Supporting Information for

Modular reductive radical-polar crossover-based acyl migration reactions of *N*-vinylimides with alkyl, silyl, and acyl radicals

Yutao Jing, Li Zhang, Li Qiu and Yewen Fang

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1. General information

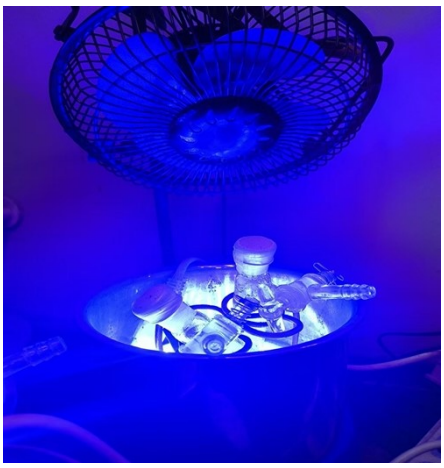
1.1 Solvents, Reagents, and Starting Materials

All reactions were carried out in glassware under inert (nitrogen) atmosphere unless otherwise noted. DMF and CH_2Cl_2 were dried from CaH. The dehydrated solvents DMSO, DMA, and acetonitrile were purchased from Energy Chemical Chemicals. The enamides were prepared according to our previous works[1] and literature procedures.[2] All dihydroquinazolinones (**2**,[3a-b] **4**,[3c] and **10a**[3d]) and benzothiazolines **10c-10d**,[4] and acyl oxime esters **13**[5-6] were well prepared according to the known procedures. Photocatalysts and all other chemicals were purchased from local vendors and used as supplied unless otherwise stated.

1.2 Instruments

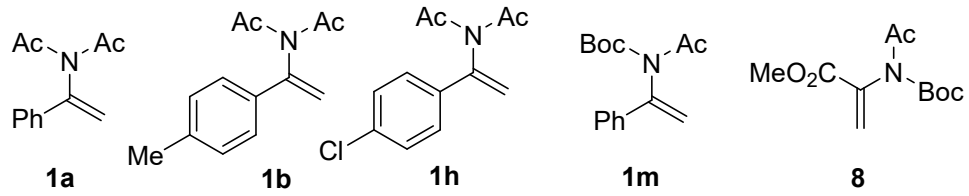
NMR spectra were recorded on a Bruker Avance 500 spectrometer (500 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent ($CHCl_3$ at 7.26 ppm ^{1}H NMR, 77.0 ppm ^{13}C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q =quartet, m = multiplet), coupling constants (Hz) and integration. High resolution mass spectra (HRMS) were recorded on an ESI-Q-TOF spectrometer Agilent 6210 ESI/TOF. Single Crystal X-ray Diffraction (SC-XRD) recorded on a Bruker D8 Quest. TLC analyses were performed on precoated GF254 silica gel plates and were visualized under UV254 nm light or by I_2 staining. Column chromatography was carried out using 300-400 mesh silica gel and eluted with petroleum/ethyl acetate unless otherwise noted.

1.3 Picture of a typical reaction setup

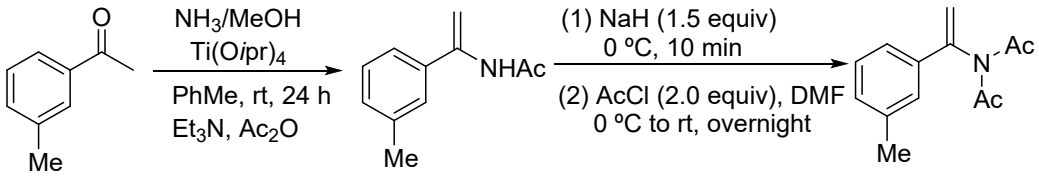


2. Preparation of *N*-vinylimides

2.1 Know substrates reported in our previous work[1]



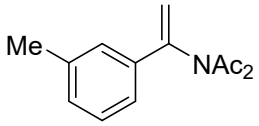
2.2 Synthesis of *N*-vinylimides 1c[1,2]



(a) To a 100 mL oven-dried single neck round flask equipped with a magnetic stirrer was added 3-methylacetophenone (1.35 g, 10.0 mmol, 1.0 equiv) and toluene (6 mL). The resultant solution was stirred and cooled in an ice/water bath. To the resultant cold stirring solution was added 7 N NH_3 in MeOH (2.14 mL, 15.0 mmol, 1.5 equiv) followed by dropwise addition of Ti(O*i*-Pr)$_4$ (5.92 mL, 20.0 mmol, 2.0 equiv). After 10 min, the ice/water cooling bath was removed, and the solution was stirred at room temperature for 24 h. The reaction mixture was then cooled in an ice/water bath (~5 °C) and treated with Et_3N (5.58 mL, 40.0 mmol, 4.0 equiv) followed by Ac_2O (1.89 mL, 20.0 mmol, 2.0 equiv). The cooling bath was then removed and the solution was stirred at room temperature for 3 h. The reaction mixture was then treated with *N,N,N'N'*-tetrakis(2-hydroxyethyl) ethylenedianmine (75% W.t., 5.95 mL, 21.0 mmol, 2.1 equiv) at room temperature, and the solution was then heated at about 55 °C for 20 min. The reaction mixture was cooled to room temperature and diluted with NH_4OH (20 mL), water (20 mL), and EtOAc (20 mL). After separation of the organic phase, the resulting aqueous phase was extracted with additional EtOAc (3×10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and evaporated in vacuo. The desired product enamide was obtained after purification by flash chromatography on silica gel with hexane/ethyl acetate as the eluent.

(b) The above prepared enamide (5 mmol) was dissolved in dry DMF (10 mL) in a dry round-bottom flask. The solution was cooled to 0°Cand sodium hydride (60% dispersion in mineral oil) (300 mg, 7.5 mmol) was added in portions. The resulting suspension was stirred at the same temperature for 10 min. Then AcCl (0.72 mL, 10 mmol) was added dropwise and the final solution was continued to

stir for overnight at room temperature. The completion of the reaction was confirmed by checking TLC and the excess of sodium hydride was quenched by adding water (10 mL) at 0 ℃. The resulting solution was extracted with ethyl acetate (5×10 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and evaporated under reduced pressure to give the crude product, which was purified by column chromatography over silica gel to give the pure product **1c**.

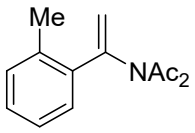


***N*-Acetyl-*N*-(1-(m-tolyl)vinyl)acetamide (1c).** Flash column chromatography to afford product as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26 – 7.12 (m, 4H), 5.99 (s, 1H), 5.27 (s, 1H), 2.39 (s, 6H), 2.39 (s, 6H), 2.34 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ172.7, 144.5, 138.5, 135.0, 129.8, 128.7, 125.4, 122.0, 115.4, 26.1, 21.3.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{15}NO_2Na$: 240.1000, found 240.1003.

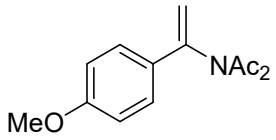


***N*-Acetyl-*N*-(1-(o-tolyl)vinyl)acetamide (1d).** Flash column chromatography to afford product as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 2H), 7.20 – 7.16 (m, 1H), 7.15 – 7.12 (m, 1H), 5.59 (s, 1H), 5.52 (s, 1H), 2.52 (s, 3H), 2.40 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.1, 142.7, 136.2, 135.8, 131.7, 128.5, 126.7, 126.1, 119.9, 26.3, 21.1.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{15}NO_2Na$: 240.1000, found 240.1003.

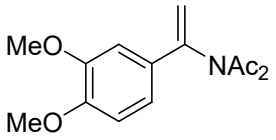


***N*-Acetyl-*N*-(1-(4-methoxyphenyl)vinyl)acetamide (1e).** Flash column chromatography to afford product as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, J = 8.9 Hz, 2H), 6.87 (d, J = 8.9 Hz, 2H), 5.87 (s, 1H), 5.18 (s, 1H), 3.79 (s, 3H), 2.39 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.8, 160.2, 144.0, 127.7, 126.3, 114.3, 113.5, 55.3, 26.1.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{15}NO_3Na$: 256.0950, found 256.0953.

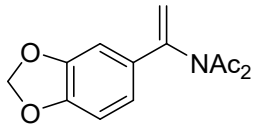


***N*-Acetyl-*N*-(1-(3,4-dimethoxyphenyl)vinyl)acetamide (1f).** Flash column chromatography to afford product as a yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.97 (d, *J* = 2.1 Hz, 1H), 6.89 – 6.85 (m, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 5.89 (s, 1H), 5.21 (s, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 2.40 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.9, 150.1, 149.3, 144.2, 128.1, 117.7, 113.8, 111.1, 108.1, 55.9, 26.2.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{17}NO_4Na$: 286.1055, found 286.1059.

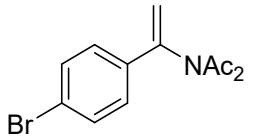


***N*-Acetyl-*N*-(1-(benzo[d][1,3]dioxol-5-yl)vinyl)acetamide (1g).** Flash column chromatography to afford product as a yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.91 (d, *J* = 1.8 Hz, 1H), 6.85 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.77 (d, *J* = 8.2 Hz, 1H), 5.98 (s, 2H), 5.85 (s, 1H), 5.20 (s, 1H), 2.40 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.8, 148.5, 148.4, 144.1, 129.6, 119.1, 114.2, 108.5, 105.5, 101.5, 26.2.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{13}NO_4Na$: 270.0742, found 270.0746.

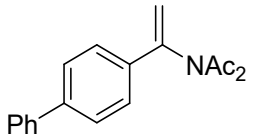


***N*-Acetyl-*N*-(1-(4-bromophenyl)vinyl)acetamide (1i).** Flash column chromatography to afford product as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 – 7.47 (m, 2H), 7.28 – 7.25 (m, 2H), 6.01 (d, *J*=1.1 Hz, 1H), 5.33 (d, *J* = 1.1 Hz, 1H), 2.39 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.6, 143.7, 134.2, 132.1, 126.5, 123.3, 116.3, 26.2.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{12}H_{12}NO_2NaBr$: 303.9949, found 303.9953.

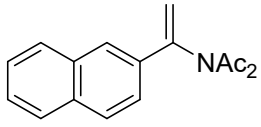


***N*-(1-([1,1'-Biphenyl]-4-yl)vinyl)-*N*-acetylacetamide (1j).** Flash column chromatography to afford product as a yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (t, *J* = 9.0 Hz, 4H), 7.50 – 7.42 (m, 4H), 7.37 (t, *J* = 7.4 Hz, 1H), 6.07 (s, 1H), 5.34 (s, 1H), 2.45 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.8, 144.3, 142.0, 140.1, 134.0, 128.9, 127.7, 127.0, 125.4, 115.6, 26.3.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{18}H_{17}NO_2Na$: 302.1157, found 302.1160.

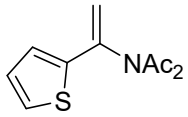


***N*-Acetyl-*N*-(1-(naphthalen-2-yl)vinyl)acetamide (1k).** Flash column chromatography to afford product as a yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.50 (d, *J* = 8.0 Hz, 1H), 7.88 (d, *J* = 9.5 Hz, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.59 – 7.55 (m, 1H), 7.54 – 7.49 (m, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 7.36 (dd, *J* = 7.3, 1.2 Hz, 1H), 5.87 (s, 1H), 5.74 (s, 1H), 2.46 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 173.3, 141.4, 134.3, 134.2, 130.9, 129.4, 128.7, 126.9, 126.0, 124.9, 124.8, 124.2, 121.1, 26.4.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{15}NO_2Na$: 276.1000, found 276.1001.

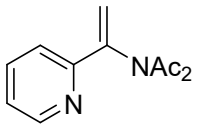


***N*-Acetyl-*N*-(1-(thiophen-2-yl)vinyl)acetamide (1l).** Flash column chromatography to afford product as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 (d, *J* = 4.7 Hz, 1H), 7.00 – 6.93 (m, 2H), 5.86 (s, 1H), 5.18 (s, 1H), 2.42 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 140.3, 139.2, 127.8, 126.3, 125.0, 114.6, 26.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{10}H_{11}NO_2NaS$: 232.0408, found 232.0412.



***N*-Acetyl-*N*-(1-(pyridin-3-yl)vinyl)acetamide (6).** Flash column chromatography to afford product as a brown oil.

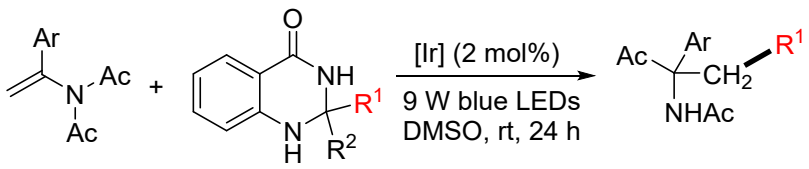
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.56 – 8.52 (m, 1H), 7.70 – 7.64 (m, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.22 – 7.18 (m, 1H), 6.38 (s, 1H), 5.51 (s, 1H), 2.38 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.9, 152.7, 149.5, 144.5, 136.8, 123.3, 119.7, 118.5, 26.1.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{11}H_{12}N_2O_2Na$: 227.0796, found 227.0800.

3. General procedure of photoredox-catalyzed acyl migration reactions.

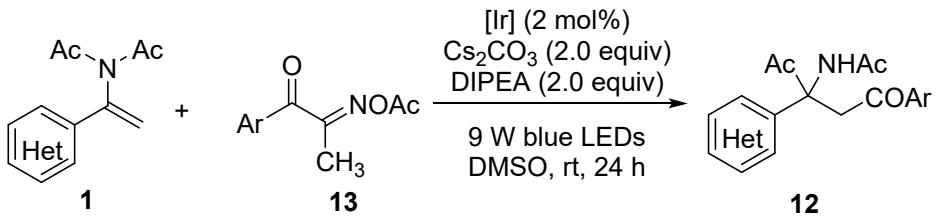
3.1 General procedure of photoredox-catalyzed reactions of enamides with dihydroquinazolinones or benzothiazolines



1 **2**, **10** **3**, **11**, **12**

To a 10 mL oven-dried Schlenk tube equipped with a magnetic stirrer was added Ir[dF(CF_3)ppy)]$_2$(dtbbpy)PF_6 (4.5 mg, 0.004 mmol, 0.02 equiv), *N*-vinylimide **1** (0.2 mmol, 1.0 equiv), and dihydroquinazolinone **2** or **10** (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, DMSO (6.0 mL, 0.033 M) was added by syringe under nitrogen atmosphere. The reaction mixture was then irradiated with 9 W blue LEDs at room temperature for 24 h. The reaction mixture was quenched with brine (10 mL) and extracted with EtOAc (4×5 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel to afford the acyl migrated product. (For some reactions listed in Table 4, 2.0 equivalents of base were necessary.)

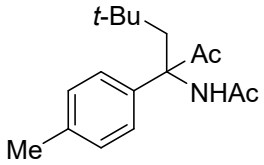
3.2 General procedure of photoredox-catalyzed acyl migration reactions between *N*-vinylimides with acyl oxime esters



1 **13** **12**

To a 10 mL oven-dried Schlenk tube equipped with a magnetic stirrer was added Ir[dF(CF_3)ppy)]$_2$(dtbbpy)PF_6 (4.5 mg, 0.004 mmol), *N*-vinylimide **1** (0.2 mmol, 1.0 equiv), acyl oxime ester **13** (0.4 mmol, 2.0 equiv), and Cs_2CO_3 (130 mg, 0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, DMSO (6.0 mL) and DIPEA (70 μL, 0.4 mmol, 2.0 equiv) were added by syringe under nitrogen atmosphere. The reaction mixture was then irradiated with 9 W blue LEDs at room temperature for 24 h. The reaction mixture was quenched

with brine (10 mL) and extracted with EtOAc (4×5 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel to afford compound the product **12**.

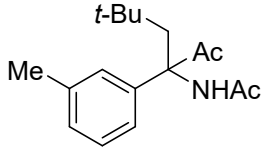


***N*-(5,5-Dimethyl-2-oxo-3-(*p*-tolyl)hexan-3-yl)acetamide (3b).** White solid (28.7 mg, 52% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.23 – 7.19 (m, 2H), 7.14 – 7.10 (m, 2H), 3.09 (d, *J* = 14.6 Hz, 1H), 2.37 (d, *J* = 14.7 Hz, 1H), 2.30 (s, 3H), 1.96 (s, 3H), 1.92 (s, 3H), 0.97 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 207.0, 168.4, 138.0, 137.4, 129.6, 125.6, 68.9, 42.2, 31.4, 30.6, 24.4, 24.2, 21.0.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{25}NO_2Na$: 298.1783, found 298.1787.

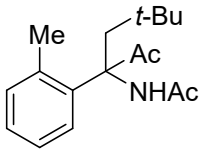


***N*-(5,5-Dimethyl-2-oxo-3-(*m*-tolyl)hexan-3-yl)acetamide (3c)**. White solid (36.4 mg, 66% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.23 – 7.18 (m, 1H), 7.16 – 7.09 (m, 2H), 7.07 – 7.01 (m, 1H), 3.10 (d, *J* = 14.7 Hz, 1H), 2.38 (d, *J* = 14.7 Hz, 1H), 2.32 (s, 3H), 1.96 (s, 3H), 1.91 (s, 3H), 0.97 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.9, 168.4, 140.9, 138.3, 128.6, 128.5, 126.3, 122.8, 69.0, 42.1, 31.4, 30.6, 24.3, 24.2, 21.6.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{25}NO_2Na$: 298.1783, found 298.1787.

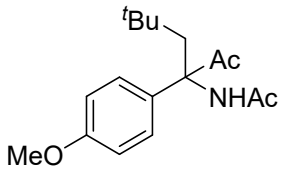


***N*-(5,5-Dimethyl-2-oxo-3-(*o*-tolyl)hexan-3-yl)acetamide (3d).** White solid (17.1 mg, 31% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ7.76 – 7.70 (m, 1H), 7.29 – 7.21 (m, 2H), 7.19 – 7.16 (m, 1H), 7.07 – 7.02 (m, 1H), 3.21 (d, *J* = 14.2 Hz, 1H), 2.31 (d, *J* = 14.3 Hz, 1H), 2.08 (s, 3H), 1.95 (s, 3H), 1.92 (s, 3H), 0.99 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 207.5, 167.6, 138.3, 135.4, 132.8, 127.9, 127.2, 126.3, 69.1, 43.8, 31.6, 30.7, 25.2, 23.7, 20.6.

ERMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{25}NO_2Na$: 298.1783, found 298.1787.

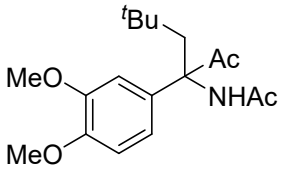


***N*-(3-(4-Methoxyphenyl)-5,5-dimethyl-2-oxohexan-3-yl)acetamide (3e).** Yellow oil (46.1 mg, 79% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.26 – 7.22 (m, 2H), 6.87 – 6.82 (m, 2H), 3.78 (s, 3H), 3.10 (d, *J* = 14.6 Hz, 1H), 2.35 (d, *J* = 14.7 Hz, 1H), 1.96 (s, 3H), 1.92 (s, 3H), 0.97 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 207.0, 168.4, 158.9, 133.0, 127.0, 114.2, 68.6, 55.2, 42.3, 31.4, 30.6, 24.3, 24.2.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{25}NO_3Na$: 314.1732, found 314.1737.

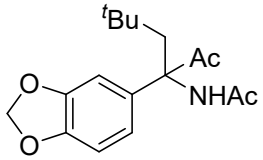


***N*-(3-(3,4-Dimethoxyphenyl)-5,5-dimethyl-2-oxohexan-3-yl)acetamide (3f).** Yellow solid (41.2 mg, 64% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 (s, 1H), 6.96 – 6.91 (m, 1H), 6.82 – 6.78 (m, 1H), 6.73 – 6.69 (m, 1H), 3.83 (s, 3H), 3.82 (s, 3H), 3.08 (d, *J* = 14.6 Hz, 1H), 2.33 (d, *J* = 14.6 Hz, 1H), 1.96 (s, 3H), 1.92 (s, 3H), 0.96 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.9, 168.4, 149.1, 148.6, 133.5, 118.5, 111.1, 109.0, 68.7, 55.9, 55.7, 42.2, 31.4, 30.6, 24.2(2), 24.2(4).

HRMS (ESI) $[M+Na]^+$: calculated for $C_{18}H_{27}NO_4Na$: 344.1838, found 344.1842.

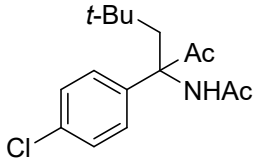


***N*-(3-(benzo[d][1,3]dioxol-5-yl)-5,5-dimethyl-2-oxohexan-3-yl)acetamide (3g)**. White solid (45.8 mg,75% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (s, 1H), 6.83 (d, *J* = 9.9 Hz, 1H), 6.79 – 6.70 (m, 2H), 5.95 – 5.92 (m, 2H), 3.05 (d, *J* = 14.5 Hz, 1H), 2.30 (d, *J* = 14.6 Hz, 1H), 1.96 (s, 3H), 1.94 (s, 3H), 0.96 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.7, 168.5, 148.2, 147.1, 135.1, 119.4, 108.4, 106.4, 101.3, 68.7, 42.4, 31.4, 30.6, 24.2.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{23}NO_4Na$: 328.1525, found 328.1528.

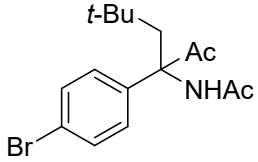


***N*-(3-(4-Chlorophenyl)-5,5-dimethyl-2-oxohexan-3-yl)acetamide (3h).** Yellow solid (49.1 mg, 83% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.31 – 7.26 (m, 4H), 3.08 (d, *J* = 14.6 Hz, 1H), 2.34 (d, *J* = 14.5 Hz, 1H), 1.96 (s, 3H), 1.93 (s, 3H), 0.97 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.1, 168.2, 139.5, 133.4, 128.7, 127.0, 68.5, 42.0, 31.2, 30.3, 24.2, 23.9.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{22}NO_2NaCl$: 318.1243, found 318.1243.

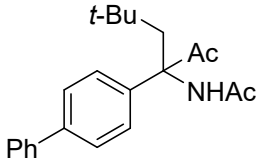


***N*-(3-(4-Bromophenyl)-5,5-dimethyl-2-oxohexan-3-yl)acetamide (3i).** Yellow solid (30.0 mg, 44% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 – 7.44 (m, 3H), 7.22 – 7.20 (m, 2H), 3.08 (d, *J* = 14.6 Hz, 1H), 2.34 (d, *J* = 14.6 Hz, 1H), 1.96 (s, 3H), 1.93 (s, 3H), 0.97 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.1, 168.2, 140.1, 131.7, 127.3, 121.7, 68.5, 41.9, 31.2, 30.3, 24.2, 23.9.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{22}NO_2NaBr$: 362.0732, found 362.0740.

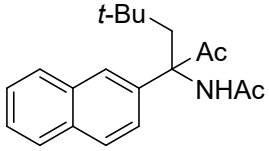


***N*-(3-([1,1'-Biphenyl]-4-yl)-5,5-dimethyl-2-oxohexan-3-yl)acetamide (3j).** White solid (44.6 mg, 66% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 – 7.54 (m, 4H), 7.51 (s, 1H), 7.43-7.40 (m, 4H), 7.35-7.32 (m, 1H), 3.17 (d, *J* = 14.6 Hz, 1H), 2.44 (d, *J* = 14.6 Hz, 1H), 2.00 (s, 3H), 1.98 (s, 3H), 1.00 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.7, 168.5, 140.4, 140.3, 140.0, 128.7, 127.5, 127.4, 127.0, 126.2, 69.0, 42.3, 31.4, 30.6, 24.5, 24.2.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{22}H_{27}NO_2Na$: 360.1939, found 360.1944.

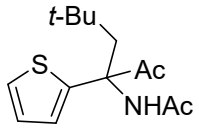


***N*-(5,5-Dimethyl-3-(naphthalen-2-yl)-2-oxohexan-3-yl)acetamide (3k).** Yellow solid (27.4 mg, 44% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.97 (m, 1H), 7.85 – 7.77 (m, 3H), 7.55 – 7.48 (m, 2H), 7.42 – 7.34 (m, 2H), 3.40 (d, *J* = 14.1 Hz, 1H), 2.42 (d, *J* = 14.1 Hz, 1H), 1.87 – 1.86 (m, 6H), 1.06 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 209.5, 167.9, 135.8, 134.7, 130.4, 129.7, 129.5, 126.2, 126.1, 125.2, 125.1, 123.4, 69.4, 44.3, 31.8, 30.8, 25.5, 23.9.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{20}H_{25}NO_2Na$: 334.1783, found 334.1787.

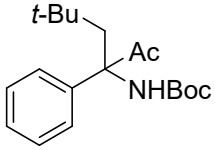


***N*-(5,5-Dimethyl-2-oxo-3-(thiophen-2-yl)hexan-3-yl)acetamide (3l).** White solid (45.5 mg, 85% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (s, 1H), 7.24 – 7.21 (m, 1H), 7.00 – 6.97 (m, 1H), 6.96 – 6.93 (m, 1H), 3.18 (d, *J* = 14.7 Hz, 1H), 2.39 (d, *J* = 14.7 Hz, 1H), 2.05 (s, 3H), 1.99 (s, 3H), 0.96 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 205.3, 168.8, 146.4, 127.2, 125.3, 124.9, 67.7, 44.1, 31.6, 30.4, 24.2, 23.9.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{21}NO_2NaS$: 290.1191, found 290.1196.

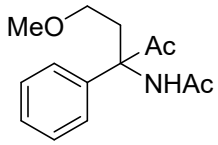


***tert*-Butyl(5,5-dimethyl-2-oxo-3-phenylhexan-3-yl)carbamate (3m).** White solid (47.3 mg, 74% yield).

^{1}H NMR (500 MHz, $CDCl_3$, observed as a mixture of rotamers**) δ** 7.39 – 7.28 (m, 4H), 7.28 – 7.20 (m, 1H), [6.50 (br, 0.78H), 6.46 (br, 0.22H)], [2.90 (d, *J* = 14.8 Hz, 0.78H) 2.68 (d, *J* = 14.6 Hz, 0.22H)], 2.45 – 2.37 (m, 1H), [1.92 (s, 1H) 1.91 (s, 2H)], [1.34 (s, 7H), 1.07 (s, 2H)], [0.98 (s, 2.6H), 0.96 (s, 6.4H)].

^{13}C NMR (126 MHz, $CDCl_3$, observed as a mixture of rotamers**) δ** 206.3, 153.4, 128.7, 128.6, 127.5, 125.9, 125.8, 79.1, 68.7, 44.1, 43.0, 31.3, 30.8, 30.6, 28.4, 27.9, 24.2.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{19}H_{29}NO_3Na$: 342.2045, found 342.2049.

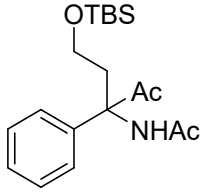


***N*-(1-Methoxy-4-oxo-3-phenylpentan-3-yl)acetamide (3n).** White solid (40.9 mg, 82% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.36 – 7.30 (m, 4H), 7.28 – 7.25 (m, 1H), 3.56 – 3.49 (m, 1H), 3.33 – 3.27 (m, 1H), 3.21 (s, 3H), 3.19 – 3.15 (m, 1H), 2.79 – 2.72 (m, 1H), 2.01 (s, 3H), 1.93 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 204.7, 168.5, 139.4, 128.7(7), 128.7(5), 127.8, 126.0(8), 126.0(6), 68.0, 58.8, 31.7, 23.8.

The analytical data were in good agreement with our previous report.[1b]

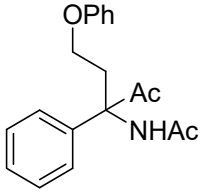


***N*-(1-((*tert*-Butyldimethylsilyl)oxy)-4-oxo-3-phenylpentan-3-yl)acetamide (3o).** Colorless oil (60.1 mg, 86% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.36 – 7.32 (m, 4H), 7.28 – 7.23 (m, 1H), 3.79 – 3.74 (m, 1H), 3.65 – 3.60 (m, 1H), 3.15 – 3.11 (m, 1H), 2.83 – 2.77 (m, 1H), 2.02 (s, 3H), 1.98 (s, 3H), 0.87 (s, 9H), 0.03 (s, 3H), 0.02 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 204.5, 168.4, 139.6, 128.8, 127.8, 126.1, 67.9, 58.9, 34.1, 25.9, 23.9, 23.8, 18.3, -5.9(0), -5.9(3).

HRMS (ESI) $[M+Na]^+$: calculated for $C_{19}H_{31}NO_3NaSi$: 372.1971, found 372.1979.

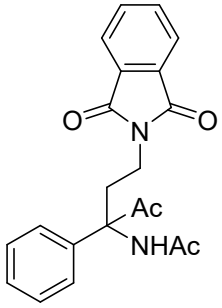


***N*-(4-Oxo-1-phenoxy-3-phenylpentan-3-yl)acetamide (3p).** White solid (41.1 mg, 66% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.42 – 7.35 (m, 4H), 7.34 – 7.24 (m, 3H), 7.00 – 6.92 (m, 1H), 6.84 – 6.77 (m, 2H), 4.19 – 4.13 (m, 1H), 3.97 – 3.90 (m, 1H), 3.49 – 3.42 (m, 1H), 3.09 – 3.01 (m, 1H), 2.08 (s, 3H), 1.99 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 204.2, 168.7, 158.2, 139.2, 129.5, 128.9, 128.0, 126.0, 121.1, 114.0, 67.9, 63.2, 31.1, 23.8(4), 23.8(1).

HRMS (ESI) [$M+H]^+$: calculated for $C_{19}H_{21}NO_3$: 334.1419, found 334.1413.

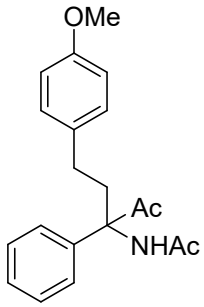


***N*-(1-(1,3-Dioxoisoindolin-2-yl)-4-oxo-3-phenylpentan-3-yl)acetamide (3q).** Colorless oil (51.7 mg, 71% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 – 7.65 (m, 2H), 7.66 – 7.61 (m, 2H), 7.32 – 7.26 (m, 3H), 7.18 – 7.11 (m, 2H), 7.02 – 6.96 (m, 1H), 3.76 – 3.66 (m, 1H), 3.64 – 3.55 (m, 1H), 3.27 – 3.18 (m, 1H), 2.81 – 2.72 (m, 1H), 2.16 (s, 3H), 1.87 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 203.8, 169.7, 168.3, 137.4, 133.9, 131.6, 128.7, 127.6, 125.5, 123.0, 68.4, 33.5, 29.8, 23.4, 23.1.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{21}H_{20}N_2O_4Na$: 387.1321, found 387.1318.

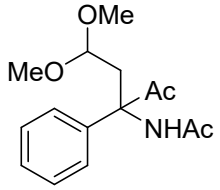


***N*-(1-(4-Methoxyphenyl)-4-oxo-3-phenylpentan-3-yl)acetamide (3r).** White solid (39.0 mg, 60% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 (d, *J* = 4.3 Hz, 4H), 7.33 (s, 1H), 7.31 – 7.27 (m, 1H), 7.10 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 3.79 (s, 3H), 3.43-3.37 (m, 1H), 2.63 – 2.52 (m, 2H), 2.23-2.18 (m, 1H), 1.99 (s, 3H), 1.93 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 205.3, 168.3, 158.0, 139.2, 132.9, 129.3, 128.9, 128.0, 126.1, 113.9, 69.8, 55.3, 33.0, 29.5, 23.8, 23.4.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{20}H_{23}NO_3Na$: 348.1576, found 348.1580.

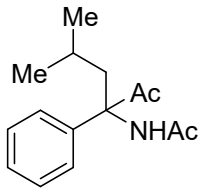


***N*-(1,1-Dimethoxy-4-oxo-3-phenylpentan-3-yl)acetamide (3s).** White solid (22.4 mg, 40% yeild).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (s, 1H), 7.37 – 7.28 (m, 4H), 7.27 (d, *J* = 10.7 Hz, 1H), 4.27 (dd, *J* = 9.0, 2.0 Hz, 1H), 3.41 (dd, *J* = 14.0, 2.4 Hz, 1H), 3.38 (s, 3H), 3.30 (s, 3H), 2.71 (dd, *J* = 14.0, 8.8 Hz, 1H), 2.02 (s, 3H), 1.93 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 204.3, 168.5, 139.1, 128.9, 128.0, 126.0, 103.7, 67.3, 56.3, 53.8, 35.8, 23.9, 23.8.

HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{21}NO_3$: 302.1368, found 302.1373.

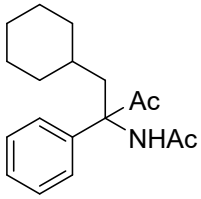


***N*-(5-Methyl-2-oxo-3-phenylhexan-3-yl)acetamide (3t).** Yellow solid (25.7 mg, 52% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.37 – 7.32 (m, 4H), 7.29 – 7.26 (m, 1H), 3.06 (dd, *J* = 14.2, 5.6 Hz, 1H), 2.24 (dd, *J* = 14.2, 6.9 Hz, 1H), 2.00 (s, 3H), 1.94 (s, 3H), 1.62 – 1.55 (m, 1H), 0.99 (d, *J* = 6.7 Hz, 3H), 0.89 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.1, 168.3, 140.1, 128.8, 127.8, 126.0, 69.6, 39.3, 24.6, 24.0, 23.9, 23.8, 23.3.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{21}NO_2Na$: 270.1470, found 270.1472.

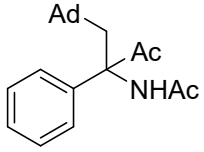


***N*-(1-Cyclohexyl-3-oxo-2-phenylbutan-2-yl)acetamide (3u).** Yellow solid (29.9 mg, 52% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.36 – 7.31 (m, 4H), 7.28 – 7.23 (m, 1H), 3.03 (dd, *J* = 14.3, 5.2 Hz, 1H), 2.21 (dd, *J* = 14.3, 6.5 Hz, 1H), 1.98 (s, 3H), 1.92 (s, 3H), 1.74 – 1.50 (m, 5H), 1.30 – 0.94 (m, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.2, 168.3, 140.2, 128.8, 127.8, 126.1, 69.4, 38.2, 34.4, 34.0, 33.9, 26.3, 26.3, 26.1, 24.0, 23.9.

The analytical data were in good agreement with our previous report.[1b]

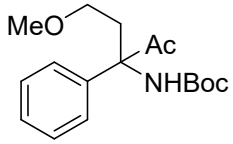


***N*-(1-((3*r*,5*r*,7*r*)-Adamantan-1-yl)-3-oxo-2-phenylbutan-2-yl)acetamide (3v).** White solid (47.5 mg, 70% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.32 (d, *J* = 6.5 Hz, 4H), 7.23 (s, 1H), 3.03 (d, *J* = 14.7 Hz, 1H), 2.21 (d, *J* = 14.7 Hz, 1H), 1.96 (s, 3H), 1.93 – 1.87 (m, 5H), 1.71 – 1.53 (m, 13H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 206.9, 168.3, 141.2, 128.7, 127.6, 125.7, 68.5, 43.6, 42.9, 36.7, 33.7, 28.6, 24.5, 24.2.

HRMS (ESI) $[M+H]^+$: calculated for $C_{22}H_{30}NO_2$: 340.2277, found 340.2273.

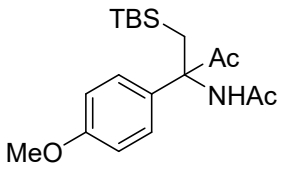


***tert*-Butyl (1-methoxy-4-oxo-3-phenylpentan-3-yl)carbamate (3w).** White solid (48.6 mg, 79% yield).

^{1}H NMR (500 MHz, $CDCl_3$, observed as a mixture of rotamers**) δ** 7.38 – 7.30 (m, 4H), 7.30 – 7.24 (m, 1H), [6.61 (br, 0.75H), 6.50(br, 0.25H)], 3.57 – 3.51 (m, 1H), 3.41 – 3.33 (m, 1H), 3.22 (s, 3H), [3.02 – 2.95 (m, 0.75H), 2.79 – 2.73 (m, 1.25H)], 1.93 (s, 3H), [1.35 (s, 6.75H), 1.06 (s, 2.25H)].

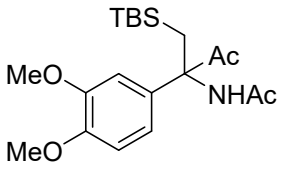
^{13}C NMR (126 MHz, $CDCl_3$, observed as a mixture of rotamers**) δ** 204.3, 153.7, 140.0, 128.8, 127.7, 126.1, 79.2, 68.0, 67.6, 58.7, 32.3, 28.3, 27.8, 23.9.

The analytical data were in good agreement with our previous report.[1b]

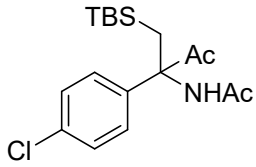


***N*-(1-(*tert*-Butyldimethylsilyl)-2-(4-methoxyphenyl)-3-oxobutan-2-yl)acetamide**

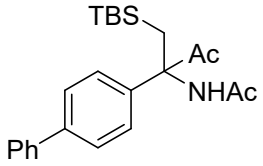
(11a). White solid (29.4 mg, 42% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (s, 1H), 7.33 – 7.28 (m, 2H), 6.89 – 6.82 (m, 2H), 2.60 (d, *J* = 14.7 Hz, 1H), 1.95 (s, 3H), 1.90 (s, 3H), 1.60 (d, *J* = 14.7 Hz, 1H), 0.90 (s, 9H), -0.04 (s, 3H), -0.10 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 206.1, 168.2, 159.0, 133.4, 127.2, 114.1, 67.8, 55.2, 26.2, 24.2, 23.5, 16.7, 15.4, -5.1, -5.6.
HRMS (ESI) $[M+Na]^+$: calculated for $C_{19}H_{31}NO_3NaSi$: 372.1971, found 372.1974.



***N*-(1-(*tert*-Butyldimethylsilyl)-2-(3,4-dimethoxyphenyl)-3-oxobutan-2-yl)acetamide (11b).** White solid (34.2 mg, 45% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (s, 1H), 7.03 (dd, *J* = 8.5, 2.3 Hz, 1H), 6.83 (d, *J* = 8.4 Hz, 1H), 6.76 (d, *J* = 2.3 Hz, 1H), 3.85 (s, 3H), 3.84 (s, 3H), 2.59 (d, *J* = 14.7 Hz, 1H), 1.96 (s, 3H), 1.91 (s, 3H), 1.59 (d, *J* = 14.7 Hz, 1H), 0.90 (s, 9H), -0.04 (s, 3H), -0.10 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 206.0, 168.2, 149.0, 148.6, 133.9, 118.7, 111.0, 109.2, 68.0, 55.9, 55.8, 26.2, 24.2, 23.4, 16.7, 15.4, -5.1, -5.6.
HRMS (ESI) $[M+Na]^+$: calculated for $C_{20}H_{33}NO_4NaSi$: 402.2077, found 402.2079.

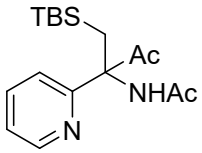


***N*-(1-(*tert*-Butyldimethylsilyl)-2-(4-chlorophenyl)-3-oxobutan-2-yl)acetamide (11c).** Yellow solid (28.4 mg, 40% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.36 – 7.28 (m, 4H), 2.59 (d, *J* = 14.6 Hz, 1H), 1.95 (s, 3H), 1.90 (s, 3H), 1.58 (d, *J* = 14.6 Hz, 1H), 0.90 (s, 9H), -0.03 (s, 3H), -0.10 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 205.5, 168.2, 140.2, 133.7, 128.8, 127.5, 67.9, 26.2, 24.1, 23.6, 16.7, 15.3, -5.1, -5.7.
HRMS (ESI) $[M+Na]^+$: calculated for $C_{18}H_{28}NO_2NaSiCl$: 376.1470, found 376. 1476.

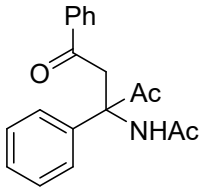


***N*-(2-([1,1'-Biphenyl]-4-yl)-1-(*tert*-butyldimethylsilyl)-3-oxobutan-2-yl)acetamide(11d).** Yellow solid (28.5 mg, 36% yield).

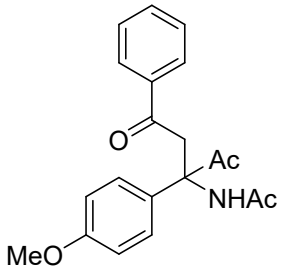
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.54 (m, 4H), 7.50 – 7.39 (m, 5H), 7.37 – 7.30 (m, 1H), 2.67 (d, *J* = 14.7 Hz, 1H), 1.99 (s, 3H), 1.96 (s, 3H), 1.68 (d, *J* = 14.6 Hz, 1H), 0.93 (s, 9H), -0.01 (s, 3H), -0.07 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 205.9, 168.3, 140.5, 129.2, 128.7, 127.4(3), 127.3(9), 127.1, 126.9, 126.4, 68.2, 26.3, 24.2, 23.7, 16.8, 15.4, -5.1, -5.6.
HRMS (ESI) $[M+Na]^+$: calculated for $C_{24}H_{33}NO_2NaSi$: 418.2178, found 418.2173.



***N*-(1-(*tert*-butyldimethylsilyl)-3-oxo-2-(pyridin-2-yl)butan-2-yl)acetamide (11e).** White solid (17.3 mg, 27% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.36 – 7.29 (m, 4H), 2.59 (d, *J* = 14.6 Hz, 1H), 1.95 (s, 3H), 1.90 (s, 3H), 1.58 (d, *J* = 14.6 Hz, 1H), 0.90 (s, 9H), -0.03 (s, 3H), -0.10 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 206.2, 169.0, 156.2, 148.0, 137.4, 123.1, 121.5, 69.6, 26.2, 23.7, 23.6, 18.0, 16.5, -5.0, -6.1.
HRMS (ESI) [$M+H]^+$: calculated for $C_{17}H_{29}N_2O_2Si$: 321.1998, found 321.2000.

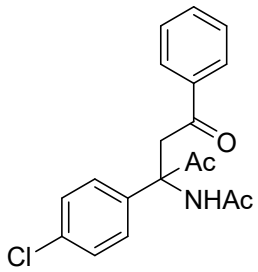


***N*-(1,4-Dioxo-1,3-diphenylpentan-3-yl)acetamide (12a).** Yellow solid (40.2 mg, 65% yeild).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 – 7.98 (m, 2H), 7.62 – 7.58 (m, 1H), 7.50 – 7.41 (m, 5H), 7.40 – 7.36 (m, 2H), 7.33 – 7.29 (m, 1H), 5.17 (d, *J* = 17.4 Hz, 1H), 3.87 (d, *J* = 17.5 Hz, 1H), 2.03 (s, 3H), 1.91 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 203.0, 198.0, 169.4, 137.7, 136.6, 133.8, 129.0, 128.7, 128.4, 128.2, 125.7, 67.5, 41.0, 23.7, 23.1.
HRMS (ESI) $[M+Na]^+$: calculated for $C_{19}H_{19}NO_3Na$: 332.1263, found 332.1268.

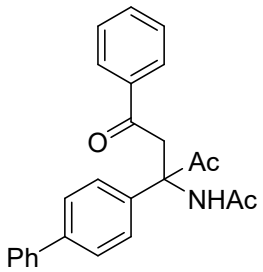


***N*-(3-(4-Methoxyphenyl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (12b).** Yellow oil (30.6 mg, 45% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 – 7.98 (m, 2H), 7.61 – 7.57 (m, 1H), 7.49 – 7.45 (m, 3H), 7.35 – 7.31 (m, 2H), 6.92 – 6.87 (m, 2H), 5.16 (d, *J* = 17.4 Hz, 1H), 3.82 (d, *J* = 17.5 Hz, 1H), 3.79 (s, 3H), 2.02 (s, 3H), 1.90 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 203.1, 198.1, 169.5, 159.3, 136.6, 133.8, 129.6,

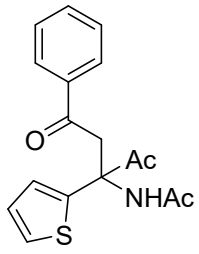
128.7, 128.4, 127.0, 114.4, 66.9, 55.2, 41.0, 23.7, 23.0.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{20}H_{21}NO_4Na$: 362.1368, found 362.1371.



***N*-(3-(4-Chlorophenyl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (12c).** Yellow oil (26.1 mg, 38% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 – 7.95 (m, 2H), 7.63 – 7.58 (m, 1H), 7.52 – 7.45 (m, 3H), 7.40 – 7.32 (m, 4H), 5.14 (d, *J* = 17.3 Hz, 1H), 3.82 (d, *J* = 17.4 Hz, 1H), 2.03 (s, 3H), 1.90 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 202.5, 197.7, 169.4, 136.4(1), 136.3(7), 134.3, 133.9, 129.2, 128.8, 128.4, 127.2, 67.1, 41.0, 23.6, 23.1.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{19}H_{18}NO_3NaCl$: 366.0873, found 366.0875.

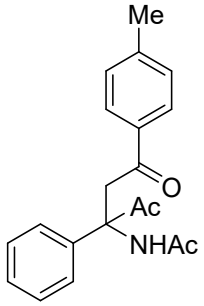


***N*-(3-([1,1'-Biphenyl]-4-yl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (12d).** Yellow oil (27.0 mg, 35% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.62 – 7.47 (m, 10H), 7.46 – 7.41 (m, 2H), 7.37 – 7.33 (m, 1H), 5.22 (d, *J* = 17.4 Hz, 1H), 3.91 (d, *J* = 17.4 Hz, 1H), 2.07 (s, 3H), 1.94 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 202.9, 198.0, 169.6, 141.0, 140.1, 136.6, 136.5, 133.8, 133.3, 128.7, 128.4, 127.7, 127.5, 127.0, 126.2, 67.4, 41.1, 23.6, 23.1.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{25}H_{23}NO_3Na$: 408.1576, found 408.1577.

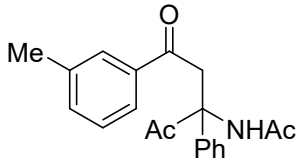


***N*-(1,4-Dioxo-1-phenyl-3-(thiophen-2-yl)pentan-3-yl)acetamide (12e).** Yellow oil (23.4 mg, 37% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 – 7.96 (m, 2H), 7.63 – 7.58 (m, 1H), 7.54 (s, 1H), 7.51 – 7.46 (m, 2H), 7.30 – 7.27 (m, 1H), 7.09 – 7.05 (m, 1H), 7.01 – 6.98 (m, 1H), 5.23 (d, *J* = 17.8 Hz, 1H), 3.88 (d, *J* = 17.8 Hz, 1H), 2.13 (s, 3H), 1.94 (s, 3H).

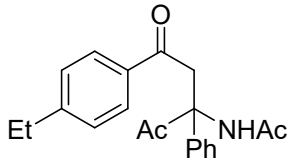
^{13}C NMR (126 MHz, $CDCl_3$) δ 201.5, 197.3, 169.7, 142.6, 136.2, 133.9, 128.8, 128.4, 127.4, 125.8, 125.3, 65.6, 42.6, 23.8, 22.6.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{17}H_{17}NO_3NaS$: 338.0827, found 338.0829.



***N*-(1,4-Dioxo-3-phenyl-1-(p-tolyl)pentan-3-yl)acetamide (12f).** Yellow solid (27.2 mg, 42% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 – 7.84 (m, 2H), 7.51 (s, 1H), 7.45 – 7.40 (m, 2H), 7.40 – 7.33 (m, 2H), 7.33 – 7.24 (m, 3H), 5.13 (d, *J* = 17.3 Hz, 1H), 3.83 (d, *J* = 17.3 Hz, 1H), 2.41 (s, 3H), 2.02 (s, 3H), 1.90 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 203.1, 197.6, 169.4, 144.8, 137.8, 134.1, 129.4, 129.0, 128.5, 128.1, 125.8, 67.5, 40.9, 23.7, 23.1, 21.7.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{20}H_{21}NO_3Na$: 346.1419, found 346.1423.

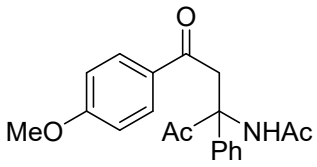


***N*-(1,4-Dioxo-3-phenyl-1-(*m*-tolyl)pentan-3-yl)acetamide (12g).** Colorless oil (20.1 mg, 31% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 – 7.77 (m, 2H), 7.50 (s, 1H), 7.45 – 7.34 (m, 6H), 7.34 – 7.27 (m, 1H), 5.14 (d, *J* = 17.5 Hz, 1H), 3.87 (d, *J* = 17.5 Hz, 1H), 2.41 (s, 3H), 2.01 (s, 3H), 1.91 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 203.0, 198.1, 169.4, 138.6, 137.8, 136.6, 134. 5, 129.0, 128.8, 128.6, 128.1, 125.7, 125.6, 77.3, 77.0, 76.7, 67.5, 41.1, 23.6, 23.1, 21.3.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{20}H_{21}NO_3Na$: 346.1419, found 346.1422.

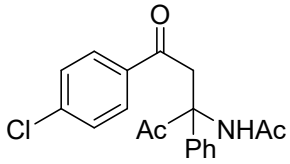


***N*-(1-(4-Ethylphenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (12h).** Yellow oil (18.2 mg, 27% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.49 (s, 1H), 7.45 – 7.41 (m, 2H), 7.40 – 7.35 (m, 2H), 7.33 – 7.28 (m, 3H), 5.14 (d, *J* = 17.4 Hz, 1H), 3.84 (d, *J* = 17.3 Hz, 1H), 2.71 (q, *J* = 7.6 Hz, 2H), 2.02 (s, 3H), 1.91 (s, 3H), 1.27 – 1.24 (m, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 203.1, 197.6, 169.3, 150.9, 137.9, 134.4, 129.0,

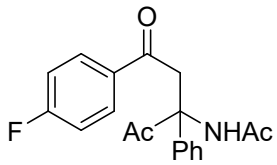
128.6, 128.3, 128.1, 125.8, 67.5, 40.9, 29.0, 23.7, 23.1, 15.1.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{21}H_{23}NO_3Na$: 360.1576, found 360.1577.



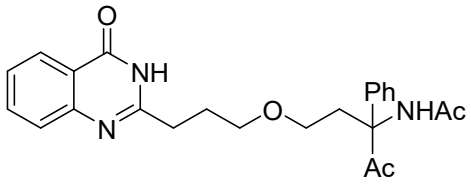
***N*-(1-(4-Methoxyphenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (12i).** Yellow oil (22.4 mg, 33% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.97 (m, 2H), 7.51 (s, 1H), 7.44 – 7.40 (m, 2H), 7.39 – 7.34 (m, 2H), 7.32 – 7.27 (m, 1H), 6.95 – 6.91 (m, 2H), 5.12 (d, *J* = 17.1 Hz, 1H), 3.87 (s, 3H), 3.79 (d, *J* = 17.0 Hz, 1H), 2.03 (s, 3H), 1.89 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 203.1, 196.4, 169.3, 164.1, 137.9, 130.8, 129.7, 129.0, 128.1, 125.8, 113.9, 67.6, 55.5, 40.6, 23.7, 23.2.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{20}H_{21}NO_4Na$: 362.1368, found 362.1371.



***N*-(1-(4-Chlorophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (12j).** Yellow oil (35.1 mg, 51% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 – 7.91 (m, 2H), 7.48 (s, 1H), 7.46 – 7.35 (m, 6H), 7.33 – 7.29 (m, 1H), 5.13 (d, *J* = 17.3 Hz, 1H), 3.82 (d, *J* = 17.3 Hz, 1H), 2.02 (s, 3H), 1.91 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 202.8, 197.0, 169.5, 140.4, 137.6, 134.9, 129.8, 129.1, 128.2, 125.7, 67.5, 40.9, 23.6, 23.1.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{19}H_{18}NO_3NaCl$: 366.0873, found 366.0875.



***N*-(1-(4-Fluorophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (12k).** Yellow oil (13.1 mg, 20% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 – 7.99 (m, 2H), 7.50 (s, 1H), 7.44 – 7.35 (m, 4H), 7.34 – 7.29 (m, 1H), 7.17 – 7.12 (m, 2H), 5.14 (d, *J* = 17.3 Hz, 1H), 3.83 (d, *J* = 17.3 Hz, 1H), 2.03 (s, 3H), 1.91 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 202.9, 196.5, 169.5, 166.2 (d, *J* = 256.3 Hz), 137.6, 133.0 (d, *J* = 3.0 Hz), 131.2 (d, *J* = 9.5 Hz), 129.1, 128.2, 125.7, 115.9 (d, *J* = 21.8 Hz), 67.5, 40.9, 23.7, 23.1.
^{19}F NMR (471 MHz, $CDCl_3$) δ -103.7.
HRMS (ESI) [M+Na]$^+$: calculated for $C_{19}H_{18}NO_3NaF$: 350.1168, found 350.1172.

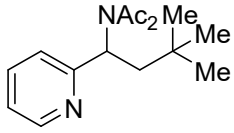


***N*-(4-Oxo-1-(3-(4-oxo-3,4-dihydroquinazolin-2-yl)propoxy)-3-phenylpentan-3-yl)acetamide (5).** White solid（45.6 mg, 54% yield）.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 – 8.25 (m, 1H), 7.90 (s, 1H), 7.79 – 7.72 (m, 1H), 7.70 – 7.64 (m, 1H), 7.49 – 7.45 (m, 1H), 7.43 – 7.38 (m, 2H), 7.38 – 7.32 (m, 2H), 7.31 – 7.26 (m, 1H), 3.61 (d, *J* = 9.9 Hz, 1H), 3.50 – 3.42 (m, 2H), 3.31 – 3.21 (m, 2H), 2.90 – 2.82 (m, 1H), 2.78 – 2.70 (m, 1H), 2.66 – 2.59 (m, 1H), 2.09 (s, 3H), 2.03 (s, 3H), 2.02 – 1.96 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 207.0, 169.4, 162.7, 156.0, 149.3, 139.3, 134.6, 128.9, 128.0, 127.1, 126.4(1), 126.3(6), 126.1, 120.8, 69.4, 68.4, 66.7, 32.3, 32.0, 27.3, 24.3, 23.7.

HRMS (ESI) $[M+Na]^+$: calculated for $C_{24}H_{27}N_3O_4Na$: 444.1899, found 444.1901.

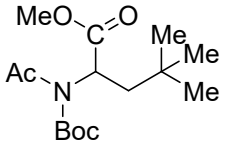


***N*-(5,5-Dimethyl-2-oxo-3-(pyridin-2-yl)hexan-3-yl)acetamide (7).** Yellow solid (21 mg, 40% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.55 – 8.47 (m, 1H), 7.68 – 7.59 (m, 1H), 7.45 – 7.36 (m, 1H), 7.17 – 7.11 (m, 1H), 5.74 – 5.66 (m, 1H), 2.71 (dd, *J* = 14.7, 5.3 Hz, 1H), 2.38 (s, 6H), 1.91 (dd, *J* = 14.7, 6.2 Hz, 1H), 0.96 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 174.6, 160.1, 148.6, 136.4, 122.1, 122.0, 56.9, 44.5, 30.9, 29.7, 27.1.

HRMS (ESI) $[M+H]^+$: calculated for $C_{15}H_{23}N_2O_2$: 263.1760, found 263.1768.



Methyl 2-(*N*-(*tert*-butoxycarbonyl)acetamido)-4,4-dimethylpentanoate (9). Yellow oil (47.1mg, 78% yield)

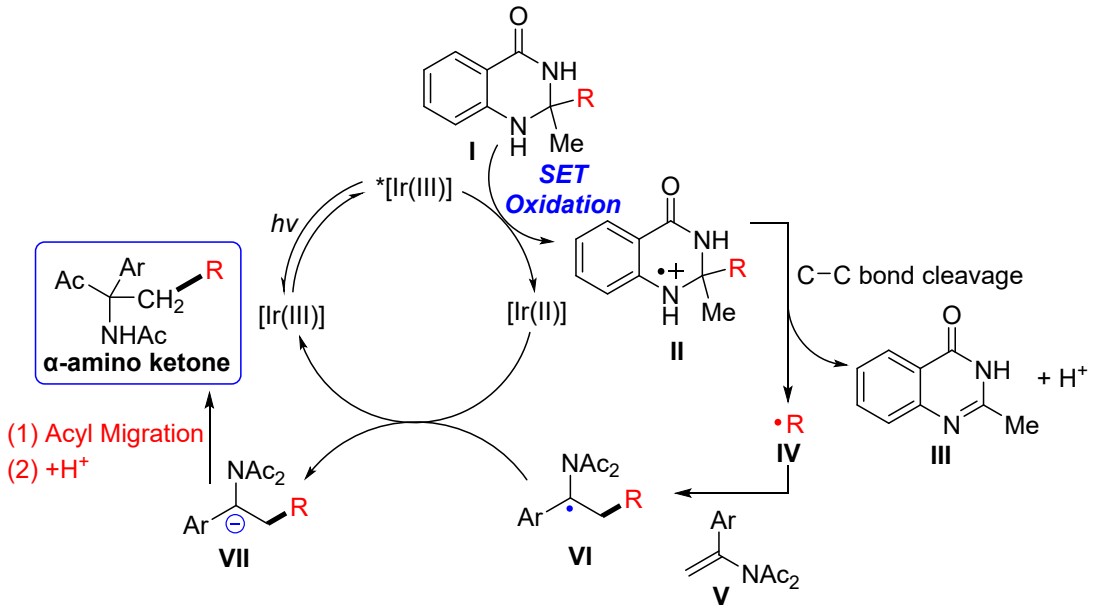
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.31 (dd, *J* = 7.4, 3.5 Hz, 1H), 3.65 (s, 3H), 2.47 (s, 3H), 2.26 (dd, *J* = 15.1, 3.5 Hz, 1H), 1.57 (dd, *J* = 15.1, 7.3 Hz, 1H), 1.47 (s, 9H), 0.88 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.6, 171.9, 152.2, 83.8, 52.9, 52.2, 43.7, 30.3, 29.3, 27.9, 26.7.

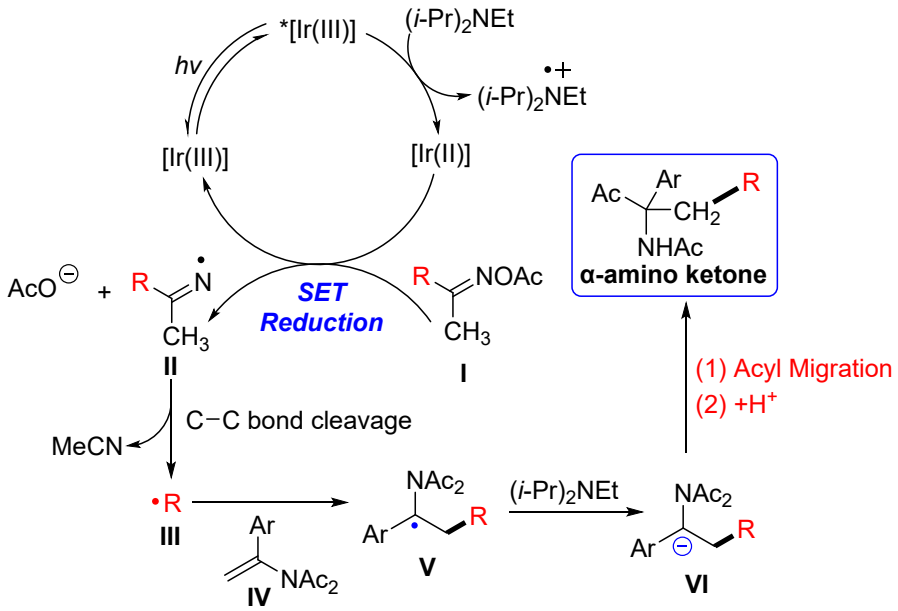
HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{27}NO_5Na$: 324.1787, found 324.1790.

4. Proposed mechanisms[1b,3-6]

Path I: ***SET oxidation-based generation of radical***



Path II: ***SET reduction-based generation of radical***



5. References

[1] (*a*) L. Huai, L. Zhang, Z. Wang, H. Wu and Y. Fang, *Org. Chem. Front.*, 2023, **10**, 1245; (*b*) L. Huai, L. Zhang, Z. Wang and Y. Fang, *Org. Chem. Front.*, 2024, **11**, 2344.

[2] J. T. Reeves, Z. Tan, Z. S. Han, G. Li, Y. Zhang, Y. Xu, D. C. Reeves, N. C. Gonnella, S. Ma, H. Lee, B. Z. Lu and C. H. Senanayake, *Angew. Chem. Int. Ed.*, 2012, **51**, 1400.

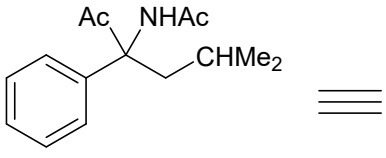
[3] (*a*) L. Li, L. Fang, W. Wu and J. Zhu, *Org. Lett.*, 2020, **22**, 5401; (*b*) Y. Zhang, T. Zhu, Y. Lin, X. Wei, X. Xie, R. Lin, Z. Zhang, W. Fang, J.-J. Zhang, Y. Zhang, M.-Y. Hu, L. Cai and Z. Chen, *Org. Biomol. Chem.*, 2024, **22**, 5561; (*c*) T. Uchikura, H. Nakamura, H. Sakai and T. Akiyama, *Chem. Eur. J.*, 2023, **29**, e202301090; (*d*) H.-J. Miao, J.-H. Zhang, W. Li, W. Yang, H. Xin, P. Gao, X.-H. Duan and L.-N. Guo, *Chem. Sci.*, 2024, **15**, 8993.

[4] L. Li, S. Guo, Q. Wang and J. Zhu, *Org. Lett.*, 2019, **21**, 5462.

[5] Y.-Y. Cheng, T. Lei, L. Su, X. Fan, B. Chen, C.-H. Tung, L.-Z. Wu, *Org. Lett.*, 2019, **21**, 8789.

[6] Y. Bao, Z.-J. Song, J.-L. Dai, S. Yan, Y. Zhang, J.-Y. Wang and G. Li, *Chin. J. Chem.*, 2024, **42**, 1399.

6. X-ray crystal data for compounds 3t



3t

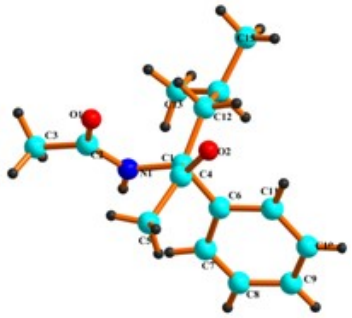
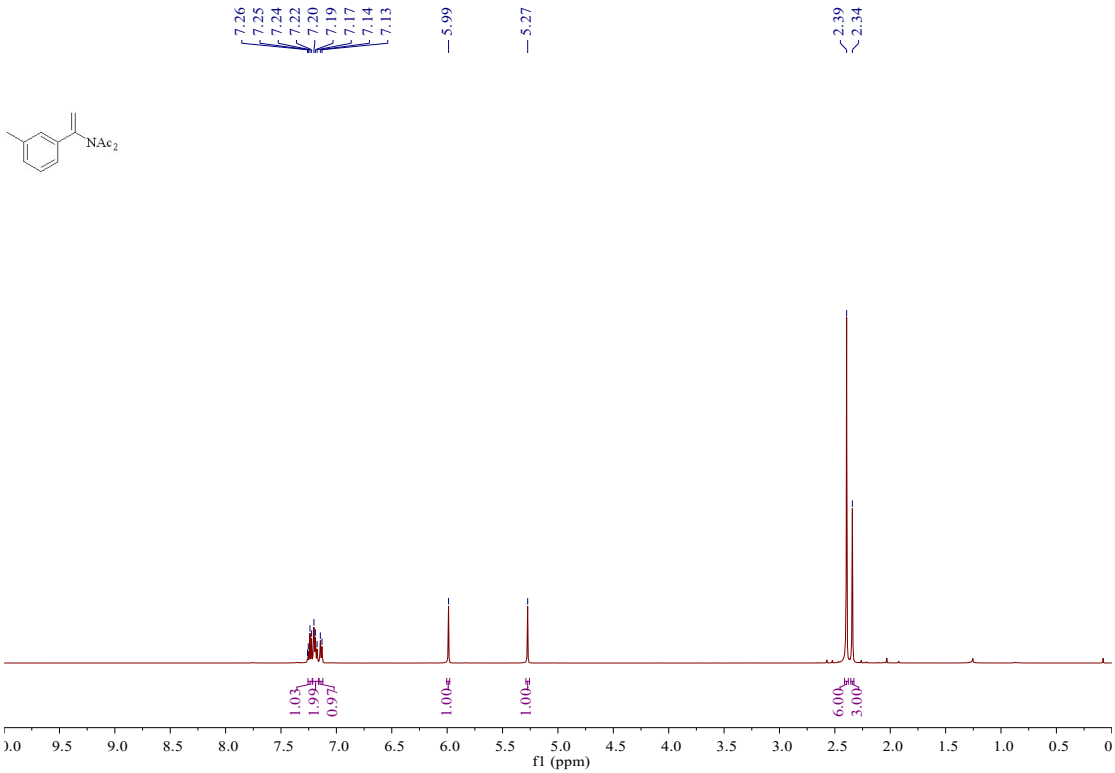
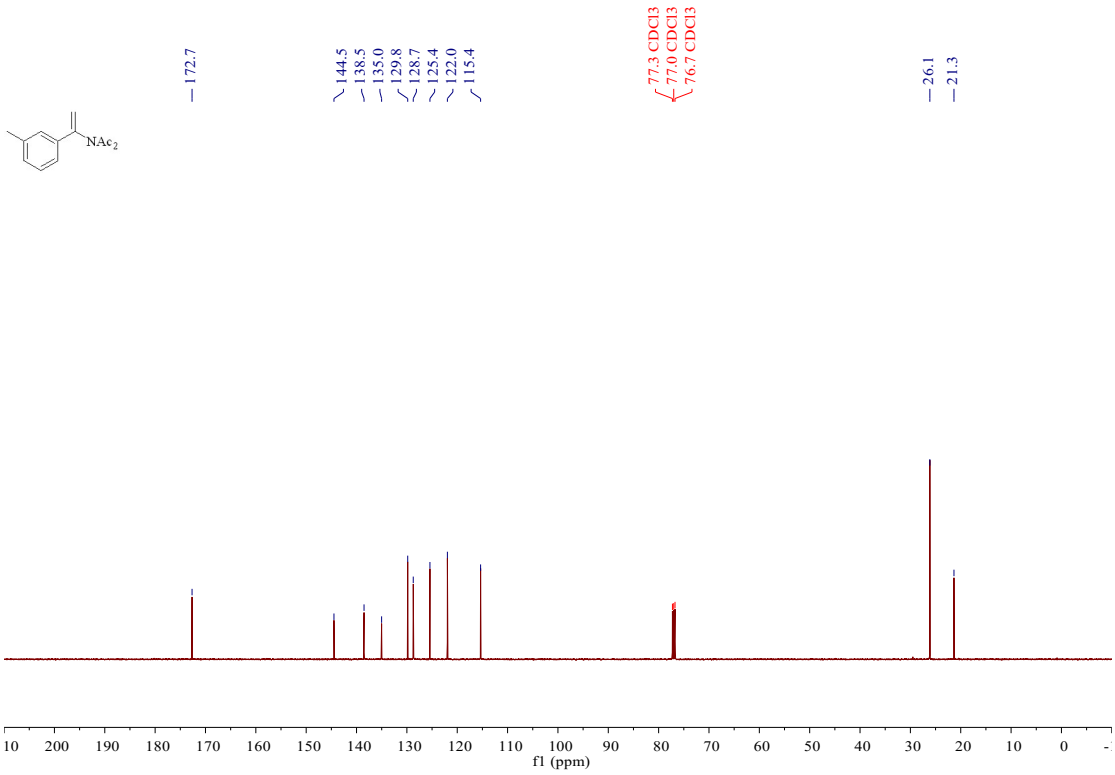
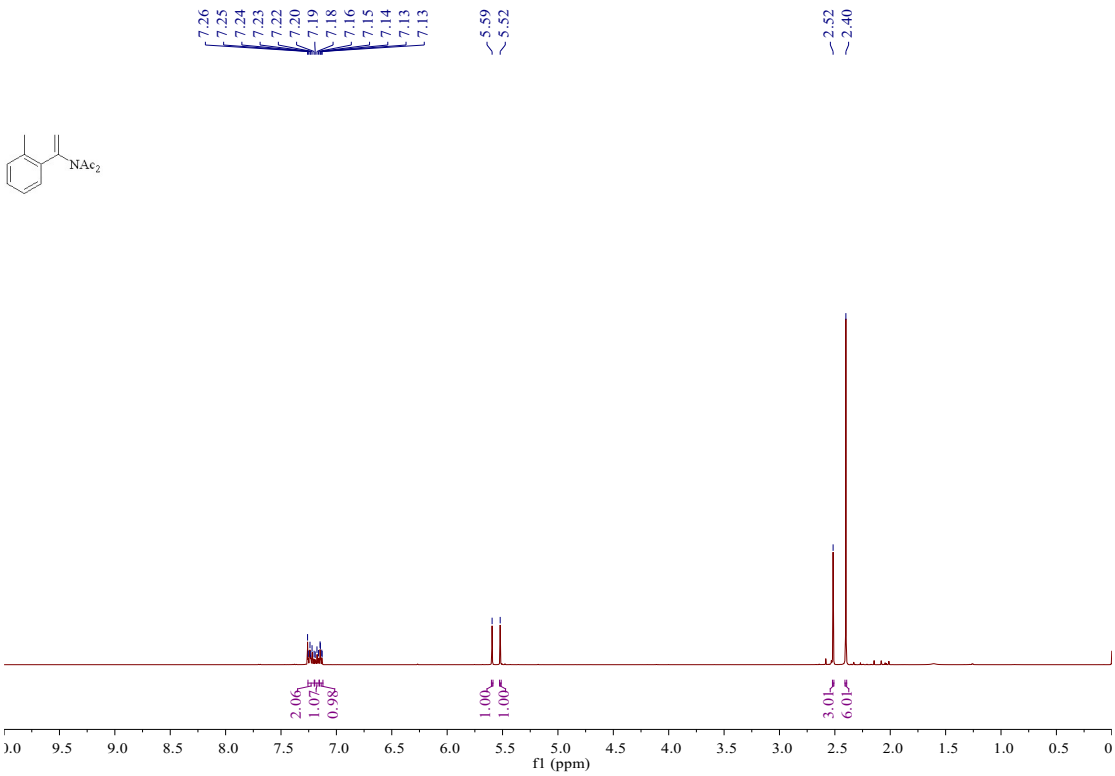


Table 1 Crystal data and structure refinement for **3**t	
Identification code	CCDC 2415008
Empirical formula	$C_{15}H_{21}NO_2$
Formula weight	247.34
Temperature/K	265(2)
Crystal system	monoclinic
Space group	Cc
a/Å	13.6440(1)
b/Å	13.7725(1)
c/Å	16.2084(2)
α/°	90
β/°	105.381(3)
γ/°	90
Volume/Å3	2936.7(5)
Z	1
ρ_{calc}g/cm^3	1.119
μ/mm^{-1}	0.074
F(000)	1072.5
Crystal size/mm^3	0.3 × 0.15 × 0.2
Radiation	M_oK_α (λ = 0.71073)
2Θ range for data collection/°	4.28 to 50
Index ranges	-16≤h≤15, -16≤k≤16, -19≤l≤19
Reflections collected	15769
Independent reflections	5046[R_{int} = 0.0441, R_{sigma} = 0.0479]
Data/restraints/parameters	5046/4/333
Goodness-of-fit on F^2	1.046
Final R indexes [I>=2σ (I)]	R_1 = 0.0624, wR_2 = 0.1600
Final R indexes [all data]	R_1 = 0.0873, wR_2 = 0.1848
Largest diff. peak/hole / e Å^{-3}	0.34/-0.39

7. NMR Spectra of new compounds



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7.13
5.59
5.52
2.52
2.40
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1.07
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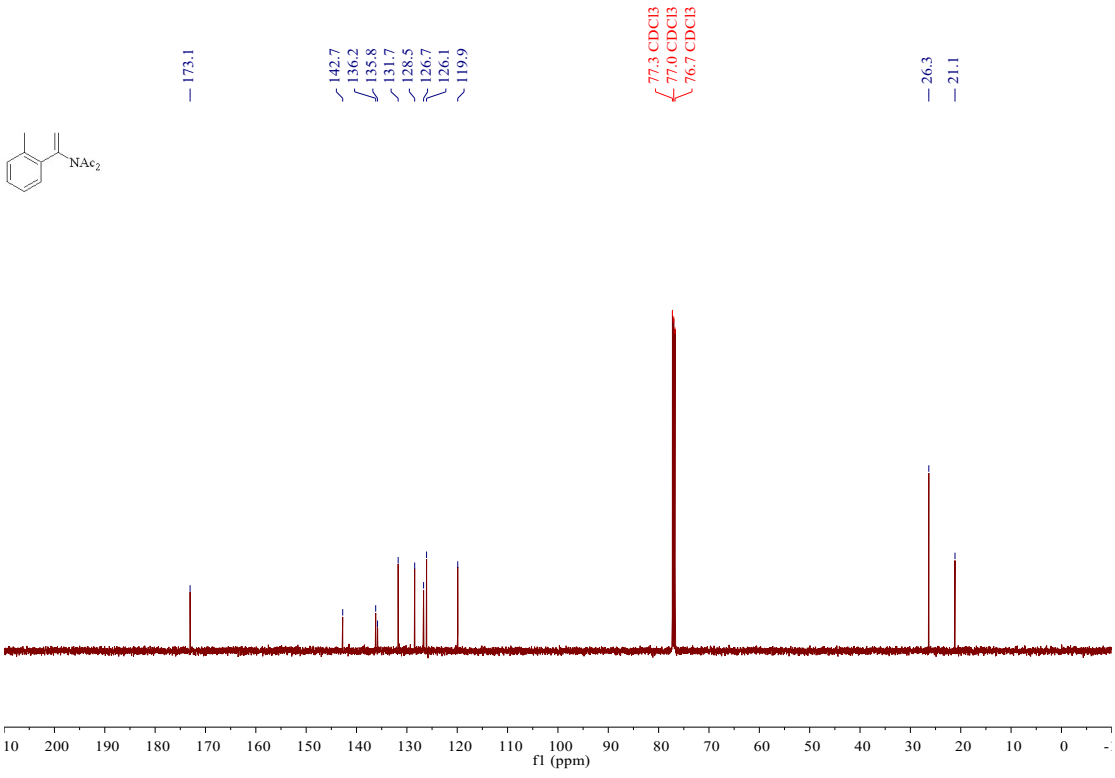
1.00
1.00
3.01
6.01
f1 (ppm)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131.7
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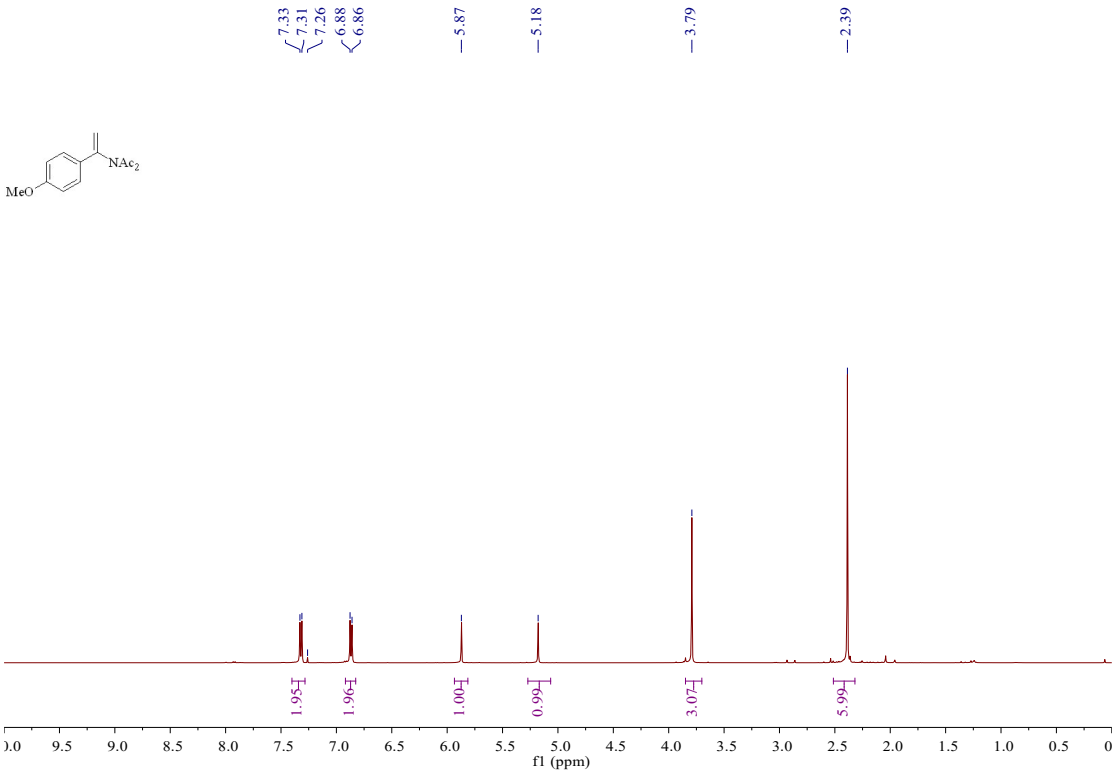
NAc2
f1 (ppm)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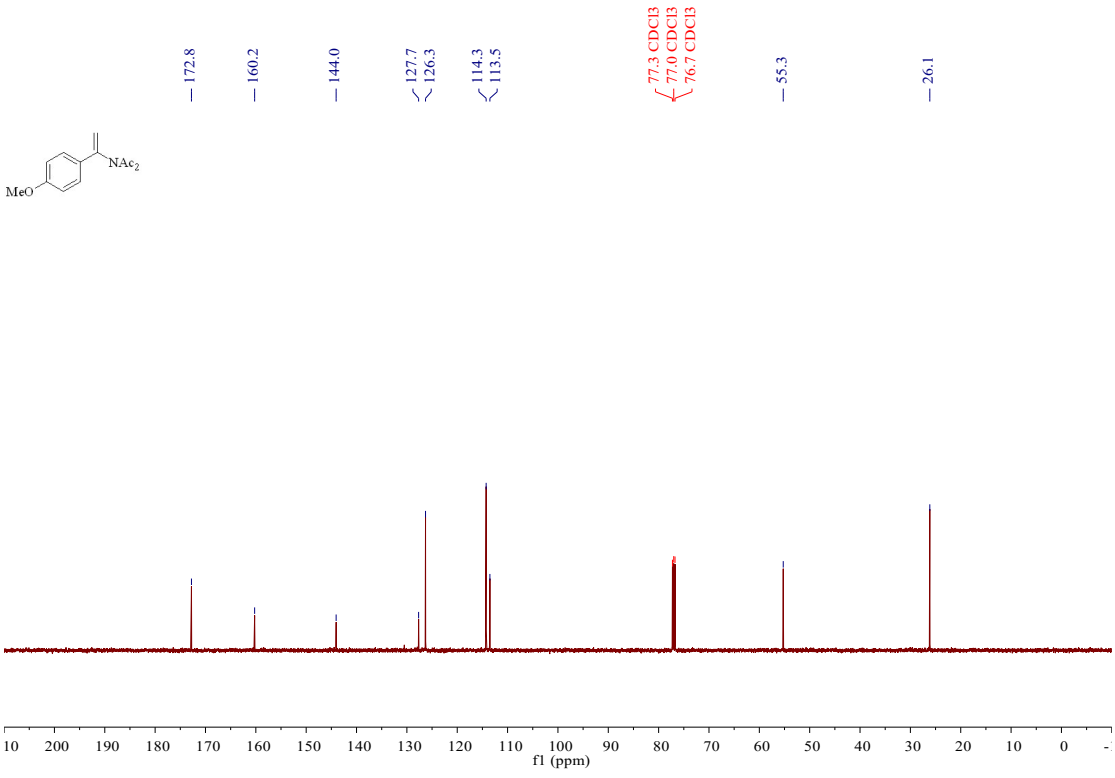
5.99
f1 (ppm)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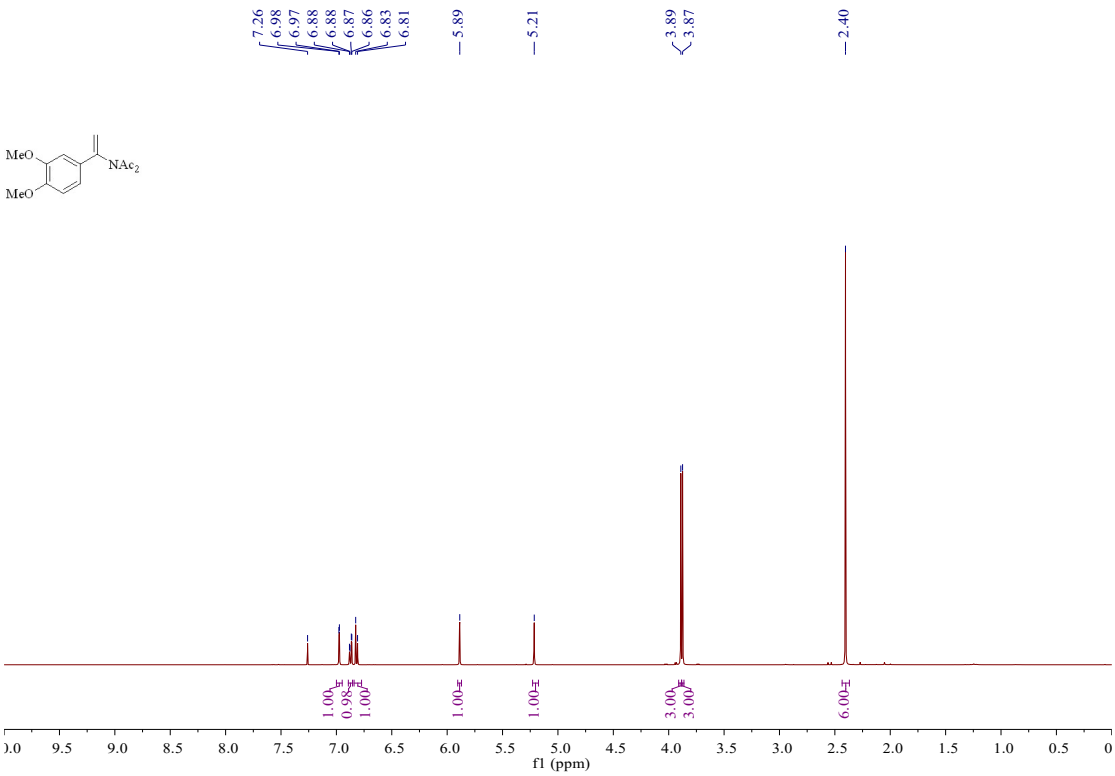
f1 (ppm)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2.40
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1.00
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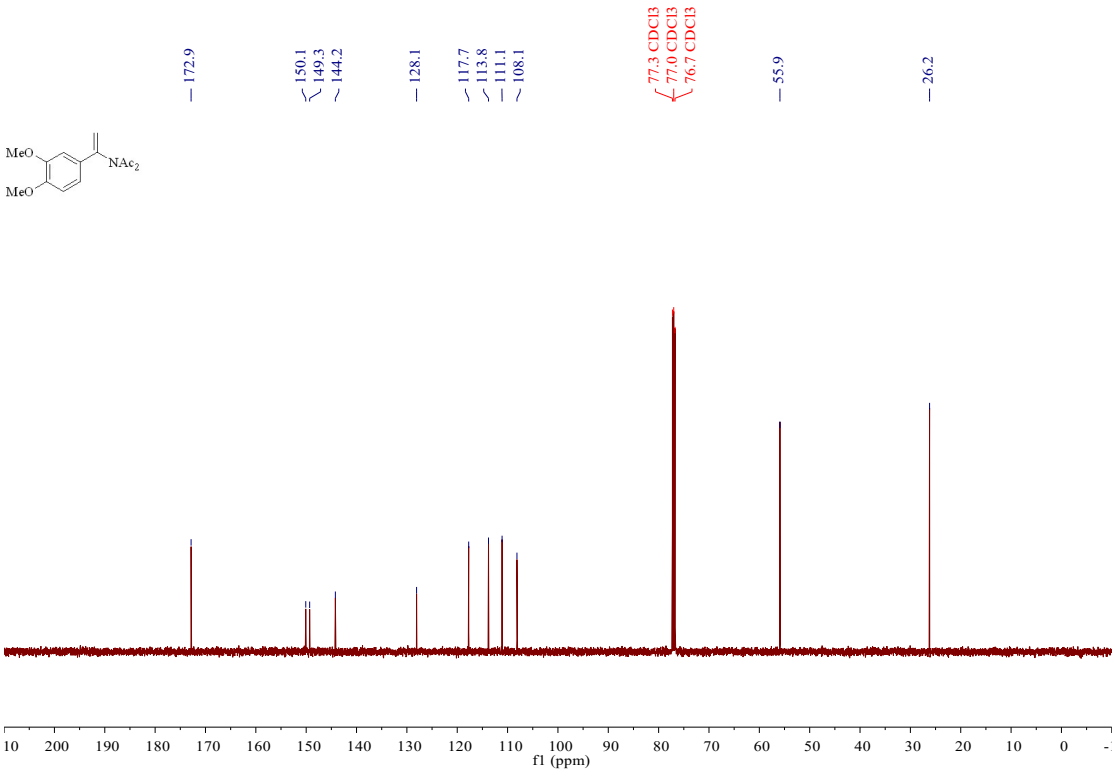
6.00
f1 (ppm)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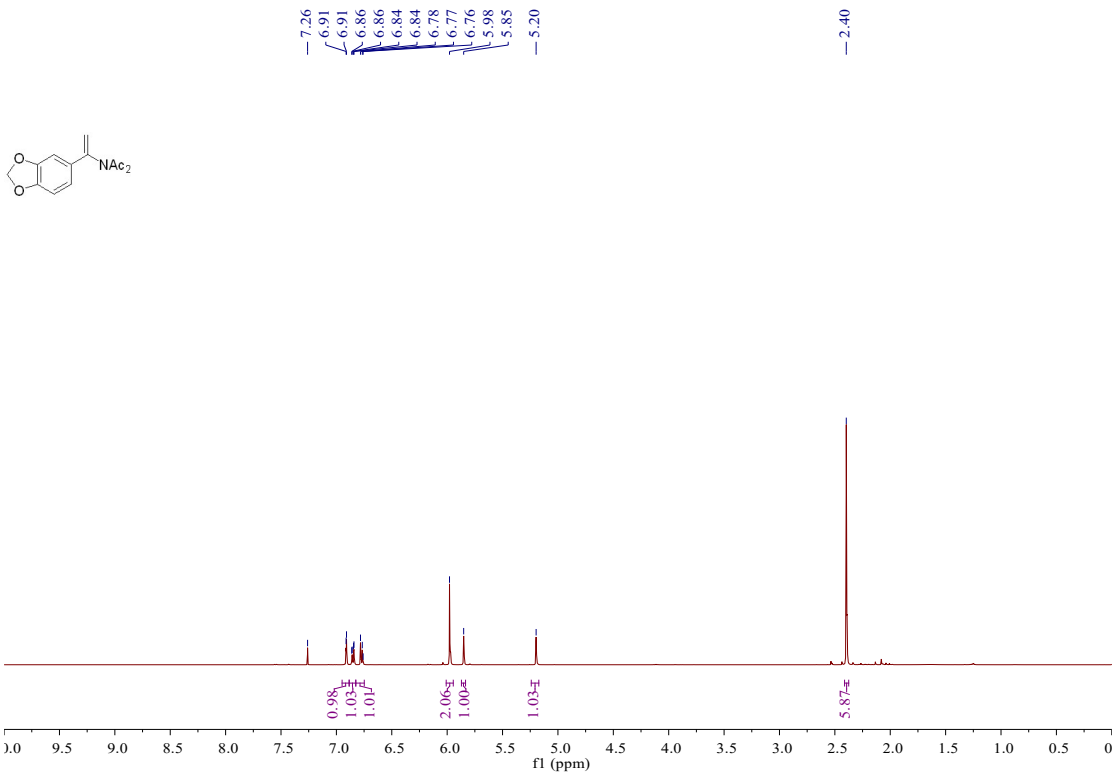
f1 (ppm)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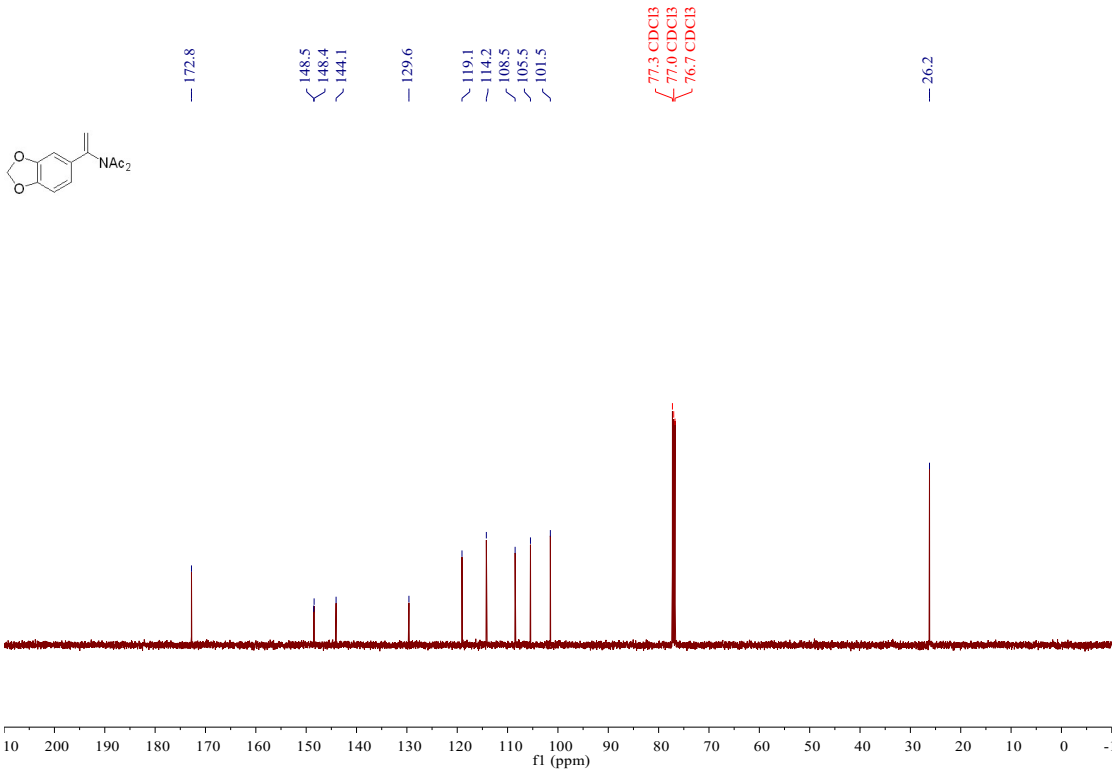
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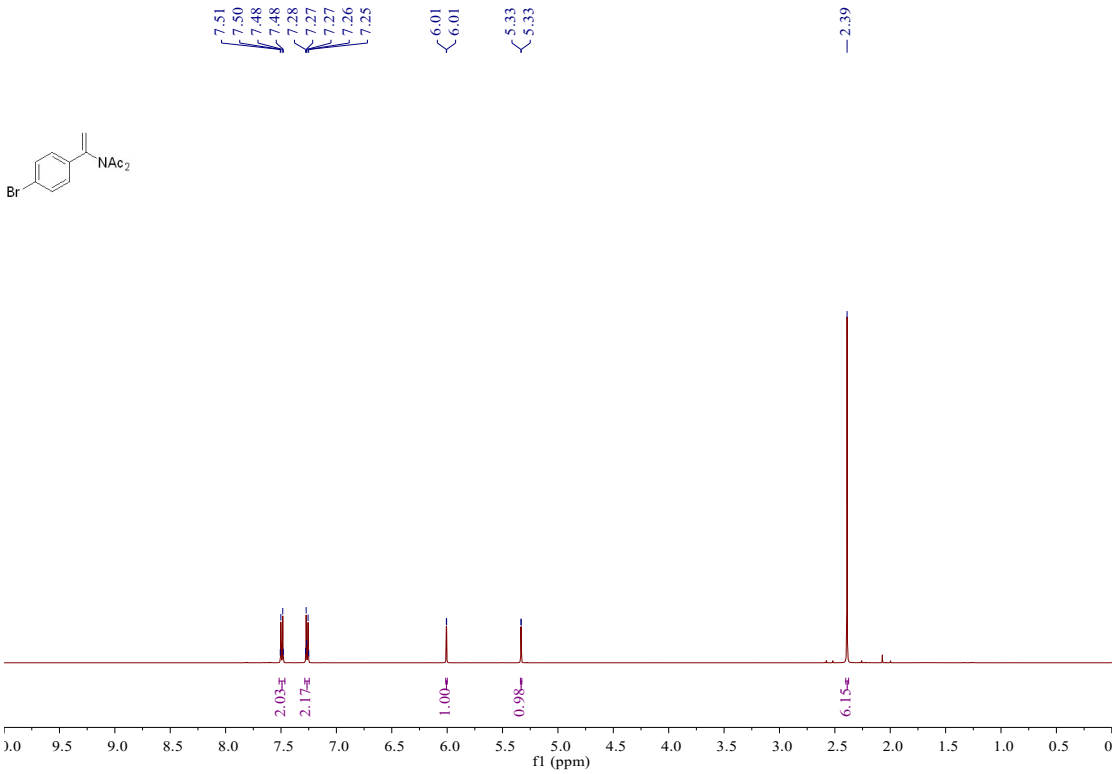
f1 (ppm)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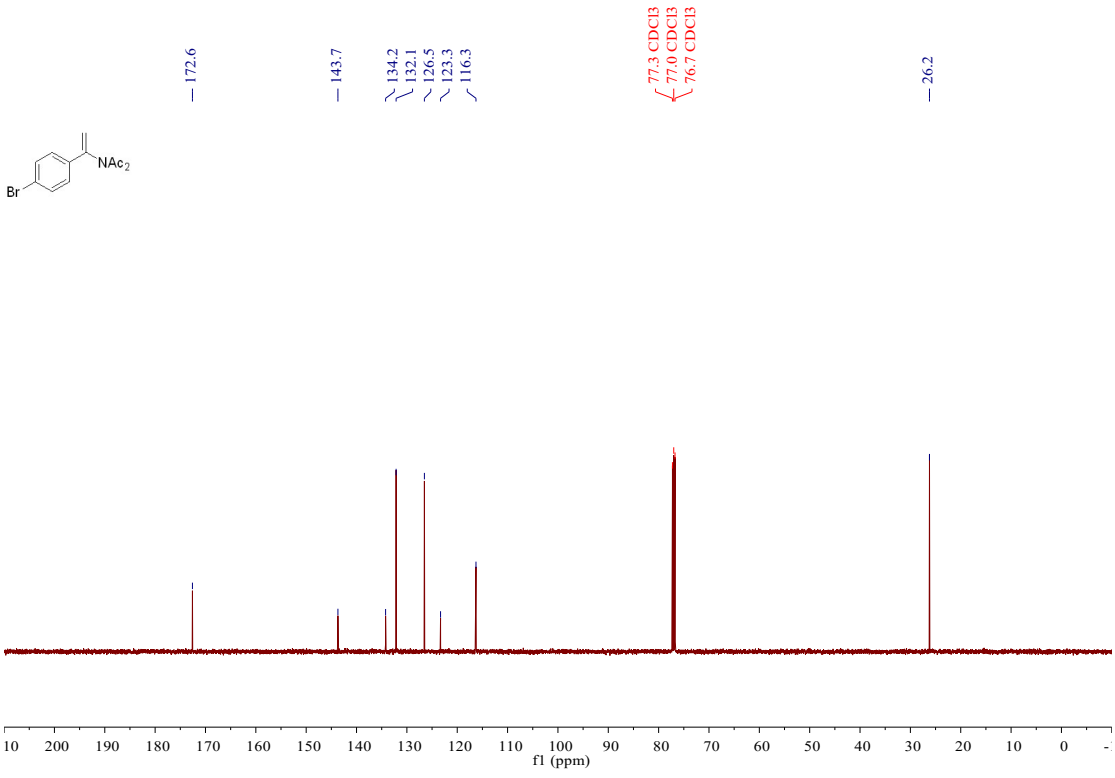
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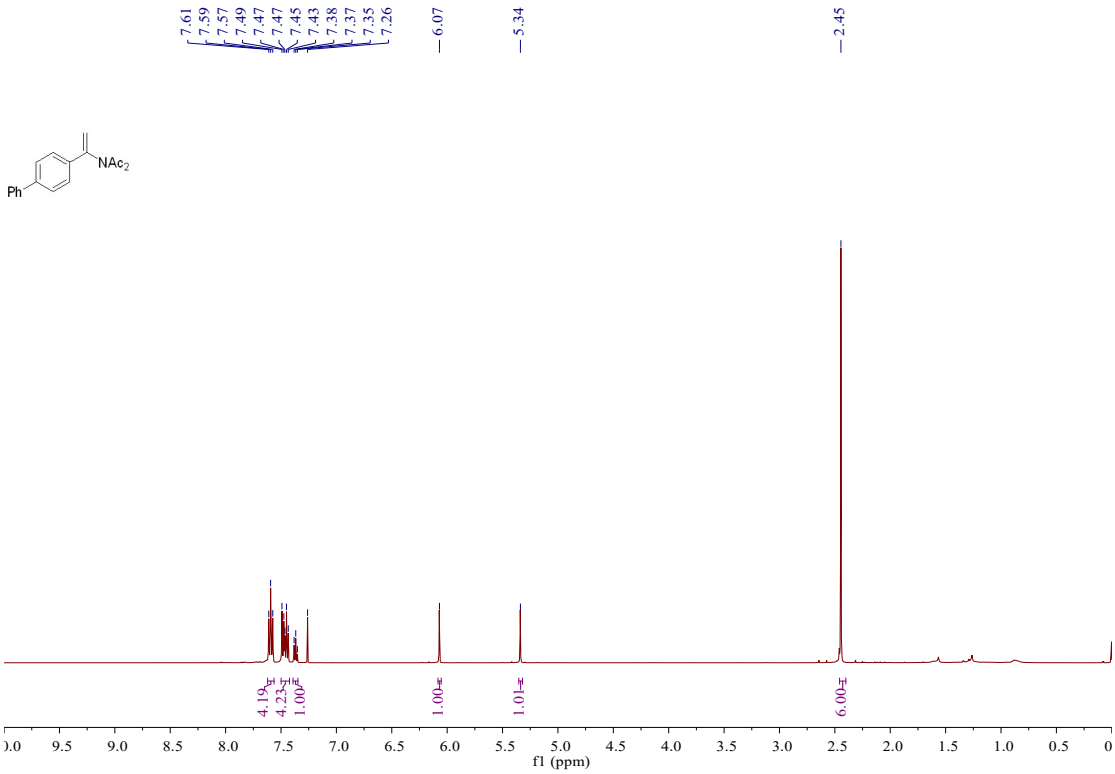
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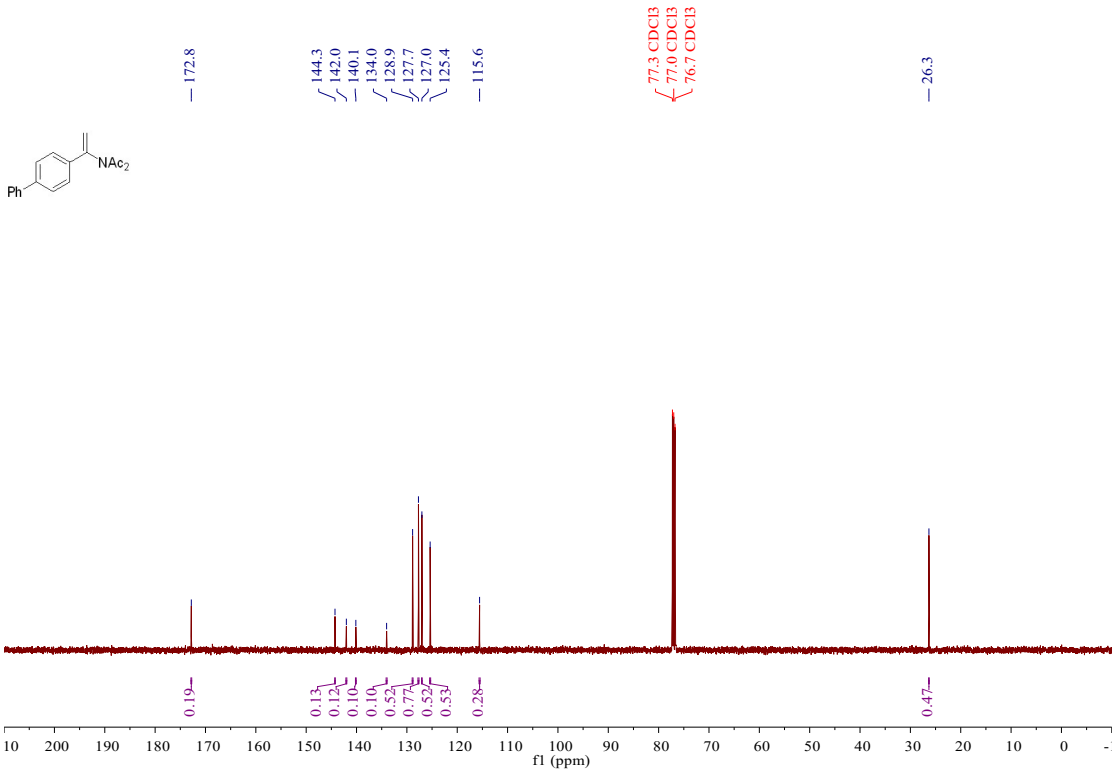
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76.7 CDCl3
26.3
NAc2
Ph
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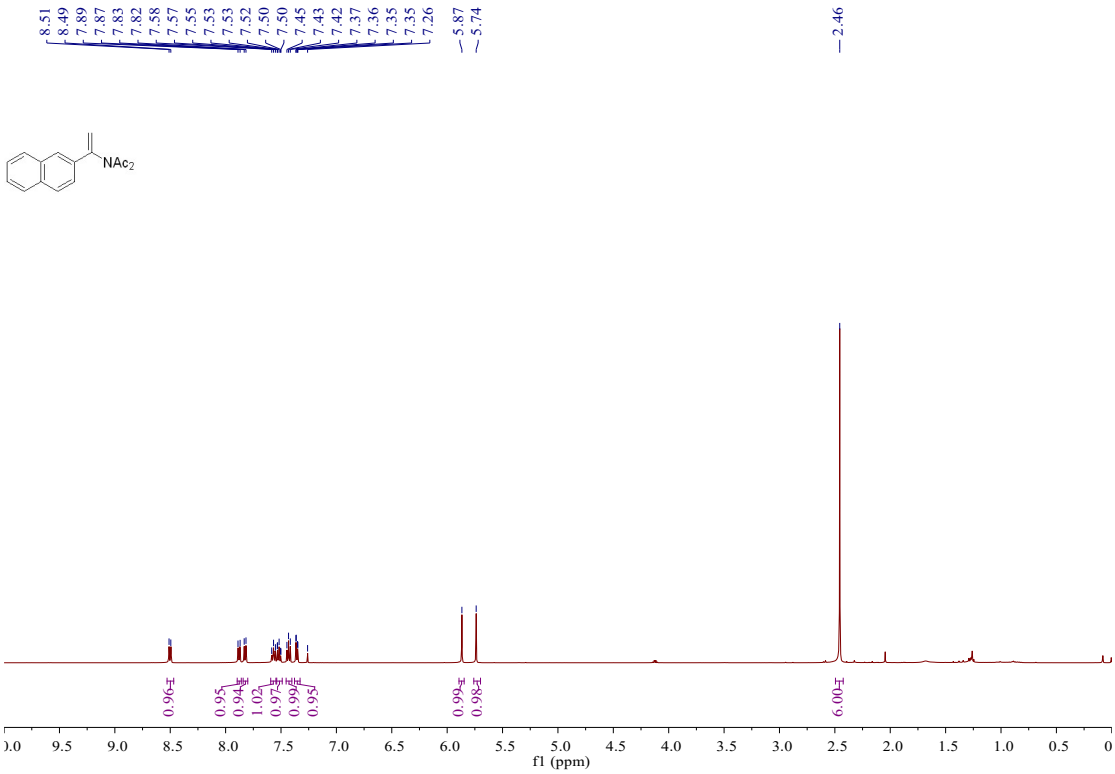
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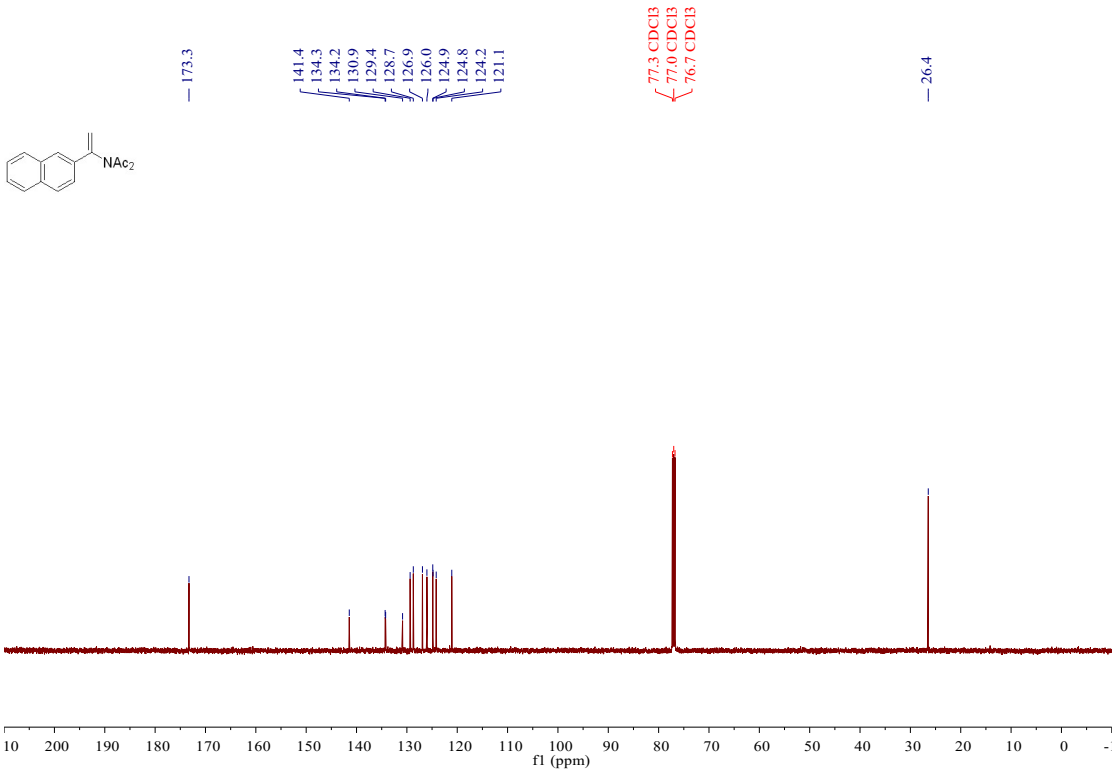
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76.7 CDCl3
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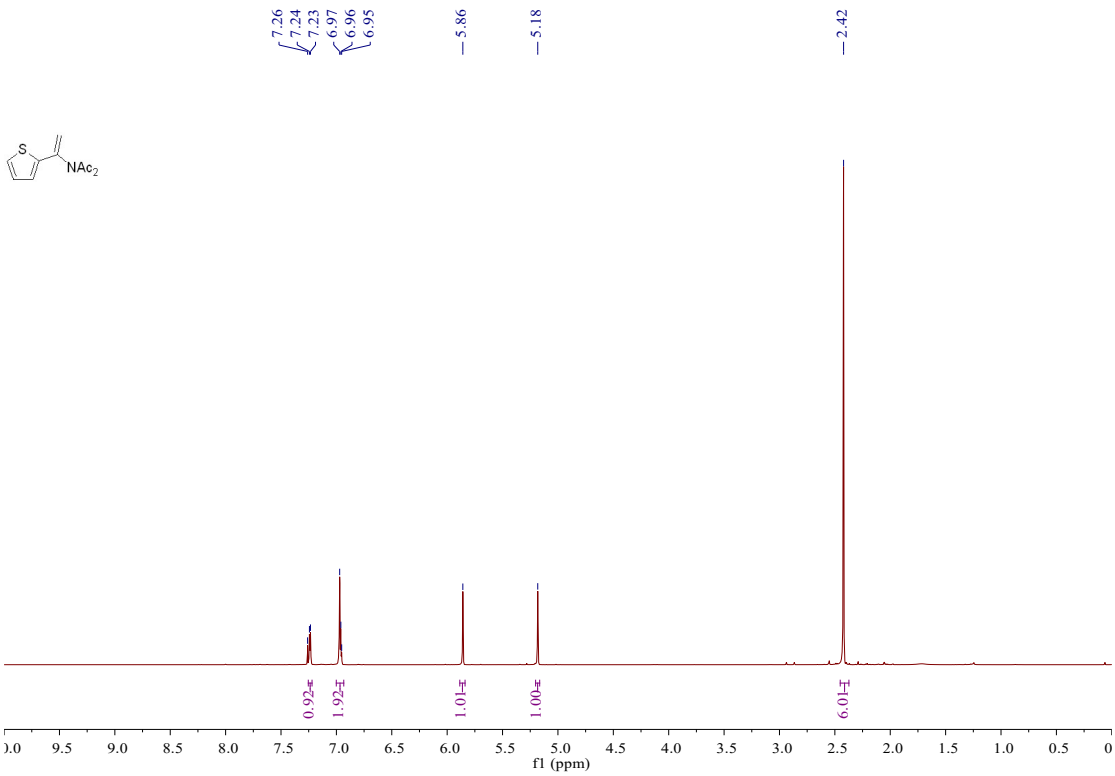
f1 (ppm)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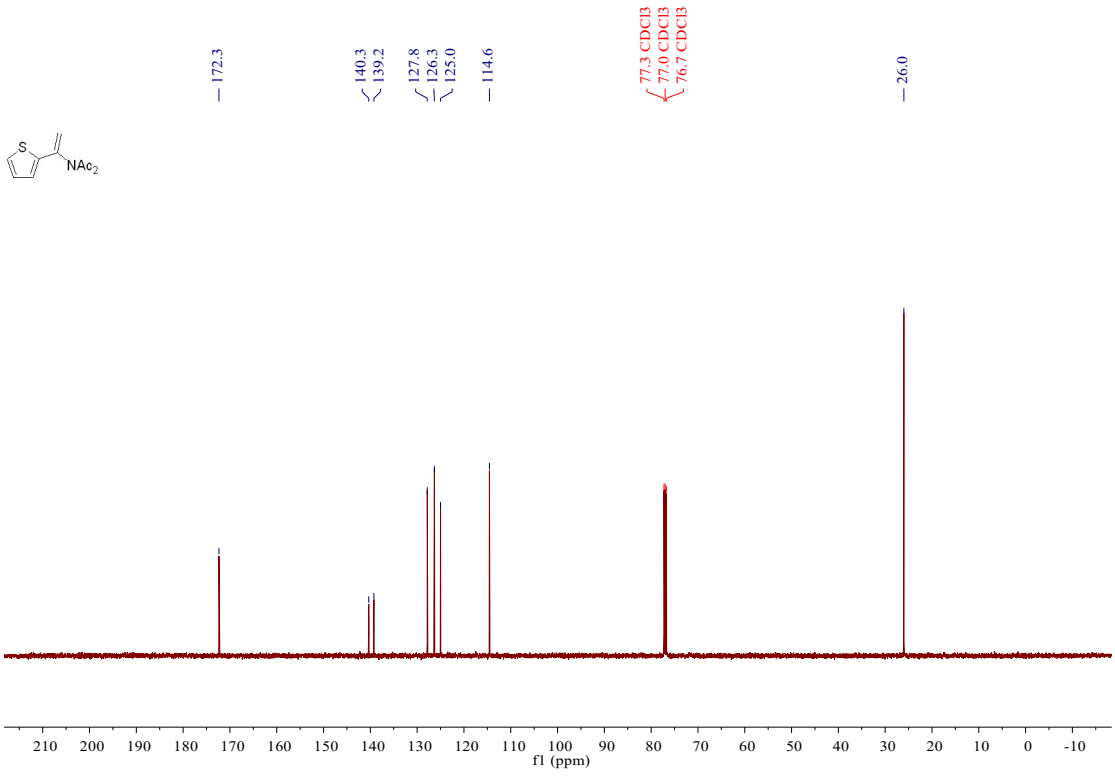
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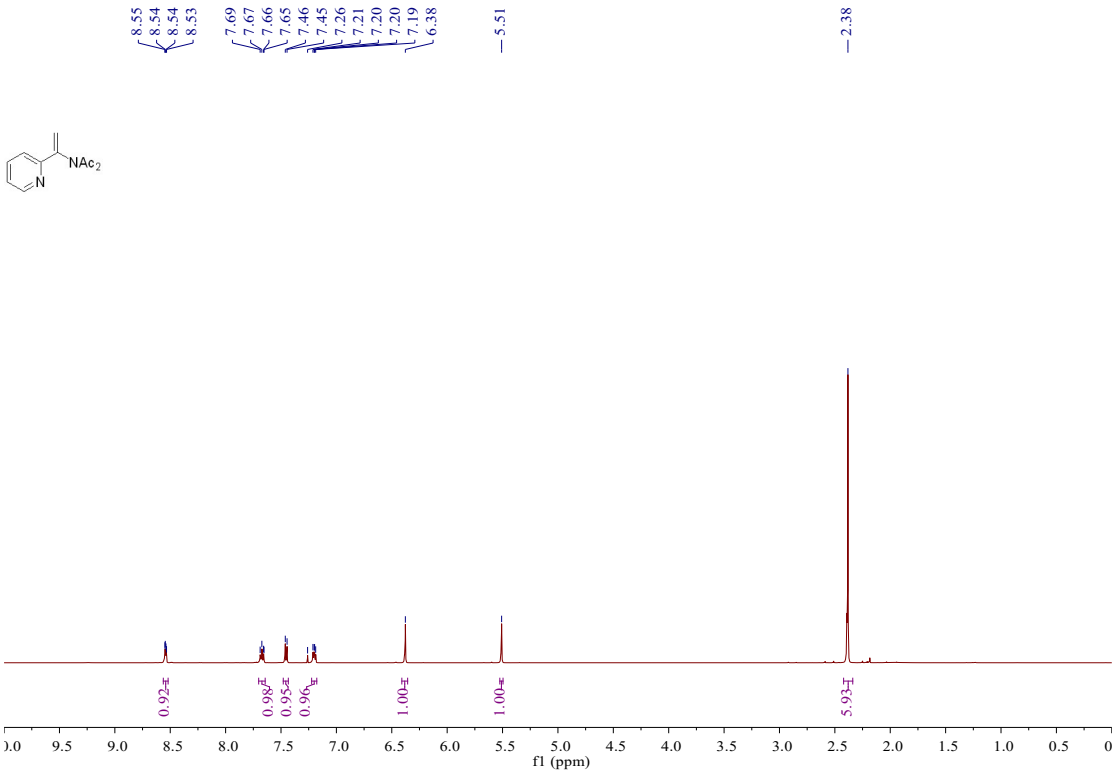
f1 (ppm)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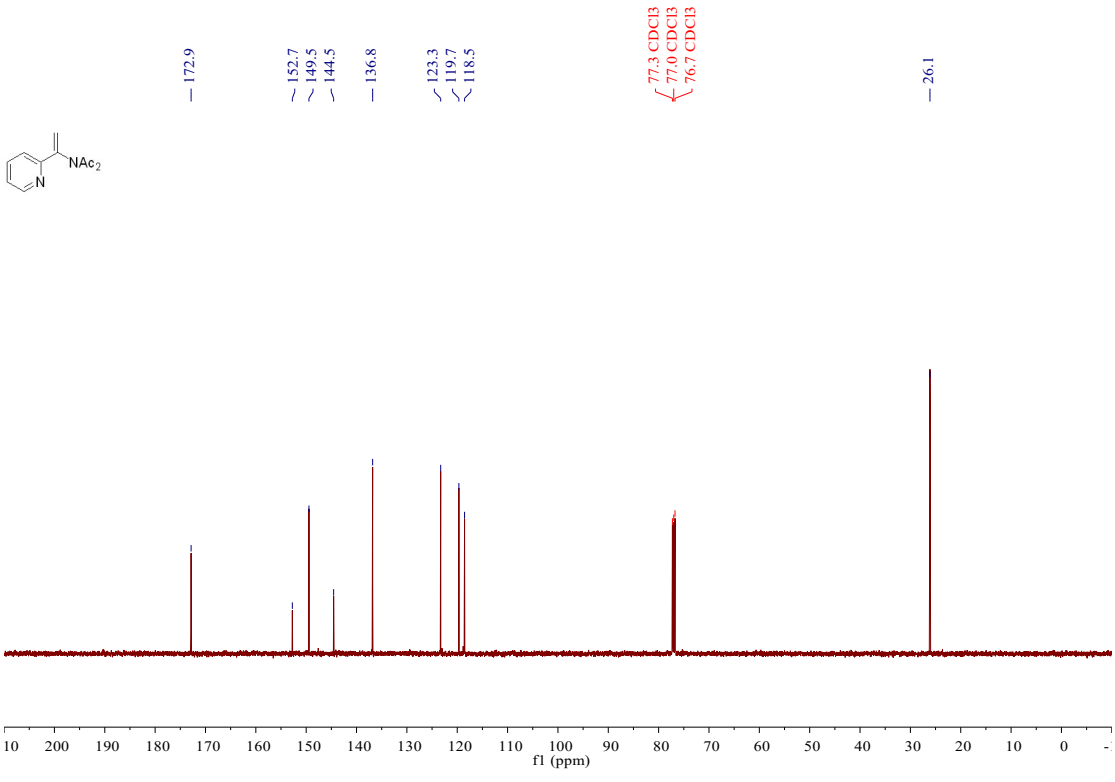
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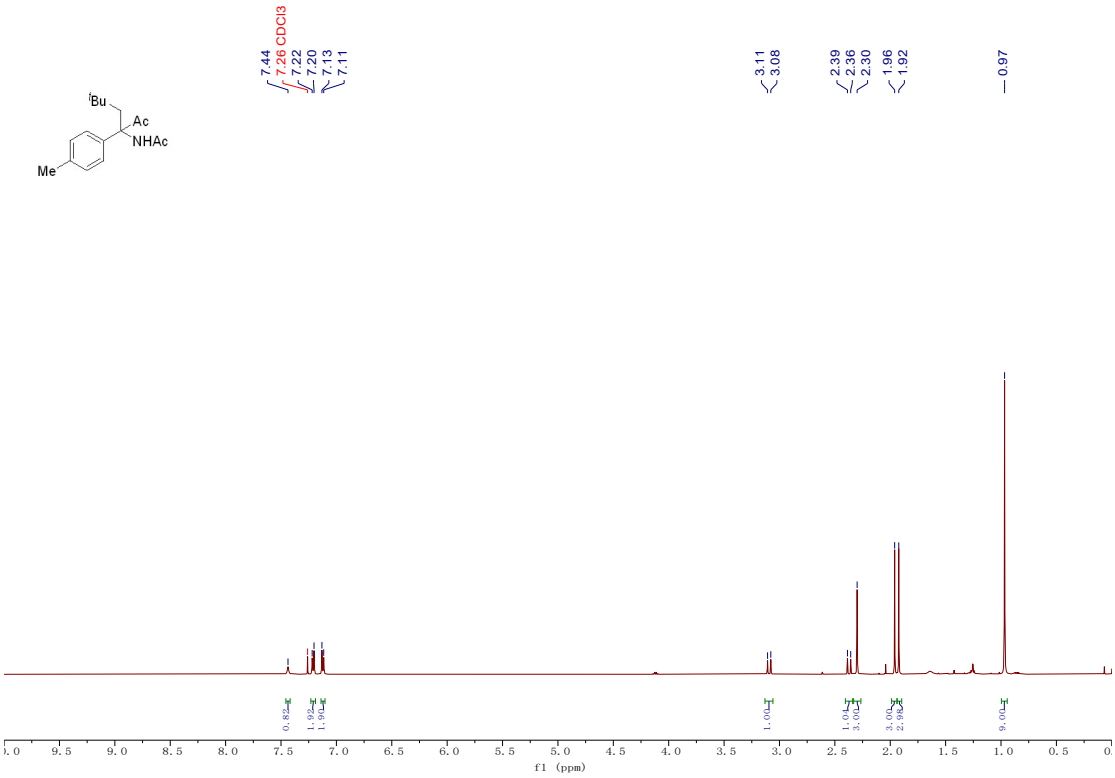
N
f1 (ppm)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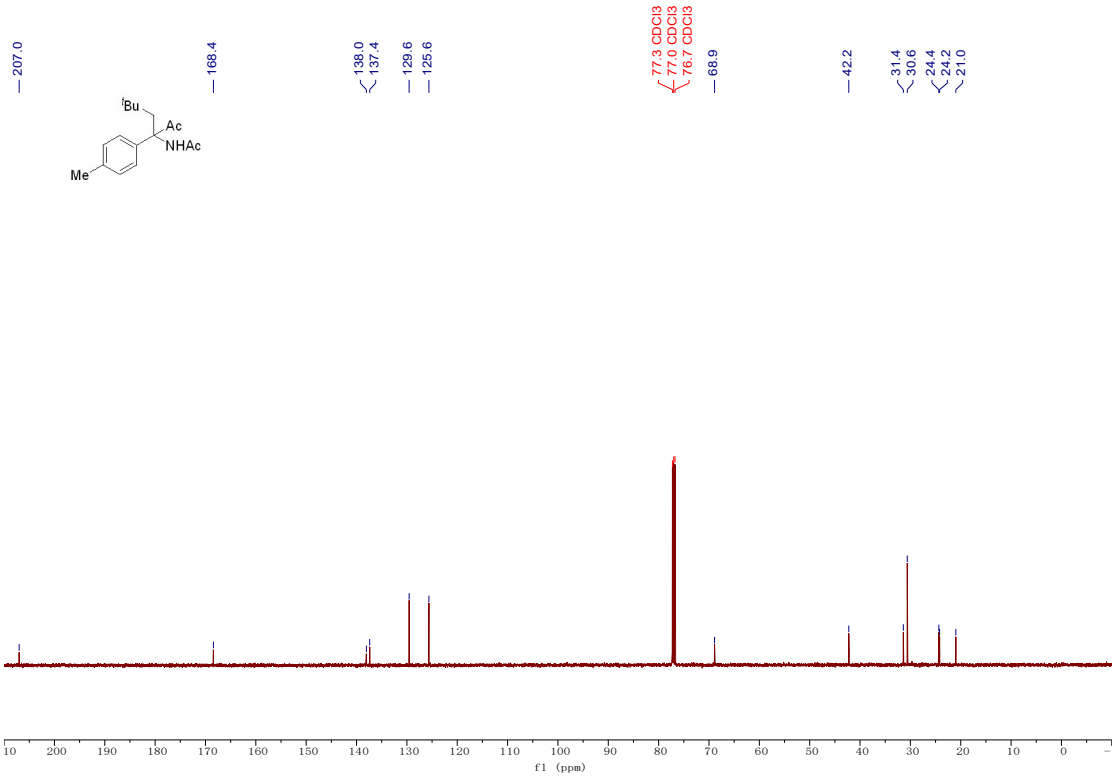
Ac
NHAc
Me
f1 (ppm)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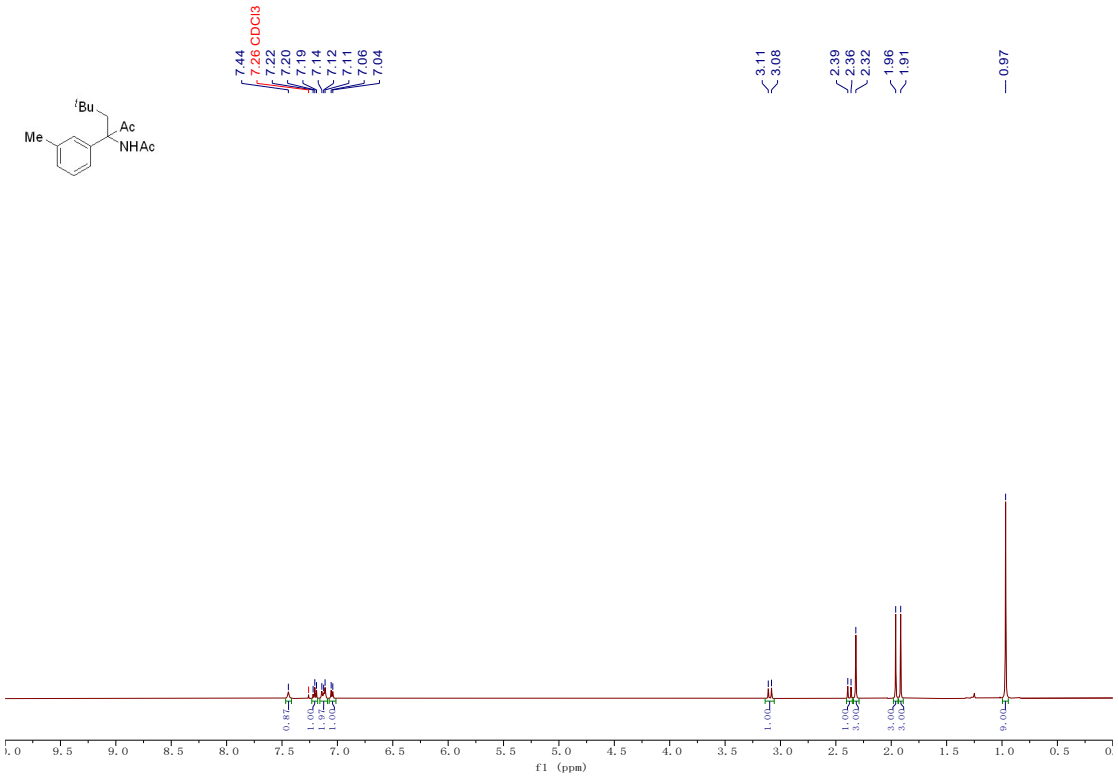
Ac
NHAc
Me
f1 (ppm)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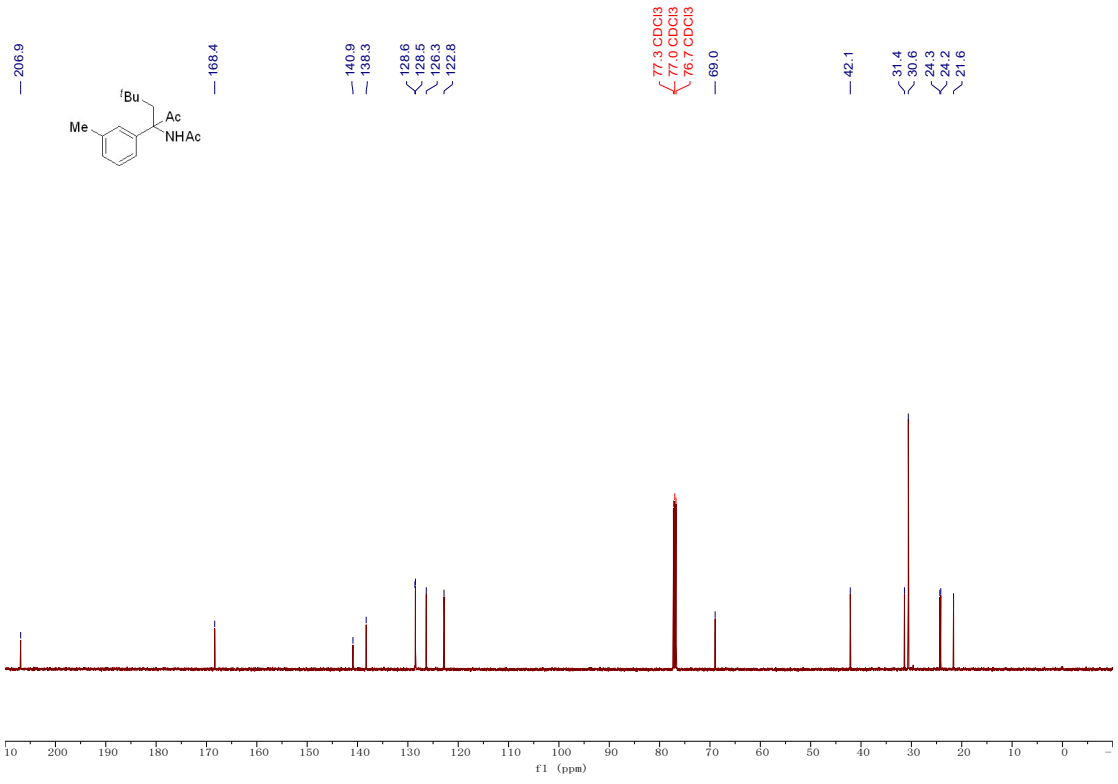
f1 (ppm)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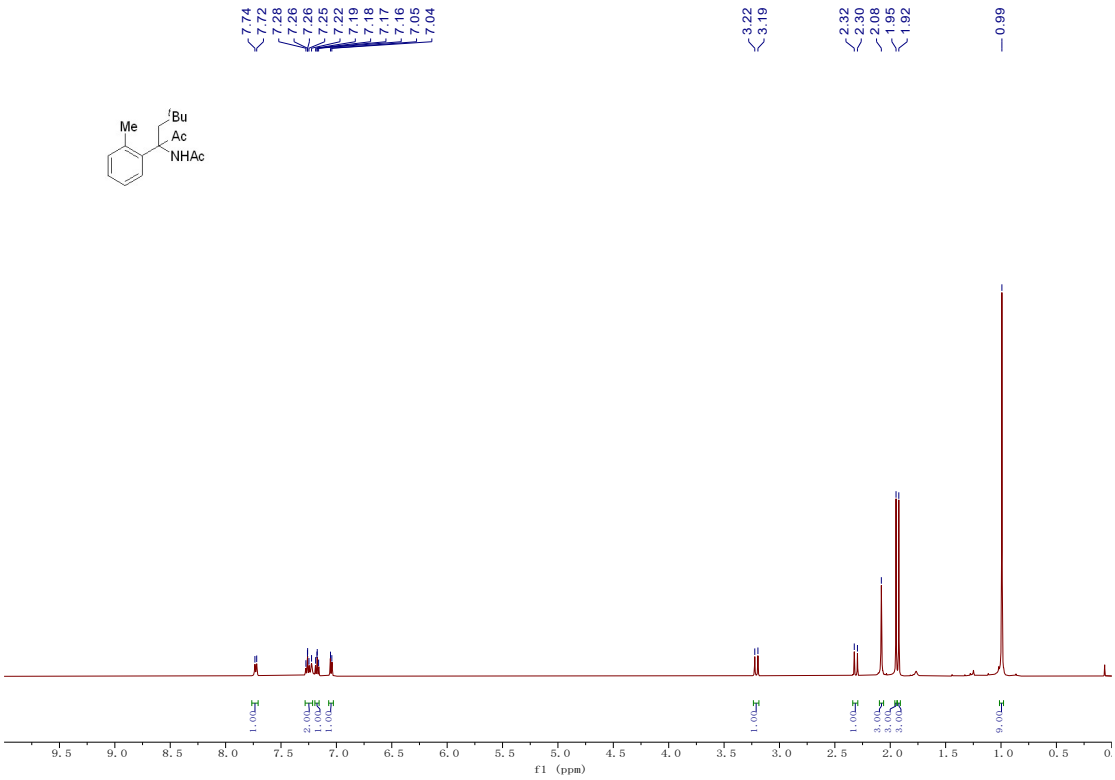
f1 (ppm)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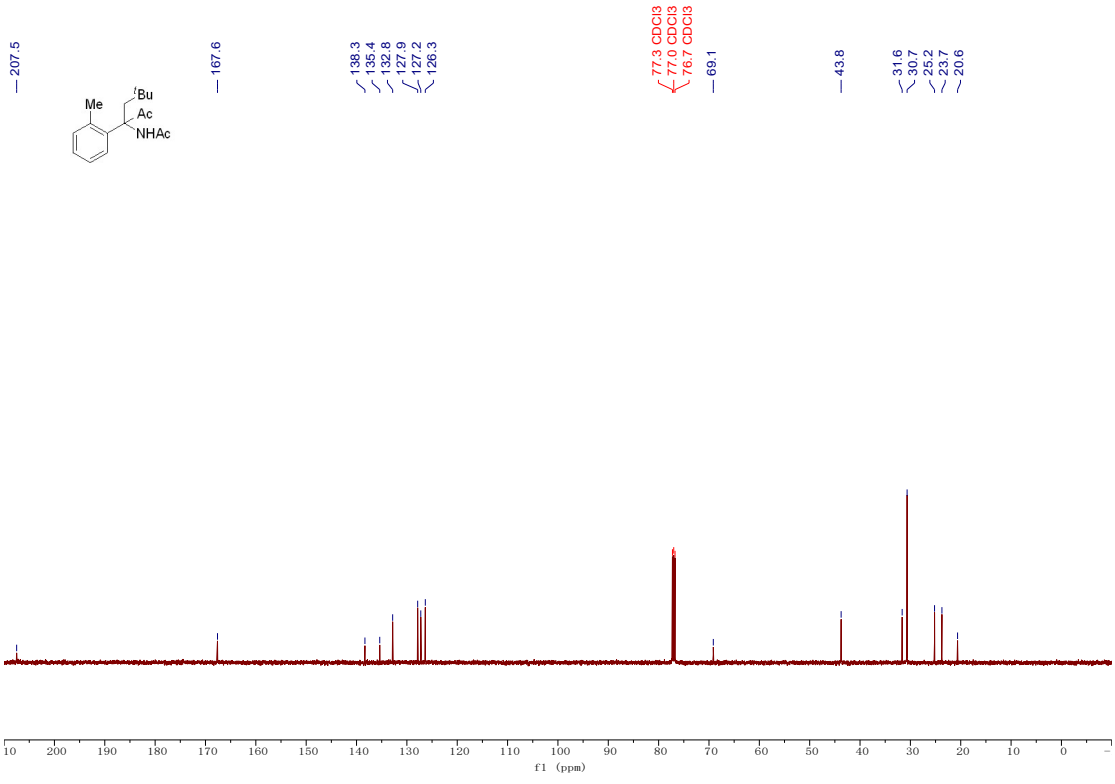
3.22
3.19
2.32
2.30
2.08
1.95
1.92
0.99
Me
tBu
Ac
NHAc
1.00
2.04
1.00
1.00
1.04
1.04
3.04
3.04
9.00
f1 (ppm)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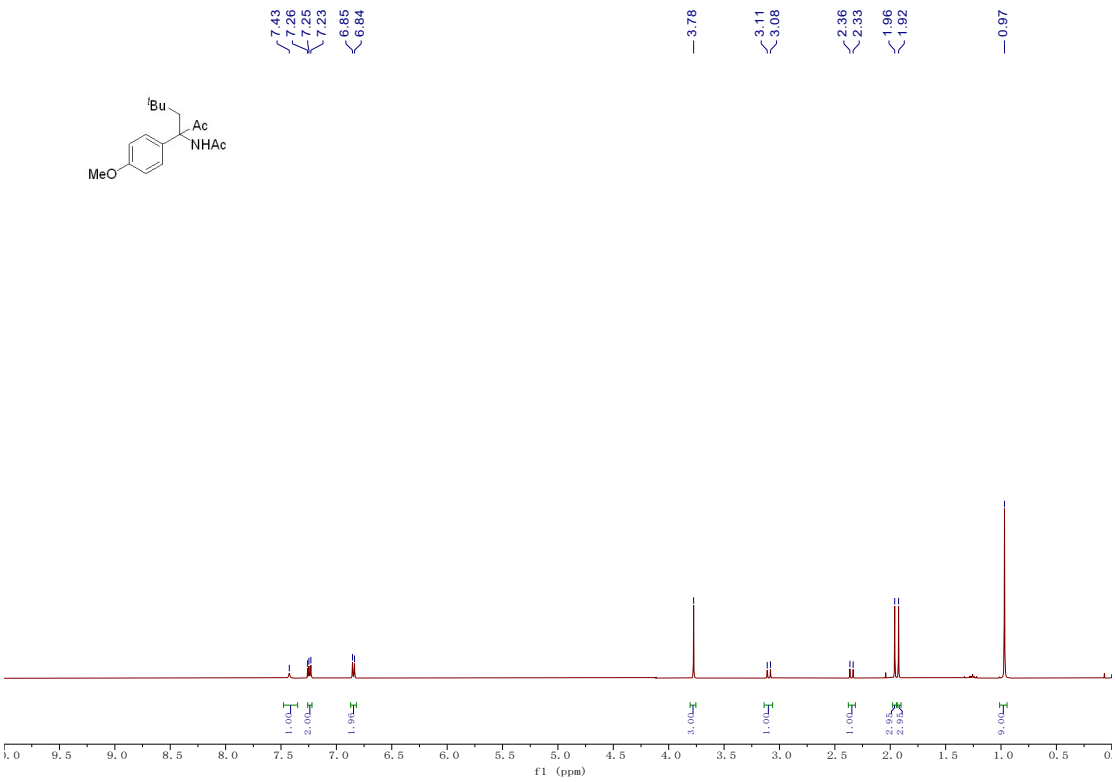
167.6
138.3
135.4
132.8
127.9
127.2
126.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.1
43.8
31.6
30.7
25.2
23.7
20.6
Me
tBu
Ac
NHAc
f1 (ppm)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7.26
7.25
7.23
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6.84
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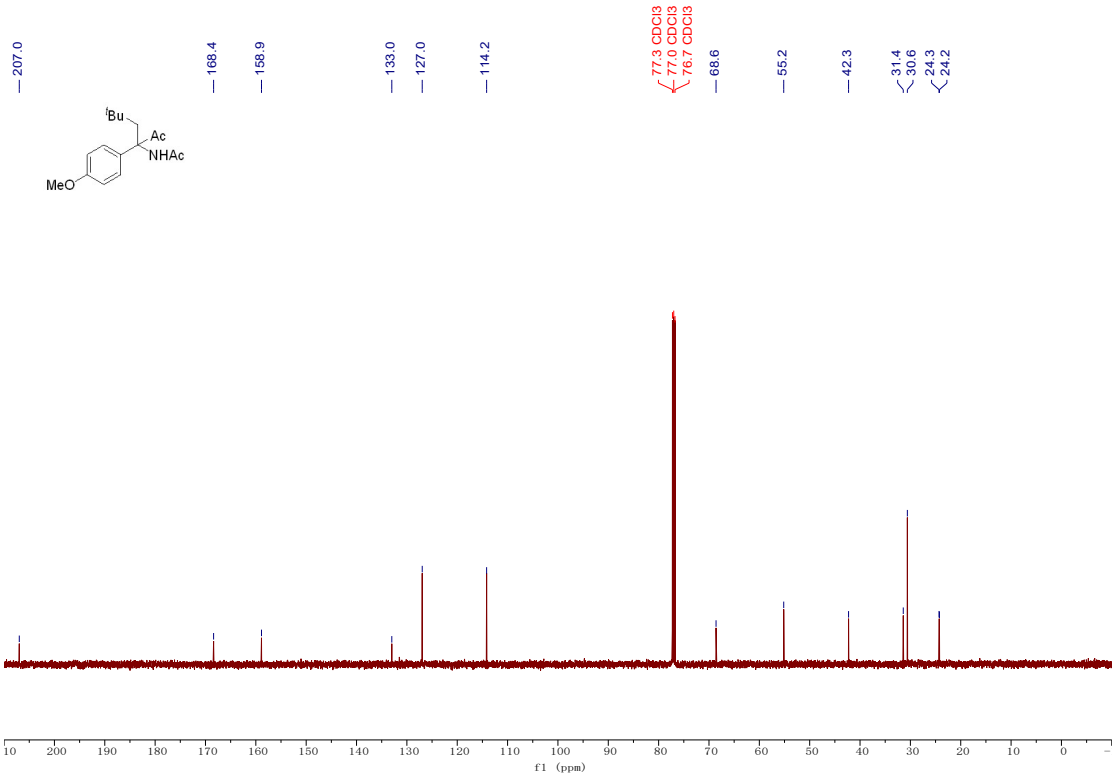
3.11
3.08
2.36
2.33
1.96
1.92
0.97
tBu
Ac
NHAc
MeO
f1 (ppm)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133.0
127.0
114.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.6
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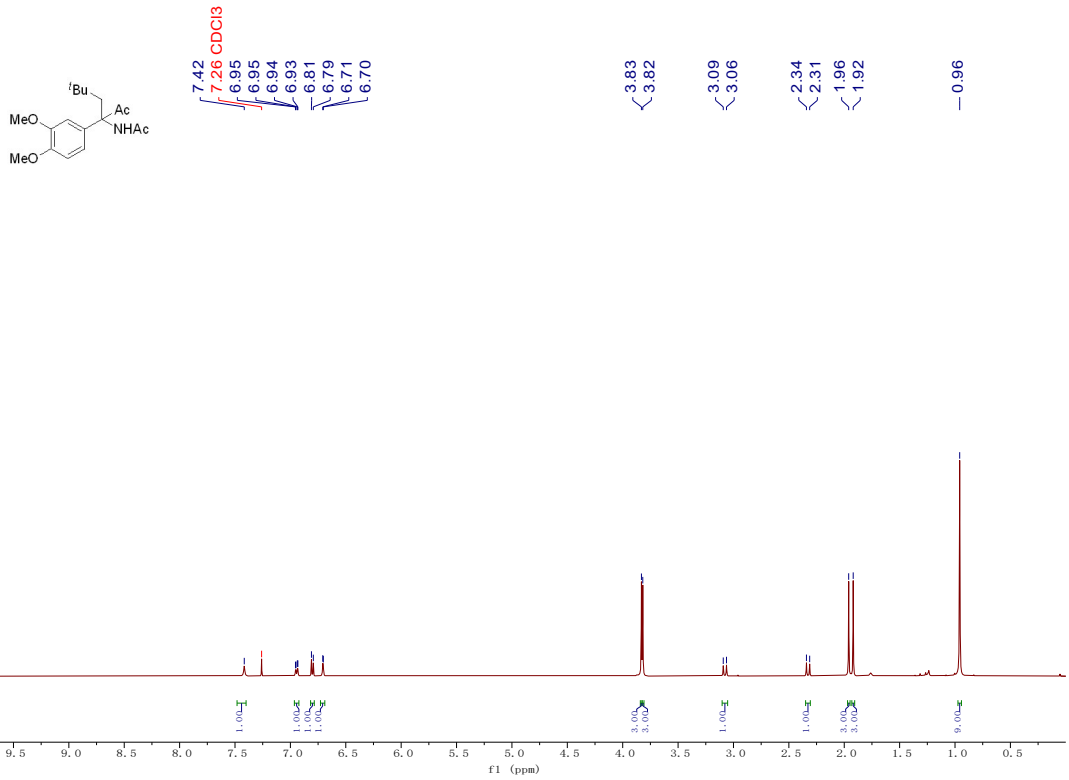
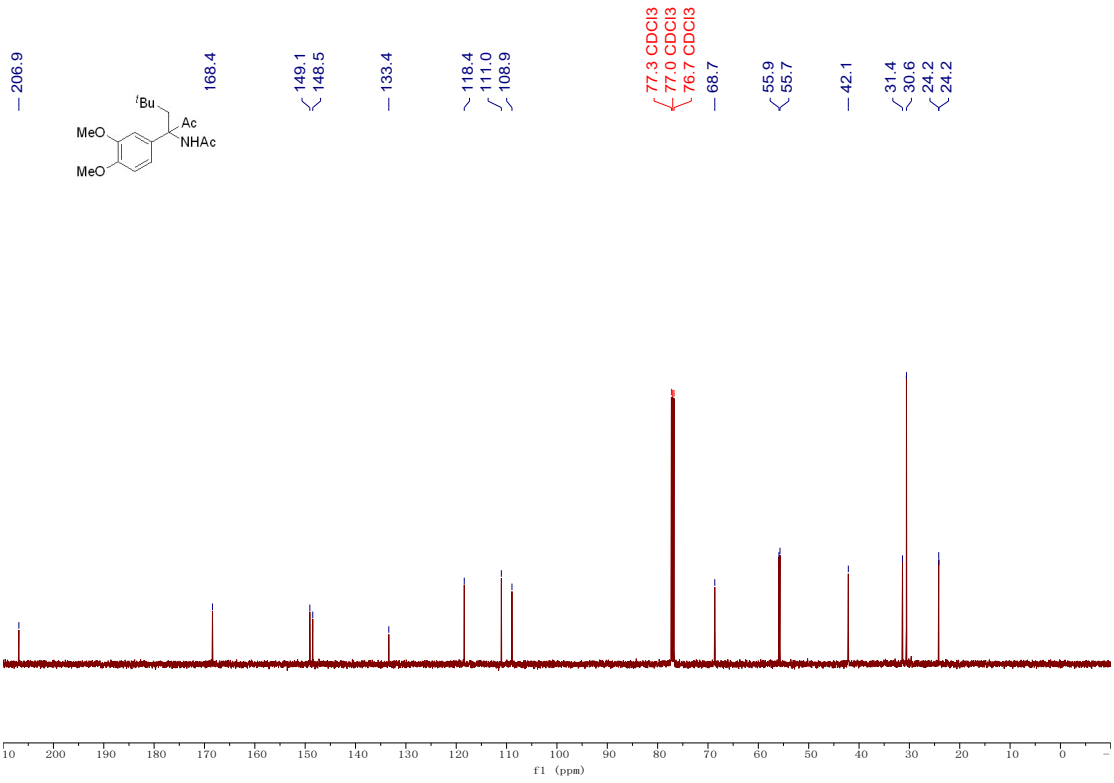
42.3
31.4
30.6
24.3
24.2
tBu
Ac
NHAc
MeO
f1 (ppm)


tBu
Ac
NHAc
MeO
MeO
7.42
7.26 CDCl3
6.95
6.95
6.94
6.93
6.81
6.79
6.71
6.70
3.83
3.82
3.09
3.06
2.34
2.31
1.96
1.92
0.96
f1 (ppm)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148.5
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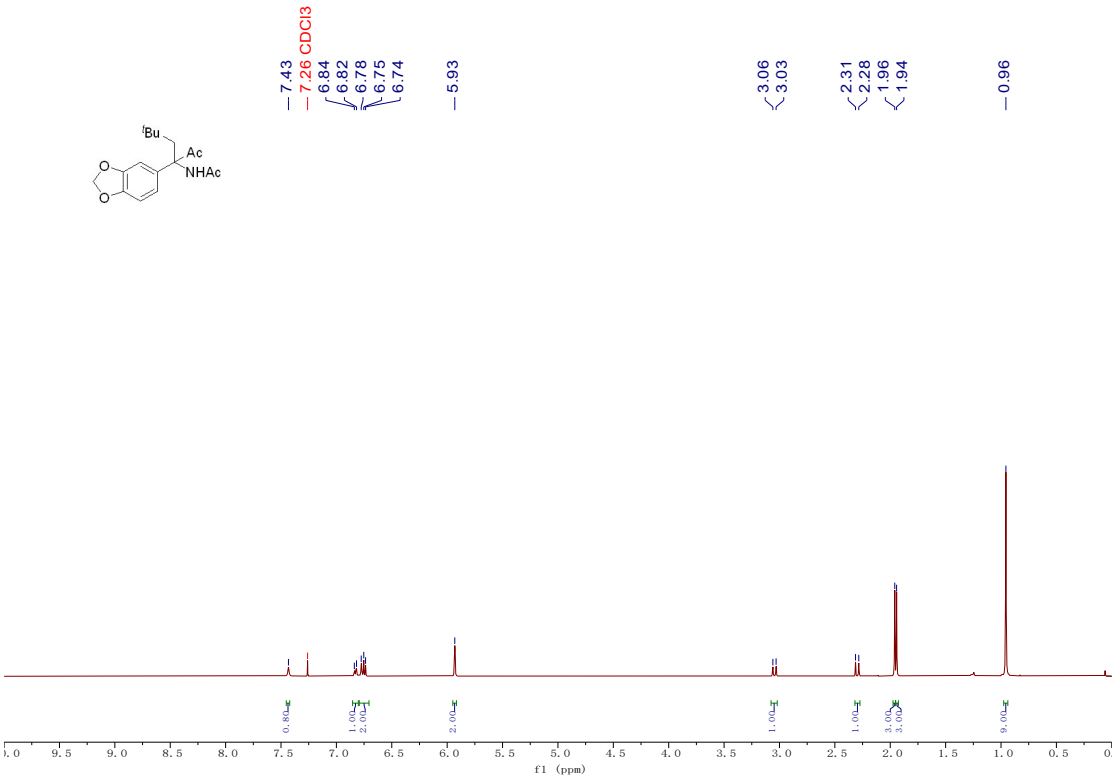
118.4
111.0
108.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.7
55.9
55.7
42.1
31.4
30.6
24.2
24.2
tBu
Ac
NHAc
MeO
MeO
f1 (ppm)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7.26 CDCl3
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6.78
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6.74
5.93
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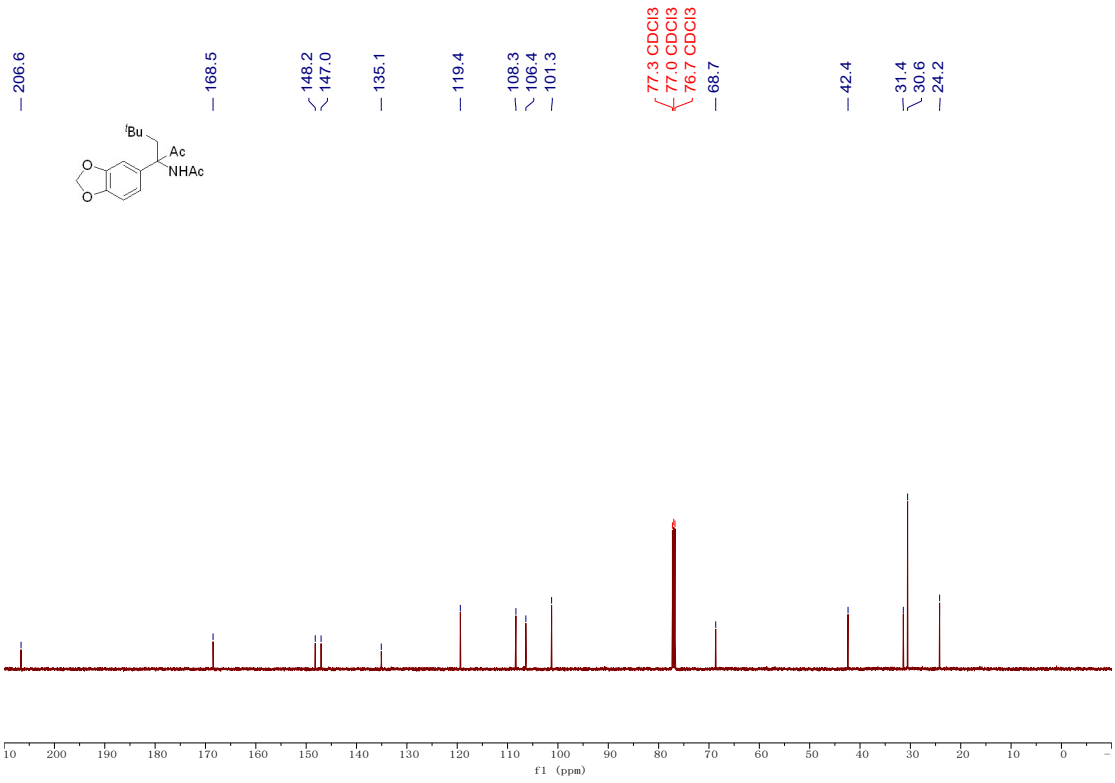
3.03
2.31
2.28
1.96
1.94
0.96
tBu
Ac
NHAc
f1 (ppm)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106.4
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77.3 CDCl3
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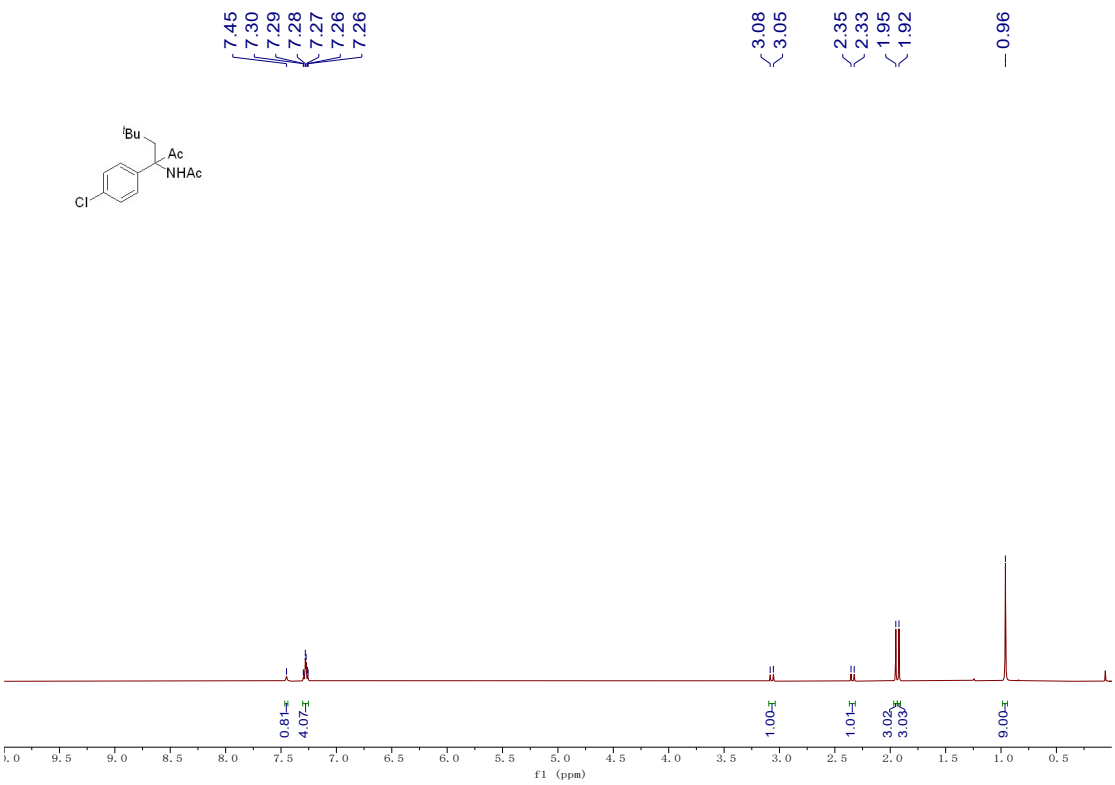
76.7 CDCl3
68.7
42.4
31.4
30.6
24.2
tBu
Ac
NHAc
f1 (ppm)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7.26
7.26
3.08
3.05
2.35
2.33
1.95
1.92
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NHAc
Cl
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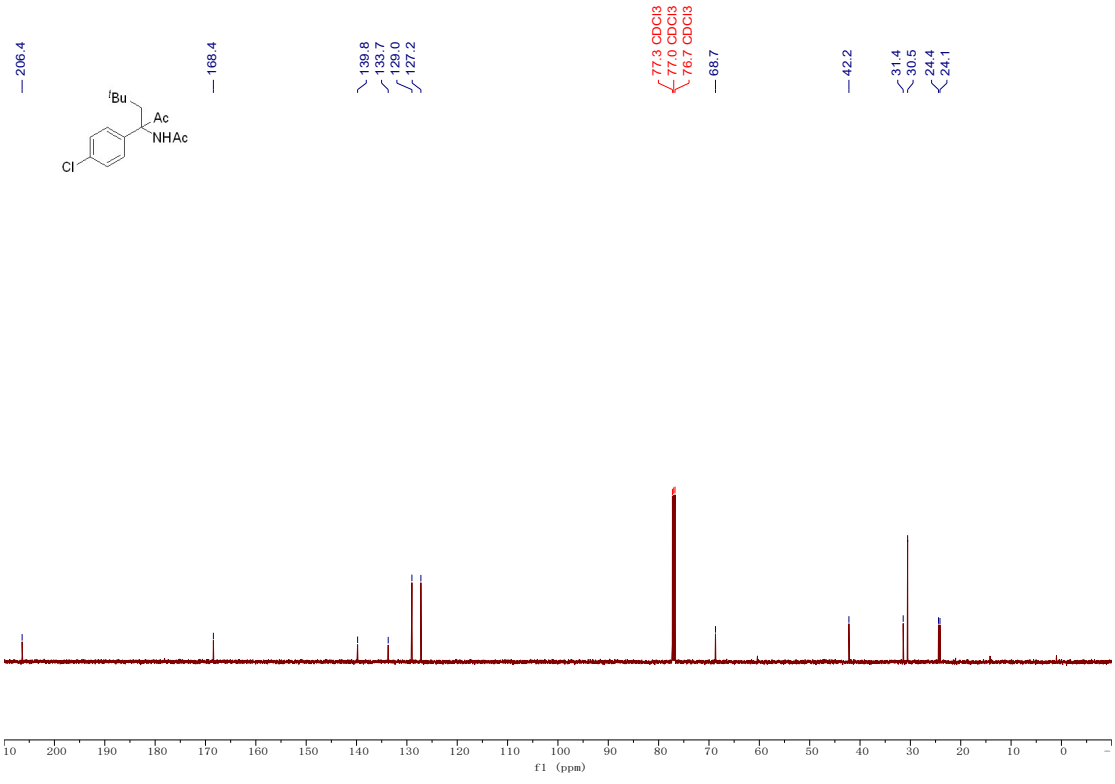
4.07
1.00
1.01
3.02
3.03
9.00
f1 (ppm)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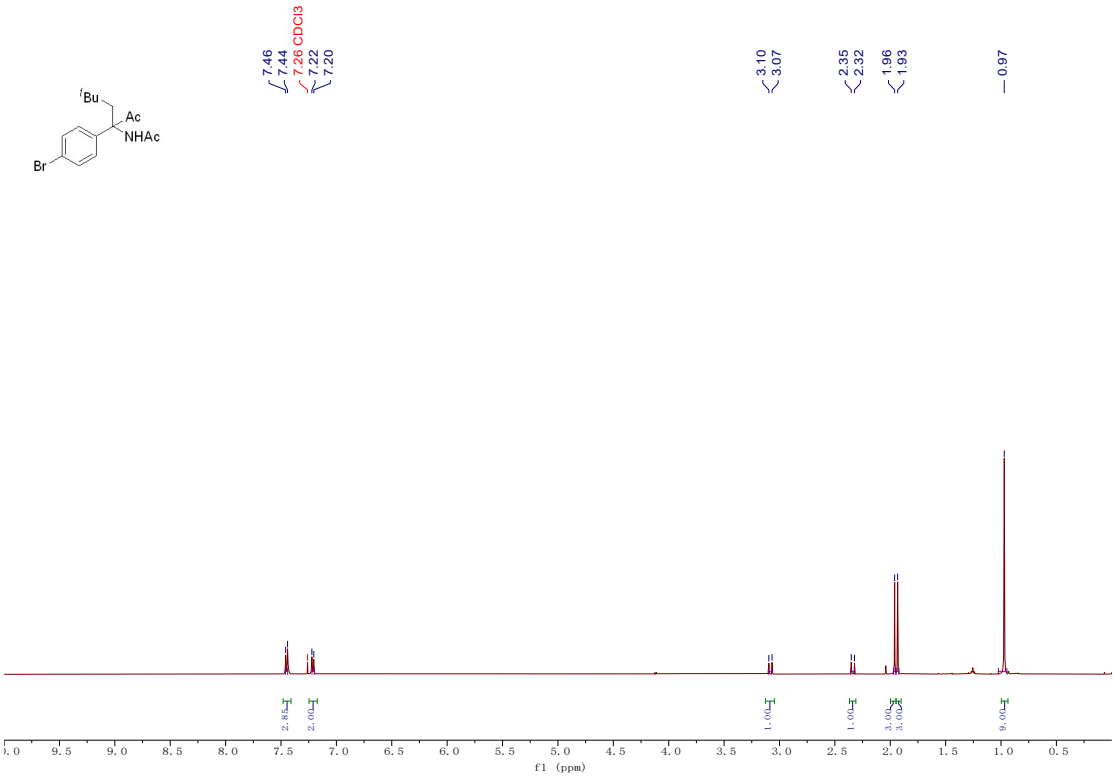
168.4
139.8
133.7
129.0
127.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.7
42.2
31.4
30.5
24.4
24.1
tBu
Ac
NHAc
Cl
f1 (ppm)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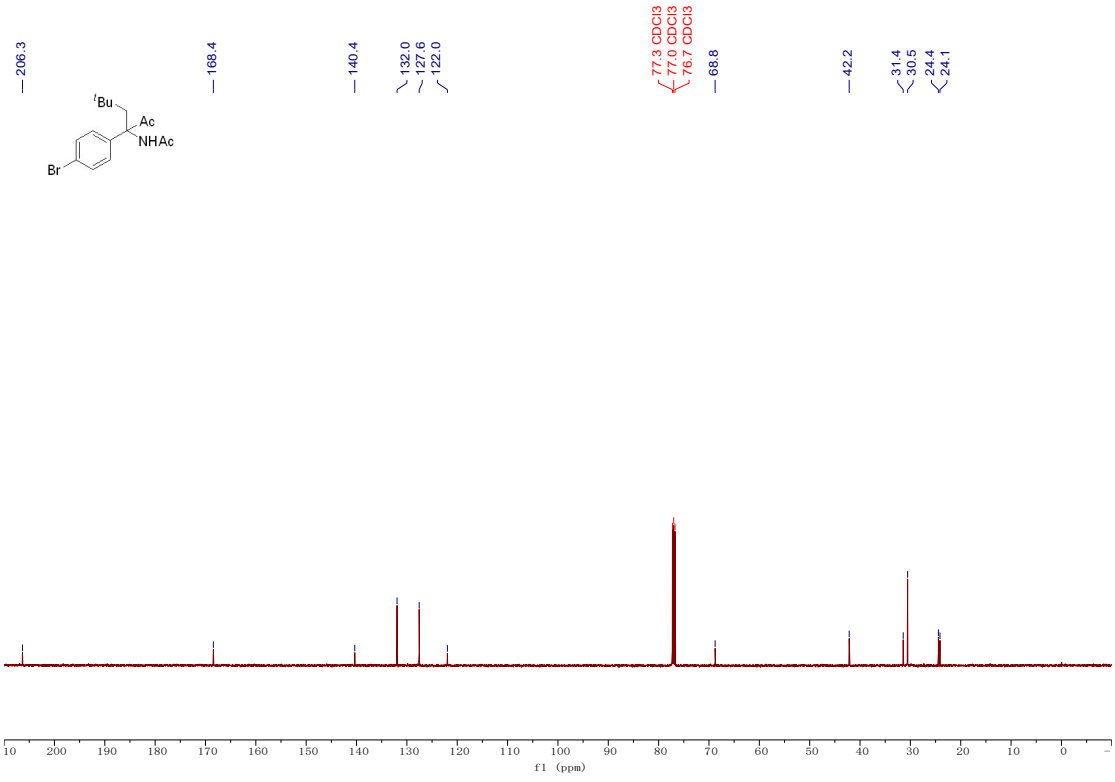
7.44
7.26 CDCl3
7.22
7.20
3.10
3.07
2.35
2.32
1.96
1.93
0.97
tBu
Ac
NHAc
Br
f1 (ppm)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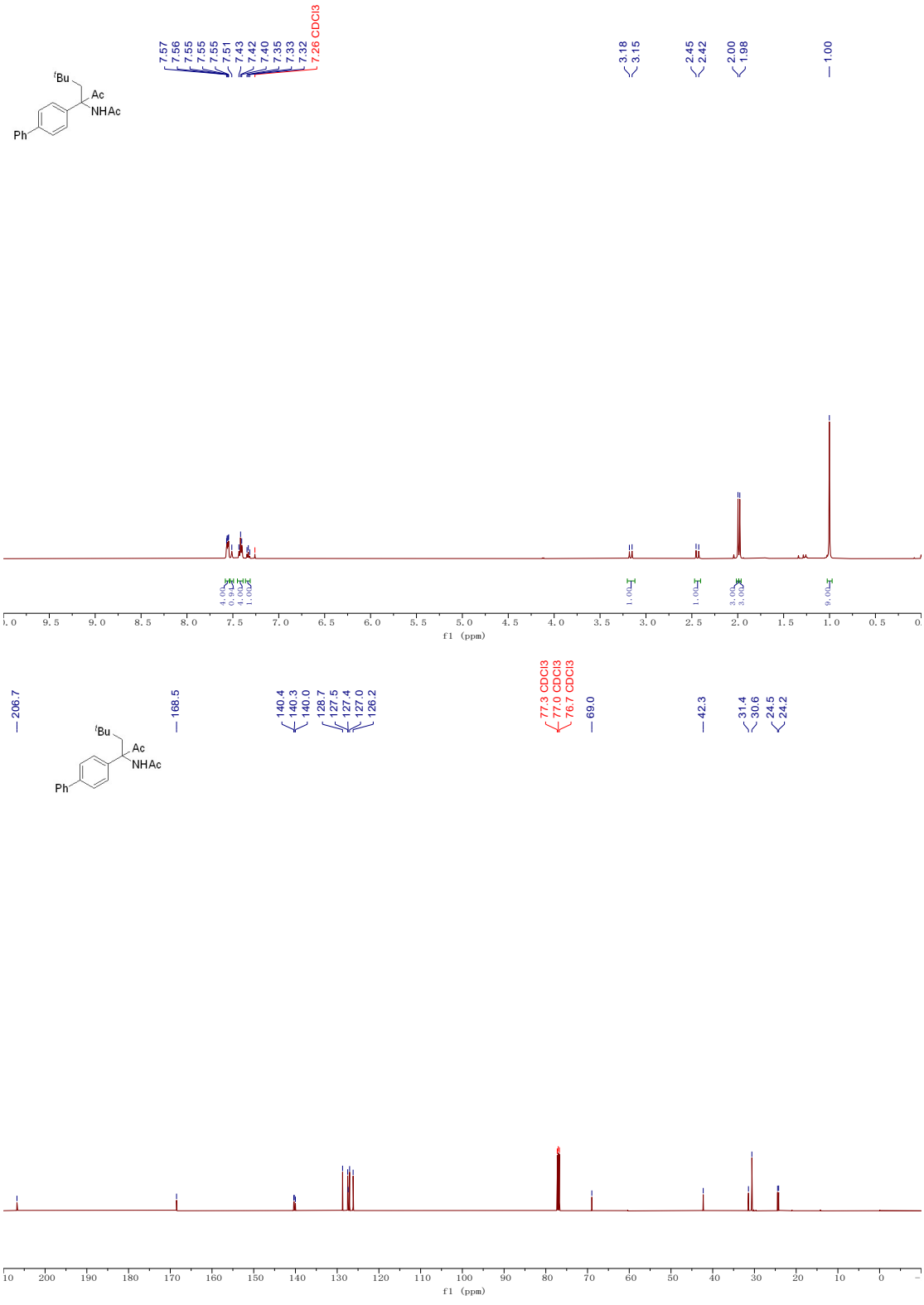
132.0
127.6
122.0
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.8
42.2
31.4
30.5
24.4
24.1
tBu
Ac
NHAc
Br
f1 (ppm)


tBu
Ac
NHAc
Ph
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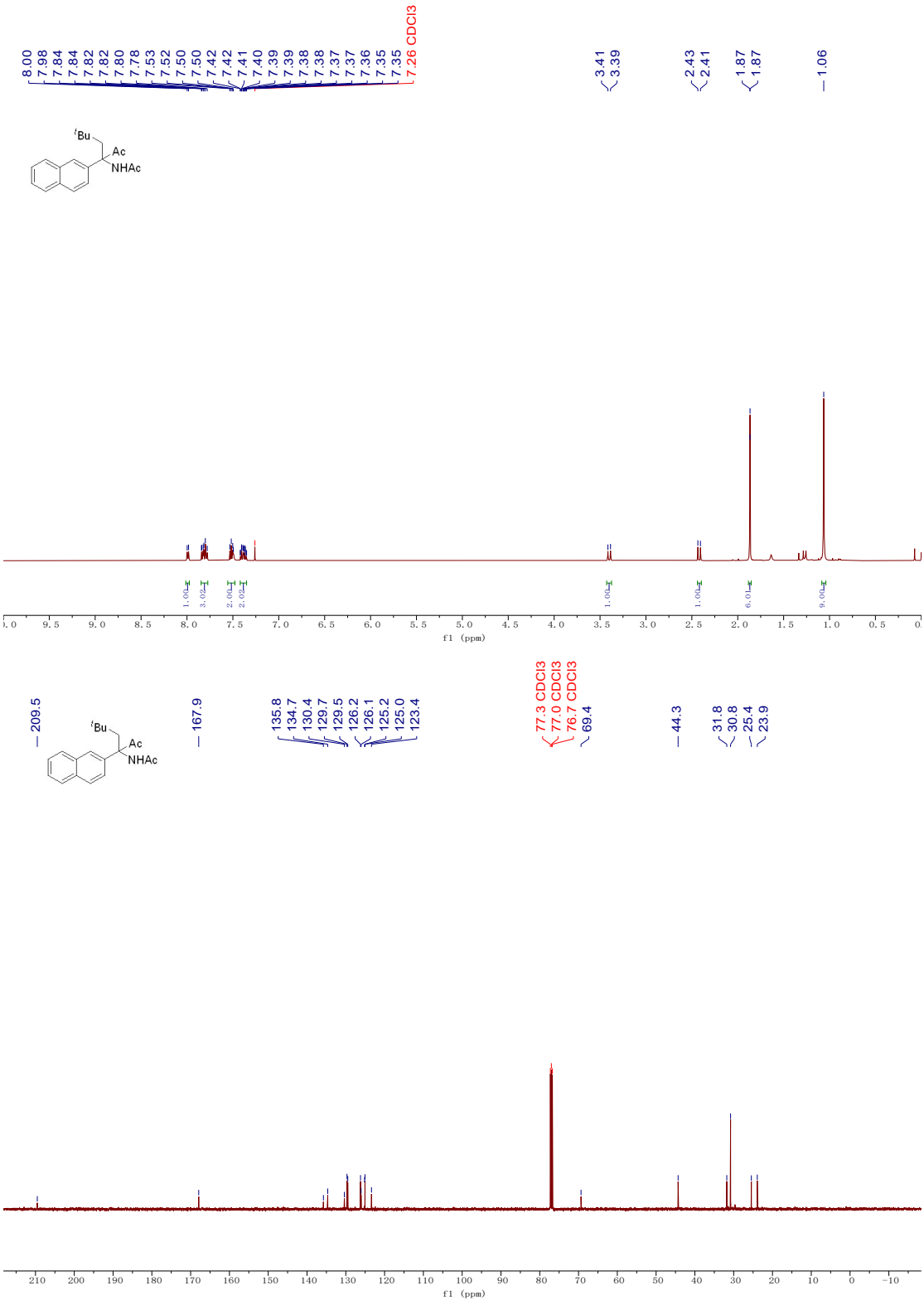
7.55
7.51
7.43
7.42
7.40
7.35
7.33
7.32
7.26 CDCl3
3.18
3.15
2.45
2.42
2.00
1.98
1.00
f1 (ppm)
206.7
168.5
140.4
140.3
140.0
128.7
127.5
127.4
127.0
126.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.0
42.3
31.4
30.6
24.5
24.2
f1 (ppm)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7.52
7.50
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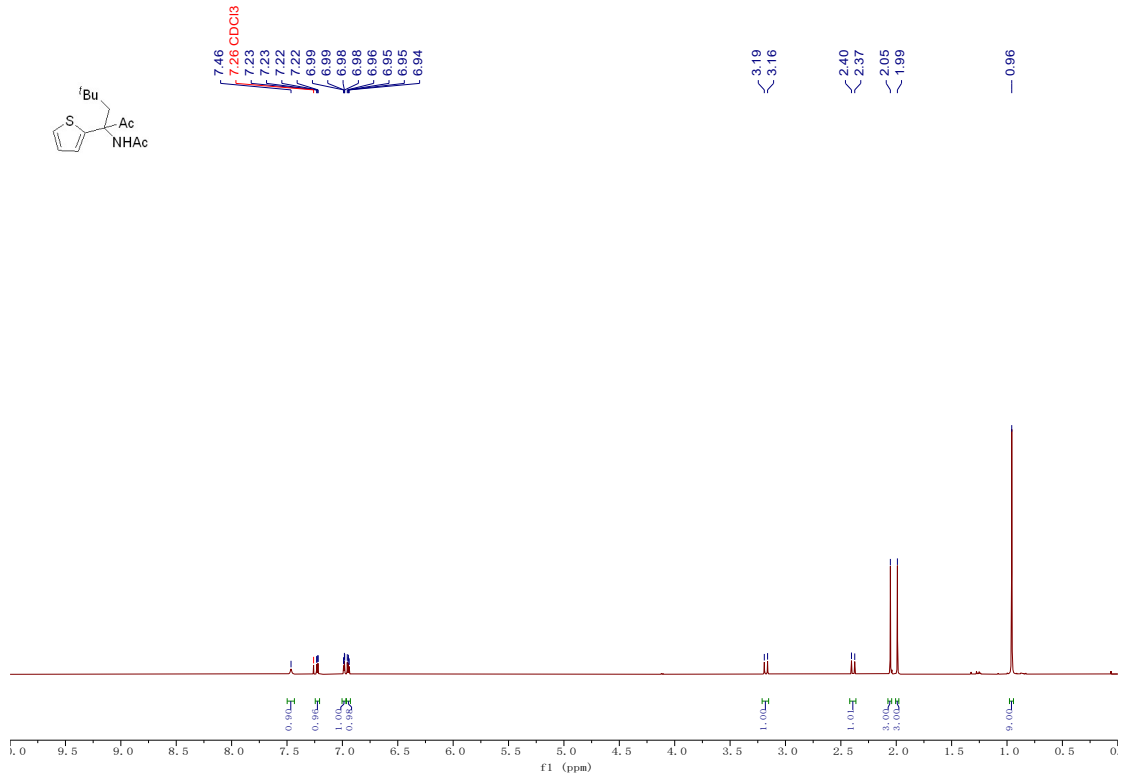
7.42
7.42
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.37
7.36
7.35
7.35
7.26 CDCl3
3.41
3.39
2.43
2.41
1.87
1.87
1.06
tBu
Ac
NHAc
1.00
3.02
2.00
2.02
1.00
1.00
6.01
9.04
f1 (ppm)
209.5
167.9
135.8
134.7
130.4
129.7
129.5
126.2
126.1
125.2
125.0
123.4
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.4
44.3
31.8
30.8
25.4
23.9
tBu
Ac
NHAc
f1 (ppm)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6.99
6.98
6.98
6.96
6.95
6.95
6.94
3.19
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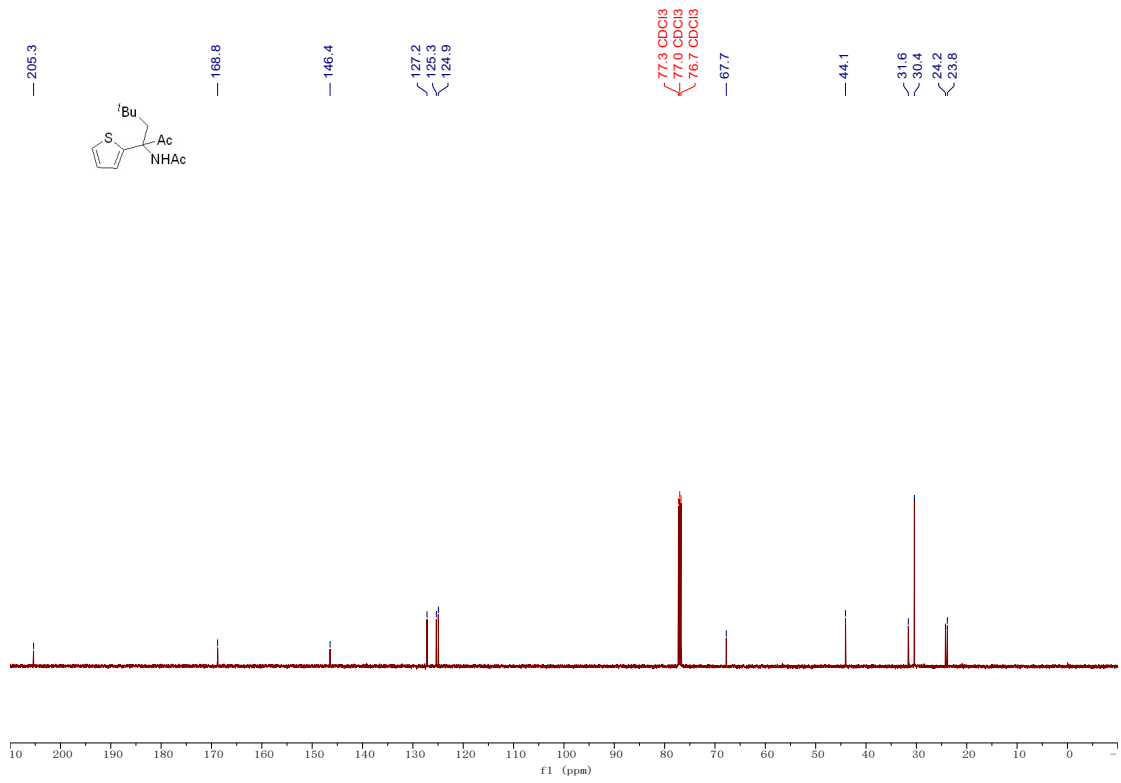
2.40
2.37
2.05
1.99
0.96
tBu
S
Ac
NHAc
f1 (ppm)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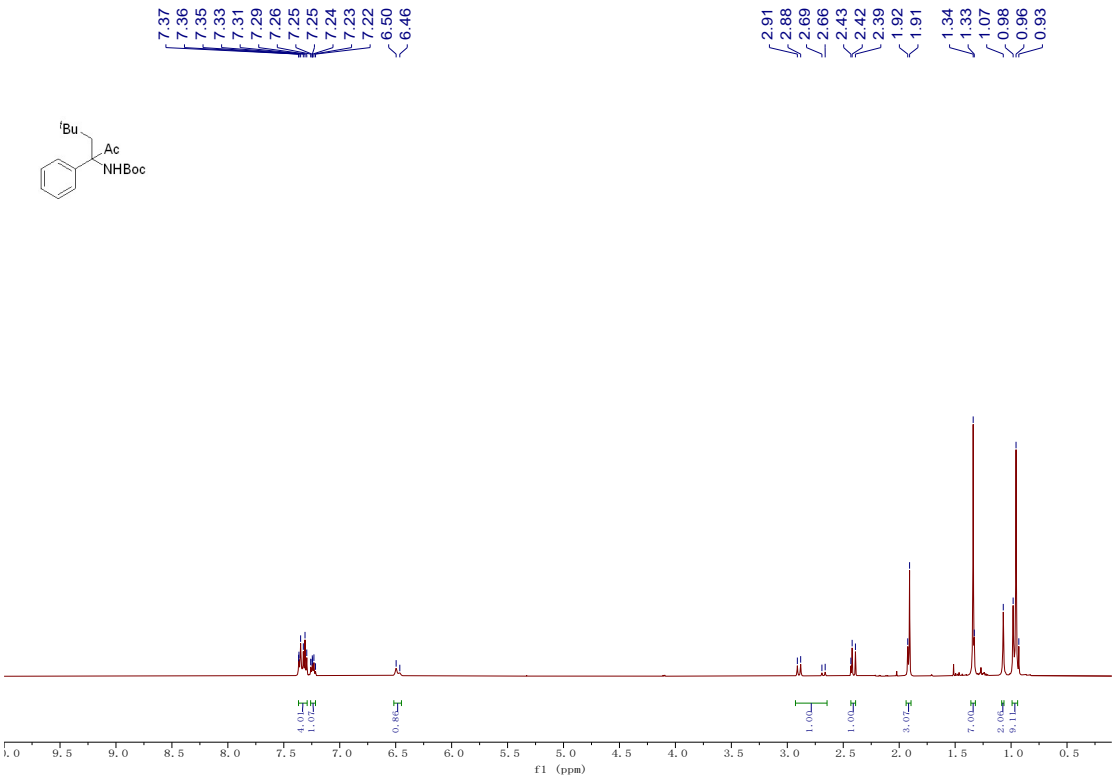
146.4
127.2
125.3
124.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.7
44.1
31.6
30.4
24.2
23.8
tBu
S
Ac
NHAc
f1 (ppm)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7.25
7.25
7.24
7.23
7.22
6.50
6.46
2.91
2.88
2.69
2.66
2.43
2.42
2.39
1.92
1.91
1.34
1.33
1.07
0.98
0.96
0.93
tBu
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NHBoc
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1.07
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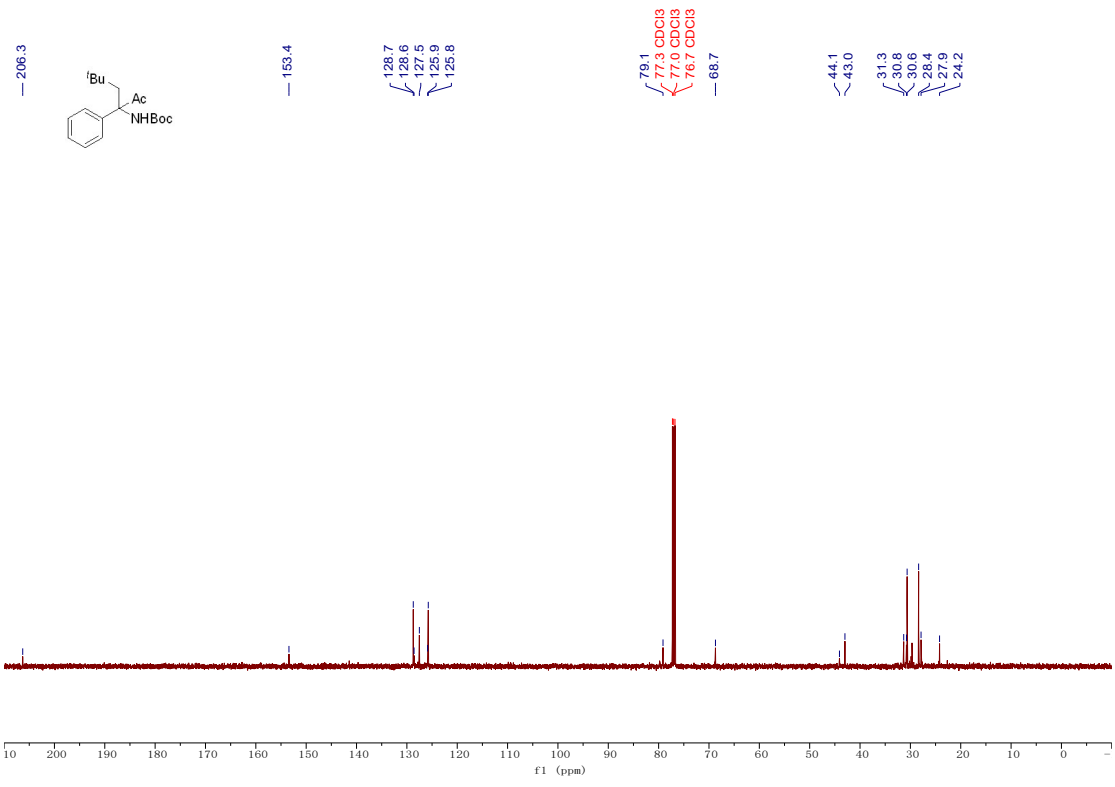
1.00
1.00
3.07
7.04
2.04
9.11
f1 (ppm)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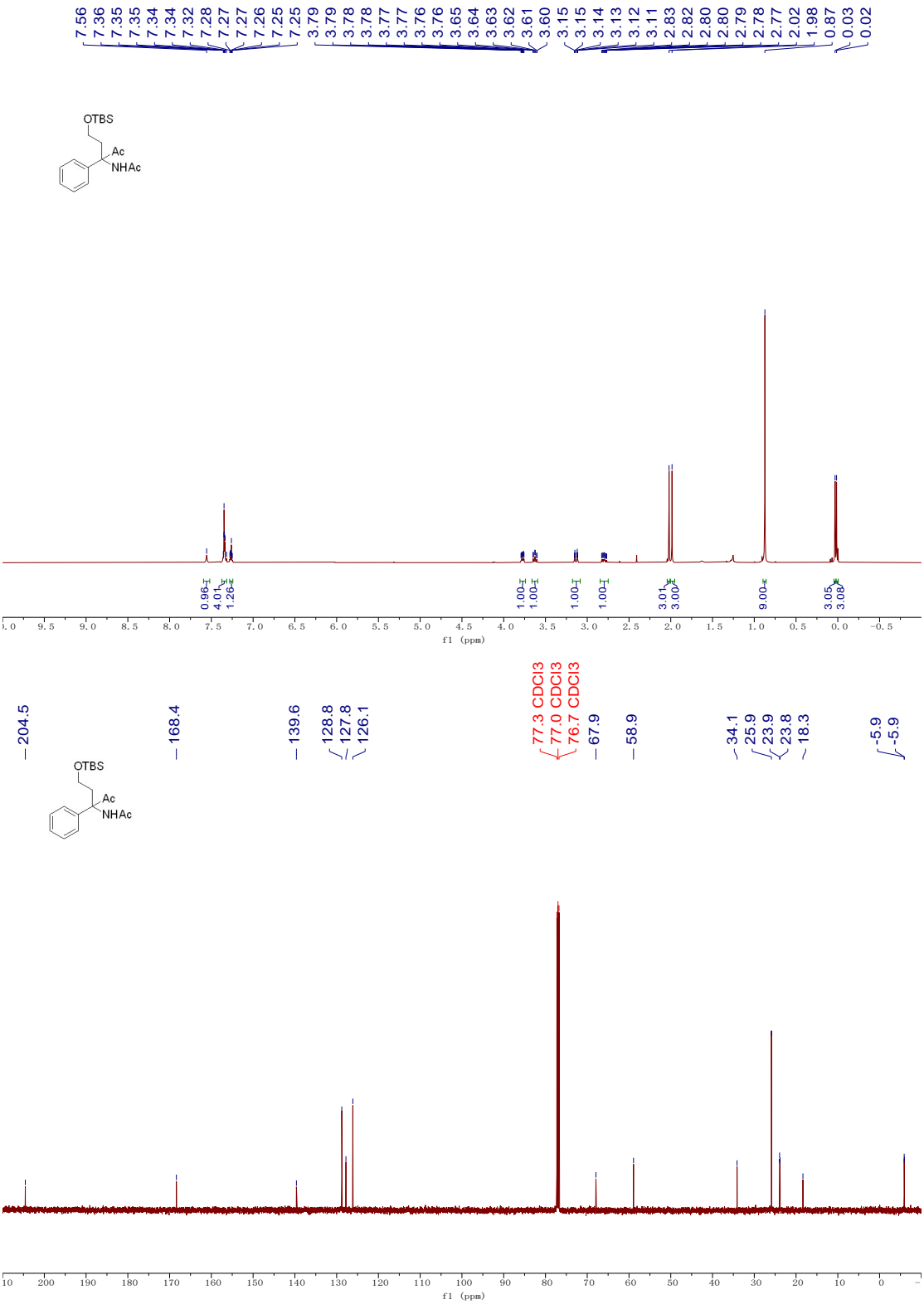
128.7
128.6
127.5
125.9
125.8
79.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.7
44.1
43.0
31.3
30.8
30.6
28.4
27.9
24.2
tBu
Ac
NHBoc
f1 (ppm)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7.25
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3.79
3.78
3.78
3.77
3.77
3.76
3.76
3.65
3.64
3.63
3.62
3.61
3.60
3.15
3.15
3.14
3.13
3.12
3.11
2.83
2.82
2.80
2.80
2.79
2.78
2.77
2.02
1.98
0.87
0.03
0.02
OTBS
Ac
NHAc
0.96
4.01
1.26
1.00
1.00
1.00
1.00
3.01
3.00
9.00
3.05
3.08
f1 (ppm)
204.5
168.4
139.6
128.8
127.8
126.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.9
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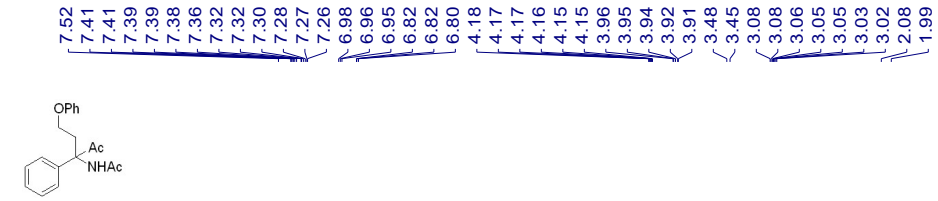
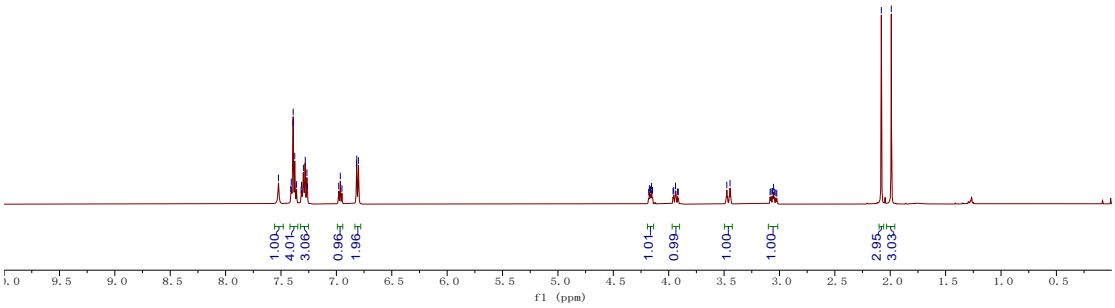
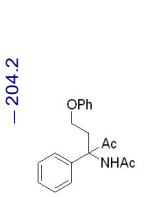
25.9
23.9
23.8
18.3
-5.9
-5.9
OTBS
Ac
NHAc
f1 (ppm)







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139.2
129.5
128.9
128.0
126.0
121.1
114.0
77.3 CDCl3
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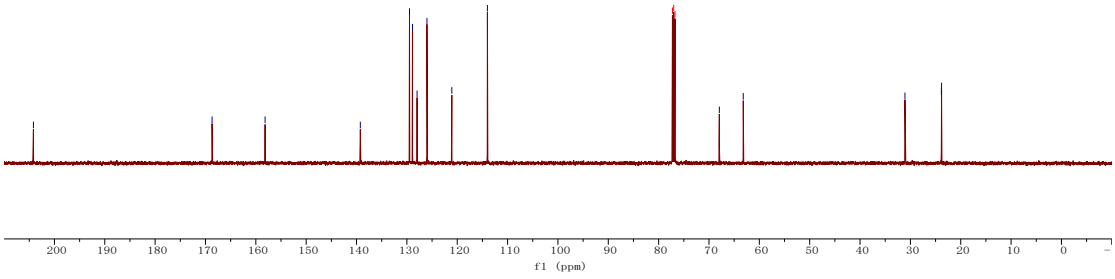
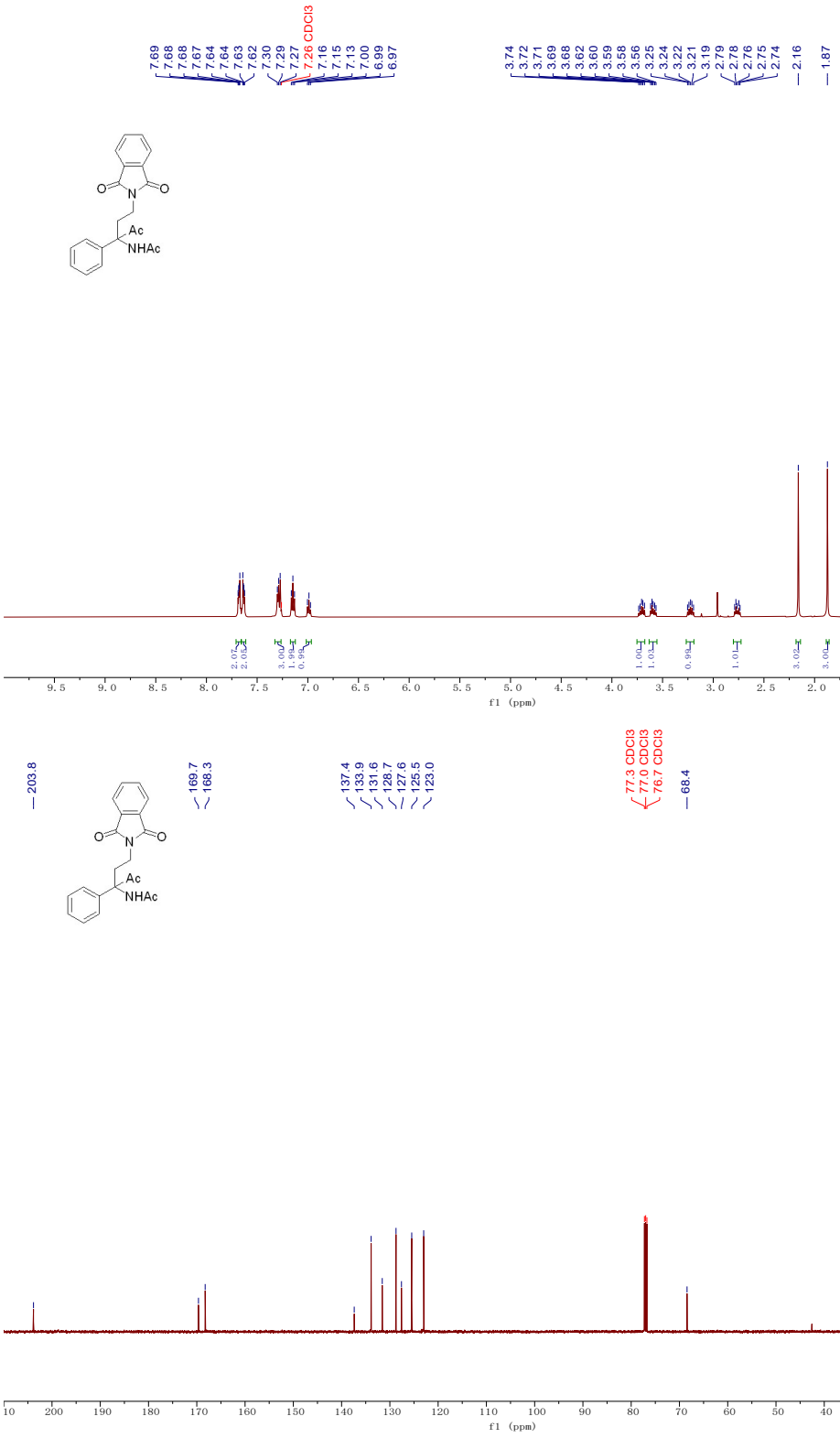
67.9
63.2
31.1
23.8
23.8

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7.62
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7.29
7.27
7.26 CDCl3
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7.15
7.13
7.00
6.99
6.97
3.74
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3.71
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3.68
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3.59
3.58
3.56
3.25
3.24
3.22
3.21
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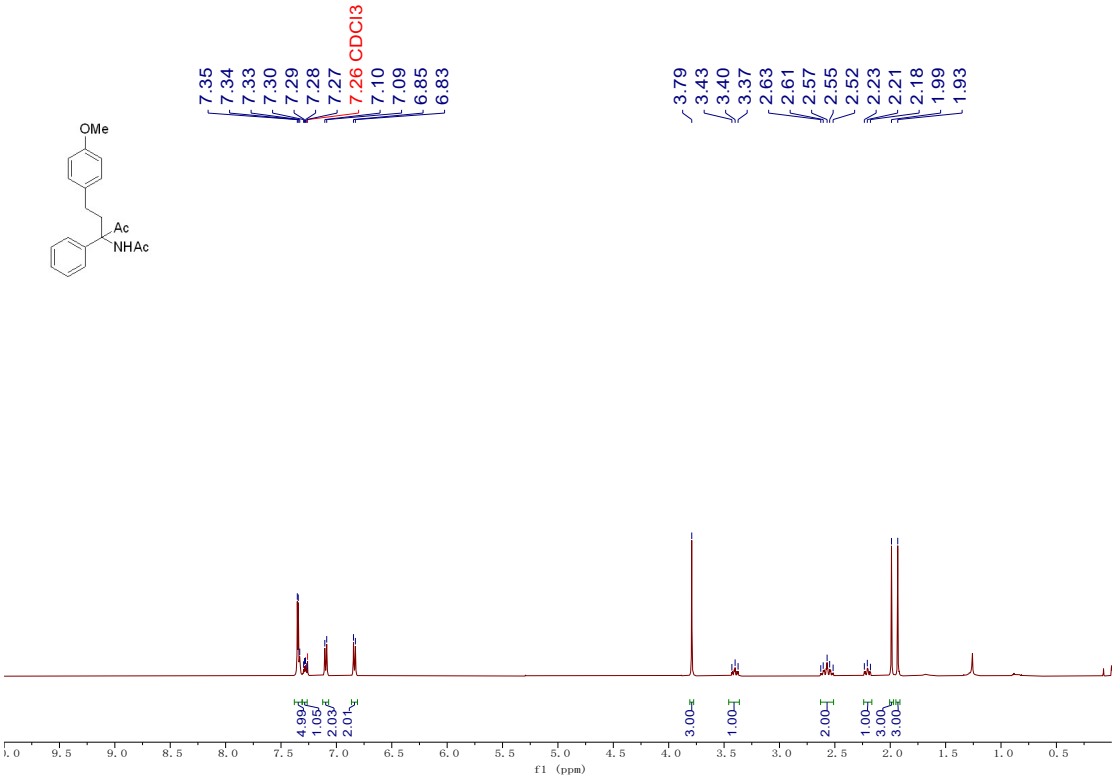
2.79
2.78
2.76
2.75
2.74
2.16
1.87
O
N
O
Ac
NHAc
2.07
2.02
3.00
1.94
0.99
1.04
1.03
0.94
1.01
3.02
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
f1 (ppm)
203.8
169.7
168.3
137.4
133.9
131.6
128.7
127.6
125.5
123.0
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.4
33.5
29.8
23.4
23.1
O
N
O
Ac
NHAc
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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7.34
7.33
7.30
7.29
7.28
7.27
7.26 CDCl3
7.10
7.09
6.85
6.83
3.79
3.43
3.40
3.37
2.63
2.61
2.57
2.55
2.52
2.23
2.21
2.18
1.99
1.93
OMe
Ac
NHAc
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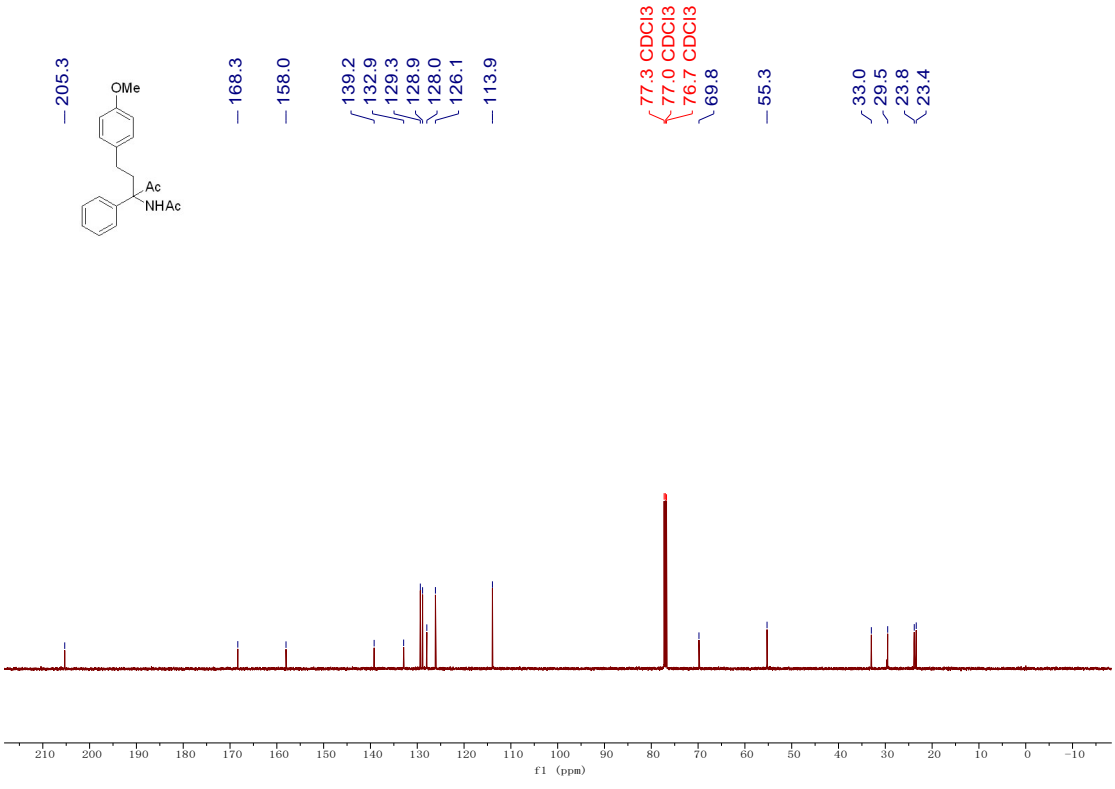
2.03
2.01
3.00
1.00
2.00
1.00
3.00
3.00
f1 (ppm)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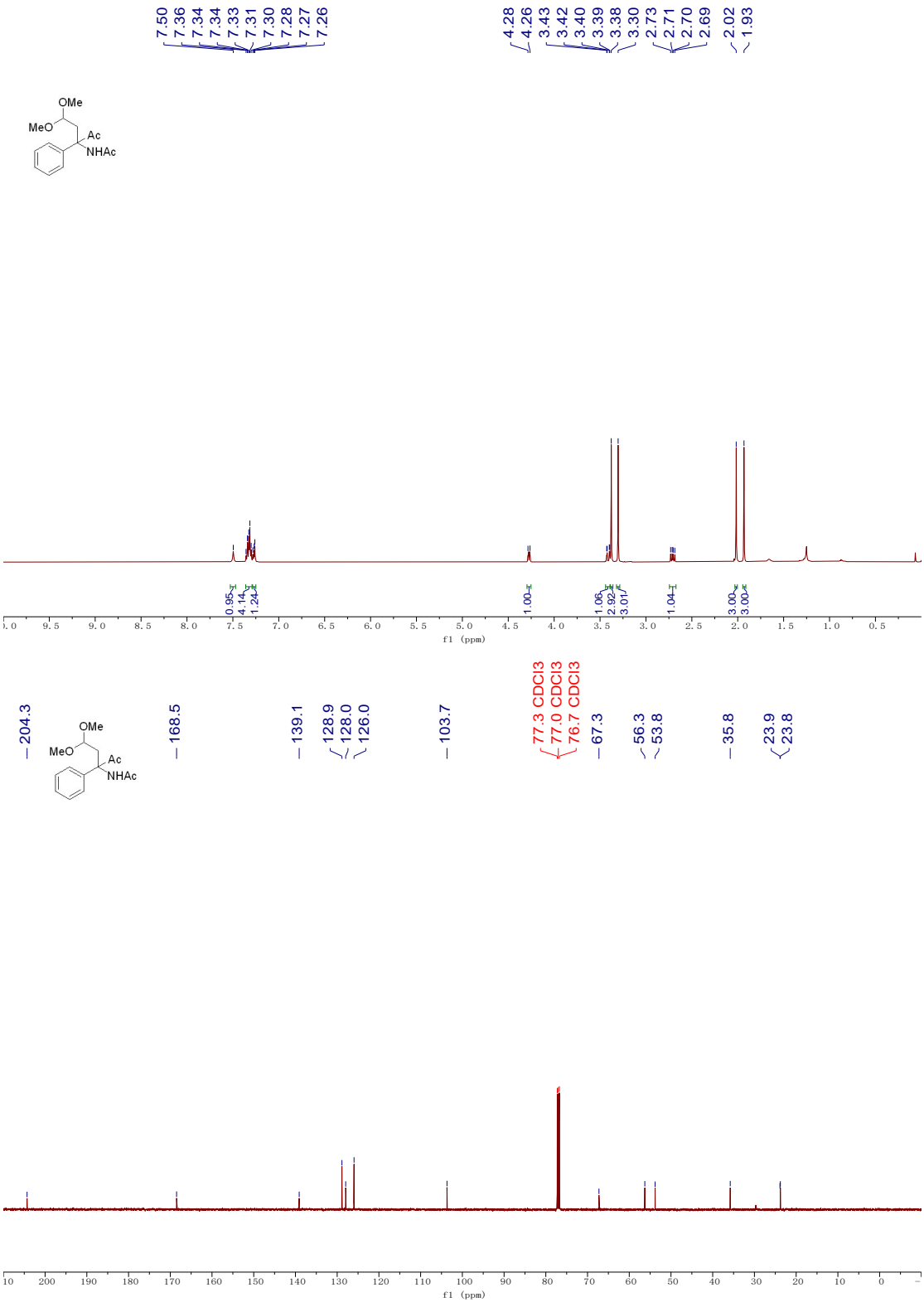
139.2
132.9
129.3
128.9
128.0
126.1
113.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.8
55.3
33.0
29.5
23.8
23.4
OMe
Ac
NHAc
f1 (ppm)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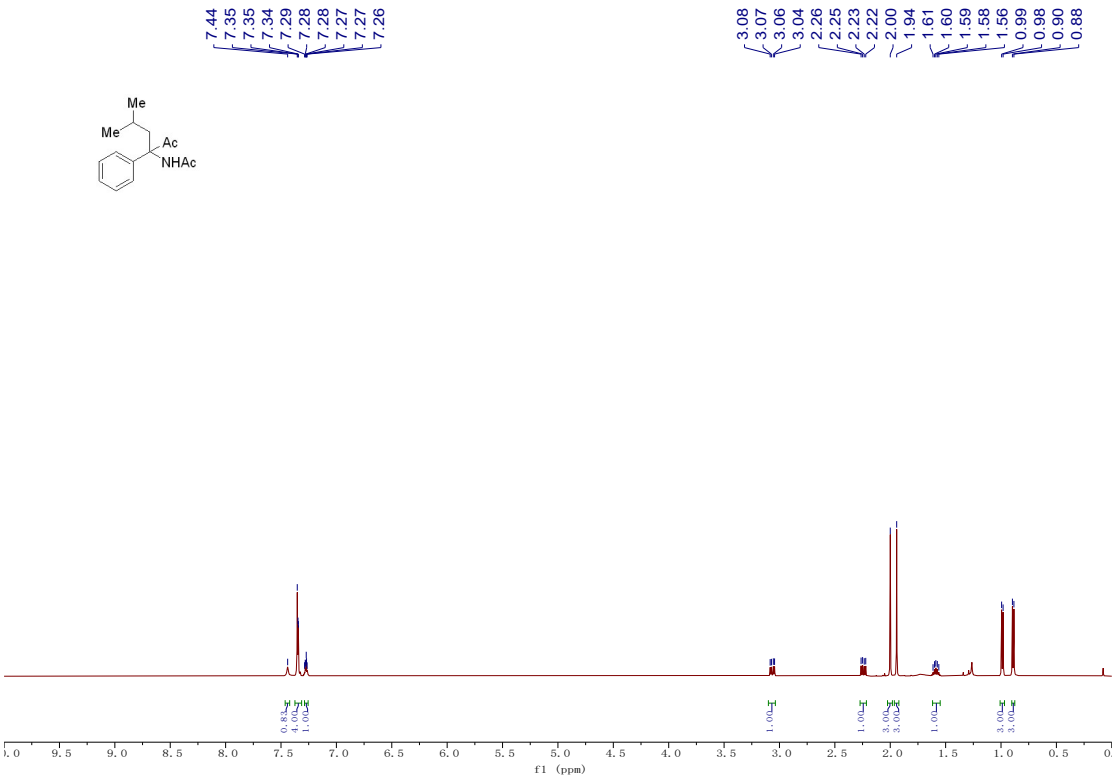
7.28
7.27
7.26
4.28
4.26
3.43
3.42
3.40
3.39
3.38
3.30
2.73
2.71
2.70
2.69
2.02
1.93
OMe
MeO
Ac
NHAc
0.95
4.14
1.24
1.00
1.06
2.92
3.01
1.04
3.00
3.00
f1 (ppm)
204.3
168.5
139.1
128.9
128.0
126.0
103.7
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.3
56.3
53.8
35.8
23.9
23.8
OMe
MeO
Ac
NHAc
f1 (ppm)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7.34
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7.28
7.27
7.27
7.26
3.08
3.07
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3.04
2.26
2.25
2.23
2.22
2.00
1.94
1.61
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1.59
1.58
1.56
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0.98
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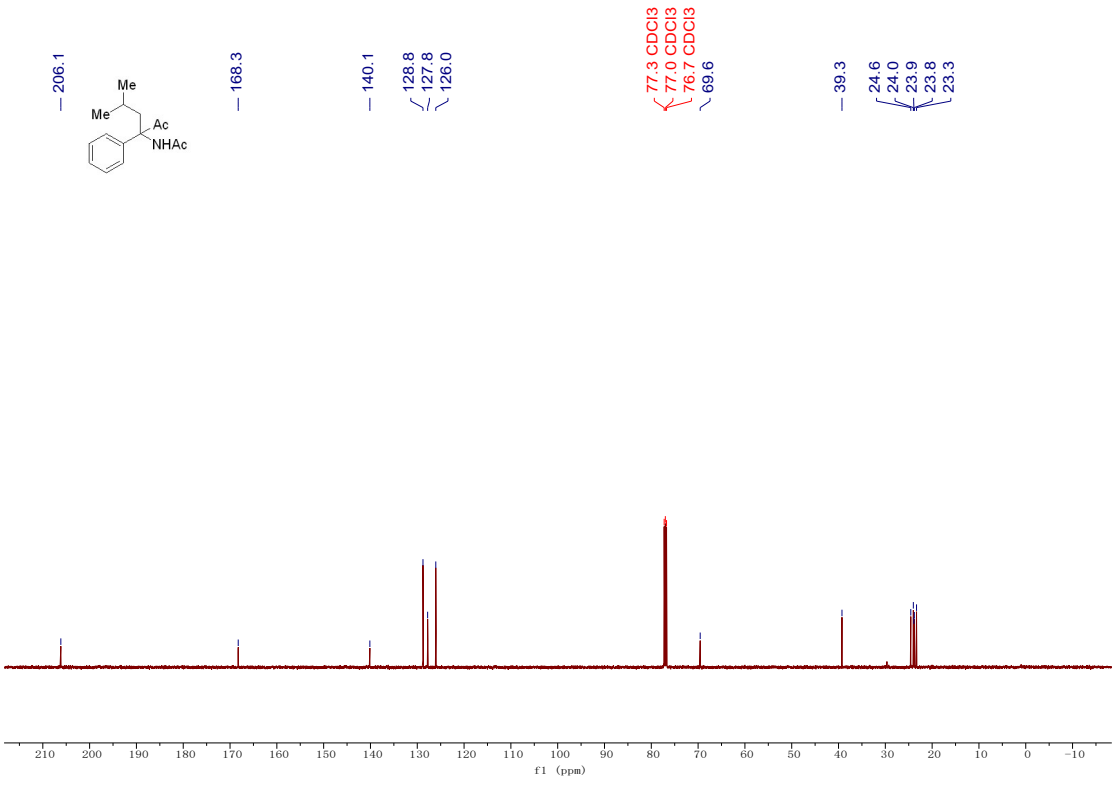
0.88
Me
Me
Ac
NHAc
f1 (ppm)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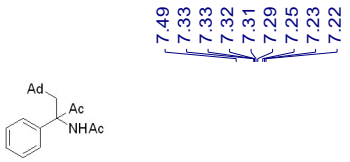
168.3
140.1
128.8
127.8
126.0
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.6
39.3
24.6
24.0
23.9
23.8
23.3
Me
Me
Ac
NHAc
f1 (ppm)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7.29
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7.23
7.22
3.04
3.01
2.22
2.19
1.96
1.92
1.91
1.90
1.68
1.65
1.62
1.58
1.55
Ad
Ac
NHAc
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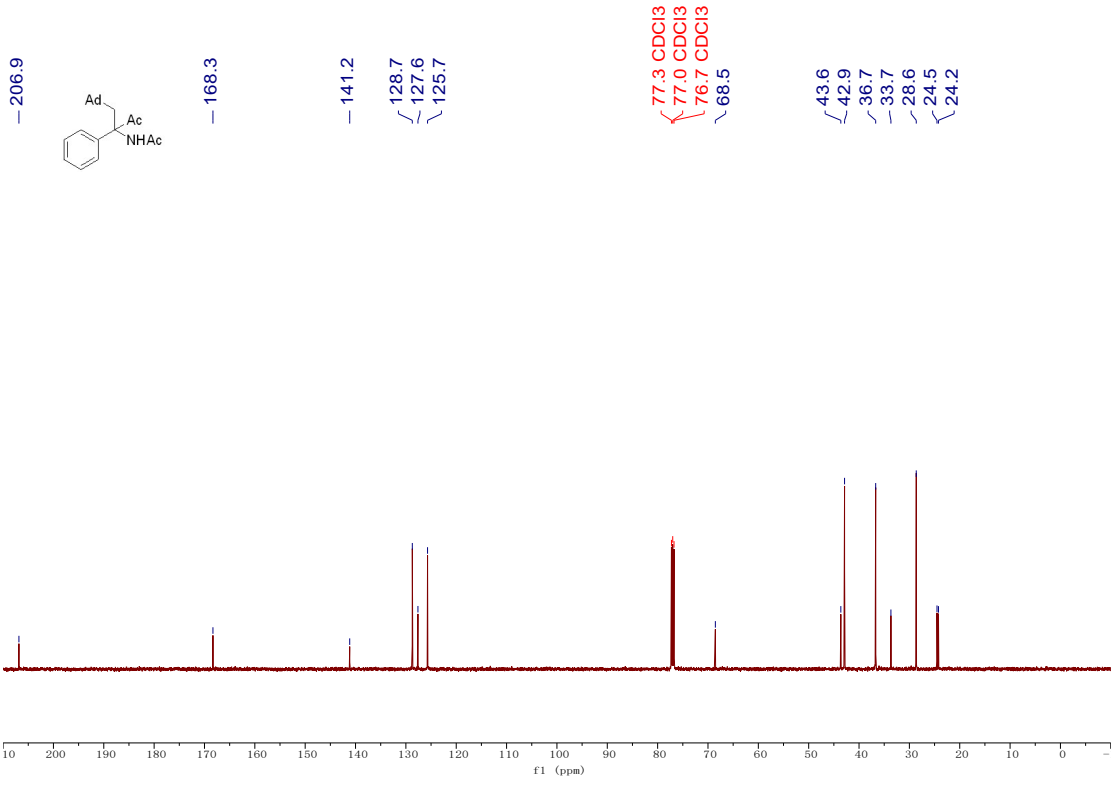
1.04
1.00
1.07
3.00
6.00
13.00
f1 (ppm)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168.3
141.2
128.7
127.6
125.7
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
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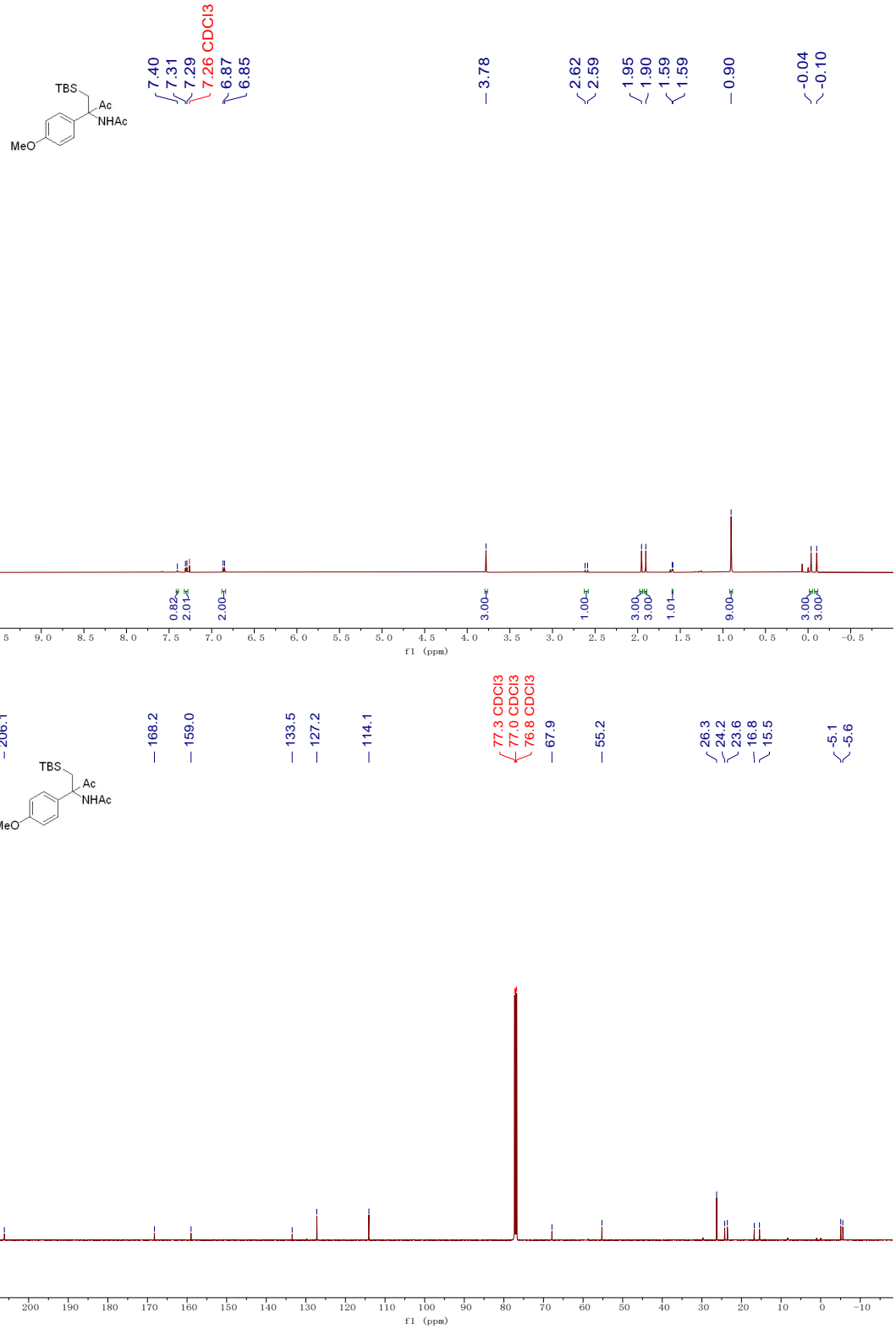
42.9
36.7
33.7
28.6
24.5
24.2
Ad
Ac
NHAc
f1 (ppm)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Ac
NHAc
MeO
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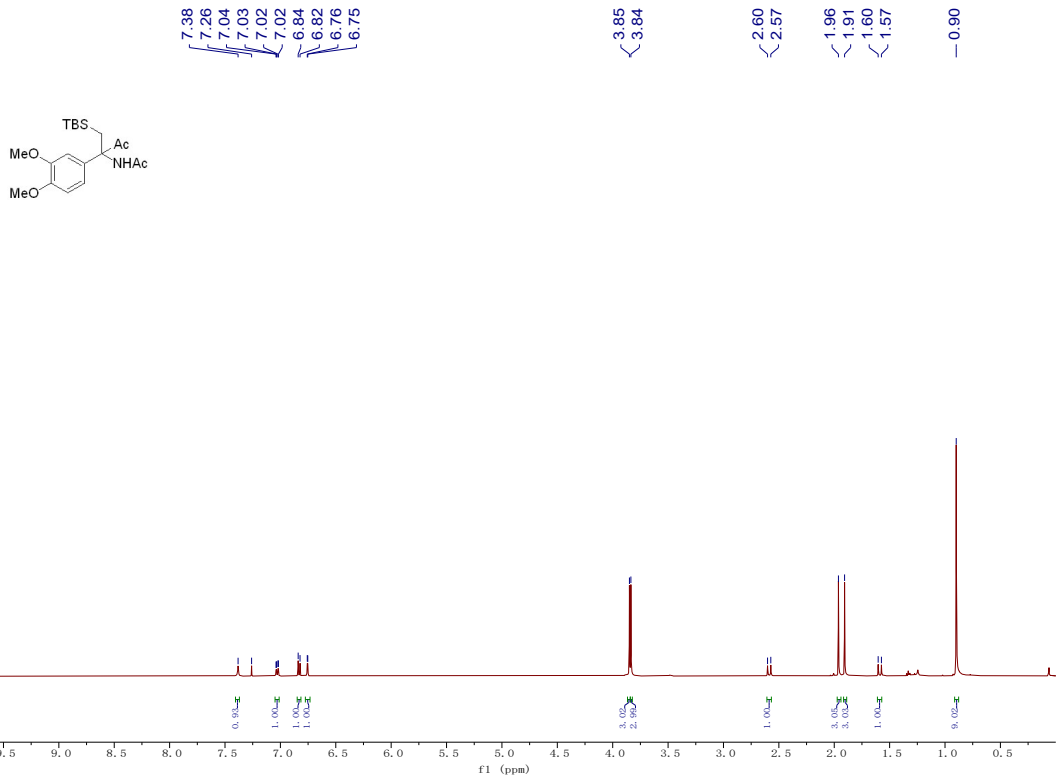
7.29
7.26 CDCl3
6.87
6.85
3.78
2.62
2.59
1.95
1.90
1.59
1.59
0.90
-0.04
-0.10
0.82
2.01
2.00
3.00
1.00
3.00
3.00
1.01
9.00
3.00
3.00
f1 (ppm)
206.1
168.2
159.0
133.5
127.2
114.1
77.3 CDCl3
77.0 CDCl3
76.8 CDCl3
67.9
55.2
26.3
24.2
23.6
16.8
15.5
-5.1
-5.6
f1 (ppm)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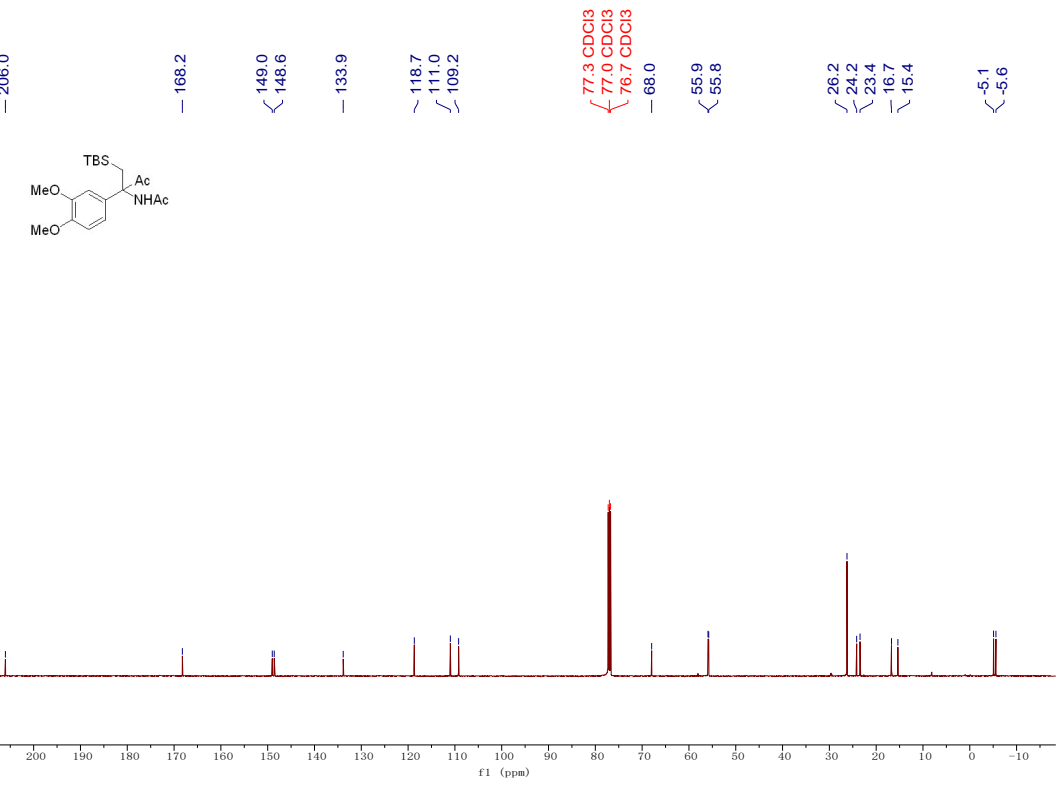
7.02
7.02
6.84
6.82
6.76
6.75
3.85
3.84
2.60
2.57
1.96
1.91
1.60
1.57
0.90
TBS
Ac
NHAc
MeO
MeO
f1 (ppm)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168.2
149.0
148.6
133.9
118.7
111.0
109.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.0
55.9
55.8
26.2
24.2
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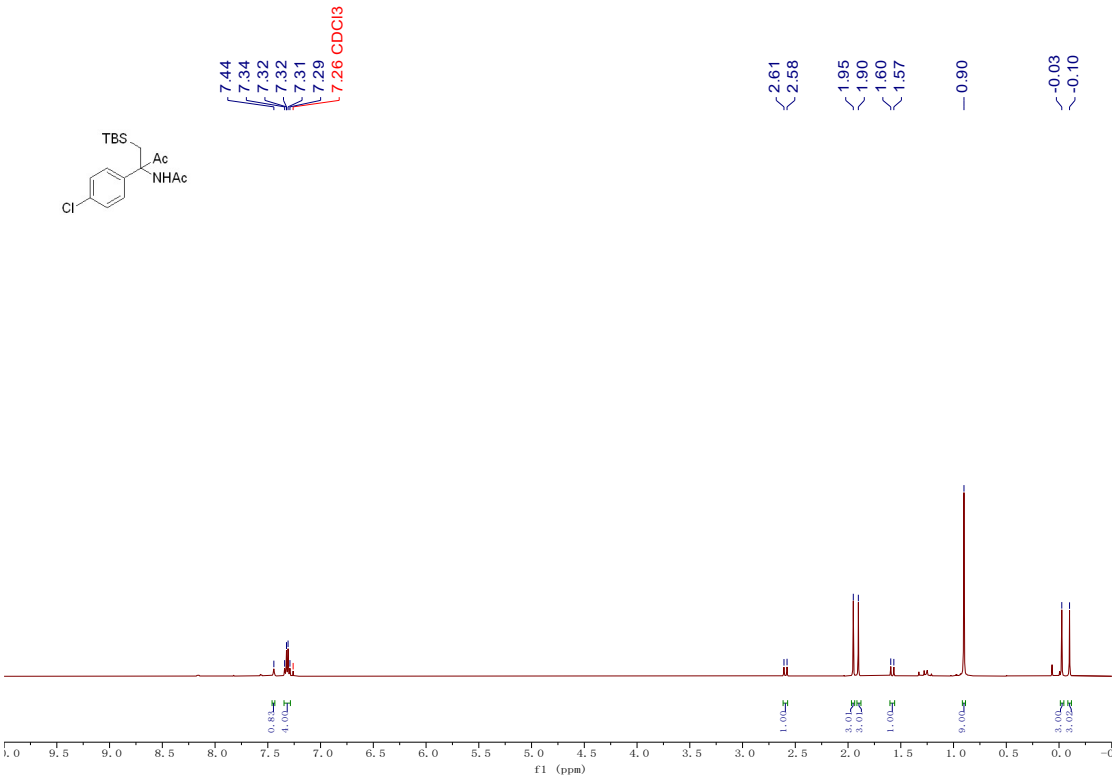
16.7
15.4
-5.1
-5.6
TBS
Ac
NHAc
MeO
MeO
f1 (ppm)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7.34
7.32
7.32
7.31
7.29
7.26 CDCl3
2.61
2.58
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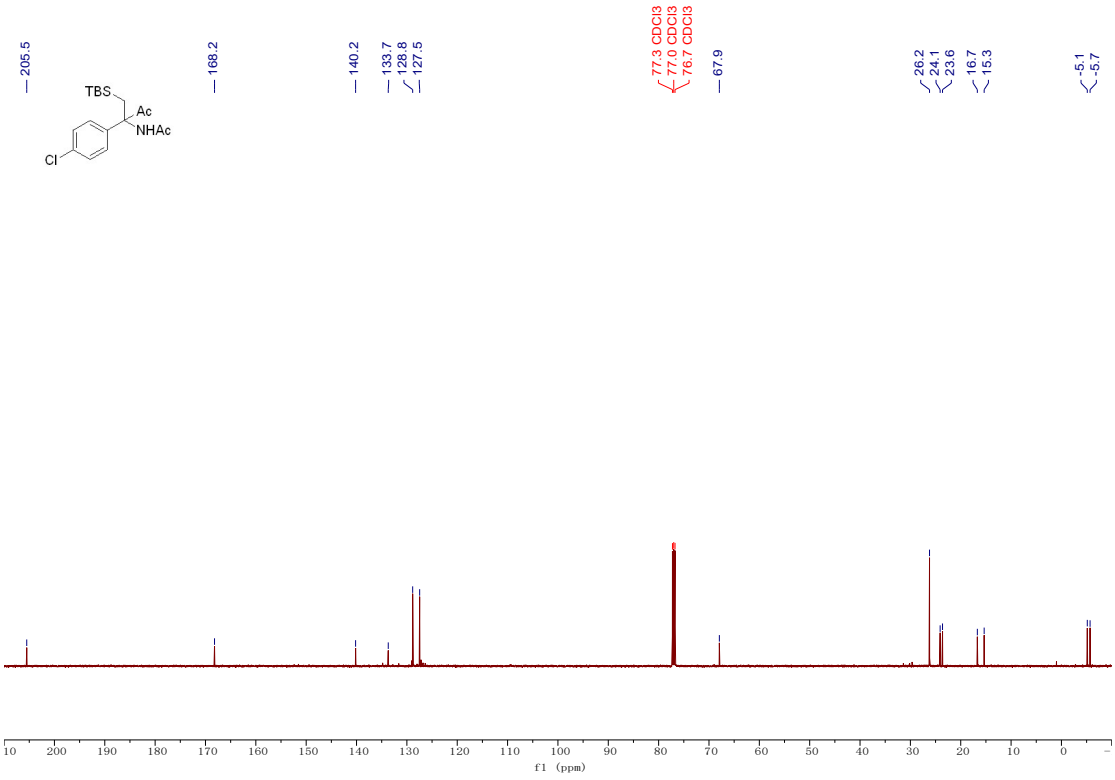
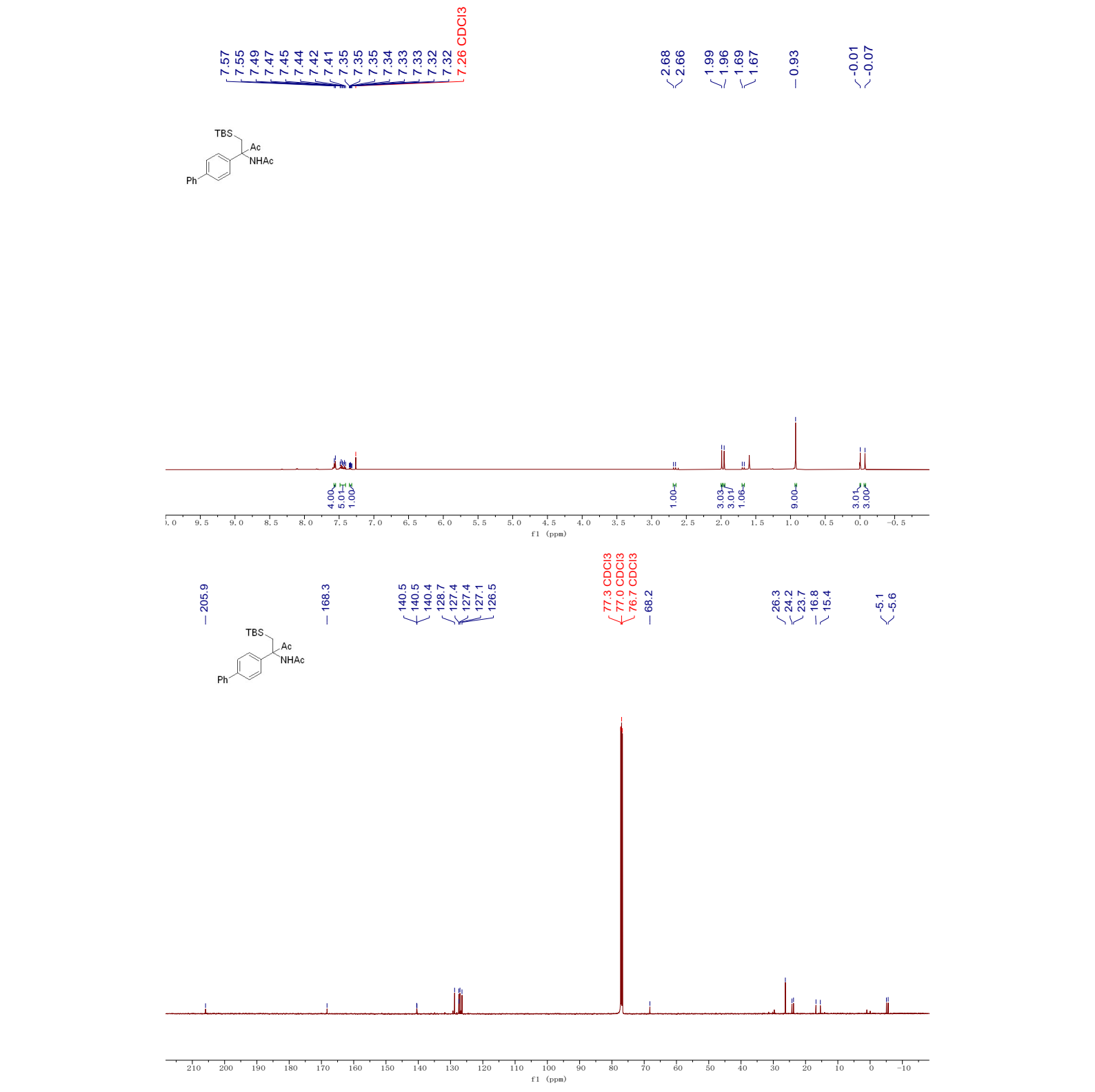
1.60
1.57
0.90
-0.03
-0.10
TBS
Ac
NHAc
Cl
f1 (ppm)


205.5
168.2
140.2
133.7
128.8
127.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.9
26.2
24.1
23.6
16.7
15.3
-5.1
-5.7
TBS
Ac
NHAc
Cl
f1 (ppm)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7.44
7.42
7.41
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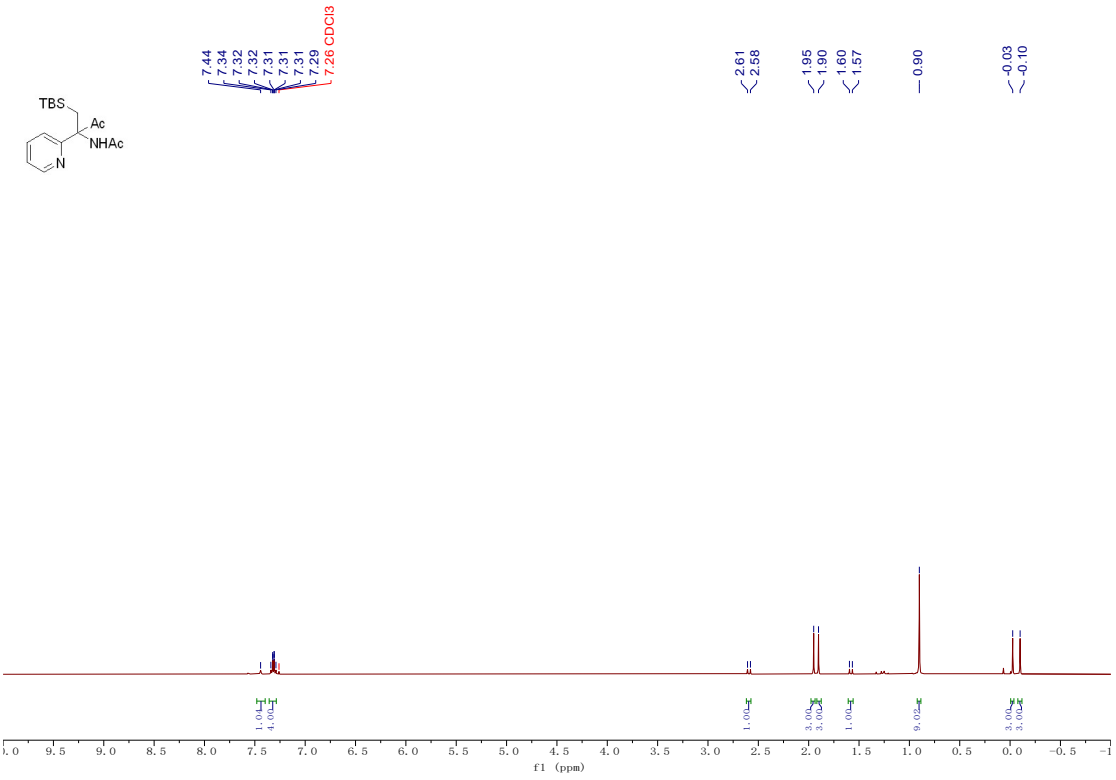
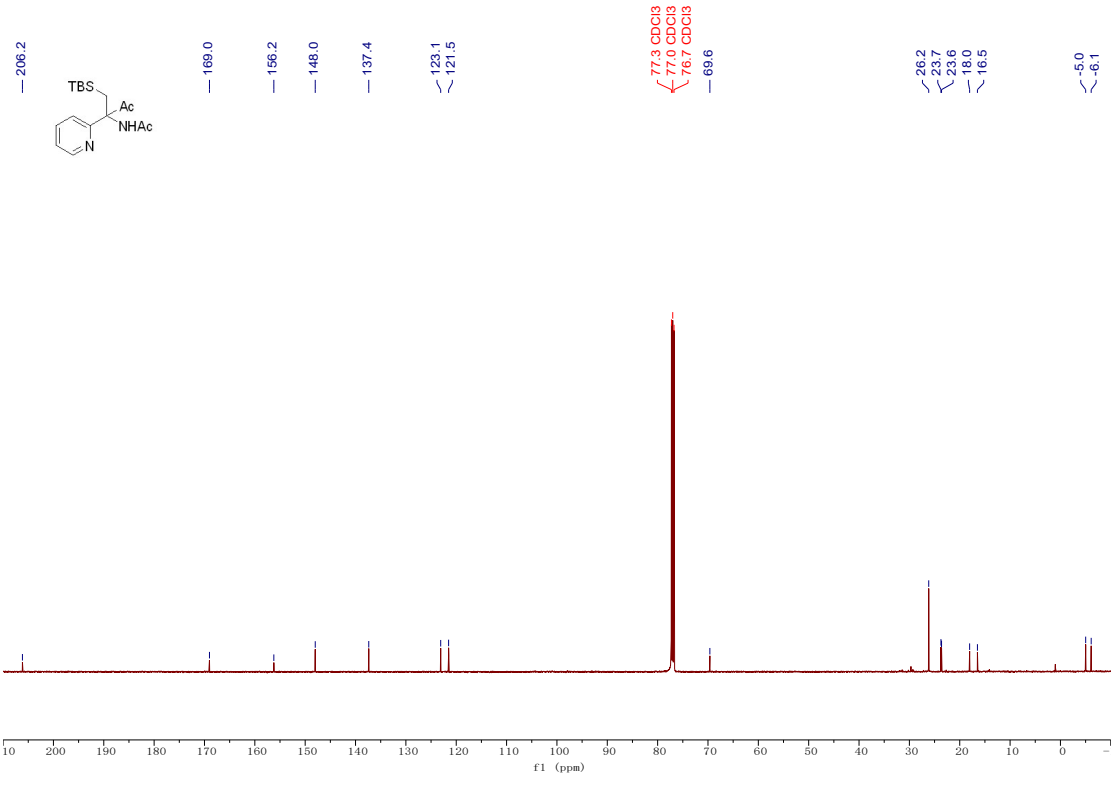
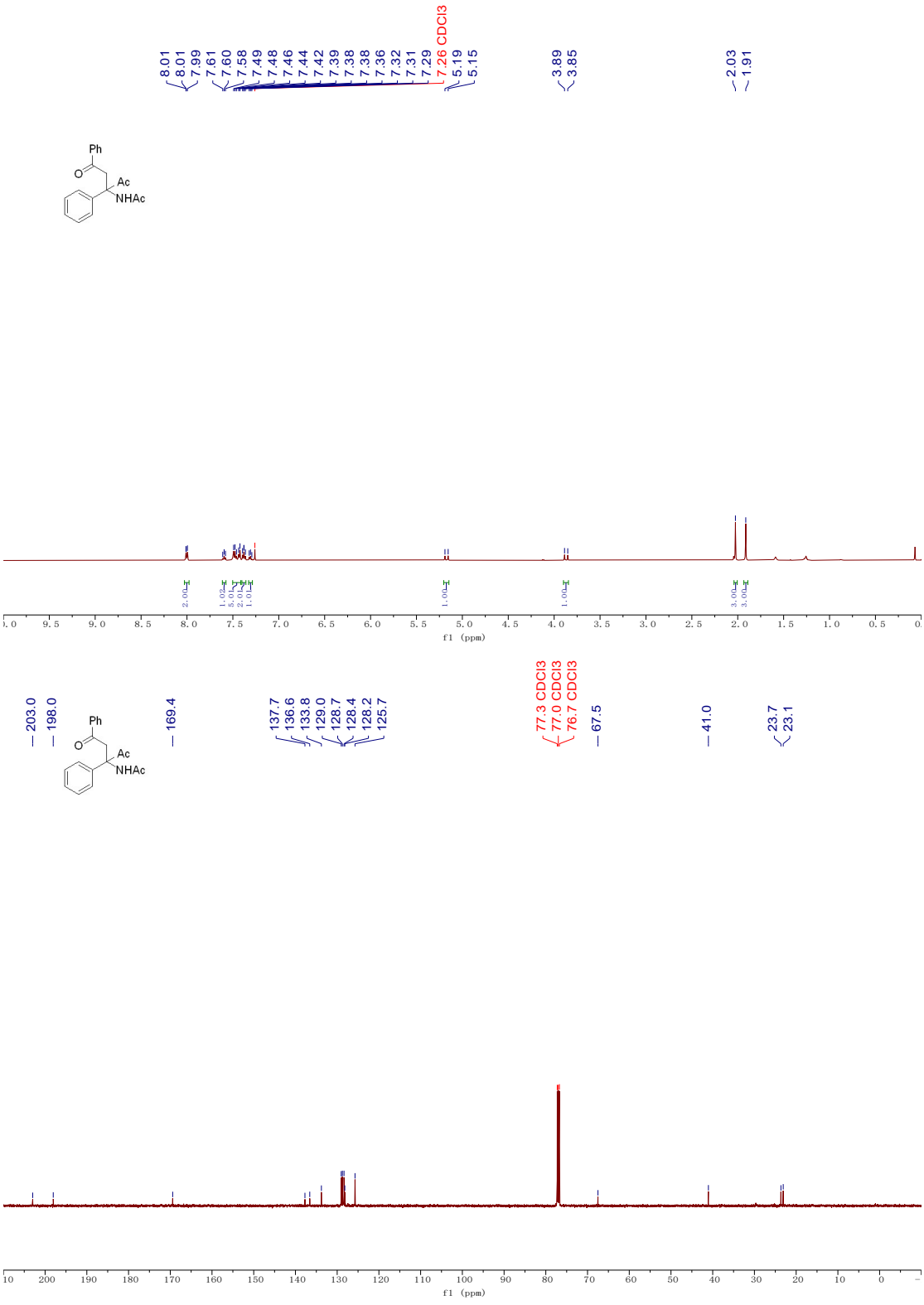
7.35
7.35
7.34
7.33
7.33
7.32
7.32
7.26 CDCl3
2.68
2.66
1.99
1.96
1.69
1.67
0.93
-0.01
-0.07
TBS
Ac
NHAc
Ph
4.00
5.01
1.00
1.00
3.03
3.01
1.06
9.00
3.01
3.00
f1 (ppm)
205.9
168.3
140.5
140.5
140.4
128.7
127.4
127.4
127.1
126.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
68.2
26.3
24.2
23.7
16.8
15.4
-5.1
-5.6
TBS
Ac
NHAc
Ph
f1 (ppm)


7.44
7.34
7.32
7.32
7.31
7.31
7.31
7.29
7.26 CDCl3
2.61
2.58
1.95
1.90
1.60
1.57
0.90
-0.03
-0.10
TBS
Ac
NHAc
N
f1 (ppm)


206.2
169.0
156.2
148.0
137.4
123.1
121.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.6
26.2
23.7
23.6
18.0
16.5
-5.0
-6.1
TBS
Ac
NHAc
N
f1 (ppm)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8.01
7.99
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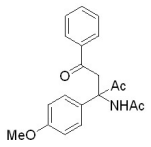
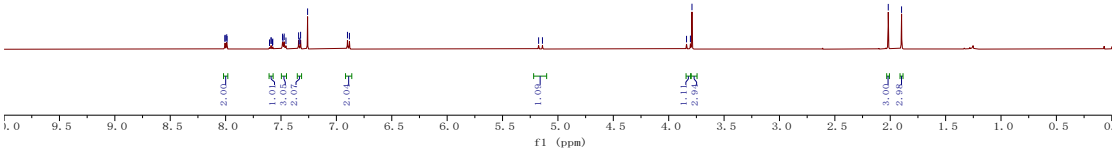
7.60
7.58
7.49
7.48
7.46
7.44
7.42
7.39
7.38
7.38
7.36
7.32
7.31
7.29
7.26 CDCl3
5.19
5.15
3.89
3.85
2.03
1.91
Ph
O
Ac
NHAc
f1 (ppm)
203.0
198.0
169.4
137.7
136.6
133.8
129.0
128.7
128.4
128.2
125.7
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.5
41.0
23.7
23.1
Ph
O
Ac
NHAc
f1 (ppm)


8.01
8.00
7.99
7.99
7.60
7.59
7.59
7.58
7.57
7.49
7.48
7.47
7.46
7.34
7.34
7.33
7.32
7.26
6.90
6.88
5.17
5.14
3.84
3.80
3.79
2.02
1.90


O
Ac
NHAc
MeO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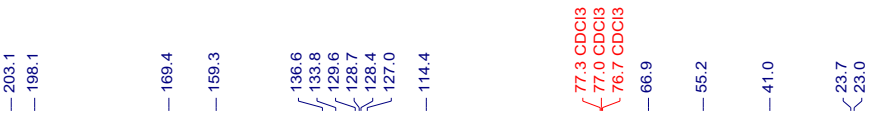
1.01
3.02
2.00
2.04
1.04
1.11
2.91
3.00
2.96
f1 (ppm)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198.1
169.4
159.3
136.6
133.8
129.6
128.7
128.4
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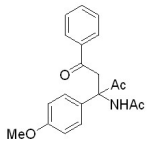
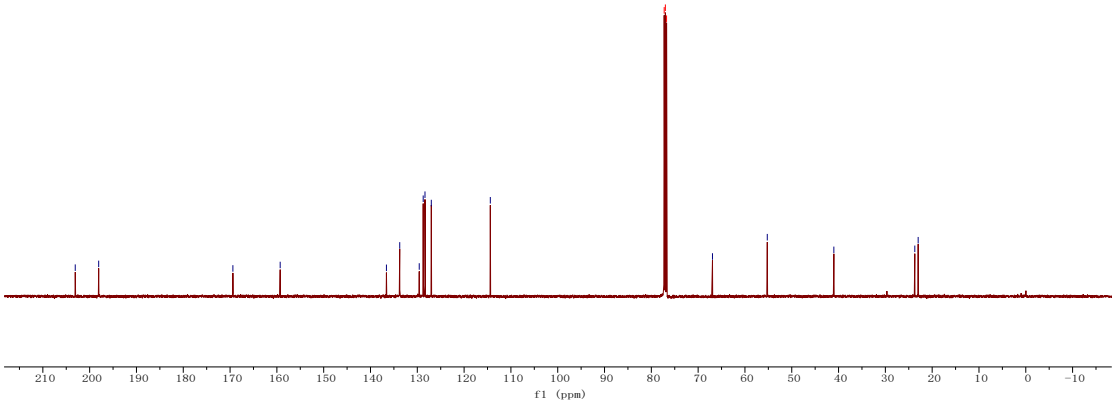
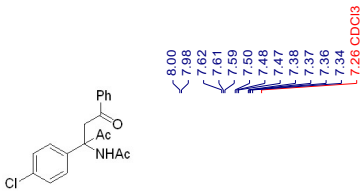
114.4
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
66.9
55.2
41.0
23.7
23.0


O
Ac
NHAc
MeO


f1 (ppm)


Ph
O
Ac
NHAc
Cl

8.00
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7.50
7.48
7.47
7.38
7.37
7.36
7.34
7.26 CDCl3
5.16
5.13
3.84
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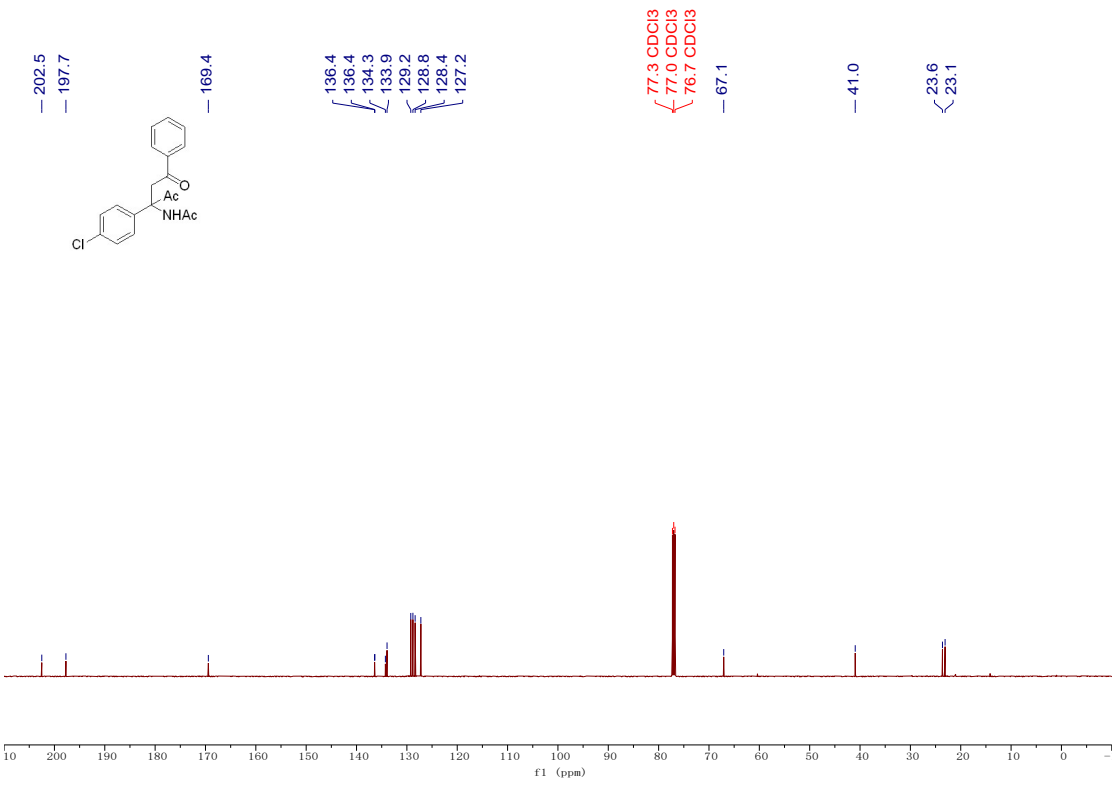
2.00
1.00
3.00
4.00
1.00
1.00
3.00
3.00
f1 (ppm)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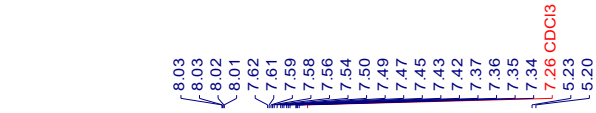
197.7
169.4
136.4
136.4
134.3
133.9
129.2
128.8
128.4
127.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.1
41.0
23.6
23.1
O
Ac
NHAc
Cl
f1 (ppm)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8.03
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7.47
7.45
7.43
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7.34
7.26 CDCl3
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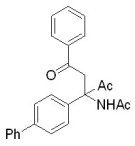
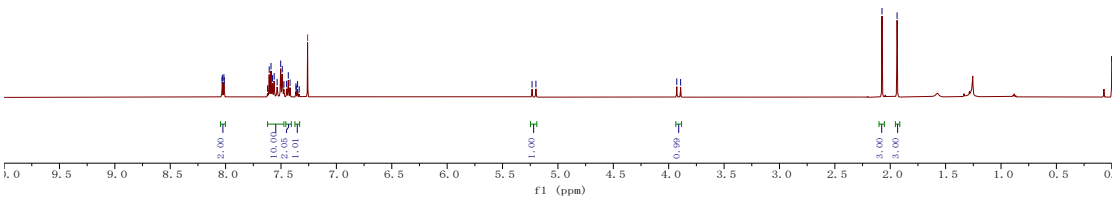
1.94


O
Ac
NHAc
Ph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0.99
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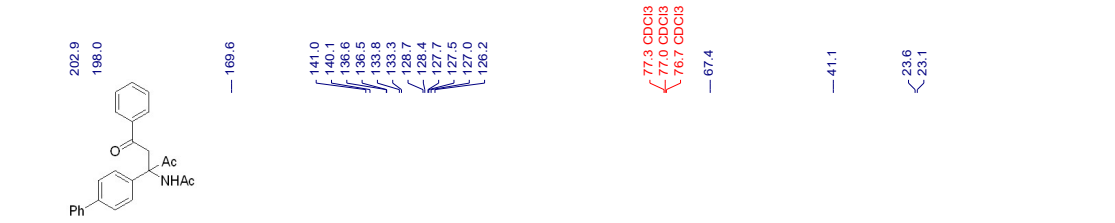
3.00
f1 (ppm)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136.5
133.8
133.3
128.7
128.4
127.7
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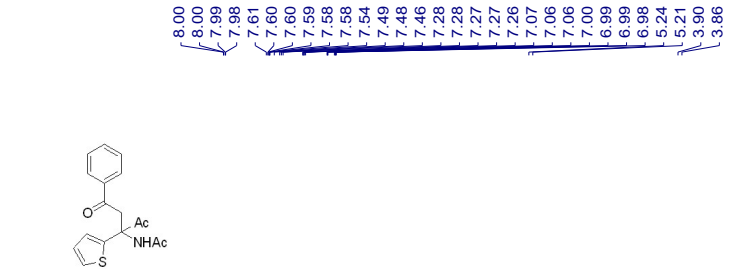
127.0
126.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.4
41.1
23.6
23.1
f1 (ppm)

O
Ac
NHAc
Ph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7.58
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7.49
7.48
7.46
7.28
7.28
7.27
7.27
7.26
7.07
7.06
7.06
7.00
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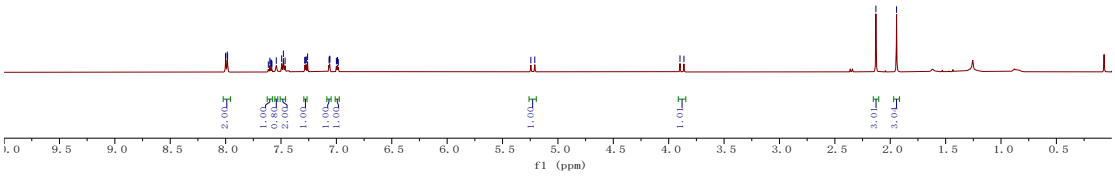
6.98
5.24
5.21
3.90
3.86
2.13
1.94

O
Ac
NHAc
S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1.00
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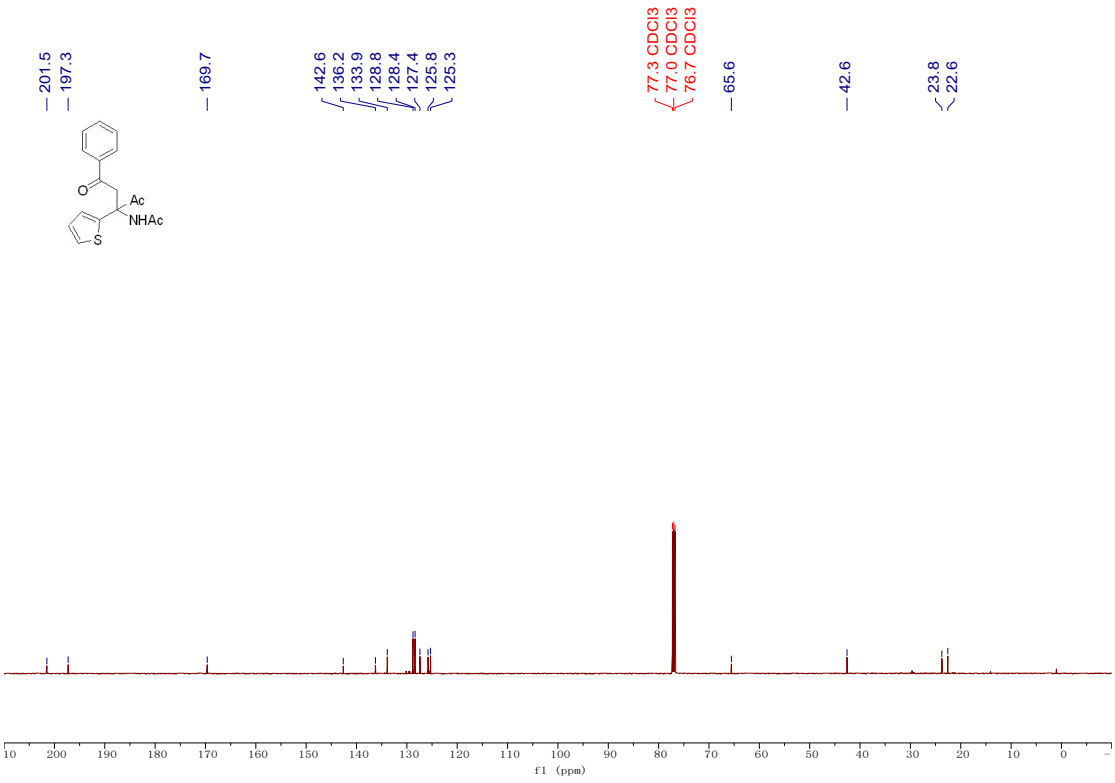
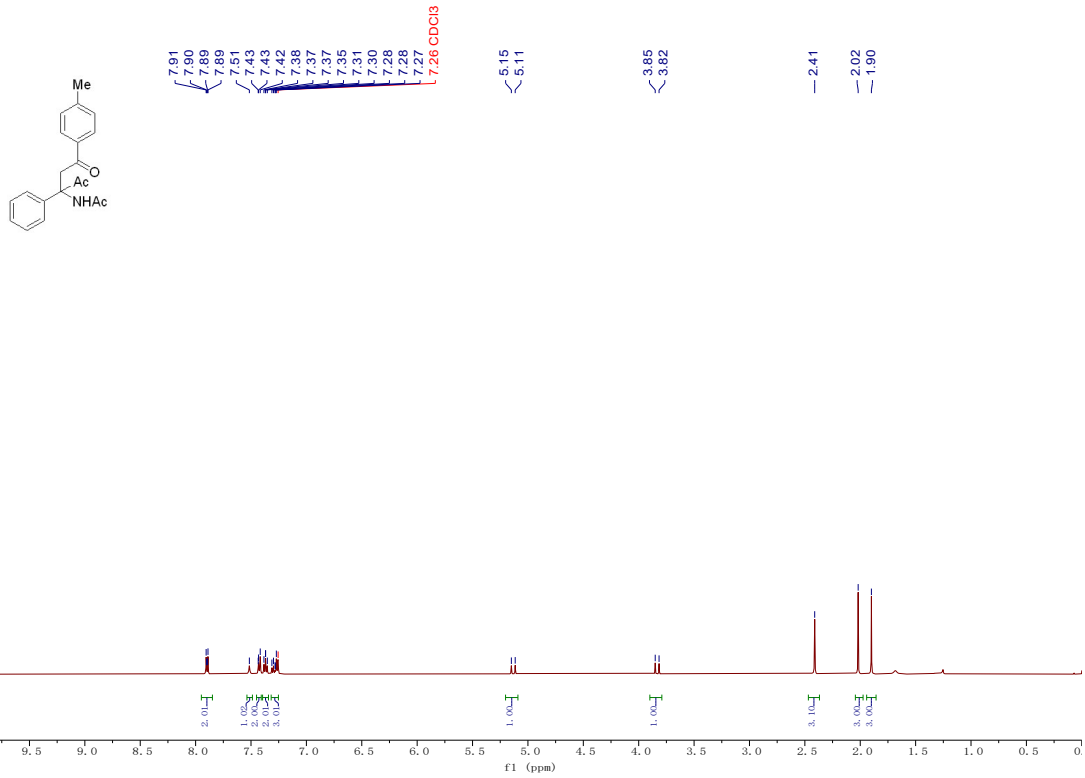
3.04
f1 (ppm)


201.5
197.3
169.7
142.6
136.2
133.9
128.8
128.4
127.4
125.8
125.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
65.6
42.6
23.8
22.6
f1 (ppm)

O
Ac
NHAc
S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7.90
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7.37
7.37
7.35
7.31
7.30
7.28
7.28
7.27
7.26 CDCl3
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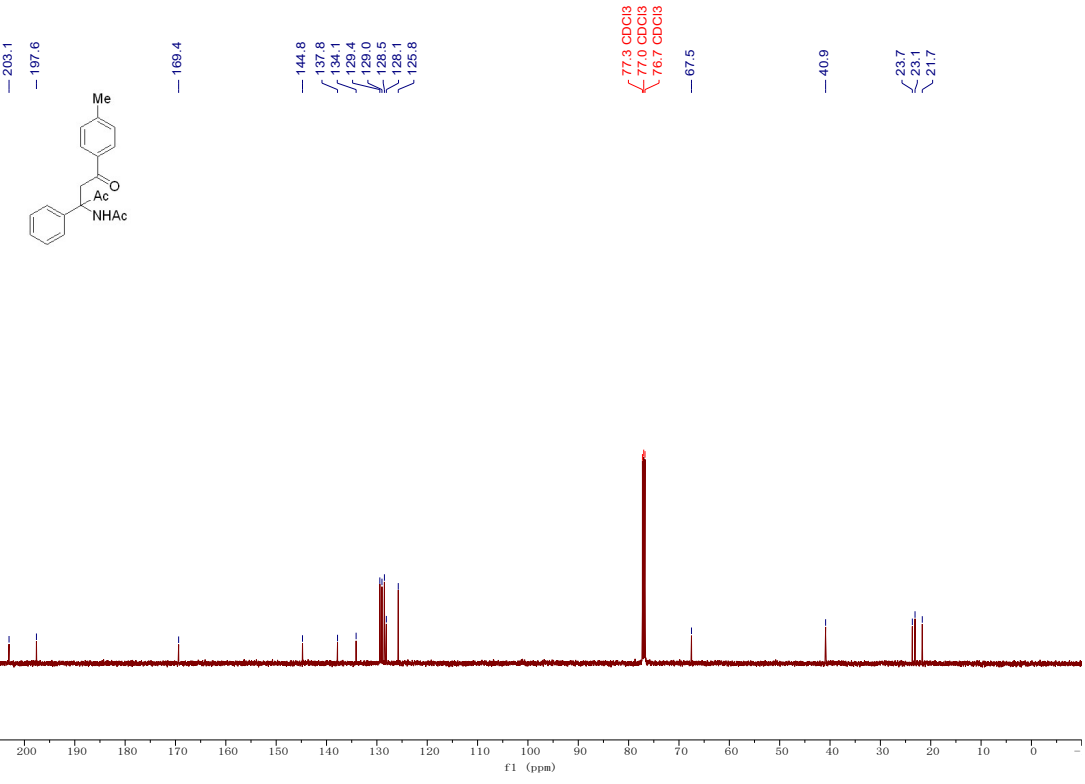
3.85
3.82
2.41
2.02
1.90
Me
O
Ac
NHAc
f1 (ppm)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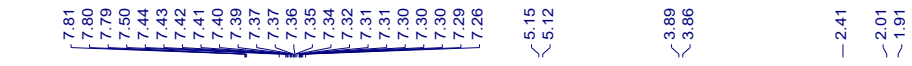
197.6
169.4
144.8
137.8
134.1
129.4
129.0
128.5
128.1
125.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.5
40.9
23.7
23.1
21.7
Me
O
Ac
NHAc
f1 (ppm)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7.32
7.31
7.31
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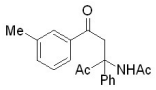
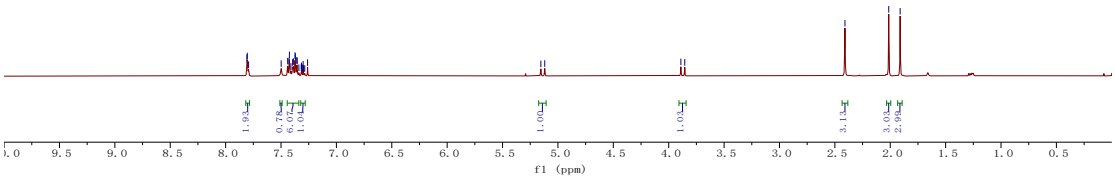
1.91


O
Me
Ac
NHAc
Ph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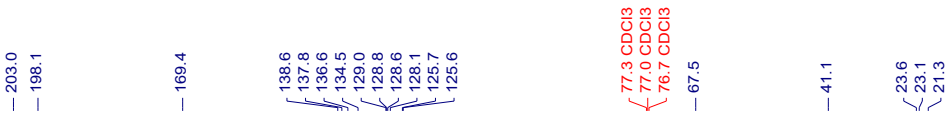
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169.4
138.6
137.8
136.6
134.5
129.0
128.8
128.6
128.1
125.7
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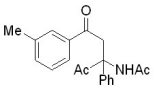
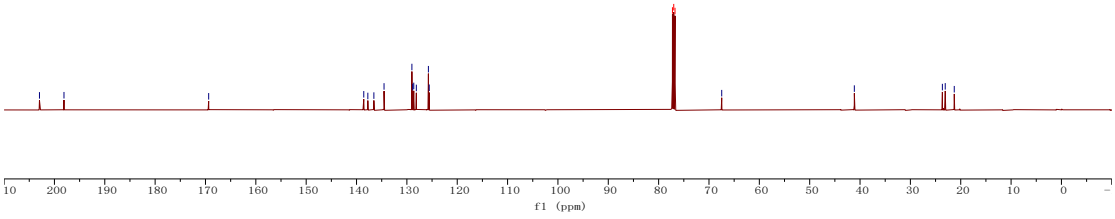
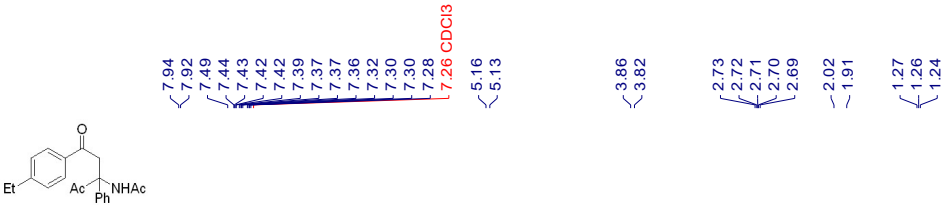
76.7 CDCl3
67.5
41.1
23.6
23.1
21.3


O
Me
Ac
NHAc
Ph


f1 (ppm)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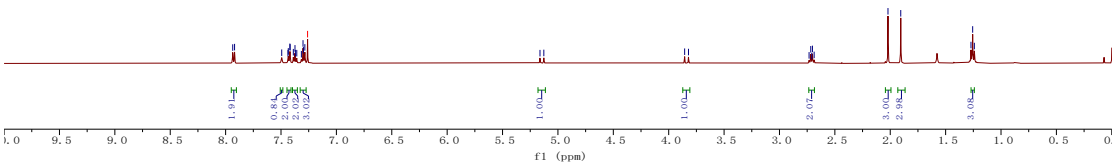
7.30
7.28
7.26 CDCl3
5.16
5.13
3.86
3.82
2.73
2.72
2.71
2.70
2.69
2.02
1.91
1.27
1.26
1.24
O
Et
Ac
NHAc
Ph


1.91
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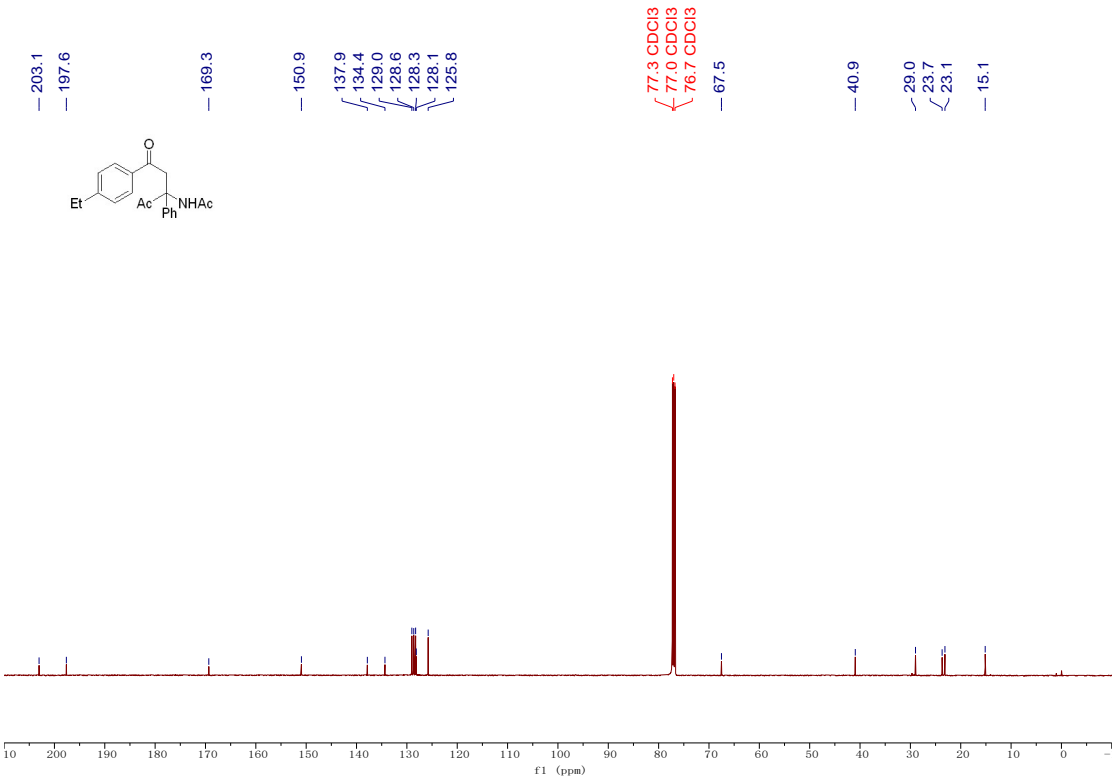
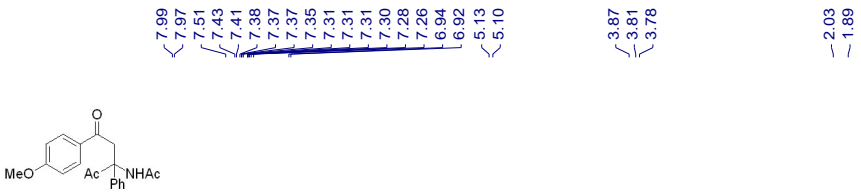
2.03
3.02
1.00
1.00
2.07
3.00
2.96
3.08
f1 (ppm)


203.1
197.6
169.3
150.9
137.9
134.4
129.0
128.6
128.3
128.1
125.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.5
40.9
29.0
23.7
23.1
15.1
O
Et
Ac
NHAc
Ph
f1 (ppm)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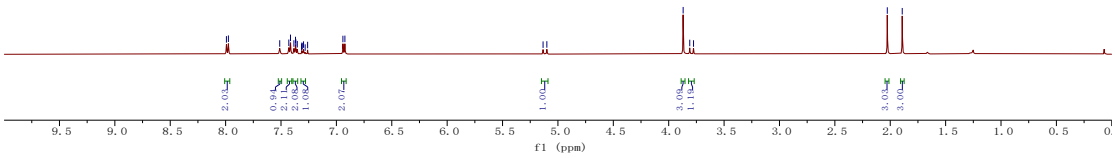
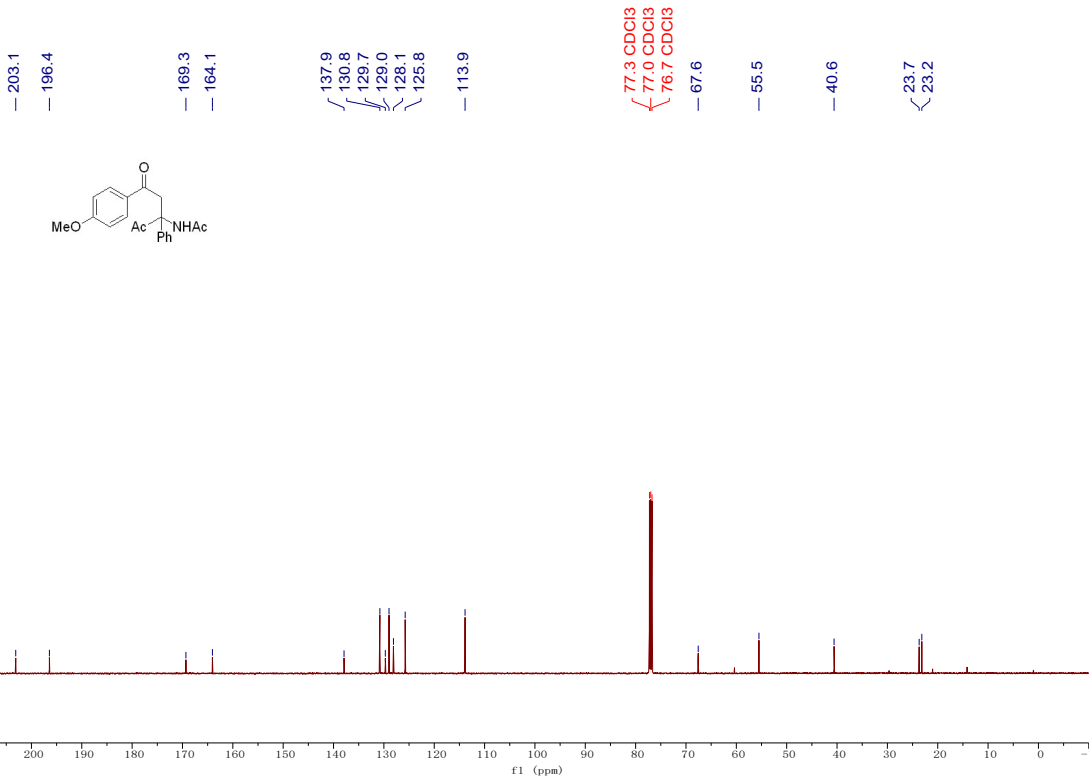
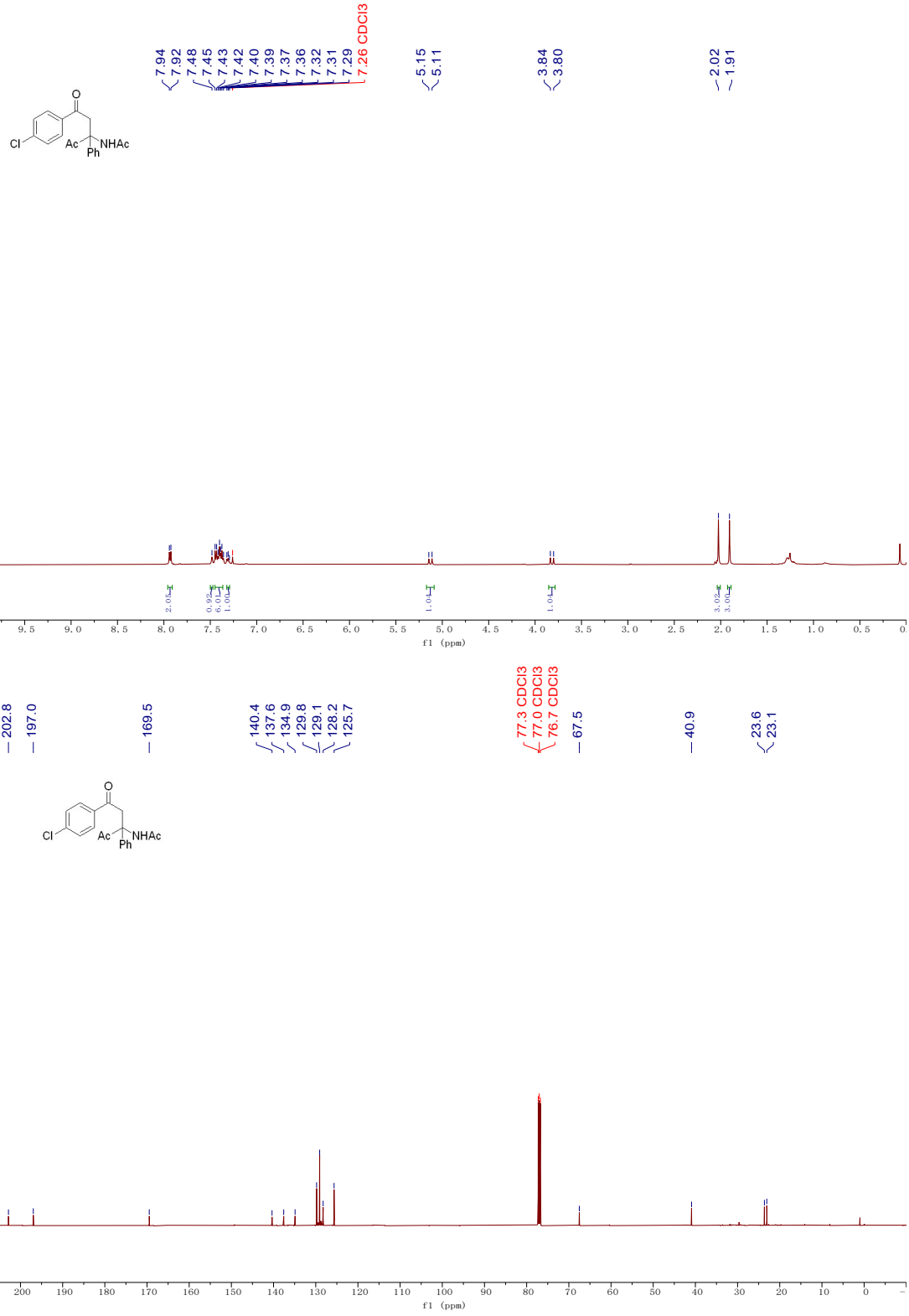
7.97
7.51
7.43
7.41
7.38
7.37
7.37
7.35
7.31
7.31
7.31
7.30
7.28
7.26
6.94
6.92
5.13
5.10
3.87
3.81
3.78
2.03
1.89
O
MeO
Ac
NHAc
Ph


2.04
0.94
2.11
2.08
1.00
2.07
1.00
3.04
1.11
3.03
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)


203.1
196.4
169.3
164.1
137.9
130.8
129.7
129.0
128.1
125.8
113.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.6
55.5
40.6
23.7
23.2
O
MeO
Ac
NHAc
Ph
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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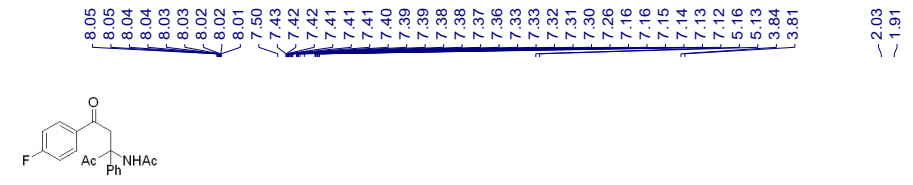
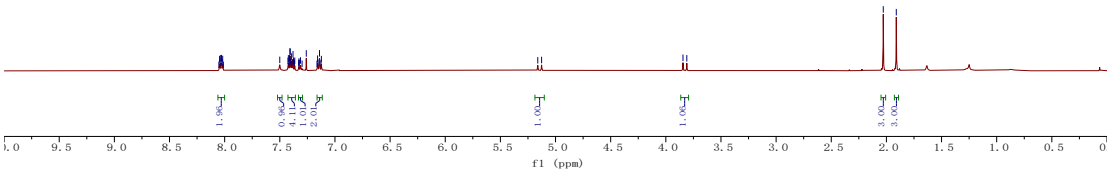
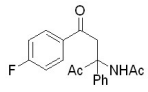
7.92
7.48
7.45
7.43
7.42
7.40
7.39
7.37
7.36
7.32
7.31
7.29
7.26 CDCl3
5.15
5.11
3.84
3.80
2.02
1.91
Cl
Ac
NHAc
Ph
O
f1 (ppm)
202.8
197.0
169.5
140.4
137.6
134.9
129.8
129.1
128.2
125.7
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.5
40.9
23.6
23.1

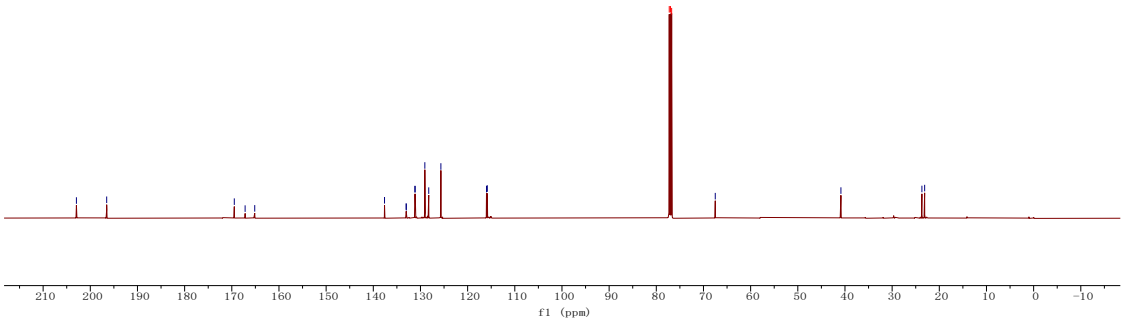

8.05
8.05
8.04
8.04
8.03
8.03
8.02
8.02
8.01
7.50
7.43
7.42
7.42
7.41
7.41
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.36
7.33
7.33
7.32
7.31
7.30
7.26
7.16
7.16
7.15
7.14
7.13
7.12
5.16
5.13
3.84
3.81
2.03
1.91

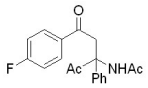
O
F
Ac
NHAc
Ph


1.96
0.98
4.11
1.01
2.01
1.00
1.00
3.00
3.00
f1 (ppm)

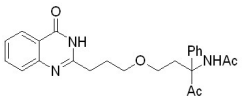

202.9
196.5
169.5
167.2
165.1
137.6
133.0
133.0
131.2
131.1
129.1
128.2
125.7
116.0
115.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
67.5
40.9
23.7
23.1

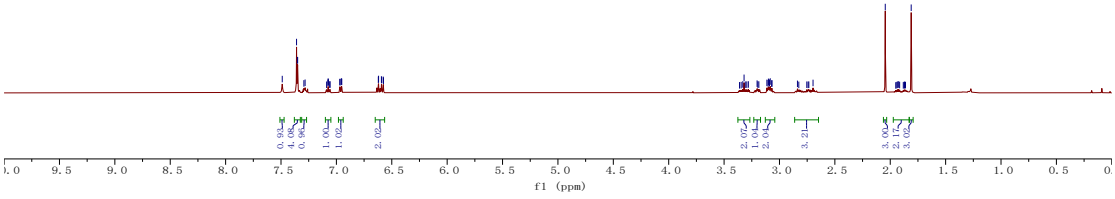

O
F
Ac
NHAc
Ph

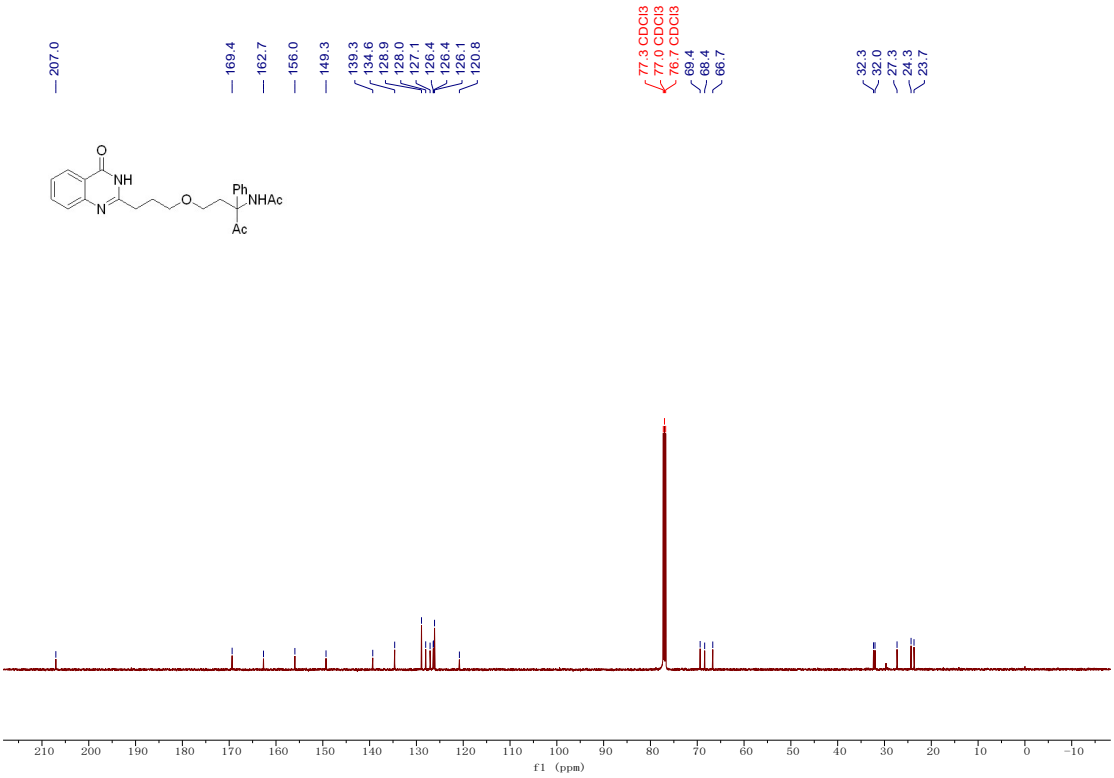

f1 (ppm)

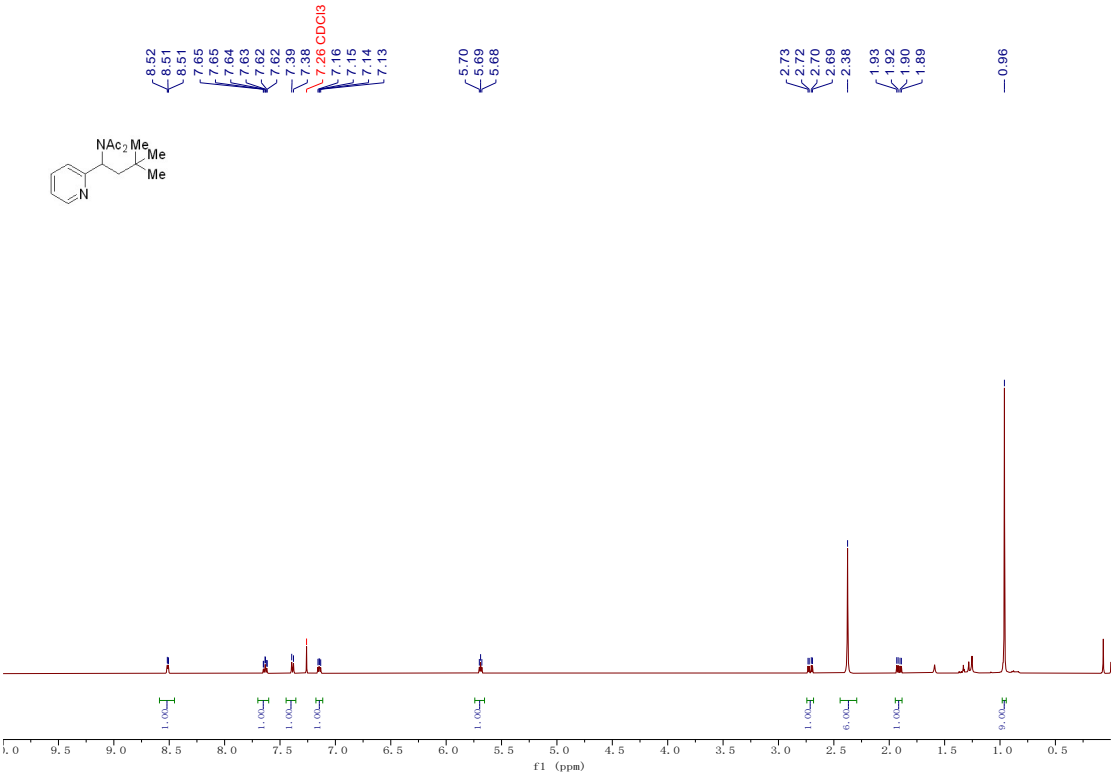

O
F
Ac
NHAc
Ph

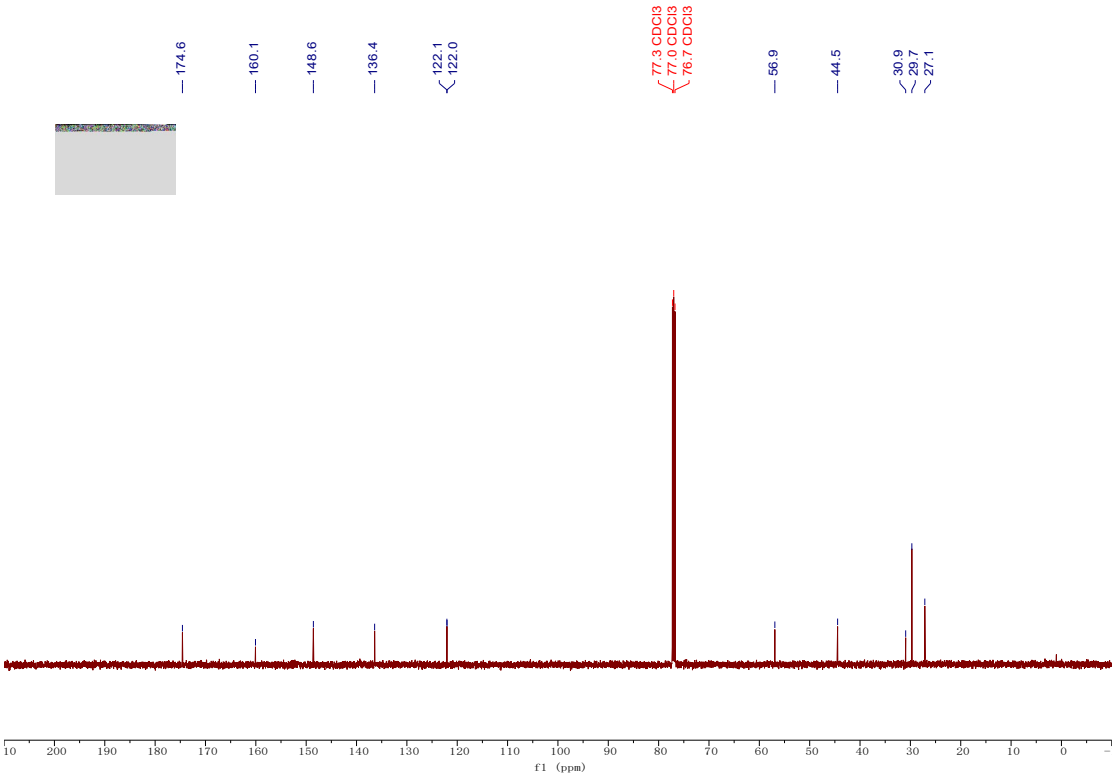
-103.67
f1 (ppm)
7.49 7.36 7.35 7.35 7.29 7.28 7.09 7.08 7.07 7.07 7.06 7.06 6.97 6.95 6.95 6.62 6.62 6.59 6.59 6.58 6.58
3.36 3.35 3.33 3.32 3.31 3.29 3.28 3.20 3.20 3.19 3.11 3.10 3.10 3.09 3.09 3.08 3.07 3.07 2.84 2.83 2.75 2.74 2.73 2.70 2.04 1.95 1.94 1.93 1.92 1.92 1.88 1.87 1.87 1.87 1.86 1.81

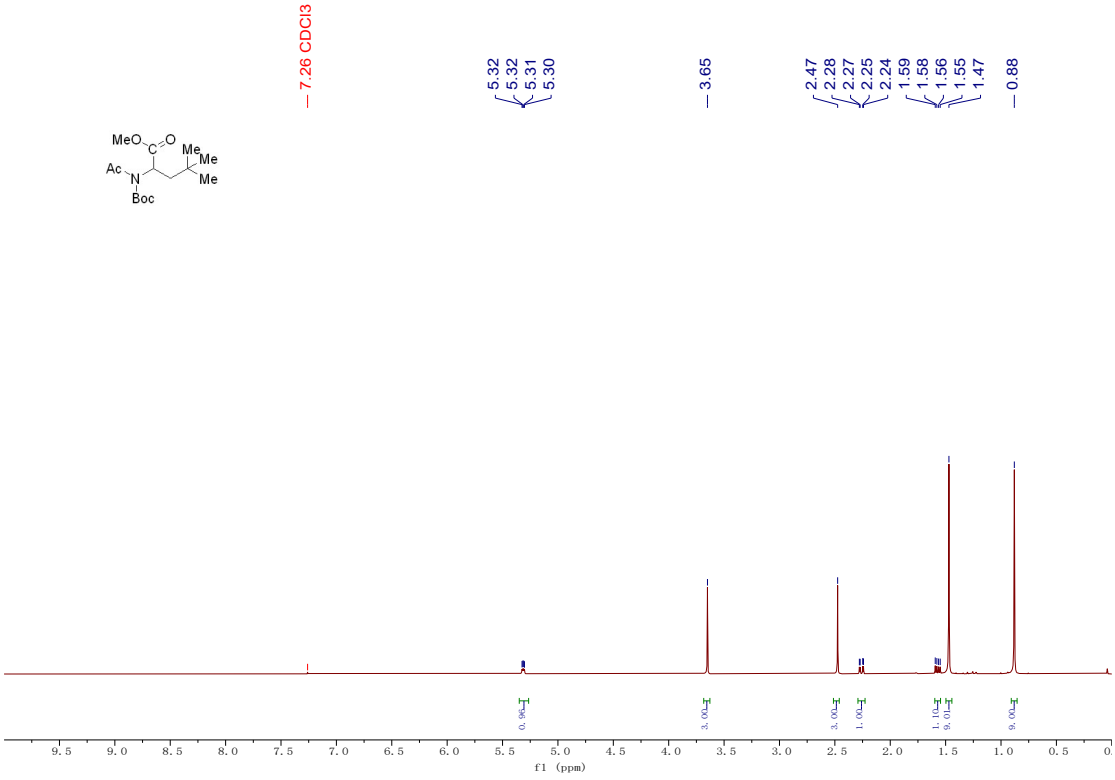

O
NH
N
O
Ph
NHAc
Ac

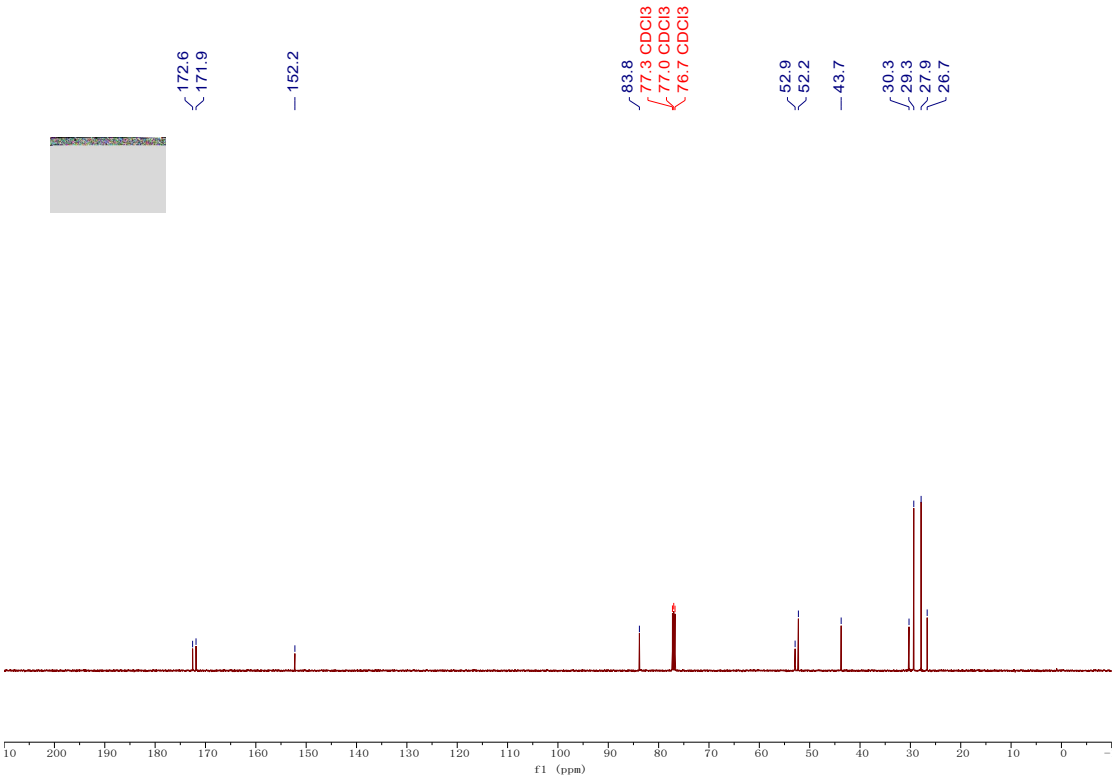

0.93
4.08
0.96
1.00
1.02
2.02
2.07
1.04
2.04
3.21
3.00
3.17
3.02
f1 (ppm)


207.0
169.4
162.7
156.0
149.3
139.3
134.6
128.9
128.0
127.1
126.4
126.4
126.1
120.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.4
68.4
66.7
32.3
32.0
27.3
24.3
23.7
O
NH
N
O
Ph
NHAc
Ac
f1 (ppm)


8.52
8.51
8.51
7.65
7.65
7.64
7.63
7.62
7.62
7.39
7.38
7.26 CDCl3
7.16
7.15
7.14
7.13
5.70
5.69
5.68
2.73
2.72
2.70
2.69
2.38
1.93
1.92
1.90
1.89
0.96
NAc2
Me
Me
Me
N
1.00
1.00
1.00
1.00
1.00
1.00
6.00
1.00
9.00
f1 (ppm)


174.6
160.1
148.6
136.4
122.1
122.0
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
56.9
44.5
30.9
29.7
27.1
f1 (ppm)


7.26 CDCl3
5.32
5.32
5.31
5.30
3.65
2.47
2.28
2.27
2.25
2.24
1.59
1.58
1.56
1.55
1.47
0.88
MeO
O
Me
Me
Me
Ac
N
Boc
f1 (ppm)


172.6
171.9
152.2
83.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
52.9
52.2
43.7
30.3
29.3
27.9
26.7
f1 (ppm)