Supporting Information
for
Chiral Allene-Containing Phosphines in Asymmetric Catalysis

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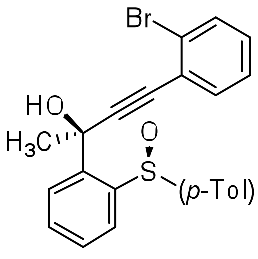
Method and Materials

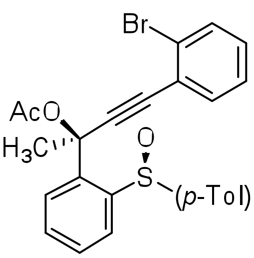
General. Unless otherwise stated, reactions were performed using freshly purified solvents which were purified using solvent purification columns purchased from Glass Contour, Laguna Beach, CA. All reactions were monitored by thin-layer chromatography with E. Merck silica gel 60 F254 pre-coated plates (0.25 mm). Gas chromatography (GC) was performed on an HP 6890N autosampling GC with an HP-5 capillary column and equipped with a FID detector. Flash chromatography was performed with indicated solvents using silica gel (particle size 0.032-0.063m) purchased from Sorbent Technologies. ^{1}H and ^{13}C NMR spectra were recorded on Varian Inova-400 MHz or 500 MHz spectrometer. Chemical shift are reported relative to internal chloroform (CDCl3: 1H, δ = 7.27, 13C, δ = 77.26). Coupling constants are in Hz and are reported as d (doublet), t (triplet), q (quartet). For signals having multiple coupling patterns, the coupling constant are listed in the same order as the pattern (e.g. dt, *J* = 2.0, 4.0; 2.0 is the coupling constant for the doublet and 4.0 is for the coupling constant for the triplet). HPLC analyses were carried out on an Agilent 1200 series or a Shimadzu LC-2010A system. Optical rotations were measured on a Rudolph Research Analytical Autopol® IV Polarimeter (50/60 Hz). Mass spectra were acquired on an Agilent technologies 1200 series LC/MS using indicated ionization methods.

Materials. Chemicals were purchased from Aldrich, Fisher or Alfa Aesar and used without purification.

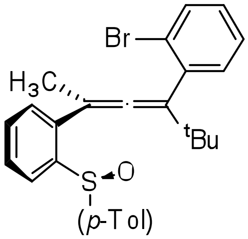
Preparation of Ligands.



(Ss, R)-2-(2'-(p-tolylsulfinyl)phenyl)-4-(2'-bromophenyl)but-3-yn-2-ol (2).[1] To a 100 mL flask charged with anhydrous cerium chloride (2.2 g, 8.9 mmol, cerium chloride was dried under vacuum overnight at 140 ^{o}C) was added anhydrous THF (20 mL) under nitrogen at rt. After stirring 2 h at rt, the resulting suspension was cooled to -78 ^{o}C. Separately, the alkynyllithium was prepared by adding LHMDS (6.4 mL, 6.4 mmol, 1.0 M solution in hexanes) to a solution of 2-bromophenylacetylene (1.12 g, 6.2 mmol) in anhydrous THF (10 mL) at -78 ^{o}C and stirring for 30 min. The alkynyl lithium solution was warmed to -40 ^{o}C and then added via canula to the suspension of $CeCl_3$ at -78 ^{o}C. The reaction mixture was stirred for 30 min at -78 ^{o}C and a solution of (*S*)-1-(2-(*p*-tolylsulfinyl)phenyl)ethanone (1.14 g, 4.43 mmol) in dry THF (10 mL) was added. After 3 h at -78 ^{o}C, the reaction mixture was slowly warmed up to rt and then treated with saturated aqueous NH_4Cl (10 mL). The crude mixture was filtered through Celite and washed with acetone. The filtrate was concentrated and extracted with EtOAc (3X). The combined organic layers were dried over $MgSO_4$, filtered, and evaporated to give crude product, which was purified by flash column chromatography on silica gel to afford **2** (1.68 g, 86%) as a single diastereomer. $[\alpha]^{25}_{D}$ = -192.8 ° (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.73 (s, 3H), 2.32 (s, 3H), 3.59 (br s, 1H), 7.15-7.18 (m, 3H), 7.20-7.25 (m, 1H), 7.40-7.55 (m, 6H), 7.75-7.77 (m, 1H), 8.08-8.10 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.6, 32.5, 71.5, 83.8, 97.7, 124.8, 125.9, 126.2, 127.14, 127.19, 127.22, 128.9, 129.7, 129.9, 130.7, 132.5, 133.9, 141.1, 143.0, 143.2, 143.7. ESI MS m/z: 438.8, $[M+H]^+$.

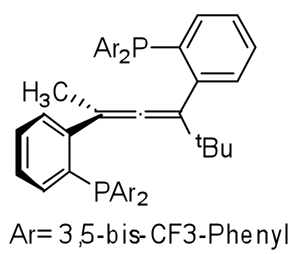


(*Ss*, *R*)-2-(2'-(*p*-tolylsulfinyl)phenyl)-4-(2'-bromophenyl)but-3-yn-2-yl acetate (3). A mixture of **2** (1.2 g, 2.7mmol), Ac_2O (1.0 g, 9.2mmol), Et_3N (1.0 g, 9.9 mmol) and DMAP (6 mg, 0.05 mmol) in CH_2Cl_2 (10 ml) was stirred at rt overnight. The reaction solution was concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel (hexane/ethyl acetate, 10/1) to afford the title compound in 98% yield (1.3 g). $[\alpha]^{24}_{D}$ = -277.0 ° (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.97 (s, 3H), 2.10 (s, 3H), 2.27 (s, 3H), 7.06 (dt, *J* = 2.0 Hz, *J* = 10.0 Hz, 1H), 7.12 (dt, *J* = 2.0 Hz, *J* = 10.0 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.34 (dd, *J* = 2.0 Hz, *J* = 7.5 Hz, 1H), 7.37-7.47 (m, 5H), 7.81 (d, *J* = 7.5 Hz, 1H), 7.87 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.6, 22.2, 31.1, 76.2, 86.8, 93.7, 124.3, 125.9, 126.6, 126.7, 127.2, 128.0, 129.7, 130.0, 130.3, 131.6, 132.5, 133.8, 141.3, 141.4, 142.4, 142.5, 168.7. ESI MS m/z: 503.0, $[M+Na]^+$.

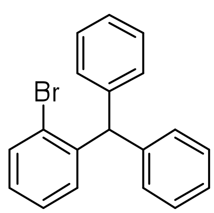


(*Ss*, *S*)-1-(5',5'-dimethyl-4'-(2"-bromophenyl)hexa-2',3'-dien-2'-yl)-2-*p*-tolylsulfinylbenzene (4). To a stirred suspension of CuCN (2.0 g, 22.3 mmol) in anhydrous THF (15 ml) at -42 ^{o}C

(CH_3CN-dry ice) under N_2 was added *t*BuLi solution (11mL, 1.7M in pentane). The resulting solution was stirred another 10 min before a solution of **3** in anhydrous THF (10 mL) was added. The reaction mixture was stirred for 1.5 h under the same conditions. Sat. NH_4Cl aq. was added to quench the reaction. The water layer was extracted by Et_2O. The combined organic layer was concentrated under reduced pressure and the residue was purified by silica gel chromatography (hexane/ethyl acetate, 10/1) to afford the title compound in 88% yield (899mg). $[\alpha]^{25}_D$ = -195.8 ° (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.06 (s, 9H), 1.54 (s, 3H), 2.20 (s, 3H), 6.84 (d, *J* = 6.5 Hz, 2H), 7.00 (d, *J* = 6.5 Hz, 2H), 7.13 (t, *J* = 7.0 Hz, 1H), 7.18 (d, *J* =7.0 Hz, 1H), 7.30-7.50 (m, 3H), 7.42 (t, *J* = 6.5 Hz, 1H), 7.68 (d, *J* = 6.5 Hz, 1H), 8.09 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 19.1, 21.6, 29.8, 36.5, 99.3, 114.7, 124.2, 125.6, 127.0, 127.2, 127.9, 129.1, 129.8, 130.5, 136.3, 137.6, 141.7, 142.4, 143.2, 200.3. ESI MS m/z: 500.8, $[M+Na]^+$.

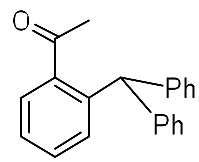


(*S*)-2,4-bis(2-(bis(3,5-bis(trifluoromethyl)phenyl) phosphino)phenyl)-5, 5-dimethyl-hexa-2,3-diene (5) To a stirred solution of allene **4** (768 mg, 1.6 mmol) in anhydrous diethyl ether (7 mL) at -78°C under nitrogen was added tert-butyllithium (5.7 ml, 9.68 mmol, 1.7 M solution in hexane). The reaction mixture was then stirred for 10 min at -78 °C under nitrogen before the addition of a solution of chlorobis(3,5-bistrifluoromethylphenyl)phosphine (4.9 g, 10.0 mmol) in anhydrous diethyl ether (5 mL). The resulting solution was allowed to warm to room temperature and stirred overnight. Sat. NH_4Cl solution (5 mL) was added to the reaction to quench the unreacted reagent. The mixture was extracted by Et_2O, the organic layers were combined, and the solvent was removed under vacuum. The residue was purified by silica gel chromatography with hexane as the eluent to afford the allene ligand (750 mg, 40%) as a white solid. Mp, 147-148 °C; $[\alpha]^{24}_D$ = +82 ° (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.06 (s, 9H), 1.90 (s, 3H), 6.61 (s, 1H), 6.81 (dd, *J* = 7.7, 4.0 Hz, 1H), 6.97 (dd, *J* = 7.5, 3.3 Hz, 1H), 7.02 (t, *J* = 7.1 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.36 – 7.24 (m, 4H), 7.42 (d, *J* = 6.2 Hz, 2H), 7.46 (s, 1H), 7.57 (d, *J* = 5.7 Hz, 2H), 7.72 (d, *J* = 5.8 Hz, 2H), 7.90 – 7.82 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 16.8, 25.2-25.5, (multiple signals of CH_3 in *t*-Bu due to atropisomers; a single peak at 30.2 ppm was observed when the ^{13}C NMR was recorded at 50 °C), 31.6, 97.8 (d, $^3J_{C\text{-}P}$ = 8.5 Hz), 108.8 (d, $^3J_{C\text{-}P}$ = 6.9 Hz), 196.6 (central C of allene). Other peaks cannot be interpreted because of the overlap due to C-P and C-F coupling. Please see the attached spectrum. ^{31}P NMR (202 MHz, $CDCl_3$) δ: -12.0, -11.3; ESI MS m/z: 1174.9, $[M+H]^+$.

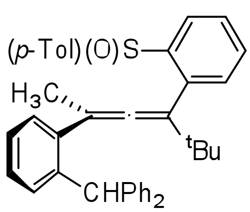


1-(2'-bromophenyl)-1,1-diphenylmethane.[2] To a stirred solution of methyl 2-bromobenzoate (6.0 g, 28 mmoL) in anhydrous Et_2O (90 mL) at 0 °C under nitrogen was added PhMgBr (21.7 mL, 3.0 M in Et_2O). The reaction mixture was stirred at rt overnight. Sat. NH_4Cl solution (15 mL) was added the reaction to quench the unreacted reagent. The mixture was extracted by Et_2O, the organic layers were combined and the solvent was removed under vacuum. The residue was purified by silica gel chromatography with hexane as the eluent to afford 1-(2'-

bromophenyl)-1,1-diphenylmethanol (7.8 g, 82%). A mixture of 1-(2'-bromophenyl)-1,1-diphenylmethanol (7.0 g, 20.6 mmol) and HCOOH (30 mL) in toluene (100 mL) was refluxed overnight. The toluene layer was then washed by sat. $NaHCO_3$ and concentrated under reduced pressure. The residue was purified by silica gel chromatography with hexane as the eluent to afford the title compound (5.0 g) in 75% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 5.96 (s, 1H), 6.95 (dd, *J* = 1.0, 7.5 Hz, 1H), 7.07-7.14 (m, 5H), 7.21-7.32 (m, 7H), 7.59 (dd, *J* = 1.0, 8.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 56.2, 125.8, 126.7, 127.5, 128.3, 128.6, 129.9, 131.6, 133.3, 142.8, 143.5.

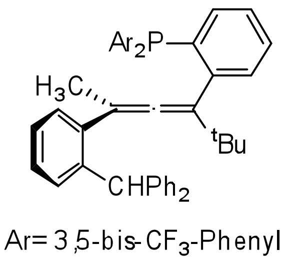


1-(2-benzhydrylphenyl)ethanone. To a stirred solution of 1-(2'-bromophenyl)-1,1-diphenylmethane (2.73 g, 8.48 mmol) in anhydrous Et_2O (70 mL) at -78 °C under a nitrogen atmosphere was added tert-butyllithium (10.0 mL, 17.0 mmol). After 10 min, acetaldehyde (2.9 mL, 51 mmol) was added at -78°C. After 2h, sat. NH_4Cl solution (25 mL) was added. The mixture was extracted by Et_2O, the organic layers were combined and the solvent was removed under reduced pressure. The residue was redissolved in CH_2Cl_2 (40 mL), and Dess-Martin periodane (3.96 g, 9.4 mmol) was added to this solution. The reaction mixture was stirred at rt overnight. A 1:1 mixture of 10% $NaHCO_3$ and sat. $Na_2S_2O_3$ solution was added to the reaction mixture, which was stirred for another 30 min and became a clear solution. The mixture was extracted by ethyl acetate, the organic layers were combined, and the solvent was removed under vacuum. The residue was purified by silica gel chromatography (hexane: ethyl acetate 10:1) to afford the title compound (1.6 g, 66% in 2 steps). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.26 (s, 3H), 6.40 (s, 1H), 7.00-7.40 (m, 13 H), 7.56 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 30.2, 52.1, 126.4, 126.5, 128.3, 128.5, 130.1, 130.9, 131.1, 139.7, 143.1, 143.7, 203.7. ESI MS m/z 308.9 $[M+Na]^+$.

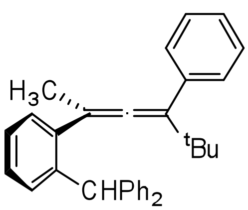


***(Ss, R*)-2-(2-(diphenylmethyl)phenyl)-4-(2'-(p-tolylsulfinyl)phenyl)-5, 5-dimethyl-hexa-2,3-diene and *(Ss, S*)-2-(2-(diphenylmethyl)phenyl)-4-(2'-(p-tolylsulfinyl)phenyl)-5, 5-dimethyl-hexa-2,3-diene (13a** and **b)** (allene stereochemistry unassigned)

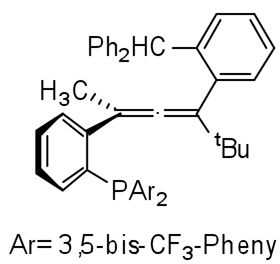
These diastereomers were made analogously to allene **4** from (*S*)-2-(*p*-tolylsulfinyl)phenylacetylene and 1-(2-benzhydrylphenyl)ethanone. The diastereomers were resolved by flash column chromatography on silica gel (hexane: ethyl acetate 10-6:1). **13a:** $[\alpha]^{25}_D$ = -303.3 ° (c = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.06 (s, 9H), 2.03 (s, 3H), 2.35 (s, 3H), 5.79 (s, 1H), 6.80-6.82 (m, 2H), 6.94 (d, *J* = 7.2 Hz, 2H), 7.05 (d, *J* = 7.2 Hz, 1H), 7.08-7.12 (m, 3H), 7.15-7.32 (m, 10H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.51 (d, *J* = 6.8 Hz, 2H), 7.93 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 20.8, 21.7, 30.6, 35.6, 52.8, 102.8, 126.1, 126.4, 126.6, 126.7, 126.8, 127.2, 128.2, 128.4, 128.5, 128.9, 129.6, 129.8, 130.1, 130.4, 130.6, 137.6, 139.7, 141.6, 142.2, 144.1, 144.5, 144.8, 201.4; ESI MS m/z: 566.9, $[M+H]^+$. **13b**: $[\alpha]^{25}_D$ = +28.4 ° (c = 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.24 (s, 9H), 1.63 (s, 3H), 2.30 (s, 3H), 5.87 (s, 1H), 6.75-6.77 (m, 2H), 6.90-6.92 (m, 2H), 7.02 (d, *J* = 6.8 Hz, 1H), 7.05-7.09 (m, 3H), 7.15-7.35 (m, 12H), 7.41 (t, *J* = 6.8 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 21.4, 21.5, 30.2, 36.4, 51.9, 101.8, 110.7, 125.8, 126.3, 126.4, 126.8, 127.3, 127.4, 128.4, 128.5, 128.7, 129.0, 129.55, 129.57, 129.9, 130.5, 131.0, 131.1, 136.9, 139.3, 140.6, 140.9, 143.0, 144.1, 145.0, 145.3, 201.1; ESI MS m/z: 566.9, $[M+H]^+$.



(-)-2-(2-(diphenylmethyl)phenyl)- 4-(2-(bis-(3,5-bistrifluoro methylphenyl)phosphino)phenyl-5, 5-dimethyl-hexa-2,3-diene (12). It was made by the same method of **5** from 1**3a** in 26% yield. $[\alpha]^{25}_D$ = -64.4 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.14 (s, 9H), 1.41 (s, 3H), 5.79 (s, 1H), 6.62-6.64 (m, 1H), 6.80-6.85 (m, 2H), 6.88-7.04 (m, 5H), 7.12-7.23 (m, 9H), 7.33 (t, J = 7.5 Hz, 1H), 7.37 (d, J = 7.5 Hz, 1H), 7.59-7.61 (m, 3H), 7.86 (d, J = 15.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 30.1, 30.3 (d, atropisomers), 36.1, 52.5, 102.3, 112.4 (d, $^3J_{C-P}$ = 6.2 Hz), 200.5 (center C of allene). Other peaks cannot be interpreted because of the overlap due to C-P and C-F coupling. Please see the attached spectrum. ^{31}P NMR (202 MHz, $CDCl_3$) δ: -10.8; ESI MS m/z: 885.2, $[M+H]^+$.

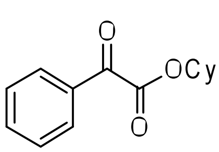


(-)-2-(2-(diphenylmethyl)phenyl)- 4-phenyl-5, 5-dimethyl-hexa-2,3-diene (14). The title compound was isolated as a side product in the preparation of **12** in 52% yield. $[\alpha]^{25}_D$ = -114.3 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.15 (s, 9H), 1.66 (s, 3H), 5.76 (s, 1 H), 6.64 (d, J = 7.0 Hz, 2H), 6.88-6.93 (m, 3H), 7.12-7.23 (m, 8H), 7.24-7.32 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 22.2, 30.1, 35.2, 52.5, 100.3, 115.1, 126.1, 126.5, 126.8, 126.9, 128.0, 128.1, 128.2, 128.9, 129.8, 130.1, 130.3, 137.8, 140.4, 141.8, 144.3, 144.6, 200.0; ESI MS m/z: 429.2, $[M+H]^+$.



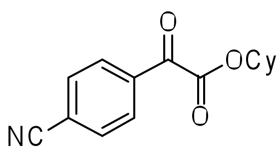
(-)-2-(2-(bis-(3,5-bistrifluoromethylphenyl)phosphino) phenyl)-4-(2-(diphenylmethyl)phenyl)-5, 5-dimethyl-hexa-2,3-diene (11). $[\alpha]^{23}_D$ = -36.8 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 0.90 (s, 9H), 1.96 (s, 3H), 6.08 (s, 1H), 6.53 (br s, 1H), 6.78 (m, 1H), 6.82-6.84 (m, 1H), 6.97 (d, J = 7.5 Hz, 1H), 7.06-7.14 (m, 5H), 7.15-7.24 (m, 4H), 7.25-7.29 (m, 3H), 7.30-7.35 (m, 2H), 7.40-7.52 (m, 4H), 7.85 (d, J = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 22.0, 30.8, 36.1, 52.6 (d, atropisomers), 101.5 (J^3_{C-P} = 8.9 Hz), 201.6 (J^4_{C-P} = 1.5 Hz, center C of allene). Other peaks cannot be interpreted because of the overlap due to C-P and C-F coupling. Please see the attached spectrum. ^{31}P NMR (202 MHz, $CDCl_3$) δ: -10.7; ESI MS m/z: 885.2, $[M+H]^+$.

Preparation of the substrates.

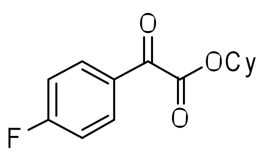


Cyclohexyl 2-phenyl-2-oxoacetate (15b). To a stirred solution of phenylglyoxylic acid (1.5 g, 10.0 mmol) and cyclohexanol (1.5 g, 15.0 mmol) in CH_2Cl_2 (8 mL) was added DCC (2.2 g, 11.0 mmol) and

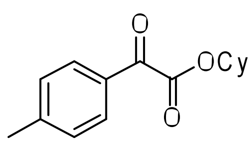
DMAP (30 mg, 0.24 mmol) at rt. The reaction mixture was stirred overnight and then filtered through Celite. The filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane: ethyl acetate 10:1) to afford the title compound (1.8 g) in 78% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26-1.34 (m, 1H), 1.39-1.46 (m, 2H), 1.55-1.64 (m, 3H), 1.72-1.84 (m, 2H), 1.97-2.05 (m, 2H), 5.11 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.53 (t, J = 7.5 Hz, 2H), 7.67 (t, J = 7.5 Hz, 1H), 8.01 (d, J = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.8, 25.4, 31.6, 75.5, 129.1, 130.0, 132.7, 135.0, 163.9, 187.0; ESI MS m/z: 254.9, $[M+Na]^+$.



Cyclohexyl 2-(4-cynophenyl)-2-oxoacetate. From 4-cynophenylglyoxylic acid, the same method as used in the preparation of cyclohexanyl 2-phenyl-2-oxoacetate gave the title compound in 80% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26-1.34 (m, 1H), 1.36-1.46 (m, 2H), 1.54-1.64 (m, 3H), 1.77-1.81 (m, 2H), 1.98-2.01 (m, 2H), 5.09 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.81 (d, J = 8.0 Hz, 2H), 8.13 (d, J = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.8, 25.3, 31.6, 76.4, 117.8, 118.0, 130.6, 132.8, 135.9, 162.4, 185.1; ESI MS m/z: 257.9 $[M+H]^+$.

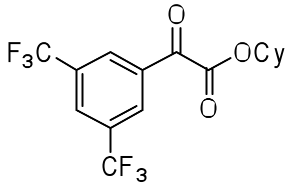


Cyclohexyl 2-(4-fluorophenyl)-2-oxoacetate.[3] Cyclohexanol (0.97 mL, 9.2 mmol) was added slowly to a solution of oxalyl chloride (0.8 mL, 9.5 mmol) in anhydrous THF (15 mL) at 0 °C within 15 min. After 4 h, a solution of imidazole (1.9 g, 27.6 mmol) in THF (8 mL) was added into the above solution at 0 °C under nitrogen. After an additional 2 h of being stirred at room temperature, the mixture was filtered and the precipitate was washed with anhydrous THF (8 mL). The filtrate was cooled to –50 °C under nitrogen. A solution of 4-flourophenyl magnesium bromide (9 mmol in 9 mL THF) was added dropwise over 30 min with stirring. The solution was allowed to come to room temperature over 3 h and poured into ice-water. The solution was extracted with ether, washed with brine and dried over $NaSO_4$. Removal of solvents with reduced pressure and purification by chromatography on silica gel (hexane:ethyl acetate 10:1) gave the title compound (1.3 g) in 58% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ1.24-1.34 (m, 1H), 1.37-1.46 (m, 2H), 1.54-1.63 (m, 3H), 1.75-1.82 (m, 2H), 1.96-2.04 (m, 2H), 5.08 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.16-7.20 (m, 2H), 8.00-8.08 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.9, 25.4, 31.6, 75.9, 116.4 (d, J = 22.0 Hz), 129.3 (d, J = 4.3 Hz),133.5 (d, J = 9.8 Hz), 163.5, 166.9 (d, J = 256.6 Hz), 186.2; ESI MS m/z: 272.9, $[M+Na]^+$.

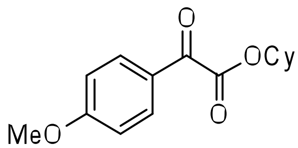


Cyclohexyl 2-p-tolyl-2-oxoacetate. From *p*-tolylmagnesium bromide; the same method as was used in the preparation of cyclohexanyl 2-(4-flourophenyl)-2-oxoacetate gave the title comound in 45% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.34 (m, 1H), 1.37-1.48 (m, 2H), 1.52-1.65 (m, 3H), 1.74-1.82 (m, 2H), 1.96-2.05 (m, 2H), 2.42 (s, 3H), 5.08 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.16 (d, J = 8.0 Hz, 2H), 7.88 (d, J =

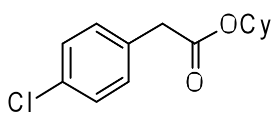
8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 22.1, 23.9, 25.4, 31.7, 75.5, 129.8, 130.2, 130.3, 146.3, 164.1, 186.7; ESI MS m/z: 269.0, $[M+Na]^+$.



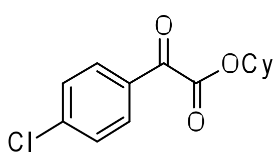
Cyclohexyl 2-(3,5-bistrifluoromethylphenyl)-2-oxoacetate. From 3,5-bis(trifluoromethyl)phenylmagnesium bromide; the same method as was used in the preparation of cyclohexanyl 2-(4-flourophenyl)-2-oxoacetate gave the title compound in 53% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.29-1.38 (m, 1H), 1.41-1.52 (m, 2H), 1.58-1.68 (m, 3H), 1.78-1.84 (m, 2H), 1.96-2.05 (m, 2H), 5.13 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 8.15 (s, 1H), 8.53 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.8, 25.3, 31.6, 76.8, 122.9 (q, J = 271.9 Hz), 127.8 (q, J = 3.6 Hz), 130.2 (q, J = 4.3 Hz),, 132.9 (q, J = 34.1 Hz), 134.7, 161.6, 183.3; ESI MS m/z: 368.9, $[M+H]^+$.



Cyclohexyl 2-(4-methoxylphenyl)-2-oxoacetate. From 4-methoxyphenylmagnesium bromide; the same method as was used in the preparation of Cyclohexanyl 2-(4-flourophenyl)-2-oxoacetate was used to give the title compound in 38% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.24-1.34 (m, 1H), 1.37-1.48 (m, 2H), 1.54-1.65 (m, 3H), 1.74-1.83 (m, 2H), 1.96-2.05 (m, 2H), 3.90 (s, 3H), 5.08 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.98 (d, J = 9.0 Hz, 2H), 7.98 (d, J = 9.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.8, 25.4, 31.6, 55.8, 75.3, 114.4, 125.7, 132.6, 164.2, 165.1, 185.5; ESI MS m/z: 263.0 $[M+H]^+$.

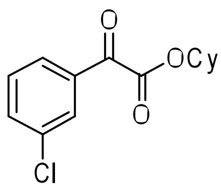


Cyclohexyl 2-(4-chlorophenyl)acetate. From 2-(4'-chlorophenyl)acetic acid; the same method as was used in the preparation of cyclohexanyl 2-phenyl-2-oxoacetate was used to give the title compound in 86% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.45 (m, 5H), 1.47-1.54 (m, 1H), 1.64-1.71 (m, 2H), 1.77-1.85 (m, 2H), 3.56 (s, 2H), 4.78 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.22 (d, J = 7.5 Hz, 2H), 7.28 (d, J = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.8, 25.6, 31.7, 41.3, 73.4, 128.8, 130.8, 133.10, 133.12, 170.7; ESI MS m/z: 253.1, $[M+H]^+$.

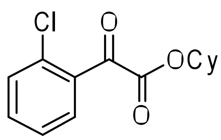


Cyclohexyl 2-(4'-chlorophenyl)-2-oxoacetate.[4] To a stirred solution of cyclohexanyl 2-(4-chlorophenyl)acetate (1.36 g, 5.4 mmol) in anhydrous MeCN (30 mL) was added DBU (0.99 g, 6.5 mmol). After the solution was stirred at room temperature under N_2 for 20 min, *p*-acetamidobenzenesulfonyl azide (1.43 mg, 5.94 mmol) was added at 0 °C in several portions. The solution was stirred at room temperature for 24 h. To the above solution was added benzene (18 mL), acetone (14 mL), H_2O (20 mL), $NaHCO_3$ (17.2 g, 204 mmol) and Oxone® (32g, 52 mmol) at 0°C. The reaction mixture was vigorously stirred overnight. Water (60 mL) was added and the mixture was extracted with diethyl ether (3 × 60 mL), and combined organic layer was dried over anhydrous

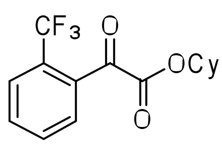
Na_2SO_4. Removal of the solvent gave a crude product, which was purified by column chromatography on silica gel to give the title product (1.08 g) in 75% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.27-1.36 (m, 1H), 1.39-1.49 (m, 2H), 1.57-1.65 (m, 3H), 1.76-1.84 (m, 2H), 1.98-2.05 (m, 2H), 5.09 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.50 (d, J = 8.5 Hz, 2H), 7.97 (d, J = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.8, 25.4, 31.7, 75.9, 129.5, 131.2, 131.5, 141.7, 163.2, 185.5; ESI MS m/z: 288.9, $[M+Na]^+$.



Cyclohexyl 2-(3'-chlorophenyl)-2-oxoacetate. From 2-(3'-chlorophenyl)acetic acid; the same method as was used for the preparation of cyclohexanyl 2-(4'-chlorophenyl)-2-oxoacetate was used to give the title compound in 68% yield in 2 steps. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.27-1.36 (m, 1H), 1.40-1.49 (m, 2H), 1.55-1.67 (m, 3H), 1.78-1.84 (m, 2H), 1.97-2.05 (m, 2H), 5.09 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.47 (t, J = 8.0 Hz, 1H), 7.63 (d, J = 8.0 Hz, 1H), 7.90 (d, J = 8.0 Hz, 1H), 8.01 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.9, 25.4, 31.6, 76.1, 128.4, 130.0, 130.4, 134.4, 134.9, 135.4, 163.0, 185.5; ESI MS m/z: 288.9, $[M+Na]^+$.



Cyclohexyl 2-(2'-chlorophenyl)-2-oxoacetate. From 2-(2'-chlorophenyl)acetic acid; the same method as was used in the preparation of cyclohexanyl 2-(4'-chlorophenyl)-2-oxoacetate was used to give the title compound in 70% yield in 2 steps. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.31 (m, 1H), 1.33-1.44 (m, 2H), 1.51-1.62 (m, 3H), 1.72-1.80 (m, 2H), 1.92-2.00 (m, 2H), 5.02 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.38-7.44 (m, 2H), 7.51 (dt, J_1 = 1.5 Hz, J_2= 8.0 Hz, 1H), 7.75 (dd, J_1 = 1.5 Hz, J_2= 8.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.7, 25.3, 31.3, 75.7, 127.4, 130.6, 131.7, 133.4, 133.8, 134.5, 162.8, 186.9; ESI MS m/z: 288.9, $[M+Na]^+$.

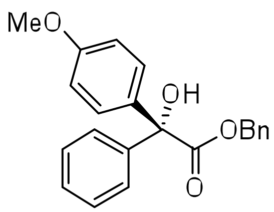


Cyclohexyl 2-(2-triflouromethylphenyl)-2-oxoacetate. From 2-(2'-trifluoromethylphenyl)acetic acid; the same method as was used in the preparation of cyclohexanyl 2-(4'-chlorophenyl)-2-oxoacetate was used to give the title compound in 38% yield in 2 steps. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.34 (m, 1H), 1.34-1.45 (m, 2H), 1.52-1.62 (m, 3H), 1.72-1.82 (m, 2H), 1.91-2.02 (m, 2H), 5.00 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.58-7.82 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.8, 25.3, 31.4, 76.4, 123.7 (q, J = 272.4 Hz), 127.1 (q, J = 4.8 Hz), 128.7 (q, J = 32.6 Hz), 129.8, 132.09, 132.10, 134.8 (q, J = 1.6 Hz), 161.0, 187.4; ESI MS m/z: 322.9, $[M+Na]^+$.

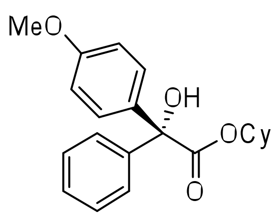
Enantioselective Rhodium(I)-Catalyzed Addition of Arylboronic Acids to α-Ketoesters

A solution of $[ClRh(ethylene)_2]_2$ (0.30 mg, 0.77 μmol) in degassed methylene chloride (200 μL) was added to a reaction vial with allene ligand **5** (1.9 mg, 1.6 μmol) under the protection of nitrogen. The resulting solution was stirred for 30min at room temperature under nitrogen. The methylene chloride solvent was then removed under vacuum and the residue was redissolved in degassed isopropanol (600 μL) under the protection of nitrogen. The catalyst solution was then transferred into another vial containing α-ketoester (77 μmol), arylboronic acid (154 μmol) and KF (154 μmol) under nitrogen. Degassed water (120 μL) was then added to the above reaction mixture. The resulting reaction solution was then stirred at room temperature (unless otherwise indicated) until reaction was complete as judged by TLC. The reaction mixture was concentrated under reduce pressure. The residue was purified by flash chromatography on silica gel with hexane and ethyl acetate as the eluent in the ratio of 20-10:1 to afford the addition products.

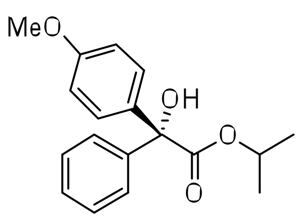
Absolute stereochemistry assigned by comparing HPLC data for Table 1, entry 1 to Zhou et al.[5] All others were assigned by analogy.



Benzyl 2-hydroxyl-2-(4-methoxyphenyl)-phenylacetate. (Table 1, entry 1) $[\alpha]^{25}_{D}$ = -1.5º (c = 0.5, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 3.83 (s, 3H), 4.21 (br s, 1H), 5.31 (s, 2H), 6.87 (d, J = 9.0 Hz, 2H), 7.23-7.45 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 55.6, 68.6, 81.0, 113.6, 127.7, 128.27, 128.32, 128.4, 128.7, 128.8, 129.0, 134.3, 135.1, 142.3, 159.5, 174.7; ESI MS m/z: 370.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AS-H column (25 cm × 0.46 cm ID), conditions: n-hexane/i-PrOH = 95:5, 1.0 mL/min, 230 nm; t_R = 22.7 min (minor) and 26.3 min (major).

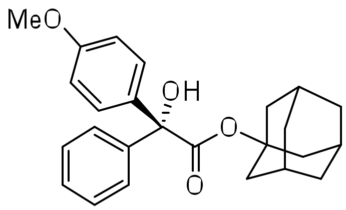


Cyclohexyl 2-hydroxyl-2-(4-methoxyphenyl)-phenylacetate. (Table 1, entry 9) $[\alpha]^{25}_{D}$ = -2.8 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ : 1.24-1.38 (m, 3H), 1.43-1.53 (m, 3H), 1.54-1.63 (m, 2H), 1.77-1.84 (m, 2H), 3.82 (s, 3H), 4.29 (br s, 1H), 4.96 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.86 (d, J = 9.0 Hz, 2H), 7.30-7.38 (m, 5H), 7.45 (d, J = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 25.4, 31.3, 55.5, 75.8, 80.7, 113.5, 127.6, 128.1, 128.2, 129.0, 134.6, 142.6, 159.4, 174.4; ESI MS m/z: 362.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AS-H column (25 cm × 0.46 cm ID), conditions: n-hexane/i-PrOH = 99.5: 0.5, 1.0 mL/min, 230 nm; t_R = 33.1 min (minor) and 37.3 min (major).

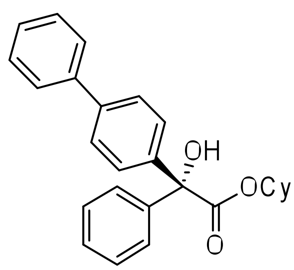


2-Propanyl 2-hydroxyl-2-(4-methoxyphenyl)-phenylacetate. (Table 1, entry 10) $[\alpha]^{25}_{D}$ = -3.0 º (c = 0.8, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26-1.28 (m, 6H), 3.83 (s, 3H), 4.28 (br s, 1H), 5.17 (quintet, J_1 = 6.0 Hz, 1H), 6.88 (d, J = 8.5 Hz, 2H), 7.28 (br s, 1H), 7.34-7.38 (m, 4H), 7.46 (d, J = 7.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.8, 55.5, 71.3, 80.7, 113.5, 127.6, 128.1, 128.2, 128.9, 134.5, 142.5, 159.4, 174.4; ESI MS m/z: 322.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AS-H column (25 cm × 0.46

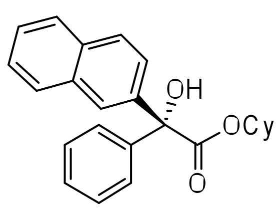
cm ID), conditions: *n*-hexane/*i*-PrOH = 99.5: 0.5, 1.0 mL/min, 230 nm; t_R = 27.9 min (minor) and 30.4 min (major).



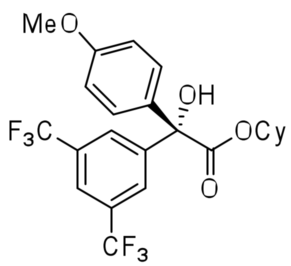
1-adamental 2-hydroxyl-2-(4-methoxyphenyl)-phenylacetate. (Table 1, entry 11) $[\alpha]^{25}_D$ = -2.0 º (c = 0.5, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.65 (br s, 6H), 2.09 (br s, 6H), 2.17 (s, 3H), 3.81 (s, 3H), 4.30 (br s, 1H), 6.86 (d, J_1 = 7.0 Hz, 2H), 7.31-7.37 (m, 5H), 7.45 (d, J = 6.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 31.1, 36.2, 41.3, 55.5, 80.7, 84.2, 113.5, 127.7, 127.9, 128.1, 129.0, 135.0, 143.0, 159.3, 173.5; ESI MS m/z: 414.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 95:5, 1.0 mL/min, 230 nm; t_R = 17.1 min (minor) and 20.1 min (major).



Cyclohexyl 2-hydroxyl-2-(4-phenyl-phenyl)-phenylacetate. (Table 2, entry 2) $[\alpha]^{25}_D$ = -2.0 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.42 (m, 3H), 1.46-1.68 (m, 5H), 1.80-1.89 (m, 2H), 4.40 (s, 1H), 5.01 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.34-7.42 (m, 3H), 7.46 (t, J = 7.5 Hz, 2H), 7.49-7.56 (m, 4H), 7.58-7.63 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 25.4, 31.3, 76.0, 80.9, 126.9, 127.4, 127.6, 127.7, 128.1, 128.2, 128.3, 129.0, 140.8, 140.9, 141.4, 142.4, 174.2; ESI MS m/z: 408.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99: 1, 1.0 mL/min, 230 nm; t_R = 17.4 min (major) and 20.6 min (minor).

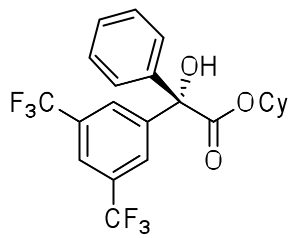


Cyclohexyl 2-hydroxyl-2-(naphthalen-2-yl)-phenylacetate. (Table 2, entry 3) ^1H NMR (500 MHz, $CDCl_3$) δ: 1.21-1.40 (m, 3H), 1.42-1.65 (m, 5H), 1.78-1.86 (m, 2H), 4.46 (s, 1H), 5.02 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.35-7.40 (m, 3H), 7.46-7.55 (m, 5H), 7.80-7.88 (m, 3H), 7.99 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 25.4, 31.4, 76.0, 81.2, 126.1, 126.4, 126.6, 127.7, 127.8, 127.9, 128.2, 128.3, 128.7, 133.0, 133.1, 139.6, 142.4, 174.2; ESI MS m/z: 382.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99: 1, 1.0 mL/min, 230 nm; t_R = 15.4 min (major) and 22.1 min (minor).

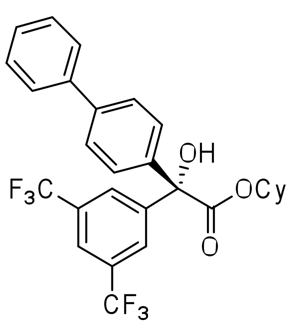


Cyclohexyl 2-(3,5-bistrifluoromethylphenyl)-2-hydroxyl-2-(4-methoxylphenyl) acetate. (Table 2, entry 4) $[\alpha]^{23}_D$ = 28.4 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.67 (m, 6H), 1.70-1.77 (m, 2H), 1.85-1.94 (m, 2H), 3.83 (s, 3H), 4.45 (s, 1H), 5.01 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.90 (d, J = 9.0 Hz, 2H), 7.26 (d, J = 9.0 Hz, 2H), 7.85 (s, 1H), 8.06 (s, 2H);

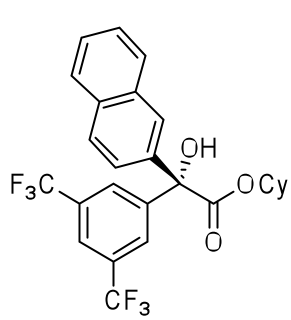
^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.2, 23.3, 25.3, 31.1, 31.3, 55.5, 76.7, 80.1, 114.2, 122.0 (m), 123.5 (q, J = 271.4 Hz), 128.2, 128.5, 131.4 (q, J = 33.1 Hz), 133.8, 144.8, 159.9, 173.0. ESI MS m/z: 498.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99: 1, 1.0 mL/min, 230 nm; t_R = 6.5 min (major) and 7.5 min (minor).



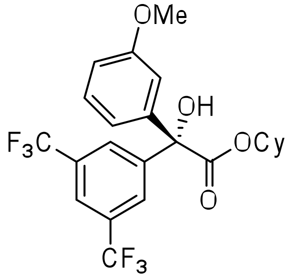
Cyclohexyl 2-(3,5-bistrifluoromethylphenyl)-2-hydroxy-phenylacetate. (Table 2, entry 5) $[\alpha]^{25}_D$ = 26.4 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.68 (m, 8H), 1.71-1.79 (m, 1H), 1.83-1.92 (m, 1H), 4.52 (br s, 1H), 5.02 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.31-7.40 (m, 5H), 7.86 (s, 1H), 8.07 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.2, 23.3, 25.2, 31.1, 31.3, 76.8, 80.3, 122.1, 123.5 (q, J = 271.4 Hz), 127.1, 128.2, 128.9, 131.4, (q, J = 33.3 Hz), 141.6, 144.5, 172.8; ESI MS m/z: 447.0, $[M+H]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99: 1, 1.0 mL/min, 230 nm; t_R = 4.3 min (major) and 5.5 min (minor).



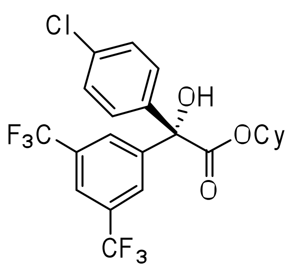
Cyclohexyl 2-(3,5-bistrifluoromethylphenyl)-2-hydroxy-2-(4-phenyl-phenyl) acetate. (Table 2, entry 6) $[\alpha]^{25}_D$ = 32.2 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.27-1.58 (m, 8H), 1.73-1.82 (m, 1H), 1.84-1.95 (m, 1H), 4.55 (br s, 1H), 5.04 (m, 1H), 7.38-7.42 (m, 3H), 7.47 (t, J = 6.5 Hz, 2H), 7.61 (d, J = 8.5 Hz, 4H), 7.88 (s, 1H), 8.11 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.3, 23.4, 25.3, 31.1, 31.3, 77.0, 80.2, 122.2, 123.5 (q, J = 271.5 Hz), 127.4, 127.6, 127.9, 128.2, 129.1, 131.5, (q, J = 33.3 Hz), 140.4, 140.6, 141.8, 144.5, 172.8; ESI MS m/z: 544.8, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99.5: 0.5, 1.0 mL/min, 230 nm; t_R = 11.8 min (minor) and 13.7 min (major).



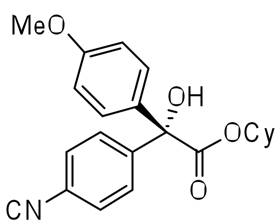
Cyclohexyl 2-hydroxy-2-(3,5-bistrifluoromethylphenyl)-2-(naphthalen-2-yl)acetate. (Table 2, entry 7) $[\alpha]^{25}_D$ = 50.8 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.27-1.63 (m, 8H), 1.73-1.82 (m, 1H), 1.87-1.93 (m, 1H), 4.59 (br s, 1H), 5.02 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.40 (d, J = 8.5 Hz, 1H), 7.53-7.55 (m, 2H), 7.82-7.88 (m, 5H), 8.10 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.2, 23.3, 25.3, 31.1, 31.3, 76.9, 80.5, 122.3, 123.5 (q, J = 271.4 Hz), 125.0, 126.20, 126.23, 126.9, 127.2, 127.9, 128.28, 128.3, 128.7, 128.9, 131.5 (q, J = 35.8 Hz), 133.0, 133.3, 138.8, 172.8; ESI MS m/z: 518.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99:1, 1.0 mL/min, 230 nm; t_R = 7.0 min (major) and 8.5 min (minor).



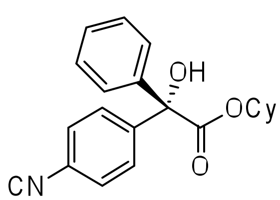
Cyclohexyl 2-(3,5-bistrifluoromethylphenyl)-2-hydroxyl-2-(3-methoxylphenyl)acetate. (Table 2, entry 8) $[\alpha]^{25}_{D}$ = 28.3 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.68 (m, 6H), 1.72-1.78 (m, 2H), 1.85-1.92 (m, 2H), 3.79 (s, 3H), 4.50 (br s, 1H), 5.02 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.87-6.94 (m, 3H), 7.30 (t, J = 8.0 Hz, 1H), 7.95 (s, 1H), 8.06 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.2, 23.3, 25.3, 31.1, 31.3, 55.5, 76.8, 80.2, 113.1, 114.2, 119.4, 122.1 (m), 123.5 (q, J = 271.4 Hz), 128.2, 129.9, 131.7 (q, J = 33.0 Hz), 143.0, 144.4, 160.0, 172.6; ESI MS m/z: 498.8, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99.5:0.5, 1.0 mL/min, 230 nm; t_R = 6.6 min (major) and 9.5 min (minor).



Cyclohexyl 2-(3,5-bistrifluoromethylphenyl)- 2-(4-chlorophenyl)-2-hydroxyacetate. (Table 2, entry 9) $[\alpha]^{25}_{D}$ = 20.5 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.68 (m, 8H), 1.73-1.80 (m, 1H), 1.82-1.91 (m, 1H), 4.53 (br s, 1H), 5.02 (m, 1H), 7.31 (d, J = 7.5 Hz, 2H), 7.37 (d, J = 7.5 Hz, 2H), 7.87 (s, 1H), 8.02 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.27, 23.34, 25.2, 31.2, 31.3, 77.2, 79.8, 122.3, 123.4 (q, J = 271.4 Hz), 128.0, 128.6, 129.0, 131.6 (q, J = 33.3 Hz), 135.0, 140.0, 144.2, 172.4; ESI MS m/z: 524.8, $[M+HCO_2]^+$; 462.8 $[M-OH]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99:1, 1.0 mL/min, 230 nm; t_R = 4.6 min (major) and 5.1 min (minor).

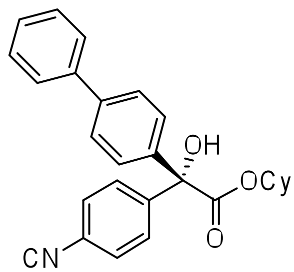


Cyclohexyl 2-(4-cynophenyl)-2-hydroxyl-2-(4-methoxyl phenyl) acetate. (Table 2, entry 10) $[\alpha]^{25}_{D}$ = 34.2 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.45-1.70 (m, 10H), 3.82 (s, 3H), 4.38 (br s, 1H), 4.97 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.88 (d, J = 8.0 Hz, 2H), 7.28 (, J = 8.0 Hz, 2H), 7.54-7.68 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 23.5, 25.3, 31.3, 31.4, 55.6, 76.4, 80.4, 111.9, 113.9, 119.0, 128.58, 128.63, 131.9, 133.9, 147.6, 159.7, 173.2. ESI MS m/z: 363.9, $[M-H]^-$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/EtOH = 90:10, 1.0 mL/min, 229 nm; t_R = 22.8 min (major) and 25.7 min (minor).

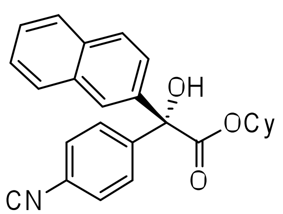


Cyclohexyl 2-(4-cynophenyl)-2-hydroxyl-phenylacetate. (Table 2, entry 11) $[\alpha]^{25}_{D}$ = 38.4 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.23-1.64 (m, 8H), 1.77 (br m, 1H), 1.85 (br m, 1H), 4.42 (br s, 1H), 4.99 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.37 (br m, 5H), 7.63 (br m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.39, 23.43, 25.3, 31.28, 31.34, 76.6, 80.7, 112.0, 118.9, 127.3, 128.60, 128.64, 128.67, 131.9, 141.7, 147.3, 173.1; ESI MS m/z: 333.9, $[M-H]^-$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46

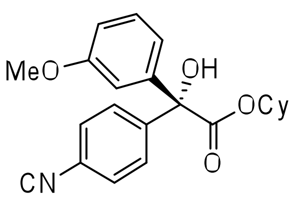
cm ID), conditions: *n*-hexane/*i*-PrOH = 90:10, 1.0 mL/min, 229 nm; t_R = 14.2 min (major) and 15.2 min (minor).



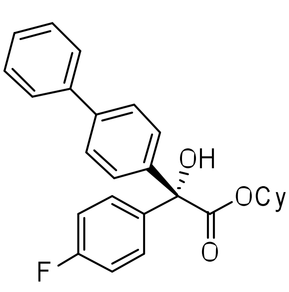
Cyclohexyl 2-(4-cynophenyl)-2-hydroxyl-2-(4-phenyl-phenyl) acetate. (Table 2, entry 12) $[\alpha]^{25}_D$ = 26.2 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ : 1.21-1.66 (m, 8H), 1.72-1.92 (m, 2H), 4.45 (br s, 1H), 5.01 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.36 (t, J = 7.5 Hz, 1H), 7.44-7.48 (m, 4H), 7.60 (d, J = 8.0 Hz, 4H), 7.65-7.68 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 23.5, 25.3, 31.3, 31.4, 76.7, 80.6, 112.1, 118.9, 127.2, 127.3, 127.8, 127.9, 128.6, 129.1, 132.0, 140.5, 140.7, 141.5, 147.3, 173.0; ESI MS m/z: 410.0, $[M-H]^-$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90:10, 1.0 mL/min, 229 nm; t_R = 20.0 min (minor) and 24.5 min (major).



Cyclohexyl 2-hydroxyl-2-(4-cynophenyl)-2-(naphthalen-2-yl)acetate. (Table 2, entry 13) $[\alpha]^{25}_D$ = 44.8 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.23-1.67 (m, 8H), 1.80 (br m, 1H), 1.88 (br m, 1H), 4.52 (br s, 1H), 5.04 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.44 (dd, J_1 = 1.5 Hz, J_2 = 8.5Hz, 1H), 7.51-7.53 (m, 2H), 7.65 (br m, 4H), 7.81-7.86 (m, 3H), 7.88 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 23.5, 25.3, 31.3, 31.4, 76.7, 80.9, 112.1, 118.9, 125.4, 126.2, 127.0, 128.5, 128.6, 128.7, 132.0, 132.9, 133.2, 138.9, 147.2, 173.0; ESI MS m/z: 383.9, $[M-H]^-$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90:10, 1.0 mL/min, 229 nm; t_R = 26.8 min (minor) and 32.1 min (major).

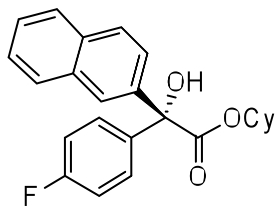


Cyclohexyl 2-(4-cynophenyl)-2-hydroxyl-2-(3-methoxyl phenyl) acetate. (Table 2, entry 14) $[\alpha]^{25}_D$ = 31.8 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ : 1.22-1.44 (m, 4H), 1.47-1.66 (m, 4H), 1.73-1.81 (m, 1H), 1.83-1.91(m, 1H), 3.78 (s, 3H), 4.39 (s, 1H), 4.98 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.89 (d, J =9.0 Hz, 1H), 6.96 (d, J = 7.0 Hz, 2H), 7.28 (d, J = 7.0 Hz, 2H), 7.60-7.64 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 23.5, 25.3, 31.3, 31.4, 55.5, 76.6, 80.6, 112.0, 113.2, 114.0, 118.9, 119.6, 128.6, 129.6, 131.9, 143.1, 147.2, 159.8, 172.9; ESI MS m/z: 363.9, $[M-H]^-$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90:10, 1.0 mL/min, 229 nm; t_R = 18.9 min (major) and 28.6 min (minor).

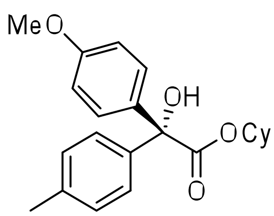


Cyclohexyl 2-hydroxyl-2-(4-fluorophenyl)-2-(4-phenyl-phenyl)acetate. (Table 2, entry 15) $[\alpha]^{23}_D$ = 14.5 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.42 (m, 3H), 1.44-1.65 (m, 5H), 1.78-1.89 (m, 2H), 4.39 (s, 1H), 4.99 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.05 (t, J = 9.0 Hz, 2H), 7.37 (t, J = 7.5 Hz, 1H), 7.44-7.52 (m, 6H), 7.57-7.63 (m, 4H); ^{13}C NMR (125 MHz,

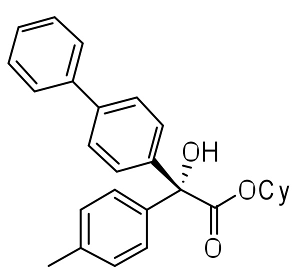
$CDCl_3$) δ: 23.4, 25.4, 31.3, 31.4, 76.1, 80.5, 105.0, 115.1 (d, J = 21.3 Hz), 127.1, 127.4, 127.7, 128.0, 129.0, 129.5, 129.6, 140.7, 141.1, 141.3, 174.0; ESI MS m/z: 426.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 95:5, 1.0 mL/min, 254 nm; t_R = 15.1 min (minor) and 18.7 min (major).



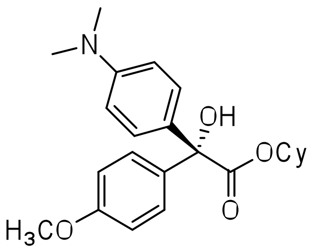
Cyclohexyl 2-hydroxy-2-(4-fluorophenyl)-2-(naphthalen-2-yl)acetate. (Table 2, entry 16) $[\alpha]^{25}_D$ = 17.6 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.23-1.42 (m, 3H), 1.45-1.55 (m, 3H), 1.57-1.66 (m, 2H), 1.84 (br s, 2H), 4.50 (br s, 1H), 5.02 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.05 (t, J = 8.0 Hz, 2H), 7.47-7.52 (m, 5H), 7.82-7.86 (m, 3H), 7.96 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.45, 23.46, 25.4, 31.36, 31.38, 76.2, 80.8, 115.1 (d, J = 21.4 Hz), 125.9, 126.3, 126.5, 126.7, 127.8, 128.1, 128.7, 129.7 (d, J = 8.0 Hz), 133.1 (d, J = 19.9 Hz), 128.1, 139.6, 162.7 (d, J = 245.6Hz), 174.0; ESI MS m/z: 400.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99:1, 1.0 mL/min, 230 nm; t_R = 11.6 min (minor) and 12.3 min (major).



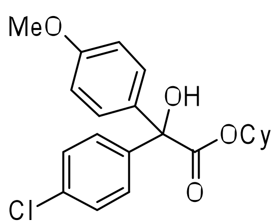
Cyclohexyl 2-hydroxyl-2-(4-methoxylphenyl)-2-*p*-tolylacetate. (Table 2, entry 17) $[\alpha]^{25}_D$ = -7.6 º (c = 0.5, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.39 (m, 3H), 1.42-1.52 (m, 3H), 1.56-1.65 (m, 2H), 1.77-1.86 (m, 2H), 2.36 (s, 3H), 3.82 (s, 3H), 4.25 (br s, 1H), 4.95 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.86 (d, J = 7.5 Hz, 2H), 7.15 (d, J = 7.5 Hz, 2H), 7.33 (d, J = 7.0 Hz, 2H), 7.38 (d, J = 7.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.3, 23.5, 25.4, 31.4, 55.5, 75.7, 80.6, 113.5, 127.5, 128.9, 129.0, 134.8, 137.8, 139.8, 159.3, 174.5; ESI MS m/z: 376.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 98:2, 1.0 mL/min, 230 nm; t_R = 32.4 min (major) and 35.5 min (minor).



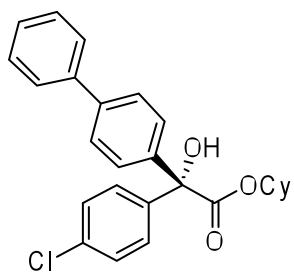
Cyclohexyl 2-hydroxyl-2-(4-phenyl-phenyl)-2-(p-tolyl)acetate. (Table 2, entry 18) $[\alpha]^{25}_D$ = -6.5 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.23-1.43 (m, 3H), 1.46-1.56 (m, 3H), 1.58-1.68 (m, 2H), 1.80-1.88 (m, 2H), 2.38 (s, 3H), 4.35 (br s, 1H), 5.00 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.18 (d, J = 8.0 Hz, 2H), 7.36-7.40 (m, 3H), 7.46 (t, J = 7.5 Hz, 2H), 7.54 (d, J = 7.5 Hz, 2H), 7.58 (d, J = 8.5 Hz, 2H), 7.62 (d, J = 7.5 Hz, 2H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.3, 23.5, 25.4, 31.4, 75.9, 80.8, 126.9, 127.3, 127.57, 127.6, 128.1, 129.0, 137.9, 139.6, 140.8, 140.9, 141.6, 174.2; ESI MS m/z: 422.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99:1, 1.0 mL/min, 230 nm; t_R = 39.9 min (minor) and 44.9 min (major).



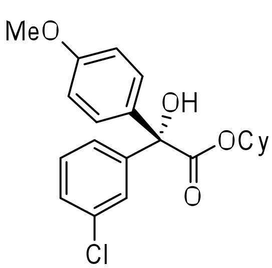
Cyclohexyl 2-hydroxyl-2-(4-*N,N*-dimethylphenyl)-2-(4-methoxylphenyl)acetate. (Table 2, entry 19) $[\alpha]^{25}_{D}$ = 8.4 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.39 (m, 3H), 1.41-1.52 (m, 3H), 1.56-1.65 (m, 2H), 1.75-1.86 (m, 2H), 2.96 (s, 6H), 3.81 (s, 3H), 4.18 (br s, 1H), 4.93 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.68 (d, J = 7.5 Hz, 2H), 6.85 (d, J = 8.5 Hz, 2H), 7.28 (d, J = 7.5 Hz, 2H), 7.38 (d, J = 9.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.5, 23.6, 25.5, 31.38, 31.44, 40.8, 55.5, 75.5, 80.5, 112.0, 113.3, 128.5, 129.0, 135.1, 150.2, 159.2, 174.8, ESI MS m/z: 384.0, $[M+H]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 97:3, 1.0 mL/min, 230 nm; t_R = 53.8 min (major) and 59.5 min (minor).



Cyclohexyl 2-(4-chlorophenyl)-2-hydroxyl-2-(4-methoxyl phenyl) acetate. (Table 2, entry 20) $[\alpha]^{25}_{D}$ = 9.2 º (c, 1.0, $CHCl_3$); (Table 2, entry 21) $[\alpha]^{25}_{D}$ = -6.5 º (c = 0.3, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.22-1.40 (m, 3H), 1.41-1.54 (m, 3H), 1.54-1.65 (m, 2H), 1.75-1.87 (m, 2H), 3.82 (s, 3H), 4.31 (br s, 1H), 4.95 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.87 (d, J = 8.0 Hz, 2H), 7.29-7.32 (m, 4H), 7.41 (d, J = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.4, 23.5, 25.4, 31.3, 31.4, 55.5, 76.1, 80.3, 113.7, 128.3, 128.8, 129.2, 134.0, 134.4, 141.1, 159.5, 174.0; ESI MS m/z: 396.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 95:5, 1.0 mL/min, 230 nm; t_R = 16 min and 17 min.

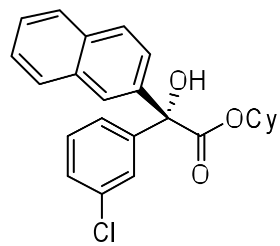


Cyclohexyl 2-(4-cynophenyl)-2-hydroxyl-2-(4-phenyl-phenyl)acetate. (Table 2, entry 22) $[\alpha]^{25}_{D}$ = 18.4 º (c = 0.5, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.42 (m, 3H), 1.42-1.55 (m, 3H), 1.55-1.66 (m, 2H), 1.78-1.90 (m, 2H), 4.39 (br s, 1H), 4.99 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 7.33-7.38 (m, 3H), 7.44-7.49 (m, 6H), 7.58-7.61 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.5, 25.4, 31.3, 31.4, 76.3, 80.5, 127.1, 127.4, 127.7, 128.0, 128.4, 129.1, 129.2, 134.2, 140.7, 140.8, 141.2, 173.8; ESI MS m/z: 442.8, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 95:5, 0.5 mL/min, 229 nm; t_R = 16.5 min (minor) and 19.0 min (major).

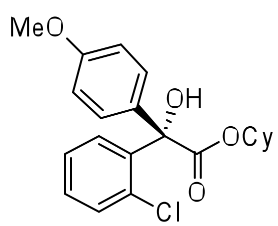


Cyclohexyl 2-(3-chlorophenyl)-2-hydroxyl-2-(4-methoxyl phenyl)acetate. (Table 2, entry 23) $[\alpha]^{25}_{D}$ = 14.2 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.66 (m, 8H), 1.75-1.86 (m, 2H), 2.82 (s, 3H), 4.34 (br s, 1H), 4.97 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.88 (d, J = 8.5 Hz, 2H), 7.27-7.38 (m, 5H), 7.50 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.37, 23.42, 25.4, 31.3, 31.4, 55.5, 76.1, 80.3, 113.7, 126.0, 128.0, 128.2, 128.8, 129.4, 134.16, 134.18, 144.5, 159.6, 173.8; ESI MS m/z: 396.9, $[M+Na]^+$.

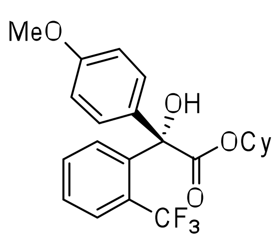
The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 98:2, 1.0 mL/min, 229 nm; t_R = 28.4 min (minor) and 30.6 min (major).



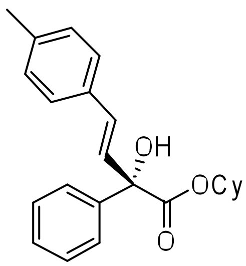
Cyclohexyl 2-hydroxyl-2-(3-chlorophenyl)-2-(naphthalen-2-yl)acetate. (Table 2, entry 24) $[\alpha]^{25}_D$ = 25.0 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.24-1.41 (m, 4H), 1.42-1.65 (m, 4H), 1.79-1.88 (m, 2H), 4.47 (br s, 1H), 5.02 (m, 1H), 7.27-7.33 (m, 3H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.46-7.55 (m, 3H), 7.81-7.87 (m, 3H), 7.92 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.38, 23.42, 25.4, 31.3, 31.4, 76.3, 80.8, 125.7, 126.1, 126.3, 126.6, 126.8, 127.8, 128.1, 128.2, 128.4, 128.7, 129.5, 133.0, 133.2, 134.3, 139.2, 144.2, 173.6; ESI MS m/z: 416.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 98:2, 1.0 mL/min, 229 nm; t_R = 20.3 min (major) and 28.7 min (minor).



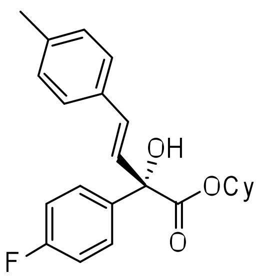
Cyclohexyl 2-hydroxyl-2-(2-chlorophenyl)-2-(4-methoxyl phenyl)acetate. (Table 2, entry 25) The reaction was run as general procedure but at 40ºC. $[\alpha]^{25}_D$ = 35.2 º (c = 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.16-1.39 (m, 3H), 1.41-1.67 (m, 3H), 1.56-1.67 (m, 2H), 1.78-1.84 (m, 1H), 1.86-1.94 (m, 1H), 3.86 (s, 3H), 4.36 (br s, 1H), 4.96 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.87 (d, *J* = 7.5 Hz, 1H), 6.95 (d, *J* = 8.5 Hz, 2H), 7.09 (t, *J* = 7.0 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.63 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.76, 23.81, 25.4, 31.36, 31.41, 55.6, 75.9, 80.2, 113.7, 126.4, 128.6, 129.7, 130.9, 131.0, 131.8, 134.5, 140.4, 159.8, 174.0; ESI MS m/z: 396.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel OD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 95:5, 1.0 mL/min, 230 nm; t_R = 6.8 min (minor) and 9.0 min (major).



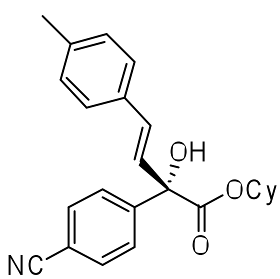
Cyclohexyl 2-hydroxyl-2-(2-trifluoromethylphenyl)-2-(4-methoxylphenyl)acetate. (Table 2, entry 26) $[\alpha]^{25}_D$ = 52.0 º (c = 0.5, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ: 1.17-1.42 (m, 4H), 1.44-1.65 (m, 4H), 1.76-1.82 (m, 1H), 1.85-1.92 (m, 1H), 3.86 (s, 3H), 4.03 (br s, 1H), 4.98 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 9.0 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.47 (d, *J* = 9.0 Hz, 2H), 7.77 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 23.5, 23.6, 25.4, 31.2, 31.5, 55.6, 75.6, 81.2, 113.9, 124.8 (q, *J* = 271.9 Hz), 128.4, 128.5, 131.0, 131.8, 134.4, 140.7, 159.8, 173.4; ESI MS m/z: 430.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90:10, 1.0 mL/min, 229 nm; t_R = 10.2 min (major) and 14.5 min (minor).



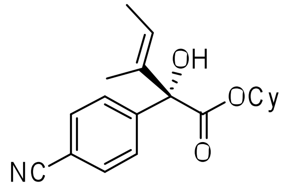
Cyclohexyl 2-hydroxy-2-phenyl-4-*p*-tolylbut-3-enoate. (Table 2, entry 27) $[\alpha]^{25}_{D}$ = 24.6 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.65 (m, 7H), 1.67-1.90 (m, 3H), 2.35 (s, 3H), 4.08 (br s, 1H), 4.91 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.67 (d, J = 16.0 Hz, 1H), 6.93 (s, J = 16.0 Hz, 1H), 7.15 (d, J = 7.5 Hz, 2H), 7.28-7.38 (m, 5H), 7.58 (d, J = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.5, 23.5, 23.6, 25.4, 31.3, 31.5, 75.8, 78.5, 126.4, 126.9, 128.2, 128.5, 128.6, 129.5, 130.4, 133.9, 138.0, 142.0, 174.0; ESI MS m/z: 372.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AS-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99.5:0.5, 1.0 mL/min, 230 nm; t_R = 15.7 min (major) and 23.6 min (minor).



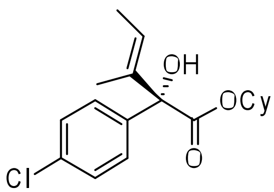
Cyclohexyl 2-hydroxy-2-4-(fluorophenyl)-4-*p*-tolylbut-3-enoate. (Table 2, entry 28) $[\alpha]^{25}_{D}$ = 38.2 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26-1.47 (m, 4H), 1.48-1.64 (m, 3H), 1.66-1.80 (m, 2H), 1.87 (br m, 1H), 2.36 (s, 3H), 4.12 (s, 1H), 4.92 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.64 (d, J = 15.5Hz, 1H), 6.92 (d, J = 15.5 Hz, 1H), 7.05 (t, J = 9.0 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 7.55-7.59 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.5, 23.5, 23.6, 25.4, 31.3, 31.5, 75.9, 78.0, 115.3, 115.4, 127.0, 128.30, 128.36, 128.43, 129.6, 130.7, 133.7, 137.8 (d, J = 3.1 Hz), 138.2, 162.6 (d, J = 245.4 Hz), 173.8; ESI MS m/z: 390.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AS-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99.5:0.5, 1.0 mL/min, 230 nm; t_R = 15.4 min (major) and 19.6 min (minor).



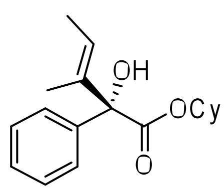
Cyclohexyl 2-hydroxy-2-(4-cynophenyl)-4-*p*-tolylbut-3-enoate. (Table 2, entry 29) $[\alpha]^{25}_{D}$ = 26.6 º (c = 0.75, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26-1.44 (m, 4H), 1.50-1.62 (m, 3H), 1.66-1.79 (m, 2H), 1.85-1.92 (m, 1H), 2.36 (s, 3H), 4.18 (s, 1H), 4.92 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 6.59 (d, J = 16.0 Hz, 1H), 6.90 (d, J = 16.0 Hz, 1H), 7.16 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 7.67 (d, J = 8.5 Hz, 2H), 7.76 (d, J = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.5, 23.5, 23.6, 25.3, 31.3, 31.5, 76.5, 78.1, 112.0, 118.9, 127.0, 127.4, 127.5, 129.6, 131.4, 132.3, 133.4, 138.5, 147.1, 173.0; ESI MS m/z: 397.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AS-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 97:3, 1.0 mL/min, 254 nm; t_R = 17.1 min (major) and 20.3 min (minor).



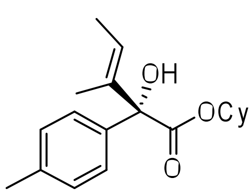
Cyclohexyl 2-hydroxy-2-(4-cynophenyl)-3-methylpent-3-enoate. (Table 2, entry 30) $[\alpha]^{25}_{D}$ = 33.5 º (c = 0.5, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.45 (m, 4H), 1.47-1.58 (m, 3H), 1.61 (s, 3H), 1.66 (d, *J* = 6.5 Hz, 3H), 1.66-1.74 (m, 2H), 1.86-1.92 (m, 1H), 3.96 (s, 1H), 4.91 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 5.37 (q, *J* = 6.5 Hz, 1H), 7.64 (d, *J* = 8.5 Hz, 2H), 7.71 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 13.2, 13.9, 23.5, 23.6, 25.4, 31.3, 31.5, 76.1, 82.6, 111.7, 119.1, 124.56, 124.60, 128.4, 131.8, 136.6, 146.7, 173.2; ESI MS m/z: 313.9, $[M+H]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/EtOH = 90:10, 1.0 mL/min, 230 nm; t_R = 8.2 min (minor) and 8.5 min (major).



Cyclohexyl 2-hydroxy-2-(4-chlorophenyl)-3-methylpent-3-enoate. (Table 2, entry 31) $[\alpha]^{23}_{D}$ = 27.1 º (c = 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.25-1.45 (m, 5H), 1.46-1.77 (m, 10H), 1.86-1.92 (m, 1H), 3.93 (s, 1H), 4.90 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 5.38 (q, *J* = 6.5 Hz, 1H), 7.30 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 13.4, 13.9, 23.5, 23.6, 25.4, 31.3, 31.6, 75.8, 82.4, 124.2, 124.3, 128.1, 129.0, 133.7, 133.8, 138.8, 174.0; ESI MS m/z: 344.9, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 99:1, 1.0 mL/min, 229 nm; t_R = 8.1 min (minor) and 8.9 min (major).



Cyclohexyl 2-hydroxy-2-phenyl-3-methylpent-3-enoate. (Table 2, entry 32) $[\alpha]^{25}_{D}$ = 7.8 º (c = 0.8, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26-1.47 (m, 4H), 1.47-1.62 (m, 3H), 1.65 (d, *J* = 6.5 Hz, 3H), 1.67 (s, 3H), 1.67-1.80 (m, 2H), 1.86-1.92 (m, 1H), 3.91 (s, 1H), 4.92 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 5.41 (q, *J* = 6.5 Hz, 1H), 7.27-7.31 (m, 3H), 7.57 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 13.6, 13.8, 23.5, 23.6, 25.5, 31.3, 31.6, 75.4, 82.8, 124.11, 124.15, 127.5, 127.8, 128.0, 136.9, 140.4, 174.4; ESI MS m/z: 311.0, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/EtOH = 90:10, 0.5 mL/min, 229 nm; t_R = 4.8 min (minor) and 5.1 min (major).



Cyclohexyl 2-hydroxy-2-*p*-tolyl-3-methylpent-3-enoate. (Table 2, entry 33) $[\alpha]^{25}_{D}$ = 4.0 º (c = 0.4, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.26-1.59 (m, 7H), 1.64 (d, *J* = 6.5 Hz, 3H), 1.67 (s, 3H), 1.69-1.82 (m, 2H), 1.87-1.93 (m, 1H), 2.36 (s, 3H), 3.88 (s, 1H), 4.91 (tt, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 1H), 5.41 (q, *J* = 6.5 Hz, 1H), 7.15 (d, *J* = 7.5 Hz, 2H), 7.44 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 13.6, 13.8, 21.3, 23.6, 23.7, 25.5, 31.4, 31.6, 75.4, 82.7, 124.0, 124.1, 127.3, 128.7, 137.0, 137.4, 137.5, 174.5; ESI MS m/z: 325.0, $[M+Na]^+$. The ee was determined by HPLC analysis using Daicel Chiralcel AD-H column (25 cm × 0.46 cm ID), conditions: *n*-hexane/EtOH = 90:10, 1.0 mL/min, 229 nm; t_R = 5.2 min (minor) and 5.4 min (major).

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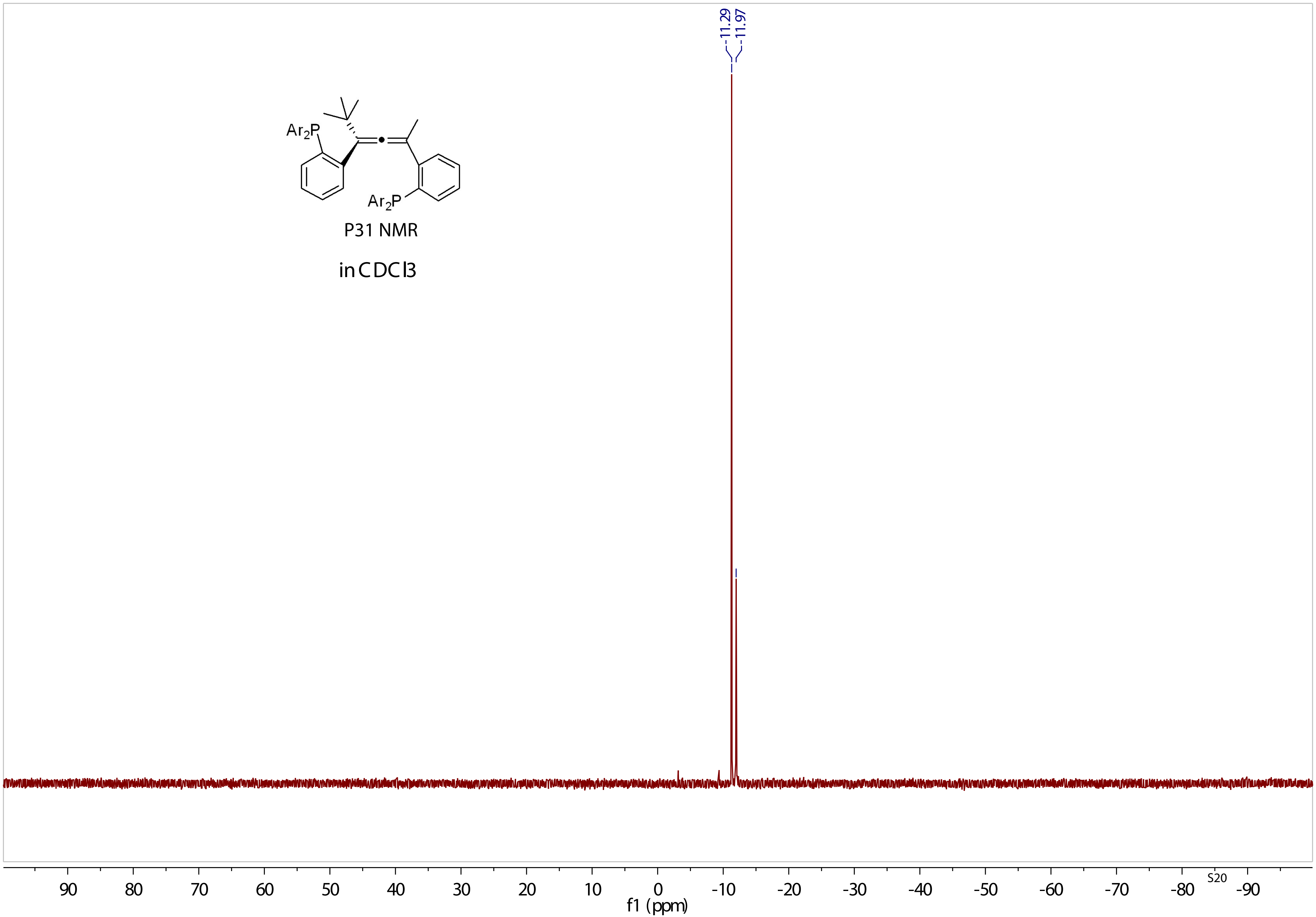

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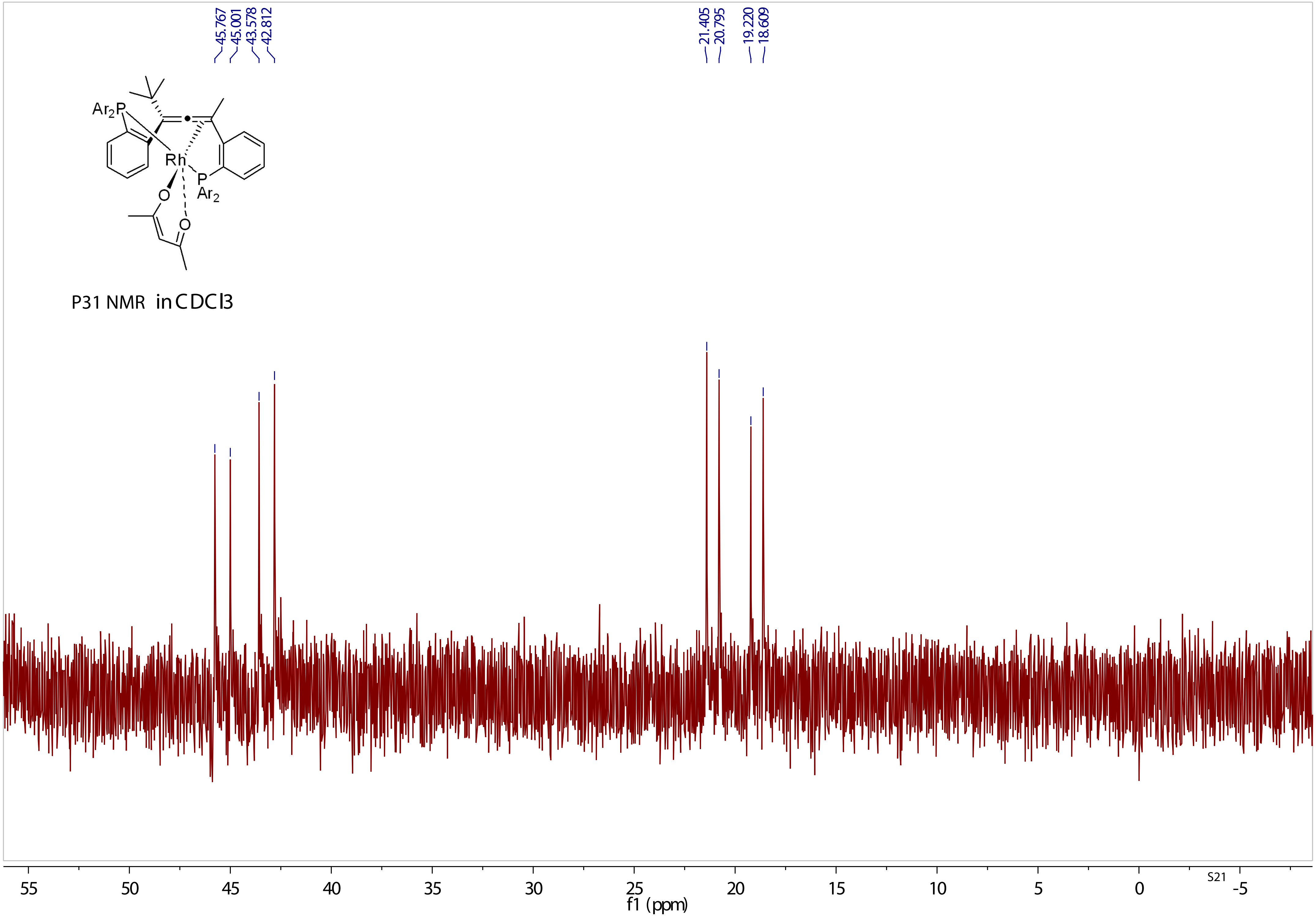
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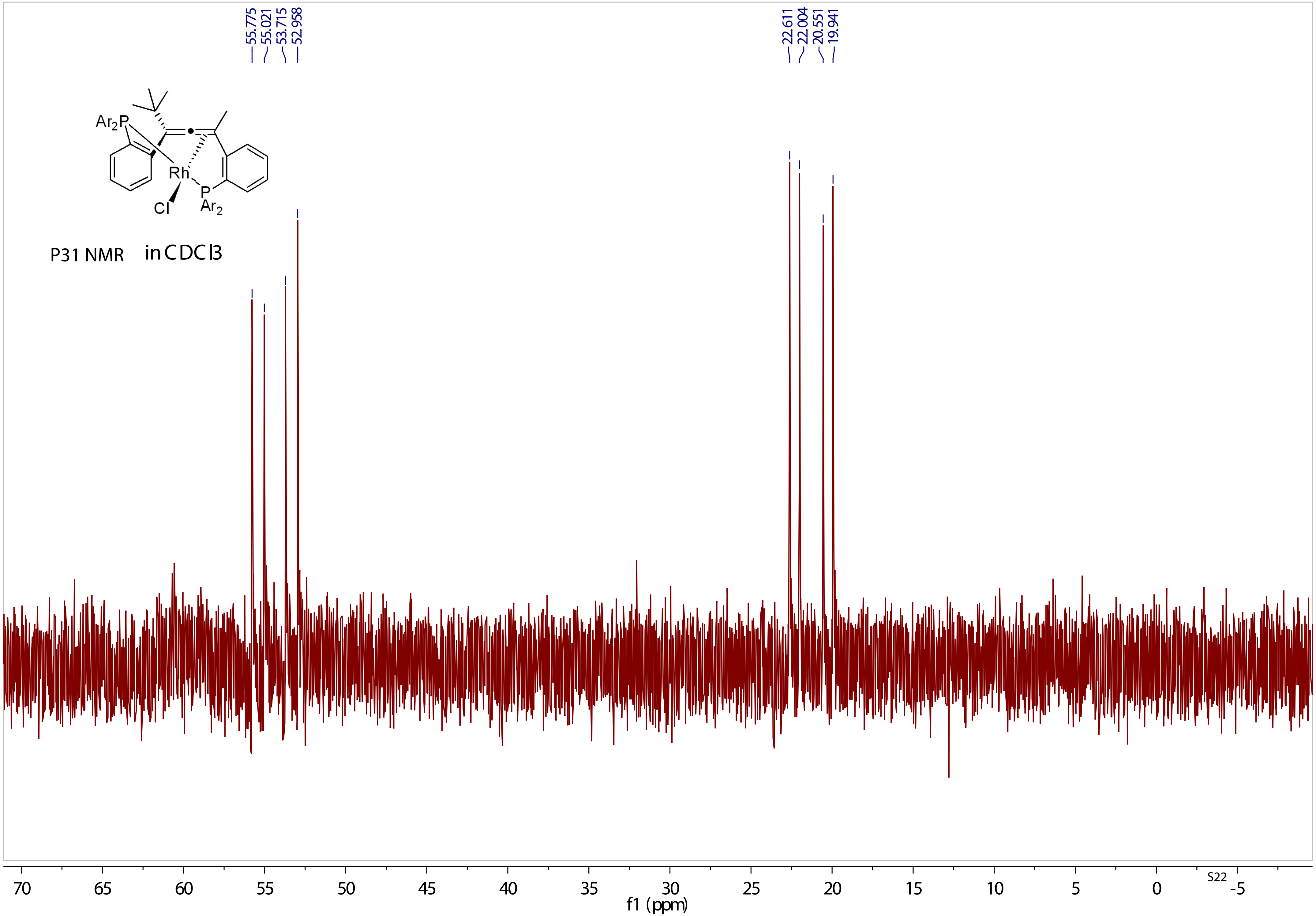
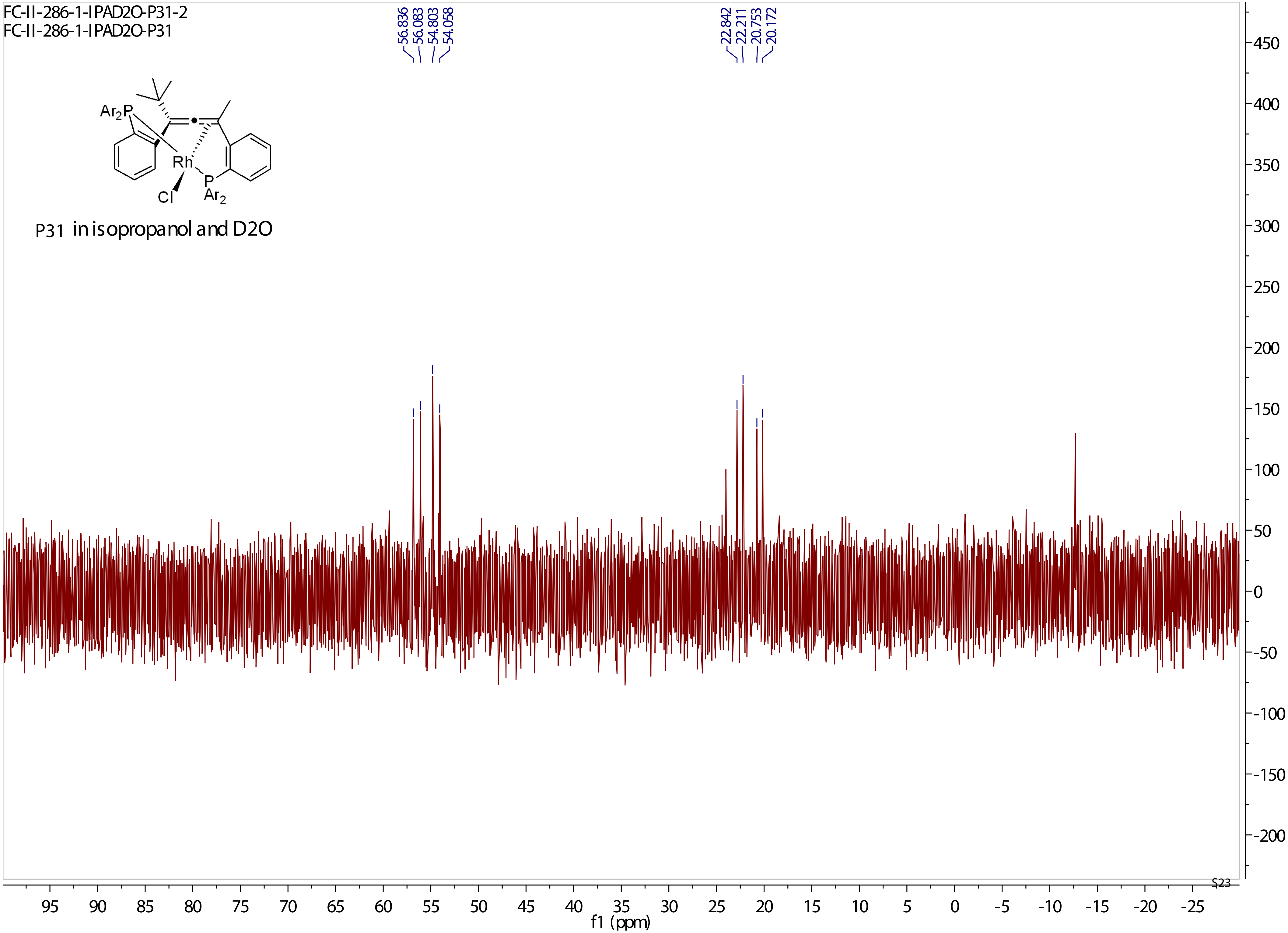
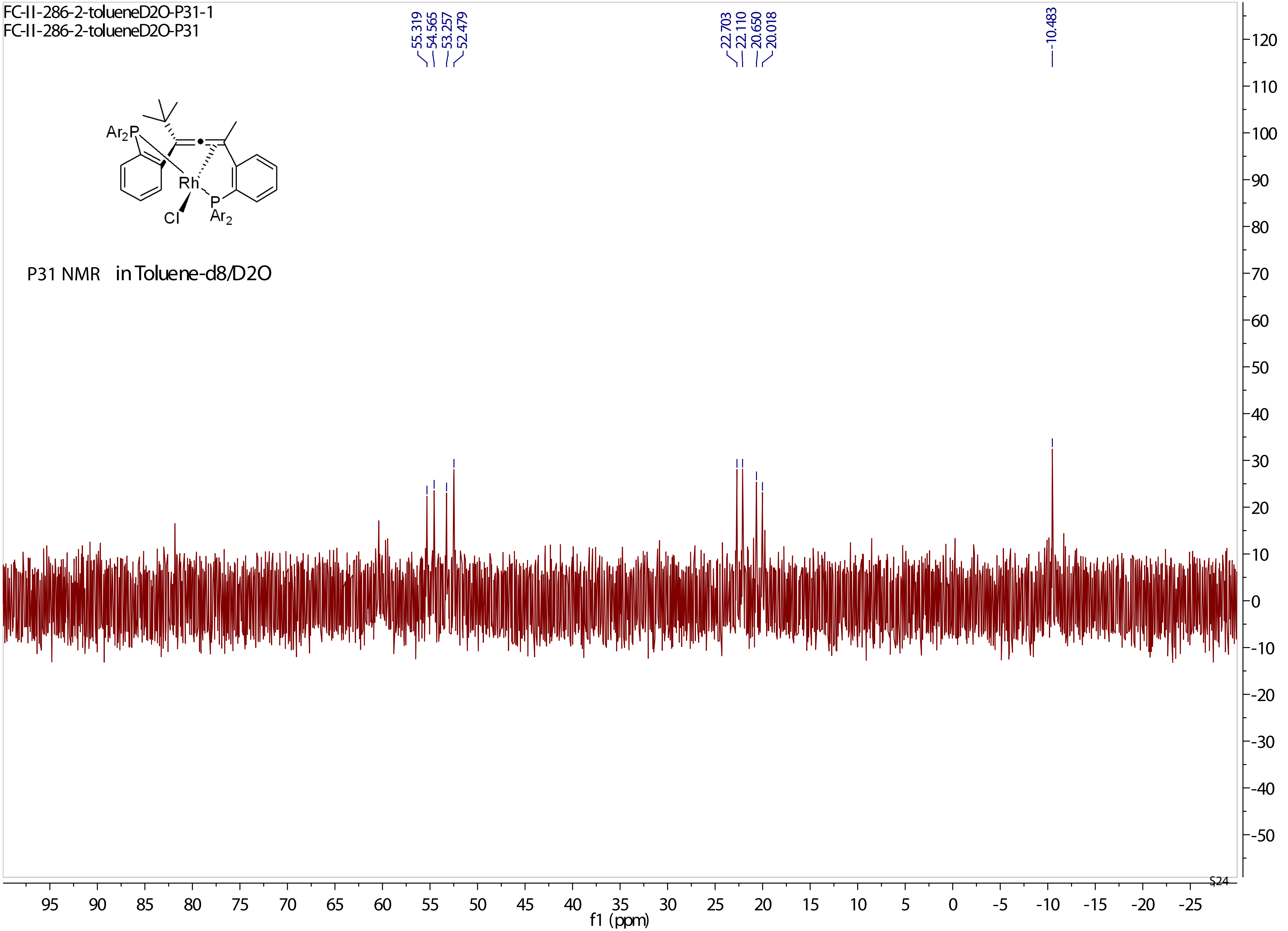
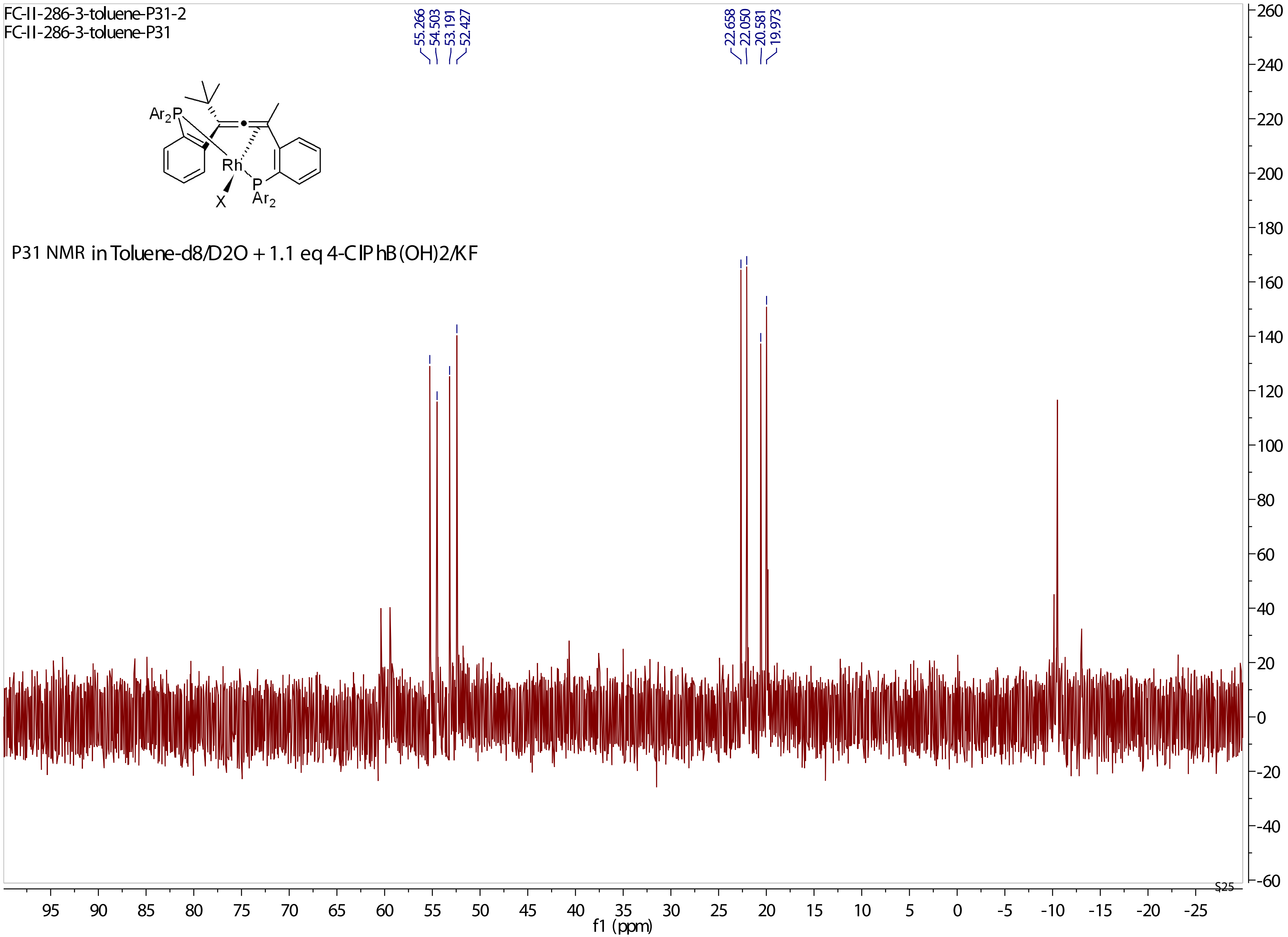

55.775
55.021
53.715
52.958
22.611
22.004
20.551
19.941
Ar2P
Rh
Cl
P
Ar2
P31 NMR in CDCl3
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5
f1 (ppm)



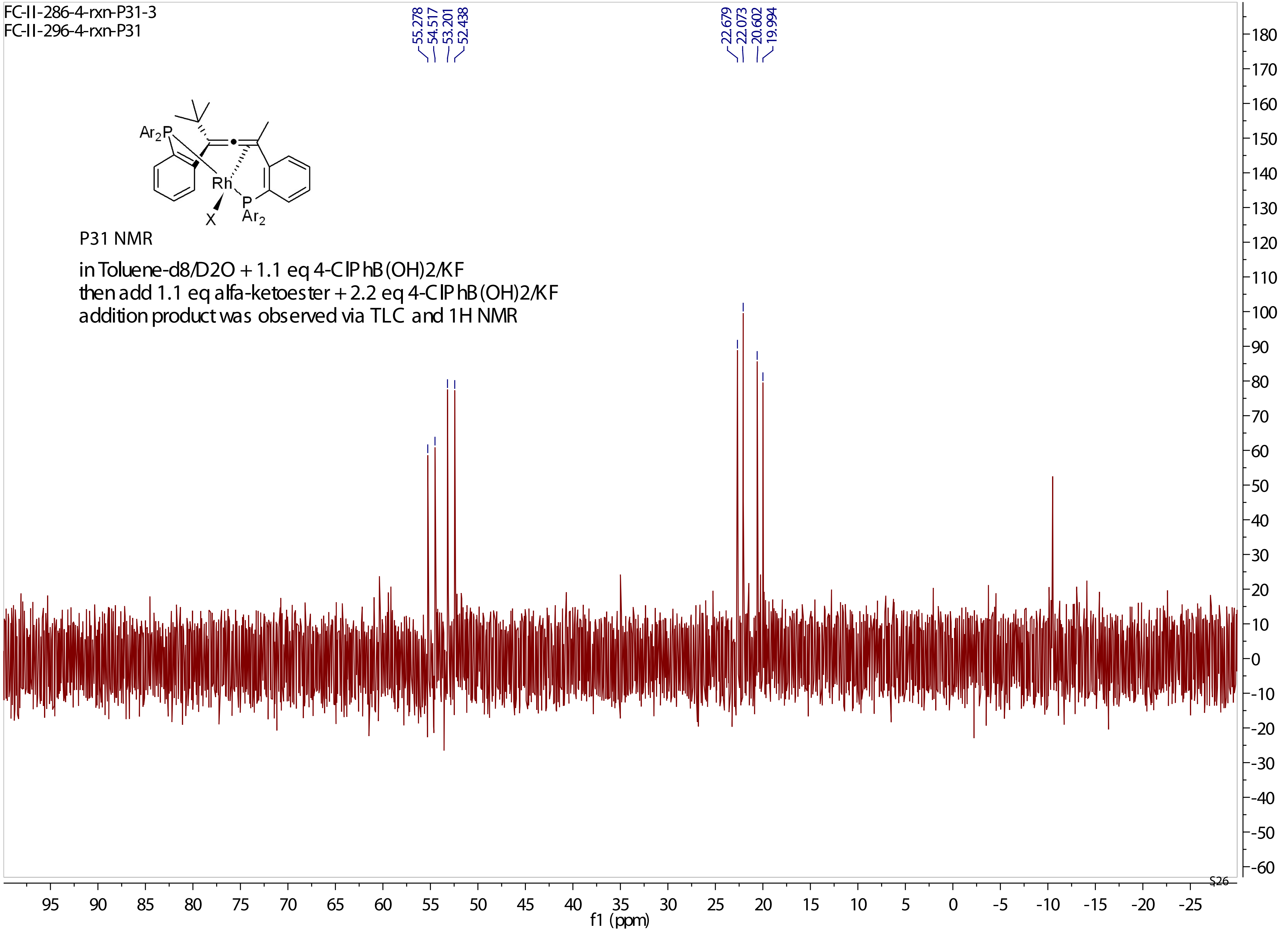
FC-II-286-1-IPAD2O-P31-2
FC-II-286-1-IPAD2O-P31
56.836
56.083
54.803
54.058
22.842
22.211
20.753
20.172
Ar2P
Rh
Cl
P
Ar2
P31 in isopropanol and D2O
f1 (ppm)

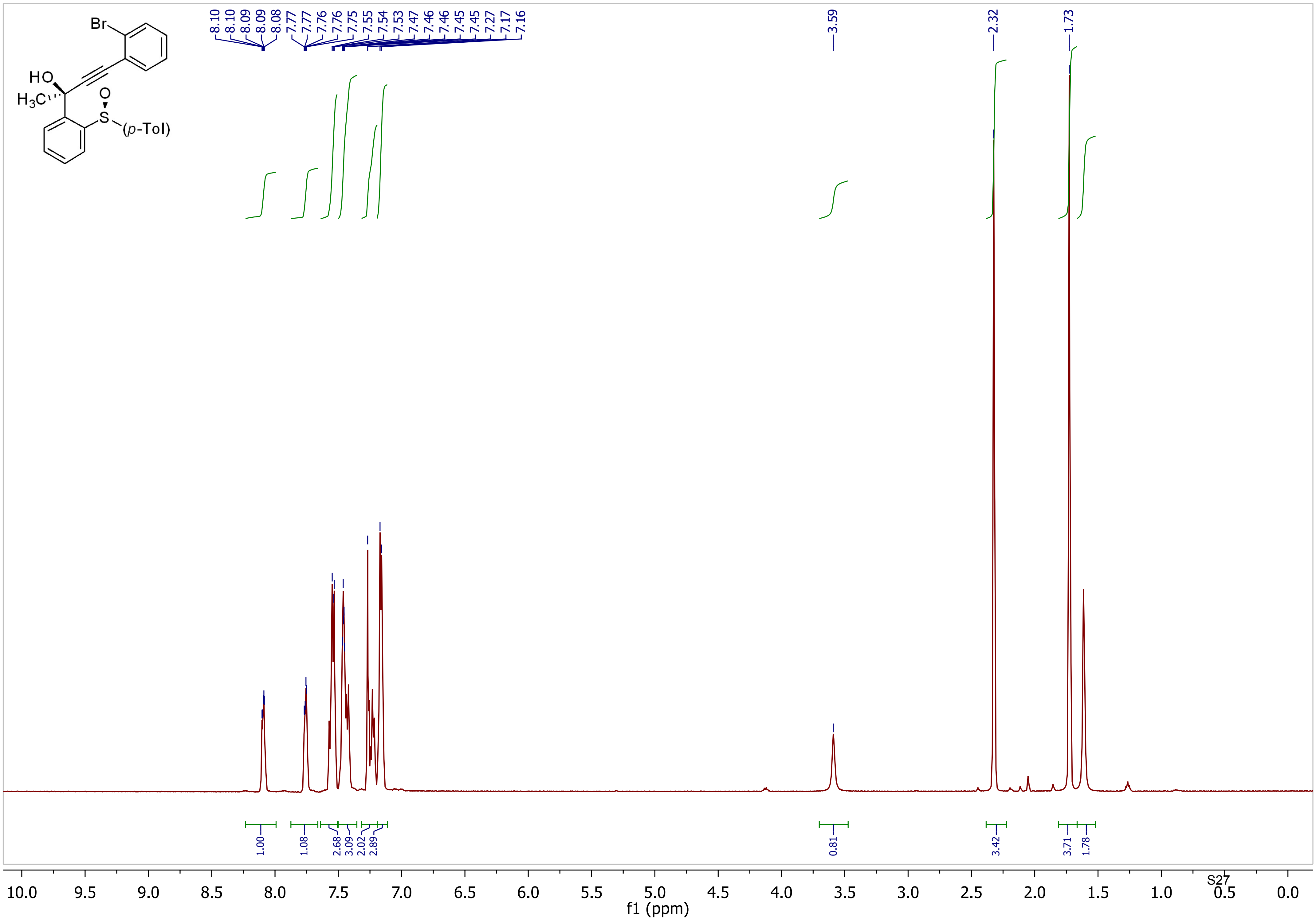


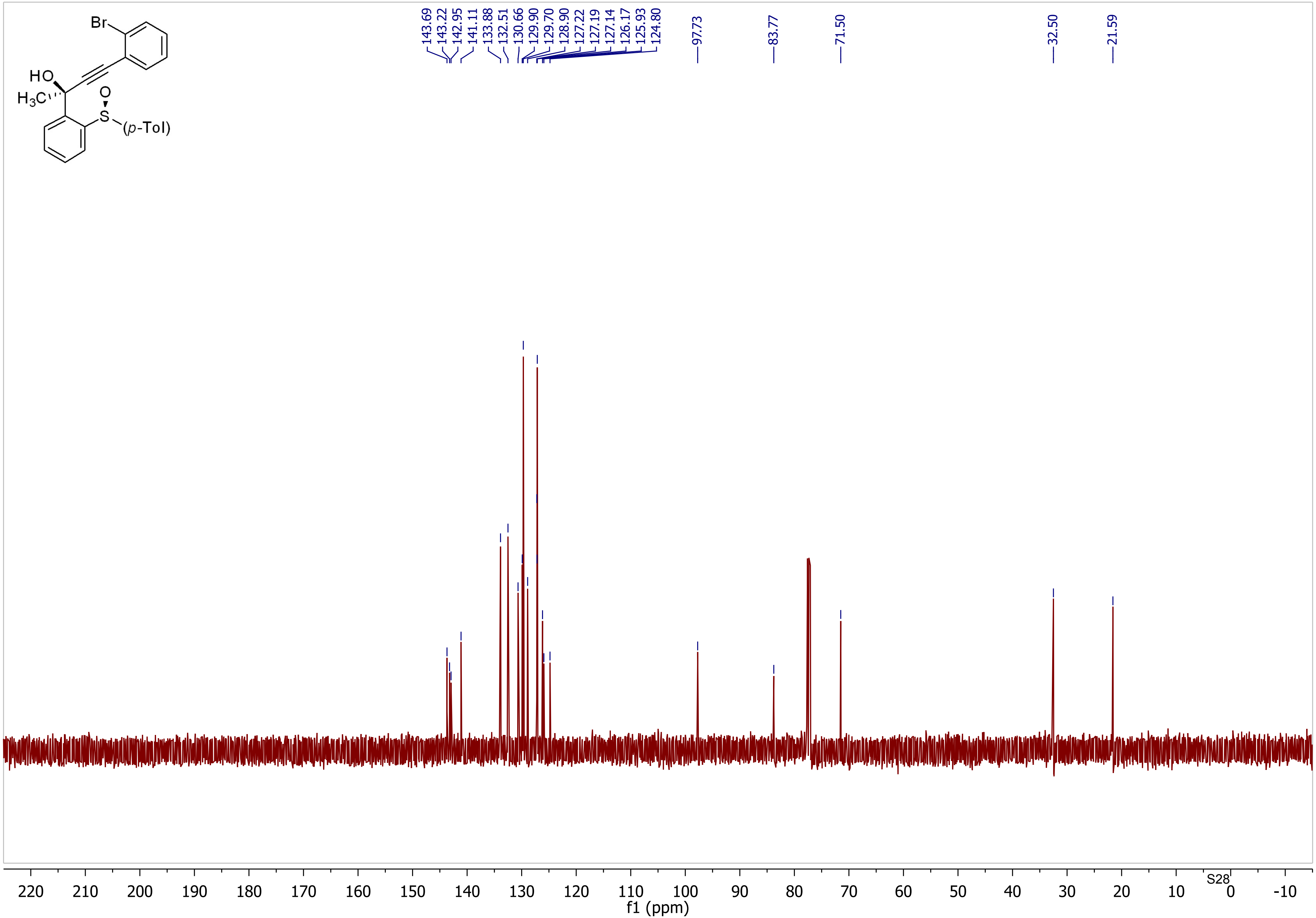
FC-II-286-2-tolueneD2O-P31-1
FC-II-286-2-tolueneD2O-P31
55.319
54.565
53.257
52.479
22.703
22.110
20.650
20.018
-10.483
Ar2P
Rh
Cl
P
Ar2
P31 NMR in Toluene-d8/D2O
f1 (ppm)

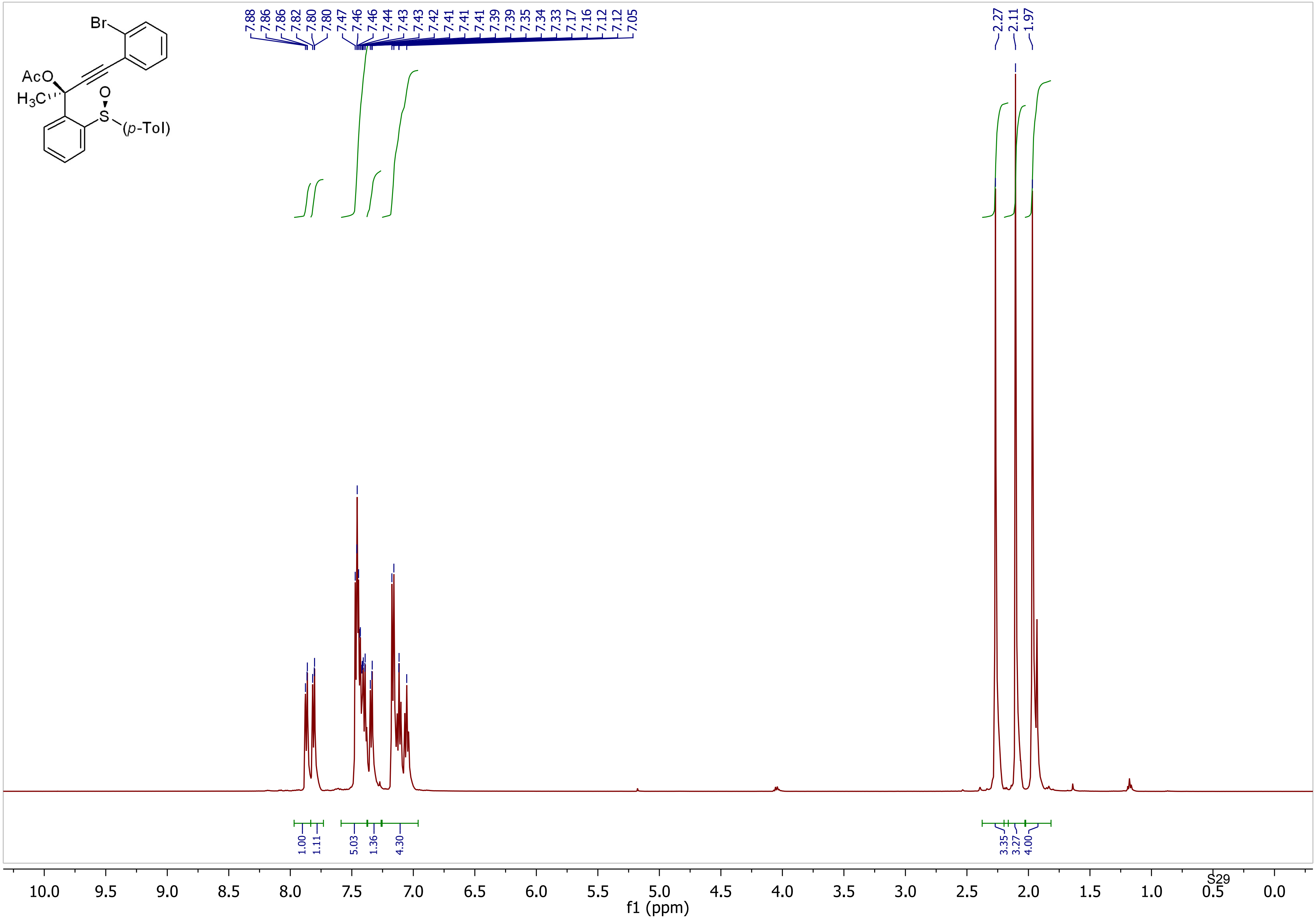


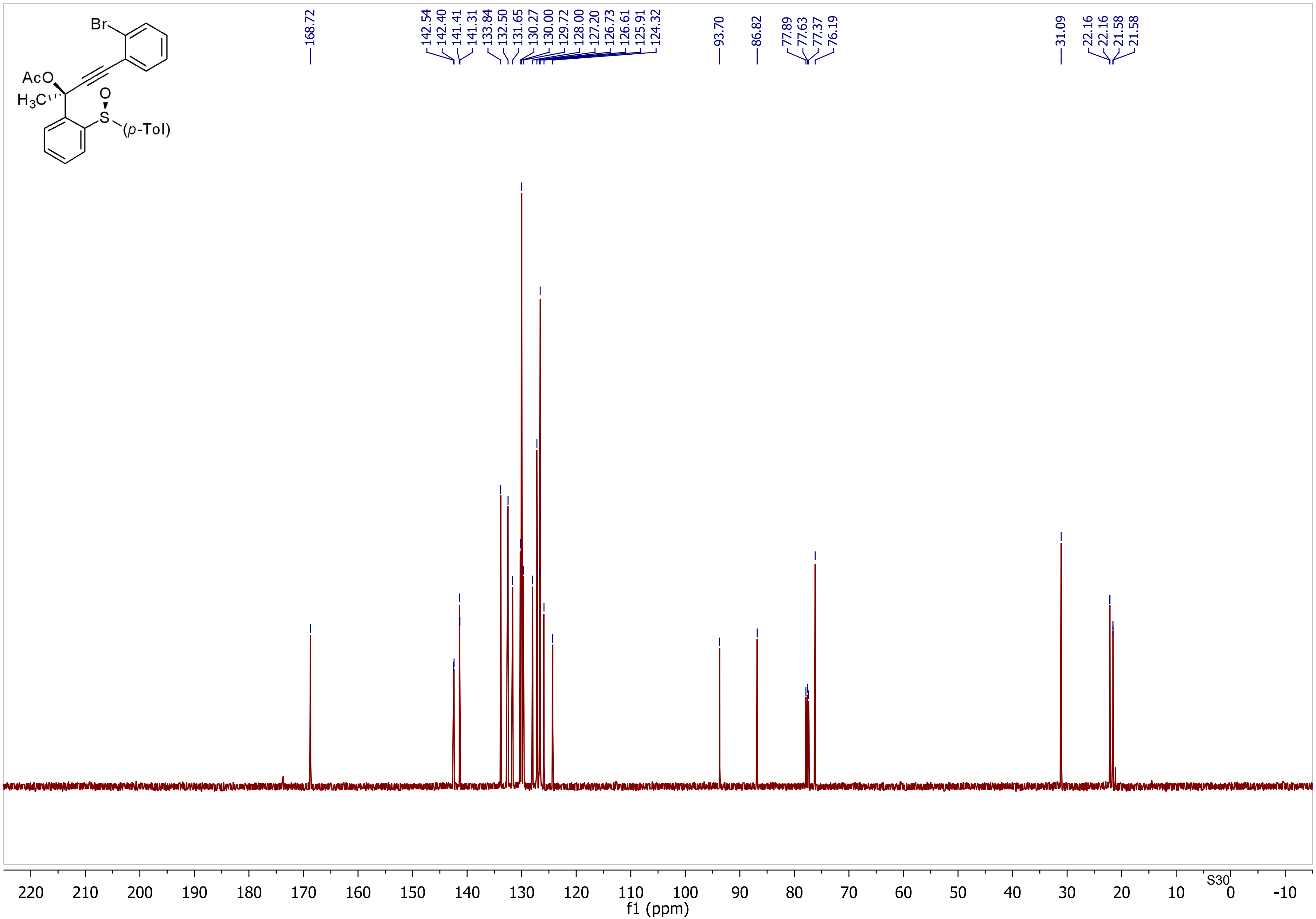
FC-II-286-3-toluene-P31-2
FC-II-286-3-toluene-P31
55.266
54.503
53.191
52.427
22.658
22.050
20.581
19.973
Ar2P
Rh
X
P
Ar2
P31 NMR in Toluene-d8/D2O + 1.1 eq 4-ClPhB(OH)2/KF
f1 (ppm)

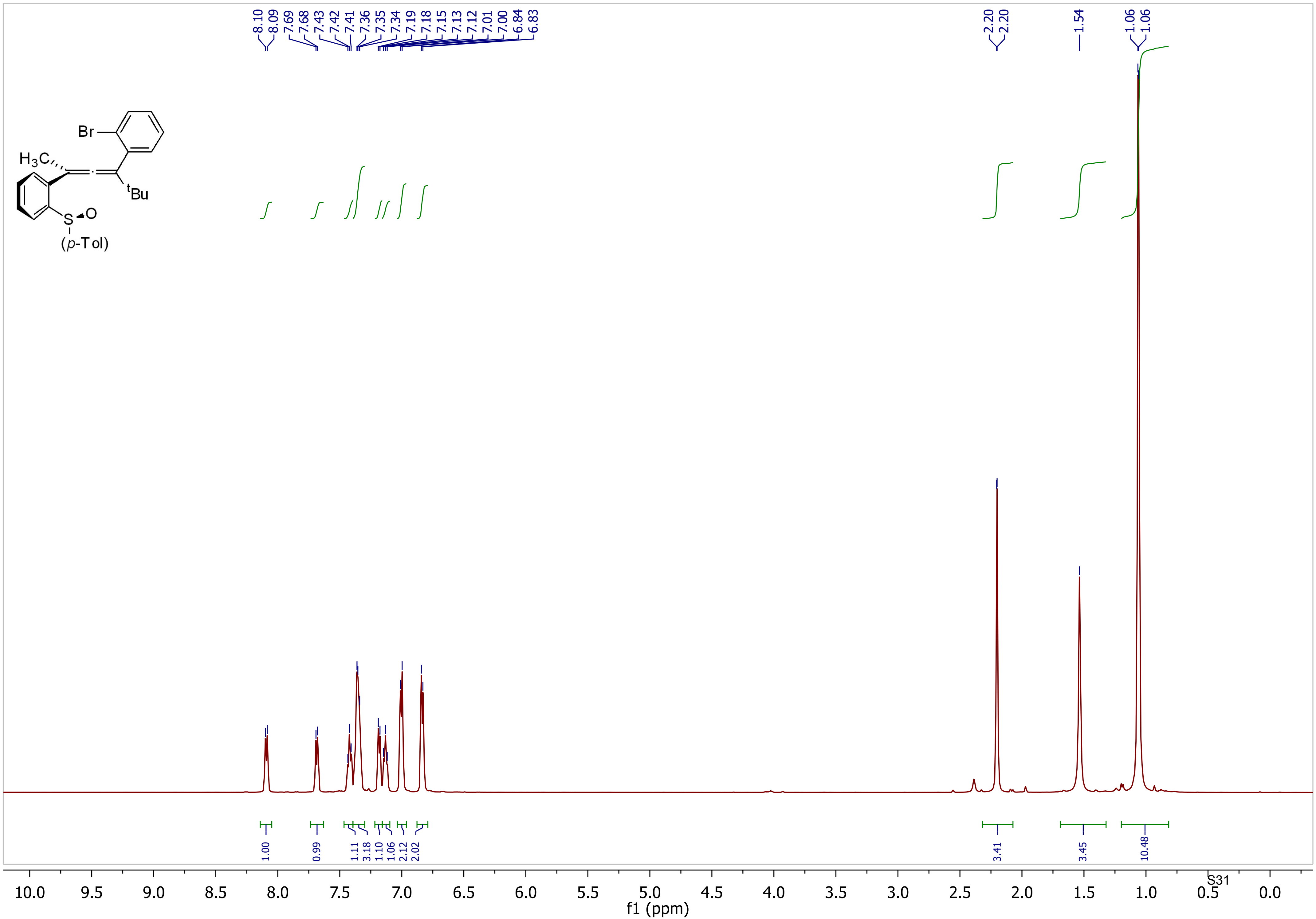

FC-II-286-4-rxn-P31-3
FC-II-296-4-rxn-P31
55.278
54.517
53.201
52.438
22.679
22.073
20.602
19.994
Ar2P
Rh
X
P
Ar2
P31 NMR
in Toluene-d8/D2O + 1.1 eq 4-ClPhB(OH)2/KF
then add 1.1 eq alfa-ketoester + 2.2 eq 4-ClPhB(OH)2/KF
addition product was observed via TLC and 1H NMR
f1 (ppm)


Br
HO
H3C
O
S
(p-Tol)
8.10
8.10
8.09
8.09
8.08
7.77
7.77
7.76
7.76
7.75
7.55
7.54
7.53
7.47
7.46
7.46
7.45
7.45
7.27
7.17
7.16
3.59
2.32
1.73
1.00
1.08
2.68
3.09
2.02
2.89
0.81
3.42
3.71
1.78
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
f1 (ppm)

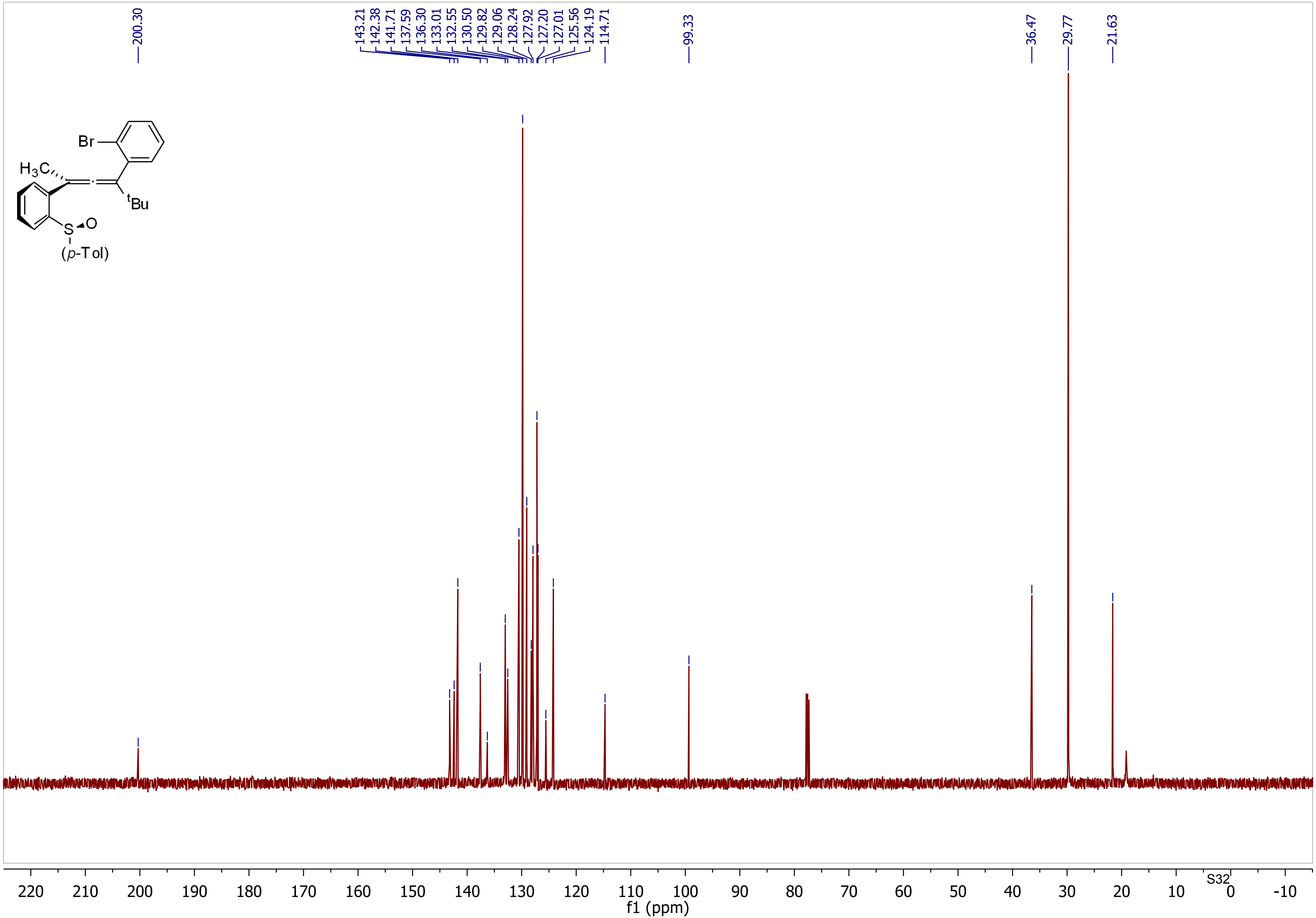

143.69
143.22
142.95
141.11
133.88
132.51
130.66
129.90
129.70
128.90
127.22
127.19
127.14
126.17
125.93
124.80
97.73
83.77
71.50
32.50
21.59
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Br
AcO
H3C
O
S
(p-Tol)
7.88 7.86 7.86 7.82 7.80 7.80 7.47 7.46 7.46 7.44 7.43 7.43 7.42 7.41 7.41 7.41 7.39 7.39 7.35 7.34 7.33 7.17 7.16 7.12 7.12 7.05
2.27 2.11 1.97
1.00 1.11 5.03 1.36 4.30
3.35 3.27 4.00
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
f1 (ppm)

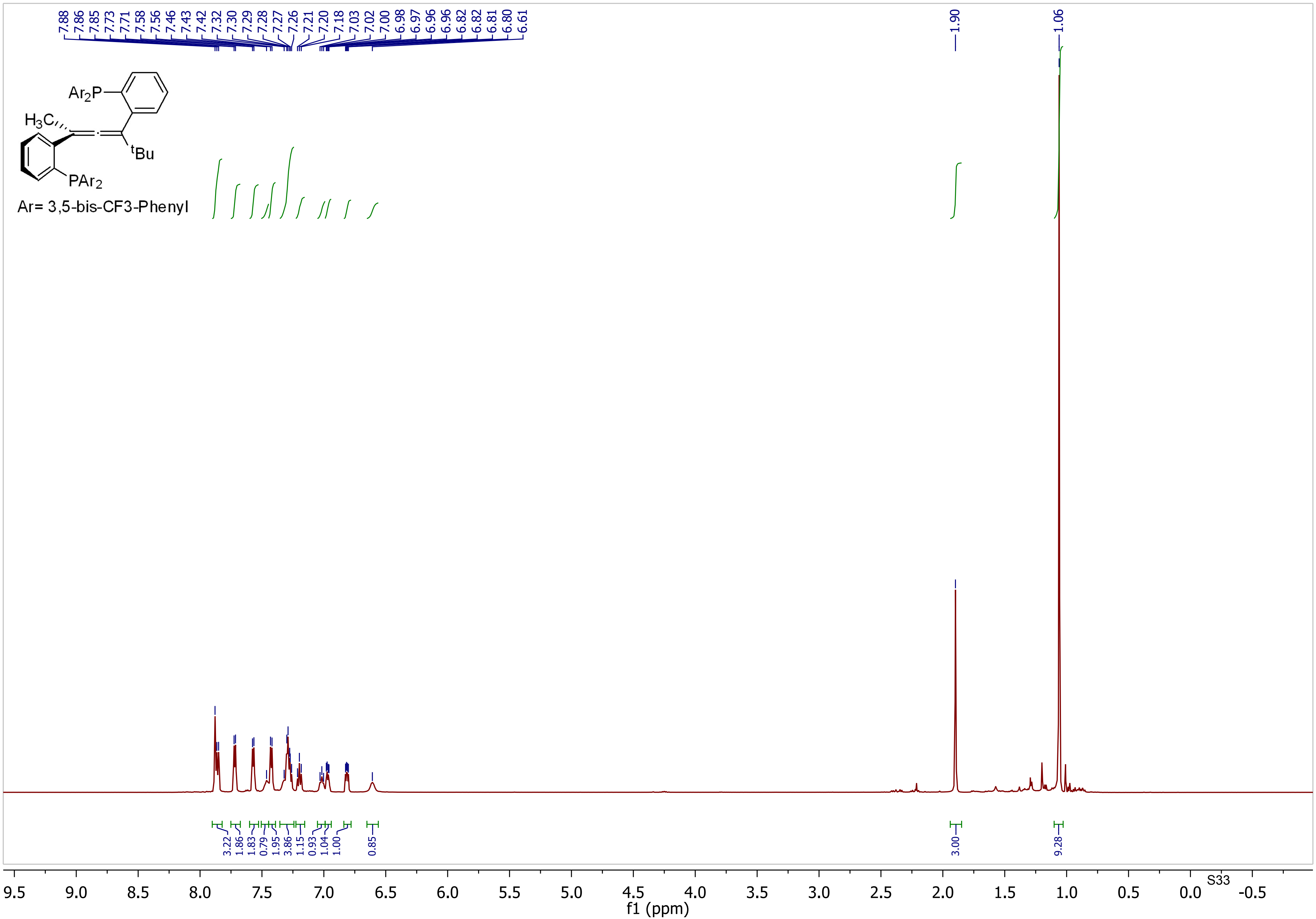

Br
AcO
H3C
O
S
(p-Tol)
168.72
142.54
142.40
141.41
141.31
133.84
132.50
131.65
130.27
130.00
129.72
128.00
127.20
126.73
126.61
125.91
124.32
93.70
86.82
77.89
77.63
77.37
76.19
31.09
22.16
22.16
21.58
21.58
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

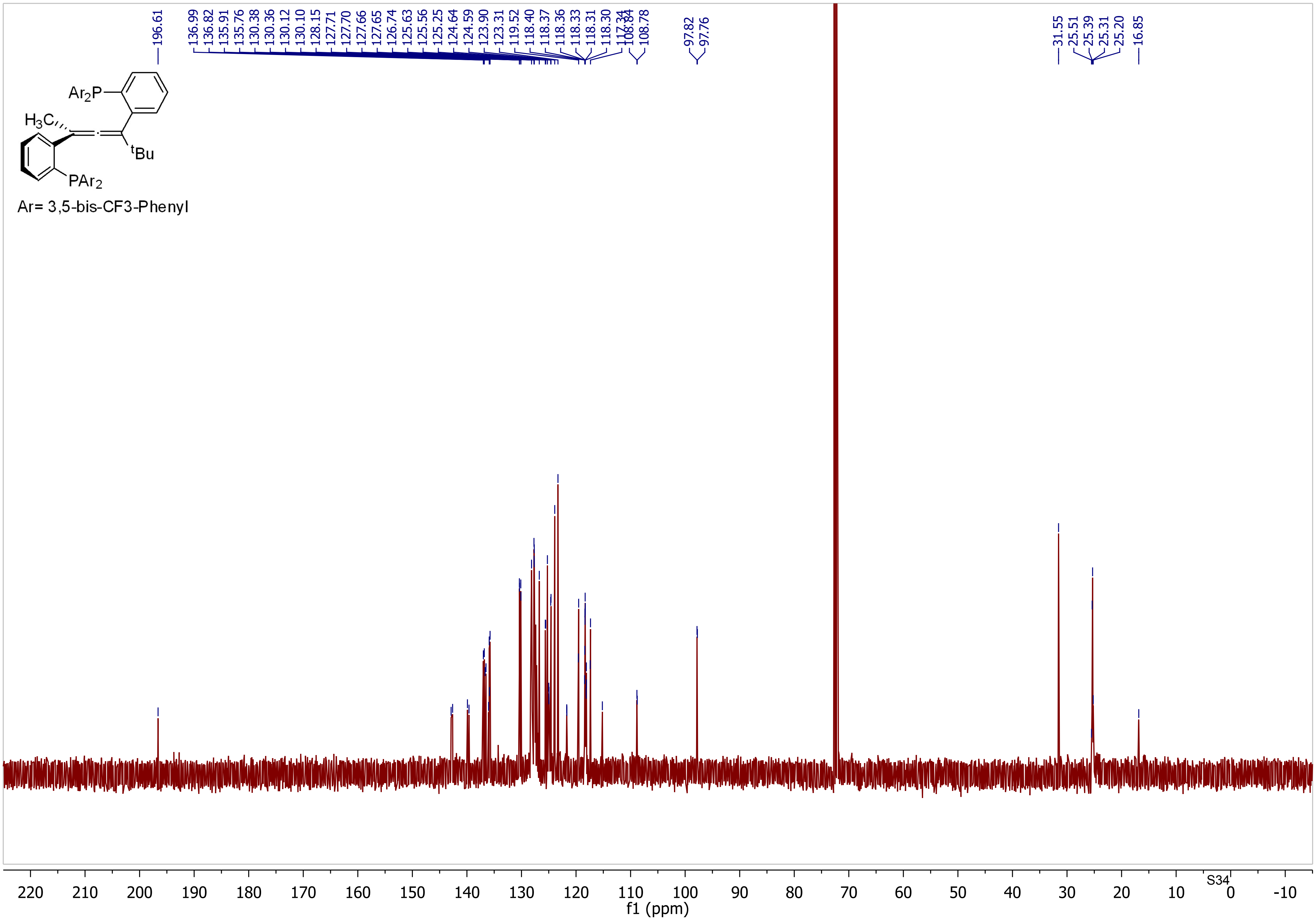


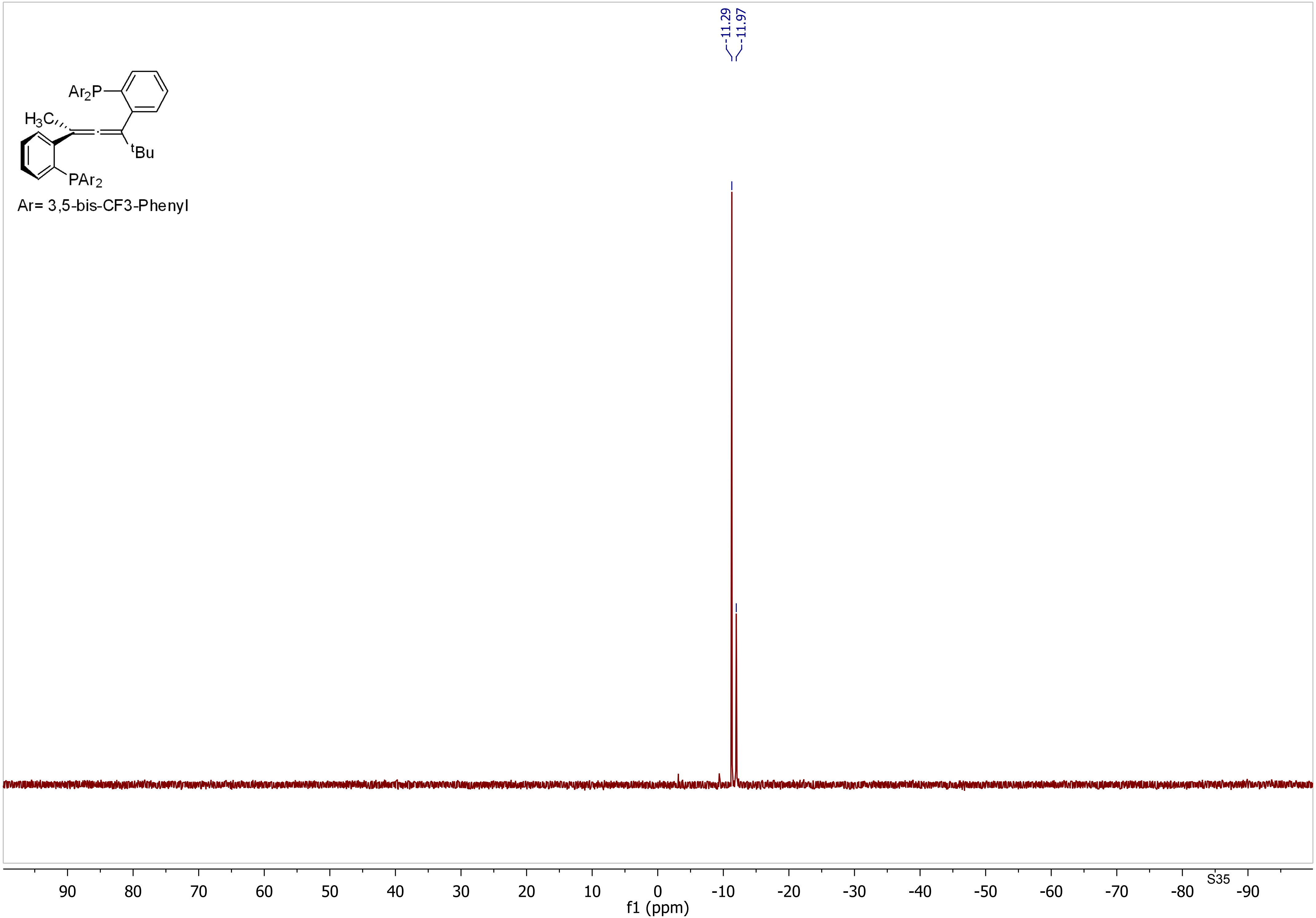
8.10
8.09
7.69
7.68
7.43
7.42
7.41
7.36
7.35
7.34
7.19
7.18
7.15
7.13
7.12
7.01
7.00
6.84
6.83
2.20
2.20
1.54
1.06
1.06
Br
H3C
tBu
S
O
(p-Tol)
1.00
0.99
1.11
3.18
1.10
1.06
2.12
2.02
3.41
3.45
10.48
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



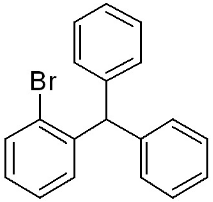
200.30
143.21
142.38
141.71
137.59
136.30
133.01
132.55
130.50
129.82
129.06
128.24
127.92
127.20
127.01
125.56
124.19
114.71
99.33
36.47
29.77
21.63
Br
H_3C
tBu
S
O
(p-Tol)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

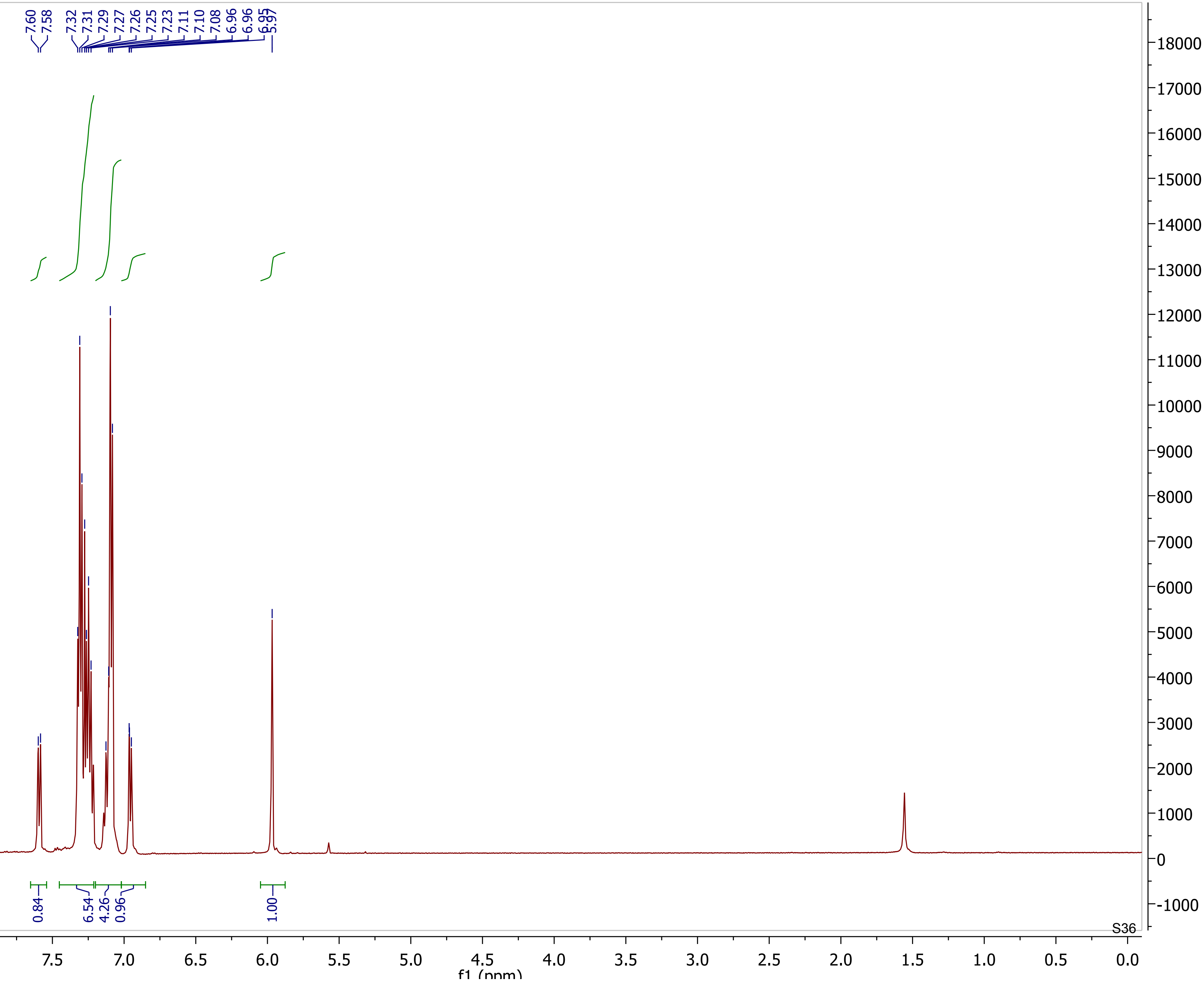

Ar= 3,5-bis-CF3-Phenyl
f1 (ppm)

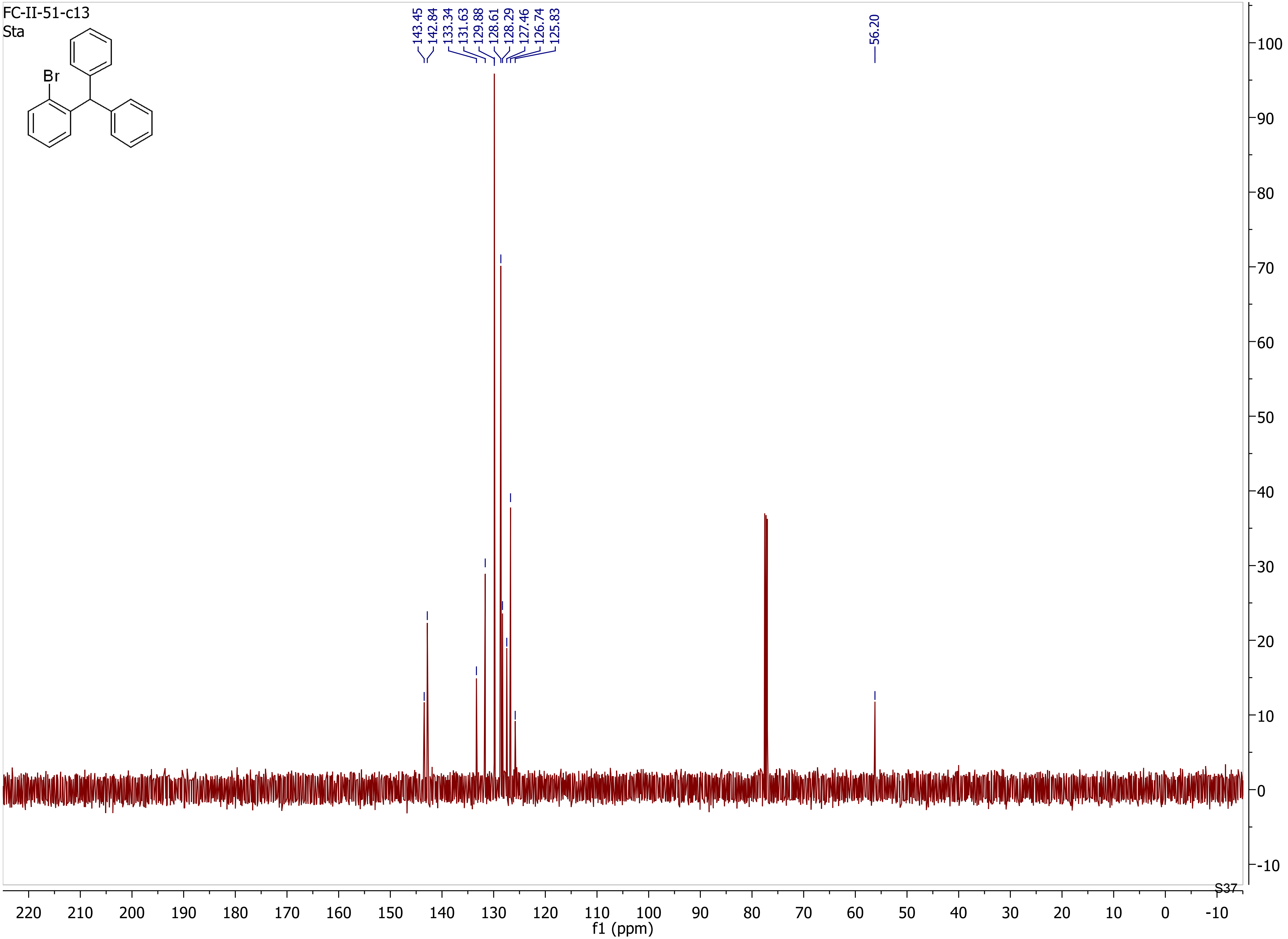

196.61
136.99
136.82
135.91
135.76
130.38
130.36
130.12
130.10
128.15
127.71
127.70
127.66
127.65
126.74
125.63
125.56
125.25
124.64
124.59
123.90
123.31
119.52
118.40
118.37
118.36
118.33
118.31
118.30
117.34
108.84
108.78
97.82
97.76
31.55
25.51
25.39
25.31
25.20
16.85
Ar2P
H3C
tBu
PAr2
Ar= 3,5-bis-CF3-Phenyl
f1 (ppm)

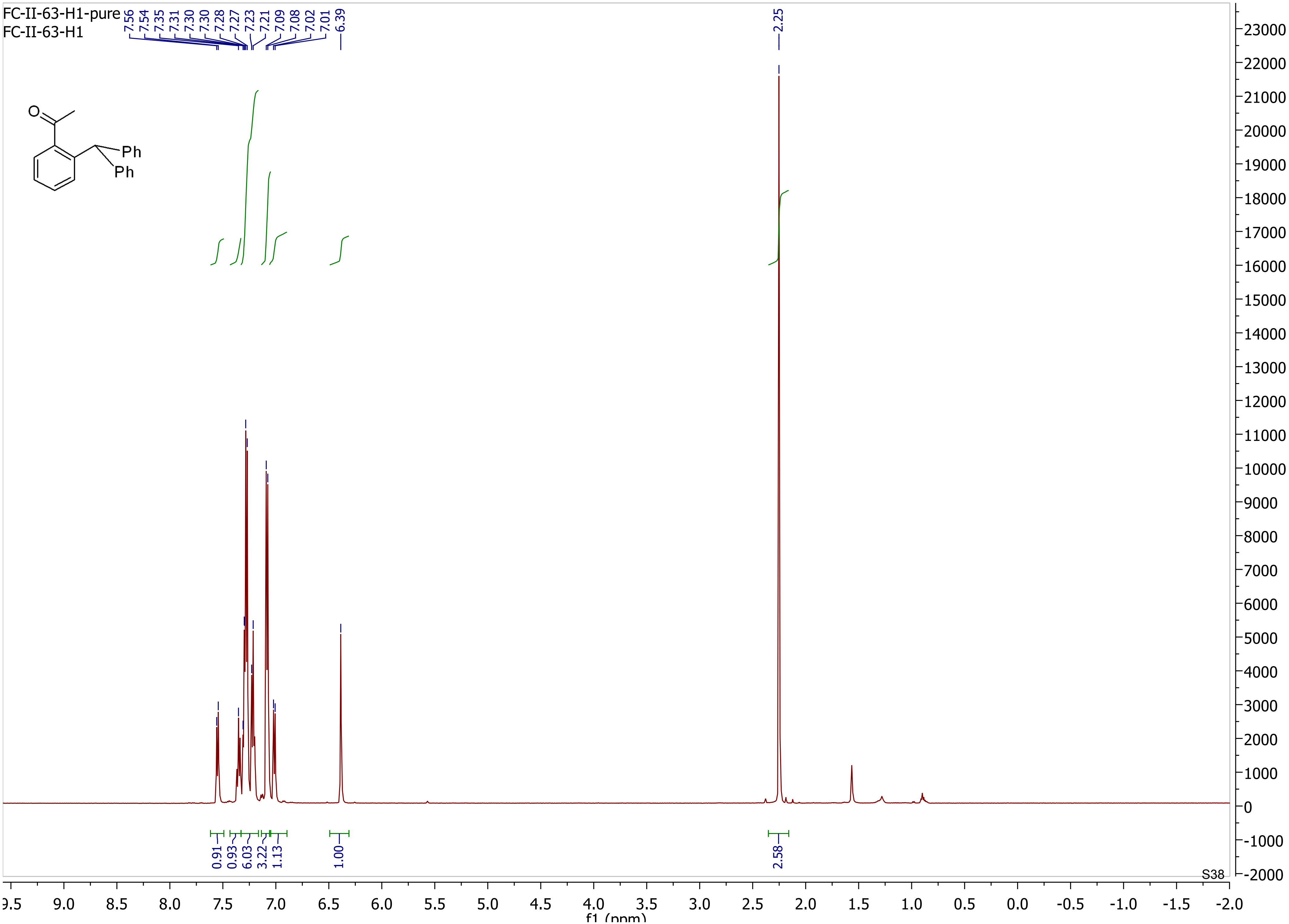

-11.29
-11.97
Ar₂P
H₃C
ᵗBu
PAr₂
Ar= 3,5-bis-CF3-Phenyl
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

FC-II-51pure-H1
FC-

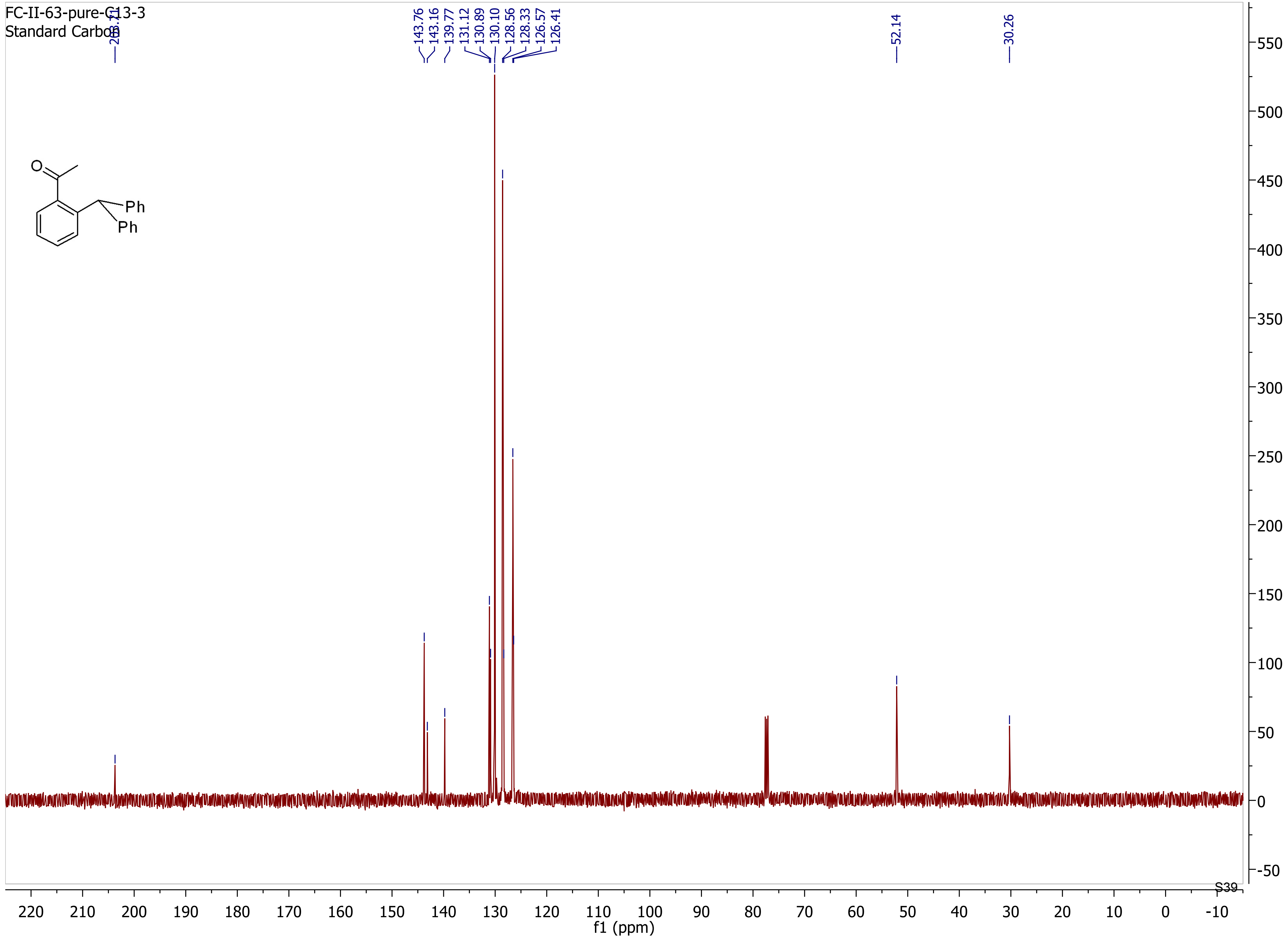




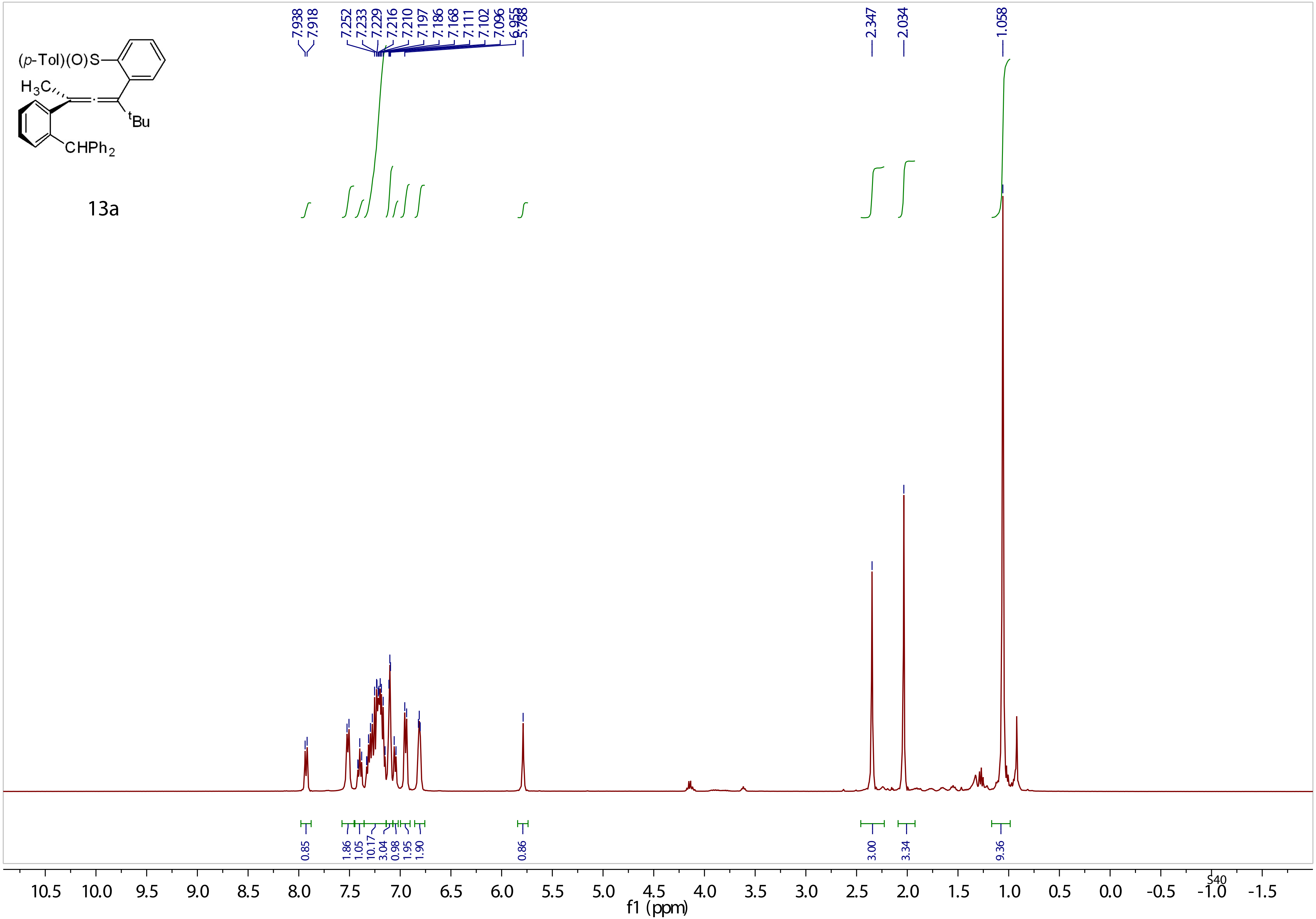

FC-II-51-c13
Sta
Br
143.45
142.84
133.34
131.63
129.88
128.61
128.29
127.46
126.74
125.83
56.20
f1 (ppm)

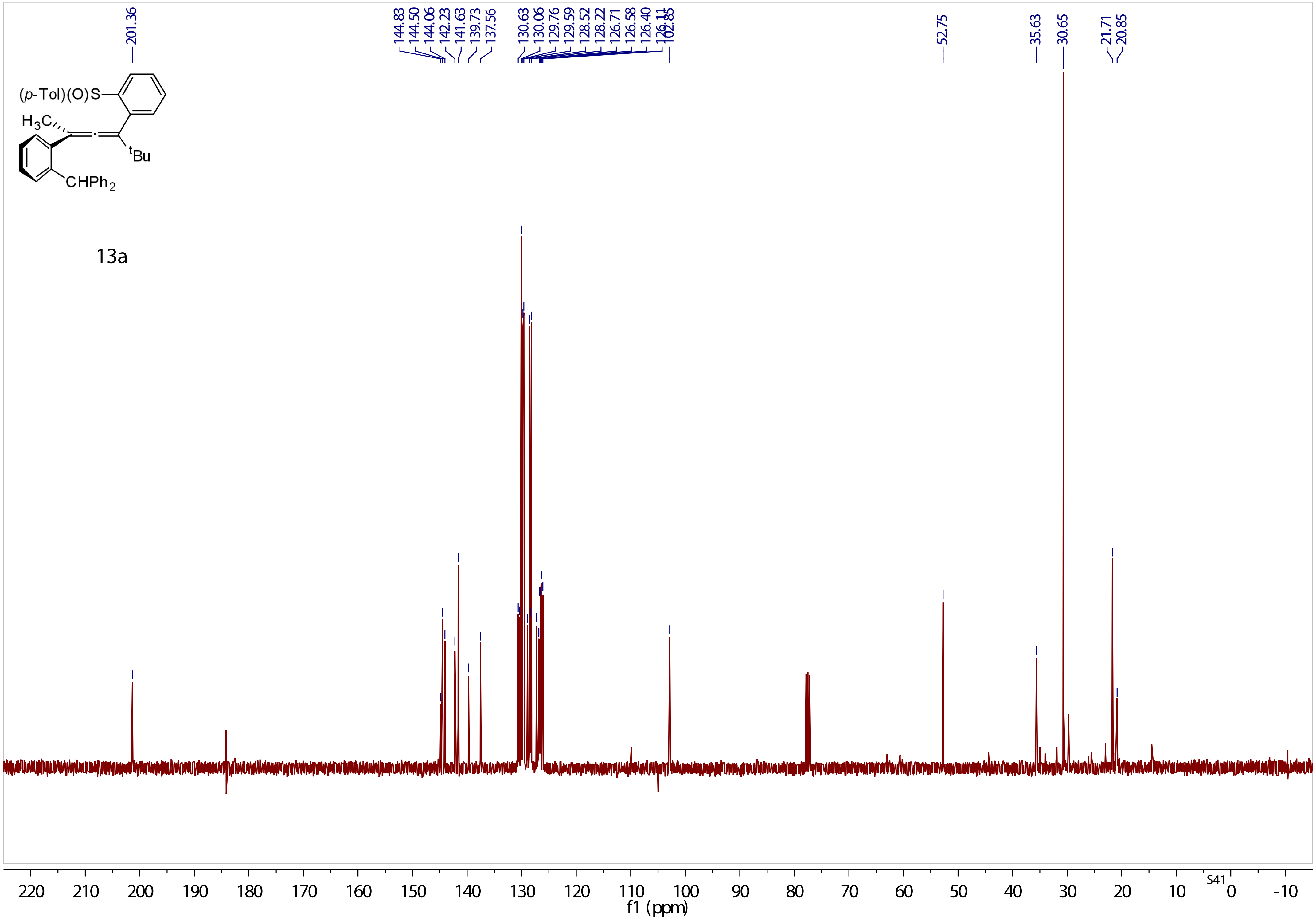


FC-II-63-H1-pure
FC-II-63-H1
7.56
7.54
7.35
7.31
7.30
7.30
7.28
7.27
7.23
7.21
7.09
7.08
7.02
7.01
6.39
2.25
O
Ph
Ph
0.91
0.93
6.03
3.22
1.13
1.00
2.58
f1 (ppm)

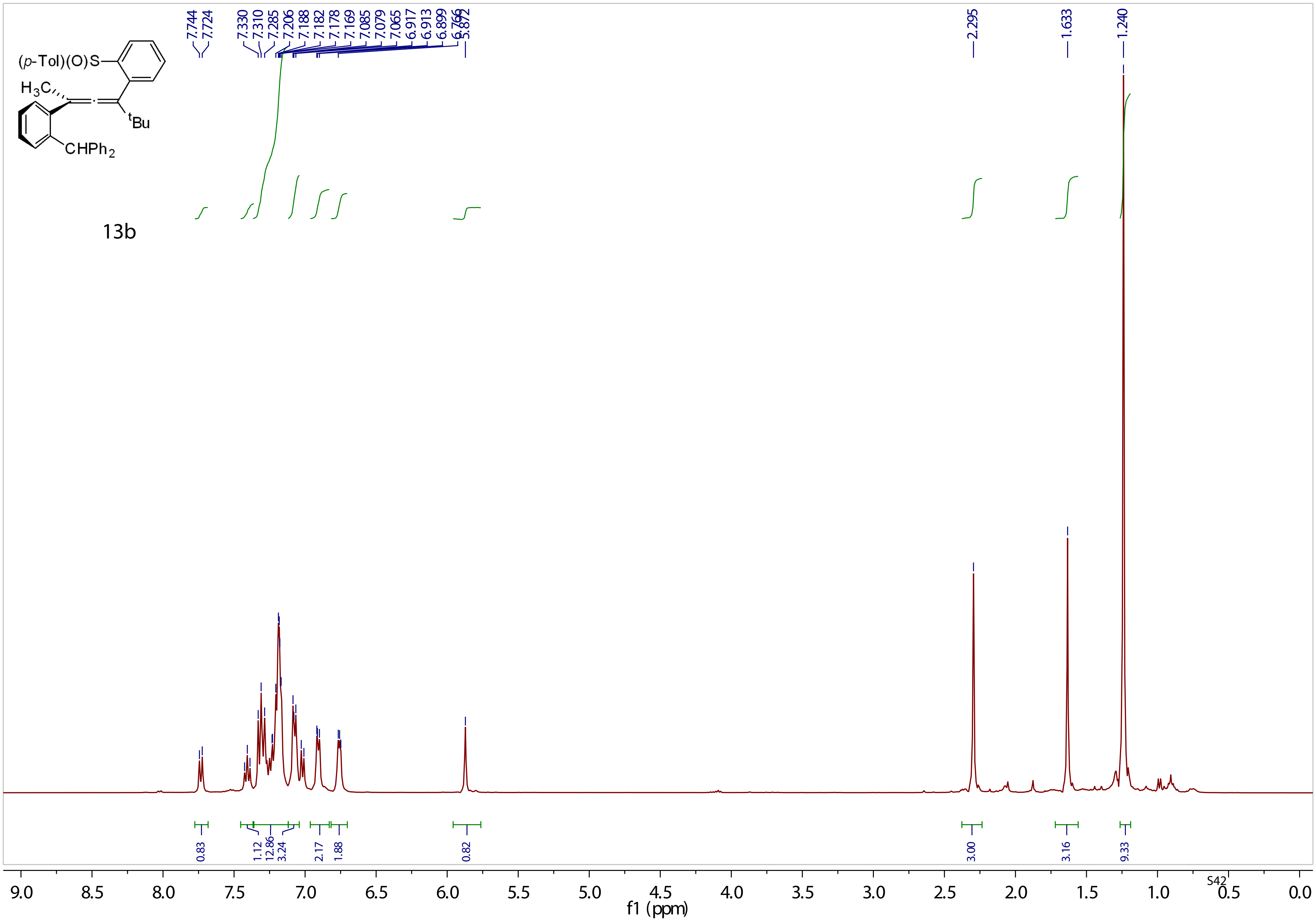


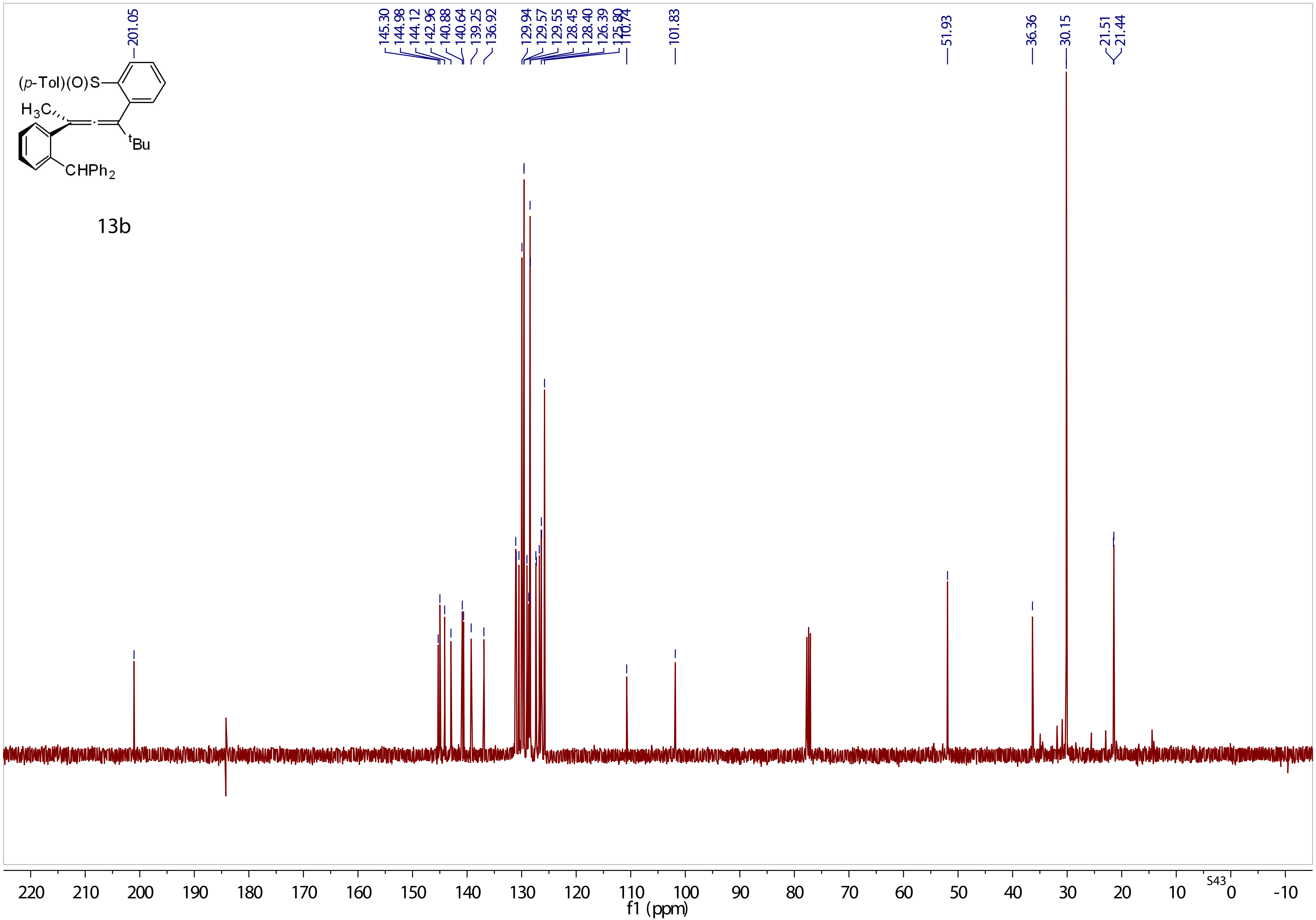
FC-II-63-pure-C13-3
Standard Carbon
203.71
143.76
143.16
139.77
131.12
130.89
130.10
128.56
128.33
126.57
126.41
52.14
30.26
O
Ph
Ph
f1 (ppm)

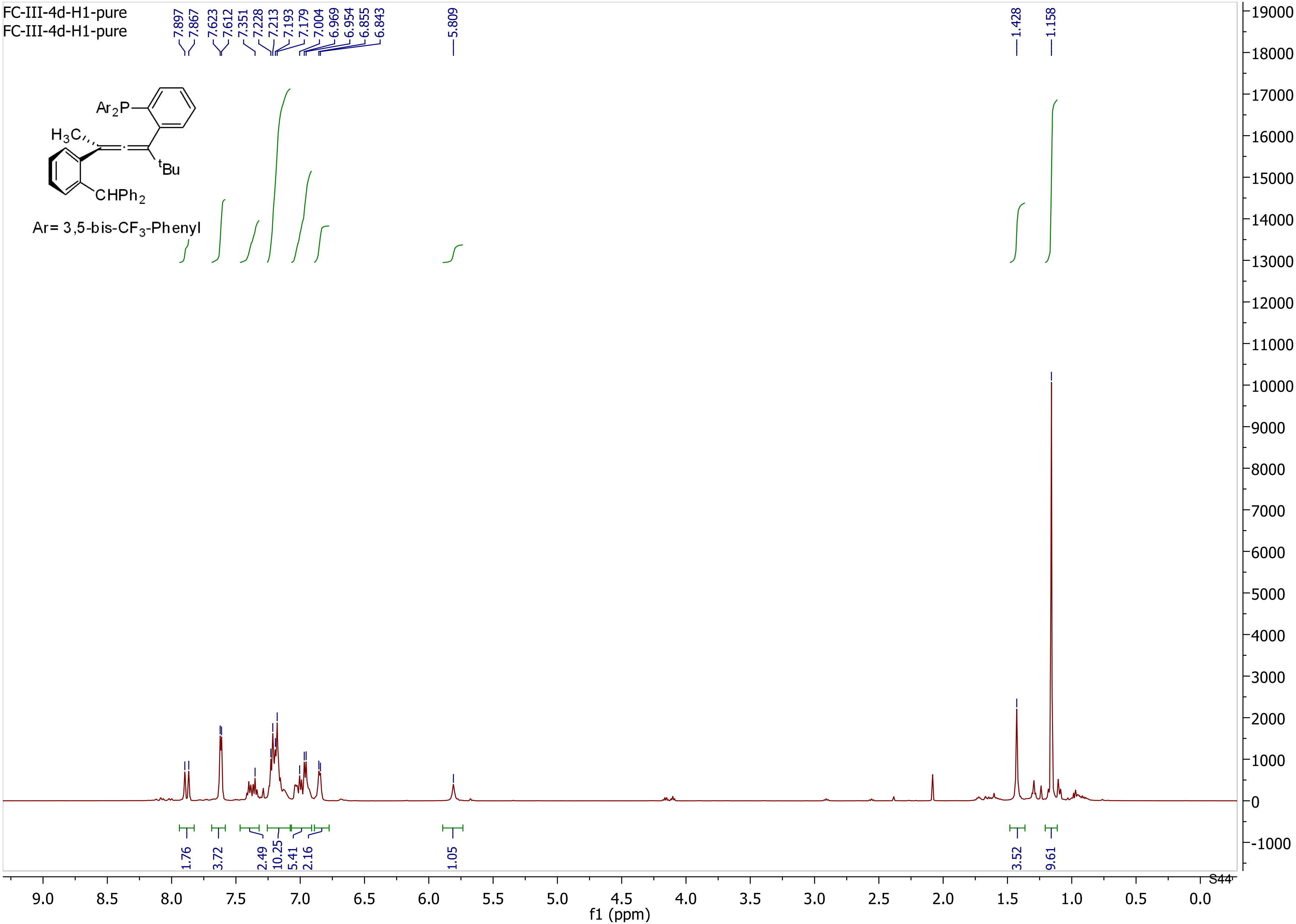

(p-Tol)(O)S
H3C
tBu
CHPh2
13a
7.938
7.918
7.252
7.233
7.229
7.216
7.210
7.197
7.186
7.168
7.111
7.102
7.096
6.955
5.788
2.347
2.034
1.058
0.85
1.86
1.05
10.17
3.04
0.98
1.95
1.90
0.86
3.00
3.34
9.36
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

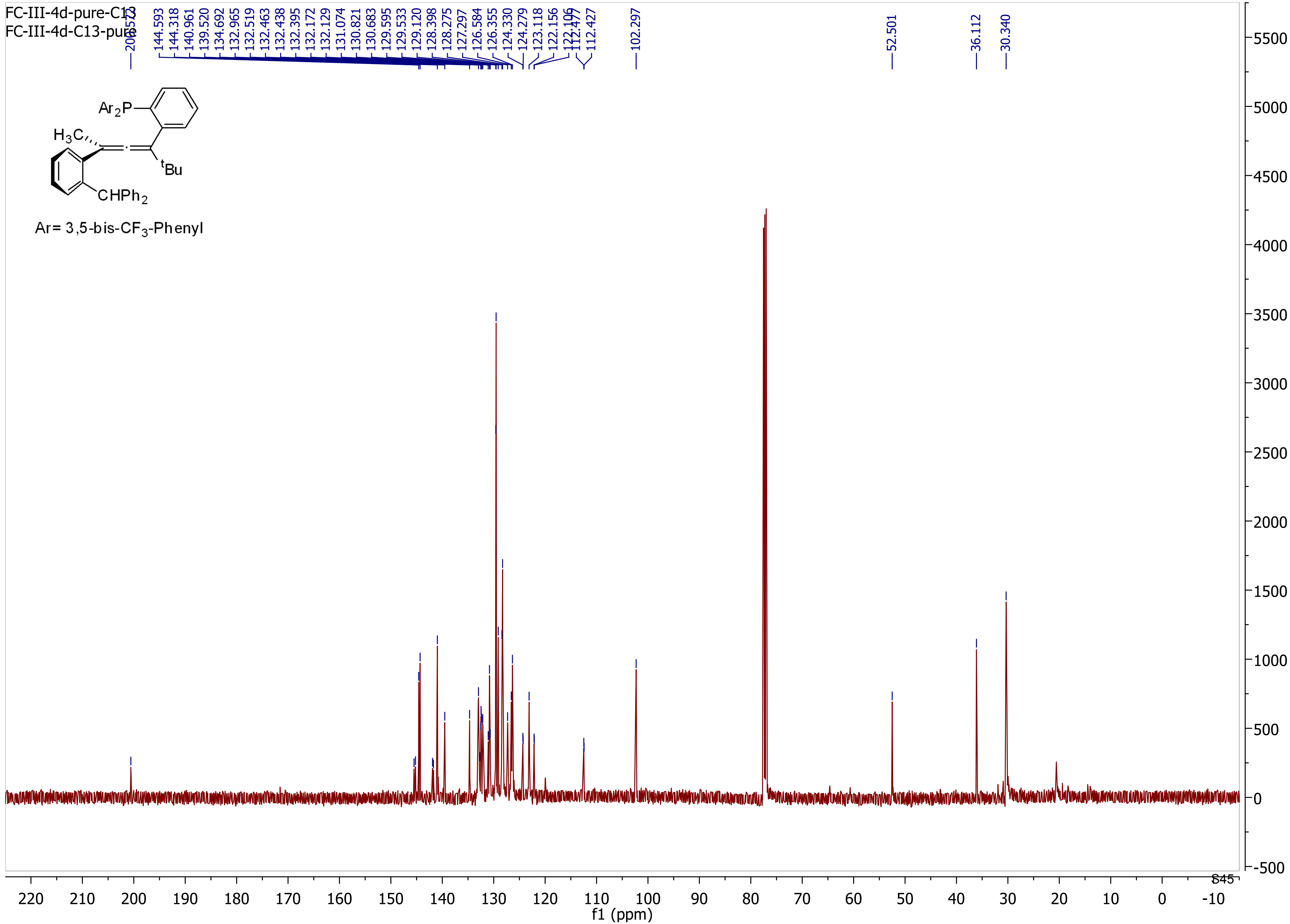


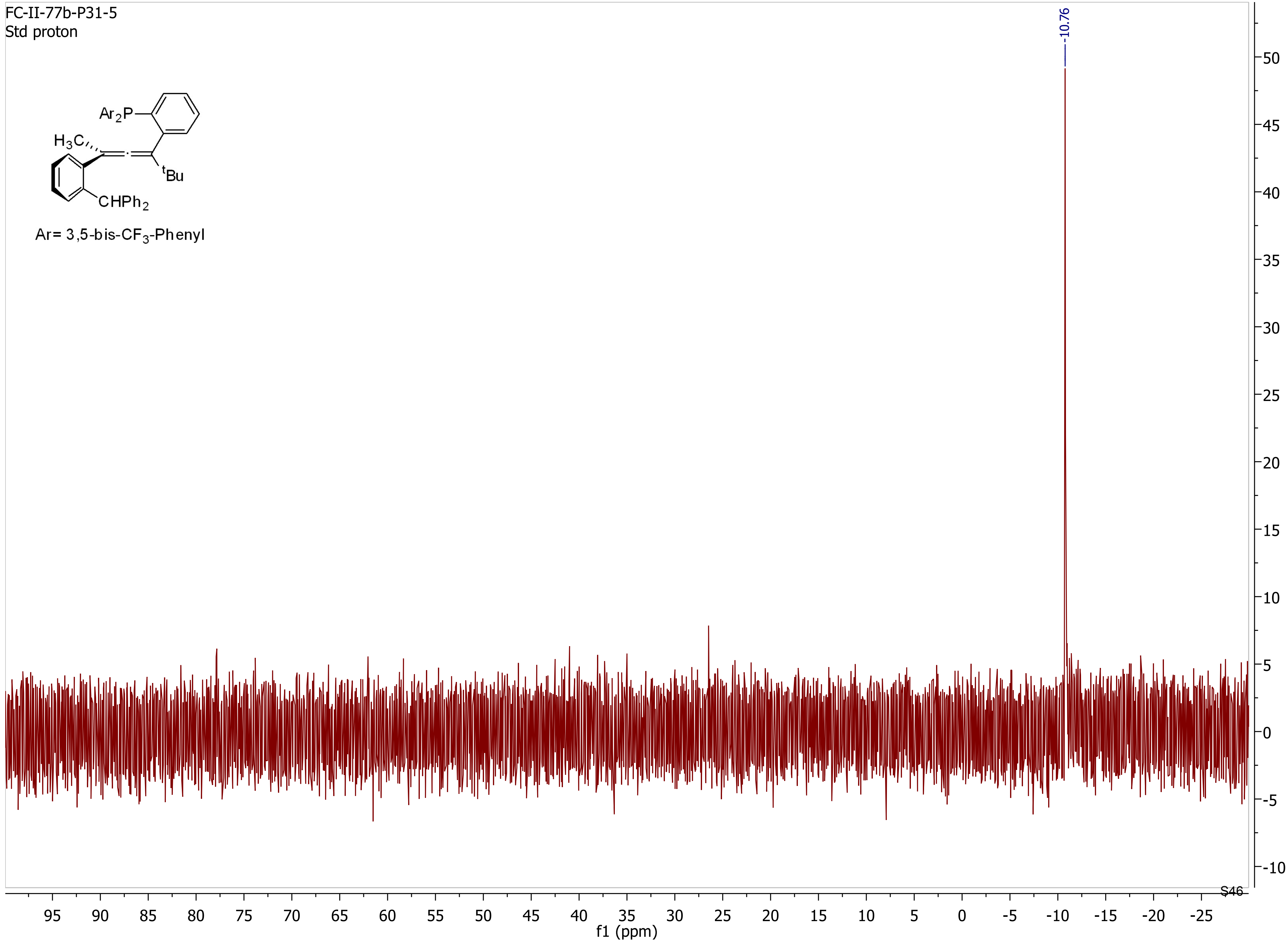
201.36
144.83
144.50
144.06
142.23
141.63
139.73
137.56
130.63
130.06
129.76
129.59
128.52
128.22
126.71
126.58
126.40
102.85
52.75
35.63
30.65
21.71
20.85
(p-Tol)(O)S
H3C
tBu
CHPh2
13a
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

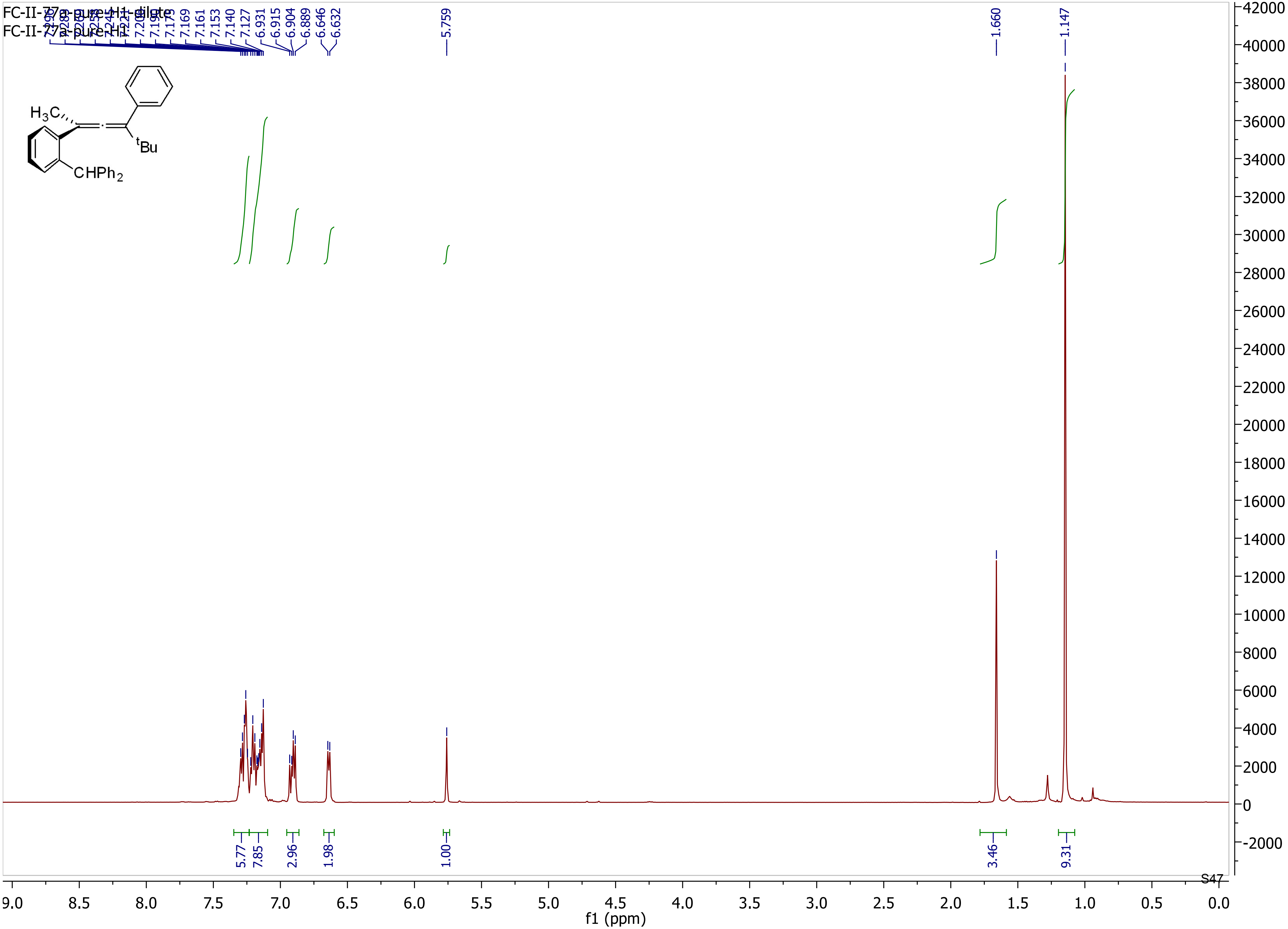

(p-Tol)(O)S
H3C
tBu
CHPh2
13b
7.744
7.724
7.330
7.310
7.285
7.206
7.188
7.182
7.178
7.169
7.085
7.079
7.065
6.917
6.913
6.899
6.766
5.872
2.295
1.633
1.240
0.83
1.12
12.86
3.24
2.17
1.88
0.82
3.00
3.16
9.33
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

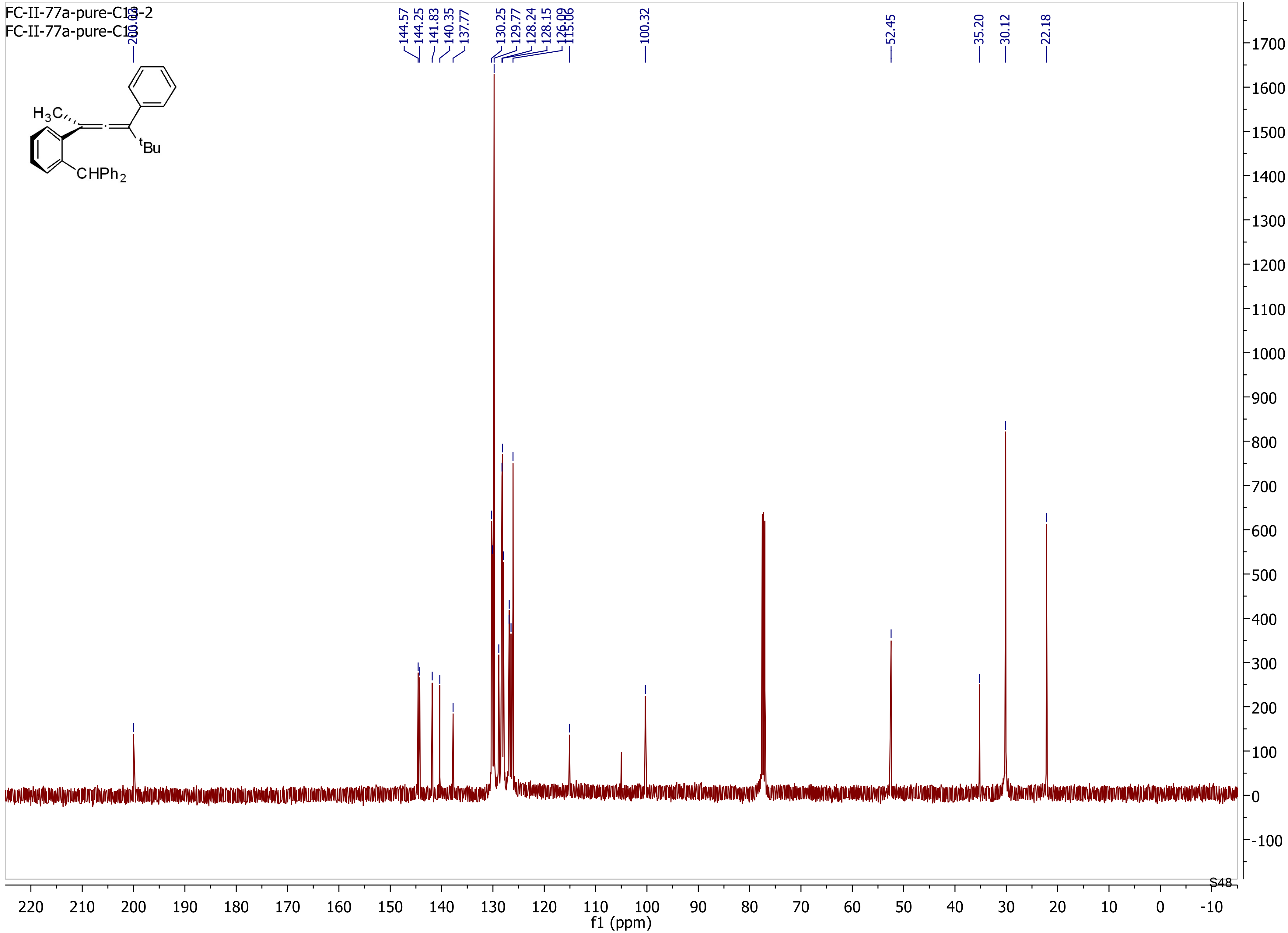

201.05
145.30
144.98
144.12
142.96
140.88
140.64
139.25
136.92
129.94
129.57
129.55
128.45
128.40
126.39
125.80
110.74
101.83
51.93
36.36
30.15
21.51
21.44
(p-Tol)(O)S
H3C
tBu
CHPh2
13b
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


FC-III-4d-H1-pure
FC-III-4d-H1-pure
7.897
7.867
7.623
7.612
7.351
7.228
7.213
7.193
7.179
7.004
6.969
6.954
6.855
6.843
5.809
1.428
1.158
Ar2P
H3C
tBu
CHPh2
Ar= 3,5-bis-CF3-Phenyl
1.76
3.72
2.49
10.25
5.41
2.16
1.05
3.52
9.61
f1 (ppm)

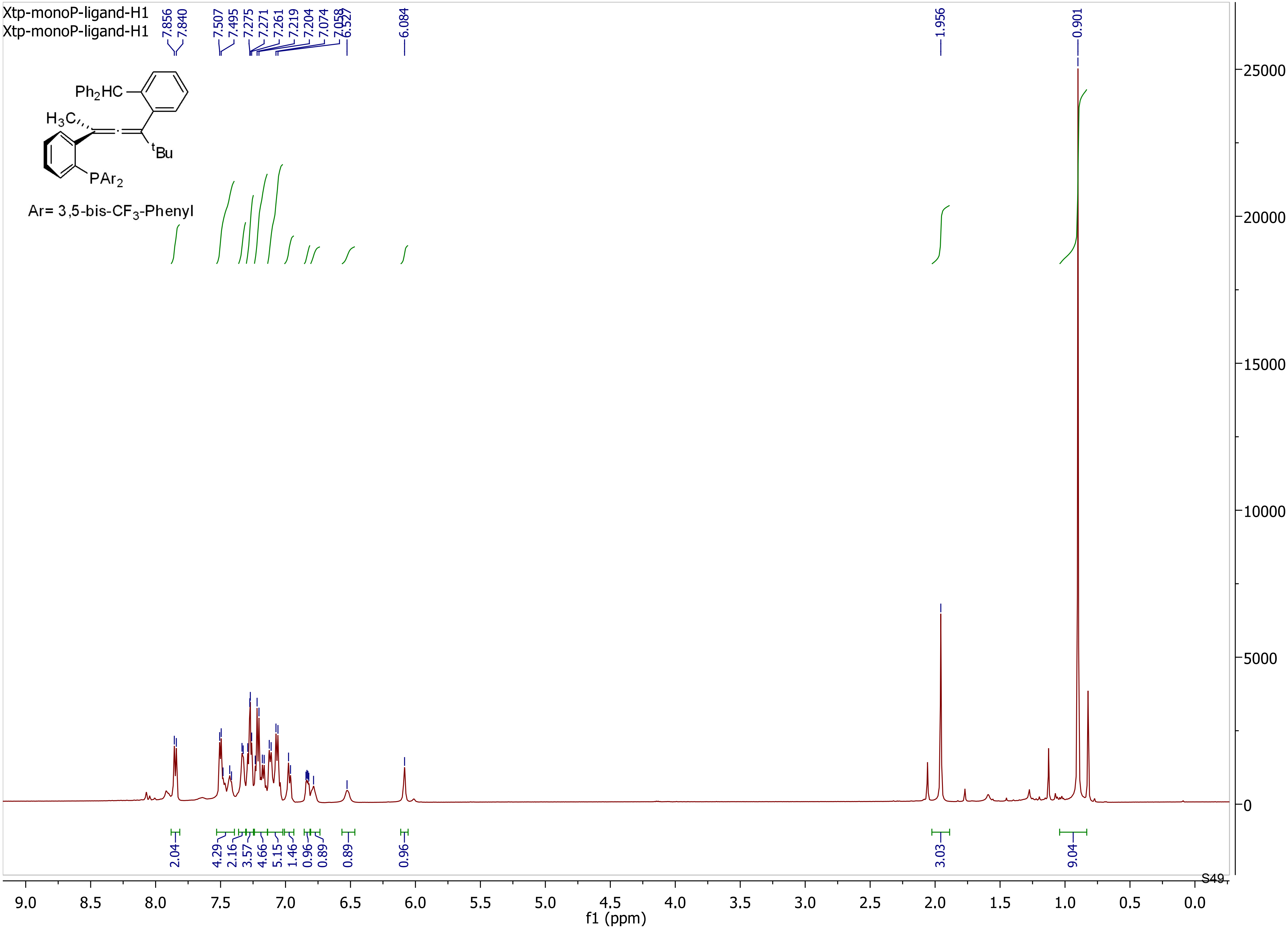

FC-III-4d-pure-C13
FC-III-4d-C13-pure
200.573
144.593
144.318
140.961
139.520
134.692
132.965
132.519
132.463
132.438
132.395
132.172
132.129
131.074
130.821
130.683
129.595
129.533
129.120
128.398
128.275
127.297
126.584
126.355
124.330
124.279
123.118
122.156
122.106
112.477
112.427
102.297
52.501
36.112
30.340
Ar₂P
H₃C
tBu
CHPh₂
Ar= 3,5-bis-CF₃-Phenyl
f1 (ppm)

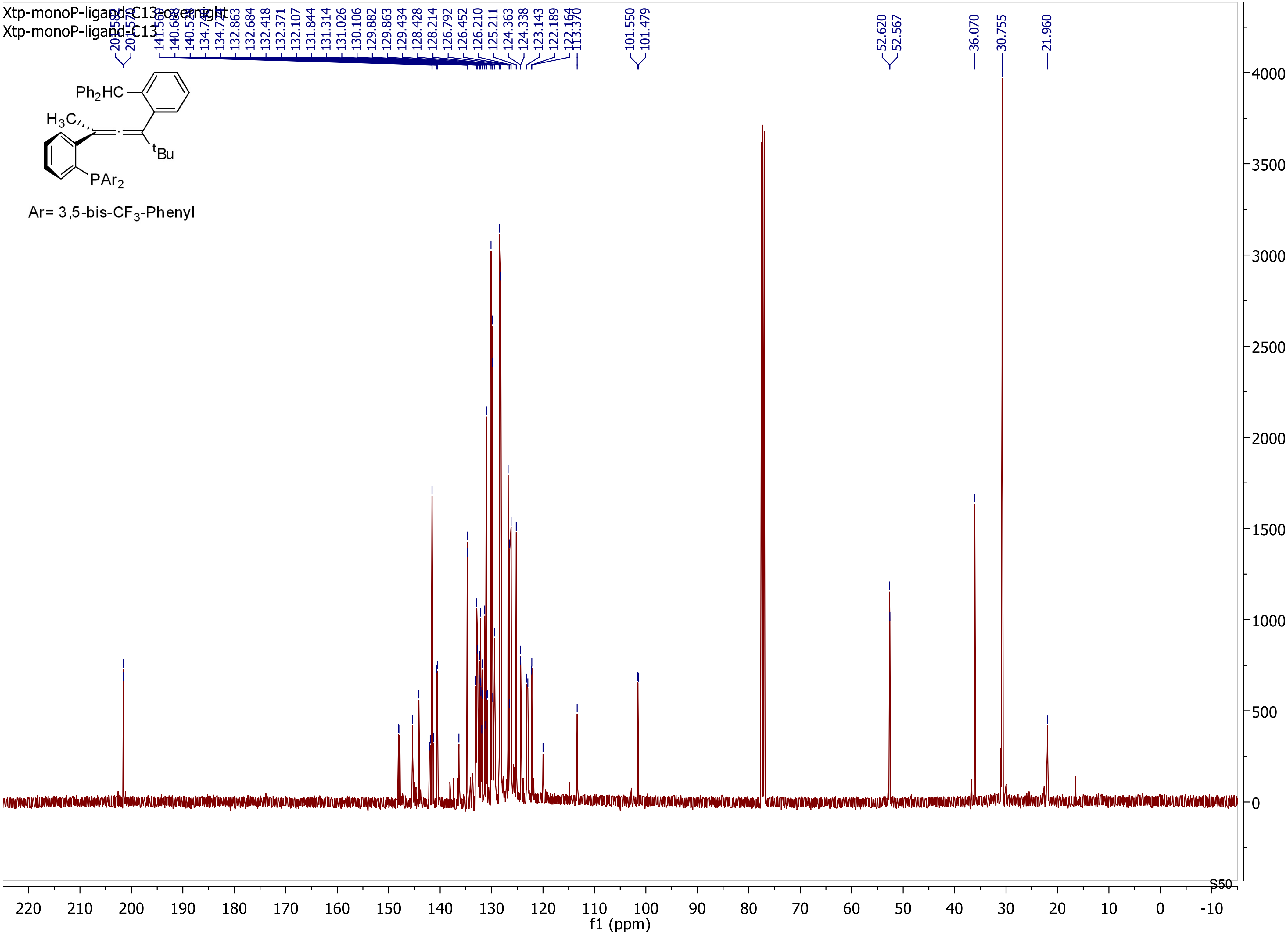

FC-II-77b-P31-5
Std proton
Ar2P
H3C
tBu
CHPh2
Ar= 3,5-bis-CF3-Phenyl
-10.76
f1 (ppm)

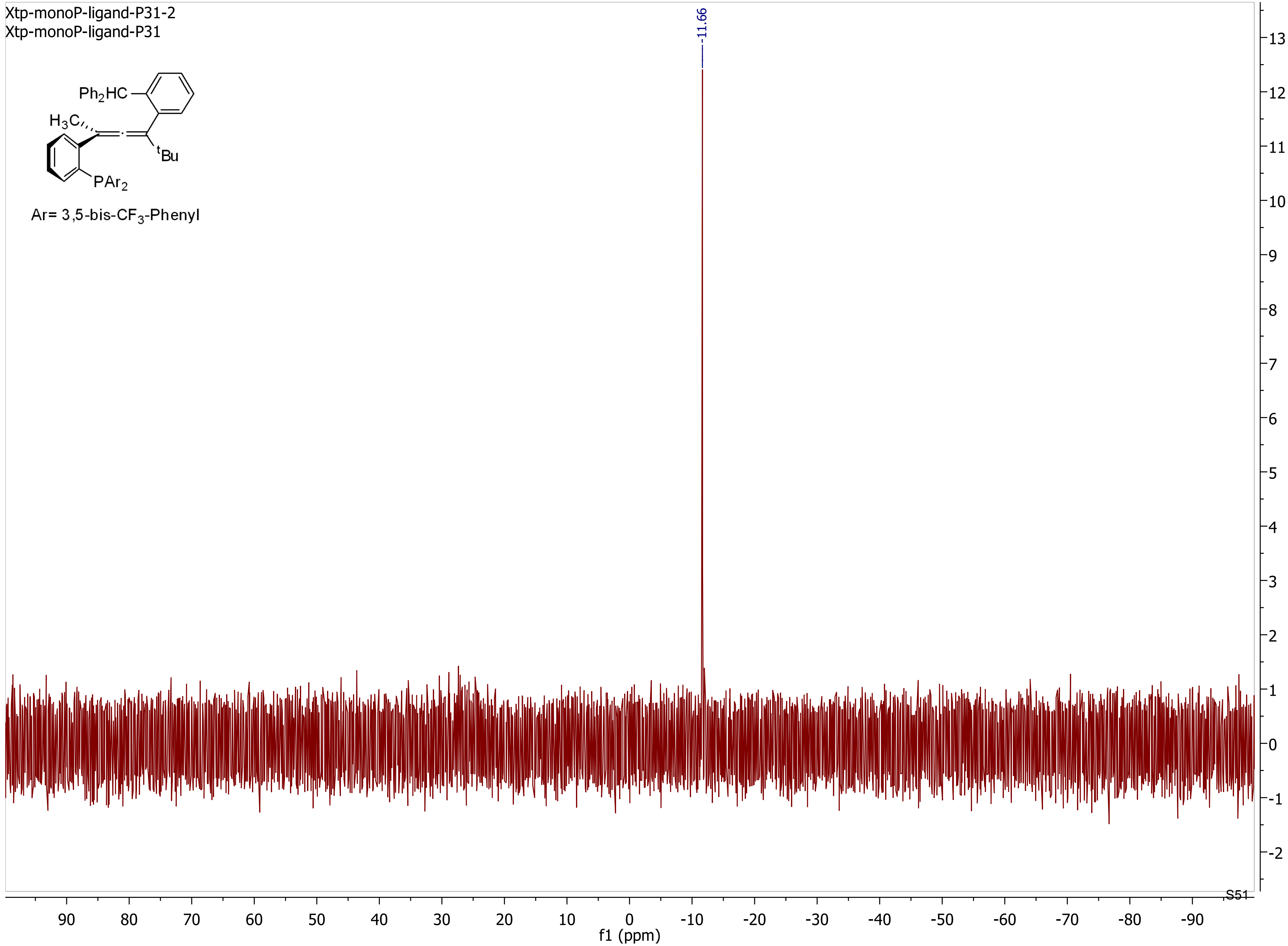

FC-II-77a-pure-H1
7.169
7.161
7.153
7.140
7.127
6.931
6.915
6.904
6.889
6.646
6.632
5.759
1.660
1.147
H3C
tBu
CHPh2
5.77
7.85
2.96
1.98
1.00
3.46
9.31
f1 (ppm)

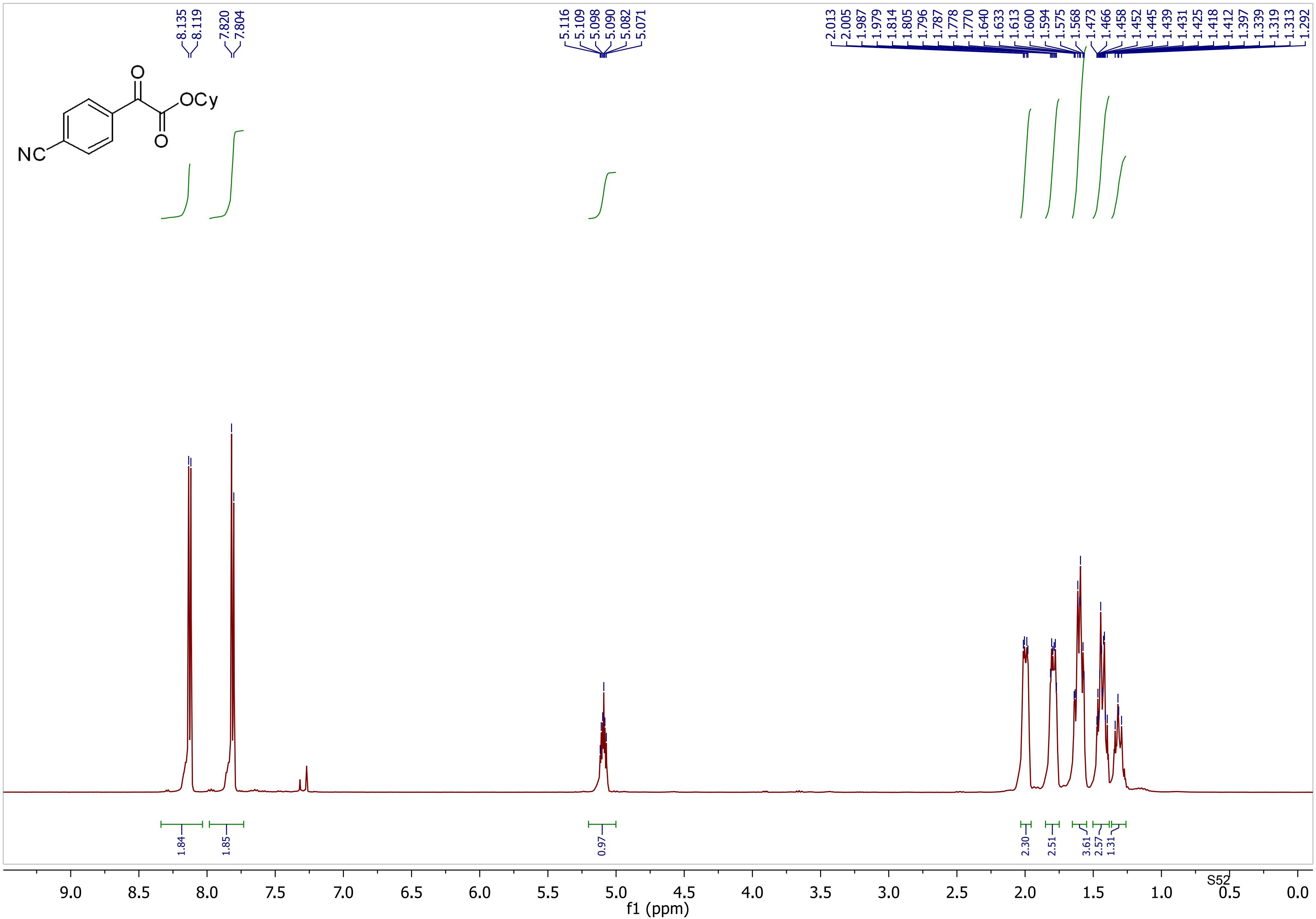


FC-II-77a-pure-C13-2
FC-II-77a-pure-C13
200.03
144.57
144.25
141.83
140.35
137.77
130.25
129.77
128.24
128.15
126.09
115.06
100.32
52.45
35.20
30.12
22.18
H3C
tBu
CHPh2
f1 (ppm)

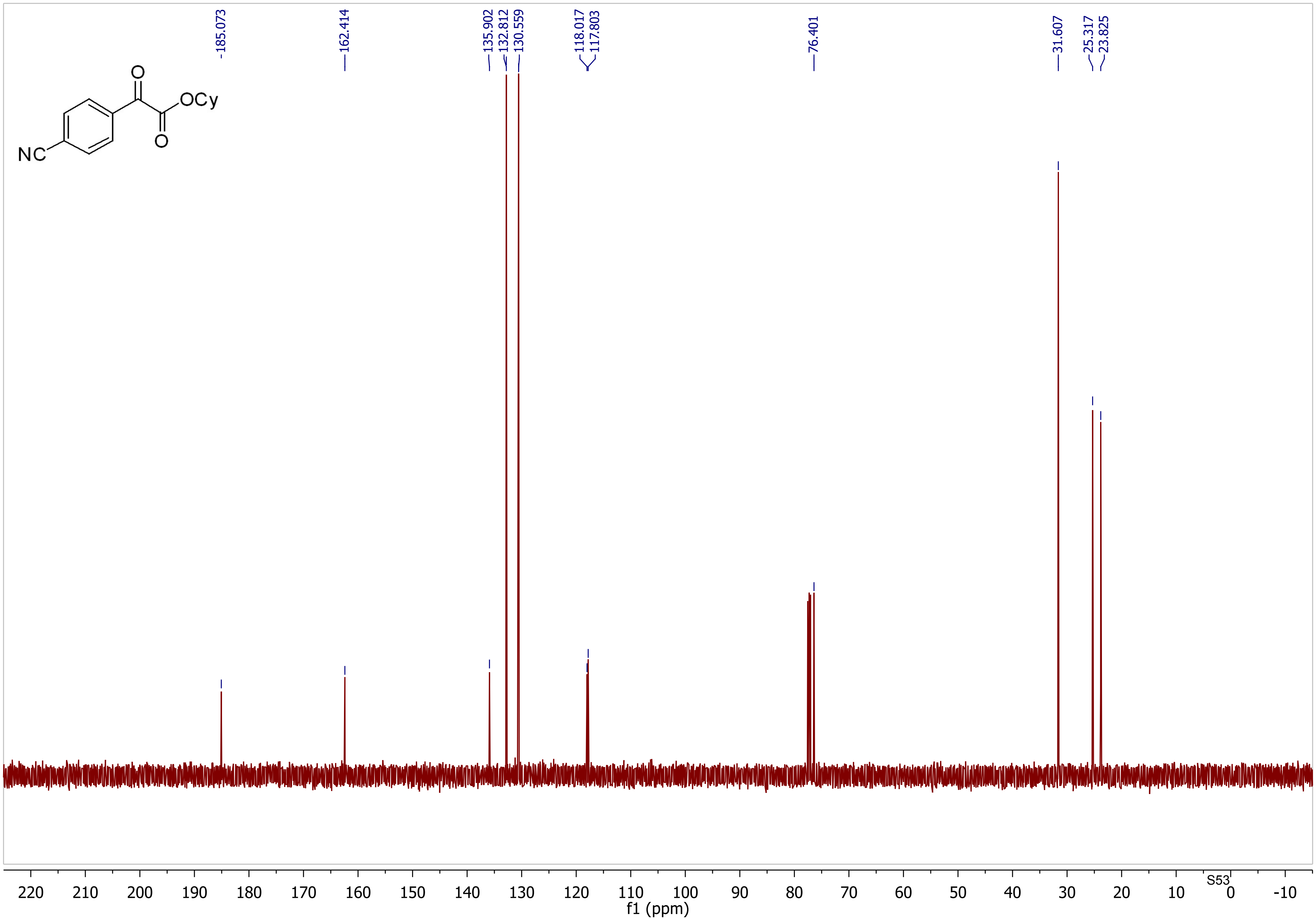

Xtp-monoP-ligand-H1
Xtp-monoP-ligand-H1
Ph2HC
H3C
tBu
PAr2
Ar= 3,5-bis-CF3-Phenyl
7.856
7.840
7.507
7.495
7.275
7.271
7.261
7.219
7.204
7.074
7.058
6.527
6.084
1.956
0.901
2.04
4.29
2.16
3.57
4.66
5.15
1.46
0.96
0.89
0.89
0.96
3.03
9.04
f1 (ppm)


Xtp-monoP-ligand-C13-overnight
Xtp-monoP-ligand-C13
132.863
132.684
132.418
132.371
132.107
131.844
131.314
131.026
130.106
129.882
129.863
129.434
128.428
128.214
126.792
126.452
126.210
125.211
124.363
124.338
123.143
122.189
113.370
101.550
101.479
52.620
52.567
36.070
30.755
21.960
Ph2HC
H3C
tBu
PAr2
Ar= 3,5-bis-CF3-Phenyl
f1 (ppm)

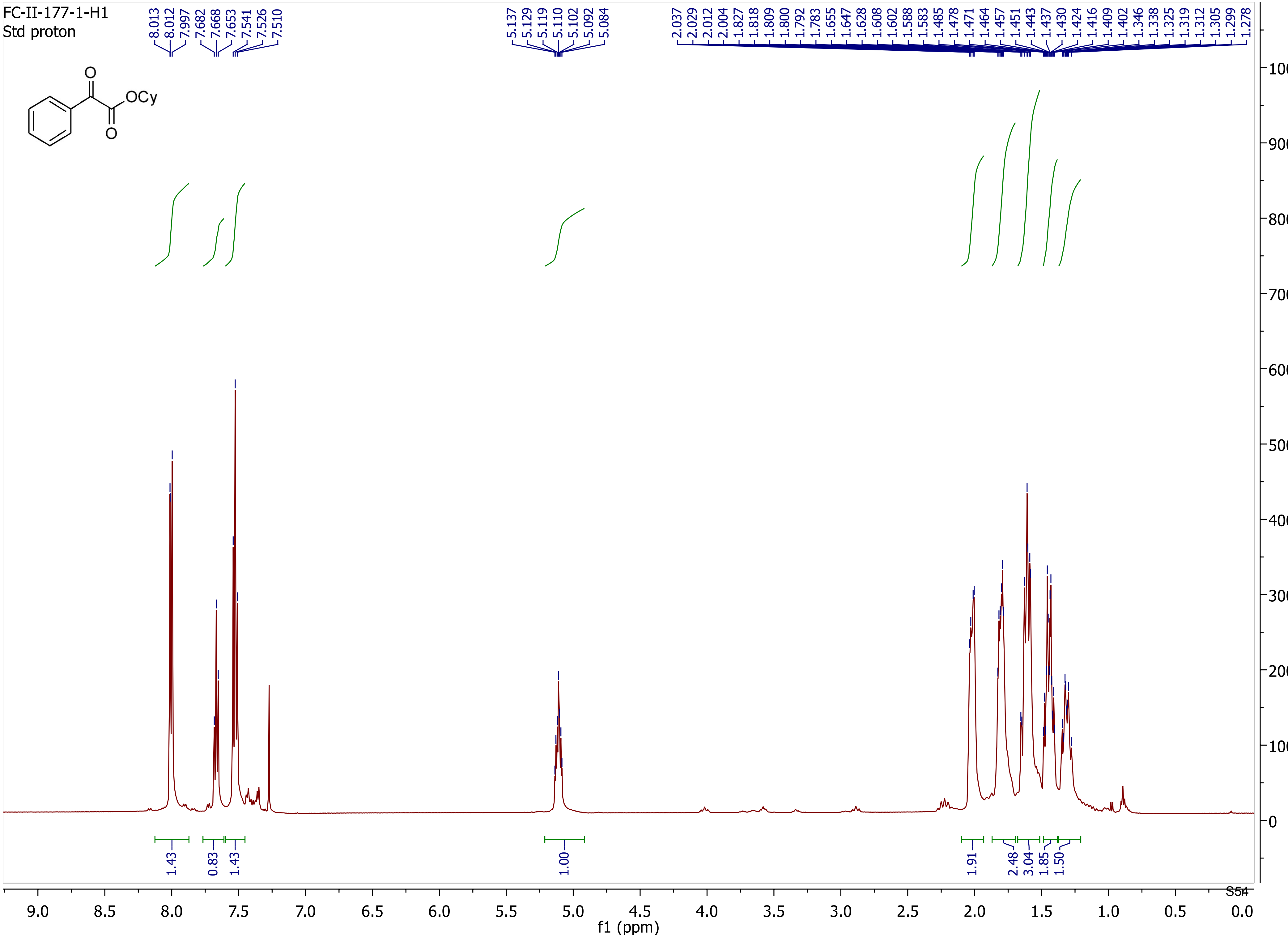

Xtp-monoP-ligand-P31-2
Xtp-monoP-ligand-P31
Ph2HC
H3C
tBu
PAr2
Ar= 3,5-bis-CF3-Phenyl
-11.66
f1 (ppm)

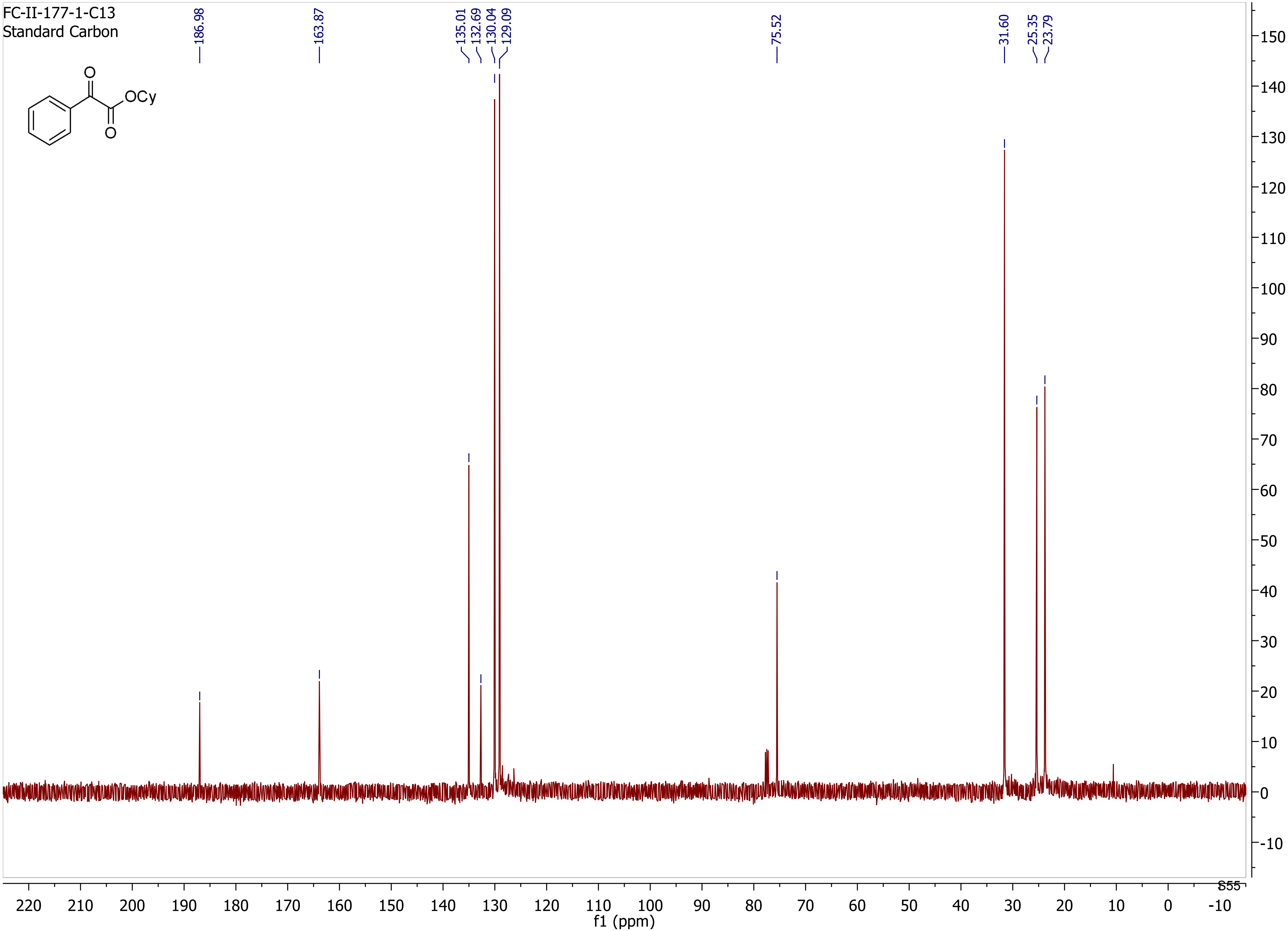


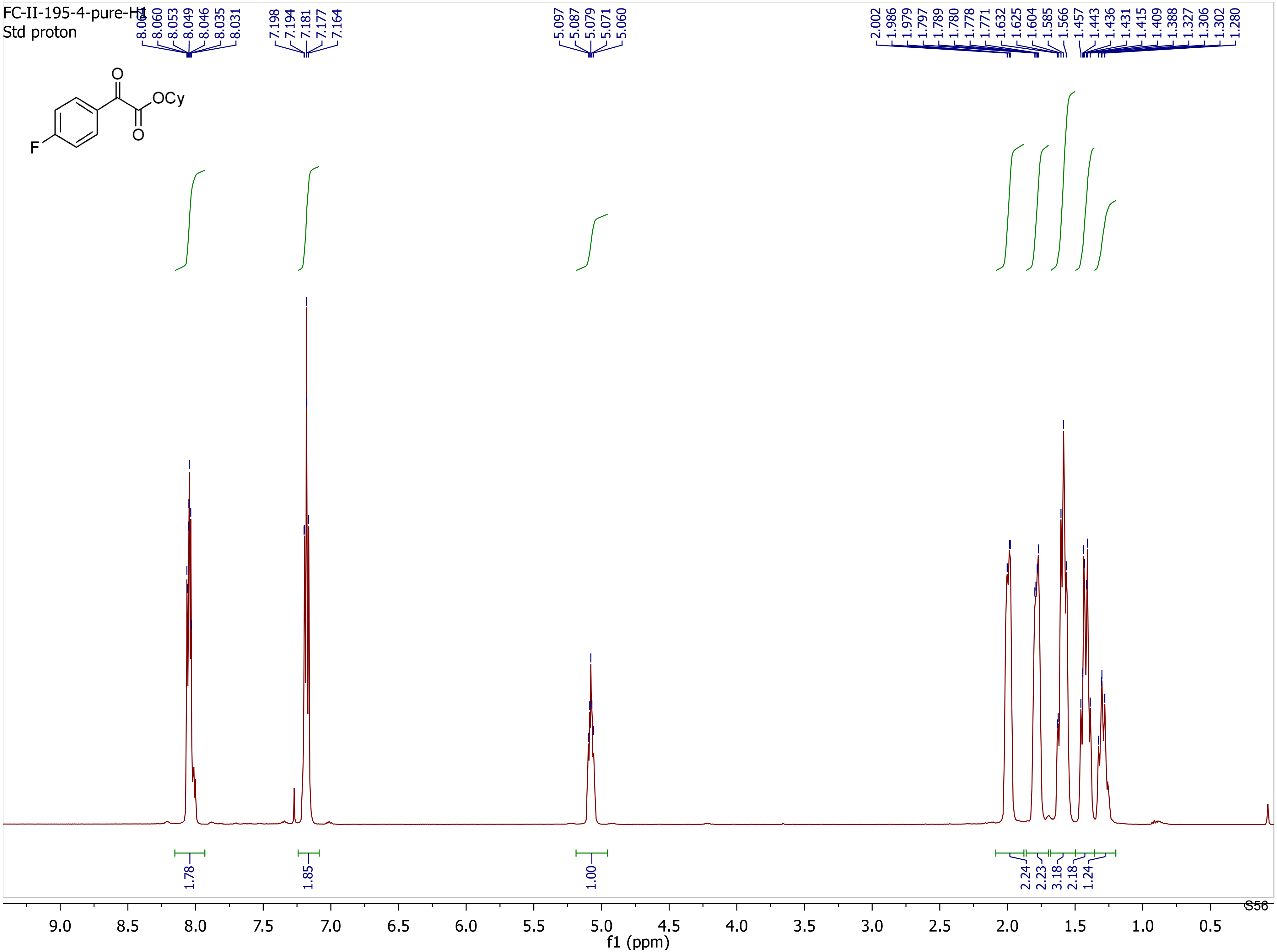
8.135
8.119
7.820
7.804
5.116
5.109
5.098
5.090
5.082
5.071
2.013
2.005
1.987
1.979
1.814
1.805
1.796
1.787
1.778
1.770
1.640
1.633
1.613
1.600
1.594
1.575
1.568
1.473
1.466
1.458
1.452
1.445
1.439
1.431
1.425
1.418
1.412
1.397
1.339
1.319
1.313
1.292
NC
O
OCy
O
1.84
1.85
0.97
2.30
2.51
3.61
2.57
1.31
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
f1 (ppm)

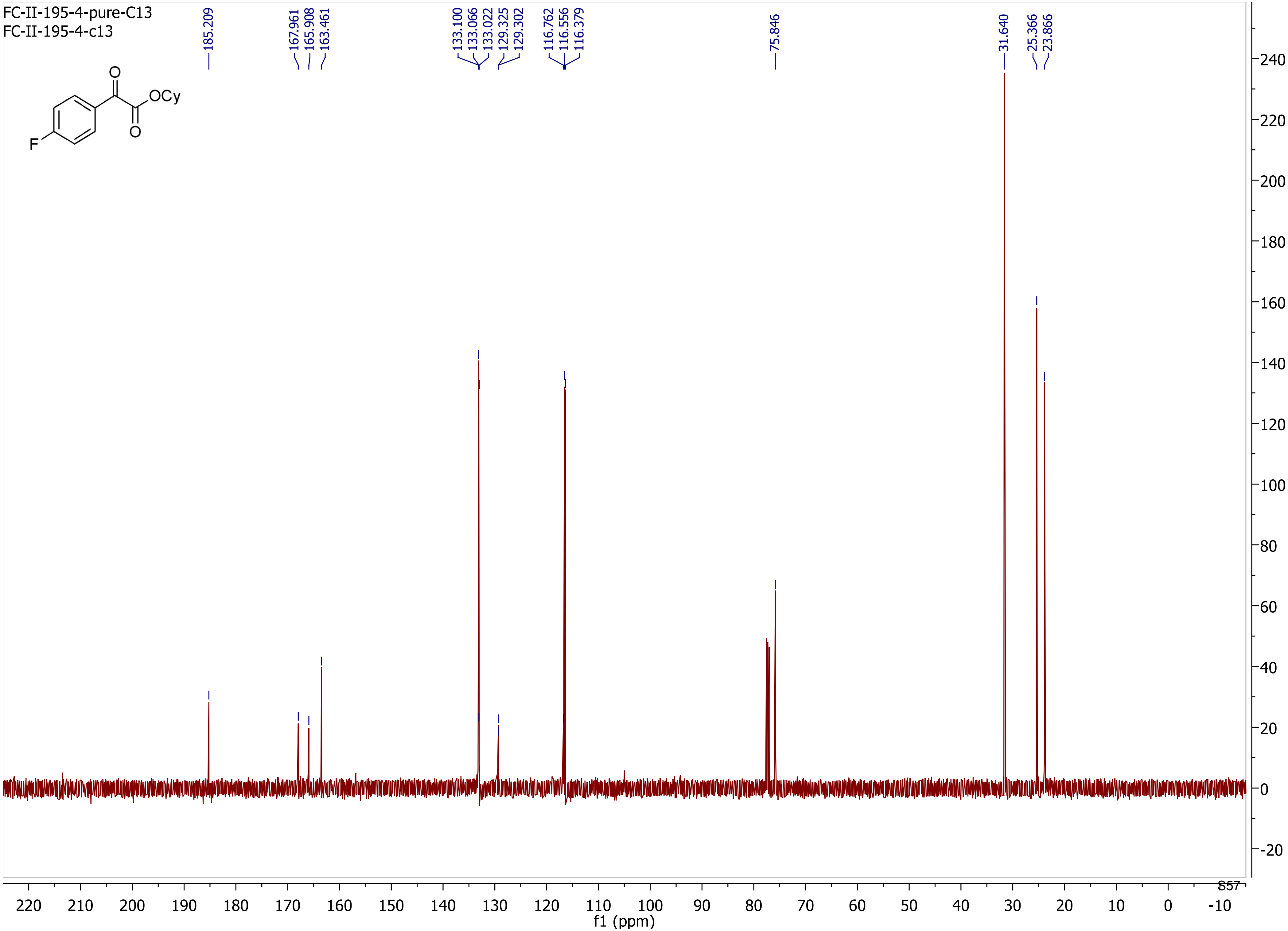


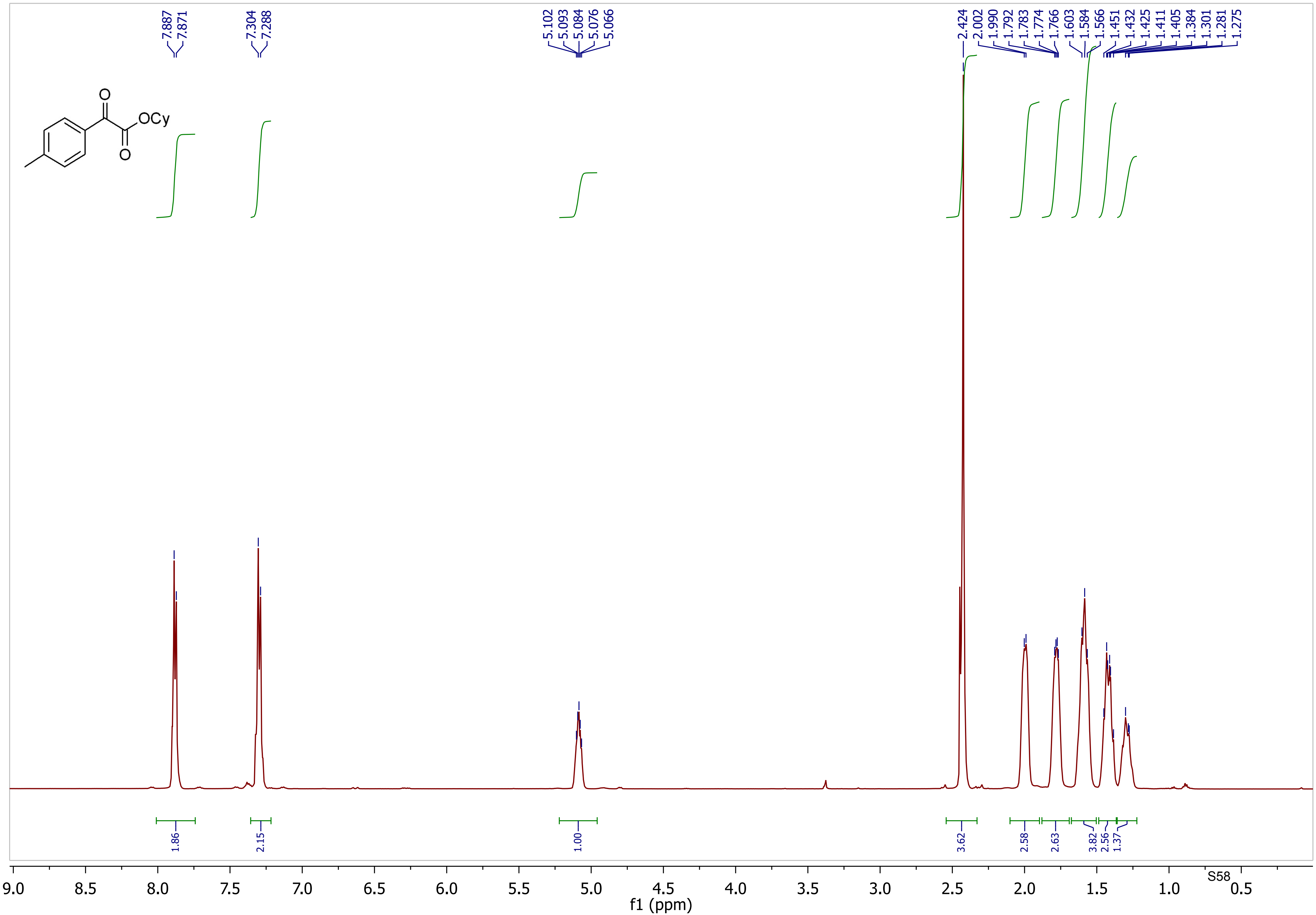
185.073
162.414
135.902
132.812
130.559
118.017
117.803
76.401
31.607
25.317
23.825
NC
O
OCy
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

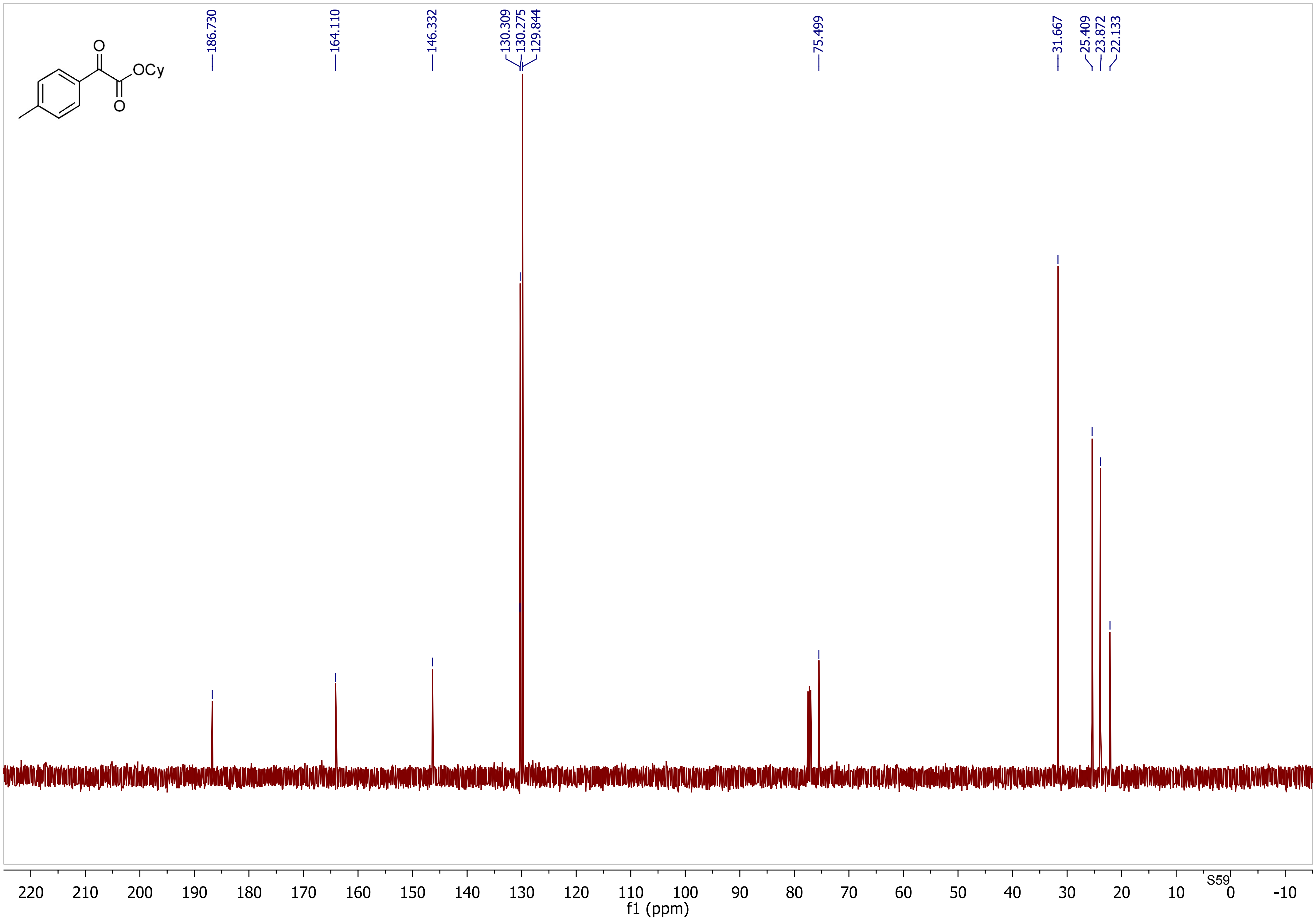

FC-II-177-1-H1
Std proton
OCy
O
O
8.013
8.012
7.997
7.682
7.668
7.653
7.541
7.526
7.510
5.137
5.129
5.119
5.110
5.102
5.092
5.084
2.037
2.029
2.012
2.004
1.827
1.818
1.809
1.800
1.792
1.783
1.655
1.647
1.628
1.608
1.602
1.588
1.583
1.485
1.478
1.471
1.464
1.457
1.451
1.443
1.437
1.430
1.424
1.416
1.409
1.402
1.346
1.338
1.325
1.319
1.312
1.305
1.299
1.278
1.43
0.83
1.43
1.00
1.91
2.48
3.04
1.85
1.50
f1 (ppm)


FC-II-177-1-C13
Standard Carbon
OCy
186.98
163.87
135.01
132.69
130.04
129.09
75.52
31.60
25.35
23.79
f1 (ppm)

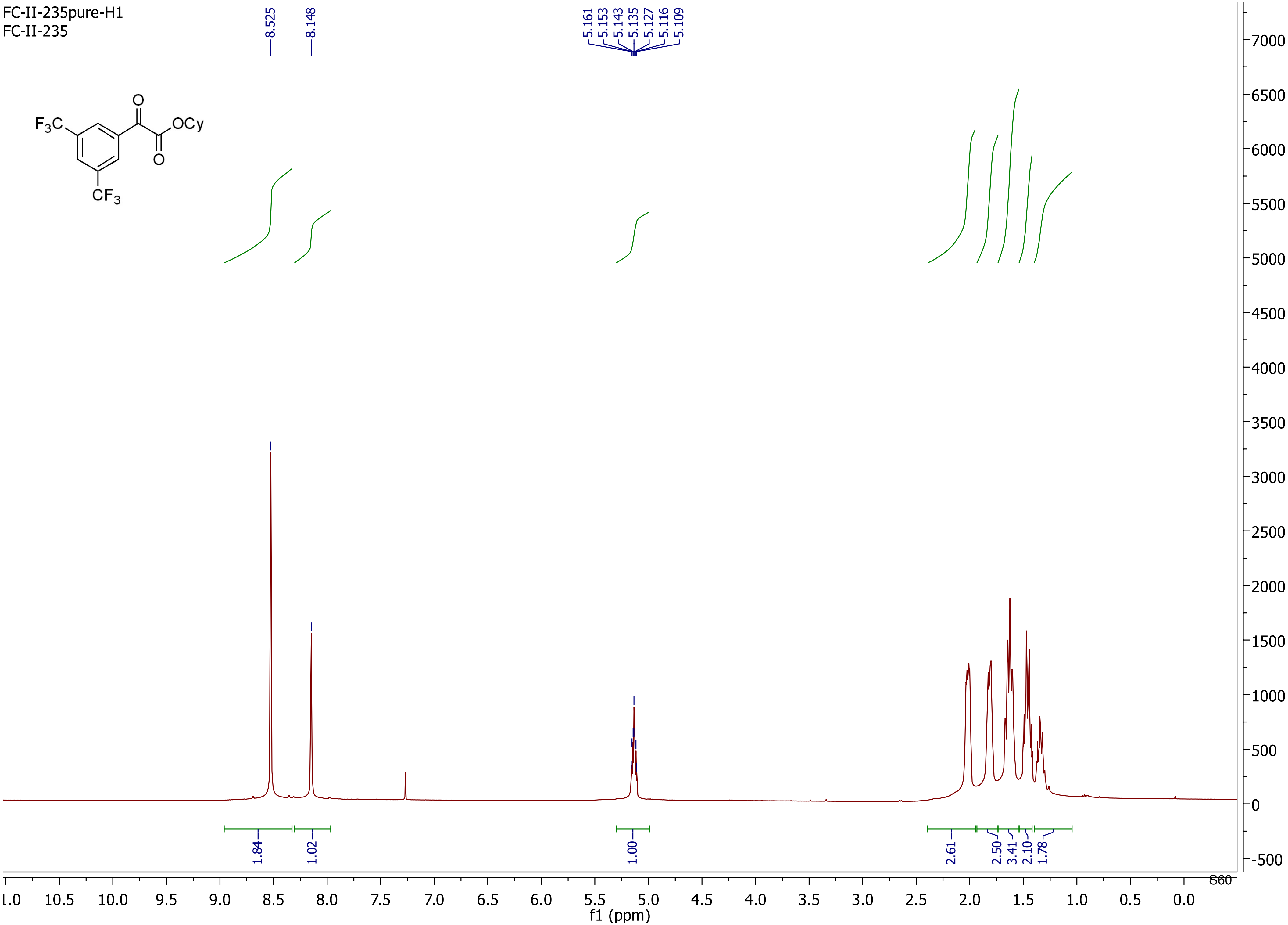

FC-II-195-4-pure-H1
Std proton
OCy
F
O
8.064 8.060 8.053 8.049 8.046 8.035 8.031
7.198 7.194 7.181 7.177 7.164
5.097 5.087 5.079 5.071 5.060
2.002 1.986 1.979 1.797 1.789 1.780 1.778 1.771 1.632 1.625 1.604 1.585 1.566 1.457 1.443 1.436 1.431 1.415 1.409 1.388 1.327 1.306 1.302 1.280
1.78
1.85
1.00
2.24
2.23
3.18
2.18
1.24
f1 (ppm)

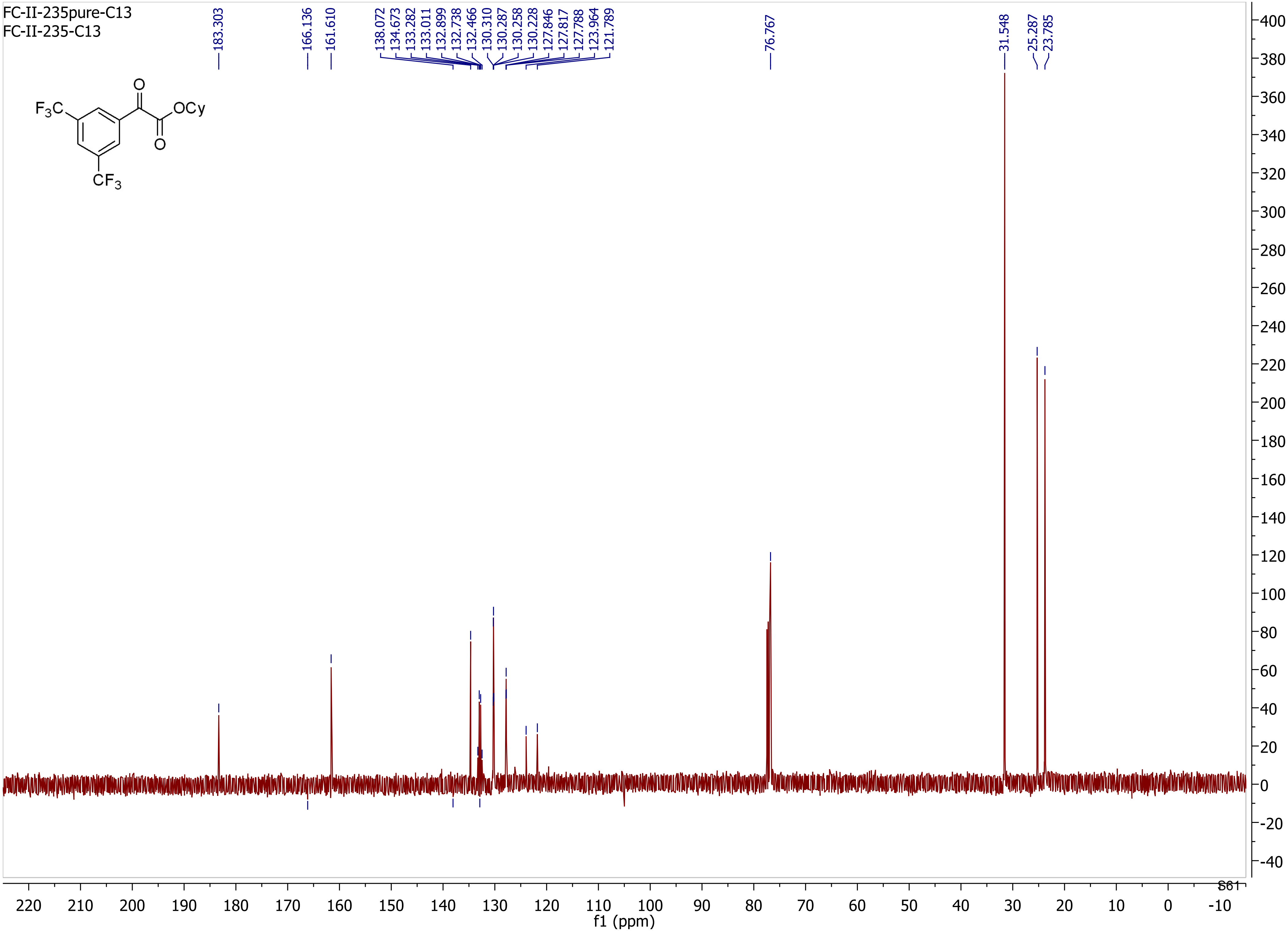

FC-II-195-4-pure-C13
FC-II-195-4-c13
OCy
F
185.209
167.961
165.908
163.461
133.100
133.066
133.022
129.325
129.302
116.762
116.556
116.379
75.846
31.640
25.366
23.866
f1 (ppm)


OCy
O
O
7.887
7.871
7.304
7.288
5.102
5.093
5.084
5.076
5.066
2.424
2.002
1.990
1.792
1.783
1.774
1.766
1.603
1.584
1.566
1.451
1.432
1.425
1.411
1.405
1.384
1.301
1.281
1.275
1.86
2.15
1.00
3.62
2.58
2.63
3.82
2.56
1.37
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
f1 (ppm)

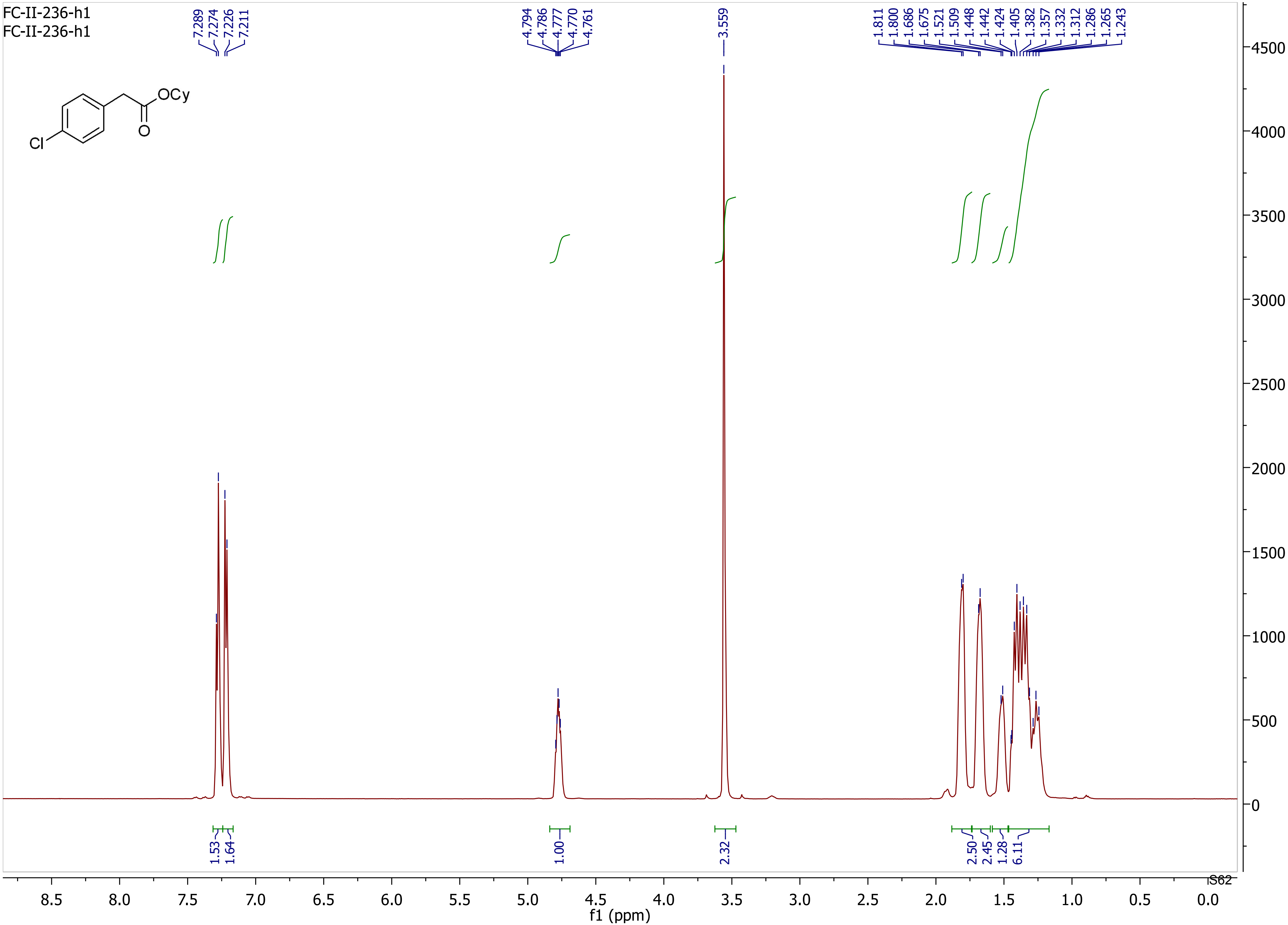

OCy
186.730
164.110
146.332
130.309
130.275
129.844
75.499
31.667
25.409
23.872
22.133
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

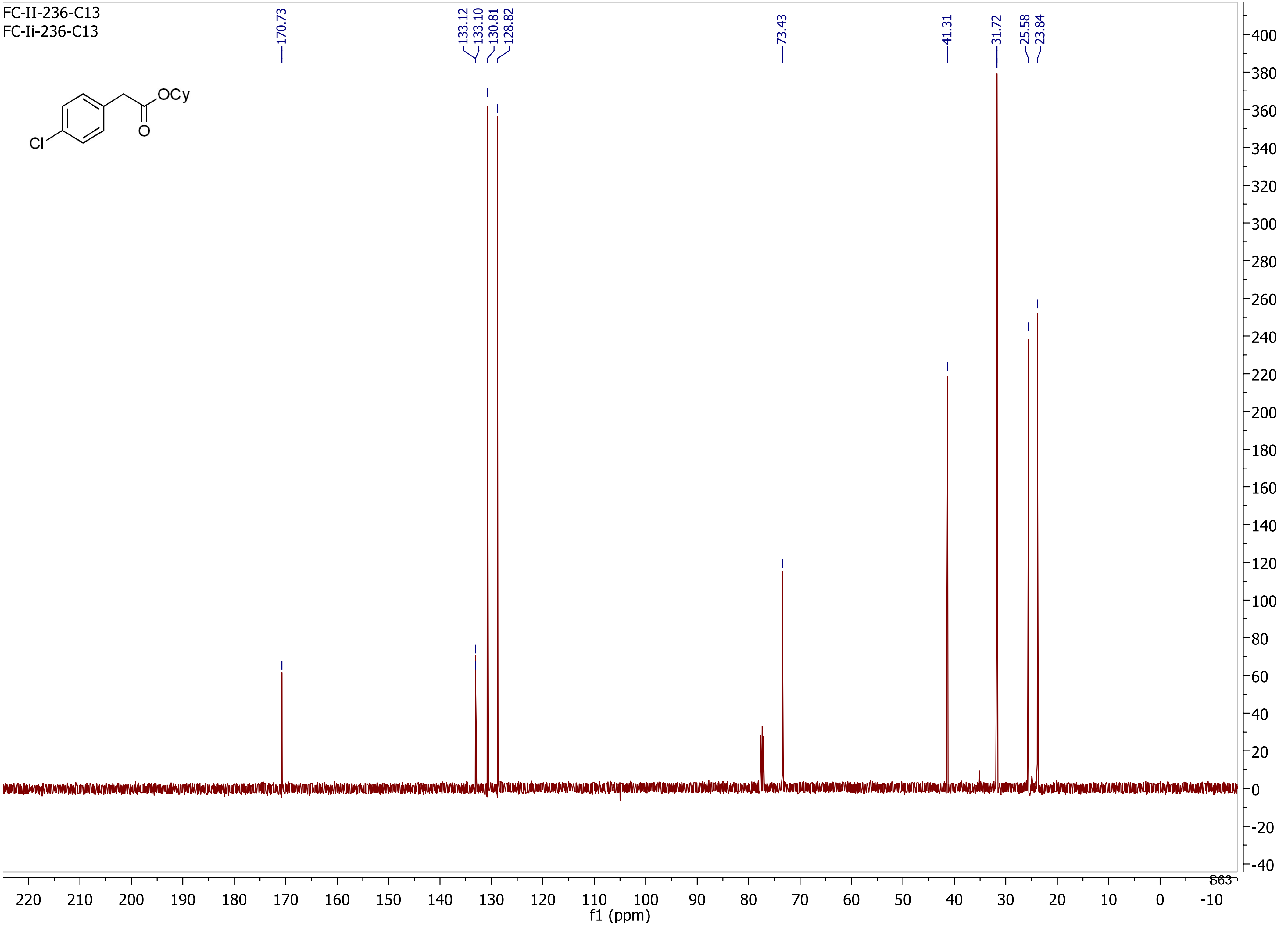
FC-II-235pure-H1
FC-II-235

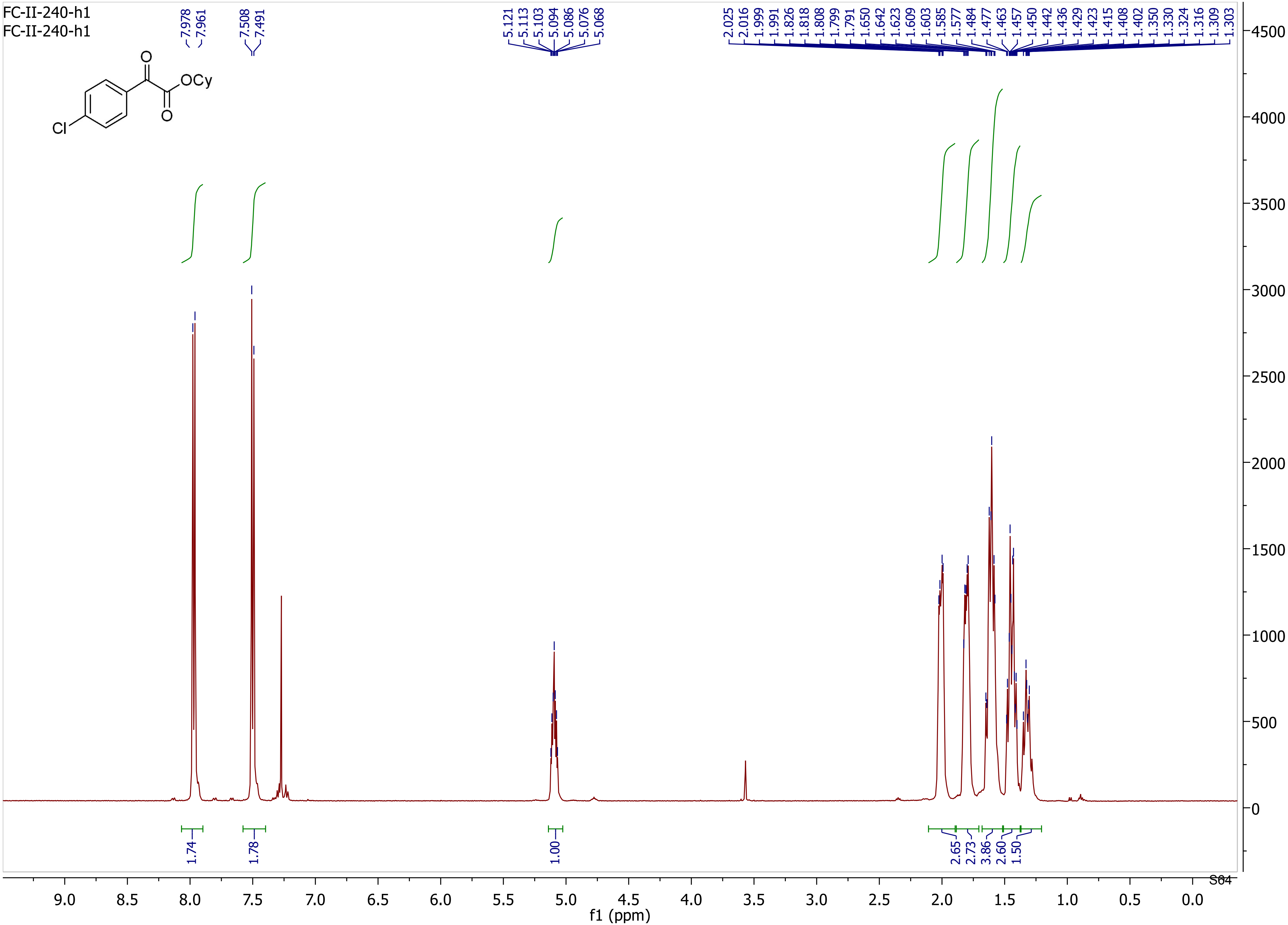


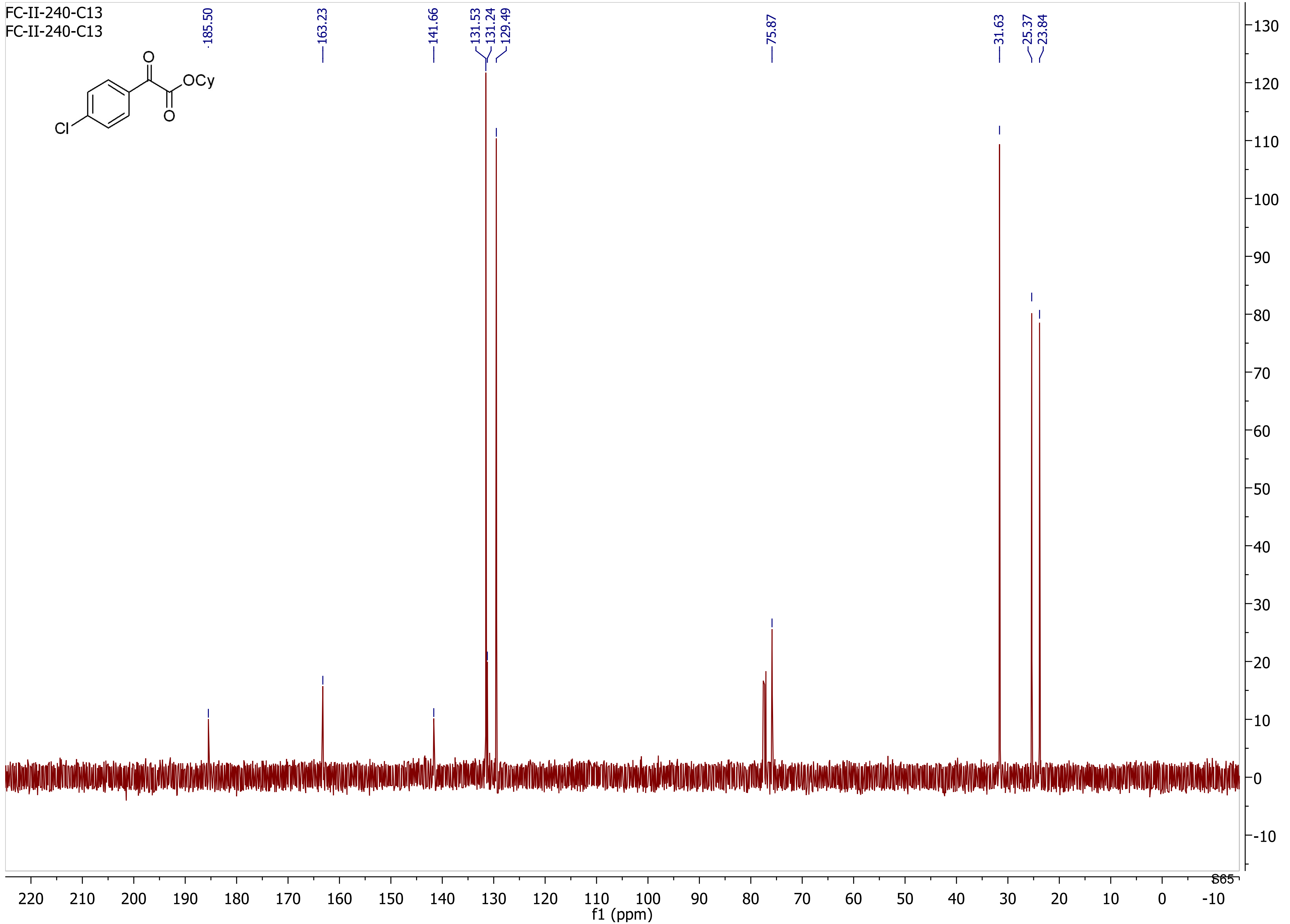


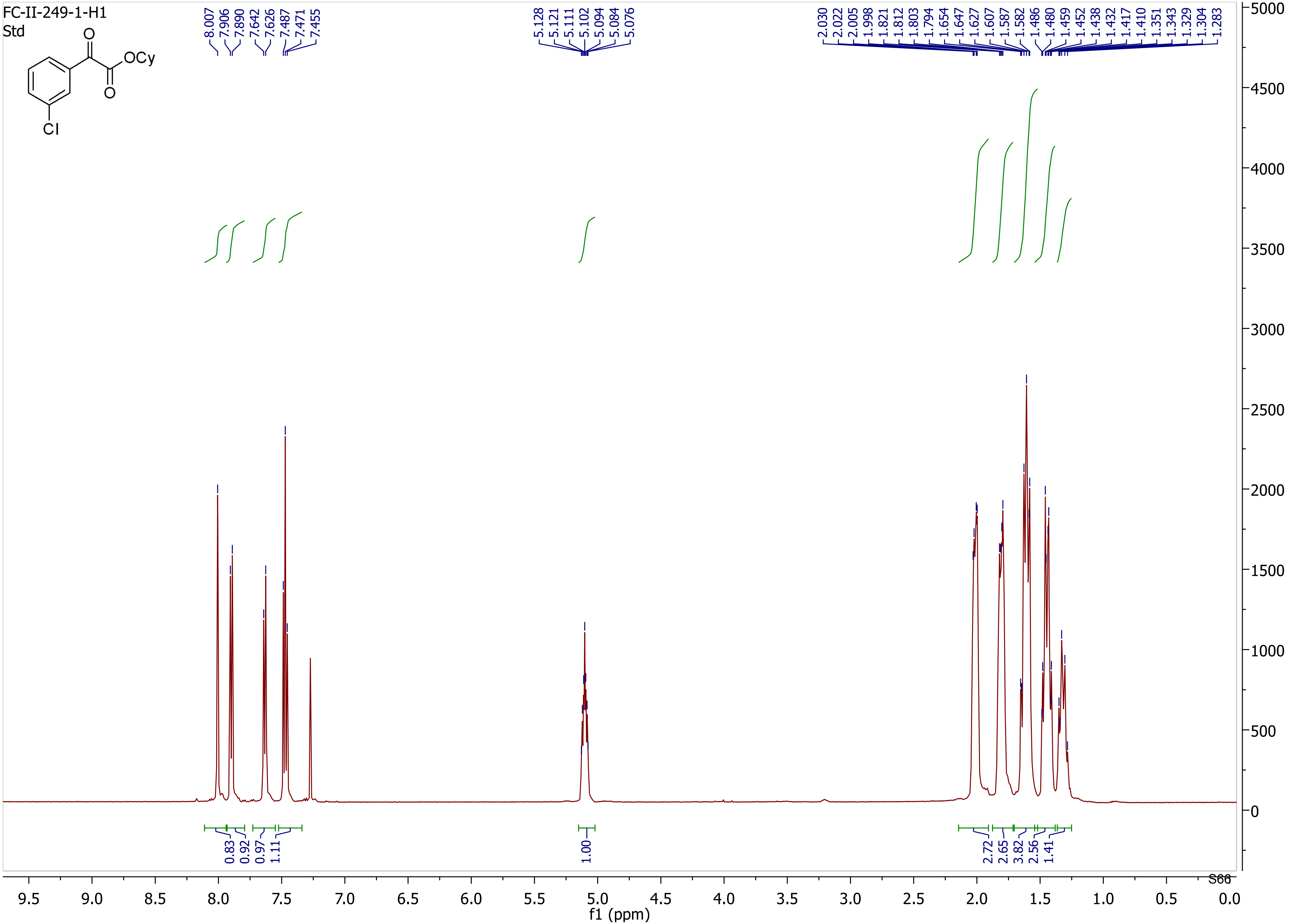
FC-II-235pure-C13
FC-II-235-C13
183.303
166.136
161.610
138.072
134.673
133.282
133.011
132.899
132.738
132.466
130.310
130.287
130.258
130.228
127.846
127.817
127.788
123.964
121.789
76.767
31.548
25.287
23.785
F3C
OCy
CF3
f1 (ppm)

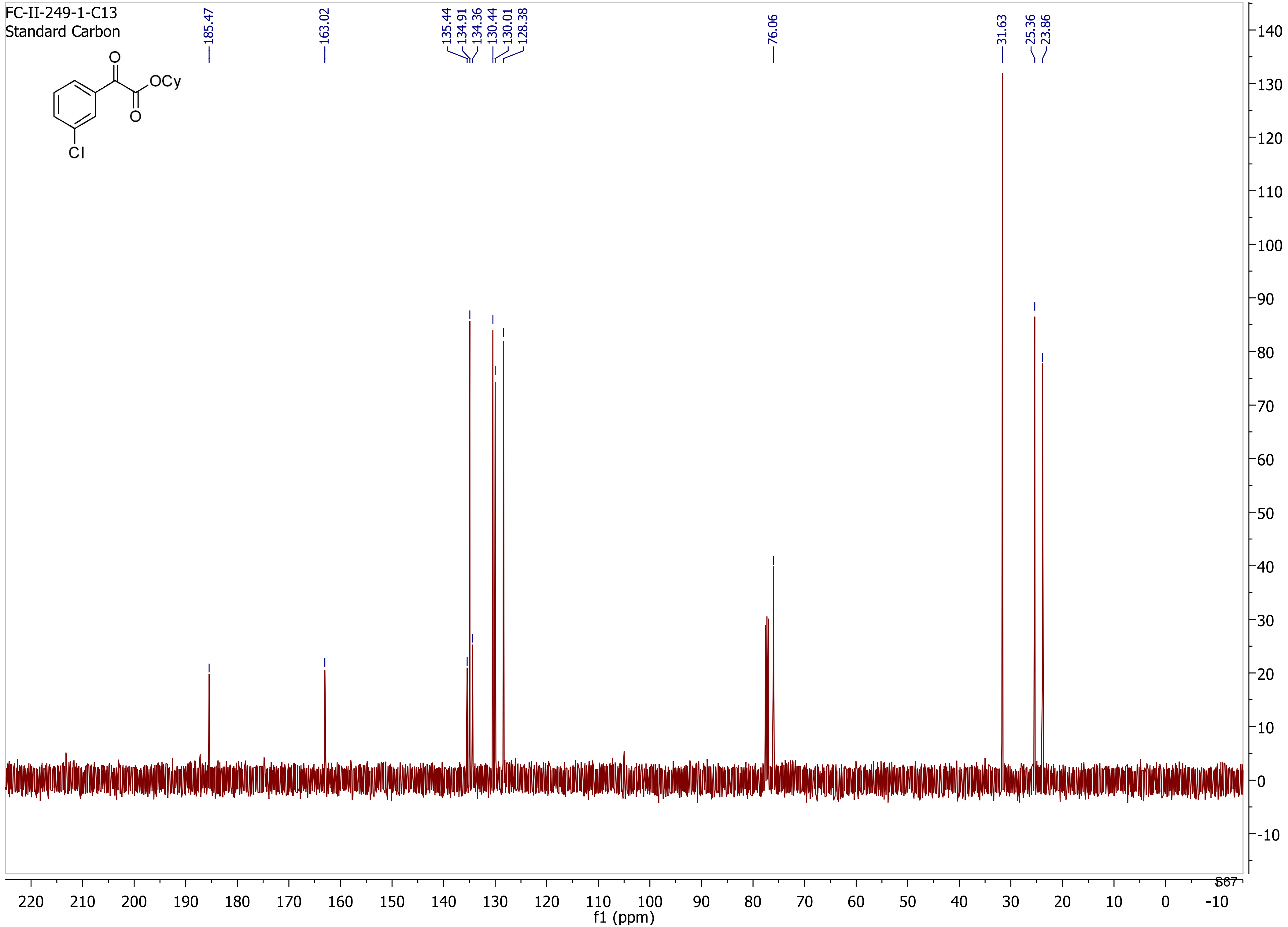

FC-II-236-h1
FC-II-236-h1
OCy
O
Cl
7.289
7.274
7.226
7.211
4.794
4.786
4.777
4.770
4.761
3.559
1.811
1.800
1.686
1.675
1.521
1.509
1.448
1.442
1.424
1.405
1.382
1.357
1.332
1.312
1.286
1.265
1.243
1.53
1.64
1.00
2.32
2.50
2.45
1.28
6.11
f1 (ppm)

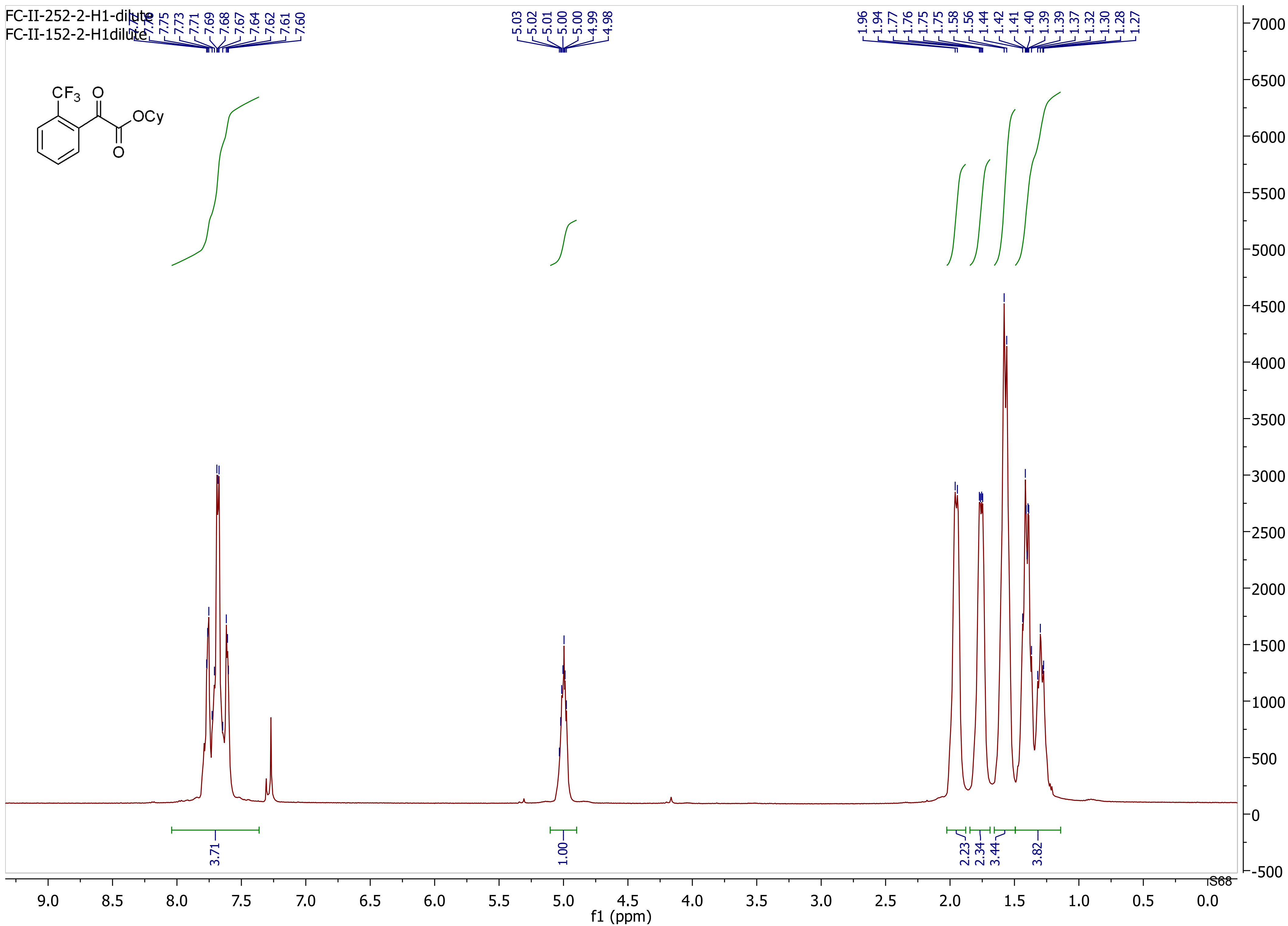

FC-II-236-C13
FC-Ii-236-C13
OCy
Cl
O
170.73
133.12
133.10
130.81
128.82
73.43
41.31
31.72
25.58
23.84
f1 (ppm)

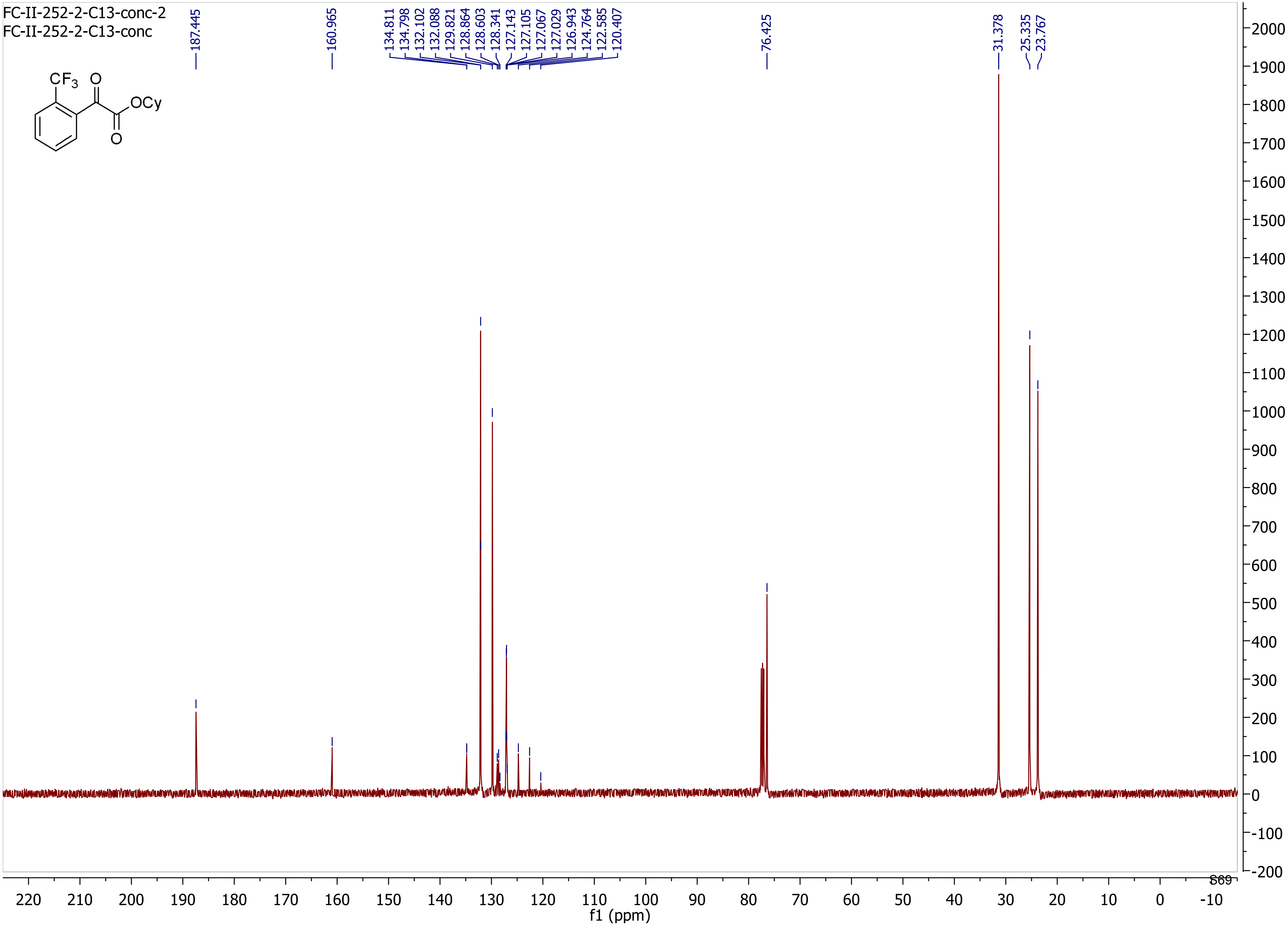

FC-II-240-h1
FC-II-240-h1
OCy
O
O
Cl
7.978
7.961
7.508
7.491
5.121
5.113
5.103
5.094
5.086
5.076
5.068
2.025
2.016
1.999
1.991
1.826
1.818
1.808
1.799
1.791
1.650
1.642
1.623
1.609
1.603
1.585
1.577
1.484
1.477
1.463
1.457
1.450
1.442
1.436
1.429
1.423
1.415
1.408
1.402
1.350
1.330
1.324
1.316
1.309
1.303
1.74
1.78
1.00
2.65
2.73
3.86
2.60
1.50
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
f1 (ppm)
4500
4000
3500
3000
2500
2000
1500
1000
500
0

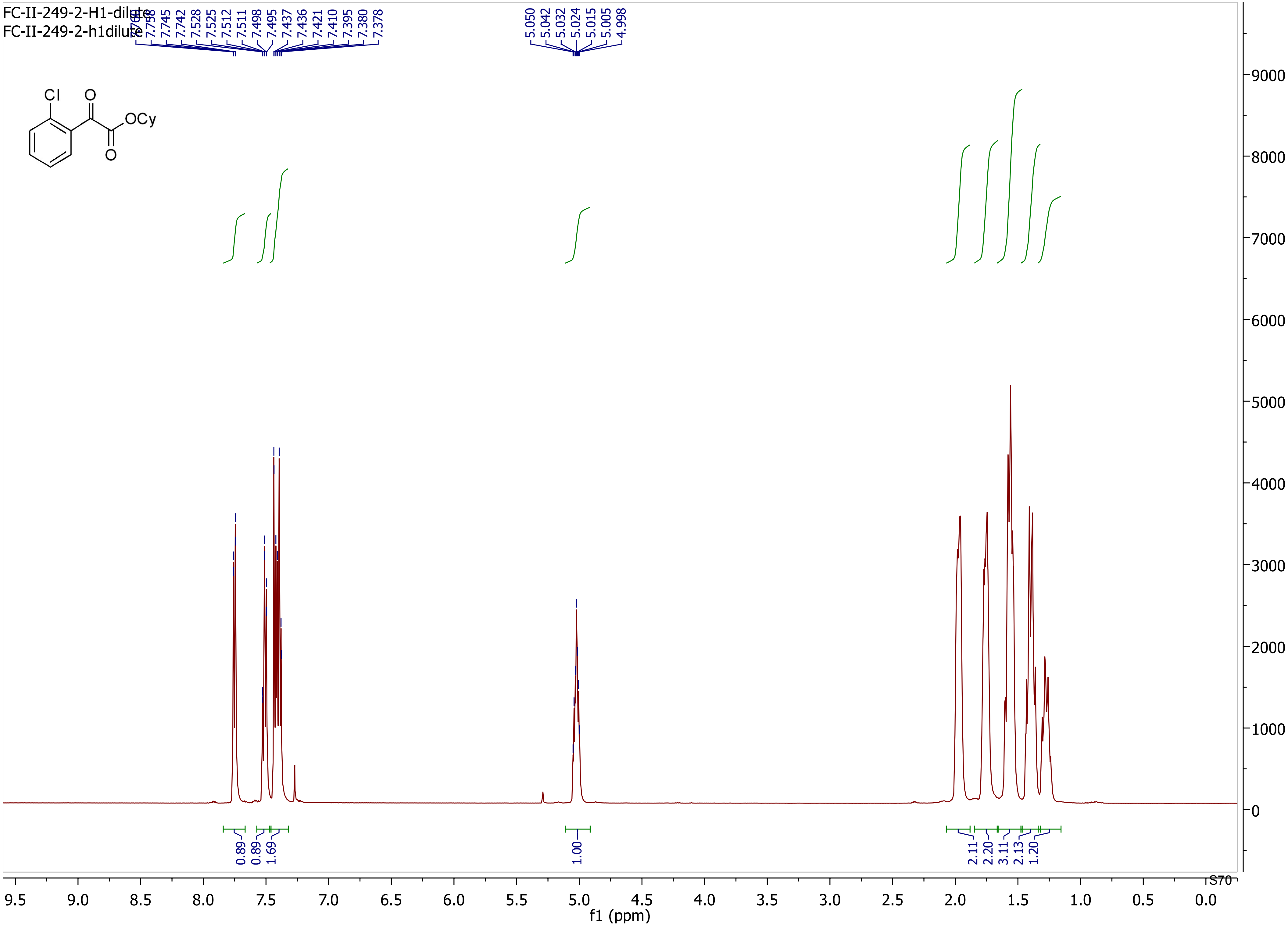

FC-II-240-C13
FC-II-240-C13
O
OCy
O
Cl
185.50
163.23
141.66
131.53
131.24
129.49
75.87
31.63
25.37
23.84
f1 (ppm)

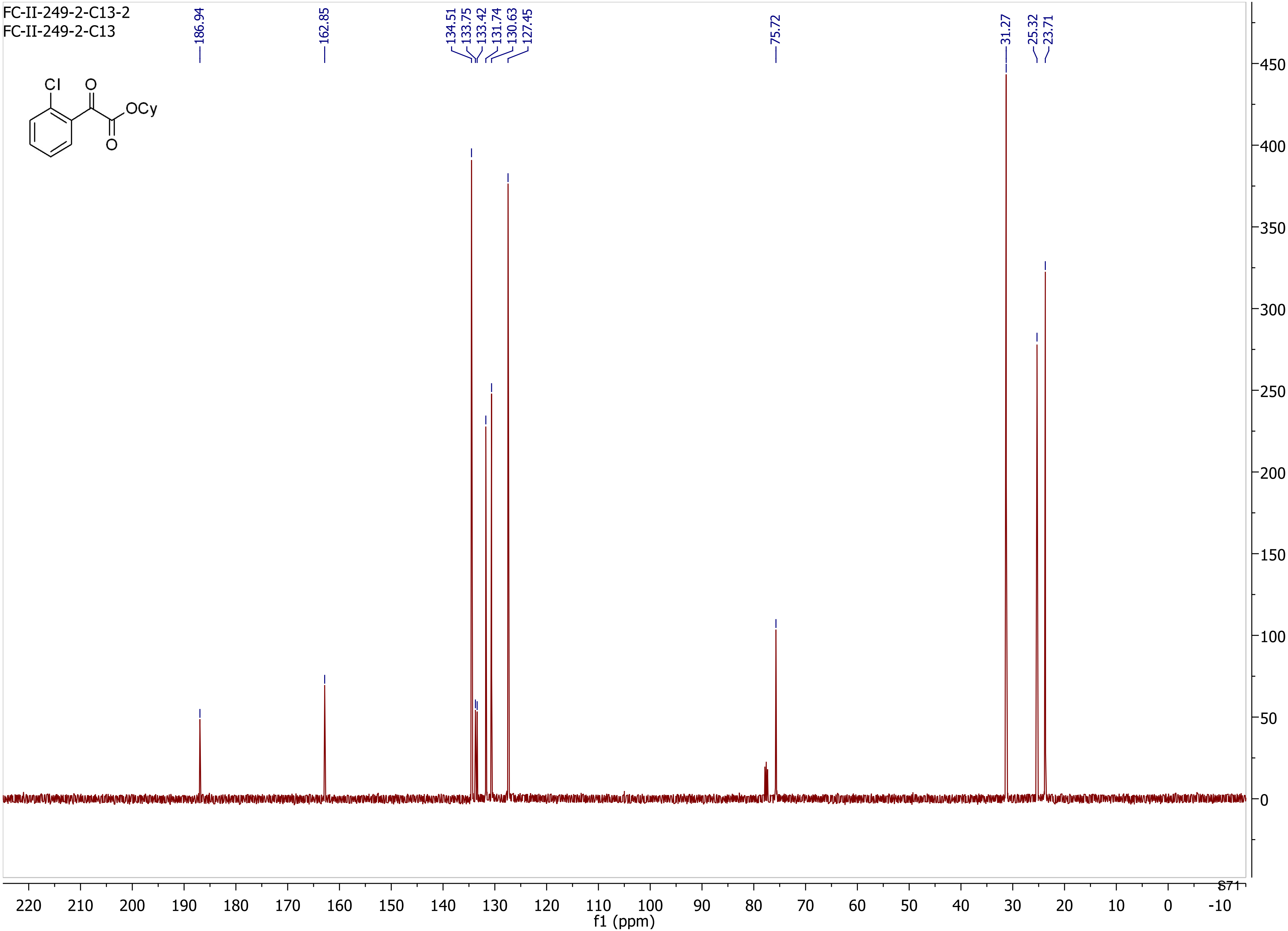

FC-II-249-1-H1
Std
OCy
Cl
8.007
7.906
7.890
7.642
7.626
7.487
7.471
7.455
5.128
5.121
5.111
5.102
5.094
5.084
5.076
2.030
2.022
2.005
1.998
1.821
1.812
1.803
1.794
1.654
1.647
1.627
1.607
1.587
1.582
1.486
1.480
1.459
1.452
1.438
1.432
1.417
1.410
1.351
1.343
1.329
1.304
1.283
0.83
0.92
0.97
1.11
1.00
2.72
2.65
3.82
2.56
1.41
f1 (ppm)

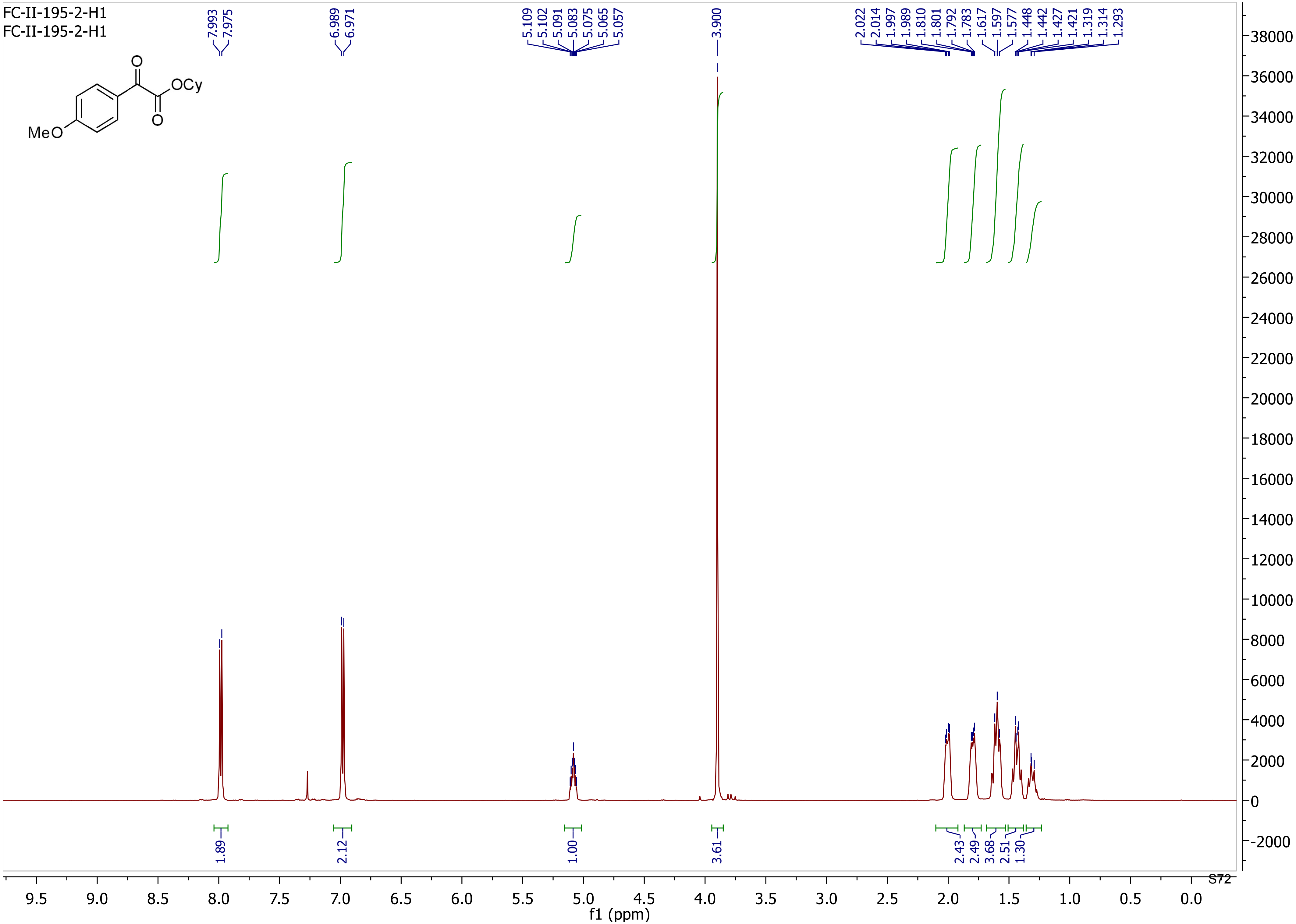

FC-II-249-1-C13
Standard Carbon
OCy
Cl
185.47
163.02
135.44
134.91
134.36
130.44
130.01
128.38
76.06
31.63
25.36
23.86
f1 (ppm)

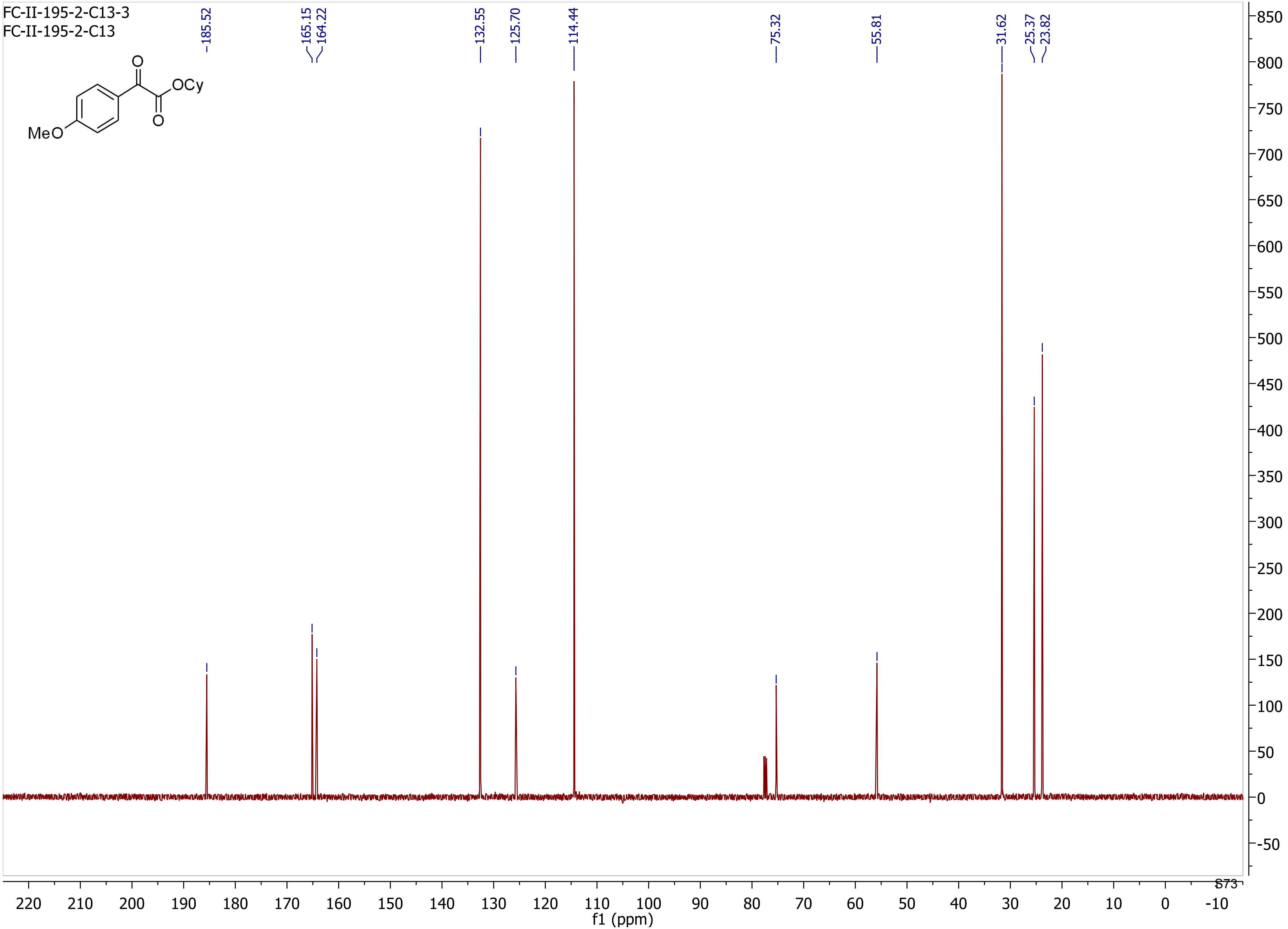

FC-II-252-2-H1-dilute
FC-II-152-2-H1dilute
f1 (ppm)

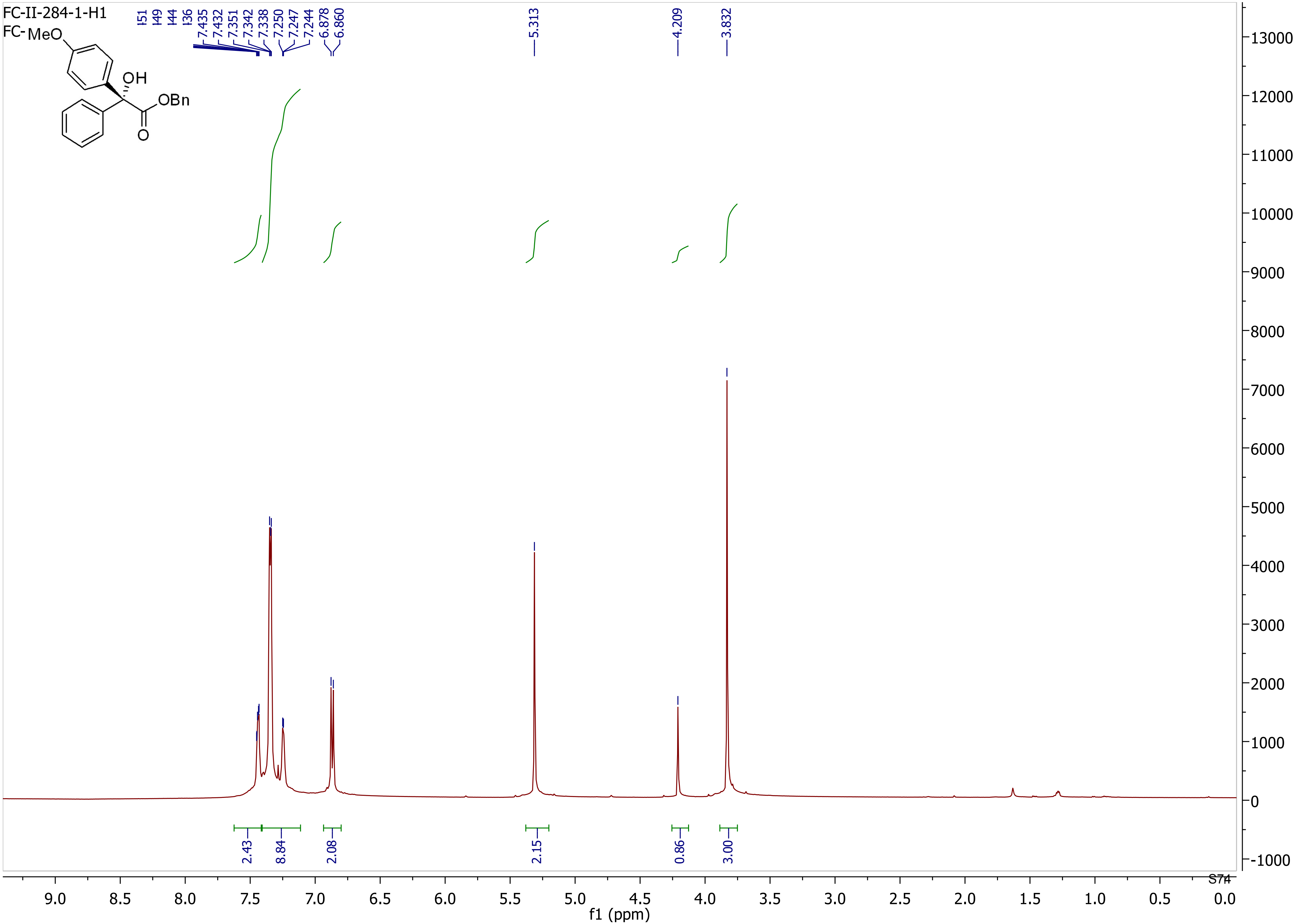

FC-II-252-2-C13-conc-2
FC-II-252-2-C13-conc
CF3
O
OCy
O
187.445
160.965
134.811
134.798
132.102
132.088
129.821
128.864
128.603
128.341
127.143
127.105
127.067
127.029
126.943
124.764
122.585
120.407
76.425
31.378
25.335
23.767
f1 (ppm)

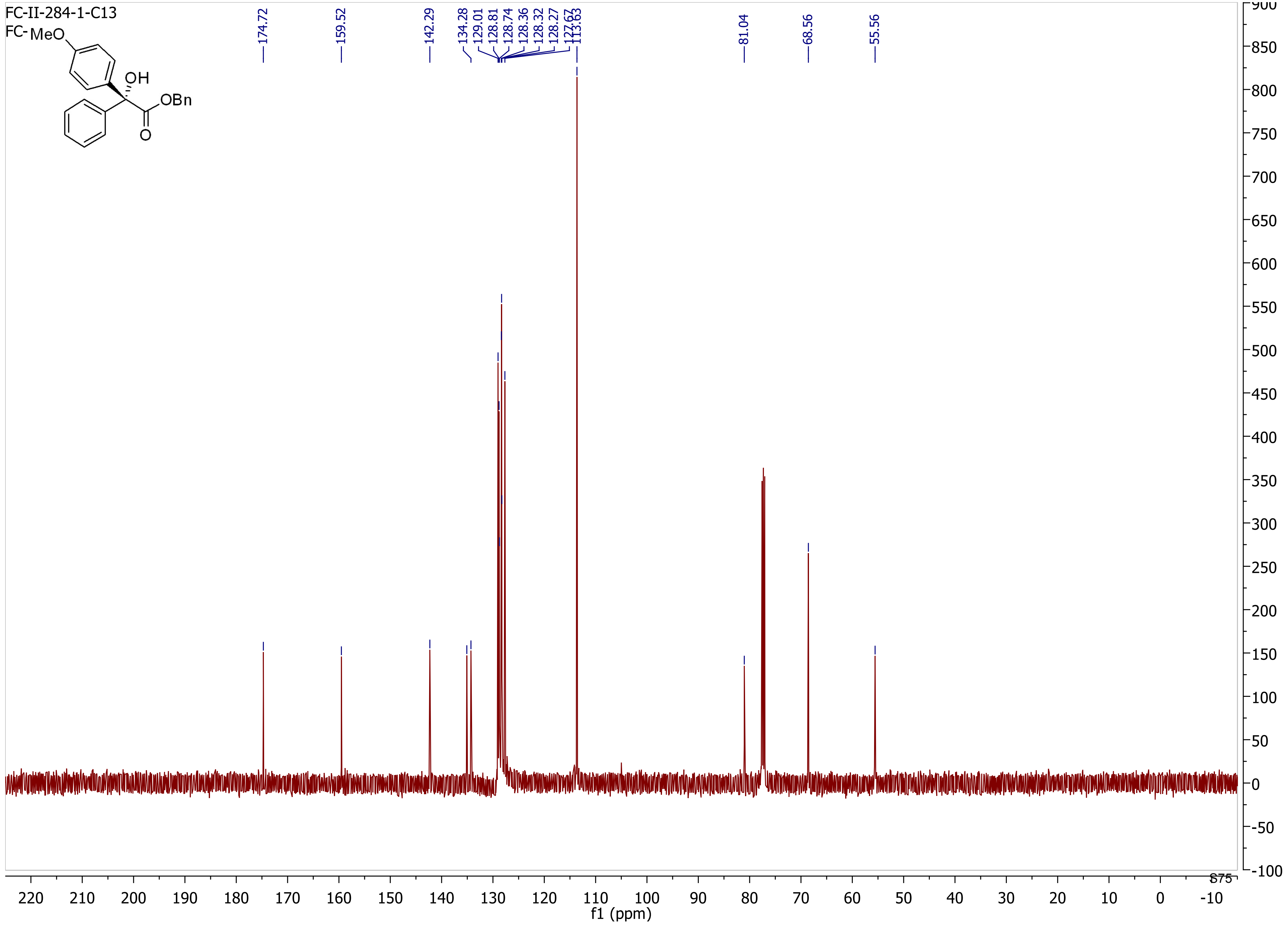

FC-II-249-2-H1-dilute
FC-II-249-2-h1dilute
7.760
7.758
7.745
7.742
7.528
7.525
7.512
7.511
7.498
7.495
7.437
7.436
7.421
7.410
7.395
7.380
7.378
5.050
5.042
5.032
5.024
5.015
5.005
4.998
Cl
O
OCy
O
0.89
0.89
1.69
1.00
2.11
2.20
3.11
2.13
1.20
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
9.5
9.0
8.5
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
f1 (ppm)


FC-II-249-2-C13-2
FC-II-249-2-C13
Cl
O
OCy
O
186.94
162.85
134.51
133.75
133.42
131.74
130.63
127.45
75.72
31.27
25.32
23.71
450
400
350
300
250
200
150
100
50
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

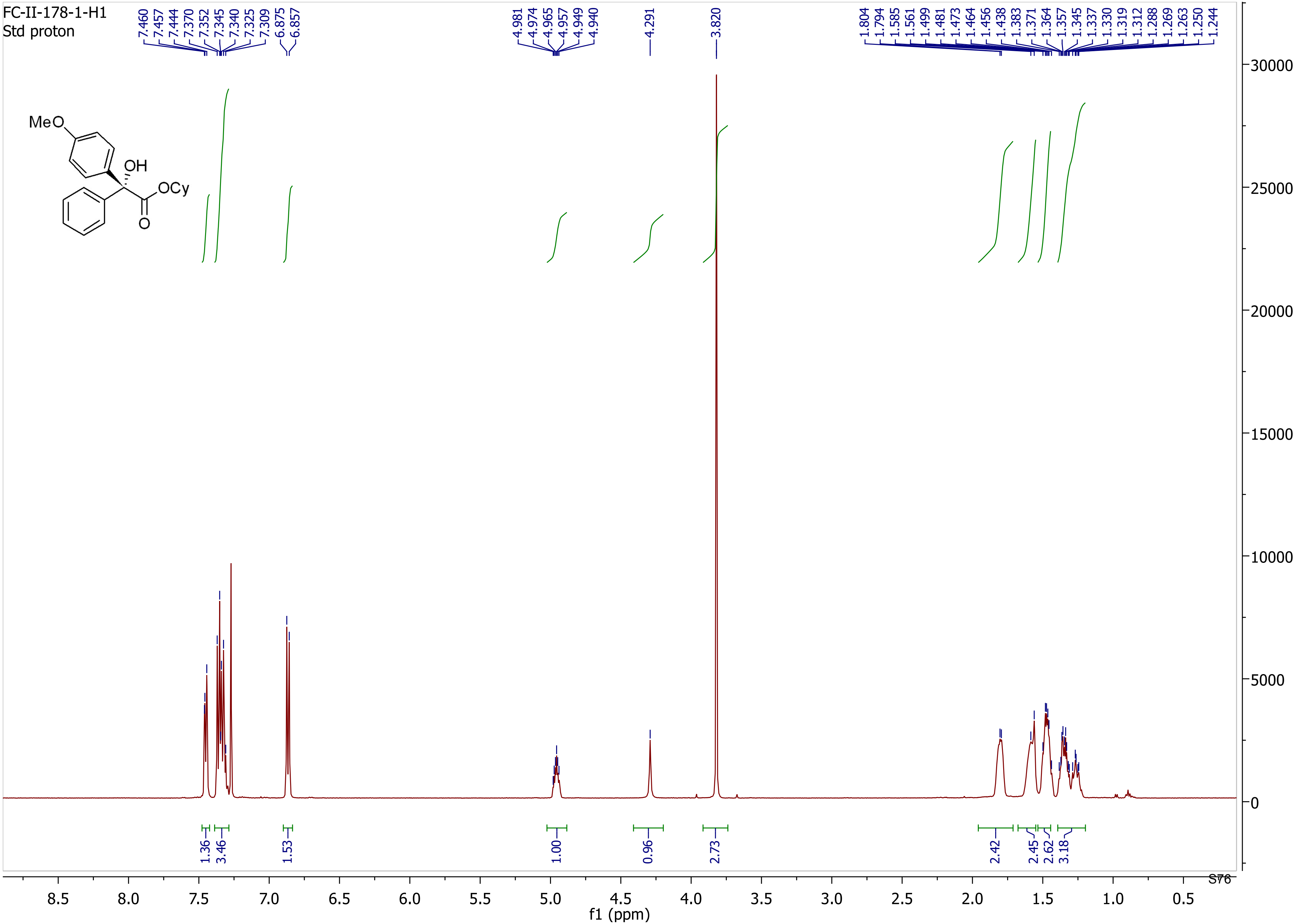

FC-II-195-2-H1
FC-II-195-2-H1
MeO
OCy
f1 (ppm)

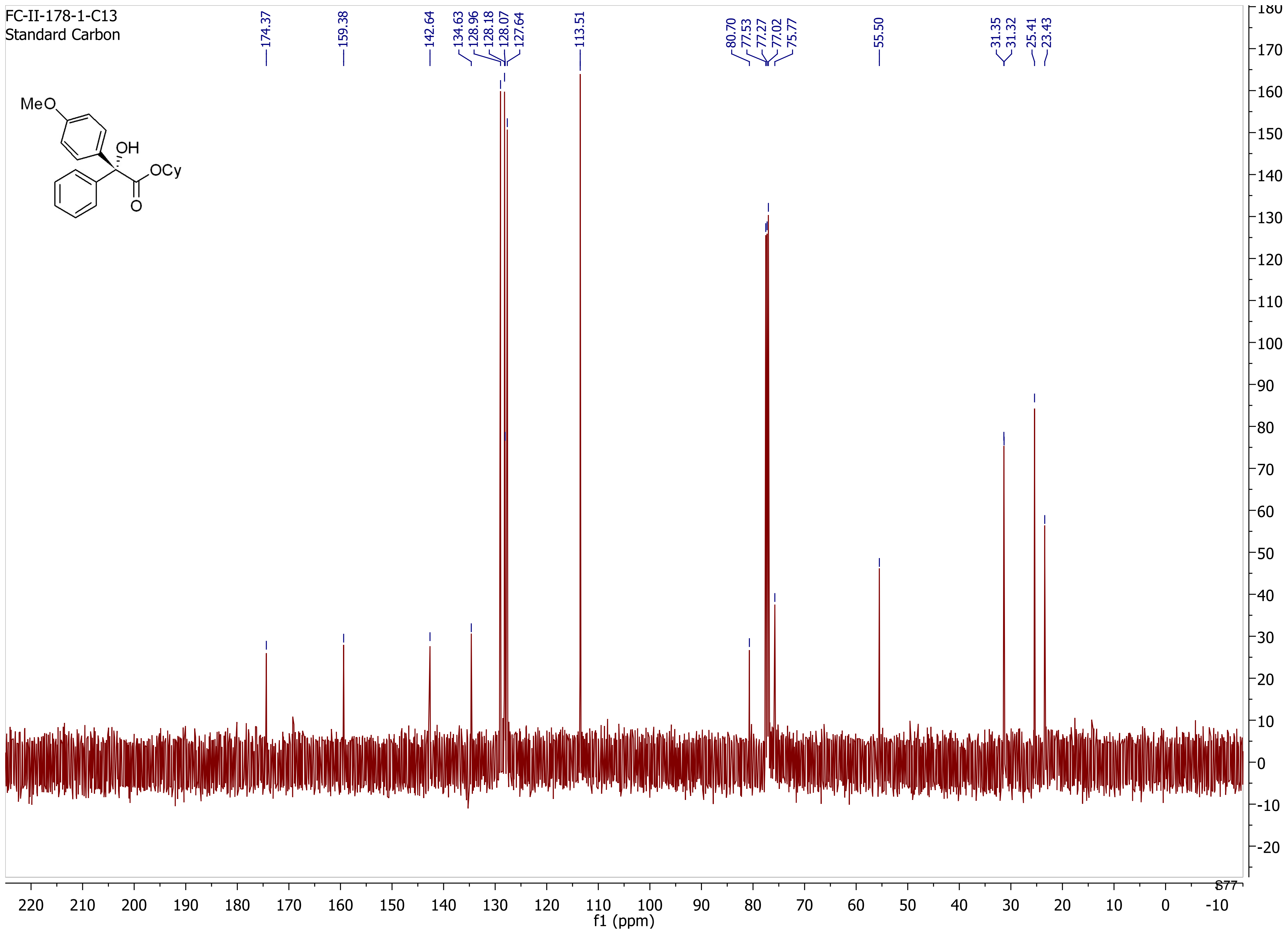

FC-II-195-2-C13-3
FC-II-195-2-C13
MeO
OCy
185.52
165.15
164.22
132.55
125.70
114.44
75.32
55.81
31.62
25.37
23.82
f1 (ppm)

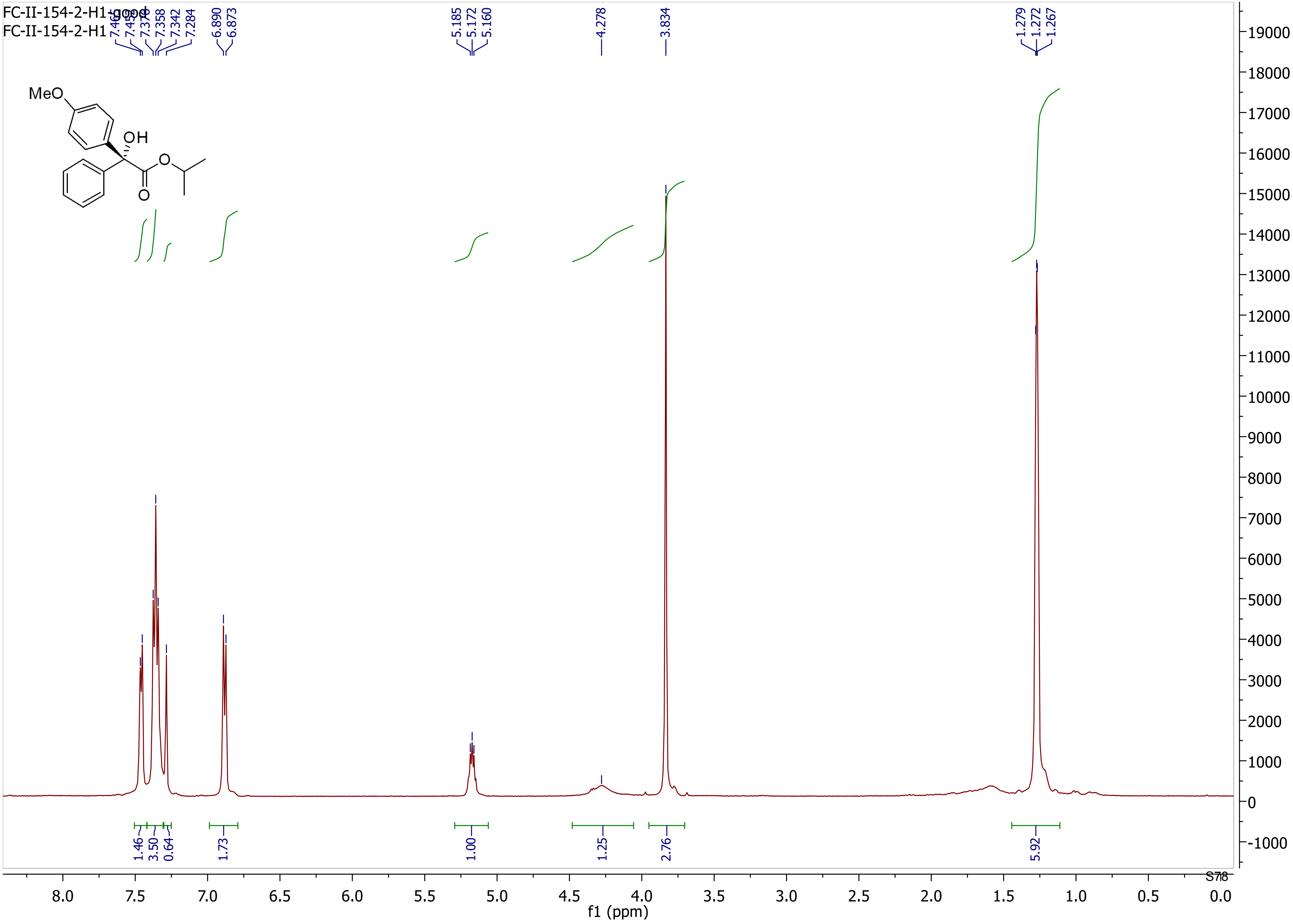

FC-II-284-1-H1
FC-
MeO
OH
OBn
O
7.435
7.432
7.351
7.342
7.338
7.250
7.247
7.244
6.878
6.860
5.313
4.209
3.832
2.43
8.84
2.08
2.15
0.86
3.00
f1 (ppm)

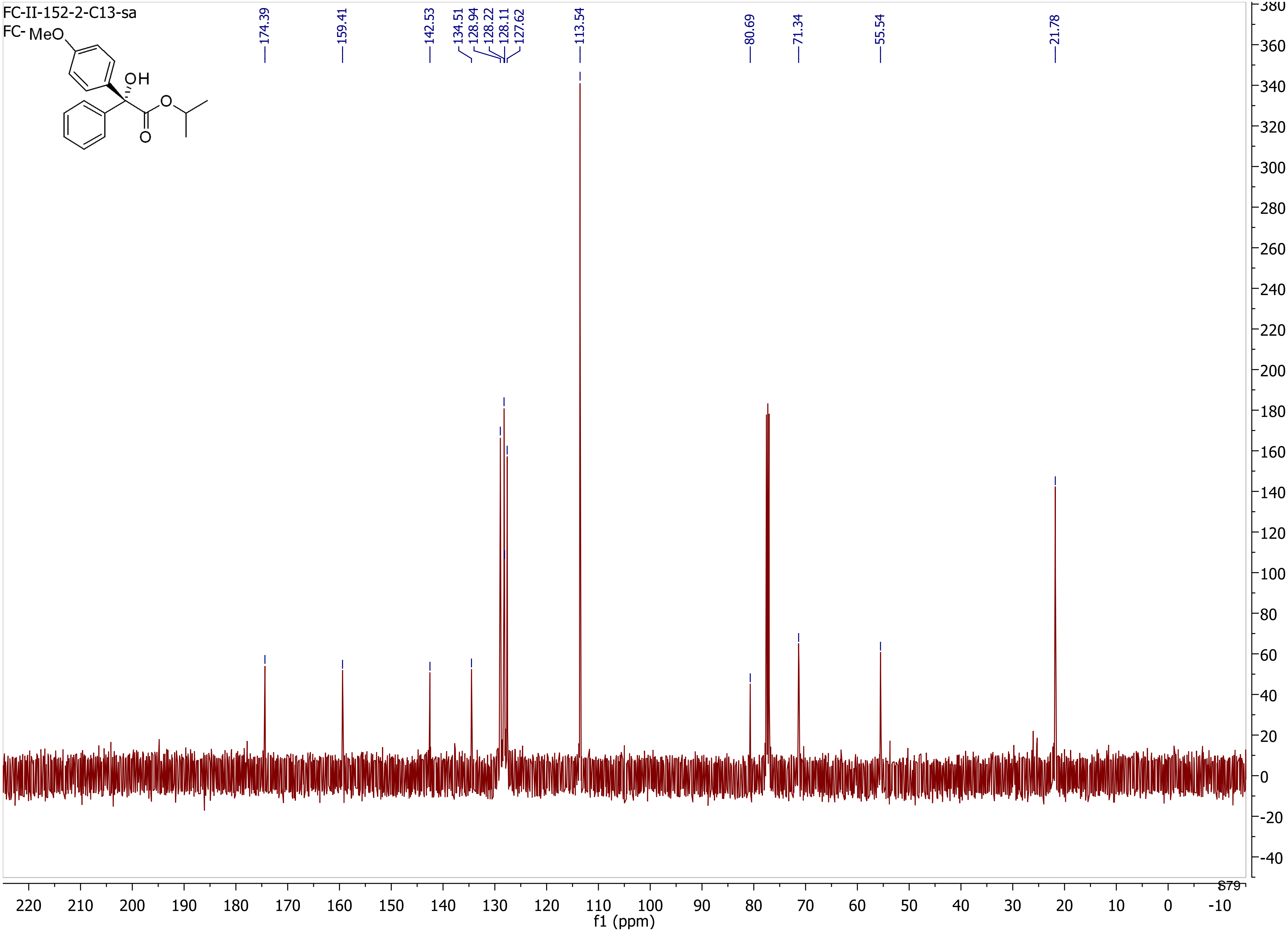

FC-II-284-1-C13
FC-
MeO
OH
OBn
O
174.72
159.52
142.29
134.28
129.01
128.81
128.74
128.36
128.32
128.27
127.67
113.63
81.04
68.56
55.56
f1 (ppm)

FC-II-178-1-H1
Std proton

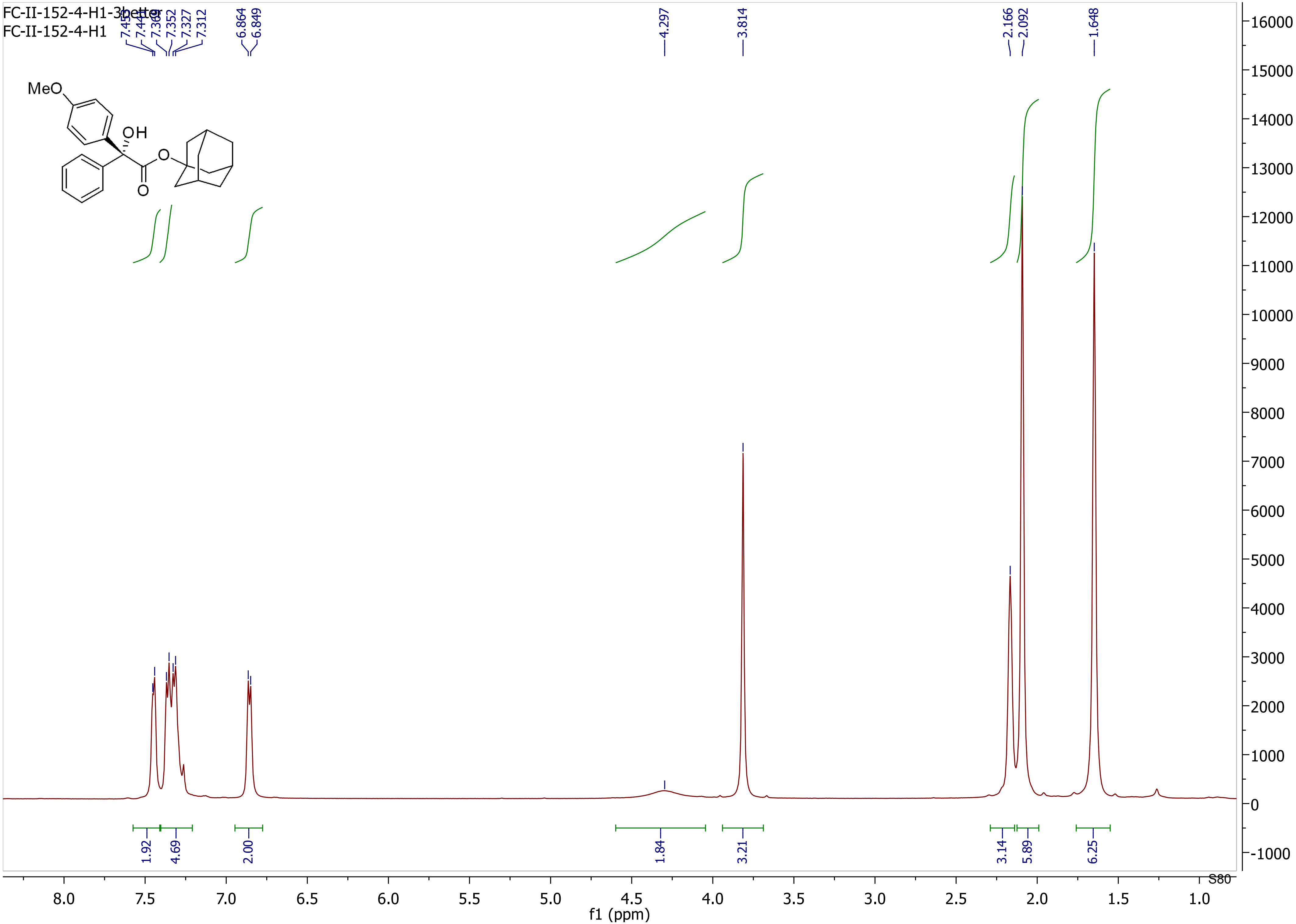



FC-II-178-1-C13
Standard Carbon
MeO
OH
OCy
O
174.37
159.38
142.64
134.63
128.96
128.18
128.07
127.64
113.51
80.70
77.53
77.27
77.02
75.77
55.50
31.35
31.32
25.41
23.43
f1 (ppm)

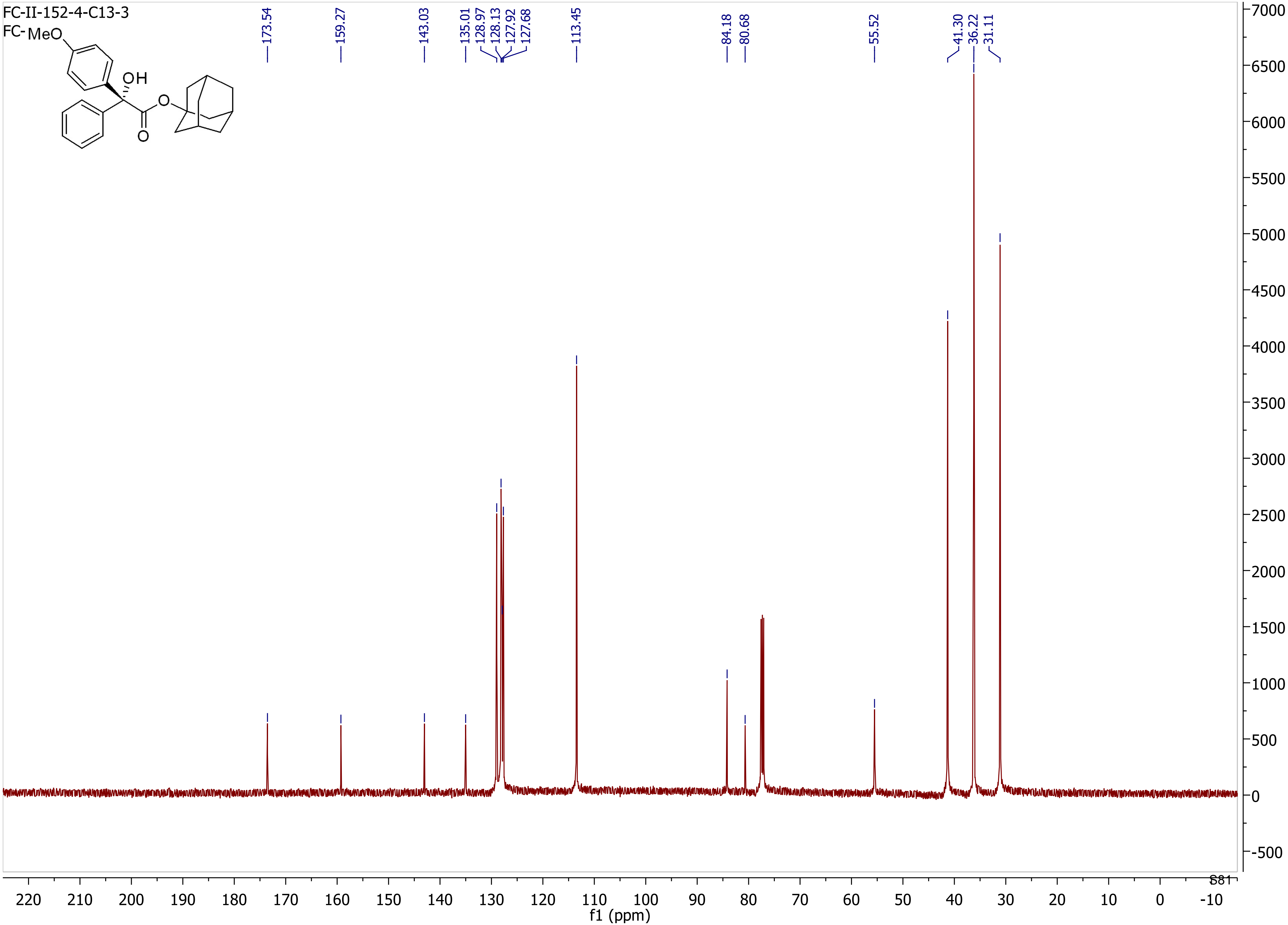

FC-II-154-2-H1-good
FC-II-154-2-H1
7.376
7.358
7.342
7.284
6.890
6.873
5.185
5.172
5.160
4.278
3.834
1.279
1.272
1.267
MeO
OH
O
O
1.46
3.50
0.64
1.73
1.00
1.25
2.76
5.92
f1 (ppm)
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000



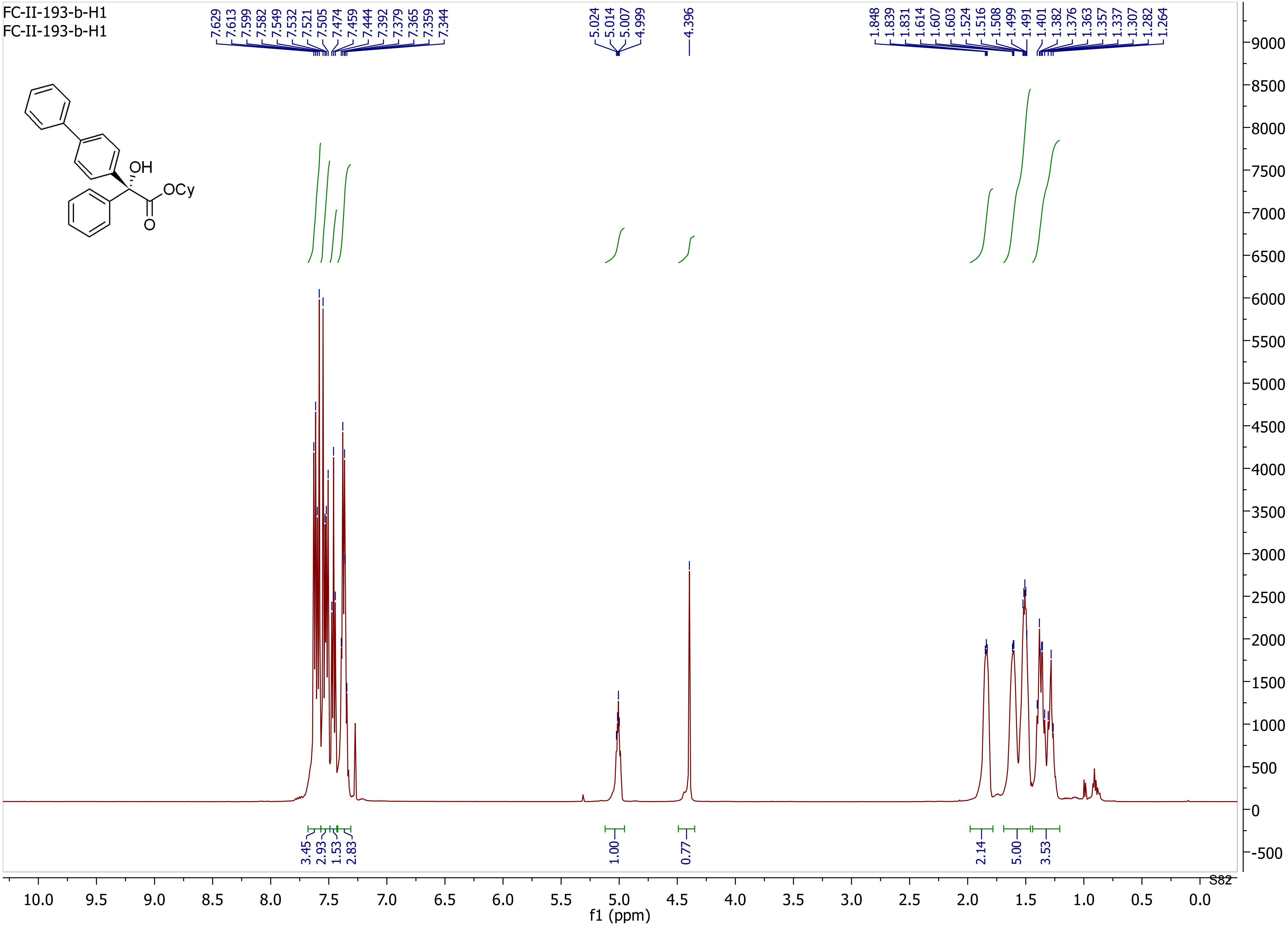
FC-II-152-2-C13-sa
FC-
MeO
OH
O
O
174.39
159.41
142.53
134.51
128.94
128.22
128.11
127.62
113.54
80.69
71.34
55.54
21.78
f1 (ppm)

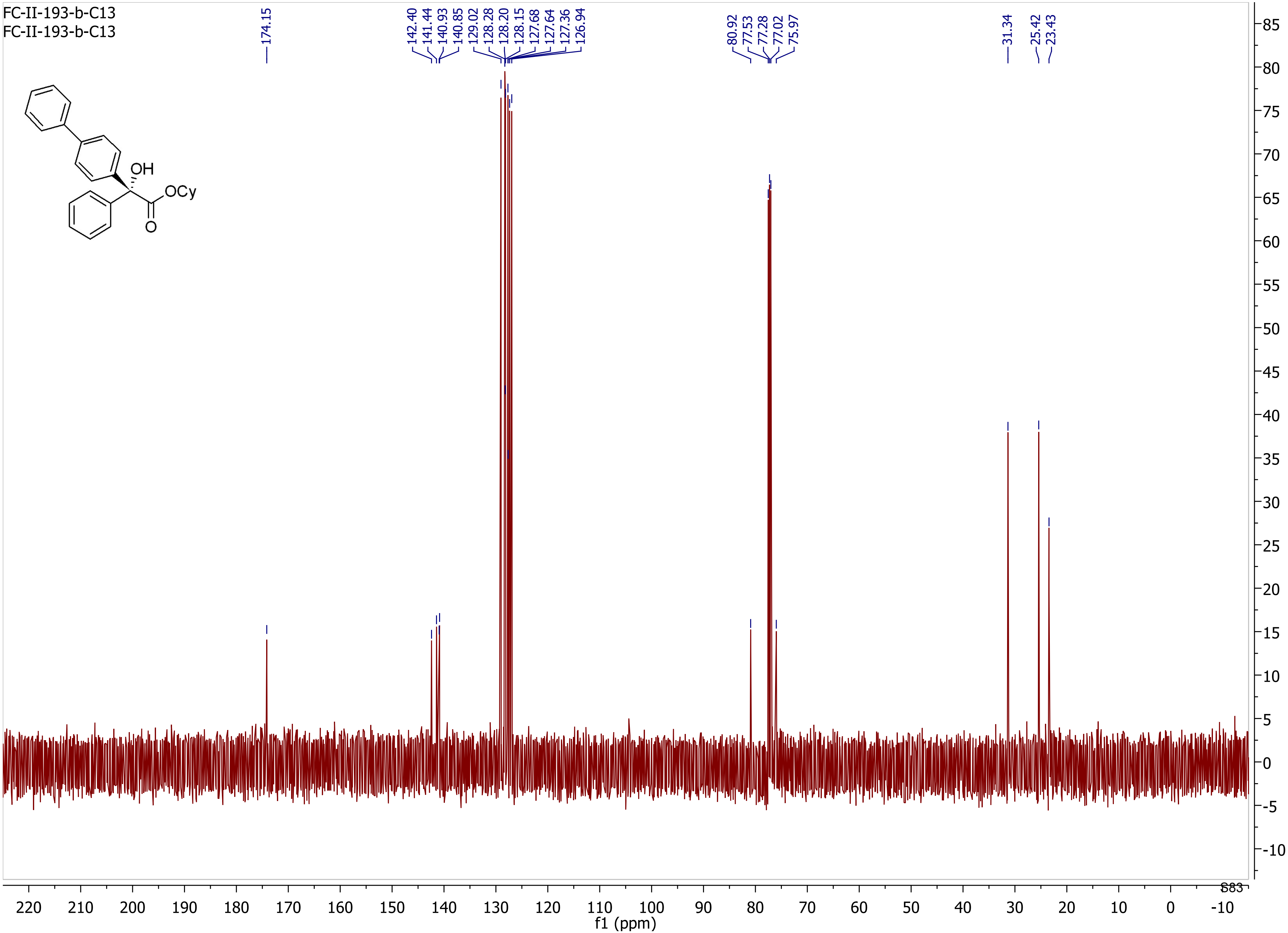

FC-II-152-4-H1-3better
FC-II-152-4-H1
7.451
7.444
7.369
7.352
7.327
7.312
6.864
6.849
4.297
3.814
2.166
2.092
1.648
MeO
OH
O
O
1.92
4.69
2.00
1.84
3.21
3.14
5.89
6.25
f1 (ppm)

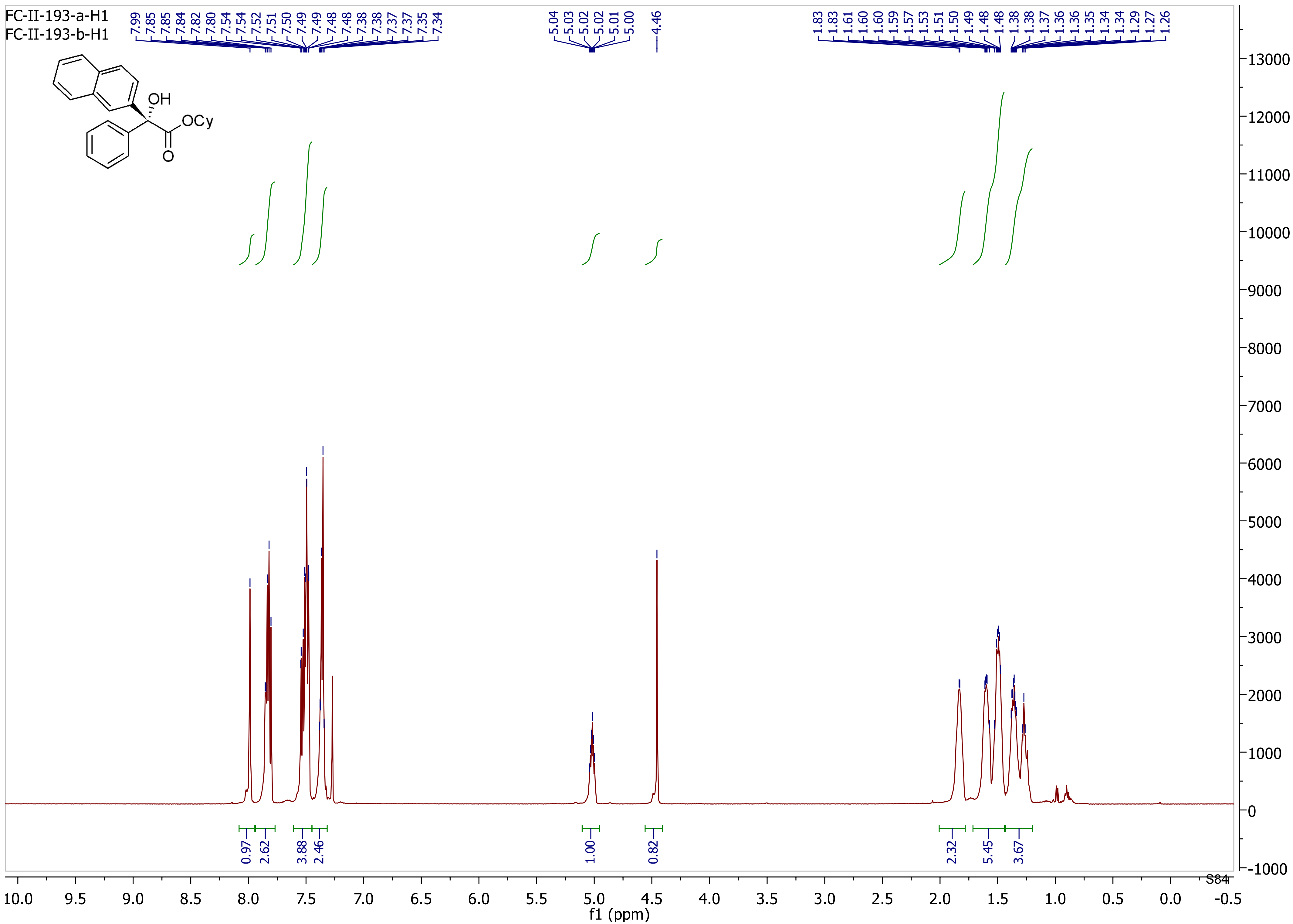
FC-II-152-4-C13-3
FC-



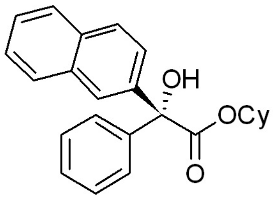
FC-II-193-b-H1
FC-II-193-b-H1

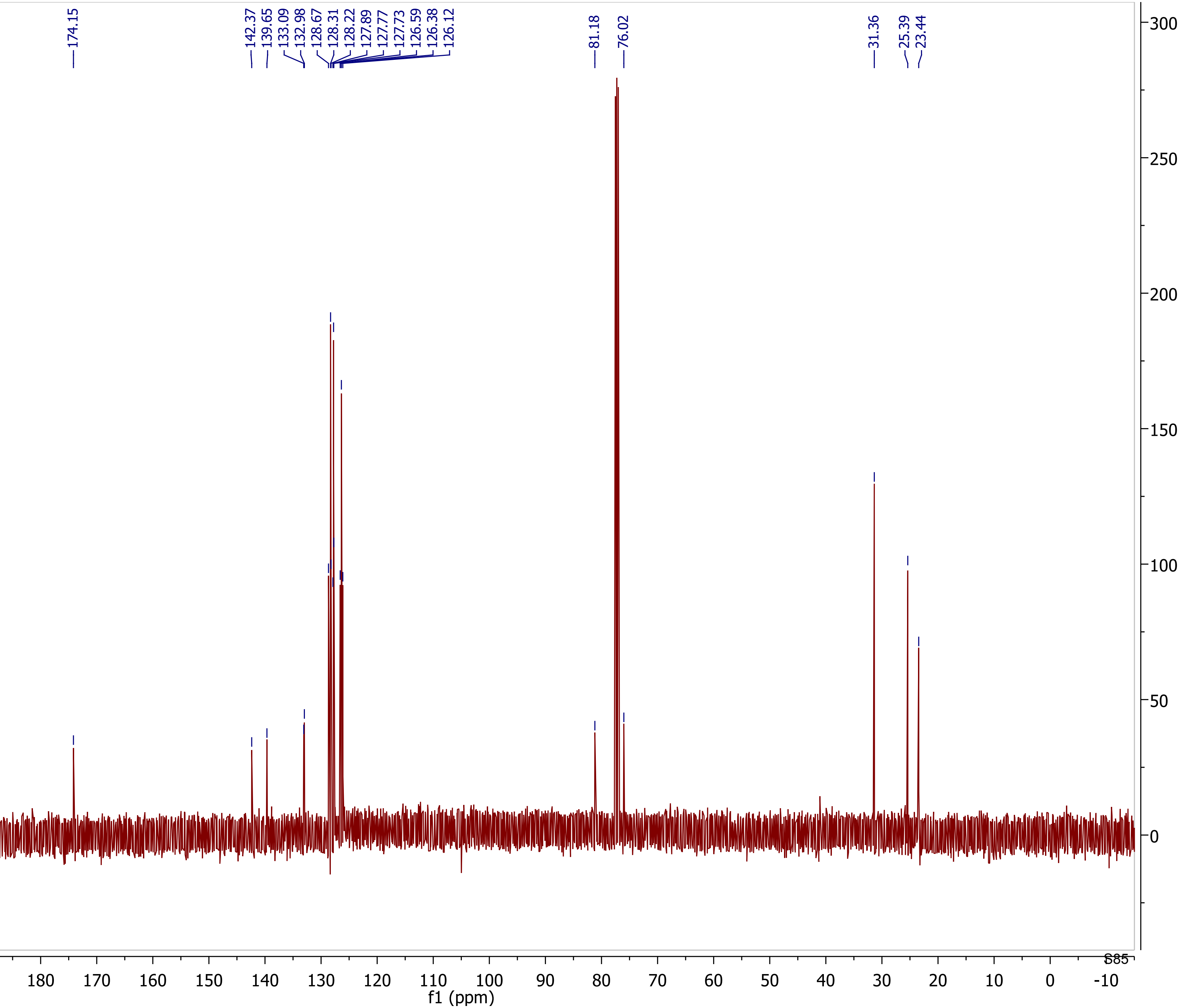


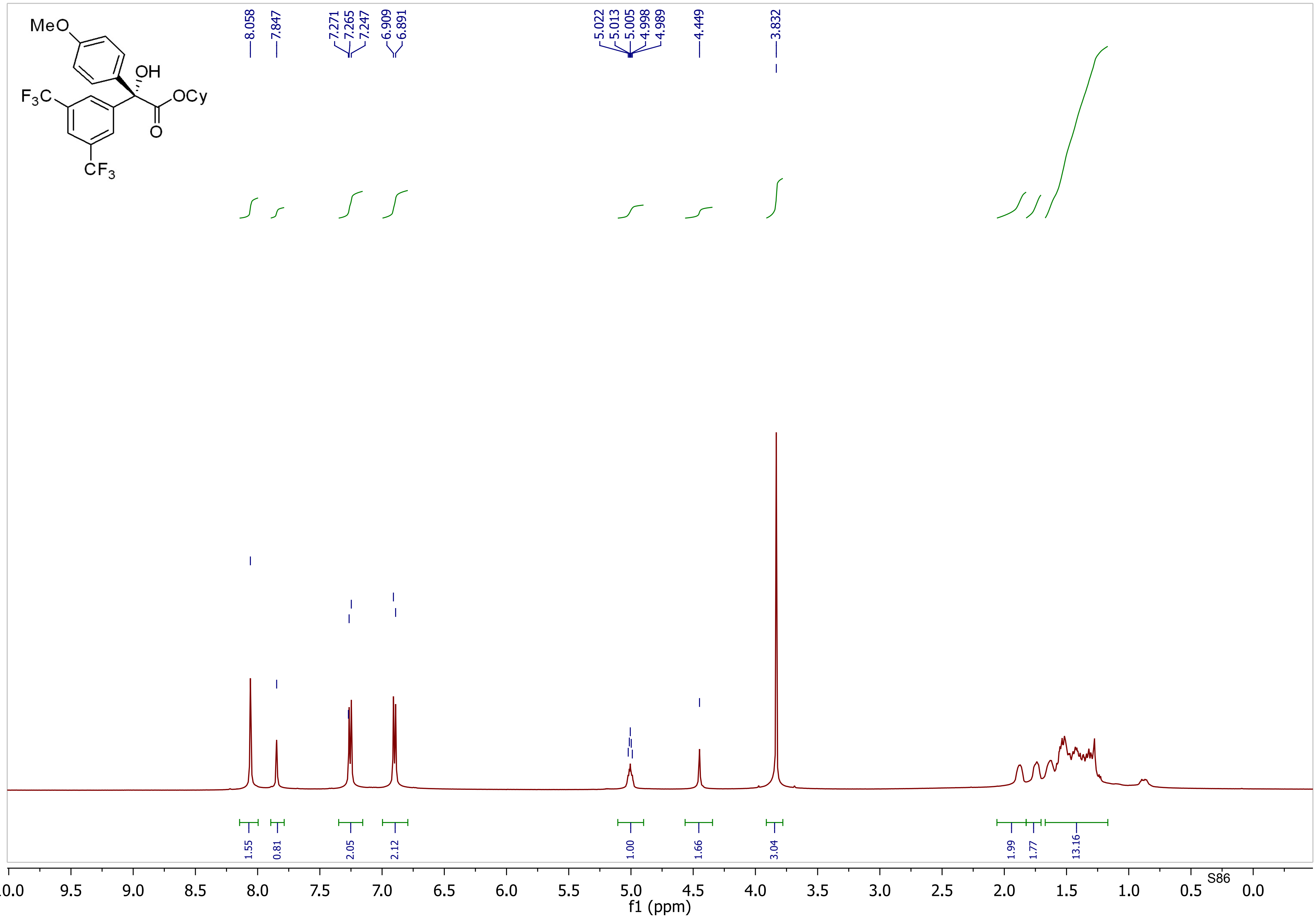

FC-II-193-b-C13
FC-II-193-b-C13
OH
OCy
O
174.15
142.40
141.44
140.93
140.85
129.02
128.28
128.20
128.15
127.68
127.64
127.36
126.94
80.92
77.53
77.28
77.02
75.97
31.34
25.42
23.43
f1 (ppm)

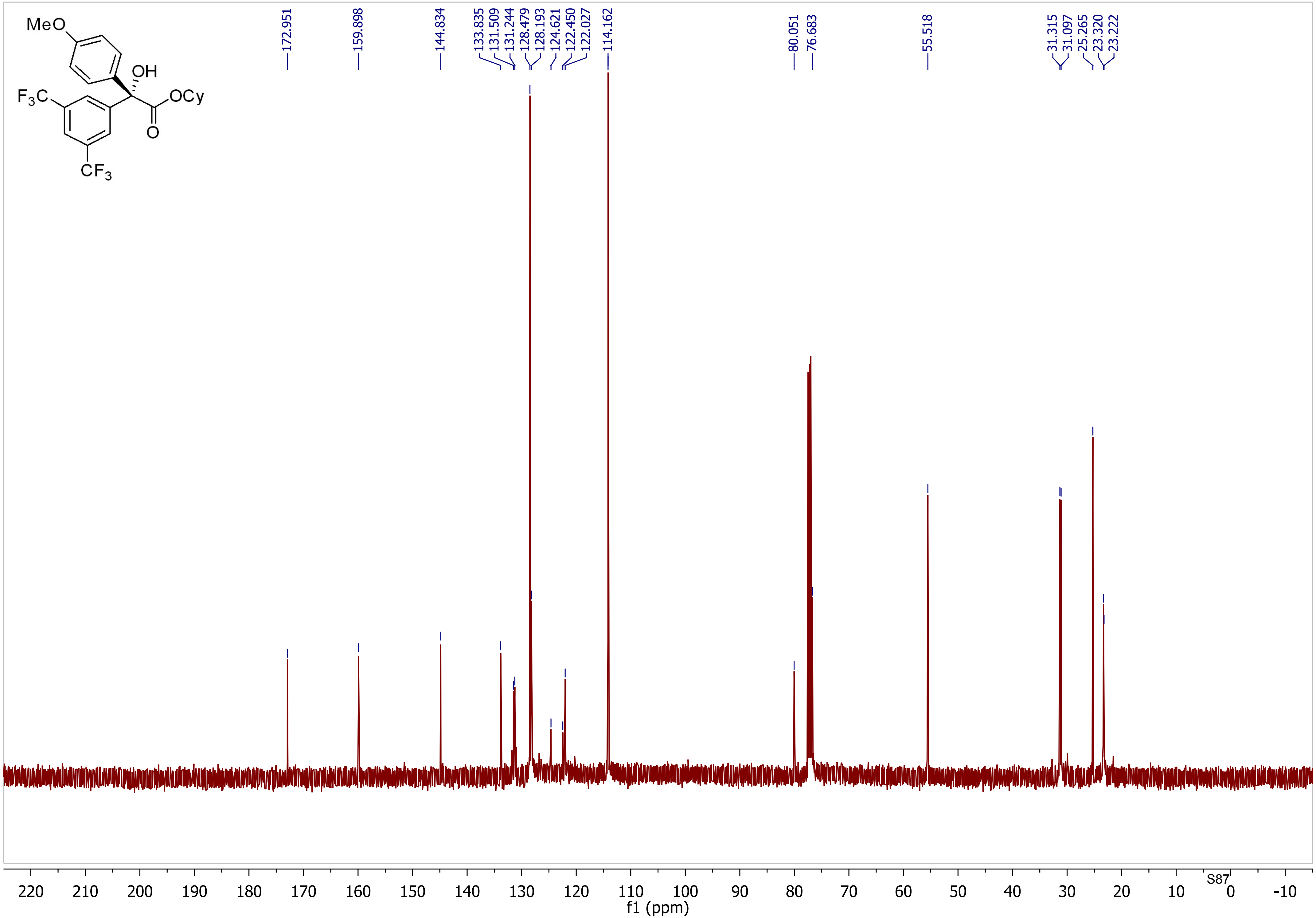

FC-II-193-a-H1
FC-II-193-b-H1
OH
OCy
O
7.99 7.85 7.85 7.84 7.82 7.80 7.54 7.54 7.52 7.51 7.50 7.49 7.49 7.48 7.48 7.38 7.38 7.37 7.37 7.35 7.34
5.04 5.03 5.02 5.02 5.01 5.00
4.46
1.83 1.83 1.61 1.60 1.60 1.59 1.57 1.53 1.51 1.50 1.49 1.48 1.48 1.38 1.38 1.37 1.36 1.36 1.35 1.34 1.34 1.29 1.27 1.26
0.97 2.62 3.88 2.46 1.00 0.82 2.32 5.45 3.67
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 -0.5
f1 (ppm)
13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000

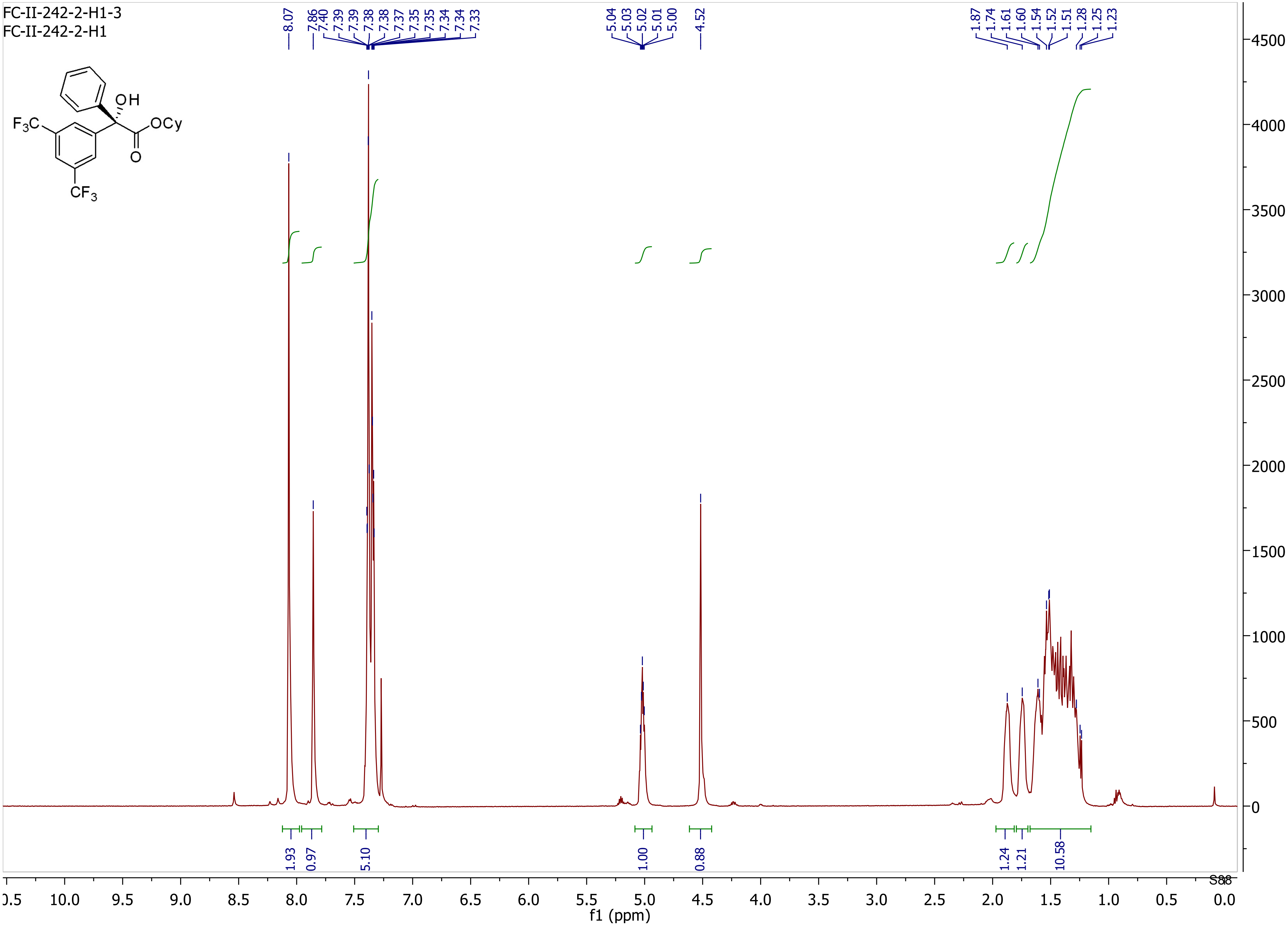
FC-II-193-a-C13-2
FC-II-193-a-C13



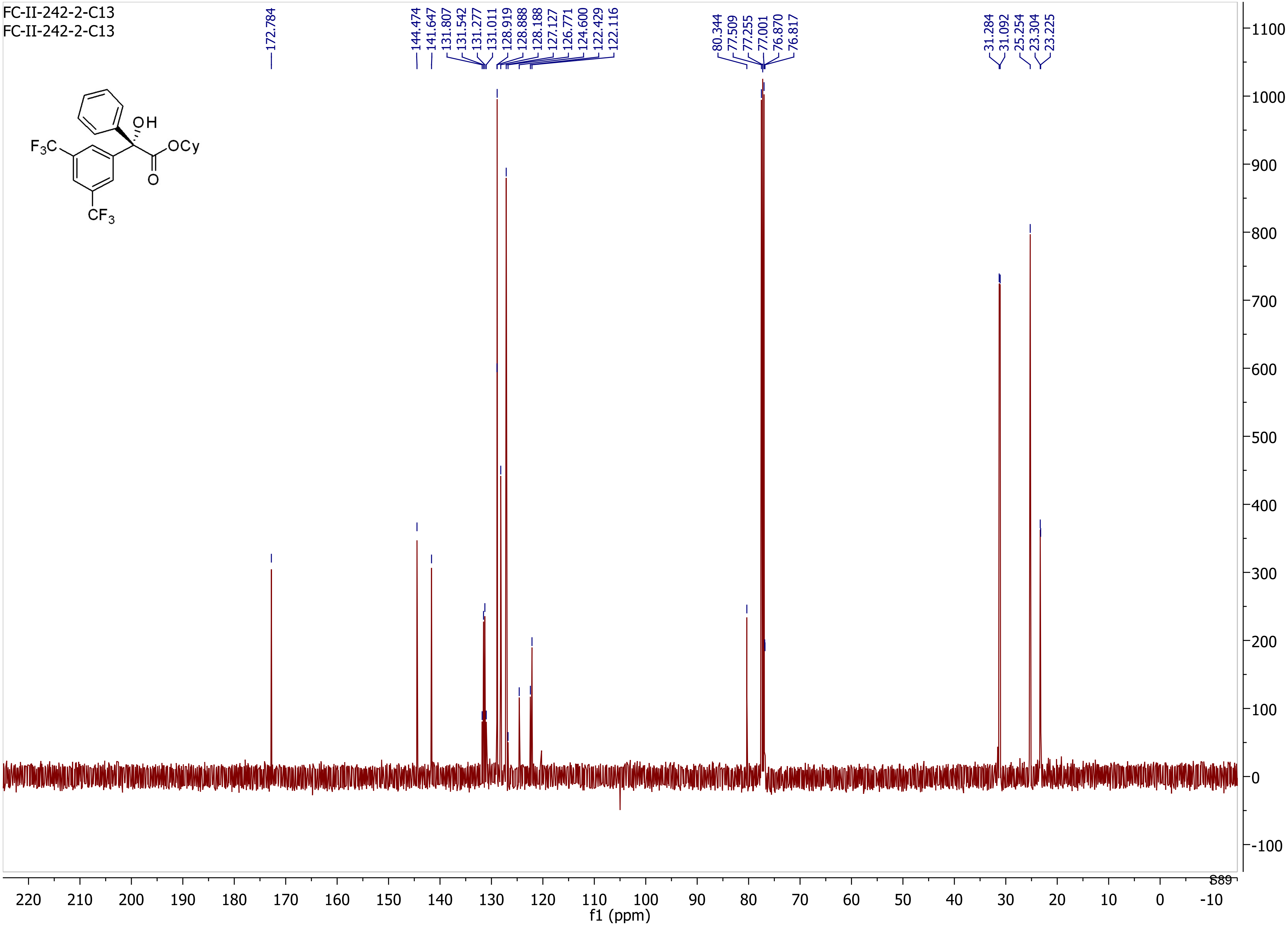


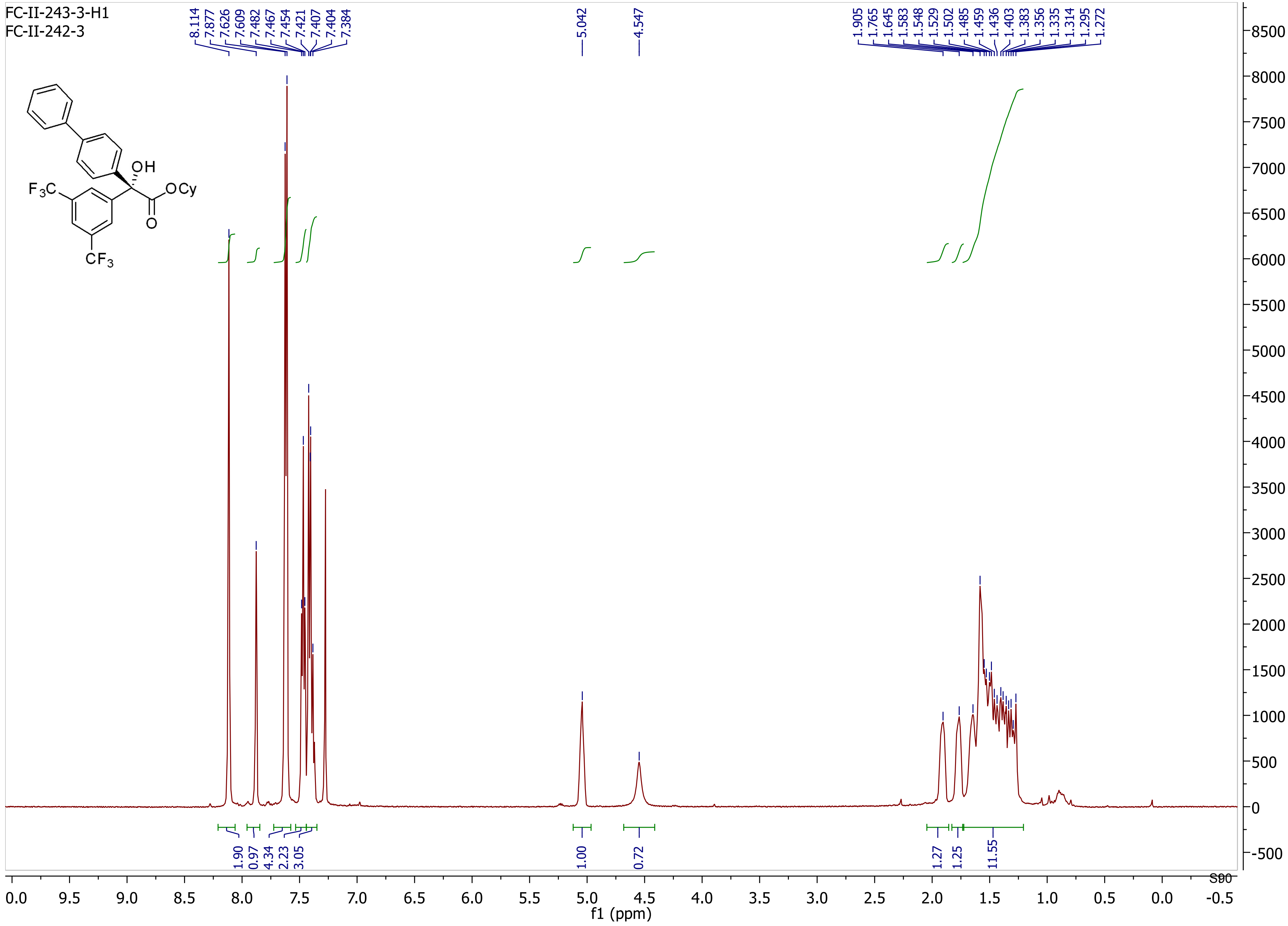

MeO
OH
F3C
OCy
O
CF3
8.058
7.847
7.271
7.265
7.247
6.909
6.891
5.022
5.013
5.005
4.998
4.989
4.449
3.832
1.55
0.81
2.05
2.12
1.00
1.66
3.04
1.99
1.77
13.16
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

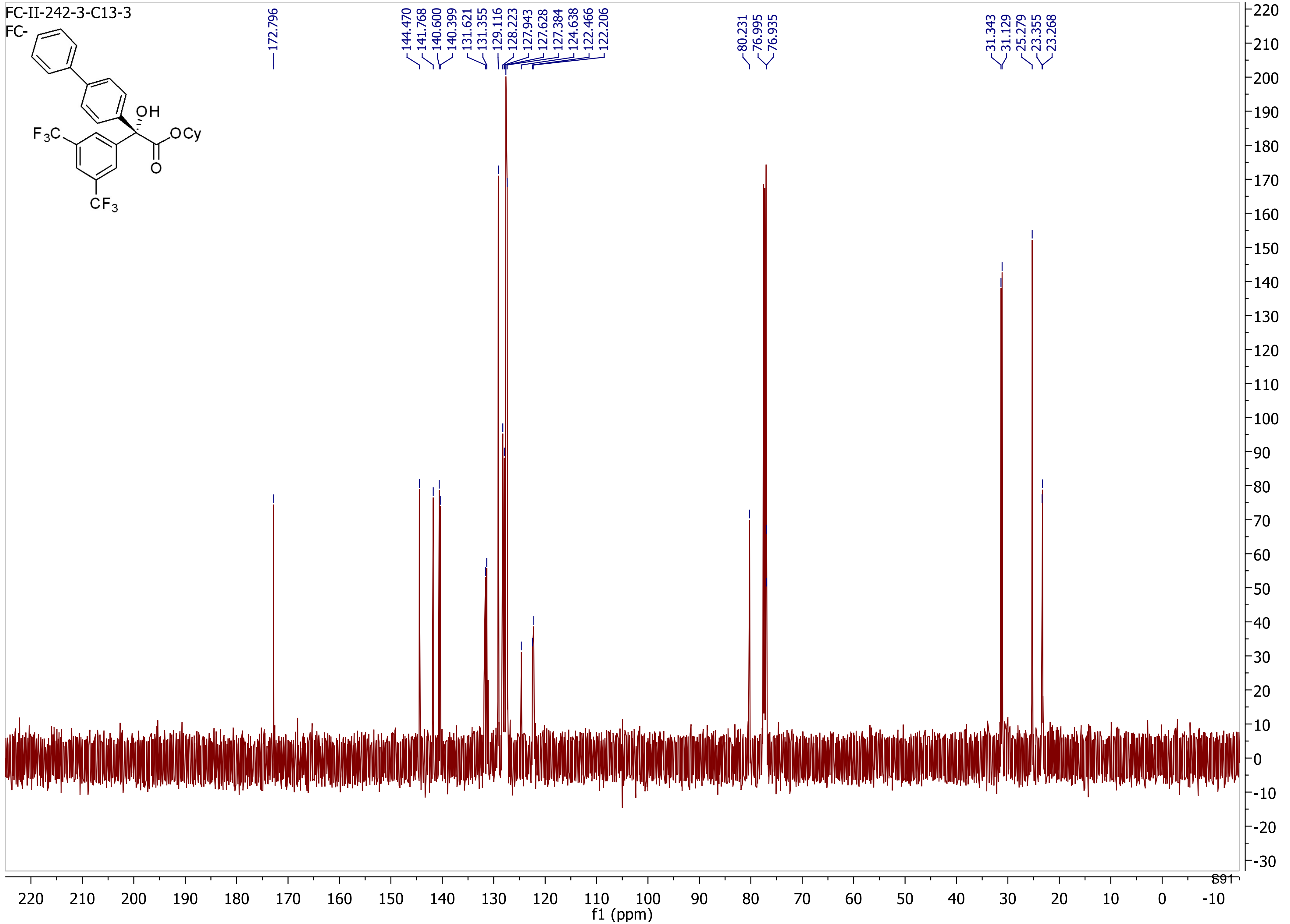

MeO
OH
F3C
OCy
O
CF3
172.951
159.898
144.834
133.835
131.509
131.244
128.479
128.193
124.621
122.450
122.027
114.162
80.051
76.683
55.518
31.315
31.097
25.265
23.320
23.222
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


FC-II-242-2-H1-3
FC-II-242-2-H1
OH
OCy
F_3C
CF_3
O
8.07
7.86
7.40
7.39
7.39
7.38
7.38
7.37
7.35
7.35
7.34
7.34
7.33
5.04
5.03
5.02
5.01
5.00
4.52
1.87
1.74
1.61
1.60
1.54
1.52
1.51
1.28
1.25
1.23
1.93
0.97
5.10
1.00
0.88
1.24
1.21
10.58
f1 (ppm)

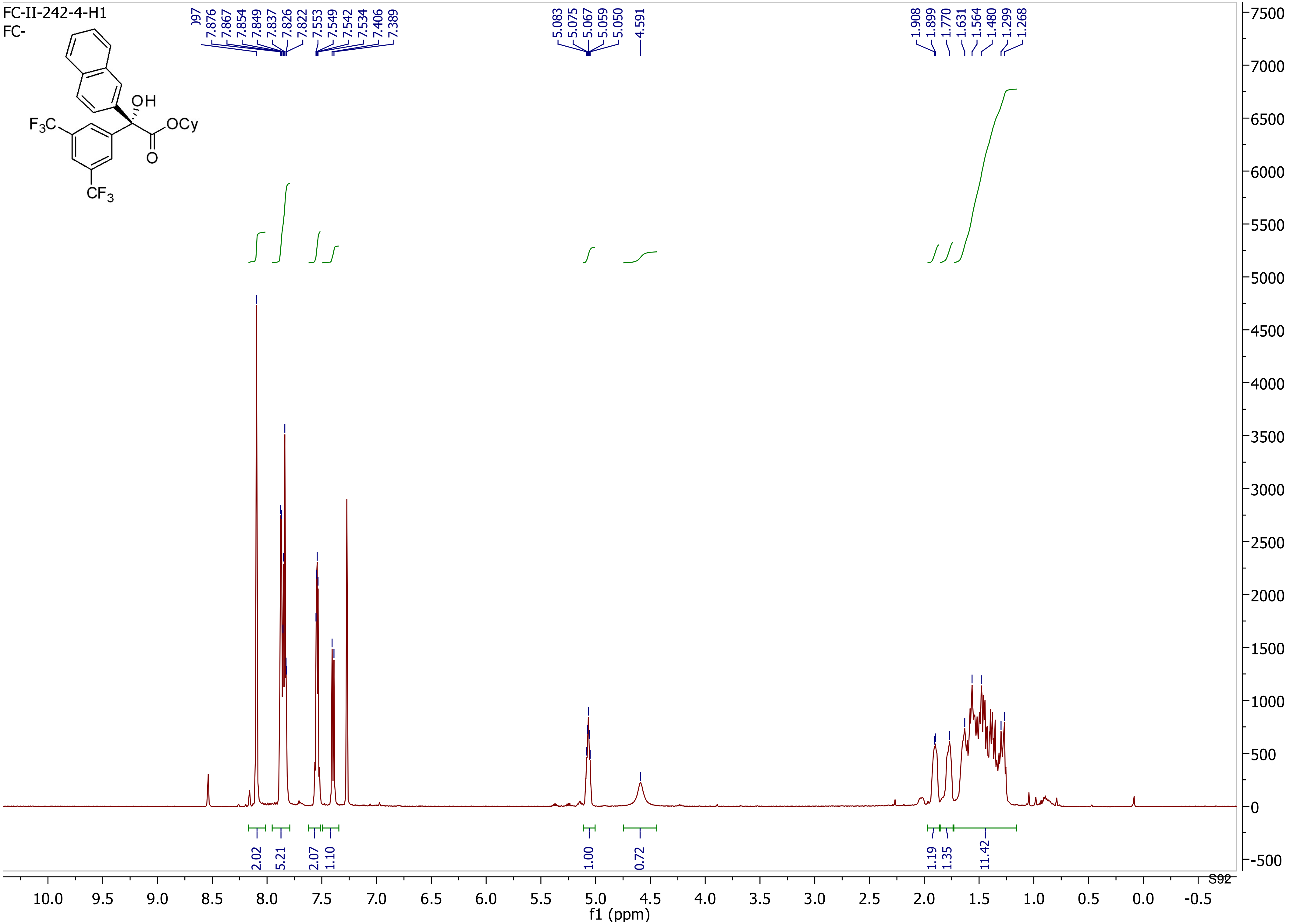
FC-II-242-2-C13
FC-II-242-2-C13

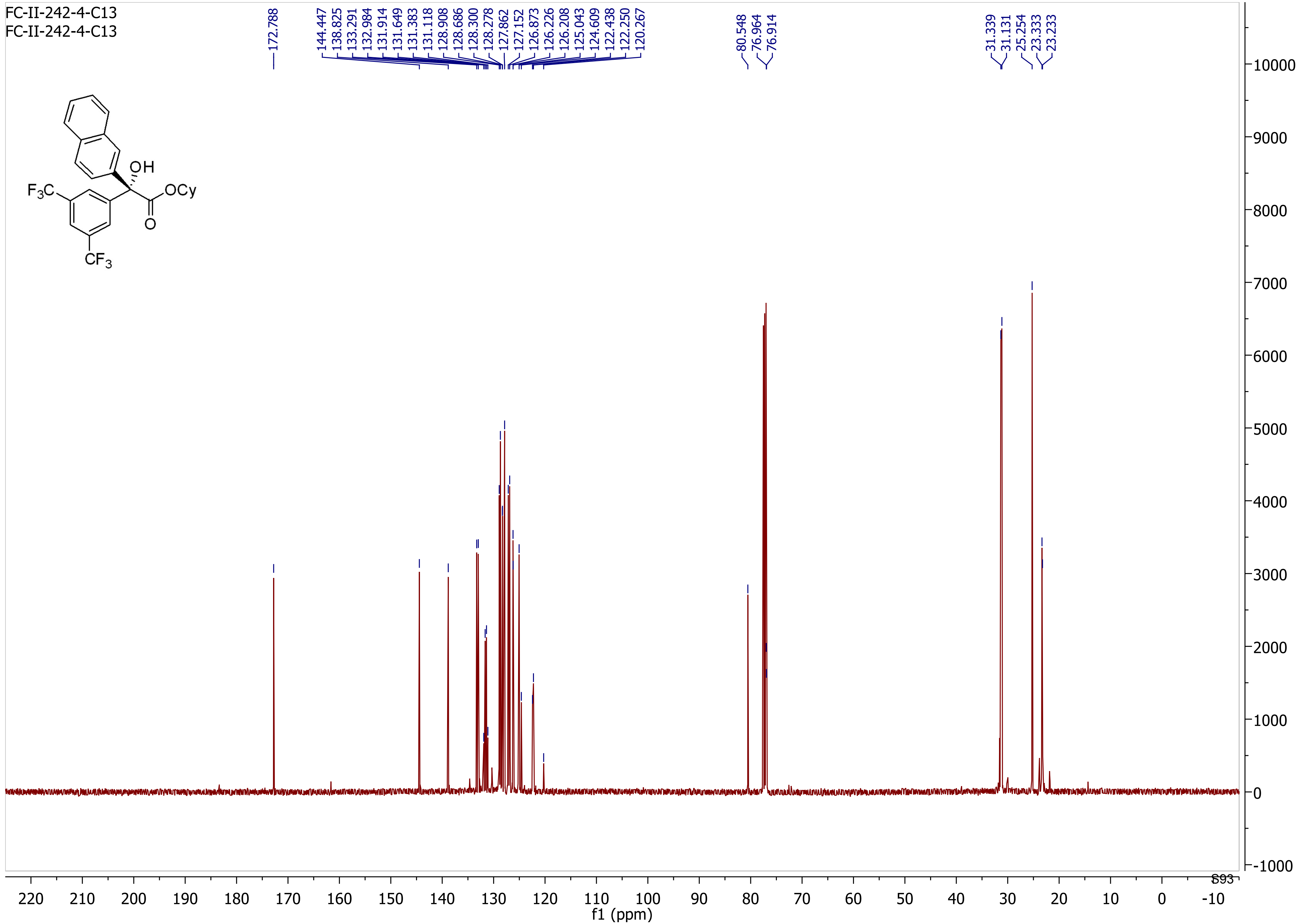


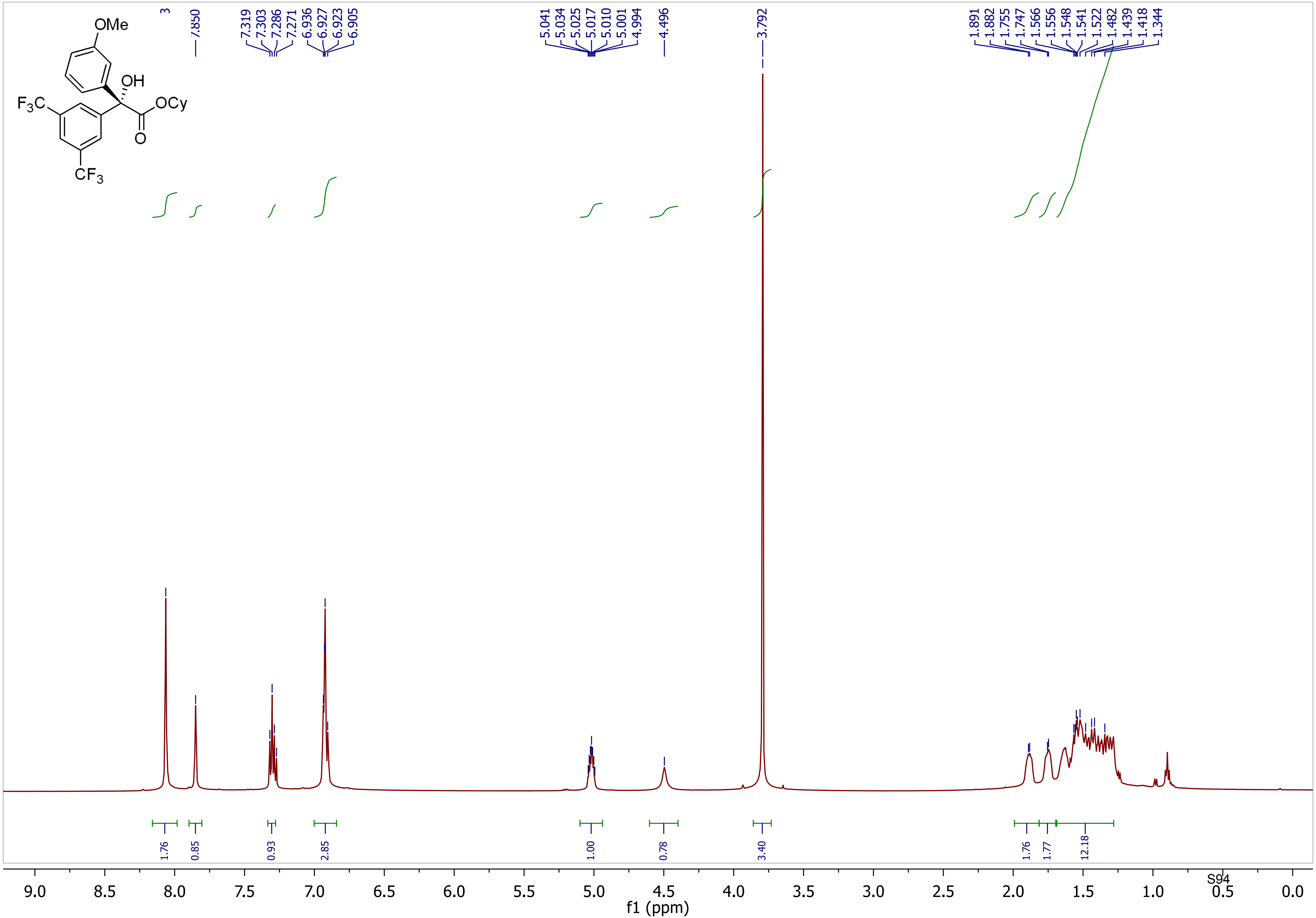

FC-II-243-3-H1
FC-II-242-3
OH
OCy
F3C
CF3
O
8.114
7.877
7.626
7.609
7.482
7.467
7.454
7.421
7.407
7.404
7.384
5.042
4.547
1.905
1.765
1.645
1.583
1.548
1.529
1.502
1.485
1.459
1.436
1.403
1.383
1.356
1.335
1.314
1.295
1.272
1.90
0.97
4.34
2.23
3.05
1.00
0.72
1.27
1.25
11.55
f1 (ppm)

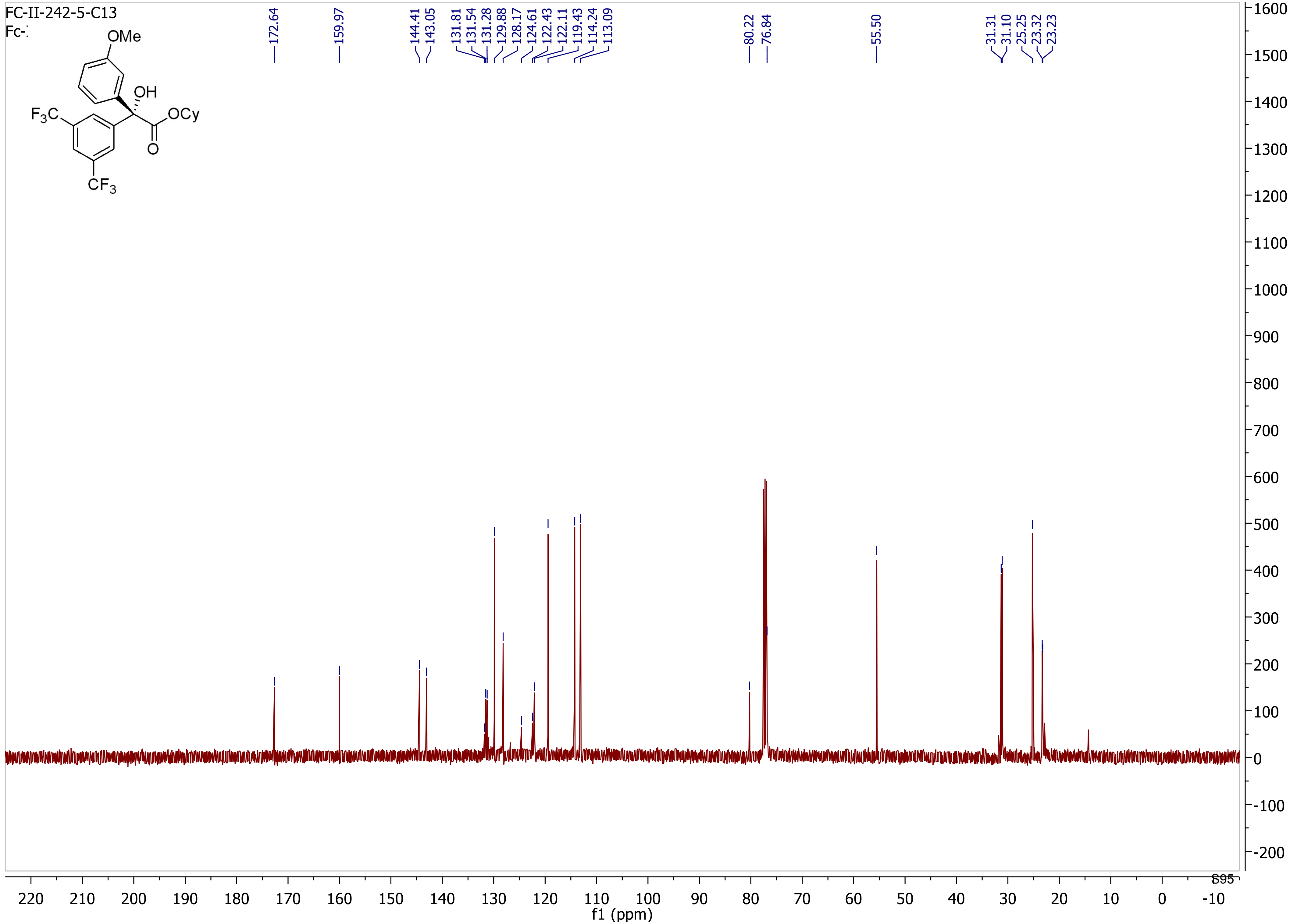


FC-II-242-3-C13-3
FC-
OH
OCy
F3C
CF3
O
172.796
144.470
141.768
140.600
140.399
131.621
131.355
129.116
128.223
127.943
127.628
127.384
124.638
122.466
122.206
80.231
76.995
76.935
31.343
31.129
25.279
23.355
23.268
f1 (ppm)


FC-II-242-4-H1
FC-
OH
OCy
F3C
CF3
O
)97
7.876
7.867
7.854
7.849
7.837
7.826
7.822
7.553
7.549
7.542
7.534
7.406
7.389
5.083
5.075
5.067
5.059
5.050
4.591
1.908
1.899
1.770
1.631
1.564
1.480
1.299
1.268
2.02
5.21
2.07
1.10
1.00
0.72
1.19
1.35
11.42
f1 (ppm)

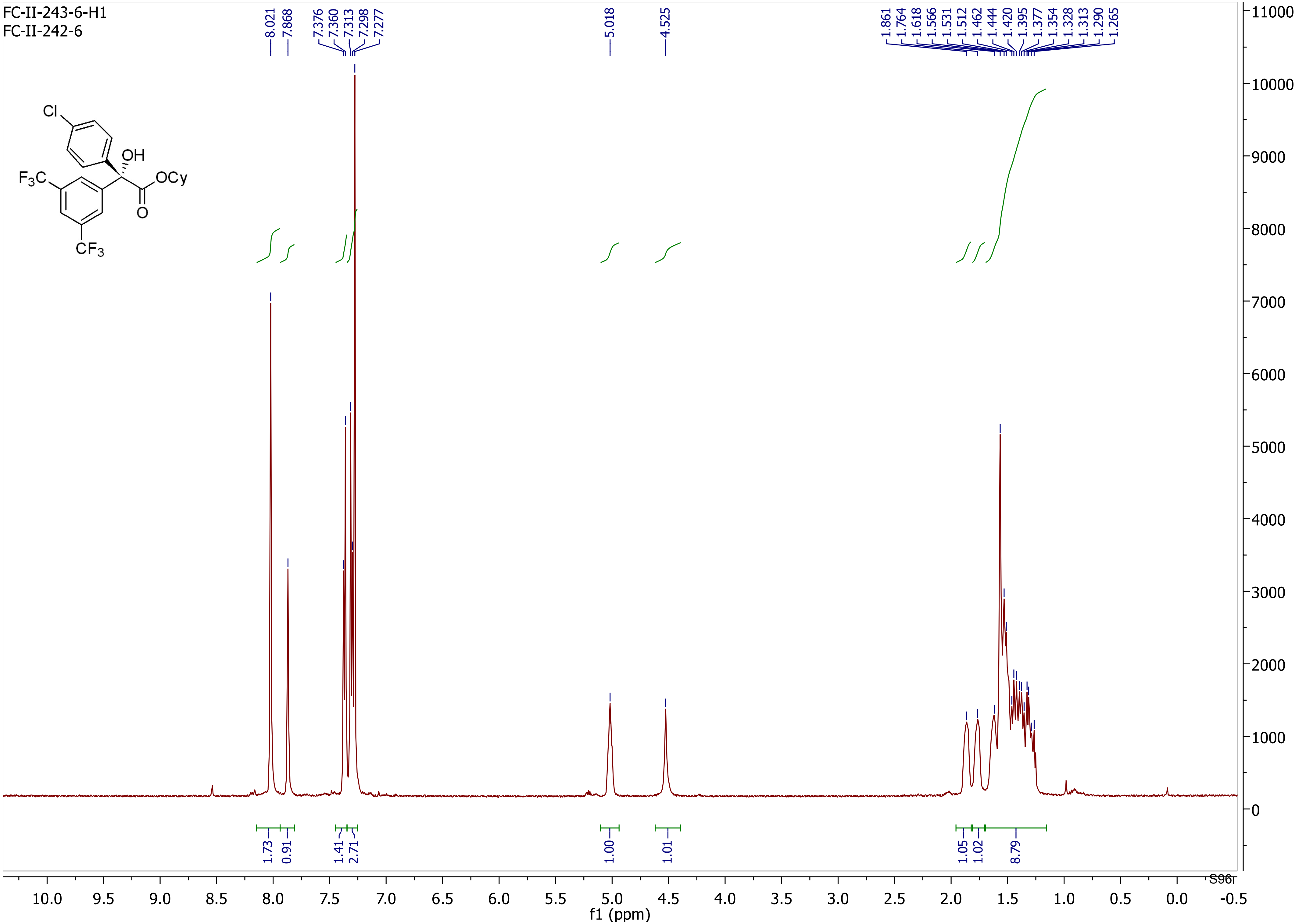

FC-II-242-4-C13
FC-II-242-4-C13
172.788
144.447
138.825
133.291
132.984
131.914
131.649
131.383
131.118
128.908
128.686
128.300
128.278
127.862
127.152
126.873
126.226
126.208
125.043
124.609
122.438
122.250
120.267
80.548
76.964
76.914
31.339
31.131
25.254
23.333
23.233
OH
OCy
F_3C
O
CF_3
f1 (ppm)

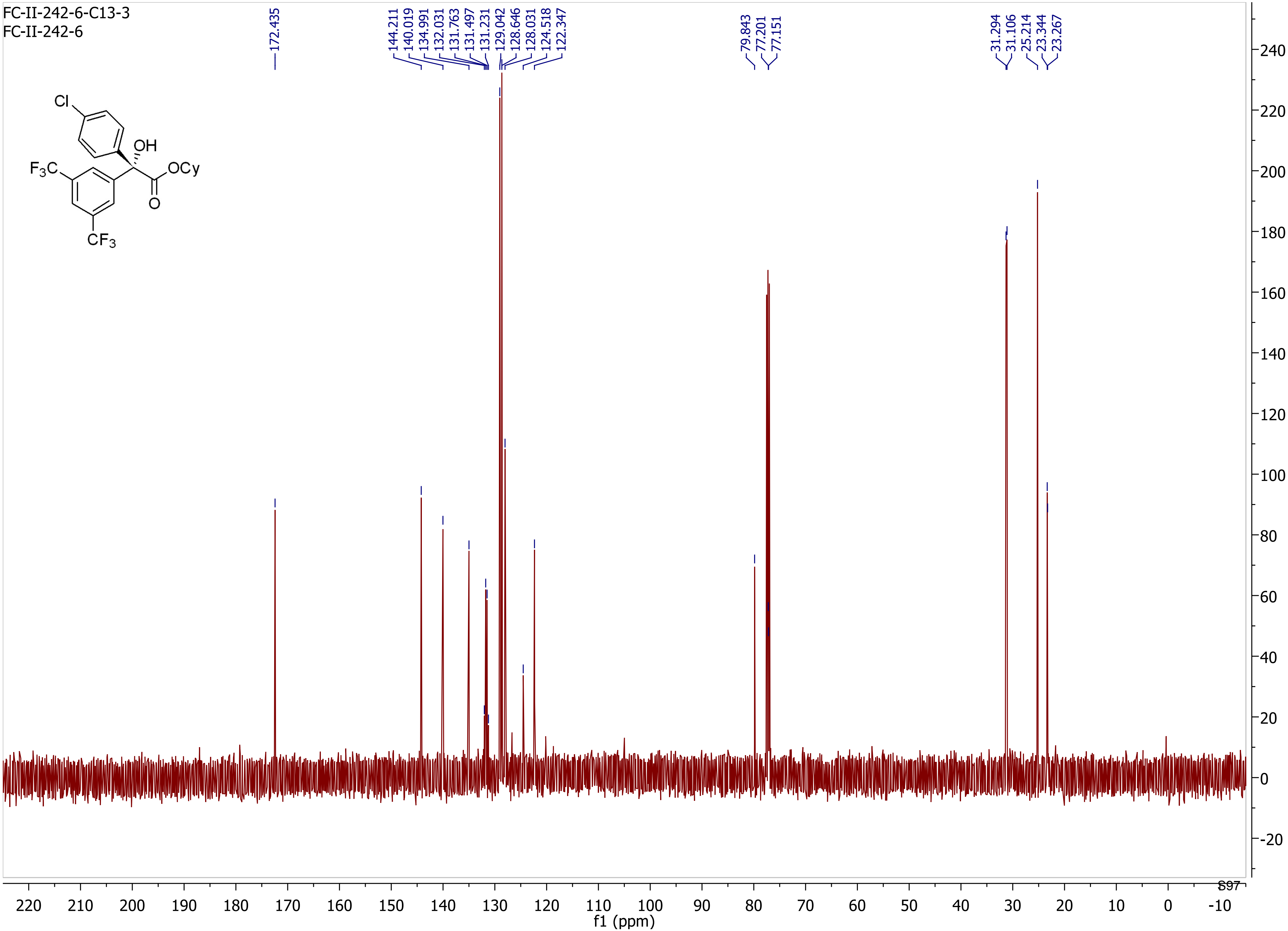

OMe
OH
OCy
F_3C
O
CF_3
3
7.850
7.319
7.303
7.286
7.271
6.936
6.927
6.923
6.905
5.041
5.034
5.025
5.017
5.010
5.001
4.994
4.496
3.792
1.891
1.882
1.755
1.747
1.566
1.556
1.548
1.541
1.522
1.482
1.439
1.418
1.344
1.76
0.85
0.93
2.85
1.00
0.78
3.40
1.76
1.77
12.18
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



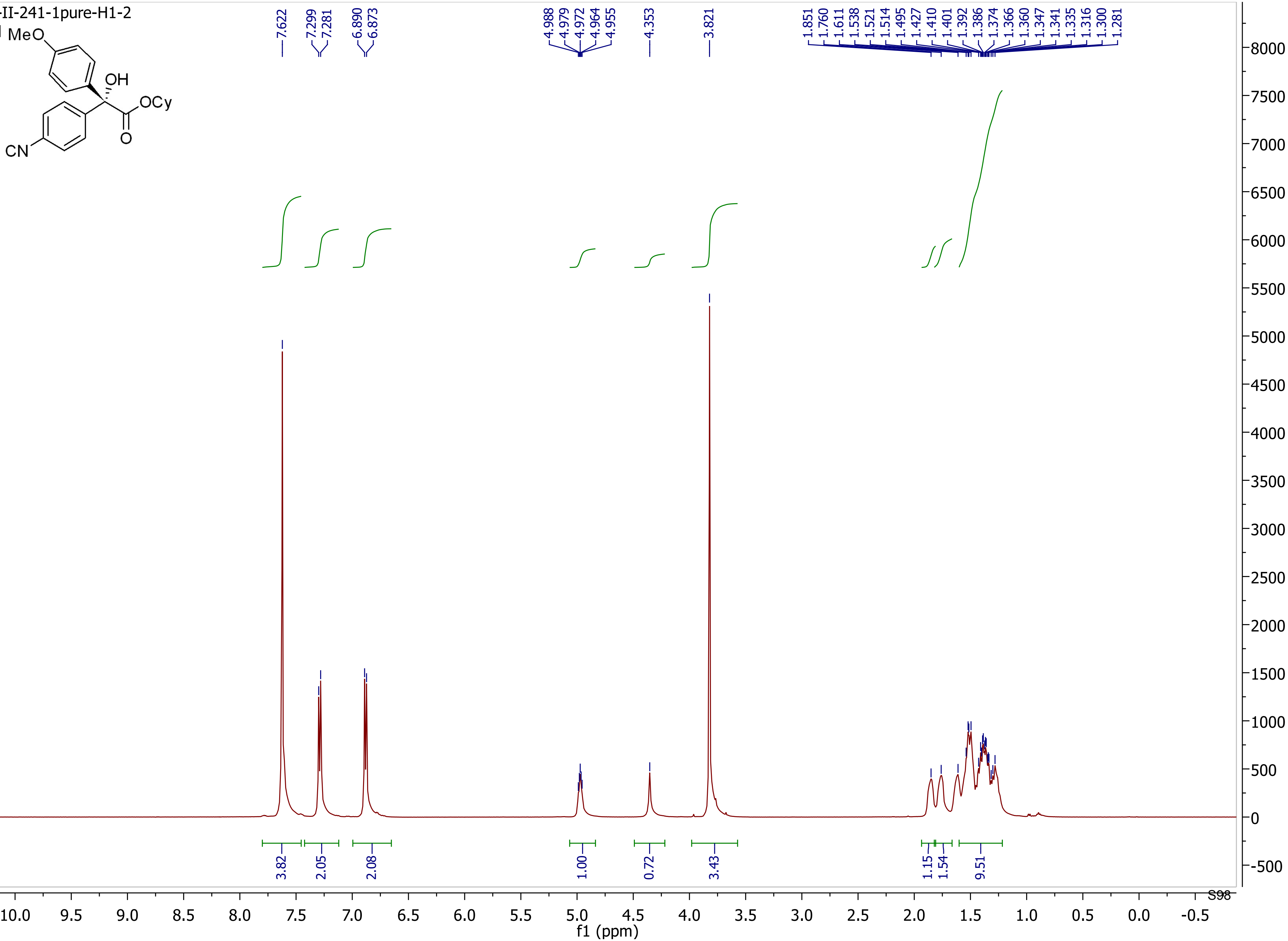
FC-II-242-5-C13
Fc-:
OMe
OH
F3C
OCy
O
CF3
172.64
159.97
144.41
143.05
131.81
131.54
131.28
129.88
128.17
124.61
122.43
122.11
119.43
114.24
113.09
80.22
76.84
55.50
31.31
31.10
25.25
23.32
23.23
f1 (ppm)

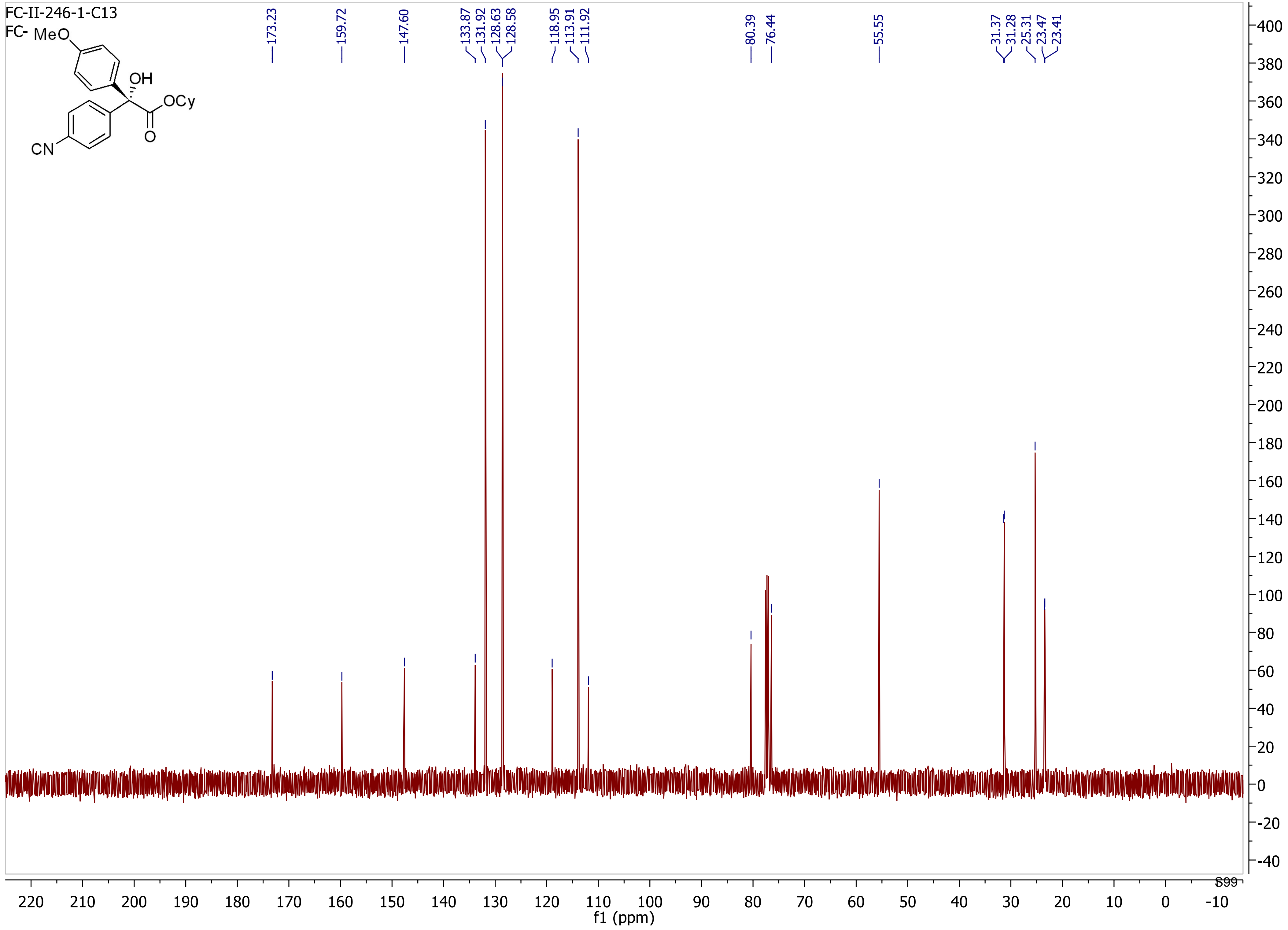
FC-II-243-6-H1
FC-II-242-6

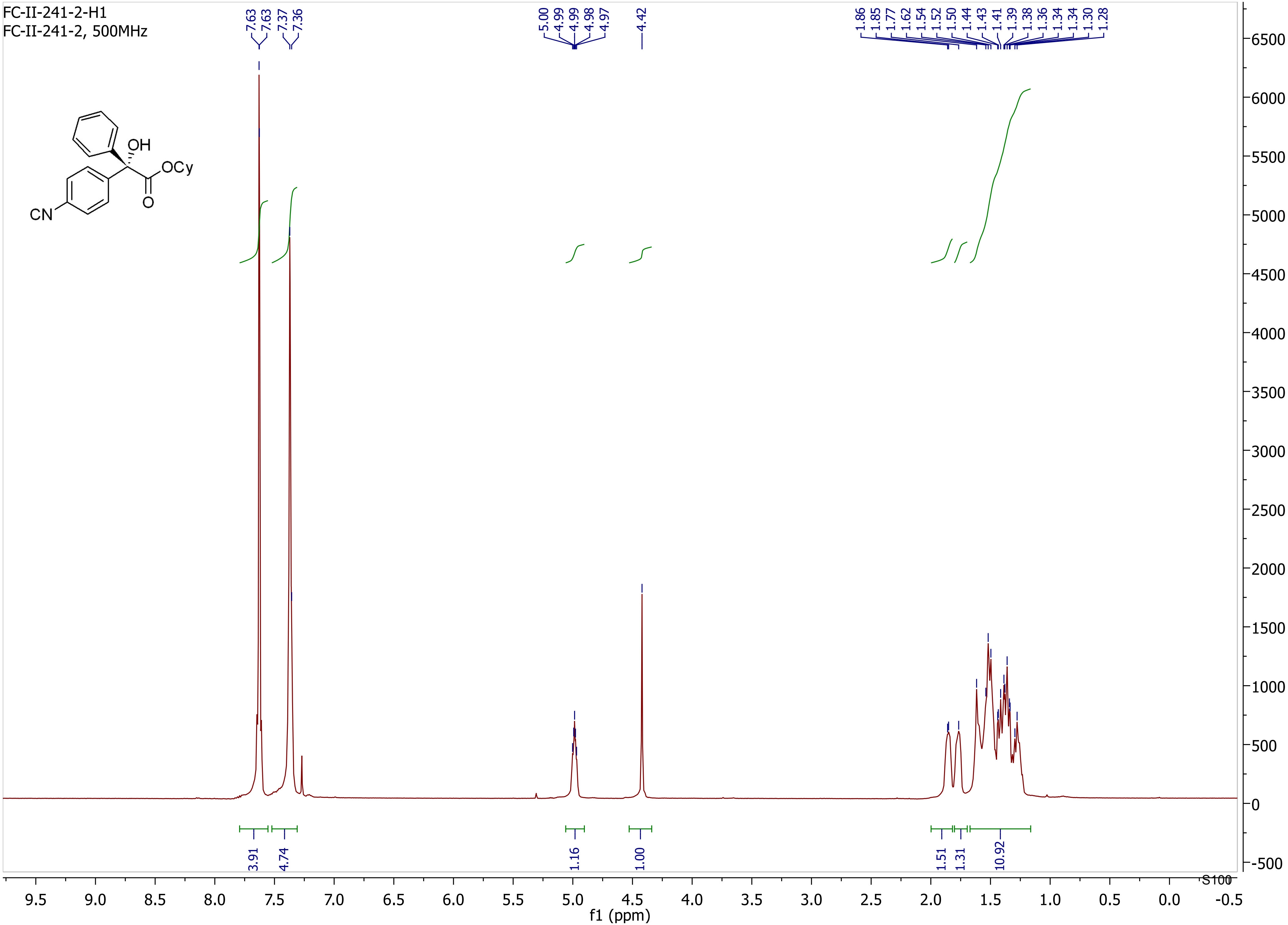


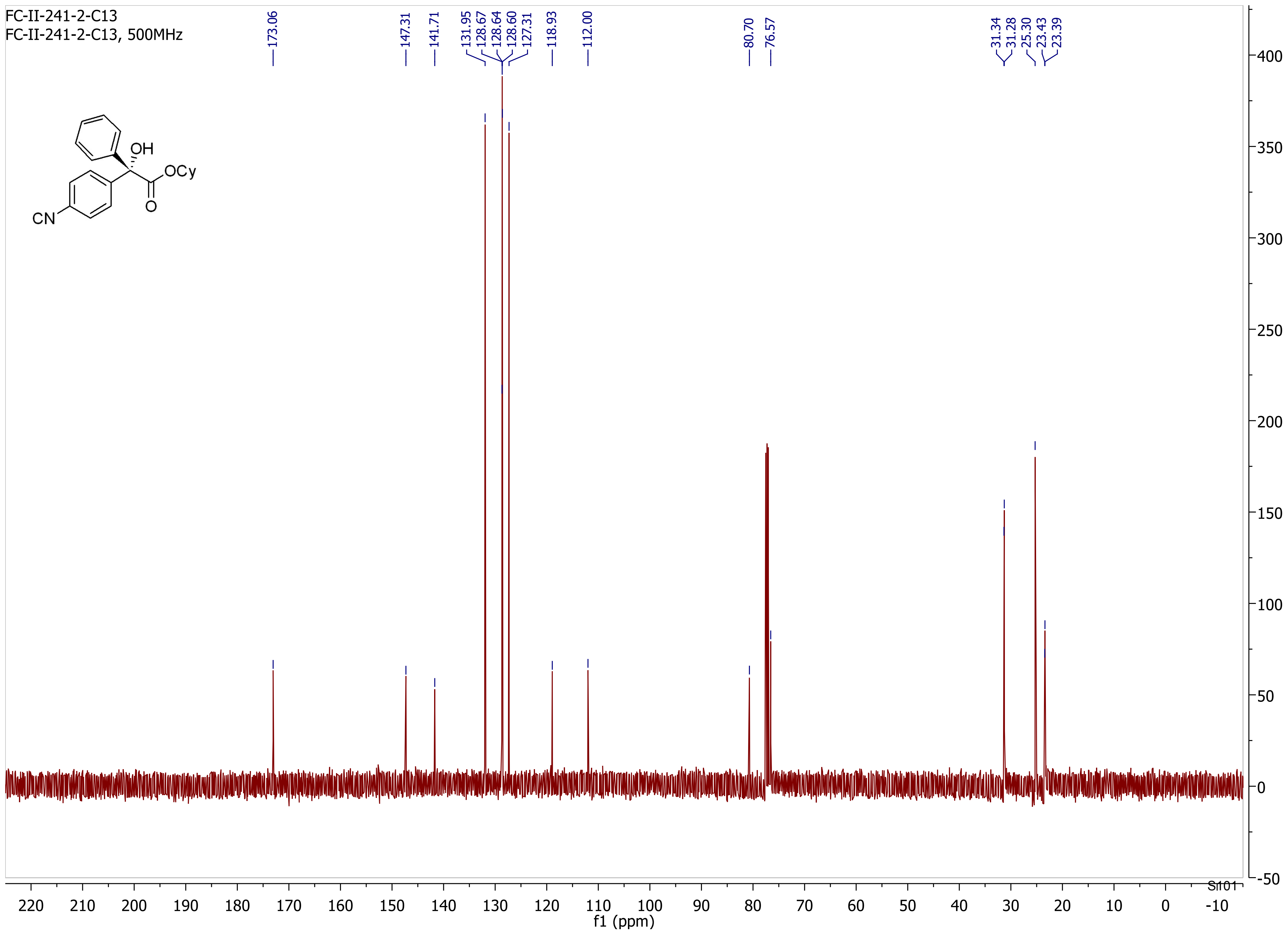

FC-II-242-6-C13-3
FC-II-242-6
172.435
144.211
140.019
134.991
132.031
131.763
131.497
131.231
129.042
128.646
128.031
124.518
122.347
79.843
77.201
77.151
31.294
31.106
25.214
23.344
23.267
Cl
OH
F_3C
OCy
O
CF_3
f1 (ppm)

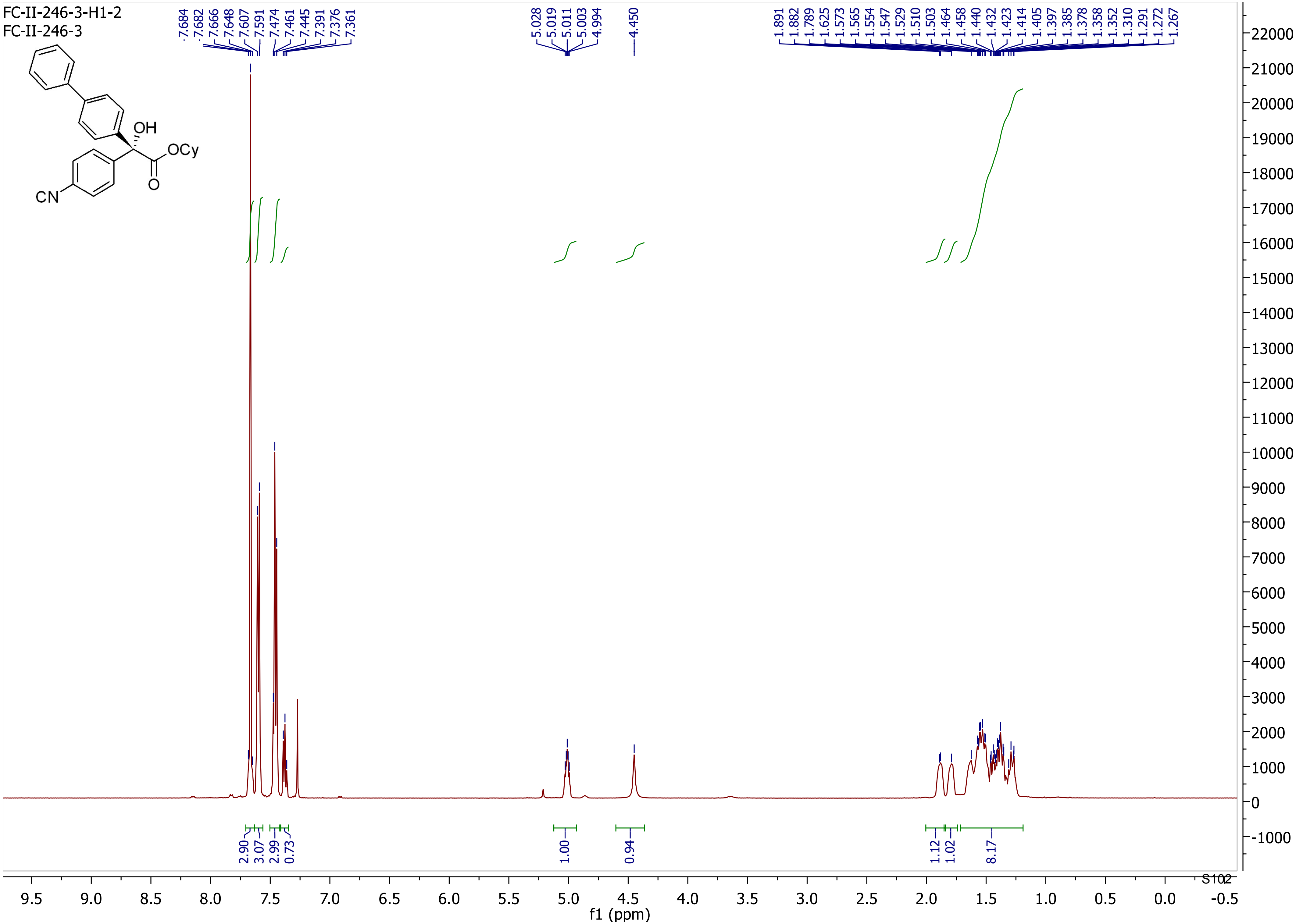
FC-II-241-1pure-H1-2
Std



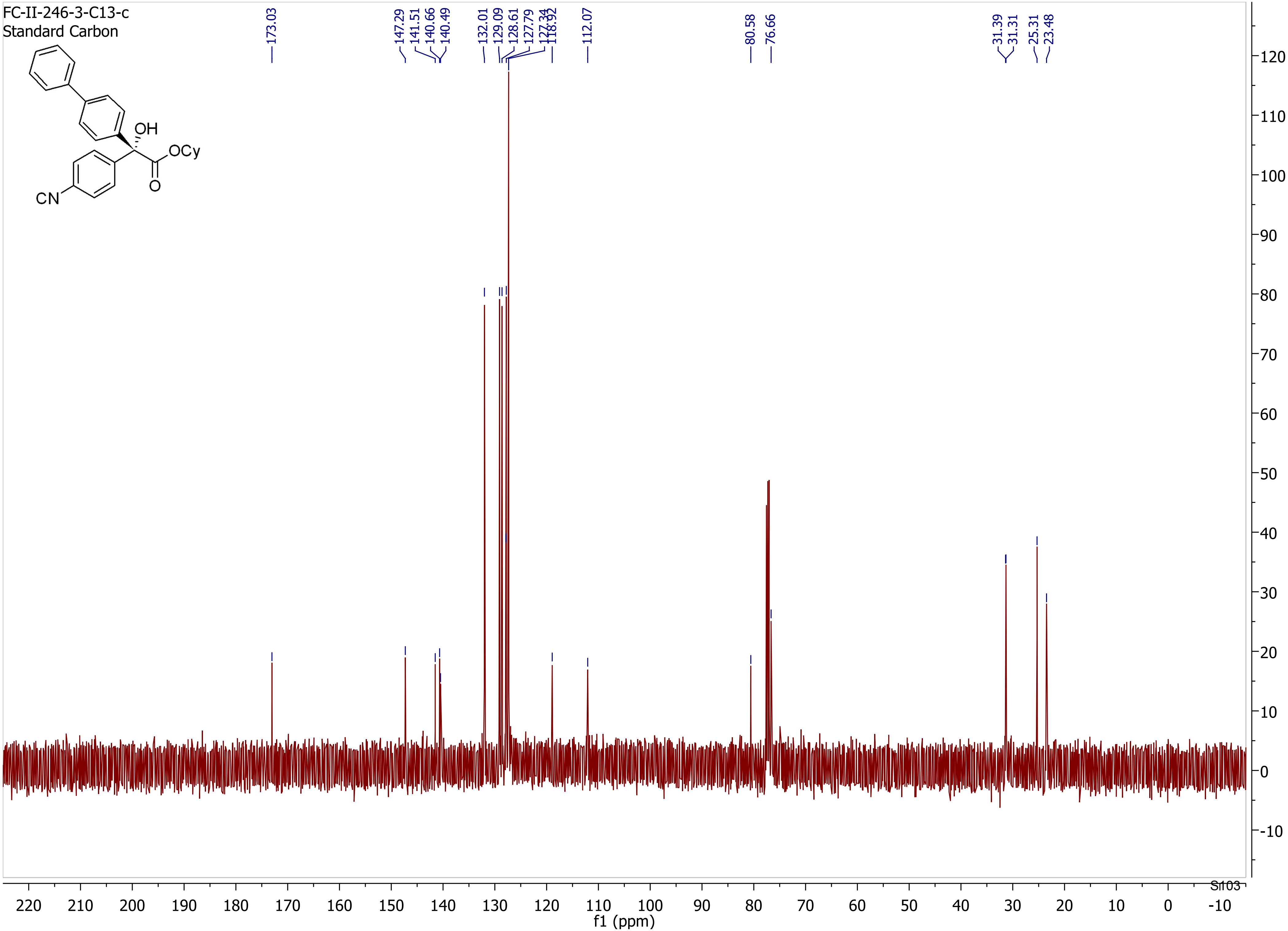

FC-II-246-1-C13
FC-
MeO
OH
OCy
O
CN
173.23
159.72
147.60
133.87
131.92
128.63
128.58
118.95
113.91
111.92
80.39
76.44
55.55
31.37
31.28
25.31
23.47
23.41
f1 (ppm)

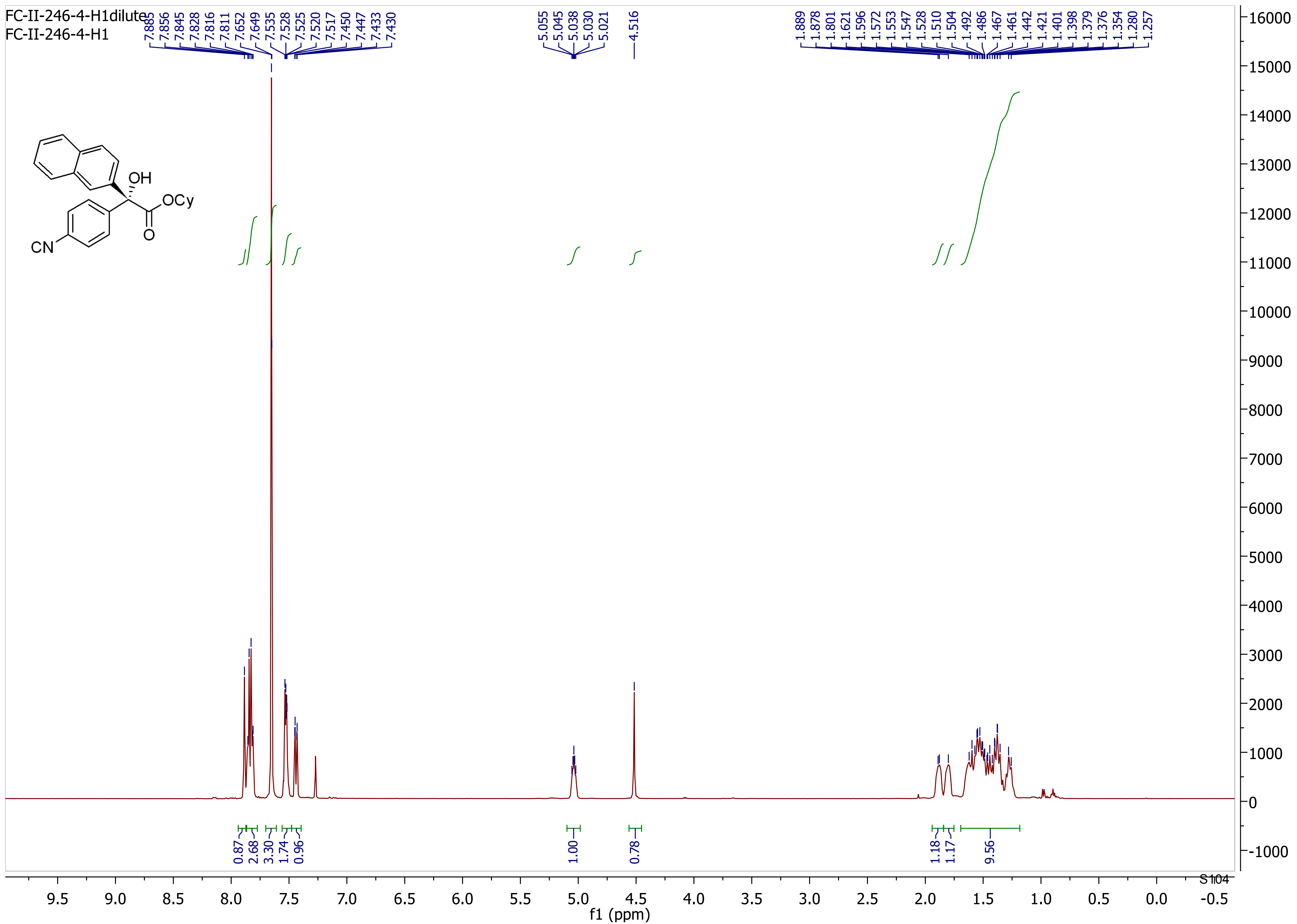

FC-II-241-2-H1
FC-II-241-2, 500MHz
7.63 7.63 7.37 7.36
5.00 4.99 4.99 4.98 4.97
4.42
1.86 1.85 1.77 1.62 1.54 1.52 1.50 1.44 1.43 1.41 1.39 1.38 1.36 1.34 1.34 1.30 1.28
OH
OCy
O
CN
3.91 4.74 1.16 1.00 1.51 1.31 10.92
f1 (ppm)

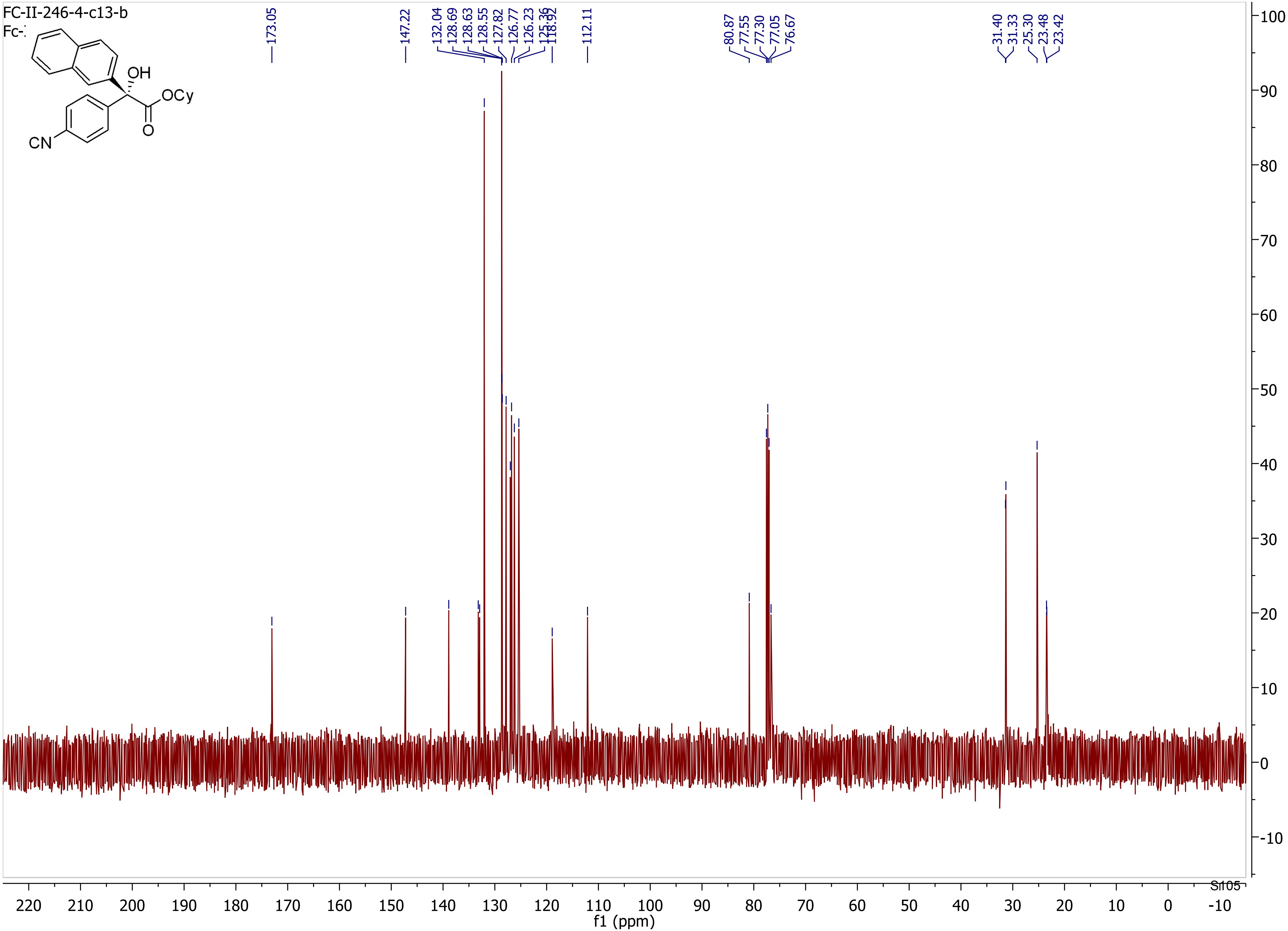

FC-II-241-2-C13
FC-II-241-2-C13, 500MHz
173.06
147.31
141.71
131.95
128.67
128.64
128.60
127.31
118.93
112.00
80.70
76.57
31.34
31.28
25.30
23.43
23.39
OH
OCy
O
CN
f1 (ppm)

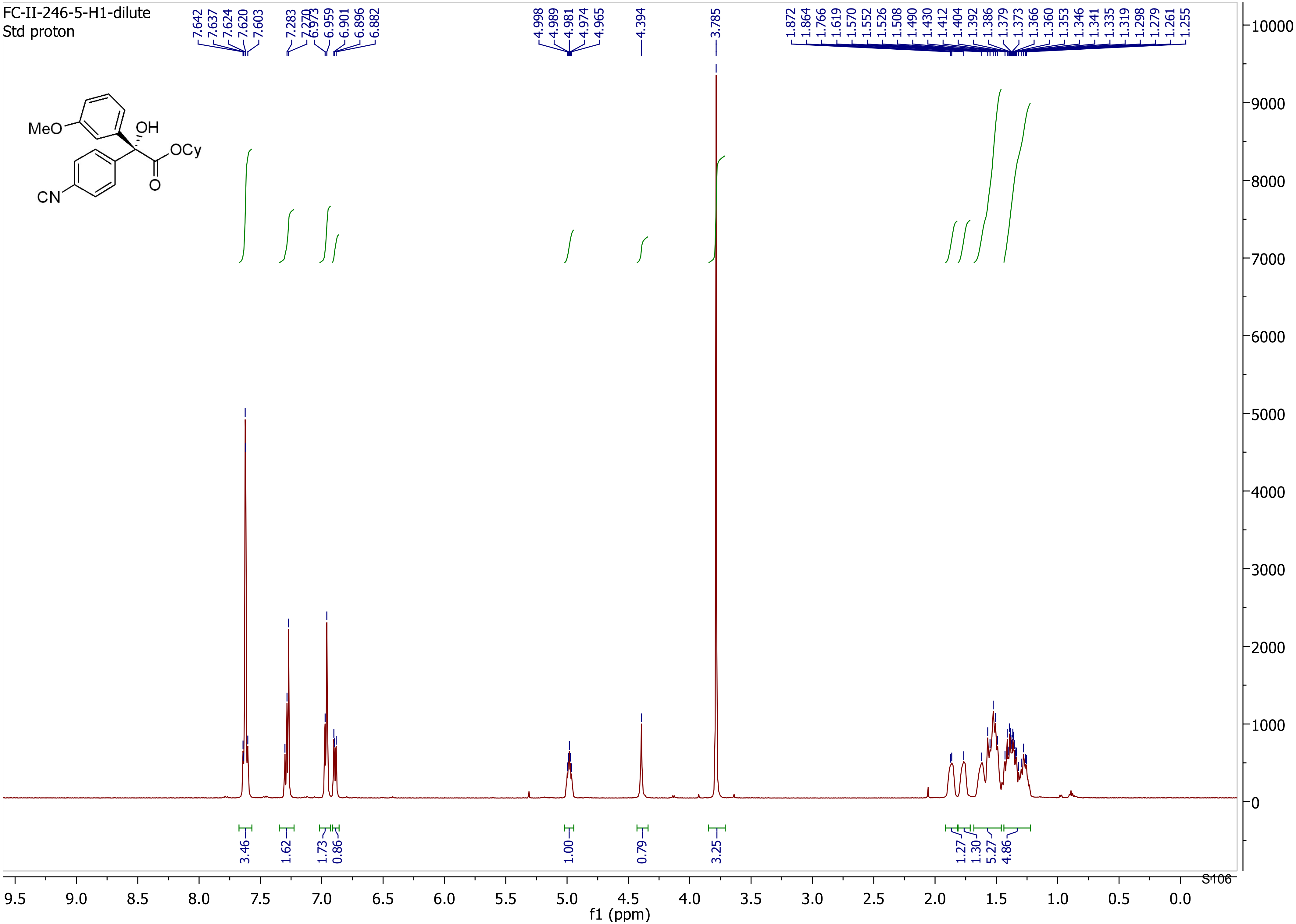

FC-II-246-3-H1-2
FC-II-246-3
OH
OCy
O
CN
7.684
7.682
7.666
7.648
7.607
7.591
7.474
7.461
7.445
7.391
7.376
7.361
5.028
5.019
5.011
5.003
4.994
4.450
1.891
1.882
1.789
1.625
1.573
1.565
1.554
1.547
1.529
1.510
1.503
1.464
1.458
1.440
1.432
1.423
1.414
1.405
1.397
1.385
1.378
1.358
1.352
1.310
1.291
1.272
1.267
2.90
3.07
2.99
0.73
1.00
0.94
1.12
1.02
8.17
f1 (ppm)

FC-II-246-3-C13-c
Standard Carbon

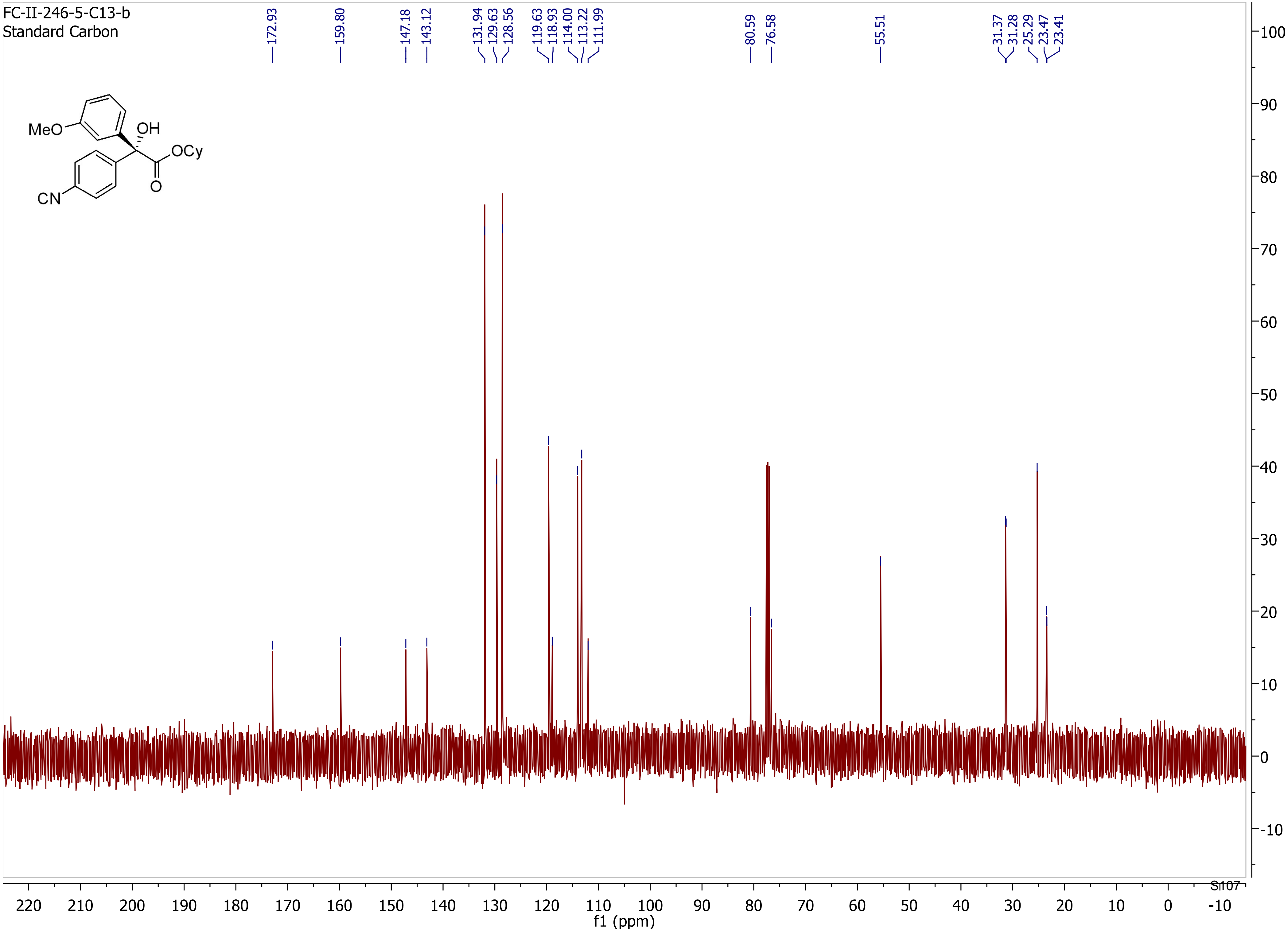


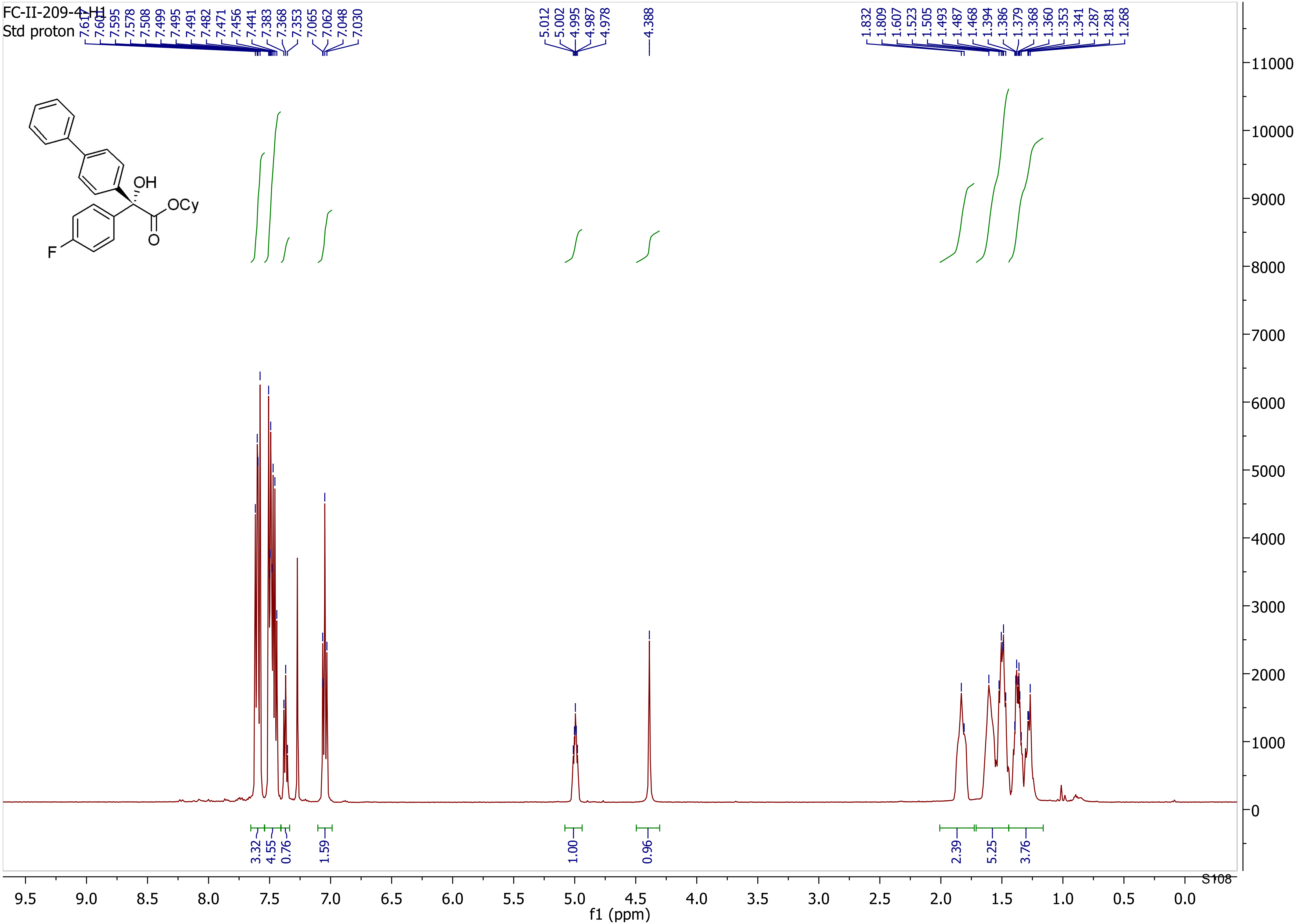

FC-II-246-4-H1dilute
FC-II-246-4-H1
7.885
7.856
7.845
7.828
7.816
7.811
7.652
7.649
7.535
7.528
7.525
7.520
7.517
7.450
7.447
7.433
7.430
5.055
5.045
5.038
5.030
5.021
4.516
1.889
1.878
1.801
1.621
1.596
1.572
1.553
1.547
1.528
1.510
1.504
1.492
1.486
1.467
1.461
1.442
1.421
1.401
1.398
1.379
1.376
1.354
1.280
1.257
OH
OCy
O
CN
0.87
2.68
3.30
1.74
0.96
1.00
0.78
1.18
1.17
9.56
f1 (ppm)

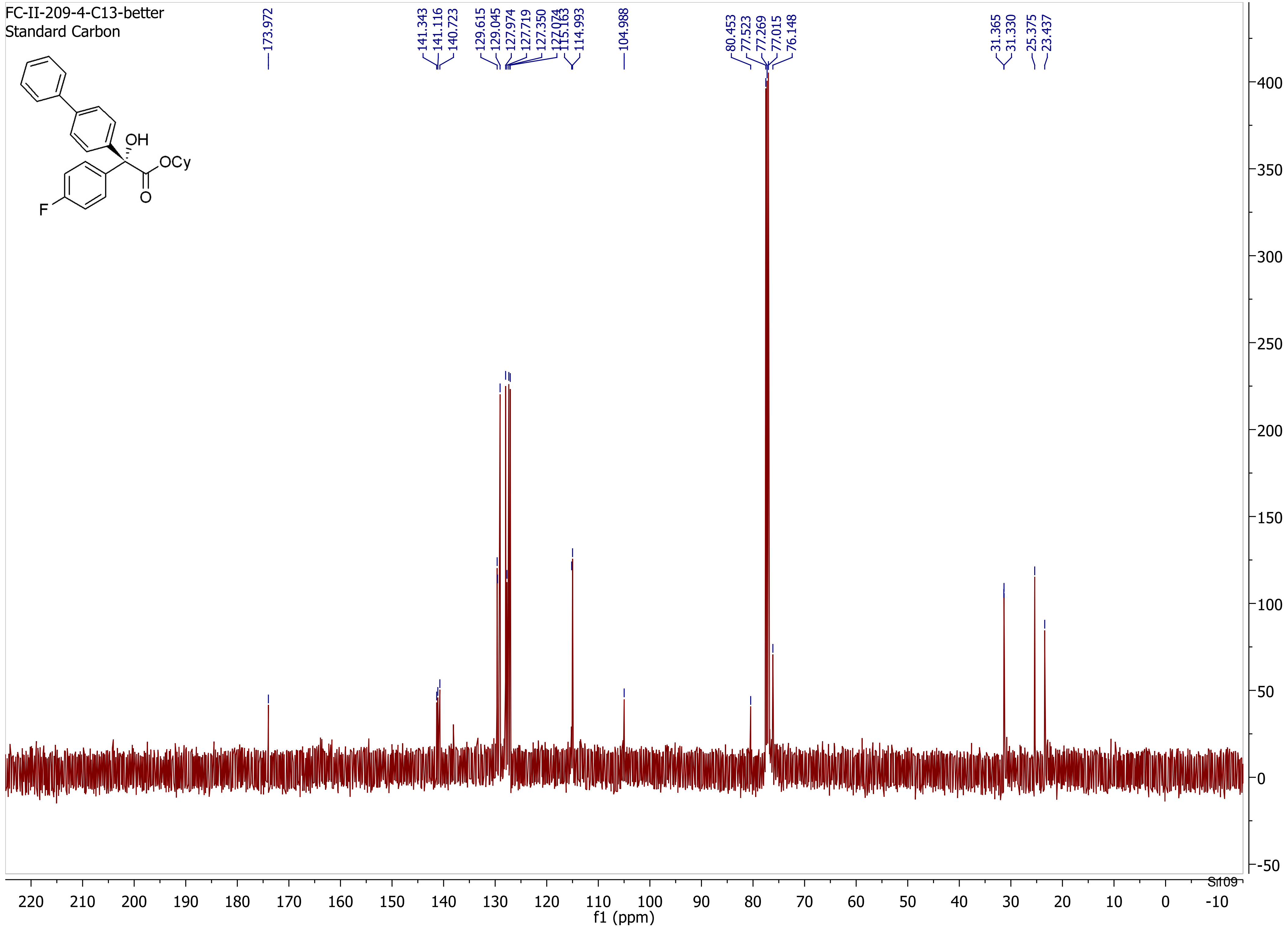

FC-II-246-4-c13-b
OH
OCy
O
CN
173.05
147.22
132.04
128.69
128.63
128.55
127.82
126.77
126.23
125.36
118.92
112.11
80.87
77.55
77.30
77.05
76.67
31.40
31.33
25.30
23.48
23.42
f1 (ppm)

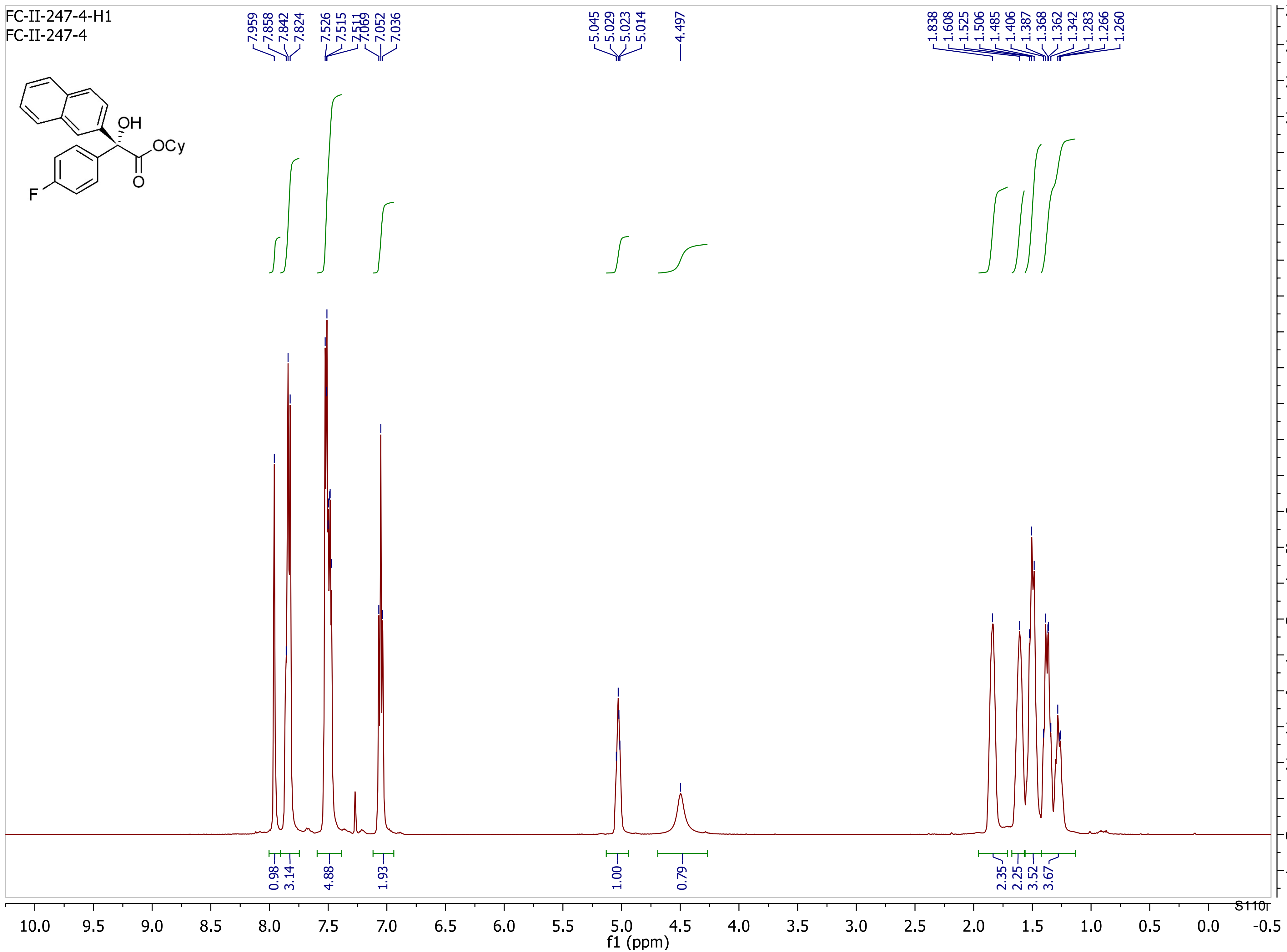


FC-II-246-5-H1-dilute
Std proton
MeO
OH
OCy
O
CN
7.642
7.637
7.624
7.620
7.603
7.283
7.270
6.973
6.959
6.901
6.896
6.882
4.998
4.989
4.981
4.974
4.965
4.394
3.785
1.872
1.864
1.766
1.619
1.570
1.552
1.526
1.508
1.490
1.430
1.412
1.404
1.392
1.386
1.379
1.373
1.366
1.360
1.353
1.346
1.341
1.335
1.319
1.298
1.279
1.261
1.255
3.46
1.62
1.73
0.86
1.00
0.79
3.25
1.27
1.30
5.27
4.86
f1 (ppm)

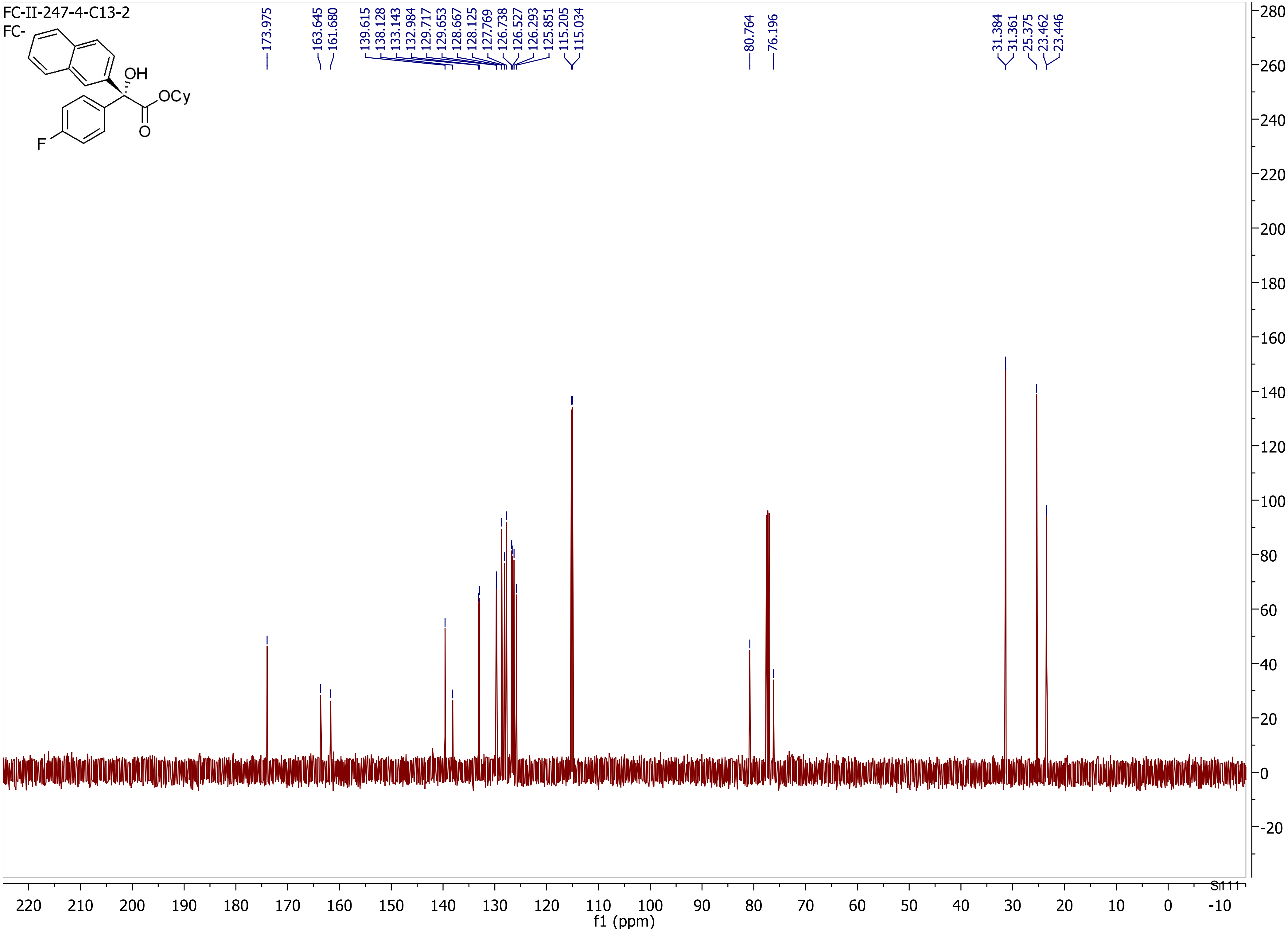

FC-II-246-5-C13-b
Standard Carbon
172.93
159.80
147.18
143.12
131.94
129.63
128.56
119.63
118.93
114.00
113.22
111.99
80.59
76.58
55.51
31.37
31.28
25.29
23.47
23.41
MeO
OH
OCy
O
CN
f1 (ppm)

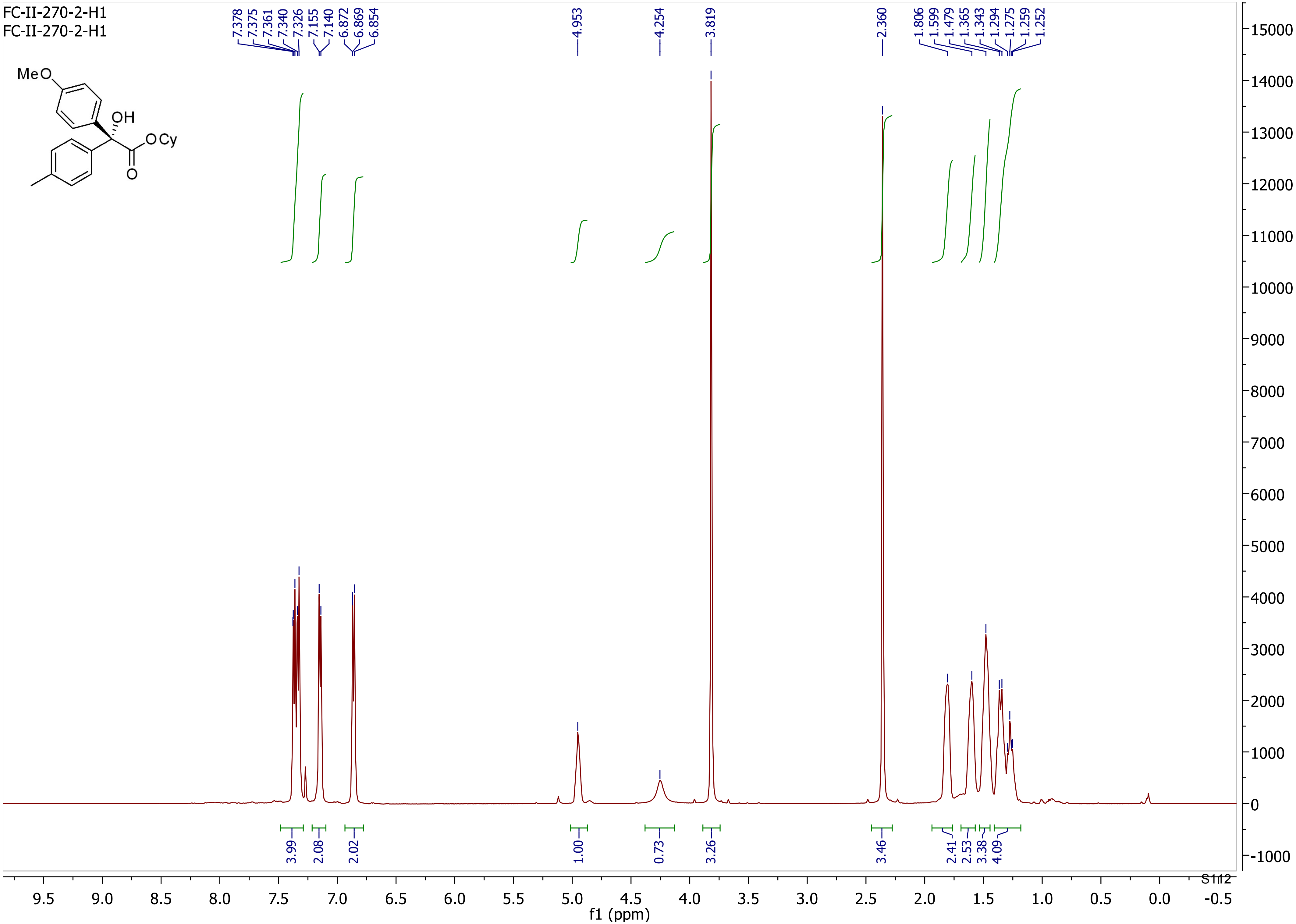

FC-II-209-4-H1
Std proton
OH
OCy
O
F
f1 (ppm)

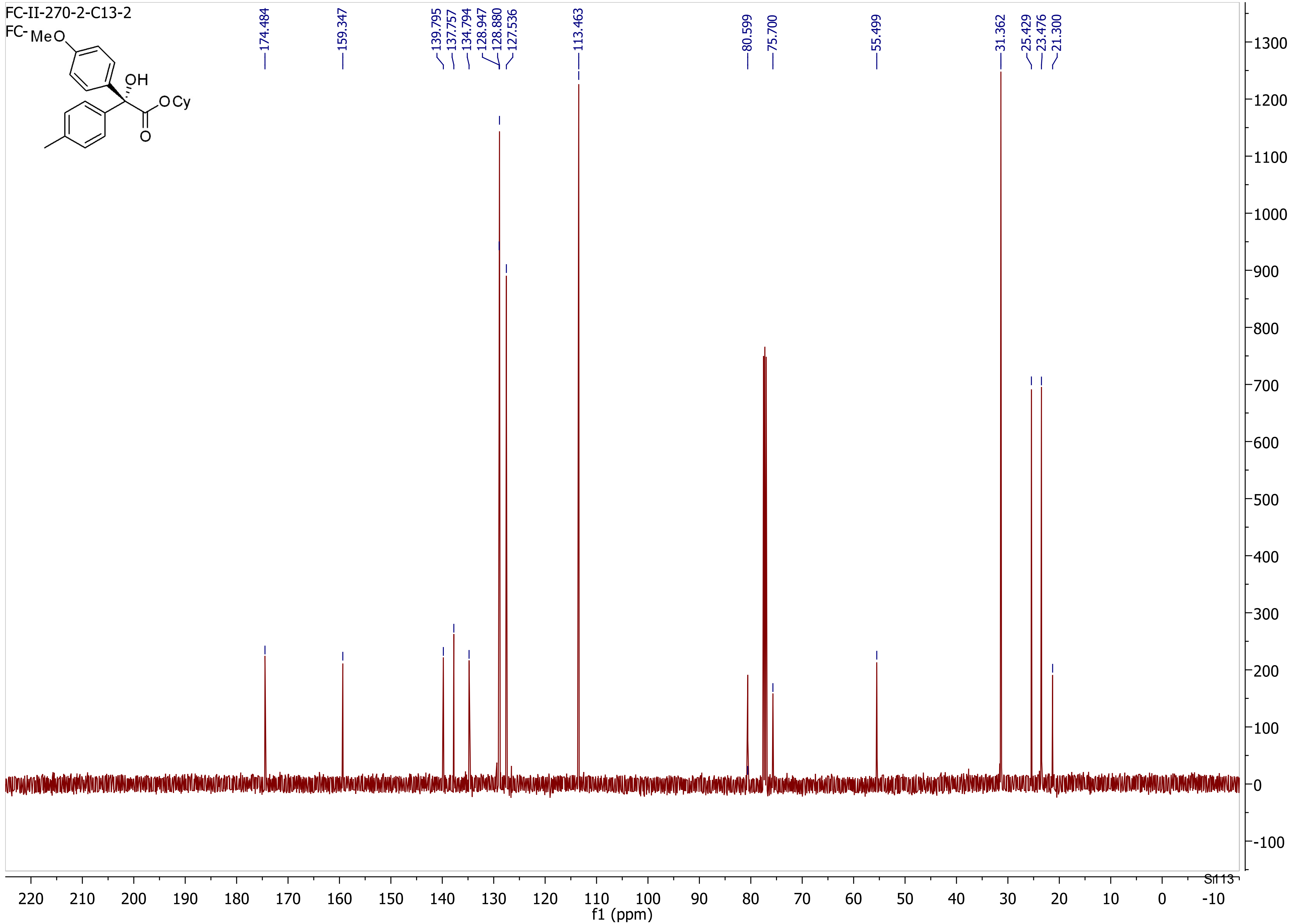

FC-II-209-4-C13-better
Standard Carbon
OH
OCy
O
F
173.972
141.343
141.116
140.723
129.615
129.045
127.974
127.719
127.350
127.074
115.163
114.993
104.988
80.453
77.523
77.269
77.015
76.148
31.365
31.330
25.375
23.437
f1 (ppm)

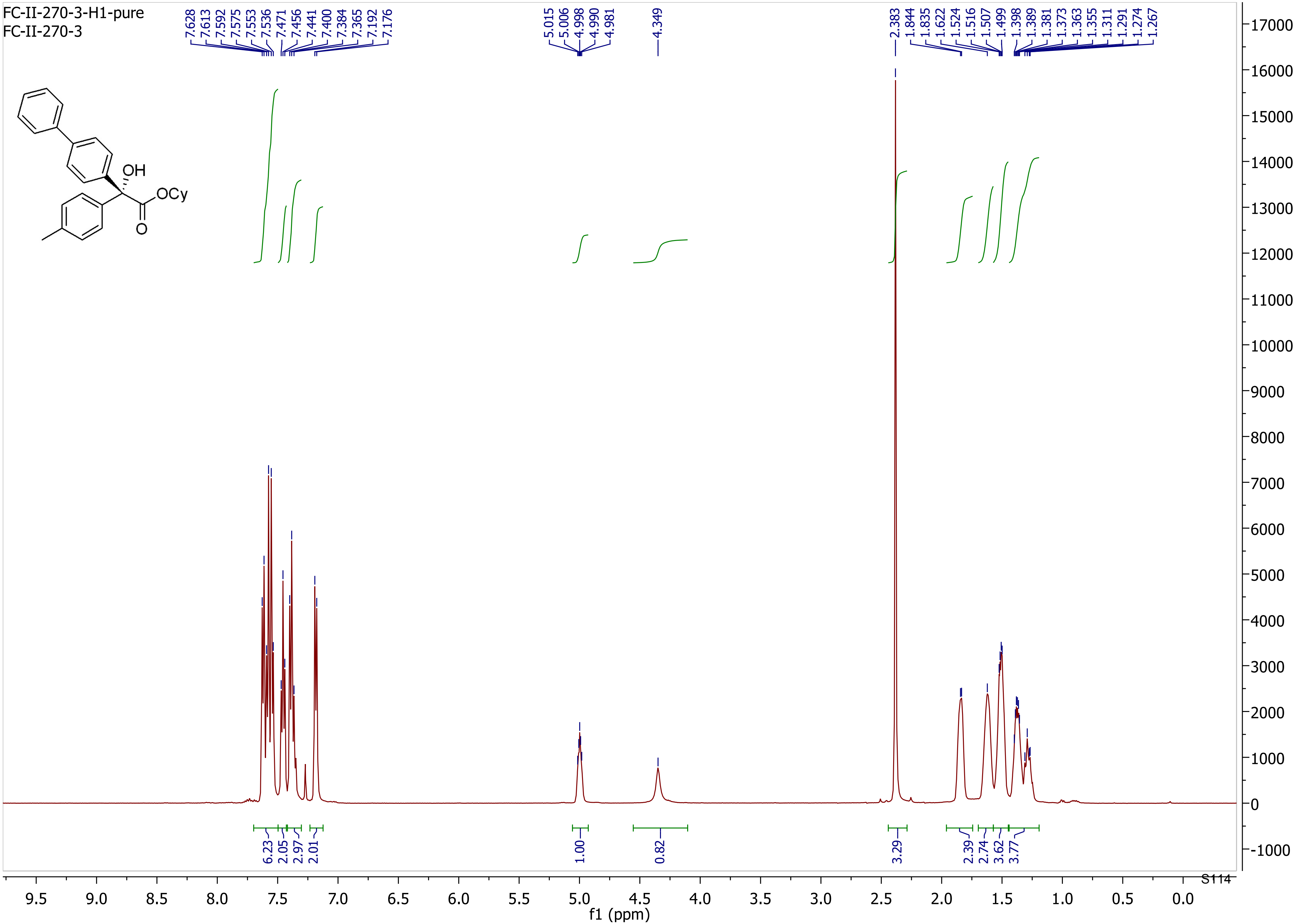


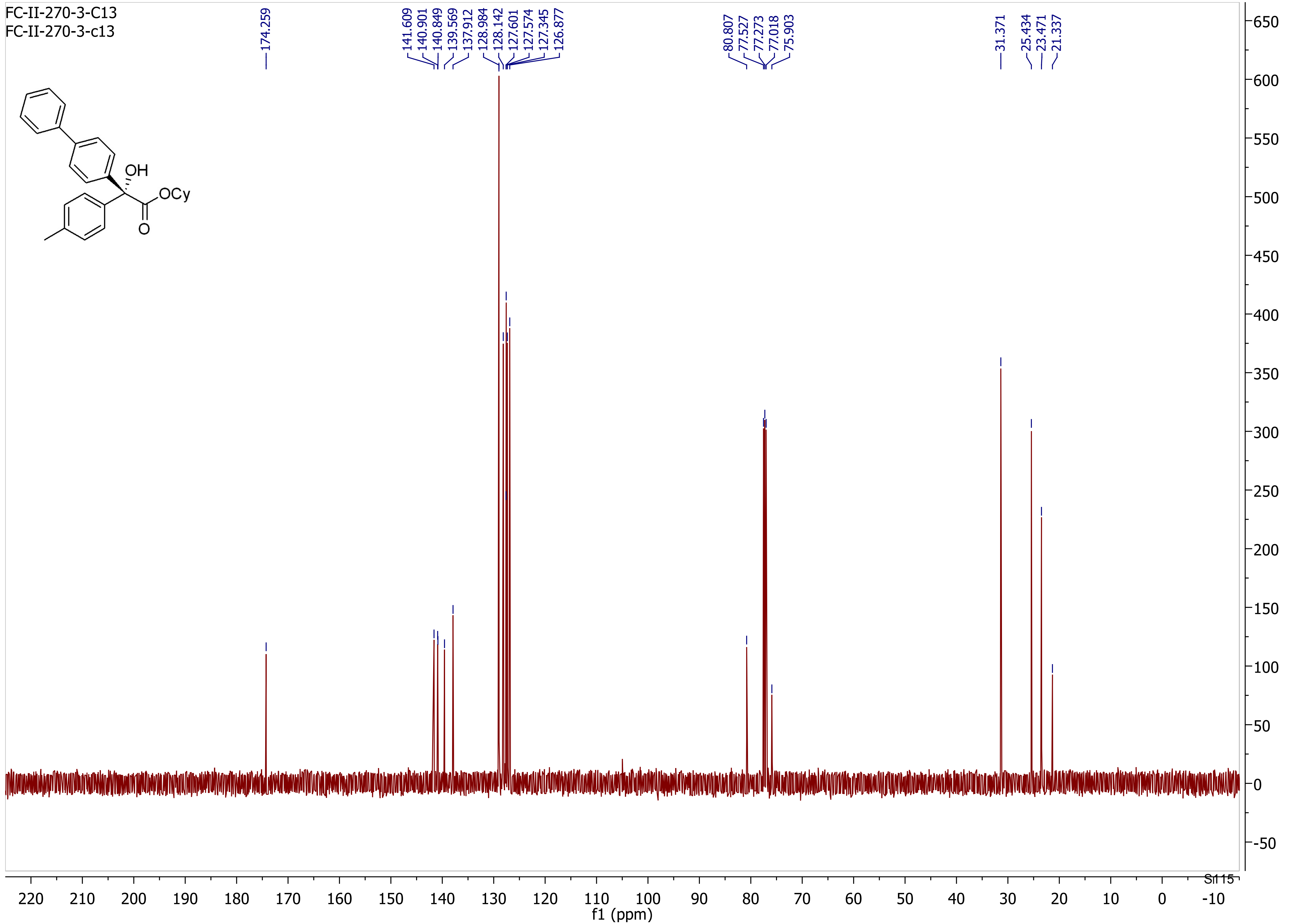
FC-II-247-4-H1
FC-II-247-4
OH
OCy
O
F
7.959
7.858
7.842
7.824
7.526
7.515
7.511
7.069
7.052
7.036
5.045
5.029
5.023
5.014
4.497
1.838
1.608
1.525
1.506
1.485
1.406
1.387
1.368
1.362
1.342
1.283
1.266
1.260
0.98
3.14
4.88
1.93
1.00
0.79
2.35
2.25
3.52
3.67
f1 (ppm)


FC-II-247-4-C13-2
FC-
OH
OCy
O
F
173.975
163.645
161.680
139.615
138.128
133.143
132.984
129.717
129.653
128.667
128.125
127.769
126.738
126.527
126.293
125.851
115.205
115.034
80.764
76.196
31.384
31.361
25.375
23.462
23.446
f1 (ppm)

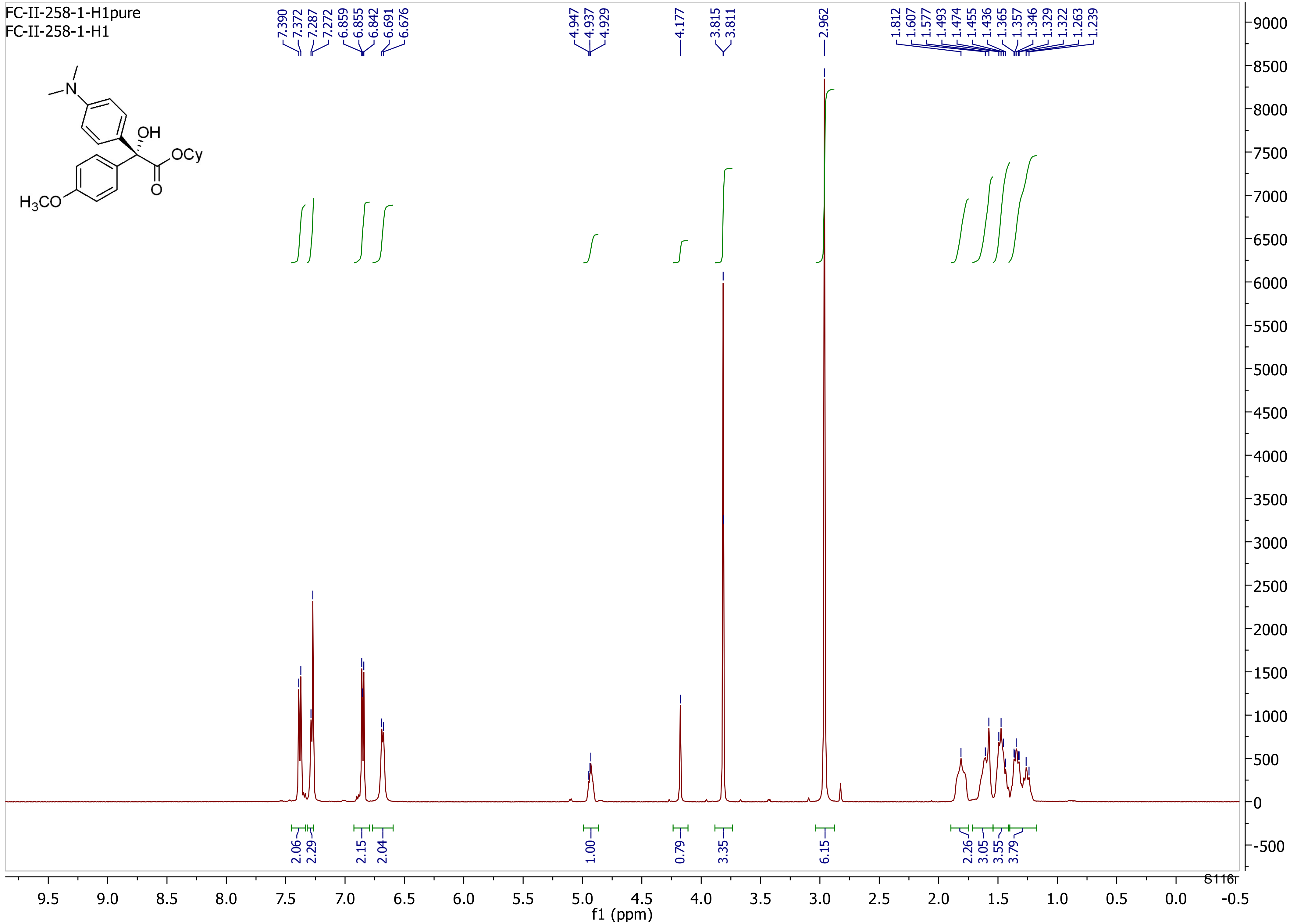

FC-II-270-2-H1
FC-II-270-2-H1
MeO
OH
OCy
O
7.378
7.375
7.361
7.340
7.326
7.155
7.140
6.872
6.869
6.854
4.953
4.254
3.819
2.360
1.806
1.599
1.479
1.365
1.343
1.294
1.275
1.259
1.252
3.99
2.08
2.02
1.00
0.73
3.26
3.46
2.41
2.53
3.38
4.09
f1 (ppm)

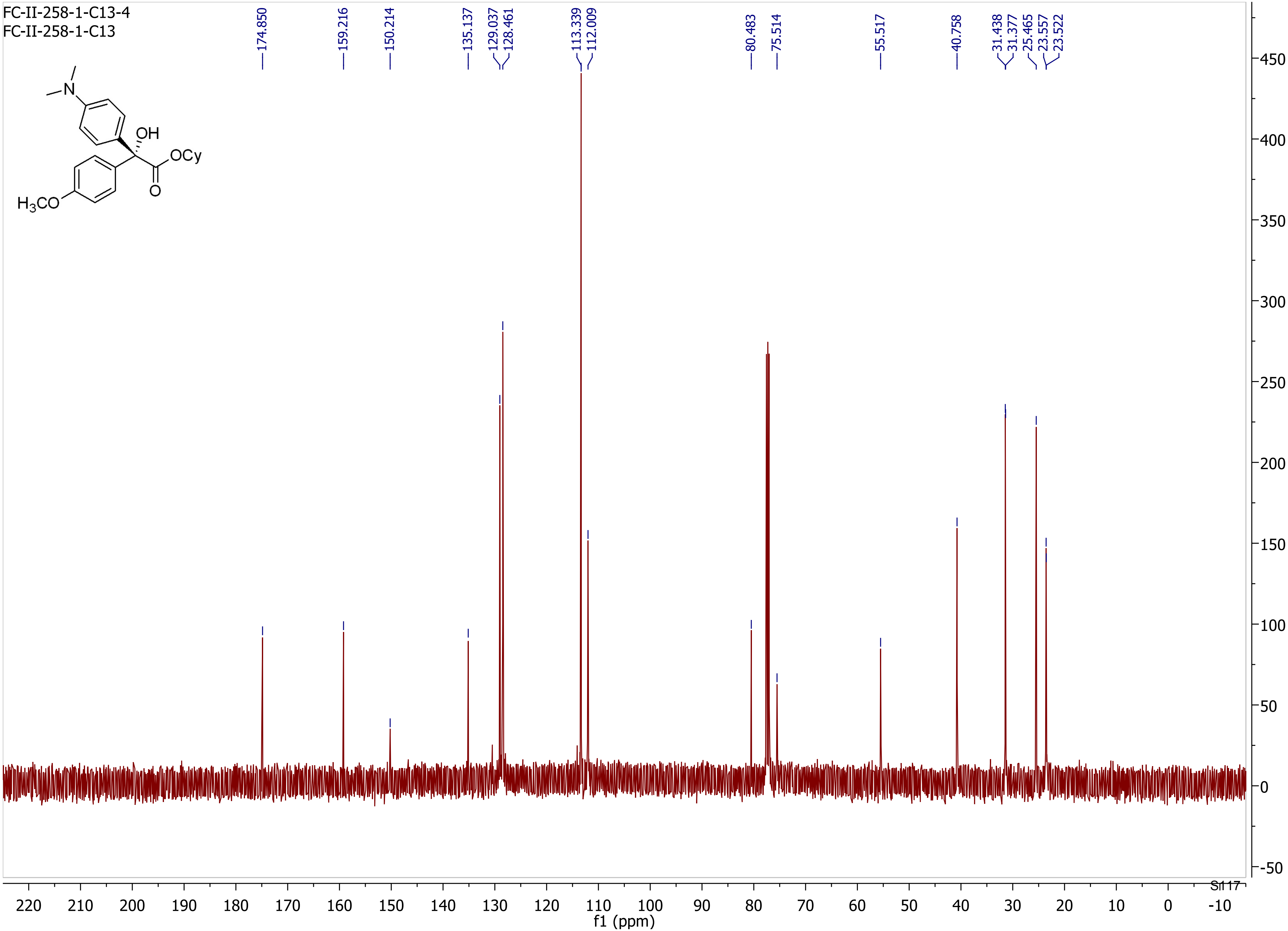

FC-II-270-2-C13-2
FC-
MeO
OH
OCy
O
174.484
159.347
139.795
137.757
134.794
128.947
128.880
127.536
113.463
80.599
75.700
55.499
31.362
25.429
23.476
21.300
f1 (ppm)

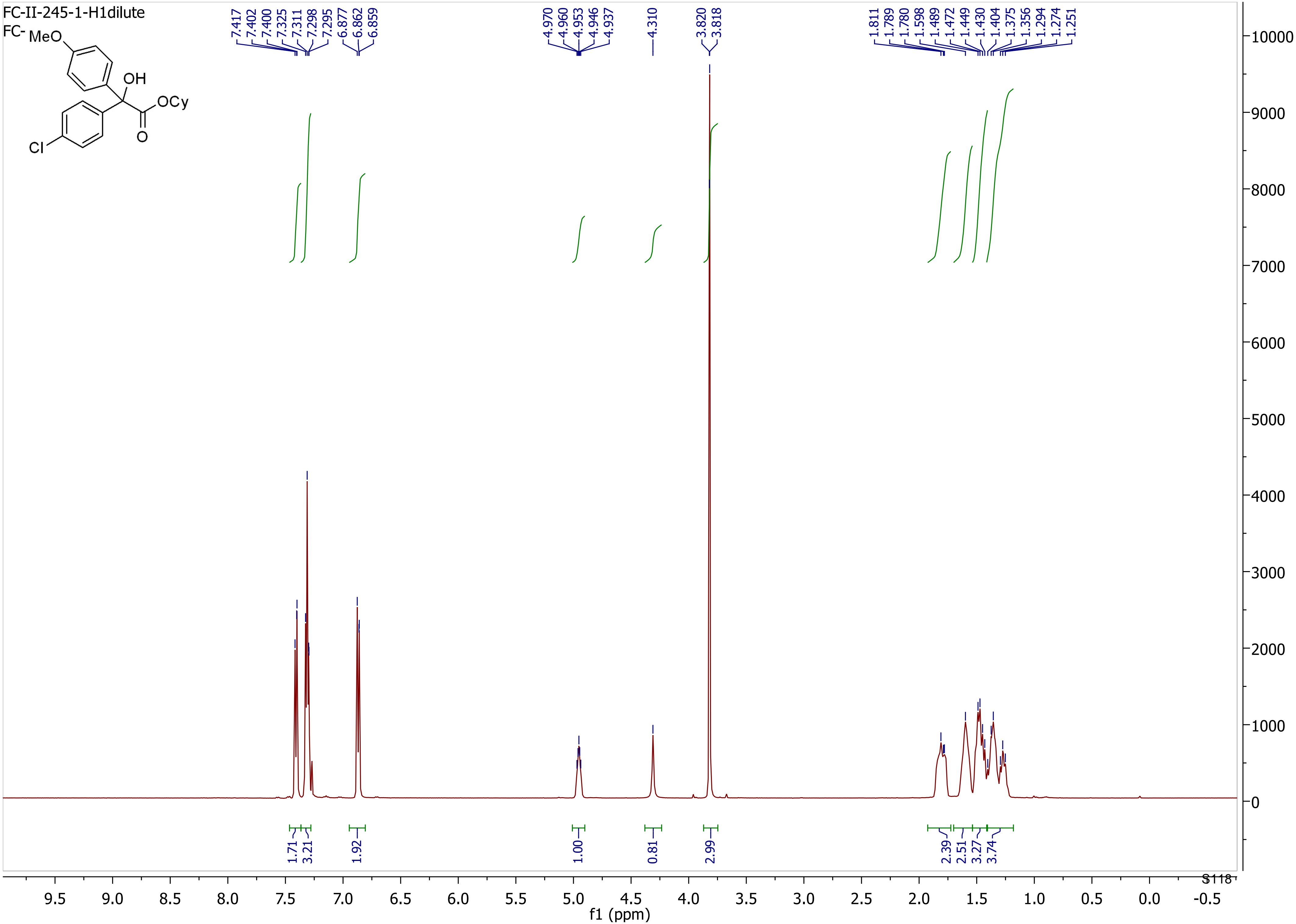

FC-II-270-3-H1-pure
FC-II-270-3
OH
OCy
O
7.628 7.613 7.592 7.575 7.553 7.536 7.471 7.456 7.441 7.400 7.384 7.365 7.192 7.176
5.015 5.006 4.998 4.990 4.981
4.349
2.383 1.844 1.835 1.622 1.524 1.516 1.507 1.499 1.398 1.389 1.381 1.373 1.363 1.355 1.311 1.291 1.274 1.267
6.23 2.05 2.97 2.01
1.00
0.82
3.29
2.39 2.74 3.62 3.77
f1 (ppm)



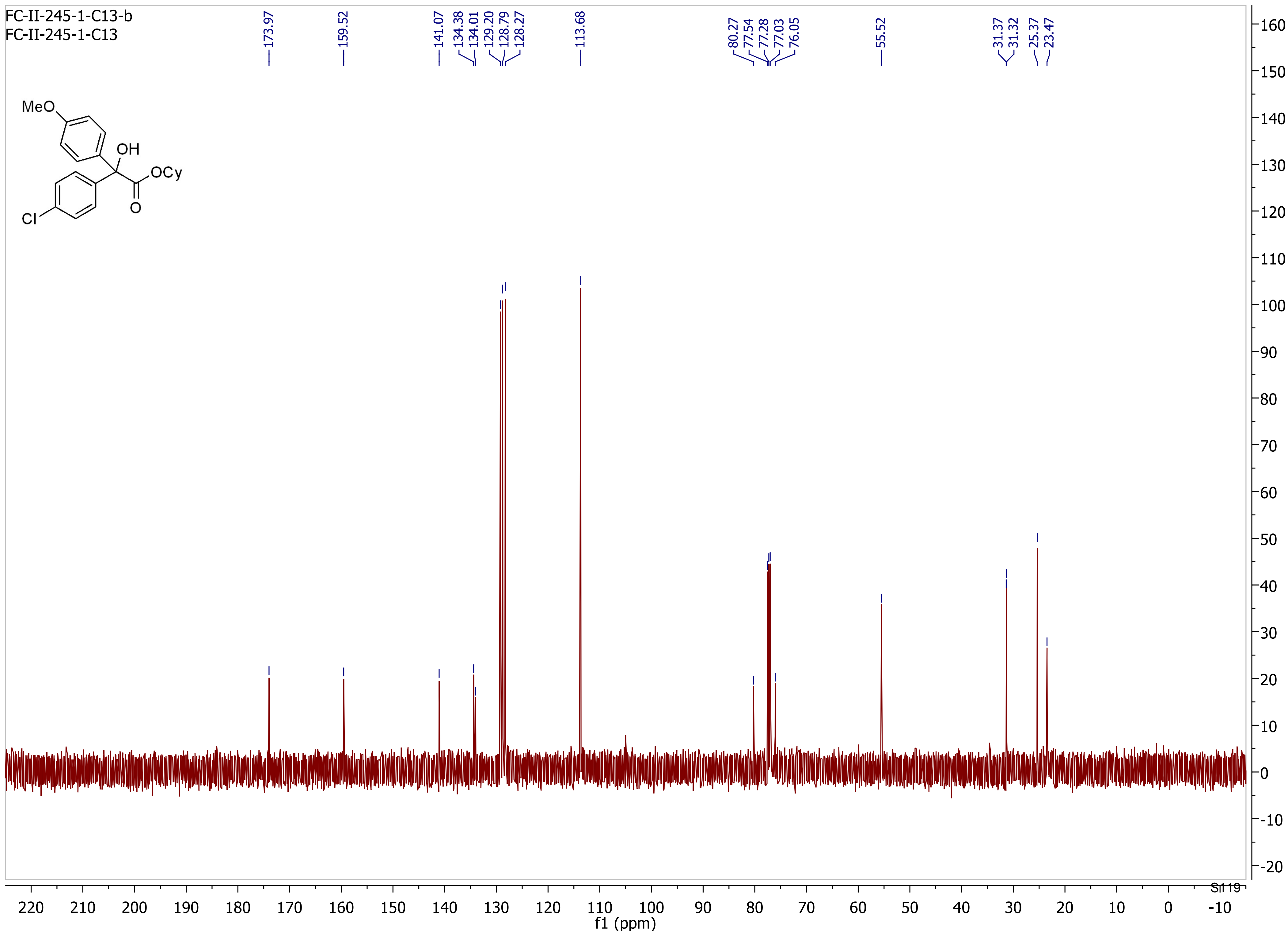
FC-II-270-3-C13
FC-II-270-3-c13
174.259
141.609
140.901
140.849
139.569
137.912
128.984
128.142
127.601
127.574
127.345
126.877
80.807
77.527
77.273
77.018
75.903
31.371
25.434
23.471
21.337
OH
OCy
O
f1 (ppm)

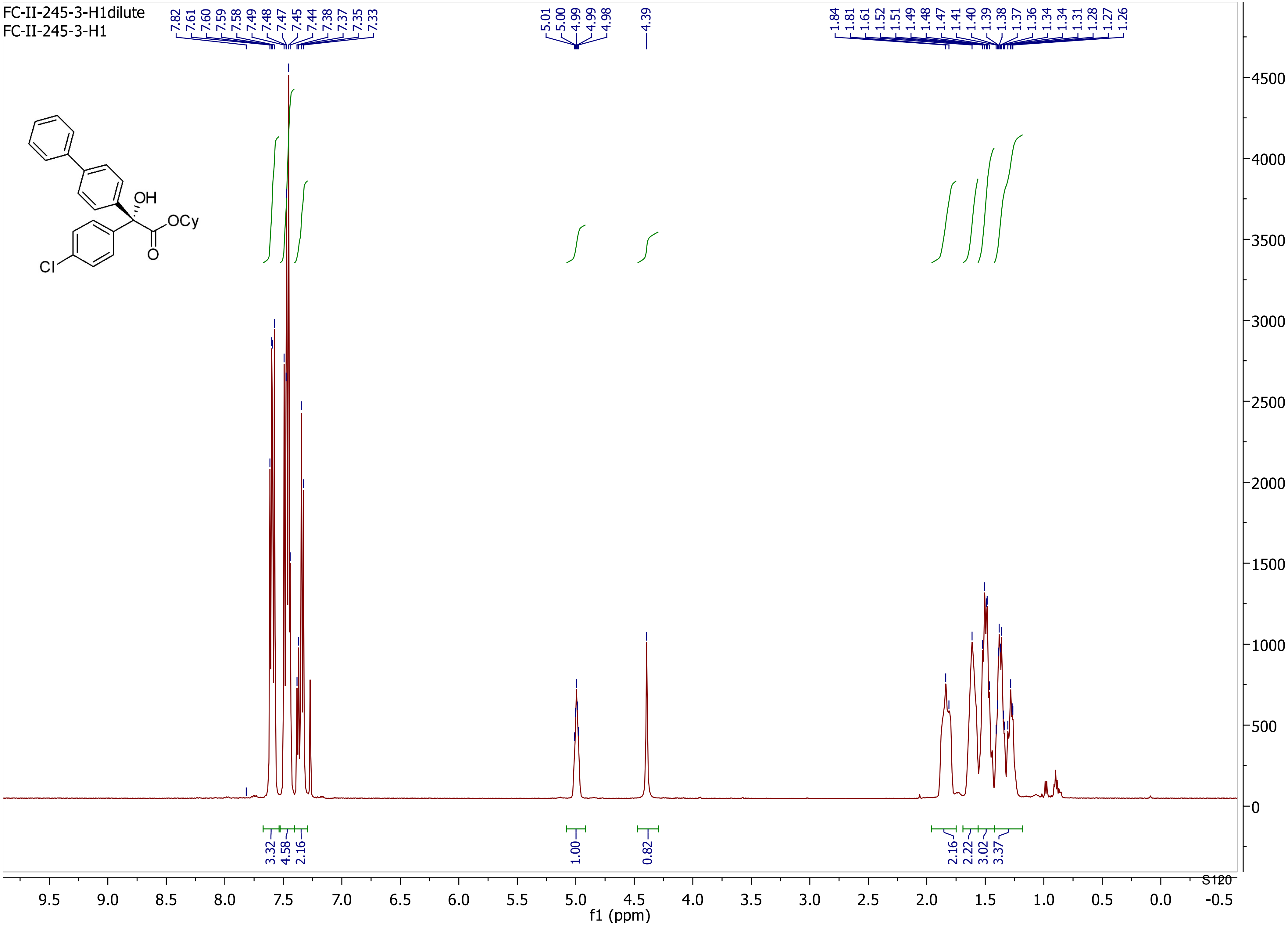

FC-II-258-1-H1pure
FC-II-258-1-H1
OH
OCy
O
N
H3CO
7.390
7.372
7.287
7.272
6.859
6.855
6.842
6.691
6.676
4.947
4.937
4.929
4.177
3.815
3.811
2.962
1.812
1.607
1.577
1.493
1.474
1.455
1.436
1.365
1.357
1.346
1.329
1.322
1.263
1.239
2.06
2.29
2.15
2.04
1.00
0.79
3.35
6.15
2.26
3.05
3.55
3.79
f1 (ppm)

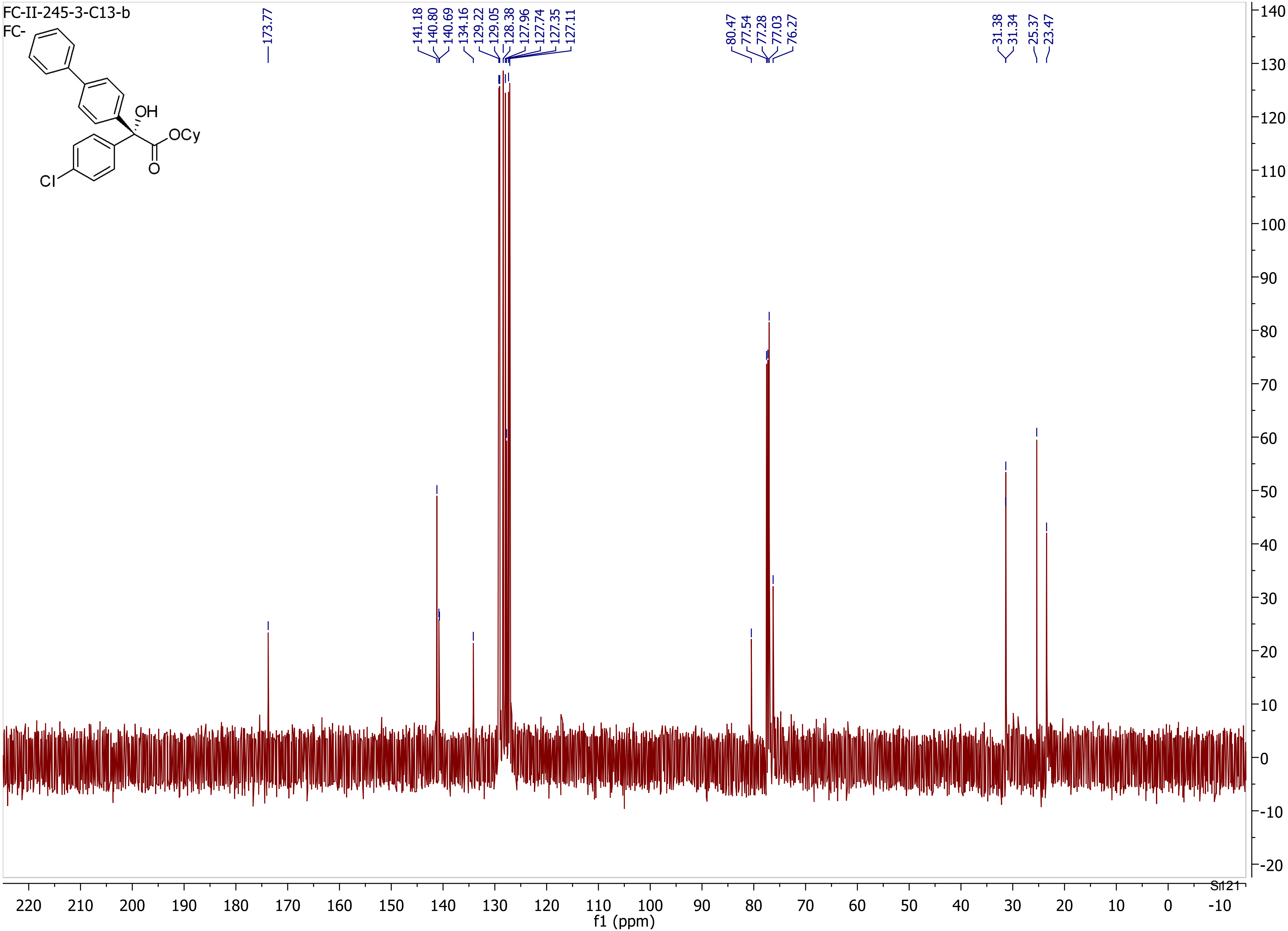

FC-II-258-1-C13-4
FC-II-258-1-C13
OH
OCy
O
N
H3CO
174.850
159.216
150.214
135.137
129.037
128.461
113.339
112.009
80.483
75.514
55.517
40.758
31.438
31.377
25.465
23.557
23.522
f1 (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
450 400 350 300 250 200 150 100 50 0 -50

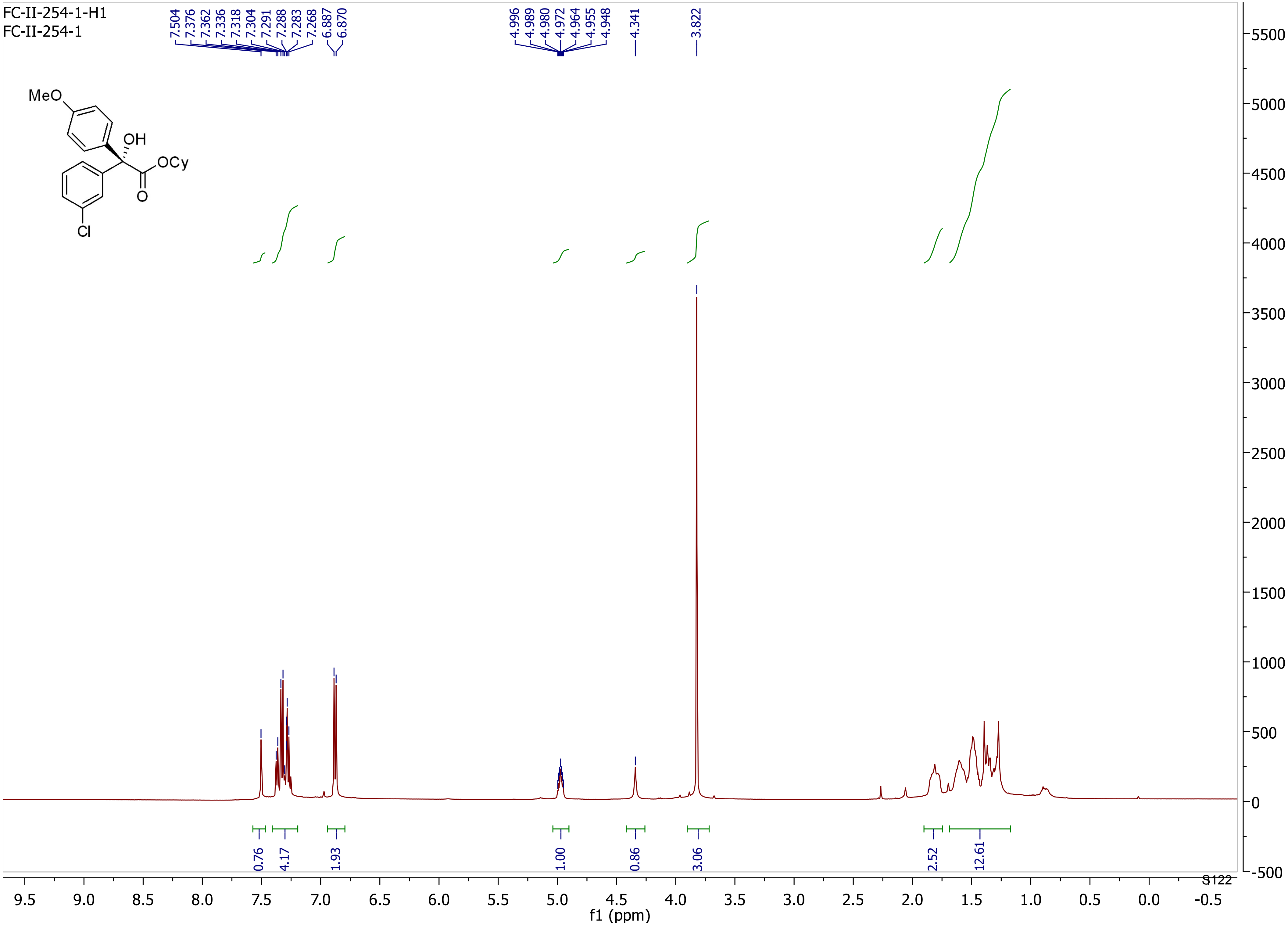


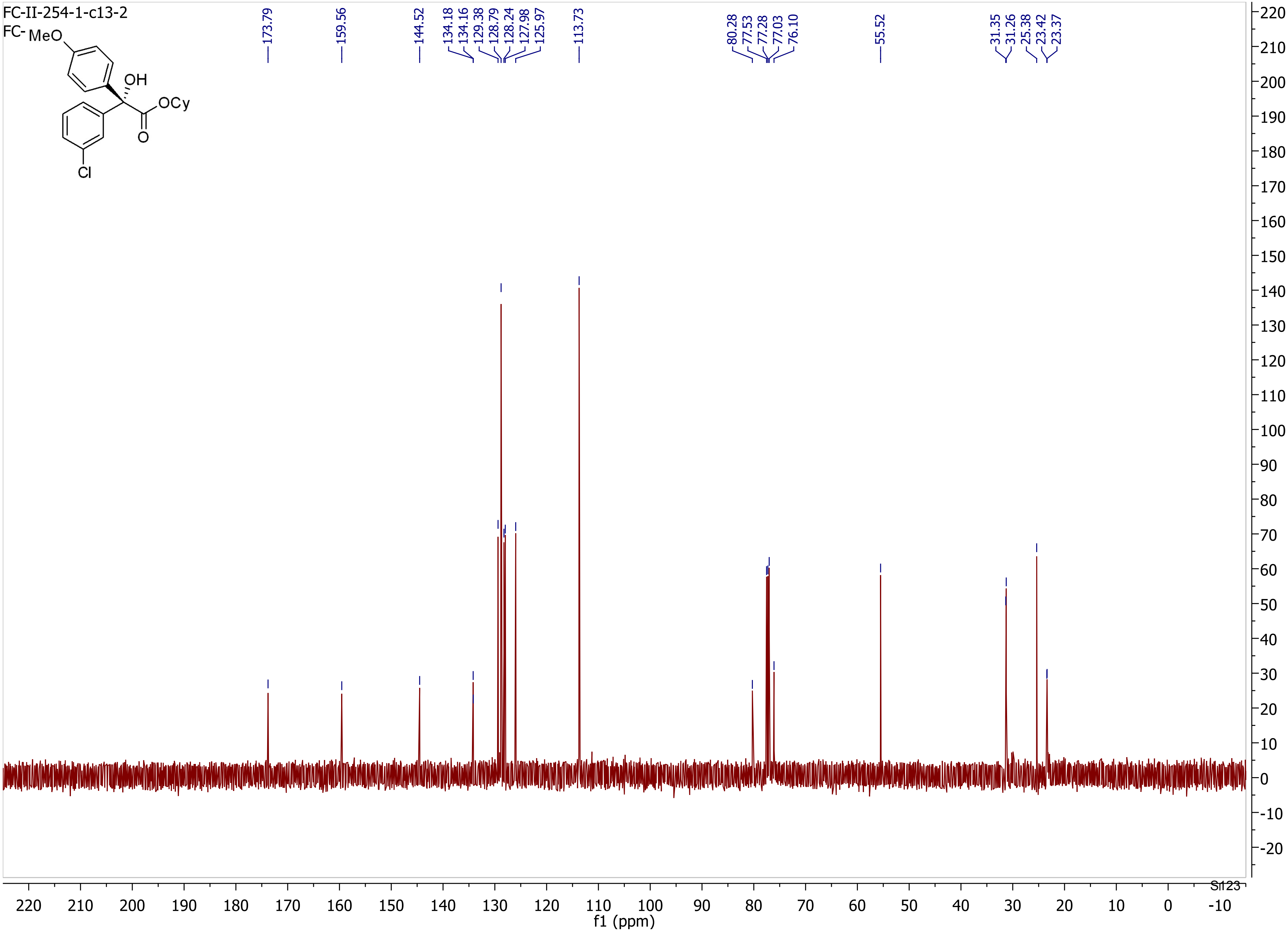
FC-II-245-1-H1dilute
FC-
MeO
OH
OCy
O
Cl
7.417
7.402
7.400
7.325
7.311
7.298
7.295
6.877
6.862
6.859
4.970
4.960
4.953
4.946
4.937
4.310
3.820
3.818
1.811
1.789
1.780
1.598
1.489
1.472
1.449
1.430
1.404
1.375
1.356
1.294
1.274
1.251
1.71
3.21
1.92
1.00
0.81
2.99
2.39
2.51
3.27
3.74
f1 (ppm)

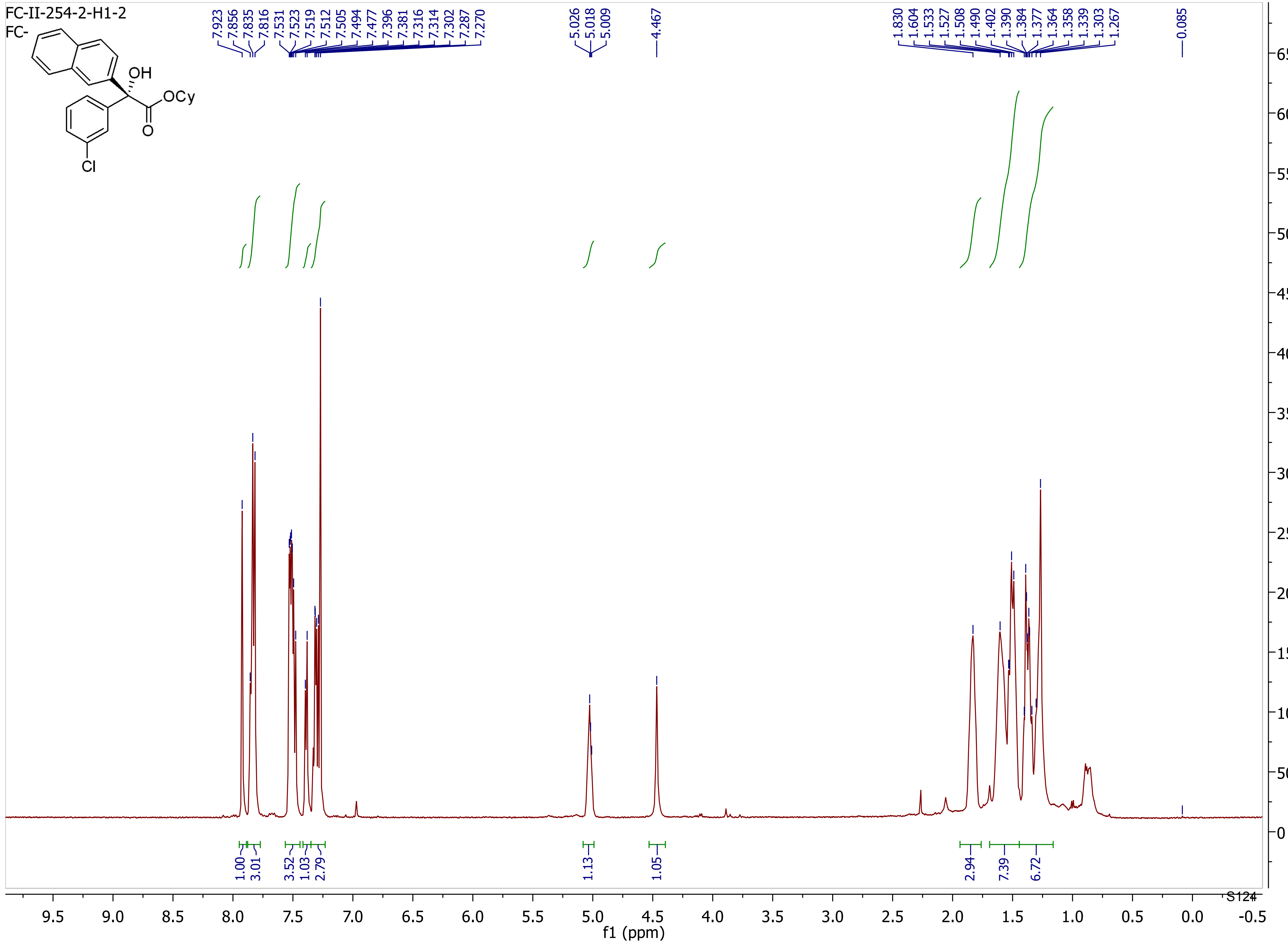

FC-II-245-1-C13-b
FC-II-245-1-C13
MeO
OH
OCy
Cl
O
173.97
159.52
141.07
134.38
134.01
129.20
128.79
128.27
113.68
80.27
77.54
77.28
77.03
76.05
55.52
31.37
31.32
25.37
23.47
f1 (ppm)

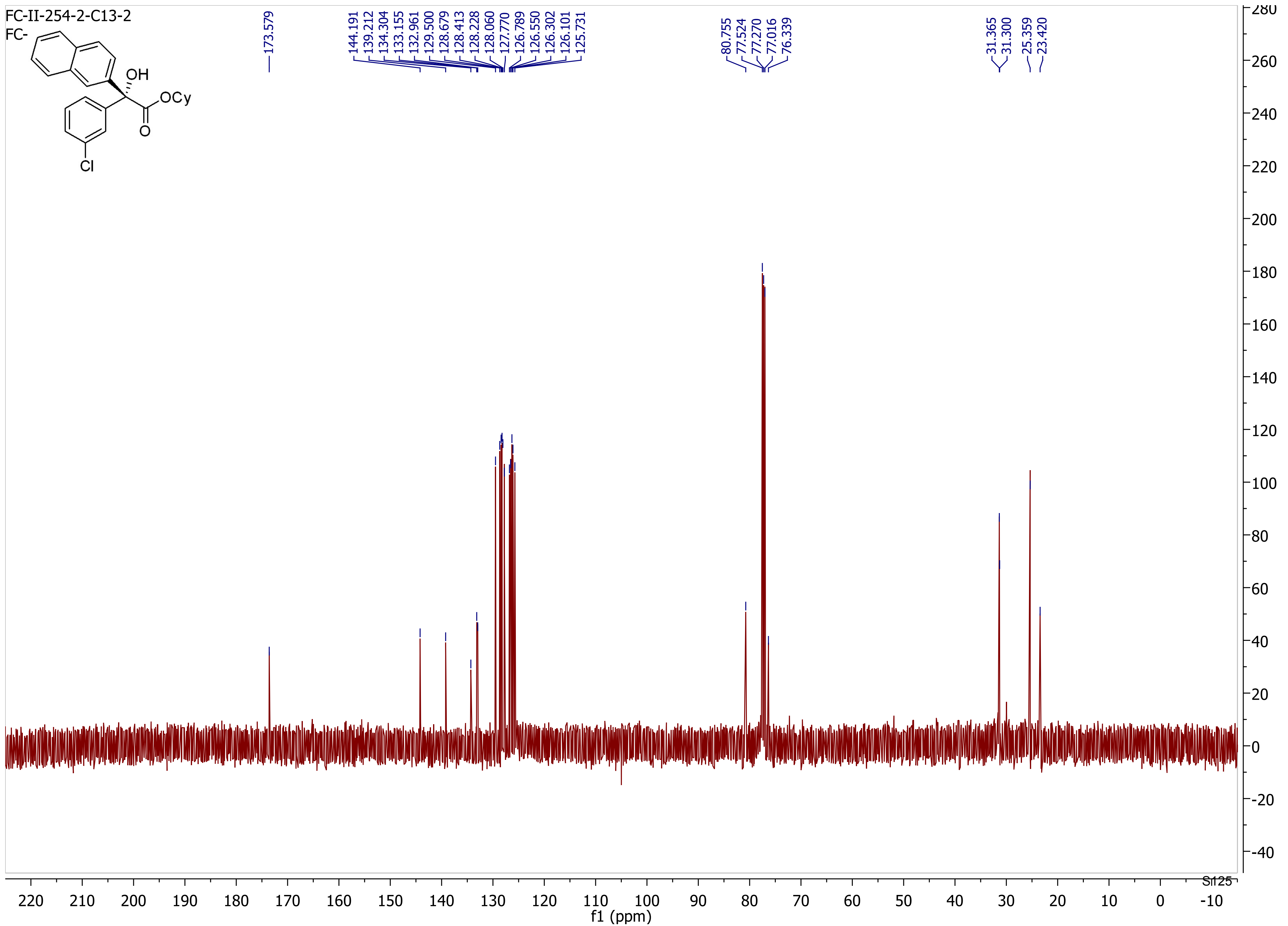

FC-II-245-3-H1dilute
FC-II-245-3-H1
7.82 7.61 7.60 7.59 7.58 7.49 7.48 7.47 7.45 7.44 7.38 7.37 7.35 7.33
5.01 5.00 4.99 4.99 4.98
4.39
1.84 1.81 1.61 1.52 1.51 1.49 1.48 1.47 1.41 1.40 1.39 1.38 1.37 1.36 1.34 1.34 1.31 1.28 1.27 1.26
OH
OCy
O
Cl
3.32 4.58 2.16 1.00 0.82 2.16 2.22 3.02 3.37
f1 (ppm)


FC-II-245-3-C13-b
FC-
OH
OCy
O
Cl
173.77
141.18
140.80
140.69
134.16
129.22
129.05
128.38
127.96
127.74
127.35
127.11
80.47
77.54
77.28
77.03
76.27
31.38
31.34
25.37
23.47
f1 (ppm)

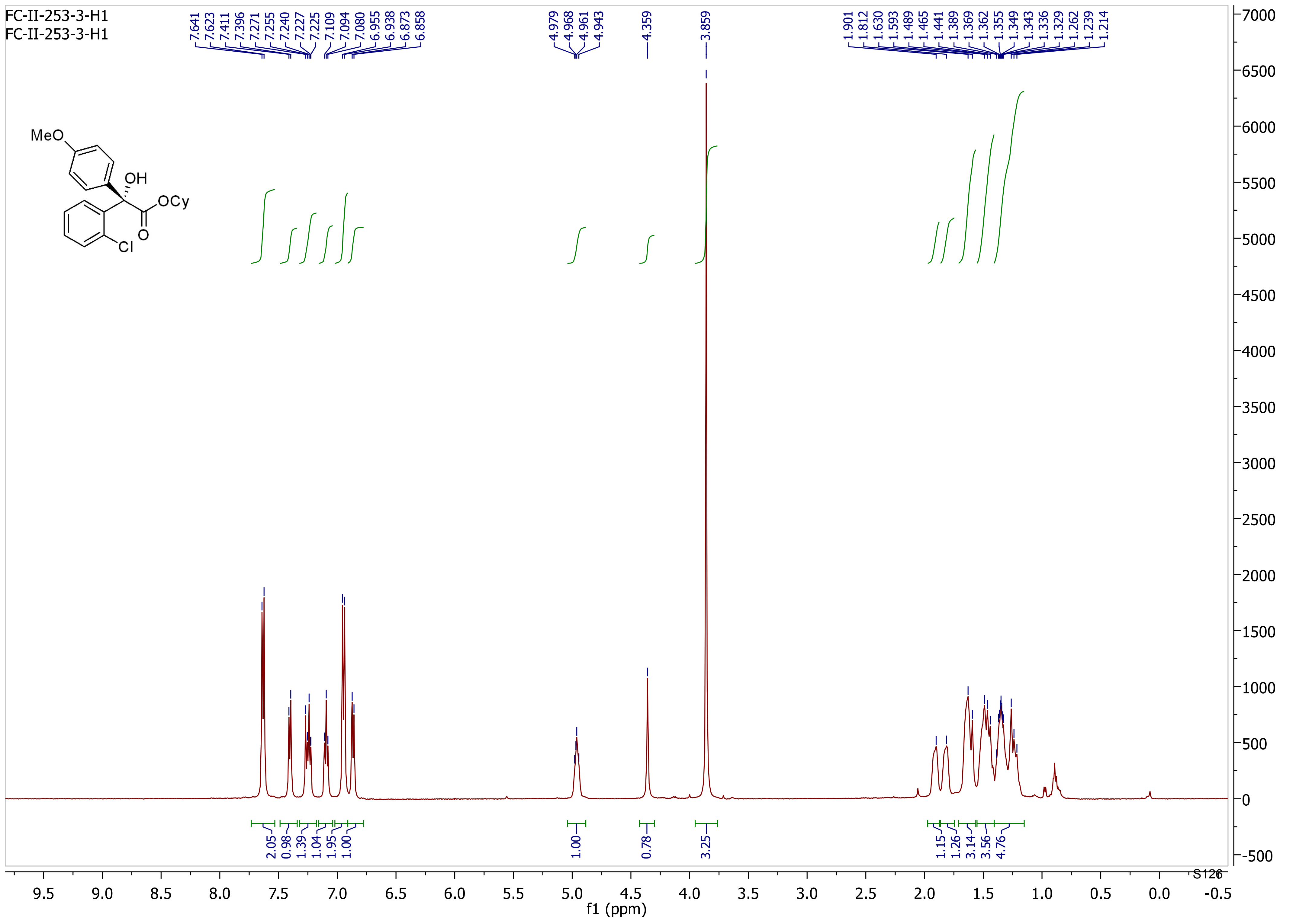

FC-II-254-1-H1
FC-II-254-1
7.504
7.376
7.362
7.336
7.318
7.304
7.291
7.288
7.283
7.268
6.887
6.870
4.996
4.989
4.980
4.972
4.964
4.955
4.948
4.341
3.822
MeO
OH
OCy
O
Cl
0.76
4.17
1.93
1.00
0.86
3.06
2.52
12.61
f1 (ppm)


FC-II-254-1-c13-2
FC-
MeO
OH
OCy
O
Cl
173.79
159.56
144.52
134.18
134.16
129.38
128.79
128.24
127.98
125.97
113.73
80.28
77.53
77.28
77.03
76.10
55.52
31.35
31.26
25.38
23.42
23.37
f1 (ppm)


FC-II-254-2-H1-2
FC-
OH
OCy
O
Cl
7.923
7.856
7.835
7.816
7.531
7.523
7.519
7.512
7.505
7.494
7.477
7.396
7.381
7.316
7.314
7.302
7.287
7.270
5.026
5.018
5.009
4.467
1.830
1.604
1.533
1.527
1.508
1.490
1.402
1.390
1.384
1.377
1.364
1.358
1.339
1.303
1.267
0.085
1.00
3.01
3.52
1.03
2.79
1.13
1.05
2.94
7.39
6.72
f1 (ppm)

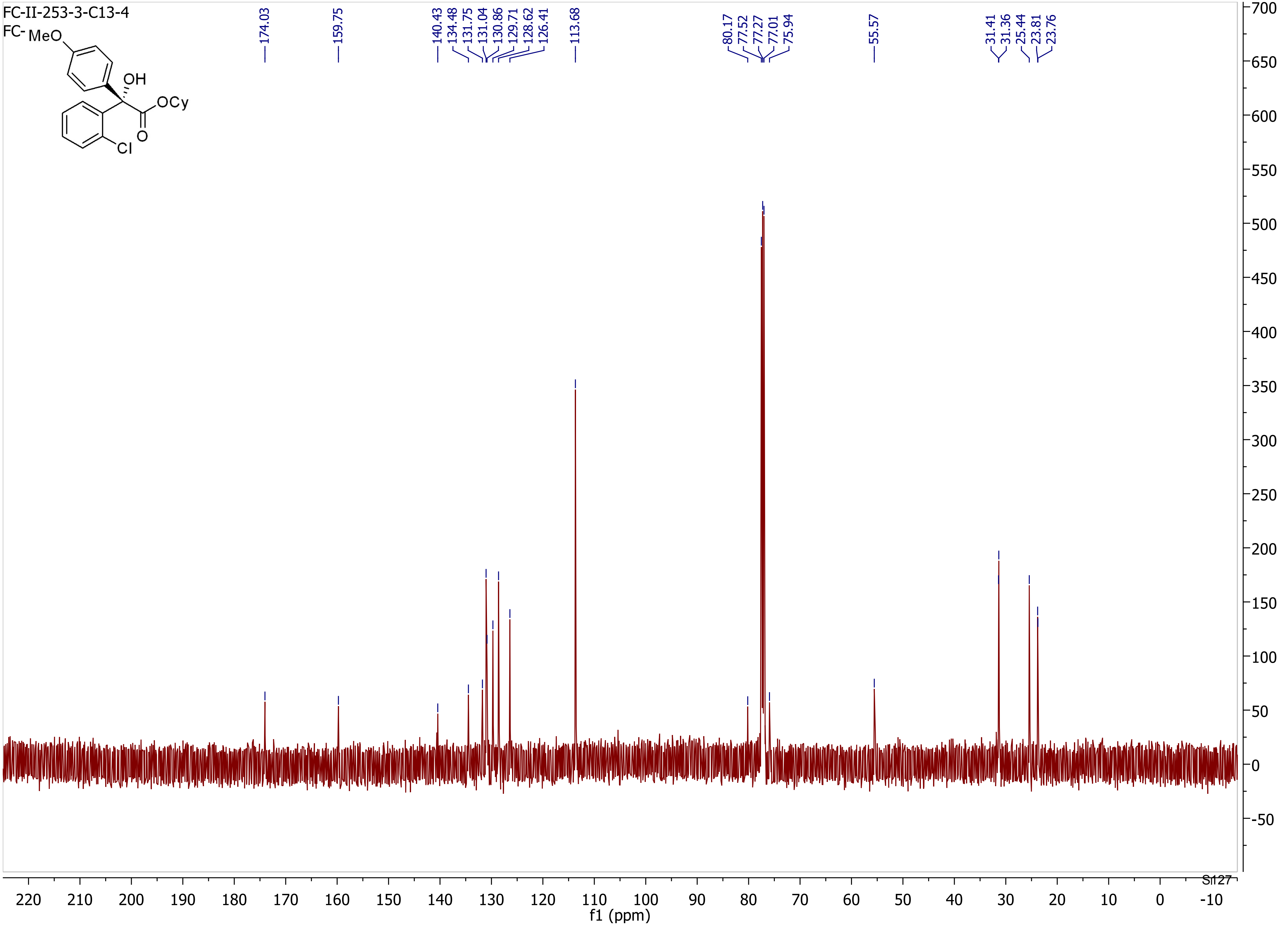


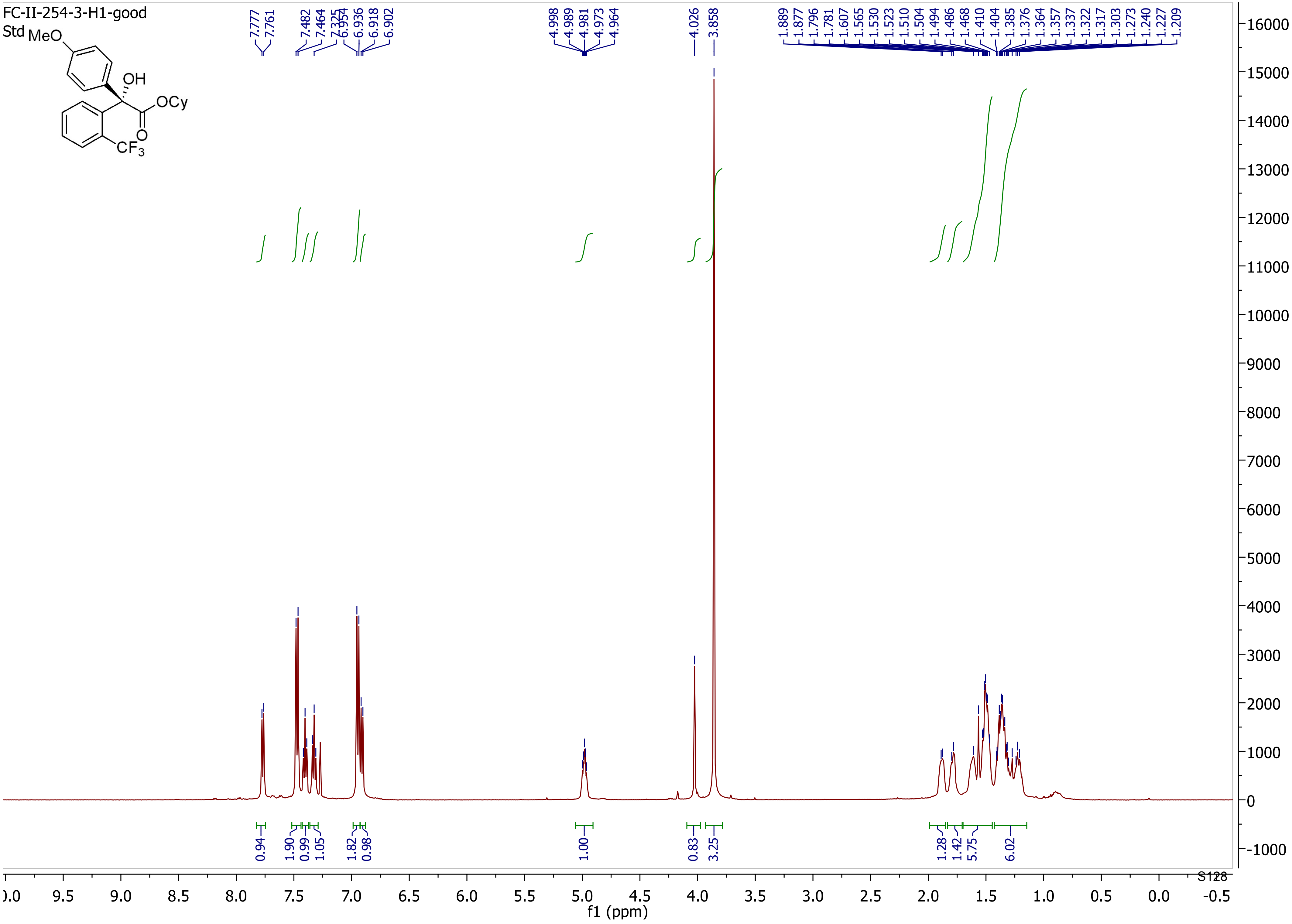
FC-II-254-2-C13-2
FC-
OH
OCy
O
Cl
173.579
144.191
139.212
134.304
133.155
132.961
129.500
128.679
128.413
128.228
128.060
127.770
126.789
126.550
126.302
126.101
125.731
80.755
77.524
77.270
77.016
76.339
31.365
31.300
25.359
23.420
f1 (ppm)

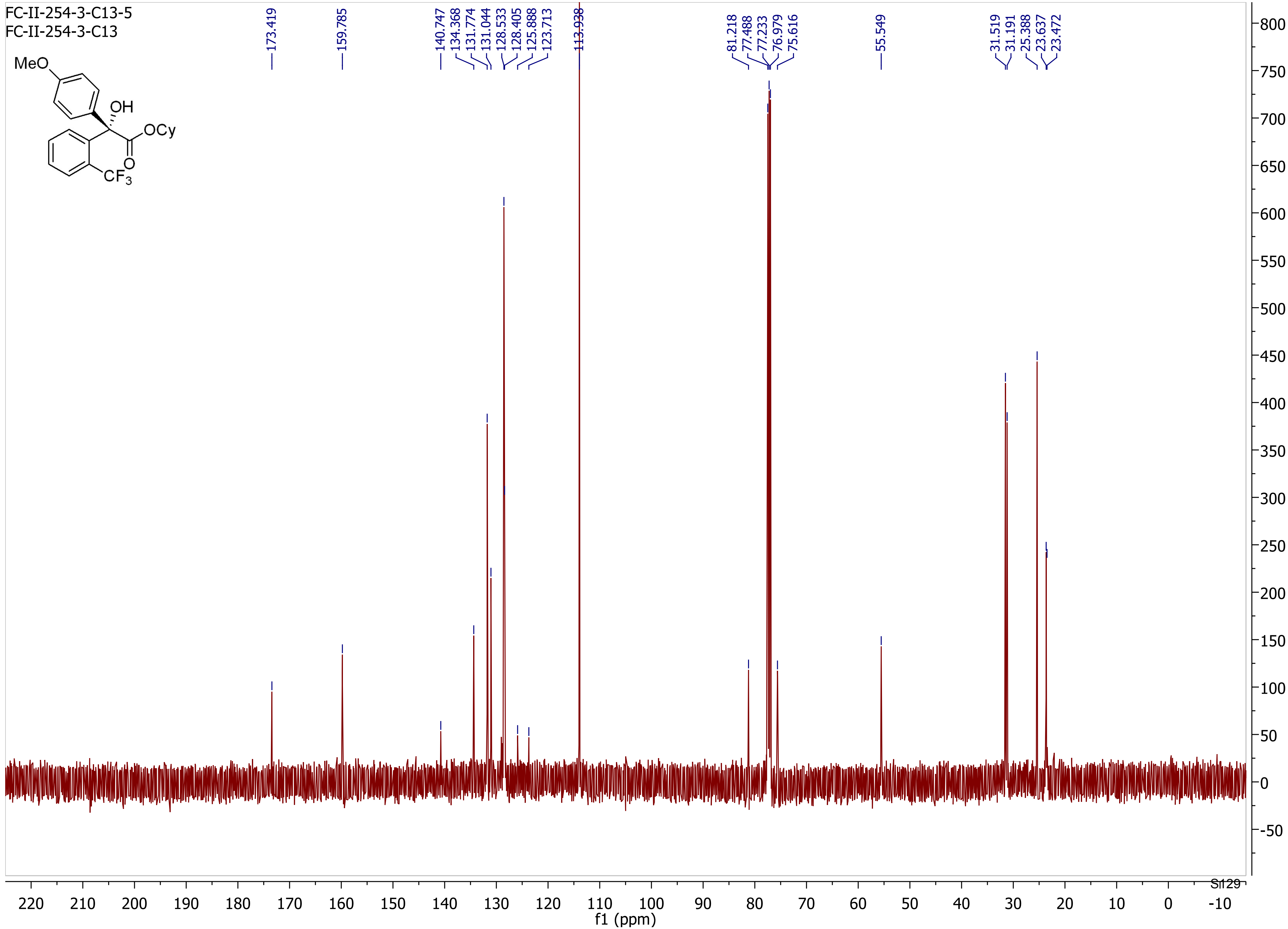

FC-II-253-3-H1
FC-II-253-3-H1
MeO
OH
OCy
O
Cl
7.641
7.623
7.411
7.396
7.271
7.255
7.240
7.227
7.225
7.109
7.094
7.080
6.955
6.938
6.873
6.858
4.979
4.968
4.961
4.943
4.359
3.859
1.901
1.812
1.630
1.593
1.489
1.465
1.441
1.389
1.369
1.362
1.355
1.349
1.343
1.336
1.329
1.262
1.239
1.214
2.05
0.98
1.39
1.04
1.95
1.00
1.00
0.78
3.25
1.15
1.26
3.14
3.56
4.76
f1 (ppm)

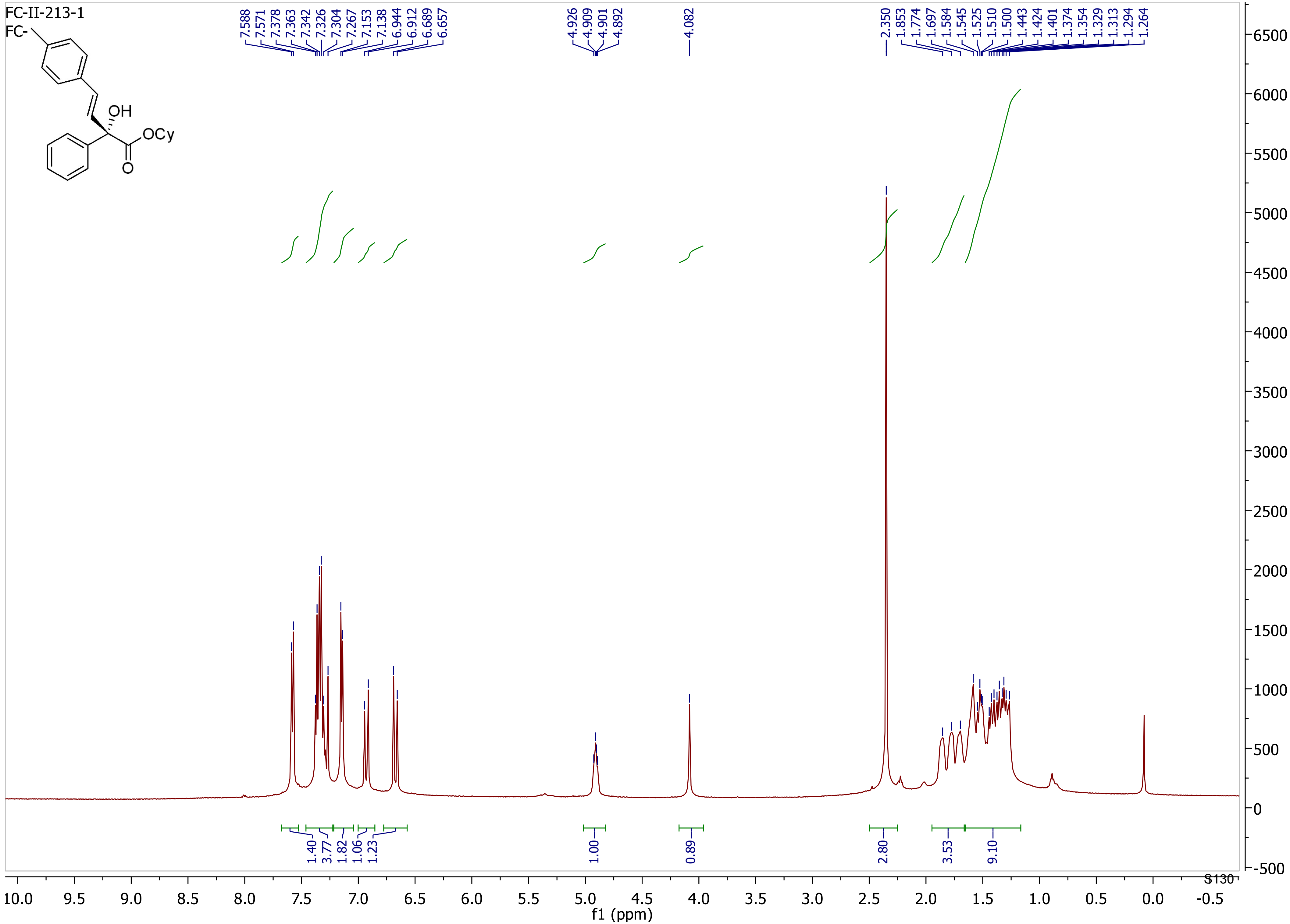
FC-II-253-3-C13-4

FC-

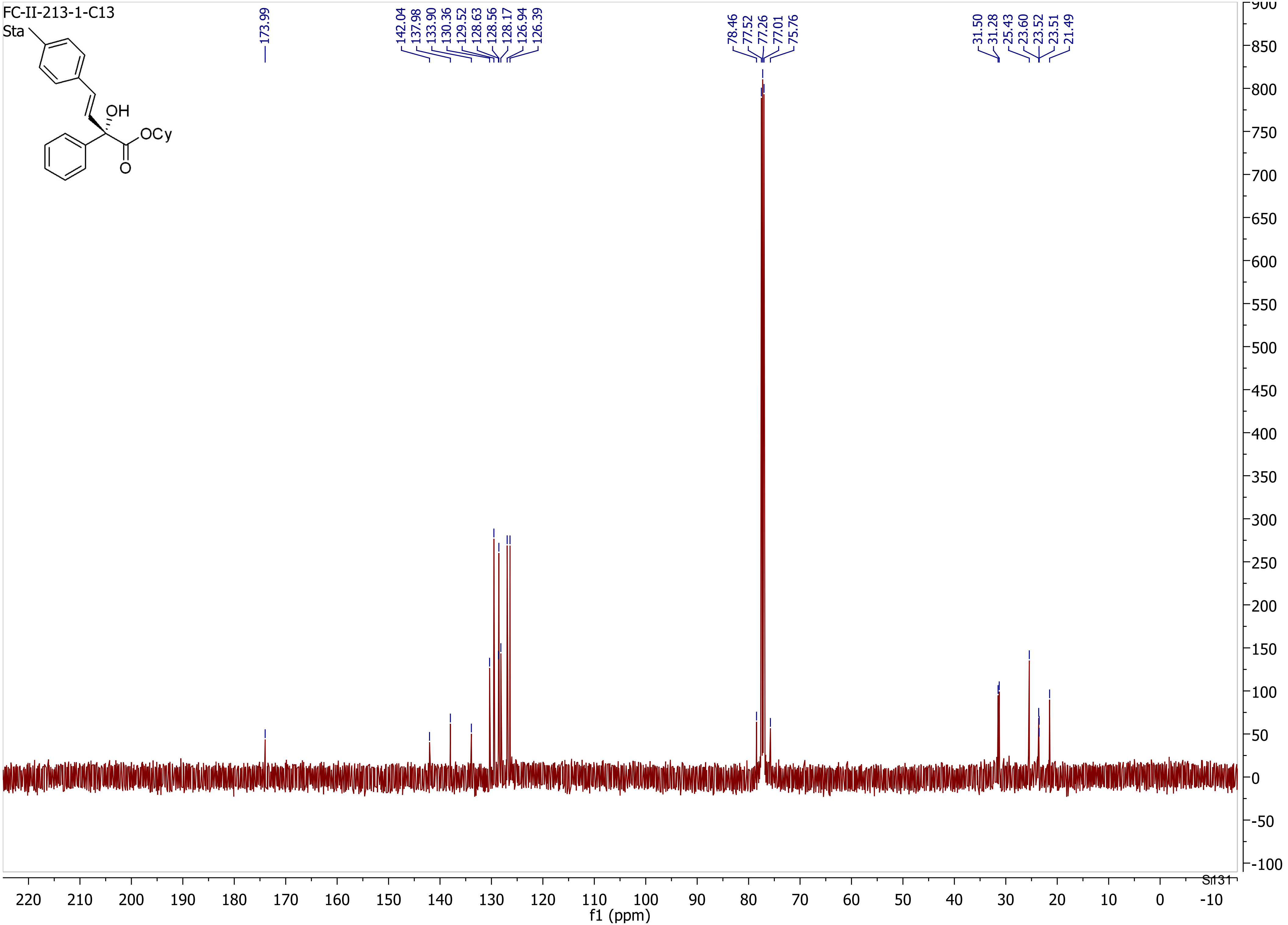


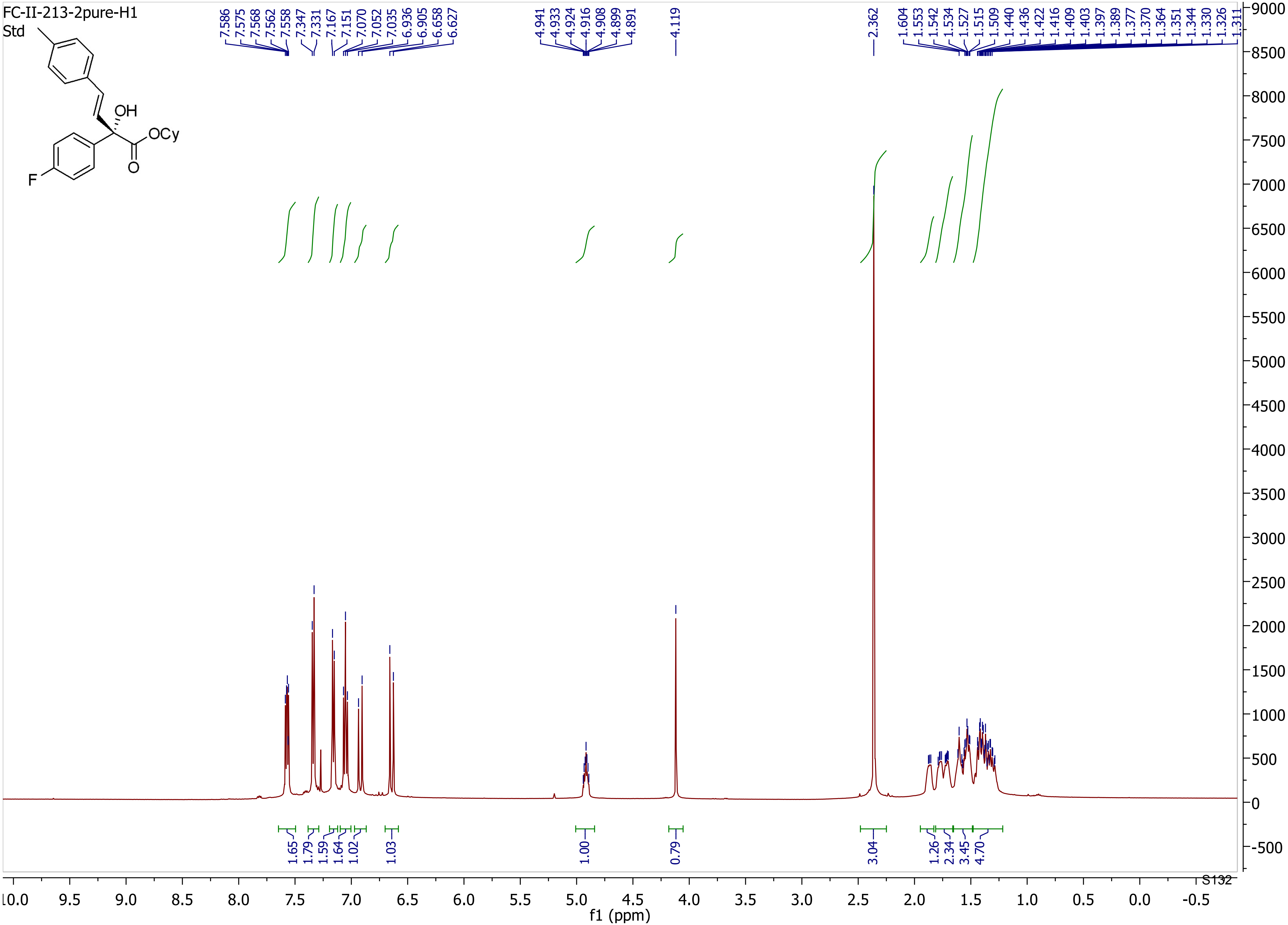

FC-II-254-3-H1-good
Std
MeO
OH
OCy
O
CF3
7.777
7.761
7.482
7.464
7.325
6.954
6.936
6.918
6.902
4.998
4.989
4.981
4.973
4.964
4.026
3.858
1.889
1.877
1.796
1.781
1.607
1.565
1.530
1.523
1.510
1.504
1.494
1.486
1.468
1.410
1.404
1.385
1.376
1.364
1.357
1.337
1.322
1.317
1.303
1.273
1.240
1.227
1.209
0.94
1.90
0.99
1.05
1.82
0.98
1.00
0.83
3.25
1.28
1.42
5.75
6.02
f1 (ppm)

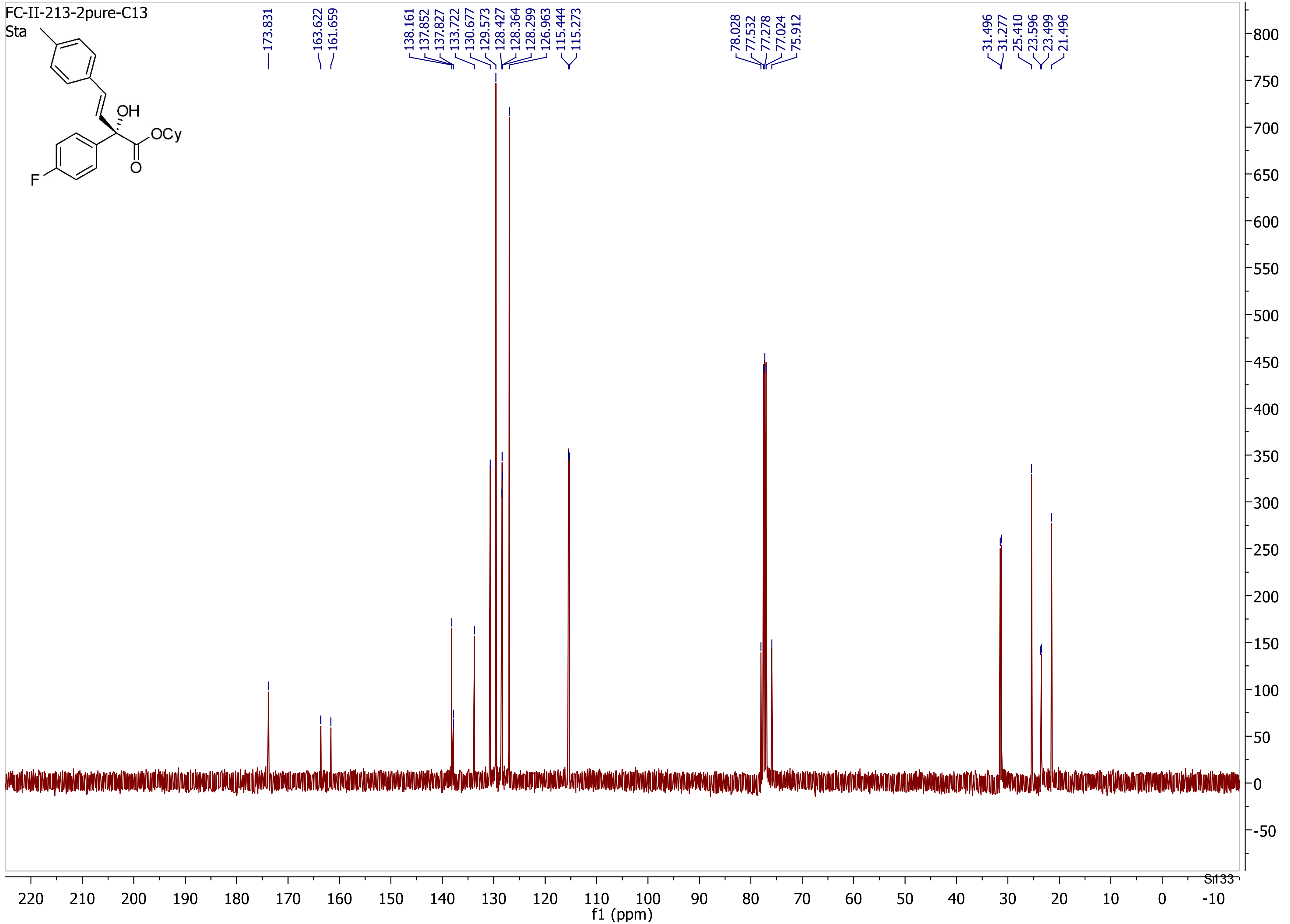

FC-II-254-3-C13-5
FC-II-254-3-C13
MeO
OH
OCy
O
CF3
173.419
159.785
140.747
134.368
131.774
131.044
128.533
128.405
125.888
123.713
113.938
81.218
77.488
77.233
76.979
75.616
55.549
31.519
31.191
25.388
23.637
23.472
800
750
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

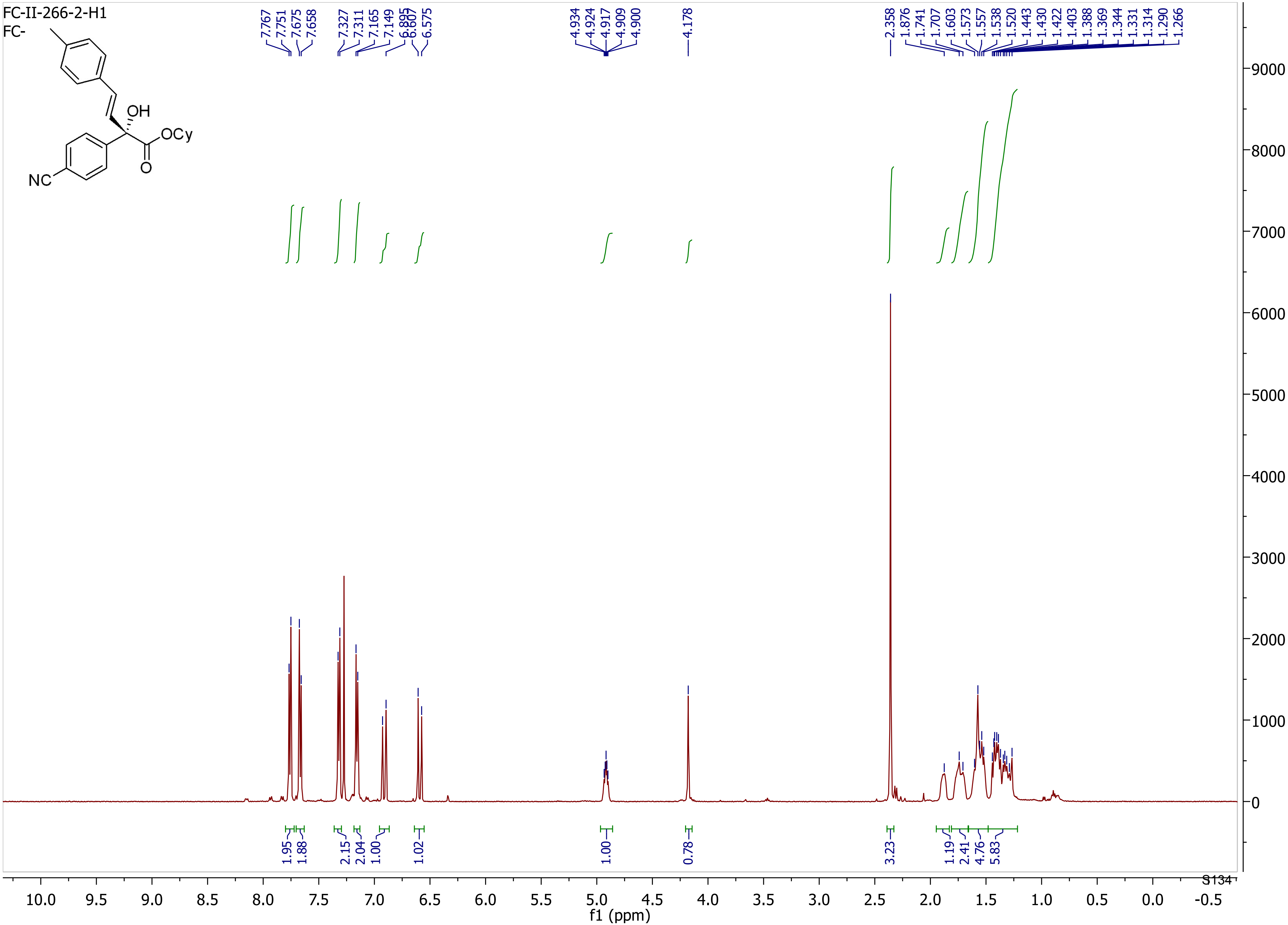

FC-II-213-1
FC-
OH
OCy
O
7.588
7.571
7.378
7.363
7.342
7.326
7.304
7.267
7.153
7.138
6.944
6.912
6.689
6.657
4.926
4.909
4.901
4.892
4.082
2.350
1.853
1.774
1.697
1.584
1.545
1.525
1.510
1.500
1.443
1.424
1.401
1.374
1.354
1.329
1.313
1.294
1.264
1.40
3.77
1.82
1.06
1.23
1.00
0.89
2.80
3.53
9.10
f1 (ppm)

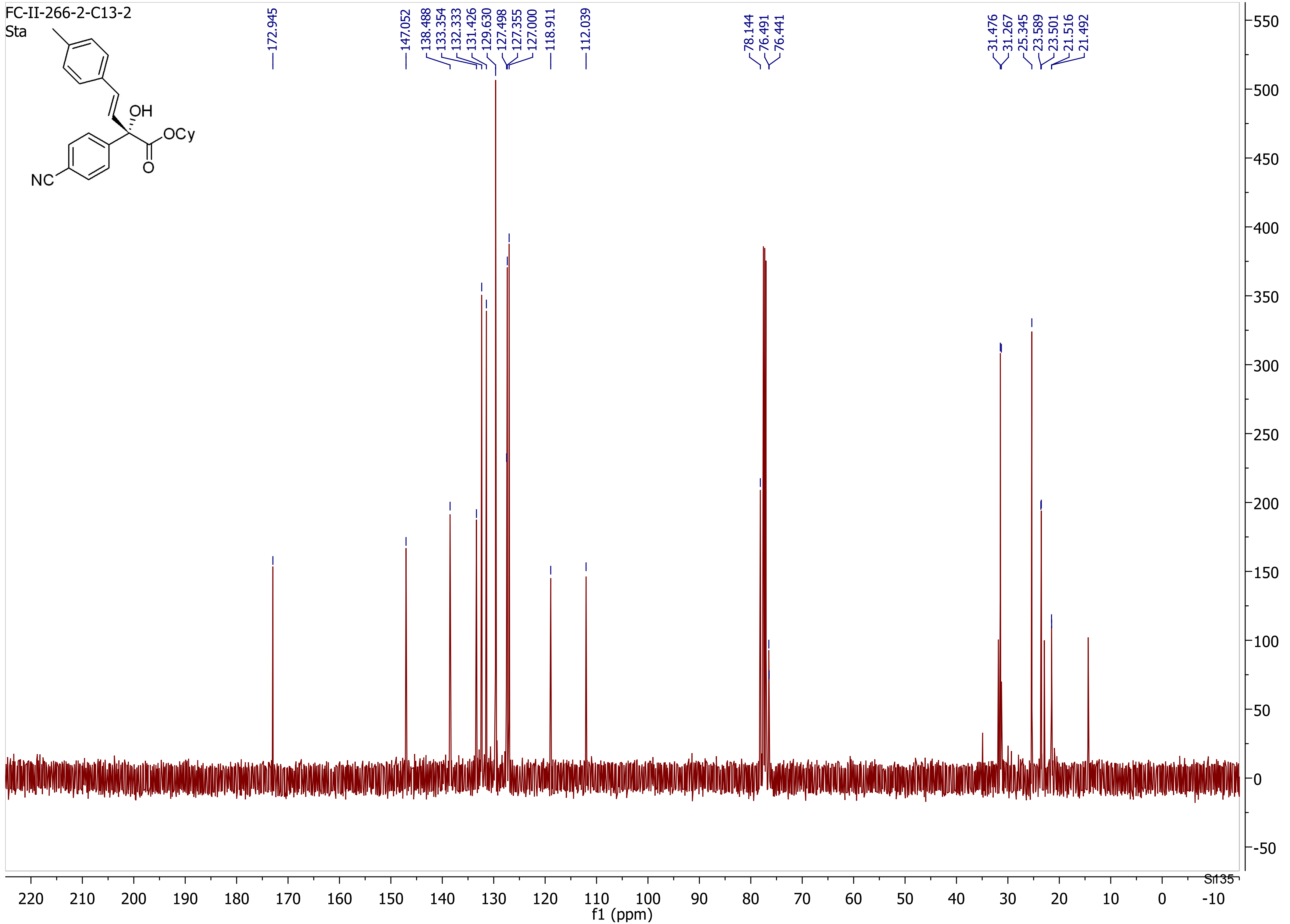
FC-II-213-1-C13
Sta

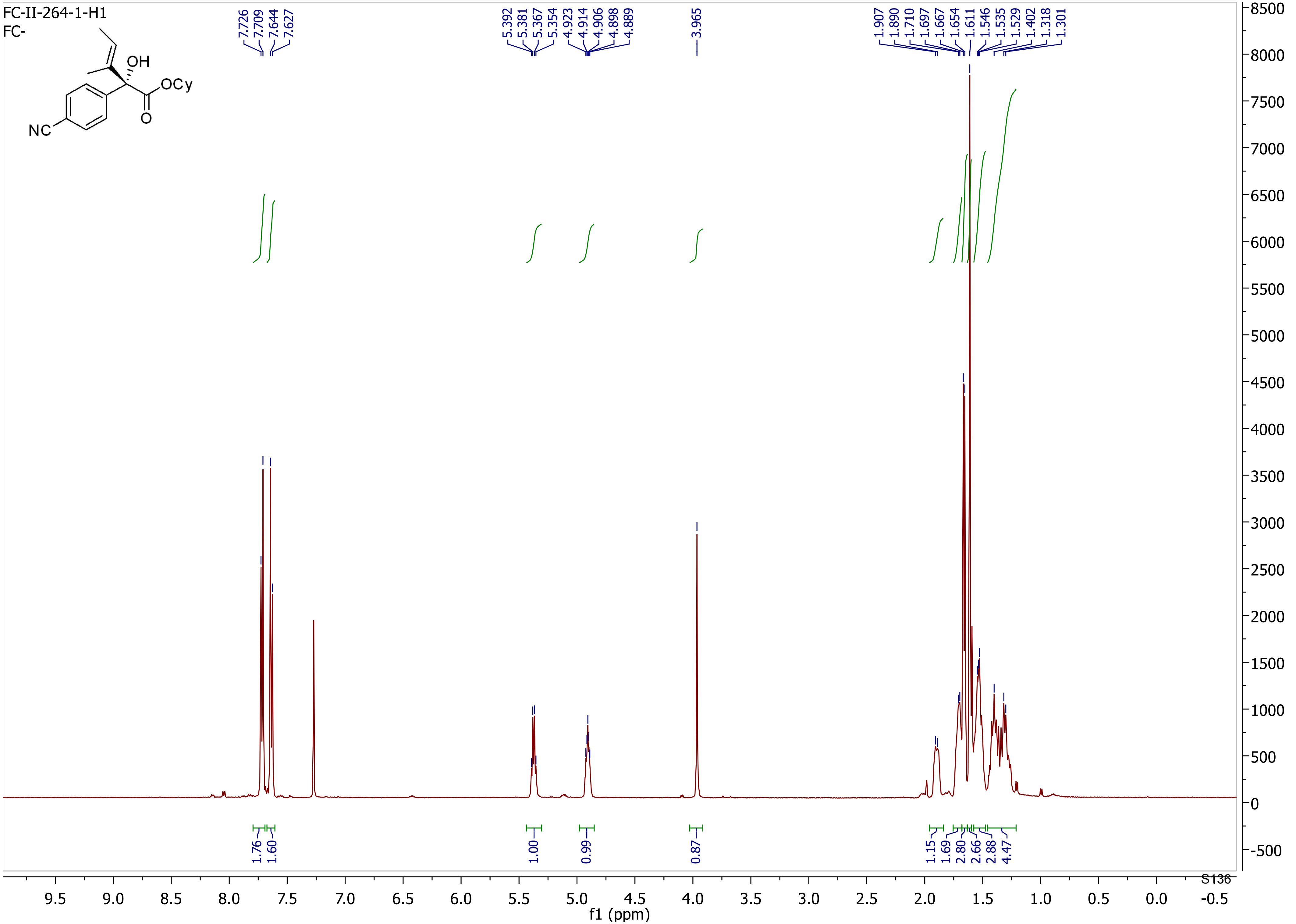


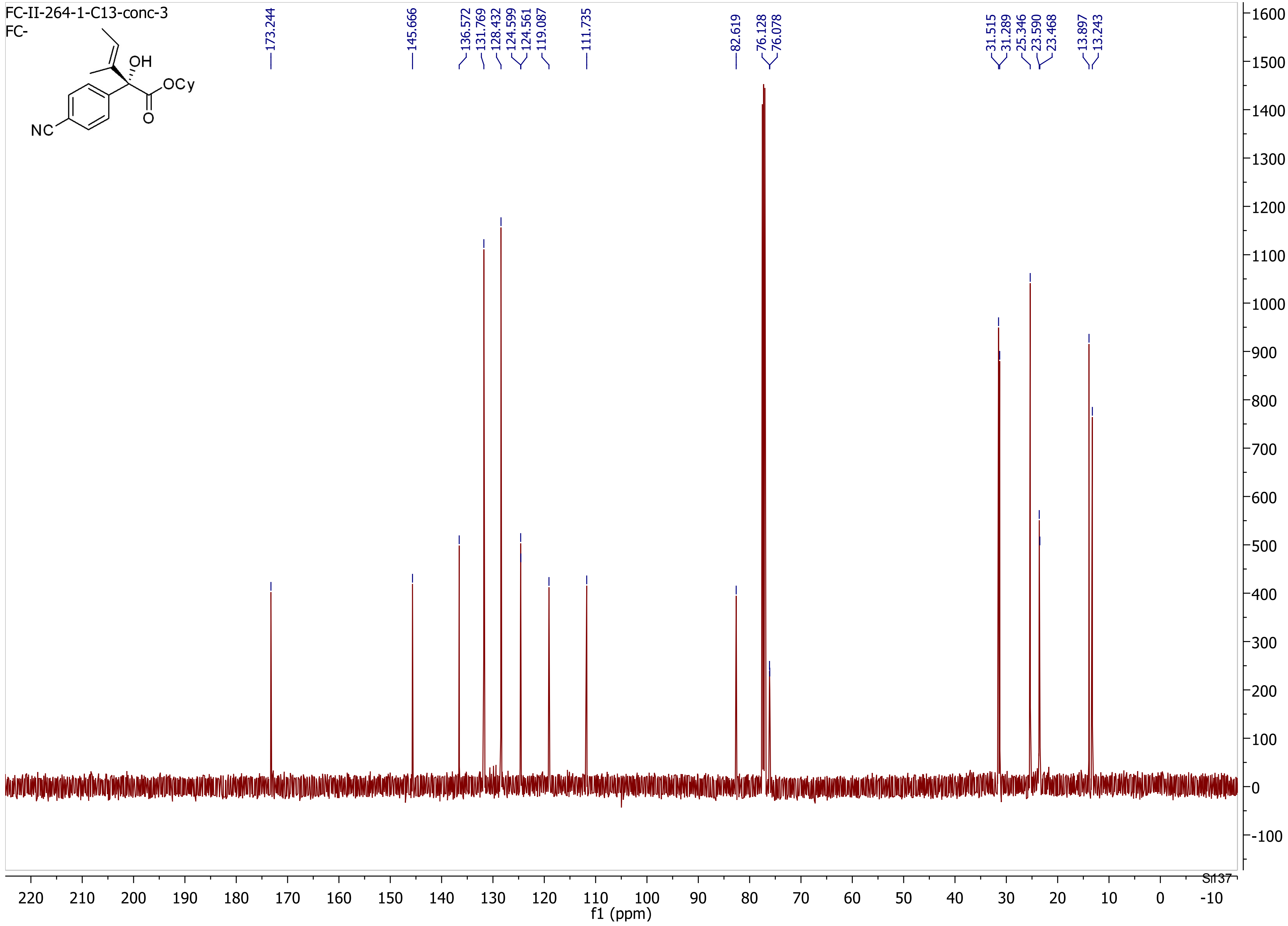

FC-II-213-2pure-H1
Std
OH
OCy
O
F
7.586
7.575
7.568
7.562
7.558
7.347
7.331
7.167
7.151
7.070
7.052
7.035
6.936
6.905
6.658
6.627
4.941
4.933
4.924
4.916
4.908
4.899
4.891
4.119
2.362
1.604
1.553
1.542
1.534
1.527
1.515
1.509
1.440
1.436
1.422
1.416
1.409
1.403
1.397
1.389
1.377
1.370
1.364
1.351
1.344
1.330
1.326
1.311
1.65
1.79
1.59
1.64
1.02
1.03
1.00
0.79
3.04
1.26
2.34
3.45
4.70
f1 (ppm)

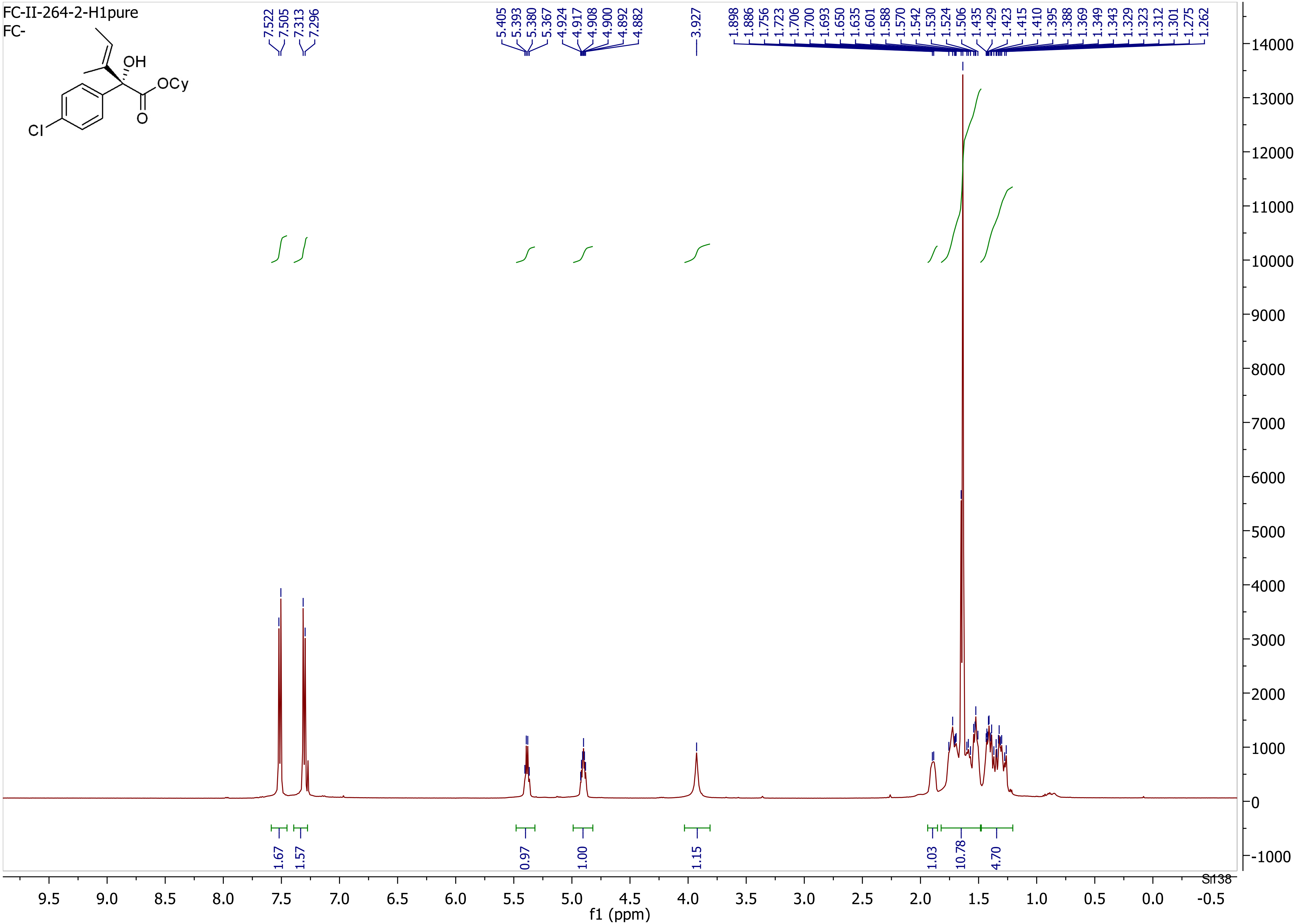

FC-II-213-2pure-C13
Sta
OH
OCy
O
F
173.831
163.622
161.659
138.161
137.852
137.827
133.722
130.677
129.573
128.427
128.364
128.299
126.963
115.444
115.273
78.028
77.532
77.278
77.024
75.912
31.496
31.277
25.410
23.596
23.499
21.496
f1 (ppm)

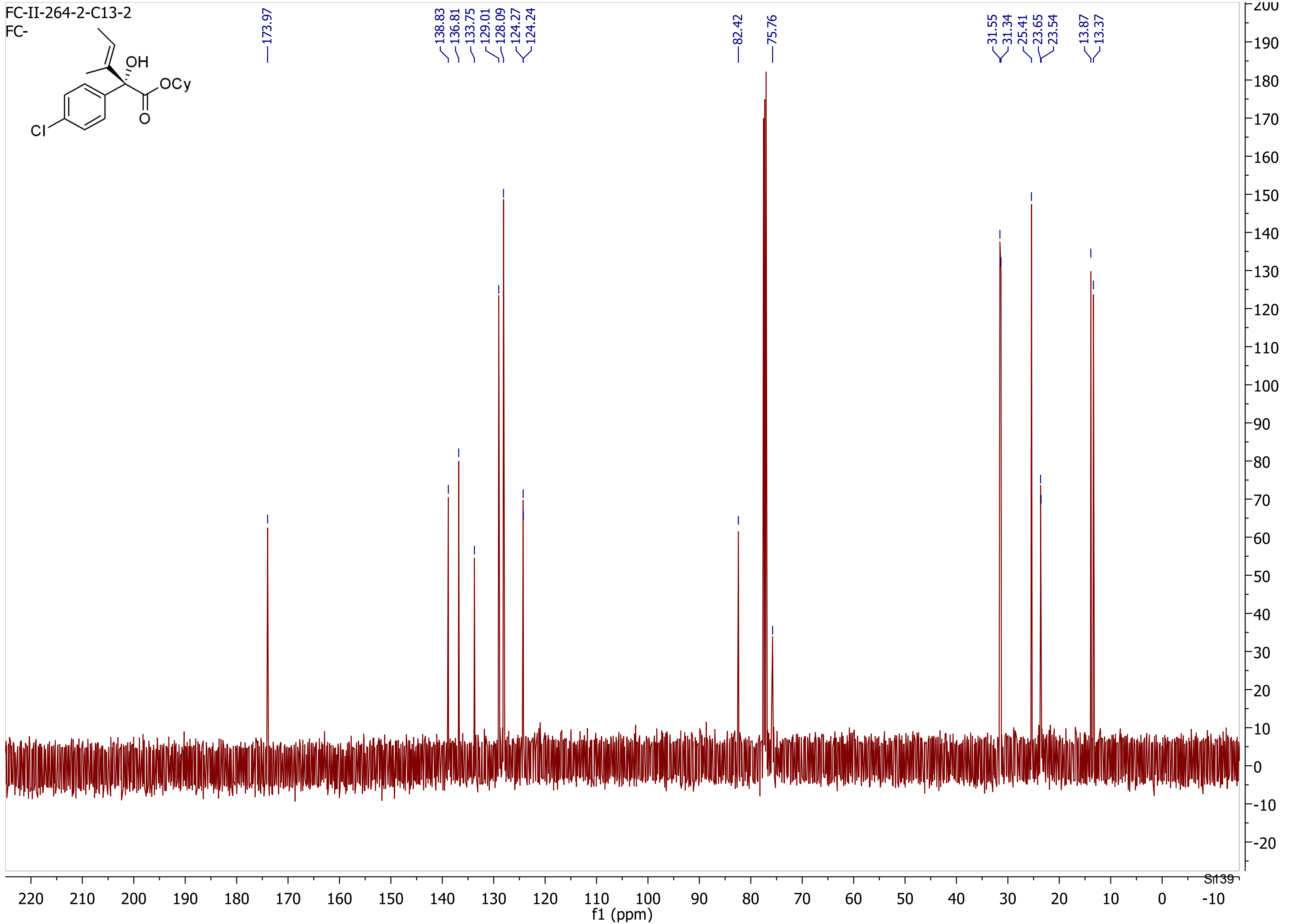

FC-II-266-2-H1
FC-
OH
OCy
O
NC
7.767
7.751
7.675
7.658
7.327
7.311
7.165
7.149
6.895
6.607
6.575
4.934
4.924
4.917
4.909
4.900
4.178
2.358
1.876
1.741
1.707
1.603
1.573
1.557
1.538
1.520
1.443
1.430
1.422
1.403
1.388
1.369
1.344
1.331
1.314
1.290
1.266
1.95
1.88
2.15
2.04
1.00
1.02
1.00
0.78
3.23
1.19
2.41
4.76
5.83
f1 (ppm)

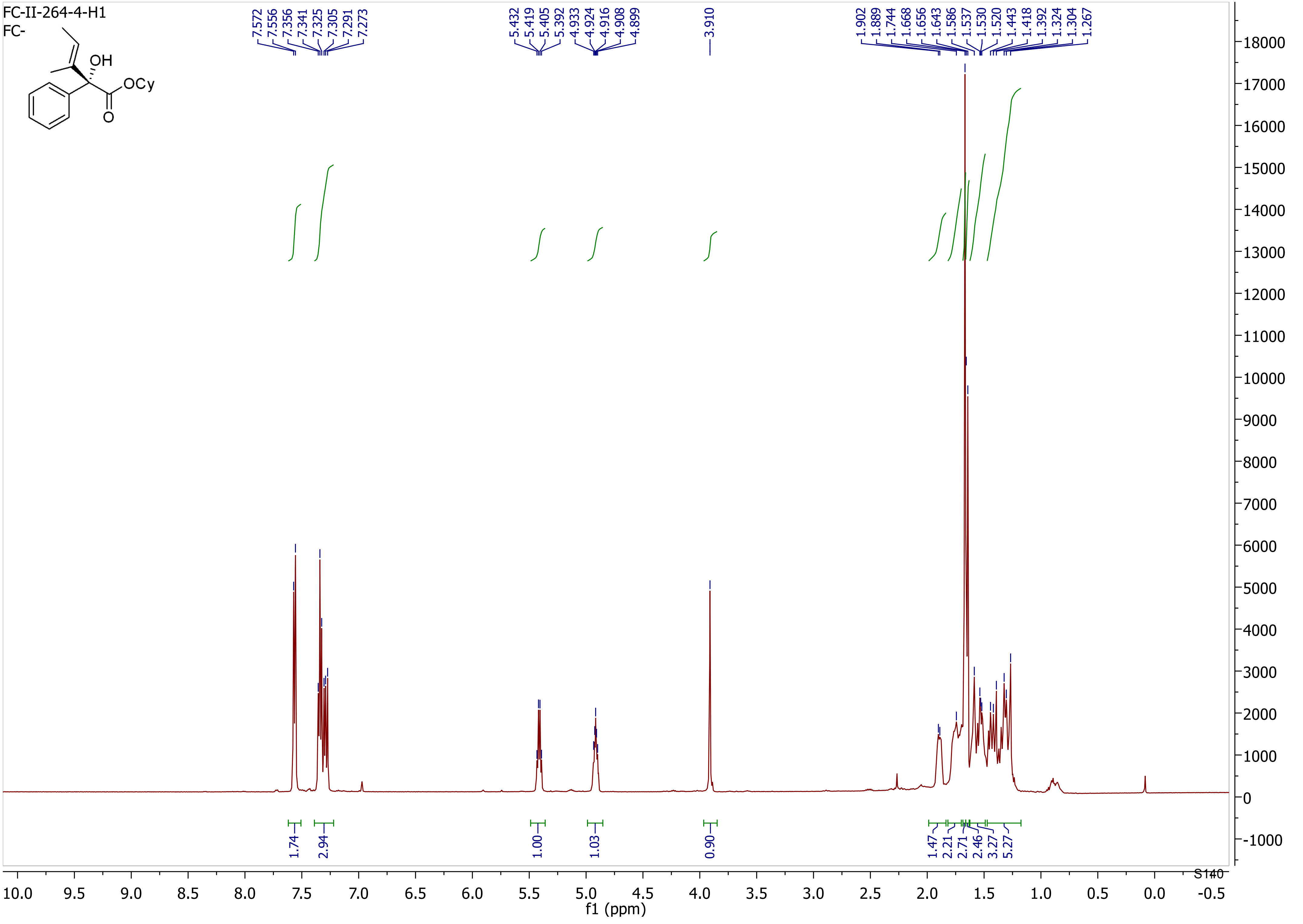

FC-II-266-2-C13-2
Sta
OH
OCy
O
NC
172.945
147.052
138.488
133.354
132.333
131.426
129.630
127.498
127.355
127.000
118.911
112.039
78.144
76.491
76.441
31.476
31.267
25.345
23.589
23.501
21.516
21.492
550
500
450
400
350
300
250
200
150
100
50
0
-50
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

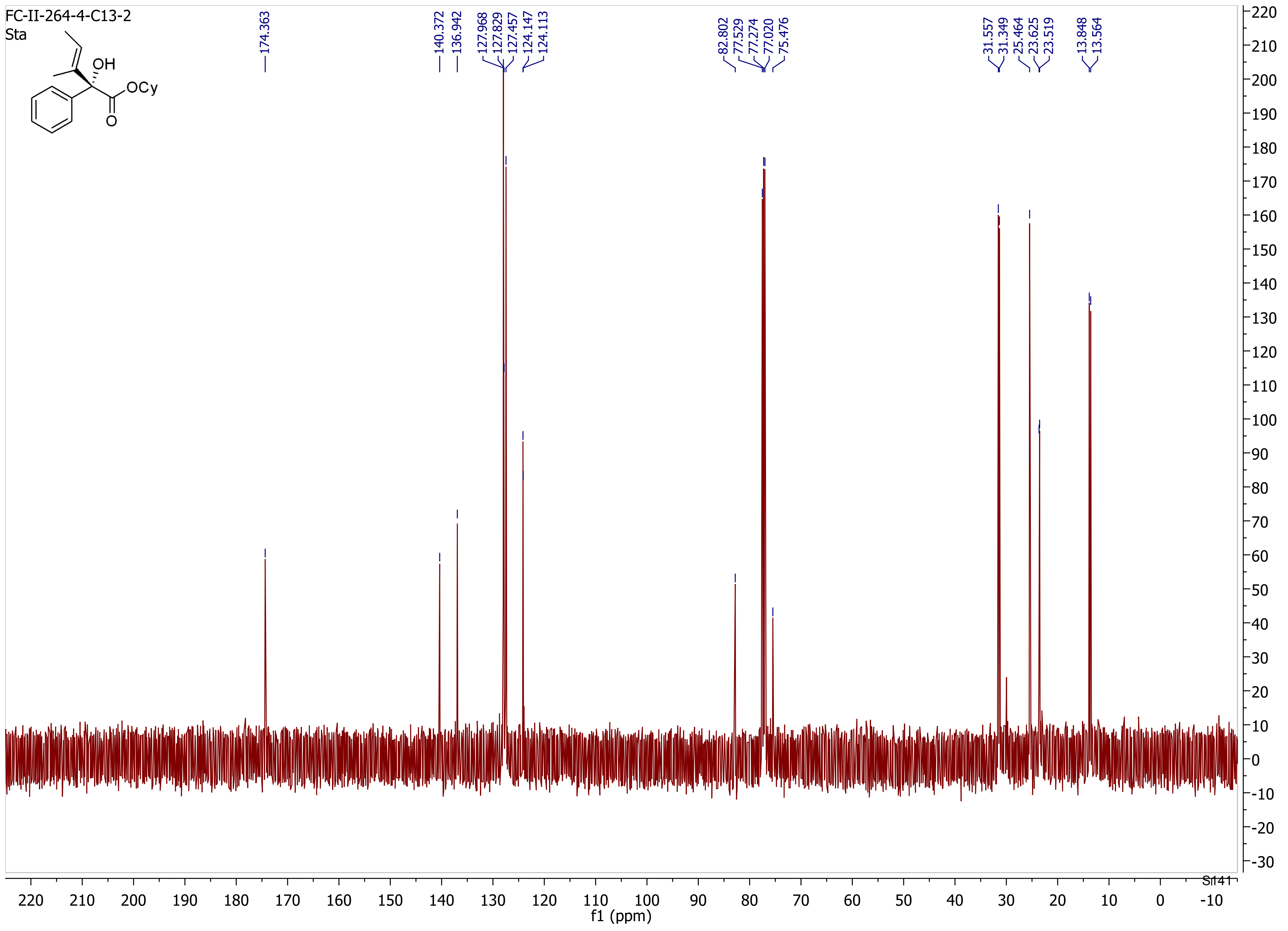

FC-II-264-1-H1
FC-
OH
OCy
NC
O
7.726
7.709
7.644
7.627
5.392
5.381
5.367
5.354
4.923
4.914
4.906
4.898
4.889
3.965
1.907
1.890
1.710
1.697
1.667
1.654
1.611
1.546
1.535
1.529
1.402
1.318
1.301
1.76
1.60
1.00
0.99
0.87
1.15
1.69
2.80
2.66
2.88
4.47
f1 (ppm)

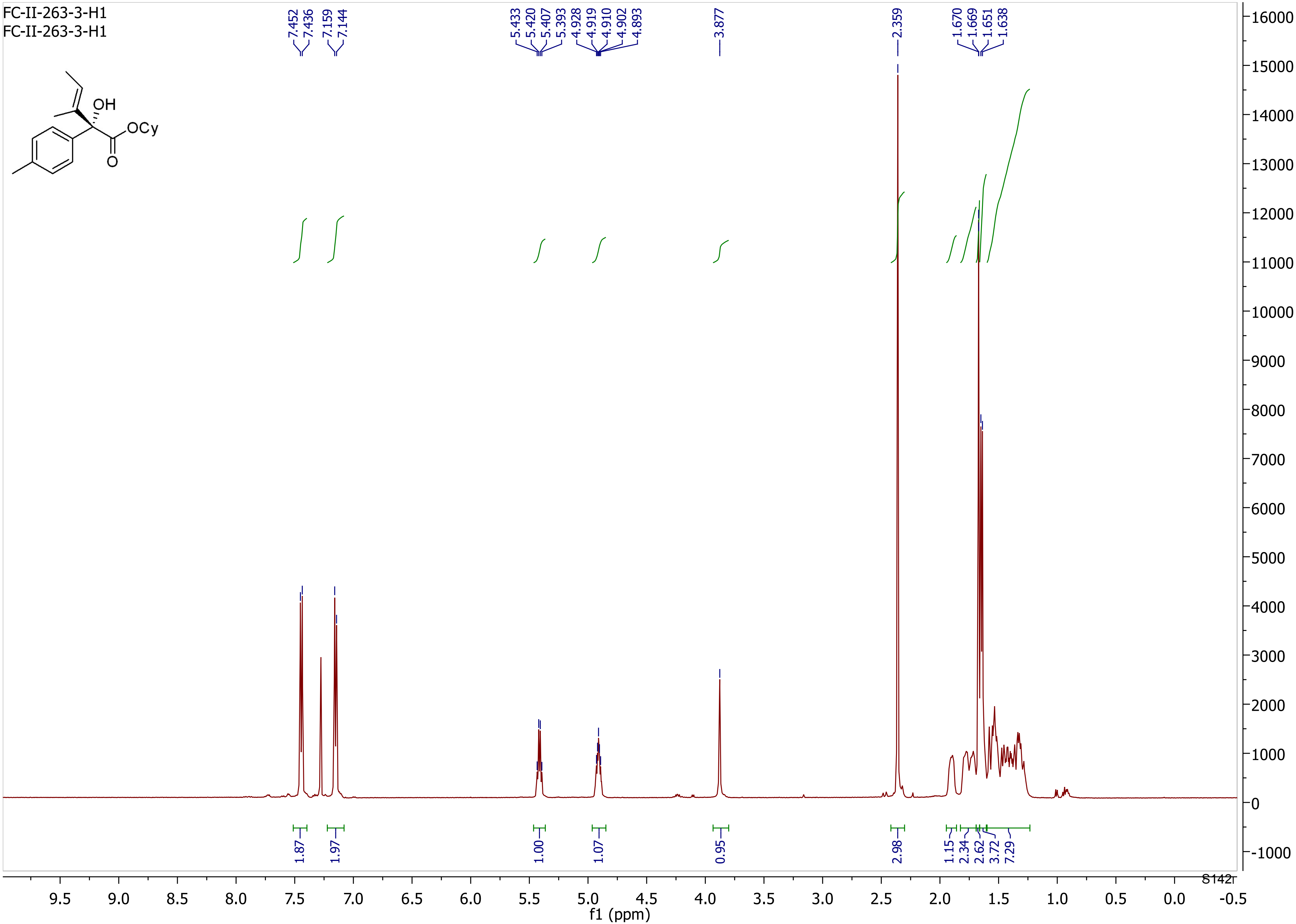

FC-II-264-1-C13-conc-3
FC-
OH
OCy
NC
O
173.244
145.666
136.572
131.769
128.432
124.599
124.561
119.087
111.735
82.619
76.128
76.078
31.515
31.289
25.346
23.590
23.468
13.897
13.243
f1 (ppm)


FC-II-264-2-H1pure
FC-
OH
OCy
O
Cl
7.522
7.505
7.313
7.296
5.405
5.393
5.380
5.367
4.924
4.917
4.908
4.900
4.892
4.882
3.927
1.898
1.886
1.756
1.723
1.706
1.700
1.693
1.650
1.635
1.601
1.588
1.570
1.542
1.530
1.524
1.506
1.435
1.429
1.423
1.415
1.410
1.395
1.388
1.369
1.349
1.343
1.329
1.323
1.312
1.301
1.275
1.262
1.67
1.57
0.97
1.00
1.15
1.03
10.78
4.70
f1 (ppm)


FC-II-264-2-C13-2
FC-
OH
OCy
O
Cl
173.97
138.83
136.81
133.75
129.01
128.09
124.27
124.24
82.42
75.76
31.55
31.34
25.41
23.65
23.54
13.87
13.37
f1 (ppm)


FC-II-264-4-H1
FC-
OH
OCy
O
7.572
7.556
7.356
7.341
7.325
7.305
7.291
7.273
5.432
5.419
5.405
5.392
4.933
4.924
4.916
4.908
4.899
3.910
1.902
1.889
1.744
1.668
1.656
1.643
1.586
1.537
1.530
1.520
1.443
1.418
1.392
1.324
1.304
1.267
1.74
2.94
1.00
1.03
0.90
1.47
2.21
2.71
2.46
3.27
5.27
f1 (ppm)


FC-II-264-4-C13-2
Sta
OH
OCy
O
174.363
140.372
136.942
127.968
127.829
127.457
124.147
124.113
82.802
77.529
77.274
77.020
75.476
31.557
31.349
25.464
23.625
23.519
13.848
13.564
f1 (ppm)


FC-II-263-3-H1
FC-II-263-3-H1
OH
OCy
O
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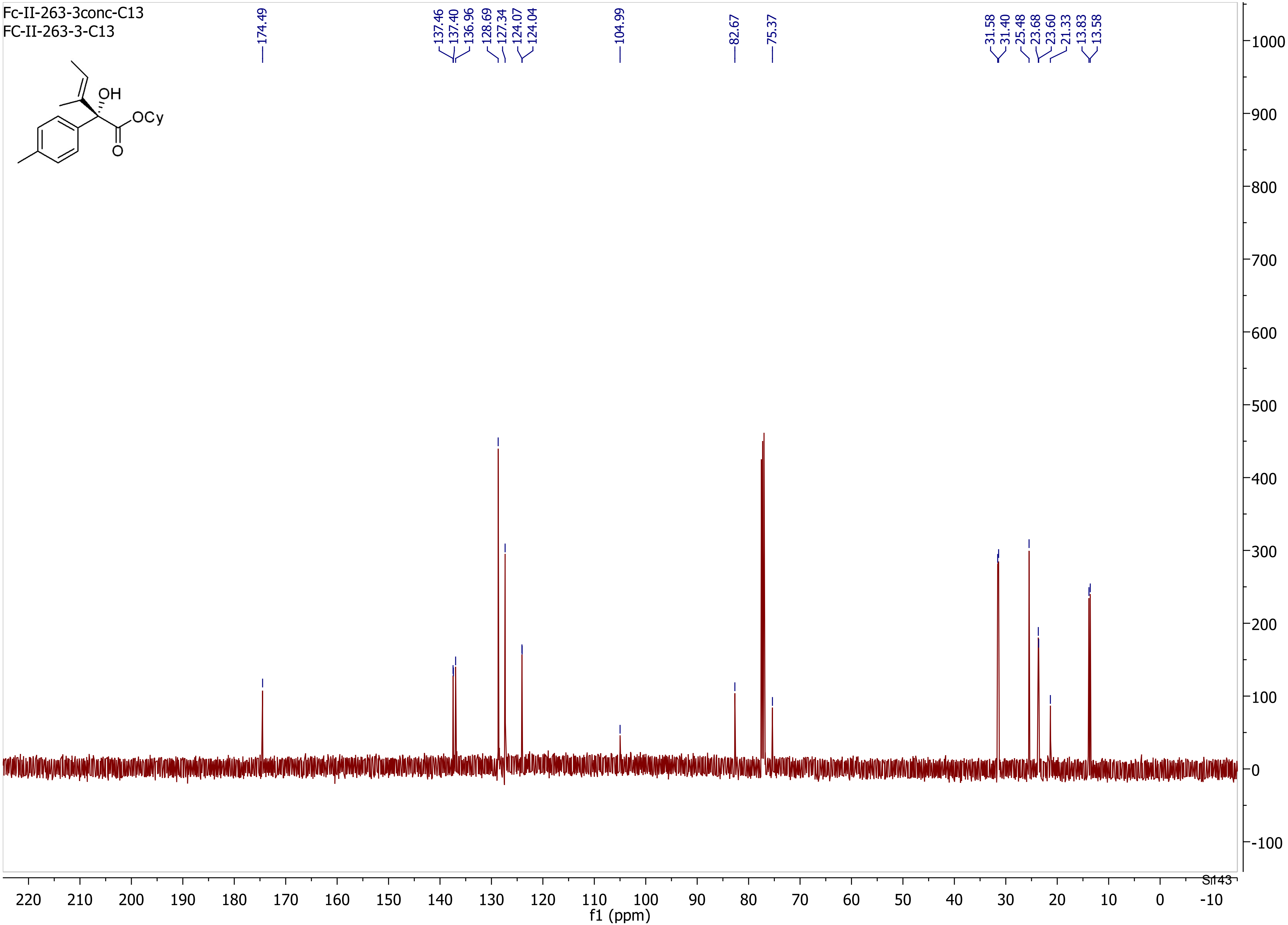
7.436
7.159
7.144
5.433
5.420
5.407
5.393
4.928
4.919
4.910
4.902
4.893
3.877
2.359
1.670
1.669
1.651
1.638
1.87
1.97
1.00
1.07
0.95
2.98
1.15
2.34
2.62
3.72
7.29
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000


Fc-II-263-3conc-C13
FC-II-263-3-C13
OH
OCy
O
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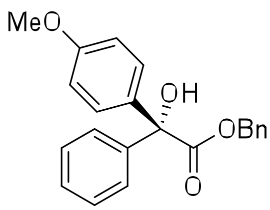
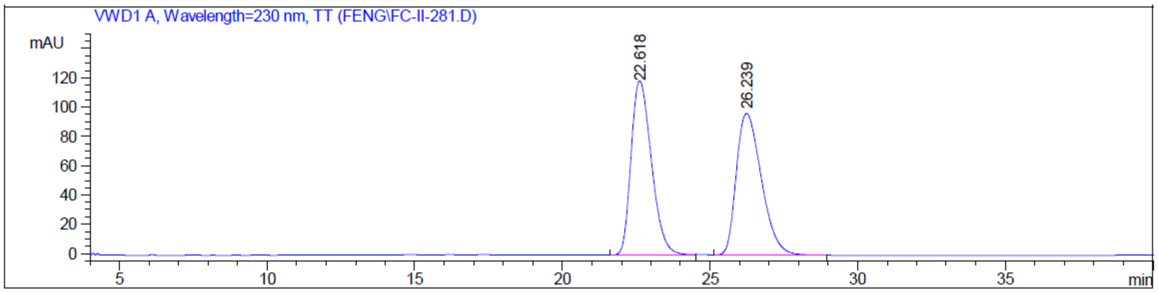
137.46
137.40
136.96
128.69
127.34
124.07
124.04
104.99
82.67
75.37
31.58
31.40
25.48
23.68
23.60
21.33
13.83
13.58
f1 (ppm)

Table 1, entry 1

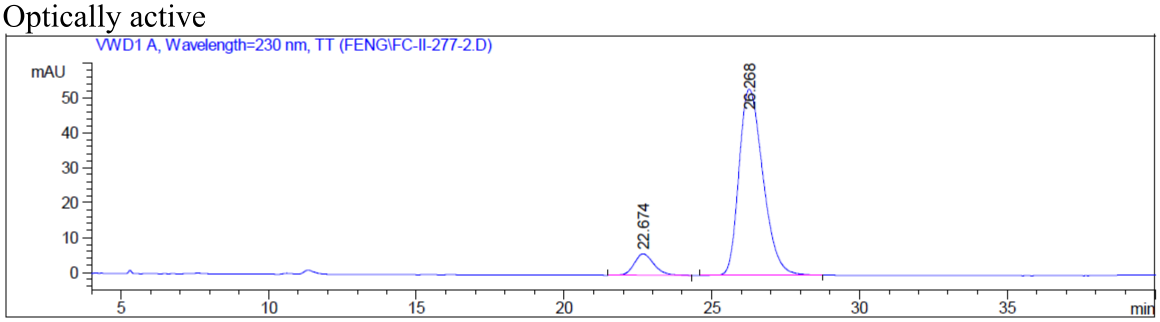


Racemic



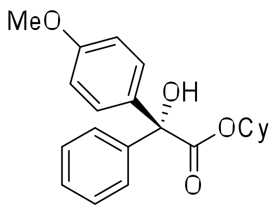
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	22.618	VV	0.7544	5730.85498	118.92757	49.9602
2	26.239	VB	0.9422	5739.97705	96.79990	50.0398

Optically active

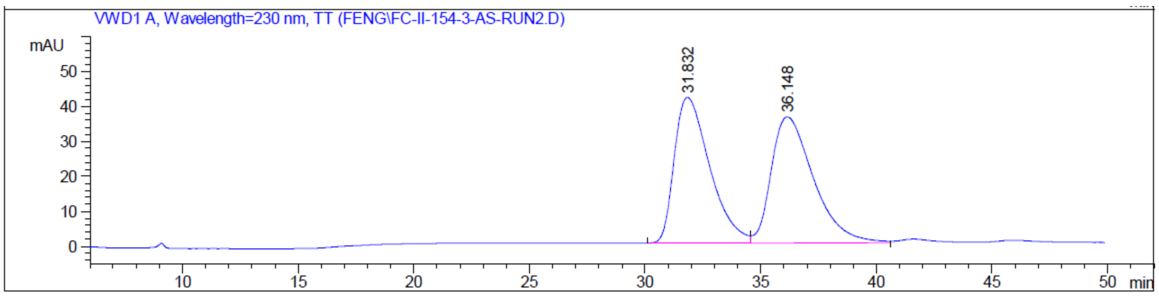


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	22.674	BB	0.7110	283.74319	6.10647	8.6141
2	26.268	BB	0.8748	3010.17773	53.26956	91.3859

Table 1, entry 9

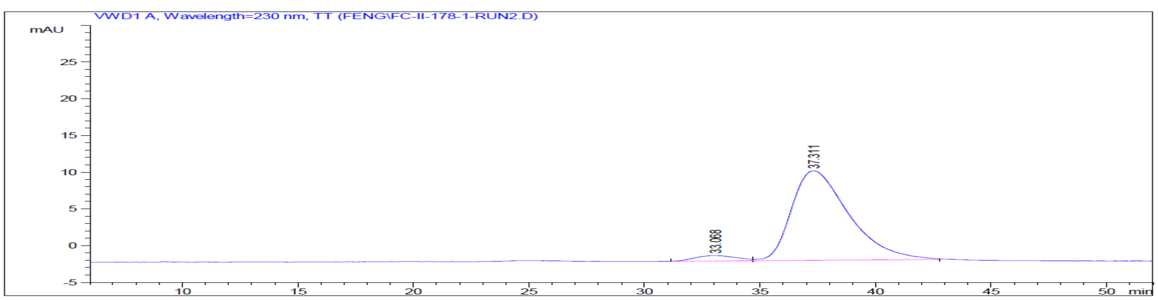


Racemic



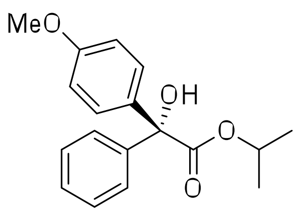
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	31.832	BV	1.6380	4432.55420	41.63805	49.2045
2	36.148	VB	1.9606	4575.87451	35.99717	50.7955

Optically active

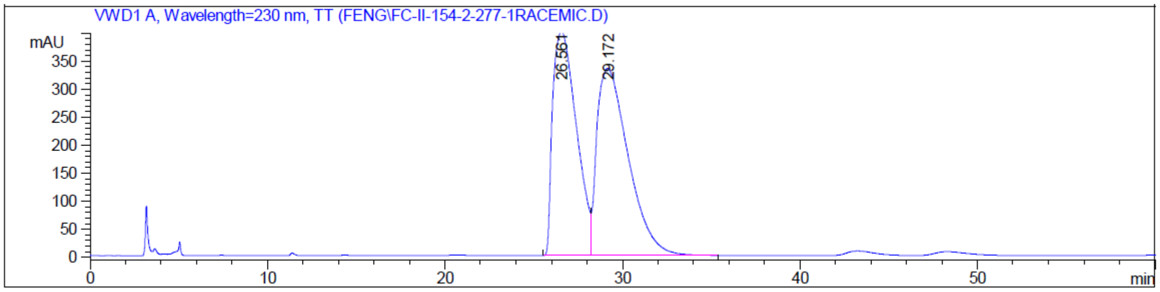


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	33.068	BV	1.4978	91.75425	7.43590e-1	4.1753
2	37.311	VB	2.5201	2105.78174	12.20189	95.8247

Table 1, entry 10

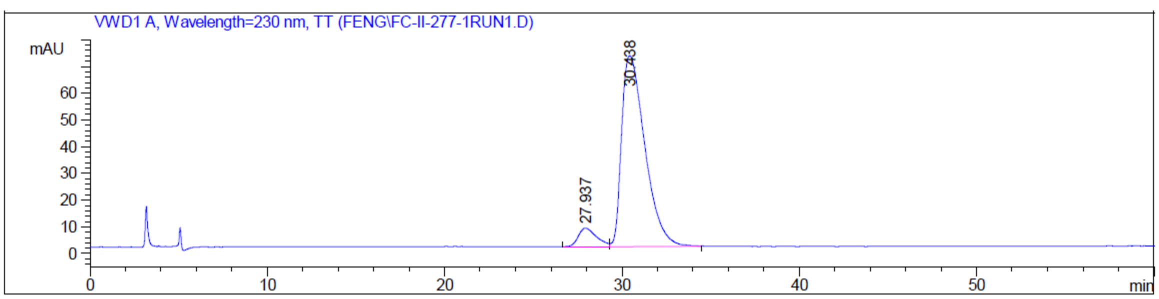


Racemic



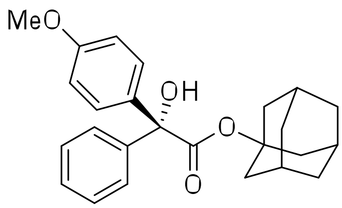
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	26.561	VV	1.4712	3.70548e4	399.91202	47.3778
2	29.172	VB	1.8870	4.11566e4	333.78186	52.6222

Optically active

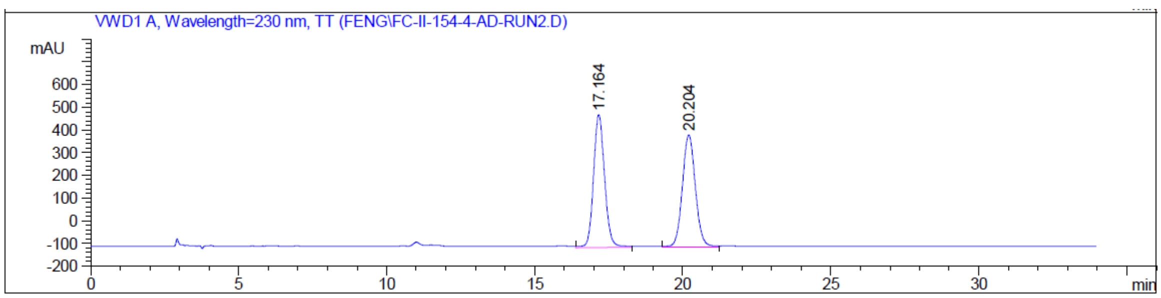


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	27.937	VV	1.0567	494.18161	6.94956	7.0651
2	30.438	VB	1.4110	6500.54932	71.43871	92.9349

Table 1, entry 11

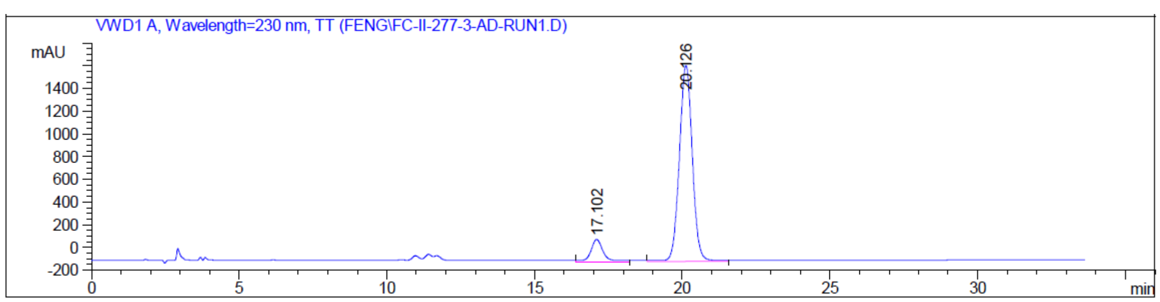


Racemic



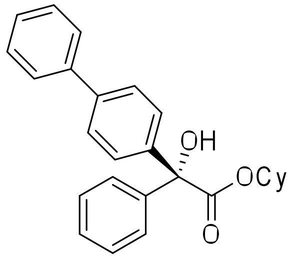
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.164	VV	0.4041	1.53152e4	585.56006	50.0675
2	20.204	VV	0.4761	1.52739e4	495.47067	49.9325

Optically active

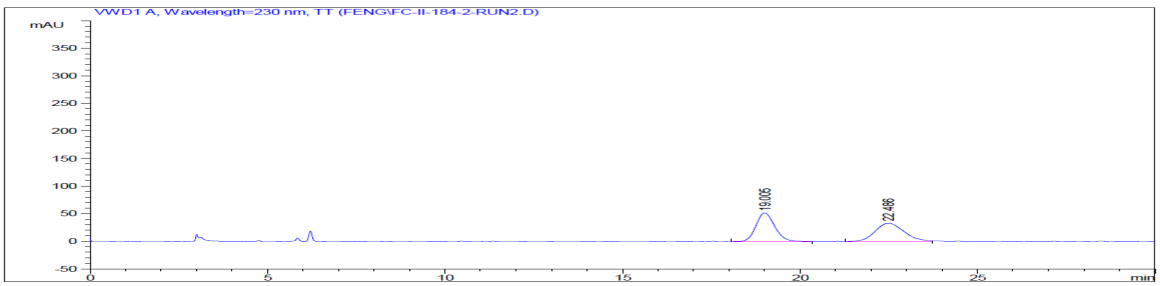


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.102	VV	0.4427	5873.42529	196.07744	9.9746
2	20.126	VV	0.4805	5.30104e4	1726.79480	90.0254

Table 2, entry 2.

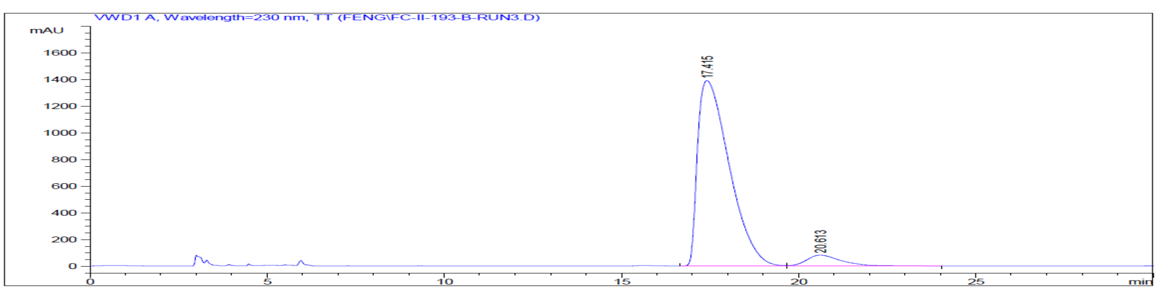


Racemic



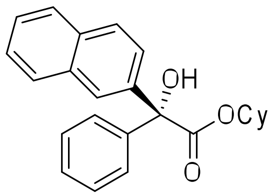
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.005	VV	0.5811	1984.52844	52.46295	50.2483
2	22.486	VV	0.9240	1964.91589	33.74318	49.7517

Optically active

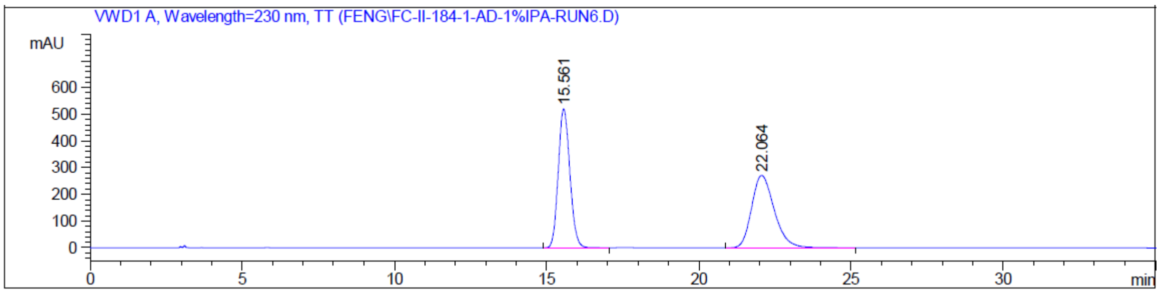


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.415	VV	0.9693	8.63712e4	1390.42664	93.8813
2	20.613	VB	1.0208	5629.19775	82.14567	6.1187

Table 2, entry 3

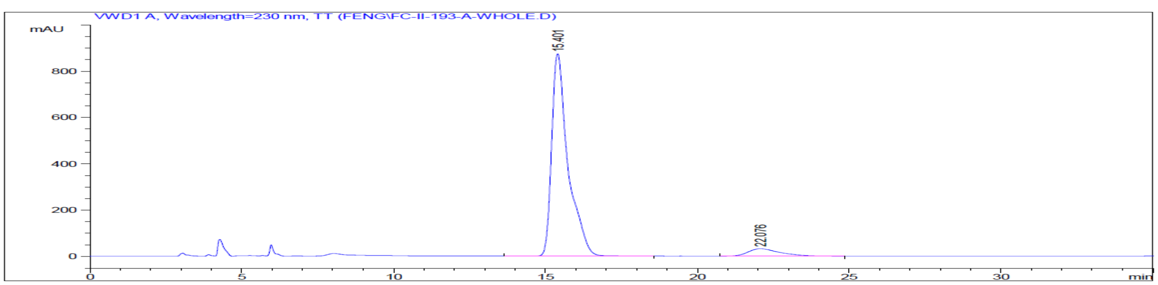


Racemic



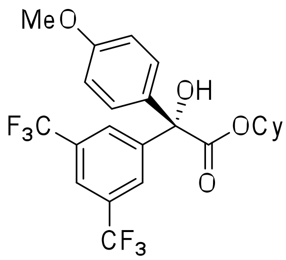
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.561	BV	0.4153	1.38570e4	520.75702	49.6477
2	22.064	BB	0.8025	1.40536e4	271.21817	50.3523

Optically active

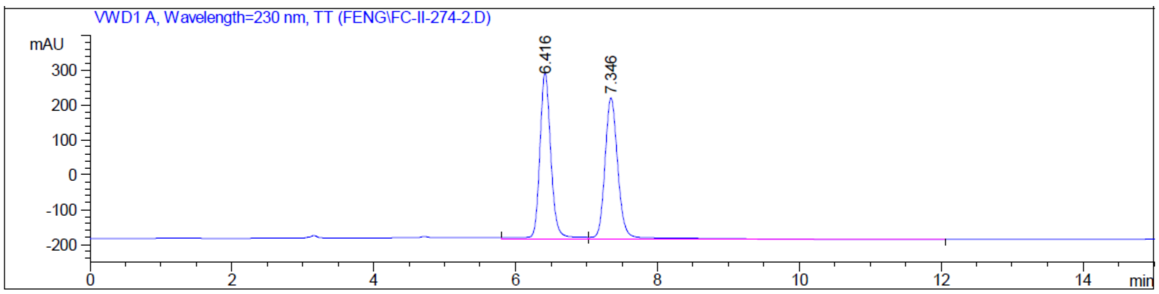


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.401	VB	0.5487	3.26932e4	873.63202	93.4287
2	22.076	VB	1.0634	2299.49219	31.96926	6.5713

Table 2, entry 4.

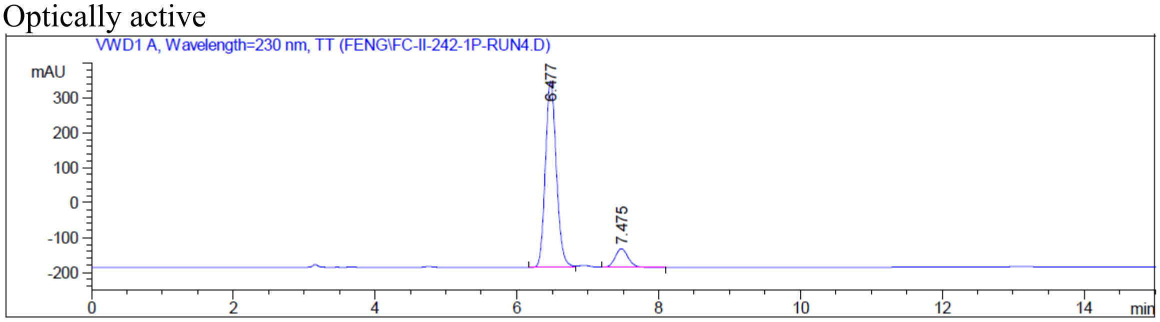


Racemic



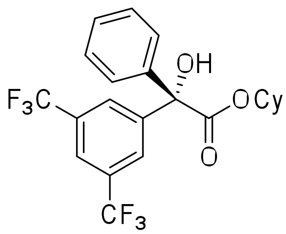
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.416	VV	0.1695	5244.65283	475.32874	49.3269
2	7.346	VB	0.2078	5387.79004	404.64053	50.6731

Optically active

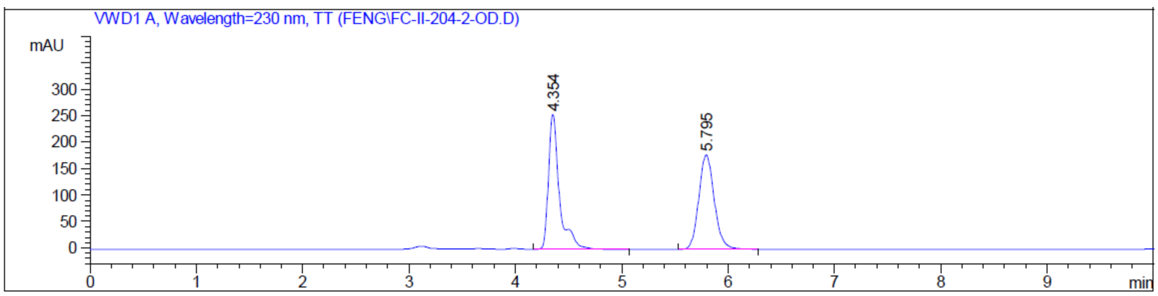


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.477	VV	0.1666	5730.73389	531.45807	89.4618
2	7.475	VB	0.1942	675.05652	53.33956	10.5382

Table 2, entry 5

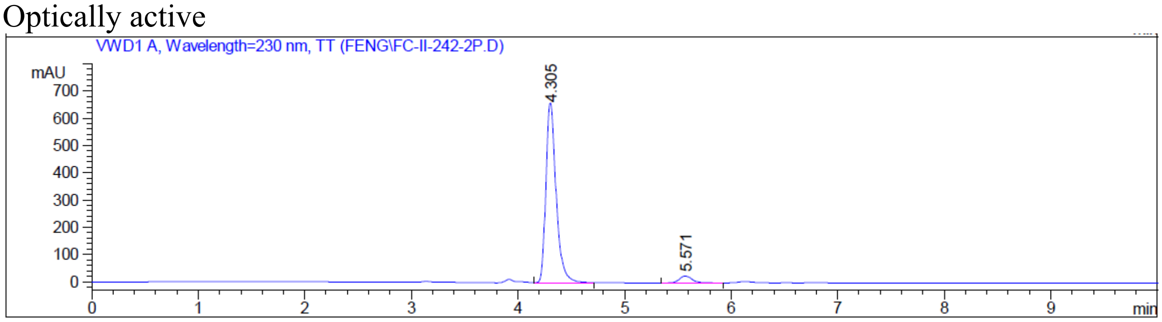


Racemic



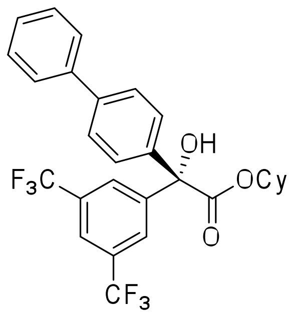
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.354	VB	0.1141	1835.57813	251.96439	50.3572
2	5.795	VV	0.1594	1809.53894	177.92149	49.6428

Optically active

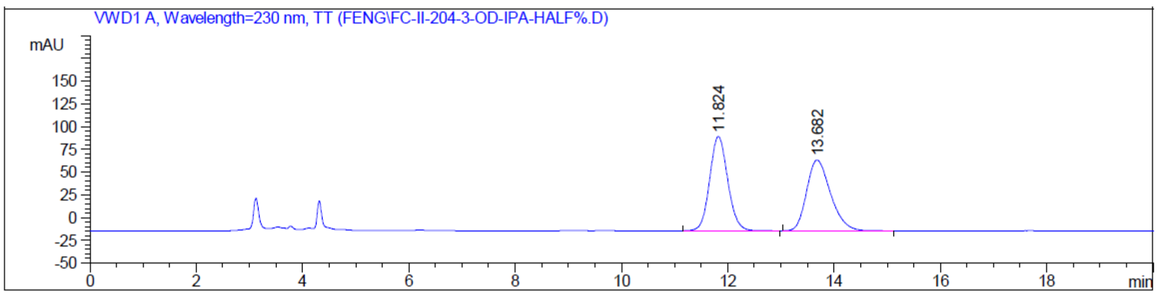


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.305	VV	0.1018	4408.07275	654.70471	95.4170
2	5.571	VV	0.1325	211.72343	25.32659	4.5830

Table 2, entry 6

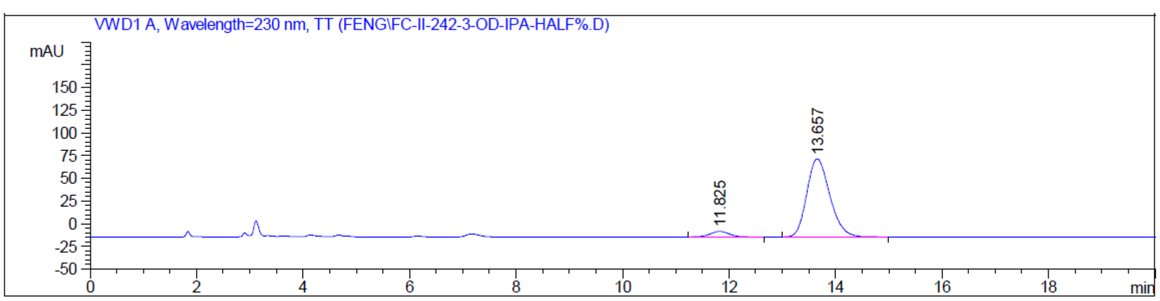


Racemic



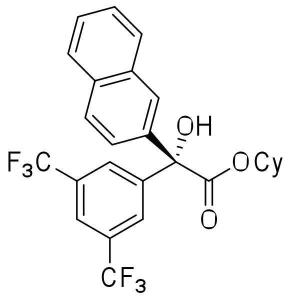
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.824	BB	0.3691	2452.93311	103.71603	50.2369
2	13.682	BB	0.4810	2429.79614	77.76572	49.7631

Optically active

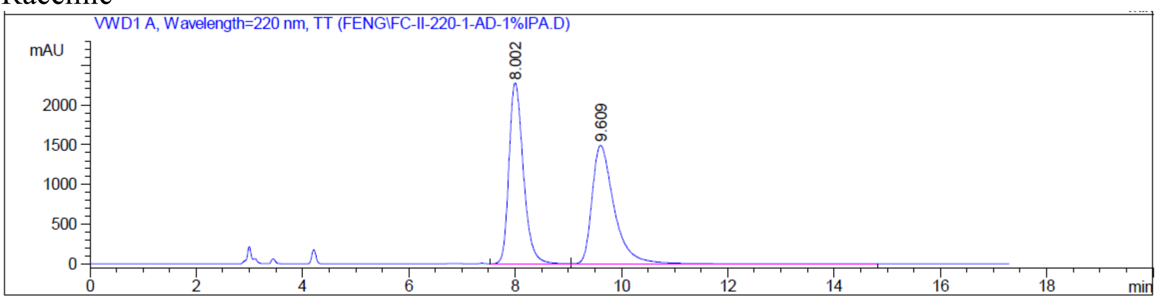


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.825	BB	0.3770	147.14075	6.04920	5.4098
2	13.657	BB	0.4658	2572.74951	85.92315	94.5902

Table 2, entry 7

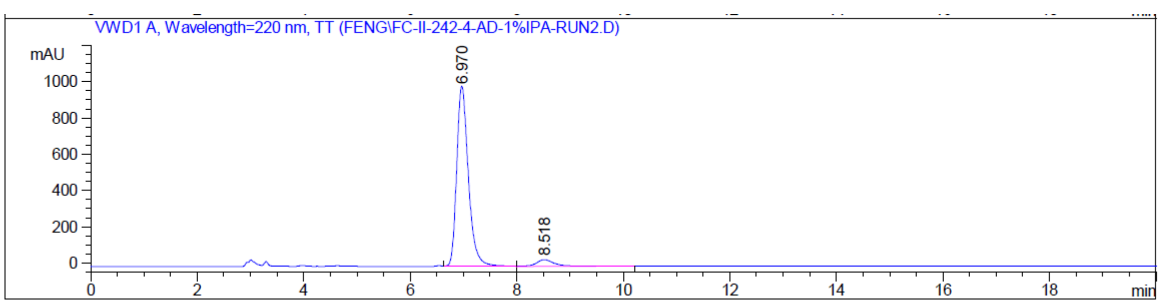


Racemic



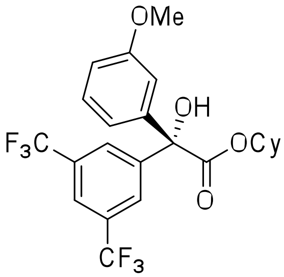
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.002	VV	0.2966	4.30617e4	2274.64526	49.7221
2	9.609	VB	0.4401	4.35429e4	1489.53442	50.2779

Optically active

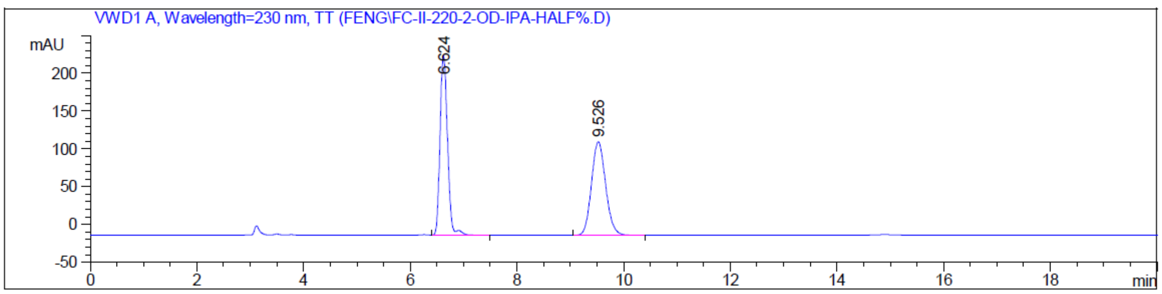


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.969	VV	0.2332	1.51337e4	992.70844	94.8759
2	8.518	VB	0.3402	817.35376	36.05864	5.1241

Table 2, entry 8

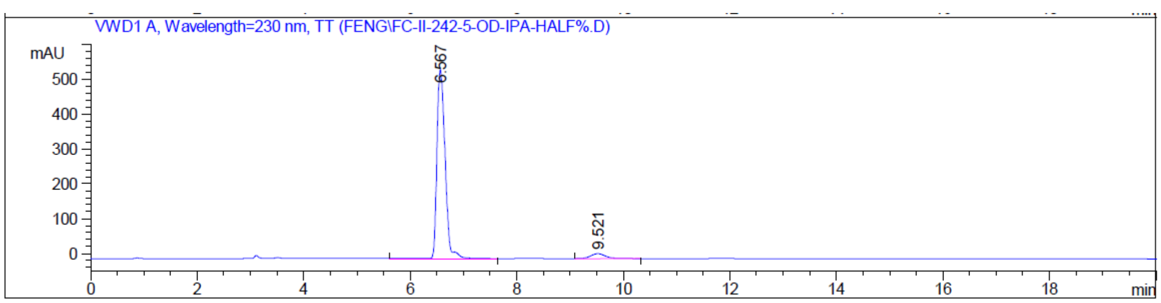


Racemic



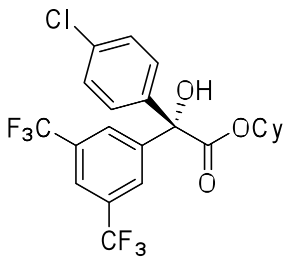
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.624	VB	0.1554	2329.90356	237.22830	50.4177
2	9.526	BB	0.2858	2291.29858	123.82945	49.5823

Optically active

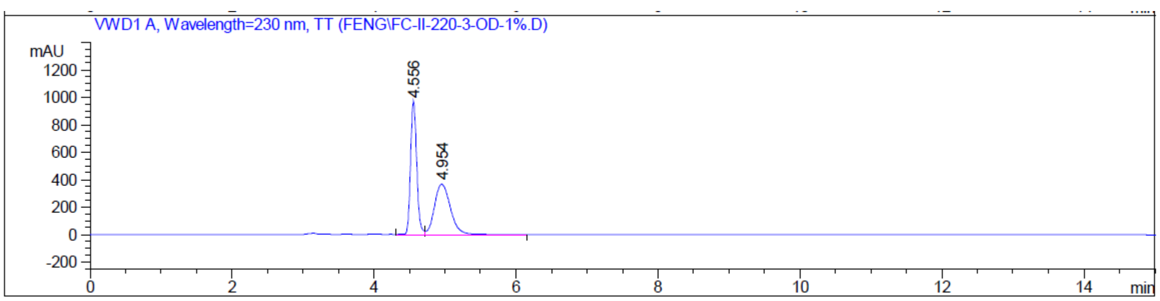


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.567	VB	0.1708	5744.64404	539.49164	95.4506
2	9.521	BB	0.3038	273.80377	14.00332	4.5494

Table 2, entry 9

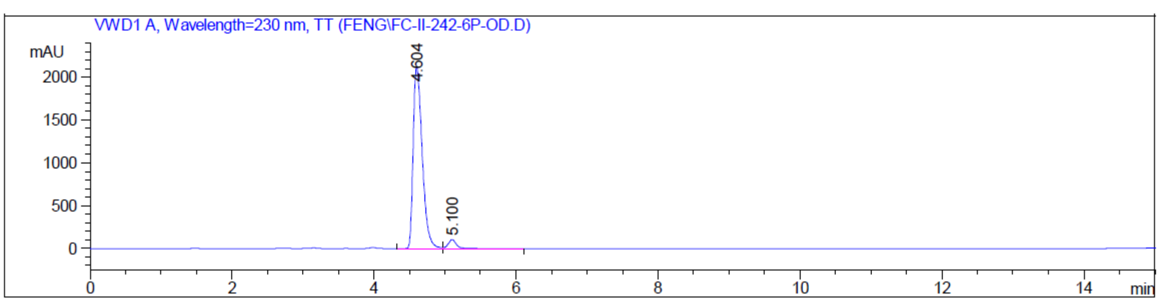


Racemic



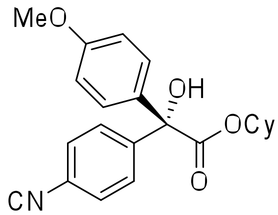
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.556	VV	0.0943	5844.78076	960.37909	50.7149
2	4.954	VV	0.2453	5679.99609	365.63089	49.2851

Optically active

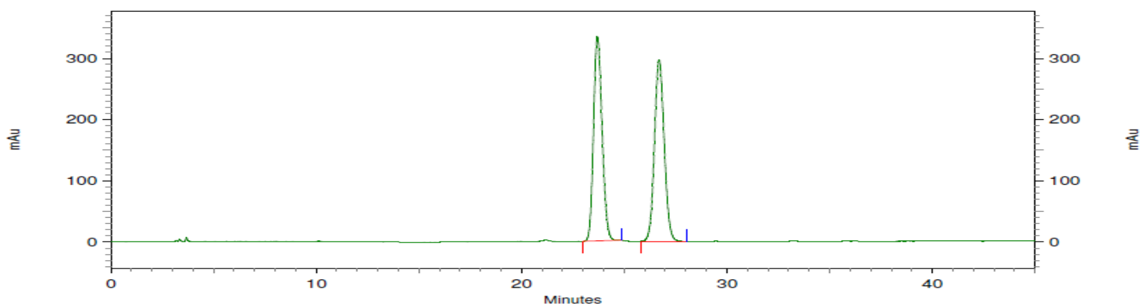


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.604	VV	0.1390	1.87158e4	2098.99927	95.4615
2	5.100	VB	0.1280	889.79761	104.95155	4.5385

Table 2, entry 10



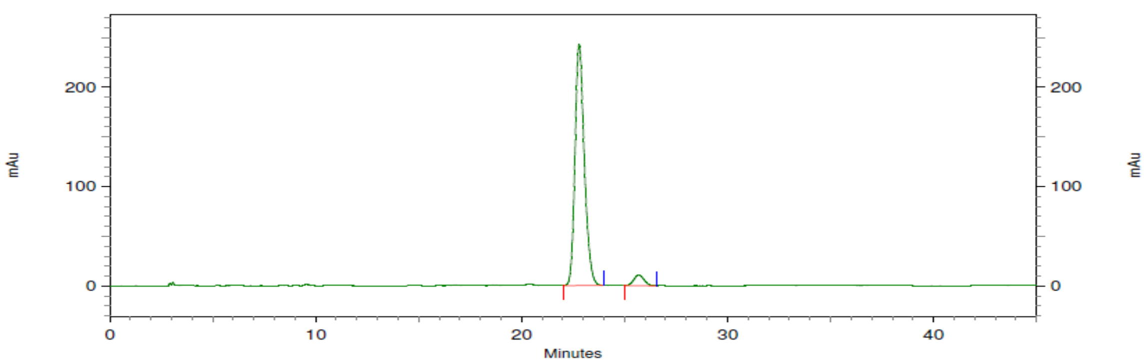
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
23.691	9690162	49.73	333591	52.96
26.699	9796544	50.27	296306	47.04
Totals	19486706	100.00	629897	100.00

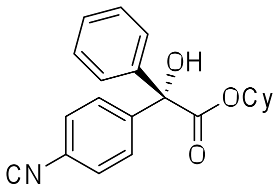
Optically active



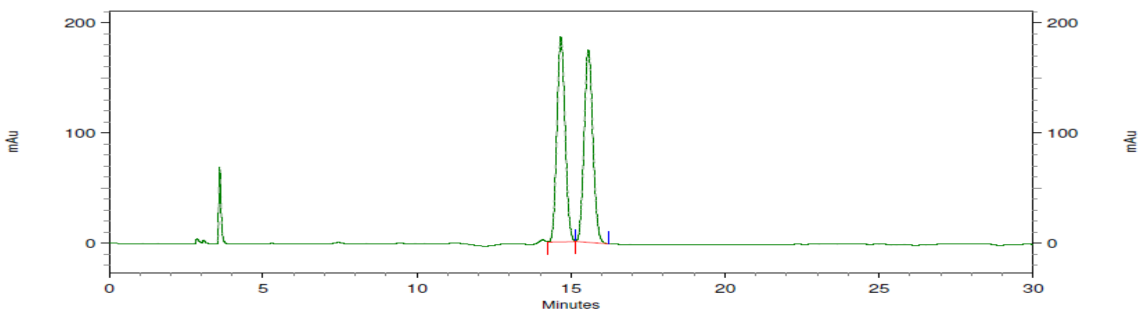
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
22.795	7721175	95.39	242417	95.75
25.691	373062	4.61	10755	4.25
Totals	8094237	100.00	253172	100.00

Table 2, entry 11



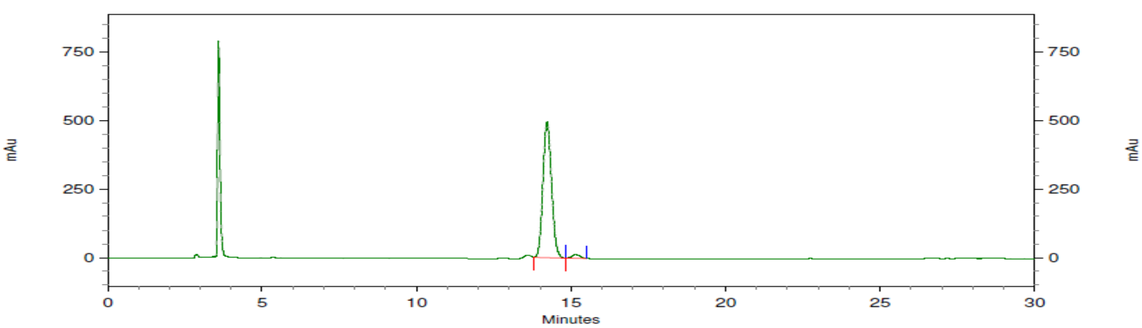
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
14.672	3440521	49.84	185694	51.57
15.563	3463123	50.16	174393	48.43
Totals	6903644	100.00	360087	100.00

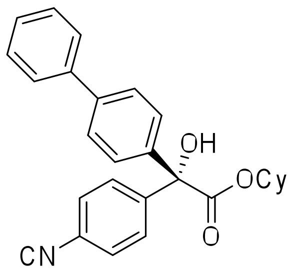
Optically active



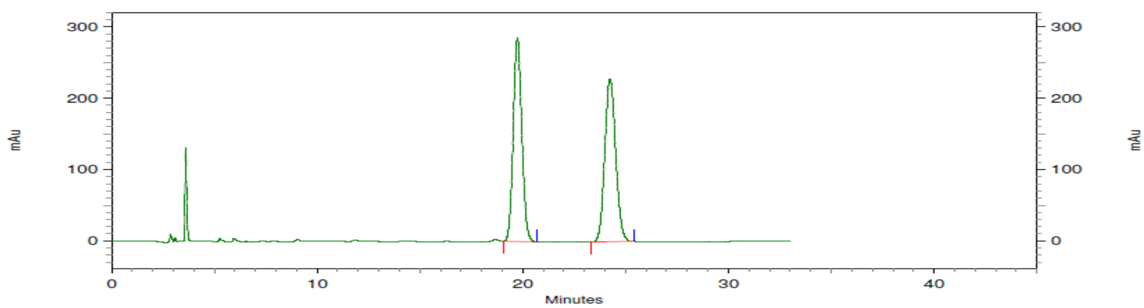
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
14.224	9592531	97.53	494652	97.46
15.152	242795	2.47	12892	2.54
Totals	9835326	100.00	507544	100.00

Table 2, entry 12



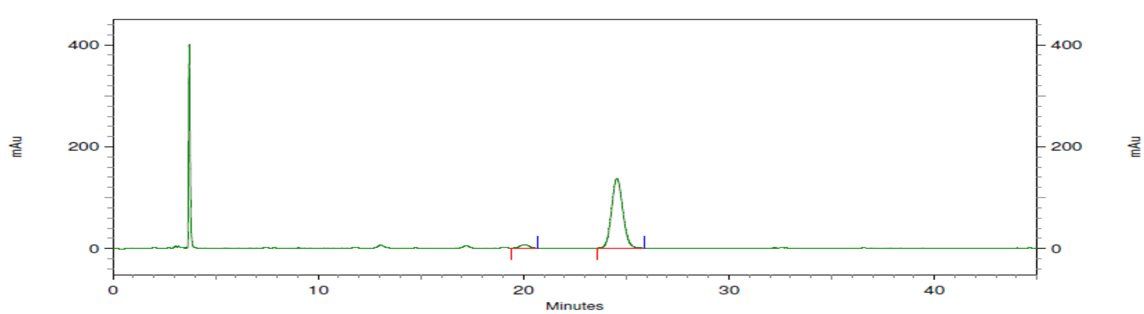
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
19.739	8299322	49.67	285264	55.51
24.245	8411162	50.33	228654	44.49
Totals	16710484	100.00	513918	100.00

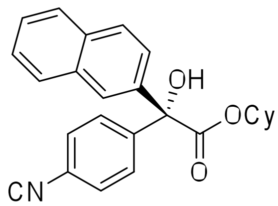
Optically active



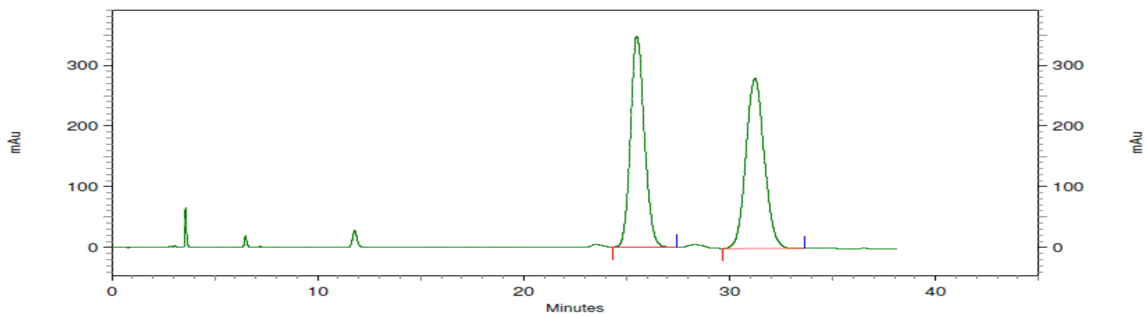
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
20.048	185508	3.51	6428	4.50
24.549	5106070	96.49	136543	95.50
Totals	5291578	100.00	142971	100.00

Table 2, entry 13



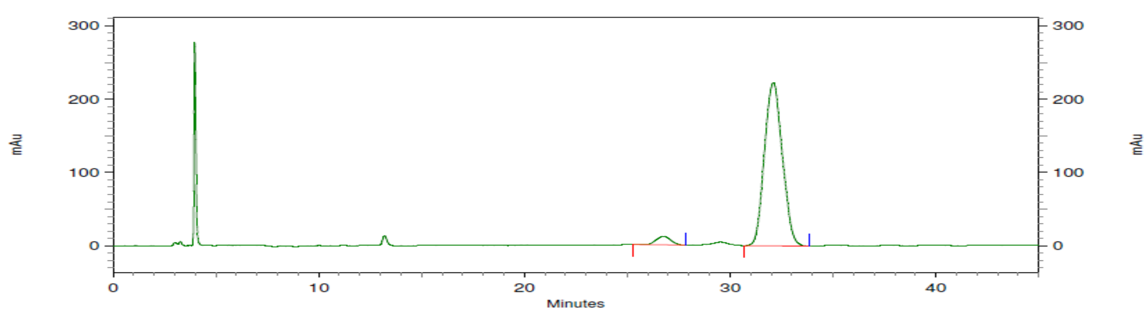
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
25.488	16339815	48.39	346937	55.30
31.243	17424910	51.61	280396	44.70
Totals	33764725	100.00	627333	100.00

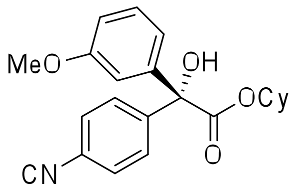
Optically active



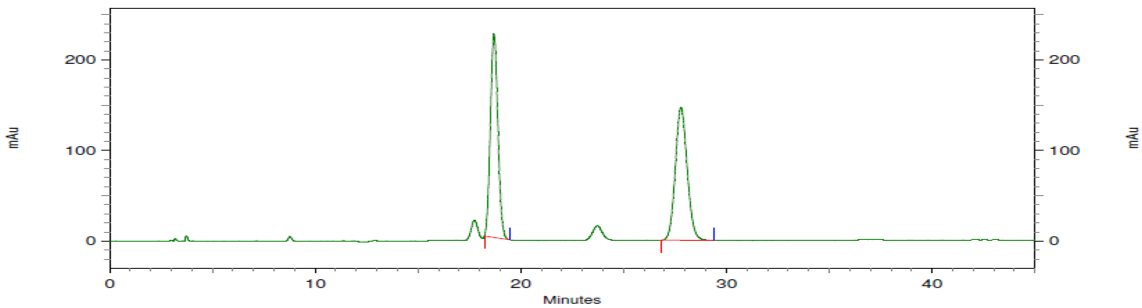
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
26.757	580217	4.12	11895	5.07
32.107	13492721	95.88	222910	94.93
Totals	14072938	100.00	234805	100.00

Table 2, entry 14



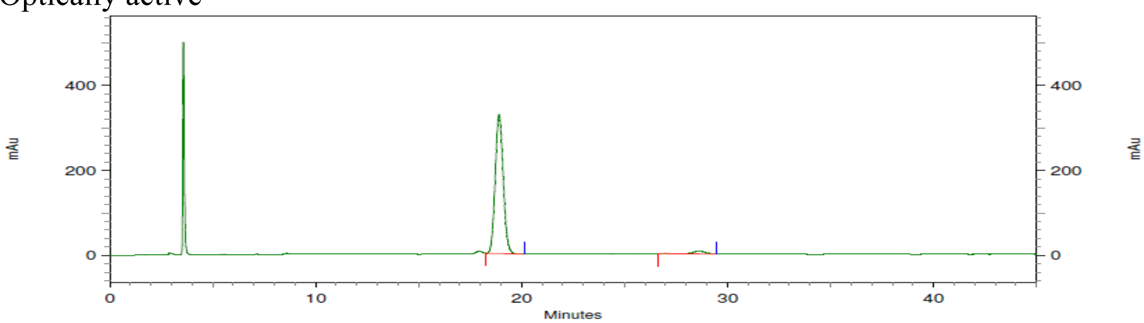
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
18.699	5710889	49.08	224648	60.53
27.797	5924439	50.92	146481	39.47
Totals	11635328	100.00	371129	100.00

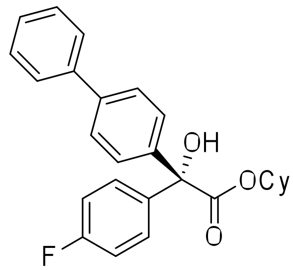
Optically active



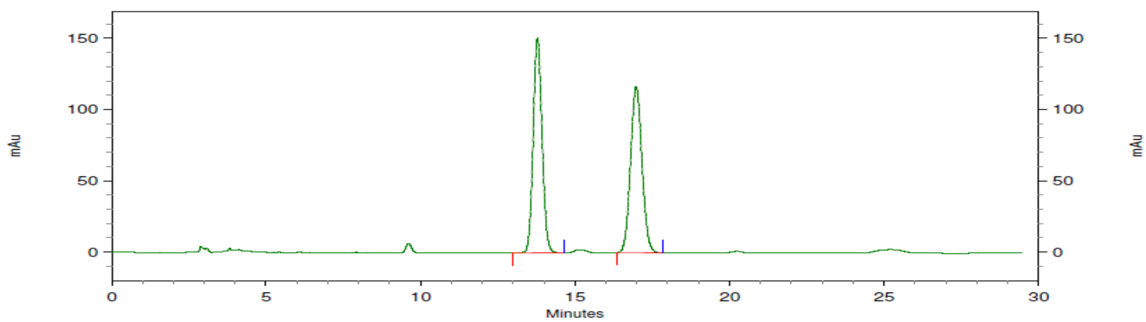
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
18.907	8918163	97.04	327529	98.04
28.613	271912	2.96	6545	1.96
Totals	9190075	100.00	334074	100.00

Table 2, entry 15



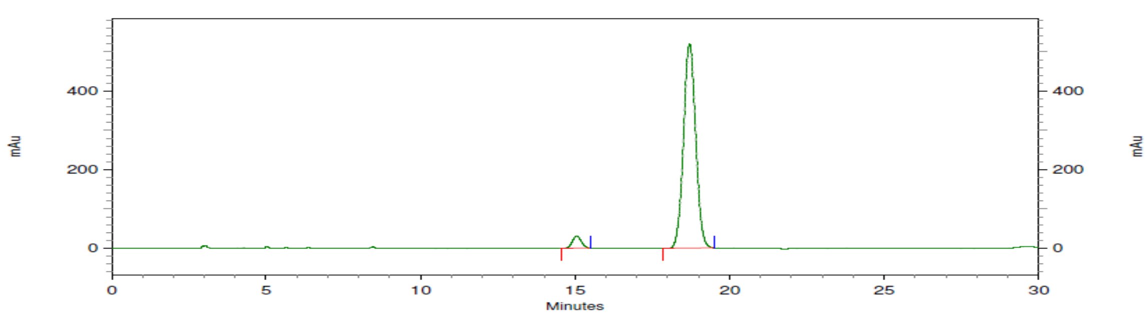
Racemic



4: 254 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
13.781	3015962	50.08	150460	56.40
16.976	3006328	49.92	116326	43.60
Totals	6022290	100.00	266786	100.00

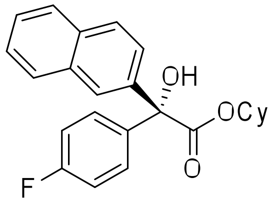
Optically active



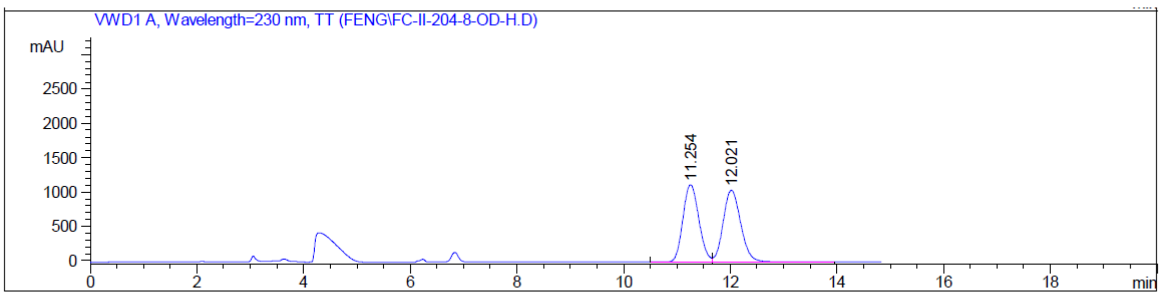
4: 254 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
15.051	617829	4.16	30929	5.62
18.704	14236815	95.84	519354	94.38
Totals	14854644	100.00	550283	100.00

Table 2, entry 16

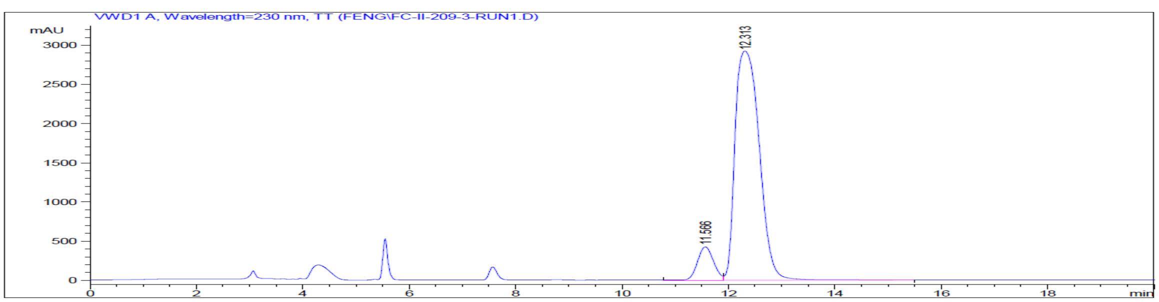


Racemic



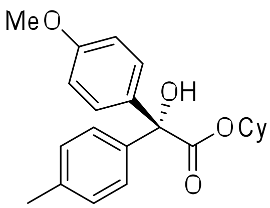
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.254	VV	0.3278	2.35532e4	1128.98962	49.3169
2	12.021	VV	0.3623	2.42056e4	1049.40320	50.6831

Optically active

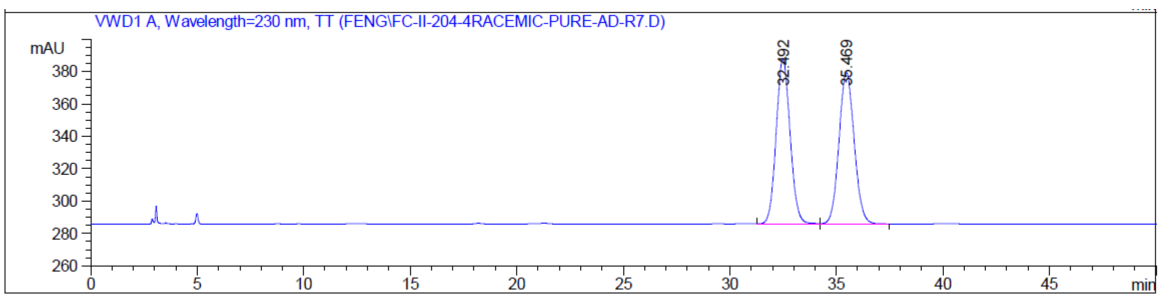


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.566	VV	0.3278	8855.95410	424.63104	8.7249
2	12.313	VV	0.5093	9.26465e4	2924.71606	91.2751

Table 2, entry 17

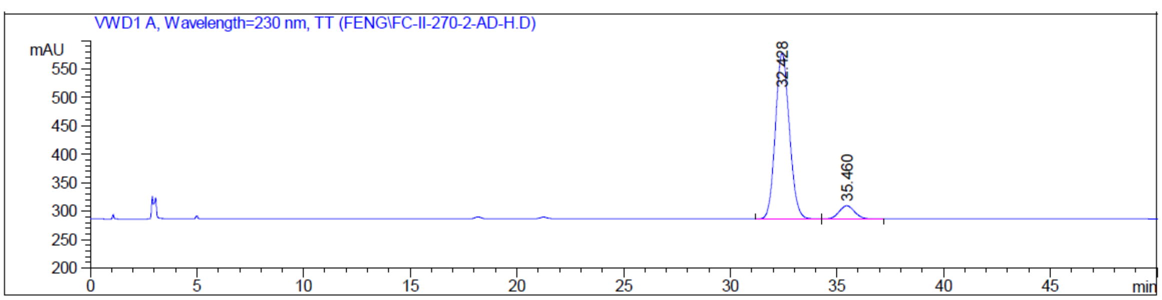


Racemic



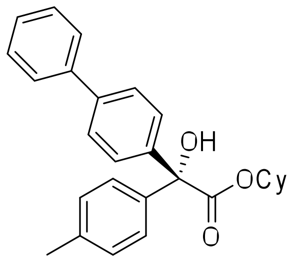
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	32.492	BB	0.7045	4599.35449	101.56059	49.9096
2	35.469	BB	0.7719	4616.01123	93.36088	50.0904

Optically active

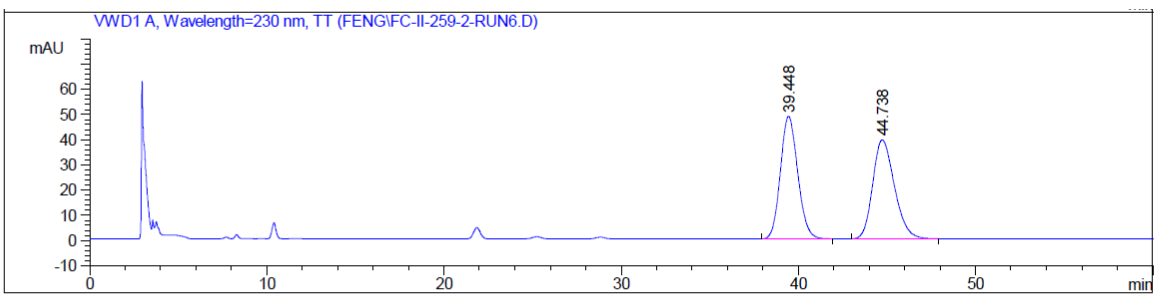


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	32.428	BB	0.7078	1.33153e4	291.42218	91.9277
2	35.461	BB	0.7808	1169.23279	23.29166	8.0723

Table 2, entry 18

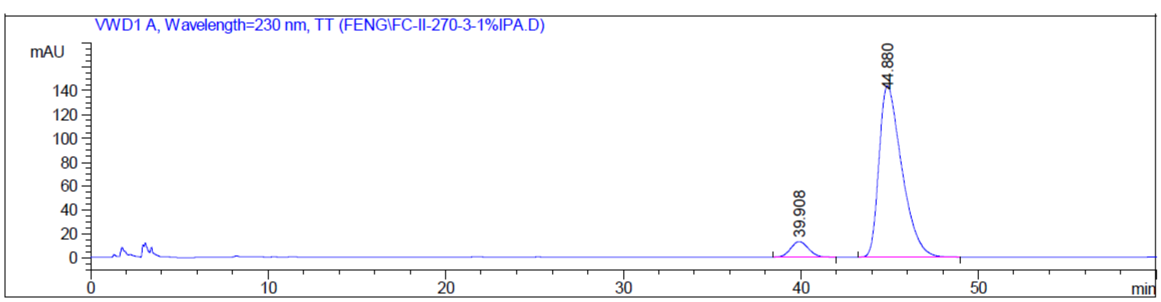


Racemic



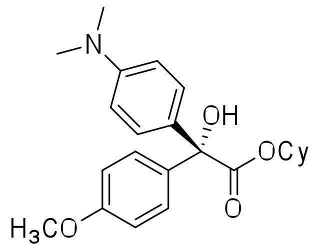
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	39.448	BB	1.0572	3315.71143	48.64926	49.6924
2	44.738	BB	1.3105	3356.76367	39.31984	50.3076

Optically active

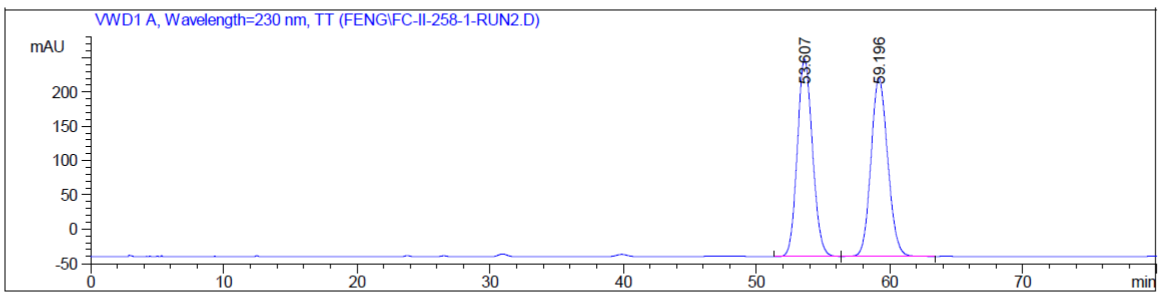


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	39.908	BB	1.0666	910.79620	13.11300	6.6616
2	44.880	BB	1.3359	1.27616e4	143.31456	93.3384

Table 2, entry 19

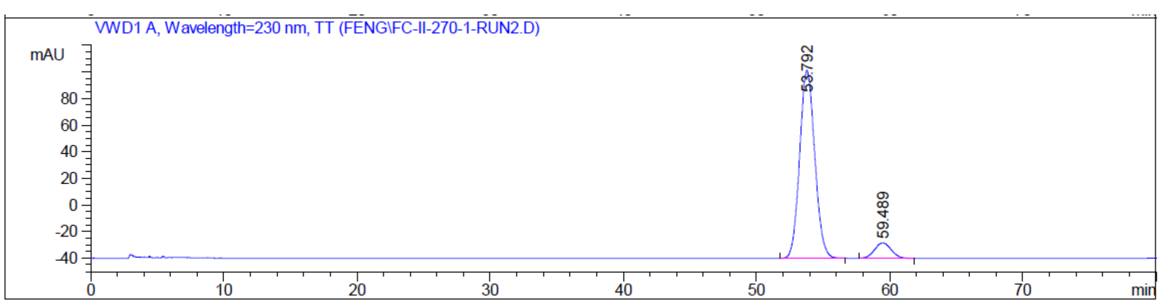


Racemic



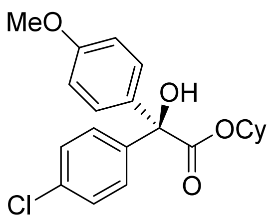
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	53.607	VV	1.2285	2.28821e4	288.39093	49.9227
2	59.196	VB	1.3584	2.29529e4	260.89792	50.0773

Optically active

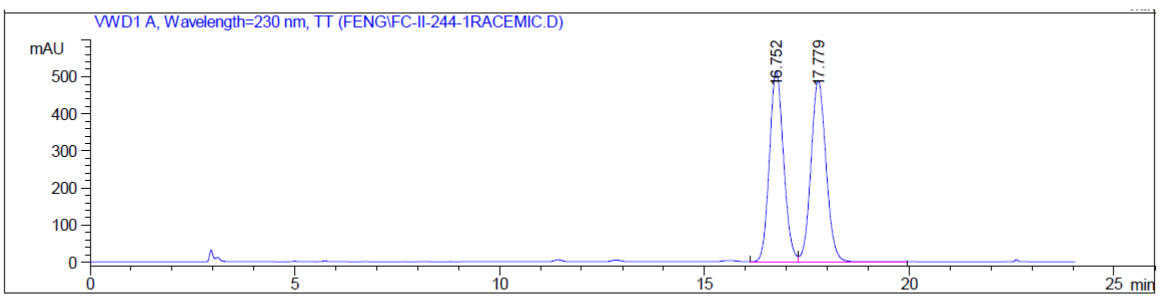


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	53.792	BB	1.2559	1.11751e4	141.11200	91.6081
2	59.489	BB	1.3612	1023.70441	11.73689	8.3919

Table 2, entry 20

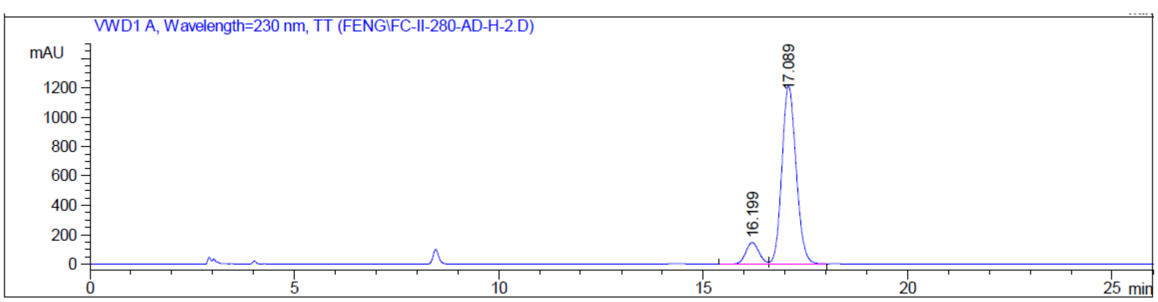


Racemic



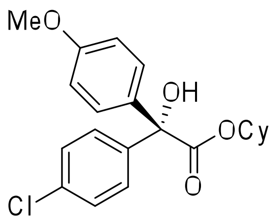
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.752	VV	0.3711	1.22956e4	513.55023	49.6945
2	17.779	VB	0.3951	1.24468e4	487.99680	50.3055

Optically active

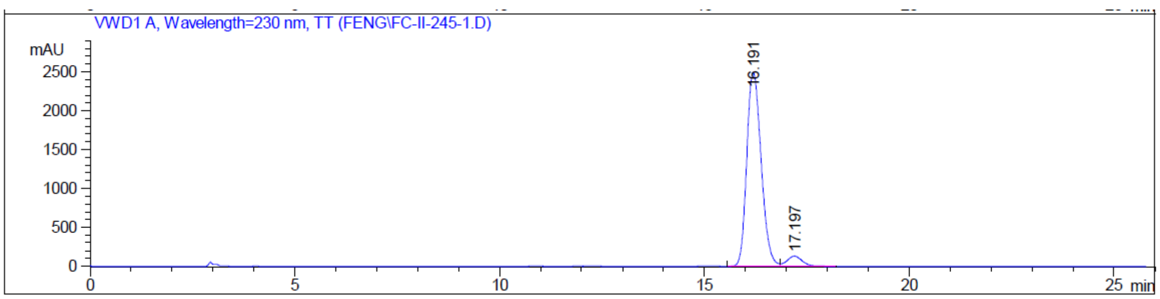


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.199	VV	0.3504	3319.93994	147.23799	10.1513
2	17.089	VV	0.3764	2.93845e4	1210.44592	89.8487

Table 2, entry 21

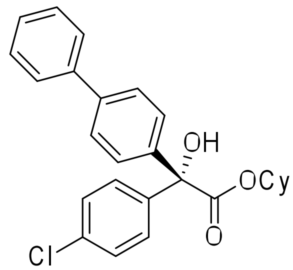


Optically active

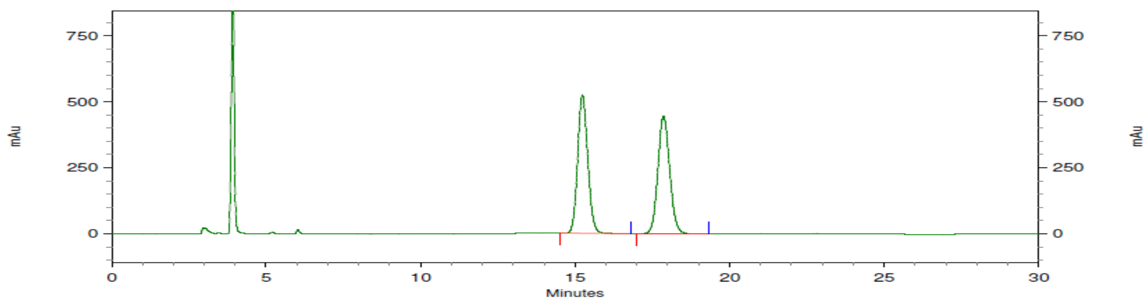


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.191	VV	0.3831	6.07033e4	2493.05640	94.8536
2	17.197	VV	0.3912	3293.52832	128.92398	5.1464

Table 2, entry 22



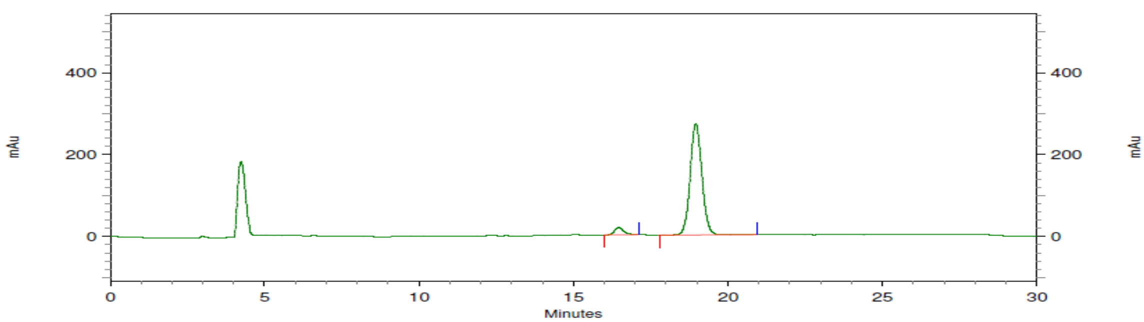
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
15.237	11905865	50.07	523817	54.09
17.861	11873556	49.93	444538	45.91
Totals	23779421	100.00	968355	100.00

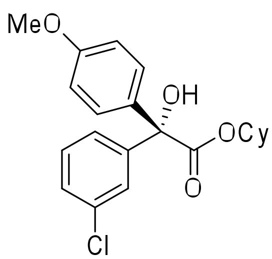
Optically active



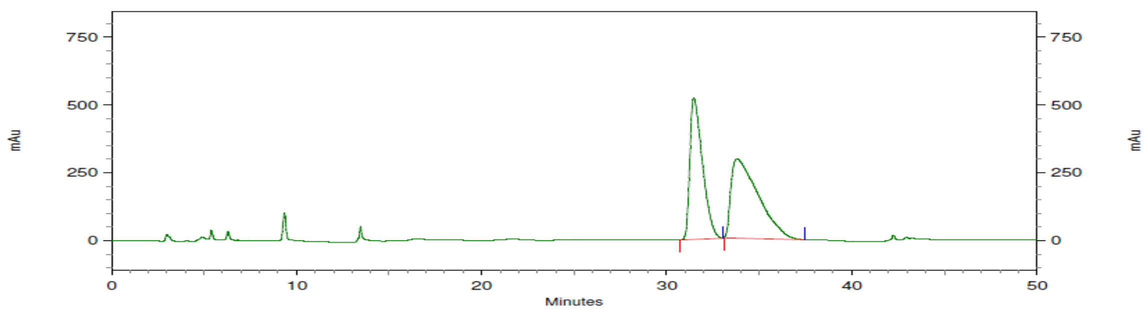
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
16.464	386466	5.12	18082	6.25
18.960	7163989	94.88	271247	93.75
Totals	7550455	100.00	289329	100.00

Table 2, entry 23



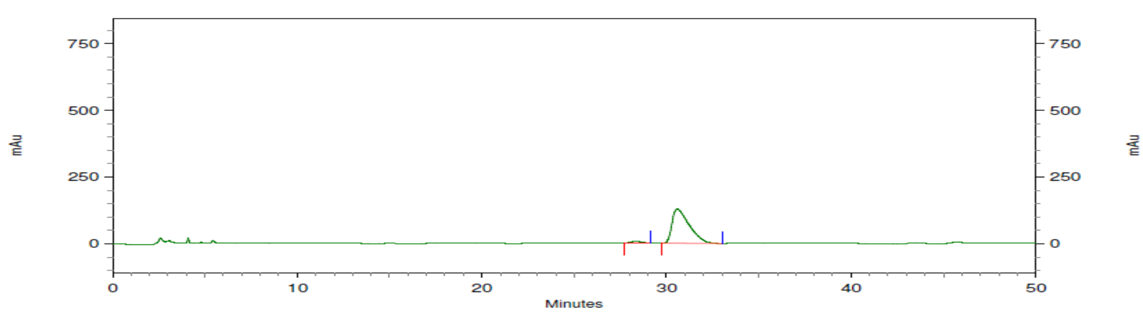
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
31.467	24662807	45.14	520485	64.13
33.792	29968008	54.86	291186	35.87
Totals	54630815	100.00	811671	100.00

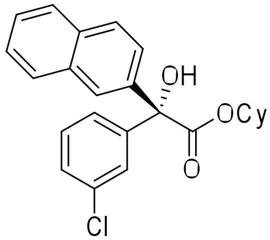
Optically active



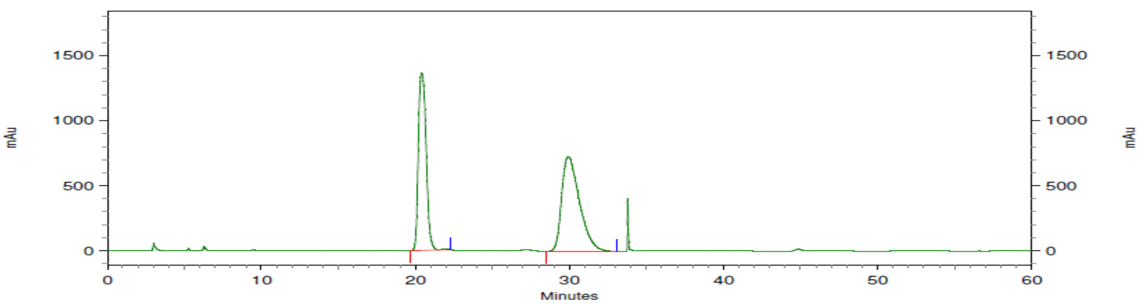
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
28.363	311292	3.60	7861	5.77
30.581	8333643	96.40	128344	94.23
Totals	8644935	100.00	136205	100.00

Table 2, entry 24



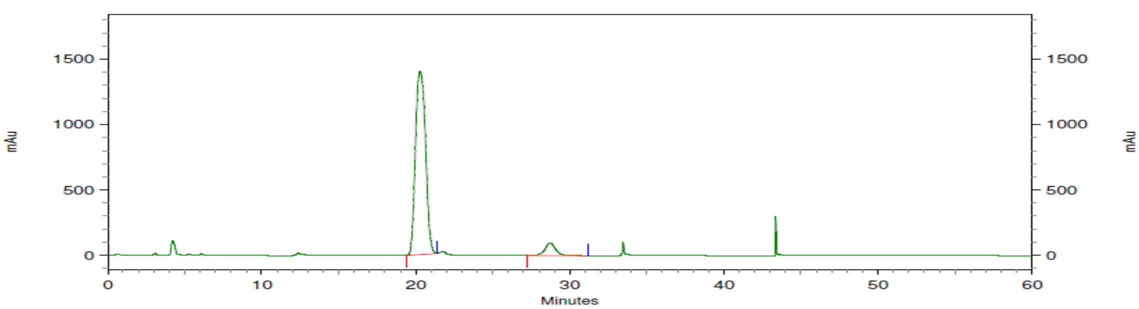
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
20.411	48057897	45.05	1365968	65.18
29.920	58619233	54.95	729702	34.82
Totals	106677130	100.00	2095670	100.00

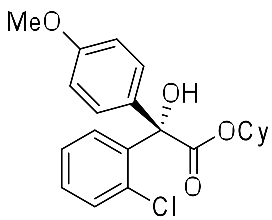
Opyical active



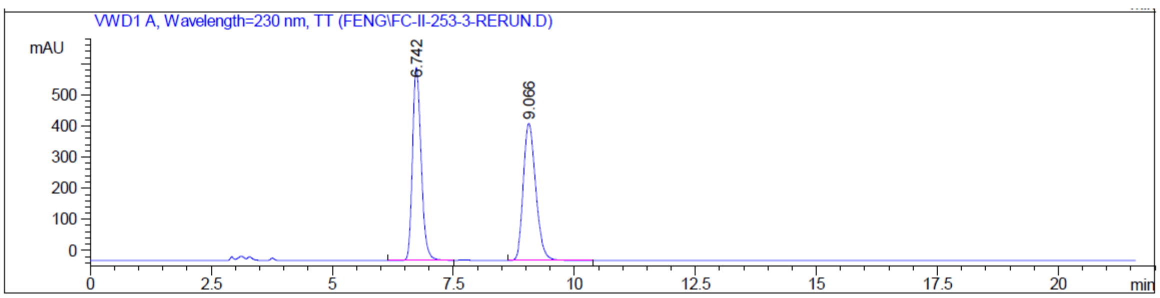
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
20.277	63766802	92.92	1403763	93.45
28.725	4857445	7.08	98460	6.55
Totals	68624247	100.00	1502223	100.00

Table 2, entry 25

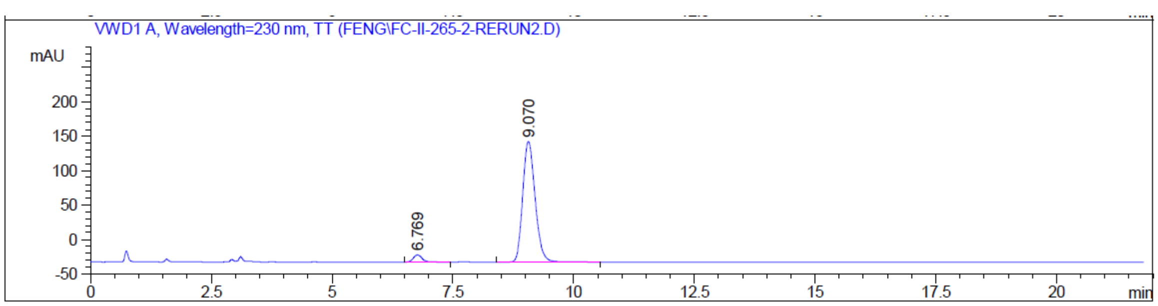


Racemic



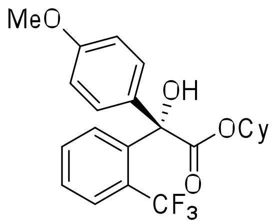
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.742	VV	0.1923	7729.17139	619.03796	49.3524
2	9.066	VV	0.2799	7932.02734	440.56595	50.6476

Optically active

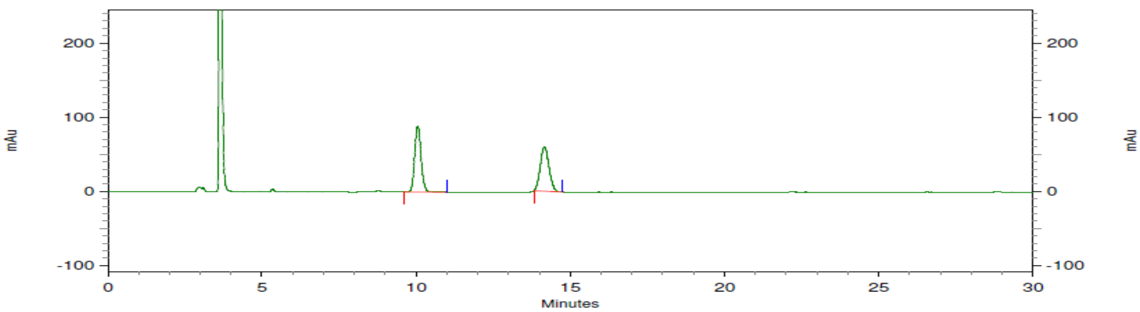


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.769	BB	0.1974	137.50420	10.63715	4.2019
2	9.070	VB	0.2785	3134.91113	175.35664	95.7981

Table 2, entry 26



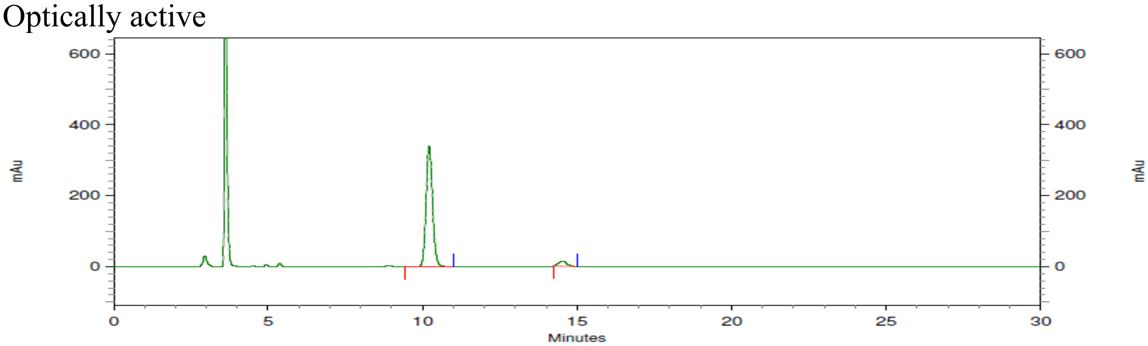
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
10.048	1279970	51.75	89286	59.80
14.160	1193301	48.25	60020	40.20
Totals	2473271	100.00	149306	100.00

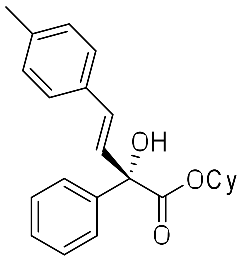
Optically active



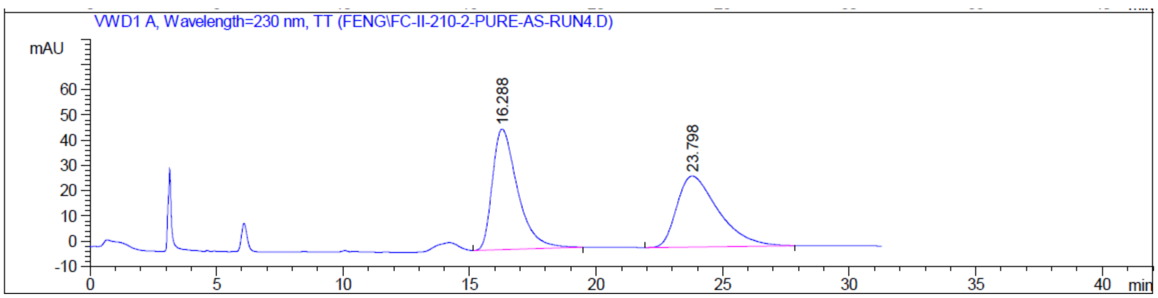
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
10.208	4943752	95.00	339284	96.14
14.528	260386	5.00	13614	3.86
Totals	5204138	100.00	352898	100.00

Table 2, entry 27

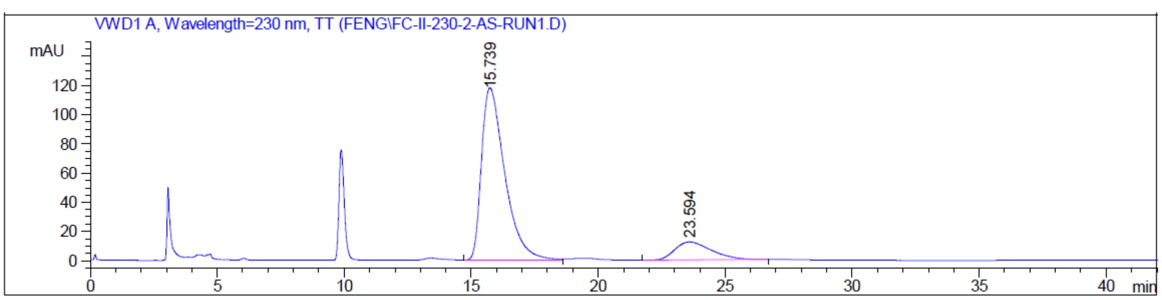


Racemic



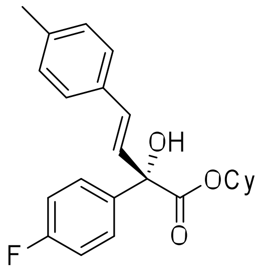
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.288	VB	1.0545	3319.57568	47.82064	50.9462
2	23.798	BB	1.7192	3196.26709	28.05784	49.0538

Optically active

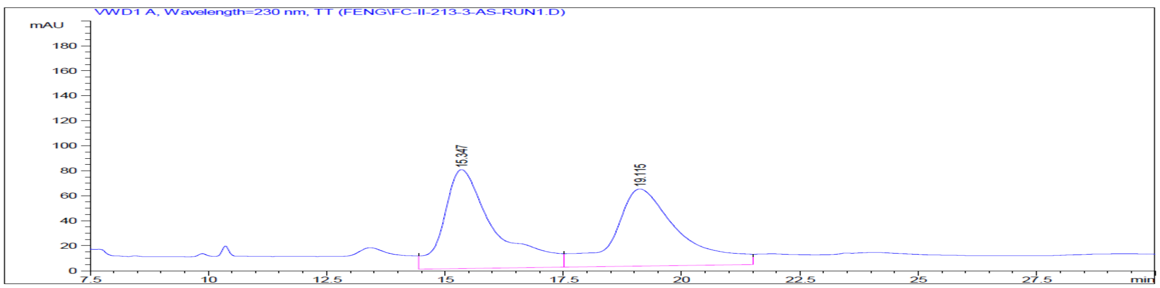


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.739	VV	0.9748	7566.45264	118.06339	85.9109
2	23.594	VB	1.5245	1240.86841	12.32055	14.0891

Table 2, entry 28

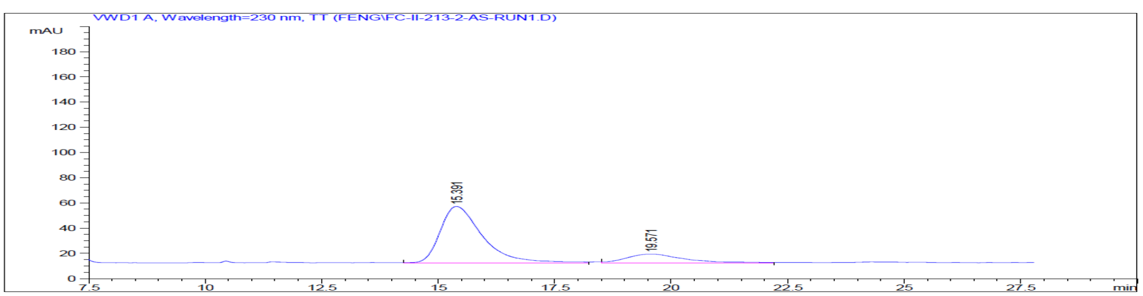


Racemic



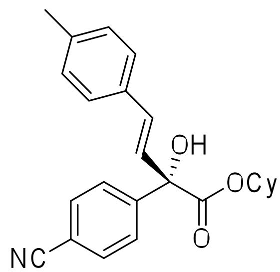
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.347	VV	1.0735	5899.48926	79.14110	49.9922
2	19.115	VV	1.3656	5901.32520	61.74097	50.0078

Optically active

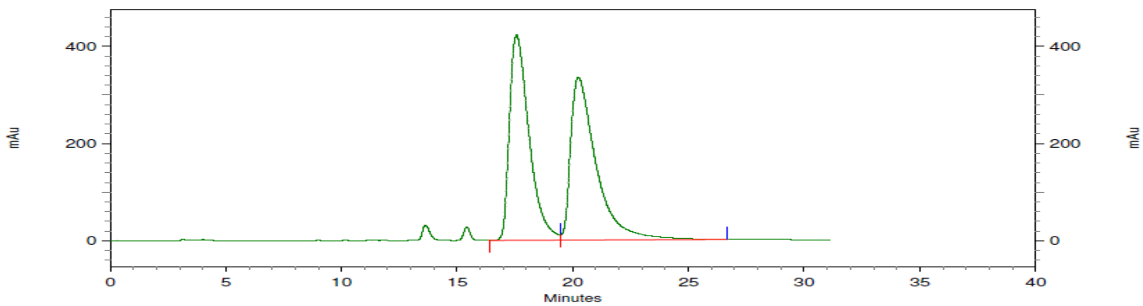


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.391	VV	0.9540	2838.90405	44.84940	82.3325
2	19.571	VV	1.2763	609.19263	7.00787	17.6675

Table 2, entry 29



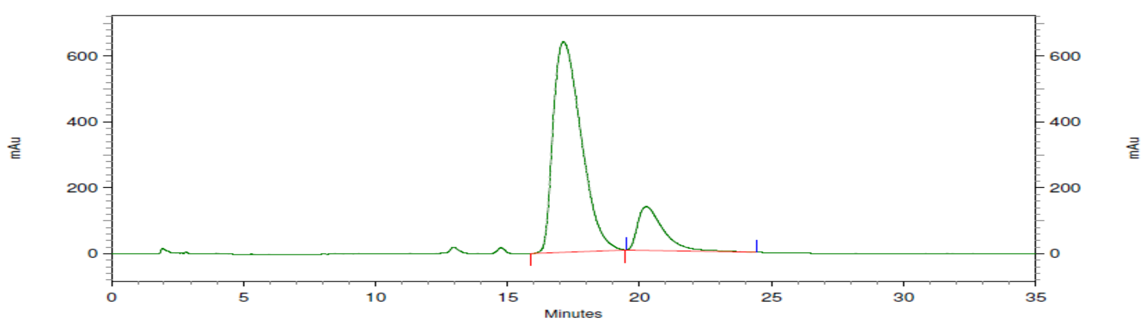
Racemic



4: 254 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
17.568	24901014	49.08	422352	55.78
20.240	25837934	50.92	334849	44.22
Totals	50738948	100.00	757201	100.00

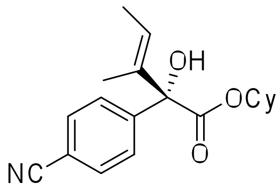
Optically active



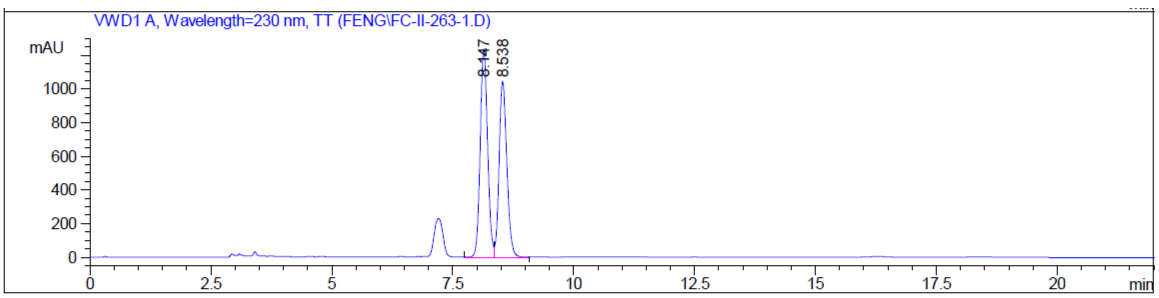
4: 254 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
17.125	59068726	82.63	807679	82.68
20.251	12413081	17.37	169164	17.32
Totals	71481807	100.00	976843	100.00

Table 2, entry 30

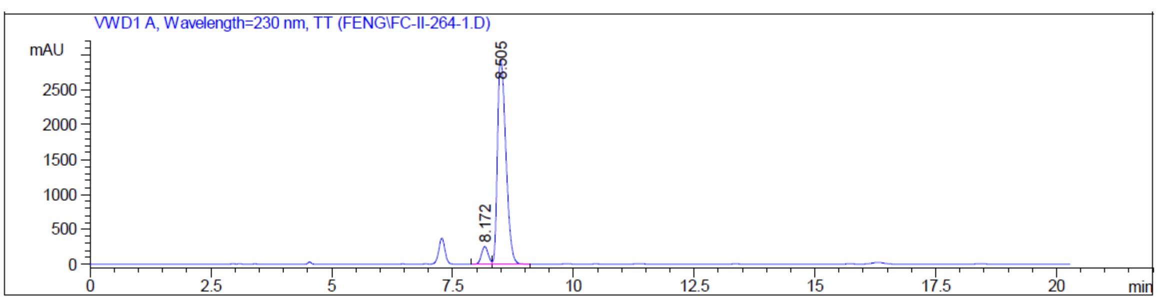


Racemic



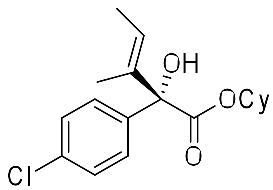
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.147	VV	0.1641	1.30437e4	1233.96631	52.9778
2	8.538	VV	0.1762	1.15774e4	1042.42041	47.0222

Optically active

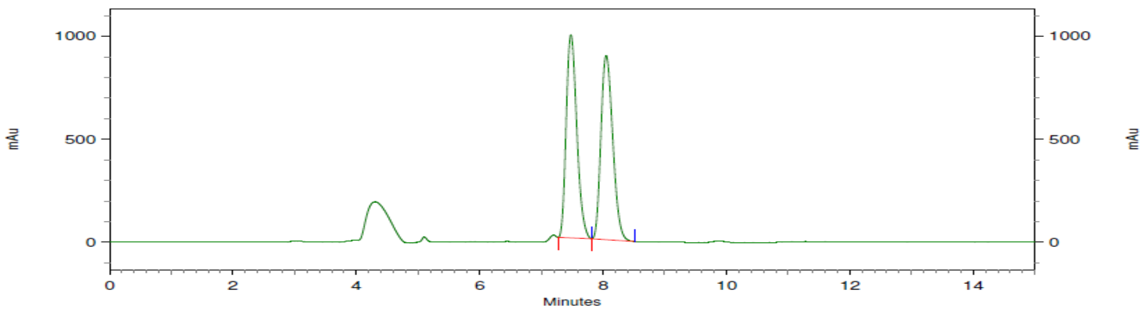


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.172	VV	0.1493	2508.14673	255.87541	6.6010
2	8.505	VV	0.1884	3.54886e4	2920.02710	93.3990

Table 2, entry 31



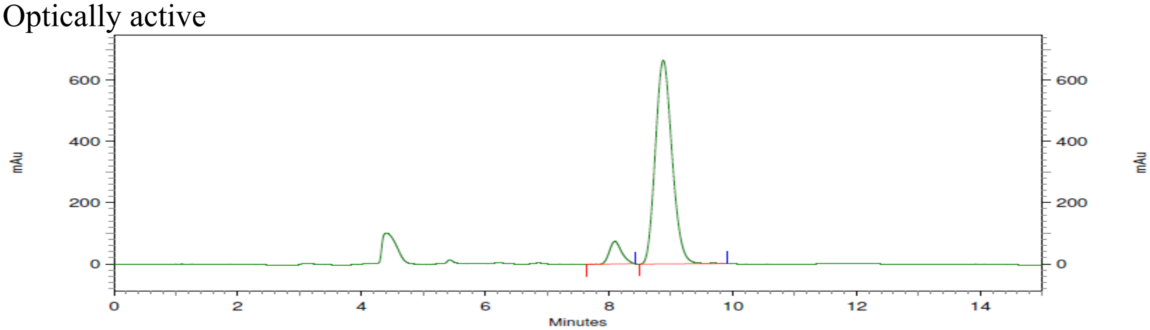
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
7.477	11758988	49.63	986694	52.45
8.048	11933277	50.37	894674	47.55
Totals	23692265	100.00	1881368	100.00

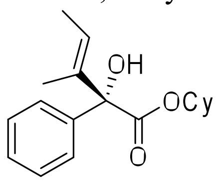
Optically active



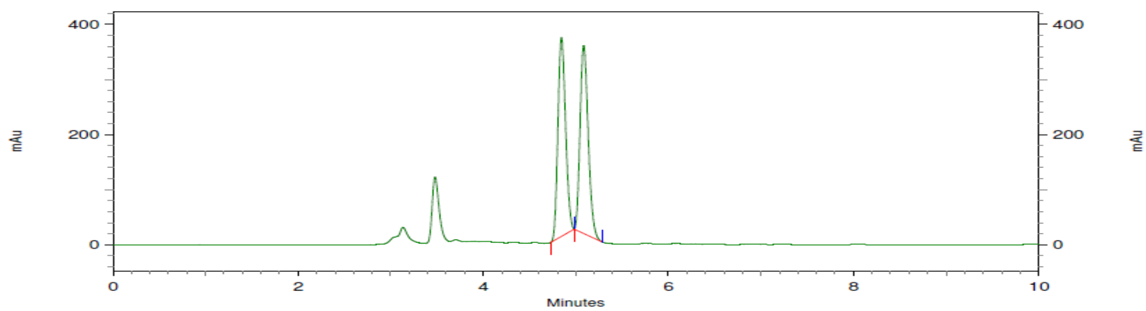
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
8.096	1031800	7.62	73886	10.00
8.875	12517296	92.38	664735	90.00
Totals	13549096	100.00	738621	100.00

Table 2, entry 32



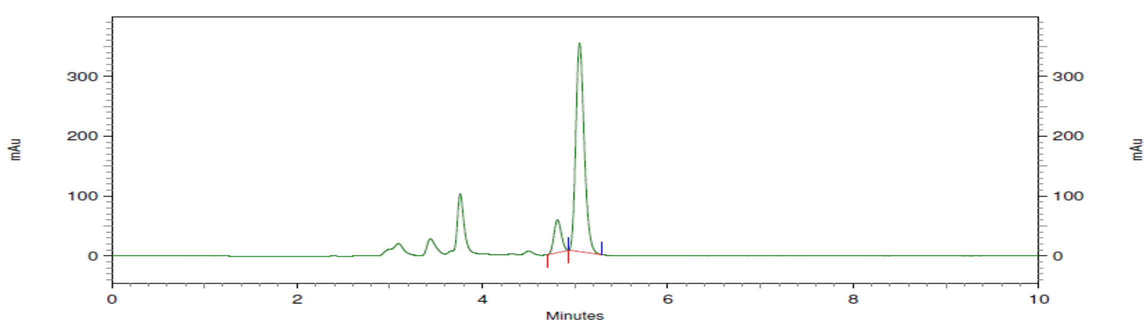
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
4.843	2079757	49.92	361478	51.46
5.083	2086602	50.08	340899	48.54
Totals	4166359	100.00	702377	100.00

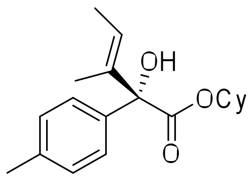
Optically active



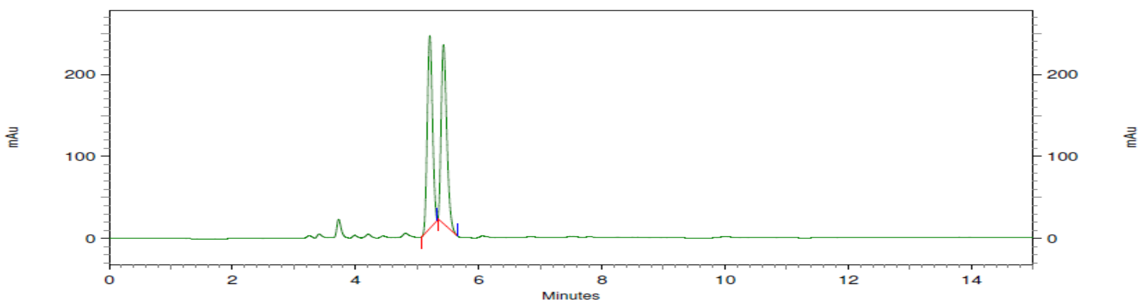
1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
4.811	298812	11.96	54725	13.59
5.051	2199024	88.04	347996	86.41
Totals	2497836	100.00	402721	100.00

Table 2, entry 33



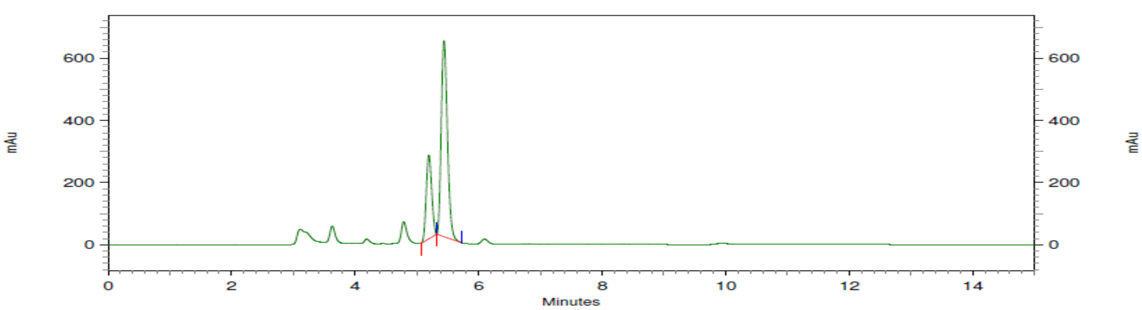
Racemic



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
5.205	1378555	49.91	234975	51.84
5.429	1383517	50.09	218300	48.16
Totals	2762072	100.00	453275	100.00

Optically active



1: 229 nm, 8 nm
Results

Retention Time	Area	Area %	Height	Height %
5.195	1655070	27.43	269141	29.92
5.440	4377815	72.57	630411	70.08
Totals	6032885	100.00	899552	100.00