Supplementary Material (ESI) for Chemical Communications

Direct synthesis of ester-containing indium homoenolate and its application in palladium-catalyzed cross-coupling with aryl halide

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Supporting Information

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General methods

All β-halo esters and aryl halides were commercially available and used without further purification. Analytical grade THF and DMA were used in all the reactions without purification such as dehydration or re-distillation. All indium, copper iodide, palladium catalysts and lithium chloride were purchased from chemical companies and used directly without further purifications.

Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible by staining with acidic solution of ceric molybdate.

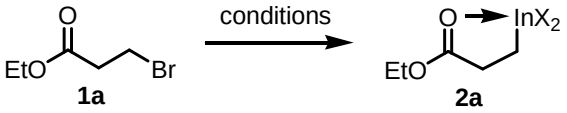
Flash chromatography was performed using Merck silica gel 60 with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use.

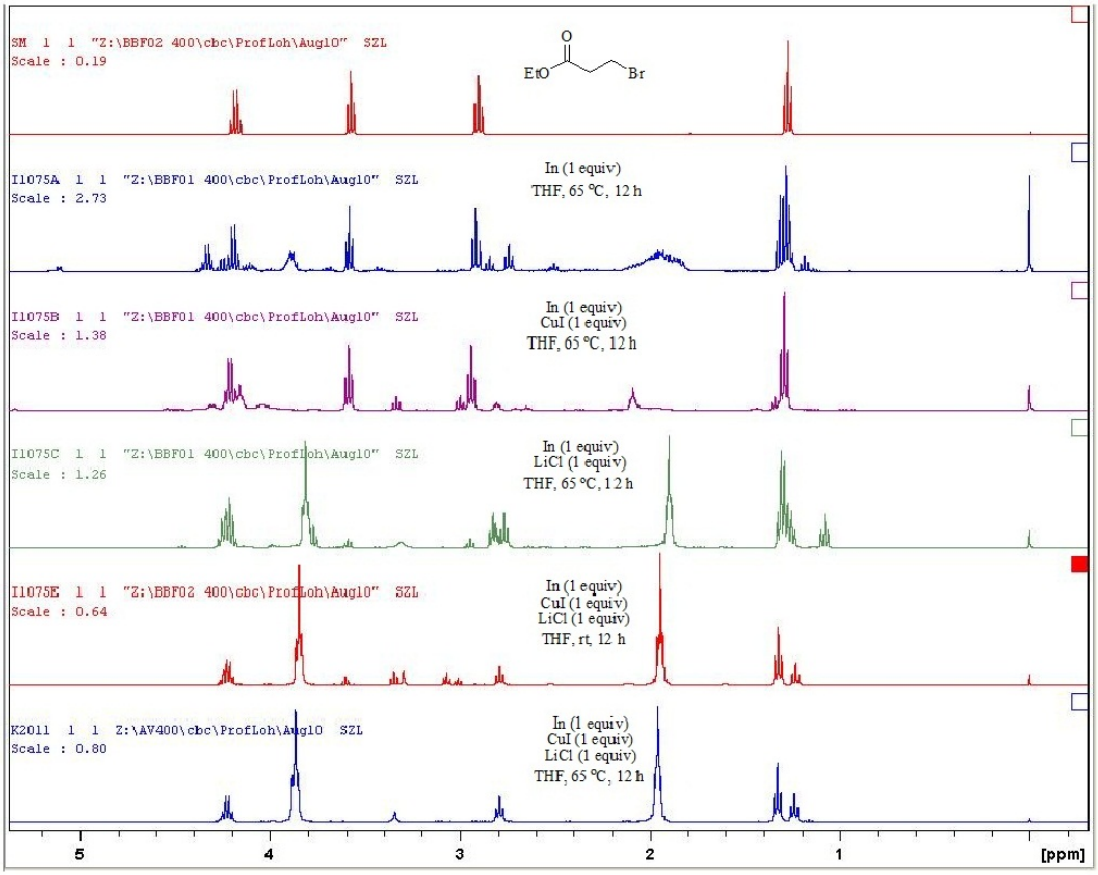
Infrared spectra were recorded on a Bio-Rad FTS 165 FTIR spectrometer. The oil samples were examined under neat conditions.

High Resolution Mass (HRMS) spectra were obtained using Finnigan MAT95XP GC/HRMS (Thermo Electron Corporation).

Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded on a Bruker Avance DPX 300 and Bruker AMX 400 and 500 spectrophotometer ($CDCl_3$ as solvent). Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 7.2600, singlet). Multiplicities were given as: s (singlet); d (doublet); t (triplet); q (quartet); or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 77.03, triplet).

^{1}H NMR comparison of the reaction products performed under different conditions

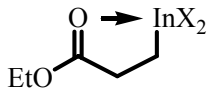




Experimental procedure

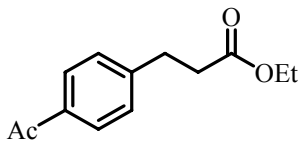
General procedure for the synthesis of ester-containing indium homoenolate from β-halo ester and palladium-catalyzed cross-coupling with aryl halide (Tables 3-4): To an 8 mL sample vial was added β-halo ester (1 mmol), indium (1 mmol), CuI (1 mmol), LiCl (1 mmol), and analytical grade THF (3 mL) sequentially. The reaction was stirred vigorously at 65 ^{o}C for 12 hrs. After reaction, it was kept still for around 10 minutes. Then the upper clear solution was carefully separated from the bottom black precipitate by syringe. The residual black precipitate was washed with 3 mL THF and the THF layer was carefully separated by syringe. The combined organic layers were concentrated under vacuo. Then the residue was dissolved in 3 mL DMA and transferred to another 8 mL sample vial. Aryl halide (0.7 mmol), LiCl (1 mmol), and $Pd(PPh_3)_4$ (0.05 mmol, 0.05 equiv) was added to the sample vial sequentially. The reaction mixture was stirred at 90 ^{o}C for 24 hrs. After reaction, it was directly purified by silica gel column chromatography using EtOAc/hexane as eluant to afford the desired product.

Spectroscopic data of products



Indium homoenolate 2a

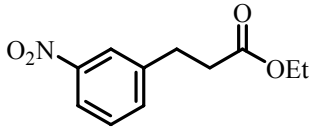
^{1}H NMR (500 MHz, $CDCl_3$): δ 1.24 (t, *J* = 7.18 Hz, 2H), 1.33 (t, *J* = 7.14 Hz, 3H), 2.80 (t, *J* = 7.18 Hz, 2H), 4.23 (q, *J* = 7.14 Hz, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 14.3, 31.6, 62.8, 180.4 ppm.



Ethyl 3-(4-acetylphenyl)propanoate[1,2]

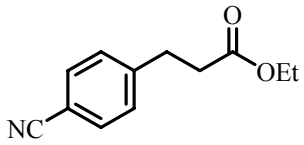
^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.23 (t, *J* = 7.20 Hz, 3H), 2.58 (s, 3H), 2.65 (t, *J* = 7.64 Hz, 2H), 3.01 (t, *J* = 7.64 Hz, 2H), 4.12 (q, *J* = 7.20 Hz, 2H), 7.30 (d, *J* = 8.08 Hz, 2H), 7.89 (d, *J*

= 8.08 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.1, 26.5, 30.8, 35.2, 60.5, 128.5, 128.5, 135.3, 146.2, 172.4, 197.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{13}H_{17}O_3$: 221.1178, found: 221.1181. FTIR (KBr, neat): *v* 1732, 1682 cm^{-1}.



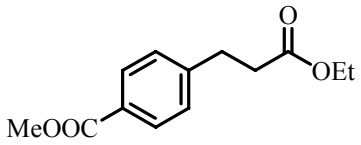
Ethyl 3-(3-nitrophenyl)propanoate[3,4]

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.24 (t, *J* = 7.12 Hz, 3H), 2.68 (t, *J* = 7.53 Hz, 2H), 3.07 (t, *J* = 7.52 Hz, 2H), 4.14 (q, *J* = 7.13 Hz, 2H), 7.44-7.58 (m, 2H), 8.06-8.09 (m, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.1, 30.4, 35.2, 60.7, 121.5, 123.2, 129.3, 134.7, 142.5, 148.3, 172.1 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{11}H_{14}NO_4$: 224.0923, found: 224.0920. FTIR (KBr, neat): *v* 1732 cm^{-1}.



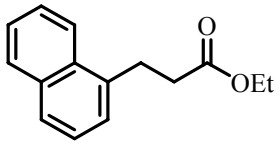
Ethyl 3-(4-cyanophenyl)propanoate[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (t, *J* = 7.18 Hz, 3H), 2.64 (t, *J* = 7.60 Hz, 2H), 3.01 (t, *J* = 7.60 Hz, 2H), 4.12 (q, *J* = 7.18 Hz, 2H), 7.32 (d, *J* = 8.17 Hz, 2H), 7.58 (d, *J* = 8.20 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.2, 30.9, 35.1, 60.7, 110.2, 118.9, 129.2, 132.3, 146.2, 172.2 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{12}H_{14}NO_2$: 204.1025, found: 204.1031. FTIR (KBr, neat): *v* 2230, 1732 cm^{-1}.



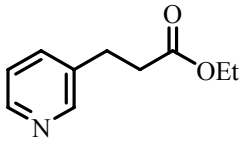
Methyl 4-(3-ethoxy-3-oxopropyl)benzoate[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.22 (t, *J* = 7.18 Hz, 3H), 2.63 (t, *J* = 7.94 Hz, 2H), 3.00 (t, *J* = 7.64 Hz, 2H), 3.89 (s, 3H), 4.12 (q, *J* = 7.18 Hz, 2H), 7.27 (d, *J* = 8.36 Hz, 2H), 7.96 (d, *J* = 8.36 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 30.7, 35.2, 51.8, 60.4, 128.1, 128.2, 129.7, 145.9, 166.8, 172.3 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{13}H_{17}O_4$: 237.1127, found: 237.1119. FTIR (KBr, neat): *v* 1720 cm^{-1}.



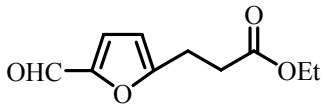
Ethyl 3-(naphthalen-1-yl)propanoate[5]

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (t, *J* = 7.12 Hz, 3H), 2.74 (t, *J* = 8.14 Hz, 2H), 3.41 (t, *J* = 7.72 Hz, 2H), 4.14 (q, *J* = 7.12 Hz, 2H), 7.33-7.40 (m, 2H), 7.45-7.53 (m, 2H), 7.71 (d, *J* = 7.89 Hz, 1H), 7.84 (d, *J* = 7.73 Hz, 1H), 8.02 (d, *J* = 8.24 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.2, 28.1, 35.2, 60.5, 123.4, 125.5, 125.6, 125.9, 126.0, 127.1, 128.9, 131.6, 133.9, 136.6, 173.0 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{15}H_{17}O_2$: 229.1229, found: 229.1238. FTIR (KBr, neat): *v* 1732 cm^{-1}.



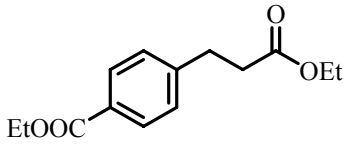
Ethyl 3-(pyridin-3-yl)propanoate[1]

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.23 (t, *J* = 7.14 Hz, 3H), 2.63 (t, *J* = 7.71 Hz, 2H), 2.95 (t, *J* = 7.53 Hz, 2H), 4.12 (q, *J* = 7.14 Hz, 2H), 7.21 (dd, *J* = 4.83, 7.75 Hz, 1H), 7.53 (d, *J* = 7.84 Hz, 1H), 8.45-8.48 (m, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.1, 28.1, 35.4, 60.6, 123.3, 135.8, 135.8, 147.8, 149.8, 172.3 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{10}H_{14}NO_2$: 180.1025, found: 180.1030. FTIR (KBr, neat): *v* 1732 cm^{-1}.



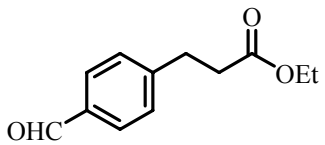
Ethyl 3-(5-formylfuran-2-yl)propanoate

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.25 (t, *J* = 7.16 Hz, 3H), 2.73 (t, *J* = 7.52 Hz, 2H), 3.07 (t, *J* = 7.36 Hz, 2H), 4.15 (q, *J* = 7.16 Hz, 2H), 6.31 (d, *J* = 3.52 Hz, 1H), 7.18 (d, *J* = 3.52 Hz, 1H), 9.53 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 23.7, 31.8, 60.7, 109.1, 123.2, 151.9, 161.4, 171.7, 177.0 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{10}H_{13}O_4$: 197.0814, found: 197.0810. FTIR (KBr, neat): *v* 1732, 1670 cm^{-1}.



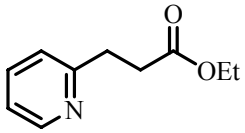
Ethyl 4-(3-ethoxy-3-oxopropyl)benzoate[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.22 (t, *J* = 7.18 Hz, 3H), 1.38 (t, *J* = 7.16 Hz, 3H), 2.63 (t, *J* = 7.85 Hz, 2H), 3.00 (t, *J* = 7.63 Hz, 2H), 4.12 (q, *J* = 7.18 Hz, 2H), 4.36 (q, *J* = 7.16 Hz, 2H), 7.27 (d, *J* = 8.24 Hz, 2H), 7.97 (d, *J* = 8.24 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 14.2, 30.8, 35.3, 60.4, 60.7, 128.2, 128.6, 129.7, 145.8, 166.4, 172.4 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{14}H_{19}O_4$: 251.1283, found: 251.1280. FTIR (KBr, neat): *v* 1720 cm^{-1}.



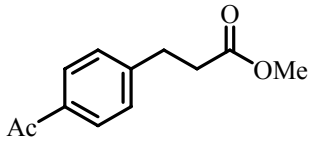
Ethyl 3-(4-formylphenyl)propanoate[6]

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (t, *J* = 7.18 Hz, 3H), 2.66 (t, *J* = 7.77 Hz, 2H), 3.03 (t, *J* = 7.60 Hz, 2H), 4.13 (q, *J* = 7.18 Hz, 2H), 7.38 (d, *J* = 8.08 Hz, 2H), 7.81 (d, *J* = 8.08 Hz, 2H), 9.97 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 30.9, 35.1, 60.5, 128.9, 129.9, 134.8, 147.8, 172.2, 191.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{12}H_{15}O_3$: 207.1021, found: 207.1023. FTIR (KBr, neat): *v* 1732, 1701 cm^{-1}.



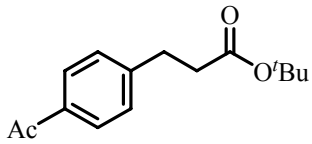
Ethyl 3-(pyridin-2-yl)propanoate[7]

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (t, *J* = 7.12 Hz, 3H), 2.80 (t, *J* = 7.53 Hz, 2H), 3.12 (t, *J* = 7.47 Hz, 2H), 4.13 (q, *J* = 7.12 Hz, 2H), 7.11-7.14 (m, 1H), 7.19 (d, *J* = 7.79 Hz, 1H), 7.59 (td, *J* = 1.85, 7.70 Hz, 1H), 8.53 (d, *J* = 4.36 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.2, 32.9, 33.5, 60.4, 121.3, 123.0, 136.4, 149.3, 160.1, 173.1 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{10}H_{14}NO_2$: 180.1025, found: 180.1021. FTIR (KBr, neat): *v* 1728 cm^{-1}.



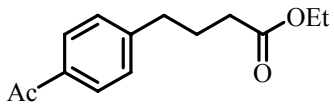
Methyl 3-(4-acetylphenyl)propanoate[8]

^{1}H NMR (300 MHz, $CDCl_3$): δ 2.58 (s, 3H), 2.66 (t, *J* = 7.83 Hz, 2H), 3.01 (t, *J* = 7.60 Hz, 2H), 3.67 (s, 3H), 7.30 (d, *J* = 8.30 Hz, 2H), 7.89 (d, *J* = 8.30 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.5, 30.8, 35.1, 51.7, 128.5, 128.6, 135.4, 146.2, 172.9, 197.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{12}H_{15}O_3$: 207.1021, found: 207.1030. FTIR (KBr, neat): *v* 1736, 1682 cm^{-1}.



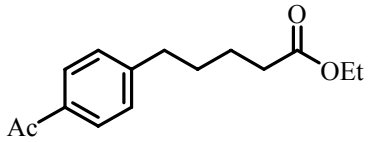
***Tert*-Butyl 3-(4-acetylphenyl)propanoate**

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.41 (s, 9H), 2.54-2.59 (m, 5H), 2.97 (t, *J* = 7.57 Hz, 2H), 7.30 (d, *J* = 8.30 Hz, 2H), 7.88 (d, *J* = 8.30 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 26.5, 28.0, 31.0, 36.4, 80.6, 128.5 (CHx4), 135.3, 146.5, 171.8, 197.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{15}H_{21}O_3$: 249.1491, found: 249.1495. FTIR (KBr, neat): *v* 1732, 1682 cm^{-1}.



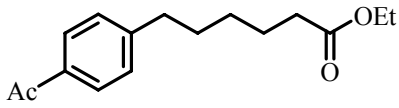
Ethyl 4-(4-acetylphenyl)butanoate[2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.26 (t, *J* = 7.13 Hz, 3H), 1.92-2.02 (m, 2H), 2.32 (t, *J* = 7.40 Hz, 2H), 2.58 (s, 3H), 2.72 (t, *J* = 7.41 Hz, 2H), 4.13 (q, *J* = 7.13 Hz, 2H), 7.28 (d, *J* = 8.26 Hz, 2H), 7.89 (d, *J* = 8.26 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.2, 26.1, 26.5, 33.5, 35.1, 60.3, 128.5, 128.7, 135.2, 147.2, 173.2, 197.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{14}H_{19}O_3$: 253.1334, found: 253.1336. FTIR (KBr, neat): *v* 1732, 1682 cm^{-1}.



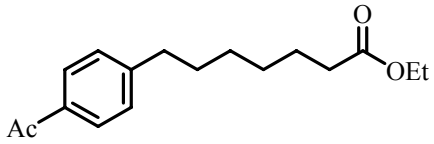
Ethyl 5-(4-acetylphenyl)pentanoate[2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.25 (t, *J* = 7.14 Hz, 3H), 1.65-1.70 (m, 4H), 2.32 (t, *J* = 6.95 Hz, 2H), 2.58 (s, 3H), 2.69 (t, *J* = 7.05 Hz, 2H), 4.12 (q, *J* = 7.14 Hz, 2H), 7.26 (d, *J* = 8.22 Hz, 2H), 7.88 (d, *J* = 8.22 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.2, 24.5, 26.5, 30.4, 34.1, 35.6, 60.2, 128.5, 128.6, 135.1, 148.0, 173.4, 197.8 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{15}H_{21}O_3$: 249.1491, found: 249.1490. FTIR (KBr, neat): *v* 1732, 1682 cm^{-1}.



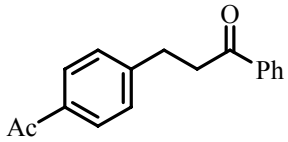
Ethyl 6-(4-acetylphenyl)hexanoate

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.24 (t, *J* = 7.15 Hz, 3H), 1.29-1.42 (m, 2H), 1.61-1.71 (m, 4H), 2.29 (t, *J* = 7.42 Hz, 2H), 2.58 (s, 3H), 2.67 (t, *J* = 7.57 Hz, 2H), 4.12 (q, *J* = 7.15 Hz, 2H), 7.26 (d, *J* = 8.19 Hz, 2H), 7.88 (d, *J* = 8.19 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.2, 24.7, 26.5, 28.7, 30.7, 34.2, 35.7, 60.2, 128.5, 128.6, 135.0, 148.4, 173.7, 197.8 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{16}H_{23}O_3$: 263.1647, found: 263.1656. FTIR (KBr, neat): *v* 1732, 1682 cm^{-1}.



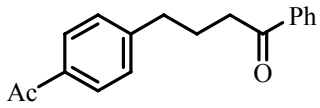
Ethyl 7-(4-acetylphenyl)heptanoate

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.25 (t, *J* = 7.12 Hz, 3H), 1.33-1.37 (m, 4H), 1.60-1.66 (m, 4H), 2.28 (t, *J* = 7.45 Hz, 2H), 2.58 (s, 3H), 2.66 (t, *J* = 7.56 Hz, 2H), 4.12 (q, *J* = 7.12 Hz, 2H), 7.26 (d, *J* = 8.57 Hz, 2H), 7.88 (d, *J* = 8.24 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.2, 24.8, 26.5, 28.8, 28.9, 30.9, 34.3, 35.9, 60.2, 128.5, 128.6, 134.9, 148.6, 173.8, 197.9 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{17}H_{25}O_3$: 277.1804, found: 277.1803. FTIR (KBr, neat): *v* 1732, 1682 cm^{-1}.



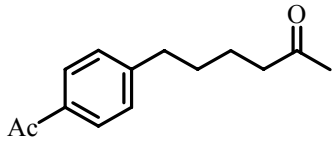
3-(4-Acetylphenyl)-1-phenylpropan-1-one[9]

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.58 (s, 3H), 3.13 (t, *J* = 7.60 Hz, 2H), 3.33 (t, *J* = 7.22 Hz, 2H), 7.35 (d, *J* = 8.21 Hz, 2H), 7.46 (t, *J* = 7.81 Hz, 2H), 7.56 (t, *J* = 7.45 Hz, 1H), 7.90 (d, *J* = 8.21 Hz, 2H), 7.96 (d, *J* = 7.24 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.6, 29.9, 39.7, 128.0, 128.6, 128.7, 133.2, 135.3, 136.6, 147.1, 197.7, 198.6 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{17}H_{17}O_2$: 253.1229, found: 253.1229.FTIR (KBr, neat): *v* 1670, 1676 cm^{-1}.



4-(4-Acetylphenyl)-1-phenylbutan-1-one

^{1}H NMR (400 MHz, $CDCl_3$): δ 2.07-2.15 (m, 2H), 2.59 (s, 3H), 2.78 (t, *J* = 7.41 Hz, 2H), 2.99 (t, *J* = 7.20 Hz, 2H), 7.31 (d, *J* = 8.20 Hz, 2H), 7.45 (t, *J* = 7.89 Hz, 2H), 7.54-7.58 (m, 1H), 7.88-7.93 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 25.2, 26.6, 35.2, 37.5, 128.0, 128.6, 128.7, 133.1, 135.2, 136.9, 147.6, 197.9, 199.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{18}H_{19}O_2$: 267.1385, found: 267.1380. FTIR (KBr, neat): *v* 1678 cm^{-1}.



6-(4-Acetylphenyl)hexan-2-one[10]

^{1}H NMR (400 MHz, $CDCl_3$): δ 1.62-1.64 (m, 4H), 2.13 (s, 3H), 2.46 (t, *J* = 6.76 Hz, 2H), 2.58 (s, 3H), 2.68 (t, *J* = 7.04 Hz, 2H), 7.26 (d, *J* = 8.22 Hz, 2H), 7.88 (d, *J* = 8.22 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.3, 26.6, 29.9, 30.5, 35.7, 43.4, 128.5, 128.6, 135.1, 148.0, 197.8, 208.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{14}H_{19}O_2$: 219.1385, found: 219.1385. FTIR (KBr, neat): *v* 1713, 1678 cm^{-1}.

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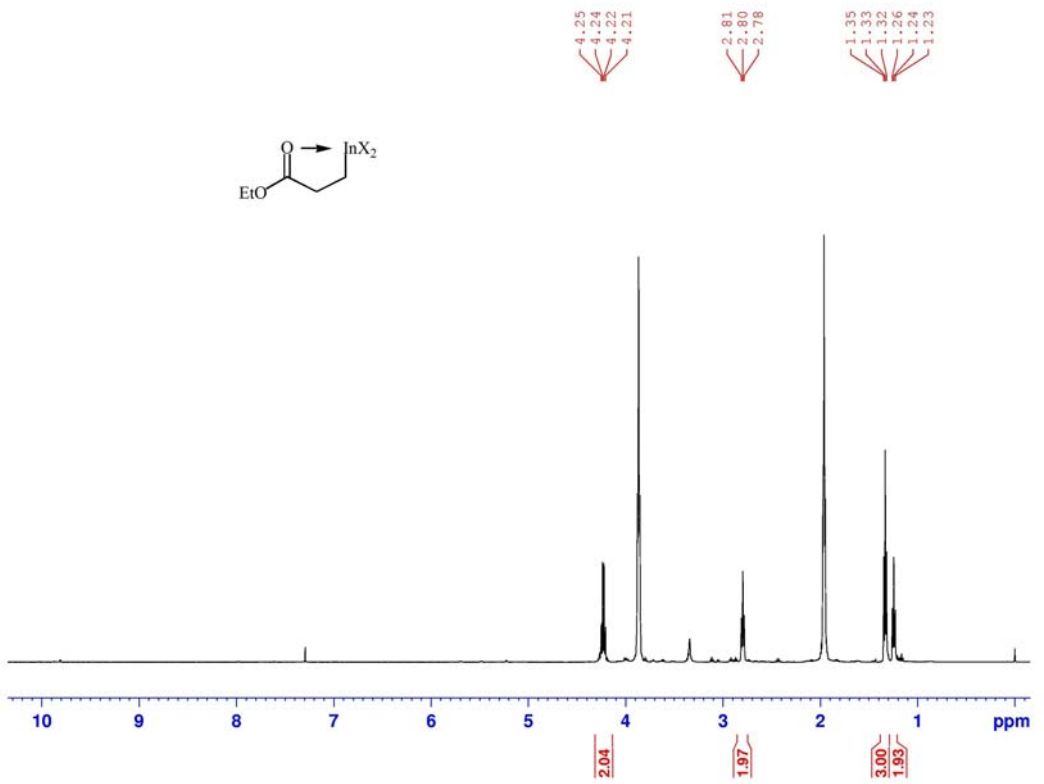
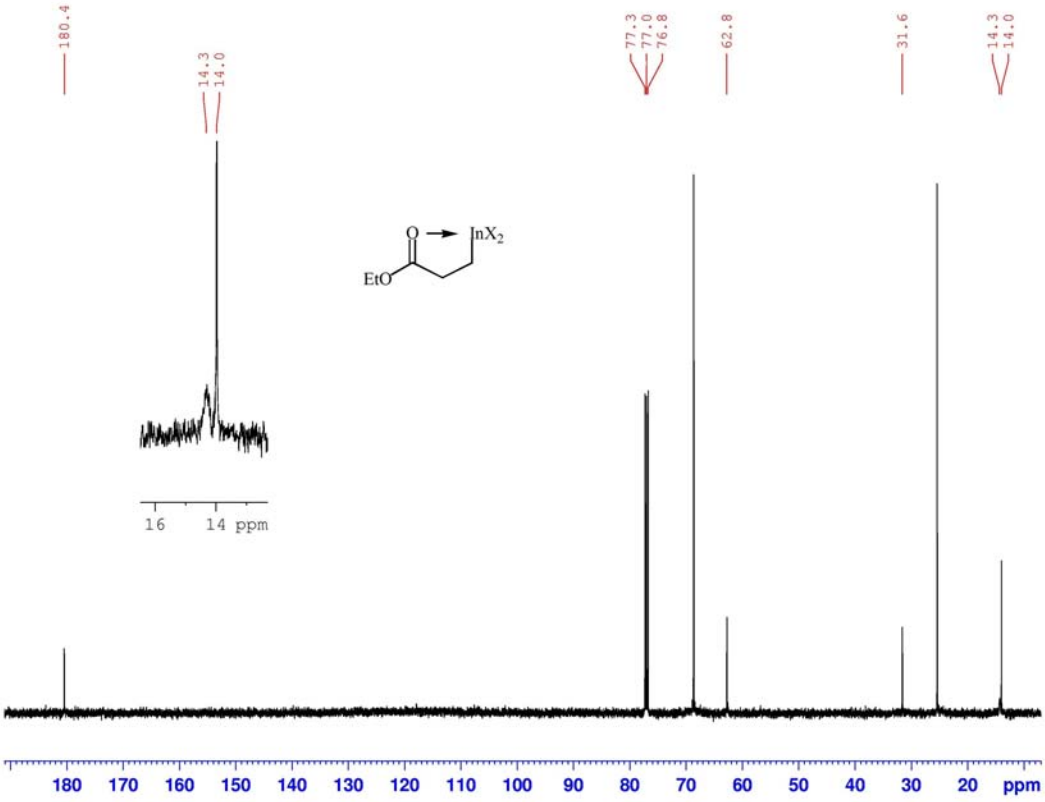
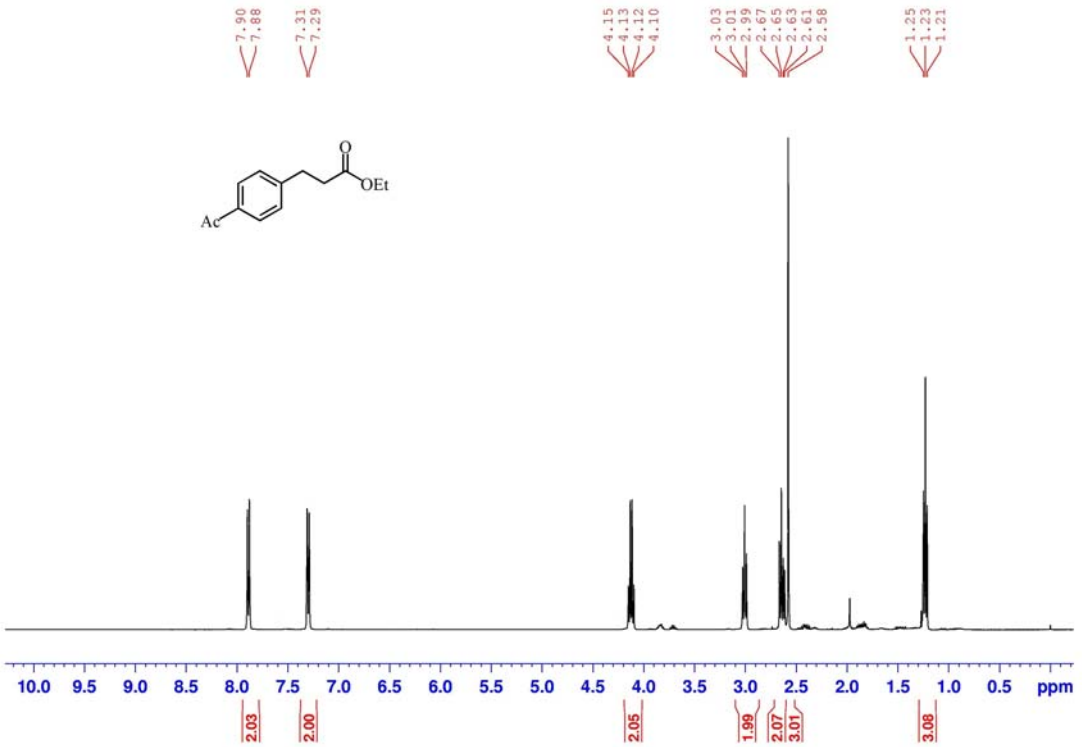
Copies of ^{1}H NMR and ^{13}C NMR spectra of products



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1.21
O
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Ac
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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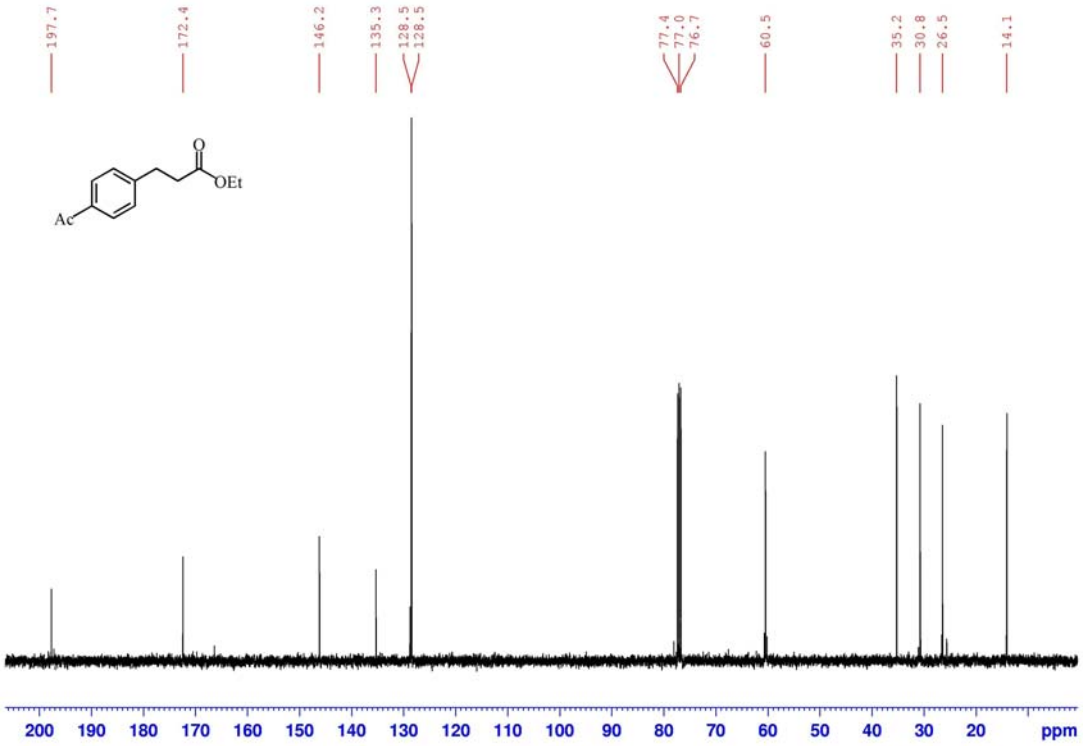
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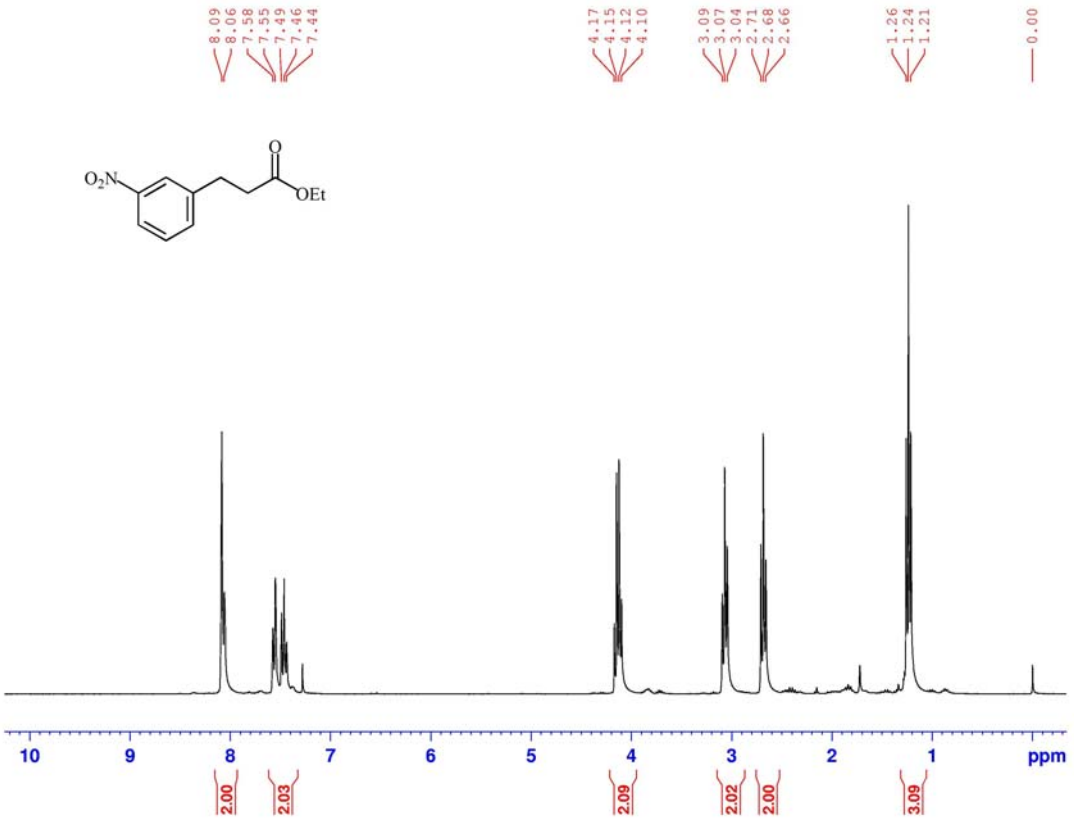
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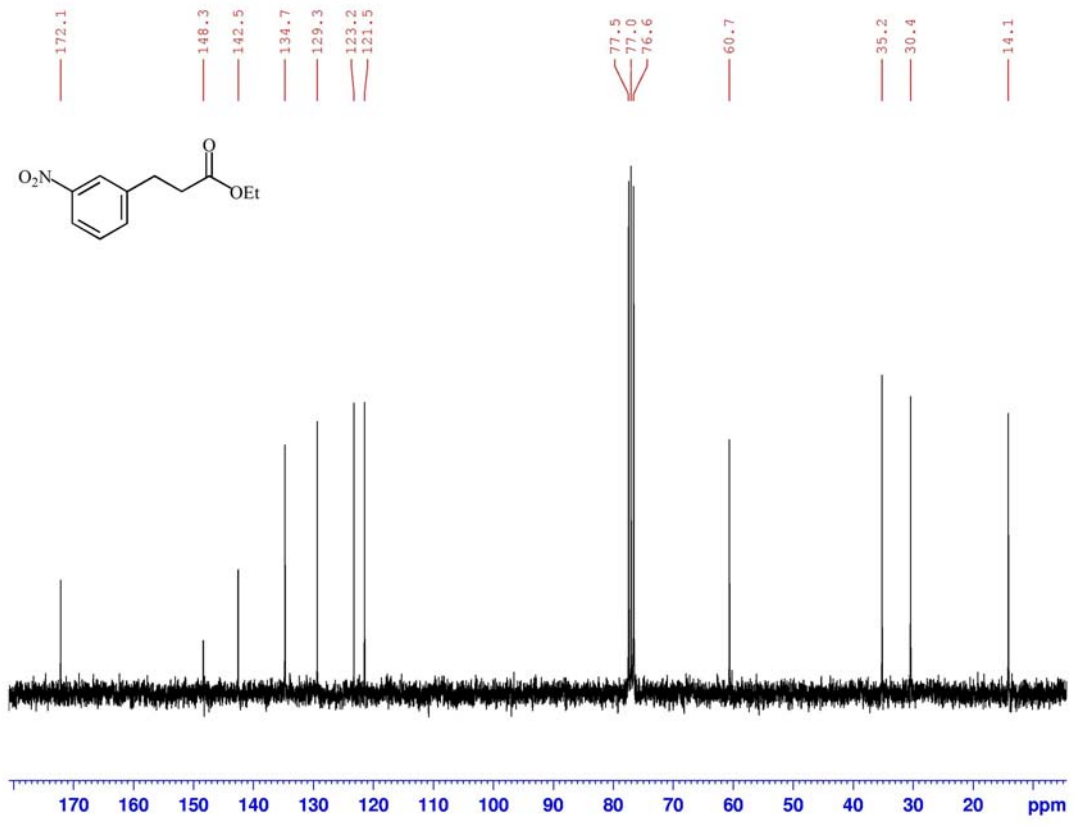
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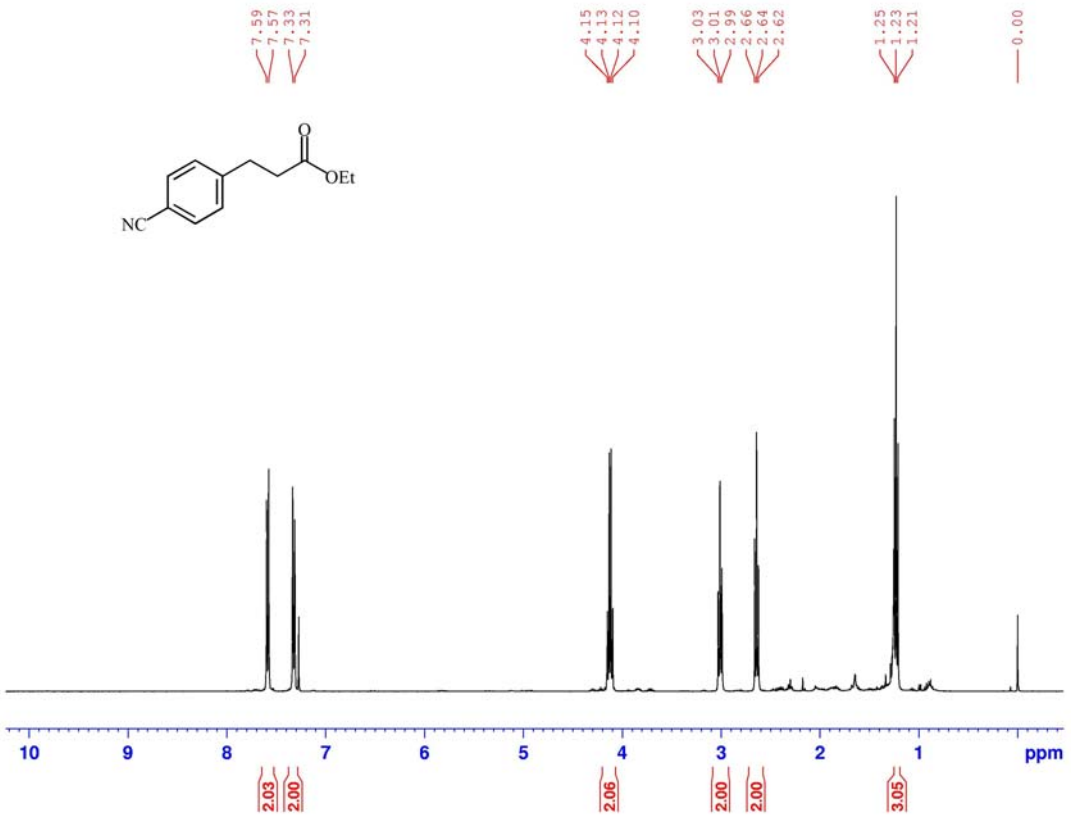
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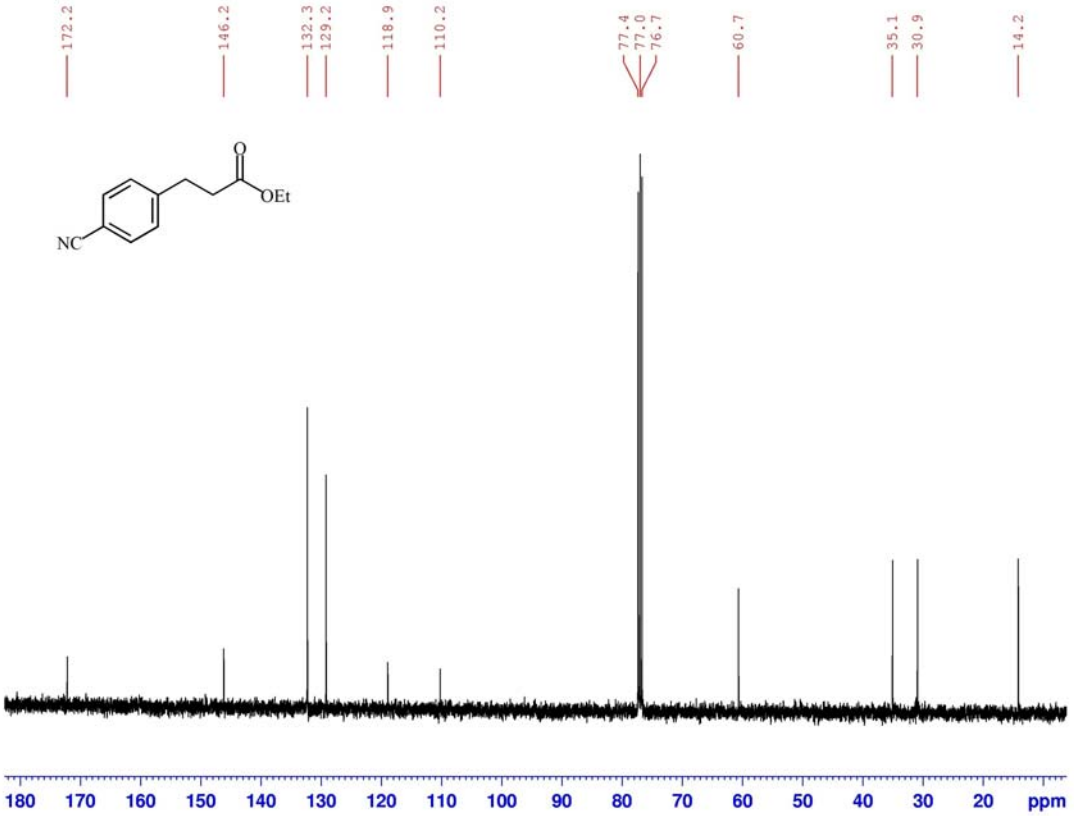
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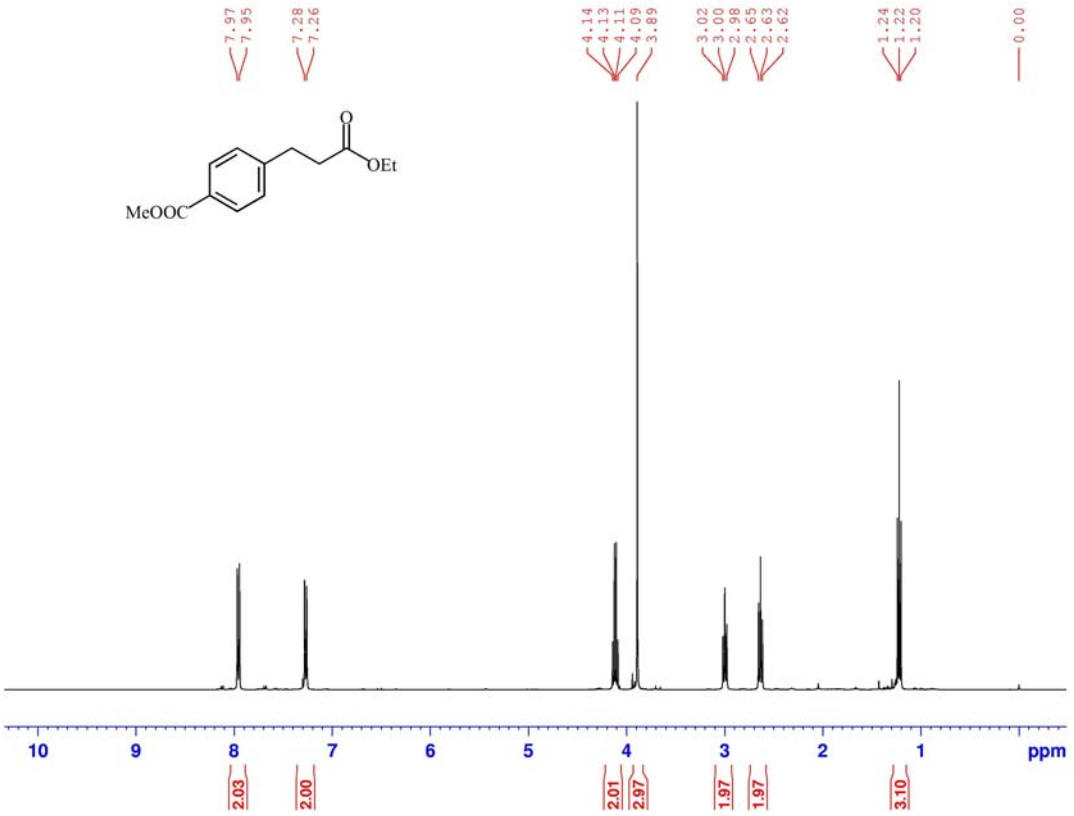
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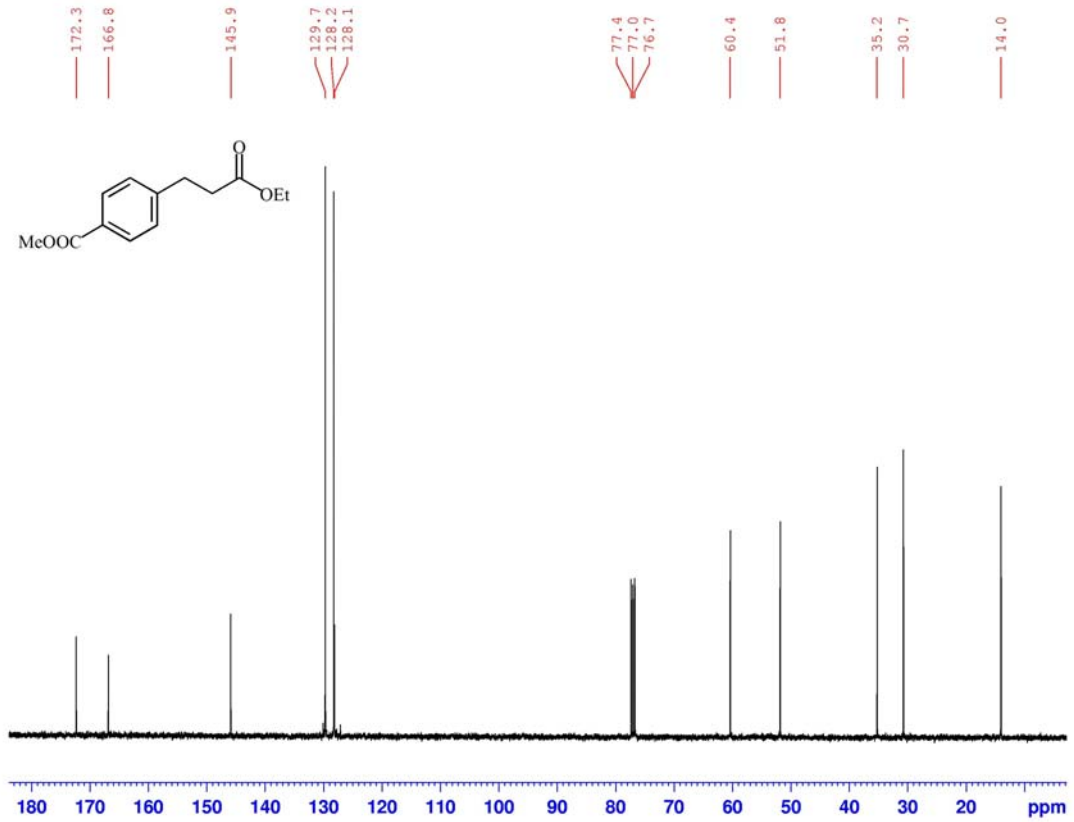
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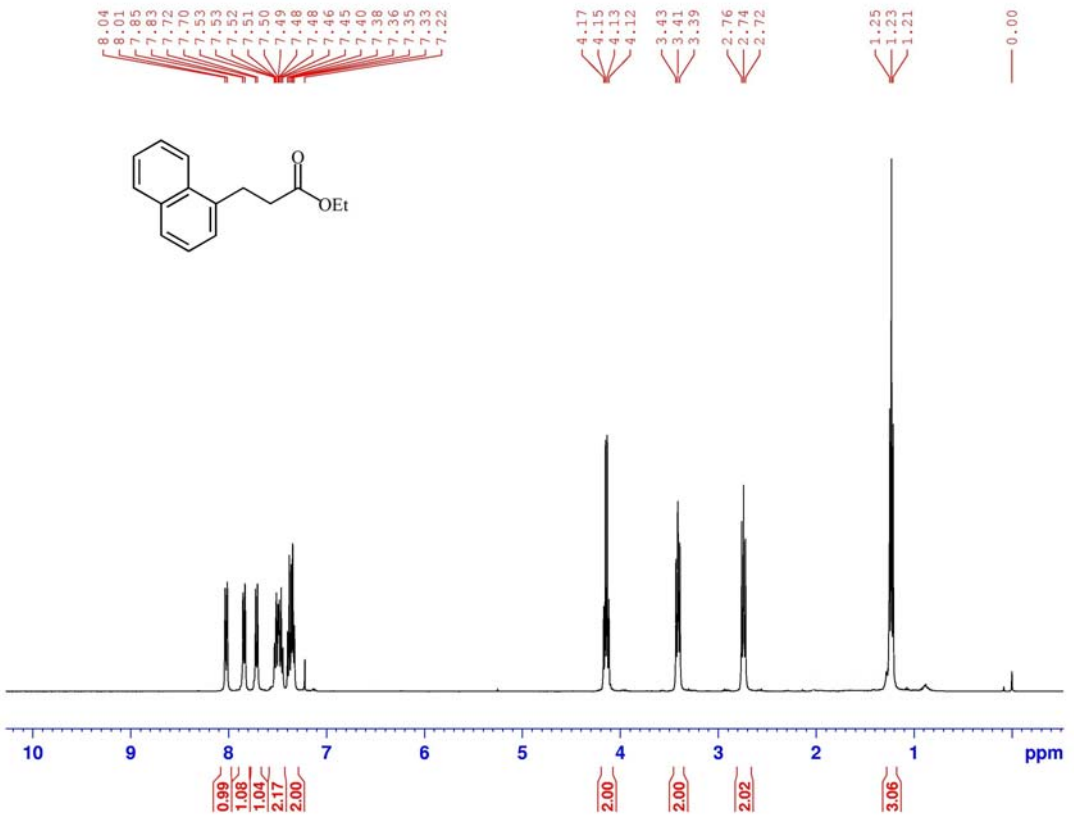
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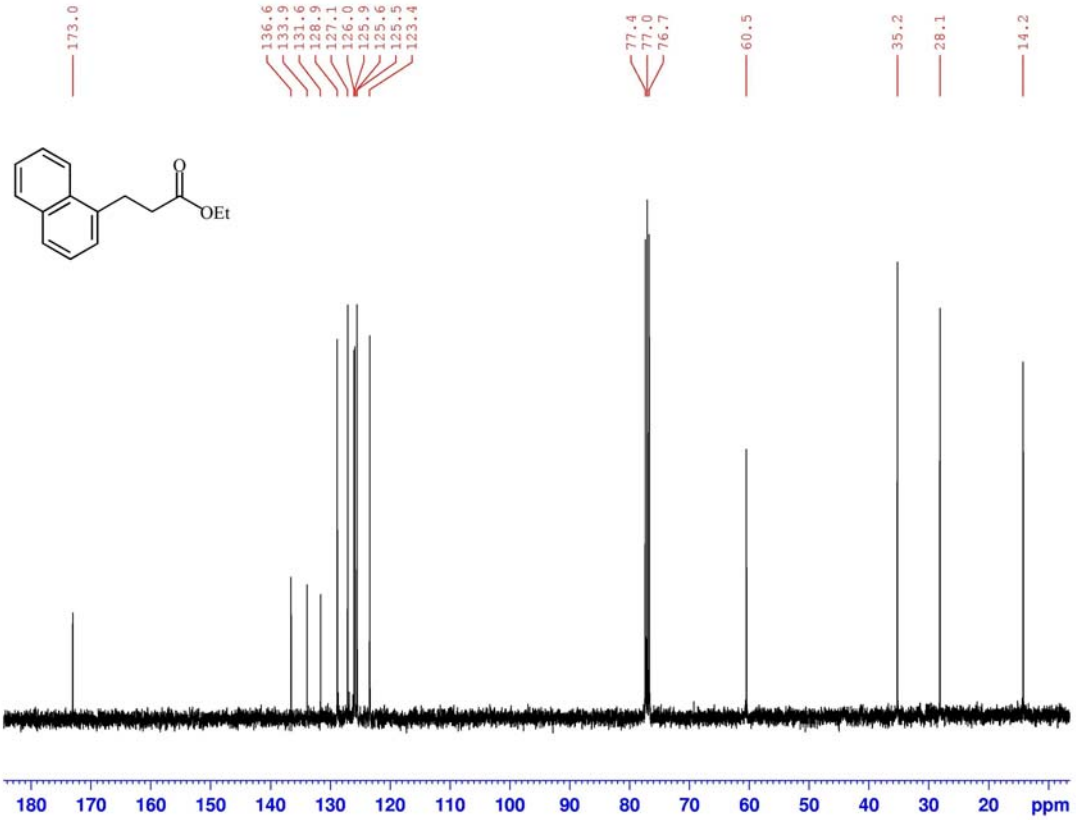
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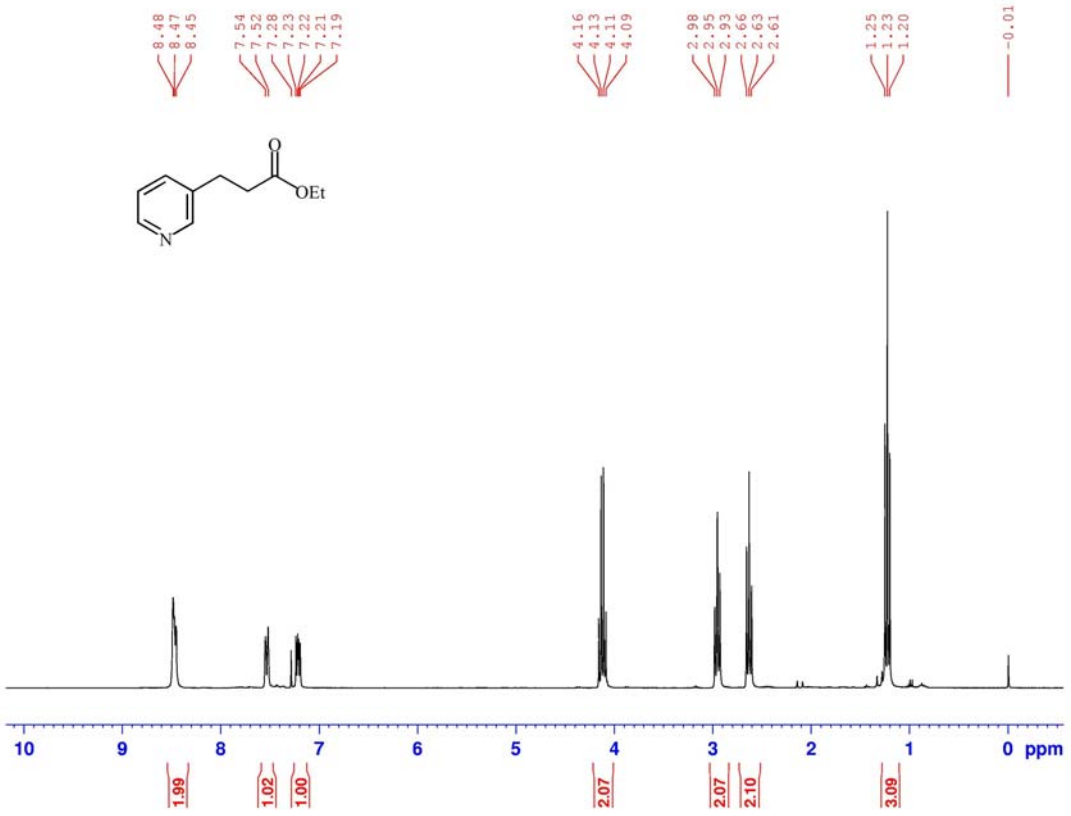
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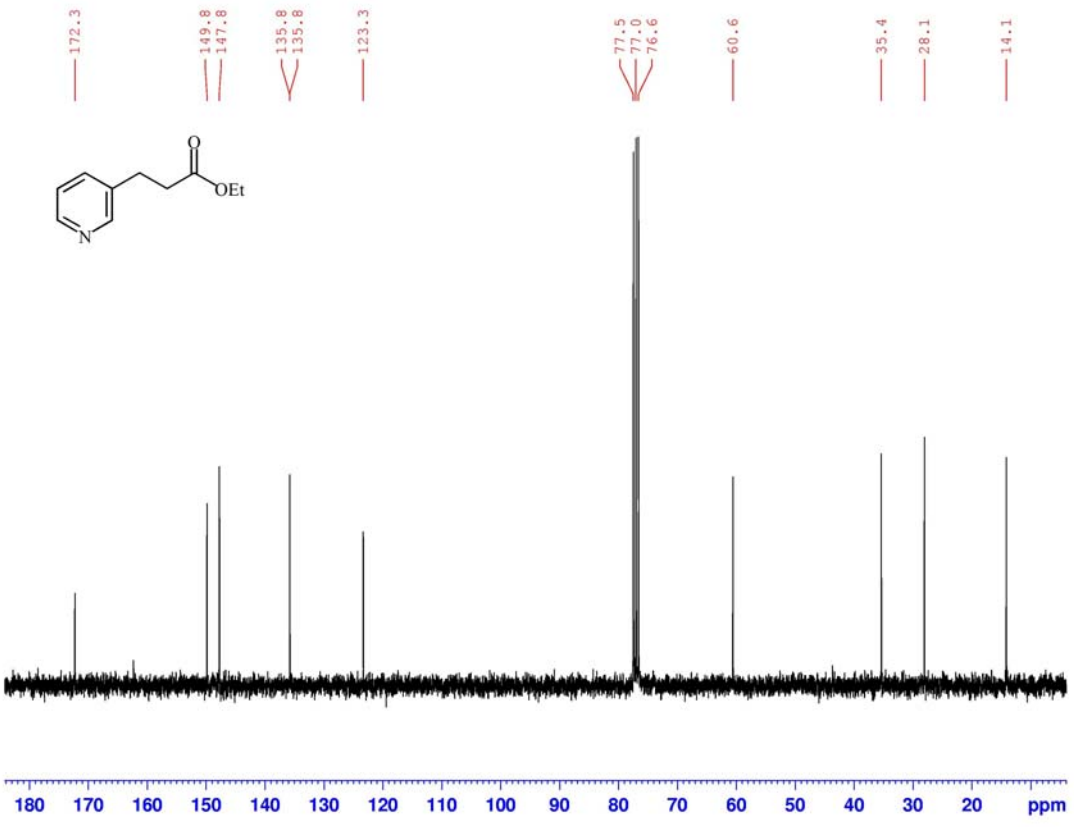
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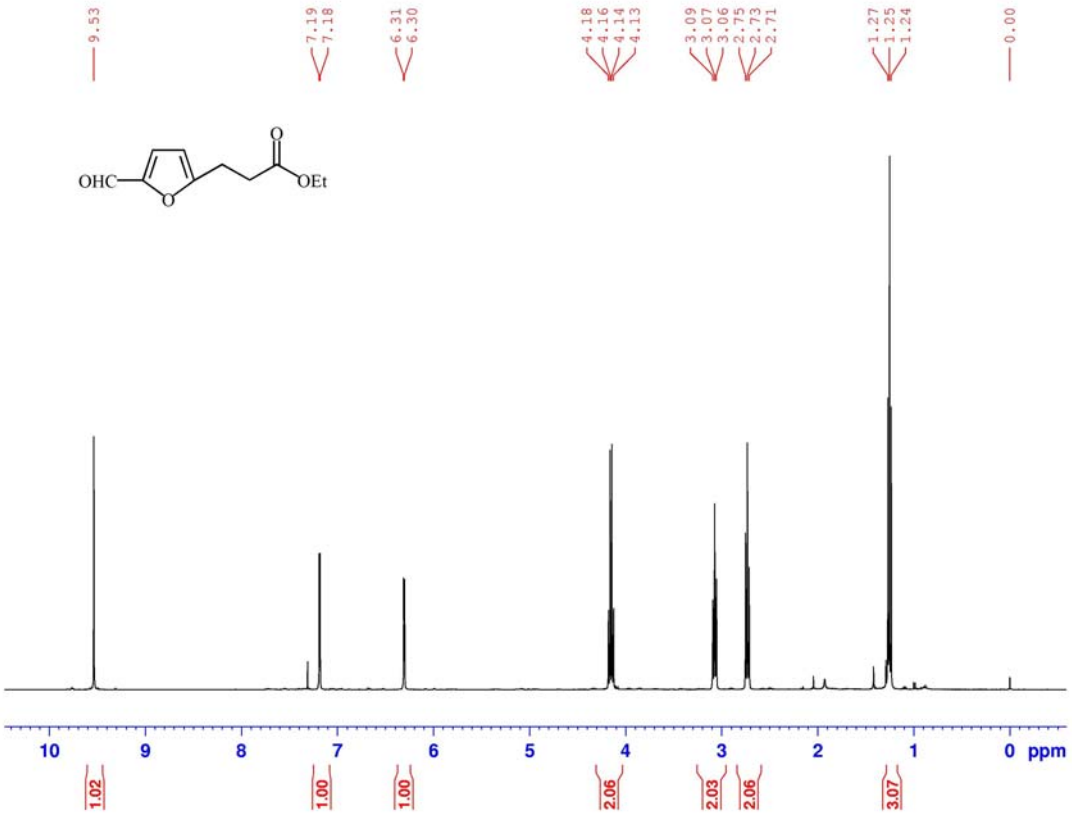
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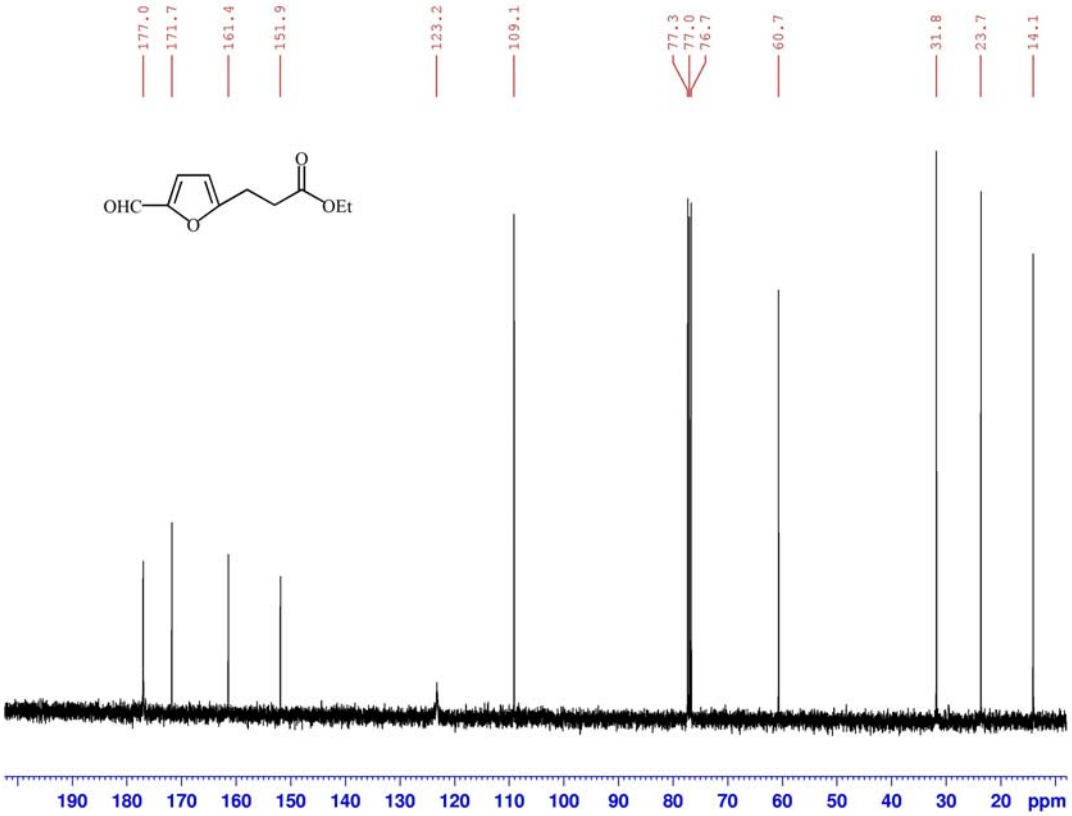
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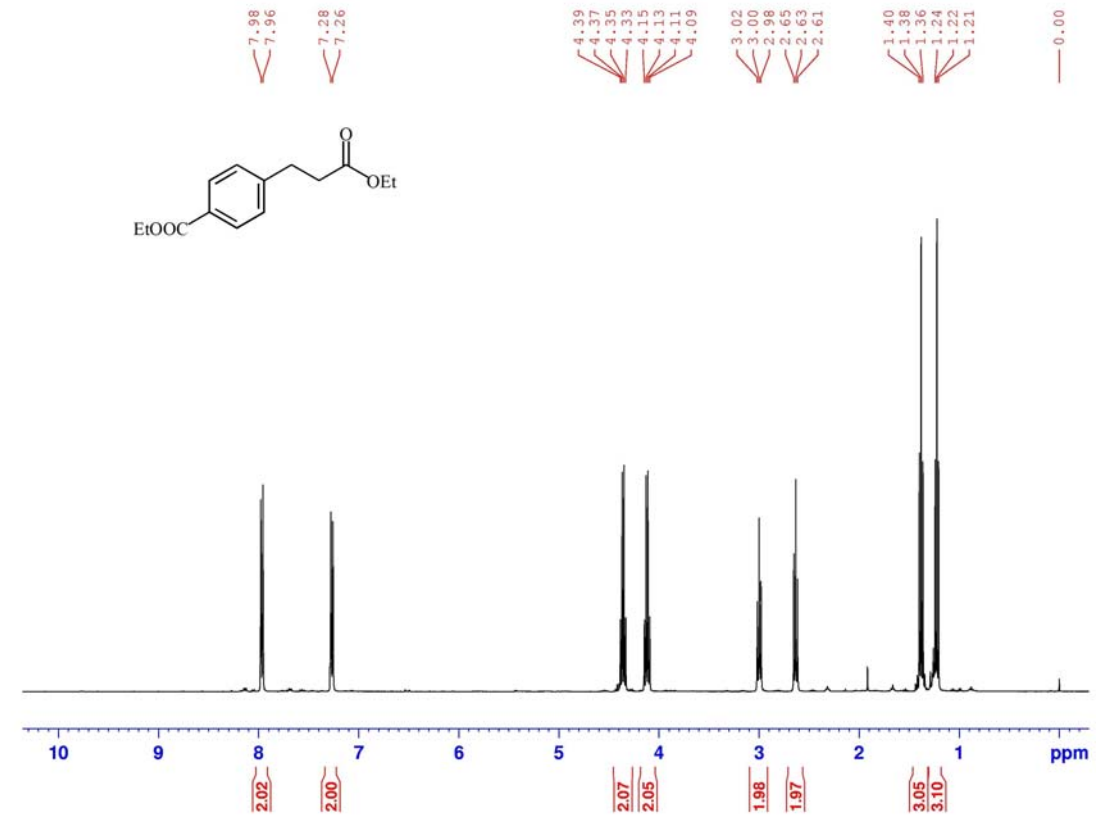
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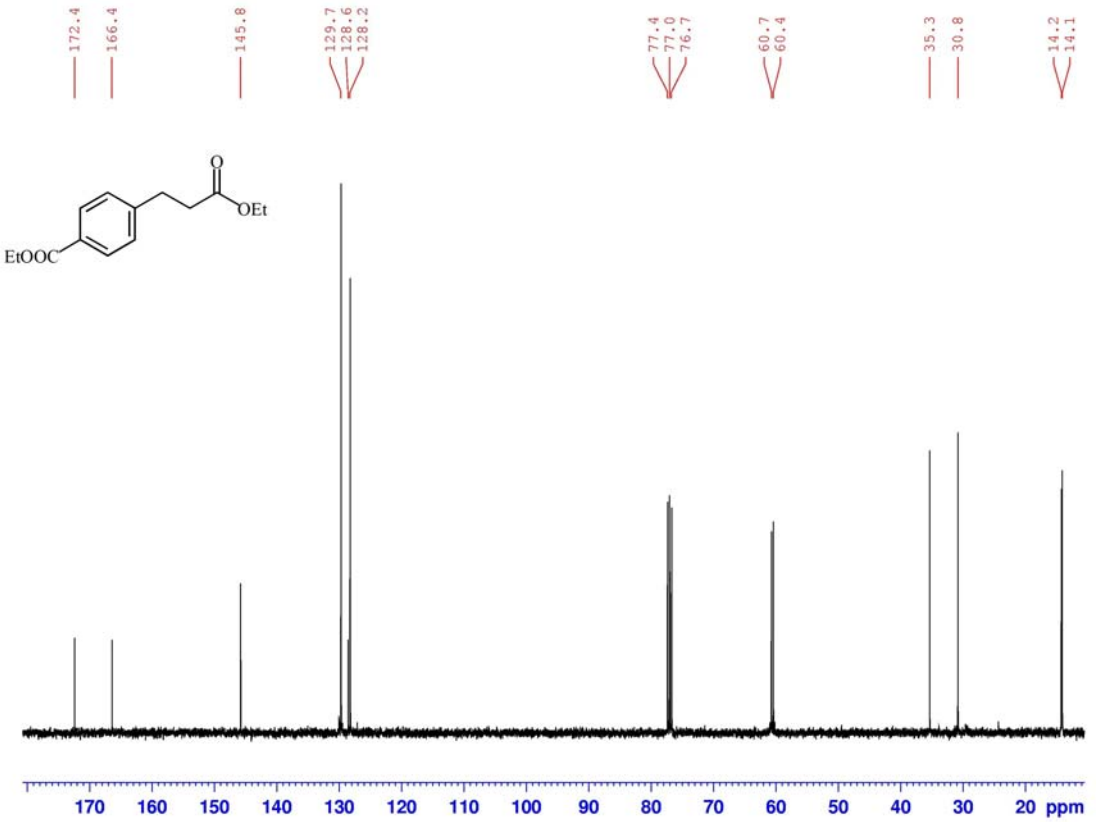
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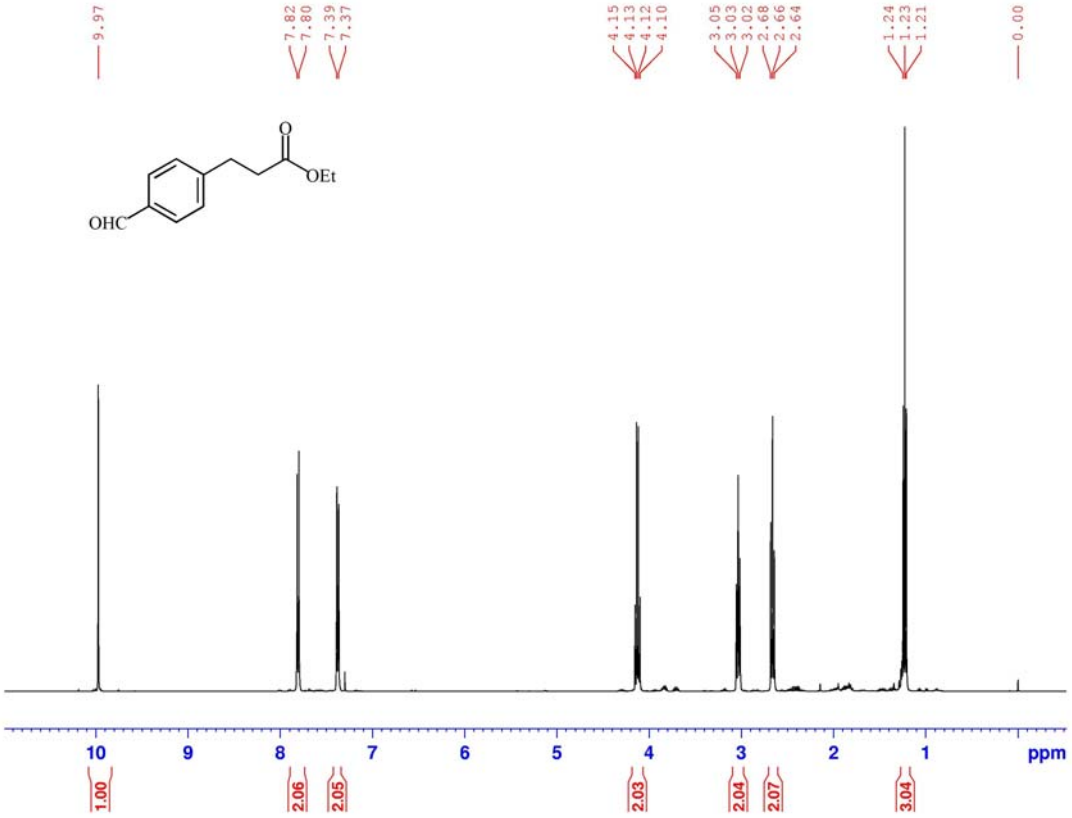
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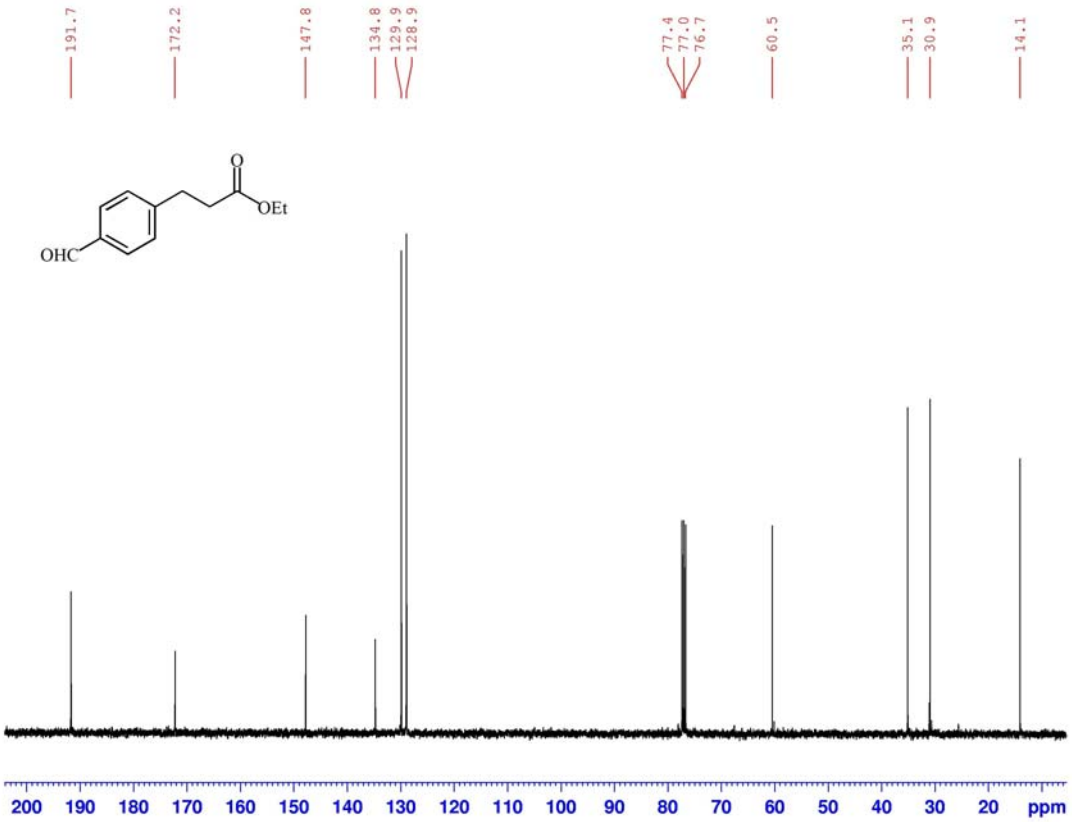
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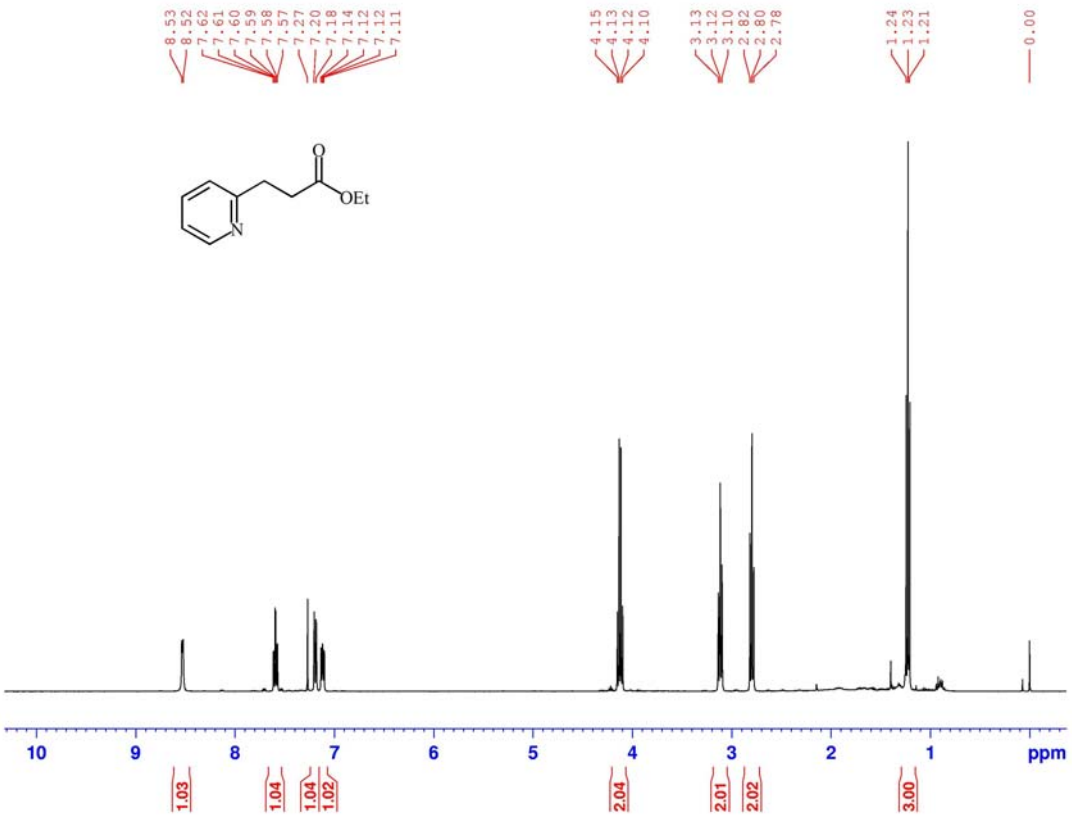
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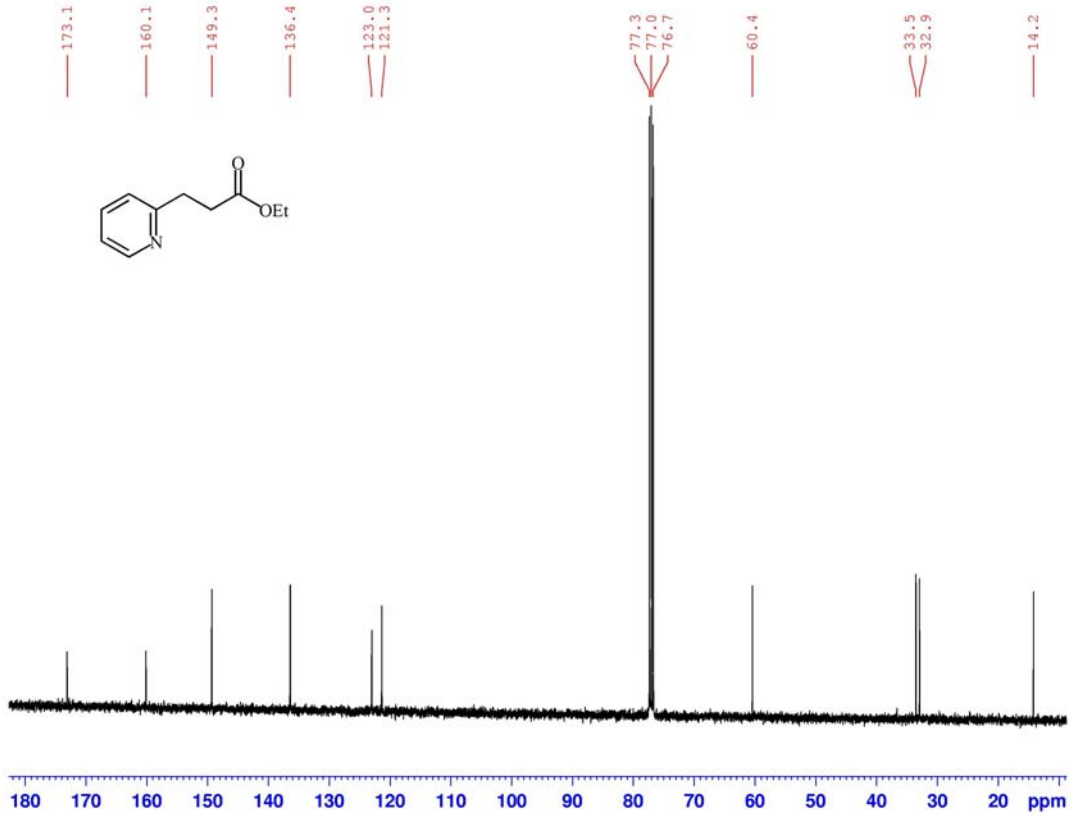
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14.2
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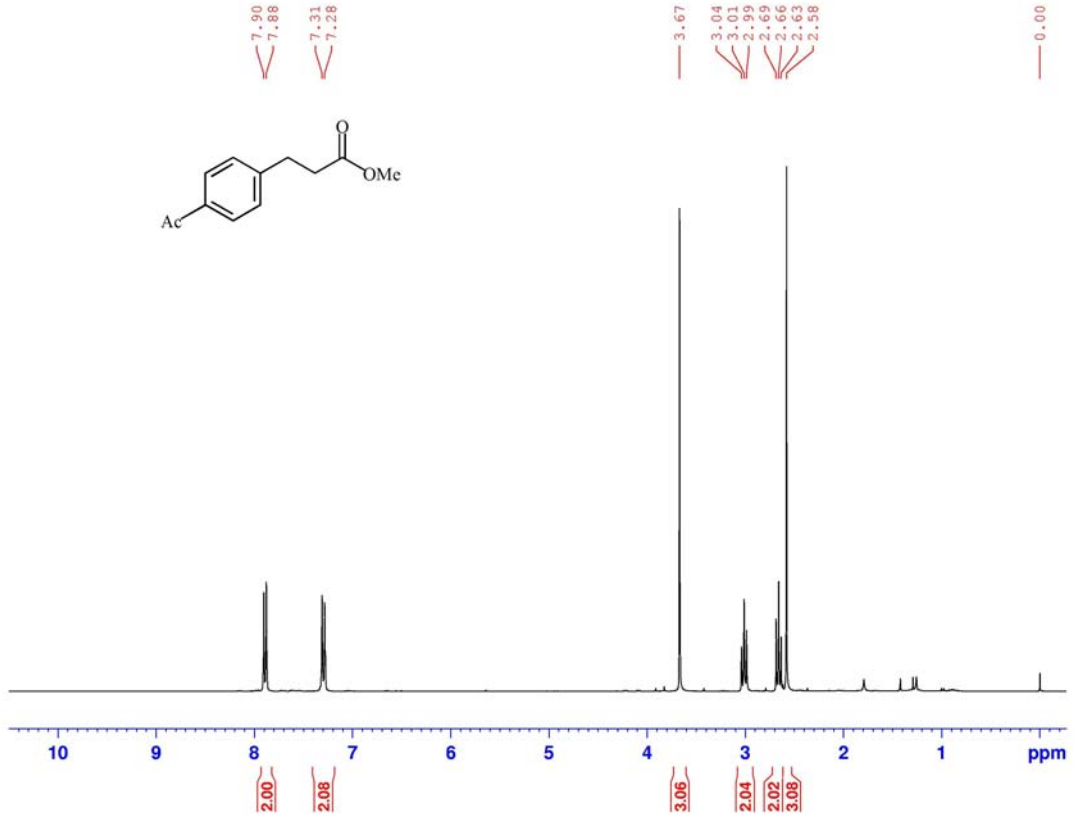
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OMe
Ac
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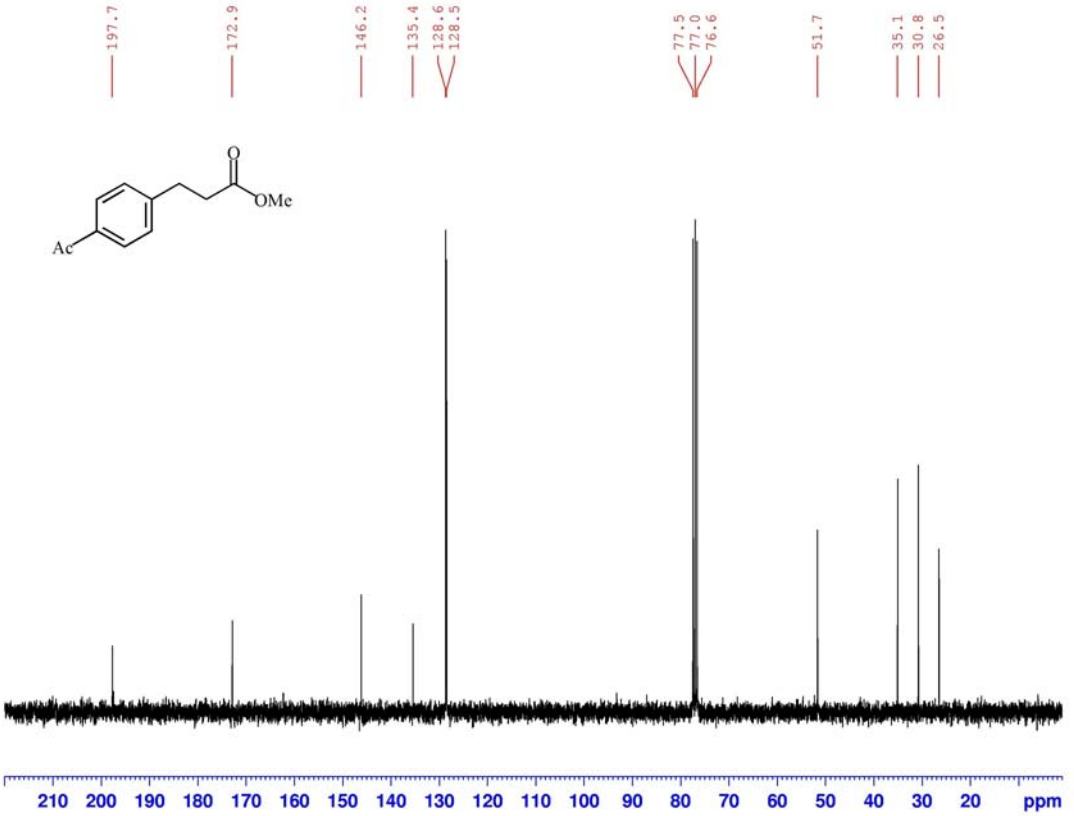
2.04
2.02
3.08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OMe
Ac
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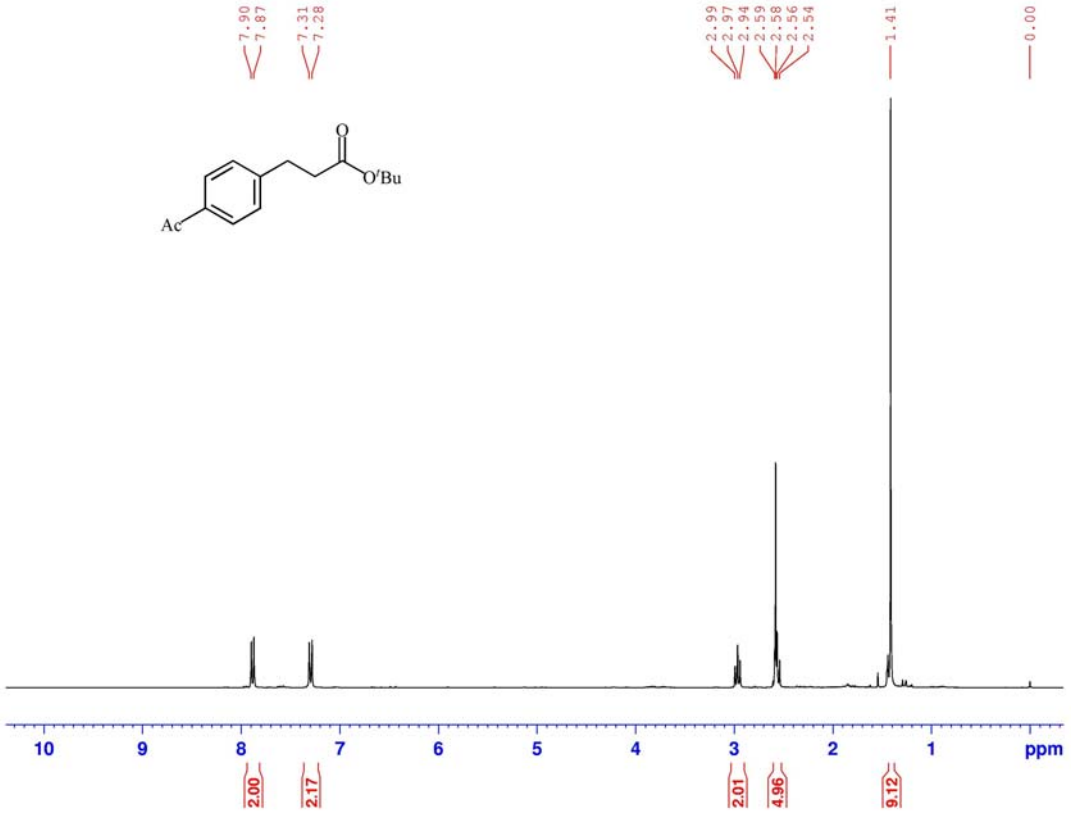
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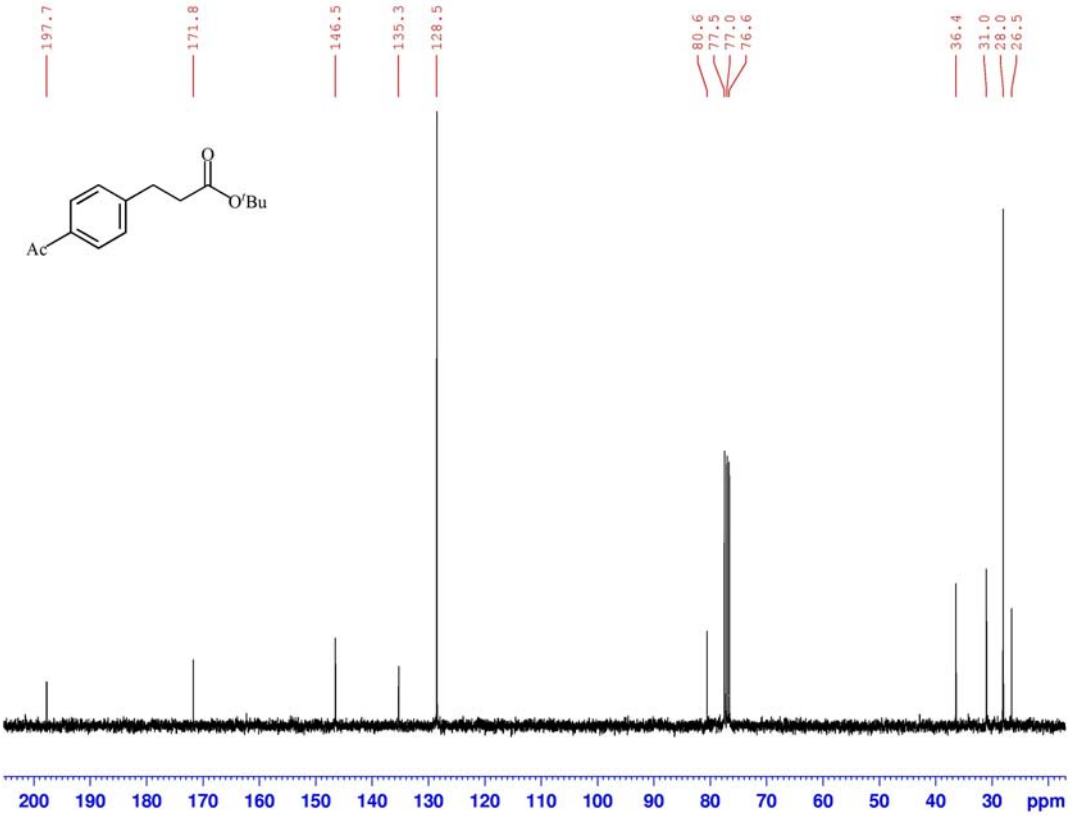
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O
O'Bu
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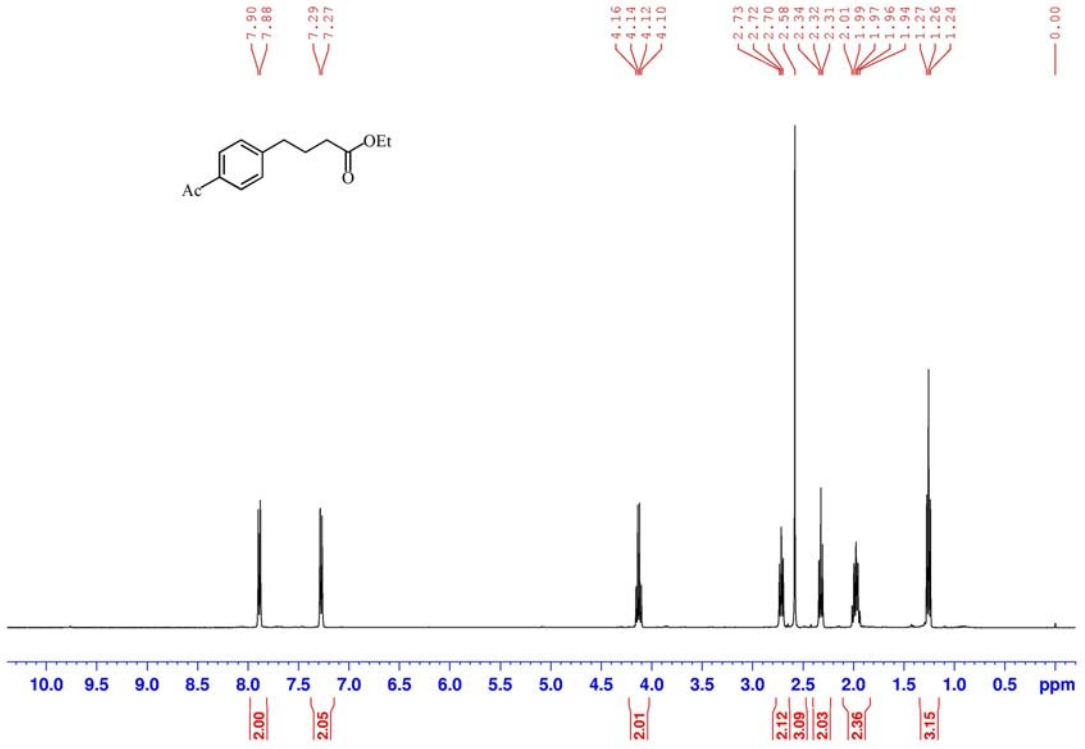
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2.31
2.01
1.99
1.97
1.96
1.94
1.27
1.26
1.24
0.00
OEt
O
Ac
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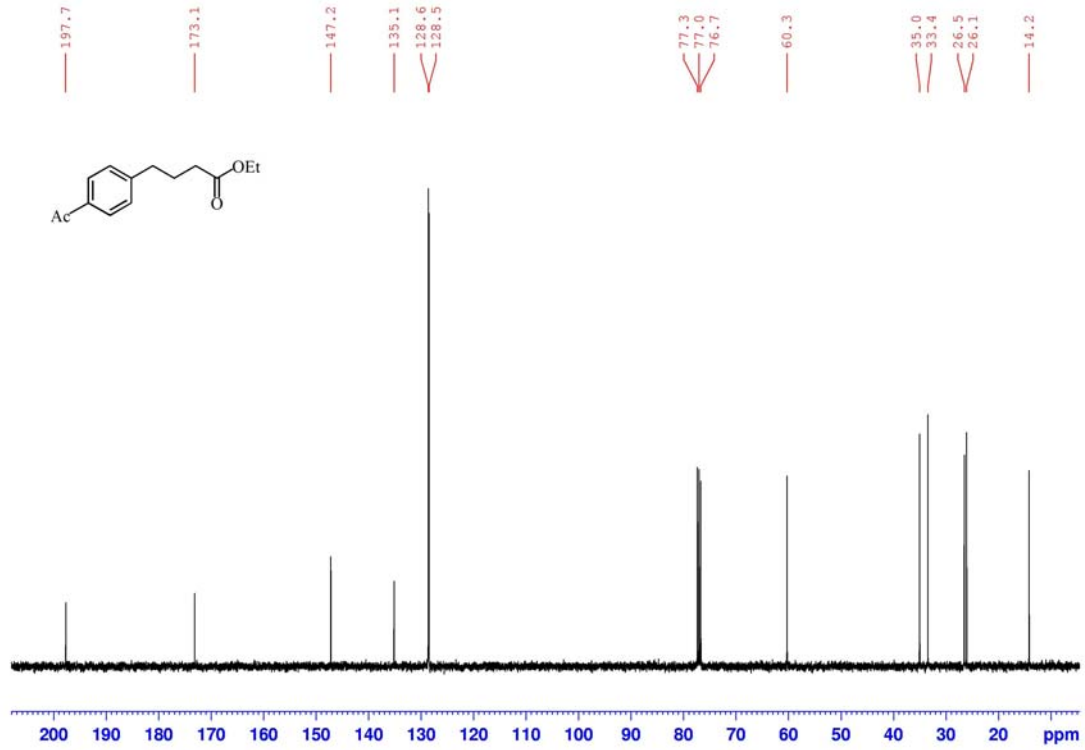
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O
Ac
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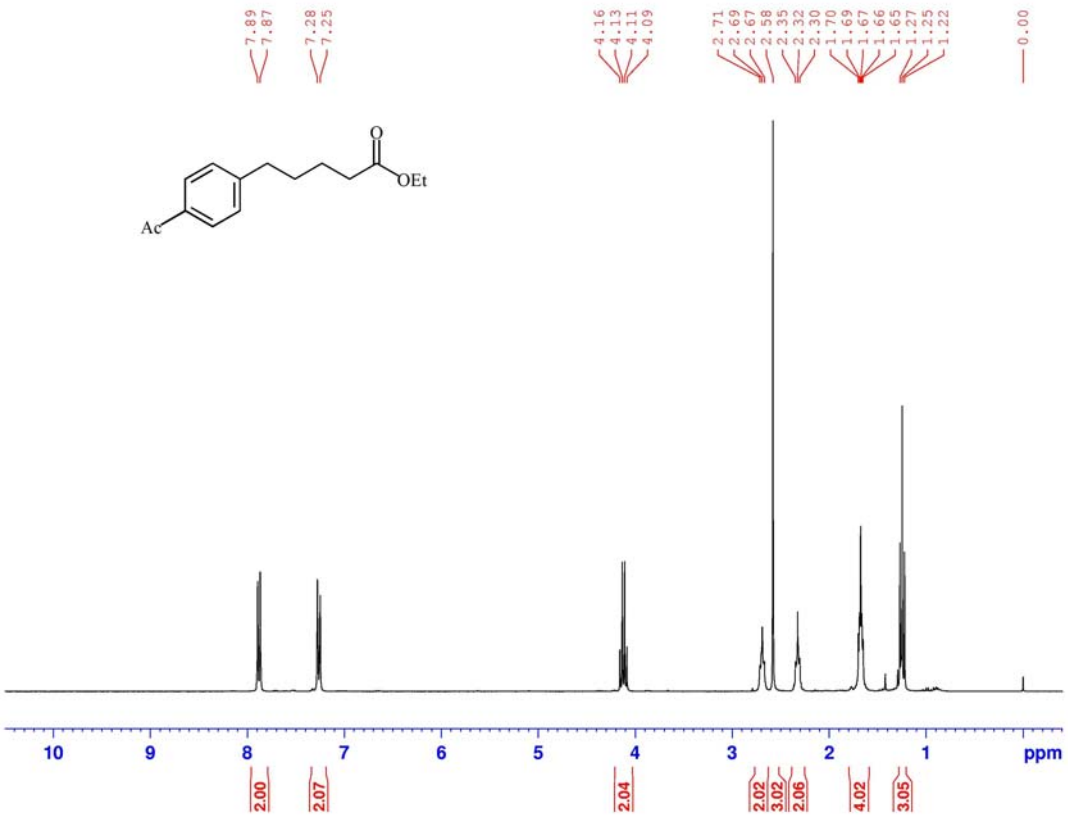
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2.30
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1.69
1.67
1.66
1.65
1.27
1.25
1.22
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OEt
Ac
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7
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ppm
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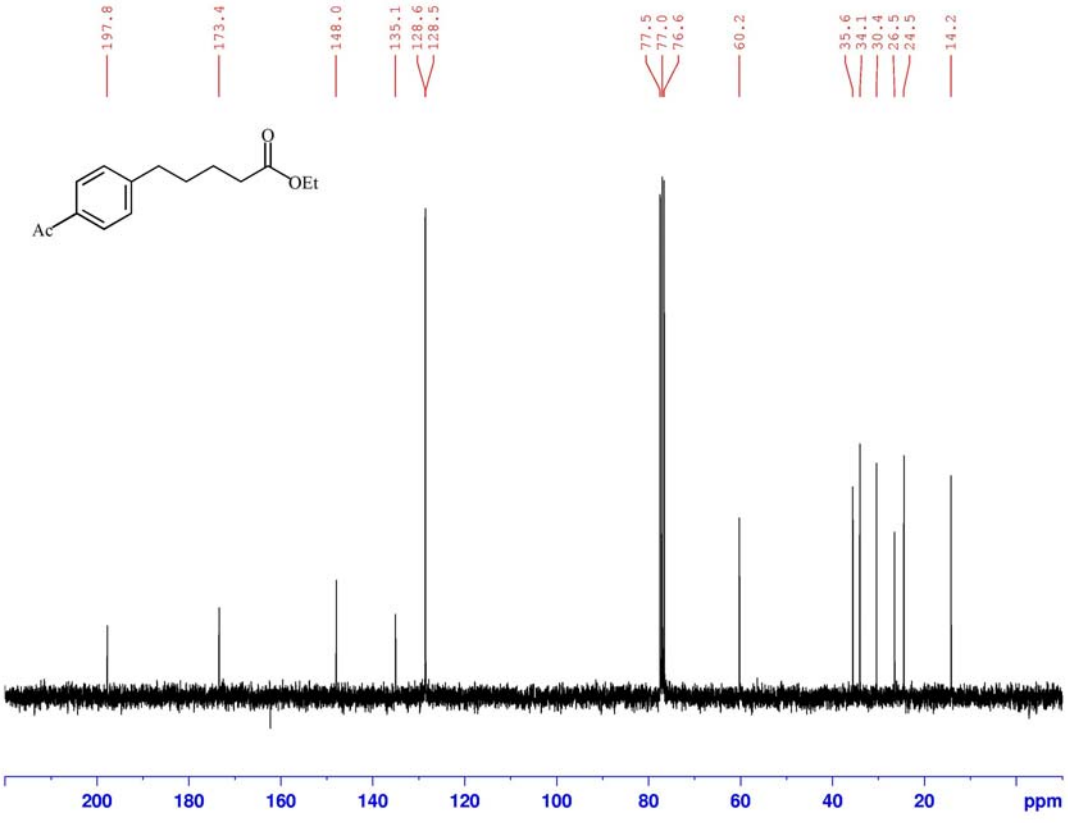
2.02
3.02
2.06
4.02
3.05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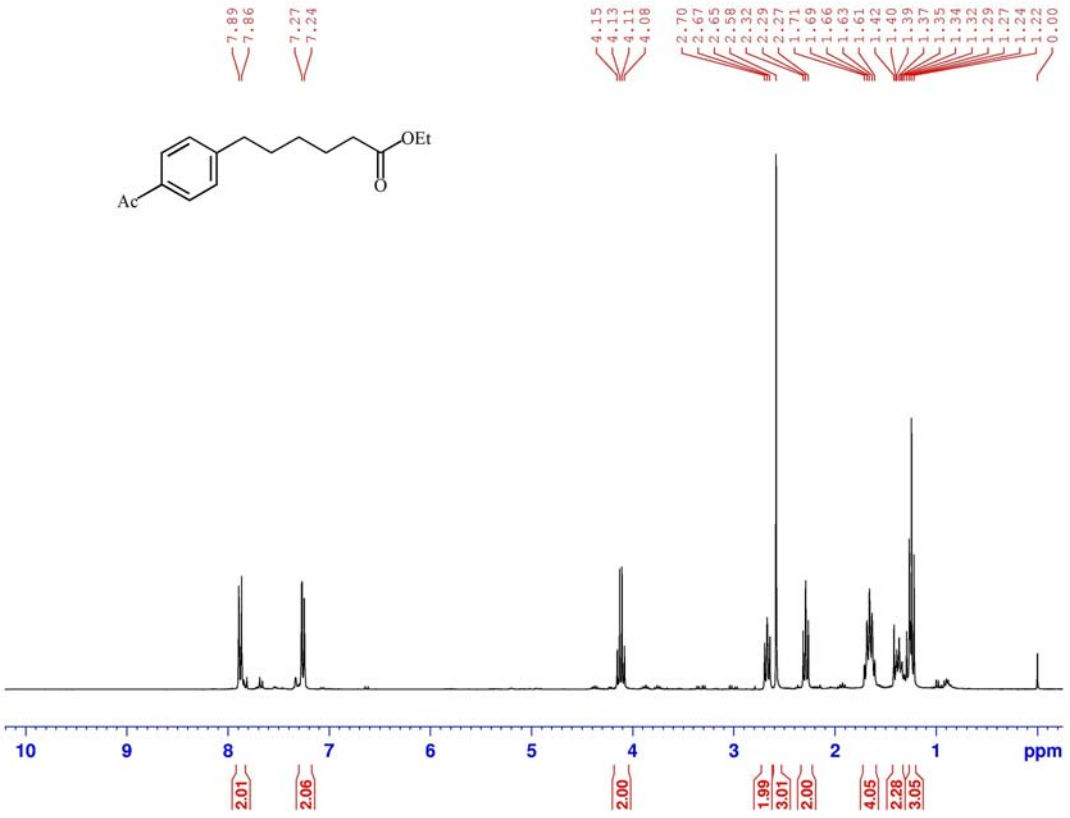
40
20
ppm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1.37
1.35
1.34
1.32
1.29
1.27
1.24
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O
Ac
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6
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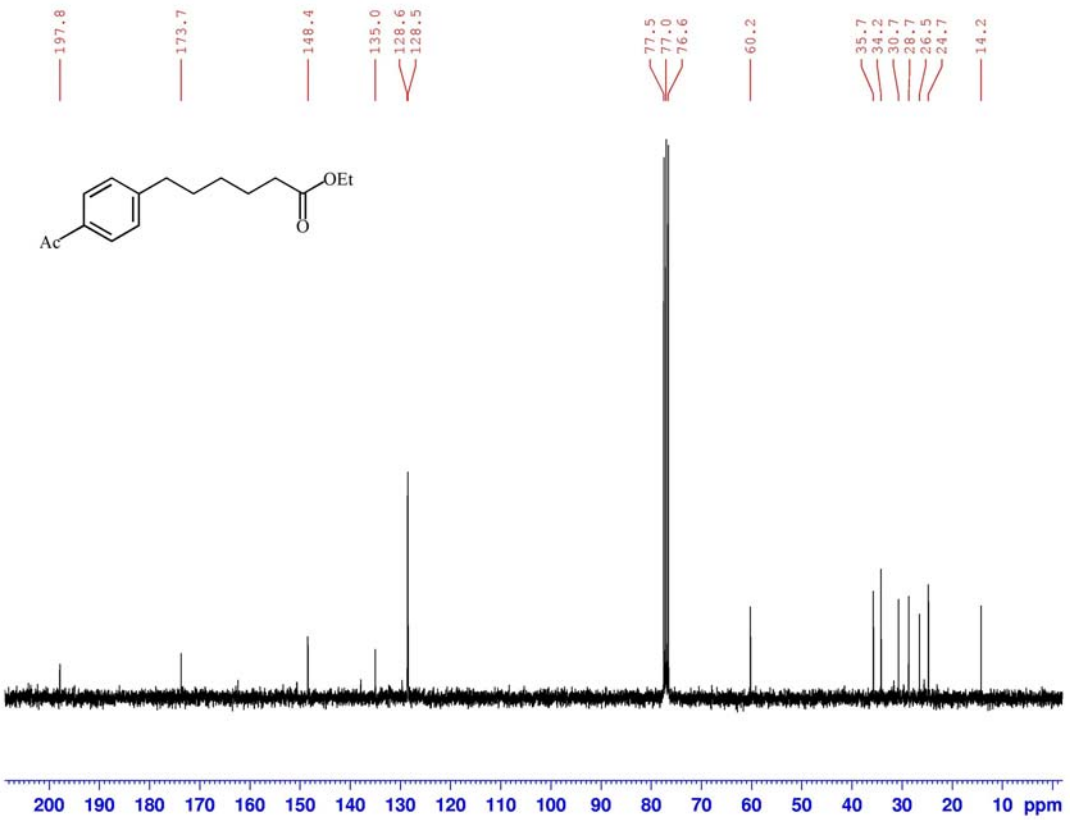
3.05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35.7
34.2
30.7
28.7
26.5
24.7
14.2
OEt
O
Ac
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180
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160
150
140
130
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70
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30
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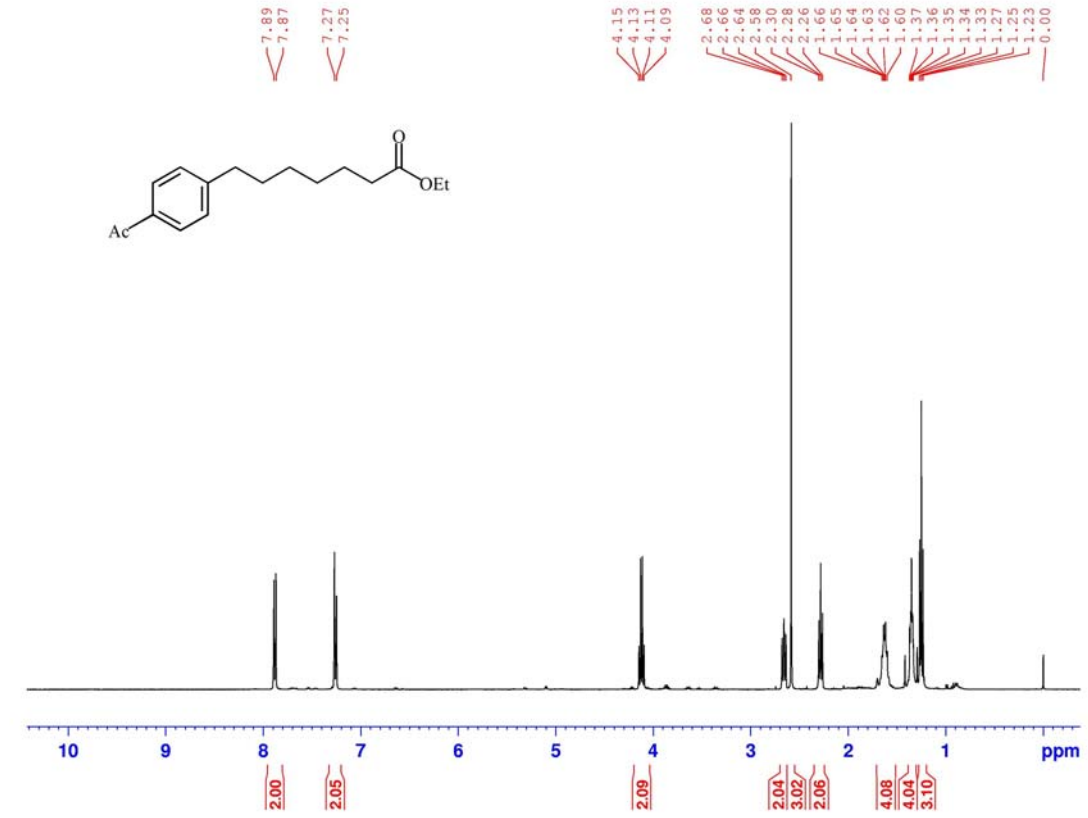
10
ppm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1.65
1.64
1.63
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1.37
1.36
1.35
1.34
1.33
1.27
1.25
1.23
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O
OEt
Ac
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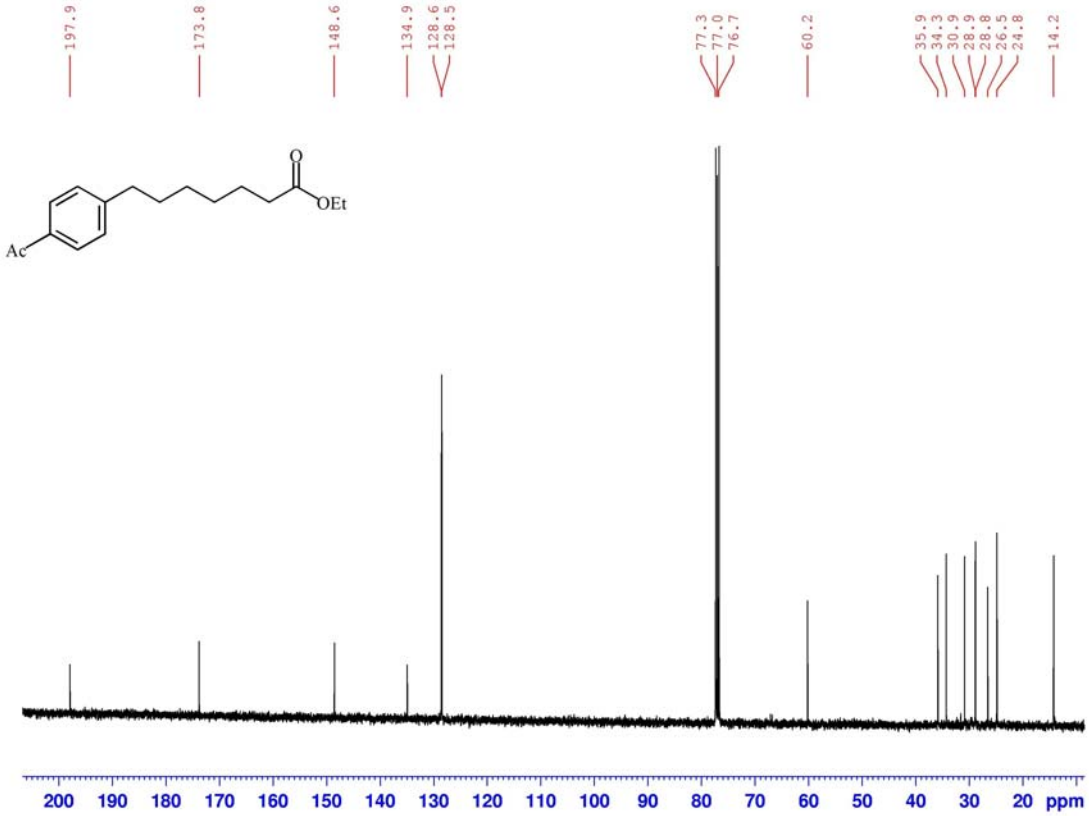
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28.8
26.5
24.8
14.2
O
OEt
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150
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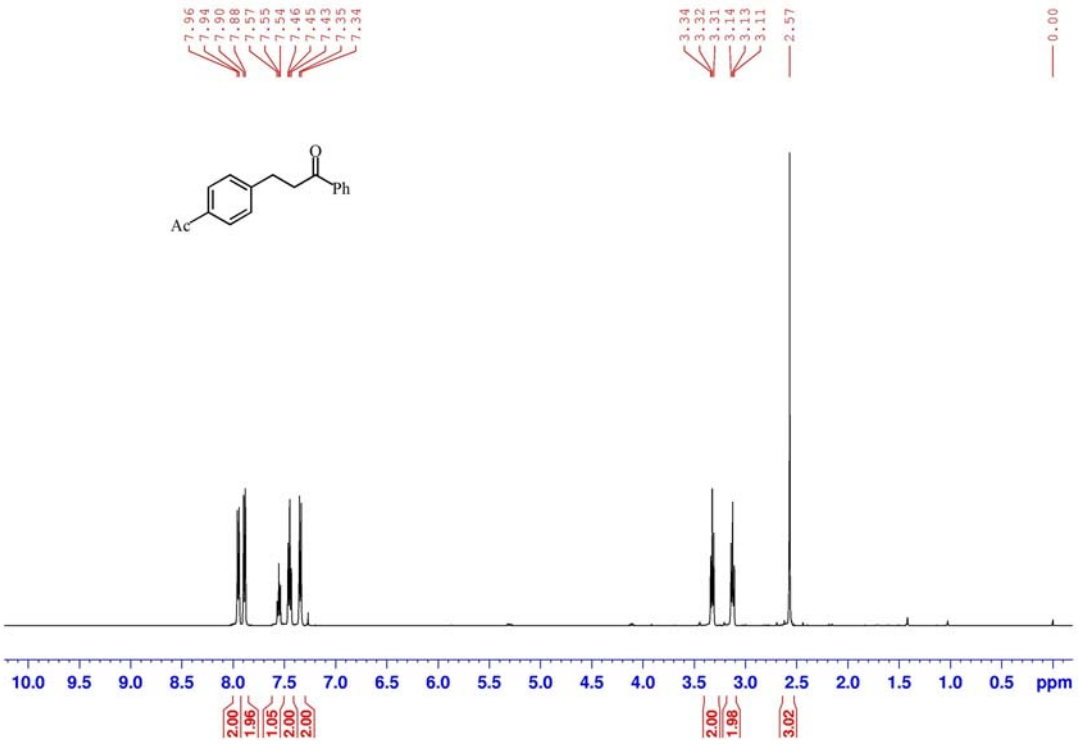
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3.32
3.31
3.14
3.13
3.11
2.57
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O
Ph
Ac
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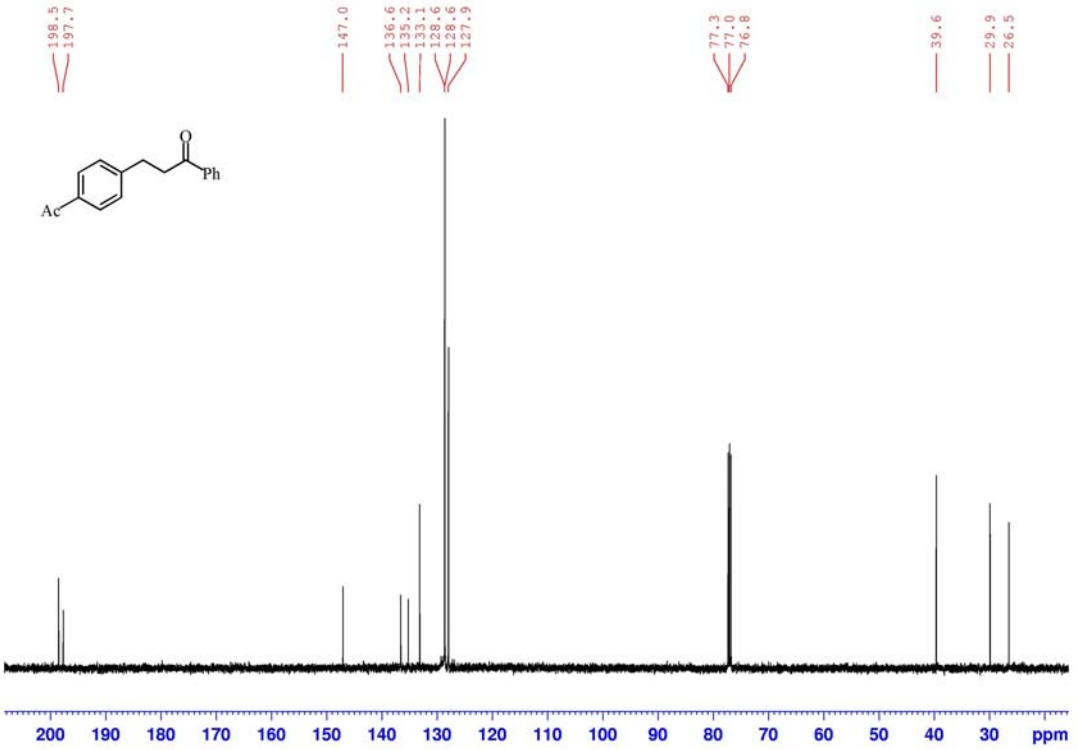
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Ac
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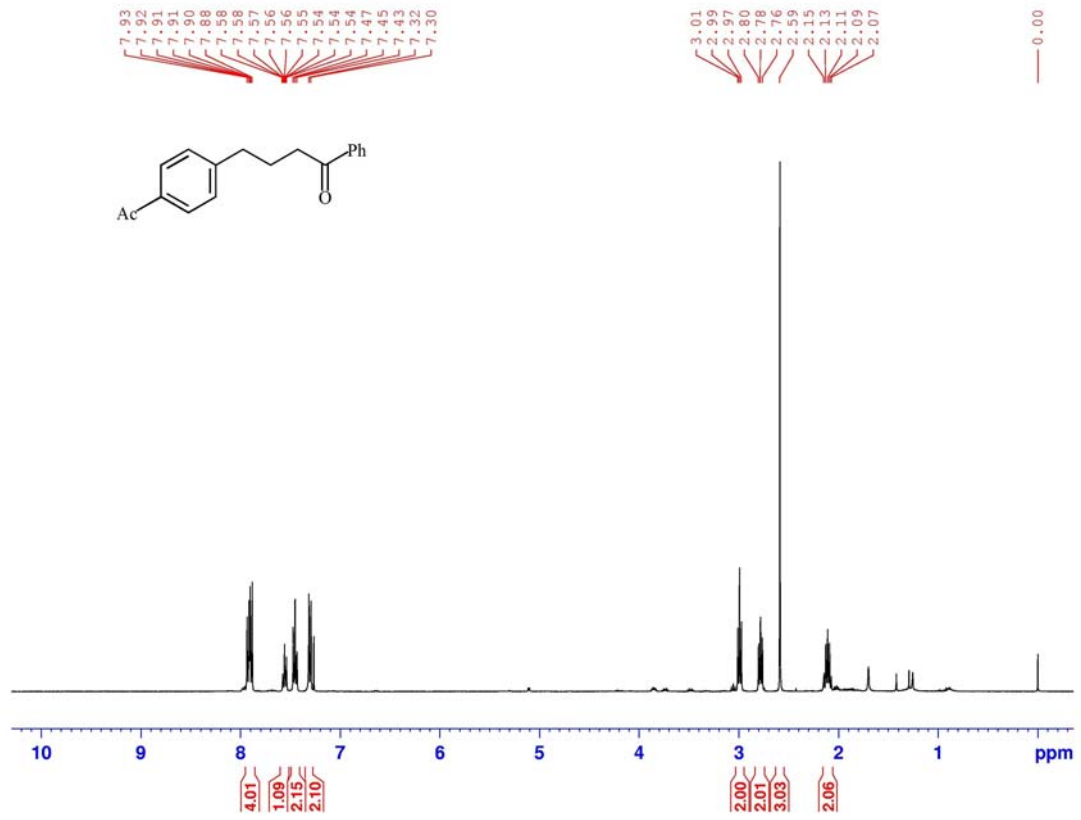
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Ph
O
Ac
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ppm
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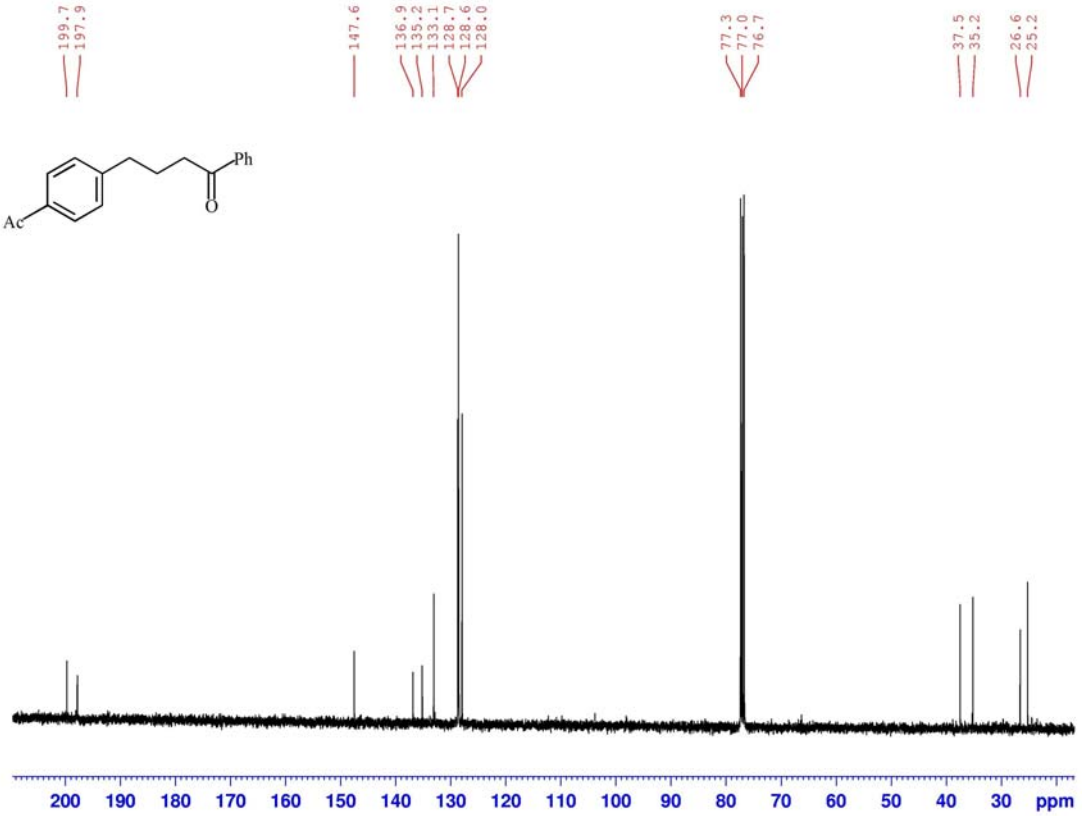
2.06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37.5
35.2
26.6
25.2
Ph
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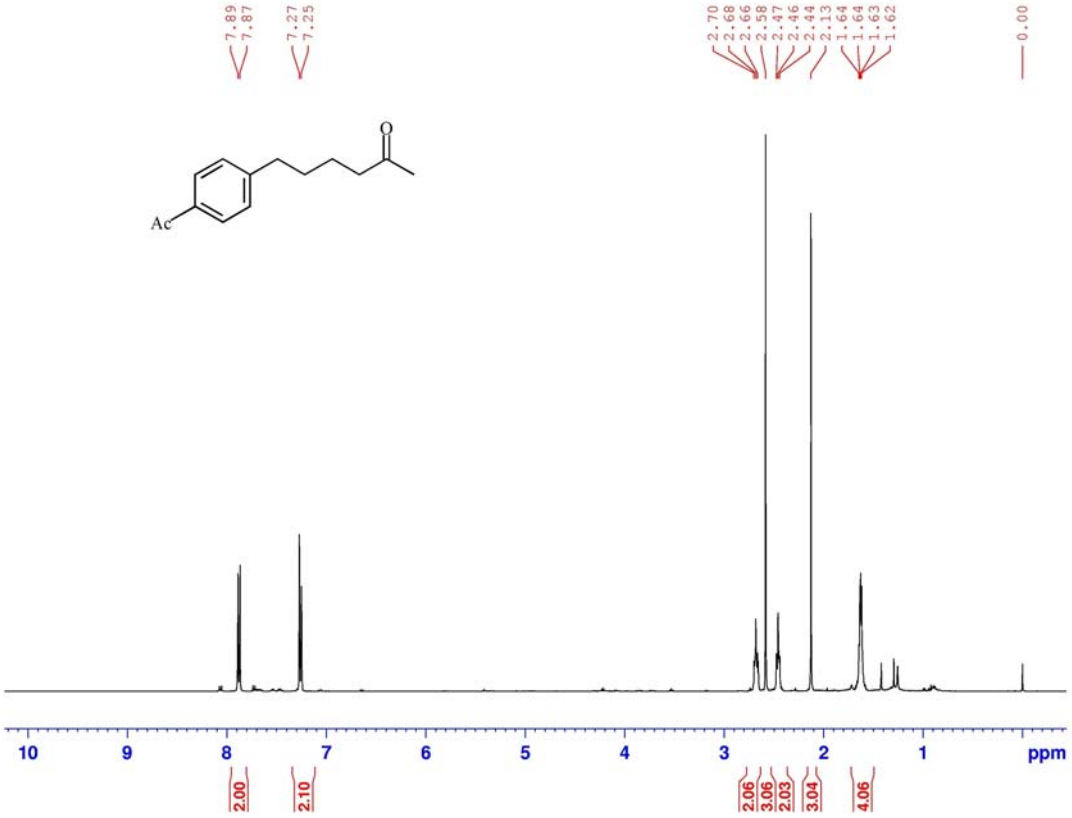
ppm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Ac
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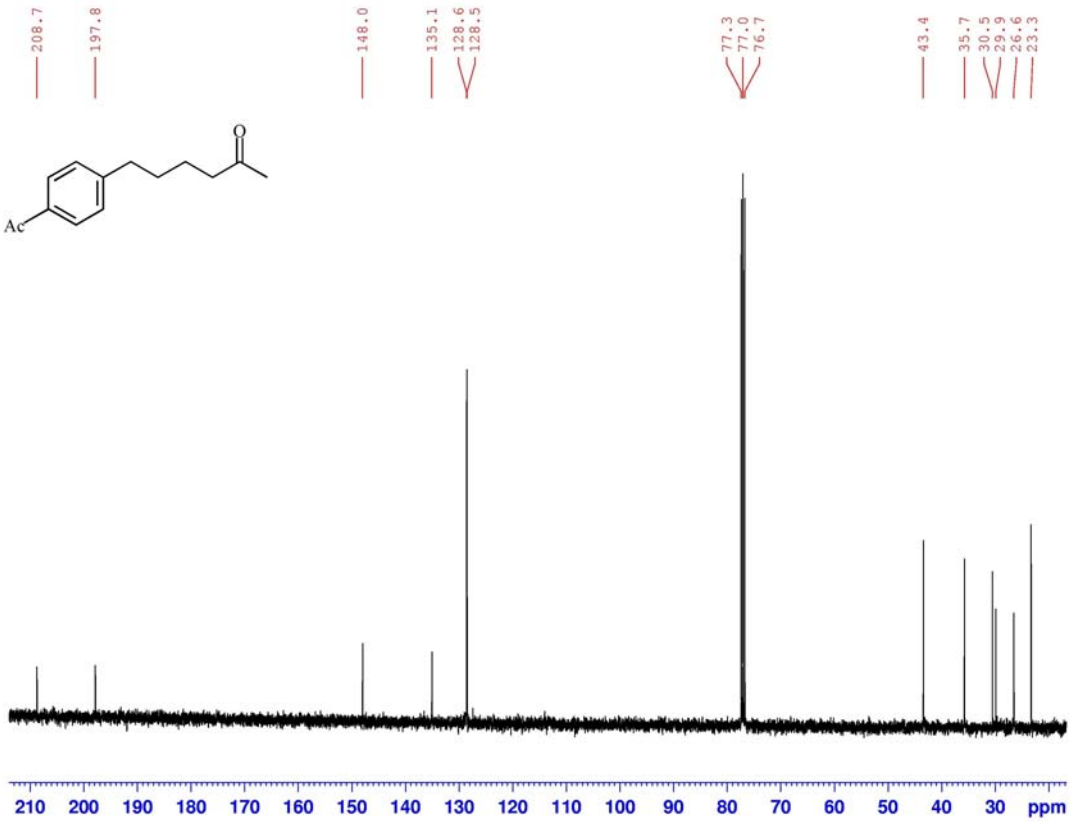
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26.6
23.3
O
Ac
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ppm