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Mechanical metal activation for Ni-catalyzed, Mn-mediated cross-electrophile coupling between aryl and alkyl bromides

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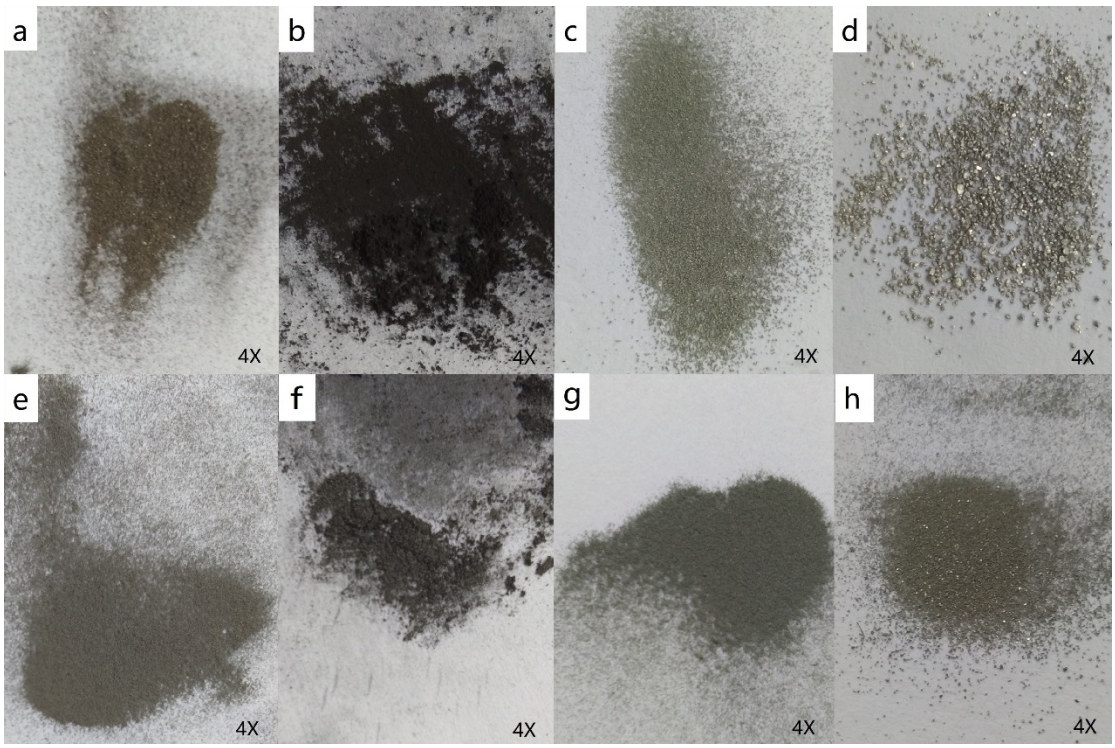
Contents

1. General information

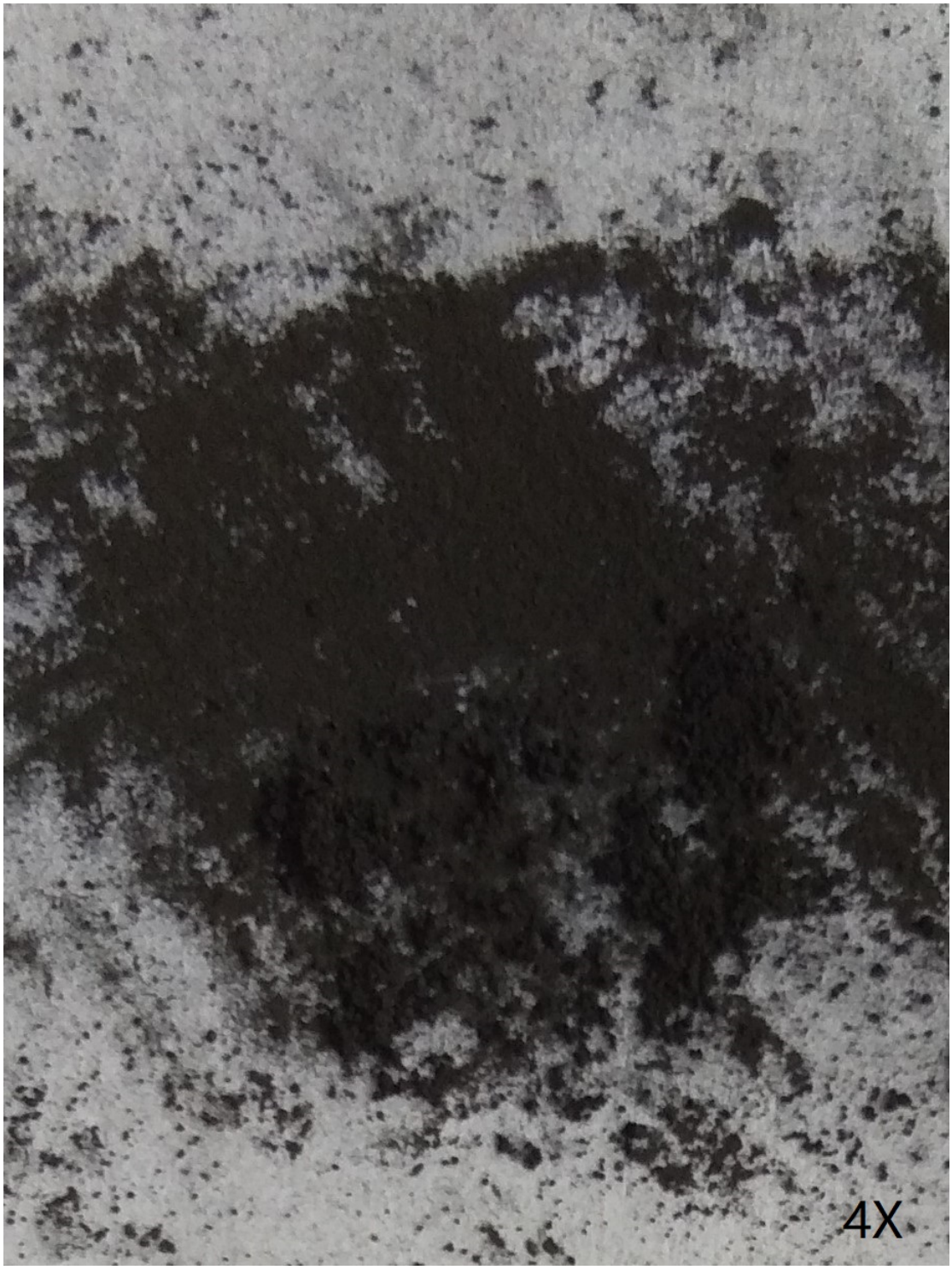
Reactions were carried out under nitrogen atmosphere unless otherwise stated. All mechanochemical reactions were conducted in a planetary ball mill (QM-3SP2) equipped with four (50mL) milling jars made of stainless steel and 8 or 16 stainless-steel balls (ø = 7 or 10 mm) at 300 rpm, 525 rpm. Chemicals obtained from commercial sources were used as received. (bpy)$NiCl_2$ was prepared according to previously reported procedure. Reaction progress was monitored by TLC. Column chromatography was performed on 300-400 mesh silica gal. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ at ambient temperature.

2. Photograph for morphology changes of metal powders before and after ball-milling



Photographs of metal powders before and after ball-mlling, a (Mn), c (Al), e (Fe) and g (Zn) before vs b (Mn), d(Al), f(Fe) and h(Zn) after ball-milling at 525rpm for 30 minutes

Stock Mn powders, (a) in Fig 1.



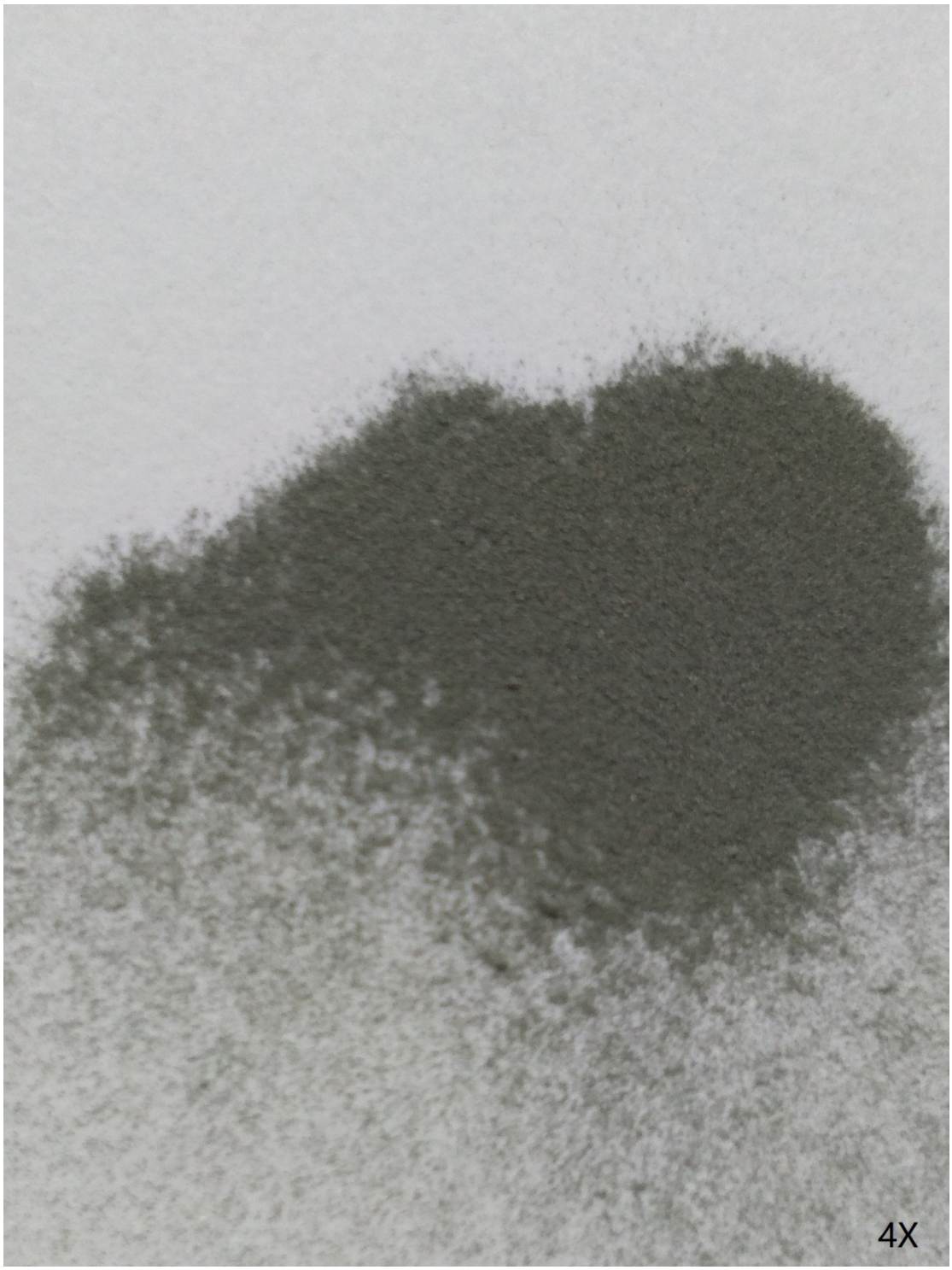
Mn powders after ball-milling, (b) in Fig 1.

Stock Al powders, (c) in Fig 1.

Al powders after ball-milling, (d) in Fig 1.

Stock Fe powders, (e) in Fig 1.

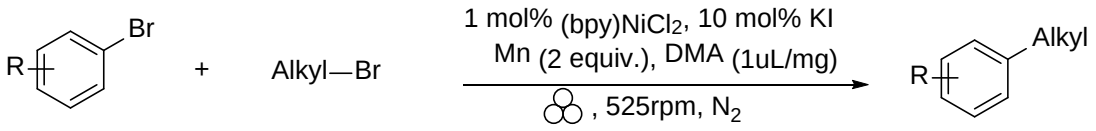
Fe powders after ball-milling, (f) in Fig 1



Stock Zn powders, (g) in Fig 1.

Zn powders after ball-milling, (h) in Fig 1

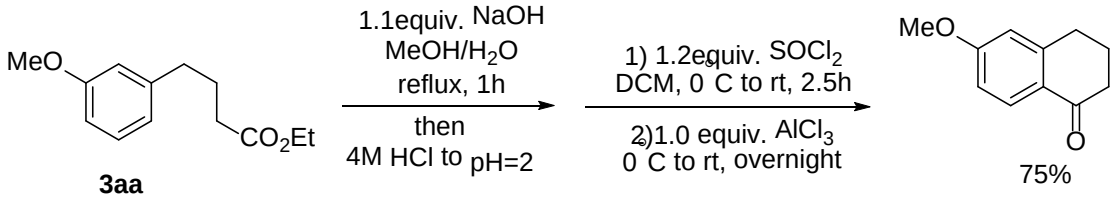
3. Typical procedure



Ball milling Procedure: A mixture of aryl bromides (1.0 mmol), alkyl bromides (1.5 mmol), (bpy)$NiCl_2$ (1 mol%), Mn power (2 equiv.) , DMA (η = 1) and KI (10 mol%) were added in a 50 mL stainless steel milling jar. The milling jar was flushed with N_2 through the lid with a gas inlet , screwed tightly and then placed on planetary ball mill at 525 rpm , using 16 stainless-steel balls (ø =7 mm) for 2-6 h. After the elapsed time, the reaction mixture was taken sample for TLC and transferred from the milling jar into a flask with aid of CH_2Cl_2 (3 x 5 mL) and then was concentrated by rotavapor to afford the crude product, which was purified by flash column chromatography on silica gel using a gradient of ethyl acetate in petroleum ether.

Procedure in solution: Under a N_2 atmosphere, to a 25 ml dry flask were added 3-bromoanisole (1.0 mmol), 4-bromobutyrate (1.5 mmol), (bpy)$NiCl_2$ (3 mol%), Mn power (2 equiv.) , KI (10 mol%) and DMA (2 mL). The mixture was stirred at room temperature for 24 h and monitored by TLC. The reaction mixture was poured into water (40 mL). This aqueous mixture was then extracted with ethyl acetate (3 × 40 mL). The organic layers were combined, washed with 50 mL of brine, and dried over anhydrous $MgSO_4$. After filtration of the organic layer, volatile materials were removed on a rotary evaporator. The crude product was purified by flash column chromatography on silica gel using a gradient of ethyl acetate in petroleum ether.

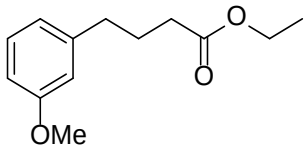
Synthesis of 6-methoxy-1-tetralone:



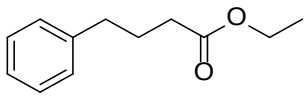
NaOH (0.36 g, 8.9 mmol) in H_2O (15 mL) were added to a solution of Ethyl 4-(3-methoxyphenyl)butanoate **3aa**(1.8 g, 8.1 mmol) in methanol (15 mL) and the mixture refluxed for 1 hous. Then, methanol was removed by evaporation under vacuum. Remaining liquid was acidified

with 4 M HCl to pH = 2. The aqueous solution was extracted with EtOAc three times, and the combined organic layer was dried over anhydrous Na_2SO_4. After filtration, solvent was evaporated by a rotary evaporator to obtain a light yellow solid, which was dried in an oven. The dried solid was dissolved in CH_2Cl_2 (15 mL). Then, $SOCl_2$ (1.2 g, 9.7 mmol) was added dropwise at 0°C. The solution was first stirred at 0°C for 30 min, then at room temperature for 2h. The mixture was cooled to 0°C again, and $AlCl_3$ (1.09 g, 8.1 mmol) was added. The reaction was then warmed to room temperature and stirred overnight. The reaction solution was poured into ice water and quenched with 10% hydrochloric acid. The mixture was diluted with EtOAc (100 mL) and washed with brine (50 mL). The organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (eluent petroleum ether/ethyl acetate: 3/1, v/v) to afford the desired product as light yellow solid（1.07 g, 75%）.

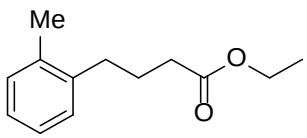
4. Product Characterization



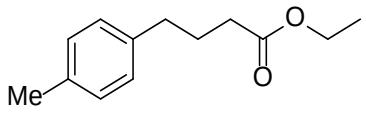
Ethyl 4-(3-methoxyphenyl)butanoate (**3aa**)[2]: pale yellow oil (0.191g, 86%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.20 (t, *J* = 8.1 Hz, 1H), 6.76 (dd, *J* = 13.7, 6.9 Hz, 3H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 3H), 2.63 (t, *J* = 7.6 Hz, 2H), 2.32 (t, *J* = 7.5 Hz, 2H), 2.01 – 1.90 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.5, 159.7, 143.1, 129.4, 120.9, 114.2, 111.3, 60.3, 55.8, 35.2, 33.6, 26.5, 14.3.



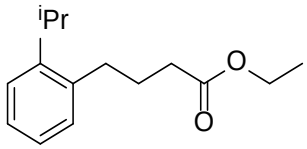
Ethyl 4-phenylbutanoate (**3ba**)[2]: pale yellow oil (0.154g, 80%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.29 (t, *J* = 7.5 Hz, 2H), 7.20 (t, *J* = 7.0 Hz, 3H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.66 (t, *J* = 7.6 Hz, 2H), 2.33 (t, *J* = 7.5 Hz, 2H), 2.03 – 1.92 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.6, 141.5, 128.6, 128.5, 126.1, 60.3, 35.2, 33.8, 26.6, 14.3.



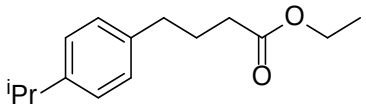
Ethyl 4-(2-tolyl)butanoate (**3ca**)[2]: pale yellow oil (0.167g, 81%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.16 – 7.10 (m, 4H), 4.15 (q, *J* = 7.1 Hz, 2H), 2.66 (t, *J* = 7.6 Hz, 2H), 2.38 (t, *J* = 7.4 Hz, 2H), 2.33 (s, 3H), 1.97 – 1.87 (m, 2H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.6, 139.8, 136.0, 130.3, 129.1, 126.2, 126.0, 60.4, 34.1, 32.7, 25.5, 19.3, 14.4.



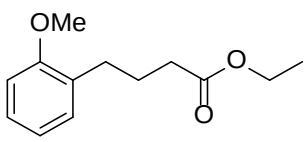
Ethyl 4-(4-tolyl)butanoate (**3da**)[3]: pale yellow oil (0.175g, 85%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.12 – 7.07 (m, 4H), 4.14 (q, *J* = 7.1 Hz, 2H), 2.63 (t, *J* = 7.6 Hz, 2H), 2.34 – 2.31 (m, 5H), 2.00 – 1.89 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.6, 138.4, 135.4, 129.1, 128.4, 60.3, 34.8, 33.7, 26.7, 21.0, 14.3.



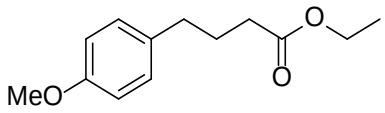
Ethyl 4-(2-isopropylphenyl)butanoate (**3ea**): pale yellow oil (0.201g, 86%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.29 (d, *J* = 7.7 Hz, 1H), 7.24 – 7.18 (m, 1H), 7.16 – 7.09 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.25 – 3.13 (m, 1H), 2.74 – 2.65 (m, 2H), 2.39 (t, *J* = 7.3 Hz, 2H), 1.97 – 1.86 (m, 2H), 1.30 – 1.25 (m, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.5, 146.7, 138.3, 129.5, 126.6, 125.7, 125.4, 60.3, 34.1, 32.2, 28.6, 26.8, 24.1, 14.3. HRMS (EI): *m/z* calcd for $C_{15}H_{22}O_2$: 234.1620; found: 234.1618.



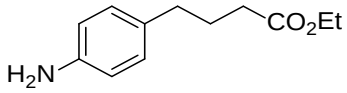
Ethyl 4-(4-isopropylphenyl)butanoate (**3fa**)[4]: pale yellow oil (0.197g, 84%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (dd, *J* = 17.4, 8.0 Hz, 4H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.93 – 2.86 (m, 1H), 2.63 (t, *J* = 7.6 Hz, 2H), 2.33 (t, *J* = 7.5 Hz, 2H), 2.02 – 1.91 (m, 2H), 1.26 (t, *J* = 7.3 Hz, 9H). ^{13}C-NMR (150 MHz, $CDCl_3$) δ (ppm): 173.7, 146.6, 138.8, 128.5, 126.5, 60.3, 34.8, 33.9, 33.8, 26.7, 24.2, 14.4.



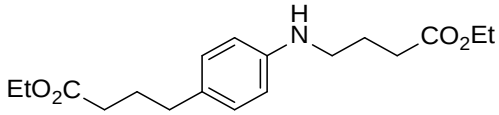
Ethyl 4-(2-methoxyphenyl)butanoate (**3ga**)[5]: pale yellow oil (0.186g, 84%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.21 – 7.12 (m, 2H), 6.90 – 6.83 (m, 2H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.82 (s, 3H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.33 (t, *J* = 7.6 Hz, 2H), 1.97 – 1.89 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 172.7, 156.5, 129.0, 128.8, 126.2, 119.4, 109.2, 59.2, 54.2, 32.9, 28.6, 24.1, 13.3.



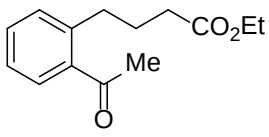
Ethyl 4-(4-methoxyphenyl)butanoate (**3ha**)[3]: pale yellow oil (0.190g, 86%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.09 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.78 (s, 3H), 2.59 (t, *J* = 7.6 Hz, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 1.99 – 1.86 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$) δ (ppm): 173.5, 157.9, 133.5, 129.4, 113.8, 77.3, 77.1, 76.9, 60.2, 55.2, 34.2, 33.6, 26.8, 14.3.



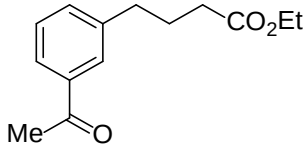
Ethyl 4-(4-aminophenyl)butanoate (**3ia**)[6]: pale yellow oil (0.068g, 33%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 6.96 (d, *J* = 8.2 Hz, 2H), 6.62 (d, *J* = 8.3 Hz, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.38 (s, 2H), 2.54 (t, *J* = 7.6 Hz, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 1.95 – 1.84 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). δ 173.7, 144.5, 131.4, 129.3, 115.3, 60.2, 34.3, 33.7, 26.9, 14.3. ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.7, 144.5, 131.4, 129.3, 115.3, 60.2, 34.3, 33.7, 26.9, 14.3.



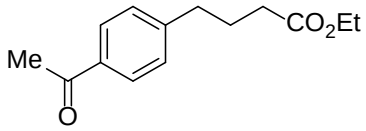
Ethyl 4-(4-((4-ethoxy-4-oxobutyl)amino)phenyl)butanoate: yellow oil (0.099g, 31%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 6.98 (d, J = 8.3 Hz, 2H), 6.54 (d, J = 8.4 Hz, 2H), 4.16 – 4.09 (m, 4H), 3.63 (br, 1H), 3.15 (t, J = 6.9 Hz, 2H), 2.53 (t, J = 7.5 Hz, 2H), 2.41 (t, J = 7.2 Hz, 2H), 2.29 (t, J = 7.5 Hz, 2H), 1.97 – 1.85 (m, 4H), 1.25 (td, J = 7.1, 2.2 Hz, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.8, 173.6, 146.5, 130.3, 129.4, 112.9, 60.6, 60.3, 43.6, 34.3, 33.8, 32.0, 27.0, 24.8, 14.4, 14.3. HRMS (EI): *m/z* calcd for $C_{18}H_{27}NO_4$: 321.1940; found: 321.1938.



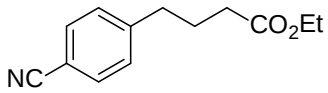
Ethyl 4-(2-acetylphenyl)butanoate (**3ja**)[7]: pale yellow oil (0.110g, 47%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.52 (d, *J* = 7.5 Hz, 1H), 7.25 (t, *J* = 7.4 Hz, 1H), 7.13 (t, *J* = 7.3 Hz, 2H), 3.97 (q, *J* = 7.1 Hz, 2H), 2.74 (t, *J* = 7.6 Hz, 2H), 2.43 (s, 3H), 2.21 (t, *J* = 7.5 Hz, 2H), 1.80 – 1.73 (m, 2H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 202.0, 173.6, 141.9, 137.8, 131.6, 131.5, 129.5, 126.1, 60.3, 34.1, 33.3, 29.9, 26.9, 14.3.



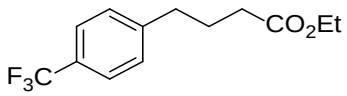
Ethyl 4-(3-acetylphenyl)butanoate (**3ka**): pale yellow oil (0.164g, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.77 (t, *J* = 3.6 Hz, 2H), 7.38 – 7.35 (m, 2H), 4.12 (q, *J* = 7.1 Hz), 2.70 (t, *J* = 7.6 Hz, 2H), 2.59 (s, 3H), 2.31 (t, *J* = 7.4 Hz, 2H), 2.01 – 1.92 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 198.3, 173.3, 142.0, 137.3, 133.3, 128.7, 128.2, 126.2, 60.3, 35.0, 33.5, 26.7, 26.4, 14.3. HRMS (EI): *m/z* calcd for $C_{14}H_{18}O_3$: 234.1256; found: 234.1258.



Ethyl 4-(4-acetylphenyl)butanoate (**3la**)[2]: pale yellow oil (0.138g, 59%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.89 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 2.59 (s, 3H), 2.33 (t, *J* = 7.4 Hz, 2H), 2.04 – 1.92 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$) δ (ppm): 197.8, 173.2, 147.3, 135.2, 128.7, 128.6, 60.3, 35.1, 33.5, 26.5, 26.1, 14.2.

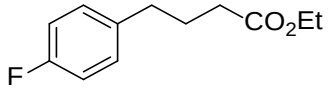


Ethyl 4-(4-cyanophenyl)butanoate (**3ma**)[2]: colorless oil (0.109g, 50%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.58 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 2.33 (t, *J* = 7.3 Hz, 2H), 2.03 – 1.89 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 172.9, 147.1, 132.1, 129.2, 118.9, 109.8, 60.3, 35.1, 33.3, 25.9, 14.2.

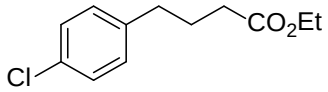


Ethyl 4-(4-(trifluoromethyl)phenyl)butanoate (**3na**)[2]: yellow oil (0.201g, 77%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.53 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.71 (t, *J* =

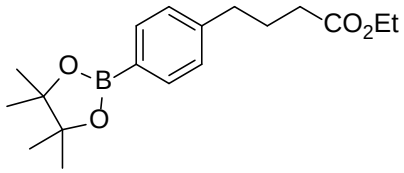
7.7 Hz, 2H), 2.32 (t, *J* = 7.4 Hz, 2H), 2.00 – 1.93 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.3, 145.7, 128.9, 128.4 (q, *J* = 32.1 Hz), 125.4 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 271.8 Hz), 60.5, 35.0, 33.6, 26.3, 14.3.



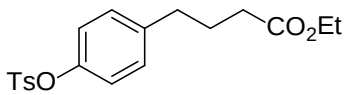
Ethyl 4-(4-fluorophenyl)butanoate (**3oa**)[2]: pale yellow oil (0.162g, 77%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.12 (dd, *J* = 8.3, 5.6 Hz, 2H), 6.96 (t, *J* = 8.7 Hz, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 2.62 (t, *J* = 7.6 Hz, 2H), 2.30 (t, *J* = 7.4 Hz, 2H), 2.00 – 1.87 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.5, 161.4 (d, *J* = 242.0 Hz), 137.1 (d, *J* = 3.3 Hz), 129.9 (d, *J* = 7.9 Hz), 115.2 (d, *J* = 21.2 Hz), 60.4, 34.4, 33.6, 26.7, 14.3.



Ethyl 4-(4-chlorophenyl)butanoate (**3pa**)[5]: pale yellow oil (0.177g, 78%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.24 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 2.30 (t, *J* = 7.4 Hz, 2H), 1.97 – 1.87 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.3, 139.9, 131.7, 129.9, 128.5, 60.4, 34.5, 33.5, 26.5, 14.3.

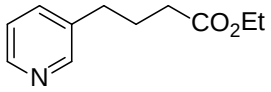


Ethyl 4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)butanoate (**3qa**)[2]: pale yellow oil (0.248g, 78%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.74 (d, *J* = 7.8 Hz, 2H), 7.19 (d, *J* = 7.8 Hz, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 2.66 (t, *J* = 7.6 Hz, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 1.99 – 1.91 (m, 2H), 1.34 (s, 12H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.5, 144.9, 135.0, 128.0, 83.7, 60.3, 35.4, 33.7, 26.5, 24.9, 14.3.

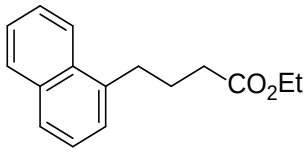


Ethyl 4-(4-(tosyloxy)phenyl)butanoate (**3ra**)[2]: colorless oil (0.257g, 71%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.69 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 2.60 (t, *J* = 7.7 Hz, 2H), 2.44 (s, 3H), 2.28 (t, *J* = 7.4 Hz, 2H), 1.93 – 1.86

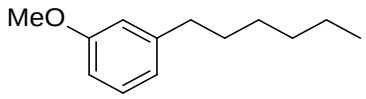
(m, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.4, 147.9, 145.4, 140.9, 132.5, 129.8, 129.6, 128.6, 122.3, 60.4, 34.5, 33.6, 26.4, 21.8, 14.3.



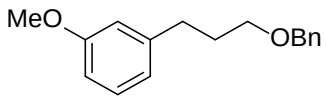
Ethyl 4-(pyridin-3-yl)butanoate (**3sa**)[8]: colorless oil (0.114g, 59%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 8.42 (d, *J* = 2.7 Hz, 2H), 7.48 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.19 (dd, *J* = 7.6, 4.9 Hz, 1H), 4.10 (q, *J* = 7.1 Hz, 2H), 2.63 (t, *J* = 7.7 Hz, 2H), 2.30 (t, *J* = 7.4 Hz, 2H), 1.97 – 1.89 (m, 2H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$) δ (ppm): 173.2, 150.0, 147.6, 136.7, 135.9, 123.4, 60.4, 33.5, 32.2, 26.2, 14.3.



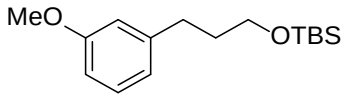
Ethyl 4-(naphthalen-1-yl)butanoate (**3ta**)[2]: yellow oil (0.201g, 83%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 8.10 (d, *J* = 8.3 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.74 (d, *J* = 8.1 Hz), 7.56 – 7.48 (m, 2H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 6.9 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.14 (t, *J* = 7.6 Hz, 2H), 2.43 (t, *J* = 7.3 Hz, 2H), 2.16 – 2.08 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 173.5, 137.6, 134.0, 131.9, 128.8, 126.9, 126.2, 125.9, 125.5, 123.8, 60.4, 34.0, 32.4, 25.9, 14.3.



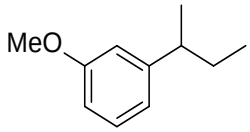
1-Hexyl-3-methoxybenzene (**3ab**)[9]: yellow oil (0.156g, 81%).^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.21 (t, *J* = 7.7 Hz, 1H), 6.77 (dd, *J* = 20.7, 7.6 Hz, 3H), 3.81 (s, 3H), 2.60 (t, *J* = 7.6 Hz, 2H), 1.66–1.59 (m, 2H), 1.39 – 1.27 (m, 6H), 0.90 (t, *J* = 6.5 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 159.7, 144.8, 129.3, 121.0, 114.3, 110.9, 55.2, 36.2, 31.9, 31.5, 29.2, 22.8, 14.2.



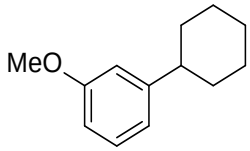
1-(3-(Benzyloxy)propyl)-3-methoxybenzene (**3ac**): yellow oil (0.177g, 69%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.39 – 7.29 (m, 5H), 7.22 (t, *J* = 7.6 Hz, 1H), 6.79 (dd, *J* = 17.9, 7.9 Hz, 3H), 4.54 (s, 2H), 3.81 (s, 3H), 3.53 (t, *J* = 6.3 Hz, 2H), 2.73 (t, *J* = 7.6 Hz, 2H), 2.01 – 1.94 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 159.7, 143.7, 138.7, 129.3, 128.5, 127.7, 127.6, 121.0, 114.3, 111.2, 73.0, 69.6, 55.2, 32.6, 31.4. HRMS (EI): *m/z* calcd for $C_{17}H_{20}O_2$: 256.1463; found: 256.1460.



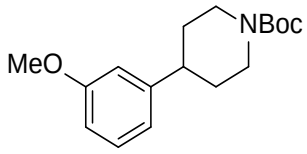
tert-Butyl(3-(3-methoxyphenyl)propoxy)dimethylsilane (**3ad**)[13]: yellow oil (0.196g, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.20 (t, *J* = 7.7 Hz, 1H), 6.81 – 6.73 (m, 3H), 3.81 (s, 3H), 3.65 (t, *J* = 6.3 Hz, 2H), 2.67 (t, *J* = 7.7 Hz, 2H), 1.88 – 1.81 (m, 2H), 0.92 (s, 9H), 0.07 (s, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 159.7, 144.0, 129.3, 121.0, 114.3, 111.1, 62.5, 55.2, 34.5, 32.3, 26.1, 18.5, -5.1.



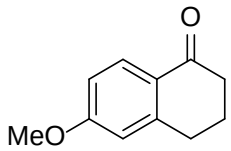
1-(sec-Butyl)-3-methoxybenzene (**3ae**)[10]: yellow oil (0.028g, 17%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.23 (t, *J* = 7.7 Hz, 1H), 6.82 – 6.74 (m, 3H), 3.82 (s, 3H), 2.64 – 2.55 (m, 1H), 1.67 – 1.56 (m, 2H), 1.26 (d, *J* = 6.9 Hz, 3H), 0.86 (t, *J* = 7.4 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 159.7, 149.6, 129.3, 119.7, 113.2, 110.8, 55.2, 41.9, 31.2, 21.9, 12.4.



1-Cyclohexyl-3-methoxybenzene (**3af**)[10]: yellow oil (0.044g, 23%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.23 (t, *J* = 7.9 Hz, 1H), 6.84 – 6.73 (m, 3H), 3.82 (s, 3H), 2.53 – 2.46 (m, 1H), 1.91 – 1.75 (m, 5H), 1.49 – 1.25 (m, 5H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 159.7, 150.0, 129.3, 119.4, 112.9, 110.9, 55.2, 44.8, 34.5, 27.0, 26.3.



N-tert-Butylcarboxylate 4-(3-methoxyphenyl) piperidine (**3ag**)[11]: yellow oil (0.073g, 25%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.25 – 7.20 (m, 1H), 6.81 – 6.75 (m, 3H), 4.23 (br, 2H), 3.80 (s, 3H), 2.79 (t, *J* = 12.2 Hz, 2H), 2.62 (tt, *J* = 12.1, 3.5 Hz, 1H), 1.82 (d, *J* = 13.1 Hz, 2H), 1.61 (qd, *J* = 12.7, 4.3 Hz, 2H), 1.48 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 159.8, 155.0, 147.6, 129.6, 119.3, 112.9, 111.5, 79.5, 55.2, 44.6 (br), 42.9, 33.2, 28.6.



6-Methoxy-1-tetralone[12] : light yellow solid（1.07 g, 75%）, m. p.: 77-79 °C. ^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm): 7.99 (d, *J* = 8.7 Hz, 1H), 6.80 (dd, *J* = 8.7, 2.1 Hz, 1H), 6.68 (s, 1H), 3.83 (s, 3H), 2.91 (t, *J* = 6.1 Hz, 2H), 2.59 (t, *J* = 6.1 Hz, 2H), 2.17 – 2.02 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm): 197.3, 163.6, 147.0, 129.7, 126.4, 113.1, 112.7, 55.5, 39.0 30.2, 23.5.

4. References

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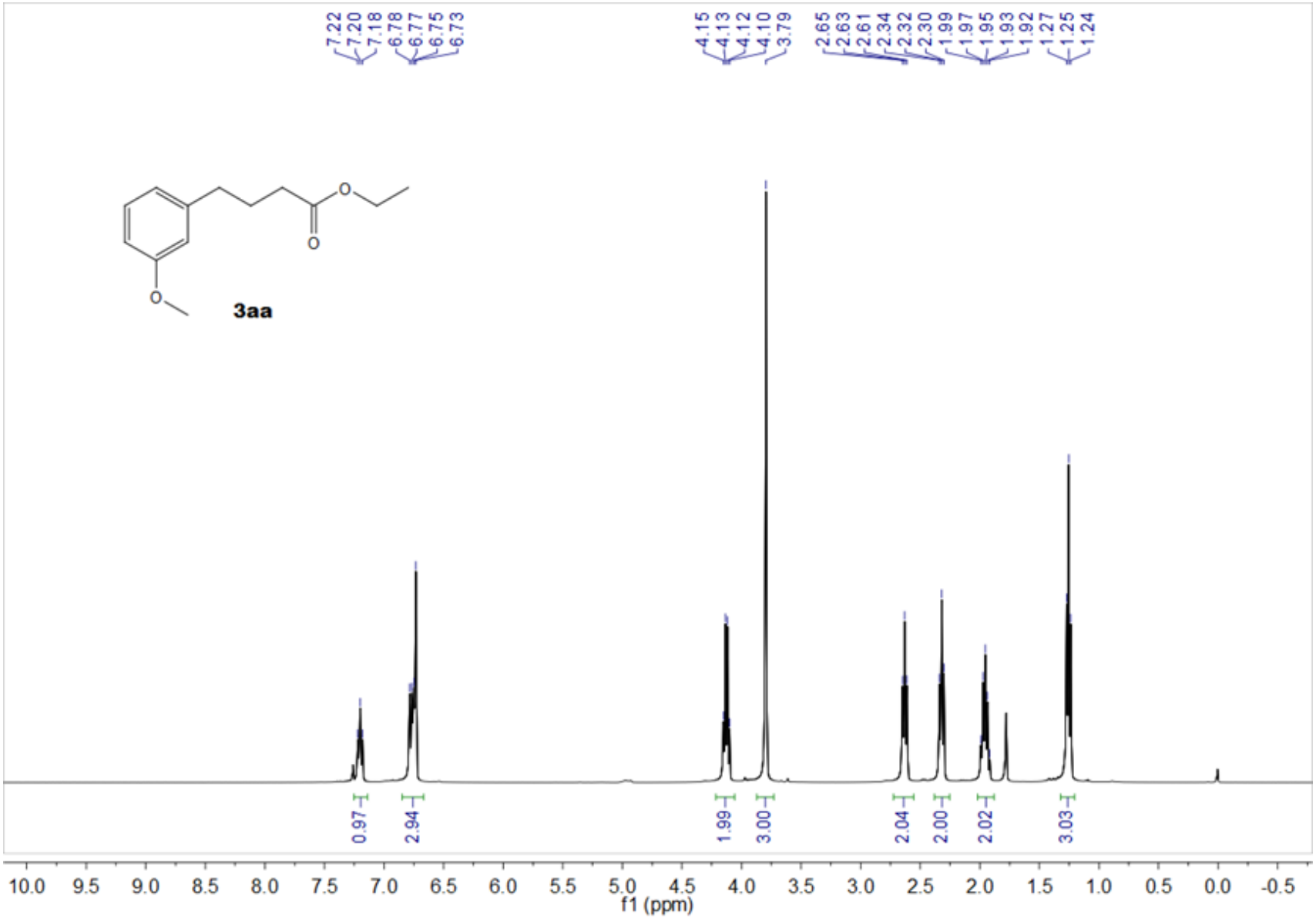
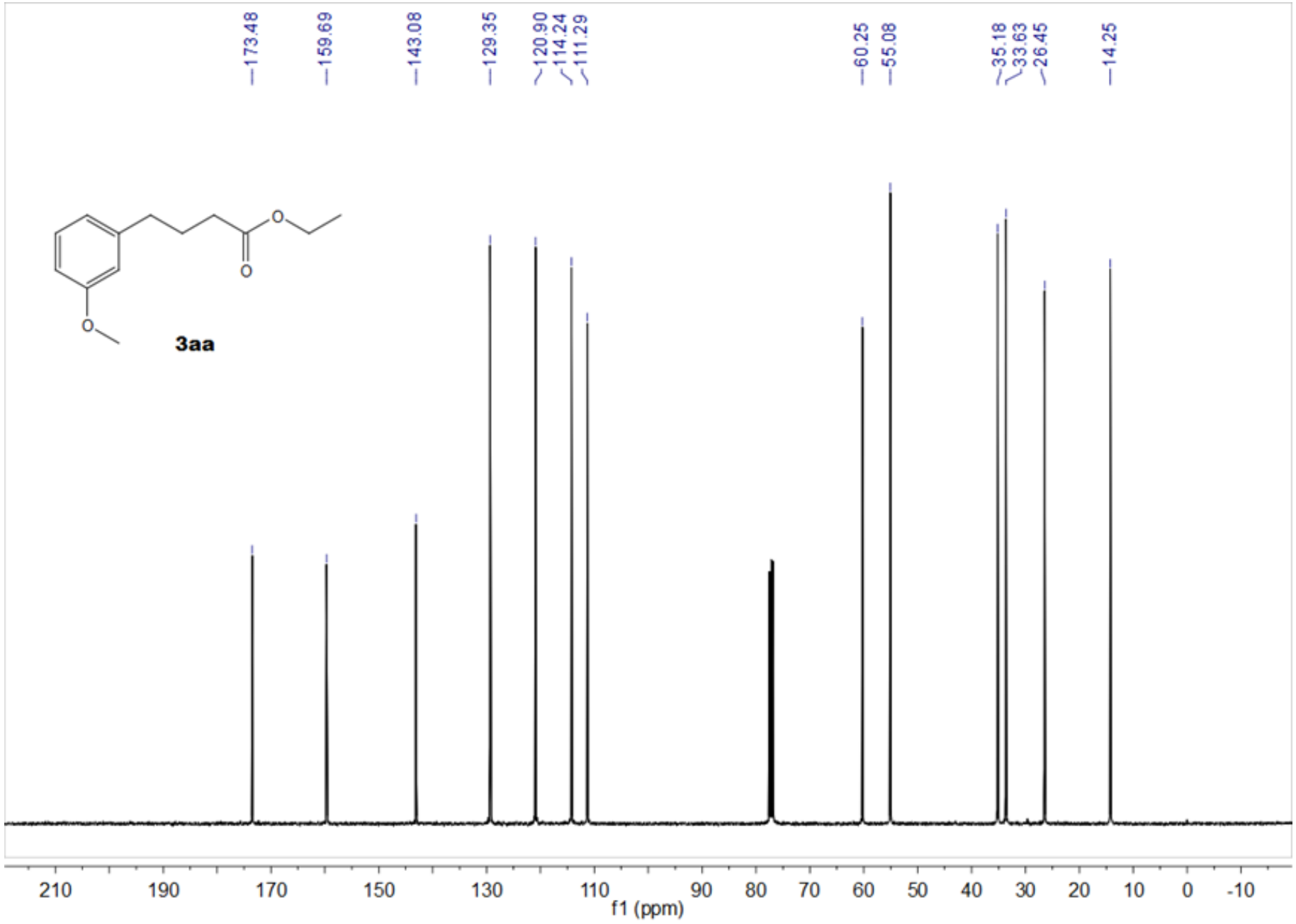
5. NMR spectra and HRMS

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O
O
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110
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80
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60
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40
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20
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0
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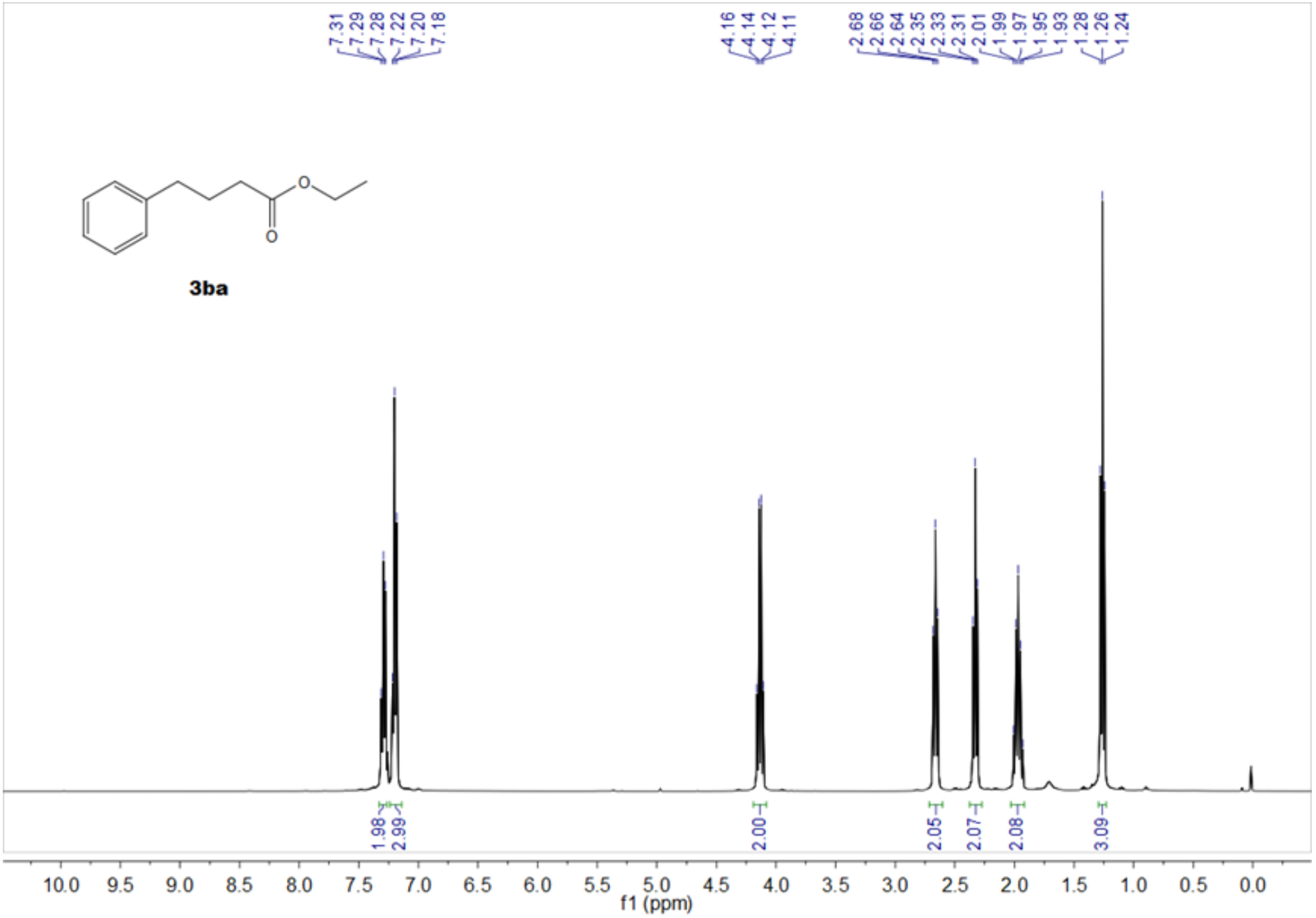
f1 (ppm)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2.35
2.33
2.31
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1.97
1.95
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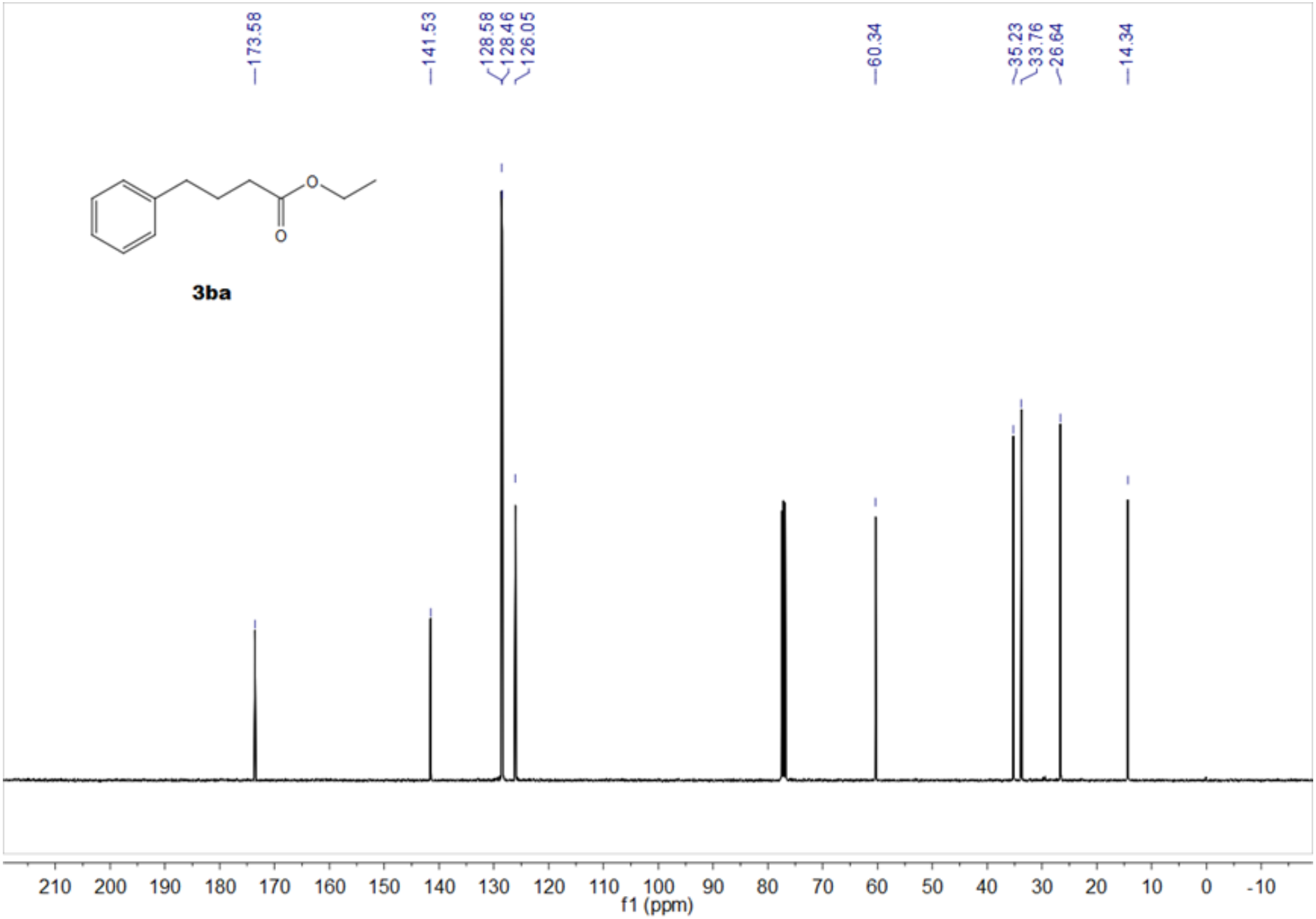
1.28
1.26
1.24
O
O
3ba
1.98
2.99
2.00
2.05
2.07
2.08
3.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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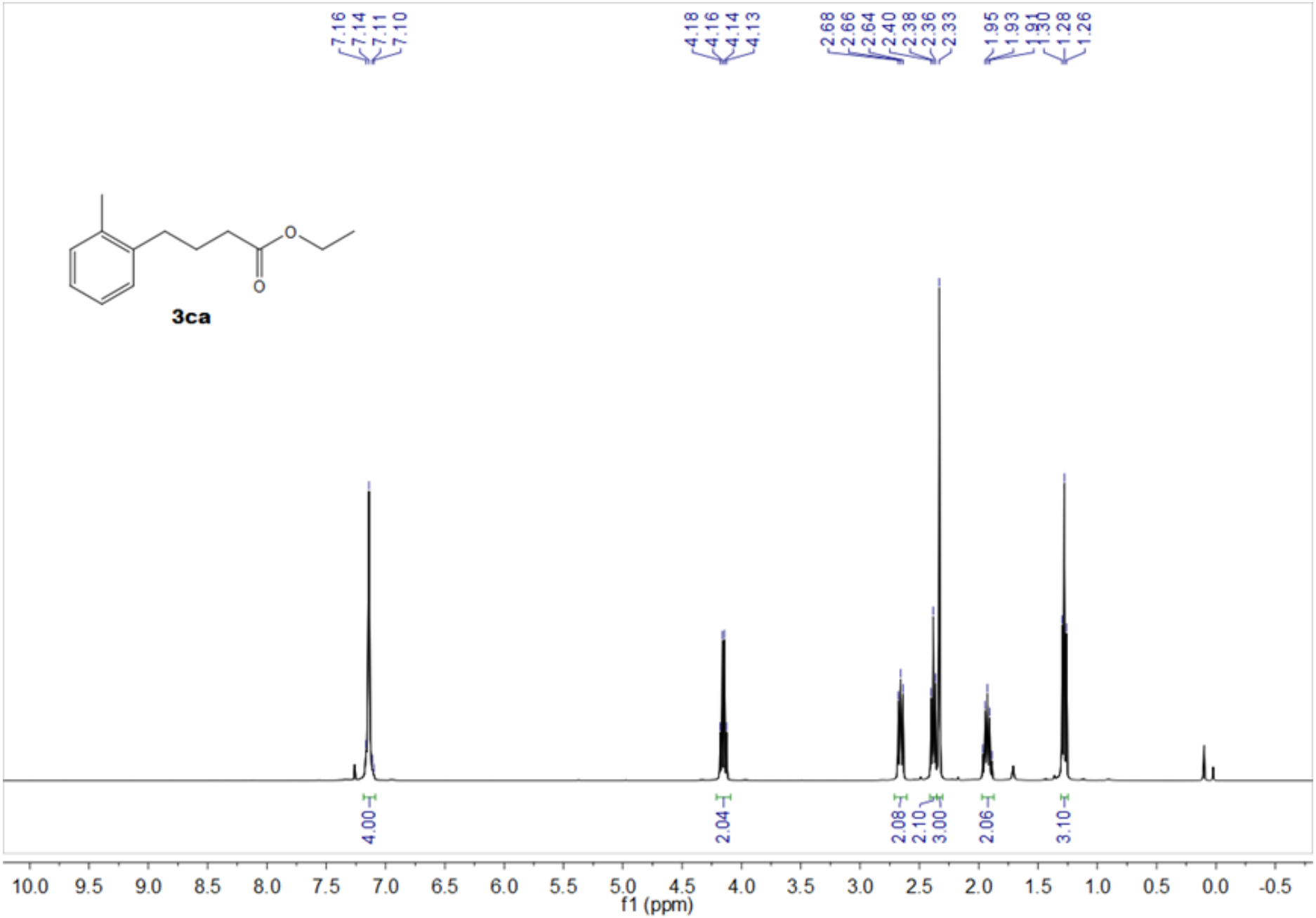
f1 (ppm)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1.91
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O
O
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2.08
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3.00
2.06
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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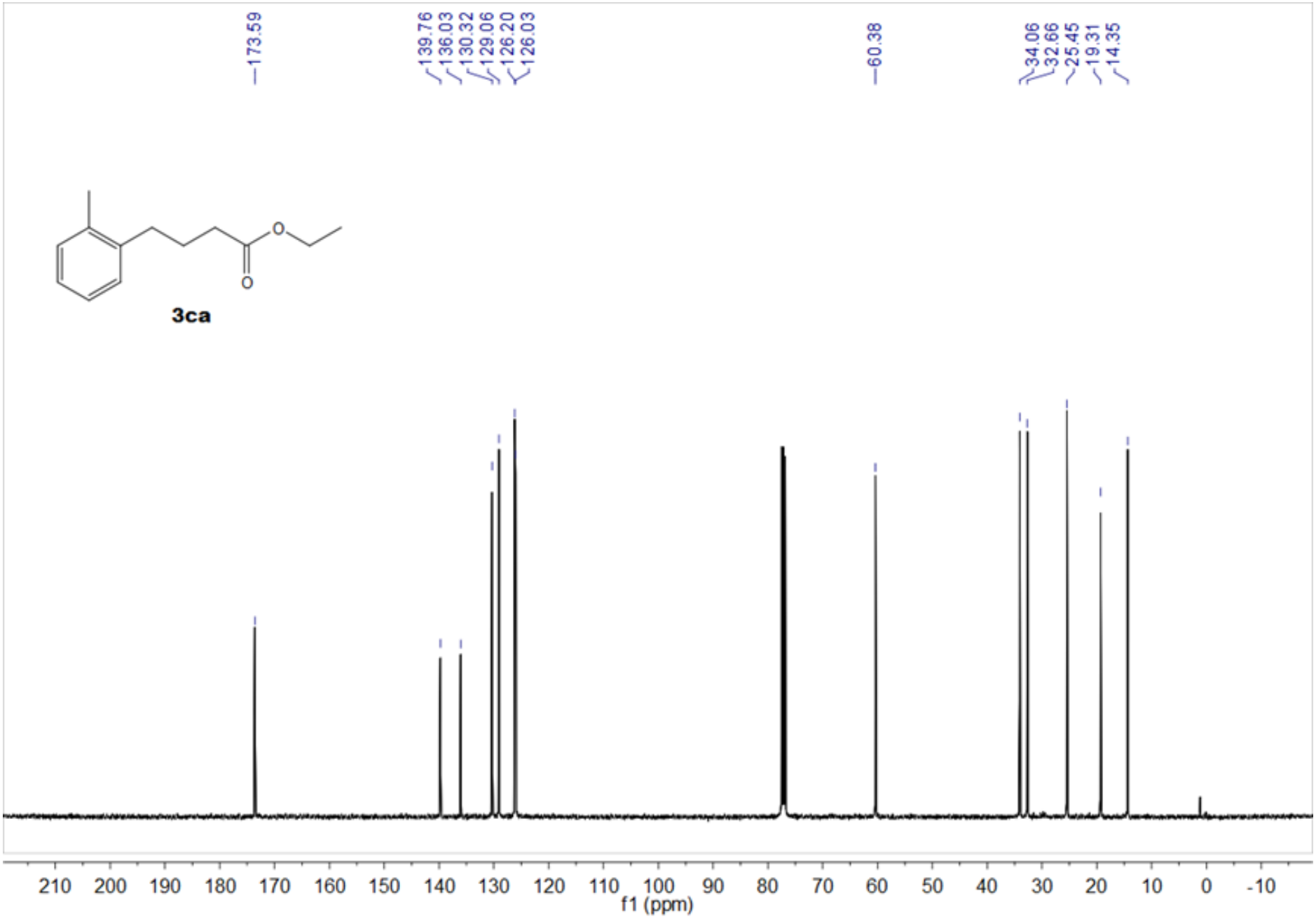
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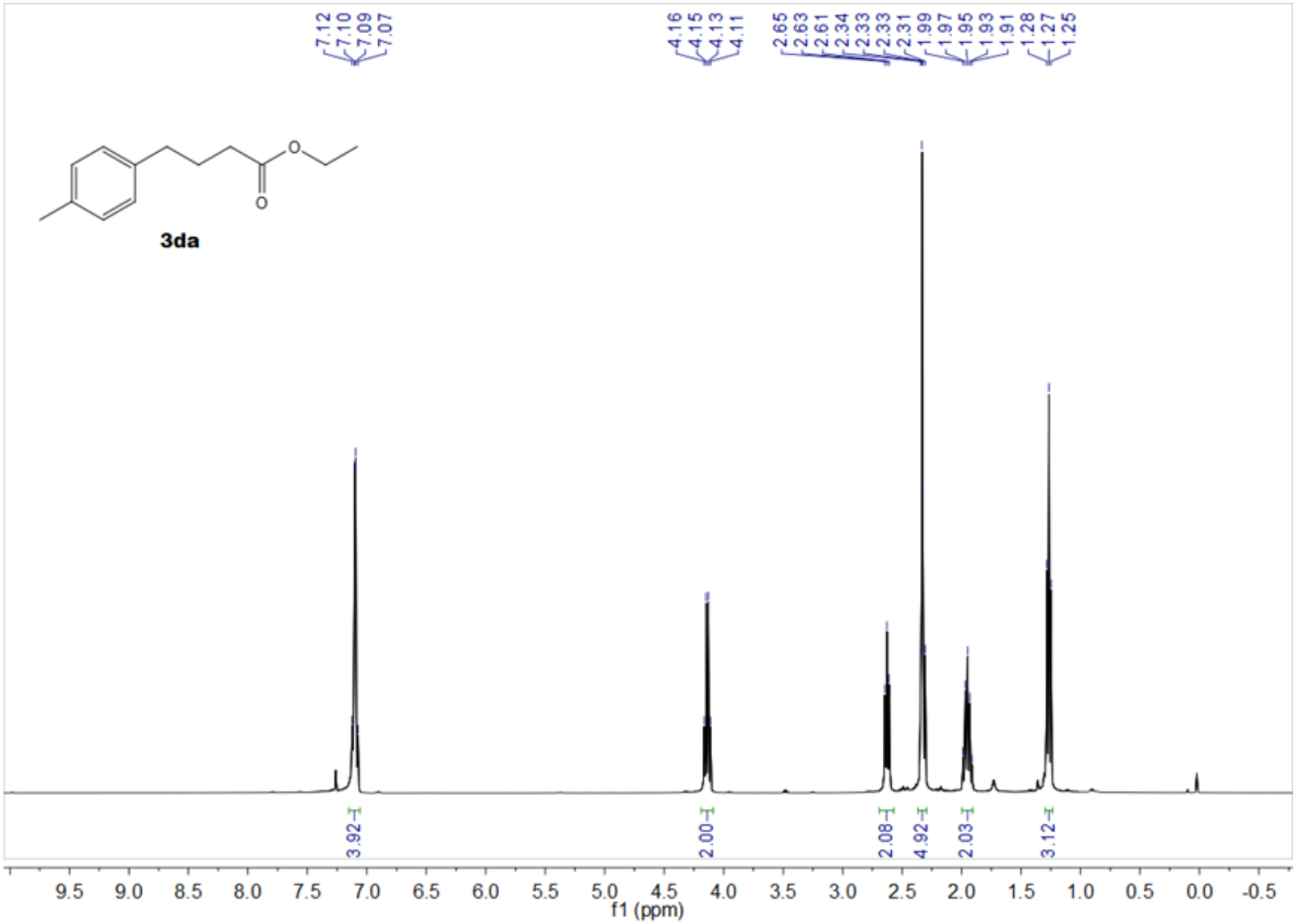
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4.92
2.03
3.12
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9.0
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7.0
6.5
6.0
5.5
5.0
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4.0
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3.0
2.5
2.0
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1.0
0.5
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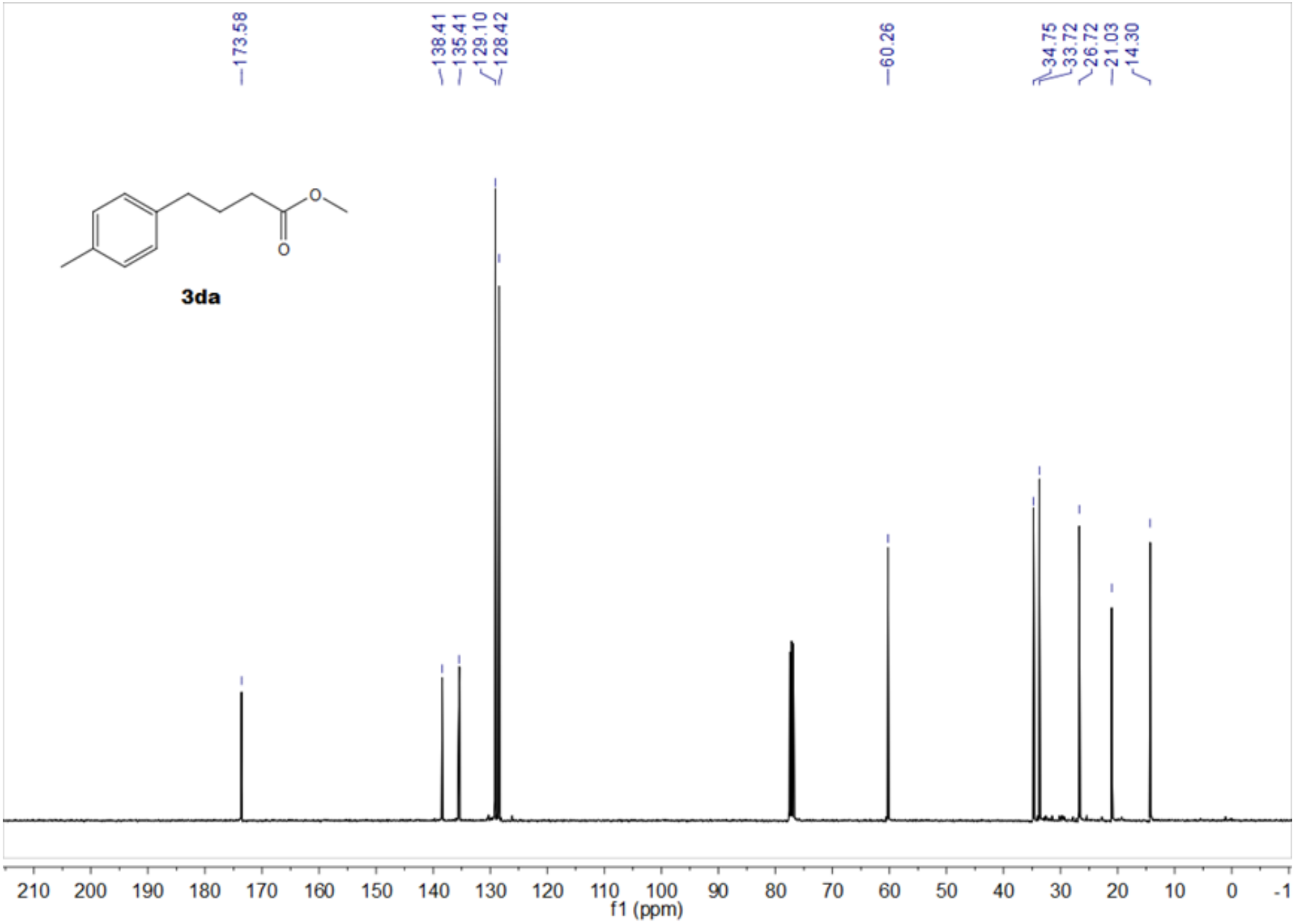
f1 (ppm)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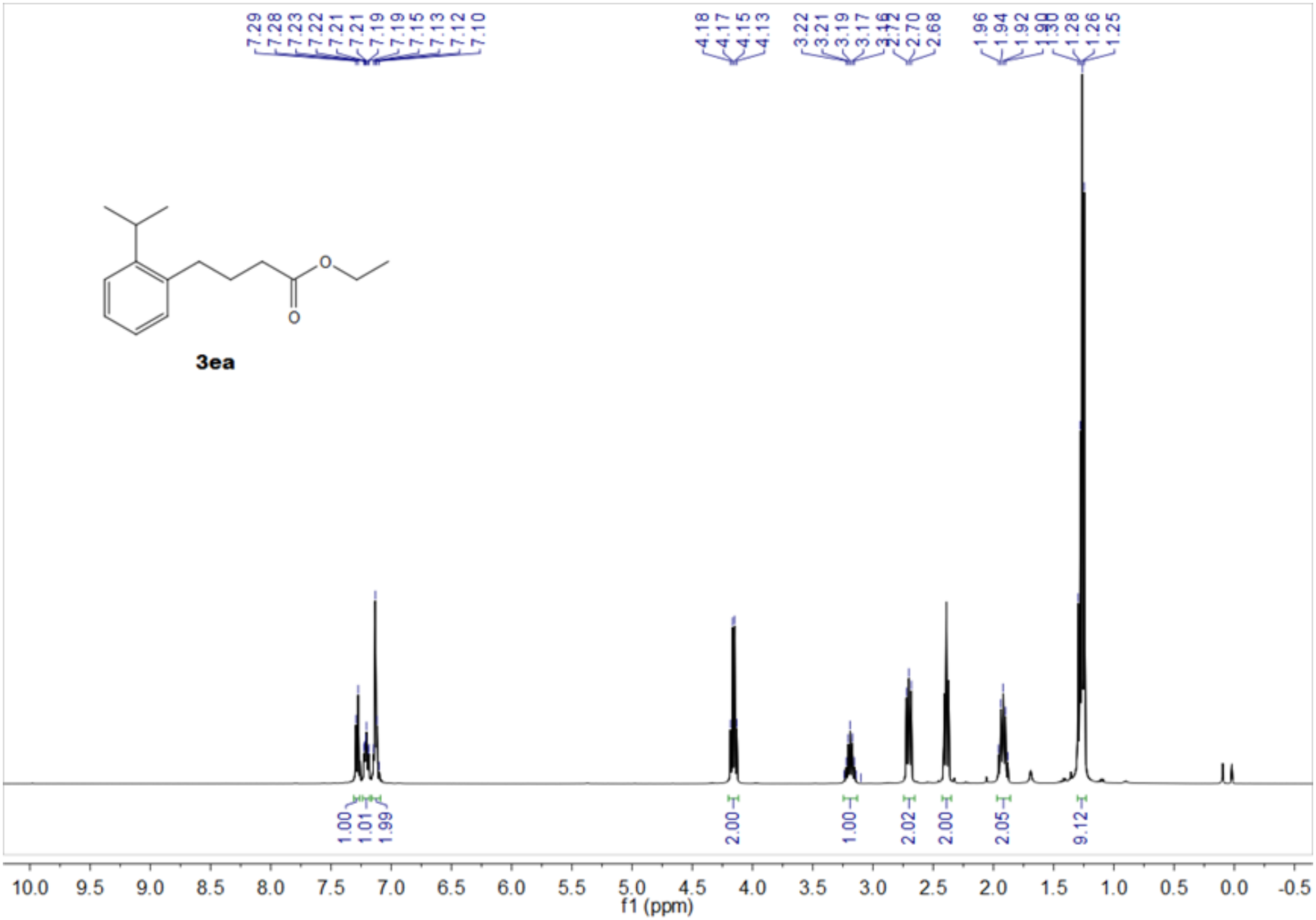
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4.15
4.13
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3.21
3.19
3.17
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1.94
1.92
1.28
1.26
1.25
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O
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1.01
1.99
2.00
1.00
2.02
2.00
2.05
9.12
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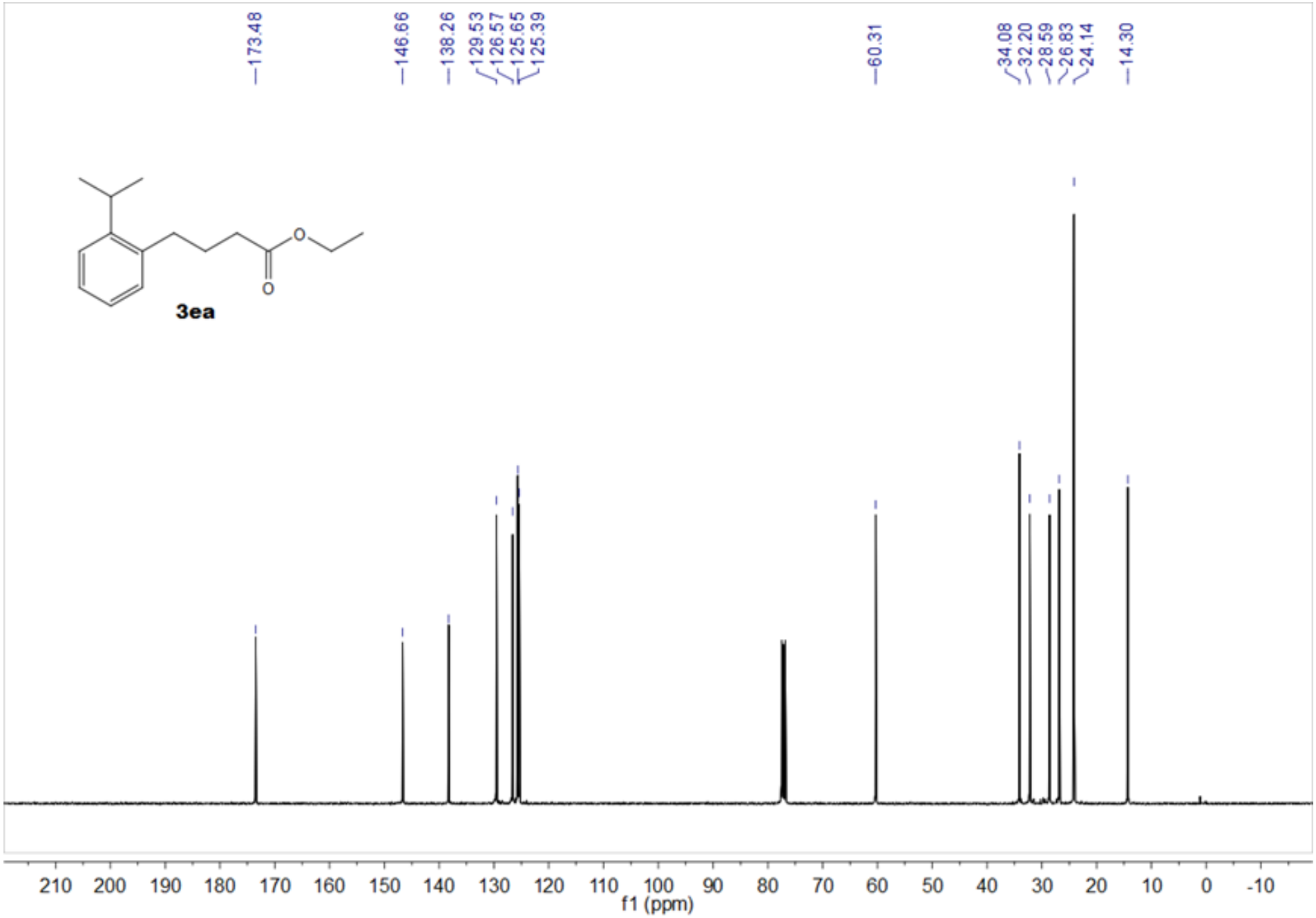
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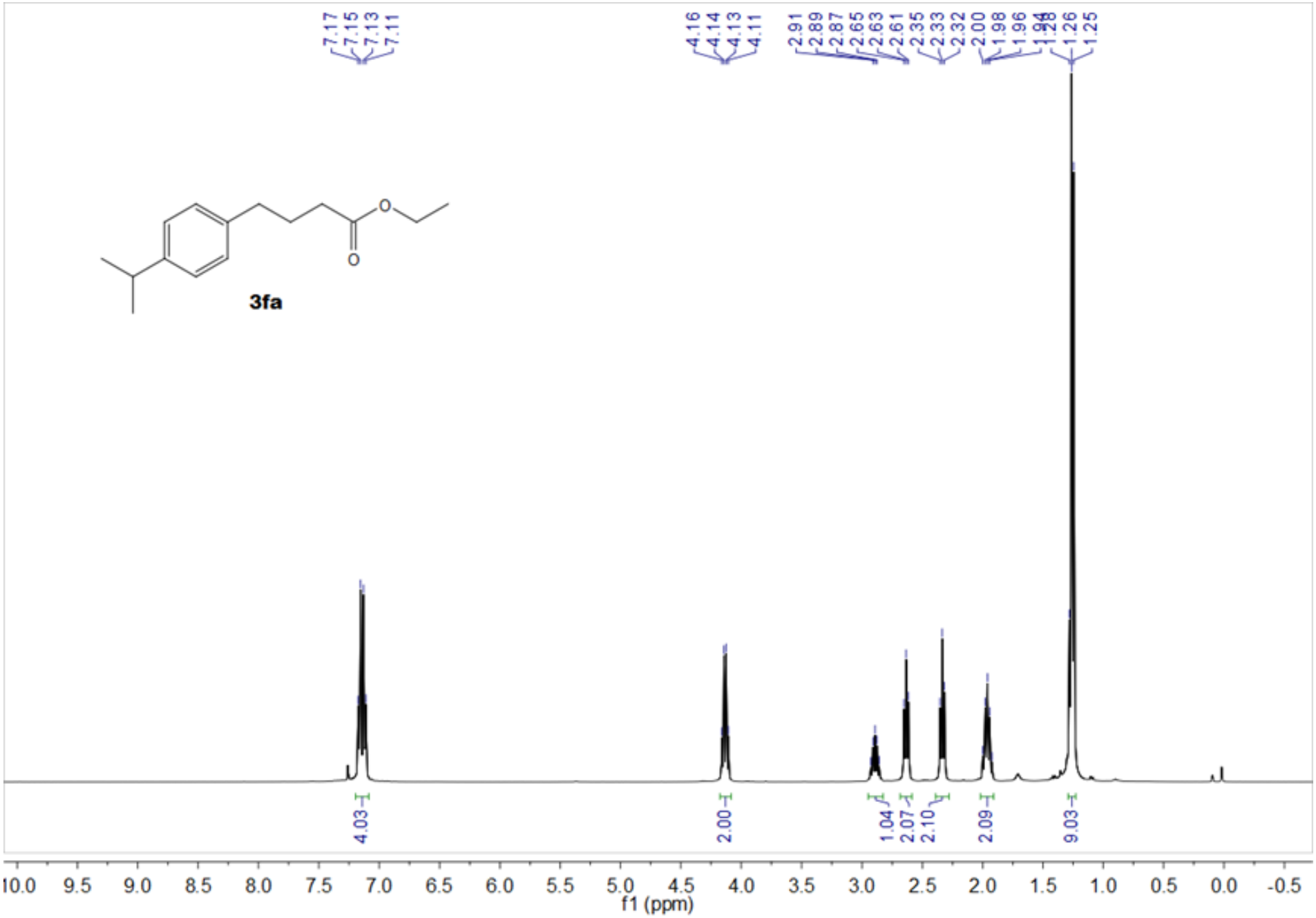
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2.61
2.35
2.33
2.32
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1.96
1.26
1.25
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O
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4.03
2.00
1.04
2.07
2.10
2.09
9.03
10.0 9.5 9.0 8.5 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
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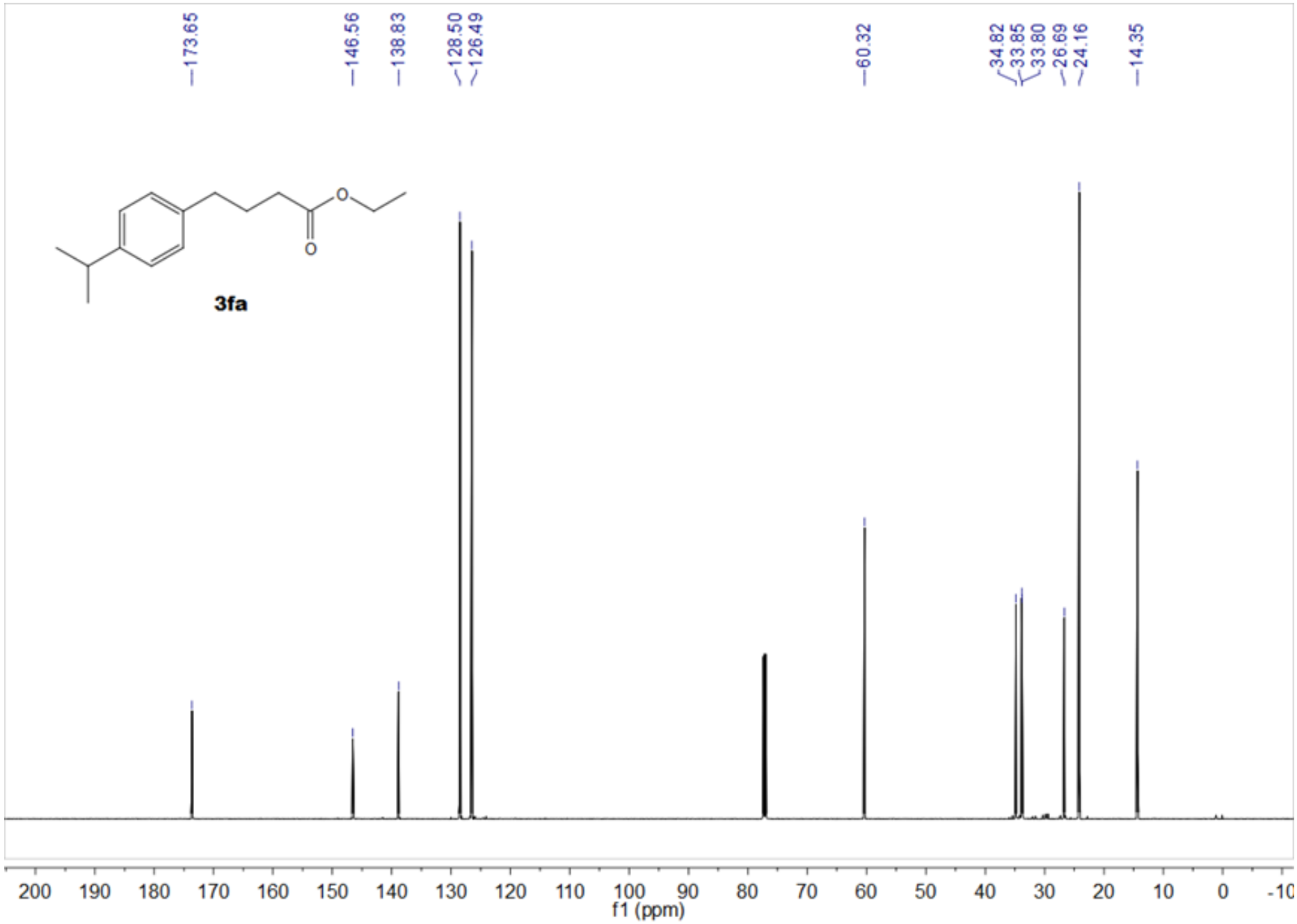
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O
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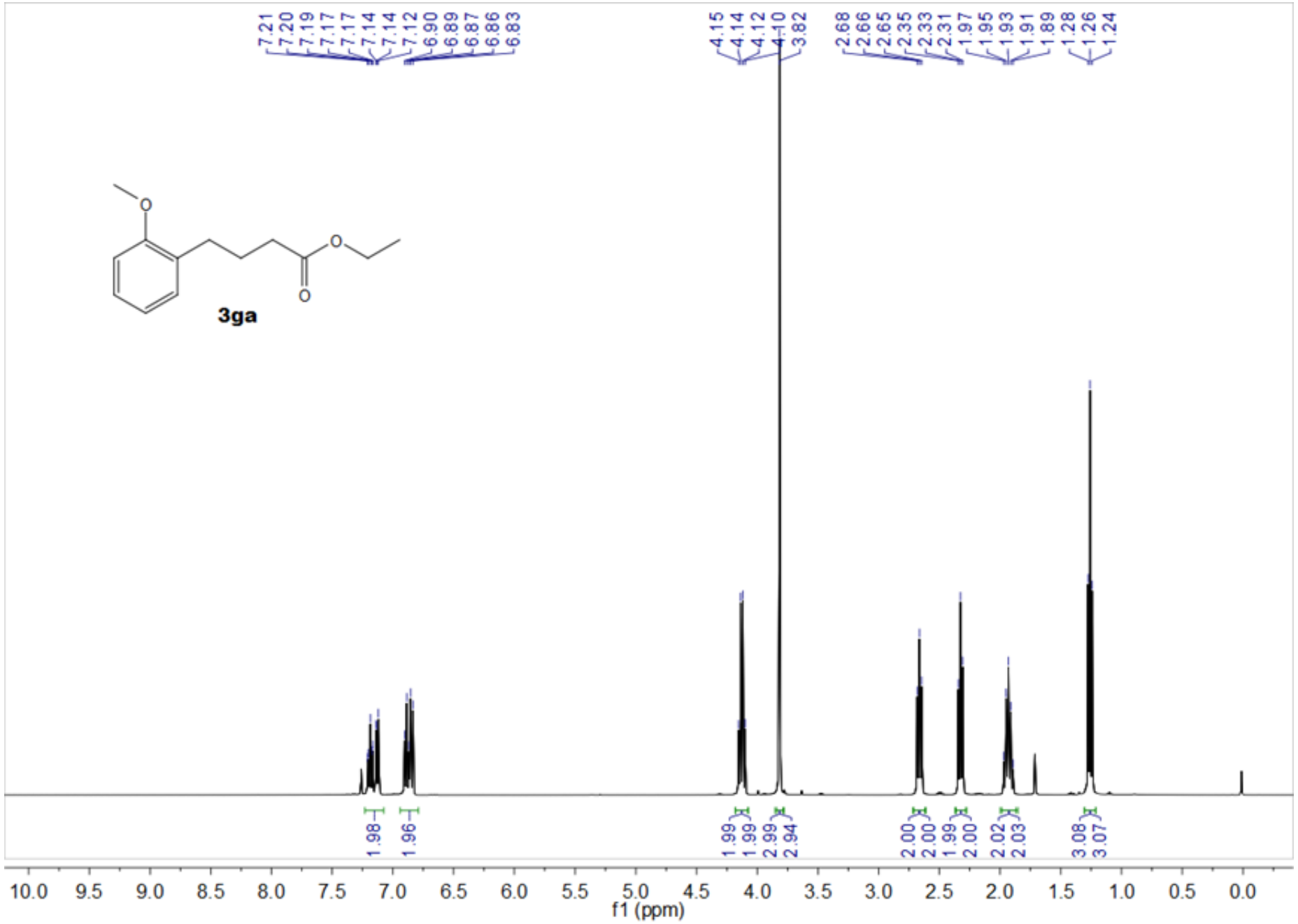
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1.28
1.26
1.24
O
O
O
3ga
1.98
1.96
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2.00
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2.03
3.08
3.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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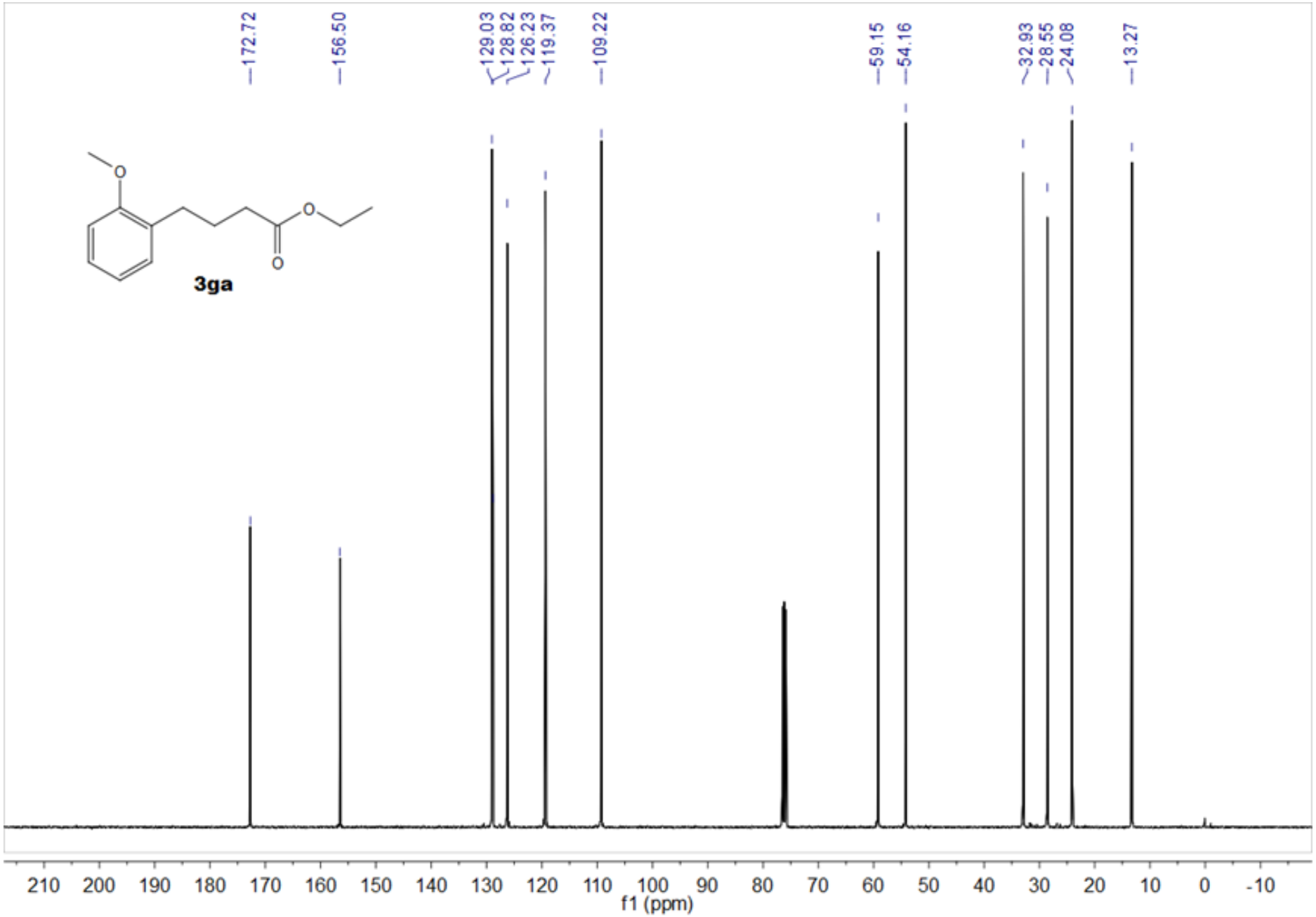
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O
3ga
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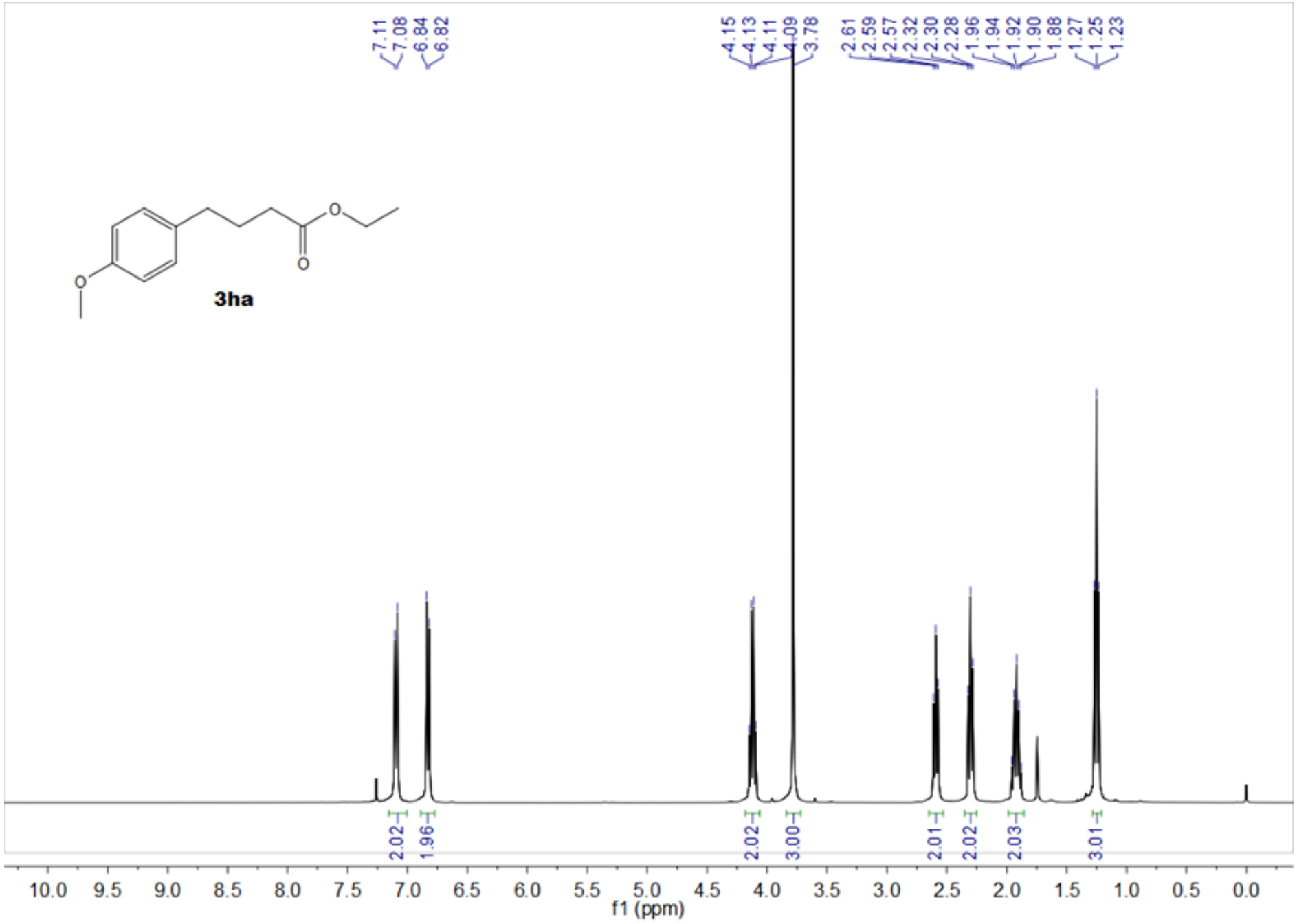
f1 (ppm)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1.94
1.92
1.90
1.88
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1.25
1.23
3ha
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2.02
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6.0
5.5
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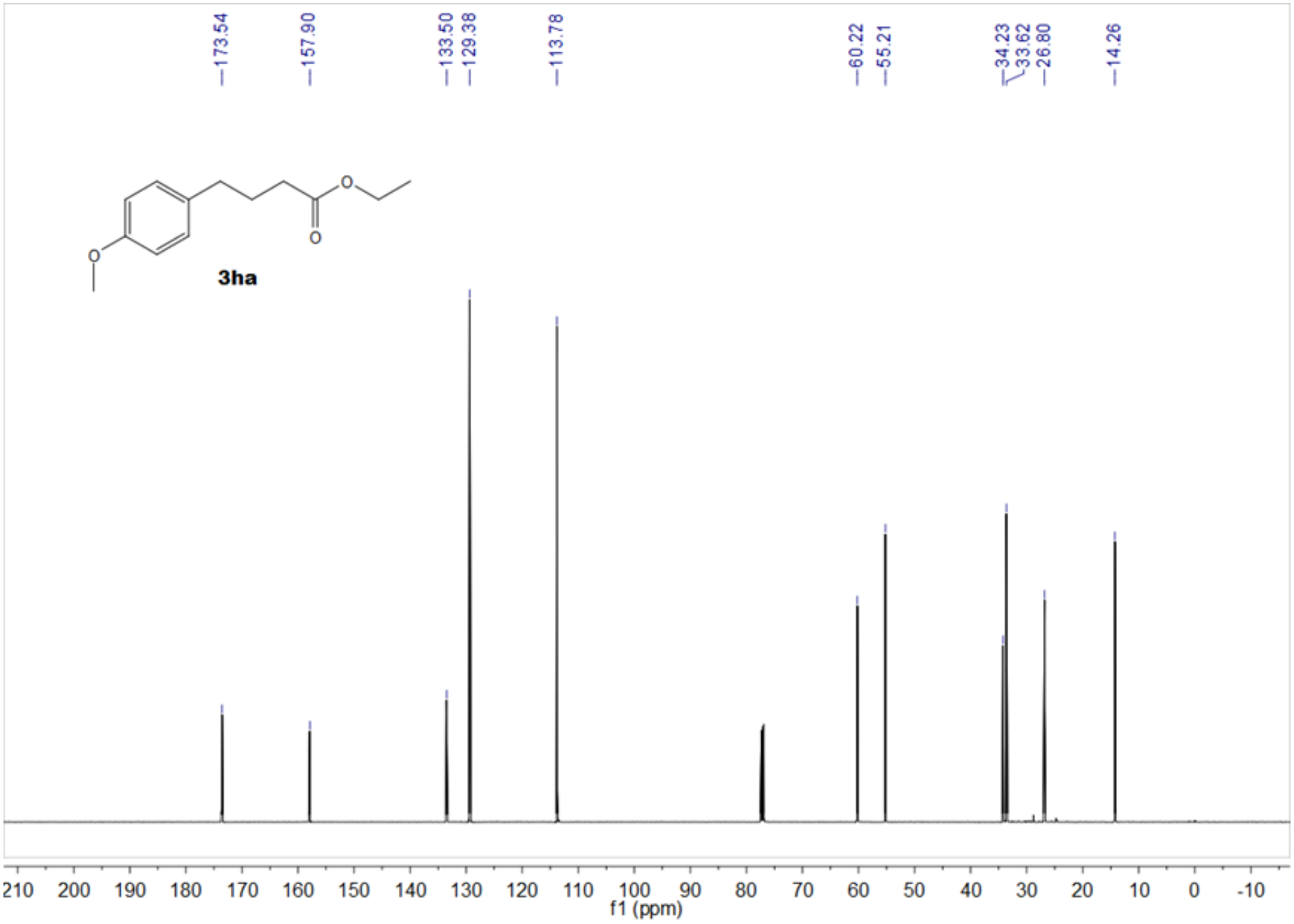
0.0
f1 (ppm)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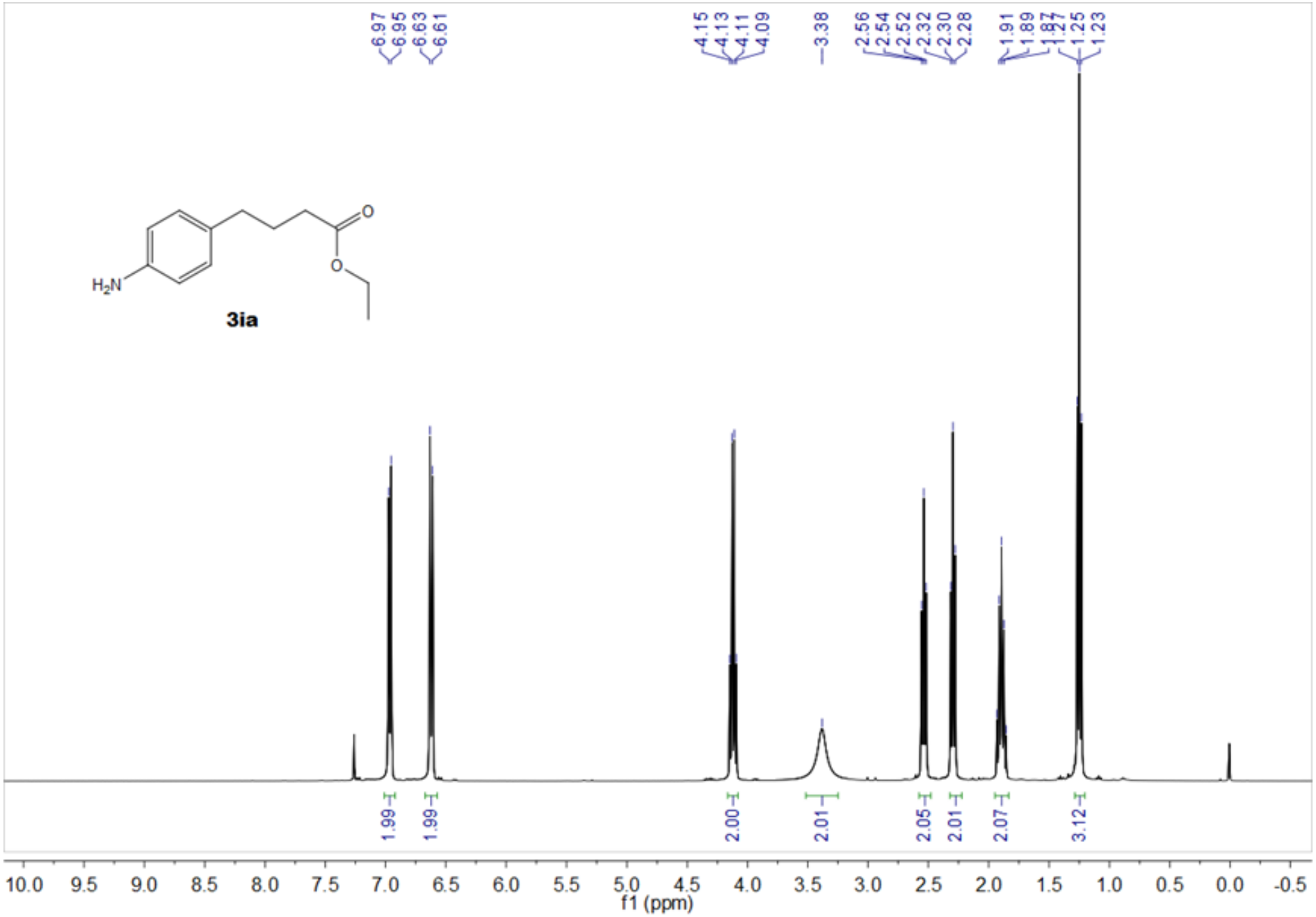
f1 (ppm)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2.30
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1.25
1.23
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O
H2N
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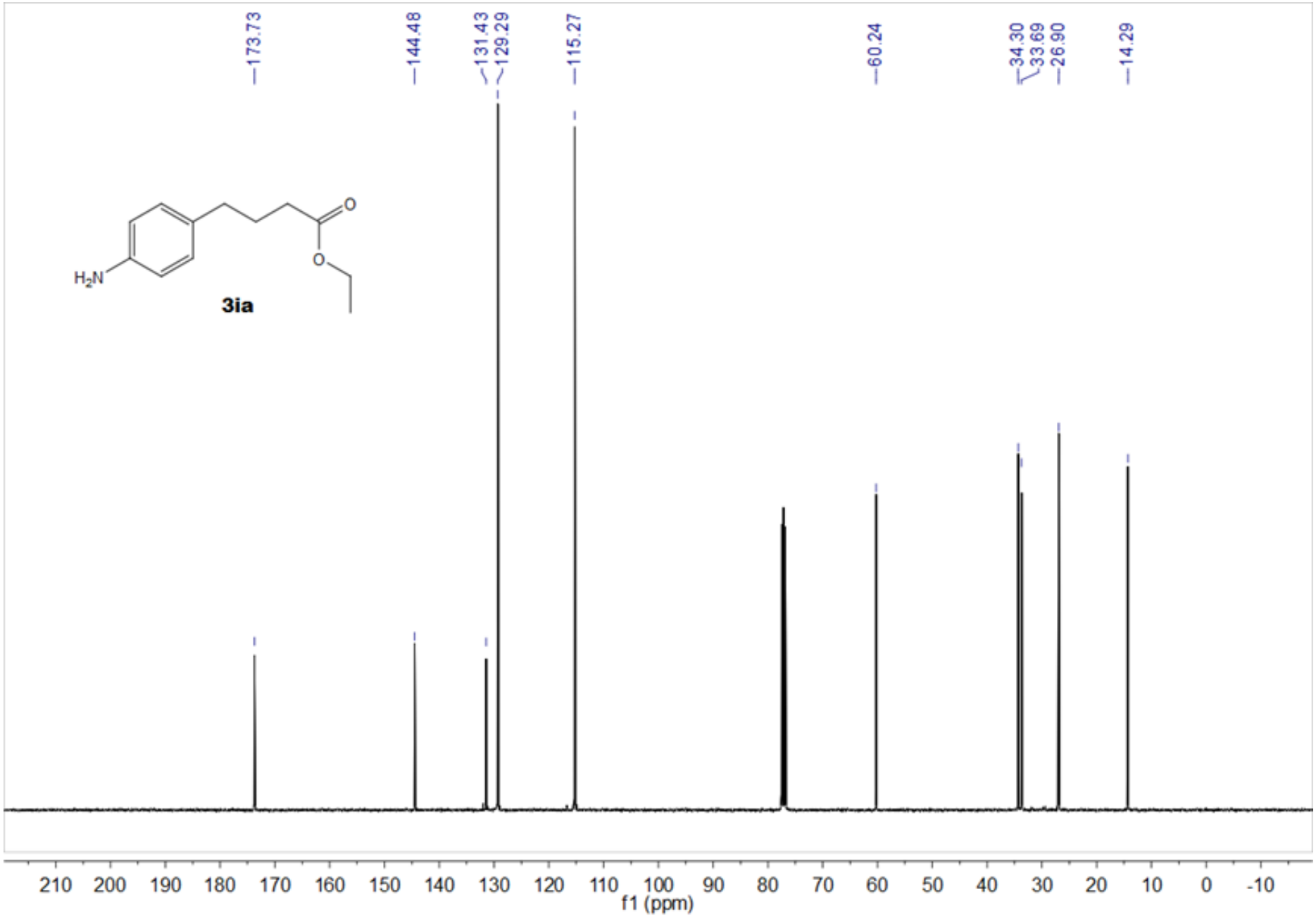
1.99
2.00
2.01
2.05
2.01
2.07
3.12
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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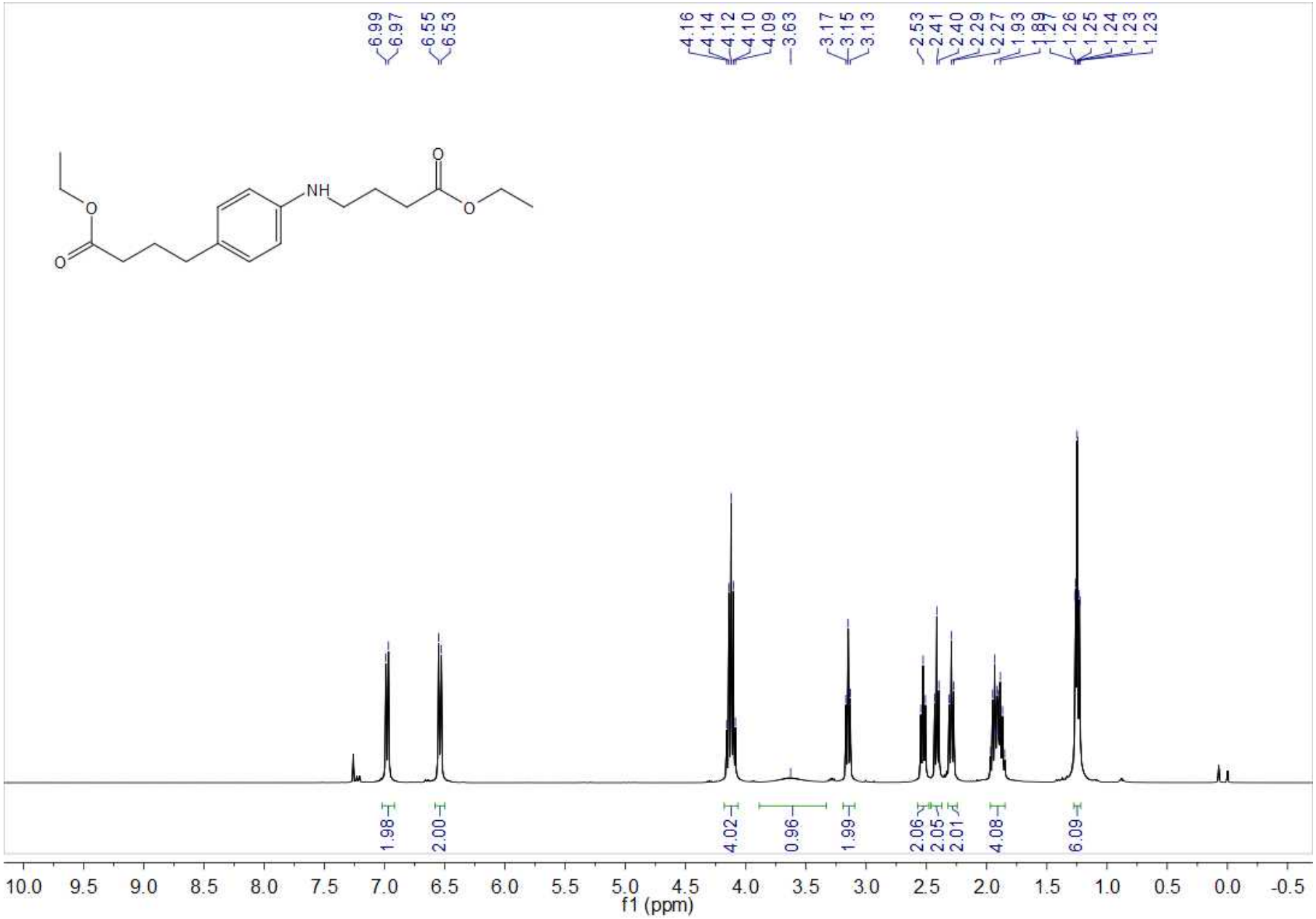
f1 (ppm)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1.27
1.26
1.25
1.24
1.23
1.23
NH
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O
O
O
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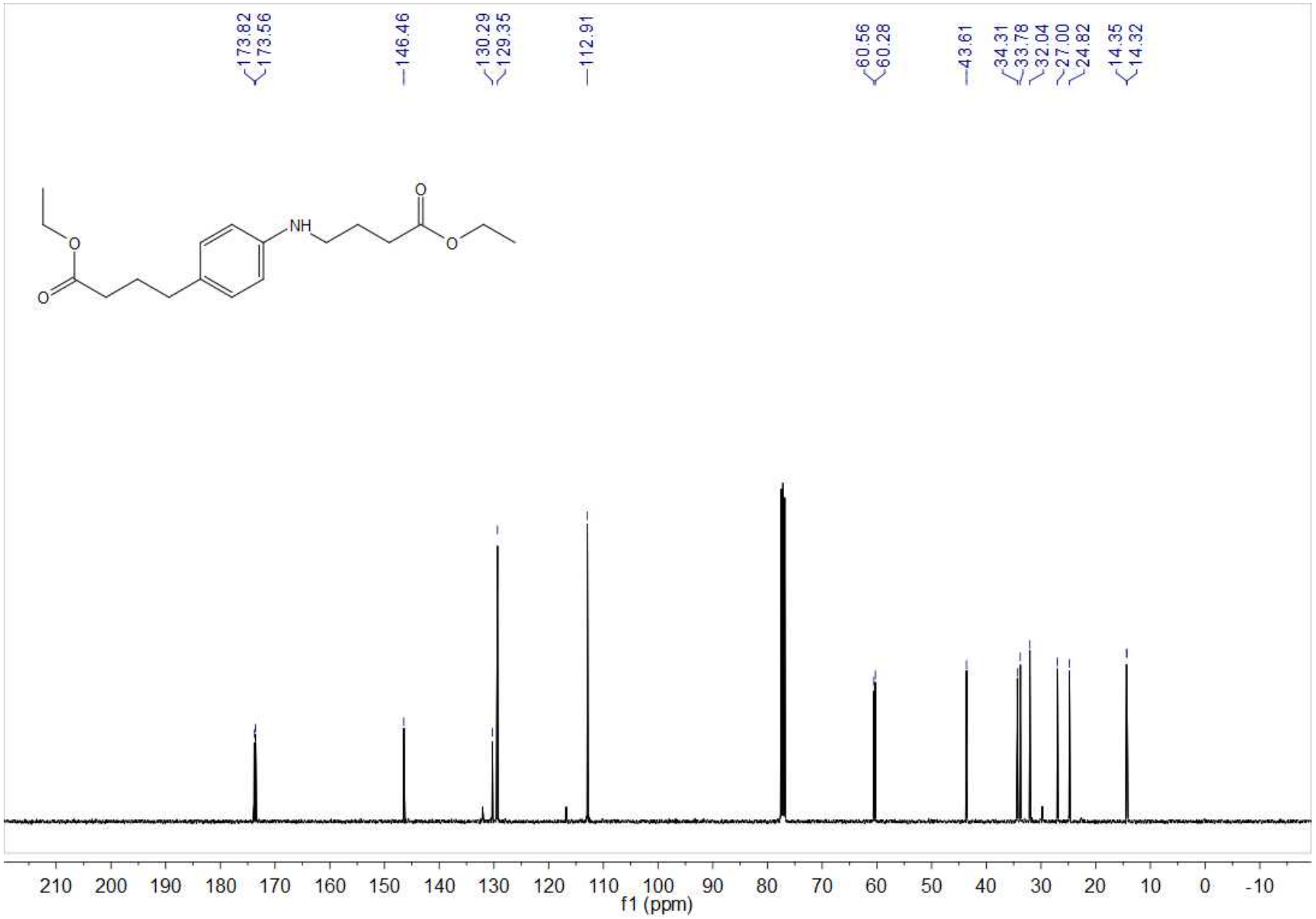
2.00
4.02
0.96
1.99
2.06
2.05
2.01
4.08
6.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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O
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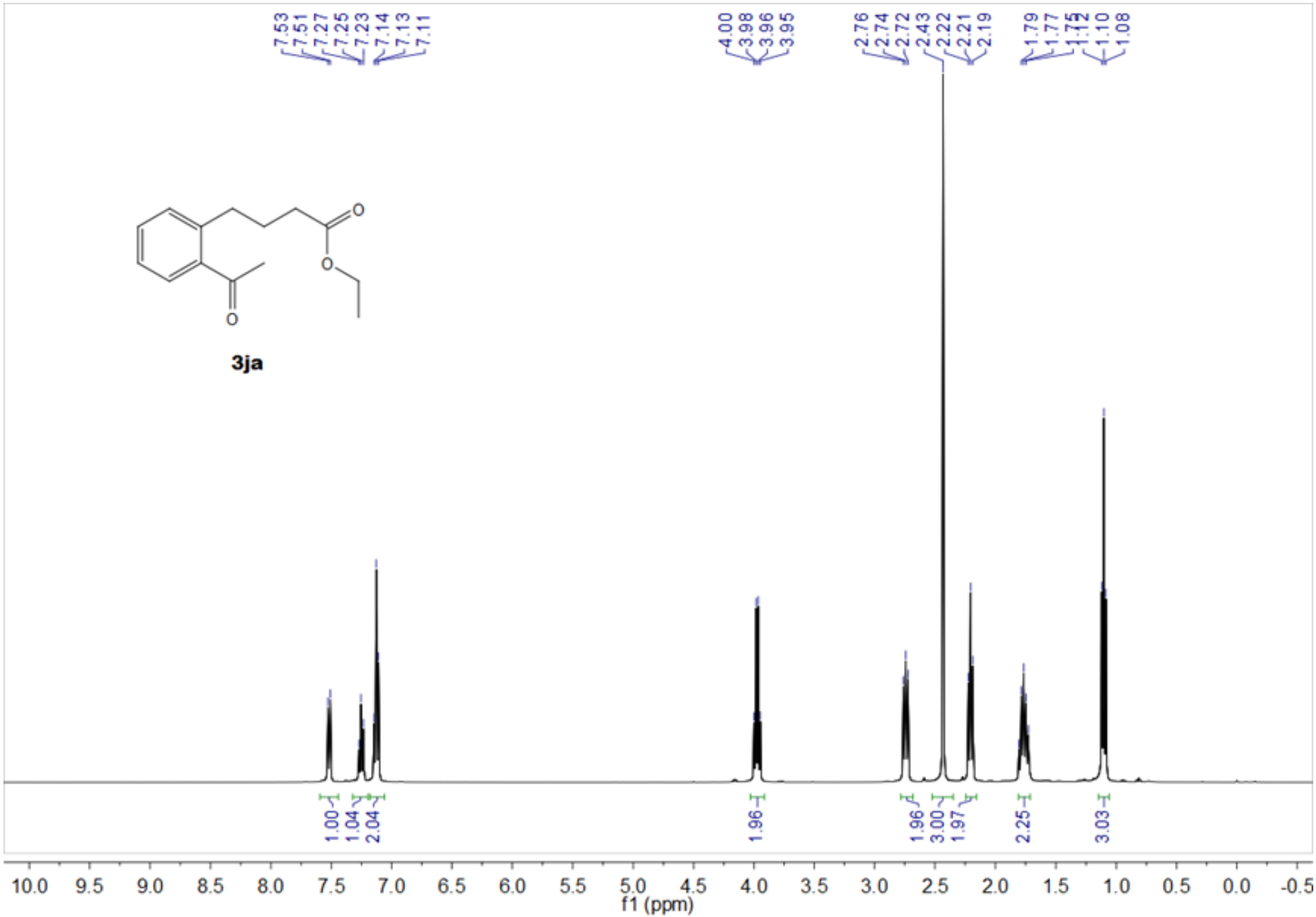
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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5.0
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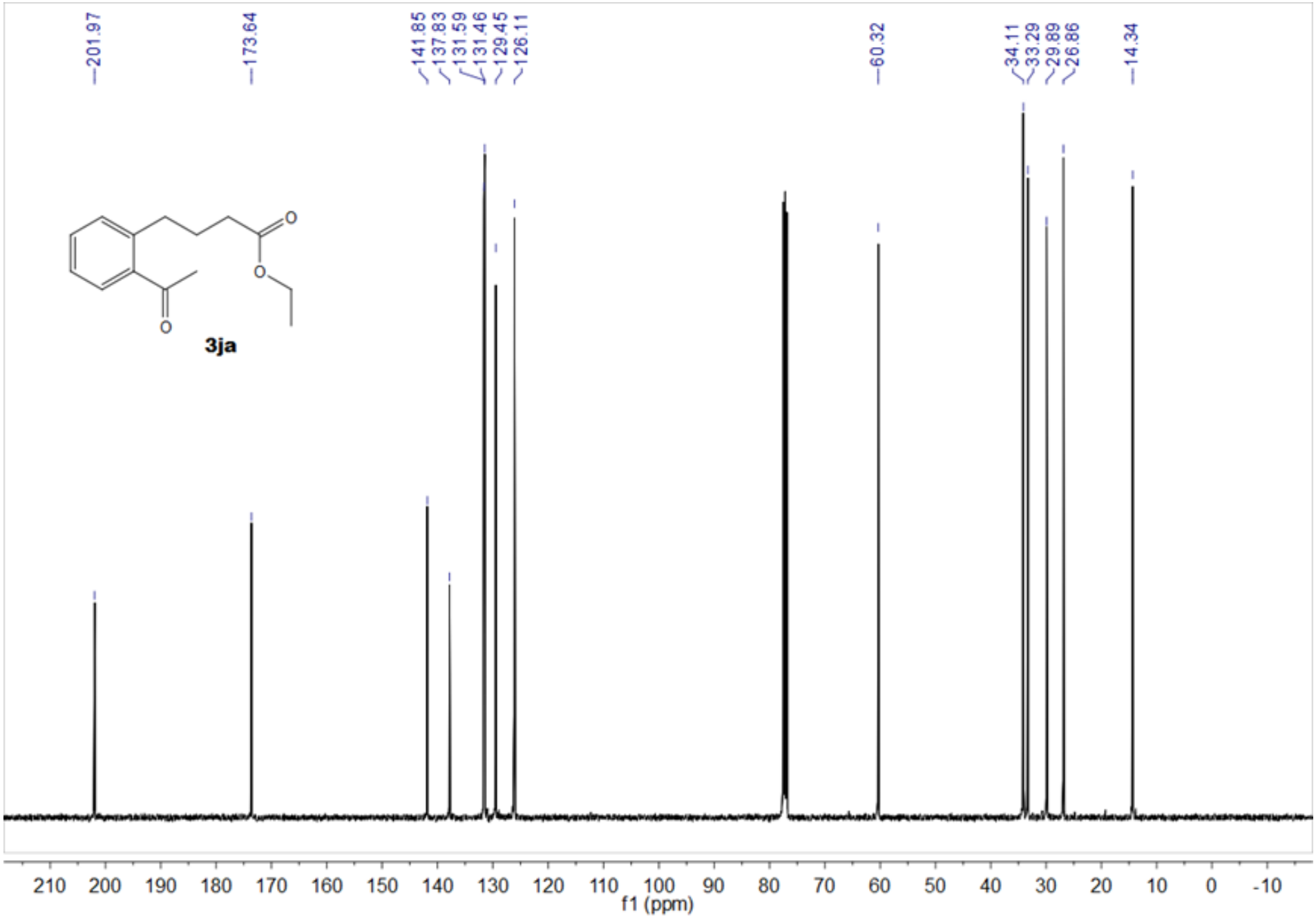
-0.5
f1 (ppm)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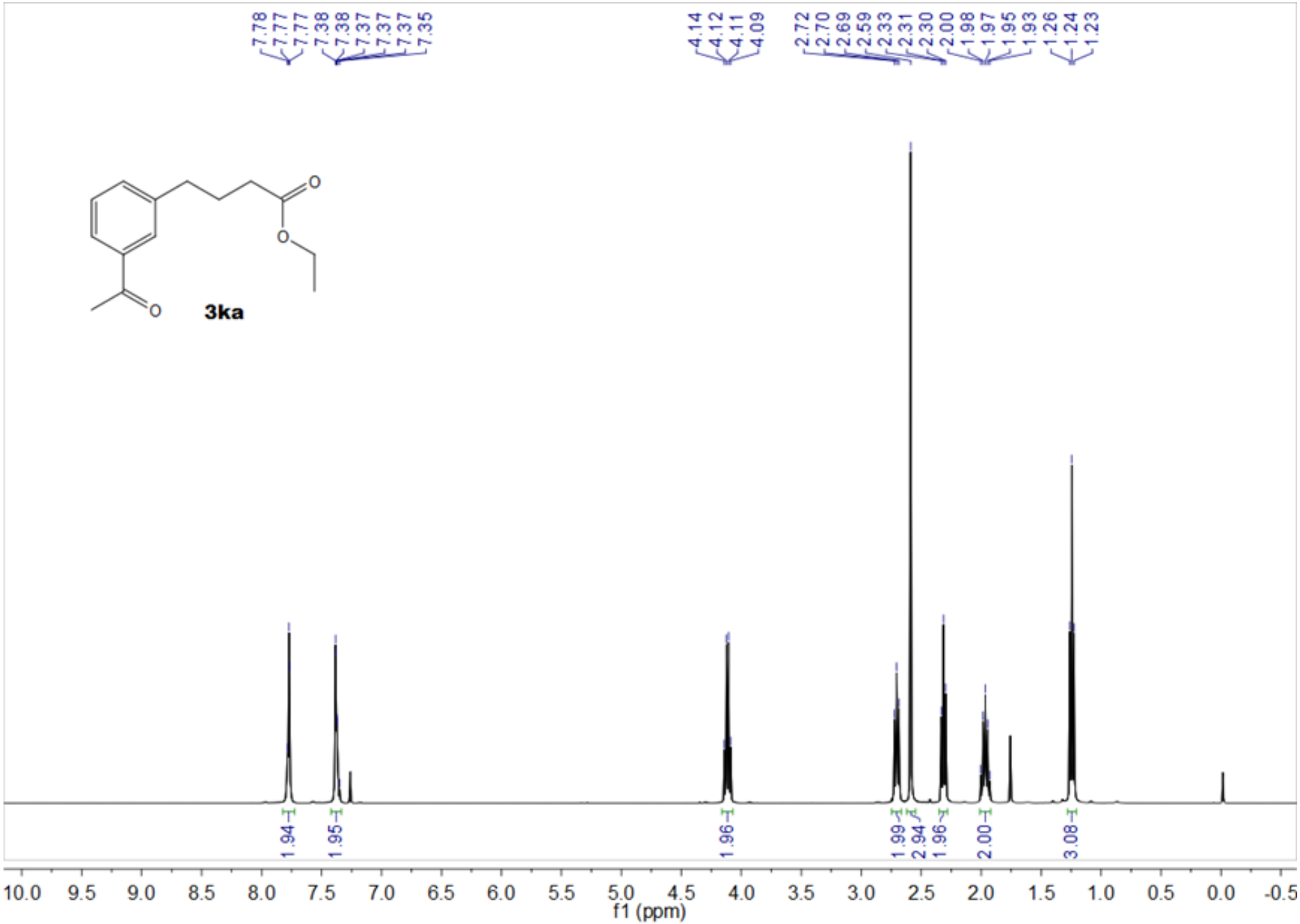
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1.23
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O
O
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2.94
1.96
2.00
3.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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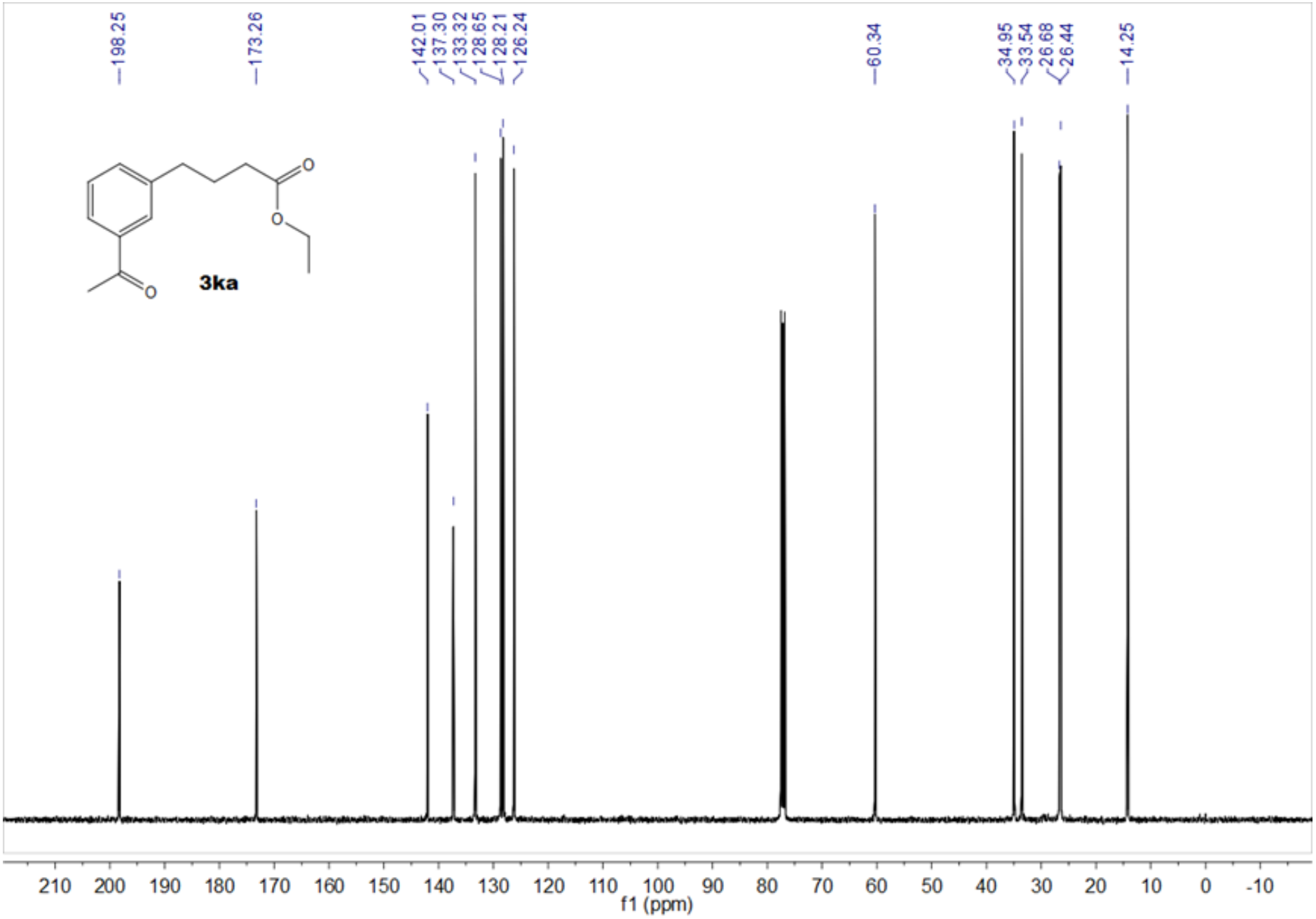
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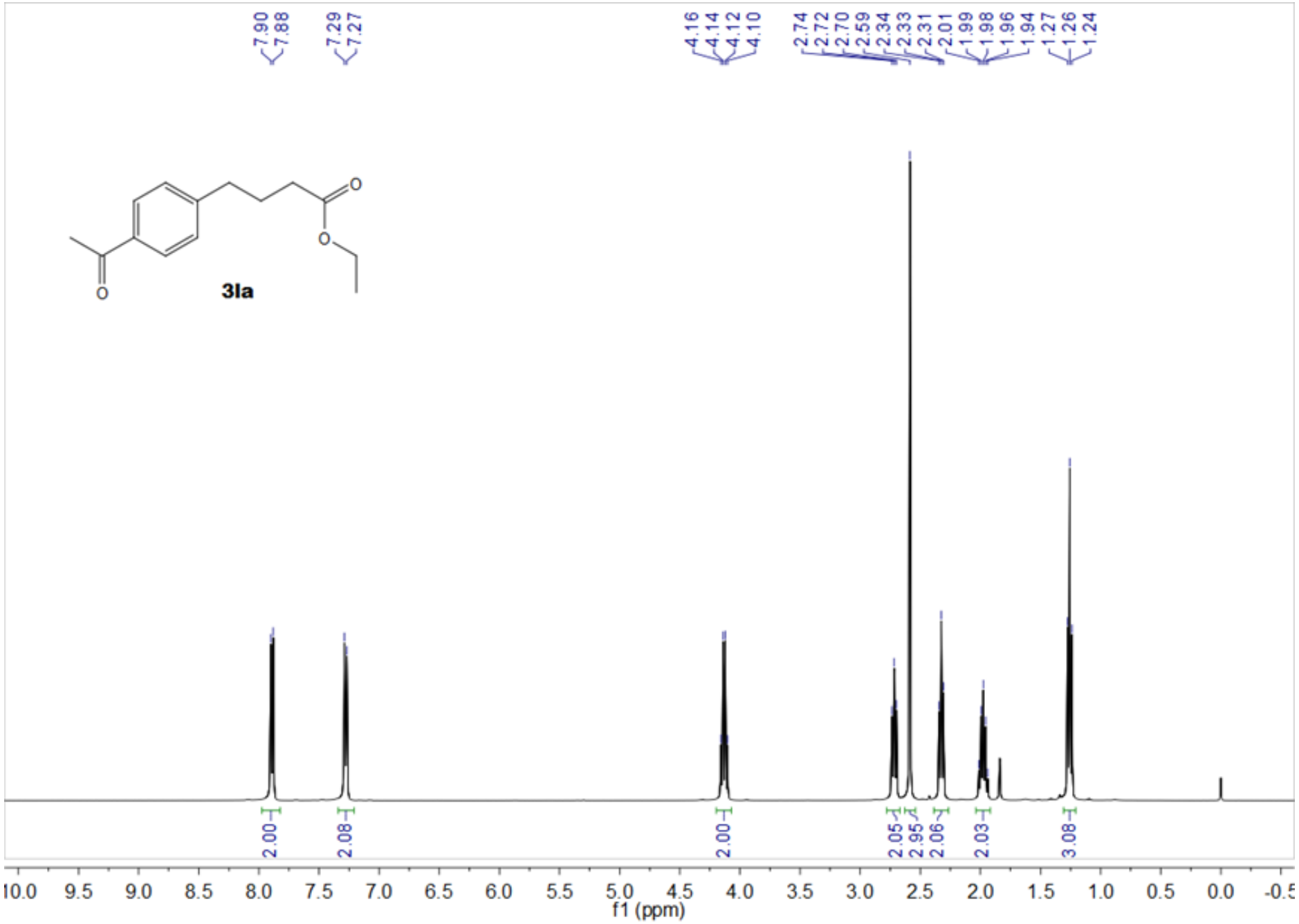
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O
O
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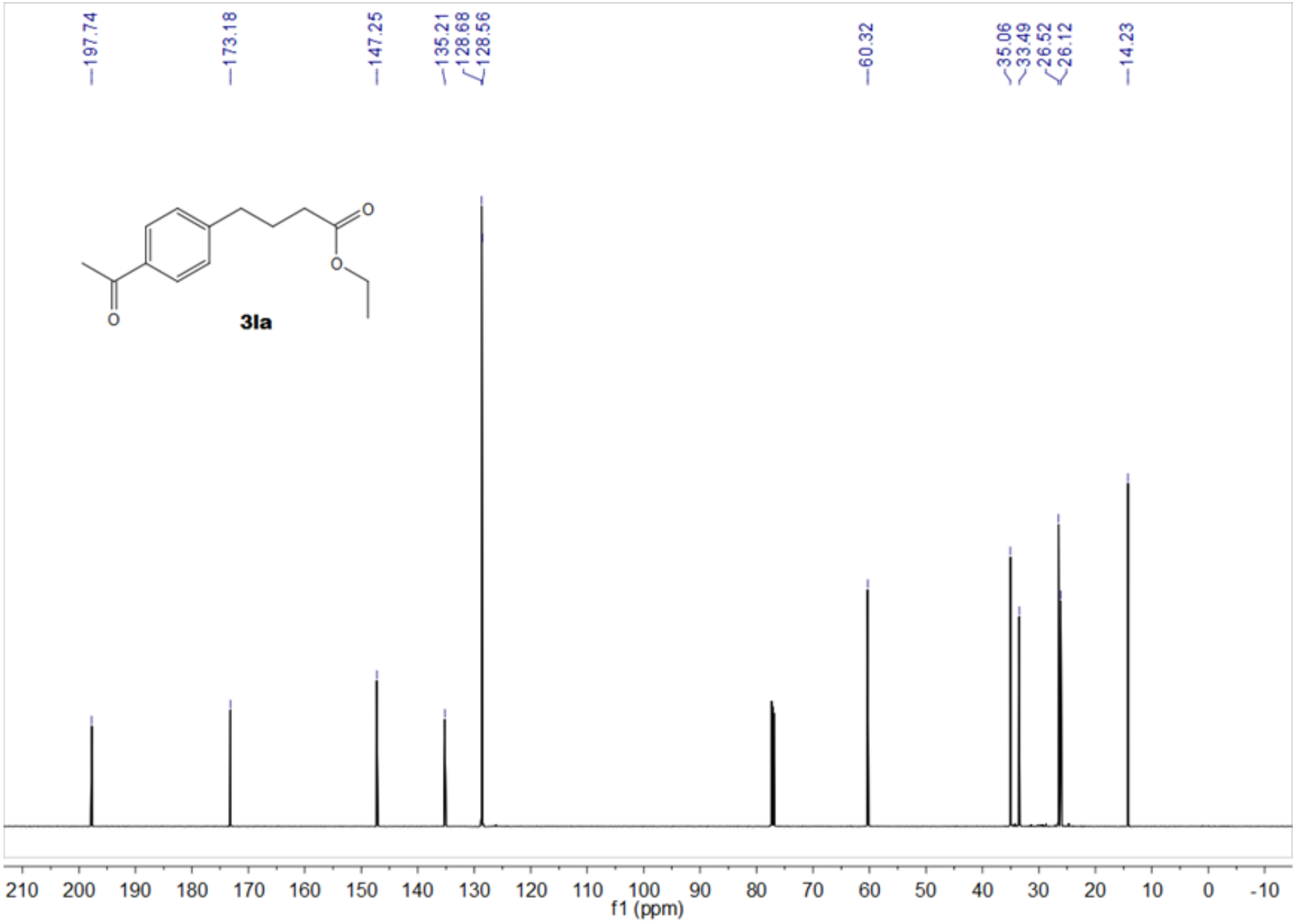
f1 (ppm)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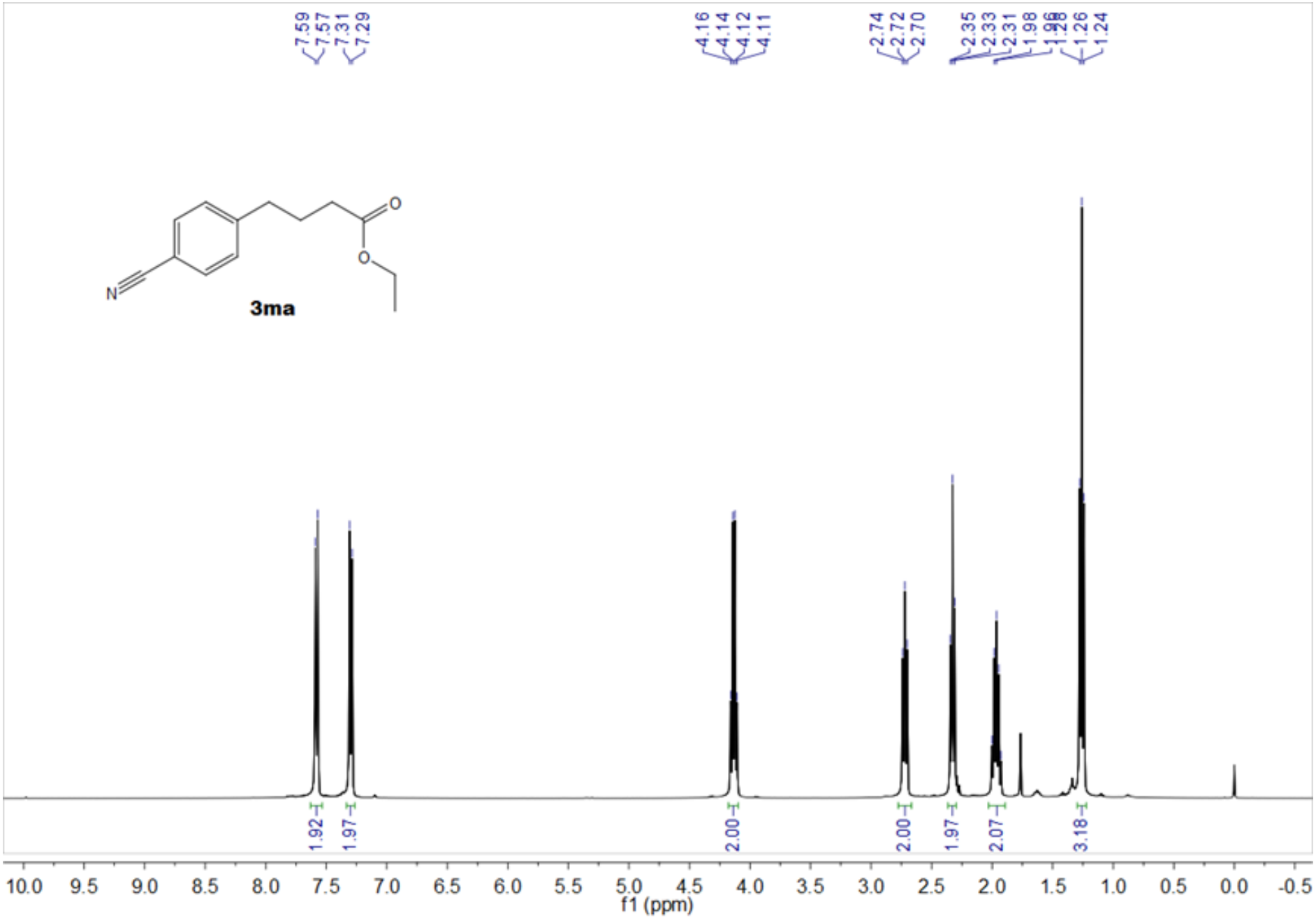
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1.24
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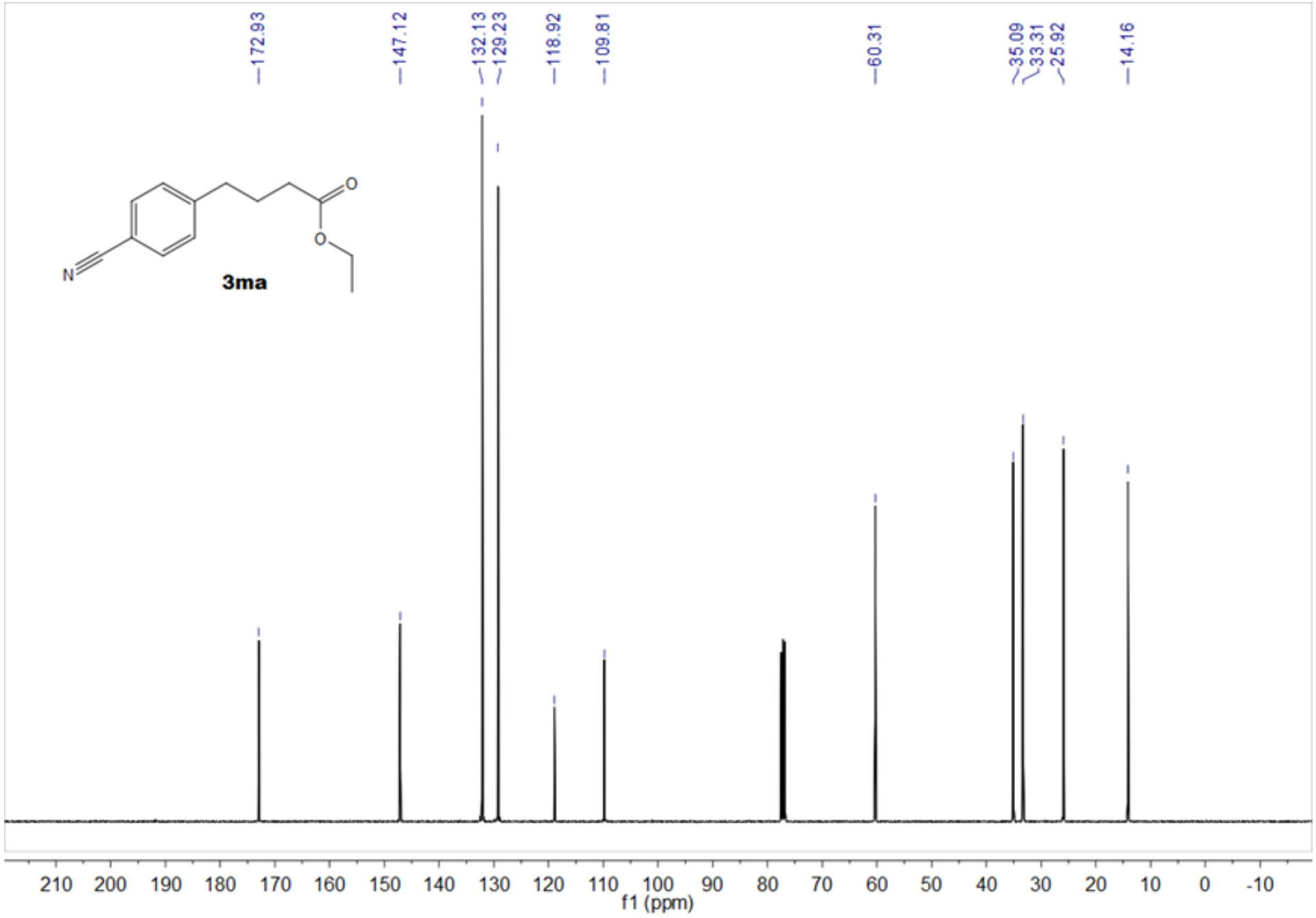
1.97
2.07
3.18
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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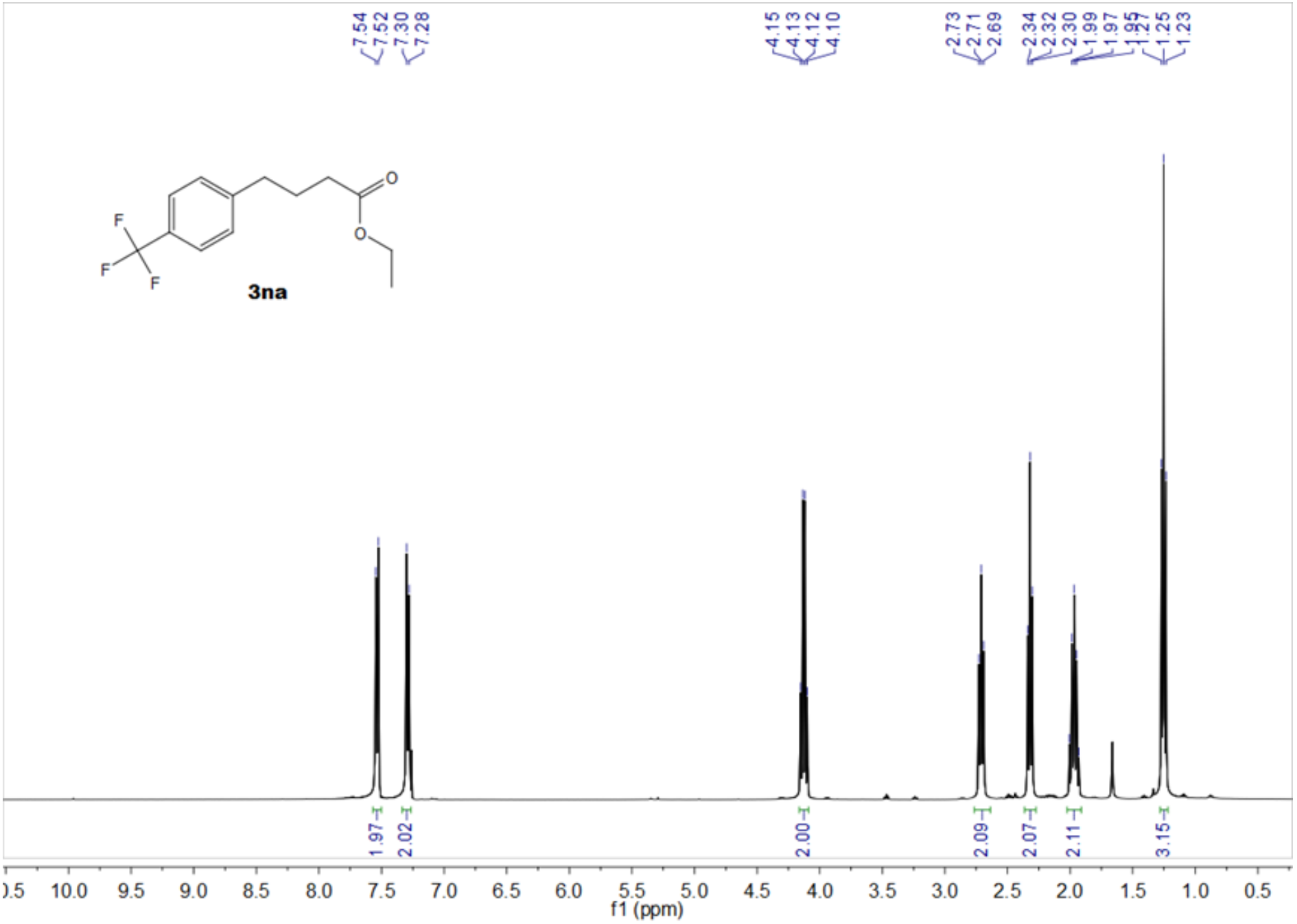
f1 (ppm)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1.97
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1.27
1.25
1.23
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2.07
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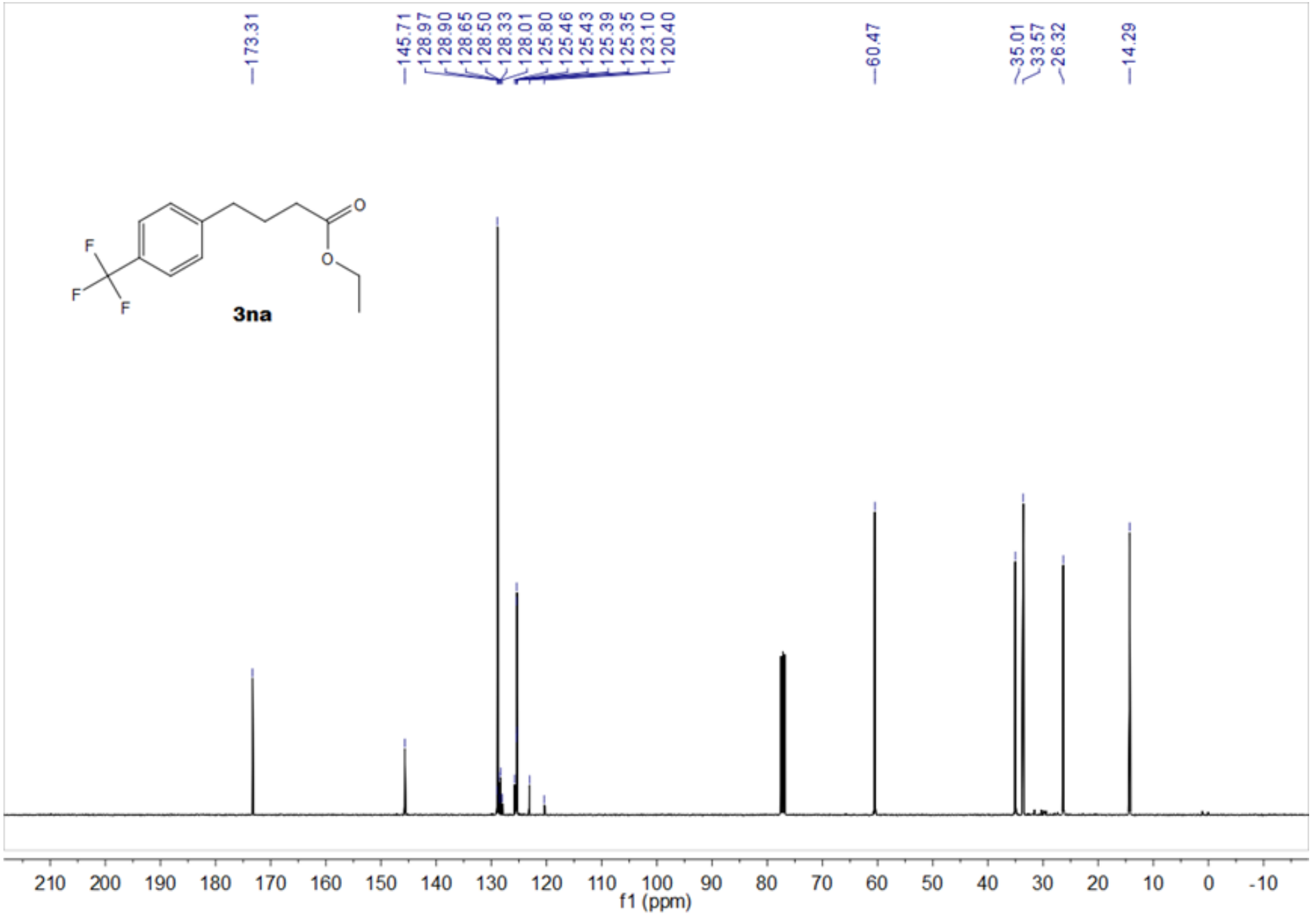
f1 (ppm)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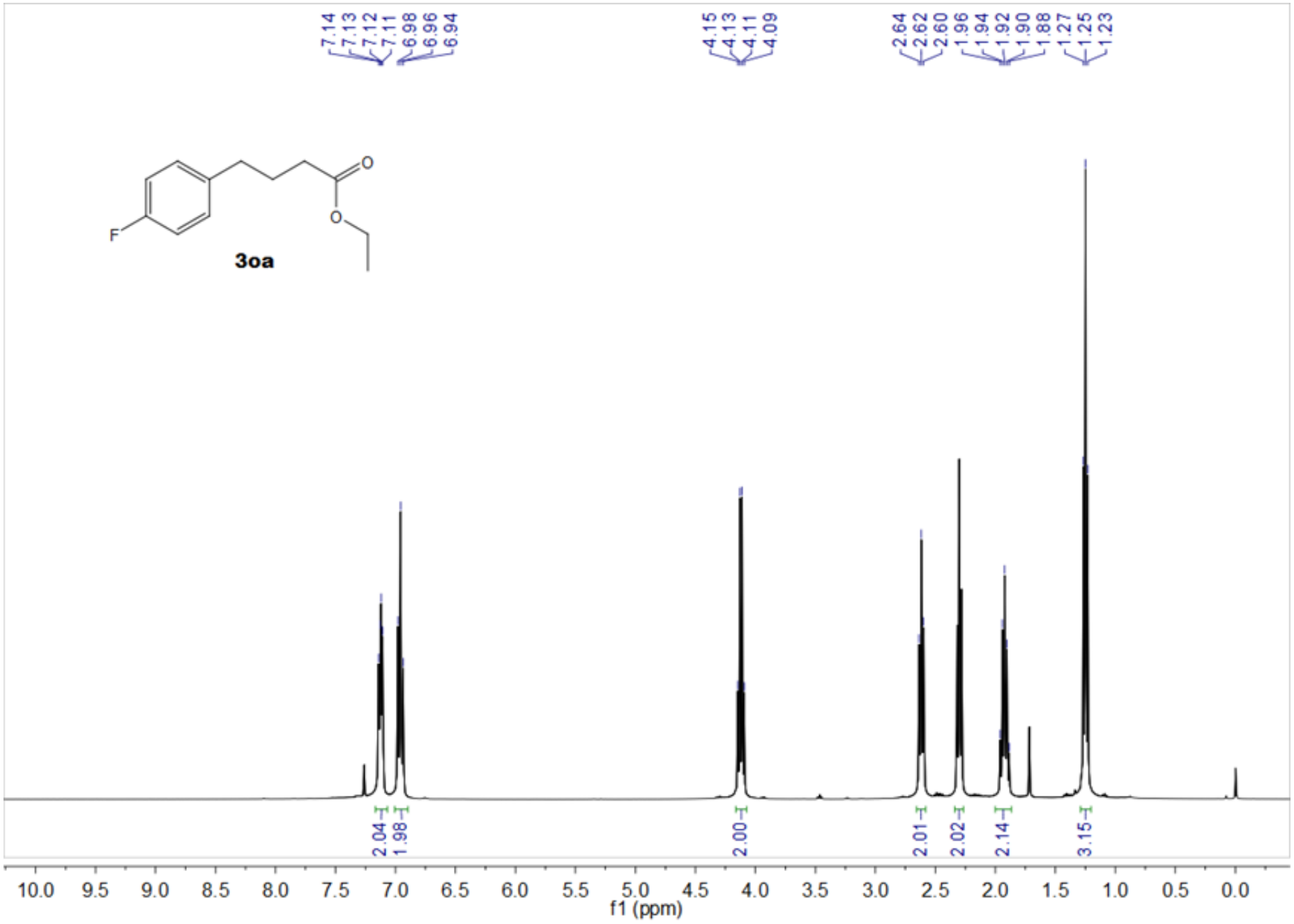
f1 (ppm)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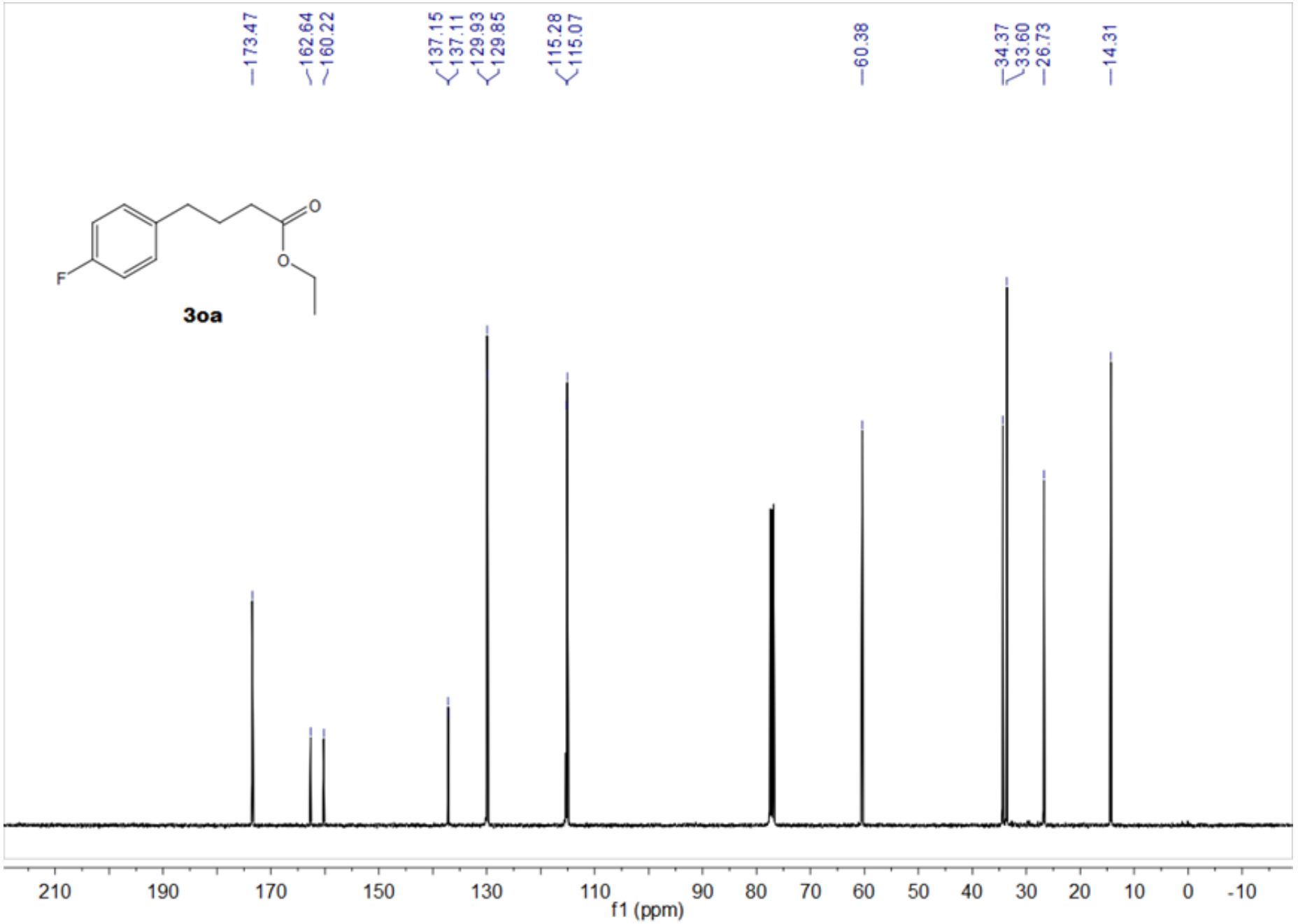
f1 (ppm)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F
O
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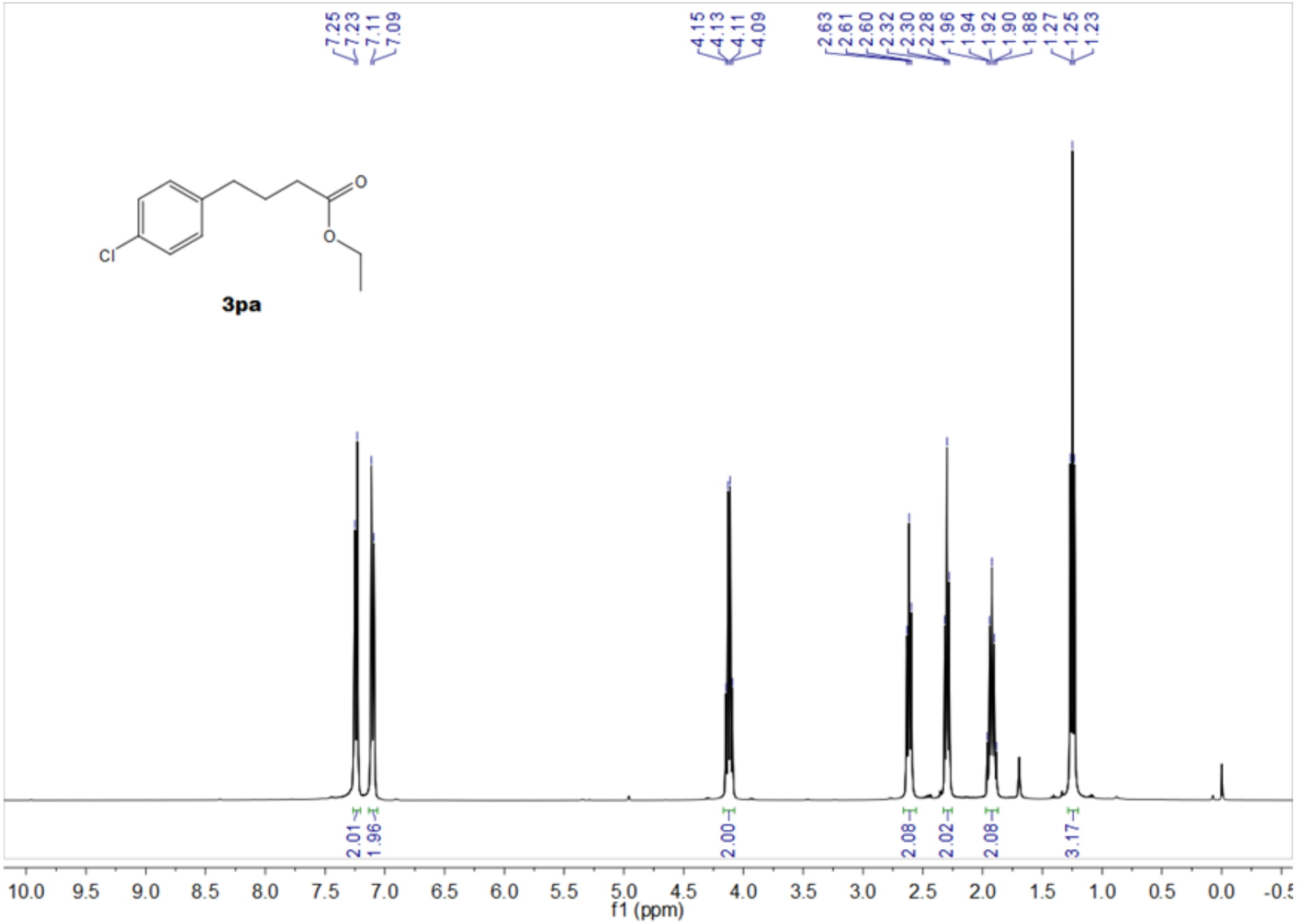
f1 (ppm)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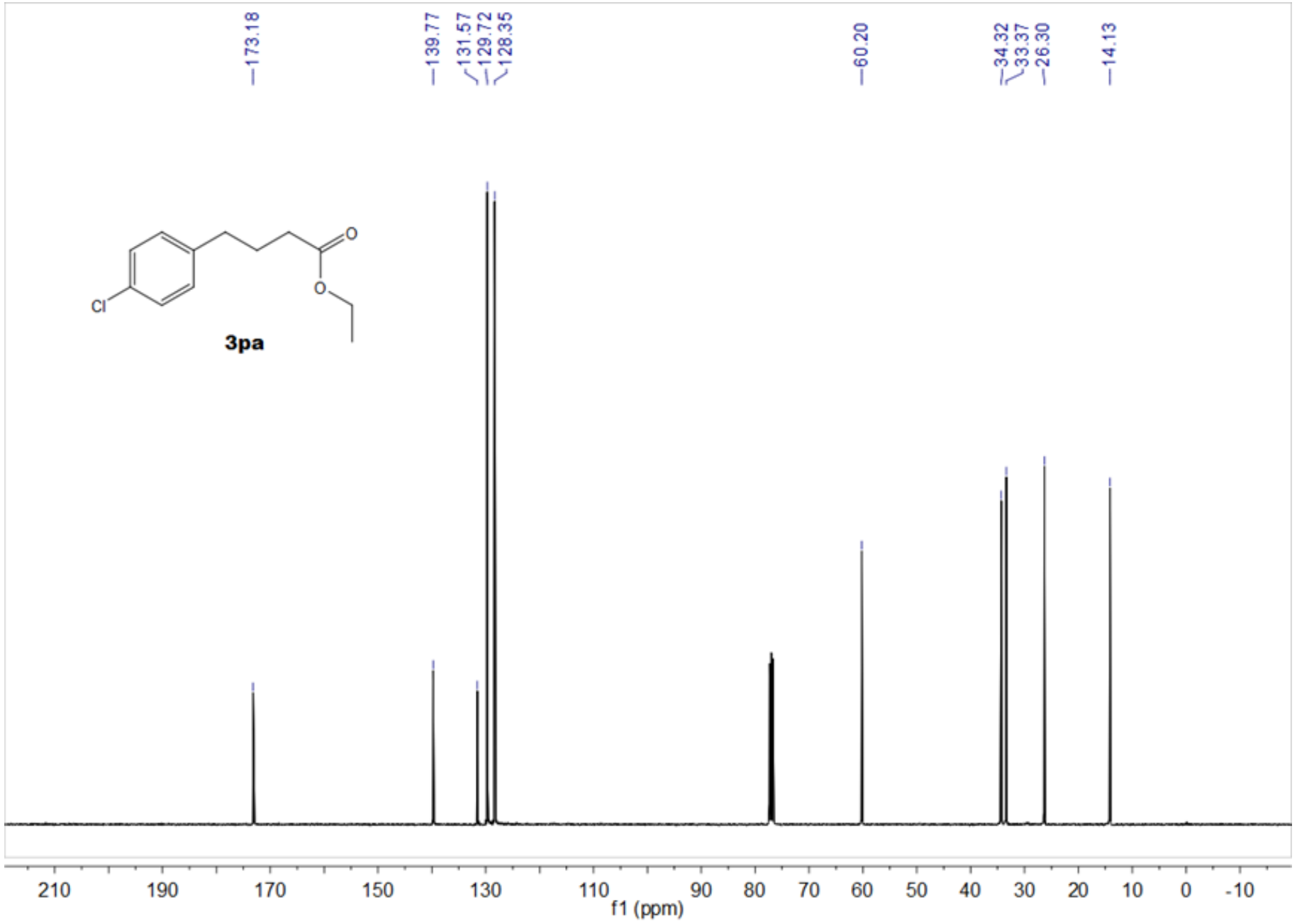
f1 (ppm)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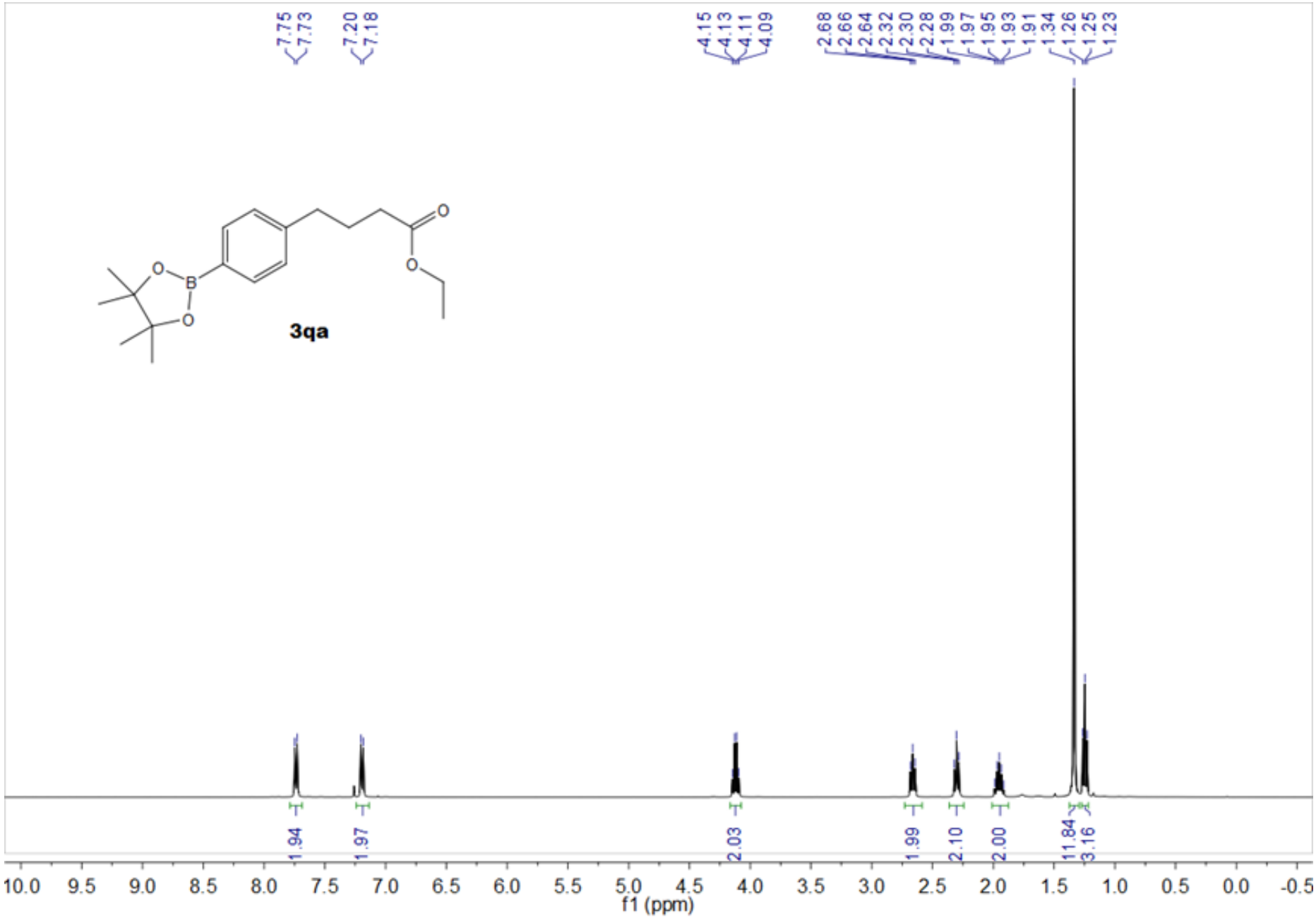
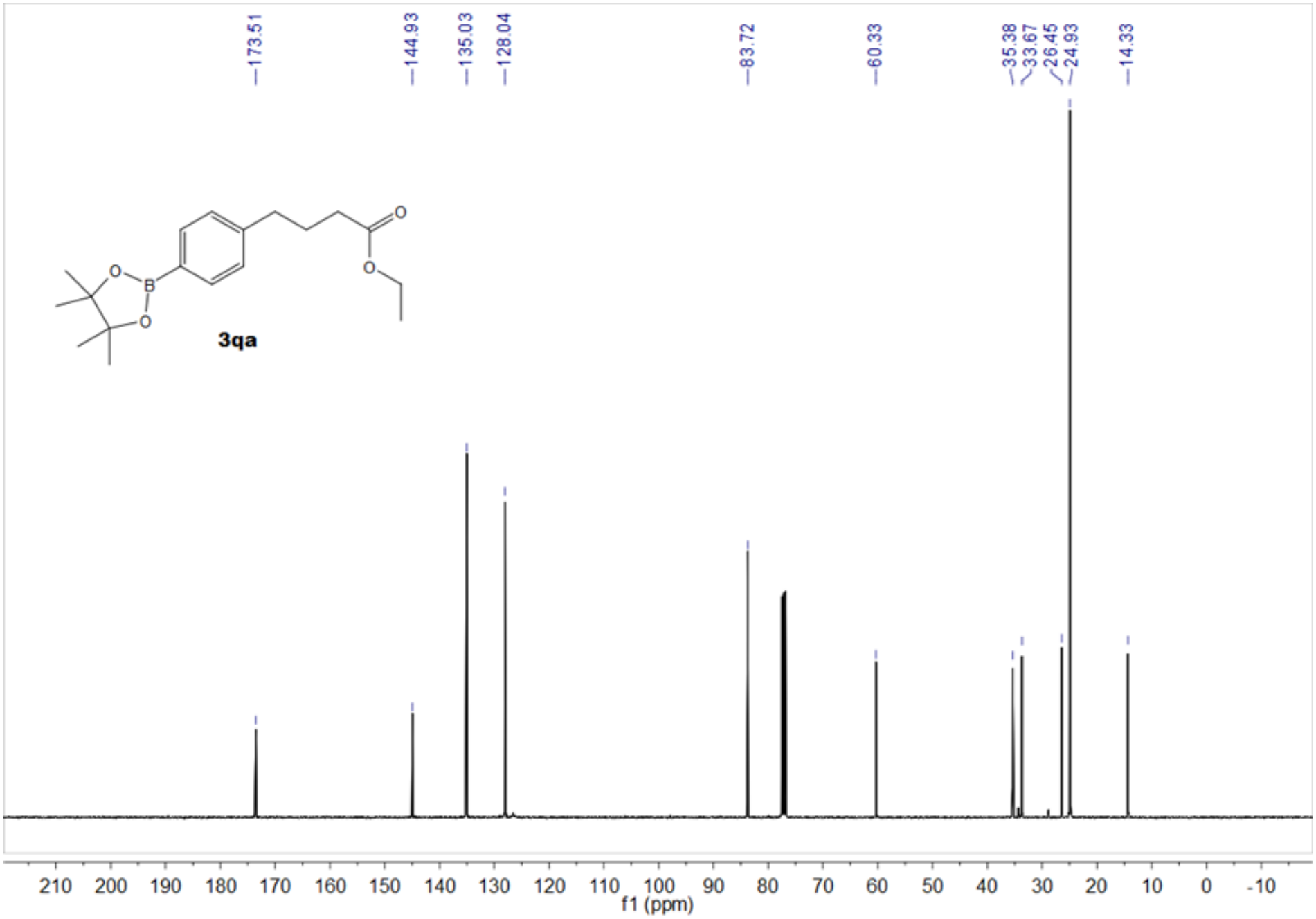
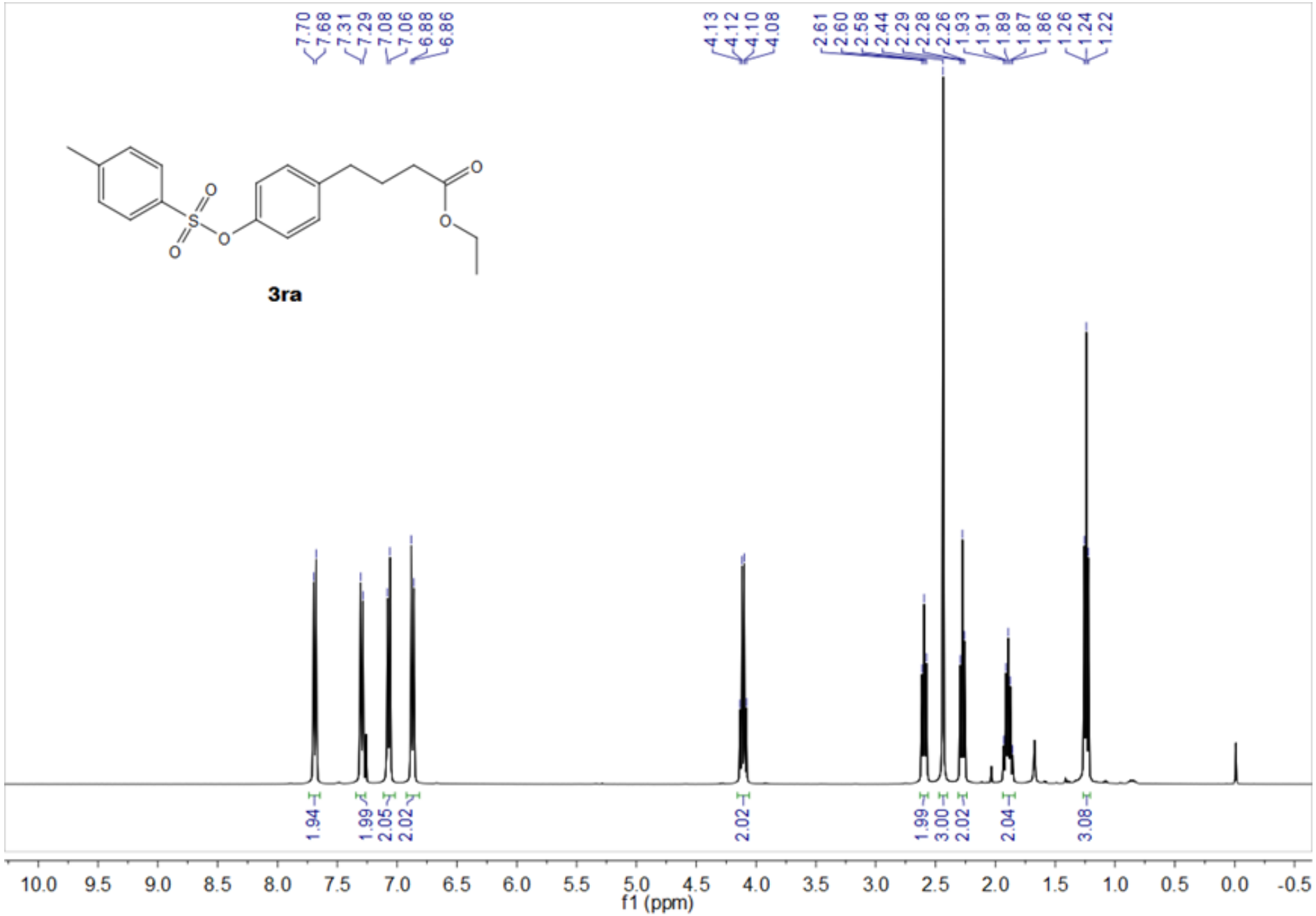
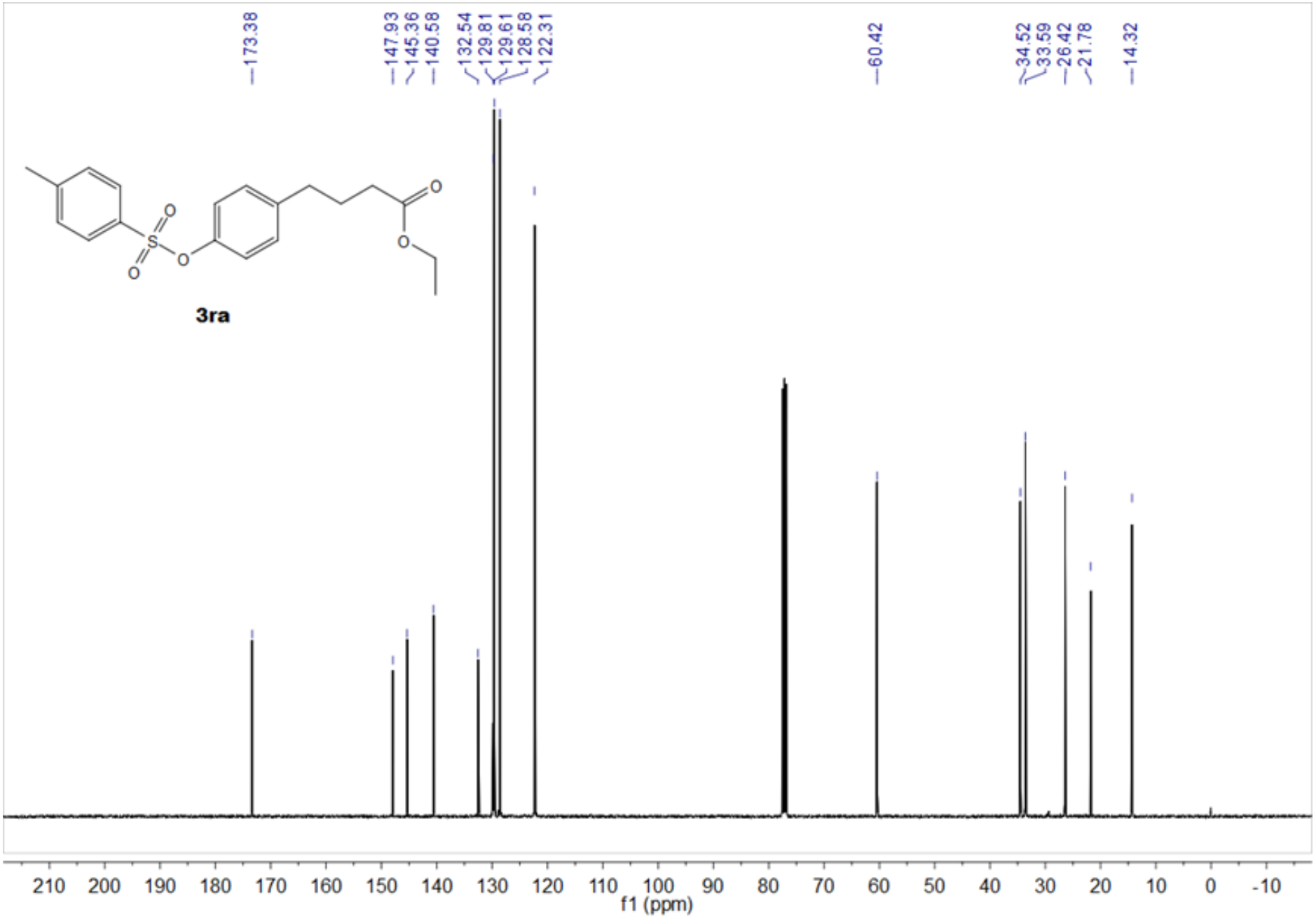
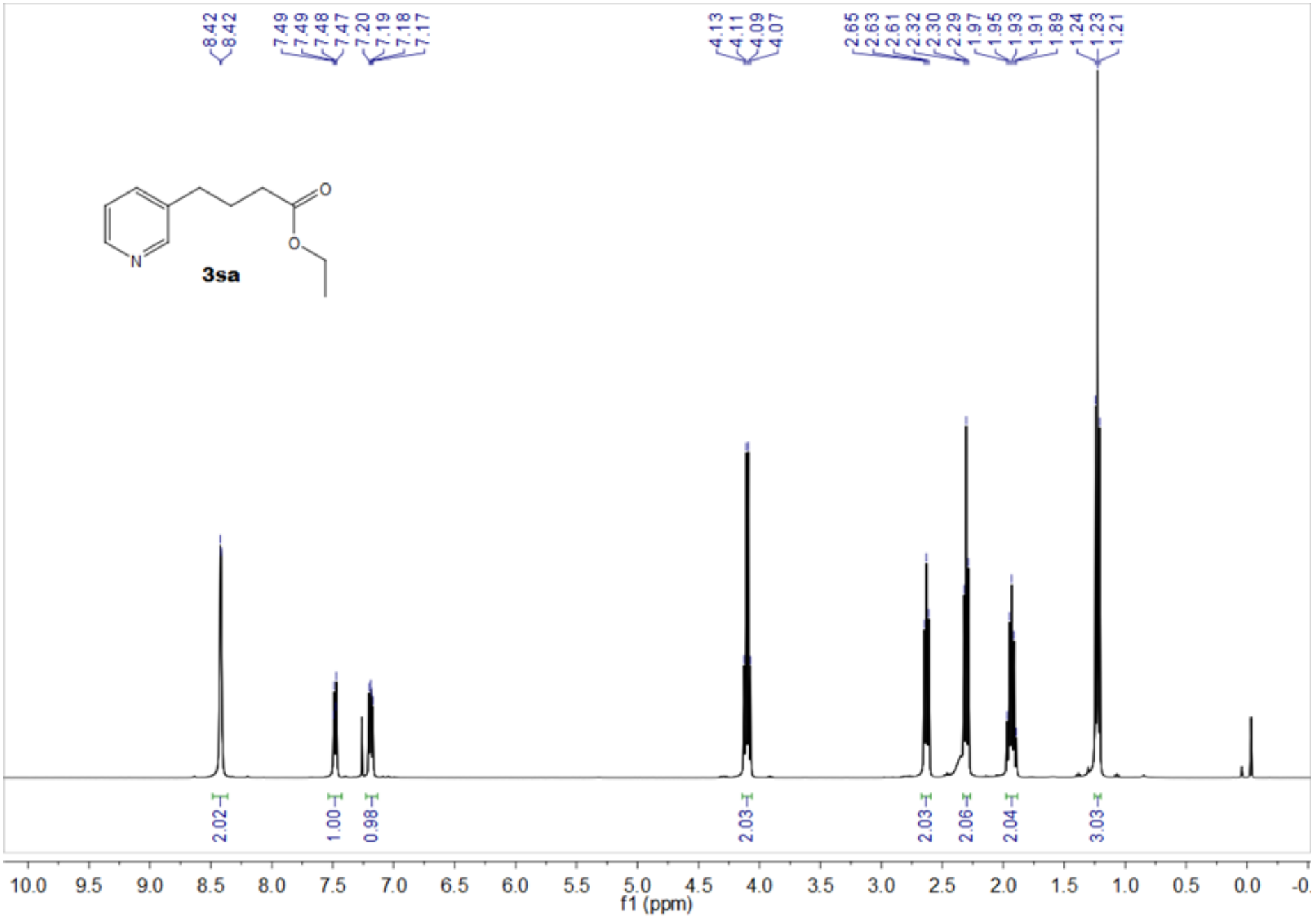
60.20
34.32
33.37
26.30
14.13
Cl
O
O
3pa
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

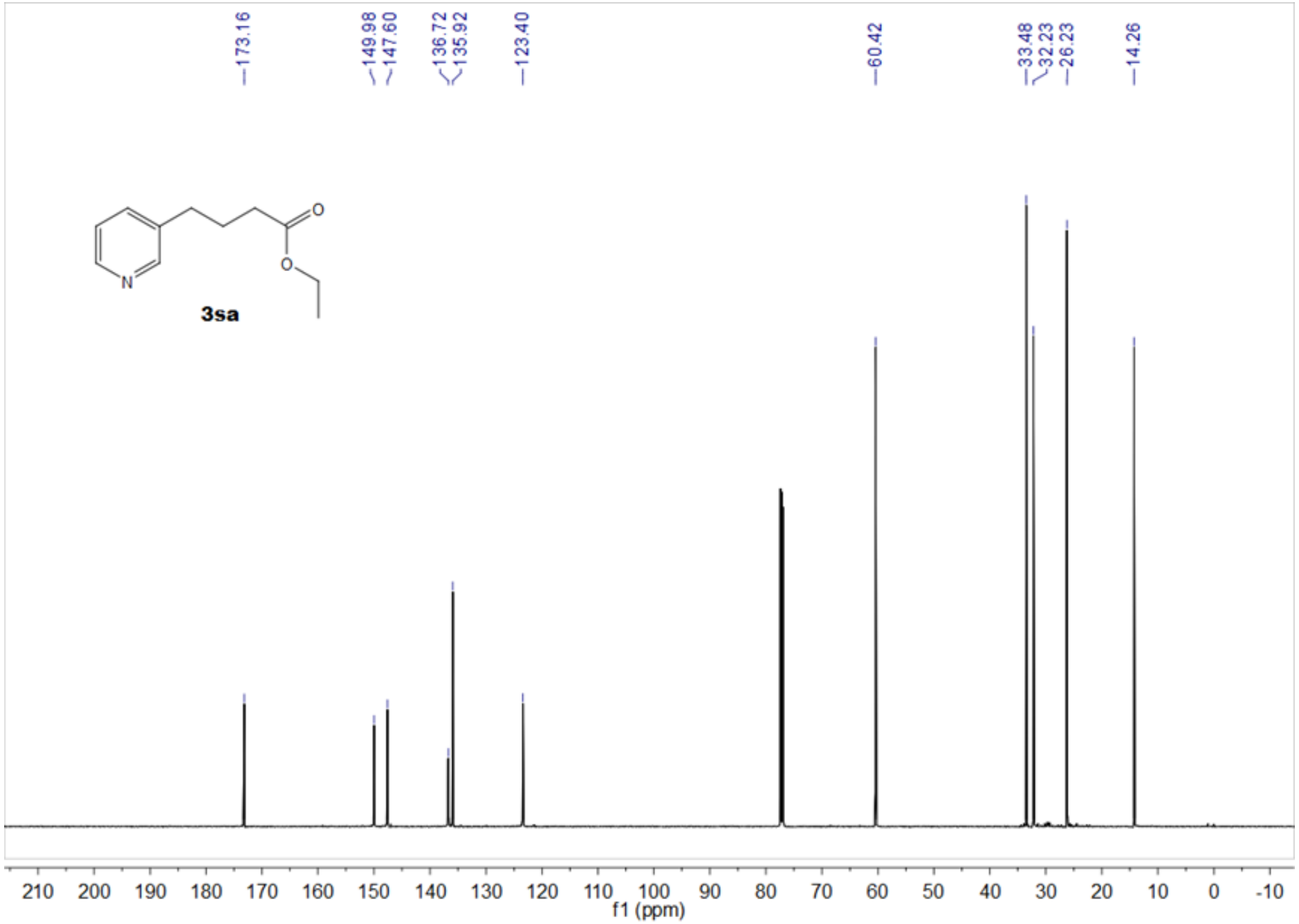

7.75
7.73
7.20
7.18
4.15
4.13
4.11
4.09
2.68
2.66
2.64
2.32
2.30
2.28
1.99
1.97
1.95
1.93
1.91
1.34
1.26
1.25
1.23
O
O
B
O
O
O
3qa
1.94
1.97
2.03
1.99
2.10
2.00
11.84
3.16
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

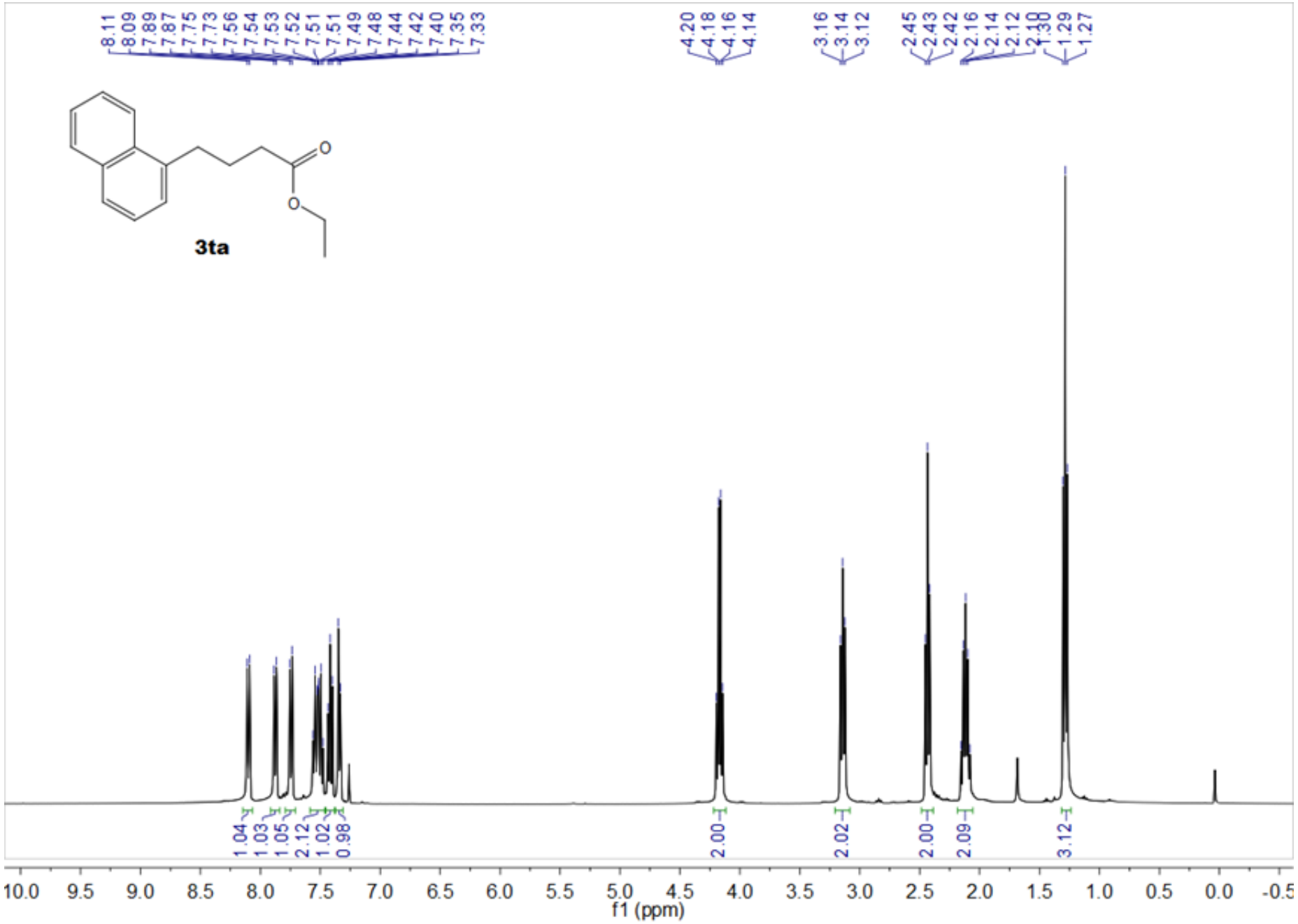

173.51
144.93
135.03
128.04
83.72
60.33
35.38
33.67
26.45
24.93
14.33
O
O
O
B
O
3qa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

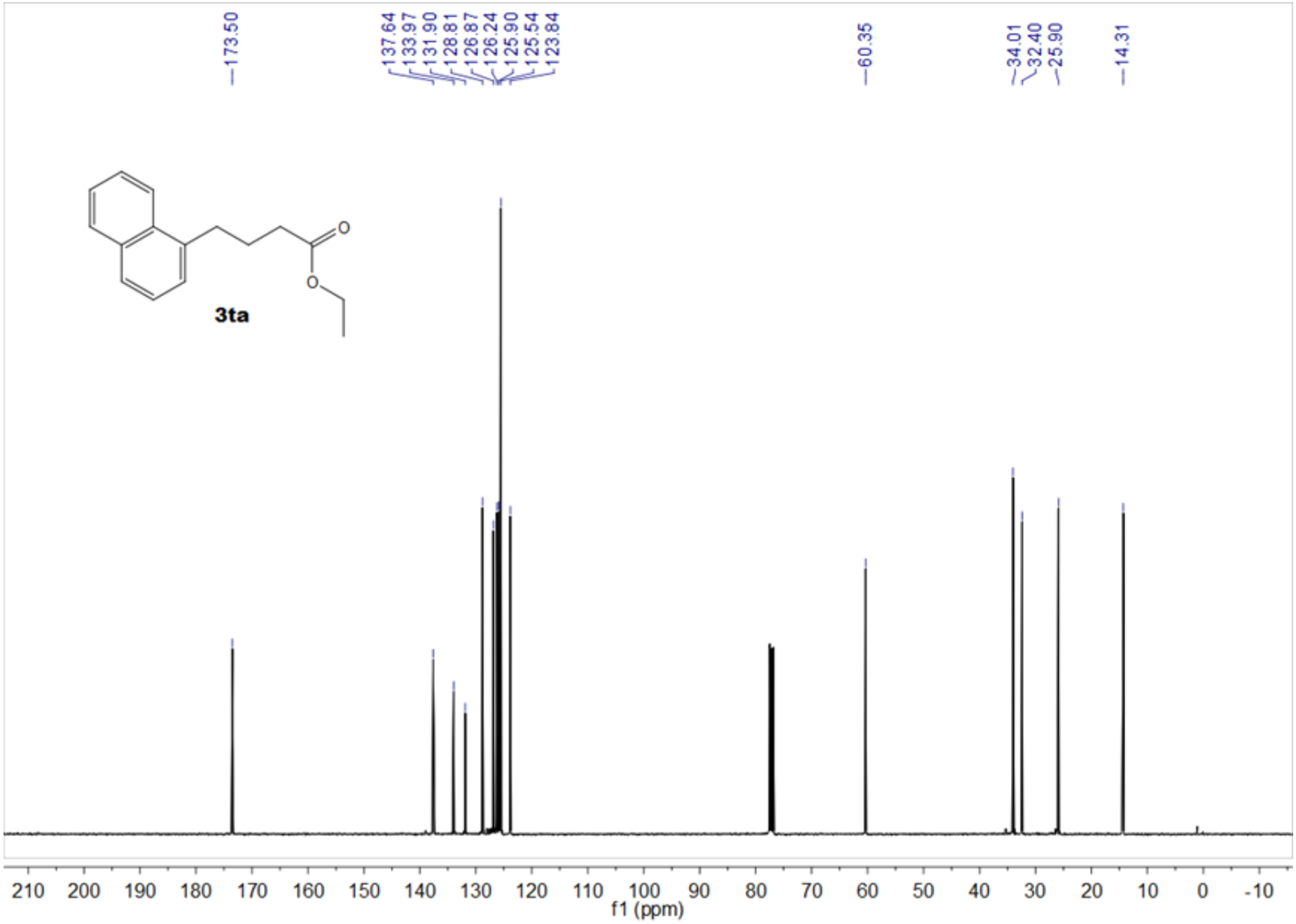

7.70
7.68
7.31
7.29
7.08
7.06
6.88
6.86
4.13
4.12
4.10
4.08
2.61
2.60
2.58
2.44
2.29
2.28
2.26
1.93
1.91
1.89
1.87
1.86
1.26
1.24
1.22
O
S
O
O
O
O
3ra
1.94
1.99
2.05
2.02
2.02
1.99
3.00
2.02
2.04
3.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

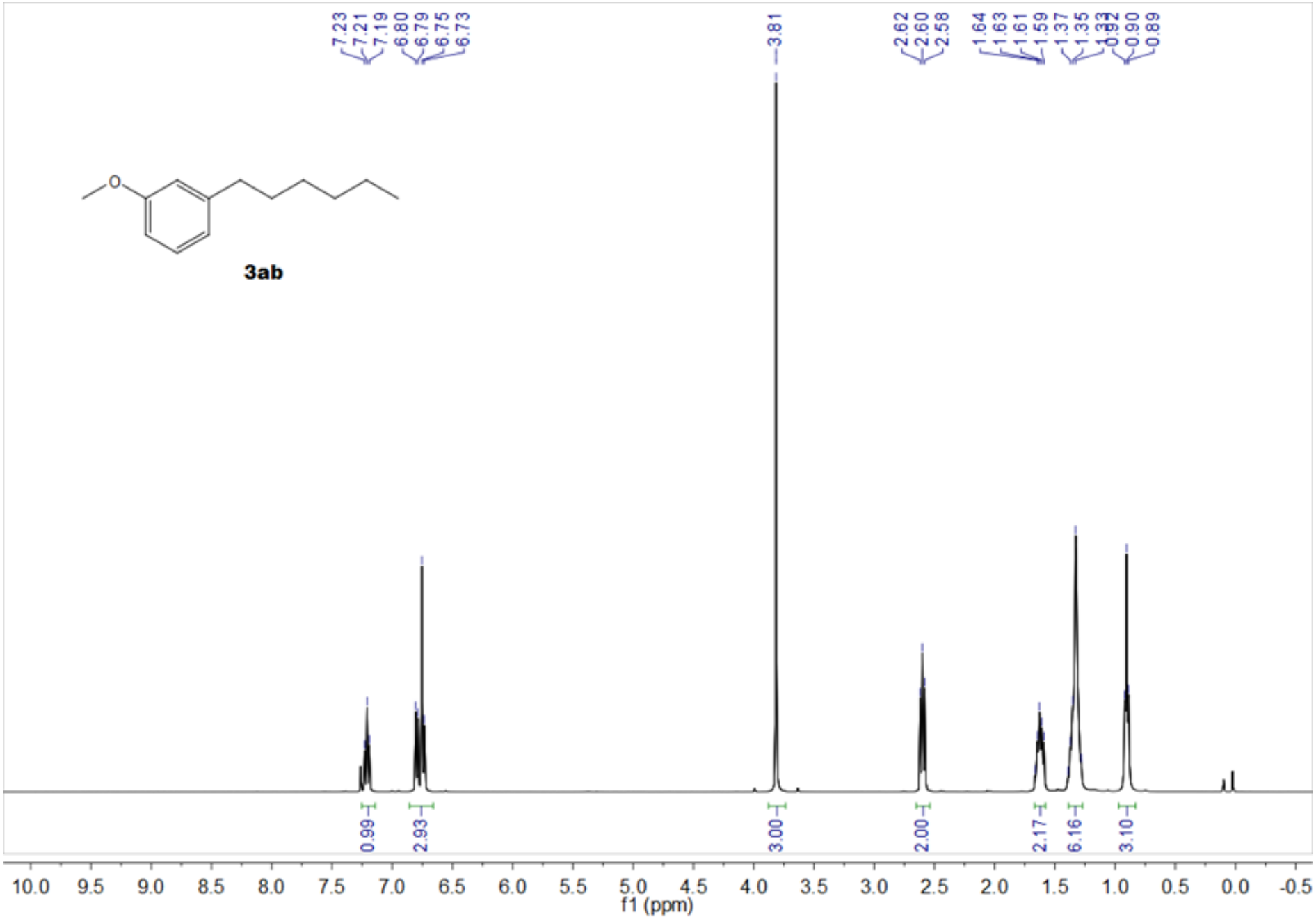

173.38
147.93
145.36
140.58
132.54
129.81
129.61
128.58
122.31
60.42
34.52
33.59
26.42
21.78
14.32
3ra
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

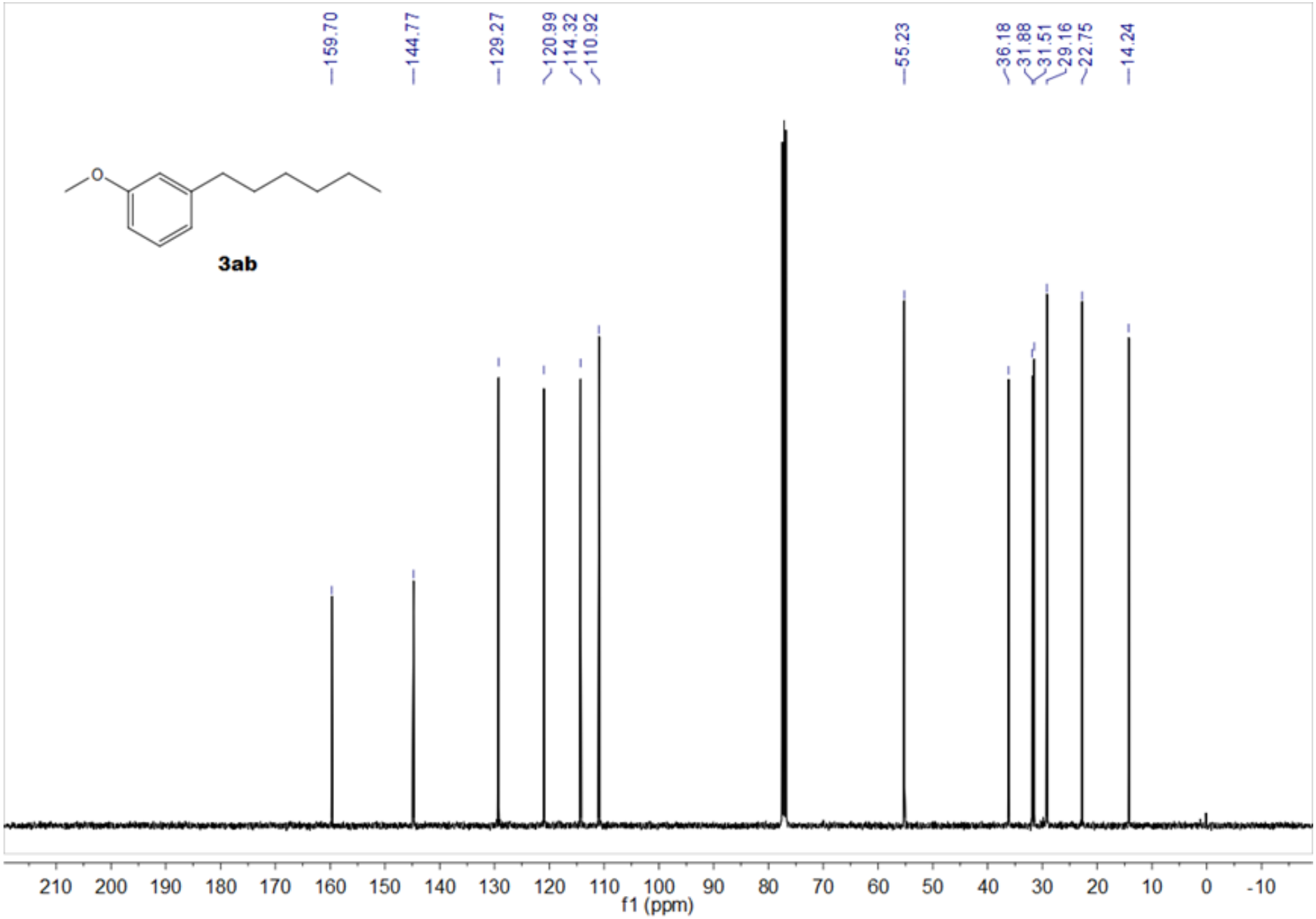

8.42
8.42
7.49
7.49
7.48
7.47
7.20
7.19
7.18
7.17
4.13
4.11
4.09
4.07
2.65
2.63
2.61
2.32
2.30
2.29
1.97
1.95
1.93
1.91
1.89
1.24
1.23
1.21
O
O
N
3sa
2.02
1.00
0.98
2.03
2.03
2.06
2.04
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

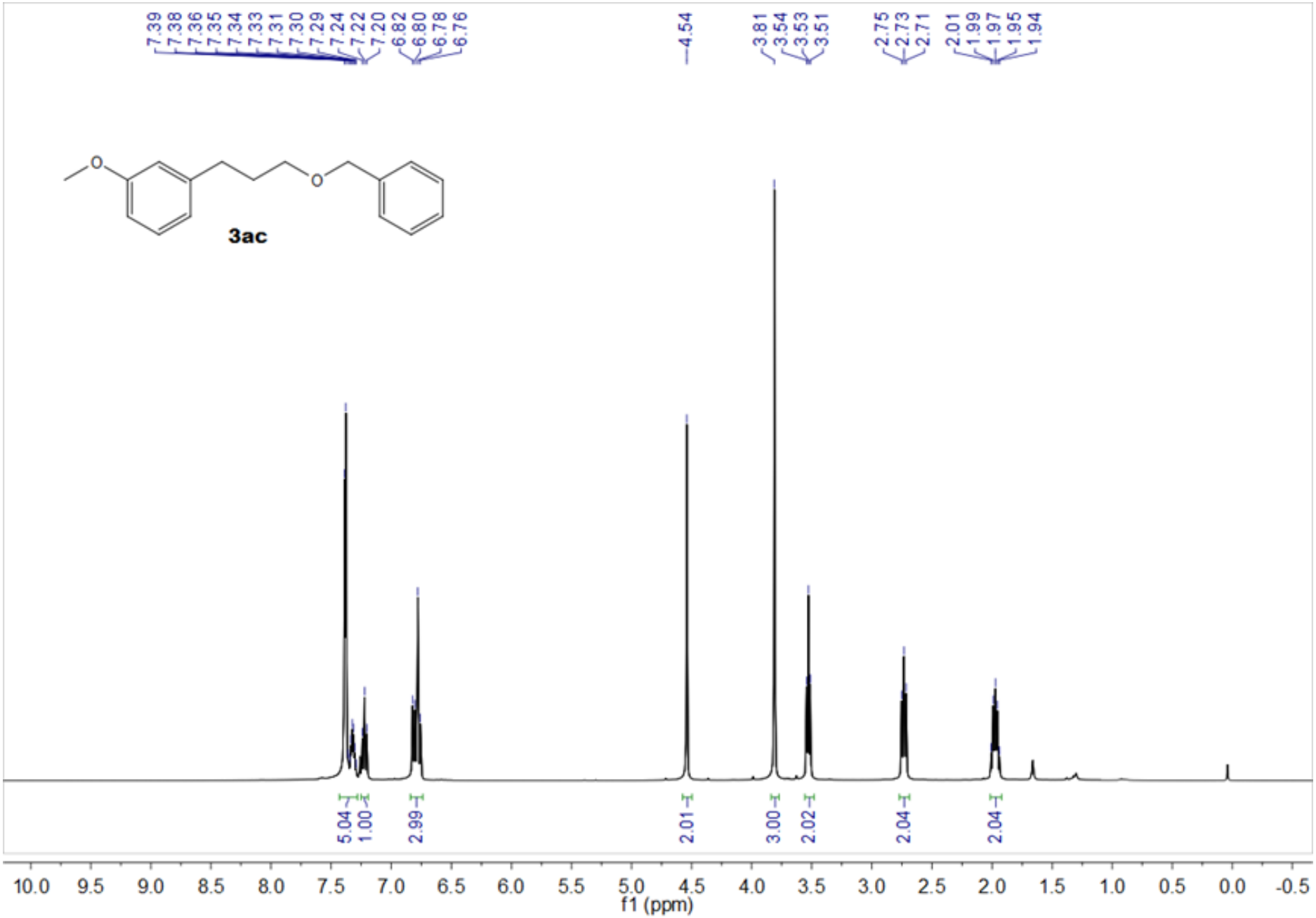

173.16
149.98
147.60
136.72
135.92
123.40
60.42
33.48
32.23
26.23
14.26
O
O
N
3sa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

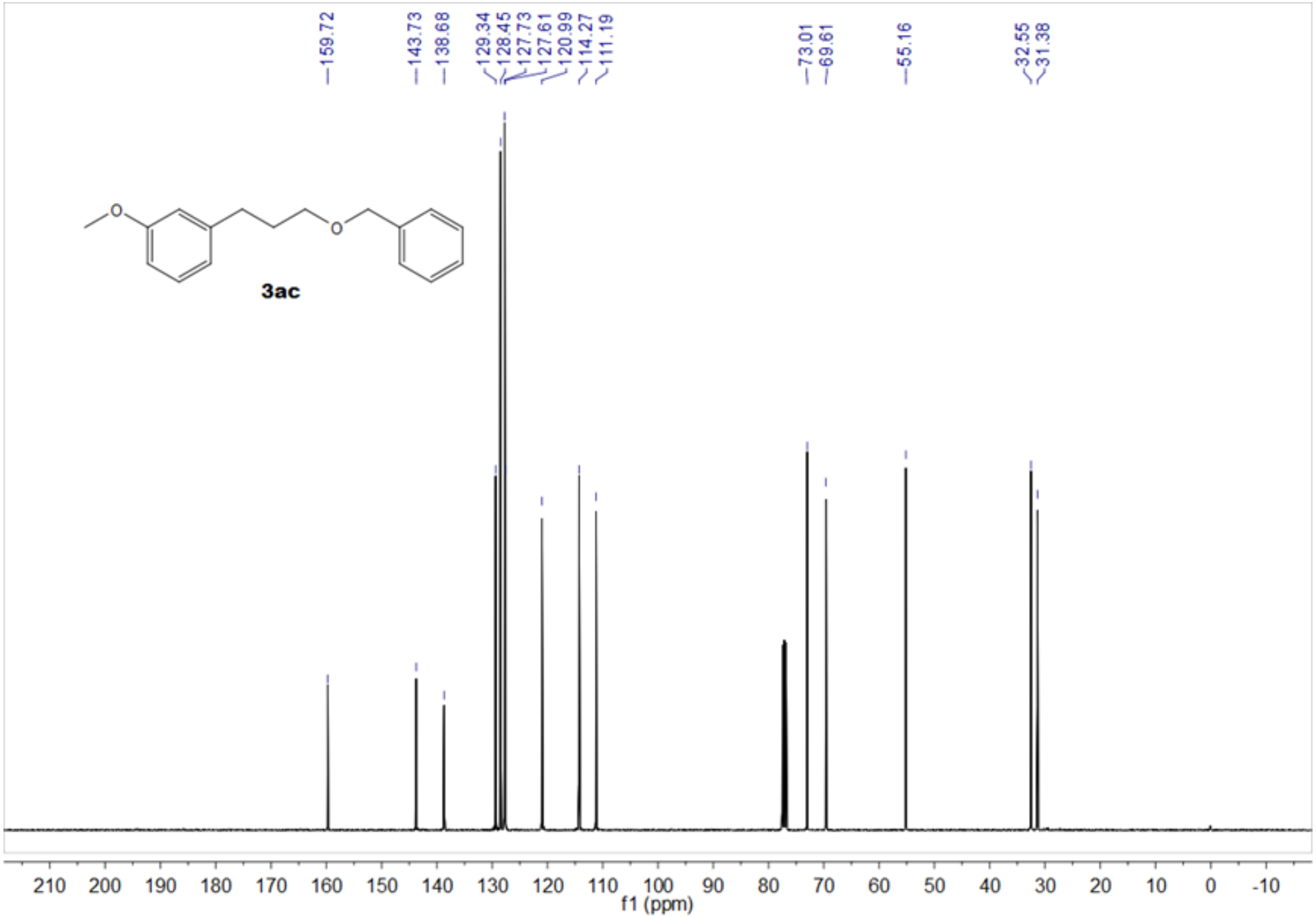

8.11
8.09
7.89
7.87
7.75
7.73
7.56
7.54
7.53
7.52
7.51
7.51
7.49
7.48
7.44
7.42
7.40
7.35
7.33
4.20
4.18
4.16
4.14
3.16
3.14
3.12
2.45
2.43
2.42
2.16
2.14
2.12
2.10
1.30
1.29
1.27
O
O
3ta
1.04
1.03
1.05
2.12
1.02
0.98
2.00
2.02
2.00
2.09
3.12
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

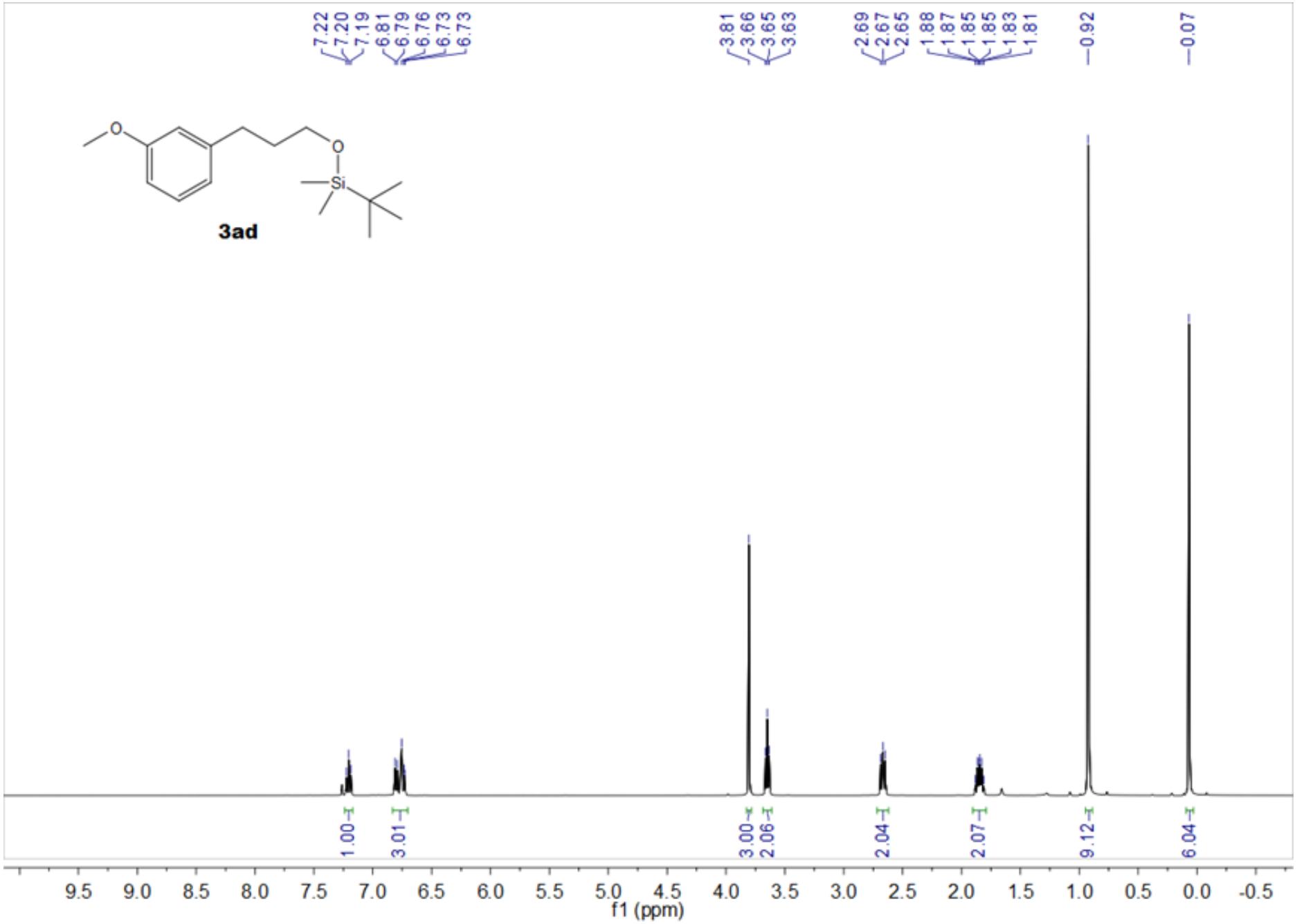

173.50
137.64
133.97
131.90
128.81
126.87
126.24
125.90
125.54
123.84
60.35
34.01
32.40
25.90
14.31
O
O
3ta
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

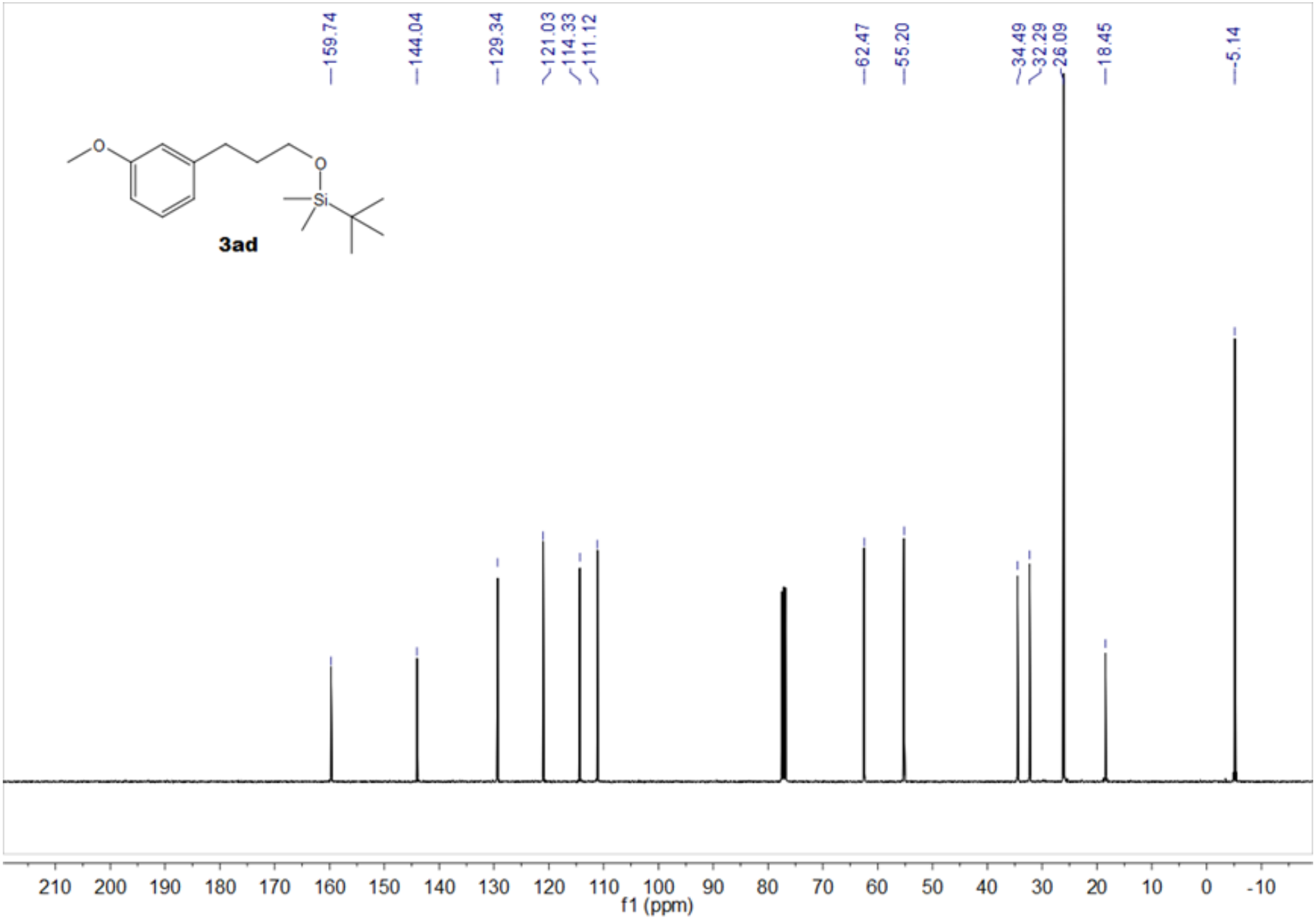

7.23
7.21
7.19
6.80
6.79
6.75
6.73
3.81
2.62
2.60
2.58
1.64
1.63
1.61
1.59
1.37
1.35
0.92
0.90
0.89
O
3ab
0.99
2.93
3.00
2.00
2.17
6.16
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

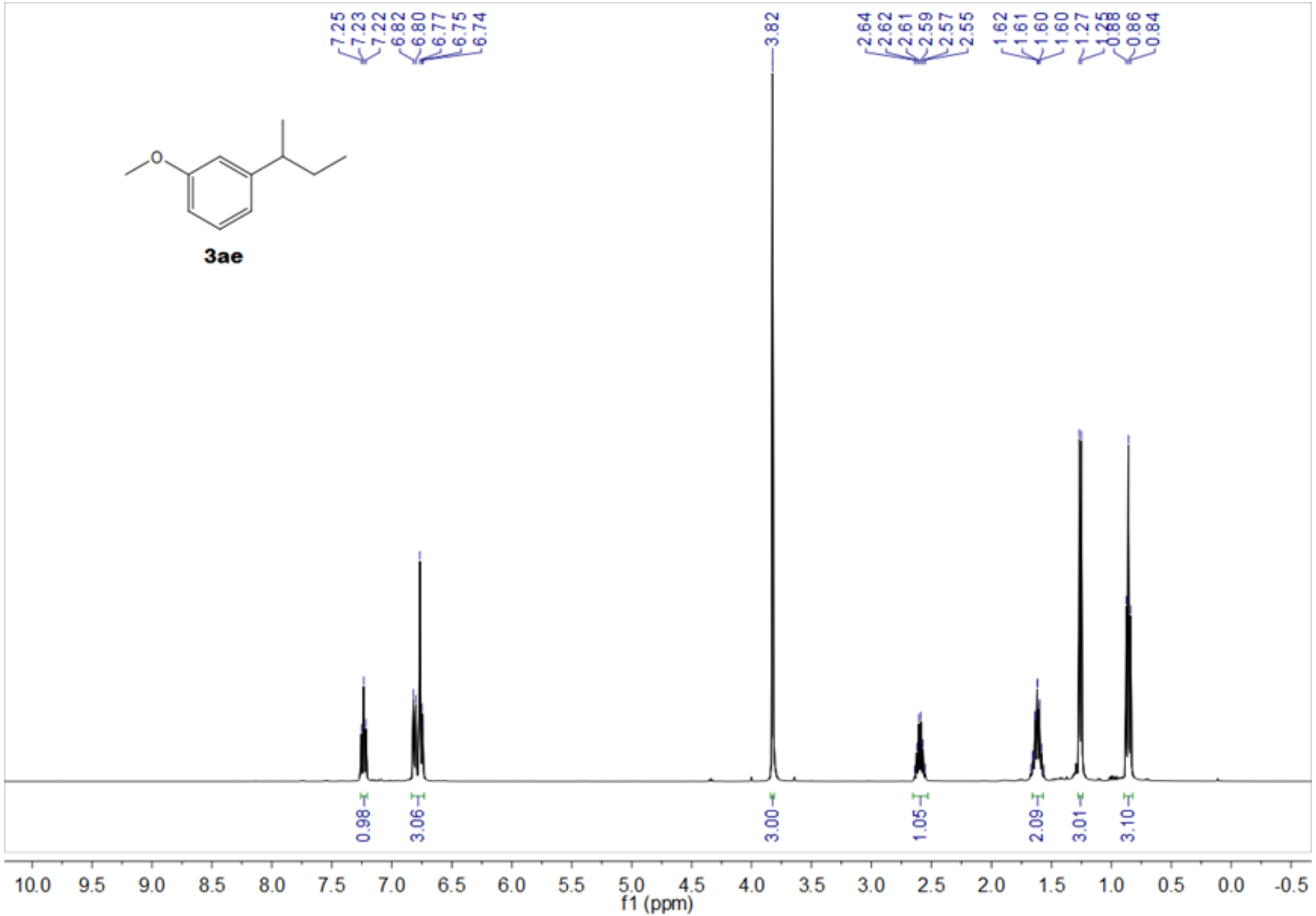

159.70
144.77
129.27
120.99
114.32
110.92
55.23
36.18
31.88
31.51
29.16
22.75
14.24
O
3ab
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

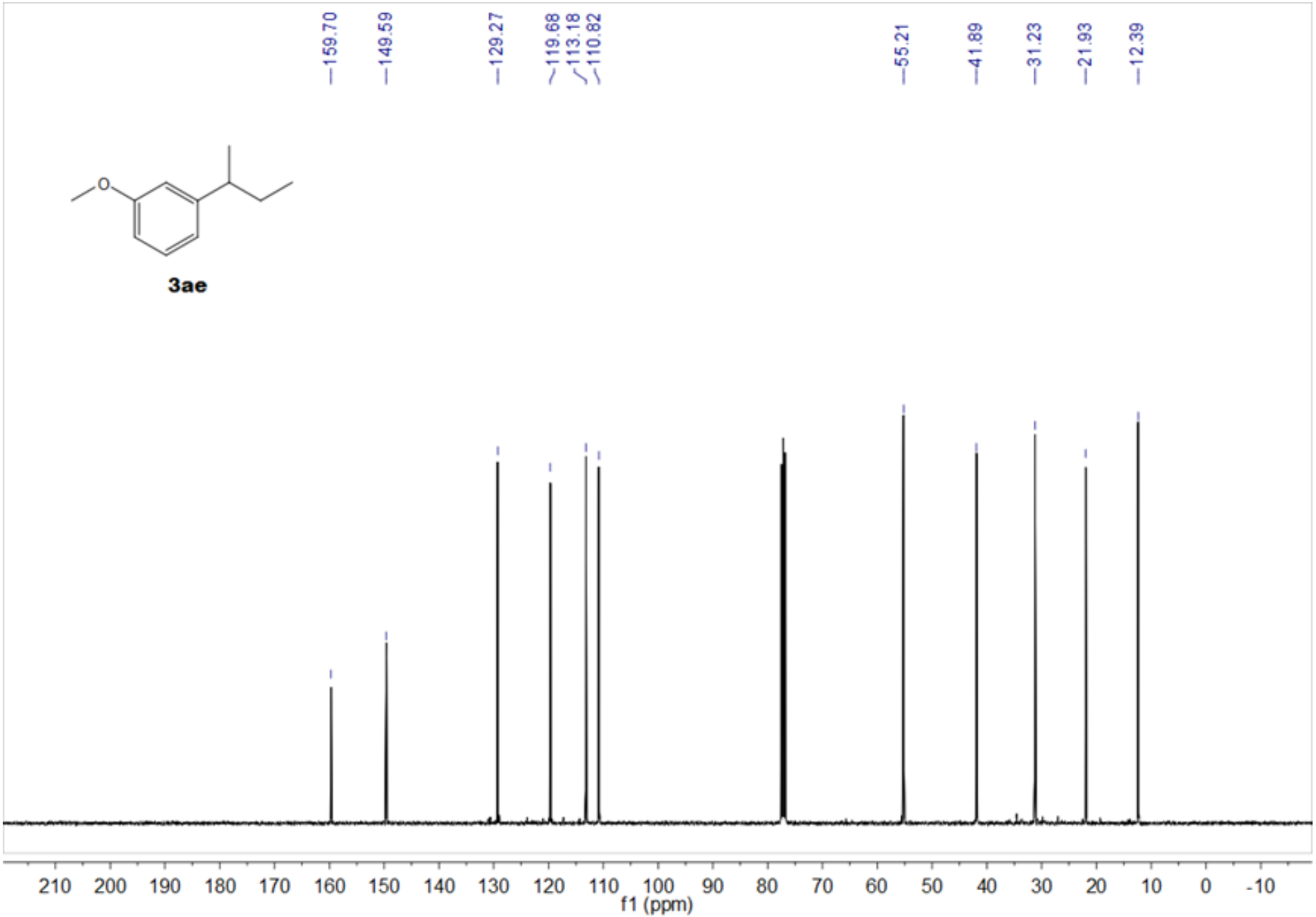

7.39
7.38
7.36
7.35
7.34
7.33
7.31
7.30
7.29
7.24
7.22
7.20
6.82
6.80
6.78
6.76
4.54
3.81
3.54
3.53
3.51
2.75
2.73
2.71
2.01
1.99
1.97
1.95
1.94
O
O
3ac
5.04
1.00
2.99
2.01
3.00
2.02
2.04
2.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

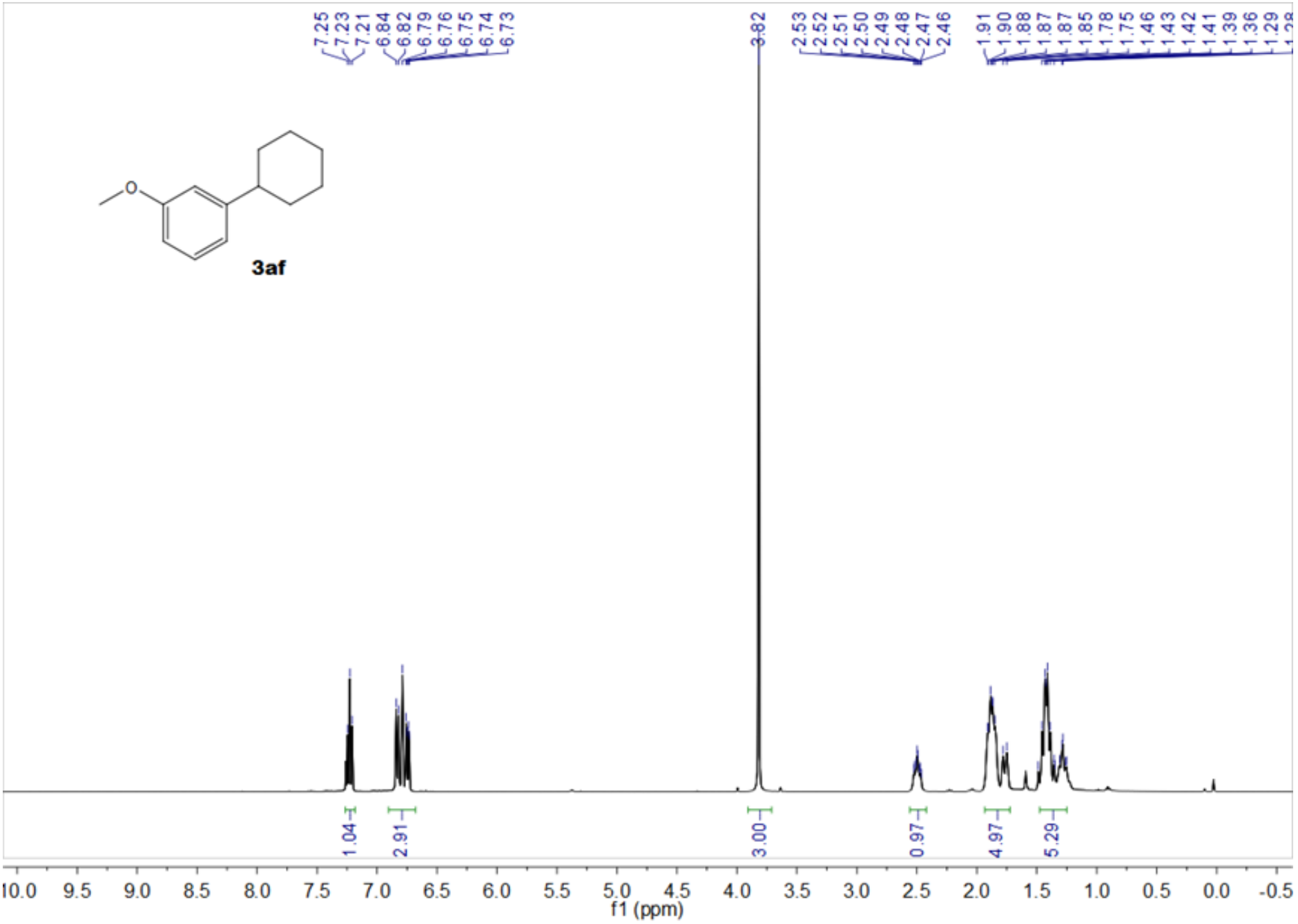

159.72
143.73
138.68
129.34
128.45
127.73
127.61
120.99
114.27
111.19
73.01
69.61
55.16
32.55
31.38
O
O
3ac
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

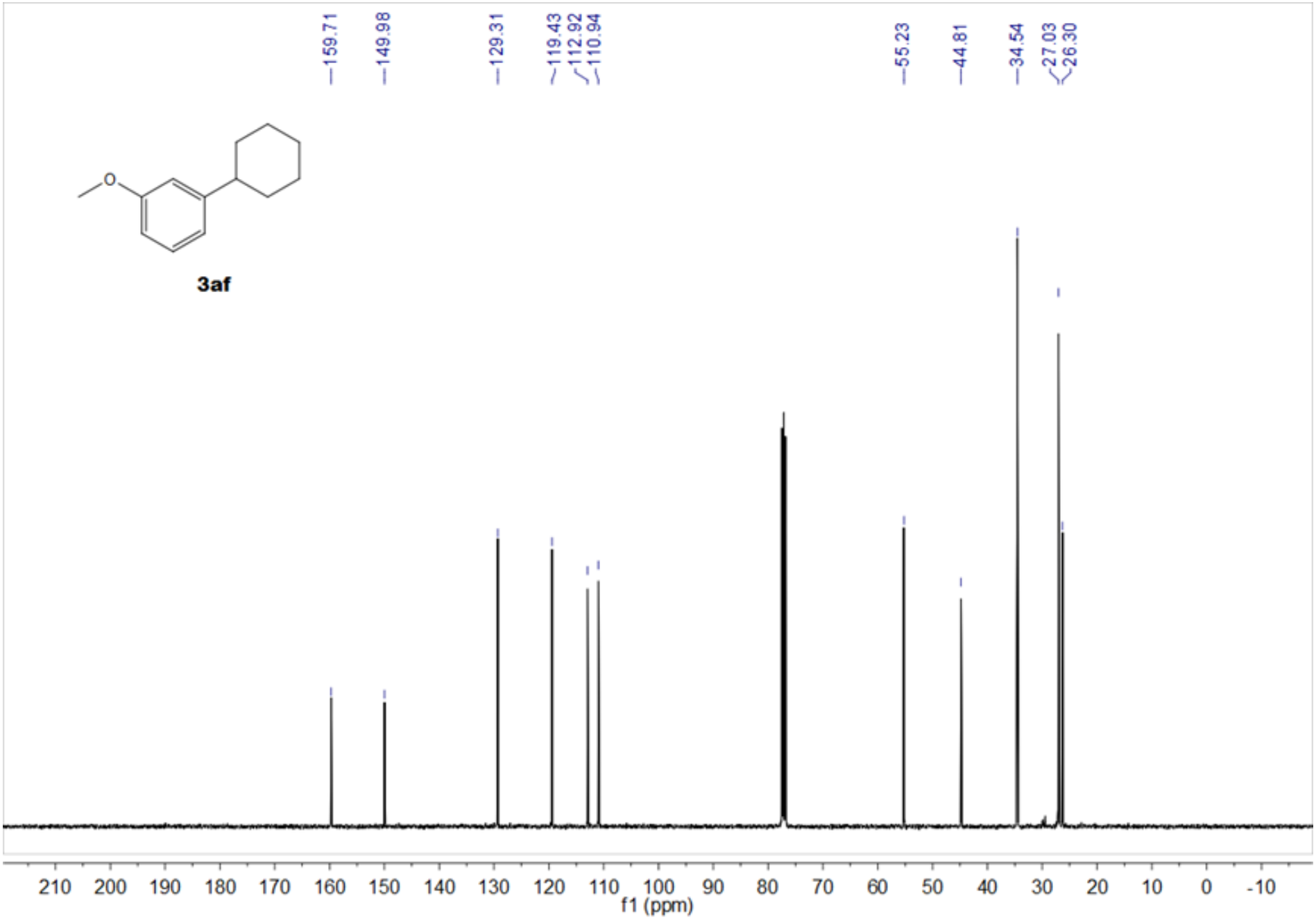

7.22
7.20
7.19
6.81
6.79
6.76
6.73
6.73
3.81
3.66
3.65
3.63
2.69
2.67
2.65
1.88
1.87
1.85
1.85
1.83
1.81
0.92
0.07
O
O
Si
3ad
1.00
3.01
3.00
2.06
2.04
2.07
9.12
6.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


159.74
144.04
129.34
121.03
114.33
111.12
62.47
55.20
34.49
32.29
26.09
18.45
5.14
O
O
Si
3ad
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.25
7.23
7.22
6.82
6.80
6.77
6.75
6.74
3.82
2.64
2.62
2.61
2.59
2.57
2.55
1.62
1.61
1.60
1.60
1.27
1.25
0.88
0.86
0.84
O
3ae
0.98
3.06
3.00
1.05
2.09
3.01
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


159.70
149.59
129.27
119.68
113.18
110.82
55.21
41.89
31.23
21.93
12.39
O
3ae
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.25
7.23
7.21
6.84
6.82
6.79
6.76
6.75
6.74
6.73
3.82
2.53
2.52
2.51
2.50
2.49
2.48
2.47
2.46
1.91
1.90
1.88
1.87
1.87
1.85
1.78
1.75
1.46
1.43
1.42
1.41
1.39
1.36
1.29
O
3af
1.04
2.91
3.00
0.97
4.97
5.29
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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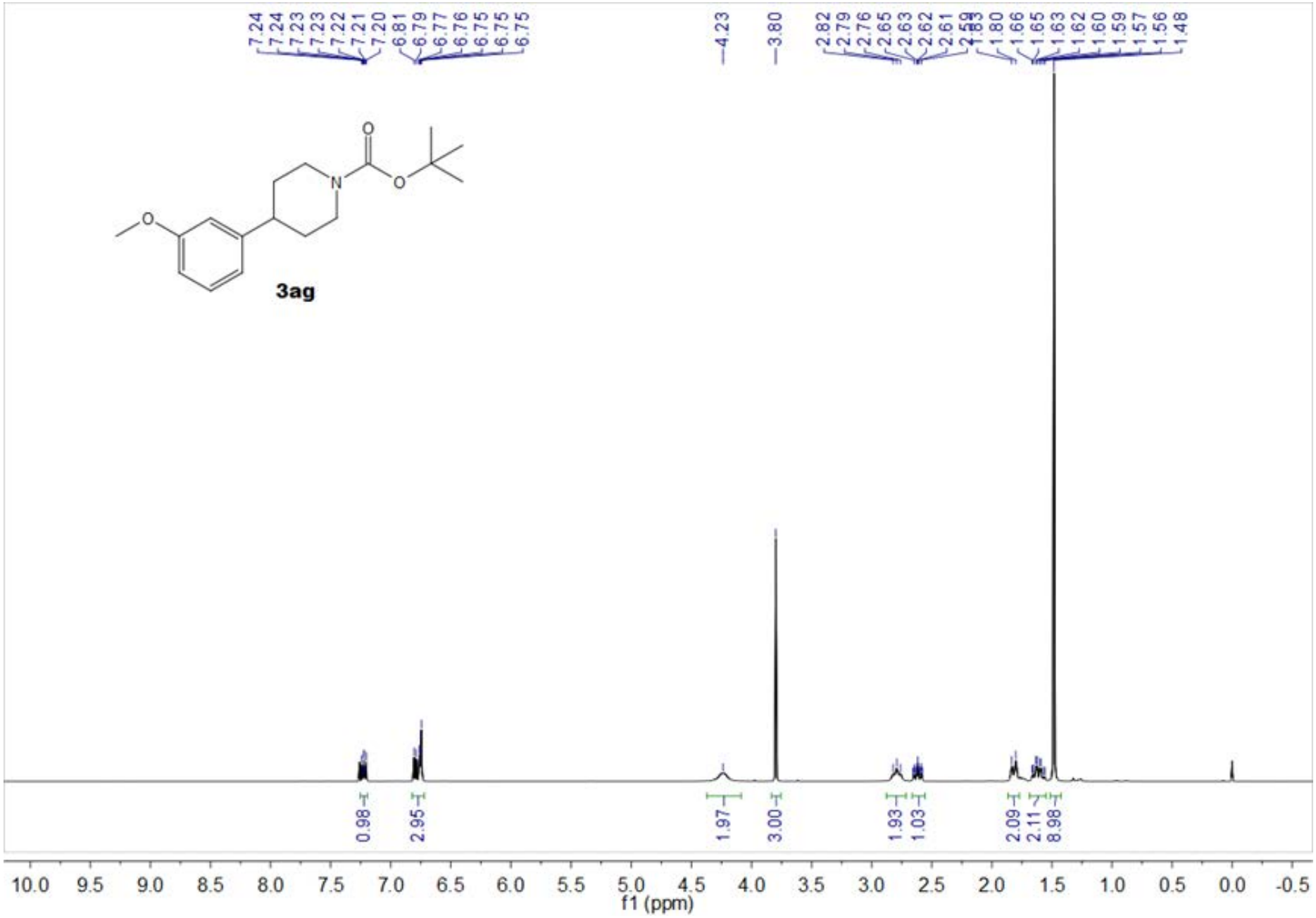
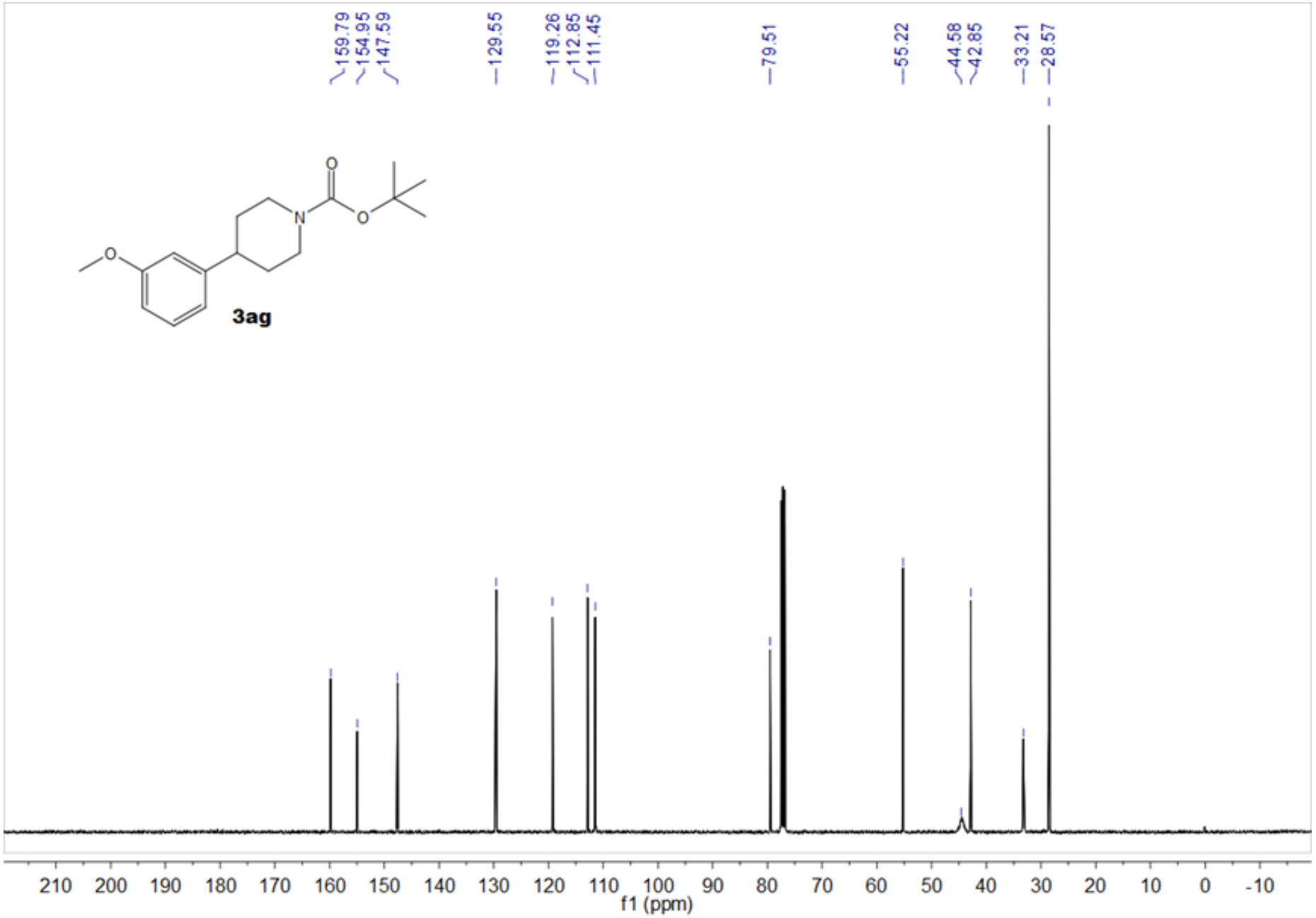
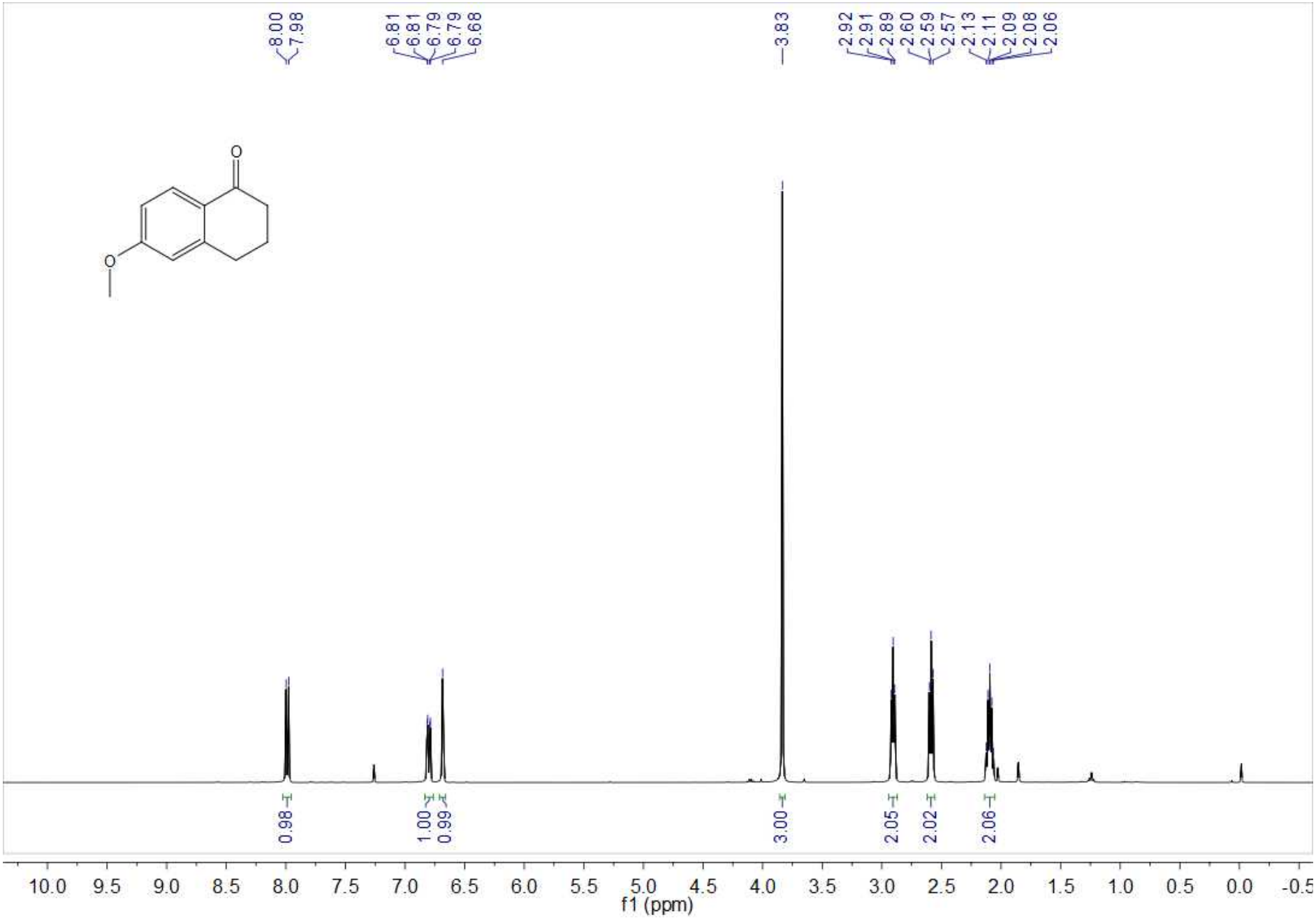
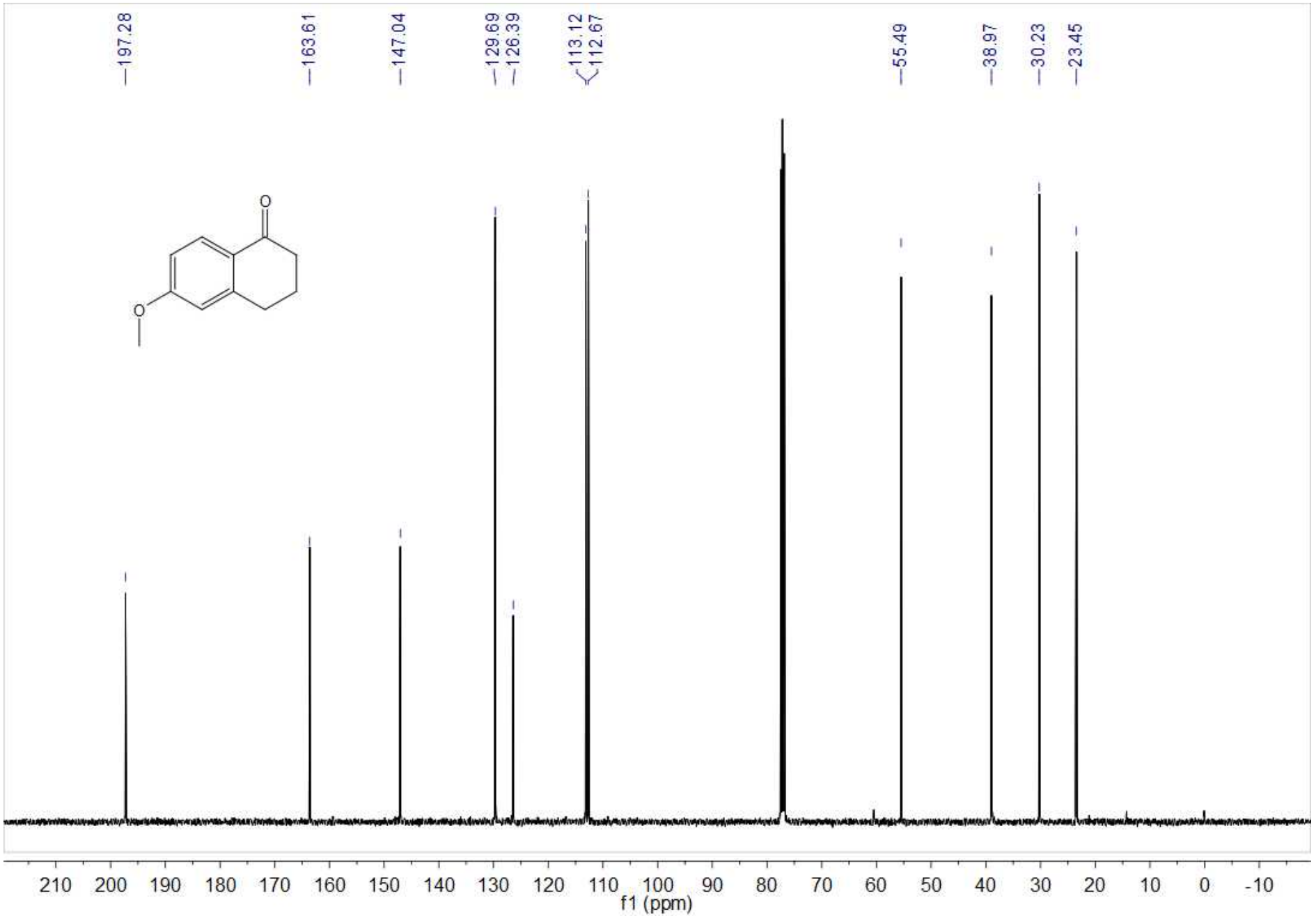
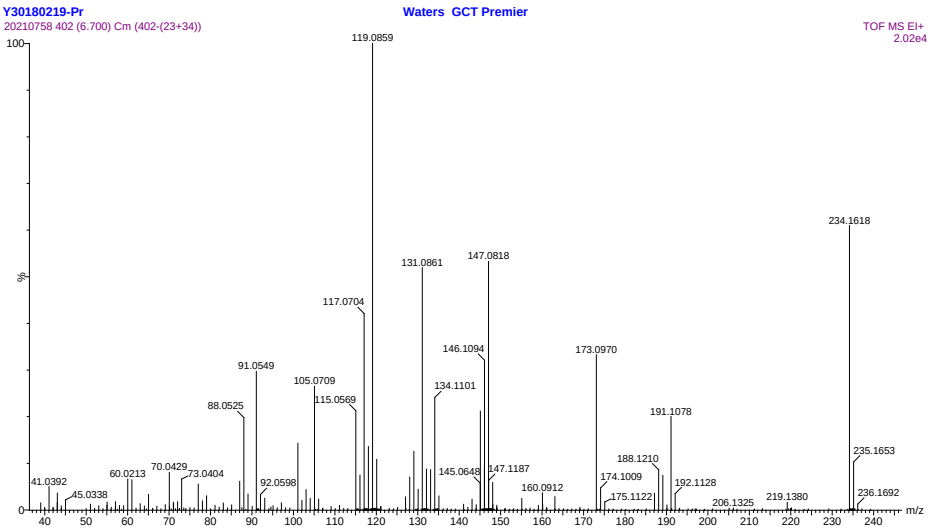
149.98
129.31
119.43
112.92
110.94
55.23
44.81
34.54
27.03
26.30
O
3af
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

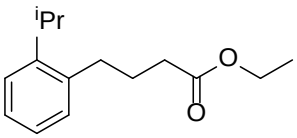

7.24
7.24
7.23
7.23
7.22
7.21
7.20
6.81
6.79
6.77
6.76
6.75
6.75
6.75
4.23
3.80
2.82
2.79
2.76
2.65
2.63
2.62
2.61
1.80
1.66
1.65
1.63
1.62
1.60
1.59
1.57
1.56
1.48
O
N
O
O
3ag
0.98
2.95
1.97
3.00
1.93
1.03
2.09
2.11
8.98
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


159.79
154.95
147.59
129.55
119.26
112.85
111.45
79.51
55.22
44.58
42.85
33.21
28.57
O
N
O
O
3ag
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.00
7.98
6.81
6.81
6.79
6.79
6.68
3.83
2.92
2.91
2.89
2.60
2.59
2.57
2.13
2.11
2.09
2.08
2.06
O
O
0.98
1.00
0.99
3.00
2.05
2.02
2.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


197.28
163.61
147.04
129.69
126.39
113.12
112.67
55.49
38.97
30.23
23.45
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)





Ethyl 4-(2-isopropylphenyl)butanoate (**3ea**)

Elemental Composition Report

Multiple Mass Analysis: 46 mass(es) processed

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions

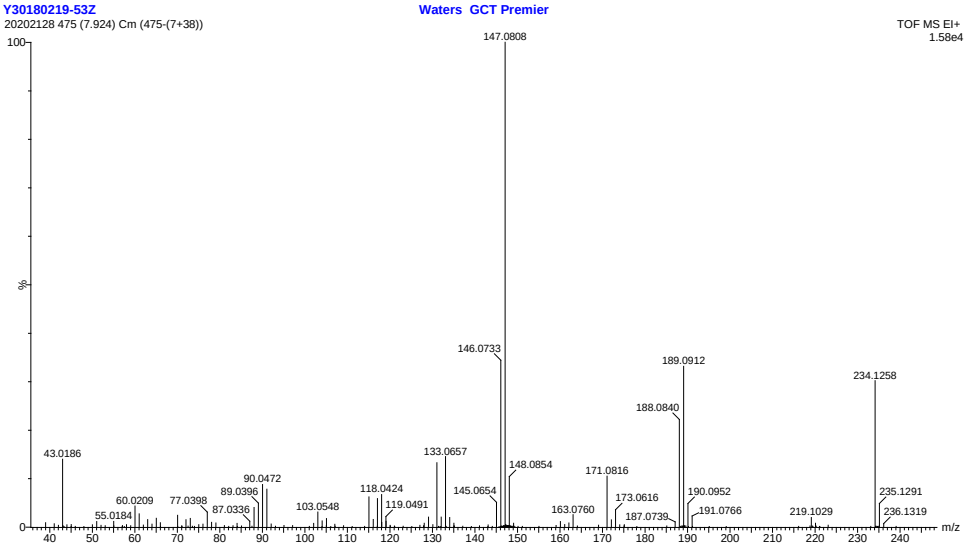
248 formula(e) evaluated with 43 results within limits (up to 50 closest results for each mass)

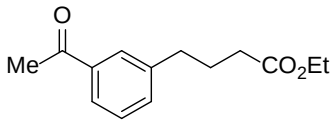
Elements Used:

C: 0-15 H: 0-22 O: 0-2

Minimum:	3.00					-1.5
Maximum:	100.00		5.0	10.0		100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
234.1618	60.95	234.1620	-0.2	-0.9	5.0	1.7	C15 H22 O2





Ethyl 4-(3-acetylphenyl)butanoate (**3ka**)

Elemental Composition Report

Multiple Mass Analysis: 24 mass(es) processed

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

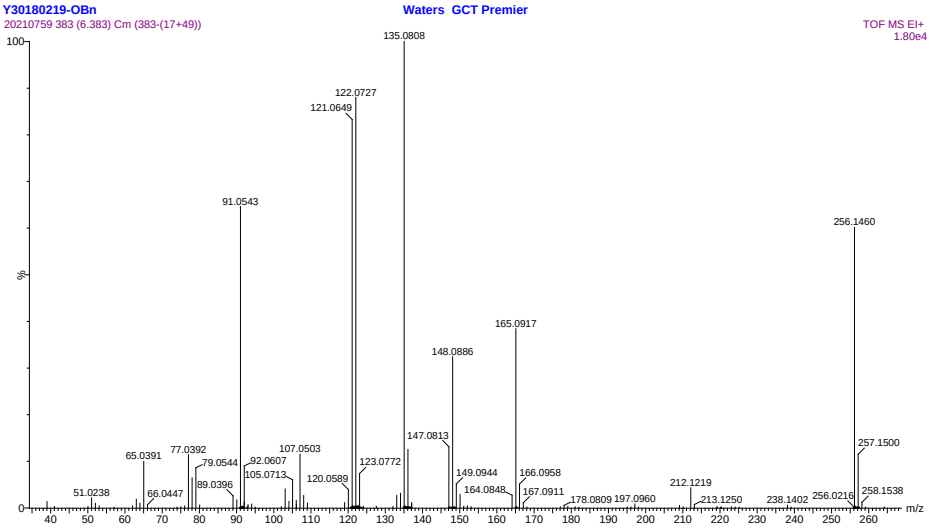
Monoisotopic Mass, Odd and Even Electron Ions

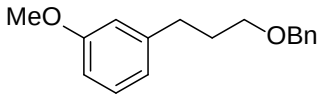
141 formula(e) evaluated with 23 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-14 H: 0 -18 O: 0-3

Minimum:	3.00				-1.5	
Maximum:	100.00		5.0	10.0	100.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	
i-FIT	Formula					
234.1258	30.18	234.1256	0.2	0.9	6.0	2.5
	C14 H18 O3					





1-(3-(Benzyloxy)propyl)-3-methoxybenzene (**3ac**)

Elemental Composition Repor

Multiple Mass Analysis: 24 mass(es) processed

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions

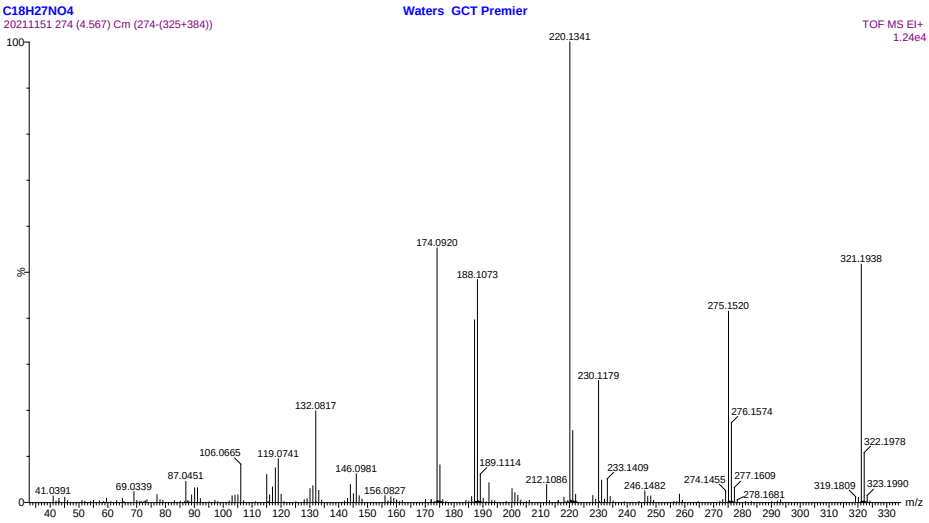
125 formula(e) evaluated with 23 results within limits (up to 50 closest results for each mass)

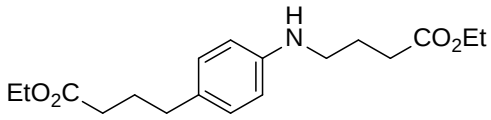
Elements Used:

C: 0-17 H: 0-20 O: 0-2

Minimum:	3.00					-1.5
Maximum:	100.00		5.0	10.0		100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
256.1460	60.11	256.1463	-0.3	-1.2	8.0	1.7	C17 H20 O2





Ethyl 4-(4-((4-ethoxy-4-oxobutyl)amino)phenyl)butanoate

Elemental Composition Report

Multiple Mass Analysis: 29 mass(es) processed

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions

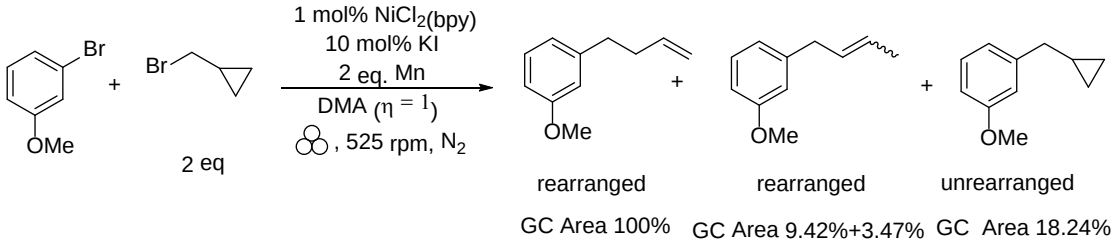
562 formula(e) evaluated with 38 results within limits (up to 50 closest results for each mass)

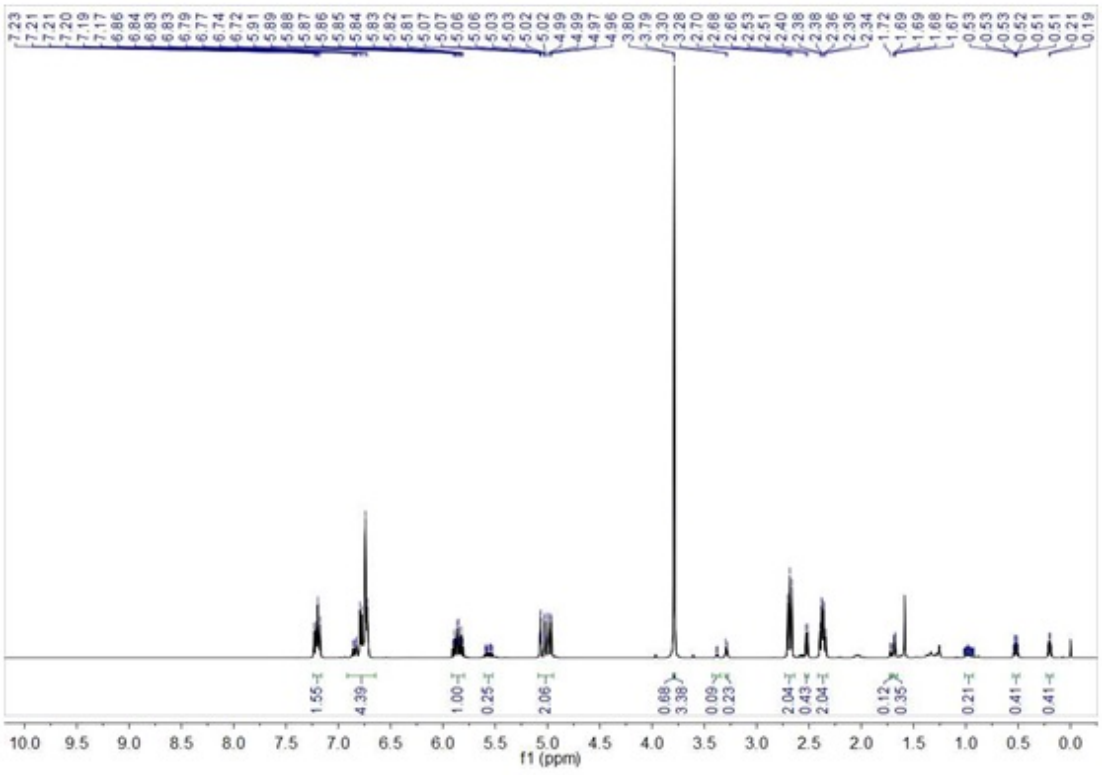
Elements Used:

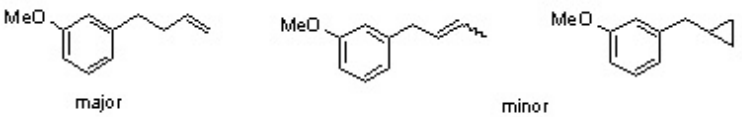
C: 0-18 H: 0-27 N: 0-1 O: 0-4

Minimum:	3.00				-1.5	
Maximum:	100.00		5.0	10.0	100.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT
Formula						
321.1938	51.68	321.1940	-0.2	-0.6	6.0	0.1
C18 H27 N O4						

[1]HNMR of the product mixture from radical clock experiment







major

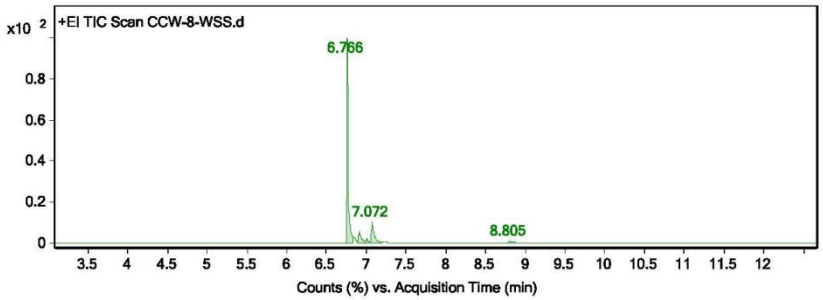
minor

GC-MS of the product mixture from radical clock experiment

Qualitative Analysis Report

Chromatograms

Fragmentor Voltage **Collision Energy** 0 **Ionization Mode** EI

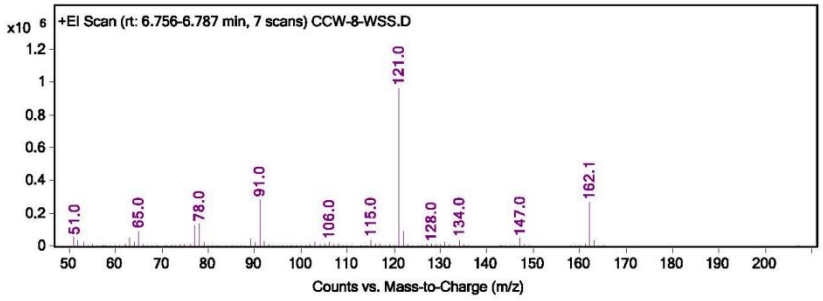


Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6.74	6.766	6.833	5297133.94	6788786.33	100
2	6.89	6.911	6.984	307211.2	639583.56	9.42
3	6.984	7.005	7.046	116894.29	235684.49	3.47
4	7.046	7.072	7.202	529376.29	1238565.4	18.24
5	8.776	8.805	8.982	57738.77	197061.58	2.9

Spectra

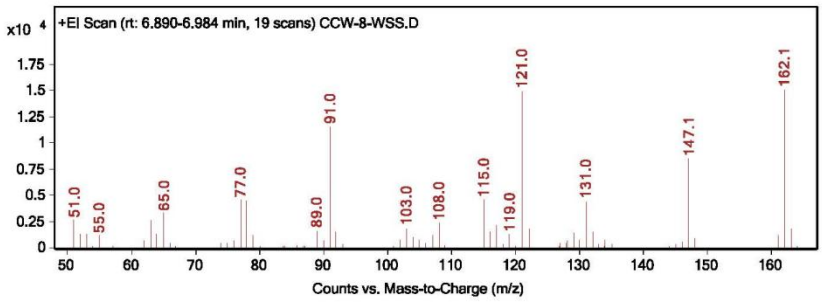
Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"		0	EI



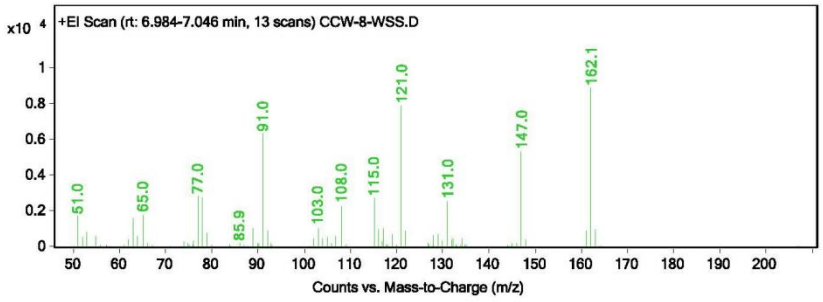
Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode

Qualitative Analysis Report

Peak (2) in "+ TIC Scan"	0	EI

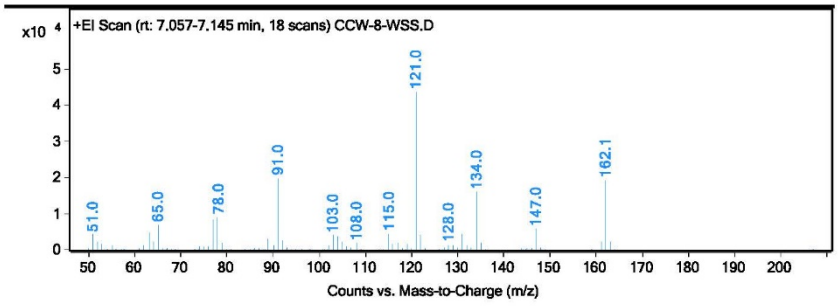


Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (3) in "+ TIC Scan"		0	EI

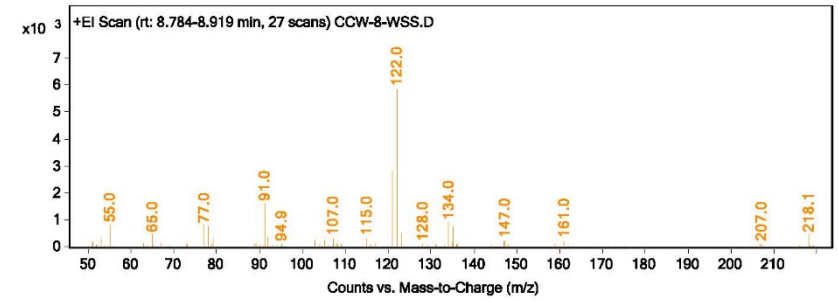


Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (4) in "+ TIC Scan"		0	EI

Qualitative Analysis Report



Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (5) in "+ TIC Scan"		0	EI



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