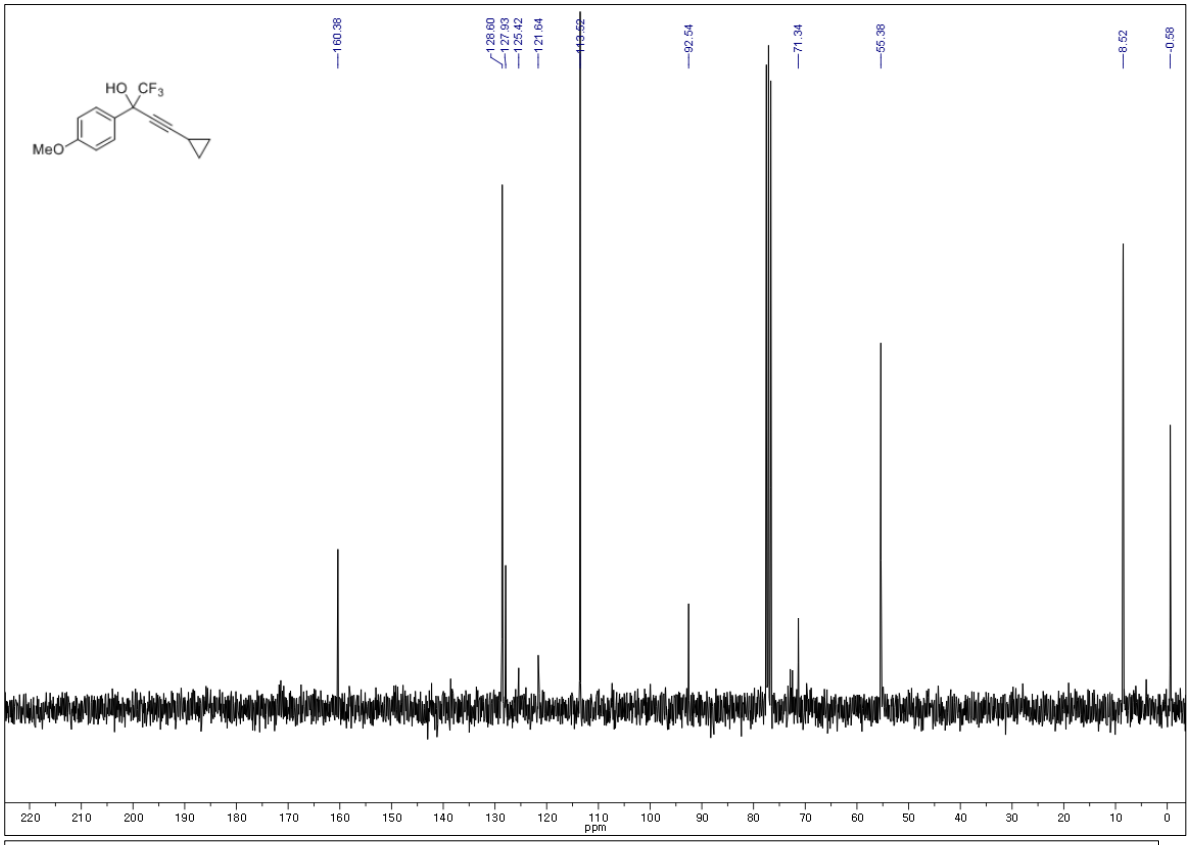

HO
CF3
Cl
F
7.74
7.71
7.50
7.40
7.26
7.07
6.91
3.10
2.02
2.10
2.03
2.00
1.00
10,0 9,5 9,0 8,5 8,0 7,5 7,0 6,5 6,0 5,5 5,0 4,5 4,0 3,5 3,0 2,5 2,0 1,5 1,0 0,5 0,0
ppm

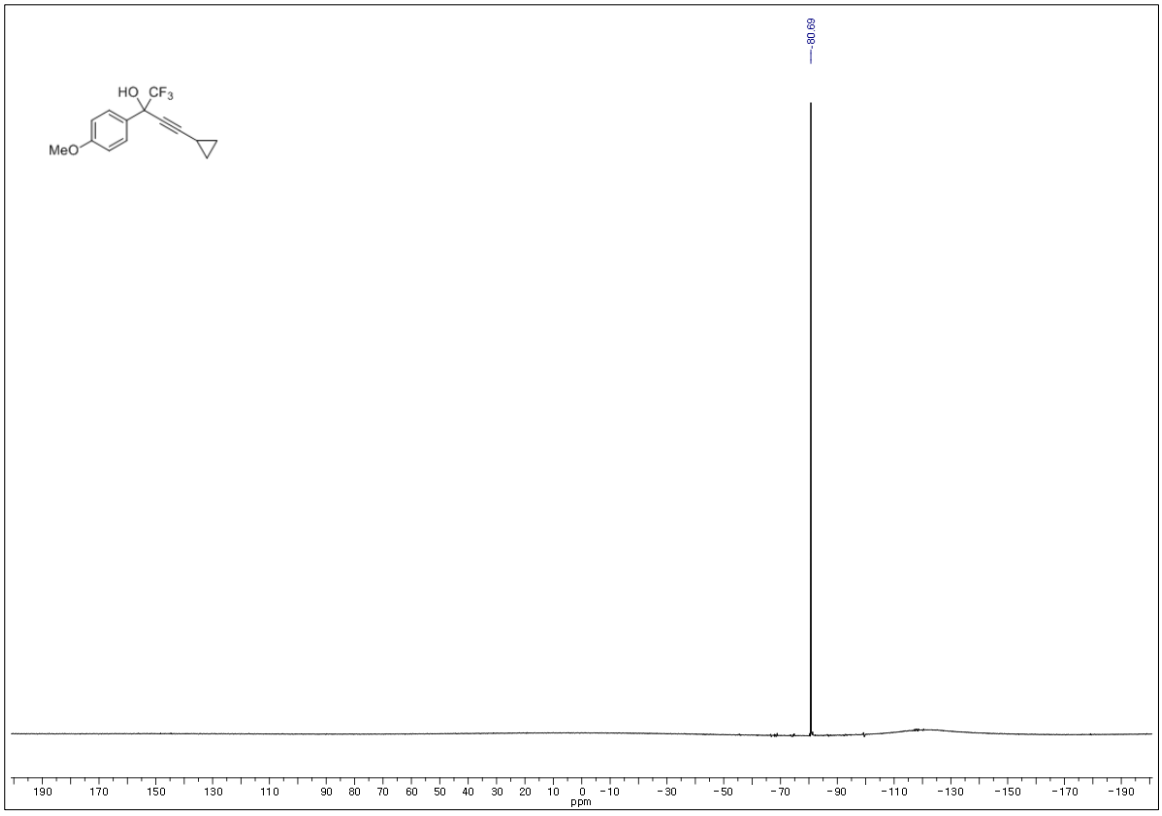

165.07
161.74
135.86
134.30
134.19
133.81
128.73
128.59
125.15
121.37
116.88
116.17
115.88
87.45
83.82
73.25
72.81
29.81
HO
CF3
Cl
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

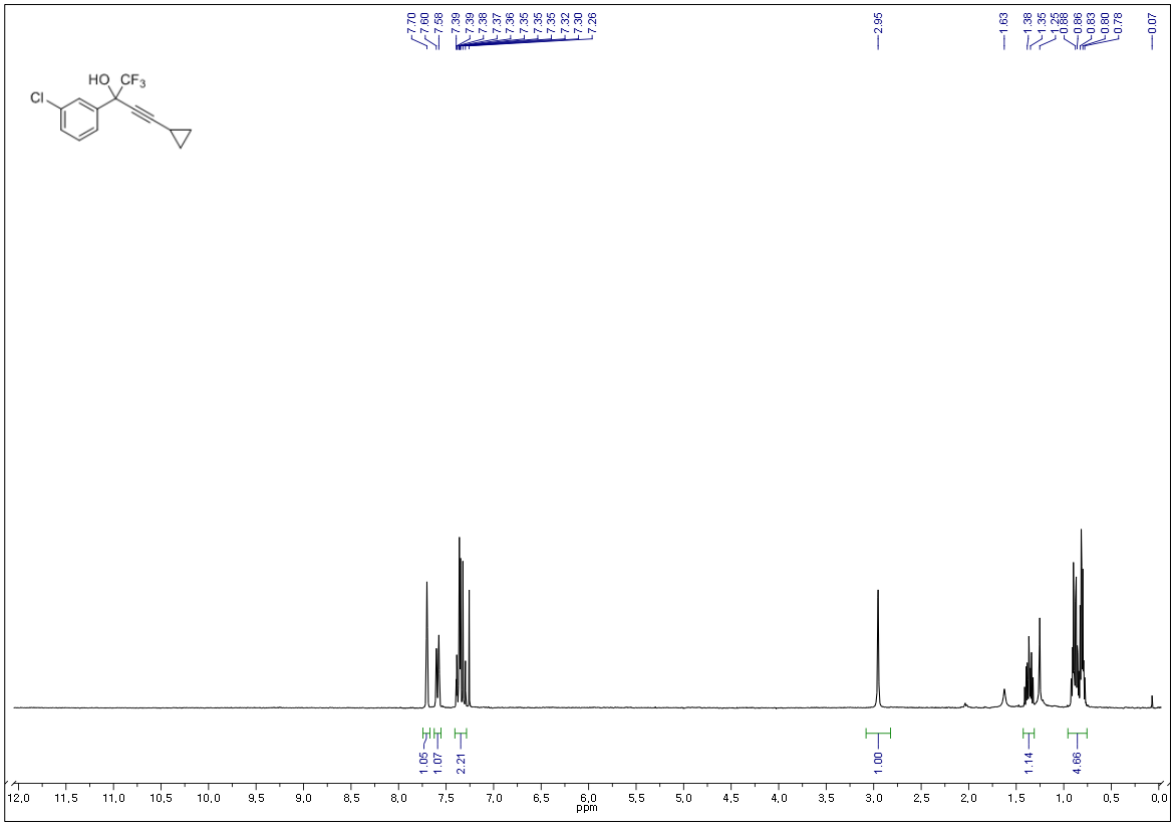

-80.27
-108.15
HO
CF3
Cl
F
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
ppm

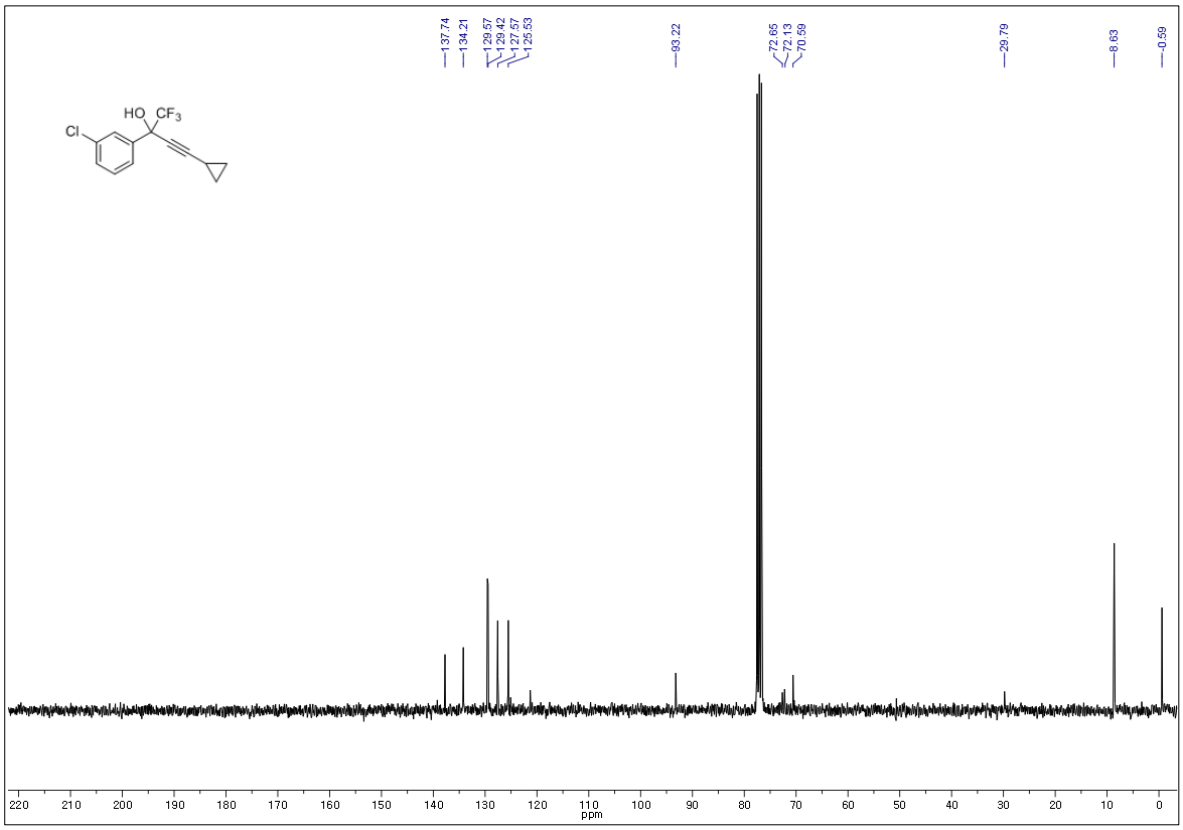

HO
CF3
MeO
7.81
7.80
7.43
7.41
7.18
7.15
3.09
2.38
1.57
2.07
5.37
2.17
1.00
3.29
2.40
ppm

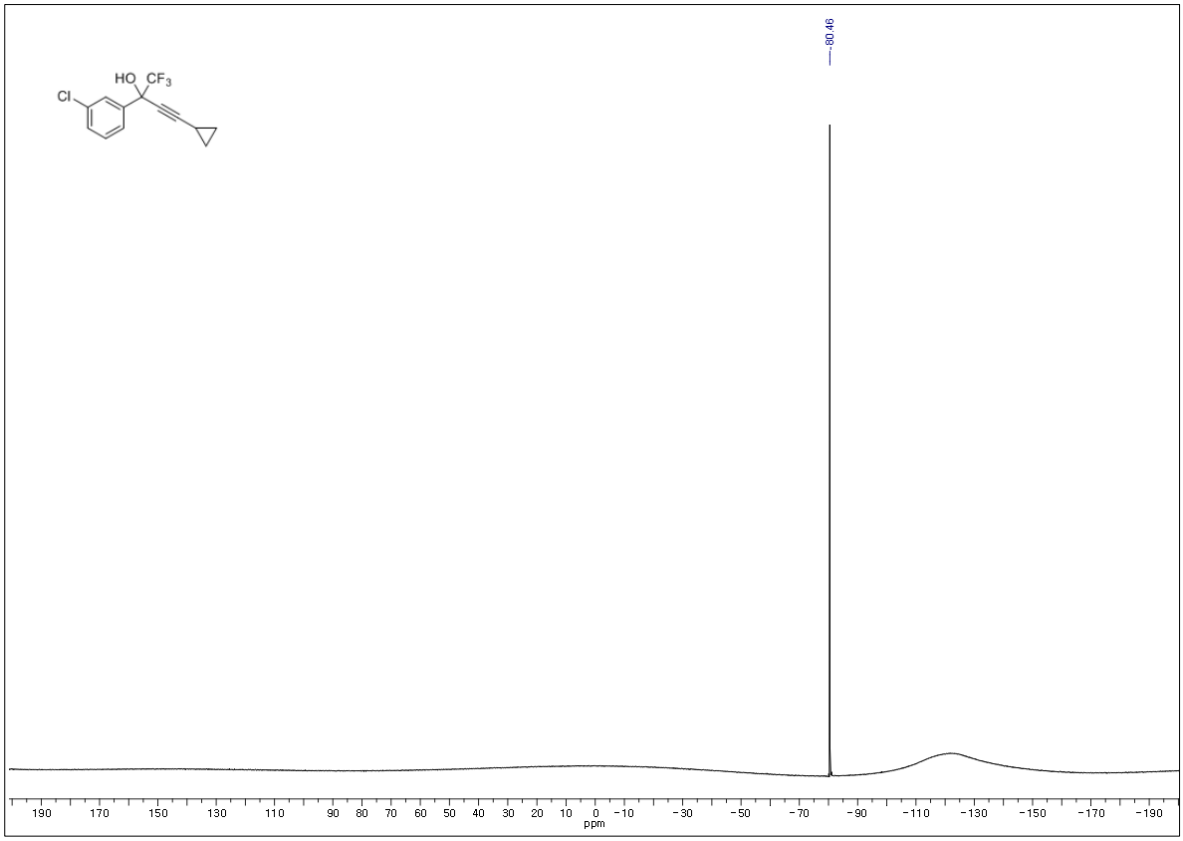

HO
CF3
MeO
2.09
2.10
3.20
1.00
1.30
4.36
ppm


160.38
128.60
127.93
125.42
121.64
113.52
92.54
71.34
55.38
6.52
-0.58
HO
CF3
MeO
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


-80.69
HO
CF3
MeO
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
ppm


HO
CF3
Cl
7.70
7.60
7.58
7.39
7.39
7.38
7.37
7.36
7.35
7.35
7.35
7.32
7.30
7.26
2.95
1.63
1.38
1.35
0.86
0.83
0.80
0.78
0.07
1.05
1.07
2.21
1.00
1.14
4.66
ppm


HO
CF3
Cl
137.74
134.21
129.57
129.42
127.57
125.53
93.22
72.65
72.13
70.59
29.79
8.63
-0.59
ppm


HO
CF3
Cl
-80.46
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
ppm