

Supporting Information

For

Dehydrogenative Cyclization of *N*-Acyl Dipeptide Esters for the Synthesis of
Imidazolidin-4-ones

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(A) General Methods

Commercially available reagents were used as received without further purification unless otherwise indicated. Reactions were magnetically stirred and monitored by thin layer chromatography (TLC) using Silica Gel 60 F254 plates and were visualized by fluorescence quenching at 254 nm. For chromatographic purifications, analytically pure solvents were used and the silica gel 300-400 mesh was used as the solid support. ^1H NMR and ^{13}C NMR chemical shifts were reported in δ units, parts per million (ppm) relative to the chemical shift of residual solvent. Reference peaks for chloroform in ^1H NMR and ^{13}C NMR spectra were set at 7.26 ppm and 77.0 ppm, respectively.

(B) Analytical data for the compounds

Typical experimental procedure for the synthesis of ethyl 2-(2-benzamidoacetamido)-2-phenylacetate (1a-1r) ^[1]

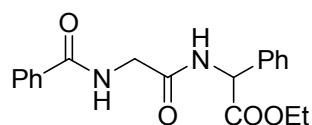
In a 25 mL dry round-bottom flask, mixture of 2-benzamidoacetic acid(0.895 g, 5 mmol), Phenylglycine ethyl ester-HCl(1.075g, 5mmol), EDCI^a (1.054 g 5.5 mmol), HOBT^b (0.743g 5.5 mmol), DIPEA^c (1.935g 15mmol) was stirred in dichloromethane(15 mL)at room temperature for 4h until complete consumption of starting material as monitored by TLC. After the reaction was finished, the mixture was quenched with water, then extracted with dichloromethane, dried over anhydrous Na_2SO_4 , and evaporated in vacuum. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate, 3/1) to give the product **1a**.

^aEDCI=1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide Hydrochloride

^bHOBT=NHydroxybenzotriazole

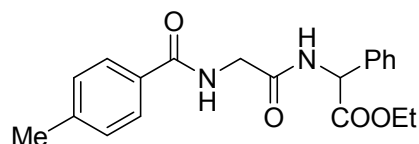
^cDIPEA=N,N-Diisopropylethylamine

Ethyl 2-(2-benzamidoacetamido)-2-phenylacetate(1a)



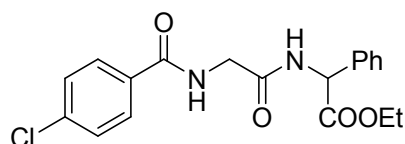
White solid, Mp. 113.2-114.0 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.89 (d, J = 7.0 Hz, 1H), 7.77 (d, J = 7.3 Hz, 2H), 7.46 (dt, J = 9.2, 6.0 Hz, 2H), 7.37 (dd, J = 8.5, 4.9 Hz, 4H), 7.33 – 7.27 (m, 3H), 5.56 (d, J = 7.0 Hz, 1H), 4.42 – 3.96 (m, 4H), 1.18 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 170.45, 168.77, 167.73, 136.05, 133.42, 131.64, 128.82, 128.40, 127.25, 127.14, 61.81, 56.76, 43.51, 13.86. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{19}\text{H}_{20}\text{N}_2\text{NaO}_4^+$: 363.1315 ($\text{M} + \text{Na}$) $^+$, found: 363.1314.

Ethyl 2-(2-(4-methylbenzamido)acetamido)-2-phenylacetate(1b)



White solid, Mp. 141.9-143.6 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.68 (d, J = 8.1 Hz, 2H), 7.47 (d, J = 6.7 Hz, 1H), 7.41 – 7.28 (m, 5H), 7.21 (d, J = 8.0 Hz, 2H), 7.07 (d, J = 3.8 Hz, 1H), 5.56 (d, J = 7.0 Hz, 1H), 4.34 – 4.04 (m, 4H), 2.39 (s, 3H), 1.20 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 170.45, 168.53, 167.65, 142.25, 136.14, 130.69, 129.20, 128.94, 128.55, 127.25, 127.14, 61.98, 56.76, 43.50, 21.43, 13.94. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{20}\text{H}_{23}\text{N}_2\text{NaO}_4^+$: 355.1652 ($\text{M} + \text{Na}$) $^+$, found: 355.1643.

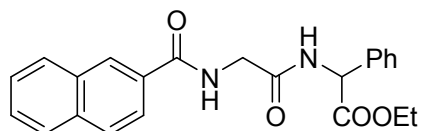
Ethyl 2-(2-(4-chlorobenzamido)acetamido)-2-phenylacetate(1c)



White solid, Mp. 154.8-156.6 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.72 (d, J = 8.5 Hz, 2H), 7.44 – 7.27 (m, 8H), 7.08 (s, 1H), 5.55 (d, J = 6.9 Hz, 1H), 4.30 – 4.10 (m,

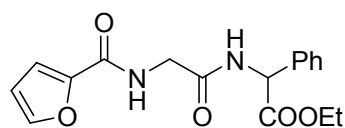
4H), 1.21 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 170.45, 168.20, 166.56, 138.09, 136.08, 131.90, 129.00, 128.83, 128.65, 128.58, 127.24, 62.08, 56.80, 43.42, 13.95$. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{19}\text{H}_{19}\text{ClN}_2\text{NaO}_4^+$: 397.0926 ($\text{M} + \text{Na}$) $^+$, found: 397.0919.

Ethyl 2-(2-(2-naphthamido)acetamido)-2-phenylacetate(1d)

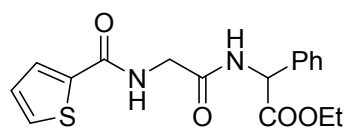


White solid, Mp. 133.3-135.8 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): $\delta = 8.33$ (s, 1H), 7.91 – 7.79 (m, 4H), 7.55 (ddd, $J = 19.8, 17.4, 6.8$ Hz, 3H), 7.39 (s, 3H), 7.32 (d, $J = 6.1$ Hz, 3H), 5.59 (d, $J = 7.0$ Hz, 1H), 4.38 – 4.10 (m, 4H), 1.19 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 170.48, 168.58, 167.77, 136.17, 134.87, 132.56, 130.72, 129.01, 128.95, 128.55, 128.40, 127.84, 127.73, 127.68, 127.26, 126.69, 123.62, 61.98, 56.83, 43.65, 13.93$. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{23}\text{H}_{23}\text{N}_2\text{NaO}_4^+$: 413.1472 ($\text{M} + \text{Na}$) $^+$, found: 413.1463.

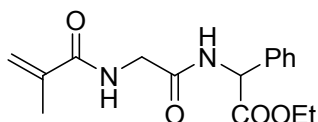
Ethyl 2-(2-(furan-2-carboxamido)acetamido)-2-phenylacetate(1e)



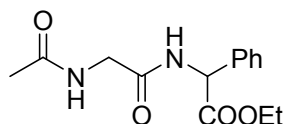
White solid, Mp. 110.8-111.5 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): $\delta = 7.44$ (s, 1H), 7.41 – 7.28 (m, 5H), 7.21 (d, $J = 6.7$ Hz, 1H), 7.11 (d, $J = 3.3$ Hz, 1H), 7.05 (s, 1H), 6.49 (dd, $J = 3.3, 1.6$ Hz, 1H), 5.57 (d, $J = 7.1$ Hz, 1H), 4.29 – 4.09 (m, 4H), 1.21 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 170.49, 168.03, 158.60, 147.28, 144.35, 136.15, 128.93, 128.53, 127.24, 114.82, 112.10, 62.00, 56.66, 42.65, 13.93$. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{NaO}_5^+$: 353.1108 ($\text{M} + \text{Na}$) $^+$, found: 353.1099.

Ethyl 2-phenyl-2-(2-(thiophene-2-carboxamido)acetamido)acetate(1f)

White solid, Mp. 141.8-145.2 °C; ¹H NMR (400 MHz, CDCl₃): δ= 7.55 (d, *J* = 2.7 Hz, 1H), 7.48 (d, *J* = 5.0 Hz, 1H), 7.34 (dd, *J* = 12.9, 5.4 Hz, 6H), 7.10 – 7.03 (m, 1H), 6.94 (s, 1H), 5.55 (d, *J* = 7.0 Hz, 1H), 4.28 – 4.12 (m, 4H), 1.20 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ= 170.43, 168.24, 162.25, 138.02, 136.16, 130.44, 128.98, 128.74, 128.59, 127.69, 127.23, 62.04, 56.78, 43.36, 13.94. HRMS (ESI-TOF) *m/z*: calcd for C₁₇H₁₈N₂NaO₄S⁺: 369.0879 (*M* + Na)⁺, found: 369.0874.

Ethyl 2-(2-methacrylamidoacetamido)-2-phenylacetate(1g)

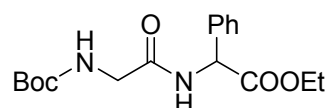
White solid, Mp. 93.3-95.1 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.46 – 7.28 (m, 6H), 6.70 (s, 1H), 5.76 (s, 1H), 5.54 (d, *J* = 7.0 Hz, 1H), 5.36 (s, 1H), 4.29 – 4.00 (m, 4H), 1.95 (s, 3H), 1.21 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ = 170.44, 168.54, 168.35, 139.05, 136.16, 128.93, 128.55, 127.22, 120.61, 61.98, 56.70, 43.20, 18.45, 13.94. HRMS (ESI-TOF) *m/z*: calcd for C₁₆H₂₀N₂NaO₄⁺: 327.1315 (*M* + Na)⁺, found: 327.1310.

Ethyl 2-(2-acetamidoacetamido)-2-phenylacetate (1h)

White solid, Mp. 134.2-136.7 °C; ¹H NMR (400 MHz, CDCl₃): δ= 7.44 (d, *J* = 6.9 Hz, 1H), 7.40 – 7.29 (m, 5H), 6.57 (s, 1H), 5.52 (d, *J* = 7.1 Hz, 1H), 4.28 – 4.08 (m, 2H), 3.99 (qd, *J* = 16.7, 5.1 Hz, 2H), 1.99 (s, 3H), 1.21 (t, *J* = 7.1 Hz, 3H); ¹³C NMR

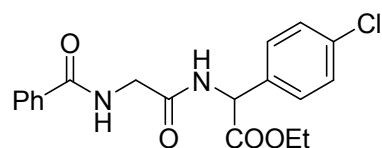
(101 MHz, CDCl₃): δ = 170.67, 170.47, 168.44, 136.08, 128.91, 128.54, 127.22, 61.96, 56.64, 43.07, 22.81, 13.94. HRMS (ESI-TOF) m/z : calcd for C₁₄H₁₈N₂NaO₄⁺: 301.1159 (M + Na)⁺, found: 301.1164.

Ethyl 2-(2-(tert-butoxycarbonylamino)acetamido)-2-phenylacetate(1i)



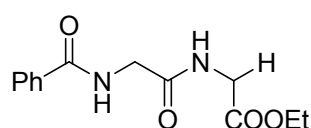
Yellow liquid, ¹H NMR (400 MHz, CDCl₃): δ = 7.50 – 7.28 (m, 6H), 5.52 (d, J = 29.5 Hz, 2H), 4.16 (ddt, J = 21.3, 14.2, 7.1 Hz, 2H), 3.94 – 3.63 (m, 2H), 1.43 (s, 9H), 1.20 (t, J = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ = 170.57, 168.93, 156.04, 136.28, 128.84, 128.42, 127.11, 80.19, 61.90, 56.30, 44.17, 28.19, 13.90.

Ethyl 2-(2-benzamidoacetamido)-2-(4-chlorophenyl)acetate(1j)



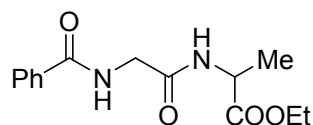
White solid, Mp. 166.3-167.5 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.78 (d, J = 7.3 Hz, 2H), 7.61 (d, J = 6.7 Hz, 1H), 7.51 (t, J = 7.4 Hz, 1H), 7.42 (t, J = 7.5 Hz, 2H), 7.36 – 7.27 (m, 4H), 7.11 (s, 1H), 5.53 (d, J = 6.8 Hz, 1H), 4.34 – 4.09 (m, 4H), 1.20 (t, J = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ = 170.07, 168.44, 167.79, 134.75, 134.54, 133.43, 131.89, 129.13, 128.64, 128.60, 127.11, 62.22, 56.17, 43.60, 13.94. HRMS (ESI-TOF) m/z : calcd for C₁₉H₁₉ClN₂NaO₄⁺: 397.0926 (M + Na)⁺, found: 397.0914.

Ethyl 2-(2-benzamidoacetamido)acetate (1k)^[2]



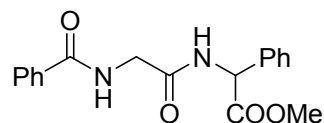
¹H NMR (400 MHz, CDCl₃): δ = 7.84 (d, *J* = 7.2 Hz, 2H), 7.57 – 7.47 (m, 2H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.30 (d, *J* = 5.0 Hz, 1H), 4.25 – 4.13 (m, 4H), 4.03 (d, *J* = 5.5 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ = 169.71, 169.63, 167.92, 133.35, 131.84, 128.54, 127.16, 77.32, 77.00, 76.68, 61.55, 43.52, 41.32, 14.04.

Ethyl 2-(2-benzamidoacetamido)propanoate (1k')^[2]



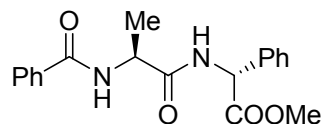
¹H NMR (400 MHz, CDCl₃): δ = 7.84 (d, *J* = 7.2 Hz, 2H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.33 (s, 1H), 7.15 (d, *J* = 7.0 Hz, 1H), 4.57 (p, *J* = 7.2 Hz, 1H), 4.27 – 4.14 (m, 4H), 1.43 (d, *J* = 7.2 Hz, 3H), 1.26 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ = 172.66, 168.64, 167.73, 133.49, 131.80, 128.55, 127.13, 77.32, 77.00, 76.68, 61.55, 48.32, 43.50, 18.09, 14.06.

Methyl 2-(2-benzamidoacetamido)-2-phenylacetate(1l)^[3]



¹H NMR (400 MHz, CDCl₃): δ = 7.78 (d, *J* = 7.4 Hz, 2H), 7.50 (d, *J* = 7.4 Hz, 2H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.39 – 7.29 (m, 5H), 7.12 (s, 1H), 5.58 (d, *J* = 6.9 Hz, 1H), 4.25 (qd, *J* = 16.9, 4.9 Hz, 2H), 3.72 (s, 3H); ¹³C NMR (101 MHz, CDCl₃): δ = 169.62, 169.39, 168.71, 136.05, 134.51, 131.13, 129.14, 128.62, 128.06, 127.57, 127.06, 79.81, 53.48, 50.83.

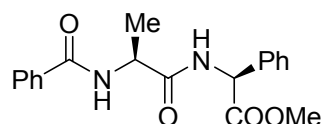
(L)-methyl 2-((D)-2-benzamidopropanamido)-2-phenylacetate(1m')^[4]



White solid, Mp. 151.9-154.4 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.80 (d, *J* = 7.2 Hz, 2H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.40 – 7.30 (m, 6H), 6.88 (d, *J* = 7.1 Hz, 1H), 5.55 (d, *J* = 7.0 Hz, 1H), 4.84 (p, *J* = 7.0 Hz, 1H), 3.69 (s, 3H), 1.48 (d,

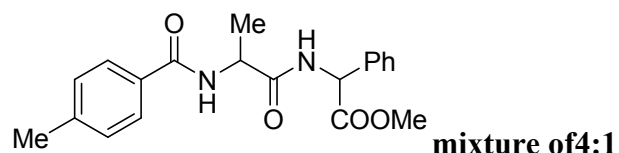
$J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 171.66, 170.94, 167.32, 136.04, 133.81, 131.79, 129.05, 128.65, 128.60, 127.18, 127.07, 56.66, 52.87, 48.91, 18.31$. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{19}\text{H}_{20}\text{N}_2\text{NaO}_4^+$: 363.1315 ($\text{M} + \text{Na}$) $^+$, found: 363.1324.

(L)-methyl 2-((L)-2-benzamidopropanamido)-2-phenylacetate(1m'')^[4]



^1H NMR (400 MHz, CDCl_3): $\delta = 7.73$ (d, $J = 7.1$ Hz, 2H), 7.49 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.5$ Hz, 3H), 7.37 – 7.33 (m, 2H), 7.32 – 7.27 (m, 3H), 6.86 (d, $J = 7.3$ Hz, 1H), 5.55 (d, $J = 7.1$ Hz, 1H), 4.85 (p, $J = 7.0$ Hz, 1H), 3.75 (s, 3H), 1.55 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 171.67, 170.93, 167.12, 135.94, 133.73, 131.74, 128.99, 128.62, 128.54, 127.22, 127.02, 77.32, 77.00, 76.68, 56.67, 52.87, 49.00, 18.61$.

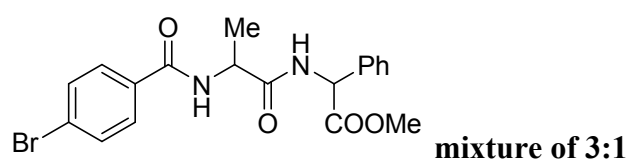
Methyl 2-(2-(4-methylbenzamido)propanamido)-2-phenylacetate(1n)



White solid, Mp. 143.1-144.3 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, $J = 7.2$ Hz, 0.2H)(minor), 7.86 (d, $J = 7.1$ Hz, 0.7H)(major), 7.68 (d, $J = 8.1$ Hz, 1.59H)(major), 7.63 (d, $J = 8.1$ Hz, 0.41H)(minor), 7.40 – 7.24 (m, 5H)(major/minor), 7.19 (d, $J = 8.1$ Hz, 2.28H)(major), 7.12 (d, $J = 7.5$ Hz, 0.79H)(minor), 5.56 (dd, $J = 7.1, 3.1$ Hz, 1H)(major/minor), 4.92 (p, $J = 7.1$ Hz, 1H)(major/minor), 3.72 (s, 0.64H)(minor), 3.65 (s, 2.39H)(major), 2.38 (s, 2.32H)(major), 2.37 (s, 0.69H)(minor), 1.51 (d, $J = 6.9$ Hz, 0.62H)(minor), 1.42 (d, $J = 7.0$ Hz, 2.38H)(major); ^{13}C NMR (101 MHz,

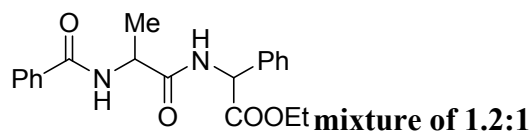
CDCl₃): δ = 172.10, 170.93 (major), 170.88 (minor), 167.19 (major), 167.07 (minor), 142.08 (major), 142.06 (minor), 136.06 (major), 135.94 (minor), 130.92 (major), 130.85 (minor), 129.11 (major), 129.07 (minor), 128.92 (major), 128.85 (minor), 128.49 (major), 128.44 (minor), 127.21 (major), 127.26 (minor), 127.11, 56.60 (major), 56.67 (minor), 52.70, 48.84, 21.39, 18.41 (major), 18.49 (minor). HRMS (ESI-TOF) m/z : calcd for C₂₀H₂₂N₂NaO₄⁺: 377.1472 (M + Na)⁺, found: 377.1479.

Methyl 2-(2-(4-bromobenzamido)propanamido)-2-phenylacetate(1o)



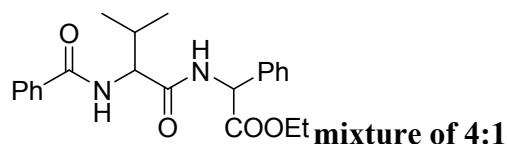
White solid, Mp. 159.9-160.7 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.73 – 7.57 (m, 3H)(major/minor), 7.51 (dd, J = 12.6, 8.6 Hz, 2H)(major/minor), 7.41 – 7.27 (m, 5H)(major/minor), 7.21 (d, J = 7.4 Hz, 0.21H)(minor), 7.17 (d, J = 7.3 Hz, 0.77H)(major), 5.54 (d, J = 7.0 Hz, 1H)(major/minor), 4.95 – 4.82 (m, 1H)(major/minor), 3.74 (s, 0.58H)(minor), 3.68 (s, 2.34H)(major), 1.51 (d, J = 7.0 Hz, 0.61H)(minor), 1.41 (d, J = 7.0 Hz, 2.44H)(major); ¹³C NMR (101 MHz, CDCl₃): δ = 171.82 (major), 171.75 (minor), 170.96 (major), 170.86 (minor), 166.26(major), 166.13 (minor), 135.96 (major), 135.90 (minor), 132.58 (major), 135.50 (minor), 131.73 (major), 131.70 (minor), 129.01 (major), 128.95 (minor), 128.75 (major), 128.73 (minor), 128.64 (major), 128.61 (minor), 127.18 (major), 127.23 (minor), 126.45, 56.65 (major), 56.69 (minor), 52.84, 48.97 (major), 49.01 (minor), 18.41 (major), 18.53 (minor). HRMS (ESI-TOF) m/z : calcd for C₁₉H₁₉BrN₂NaO₄⁺: 441.0420 (M + Na)⁺, found: 441.0425.

Ethyl 2-(2-benzamidopropanamido)-2-phenylacetate(1p)



White solid, Mp. 109.5-112.9 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.85 – 7.68 (m, 2H)(major/minor), 7.53 – 7.27 (m, 9H)(major/minor), 6.95 (d, J = 6.7 Hz, 1H)(major/minor), 5.54 (d, J = 7.1 Hz, 1H)(major/minor), 4.95 – 4.74 (m, 1H)(major/minor), 4.32 – 4.04 (m, 2H)(major/minor), 1.57 – 1.40 (m, 3H)(major/minor), 1.22 (t, J = 7.1 Hz, 1.75H)(major), 1.17 (t, J = 7.1 Hz, 1.45H)(minor); ^{13}C NMR (101 MHz, CDCl_3): δ = 171.85 (major), 171.88 (minor), 170.43 (major), 170.48 (minor), 167.14 (major), 167.25 (minor), 136.27, 136.16, 133.82 (major), 133.84 (minor), 131.72 (major), 131.75 (minor), 128.93 (major), 128.97 (minor), 128.53 (major), 128.57 (minor), 127.25 (major), 127.22 (minor), 127.11 (major), 127.15 (minor), 61.94, 56.82 (major), 56.78 (minor), 49.05 (major), 48.98 (minor), 18.70 (major), 18.46 (minor), 14.00 (major), 13.95 (minor). HRMS (ESI-TOF) m/z : calcd for $\text{C}_{20}\text{H}_{22}\text{N}_2\text{NaO}_4^+$: 377.1472 ($\text{M} + \text{Na}$) $^+$, found: 377.1468.

Ethyl 2-(2-benzamido-3-methylbutanamido)-2-phenylacetate(1q)



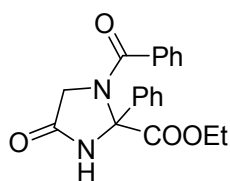
White solid, Mp. 172.3-173.6 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.80 (d, J = 7.2 Hz, 1.46H)(major), 7.75 (d, J = 8.4 Hz, 0.54H)(minor), 7.50 (q, J = 7.4 Hz, 1H)(major/minor), 7.46 – 7.27 (m, 7H)(major/minor), 7.22 (t, J = 6.7 Hz, 1H)(major/minor), 6.92 (d, J = 8.6 Hz, 0.26H)(minor), 6.87 (d, J = 8.5 Hz, 0.69H)(major), 5.53 (d, J = 6.8 Hz, 1H)(major/minor), 4.65 (dd, J = 15.5, 9.0 Hz, 1H)(major/minor), 4.30 – 4.07 (m, 2H)(major/minor), 2.21 (ddq, J = 26.9, 13.5, 6.8 Hz, 1H)(major/minor), 1.22 (t, J = 7.1 Hz, 0.91H)(minor), 1.17 (t, J = 7.1 Hz,

2.22H)(major/minor), 1.06 (t, $J = 7.1$ Hz, 1.64H)(minor), 0.91 (dd, $J = 6.8, 1.8$ Hz, 4.41H)(major); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 170.60$ (major), 170.65 (minor), 170.34 (major), 170.37 (minor), 167.41 (major), 167.32 (minor), 136.42, 135.87, 134.07 (major), 134.01 (minor), 131.68 (major), 131.65 (minor), 128.89 (major), 128.92 (minor), 128.56 (major), 128.50(minor), 127.24 (major), 127.27 (minor), 127.08, 62.00 (major), 61.91(minor), 58.29(major), 58.43 (minor), 56.72 (major), 56.85 (minor), 31.67 (major), 31.72 (minor), 19.17 (major), 19.15 (minor), 18.06 (major), 18.31(minor), 13.92 (major), 13.96 (minor).HRMS (ESI-TOF) m/z : calcd for $\text{C}_{22}\text{H}_{26}\text{N}_2\text{NaO}_4^+$: 405.1785 ($\text{M} + \text{Na}$) $^+$, found:405.1778.

Typical experimental procedure for the synthesis of ethyl 1-benzoyl-4-oxo-2-phenylimidazolidine-2-carboxylate (2a-2r)

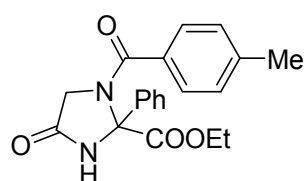
In a 15 mL reaction tube, a mixture of ethyl 2-(2-benzamidoacetamido)-2-phenylacetate(68 mg, 0.2 mmol), KI (16 mg, 0.1 mmol), K_2CO_3 (3 mg, 10% mmol), tert-Butyl hydroperoxide(70 % in decane, 0.3 mmol), in acetonitrile(1.5 mL) was stirred at 45°C under air for 3 h, until complete consumption of starting material as monitored by TLC. After the reaction was finished, the mixture was queched with saturate sodium thiosulfate solution, then extracted with EtOAc, dried over anhydrous Na_2SO_4 , and evaporated in vacuum. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate, 3/1) to give the product **2a**.

Ethyl 1-benzoyl-4-oxo-2-phenylimidazolidine-2-carboxylate(2a)



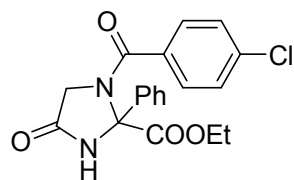
White solid, 92% yield, Mp. 183.3-185.6 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.66 (s, 2H), 7.57 – 7.29 (m, 9H), 4.48 – 4.34 (m, 1H), 4.34 – 4.22 (m, 2H), 4.10 (d, J = 15.7 Hz, 1H), 1.33 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 169.53, 169.25, 168.03, 136.05, 134.64, 131.07, 129.12, 128.63, 128.01, 127.72, 126.99, 79.73, 62.75, 50.85, 14.12. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{19}\text{H}_{18}\text{N}_2\text{NaO}_4^+$: 361.1159 ($M + \text{Na}$) $^+$, found: 361.1169.

Ethyl 1-(4-methylbenzoyl)-4-oxo-2-phenylimidazolidine-2-carboxylate(2b)



White solid, 84% yield, Mp. 195.3-197.6 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.86 (s, 1H), 7.64 (s, 2H), 7.36 (dd, J = 10.4, 5.5 Hz, 5H), 7.21 (d, J = 7.7 Hz, 2H), 4.43 – 4.06 (m, 4H), 2.38 (s, 3H), 1.30 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 169.67, 169.60, 168.15, 141.58, 136.11, 131.63, 129.20, 128.96, 127.92, 127.69, 127.17, 79.78, 62.60, 51.01, 21.42, 14.07. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{NaO}_4^+$: 375.1315 ($M + \text{Na}$) $^+$, found: 375.1311.

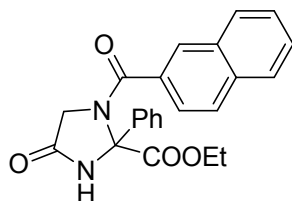
Ethyl 1-(4-chlorobenzoyl)-4-oxo-2-phenylimidazolidine-2-carboxylate(2c)



White solid, 67% yield, Mp. 220.5-223.6 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.48 (dd, J = 136.4, 29.7 Hz, 10H), 4.46 – 4.03 (m, 4H), 1.32 (t, J = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 169.09, 168.57, 167.94, 137.32, 135.92, 133.01, 129.27,

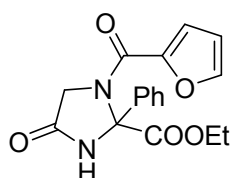
128.95, 128.55, 128.08, 127.76, 79.96, 62.88, 50.74, 14.14, -0.00. HRMS (ESI-TOF) m/z : calcd for $C_{19}H_{17}ClN_2NaO_4^+$: 395.0769 ($M + Na$)⁺, found: 395.0764.

Ethyl 1-(2-naphthoyl)-4-oxo-2-phenylimidazolidine-2-carboxylate(2d)



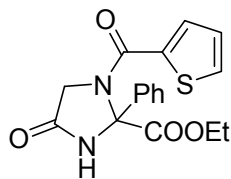
White solid, 50% yield, Mp. 198.1-200.6 °C; ¹H NMR (400 MHz, CDCl₃): δ= 7.96 (s, 1H), 7.86 (d, J = 7.0 Hz, 3H), 7.71 (d, J = 16.8 Hz, 3H), 7.55 (dt, J = 13.3, 6.5 Hz, 3H), 7.35 (s, 3H), 4.48 – 4.11 (m, 4H), 1.34 (t, J = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ= 169.69, 169.48, 168.16, 136.17, 134.29, 132.47, 131.84, 129.14, 128.65, 128.04, 127.86, 127.81, 127.51, 127.03, 123.62, 79.98, 62.80, 51.03, 14.17, -0.00. HRMS (ESI-TOF) m/z : calcd for $C_{23}H_{20}N_2NaO_4^+$: 411.1315 ($M + Na$)⁺, found: 411.1321.

Ethyl 1-(furan-2-carbonyl)-4-oxo-2-phenylimidazolidine-2-carboxylate(2e)



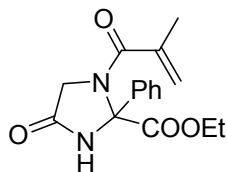
White solid, 50% yield, Mp. 192.3-195.1 °C; ¹H NMR (400 MHz, CDCl₃): δ= 7.64 (dd, J = 7.5, 1.7 Hz, 3H), 7.54 (s, 1H), 7.41 – 7.29 (m, 3H), 7.18 (d, J = 3.3 Hz, 1H), 6.51 (dd, J = 3.4, 1.6 Hz, 1H), 4.66 (dd, J = 41.4, 16.1 Hz, 2H), 4.44 – 4.23 (m, 2H), 1.28 (t, J = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ= 170.04, 167.77, 156.90, 147.05, 145.16, 136.10, 128.99, 127.88, 127.84, 118.52, 112.03, 80.18, 62.74, 49.60, 13.98. HRMS (ESI-TOF) m/z : calcd for $C_{17}H_{16}N_2NaO_5^+$: 351.0951 ($M + Na$)⁺, found: 351.0952.

Ethyl 4-oxo-2-phenyl-1-(thiophene-2-carbonyl)imidazolidine-2-carboxylate(2f)



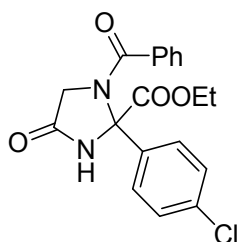
White solid, 70% yield, Mp. 205.6-208.4 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.83 (s, 1H), 7.65 (dd, J = 7.7, 1.8 Hz, 2H), 7.56 (dd, J = 5.9, 4.6 Hz, 2H), 7.35 (dd, J = 9.2, 3.1 Hz, 3H), 7.17 – 7.07 (m, 1H), 4.58 – 4.48 (m, 2H), 4.42 – 4.20 (m, 2H), 1.27 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 169.44, 167.82, 161.32, 136.73, 135.95, 131.53, 131.17, 128.99, 127.89, 127.83, 127.65, 80.29, 62.71, 50.42, 14.01. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{17}\text{H}_{16}\text{N}_2\text{NaO}_4\text{S}^+$: 367.0723 ($\text{M} + \text{Na}$) $^+$, found: 367.0723.

Ethyl 1-methacryloyl-4-oxo-2-phenylimidazolidine-2-carboxylate(2g)



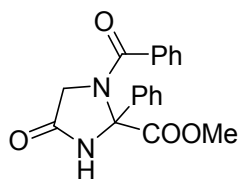
White solid, 85% yield, Mp. 162.7-165.4 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.86 (s, 1H), 7.64 – 7.52 (m, 2H), 7.49 – 7.29 (m, 4H), 5.28 (d, J = 60.0 Hz, 2H), 4.40 – 4.15 (m, 4H), 1.90 (s, 3H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 170.13, 169.58, 167.97, 139.84, 136.17, 129.07, 128.00, 127.67, 118.38, 79.51, 62.75, 50.21, 19.28, 14.05. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{16}\text{H}_{18}\text{N}_2\text{NaO}_4^+$: 325.1159 ($\text{M} + \text{Na}$) $^+$, found: 325.1156.

Ethyl 1-benzoyl-2-(4-chlorophenyl)-4-oxoimidazolidine-2-carboxylate(2j)



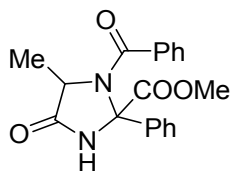
White solid, 81% yield, Mp. 234.5-237.9 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.92 (s, 1H), 7.61 (d, J = 6.9 Hz, 2H), 7.54 – 7.38 (m, 5H), 7.33 (d, J = 8.5 Hz, 2H), 4.38 (dt, J = 14.3, 7.1 Hz, 1H), 4.26 (d, J = 16.2 Hz, 2H), 4.08 (d, J = 15.5 Hz, 1H), 1.32 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 169.65, 169.42, 167.86, 135.16, 134.58, 134.31, 131.30, 129.35, 128.71, 128.15, 126.98, 79.30, 62.89, 50.84, 14.09. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{19}\text{H}_{17}\text{ClN}_2\text{NaO}_4^+$: 395.0769 ($\text{M} + \text{Na}$) $^+$, found: 395.0760.

Methyl 1-benzoyl-4-oxo-2-phenylimidazolidine-2-carboxylate(2l)



White solid, 78% yield, Mp. 163.5-167.8 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.66 (d, J = 20.2 Hz, 3H), 7.55 – 7.29 (m, 8H), 4.19 (dd, J = 65.2, 15.6 Hz, 2H), 3.87 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ = 169.62, 169.39, 168.71, 136.05, 134.51, 131.13, 129.14, 128.62, 128.06, 127.57, 127.06, 79.81, 53.48, 50.83. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{NaO}_4^+$: 347.1002 ($\text{M} + \text{Na}$) $^+$, found: 347.0998.

Methyl 1-benzoyl-5-methyl-4-oxo-2-phenylimidazolidine-2-carboxylate (2m)^[4]

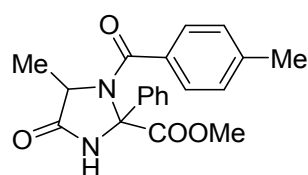


dr = 3:1

White solid, cis:trans = 3:1, 73% yield, Mp. 185.6-187.8 °C; ^1H NMR (400 MHz, CDCl_3): δ = 8.03 (s, 0.25H)(minor), 7.72 (s, 0.8H)(major), 7.68 – 7.27 (m,

8H)(major/minor), 4.58 (d, $J = 5.9$ Hz, 0.71H)(major), 4.41 (d, $J = 6.8$ Hz, 0.25H)(minor), 3.89 (s, 2.23H)(major), 3.85 (s, 0.74H)(minor), 1.23 (d, $J = 5.6$ Hz, 2.25H)(major), 1.12 (d, $J = 6.7$ Hz, 0.8H)(minor); ^{13}C NMR (101 MHz, CDCl_3): $\delta =$ 172.83(major/minor), 168.72(major/minor), 168.66(major/minor), 137.05(minor), 136.40(major), 135.5(minor), 135.15(major), 130.97(major), 130.01(minor), 129.02(major), 128.96(minor), 128.65(major), 128.02(minor), 127.85(major), 127.82(minor), 127.45(minor), 126.77(major), 125.89(minor), 79.32(major/minor), 60.38(major), 56.53(major), 55.85(minor), 53.63(minor), 53.42(major), 18.86(minor), 17.81(major). HRMS (ESI-TOF) m/z : calcd for $\text{C}_{19}\text{H}_{18}\text{N}_2\text{NaO}_4^+$: 361.1159 ($\text{M} + \text{Na}$) $^+$, found: 361.1150.

Methyl 5-methyl-1-(4-methylbenzoyl)-4-oxo-2-phenylimidazolidine-2-carboxylate (2n)

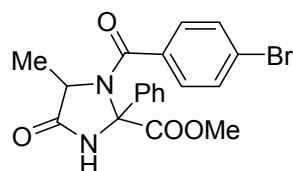


dr = 4.5:1

White solid, cis:trans = 4.5:1, 79% yield, Mp. 213.0-217.4 °C; ^1H NMR (400 MHz, CDCl_3): $\delta =$ 7.61 (s, 2H)(major/minor), 7.40 (dd, $J = 21.9, 18.5$ Hz, 5H)(major/minor), 7.18 (d, $J = 7.6$ Hz, 2H)(major/minor), 4.60 (q, $J = 6.6$ Hz, 0.77H)(major), 4.44 (q, $J = 6.8$ Hz, 0.19H)(minor), 3.89 (s, 2.3H)(major), 3.85 (s, 0.53H)(minor), 2.38 (s, 0.51H)(minor), 2.37 (s, 2.27H)(major), 1.25 (d, $J = 7.1$ Hz, 2.67H)(major), 1.14 (d, $J = 7.3$ Hz, 0.48H)(minor); ^{13}C NMR (101 MHz, CDCl_3): $\delta =$ 172.97(major/minor), 172.93(major), 168.86(minor), 168.80(major), 168.71(minor), 141.44(major), 140.20(minor), 137.20(minor), 136.54(major), 132.69(minor), 132.31(major), 129.29(major), 129.21(minor), 128.96(major), 128.91(minor), 127.99(minor), 127.83(major), 127.48(minor), 126.88(major), 125.94(minor), 79.33(minor), 79.27(major), 56.61(major), 55.85(minor), 53.58(minor), 53.36(major), 21.44(major),

21.39(minor), 18.93(minor), 17.84(major). HRMS (ESI-TOF) m/z: calcd for $C_{20}H_{20}N_2NaO_4^+$: 375.1315 (M + Na)⁺, found: 375.1310.

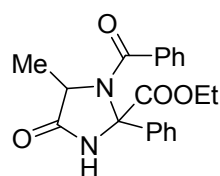
Methyl 1-(4-bromobenzoyl)-5-methyl-4-oxo-2-phenylimidazolidine-2-carboxylate (2o)



dr = 2.5:1

White solid, cis:trans = 2.5:1, 74% yield, Mp. 233.2-235.9 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.50 (s, 4H)(major/minor), 7.42 – 7.01 (m, 6H)(major/minor), 4.57 (s, 0.68H)(major), 4.39 (d, *J* = 6.1 Hz, 0.34H)(minor), 3.90 (s, 2.01H)(major), 3.87 (s, 0.84H)(minor), 1.28 (s, 2.14H)(major), 1.20 (s, 0.98H)(minor); ¹³C NMR (101 MHz, CDCl₃): δ = 172.46(major/minor), 172.40(major/minor), 168.64(major), 168.53(minor), 136.89(minor), 136.23(major), 134.41(minor), 134.09(minor), 131.88(major), 129.22(major), 129.17(minor), 128.38(major), 128.11(minor), 127.95(major), 127.81(major), 127.49(minor), 79.52(minor), 79.44(major), 60.38(major), 56.38(major), 55.71(minor), 53.71(minor), 53.50(major), 21.00(minor). HRMS (ESI-TOF) m/z: calcd for $C_{19}H_{17}BrN_2NaO_4^+$: 439.0264 (M + Na)⁺, found: 439.0262.

Ethyl 1-benzoyl-5-methyl-4-oxo-2-phenylimidazolidine-2-carboxylate (2p)

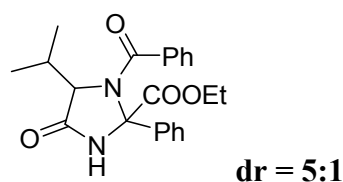


dr = 2:1

White solid, cis:trans = 2:1, 83% yield, Mp. 197.3-200.6 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.90 – 7.28 (m, 11H)(major/minor), 4.60 (d, *J* = 5.5 Hz, 0.63H)(minor), 4.49 – 4.26 (m, 2.43H)(major), 1.35 (t, *J* = 8.6, 7.1 Hz, 2.04H)(major), 1.32 (t, *J* = 7.1

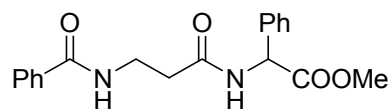
Hz, 1.17H)(minor), 1.25 (s, 2.20H)(major), 1.14 (s, 1.08H)(minor); ^{13}C NMR (101 MHz, CDCl_3): δ = 172.67(major/minor), 168.02(major/minor), 167.99(major/minor), 137.10(minor), 136.50(major), 135.69(minor), 135.31(major), 135.28(minor), 129.97(major), 129.03(major), 128.98(minor), 128.67(major/minor), 128.01(minor), 127.93(major), 127.85(major), 127.58(minor), 126.72(major), 125.90(minor), 79.19(major/minor), 62.98(minor), 62.72(major), 56.44(major), 55.82(minor), 18.90(minor), 17.82(major), 14.07(major), 14.04(minor).HRMS (ESI-TOF) m/z : calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{NaO}_4^+$: 375.1315 ($\text{M} + \text{Na}$) $^+$, found: 375.1312.

Ethyl 1-benzoyl-5-isopropyl-4-oxo-2-phenylimidazolidine-2-carboxylate (2q)



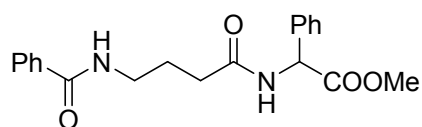
White solid, cis:trans = 5:1, 80% yield, Mp. 233.6-236.4 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.40 (dd, J = 73.7, 55.7 Hz, 1H)(major/minor), 4.41 (d, J = 3.5 Hz, 1H)(major/minor), 4.28 (tdd, J = 10.7, 7.1, 3.6 Hz, 2H)(major/minor), 1.35 (dd, J = 12.1, 4.9 Hz, 0.58H)(minor), 1.32 – 1.22 (m, 3.34H)(major), 0.99 (d, J = 21.8 Hz, 1H)(minor), 0.74 (d, J = 46.6 Hz, 5H)(major); ^{13}C NMR (101 MHz, CDCl_3): δ = 171.70(major/minor), 171.63(major/minor), 168.71(minor), 168.64(major), 137.55(minor), 136.71(major), 135.84(major), 129.83(major), 129.00(minor), 128.77(major/minor), 128.54(major), 128.23(minor), 128.00(minor), 127.99(major), 127.96(minor), 127.74(major), 127.32(minor), 126.41(minor), 125.95(major), 79.92(major), 79.89(minor), 64.52(major), 62.97(major), 62.65(minor), 60.37(minor), 19.36(minor), 18.87(major), 16.52(minor), 16.47(major), 14.14(minor), 14.03(major).HRMS (ESI-TOF) m/z : calcd for $\text{C}_{22}\text{H}_{26}\text{N}_2\text{NaO}_4^+$: 403.1622 ($\text{M} + \text{Na}$) $^+$, found: 403.1628.

Methyl 2-(3-benzamidopropanamido)-2-phenylacetate (3a)



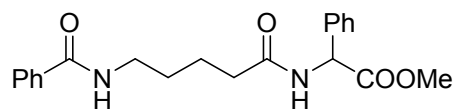
White solid, Mp. 132.6-135.1 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.74 (d, J = 7.2 Hz, 2H), 7.48 (t, J = 7.4 Hz, 1H), 7.39 (t, J = 8.0 Hz, 2H), 7.30 (s, 5H), 7.20 (t, J = 5.3 Hz, 1H), 6.97 (d, J = 6.8 Hz, 1H), 5.53 (d, J = 6.9 Hz, 1H), 3.75 – 3.64 (m, 5H), 2.58 (dt, J = 6.6, 4.6 Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ = 171.43, 171.21, 167.46, 135.93, 134.24, 131.38, 128.98, 128.62, 128.42, 127.22, 126.99, 56.64, 52.81, 35.93, 35.27. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{19}\text{H}_{20}\text{N}_2\text{NaO}_4^+$: 363.1315 ($\text{M} + \text{Na}$) $^+$, found: 363.1305.

Methyl 2-(4-benzamidobutanamido)-2-phenylacetate (3b)



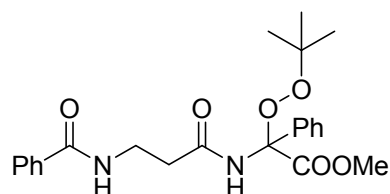
White solid, Mp. 140.9-143.5 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.77 (d, J = 7.1 Hz, 2H), 7.49 (t, J = 7.3 Hz, 1H), 7.41 (t, J = 7.4 Hz, 2H), 7.33 (dd, J = 9.1, 3.9 Hz, 5H), 6.95 (d, J = 6.7 Hz, 2H), 5.55 (d, J = 7.1 Hz, 1H), 3.71 (s, 3H), 3.62 – 3.46 (m, 2H), 2.41 (q, J = 6.6 Hz, 2H), 1.98 (dd, J = 12.8, 6.4 Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ = 172.55, 171.34, 167.66, 136.16, 134.33, 131.32, 129.01, 128.62, 128.47, 127.28, 126.93, 56.63, 52.81, 39.67, 33.80, 24.77. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{20}\text{H}_{22}\text{N}_2\text{NaO}_4^+$: 377.1472 ($\text{M} + \text{Na}$) $^+$, found: 377.1460.

Methyl 2-(5-benzamidopentanamido)-2-phenylacetate (3c)



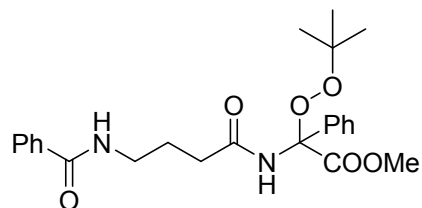
White solid, Mp. 139.8-140.9 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.77 (d, J = 7.1 Hz, 2H), 7.48 (t, J = 7.3 Hz, 1H), 7.40 (t, J = 7.4 Hz, 2H), 7.37 – 7.28 (m, 5H), 6.77 – 6.62 (m, 2H), 5.55 (d, J = 7.1 Hz, 1H), 3.71 (s, 3H), 3.43 (ddd, J = 12.5, 6.5, 3.4 Hz, 2H), 2.33 (td, J = 7.1, 3.5 Hz, 2H), 1.74 (dq, J = 13.4, 6.8 Hz, 2H), 1.64 (dd, J = 13.2, 6.6 Hz, 2H); ^{13}C NMR (101 MHz, CDCl_3): δ = 172.44, 171.40, 167.56, 136.29, 134.57, 131.26, 128.96, 128.56, 128.43, 127.29, 126.92, 56.49, 52.74, 39.06, 35.23, 28.67, 22.24. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{21}\text{H}_{24}\text{N}_2\text{NaO}_4^+$: 391.1628 ($\text{M} + \text{Na}$) $^+$, found: 391.1619.

Methyl 2-(3-benzamidopropanamido)-2-(tert-butylperoxy)-2-phenylacetate (4a)



White solid, 35% yield, Mp. 175.5-178.3 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.70 (d, J = 7.3 Hz, 2H), 7.53 (s, 1H), 7.51 – 7.44 (m, 3H), 7.41 – 7.29 (m, 5H), 7.08 (s, 1H), 3.81 – 3.62 (m, 5H), 2.68 (t, J = 5.6 Hz, 2H), 1.22 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3): δ = 170.81, 169.12, 167.25, 135.44, 134.28, 131.35, 128.79, 128.43, 128.38, 126.95, 125.62, 89.72, 81.36, 53.46, 35.68, 35.36, 26.44. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{23}\text{H}_{28}\text{N}_2\text{NaO}_6^+$: 451.1840 ($\text{M} + \text{Na}$) $^+$, found: 451.1847.

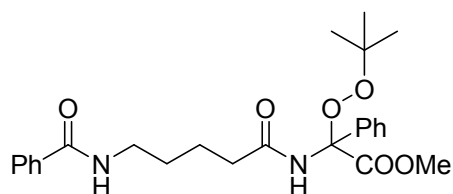
Methyl 2-(4-benzamidobutanamido)-2-(tert-butylperoxy)-2-phenylacetate (4b)



White solid, 42% yield, Mp. 157.5-158.2 °C; ^1H NMR (400 MHz, CDCl_3): δ = 7.69 (d, J = 7.4 Hz, 2H), 7.63 (s, 1H), 7.49 (d, J = 7.9 Hz, 2H), 7.43 (d, J = 7.4 Hz, 1H),

7.37 – 7.27 (m, 5H), 7.14 (s, 1H), 3.71 (s, 3H), 3.48 (dd, $J = 11.8, 6.1$ Hz, 2H), 2.49 (td, $J = 6.2, 2.6$ Hz, 2H), 1.95 (dd, $J = 7.2, 5.4$ Hz, 2H), 1.26 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 172.00, 169.19, 167.48, 135.60, 134.27, 131.10, 128.69, 128.35, 128.30, 126.96, 125.62, 89.68, 81.32, 53.36, 39.89, 34.65, 26.49, 23.86$. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{24}\text{H}_{30}\text{N}_2\text{NaO}_6^+$: 465.1996 ($\text{M} + \text{Na}$) $^+$, found: 465.1992.

Methyl 2-(5-benzamidopentanamido)-2-(tert-butylperoxy)-2-phenylacetate (4c)



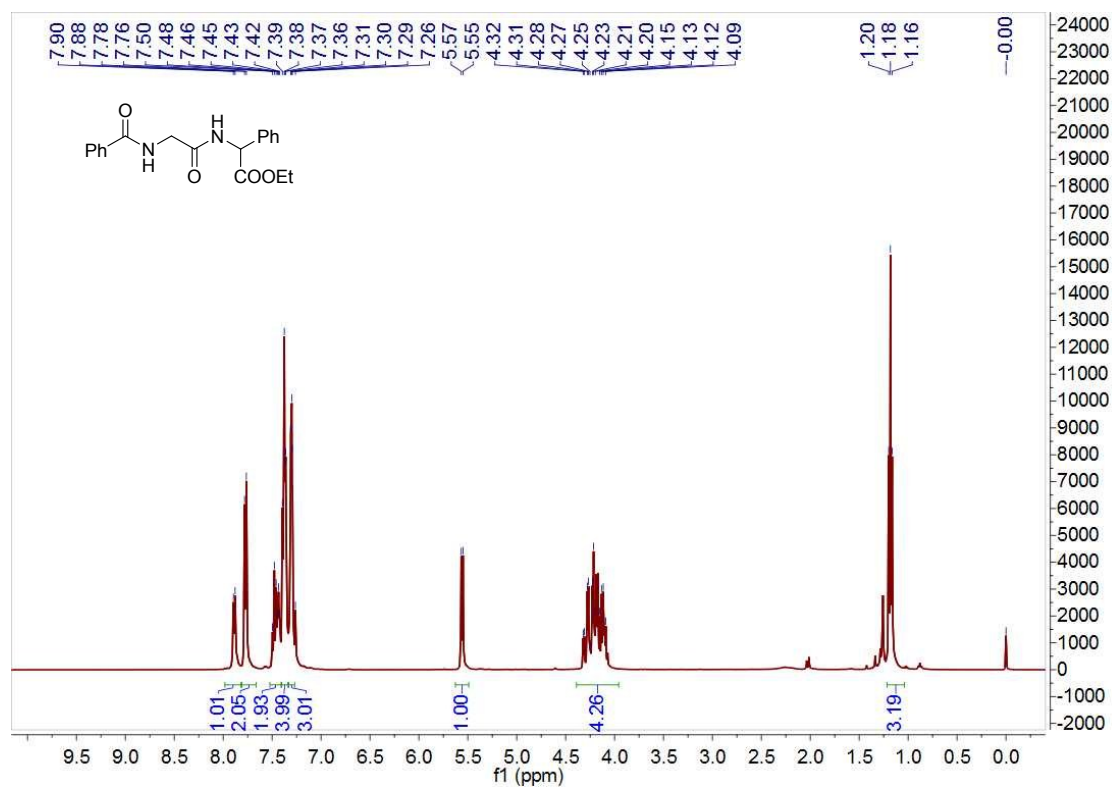
White solid, 53% yield, Mp. 154.3-156.7°C; ^1H NMR (400 MHz, CDCl_3): $\delta = 7.73$ (d, $J = 7.3$ Hz, 2H), 7.53 – 7.28 (m, 10H), 6.71 (s, 1H), 3.71 (s, 3H), 3.43 (q, $J = 6.1$ Hz, 2H), 2.39 (t, $J = 6.6$ Hz, 2H), 1.70 (dt, $J = 30.1, 6.4$ Hz, 4H), 1.26 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3): $\delta = 171.73, 169.27, 167.46, 135.77, 134.50, 131.14, 128.64, 128.36, 128.27, 126.94, 125.67, 89.59, 81.24, 53.33, 39.07, 35.92, 28.51, 26.51, 21.65$. HRMS (ESI-TOF) m/z : calcd for $\text{C}_{25}\text{H}_{32}\text{N}_2\text{NaO}_6^+$: 479.2153 ($\text{M} + \text{Na}$) $^+$, found: 479.2136.

(C) References

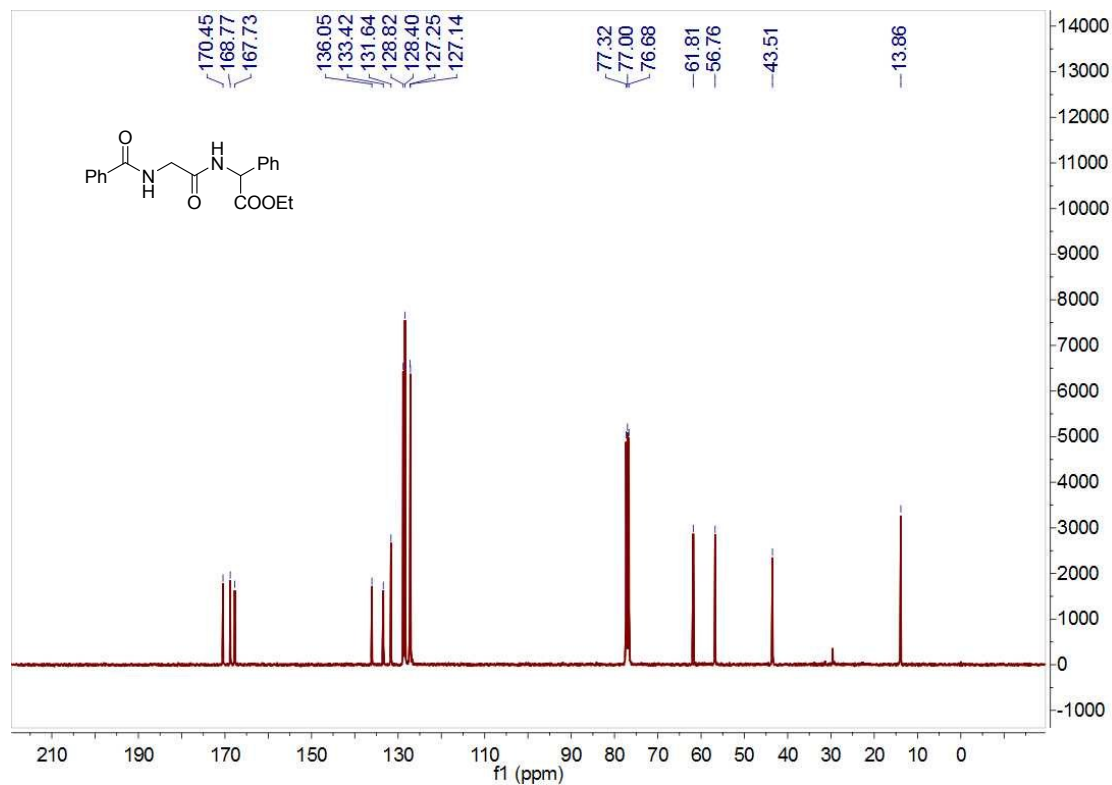
- [1] G. He, Y. s. Zhao, S. y. Zhang, C. x. Lu, and G. Chen, *J. Am. Chem. Soc.* **2012**, 134, 3
- [2] S. Oskar, J. Liebigs *Annalen der Chemie* **1951**, 572, 96
- [3] A. Ghaffar, S. A. *Al-Azhar Bulletin of Science.* **1991**, 2, 415
- [4] A. Papadopoulos, B. Lewalla. E. Steckhan, K. D. Ginzel, F. Knoch, M. Nieger, *Tetrahedron.* **1991**, 47, 563

(D) Spectra

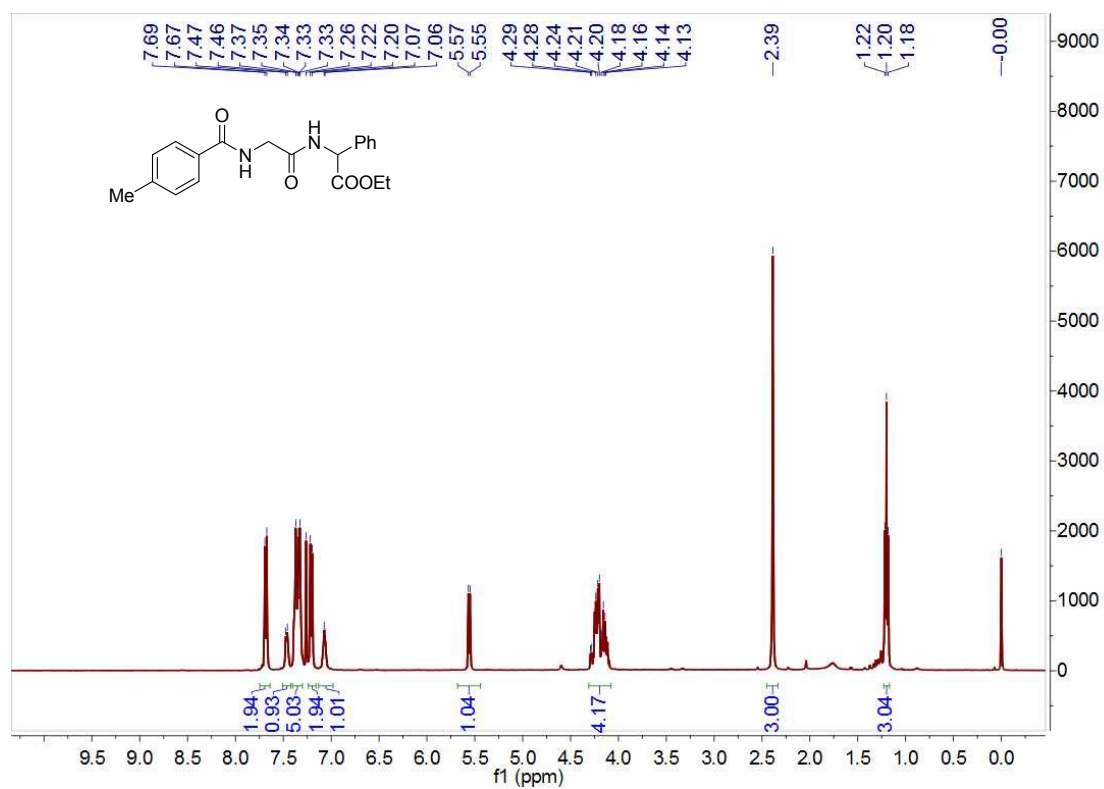
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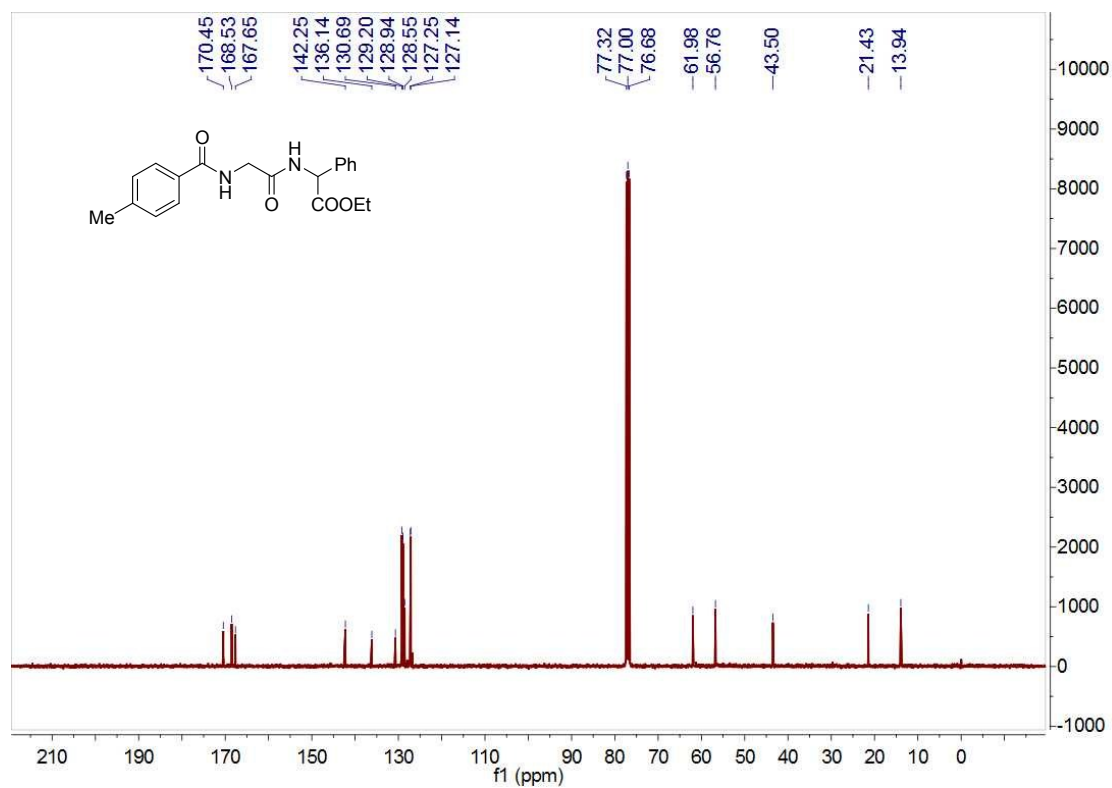
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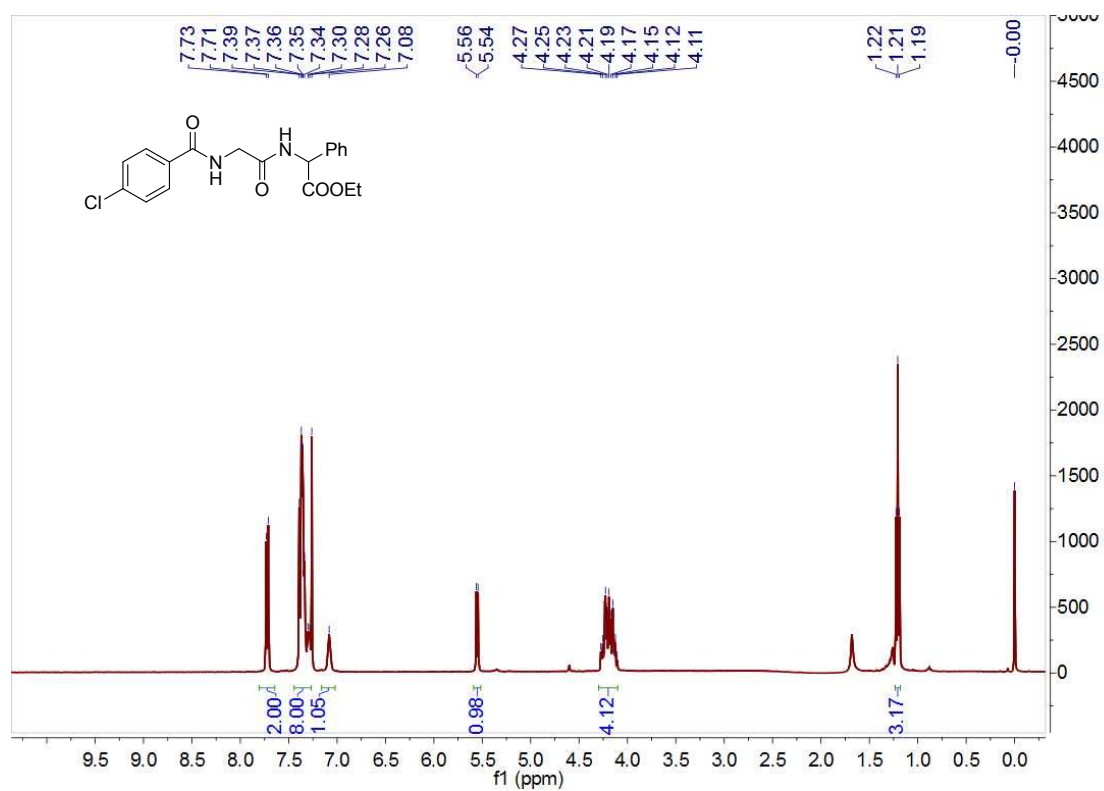
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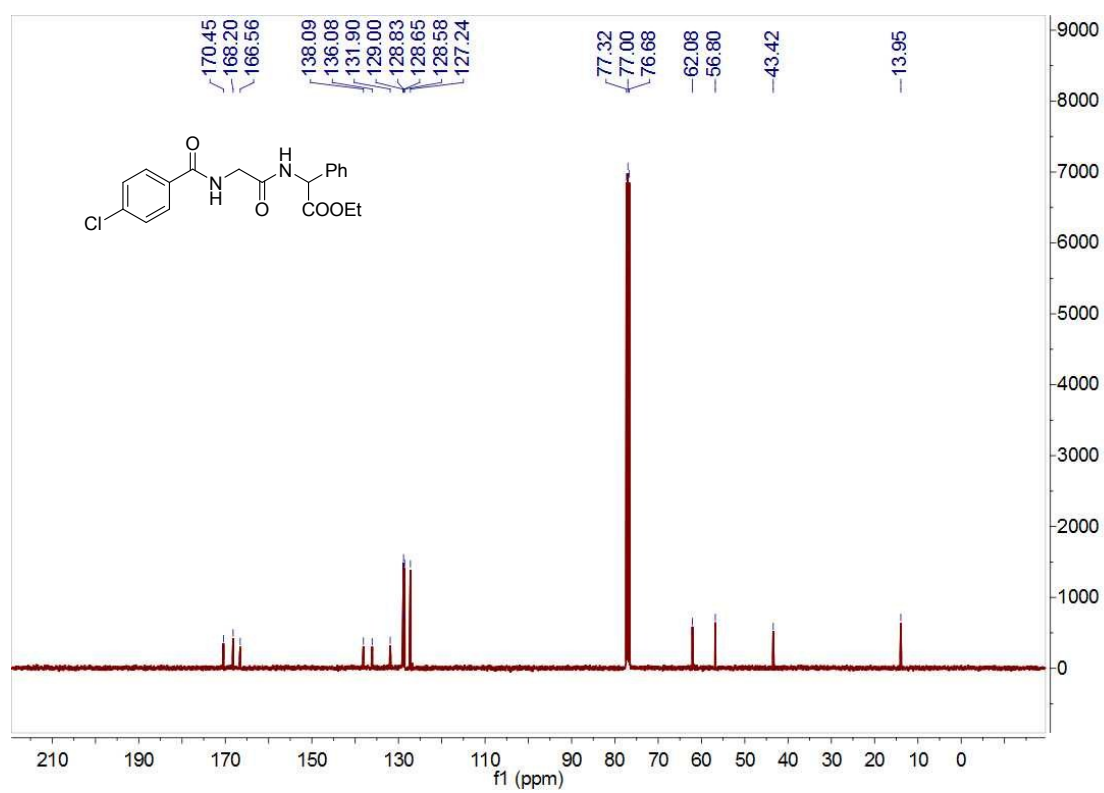
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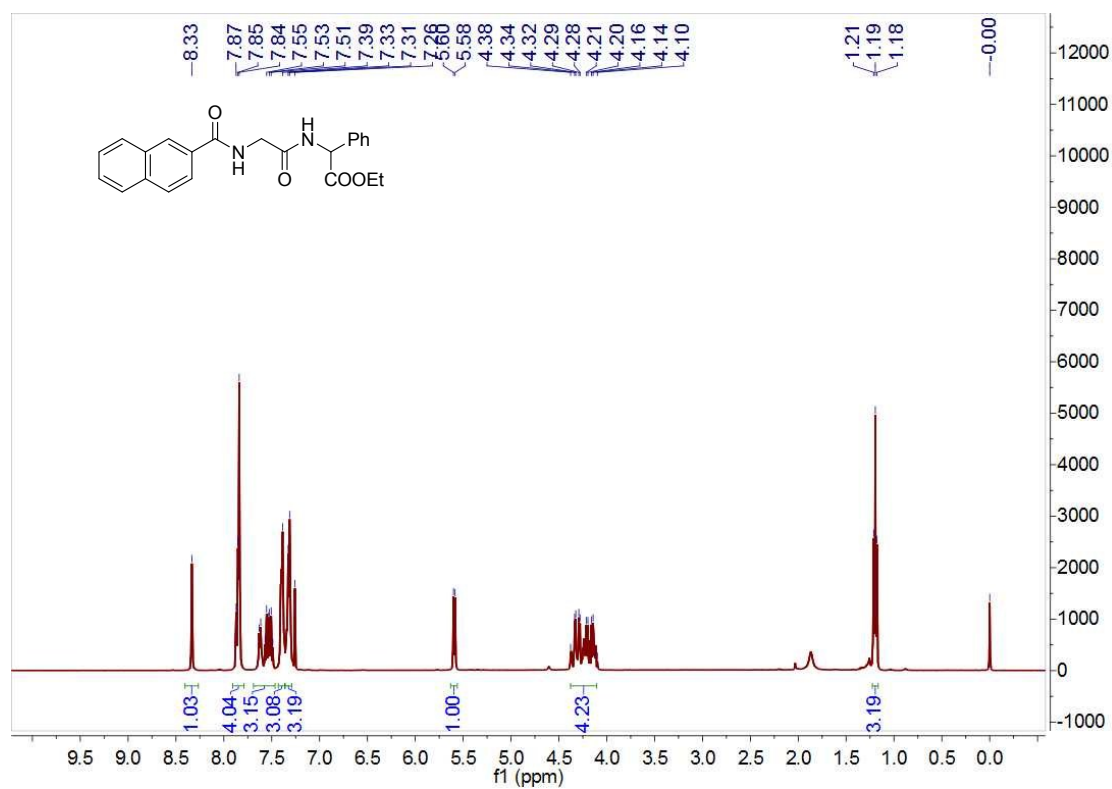
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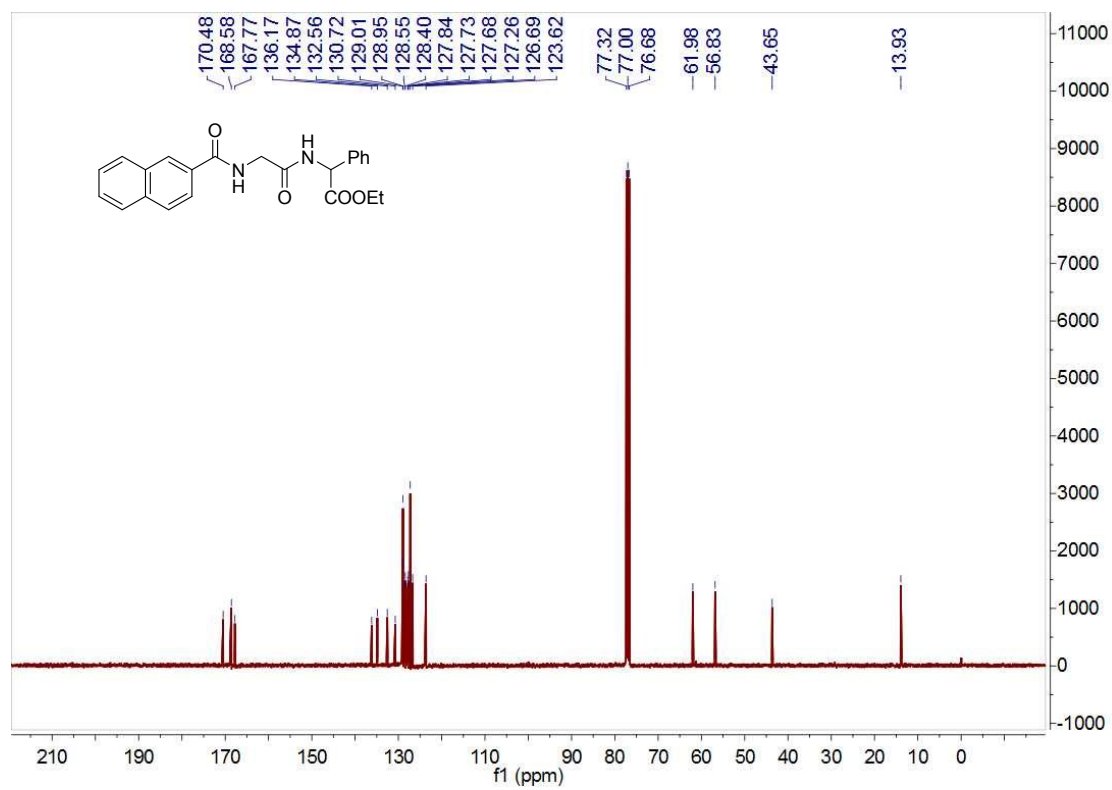
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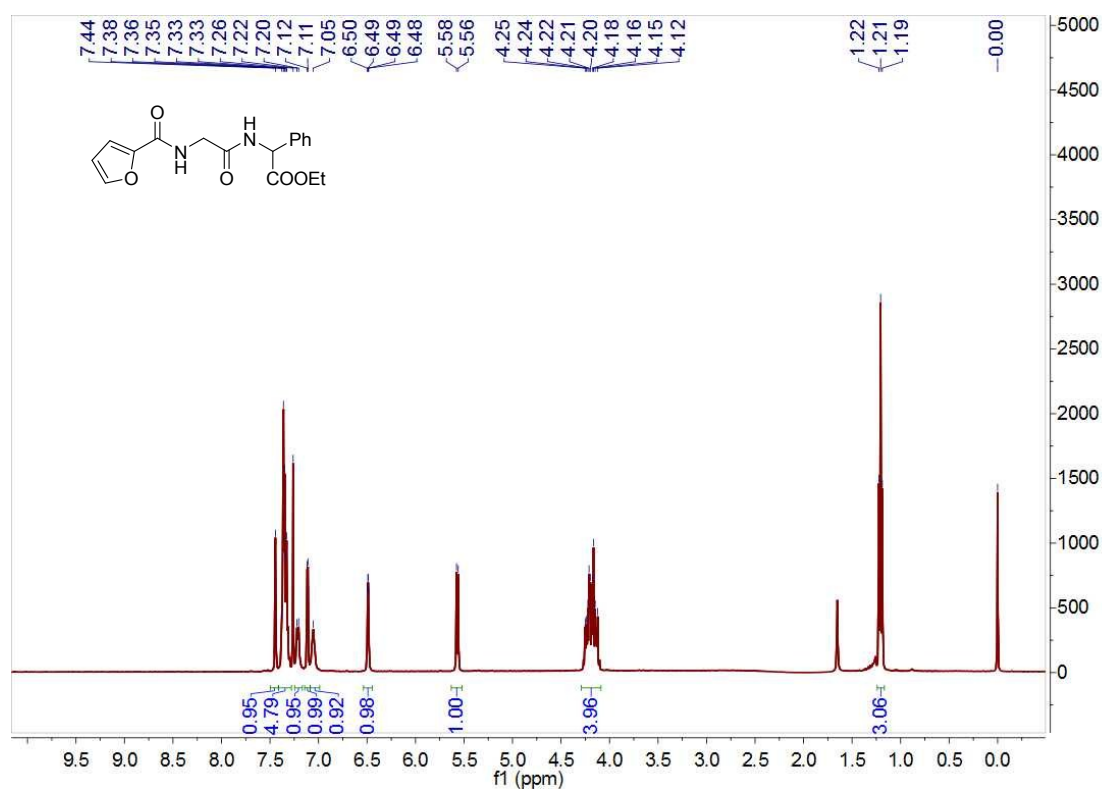
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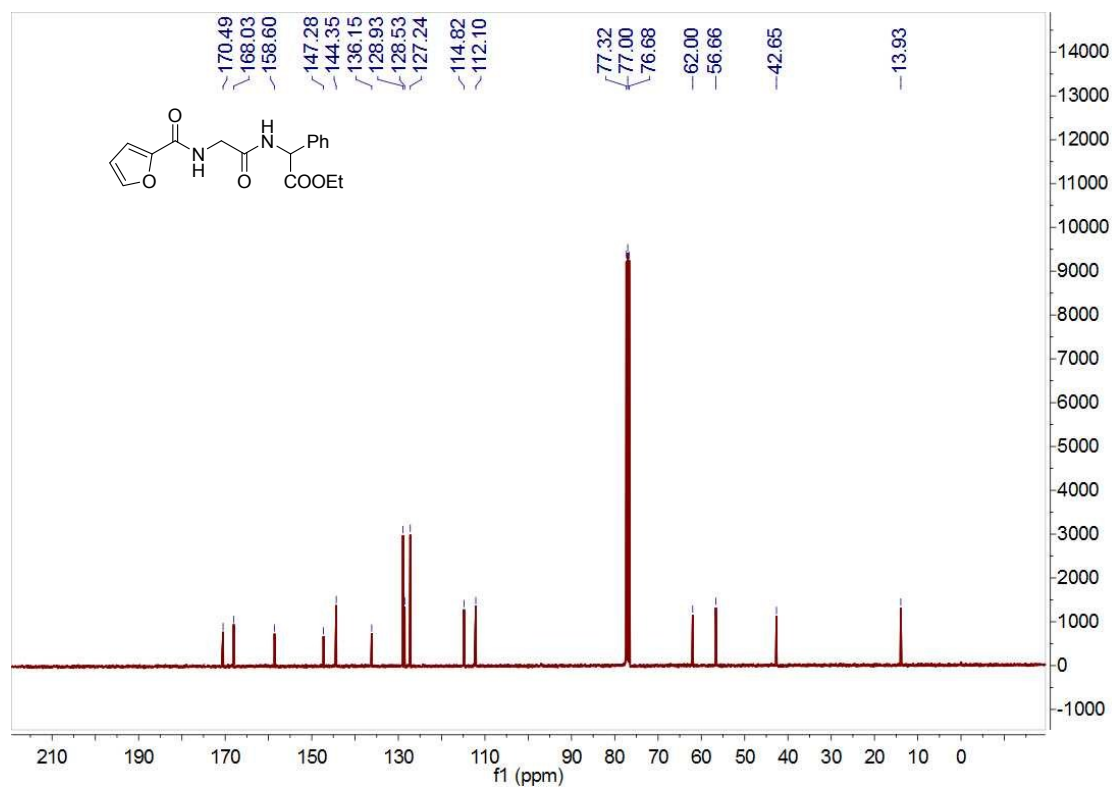
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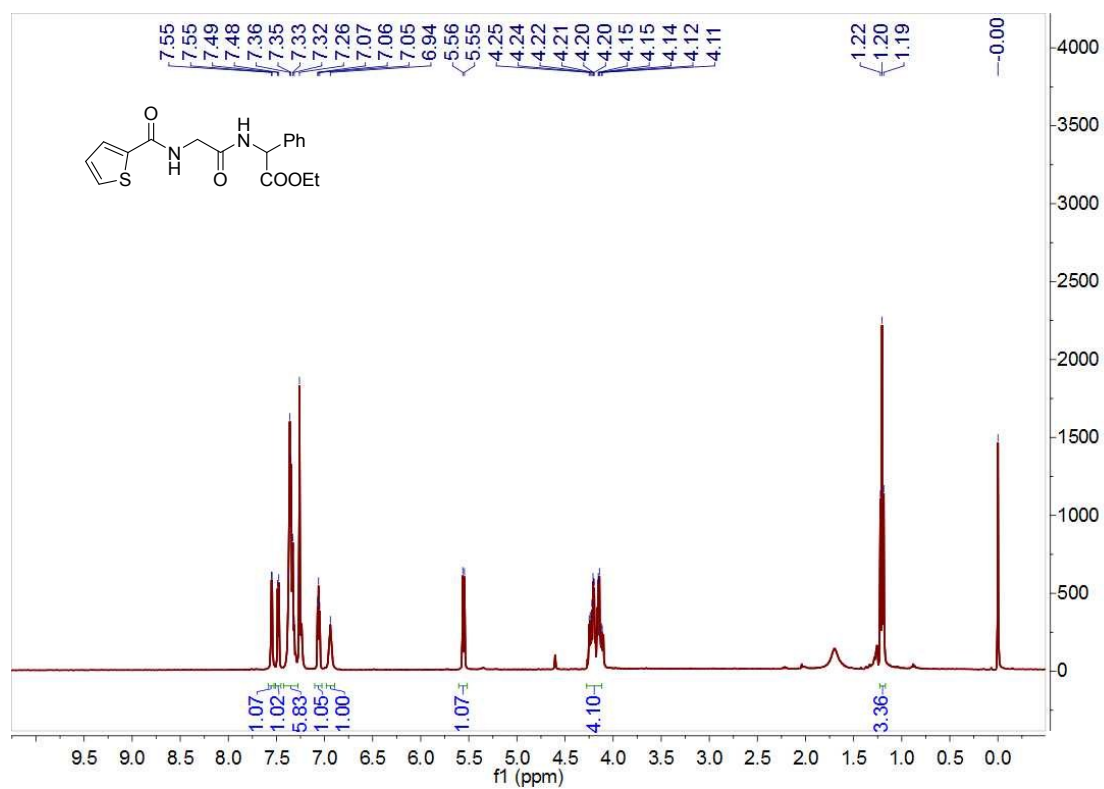
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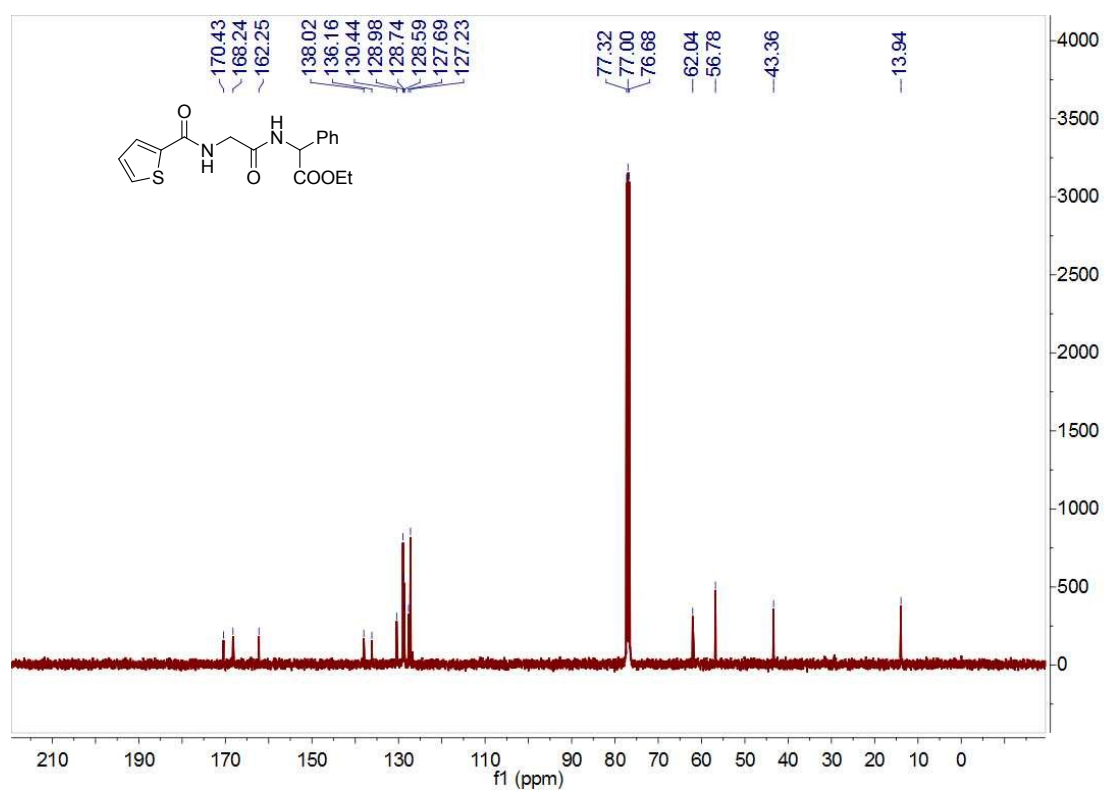
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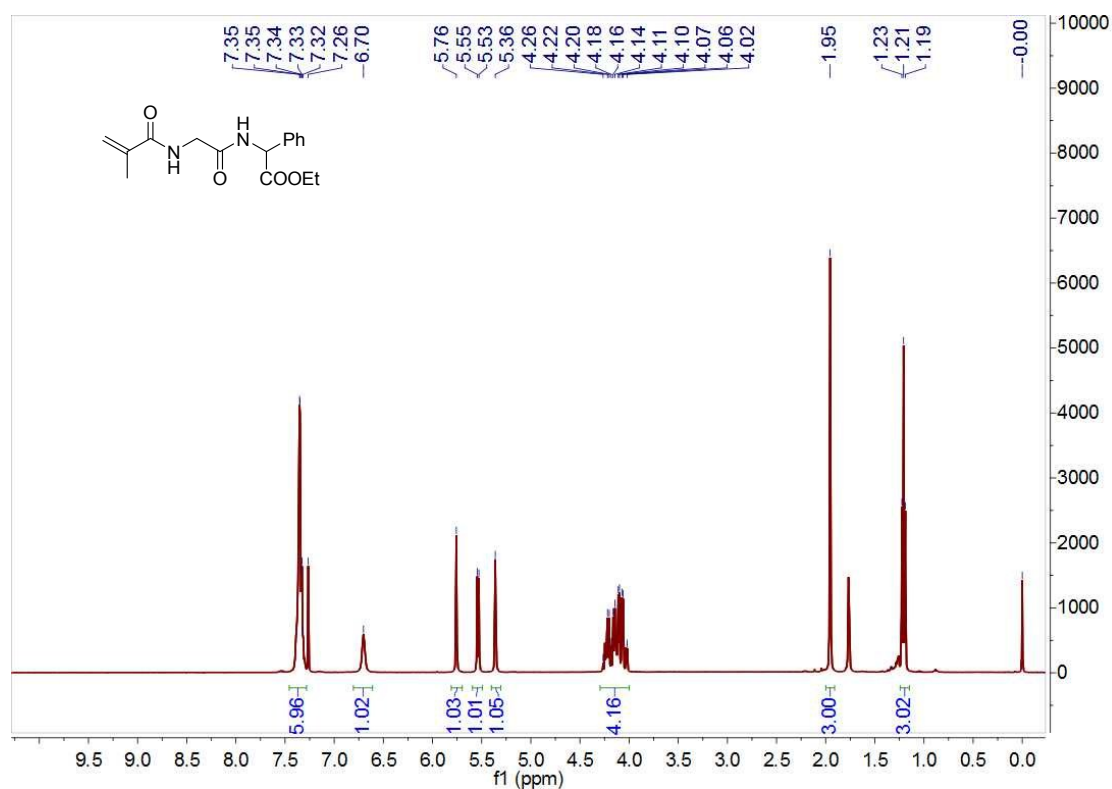
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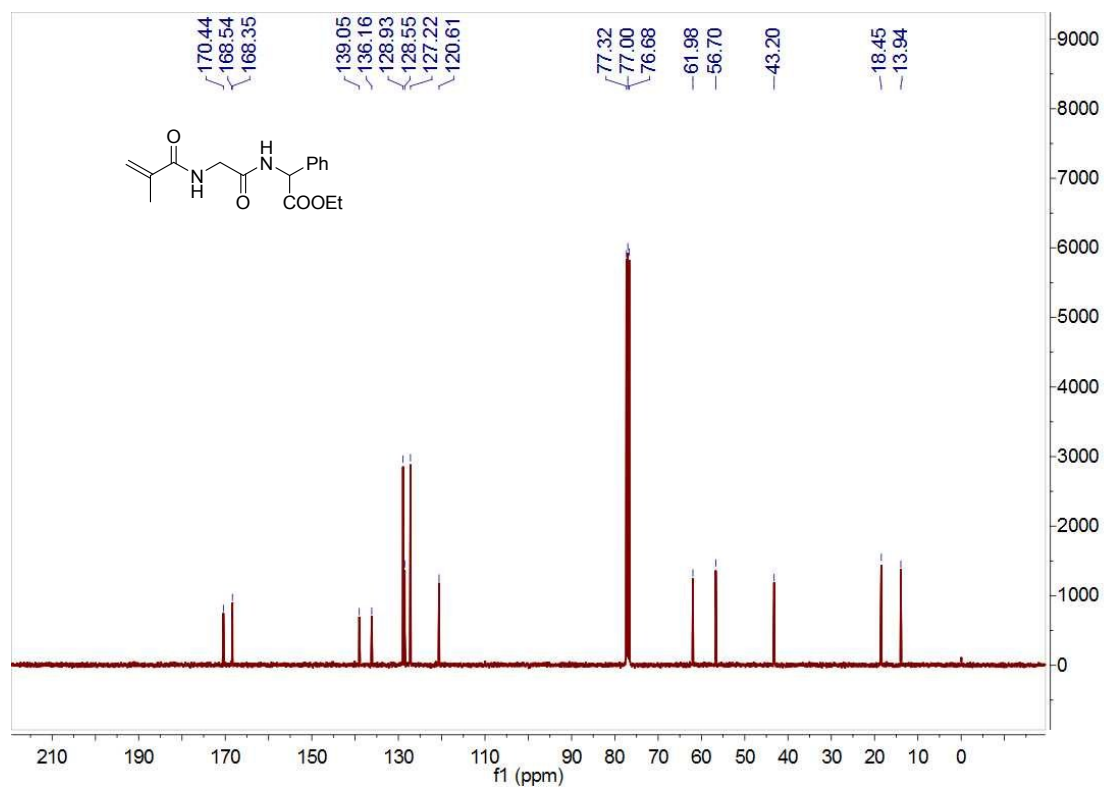
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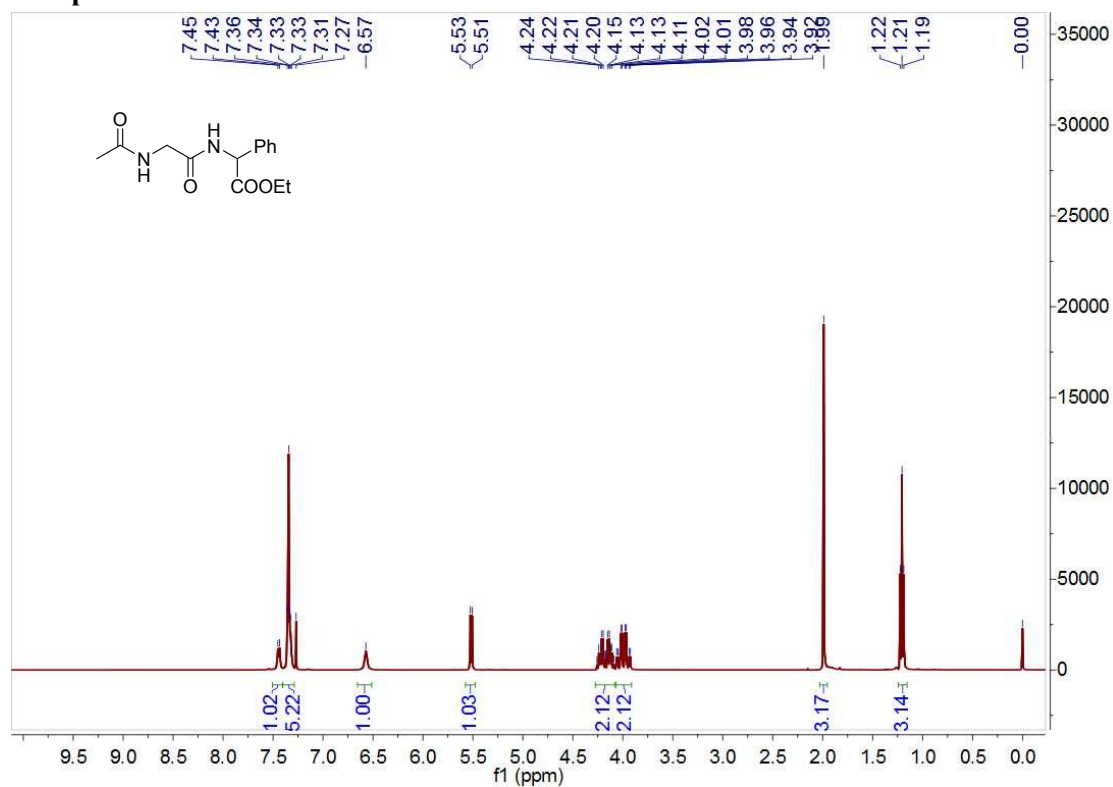
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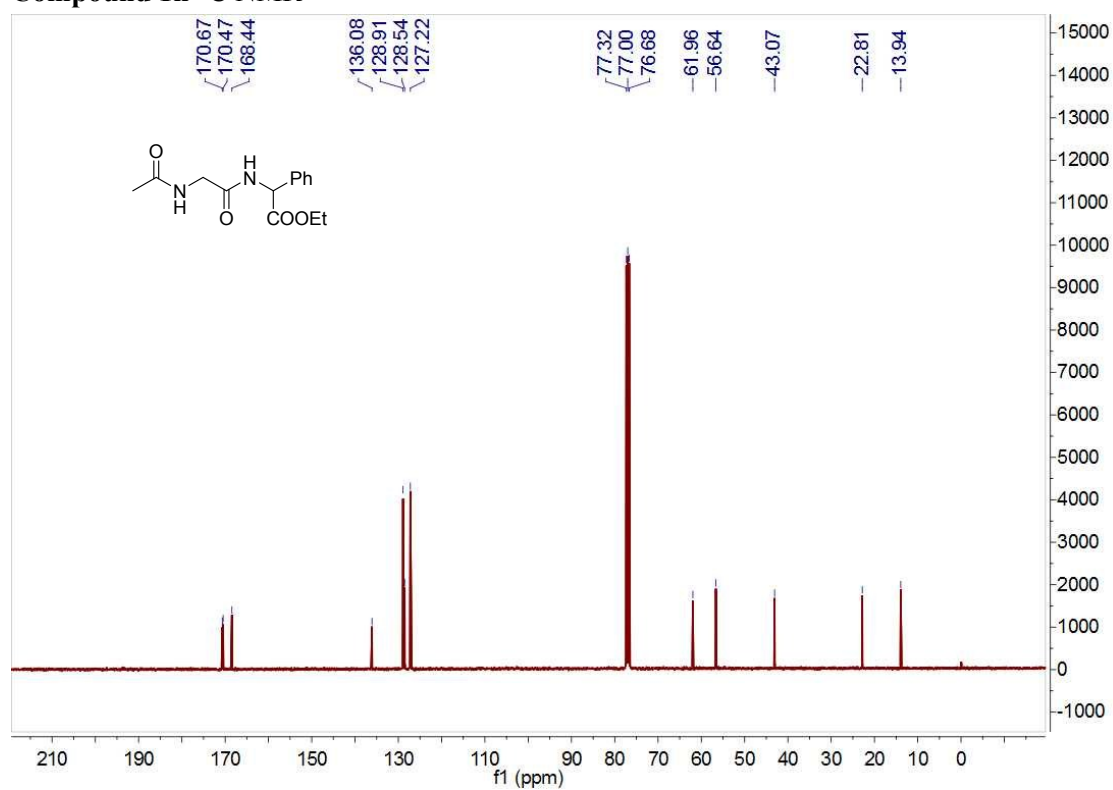
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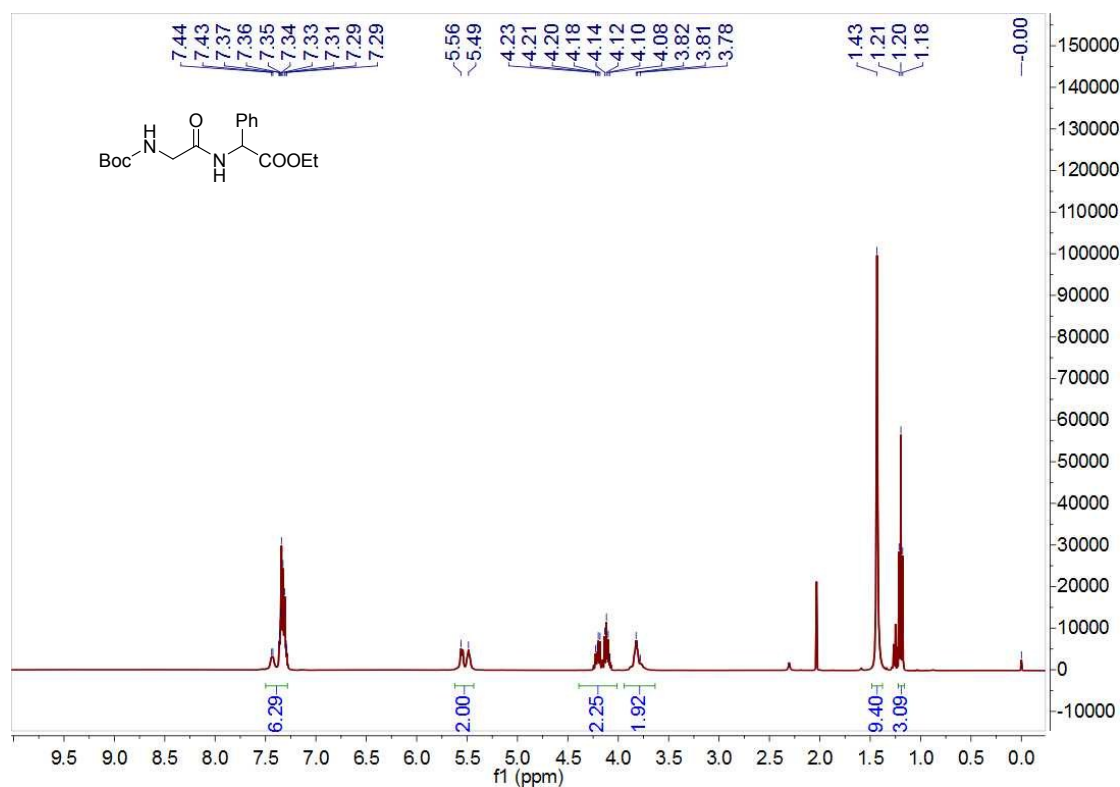
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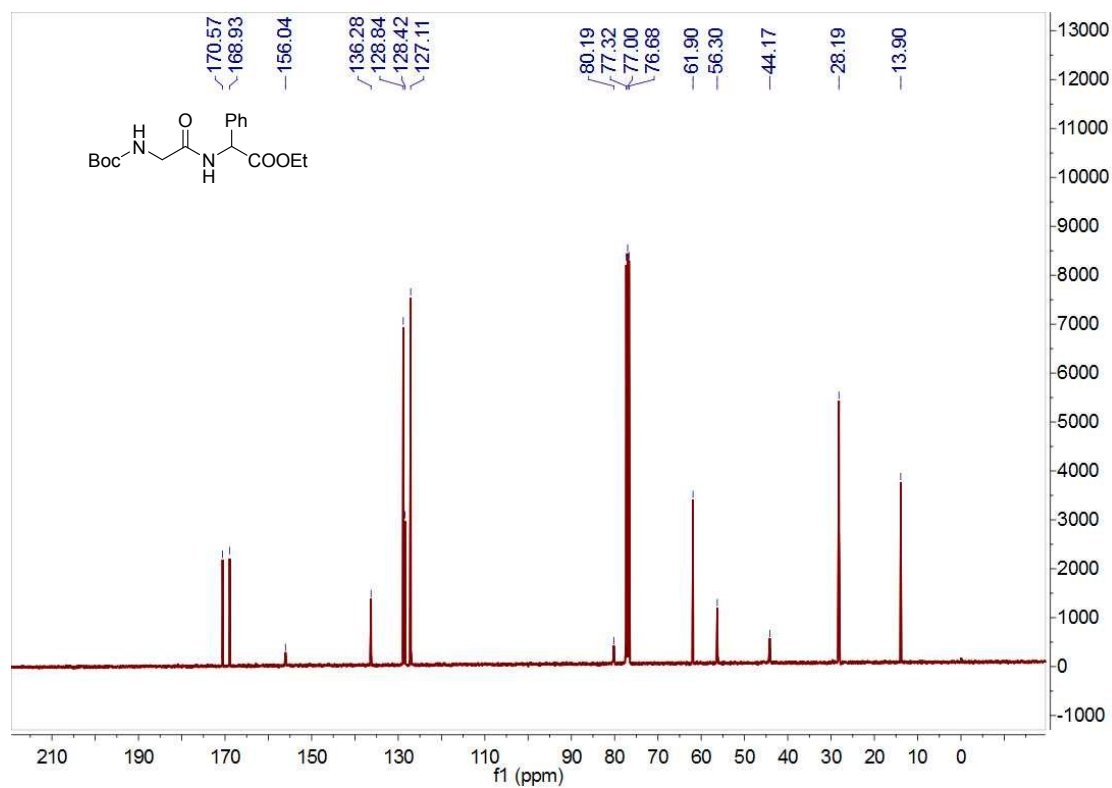
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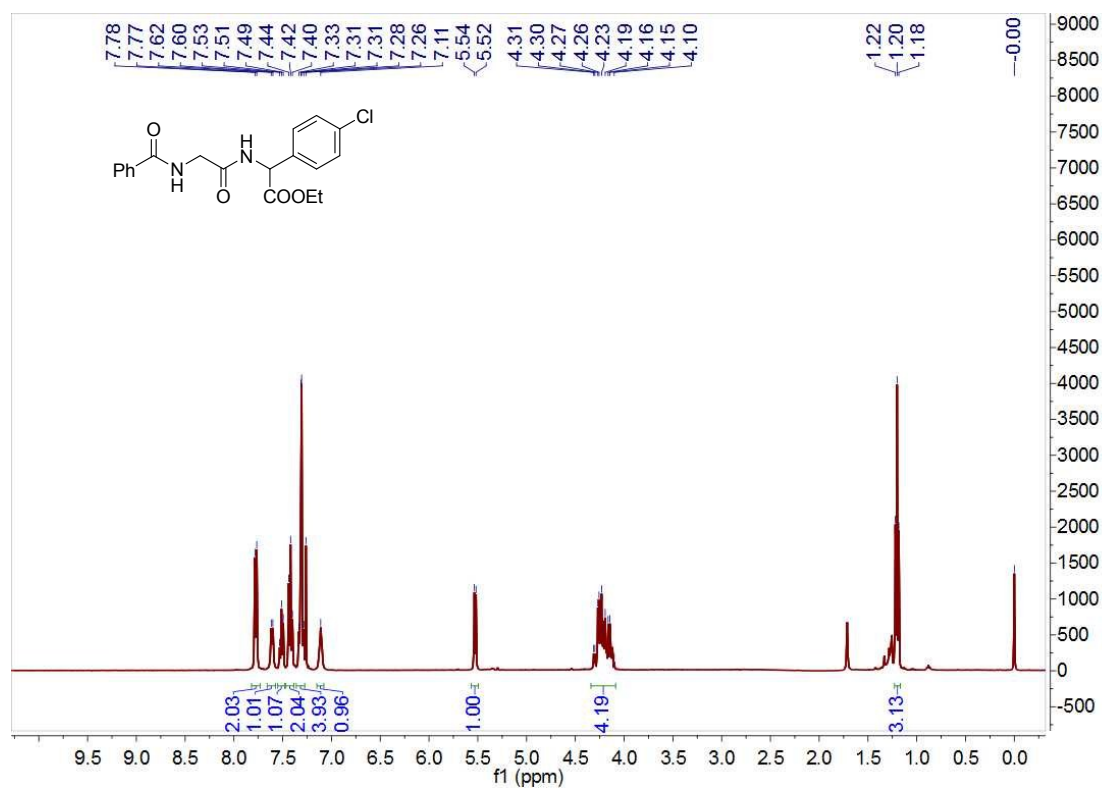
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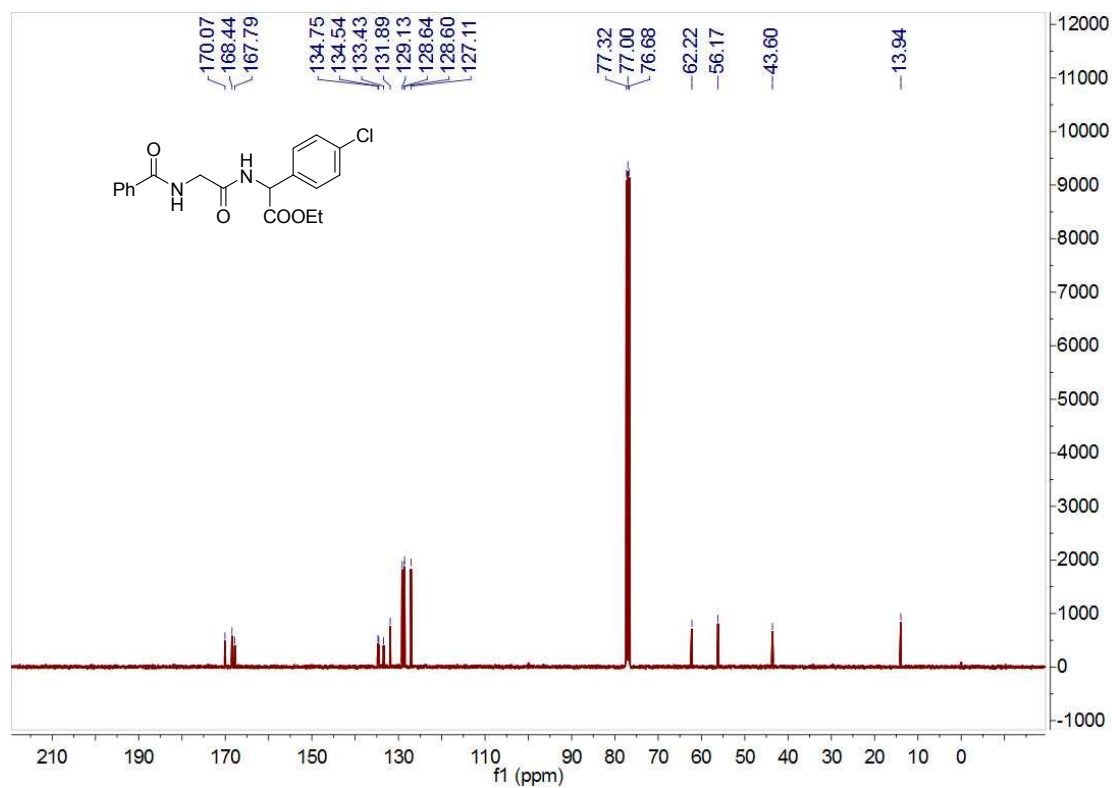
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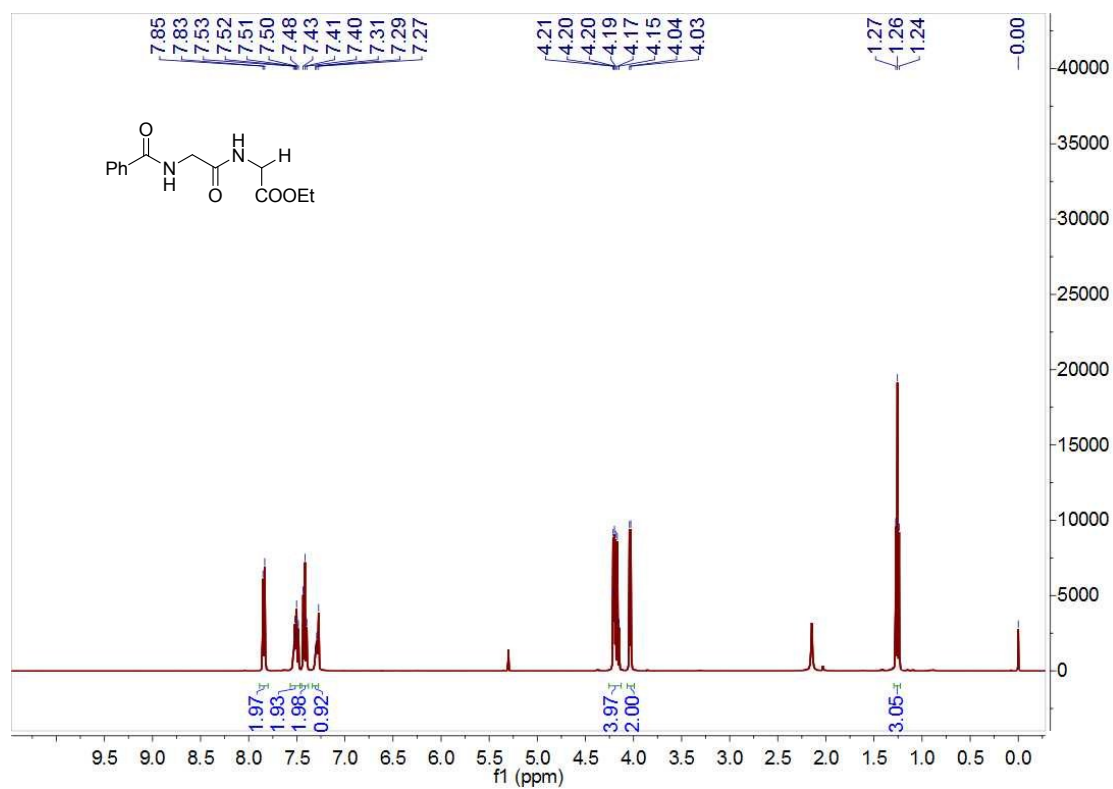
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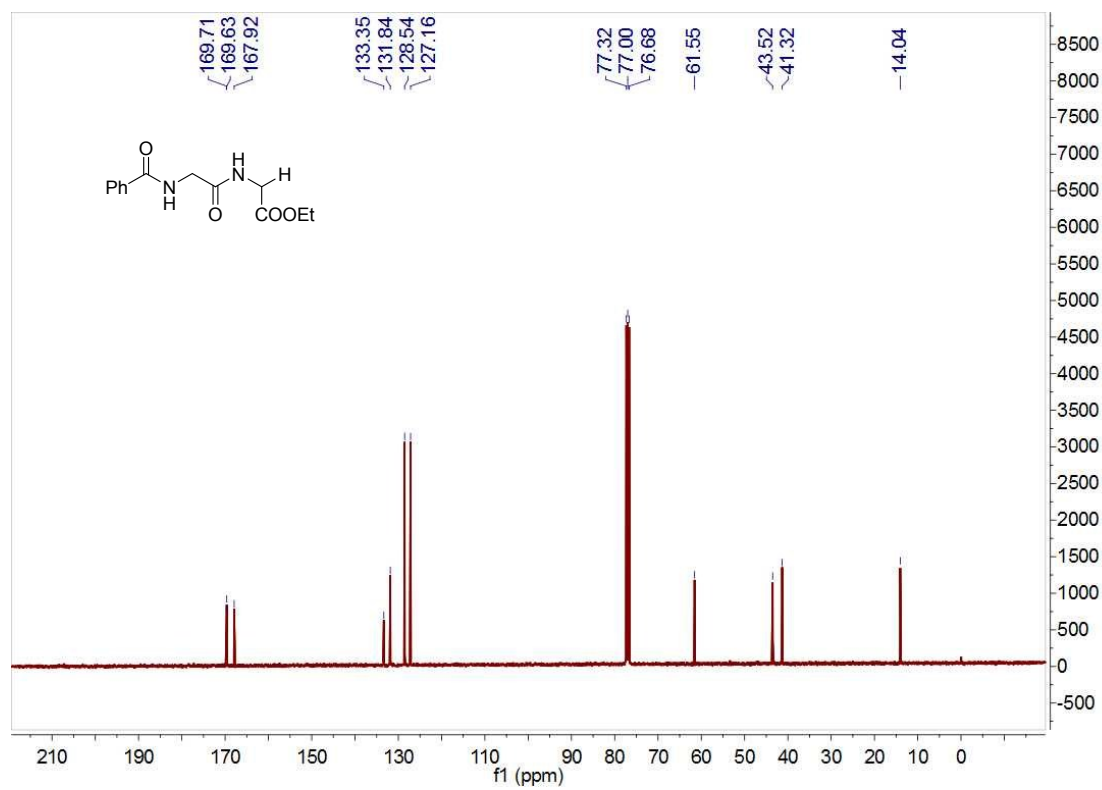
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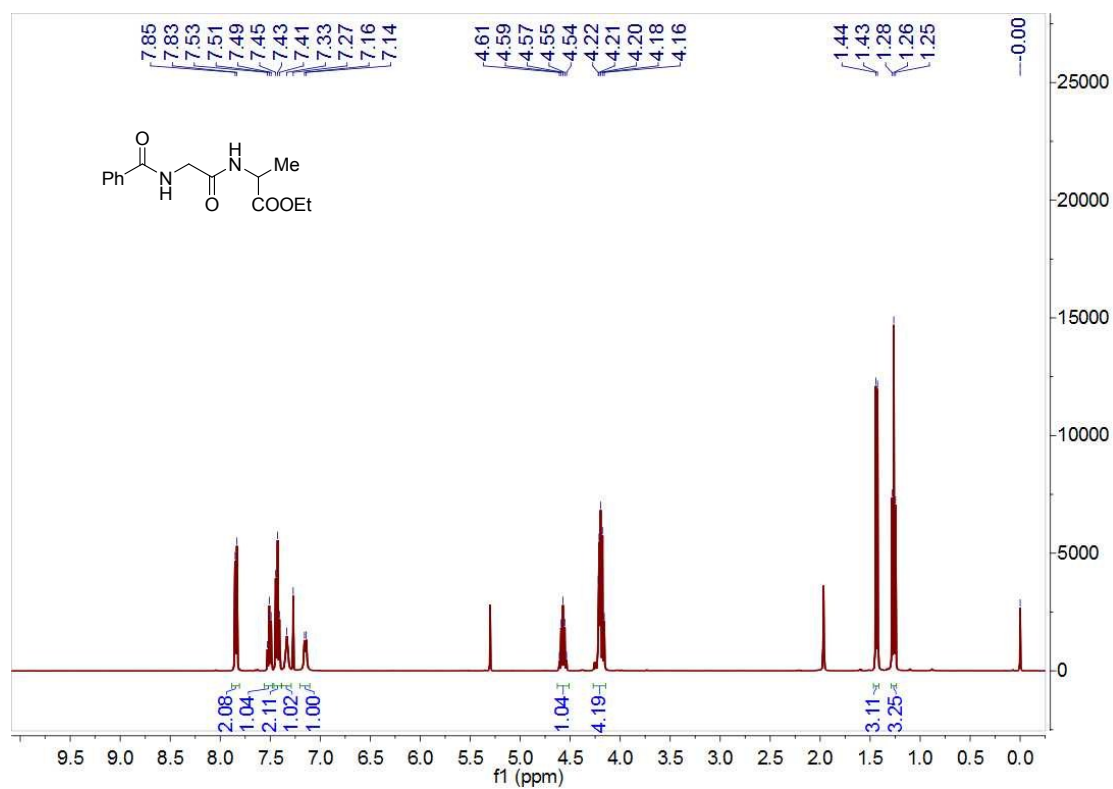
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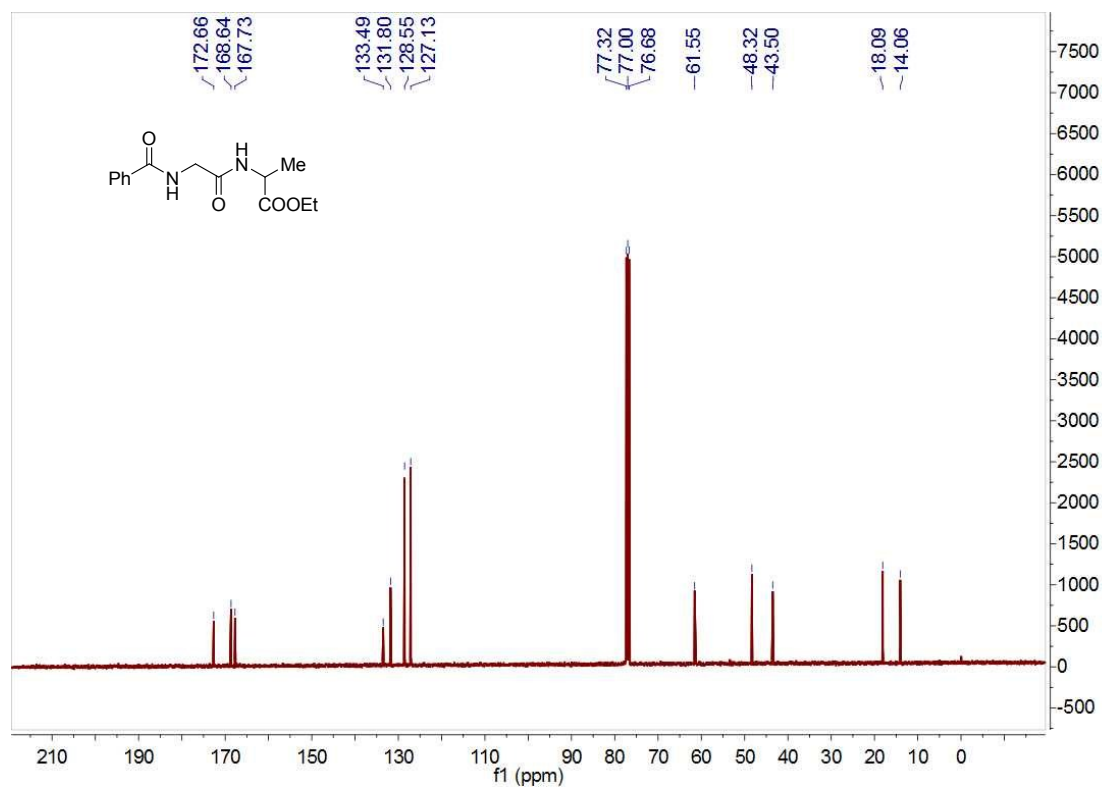
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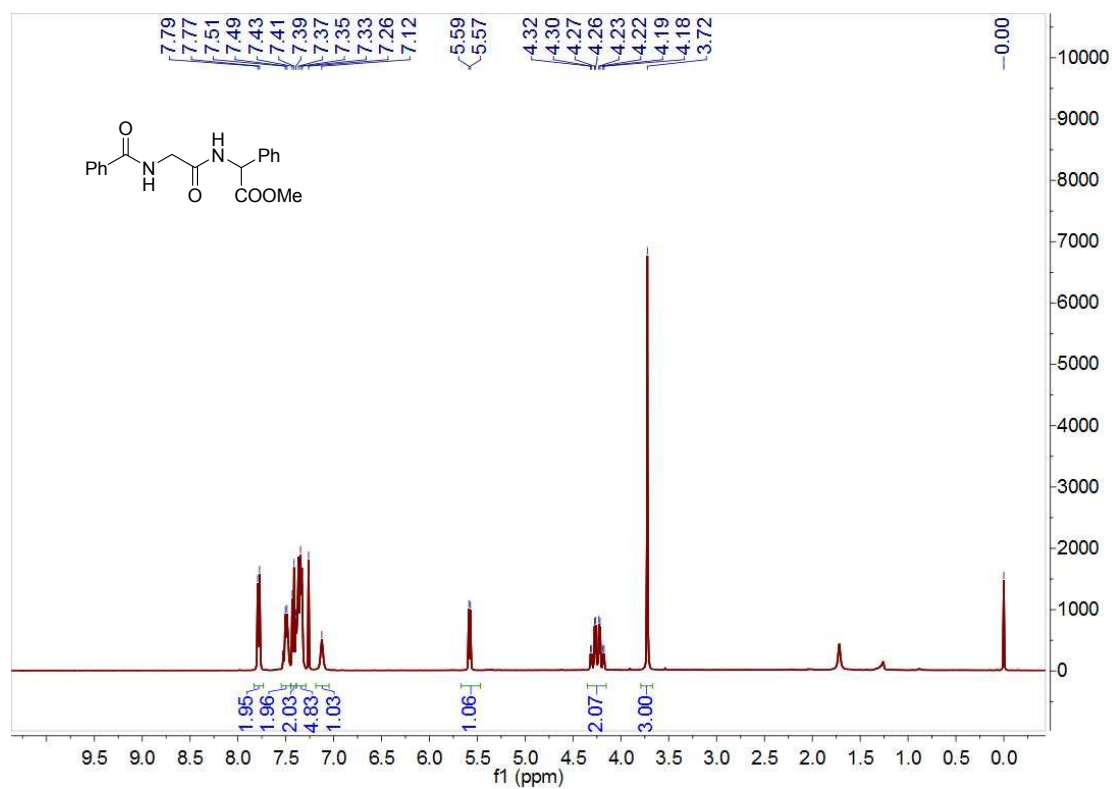
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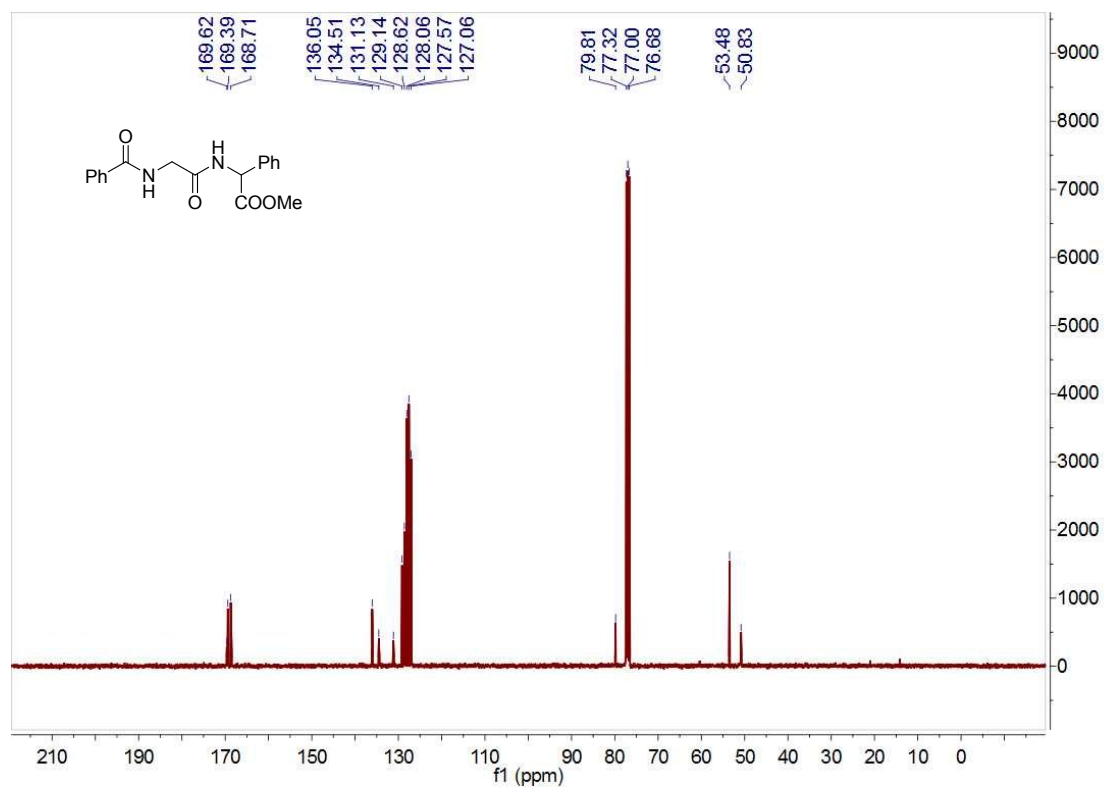
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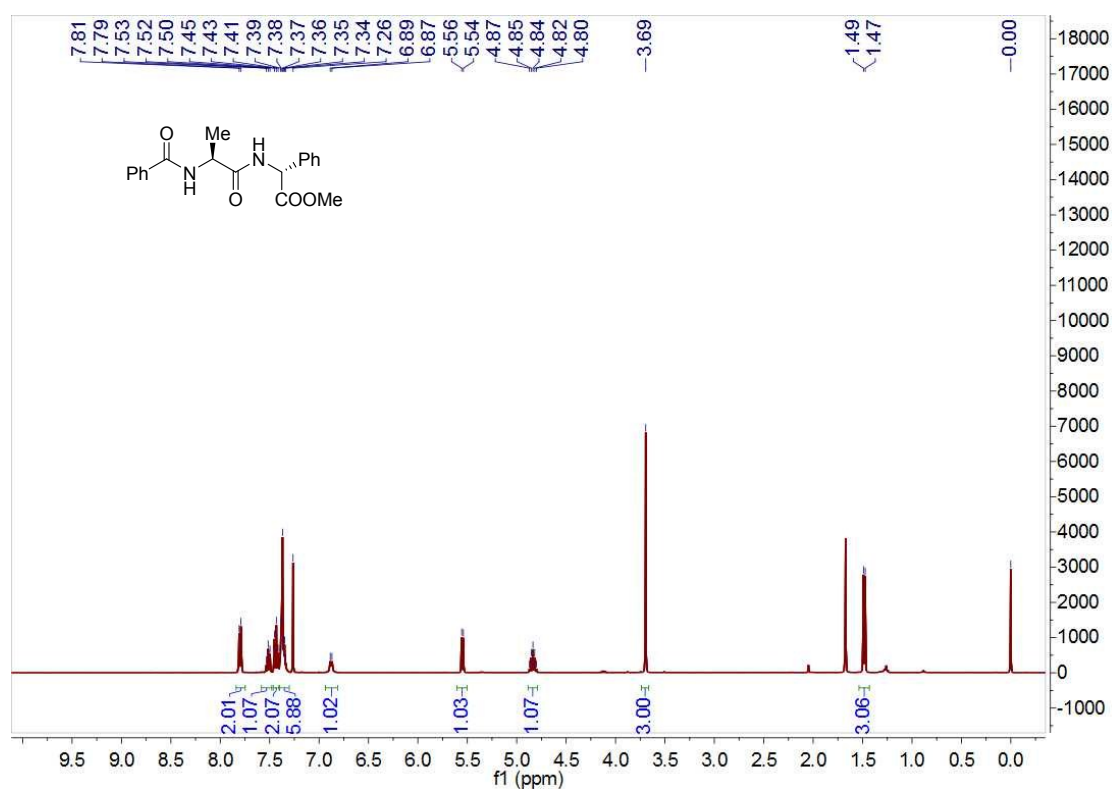
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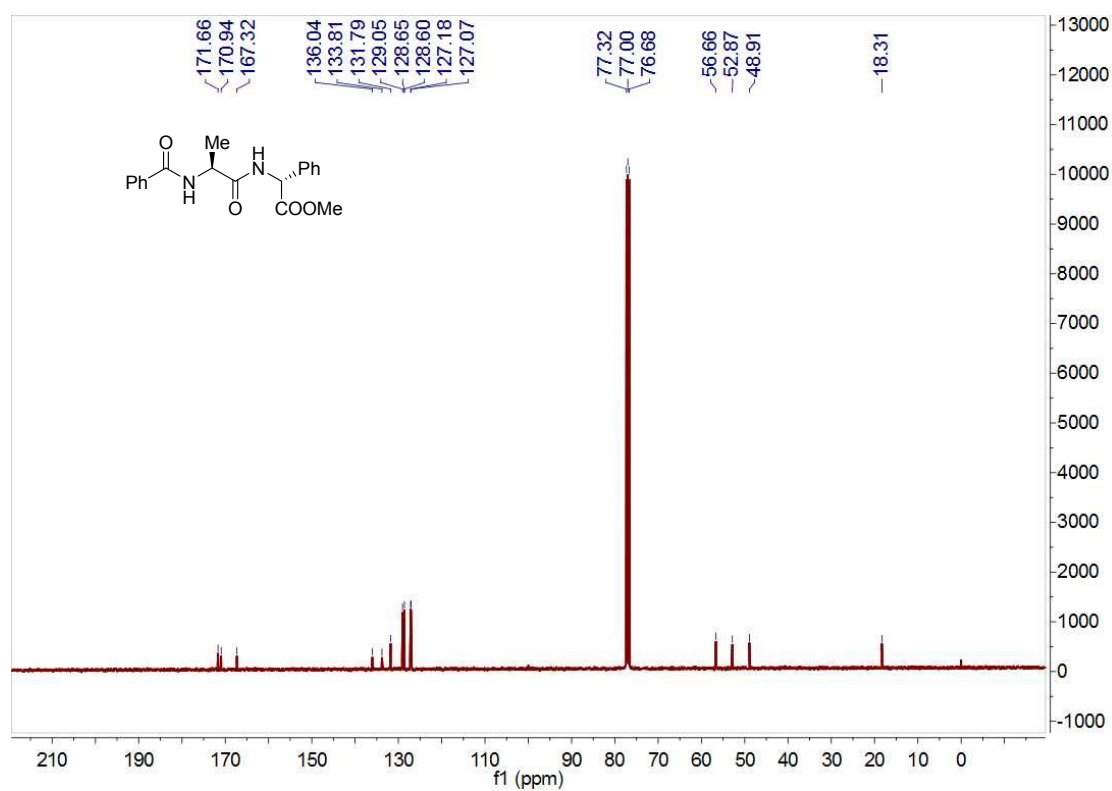
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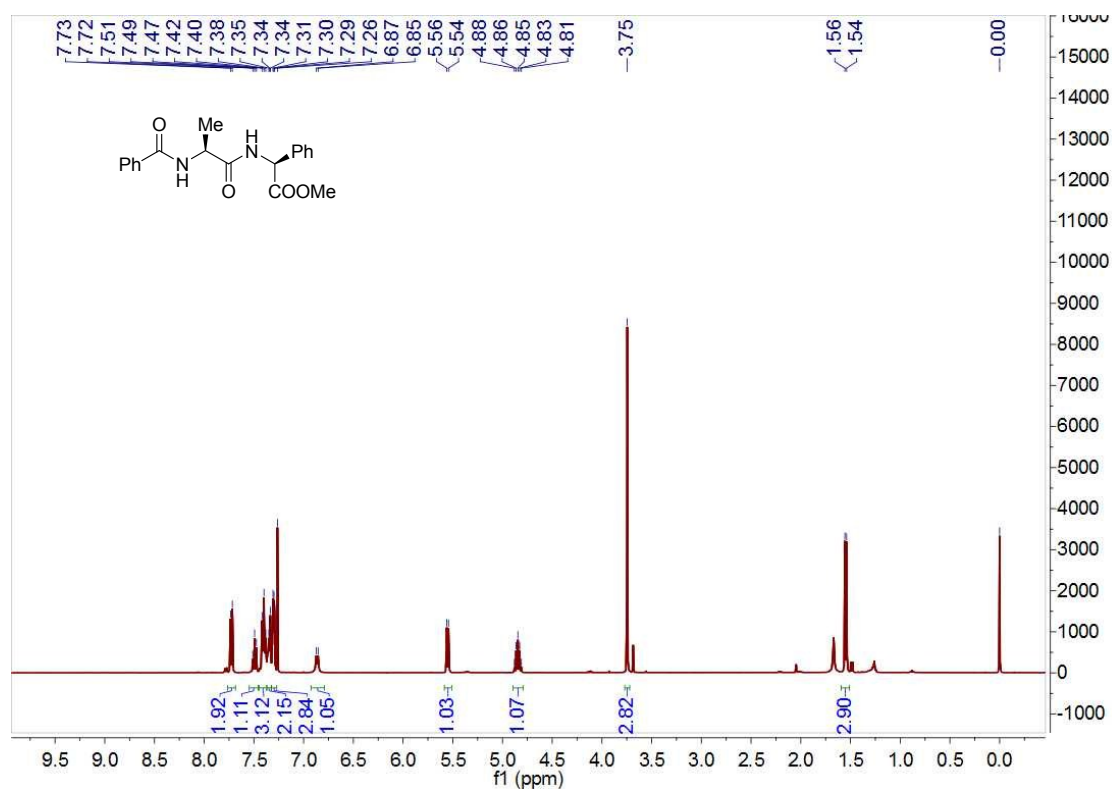
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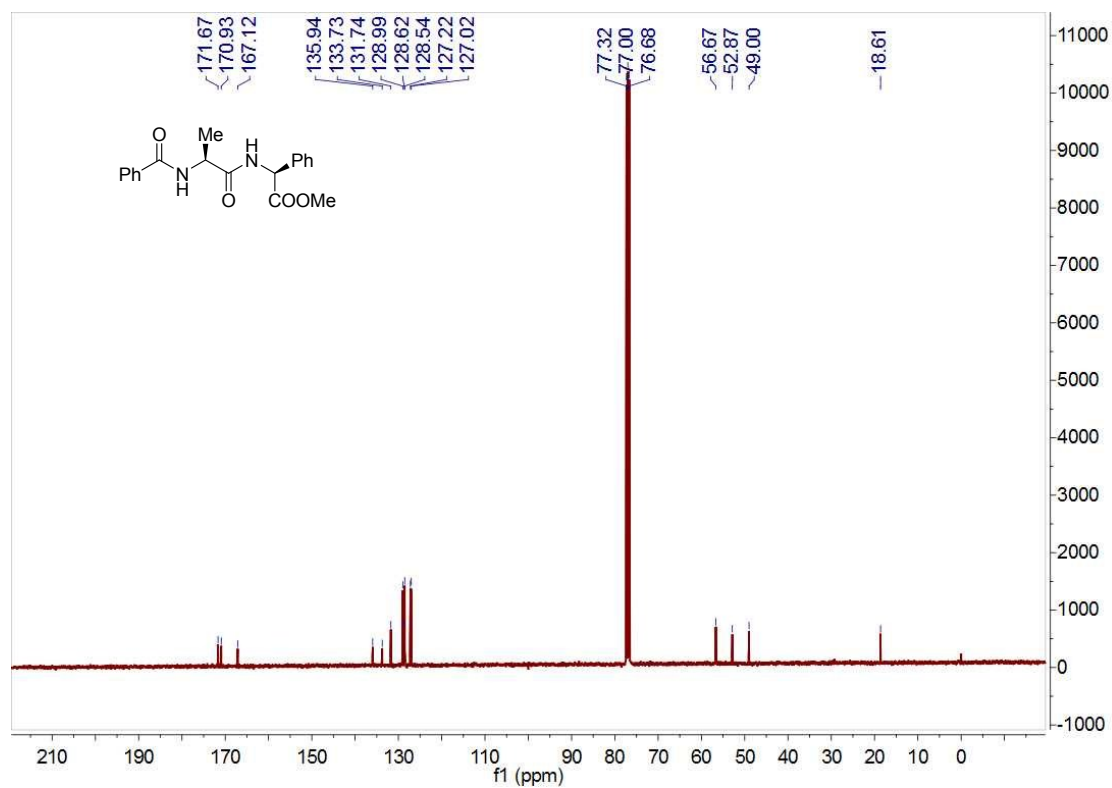
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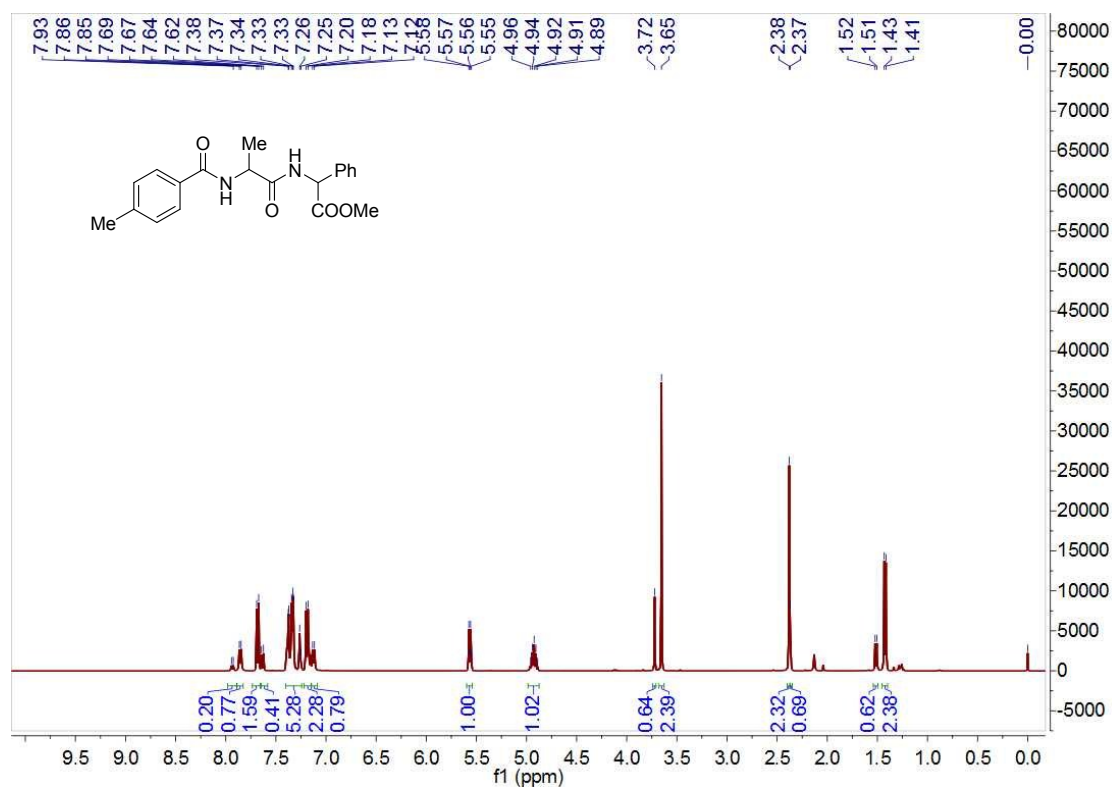
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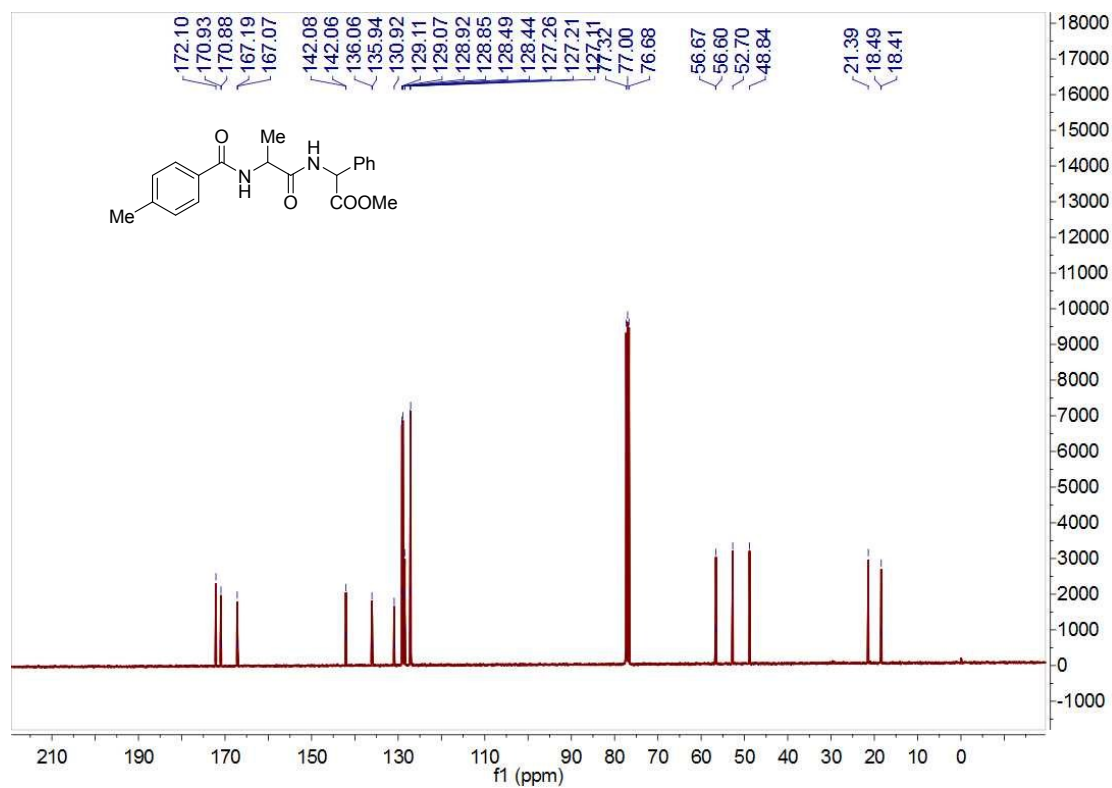
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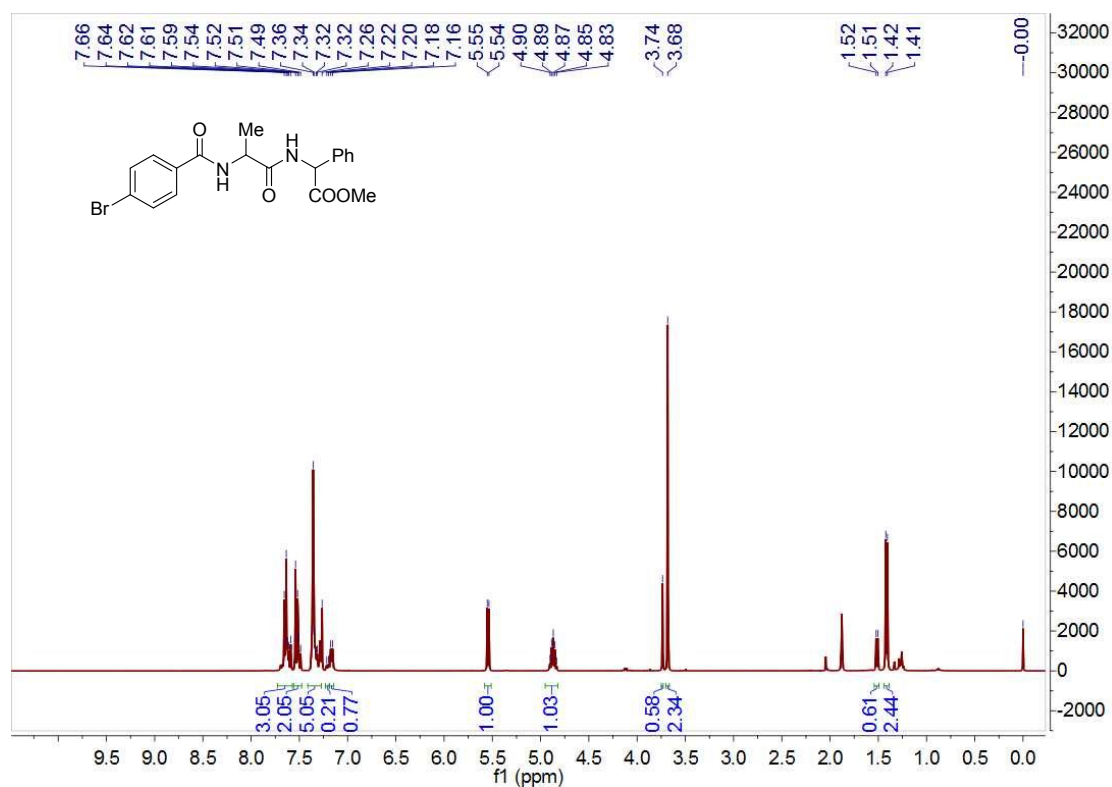
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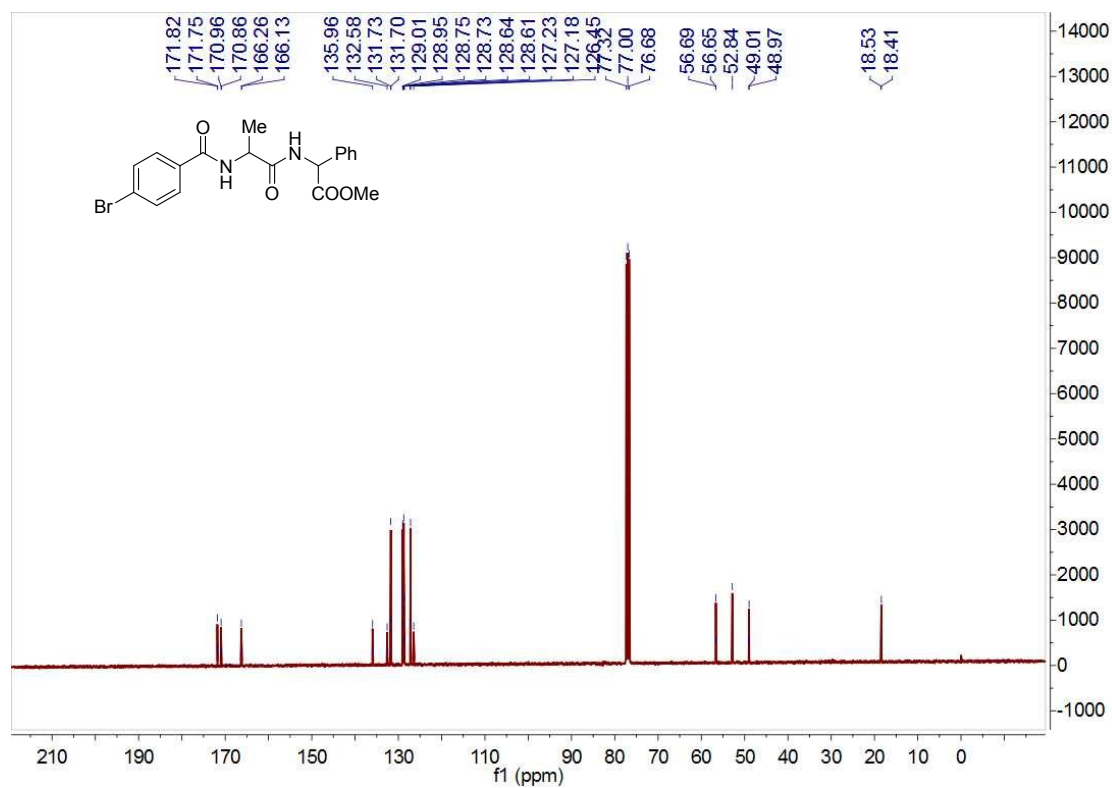
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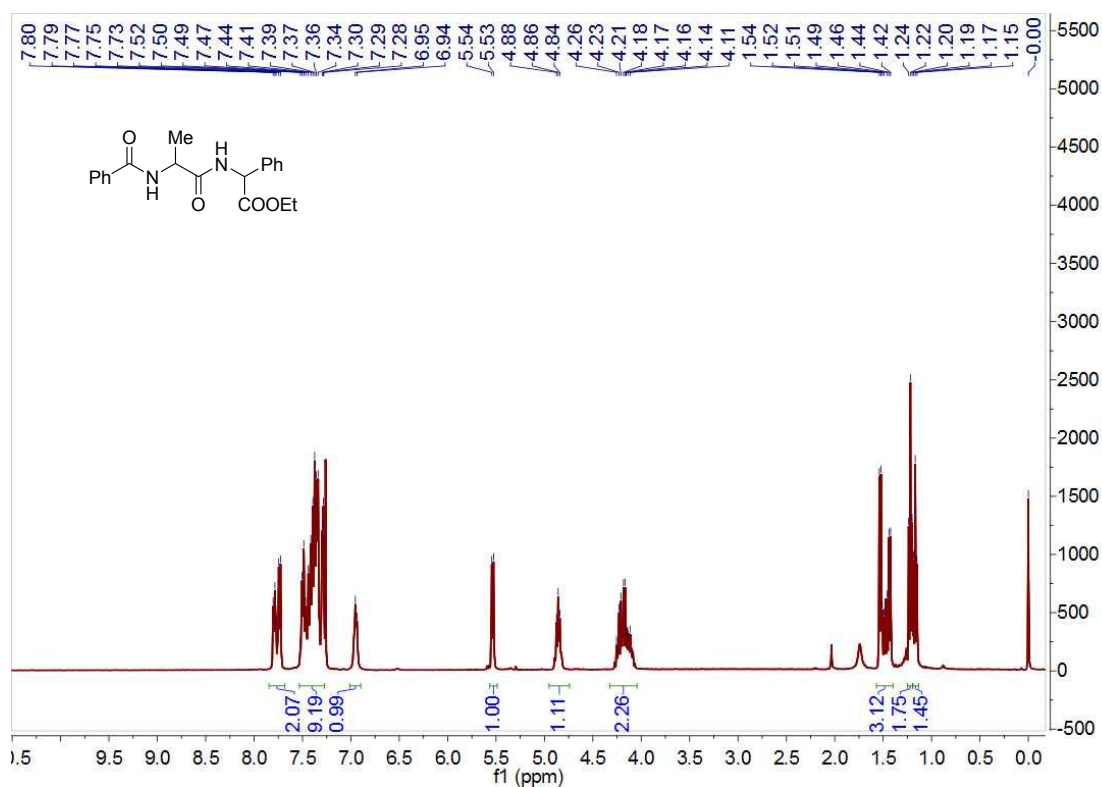
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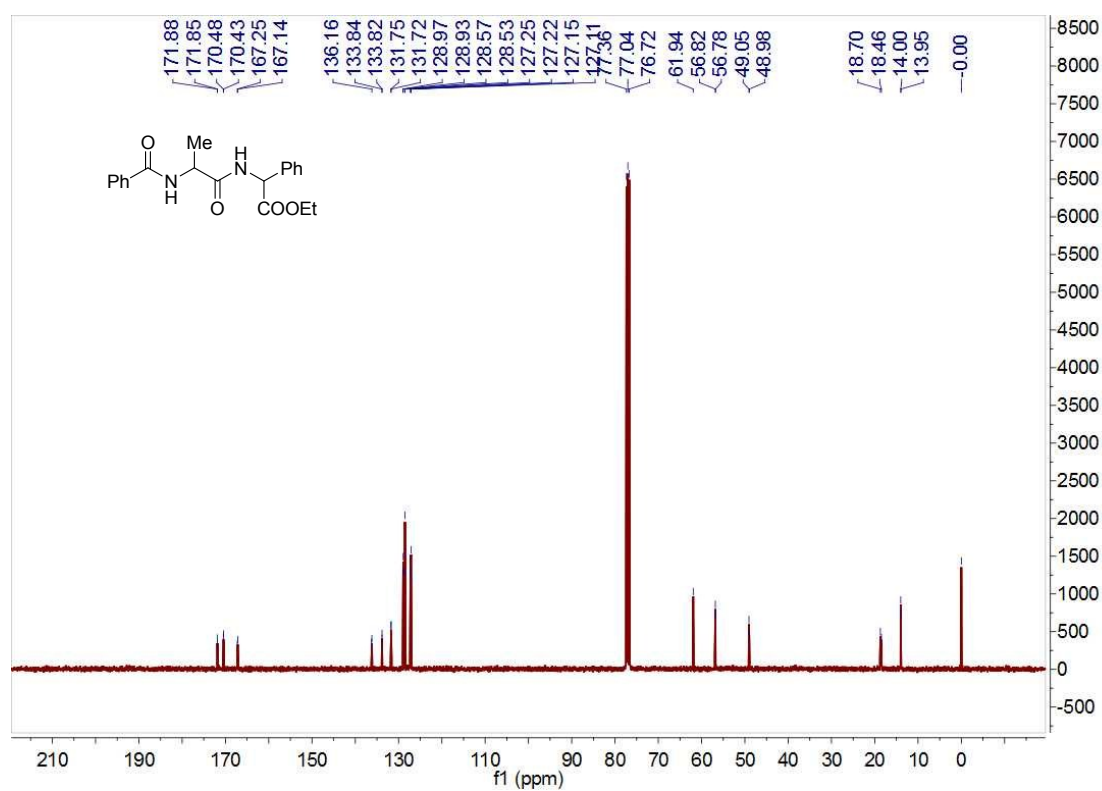
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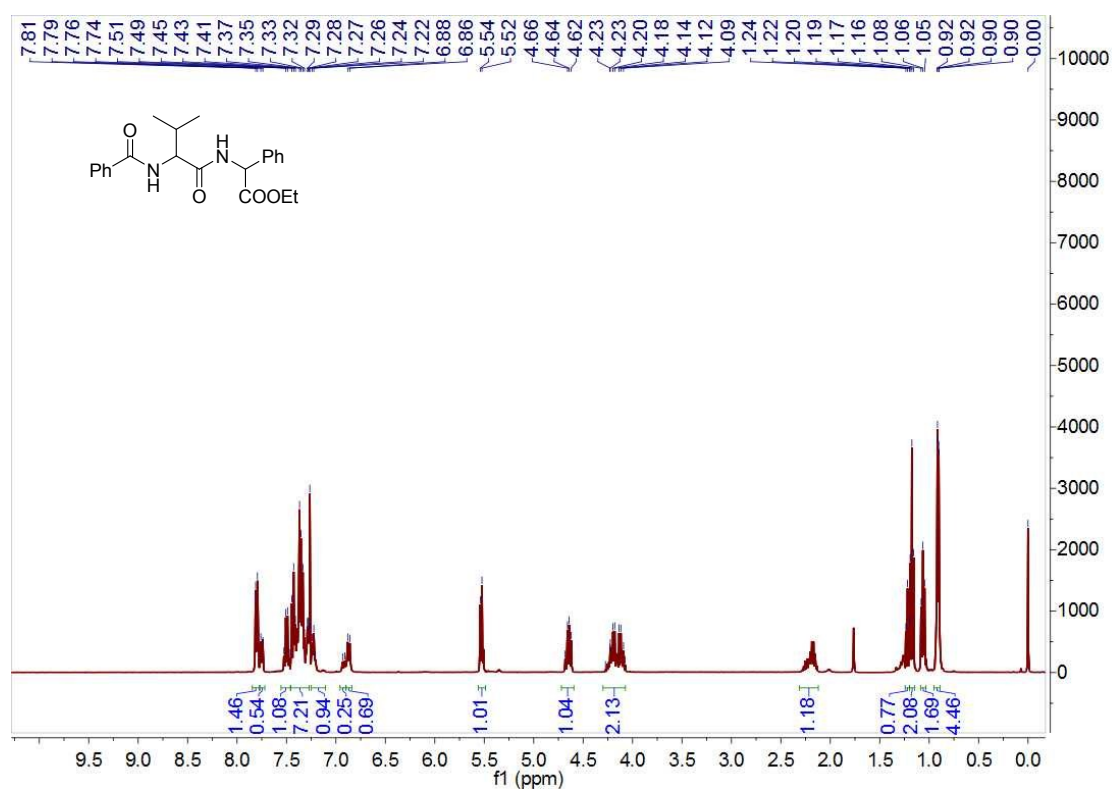
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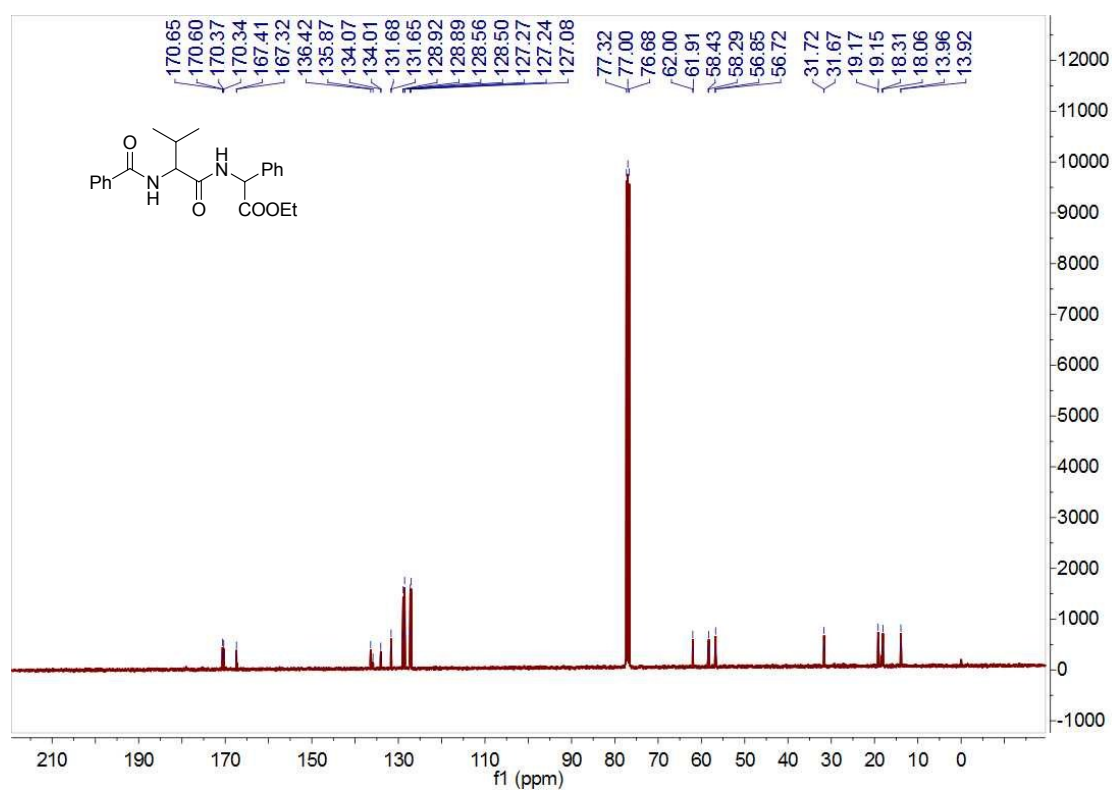
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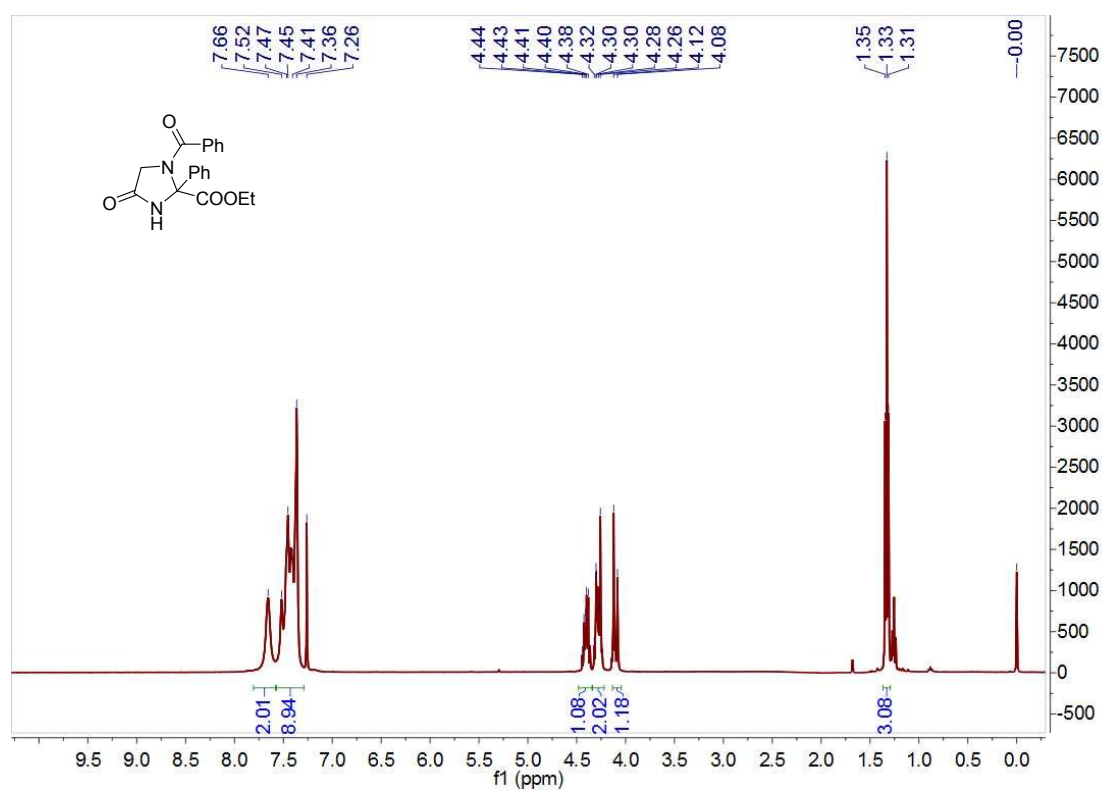
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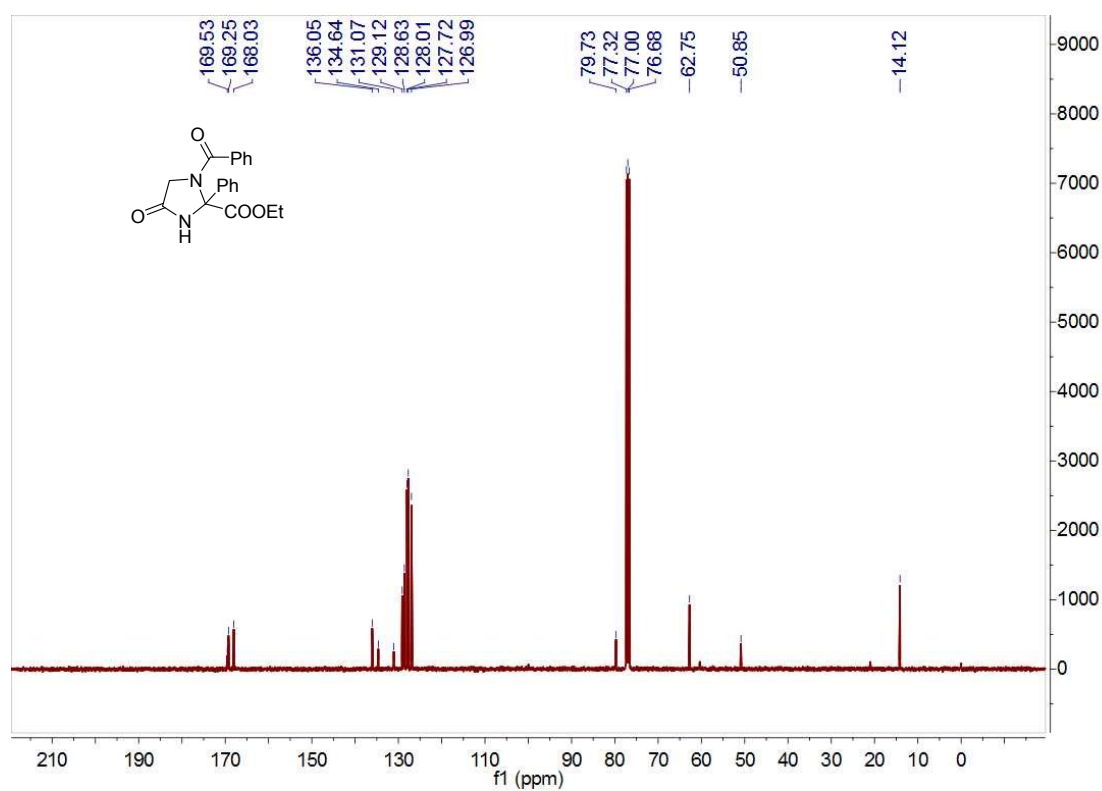
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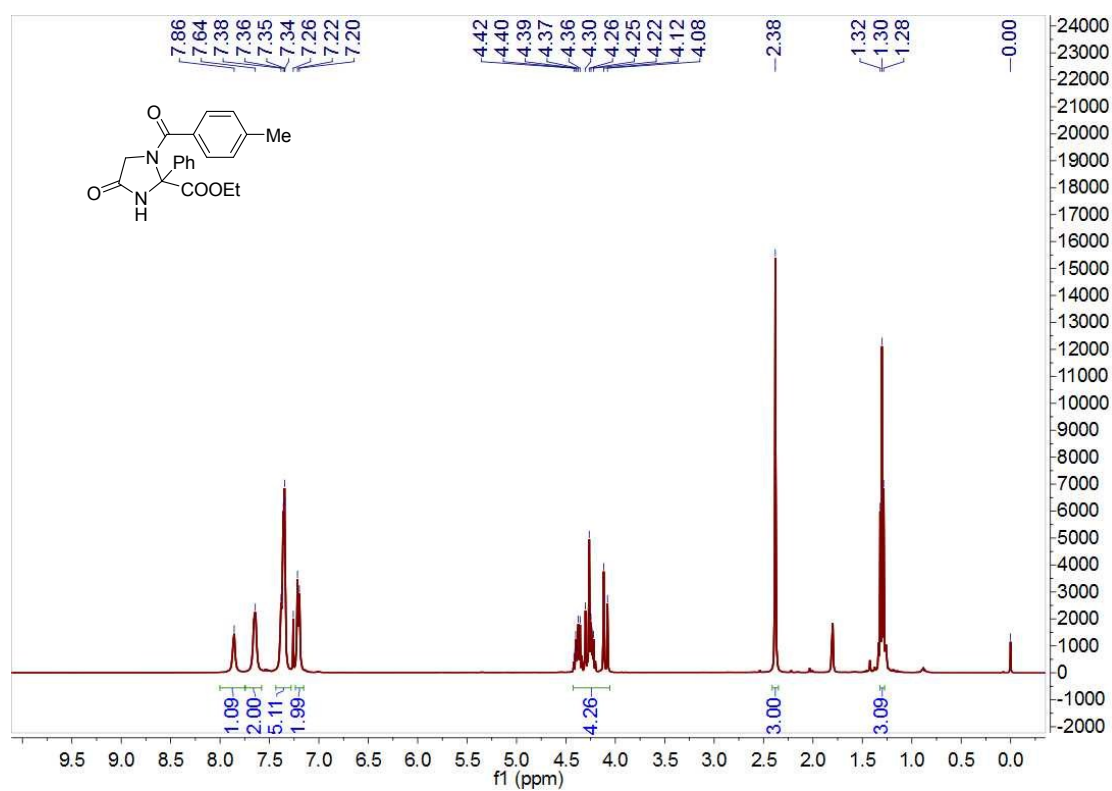
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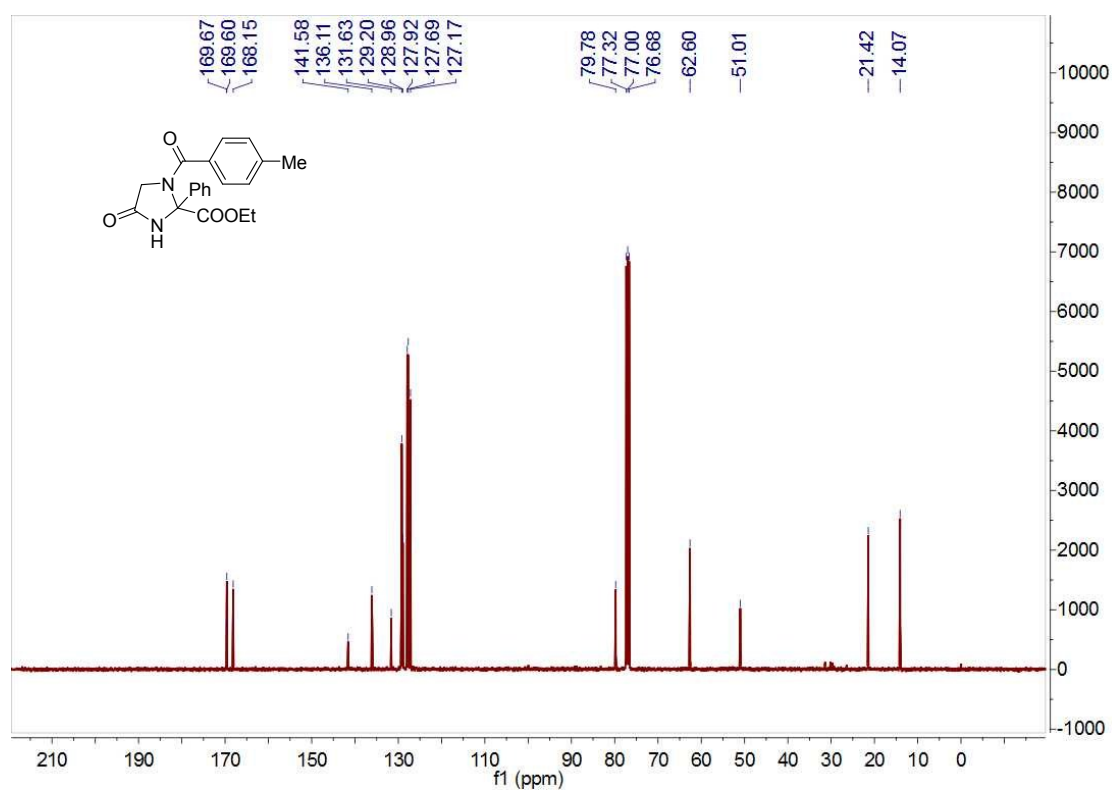
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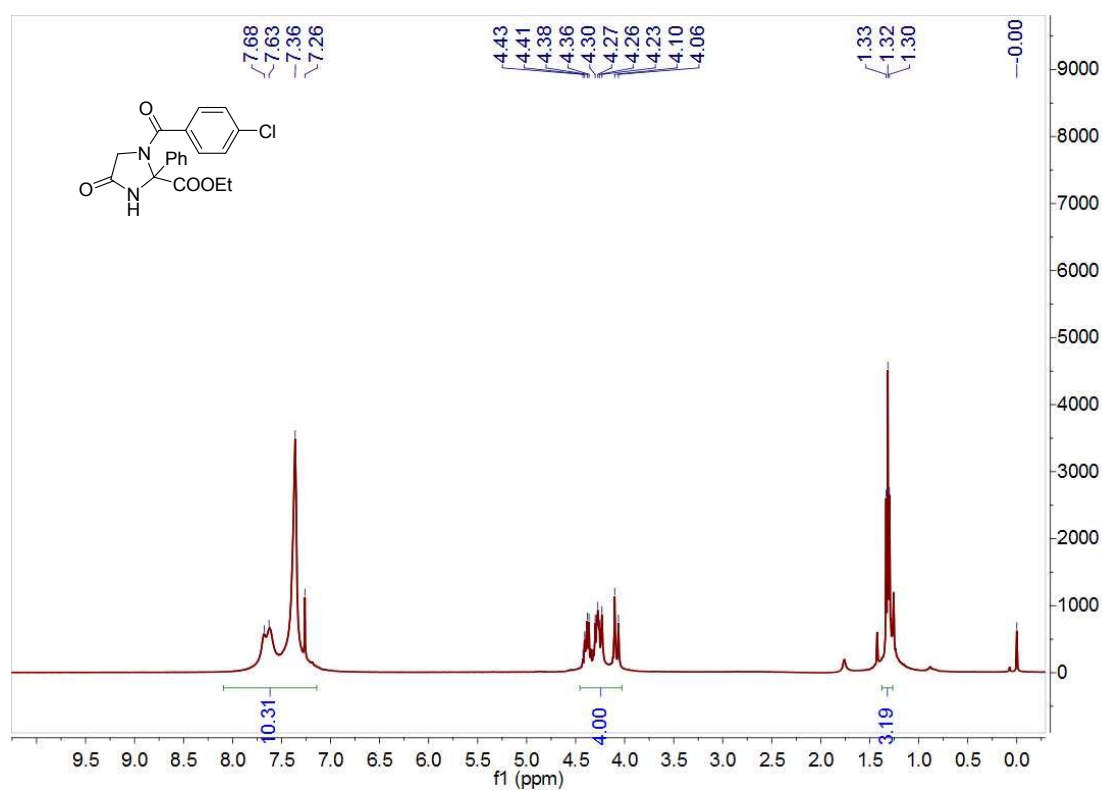
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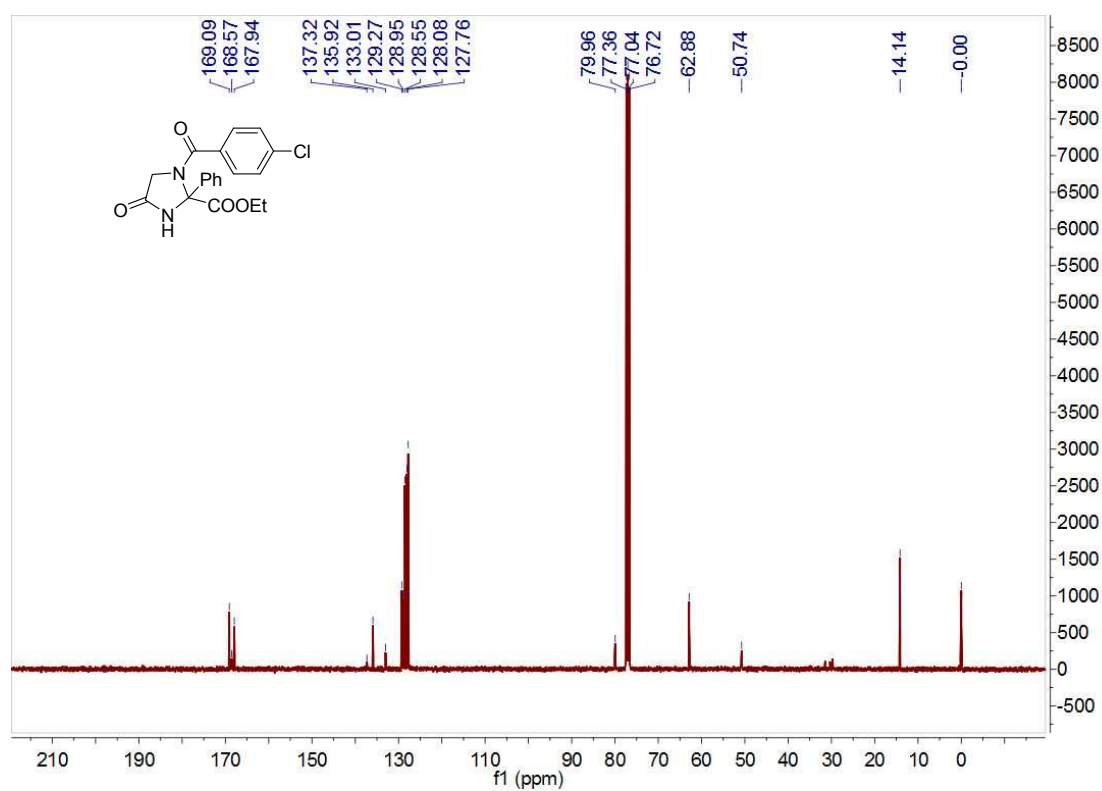
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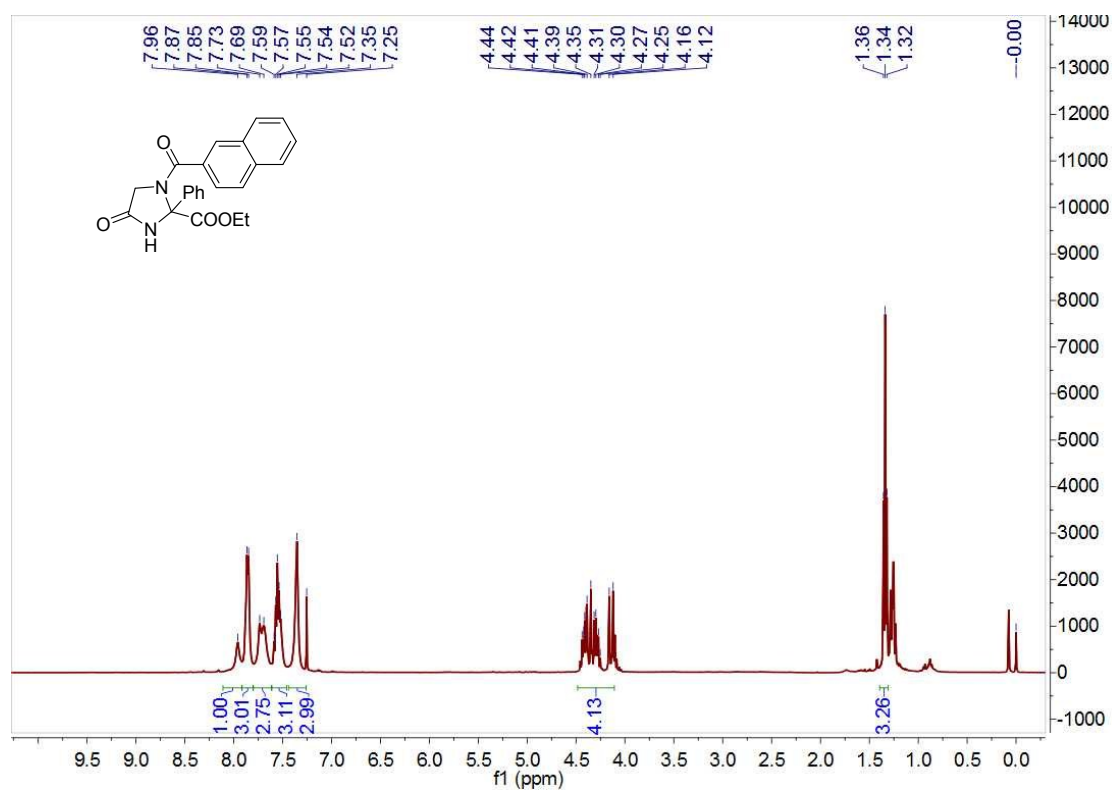
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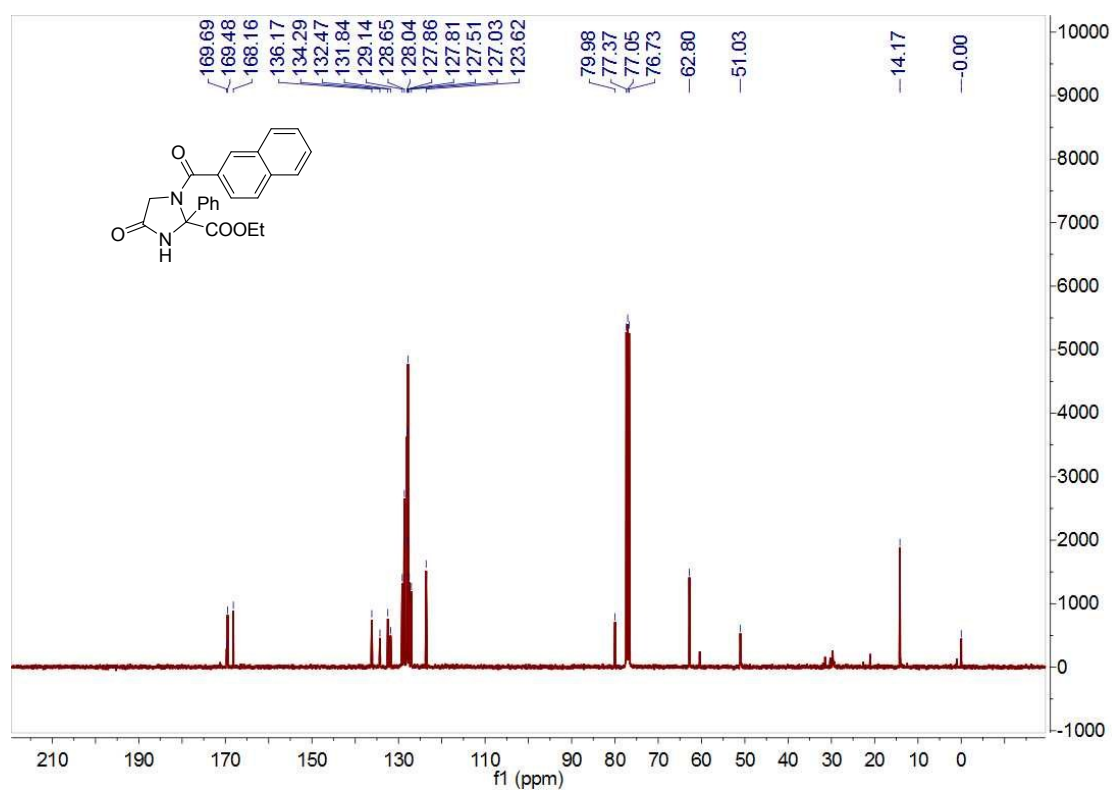
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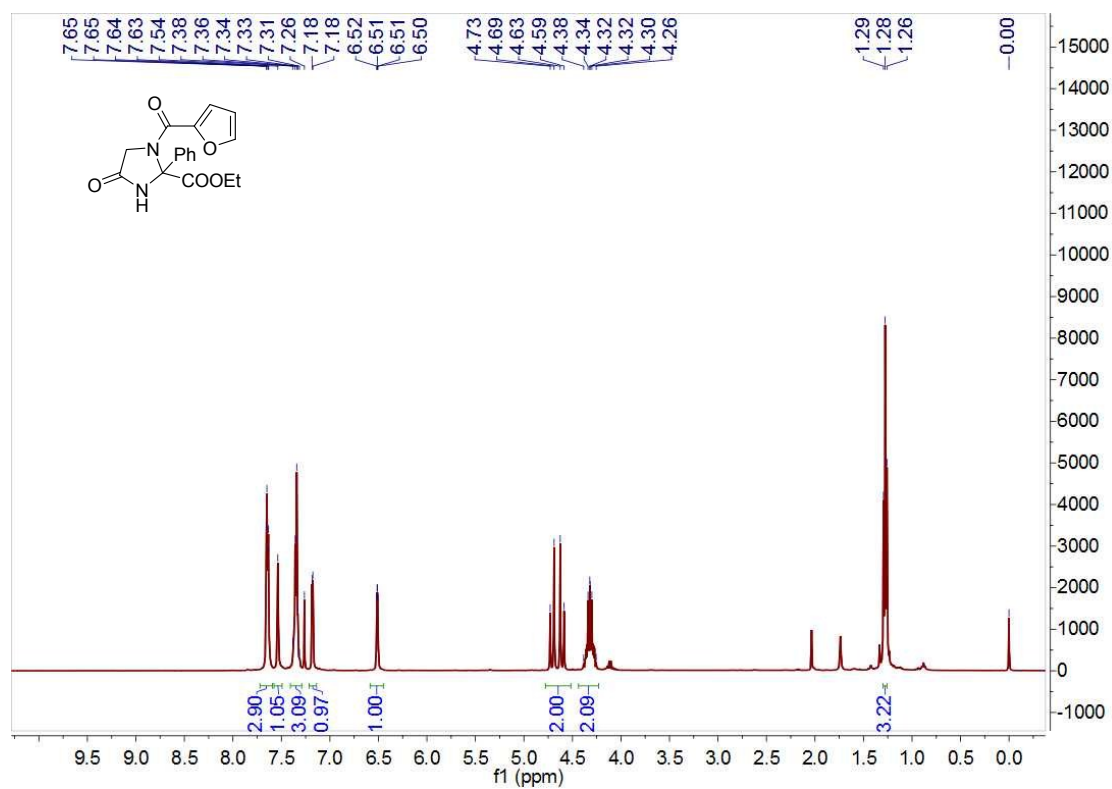
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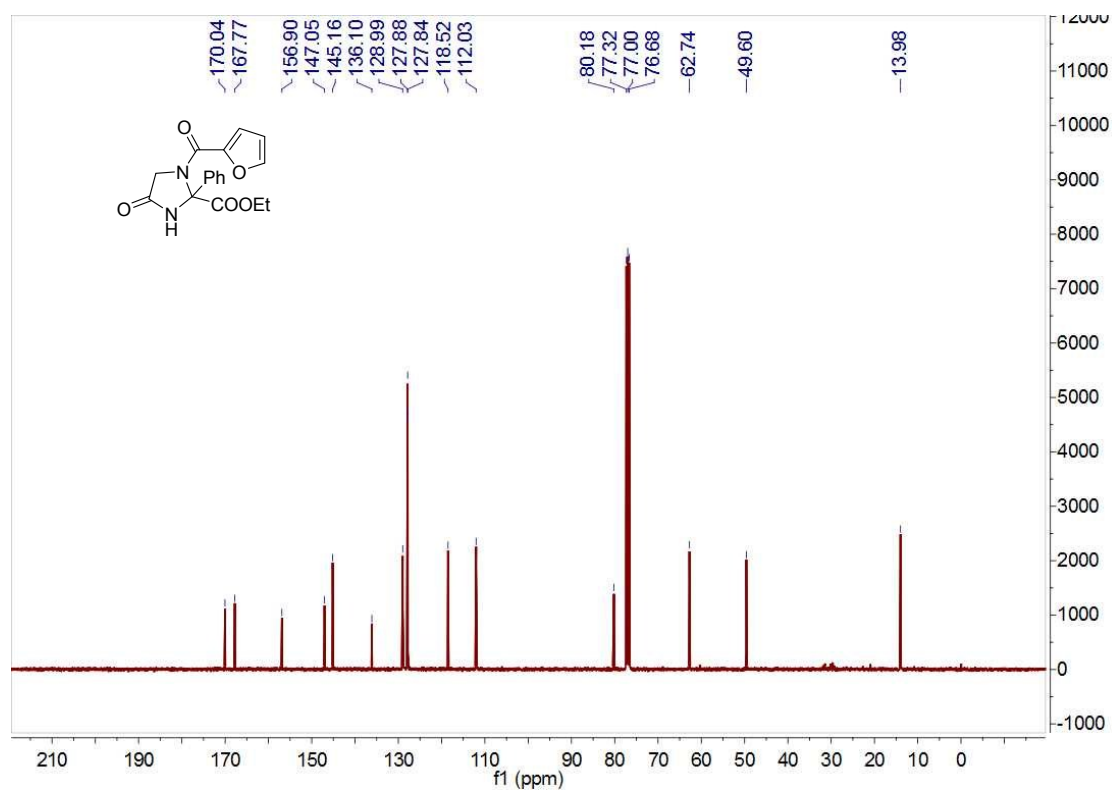
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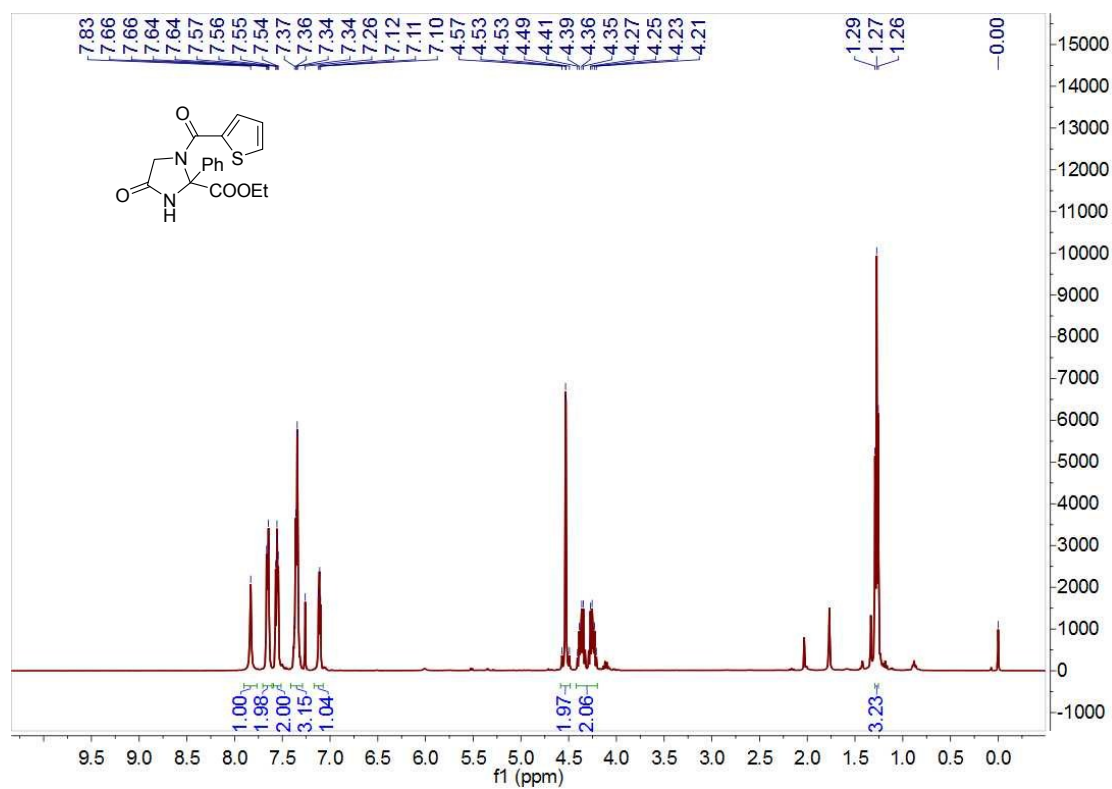
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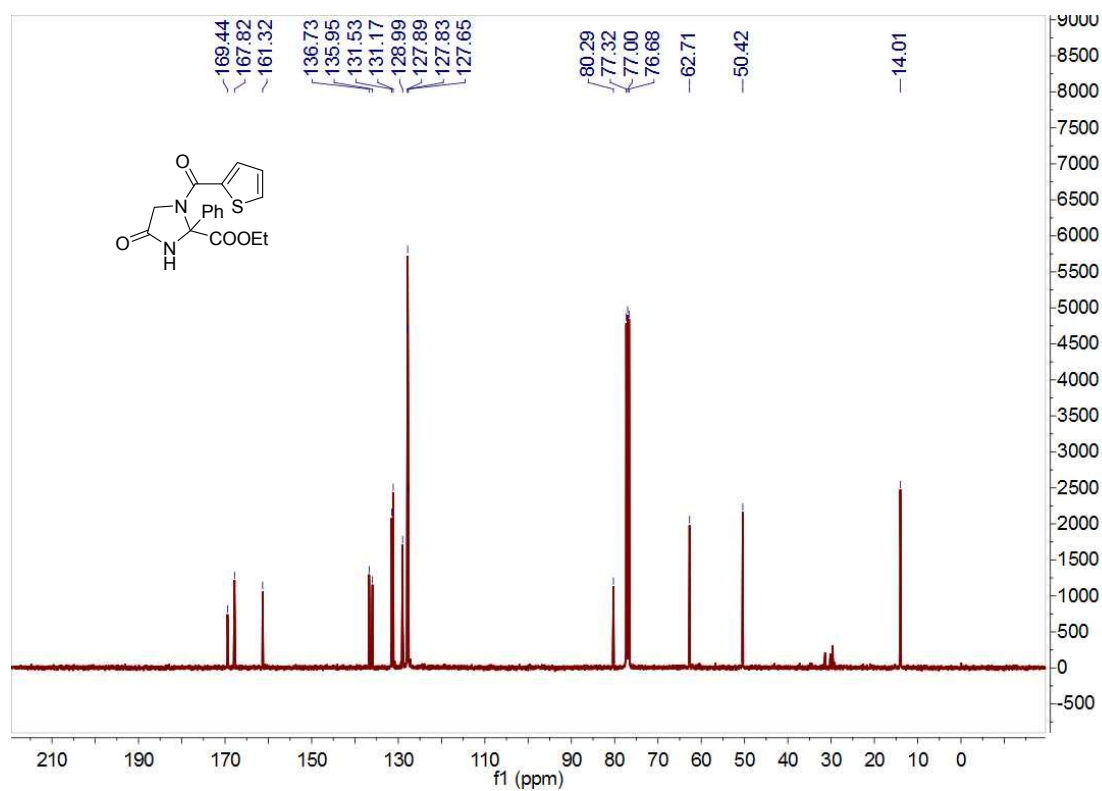
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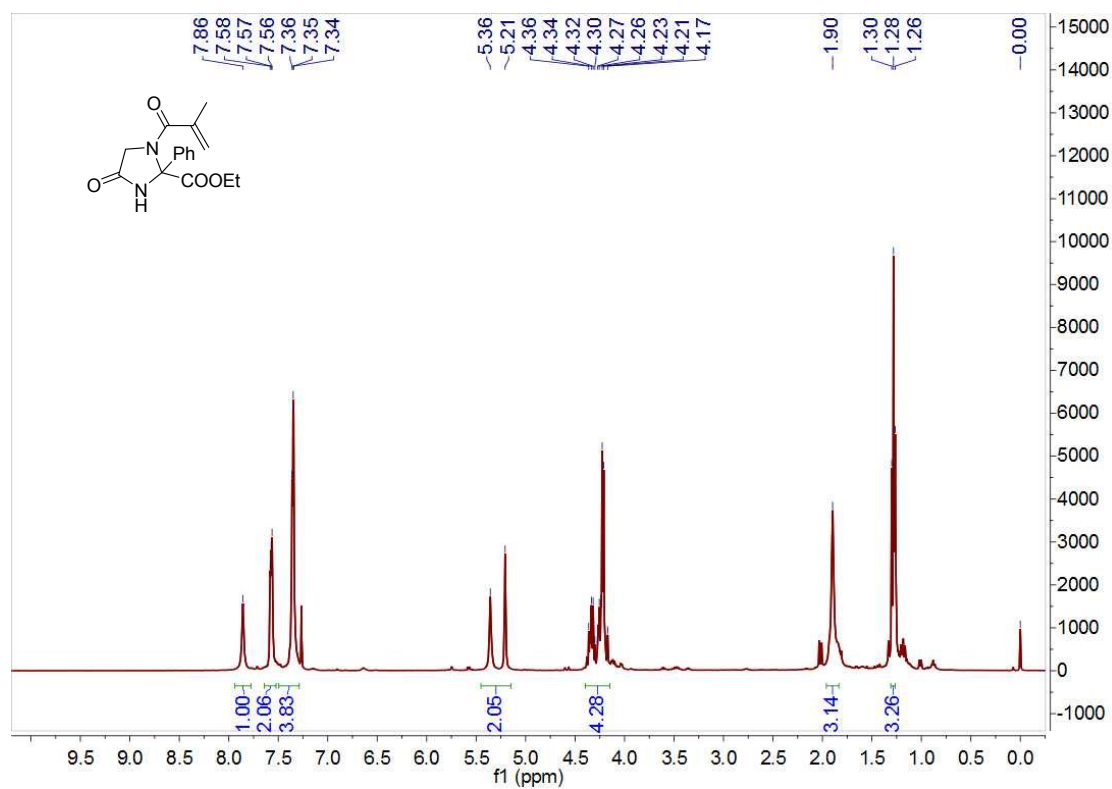
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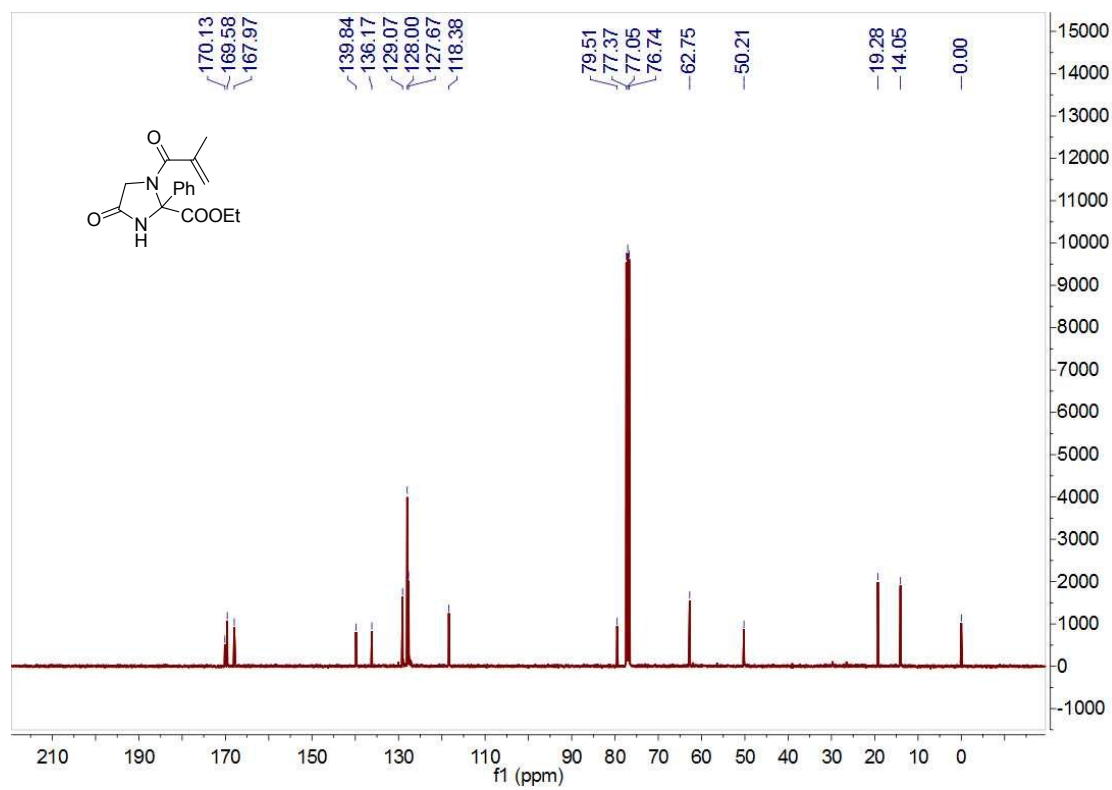
Compound 2f¹³C NMR



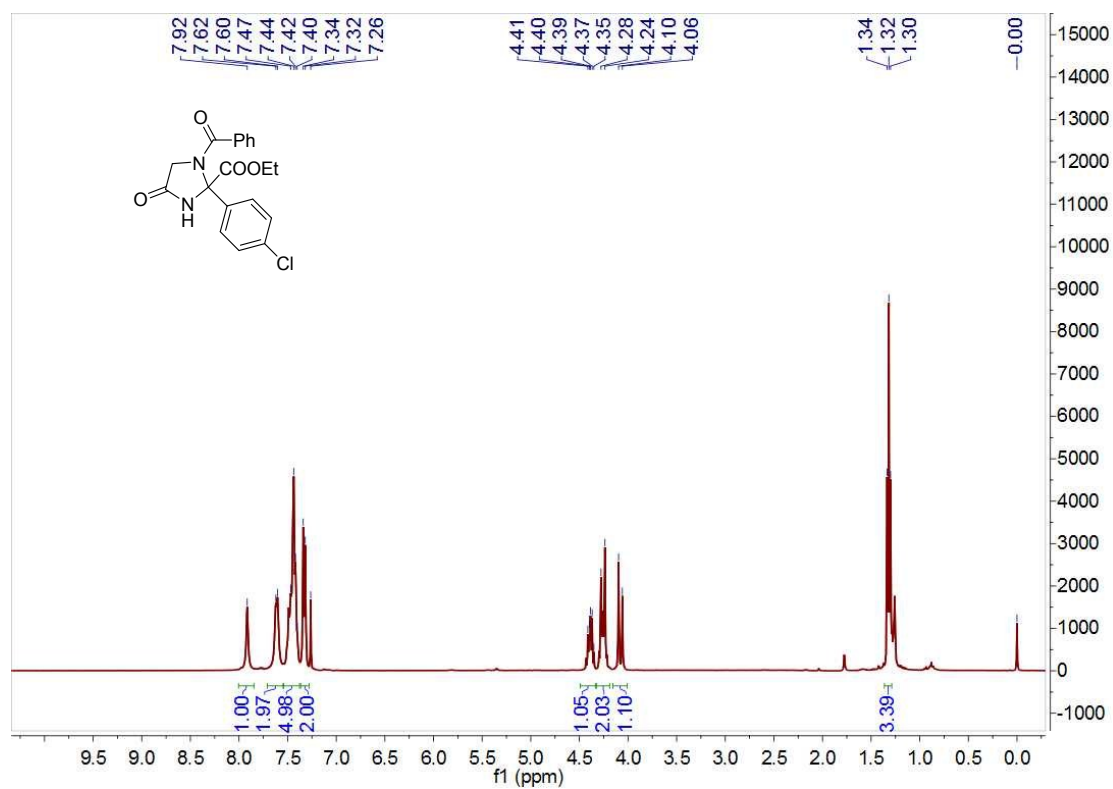
Compound 2g¹H NMR



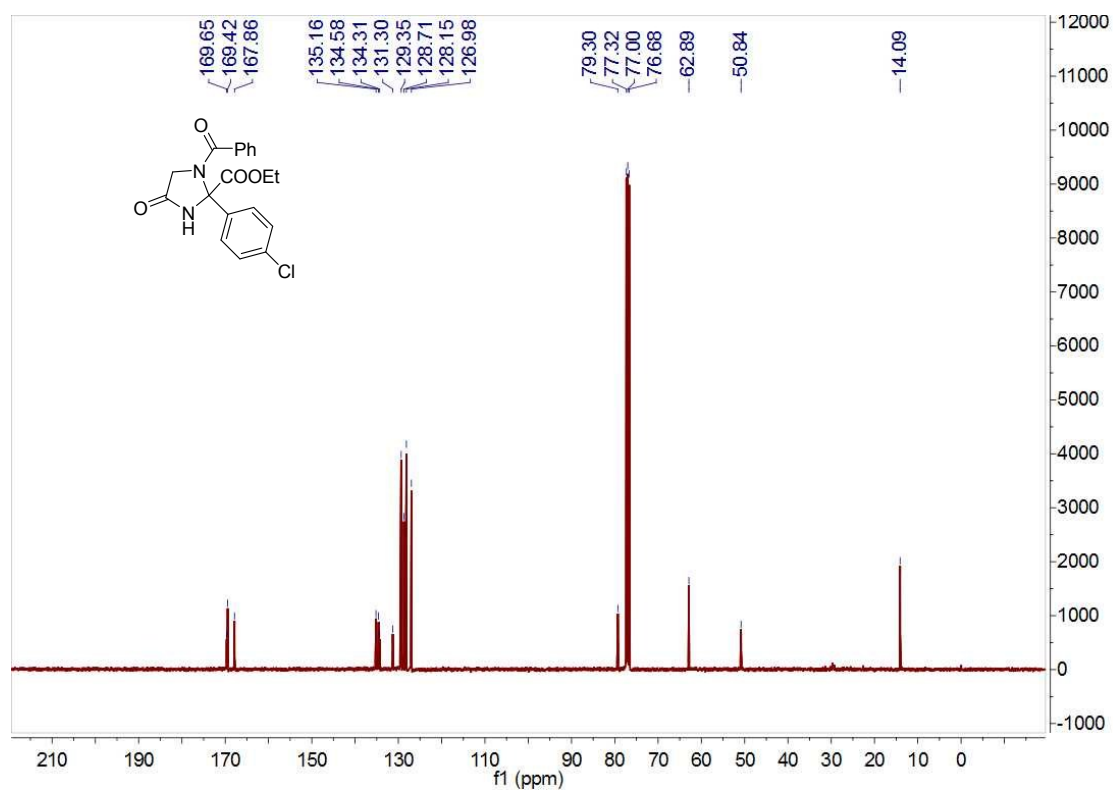
Compound 2g¹³C NMR



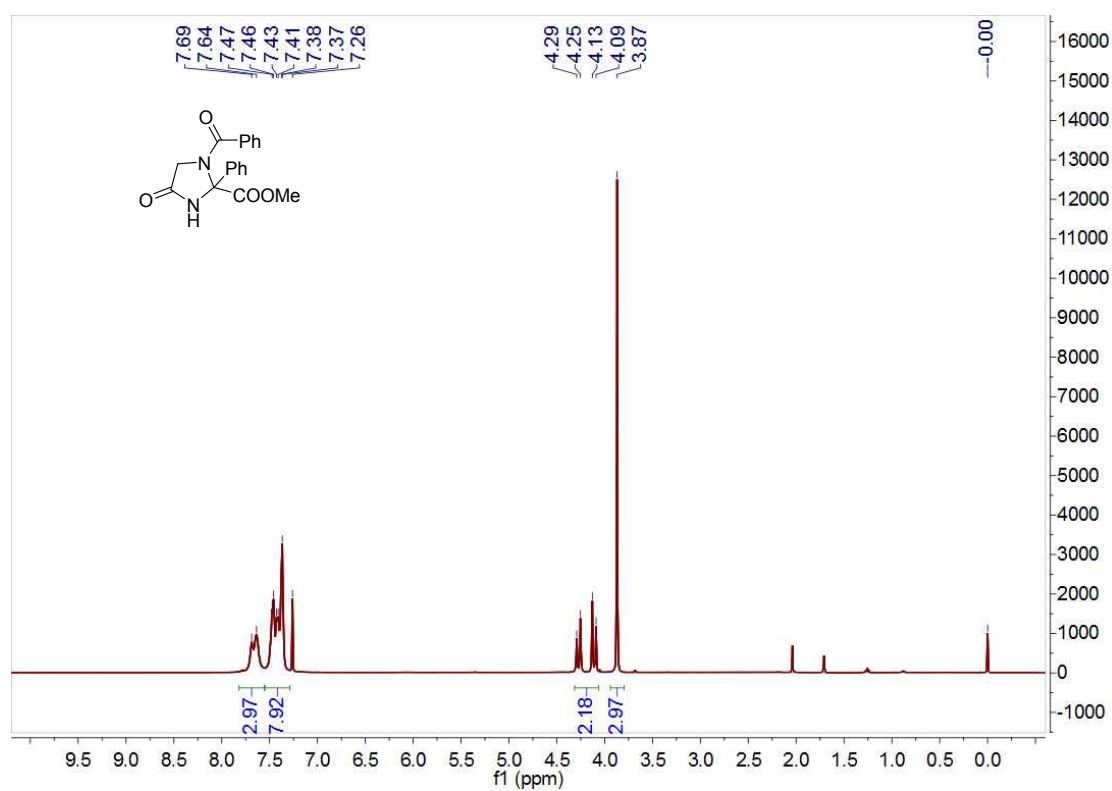
Compound 2j ^1H NMR



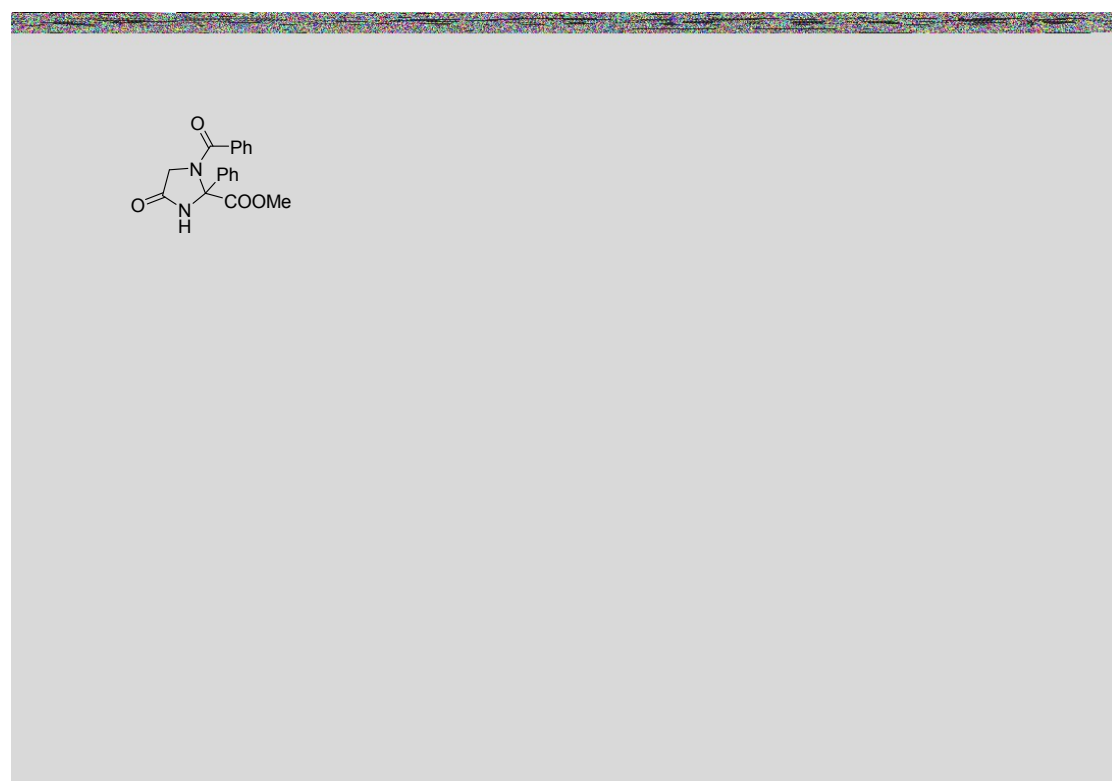
Compound 2j ^{13}C NMR



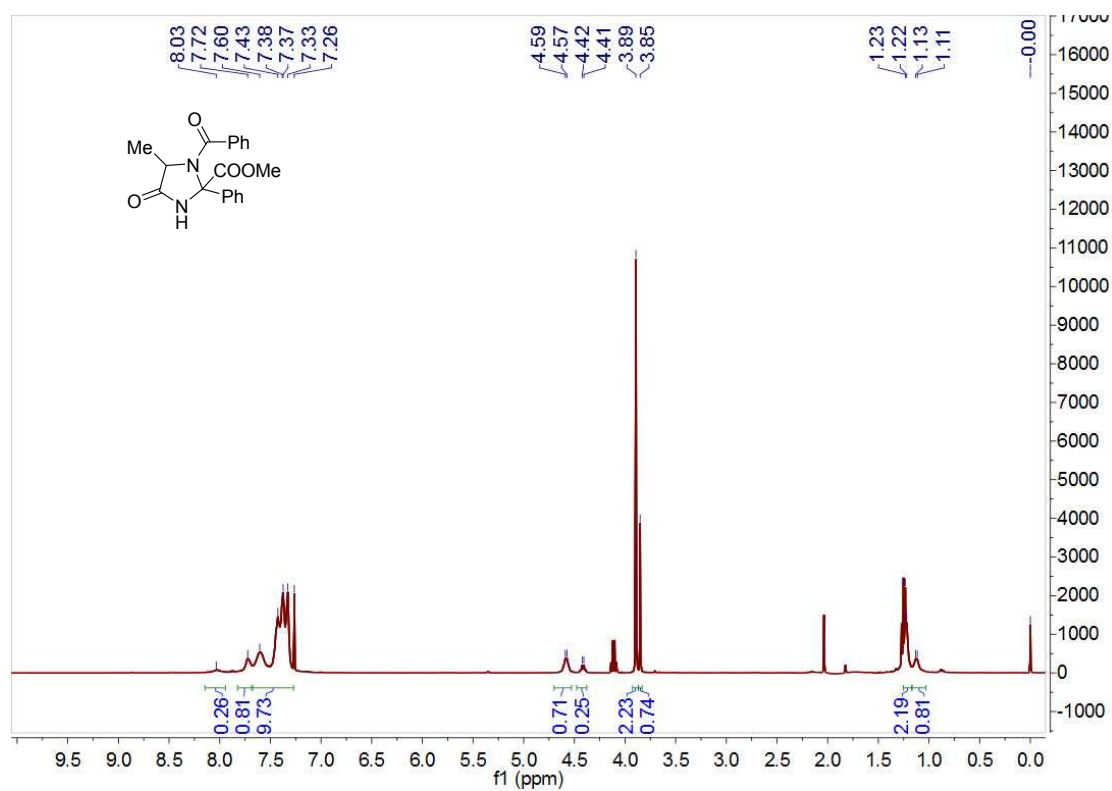
Compound 2l¹H NMR



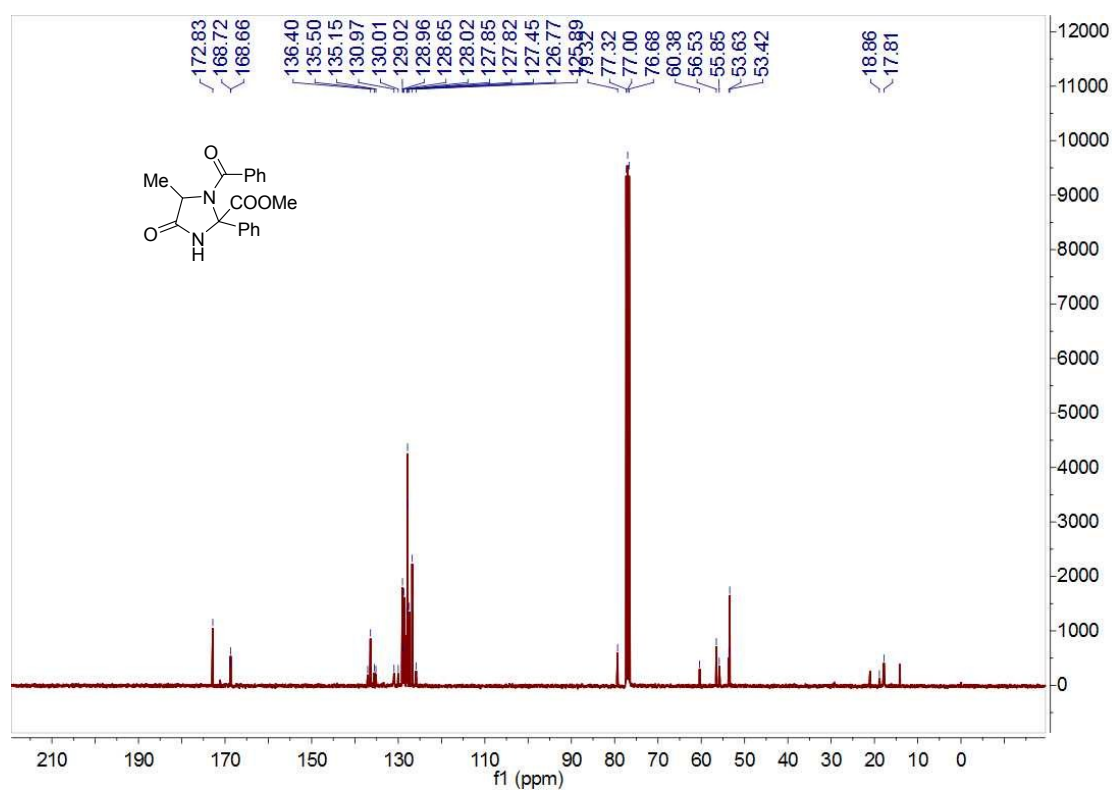
Compound 2l¹³C NMR



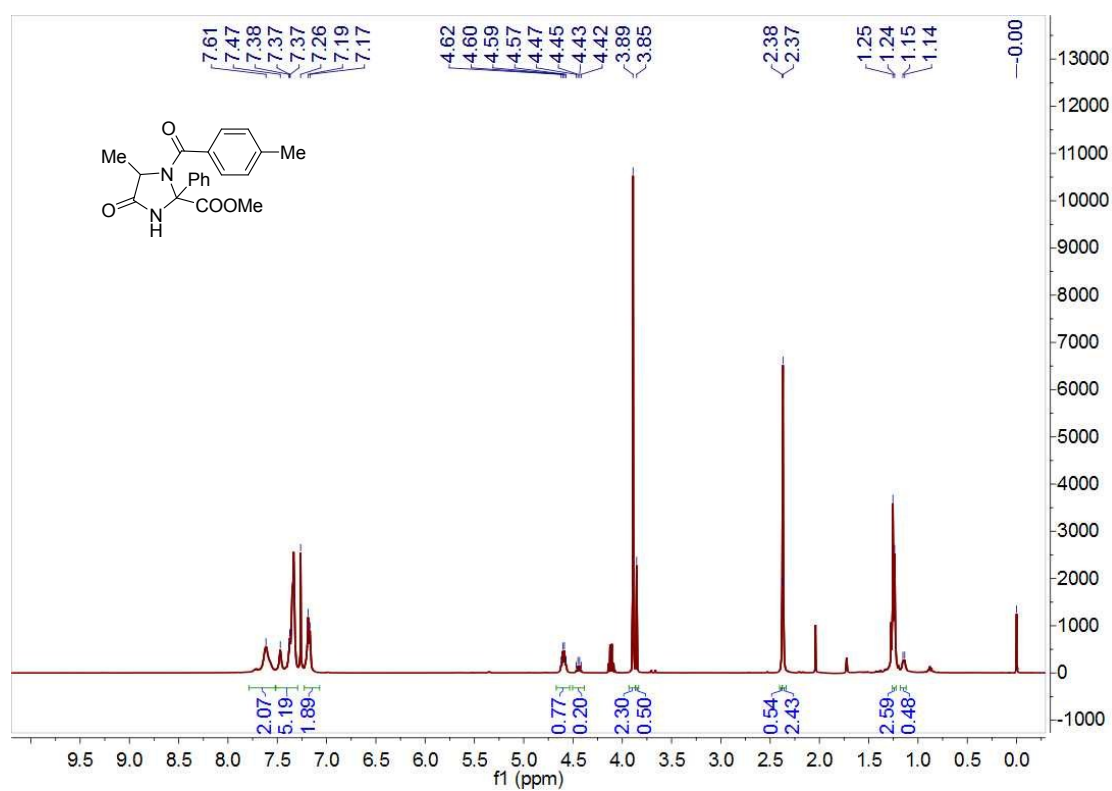
Compound 2m ¹H NMR



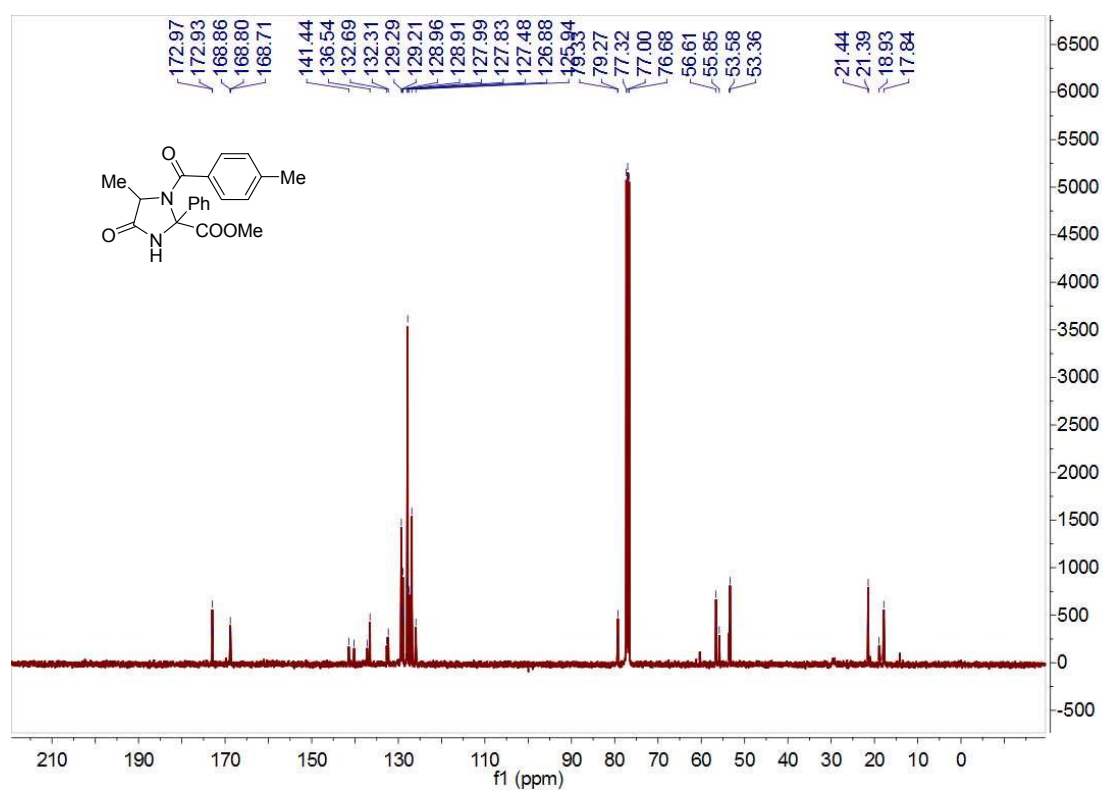
Compound 2m ¹³C NMR



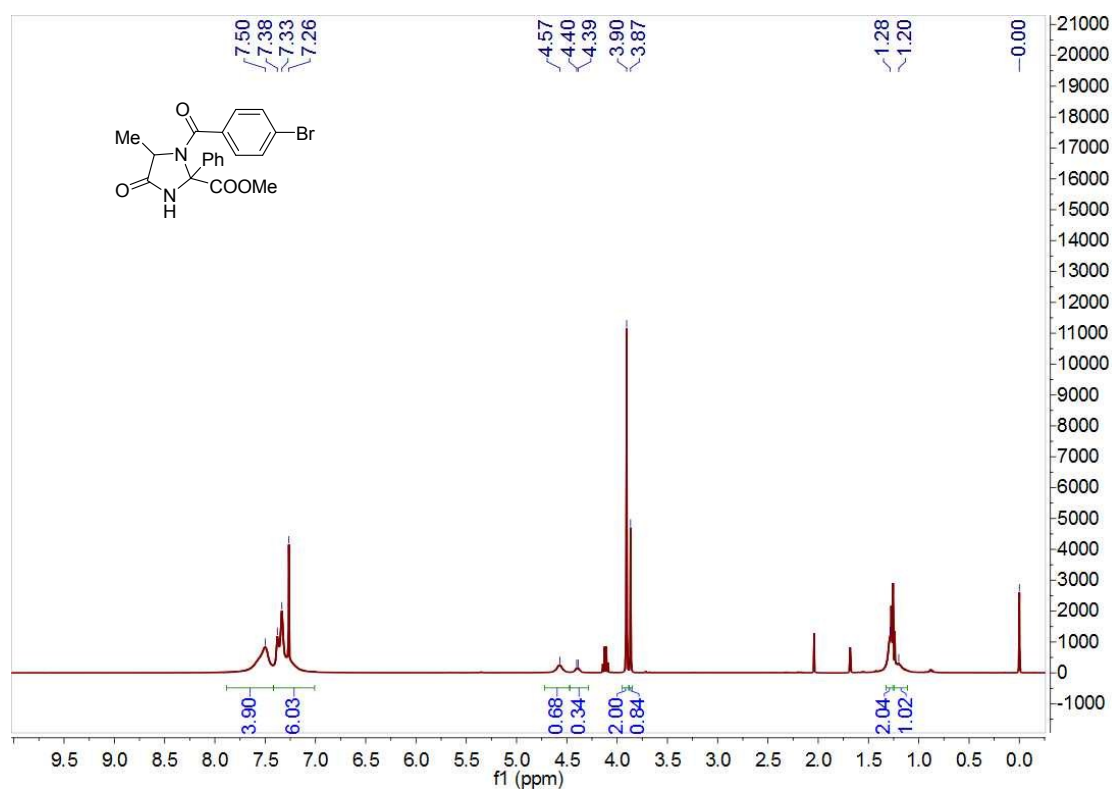
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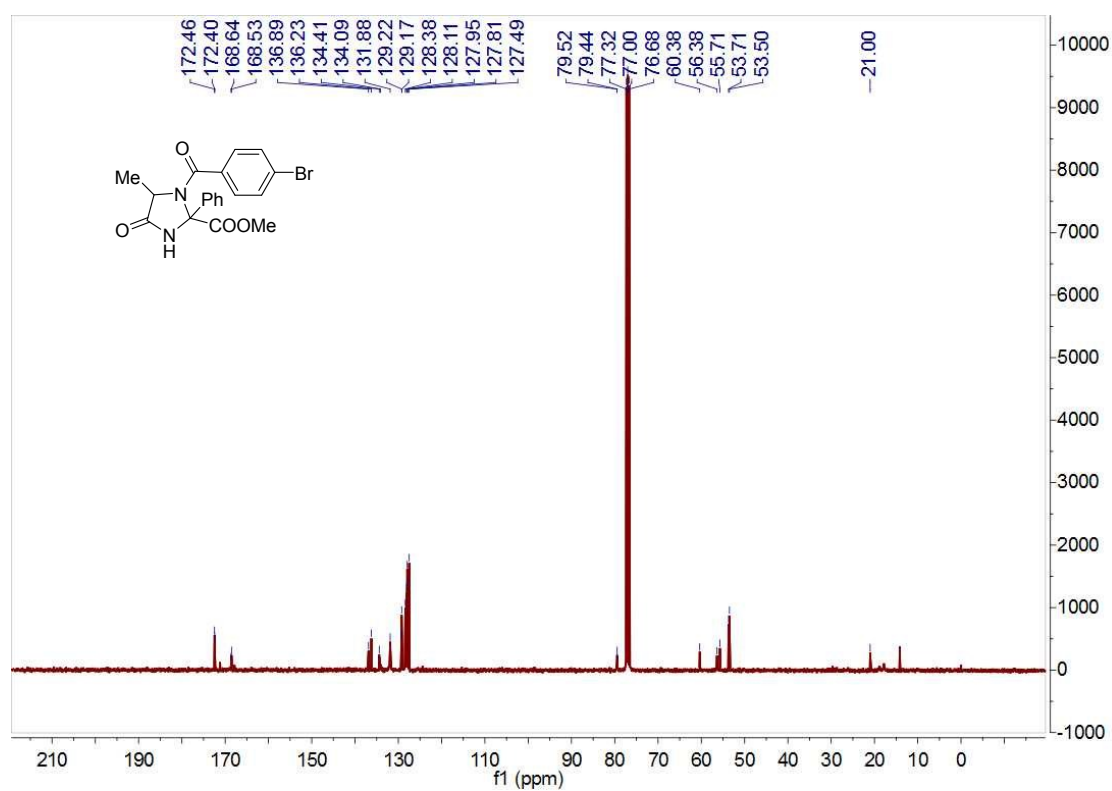
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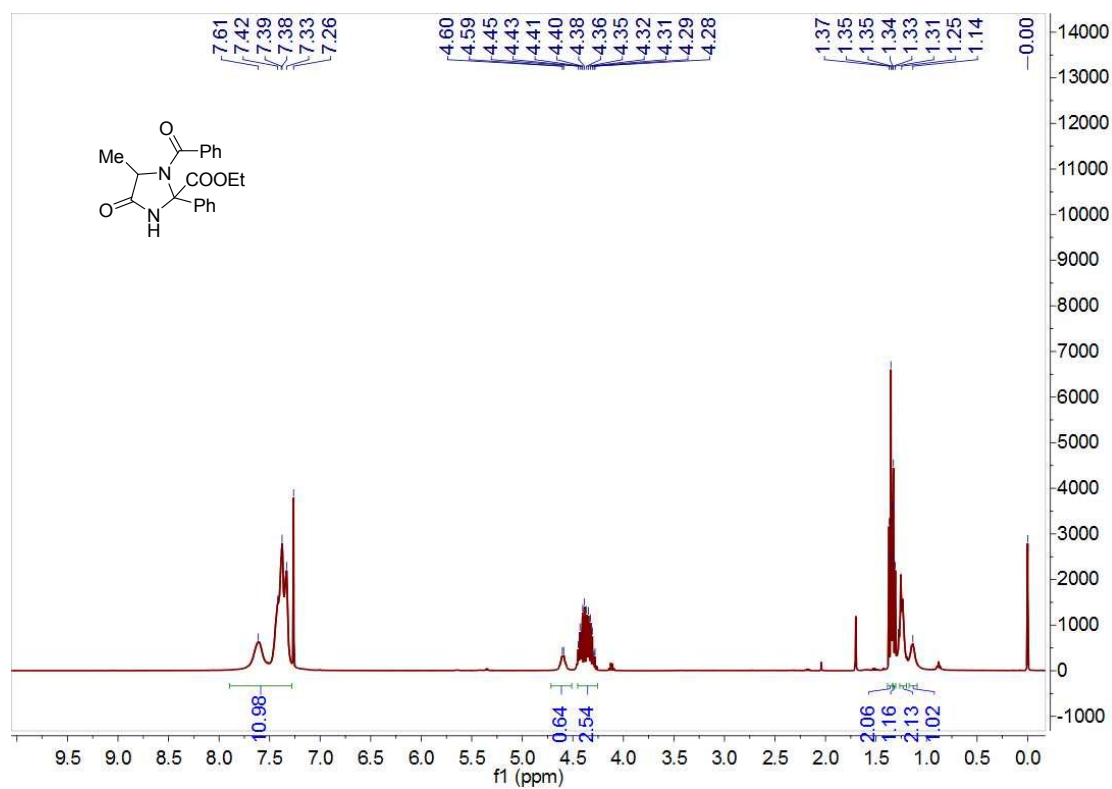
Compound 2o¹H NMR



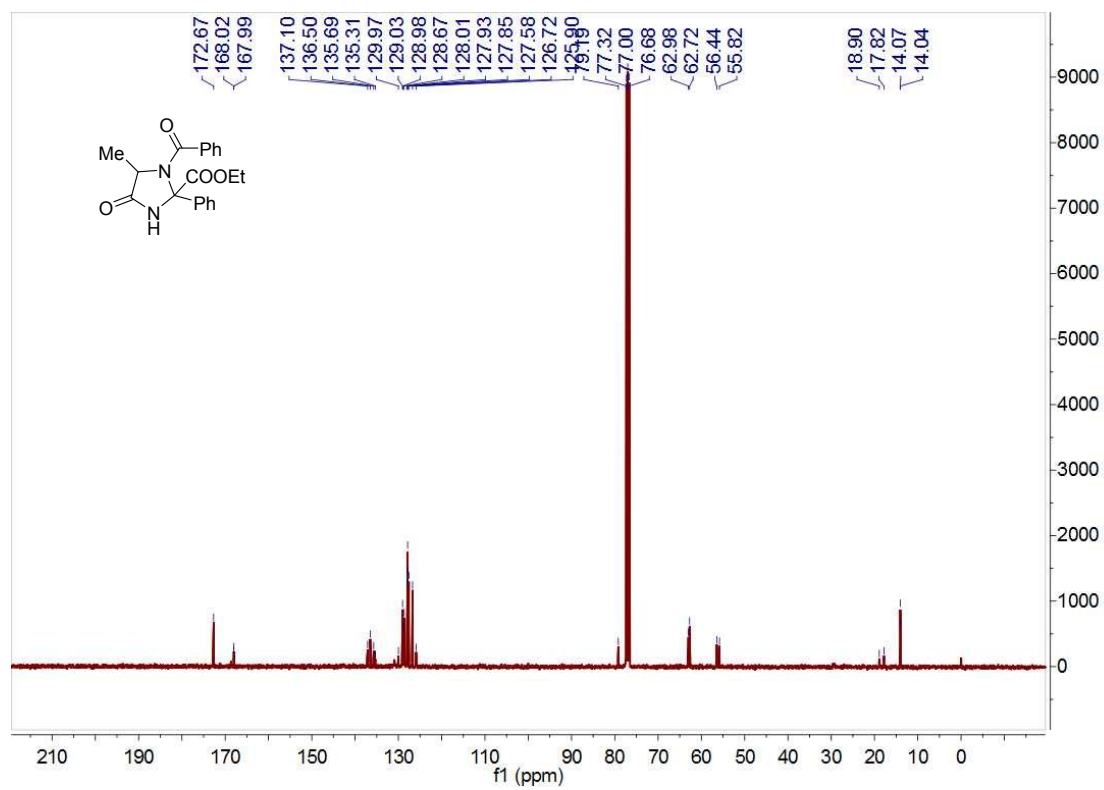
Compound 2o¹³C NMR



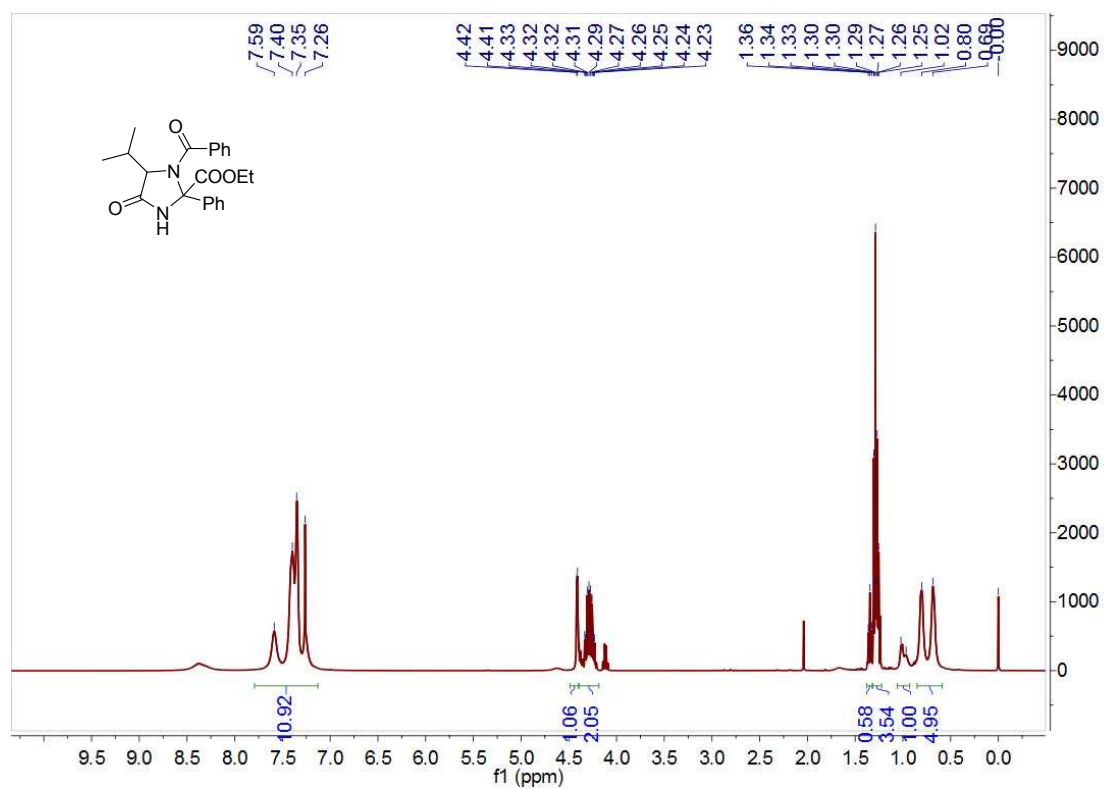
Compound 2p¹H NMR



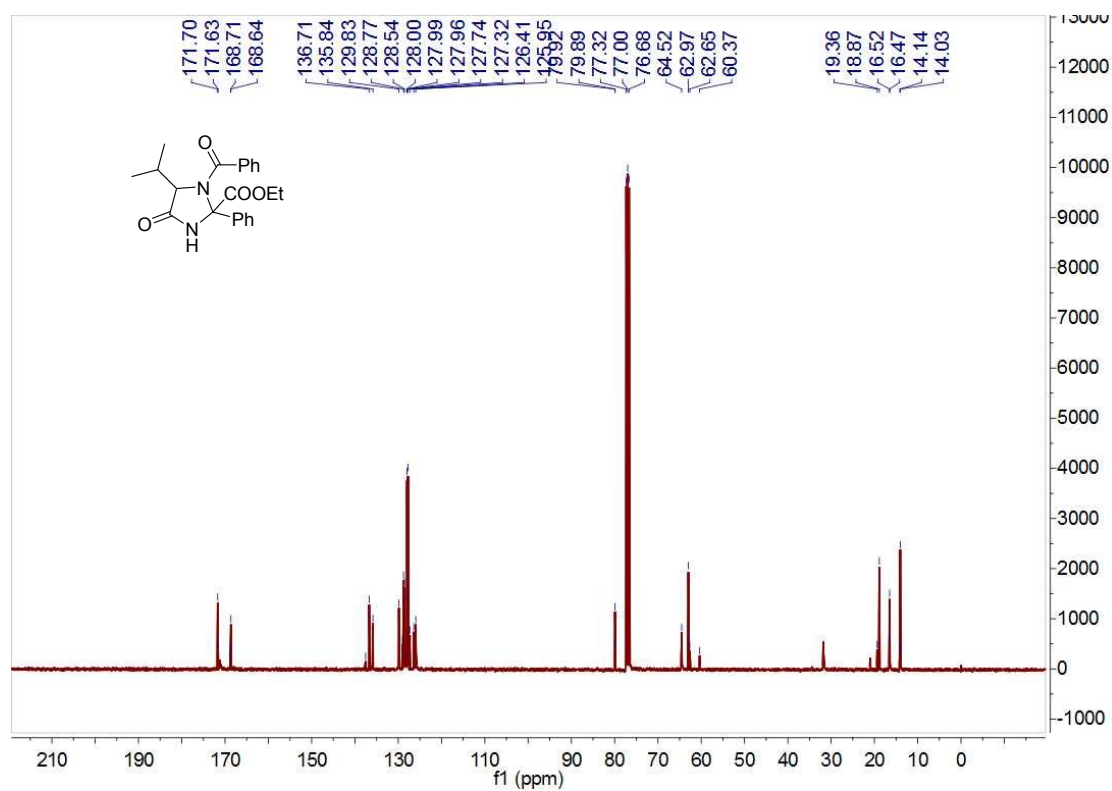
Compound 2p¹³C NMR



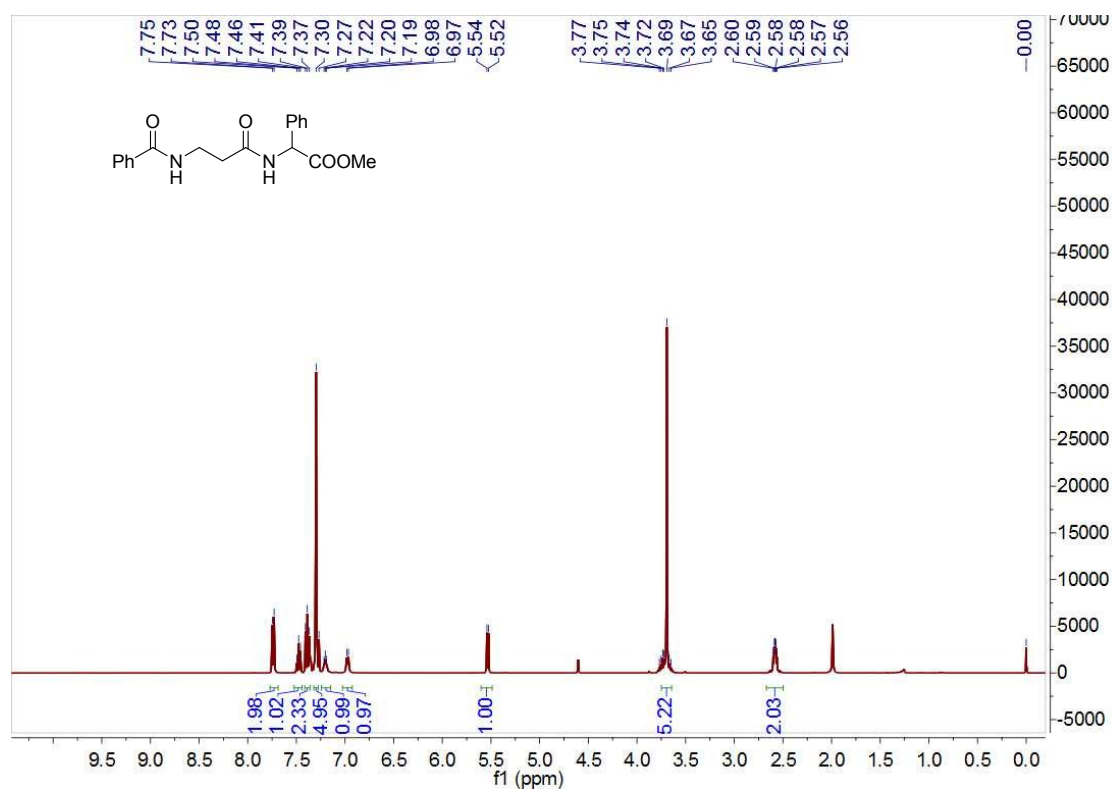
Compound 2q ¹H NMR



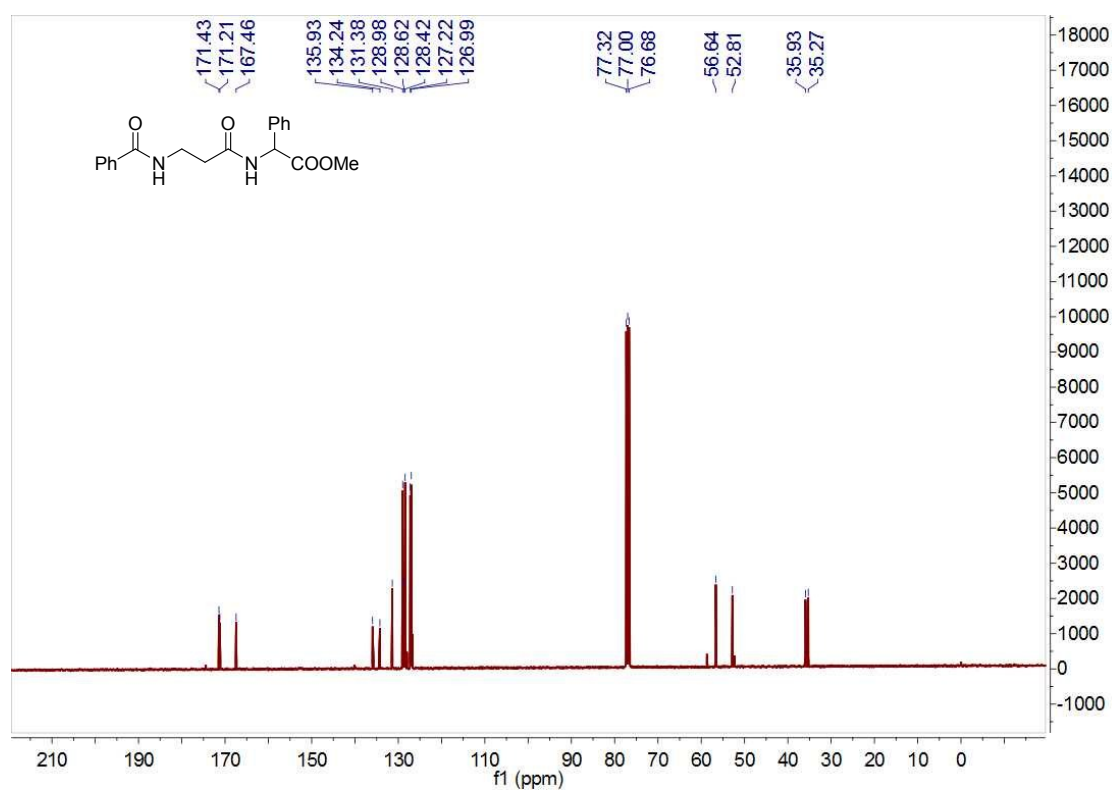
Compound 2q ¹³C NMR



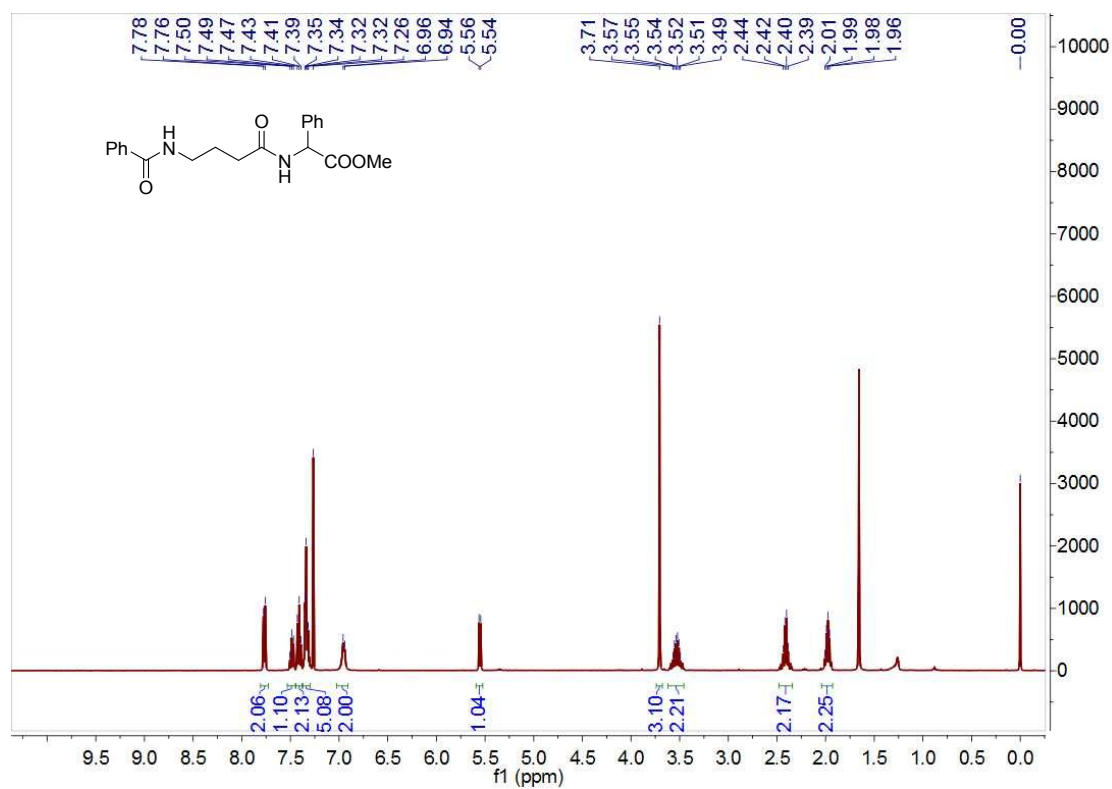
Compound 3a ¹H NMR



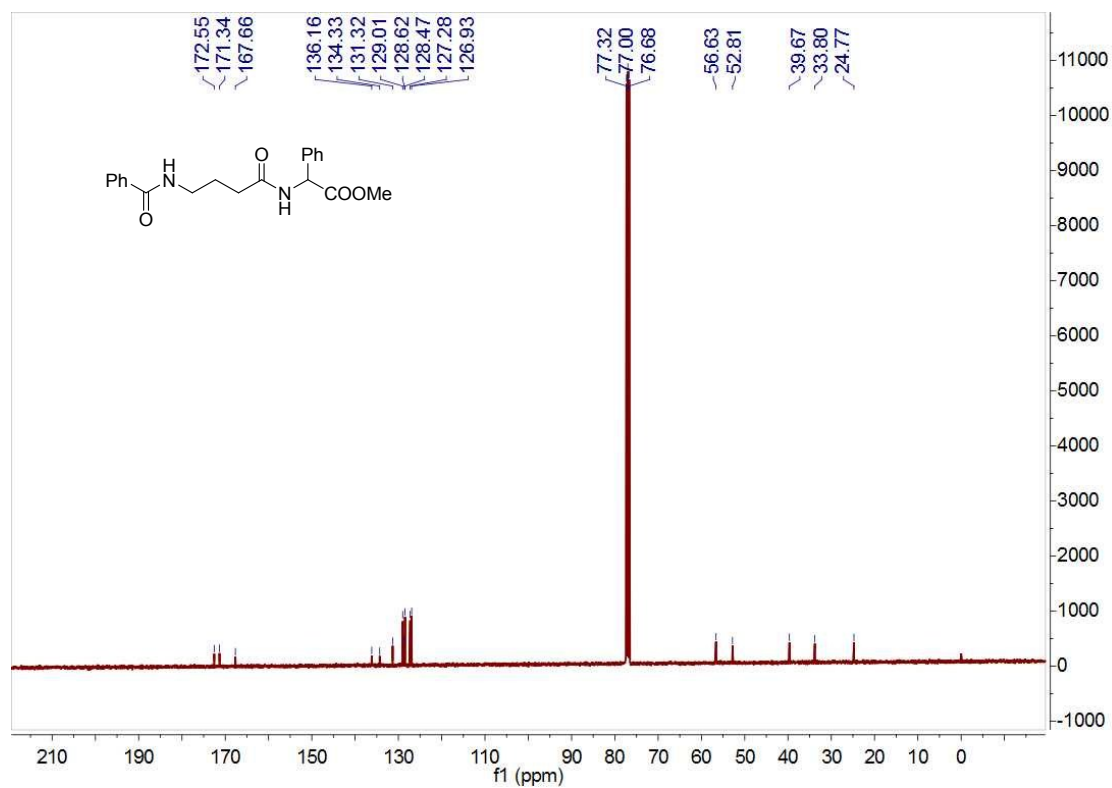
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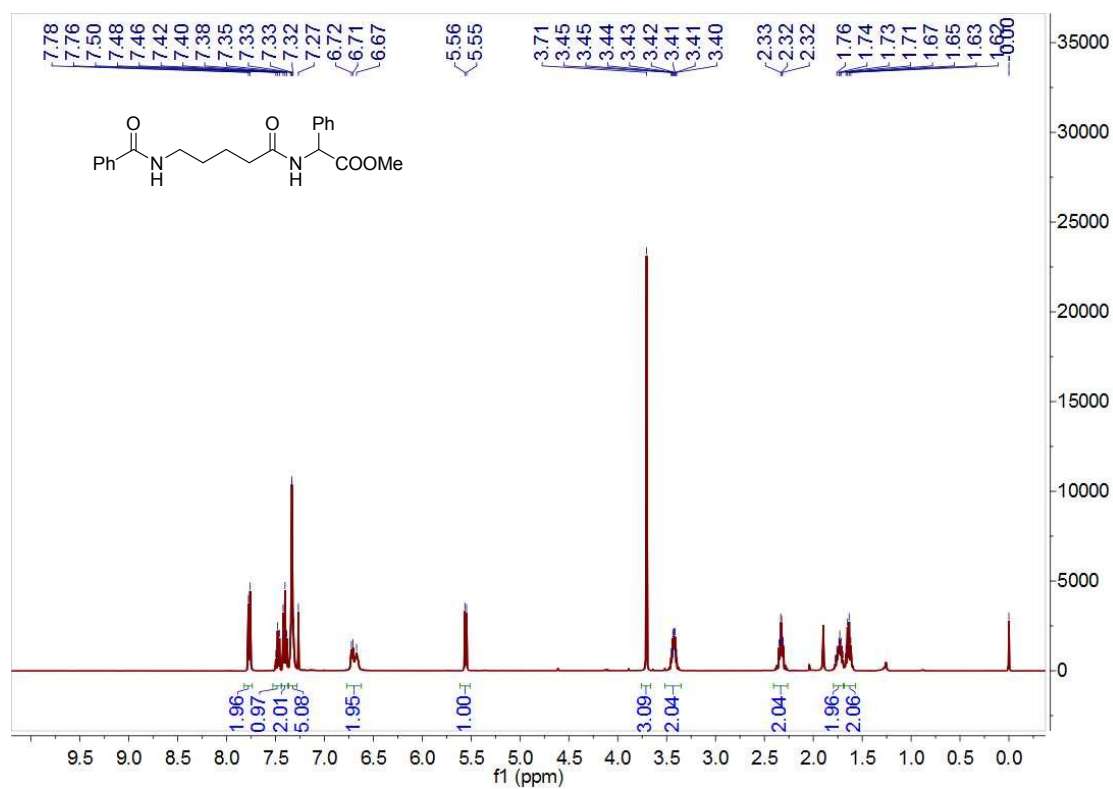
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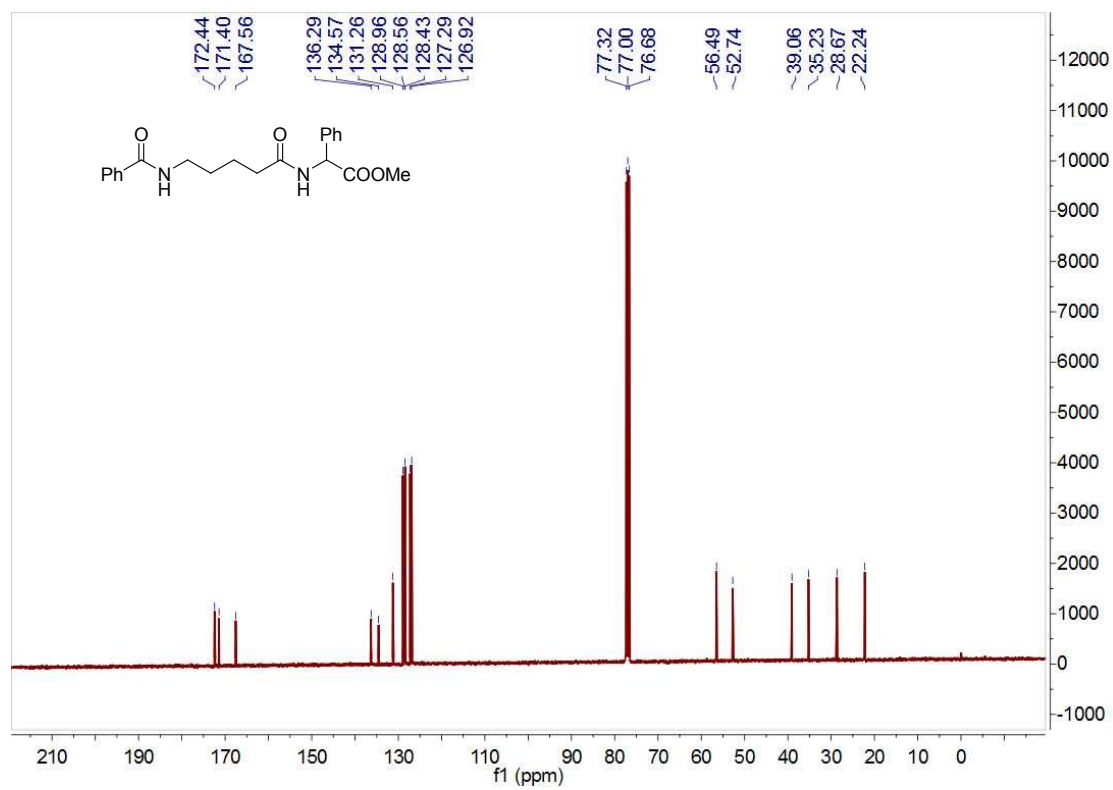
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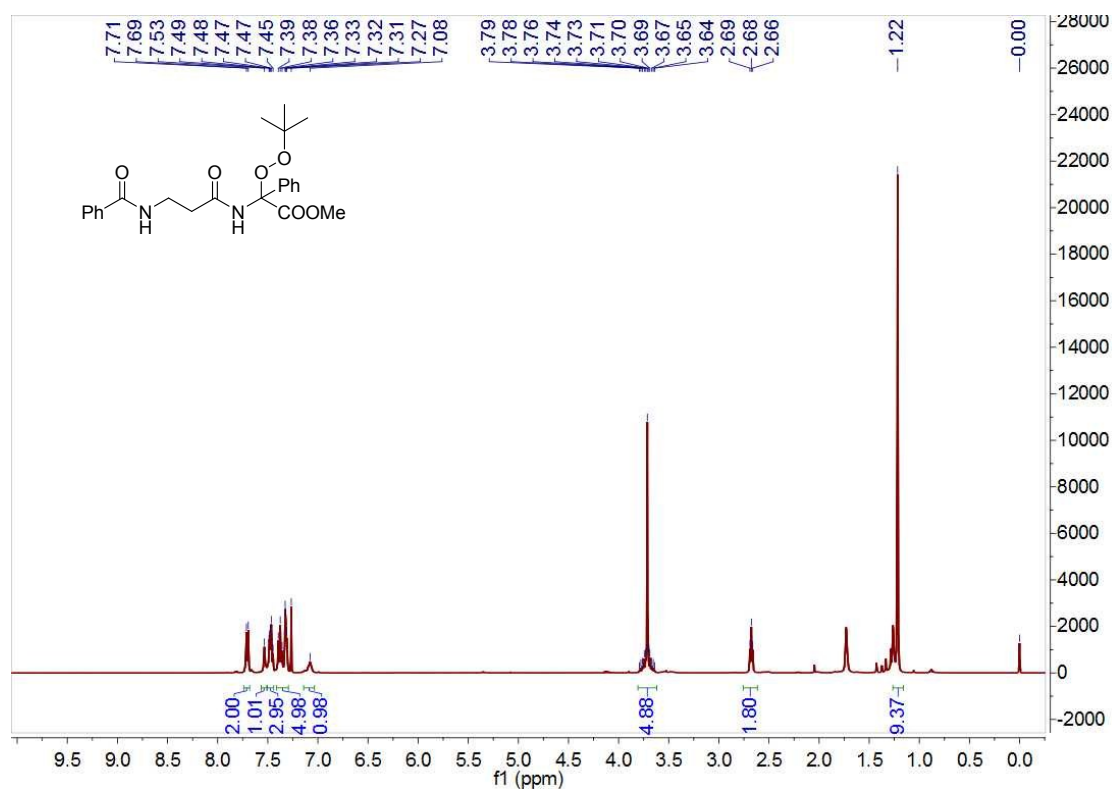
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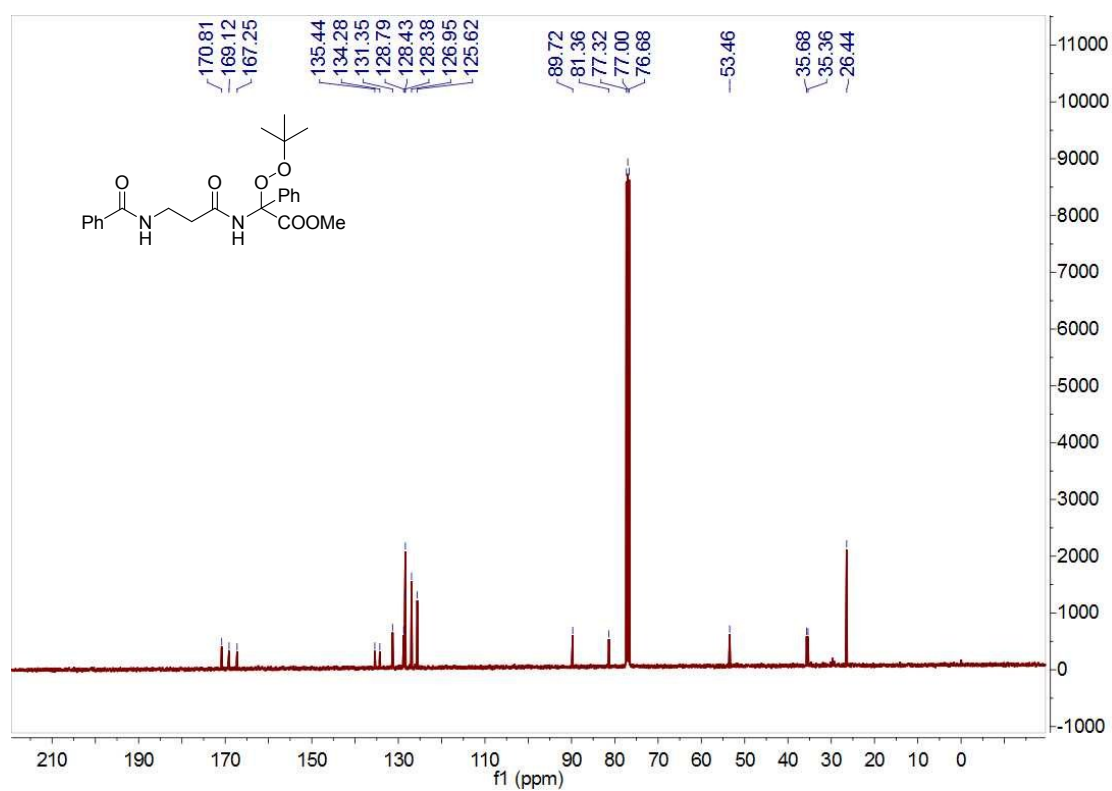
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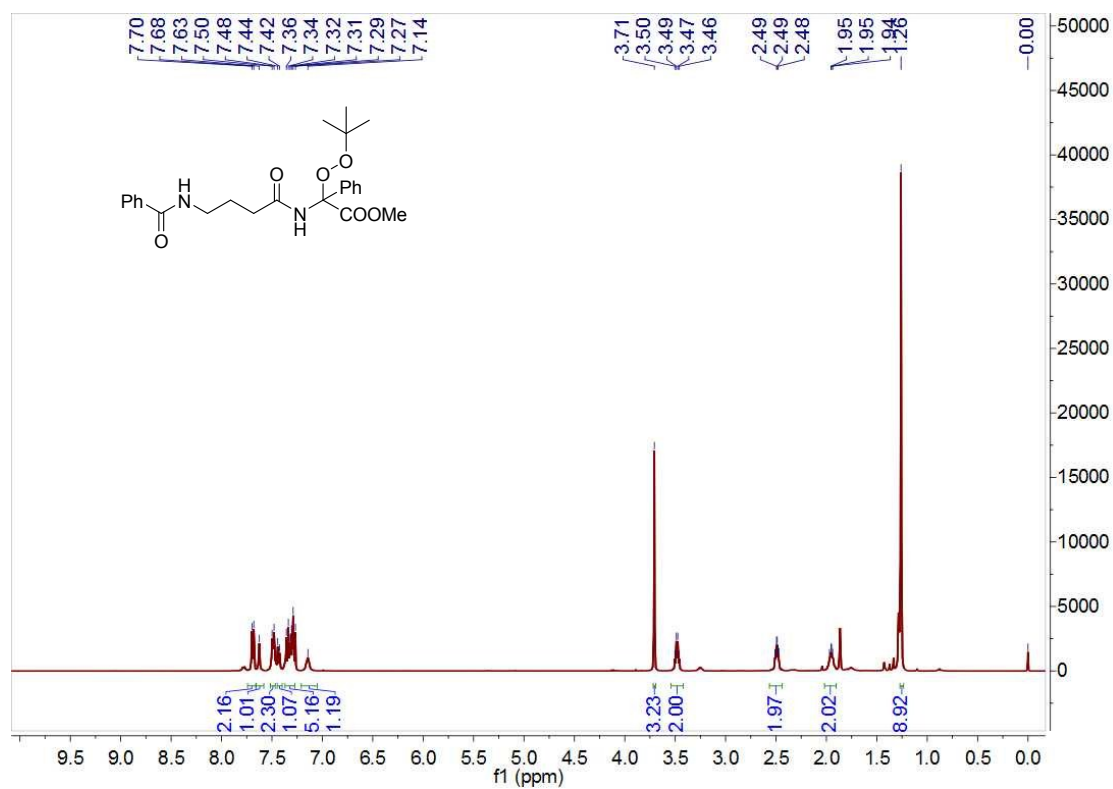
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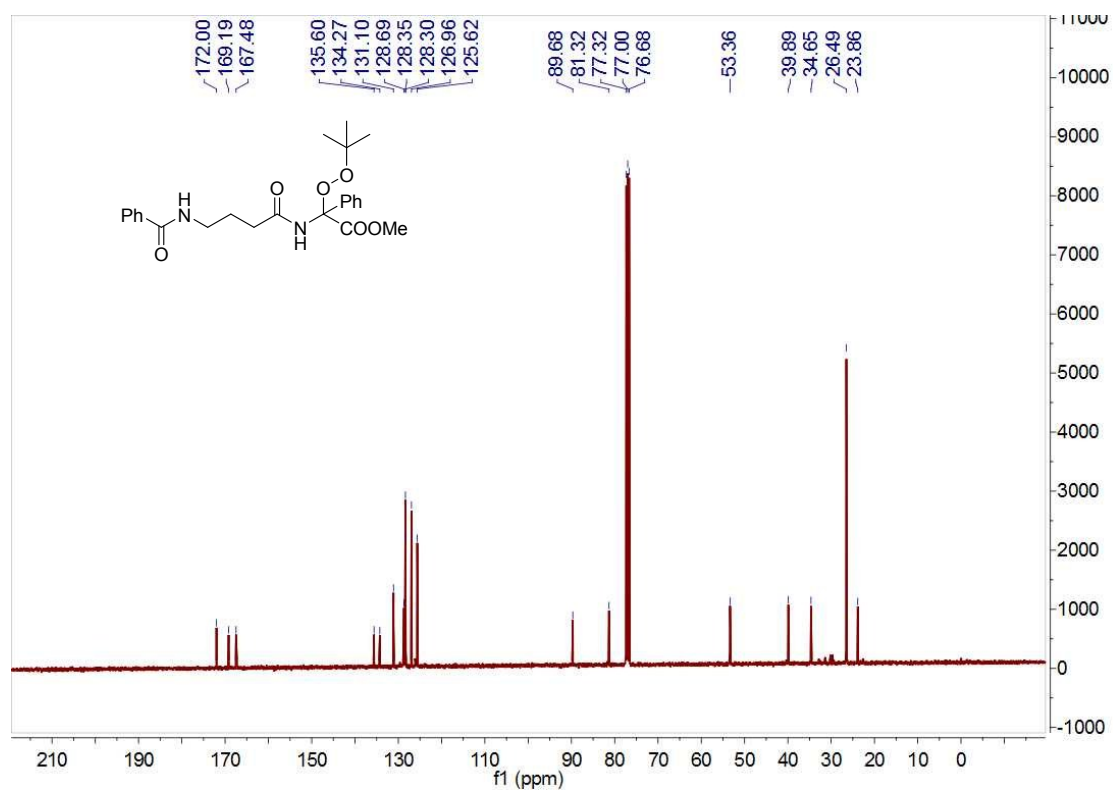
Compound 4a ¹³C NMR



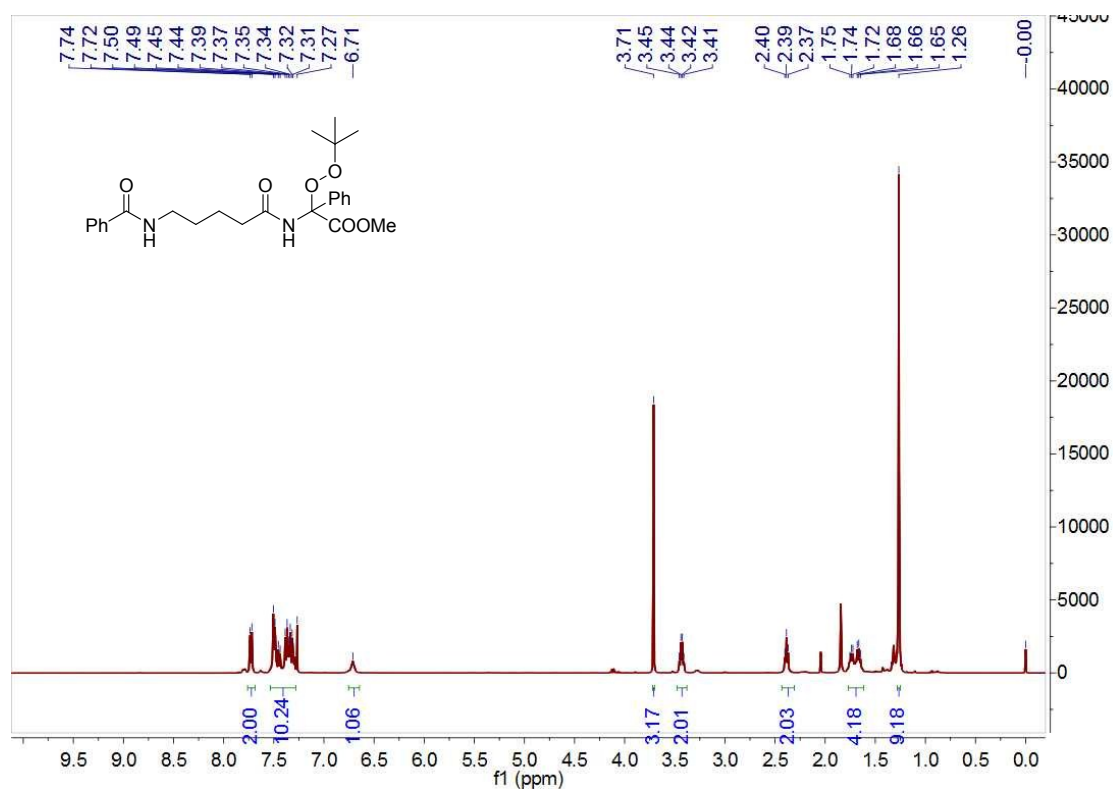
Compound 4b¹H NMR



Compound 4b¹³C NMR



Compound 4c ¹H NMR



Compound 4c ¹³C NMR

