Electronic Supporting Information

Manganese(III) Acetate Catalyzed Oxidative Amination of Benzylic C(sp^3)–H Bonds with Nitriles

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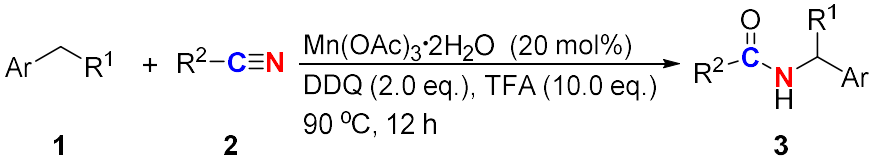
1. General Information

The reactions were carried out in schlenk tubes of 25 mL under N_2 atmosphere. Reagents were used as received unless otherwise noted, and solvents were purified according to standard operation procedure. Column chromatography was performed using Silica Gel 60 (300–400 mesh). The reactions were monitored by GC-MS and GC. GC-MS results were recorded on GC-MS QP 2010, and GC analysis was performed on GC 2010 plus. The ^{1}H and ^{13}C NMR spectra were recorded on a Brucker ADVANCE III spectrometer at 400 MHz and 100 MHz respectively, and chemical shifts were reported in parts per million (ppm). The electron ionization (EI) method was used as the ionization method for the HRMS measurement, and the mass analyzer type is TOF for EI. All solvents and reagents were purchased from Energy Chemical, Alfa Aesar, and Aladdin.

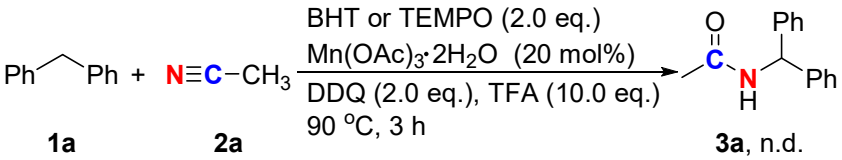
2. Experimental Procedure

a) General Experimental Procedure for the Synthesis of Amides



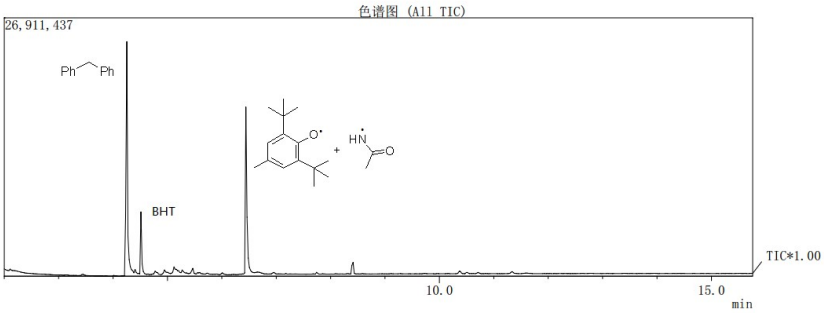
An oven-dried 25 mL Schlenk tube, which was equipped with a magnetic stir bar and charged with $Mn(OAc)_3 \cdot 2H_2O$ and DDQ. After charging nitrogen for three times, TFA (10.0 equiv.), benzylic compound **1** (0.2 mmol), nitrile **2**, and solvent were added. Then the reaction mixture was heated to 90 ℃ for 12 h. After completion of the reaction, the reaction mixture was cooled down to room temperature, and neutralized by K_2CO_3. The crude product was extracted with diethyl ether, dried over anhydrous Na_2SO_4, and concentrated under vacuum. The residue was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate to afford the desired product **3**.

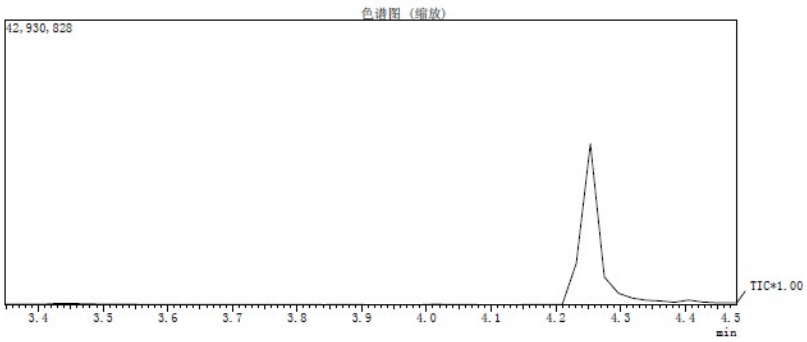
b) **Control Experiments**

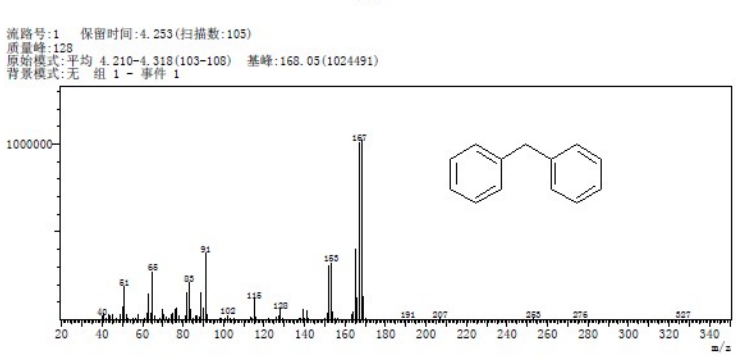


1a **2a** **3a**, n.d.

An oven-dried 25 mL Schlenk tube, which was equipped with a magnetic stir bar and charged with $Mn(OAc)_3 \cdot 2H_2O$ (20 mol%), DDQ (2.0 equiv.) and BHT or TEMPO (2.0 equiv.). After charging nitrogen for three times, TFA (10.0 equiv.), diphenylmethane **1a** (0.2 mmol), acetonitrile **2a** (0.5 mL) were added. Then the reaction mixture was heated to 90 ℃ for 3 h, the desired product was not observed, but the radical adduct of BHT with acetamide was observed. These results suggested that free-radical was involved in the reaction. The GC-MS spectra are as follows:







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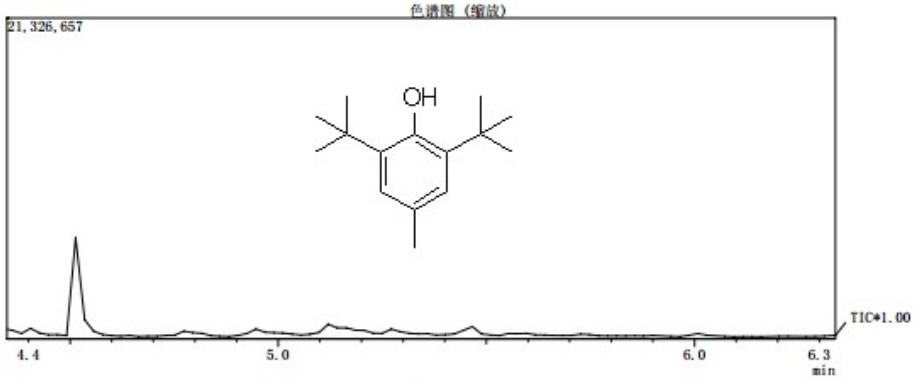

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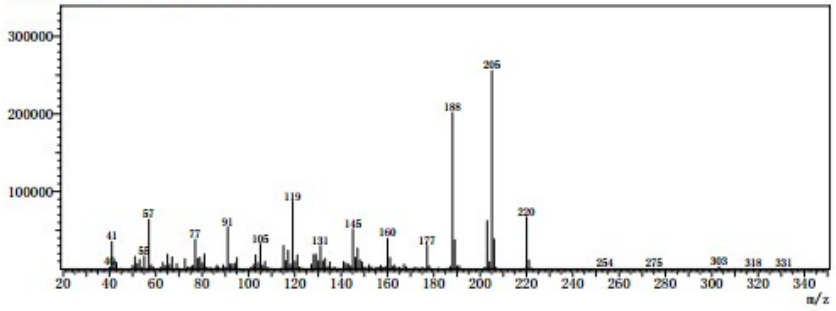

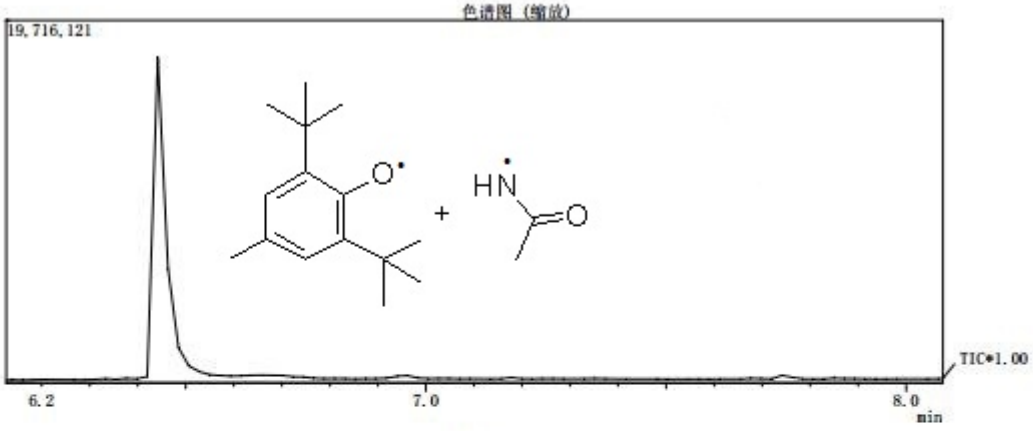

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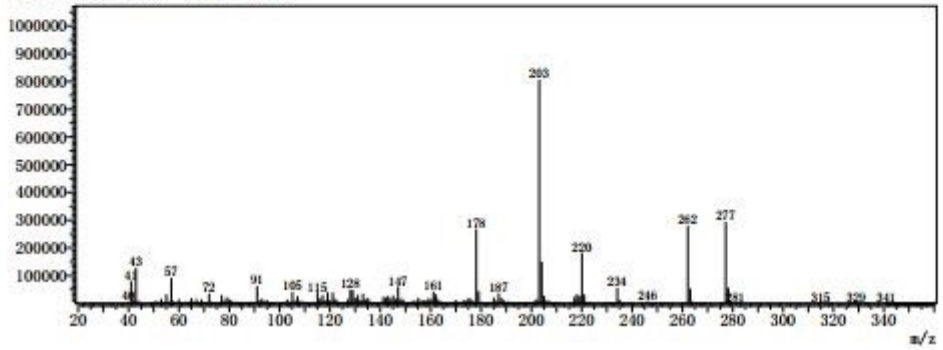
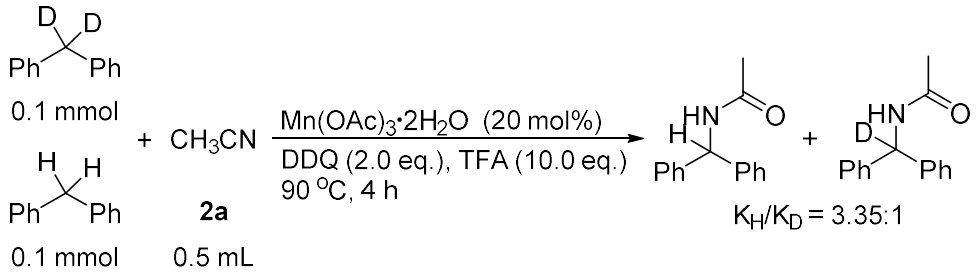
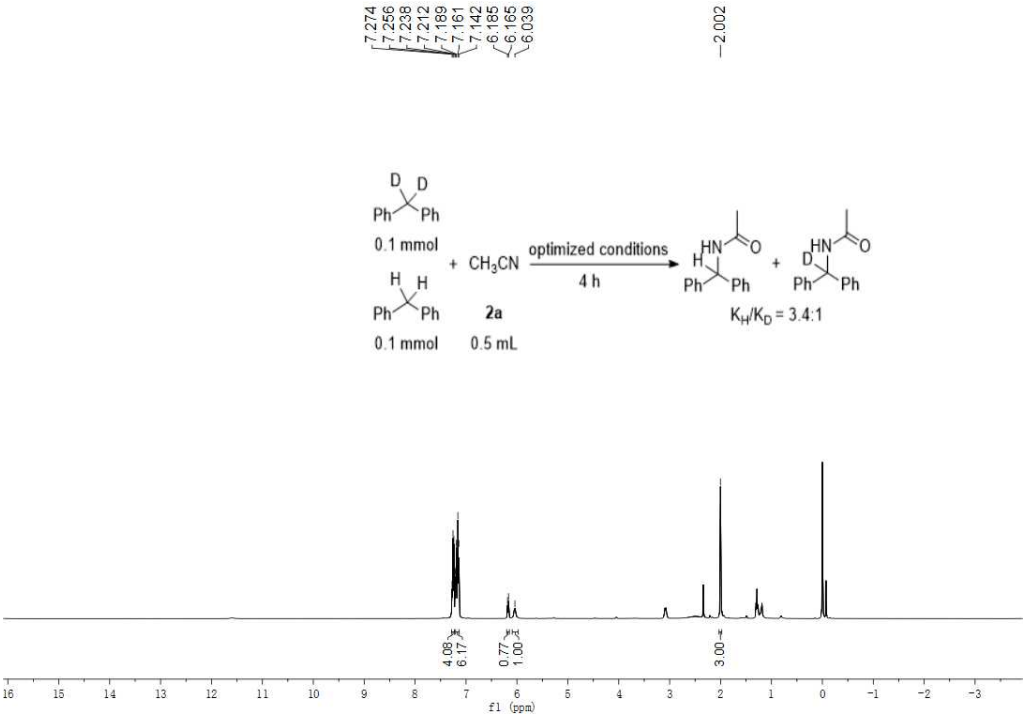


c) **Kinetic Isotopic Effect Experiments**

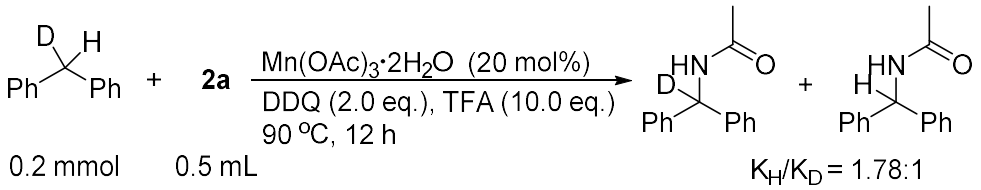
Procedure for intermolecular KIE:



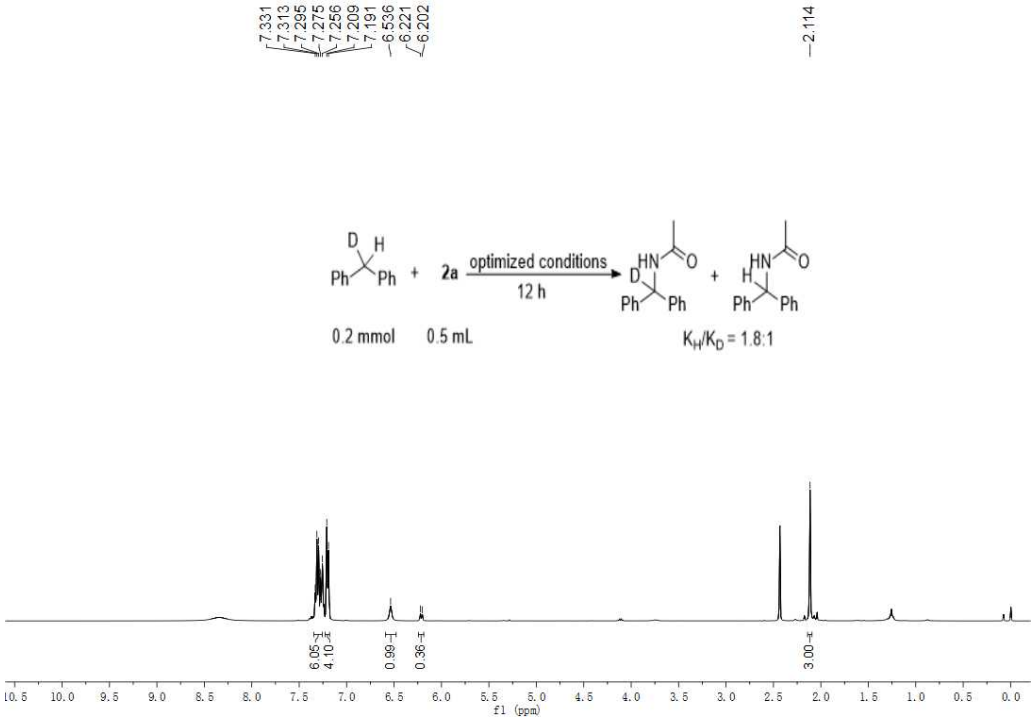
An oven-dried 25 mL Schlenk tube, which was equipped with a magnetic stir bar and charged with $Mn(OAc)_3 \cdot 2H_2O$ (0.04 mmol), DDQ (0.4 mmol). After charging nitrogen for three times, di-deuterated diphenylmethane (0.1 mmol), diphenylmethane (0.1 mmol), acetonitrile (0.5 mL), and TFA (10.0 equiv.) were added. Then the reaction mixture was heated to 90 °C for 4 h. Then the reaction mixture was cooled down to room temperature, and neutralized by K_2CO_3. The crude product was extracted with diethyl ether, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate to afford the desired product in a 27% yield.



Procedure for intramolecular KIE:

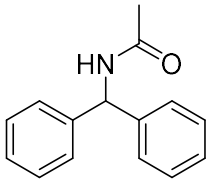


An oven-dried 25 mL Schlenk tube, which was equipped with a magnetic stir bar and charged with $Mn(OAc)_3 \cdot 2H_2O$ (0.04 mmol), DDQ (0.4 mmol), after charging nitrogen for three times, mono-deuterated diphenylmethane (0.2 mmol), acetonitrile (0.5 mL), and TFA (10.0 equiv.) were added. Then the reaction mixture was heated to 90 °C for 12 h. Then the reaction mixture was cooled down to room temperature, and neutralized by K_2CO_3. The crude product was extracted with diethyl ether, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate to afford the desired product in a 75% yield.



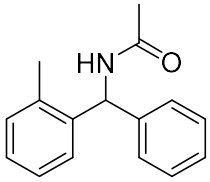
3. Characterization Data for the Products

N-benzhydrylacetamide (*3a*)[1]



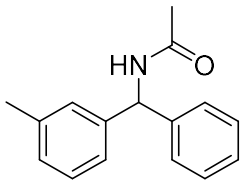
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield: 90%; mp 147–149 °C ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.26 (m, 6H), 7.22 (d, *J* = 7.2 Hz, 4H), 6.24 (d, *J* = 8.0 Hz, 1H), 6.09 (d, *J* = 6.0 Hz, 1H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.1, 141.4, 128.6, 127.4, 127.3, 57.0, 23.3.

N-(phenyl(o-tolyl)methyl)acetamide (*3b*)[2]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 81%; mp 130–132 °C ; ^{1}H NMR (400 M, $CDCl_3$): δ 7.31–7.27 (m, 2H), 7.24–7.13 (m, 6H), 7.10–7.08 (m, 1H), 6.39 (d, *J* = 8.0 Hz, 1H), 6.13 (s, 1H), 2.27 (s, 3H), 2.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.9, 141.0, 139.4, 136.2, 130.7, 128.5, 127.4, 127.4, 127.3, 126.6, 126.1, 53.9, 23.1, 19.4.

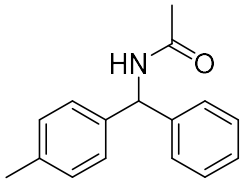
N-(phenyl(m-tolyl)methyl)acetamide (*3c*)[2]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 83%; mp 99–101 ^{o}C ; ^{1}H NMR (400 M, $CDCl_3$): δ 7.40–7.27 (m, 6H), 7.15–7.06 (m, 3H), 7.25–6.27 (m, 2H), 2.38 (s, 3H), 2.10 (s,

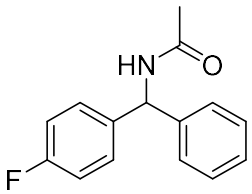
3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.2, 141.5, 141.3, 138.3, 128.5, 128.5, 128.2, 128.1, 127.3, 127.3, 124.3, 57.0, 23.2, 21.4.

N-(phenyl(p-tolyl)methyl)acetamide (*3d*)[2]



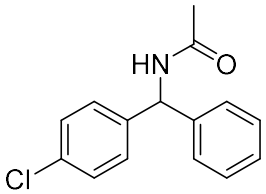
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield: 88%; mp 126–128 °C. ^{1}H NMR (400 M, $CDCl_3$): δ 7.33–7.27 (m, 3H), 7.22–7.20 (m, 2H), 7.14–7.09 (m, 4H), 6.20 (d, J = 8.0 Hz, 1H), 6.12 (d, J = 6.8 Hz, 1H), 2.32 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.1, 141.5, 138.5, 137.1, 129.3, 128.5, 127.3, 127.3, 127.2, 56.7, 23.3, 21.0.

N-((4-fluorophenyl)(phenyl)methyl)acetamide (*3e*)[2]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 85%; mp 147–149 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.33–7.26 (m, 3H), 7.19–7.15 (m, 4H), 7.01–6.96 (m, 2H), 6.35 (d, J = 7.2 Hz, 1H), 6.19 (d, J = 7.6 Hz, 1H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.5, 162.0 (d, J_{C-F} = 244.7 Hz), 141.1, 137.1 (d, J_{C-F} = 3.2 Hz), 128.9 (d, J_{C-F} = 8.1 Hz), 128.7, 127.6, 127.3, 115.4 (d, J_{C-F} = 21.4 Hz), 56.4, 23.1.

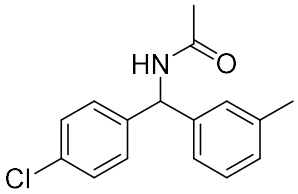
N-((4-chlorophenyl)(phenyl)methyl)acetamide (*3f*)[2]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 93%; mp

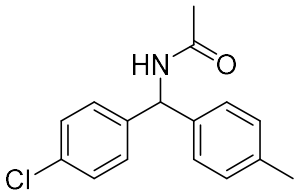
133–135 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.34–7.26 (m, 5H), 7.18–7.13 (m, 4H), 6.25 (d, *J* = 6.8 Hz, 1H), 6.18 (d, *J* = 7.6 Hz, 1H), 2.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.3, 140.8, 139.8, 133.2, 128.8, 128.7, 128.6, 127.7, 127.4, 56.5, 23.2.

N-((4-chlorophenyl)(m-tolyl)methyl)acetamide (*3g*)



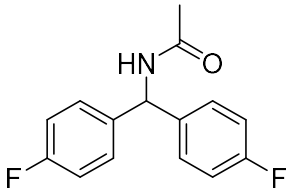
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 76%; mp 187–189 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.29 (s, 1H), 7.27 (s, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.15 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 7.6 Hz, 1H), 6.99–6.96 (m, 2H), 6.16 (d, *J* = 8.0 Hz, 1H), 6.02 (s, 1H), 2.31 (s, 3H), 2.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.2, 140.9, 140.1, 138.5, 133.0, 128.6 (2), 128.6, 128.4, 128.1, 124.4, 56.4, 23.2, 21.3. HRMS (EI) m/z: [M]+ calcd. for $C_{16}H_{16}ClNO$ 273.0920; found: 273.0910.

N-((4-chlorophenyl)(p-tolyl)methyl)acetamide (*3h*)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 85%; mp 162–164 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.27 (d, *J* = 8.4 Hz, 2H), 7.15–7.12 (m, 4H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.16–6.14 (m, 2H), 2.32 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.1, 140.1, 138.0, 137.5, 133.1, 129.4, 128.6, 128.5, 127.3, 56.2, 23.2, 21.0. HRMS (EI) m/z: [M]+ calcd. for $C_{16}H_{16}ClNO$ 273.0920; found: 273.0910.

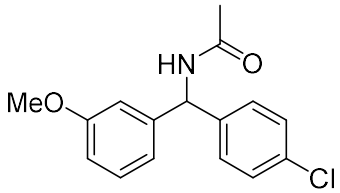
N-(bis(4-fluorophenyl)methyl)acetamide (*3i*)[1]



The title compound was prepared according to the general procedure and purified by column chromatography on

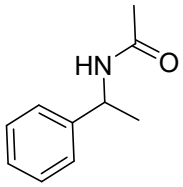
silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 86%; mp 160–162 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.17–7.14 (m, 4H), 7.03–6.99 (m, 4H), 6.19 (d, J = 8.0 Hz, 1H), 6.05 (d, J = 5.6 Hz, 1H), 2.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.1, 162.1 (d, J_{C-F} = 245.0 Hz), 137.0 (d, J_{C-F} = 3.1 Hz), 128.9 (d, J_{C-F} = 8.1 Hz), 115.6 (d, J_{C-F} = 21.4 Hz), 55.7, 23.2.

N-((4-chlorophenyl)(3-methoxyphenyl)methyl)acetamide (*3j*)



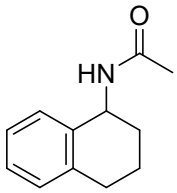
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 75%; mp 176–178 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.23–7.19 (m, 3H), 7.12 (d, J = 8.0 Hz, 2H), 6.79–6.70 (m, 3H), 6.29 (br, 1H), 6.11 (d, J = 7.6 Hz, 1H), 3.73 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.6, 159.8, 142.3, 139.7, 133.2, 129.8, 128.7, 128.6, 119.6, 113.5, 112.6, 56.5, 55.2, 23.1. HRMS (EI) m/z: [M]+ calcd. for $C_{16}H_{16}ClNO_2$ 289.0870; found: 289.0858.

N-(1-phenylethyl)acetamide (*3k*)[1]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 59%; mp 74–76 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.27–7.18 (m, 5H), 5.83 (s, 1H), 5.07–4.99 (m, 1H), 1.89 (s, 3H), 1.40 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.2, 143.0, 128.6, 127.3, 126.1, 48.8, 23.3, 21.6.

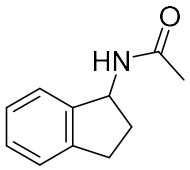
N-(1,2,3,4-tetrahydronaphthalen-1-yl)acetamide (*3l*)[3]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 55%; mp

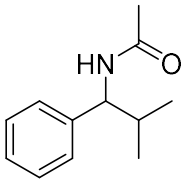
145–147 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.16–7.09 (m, 4H), 5.80 (br, 1H), 5.16–5.14 (m, 1H), 2.77–2.76 (m, 2H), 2.02–1.99 (m, 4H), 1.81 (br, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.2, 137.5, 136.6, 129.1, 128.6, 127.2, 126.1, 47.3, 30.0, 29.1, 23.4, 19.8.

N-(2,3-dihydro-1H-inden-1-yl)acetamide (*3m*)[4]



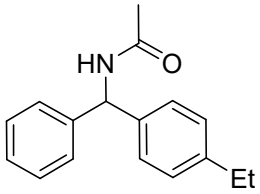
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a off white solid. Yield 75%; mp 206–208 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.18–7.08 (m, 4H), 5.99(br, 1H), 5.34–5.28 (m, 1H), 2.90–2.83 (m, 1H), 2.78–2.70 (m, 1H), 2.49–2.40 (m, 1H), 1.89 (s, 3H), 1.73–1.64 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.0, 143.2, 143.1, 127.9, 126.7, 124.7, 124.0, 54.7, 33.9, 30.2, 23.3.

N-(2-methyl-1-phenylpropyl)acetamide (*3n*)[1]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 20%; mp 115–117 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.33–7.29 (m, 2H), 7.24–7.21 (m, 3H), 5.74 (d, J = 7.6 Hz, 1H), 4.75 (t, J = 8.4 Hz, 1H), 2.05–2.01 (m, 1H), 1.99 (s, 3H), 0.96 (d, J = 6.8 Hz, 3H), 0.82 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.2, 141.5, 128.4, 127.1, 126.9, 59.1, 33.3, 23.5, 19.7, 18.8.

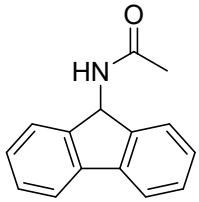
N-((4-ethylphenyl)(phenyl)methyl)acetamide (*3o*)



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a pale yellow solid. Yield 63%; mp 133–135 °C; ^{1}H NMR (400 M, $CDCl_3$): δ7.33–7.27 (m, 3H), 7.22 (d, J = 7.6 Hz, 2H), 7.16–7.11 (m, 4H), 6.21 (d,

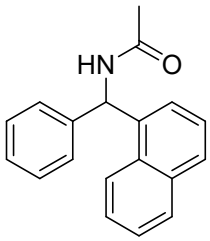
J = 8.0 Hz, 1H), 6.08 (d, *J* = 7.2 Hz, 1H), 2.62 (q, *J* = 15.2 Hz, 7.6 Hz, 2H), 2.05 (s, 3H), 1.22 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.0, 143.5, 141.6, 138.7, 128.5, 128.1, 127.4, 127.3, 127.2, 56.7, 28.4, 23.3, 15.4. HRMS (EI) m/z: [M]+ calcd. for $C_{17}H_{19}NO$ 253.1467; found: 253.1451.

N-(9H-fluoren-9-yl)acetamide (*3p*)[1]



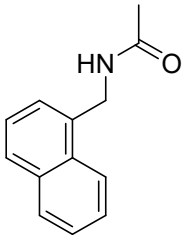
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 48%; mp 255–257 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.61 (d, *J* = 7.2 Hz, 2H), 7.49 (d, *J* = 7.6 Hz, 2H), 7.34–7.31 (m, 2H), 7.23–7.19 (m, 2H), 6.13 (d, *J* = 8.8 Hz, 1H), 5.79 (d, *J* = 8.0 Hz, 1H), 2.04 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 144.0, 140.5, 128.7, 127.7, 125.1, 119.9, 54.8, 23.3.

N-(naphthalen-1-yl(phenyl)methyl)acetamide (*3q*)[5]



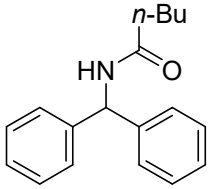
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 80%; mp 228–230 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 8.01–7.98 (m, 1H), 7.88–7.86 (m, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.49–7.47 (m, 2H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.34–7.27 (m, 5H), 7.22 (d, *J* = 7.2 Hz, 1H), 6.99 (d, *J* = 8.0 Hz, 1H), 6.15 (d, *J* = 7.6 Hz, 1H), 2.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.9, 141.1, 137.1, 134.0, 131.1, 128.8, 128.6, 128.5, 127.4, 127.3, 126.6, 125.8, 125.4, 125.1, 123.7, 53.8, 23.2.

N-(naphthalen-1-ylmethyl)acetamide (*3r*)[6]



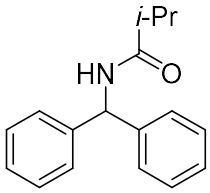
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 45%; mp 124–126 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 8.00 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 7.6 Hz, 1H), 7.82–7.80 (m, 1H), 7.56–7.49 (m, 2H), 7.42–7.39 (m, 2H), 5.78 (s, 1H), 4.86 (d, *J* = 5.2 Hz, 2H), 1.98 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.6, 133.8, 133.4, 131.3, 128.7, 128.6, 126.7, 126.6, 126.0, 125.3, 123.4, 41.8, 23.1.

N-benzhydrylpentanamide (3s)[7]



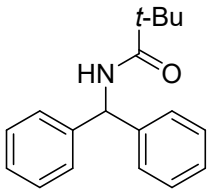
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 84%; mp 122–124 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.23–7.12 (m, 10H), 6.17 (d, *J* = 8.0 Hz, 1H), 6.08 (br, 1H), 2.19–2.15 (m, 2H), 1.60–1.52 (m, 2H), 1.31–1.24 (m, 2H), 0.83 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 141.5, 128.6, 127.3, 127.3, 56.8, 36.4, 27.7, 22.3, 13.7.

N-benzhydrylisobutyramide (*3t*)[8]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 75%; mp 143–145 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.35–7.31 (m, 4H), 7.29–7.27 (m, 2H), 7.23–7.21 (m, 4H), 6.25 (d, *J* = 8.0 Hz, 1H), 6.10 (d, *J* = 6.4 Hz, 1H), 2.49–2.39 (m, 1H), 1.20 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 175.8, 141.6, 128.6, 127.3, 127.3, 56.6, 35.6, 19.5.

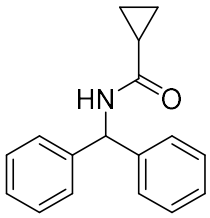
N-benzhydrylpivalamide (*3u*)[9]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 51%; mp

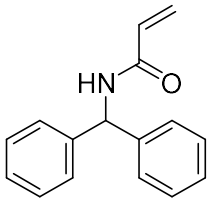
152–154 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.34–7.26 (m, 6H), 7.24–7.19 (m, 4H), 6.21 (d, *J* = 7.2 Hz, 1H), 6.18 (br, 1H), 1.24 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 177.3, 141.7, 128.6, 127.3, 127.2, 56.7, 38.7, 27.5.

N-benzhydrylcyclopropanecarboxamide (*3v*)[7]



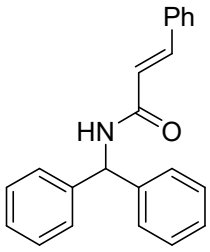
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 90%; mp 173–175 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.35–7.33 (m, 4H), 7.30–7.25 (m, 6H), 6.27 (br, 2H), 1.44 (br, 1H), 1.02 (br, 2H), 0.78–0.77 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.5, 141.6, 128.6, 127.4 (2), 57.0, 14.8, 7.3.

N-benzhydrylacrylamide (*3w*)[10]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 96%; mp 179–181 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.33–7.27 (m, 5H), 7.23–7.21 (m, 5H), 6.35–6.30 (m, 2H), 6.18–6.09 (m, 2H), 5.67 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.2, 140.9, 130.1, 128.3, 127.2, 127.1, 127.0, 56.7.

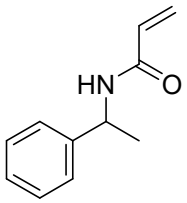
N-benzhydrylcinnamamide (*3x*)[8]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 93%; mp

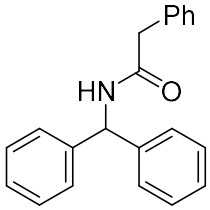
178–180 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.67 (d, *J* = 15.6 Hz, 1H), 7.48 (br, 2H), 7.40–7.25 (m, 13H), 6.46 (d, *J* = 15.6 Hz, 1H), 6.39 (d, *J* = 7.6 Hz, 1H), 6.17 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.9, 141.8, 141.3, 134.6, 129.7, 128.8, 128.7, 127.8, 127.5, 127.4, 120.1, 57.2.

N-(1-phenylethyl)acrylamide (*3y*)[10]



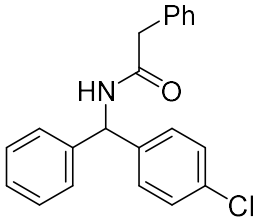
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 40%; mp 63–65 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.32–7.25 (m, 5H), 6.28 (d, *J* = 17.2 Hz, 1H), 6.11–6.04 (m, 1H), 5.88 (br, 1H), 5.63 (d, *J* = 10.0 Hz, 1H), 5.24–5.17 (m, 1H), 1.52 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.7, 142.7, 130.6, 128.6, 127.4, 126.8, 126.1, 48.8, 21.5.

N-benzhydryl-2-phenylacetamide (*3z*)[11]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 70%; mp 138–140 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.37–7.27 (m, 9H), 7.24–7.23 (m, 2H), 7.08 (d, *J* = 7.2 Hz, 4H), 6.23 (d, *J* = 8.4 Hz, 1H), 6.05 (d, *J* = 4.0 Hz, 1H), 3.64 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.1, 141.2, 134.6, 129.3, 129.0, 128.6, 127.4, 127.4, 127.1, 56.8, 43.8.

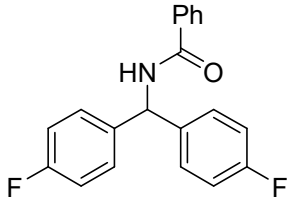
N-((4-chlorophenyl)(phenyl)methyl)-2-phenylacetamide (*3za*)



The title compound was prepared according to the general procedure and purified by column chromatography on

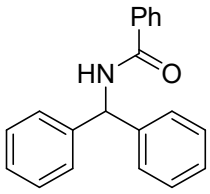
silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 70%; mp 133–135 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.37–7.27 (m, 7H), 7.22 (s, 3H), 7.05–7.00 (m, 4H), 6.19 (d, J = 8.4 Hz, 1H), 5.96 (d, J = 7.6 Hz, 1H), 3.63 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.0, 140.7, 139.8, 134.6, 133.2, 129.3, 129.1, 128.7, 128.7, 128.5, 127.6, 127.5, 127.2, 56.2, 43.8. HRMS (EI) m/z: [M]+ calcd. for $C_{21}H_{18}ClNO$ 335.1077; found: 335.1072.

N-(bis(4-fluorophenyl)methyl)benzamide (*3zb*)[12]



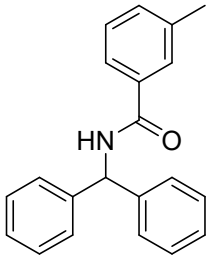
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 75%; mp 177–179 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.79 (d, J = 7.6 Hz, 2H), 7.53–7.49 (m, 1H), 7.45–7.41 (m, 2H), 7.24–7.22 (m, 4H), 7.03 (t, J = 8.4 Hz, 4H), 6.57 (d, J = 7.2 Hz, 1H), 6.40 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.4, 162.2 (d, J_{C-F} = 245.3 Hz), 136.9 (d, J_{C-F} = 3.3 Hz), 133.8, 131.9, 129.0 (d, J_{C-F} = 8.1 Hz), 128.7, 127.0, 115.7 (d, J_{C-F} = 21.5 Hz), 56.2.

N-benzhydrylbenzamide (*3zc*)[11]



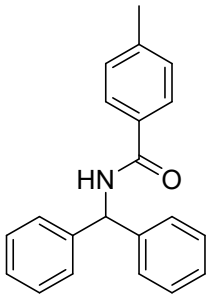
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 57%; mp 168–170 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.81 (d, J = 7.6 Hz, 2H), 7.52–7.49 (m, 1H), 7.45–7.41 (m, 2H), 7.36–7.28 (m, 10H), 6.70 (d, J = 7.2 Hz, 1H), 6.46 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.4, 141.4, 134.2, 131.6, 128.7, 128.6, 127.5, 127.4, 127.0, 57.4.

N-benzhydryl-3-methylbenzamide (*3zd*)[8]



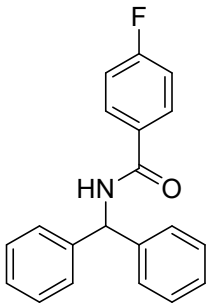
The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 45%; mp 151–153 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.55 (s, 1H), 7.53–7.51 (m, 1H), 7.29–7.27 (m, 3H), 7.24–7.18 (m, 9H), 6.58 (d, *J* = 7.2 Hz, 1H), 6.37 (d, *J* = 7.6 Hz, 1H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.2, 141.4, 138.5, 134.2, 132.4, 128.7, 128.4, 127.7, 127.5, 127.5, 123.9, 57.3, 21.3.

N-benzhydryl-4-methylbenzamide (*3ze*)[8]



The title compound was prepared according to the general procedure and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 40%; mp 174–176 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.65 (d, *J* = 7.6 Hz, 2H), 7.29–7.27 (m, 3H), 7.23–7.15 (m, 9H), 6.62 (d, *J* = 7.2 Hz, 1H), 6.38 (d, *J* = 7.6 Hz, 1H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.4, 142.1, 141.5, 131.3, 129.2, 128.6, 127.4 (2), 127.0, 57.3, 21.4 .

N-benzhydryl-4-fluorobenzamide (*3zf*)[13]



The title compound was prepared according to the general procedure and purified by column chromatography on

silica gel and eluted with petroleum ether/ethyl acetate (10/1–1/1) to afford a white solid. Yield 66%; mp 217–219 °C; ^{1}H NMR (400 M, $CDCl_3$): δ 7.75 (dd, J = 7.6, 5.6 Hz, 2H), 7.29–7.27 (m, 3H), 7.22–7.18 (m, 7H), 7.04–7.00 (m, 2H), 6.59 (d, J = 4.0 Hz, 1H), 6.35 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.0, 164.5 (d, J_{C-F} = 189.3 Hz), 141.2, 130.3 (d, J_{C-F} = 3.2 Hz), 129.3 (d, J_{C-F} = 8.9 Hz), 128.7, 127.6, 127.4, 115.6 (d, J_{C-F} = 21.7 Hz), 57.5.

4. References

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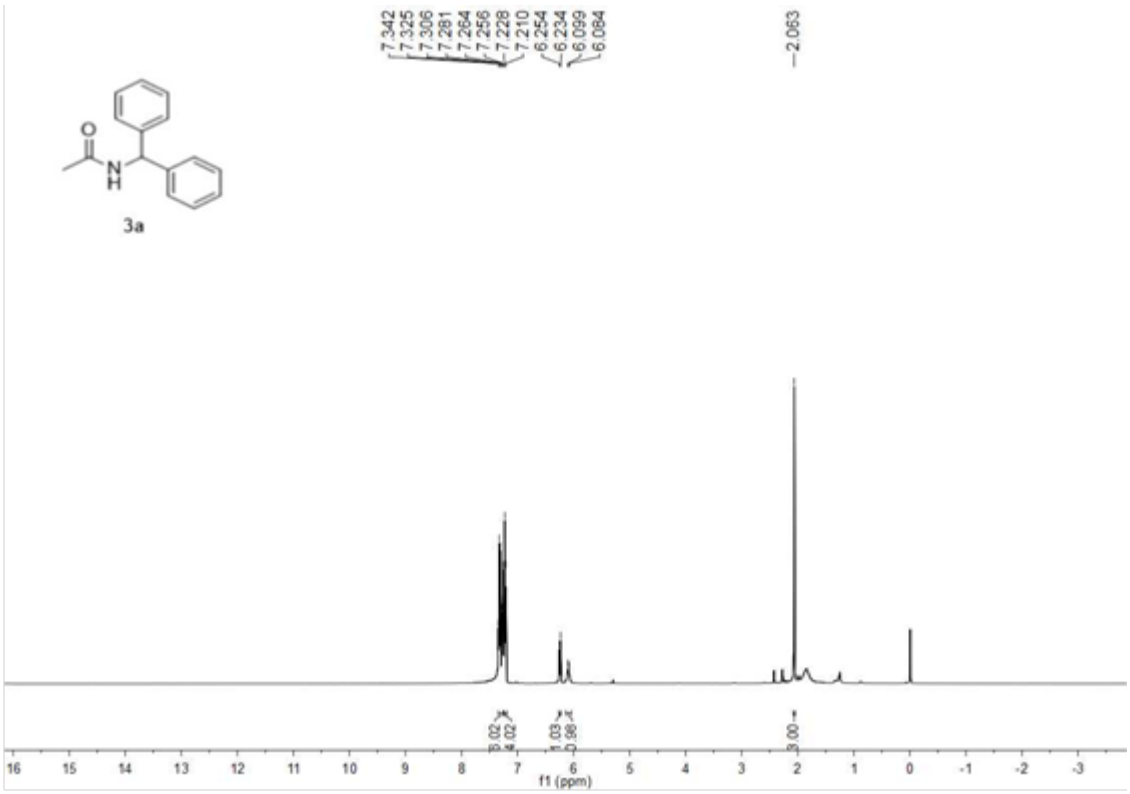
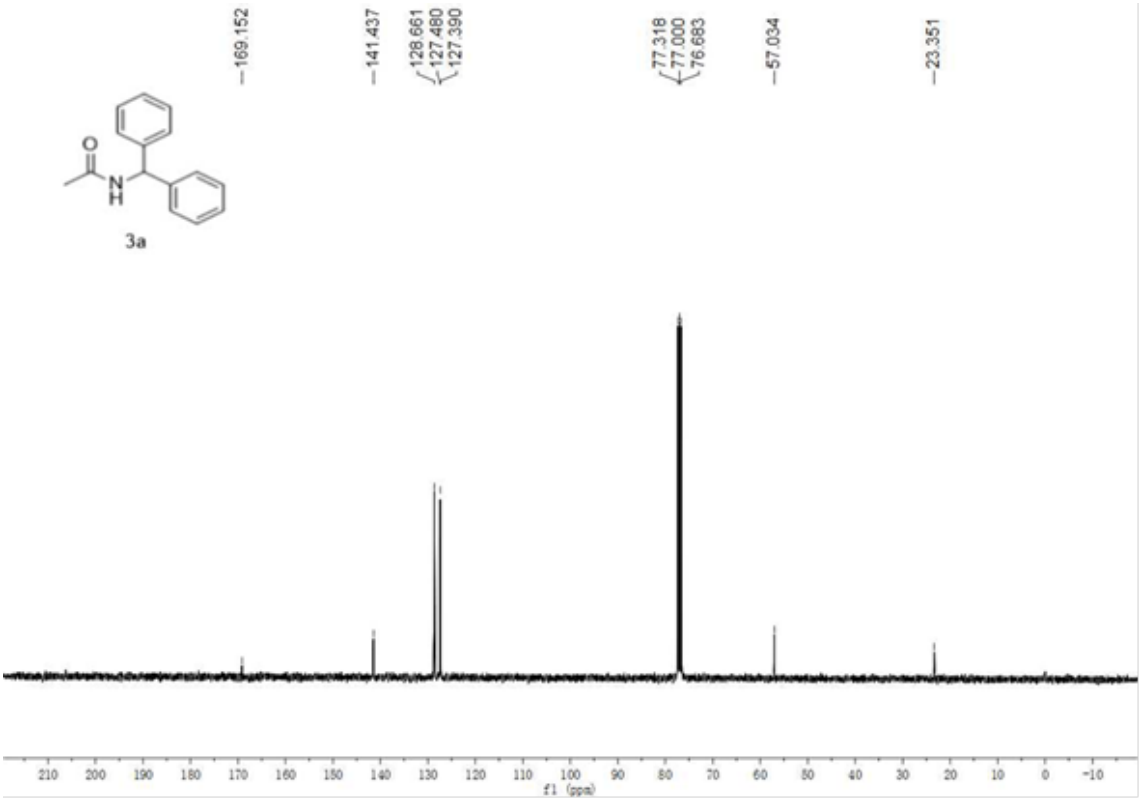
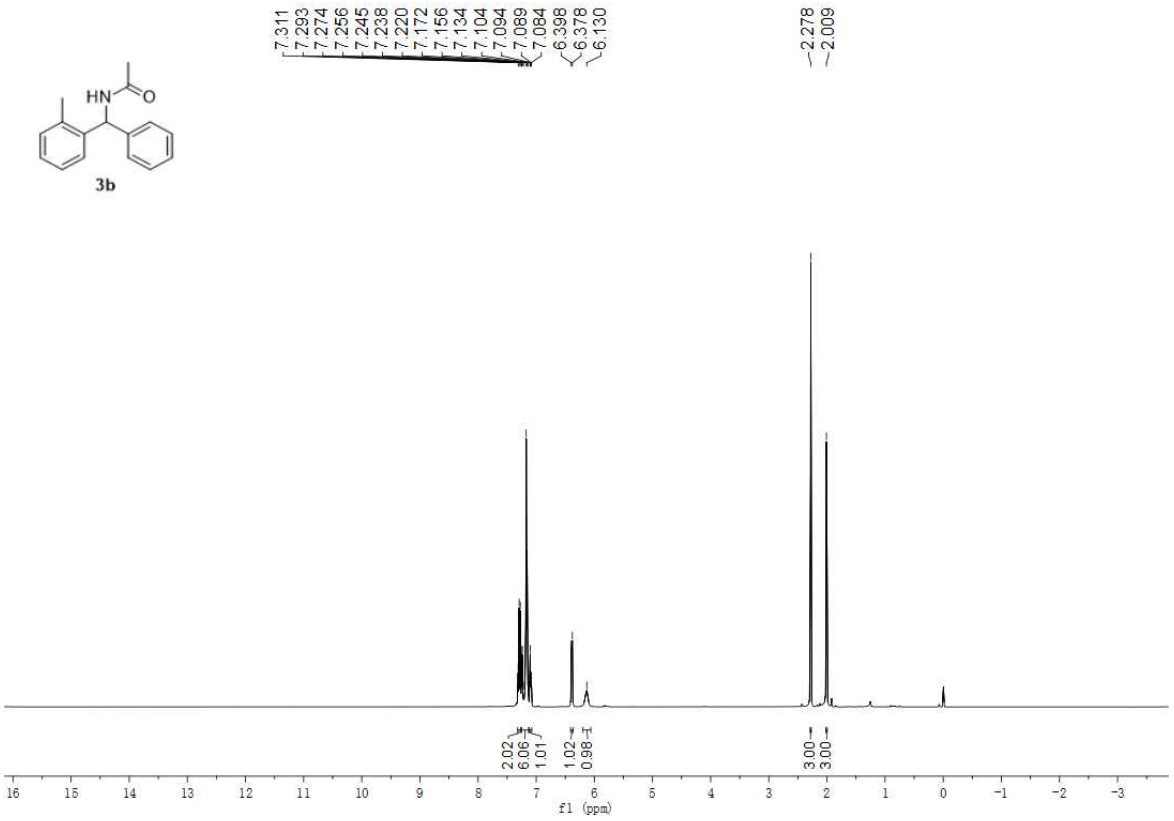
(13) X.-N. Zhao, H.-C. Hu, F.-J. Zhang, Z.-H. Zhang, *Appl. Catal. A. Gen.,* **2014**, *482*, 258.

5. Copies of ^{1}H and ^{13}C NMR Spectra of the Products



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7.094
7.089
7.084
6.398
6.378
6.130
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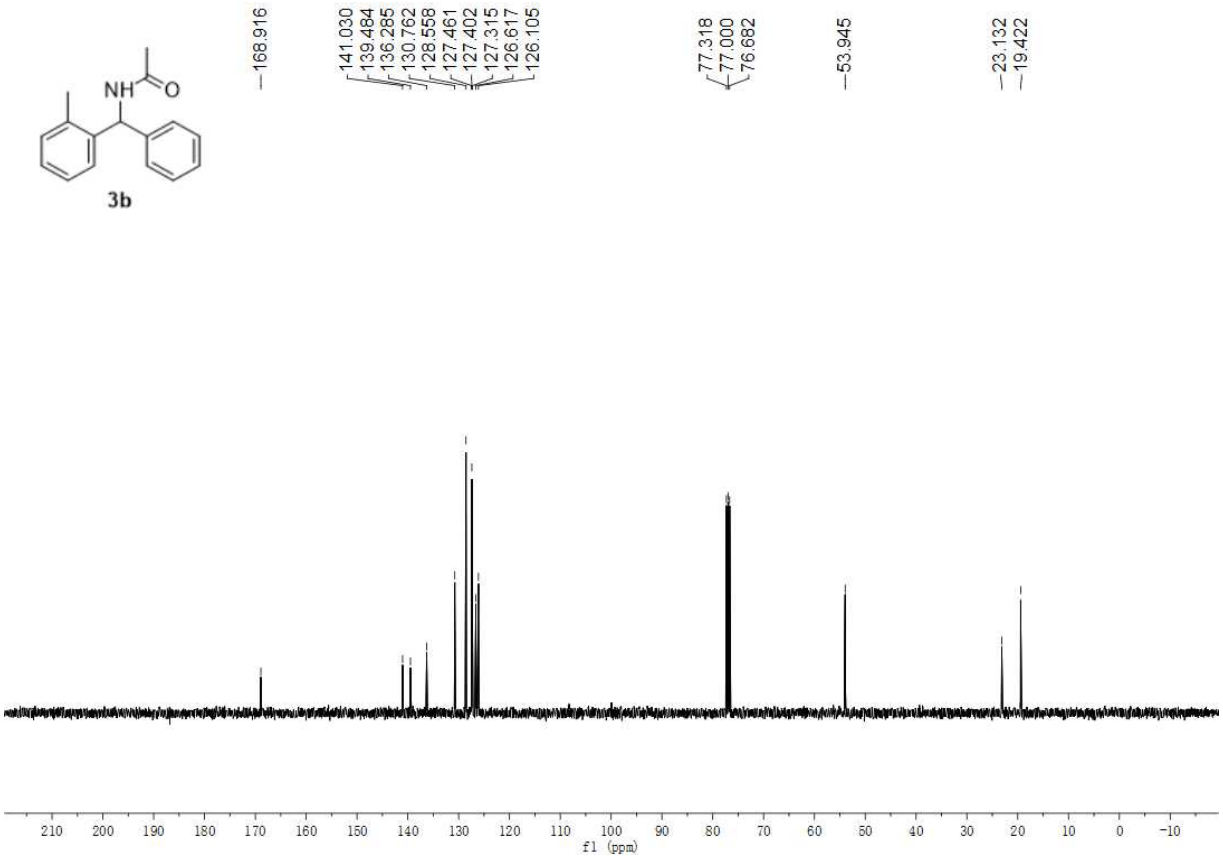
2.009
HN
O
3b
2.02
6.06
1.01
1.02
0.98
3.00
3.00
f1 (ppm)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77.000
76.682
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23.132
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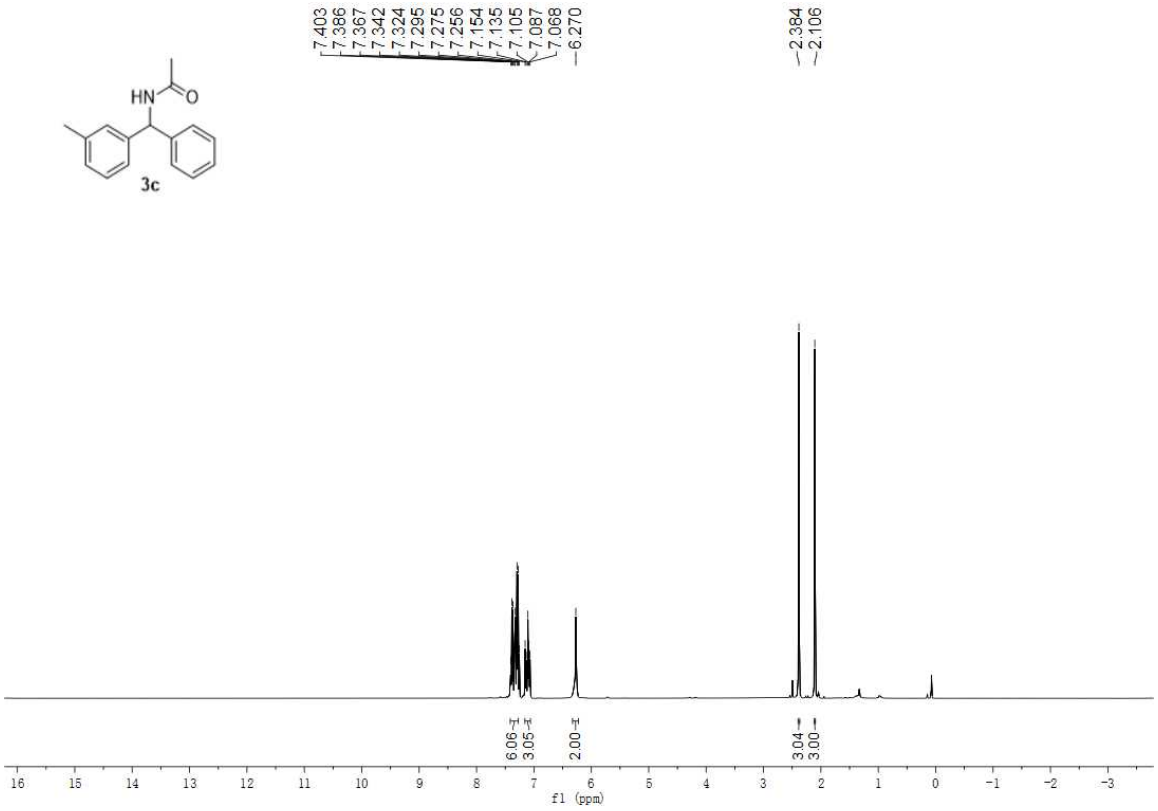
NH
O
3b
f1 (ppm)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2.106
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2.00
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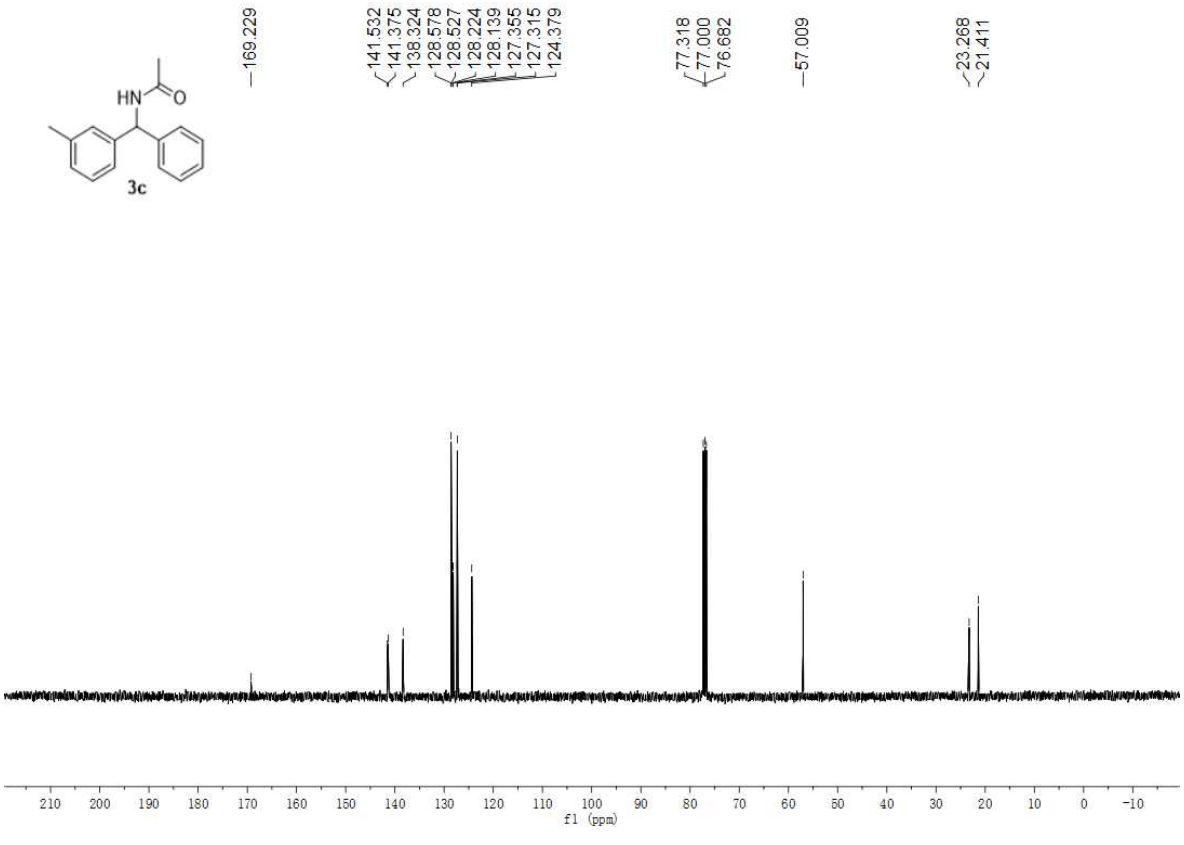
3.00
f1 (ppm)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76.682
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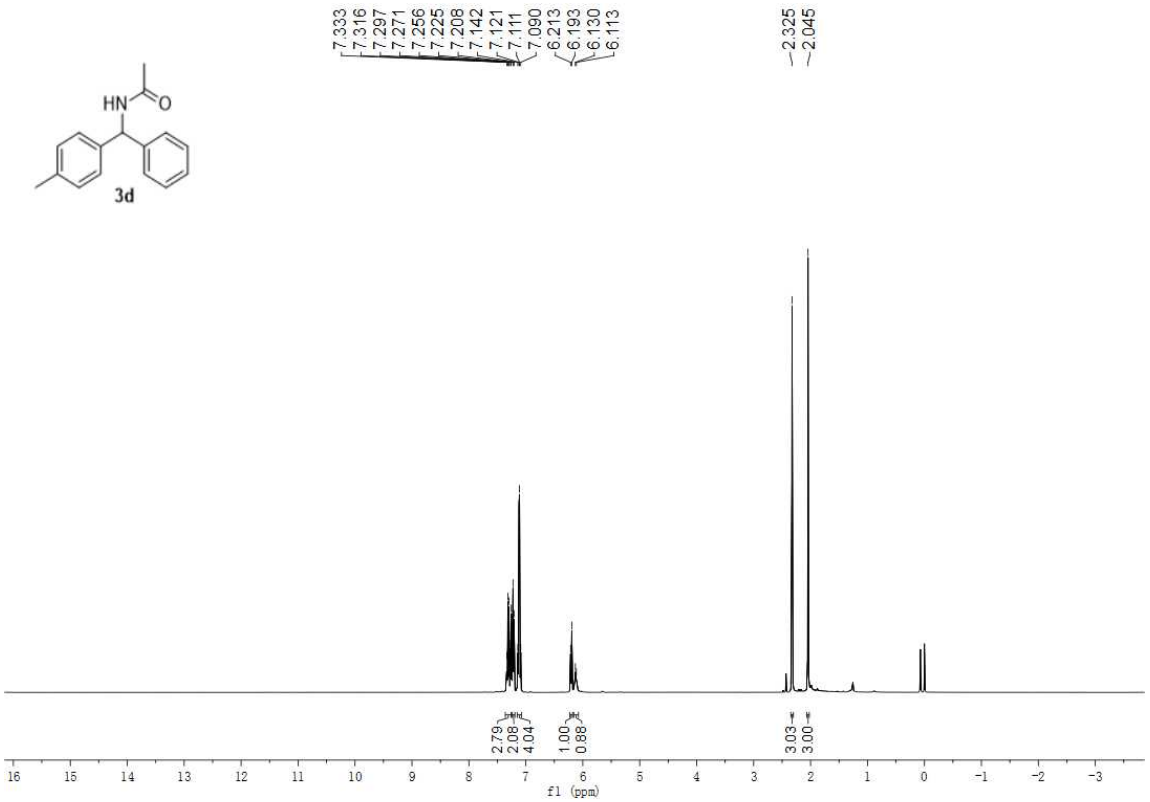
HN
O
3c
f1 (ppm)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6.130
6.113
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O
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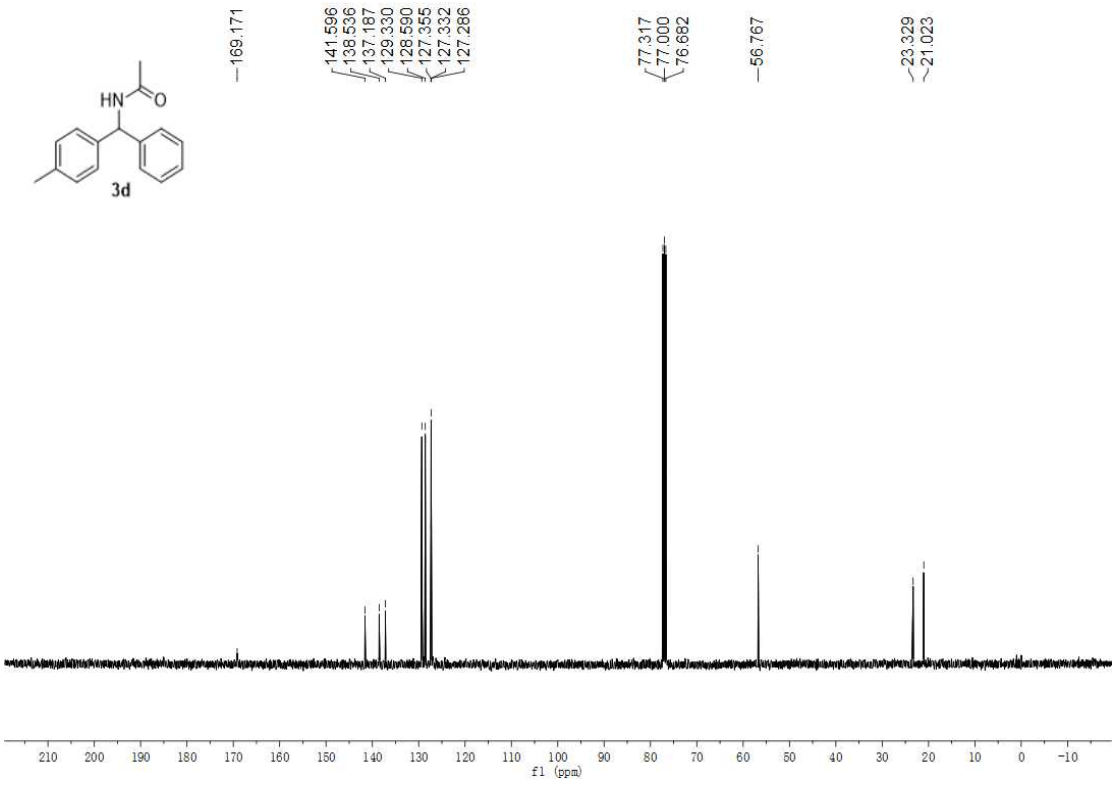
f1 (ppm)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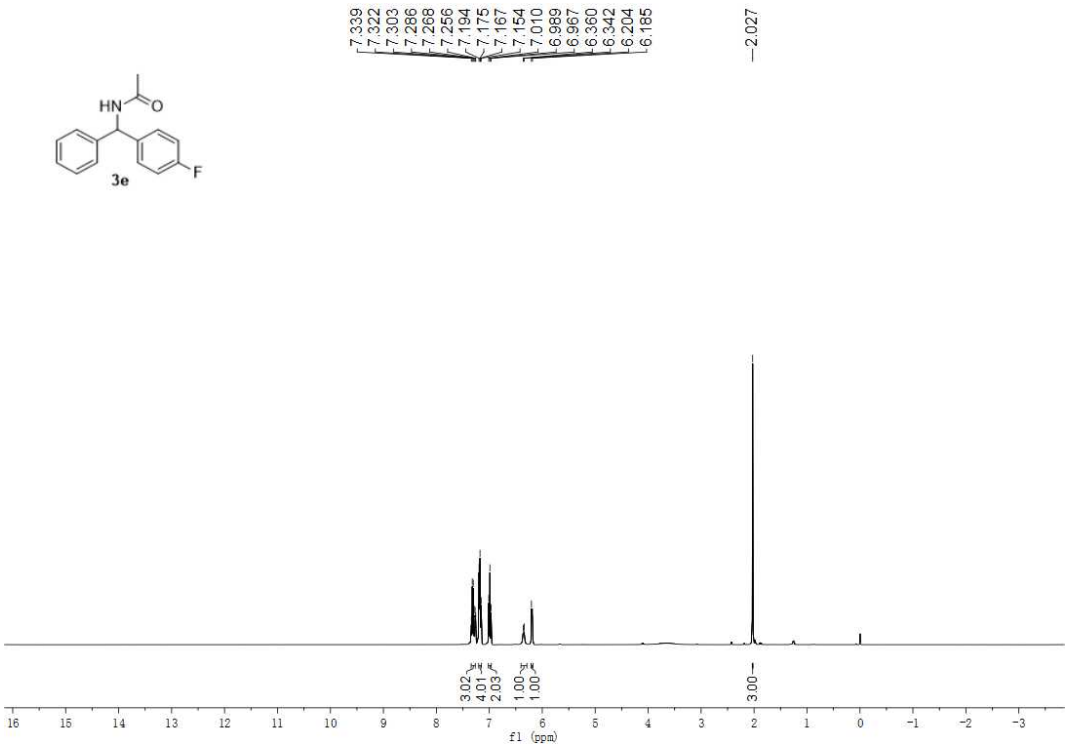
O
3d
f1 (ppm)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6.967
6.360
6.342
6.204
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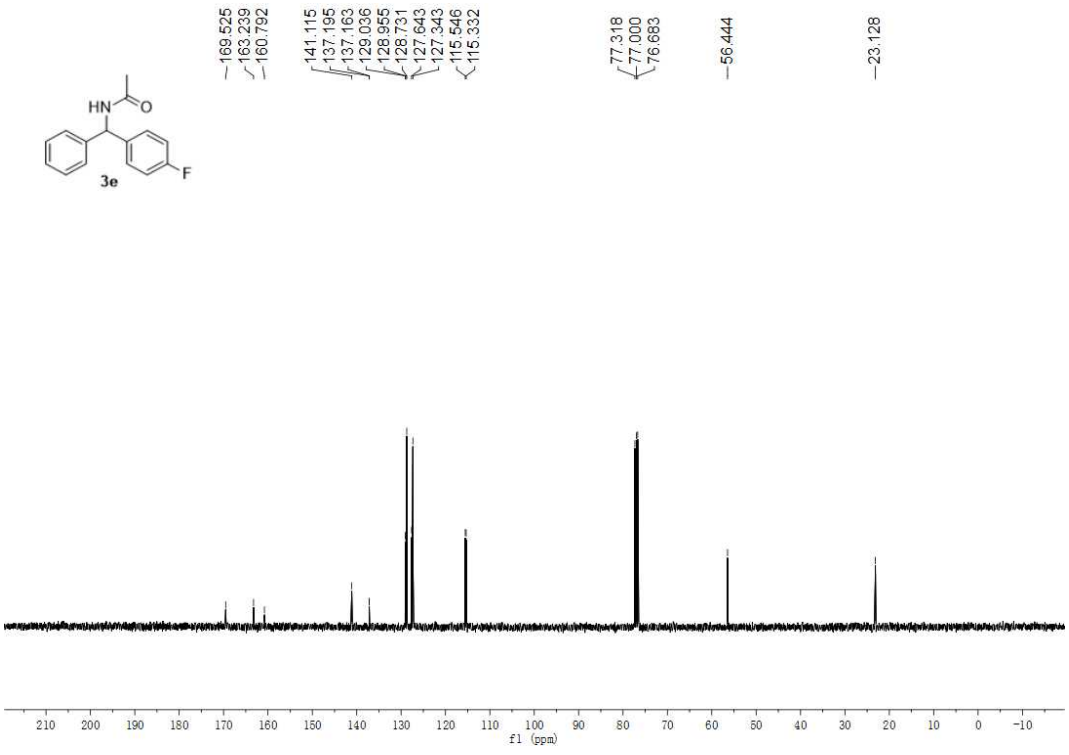
HN
O
F
3e
3.02
4.01
2.03
1.00
1.00
3.00
f1 (ppm)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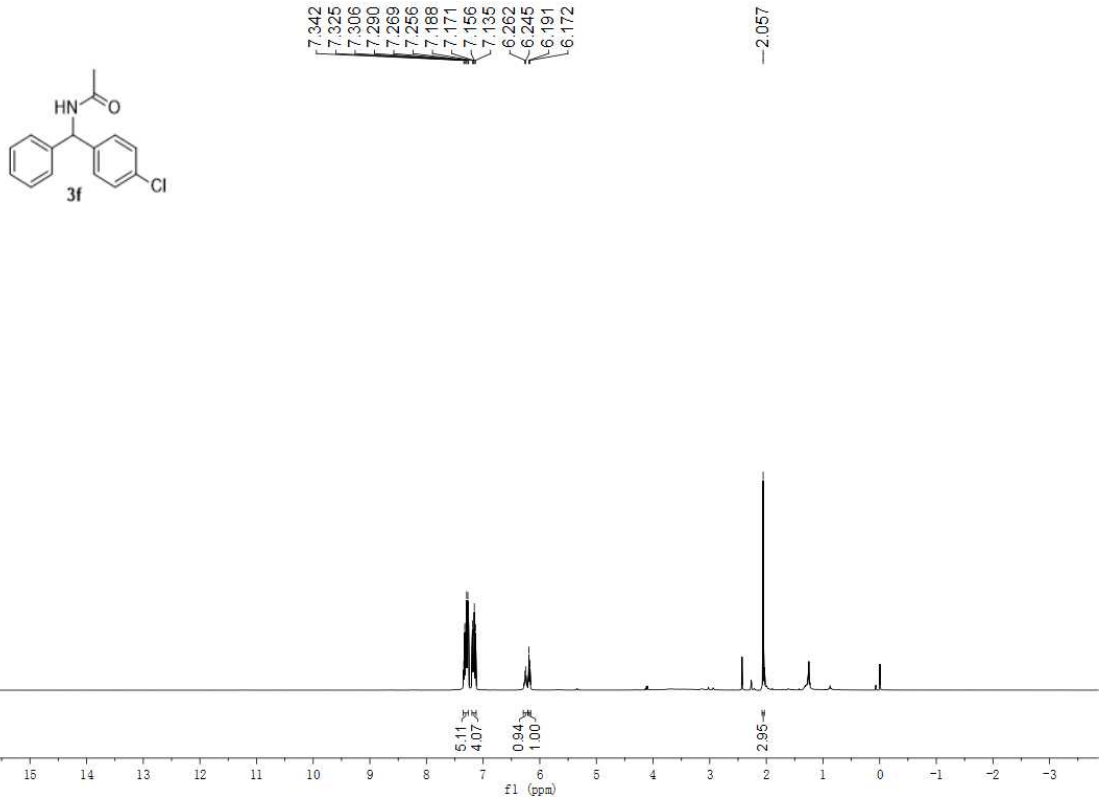
HN
O
F
3e
f1 (ppm)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O
Cl
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0.94
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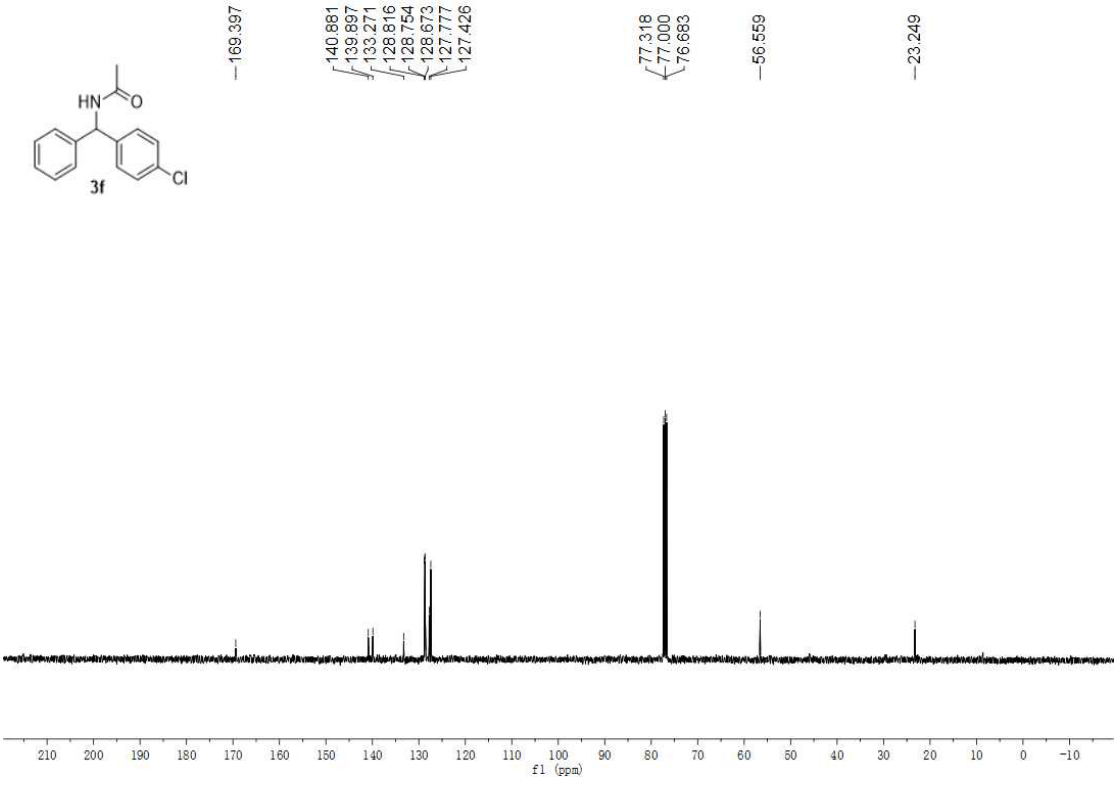
2.95
f1 (ppm)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128.754
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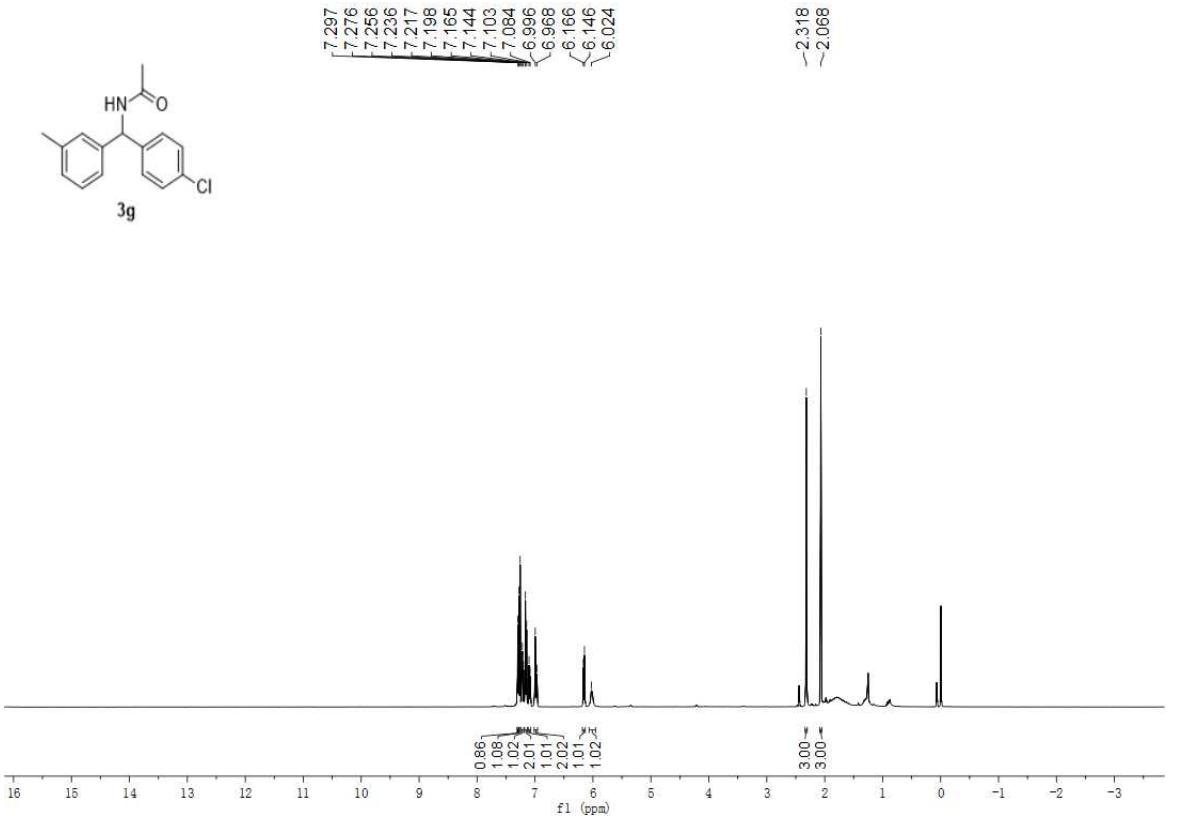
23.249
HN
O
Cl
3f
f1 (ppm)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7.144
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7.084
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6.968
6.166
6.146
6.024
2.318
2.068
HN
O
Cl
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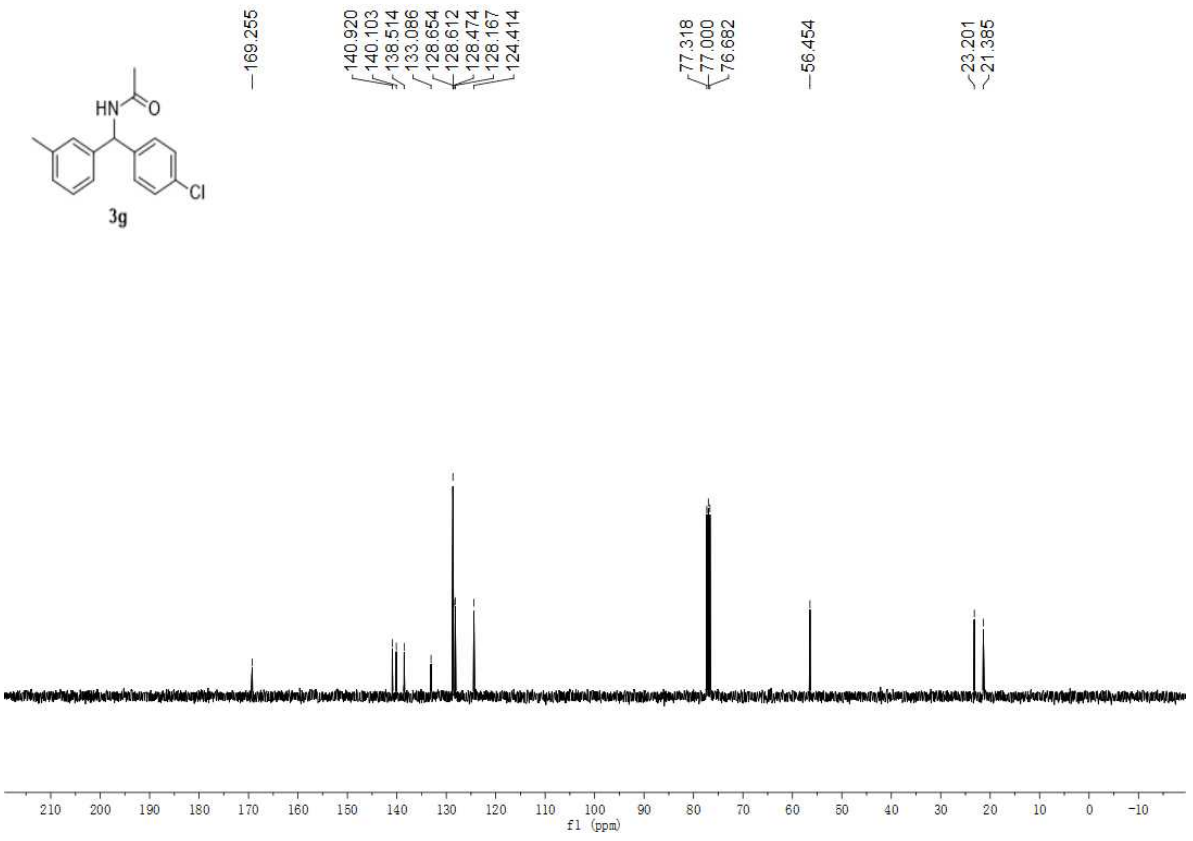
1.01
2.02
1.01
1.02
3.00
3.00
f1 (ppm)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Cl
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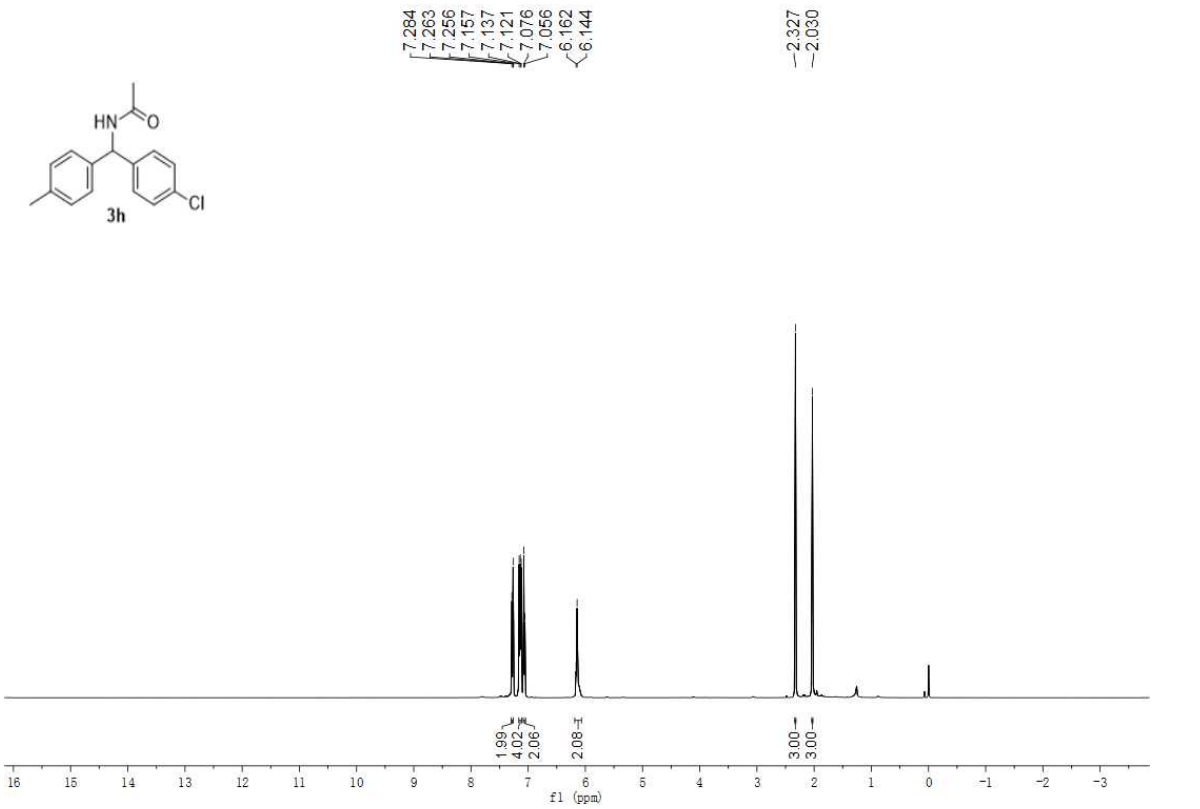
f1 (ppm)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Cl
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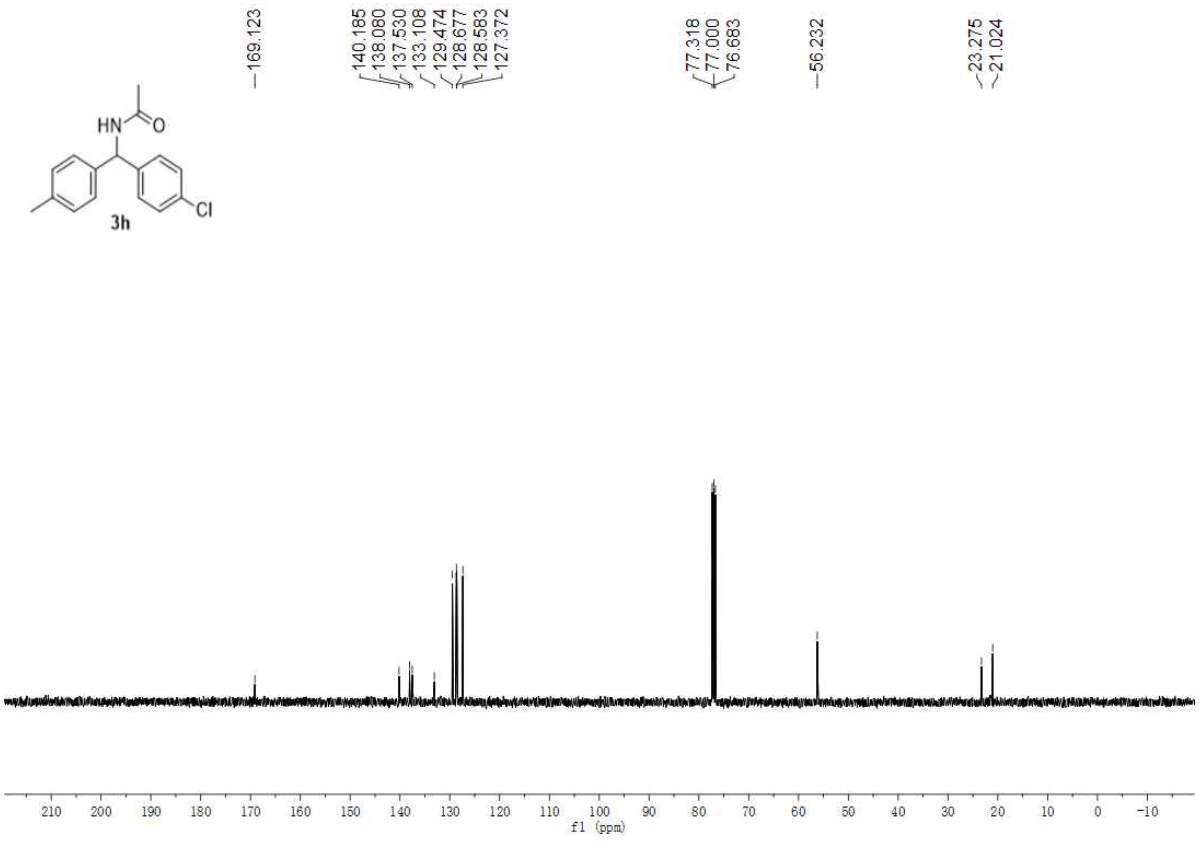
3.00
f1 (ppm)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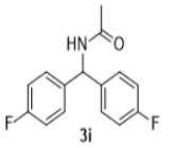
Cl
3h
f1 (ppm)


HN
O
F
F

3i

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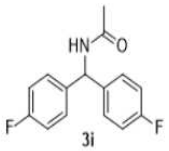
f1 (ppm)


HN
O
F
F

3i

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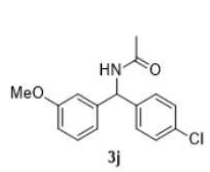
HN
O
MeO
Cl

3j


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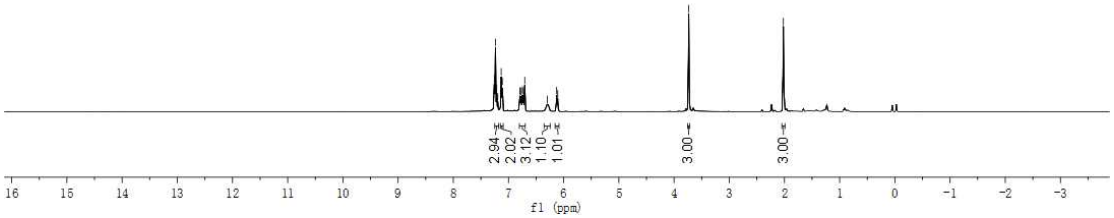
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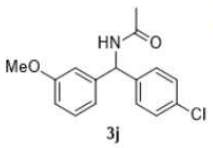
HN
O
MeO
Cl

3j


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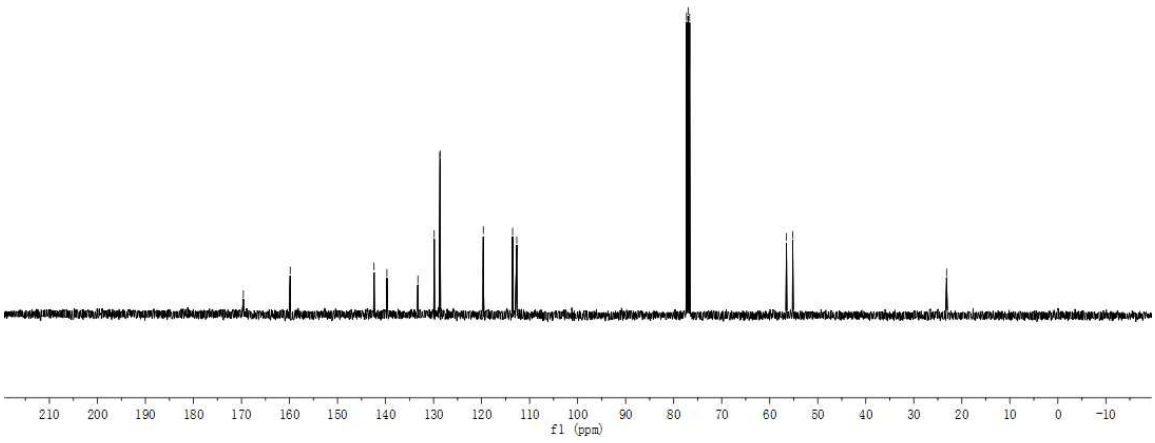
23.168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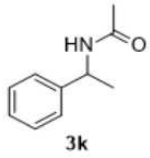

3k

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7.195
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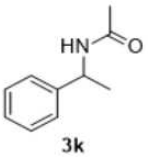
3.00
f1 (ppm)



3k

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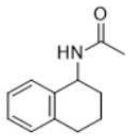

3l

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5.163
5.147
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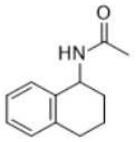
f1 (ppm)



3l

169.205
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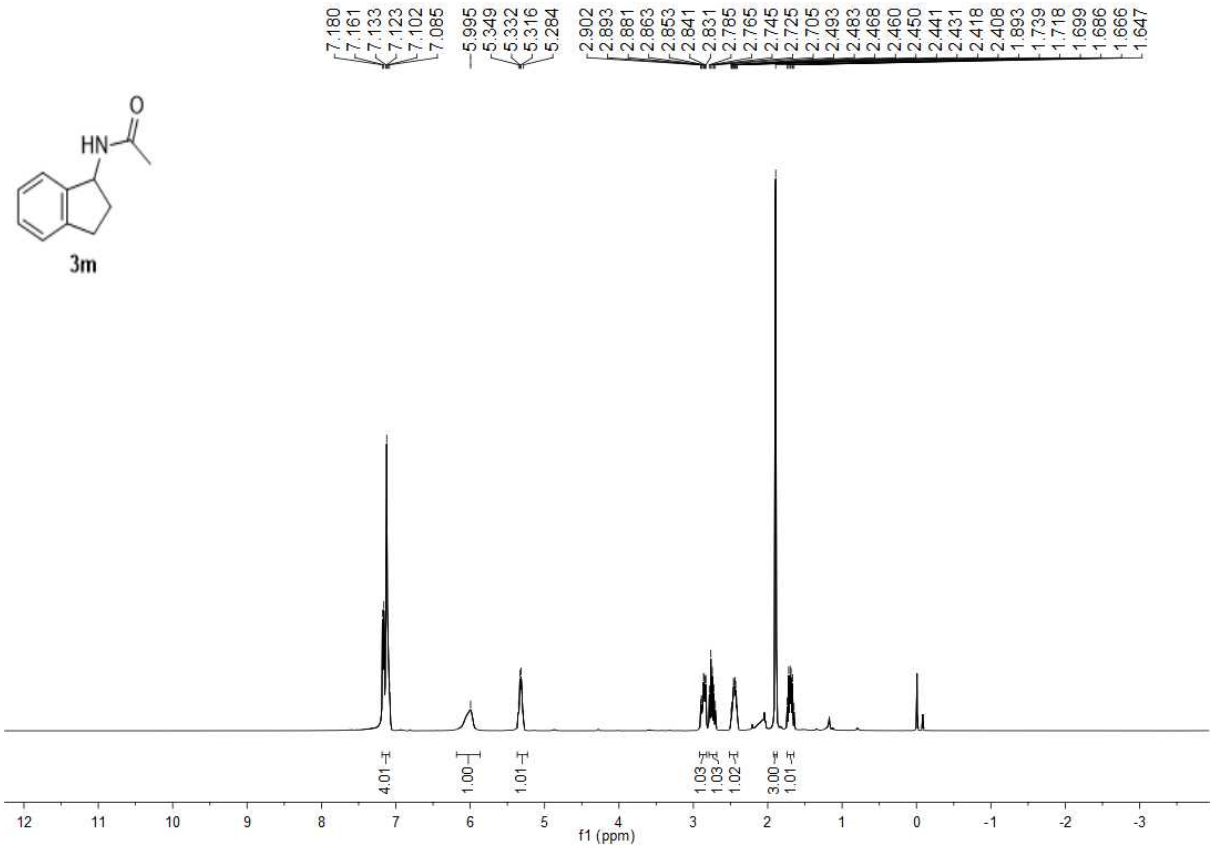
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2.765
2.745
2.725
2.705
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2.483
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2.441
2.431
2.418
2.408
1.893
1.739
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1.686
1.666
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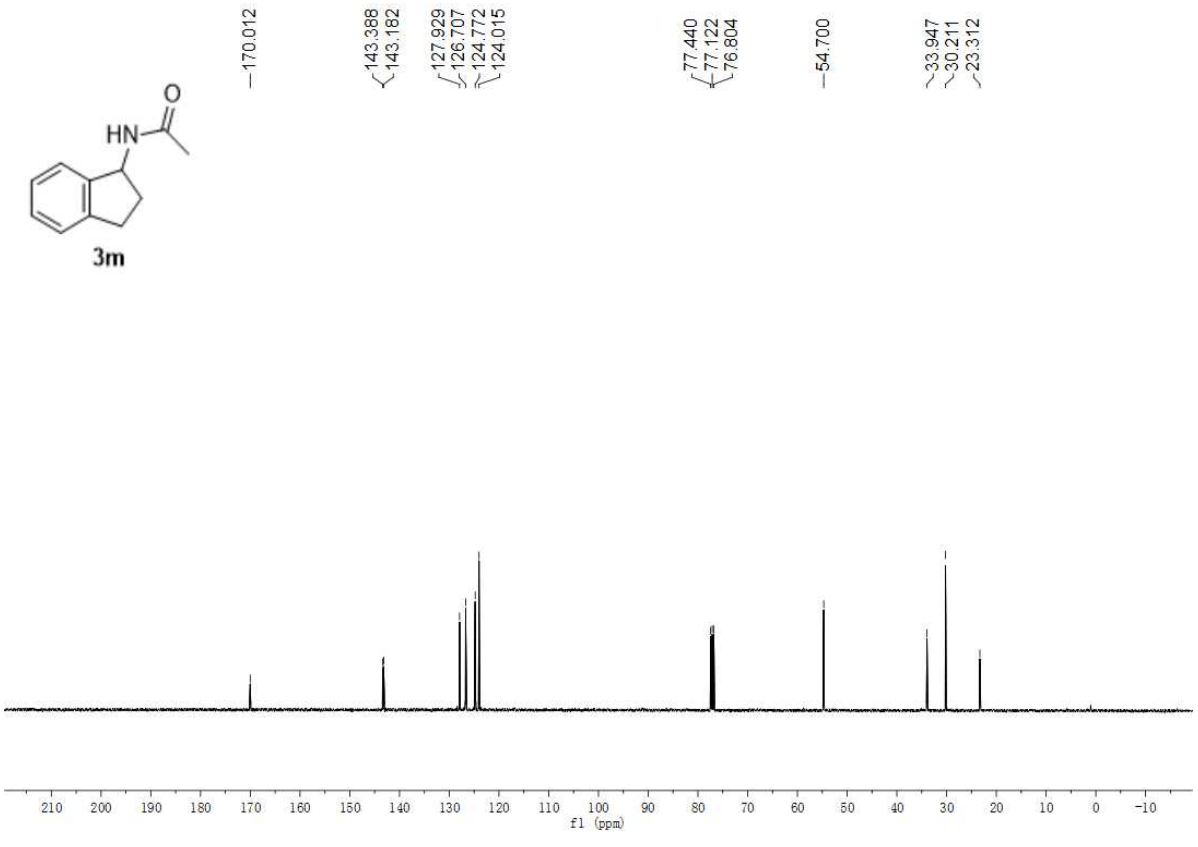
f1 (ppm)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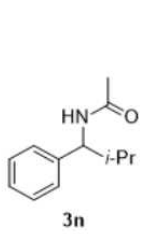
30.211
23.312
HN
O
3m
f1 (ppm)


HN
O
i-Pr

3n

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4.775
4.754
4.733
2.050
2.031
2.014
1.997
0.970
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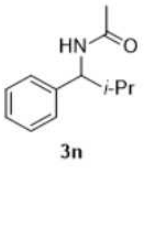
1.06
3.02
3.03
3.00
f1 (ppm)


HN
O
i-Pr

3n

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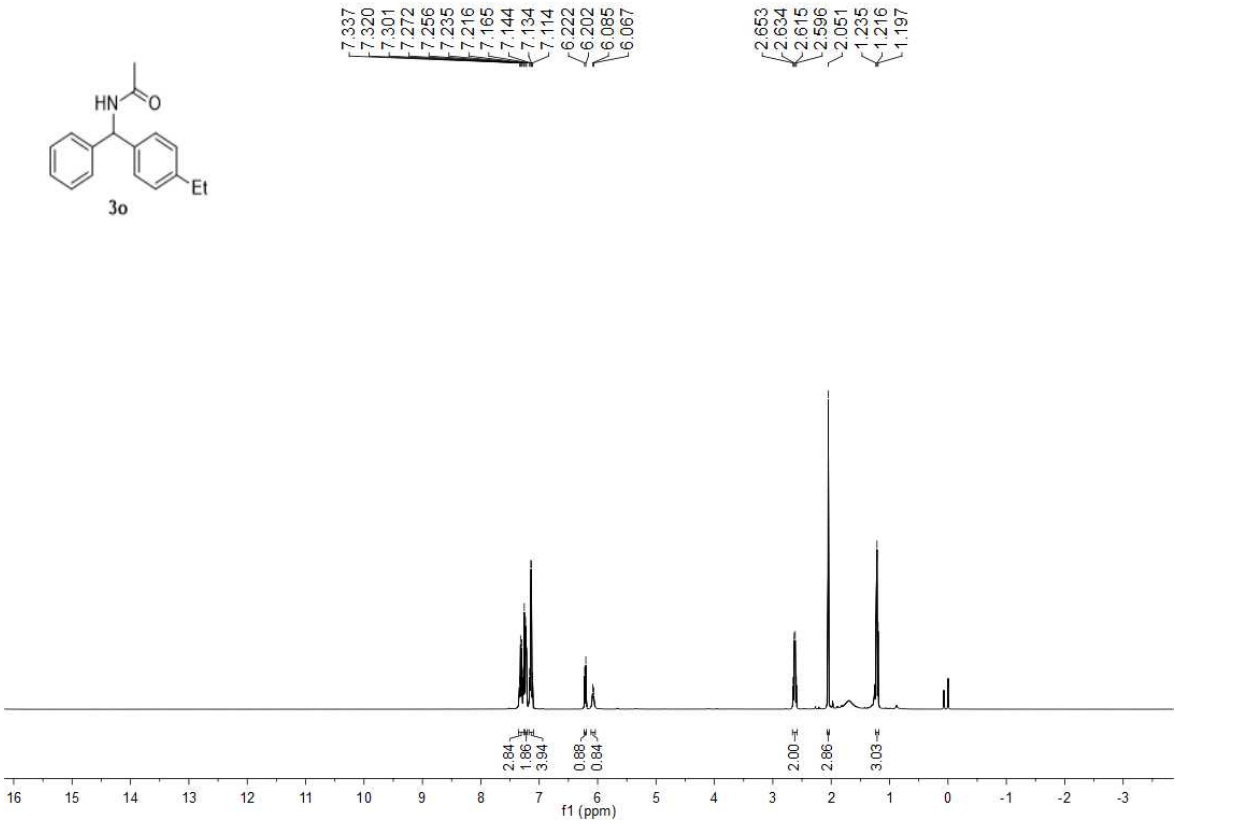
f1 (ppm)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7.134
7.114
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6.085
6.067
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2.615
2.596
2.051
1.235
1.216
1.197
HN
O
Et
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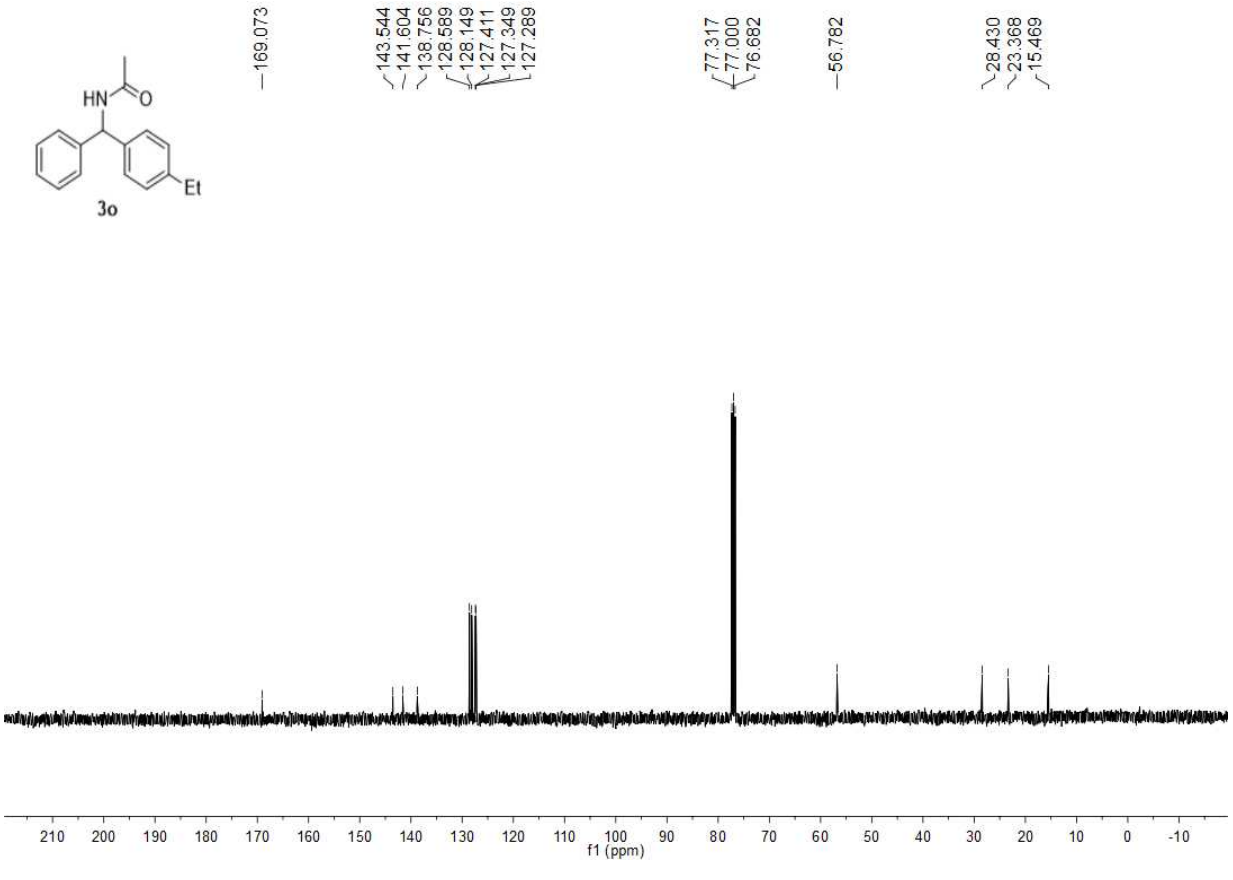
f1 (ppm)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15.469
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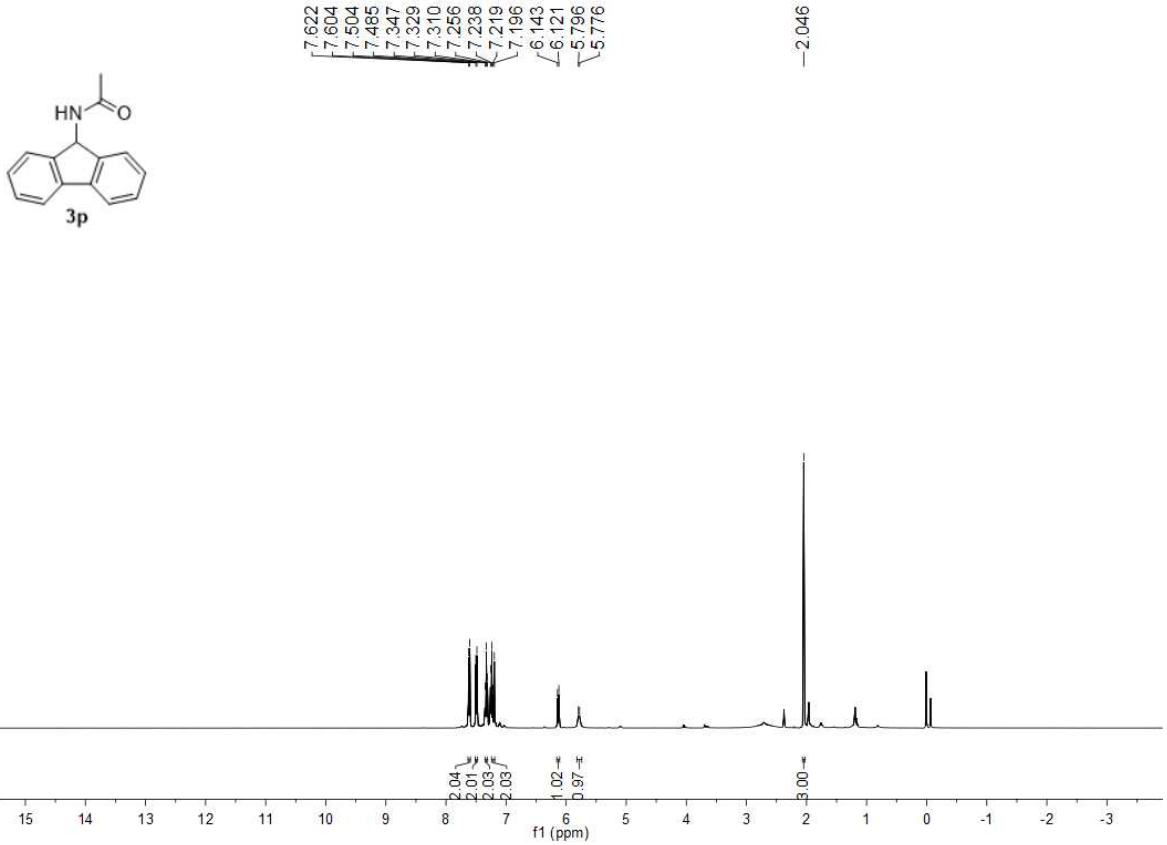
O
Et
3o
f1 (ppm)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7.238
7.219
7.196
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6.121
5.796
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HN
O
3p
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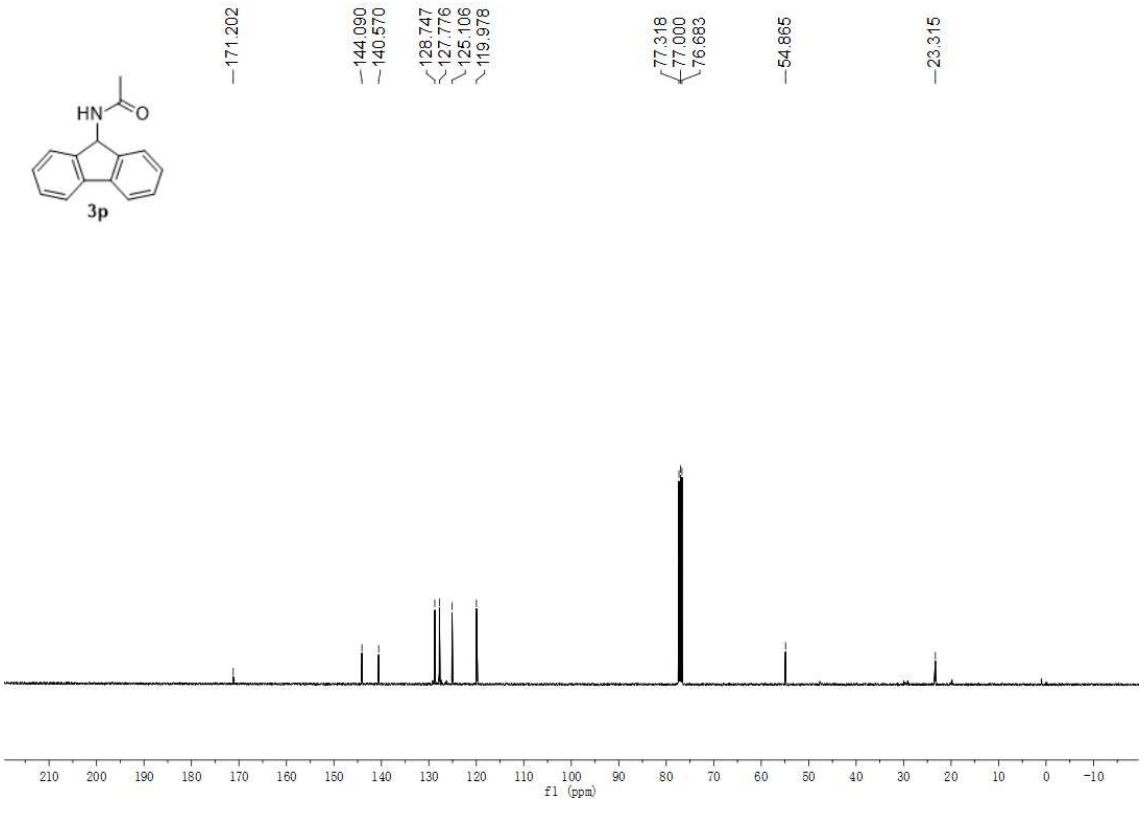
2.01
2.03
2.03
1.02
0.97
3.00
f1 (ppm)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128.747
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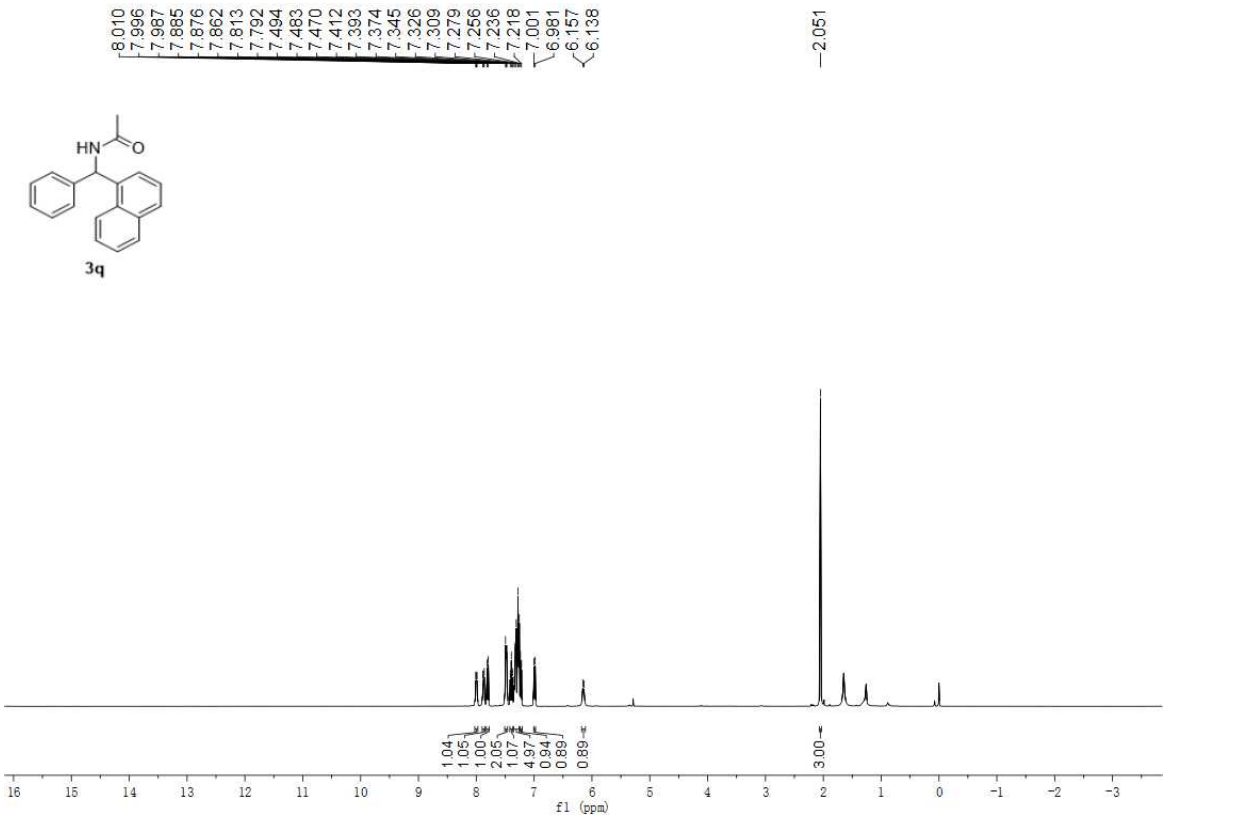
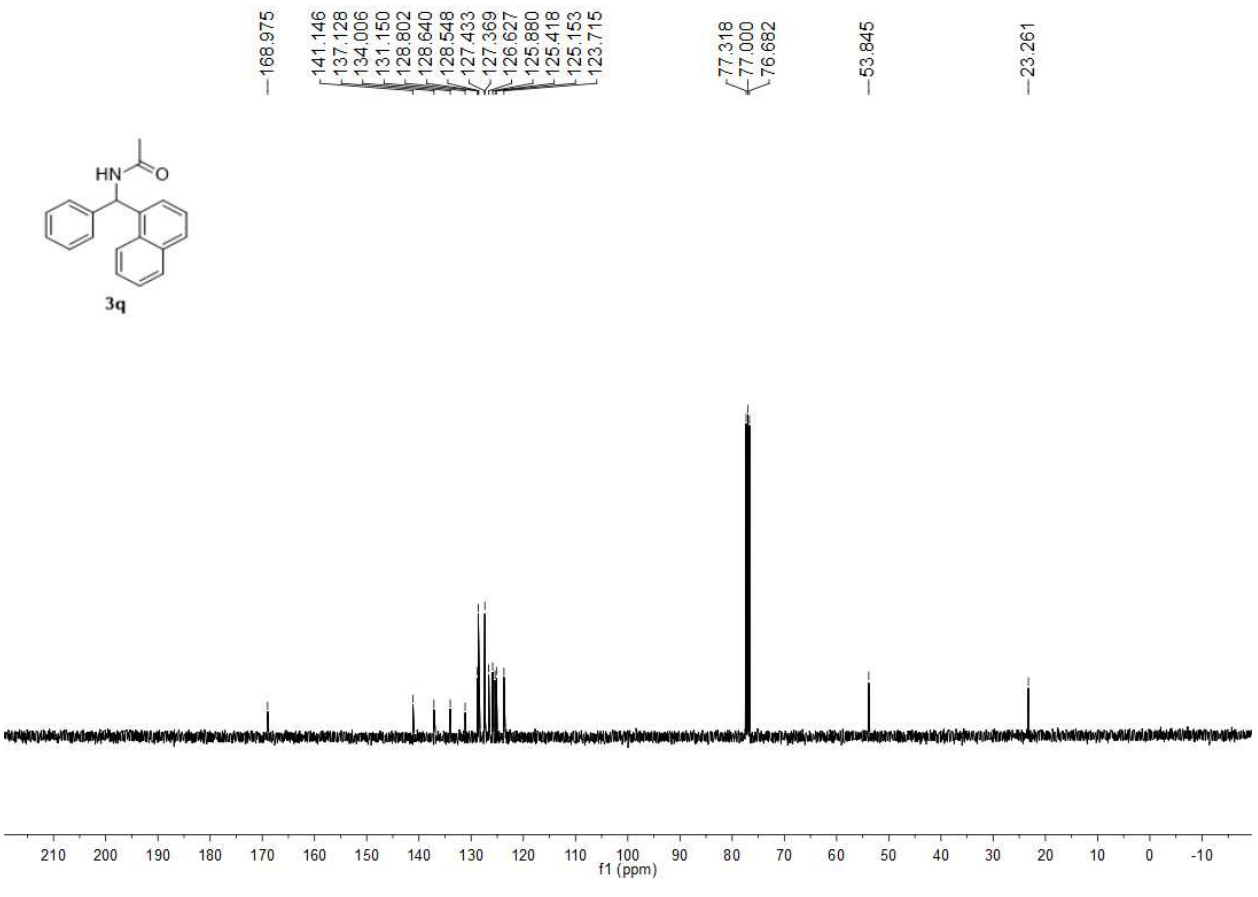
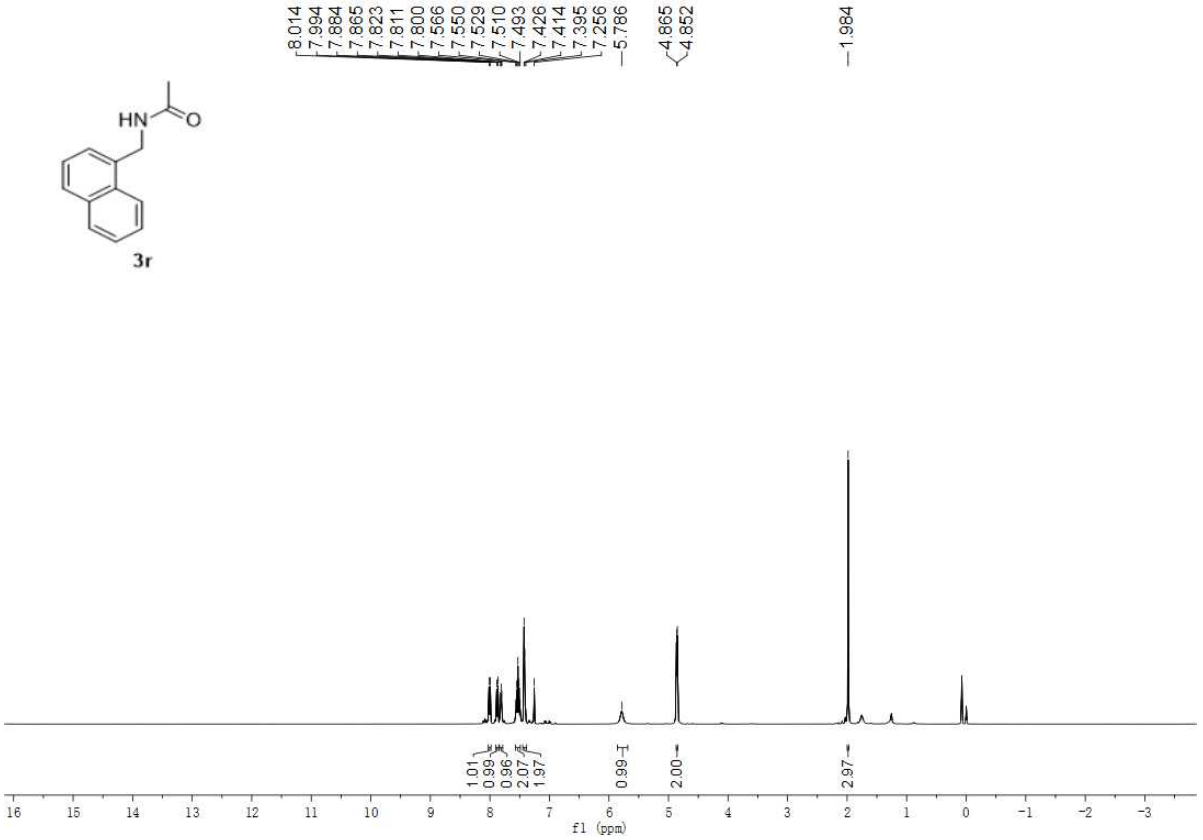
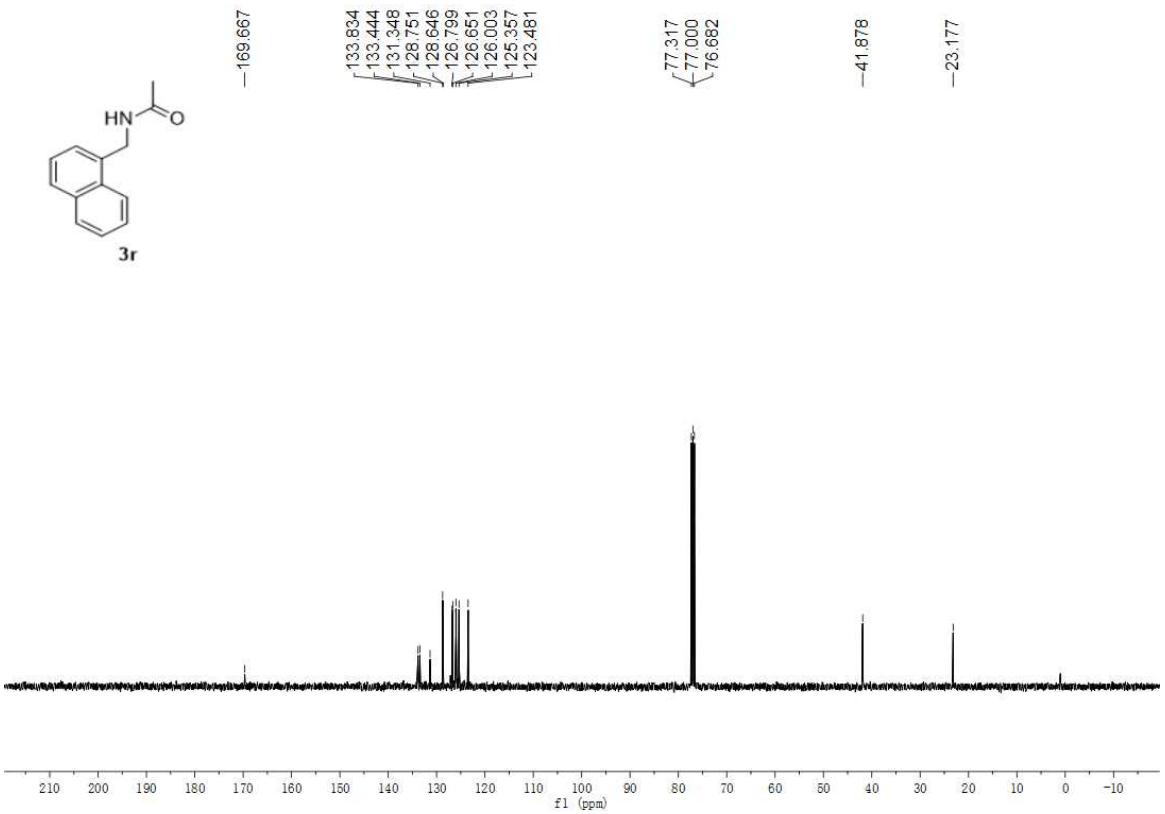
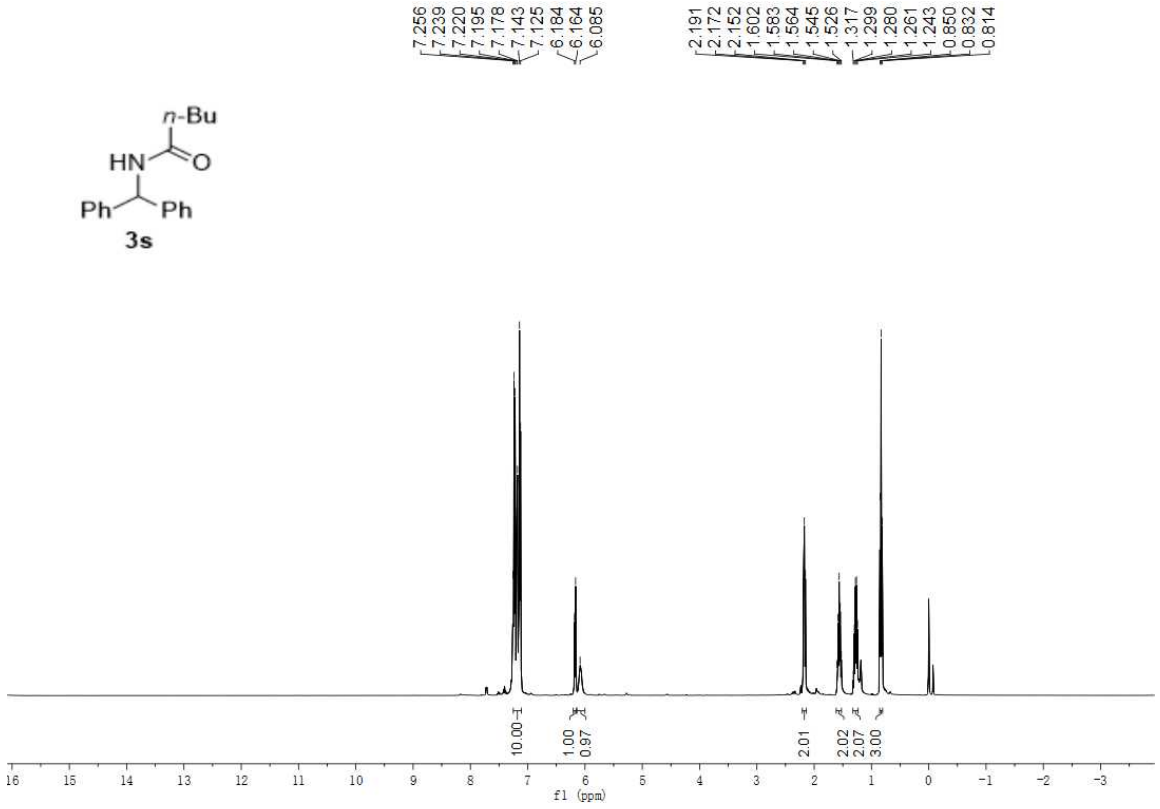
125.106
119.978
77.318
77.000
76.683
54.865
23.315
HN
O
3p
f1 (ppm)

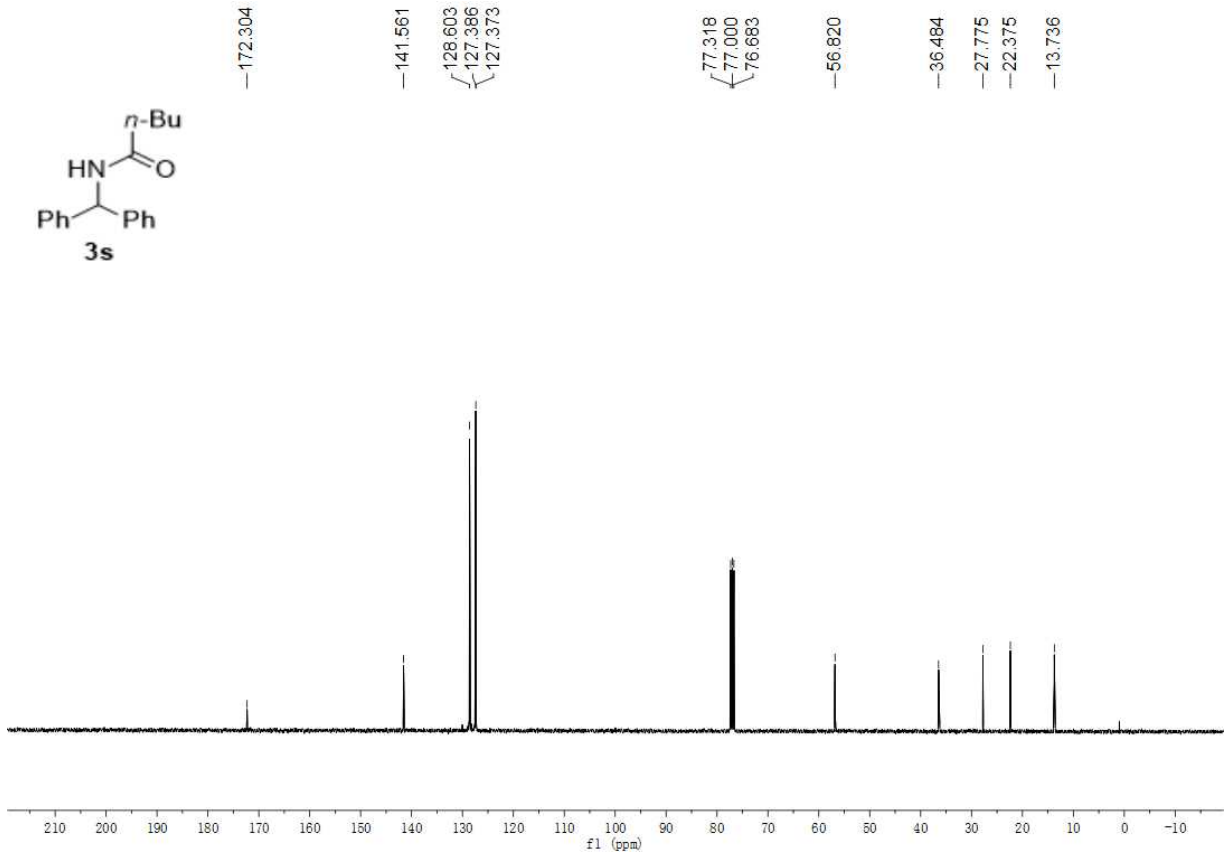

8.010
7.996
7.987
7.885
7.876
7.862
7.813
7.792
7.494
7.483
7.470
7.412
7.393
7.374
7.345
7.326
7.309
7.279
7.256
7.236
7.218
7.001
6.981
6.157
6.138
2.051
HN
O
3q
1.04
1.05
1.00
2.05
1.07
4.97
0.94
0.89
0.89
3.00
f1 (ppm)

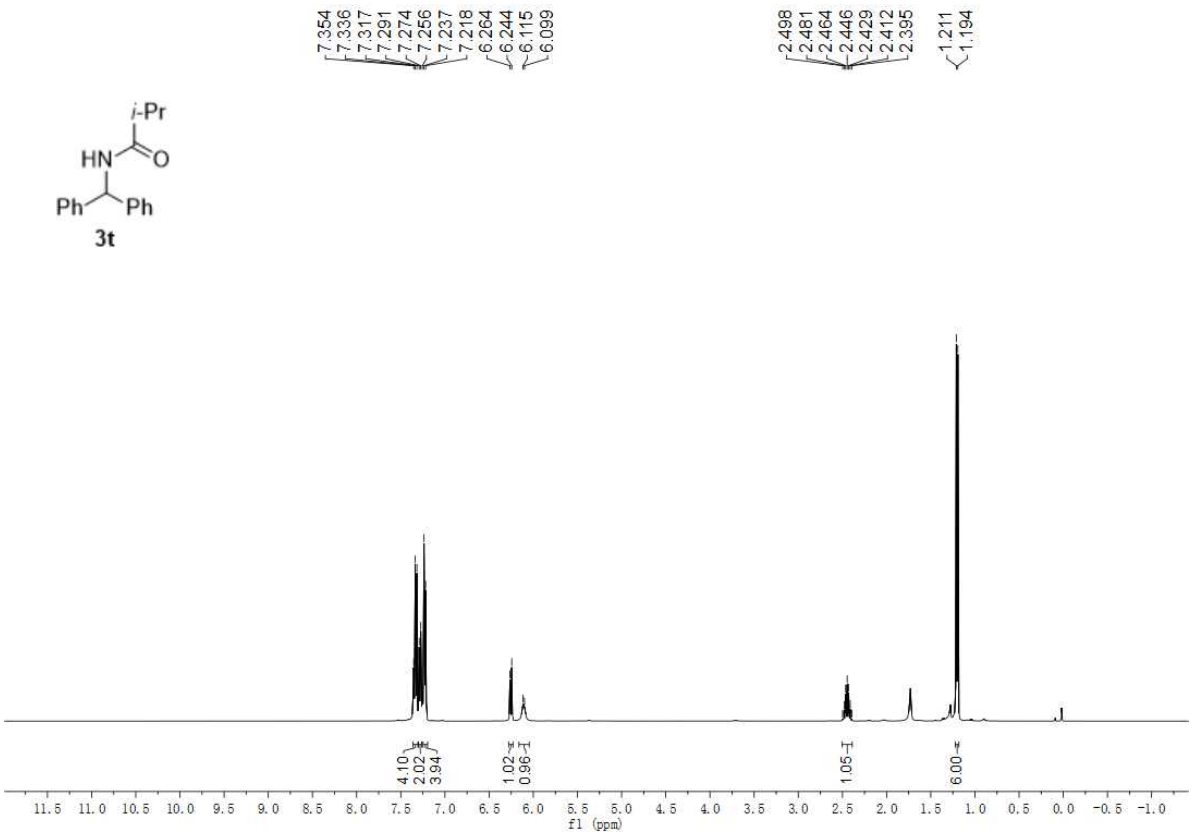

168.975
141.146
137.128
134.006
131.150
128.802
128.640
128.548
127.433
127.369
126.627
125.880
125.418
125.153
123.715
77.318
77.000
76.682
53.845
23.261
HN
O
3q
f1 (ppm)

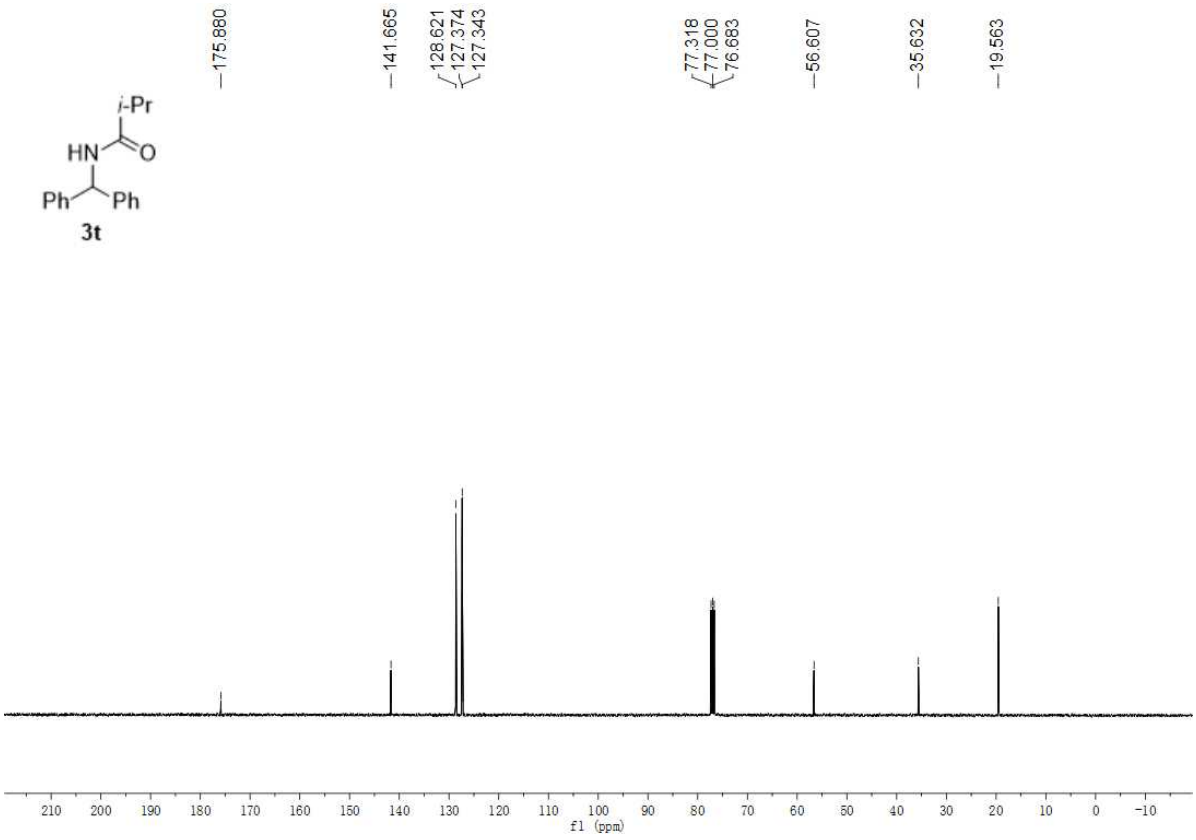

8.014
7.994
7.884
7.865
7.823
7.811
7.800
7.566
7.550
7.529
7.510
7.493
7.426
7.414
7.395
7.256
5.786
4.865
4.852
1.984
HN
O
3r
1.01
0.99
0.96
2.07
1.97
0.99
2.00
2.97
f1 (ppm)

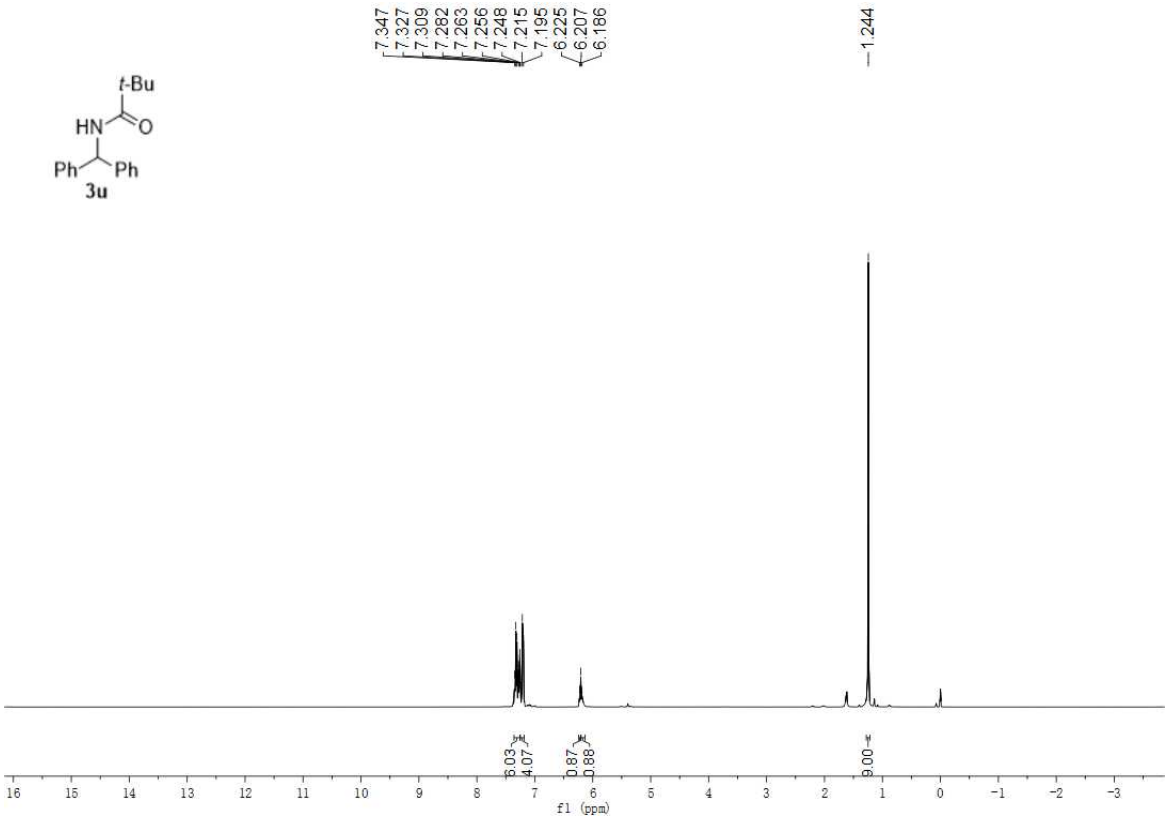

169.667
133.834
133.444
131.348
128.751
128.646
126.799
126.651
126.003
125.357
123.481
77.317
77.000
76.682
41.878
23.177
HN
O
3r
f1 (ppm)

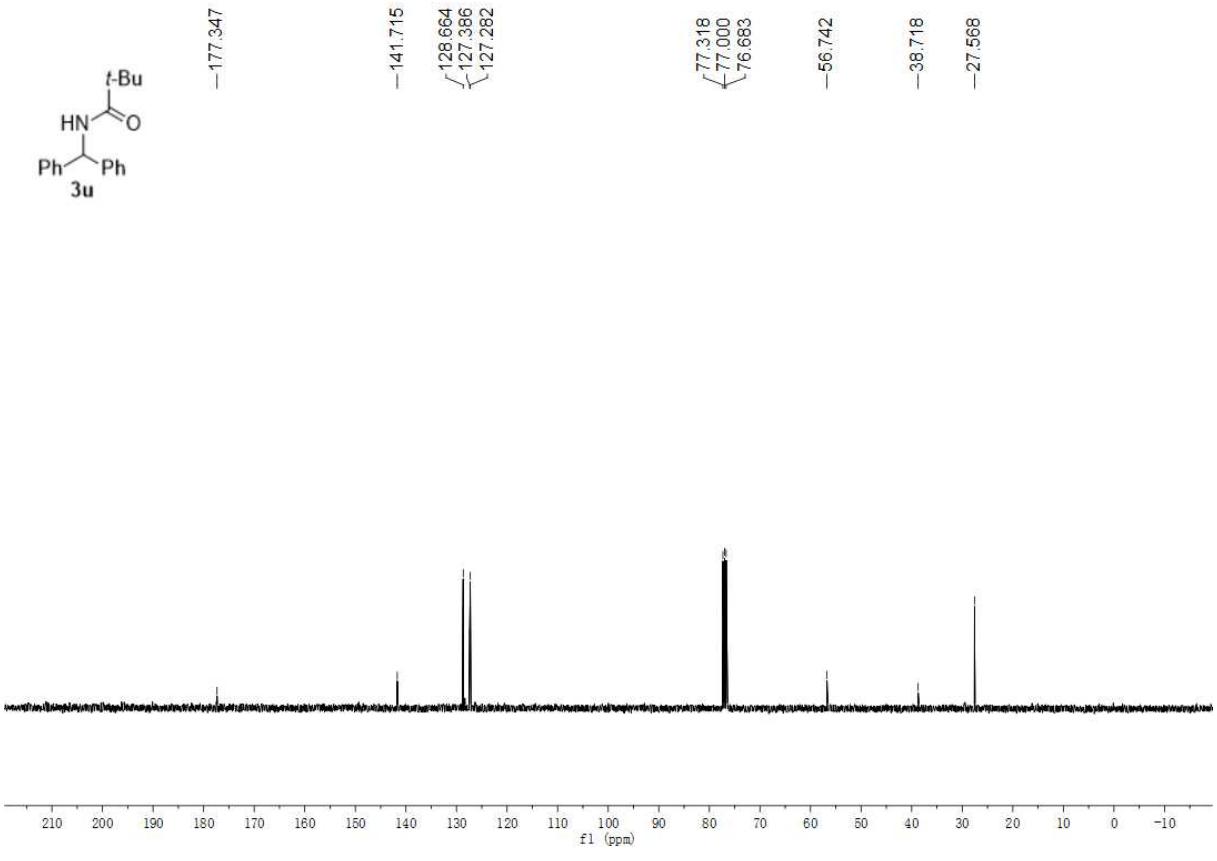

7.256
7.239
7.220
7.195
7.178
7.143
7.125
6.184
6.164
6.085
2.191
2.172
2.152
1.602
1.583
1.564
1.545
1.526
1.317
1.299
1.280
1.261
1.243
0.850
0.832
0.814
n-Bu
HN
O
Ph
Ph
3s
10.00
1.00
0.97
2.01
2.02
2.07
3.00
f1 (ppm)

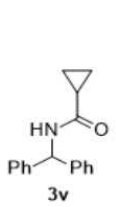

172.304
141.561
128.603
127.386
127.373
77.318
77.000
76.683
56.820
36.484
27.775
22.375
13.736
n-Bu
HN
O
Ph
Ph
3s
f1 (ppm)


7.354
7.336
7.317
7.291
7.274
7.256
7.237
7.218
6.264
6.244
6.115
6.099
2.498
2.481
2.464
2.446
2.429
2.412
2.395
1.211
1.194
i-Pr
HN
O
Ph
Ph
3t
4.10
2.02
3.94
1.02
0.96
1.05
6.00
f1 (ppm)


175.880
141.665
128.621
127.374
127.343
77.318
77.000
76.683
56.607
35.632
19.563
i-Pr
HN
O
Ph
Ph
3t
f1 (ppm)

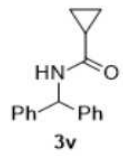

7.347
7.327
7.309
7.282
7.263
7.256
7.248
7.215
7.195
6.225
6.207
6.186
1.244
t-Bu
HN
O
Ph
Ph
3u
6.03
4.07
0.87
0.88
9.00
f1 (ppm)


177.347
141.715
128.664
127.386
127.282
77.318
77.000
76.683
56.742
38.718
27.568
t-Bu
HN
O
Ph
Ph
3u
f1 (ppm)


HN
O
Ph
Ph

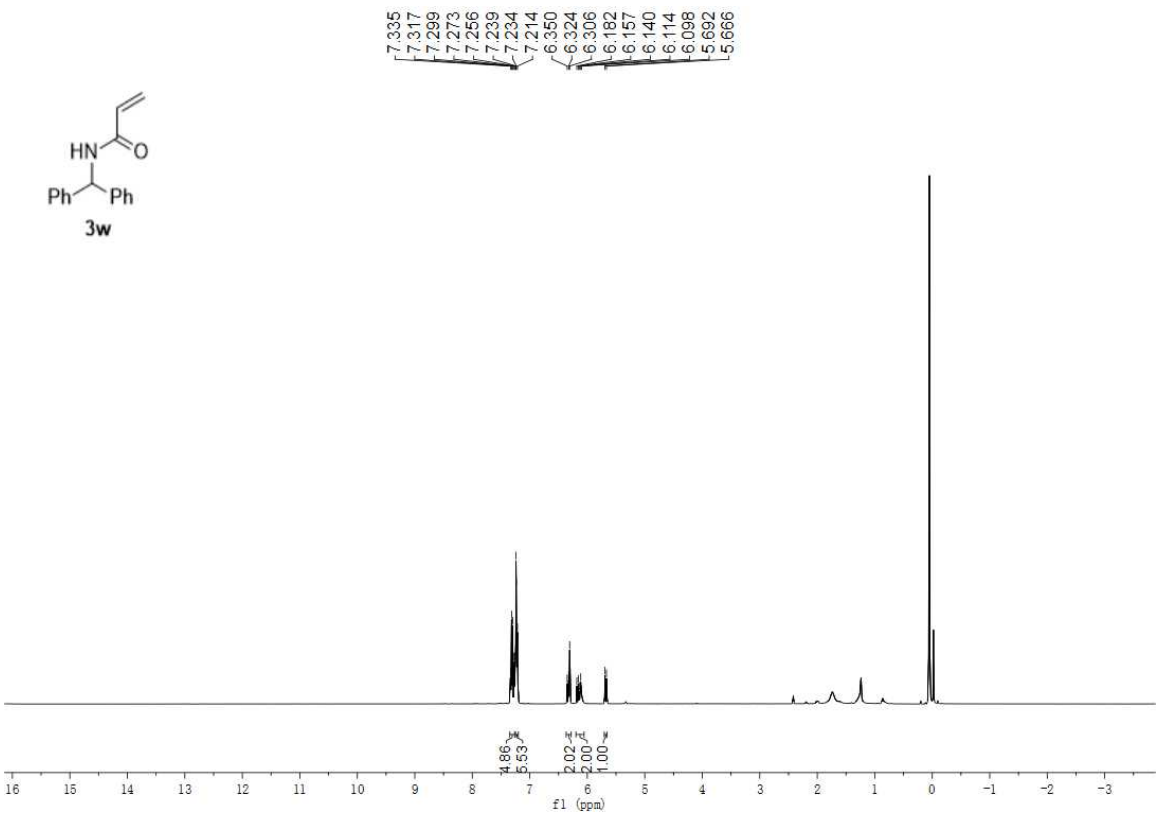
3v

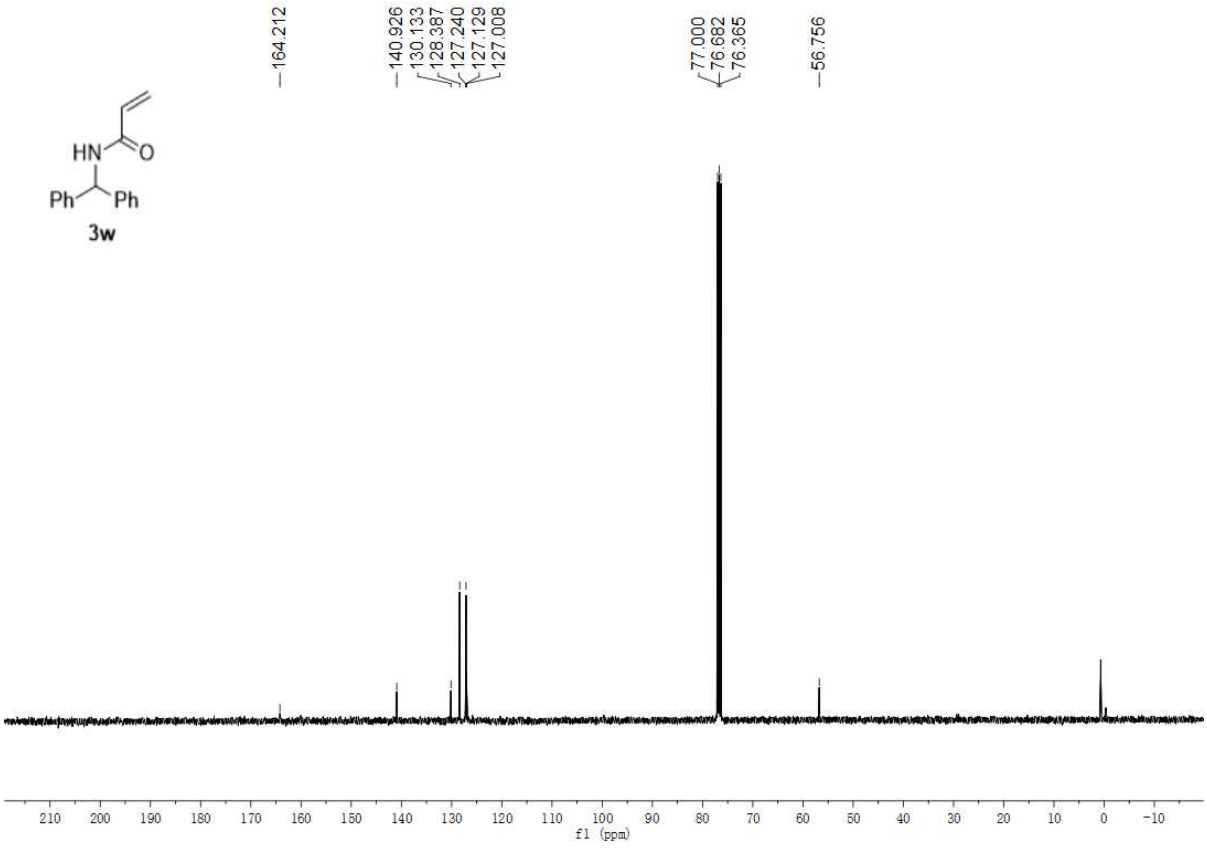
7.352
7.335
7.304
7.276
7.256
6.278
1.441
1.025
0.783
0.770
4.01
6.01
1.97
1.09
2.01
2.00
f1 (ppm)

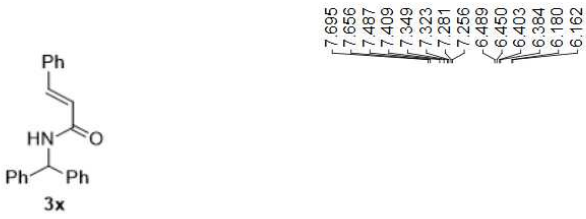

HN
O
Ph
Ph

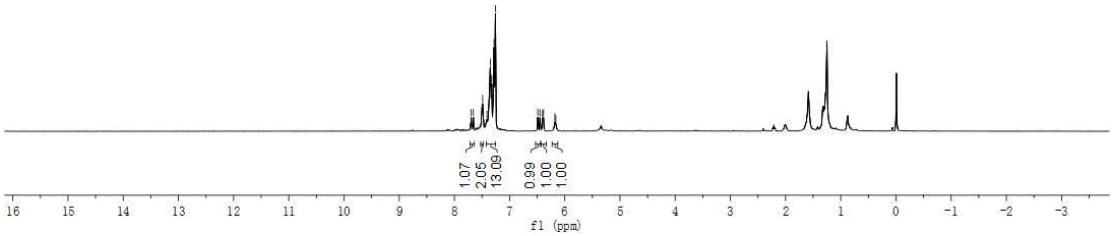
3v

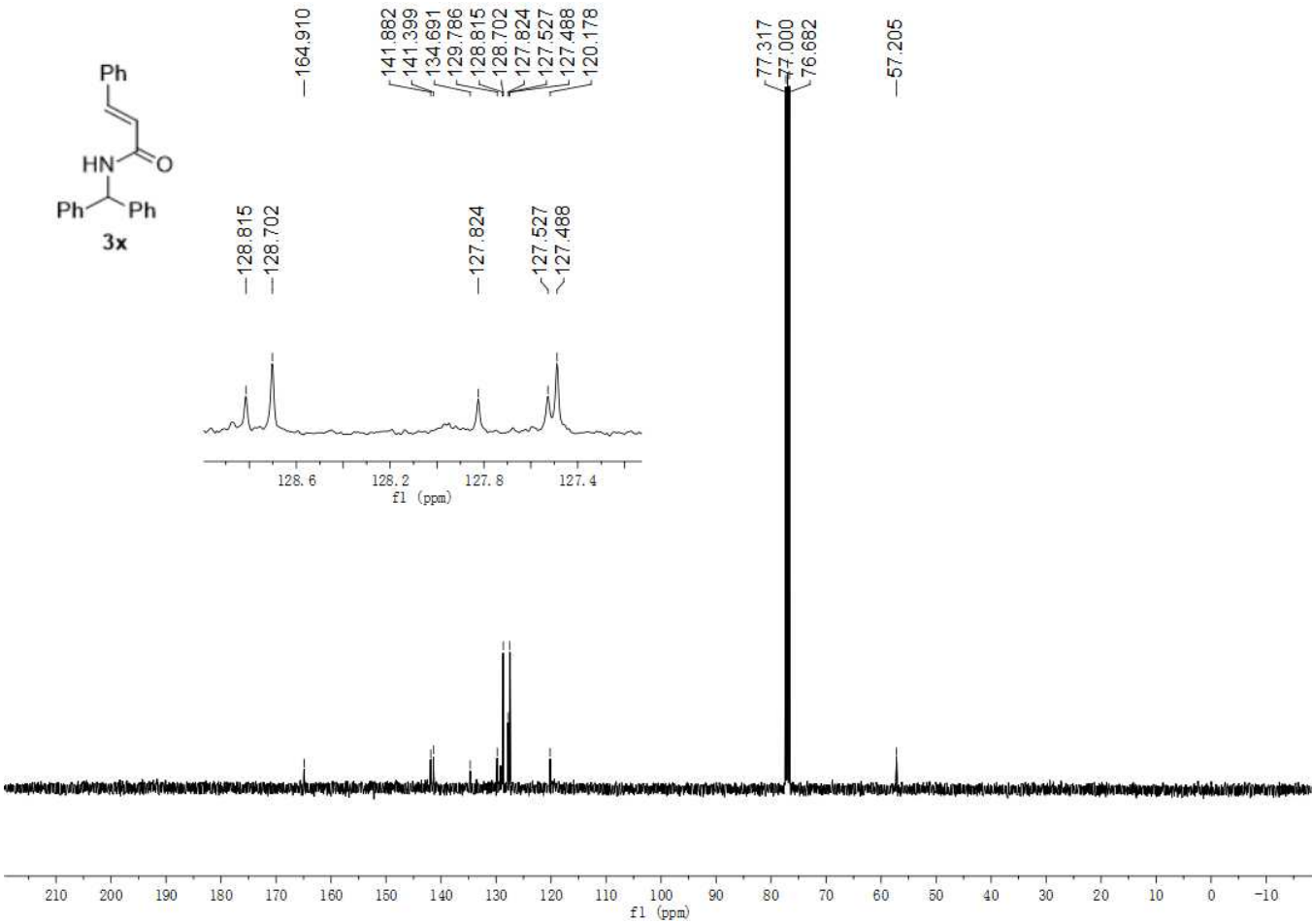
172.584
141.689
128.642
127.406
77.317
77.000
76.682
57.067
14.826
7.354
f1 (ppm)

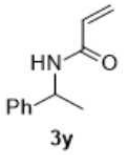

7.335
7.317
7.299
7.273
7.256
7.239
7.234
7.214
6.350
6.324
6.306
6.182
6.157
6.140
6.114
6.098
5.692
5.666
HN
O
Ph
Ph
3w
4.86
5.53
2.02
2.00
1.00
f1 (ppm)


164.212
140.926
130.133
128.387
127.240
127.129
127.008
77.000
76.682
76.365
56.756
HN
O
Ph
Ph
3w
f1 (ppm)

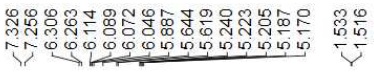

7.695
7.656
7.487
7.409
7.349
7.323
7.281
7.256
6.489
6.450
6.403
6.384
6.180
6.162
Ph
HN
O
Ph
Ph
3x

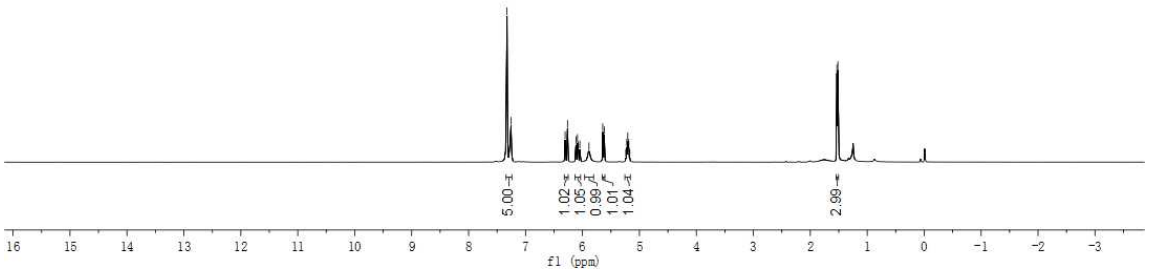

1.07
2.05
13.09
0.99
1.00
1.00
f1 (ppm)

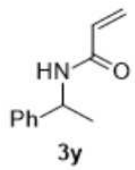

164.910
141.882
141.399
134.691
129.786
128.815
128.702
127.824
127.527
127.488
120.178
77.317
77.000
76.682
57.205
128.815
128.702
127.824
127.527
127.488
Ph
HN
O
Ph
Ph
3x
128.6
128.2
127.8
127.4
f1 (ppm)
f1 (ppm)


HN
O
Ph

3y

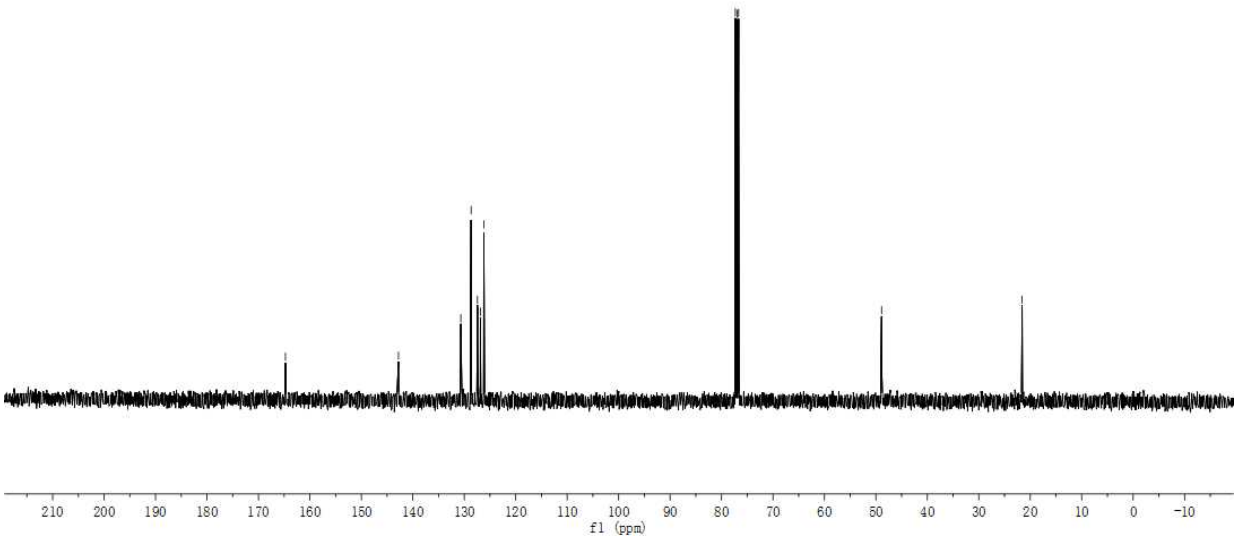

7.326
7.256
6.306
6.263
6.114
6.089
6.072
6.046
5.887
5.644
5.619
5.240
5.223
5.205
5.187
5.170
1.533
1.516

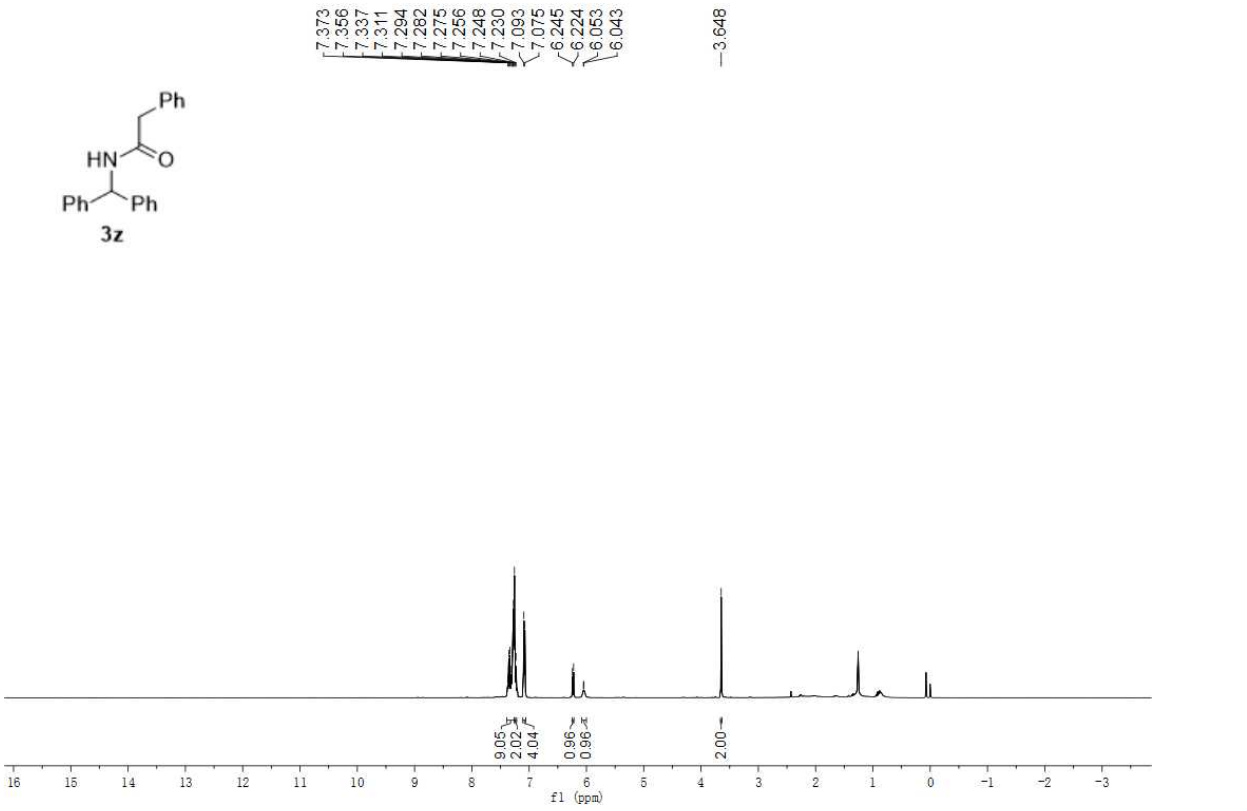

5.00
1.02
1.05
0.99
1.01
1.04
2.99
f1 (ppm)


HN
O
Ph

3y


164.760
142.784
130.668
128.672
127.434
126.865
126.189
77.318
77.000
76.682
48.893
21.576


f1 (ppm)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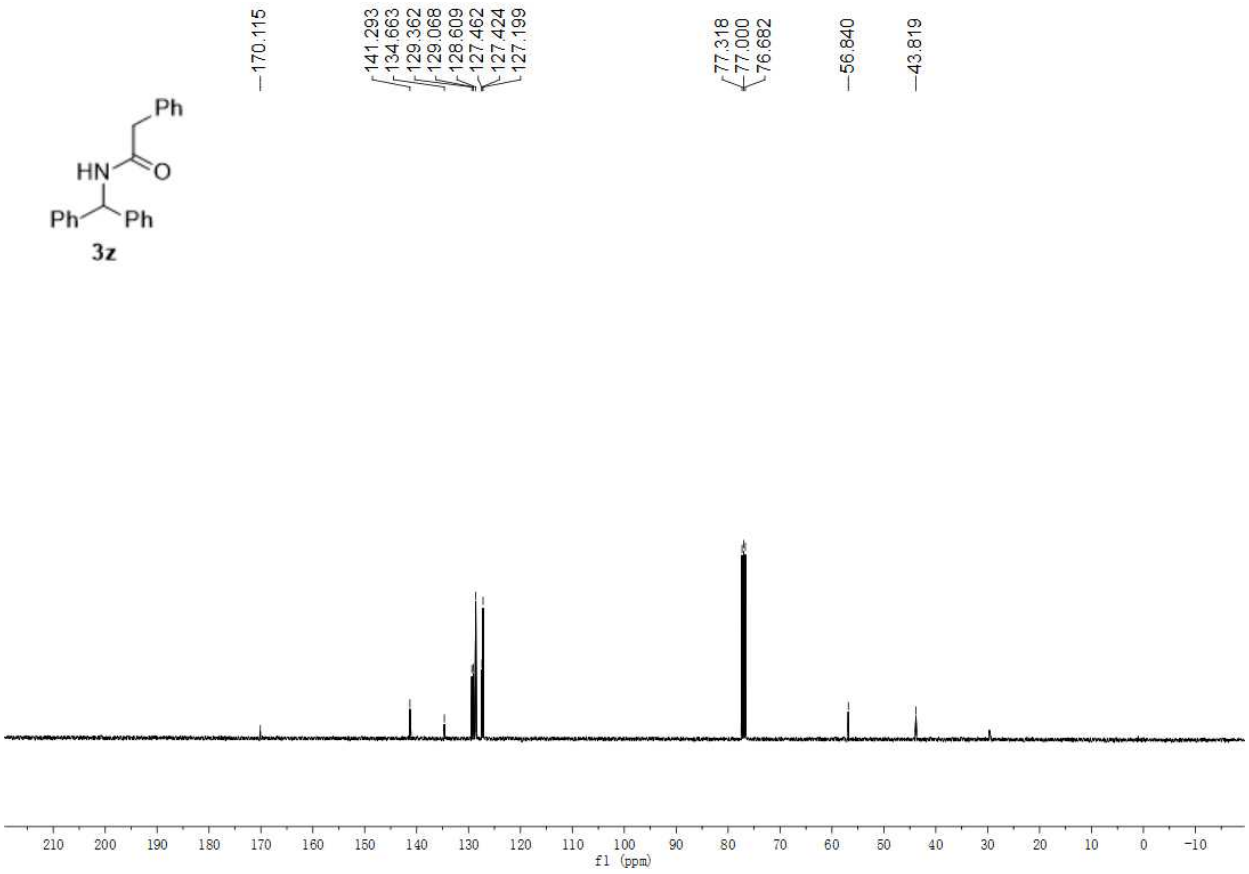
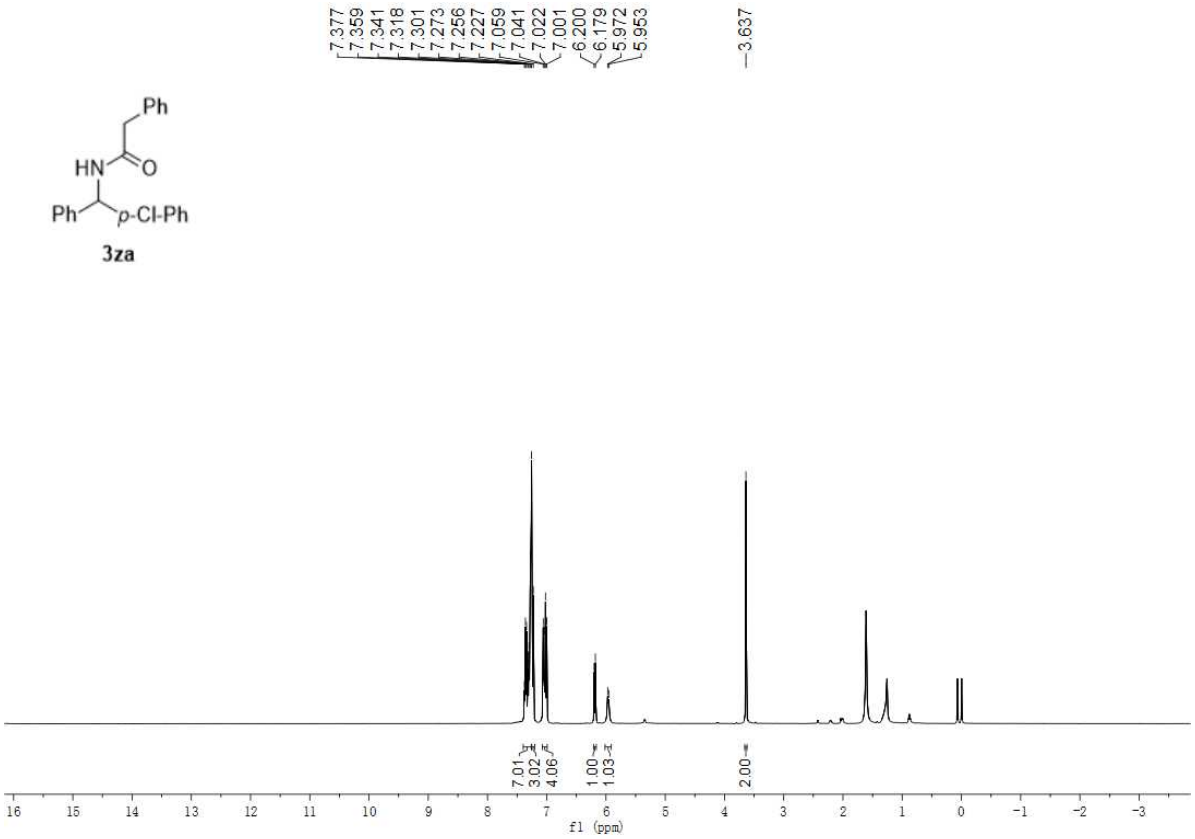
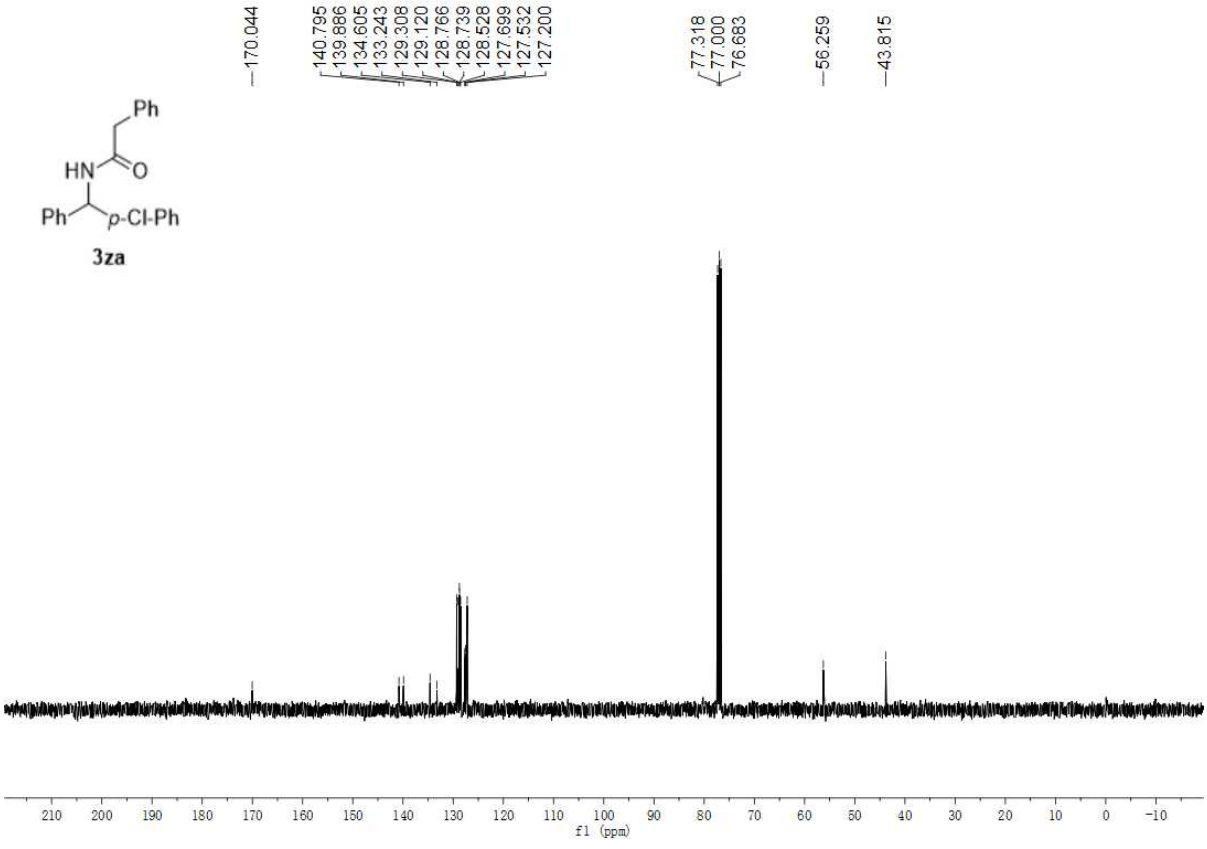
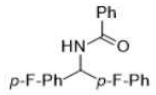
7.356
7.337
7.311
7.294
7.282
7.275
7.256
7.248
7.230
7.093
7.075
6.245
6.224
6.053
6.043
3.648
Ph
HN
O
Ph
Ph
3z
9.05
2.02
4.04
0.96
0.98
2.00
f1 (ppm)

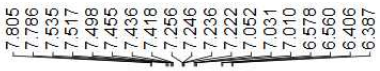

170.115
141.293
134.663
129.362
129.068
128.609
127.462
127.424
127.199
77.318
77.000
76.682
56.840
43.819
Ph
HN
O
Ph
Ph
3z
f1 (ppm)


7.377
7.359
7.341
7.318
7.301
7.273
7.256
7.227
7.059
7.041
7.022
7.001
6.200
6.179
5.972
5.953
3.637
Ph
HN
O
Ph
p-Cl-Ph
3za
7.01
3.02
4.06
1.00
1.03
2.00
f1 (ppm)


170.044
140.795
139.886
134.605
133.243
129.308
129.120
128.766
128.739
128.528
127.699
127.532
127.200
77.318
77.000
76.683
56.259
43.815
Ph
HN
O
Ph
p-Cl-Ph
3za
f1 (ppm)


Ph
HN
O
p-F-Ph
p-F-Ph

3zb


7.805
7.786
7.535
7.517
7.498
7.455
7.436
7.418
7.256
7.246
7.236
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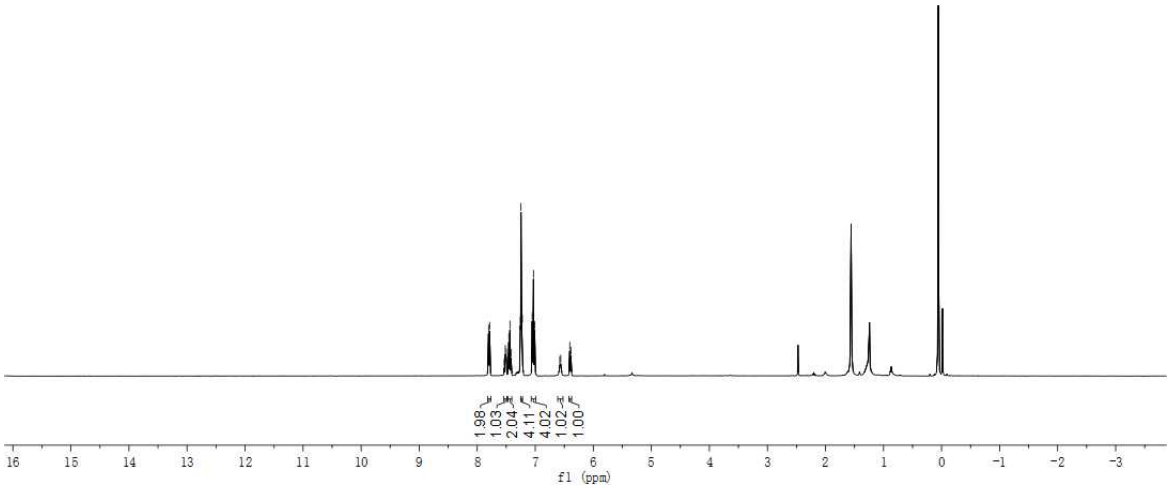
7.031
7.010
6.578
6.560
6.406
6.387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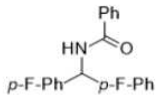
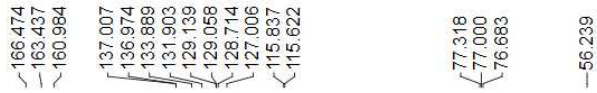
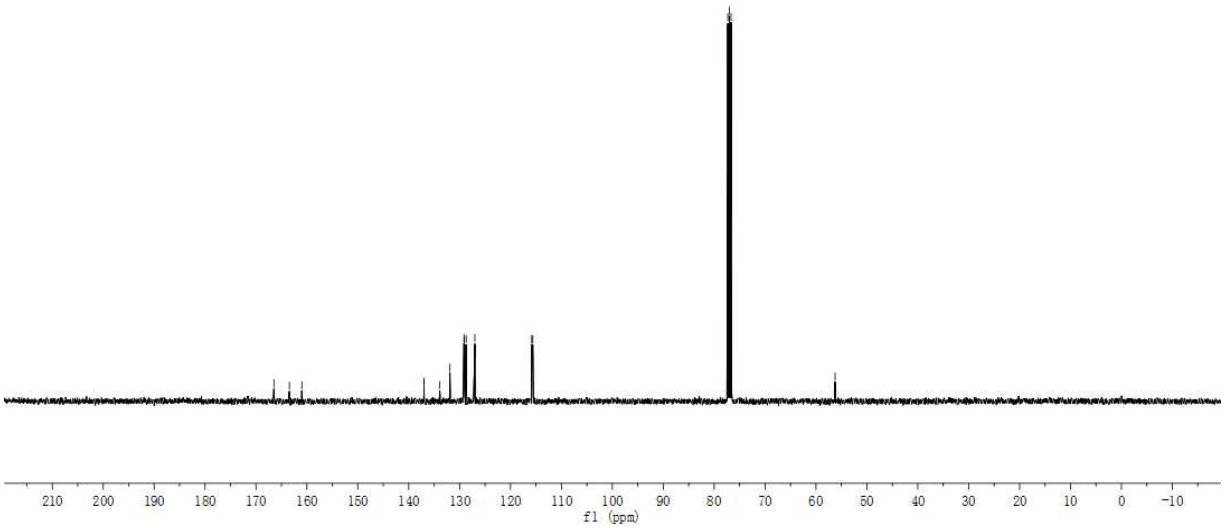
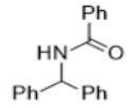
1.03
2.04
4.11
4.02
1.02
1.00
f1 (ppm)


Ph
HN
O
p-F-Ph
p-F-Ph

3zb

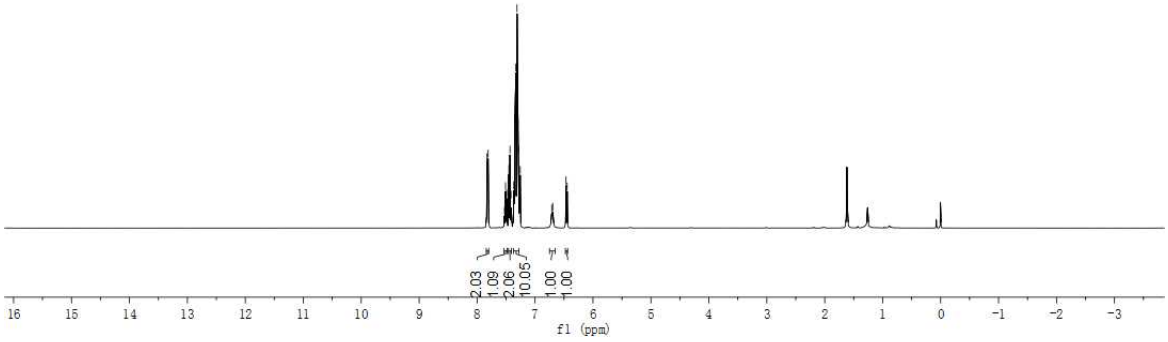

166.474
163.437
160.984
137.007
136.974
133.889
131.903
129.139
129.058
128.714
127.006
115.837
115.622
77.318
77.000
76.683
56.239

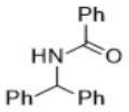

f1 (ppm)


Ph
HN
O
Ph
Ph

3zc

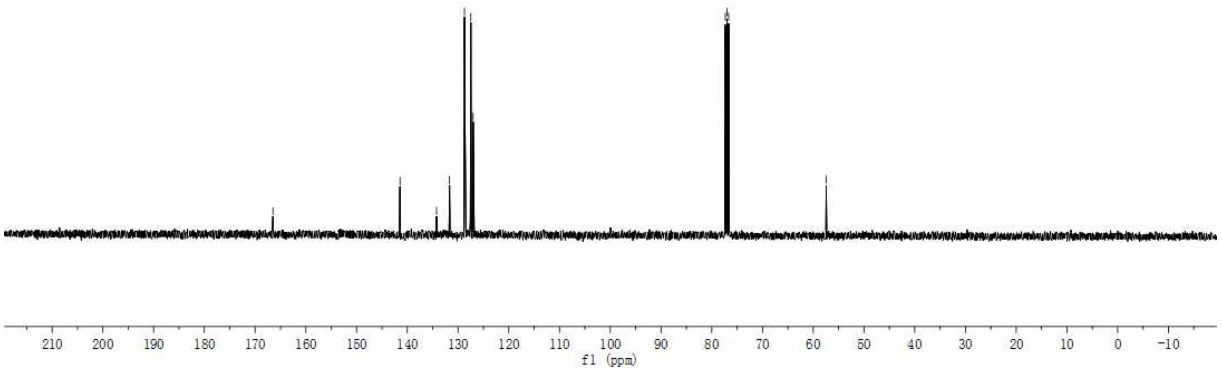

7.828
7.809
7.527
7.509
7.490
7.452
7.432
7.414
7.367
7.348
7.331
7.311
7.304
7.293
7.285
7.256
6.712
6.694
6.465
6.446

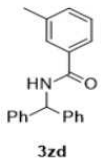

2.03
1.09
2.06
10.05
1.00
1.00
f1 (ppm)


Ph
HN
O
Ph
Ph

3zc

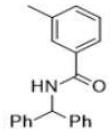

166.482
141.437
134.238
131.673
128.735
128.619
127.561
127.484
127.027
77.317
77.000
76.682
57.445


f1 (ppm)


HN
O
Ph
Ph

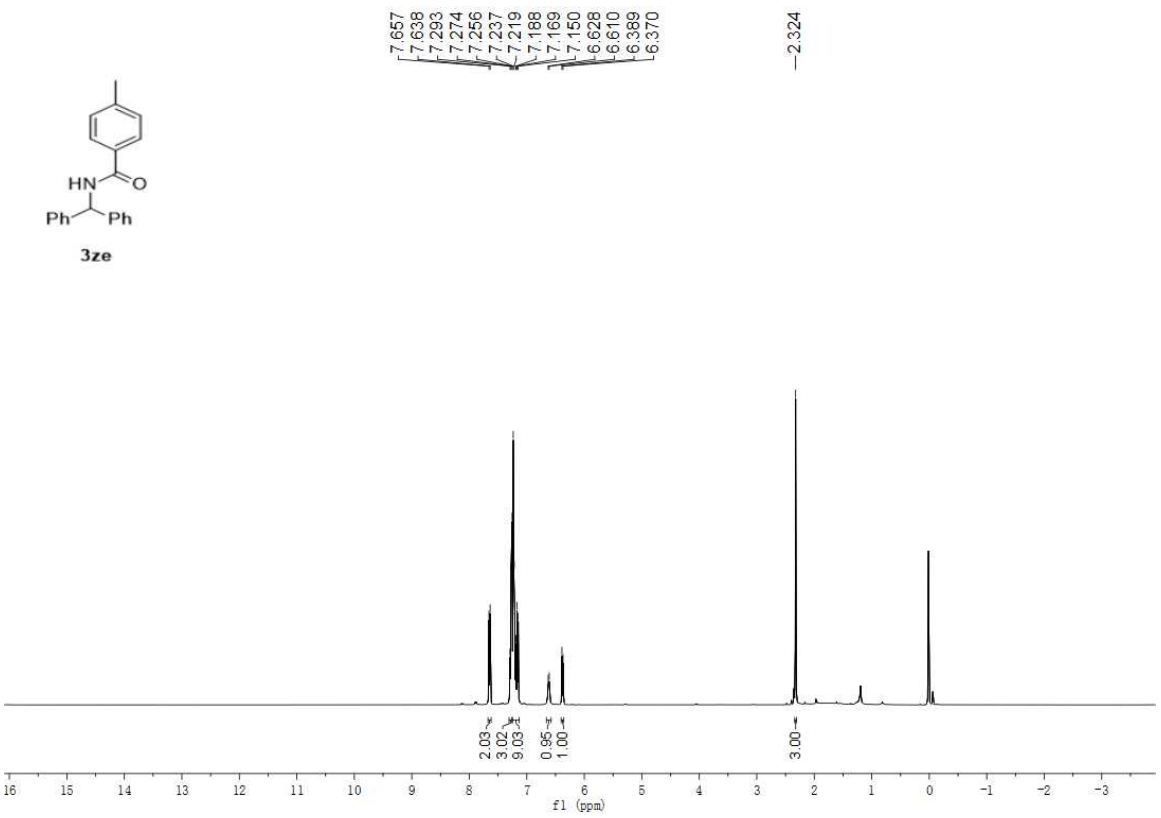
3zd

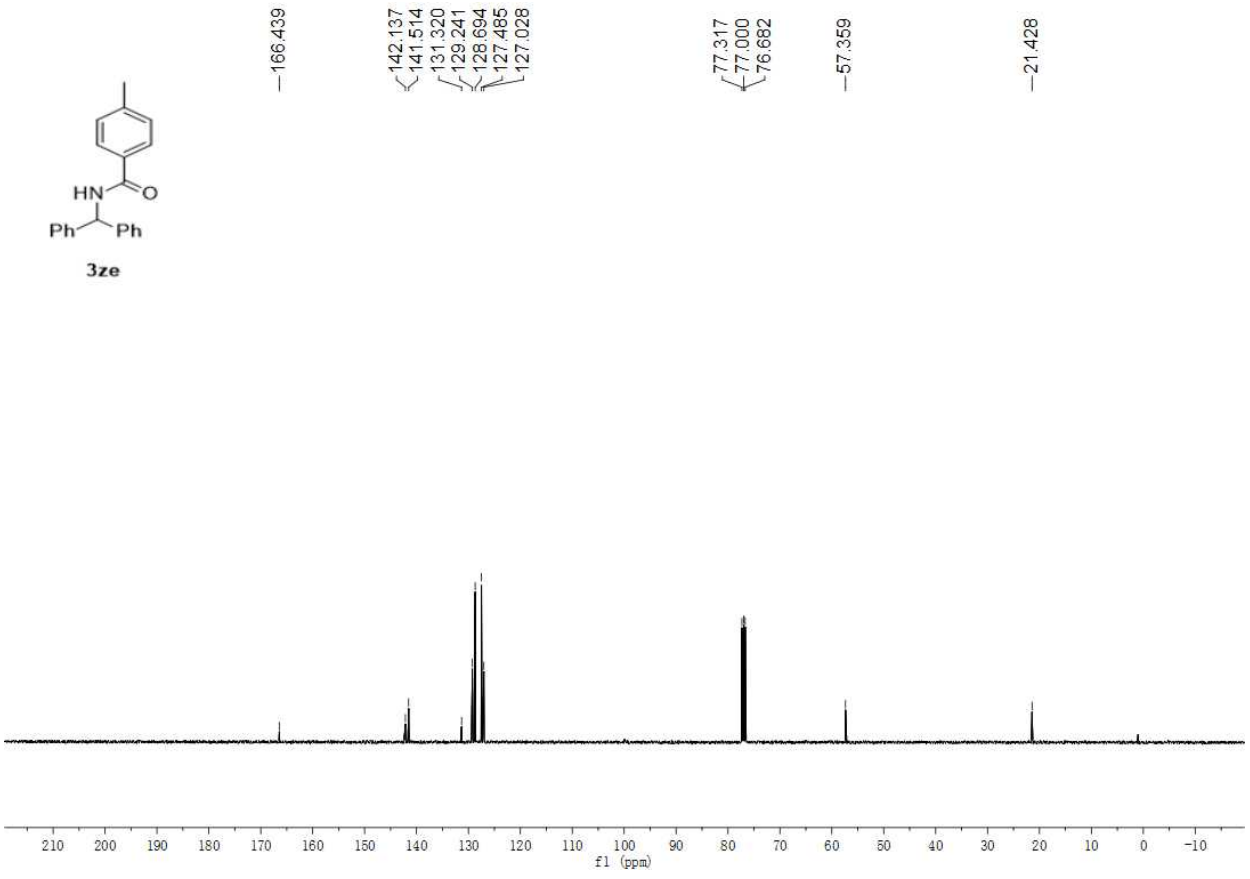
7.559
7.531
7.520
7.510
7.293
7.274
7.256
7.246
7.235
7.217
7.192
7.181
6.594
6.576
6.388
6.369
2.318
1.02
1.04
3.00
8.96
1.00
1.00
3.00
f1 (ppm)

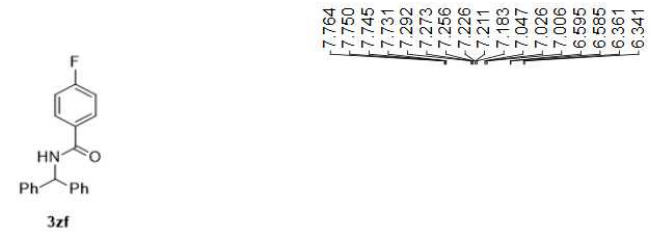

HN
O
Ph
Ph

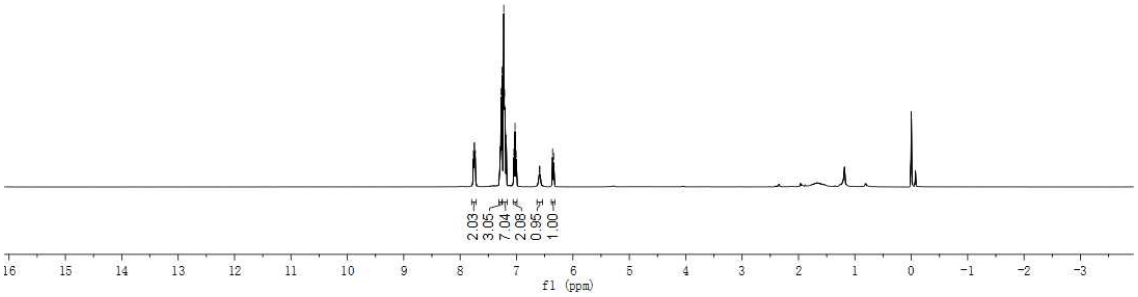
3zd

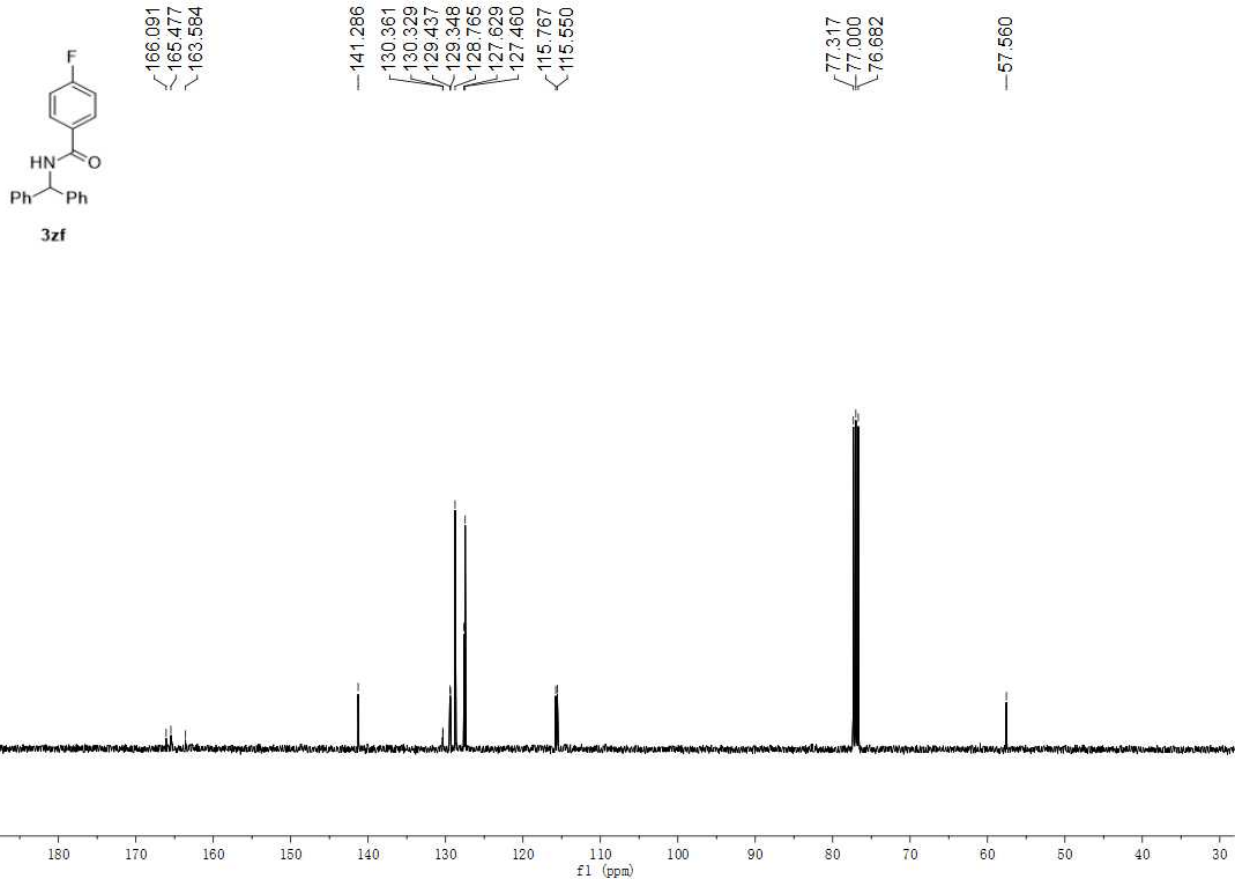
166.672
141.494
138.521
134.228
132.412
128.725
128.496
127.750
127.540
127.506
123.986
77.318
77.000
76.683
57.395
21.330
f1 (ppm)


7.657
7.638
7.293
7.274
7.256
7.237
7.219
7.188
7.169
7.150
6.628
6.610
6.389
6.370
2.324
HN
O
Ph
Ph
3ze
2.03
3.02
9.03
0.95
1.00
3.00
f1 (ppm)

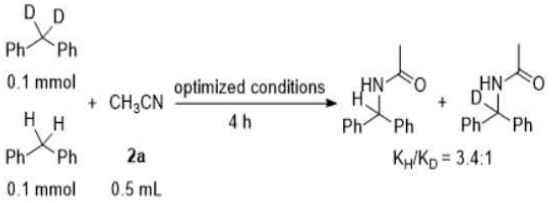

166.439
142.137
141.514
131.320
129.241
128.694
127.485
127.028
77.317
77.000
76.682
57.359
21.428
HN
O
Ph
Ph
3ze
f1 (ppm)

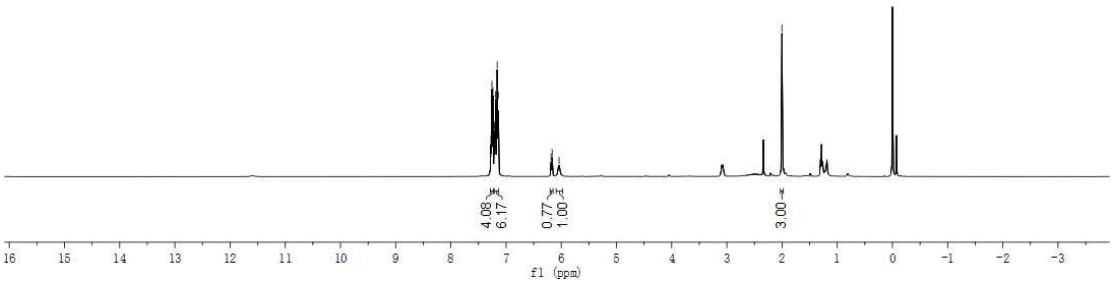

7.764
7.750
7.745
7.731
7.292
7.273
7.256
7.226
7.211
7.183
7.047
7.026
7.006
6.595
6.585
6.361
6.341
F
HN
O
Ph
Ph
3zf


2.03
3.05
7.04
2.08
0.95
1.00
f1 (ppm)

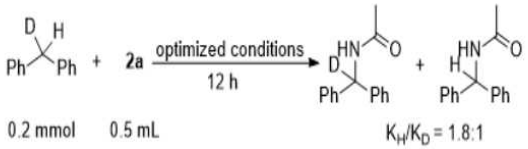

166.091
165.477
163.584
141.286
130.361
130.329
129.437
129.348
128.765
127.629
127.460
115.767
115.550
77.317
77.000
76.682
57.560
F
HN
O
Ph
Ph
3zf
f1 (ppm)

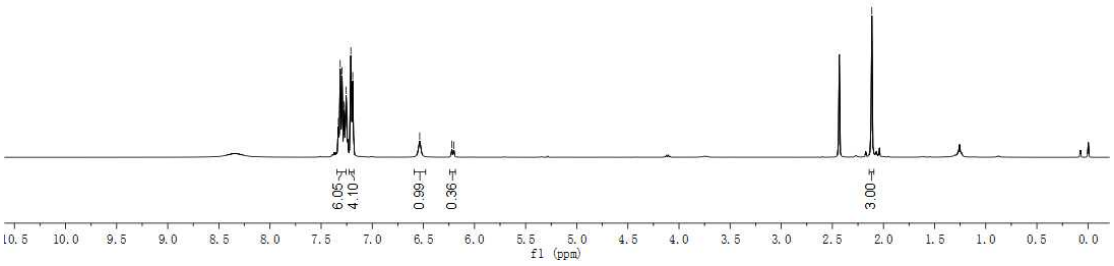

7.274
7.256
7.238
7.212
7.189
7.161
7.142
6.185
6.165
6.039
2.002


D D
Ph Ph
0.1 mmol
+ CH3CN
optimized conditions
4 h
HN O
H
Ph Ph
+
HN O
D
Ph Ph
H H
Ph Ph
0.1 mmol
2a
0.5 mL
KH/KD = 3.4:1


4.08
6.17
0.77
1.00
3.00
f1 (ppm)


7.331
7.313
7.295
7.275
7.256
7.209
7.191
6.536
6.221
6.202
2.114


D H
Ph Ph
+
2a
optimized conditions
12 h
HN O
D
Ph Ph
+
HN O
H
Ph Ph
0.2 mmol
0.5 mL
KH/KD = 1.8:1


6.05
4.10
0.99
0.36
3.00
f1 (ppm)