Palladium-Catalyzed Carbonylative Arylacetamides Synthesis from Benzyl Formates and Tertiary Amines

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Supporting Information

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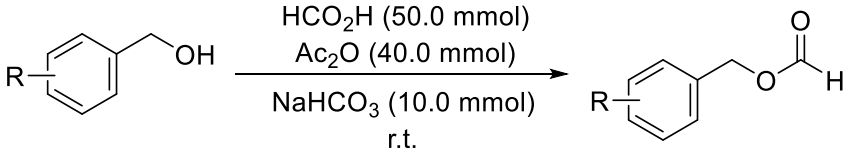
1. General Information

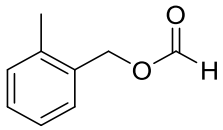
Unless otherwise noted, all reactions were carried out under nitrogen atmosphere. organic carbonates were from commercial sources and used as received without further purification. Benzyl formates that were not commercially available were synthesized according to existing method. Column chromatography was performed on silica gel (200-300 meshes) using petroleum ether (Bp. 60-90 °C) and ethyl acetate as eluent. ^{1}H and ^{13}C NMR spectra were taken on 400 MHz instruments and spectral data were reported in ppm relative to tetramethylsilane (TMS) as internal standard and $CDCl_3$ (^{1}H NMR δ 7.26, ^{13}C NMR δ 77.0) as solvent. All coupling constants (J) are reported in Hz with the following abbreviations: s = singlet, d = doublet, dd = double doublet, ddd = double doublet of doublets, t = triplet, dt = double triplet, q = quartet, m = multiplet, br = broad. Gas chromatography (GC) analyses were performed on a Shimadzu GC-2014C chromatograph equipped with a FID detector. Mass spectra (MS) were measured on spectrometer by direct inlet at 70 eV. IR spectra were collected with Bruker-VERTEX 70 spectrometer and only major peaks were reported in cm^{-1}.

2. General Procedure for the Syntheses of the Benzyl Formates[1]

Formic acid (50.0 mmol) was added to acetic anhydride (40.0 mmol) at room temperature. The resulting mixture was stirred at 60 °C for 1 h and then cooled at room temperature. Benzyl alcohols (5.0 mmol) and $NaHCO_3$ (10.0 mmol) were added to the solution, and the mixture was stirred until starting material was consumed. The reaction was quenched by adding a mixture of EA and water, and the biphasic system was stirred vigorously. Then the organic phase was separated, and the aqueous phase was extracted with EA for 2 times. The organic phases were combined and washed with water and brine, and then dried over anhydrous Na_2SO_4. The resulting mixture was concentrated by rotary evaporation. The crude mixture was purified by silica gel column chromatography to afford the desired product.

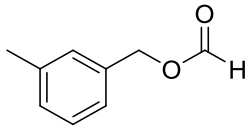


3. Characterization of the Benzyl Formates



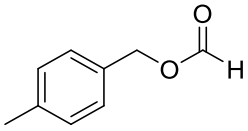
2-Methylbenzyl formate, 1b[2]

0.56 g, 75% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.24 (d, J = 6.9 Hz, 1H), 7.18-7.08 (m, 3H), 5.11 (s, 2H), 2.26 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 136.9, 133.1, 130.3, 129.4, 128.7, 126.0, 63.9, 18.7.



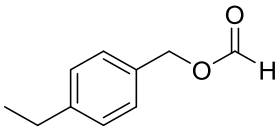
3-Methylbenzyl formate, 1c[3]

0.70 g, 93% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.10 (t, J = 7.5 Hz, 1H), 7.04-6.95 (m, 3H), 5.00 (s, 2H), 2.20 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.5, 138.0, 135.0, 128.9, 128.8, 128.3, 125.2, 65.4, 21.0.



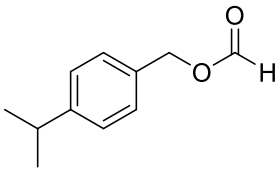
4-Methylbenzyl formate, 1d[4]

0.41 g, 55% yield, pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.20 (d, J = 7.9 Hz, 2H), 7.10 (d, J = 7.8 Hz, 2H), 5.08 (s, 2H), 2.29 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 137.9, 132.1, 128.9, 128.2, 65.1, 20.7.



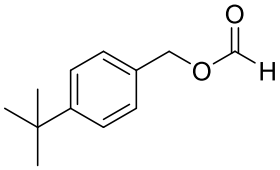
4-Ethylbenzyl formate, 1e[5]

0.49 g, 60% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.27 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 7.9 Hz, 2H), 5.14 (s, 2H), 2.63 (q, J = 7.5 Hz, 2H), 1.22 (td, J = 7.6, 2.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 144.6, 132.4, 128.5, 128.0, 65.5, 28.5, 15.4.



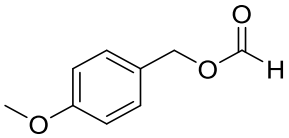
4-Isopropylbenzyl formate, 1f[5]

0.51 g, 57% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.29 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 8.1 Hz, 2H), 5.14 (s, 2H), 2.89 (hept, J = 6.8 Hz, 1H), 1.23 (d, J = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 149.2, 132.5, 128.5, 126.6, 65.4, 33.8, 23.8.



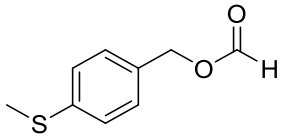
4-(*tert*-Butyl)benzyl formate, 1g[5]

0.60 g, 62% yield, pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.29 (d, J = 8.3 Hz, 2H), 7.20 (d, J = 8.1 Hz, 2H), 5.06 (s, 2H), 1.21 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6, 151.4, 132.1, 128.2, 125.4, 65.3, 34.4, 31.1.



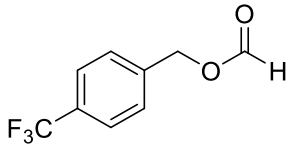
4-Methoxybenzyl formate, 1h

0.71 g, 86% yield, colourless oil. ^{1}H NMR (400 MHz, CDCl3) δ 8.08 (s, 1H), 7.29 (d, J = 8.7 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 5.11 (s, 2H), 3.78 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 159.6, 130.1, 127.2, 113.8, 65.4, 55.1. HRMS (ESI): [M+H$^+$] calcd. for $C_9H_{11}O_3^+$, 167.0703; found, 167.0713.



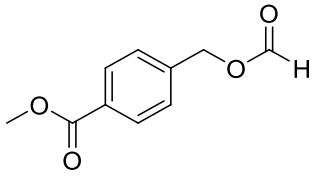
4-(Methylthio)benzyl formate, 1i[5]

0.77 g, 85% yield, pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 7.29 (d, J = 8.4 Hz, 2H), 7.24 (d, J = 8.4 Hz, 2H), 5.15 (s, 2H), 2.48 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6, 139.1, 131.8, 128.9, 126.4, 65.2, 15.5.



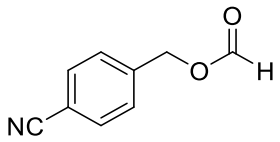
4-(Trifluoromethyl)benzyl formate, 1j[6]

0.69 g, 68% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.62 (d, J = 8.2 Hz, 2H), 7.47 (d, J = 8.1 Hz, 2H), 5.24 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 139.2, 130.4 (q, J = 32.5 Hz), 128.1, 125.5 (d, J = 3.5 Hz), 123.9 (q, J = 272.1 Hz), 64.5.



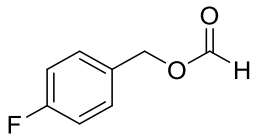
Methyl 4-((formyloxy)methyl)benzoate, 1k[5]

0.82 g, 85% yield, white solid, Mp. 67.9-70.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.94 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.4 Hz, 2H), 5.14 (s, 2H), 3.81 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.1, 160.2, 139.9, 129.7, 129.4, 127.4, 64.3, 51.7.



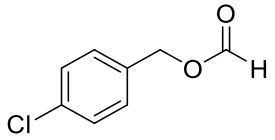
4-Cyanobenzyl formate, 1l[6]

0.56 g, 69% yield, pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.68 (d, J = 8.1 Hz, 2H), 7.49 (d, J = 8.0 Hz, 2H), 5.26 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.2, 140.3, 132.3, 128.2, 118.3, 112.1, 64.2.



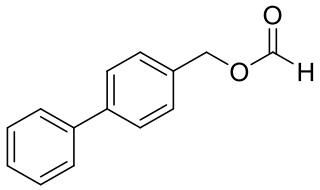
4-Fluorobenzyl formate, 1m[5]

0.50 g, 65% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.32-7.22 (m, 2H), 7.04-6.91 (m, 2H), 5.09 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7 (d, J = 247.3 Hz), 160.6, 131.1, 130.4 (d, J = 8.3 Hz), 115.6 (d, J = 21.6 Hz), 64.9.



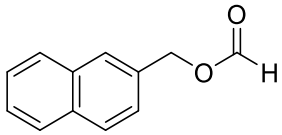
4-Chlorobenzyl formate, 1n[7]

0.79 g, 93% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (s, 1H), 7.34 (d, J = 8.4 Hz, 2H), 7.30 (d, J = 8.5 Hz, 2H), 5.16 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6, 134.4, 133.6, 129.7, 128.8, 64.8.



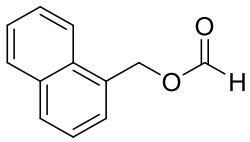
[1,1'-Biphenyl]-4-ylmethyl formate, 1o[5]

0.62 g, 58% yield, white solid, Mp. 56.0-57.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.58-7.29 (m, 9H), 5.19 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 141.5, 140.5, 134.1, 128.8, 128.8, 127.5, 127.4, 127.1, 65.4.



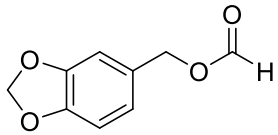
Naphthalen-2-ylmethyl formate, 1p[6]

0.80 g, 86% yield, white solid, Mp. 81.1-82.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (s, 1H), 7.86-7.78 (m, 4H), 7.52-7.41 (m, 3H), 5.34 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 133.1, 132.6, 128.4, 127.9, 127.7, 127.5, 126.4, 125.7, 65.8.



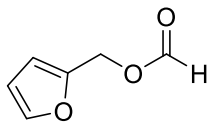
Naphthalen-1-ylmethyl formate, 1q[6]

0.82 g, 88% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.96-7.33 (m, 7H), 5.57 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6, 133.6, 131.4, 130.6, 129.4, 128.6, 127.5, 126.5, 125.9, 125.1, 123.2, 63.7.



Benzo[*d*][1,3]dioxol-5-ylmethyl formate, 1r[5]

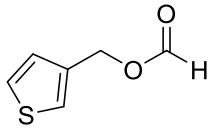
0.68 g, 75% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 6.82 (d, J = 9.0 Hz, 2H), 6.76 (d, J = 7.6 Hz, 1H), 5.92 (s, 2H), 5.06 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.5, 147.5, 147.5, 128.7, 122.1, 108.7, 107.9, 100.9, 65.2.



Furan-2-ylmethyl formate, 1s[8]

0.51 g, 81% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.43 (s, 1H), 6.41 (d, J = 26.1 Hz,

2H), 5.15 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 148.7, 143.4, 111.0, 110.6, 57.3.

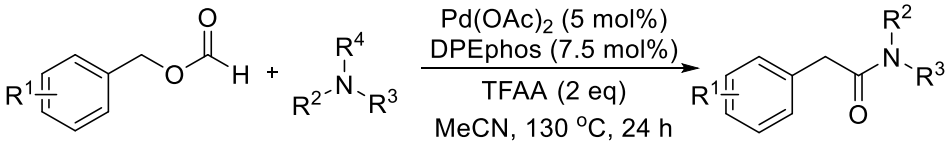


Thiophen-3-ylmethyl formate, 1t

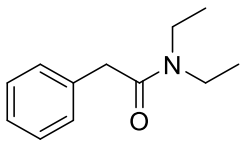
0.53 g, 75% yield, colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.33-7.25 (m, 2H), 7.11-7.03 (m, 1H), 5.18 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.5, 135.9, 127.4, 126.3, 124.6, 60.4. HRMS (ESI): [M+Na^+] calcd. for $C_6H_6NaO_2S^+$, 164.9981; found, 164.9967.

4. General Procedure for the Syntheses of the Arylacetamides

Under nitrogen, $Pd(OAc)_2$ (5 mol%), DPEphos (7.5 mol%) was added to a 15 mL tube. After refilled the tube with nitrogen, benzyl formates (1.0 mmol), tertiary amines (5.0 mmol), TFAA (2.0 equiv.) and CH_3CN (2 mL) were added by a syringe. Then the reaction mixture was stirred at 130 °C for 24 h. After the reaction was completed, the reaction mixture was concentrated by rotary evaporation. The crude mixture was purified by silica gel column chromatography (PE/1,4-dioxane = 9/1) to provide the desired products.

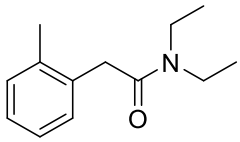


5. Characterization of the Arylacetamides



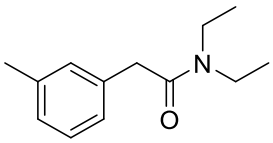
***N,N*-Diethyl-2-phenylacetamide, 3aa**[9]

143.3 mg, 75% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.20 (m, 5H), 3.69 (s, 2H), 3.38 (q, J = 7.1 Hz, 2H), 3.29 (q, J = 7.1 Hz, 2H), 1.10 (dt, J = 17.4, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 135.3, 128.5, 128.4, 126.5, 42.2, 40.7, 40.0, 14.0, 12.7.



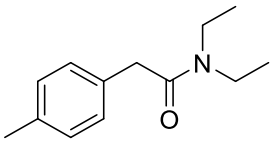
***N,N*-Diethyl-2-(*o*-tolyl)acetamide, 3ba**[10]

176.3 mg, 86% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18-7.11 (m, 4H), 3.65 (s, 2H), 3.42 (q, J = 7.1 Hz, 2H), 3.28 (q, J = 7.1 Hz, 2H), 2.27 (s, 3H), 1.15 (dd, J = 15.6, 7.2 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 136.3, 134.1, 130.2, 128.7, 126.8, 126.1, 42.3, 40.2, 38.4, 19.6, 14.2, 13.0.



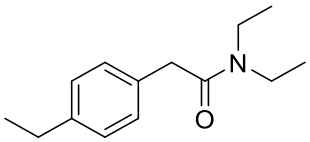
***N,N*-Diethyl-2-(*m*-tolyl)acetamide, 3ca**[10]

159.9 mg, 78% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (t, J = 7.5 Hz, 1H), 7.09-7.01 (m, 3H), 3.66 (s, 2H), 3.39 (q, J = 7.1 Hz, 2H), 3.29 (q, J = 7.1 Hz, 2H), 2.33 (s, 3H), 1.11 (dt, J = 14.1, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 138.2, 135.3, 129.4, 128.4, 127.4, 125.6, 42.3, 40.8, 40.1, 21.3, 14.2, 12.9.



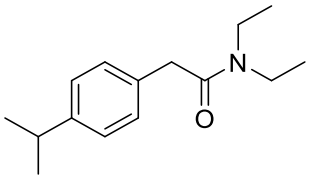
***N,N*-Diethyl-2-(*p*-tolyl)acetamide, 3da**[10]

153.8 mg, 75% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (q, J = 8.2 Hz, 4H), 3.65 (s, 2H), 3.38 (q, J = 7.1 Hz, 2H), 3.28 (q, J = 7.1 Hz, 2H), 2.32 (s, 3H), 1.10 (dt, J = 12.6, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 136.1, 132.3, 129.2, 128.4, 42.2, 40.5, 40.0, 21.0, 14.1, 12.9.



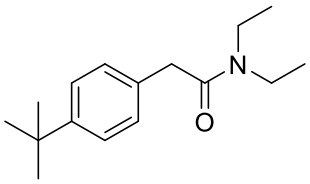
***N,N*-Diethyl-2-(4-ethylphenyl)acetamide, 3ea**

175.2 mg, 80% yield, pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18-7.12 (m, 4H), 3.66 (s, 2H), 3.38 (q, J = 7.1 Hz, 2H), 3.29 (q, J = 7.1 Hz, 2H), 2.62 (q, J = 7.6 Hz, 2H), 1.22 (t, J = 7.6 Hz, 3H), 1.10 (dt, J = 12.7, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 142.5, 132.6, 128.5, 128.1, 42.3, 40.5, 40.1, 28.4, 15.5, 14.2, 12.9. HRMS (ESI): [M+H$^+$] calcd. for $C_{14}H_{22}NO^+$, 220.1696; found, 220.1705.



***N,N*-Diethyl-2-(4-isopropylphenyl)acetamide, 3fa**

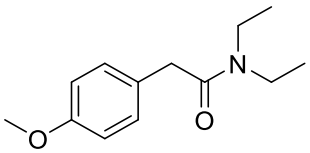
202.7 mg, 87% yield, pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (s, 4H), 3.66 (s, 2H), 3.38 (q, J = 7.1 Hz, 2H), 3.29 (q, J = 7.1 Hz, 2H), 2.87 (hept, J = 6.9 Hz, 1H), 1.23 (d, J = 6.9 Hz, 6H), 1.10 (dt, J = 14.6, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.3, 147.2, 132.7, 128.5, 126.6, 42.3, 40.4, 40.1, 33.7, 24.0, 14.2, 12.9. HRMS (ESI): [M+H$^+$] calcd. for $C_{15}H_{24}NO^+$, 234.1852; found, 234.1866.



2-(4-(*tert*-Butyl)phenyl)-*N,N*-diethylacetamide, 3ga[11]

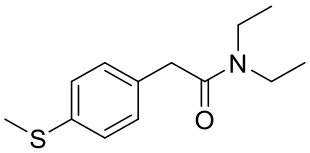
197.6 mg, 80% yield, brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, J = 8.2 Hz, 2H), 7.18 (d, J = 8.1 Hz, 2H), 3.66 (s, 2H), 3.39 (q, J = 7.1 Hz, 2H), 3.31 (q, J = 7.1 Hz, 2H), 1.30 (s, 9H), 1.12 (dt, J = 11.2, 7.1 Hz, 6H). ^{13}C

NMR (101 MHz, $CDCl_3$) δ 170.3, 149.4, 132.4, 128.3, 125.5, 42.3, 40.2, 40.1, 34.4, 31.3, 14.2, 12.9.



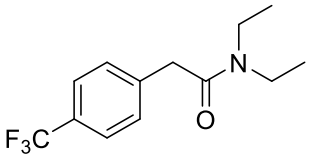
***N,N*-Diethyl-2-(4-methoxyphenyl)acetamide, 3ha**[12]

179.0 mg, 81% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, J = 8.6 Hz, 2H), 6.85 (d, J = 8.7 Hz, 2H), 3.79 (s, 3H), 3.63 (s, 2H), 3.38 (q, J = 7.1 Hz, 2H), 3.30 (q, J = 7.1 Hz, 2H), 1.10 (dt, J = 10.4, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 158.4, 129.6, 127.5, 114.0, 55.2, 42.3, 40.2, 39.9, 14.2, 12.9.



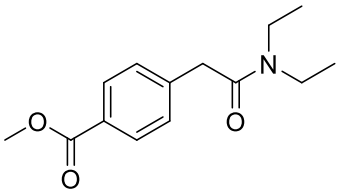
***N,N*-Diethyl-2-(4-(methylthio)phenyl)acetamide, 3ia**

177.8 mg, 75% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.16 (m, 4H), 3.65 (s, 2H), 3.39 (q, J = 7.1 Hz, 2H), 3.29 (q, J = 7.1 Hz, 2H), 2.47 (s, 3H), 1.11 (td, J = 7.1, 4.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 136.5, 132.4, 129.2, 127.1, 42.3, 40.3, 40.2, 16.1, 14.3, 12.9. HRMS (ESI): [M+H$^+$] calcd. for $C_{13}H_{20}NOS^+$, 238.1260; found, 238.1272.



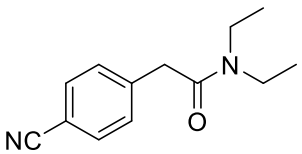
***N,N*-Diethyl-2-(4-(trifluoromethyl)phenyl)acetamide, 3ja**[11]

225.3 mg, 87% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, J = 8.1 Hz, 2H), 7.38 (d, J = 8.0 Hz, 2H), 3.74 (s, 2H), 3.40 (q, J = 7.1 Hz, 2H), 3.32 (q, J = 7.2 Hz, 2H), 1.14 (dd, J = 13.5, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 139.6, 129.2, 129.0 (d, J = 32.9 Hz), 125.4 (d, J = 3.6 Hz), 124.2 (q, J = 271.9 Hz), 42.3, 40.3, 40.2, 14.2, 12.8.



Methyl 4-(2-(diethylamino)-2-oxoethyl)benzoate, 3ka[11]

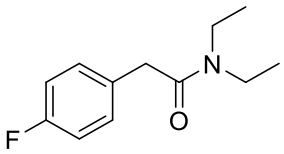
174.3 mg, 70% yield, brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 3.91 (s, 3H), 3.75 (s, 2H), 3.40 (q, J = 7.1 Hz, 2H), 3.30 (q, J = 7.1 Hz, 2H), 1.12 (q, J = 7.3 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 166.9, 140.8, 129.9, 128.8, 128.6, 52.0, 42.4, 40.8, 40.2, 14.2, 12.9.



2-(4-Cyanophenyl)-*N,N*-diethylacetamide, 3la[11]

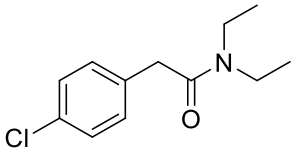
183.6 mg, 85% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, J = 8.3 Hz, 2H), 7.38 (d, J = 8.2 Hz, 2H),

3.74 (s, 2H), 3.40 (q, *J* = 7.1 Hz, 2H), 3.32 (q, *J* = 7.2 Hz, 2H), 1.15 (dt, *J* = 12.4, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 141.0, 132.2, 129.8, 118.8, 110.6, 42.4, 40.4, 40.3, 14.3, 12.8.



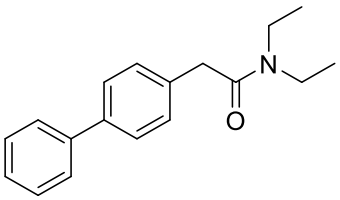
***N,N*-Diethyl-2-(4-fluorophenyl)acetamide, 3ma**[10]

146.3 mg, 70% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (dd, *J* = 8.6, 5.4 Hz, 2H), 7.06−7.00 (m, 2H), 3.69 (s, 2H), 3.42 (q, *J* = 7.1 Hz, 2H), 3.34 (q, *J* = 7.1 Hz, 2H), 1.15 (td, *J* = 7.1, 2.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 161.7 (d, *J* = 244.9 Hz), 131.1 (d, *J* = 2.8 Hz), 130.2 (d, *J* = 7.9 Hz), 115.4 (d, *J* = 21.3 Hz), 42.3, 40.2, 39.8, 14.2, 12.9.



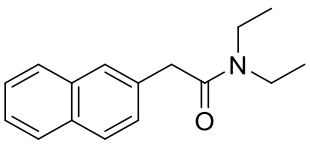
2-(4-Chlorophenyl)-*N,N*-diethylacetamide, 3na[11]

162.0 mg, 72% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 3.65 (s, 2H), 3.39 (q, *J* = 7.1 Hz, 2H), 3.30 (q, *J* = 7.1 Hz, 2H), 1.12 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 134.0, 132.6, 130.1, 128.7, 42.3, 40.2, 40.0, 14.3, 12.9.



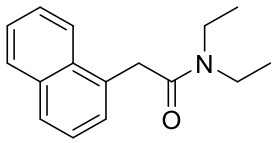
2-([1,1'-Biphenyl]-4-yl)-*N,N*-diethylacetamide, 3oa[13]

181.6 mg, 68% yield, brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.53 (m, 4H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.37-7.31 (m, 3H), 3.74 (s, 2H), 3.41 (q, *J* = 7.1 Hz, 2H), 3.34 (q, *J* = 7.2 Hz, 2H), 1.14 (td, *J* = 7.1, 4.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 140.7, 139.5, 134.4, 129.0, 128.6, 127.2, 127.1, 126.9, 42.3, 40.3, 40.2, 14.1, 12.8.



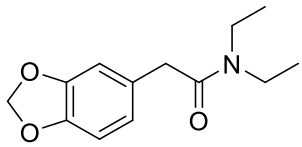
***N,N*-Diethyl-2-(naphthalen-2-yl)acetamide, 3pa**[12]

209.7 mg, 87% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.74 (m, 3H), 7.69 (s, 1H), 7.49-7.38 (m, 3H), 3.86 (s, 2H), 3.42 (q, *J* = 7.1 Hz, 2H), 3.33 (q, *J* = 7.1 Hz, 2H), 1.12 (dt, *J* = 14.0, 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 133.6, 133.0, 132.3, 128.2, 127.6, 127.6, 127.0, 126.0, 125.6, 42.4, 41.1, 40.2, 14.2, 12.9.



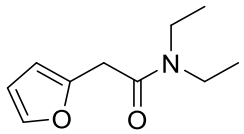
***N,N*-Diethyl-2-(naphthalen-1-yl)acetamide, 3qa**

188.0 mg, 78% yield, brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 8.3 Hz, 1H), 7.88-7.84 (m, 1H), 7.77 (d, J = 8.2 Hz, 1H), 7.55-7.46 (m, 2H), 7.45-7.39 (m, 1H), 7.33 (d, J = 6.9 Hz, 1H), 4.12 (s, 2H), 3.45 (q, J = 7.1 Hz, 2H), 3.32 (q, J = 7.1 Hz, 2H), 1.18 (t, J = 7.1 Hz, 3H), 1.12 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 133.8, 132.1, 131.8, 128.8, 127.5, 126.2, 126.1, 125.7, 125.5, 123.4, 42.4, 40.2, 38.3, 14.2, 13.0. HRMS (ESI): [M+H$^+$] calcd. for $C_{16}H_{20}NO^+$, 242.1539; found, 242.1550.



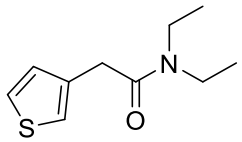
2-(Benzo[*d*][1,3]dioxol-5-yl)-*N,N*-diethylacetamide, 3ra

173.9 mg, 74% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79-6.65 (m, 3H), 5.93 (s, 2H), 3.60 (s, 2H), 3.38 (q, J = 7.1 Hz, 2H), 3.30 (q, J = 7.1 Hz, 2H), 1.12 (td, J = 7.1, 2.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 147.8, 146.3, 129.1, 121.7, 109.2, 108.2, 100.9, 42.3, 40.4, 40.2, 14.2, 12.9. HRMS (ESI): [M+H$^+$] calcd. for $C_{13}H_{18}NO_3^+$, 236.1281; found, 236.1291.



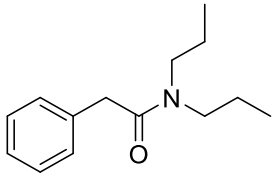
***N,N*-Diethyl-2-(furan-2-yl)acetamide, 3sa**

153.9 mg, 85% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, J = 1.3 Hz, 1H), 6.34-6.31 (m, 1H), 6.19 (d, J = 3.1 Hz, 1H), 3.72 (s, 2H), 3.38 (dq, J = 11.9, 7.1 Hz, 4H), 1.14 (dd, J = 13.4, 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.8, 149.3, 141.6, 110.5, 107.4, 42.4, 40.3, 33.9, 14.2, 12.9. HRMS (ESI): [M+H$^+$] calcd. for $C_{10}H_{16}NO_2^+$, 182.1176; found, 182.1185.



***N,N*-Diethyl-2-(thiophen-3-yl)acetamide, 3ta**[11]

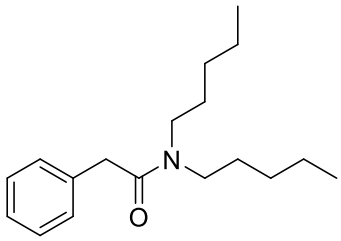
159.6 mg, 81% yield, brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (dd, J = 4.9, 2.9 Hz, 1H), 7.07 (dd, J = 2.8, 1.0 Hz, 1H), 7.03 (dd, J = 4.9, 1.0 Hz, 1H), 3.69 (s, 2H), 3.39 (q, J = 7.1 Hz, 2H), 3.31 (q, J = 7.1 Hz, 2H), 1.12 (dd, J = 15.5, 7.2 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 135.2, 128.2, 125.7, 121.7, 42.4, 40.2, 35.6, 14.2, 12.9.



2-Phenyl-*N,N*-dipropylacetamide, 3ab[14]

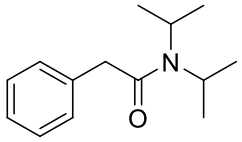
170.8 mg, 78% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.19 (m, 5H), 3.69 (s, 2H), 3.28 (t, J = 7.6

Hz, 2H), 3.20-3.14 (m, 2H), 1.60-1.46 (m, 4H), 0.89-0.81 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 135.4, 128.5, 128.4, 126.4, 49.7, 47.3, 40.8, 22.0, 20.6, 11.2, 11.0.



***N,N*-Dipentyl-2-phenylacetamide, 3ac**

195.3 mg, 71% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.28 (m, 2H), 7.27-7.22 (m, 3H), 3.69 (s, 2H), 3.35-3.27 (m, 2H), 3.22-3.16 (m, 2H), 1.58-1.42 (m, 4H), 1.37-1.17 (m, 8H), 0.90-0.86 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.5, 135.6, 128.7, 128.6, 126.6, 48.3, 45.8, 41.0, 29.2, 29.0, 28.7, 27.3, 22.5, 22.4, 14.0, 14.0. HRMS (ESI): [M+H$^+$] calcd. for $C_{18}H_{30}NO^+$, 276.2322; found, 276.2334.



***N,N*-Diisopropyl-2-phenylacetamide, 3ad**[15]

181.8 mg, 83% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.18 (m, 5H), 4.00-3.89 (m, 1H), 3.67 (s, 2H), 3.35 (s, 1H), 1.41 (dd, J = 6.7, 2.0 Hz, 6H), 0.98 (dd, J = 6.4, 2.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 135.6, 128.3, 128.2, 126.2, 49.1, 45.5, 43.2, 20.2.

6. References

1 J. M. Álvarez-Calero, Z. D. Jorge, G. M. Massanet, $TiCl_4$/Et_3N-Mediated Condensation of Acetate and Formate Esters: Direct Access to β-Alkoxy-and β-Aryloxyacrylates, *Org. Lett.*, 2016, **18**, 6344-6347.

2 F. Shirini, M. Seddighi, M. Mamaghani, Brönsted acidic ionic liquid supported on rice husk ash (RHA-[pmim]HSO_4): a highly efficient and reusable catalyst for the formylation of amines and alcohols, *RSC Adv.*, 2014, **4**, 50631-50638.

3 M. Lai, X. Qi, X.-F. Wu, Palladium-Catalyzed Carbonylative Synthesis of Benzyl Benzoates Employing Benzyl Formates as Both CO Surrogates and Benzyl Alcohol Sources, *Eur. J. Org. Chem.*, 2019, **2019**, 3776-3778.

4 S. Liang, P. Monsen, G. B. Hammond, B. Xu, Au/TiO_2 catalyzed reductive amination of aldehydes and ketones using formic acid as reductant, *Org. Chem. Front.*, 2016, **3**, 505-509.

5 X. Qi, M. Lai, X.-F. Wu, Carbonylative transformation of benzyl formates into alkyl 2-arylacetates in organic carbonates, *Org. Chem. Front.*, 2019, **6**, 3397-3400.

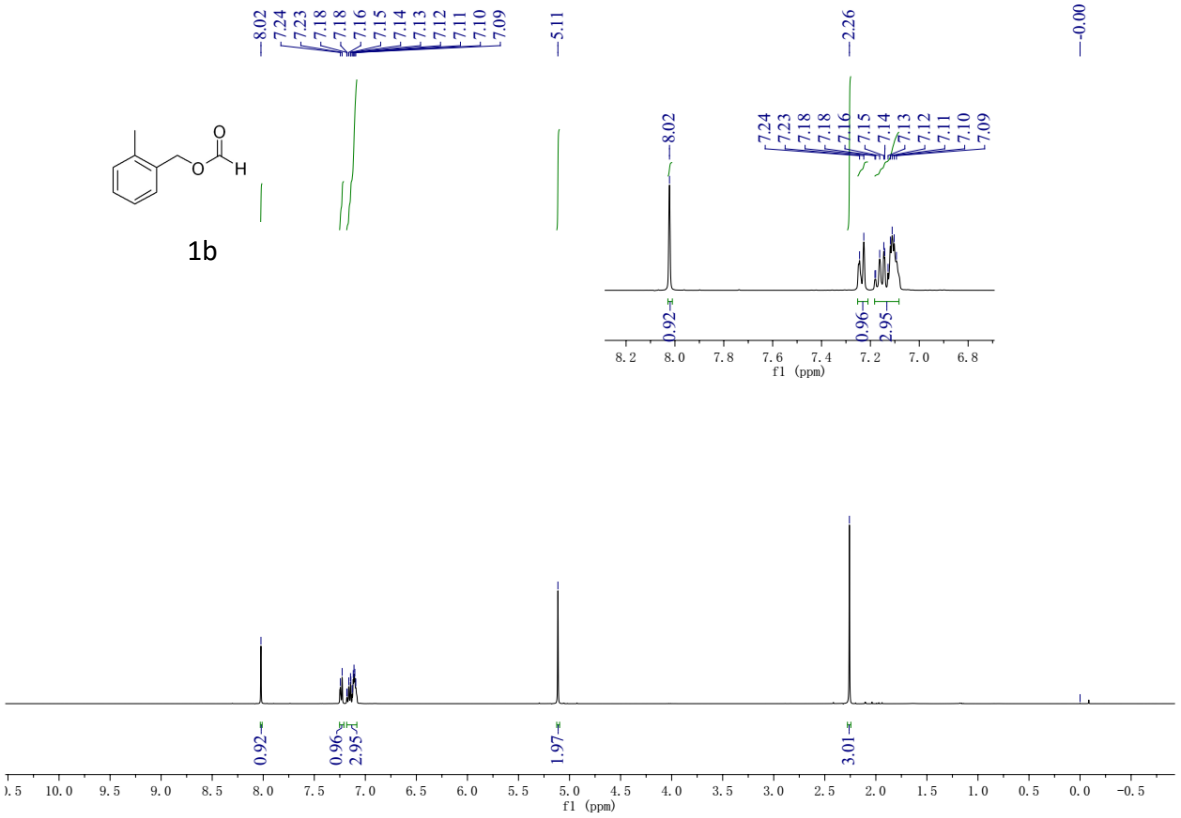
6 W. Y. Fang, G. F. Zha, H. L. Qin, Making Carbonyls of Amides Nucleophilic and Hydroxyls of Alcohols Electrophilic Mediated by SO_2F_2 for Synthesis of Esters from Amides, *Org. Lett.*, 2019, **21**, 8657-8661.

7 S. Taheri, H. Veisi, M. Hekmati, Application of polydopamine sulfamic acid-functionalized magnetic Fe_3O_4 nanoparticles (Fe_3O_4@PDA-SO_3H) as a heterogeneous and recyclable nanocatalyst for the formylation of alcohols and amines under solvent-free conditions, *New J. Chem.*, 2017, **41**, 5075-5081.

8 J. Mitra, X. Zhou, T. Rauchfuss, Pd/C-catalyzed reactions of HMF: decarbonylation, hydrogenation, and hydrogenolysis, *Green Chem.*, 2015, **17**, 307-313.

9 V. Rathore, M. Sattar, R. Kumar, S. Kumar, Synthesis of Unsymmetrical Diaryl Acetamides, Benzofurans, Benzophenones, and Xanthenes by Transition-Metal-Free Oxidative Cross-Coupling of sp^3 and sp^2 C-H Bonds, *J. Org. Chem.*, 2016, **81**, 9206-9218.
10 B. Zheng, T. Jia, P. J. Walsh, A General and Practical Palladium-Catalyzed Direct α-Arylation of Amides with Aryl Halides, *Adv. Synth. Catal.*, 2014, **356**, 165-178.
11 T. Hama, D. A. Culkin, J. F. Hartwig, Palladium-catalyzed intermolecular α-arylation of zinc amide enolates under mild conditions, *J. Am. Chem. Soc.*, 2006, **128**, 4976-4985.
12 B. Xiong, L. Zhu, X. Feng, J. Lei, T. Chen, Y. Zhou, S. F. Yin, Direct Amidation of Carboxylic Acids with Tertiary Amines: Amide Formation over Copper Catalysts through C-N Bond Cleavage, *Eur. J. Org. Chem.*, 2014, **2014**, 4244-4247.
13 S. M. Wang, C. Zhao, X. Zhang, H. L. Qin, Clickable coupling of carboxylic acids and amines at room temperature mediated by SO_2F_2: a significant breakthrough for the construction of amides and peptide linkages, *Org. Biomol. Chem.*, 2019, **17**, 4087-4101.
14 M. York, R. A. Evans, The use of polyhedral oligomeric silsesquioxane (POSS) as a soluble support for organic synthesis: A case study with a POSS-bound isocyanate scavenger reagent, *Tetrahedron Lett.*, 2010, **51**, 4677-4680.
15 T. C. Malig, D. Yu, J. E. Hein, A Revised Mechanism for the Kinugasa Reaction, *J. Am. Chem. Soc.*, 2018, **140**, 9167-9173.

7. Spectra of Benzyl Formates



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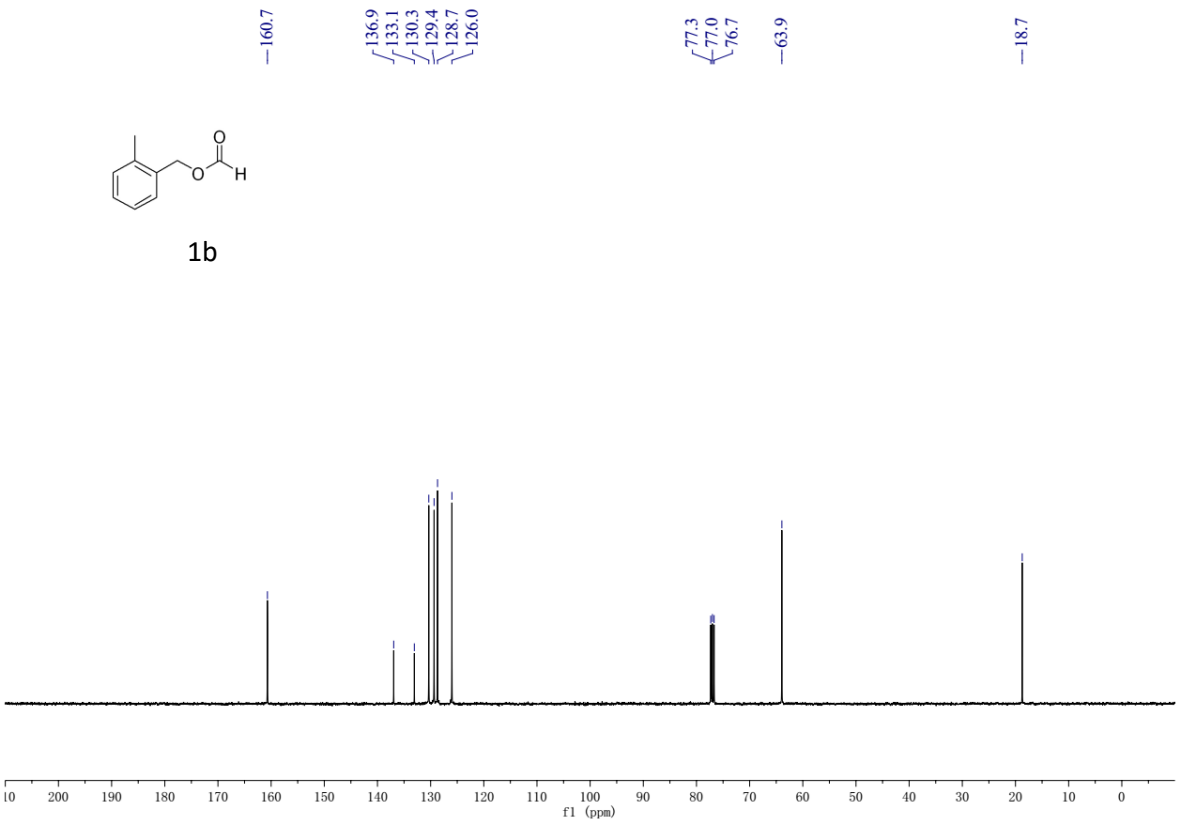

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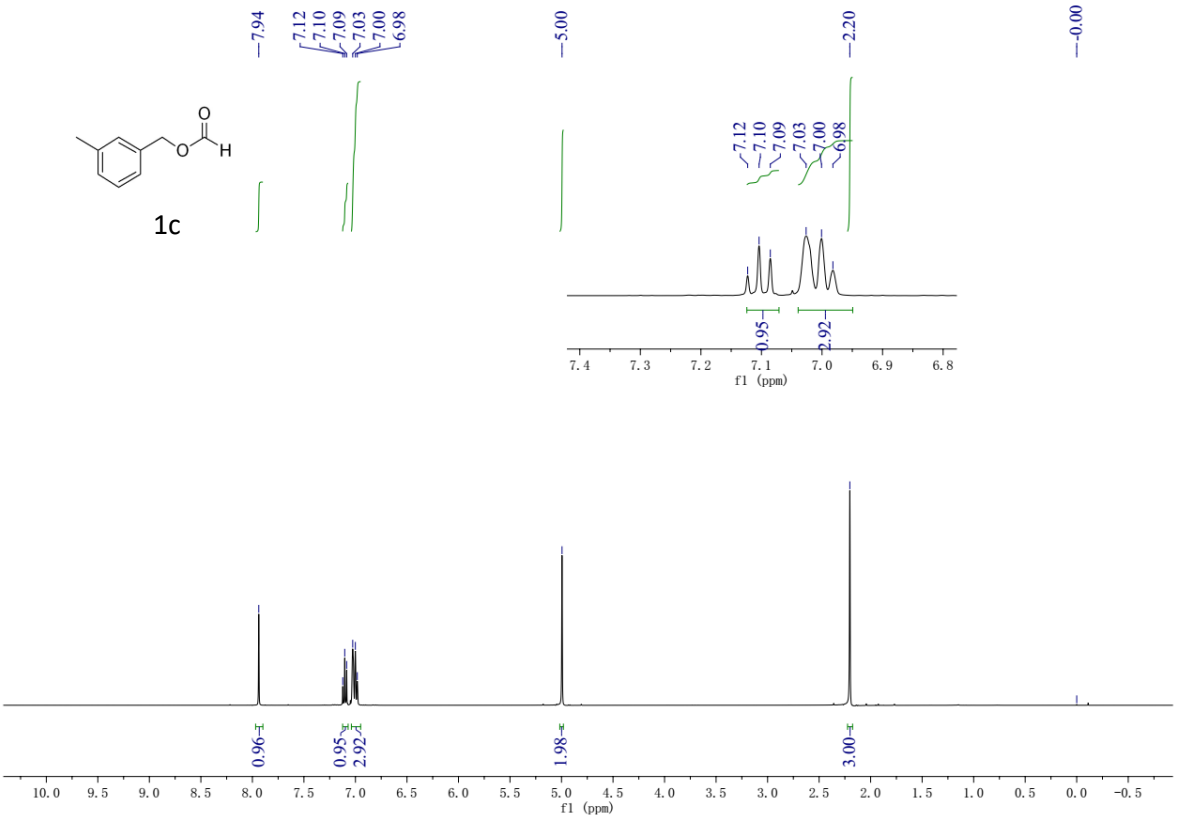
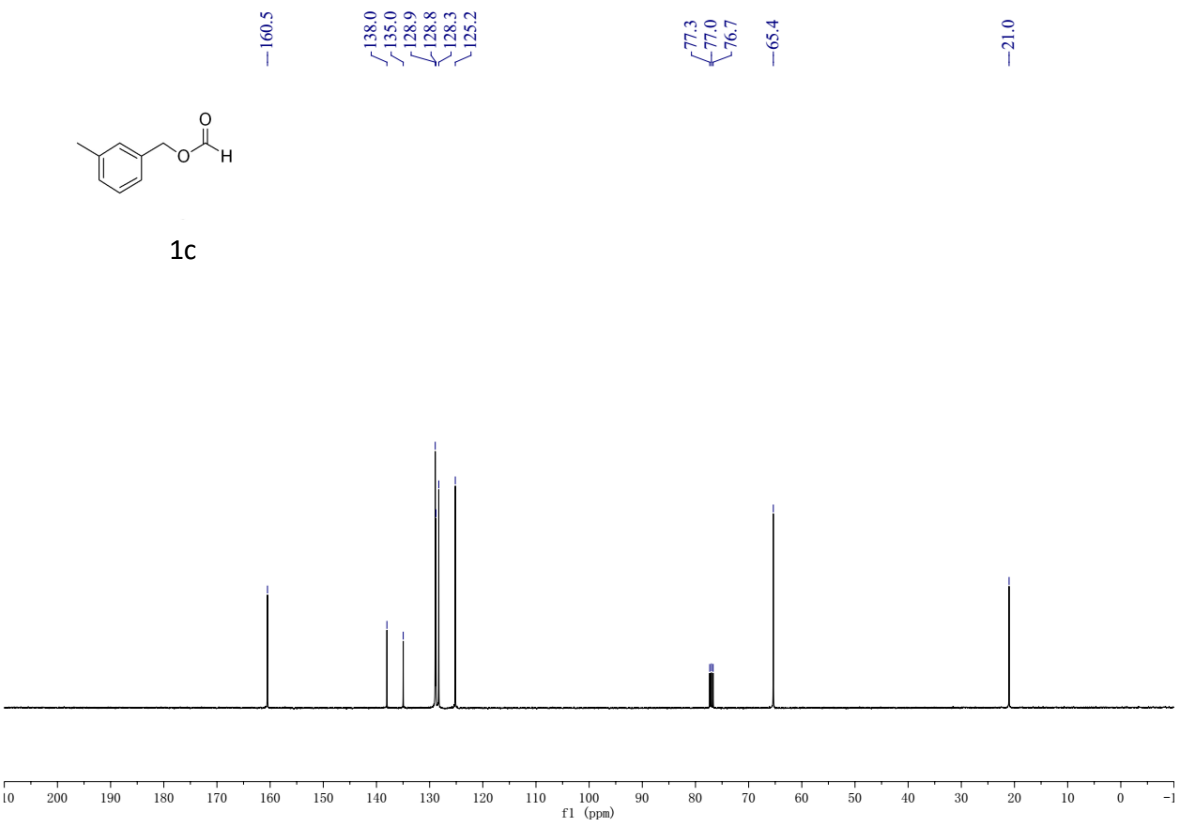
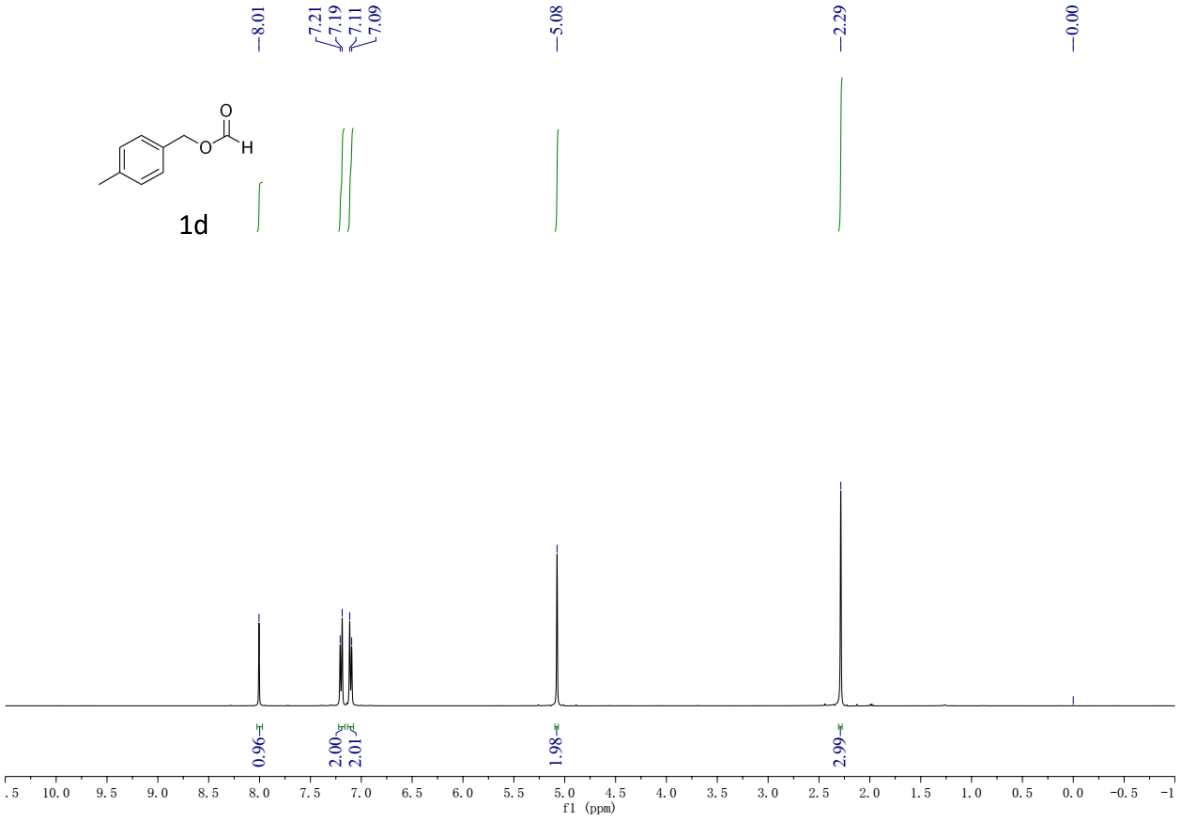
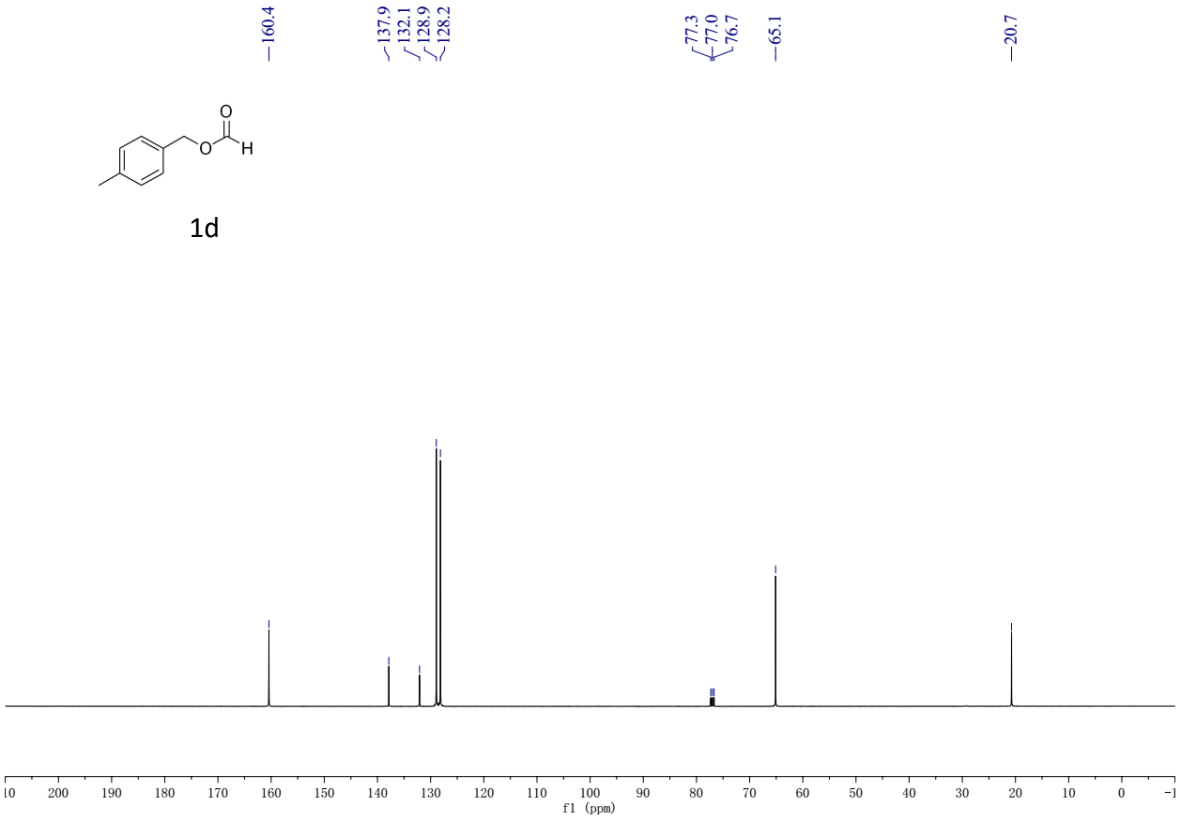

7.94
7.12
7.10
7.09
7.03
7.00
6.98
5.00
2.20
0.00
0.95
2.92
f1 (ppm)
0.96
0.95
2.92
1.98
3.00
f1 (ppm)

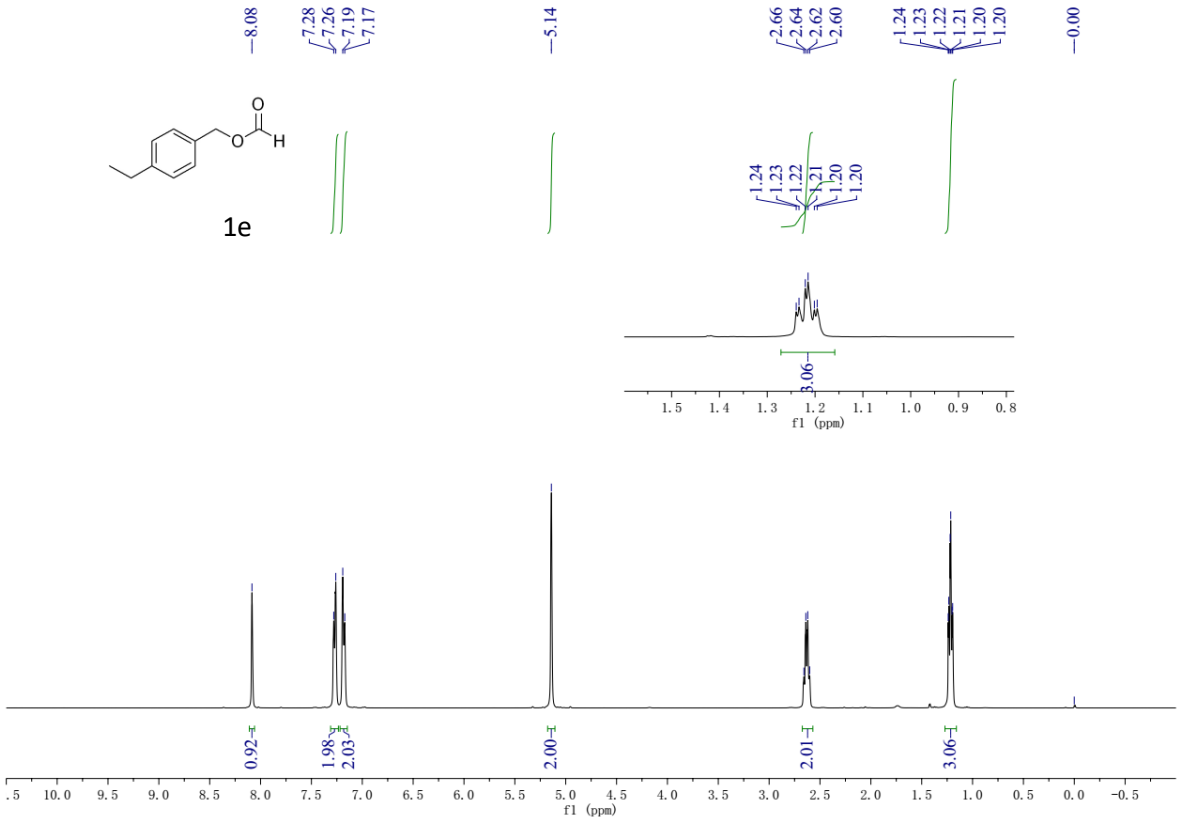
1c

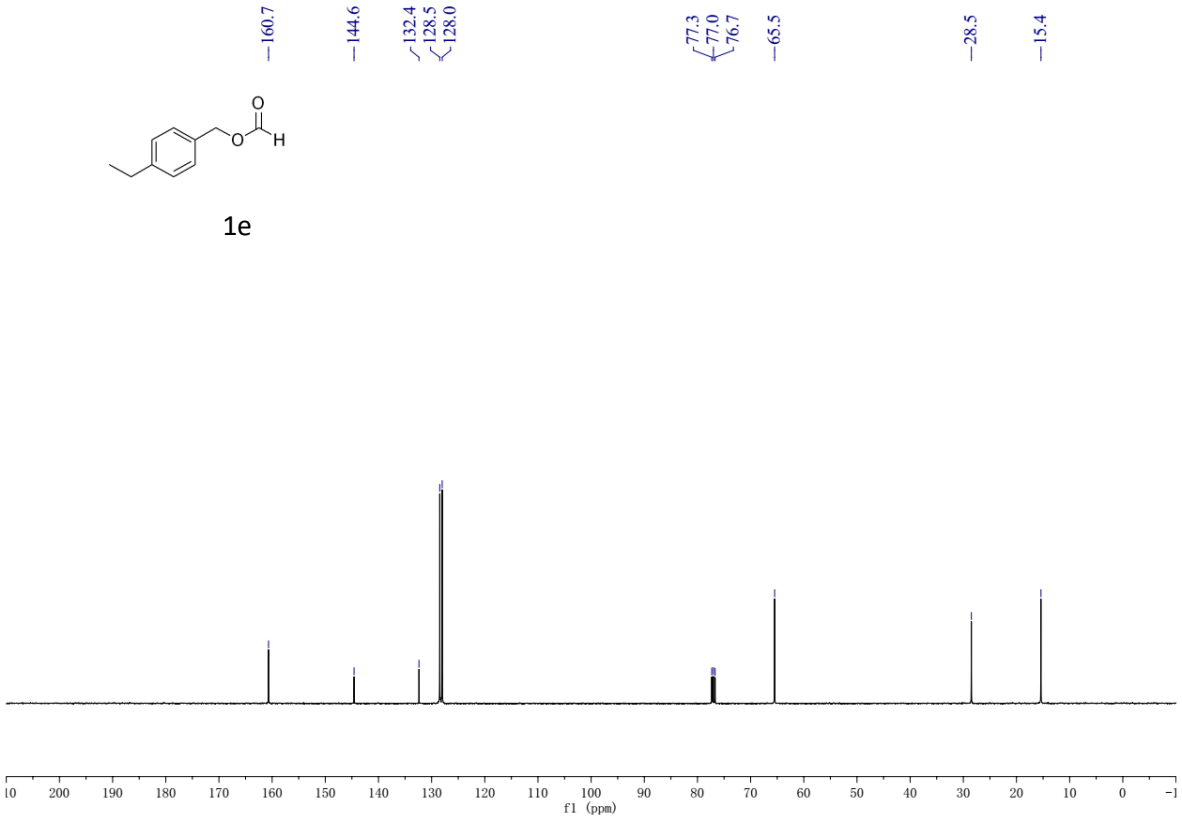

160.5
138.0
135.0
128.9
128.8
128.3
125.2
77.3
77.0
76.7
65.4
21.0
f1 (ppm)

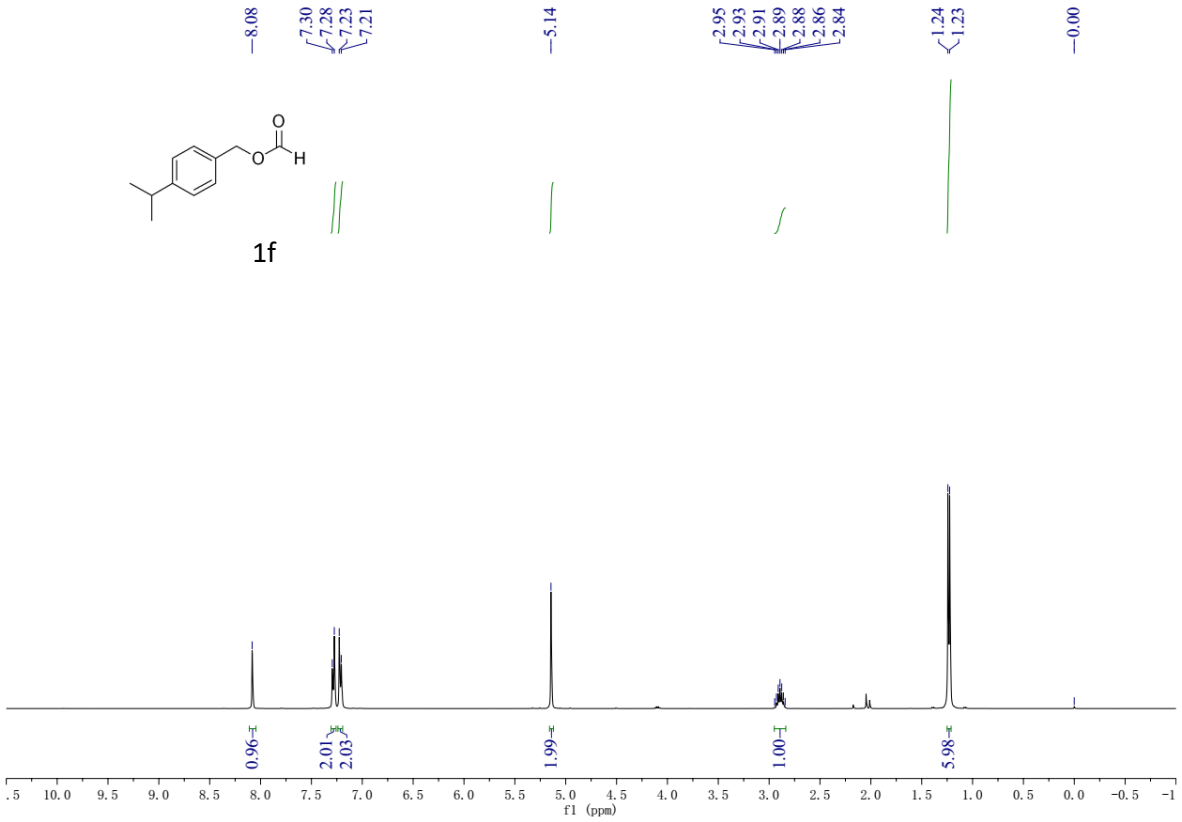
1c


8.01
7.21
7.19
7.11
7.09
5.08
2.29
0.00
O
O
H
1d
0.96
2.00
2.01
1.98
2.99
f1 (ppm)


160.4
137.9
132.1
128.9
128.2
77.3
77.0
76.7
65.1
20.7
O
O
H
1d
f1 (ppm)


8.08
7.28
7.26
7.19
7.17
5.14
2.66
2.64
2.62
2.60
1.24
1.23
1.22
1.21
1.20
1.20
0.00
O
O
H
1e
3.06
f1 (ppm)
0.92
1.98
2.03
2.00
2.01
3.06
f1 (ppm)


160.7
144.6
132.4
128.5
128.0
77.3
77.0
76.7
65.5
28.5
15.4
O
O
H
1e
f1 (ppm)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7.23
7.21
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2.93
2.91
2.89
2.88
2.86
2.84
1.24
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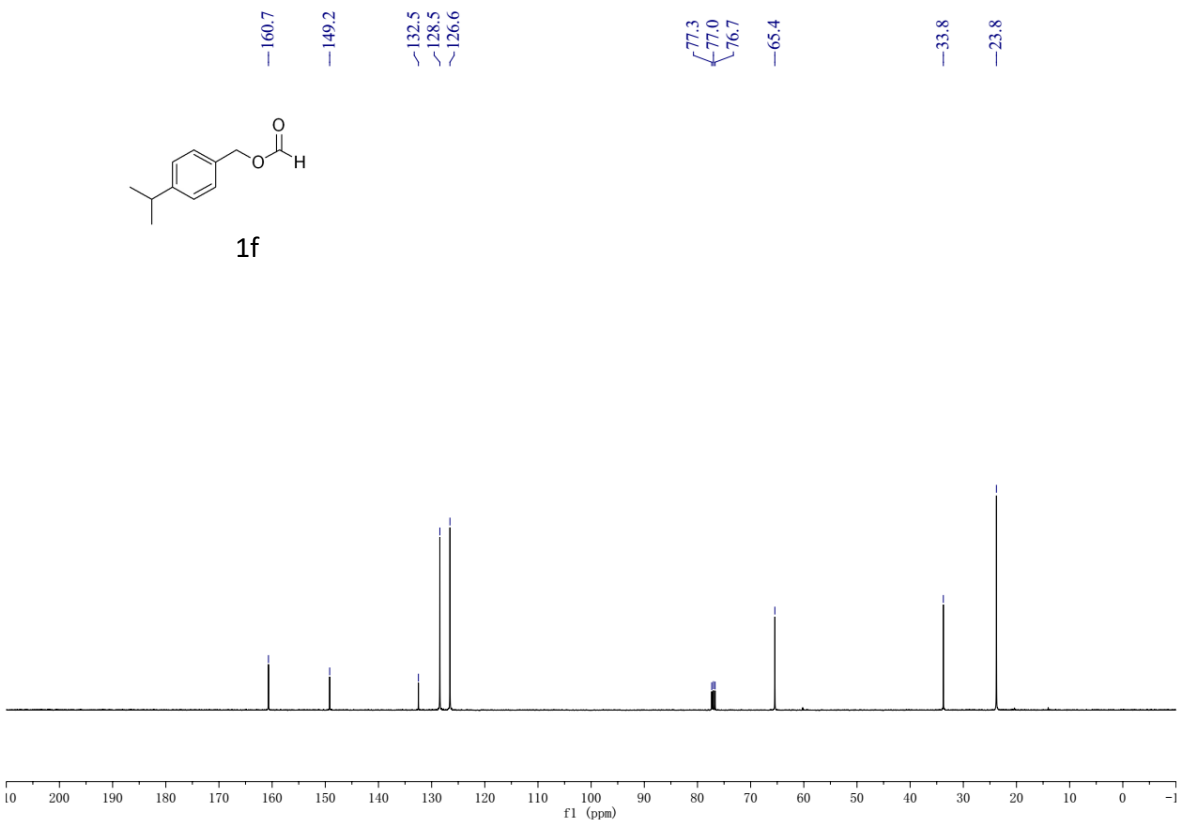
O
H
0.96
2.01
2.03
1.99
1.00
5.98
f1 (ppm)

1f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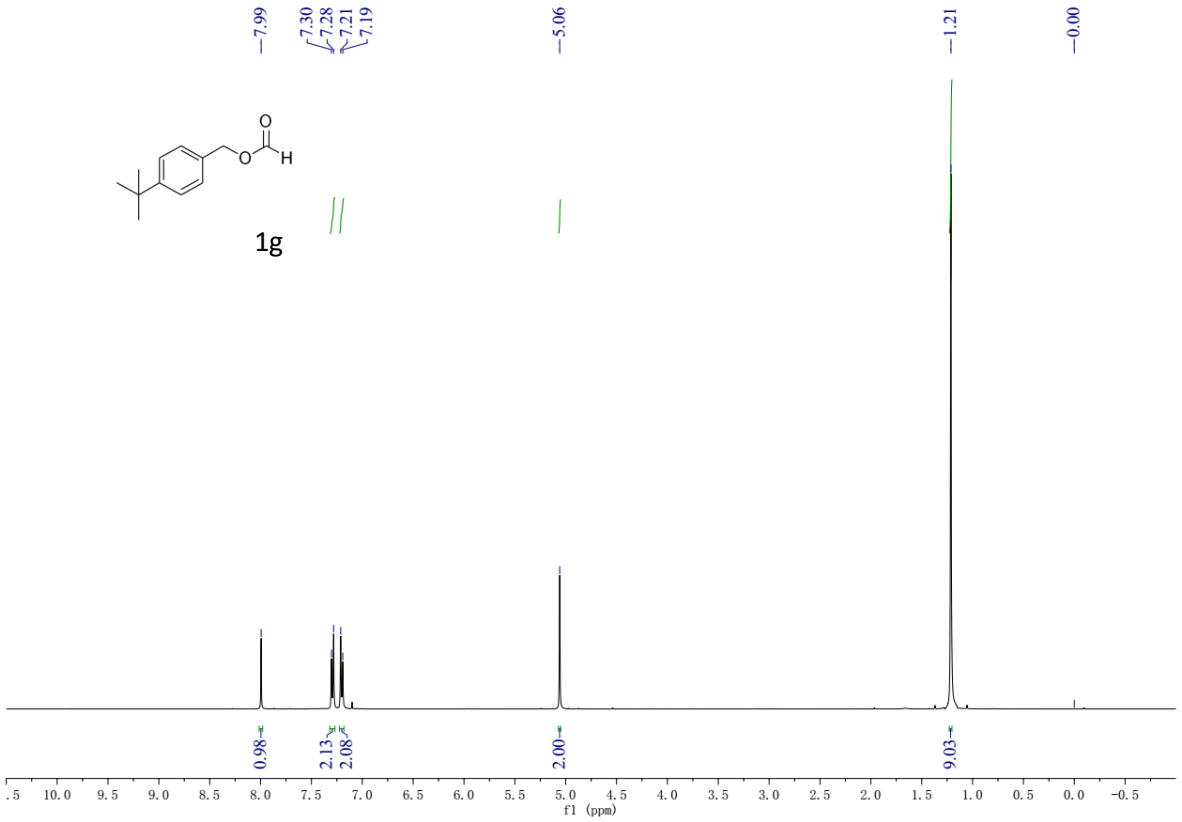
149.2
132.5
128.5
126.6
77.3
77.0
76.7
65.4
33.8
23.8
O
O
H
f1 (ppm)

1f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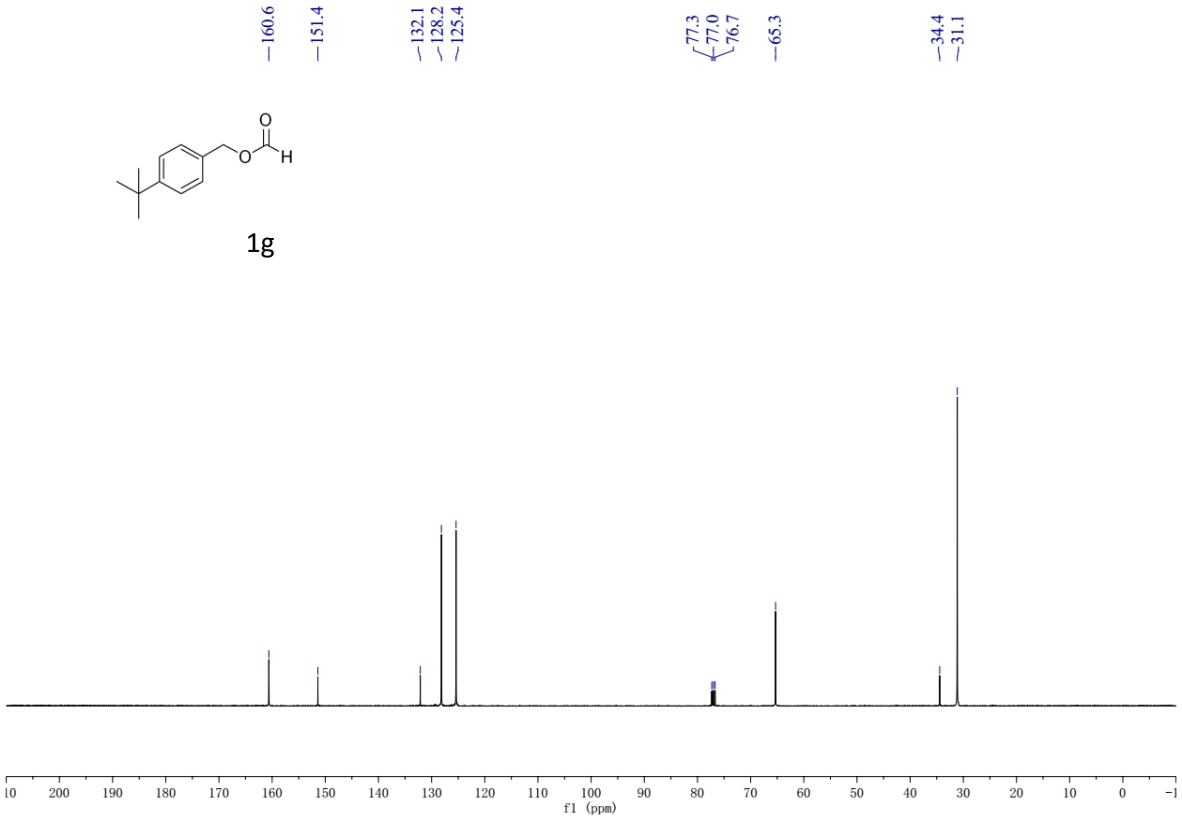
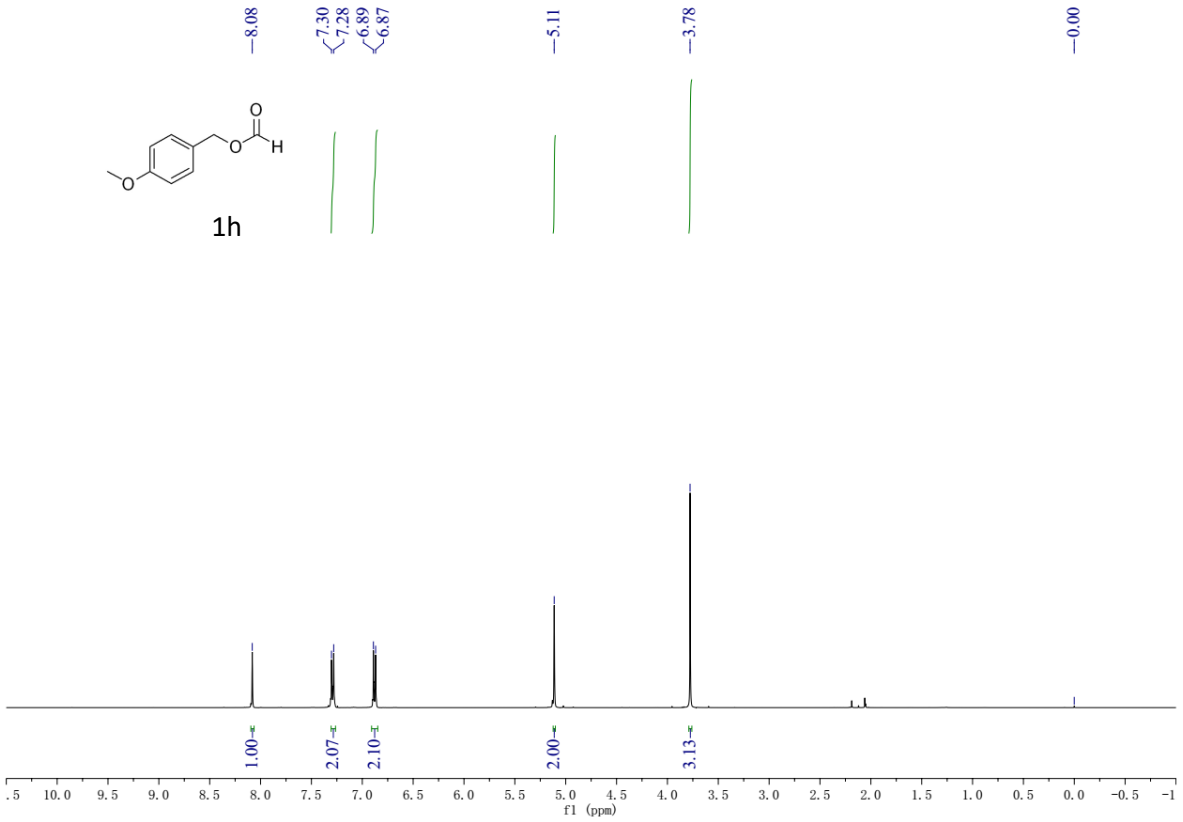
7.30
7.28
7.21
7.19
5.06
1.21
0.00
O
O
H
0.98
2.13
2.08
2.00
9.03
f1 (ppm)

1g


160.6
151.4
132.1
128.2
125.4
77.3
77.0
76.7
65.3
34.4
31.1
O
O
H
f1 (ppm)

1g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7.28
6.89
6.87
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O
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O
1h
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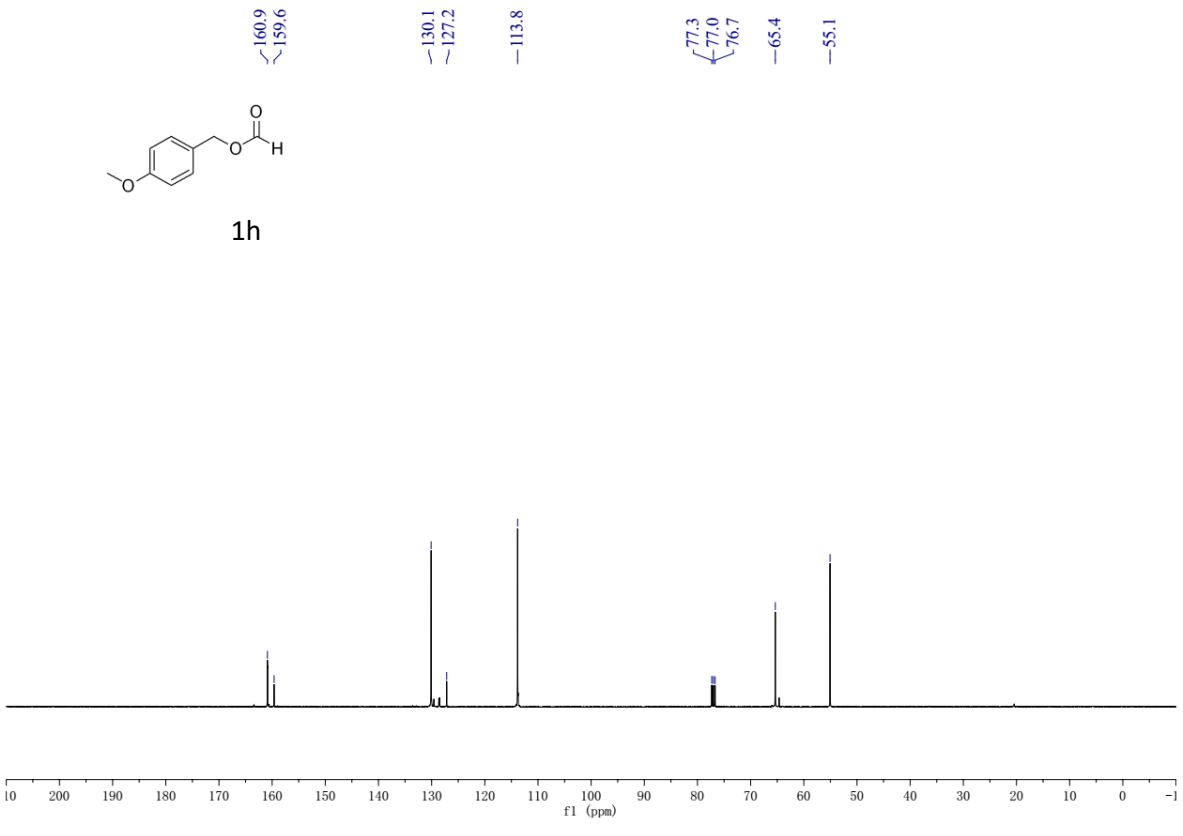
2.07
2.10
2.00
3.13
f1 (ppm)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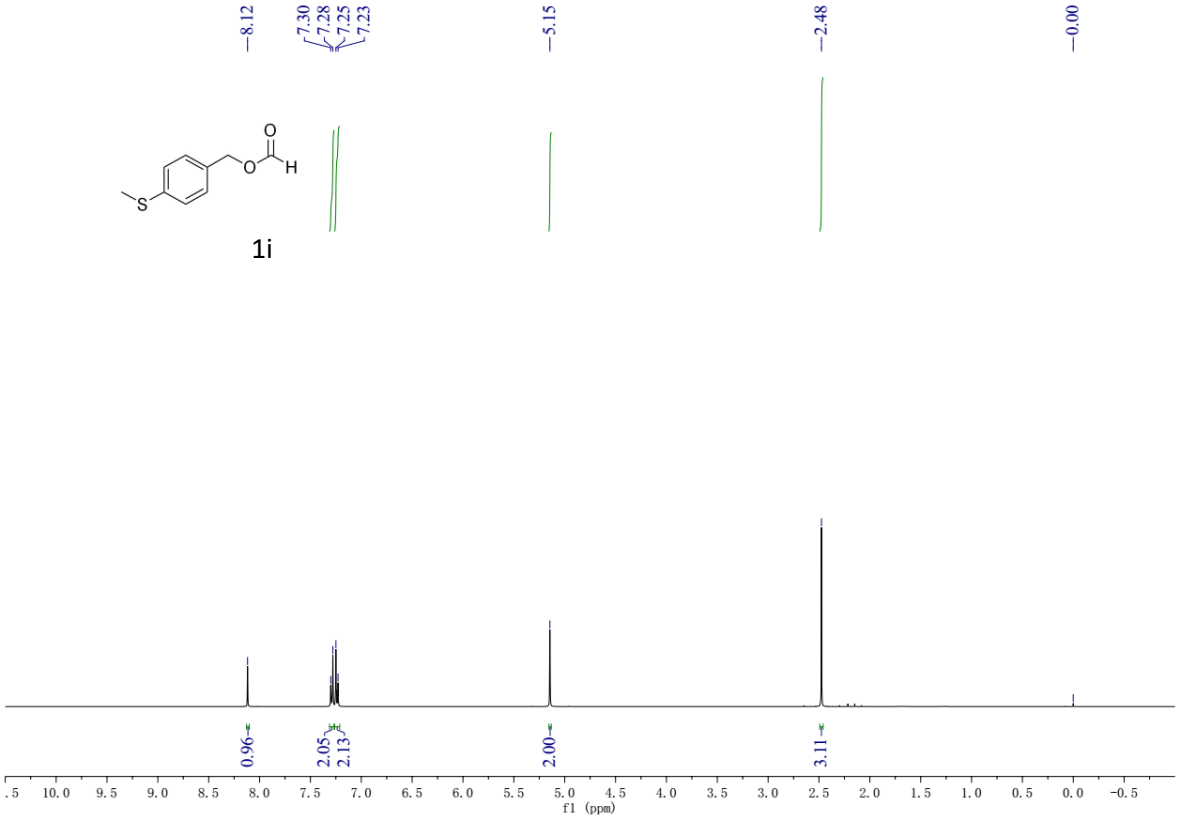
159.6
130.1
127.2
113.8
77.3
77.0
76.7
65.4
55.1
O
O
H
O
1h
f1 (ppm)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7.30
7.28
7.25
7.23
5.15
2.48
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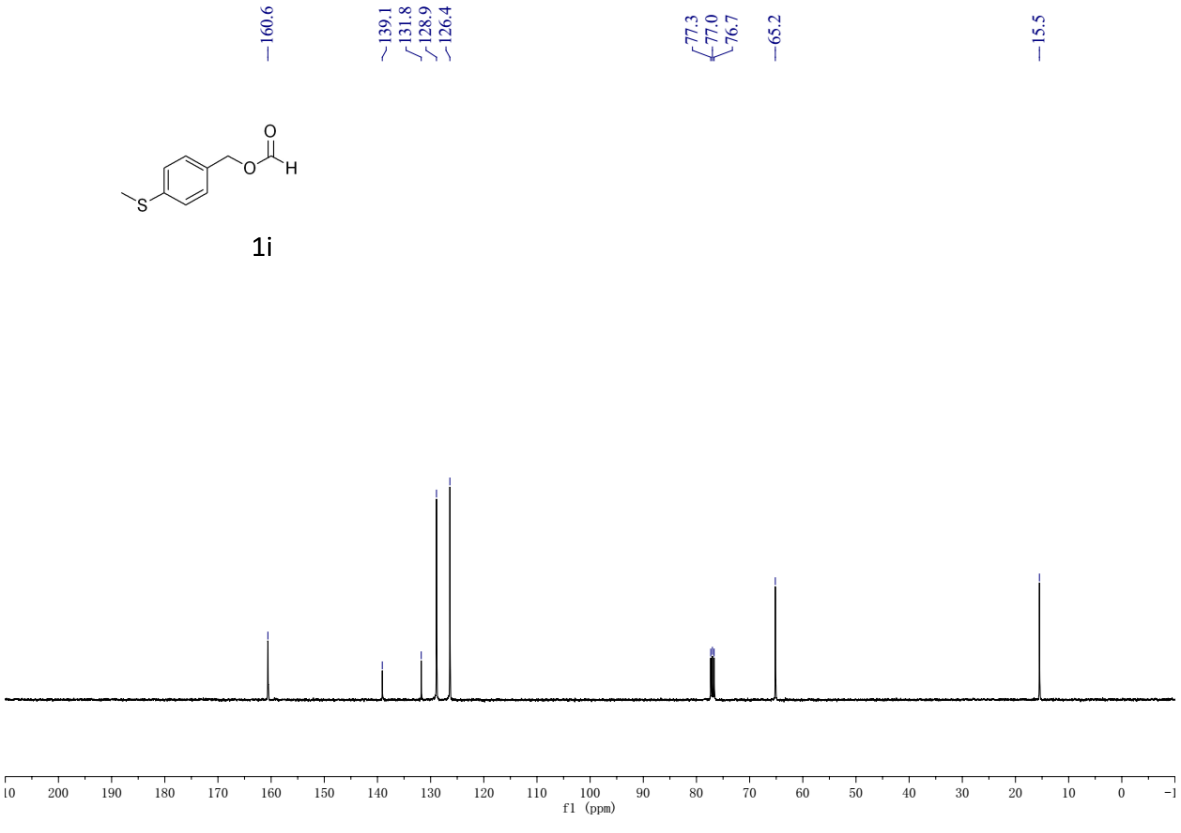
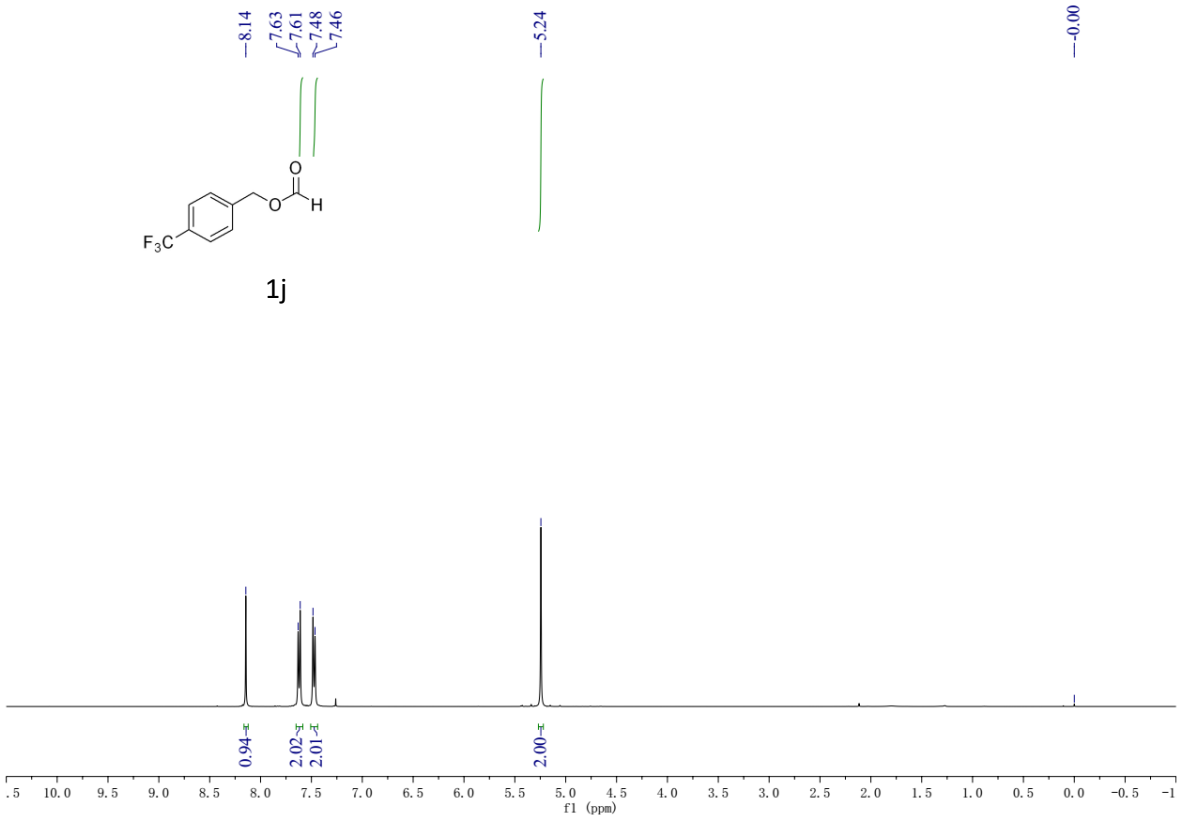
O
O
H
S
0.96
2.05
2.13
2.00
3.11
f1 (ppm)

1i


160.6
139.1
131.8
128.9
126.4
77.3
77.0
76.7
65.2
15.5
O
O
H
S
f1 (ppm)

1i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7.63
7.61
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7.46
5.24
0.00
O
O
H
F3C
0.94
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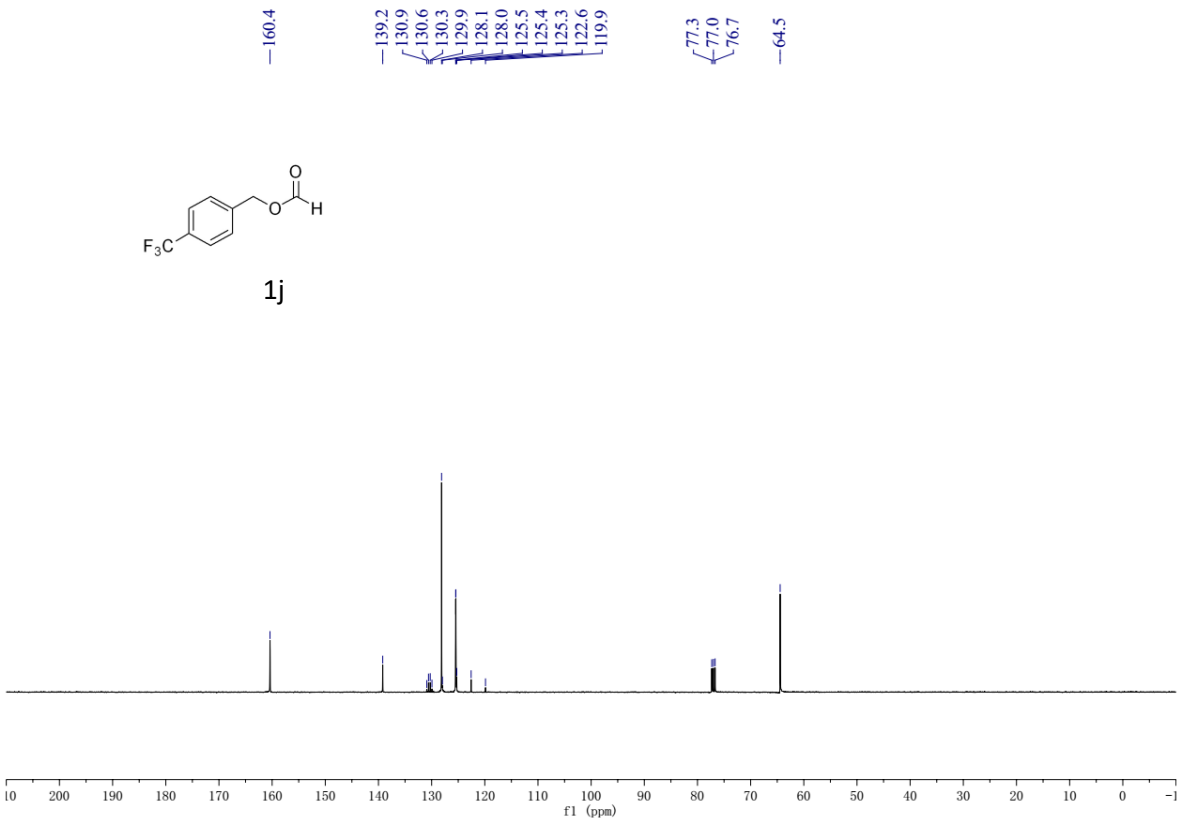
2.01
2.00
f1 (ppm)

1j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130.9
130.6
130.3
129.9
128.1
128.0
125.5
125.4
125.3
122.6
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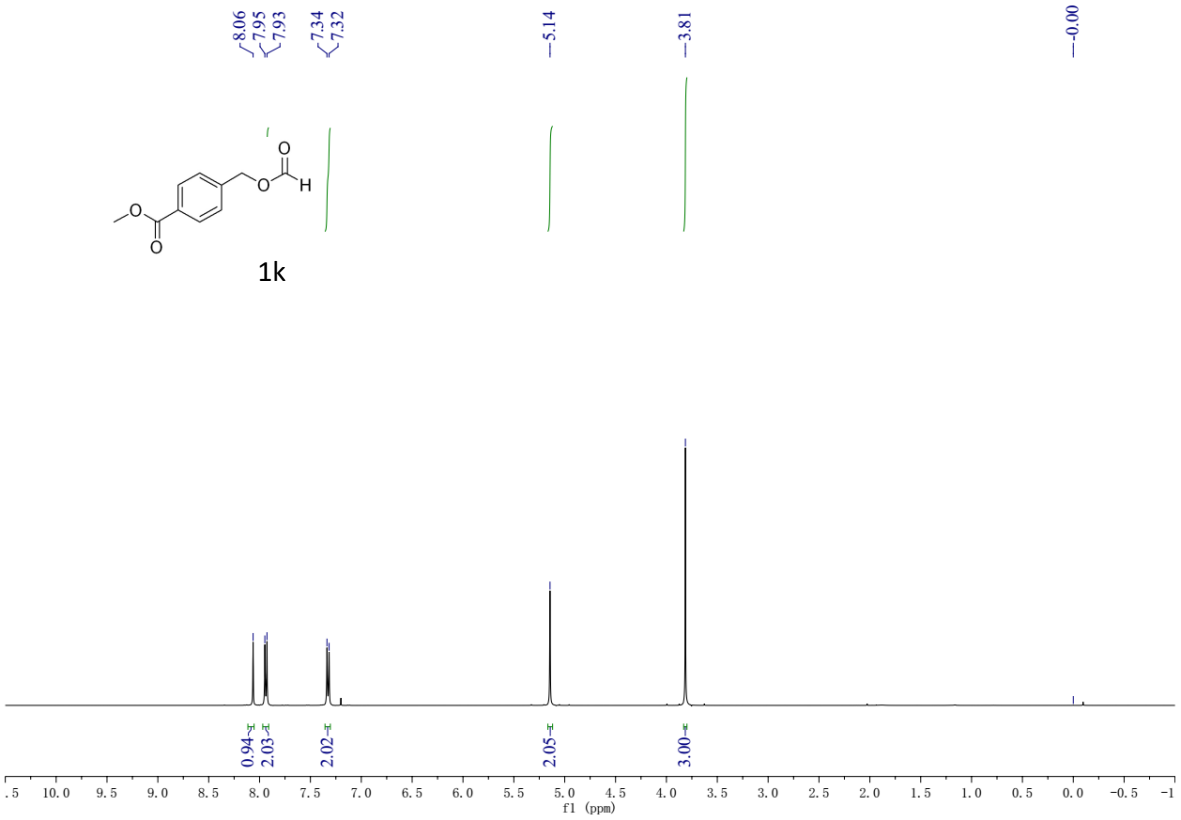
64.5
O
O
H
F3C
f1 (ppm)

1j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0.94
2.03
2.02
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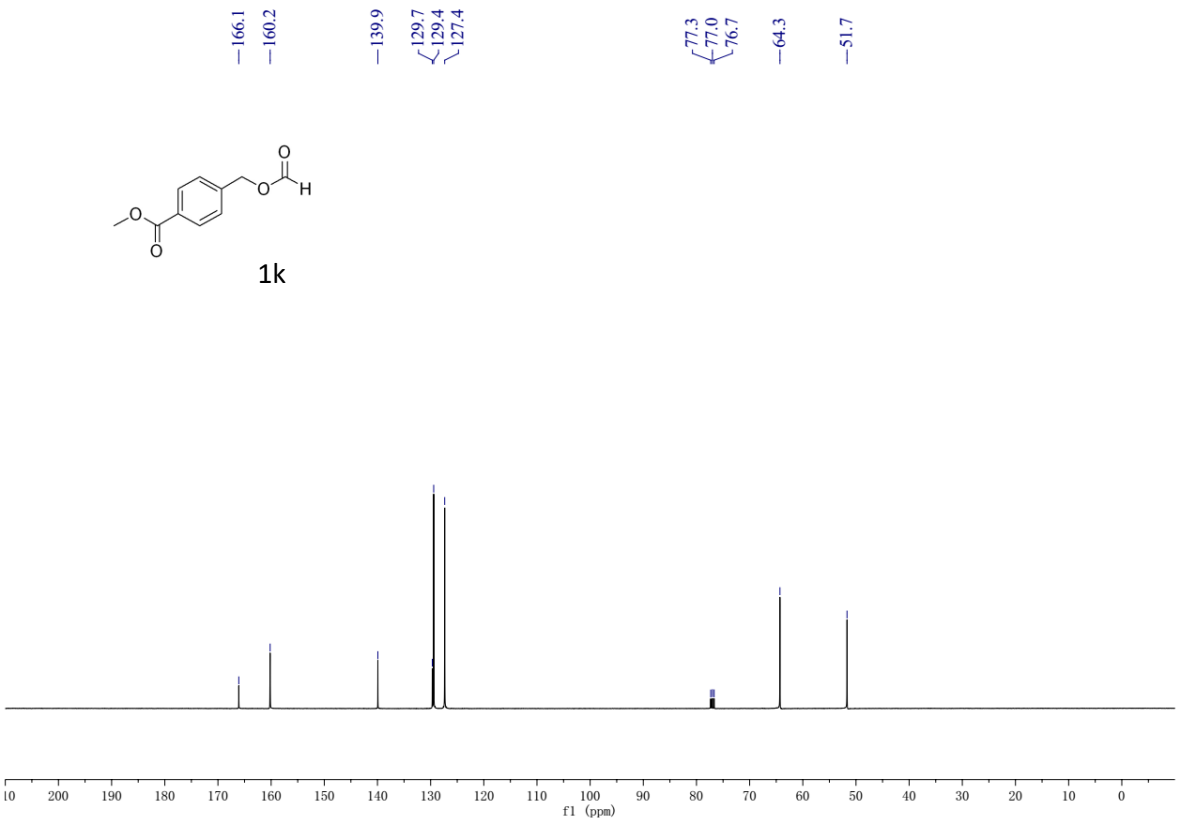
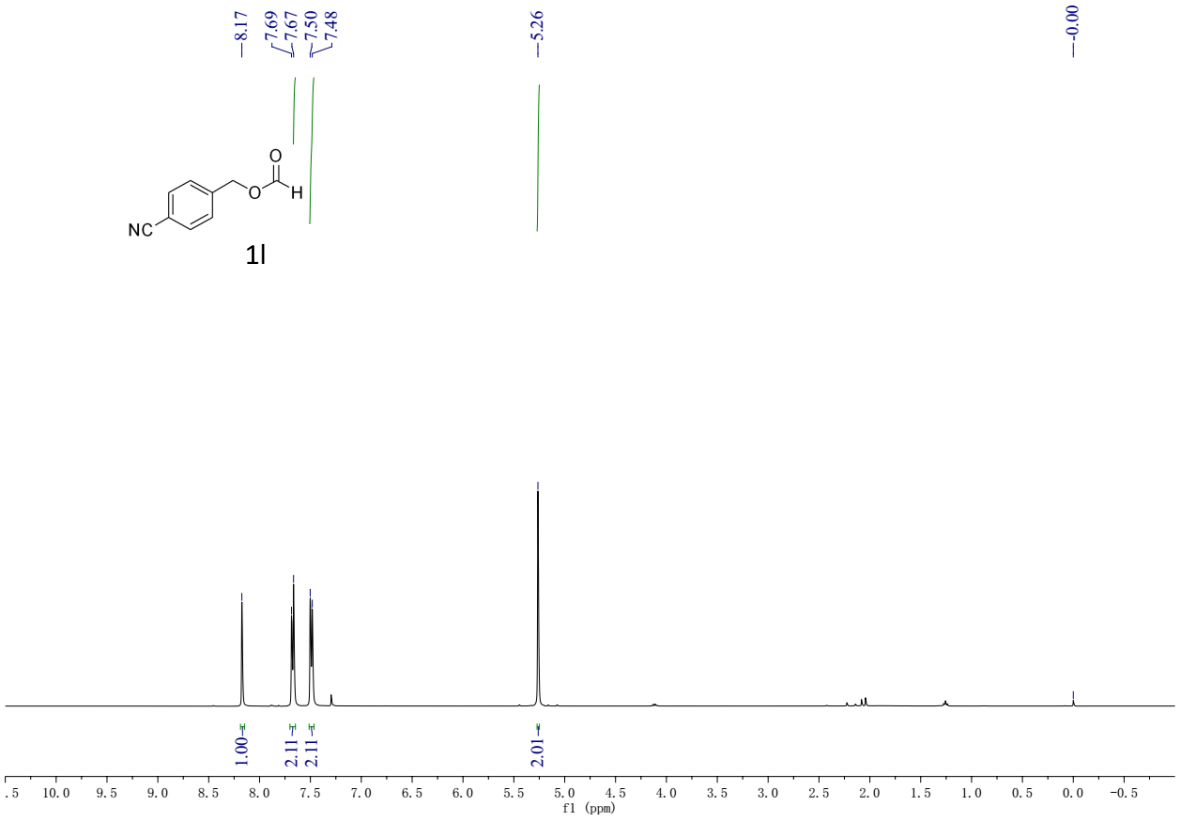
3.00
f1 (ppm)

1k


166.1
160.2
139.9
129.7
129.4
127.4
77.3
77.0
76.7
64.3
51.7
f1 (ppm)

1k


8.17
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7.67
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O
O
H
NC
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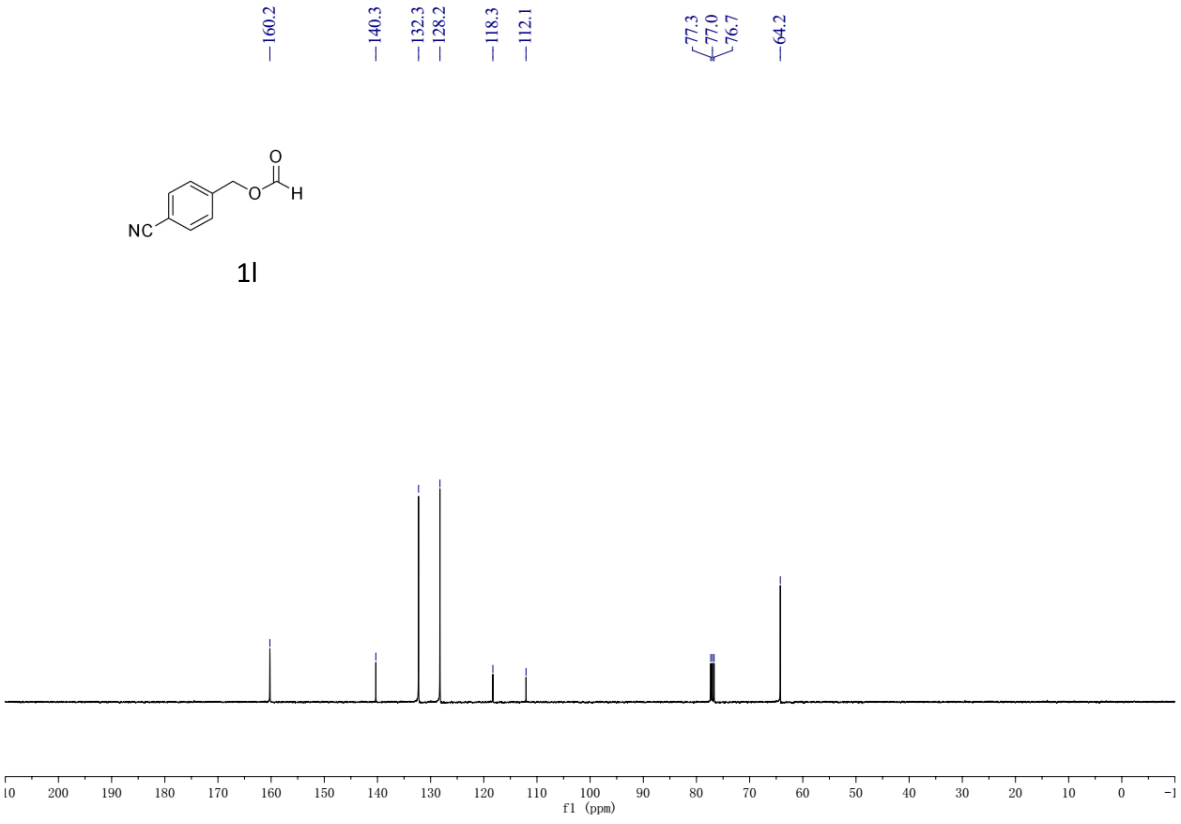
2.11
2.01
f1 (ppm)

1l


160.2
140.3
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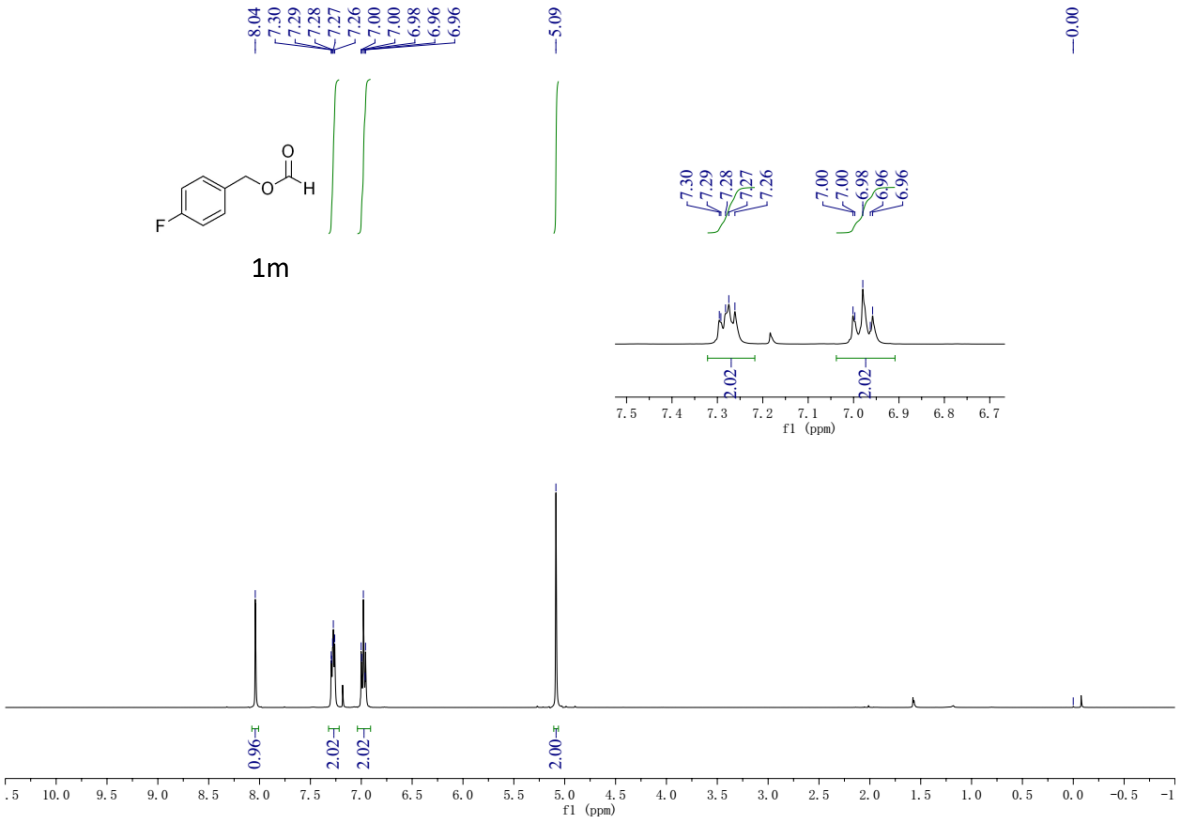
112.1
77.3
77.0
76.7
64.2
O
O
H
NC
f1 (ppm)

1l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7.30
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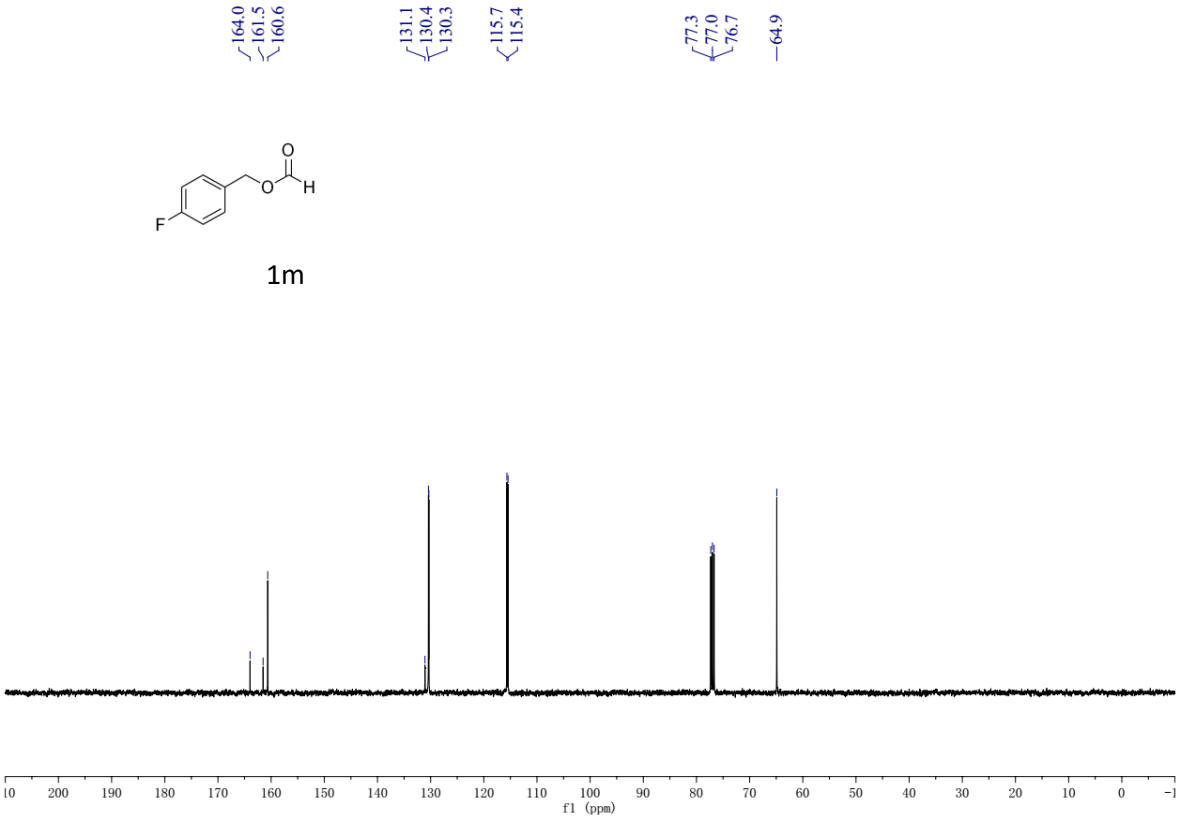
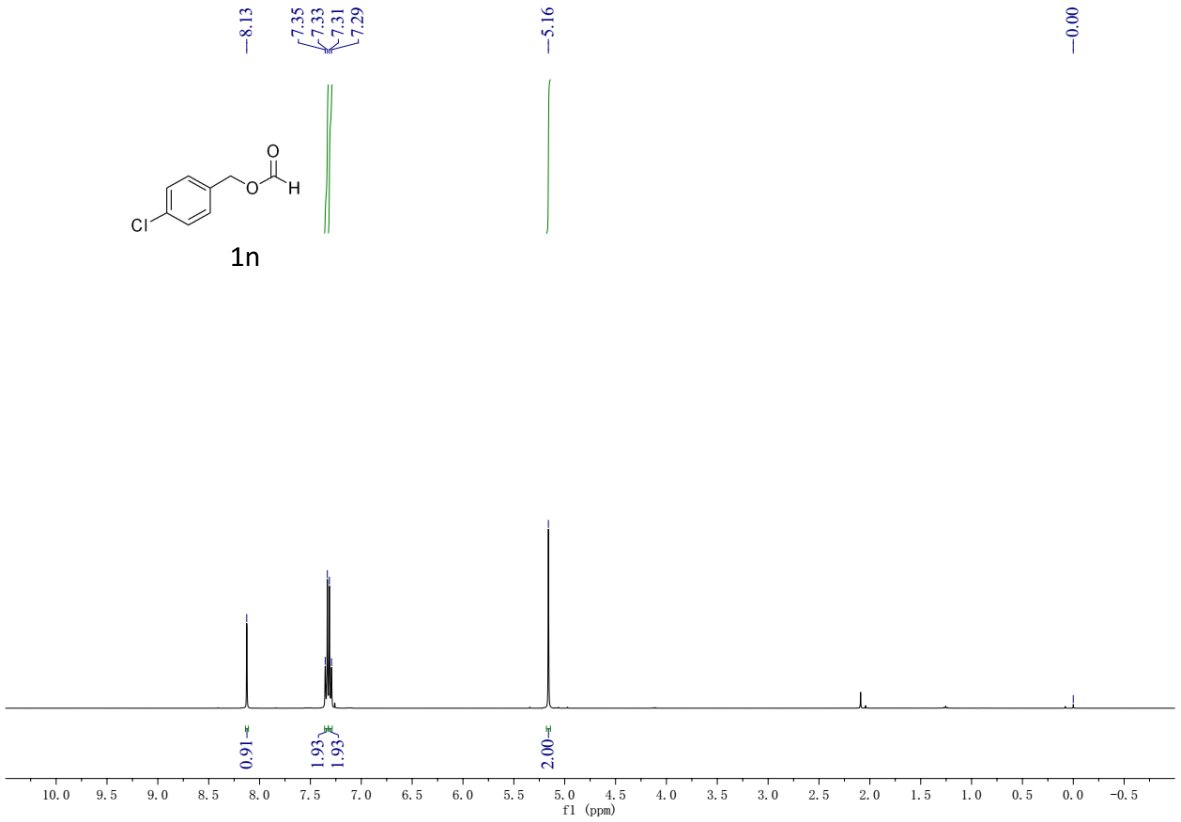
7.28
7.27
7.26
7.00
7.00
6.98
6.96
6.96
5.09
0.00
O
O
H
F
2.02
2.02
f1 (ppm)
0.96
2.02
2.02
2.00
f1 (ppm)

1m


164.0
161.5
160.6
131.1
130.4
130.3
115.7
115.4
77.3
77.0
76.7
64.9
O
O
H
F
f1 (ppm)

1m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7.33
7.31
7.29
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O
H
Cl
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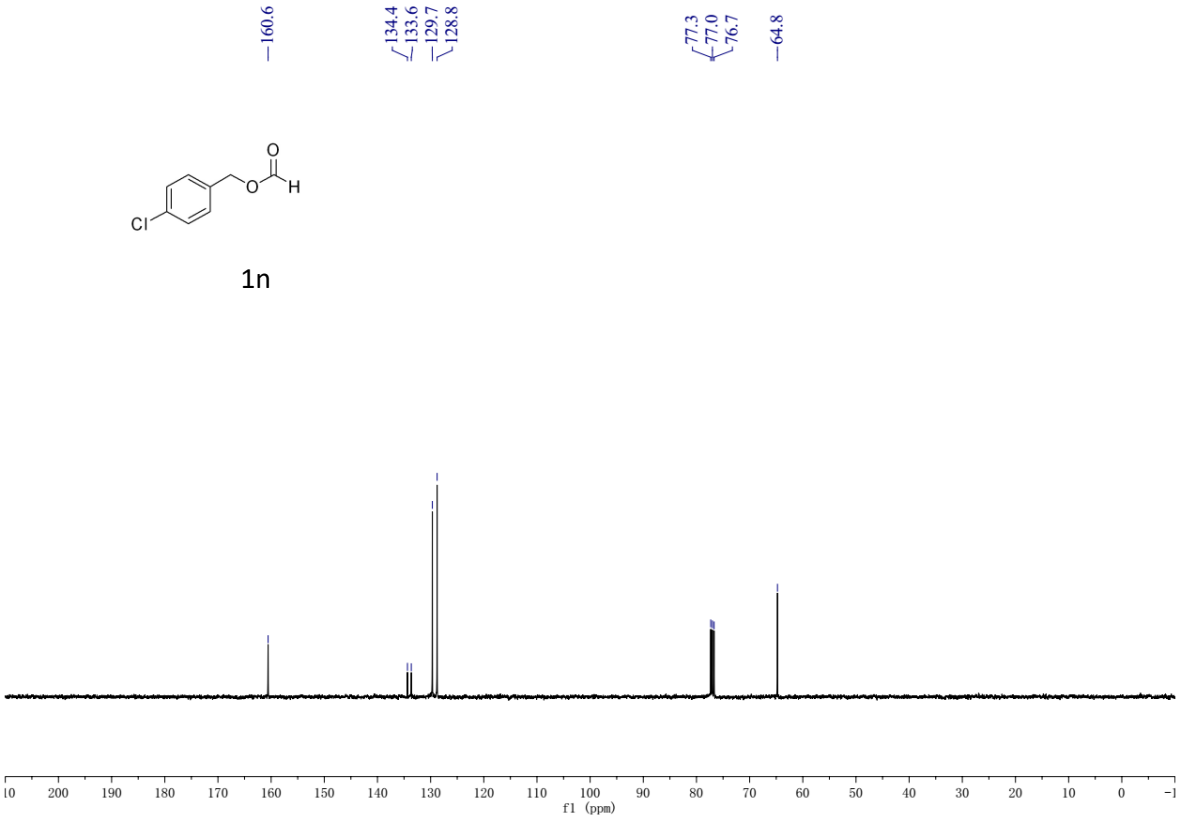
2.00
f1 (ppm)

1n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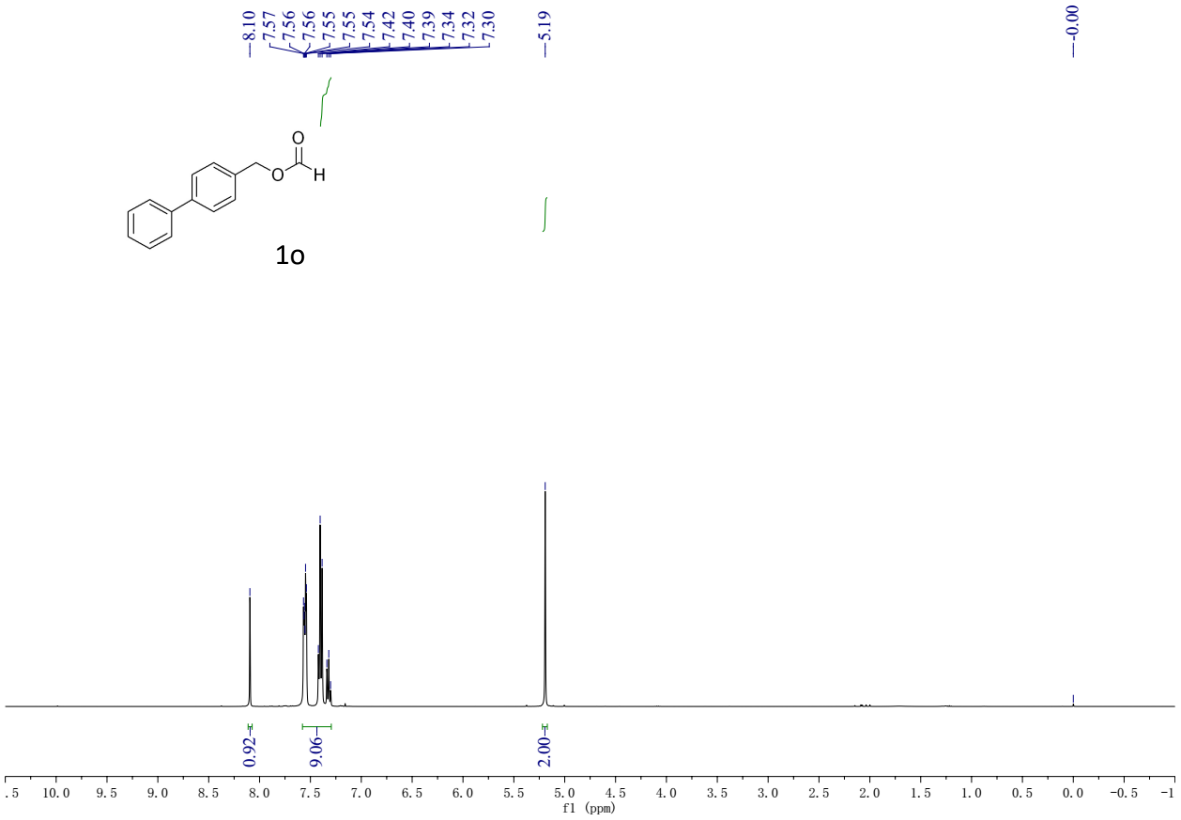
134.4
133.6
129.7
128.8
77.3
77.0
76.7
64.8
O
O
H
Cl
f1 (ppm)

1n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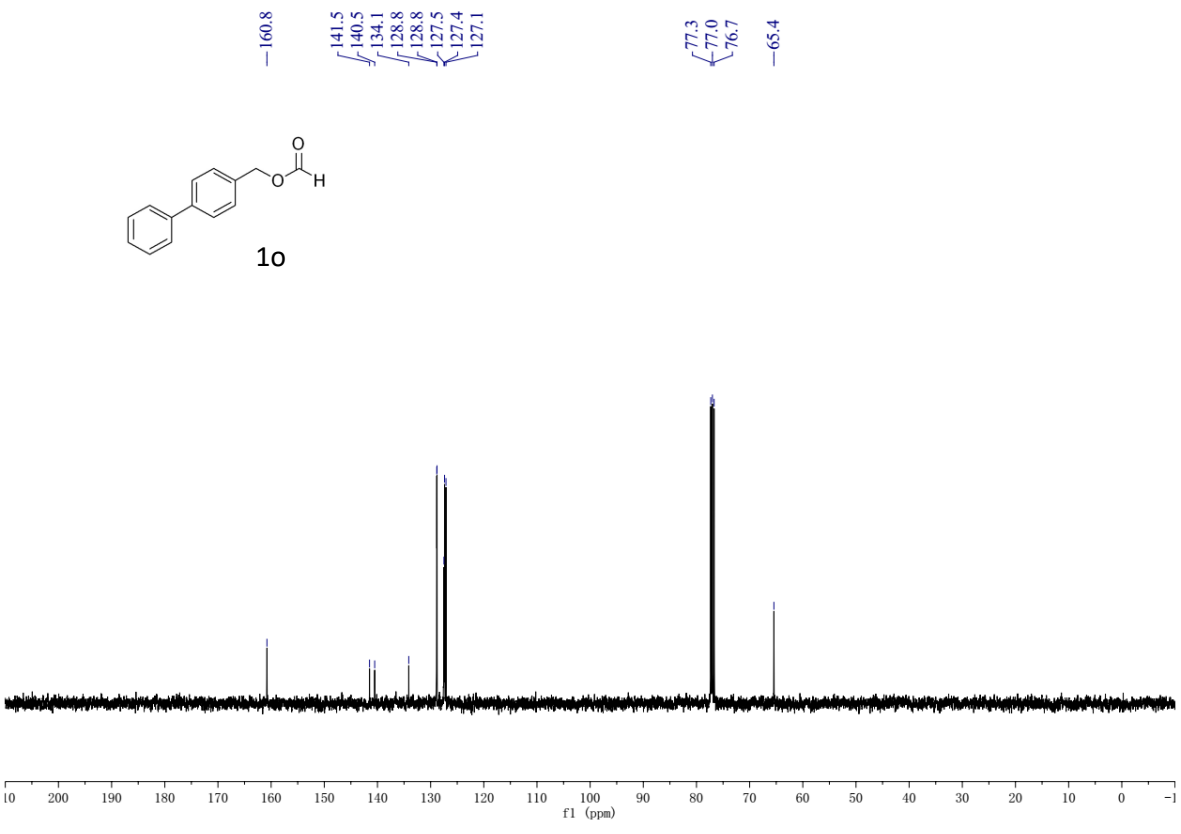
7.56
7.55
7.55
7.54
7.42
7.40
7.39
7.34
7.32
7.30
5.19
0.00
O
O
H
0.92
9.06
2.00
f1 (ppm)

1o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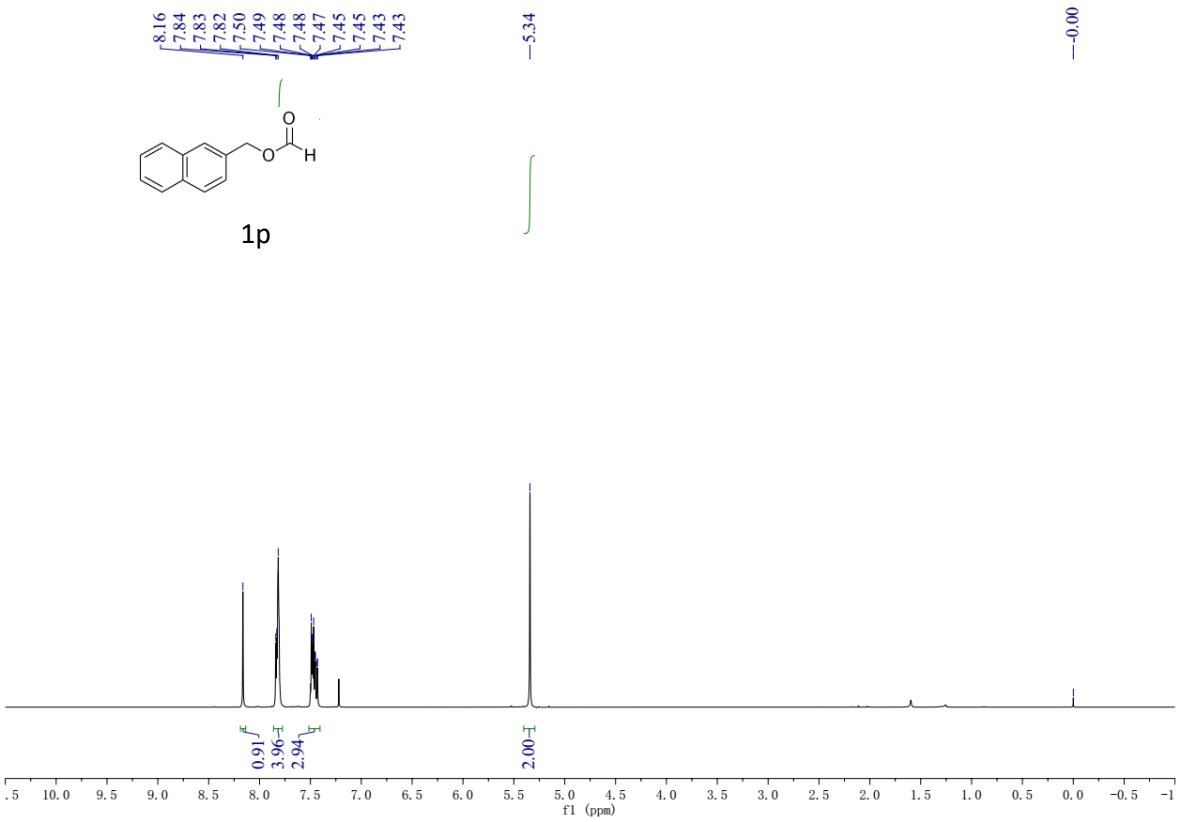
76.7
65.4
O
O
H
f1 (ppm)

1o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7.48
7.48
7.47
7.45
7.45
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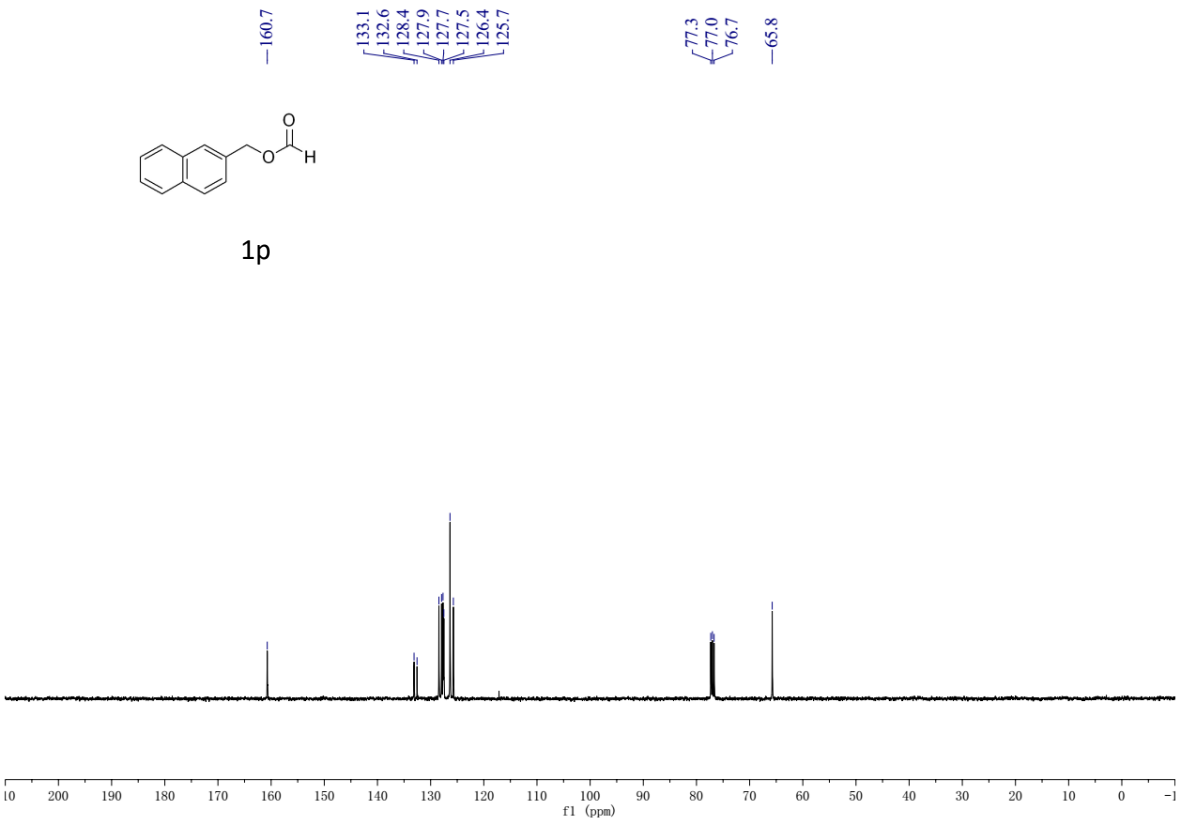
5.34
0.00
O
O
H
0.91
3.96
2.94
2.00
f1 (ppm)

1p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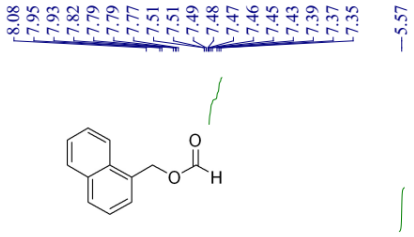
132.6
128.4
127.9
127.7
127.5
126.4
125.7
77.3
77.0
76.7
65.8
O
O
H
f1 (ppm)

1p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7.95
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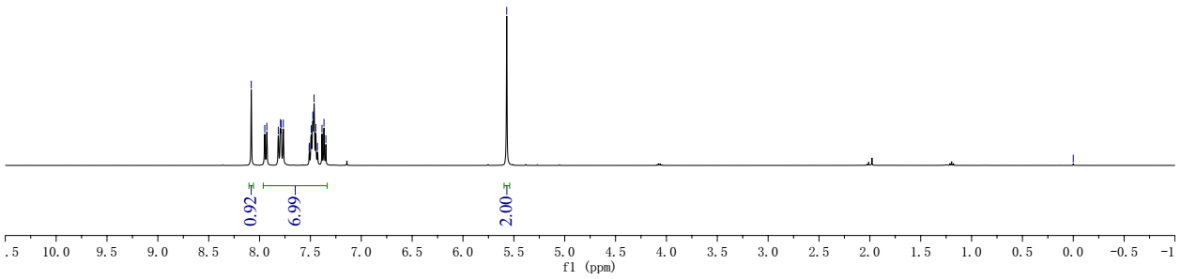
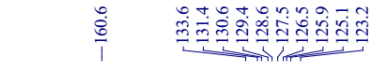
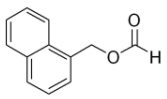
7.79
7.79
7.77
7.51
7.51
7.49
7.48
7.47
7.46
7.45
7.43
7.39
7.37
7.35
5.57
0.00
O
O
H

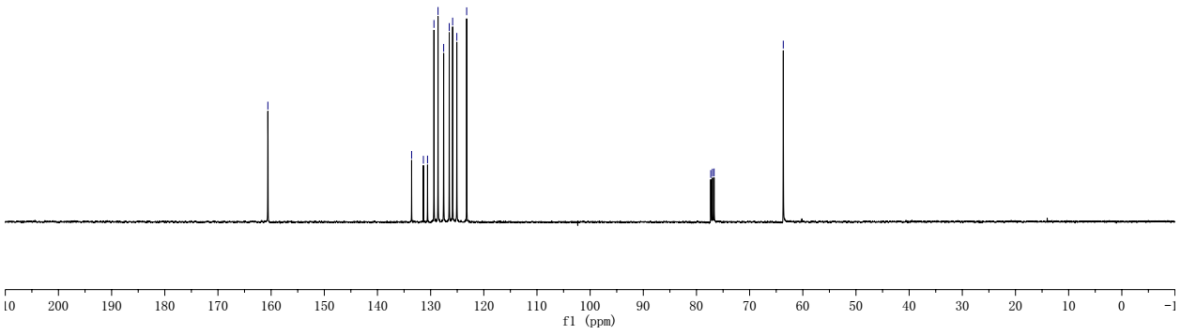
1q

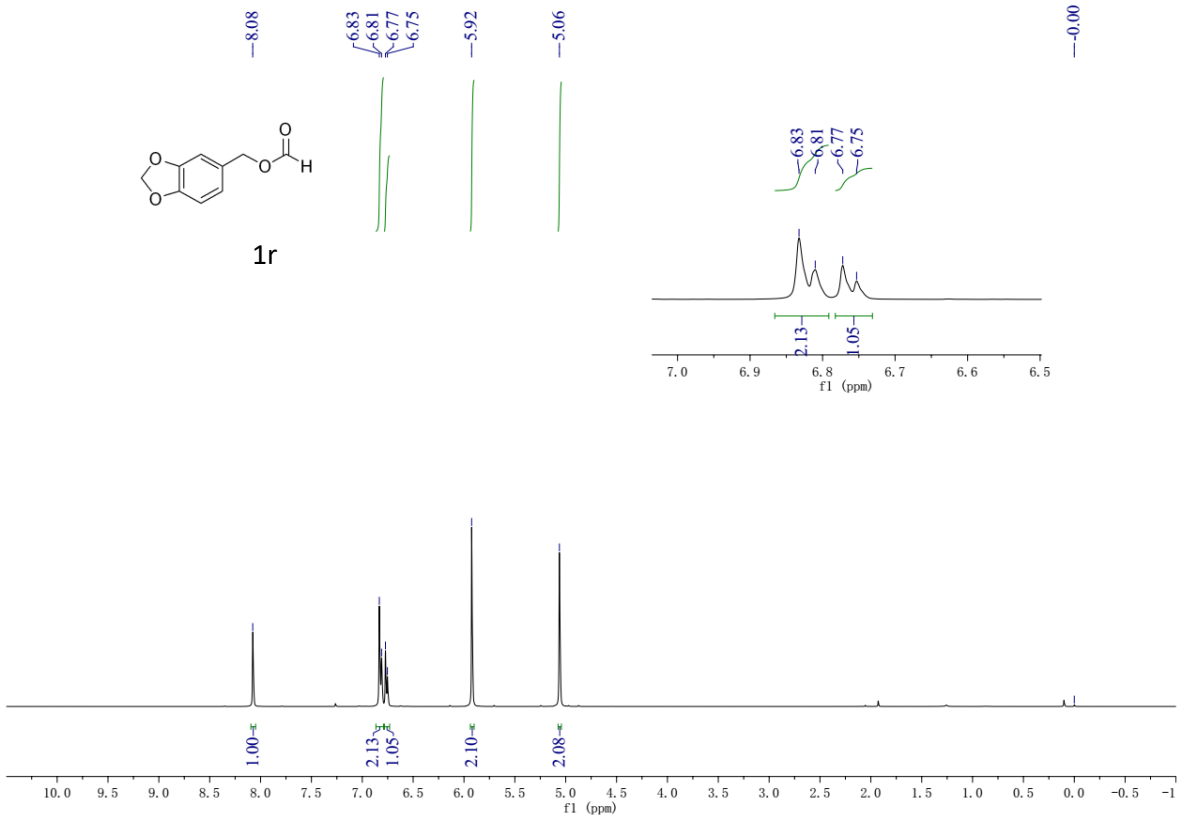

0.92
6.99
2.00
f1 (ppm)

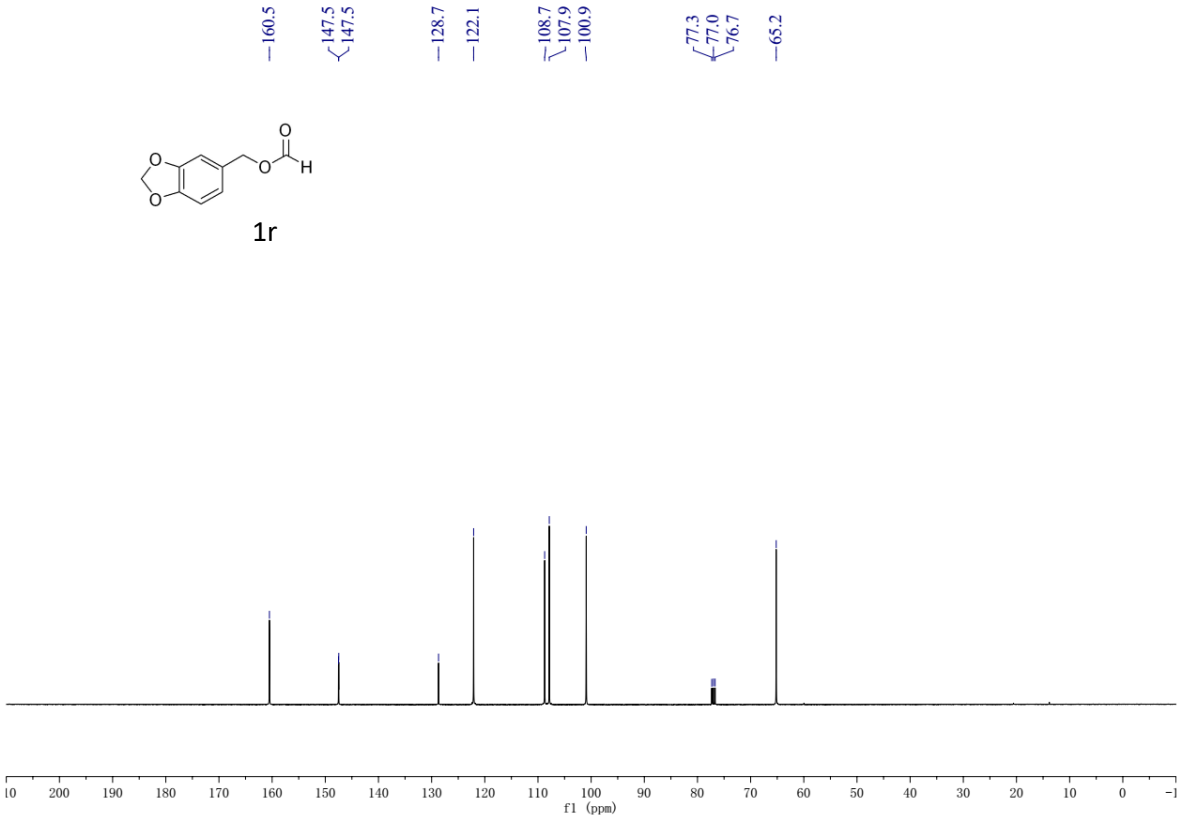

160.6
133.6
131.4
130.6
129.4
128.6
127.5
126.5
125.9
125.1
123.2
77.3
77.0
76.7
63.7

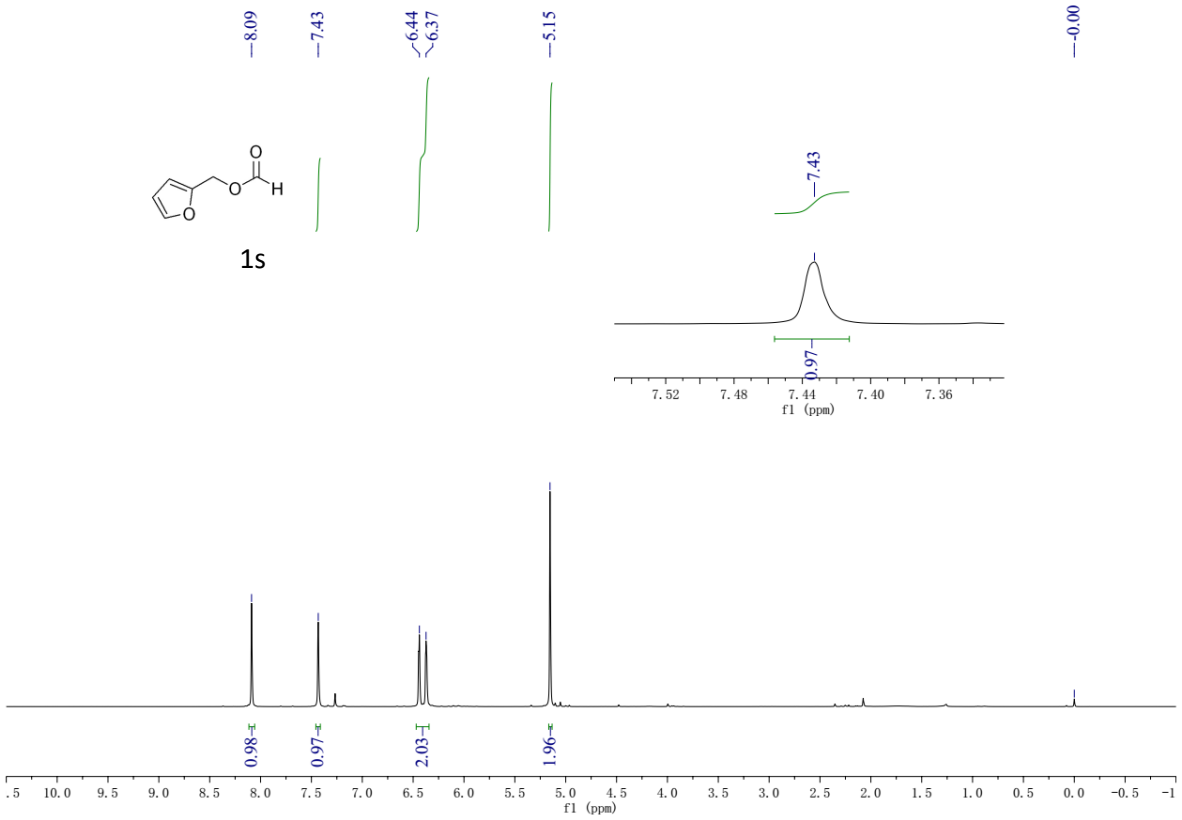

O
O
H

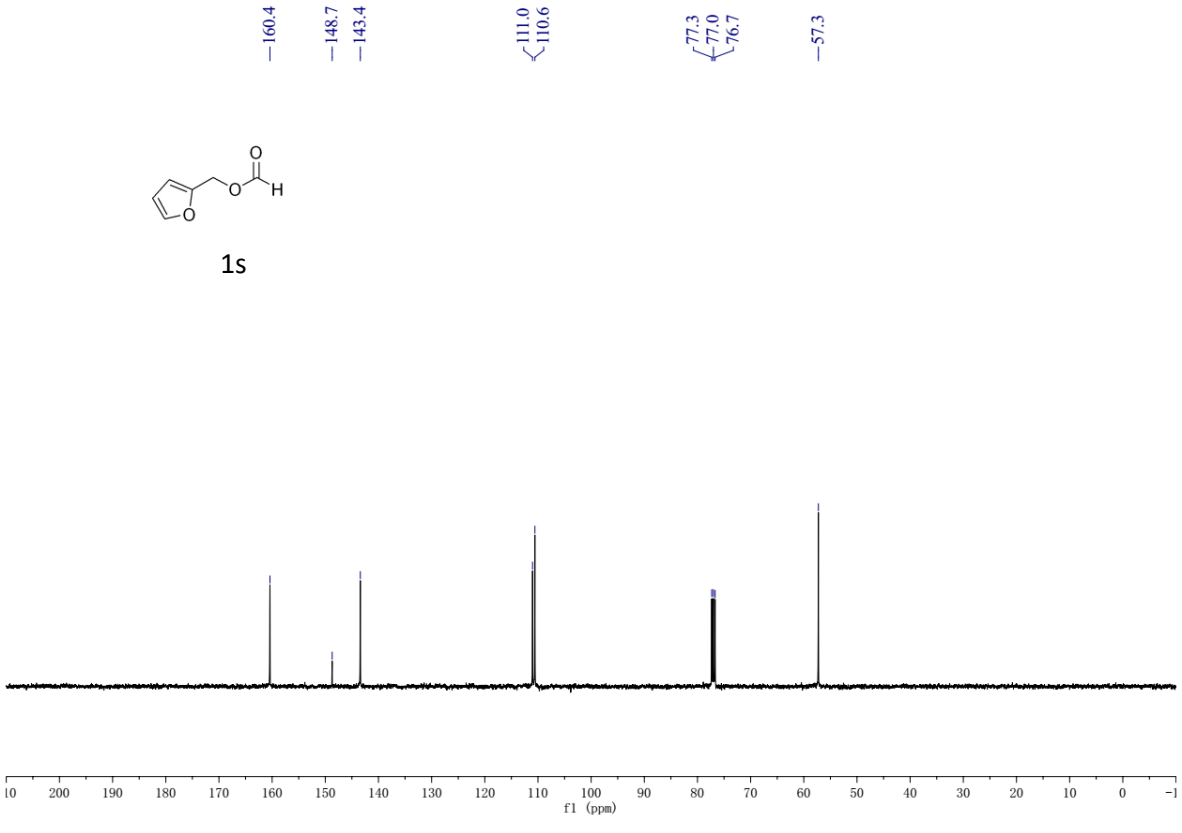
1q

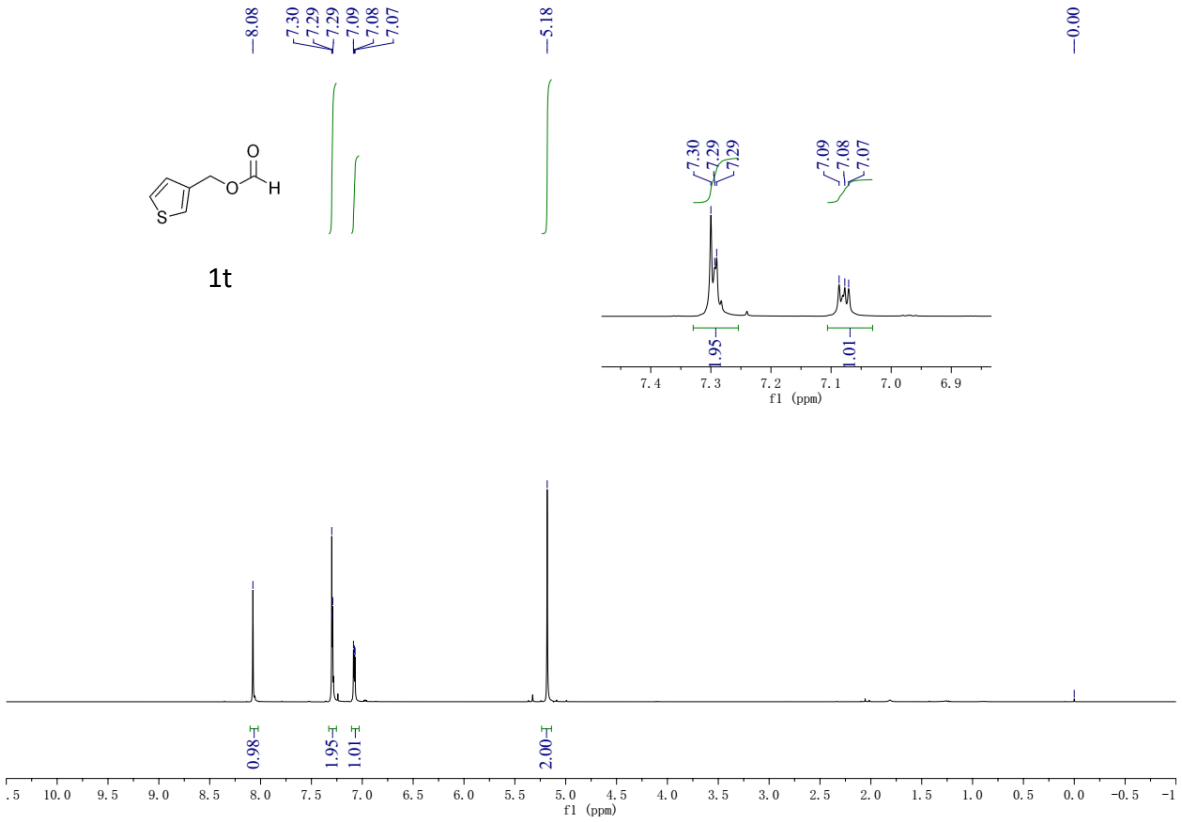

f1 (ppm)

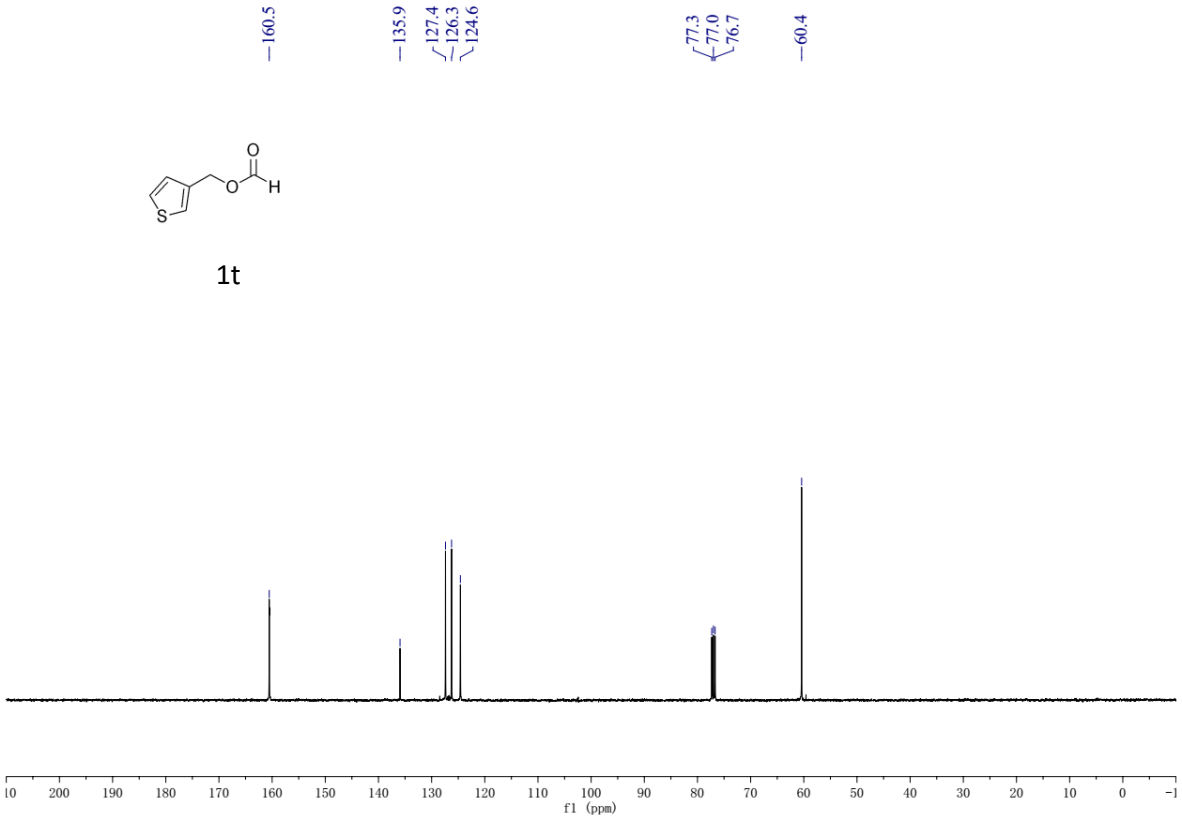

8.08
6.83
6.81
6.77
6.75
5.92
5.06
0.00
1r
2.13
1.05
f1 (ppm)
1.00
2.13
1.05
2.10
2.08
f1 (ppm)


160.5
147.5
147.5
128.7
122.1
108.7
107.9
100.9
77.3
77.0
76.7
65.2
1r
f1 (ppm)

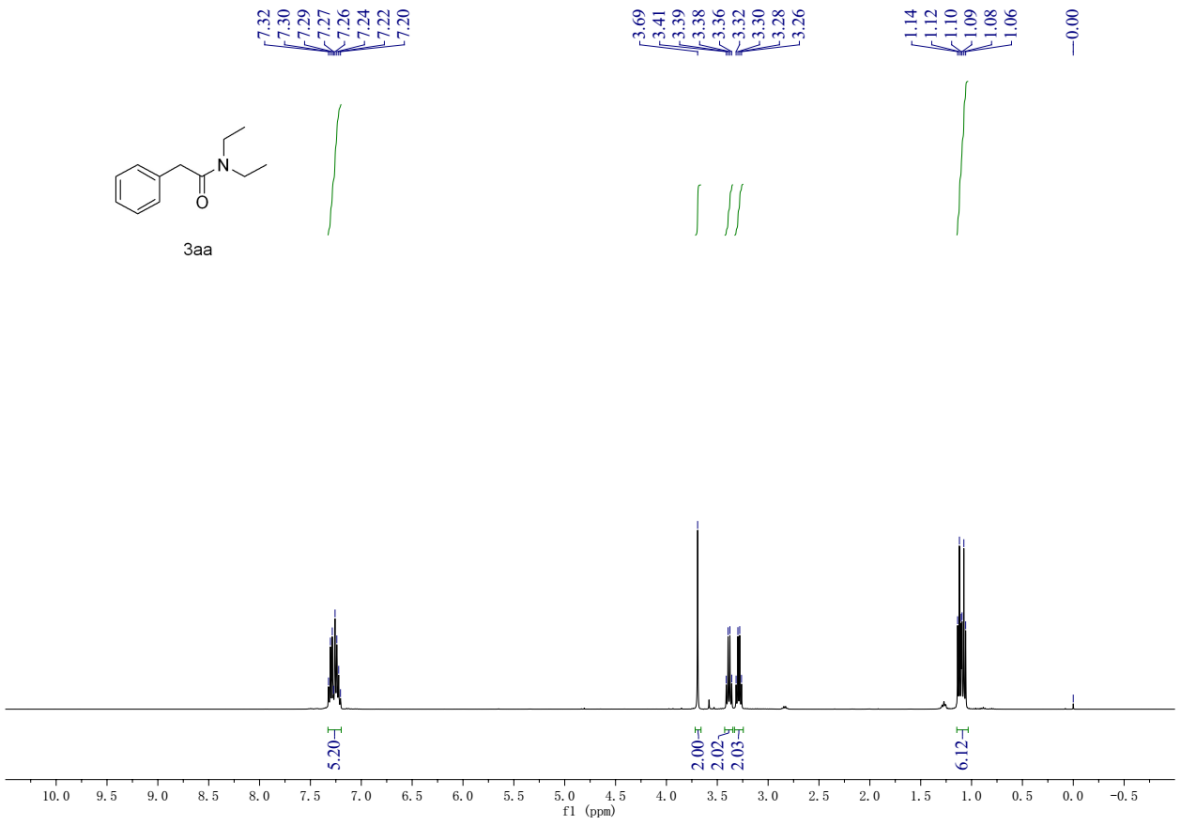

8.09
7.43
6.44
6.37
5.15
0.00
O
O
H
O
1s
7.43
0.97
7.52
7.48
7.44
7.40
7.36
f1 (ppm)
0.98
0.97
2.03
1.96
f1 (ppm)

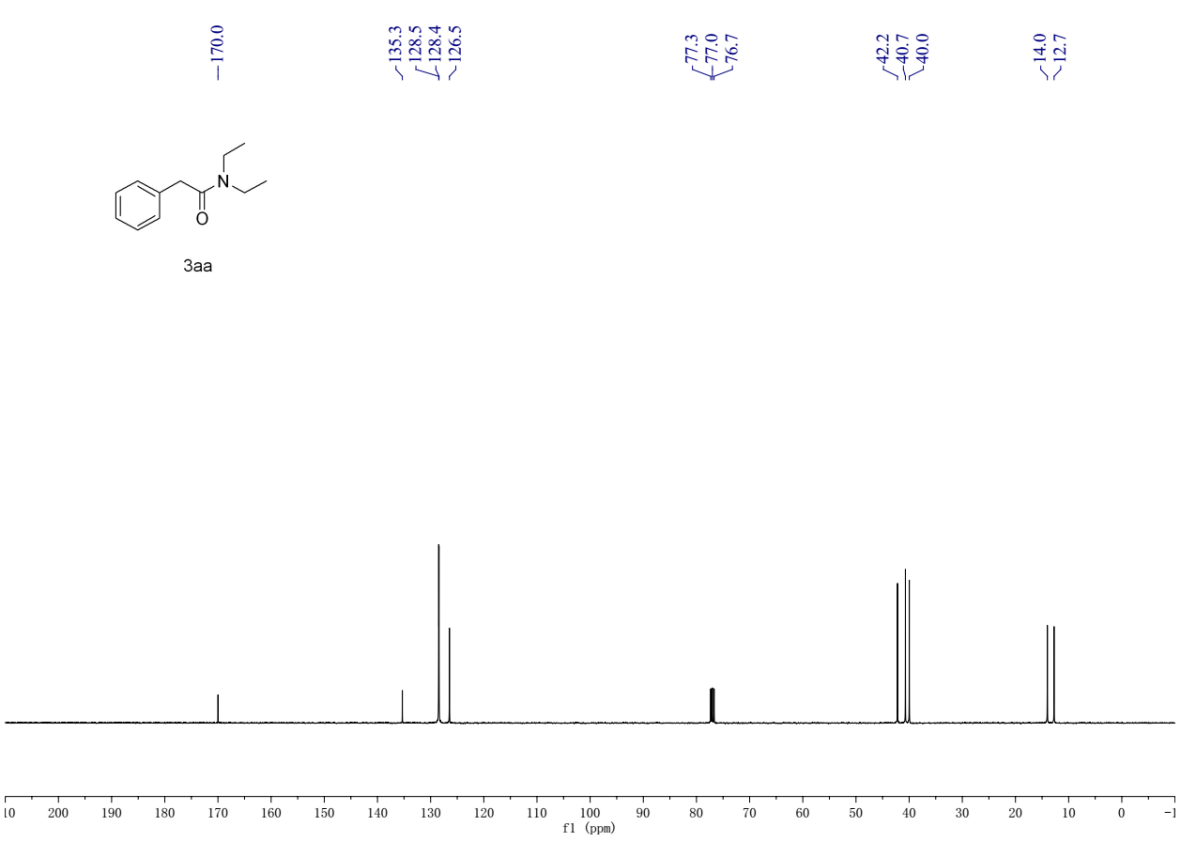

160.4
148.7
143.4
111.0
110.6
77.3
77.0
76.7
57.3
O
O
H
O
1s
f1 (ppm)

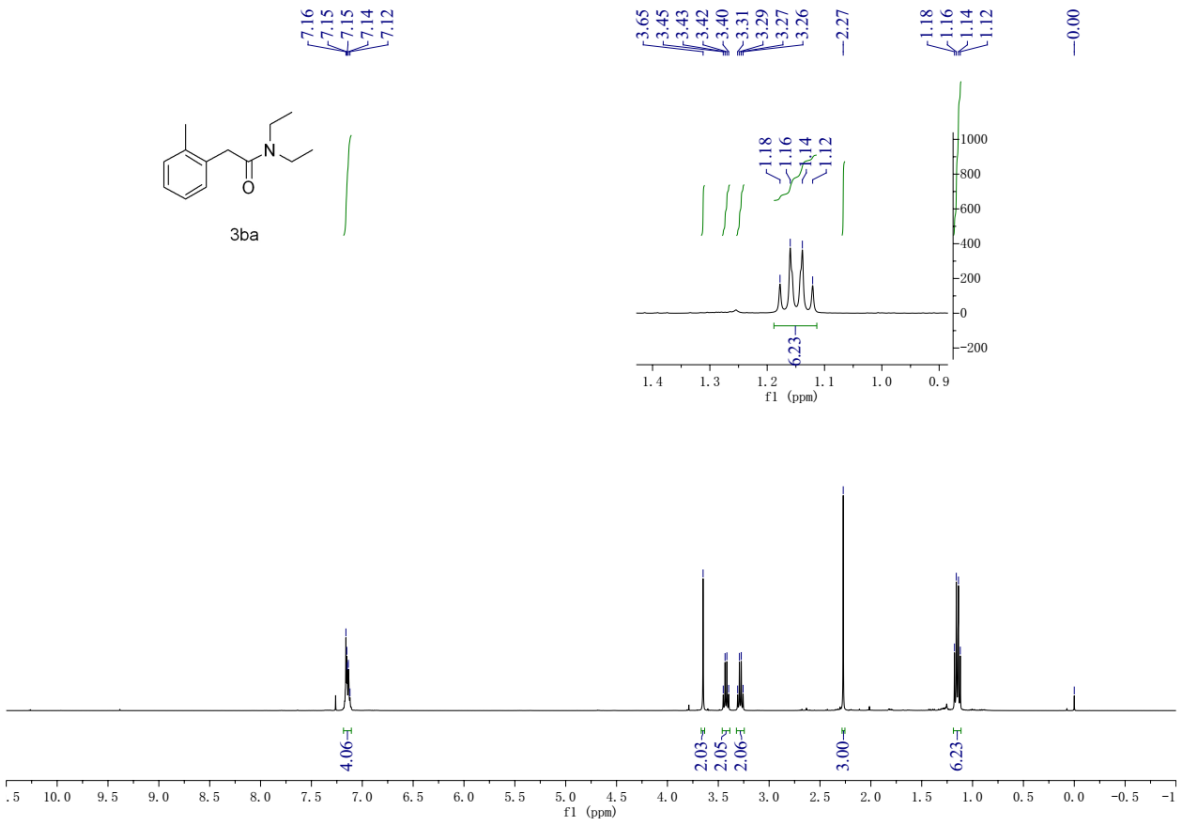

8.08
7.30
7.29
7.29
7.09
7.08
7.07
5.18
0.00
O
O
H
S
1t
7.30
7.29
7.29
7.09
7.08
7.07
1.95
1.01
7.4
7.3
7.2
7.1
7.0
6.9
f1 (ppm)
0.98
1.95
1.01
2.00
f1 (ppm)

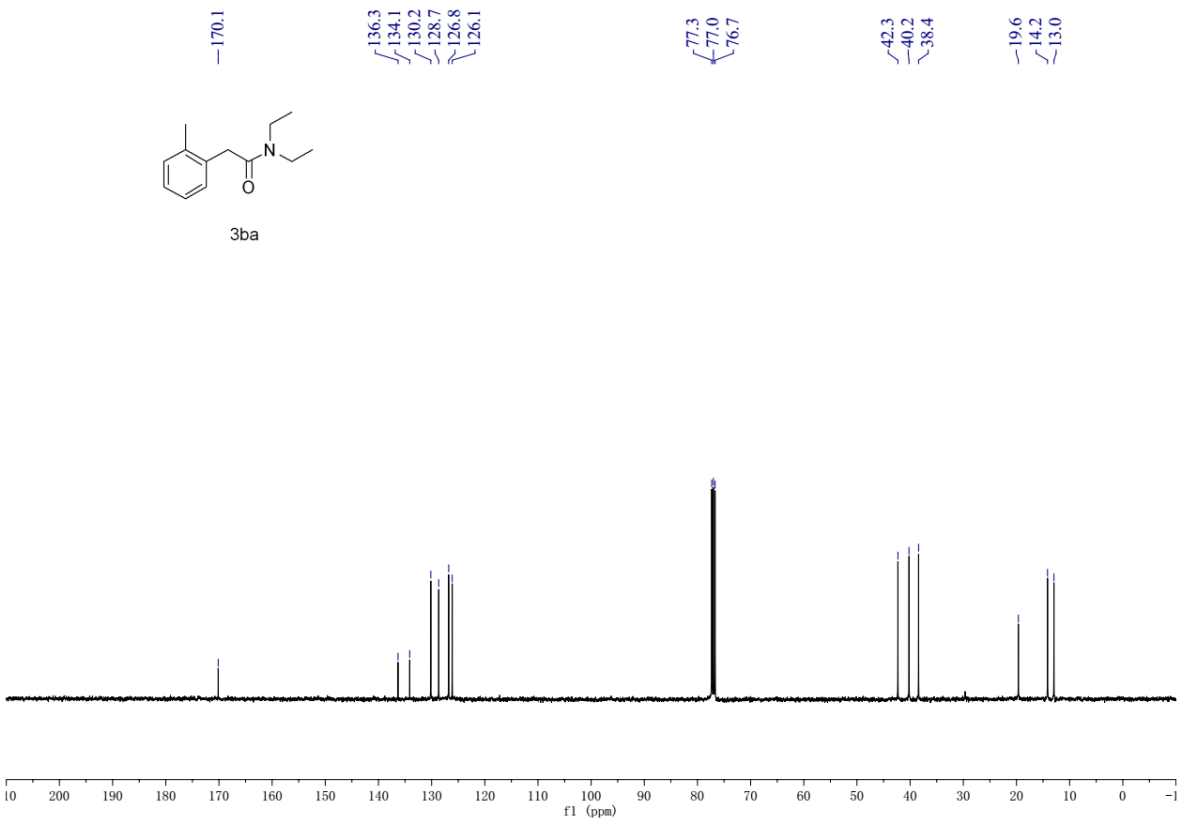

160.5
135.9
127.4
126.3
124.6
77.3
77.0
76.7
60.4
O
O
H
S
1t
f1 (ppm)

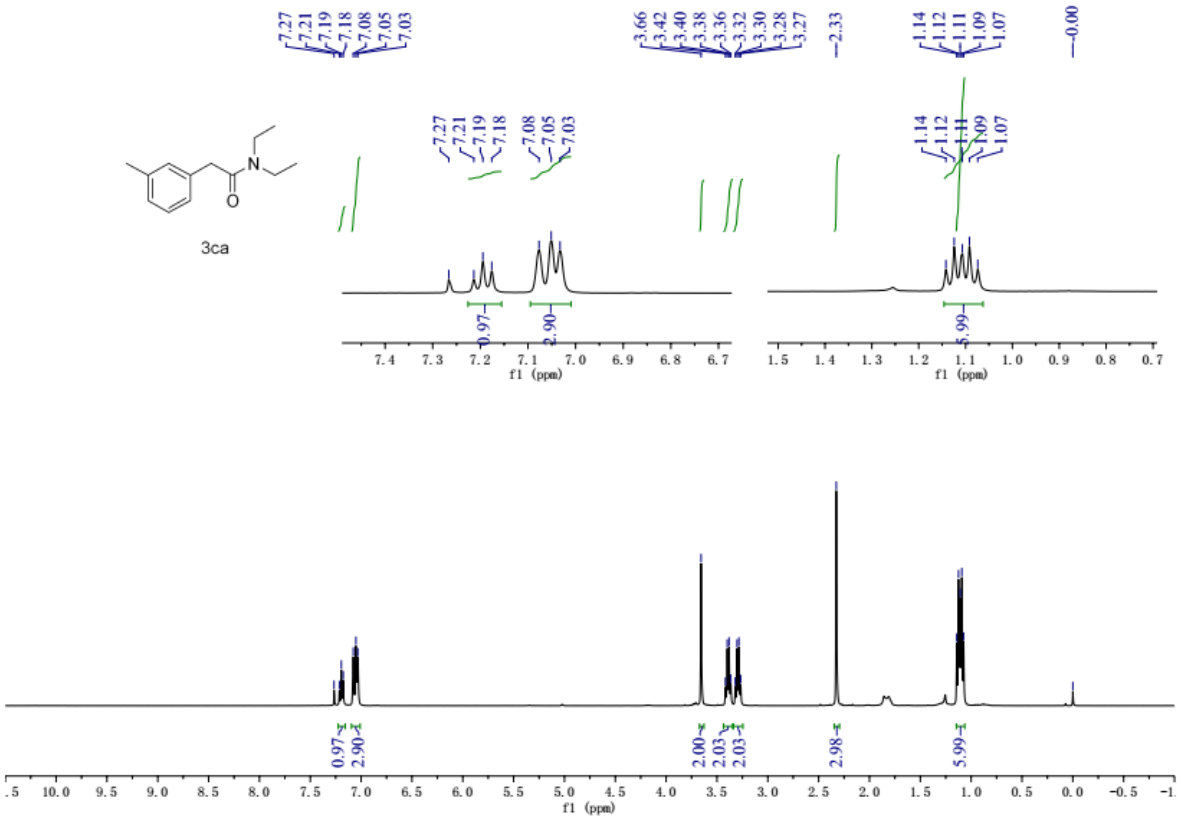
8. Spectra of Arylacetamides



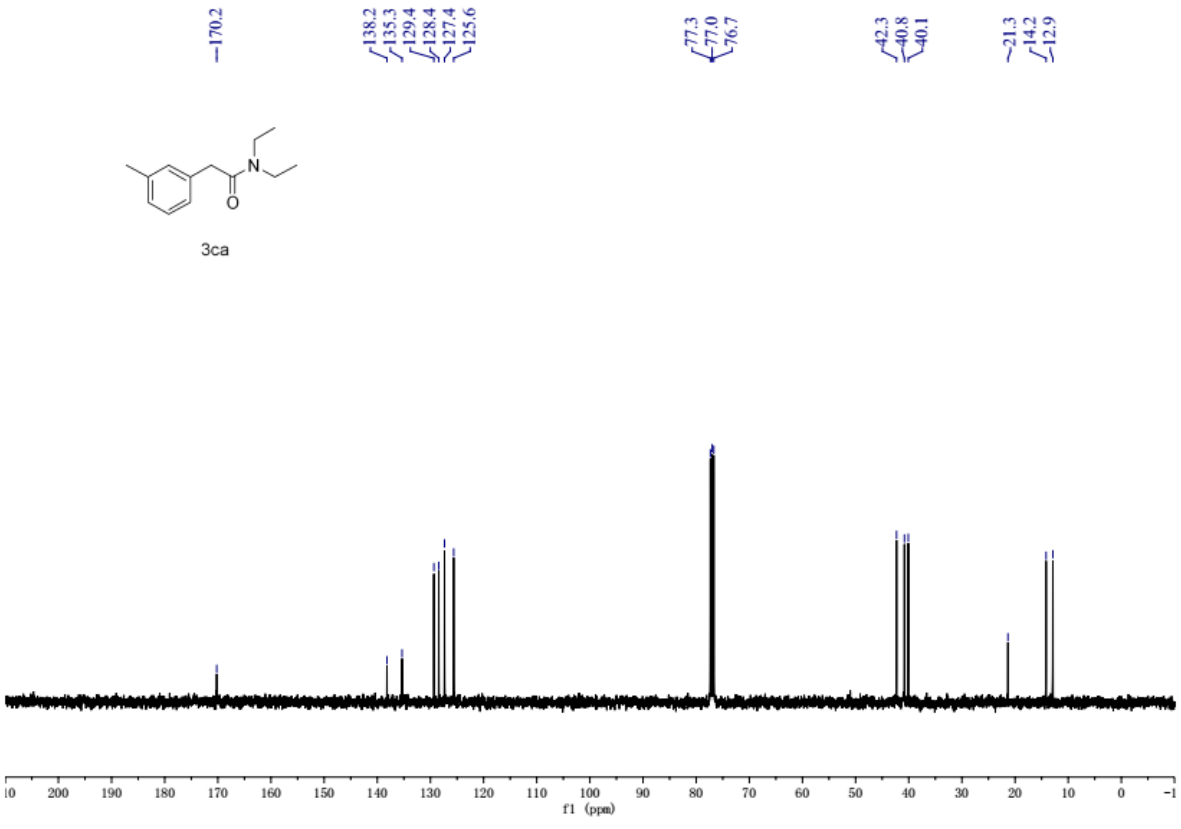


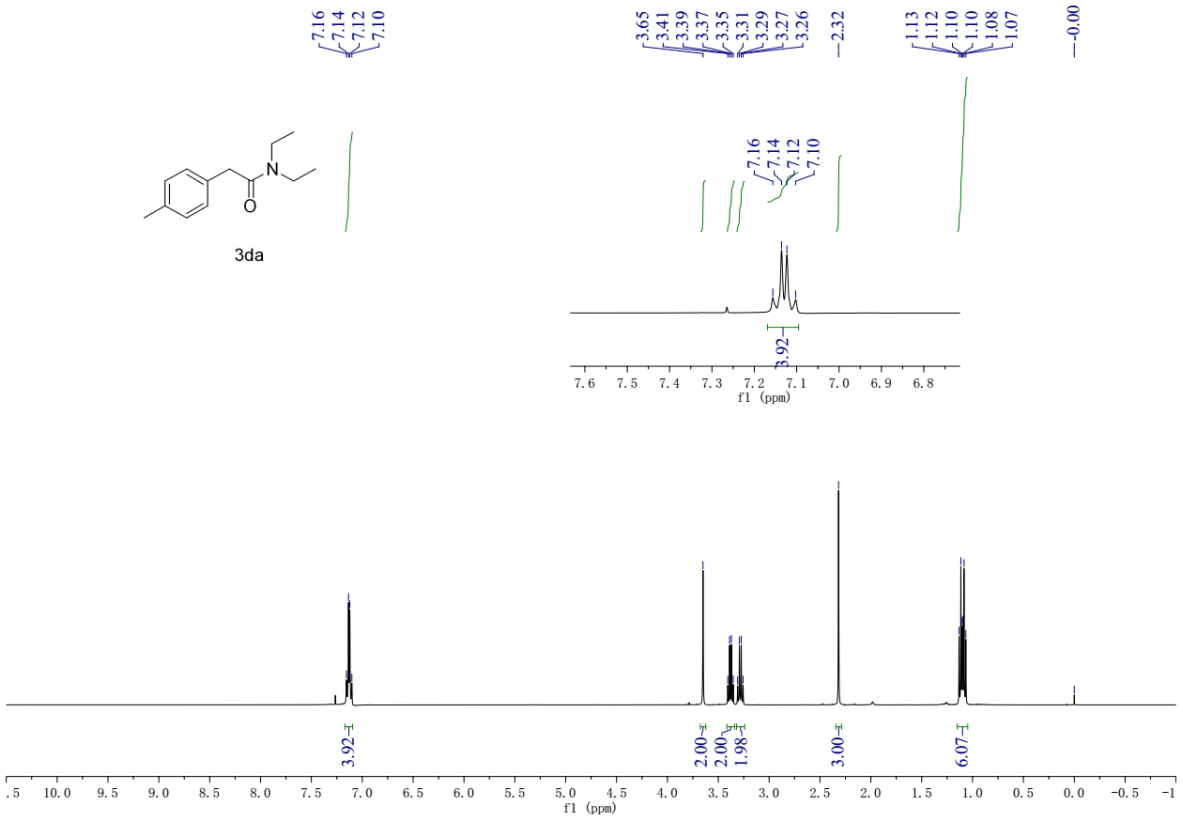

7.16
7.15
7.15
7.14
7.12
3.65
3.45
3.43
3.42
3.40
3.31
3.29
3.27
3.26
2.27
1.18
1.16
1.14
1.12
0.00
3ba
6.23
f1 (ppm)
4.06
2.03
2.05
2.06
3.00
6.23
f1 (ppm)

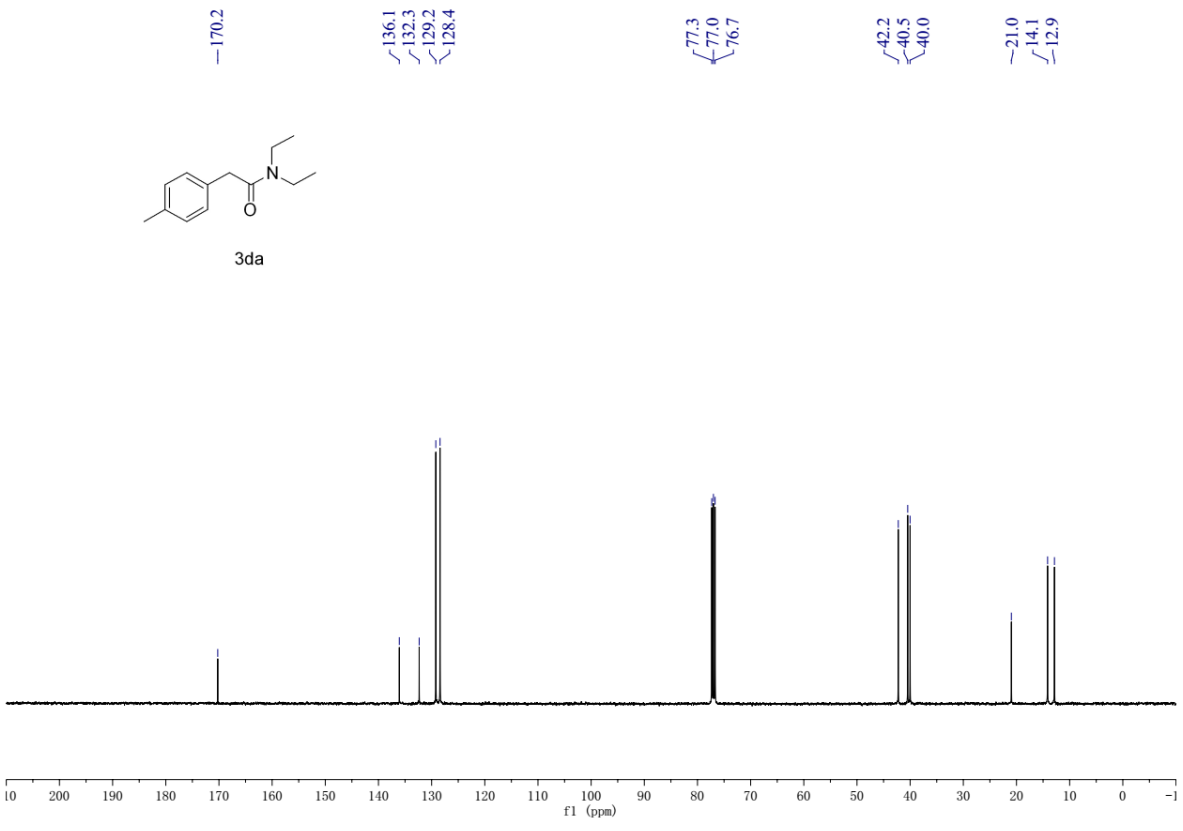

170.1
136.3
134.1
130.2
128.7
126.8
126.1
77.3
77.0
76.7
42.3
40.2
38.4
19.6
14.2
13.0
3ba
f1 (ppm)

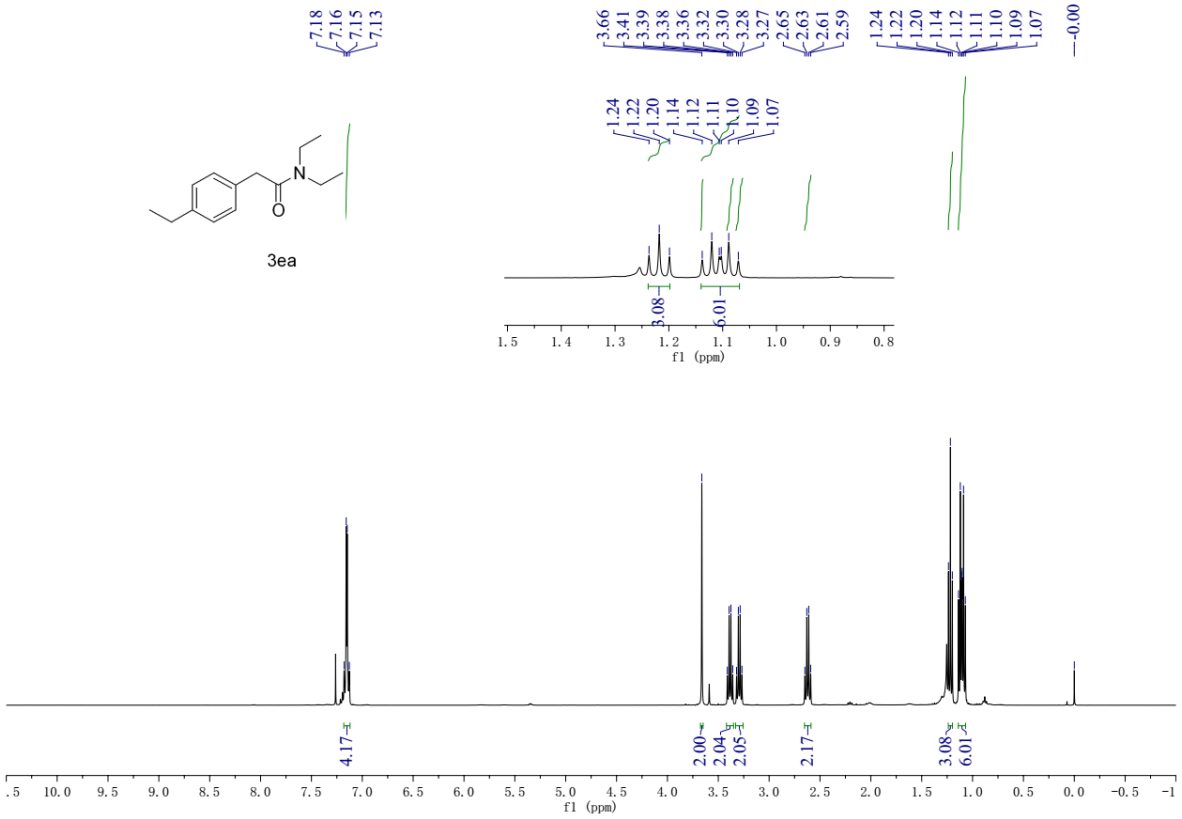

7.27
7.21
7.19
7.18
7.08
7.05
7.03
3.66
3.42
3.40
3.38
3.36
3.32
3.30
3.28
3.27
2.33
1.14
1.12
1.11
1.09
1.07
0.00
0.97
2.90
5.99
f1 (ppm)
3ca

0.97
2.90
2.00
2.03
2.03
2.98
5.99
f1 (ppm)

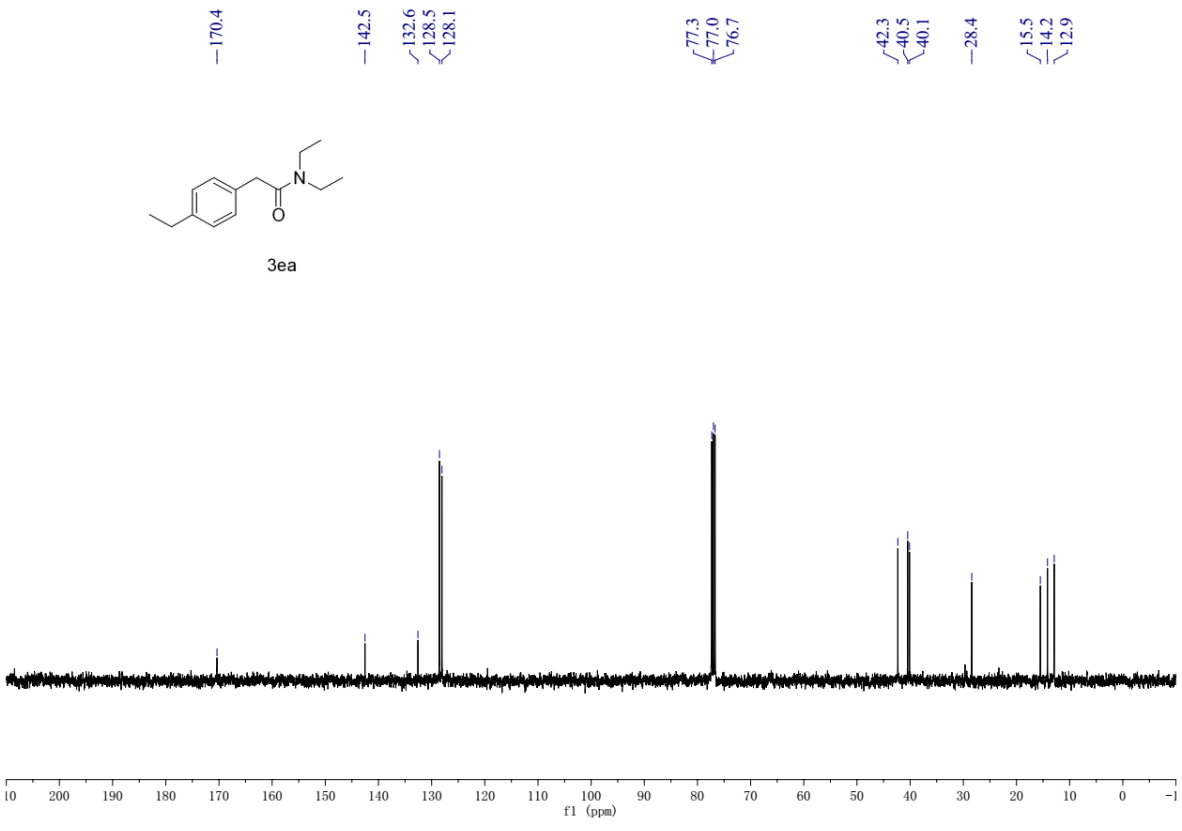

170.2
138.2
135.3
129.4
128.4
127.4
125.6
77.3
77.0
76.7
42.3
40.8
40.1
21.3
14.2
12.9
f1 (ppm)
3ca


7.16
7.14
7.12
7.10
3.65
3.41
3.39
3.37
3.35
3.31
3.29
3.27
3.26
2.32
1.13
1.12
1.10
1.10
1.08
1.07
0.00
3da
3.92
f1 (ppm)
3.92
2.00
2.00
1.98
3.00
6.07
f1 (ppm)

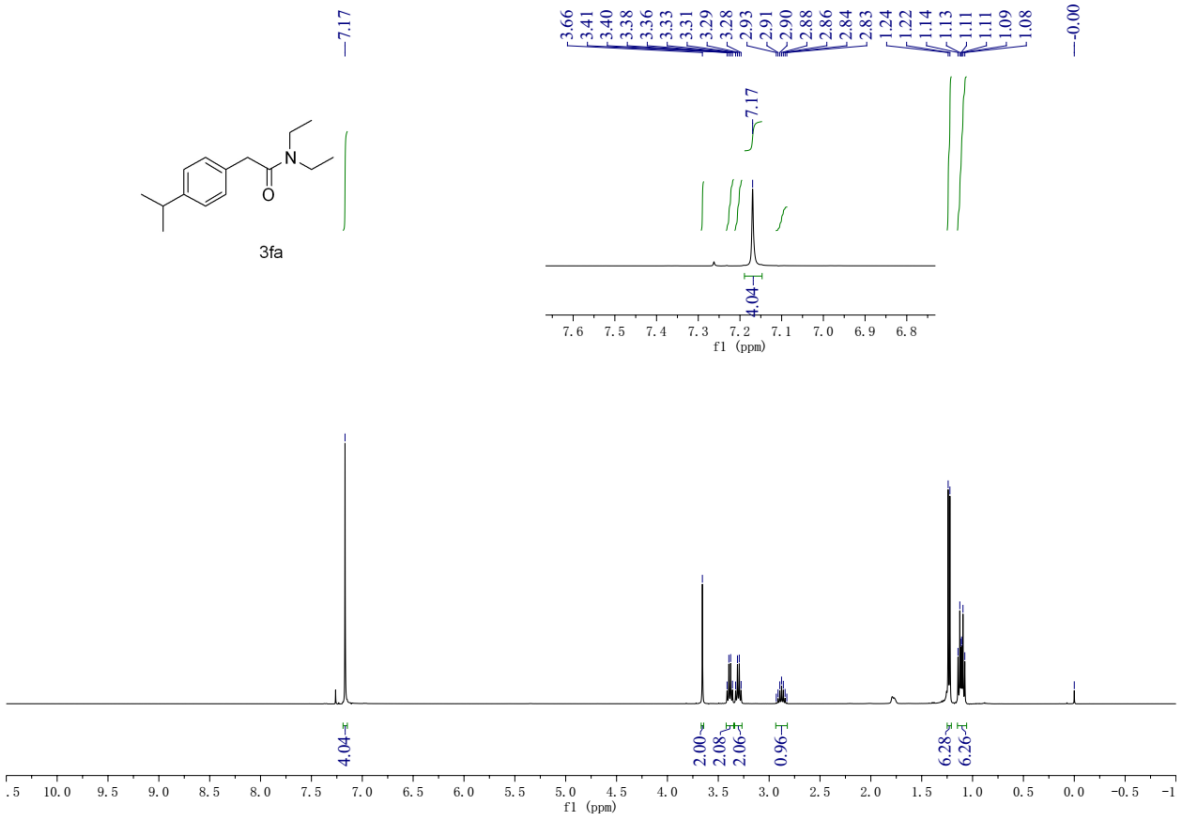

170.2
136.1
132.3
129.2
128.4
77.3
77.0
76.7
42.2
40.5
40.0
21.0
14.1
12.9
3da
f1 (ppm)

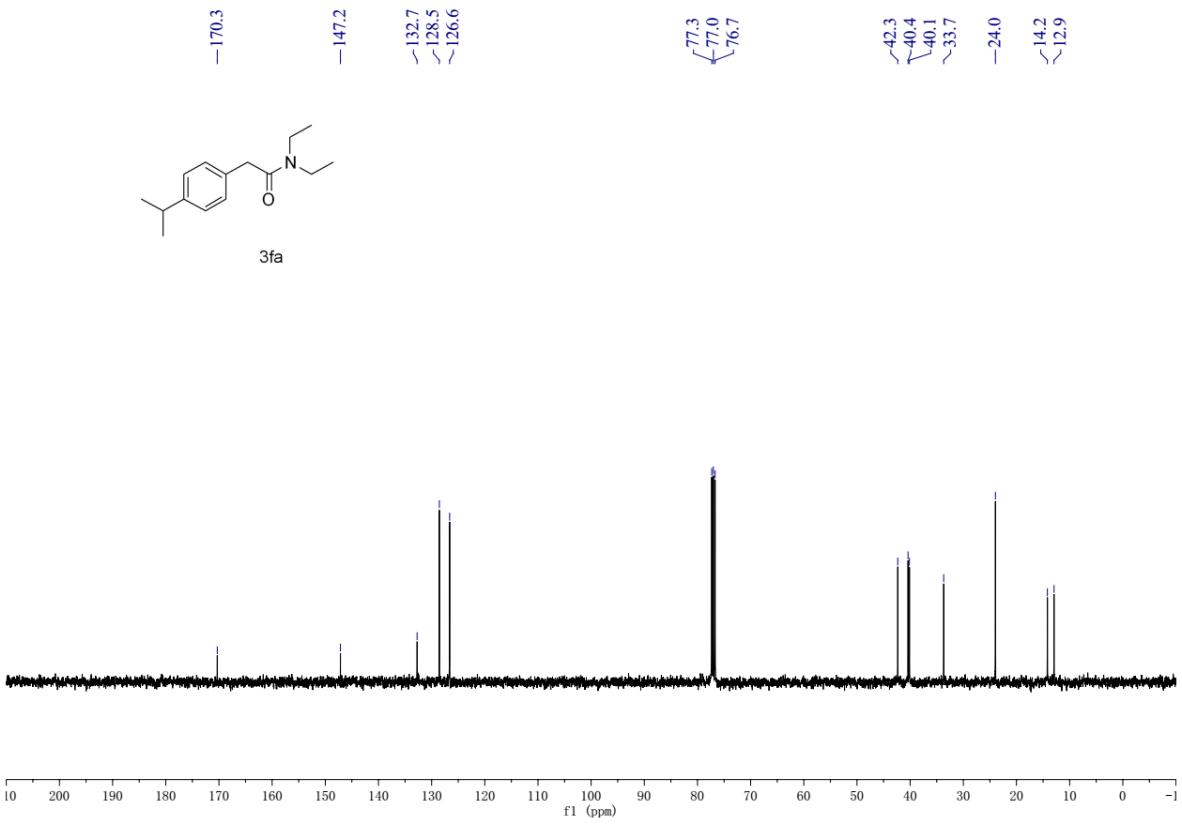

7.18
7.16
7.15
7.13
3.66
3.41
3.39
3.38
3.36
3.32
3.30
3.28
3.27
2.65
2.63
2.61
2.59
1.24
1.22
1.20
1.14
1.12
1.11
1.10
1.09
1.07
0.00
3.08
6.01
f1 (ppm)
4.17
2.00
2.04
2.05
2.17
3.08
6.01
f1 (ppm)

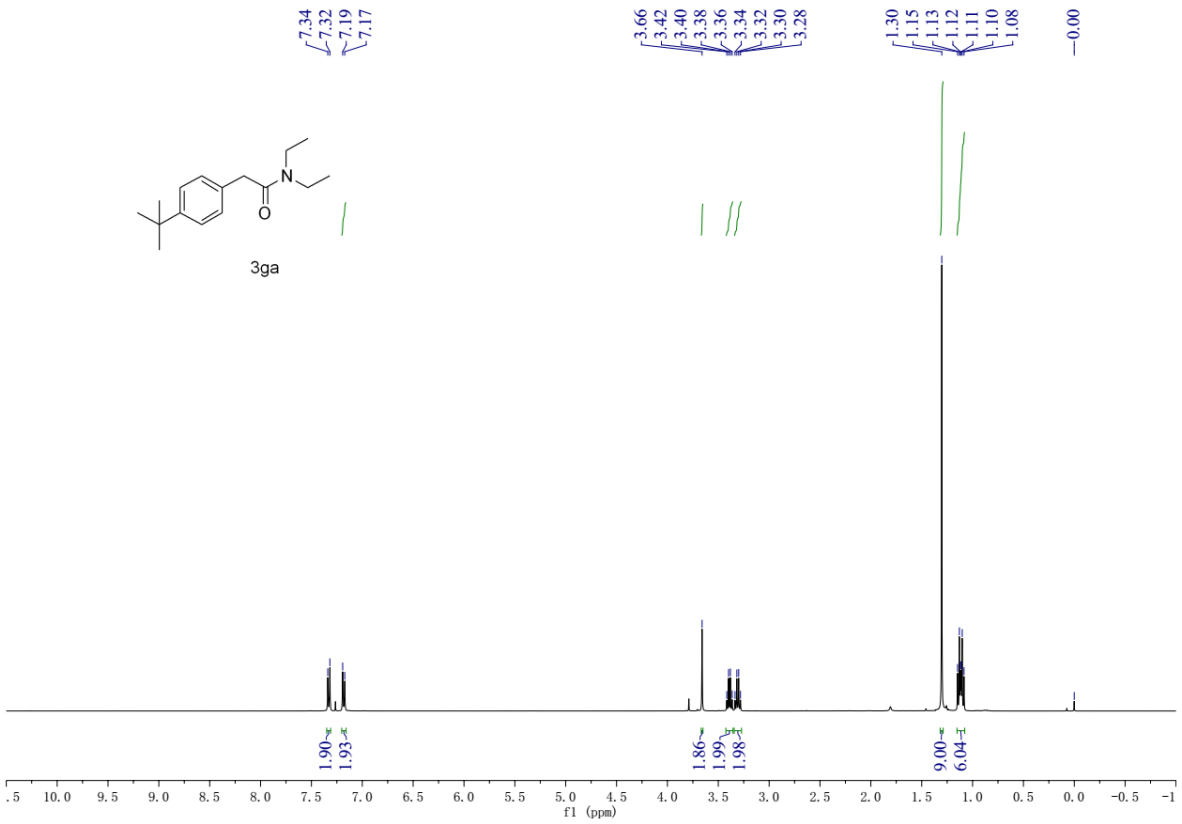
3ea

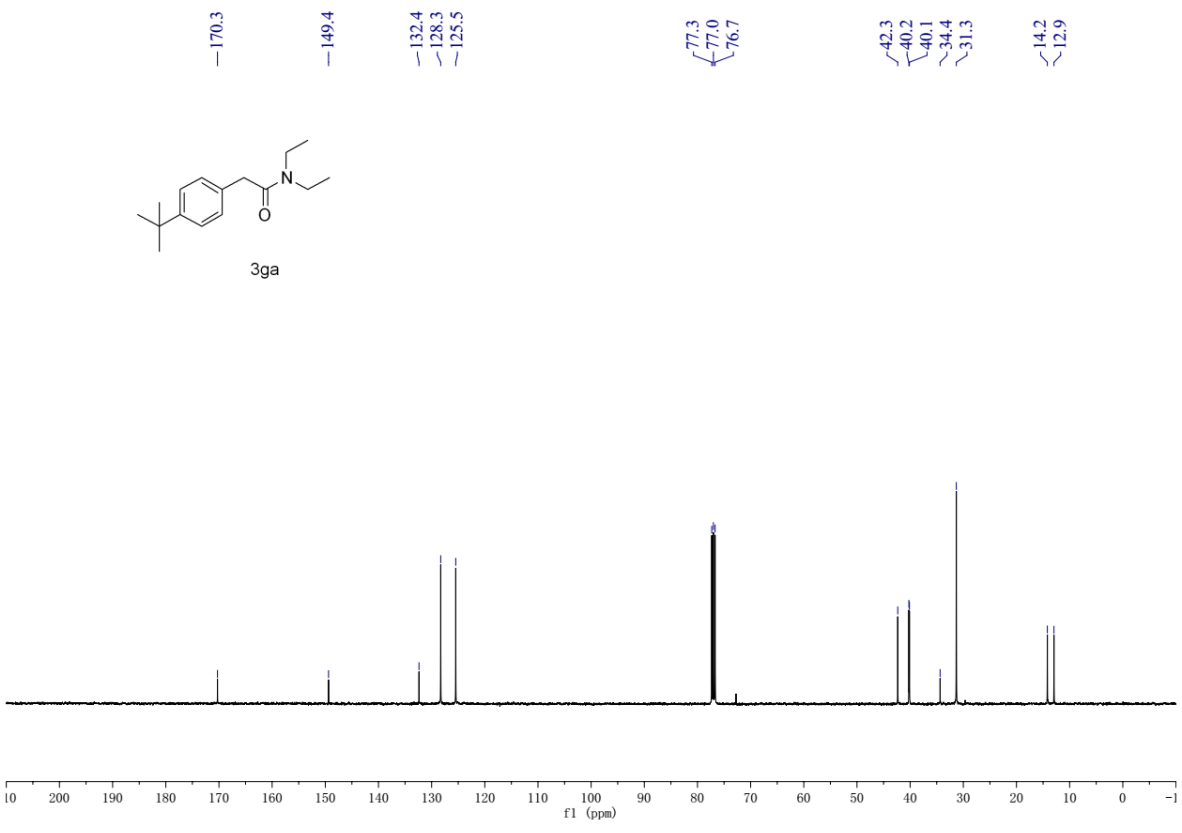

170.4
142.5
132.6
128.5
128.1
77.3
77.0
76.7
42.3
40.5
40.1
28.4
15.5
14.2
12.9
f1 (ppm)

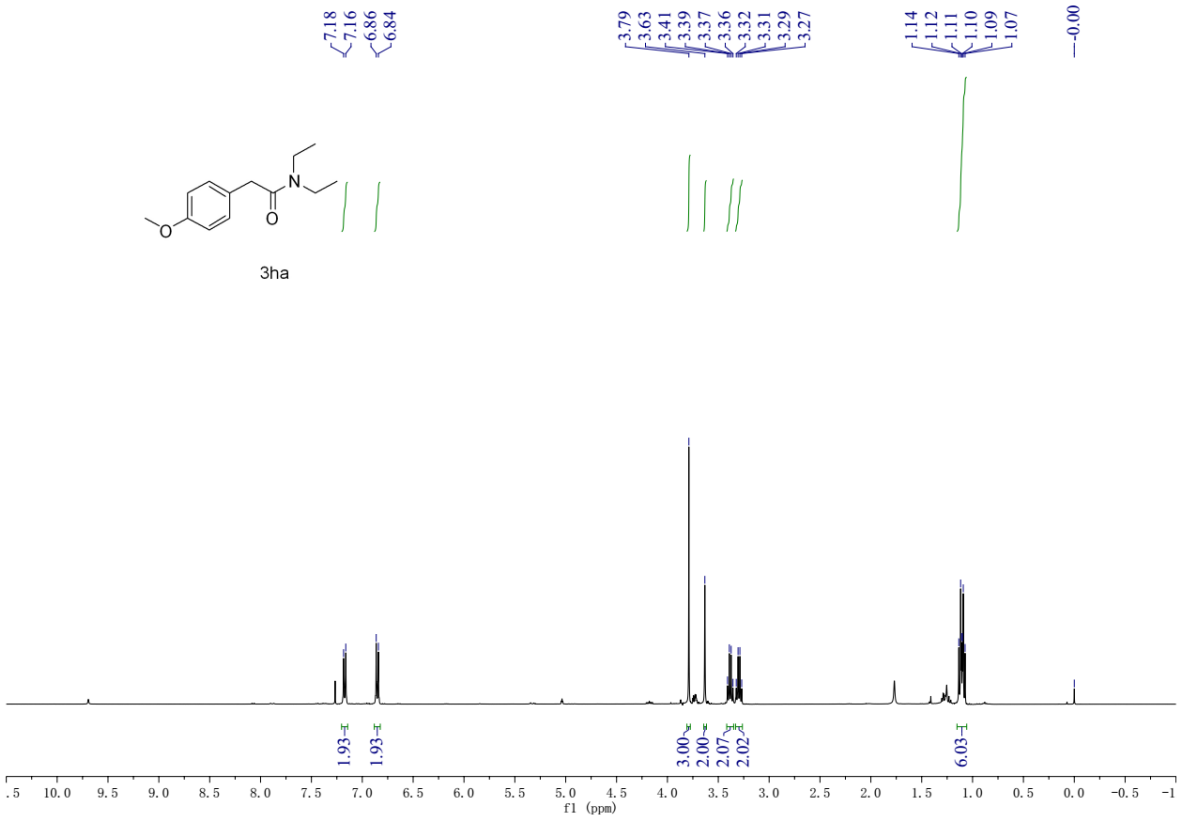
3ea

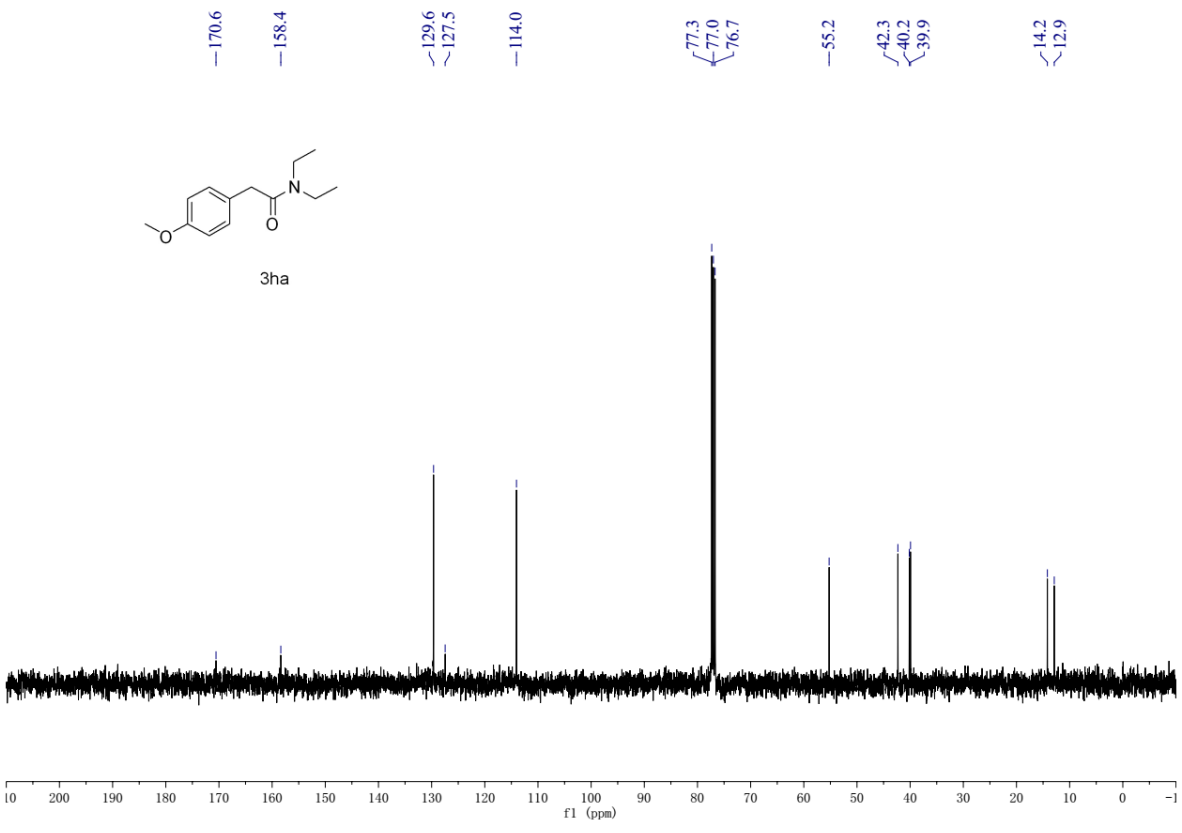

3fa
7.17
3.66
3.41
3.40
3.38
3.36
3.33
3.31
3.29
3.28
2.93
2.91
2.90
2.88
2.86
2.84
2.83
1.24
1.22
1.14
1.13
1.11
1.11
1.09
1.08
0.00
4.04
2.00
2.08
2.06
0.96
6.28
6.26
f1 (ppm)

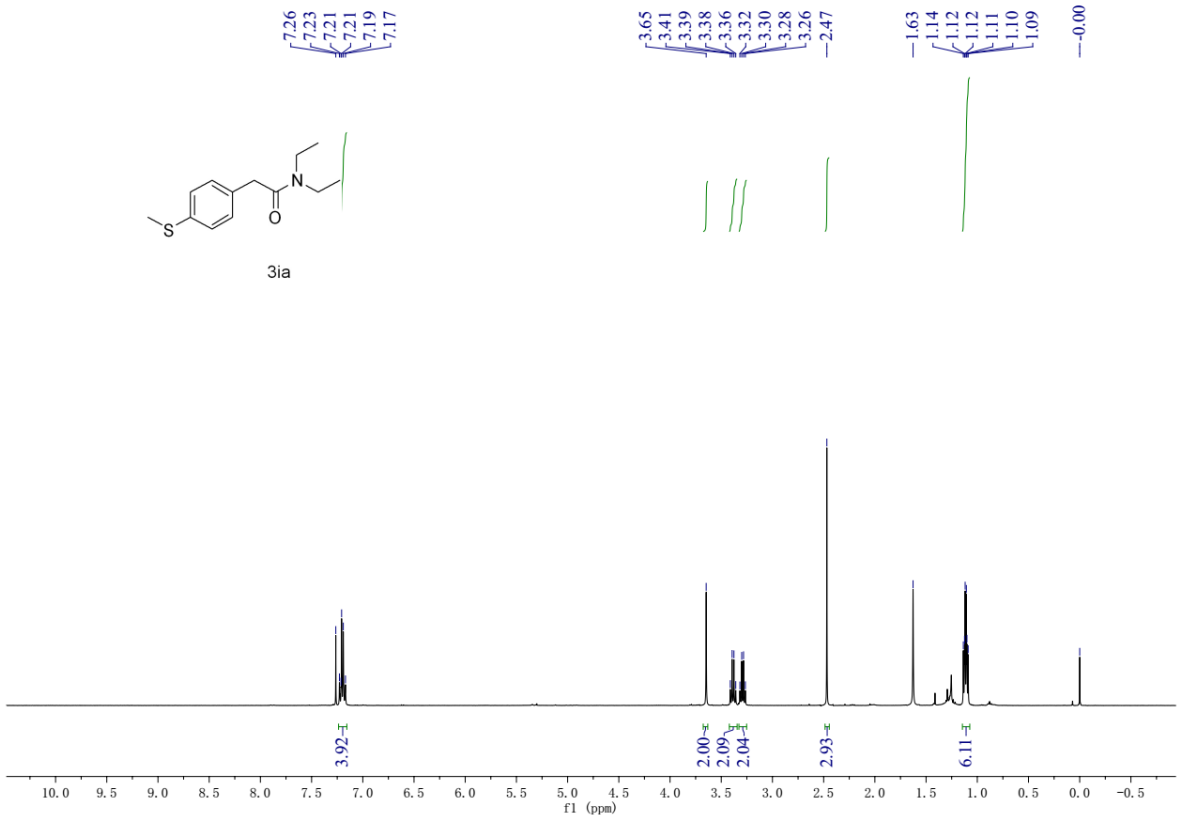

3fa
170.3
147.2
132.7
128.5
126.6
77.3
77.0
76.7
42.3
40.4
40.1
33.7
24.0
14.2
12.9
f1 (ppm)

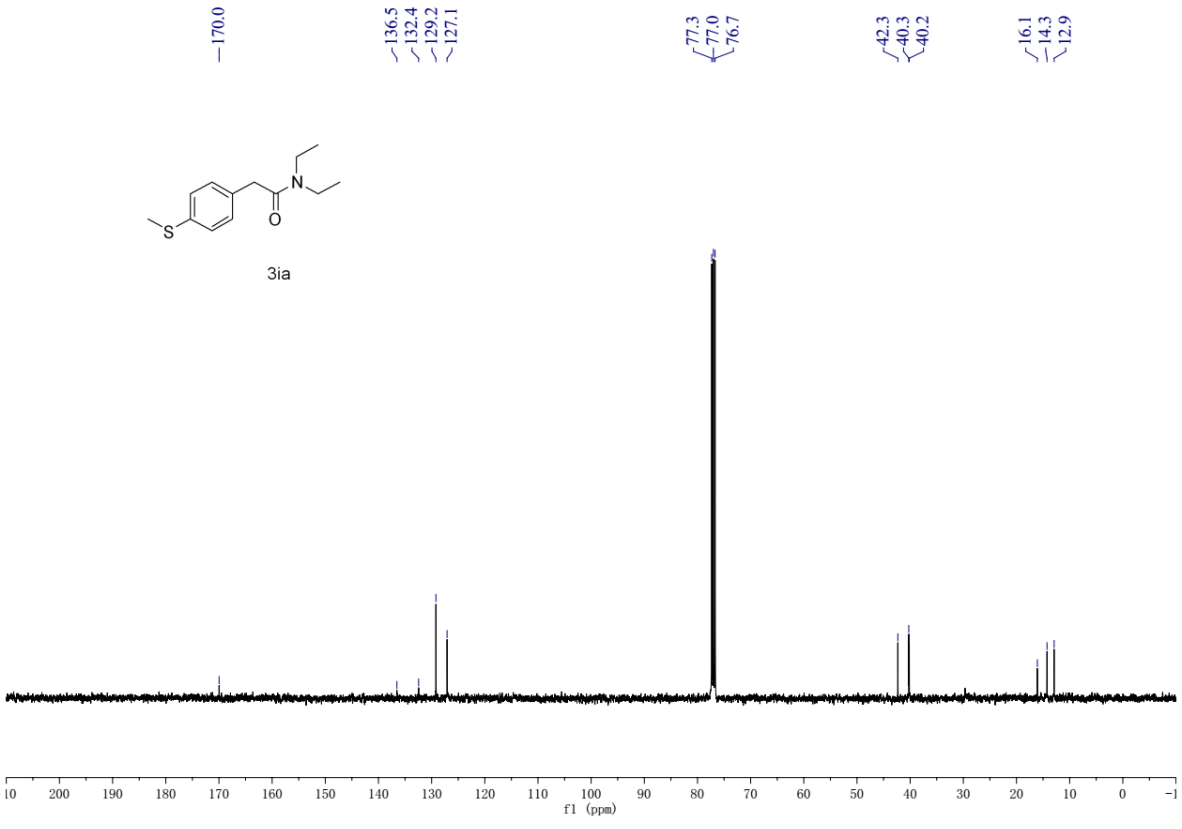

7.34
7.32
7.19
7.17
3.66
3.42
3.40
3.38
3.36
3.34
3.32
3.30
3.28
1.30
1.15
1.13
1.12
1.11
1.10
1.08
0.00
3ga
1.90
1.93
1.86
1.99
1.98
9.00
6.04
f1 (ppm)

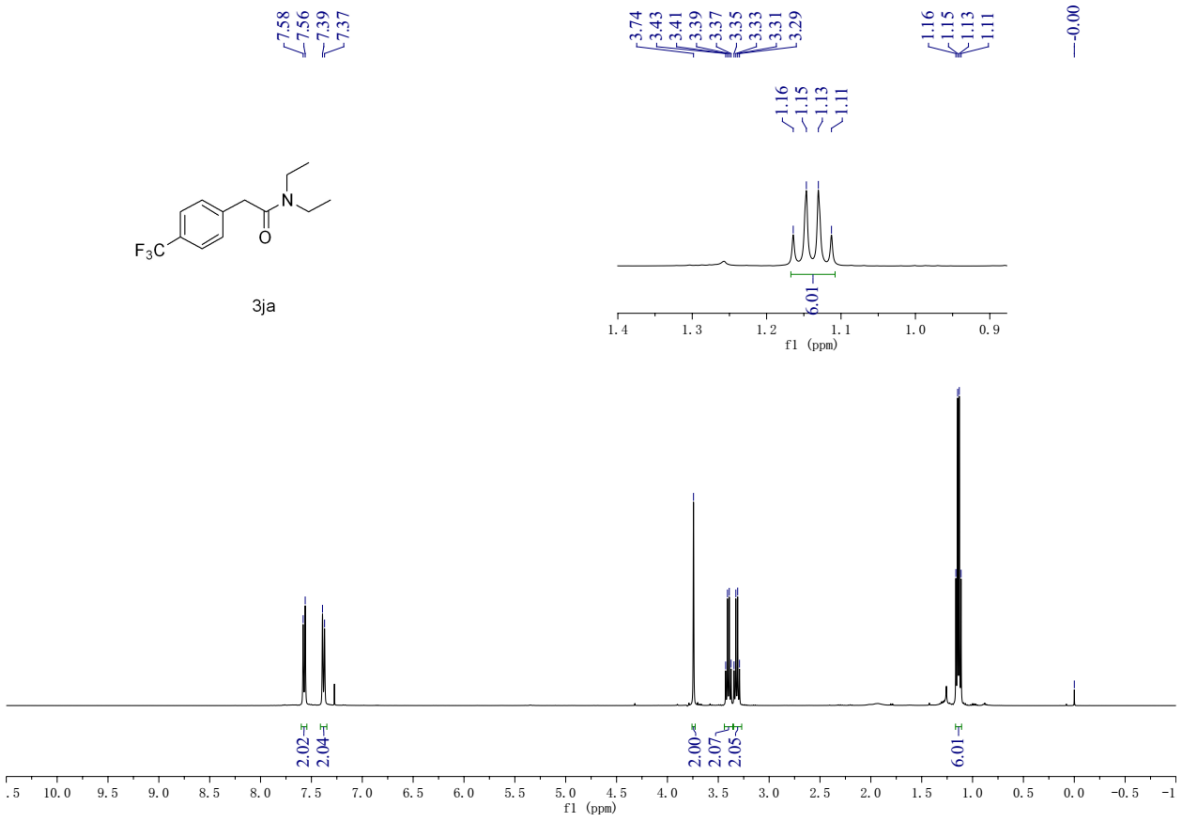

170.3
149.4
132.4
128.3
125.5
77.3
77.0
76.7
42.3
40.2
40.1
34.4
31.3
14.2
12.9
3ga
f1 (ppm)

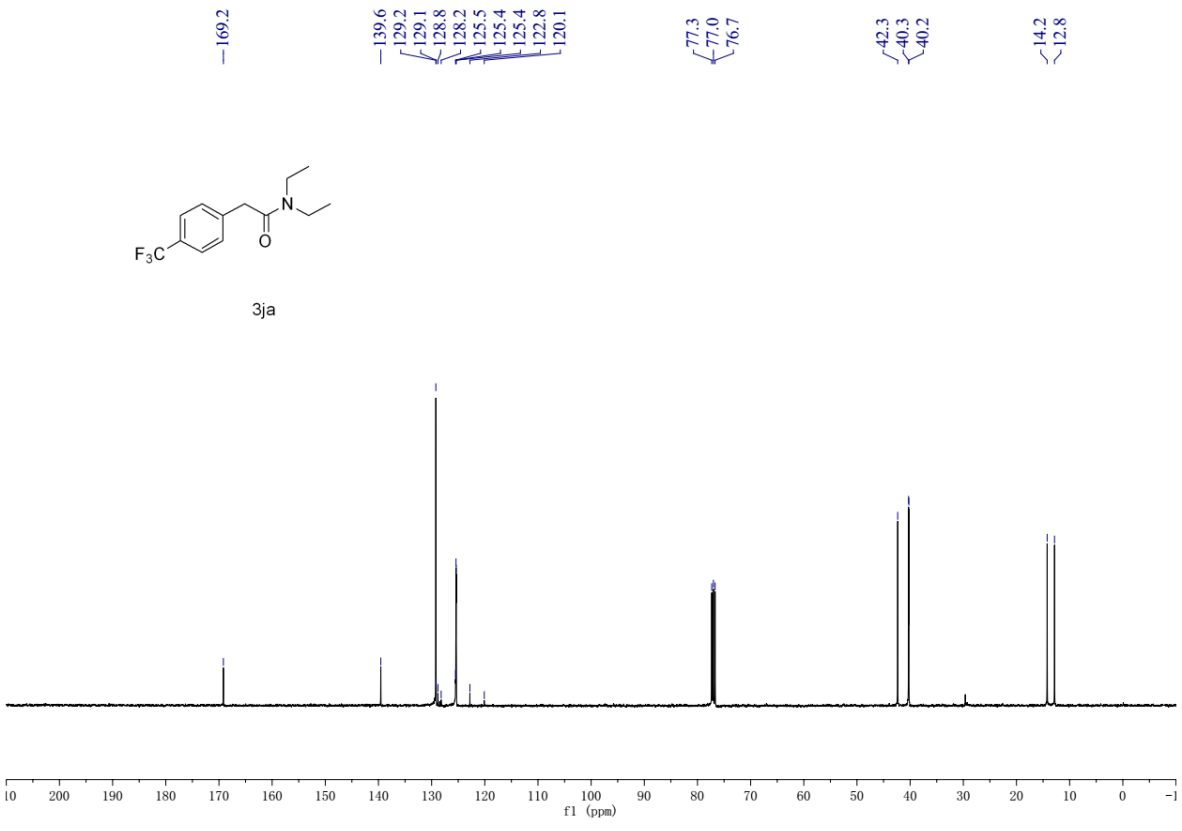

7.18
7.16
6.86
6.84
3.79
3.63
3.41
3.39
3.37
3.36
3.32
3.31
3.29
3.27
1.14
1.12
1.11
1.10
1.09
1.07
0.00
3ha
1.93
1.93
3.00
2.00
2.07
2.02
6.03
f1 (ppm)

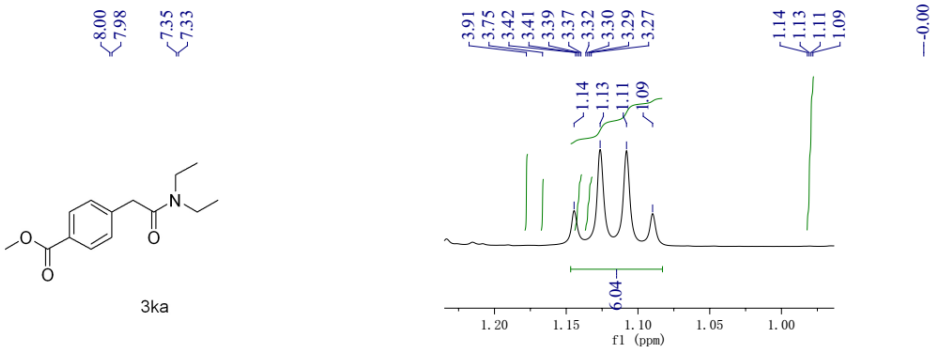

170.6
158.4
129.6
127.5
114.0
77.3
77.0
76.7
55.2
42.3
40.2
39.9
14.2
12.9
3ha
f1 (ppm)


7.26
7.23
7.21
7.21
7.19
7.17
3.65
3.41
3.39
3.38
3.36
3.32
3.30
3.28
3.26
2.47
1.63
1.14
1.12
1.12
1.11
1.10
1.09
0.00
N
O
S
3ia
3.92
2.00
2.09
2.04
2.93
6.11
f1 (ppm)

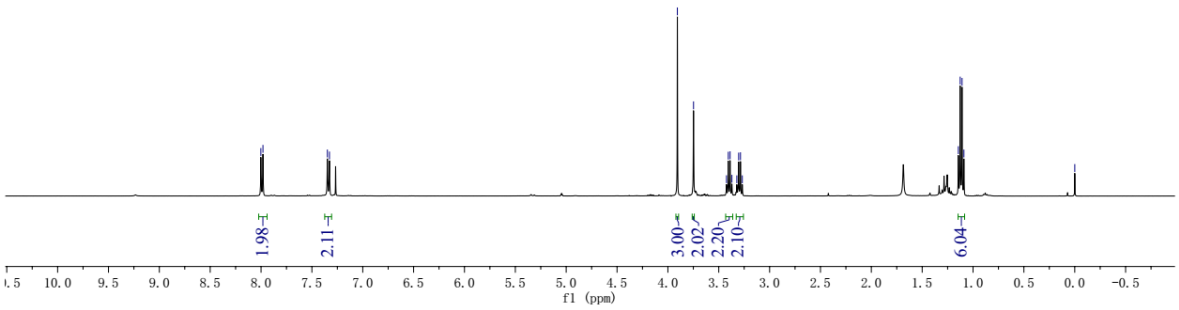

170.0
136.5
132.4
129.2
127.1
77.3
77.0
76.7
42.3
40.3
40.2
16.1
14.3
12.9
N
O
S
3ia
f1 (ppm)

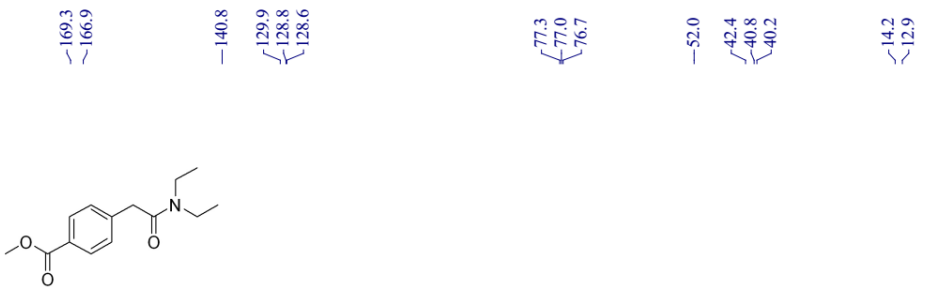

7.58
7.56
7.39
7.37
3.74
3.43
3.41
3.39
3.37
3.35
3.33
3.31
3.29
1.16
1.15
1.13
1.11
-0.00
F3C
N
O
3ja
6.01
f1 (ppm)
2.02
2.04
2.00
2.07
2.05
6.01
f1 (ppm)


169.2
139.6
129.2
129.1
128.8
128.2
125.5
125.4
125.4
122.8
120.1
77.3
77.0
76.7
42.3
40.3
40.2
14.2
12.8
F3C
N
O
3ja
f1 (ppm)

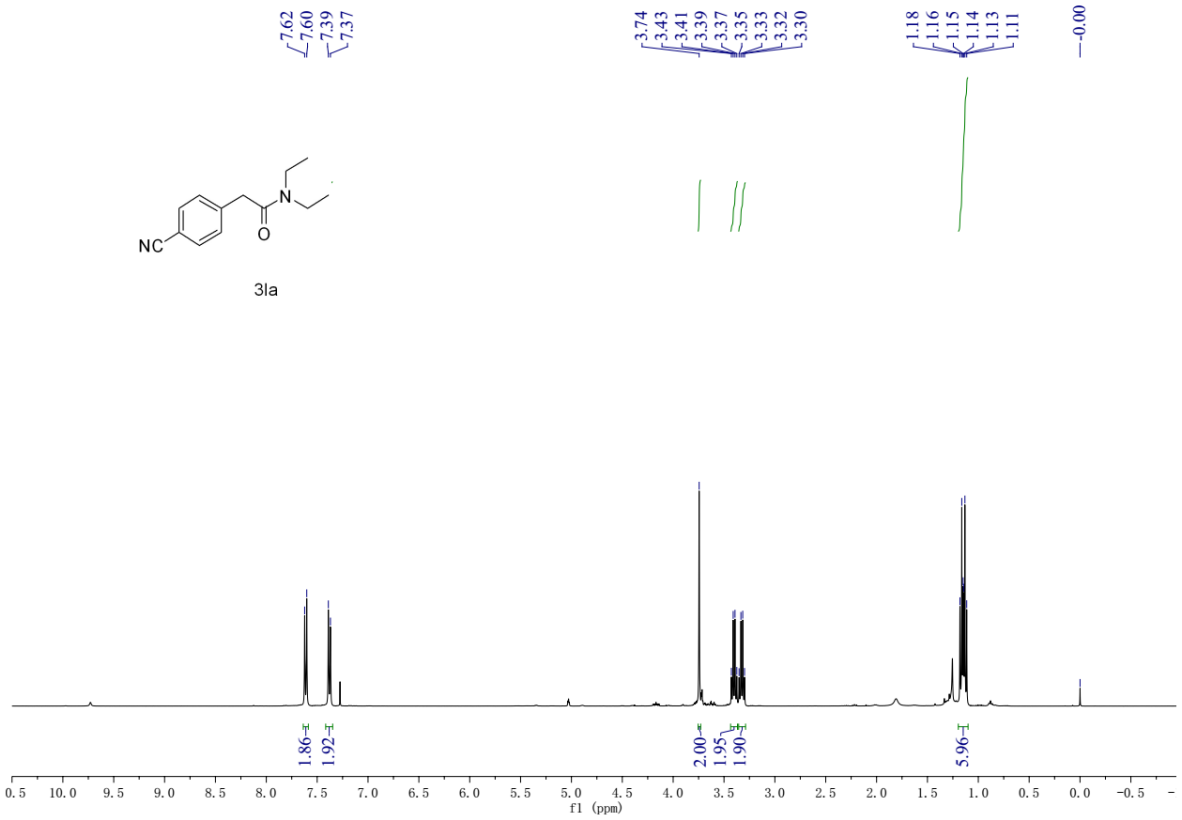

8.00
7.98
7.35
7.33
3.91
3.75
3.42
3.41
3.39
3.37
3.32
3.30
3.29
3.27
1.14
1.13
1.11
1.09
-0.00
6.04
f1 (ppm)

3ka

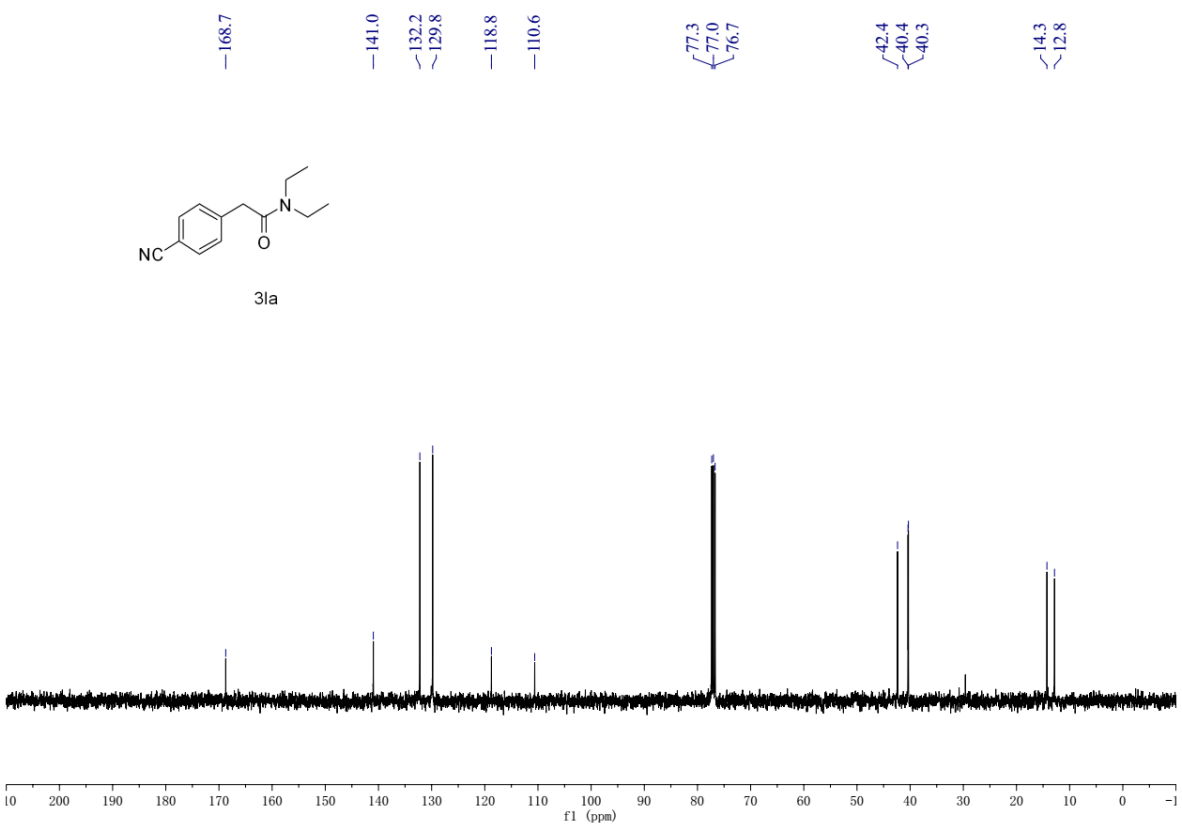

1.98
2.11
3.00
2.02
2.20
2.10
6.04
f1 (ppm)


169.3
166.9
140.8
129.9
128.8
128.6
77.3
77.0
76.7
52.0
42.4
40.8
40.2
14.2
12.9
f1 (ppm)

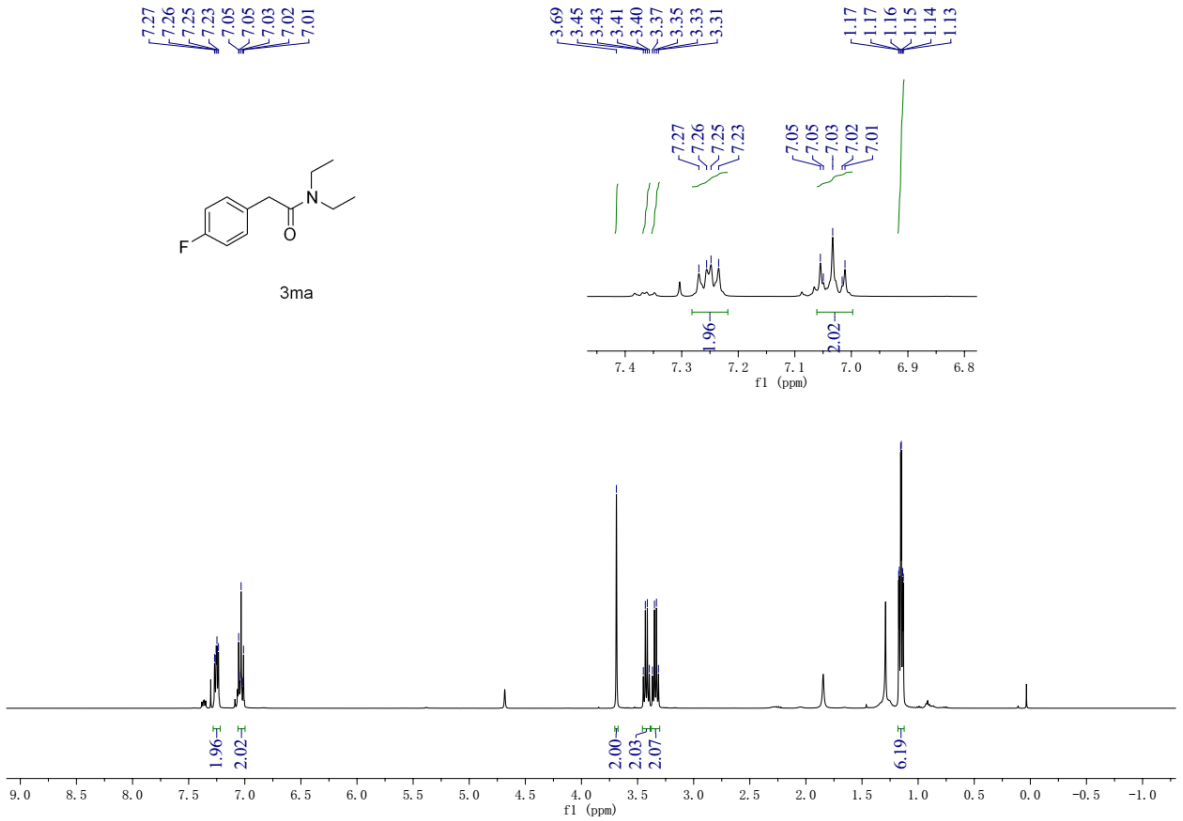
3ka

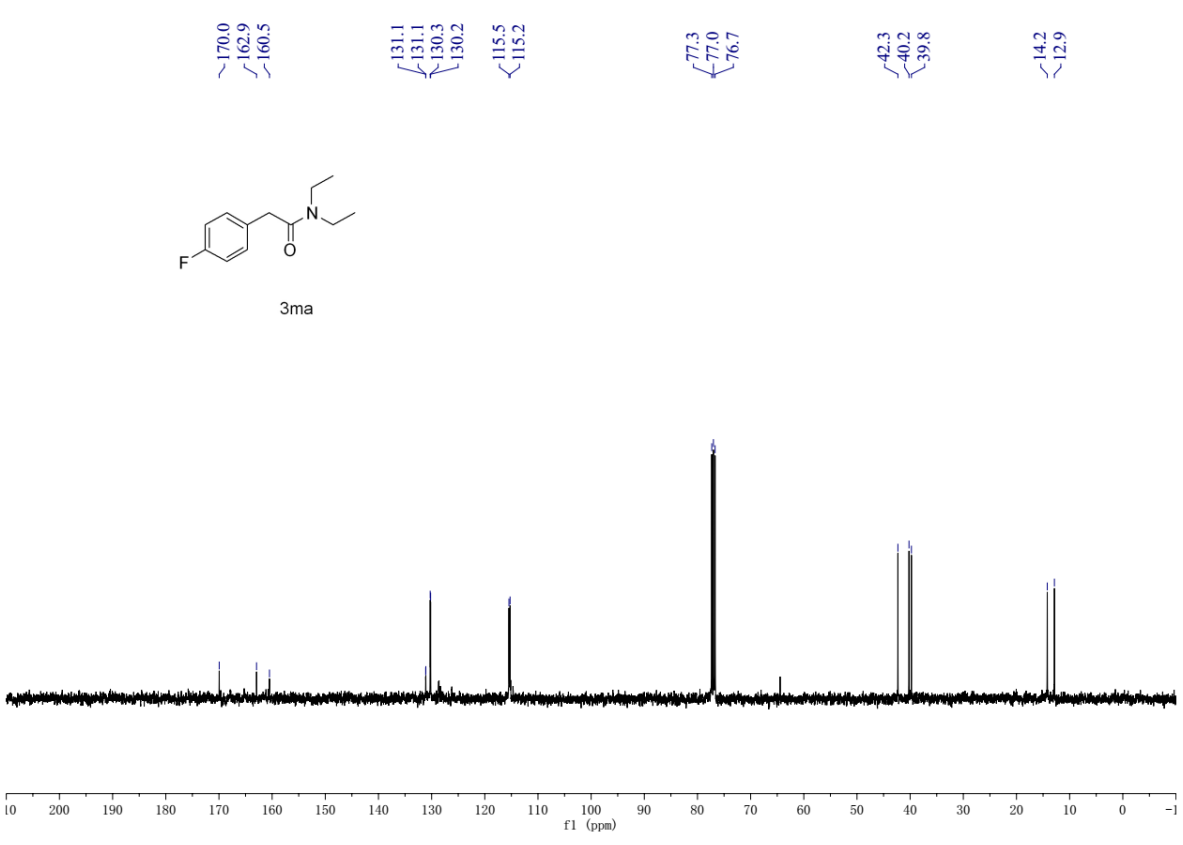

7.62
7.60
7.39
7.37
3.74
3.43
3.41
3.39
3.37
3.35
3.33
3.32
3.30
1.18
1.16
1.15
1.14
1.13
1.11
0.00
NC
O
N
1.86
1.92
2.00
1.95
1.90
5.96
f1 (ppm)

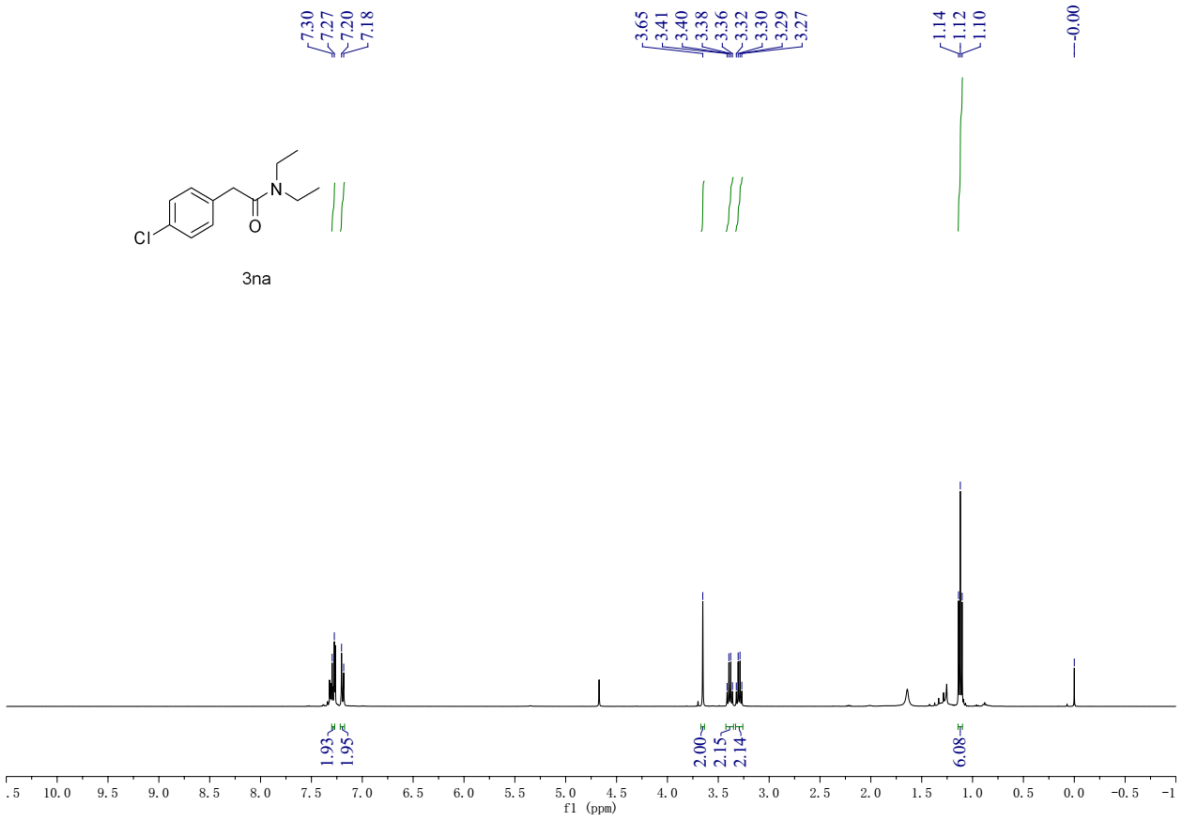
3la

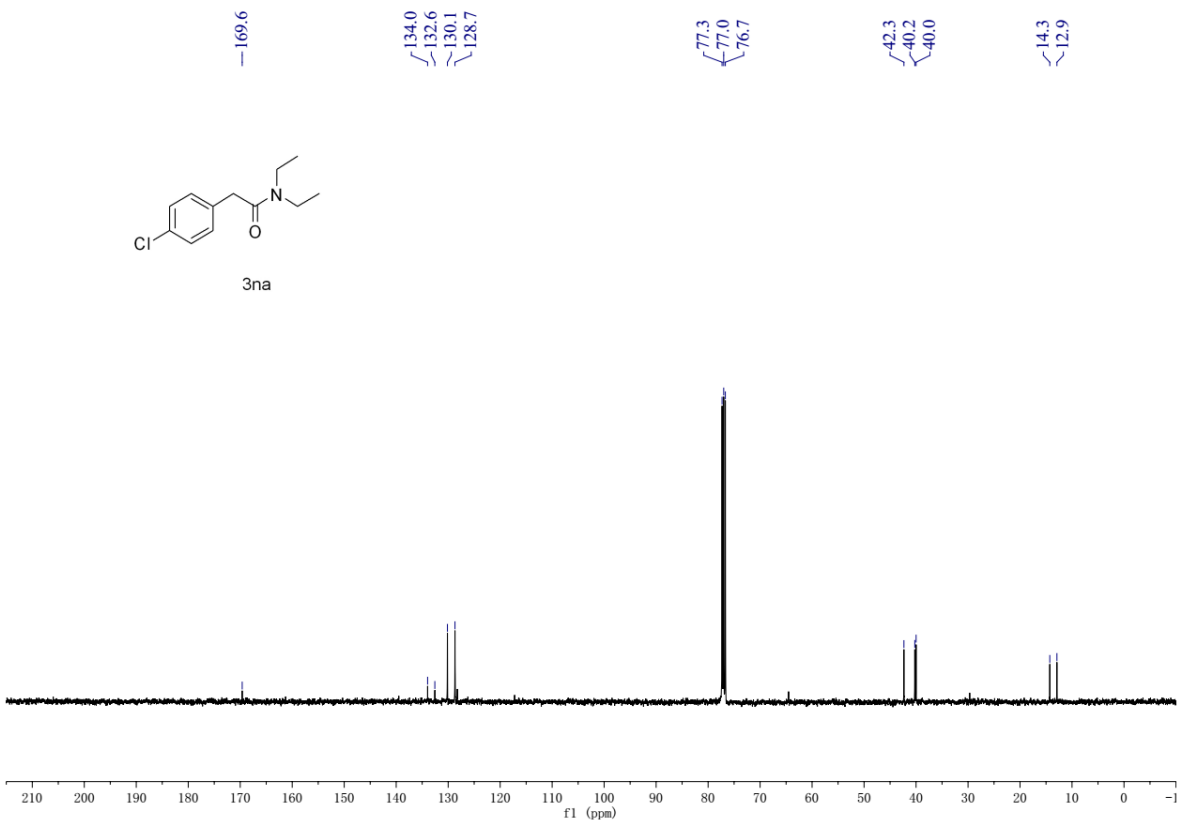

168.7
141.0
132.2
129.8
118.8
110.6
77.3
77.0
76.7
42.4
40.4
40.3
14.3
12.8
NC
O
N
f1 (ppm)

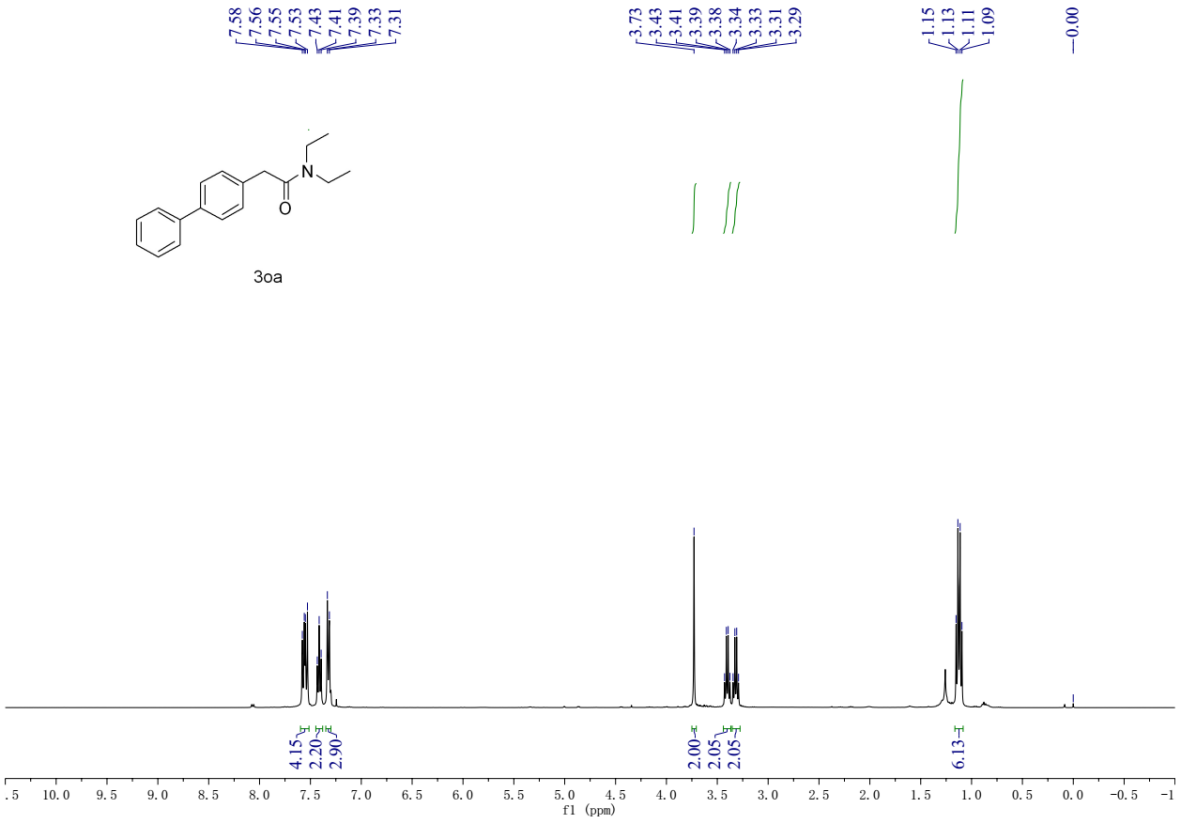
3la

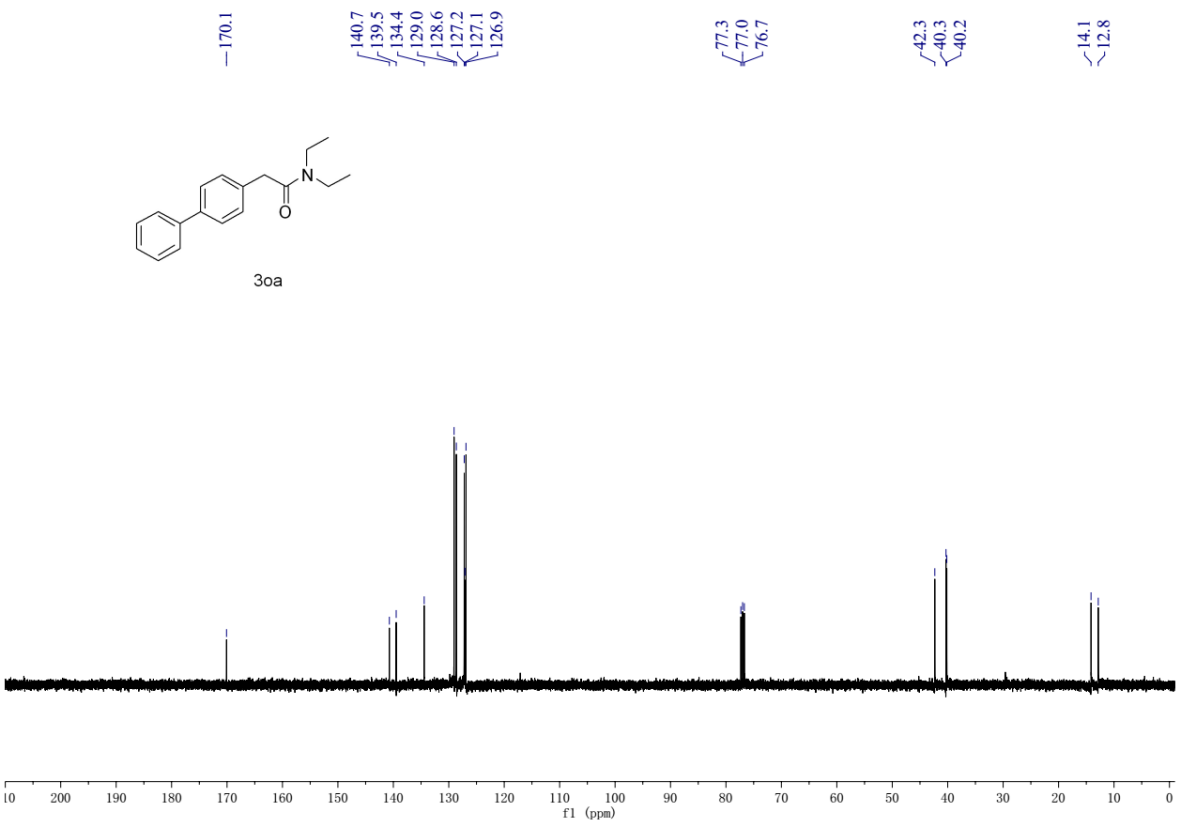

7.27
7.26
7.25
7.23
7.05
7.03
7.02
7.01
3.69
3.45
3.43
3.41
3.40
3.37
3.35
3.33
3.31
1.17
1.17
1.16
1.15
1.14
1.13
F
O
N
3ma
1.96
2.02
f1 (ppm)
2.00
2.03
2.07
6.19


170.0
162.9
160.5
131.1
131.1
130.3
130.2
115.5
115.2
77.3
77.0
76.7
42.3
40.2
39.8
14.2
12.9
F
O
N
3ma
f1 (ppm)


7.30
7.27
7.20
7.18
3.65
3.41
3.40
3.38
3.36
3.32
3.30
3.29
3.27
1.14
1.12
1.10
0.00
Cl
N
O
3na
1.93
1.95
2.00
2.15
2.14
6.08
f1 (ppm)


169.6
134.0
132.6
130.1
128.7
77.3
77.0
76.7
42.3
40.2
40.0
14.3
12.9
Cl
N
O
3na
f1 (ppm)


7.58
7.56
7.55
7.53
7.43
7.41
7.39
7.33
7.31
3.73
3.43
3.41
3.39
3.38
3.34
3.33
3.31
3.29
1.15
1.13
1.11
1.09
0.00
N
O
3oa
4.15
2.20
2.90
2.00
2.05
2.05
6.13
f1 (ppm)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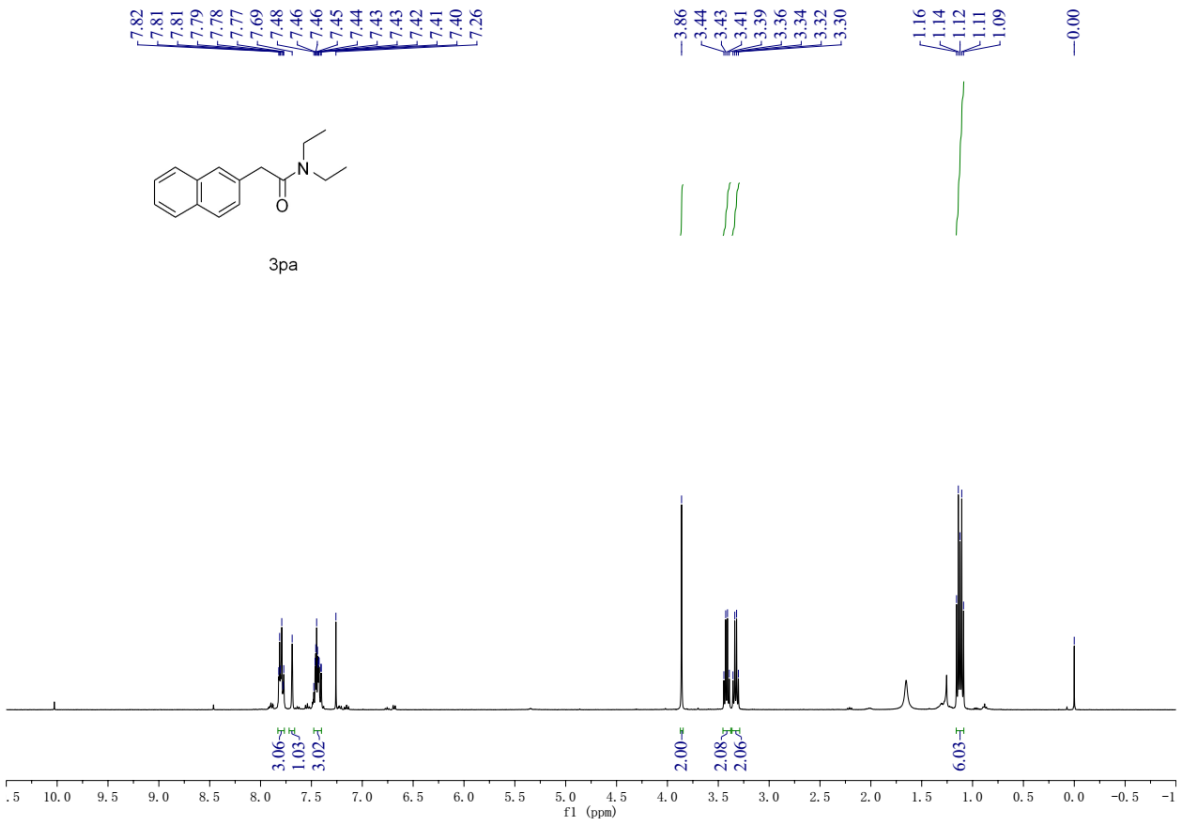
134.4
129.0
128.6
127.2
127.1
126.9
77.3
77.0
76.7
42.3
40.3
40.2
14.1
12.8
N
O
3oa
f1 (ppm)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7.48
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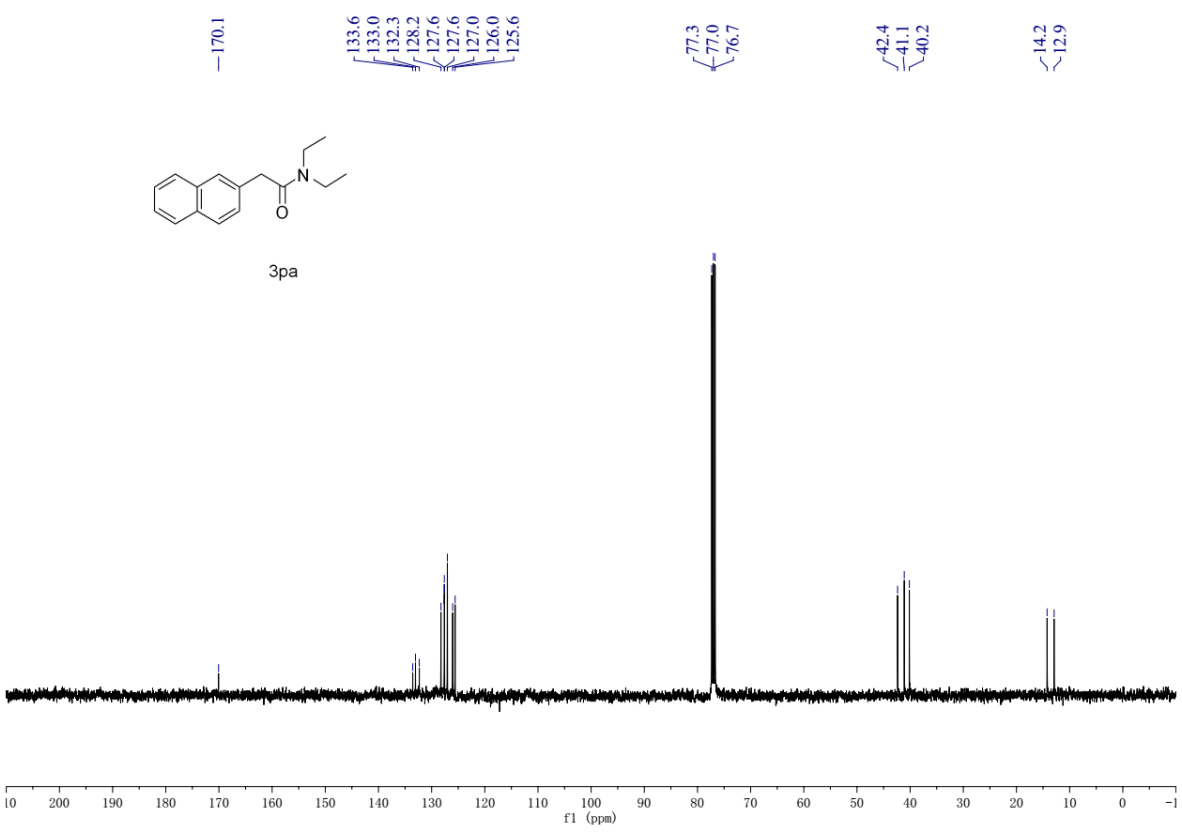
7.46
7.45
7.44
7.43
7.43
7.42
7.41
7.40
7.26
3.86
3.44
3.43
3.41
3.39
3.36
3.34
3.32
3.30
1.16
1.14
1.12
1.11
1.09
0.00
3pa
3.06
1.03
3.02
2.00
2.08
2.06
6.03
f1 (ppm)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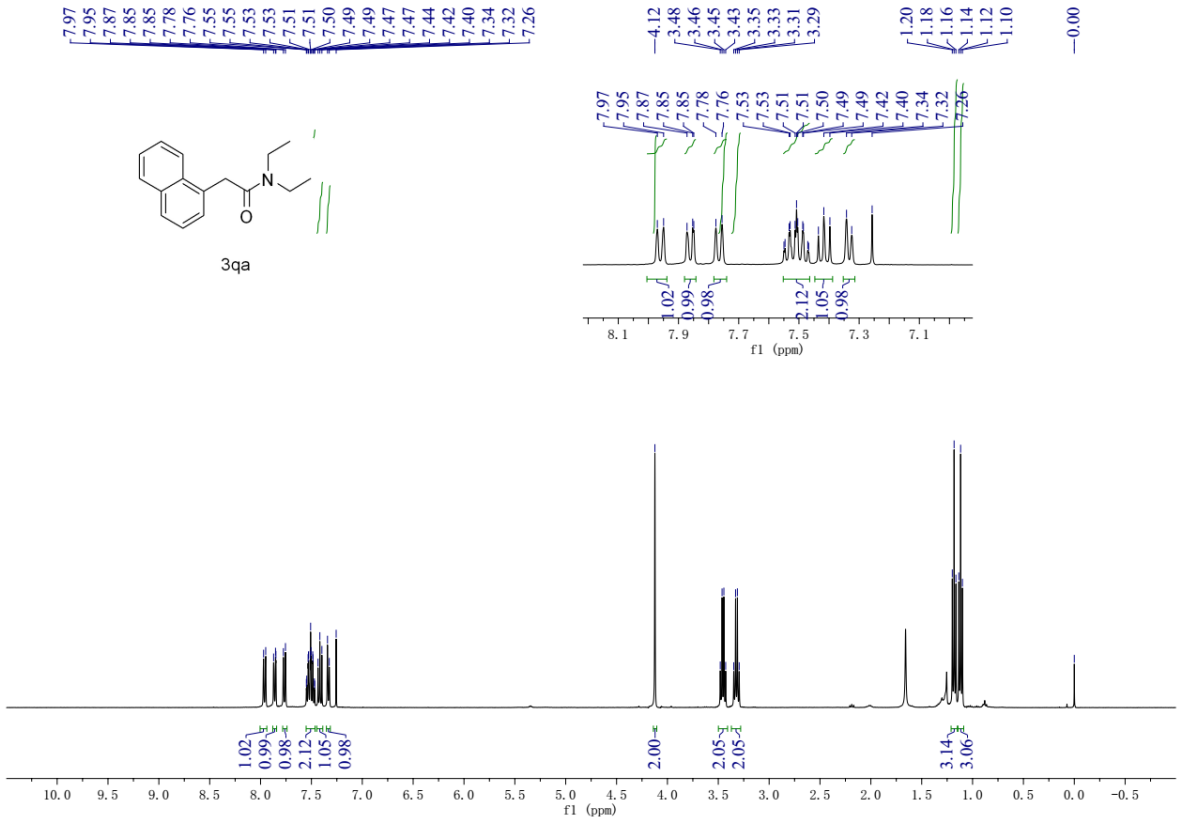
132.3
128.2
127.6
127.6
127.0
126.0
125.6
77.3
77.0
76.7
42.4
41.1
40.2
14.2
12.9
3pa
f1 (ppm)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7.76
7.55
7.55
7.53
7.53
7.51
7.51
7.50
7.49
7.49
7.47
7.47
7.44
7.42
7.40
7.34
7.32
7.26
4.12
3.48
3.46
3.45
3.43
3.35
3.33
3.31
3.29
1.20
1.18
1.16
1.14
1.12
1.10
0.00
N
O
3qa
1.02
0.99
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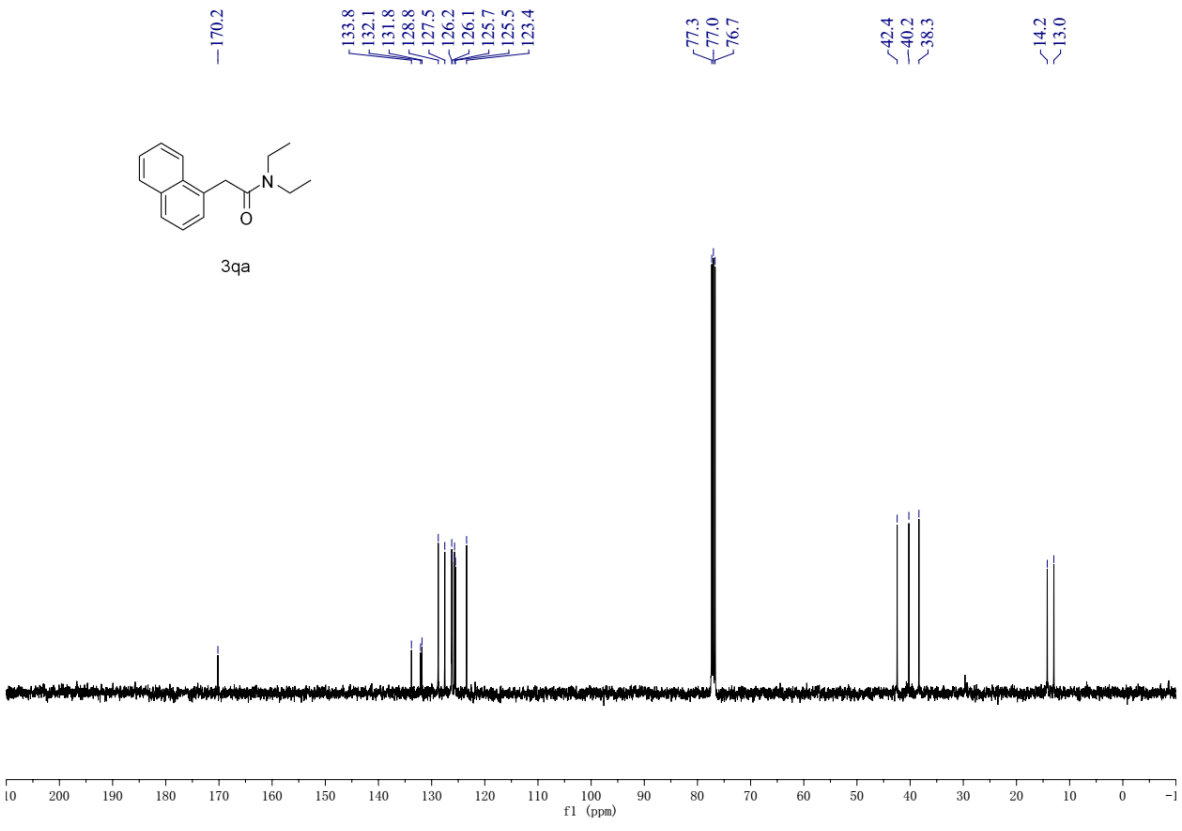
2.12
1.05
0.98
2.00
2.05
2.05
3.14
3.06
f1 (ppm)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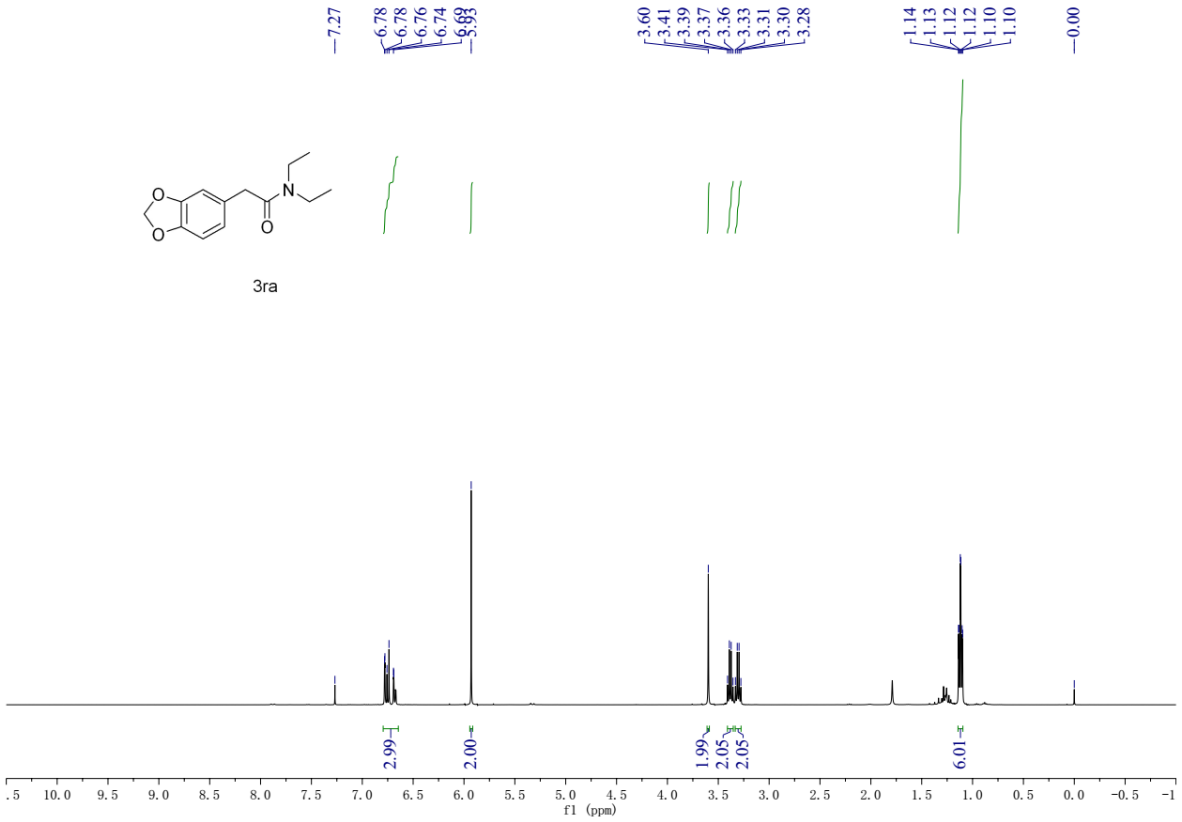
133.8
132.1
131.8
128.8
127.5
126.2
126.1
125.7
125.5
123.4
77.3
77.0
76.7
42.4
40.2
38.3
14.2
13.0
N
O
3qa
f1 (ppm)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6.78
6.78
6.76
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6.69
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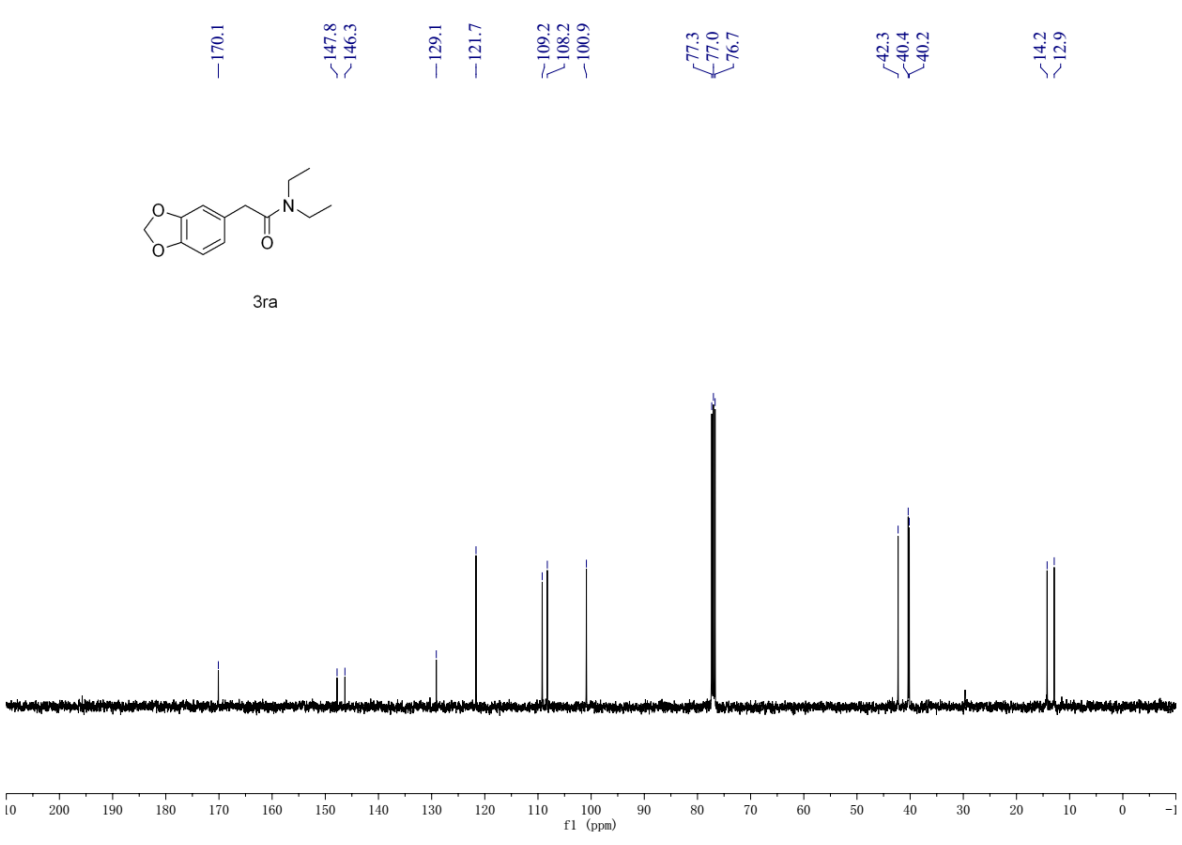
3.60
3.41
3.39
3.37
3.36
3.33
3.31
3.30
3.28
1.14
1.13
1.12
1.12
1.10
1.10
0.00
3ra
2.99
2.00
1.99
2.05
2.05
6.01
f1 (ppm)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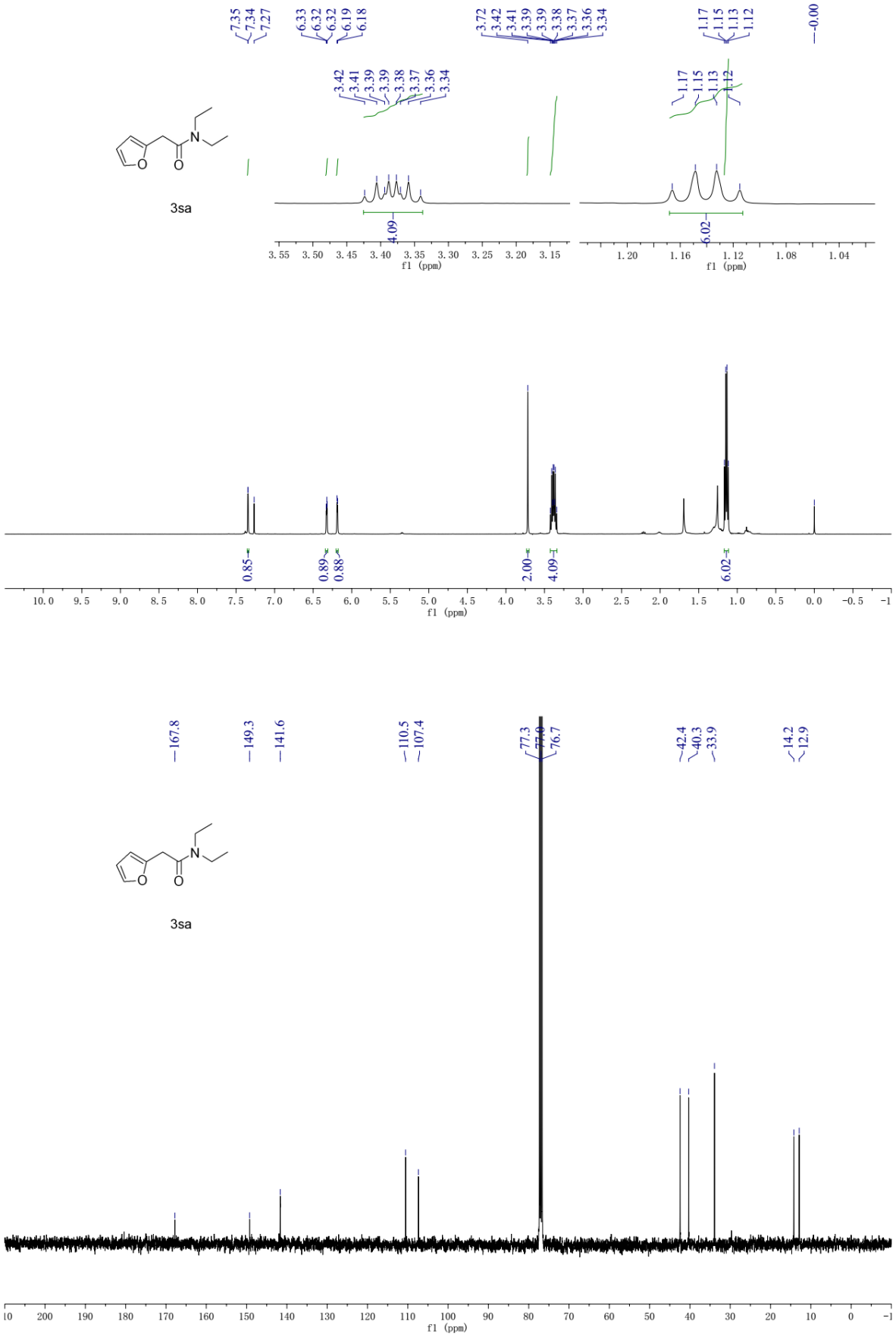
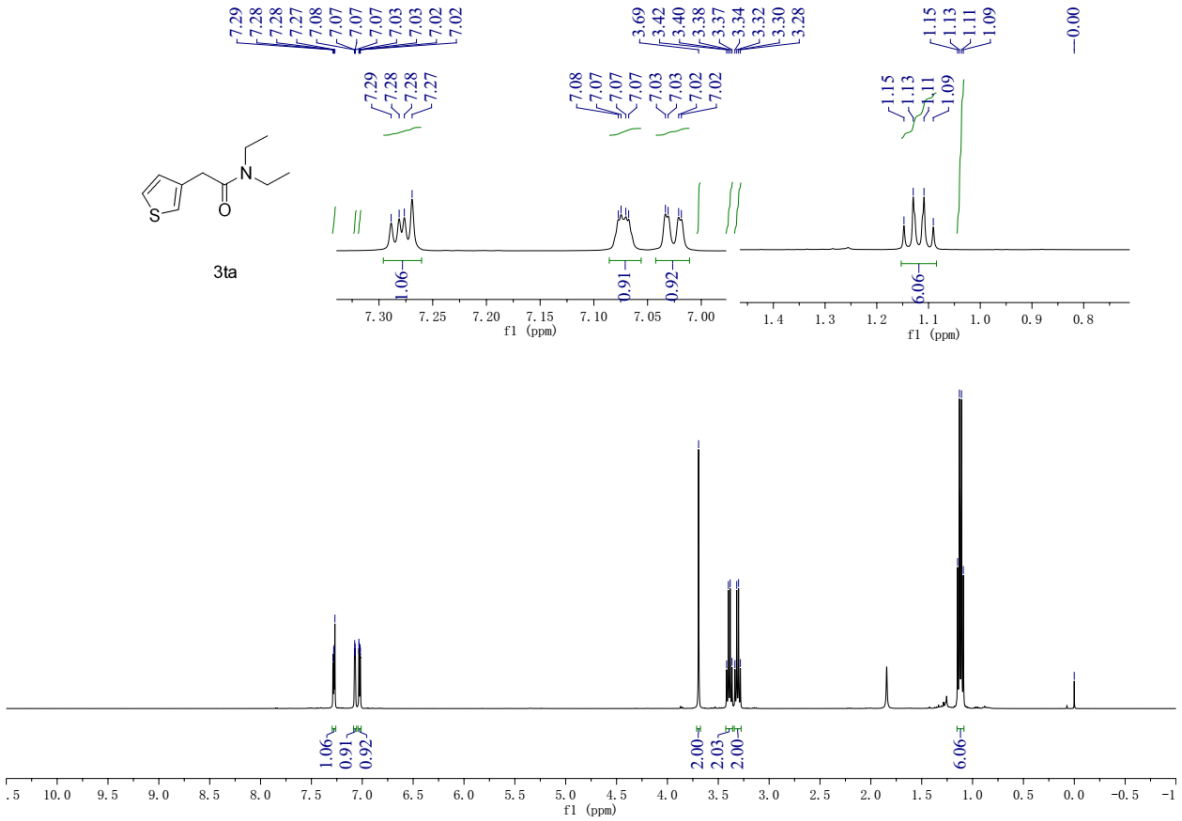
147.8
146.3
129.1
121.7
109.2
108.2
100.9
77.3
77.0
76.7
42.3
40.4
40.2
14.2
12.9
3ra
f1 (ppm)


3sa
3sa
f1 (ppm)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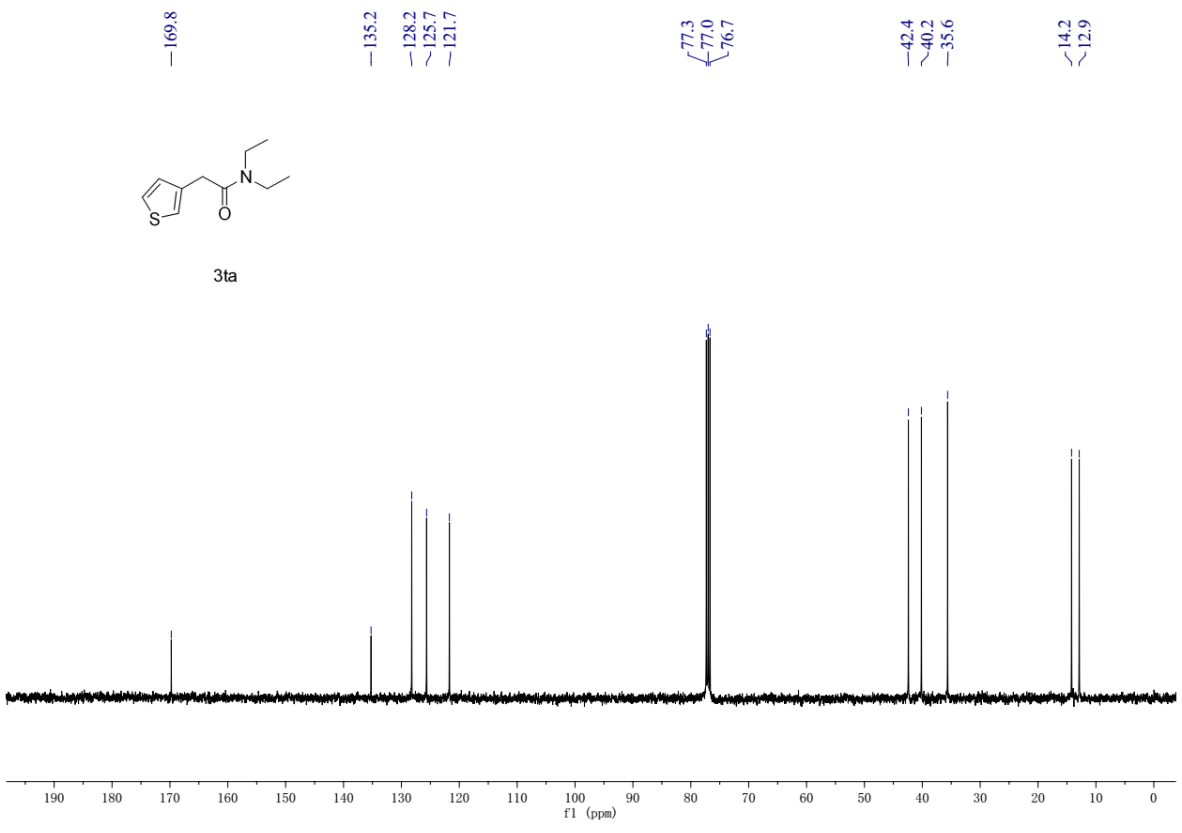
7.28
7.27
7.08
7.07
7.07
7.07
7.03
7.02
7.02
3.69
3.42
3.40
3.38
3.37
3.34
3.32
3.30
3.28
1.15
1.13
1.11
1.09
0.00
3ta
1.06
0.91
0.92
6.06
f1 (ppm)
1.06
0.91
0.92
2.00
2.03
2.00
6.06
f1 (ppm)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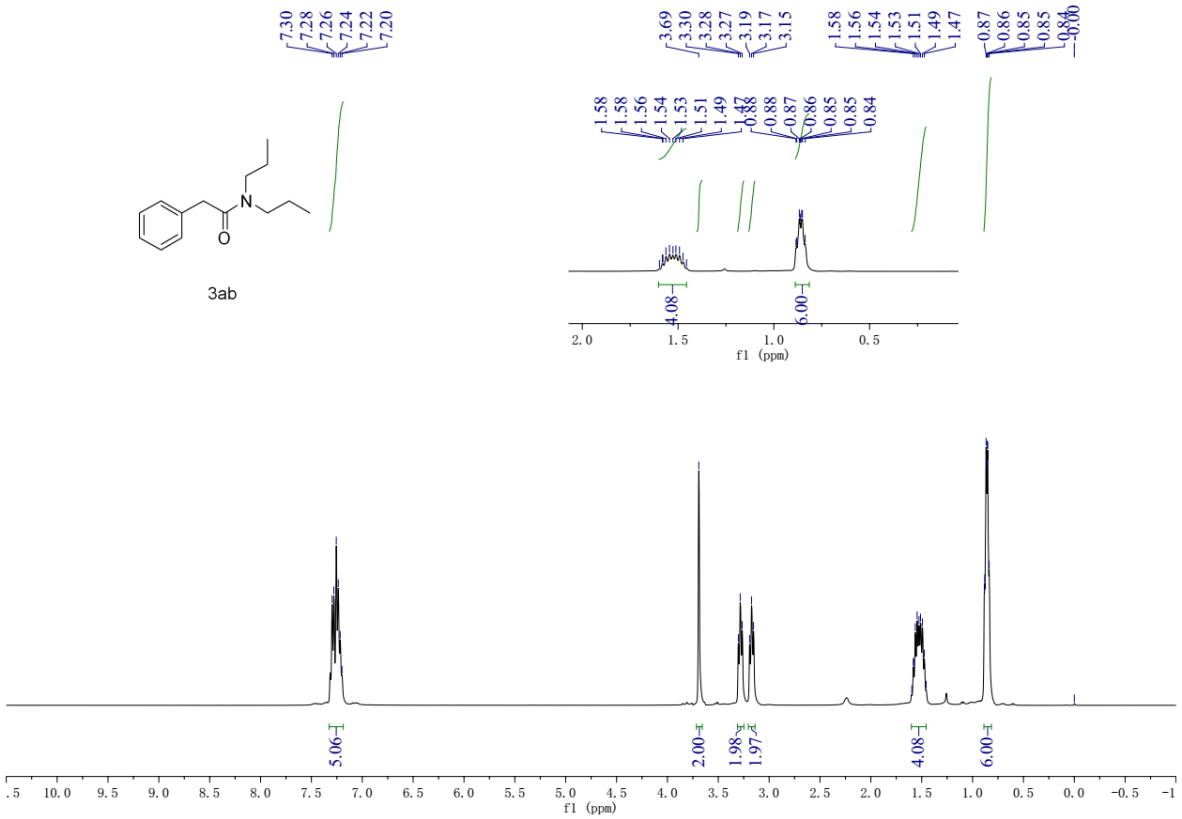
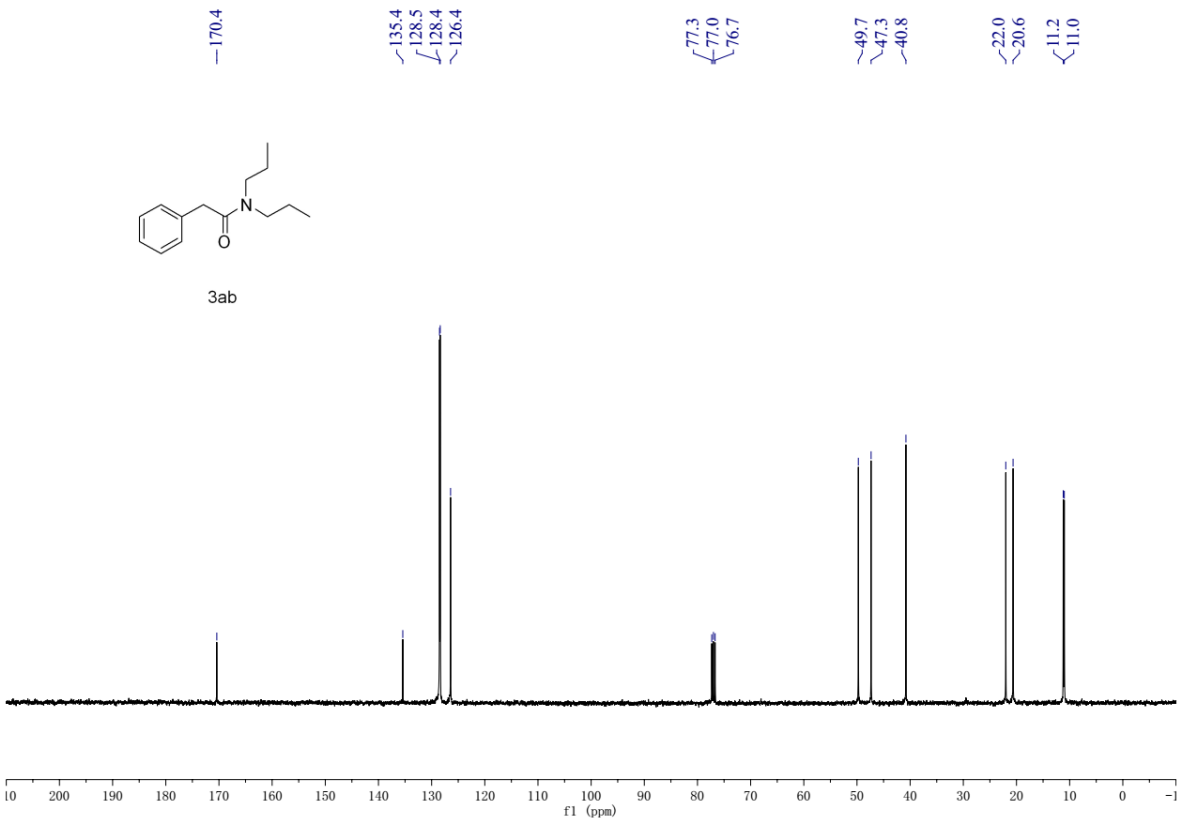
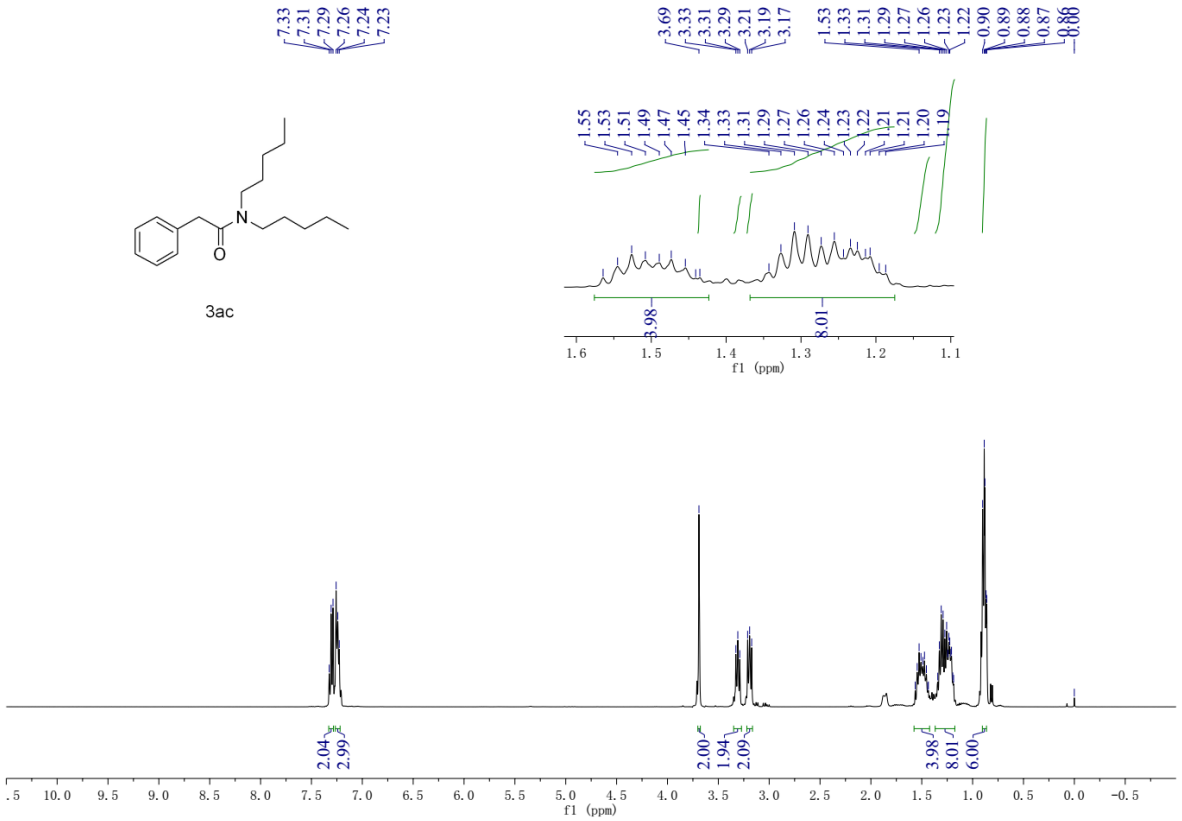
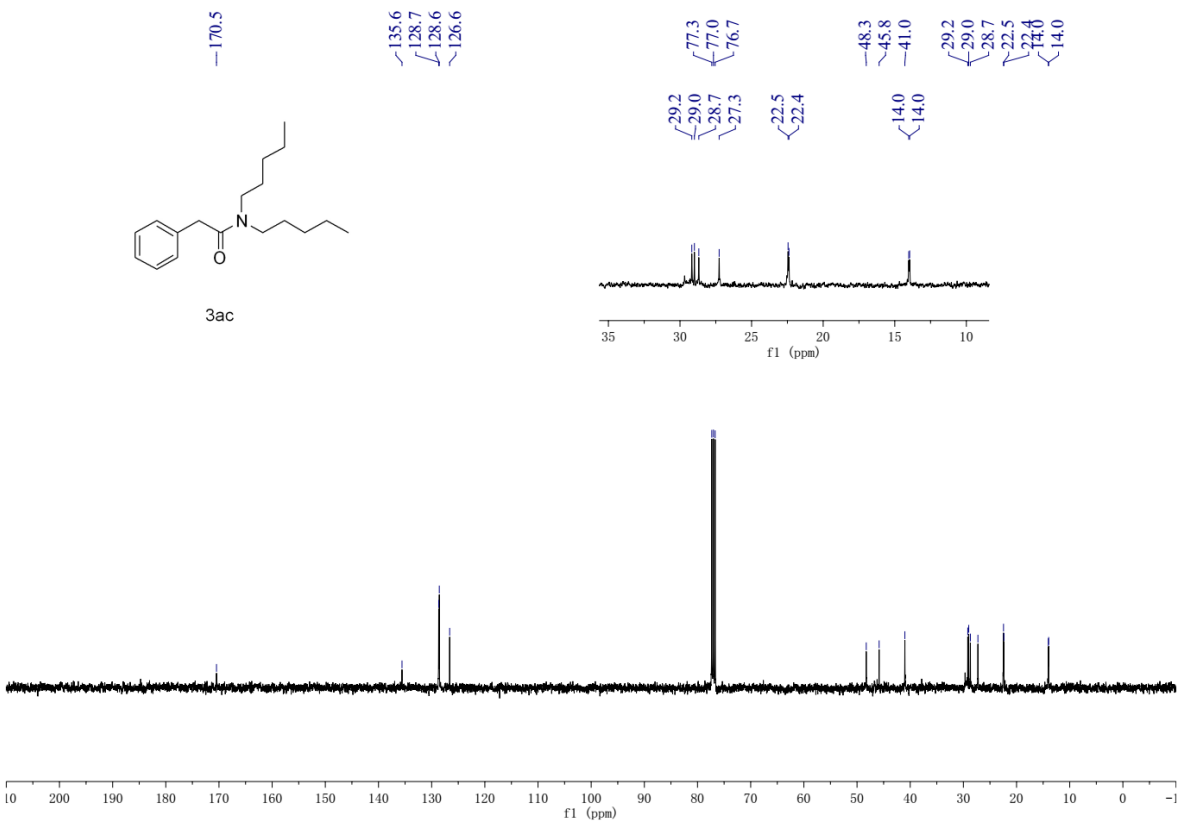
135.2
128.2
125.7
121.7
77.3
77.0
76.7
42.4
40.2
35.6
14.2
12.9
3ta
f1 (ppm)


3ab
f1 (ppm)


3ab
f1 (ppm)


7.33
7.31
7.29
7.26
7.24
7.23
3.69
3.33
3.31
3.29
3.21
3.19
3.17
1.53
1.33
1.31
1.29
1.27
1.26
1.23
1.22
0.90
0.89
0.88
0.87
1.55
1.53
1.51
1.49
1.47
1.45
1.34
1.33
1.31
1.29
1.27
1.26
1.24
1.23
1.22
1.21
1.21
1.20
1.19
3.98
8.01
1.6
1.5
1.4
1.3
1.2
1.1
f1 (ppm)
3ac
2.04
2.99
2.00
1.94
2.09
3.98
8.01
6.00
f1 (ppm)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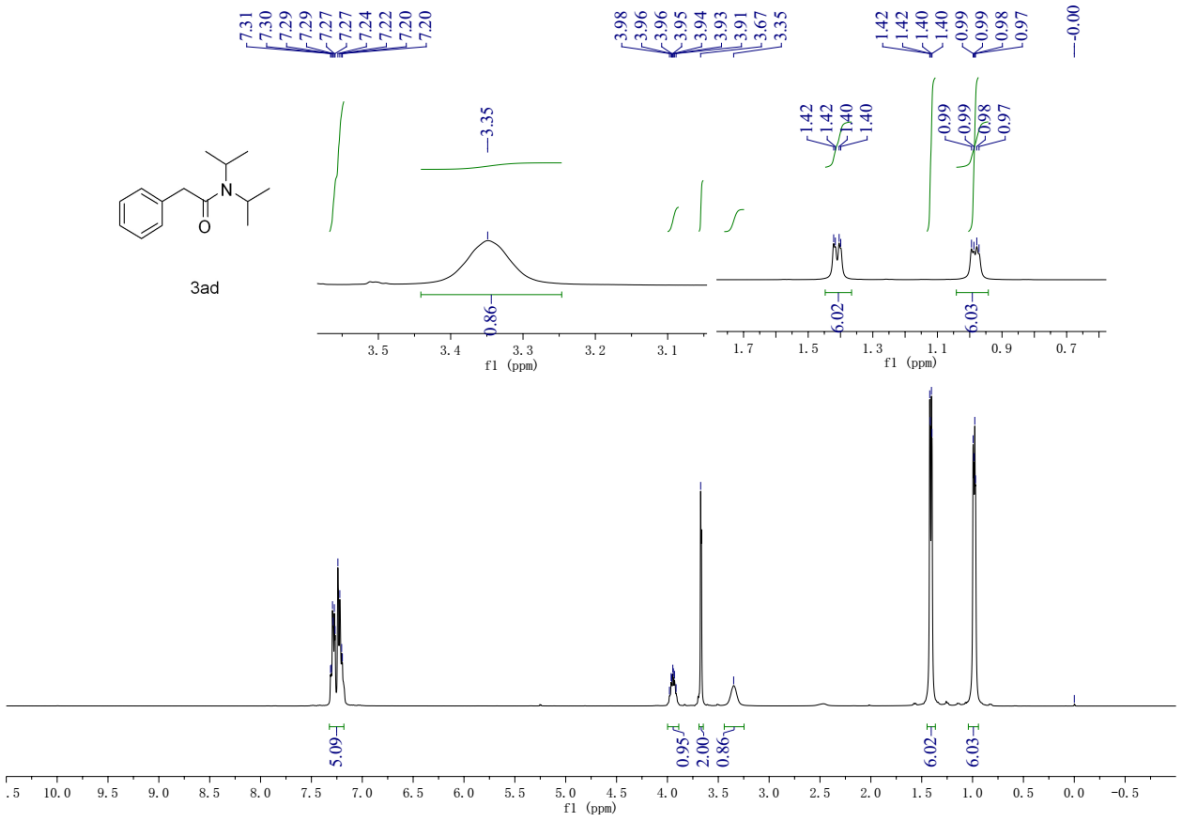
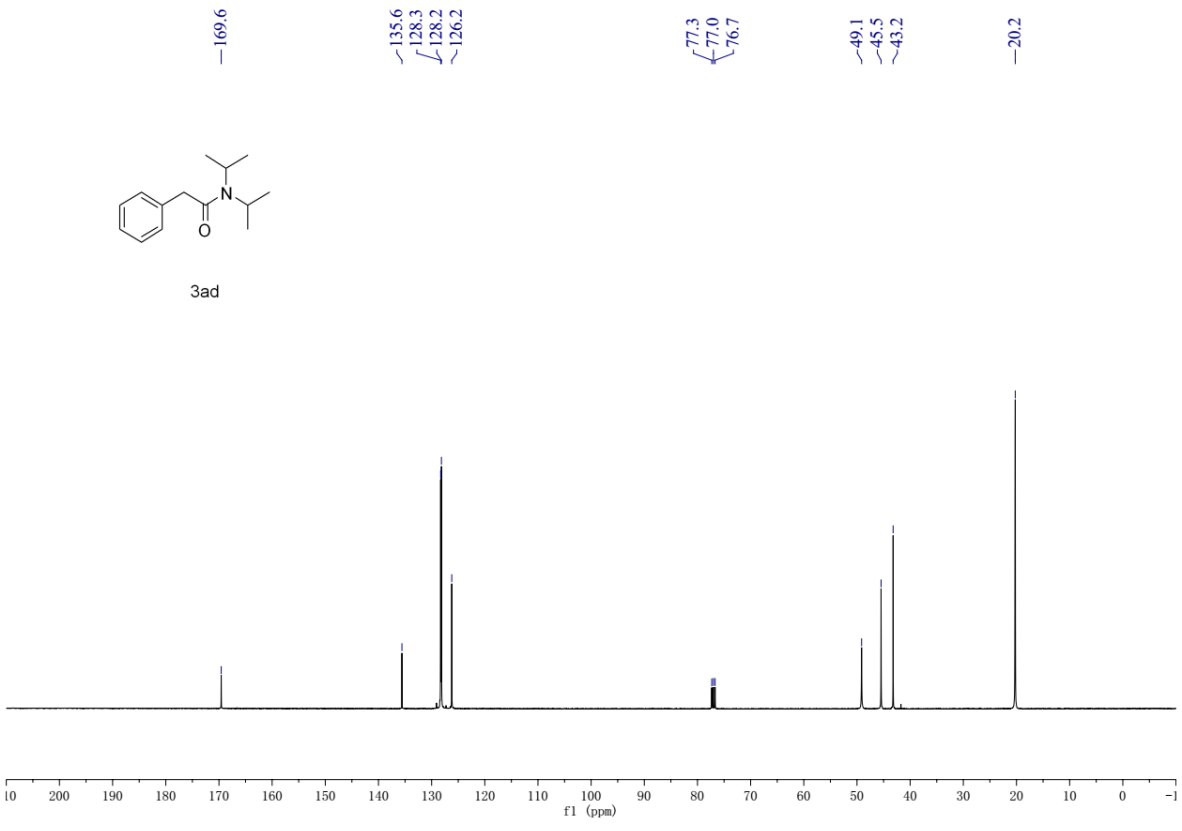
135.6
128.7
128.6
126.6
77.3
77.0
76.7
48.3
45.8
41.0
29.2
29.0
28.7
22.5
14.0
29.2
29.0
28.7
27.3
22.5
22.4
14.0
14.0
35
30
25
20
15
10
f1 (ppm)
3ac
f1 (ppm)


7.31
7.30
7.29
7.29
7.27
7.27
7.24
7.22
7.20
7.20
3.98
3.96
3.96
3.95
3.94
3.93
3.91
3.67
3.35
1.42
1.42
1.40
1.40
0.99
0.99
0.98
0.97
-0.00
3.35
0.86
6.02
6.03
f1 (ppm)
5.09
0.95
2.00
0.86
6.02
6.03
f1 (ppm)

3ad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135.6
128.3
128.2
126.2
77.3
77.0
76.7
49.1
45.5
43.2
20.2
f1 (ppm)

3ad