Supporting Information

Palladium-Catalyzed Cascade Heck-Type Cyclization and Reductive Aminocarbonylation for the Synthesis of Functionalized Amides

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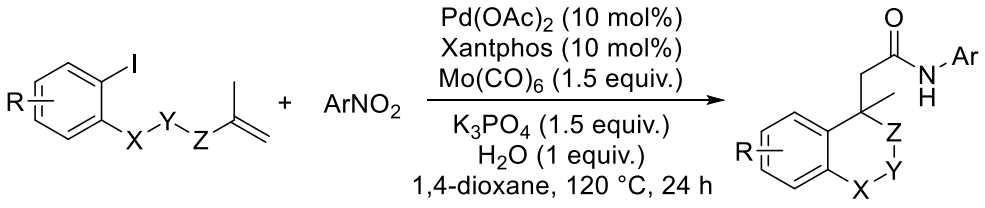
Table of Contents

1. General Information

Unless otherwise noted, all reactions were carried out under a nitrogen atmosphere. All reagents were from commercial sources and used as received without further purification. All solvents were anhydrous. Column chromatography was performed on silica gel (200-300 meshes) using petroleum ether (b.p. 60-90 °C) and ethyl acetate as the eluents. ^{1}H and ^{13}C NMR spectra were taken on 400 MHz instruments and spectral data were reported in ppm relative to tetramethylsilane (TMS) as the internal standard and $CDCl_3$ (^{1}H NMR δ 7.26, ^{13}C NMR δ 77.0) as solvent. All coupling constants (J) are reported in Hz with the following abbreviations: s = singlet, d = doublet, dd = double doublet, ddd = double doublet of doublets, t = triplet, dt = double triplet, q = quartet, m = multiplet, br = broad. Gas chromatography (GC) analyses were performed on a Shimadzu GC-2014C chromatograph equipped with FID detector. Mass spectra (MS) were measured on spectrometer by direct inlet at 70 eV.

2. General Procedure for the Synthesis of Amides



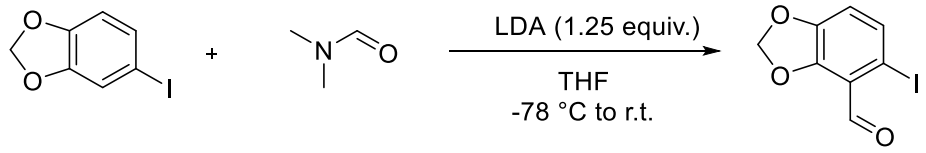
1 **2** **3**

1 (0.3 mmol), **2** (0.2 mmol), $Pd(OAc)_2$ (10 mol%, 4.5 mg), Xantphos (10 mol%, 11.6 mg), $Mo(CO)_6$ (1.5 equiv., 79.2 mg), K_3PO_4 (1.5 equiv., 63.7mg), H_2O (1 equiv., 3.6 μL) and 1,4-dioxane (1 mL) were added to an oven-dried tube (15 mL). The tube was sealed and the mixture was stirred at 120 °C for 24 hours. After the reaction was completed, the crude mixture was filtered and concentrated under vacuum. The crude product was purified by column chromatography on silica gel using PE/EA = 5/1 to afford the desired products.

3. Starting Material Synthesis

3.1 Procedure for the Synthesis of 2-Iodobenzyl Alcohols

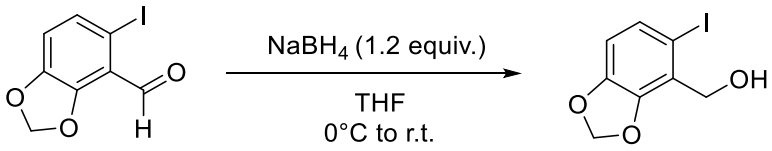
5-iodobenzo[*d*][1,3]dioxole-4-carbaldehyde[1]



To a stirred solution of LDA (1.25 equiv., 3.1 mL, 2 M in THF) at –78 °C was added neat 5-iodobenzo dioxole (5 mmol, 1.24 g) drop wise such that the internal reaction temperature did not exceed –70 °C. This was stirred at –78 °C for 2 hours, at which time anhydrous DMF (2.4 equiv., 0.92 mL) was added dropwise such that the internal temperature did not exceed –70 °C. The resulting solution was allowed to warm to room temperature over night at which time saturated aqueous NH_4Cl (10 mL) and water (30 mL) were added sequentially at 0 °C. The aqueous layer was extracted with EA (30 mL × 3), and the combined organic layers were washed with brine (30 mL), dried over anhydrous Na_2SO_4, filtered, and

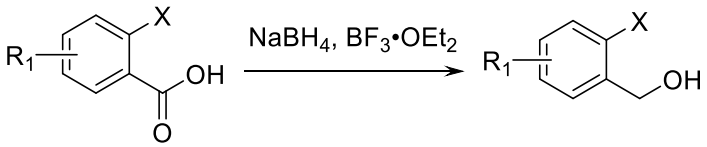
concentrated to afford a deep yellow powder which was recrystallized from PE/EA and the title compound was obtained as a deep yellow crystal.

(5-iodobenzo[*d*][1,3]dioxol-4-yl)methanol[2]



To a suspension of $NaBH_4$ (1.2 equiv., 576 mg) in 50 mL anhydrous THF at 0 °C was added a solution of 5-iodobenzo[*d*][1,3]dioxole-4-carbaldehyde (12.7 mmol, 3.5 g) in anhydrous THF (100 mL) dropwise over a period of 20 minutes after which the mixture warmed to room temperature. After three hours of stirring at room temperature, the excess $NaBH_4$ was quenched with 1 M HCl (100 mL) and the mixture was extracted with CH_2Cl_2 (150 mL × 3). The extracts were combined and washed with Na_2CO_3 (50 mL × 3), water (50 mL × 3), dried over anhydrous Na_2SO_4, filtered and evaporated to dryness. This process afforded the title compound as a yellow powder without further purification.

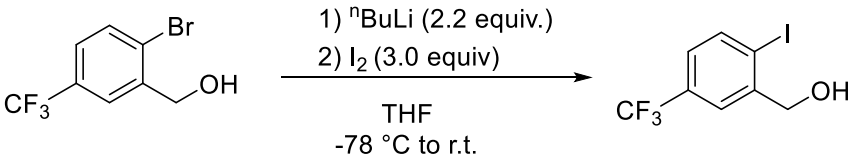
(2-iodophenyl)methanol/(2-bromophenyl)methanol[3]



X = I, Br X = I, Br

To a solution of 2-iodobenzoic acid (5 mmol) / 2-bromobenzoic acid (5 mmol) and $NaBH_4$ (2 equiv., 378.3 mg) in THF (10 mL) at 0 ºC was slowly added $BF_3 \cdot OEt_2$ (2 equiv., 1.25 mL) over a period of 30 minutes and then the mixture was vigorously stirred at room temperature. When the reaction was completed as determined by TLC analysis, the reaction mixture was cooled to 0 °C, H_2O was slowly added and then extracted with EA (20 mL × 3). The combined organic layers were dried over anhydrous Na_2SO_4, and the solvent was evaporated in vacuum obtaining the title compound as a white solid without further purification.

(2-iodo-5-(trifluoromethyl)phenyl)methanol[2]

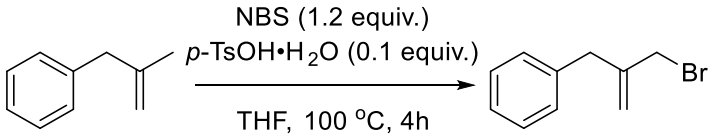


A dry round bottom flaks was charged with (2-bromo-5-(trifluoromethyl)phenyl)methanol (2.3 mmol, 0.59 g,) and anhydrous THF (20 mL). The resulting solution was cooled to –78 °C, then *n*-butyllithium (2.2 equiv., 2.1 mL, 2.4 M in hexanes) was added dropwise over 5 minutes. The mixture was stirred at –78 °C for 2.5 hours (complete conversion monitored by TLC), then solution of I_2 (3.0 equiv., 1.8 g) in anhydrous THF (5 mL) was added dropwise over 10 minutes. After an additional 10 minutes at –78 °C, the resulting mixture was warmed to room temperature and stirred for 3 hours. The mixture was poured into saturated aqueous NH_4Cl-solution (20 mL), followed by saturated NaS_2O_3 solution (20 mL), extracted with EA (50 mL × 3) and the combined organic phases were dried over Na_2SO_4. After

evaporation of the solvent under reduced pressure, the crude oil was purified via silica gel flash column chromatography using PE/EA = 5/1 as the mobile phase and the title compound was obtained as a white solid.

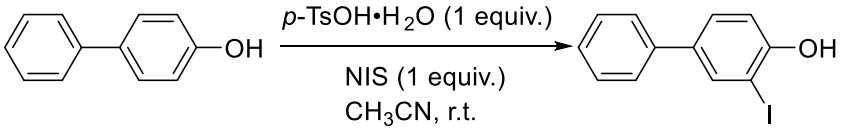
3.2 Procedure for the Synthesis of 2-Iodophenols

(2-(bromomethyl)allyl)benzene[4]



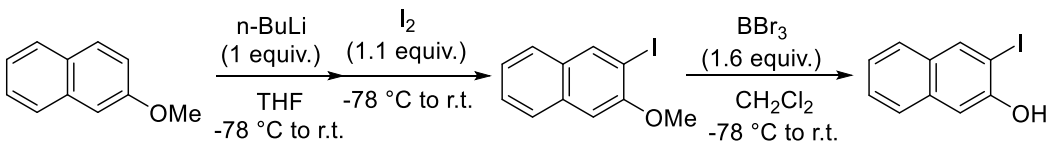
N-Bromosuccinimide (1.2 equiv., 2.1 g) and *p*-TsOH·H_2O (0.1 equiv., 190 mg) were added to the THF (20 mL) solution of 2-aryl-1-propene (10 mmol, 1.3 g) and the solution was refluxed at 100 °C in oil bath for 4 hours. After completion, the reaction mixture was filtered through celite. The filtrate was extracted with EA (20 mL × 3), washed with H_2O (20 mL), brine (20 mL), and dried over Na_2SO_4. The solution was concentrated under reduced pressure. The crude oil was purified via silica gel flash column chromatography using a gradient starting from PE to PE/EA = 100/1 as the mobile phase and the title compound was afforded as a clear and colorless oil.

3-iodo-[1,1'-biphenyl]-4-ol[5]



To a solution of the corresponding phenol (10 mmol, 1.7 g) in CH_3CN (2 M) at room temperature was added *p*-TsOH·H_2O (1 equiv., 1.9 g). After 10 min, added NIS (1 equiv., 2.25 g) to the reaction mixture. The mixture was stirred for 8 hours at room temperature and quenched by addition of aqueous Na_2SO_3 solution. It was acidified by addition of aqueous HCl (1 M), the organic solvent was evaporated, and the aqueous layer was extracted twice with EA. The combined organic layers were dried with Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified via silica gel flash column chromatography using a gradient starting from PE/EA = 10/1 to PE/EA = 6/1 as the mobile phase and the title compound was afforded as a colorless solid.

3-iodonaphthalen-2-ol[6]

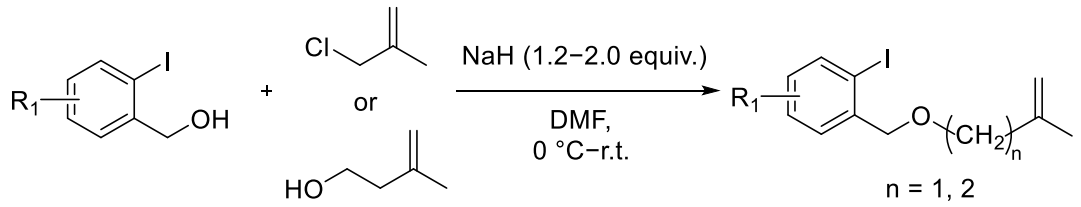


To a solution of 2-methoxynaphthalene (1.02 mmol, 161 mg) dissolved in THF (10 mL) was added *n*-butyllithium (1 equiv., 0.43 mL, 2.4 M in hexane) at –78 °C. After gradually warming to room temperature, the mixture was stirred for 1 hour and then to this was added a solution of iodine (1.1 equiv., 290 mg) dissolved in THF (5 mL) at –78 °C. After gradually warming to room temperature, the mixture was stirred for 26 hours and then to this was added saturated aqueous ammonium chloride (20 mL). The mixture was extracted with EA (10 mL × 3), and the combined organic extract was washed with brine (10 mL), dried (Na_2SO_4), and after filtration, the filtrate was concentrated under reduced pressure to

afford a crude product containing 2-methoxy-3-iodonaphthalene (ca. 1.0 mmol) as a colorless oil. To a solution of the crude product dissolved in dichloromethane (5 mL) was added boron tribromide (1.6 equiv., 1.6 mL, 1 M in DCM solution,) at –78 °C. After gradually warming to room temperature, the mixture was stirred for 14 hours and then to this was added saturated aqueous sodium bicarbonate (10 mL). The mixture was extracted with dichloromethane (10 mL × 3), and the combined organic extract was washed with brine (10 mL), dried (Na_2SO_4), and after filtration, the filtrate was concentrated under reduced pressure. The residue was purified via silica gel flash column chromatography using a gradient starting from PE/EA = 10/1 to PE/EA = 6/1 as the mobile phase and the title compound was afforded as a colorless solid.

3.3 Procedure for the Synthesis of Iodoarenes

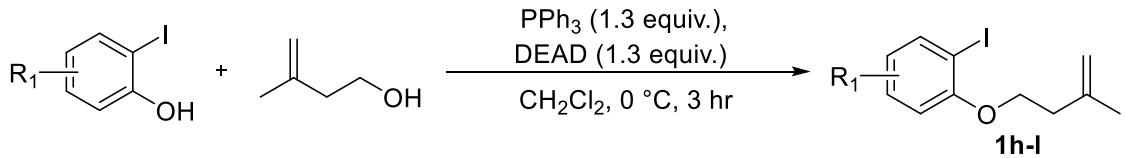
Compounds 1a-g, 1m[2]



1a-g, 1m

A dry round bottom flask was charged with NaH (1.3−2.0 equiv., 60% suspension in mineral oil), suspended in DMF (anhydrous, 0.4−0.6 M) under an N_2 atmosphere and stirred for 5 minutes at 0 °C. At 0 °C, a solution of the substituted benzyl alcohol (1.0 equiv.) in DMF (anhydrous, 0.4−0.6 M) was added dropwise over a period of 15 minutes. The mixture was stirred for 30 minutes at 0 °C, after which methallyl chloride / 3-methylbut-3-en-1-ol (1.2−1.5 equiv.) was added dropwise to the reaction mixture over a period of 20 minutes. The mixture was stirred at room temperature until full conversion was detected by TLC (usually 2−6 hours). After that, saturated aqueous NH_4Cl-solution was added and the mixture was extracted EA. The organic layers were combined, washed with twice with H_2O and brine, dried over Na_2SO_4, and the solvent was evaporated under reduced pressure. The crude oil was purified via silica gel flash column chromatography using a gradient starting from hexanes to PE/EA = 100/1 as the mobile phase and the title compound was afforded as clear and colorless oil.

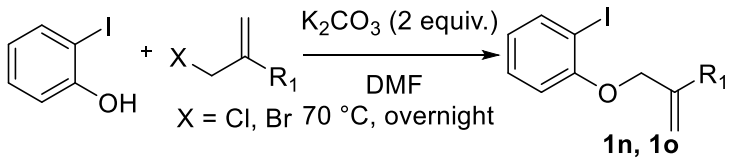
Compounds 1h-l[7]



1h-l

A 300 mL round bottom flask was charged with substituted 2-iodophenol (8.72 mmol), 2-methylbut-3-en-1-ol (1.3 equiv., 973 mg), triphenylphosphine (1.3 equiv., 2.97 g), and anhydrous CH_2Cl_2 (80 mL). The reaction mixture was stirred at 0 °C under N_2 atmosphere, then DEAD (1.3 equiv., 2 g) was added to the reaction mixture. After 3 hours, to the reaction mixture were added EA (100 mL) and brine (100 mL), and the separated organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude oil was purified via silica gel flash column chromatography using a gradient starting from PE to PE/EA = 100/1 as the mobile phase and the title compound was afforded as a clear and colorless oil.

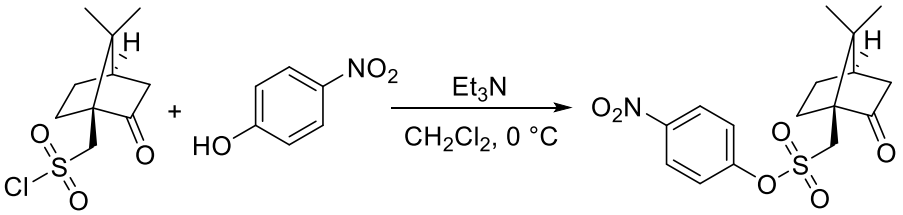
Compounds 1n, 1o[8]



2-iodophenol (5 mmol, 1.1 g) and K_2CO_3 (2 equiv., 1.38 g) were dissolved in DMF (0.2 M). 3-chloro-2-methylprop-1-ene / 3-bromo-2-methylprop-1-ene (1.2 equiv.) was added by syringe and the reaction was heated to 70 °C overnight. After cooling, the contents were diluted with EA and transferred to a separatory funnel. The organic phase was washed twice with H_2O and once with brine. and the separated organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude oil was purified via silica gel flash column chromatography using a gradient starting from PE to PE/EA = 100/1 as the mobile phase and the title compound was afforded as a clear and colorless oil.

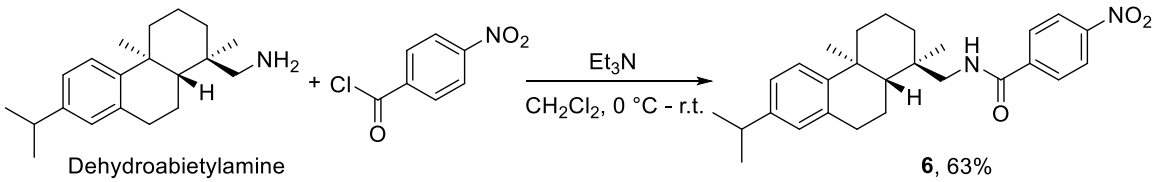
3.4 Procedure for the Synthesis of Compounds 4 and 6[9]

Compound 4



An oven-dried round-bottom flask was charged under air with D-10-Camphorsulfonyl chloride (12 mmol, 3.00 g), DCM (40 mL), 4-nitrophenol (10 mmol, 1.39 g), and triethylamine (15 mmol, 1.52 g) was stirred at 0 °C until completion (monitored by TLC). The reaction mixture was then diluted with 40 mL of H_2O and extracted with DCM (25 mL × 3). The combined organic phases were dried over Na_2SO_4, filtered through short celite pad, and concentrated under reduced pressure. The residue was purified via silica gel flash column chromatography using a gradient starting from PE/EA = 10/1 to PE/EA = 6/1 as the mobile phase and the title compound was afforded as a white solid (2.22 g, 67%).

Compound 6

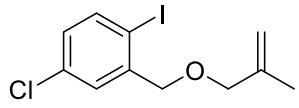


An oven-dried round-bottom flask was charged under air with dehydroabietylamine (10 mmol, 2.85 g), DCM (40 mL), and triethylamine (12 mmol, 1.21 g), 4-nitrobenzoyl chloride (11 mmol, 2.04 g) was add in slowly and stirred at 0 °C, then stir the reaction mixture at room temperature until completion (monitored by TLC). The reaction mixture was then diluted with 40 mL of H_2O and extracted with DCM. (25 mL × 3). The combined organic phases were dried over Na_2SO_4, filtered through short celite pad, and concentrated under reduced pressure. The residue was purified via silica gel flash column

chromatography using a gradient starting from PE/EA = 10/1 to PE/EA = 5/1 as the mobile phase and the title compound was afforded as a light yellow solid (2.73 g, 63%).

4. Characterization

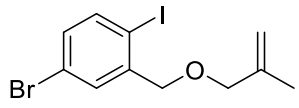
4.1 Characterization of Compounds 1f, 1g, 1h, 1i.



4-chloro-1-iodo-2-(((2-methylallyl)oxy)methyl)benzene (1f)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 8.4 Hz, 1H), 7.48 (d, J = 2.5 Hz, 1H), 6.96 (dd, J = 8.4, 2.6 Hz, 1H), 5.07 (s, 1H), 4.98 (s, 1H), 4.40 (s, 2H), 4.03 (s, 2H), 1.81 (s, 3H).

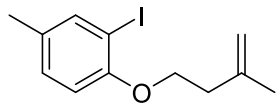
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.4, 141.5, 139.7, 134.6, 128.9, 128.3, 112.6, 93.8, 75.0, 74.7, 19.5.



4-bromo-1-iodo-2-(((2-methylallyl)oxy)methyl)benzene (1g)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.61 (m, 2H), 7.11 (dd, J = 8.3, 2.4 Hz, 1H), 5.05 (s, 1H), 4.97 (s, 1H), 4.41 (s, 2H), 4.03 (s, 2H), 1.80 (s, 3H).

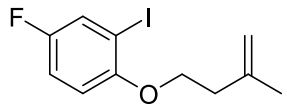
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.7, 141.6, 140.1, 131.9, 131.3, 122.7, 112.7, 94.8, 75.0, 74.8, 19.6.



2-iodo-4-methyl-1-((3-methylbut-3-en-1-yl)oxy)benzene (1h)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, J = 1.6 Hz, 1H), 7.08 (dd, J = 8.3, 1.4 Hz, 1H), 6.71 (d, J = 8.3 Hz, 1H), 4.87 (s, 1H), 4.85 (s, 1H), 4.09 (t, J = 6.8 Hz, 2H), 2.57 (t, J = 6.8 Hz, 2H), 2.26 (s, 3H), 1.86 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 155.4, 142.1, 139.7, 131.9, 129.8, 112.3, 112.0, 86.5, 68.1, 37.1, 23.0, 19.9.

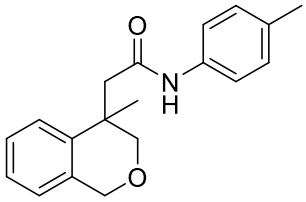


4-fluoro-2-iodo-1-((3-methylbut-3-en-1-yl)oxy)benzene (1i)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, J = 7.6, 3.0 Hz, 1H), 7.03 – 6.98 (m, 1H), 6.73 (dd, J = 9.0, 4.6 Hz, 1H), 4.87 (s, 1H), 4.84 (s, 1H), 4.07 (t, J = 6.8 Hz, 2H), 2.56 (t, J = 6.8 Hz, 2H), 1.84 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 156.7 (d, J = 243.4 Hz), 154.1 (d, J = 2.1 Hz), 141.9, 126.0 (d, J = 25.0 Hz), 115.5 (d, J = 22.6 Hz), 112. 4, 112.2 (d, J = 8.1 Hz), 86.1 (d, J = 8.5 Hz), 68.7, 37.1, 22.9.

4.2 Characterization of Products



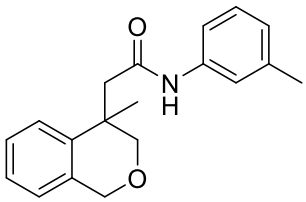
2-(4-methylisochroman-4-yl)-*N*-(*p*-tolyl)acetamide (3aa)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3aa** as a yellow oil (51.4 mg, 87%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.36 (m, 1H), 7.25 – 7.16 (m, 5H), 7.07 (d, *J* = 8.3 Hz, 2H), 7.01 – 6.99 (m, 1H), 4.83 (s, 2H), 4.04 (d, *J* = 11.6 Hz, 1H), 3.62 (d, *J* = 11.6 Hz, 1H), 2.74 (d, *J* = 13.5 Hz, 1H), 2.60 (d, *J* = 13.5 Hz, 1H), 2.29 (s, 3H), 1.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 140.5, 135.1, 133.7, 133.5, 129.3, 127.0, 126.6, 125.8, 124.4, 119.9, 74.4, 68.9, 49.1, 36.5, 23.0, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{21}NO_2$, 296.1645; Found, 296.1657.



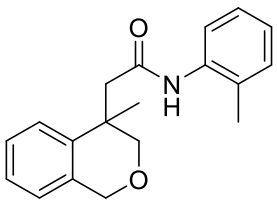
2-(4-methylisochroman-4-yl)-*N*-(*m*-tolyl)acetamide (3ab)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ab** as a yellow oil (52.0 mg, 88%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.36 (m, 1H), 7.31 (s, 1H), 7.24 – 7.18 (m, 3H), 7.17 – 7.08 (m, 2H), 7.01 – 6.99 (m, 1H), 6.89 (d, *J* = 7.3 Hz, 1H), 4.83 (s, 2H), 4.06 (d, *J* = 11.6 Hz, 1H), 3.62 (d, *J* = 11.6 Hz, 1H), 2.75 (d, *J* = 13.5 Hz, 1H), 2.61 (d, *J* = 13.5 Hz, 1H), 2.30 (s, 3H), 1.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.0, 140.5, 138.7, 137.6, 133.5, 128.6, 127.0, 126.5, 125.8, 124.9, 124.3, 120.5, 116.9, 74.3, 68.8, 49.0, 36.5, 23.0, 21.4.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{21}NO_2$, 296.1645; Found, 296.1657.



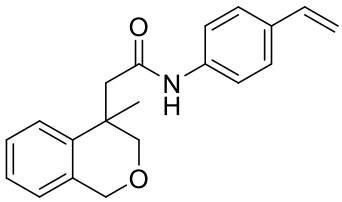
2-(4-methylisochroman-4-yl)-*N*-(*o*-tolyl)acetamide (3ac)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product as a yellow oil (46.7 mg, 79%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 7.3 Hz, 1H), 7.25 – 7.19 (m, 2H), 7.17 – 7.09 (m, 2H), 7.04 – 6.98 (m, 3H), 4.84 (s, 2H), 4.07 (d, *J* = 11.6 Hz, 1H), 3.62 (d, *J* = 11.6 Hz, 1H), 2.81 (d, *J* = 13.4 Hz, 1H), 2.62 (d, *J* = 13.4 Hz, 1H), 1.98 (s, 3H), 1.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 140.5, 135.6, 133.6, 130.3, 128.6, 127.1, 126.6, 126.5, 125.7, 124.8, 124.4, 122.6, 74.2, 68.9, 48.8, 36.5, 23.4, 17.4.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{21}NO_2$, 296.1645; Found, 296.1660.



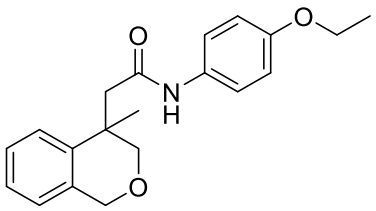
2-(4-methylisochroman-4-yl)-*N*-(4-vinylphenyl)acetamide (3ad)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ad** as a yellow oil (41.8 mg, 68%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.36 (m, 1H), 7.32 – 7.27 (m, 4H), 7.25 – 7.19 (m, 3H), 7.01 – 6.99 (m, 1H), 6.65 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.66 (d, *J* = 17.6 Hz, 1H), 5.17 (d, *J* = 11.0 Hz, 1H), 4.83 (s, 2H), 4.04 (d, *J* = 11.6 Hz, 1H), 3.62 (d, *J* = 11.6 Hz, 1H), 2.75 (d, *J* = 13.5 Hz, 1H), 2.61 (d, *J* = 13.5 Hz, 1H), 1.42 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 140.3, 137.3, 136.1, 133.5, 127.1, 126.7, 125.8, 124.4, 119.7, 112.9, 74.4, 68.9, 49.4, 36.6, 23.0.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{21}NO_2$, 308.1645; Found, 308.1656.



***N*-(4-ethoxyphenyl)-2-(4-methylisochroman-4-yl)acetamide (3ae)**

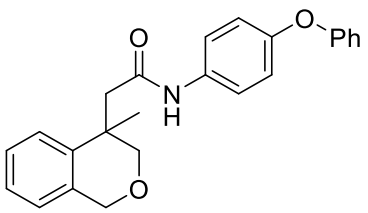
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product products **3ae** as a white solid (49.4 mg, 76%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.35 (m, 1H), 7.24 – 7.18 (m, 4H), 7.13 (s, 1H), 7.00 – 6.98 (m, 1H), 6.79 (d, *J* = 8.9 Hz, 2H), 4.83 (s, 2H), 4.04 (d, *J* = 11.6 Hz, 1H), 3.98 (q, *J* = 7.0 Hz, 2H), 3.61 (d, *J* = 11.6 Hz, 1H), 2.72 (d, *J* = 13.4 Hz, 1H), 2.59 (d, *J* = 13.4 Hz, 1H), 1.42 (s, 3H), 1.38 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 155.6, 140.5, 133.5, 130.7, 127.0, 126.6, 125.9, 124.4, 121.7, 114.6, 74.4, 68.9, 63.6, 48.9, 36.5, 23.0, 14.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{23}NO_3$, 326.1751; Found, 326.1766.

M.p. 112.8 – 114.2 °C



2-(4-methylisochroman-4-yl)-*N*-(4-phenoxyphenyl)acetamide (3af)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA

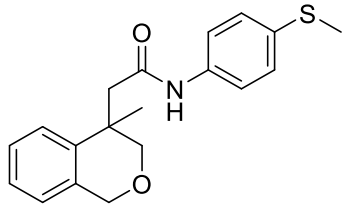
= 5/1, R_f = 0.3) to give the titled product **3af** as a white solid (53.8 mg, 72%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.36 (m, 1H), 7.33 – 7.28 (m, 3H), 7.27 – 7.21 (m, 3H), 7.19 – 7.18 (m, 1H), 7.07 (t, *J* = 7.4 Hz, 1H), 7.02 – 6.99 (m, 1H), 6.97 – 6.90 (m, 4H), 4.84 (s, 2H), 4.04 (d, *J* = 11.6 Hz, 1H), 3.62 (d, *J* = 11.6 Hz, 1H), 2.75 (d, *J* = 13.5 Hz, 1H), 2.61 (d, *J* = 13.4 Hz, 1H), 1.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 157.5, 153.3, 140.4, 133.5, 133.2, 129.7, 127.1, 126.7, 125.8, 124.4, 123.0, 121.6, 119.5, 118.3, 74.4, 68.9, 49.2, 36.5, 23.0.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{24}H_{23}NO_3$, 374.1751; Found, 374.1765.

M.p. 159.8 – 160.9 °C



2-(4-methylisochroman-4-yl)-*N*-(4-(methylthio)phenyl)acetamide (3ag)

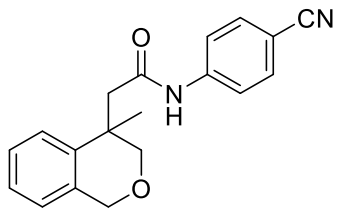
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ag** as a white solid (55.0 mg, 84%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.34 (m, 1H), 7.28 – 7.24 (m, 3H), 7.22 – 7.20 (m, 2H), 7.16 (d, *J* = 8.3 Hz, 2H), 7.00 – 6.98 (m, 1H), 4.85 – 4.77 (m, 2H), 4.02 (d, *J* = 11.6 Hz, 1H), 3.60 (d, *J* = 11.6 Hz, 1H), 2.72 (d, *J* = 13.5 Hz, 1H), 2.60 (d, *J* = 13.5 Hz, 1H), 2.43 (s, 3H), 1.41 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 140.4, 135.4, 133.6, 133.4, 127.9, 127.2, 126.7, 125.9, 124.5, 120.5, 74.5, 68.9, 49.3, 36.6, 23.2, 16.7.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{21}NO_2S$, 328.1366; Found, 328.1378.

M.p. 152.7 – 154.1 °C



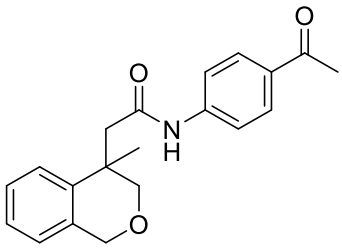
***N*-(4-cyanophenyl)-2-(4-methylisochroman-4-yl)acetamide (3ah)**

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ah** as a yellow oil (42.9 mg, 70%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.6 Hz, 2H), 7.44 – 7.35 (m, 4H), 7.24 – 7.21 (m, 2H), 7.02 – 7.00 (m, 1H), 4.87 – 4.77 (m, 2H), 4.02 (d, *J* = 11.7 Hz, 1H), 3.63 (d, *J* = 11.7 Hz, 1H), 2.78 (d, *J* = 13.6 Hz, 1H), 2.65 (d, *J* = 13.6 Hz, 1H), 1.41 (s, 3H).

^{13}C NMR (11 MHz, $CDCl_3$) δ 169.5, 141.8, 139.9, 133.6, 133.2, 127.3, 127.0, 125.8, 124.6, 119.4, 118.9, 106.9, 74.7, 69.0, 50.1, 36.7, 23.2.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{18}N_2O_2$, 307.1441; Found, 307.1452.



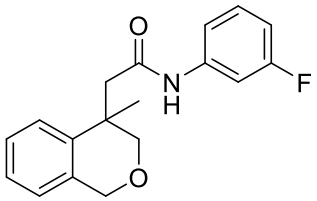
***N*-(4-acetylphenyl)-2-(4-methylisochroman-4-yl)acetamide (3ai)**

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ai** as a yellow oil (53.0 mg, 82%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.7 Hz, 2H), 7.63 (s, 1H), 7.42 (d, *J* = 8.7 Hz, 2H), 7.36 – 7.34 (m, 1H), 7.23 – 7.18 (m, 2H), 6.99 – 6.97 (m, 1H), 4.84 – 4.75 (m, 2H), 4.04 (d, *J* = 11.7 Hz, 1H), 3.61 (d, *J* = 11.7 Hz, 1H), 2.77 (d, *J* = 13.6 Hz, 1H), 2.65 (d, *J* = 13.5 Hz, 1H), 2.54 (s, 3H), 1.41 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 197.0, 169.4, 142.2, 140.0, 133.5, 132.6, 129.6, 127.1, 126.8, 125.7, 124.4, 118.7, 74.4, 68.8, 49.6, 36.6, 26.4, 23.1.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{21}NO_3$, 324.1594; Found, 324.1604.



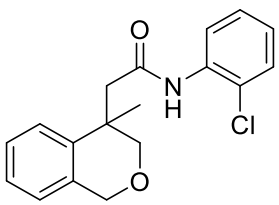
***N*-(3-fluorophenyl)-2-(4-methylisochroman-4-yl)acetamide (3aj)**

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3aj** as a yellow oil (50.9 mg, 85%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.36 – 7.30 (m, 2H), 7.24 – 7.15 (m, 3H), 7.00 – 6.98 (m, 1H), 6.93 (dd, *J* = 8.1, 1.0 Hz, 1H), 6.75 (td, *J* = 8.3, 1.8 Hz, 1H), 4.85 – 4.76 (m, 2H), 4.04 (d, *J* = 11.6 Hz, 1H), 3.61 (d, *J* = 11.6 Hz, 1H), 2.74 (d, *J* = 13.5 Hz, 1H), 2.62 (d, *J* = 13.5 Hz, 1H), 1.41 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 162.8 (d, *J* = 244.5 Hz), 140.1, 139.2 (d, *J* = 10.8 Hz), 133.4, 129.8 (d, *J* = 9.3 Hz), 127.1, 126.7, 125.7, 124.4, 114.9, 110.7 (d, *J* = 21.3 Hz), 107.2 (d, *J* = 26.2 Hz), 74.4, 68.8, 49.3, 36.5, 23.1.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{18}H_{18}FNO_2$, 300.1394; Found, 300.1406.



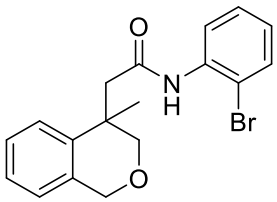
***N*-(2-chlorophenyl)-2-(4-methylisochroman-4-yl)acetamide (3ak)**

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ak** as a yellow oil (52.4 mg, 87%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.2 Hz, 1H), 7.49 (s, 1H), 7.38 (d, *J* = 7.5 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.26 – 7.17 (m, 3H), 7.02 – 6.98 (m, 2H), 4.89 – 4.81 (m, 2H), 4.10 (d, *J* = 11.6 Hz, 1H), 3.63 (d, *J* = 11.6 Hz, 1H), 2.87 (d, *J* = 13.5 Hz, 1H), 2.66 (d, *J* = 13.5 Hz, 1H), 1.45 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.0, 140.2, 134.5, 133.6, 128.9, 127.5, 127.1, 126.7, 125.6, 124.5, 122.6, 121.6, 74.0, 68.9, 49.1, 36.6, 23.2.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{18}H_{18}ClNO_2$, 316.1099; Found, 316.1115.



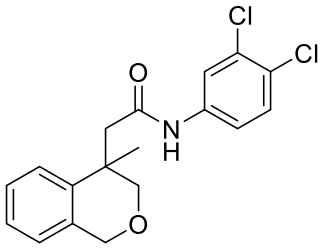
***N*-(2-bromophenyl)-2-(4-methylisochroman-4-yl)acetamide (3al)**

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3al** as a yellow oil (29.5 mg, 41%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, J = 8.1 Hz, 1H), 7.48 – 7.46 (m, 2H), 7.39 – 7.37 (m, 1H), 7.30 – 7.17 (m, 3H), 7.00 – 6.92 (m, 2H), 4.89 – 4.81 (m, 2H), 4.11 (d, J = 11.6 Hz, 1H), 3.62 (d, J = 11.6 Hz, 1H), 2.86 (d, J = 13.5 Hz, 1H), 2.66 (d, J = 13.5 Hz, 1H), 1.45 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.0, 140.3, 135.6, 133.6, 132.1, 128.2, 127.1, 126.7, 125.6, 125.0, 124.4, 121.9, 113.2, 73.9, 68.9, 49.1, 36.6, 23.2.

HRMS (ESI): [M+H] Calcd. for $C_{18}H_{18}BrNO_2$, 360.0594; Found, 360.0605.



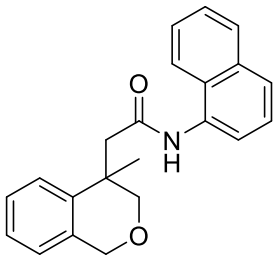
***N*-(3,4-dichlorophenyl)-2-(4-methylisochroman-4-yl)acetamide (3am)**

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3am** as a yellow oil (67.2 mg, 96%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (s, 1H), 7.34 (d, J = 5.5 Hz, 2H), 7.29 – 7.22 (m, 3H), 7.11 – 7.09 (m, 1H), 7.01 – 6.99 (m, 1H), 4.86 – 4.77 (m, 2H), 4.01 (d, J = 11.7 Hz, 1H), 3.61 (d, J = 11.7 Hz, 1H), 2.74 (d, J = 13.5 Hz, 1H), 2.62 (d, J = 13.5 Hz, 1H), 1.41 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 139.9, 137.2, 133.4, 132.5, 130.3, 127.2, 127.1, 126.8, 125.7, 124.5, 121.3, 118.9, 74.5, 68.9, 49.6, 36.6, 23.0.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{18}H_{17}Cl_2NO_2$, 350.0709; Found, 350.0720.



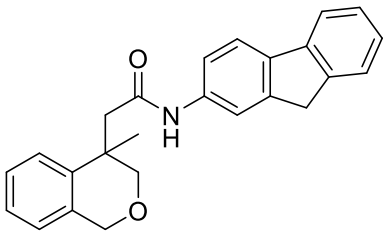
2-(4-methylisochroman-4-yl)-*N*-(naphthalen-1-yl)acetamide (3an)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product **3an** as a yellow oil (55.7 mg, 84%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, J = 20.6, 7.8 Hz, 2H), 7.70 (s, 1H), 7.64 (d, J = 8.2 Hz, 1H), 7.47 – 7.34 (m, 5H), 7.25 – 7.22 (m, 2H), 7.04 – 7.02 (m, 1H), 4.88 (s, 2H), 4.15 (d, J = 11.6 Hz, 1H), 3.67 (d, J = 11.6 Hz, 1H), 2.93 (d, J = 13.5 Hz, 1H), 2.74 (d, J = 13.5 Hz, 1H), 1.46 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.4, 140.5, 133.9, 133.7, 132.2, 128.5, 127.3, 126.7, 126.6, 126.0, 125.8, 125.6, 125.4, 124.6, 120.5, 120.2, 74.3, 69.0, 49.0, 36.6, 23.6.

HRMS (ESI): $[M+H]^+$ Calcd. For $C_{22}H_{21}NO_2$, 332.1645; Found, 332.1656.



***N*-(9*H*-fluoren-2-yl)-2-(4-methylisochroman-4-yl)acetamide (3ao)**

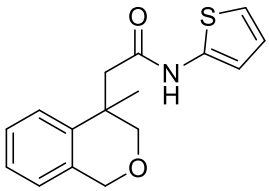
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product **3ao** as a white solid (65.0 mg, 88%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.70 (d, J = 7.5 Hz, 1H), 7.63 (d, J = 8.2 Hz, 1H), 7.50 (d, J = 7.4 Hz, 1H), 7.41 – 7.33 (m, 3H), 7.28 – 7.26 (m, 1H), 7.25 – 7.20 (m, 2H), 7.13 – 7.11 (m, 1H), 7.02 – 7.00 (m, 1H), 4.86 (s, 2H), 4.09 (d, J = 11.6 Hz, 1H), 3.83 (s, 2H), 3.64 (d, J = 11.6 Hz, 1H), 2.79 (d, J = 13.5 Hz, 1H), 2.65 (d, J = 13.5 Hz, 1H), 1.45 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 144.3, 143.2, 141.4, 140.6, 138.0, 136.7, 133.6, 127.2, 126.8, 126.7, 126.3, 125.9, 125.0, 124.5, 120.0, 119.5, 118.6, 116.9, 74.5, 69.0, 49.4, 37.0, 36.7, 23.1.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{25}H_{23}NO_2$, 370.1802; Found, 370.1808.

M.p. 123.7 – 125.2 °C



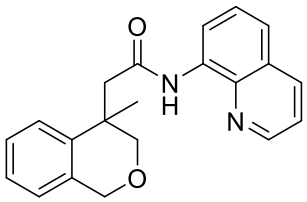
2-(4-methylisochroman-4-yl)-*N*-(thiophen-2-yl)acetamide (3ap)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ap** as a yellow oil (33.3 mg, 58%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.36 – 7.34 (m, 1H), 7.25 – 7.18 (m, 2H), 7.00 (d, J = 6.8 Hz, 1H), 6.84 (d, J = 5.4 Hz, 1H), 6.81 – 6.78 (m, 1H), 6.46 (dd, J = 3.6, 1.1 Hz, 1H), 4.84 (s, 2H), 4.02 (d, J = 11.7 Hz, 1H), 3.61 (d, J = 11.7 Hz, 1H), 2.79 (d, J = 13.7 Hz, 1H), 2.66 (d, J = 13.7 Hz, 1H), 1.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.1, 140.1, 138.7, 133.4, 127.2, 126.8, 125.8, 124.4, 123.7, 117.8, 111.5, 74.5, 68.9, 48.2, 36.5, 22.9.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{16}H_{17}NO_2S$, 288.1053; Found, 288.1064.



2-(4-methylisochroman-4-yl)-*N*-(quinolin-8-yl)acetamide (3aq)

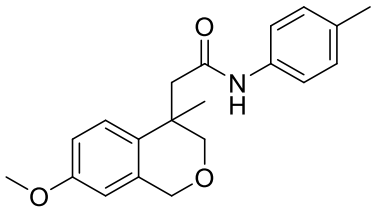
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product **3aq** as a white solid (41.9 mg, 63%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.66 (s, 1H), 8.75 (dd, *J* = 7.3, 1.3 Hz, 1H), 8.71 (dd, *J* = 4.1, 1.4 Hz, 1H), 8.12 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.53 – 7.46 (m, 2H), 7.44 – 7.39 (m, 2H), 7.21 – 7.13 (m, 2H), 6.96 (d, *J* = 7.3 Hz, 1H), 4.90 – 4.81 (m, 2H), 4.21 (d, *J* = 11.5 Hz, 1H), 3.67 (d, *J* = 11.5 Hz, 1H), 3.02 (d, *J* = 13.8 Hz, 1H), 2.80 (d, *J* = 13.8 Hz, 1H), 1.50 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.4, 148.0, 140.7, 138.3, 136.2, 134.4, 133.7, 127.8, 127.3, 126.9, 126.3, 125.8, 124.3, 121.45, 121.36, 116.3, 74.1, 68.9, 48.7, 36.6, 23.6.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{21}H_{20}N_2O_2$, 333.1598; Found, 333.1613.

M.p. 132.8 – 134.7 °C



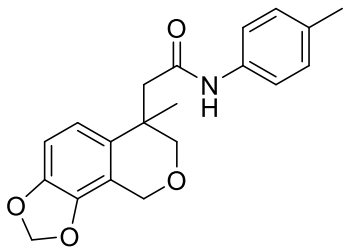
2-(7-methoxy-4-methylisochroman-4-yl)-*N*-(*p*-tolyl)acetamide (3ba)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product **3ba** as a yellow oil (58.5 mg, 90%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.8 Hz, 1H), 7.26 (d, *J* = 8.3 Hz, 2H), 7.20 (s, 1H), 7.10 (d, *J* = 8.2 Hz, 2H), 6.81 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.55 (d, *J* = 2.3 Hz, 1H), 4.83 (s, 2H), 4.05 (d, *J* = 11.6 Hz, 1H), 3.81 (s, 3H), 3.61 (d, *J* = 11.6 Hz, 1H), 2.73 (d, *J* = 13.4 Hz, 1H), 2.62 (d, *J* = 13.4 Hz, 1H), 2.32 (s, 3H), 1.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.0, 158.0, 135.2, 134.8, 133.7, 132.5, 129.3, 127.1, 119.9, 113.2, 108.9, 74.7, 69.0, 55.2, 49.3, 36.0, 23.2, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{23}NO_3$, 326.1751; Found, 326.1765.



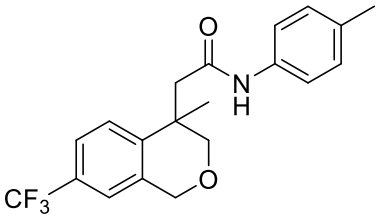
2-(6-methyl-6,9-dihydro-7*H*-[1,3]dioxolo[4,5-h]isochromen-6-yl)-*N*-(*p*-tolyl)acetamide (3ca)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 3/1, R_f = 0.4) to give the titled product as a yellow oil (50.2 mg, 74%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.23 (m, 2H), 7.11 – 7.07 (m, 3H), 6.84 (d, J = 8.2 Hz, 1H), 6.72 (d, J = 8.2 Hz, 1H), 5.95 (s, 2H), 4.87 (d, J = 15.4 Hz, 1H), 4.75 (d, J = 15.4 Hz, 1H), 3.99 (d, J = 11.6 Hz, 1H), 3.55 (d, J = 11.6 Hz, 1H), 2.70 (d, J = 13.4 Hz, 1H), 2.55 (d, J = 13.4 Hz, 1H), 2.29 (s, 3H), 1.39 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 145.5, 142.5, 135.2, 134.8, 133.9, 129.4, 120.0, 118.7, 115.9, 107.2, 101.5, 74.4, 64.4, 49.3, 36.4, 23.3, 20.9.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{21}NO_4$, 340.1543; Found, 340.1559.



2-(4-methyl-7-(trifluoromethyl)isochroman-4-yl)-*N*-(*p*-tolyl)acetamide (3da)

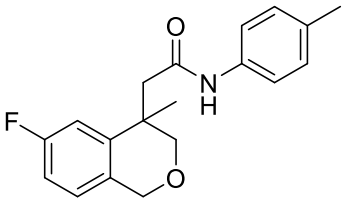
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product **3da** as a white solid (61.8 mg, 85%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.44 (m, 2H), 7.24 (d, J = 8.3 Hz, 2H), 7.20 (s, 1H), 7.09 (d, J = 8.2 Hz, 2H), 4.93 – 4.84 (m, 2H), 4.09 (d, J = 11.7 Hz, 1H), 3.60 (d, J = 11.7 Hz, 1H), 2.73 (d, J = 13.6 Hz, 1H), 2.59 (d, J = 13.6 Hz, 1H), 2.30 (s, 3H), 1.45 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 144.9, 135.0, 134.3, 134.2, 129.5, 128.8 (q, J = 32.6 Hz), 126.7, 124.0 (q, J = 272.1 Hz), 123.8 (q, J = 3.5 Hz), 121.4 (q, J = 3.7 Hz), 120.2, 74.0, 68.7, 48.4, 36.8, 22.7, 20.9.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{20}F_3NO_2$, 364.1519; Found, 364.1533.

M.p. 120.5 – 121.7 °C



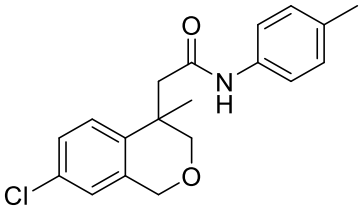
2-(6-fluoro-4-methylisochroman-4-yl)-*N*-(*p*-tolyl)acetamide (3ea)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ea** as a yellow oil (50.1 mg, 80%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (s, 1H), 7.25 (d, J = 8.5 Hz, 2H), 7.08 – 7.04 (m, 3H), 6.97 – 6.93 (m, 1H), 6.89 (td, J = 8.3, 2.3 Hz, 1H), 4.84 – 4.75 (m, 2H), 4.07 (d, J = 11.7 Hz, 1H), 3.57 (d, J = 11.7 Hz, 1H), 2.72 (d, J = 13.6 Hz, 1H), 2.55 (d, J = 13.6 Hz, 1H), 2.29 (s, 3H), 1.40 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 161.7 (d, J = 245.1 Hz), 143.0 (d, J = 6.5 Hz), 135.0, 134.0, 129.4, 129.0 (d, J = 2.8 Hz), 126.0 (d, J = 8.0 Hz), 120.1, 113.8 (d, J = 21.7 Hz), 112.6 (d, J = 21.5 Hz), 73.8, 68.5, 48.6, 36.7, 22.9, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{20}FNO_2$, 314.1551; Found, 314.1565.



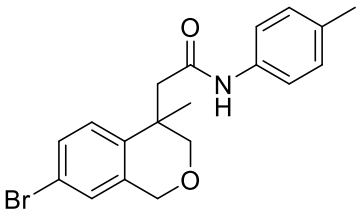
2-(7-chloro-4-methylisochroman-4-yl)-*N*-(*p*-tolyl)acetamide (3fa)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3fa** as a yellow oil (62.7 mg, 95%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (s, 1H), 7.28 – 7.25 (m, 2H), 7.23 (s, 1H), 7.15 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.07 (d, *J* = 8.2 Hz, 2H), 6.97 (d, *J* = 1.3 Hz, 1H), 4.77 (s, 2H), 4.03 (d, *J* = 11.6 Hz, 1H), 3.55 (d, *J* = 11.6 Hz, 1H), 2.67 (d, *J* = 13.5 Hz, 1H), 2.56 (d, *J* = 13.5 Hz, 1H), 2.29 (s, 3H), 1.39 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.6, 139.1, 135.3, 135.0, 133.9, 132.2, 129.3, 127.5, 127.1, 124.2, 120.1, 74.2, 68.4, 48.5, 36.3, 22.8, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{20}ClNO_2$, 330.1255; Found, 330.1264.



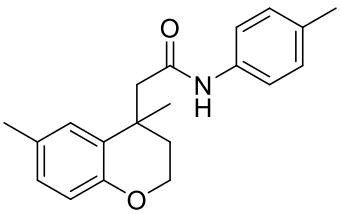
2-(7-bromo-4-methylisochroman-4-yl)-*N*-(*p*-tolyl)acetamide (3ga)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ga** as a yellow oil (59.1 mg, 79%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.26 (m, 2H), 7.22 – 7.16 (m, 3H), 7.09 (s, 1H), 7.04 (d, *J* = 8.1 Hz, 2H), 4.74 (s, 2H), 4.00 (d, *J* = 11.6 Hz, 1H), 3.51 (d, *J* = 11.6 Hz, 1H), 2.64 (d, *J* = 13.5 Hz, 1H), 2.53 (d, *J* = 13.5 Hz, 1H), 2.26 (s, 3H), 1.36 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.6, 139.7, 135.7, 135.0, 133.9, 130.0, 129.4, 127.8, 127.2, 120.3, 120.1, 74.2, 68.3, 48.4, 36.3, 22.7, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{20}BrNO_2$, 374.0750; Found, 374.0760.



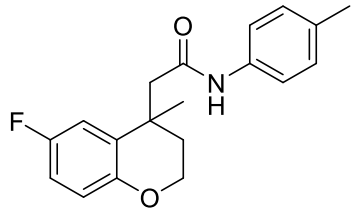
2-(4,6-dimethylchroman-4-yl)-*N*-(*p*-tolyl)acetamide (3ha)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ha** as a yellow oil (47.6 mg, 77%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 – 7.08 (m, 3H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.98 (dd, *J* = 8.3, 1.6 Hz, 1H), 6.84 (s, 1H), 6.78 (d, *J* = 8.3 Hz, 1H), 4.20 – 4.09 (m, 2H), 2.78 (d, *J* = 13.8 Hz, 1H), 2.59 (d, *J* = 13.8 Hz, 1H), 2.28 (s, 6H), 2.26 – 2.20 (m, 1H), 1.92 – 1.86 (m, 1H), 1.49 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 152.3, 134.9, 133.8, 130.0, 129.3, 128.8, 128.4, 126.8, 119.9, 117.6, 62.8, 50.6, 34.7, 33.6, 29.8, 20.8, 20.7.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{23}NO_2$, 310.1802; Found, 310.1812.



2-(6-fluoro-4-methylchroman-4-yl)-*N*-(*p*-tolyl)acetamide (3ia)

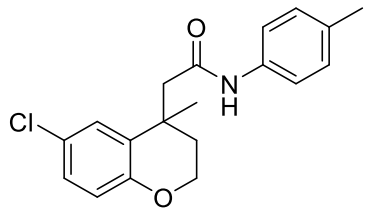
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ia** as a white solid (52.0 mg, 83%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, J = 8.3 Hz, 2H), 7.13 (s, 1H), 7.06 (d, J = 8.2 Hz, 2H), 6.97 (dd, J = 9.6, 2.9 Hz, 1H), 6.87 – 6.82 (m, 1H), 6.80 – 6.77 (m, 1H), 4.21 – 4.09 (m, 2H), 2.68 (d, J = 13.9 Hz, 1H), 2.57 (d, J = 13.9 Hz, 1H), 2.33 – 2.28 (m, 4H), 1.92 – 1.86 (m, 1H), 1.47 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 156.9 (d, J = 238.6 Hz), 150.3 (d, J = 1.4 Hz), 134.8, 134.2, 130.4 (d, J = 6.1 Hz), 129.4, 120.2, 118.6 (d, J = 7.9 Hz), 114.8 (d, J = 23.1 Hz), 112.6 (d, J = 22.9 Hz), 62.9, 49.9, 34.0, 33.9, 29.2, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{20}FNO_2$, 314.1551; Found, 314.1563.

M.p. 134.5 – 136.2 °C



2-(6-chloro-4-methylchroman-4-yl)-*N*-(*p*-tolyl)acetamide (3ja)

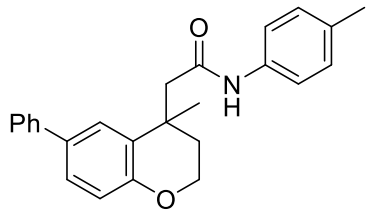
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ja** as a white solid (45.5 mg, 69%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (d, J = 2.5 Hz, 1H), 7.20 (d, J = 8.3 Hz, 2H), 7.17 (s, 1H), 7.10 – 7.05 (m, 3H), 6.77 (d, J = 8.7 Hz, 1H), 4.23 – 4.11 (m, 2H), 2.66 (d, J = 13.9 Hz, 1H), 2.56 (d, J = 13.9 Hz, 1H), 2.34 – 2.29 (m, 4H), 1.92 – 1.86 (m, 1H), 1.47 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 152.8, 134.7, 134.2, 131.0, 129.4, 127.8, 126.4, 125.3, 120.3, 119.0, 62.9, 49.7, 33.9, 33.8, 28.9, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{19}H_{20}ClNO_2$, 330.1255; Found, 330.1270.

M.p. 137.2 – 139.8 °C



2-(4-methyl-6-phenylchroman-4-yl)-*N*-(*p*-tolyl)acetamide (3ka)

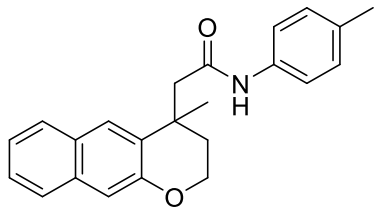
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product **3ka** as a white solid (48.3 mg, 65%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.48 (m, 3H), 7.42 – 7.38 (m, 3H), 7.33 – 7.29 (m, 1H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.3 Hz, 2H), 6.98 (s, 1H), 6.94 (d, *J* = 8.5 Hz, 1H), 4.28 – 4.17 (m, 2H), 2.79 (d, *J* = 13.8 Hz, 1H), 2.65 (d, *J* = 13.8 Hz, 1H), 2.33 – 2.27 (m, 4H), 1.98 – 1.92 (m, 1H), 1.56 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 153.9, 140.7, 134.9, 134.0, 133.9, 129.3, 129.2, 128.7, 126.80, 126.79, 126.7, 125.3, 119.9, 118.1, 62.9, 50.4, 34.5, 33.8, 29.4, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{25}H_{25}NO_2$, 372.1958; Found, 372.1970.

M.p. 155.9 – 157.8 °C



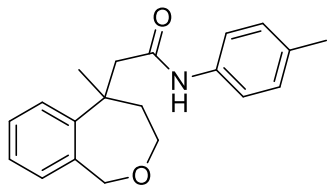
2-(4-methyl-3,4-dihydro-2*H*-benzo[*g*]chromen-4-yl)-*N*-(*p*-tolyl)acetamide (3la)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.3) to give the titled product **3la** as a yellow oil (51.8 mg, 75%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.70 – 7.67 (m, 2H), 7.42 – 7.38 (m, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.26 (s, 1H), 7.07 – 7.00 (m, 4H), 6.83 (s, 1H), 4.35 – 4.23 (m, 2H), 2.85 (d, *J* = 13.8 Hz, 1H), 2.71 (d, *J* = 13.8 Hz, 1H), 2.38 – 2.32 (m, 1H), 2.26 (s, 3H), 2.05 – 1.98 (m, 1H), 1.63 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 152.5, 134.8, 134.0, 133.5, 131.6, 129.3, 128.6, 127.3, 126.3, 126.1, 125.9, 123.7, 120.0, 112.2, 63.1, 50.5, 34.5, 34.2, 29.5, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{23}H_{23}NO_2$, 346.1802; Found, 346.1813.



2-(5-methyl-1,3,4,5-tetrahydrobenzo[*c*]oxepin-5-yl)-*N*-(*p*-tolyl)acetamide (3ma)

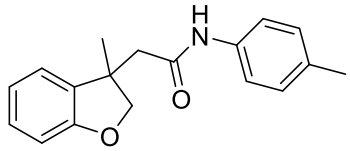
Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3ma** as a yellow oil (26.0 mg, 42%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.8 Hz, 1H), 7.29 – 7.26 (m, 1H), 7.22 (t, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 7.4 Hz, 1H), 7.08 – 7.02 (m, 4H), 6.56 (s, 1H), 4.90 (d, *J* = 14.9 Hz, 1H), 4.77 (d, *J* = 14.9

Hz, 1H), 4.15 – 4.00 (m, 2H), 2.86 (d, J = 13.8 Hz, 1H), 2.78 (d, J = 13.7 Hz, 1H), 2.27 (s, 3H), 2.18 (t, J = 5.7 Hz, 2H), 1.60 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 145.5, 139.4, 135.0, 133.8, 129.5, 129.3, 128.0, 127.8, 126.7, 119.8, 72.6, 67.6, 49.2, 41.6, 39.7, 29.1, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. for $C_{20}H_{23}NO_2$, 310.1802; Found, 310.1812.



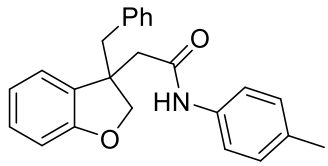
2-(3-methyl-2,3-dihydrobenzofuran-3-yl)-*N*-(*p*-tolyl)acetamide (3na)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3na** as a yellow oil (30.9 mg, 55%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.13 (m, 4H), 7.09 (d, J = 8.2 Hz, 2H), 6.95 – 6.89 (m, 2H), 6.83 (d, J = 8.0 Hz, 1H), 4.71 (d, J = 9.2 Hz, 1H), 4.35 (d, J = 9.2 Hz, 1H), 2.69 (d, J = 14.2 Hz, 1H), 2.62 (d, J = 14.2 Hz, 1H), 2.30 (s, 3H), 1.51 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 159.4, 134.9, 134.21, 134.16, 129.5, 128.9, 122.7, 120.7, 120.2, 110.3, 82.5, 47.8, 44.5, 25.2, 20.9.

HRMS (ESI): $[M+H]^+$ Calcd. For $C_{18}H_{19}NO_2$, 282.1489; Found, 282.1502.



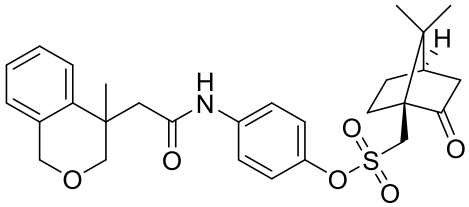
2-(3-benzyl-2,3-dihydrobenzofuran-3-yl)-*N*-(*p*-tolyl)acetamide (3oa)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1, R_f = 0.4) to give the titled product **3oa** as a yellow oil (56.5 mg, 79%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (s, 1H), 7.22 – 7.20 (m, 4H), 7.19 – 7.15 (m, 1H), 7.09 (d, J = 8.2 Hz, 2H), 7.00 – 6.97 (m, 2H), 6.95 (s, 1H), 6.88 – 6.83 (m, 2H), 6.77 (d, J = 8.0 Hz, 1H), 4.70 (d, J = 9.4 Hz, 1H), 4.48 (d, J = 9.4 Hz, 1H), 3.19 (d, J = 13.3 Hz, 1H), 3.13 (d, J = 13.3 Hz, 1H), 2.71 (s, 2H), 2.30 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 159.7, 136.8, 134.8, 134.2, 131.9, 130.6, 129.5, 129.0, 128.0, 126.6, 124.1, 120.1, 110.1, 81.2, 48.6, 44.5, 43.6, 20.8.

HRMS (ESI): $[M+H]^+$ Calcd. For $C_{24}H_{23}NO_2$, 358.1802; Found, 358.1813.



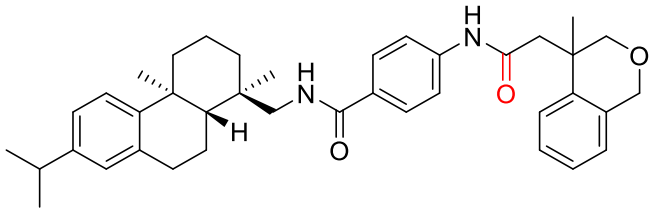
4-(2-(4-methylisochroman-4-yl)acetamido)phenyl ((1*S*,4*R*)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan -1-yl)methanesulfonate (5)

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 3/1, R_f = 0.4) to give the titled product **5** as a yellow oil (89.0 mg, 87%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.36 – 7.34 (m, 3H), 7.23 – 7.18 (m, 2H), 7.17 – 7.15 (m, 2H), 6.99 – 6.97 (m, 1H), 4.80 (s, 2H), 4.03 (d, *J* = 11.6 Hz, 1H), 3.75 (d, *J* = 15.0 Hz, 1H), 3.60 (d, *J* = 11.6 Hz, 1H), 3.16 (d, *J* = 15.0 Hz, 1H), 2.73 (d, *J* = 13.6 Hz, 1H), 2.61 (d, *J* = 13.6 Hz, 1H), 2.54 – 2.46 (m, 1H), 2.43 – 2.36 (m, 1H), 2.12 (t, *J* = 4.5 Hz, 1H), 2.09 – 2.02 (m, 1H), 1.95 (d, *J* = 18.5 Hz, 1H), 1.73 – 1.66 (m, 1H), 1.46 – 1.42 (m, 1H), 1.40 (s, 3H), 1.13 (s, 3H), 0.88 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 214.0, 169.1, 144.8, 140.1, 136.7, 133.5, 127.0, 126.6, 125.7, 124.4, 122.4, 120.9, 74.4, 68.8, 58.0, 49.0, 47.9, 47.3, 42.7, 42.3, 36.5, 26.7, 25.0, 23.2, 19.8, 19.6.

HRMS (ESI): $[M+H]^+$ Calcd. For $C_{28}H_{33}NO_6S$, 512.2101; Found, 512.2109.



***N*-(((1*R*,4a*S*,10a*R*)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl)methylisochroman-4-yl)acetamido)benzamide (7)**

Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 3/1, R_f = 0.3) to give the titled product **7** as a yellow oil (84.2 mg, 71%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.5 Hz, 2H), 7.54 (s, 1H), 7.37 – 7.33 (m, 3H), 7.23 – 7.16 (m, 3H), 7.01 – 6.98 (m, 2H), 6.89 (s, 1H), 6.16 (t, *J* = 6.2 Hz, 1H), 4.84 – 4.76 (m, 2H), 4.04 (d, *J* = 11.6 Hz, 1H), 3.62 (d, *J* = 11.6 Hz, 1H), 3.42 – 3.30 (m, 2H), 2.95 – 2.89 (m, 1H), 2.86 – 2.79 (m, 2H), 2.75 (d, *J* = 13.6 Hz, 1H), 2.64 (d, *J* = 13.5 Hz, 1H), 2.30 (d, *J* = 12.8 Hz, 1H), 1.96 (t, *J* = 9.9 Hz, 1H), 1.84 – 1.66 (m, 3H), 1.49 (t, *J* = 12.3 Hz, 2H), 1.41 (s, 3H), 1.39 – 1.31 (m, 2H), 1.23 (s, 6H), 1.22 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 167.0, 147.0, 145.5, 140.6, 140.1, 134.7, 133.6, 123.0, 127.7, 127.1, 126.9, 126.7, 125.7, 124.4, 124.2, 123.8, 119.1, 74.5, 68.8, 50.3, 49.4, 45.8, 38.3, 37.6, 37.5, 36.6, 36.3, 33.3, 30.4, 25.4, 23.9, 23.2, 19.0, 18.7, 18.6.

HRMS (ESI): $[M+H]^+$ Calcd. For $C_{39}H_{48}N_2O_3$, 593.3738; Found, 593.3746.

5. Reference

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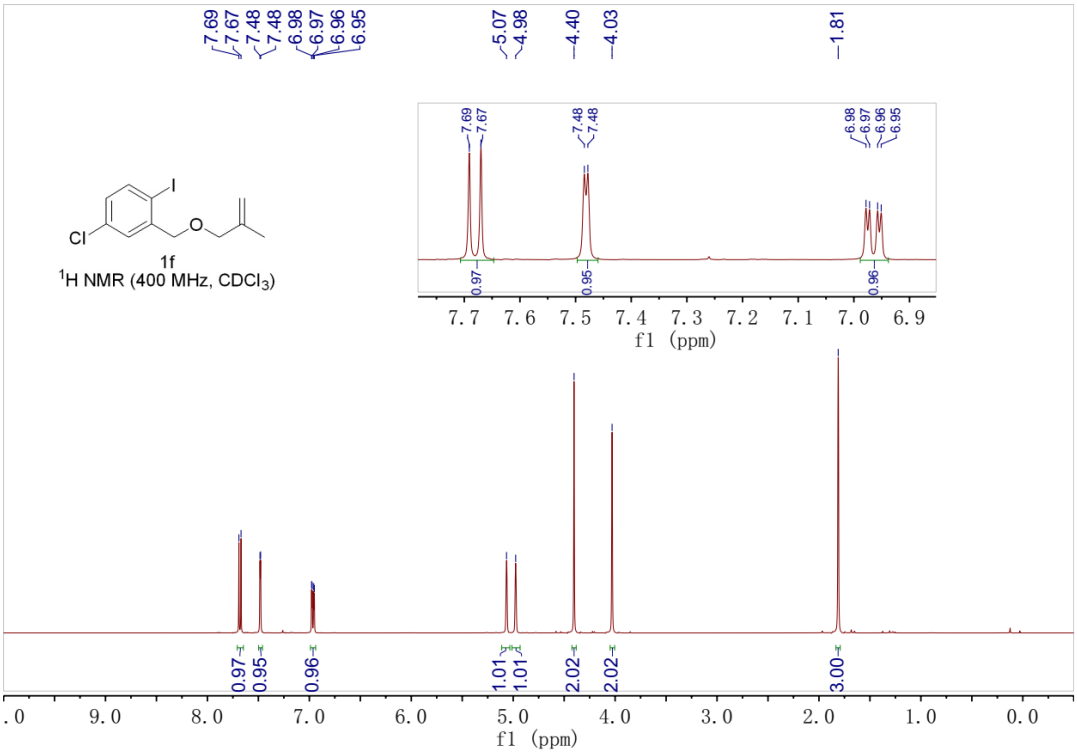
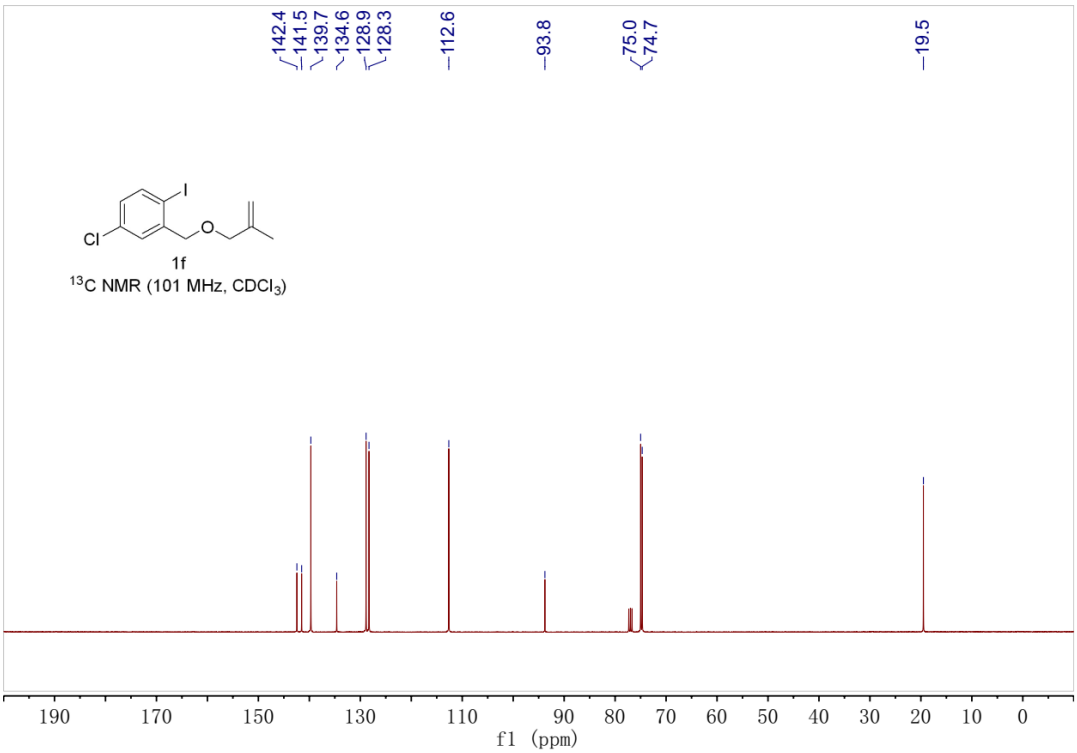
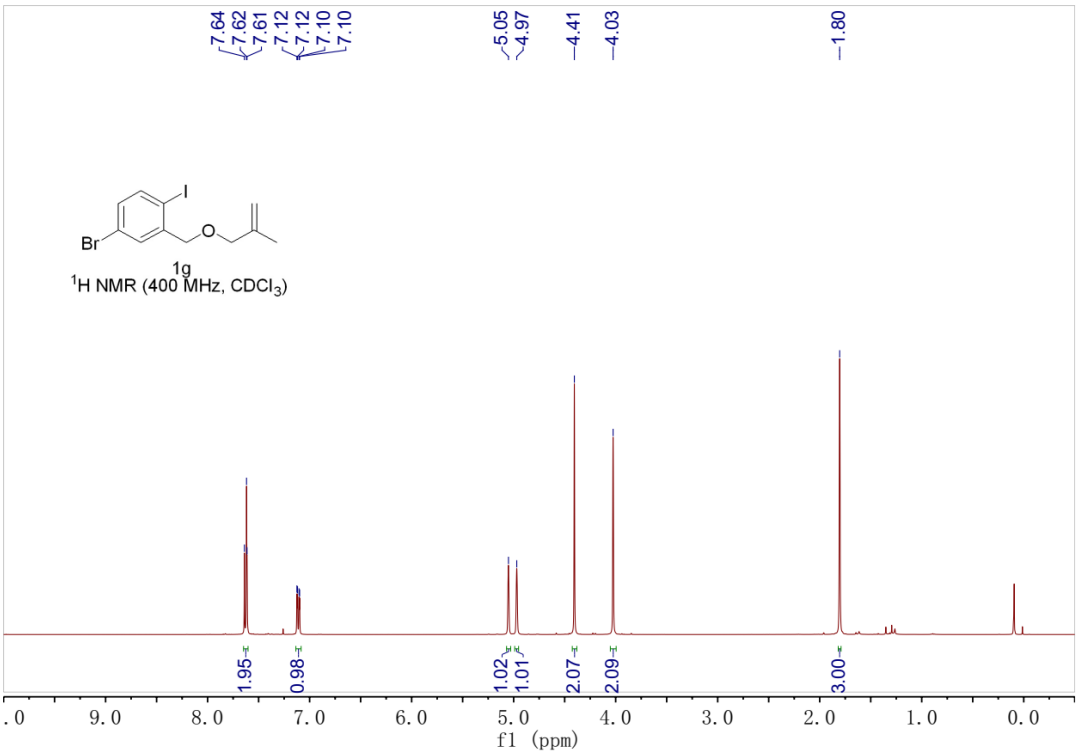
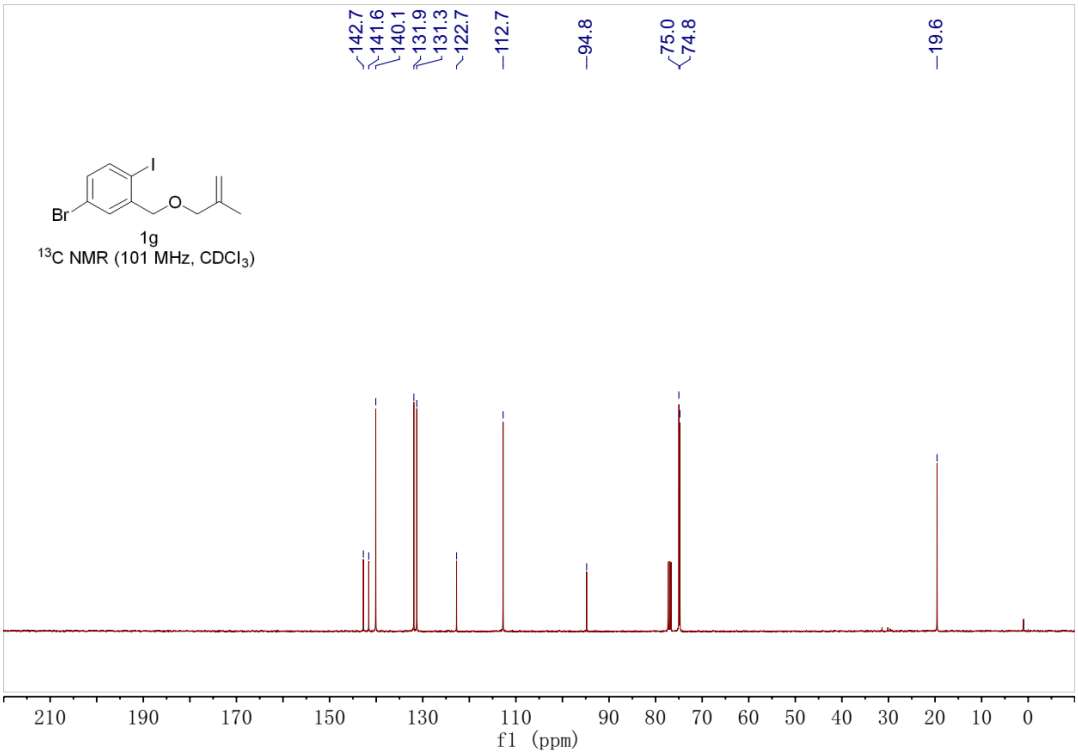
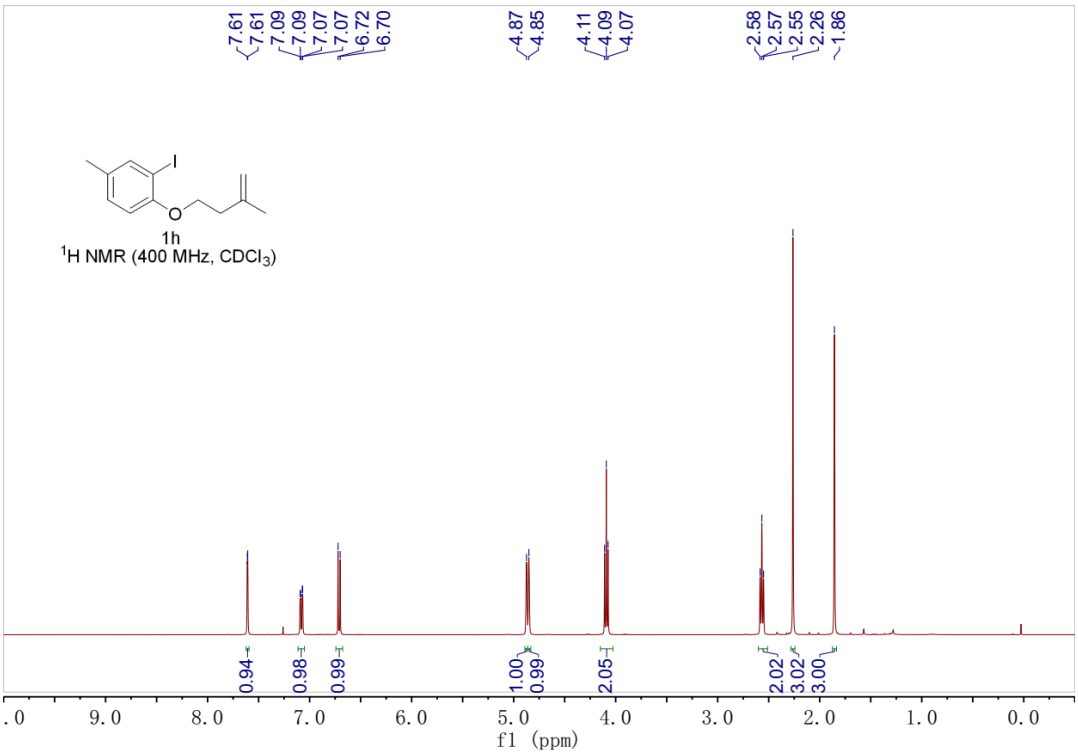
6. Copy of ^{1}H and ^{13}C NMR Spectra of Compounds 1f, 1g, 1h, 1i

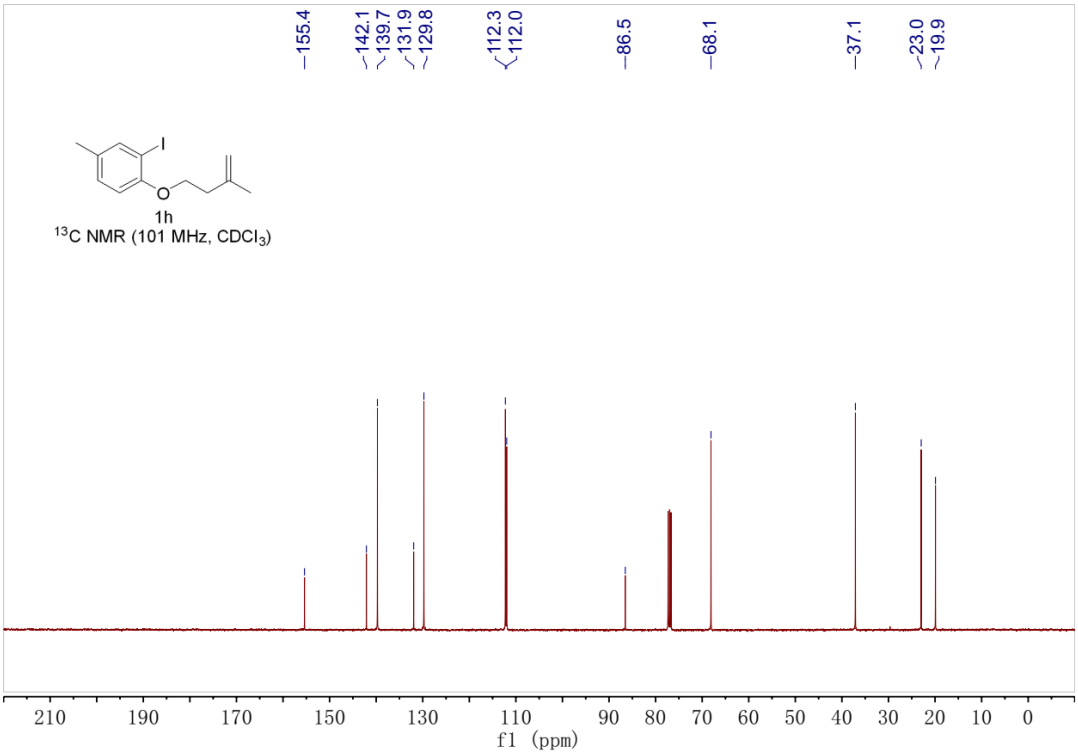


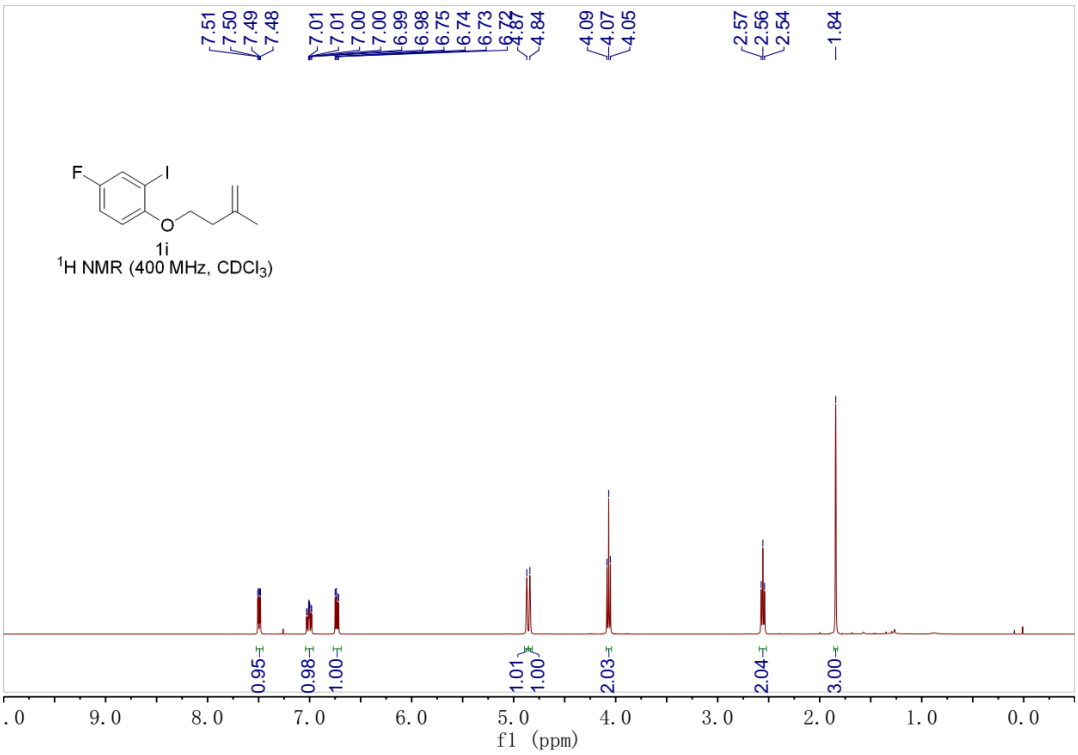


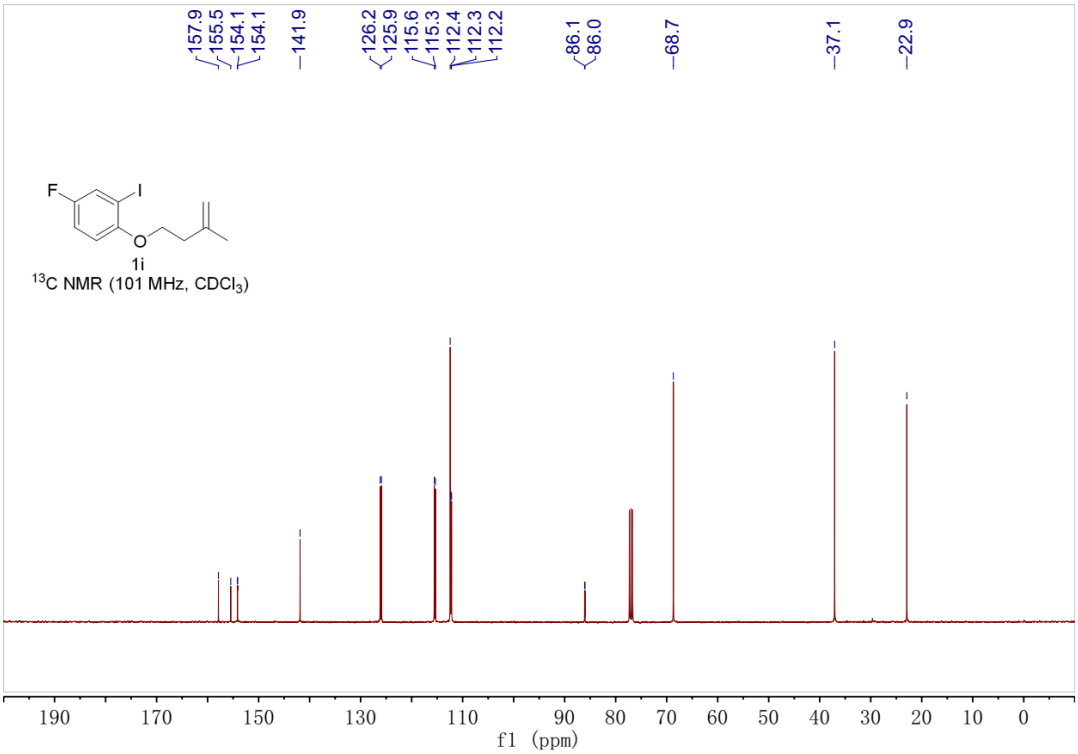

7.64
7.62
7.61
7.12
7.12
7.10
7.10
5.05
4.97
4.41
4.03
1.80
I
Br
O
1g
^{1}H NMR (400 MHz, CDCl$_3$)
1.95
0.98
1.02
1.01
2.07
2.09
3.00
f1 (ppm)


142.7
141.6
140.1
131.9
131.3
122.7
112.7
94.8
75.0
74.8
19.6
I
Br
O
1g
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

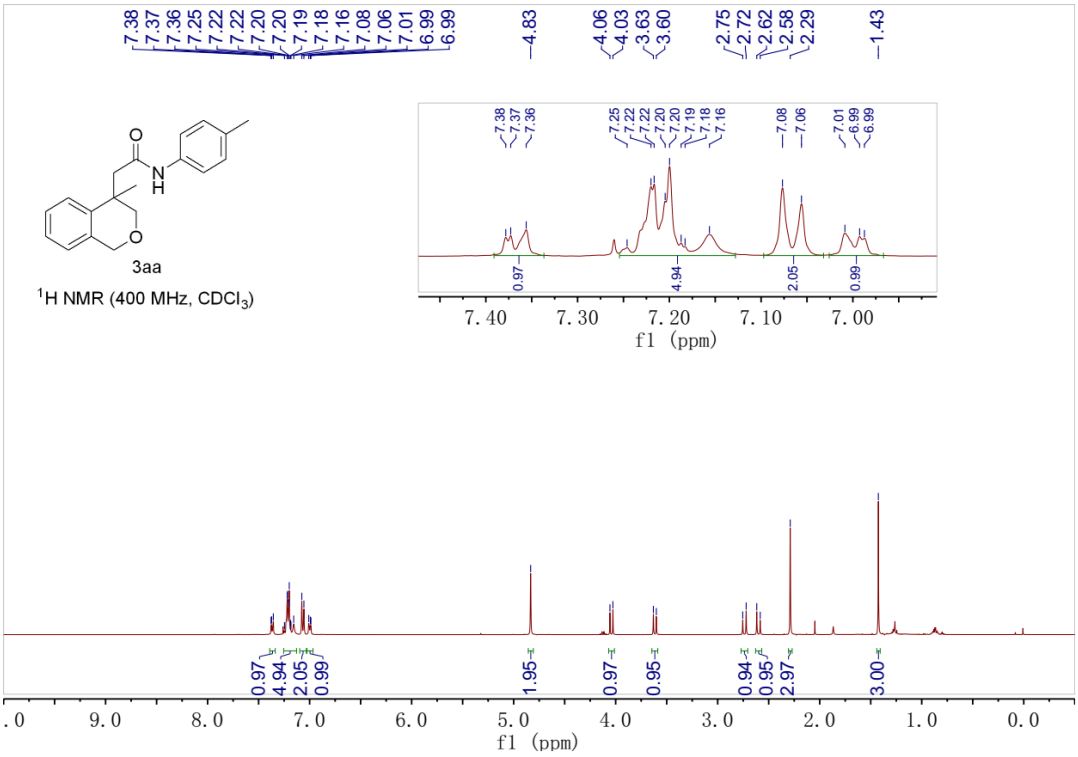

7.61
7.61
7.09
7.09
7.07
7.07
6.72
6.70
4.87
4.85
4.11
4.09
4.07
2.58
2.57
2.55
2.26
1.86
I
O
1h
1H NMR (400 MHz, CDCl3)
0.94
0.98
0.99
1.00
0.99
2.05
2.02
3.02
3.00
f1 (ppm)

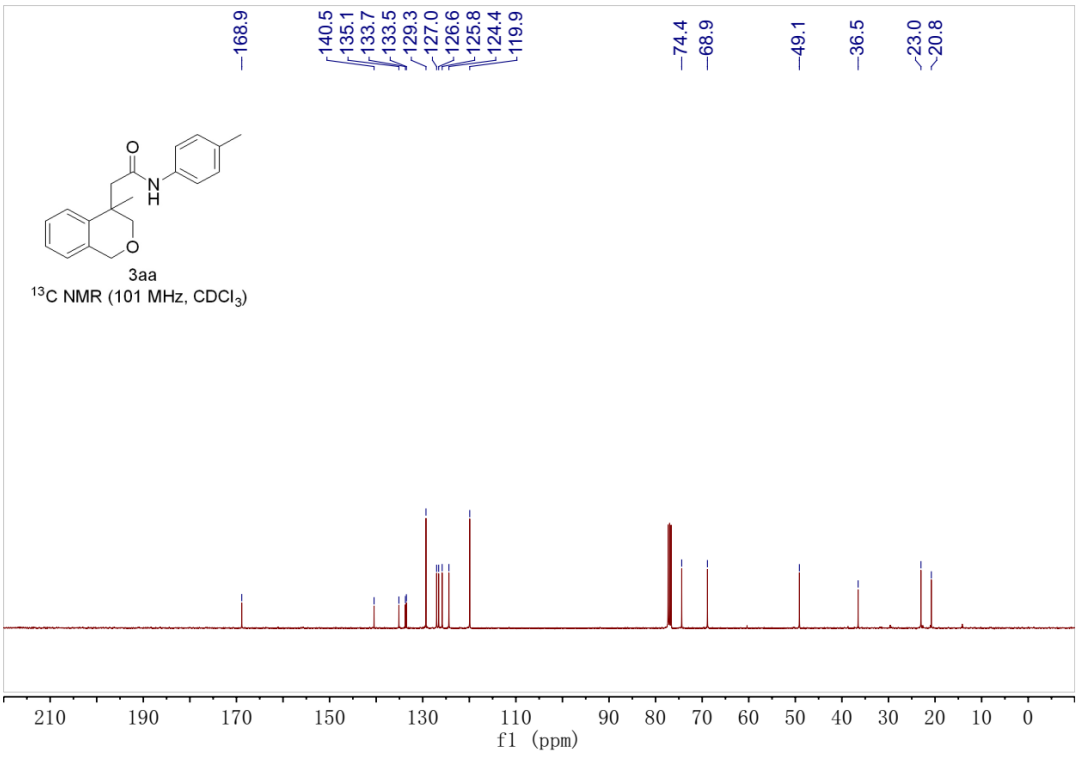

155.4
142.1
139.7
131.9
129.8
112.3
112.0
86.5
68.1
37.1
23.0
19.9
I
O
1h
13C NMR (101 MHz, CDCl3)
f1 (ppm)

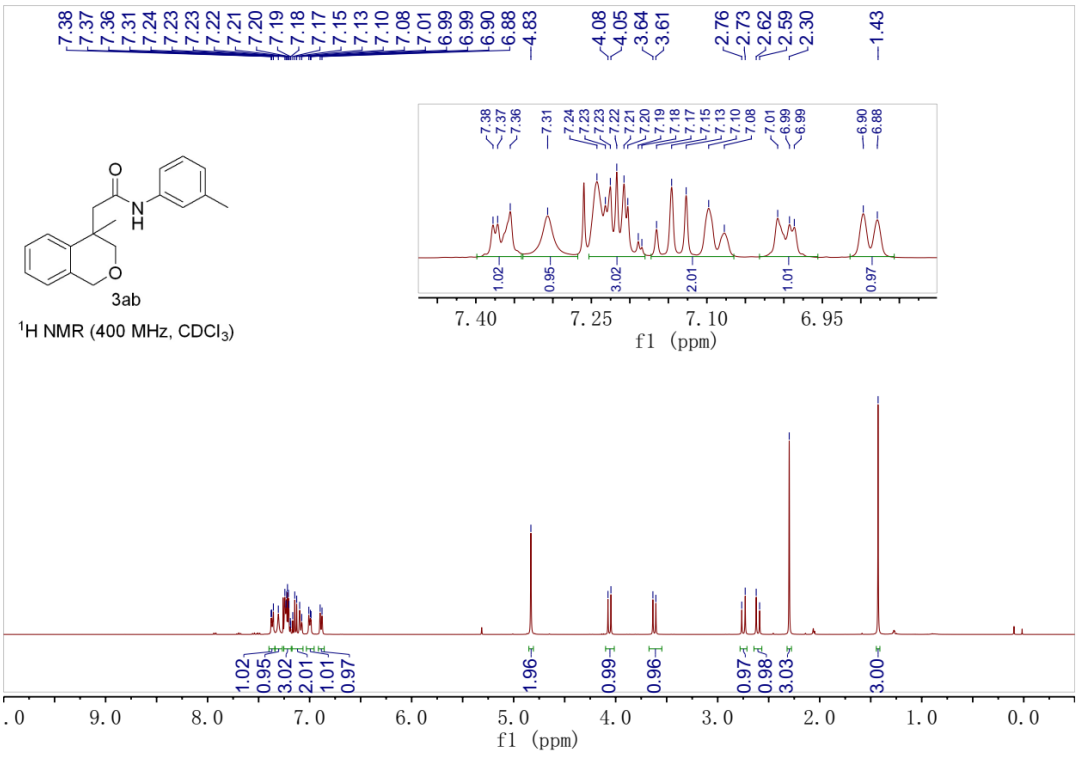

7.51
7.50
7.49
7.48
7.01
7.01
7.00
7.00
6.99
6.98
6.75
6.74
6.73
4.87
4.84
4.09
4.07
4.05
2.57
2.56
2.54
1.84
F
I
O
1i
^{1}H NMR (400 MHz, CDCl$_3$)
0.95
0.98
1.00
1.01
1.00
2.03
2.04
3.00
f1 (ppm)


157.9
155.5
154.1
154.1
141.9
126.2
125.9
115.6
115.3
112.4
112.3
112.2
86.1
86.0
68.7
37.1
22.9
F
I
O
1i
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

7. Copy of ^{1}H and ^{13}C NMR Spectra of Products



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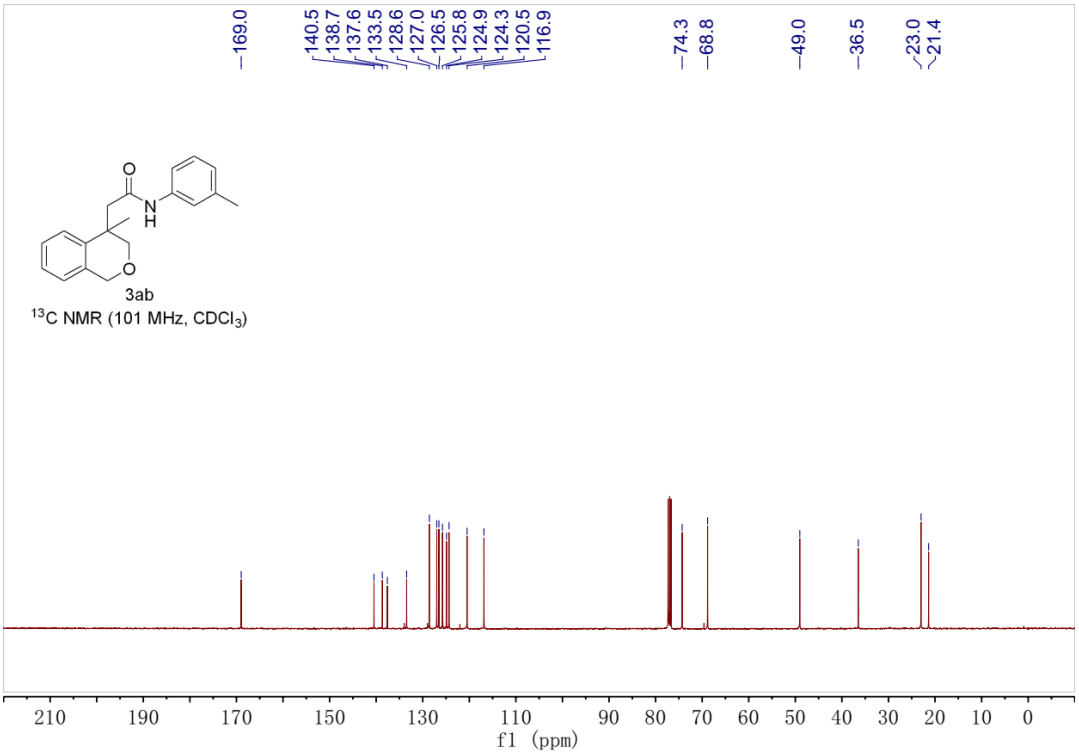
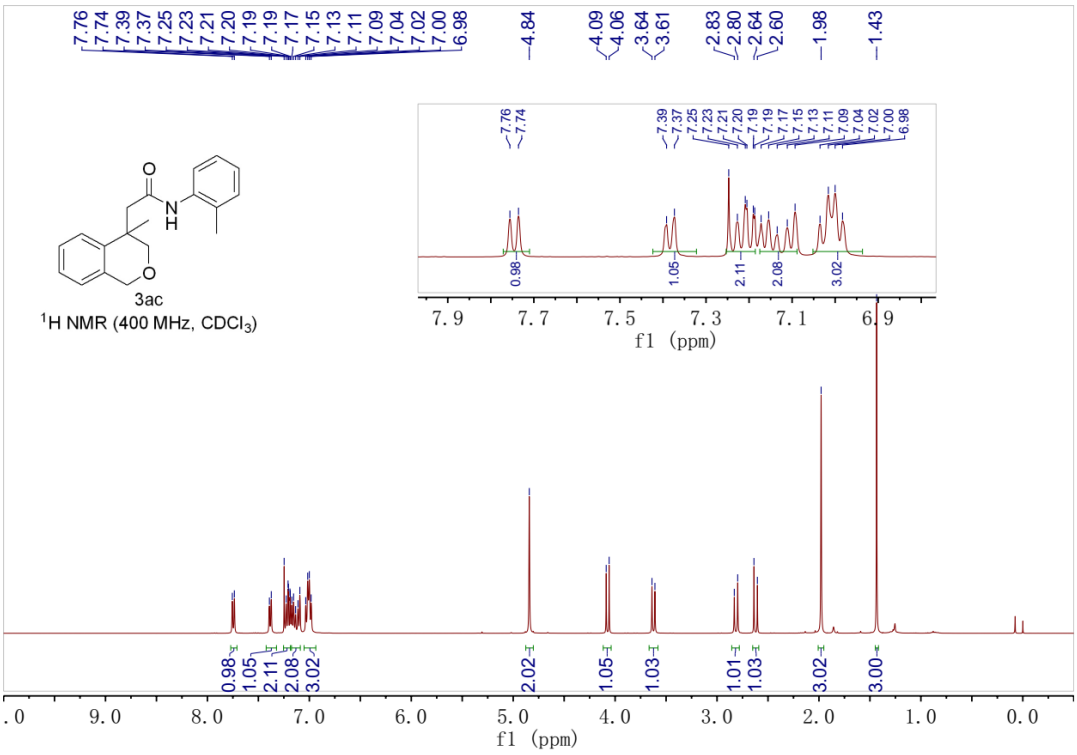
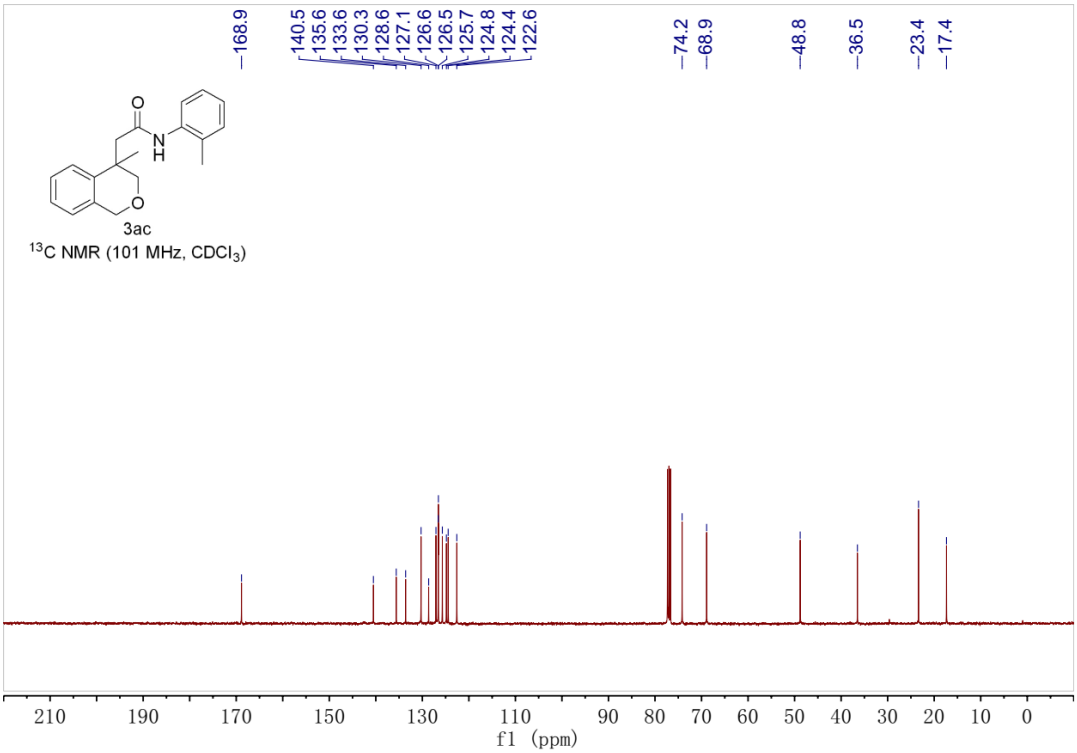
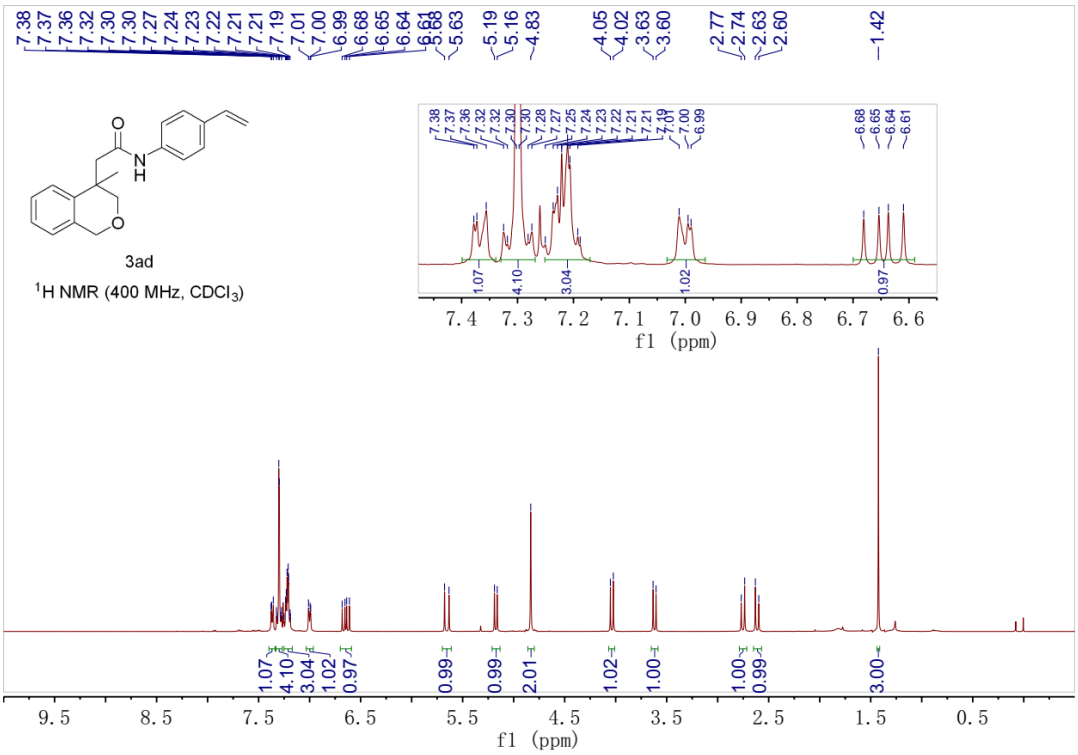
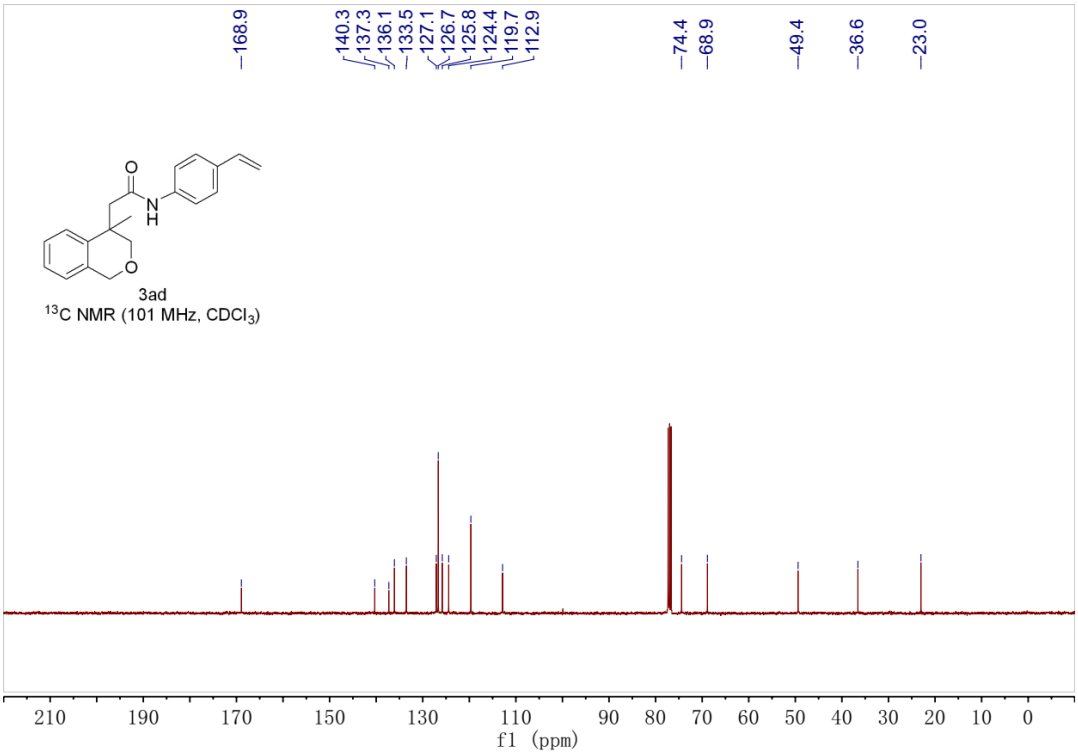
3ab
1H NMR (400 MHz, CDCl3)
1.02 0.95 3.02 2.01 1.01 0.97 1.96 0.99 0.96 0.97 0.98 3.03 3.00
f1 (ppm)

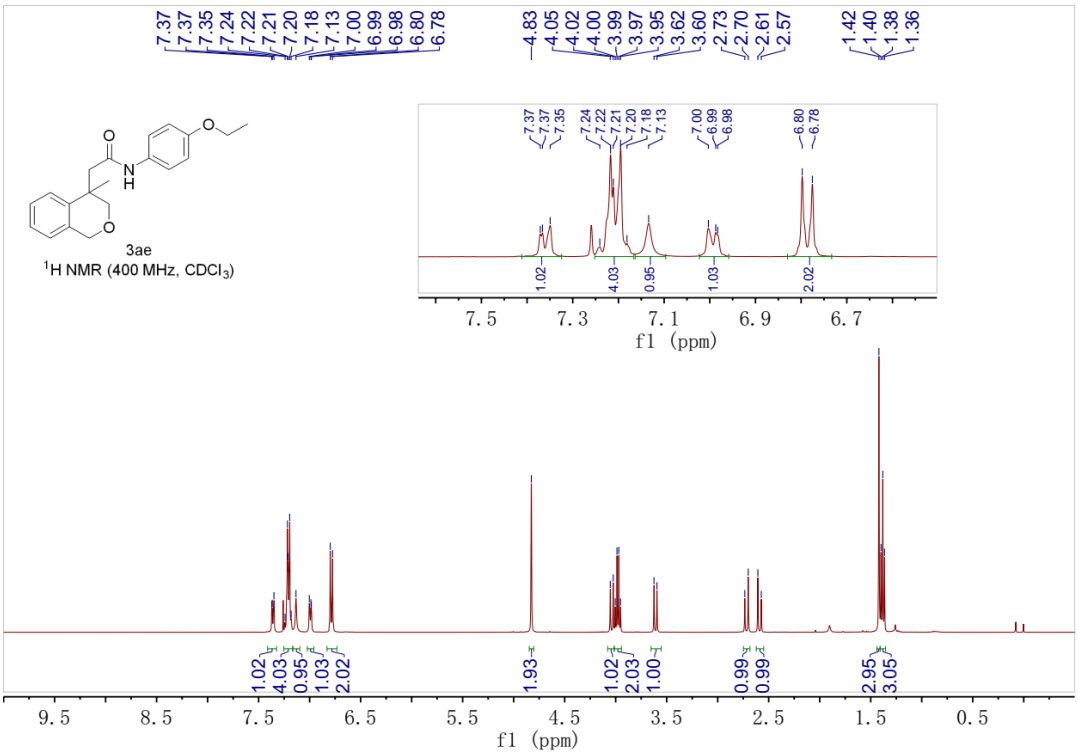

169.0 140.5 138.7 137.6 133.5 128.6 127.0 126.5 125.8 124.9 124.3 120.5 116.9 74.3 68.8 49.0 36.5 23.0 21.4
3ab
13C NMR (101 MHz, CDCl3)
f1 (ppm)

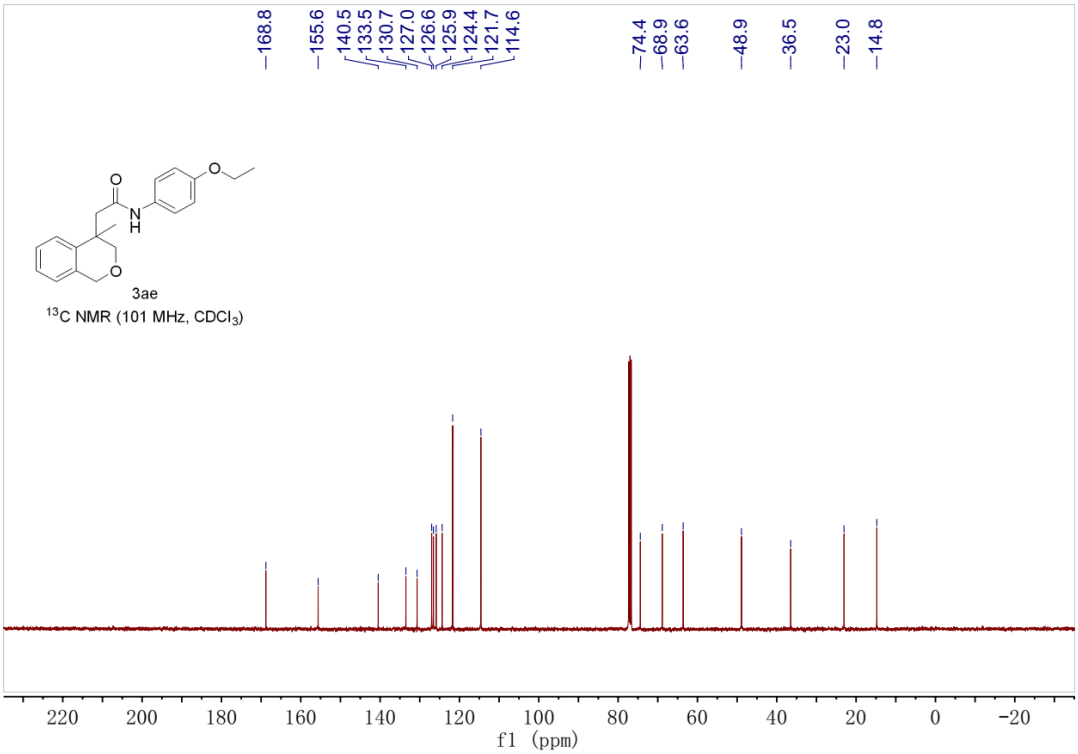

7.76
7.74
7.39
7.37
7.25
7.23
7.21
7.20
7.19
7.19
7.17
7.15
7.13
7.11
7.09
7.04
7.02
7.00
6.98
4.84
4.09
4.06
3.64
3.61
2.83
2.80
2.64
2.60
1.98
1.43
O
N
H
O
3ac
^{1}H NMR (400 MHz, CDCl$_3$)
0.98
1.05
2.11
2.08
3.02
2.02
1.05
1.03
1.01
1.03
3.02
3.00
f1 (ppm)

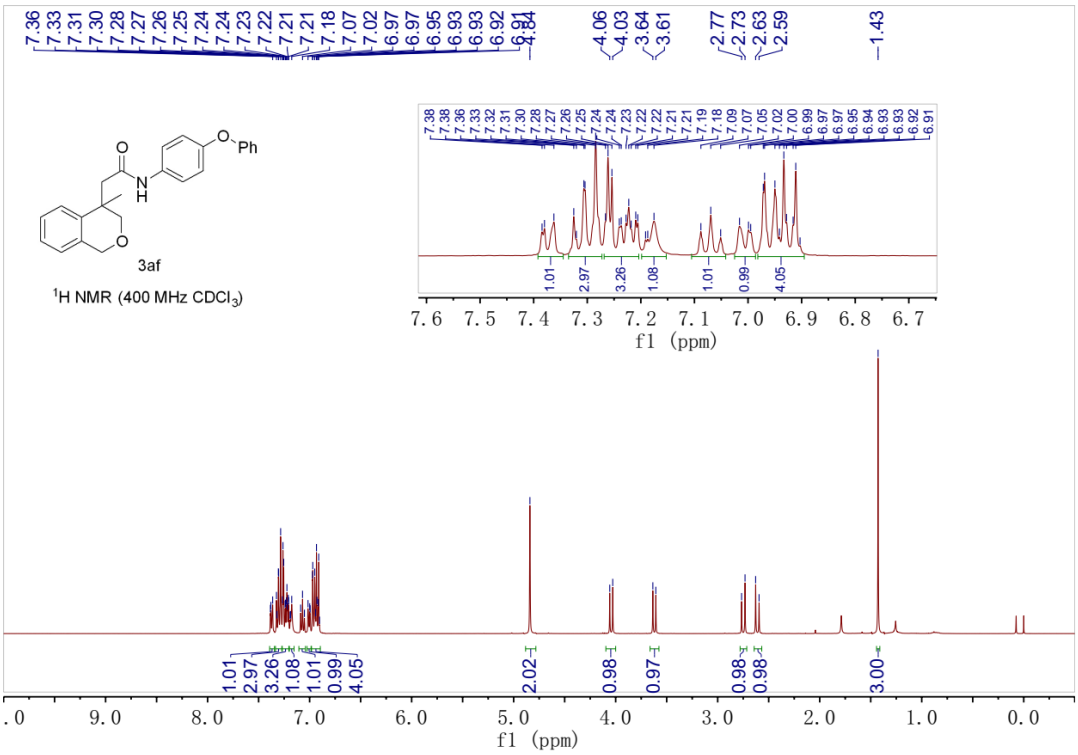

168.9
140.5
135.6
133.6
130.3
128.6
127.1
126.6
126.5
125.7
124.8
124.4
122.6
74.2
68.9
48.8
36.5
23.4
17.4
O
N
H
O
3ac
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

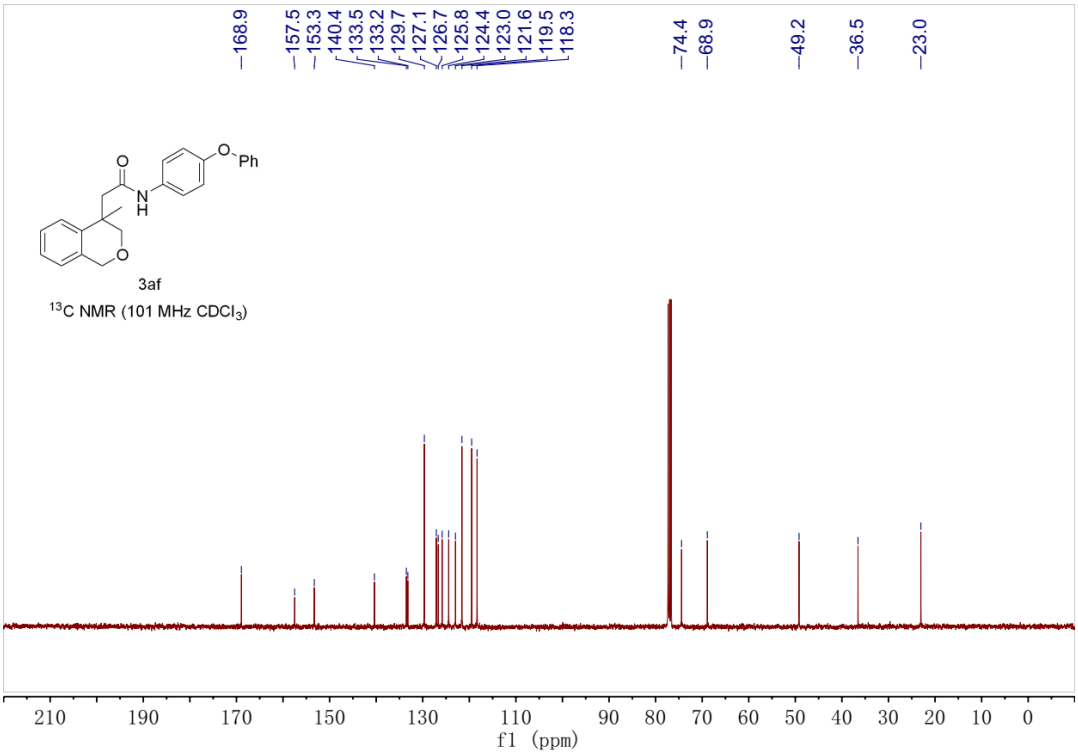

7.38 7.37 7.36 7.32 7.30 7.30 7.27 7.24 7.23 7.22 7.21 7.21 7.19 7.01 7.00 6.99 6.68 6.65 6.64 6.61 5.63 5.19 5.16 4.83 4.05 4.02 3.63 3.60 2.77 2.74 2.63 2.60 1.42
3ad
1H NMR (400 MHz, CDCl3)
1.07 4.10 3.04 1.02 0.97 0.99 0.99 2.01 1.02 1.00 1.00 0.99 3.00
f1 (ppm)

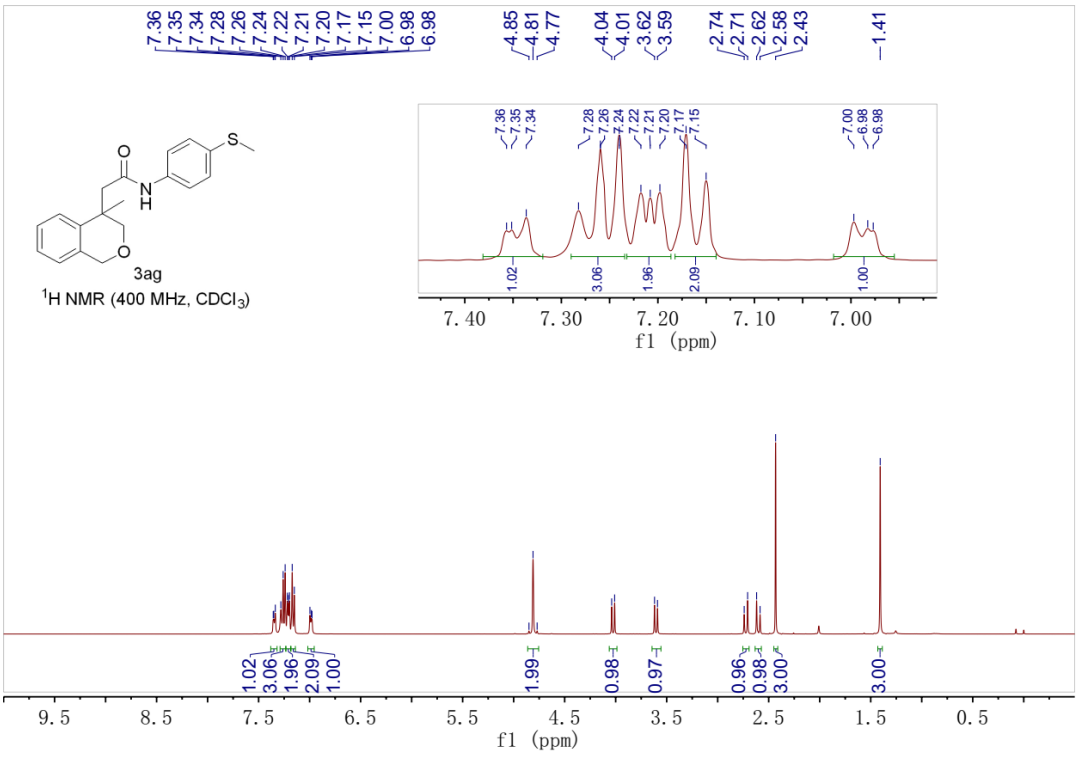

168.9 140.3 137.3 136.1 133.5 127.1 126.7 125.8 124.4 119.7 112.9 74.4 68.9 49.4 36.6 23.0
3ad
13C NMR (101 MHz, CDCl3)
f1 (ppm)

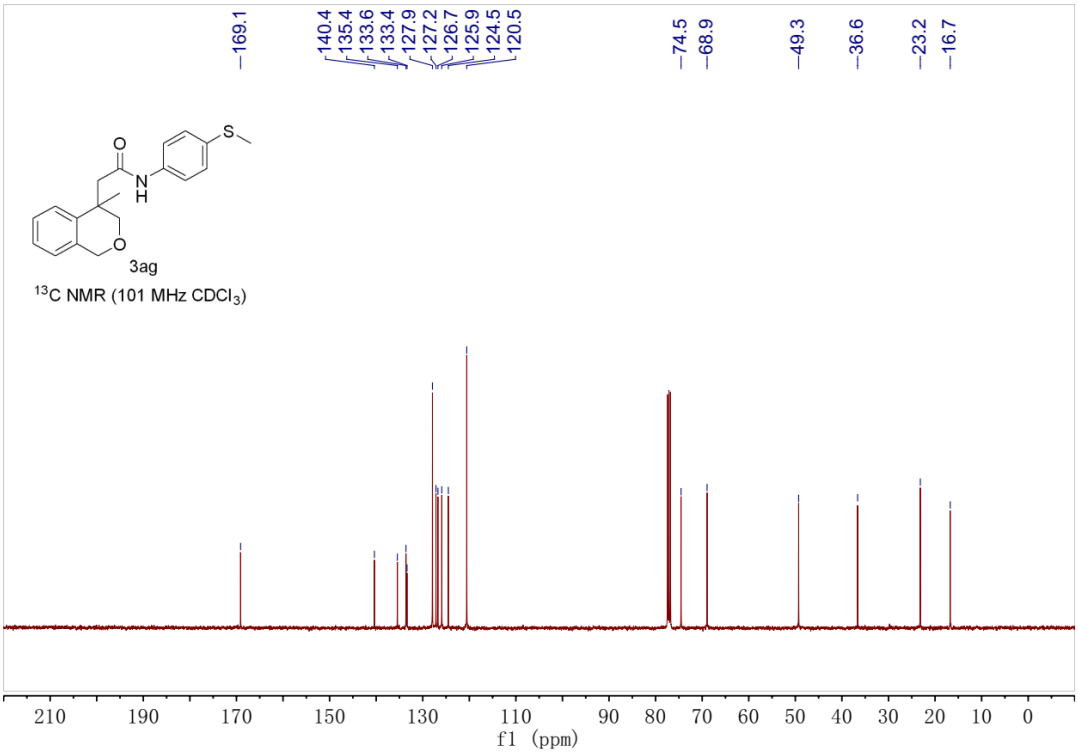

7.37
7.37
7.35
7.24
7.22
7.21
7.20
7.18
7.13
7.00
6.99
6.98
6.80
6.78
4.83
4.05
4.02
4.00
3.99
3.97
3.95
3.62
3.60
2.73
2.70
2.61
2.57
1.42
1.40
1.38
1.36
O
N
H
O
O
3ae
1H NMR (400 MHz, CDCl3)
1.02
4.03
0.95
1.03
2.02
7.5
7.3
7.1
6.9
6.7
f1 (ppm)
1.02
4.03
0.95
1.03
2.02
1.93
1.02
2.03
1.00
0.99
0.99
2.95
3.05
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


168.8
155.6
140.5
133.5
130.7
127.0
126.6
125.9
124.4
121.7
114.6
74.4
68.9
63.6
48.9
36.5
23.0
14.8
O
N
H
O
O
3ae
13C NMR (101 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
−20
f1 (ppm)


7.36 7.33 7.31 7.30 7.28 7.27 7.26 7.25 7.24 7.24 7.23 7.22 7.21 7.21 7.18 7.07 7.02 6.97 6.97 6.95 6.93 6.93 6.92 6.91 4.06 4.03 3.64 3.61 2.77 2.73 2.63 2.59 1.43
O
N
H
O
Ph
O
3af
^{1}H NMR (400 MHz CDCl$_3$)
1.01 2.97 3.26 1.08 1.01 0.99 4.05 2.02 0.98 0.97 0.98 0.98 3.00
f1 (ppm)


168.9 157.5 153.3 140.4 133.5 133.2 129.7 127.1 126.7 125.8 124.4 123.0 121.6 119.5 118.3 74.4 68.9 49.2 36.5 23.0
O
N
H
O
Ph
O
3af
^{13}C NMR (101 MHz CDCl$_3$)
f1 (ppm)


7.36
7.35
7.34
7.28
7.26
7.24
7.22
7.21
7.20
7.17
7.15
7.00
6.98
6.98
4.85
4.81
4.77
4.04
4.01
3.62
3.59
2.74
2.71
2.62
2.58
2.43
1.41
3ag
1H NMR (400 MHz, CDCl3)
1.02
3.06
1.96
2.09
1.00
1.99
0.98
0.97
0.96
0.98
3.00
3.00
f1 (ppm)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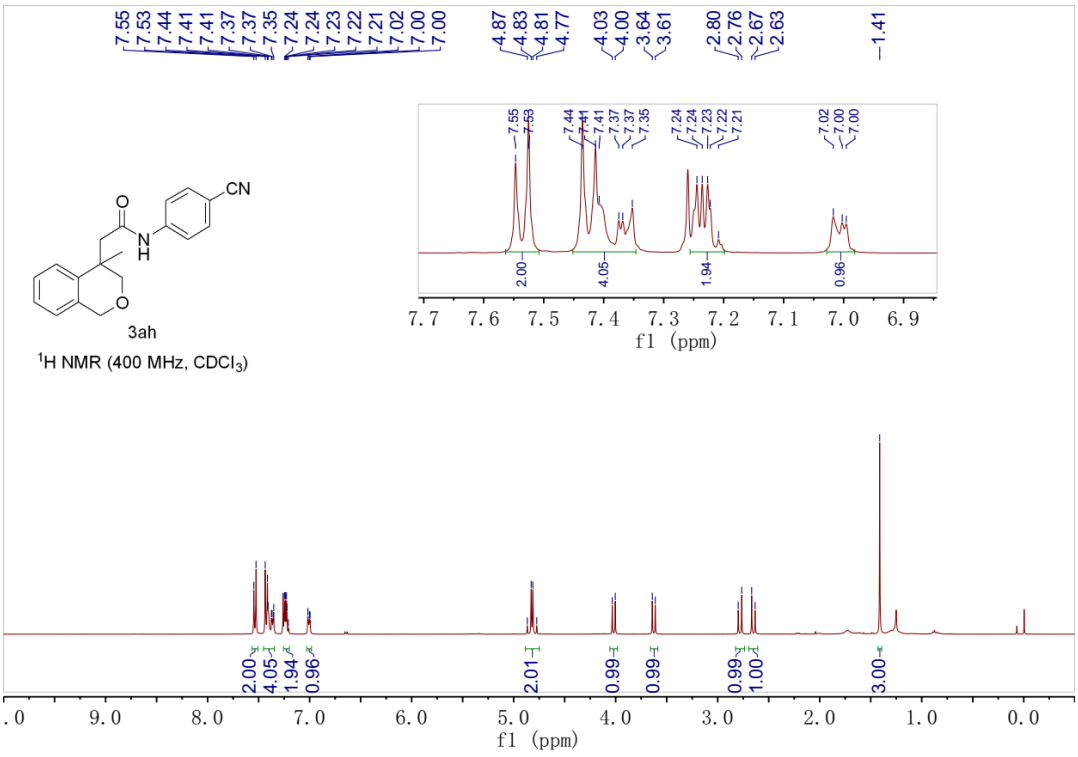
140.4
135.4
133.6
133.4
127.9
127.2
126.7
125.9
124.5
120.5
74.5
68.9
49.3
36.6
23.2
16.7
3ag
13C NMR (101 MHz CDCl3)
f1 (ppm)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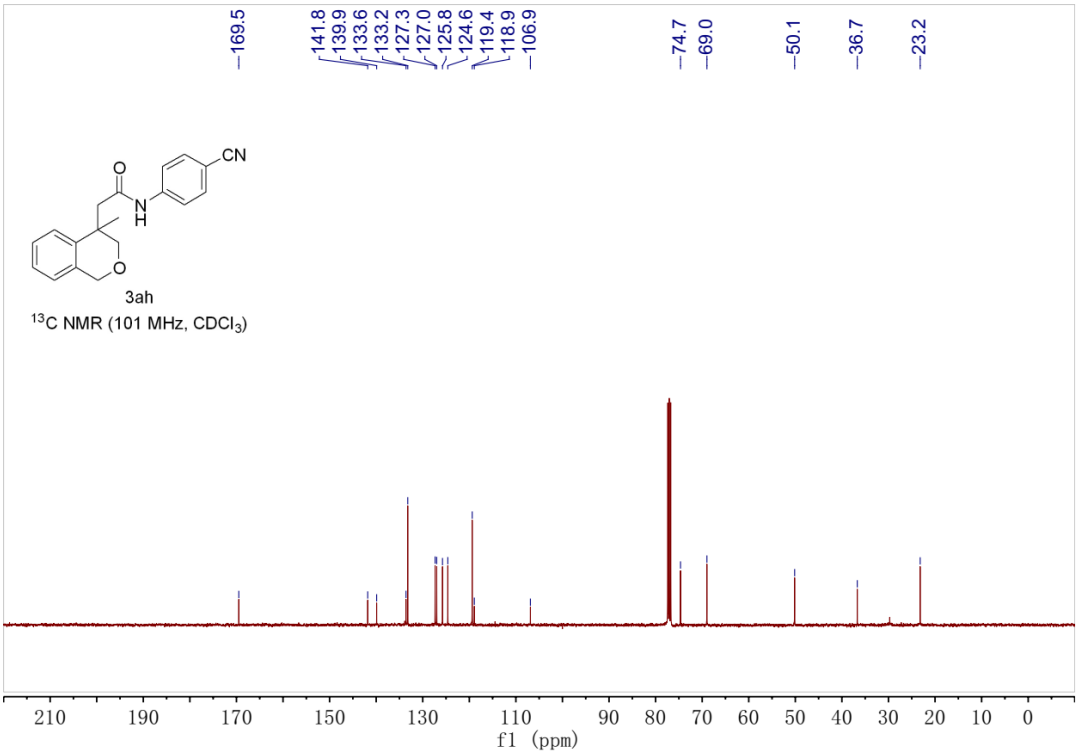
7.53
7.44
7.41
7.41
7.37
7.37
7.35
7.24
7.24
7.23
7.22
7.21
7.02
7.00
7.00
4.87
4.83
4.81
4.77
4.03
4.00
3.64
3.61
2.80
2.76
2.67
2.63
1.41
CN
O
N
H
O
3ah
1H NMR (400 MHz, CDCl3)
2.00
4.05
1.94
0.96
7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
f1 (ppm)
2.00
4.05
1.94
0.96
2.01
0.99
0.99
0.99
1.00
3.00
.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)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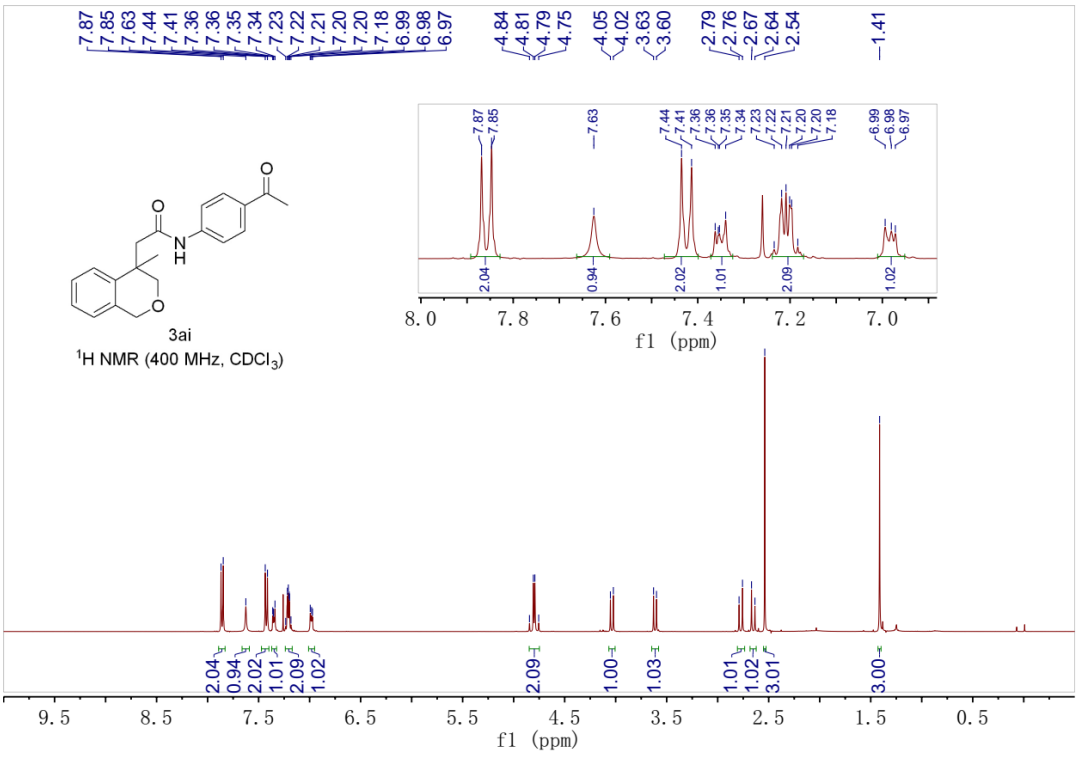
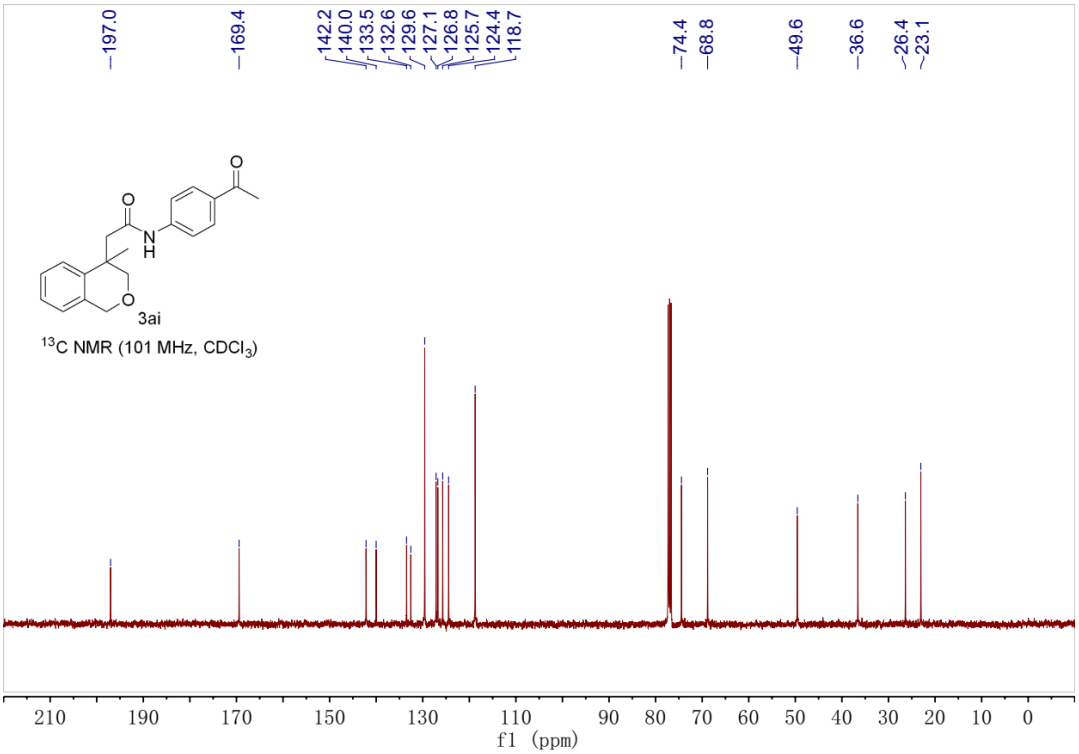
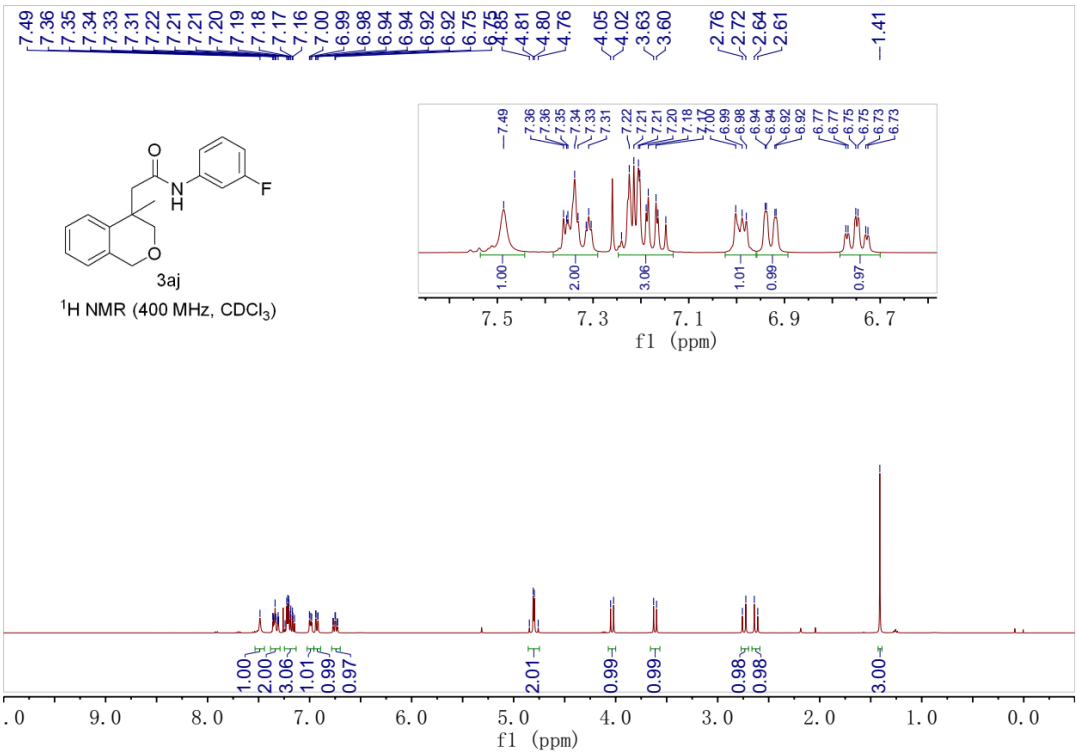
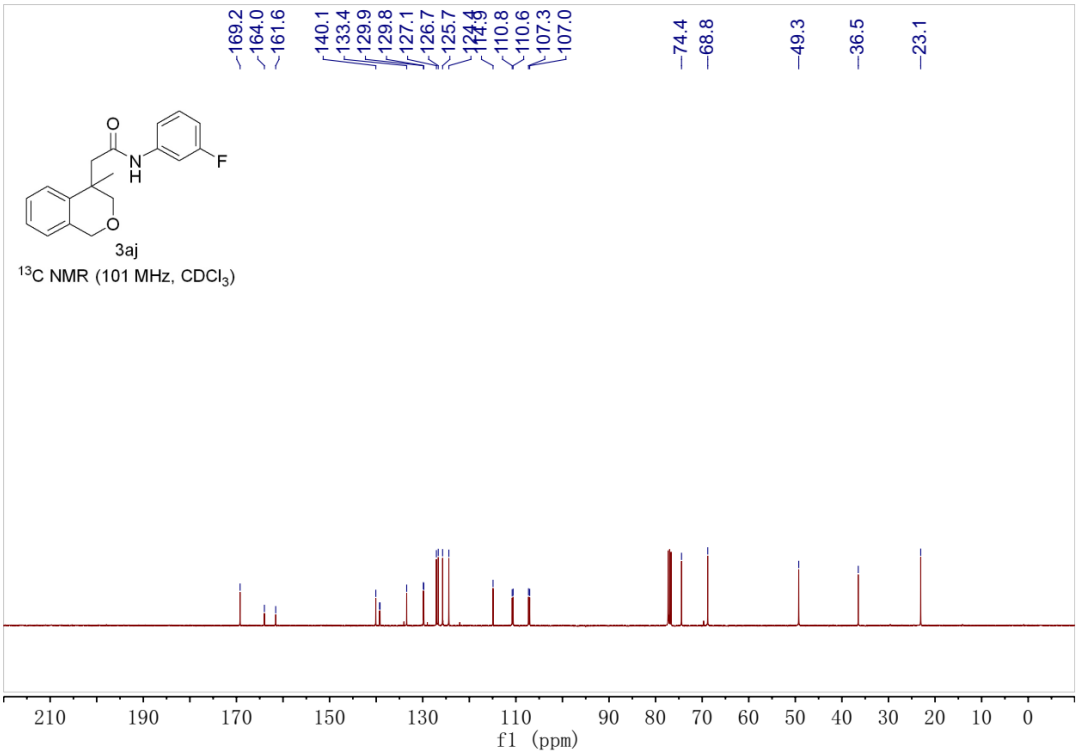
141.8
139.9
133.6
133.2
127.3
127.0
125.8
124.6
119.4
118.9
106.9
74.7
69.0
50.1
36.7
23.2
CN
O
N
H
O
3ah
13C NMR (101 MHz, CDCl3)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.87
7.85
7.63
7.44
7.41
7.36
7.36
7.35
7.34
7.23
7.22
7.21
7.20
7.20
7.18
6.99
6.98
6.97
4.84
4.81
4.79
4.75
4.05
4.02
3.63
3.60
2.79
2.76
2.67
2.64
2.54
1.41
3ai
1H NMR (400 MHz, CDCl3)
2.04
0.94
2.02
1.01
2.09
1.02
2.09
1.00
1.03
1.01
1.02
3.01
3.00
f1 (ppm)


197.0
169.4
142.2
140.0
133.5
132.6
129.6
127.1
126.8
125.7
124.4
118.7
74.4
68.8
49.6
36.6
26.4
23.1
3ai
13C NMR (101 MHz, CDCl3)
f1 (ppm)


7.49
7.36
7.35
7.34
7.33
7.31
7.22
7.21
7.21
7.20
7.19
7.18
7.17
7.16
7.00
6.99
6.98
6.94
6.94
6.92
6.92
6.75
6.75
4.85
4.81
4.80
4.76
4.05
4.02
3.63
3.60
2.76
2.72
2.64
2.61
1.41
O
N
H
F
O
3aj
1H NMR (400 MHz, CDCl3)
1.00
2.00
3.06
1.01
0.99
0.97
2.01
0.99
0.99
0.98
0.98
3.00
f1 (ppm)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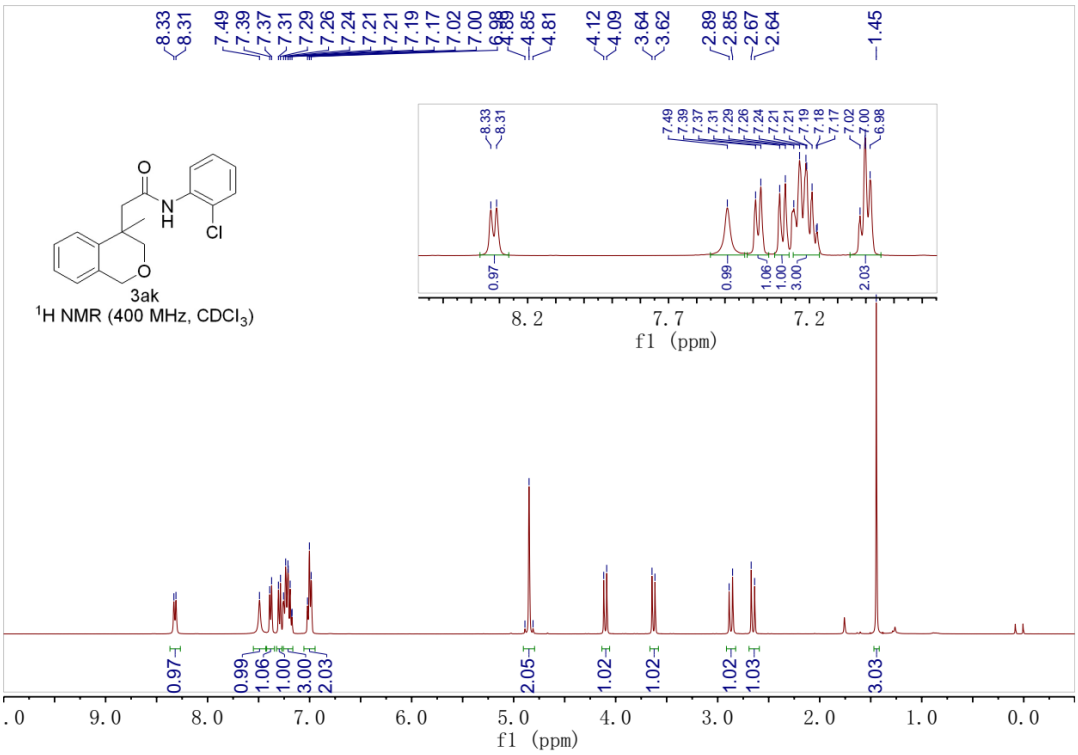
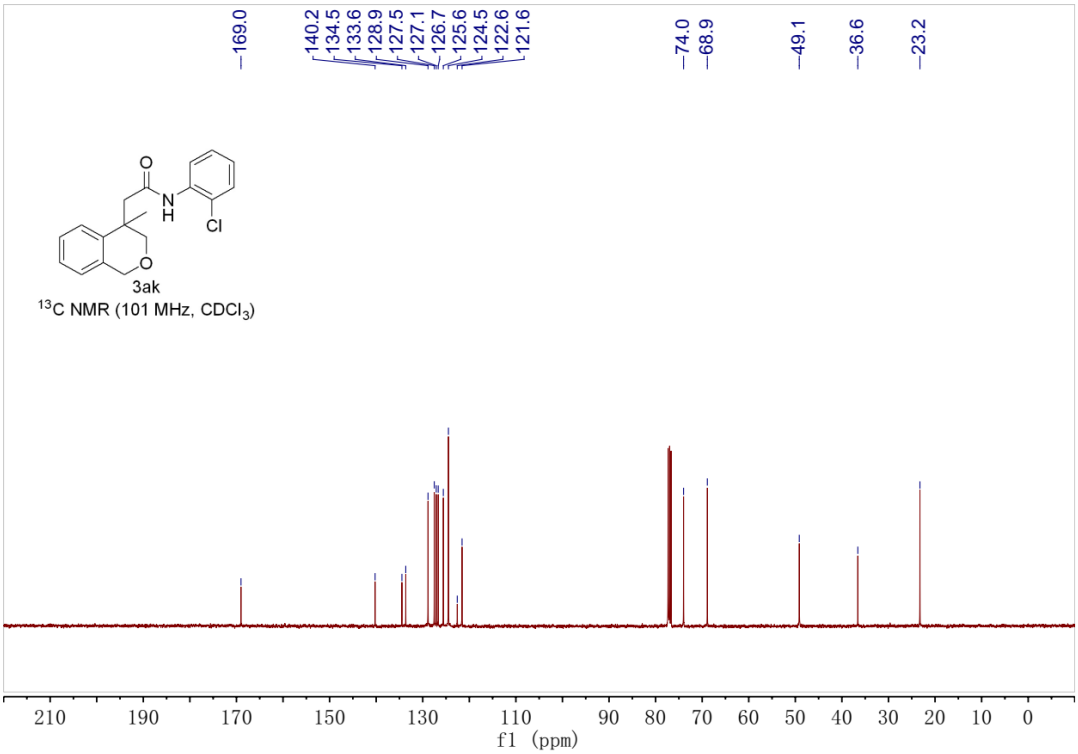
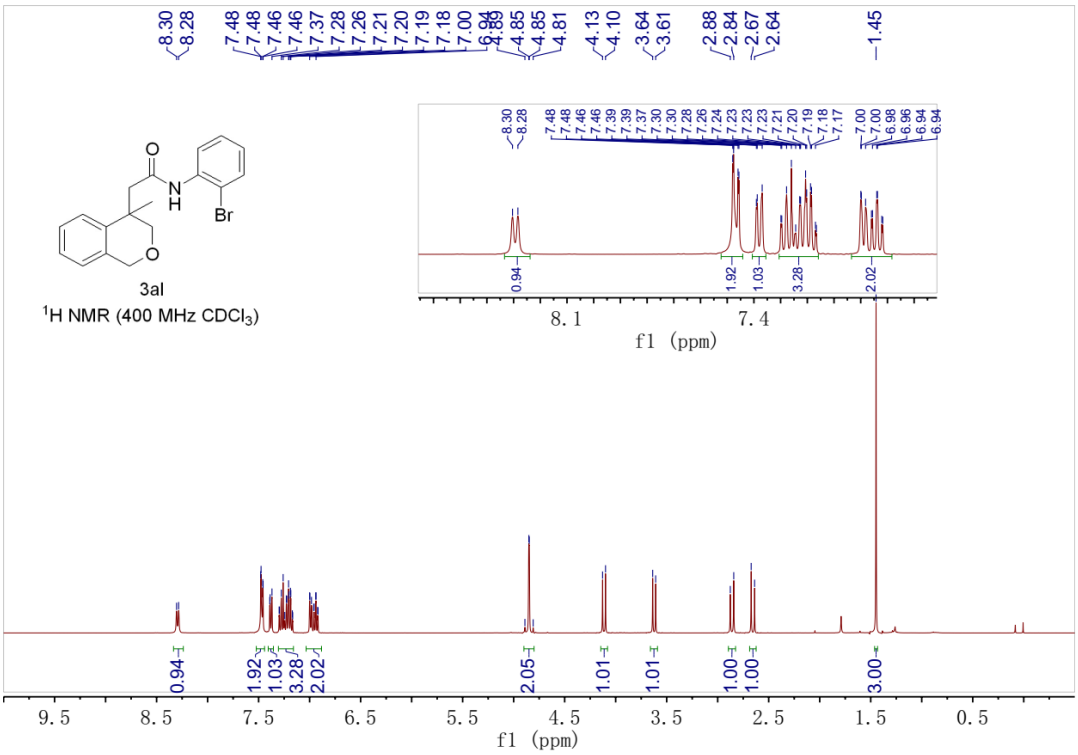
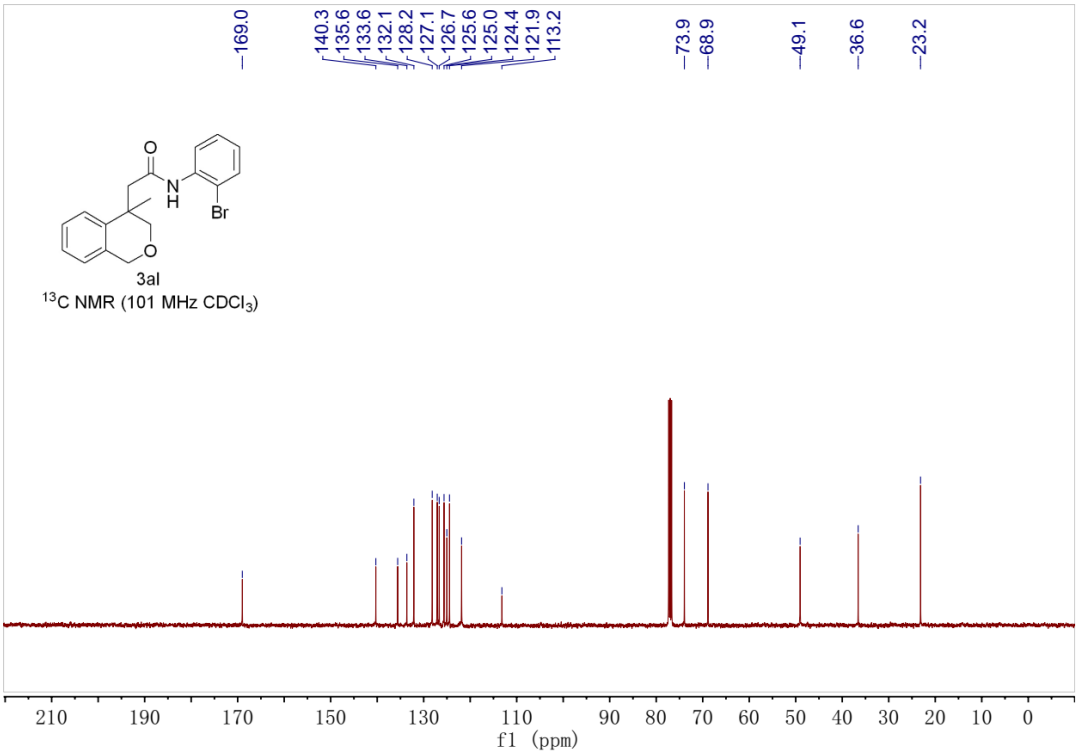
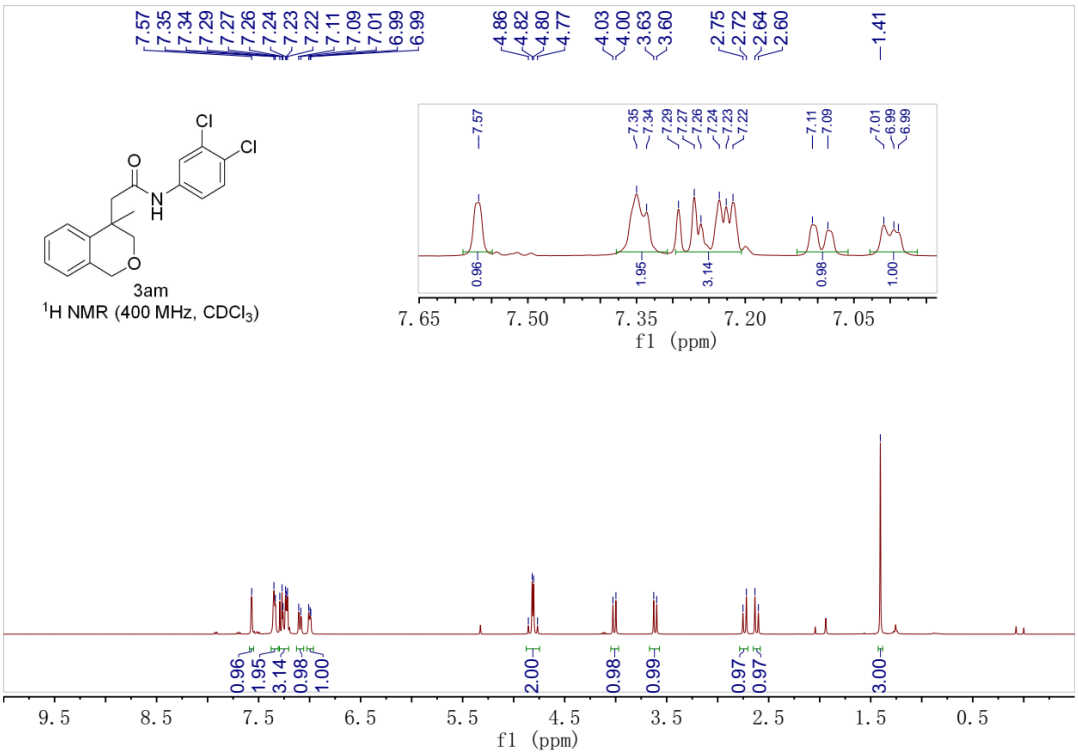
164.0
161.6
140.1
133.4
129.9
129.8
127.1
126.7
125.7
124.4
114.9
110.8
110.6
107.3
107.0
74.4
68.8
49.3
36.5
23.1
O
N
H
F
O
3aj
13C NMR (101 MHz, CDCl3)
f1 (ppm)

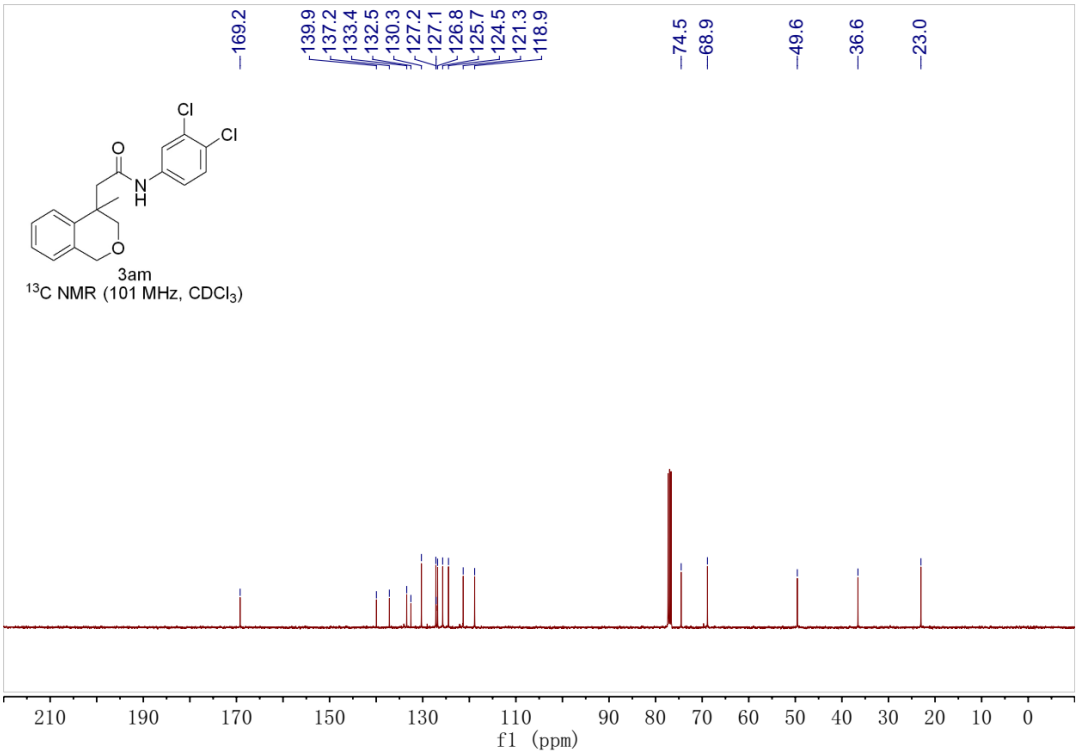

8.33
8.31
7.49
7.39
7.37
7.31
7.29
7.26
7.24
7.21
7.21
7.19
7.17
7.02
7.00
6.98
4.85
4.81
4.12
4.09
3.64
3.62
2.89
2.85
2.67
2.64
1.45
O
N
H
Cl
O
3ak
1H NMR (400 MHz, CDCl3)
0.97
0.99
1.06
1.00
3.00
2.03
2.05
1.02
1.02
1.02
1.03
3.03
f1 (ppm)

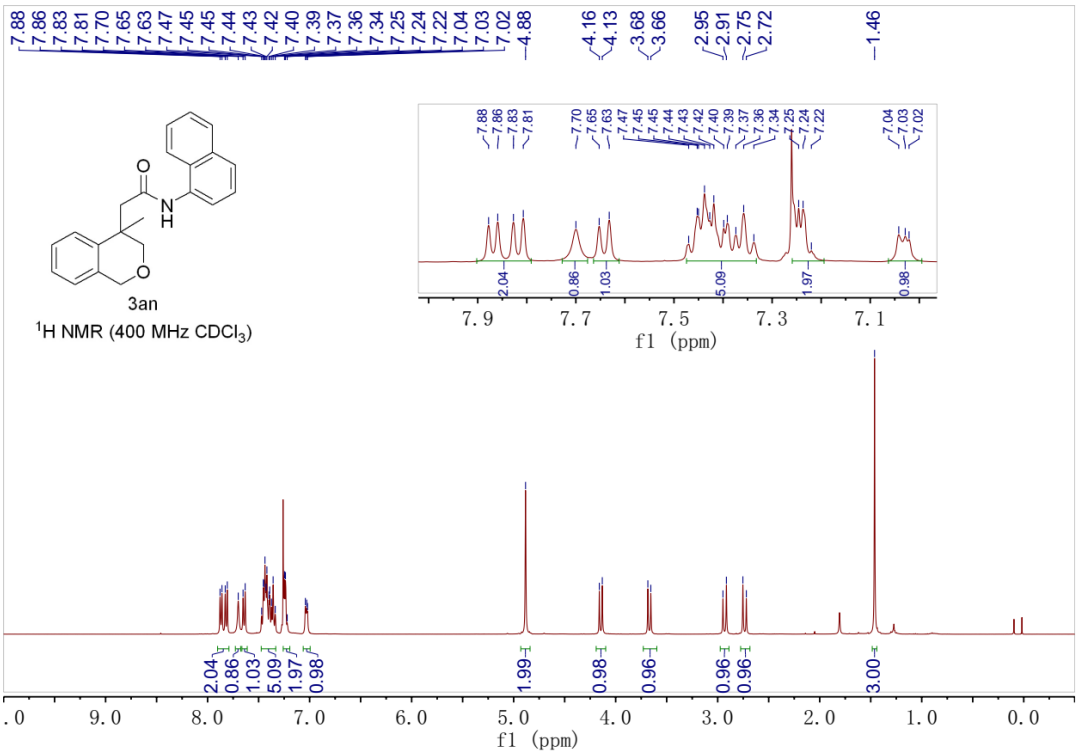

169.0
140.2
134.5
133.6
128.9
127.5
127.1
126.7
125.6
124.5
122.6
121.6
74.0
68.9
49.1
36.6
23.2
O
N
H
Cl
O
3ak
13C NMR (101 MHz, CDCl3)
f1 (ppm)

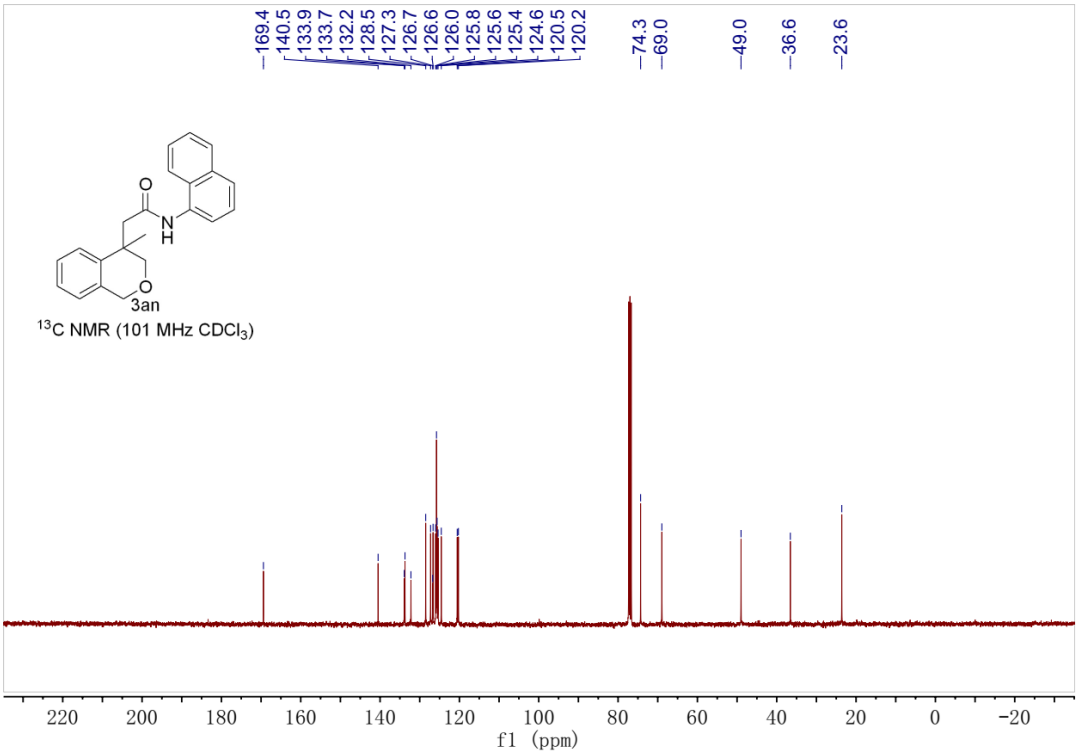

8.30
8.28
7.48
7.46
7.37
7.28
7.26
7.21
7.20
7.19
7.18
7.00
4.85
4.81
4.13
4.10
3.64
3.61
2.88
2.84
2.67
2.64
1.45
O
N
H
Br
O
3al
^{1}H NMR (400 MHz CDCl$_3$)
0.94
1.92
1.03
3.28
2.02
2.05
1.01
1.01
1.00
1.00
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)
8.1
7.4
f1 (ppm)


169.0
140.3
135.6
133.6
132.1
128.2
127.1
126.7
125.6
125.0
124.4
121.9
113.2
73.9
68.9
49.1
36.6
23.2
O
N
H
Br
O
3al
^{13}C NMR (101 MHz CDCl$_3$)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.57
7.35
7.34
7.29
7.27
7.26
7.24
7.23
7.22
7.11
7.09
7.01
6.99
6.99
4.86
4.82
4.80
4.77
4.03
4.00
3.63
3.60
2.75
2.72
2.64
2.60
1.41
Cl
Cl
O
N
H
O
3am
1H NMR (400 MHz, CDCl3)
0.96
1.95
3.14
0.98
1.00
2.00
0.98
0.99
0.97
0.97
3.00
f1 (ppm)


169.2
139.9
137.2
133.4
132.5
130.3
127.2
127.1
126.8
125.7
124.5
121.3
118.9
74.5
68.9
49.6
36.6
23.0
Cl
Cl
O
N
H
O
3am
13C NMR (101 MHz, CDCl3)
f1 (ppm)


7.88
7.86
7.83
7.81
7.70
7.65
7.63
7.47
7.45
7.45
7.44
7.43
7.42
7.40
7.39
7.37
7.36
7.34
7.25
7.24
7.22
7.04
7.03
7.02
4.88
4.16
4.13
3.68
3.66
2.95
2.91
2.75
2.72
1.46
O
N
H
O
3an
1H NMR (400 MHz CDCl3)
2.04
0.86
1.03
5.09
1.97
0.98
1.99
0.98
0.96
0.96
0.96
3.00
f1 (ppm)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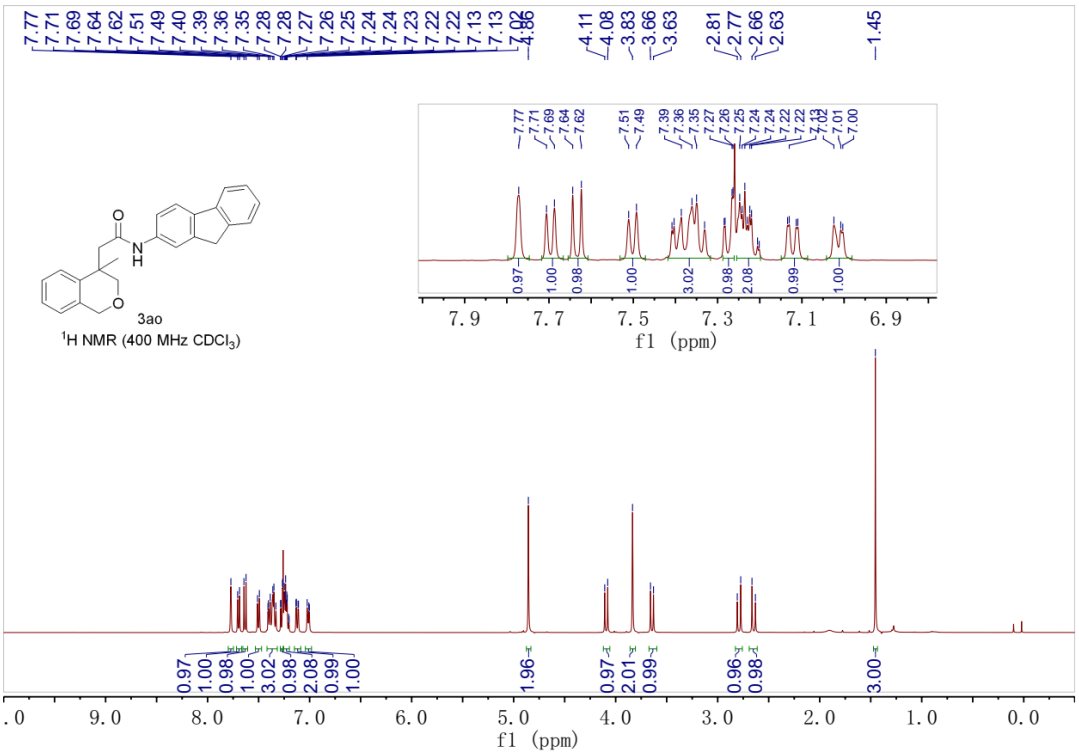
140.5
133.9
133.7
132.2
128.5
127.3
126.7
126.6
126.0
125.8
125.6
125.4
124.6
120.5
120.2
74.3
69.0
49.0
36.6
23.6
O
N
H
O
3an
13C NMR (101 MHz CDCl3)
f1 (ppm)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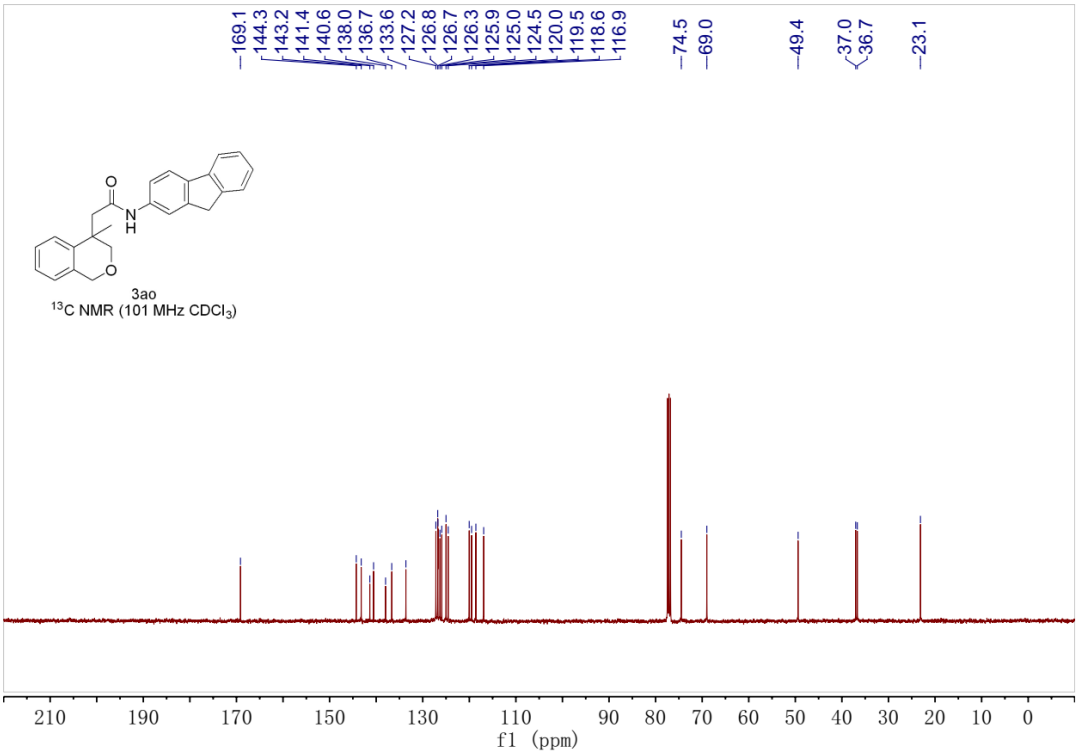
7.71
7.69
7.64
7.62
7.51
7.49
7.40
7.39
7.36
7.35
7.28
7.27
7.26
7.25
7.24
7.24
7.23
7.22
7.22
7.13
7.13
7.06
4.11
4.08
3.83
3.66
3.63
2.81
2.77
2.66
2.63
1.45
O
N
H
O
3ao
1H NMR (400 MHz CDCl3)
0.97
1.00
0.98
1.00
3.02
0.98
2.08
0.99
1.00
1.96
0.97
2.01
0.99
0.96
0.98
3.00
f1 (ppm)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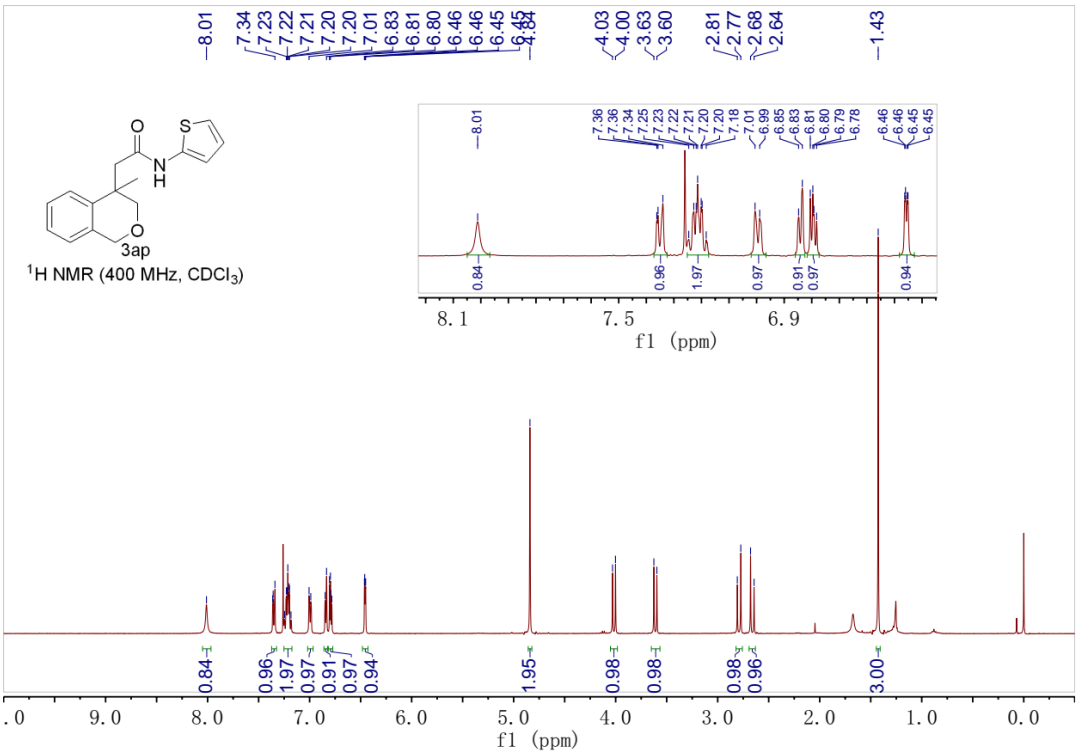
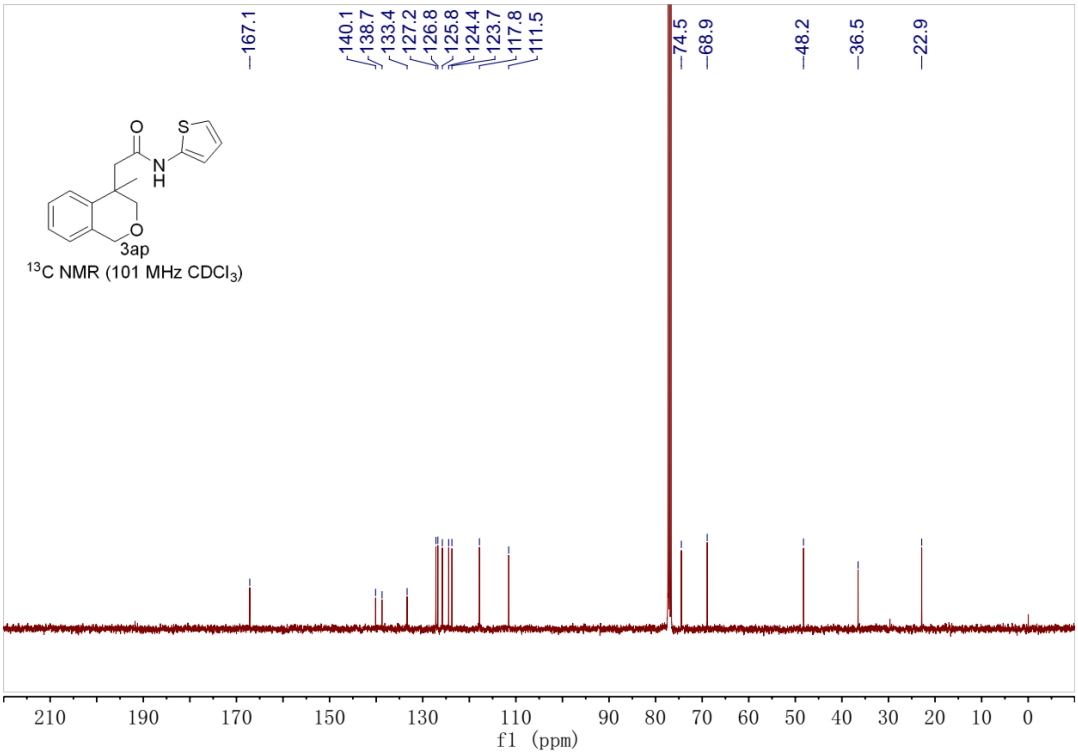
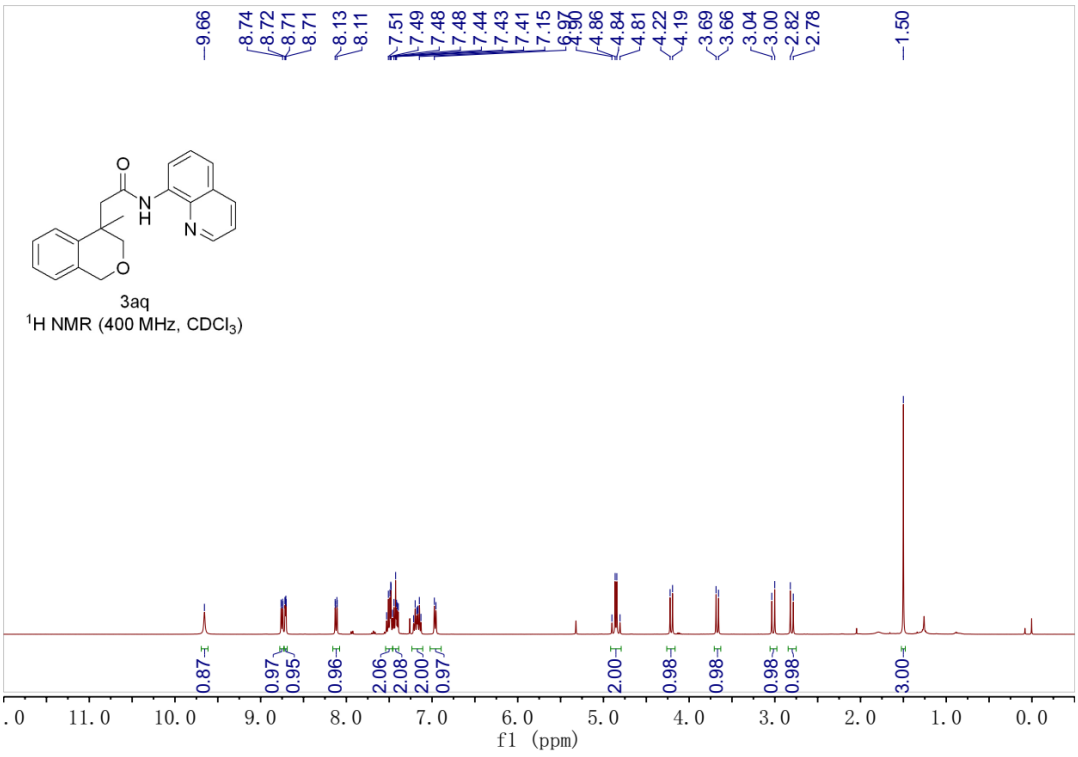
144.3
143.2
141.4
140.6
138.0
136.7
133.6
127.2
126.8
126.7
126.3
125.9
125.0
124.5
120.0
119.5
118.6
116.9
74.5
69.0
49.4
37.0
36.7
23.1
O
N
H
O
3ao
13C NMR (101 MHz CDCl3)
f1 (ppm)

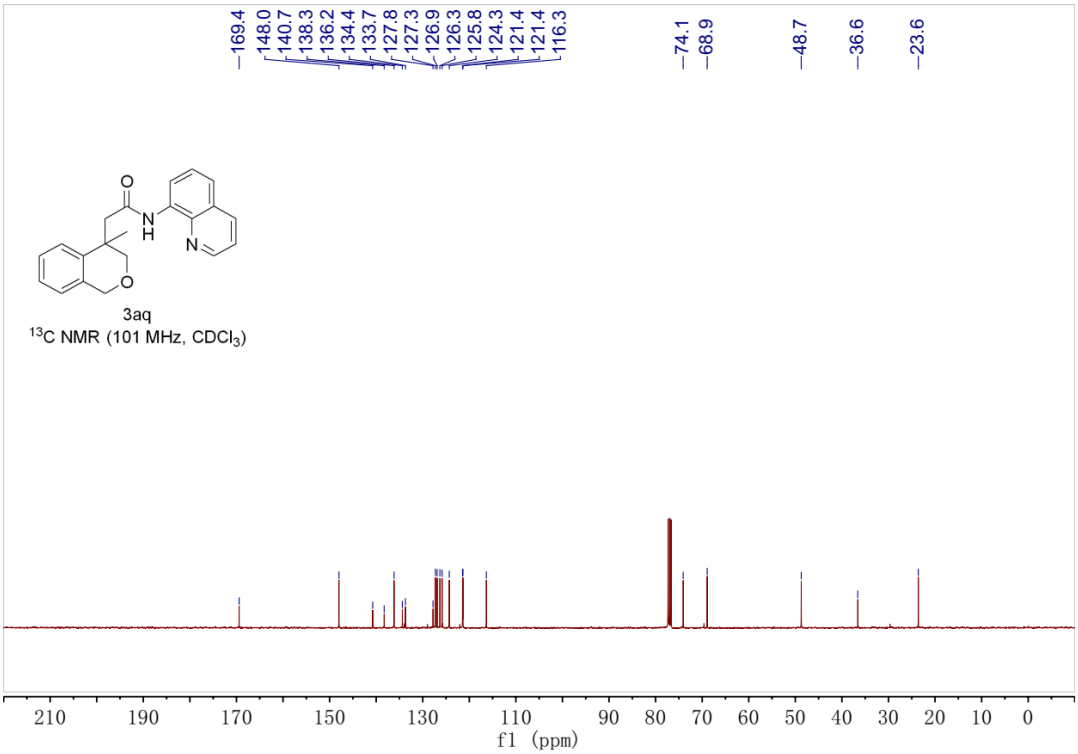

3ap
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)


3ap
^{13}C NMR (101 MHz CDCl$_3$)
f1 (ppm)



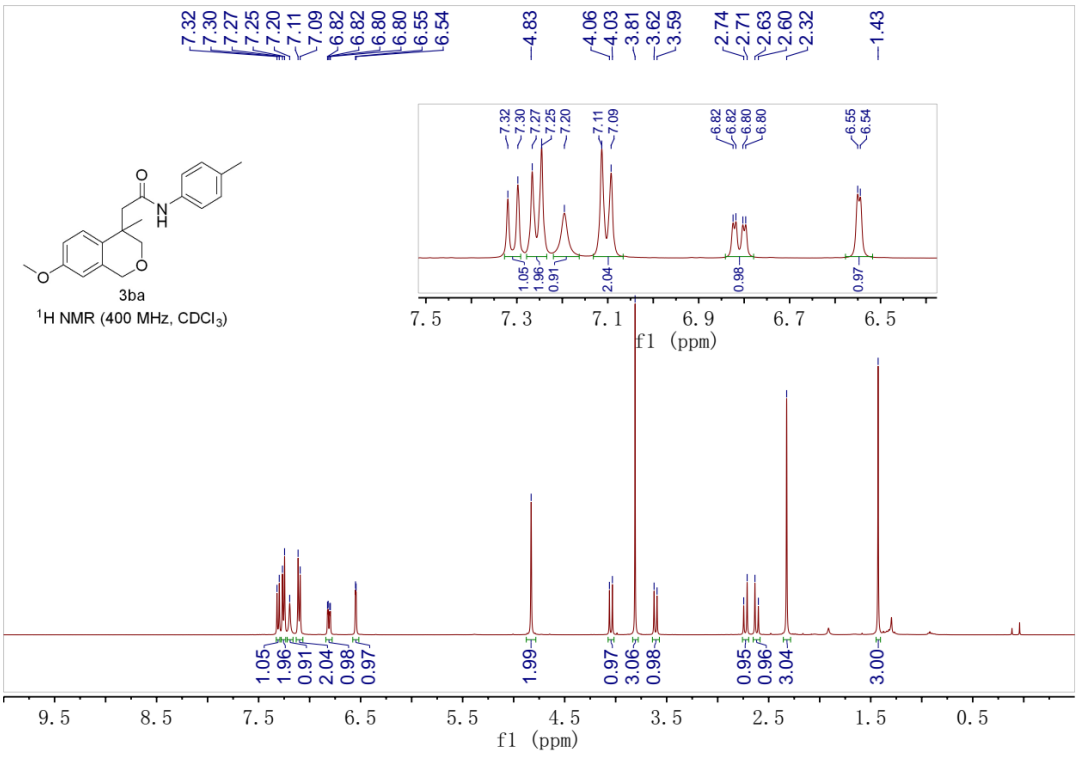
3aq

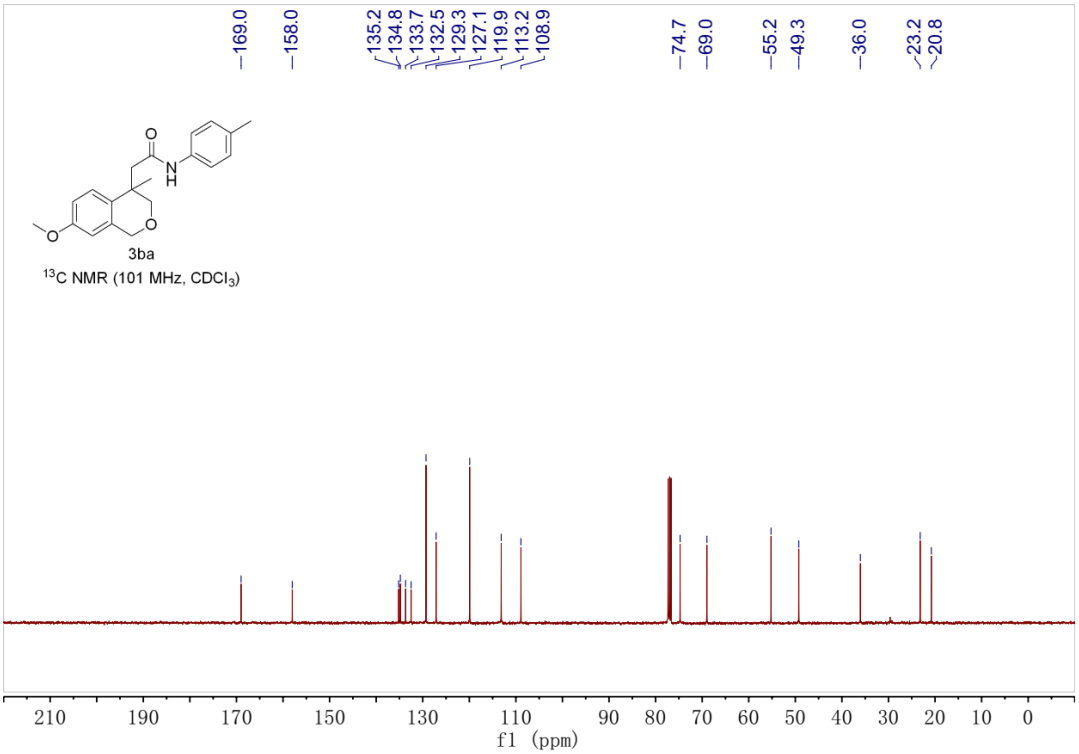
^{1}H NMR (400 MHz, $CDCl_3$)

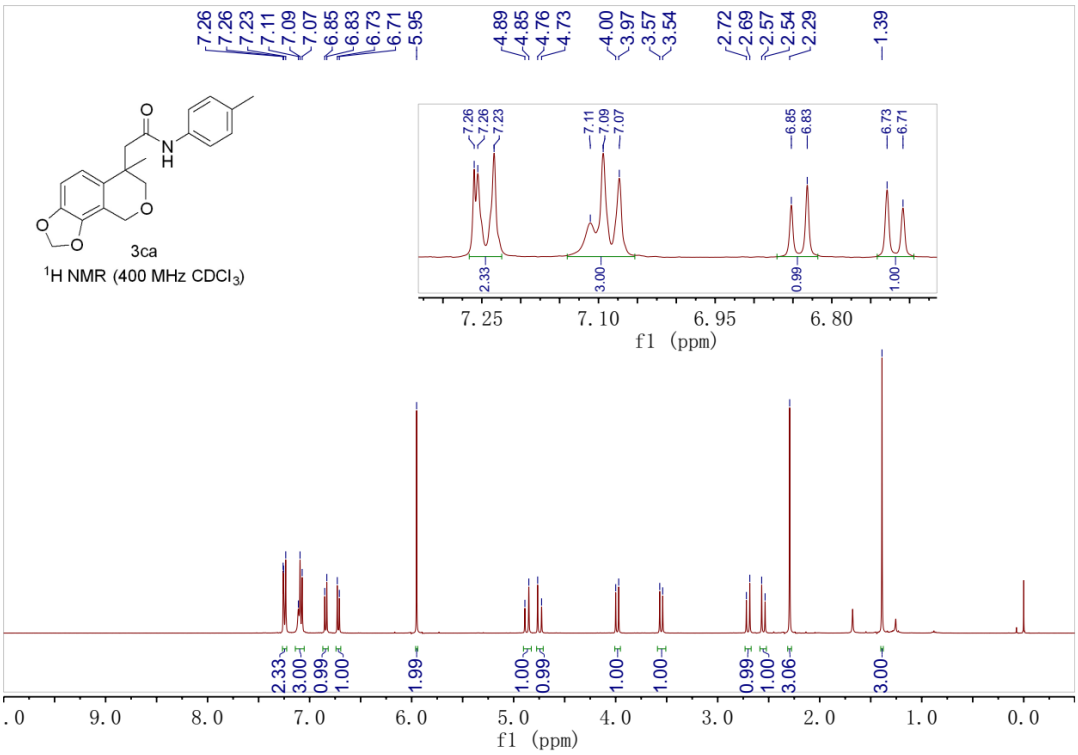


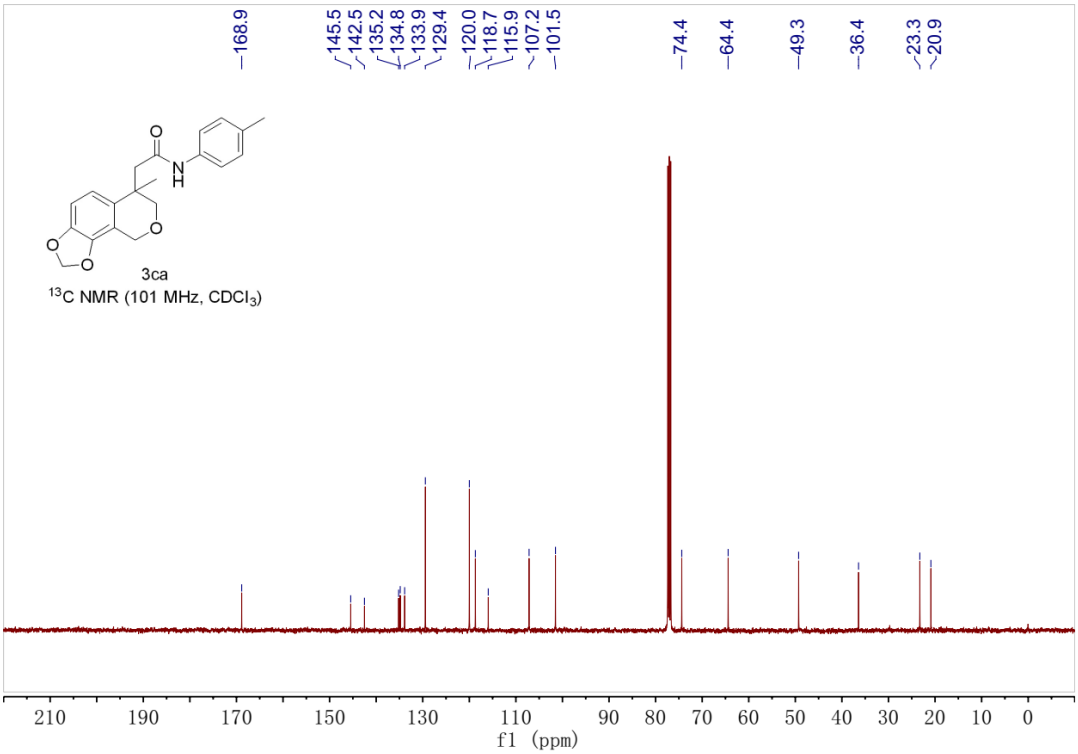
3aq

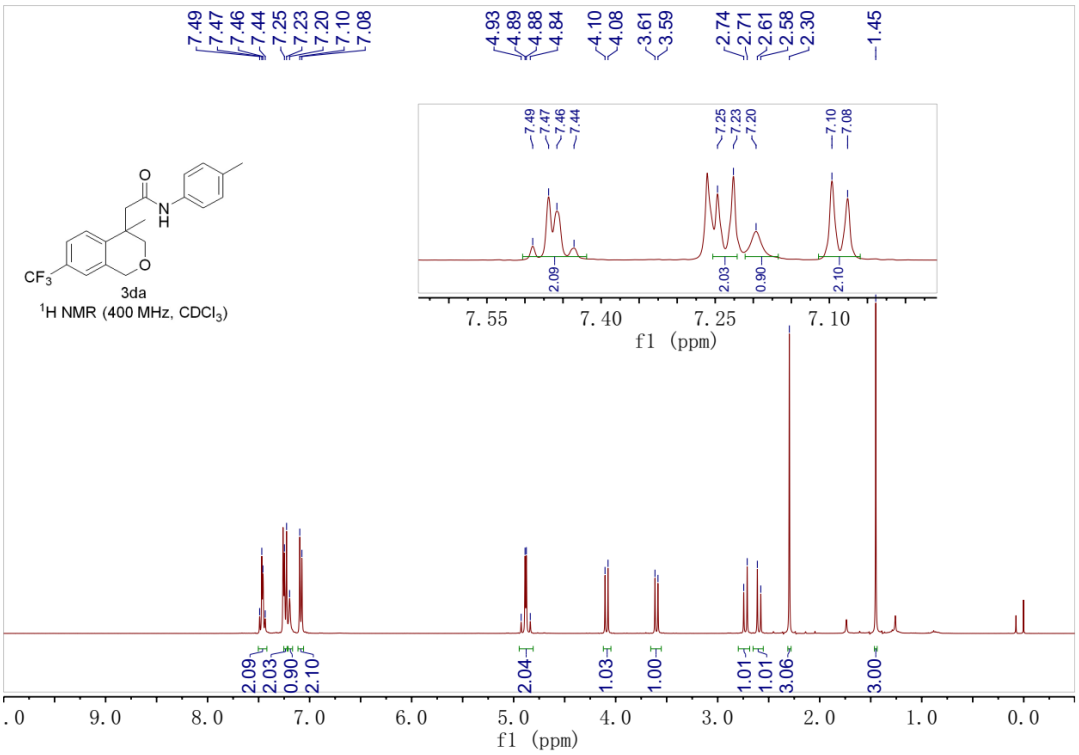
^{13}C NMR (101 MHz, $CDCl_3$)

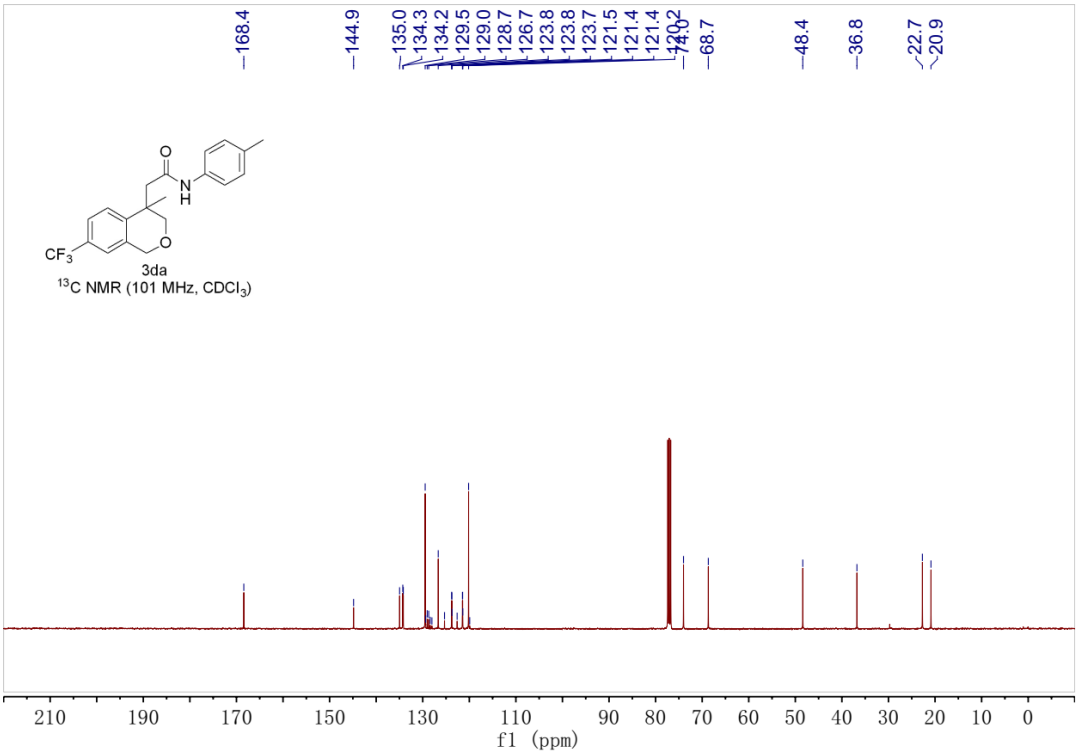

7.32
7.30
7.27
7.25
7.20
7.11
7.09
6.82
6.82
6.80
6.80
6.55
6.54
4.83
4.06
4.03
3.81
3.62
3.59
2.74
2.71
2.63
2.60
2.32
1.43
3ba
1H NMR (400 MHz, CDCl3)
1.05
1.96
0.91
2.04
0.98
0.97
1.99
0.97
3.06
0.98
0.95
0.96
3.04
3.00
f1 (ppm)

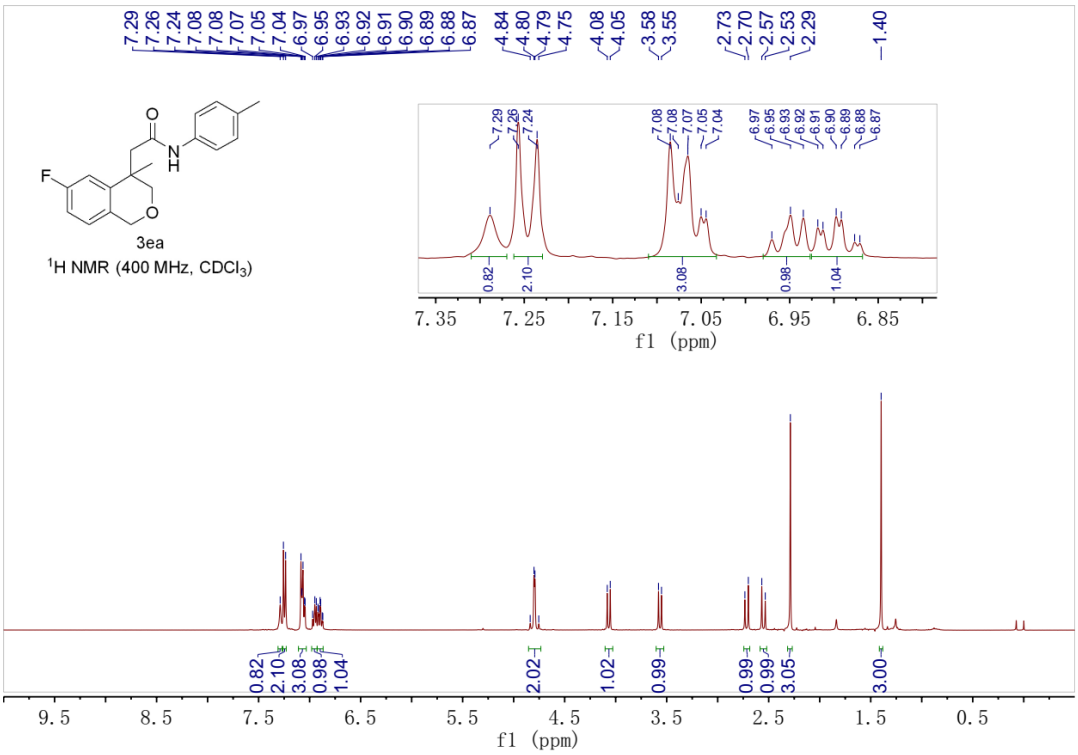

169.0
158.0
135.2
134.8
133.7
132.5
129.3
127.1
119.9
113.2
108.9
74.7
69.0
55.2
49.3
36.0
23.2
20.8
3ba
13C NMR (101 MHz, CDCl3)
f1 (ppm)

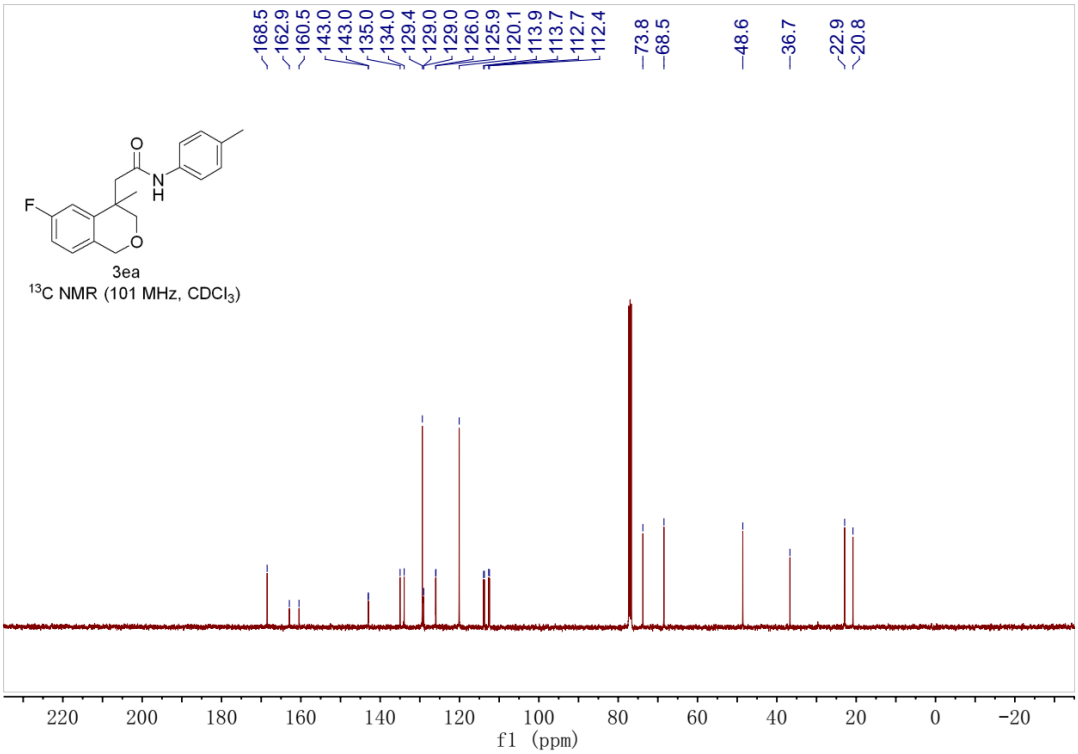

7.26
7.26
7.23
7.11
7.09
7.07
6.85
6.83
6.73
6.71
5.95
4.89
4.85
4.76
4.73
4.00
3.97
3.57
3.54
2.72
2.69
2.57
2.54
2.29
1.39
3ca
1H NMR (400 MHz CDCl3)
2.33
3.00
0.99
1.00
1.99
1.00
0.99
1.00
1.00
0.99
1.00
3.06
3.00
f1 (ppm)

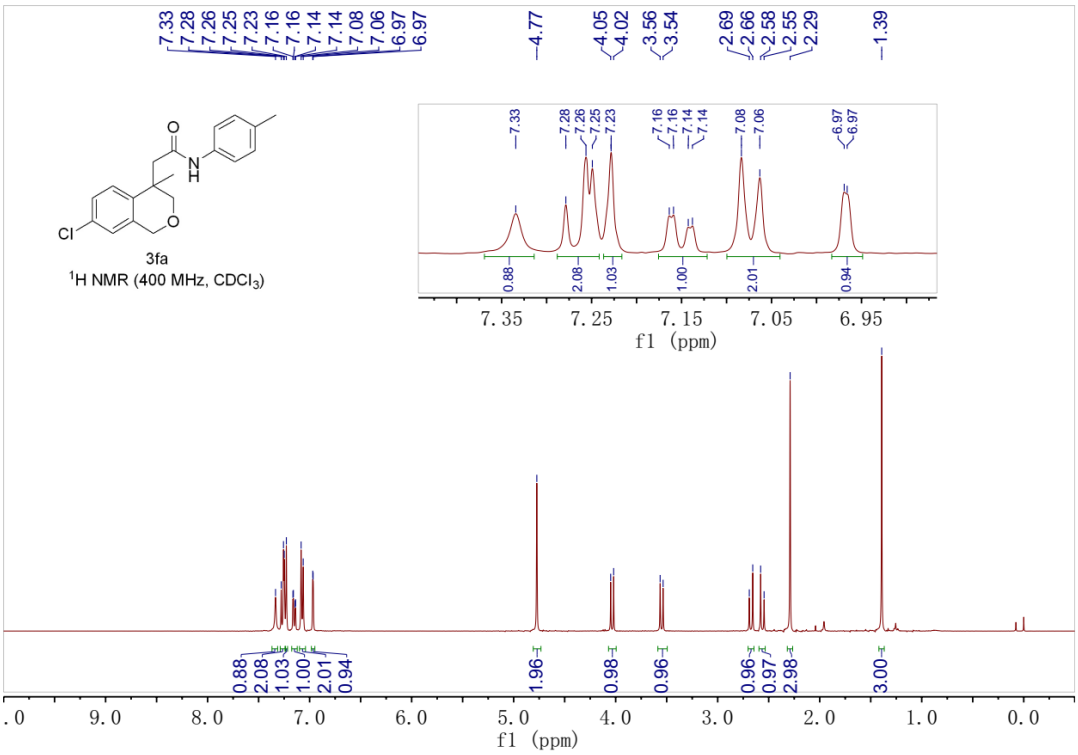

168.9
145.5
142.5
135.2
134.8
133.9
129.4
120.0
118.7
115.9
107.2
101.5
74.4
64.4
49.3
36.4
23.3
20.9
3ca
13C NMR (101 MHz, CDCl3)
f1 (ppm)

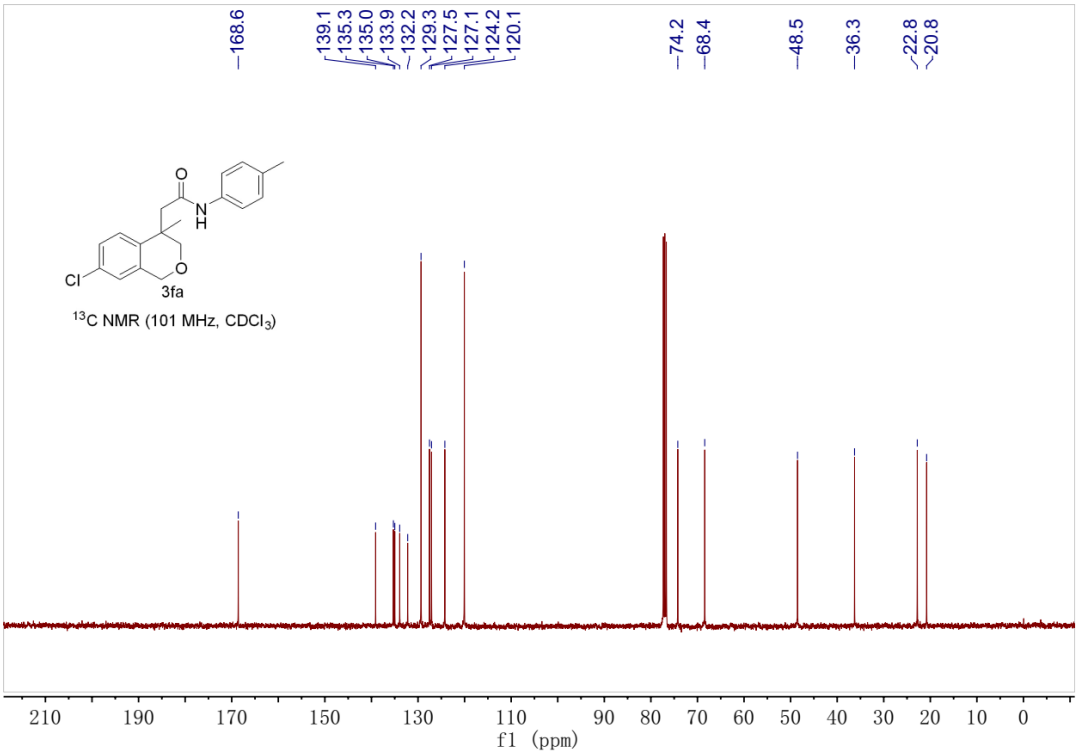

7.49
7.47
7.46
7.44
7.25
7.23
7.20
7.10
7.08
4.93
4.89
4.88
4.84
4.10
4.08
3.61
3.59
2.74
2.71
2.61
2.58
2.30
1.45
2.09
2.03
0.90
2.10
2.04
1.03
1.00
1.01
1.01
3.06
3.00
f1 (ppm)
3da
1H NMR (400 MHz, CDCl3)

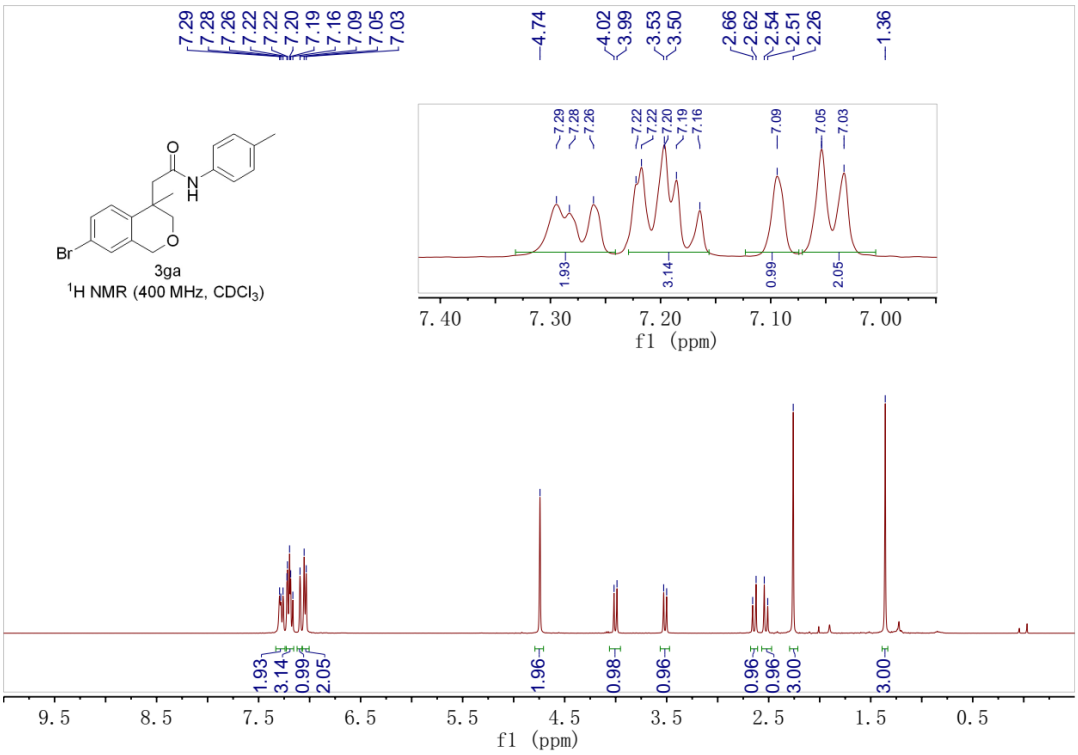

168.4
144.9
135.0
134.3
134.2
129.5
129.0
128.7
126.7
123.8
123.8
123.7
121.5
121.4
121.4
74.0
68.7
48.4
36.8
22.7
20.9
f1 (ppm)
3da
13C NMR (101 MHz, CDCl3)

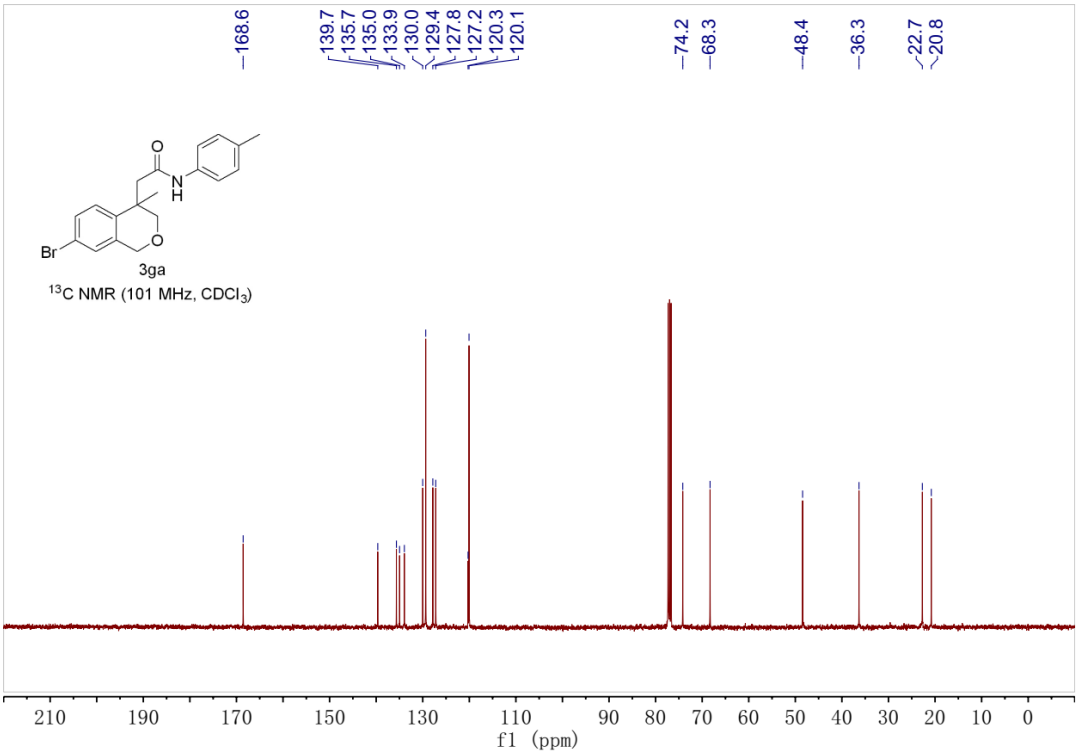

7.29
7.26
7.24
7.08
7.08
7.07
7.05
7.04
6.97
6.95
6.93
6.92
6.91
6.90
6.89
6.88
6.87
4.84
4.80
4.79
4.75
4.08
4.05
3.58
3.55
2.73
2.70
2.57
2.53
2.29
1.40
F
O
N
H
O
3ea
1H NMR (400 MHz, CDCl3)
0.82
2.10
3.08
0.98
1.04
2.02
1.02
0.99
0.99
0.99
3.05
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)
7.35
7.25
7.15
7.05
6.95
6.85
f1 (ppm)

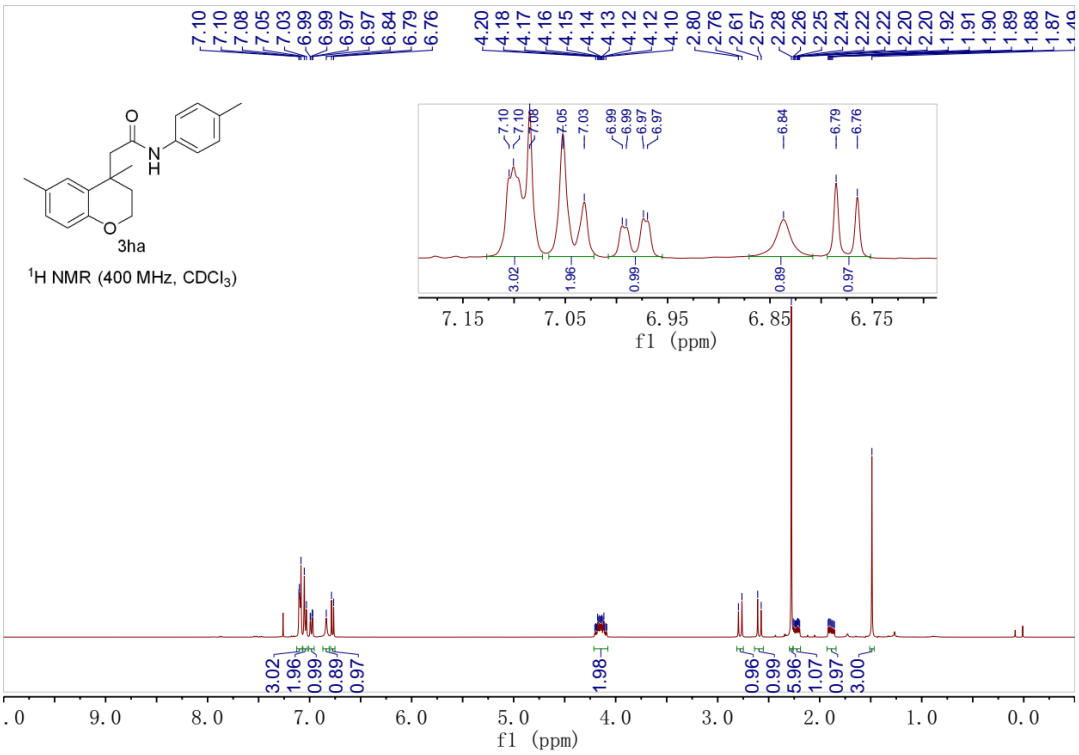

168.5
162.9
160.5
143.0
143.0
135.0
134.0
129.4
129.0
129.0
126.0
125.9
120.1
113.9
113.7
112.7
112.4
73.8
68.5
48.6
36.7
22.9
20.8
F
O
N
H
O
3ea
13C NMR (101 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
−20
f1 (ppm)

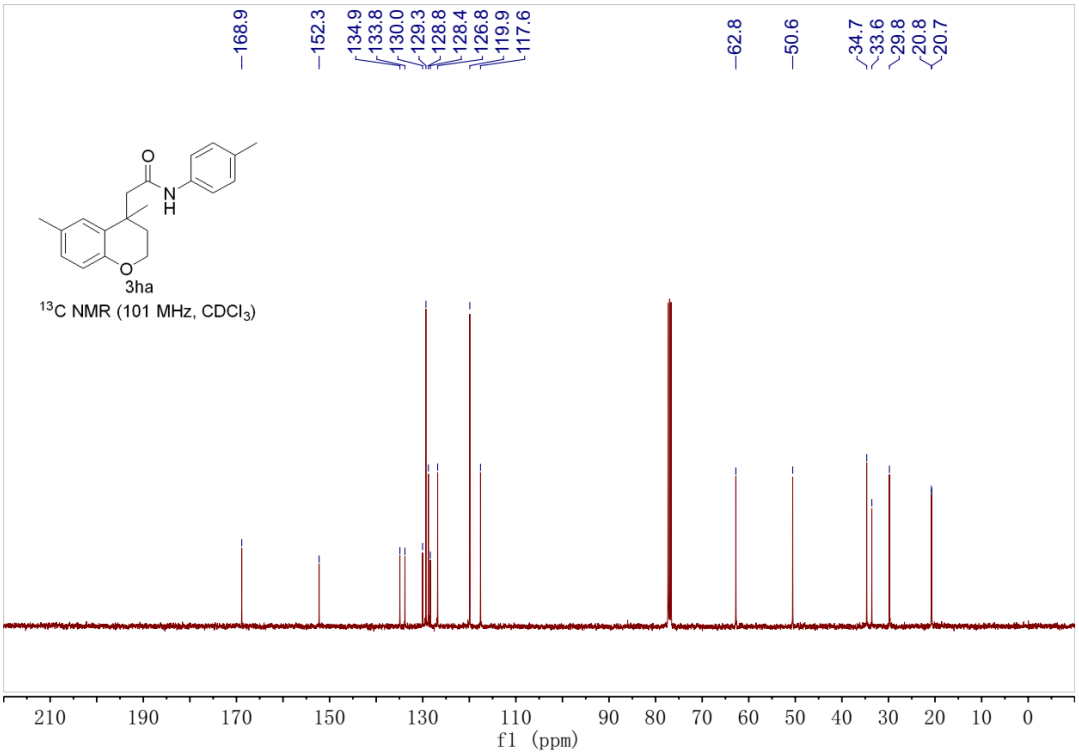

7.33
7.28
7.26
7.25
7.23
7.16
7.16
7.14
7.14
7.08
7.06
6.97
6.97
4.77
4.05
4.02
3.56
3.54
2.69
2.66
2.58
2.55
2.29
1.39
O
N
H
O
Cl
3fa
^{1}H NMR (400 MHz, CDCl$_3$)
0.88
2.08
1.03
1.00
2.01
0.94
1.96
0.98
0.96
0.96
0.97
2.98
3.00
f1 (ppm)

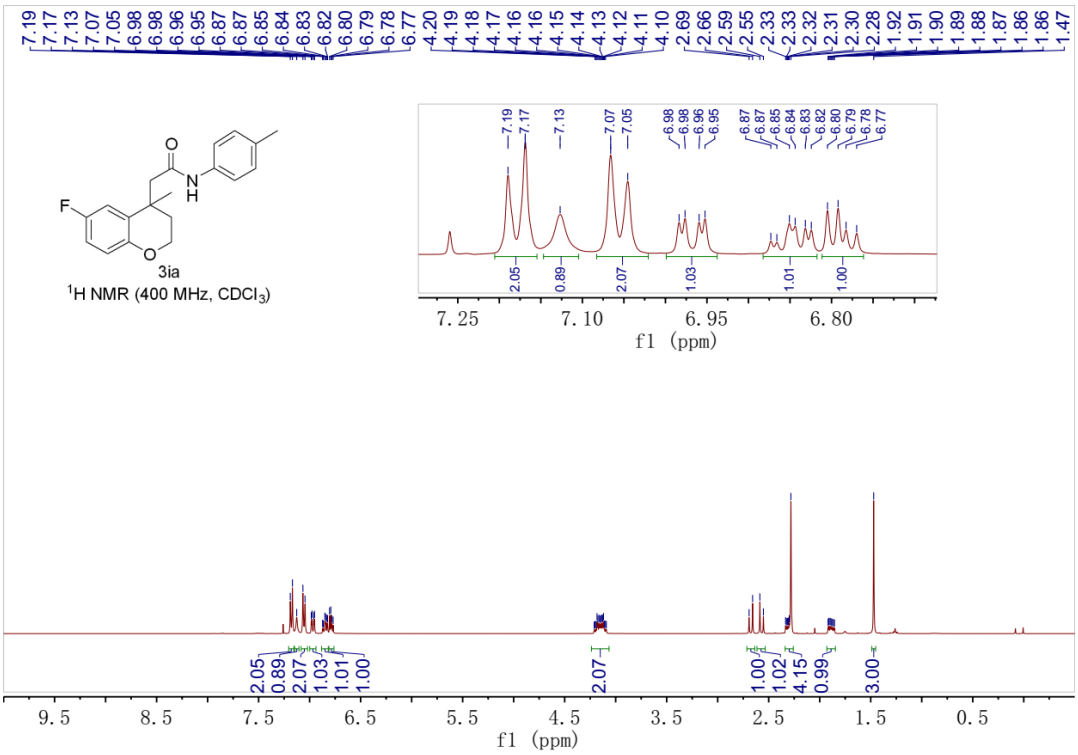

168.6
139.1
135.3
135.0
133.9
132.2
129.3
127.5
127.1
124.2
120.1
74.2
68.4
48.5
36.3
22.8
20.8
O
N
H
O
Cl
3fa
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

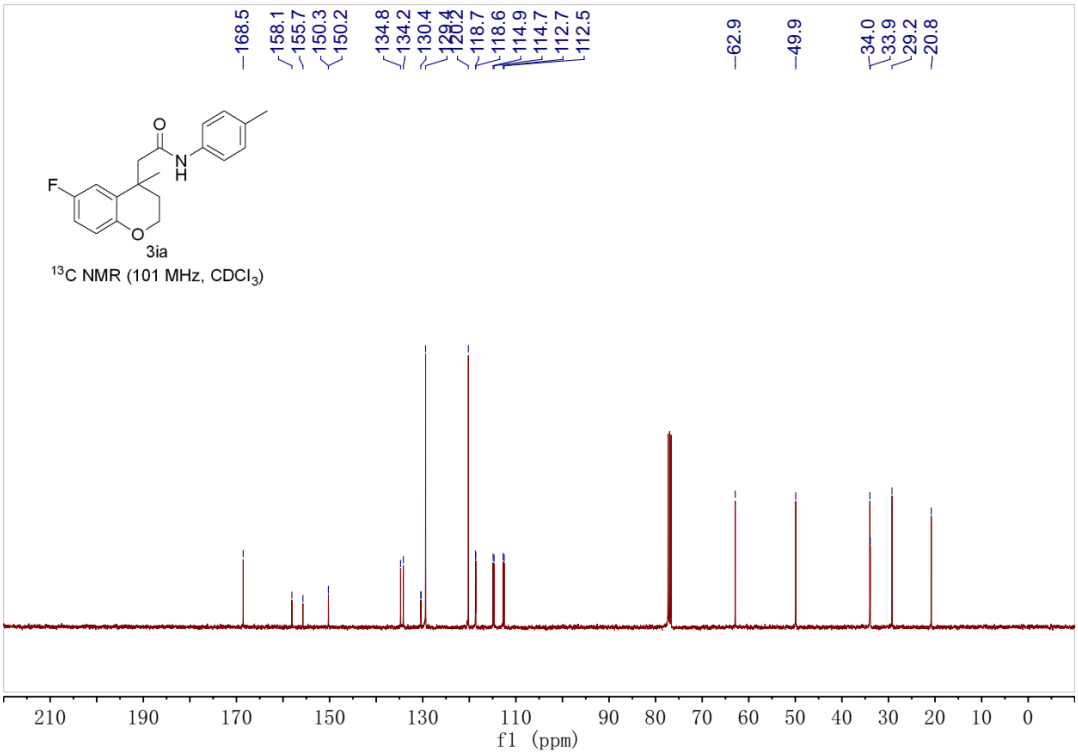

7.29
7.28
7.26
7.22
7.22
7.20
7.19
7.16
7.09
7.05
7.03
4.74
4.02
3.99
3.53
3.50
2.66
2.62
2.54
2.51
2.26
1.36
Br
O
N
H
O
3ga
^{1}H NMR (400 MHz, $CDCl_3$)
1.93
3.14
0.99
2.05
7.40
7.30
7.20
7.10
7.00
f1 (ppm)
1.93
3.14
0.99
2.05
1.96
0.98
0.96
0.96
0.96
3.00
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

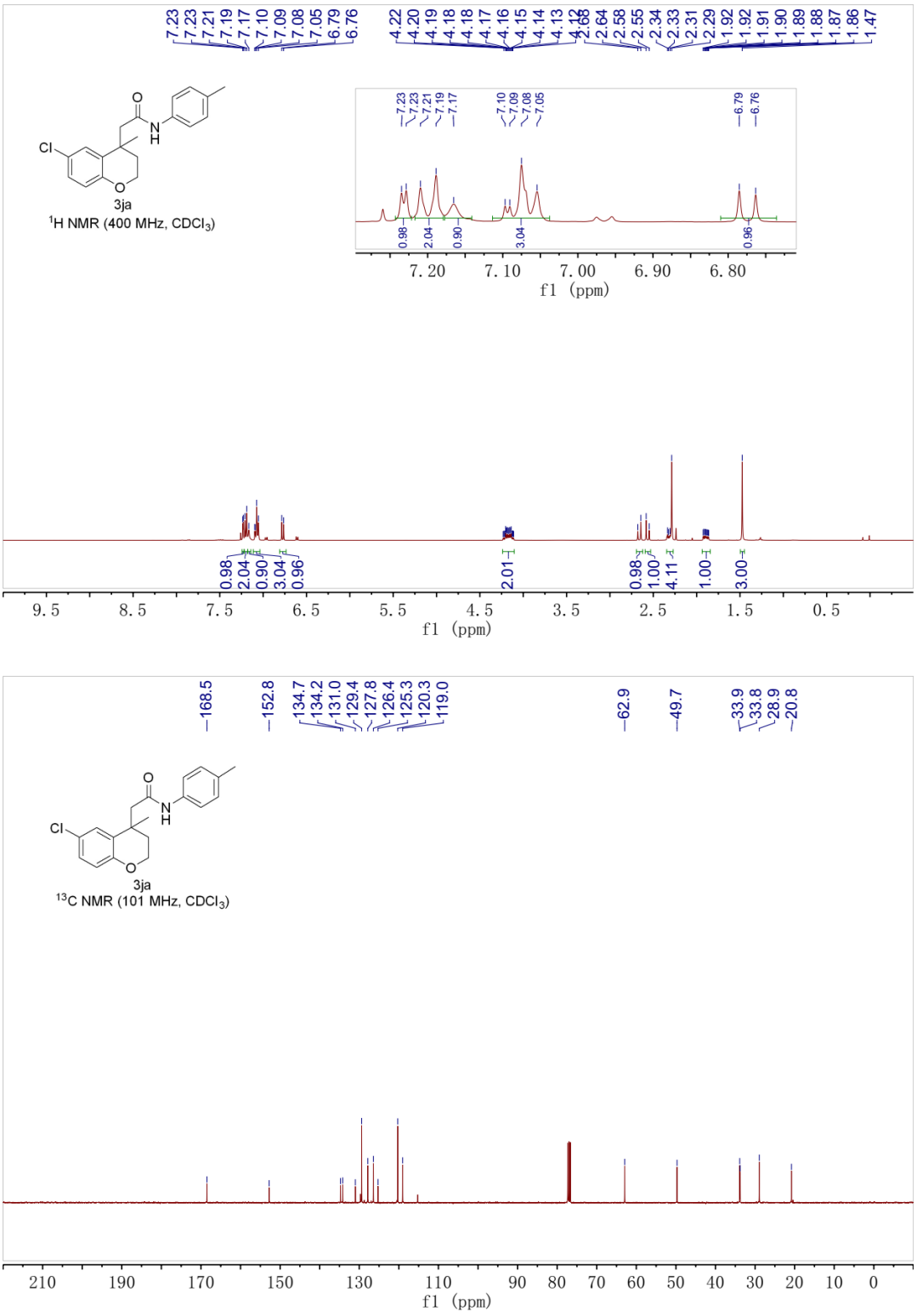

168.6
139.7
135.7
135.0
133.9
130.0
129.4
127.8
127.2
120.3
120.1
74.2
68.3
48.4
36.3
22.7
20.8
Br
O
N
H
O
3ga
^{13}C NMR (101 MHz, $CDCl_3$)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

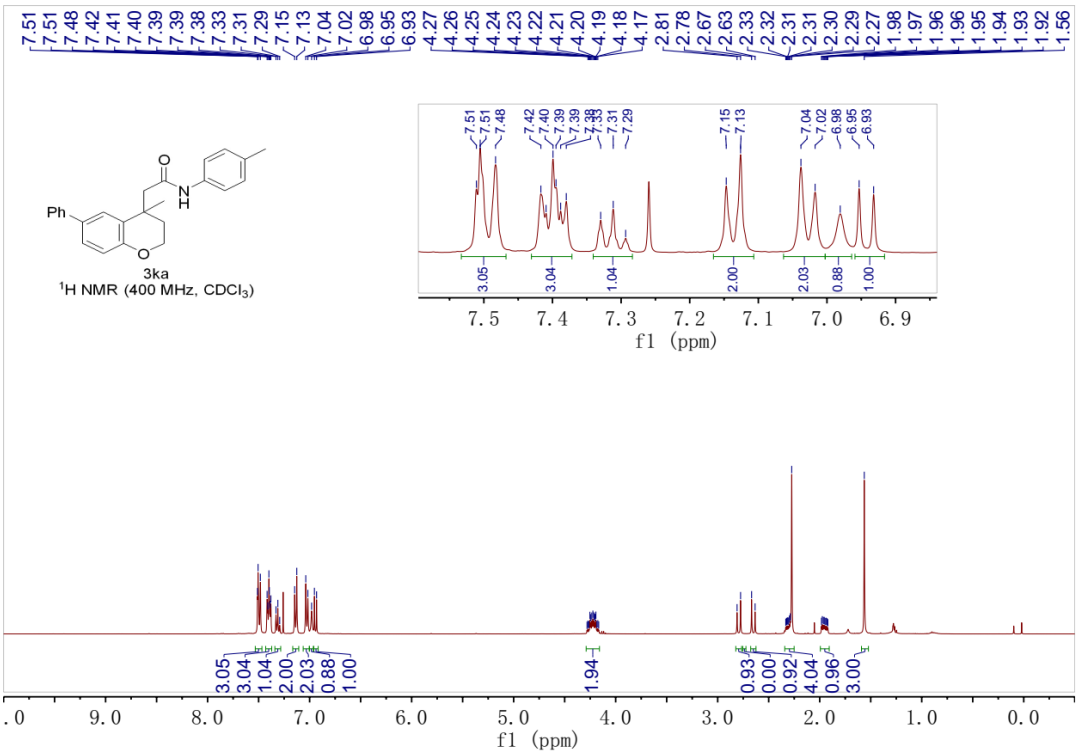

3ha
^{1}H NMR (400 MHz, $CDCl_3$)
f1 (ppm)

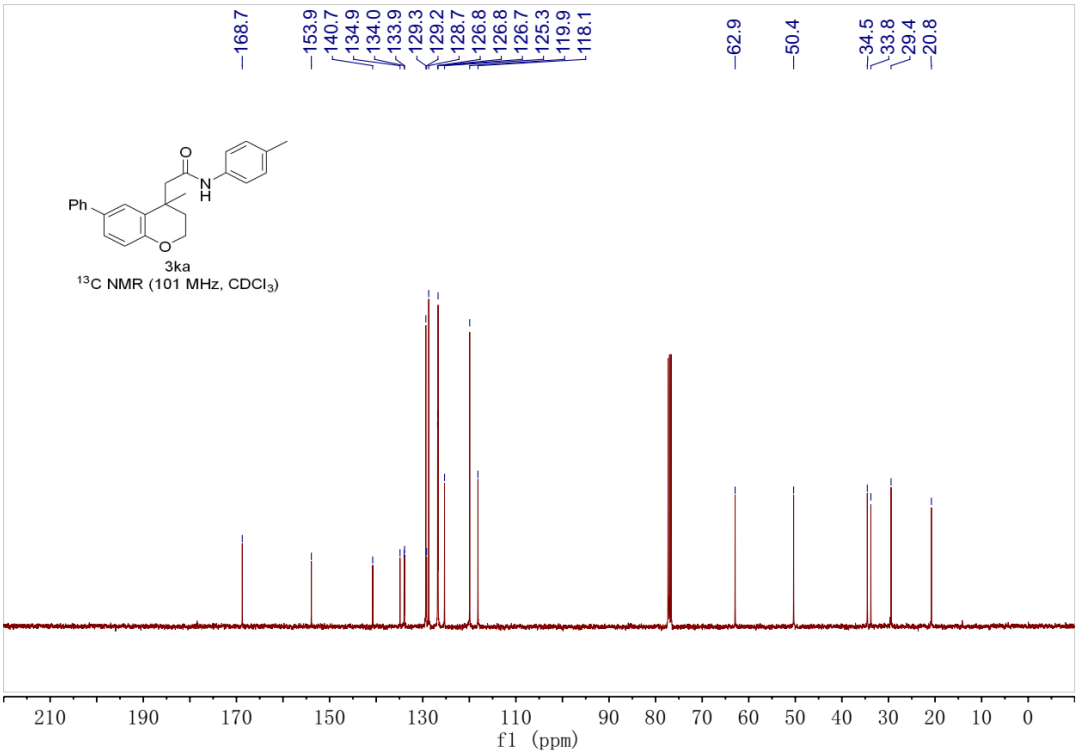

3ha
^{13}C NMR (101 MHz, $CDCl_3$)
f1 (ppm)

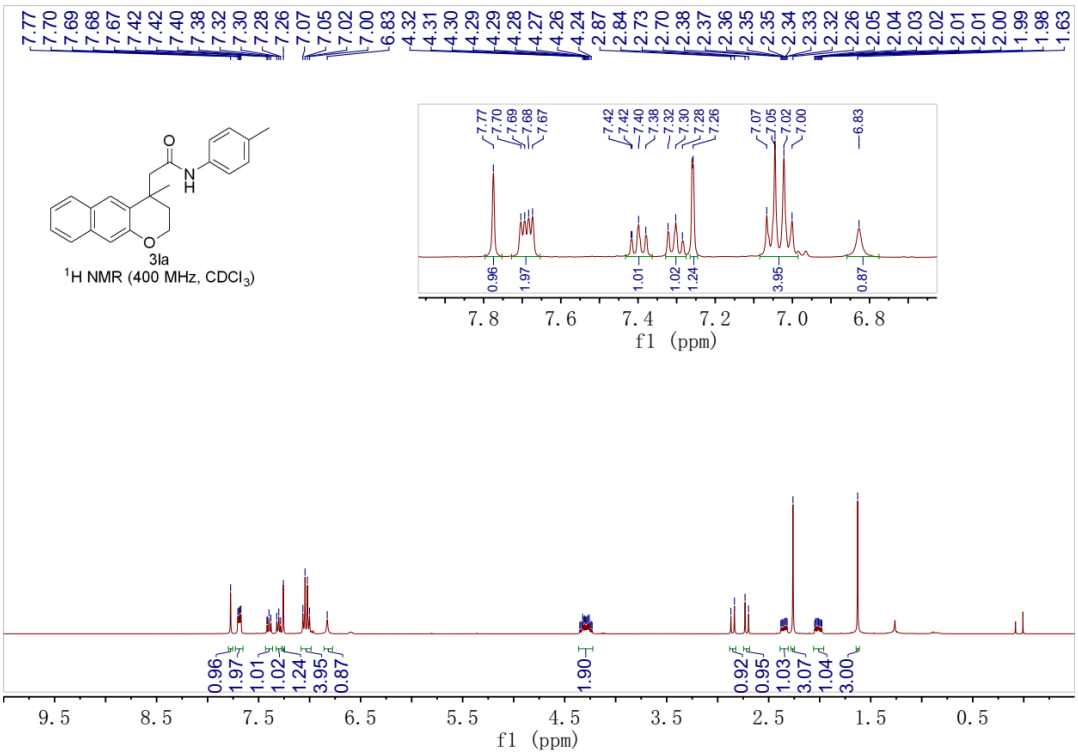

7.19 7.17 7.13 7.07 7.05 6.98 6.98 6.96 6.95 6.87 6.87 6.85 6.84 6.83 6.82 6.80 6.79 6.78 6.77 4.20 4.19 4.18 4.17 4.16 4.15 4.14 4.13 4.12 4.11 4.10 2.69 2.66 2.59 2.55 2.33 2.32 2.31 2.30 2.28 1.92 1.91 1.90 1.89 1.88 1.87 1.86 1.86 1.47
O
N
H
F
O
3ia
^{1}H NMR (400 MHz, $CDCl_3$)
2.05 0.89 2.07 1.03 1.01 1.00
7.25 7.10 6.95 6.80
f1 (ppm)
2.05 0.89 2.07 1.03 1.01 1.00 2.07 1.00 1.02 4.15 0.99 3.00
9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)

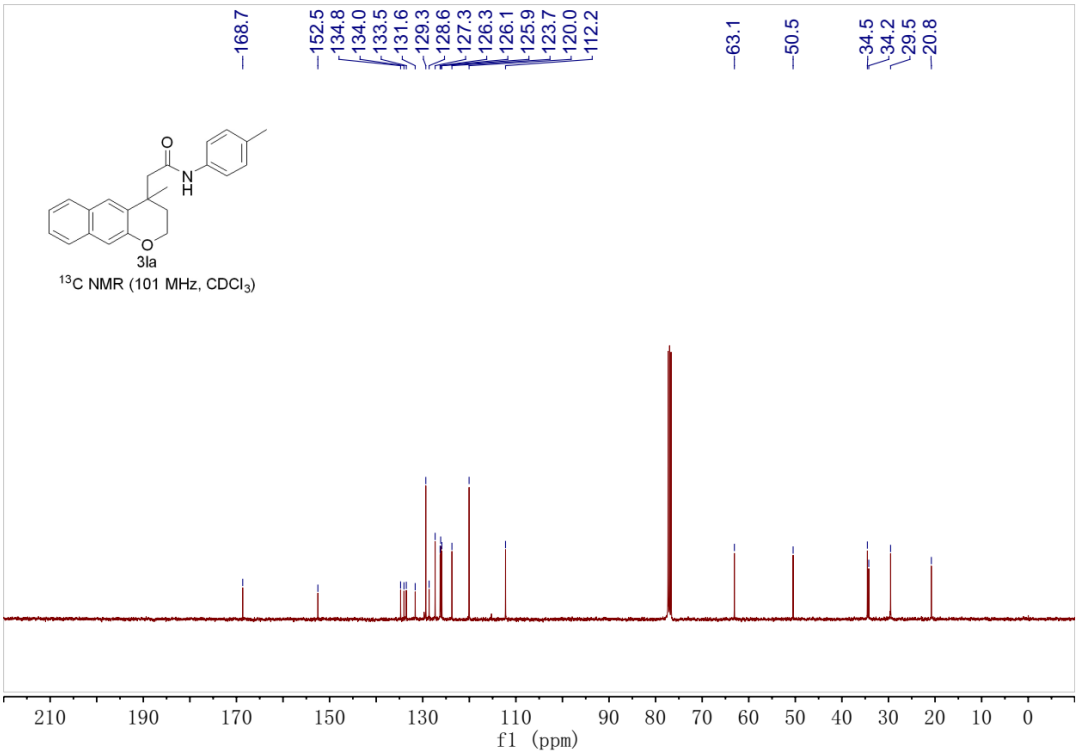

168.5 158.1 155.7 150.3 150.2 134.8 134.2 130.4 129.4 129.2 118.7 118.6 114.9 114.7 112.7 112.5 62.9 49.9 34.0 33.9 29.2 20.8
O
N
H
F
O
3ia
^{13}C NMR (101 MHz, $CDCl_3$)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

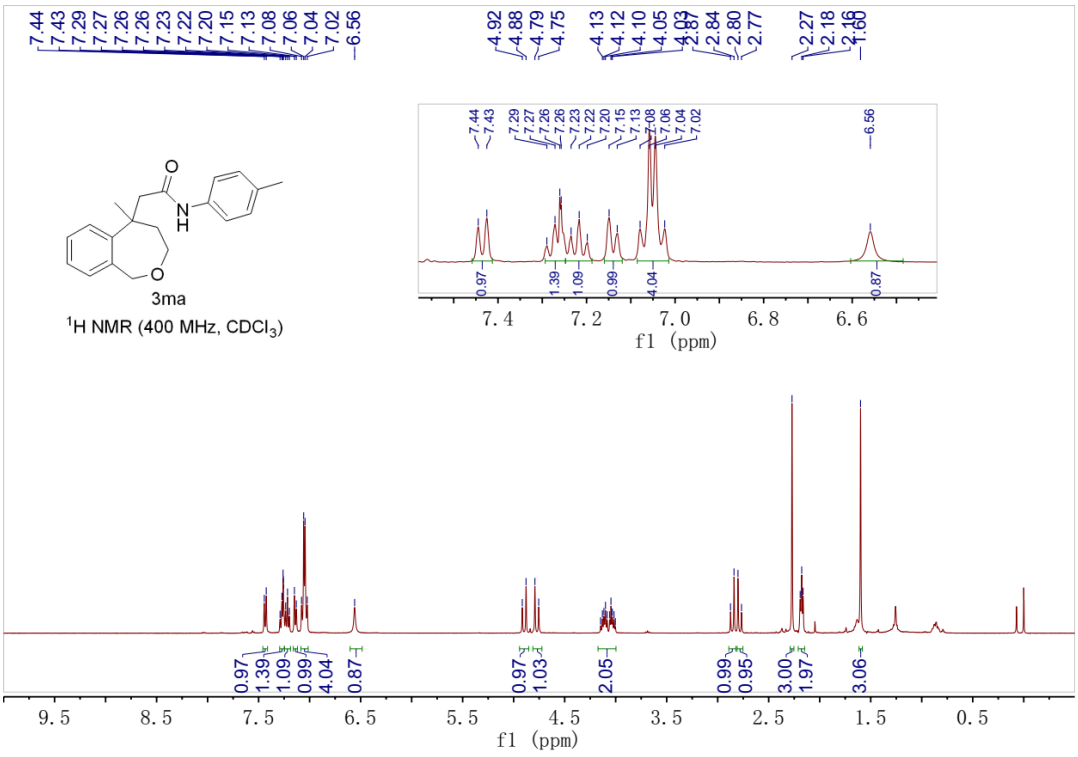

7.23 7.23 7.21 7.19 7.17 7.10 7.09 7.08 7.05 6.79 6.76
4.22 4.20 4.19 4.18 4.18 4.17 4.16 4.15 4.14 4.13 4.12 2.68 2.64 2.58 2.55 2.34 2.33 2.31 2.29 1.92 1.92 1.91 1.90 1.89 1.88 1.87 1.86 1.47
Cl
O
N
H
O
3ja
1H NMR (400 MHz, CDCl3)
0.98 2.04 0.90 3.04 0.96
2.01
0.98 1.00 4.11 1.00 3.00
f1 (ppm)
168.5 152.8 134.7 134.2 131.0 129.4 127.8 126.4 125.3 120.3 119.0
62.9 49.7 33.9 33.8 28.9 20.8
3ja
13C NMR (101 MHz, CDCl3)
f1 (ppm)

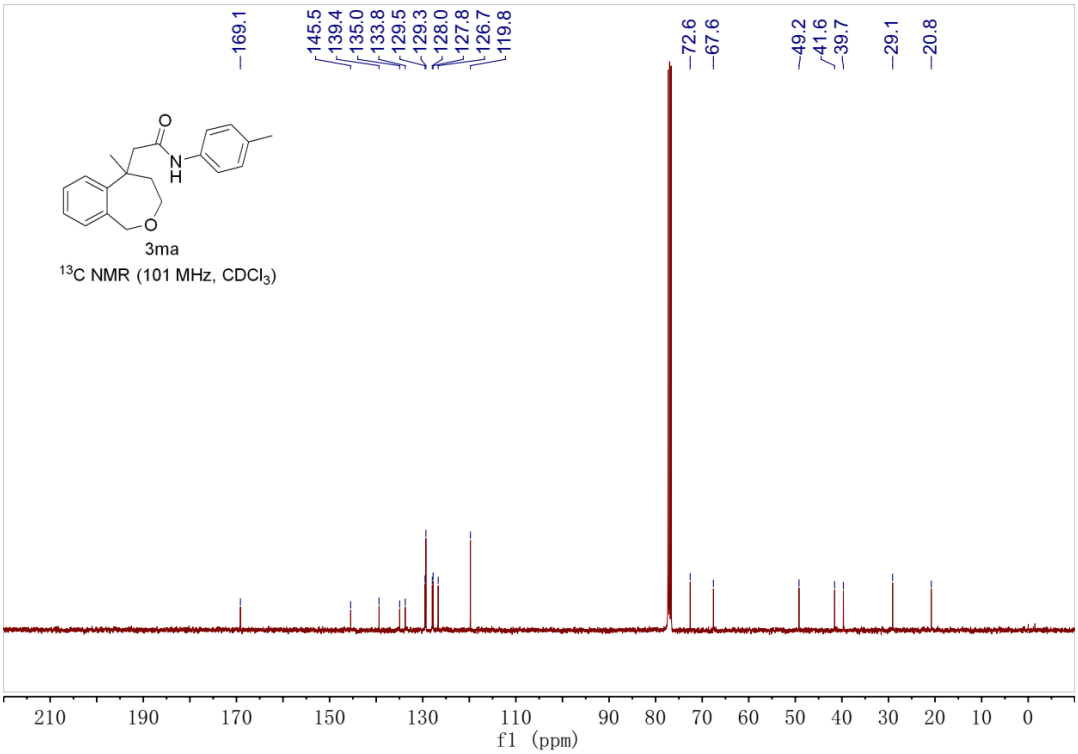

7.51 7.51 7.48 7.42 7.41 7.40 7.39 7.39 7.38 7.33 7.31 7.29 7.15 7.13 7.04 7.02 6.98 6.95 6.93 4.27 4.26 4.25 4.24 4.23 4.22 4.21 4.20 4.19 4.18 4.17 2.81 2.78 2.67 2.63 2.33 2.32 2.31 2.31 2.30 2.29 2.27 1.98 1.97 1.96 1.96 1.95 1.94 1.93 1.92 1.56
Ph
O
N
H
O
3ka
^{1}H NMR (400 MHz, CDCl$_3$)
3.05 3.04 1.04 2.00 2.03 0.88 1.00 1.94 0.93 0.00 0.92 4.04 0.96 3.00
f1 (ppm)

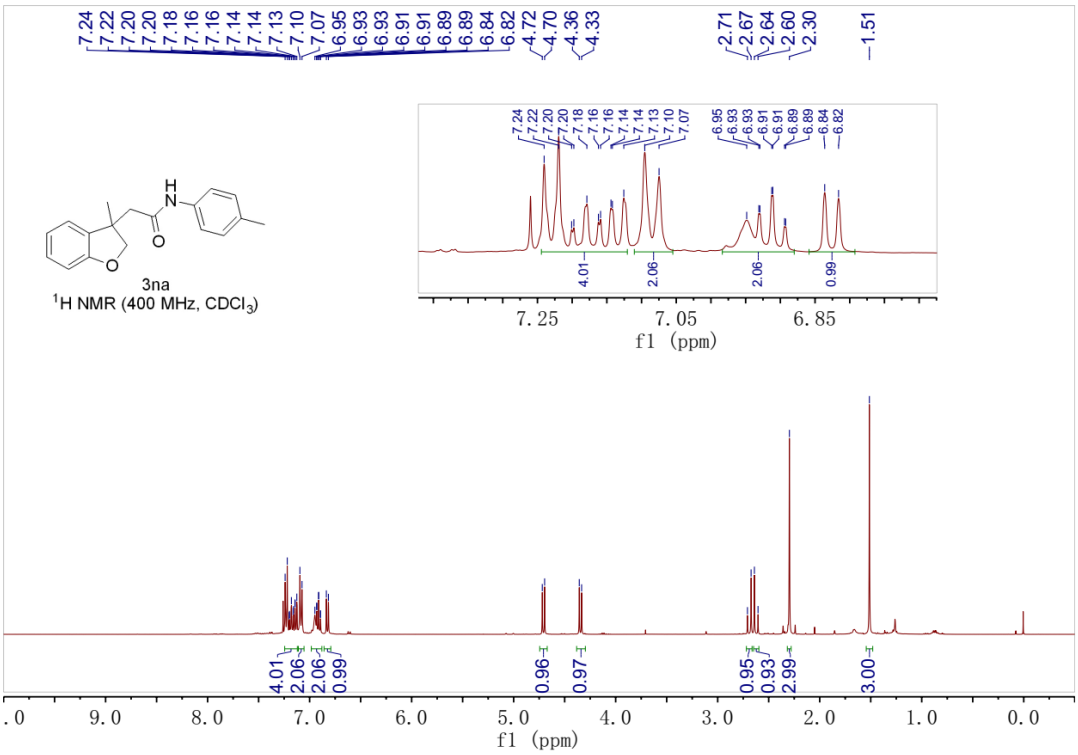

168.7 153.9 140.7 134.9 134.0 133.9 129.3 129.2 128.7 126.8 126.8 126.7 125.3 119.9 118.1 62.9 50.4 34.5 33.8 29.4 20.8
Ph
O
N
H
O
3ka
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

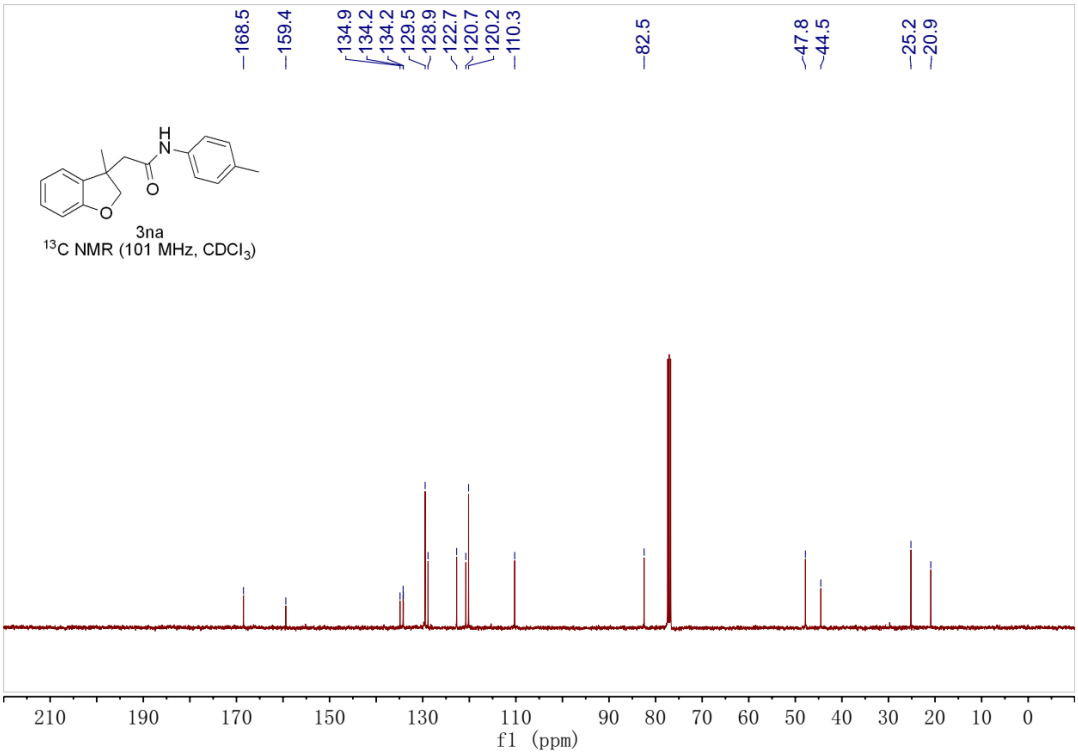

7.77 7.70 7.69 7.68 7.67 7.42 7.42 7.40 7.38 7.32 7.30 7.28 7.26 7.07 7.05 7.02 7.00 6.83 4.32 4.31 4.30 4.29 4.29 4.28 4.27 4.26 4.24 2.87 2.84 2.73 2.70 2.38 2.37 2.36 2.35 2.35 2.34 2.33 2.32 2.26 2.05 2.04 2.03 2.02 2.01 2.01 2.00 1.99 1.98 1.63
3la
^{1}H NMR (400 MHz, $CDCl_3$)
0.96 1.97 1.01 1.02 1.24 3.95 0.87 1.90 0.92 0.95 1.03 3.07 1.04 3.00
f1 (ppm)

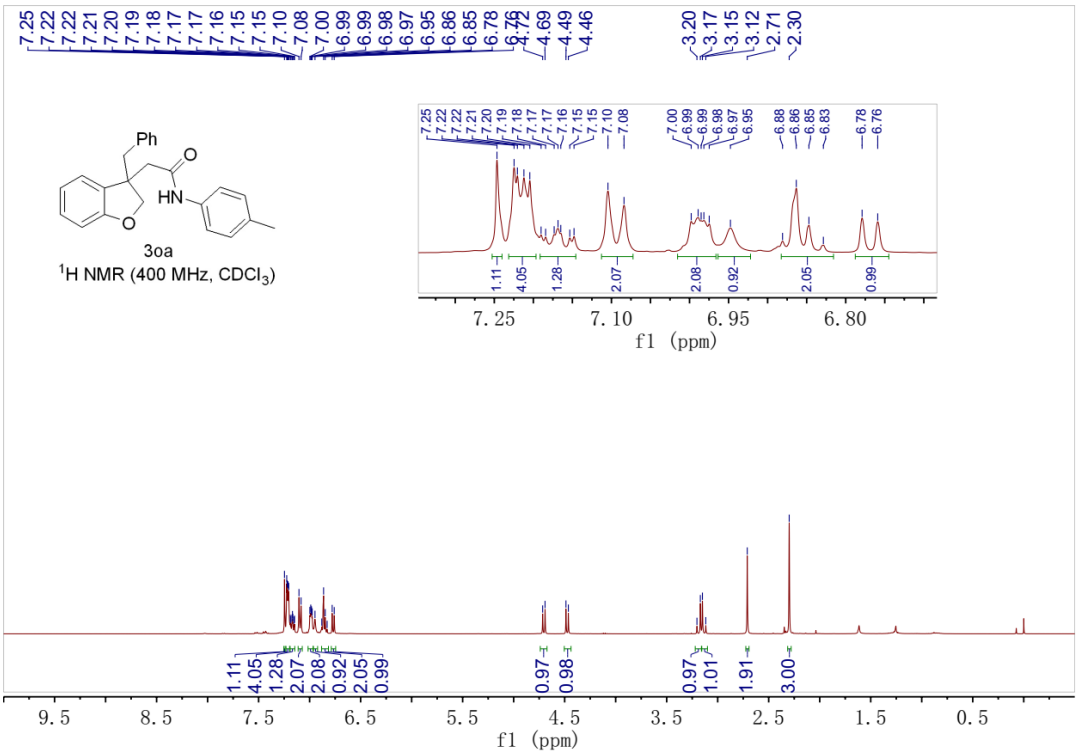

168.7 152.5 134.8 134.0 133.5 131.6 129.3 128.6 127.3 126.3 126.1 125.9 123.7 120.0 112.2 63.1 50.5 34.5 34.2 29.5 20.8
3la
^{13}C NMR (101 MHz, $CDCl_3$)
f1 (ppm)

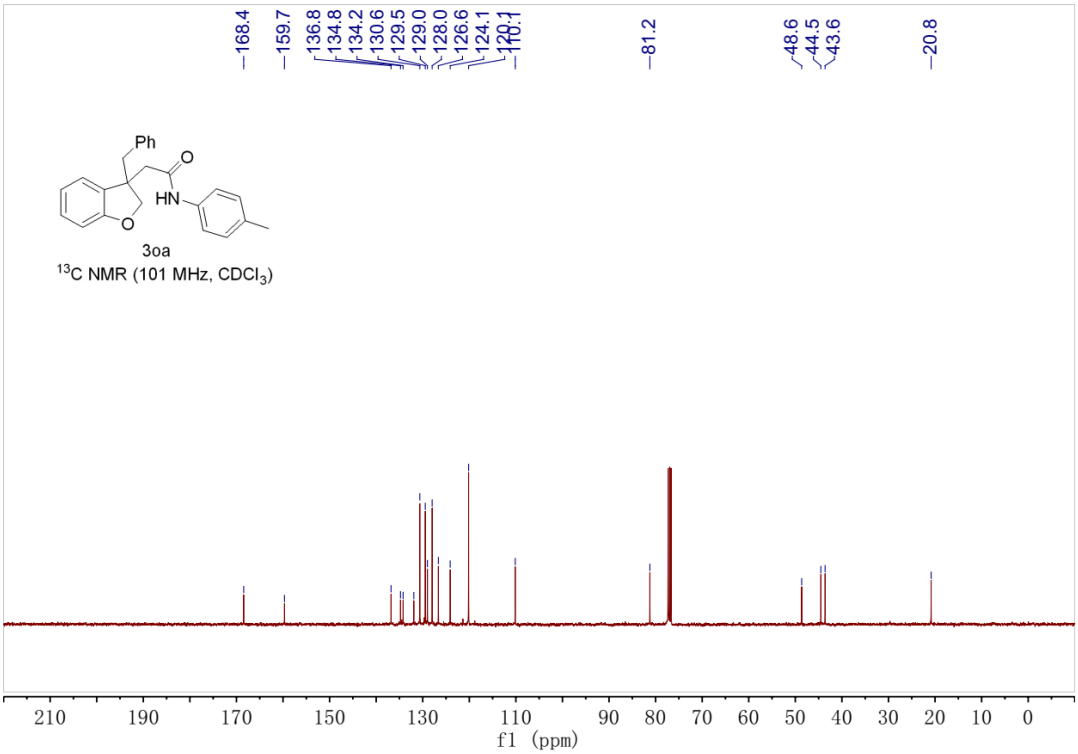

7.44
7.43
7.29
7.27
7.26
7.26
7.23
7.22
7.20
7.15
7.13
7.08
7.06
7.04
7.02
6.56
4.92
4.88
4.79
4.75
4.13
4.12
4.10
4.05
2.84
2.80
2.77
2.27
2.18
2.16
1.60
O
N
H
O
3ma
1H NMR (400 MHz, CDCl3)
0.97
1.39
1.09
0.99
4.04
0.87
0.97
1.03
2.05
0.99
0.95
3.00
1.97
3.06
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

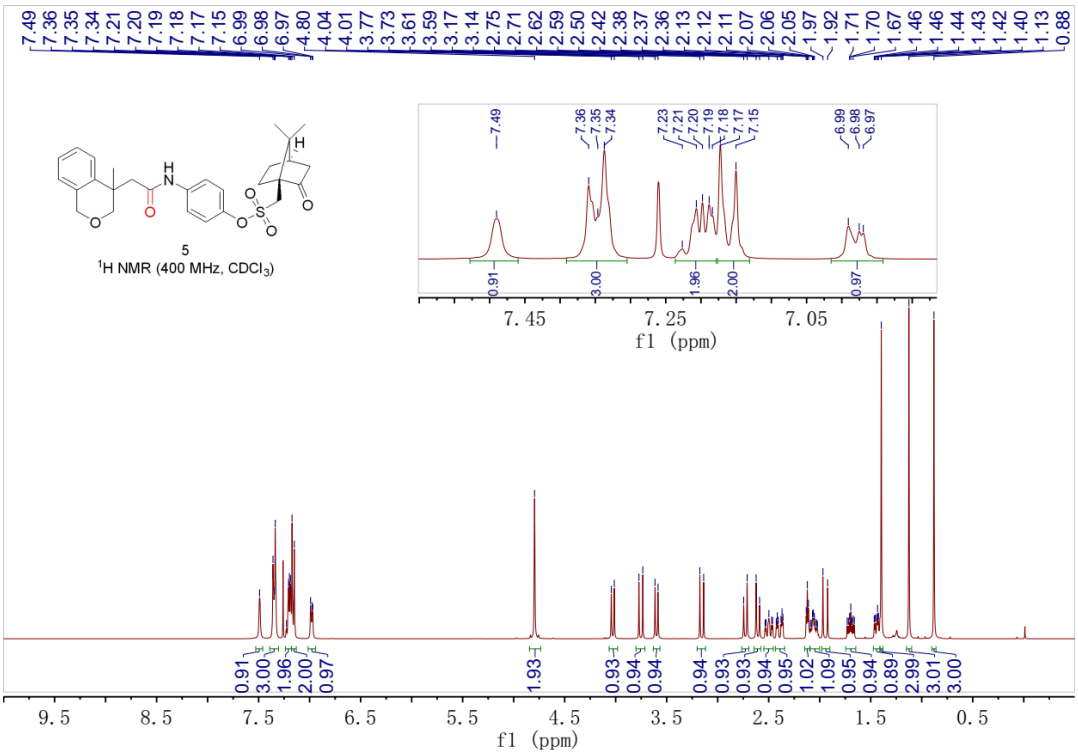

169.1
145.5
139.4
135.0
133.8
129.5
129.3
128.0
127.8
126.7
119.8
72.6
67.6
49.2
41.6
39.7
29.1
20.8
O
N
H
O
3ma
13C NMR (101 MHz, CDCl3)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

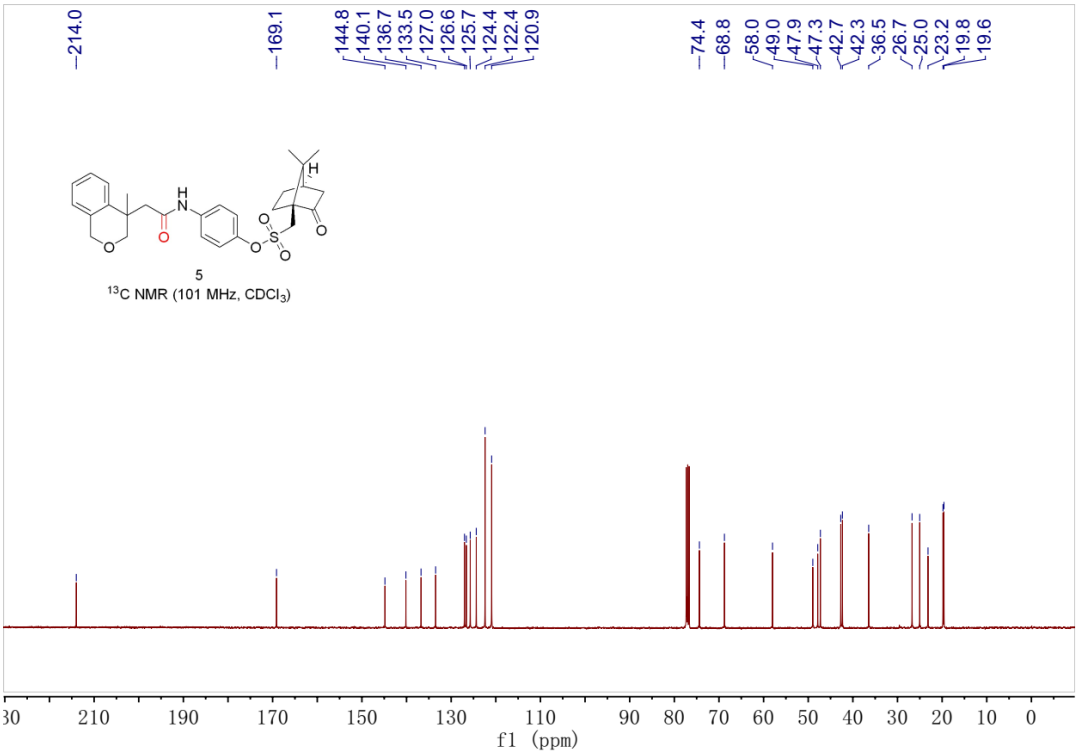

7.24 7.22 7.20 7.20 7.18 7.16 7.16 7.14 7.14 7.13 7.10 7.07 6.95 6.93 6.93 6.91 6.91 6.89 6.89 6.84 6.82 4.72 4.70 4.36 4.33 2.71 2.67 2.64 2.60 2.30 1.51
3na
1H NMR (400 MHz, CDCl3)
4.01 2.06 2.06 0.99 0.96 0.97 0.95 0.93 2.99 3.00
f1 (ppm)

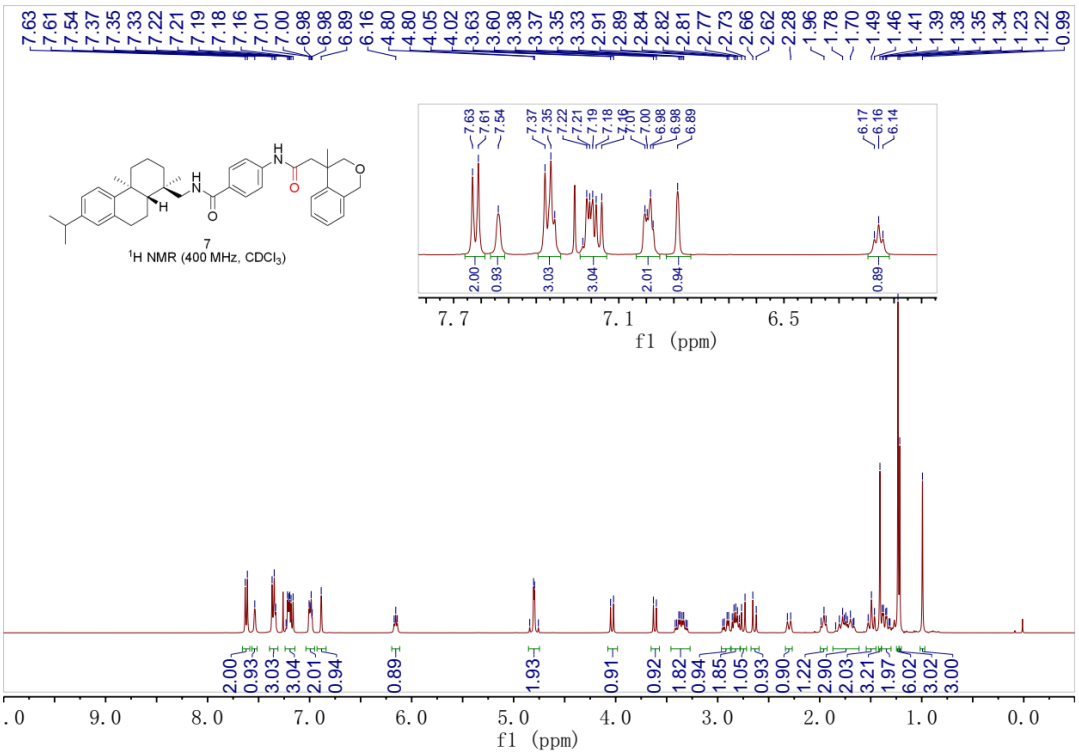

168.5 159.4 134.9 134.2 134.2 129.5 128.9 122.7 120.7 120.2 110.3 82.5 47.8 44.5 25.2 20.9
3na
13C NMR (101 MHz, CDCl3)
f1 (ppm)

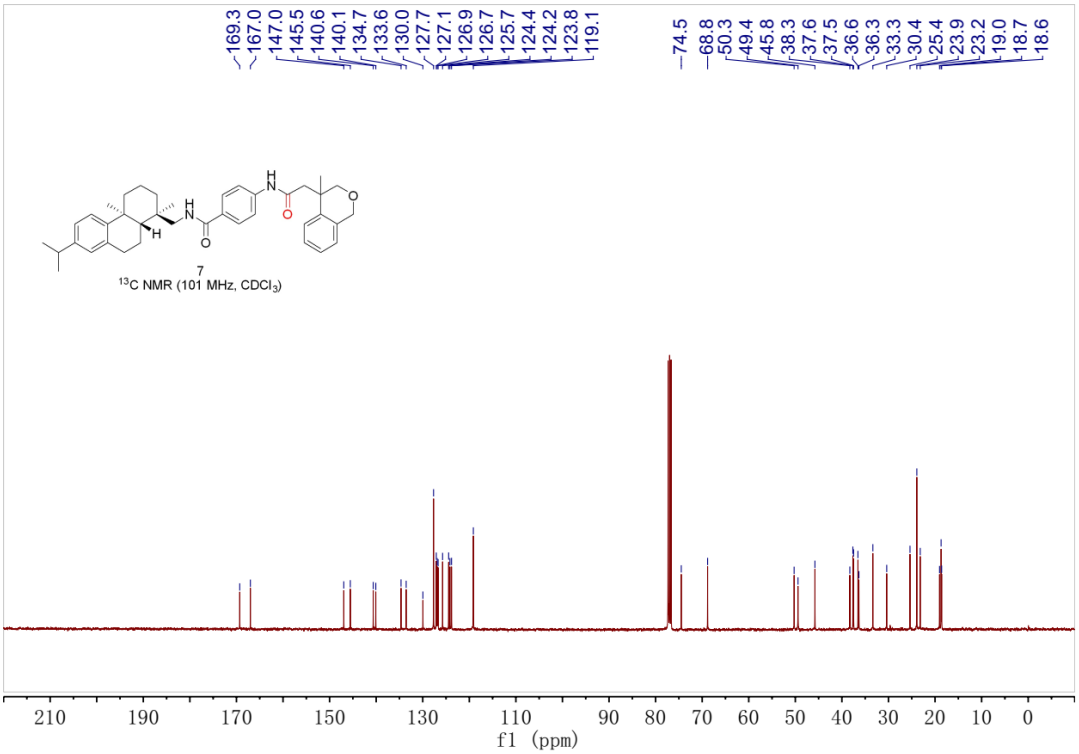

7.25 7.22 7.22 7.21 7.20 7.19 7.18 7.17 7.17 7.16 7.15 7.15 7.10 7.08 7.00 6.99 6.99 6.98 6.97 6.95 6.86 6.85 6.78 6.76 4.69 4.49 4.46
3.20 3.17 3.15 3.12 2.71 2.30
Ph
O
HN
O
3oa
^{1}H NMR (400 MHz, CDCl$_3$)
1.11 4.05 1.28 2.07 2.08 0.92 2.05 0.99
7.25 7.10 6.95 6.80
f1 (ppm)
1.11 4.05 1.28 2.07 2.08 0.92 2.05 0.99
0.97 0.98 0.97 1.01 1.91 3.00
9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)


168.4 159.7 136.8 134.8 134.2 130.6 129.5 129.0 128.0 126.6 124.1 120.1 110.1
81.2
48.6 44.5 43.6
20.8
Ph
O
HN
O
3oa
^{13}C NMR (101 MHz, CDCl$_3$)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.49 7.36 7.35 7.34 7.21 7.20 7.19 7.18 7.17 7.15 6.99 6.98 6.97 4.80 4.04 4.01 3.77 3.73 3.61 3.59 3.17 3.14 2.75 2.71 2.62 2.59 2.50 2.42 2.38 2.37 2.36 2.13 2.12 2.11 2.07 2.06 2.05 1.97 1.92 1.71 1.70 1.67 1.46 1.46 1.44 1.43 1.42 1.40 1.13 0.88
5
1H NMR (400 MHz, CDCl3)
7.49 7.36 7.35 7.34 7.23 7.21 7.20 7.19 7.18 7.17 7.15 6.99 6.98 6.97
0.91 3.00 1.96 2.00 0.97
7.45 7.25 7.05
f1 (ppm)
0.91 3.00 1.96 2.00 0.97 1.93 0.93 0.94 0.94 0.94 0.93 0.93 0.94 0.95 1.02 1.09 0.95 0.94 0.89 2.99 3.01 3.00
9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)


214.0 169.1 144.8 140.1 136.7 133.5 127.0 126.6 125.7 124.4 122.4 120.9 74.4 68.8 58.0 49.0 47.9 47.3 42.7 42.3 36.5 26.7 25.0 23.2 19.8 19.6
5
13C NMR (101 MHz, CDCl3)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.63 7.61 7.54 7.37 7.35 7.33 7.22 7.21 7.19 7.18 7.16 7.01 7.00 6.98 6.98 6.89 6.16 4.80 4.80 4.05 4.02 3.63 3.60 3.38 3.37 3.35 3.33 2.91 2.89 2.84 2.82 2.81 2.77 2.73 2.66 2.62 2.28 1.96 1.78 1.70 1.49 1.46 1.41 1.39 1.38 1.35 1.34 1.23 1.22 0.99
7
1H NMR (400 MHz, CDCl3)
6.17 6.16 6.14
2.00 0.93 3.03 3.04 2.01 0.94 0.89
7.7 7.1 6.5
f1 (ppm)
2.00 0.93 3.03 3.04 2.01 0.94 0.89 1.93 0.91 0.92 1.82 0.94 1.85 1.05 0.93 0.90 1.22 2.90 2.03 3.21 1.97 6.02 3.02 3.00
.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


169.3 167.0 147.0 145.5 140.6 140.1 134.7 133.6 130.0 127.7 127.1 126.9 126.7 125.7 124.4 124.2 123.8 119.1
74.5 68.8 50.3 49.4 45.8 38.3 37.6 37.5 36.6 36.3 33.3 30.4 25.4 23.9 23.2 19.0 18.7 18.6
7
13C NMR (101 MHz, CDCl3)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)