Supplementary Information (SI) for ChemComm.

Supporting Information

Radical-polar Crossover Reaction of Glycine Derivatives

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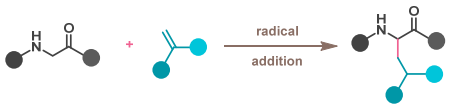

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General Information

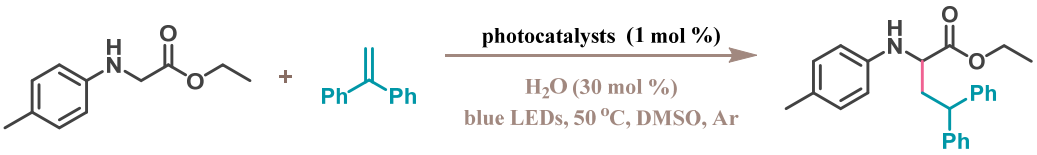
The starting materials, reagents and solvents, purchased from commercial suppliers, were used without further purification. Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. Flash chromatography was carried out using silica gel 200 - 300. ^{1}HNMR (400 MHz or 600 MHz) and ^{13}CNMR (151 MHz) spectra were measured with $CDCl_3$ as solvent. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. High-resolution mass spectra (HRMS) were performed on Agilent 6520 with an ultra-high resolution quadruple Time-of-Flight (qTOF) detector and recorded under electrospray ionization (ESI) conditions.

The synthesis of substrates

The synthesis of glycine derivatives **A** used in this work were prepared according to the methods reported in literature. [1] The synthesis of 1,1-diarylethylenes **B** used in this work were prepared according to the methods reported in literature. [2]

Optimization of reaction conditions

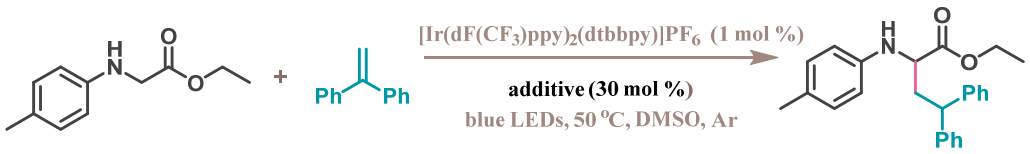
Table S1: Screening of photocatalysts



entry	photocatalyst (1 mol %)	yield (%)[b]
1	-	n.d.
2	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	72
3	$Ru(bpy)_3Cl_2$	10
4	fac-$Ir(bpy)_3$	5
5	$[Ir(dtbbpy)(ppy)_2]PF_6$	53
6	4CzIPN	54
7	Mes-Acr-ph$^{+}BF_4^{-}$	trace

[a] Reaction conditions: ethyl *p*-tolylglycinate (0.1 mmol), 1,1-diphenylethylene (0.4 mmol), photocatalysts (1 mol %), H_2O (30 mol %), DMSO (0.25 mL), Ar, blue LEDs, 50 °C, 24 h. [b] Isolated yield.

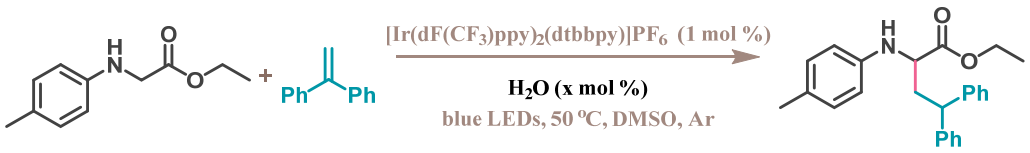
Table S2: Screening of additive



entry	additive (30 mol %)	yield (%)[b]
1	4-MePhSH	67
2	Cyclohexyl mercaptan	48
3	1-Dodecanethiol	64
4	Perfluorodecanethiopl	52
5	Benzyl mercaptan	69
6	Hantzsch ester	trace
7	AcOH	46
8	TFA	8
9	**H_2O**	**72**

[a] Reaction conditions: ethyl *p*-tolylglycinate (0.1 mmol), 1,1-diphenylethylene (0.4 mmol), [Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$ (1 mol %), additive (30 mol %), DMSO (0.25 mL), Ar, blue LEDs, 50 °C, 24 h. [b] Isolated yield.

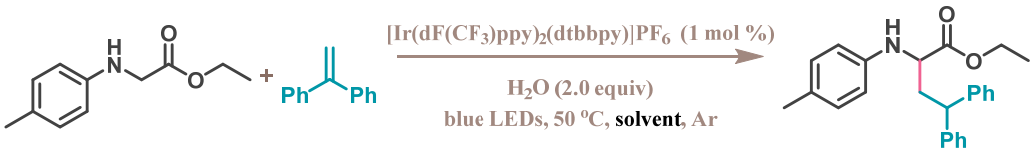
Table S3: Screening the loading of additive



entry	additive	yield (%)[b]
1	-	49
2	H_2O (30 mol %)	72
3	H_2O (50 mol %)	71
4	H_2O (1.0 equiv)	77
5	**H_2O (2.0 equiv)**	**82**
6	H_2O (3.0 equiv)	63
7	H_2O (5..0 equiv)	55
8	H_2O (8.0 equiv)	53

[a] Reaction conditions: ethyl *p*-tolylglycinate (0.1 mmol), 1,1-diphenylethylene (0.4 mmol), [Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$ (1 mol %), H_2O (x mol %), DMSO (0.25 mL), Ar, blue LEDs, 50 °C, 24 h. [b] Isolated yield.

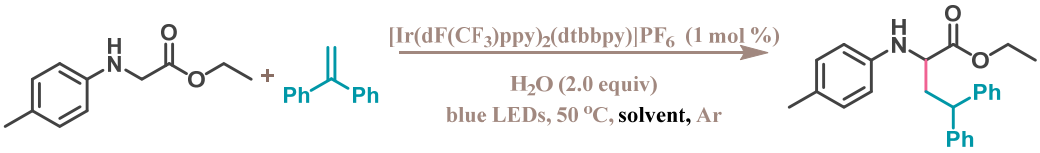
Table S4: Screening of solvent



entry	solvent	yield (%)[b]
1	DMSO	82
2	DMF	39
3	DMA	35
4	EtOH	10
5	HFIP	trace
6	DCE	45
7	DCM	9
8	EA	15
9	$CHCl_3$	8
10	MeCN	18

[a] Reaction conditions: ethyl *p*-tolylglycinate (0.1 mmol), 1,1-diphenylethylene (0.4 mmol), $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %), H_2O (2.0 equiv), solvent (0.25 mL), Ar, blue LEDs, 50 °C, 24 h. [b] Isolated yield.

Table S5: Screening the loading of solvent



entry	solvent	yield (%)[b]
1	DMSO (0.1 mL)	26
2	DMSO (0.25 mL)	82
3	DMSO (0.5 mL)	71
4	DMSO (1.0 mL)	57
5	DMSO (2.0 mL)	20

[a] Reaction conditions: ethyl *p*-tolylglycinate (0.1 mmol), 1,1-diphenylethylene (0.4 mmol), $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %), H_2O (2.0 equiv), solvent (x mL), Ar, blue LEDs, 50 °C, 24 h. [b] Isolated yield.

General procedure of radical addition of glycine derivatives with alkenes

General procedure 1: To a dried Schlenk tube (10 mL) with a magnetic stirring bar were added glycine esters (**A**, 0.2 mmol) and $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, alkenes (**B**, 0.8 mmol), H_2O (2.0 equiv) and degassed DMSO (0.5 mL) were added under argon atmosphere. Then, the resulting reaction mixture was 50 ℃ under blue LEDs (30 W) irradiation for 24 hours. The reaction progress was monitored by TLC. After the reaction was completed, the reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration, and the residue was purified by column chromatography to afford the desired compounds **C1-C31** (EA/PE = 1:3 - 1:150).

General procedure 2: To a dried Schlenk tube (10 mL) with a magnetic stirring bar were added glycine esters (**A**, 0.2 mmol) and $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, alkenes (**B**, 0.4 mmol), 2, 4, 6-triisopropylbenzenethiol (30 mol %) and degassed DCE (2 mL) were added under argon atmosphere. Then, the resulting reaction mixture was performed at 50 ℃ under blue LEDs (30 W) irradiation for 60 hours. The reaction progress was monitored by TLC. After the reaction was completed, the reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography to afford the desired compounds **C32 - C46** (EA/PE = 1:20 - 1:200).

General procedure 3: To a dried Schlenk tube (10 mL) with a magnetic stirring bar were added glycine esters (**A**, 0.2 mmol) and $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, alkenes (**B**, 0.8 mmol) and degassed DMSO (0.5 mL) were added under argon atmosphere. Then, the resulting reaction mixture was 50 ℃ under blue LEDs (30 W) irradiation for 24 hours. The reaction progress was monitored by TLC. After the reaction was completed, the reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration, and the residue was purified by column chromatography to afford the desired compounds **C47**

- C54 (EA/PE = 1:20 - 1:80).

Scale-up experiment

To a dried Schlenk tube (25 mL) with a magnetic stirring bar were added ethyl p-tolylglycinate (5 mmol), $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, 1,1-diphenylethylene (20 mmol), H_2O (2.0 equiv) and degassed DMSO (12.5 mL) were added under argon atmosphere. Then, the resulting reaction mixture was 50 ℃ under blue LEDs (30 W) irradiation for 24 hours. The reaction progress was monitored by TLC. After the reaction was completed, the reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration, and the residue was purified by column chromatography to afford the desired compounds (1.31 g, 70%).

Radical trapping experiments

To a dried Schlenk tube (10 mL) with a magnetic stirring bar were added ethyl p-tolylglycinate (0.1 mmol) and $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, 1,1-diphenylethylene (0.4 mmol), H_2O (2.0 equiv), TEMPO (2.0 equiv) and degassed DMSO (0.25 mL) were added under argon atmosphere. Then, the resulting reaction mixture was 50 ℃ under blue LEDs (30 W) irradiation for 1 hours. then reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration, and detected by HRMS analysis of the reaction mixture.

The isotope labelling experiments with D_2O

To a dried Schlenk tube (10 mL) with a magnetic stirring bar were added ethyl p-tolylglycinate (0.1 mmol) and $Ir(dF(CF_3)ppy)_2(dtbpy)PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, 1,1-diphenylethylene (0.4 mmol), D_2O

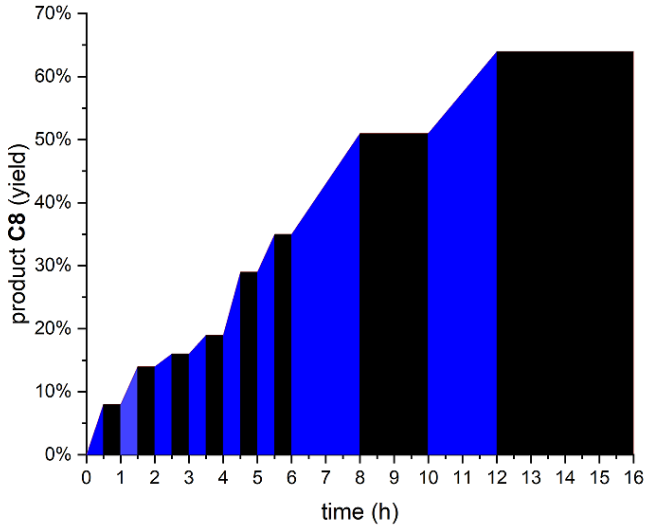
(2.0 equiv) and DMSO degassed (0.25 mL) were added under argon atmosphere. Then, the resulting reaction mixture was 50 °C under blue LEDs (30 W) irradiation for 24 hours. The reaction progress was monitored by TLC. After the reaction was completed, the reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration, and the residue was purified by column chromatography to afford the desired compounds in 58% yield with 26% deuterium incorporation.

Trapping plausible carbanionic species with CO_2

To a dried Schlenk tube (25 mL) with a magnetic stirring bar were added ethyl p-tolylglycinate (0.1 mmol) and $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %) successively. hen evacuated and back-filled with CO_2 atmosphere 3 times. Subsequently, 1,1-diphenylethylene (0.4 mmol) and DMSO (0.25 mL) were added under CO_2 atmosphere and the tube was sealed at atmospheric pressure of CO_2 (1 atm). Then, the resulting reaction mixture was performed at 50 °C under blue LEDs (30 W) irradiation for 24 hours. The reaction progress was monitored by TLC. After the reaction was completed, CH_3I (2 equiv) and Cs_2CO_3 (2 equiv) was added and stirred at 50 °C for 4 hours, the reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration, and the residue was purified by column chromatography to afford the desired compounds (EA/PE = 1:30). ^1H NMR (400 MHz, $CDCl_3$) δ 7.32 - 7.21 (m, 10H), 6.95 (d, J = 8.4 Hz, 2H), 6.51 (d, J = 8.6 Hz, 2H), 4.21 (t, J = 5.5 Hz, 1H), 3.96 - 3.76 (m, 2H), 3.58 (s, 3H), 3.22 (dd, J = 14.3, 6.0 Hz, 1H), 3.00 (dd, J = 14.3, 5.1 Hz, 1H), 2.70 (s, 3H), 2.23 (s, 3H), 1.02 (t, J = 7.1 Hz, 3H).

The light on-off experiment [3]

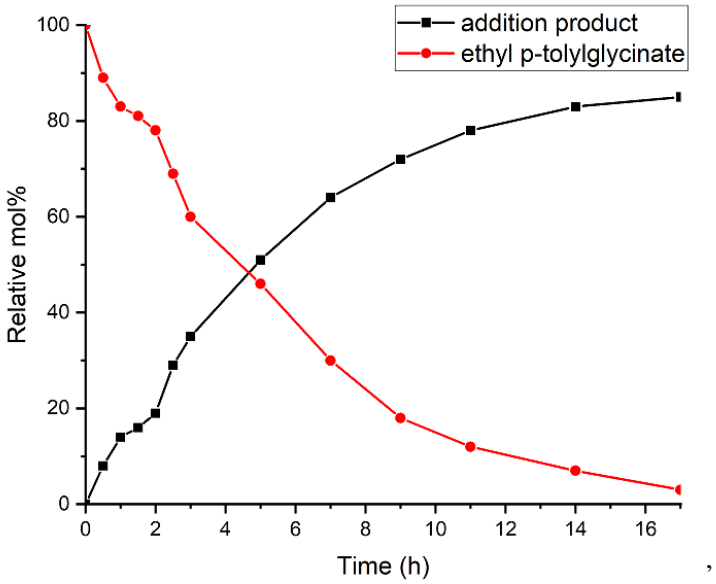
To a dried Schlenk tube (10 mL) with a magnetic stirring bar were added ethyl p-tolylglycinate (0.1 mmol) and $Ir(dF(CF_3)ppy)_2(dtbpy)PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, 1,1-diphenylethylene (0.4 mmol), H_2O (2.0 equiv) and DMSO (0.25 mL) were added under argon atmosphere. Then, the resulting reaction mixture was performed 50 ℃ under blue LEDs (30 W) irradiation. The reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration Sixteen identical reactions were carried out simultaneously and yield was determined by ^1H NMR of the crude mixture using 1,3,5-Trimethoxybenzene as internal standard. These experiments with continuous intervals of irradiation and dark periods leaded to total interruption of the reaction proceed in the absence of light, and reactivity is restored under further light. These results indicated that light is an essential component of the reaction.



Time course experiments [4]

To a dried Schlenk tube (10 mL) with a magnetic stirring bar were added ethyl p-tolylglycinate (0.1 mmol) and $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (1 mol %) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, 1,1-diphenylethylene (0.4 mmol), H_2O (2.0 equiv) and DMSO (0.25 mL) were added under argon atmosphere. Then, the resulting

reaction mixture was performed 50 ℃ under blue LEDs (30 W) irradiation. The reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine 3 times. Twelve identical reactions were carried out simultaneously and yield was determined by ^{1}H NMR of the crude mixture using 1,3,5-trimethylbenzene as internal standard. Reaction times 0.5 h, 1 h, 1.5 h, 2 h, 2.5 h, 3 h, 5 h, 7 h, 9 h, 11 h,14 h, 17 h were plotted for the increase of product with time and decrease of feedstock ethyl p-tolylglycinate with time.



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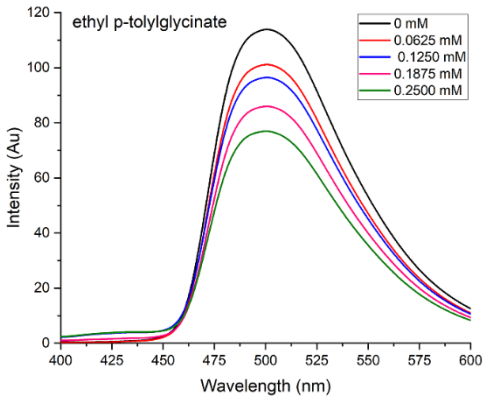
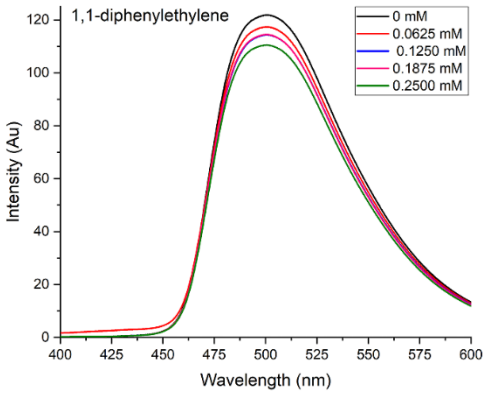
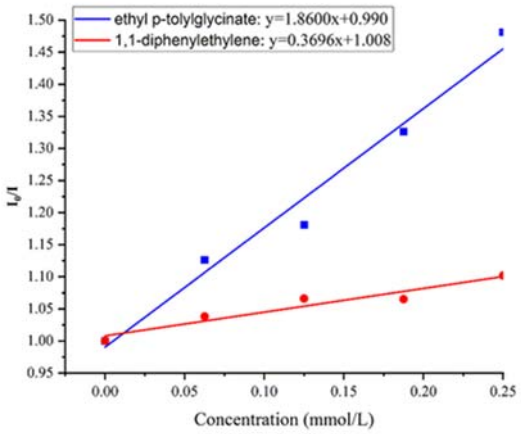
Stern-Volmer luminescence quenching analysis [5]

DMSO was degassed with a stream of argon for 1 h. $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ (3.4 mg, 15 μmol) was dissolved in 200 mL DMSO to prepare a 1.5×10^{-5} M solution. 9.5 mL of this solution was added to each of a set of 5 volumetric flasks (10 mL). Subsequently, the solution of quencher ethyl p-tolylglycinate or 1,1-diphenylethylene in DMSO (1.0 mL, 0.0125 M) was added in increasing amounts (0, 50 μL, 100 μL, 150 μL and 200 μL) to the volumetric flasks and the volume of volumetric flasks were adjusted to 10 mL by adding DMSO. Emission intensities were recorded by using F-4700 Fluorescence Spectrometer. All solutions were excited at 380 nm and the fluorescence emission spectra were recorded. The ratio of the maximum fluorescence emission intensities maximum between samples without and with quencher were plotted against the quencher concentration to generate the Stern-Volmer plots below.

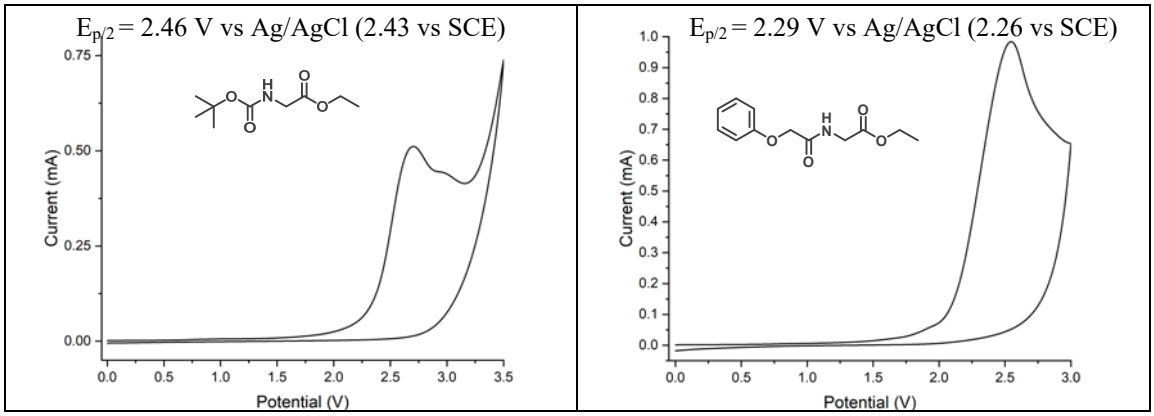




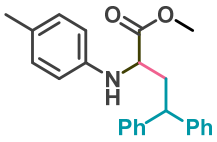


Cyclic voltammetry study

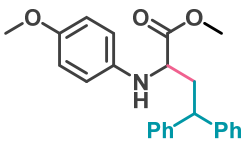
Cyclic voltammograms were performed on a CH Instruments Electrochemical Workstation model CHI660E at room temperature. Samples were prepared with 0.2 mmol of substrate in 10 mL of 0.1 M tetrabutylammonium hexafluorophosphate in dry, degassed acetonitrile. Measurements employed a glassy carbon working electrode, a Pt counter electrode, and an Ag/AgCl reference electrode, and a scan rate of 100 mV/s. The obtained value was referenced to Ag/AgCl and converted to SCE by subtracting 0.03 V. [6]



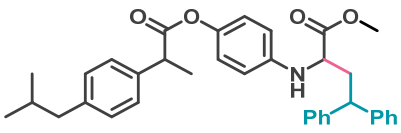
Characterization of the products



Methyl 4,4-diphenyl-2-(p-tolylamino)butanoate (C1). The desired pure product was obtained in 76% yield (54.6 mg) as a white solid. m.p. = 132 - 133 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 - 7.23 (m, 8H), 7.23 - 7.16 (m, 2H), 6.92 (d, *J* = 6.7 Hz, 2H), 6.37 (d, *J* = 8.5 Hz, 2H), 4.31 - 4.25 (m, 1H), 4.01 - 3.86 (m, 2H), 3.62 (s, 3H), 2.63 - 2.55 (m, 1H), 2.40 - 2.32 (m, 1H), 2.20 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.7, 144.6, 144.2, 143.4, 129.7, 128.6, 128.5, 128.1, 127.8, 126.5, 126.4, 114.0, 55.6, 52.0, 47.2, 39.1, 20.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{24}H_{26}NO_2$ 360.1958; found 360.1957.

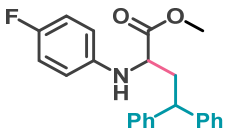


Methyl 2-((4-methoxyphenyl)amino)-4,4-diphenylbutanoate (C2). The desired pure product was obtained in 81% yield (64.0 mg) white solid. m.p. = 93 - 95 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 - 7.25 (m, 8H), 7.22 - 7.17 (m, 2H), 6.69 (d, J = 8.9 Hz, 2H), 6.42 (d, J = 8.9 Hz, 2H), 4.30 (dd, J = 9.4, 6.1 Hz, 1H), 3.84 (dd, J = 9.0, 5.1 Hz, 1H), 3.79 (s, 1H), 3.71 (s, 3H), 3.62 (s, 3H), 2.61 - 2.55(m, 1H), 2.37 - 2.31 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.0, 152.9, 144.2, 143.3, 140.9, 128.7, 128.5, 128.1, 127.8, 126.6, 126.4, 115.6, 114.8, 56.5, 55.6, 52.0, 47.2, 39.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{24}H_{26}NO_3$ 376.1907; found 376.1908.

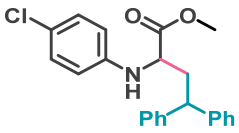


Methyl 2-((4-((2-(4-isobutylphenyl)propanoyl)oxy)phenyl)amino)-4,4-diphenylbutanoate (C3). The desired pure product was obtained in 80% yield (87.8mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 - 7.21 (m, 10H), 7.20 - 7.16 (m, 2H), 7.11 (d, *J* = 8.1 Hz, 2H), 6.73 (d, *J* = 8.9 Hz, 2H), 6.35 (d, *J* = 8.8 Hz, 2H), 4.24 (dd, *J* = 9.5, 6.5 Hz, 1H), 4.01 (s, 1H), 3.89 - 3.84(m, 2H), 3.61 (s, 2H), 2.61 - 2.55 (m, 1H), 2.46 (d, *J* = 7.1 Hz, 2H), 2.37 - 2.31 (m, 1H), 1.89 - 1.81 (m, 1H), 1.56 (d, *J* = 7.1 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.4, 173.6, 144.6, 144.1, 143.2 143.0, 140.6, 137.4, 129.4, 128.7, 128.5, 128.0,

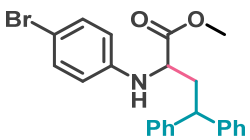
127.7, 127.2, 126.6, 126.5, 121.9, 114.2, 55.6, 52.1, 47.2, 45.2, 45.0, 39.0, 30.1, 22.4, 18.5. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{36}H_{40}NO_4$ 550.2952; found 550.2950.



Methyl 2-((4-fluorophenyl)amino)-4,4-diphenylbutanoate (C4). The desired pure product was obtained in 71% yield (51.6 mg) a white solid. m.p. = 136 - 138 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.23 - 7.16 (m, 8H), 7.15 - 7.09 (m, 2H), 6.75 - 6.70 (m, 2H), 6.31 - 6.26 (m, 2H), 4.19 (dd, *J* = 7.6, 6.9 Hz, 1H), 3.85 (s, 1H), 4.21 - 4.17 (m, 1H), 3.55 (s, 3H), 2.55 - 2.50 (m, 1H), 2.31 - 2.24 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.5, 156.4 (d, *J* = 236.4 Hz), 144.1, 143.2, 143.2 (d, *J* = 1.9 Hz), 128.7, 128.6, 128.0, 127.8, 126.6, 126.5, 115.7 (d, *J* = 22.5 Hz), 115.0 (d, *J* = 7.5 Hz), 56.1, 52.1, 47.2, 39.0. ^{19}F NMR (376 MHz, $CDCl_3$) δ -126.45. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{23}H_{23}FNO_2$ 364.1707; found 364.1708.

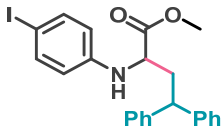


Methyl 2-((4-chlorophenyl)amino)-4,4-diphenylbutanoate(C5). The desired pure product was obtained in 66% yield (50.1 mg) a white solid. m.p. = 155 - 157 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 - 7.20 (m, 10H), 7.06 (d, *J* = 8.8 Hz, 2H), 6.36 (d, *J* = 8.9 Hz, 2H), 4.26 (dd, *J* = 9.2, 6.2 Hz, 1H), 4.08 (s, 1H), 3.93 - 3.88 (m, 1H), 3.66 (s, 3H), 2.66 - 2.60 (m, 1H), 2.42 - 2.35 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 145.4, 144.0, 143.1, 129.1, 128.7, 128.6, 128.0, 127.8, 126.7, 126.5, 123.1, 114.9, 55.3, 52.2, 47.2, 38.9. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{23}H_{23}ClNO_2$ 380.1412; found 380.1413.

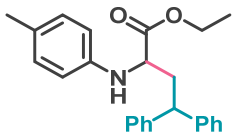


Methyl 2-((4-bromophenyl)amino)-4,4-diphenylbutanoate (C6). The desired pure product was obtained in 78% yield (66.6 mg) a white solid. m.p. = 146 - 148 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 - 7.16 (m, 10H), 7.17 (d, *J* = 8.8 Hz, 2H), 6.28 (d, *J* = 8.8 Hz, 2H), 4.23 (dd, *J* = 9.4, 6.4

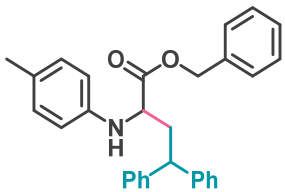
Hz, 1H), 4.07 (d, J = 9.8 Hz, 1H), 3.90 - 3.85 (m, 1H), 3.63 (s, 3H), 2.64 - 2.58 (m, 1H), 2.39 - 2.33 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 145.8, 143.9, 143.1, 131.9, 128.7, 128.6, 128.0, 127.7, 126.7, 126.5, 115.3, 110.2, 55.1, 52.2, 47.2, 38.9. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{23}H_{23}BrNO_2$ 424.0907; found 424.0906.



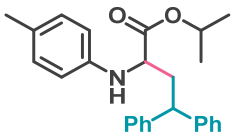
Methyl 2-((4-iodophenyl)amino)-4,4-diphenylbutanoate (C7). The desired pure product was obtained in 28% yield (26.3 mg) as yellow solid. m.p. = 157 - 159 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 (d, J = 8.8 Hz, 2H), 7.31 - 7.19 (m, 10H), 6.20 (d, J = 8.8 Hz, 2H), 4.22 (dd, J = 9.2, 6.4 Hz, 1H), 4.07 (s, 1H), 3.91 - 3.86 (m, 1H), 3.65 (s, 3H), 2.64 - 2.58 (m, 1H), 2.40 - 2.33 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.1, 146.5, 143.9, 143.1, 137.8, 128.7, 128.6, 128.0, 127.7, 126.7, 126.6, 115.9, 79.5, 55.0, 52.2, 47.2, 38.9. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{23}H_{23}INO_2$ 472.0768; found 472.0766.



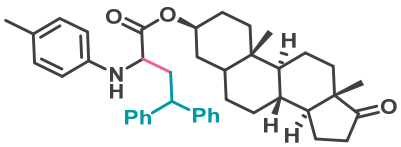
Ethyl 4,4-diphenyl-2-(p-tolylamino)butanoate (C8). The desired pure product was obtained in 82% yield (61.2 mg) as a white solid. m.p. = 113 - 115 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 - 7.23 (m, 8H), 7.21 - 7.17 (m, 2H), 6.91 (d, J = 8.3 Hz, 2H), 6.37 (d, J = 8.4 Hz, 2H), 4.28 (dd, J = 9.1, 6.5 Hz, 1H), 4.13 - 4.04 (m, 2H), 3.94 - 3.86 (m, 1H), 2.60 - 2.54 (m, 1H), 2.40 - 2.34 (m, 1H), 2.20 (s, 3H), 1.21 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 144.6, 144.2, 143.4, 129.7, 128.6, 128.5, 128.1, 127.8, 127.7, 126.5, 126.4, 114.1, 61.0, 55.7, 47.3, 39.2, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{25}H_{28}NO_2$ 374.2115; found 374.2114.



Benzyl 4,4-diphenyl-2-(p-tolylamino)butanoate (C9). The desired pure product was obtained in 75% yield (65.2 mg) as a white solid. m.p. = 166 - 168 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 - 7.35 (m, 3H), 7.35 - 7.21 (m, 10H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.44 (d, *J* = 8.0 Hz, 2H), 5.12 (s, 2H), 4.29 (t, *J* = 8.6 Hz, 1H), 4.07 - 3.96 (m, 2H), 2.69 - 2.59 (m, 1H), 2.50 - 2.41 (m, 1H), 2.27 (s, 3H).^{13}C NMR (151 MHz, $CDCl_3$) δ 174.1, 144.5, 144.0, 143.4, 135.5, 129.7, 128.6, 128.5, 128.5, 128.2, 128.2, 128.0, 127.8, 127.8, 126.5, 126.4, 114.1, 66.7, 55.7, 47.2, 39.1, 20.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{30}H_{30}NO_2$ 436.2271; found 436.2269.

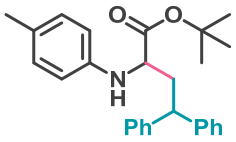


Isobutyl 4,4-diphenyl-2-(p-tolylamino)butanoate (C10). The desired pure product was obtained in 75% yield (50.4 mg) as a white solid. m.p. = 121 - 123 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 - 7.23 (m, 8H), 7.23 - 7.17 (m, 2H), 6.90 (d, *J* = 6.4 Hz, 2H), 6.36 (d, *J* = 6.2 Hz, 2H), 5.01 - 4.93 (m, 1H), 4.33 – 4.25 (m, 1H), 3.92 (s, 1H), 3.87 – 3.81 (m, 1H), 2.59 - 2.50 (m, 1H), 2.41 - 2.31 (m, 1H), 2.20 (s, 3H), 1.19 (d, *J* = 6.2 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.8, 144.6, 144.2, 143.5, 129.6, 128.6, 128.5, 128.0, 127.9, 127.7, 126.5, 126.4, 114.1, 68.6, 55.8, 47.3, 39.2, 21.8, 21.7, 20.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{26}H_{30}NO_2$ 388.2271; found 388.2272.

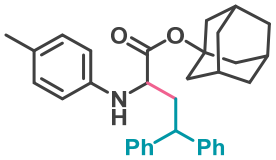


(3S,8R,9S,10S,13S,14S)-10,13-dimethyl-17-oxohexadecahydro-1H-cyclopenta[a]phenanthren-3-yl 4,4-diphenyl-2-(p-tolylamino)butanoate (C11). The desired pure product was obtained in 64% yield (78.7 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 - 7.23 (m, 8H), 7.23 - 7.17 (m, 2H) 6.90 (d, *J* = 8.0 Hz, 2H), 6.36 (d, *J* = 8.3 Hz, 2H), 4.71 - 4.61 (m, 1H), 4.32 - 4.25 (m, 1H), 3.91 (s, 1H), 3.83 (s, 1H), 2.58 - 2.49 (m, 1H), 2.48 - 2.31 (m, 2H), 2.20 (s, 3H), 2.12 - 2.00 (m, 1H), 1.97 - 1.87 (m, 1H), 1.83 - 1.69 (m, 4H), 1.67-1.42 (m, 6H), 1.38 - 1.16 (m, 6H), 1.05 - 0.91 (m, 2H), 0.85 (d, *J* = 4.4 Hz, 6H), 0.74 - 0.65 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 221.1, 173.8, 173.8, 144.6, 144.6, 144.2, 144.2, 143.5, 143.5, 129.6, 128.6, 128.5, 128.1, 128.0, 127.9, 127.7, 127.7, 127.7, 126.5, 126.4, 114.1, 114.1,

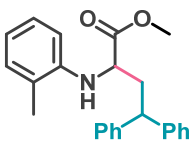
74.3, 55.8, 54.2, 51.3, 47.7, 47.3, 44.6, 44.6, 39.3, 36.6, 36.6, 35.8, 35.6, 35.0, 33.8, 33.8, 31.5, 30.8, 30.7, 28.2, 28.2, 27.4, 27.3, 21.7, 20.4, 20.4, 13.8, 12.2. HRMS (ESI-Q-TOF) $[M + H]^+$ calcd for $C_{42}H_{52}NO_3$ 618.3942; found 618.3940.



Neopentyl 4,4-diphenyl-2-(p-tolylamino)butanoate (C12). The desired pure product was obtained in 72% yield (57.7 mg) as a white solid. m.p. = 147 - 149 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 - 7.26 (m, 8H), 7.25 - 7.19 (m, 2H), 6.92 (d, J = 8.3 Hz, 2H), 6.36 (d, J = 8.5 Hz, 2H), 4.34 - 4.27 (m, 1H), 3.89 (s, 1H), 3.78 (d, J = 7.0 Hz, 1H), 2.58 - 2.52 (m, 1H), 2.38 - 2.30 (m, 1H), 2.22 (s, 3H), 1.43 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.5, 144.8, 144.3, 143.6, 129.6, 128.6, 128.5, 128.1, 127.9, 127.5, 126.5, 126.4, 114.1, 81.5, 56.2, 47.4, 39.4, 28.0, 20.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{27}H_{32}NO_2$ 402.2428; found 402.2427.

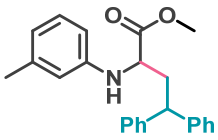


(3s,5s,7s)-adamantan-1-yl 4,4-diphenyl-2-(p-tolylamino)butanoate (C13). The desired pure product was obtained in 60% yield (57.5 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 - 7.24 (m, 8H), 7.23 - 7.17 (m, 2H), 6.90 (d, J = 8.3 Hz, 2H), 6.33 (d, J = 8.4 Hz, 2H), 4.34 - 4.25 (m, 1H), 3.91 (s, 1H), 3.80 - 3.73 (m, 1H), 2.57 - 2.49 (m, 1H), 2.38 - 2.28 (m, 1H), 2.20 (s, 3H), 2.14 (s, 3H), 2.07 (s, 6H), 1.64 (s, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.2, 144.8, 144.3, 143.6, 129.6, 128.6, 128.5 128.1, 127.9, 127.4, 126.4, 126.4, 114.1, 81.6, 56.2, 47.4, 41.3, 39.5, 36.1, 30.8, 20.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{33}H_{38}NO_2$ 480.2897; found 480.2896.

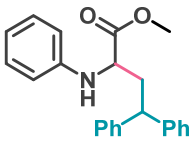


Methyl 4,4-diphenyl-2-(o-tolylamino)butanoate (C14). The desired pure product was obtained in 75% yield (53.9 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 - 7.22 (m, 8H),

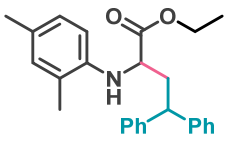
7.22 - 7.16 (m, 2H), 7.03 (d, J = 7.3 Hz, 1H), 6.99 - 6.94 (m, 1H), 6.68 - 6.63 (m, 1H), 6.24 (d, J = 8.0 Hz, 1H), 4.31 - 4.21 (m, 1H), 4.03 (dd, J = 8.1, 5.3 Hz, 1H), 3.63 (s, 3H), 2.70 - 2.61 (m, 1H), 2.51 - 2.43 (m, 1H), 2.13 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.7, 144.8, 144.2, 143.5, 130.3, 128.7, 128.6, 128.0, 127.8, 127.0, 126.6, 126.5, 122.9, 118.1, 110.7, 55.2, 52.1, 47.4, 39.2, 17.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{24}H_{26}NO_2$ 360.1958; found 360.1957.



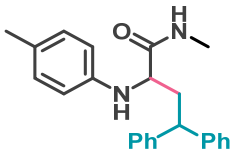
Methyl 4,4-diphenyl-2-(m-tolylamino)butanoate (C15). The desired pure product was obtained in 78% yield (56.1 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 - 7.23 (m, 8H), 7.23 - 7.17 (m, 2H) 7.02 - 6.96 (m, 1H), 6.54 (d, J = 7.4 Hz, 1H), 6.28 - 6.23 (m, 2H), 4.27 (dd, J = 9.3, 6.4 Hz, 1H), 4.05 - 3.90 (m, 2H), 3.64 (s, 3H), 2.64 - 2.55 (m, 1H), 2.41 - 2.33 (m, 1H), 2.20 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.6, 146.9, 144.2, 143.3, 139.0, 129.1, 128.6, 128.5, 128.1, 127.8, 126.5, 126.4, 119.5, 114.6, 110.9, 55.1, 52.1, 47.2, 39.2, 21.5. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{24}H_{26}NO_2$ 360.1958; found 360.1959.



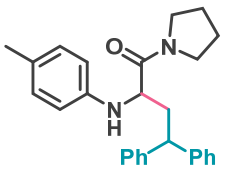
Methyl 4,4-diphenyl-2-(phenylamino)butanoate (C16). The desired pure product was obtained in 58% yield (40.0 mg) as a white solid. m.p. = 125- 127 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 - 7.23 (m, 8H), 7.22 - 7.16 (m, 2H), 7.14 - 7.07 (m, 2H), 6.74 – 6.68 (m, 1H), 6.45 (d, J = 7.6 Hz, 2H), 4.30 - 4.23 (m, 1H), 4.05 (s, 1H), 3.95 (s, 1H), 3.63 (s, 3H) 2.65 - 2.57 (m, 1H), 2.42 - 2.36 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.5, 146.8, 144.1, 143.3, 129.2, 128.7, 128.6, 128.0, 127.8, 126.6, 126.5, 118.5, 113.8, 55.2, 52.1, 47.2, 39.1. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{23}H_{24}NO_2$ 346.1802; found 346.1804.



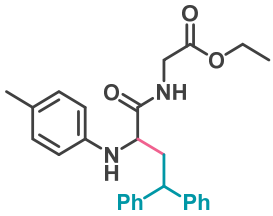
methyl 2-((2,4-dimethylphenyl)amino)-4,4-diphenylbutanoate (C17). The desired pure product was obtained in 62% yield (48.1 mg) as a white solid. m.p. = 102- 104 ℃ . ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 - 7.23 (m, 8H), 7.23 - 7.16 (m, 2H), 6.85 (d, *J* = 5.3 Hz, 1H), 6.26 (s, 1H), 6.21 (d, *J* = 7.6 Hz, 1H), 4.33 - 4.25 (m, 1H), 4.13 - 4.03 (m, 2H), 3.91 - 3.83 (m, 1H), 2.64 - 2.50 (m, 1H), 2.41 - 2.26 (m, 1H), 2.11 (s, 6H), 1.22 (t, *J* = 7.0 Hz, 3H).^{13}C NMR (151 MHz, $CDCl_3$) δ 174.3, 145.0 144.3, 143.5, 137.3, 130.2, 128.6, 128.5, 128.2, 128.1, 127.8, 126.5, 126.4, 115.8, 111.4, 61.0, 55.6, 47.2, 39.3, 19.9, 18.7, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{26}H_{30}NO_2$ 388.2271; found 388.2272.



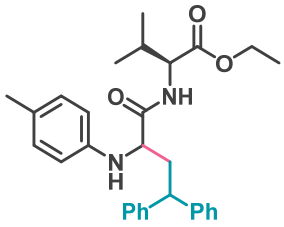
N-methyl-4,4-diphenyl-2-(p-tolylamino)butanamide (C18). The desired pure product was obtained in 58% yield (41.6 mg) as a white solid. m.p. = 193- 195 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 - 7.11 (m, 10H), 6.94 (d, *J* = 8.0 Hz, 2H), 6.76 (s, 1H), 6.27 (d, *J* = 8.0 Hz, 2H), 4.16 - 4.03 (m, 1H), 3.78 (s, 1H), 3.63 (dd, *J* = 9.3, 3.9 Hz, 1H), 2.89 - 2.79 (m, 1H), 2.76 (d, *J* = 4.9 Hz, 3H), 2.45 - 2.33 (m, 1H), 2.23 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.0, 144.3 144.1, 143.8, 129.8, 128.8, 128.7, 128.4, 127.8, 127.8, 126.6, 126.54, 113.7, 59.1, 48.7, 39.1, 26.0, 20.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{24}H_{27}N_2O$ 359.2118; found 359.2118.



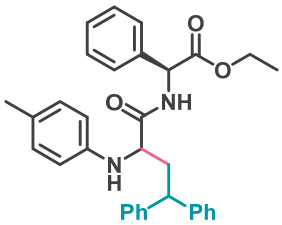
4,4-diphenyl-1-(pyrrolidin-1-yl)-2-(p-tolylamino)butan-1-one (C19). The desired pure product was obtained in 74% yield (58.9 mg) as a white solid. m.p. = 129- 131 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 - 7.25 (m, 8H), 7.23 - 7.11 (m, 2H), 6.92 (d, *J* = 8.4 Hz, 2H), 6.40 (d, *J* = 8.4 Hz, 2H), 4.33 - 4.26 (m, 1H), 4.22 (s, 1H), 3.94 - 3.87 (m, 1H), 3.45 - 3.30 (m, 2H), 3.10 - 3.03 (m, 1H), 2.92 - 2.84 (m, 1H), 2.43 - 2.33 (m, 2H), 2.21 (s, 3H), 1.81 - 1.69 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.8, 145.2, 144.2, 144.2, 129.7, 128.5, 128.5, 128.2, 127.7, 127.6, 126.5, 126.3, 114.5, 54.3, 47.3, 45.7, 45.6, 39.0, 25.9, 24.0, 20.3. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{27}H_{31}N_2O$ 399.2431; found 399.2432.



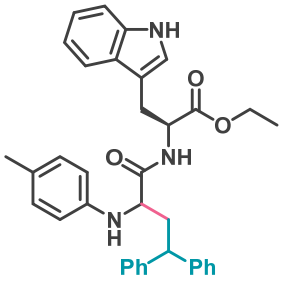
Ethyl (4,4-diphenyl-2-(p-tolylamino)butanoyl)glycinate (C20). The desired pure product was obtained in 43% yield (40.1 mg) as a white solid. m.p. = 121- 123 °C. ^{1}H NMR (600 MHz, $CDCl_3$) 7.29 - 7.24 (m, 4H), 7.24 - 7.14 6.93 (m, 6H), (d, *J* = 8.6 Hz, 2H), 6.30 (d, *J* = 8.5 Hz, 2H), 4.18 - 4.13 (m, 3H), 4.05 (dd, *J* = 18.2, 6.3 Hz, 1H), 3.84 (dd, *J* = 18.1, 5.0 Hz, 1H), 3.71 (s, 1H), 3.68 (d, *J* = 5.5 Hz, 1H), 2.84 - 2.77 (m, 1H), 2.47 - 2.37 (m, 1H), 2.22 (s, 3H), 1.23 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.9, 169.6, 144.2, 144.1, 143.6, 129.7, 128.7, 128.6, 128.2, 127.8, 127.7, 126.5, 113.7, 61.3, 58.7, 48.3, 41.0, 39.0, 20.3, 14.0. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{27}H_{31}N_2O_3$ 431.2329; found 431.2329.



Ethyl (4,4-diphenyl-2-(p-tolylamino)butanoyl)-L-valinate (C21). The desired pure product was obtained in 44% yield (41.4 mg, dr = 1.2:1) as a white solid. m.p. = 149-151 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.37 - 7.13 (m, 10H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.31 (dd, *J* = 23.2, 8.3 Hz, 2H), 4.51 - 4.46 (m, 1H), 4.22 - 4.13 (m, 2H), 4.09 (q, *J* = 7.2 Hz, 1H), 3.74 - 3.48 (m, 2H), 2.86 - 2.75 (m, 1H), 2.47 - 2.36 (m, 1H), 2.21 (s, 3H), 2.17 - 2.04 (m, 1H), 1.20 (m, 3H), 0.87 (dd, *J* = 32.9, 6.8 Hz, 3H), 0.75 (dd, *J* = 65.0, 6.8 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.6, 173.4, 171.8, 171.3, 144.3, 144.2, 144.1, 144.0, 143.6, 143.5, 129.7, 129.6, 128.8, 128.7, 128.7, 128.3, 128.2, 127.9, 127.8, 127.8, 127.7, 126.6, 126.5, 126.5, 114.2, 113.7, 61.2, 61.1, 59.1, 59.0, 57.1, 56.8, 48.4, 48.4, 39.2, 39.1, 31.1, 31.1, 20.4, 20.4, 19.0, 17.7, 17.4, 14.2, 14.0. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{30}H_{37}N_2O_3$ 473.2799; found 473.2798.

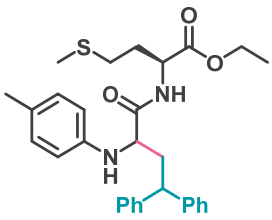


Ethyl (2S)-2-(4,4-diphenyl-2-(p-tolylamino)butanamido)-2-phenylacetate (C22). The desired pure product was obtained in 45% yield (45.5mg, dr = 1:1) as a white solid. m.p. = 180 -182 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (dd, *J* = 77.2, 7.6 Hz, 1H), 7.38 - 7.10 (m, 14H), 6.92 (dd, *J* = 22.0, 8.0 Hz, 2H), 6.30 (dd, *J* = 43.2, 8.4 Hz, 2H), 5.54 - 5.46 (m, 1H), 4.25 - 4.03 (m, 3H), 3.73 - 3.61 (m, 2H), 2.84 - 2.69 (m, 1H), 2.49 - 2.33 (m, 1H), 2.23 (d, *J* = 5.1 Hz, 3H), 1.21 - 1.11 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.11, 173.06, 170.6, 170.3, 144.14, 144.08, 143.58, 143.56, 136.42, 136.36, 129.69, 129.65, 128.9, 128.73, 128.69, 128.6, 128.44, 128.36, 128.3, 127.9, 127.84, 127.75, 127.2, 126.6, 126.5, 114.2, 114.0, 61.8, 61.7, 58.87, 58.86, 56.5, 56.3, 48.4, 48.3, 39.2, 38.9, 20.41, 20.37, 14.0, 13.9. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{33}H_{35}N_2O_3$ 507.2642; found 507.2643.

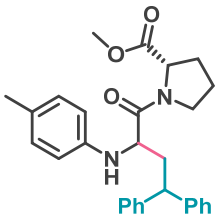


Ethyl (4,4-diphenyl-2-(p-tolylamino)butanoyl)-L-tryptophanate (C23). The desired pure product was obtained in 67% yield (74.9 mg, dr = 1.1:1) as a yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 117.2 Hz, 1H), 7.45 (dd, *J* = 72.1, 7.9 Hz, 1H), 7.31 - 7.23 (m, 3H), 7.23 - 7.13 (m, 7H), 7.13 - 7.09 (m, 1H), 7.09 - 6.97 (m, 1H), 6.91 - 6.87 (m, 2H), 6.62 (d, *J* = 238.6 Hz, 1H), 6.22 - 6.17 (m, 2H), 4.92 - 4.85 (m, 1H), 4.15 - 4.02 (m, 3H), 3.66 - 3.42 (m, 2H), 3.34 - 3.26 (m, 1H), 3.24 - 3.10 (m, 1H), 2.77 - 2.59 (m, 1H), 2.23 (d, *J* = 21.7 Hz, 3H), 2.35 - 2.10 (m, 1H), 1.21 - 1.03 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.3, 171.8, 171.5, 144.23, 144.17, 144.1, 144.0, 143.6, 143.5, 136.0, 129.6, 129.5, 128.7, 128.59, 128.56, 128.0, 127.9, 127.8, 127.7, 127.7, 127.3, 126.5, 126.4, 123.0, 122.7, 122.1, 122.0, 119.51, 119.47, 118.54, 118.47, 114.0, 113.5, 111.3, 111.0, 110.10, 110.08, 109.5, 61.4, 61.3, 58.7, 58.6, 52.9, 52.04,

52.02, 48.3, 48.2, 39.0, 38.7, 27.5, 27.4, 20.39, 20.35, 14.0, 13.9. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{36}H_{38}N_3O_3$ 560.2908; found 560.2908.

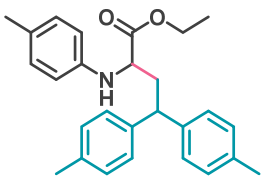


Ethyl (4,4-diphenyl-2-(p-tolylamino)butanoyl)-L-methioninate (C24). The desired pure product was obtained in 43% yield (43.4 mg, dr = 1.3:1) as a white solid. m.p. = 102 -104 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 - 7.14 (m, 10H), 6.93 (d, *J* = 8.0 Hz, 2H), 6.31 (dd, *J* = 34.4, 8.5 Hz, 2H), 4.70 - 4.58 (m, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.11 (q, *J* = 7.1 Hz, 1H), 3.81 - 3.56 (m, 2H), 2.83 - 2.75 (m, 1H), 2.44 - 2.38 (m, 2H), 2.28 -2.24 (m, 1H), 2.21 (d, *J* = 2.4 Hz, 3H), 2.16 - 2.04 (m, 1H), 1.96 (d, *J* = 65.0 Hz, 3H), 1.95 - 1.80 (m, 1H), 1.28 - 1.14 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.6, 173.4, 171.7, 171.3, 144.2, 144.1, 144.0, 143.9, 143.51, 143.49, 129.7, 129.6, 128.73, 128.70, 128.6, 128.3, 128.2, 127.84, 127.81, 127.72, 127.69, 126.6, 126.5, 114.1, 113.5, 61.53, 61.46, 58.8, 58.7, 51.5, 51.1, 48.4, 48.3, 39.1, 38.9, 31.51, 31.46, 30.0, 29.7, 20.4, 20.3, 15.4, 15.2, 14.1, 14.0. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{30}H_{37}N_2O_3S$ 505.2519; found 505.2518.

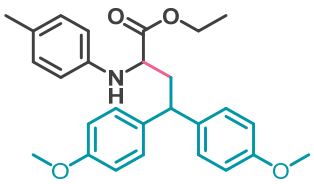


Methyl (4,4-diphenyl-2-(p-tolylamino)butanoyl)-L-prolinate (C25). The desired pure product was obtained in 56% yield (51.1 mg, dr = 6:1) as a yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 - 7.23 (m, 8H), 7.22 - 7.16 (m, 2H), 6.94 (d, *J* = 8.1 Hz, 2H), 6.44 - 6.27 (m, 2H), 4.38 (dd, *J* = 8.1, 2.7 Hz, 1H), 4.24 (t, *J* = 7.9 Hz, 1H), 4.05 (s, 1H), 3.97 - 3.88 (m, 1H), 3.54 (d, *J* = 17.8 Hz, 3H), 3.36 - 3.25 (m, 1H), 3.00 - 2.93 (m, 1H), 2.49 - 2.35 (m, 2H), 2.22 (s, 3H), 2.14 - 1.98 (m, 2H), 1.96 - 1.85 (m, 2H), 1.85 - 1.74 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.3, 172.0, 144.8, 144.2, 144.0, 129.8, 129.6, 128.53, 128.49, 128.1, 127.8, 126.47, 126.45, 114.3, 59.0,

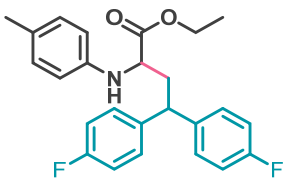
54.2, 52.0, 47.4, 46.1, 38.6, 28.9, 24.6, 20.4. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{29}H_{33}N_2O_3$ 457.2486; found 457.2486.



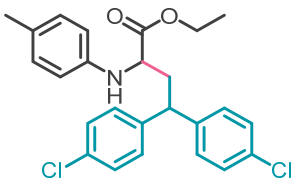
Ethyl 4,4-di-p-tolyl-2-(p-tolylamino)butanoate (C26). The desired pure product was obtained in 37% yield (29.7 mg) as a white solid. m.p. = 107 -109 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 - 7.02 (m, 8H), 6.91 (d, J = 8.0 Hz, 2H), 6.38 (d, J = 8.2 Hz, 2H), 4.23 – 4.17 (m, 1H), 4.09 (q, J = 7.1 Hz, 1H), 3.98 - 3.83 (m, 2H), 2.57 - 2.48 (m, 1H), 2.38 - 2.31 (m, 1H), 2.30 (s, 6H), 2.21 (s, 3H), 1.21 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.4, 144.5, 141.5, 140.7, 135.9, 135.8, 129.7, 129.3, 129.2, 127.8, 127.7, 127.6, 114.1, 61.0, 55.7, 46.5, 39.3, 21.0, 21.0, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{27}H_{32}NO_2$ 402.2428; found 402.2427.



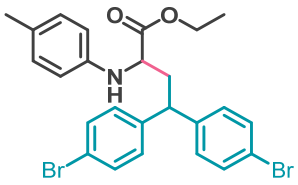
Ehyl 4,4-bis(4-methoxyphenyl)-2-(p-tolylamino)butanoate (C27). The desired pure product was obtained in32% yield (27.7 mg) as a white solid. m.p. = 82 -84 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 - 7.04 (m, 4H), 6.84 (d, J = 7.9 Hz, 2H), 6.75 (d, J = 7.6 Hz, 4H), 6.31 (d, J = 7.2 Hz, 2H), 4.14 - 4.08(m, 1H), 4.02 (q, J = 7.2 Hz, 1H), 3.81 - 3.76 (m, 1H), 3.69 (s, 6H), 2.46 - 2.37 (m, 1H), 2.26 - 2.12 (m, 1H), 2.13 (s, 3H), 1.13 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.4, 158.1, 158.0, 144.7, 136.8, 135.9, 129.7, 128.9, 128.6, 127.7, 114.1, 114.0, 113.9, 61.0, 55.7, 55.2, 55.2, 45.7, 39.6, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{27}H_{32}NO_4$ 434.2326; found 434.2325.



Ethyl 4,4-bis(4-fluorophenyl)-2-(p-tolylamino)butanoate (C28). The desired pure product was obtained in 65% yield (40.9 mg) as a white solid. m.p. = 93 -95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 - 7.16 (m, 4H), 7.03 - 6.90 (m, 6H), 6.38 (d, *J* = 7.9 Hz, 2H), 4.28 (dd, *J* = 9.0, 6.0 Hz, 1H), 4.14 - 4.02 (m, 2H), 3.92 (s, 1H), 3.83 - 3.79 (m, 1H), 2.55 - 2.45 (m, 1H), 2.35 - 2.26 (m, 1H), 2.21 (s, 3H), 1.21 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, CDCl3) δ 174.1, 161.6 (d, J = 245.1 Hz), 161.5 (d, J = 245.1 Hz), 144.5, 139.8 (d, J = 3.3 Hz), 139.0 (d, J = 3.4 Hz), 129.8, 129.4 (d, J = 7.9 Hz), 129.2 (d, J = 7.6 Hz), 128.0, 115.5 (d, J = 22.4 Hz), 115.4 (d, J = 21.4 Hz), 114.1, 61.1, 55.5, 45.7, 39.4, 20.4, 14.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ -116.22, -116.40. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{25}H_{26}F_2NO_2$ 410.1926; found 410.1928.

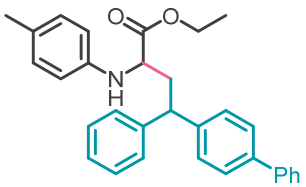


Ethyl 4,4-bis(4-chlorophenyl)-2-(p-tolylamino)butanoate (C29). The desired pure product was obtained in 59% yield (52.0 mg) as a white solid. m.p. = 114 - 116 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 - 7.21 (m, 4H), 7.19 - 7.12 (m, 4H), 6.93 (d, J = 8.0 Hz, 2H), 6.39 (d, J = 8.3 Hz, 2H), 4.26 (dd, J = 9.4, 6.0 Hz, 1H), 4.15 - 4.04 (m, 2H), 3.94 (s, 1H), 3.82 (dd, J = 8.8, 5.0 Hz, 1H), 2.55 - 2.45 (m, 1H), 2.34 - 2.25 (m, 1H), 2.21 (s, 3H), 1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.9, 144.4, 142.2, 141.4, 132.5, 132.4, 129.8, 129.3, 129.1, 128.9, 128.7, 128.1, 114.1, 61.2, 55.4, 45.9, 38.9, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{25}H_{26}Cl_2NO_2$ 442.1335; found 442.1335.

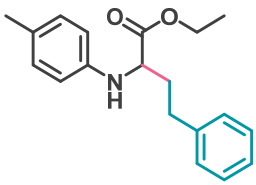


Ethyl 4,4-bis(4-bromophenyl)-2-(p-tolylamino)butanoate (C30). The desired pure product was obtained in 45% yield (47.8mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$)δ 7.42 (d, *J* = 8.2 Hz, 4H), 7.13 - 7.06 (m, 4H), 6.94 (d, *J* = 8.0 Hz, 2H), 6.39 (d, *J* = 8.0 Hz, 2H), 4.24 (dd, *J* = 9.5, 6.2 Hz, 1H), 4.15 - 4.04 (m, 2H), 3.92 (s, 1H), 3.82 (dd, *J* = 8.8, 5.2 Hz, 1H), 2.54 - 2.45 (m, 1H), 2.33 - 2.25 (m, 1H), 2.22 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz,

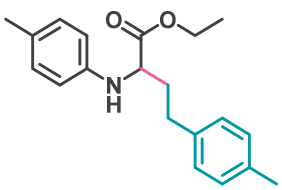
$CDCl_3$) δ 173.9, 144.4, 142.6, 141.9, 131.9, 131.7, 129.8, 129.8, 129.5, 128.1, 120.7, 120.5, 114.2, 61.2, 55.4, 46.1, 38.8, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{25}H_{26}Br_2NO_2$ 530.0324; found 530.0325.



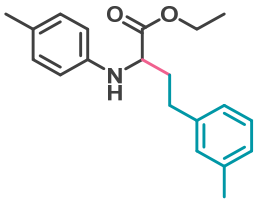
Ethyl 4-([1,1'-biphenyl]-4-yl)-4-phenyl-2-(p-tolylamino)butanoate (C31). The desired pure product was obtained in 52% yield (46.7 mg, dr = 6.1:1) as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.61 - 7.53 (m, 4H), 7.46 - 7.41 (m, 2H), 7.38 - 7.31 (m, 7H), 7.27 - 7.20 (m, 1H), 6.94 (d, *J* = 8.0 Hz, 2H), 6.42 (dd, *J* = 8.5, 2.6 Hz, 2H), 4.36 (dd, *J* = 9.1, 6.5 Hz, 1H), 4.12 - 4.07 (m, 2H), 3.98 - 3.92 (m, 2H), 2.68 - 2.59 (m, 1H), 2.48 - 2.36 (m, 1H), 2.23 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 144.6, 144.6, 144.1, 143.4, 143.3, 142.6, 140.8, 140.8, 139.4, 139.3, 129.7, 128.7, 128.7, 128.6, 128.5, 128.2, 128.1, 127.9, 127.8, 127.8, 127.3, 127.3, 127.1, 127.0, 127.0, 126.6, 126.5, 114.1, 114.1, 61.0, 55.70, 55.66, 47.0, 46.9, 39.2, 39.2, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{31}H_{32}NO_2$ 450.2428; found 450.2429.



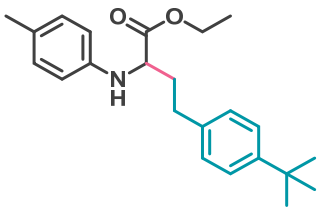
Ethyl 4-phenyl-2-(p-tolylamino)butanoate (C32). The desired pure product was obtained in 50% yield (29.7 mg) as a colorless oil. ^1H NMR (600 MHz, $CDCl_3$) δ 7.30 - 7.27 (m, 2H), 7.22 - 7.17 (m, 3H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.52 (d, *J* = 8.5 Hz, 2H), 4.16 (q, *J* = 7.2 Hz, 2H), 4.05 - 4.01 (m, 2H), 2.76 (t, *J* = 7.7 Hz, 2H), 2.23 (s, 3H), 2.19 - 2.11 (m, 1H), 2.08 - 1.99 (m, 1H), 1.24 (t, *J* = 7.1 Hz, 3H) .^{13}C NMR (151 MHz, $CDCl_3$) δ 174.1, 144.6, 141.0, 129.88, 128.5, 128.5, 127.6, 126.1, 113.8, 61.0, 56.5, 34.7, 31.8, 20.4, 14.3. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{19}H_{24}NO_2$ 298.1802; found 298.1803.



Ethyl 4-(p-tolyl)-2-(p-tolylamino)butanoate (C33). The desired pure product was obtained in 32% yield (19.9 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.11 -7.05 (m, 4H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.52 (d, *J* = 8.5 Hz, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 4.02 (t, *J* = 5.7 Hz, 1H), 2.75 - 2.69 (m, 2H), 2.32 (s, 3H), 2.22 (s, 3H), 2.16 - 2.08 (m, 1H), 2.05 - 1.97 (m, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 144.6, 137.9, 135.6, 129.8, 129.1, 128.4, 127.6, 113.8, 61.0, 56.5, 34.8, 31.4, 21.0, 20.4, 14.3. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{20}H_{26}NO_2$ 312.1958; found 312.1958.

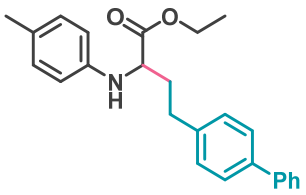


Ethyl 4-(m-tolyl)-2-(p-tolylamino)butanoate (C34). The desired pure product was obtained in 40% yield (24.8 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 - 7.14 (m, 1H), 7.03 - 6.94 (m, 5H), 6.52 (d, *J* = 8.4 Hz, 2H), 4.16 (q, *J* = 7.2 Hz, 2H), 4.02 (s, 2H), 2.72 (t, *J* = 7.9 Hz, 2H), 2.32 (s, 3H), 2.23 (s, 3H), 2.17 - 2.10 (m, 1H), 2.08 - 1.97 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 144.6, 140.9, 138.0, 129.8, 129.3, 128.4, 127.6, 126.8, 125.5, 113.8, 61.0, 56.6, 34.7, 31.8, 21.4, 20.4, 14.3. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{20}H_{26}NO_2$ 312.1958; found 312.1960.

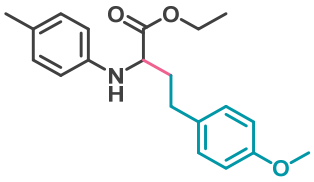


Ethyl 4-(4-(tert-butyl)phenyl)-2-(p-tolylamino)butanoate (C35). The desired pure product was obtained in 38% yield (26.8 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.2 Hz, 2H), 7.12 (d, *J* = 7.9 Hz, 2H), 6.97 (d, *J* = 7.9 Hz, 2H), 6.51 (d, *J* = 8.4 Hz, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 4.08 – 3.97 (m, 2H), 2.73 (t, *J* = 7.9 Hz, 2H), 2.22 (s, 3H), 2.19 - 2.11 (m, 1H), 2.08 - 1.98 (m, 1H), 1.31 (s, 9H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ

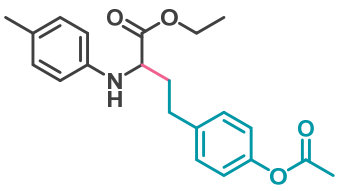
174.2, 148.9, 144.6, 137.9, 129.8, 128.1, 127.5, 125.3, 113.8, 61.0, 56.6, 34.6, 34.4, 31.4, 31.3, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{23}H_{32}NO_2$ 354.2428; found 354.2428.



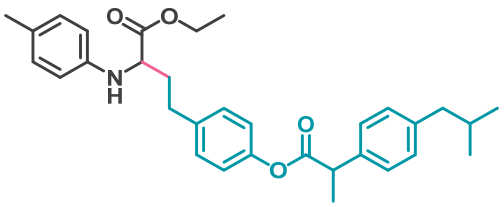
Ethyl 4-([1,1'-biphenyl]-4-yl)-2-(p-tolylamino)butanoate (C36). The desired pure product was obtained in 35% yield (26.1 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.3 Hz, 2H), 7.52 (d, *J* = 8.3 Hz, 2H), 7.45 - 7.41 (m, 2H), 7.35 - 7.31 (m, 1H), 7.26 (d, *J* = 8.4 Hz, 2H), 6.98 (d, *J* = 8.0 Hz, 2H), 6.53 (d, *J* = 8.4 Hz, 2H), 4.17 (q, *J* = 7.1 Hz, 2H), 4.08 - 4.03 (m, 2H), 2.81 (t, *J* = 7.9 Hz, 2H), 2.23 (s, 3H), 2.20 - 2.16 (m, 1H), 2.11 - 2.02 (m, 1H), 1.25 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.1, 144.6, 141.0, 140.1, 139.1, 129.8, 128.9, 128.7, 127.7, 127.2, 127.1, 127.0, 113.8, 61.1, 56.5, 34.7, 31.4, 20.4, 14.3. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{25}H_{28}NO_2$ 374.2115; found 374.2113.



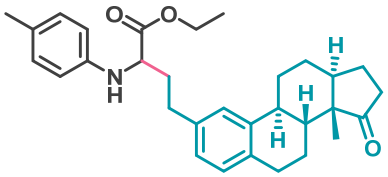
Ethyl 4-(4-methoxyphenyl)-2-(p-tolylamino)butanoate (C37). The desired pure product was obtained in 29% yield (19.0 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.6 Hz, 2H), 6.97 (d, *J* = 7.8 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 6.52 (d, *J* = 8.4 Hz, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 4.03 - 3.98 (m, 2H), 3.79 (s, 3H), 2.71 (t, *J* = 7.7 Hz, 2H), 2.23 (s, 3H), 2.16- 2.06 (m, 1H), 2.05 - 1.94 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H).^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 158.0, 144.6, 133.0, 129.8, 129.4, 127.6, 113.9, 113.8, 61.0, 56.4, 55.3, 34.9, 30.9, 20.4, 14.3. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{20}H_{26}NO_3$ 328.1907; found 328.1908.



Ethyl 4-(4-acetoxyphenyl)-2-(p-tolylamino)butanoate (C38). The desired pure product was obtained in 45% yield (31.9 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.2 Hz, 2H), 7.03 - 6.94 (m, 4H), 6.52 (d, *J* = 8.4 Hz, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 4.06 - 4.00 (m, 2H), 2.75 (t, *J* = 7.8 Hz, 2H), 2.28 (s, 3H), 2.23 (s, 3H), 2.18 - 2.10 (m, 1H), 2.09 - 1.93 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.0, 169.6, 149.0, 144.5, 138.5, 129.8, 129.4, 127.6, 121.5, 113.8, 61.1, 56.4, 34.6, 31.1, 21.1, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{26}NO_4$ 356.1856; found 356.1855.

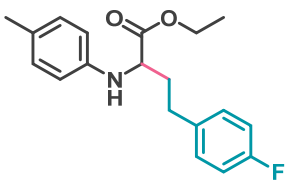


Ethyl 4-(4-((2-(4-isobutylphenyl)propanoyl)oxy)phenyl)-2-(p-tolylamino)butanoate (C39). The desired pure product was obtained in 30% yield (30.1 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 7.1 Hz, 4H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 8.5 Hz, 2H), 6.51 (d, *J* = 8.4 Hz, 2H), 4.14 (q, *J* = 7.1 Hz, 2H), 4.00 (s, 2H), 3.92 (q, *J* = 7.2 Hz, 1H), 2.76 -2.70 (m, 2H), 2.47 (d, *J* = 7.2 Hz, 2H), 2.22 (s, 3H), 2.15 - 2.07 (m, 1H), 2.02 - 1.95 (m, 1H), 1.89 – 1.83 (m, 1H), 1.59 (d, *J* = 7.1 Hz, 3H), 1.22 (t, *J* = 7.1 Hz, 3H), 0.91 (d, *J* = 6.5 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.1, 173.3, 149.2, 144.5, 140.8, 138.4, 137.26, 129.8, 129.5, 129.3, 127.7, 127.2, 121.3, 113.8, 61.0, 56.3, 45.2, 45.0, 34.6, 31.1, 30.2, 22.4, 20.4, 18.5, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{32}H_{40}NO_4$ 502.2952; found 502.2954.

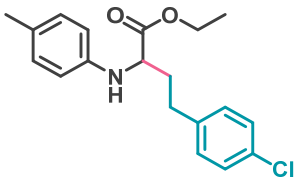


Ethyl 4-((8R,9S,13S,14S)-14-methyl-15-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-2-yl)-2-(p-tolylamino)butanoate (C40). The desired pure product was obtained in 18% yield (17.1 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.21 (d, *J* = 8.0 Hz, 1H), 7.02 - 6.94 (m, 3H), 6.92 (s, 1H), 6.53 (d, *J* = 8.3 Hz, 2H), 4.20 - 4.13 (m, 2H), 4.04 (t, *J* = 7.6, 5.4 Hz, 2H), 2.91 - 2.85 (m, 2H), 2.50 (dd, *J* = 18.6, 8.4 Hz, 1H), 2.44 - 2.37

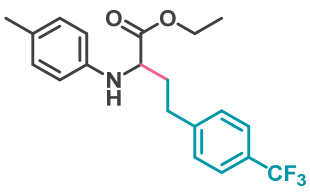
(m, 1H), 2.32 - 2.26 (m, 1H), 2.23 (s, 3H), 2.19 - 2.11 (m, 2H), 2.09 - 1.92 (m, 4H), 1.68 - 1.57 (m, 2H), 1.55 - 1.38 (m, 4H), 1.24 (t, J = 7.1 Hz, 3H), 0.91 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 220.9, 174.2, 144.6, 138.5, 137.5, 136.5, 129.8, 129.2, 129.1, 127.6, 125.93, 125.92, 125.5, 113.8, 61.0, 56.62, 56.61, 50.5, 48.0, 44.3, 38.2, 35.9, 34.8, 31.6, 31.3, 29.4, 26.5, 25.7, 21.6, 20.4, 14.3, 13.8. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{31}H_{40}NO_3$ 474.3003; found 474.3003.



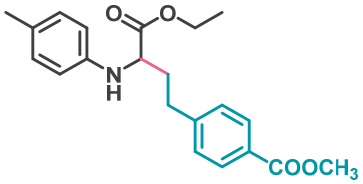
Ethyl 4-(4-fluorophenyl)-2-(p-tolylamino)butanoate (C41). The desired pure product was obtained in 32% yield (20.1 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.13 (dd, J = 8.5, 5.5 Hz, 2H), 7.01 - 6.91 (m, 4H), 6.51 (d, J = 8.3 Hz, 2H), 4.16 (q, J = 7.2 Hz, 2H), 4.04 - 3.95 (m, 2H), 2.74 (t, J = 8.4 Hz, 2H), 2.23 (s, 3H), 2.15 - 2.08 (m, 1H), 2.04 - 1.97 (m, 1H), 1.24 (t, J = 7.1 Hz, 3H).^{13}C NMR (151 MHz, $CDCl_3$) δ 174.1, 161.4 (d, J = 243.9 Hz), 144.5, 136.5 (d, J = 3.3 Hz), 129.9 (d, J = 7.6 Hz), 129.8, 127.7, 115.2 (d, J = 21.1 Hz). 113.8, 61.1, 56.3, 34.8, 31.0, 20.4, 14.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ -117.25. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{19}H_{23}FNO_2$ 316.1707; found 316.1706.



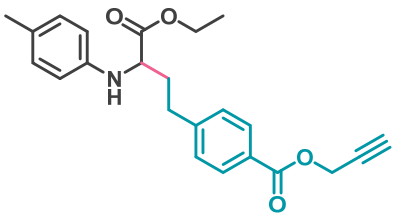
Ethyl 4-(4-chlorophenyl)-2-(p-tolylamino)butanoate (C42). The desired pure product was obtained in 36% yield (23.8 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, J = 8.4 Hz, 2H), 7.11 (d, J = 8.3 Hz, 2H), 6.97 (d, J = 8.4 Hz, 2H), 6.51 (d, J = 8.4 Hz, 2H), 4.15 (q, J = 7.1 Hz, 2H), 4.03 - 3.95 (m, 2H), 2.74 (t, J = 8.1 Hz, 2H), 2.23 (s, 3H), 2.17 - 2.08 (m, 1H), 2.06 - 1.95 (m, 1H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.0, 144.5, 139.4, 131.9, 129.9, 129.8, 128.6, 127.8, 113.8, 61.1, 56.3, 34.5, 31.1, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{19}H_{23}ClNO_2$ 332.1412; found 332.1412.



Ethyl 2-(p-tolylamino)-4-(4-(trifluoromethyl)phenyl)butanoate (C43). The desired pure product was obtained in 27% yield (19.7mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 8.2 Hz, 2H), 6.52 (d, *J* = 8.0 Hz, 2H), 4.16 (q, *J* = 7.3 Hz, 2H), 4.02 (dd, *J* = 7.4, 5.6 Hz, 1H), 2.83 (t, *J* = 8.0 Hz, 2H), 2.23 (s, 3H), 2.22 - 2.12 (m, 1H), 2.08 - 1.99 (m, 1H), 1.24 (t, *J* = 7.1 Hz, 3H).^{13}C NMR (151 MHz, $CDCl_3$) δ 173.9, 145.1, 144.4, 129.8, 128.8, 128.5 (q, *J* = 31.9 Hz), 127.9, 125.4 (q, *J* = 3.9 Hz), 124.3 (q, *J* = 271.7 Hz), 113.8, 61.2, 56.3, 34.3, 31.6, 20.4, 14.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.39. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{20}H_{23}F_3NO_2$ 366.1675; found 366.1676.

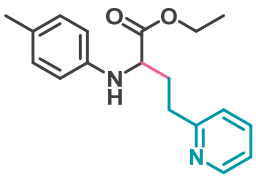


Methyl 4-(4-ethoxy-4-oxo-3-(p-tolylamino)butyl)benzoate (C44). The desired pure product was obtained in 37% yield (26.3 mg) as a white solid. m.p. = 83 - 85 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.2 Hz, 2H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.52 (d, *J* = 8.5 Hz, 2H), 4.16 (q, *J* = 7.2 Hz, 2H), 4.01 (dd, *J* = 7.4, 5.6 Hz, 1H), 3.90 (s, 3H), 2.85 - 2.78 (m, 2H), 2.23 (s, 3H), 2.19 - 2.13 (m, 1H), 2.08 - 2.01 (m, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.9, 167.0, 146.5, 144.4, 129.8, 129.8, 128.6, 128.2, 127.8, 113.9, 61.1, 56.4, 52.0, 34.2, 31.8, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{26}NO_4$ 356.1856; found 356.1857.

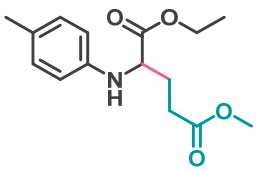


Prop-2-yn-1-yl 4-(4-ethoxy-4-oxo-3-(p-tolylamino)butyl)benzoate (C45). The desired pure product was obtained in 30% yield (22.7 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ

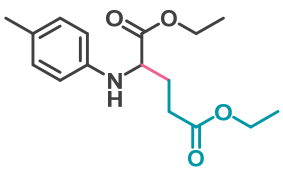
7.99 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 8.4 Hz, 2H), 6.98 (d, J = 8.2 Hz, 2H), 6.52 (d, J = 8.4 Hz, 2H), 4.92 (d, J = 2.4 Hz, 2H), 4.16 (q, J = 7.1 Hz, 2H), 4.07 - 3.98 (m, 2H), 2.87 - 2.79 (m, 2H), 2.51 (t, J = 2.5 Hz, 1H), 2.23 (s, 3H), 2.20 - 2.12 (m, 1H), 2.09 - 1.99 (m, 1H), 1.24 (t, J = 7.2 Hz, 3H).^{13}C NMR (151 MHz, $CDCl_3$) δ 173.9, 165.7, 147.0, 144.4, 130.1, 129.8, 128.6, 127.8, 127.4, 113.8, 77.8, 74.9, 61.1, 56.3, 52.3, 34.2, 31.8, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{23}H_{26}NO_4$ 380.1856; found 380.1858.



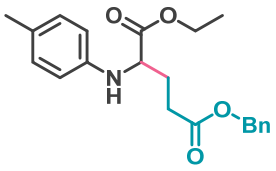
Ethyl 4-(pyridin-2-yl)-2-(p-tolylamino)butanoate (C46). The desired pure product was obtained in 40% yield (25.0 mg) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (d, J = 4.3 Hz, 1H), 7.60 - 7.54 (m, 1H), 7.21 - 7.06 (m, 2H), 6.96 (d, J = 8.0 Hz, 2H), 6.53 (d, J = 8.4 Hz, 2H), 4.20 - 4.11 (m, 3H), 4.05 (s, 1H), 2.99 - 2.92 (m, 2H), 2.36 - 2.26 (m, 1H), 2.22 (s, 3H), 2.20 -2.13 (m, 1H), 1.23 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δk, 32.6, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{18}H_{23}N_2O_2$ 299.1754; found 299.1753.



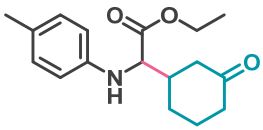
1-ethyl 5-methyl p-tolylglutamate (C47). The desired pure product was obtained in 54% yield (30.1 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (d, J = 8.0 Hz, 2H), 6.55 (d, J = 8.4 Hz, 2H), 4.17 (q, J = 7.1 Hz, 2H), 4.10 - 4.01 (m, 2H), 3.67 (s, 3H), 2.56 - 2.41 (m, 2H), 2.22 (s, 3H), 2.21- 2.13 (m, 1H), 2.11 - 2.01 (m, 1H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.6, 173.4, 144.4, 129.8, 127.8, 113.8, 61.2, 56.4, 51.7, 30.2, 27.9, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: [M + H]$^+$ calcd for $C_{15}H_{22}NO_4$ 280.1543; found 280.1544.



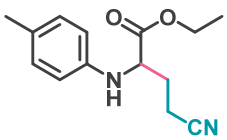
Diethyl p-tolylglutamate (C48). The desired pure product was obtained in 66% yield (38.6 mg) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (d, *J* = 8.6 Hz, 2H), 6.55 (d, *J* = 8.4 Hz, 2H), 4.20 - 4.05 (m, 5H), 2.53 - 2.39 (m, 2H), 2.23 (s, 3H), 2.20 - 2.12 (m, 1H), 2.11 - 2.00 (m, 1H), 1.28 - 1.20 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.7, 172.9, 144.4, 129.8, 127.8, 113.8, 61.2, 60.6, 56.5, 30.5, 28.0, 20.4, 14.2, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{24}NO_4$ 294.1700; found 294.1700.



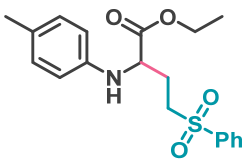
5-benzyl 1-ethyl p-tolylglutamate (C49). The desired pure product was obtained in 42% yield (29.8 mg) as a yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 5H), 6.96 (d, *J* = 8.5 Hz, 2H), 6.52 (d, *J* = 8.3 Hz, 2H), 5.14 - 5.08 (m, 2H), 4.20 - 4.12 (m, 2H), 4.10 - 4.06 (m, 1H), 4.03 (s, 1H), 2.59 - 2.48 (m, 2H), 2.22 (s, 3H), 2.21 - 2.16 (m, 1H), 2.11 - 2.04 (m, 1H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.6, 172.7, 144.4, 135.8, 129.8, 128.5, 128.3, 127.8, 113.9, 66.4, 61.2, 56.4, 30.4, 28.0, 20.4, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{26}NO_4$ 356.1856; found 356.1858.



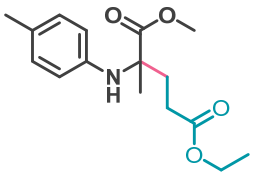
Ethyl 2-(3-oxocyclohexyl)-2-(p-tolylamino)acetate (C50). The desired pure product was obtained in 57% yield (32.9 mg, dr = 1.1:1) as a yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.99 (dd, *J* = 8.3, 2.5 Hz, 2H), 6.57 (dd, *J* = 8.5, 2.0 Hz, 2H), 4.22 - 4.15 (m, 2H), 4.12 - 4.04 (m, 1H), 4.01 - 3.90 (m, 1H), 2.49 - 2.25 (m, 5H), 2.23 (d, *J* = 2.1 Hz, 3H), 2.15 - 2.07 (m, 1H), 1.99 - 1.90 (m, 1H), 1.72 - 1.52 (m, 2H), 1.30 – 1.22 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 210.5, 210.3, 172.8, 172.6, 144.9, 144.7, 129.9, 129.8, 128.2, 128.0, 114.3, 114.1, 61.6, 61.4, 61.4, 61.3, 45.0, 43.7, 42.0, 41.8, 41.1, 41.1, 28.1, 26.6, 24.9, 24.7, 20.3, 14.2, 14.2. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{17}H_{24}NO_3$ 290.1751; found 290.1750.



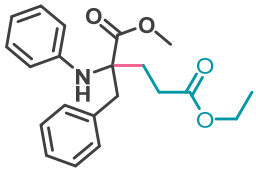
Ethyl 4-cyano-2-(p-tolylamino)butanoate (C51). The desired pure product was obtained in 61% yield (30.1 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.00 (d, J = 7.9 Hz, 2H), 6.59 (d, J = 8.4 Hz, 2H), 4.21 (q, J = 7.1 Hz, 2H), 4.16 - 4.10 (m, 1H), 4.05 (d, J = 8.9 Hz, 1H), 2.61 - 2.44 (m, 2H), 2.27 - 2.20 (m, 4H), 2.07 - 2.01 (m, 1H), 1.27 (t, J = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.6, 143.9, 129.9, 128.5, 118.9, 114.2, 61.7, 55.8, 28.6, 20.3, 14.1, 13.6. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{14}H_{19}N_2O_2$ 247.1441; found 247.1441.



Ethyl 4-(phenylsulfonyl)-2-(p-tolylamino)butanoate (C52). The desired pure product was obtained in 30% yield (21.6 mg) as a yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (d, J = 8.1 Hz, 2H), 7.67 -7.62 (m, 1H), 7.58 - 7.52 (m, 2H), 6.95 (d, J = 8.0 Hz, 2H), 6.50 (d, J = 8.4 Hz, 2H), 4.15 (q, J = 7.2 Hz, 2H), 4.11 - 4.06 (m, 1H), 3.96 (d, J = 8.9 Hz, 1H), 3.29 - 3.20 (m, 2H), 2.33 - 2.28 (m, 1H), 2.21 (s, 3H), 2.13 - 2.07 (m, 1H), 1.21 (t, J = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.8, 143.9, 138.9, 133.8, 129.9, 129.4, 128.4, 128.0, 114.1, 61.6, 55.7, 52.6, 26.0, 20.4, 14.1. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{19}H_{24}NO_4S$ 362.1421; found 362.1421.



5-ethyl 1-methyl 2-methyl-2-(p-tolylamino)pentanedioate (C53). The desired pure product was obtained in 40% yield (23.5 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.96 (d, J = 7.9 Hz, 2H), 6.53 (d, J = 8.3 Hz, 2H), 4.09 (q, J = 7.2 Hz, 2H), 3.71 (s, 3H), 2.44 - 2.38 (m, 1H), 2.36 - 2.28 (m, 2H), 2.27 - 2.23 (m, 1H), 2.22 (s, 3H), 1.50 (s, 3H), 1.22 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 176.0, 173.2, 142.6, 129.6, 128.2, 116.4, 60.5, 60.3, 52.5, 33.1, 29.1, 23.3, 20.4, 14.1. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{24}NO_4$ 294.1700; found 294.1696.



5-ethyl 1-methyl 2-benzyl-2-(phenylamino)pentanedioate (C54). The desired pure product was obtained in 10% yield (7.1 mg) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.22 - 7.17 (m, 5H), 6.95 - 6.92 (m, 2H), 6.78 - 6.72 (m, 1H), 6.70 - 6.67 (m, 2H), 4.56 (s, 1H), 4.05 (q, *J* = 7.1 Hz, 2H), 3.71 (s, 3H), 3.49 (d, *J* = 13.7 Hz, 1H), 3.17 (d, *J* = 13.8 Hz, 1H), 2.58 - 2.51 (m, 1H), 2.44 - 2.35 (m, 2H), 2.23 - 2.16 (m, 1H), 1.20 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 172.9, 144.8, 135.8, 130.0, 129.4, 128.1, 126.9, 117.8, 114.8, 65.0, 60.5, 52.6, 40.1, 31.1, 29.2, 14.1. HRMS (ESI-Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{26}NO_4$ 356.1856; found 356.1855.

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[1] a) Nithinchandra, B. Kalluraya, S. Aamir, A. R. Shabaraya, *Eur. J. Med. Chem.* **2012**, *54*, 597-604; b) H. Zhi, S. P.-M. Ung, Y. Liu, L. Zhao, C.-J. Li, *Adv. Synth. Catal.* **2016**, *358*, 2553-2557.

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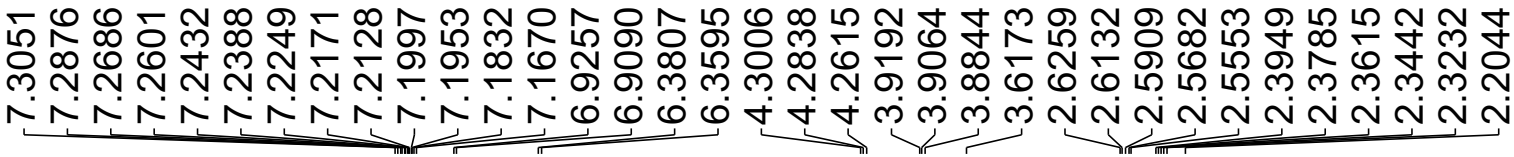

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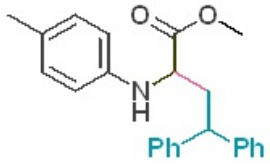

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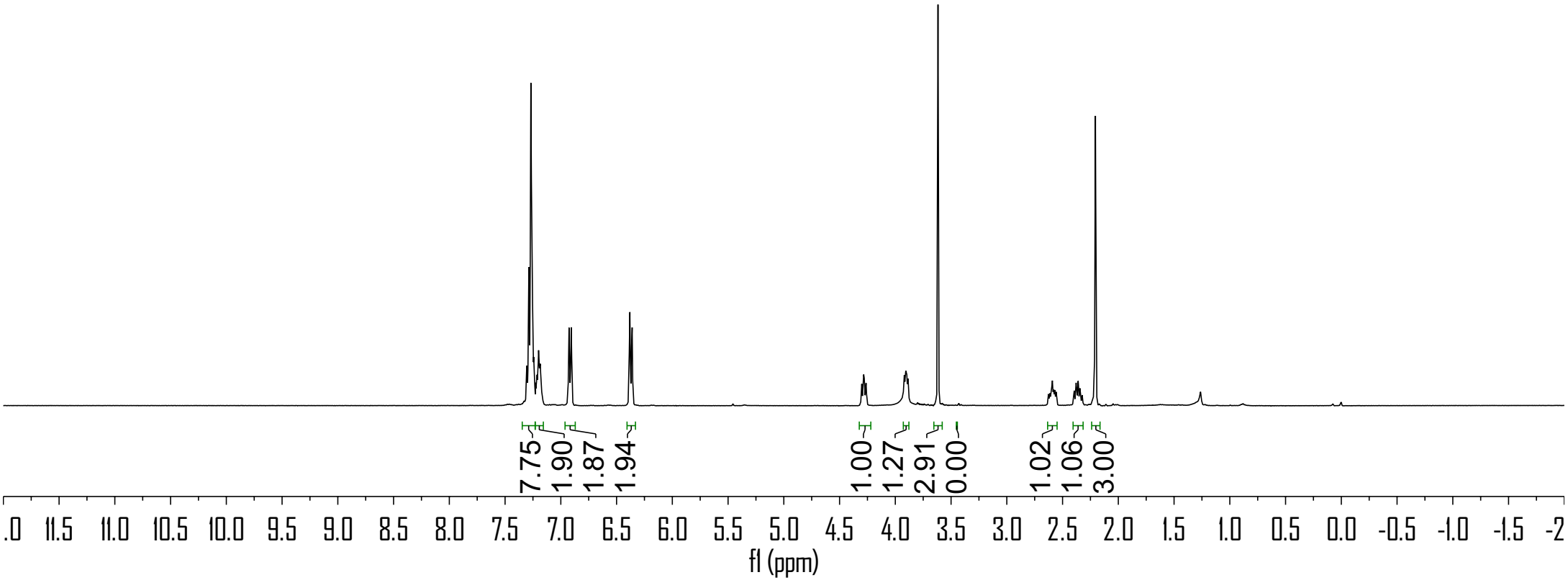

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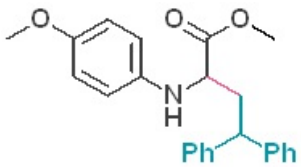
2.3205

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



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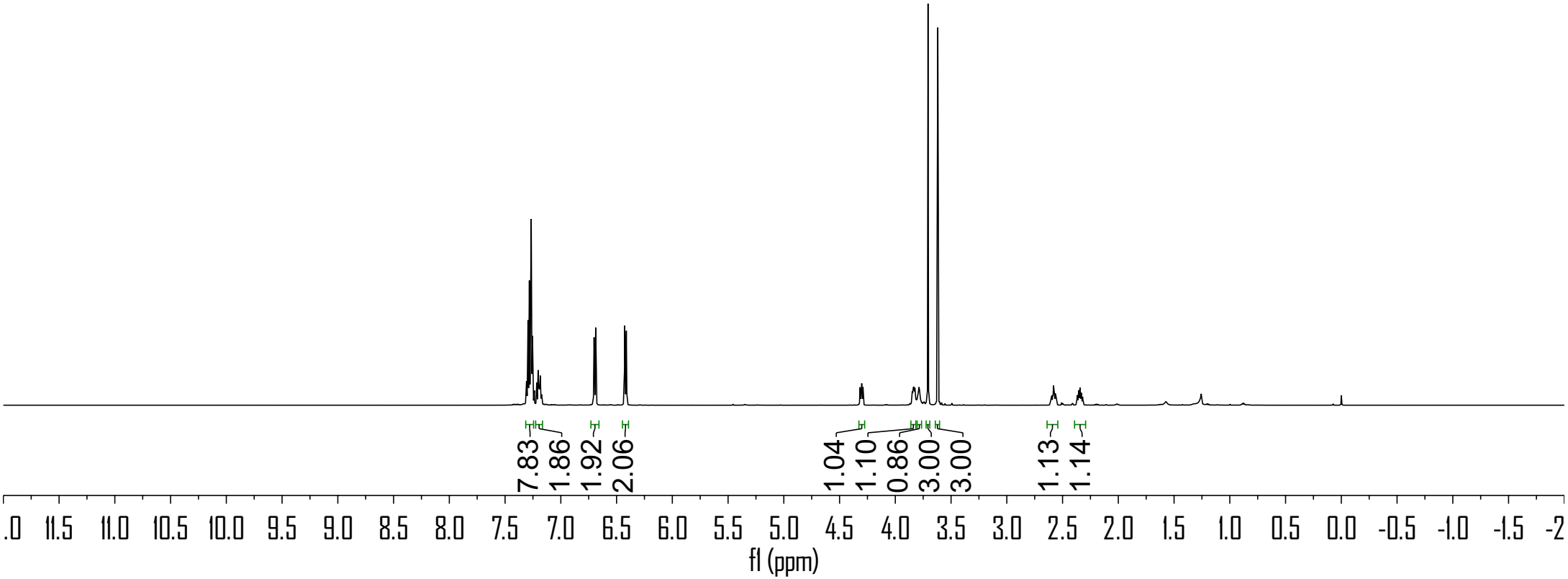

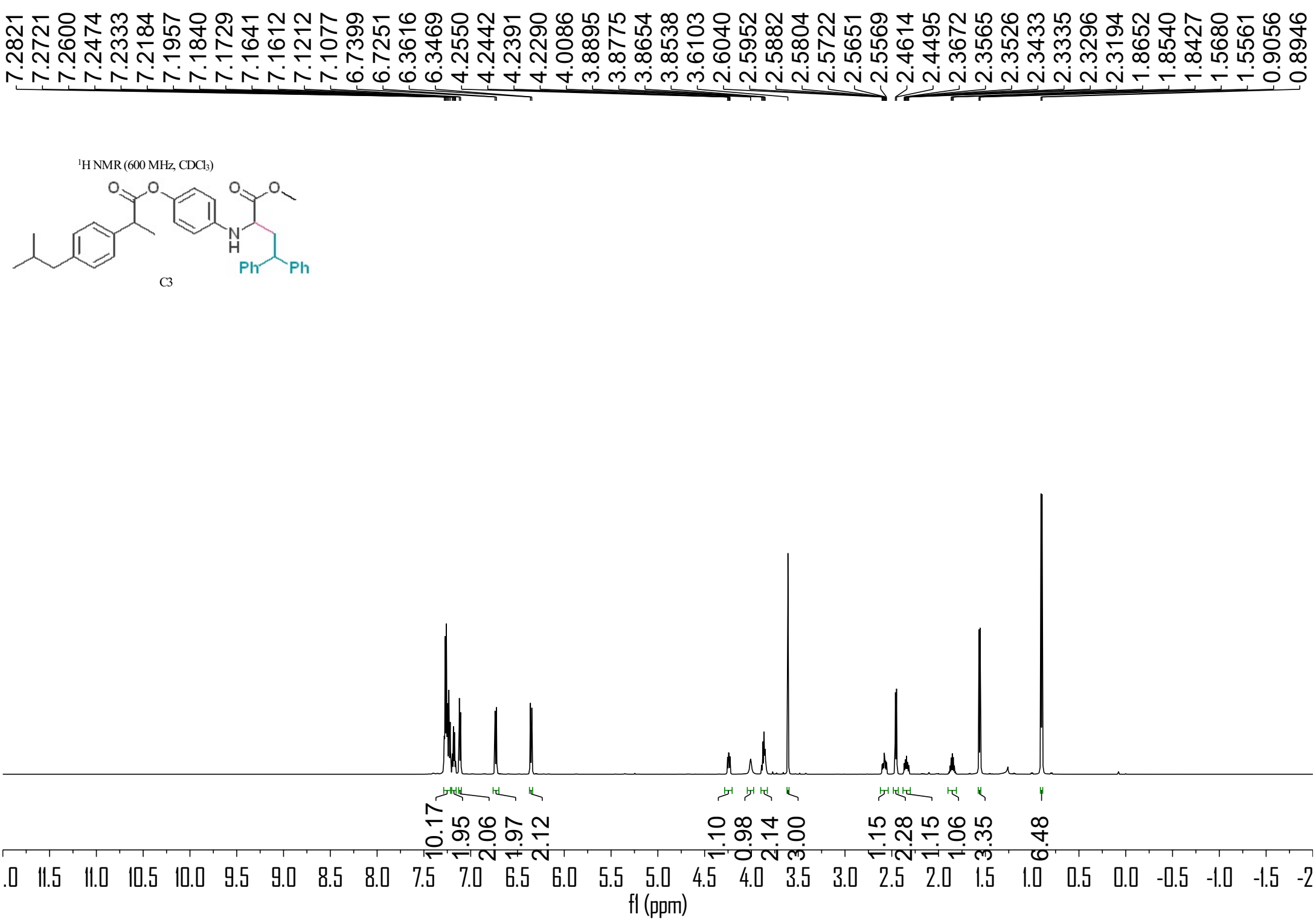
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Ph
Ph
C3
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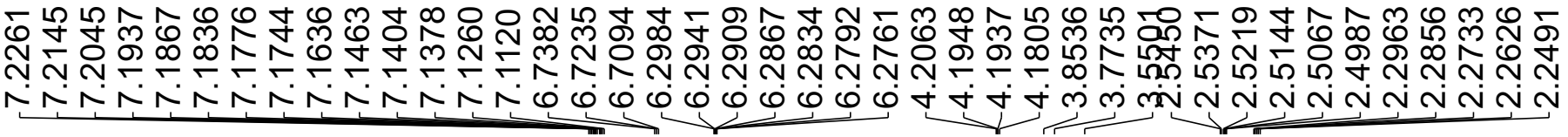

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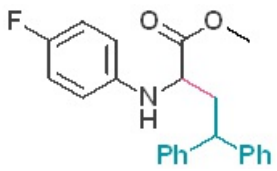

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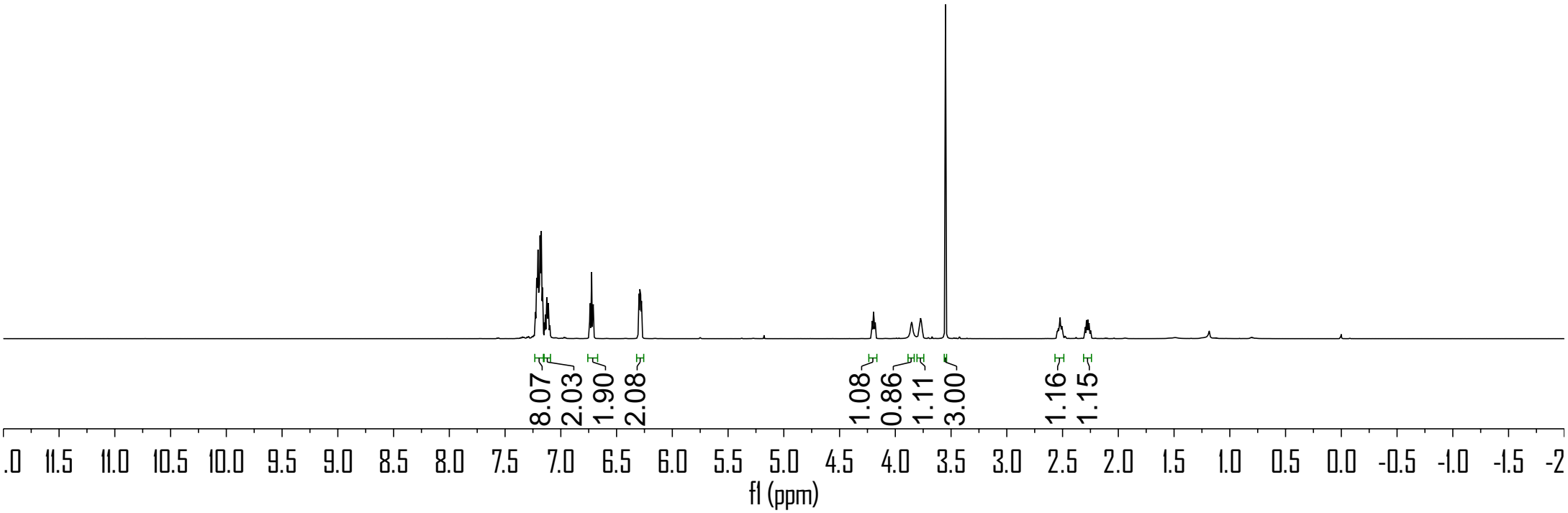

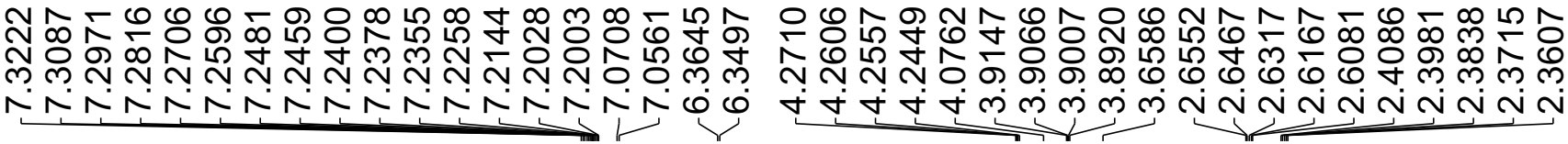

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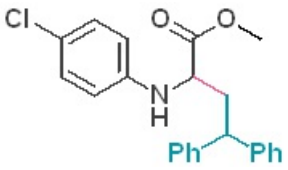
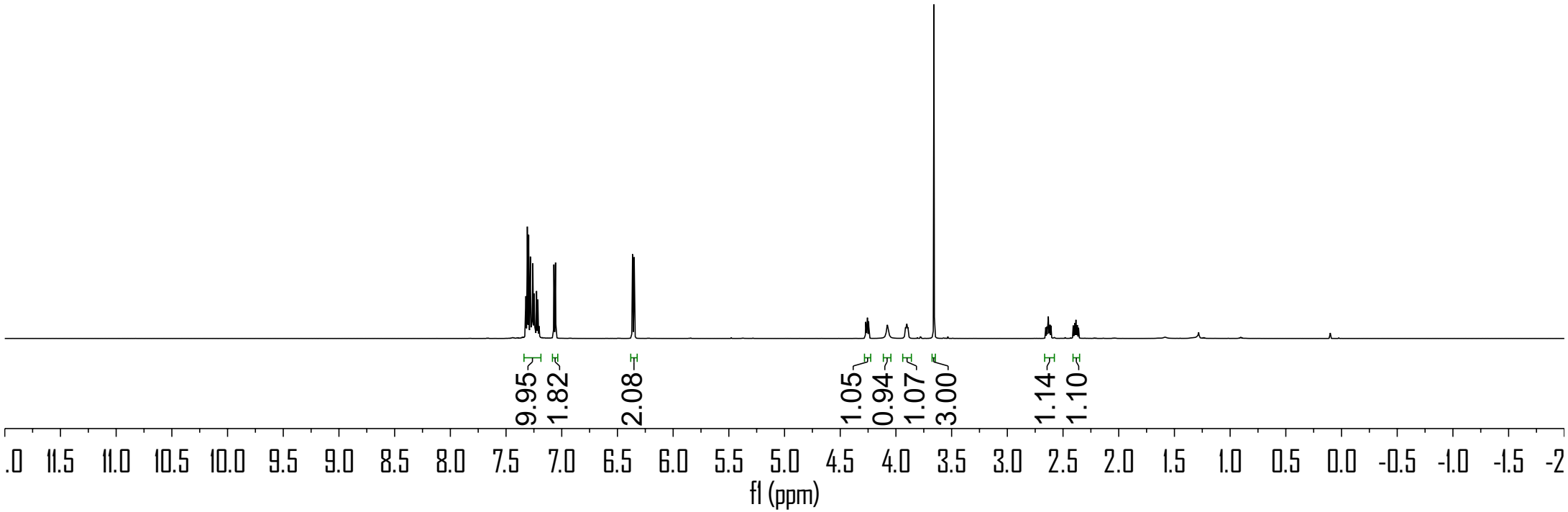


C5



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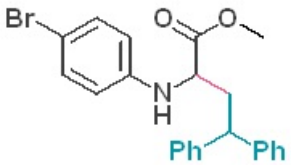
^{1}H NMR (600 MHz, $CDCl_3$)



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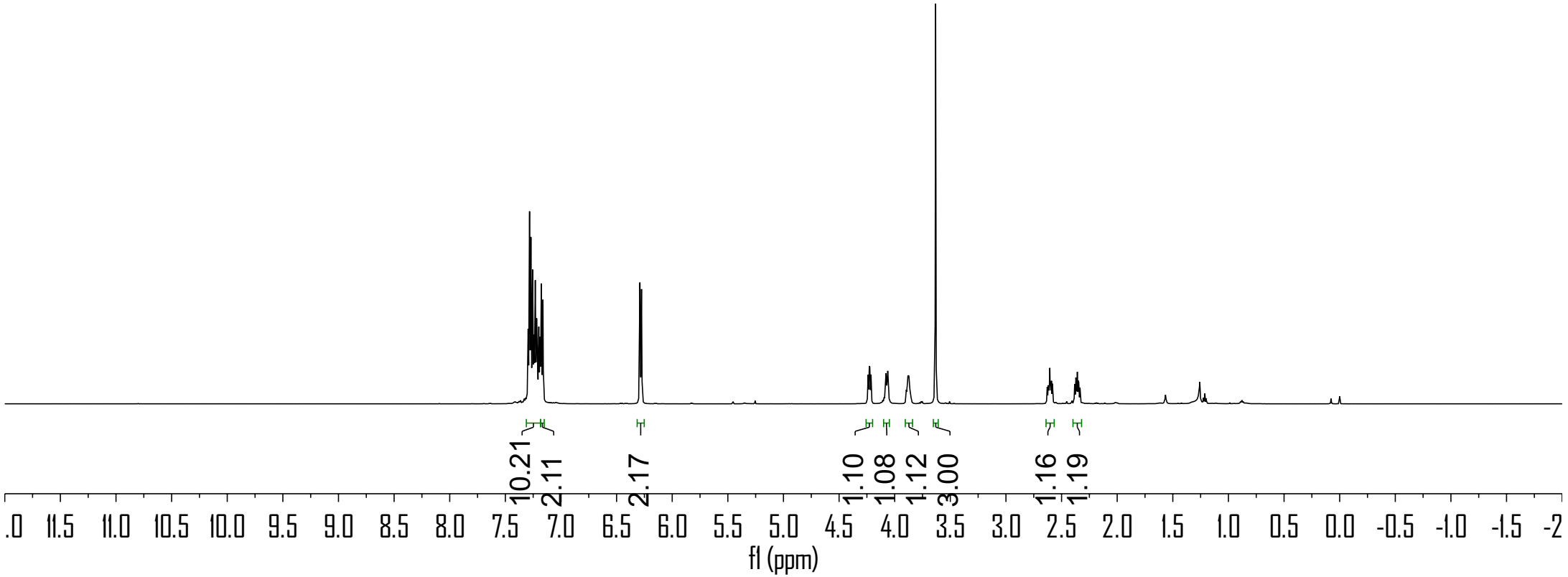

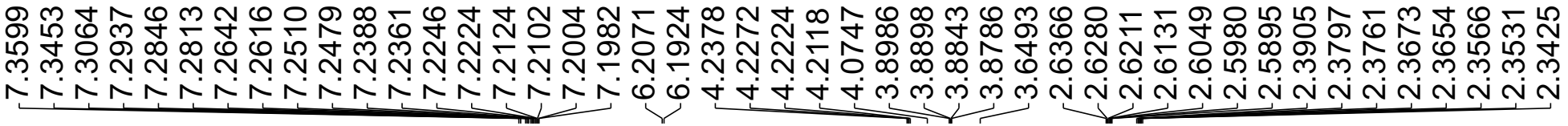

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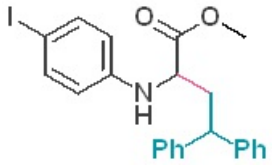

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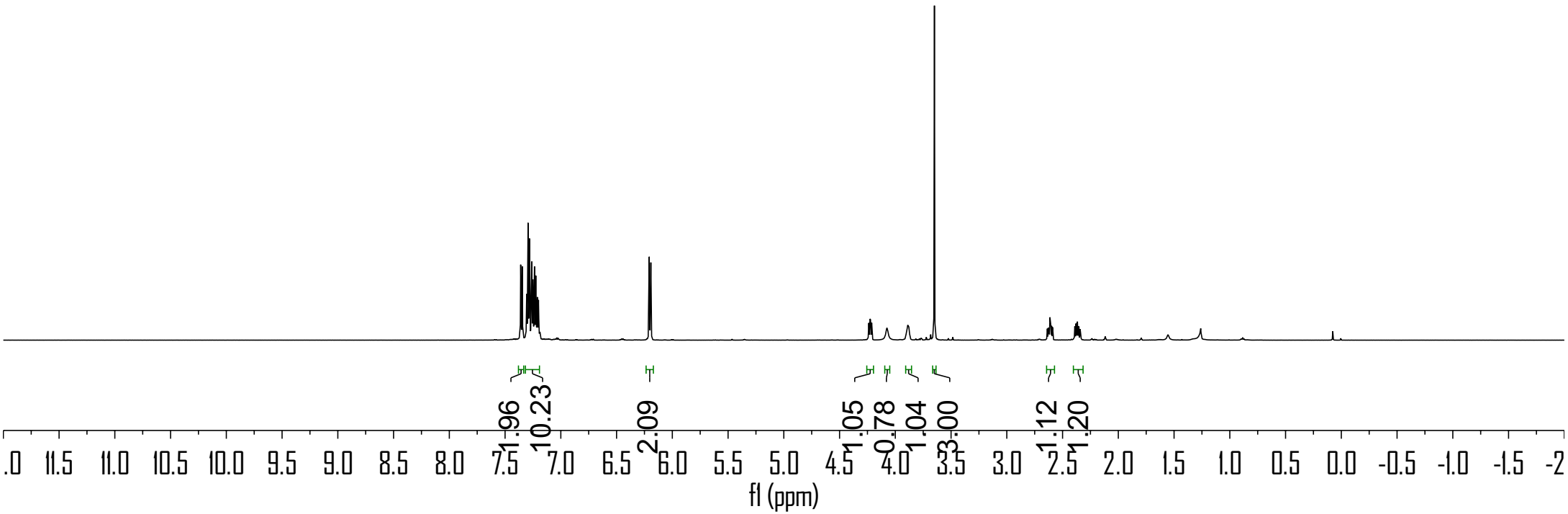

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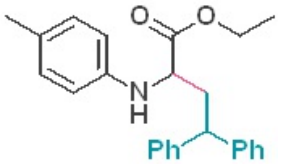
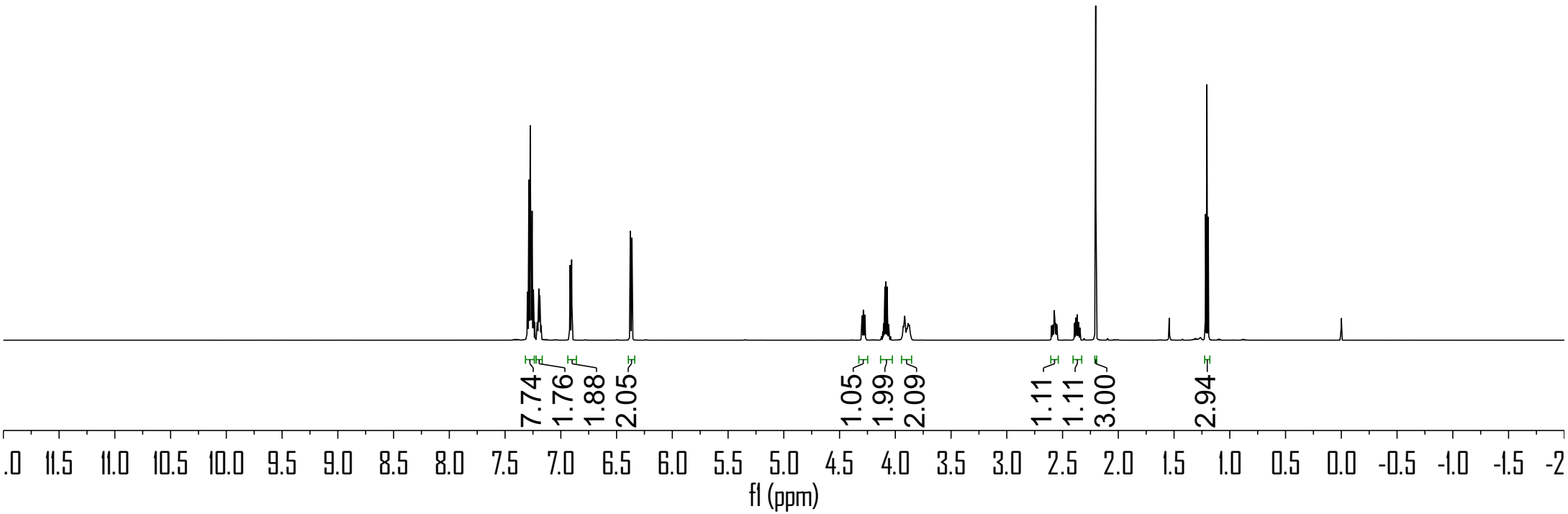
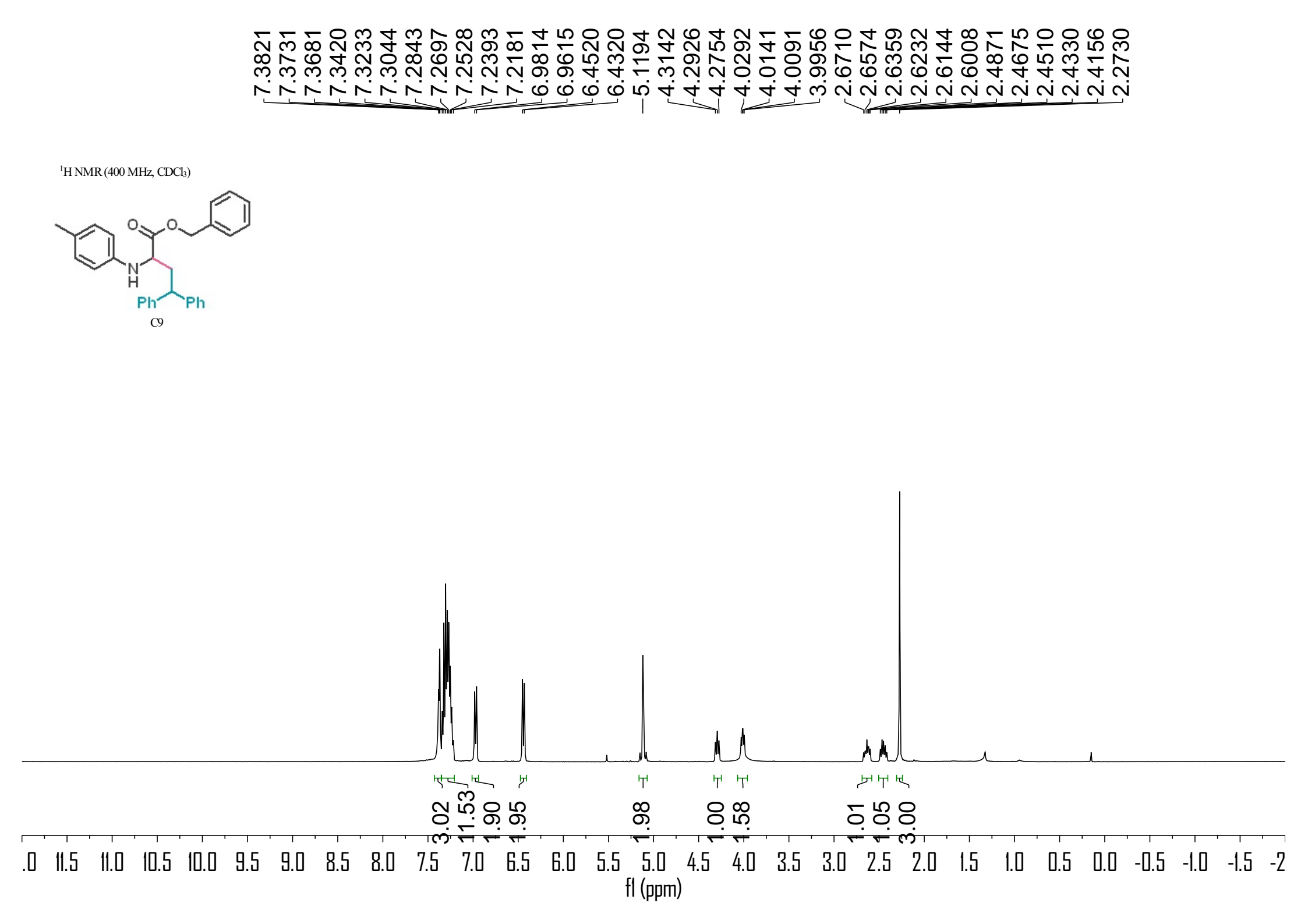
1.1937

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



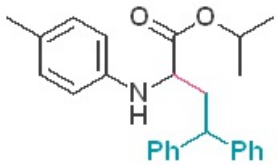
C8



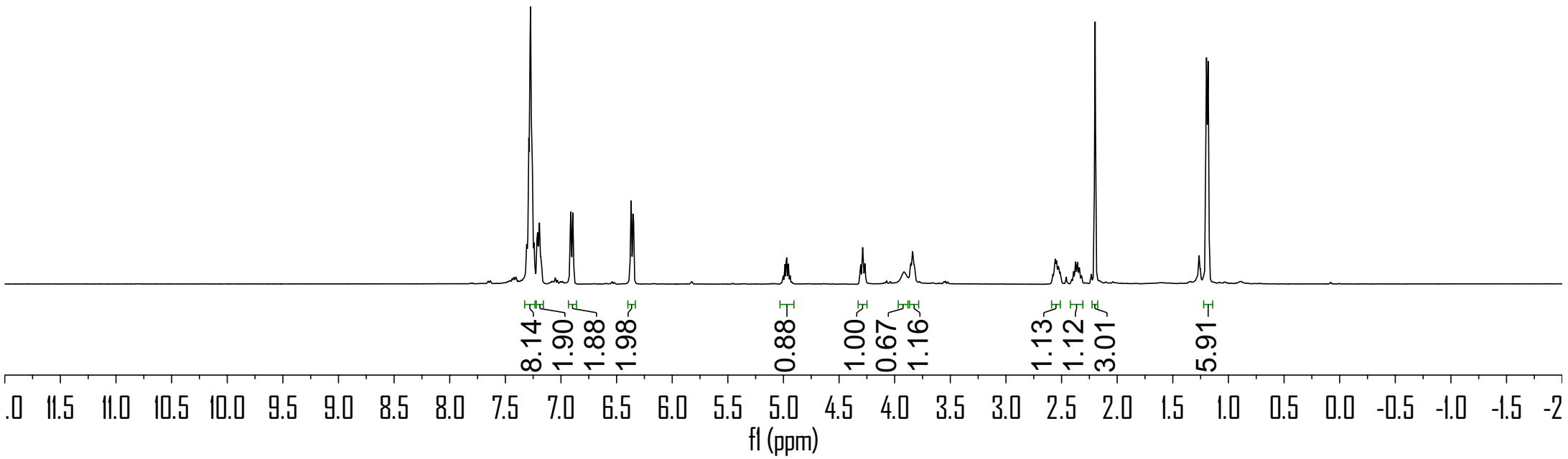

7.3821
7.3731
7.3681
7.3420
7.3233
7.3044
7.2843
7.2697
7.2528
7.2393
7.2181
6.9814
6.9615
6.4520
6.4320
5.1194
4.3142
4.2926
4.2754
4.0292
4.0141
4.0091
3.9956
2.6710
2.6574
2.6359
2.6232
2.6144
2.6008
2.4871
2.4675
2.4510
2.4330
2.4156
2.2730
1H NMR (400 MHz, CDCl3)
O
O
N
H
Ph
Ph
C9
3.02
11.53
1.90
1.95
1.98
1.00
1.58
1.01
1.05
3.00
f1 (ppm)

7.3095 7.2893 7.2841 7.2728 7.2619 7.2427 7.2161 7.2099 7.1933 7.1808 7.1767 6.9107 6.8948 6.3690 6.3534 5.0068 5.0018 4.9861 4.9755 4.9704 4.9547 4.9390 4.3049 4.2885 4.2665 3.9155 3.8549 3.8401 3.8196 2.5786 2.5565 2.5436 2.5291 2.5136 2.5082 2.4077 2.3915 2.3743 2.3565 2.3398 2.3193 2.1974 1.1975 1.1820

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

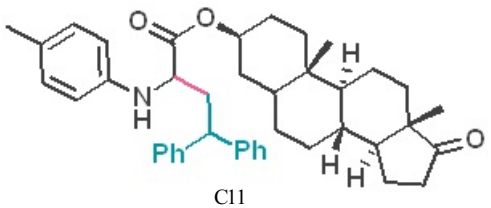


C10



7.3157
7.2966
7.2793
7.2739
7.2591
7.2545
7.2425
7.2208
7.2162
7.2032
7.1993
7.1826
6.9149
6.8949
6.3656
6.3448
4.2837
2.4176
2.3951
2.2030
2.0862
1.8081
1.8033
1.7768
1.7658
1.7461
1.7326
1.6178
1.5826
1.5588
1.5419
1.5218
1.4906
1.4862
1.4676
1.3524
1.3460
1.3233
1.3165
1.2954
1.2860
1.2659
1.2565
1.2352
1.2246
0.9969
0.9769
0.9619
0.8533
0.8423

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

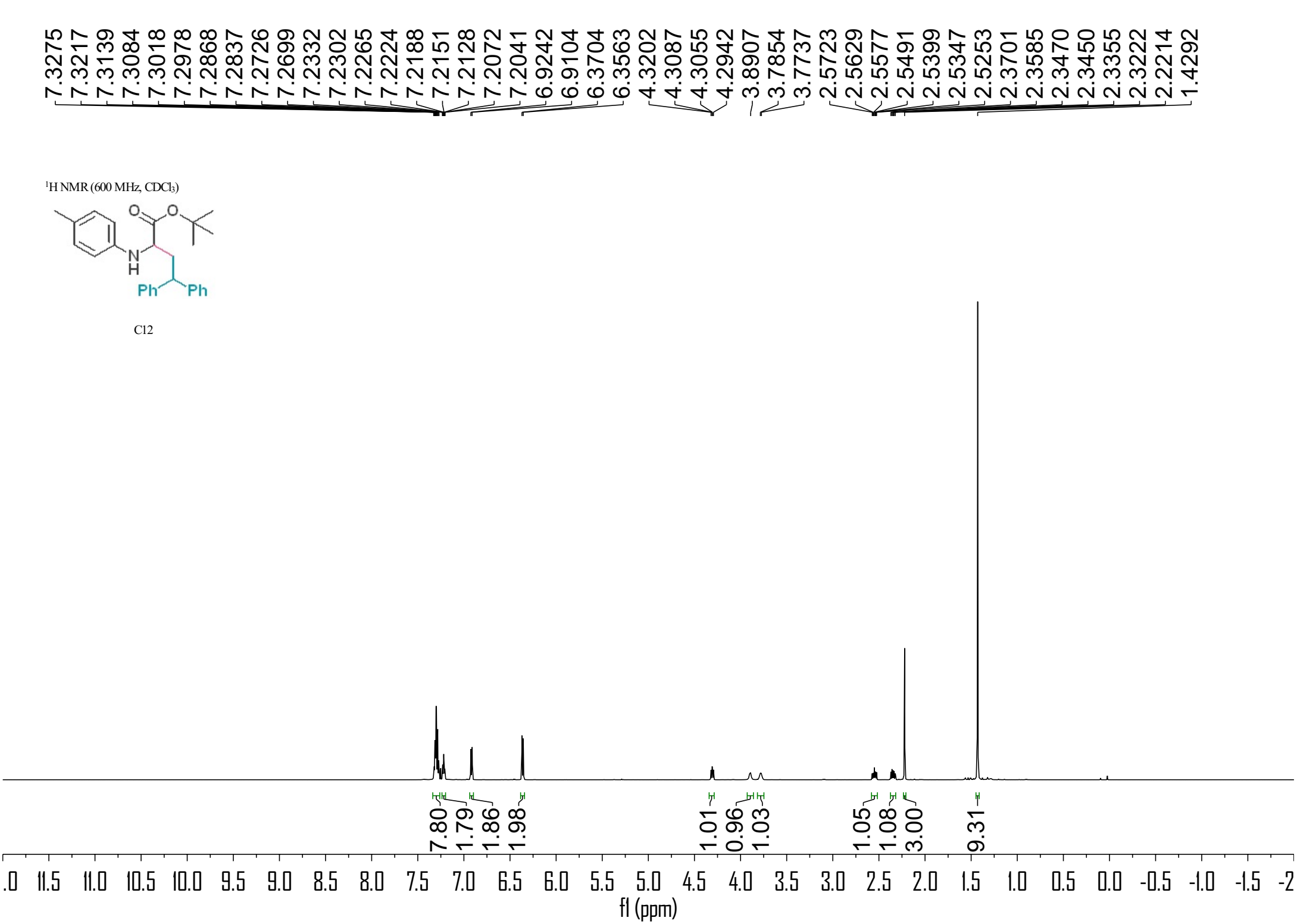


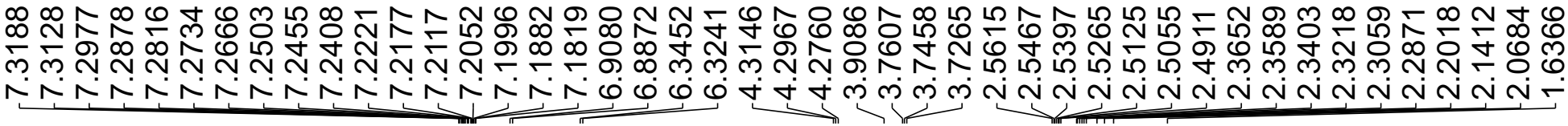
C11

8.38
2.23
1.93
1.99
1.00
1.00
1.99
0.95
2.09
3.03
1.21
1.12
4.00
6.31
6.40
2.36
6.22
1.18

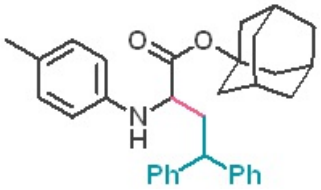
.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2

f1 (ppm)

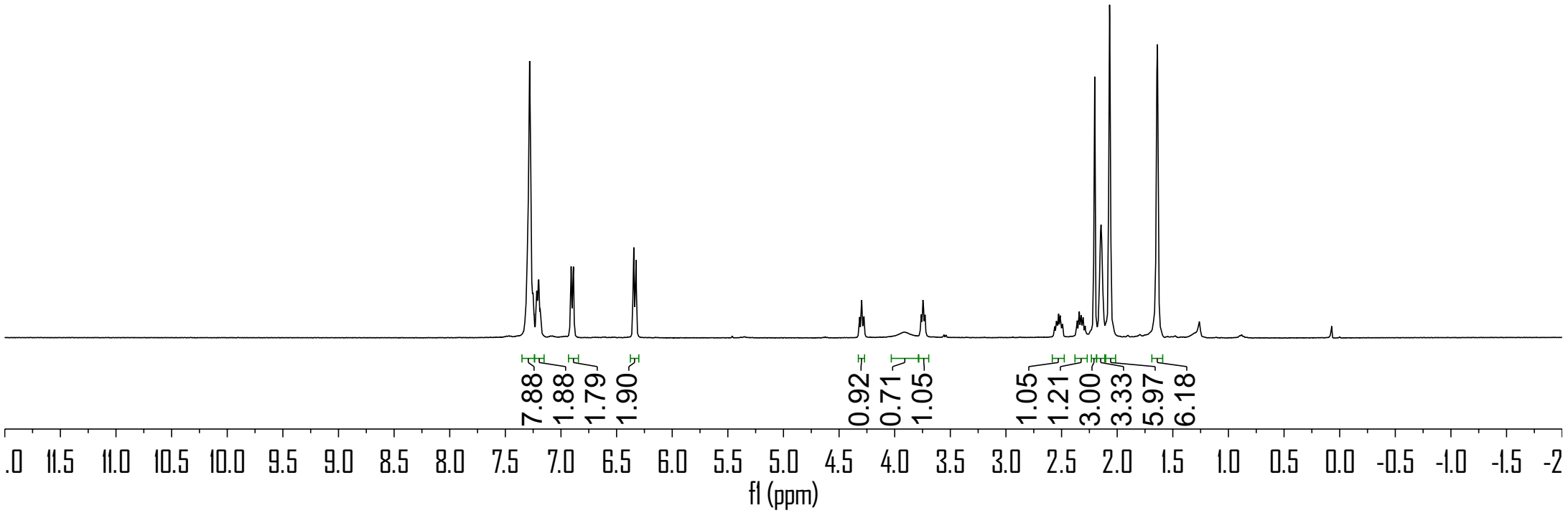

7.3275
7.3217
7.3139
7.3084
7.3018
7.2978
7.2868
7.2837
7.2726
7.2699
7.2332
7.2302
7.2265
7.2224
7.2188
7.2151
7.2128
7.2072
7.2041
6.9242
6.9104
6.3704
6.3563
4.3202
4.3087
4.3055
4.2942
3.8907
3.7854
3.7737
2.5723
2.5629
2.5577
2.5491
2.5399
2.5347
2.5253
2.3701
2.3585
2.3470
2.3450
2.3355
2.3222
2.2214
1.4292
1H NMR (600 MHz, CDCl3)
O
O
N
H
Ph
Ph
C12
7.80
1.79
1.86
1.98
1.01
0.96
1.03
1.05
1.08
3.00
9.31
.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)



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

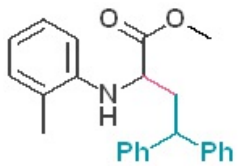


C13

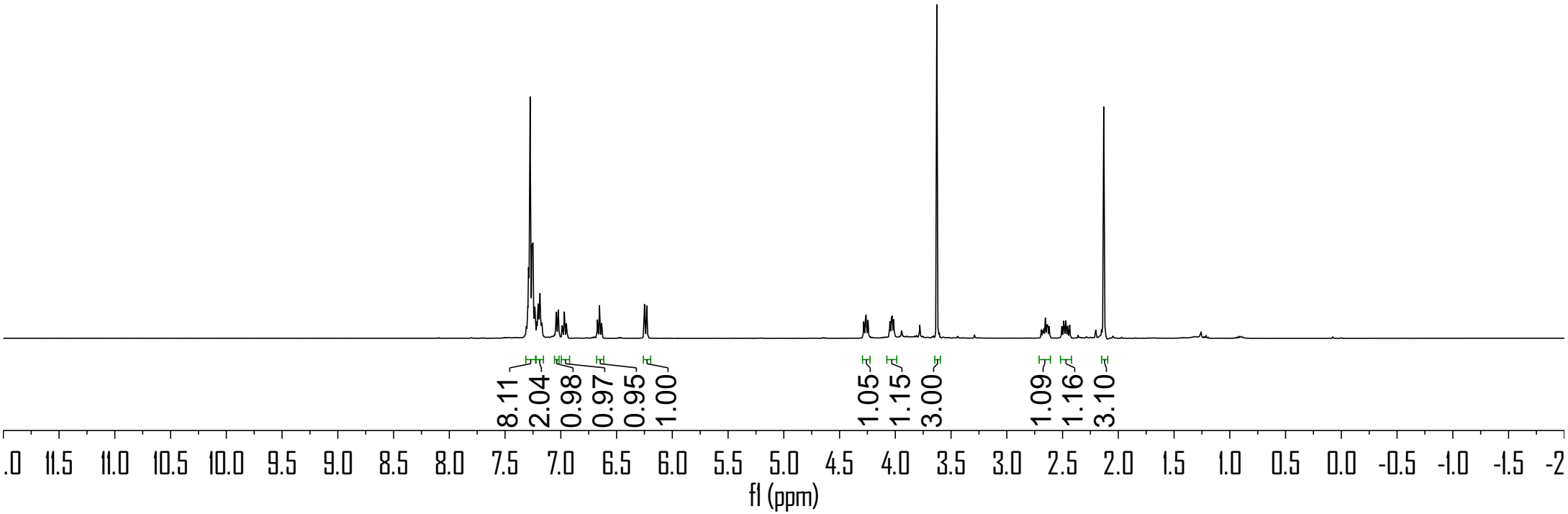


7.2986
7.2902
7.2754
7.2605
7.2491
7.2329
7.2143
7.2087
7.2034
7.1941
7.1869
7.1721
7.0424
7.0241
6.9882
6.9698
6.9509
6.6720
6.6536
6.6351
6.2492
6.2291
4.2837
4.2672
4.2616
4.2452
4.0484
4.0352
4.0282
4.0147
3.6278
2.6897
2.6764
2.6675
2.6548
2.6421
2.6331
2.6200
2.5070
2.4901
2.4698
2.4555
2.4358
2.1307

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

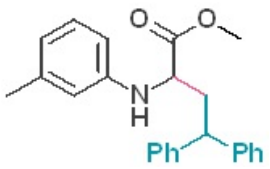


C14

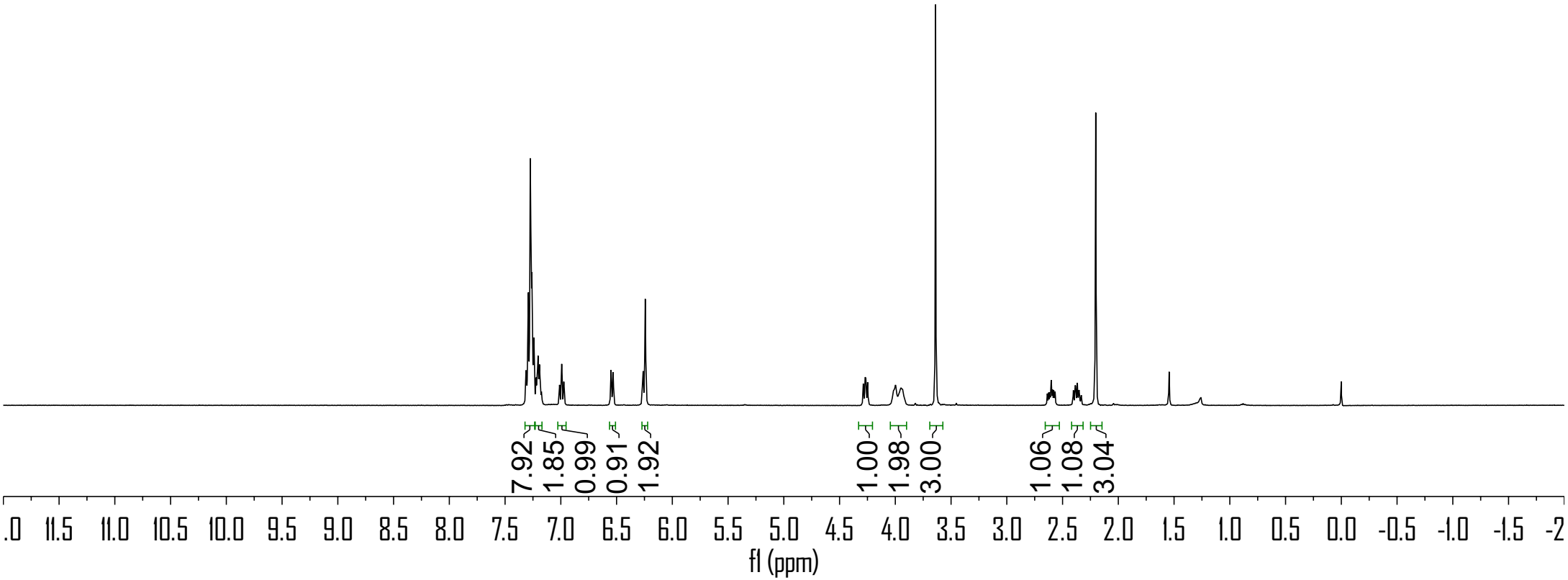


7.3122
7.2943
7.2902
7.2740
7.2681
7.2595
7.2404
7.2252
7.2212
7.2038
7.1906
7.1828
7.0117
6.9929
6.9740
6.5498
6.5312
6.2612
6.2427
4.2873
4.2712
4.2641
4.2484
3.9988
3.9501
3.9338
3.6389
2.6367
2.6242
2.6137
2.6017
2.5901
2.5791
2.5673
2.4031
2.3871
2.3672
2.3526
2.3325
2.2025

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

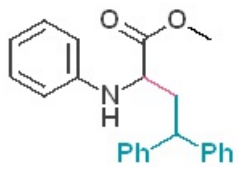


C15

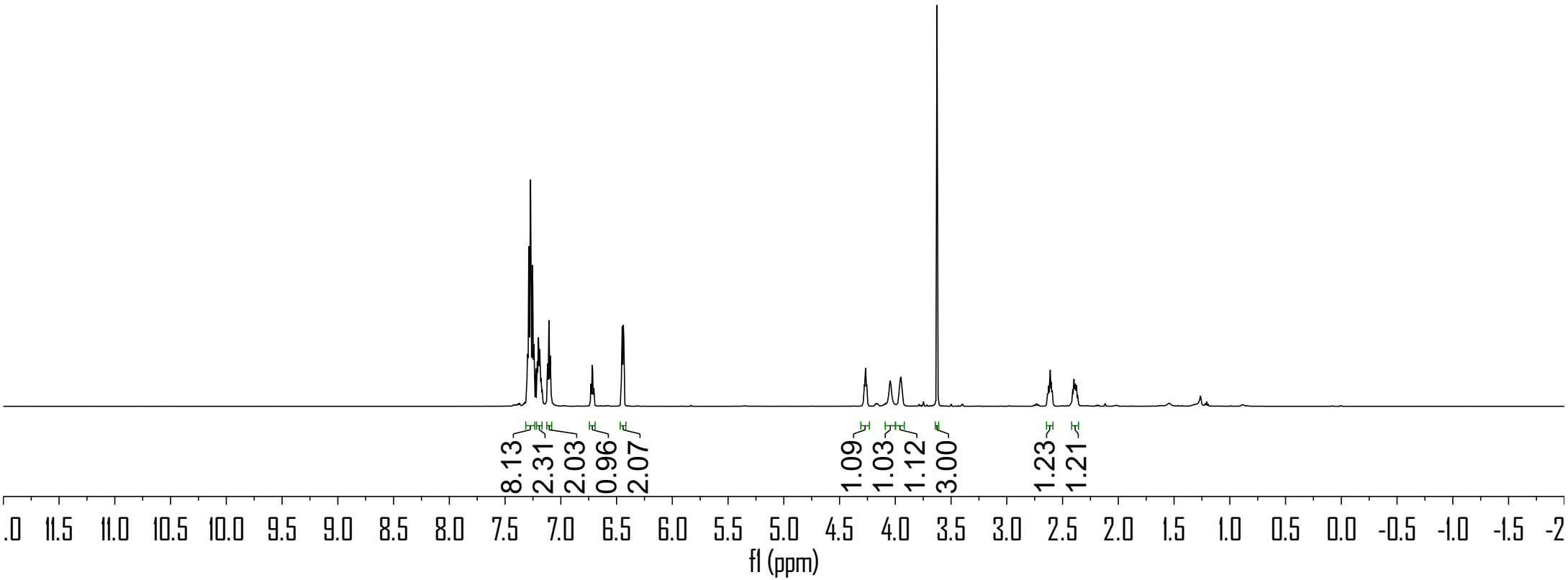


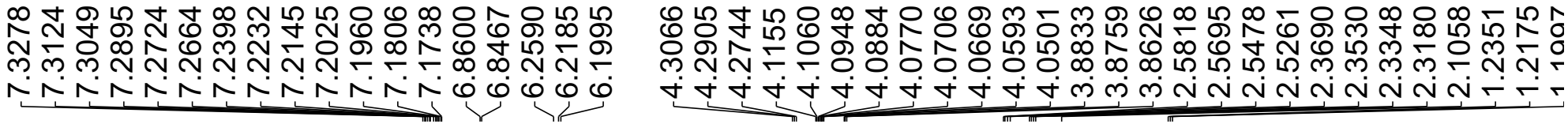
7.2993
7.2871
7.2713
7.2552
7.2520
7.2414
7.2133
7.2109
7.2011
7.1923
7.1899
7.1868
7.1781
7.1213
7.1189
7.1154
7.1067
7.0947
7.0922
7.0889
6.7303
6.7178
6.7059
6.4518
6.4392
4.2771
4.2661
4.2576
4.0454
3.9512
3.6261
2.6341
2.6253
2.6171
2.6104
2.6069
2.6023
2.5873
2.4142
2.4082
2.4004
2.3974
2.3893
2.3774
2.3744
2.3603

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

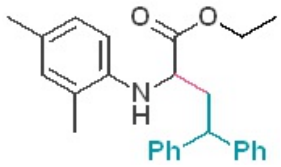


C16

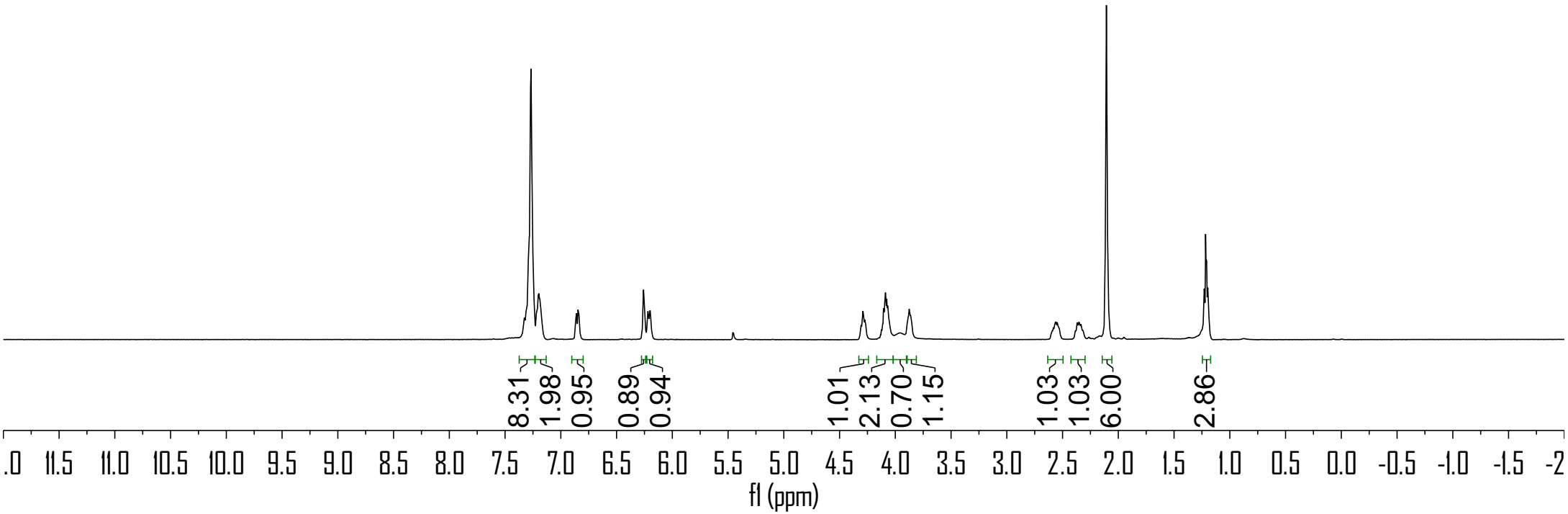




^{1}H NMR (400 MHz, $CDCl_3$)

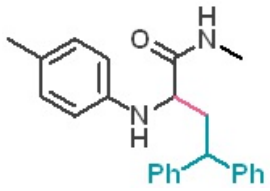


C17

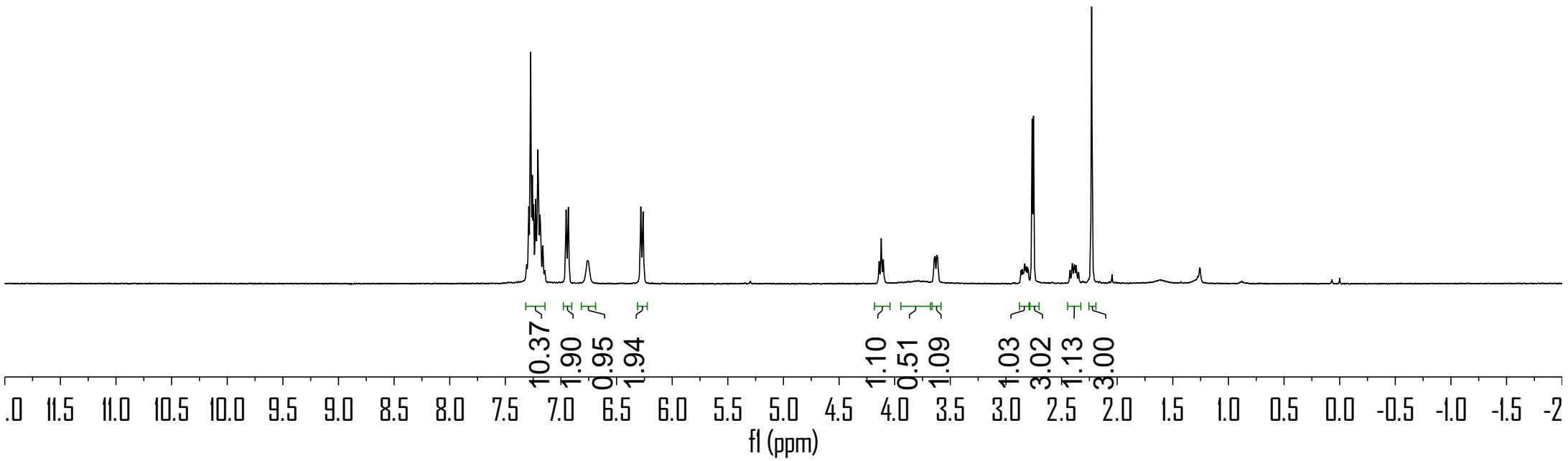


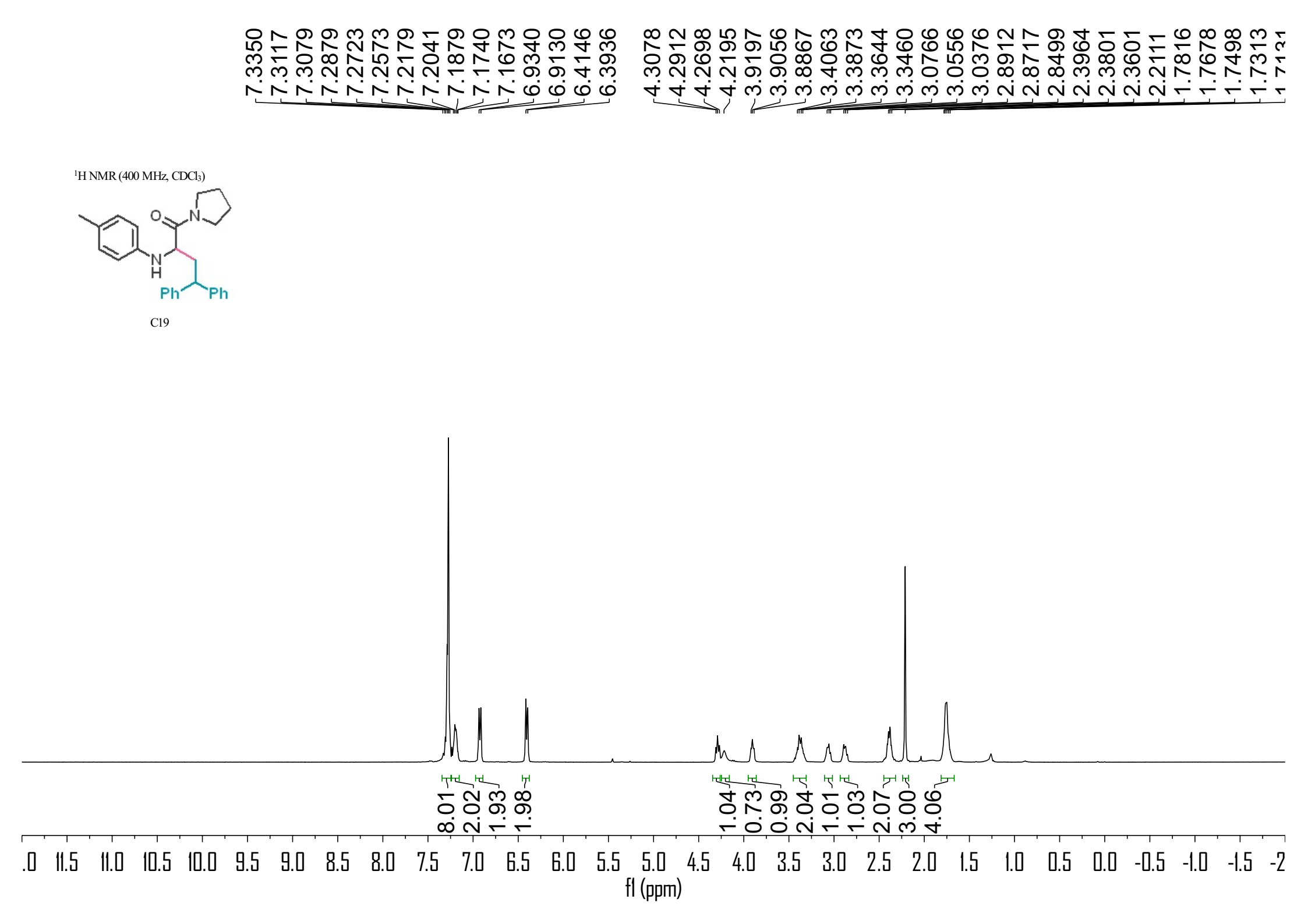
7.3096
7.2895
7.2728
7.2574
7.2469
7.2284
7.2075
7.1898
7.1840
7.1787
7.1633
7.1456
6.9536
6.9337
6.7595
6.2822
6.2622
4.1404
4.1210
4.1018
3.7846
3.6463
3.6365
3.6231
3.6129
2.8645
2.8546
2.8444
2.7644
2.7522
2.4241
2.4038
2.3889
2.3828
2.3701
2.3653
2.3472
2.2298

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



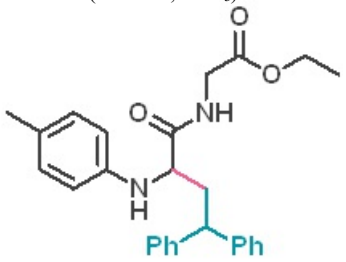
C18



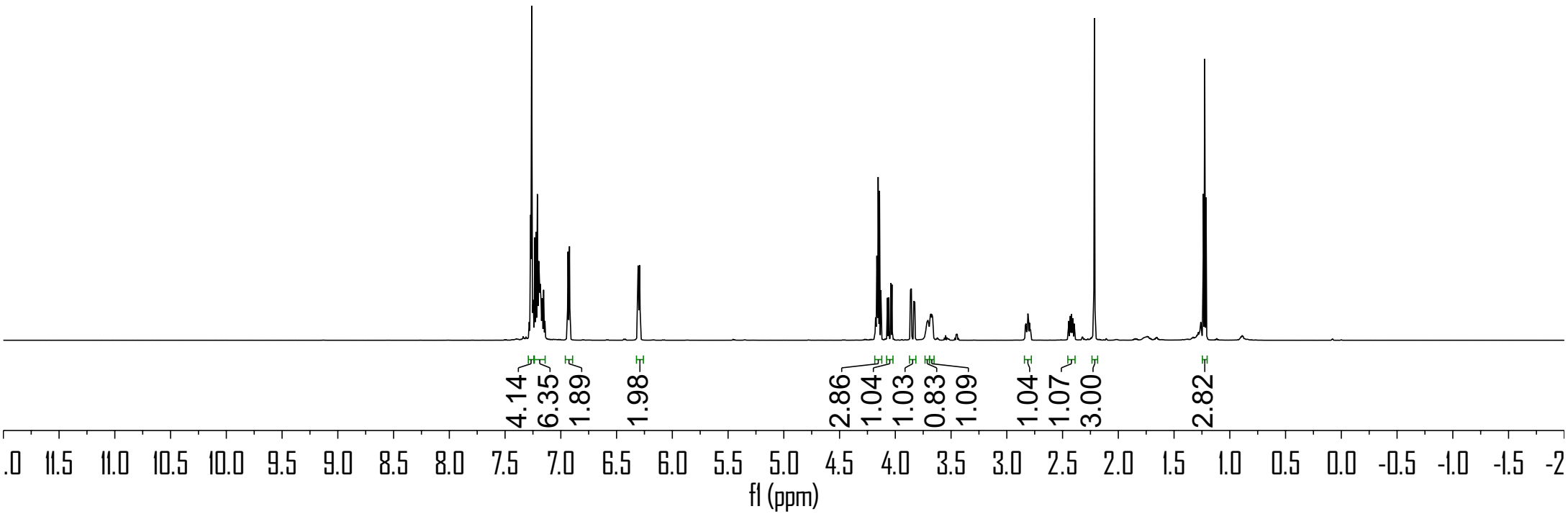

7.3350
7.3117
7.3079
7.2879
7.2723
7.2573
7.2179
7.2041
7.1879
7.1740
7.1673
6.9340
6.9130
6.4146
6.3936
4.3078
4.2912
4.2698
4.2195
3.9197
3.9056
3.8867
3.4063
3.3873
3.3644
3.3460
3.0766
3.0556
3.0376
2.8912
2.8717
2.8499
2.3964
2.3801
2.3601
2.2111
1.7816
1.7678
1.7498
1.7313
^{1}H NMR (400 MHz, CDCl$_3$)
O
N
N
H
Ph
Ph
C19
8.01
2.02
1.93
1.98
1.04
0.73
0.99
2.04
1.01
1.03
2.07
3.00
4.06
f1 (ppm)

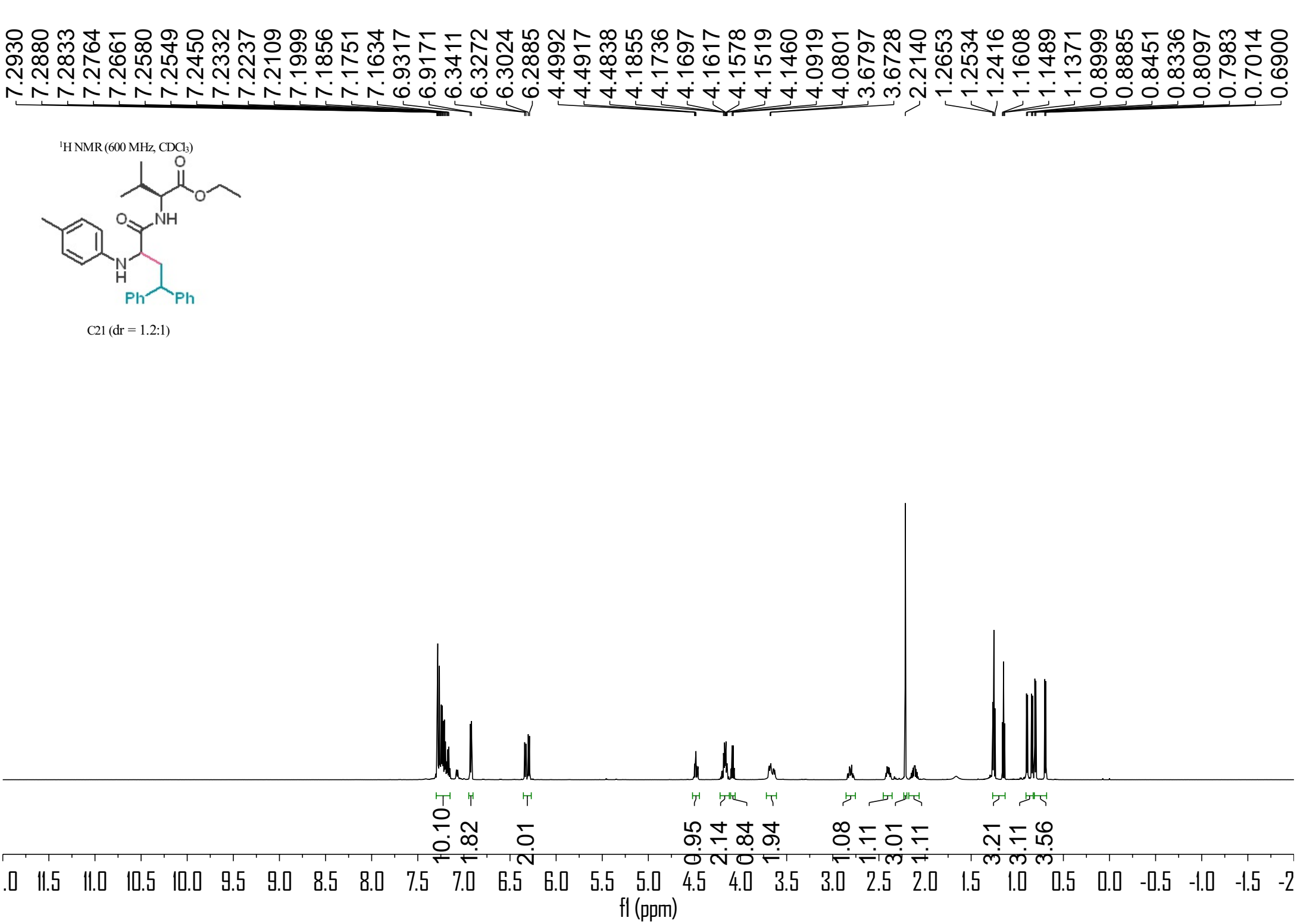
7.2840 7.2707 7.2613 7.2470 7.2339 7.2219 7.2101 7.1990 7.1905 7.1809 7.1690 7.1661 7.1544 7.1427 6.9369 6.9226 6.3068 6.2926 4.1747 4.1658 4.1538 4.1489 4.1420 4.1301 4.0703 4.0598 4.0400 4.0295 3.8641 3.8557 3.8339 3.8254 3.7066 3.6799 3.6708 2.8273 2.8198 2.8106 2.8039 2.7963 2.7895 2.4452 2.4305 2.4189 2.4069 2.3953 2.2151 1.2369 1.2250 1.2131

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



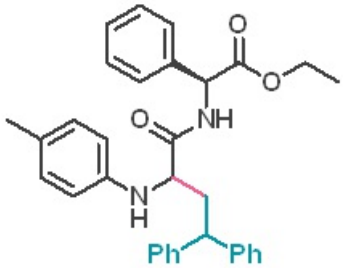
C20



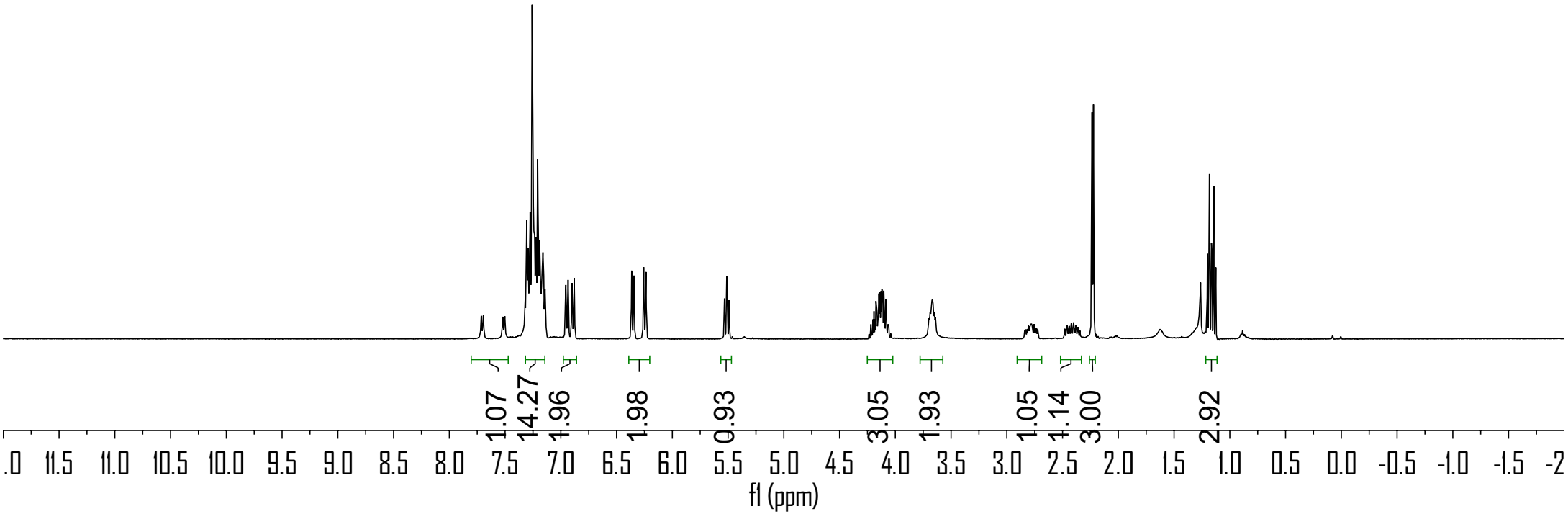

7.2930
7.2880
7.2833
7.2764
7.2661
7.2580
7.2549
7.2450
7.2332
7.2237
7.2109
7.1999
7.1856
7.1751
7.1634
6.9317
6.9171
6.3411
6.3272
6.3024
6.2885
4.4992
4.4917
4.4838
4.1855
4.1736
4.1697
4.1617
4.1578
4.1519
4.1460
4.0919
4.0801
3.6797
3.6728
2.2140
1.2653
1.2534
1.2416
1.1608
1.1489
1.1371
0.8999
0.8885
0.8451
0.8336
0.8097
0.7983
0.7014
0.6900
^{1}H NMR (600 MHz, CDCl$_3$)
O
O
NH
O
N
H
Ph
Ph
C21 (dr = 1.2:1)
10.10
1.82
2.01
0.95
2.14
0.84
1.94
1.08
1.11
3.01
1.11
3.21
3.11
3.56
f1 (ppm)

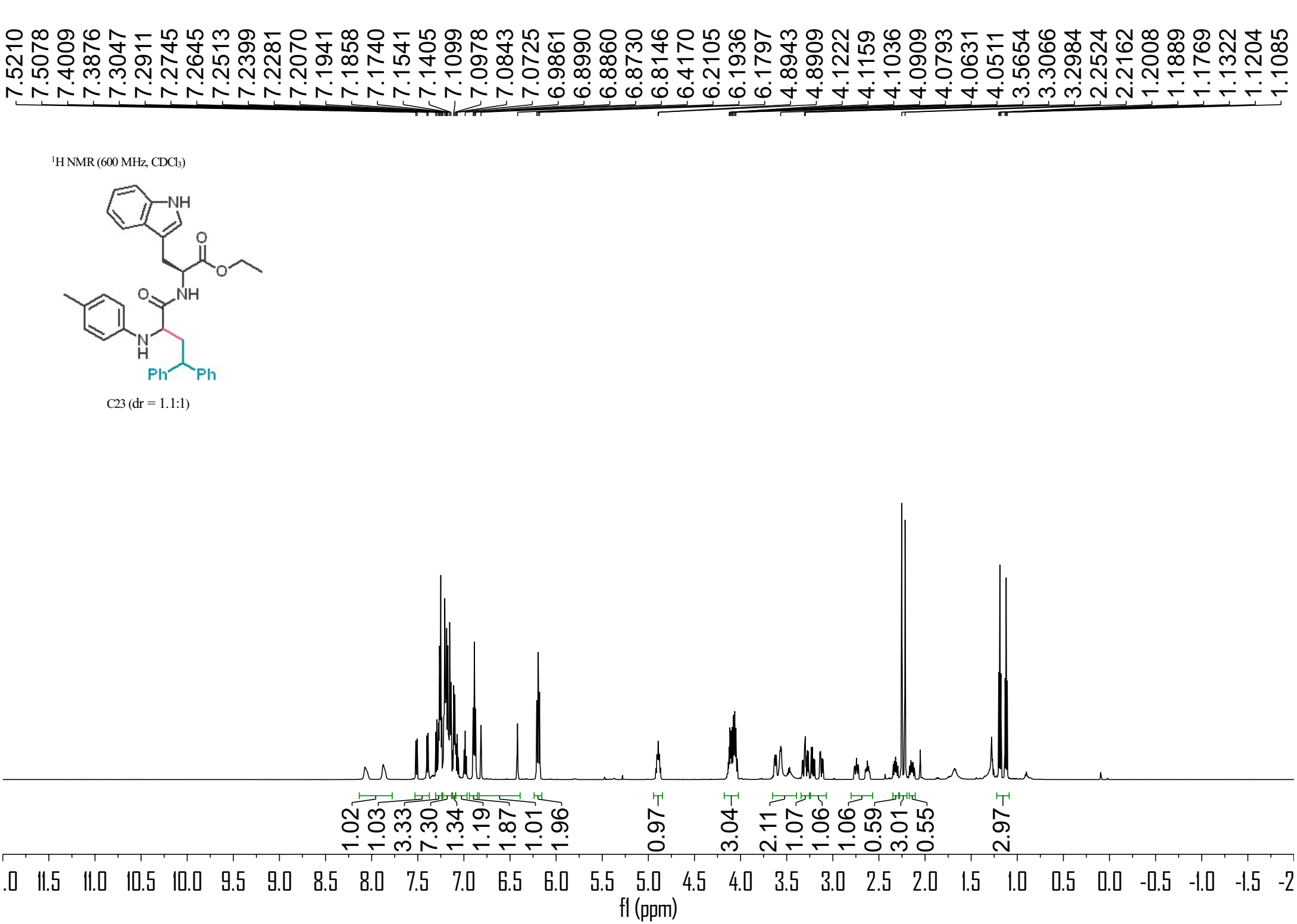
7.7139 7.6949 7.5206 7.5024 7.3243 7.3062 7.2929 7.2749 7.2581 7.2373 7.2209 7.2082 7.1908 7.1707 7.1624 7.1541 7.1426 6.9548 6.9348 6.8999 6.8798 6.3649 6.3440 6.2570 6.2361 5.5323 5.5120 5.4926 4.1917 4.1737 4.1556 4.1378 4.1200 4.1029 4.0855 3.6998 3.6886 3.6673 3.6491 3.6386 2.4202 2.4017 2.2360 2.2232 1.2010 1.1832 1.1653 1.1614 1.1437 1.1259

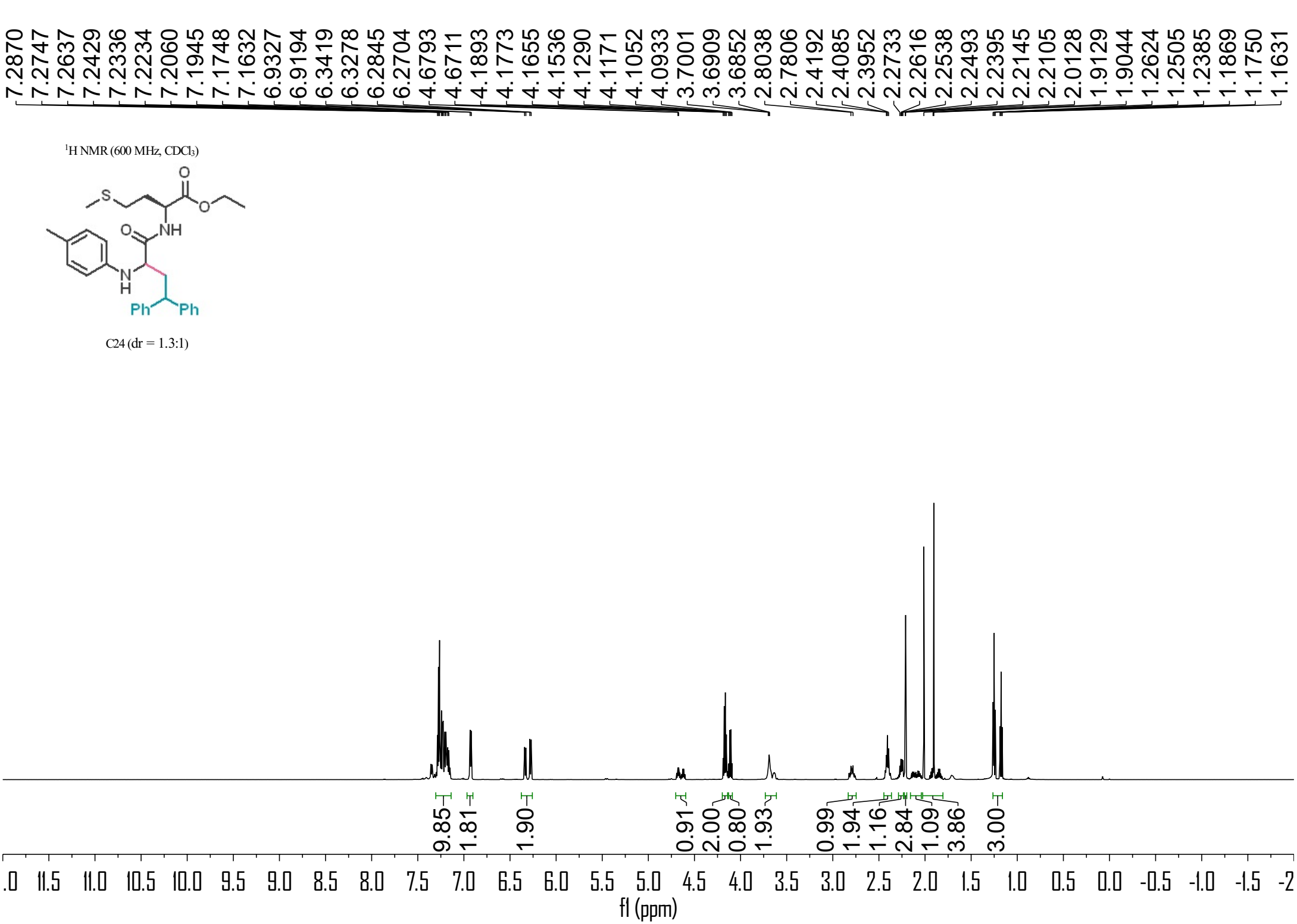
^{1}H NMR (400 MHz, $CDCl_3$)



C22 (dr = 1:1)

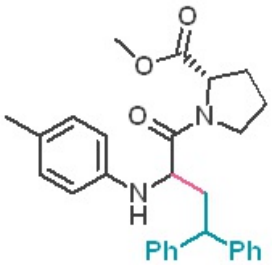



7.5210
7.5078
7.4009
7.3876
7.3047
7.2911
7.2745
7.2645
7.2513
7.2399
7.2281
7.2070
7.1941
7.1858
7.1740
7.1541
7.1405
7.1099
7.0978
7.0843
7.0725
6.9861
6.8990
6.8860
6.8730
6.8146
6.4170
6.2105
6.1936
6.1797
4.8943
4.8909
4.1222
4.1159
4.1036
4.0909
4.0793
4.0631
4.0511
3.5654
3.3066
3.2984
2.2524
2.2162
1.2008
1.1889
1.1769
1.1322
1.1204
1.1085
1H NMR (600 MHz, CDCl3)
NH
O
O
O
NH
N
H
Ph
Ph
C23 (dr = 1.1:1)
1.02
1.03
3.33
7.30
1.34
1.19
1.87
1.01
1.96
0.97
3.04
2.11
1.07
1.06
1.06
0.59
3.01
0.55
2.97
f1 (ppm)


7.2870
7.2747
7.2637
7.2429
7.2336
7.2234
7.2060
7.1945
7.1748
7.1632
6.9327
6.9194
6.3419
6.3278
6.2845
6.2704
4.6793
4.6711
4.1893
4.1773
4.1655
4.1536
4.1290
4.1171
4.1052
4.0933
3.7001
3.6909
3.6852
2.8038
2.7806
2.4192
2.4085
2.3952
2.2733
2.2616
2.2538
2.2493
2.2395
2.2145
2.2105
2.0128
1.9129
1.9044
1.2624
1.2505
1.2385
1.1869
1.1750
1.1631
1H NMR (600 MHz, CDCl3)
O
S
O
NH
O
N
H
Ph
Ph
C24 (dr = 1.3:1)
9.85
1.81
1.90
0.91
2.00
0.80
1.93
0.99
1.94
1.16
2.84
1.09
3.86
3.00
f1 (ppm)

7.3126 7.2990 7.2872 7.2698 7.2498 7.2385 7.2150 7.2125 7.2005 7.1913 7.1886 7.1802 7.1715 6.9441 6.9306 6.4243 6.4104 4.3911 4.3857 4.3768 4.3730 4.2520 4.2389 4.2259 3.9397 3.5504 3.5208 3.3319 3.3286 3.3200 2.9710 2.9549 2.4454 2.4361 2.4310 2.4154 2.4035 2.3918 2.2191 2.0734 2.0388 1.9060 1.8977 1.8894 1.8829 1.8796 1.8034 1.7972 1.7946 1.7911

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



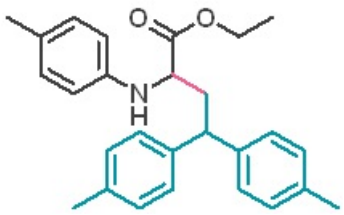
C25 (dr = 6:1)

8.22 2.07 1.78 2.08 1.04 0.95 0.92 1.01 3.00 0.97 0.95 1.87 2.95 1.83 2.20 1.24

.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2

f1 (ppm)

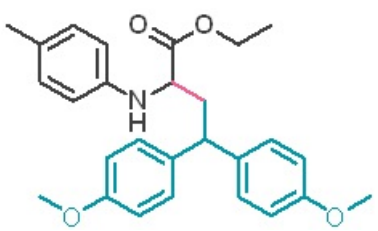
^{1}H NMR (400 MHz, $CDCl_3$)



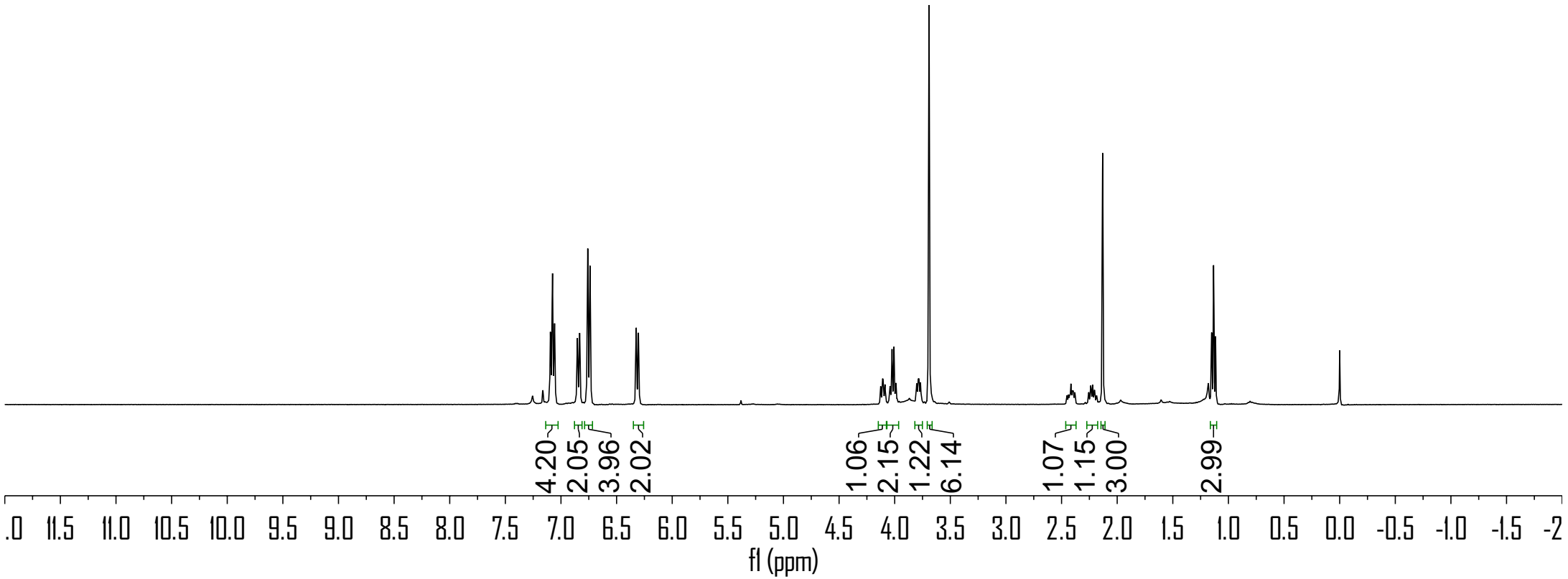
C26

7.0967
7.0756
7.0580
6.8522
6.8324
6.7583
6.7394
6.3223
6.3044
4.1264
4.1102
4.1035
4.0873
4.0419
4.0240
4.0062
3.9886
3.8025
3.7894
3.7812
3.7680
3.6911
2.4511
2.4384
2.4281
2.4161
2.4040
2.3938
2.3808
2.2548
2.2373
2.2203
2.2036
2.1850
2.1306
1.1518
1.1339
1.1162

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

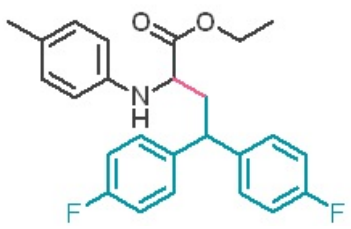


C27

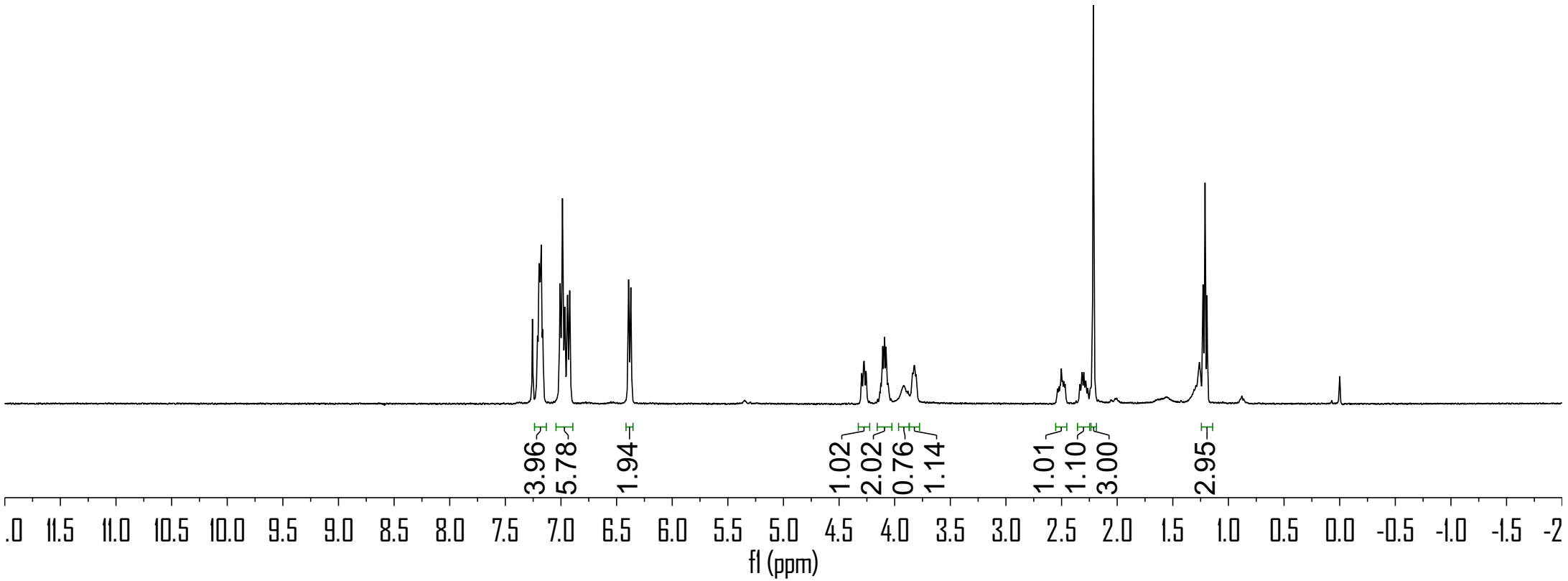


7.2113 7.1984 7.1924 7.1847 7.1770 7.1629 7.0182 7.0077 6.9866 6.9655 6.9408 6.9210 6.3915 6.3719 4.2965 4.2814 4.2739 4.2575 4.1347 4.1256 4.1161 4.1078 4.0971 4.0901 4.0788 4.0711 4.0607 4.0515 3.9184 3.8409 3.8284 3.8185 3.8056 2.5371 2.5254 2.5142 2.5022 2.4903 2.4788 2.4674 2.3353 2.3166 2.2995 2.2818 2.2642 2.2145 1.2282 1.2105 1.1928

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

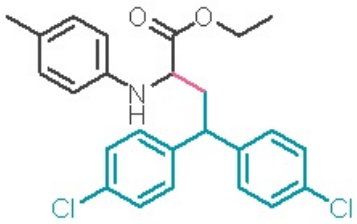


C28

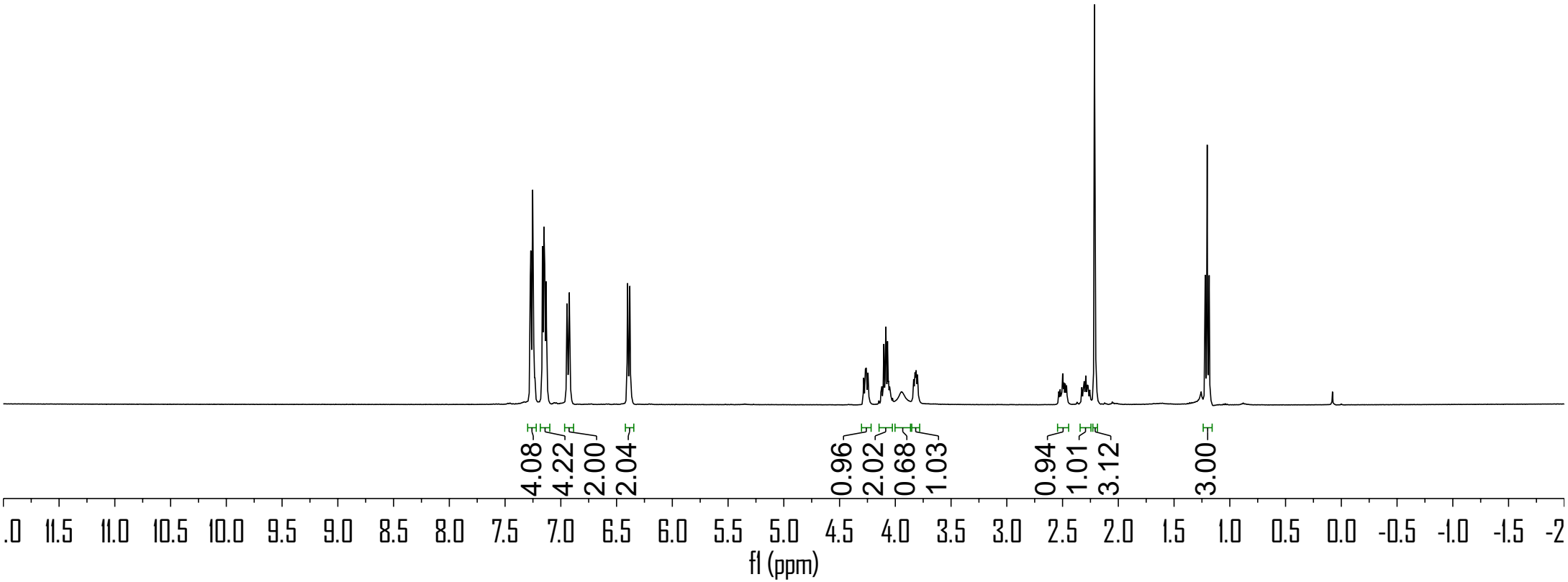


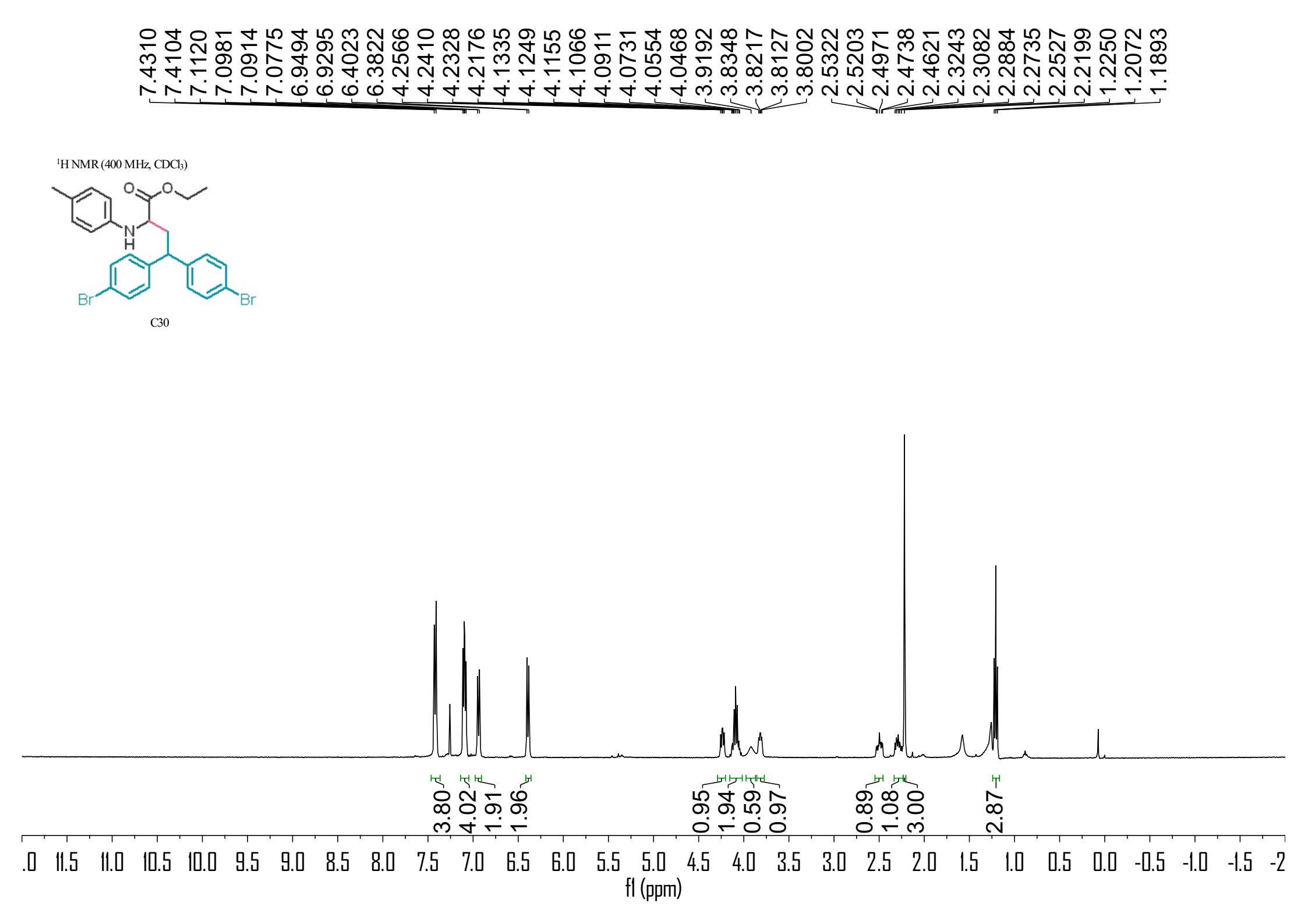
7.2740 7.2691 7.2530 7.2478 7.2331 7.1646 7.1591 7.1518 7.1439 7.1309 6.9442 6.9241 6.4020 6.3812 4.2837 4.2686 4.2601 4.2448 4.1294 4.1203 4.1027 4.0878 4.0844 4.0701 4.0523 4.0430 3.9426 3.8357 3.8231 3.8137 3.8010 2.5340 2.5218 2.4990 2.4760 2.4638 2.3274 2.3055 2.2914 2.2778 2.2560 2.2135 1.2203 1.2025 1.1847

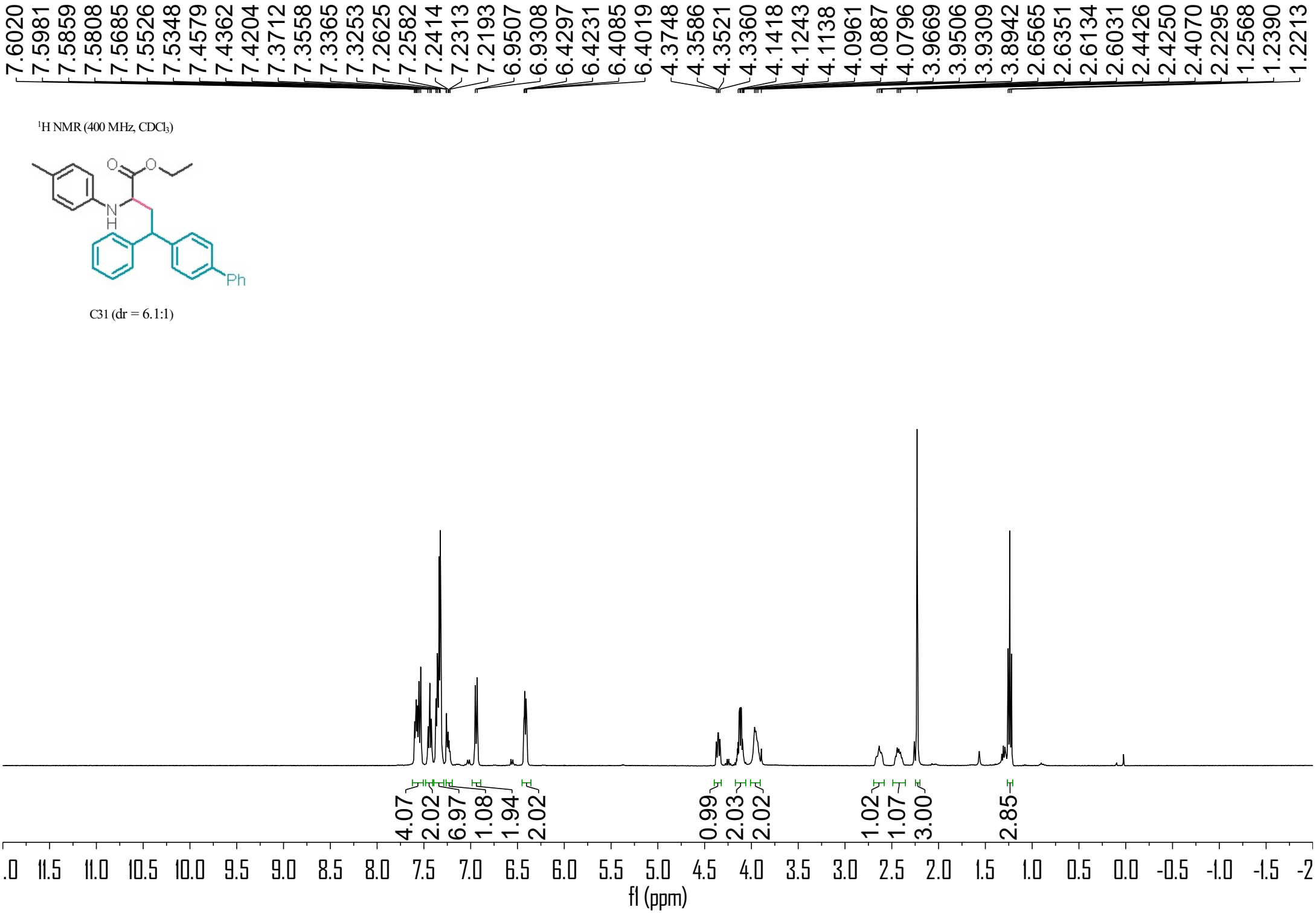
^{1}H NMR (400 MHz, $CDCl_3$)



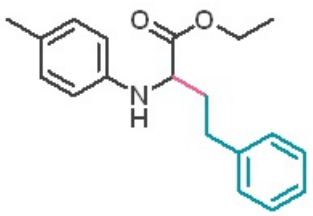
C29




7.4310
7.4104
7.1120
7.0981
7.0914
7.0775
6.9494
6.9295
6.4023
6.3822
4.2566
4.2410
4.2328
4.2176
4.1335
4.1249
4.1155
4.1066
4.0911
4.0731
4.0554
4.0468
3.9192
3.8348
3.8217
3.8127
3.8002
2.5322
2.5203
2.4971
2.4738
2.4621
2.3243
2.3082
2.2884
2.2735
2.2527
2.2199
1.2250
1.2072
1.1893
^{1}H NMR (400 MHz, $CDCl_3$)
O
O
N
H
Br
Br
C30
3.80
4.02
1.91
1.96
0.95
1.94
0.59
0.97
0.89
1.08
3.00
2.87
.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

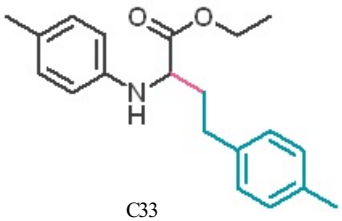

7.6020
7.5981
7.5859
7.5808
7.5685
7.5526
7.5348
7.4579
7.4362
7.4204
7.3712
7.3558
7.3365
7.3253
7.2625
7.2582
7.2414
7.2313
7.2193
6.9507
6.9308
6.4297
6.4231
6.4085
6.4019
4.3748
4.3586
4.3521
4.3360
4.1418
4.1243
4.1138
4.0961
4.0887
4.0796
3.9669
3.9506
3.9309
3.8942
2.6565
2.6351
2.6134
2.6031
2.4426
2.4250
2.4070
2.2295
1.2568
1.2390
1.2213
^{1}H NMR (400 MHz, $CDCl_3$)
O
O
N
H
Ph
C31 (dr = 6.1:1)
4.07
2.02
6.97
1.08
1.94
2.02
0.99
2.03
2.02
1.02
1.07
3.00
2.85
f1 (ppm)

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

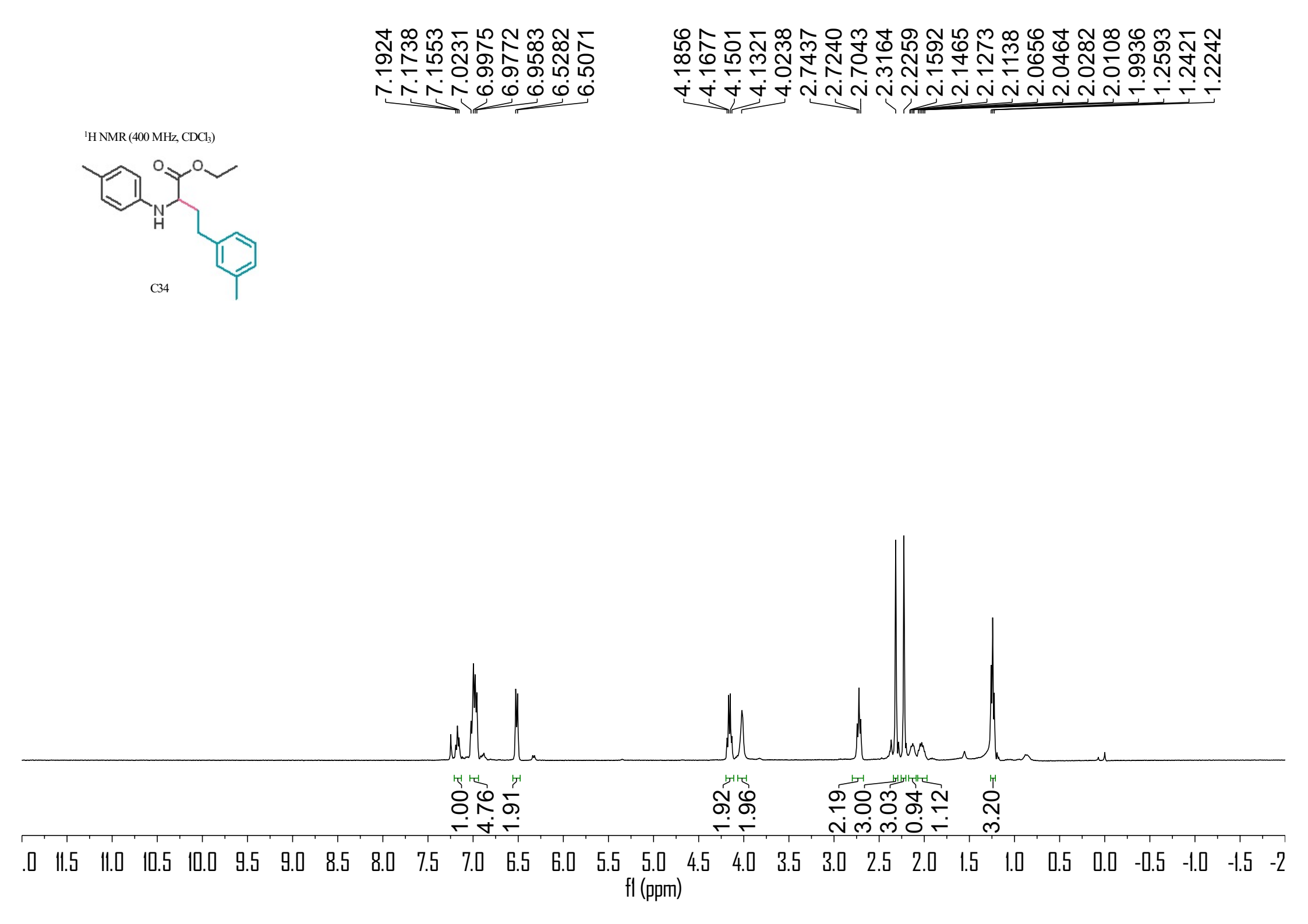


C32

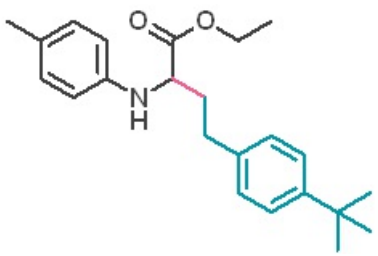
^{1}H NMR (600 MHz, $CDCl_3$)



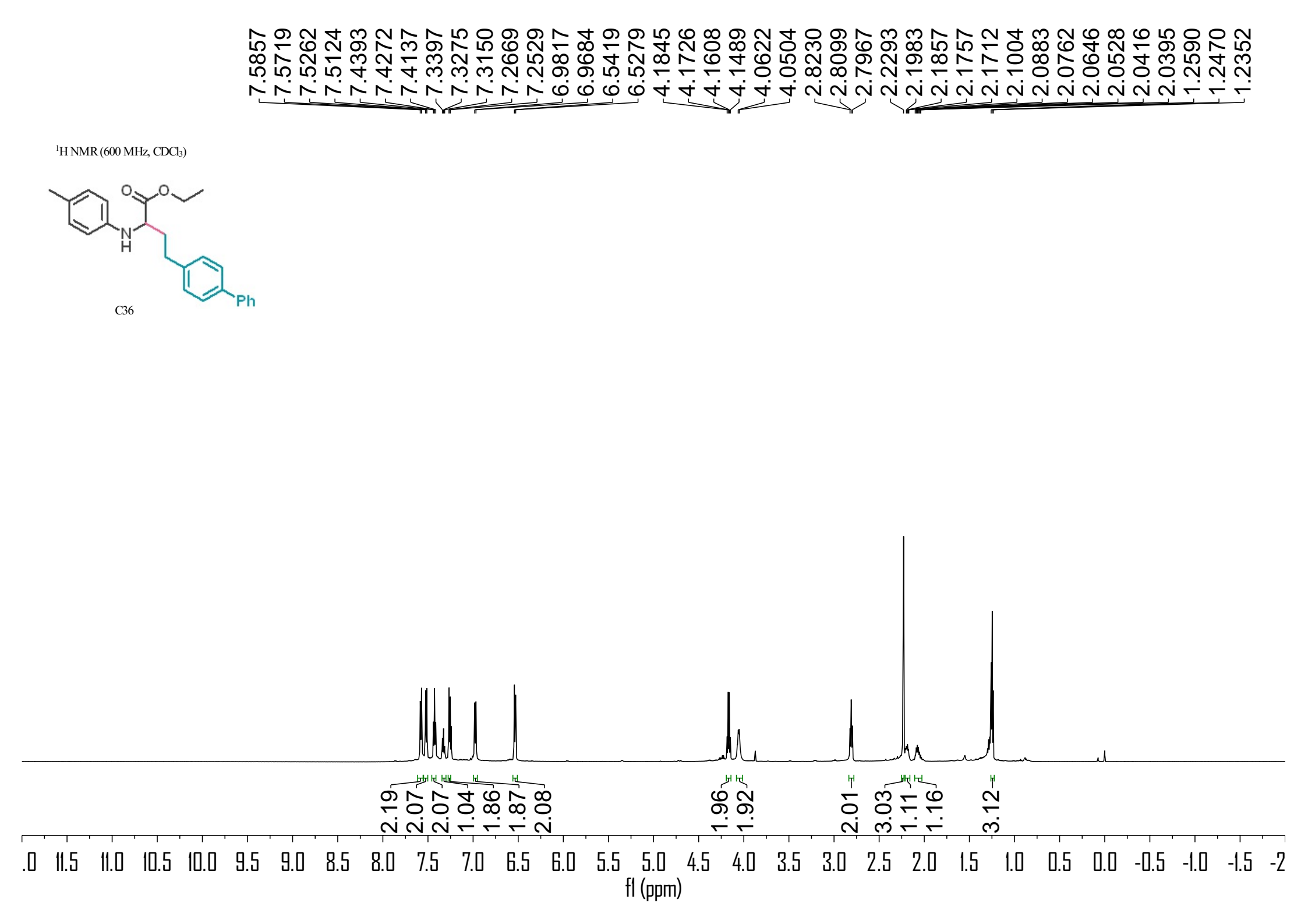
C33


7.1924
7.1738
7.1553
7.0231
6.9975
6.9772
6.9583
6.5282
6.5071
4.1856
4.1677
4.1501
4.1321
4.0238
2.7437
2.7240
2.7043
2.3164
2.2259
2.1592
2.1465
2.1273
2.1138
2.0656
2.0464
2.0282
2.0108
1.9936
1.2593
1.2421
1.2242
^{1}H NMR (400 MHz, $CDCl_3$)
O
O
N
H
C34
1.00
4.76
1.91
1.92
1.96
2.19
3.00
3.03
0.94
1.12
3.20
.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

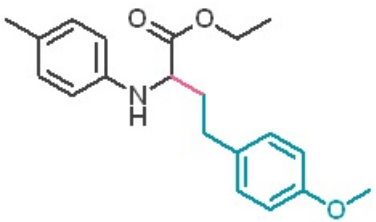
^{1}H NMR (400 MHz, $CDCl_3$)



C35

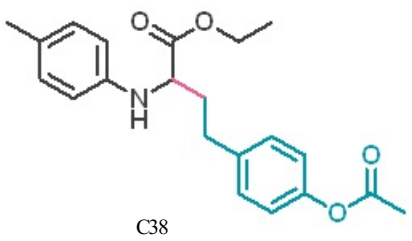

7.5857
7.5719
7.5262
7.5124
7.4393
7.4272
7.4137
7.3397
7.3275
7.3150
7.2669
7.2529
6.9817
6.9684
6.5419
6.5279
4.1845
4.1726
4.1608
4.1489
4.0622
4.0504
2.8230
2.8099
2.7967
2.2293
2.1983
2.1857
2.1757
2.1712
2.1004
2.0883
2.0762
2.0646
2.0528
2.0416
2.0395
1.2590
1.2470
1.2352
^{1}H NMR (600 MHz, $CDCl_3$)
O
O
N
H
Ph
C36
2.19
2.07
2.07
1.04
1.86
1.87
2.08
1.96
1.92
2.01
3.03
1.11
1.16
3.12
.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)

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

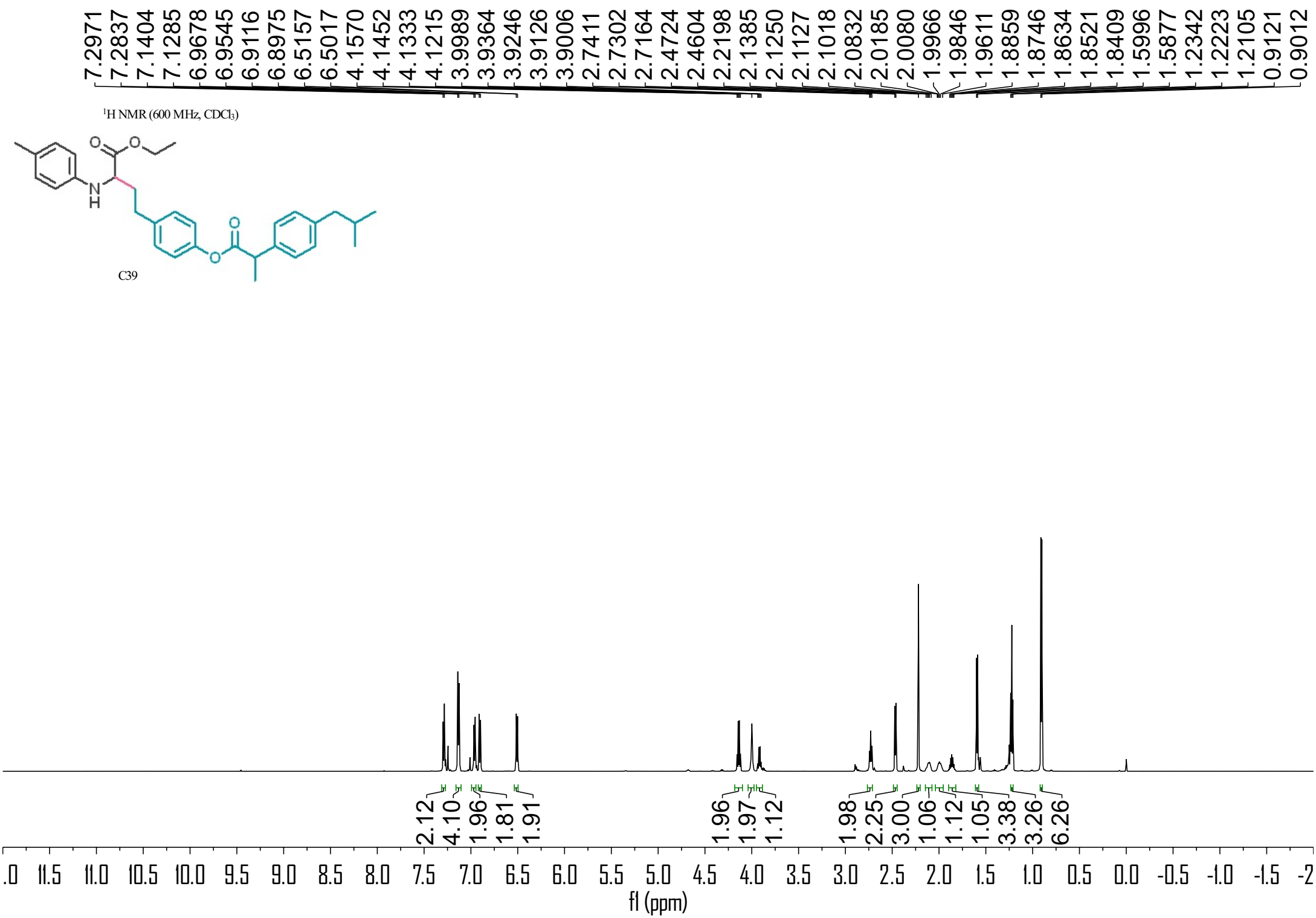


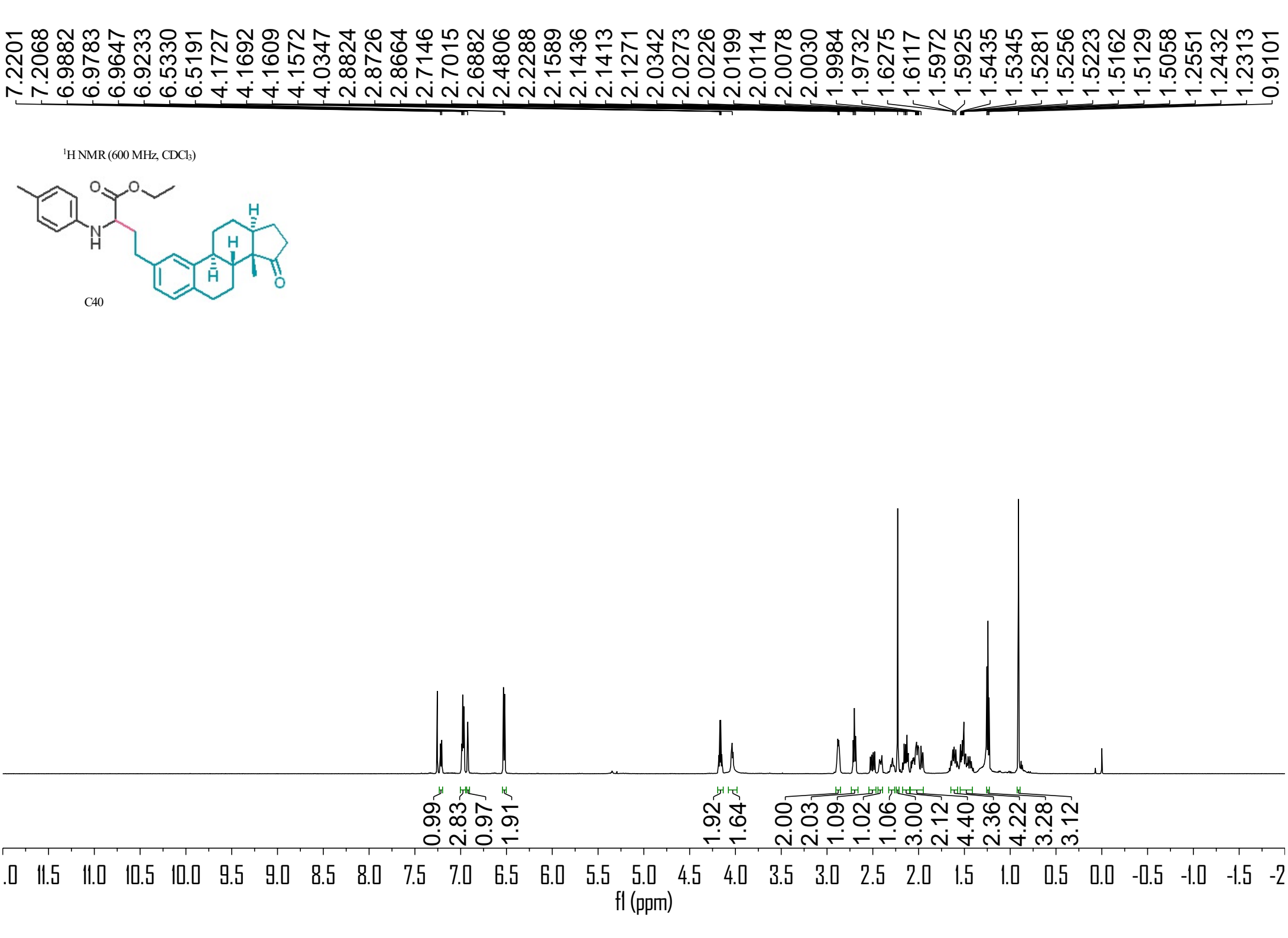
C37

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

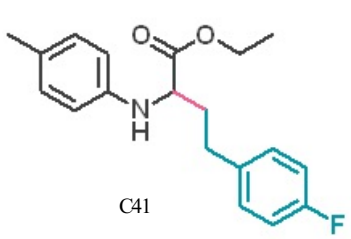


C38


7.2971
7.2837
7.1404
7.1285
6.9678
6.9545
6.9116
6.8975
6.5157
6.5017
4.1570
4.1452
4.1333
4.1215
3.9989
3.9364
3.9246
3.9126
3.9006
2.7411
2.7302
2.7164
2.4724
2.4604
2.2198
2.1385
2.1250
2.1127
2.1018
2.0832
2.0185
2.0080
1.9966
1.9846
1.9611
1.8859
1.8746
1.8634
1.8521
1.8409
1.5996
1.5877
1.2342
1.2223
1.2105
0.9121
0.9012
1H NMR (600 MHz, CDCl3)
O
O
N
H
O
O
C39
2.12
4.10
1.96
1.81
1.91
1.96
1.97
1.12
1.98
2.25
3.00
1.06
1.12
1.05
3.38
3.26
6.26
.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2
f1 (ppm)

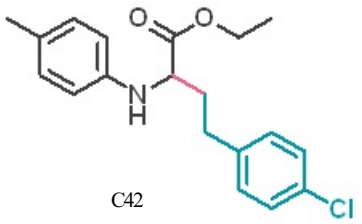

7.2201
7.2068
6.9882
6.9783
6.9647
6.9233
6.5330
6.5191
4.1727
4.1692
4.1609
4.1572
4.0347
2.8824
2.8726
2.8664
2.7146
2.7015
2.6882
2.4806
2.2288
2.1589
2.1436
2.1413
2.1271
2.0342
2.0273
2.0226
2.0199
2.0114
2.0078
2.0030
1.9984
1.9732
1.6275
1.6117
1.5972
1.5925
1.5435
1.5345
1.5281
1.5256
1.5223
1.5162
1.5129
1.5058
1.2551
1.2432
1.2313
0.9101
1H NMR (600 MHz, CDCl3)
O
O
N
H
H
H
H
O
C40
0.99
2.83
0.97
1.91
1.92
1.64
2.00
2.03
1.09
1.02
1.06
3.00
2.12
4.40
2.36
4.22
3.28
3.12
.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2
f1 (ppm)

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



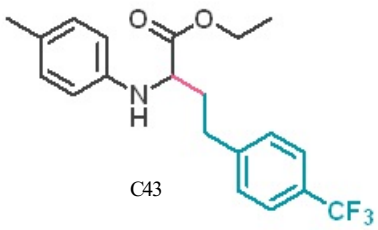
C41

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

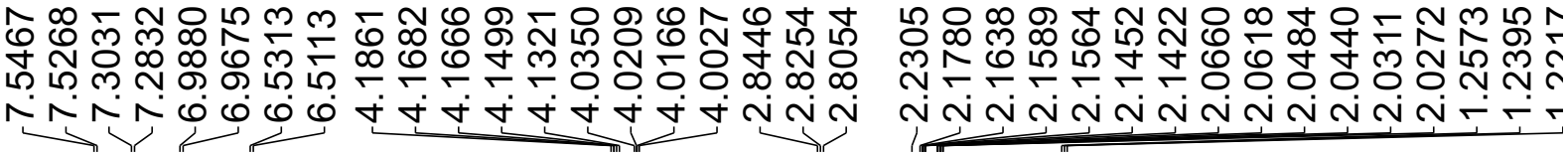


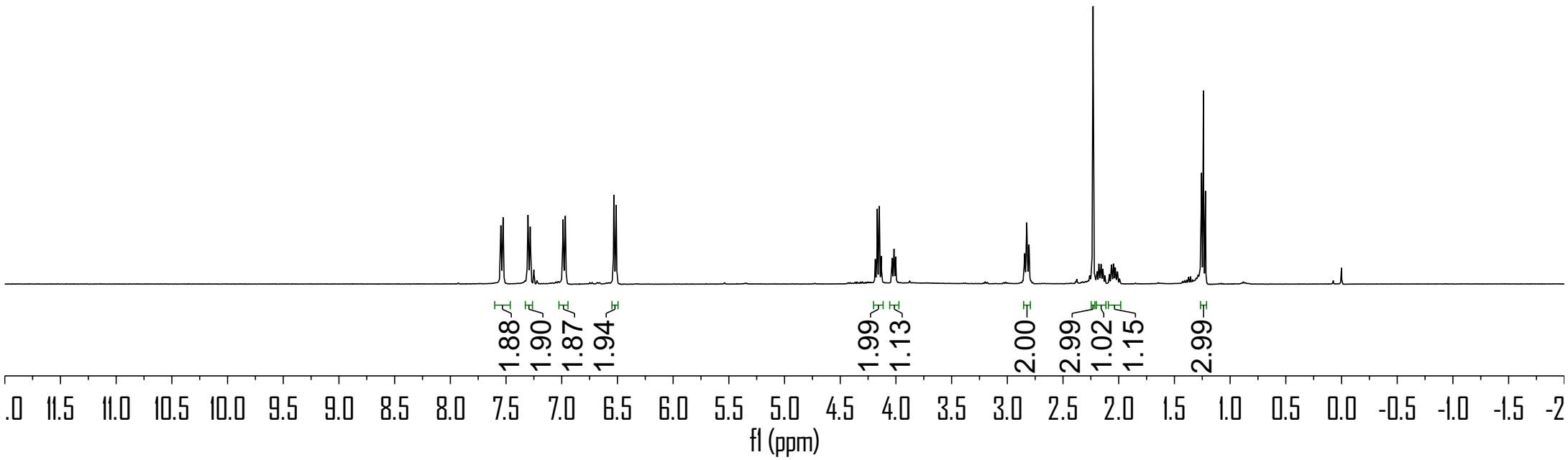
C42

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



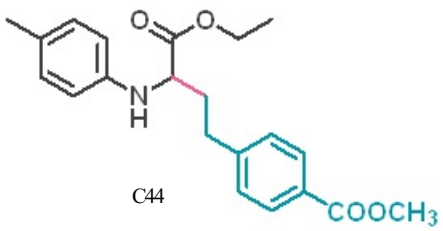
C43



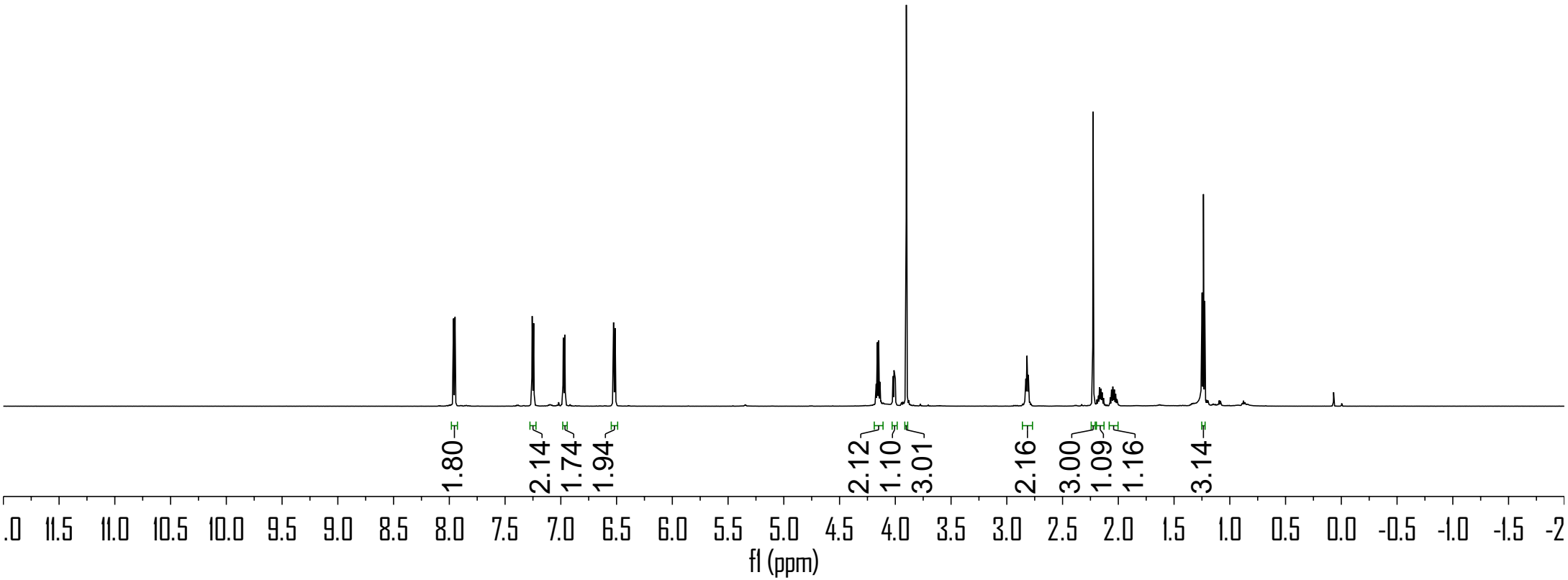


7.9624 7.9485 7.2570 7.2433 6.9776 6.9641 6.5270 6.5129 4.1732 4.1615 4.1495 4.1375 4.0214 4.0120 4.0091 3.9998 3.9013 2.8341 2.8293 2.8188 2.8072 2.2260 2.1917 2.1824 2.1792 2.1773 2.1687 2.1646 2.1594 2.1564 2.1542 2.1474 2.1445 2.1418 2.1326 2.0724 2.0612 2.0580 2.0493 2.0458 2.0380 2.0348 2.0259 2.0233 2.0118 1.2485 1.2366 1.2248

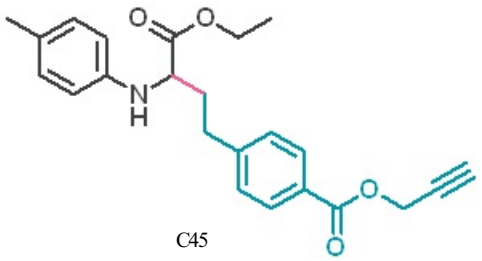
^{1}H NMR (600 MHz, $CDCl_3$)



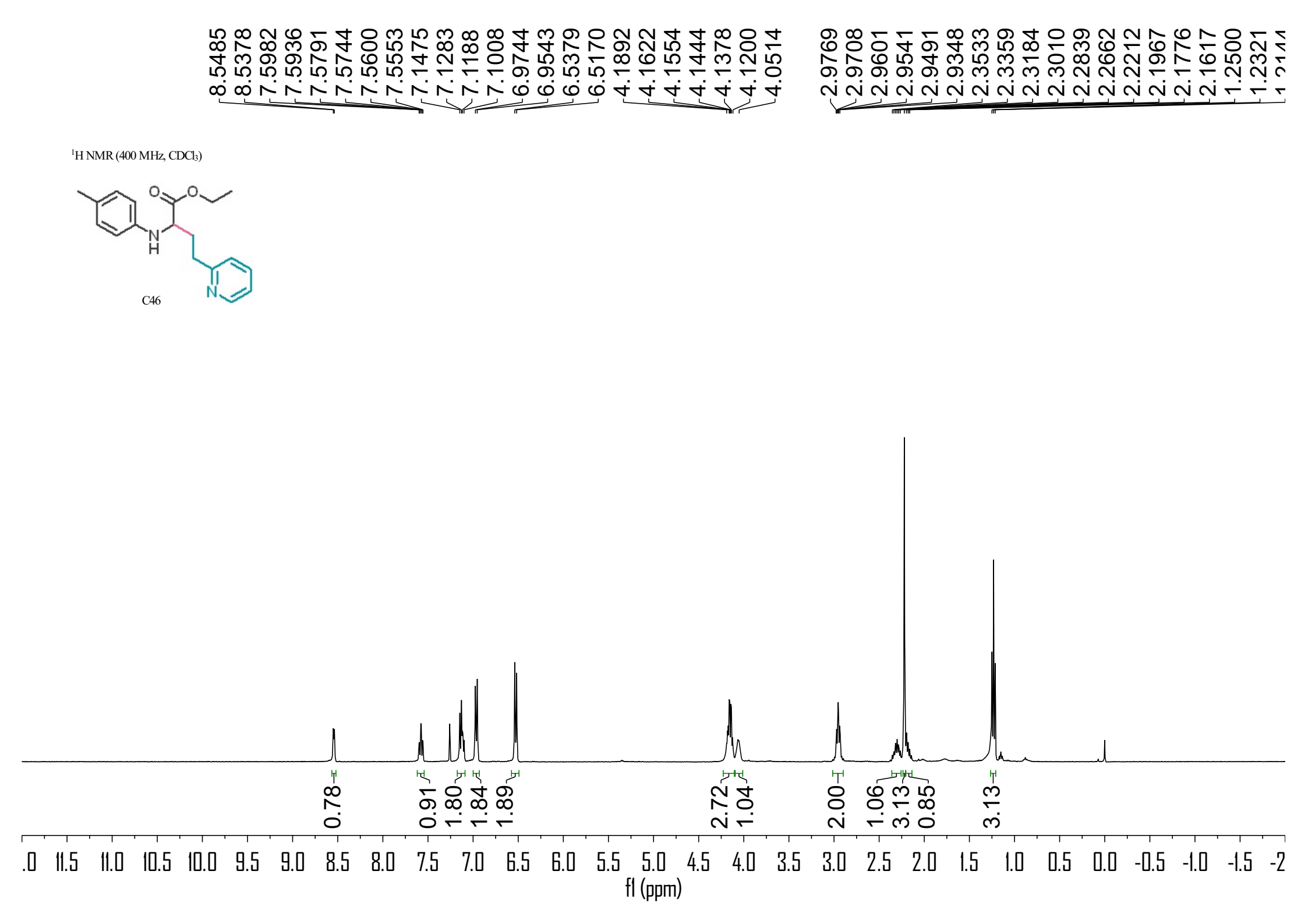
C44



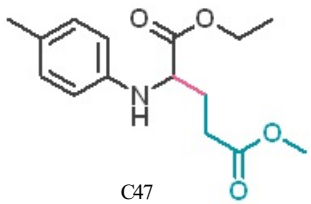
^{1}H NMR (400 MHz, $CDCl_3$)



C45

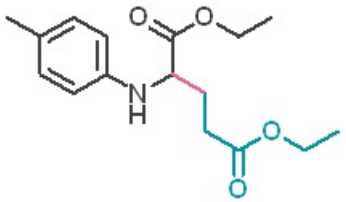

8.5485
8.5378
7.5982
7.5936
7.5791
7.5744
7.5600
7.5553
7.1475
7.1283
7.1188
7.1008
6.9744
6.9543
6.5379
6.5170
4.1892
4.1622
4.1554
4.1444
4.1378
4.1200
4.0514
2.9769
2.9708
2.9601
2.9541
2.9491
2.9348
2.3533
2.3359
2.3184
2.3010
2.2839
2.2662
2.2212
2.1967
2.1776
2.1617
2.1500
1.2321
1H NMR (400 MHz, CDCl3)
O
O
N
H
N
C46
0.78
0.91
1.80
1.84
1.89
2.72
1.04
2.00
1.06
3.13
0.85
3.13
.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2
f1 (ppm)

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



C47

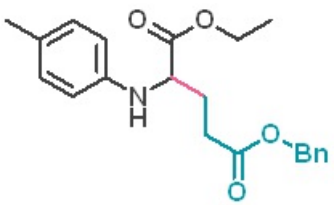
^{1}H NMR (400 MHz, $CDCl_3$)



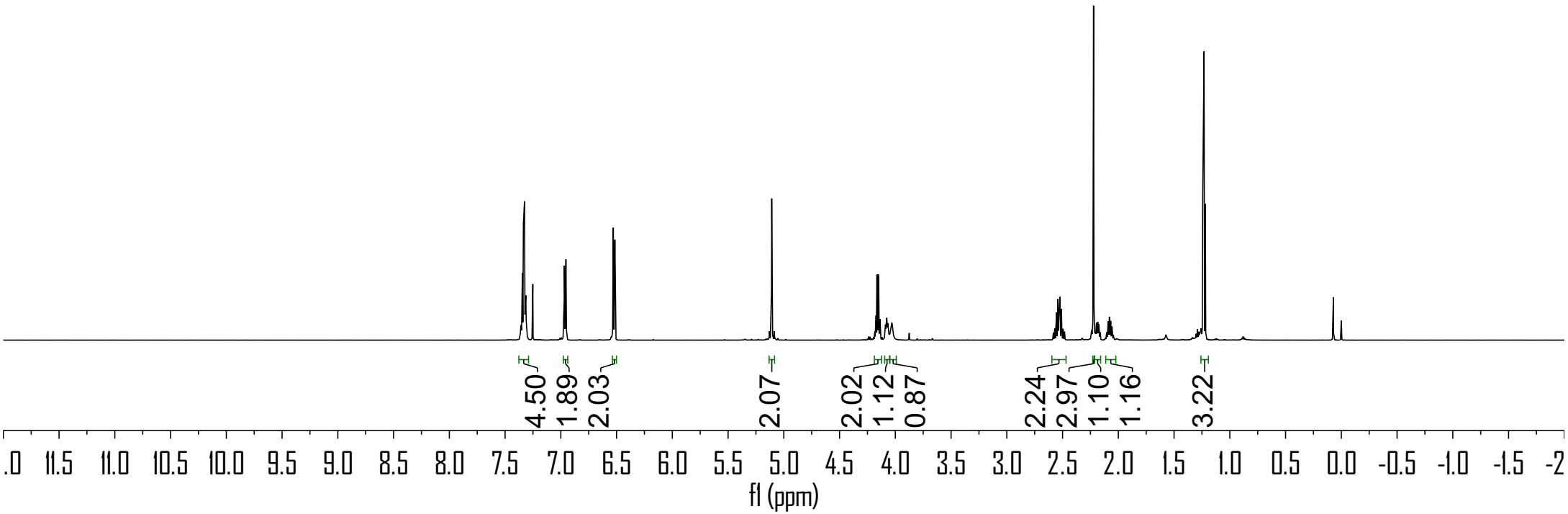
C48

7.3583 7.3447 7.3357 7.3271 7.3252 7.3171 7.3137 7.3104 6.9684 6.9542 6.5293 6.5154 5.1085 4.1835 4.1772 4.1727 4.1653 4.1608 4.1534 4.1490 4.1414 4.1370 4.1309 4.0886 4.0794 4.0767 4.0664 4.0307 2.5820 2.5541 2.5425 2.5230 2.5101 2.4823 2.2221 2.2068 2.1952 2.1837 2.1748 2.1619 2.1044 2.0913 2.0804 2.0676 2.0567 2.0437 1.2429 1.2311 1.2192

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

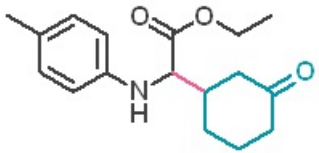


C49

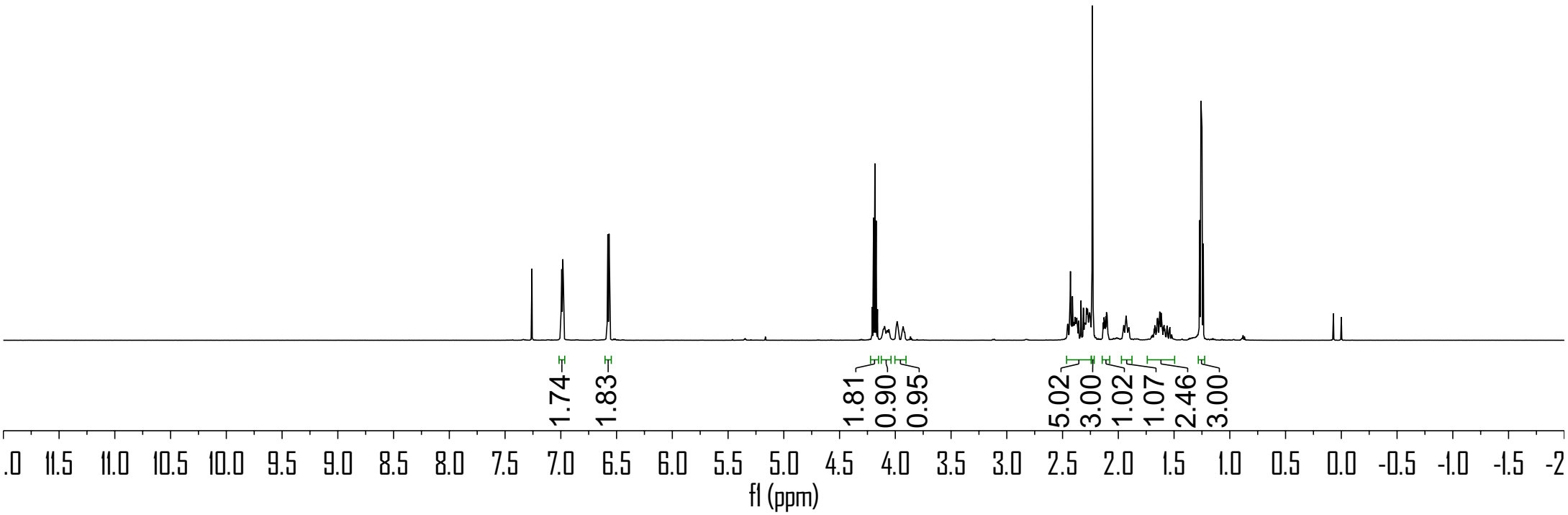


6.9956
6.9914
6.9817
6.9774
6.5814
6.5781
6.5673
6.5640
4.2066
4.1948
4.1832
4.1714
4.1596
3.9851
2.4550
2.4293
2.4117
2.3870
2.3754
2.3568
2.3340
2.3130
2.3041
2.2967
2.2924
2.2864
2.2753
2.2702
2.2654
2.2508
2.2331
2.2296
2.1339
2.1286
2.1231
2.1117
2.1064
2.1011
2.0961
1.9298
1.6451
1.6283
1.6155
1.5891
1.2711
1.2616
1.2592
1.2498
1.2474
1.2378

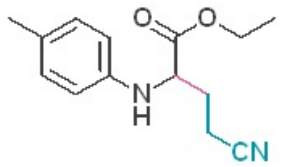
^{1}H NMR (600 MHz, $CDCl_3$)



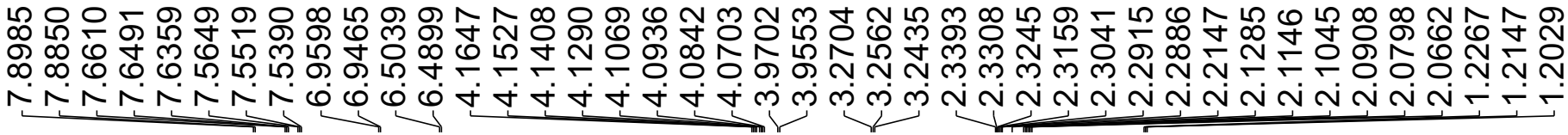
C50 (dr = 1.1:1)



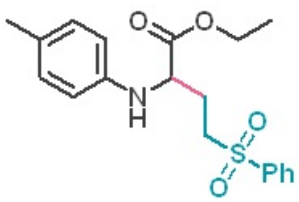
^{1}H NMR (600 MHz, $CDCl_3$)



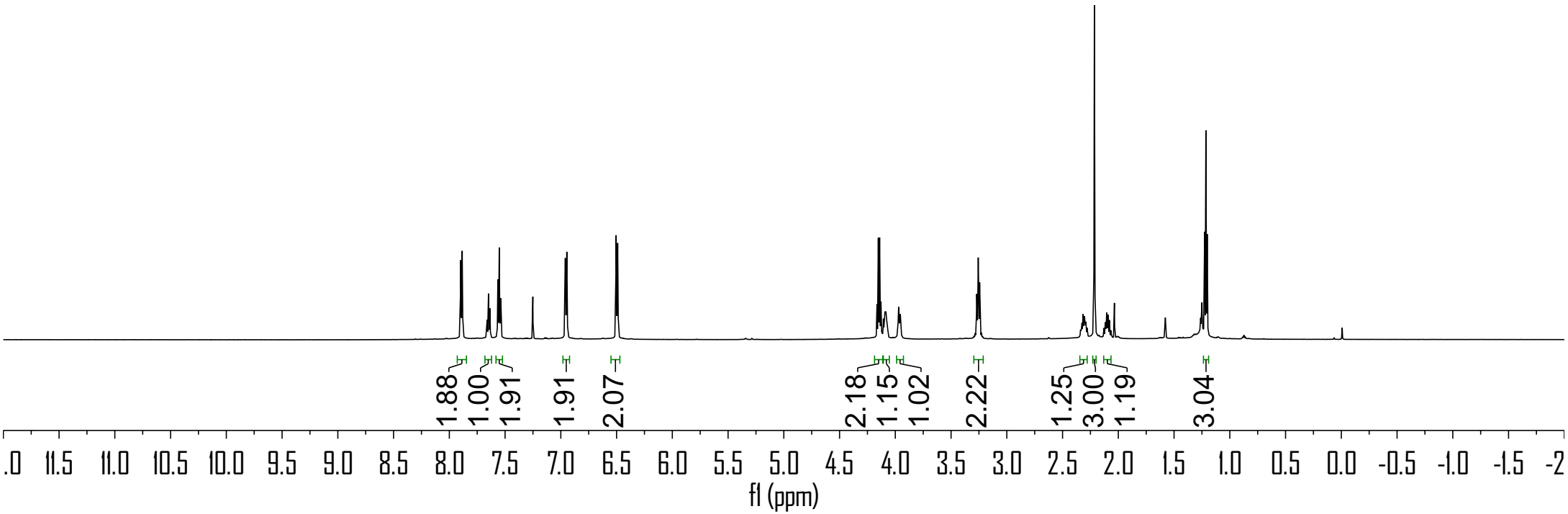
C51

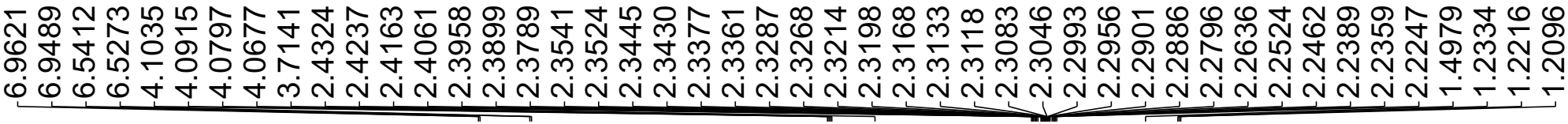


^{1}H NMR (600 MHz, $CDCl_3$)

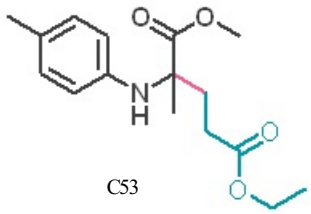


C52

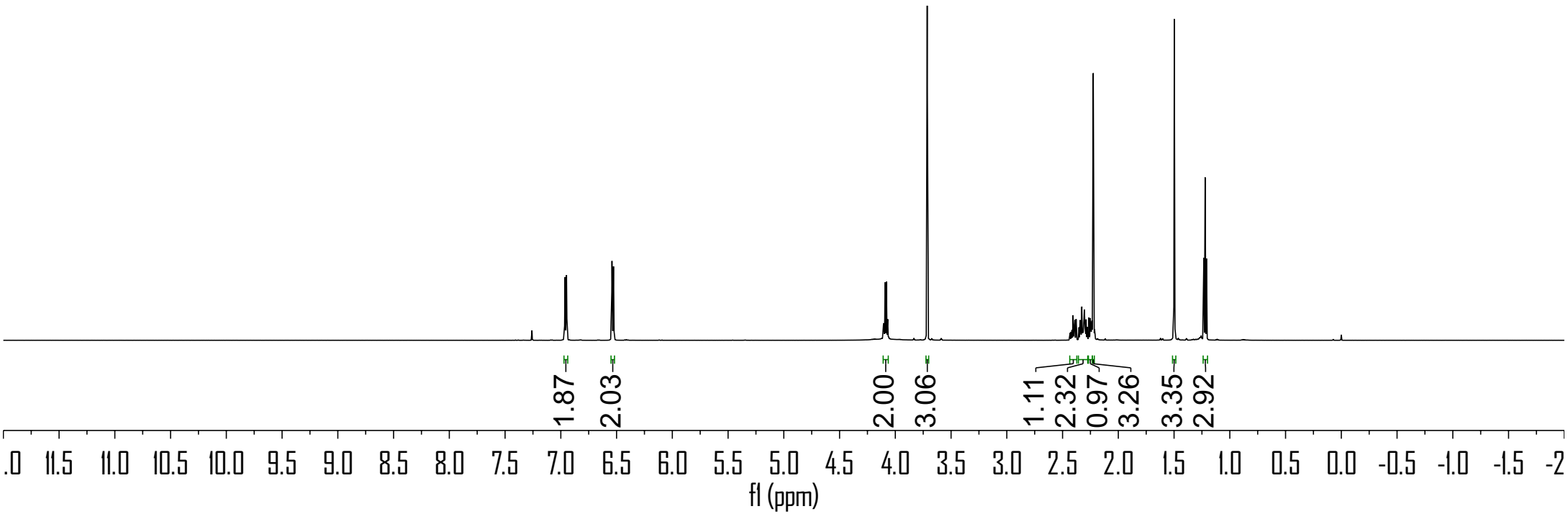




^{1}H NMR (600 MHz, $CDCl_3$)

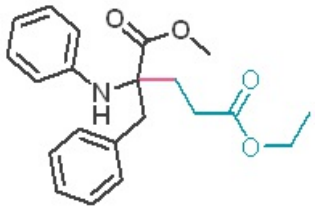


C53

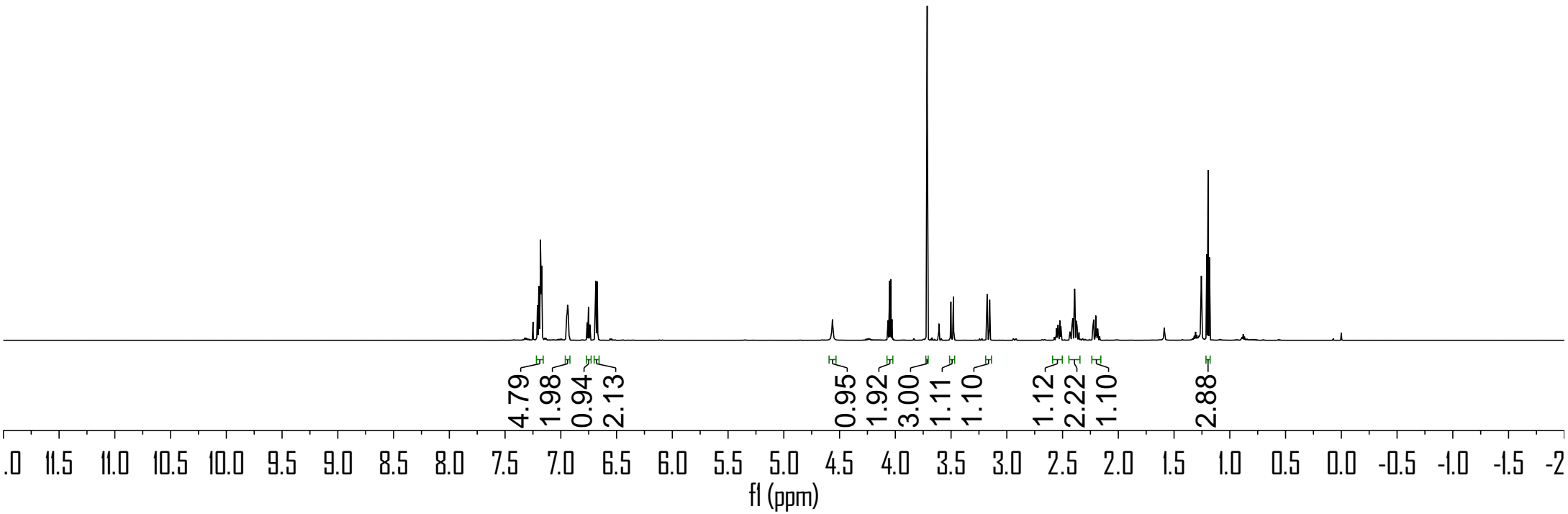


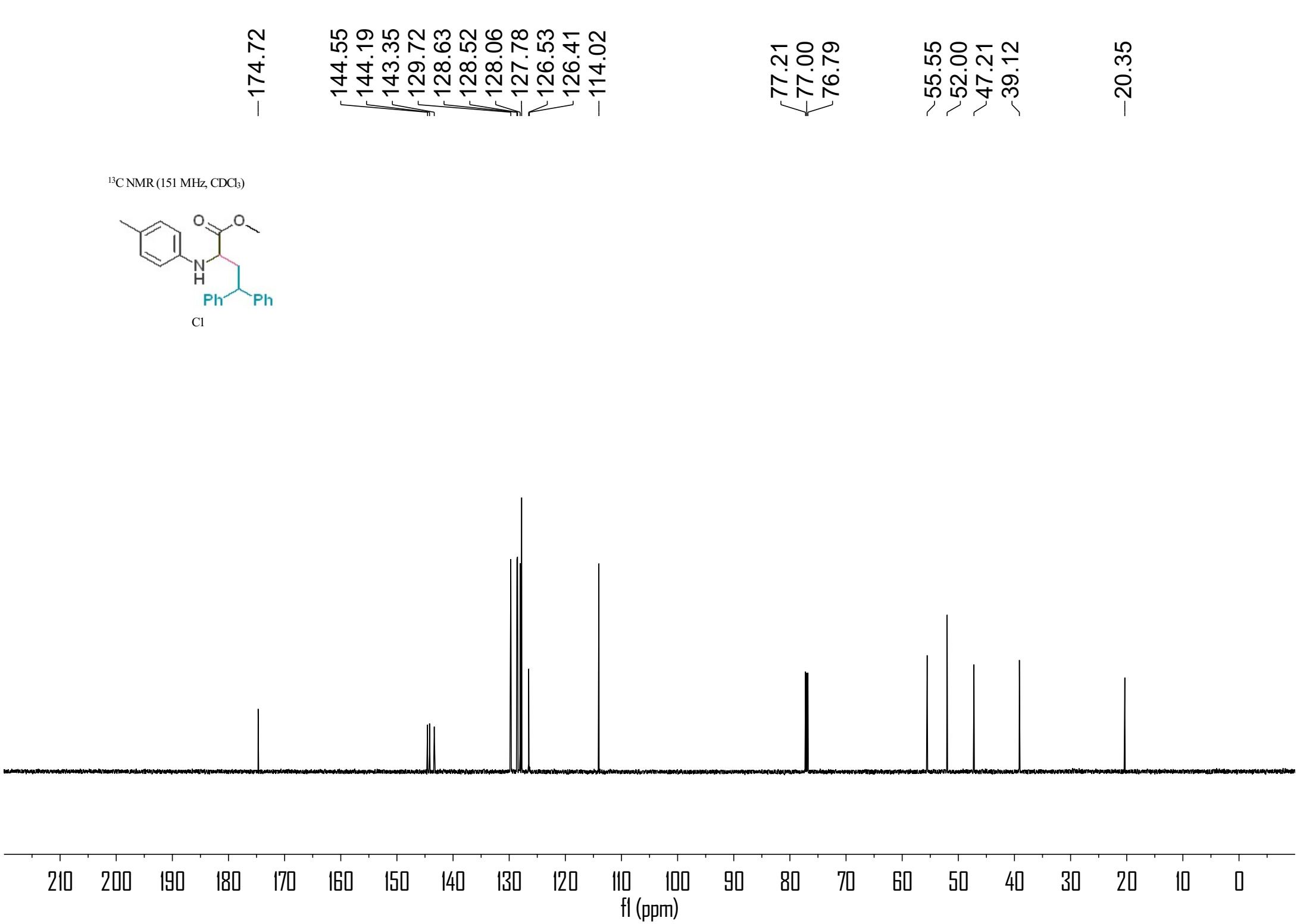
7.2096 7.1975 7.1954 7.1864 7.1831 7.1795 7.1774 7.1749 7.1723 6.9458 6.9412 6.9392 6.9369 6.9340 6.9298 6.7642 6.7520 6.7397 6.6883 6.6864 6.6737 6.6720 4.5642 4.0653 4.0534 4.0415 4.0296 3.7143 3.5016 3.4788 3.1766 3.1536 2.5453 2.5404 2.5375 2.5226 2.5150 2.4162 2.4083 2.3927 2.3899 2.3773 2.3750 2.3696 2.2282 2.2206 2.2024 1.2070 1.1950 1.1831

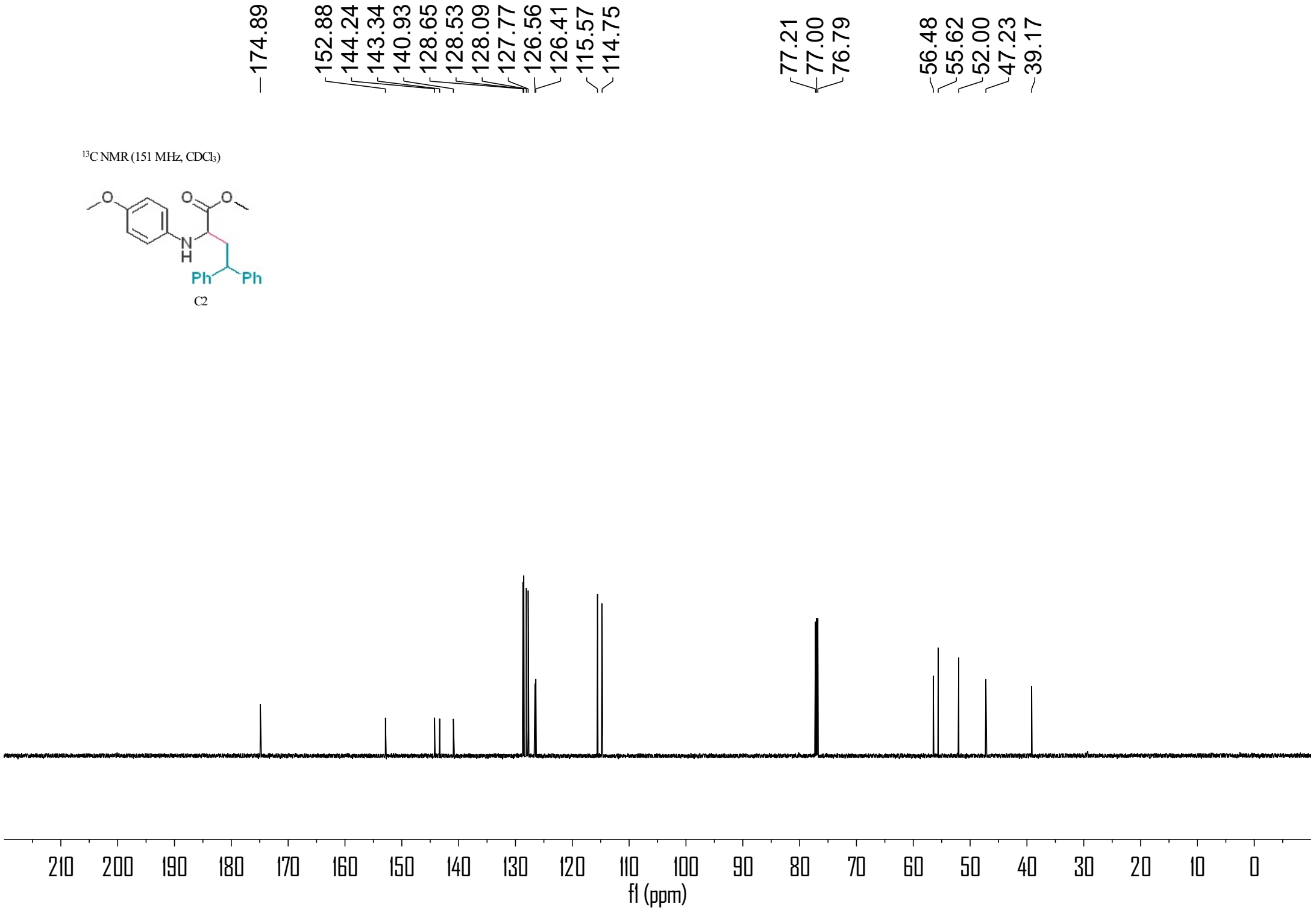
^{1}H NMR (600 MHz, $CDCl_3$)



C54

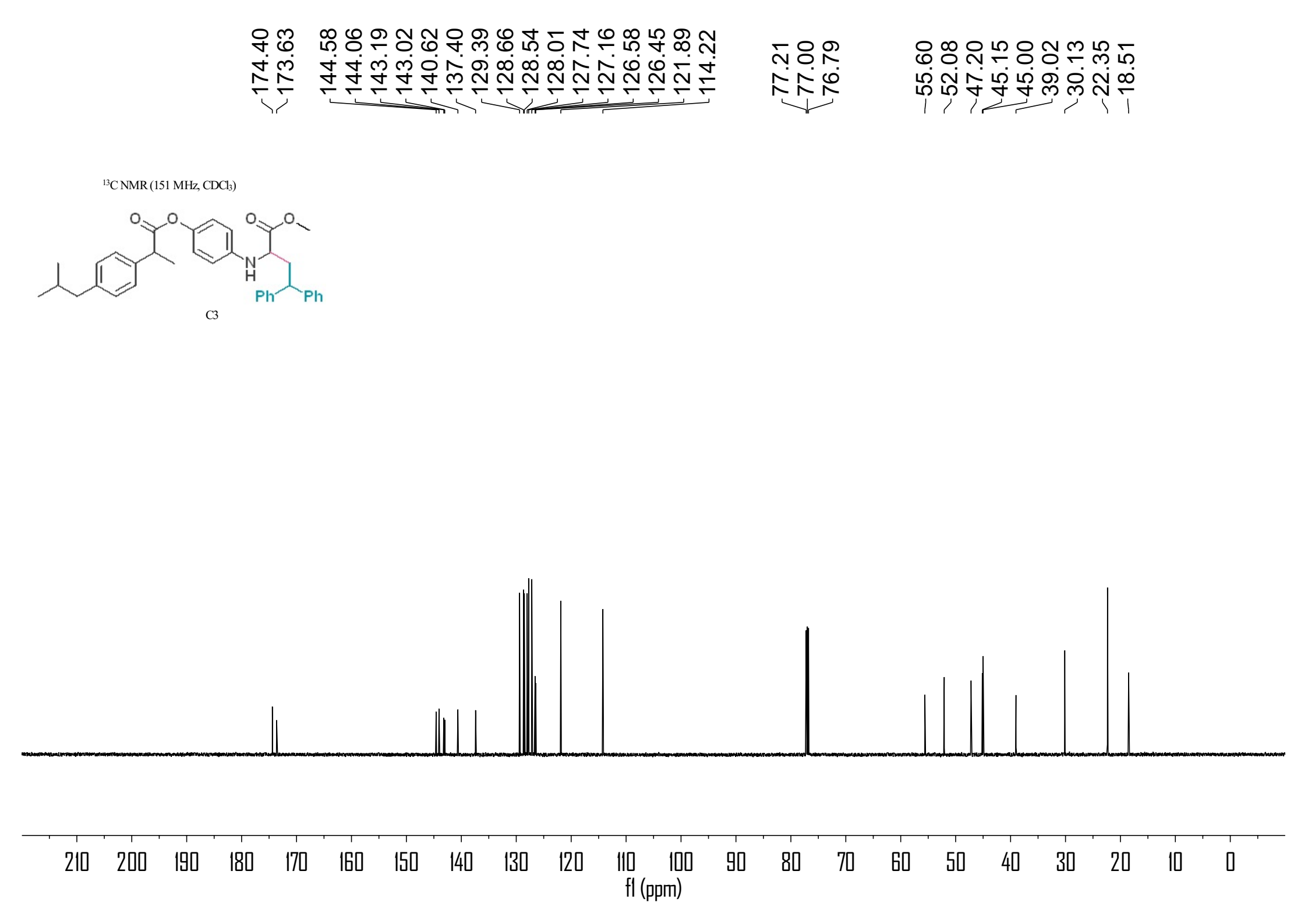


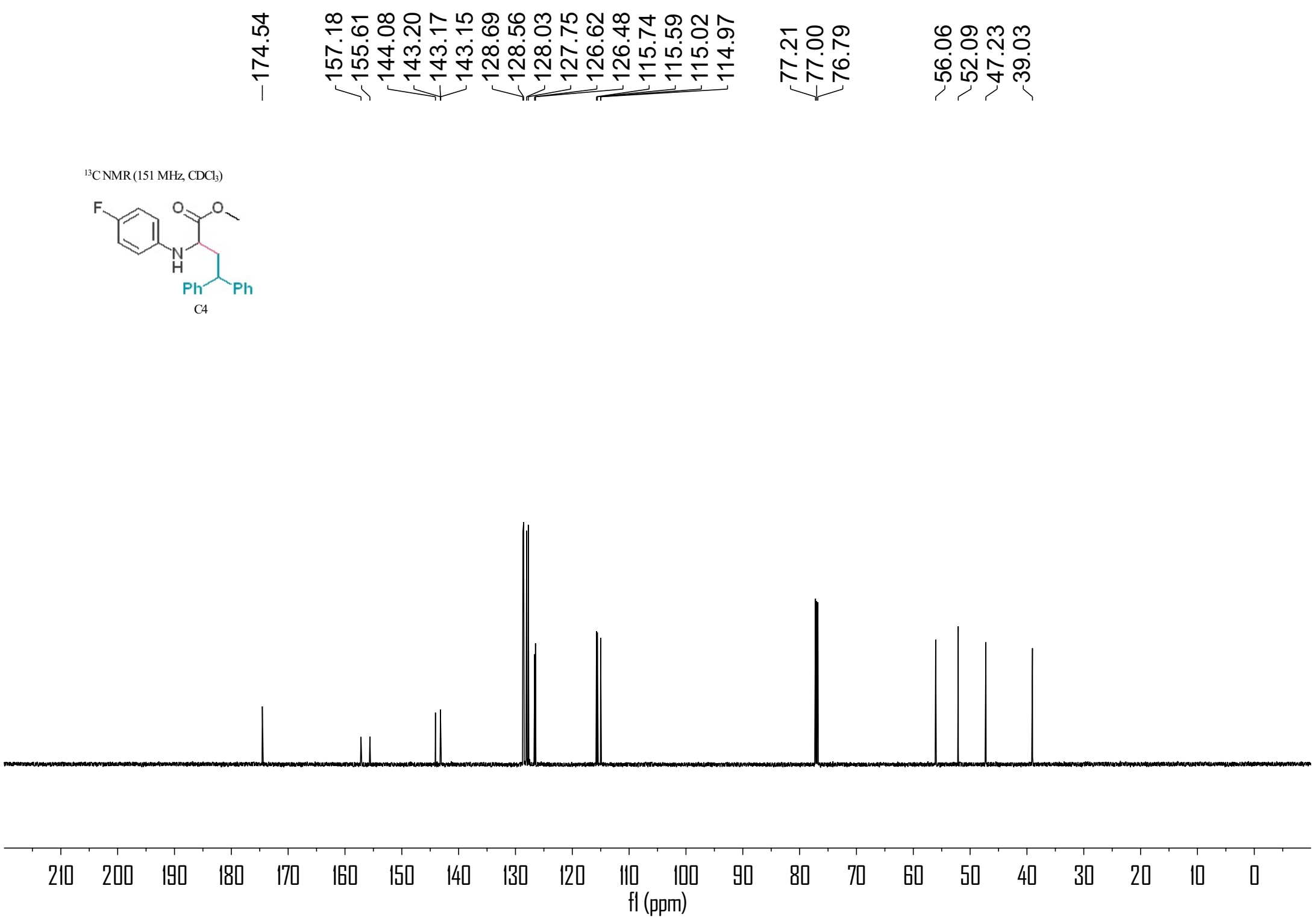

174.72
144.55
144.19
143.35
129.72
128.63
128.52
128.06
127.78
126.53
126.41
114.02
77.21
77.00
76.79
55.55
52.00
47.21
39.12
20.35
13C NMR (151 MHz, CDCl3)
O
O
N
H
Ph
Ph
C1
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

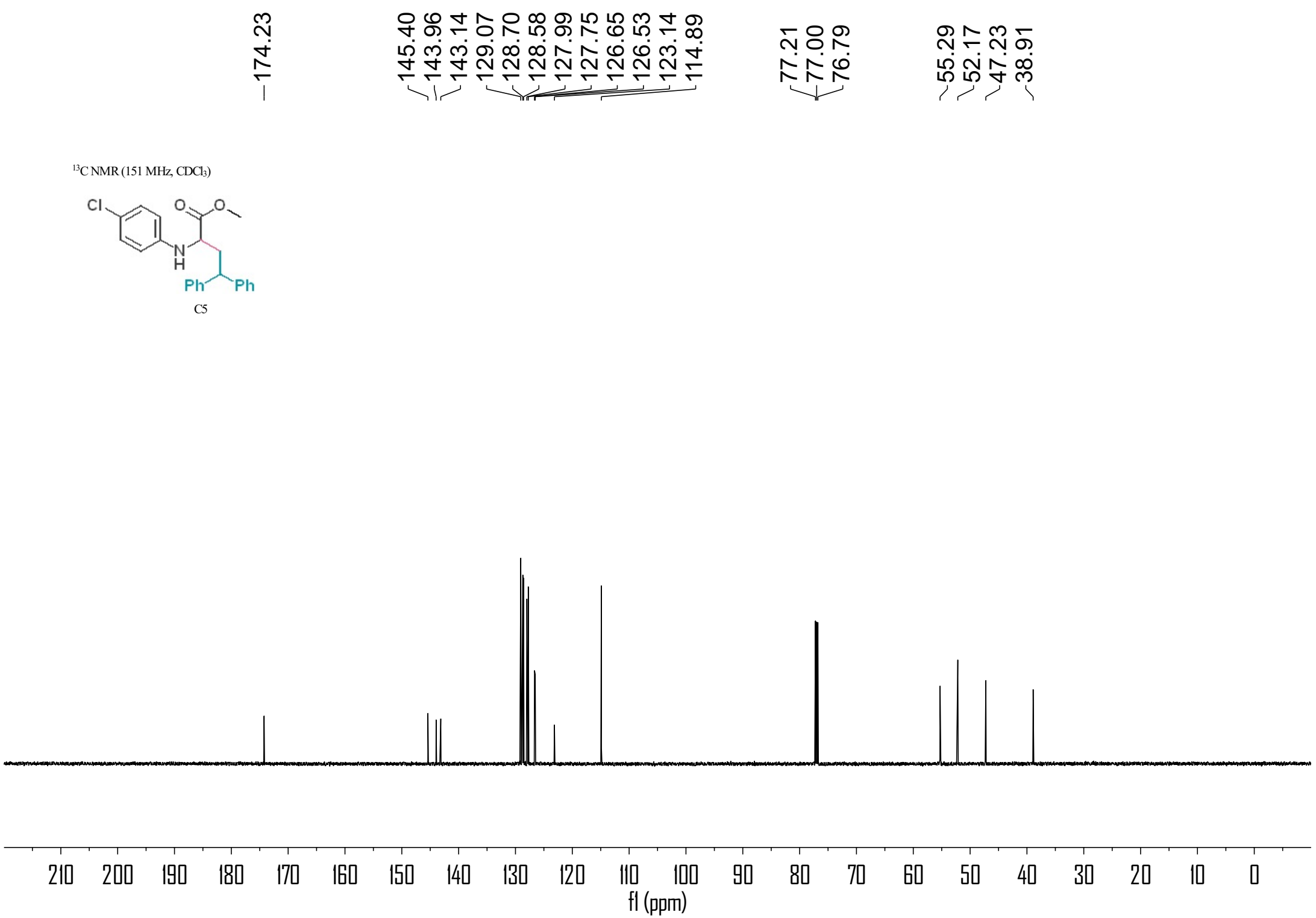


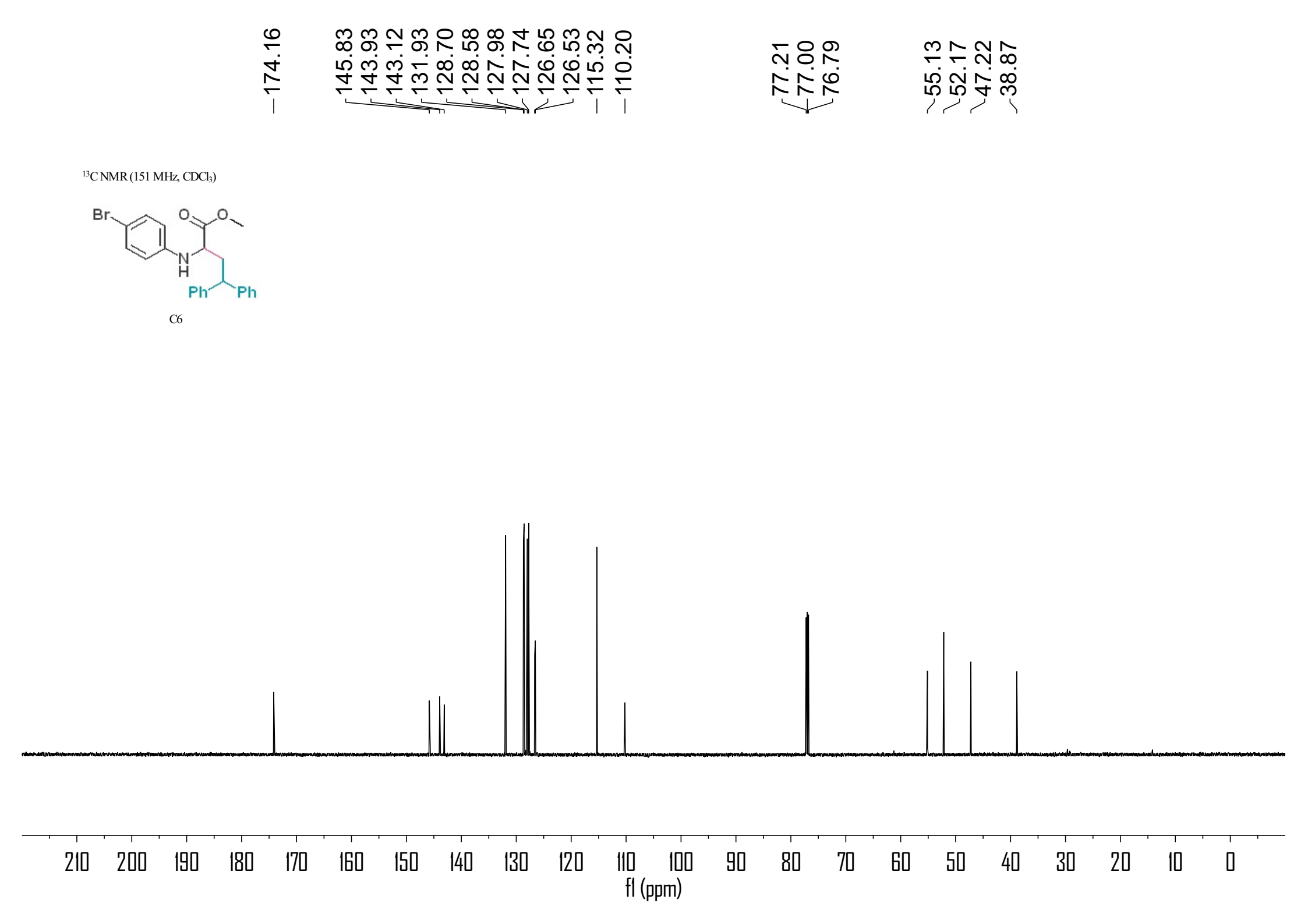
^{13}C NMR (151 MHz, $CDCl_3$)

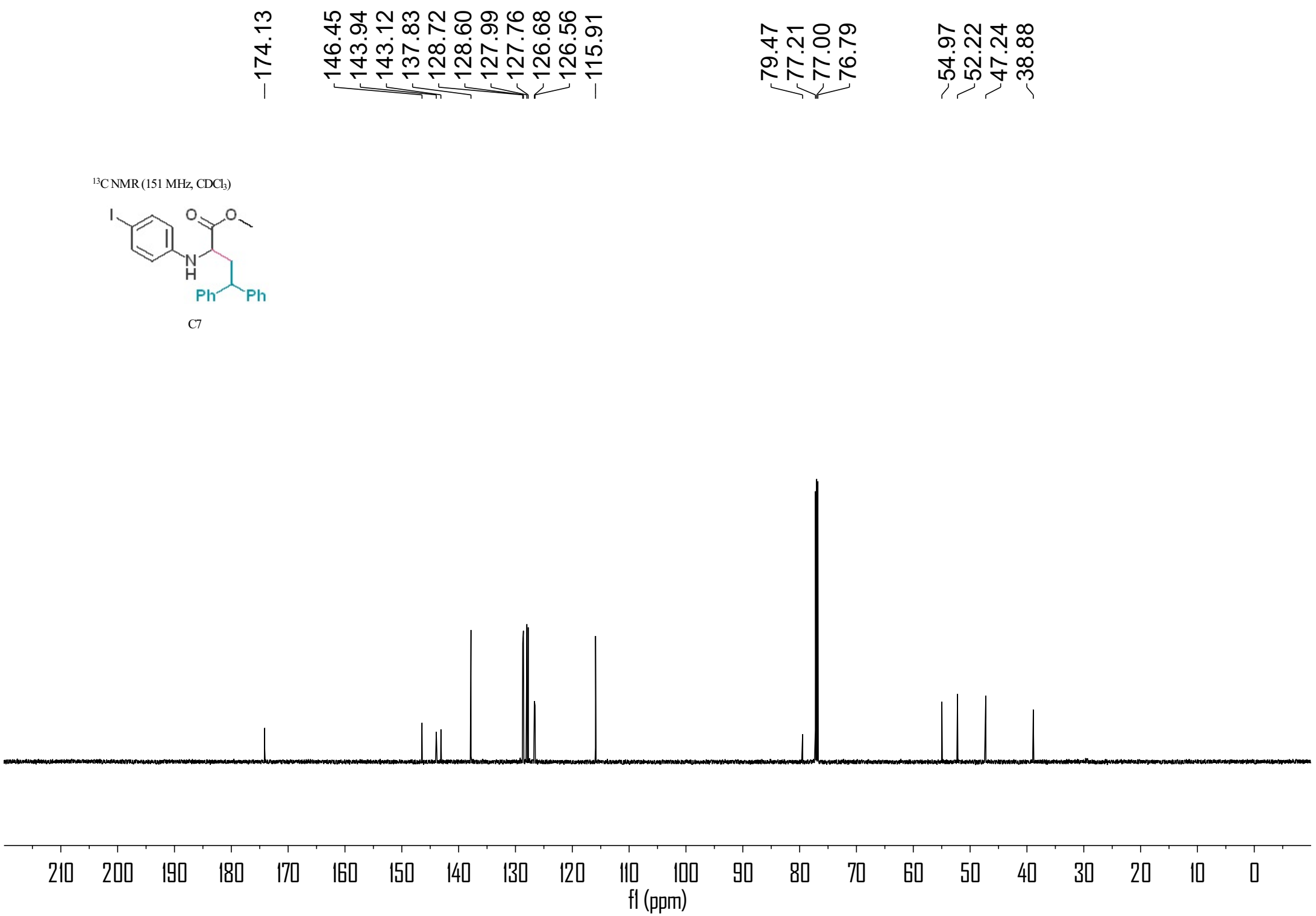
C2

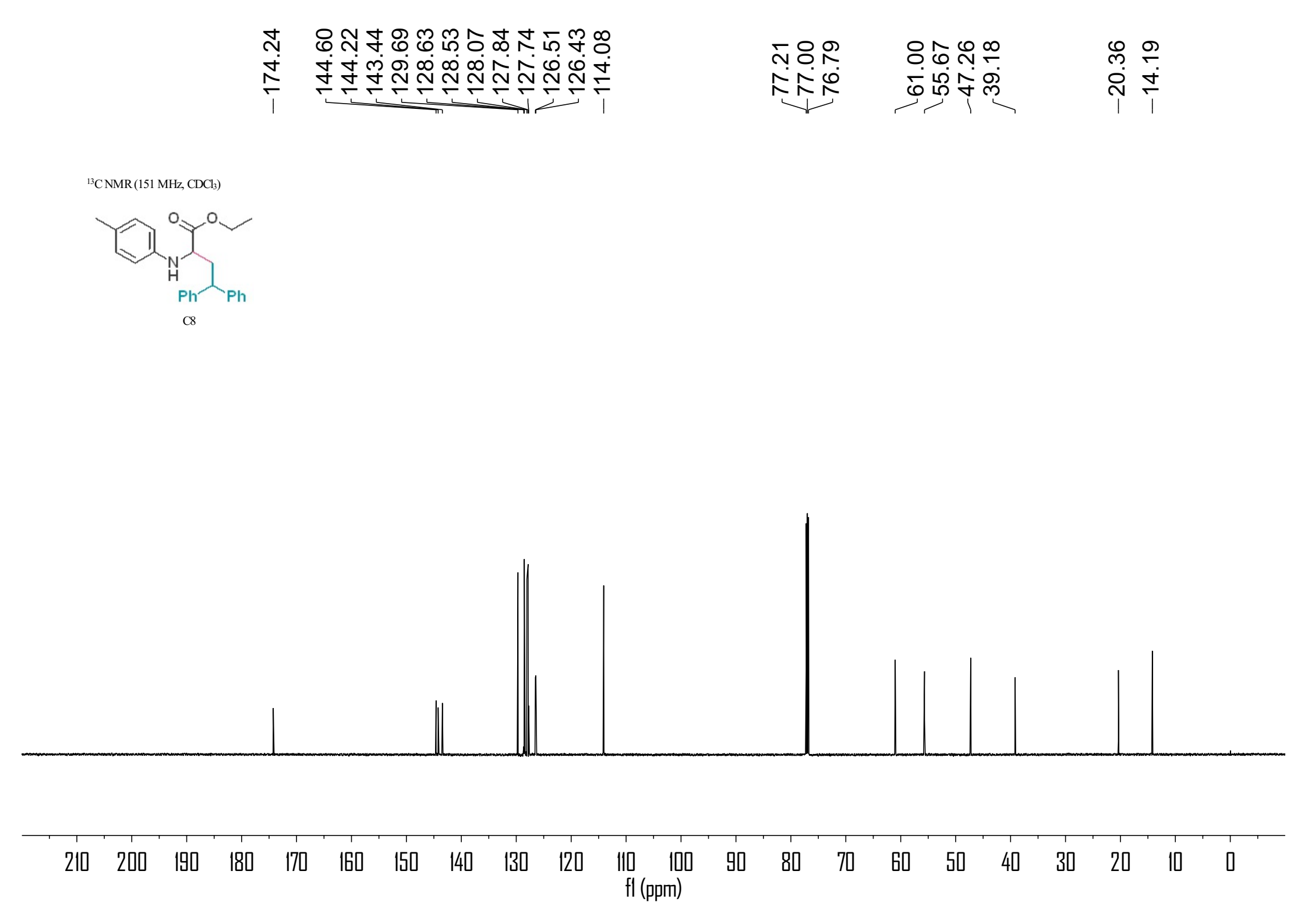

174.40
173.63
144.58
144.06
143.19
143.02
140.62
137.40
129.39
128.66
128.54
128.01
127.74
127.16
126.58
126.45
121.89
114.22
77.21
77.00
76.79
55.60
52.08
47.20
45.15
45.00
39.02
30.13
22.35
18.51
13C NMR (151 MHz, CDCl3)
O
O
O
O
N
H
Ph
Ph
C3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

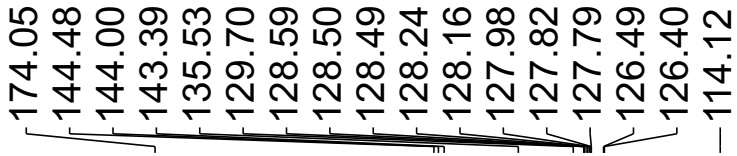

174.54
157.18
155.61
144.08
143.20
143.17
143.15
128.69
128.56
128.03
127.75
126.62
126.48
115.74
115.59
115.02
114.97
77.21
77.00
76.79
56.06
52.09
47.23
39.03
13C NMR (151 MHz, CDCl3)
F
O
O
N
H
Ph
Ph
C4
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

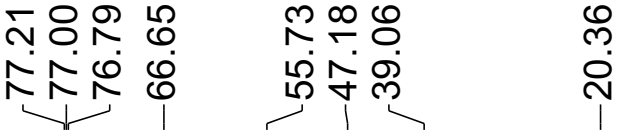

174.23
145.40
143.96
143.14
129.07
128.70
128.58
127.99
127.75
126.65
126.53
123.14
114.89
77.21
77.00
76.79
55.29
52.17
47.23
38.91
13C NMR (151 MHz, CDCl3)
Cl
O
O
N
H
Ph
Ph
C5
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


174.16
145.83
143.93
143.12
131.93
128.70
128.58
127.98
127.74
126.65
126.53
115.32
110.20
77.21
77.00
76.79
55.13
52.17
47.22
38.87
13C NMR (151 MHz, CDCl3)
Br
O
O
N
H
Ph
Ph
C6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

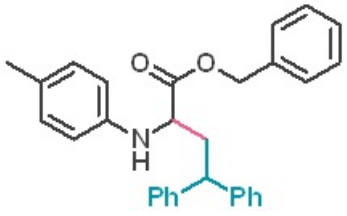

174.13
146.45
143.94
143.12
137.83
128.72
128.60
127.99
127.76
126.68
126.56
115.91
79.47
77.21
77.00
76.79
54.97
52.22
47.24
38.88
13C NMR (151 MHz, CDCl3)
I
O
O
N
H
Ph
Ph
C7
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


174.24
144.60
144.22
143.44
129.69
128.63
128.53
128.07
127.84
127.74
126.51
126.43
114.08
77.21
77.00
76.79
61.00
55.67
47.26
39.18
20.36
14.19
13C NMR (151 MHz, CDCl3)
O
O
N
H
Ph
Ph
C8
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

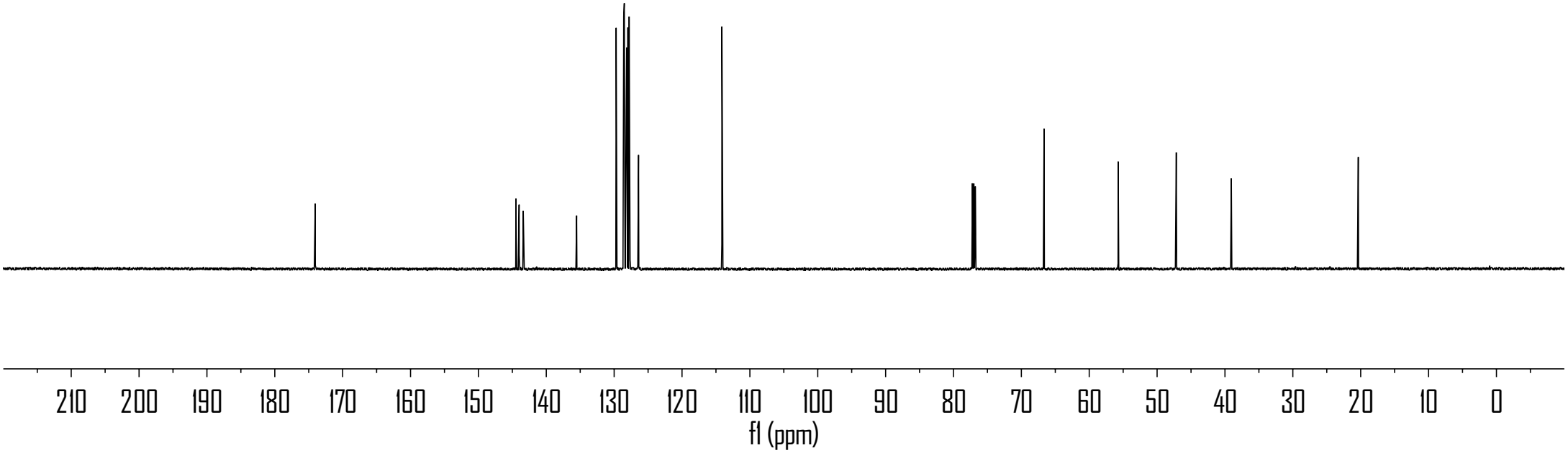


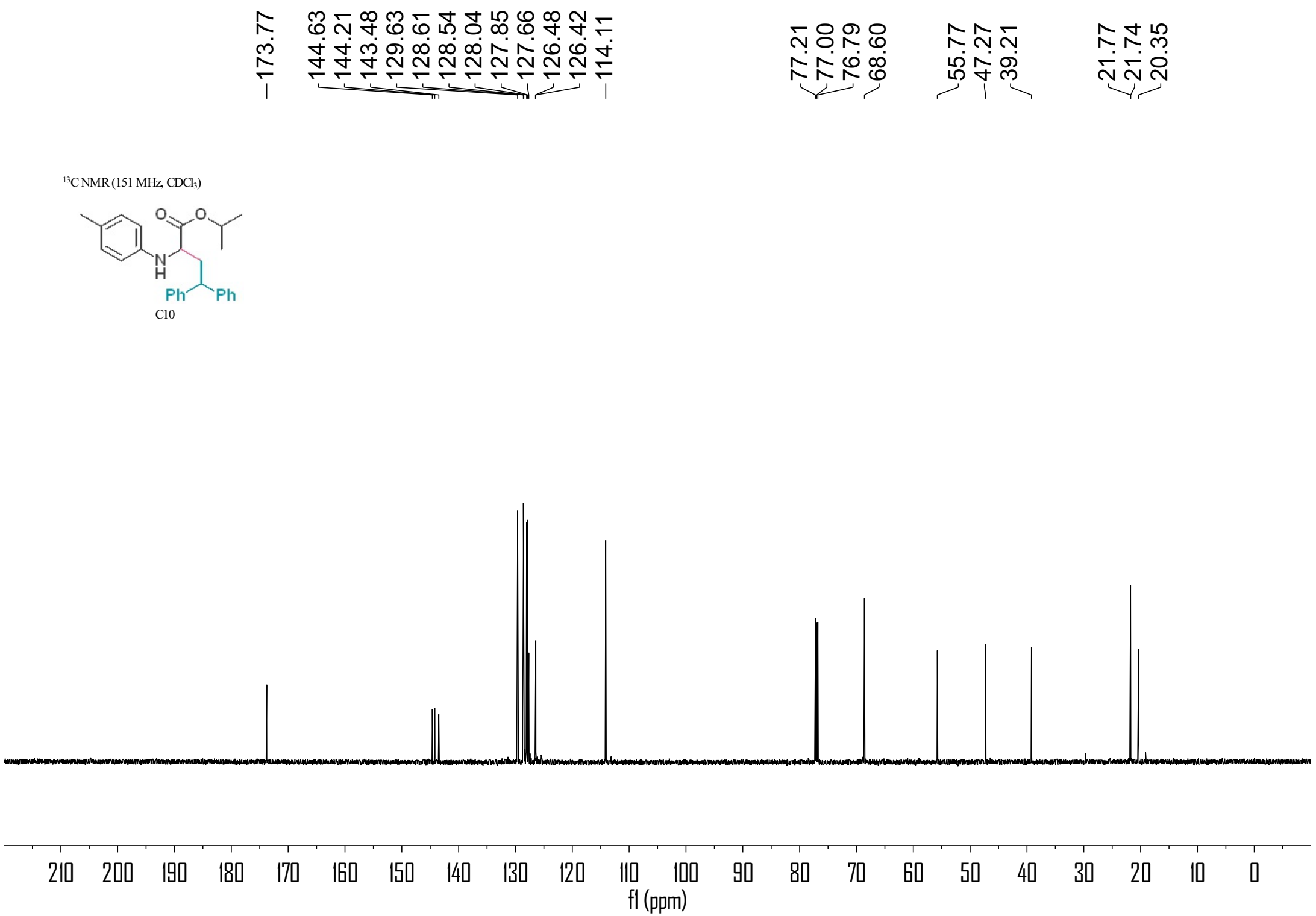


^{13}C NMR (151 MHz, $CDCl_3$)



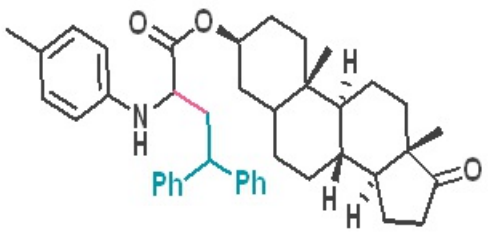
C9



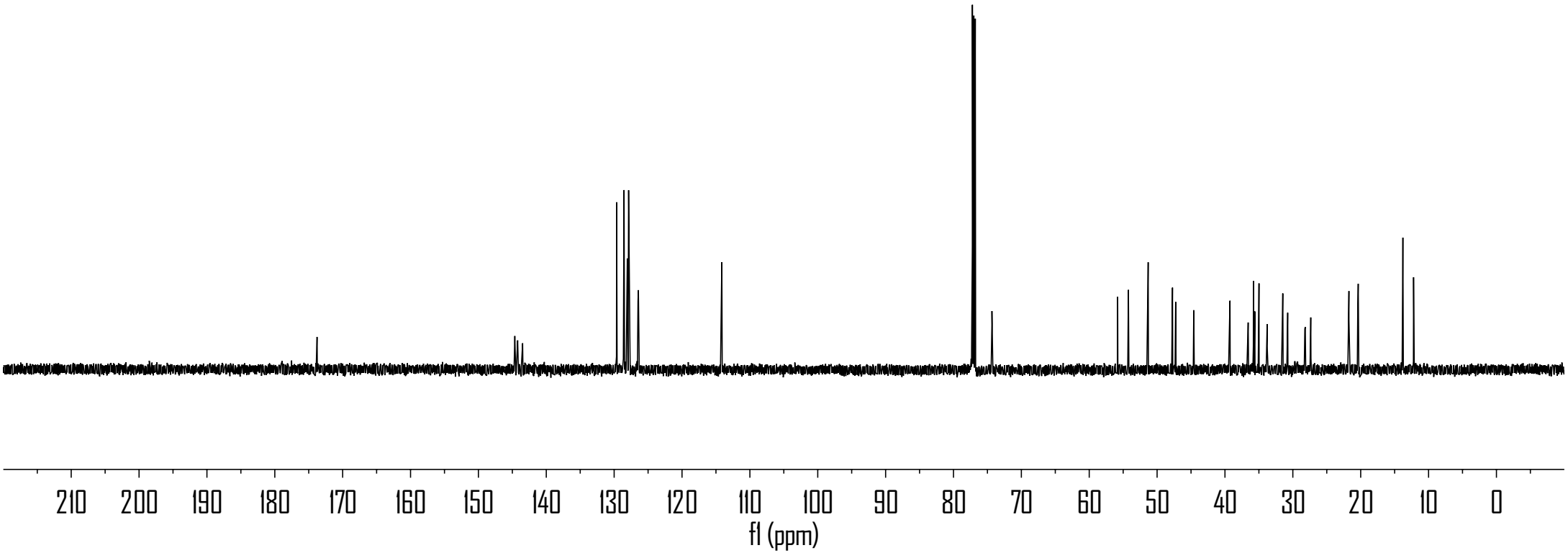

173.77
144.63
144.21
143.48
129.63
128.61
128.54
128.04
127.85
127.66
126.48
126.42
114.11
77.21
77.00
76.79
68.60
55.77
47.27
39.21
21.77
21.74
20.35
13C NMR (151 MHz, CDCl3)
O
O
N
H
Ph
Ph
C10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

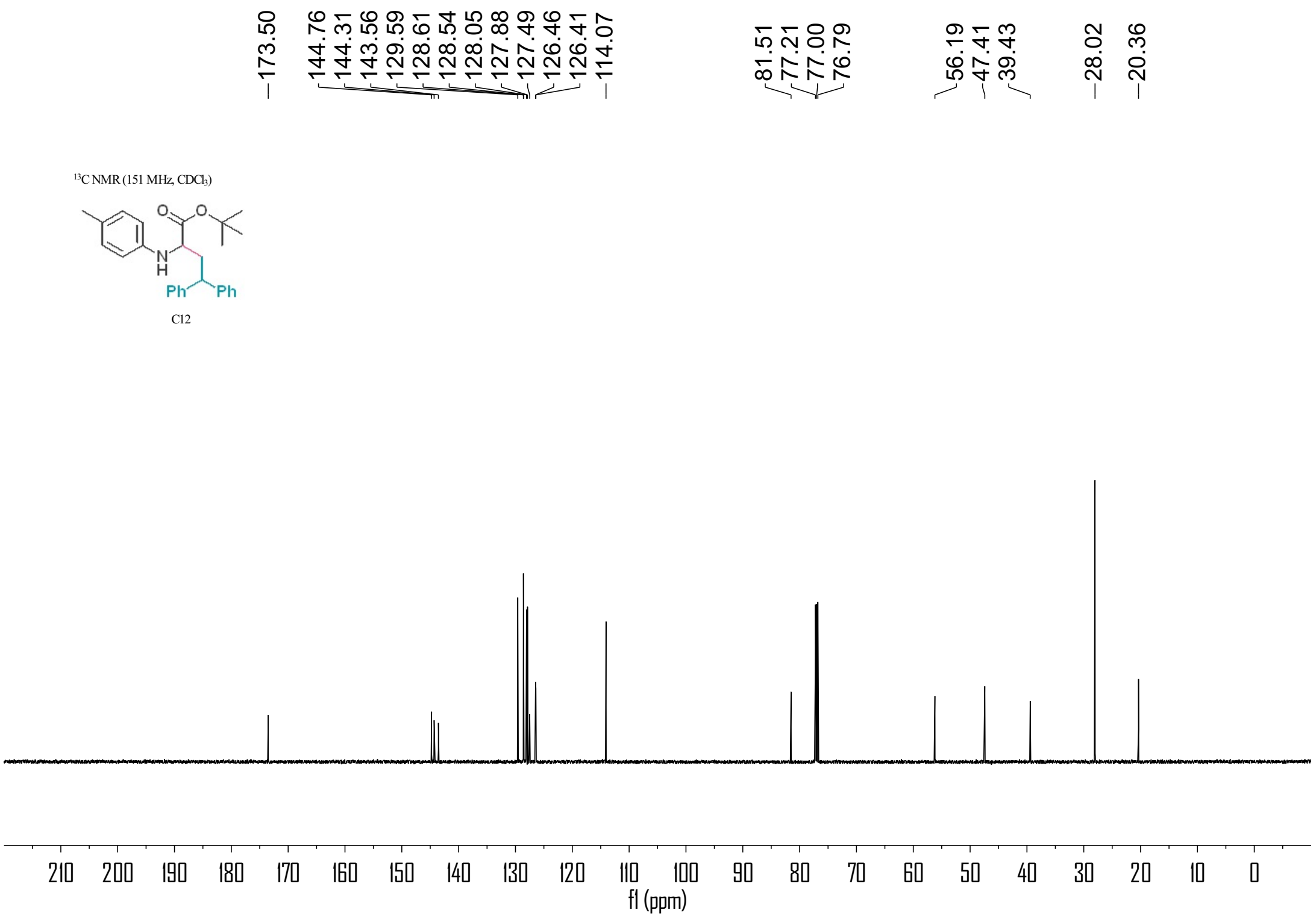
173.78
173.77
144.63
144.61
143.49
143.47
129.63
128.61
128.54
128.06
128.04
127.87
127.73
127.69
127.67
126.48
126.43
114.13
114.12
77.21
77.00
76.79
74.33
55.81
54.23
51.32
47.73
47.26
44.60
44.58
39.26
36.60
36.58
35.81
35.60
34.99
33.82
33.78
31.48
30.76
30.74
28.23
28.17
27.36
27.34
21.74
20.43
20.36
13.78
12.21

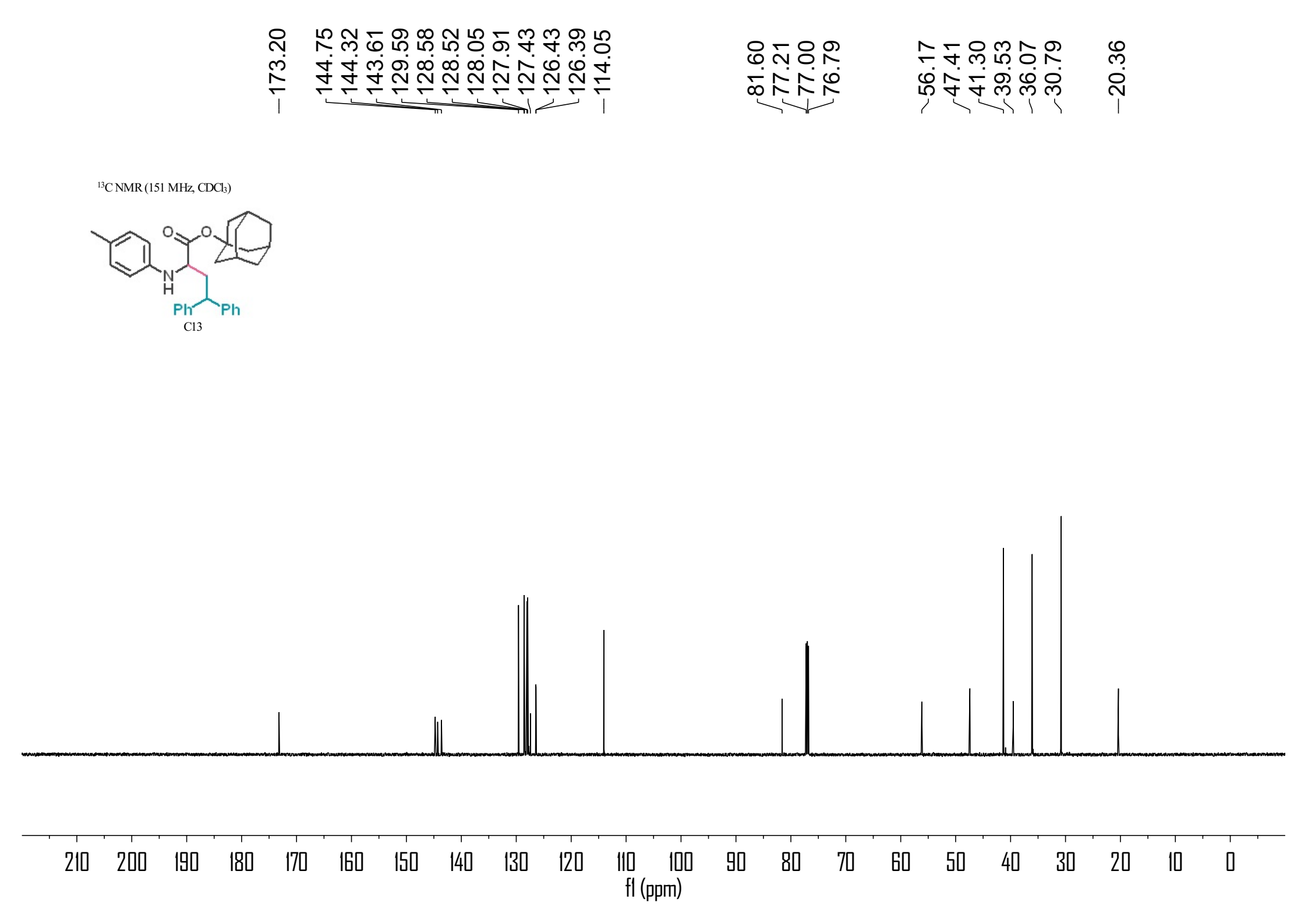
^{13}C NMR (151 MHz, $CDCl_3$)

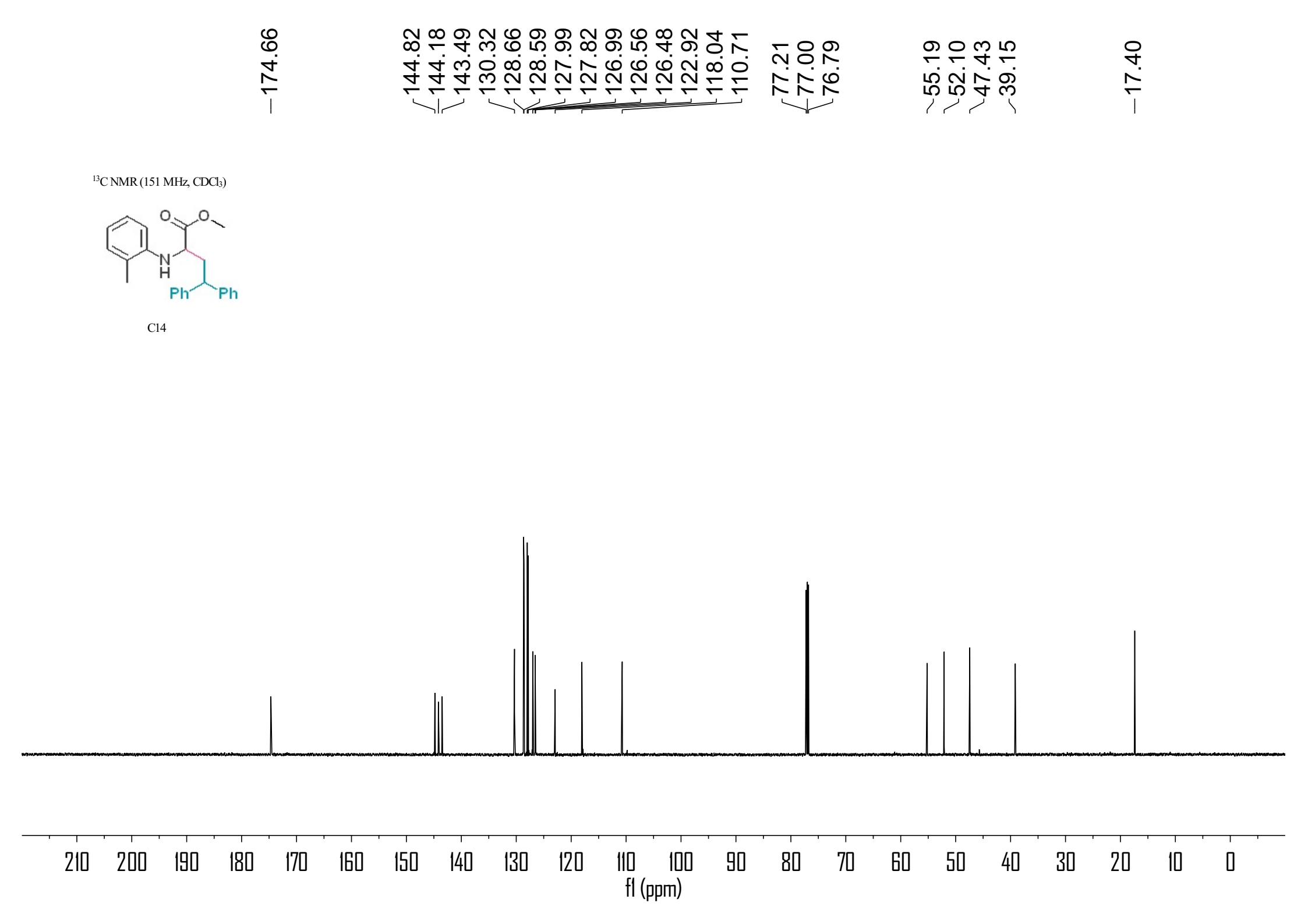


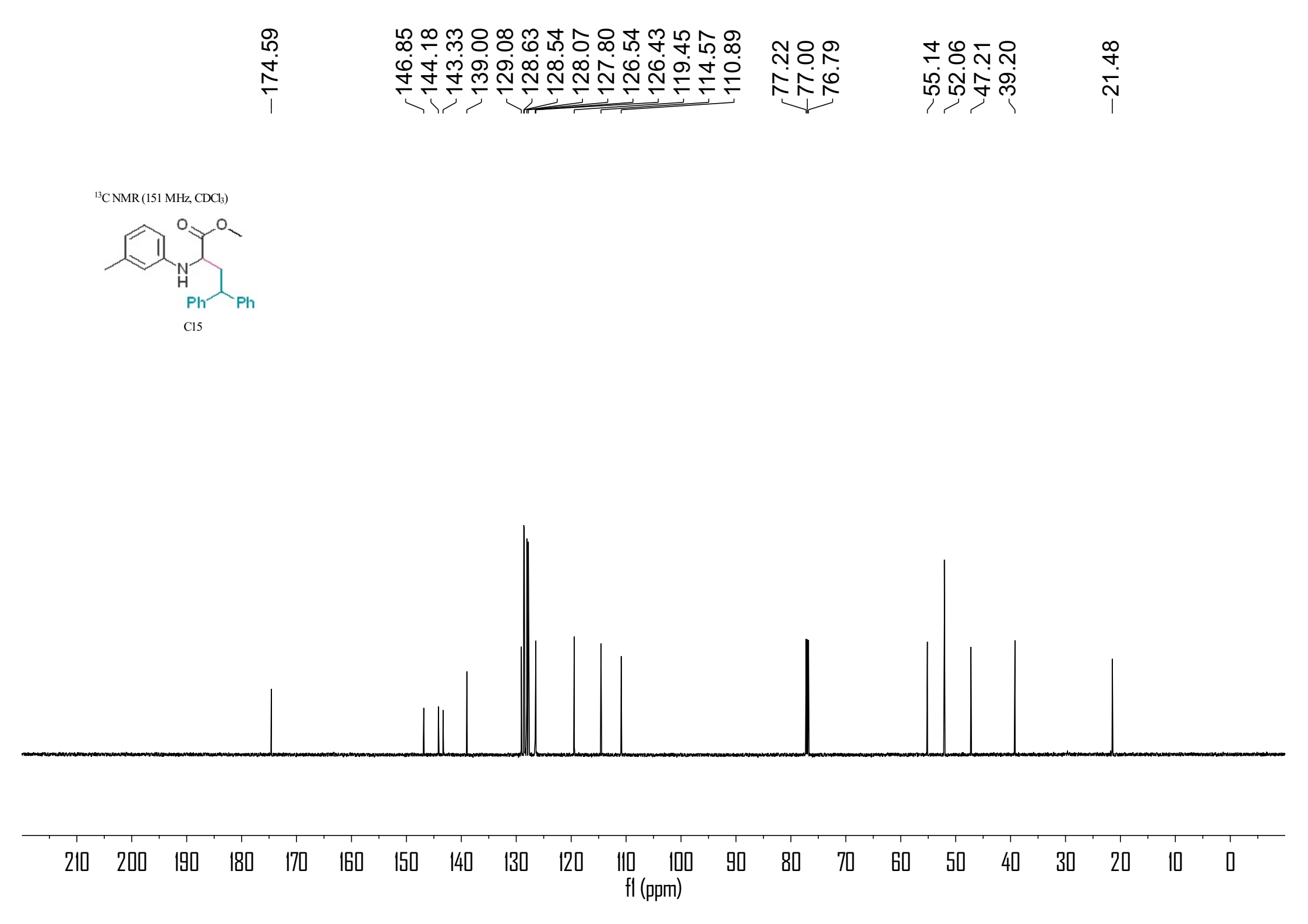
C11

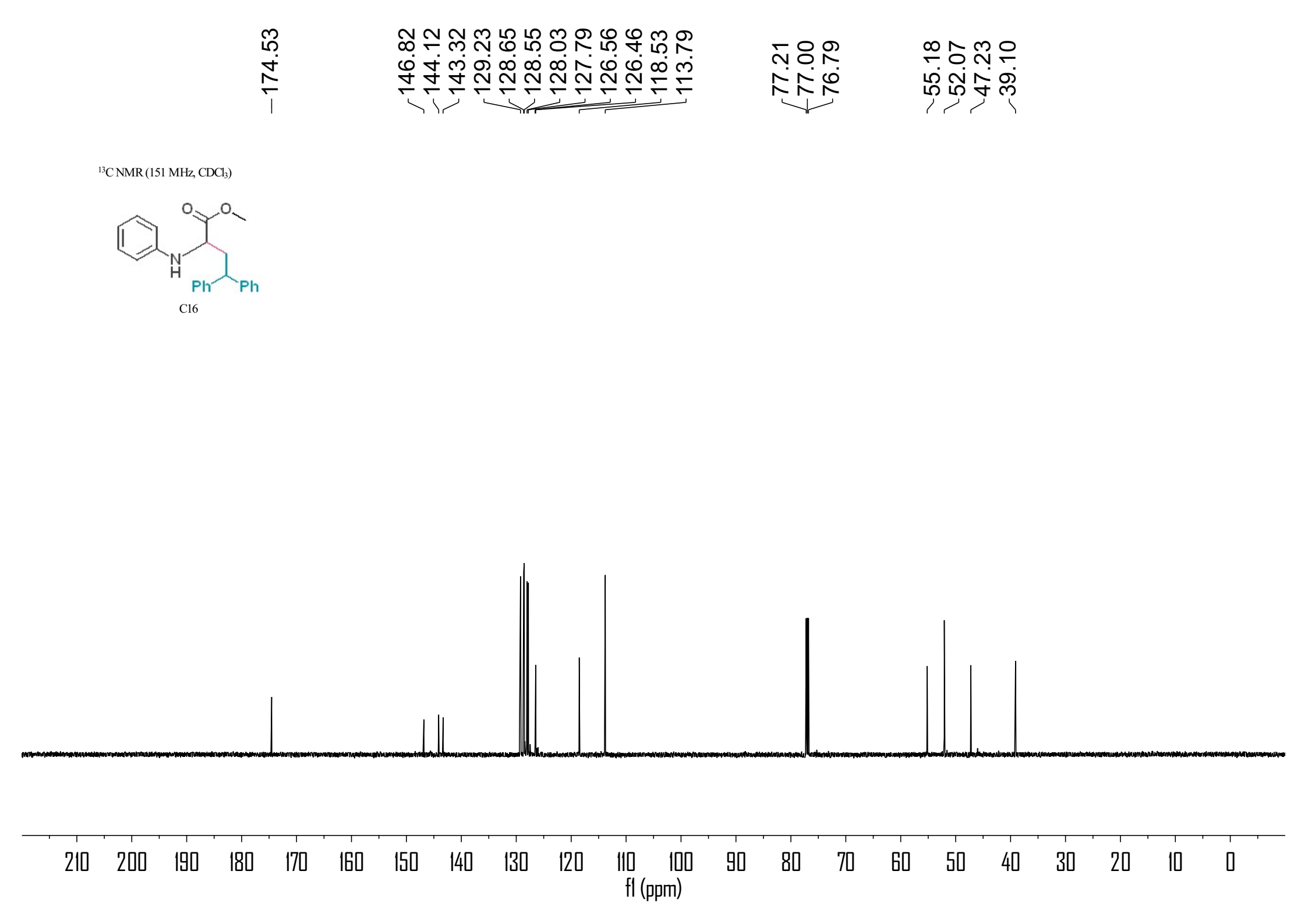



173.50
144.76
144.31
143.56
129.59
128.61
128.54
128.05
127.88
127.49
126.46
126.41
114.07
81.51
77.21
77.00
76.79
56.19
47.41
39.43
28.02
20.36
13C NMR (151 MHz, CDCl3)
O
O
N
H
Ph
Ph
C12
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


173.20
144.75
144.32
143.61
129.59
128.58
128.52
128.05
127.91
127.43
126.43
126.39
114.05
81.60
77.21
77.00
76.79
56.17
47.41
41.30
39.53
36.07
30.79
20.36
13C NMR (151 MHz, CDCl3)
O
O
N
H
Ph
Ph
C13
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


174.66
144.82
144.18
143.49
130.32
128.66
128.59
127.99
127.82
126.99
126.56
126.48
122.92
118.04
110.71
77.21
77.00
76.79
55.19
52.10
47.43
39.15
17.40
13C NMR (151 MHz, CDCl3)
O
O
N
H
Ph
Ph
C14
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

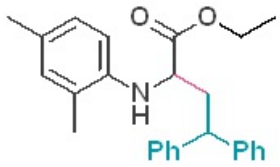

174.59
146.85
144.18
143.33
139.00
129.08
128.63
128.54
128.07
127.80
126.54
126.43
119.45
114.57
110.89
77.22
77.00
76.79
55.14
52.06
47.21
39.20
21.48
13C NMR (151 MHz, CDCl3)
O
O
N
H
Ph
Ph
C15
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



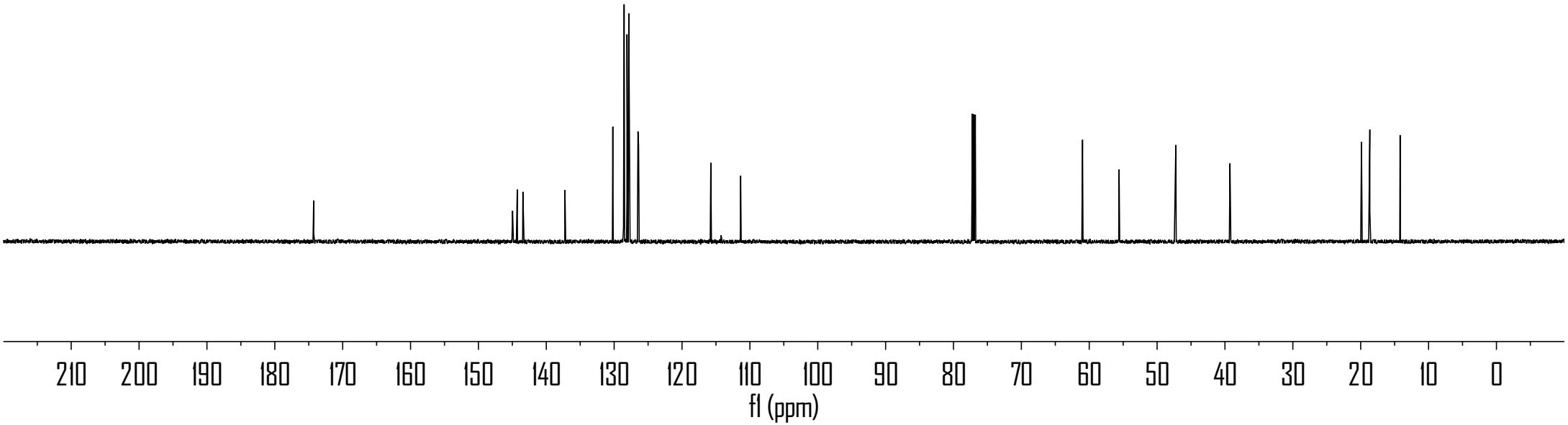
^{13}C NMR (151 MHz, $CDCl_3$)

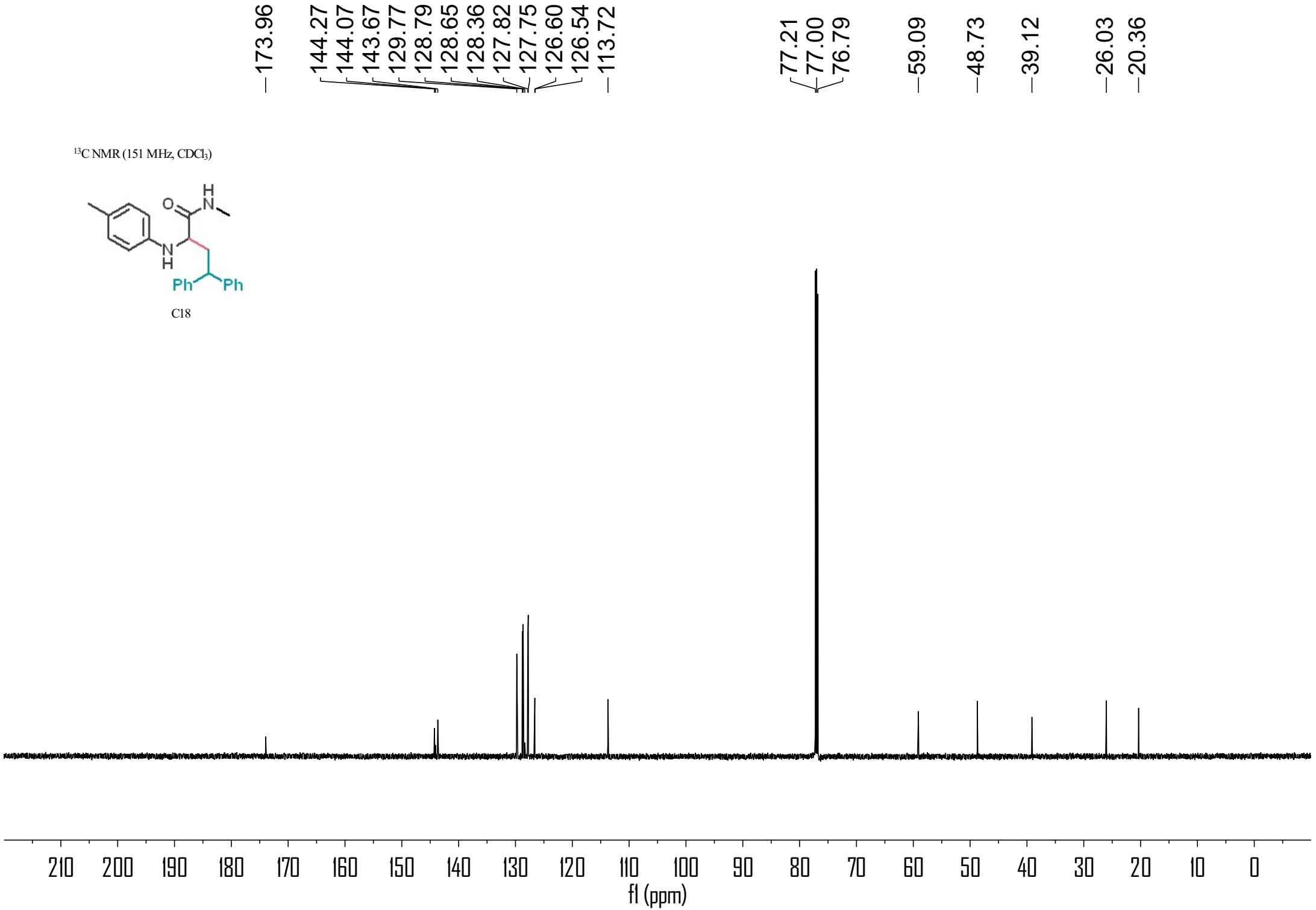
C16

^{13}C NMR (151 MHz, $CDCl_3$)



C17




173.96
144.27
144.07
143.67
129.77
128.79
128.65
128.36
127.82
127.75
126.60
126.54
113.72
77.21
77.00
76.79
59.09
48.73
39.12
26.03
20.36
13C NMR (151 MHz, CDCl3)
O
H
N
N
H
Ph
Ph
C18
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

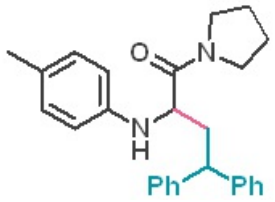
171.76
145.20
144.19
144.15
129.71
128.48
128.45
128.16
127.69
127.58
126.47
126.30
114.45

77.21
77.00
76.79

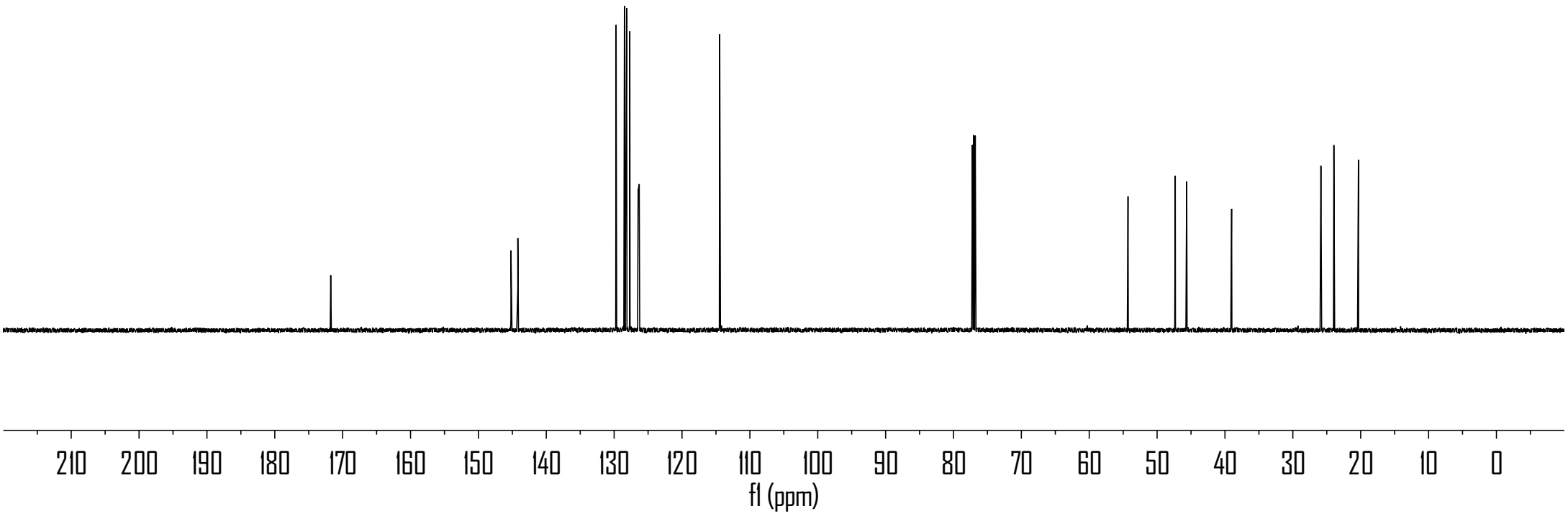
54.29
47.32
45.70
45.64
39.02

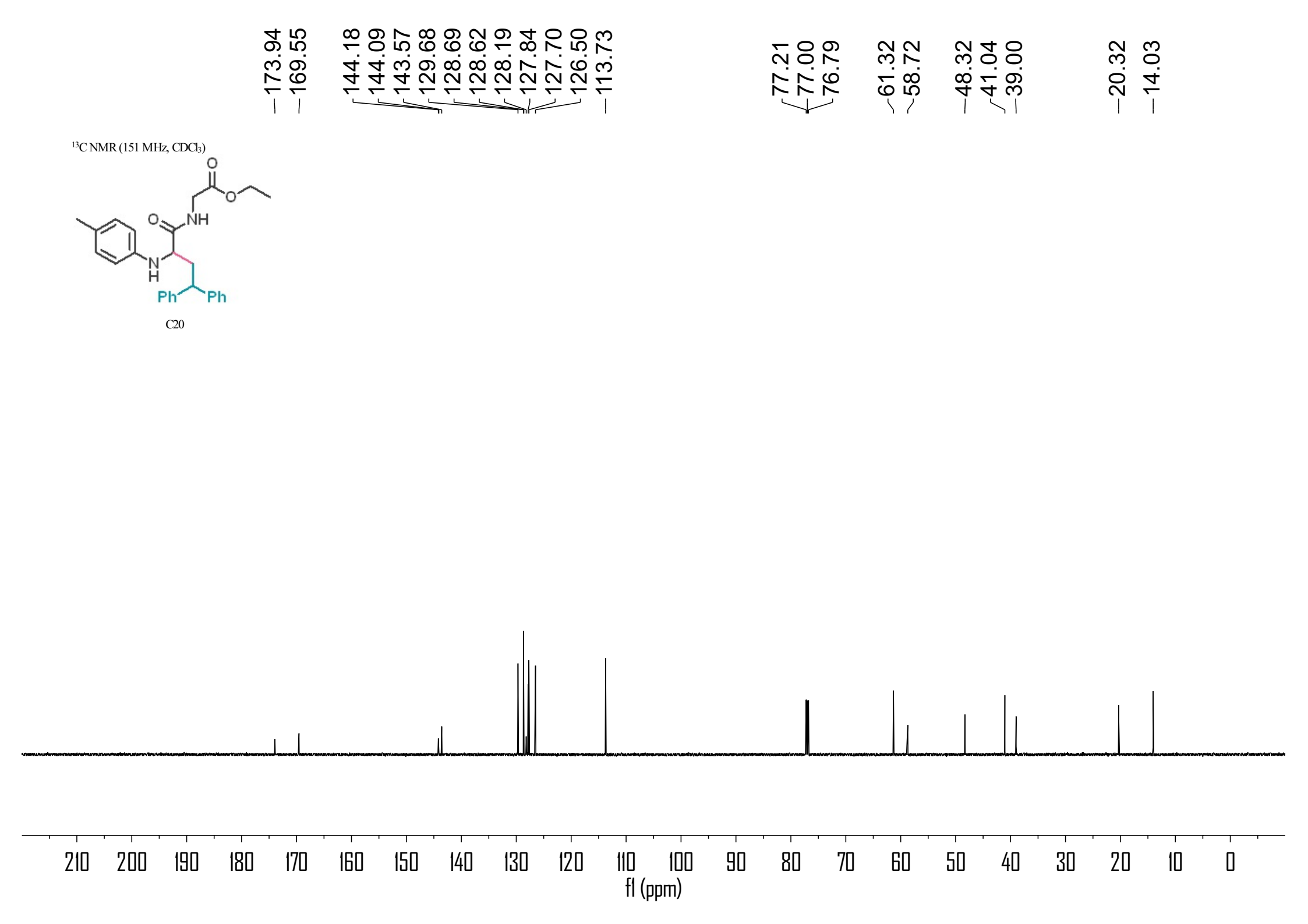
25.87
23.96
20.33

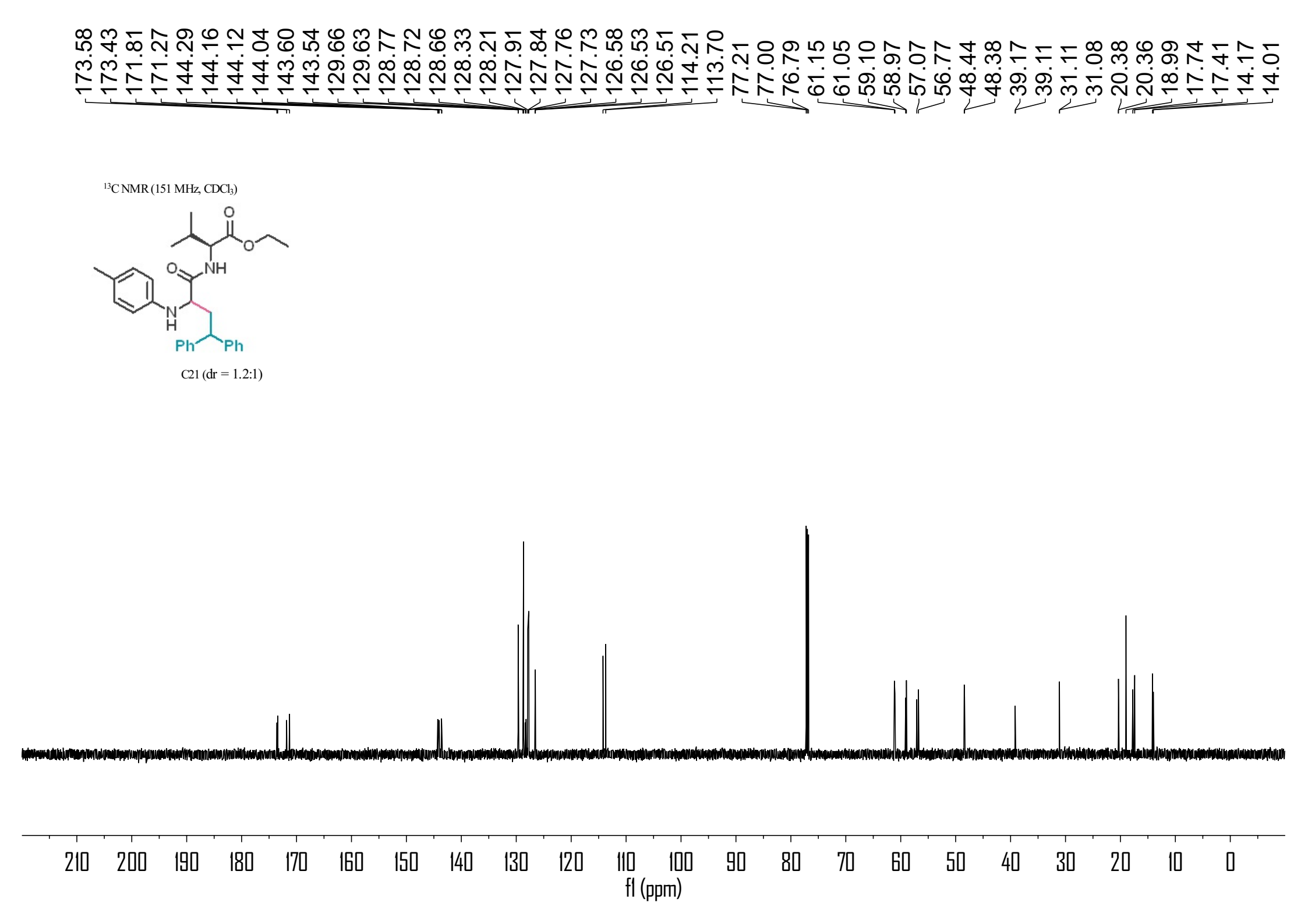
^{13}C NMR (151 MHz, $CDCl_3$)

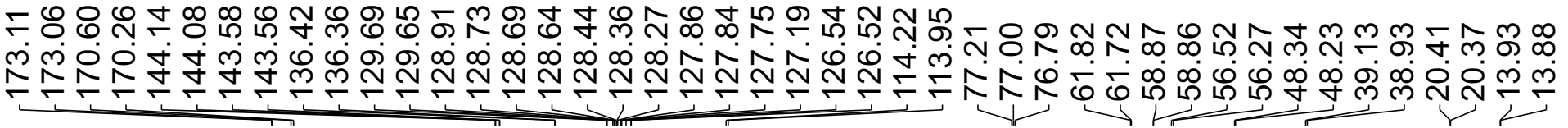


C19

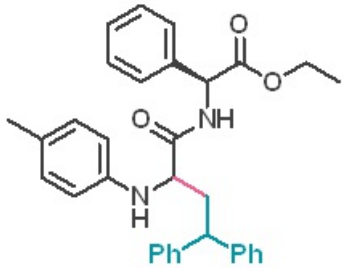



173.94
169.55
144.18
144.09
143.57
129.68
128.69
128.62
128.19
127.84
127.70
126.50
113.73
77.21
77.00
76.79
61.32
58.72
48.32
41.04
39.00
20.32
14.03
13C NMR (151 MHz, CDCl3)
O
O
NH
O
N
H
Ph
Ph
C20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

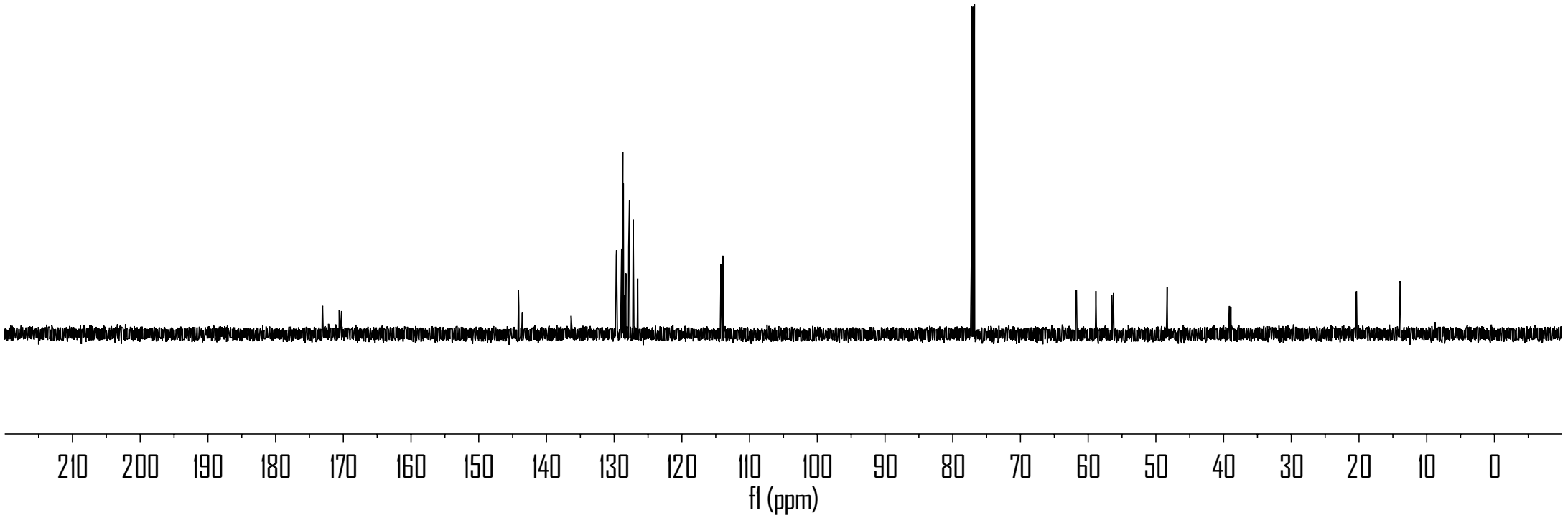

173.58
173.43
171.81
171.27
144.29
144.16
144.12
144.04
143.60
143.54
129.66
129.63
128.77
128.72
128.66
128.33
128.21
127.91
127.84
127.76
127.73
126.58
126.53
126.51
114.21
113.70
77.21
77.00
76.79
61.15
61.05
59.10
58.97
57.07
56.77
48.44
48.38
39.17
39.11
31.11
31.08
20.38
20.36
18.99
17.74
17.41
14.17
14.01
13C NMR (151 MHz, CDCl3)
O
O
NH
O
N
H
Ph
Ph
C21 (dr = 1.2:1)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

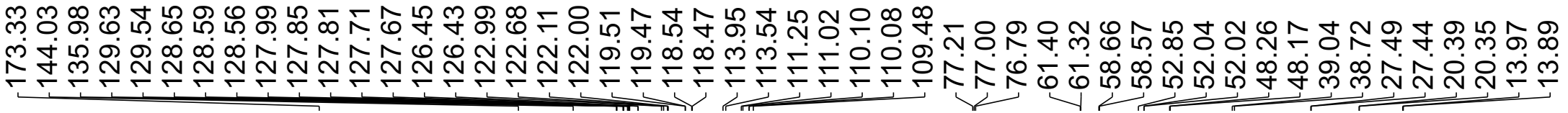


^{13}C NMR (151 MHz, $CDCl_3$)

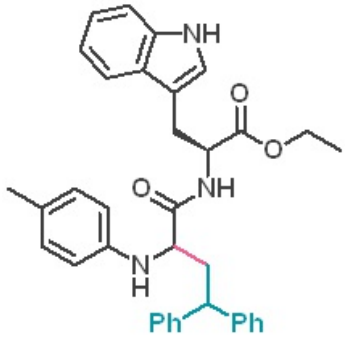


C22 (dr = 1:1)

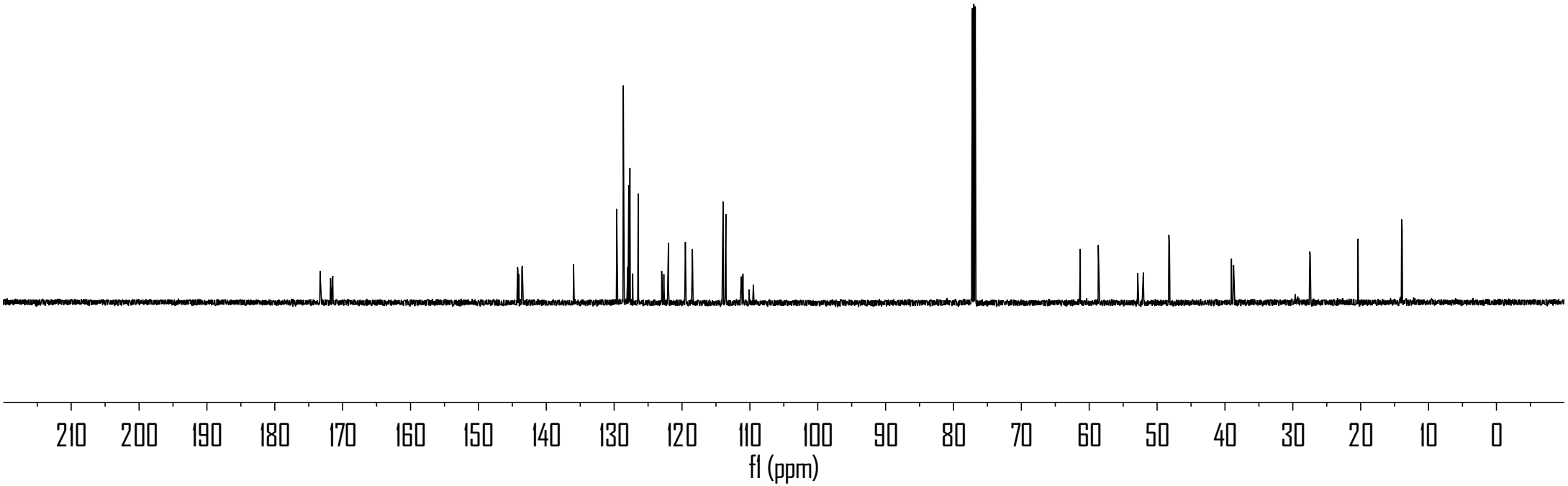


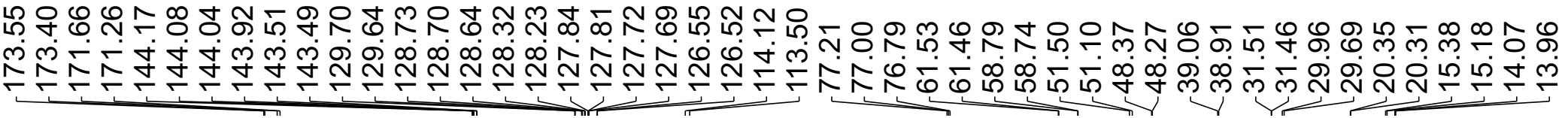


^{13}C NMR (151 MHz, $CDCl_3$)

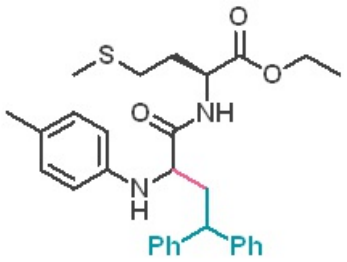


C23 (dr = 1.1:1)

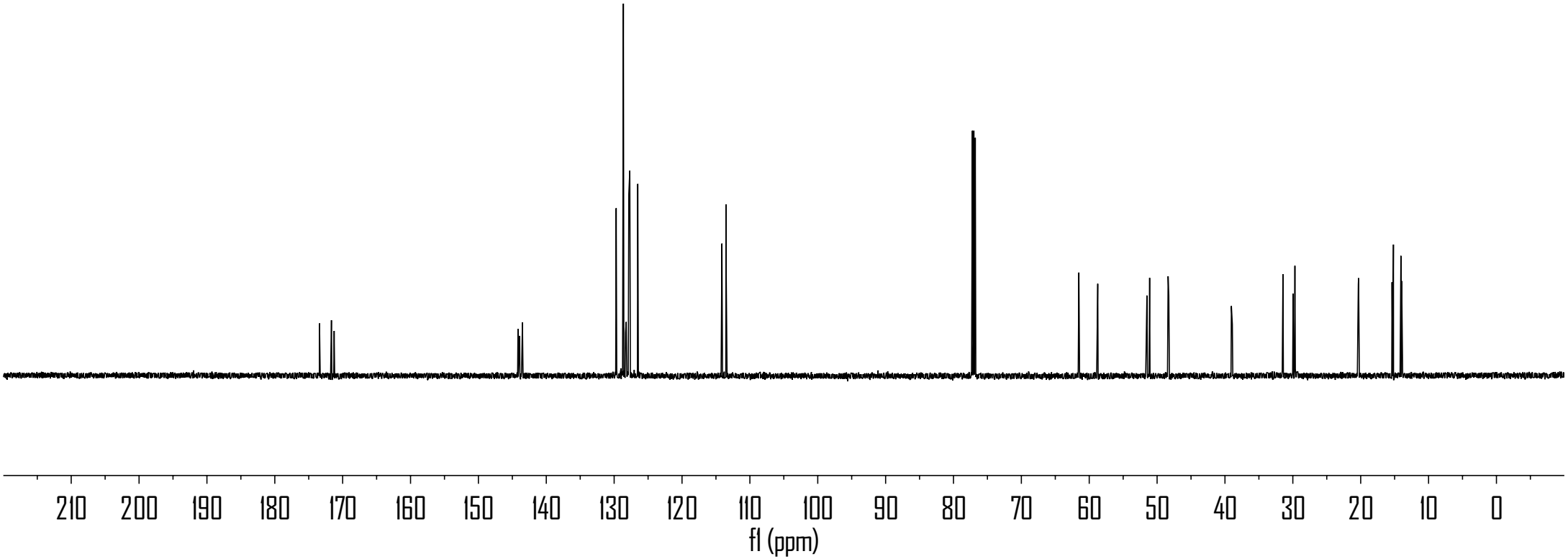




^{13}C NMR (151 MHz, $CDCl_3$)

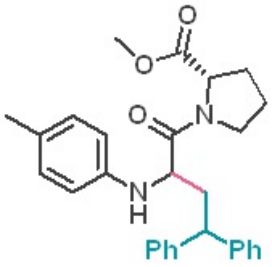


C24 (dr = 1.3:1)

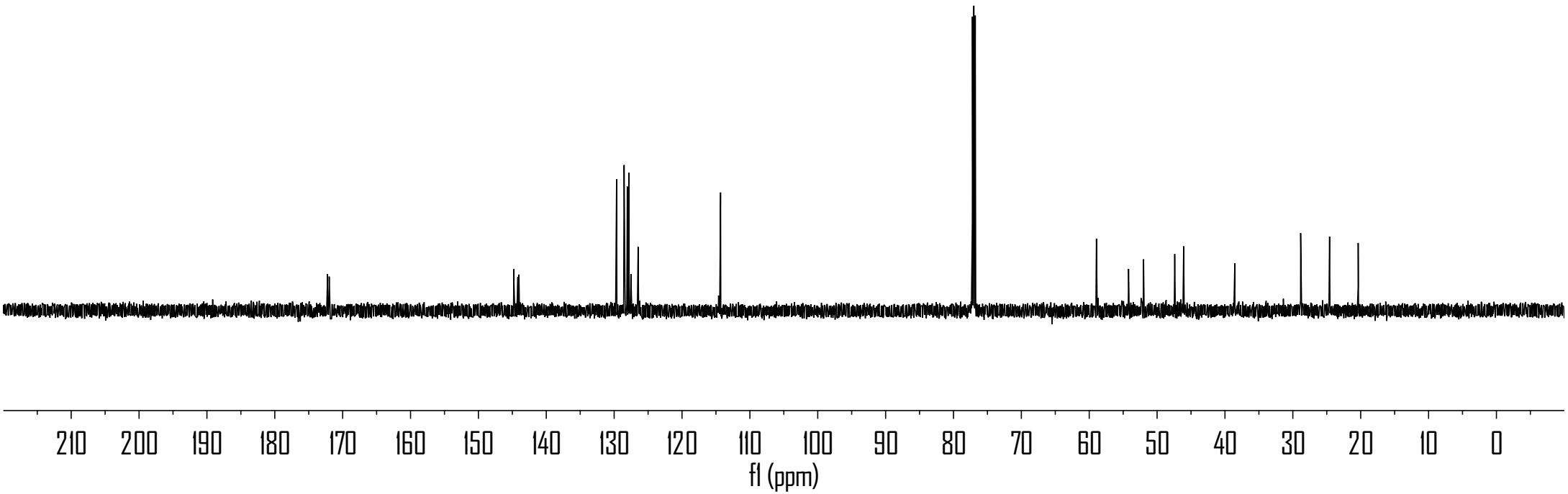


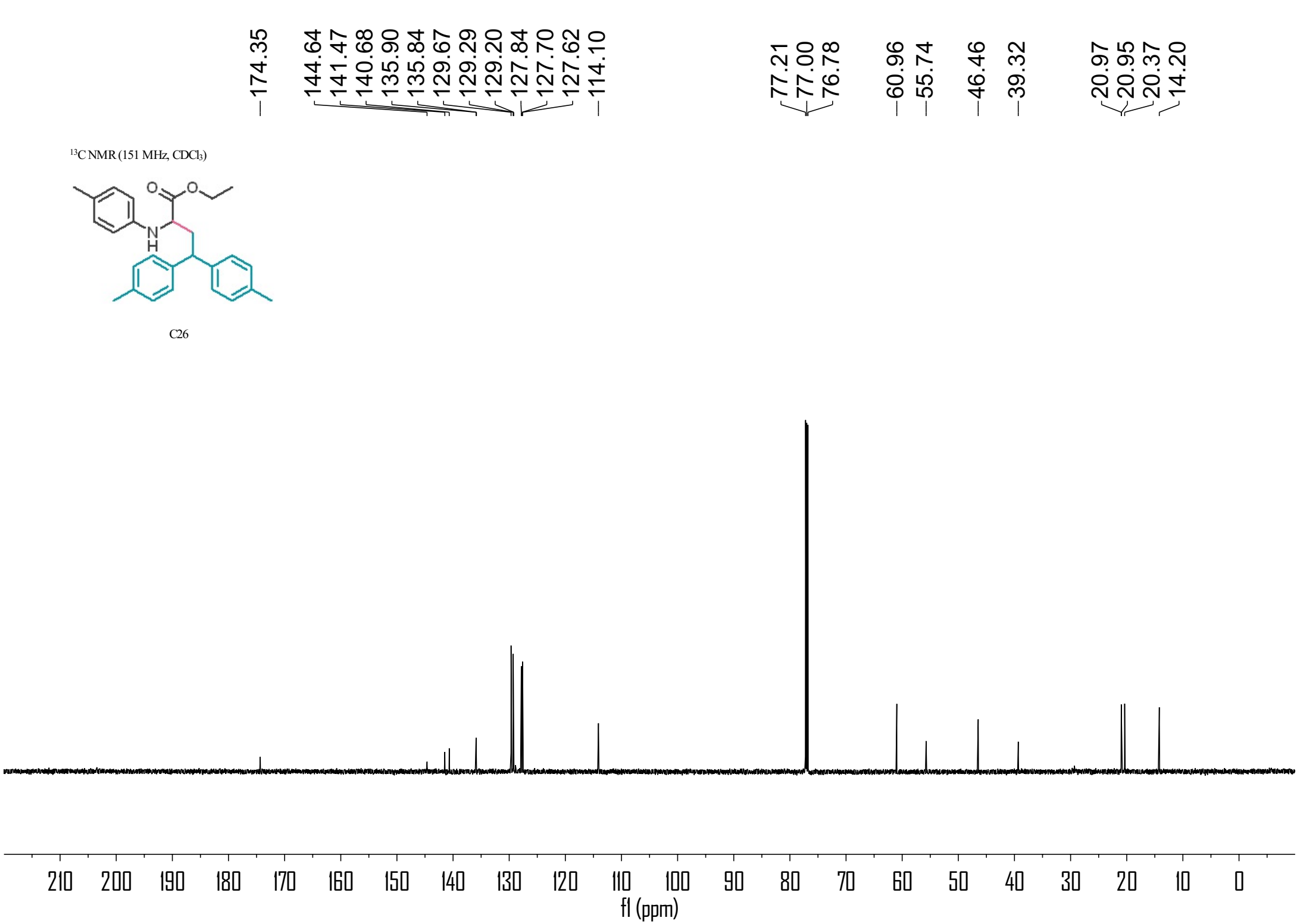
172.25
171.98
144.76
144.19
144.03
129.76
129.64
128.53
128.49
128.06
127.80
127.53
126.47
126.45
114.32
77.21
77.00
76.79
58.94
54.20
52.00
47.40
46.09
38.55
28.85
24.59
20.37

^{13}C NMR (151 MHz, $CDCl_3$)



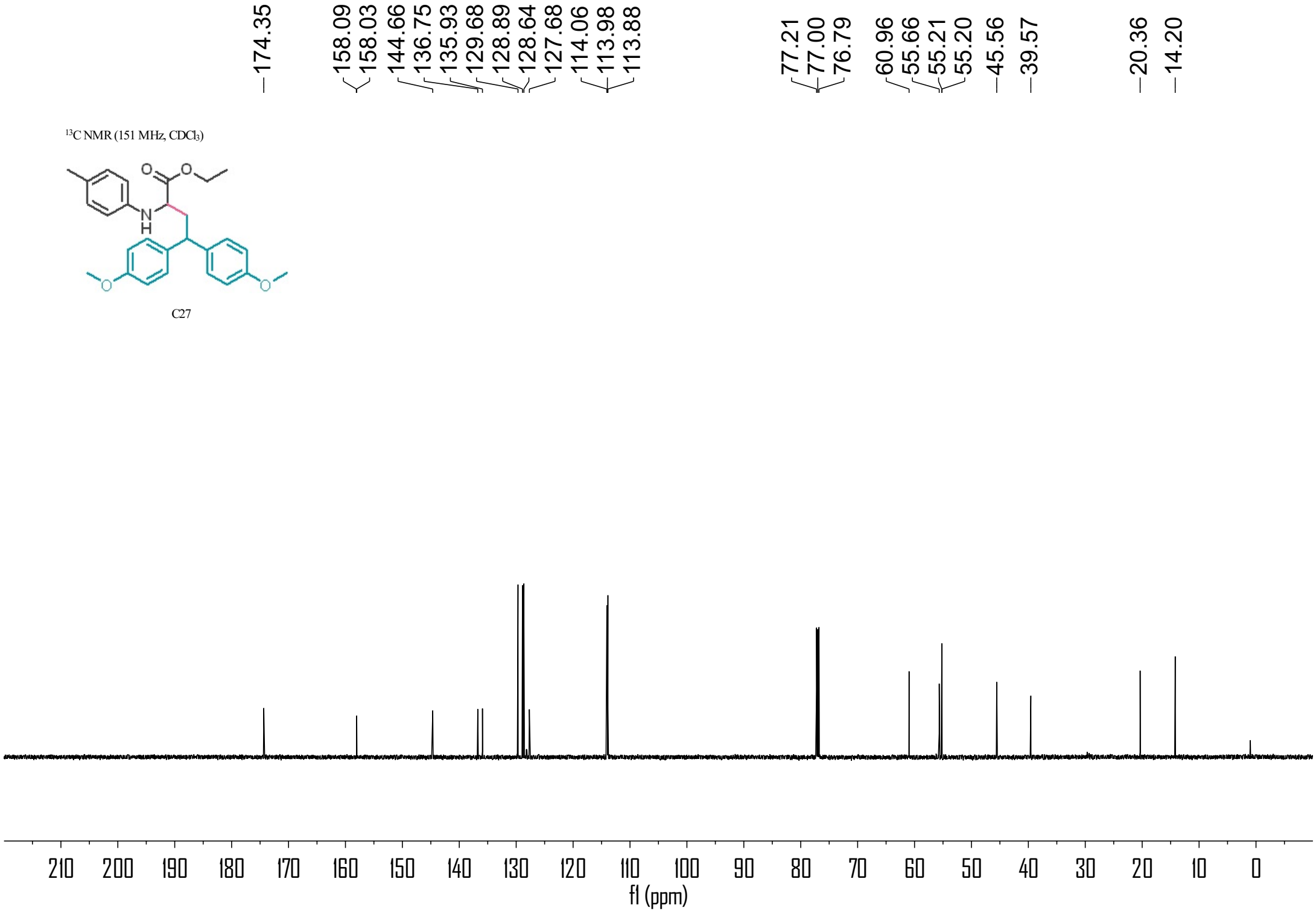
C25 (dr = 6:1)

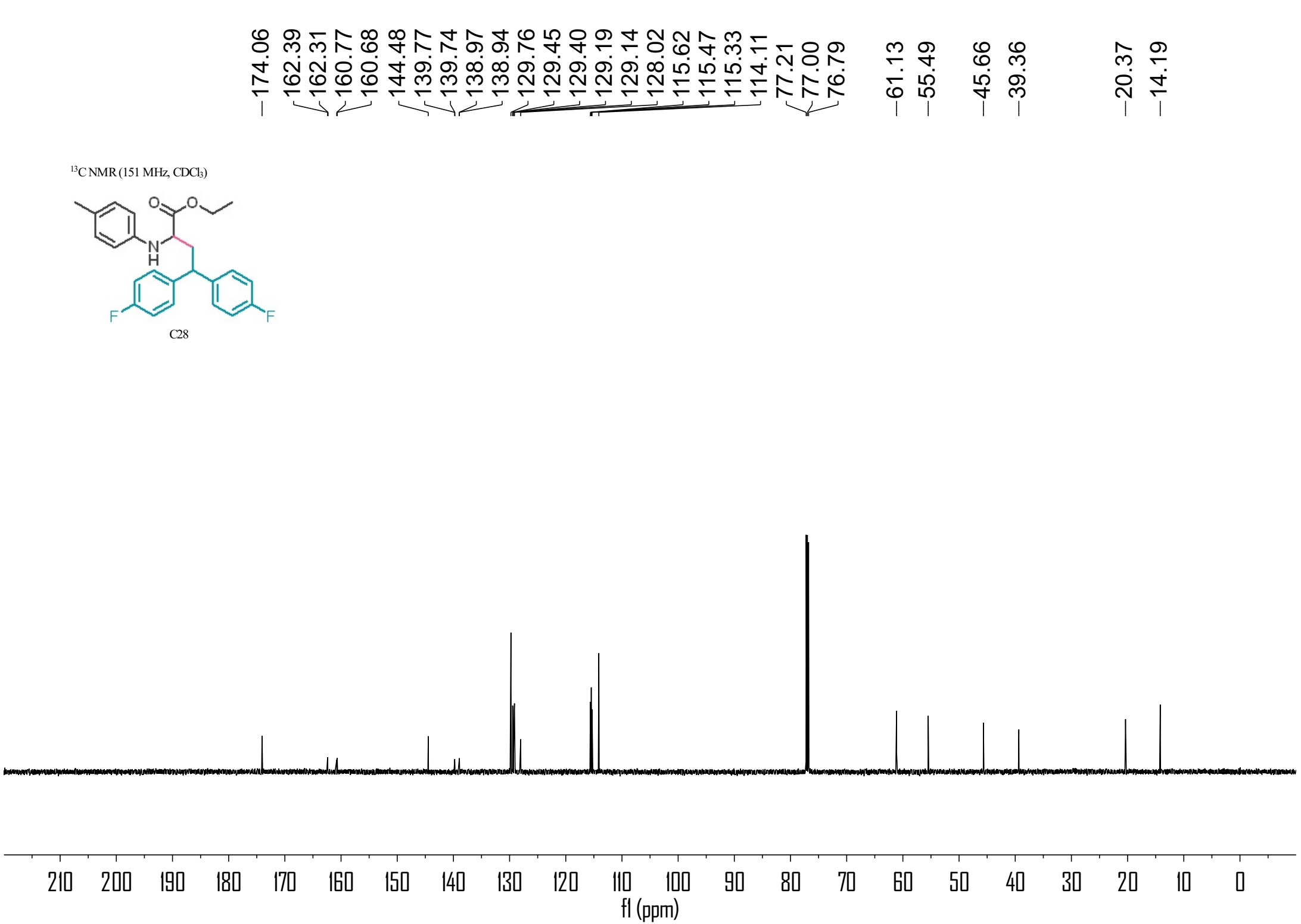


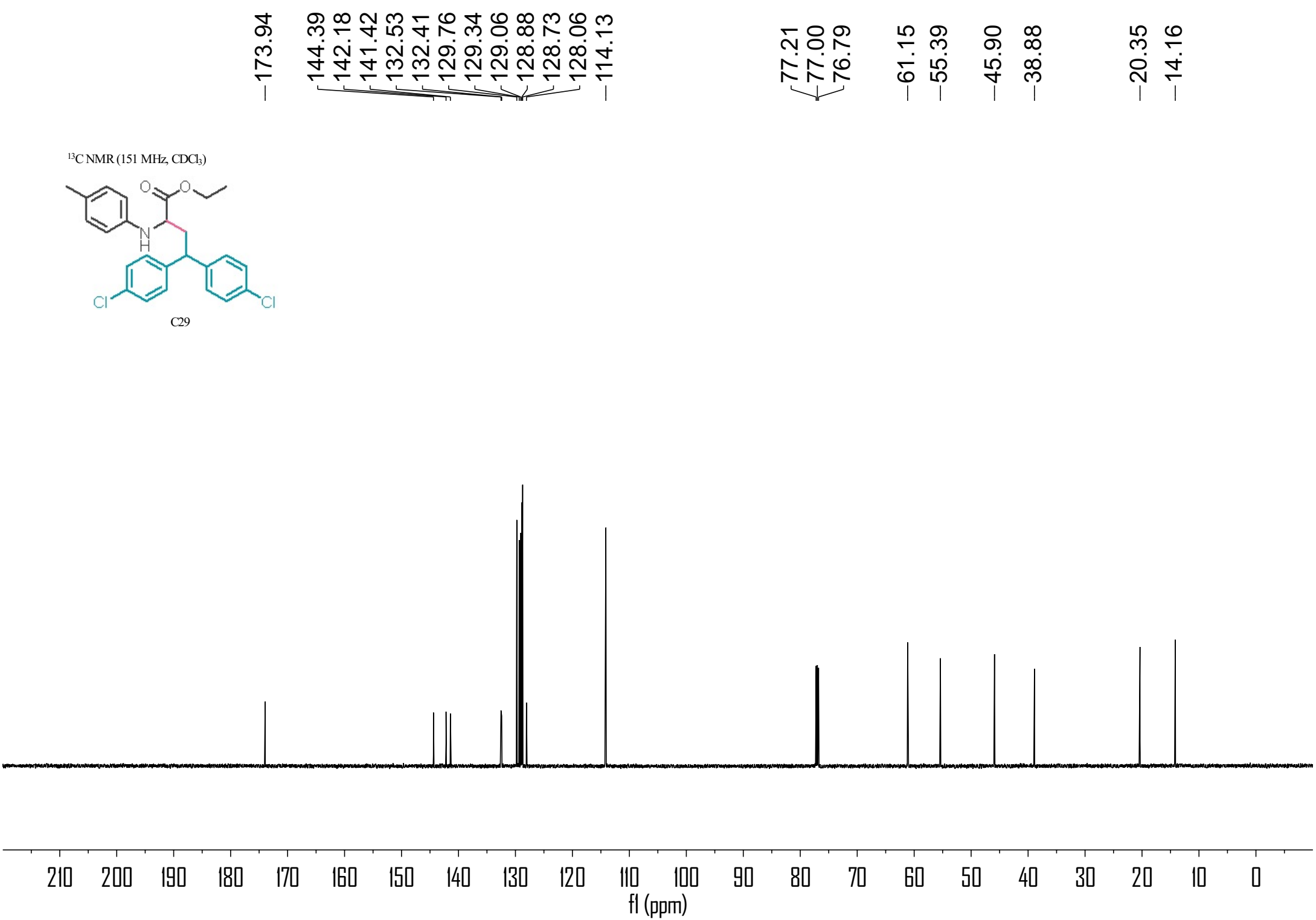


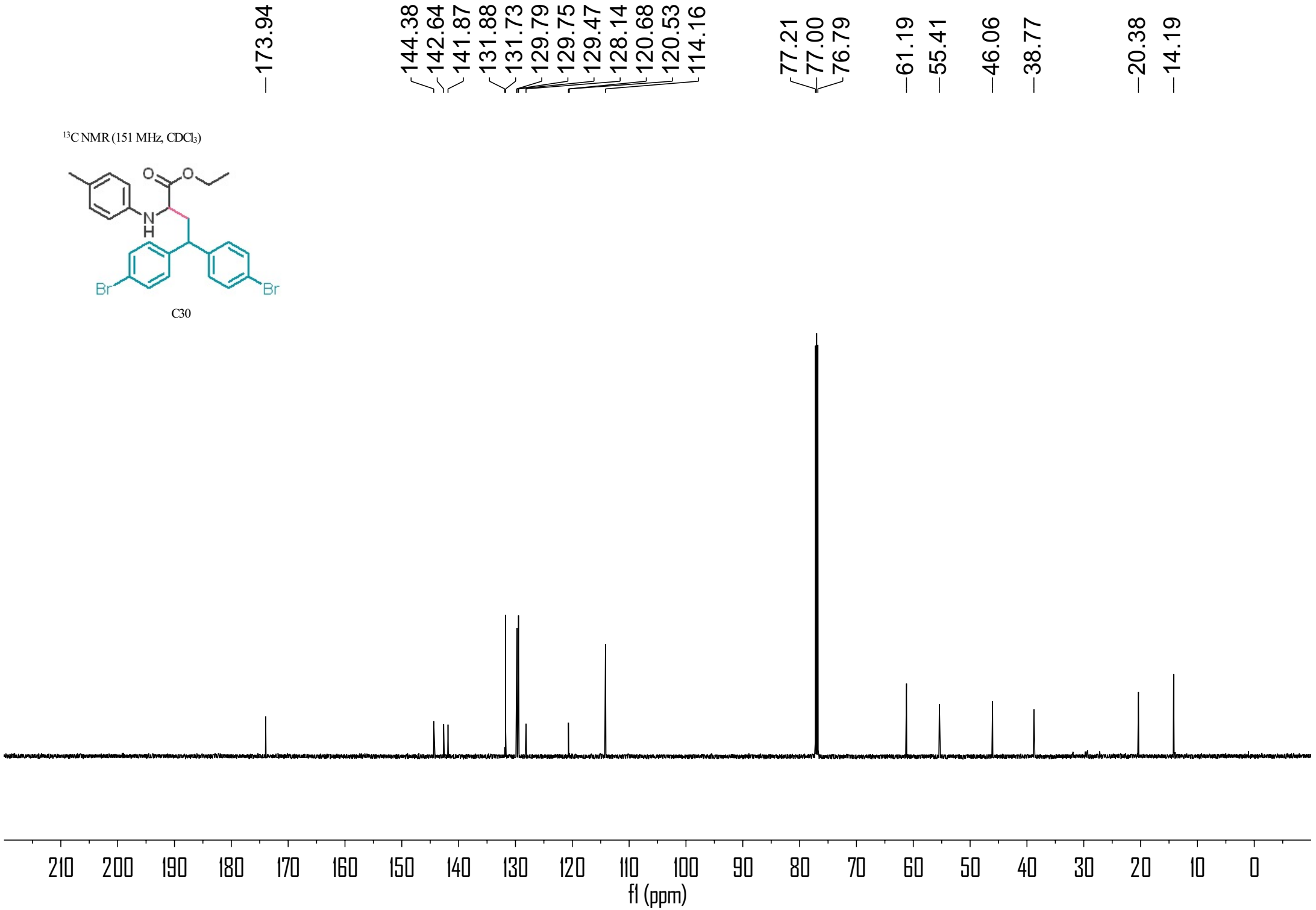
^{13}C NMR (151 MHz, $CDCl_3$)

C26


174.35
158.09
158.03
144.66
136.75
135.93
129.68
128.89
128.64
127.68
114.06
113.98
113.88
77.21
77.00
76.79
60.96
55.66
55.21
55.20
45.56
39.57
20.36
14.20
13C NMR (151 MHz, CDCl3)
O
O
N
H
O
O
C27
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


174.06
162.39
162.31
160.77
160.68
144.48
139.77
139.74
138.97
138.94
129.76
129.45
129.40
129.19
129.14
128.02
115.62
115.47
115.33
114.11
77.21
77.00
76.79
61.13
55.49
45.66
39.36
20.37
14.19
13C NMR (151 MHz, CDCl3)
O
O
N
H
F
F
C28
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


173.94
144.39
142.18
141.42
132.53
132.41
129.76
129.34
129.06
128.88
128.73
128.06
114.13
77.21
77.00
76.79
61.15
55.39
45.90
38.88
20.35
14.16
13C NMR (151 MHz, CDCl3)
O
O
N
H
Cl
Cl
C29
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

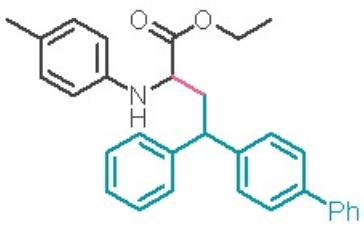


^{13}C NMR (151 MHz, $CDCl_3$)

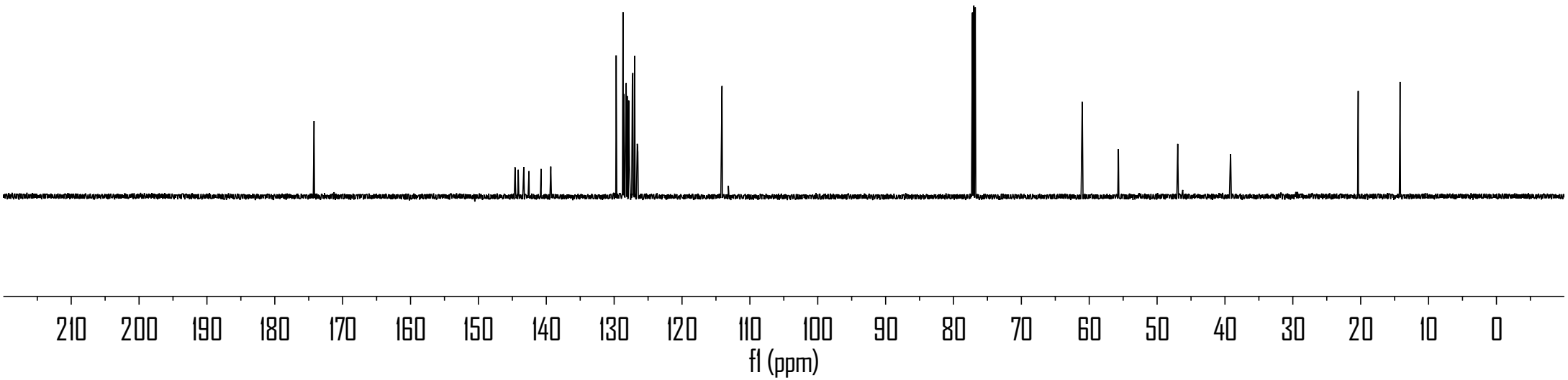
C30

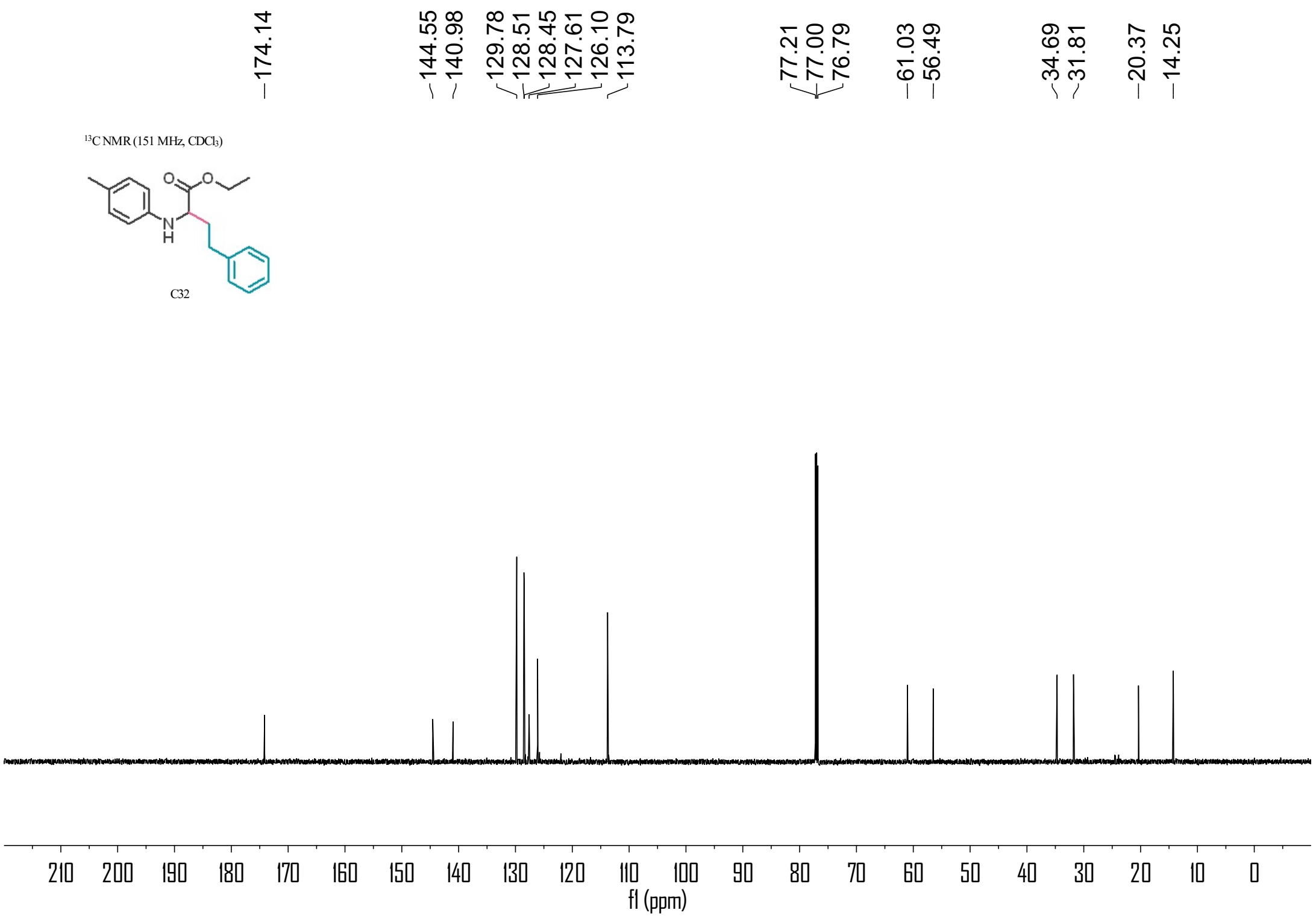
174.22
144.61
144.58
144.13
143.35
143.32
142.55
140.79
140.77
139.35
139.33
129.70
128.71
128.69
128.59
128.45
128.22
128.08
127.85
127.77
127.75
127.32
127.25
127.13
126.97
126.95
126.60
126.51
114.11
114.08
77.21
77.00
76.79
61.03
55.70
55.66
46.96
46.94
39.19
39.15
20.36
14.20

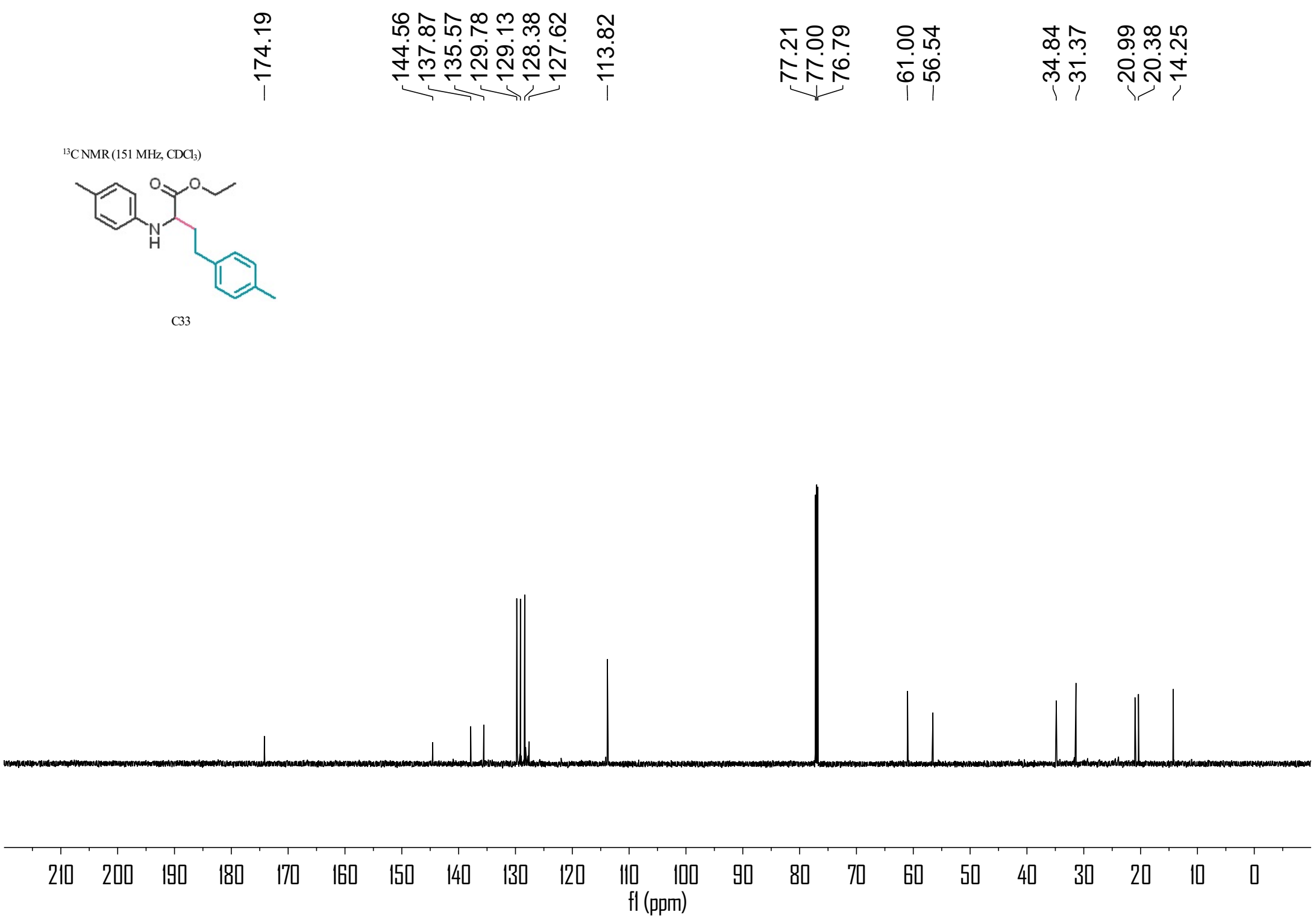
^{13}C NMR (151 MHz, $CDCl_3$)

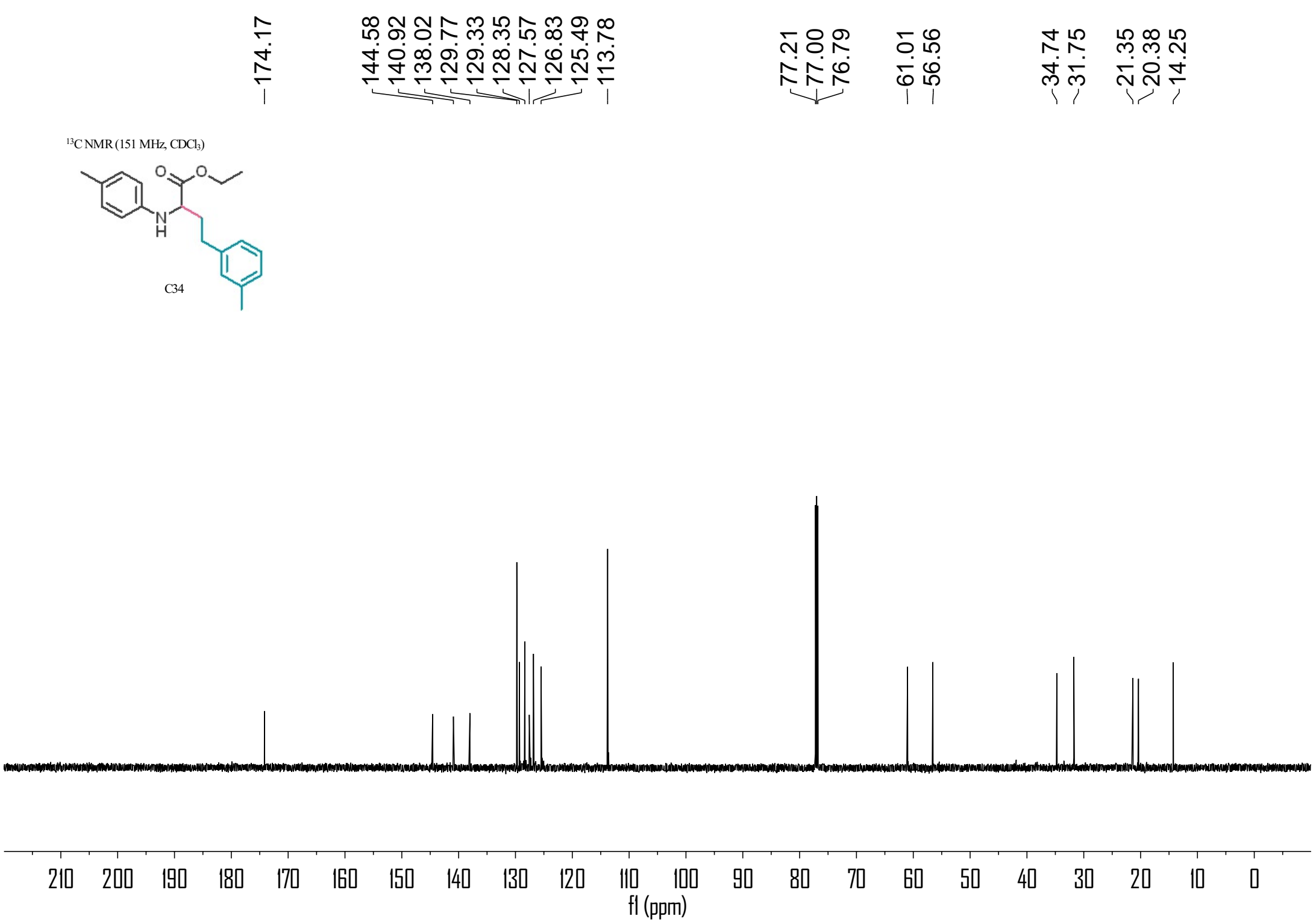


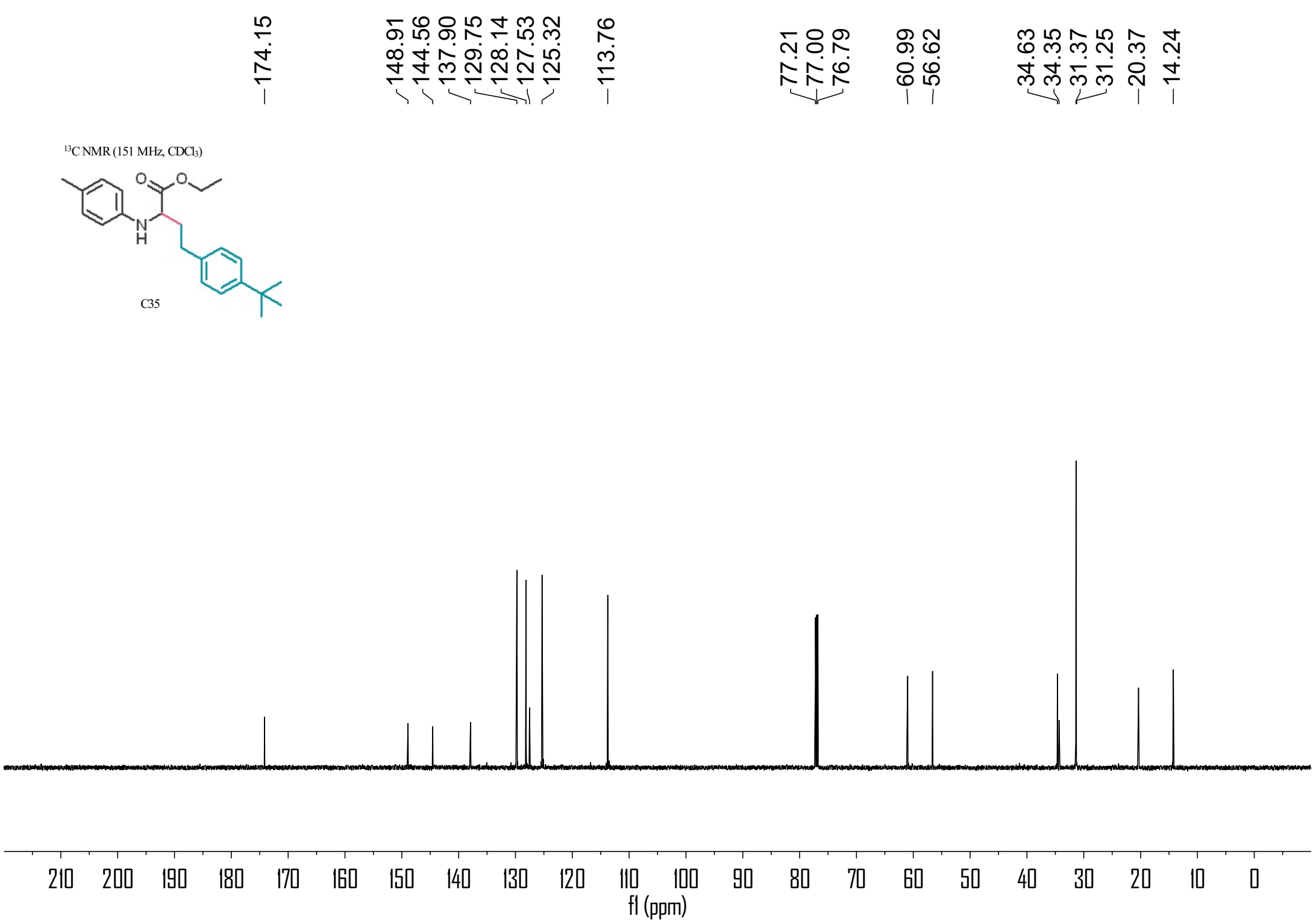
C31 (dr = 6.1:1)

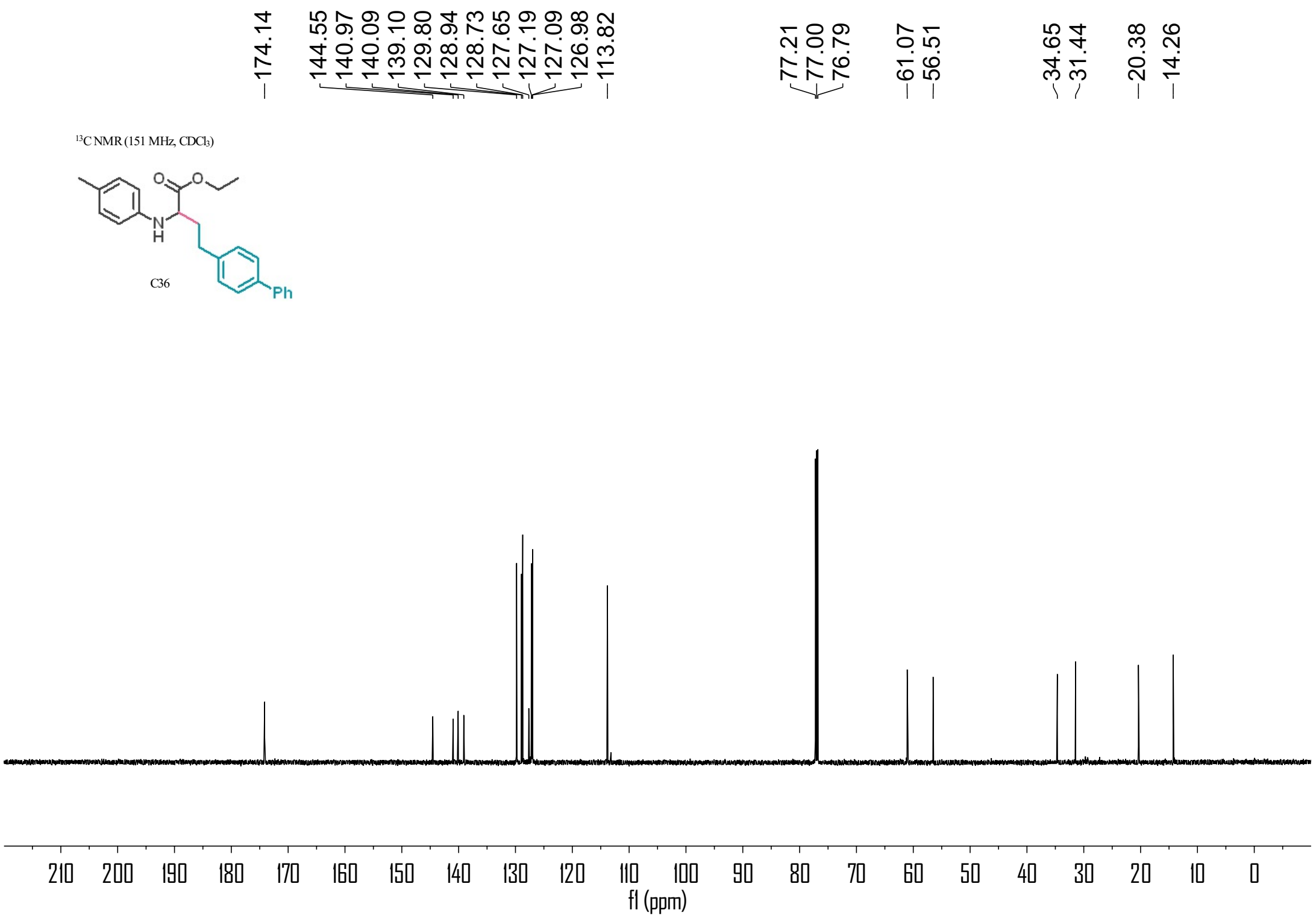


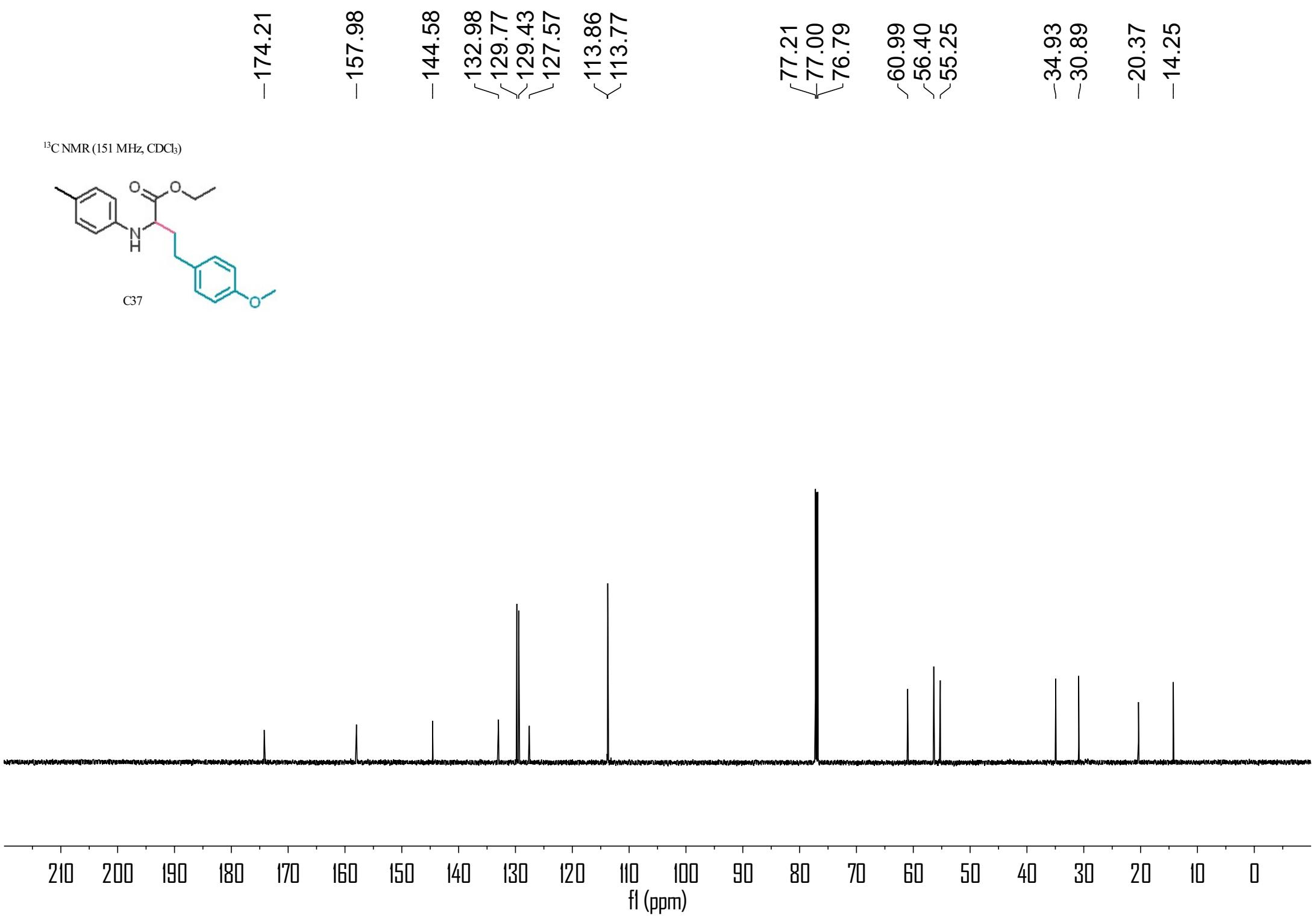

174.14
144.55
140.98
129.78
128.51
128.45
127.61
126.10
113.79
77.21
77.00
76.79
61.03
56.49
34.69
31.81
20.37
14.25
13C NMR (151 MHz, CDCl3)
O
O
N
H
C32
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

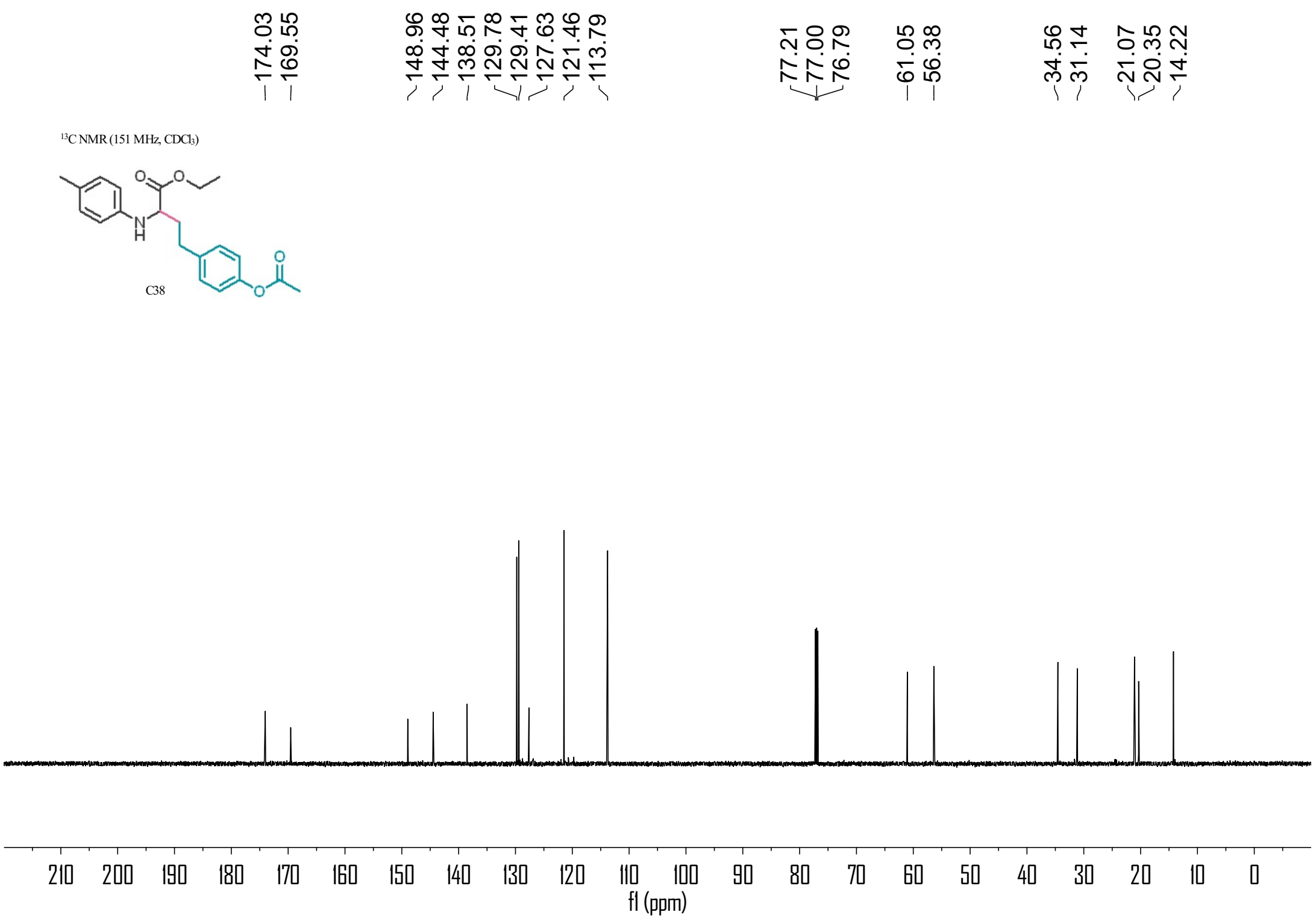

174.19
144.56
137.87
135.57
129.78
129.13
128.38
127.62
113.82
77.21
77.00
76.79
61.00
56.54
34.84
31.37
20.99
20.38
14.25
13C NMR (151 MHz, CDCl3)
O
O
N
H
C33
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

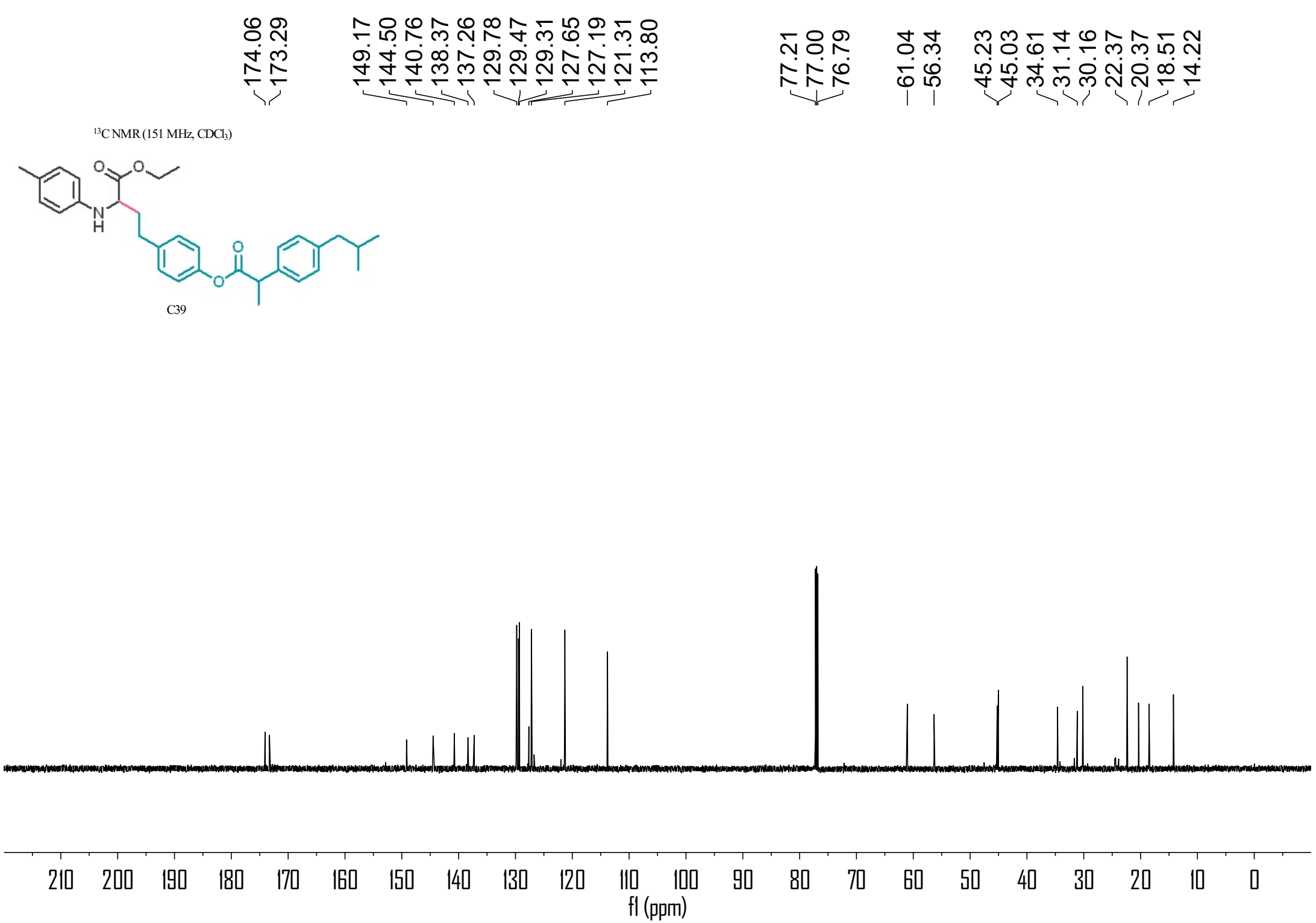

174.17
144.58
140.92
138.02
129.77
129.33
128.35
127.57
126.83
125.49
113.78
77.21
77.00
76.79
61.01
56.56
34.74
31.75
21.35
20.38
14.25
13C NMR (151 MHz, CDCl3)
O
O
N
H
C34
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

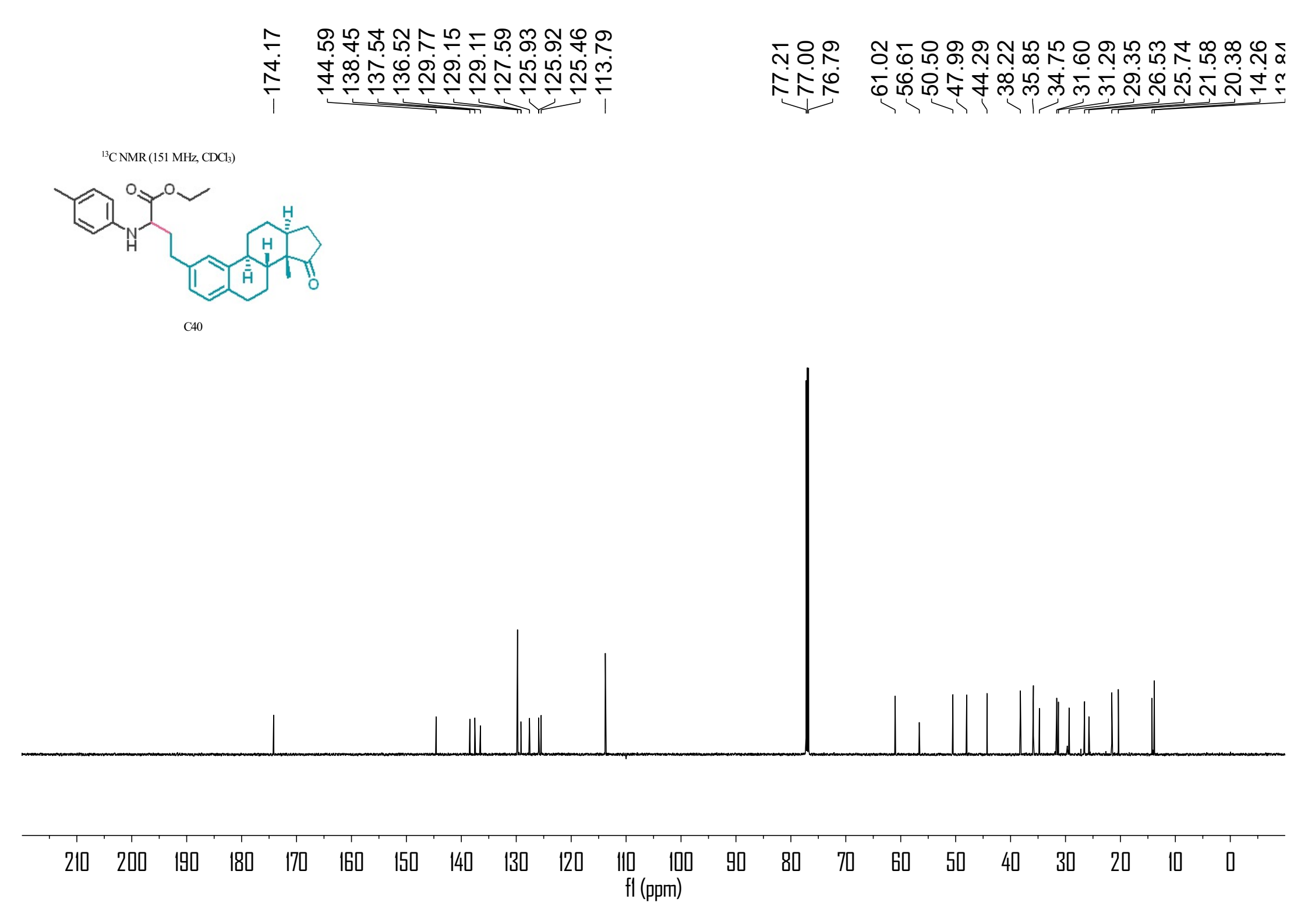

174.15
148.91
144.56
137.90
129.75
128.14
127.53
125.32
113.76
77.21
77.00
76.79
60.99
56.62
34.63
34.35
31.37
31.25
20.37
14.24
13C NMR (151 MHz, CDCl3)
O
O
N
H
C35
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

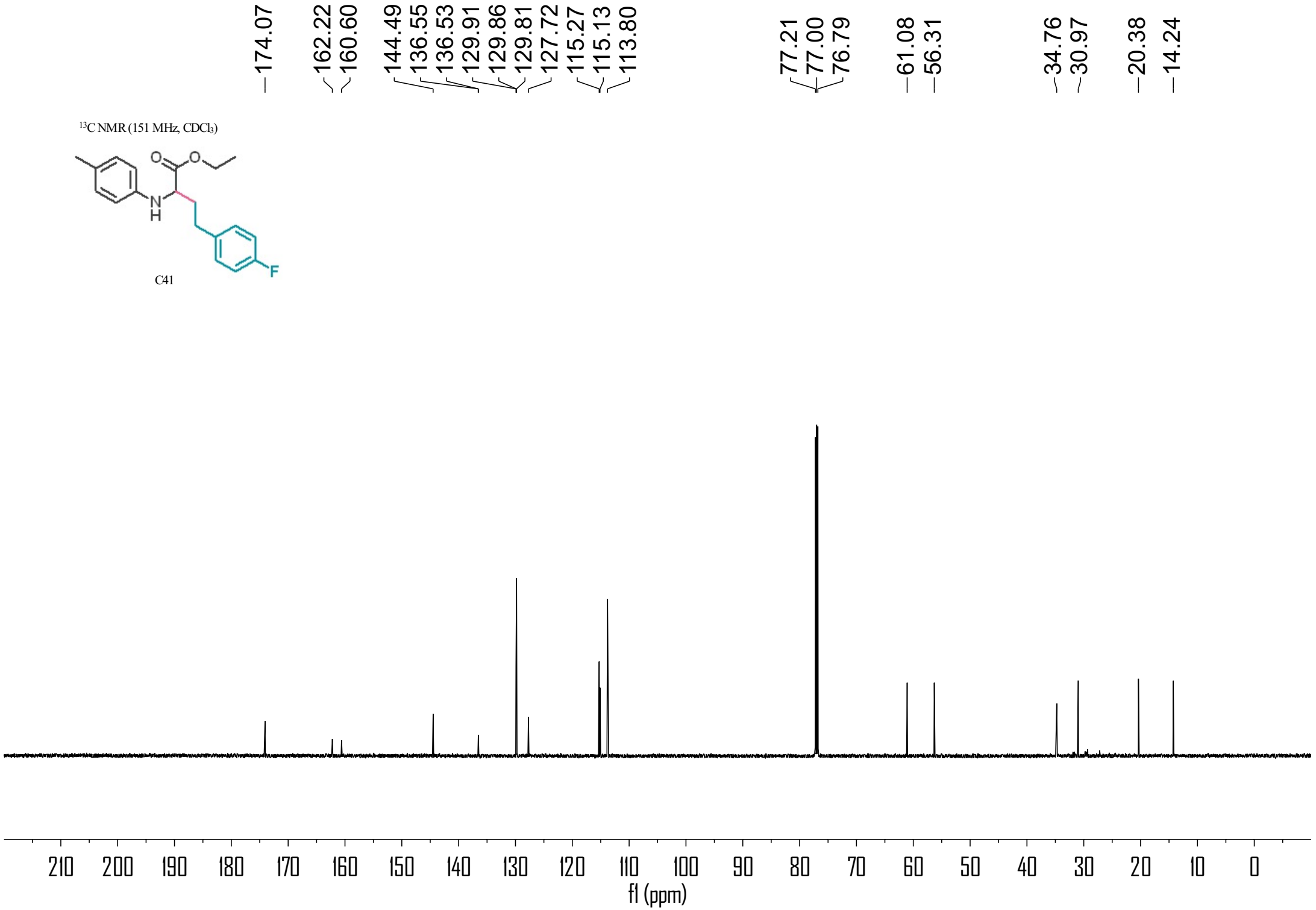

174.14
144.55
140.97
140.09
139.10
129.80
128.94
128.73
127.65
127.19
127.09
126.98
113.82
77.21
77.00
76.79
61.07
56.51
34.65
31.44
20.38
14.26
13C NMR (151 MHz, CDCl3)
O
O
N
H
C36
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

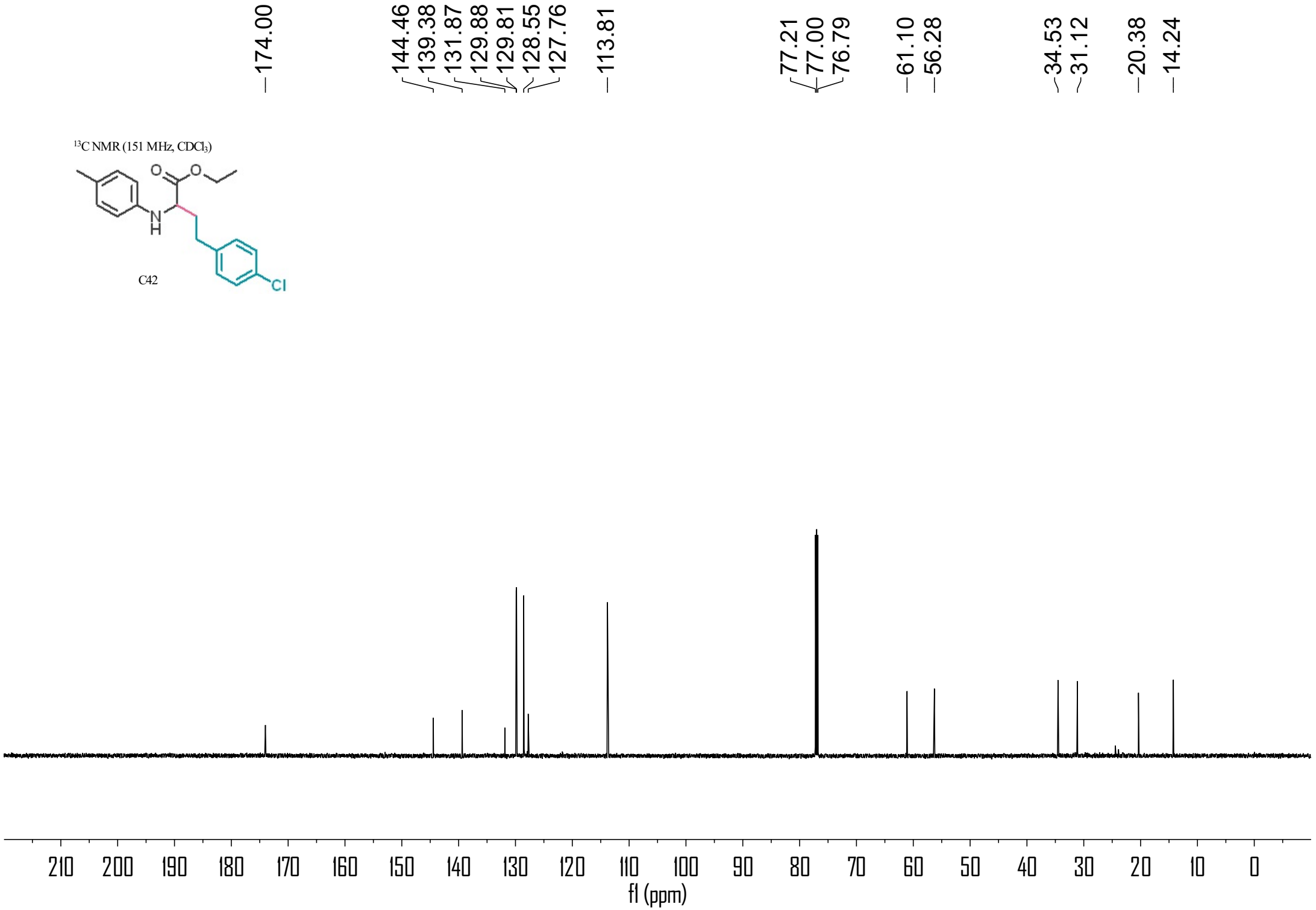

174.21
157.98
144.58
132.98
129.77
129.43
127.57
113.86
113.77
77.21
77.00
76.79
60.99
56.40
55.25
34.93
30.89
20.37
14.25
13C NMR (151 MHz, CDCl3)
O
O
N
H
O
C37
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

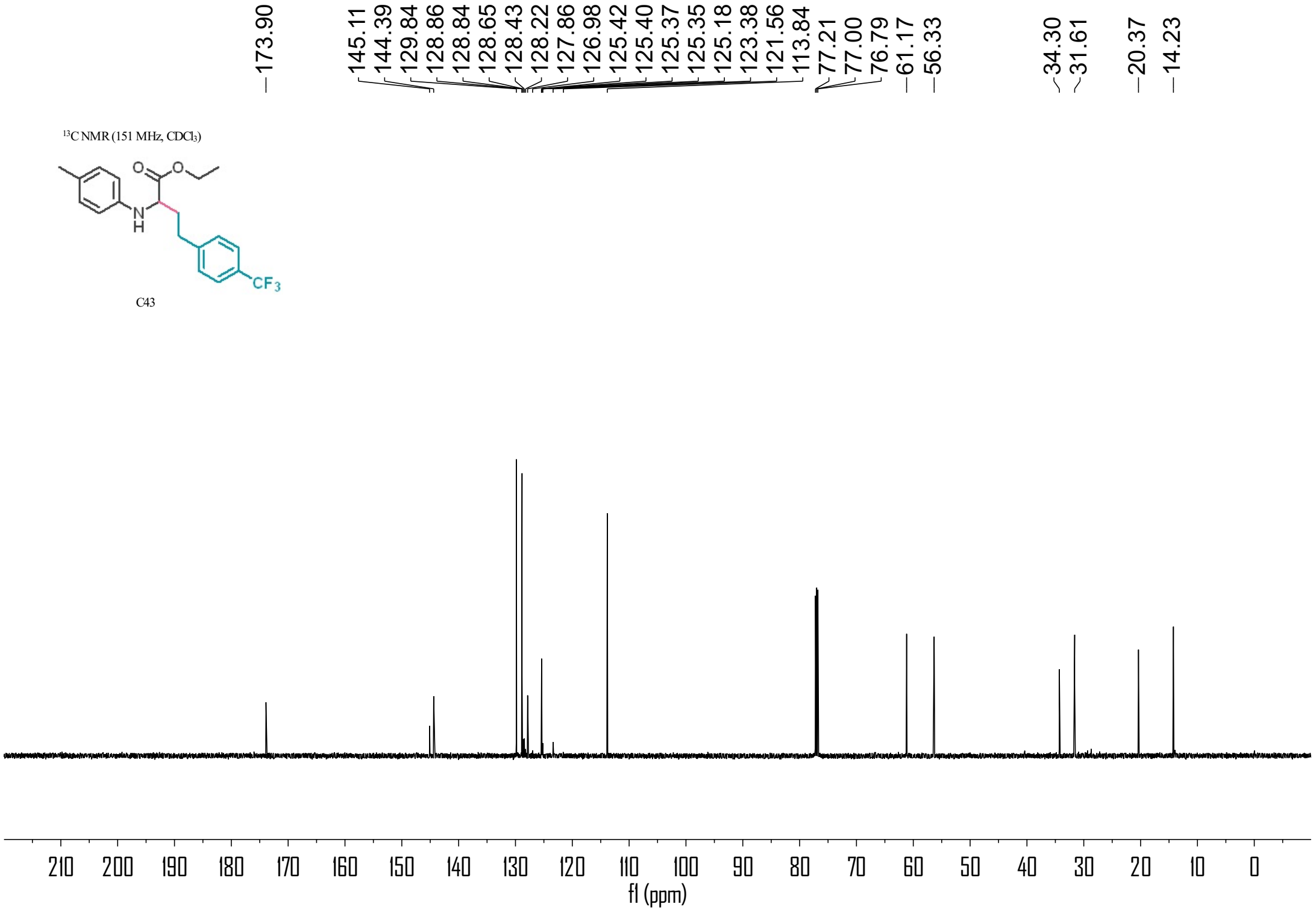

174.03
169.55
148.96
144.48
138.51
129.78
129.41
127.63
121.46
113.79
77.21
77.00
76.79
61.05
56.38
34.56
31.14
21.07
20.35
14.22
13C NMR (151 MHz, CDCl3)
C38
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

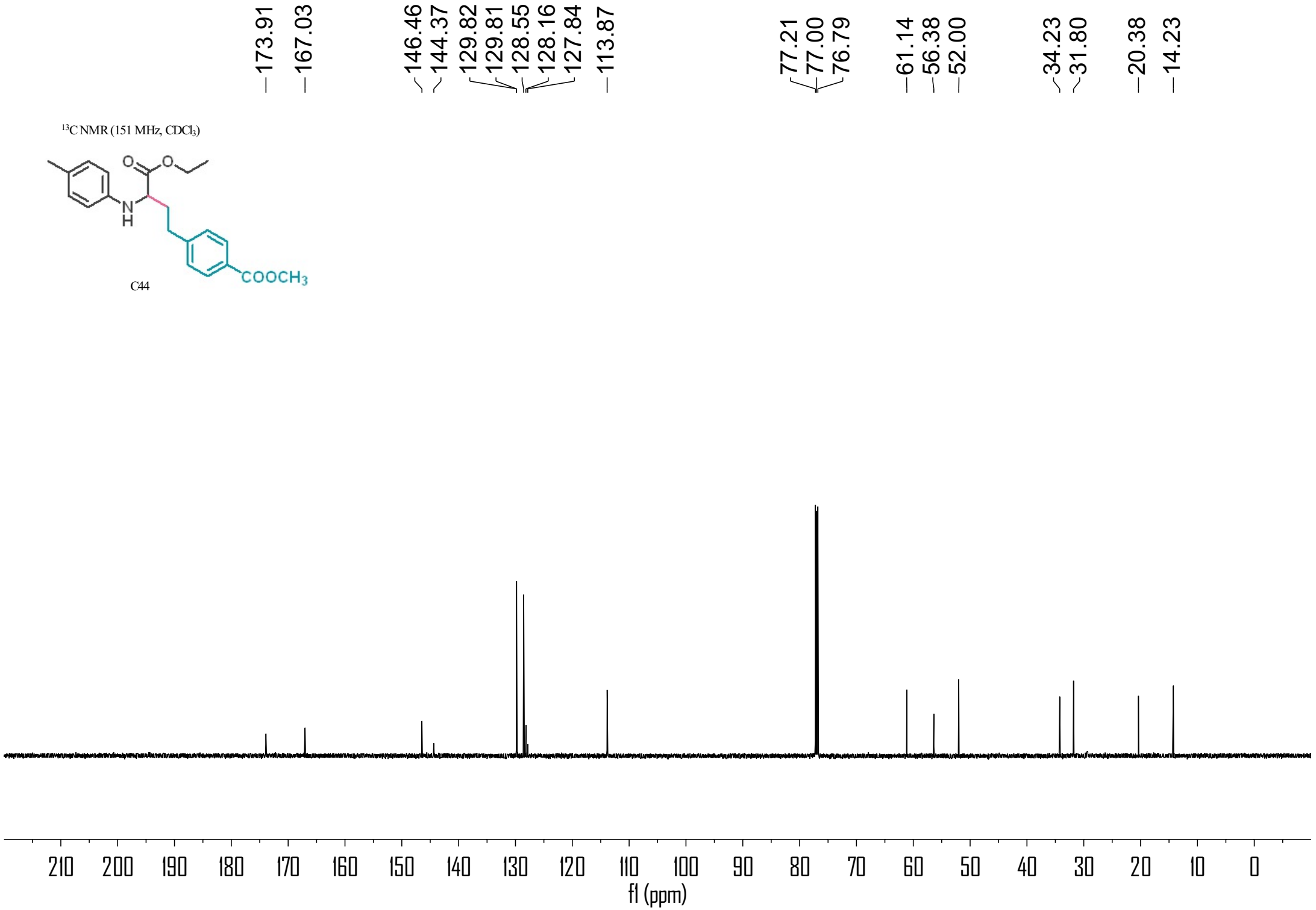

174.06
173.29
149.17
144.50
140.76
138.37
137.26
129.78
129.47
129.31
127.65
127.19
121.31
113.80
77.21
77.00
76.79
61.04
56.34
45.23
45.03
34.61
31.14
30.16
22.37
20.37
18.51
14.22
13C NMR (151 MHz, CDCl3)
O
O
N
H
O
O
C39
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

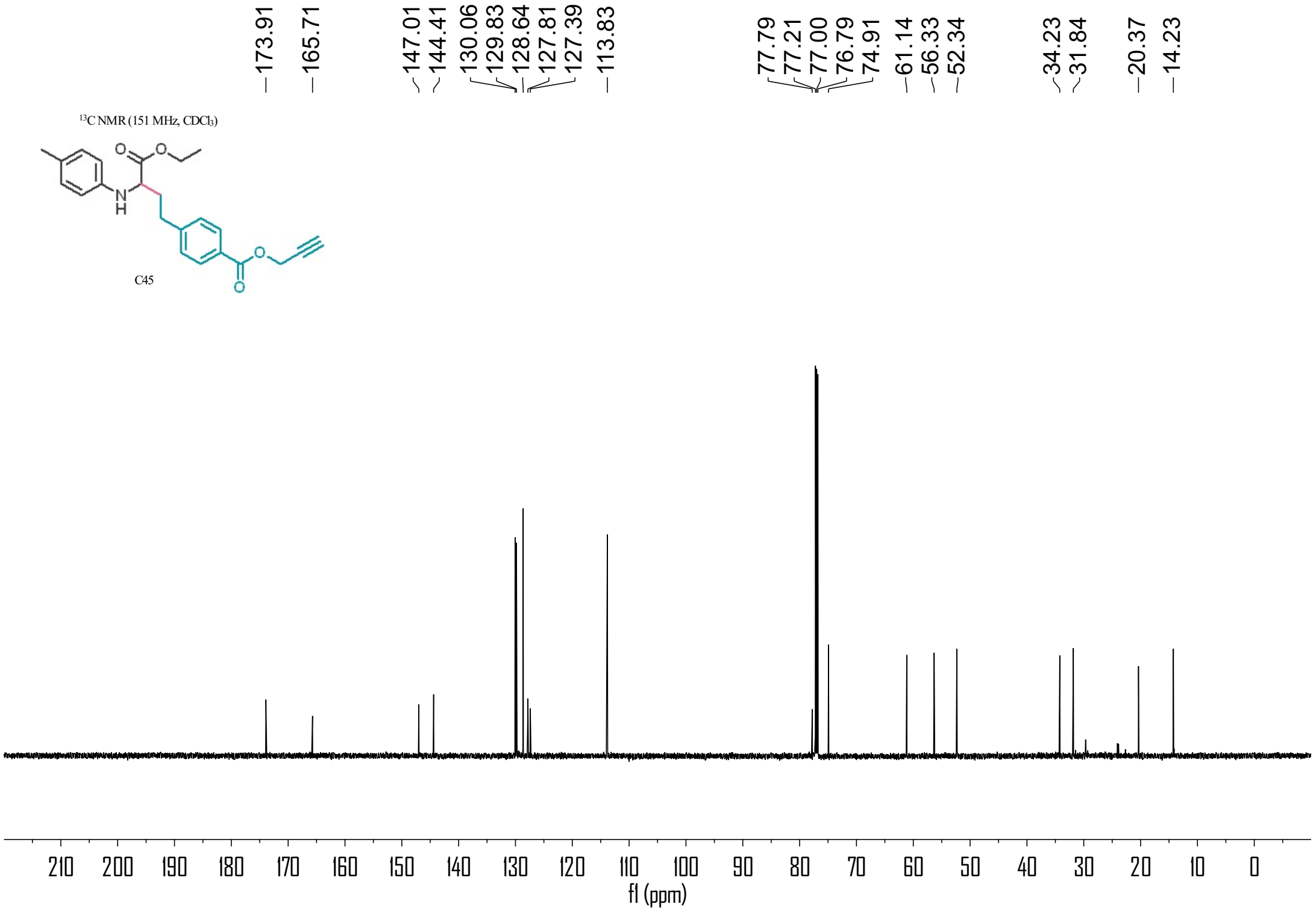

174.17
144.59
138.45
137.54
136.52
129.77
129.15
129.11
127.59
125.93
125.92
125.46
113.79
77.21
77.00
76.79
61.02
56.61
50.50
47.99
44.29
38.22
35.85
34.75
31.60
31.29
29.35
26.53
25.74
21.58
20.38
14.26
13.84
13C NMR (151 MHz, CDCl3)
O
O
N
H
H
H
H
O
C40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

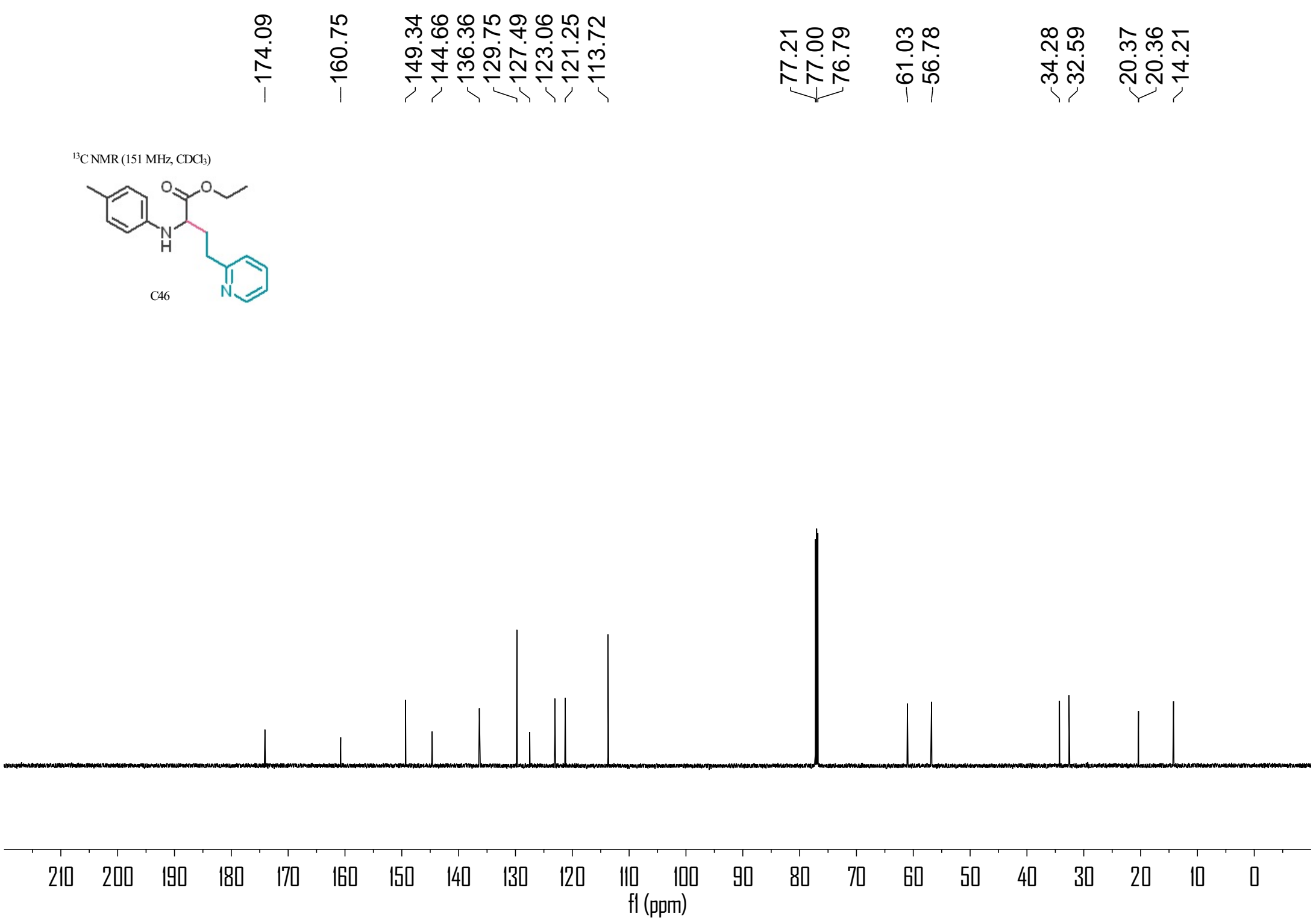

174.07
162.22
160.60
144.49
136.55
136.53
129.91
129.86
129.81
127.72
115.27
115.13
113.80
77.21
77.00
76.79
61.08
56.31
34.76
30.97
20.38
14.24
13C NMR (151 MHz, CDCl3)
O
O
N
H
F
C41
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

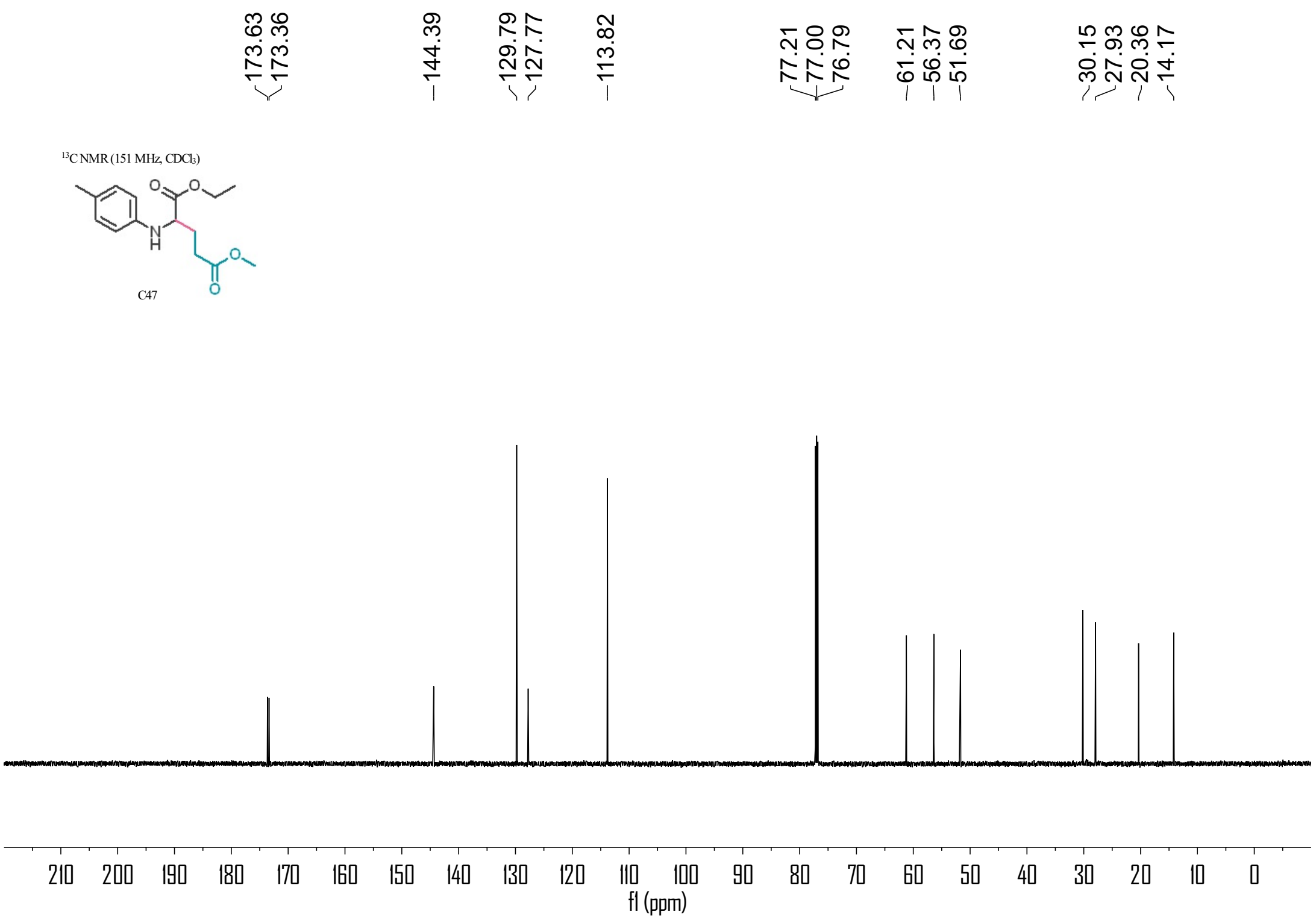

174.00
144.46
139.38
131.87
129.88
129.81
128.55
127.76
113.81
77.21
77.00
76.79
61.10
56.28
34.53
31.12
20.38
14.24
13C NMR (151 MHz, CDCl3)
O
O
N
H
Cl
C42
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

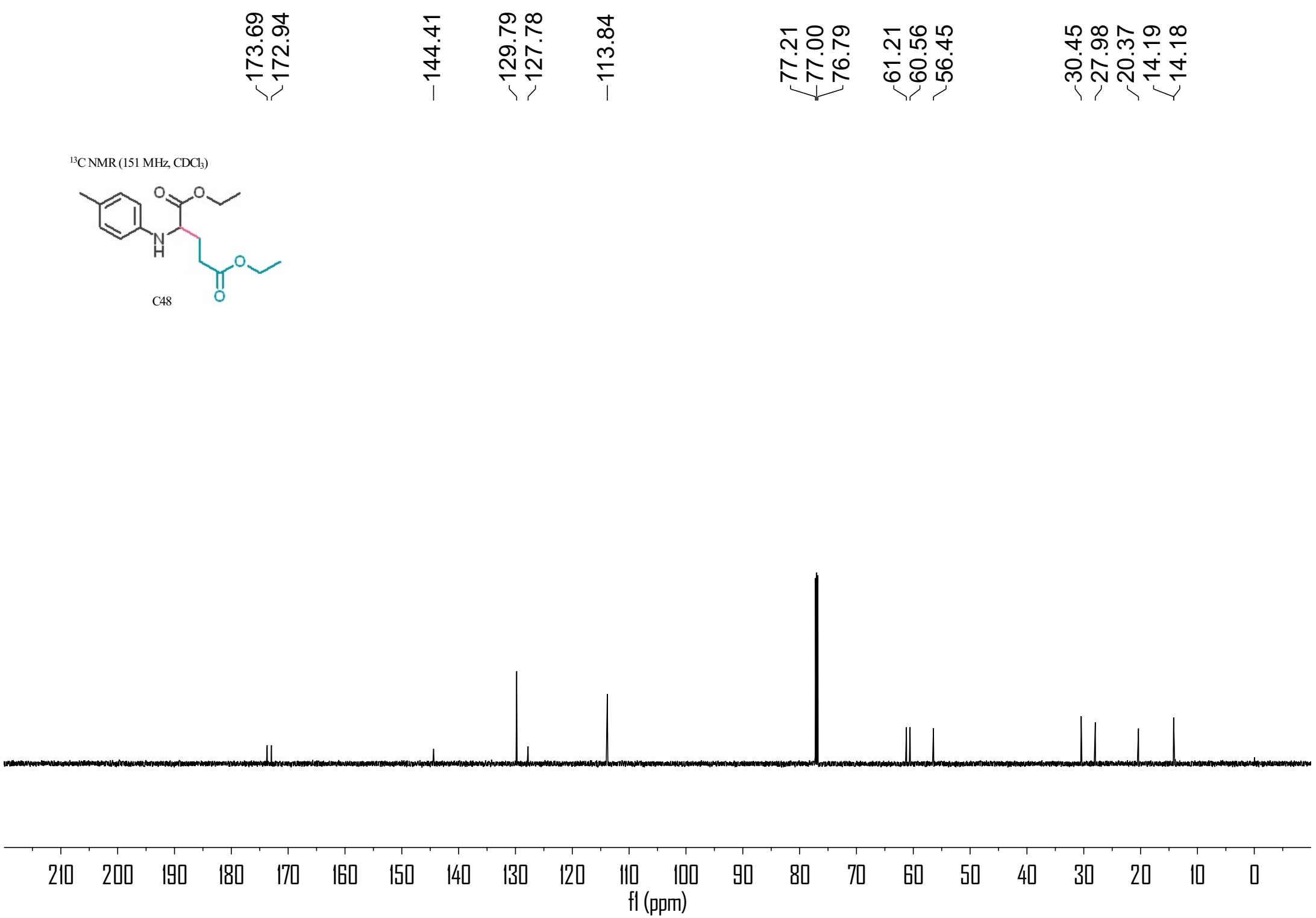

173.90
145.11
144.39
129.84
128.86
128.84
128.65
128.43
128.22
127.86
126.98
125.42
125.40
125.37
125.35
125.18
123.38
121.56
113.84
77.21
77.00
76.79
61.17
56.33
34.30
31.61
20.37
14.23
13C NMR (151 MHz, CDCl3)
O
O
N
H
CF3
C43
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

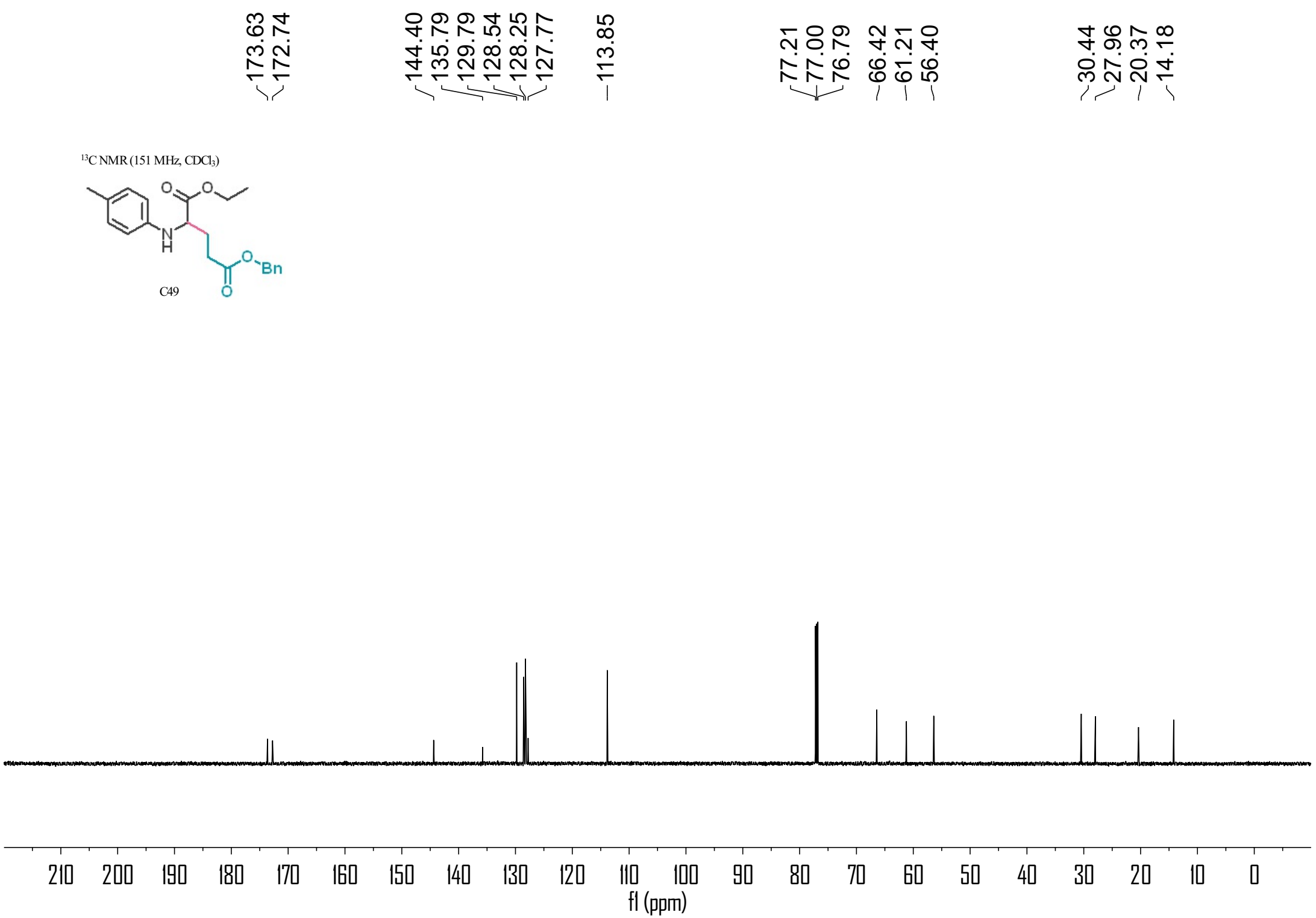

173.91
167.03
146.46
144.37
129.82
129.81
128.55
128.16
127.84
113.87
77.21
77.00
76.79
61.14
56.38
52.00
34.23
31.80
20.38
14.23
13C NMR (151 MHz, CDCl3)
O
O
N
H
COOCH3
C44
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


173.91
165.71
147.01
144.41
130.06
129.83
128.64
127.81
127.39
113.83
77.79
77.21
77.00
76.79
74.91
61.14
56.33
52.34
34.23
31.84
20.37
14.23
13C NMR (151 MHz, CDCl3)
C45
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


174.09
160.75
149.34
144.66
136.36
129.75
127.49
123.06
121.25
113.72
77.21
77.00
76.79
61.03
56.78
34.28
32.59
20.37
20.36
14.21
13C NMR (151 MHz, CDCl3)
O
O
N
H
N
C46
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

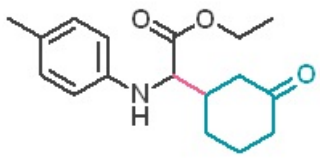

173.63
173.36
144.39
129.79
127.77
113.82
77.21
77.00
76.79
61.21
56.37
51.69
30.15
27.93
20.36
14.17
13C NMR (151 MHz, CDCl3)
C47
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


173.69
172.94
144.41
129.79
127.78
113.84
77.21
77.00
76.79
61.21
60.56
56.45
30.45
27.98
20.37
14.19
14.18
13C NMR (151 MHz, CDCl3)
C48
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

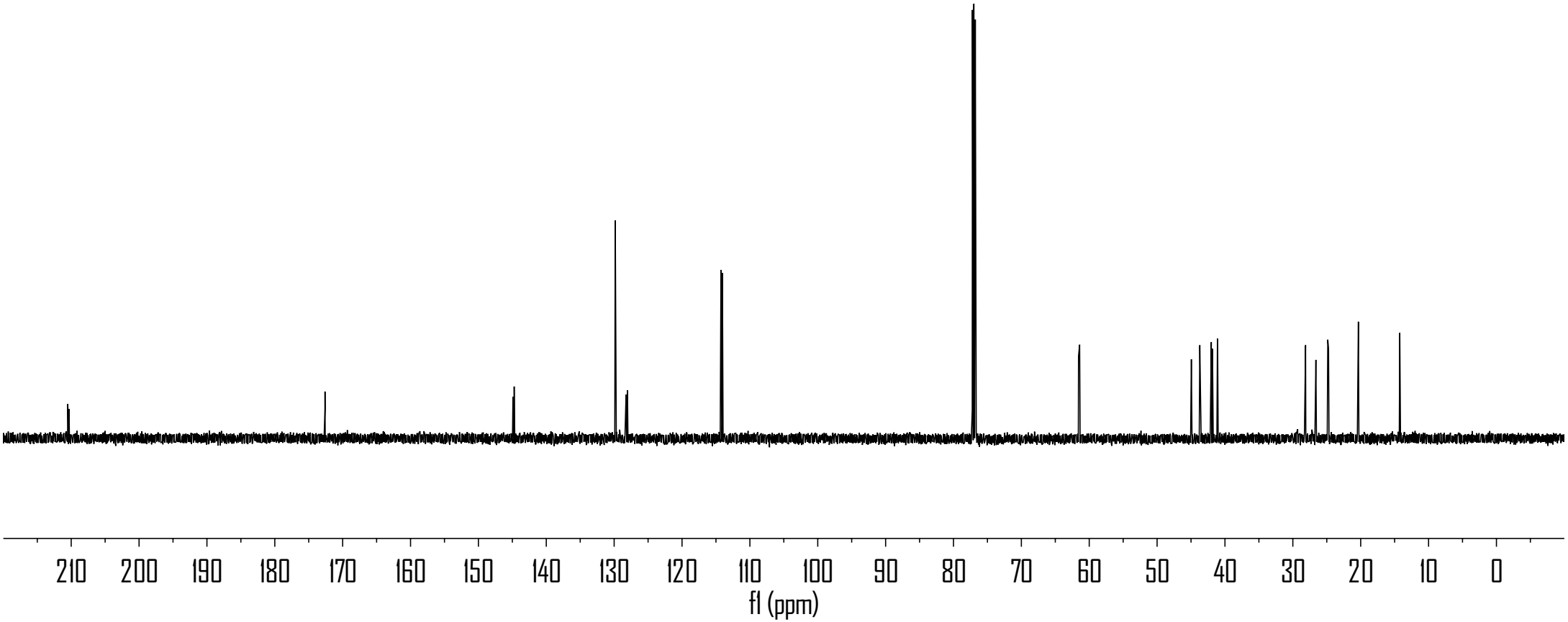

173.63
172.74
144.40
135.79
129.79
128.54
128.25
127.77
113.85
77.21
77.00
76.79
66.42
61.21
56.40
30.44
27.96
20.37
14.18
13C NMR (151 MHz, CDCl3)
O
O
N
H
O
Bn
O
C49
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

210.50
210.32
172.75
172.60
144.91
144.73
129.85
129.83
128.24
128.04
114.26
114.05
77.21
77.00
76.79
61.58
61.41
61.39
61.31
44.95
43.71
42.01
41.84
41.11
41.07
28.12
26.60
24.85
24.74
20.34
14.24
14.22

^{13}C NMR (151 MHz, $CDCl_3$)

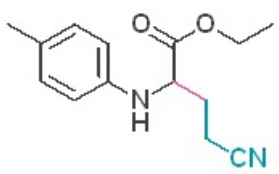


C50 (dr = 1.1:1)

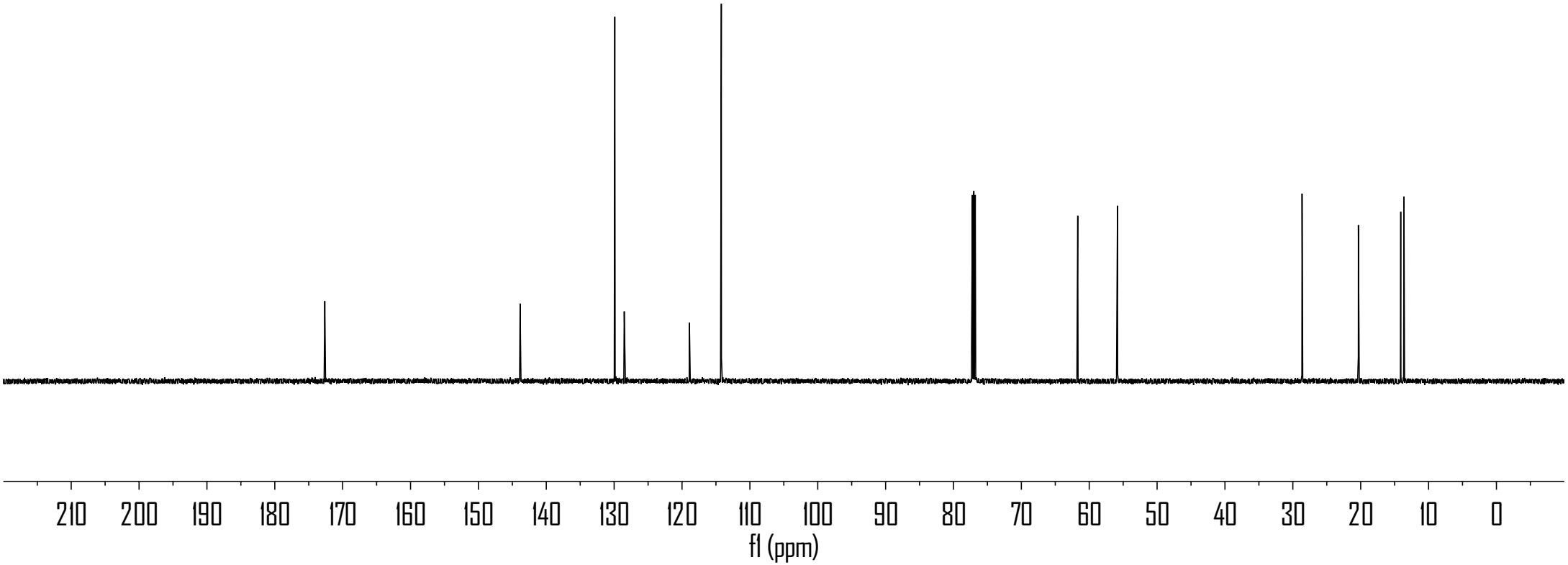


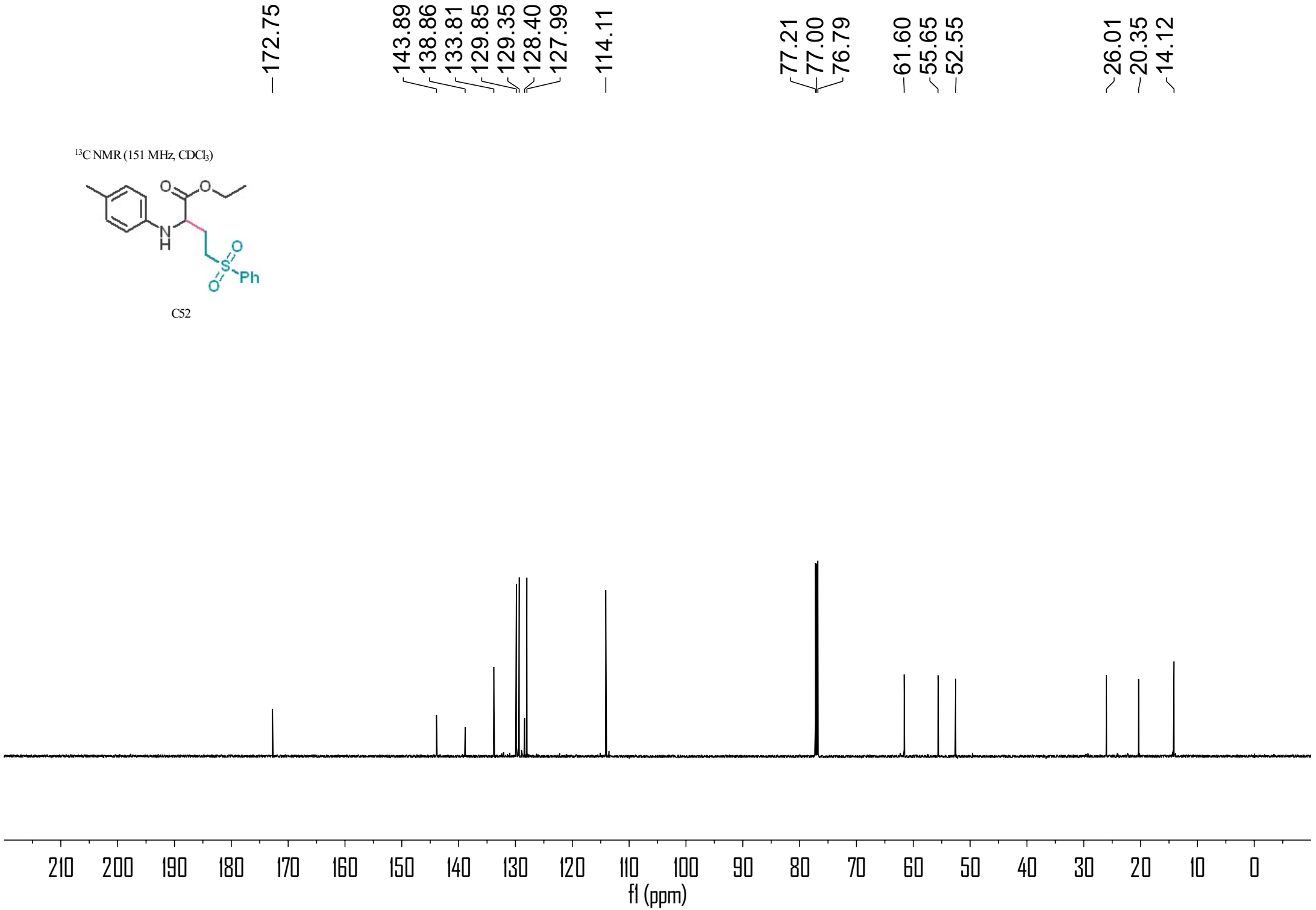
172.63
143.86
129.90
128.51
118.91
114.21
77.21
77.00
76.79
61.69
55.83
28.64
20.34
14.10
13.63

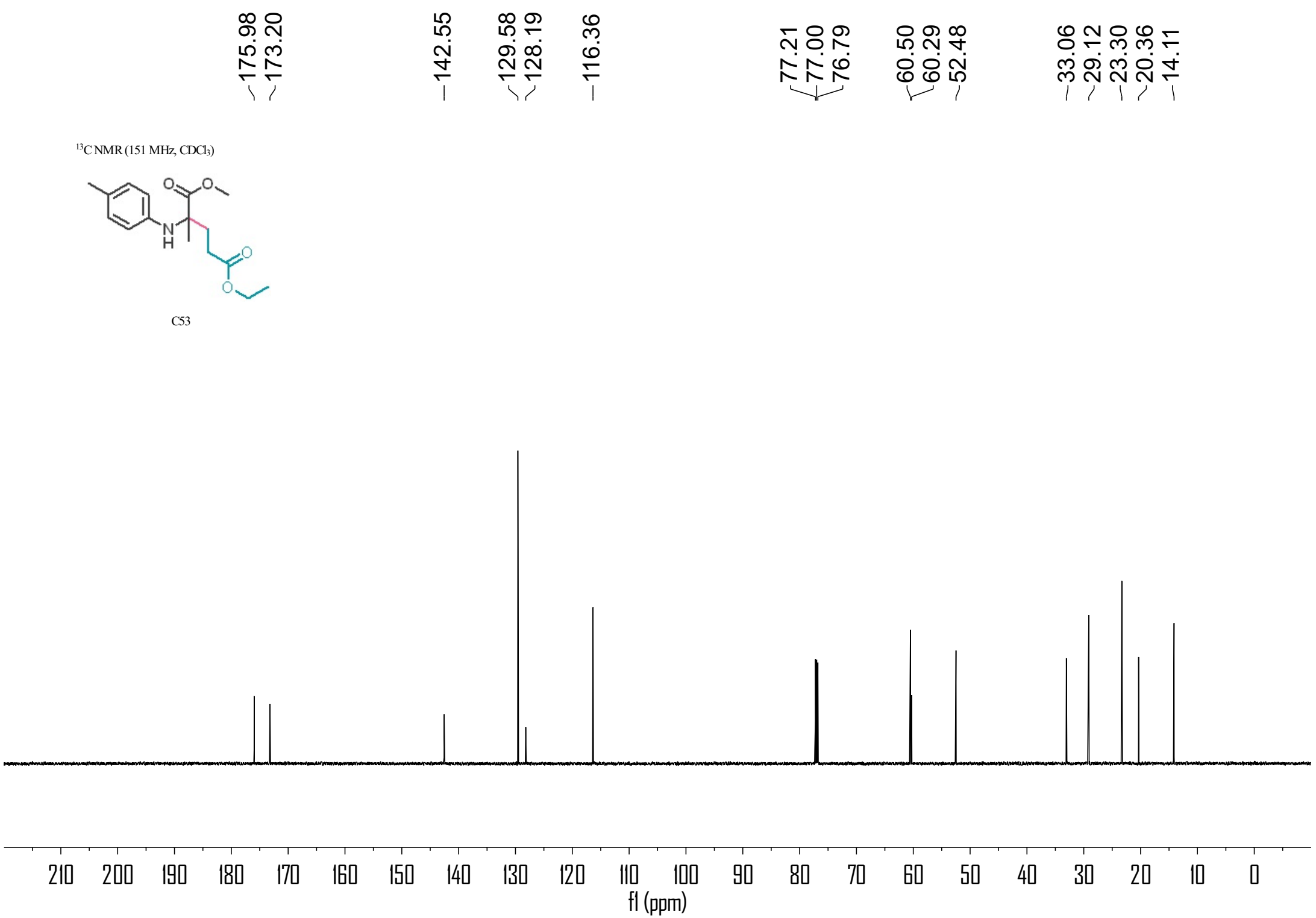
^{13}C NMR (151 MHz, $CDCl_3$)



C51

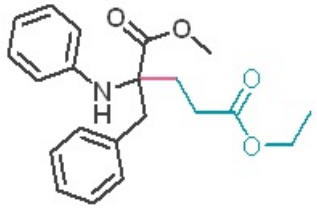



172.75
143.89
138.86
133.81
129.85
129.35
128.40
127.99
114.11
77.21
77.00
76.79
61.60
55.65
52.55
26.01
20.35
14.12
13C NMR (151 MHz, CDCl3)
O
O
N
H
S
O
O
Ph
C52
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

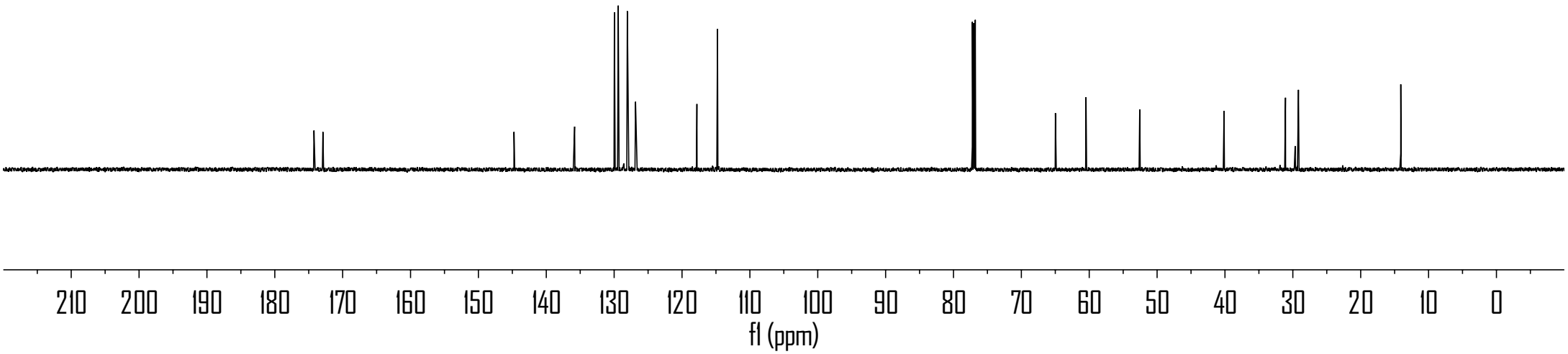

175.98
173.20
142.55
129.58
128.19
116.36
77.21
77.00
76.79
60.50
60.29
52.48
33.06
29.12
23.30
20.36
14.11
^{13}C NMR (151 MHz, $CDCl_3$)
C53
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

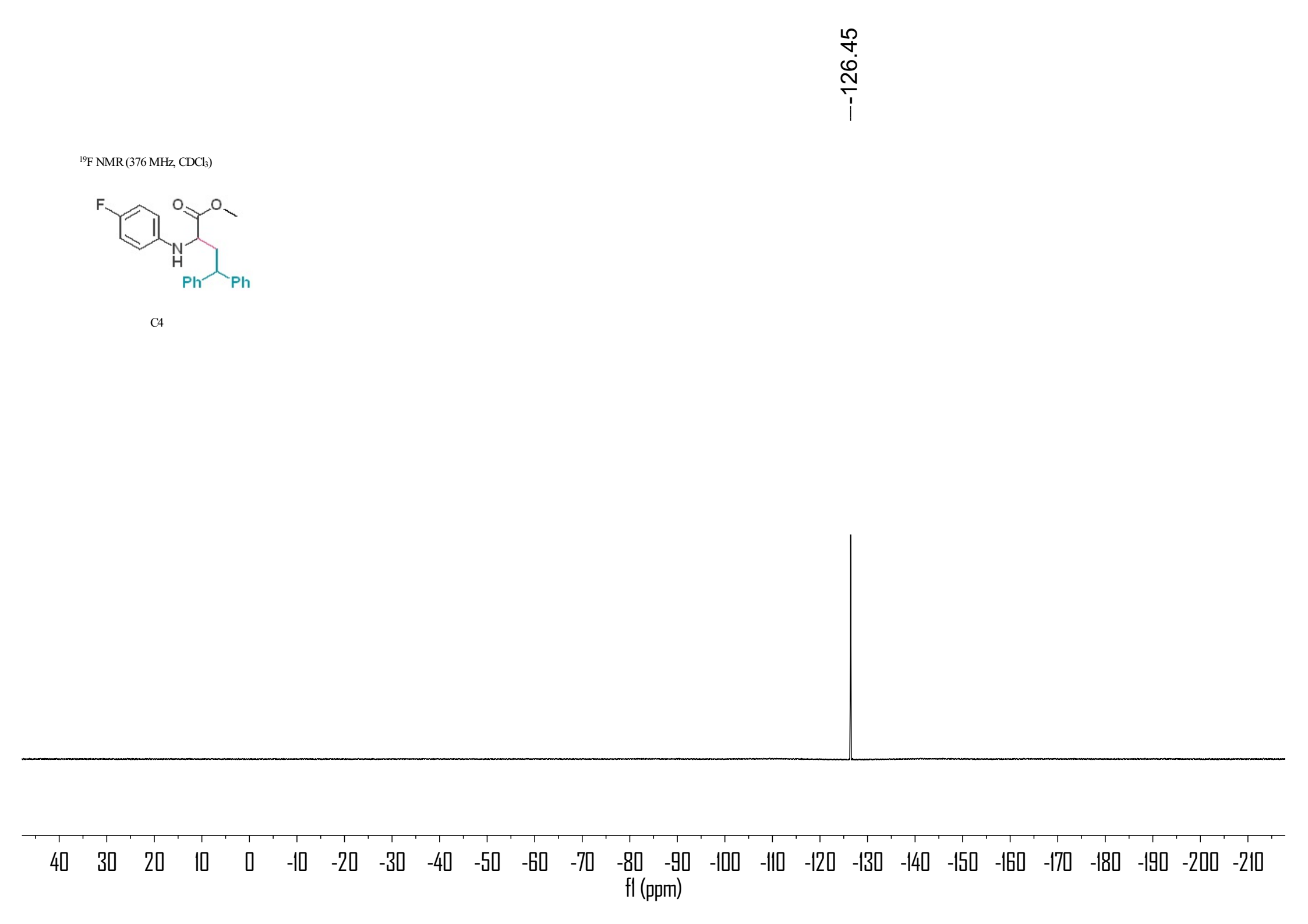
174.23
172.91
144.75
135.83
129.96
129.40
128.06
126.85
117.81
114.77
77.21
77.00
76.79
64.98
60.47
52.56
40.14
31.08
29.18
14.10

^{13}C NMR (151 MHz, $CDCl_3$)

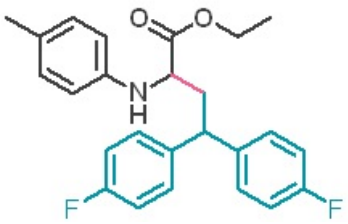


C54



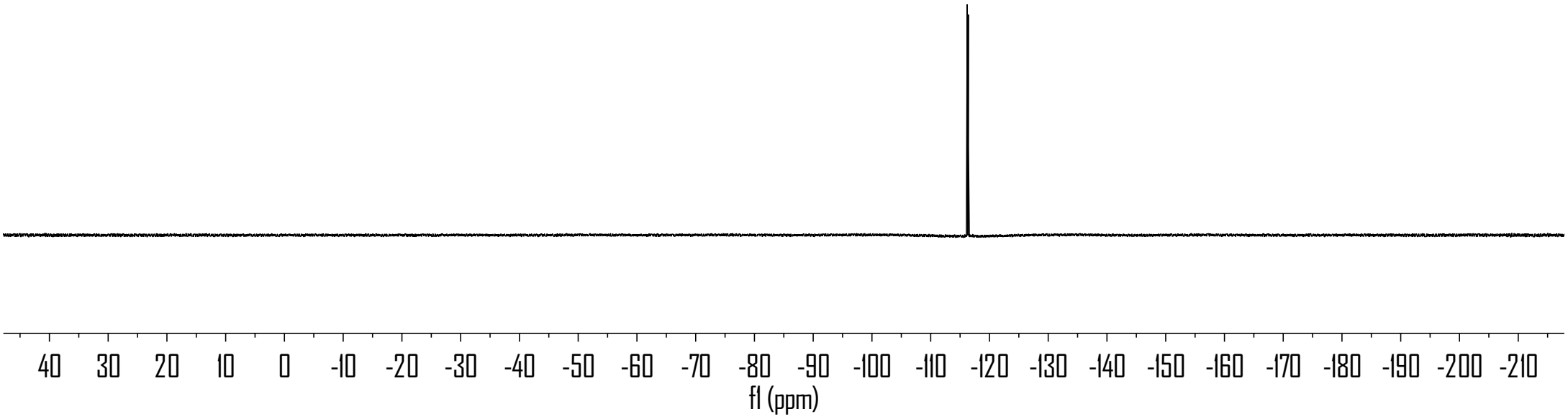

-126.45
19F NMR (376 MHz, CDCl3)
F
O
O
N
H
Ph
Ph
C4
40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

^{19}F NMR (376 MHz, $CDCl_3$)

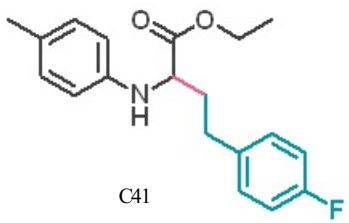


C28

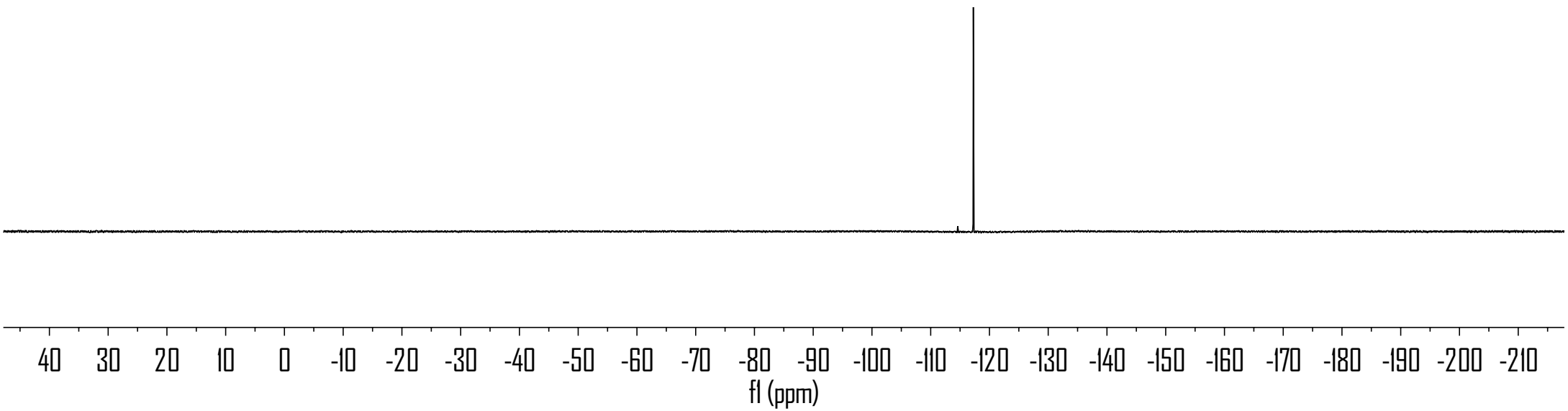
-116.22
-116.40

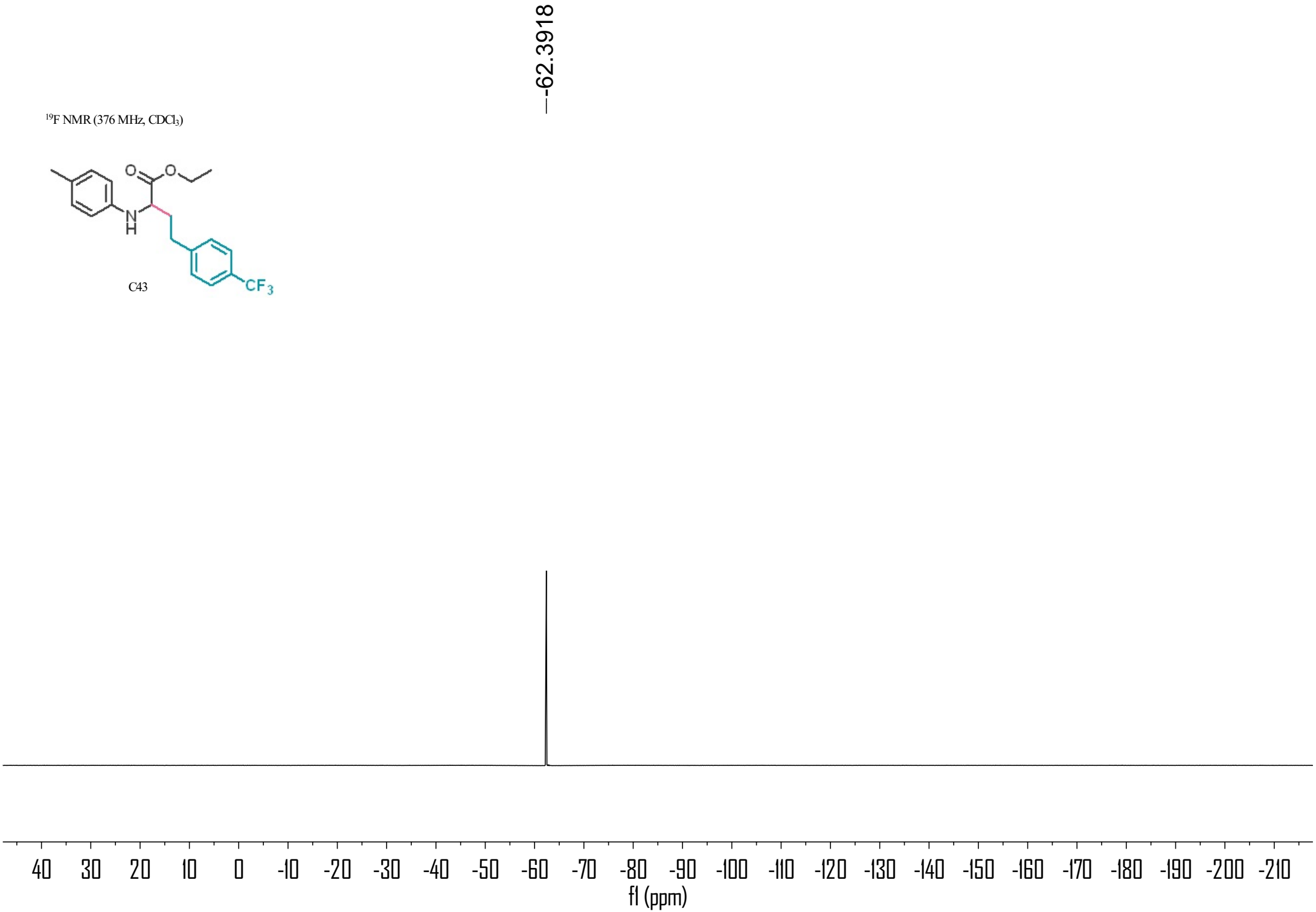


^{19}F NMR (376 MHz, $CDCl_3$)



C41




-62.3918
19F NMR (376 MHz, CDCl3)
O
O
N
H
CF3
C43
40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

X-Ray Diffraction Data of C8 (CCDC 2345482)

Table S6. Crystal data

Bond precision: C-C = 0.0018 A Wavelength=0.71073

Cell: a=11.3592(3) b=8.7279(2) c=21.5003(5)
alpha=90 beta=93.109(1) gamma=90
Temperature: 150 K

	Calculated	Reported
Volume	2128.45(9)	2128.44(9)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C25 H27 N O2	C25 H27 N O2
Sum formula	C25 H27 N O2	C25 H27 N O2
Mr	373.48	373.47
Dx,g cm-3	1.166	1.165
Z	4	4
Mu (mm-1)	0.073	0.073
F000	800.0	800.0
F000'	800.33	
h,k,lmax	14,11,27	14,11,27
Nref	4875	4819
Tmin,Tmax	0.991,0.994	0.710,0.748
Tmin'	0.987	

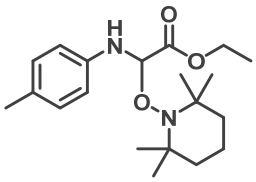
Correction method= # Reported T Limits: Tmin=0.710 Tmax=0.748
AbsCorr = NONE

Data completeness= 0.989 Theta(max)= 27.484

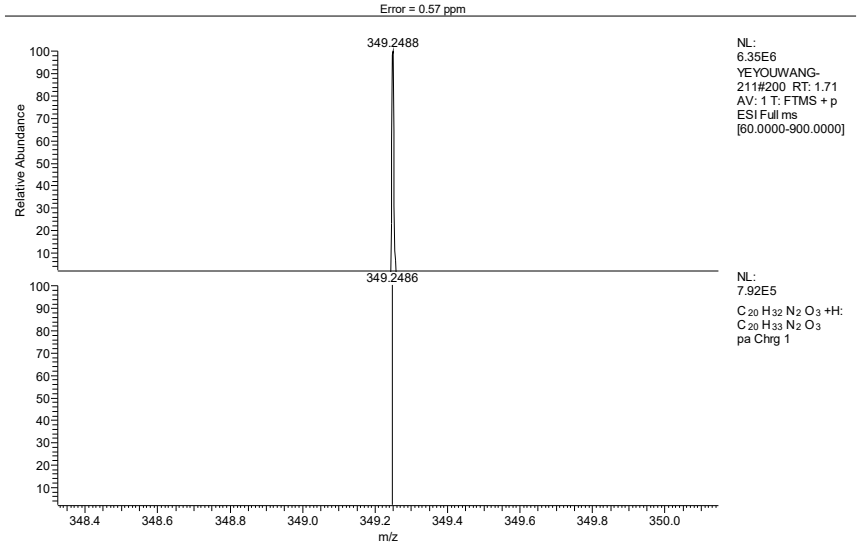
R(reflections)= 0.0480(4537) wR2(reflections)= 0.1265(4819)

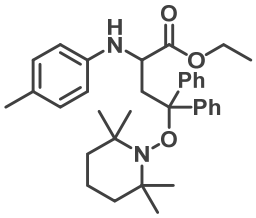
S = 1.037 Npar= 255

HRMS spectra of key intermediates

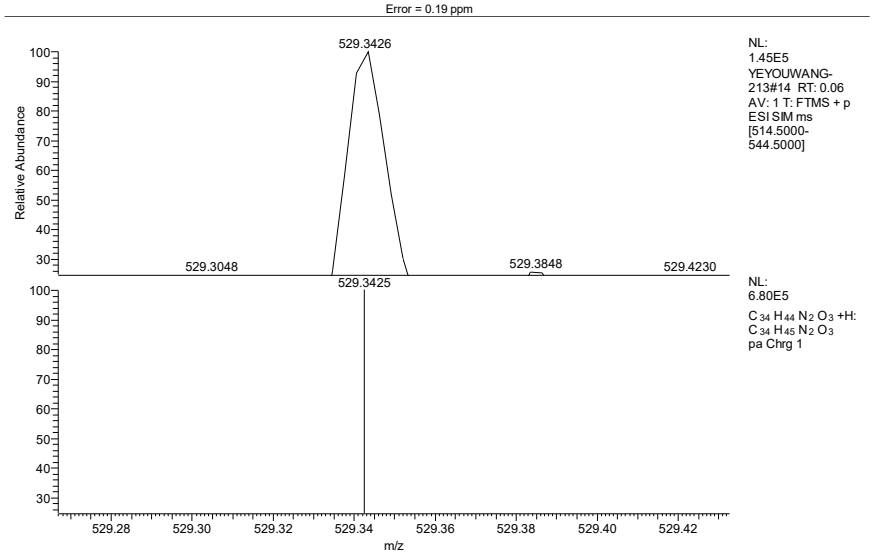


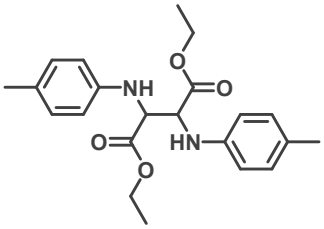
$C_{20}H_{33}N_2O_3^+$
$[M+H]^+$ m\z 349.2486
found 349.2488



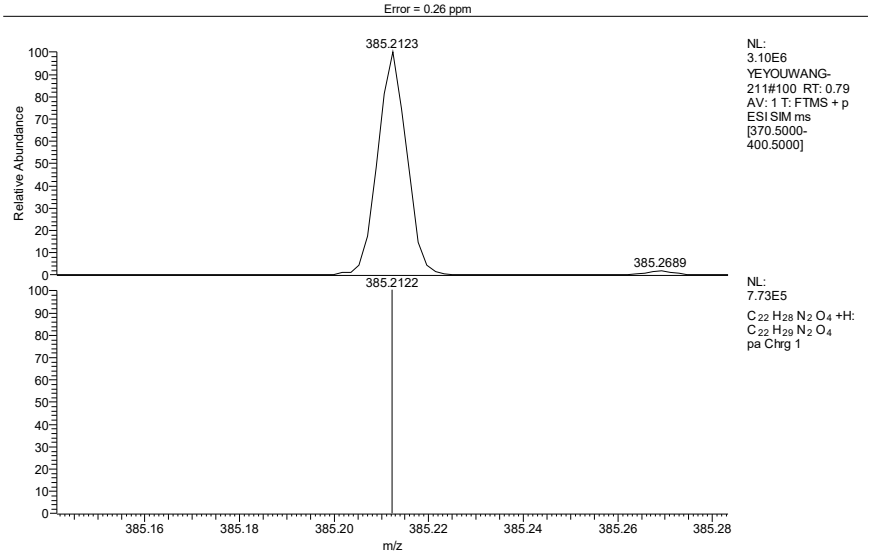


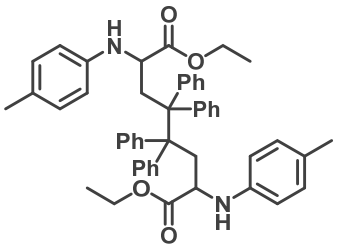
$C_{34}H_{45}N_2O_3^+$
$[M+H]^+$ m\z 529.3425
found 529.3426





$C_{22}H_{29}N_2O_4^+$
$[M+H]^+$ m\z 385.2122
found 385.2123





$C_{50}H_{53}N_2O_4^+$
$[M+H]^+$ m\z 745.4000
found 745.4006