Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Palladium-catalyzed double carbonylation of propargyl amines and aryl halides to access 1-aroyl-3-aryl-1,5-dihydro-2*H*-pyrrol-2-ones

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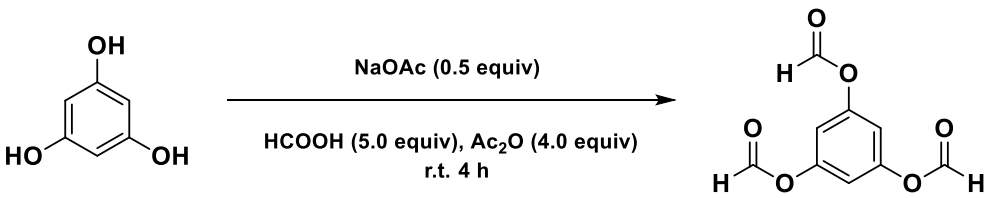
1. General Information

Unless otherwise noted, all reactions were performed under nitrogen protection unless otherwise noted. All reagents were obtained from commercial sources and used as received without further purification. Column chromatography was performed on silica gel (200−300 mesh) using petroleum ether (bp 60−90 °C) and ethyl acetate as eluent. Reactions were followed with TLC (0.25 mm silica gel 20 cm×20 cm). Visualization was accomplished with UV light. ^{1}H and ^{13}C NMR spectra were taken on 400 MHz instruments, and spectral data were reported in ppm relative to tetramethylsilane (TMS) as internal standard and $CDCl_3$ as solvent.

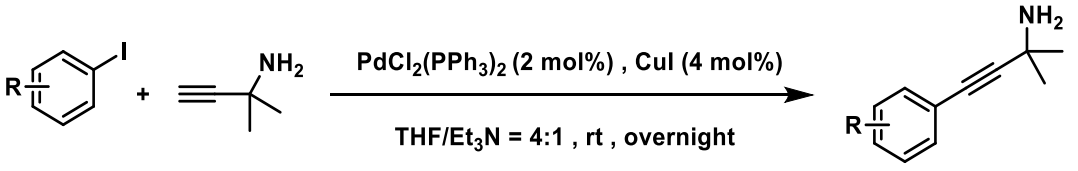
2. Preparation of TFBen



Formic acid (8.4 mL, 222.8 mmol, 5.0 equiv.) was added to acetic anhydride (16.8 mL, 178.2 mmol, 4.0 equiv) at rt. The mixture was stirred at 60 °C for 1 h and cooled to rt. The resulting solution was poured into a flask containing 1,3,5-trihydroxybenzene (5.62 g, 44.6 mmol, 1.0 equiv) and NaOAc (1.83 g, 22.3 mmol, 0.5 equiv). The mixture was stirred for 4 h in a water bath and then diluted with toluene (100 mL), washed with H_2O (50 mL) twice. Keep the organic phase in fridge (2-8 °C) overnight. Then filtered and dried in vacuo to afford the desired product benzene-1,3,5-triyl triformate (TFBen) (5.1 g, 55%) as a white solid.

3. Procedure for the Synthesis of Propargyl Amines

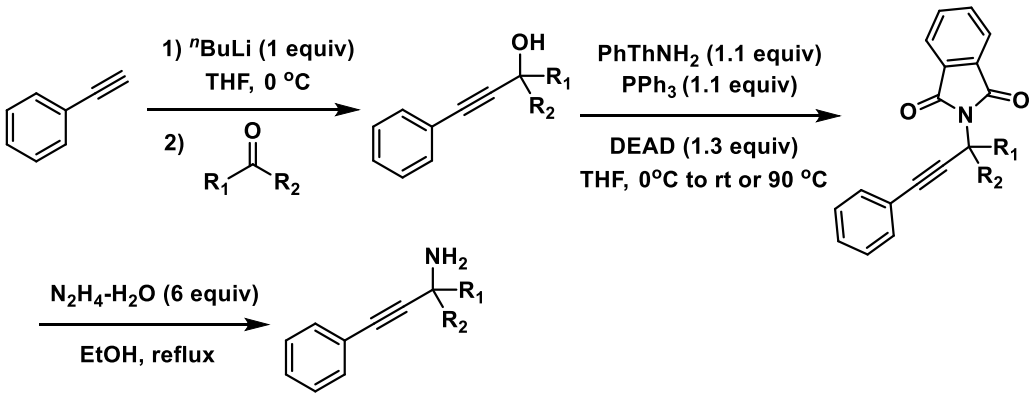
3.1 General procedure for the synthesis of propargyl amine (1a, 1aa-1am)[1]



To a solution of CuI (38.1 mg, 0.20 mmol, 4.0 mol %), $PdCl_2(PPh_3)_2$ (70.2 mg,

0.10 mmol, 2 mol %), and an aryl iodide (5.5 mmol, 1.1 equiv) in THF/Et_3N (4/1, 0.45 M) was added under nitrogen. Then, a propargyl amine (5.0 mmol, 1.0 equiv) was added and the mixture was stirred overnight at rt. A saturated NH_4Cl aqueous solution was added to the reaction mixture upon completion, then extracted with Et_2O three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (PE/EA/Et_3N=2/1/1%) to obtain the corresponding propargyl amines in high yields.

3.2 General procedure for the synthesis of propargyl amines (1ap and 1aq)[2]



3.2.1 General procedure for the synthesis of propargyl alcohols

To a solution of an alkyne (10 mmol, 1.0 equiv) in THF (28 mL) at 0 °C was added a solution of n-butyllithium (2.5 M in THF, 4 mL, 10 mmol, 1.0 equiv) drop-wise. After stirring for 30 min at 0 °C, an aldehyde (12 mmol, 1.2 equiv) was added and the mixture was stirred for an additional 30 min before a saturated aqueous solution of NH_4Cl (50 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with EtOAc (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography (PE/EA=10/1) over silica gel to afford pure propargyl alcohols.

3.2.2 General procedure for the synthesis of phthalimides

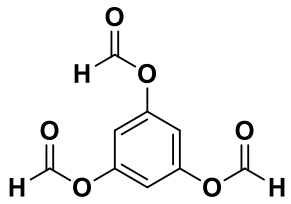
To a solution of a propargyl alcohol (10 mmol, 1.0 equiv) in THF (100 mL) at 0 °C was added triphenylphosphine (2.89 g, 11 mmol, 1.1 equiv), a phthalimide (1.62 g, 11 mmol, 1.1 equiv) and a solution of DEAD (40% wt in toluene, 5.9 mL, 13 mmol,

1.3 equiv). The reaction mixture was then stirred for 20 h at rt before the solvent was evaporated and triphenylphosphine oxide was precipitated by the addition of a 1:1 mixture of Et_2O/PE (40 mL). The precipitate was filtered through a plug of Celite and the solvent was evaporated under reduced pressure to afford a crude residue which was purified by flash column chromatography (PE/EA=10/1) over silica gel.

3.2.3 General procedure for the cleavage of phthalimides

To a solution of a phthalimide (3 mmol, 1 equiv) in EtOH (26 mL) was added hydrazine hydrate (0.9 mL, 18 mmol, 6 equiv) drop-wise and the resulting mixture was stirred at reflux for 3 h. The formed pasty precipitate was then filtered through a plug of Celite, washed with Et_2O (20 mL) and the solvent was evaporated under reduced pressure. The crude residue was finally purified by flash column chromatography (PE/EA/Et_3N=2/1/1%) over silica gel to afford the desired pure propargyl amines.

4. Characterization of TFBen and Substrates

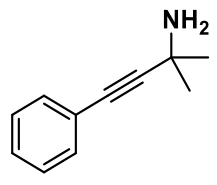


Benzene-1,3,5-triyl triformate, TFBen[3]

White solid, mp. 53.2-55.6 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.24 (s, 3H), 6.97 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.06, 150.30, 112.62.



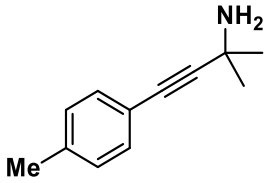
1a

2-Methyl-4-phenylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 90% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39 – 7.29 (m, 2H), 7.21 (dd, *J* = 5.1, 2.0 Hz, 3H), 1.68 (s, 2H), 1.43 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 131.4, 128.2, 127.7, 123.4, 97.1, 79.9, 45.5, 31.8.



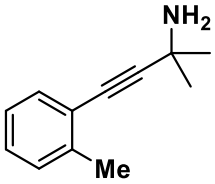
1aa

2-Methyl-4-(*p*-tolyl)but-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 86% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.30 (d, *J* = 8.1 Hz, 1H), 7.09 (d, *J* = 7.9 Hz, 2H), 2.33 (s, 2H), 1.85 (s, 2H), 1.50 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 137.7, 131.4, 128.9, 120.3, 96.2, 80.1, 45.6, 31.8, 21.37.



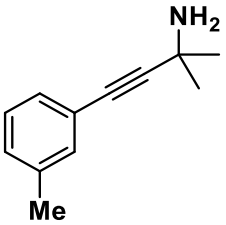
1ab

2-Methyl-4-(*m*-tolyl)but-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 78% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32 – 7.15 (m, 3H), 7.09 (d, *J* = 7.3 Hz, 1H), 2.32 (s, 3H), 1.78 (s, 2H), 1.50 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 137.8, 132.1, 128.7, 128.5, 128.1, 96.7, 80.1, 45.6, 31.8, 21.1.



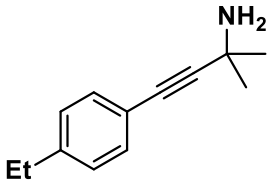
1ac

2-Methyl-4-(*m*-tolyl)but-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 78% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32 – 7.15 (m, 3H), 7.09 (d, *J* = 7.3 Hz, 1H), 2.32 (s, 3H), 1.78 (s, 2H), 1.50 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 137.8, 132.1, 128.7, 128.5, 128.1, 96.7, 80.1, 45.6, 31.8, 21.1.



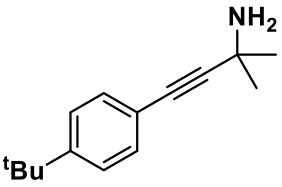
1ad

4-(4-Ethylphenyl)-2-methylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 90% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32 (d, *J* = 8.1 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 2.62 (q, *J* = 7.6 Hz, 2H), 1.87 (s, 2H), 1.49 (s, 6H), 1.21 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, CDCl3): δ = 144.1, 131.5, 127.7, 120.6, 96.3, 80.1, 45.6, 31.8, 28.7, 15.3.



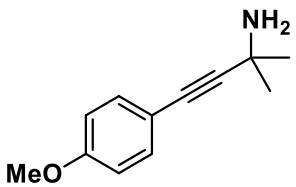
1ae

(4-(*tert*-Butyl)phenyl)-2-methylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 91% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39 – 7.30 (m, 4H), 1.80 (s, 2H), 1.52 (s, 6H), 1.33 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.9, 131.2, 125.2, 120.4, 96.4, 80.0, 45.6, 34.7, 31.9, 31.2.



1af

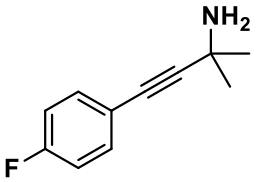
4-(4-Methoxyphenyl)-2-methylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 94% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36 (d, *J* = 7.0 Hz, 2H), 6.86 (d, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 1.88 (s, 2H), 1.53 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.3, 132.9, 115.5, 113.8, 95.5, 79.8, 55.2, 45.7, 31.9.

1ag



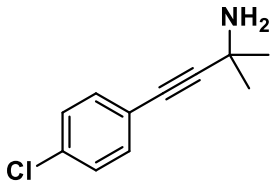
4-(4-Fluorophenyl)-2-methylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 73% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38 – 7.23 (m, 2H), 6.94 (t, *J* = 8.7 Hz, 2H), 1.71 (s, 2H), 1.45 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 162.2 (d, *J* = 246.2 Hz), 133.3 (d, *J* = 8.2 Hz), 119.4, 115.3 (d, *J* = 22.0 Hz), 96.6, 78.9, 45.6, 31.8.

1ah

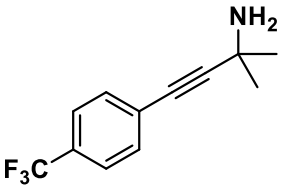


4-(4-Chlorophenyl)-2-methylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 75% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.31 – 7.28 (m, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 1.80 (s, 2H), 1.47 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 133.7, 133.0, 132.7, 128.5, 121.9, 97.9, 78.9, 45.6, 31.7.



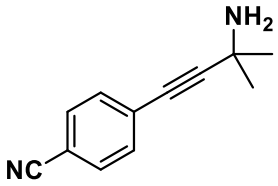
1ai

2-Methyl-4-(4-(trifluoromethyl)phenyl)but-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 80% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54 (d, *J* = 8.2 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 1.80 (s, 2H), 1.51 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 131.7, 129.6 (q, *J* = 32.5 Hz), 127.3, 125.1 (q, *J* = 3.8 Hz), 123.9 (q, *J* = 272.2 Hz), 99.5, 78.9, 45.7, 31.6.



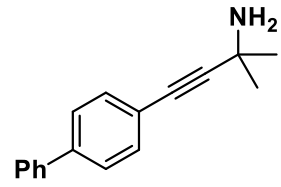
1aj

4-(3-Amino-3-methylbut-1-yn-1-yl)benzonitrile[1]

This compound was obtained as a yellow oil in 82% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 1.82 (s, 2H), 1.52 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 132.0, 131.9, 128.4, 118.5, 111.2, 101.6, 78.8, 45.8, 31.6.



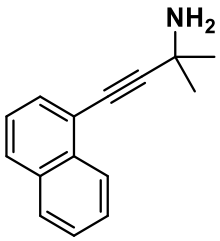
1ak

4-([1,1'-Biphenyl]-4-yl)-2-methylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 90% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.64 – 7.43 (m, 8H), 7.39 (d, *J* = 7.4 Hz, 1H), 1.81 (s, 2H), 1.57 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 140.5, 140.4, 132.0, 128.9, 127.6, 127.0, 126.9, 97.8, 79.9, 45.7, 31.9.



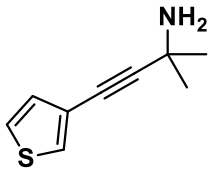
1al

2-Methyl-4-(naphthalen-1-yl)but-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 87% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (d, *J* = 8.3 Hz, 1H), 7.85 (dd, *J* = 19.0, 8.1 Hz, 2H), 7.71 – 7.52 (m, 3H), 7.49 – 7.40 (m, 1H), 1.96 (s, 2H), 1.66 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 133.3, 133.2, 130.1, 128.3, 128.2, 126.7, 126.3, 126.1, 125.2, 121.0, 102.2, 78.2, 46.0, 32.0.



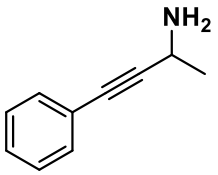
1am

2-Methyl-4-(thiophen-3-yl)but-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 75% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33 (dd, *J* = 2.9, 1.1 Hz, 1H), 7.19 (dd, *J* = 4.9, 3.0 Hz, 1H), 7.04 (dd, *J* = 5.0, 1.0 Hz, 1H), 1.77 (s, 2H), 1.44 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 129.9, 127.9, 125.1, 122.3, 96.6, 75.1, 45.6, 31.8.



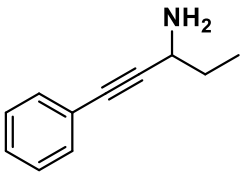
1ap

4-Phenylbut-3-yn-2-amine[1]

This compound was obtained as a yellow oil in 30% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.32 (dd, *J* = 6.8, 3.9 Hz, 3H), 3.96 (dd, *J* = 13.3, 6.6 Hz, 1H), 1.67 (s, 2H), 1.48 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 131.5, 128.2, 127.9, 123.3, 94.0, 81.5, 39.4, 24.6.



1aq

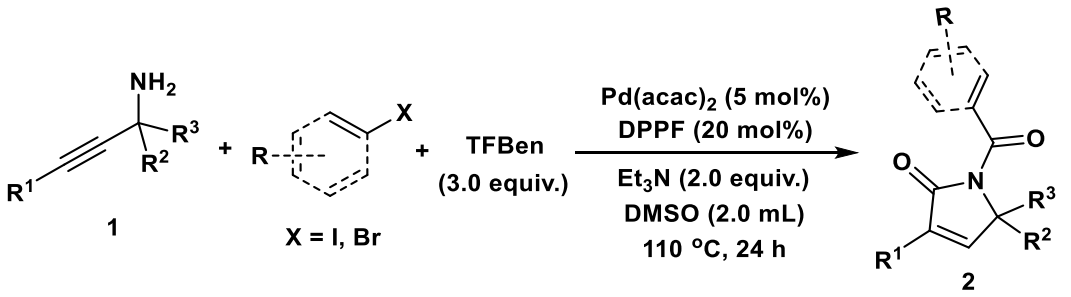
1-Phenylpent-1-yn-3-amine[1]

This compound was obtained as a yellow oil in 35% yield.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50 – 7.38 (m, 2H), 7.31 (dd, *J* = 4.5, 2.0 Hz, 3H), 3.75 (t, *J* = 6.7 Hz, 1H), 1.68 (s, 2H), 1.11 (t, *J* = 7.4 Hz, 3H).

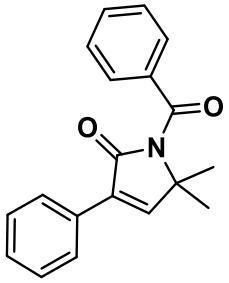
^{13}C NMR (100 MHz, $CDCl_3$): δ = 131.6, 128.2, 127.9, 123.4, 92.8, 82.6, 45.5, 31.3, 10.4.

5. General Procedure for Palladium-Catalyzed Double Carbonylation of Propargyl Amines, Aryl Halides, and TFBen



To a solution of propargyl amine **1** (0.5 mmol, 1.0 equiv), $Pd(acac)_2$ (7.6 mg, 5 mol %) , DPPF (55.4 mg, 20 mol %), and a 2.5 mL vial containing TFBen (315 mg, 1.5 mmol, 3.0 equiv) were added to an oven-dried tube (15 mL) which was then placed under vacuum and refilled with nitrogen three times. Et_3N (140 μL, 1.0 mmol, 2.0 equiv), an aryl halide (0.6 mmol, 1.2 equiv) and DMSO (2.0 mL) were added into the tube via syringe. The tube was sealed and stirred at 110 °C for 24 h. Upon the reaction was completed, the resulting mixture was filtered through a pad of tripolite and washed with EtOAc. The filtrate was concentrated under vacuum and purified by silica gel column using chromatography (petroleum ether / acetone) to obtain the desired products **2**.

6. Characterization of Products



2a

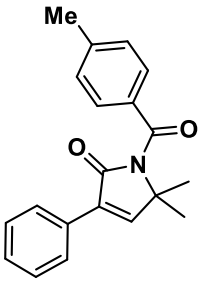
1-Benzoyl-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (116.4 mg, 80%). mp. 126-128 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89 – 7.86 (m, 2H), 7.64 – 7.55 (m, 3H), 7.52 – 7.43 (m, 5H), 7.43 (s, 1H), 1.83 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.5, 152.6, 136.3, 132.4, 131.3, 130.3, 129.1, 128.6, 128.0, 127.9, 127.3, 63.6, 24.6.

HRMS (ESI): [M+H^+]calcd for $C_{19}H_{18}NO_2^+$,292.1332; found, 292.1352.



2b

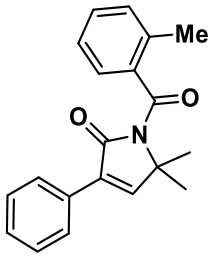
5,5-Dimethyl-1-(4-methylbenzoyl)-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (131.1 mg, 86%). mp. 127-129 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87 (dt, *J* = 4.4, 2.6 Hz, 2H), 7.53 (d, *J* = 8.1 Hz, 2H), 7.47 – 7.42 (m, 3H), 7.41 (s, 1H), 7.29 (d, *J* = 8.2 Hz, 2H), 2.46 (s, 3H), 1.82 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.2, 168.5, 152.4, 141.8, 133.4, 132.4, 130.3, 129.0, 128.7, 128.5, 128.2, 127.3, 63.6, 24.6, 21.7.

HRMS (ESI): [M+Na^+]calcd for $C_{20}H_{19}NNaO_2^+$,328.1308; found, 328.1325.



2c

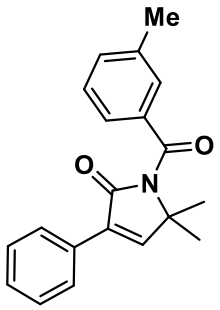
5,5-Dimethyl-1-(2-methylbenzoyl)-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **yellow oil (91.5 mg, 60%).**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84 (dt, *J* = 5.1, 3.2 Hz, 2H), 7.44 (s, 1H), 7.43 – 7.40 (m, 4H), 7.36 – 7.28 (m, 3H), 2.41 (s, 3H), 1.87 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.8, 167.9, 153.0, 137.5, 133.9, 132.3, 130.2, 130.1, 129.4, 129.1, 128.5, 127.3, 125.8, 125.6, 63.3, 24.2, 19.1.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{20}NO_2^+$,306.1489; found, 306.1509.



2d

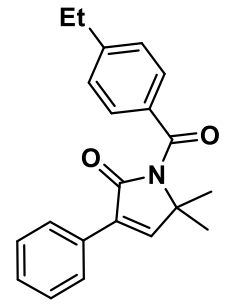
5,5-Dimethyl-1-(3-methylbenzoyl)-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (96.1 mg, 63%). mp. 116-118 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89 – 7.85 (m, 2H), 7.57 – 7.40 (m, 6H), 7.38 (s, 1H), 7.37 (s, 1H), 2.46 (s, 3H), 1.82 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.3, 168.4, 152.6, 137.8, 136.4, 132.4, 132.0, 130.3, 129.1, 128.6, 128.3, 127.8, 127.3, 63.5, 24.6, 21.4.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{20}NO_2^+$,306.1489; found, 306.1505.



2e

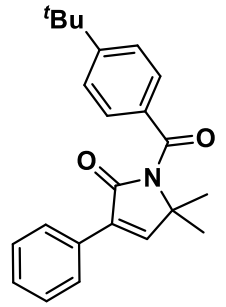
1-(4-Ethylbenzoyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (127.6 mg, 80%). mp. 141-143 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87 – 7.83 (m, 2H), 7.53 (d, *J* = 8.1 Hz, 2H), 7.45 – 7.40 (m, 3H), 7.39 (s, 1H), 7.30 (d, *J* = 8.2 Hz, 2H), 2.75 (q, *J* = 7.6 Hz, 2H), 1.80 (s, 6H), 1.31 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.5, 152.4, 148.0, 133.5, 132.5, 130.3, 129.0, 128.5, 128.3, 127.5, 127.2, 63.6, 28.9, 24.6, 15.1.

HRMS (ESI): [M+H$^+$]calcd for $C_{21}H_{22}NO_2^+$,320.1645; found, 320.1662.



2f

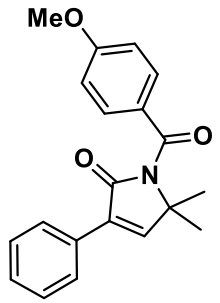
1-(4-(*tert*-Butyl)benzoyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (135.3 mg, 78%). mp. 188-190 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88 – 7.84 (m, 2H), 7.59 – 7.57 (m, 2H), 7.51 – 7.42 (m, 5H), 7.40 (s, 1H), 1.81 (s, 6H), 1.39 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.5, 154.8, 152.5, 133.2, 132.6, 130.4, 129.0, 128.6, 128.3, 127.3, 124.9, 63.6, 35.0, 31.2, 24.7.

HRMS (ESI): [M+H$^+$]calcd for $C_{23}H_{26}NO_2^+$,348.1958; found, 348.1971.



2g

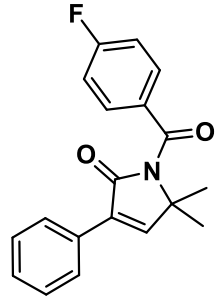
1-(4-Methoxybenzoyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (130.0 mg, 81%). mp. 130-132 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87 (dd, *J* = 7.9, 1.7 Hz, 2H), 7.63 – 7.60 (m, 2H), 7.46 – 7.40 (m, 3H), 7.39 (s, 1H), 6.98 – 6.95 (m, 2H), 3.89 (s, 3H), 1.80 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.6, 168.6, 162.4, 152.4, 132.5, 130.7, 130.4, 129.0, 128.5, 128.2, 127.2, 113.3, 63.7, 55.4, 24.8.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{20}NO_3^+$,322.1438; found, 322.1453.



2h

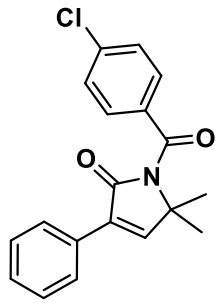
1-(4-Fluorobenzoyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (115.8 mg, 75%). mp. 131-133 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87 – 7.83 (m, 2H), 7.65 – 7.60 (m, 2H), 7.48 – 7.43 (m, 3H), 7.42 (s, 1H), 7.18 – 7.12 (m, 2H), 1.81 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ =168.9, 168.5, 164.6 (d, *J* = 251.8 Hz), 152.7 (d, *J* = 3.1 Hz), 132.4, 132.3 (d, *J* = 3.3 Hz), 130.6 (d, *J* = 8.9 Hz), 130.2, 129.3, 128.7, 127.2, 115.1 (d, J = 22.0 Hz), 63.7, 24.6.

HRMS (ESI): [M+H$^+$]calcd for $C_{19}H_{17}FNO_2^+$,310.1238; found, 310.1256.



2i

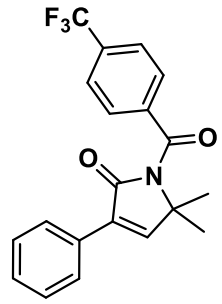
1-(4-Chlorobenzoyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (131.6 mg, 81%). mp. 135-137 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 – 7.81 (m, 2H), 7.55 – 7.51 (m, 2H), 7.45 (s, 1H), 7.44 – 7.41 (m, 5H), 1.80 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.9, 168.5, 152.7, 137.4, 134.6, 132.4, 130.1, 129.4, 129.2, 128.6, 128.3, 127.2, 63.7, 24.5.

HRMS (ESI): [M+H$^+$]calcd for $C_{19}H_{17}ClNO_2^+$,326.0942; found, 326.0958.



2j

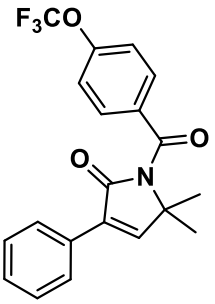
5,5-Dimethyl-3-phenyl-1-(4-(trifluoromethyl)benzoyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (149.0 mg, 83%). mp. 168-170 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84 – 7.80 (m, 2H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.67 (d, *J* = 8.2 Hz, 2H), 7.44 (s, 1H), 7.47 – 7.41 (m, 3H), 1.83 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.6, 168.4, 153.0, 139.8, 132.4, 132.5 (q, *J* = 32.7 Hz), 129.9, 129.3, 128.6, 128.0, 127.2, 125.1 (q, *J* = 3.9 Hz), 123.8 (q, *J* = 272.5 Hz), 63.7, 24.4.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{17}F_3NO_2^+$,360.1206; found, 360.1221.



2k

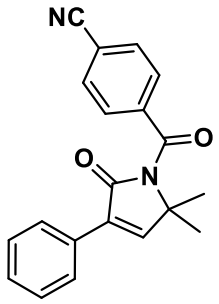
5,5-Dimethyl-3-phenyl-1-(4-(trifluoromethoxy)benzoyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (133.1 mg, 71%). mp. 124-126 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83 – 7.79 (m, 2H), 7.64 – 7.61 (m, 2H), 7.46 – 7.42 (m, 3H), 7.41 (s, 1H), 7.29 (d, *J* = 7.7 Hz, 2H), 1.80 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.6, 168.4, 152.8, 151.2, 134.5, 132.4, 130.1, 129.9, 129.2, 128.6, 127.2, 120.6, 120.4 (q, *J* = 258.3 Hz), 63.7, 24.5.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{17}F_3NO_3^+$,376.1155; found, 376.1165.



2l

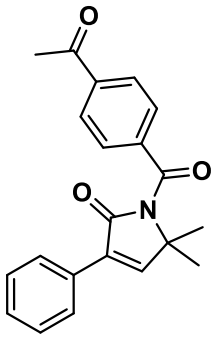
4-(2,2-Dimethyl-5-oxo-4-phenyl-2,5-dihydro-1*H*-pyrrole-1-carbonyl)benzonitrile

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (118.5 mg, 75%). mp. 158-160 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.81 – 7.78 (m, 2H), 7.77 – 7.73 (m, 2H), 7.64 – 7.61 (m, 2H), 7.46 (s, 1H), 7.44 – 7.40 (m, 3H), 1.82 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.5, 168.0, 153.2, 140.5, 132.2, 131.8, 129.8, 129.3, 128.7, 128.1, 127.2, 118.3, 114.3, 63.7, 24.3.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{17}N_2O_2^+$,317.1285; found, 317.1296.



2m

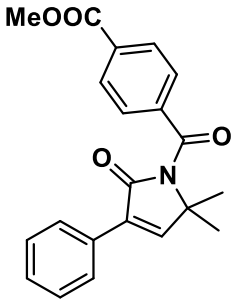
1-(4-Acetylbenzoyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (121.5 mg, 73%). mp. 156-158 ºC**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.09 (s, 2H), 7.84 (s, 2H), 7.66 (s, 2H), 7.46 (s, 1H), 7.44 (s, 3H), 2.71 (s, 3H), 1.86 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 197.4, 169.0, 168.5, 152.9, 140.6, 138.5, 132.3, 130.0, 129.2, 128.6, 128.0, 127.7, 127.2, 63.7, 26.8, 24.4.

HRMS (ESI): [M+H$^+$]calcd for $C_{21}H_{20}NO_3^+$,334.1438; found, 334.1451.



2n

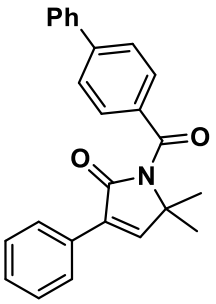
Methyl 4-(2,2-dimethyl-5-oxo-4-phenyl-2,5-dihydro-1*H*-pyrrole-1-carbonyl)benzoate

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (143.1 mg, 82%). mp. 123-125 ºC**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.10 (d, *J* = 8.5 Hz, 2H), 7.78 – 7.74 (m, 2H), 7.57 – 7.55 (m, 2H), 7.38 (s, 1H), 7.37 (dd, *J* = 5.5, 2.0 Hz, 3H), 3.93 (s, 3H), 1.77 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.1, 168.4, 166.4, 152.8, 140.5, 132.3, 132.0, 129.9, 129.3, 129.2, 128.6, 127.5, 127.2, 63.6, 52.3, 24.4.

HRMS (ESI): [M+H$^+$]calcd for $C_{21}H_{20}NO_4^+$,350.1387; found, 350.1405.



2o

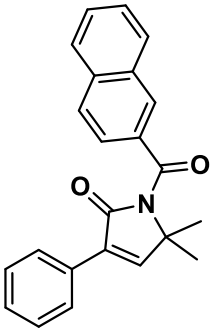
1-([1,1'-Biphenyl]-4-carbonyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (150.5 mg, 82%). mp. 201-203 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.72 – 7.64 (m, 6H), 7.50 (t, *J* = 7.5 Hz, 2H), 7.44 (s, 1H), 7.43 – 7.39 (m, 4H), 1.83 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.8, 168.5, 152.6, 144.1, 140.3, 134.9, 132.5, 130.2, 129.1, 128.8, 128.7, 128.6, 127.8, 127.3, 127.2, 126.7, 63.7, 24.6.

HRMS (ESI): [M+H$^+$]calcd for $C_{25}H_{22}NO_2^+$,368.1645; found, 368.1655.



2p

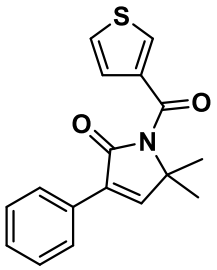
1-(2-Naphthoyl)-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (146.6 mg, 86%). mp. 170-172 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.15 (s, 1H), 7.94 (dd, *J* = 16.9, 8.2 Hz, 3H), 7.88 – 7.83 (m, 2H), 7.65 – 7.55 (m, 3H), 7.44 (s, 1H), 7.42 (dd, *J* = 5.9, 4.6 Hz, 3H), 1.87 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.4, 152.6, 134.6, 133.7, 132.5, 132.4, 130.2, 129.1, 129.0, 128.6, 127.8, 127.6, 127.5, 127.2, 126.5, 124.6, 63.6, 24.6.

HRMS (ESI): [M+H$^+$]calcd for $C_{23}H_{20}NO_2^+$,342.1489; found, 342.1502.



2q

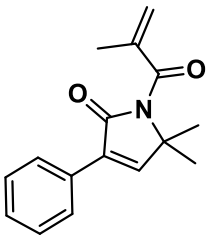
5,5-Dimethyl-3-phenyl-1-(thiophene-3-carbonyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (112.8 mg, 76%). mp. 108-110 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93 (dd, *J* = 2.9, 1.3 Hz, 1H), 7.88 – 7.85 (m, 2H), 7.46 – 7.41 (m, 3H), 7.38 (s, 1H), 7.36 (dd, *J* = 5.1, 1.3 Hz, 1H), 7.33 – 7.30 (m, 1H), 1.78 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.4, 164.3, 152.5, 137.3, 132.5, 131.0, 130.3, 129.1, 128.6, 127.8, 127.2, 124.6, 63.8, 24.7.

HRMS (ESI): [M+H$^+$]calcd for $C_{17}H_{16}NO_2S^+$,298.0896; found, 298.0911.



2r

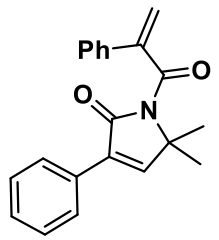
1-Methacryloyl-5,5-dimethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (89.2 mg, 70%). mp. 73-75 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 7.9, 1.5 Hz, 2H), 7.46 – 7.41 (m, 3H), 7.35 (s, 1H), 5.31 (s, 1H), 5.24 (s, 1H), 2.09 (s, 3H), 1.70 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.2, 168.2, 152.7, 142.8, 132.4, 130.2, 129.1, 128.6, 127.2, 116.7, 63.0, 24.1, 19.2.

HRMS (ESI): [M+H$^+$]calcd for $C_{16}H_{18}NO_2^+$,256.1332; found, 256.1351.



2s

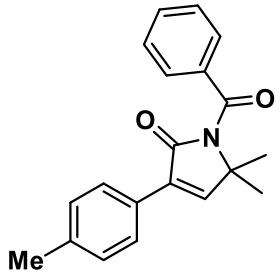
5,5-Dimethyl-3-phenyl-1-(2-phenylacryloyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **green oil (107.8 mg, 68%).**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.75 – 7.72 (m, 2H), 7.48 (dd, *J* = 5.2, 3.0 Hz, 3H), 7.39 – 7.36 (m, 5H), 7.33 (s, 1H), 5.78 (s, 1H), 5.47 (s, 1H), 1.80 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ =169.2, 167.4, 152.7, 146.9, 136.0, 132.3, 131.7, 130.0, 129.2, 129.0, 128.8, 128.6, 128.5, 128.5, 128.3, 128.2, 127.4, 127.3, 127.2, 127.1, 125.9, 114.9, 63.0, 24.0.

HRMS (ESI): [M+H$^+$]calcd for $C_{21}H_{21}NO_2^+$,318.1489; found, 318.1502.



2aa

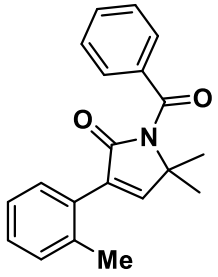
1-Benzoyl-5,5-dimethyl-3-(p-tolyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (132.7 mg, 87%). mp. 146-148 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.77 (d, *J* = 8.2 Hz, 2H), 7.63 – 7.54 (m, 3H), 7.48 (t, *J* = 7.4 Hz, 2H), 7.37 (s, 1H), 7.25 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H), 1.82 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.6, 151.7, 139.1, 136.4, 132.2, 131.2, 129.2, 128.0, 127.9, 127.4, 127.1, 63.5, 24.6, 21.4.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{20}NO_2^+$,306.1489; found, 306.1507.



2ab

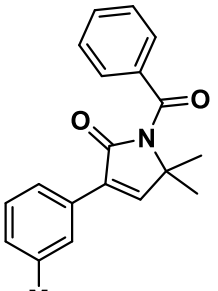
1-Benzoyl-5,5-dimethyl-3-(*o*-tolyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (106.7 mg, 70%). mp. 143-145 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.60 – 7.51 (m, 3H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.36 – 7.24 (m, 4H), 7.20 (s, 1H), 2.37 (s, 3H), 1.85 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.5, 155.7, 136.5, 136.1, 134.5, 131.2, 130.4, 130.0, 129.7, 128.8, 128.0, 127.9, 125.7, 64.1, 24.7, 20.4.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{20}NO_2^+$,306.1489; found, 306.1506.



2ac

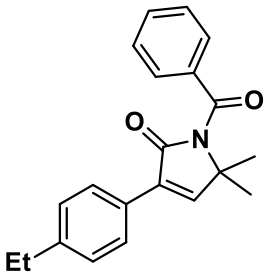
1-Benzoyl-5,5-dimethyl-3-(*m*-tolyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (122.0 mg, 80%). mp. 81-83 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.73 (s, 1H), 7.68 – 7.55 (m, 4H), 7.50 (t, *J* = 7.4 Hz, 2H), 7.42 (s, 1H), 7.37 – 7.32 (m, 1H), 7.25 (d, *J* = 7.6 Hz, 1H), 2.43 (s, 3H), 1.84 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.5, 152.6, 138.2, 136.4, 132.5, 131.2, 130.2, 129.9, 128.5, 128.0, 127.9, 127.8, 124.4, 63.5, 24.6, 21.5.

HRMS (ESI): [M+Na$^+$]calcd for $C_{20}H_{20}NO_2^+$,306.1489; found, 306.1507.



2ad

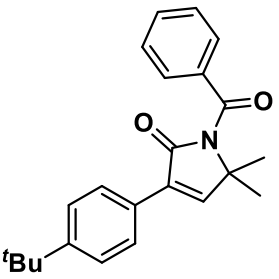
1-Benzoyl-3-(4-ethylphenyl)-5,5-dimethyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (129.2 mg, 81%). mp. 125-127 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78 (d, *J* = 8.2 Hz, 2H), 7.62 – 7.53 (m, 3H), 7.48 (t, *J* = 7.4 Hz, 2H), 7.37 (s, 1H), 7.27 (d, *J* = 8.1 Hz, 2H), 2.71 (q, *J* = 7.6 Hz, 2H), 1.81 (s, 6H), 1.28 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.6, 151.8, 145.5, 136.3, 132.3, 131.2, 128.1, 128.0, 127.9, 127.6, 127.2, 63.5, 28.7, 24.6, 15.5.

HRMS (ESI): [M+H$^+$]calcd for $C_{21}H_{22}NO_2^+$,320.1645; found, 320.1662.



2ae

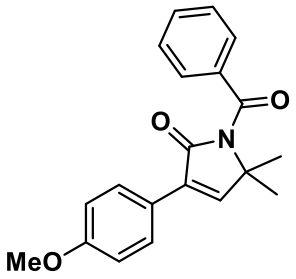
1-Benzoyl-3-(4-(*tert*-butyl)phenyl)-5,5-dimethyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (154.4 mg, 89%). mp. 127-129 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ =7.82 – 7.77 (m, 2H), 7.61 – 7.53 (m, 3H), 7.46 (dd, *J* = 11.9, 5.2 Hz, 4H), 7.37 (s, 1H), 1.81 (s, 6H), 1.37 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.6, 152.3, 151.9, 136.3, 132.3, 131.2, 128.0, 127.4, 126.9, 125.5, 63.5, 34.7, 31.2, 24.6.

HRMS (ESI): [M+H$^+$]calcd for $C_{23}H_{26}NO_2^+$,348.1958; found, 348.1978.



2af

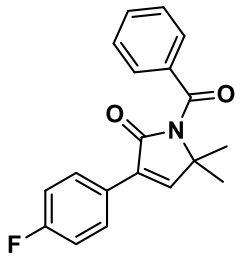
1-Benzoyl-3-(4-methoxyphenyl)-5,5-dimethyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (146.0 mg, 91%). mp. 159-161 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, *J* = 8.7 Hz, 2H), 7.61 – 7.52 (m, 3H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.30 (s, 1H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.86 (s, 3H), 1.80 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.7, 160.2, 150.6, 136.4, 131.8, 131.2, 128.5, 127.9, 127.9, 122.8, 113.9, 63.4, 55.3, 24.7.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{20}NO_3^+$,322.1438; found, 322.1456.



2ag

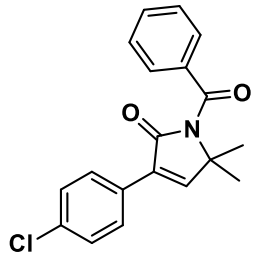
1-Benzoyl-3-(4-fluorophenyl)-5,5-dimethyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (115.9 mg, 75%). mp. 150-152 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87 – 7.82 (m, 2H), 7.60 – 7.54 (m, 3H), 7.48 (t, J*J* = 7.5 Hz, 2H), 7.37 (s, 1H), 7.11 (t, *J* = 8.7 Hz, 2H), 1.81 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.0, 168.3, 163.2 (d, *J* = 249.4 Hz), 152.1, 136.2, 131.5, 131.3, 129.1 (d, *J* = 8.1 Hz), 127.9 (d, *J* = 11.7 Hz), 126.3 (d, *J* = 3.3 Hz), 115.7, 115.5, 63.6, 24.6.

HRMS (ESI): [M+H$^+$]calcd for $C_{19}H_{17}FNO_2^+$,310.1238; found, 310.1257.



2ah

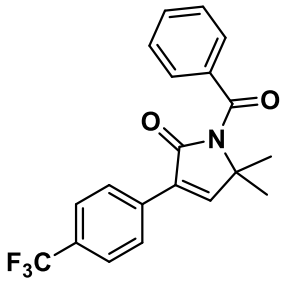
1-Benzoyl-3-(4-chlorophenyl)-5,5-dimethyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (118.6 mg, 73%). mp. 114-116 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.80 (d, *J* = 8.5 Hz, 2H), 7.60 – 7.54 (m, 3H), 7.48 (t, *J* = 7.5 Hz, 2H), 7.41 (d, *J* = 3.2 Hz, 2H), 7.38 (s, 1H), 1.81 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.0, 168.2, 152.7, 136.2, 135.1, 131.3, 128.8, 128.7, 128.5, 128.0, 127.9, 63.6, 24.5.

HRMS (ESI): [M+H$^+$]calcd for $C_{19}H_{17}ClNO_2^+$,326.0942; found, 326.0955.



2ai

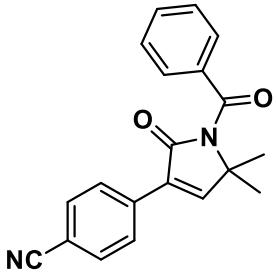
1-Benzoyl-5,5-dimethyl-3-(4-(trifluoromethyl)phenyl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (129.2 mg, 72%). mp. 84-86 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.1 Hz, 2H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.61 – 7.56 (m, 3H), 7.52 (s, 1H), 7.49 (t, *J* = 7.4 Hz, 2H), 1.84 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.0, 167.9, 154.1, 136.0, 133.7, 131.4, 131.0, 130.7, 128.0, 127.9, 127.5, 125.4 (q, *J* = 3.9 Hz), 123.9 (q, *J* = 272.2 Hz), 63.8, 24.5.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{17}F_3NO_2^+$,360.1206; found, 360.1220.



2aj

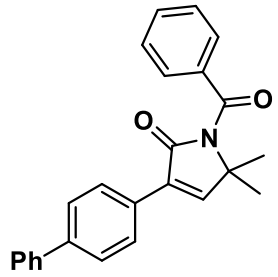
4-(1-Benzoyl-5,5-dimethyl-2-oxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzonitrile

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 5:1) to give the product as a **white solid (123.2 mg, 78%). mp. 163-165 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95 – 7.92 (m, 2H), 7.67 – 7.64 (m, 2H), 7.55 – 7.51 (m, 3H), 7.50 (s, 1H), 7.46 – 7.41 (m, 2H), 1.79 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.9, 167.6, 154.8, 135.9, 134.6, 132.3, 131.5, 131.0, 128.0, 127.9, 127.8, 118.5, 112.6, 64.0, 24.4.

HRMS (ESI): [M+H$^+$]calcd for $C_{20}H_{17}N_2O_2^+$,317.1285; found, 317.1295.



2ak

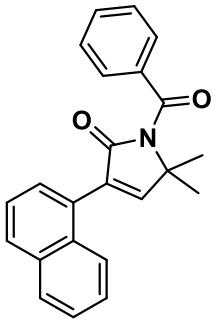
3-([1,1'-Biphenyl]-4-yl)-1-benzoyl-5,5-dimethyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (156.0 mg, 85%). mp. 157-159 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97 (d, *J* = 8.4 Hz, 2H), 7.68 (dd, *J* = 7.7, 5.1 Hz, 4H), 7.60 (ddd, *J* = 7.4, 6.9, 4.1 Hz, 3H), 7.54 – 7.49 (m, 4H), 7.46 (s, 1H), 7.43 (t, *J* = 7.3 Hz, 1H), 1.85 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.5, 152.4, 141.8, 140.4, 136.3, 132.0, 131.3, 129.2, 128.9, 128.0, 127.9, 127.7, 127.2, 127.1, 63.6, 24.6.

HRMS (ESI): [M+H$^+$]calcd for $C_{25}H_{22}NO_2^+$,368.1645; found, 368.1657.



2al

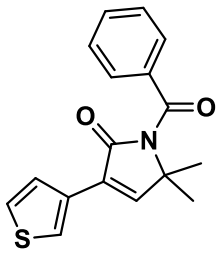
1-Benzoyl-5,5-dimethyl-3-(naphthalen-1-yl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (146.6 mg, 86%). mp. 149-151 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98 – 7.90 (m, 3H), 7.66 – 7.50 (m, 7H), 7.48 – 7.42 (m, 2H), 7.42 (s, 1H), 1.94 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.1, 168.8, 156.8, 136.1, 133.8, 133.1, 131.4, 131.2, 129.3, 128.7, 128.0, 127.9, 127.7, 126.6, 126.0, 125.1, 124.6, 64.4, 24.8.

HRMS (ESI): [M+H$^+$]calcd for $C_{23}H_{20}NO_2^+$,342.1489; found, 342.1507.



2am

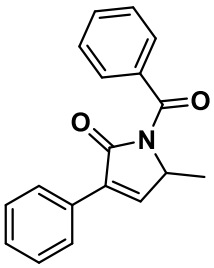
1-Benzoyl-5,5-dimethyl-3-(thiophen-3-yl)-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **white solid (118.8 mg, 80%). mp. 108-110 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.18 (dd, *J* = 2.9, 1.1 Hz, 1H), 7.61 – 7.56 (m, 3H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.44 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.41 – 7.37 (m, 1H), 7.29 (s, 1H), 1.81 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.0, 168.4, 150.1, 136.3, 131.3, 130.7, 128.0, 127.9, 125.9, 125.7, 125.0, 63.9, 24.7.

HRMS (ESI): [M+H$^+$]calcd for $C_{17}H_{16}NO_2S^+$,298.0896; found, 298.0910.



2ap

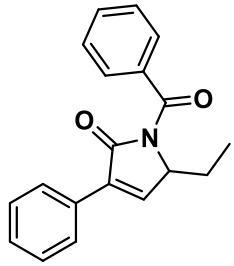
1-Benzoyl-5-methyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **black solid (55.4 mg, 40%). mp. 114-116 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83 (dd, *J* = 7.4, 1.8 Hz, 2H), 7.73 (d, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.42 (m, 4H), 7.41 (s, 1H), 5.16 (qd, *J* = 6.7, 2.0 Hz, 1H), 1.62 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.6, 168.2, 146.0, 134.8, 132.0, 130.3, 129.1, 128.9, 128.5, 127.9, 127.2, 55.0, 17.7.

HRMS (ESI): [M+H$^+$]calcd for $C_{18}H_{16}NO_2^+$,278.1176; found, 278.1192.



2aq

1-Benzoyl-5-ethyl-3-phenyl-1,5-dihydro-2*H*-pyrrol-2-one

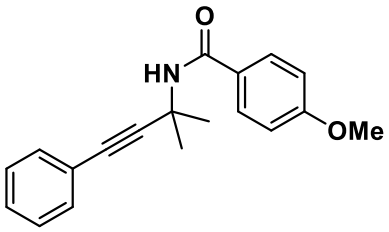
According to general procedure for reaction, the crude residue was purified by flash chromatography (petroleum ether / acetone = 10:1) to give the product as a **black solid (61.1 mg, 42%). mp. 118-120 °C**

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 – 7.82 (m, 2H), 7.77 – 7.73 (m, 2H), 7.63 – 7.58 (m, 1H), 7.54 – 7.47 (m, 4H), 7.43 (s, 1H), 7.41 (d, *J* = 1.4 Hz, 1H), 5.17 (ddd, *J* = 7.2, 3.4, 2.4 Hz, 1H), 1.30 (s, 2H), 1.05 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.7, 168.6, 144.5, 135.6, 134.7, 132.1, 130.4, 129.1, 128.5, 127.9, 127.2, 59.6, 23.9, 8.3.

HRMS (ESI): [M+H$^+$]calcd for $C_{19}H_{18}NO_2^+$,292.1332; found, 292.1343.

7. Characterization of Intermediates 2g'



2g'

4-Methoxy-*N*-(2-methyl-4-phenylbut-3-yn-2-yl)benzamide

This compound was obtained as a white solid. mp. 175-177 ºC.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78 (d, *J* = 8.7 Hz, 1H), 7.50 – 7.46 (m, 1H), 7.31 (dd, *J* = 6.0, 2.6 Hz, 1H), 6.92 (d, *J* = 8.7 Hz, 1H), 6.41 (s, 1H), 3.85 (s, 2H), 1.89 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 166.0, 162.1, 131.8, 128.7, 128.2, 127.4, 122.9, 113.6, 93.0, 81.3, 55.4, 48.9, 29.1.

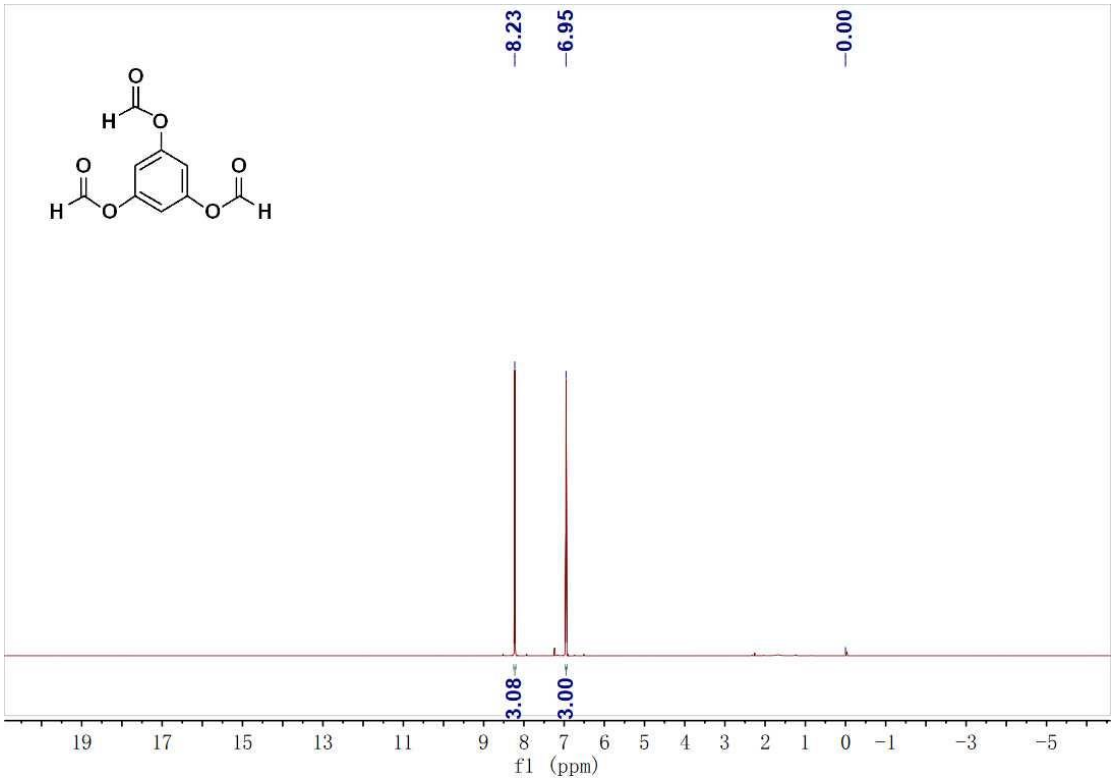
8. References

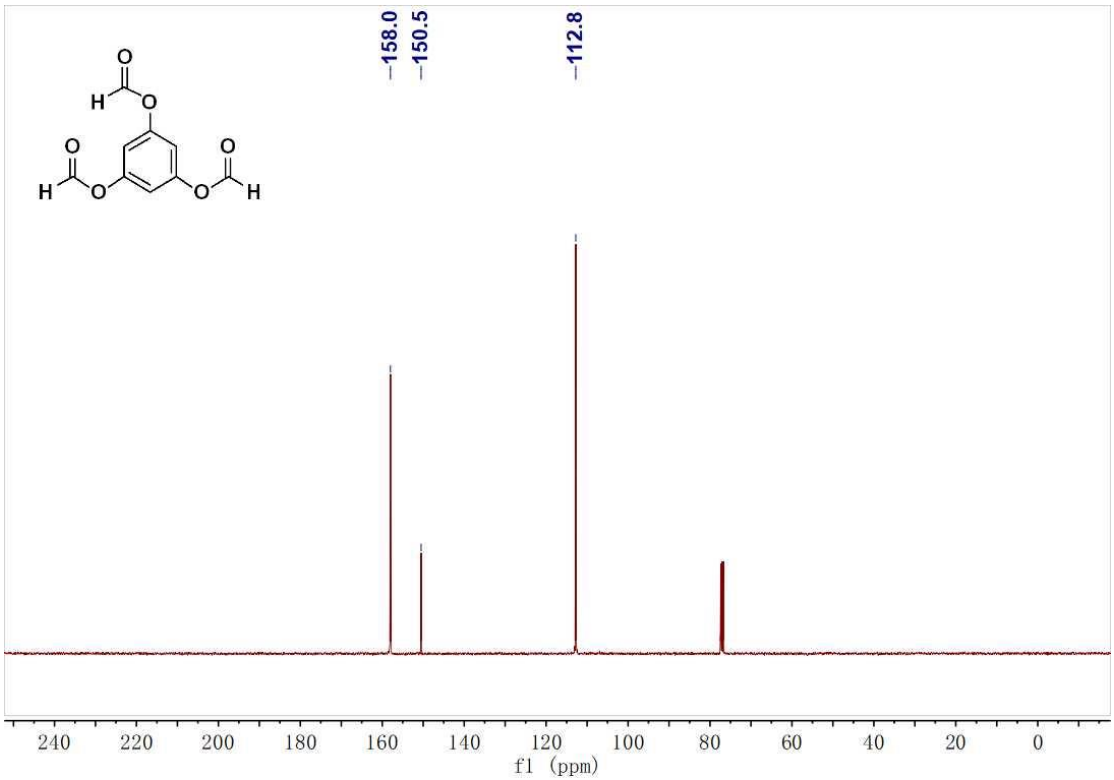
1 J. Ying, Z. Le and X. -F. Wu, *Org. Lett.*, 2020, **22**, 194-198.

2 A. Kolleth, S. Christoph, S. Arseniyadis and J. Cossy, *Chem. Commun.,* 2012, **48**, 10511-10513.

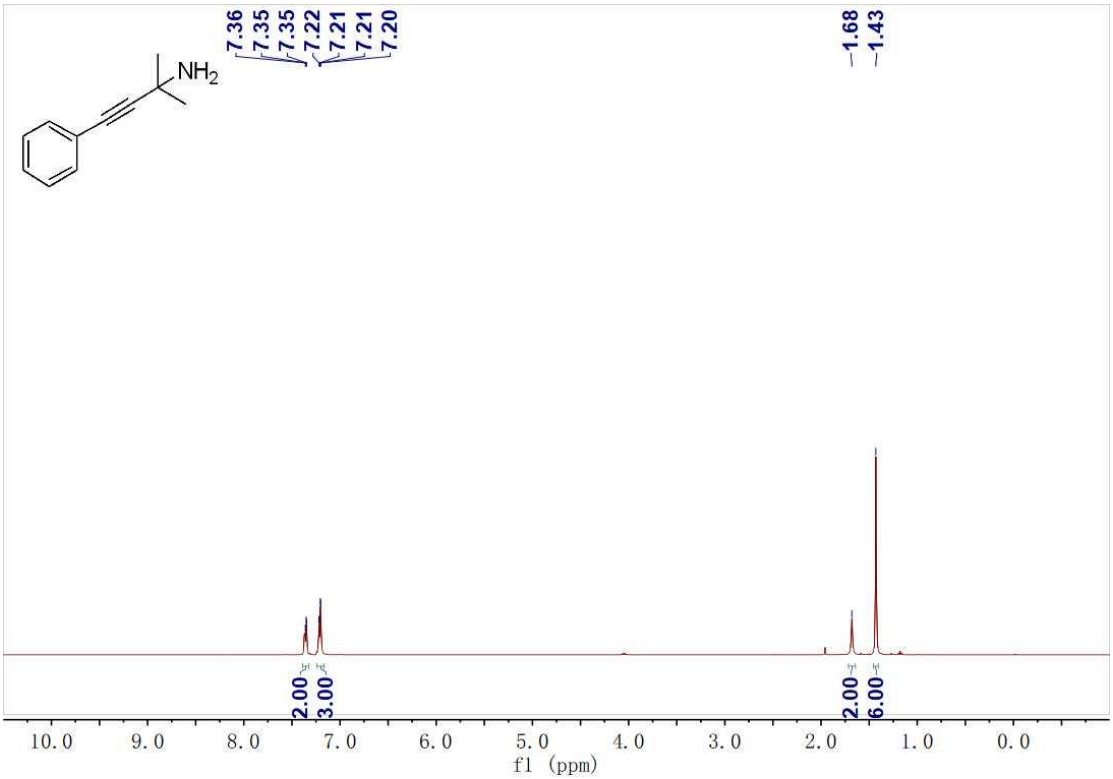
3 (a) L. Jiang, X. Qi and X.-F. Wu, *Tetrahedron*, 2016, **57**, 3368-3370; (b) L. Jiang, R. Li, C. Zhou, X. Qi, J.-B. Peng and X.-F. Wu, *Mol. Catal.*, 2017, **433**, 8-11.

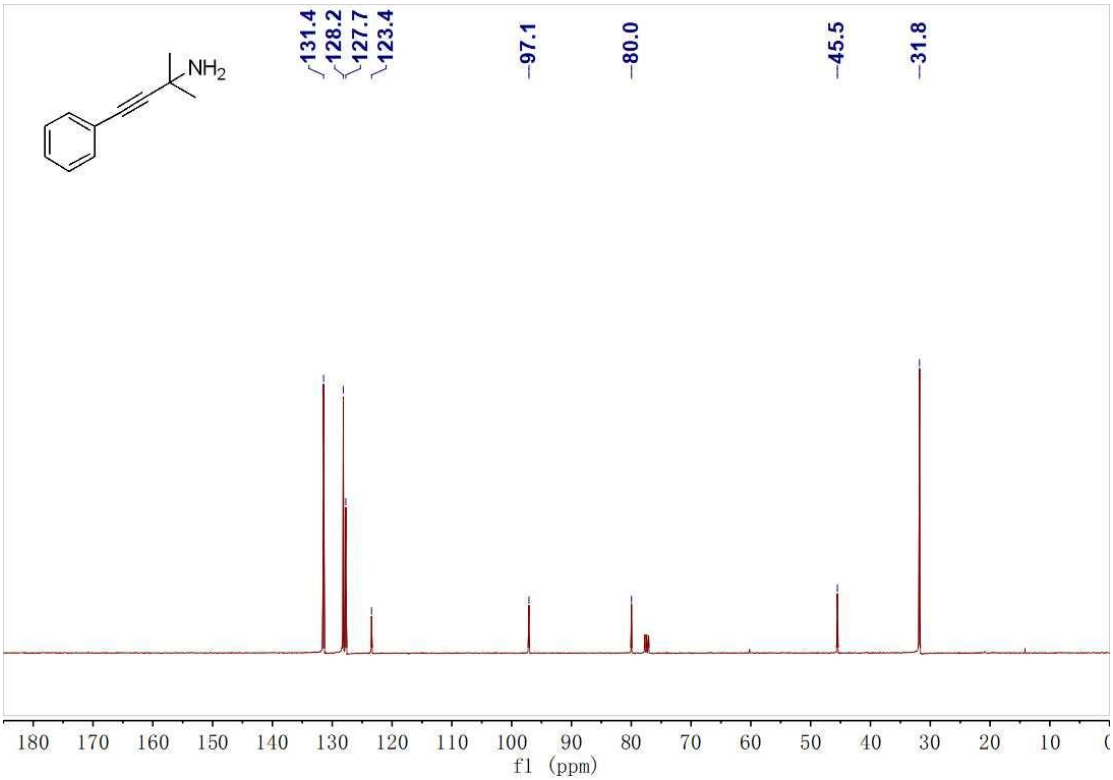
9. Spectra of TFBen and Substrates



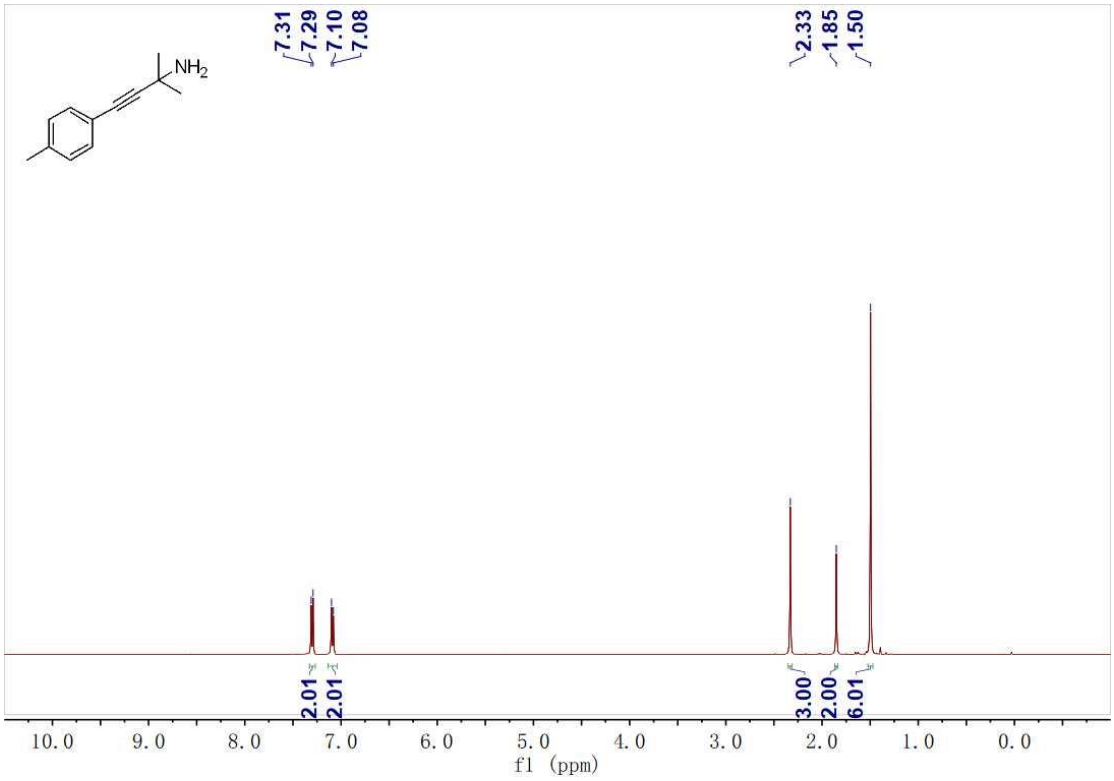


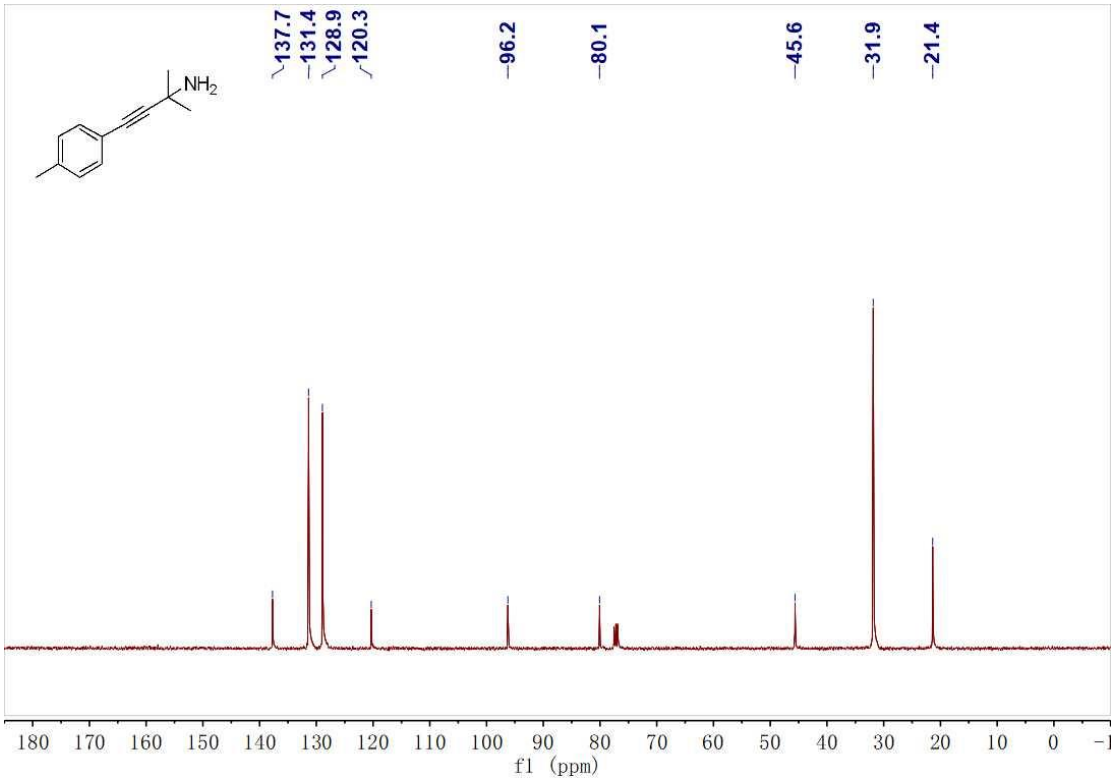
1a


7.36
7.35
7.35
7.22
7.21
7.21
7.20
1.68
1.43
NH2
2.00
3.00
2.00
6.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

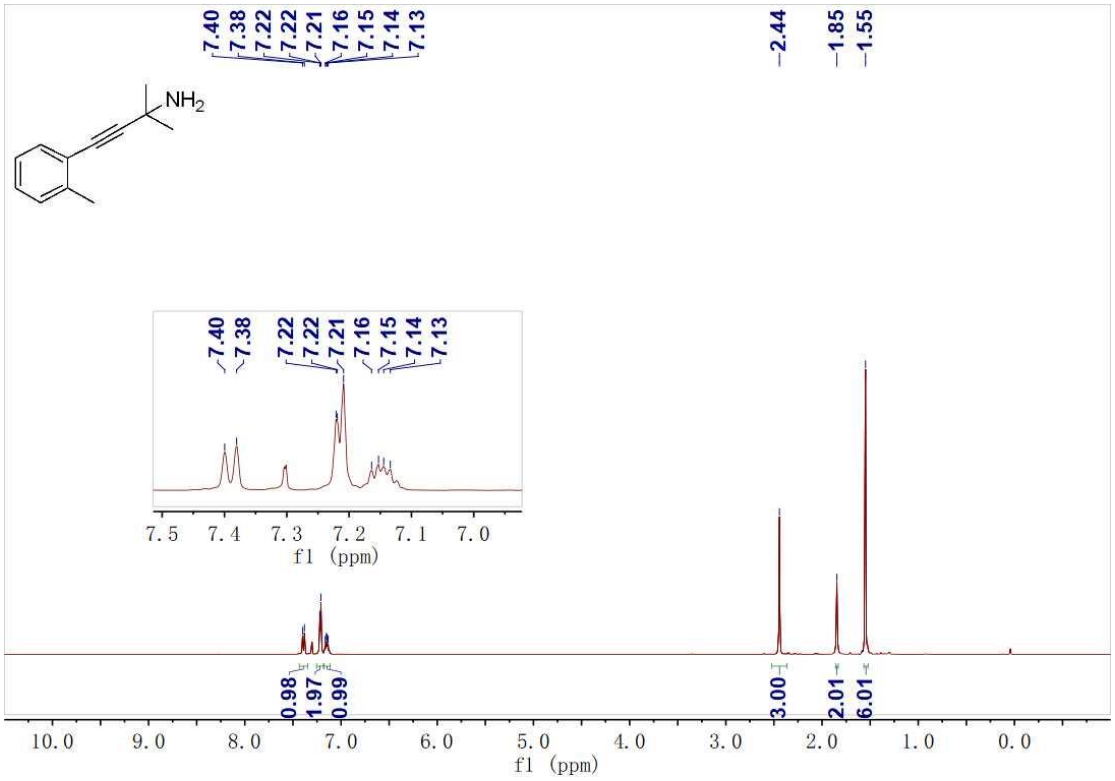

131.4
128.2
127.7
123.4
97.1
80.0
45.5
31.8
NH2
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

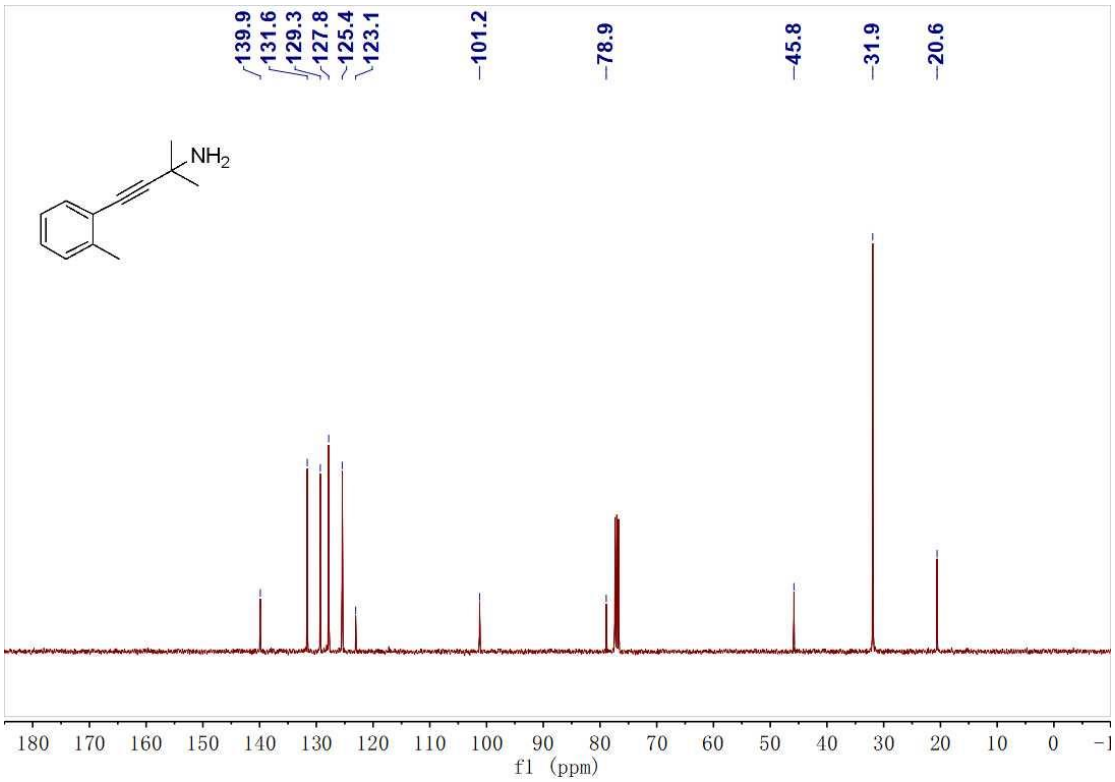
1aa


NH2
7.31
7.29
7.10
7.08
2.33
1.85
1.50
2.01
2.01
3.00
2.00
6.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

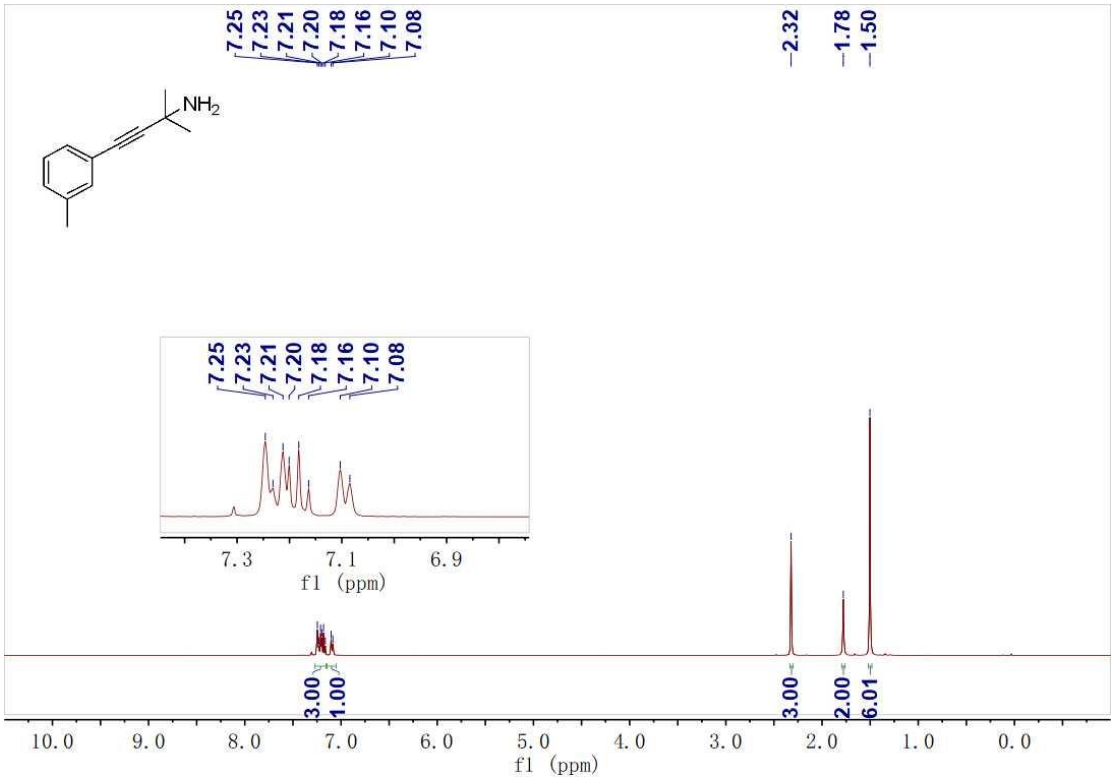

NH2
137.7
131.4
128.9
120.3
96.2
80.1
45.6
31.9
21.4
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

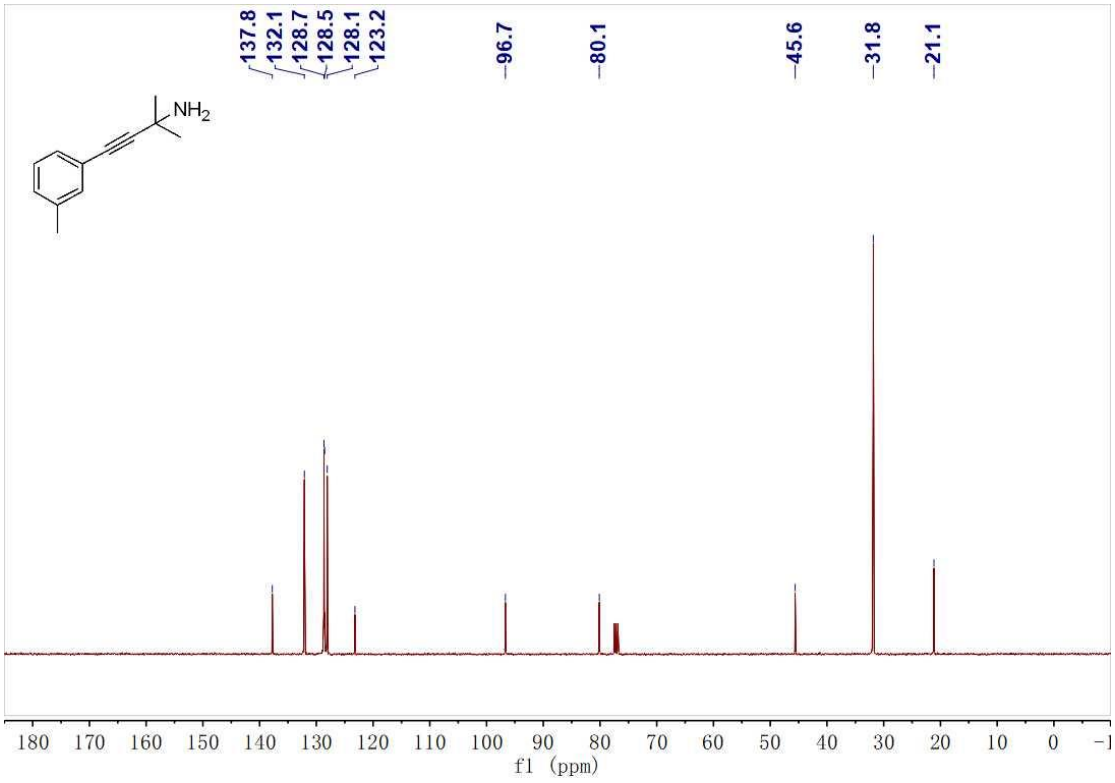
1ab


7.40
7.38
7.22
7.22
7.21
7.16
7.15
7.14
7.13
2.44
1.85
1.55
NH2
0.98
1.97
0.99
3.00
2.01
6.01
f1 (ppm)

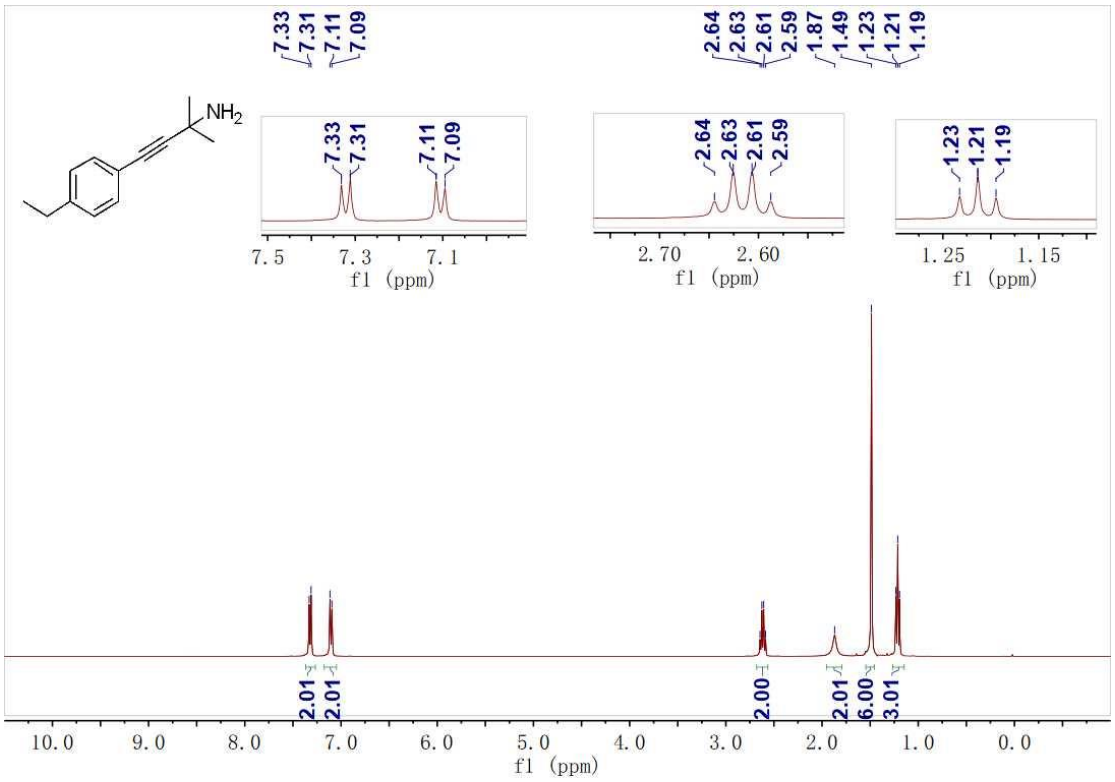

139.9
131.6
129.3
127.8
125.4
123.1
101.2
78.9
45.8
31.9
20.6
NH2
f1 (ppm)

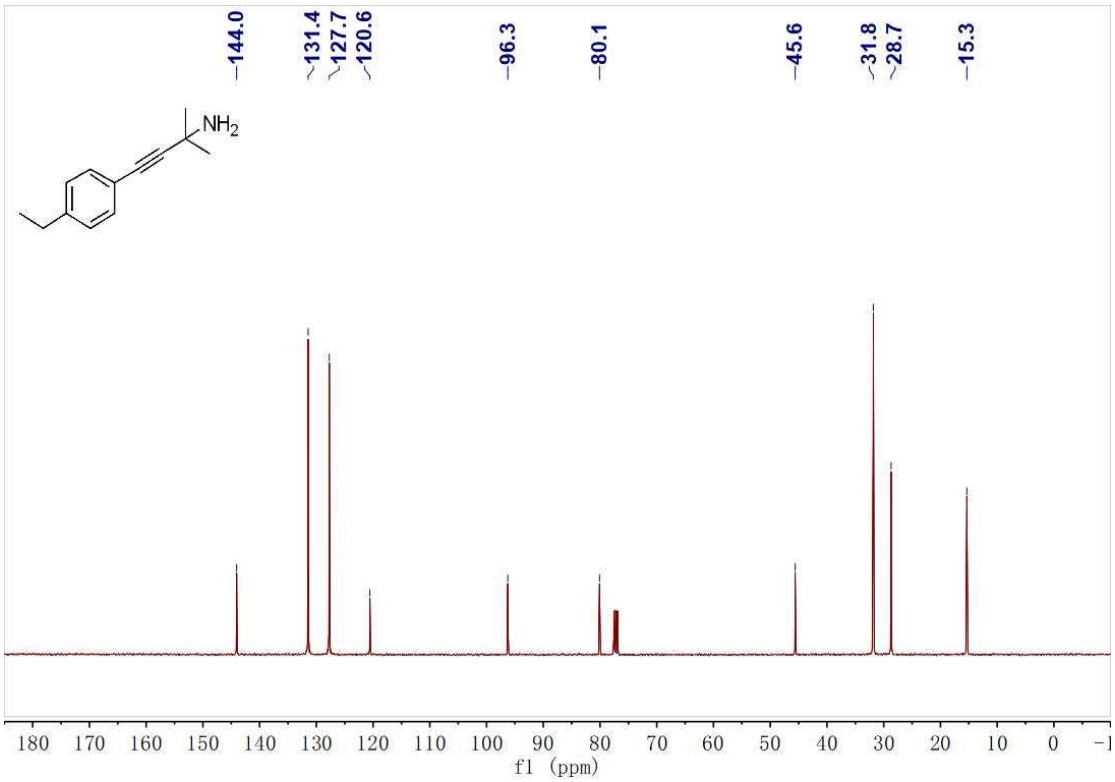
1ac


7.25
7.23
7.21
7.20
7.18
7.16
7.10
7.08
2.32
1.78
1.50
NH2
7.3
7.1
6.9
f1 (ppm)
3.00
1.00
3.00
2.00
6.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

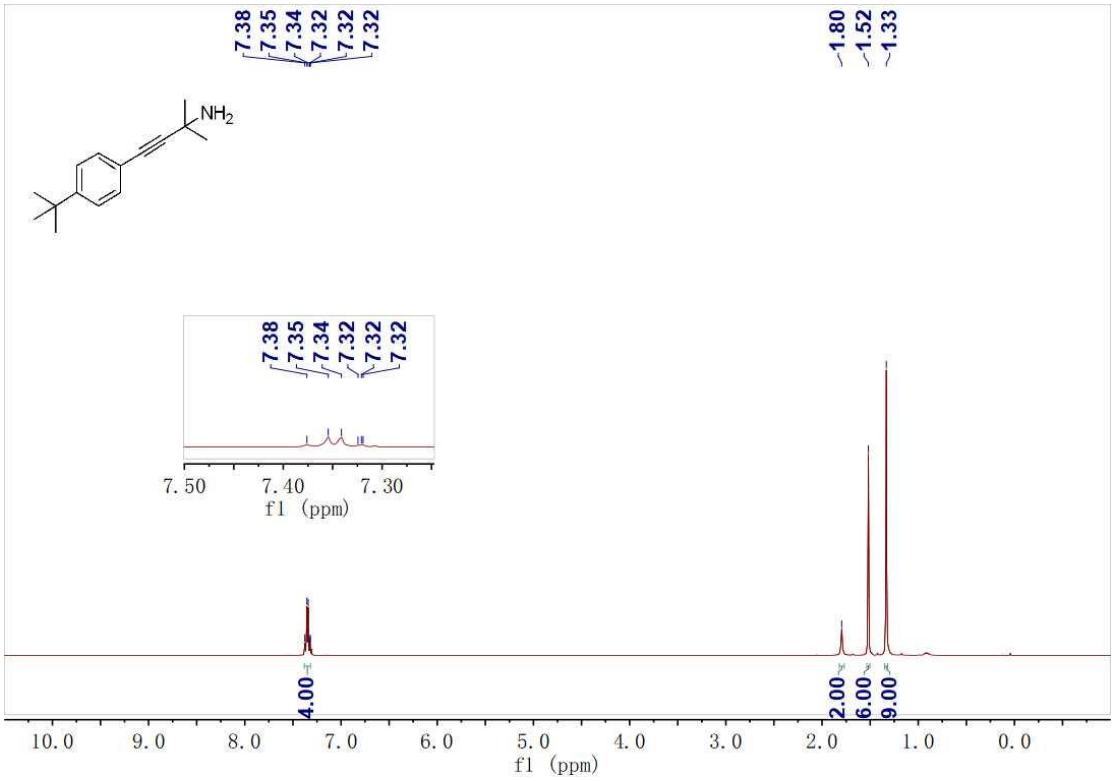

137.8
132.1
128.7
128.5
128.1
123.2
96.7
80.1
45.6
31.8
21.1
NH2
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

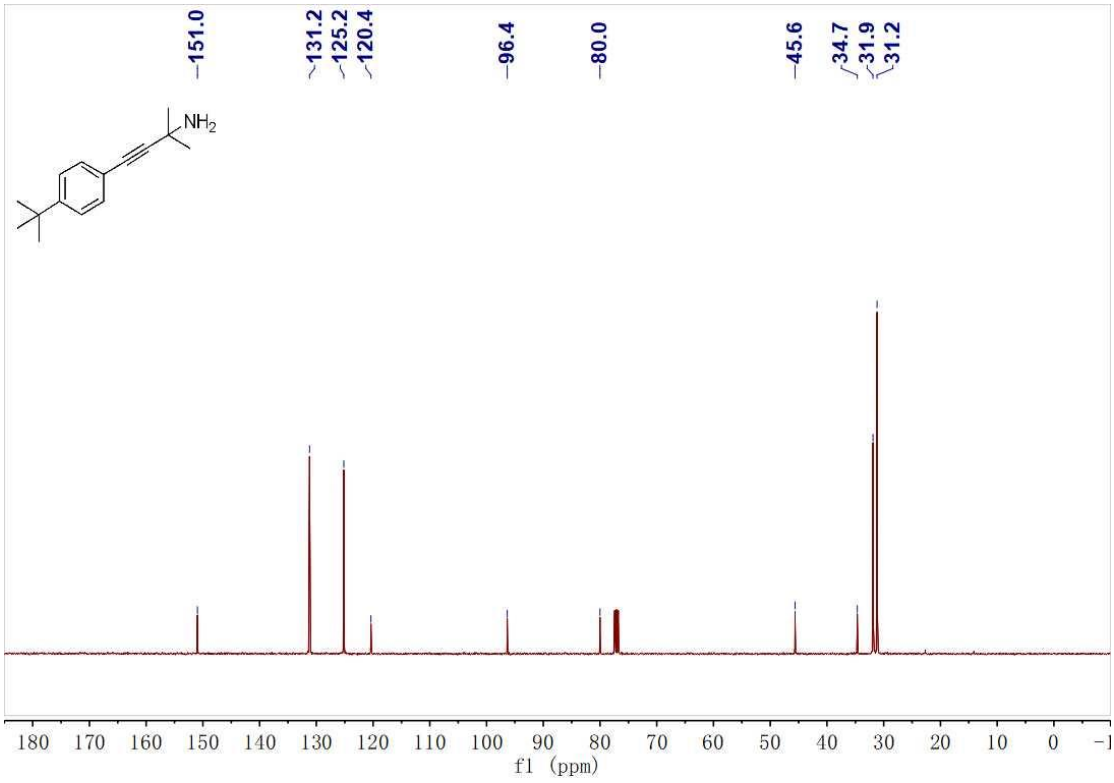
1ad


7.33
7.31
7.11
7.09
2.64
2.63
2.61
2.59
1.87
1.49
1.23
1.21
1.19
NH2
f1 (ppm)
2.01
2.01
2.00
2.01
6.00
3.01

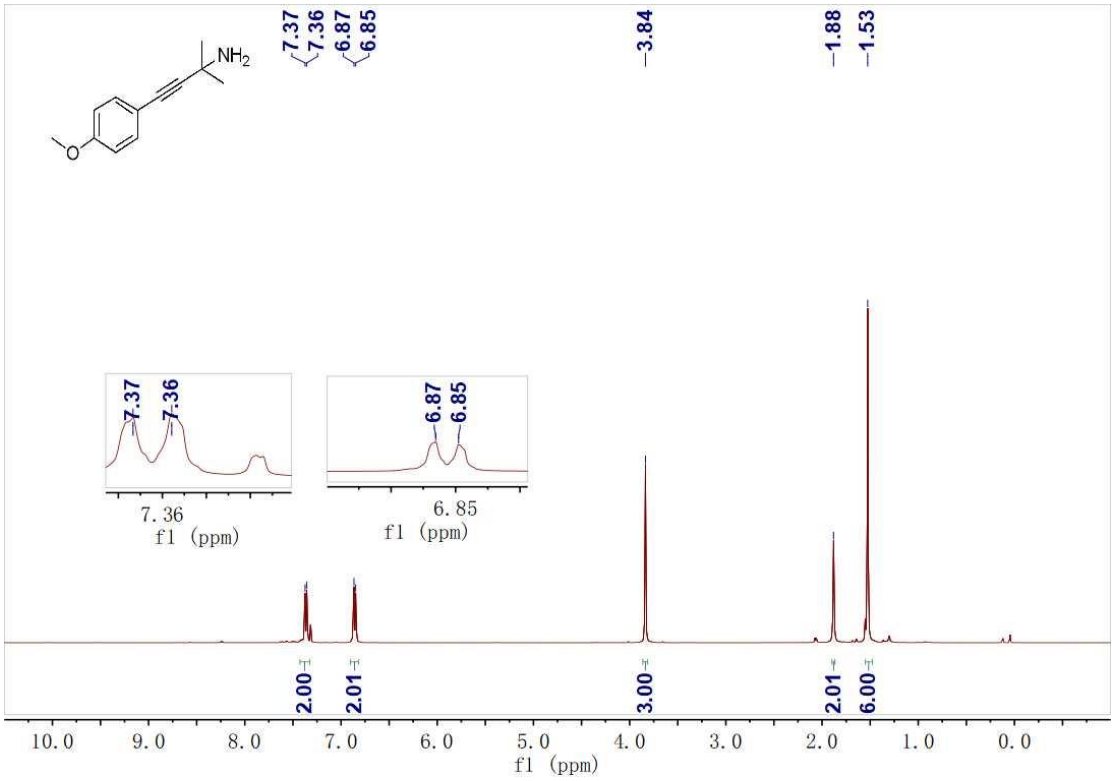

144.0
131.4
127.7
120.6
96.3
80.1
45.6
31.8
28.7
15.3
NH2
f1 (ppm)

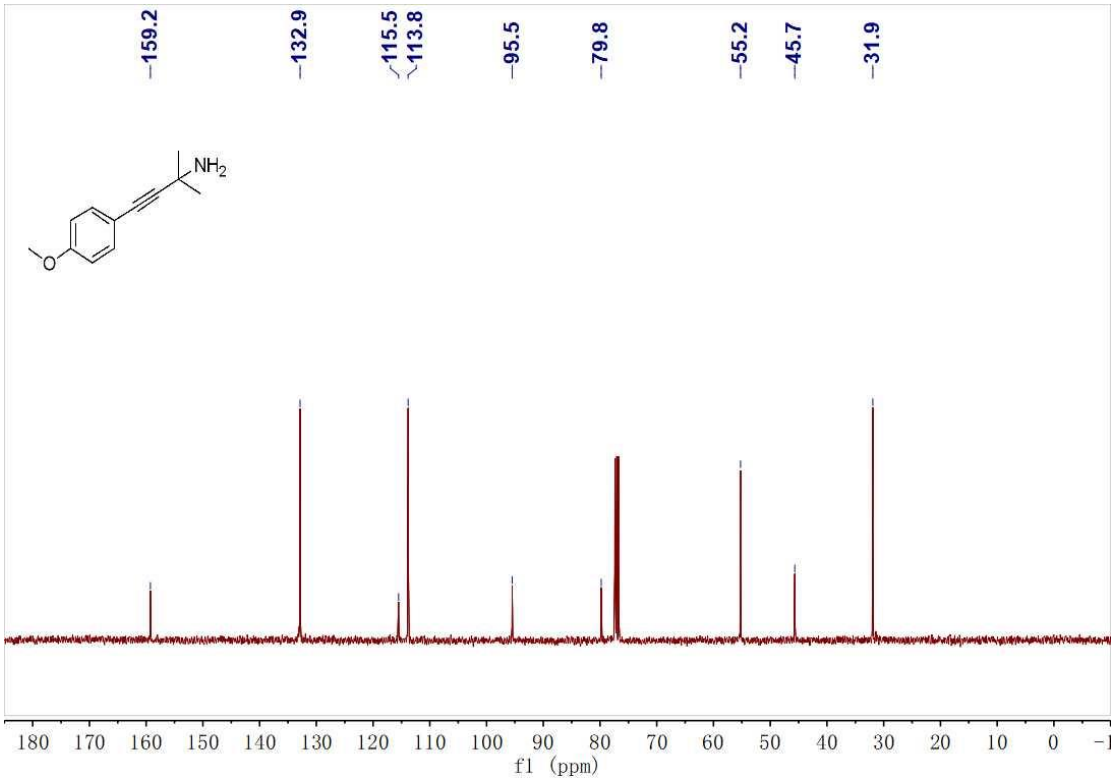
1ae


7.38
7.35
7.34
7.32
7.32
7.32
1.80
1.52
1.33
NH2
7.50
7.40
7.30
f1 (ppm)
4.00
2.00
6.00
9.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

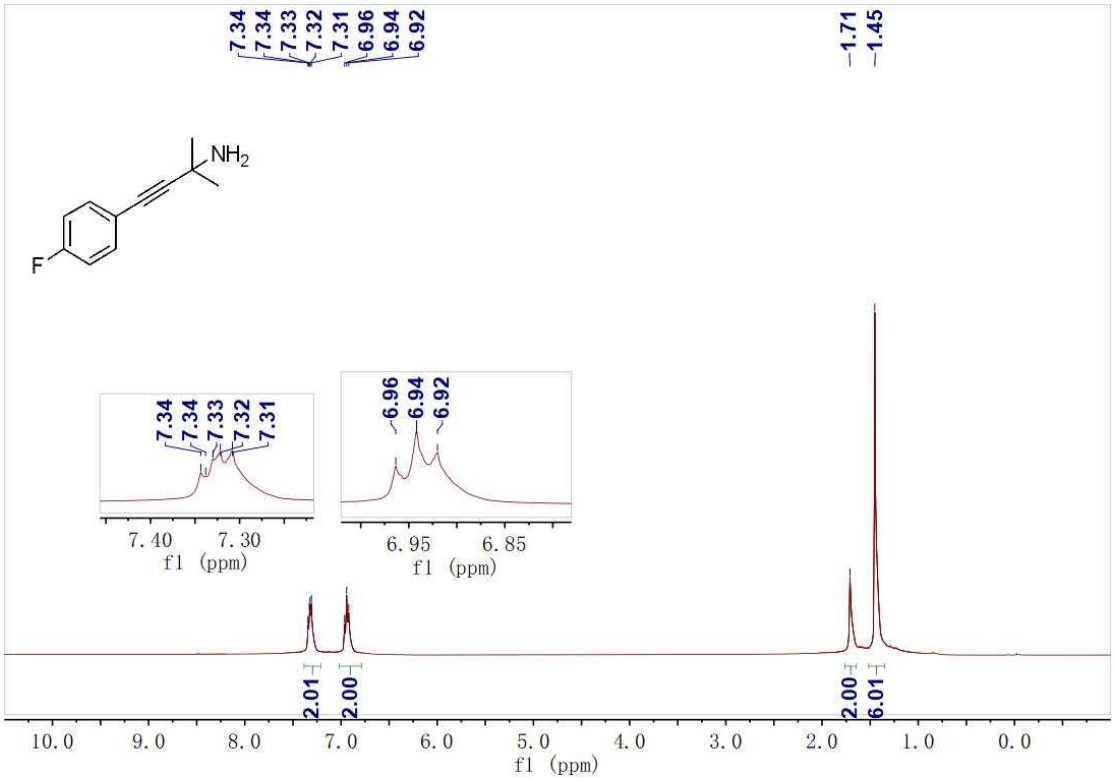

151.0
131.2
125.2
120.4
96.4
80.0
45.6
34.7
31.9
31.2
NH2
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

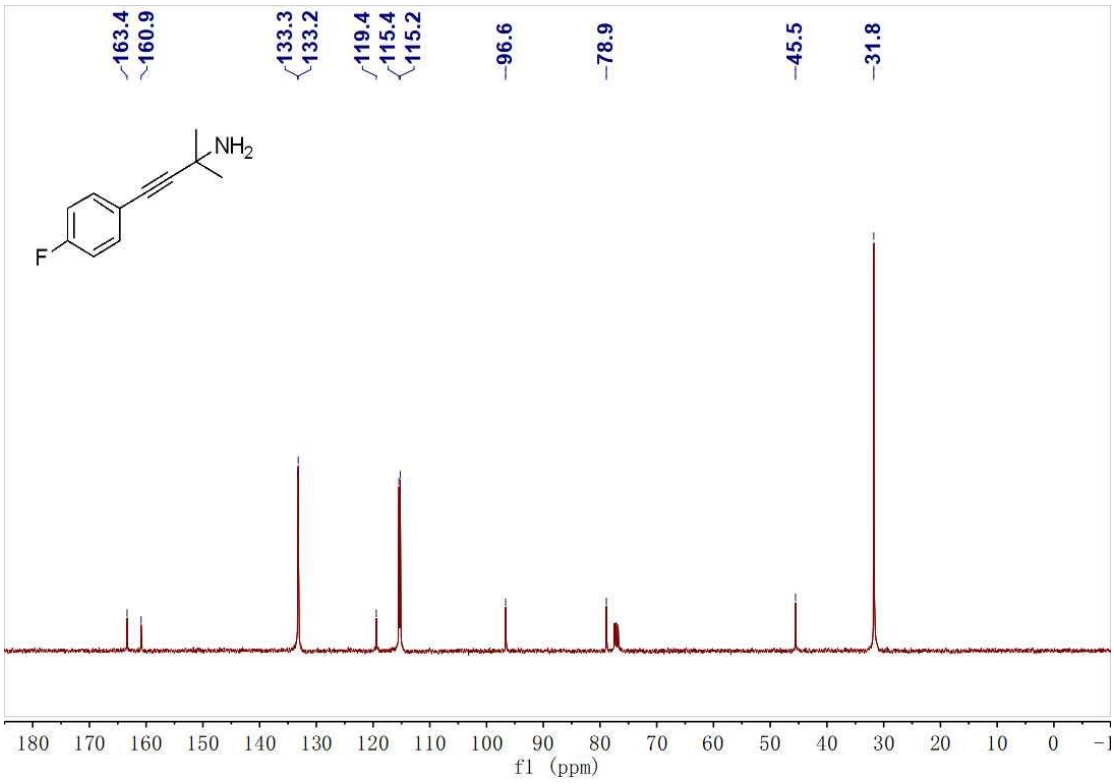
1af


7.37
7.36
6.87
6.85
3.84
1.88
1.53
NH2
O
7.36
f1 (ppm)
6.85
f1 (ppm)
2.00
2.01
3.00
2.01
6.00
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

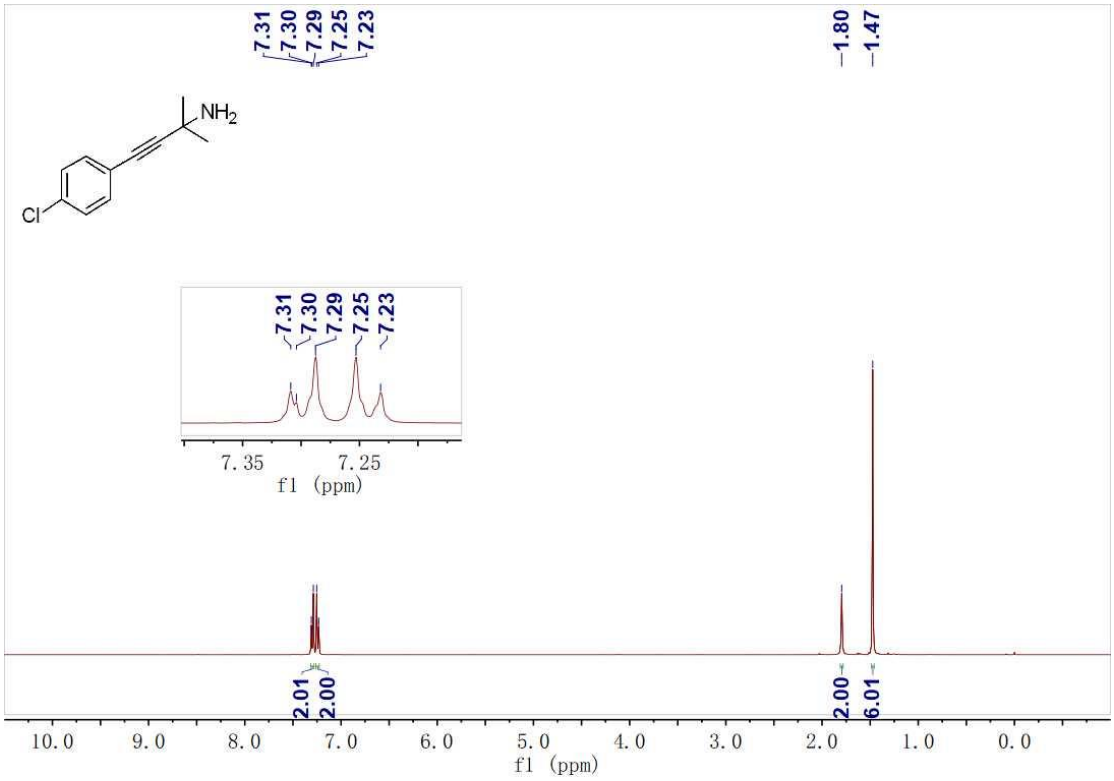

159.2
132.9
115.5
113.8
95.5
79.8
55.2
45.7
31.9
NH2
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

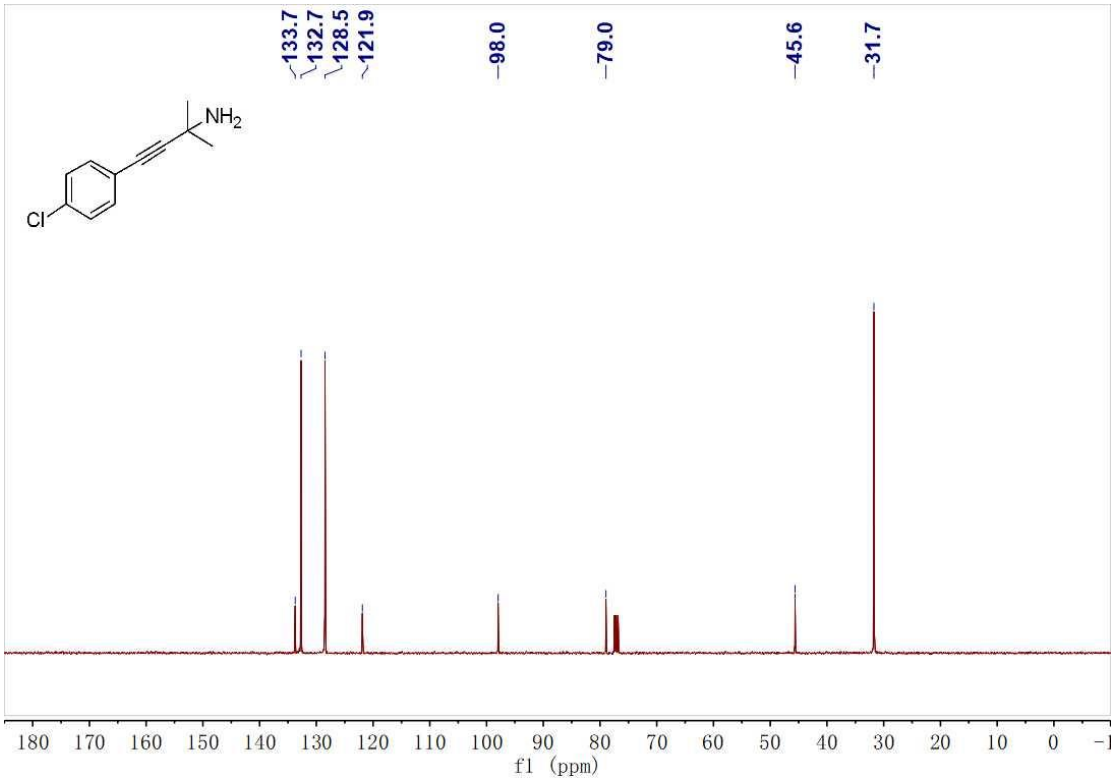
1ag


7.34
7.34
7.33
7.32
7.31
6.96
6.94
6.92
1.71
1.45
NH2
F
7.40
7.30
f1 (ppm)
6.95
6.85
f1 (ppm)
2.01
2.00
2.00
6.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

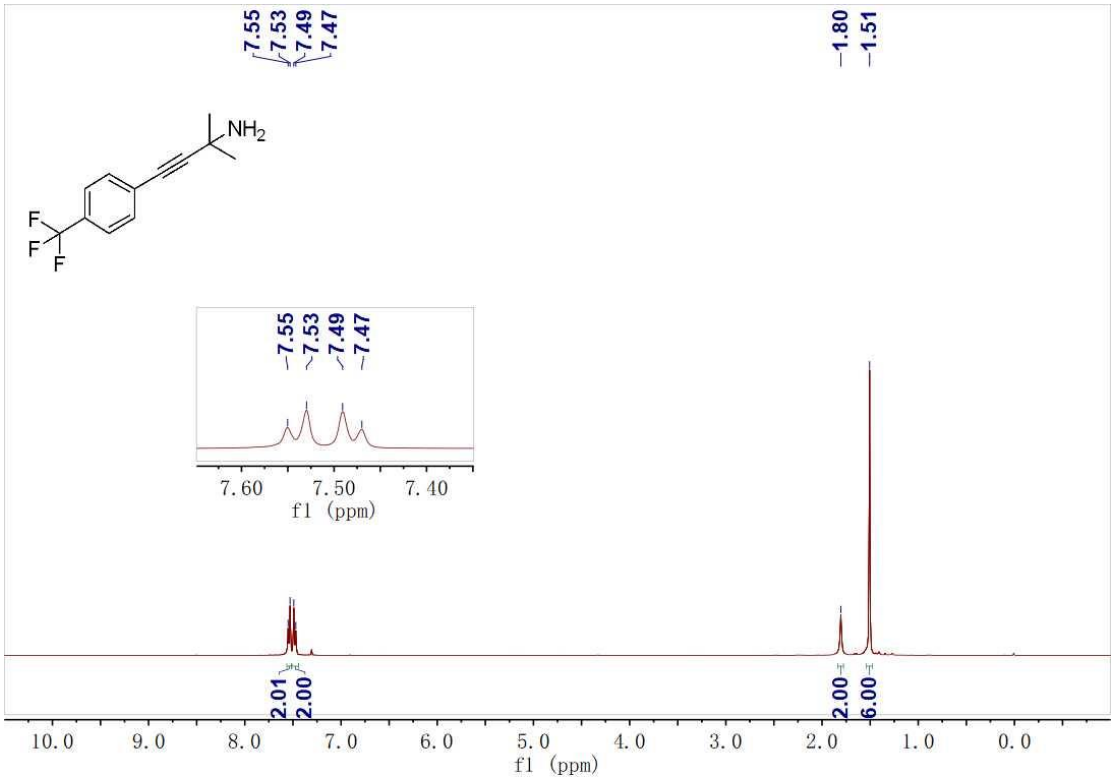

163.4
160.9
133.3
133.2
119.4
115.4
115.2
96.6
78.9
45.5
31.8
NH2
F
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

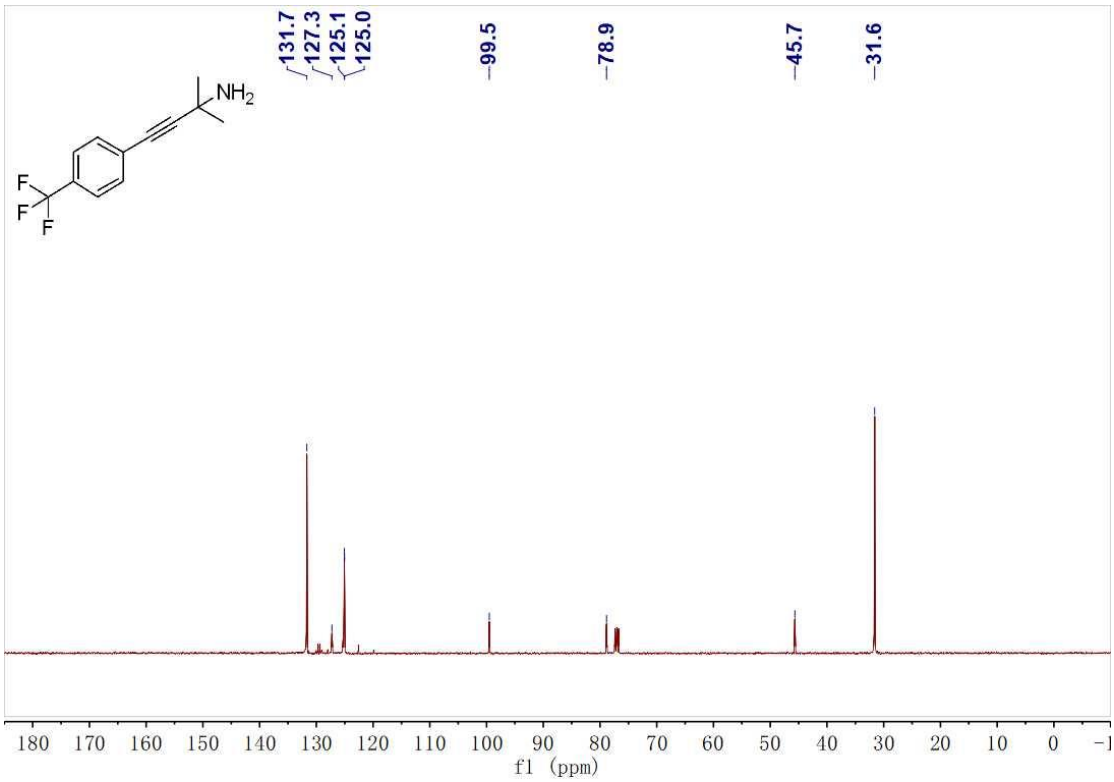
1ah


7.31
7.30
7.29
7.25
7.23
1.80
1.47
NH2
Cl
7.35
7.25
f1 (ppm)
2.01
2.00
2.00
6.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

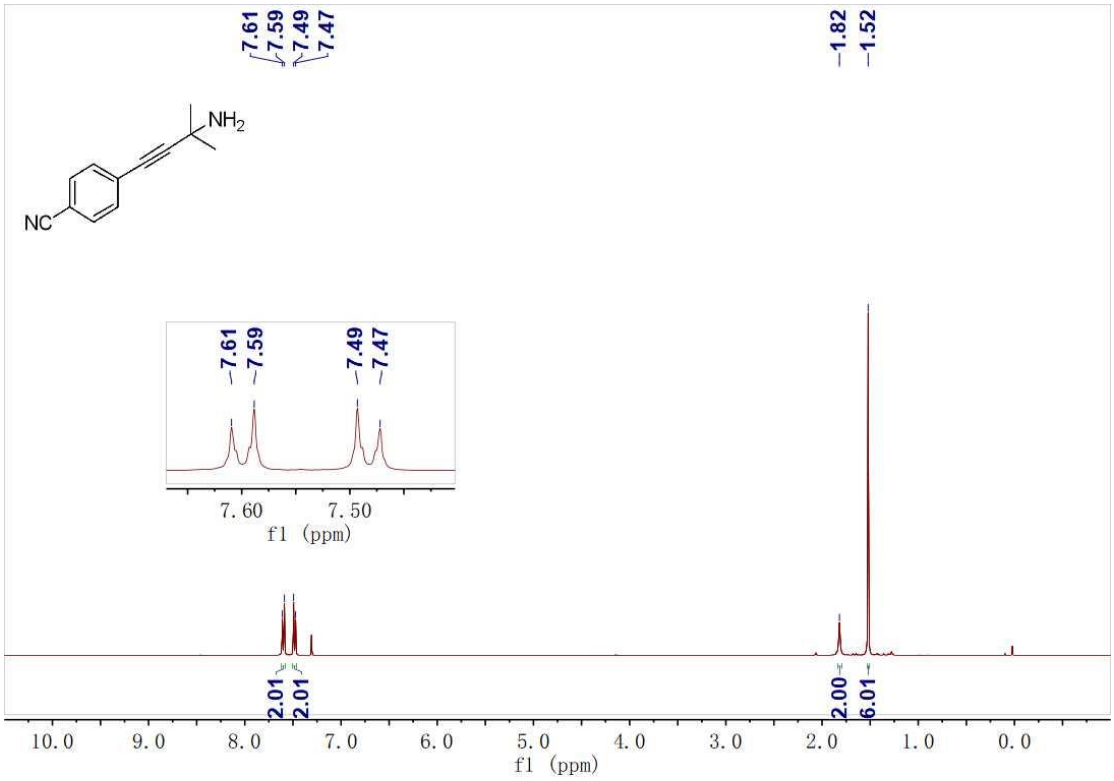

133.7
132.7
128.5
121.9
98.0
79.0
45.6
31.7
NH2
Cl
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

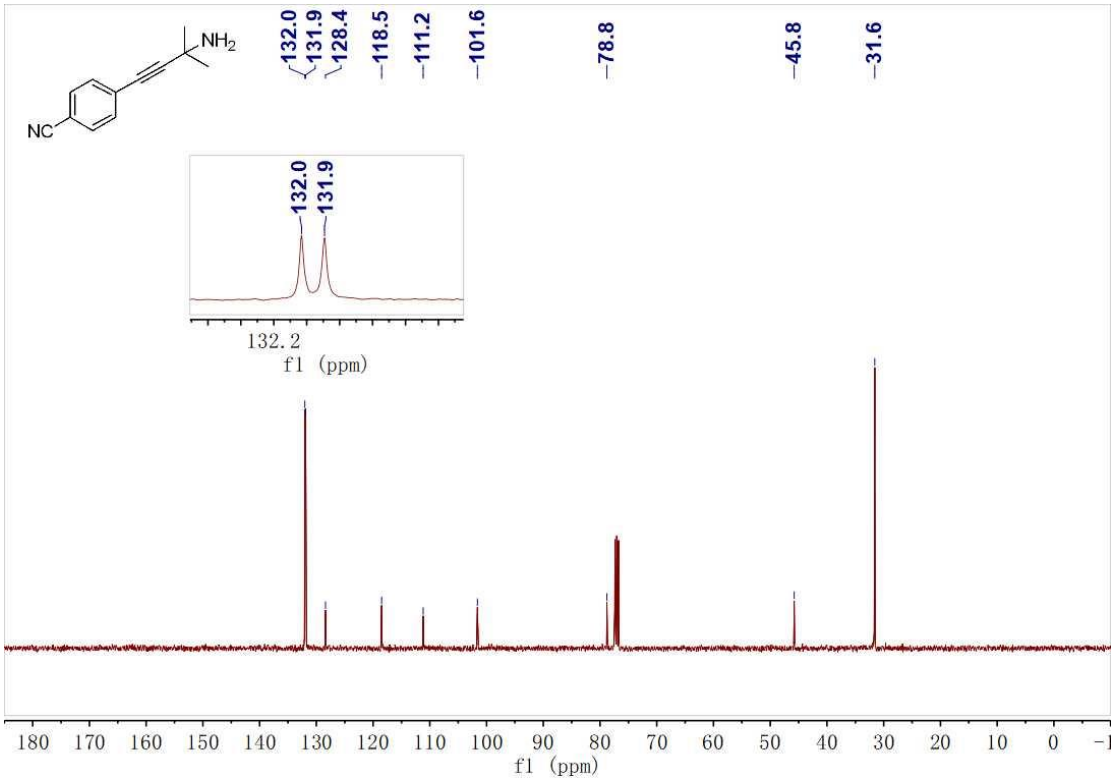
1ai


7.55
7.53
7.49
7.47
1.80
1.51
NH2
F
F
F
7.60
7.50
7.40
f1 (ppm)
2.01
2.00
2.00
6.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

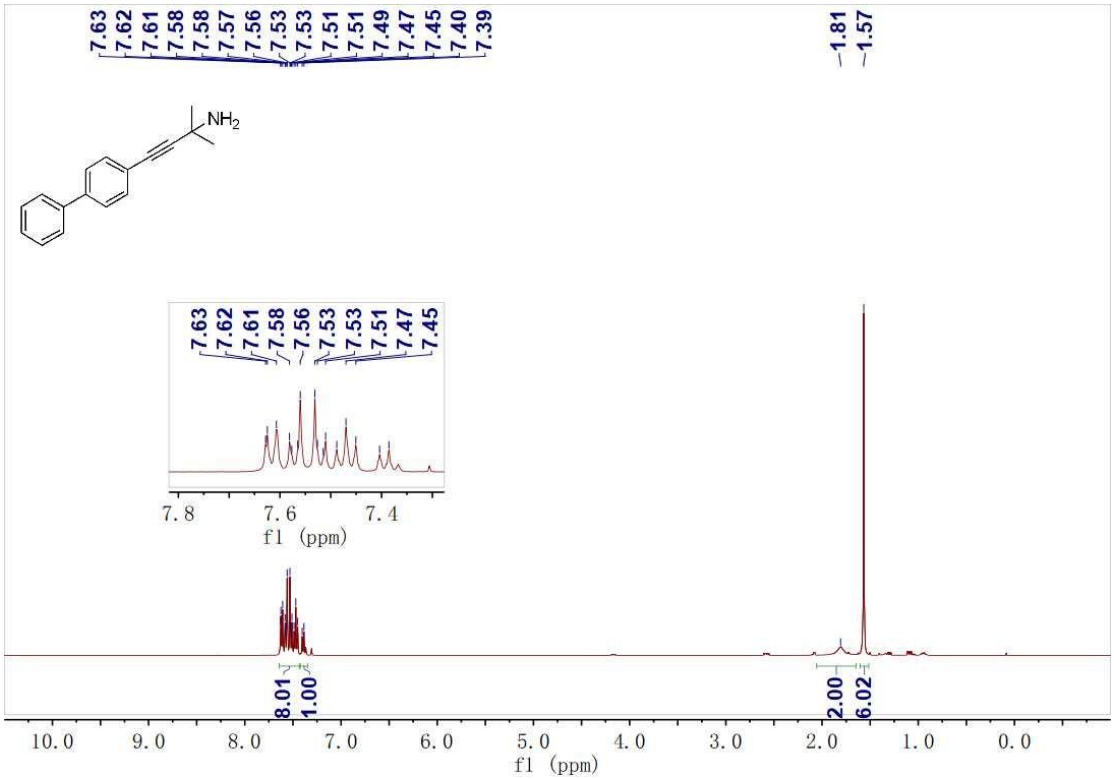

131.7
127.3
125.1
125.0
99.5
78.9
45.7
31.6
NH2
F
F
F
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

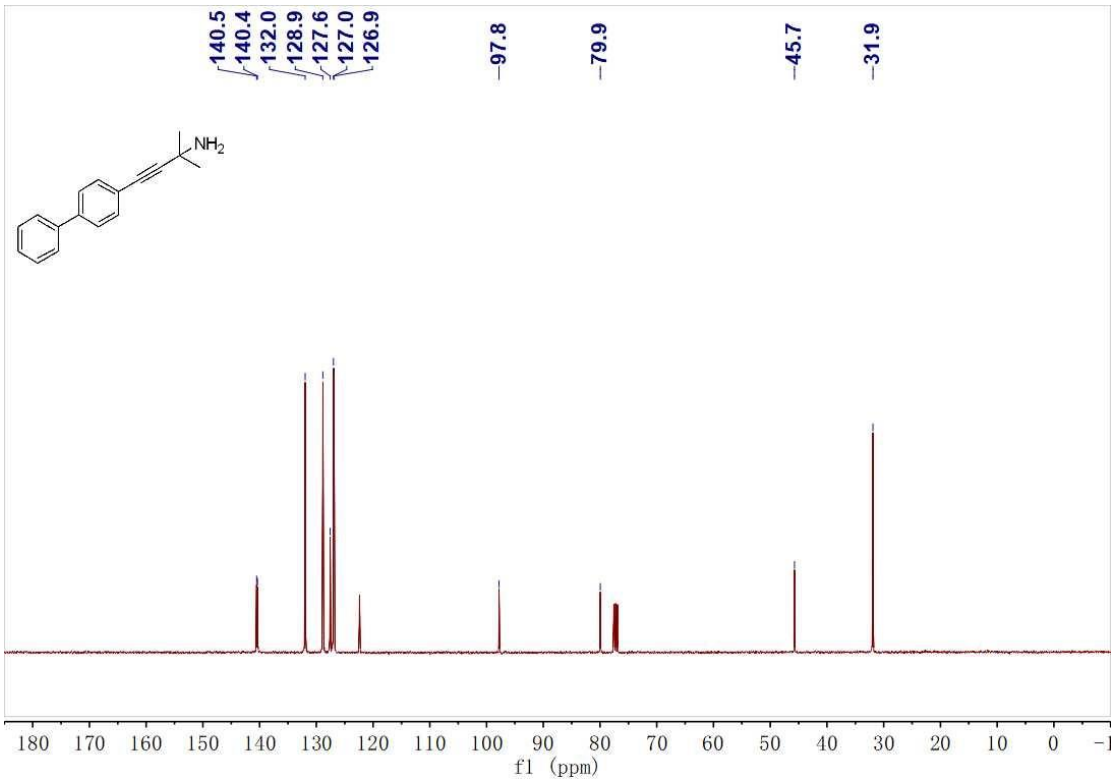
1aj


7.61
7.59
7.49
7.47
1.82
1.52
NH2
NC
7.60
7.50
f1 (ppm)
2.01
2.01
2.00
6.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

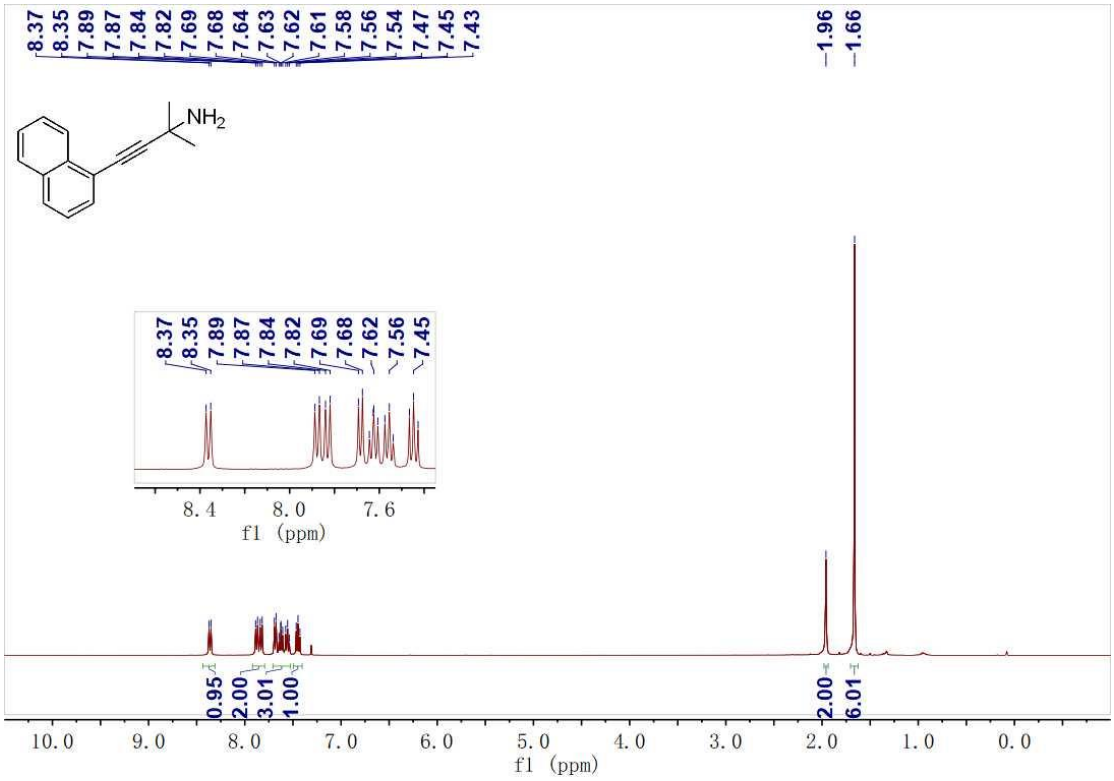

132.0
131.9
128.4
118.5
111.2
101.6
78.8
45.8
31.6
NH2
NC
132.2
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

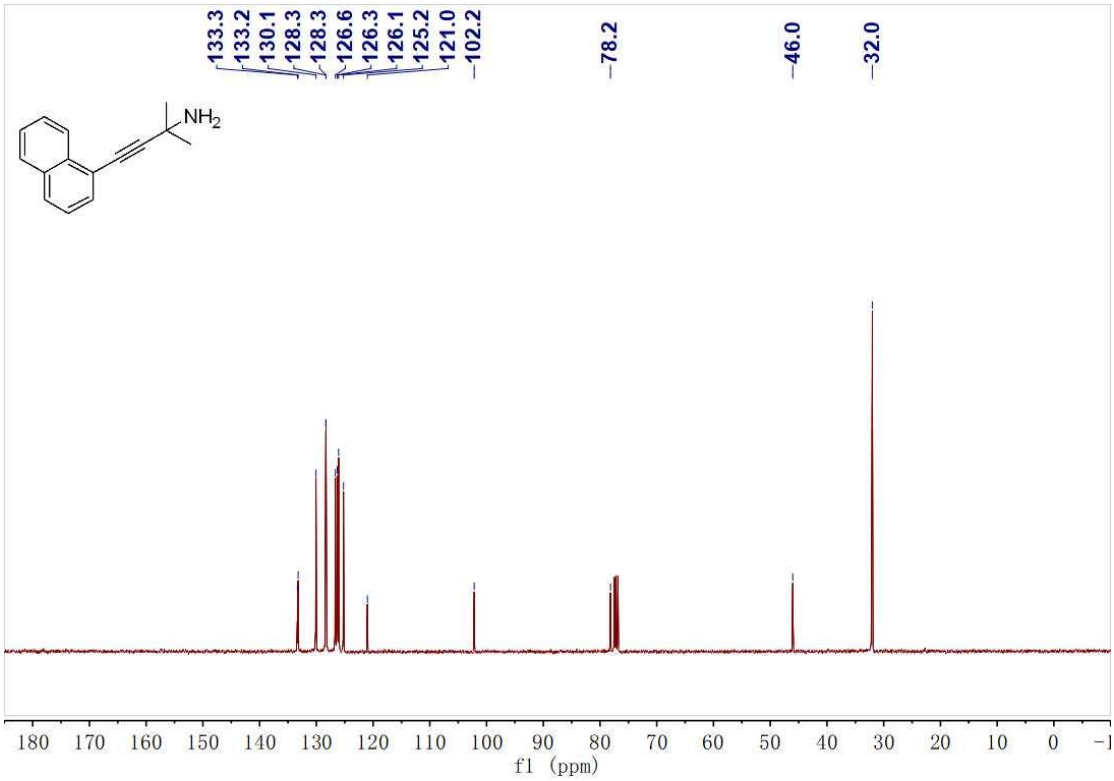
1ak


7.63
7.62
7.61
7.58
7.58
7.57
7.56
7.53
7.53
7.51
7.51
7.49
7.47
7.45
7.40
7.39
1.81
1.57
NH2
7.8
7.6
7.4
f1 (ppm)
8.01
1.00
2.00
6.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

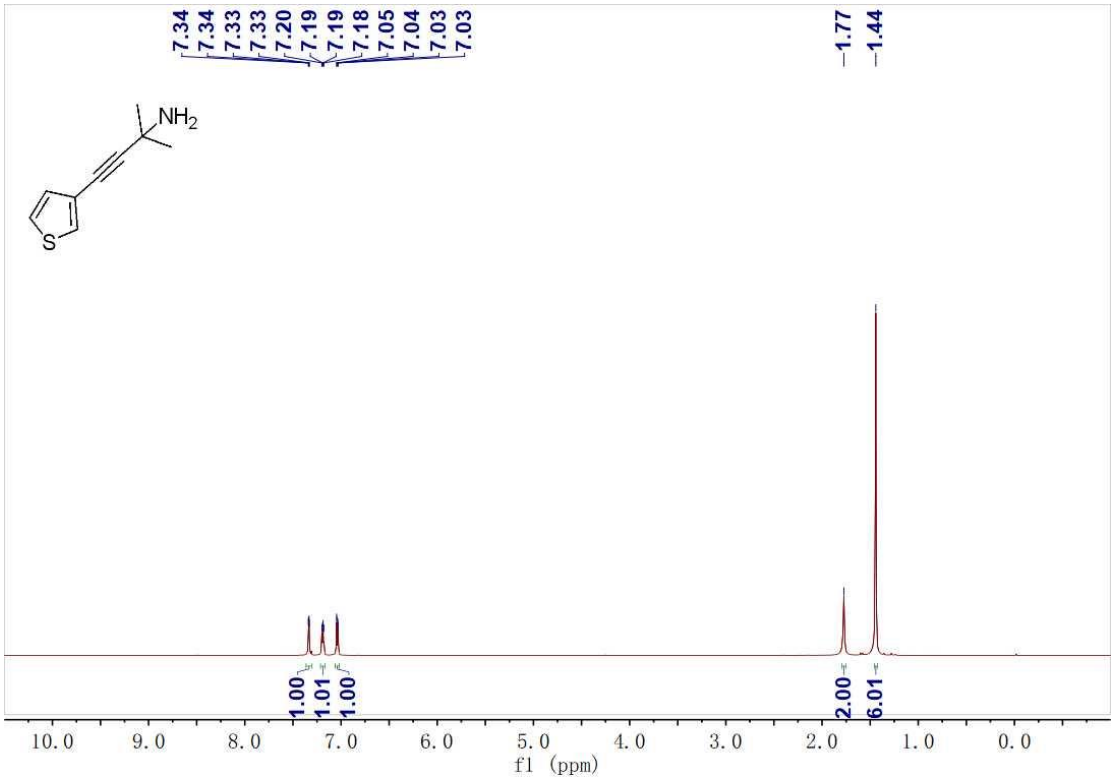

140.5
140.4
132.0
128.9
127.6
127.0
126.9
97.8
79.9
45.7
31.9
NH2
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

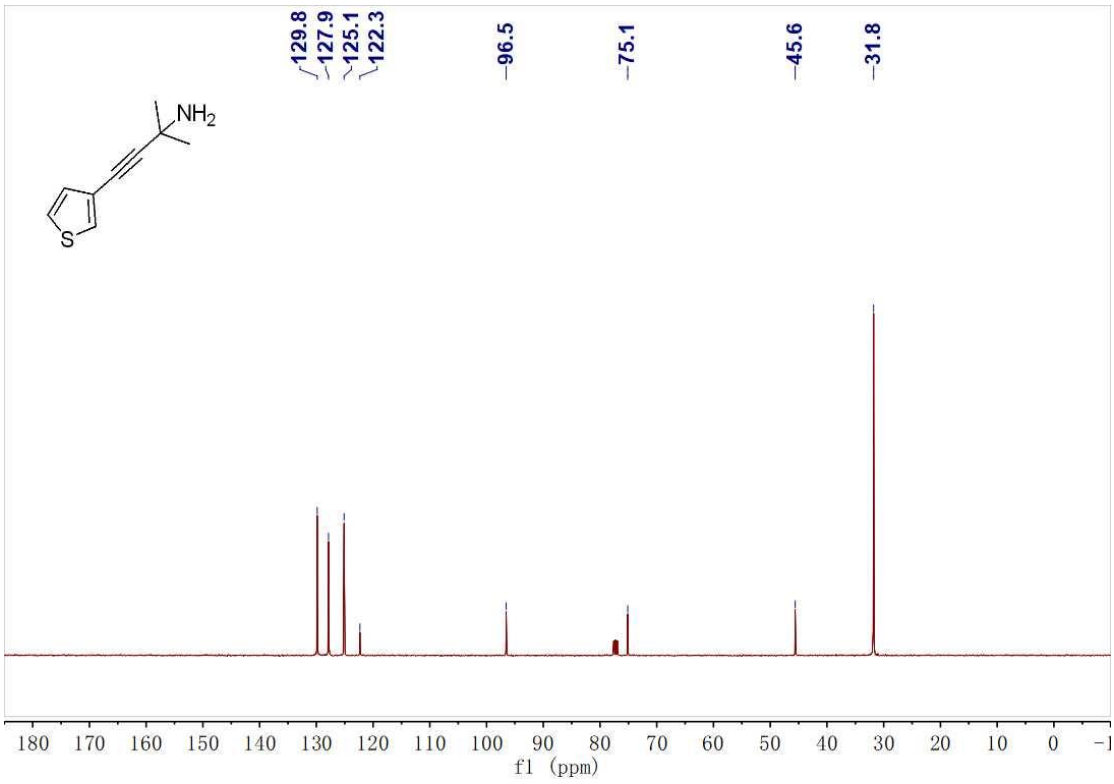
1al


8.37
8.35
7.89
7.87
7.84
7.82
7.69
7.68
7.64
7.63
7.62
7.61
7.58
7.56
7.54
7.47
7.45
7.43
1.96
1.66
NH2
f1 (ppm)
0.95
2.00
3.01
1.00
2.00
6.01

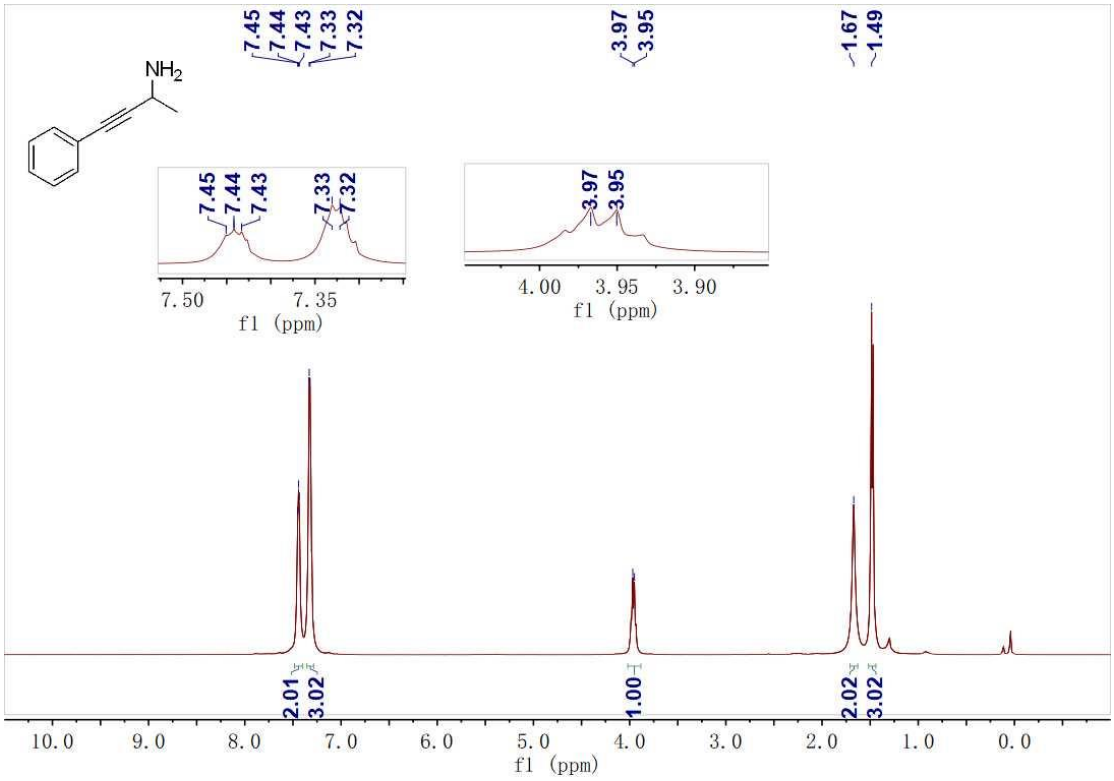

133.3
133.2
130.1
128.3
128.3
126.6
126.3
126.1
125.2
121.0
102.2
78.2
46.0
32.0
NH2
f1 (ppm)

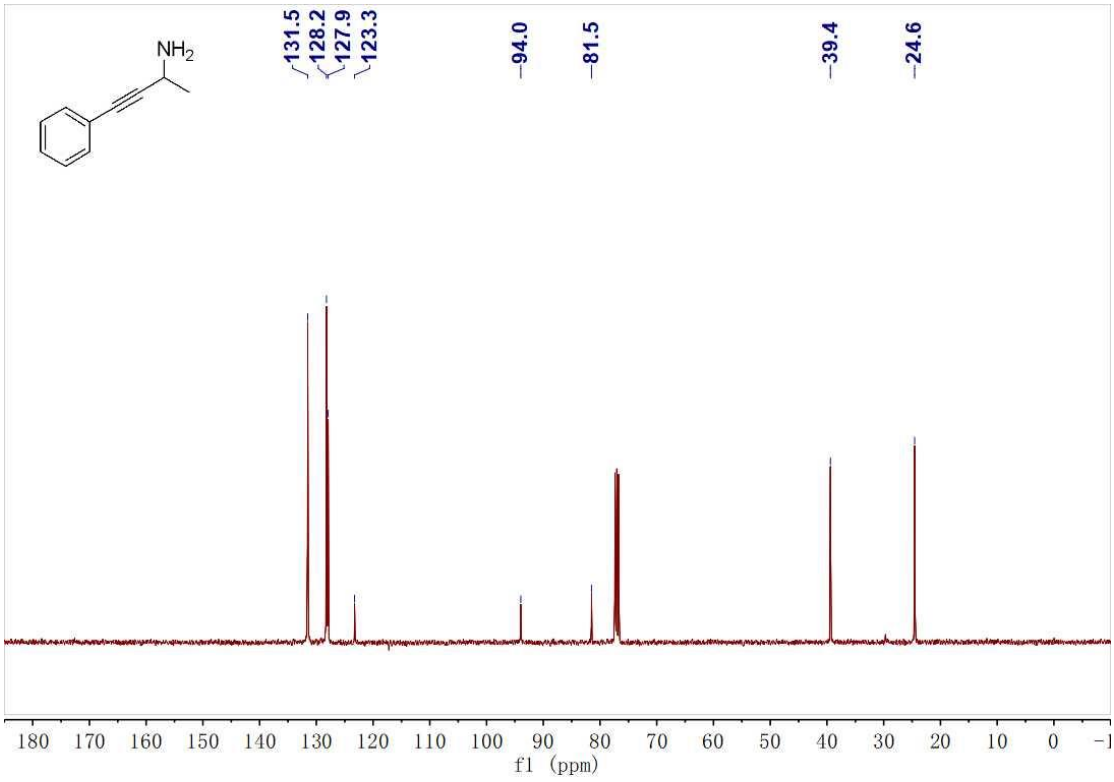
1am


7.34
7.34
7.33
7.33
7.20
7.19
7.19
7.18
7.05
7.04
7.03
7.03
1.77
1.44
NH2
S
1.00
1.01
1.00
2.00
6.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

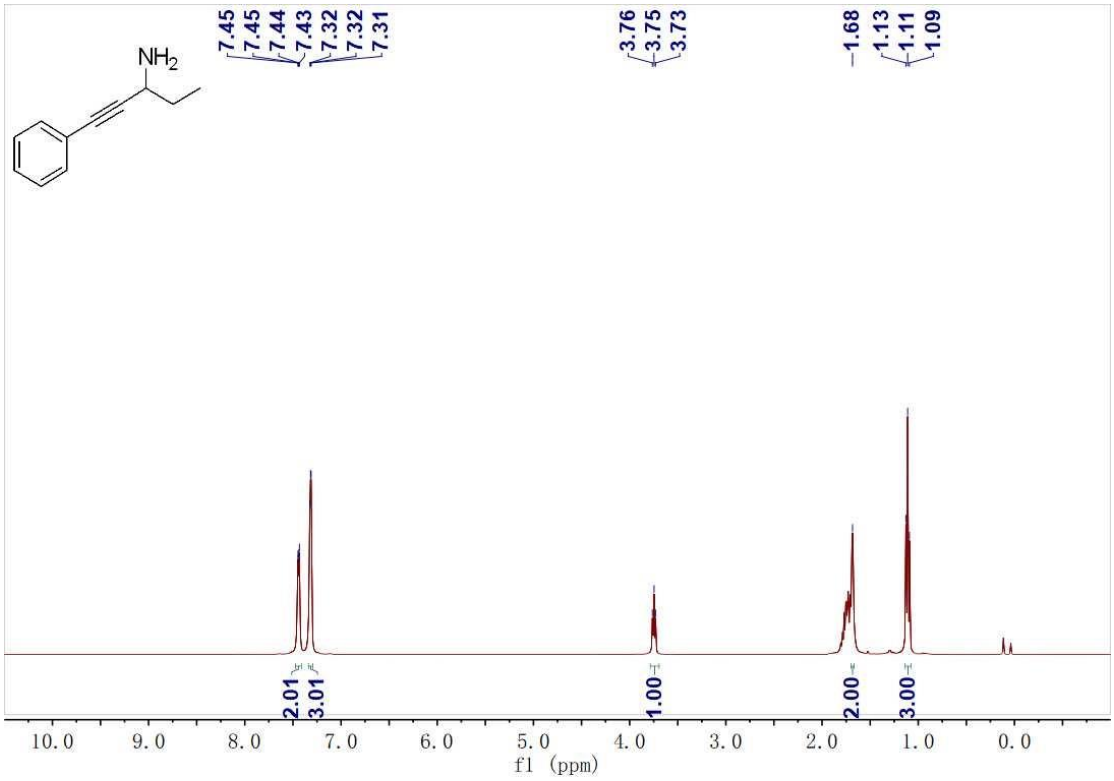

129.8
127.9
125.1
122.3
96.5
75.1
45.6
31.8
NH2
S
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

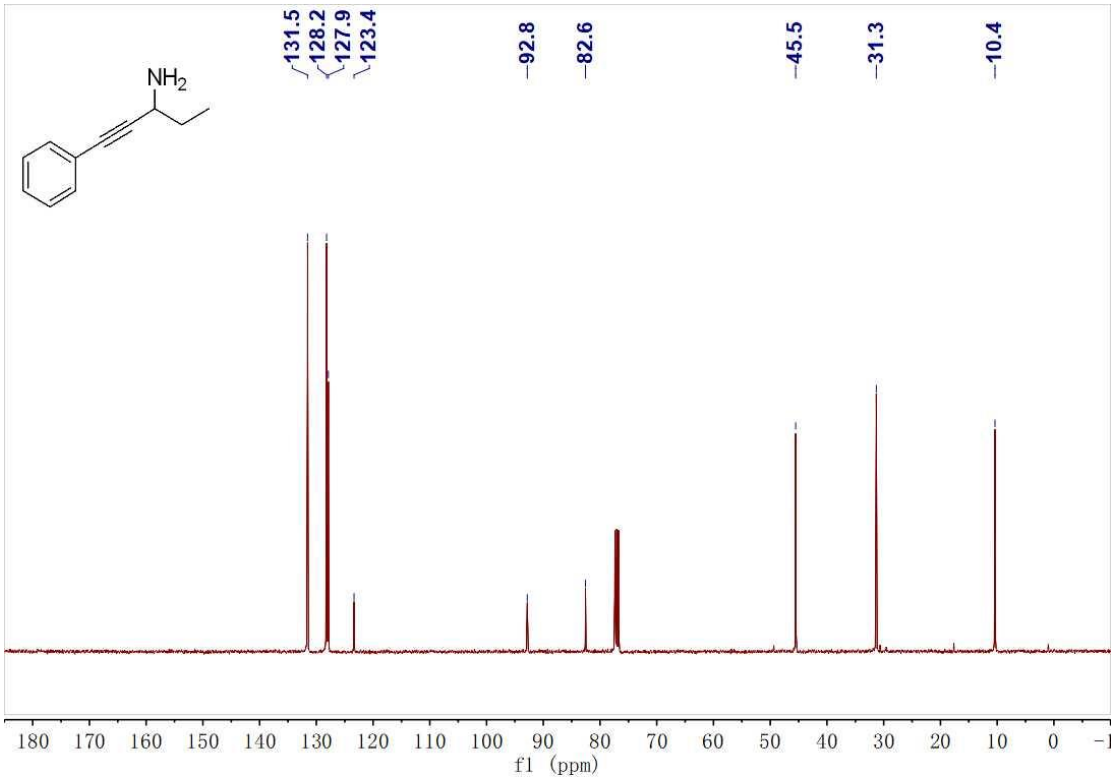
1ap


7.45
7.44
7.43
7.33
7.32
3.97
3.95
1.67
1.49
NH2
7.50
7.35
f1 (ppm)
4.00
3.95
3.90
f1 (ppm)
2.01
3.02
1.00
2.02
3.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


131.5
128.2
127.9
123.3
94.0
81.5
39.4
24.6
NH2
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

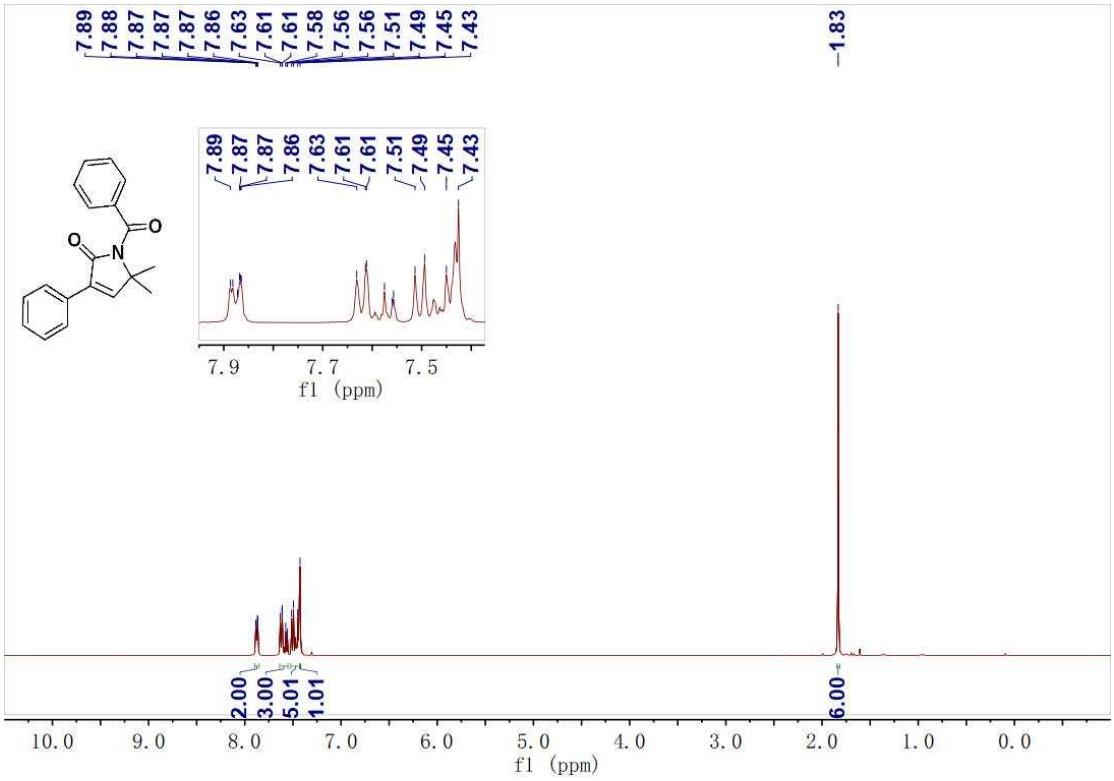
1aq

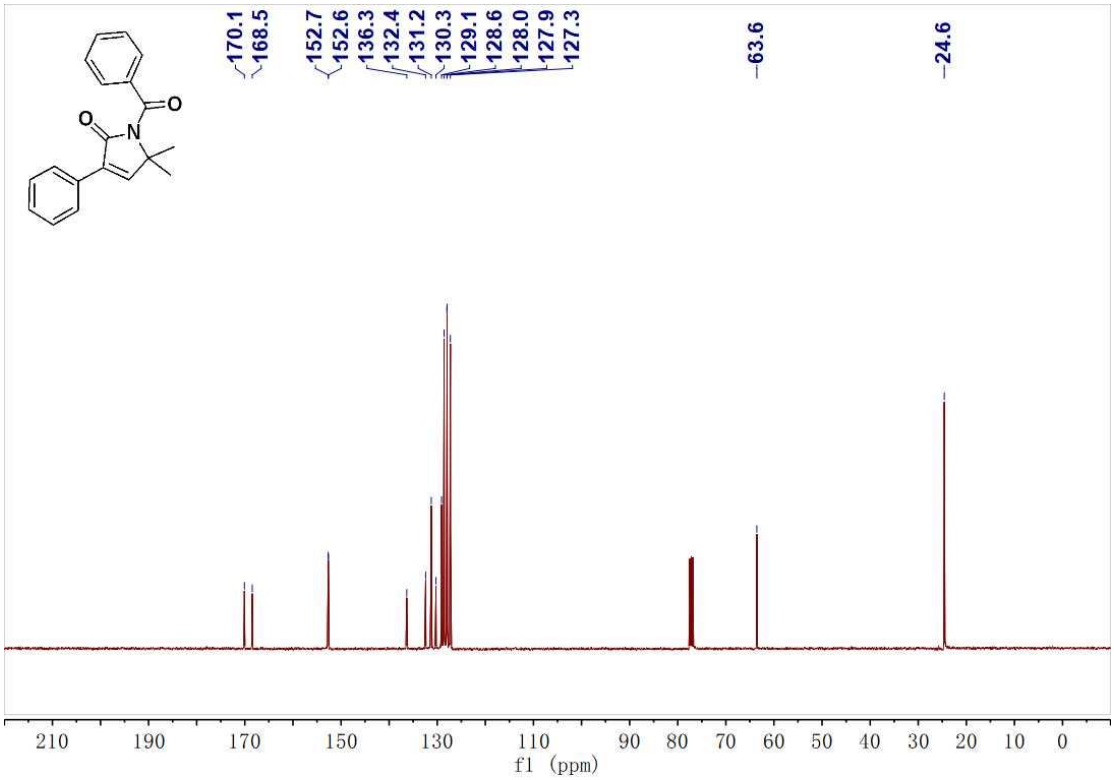

NH2
7.45
7.45
7.44
7.43
7.32
7.32
7.31
3.76
3.75
3.73
1.68
1.13
1.11
1.09
2.01
3.01
1.00
2.00
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


NH2
131.5
128.2
127.9
123.4
92.8
82.6
45.5
31.3
10.4
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

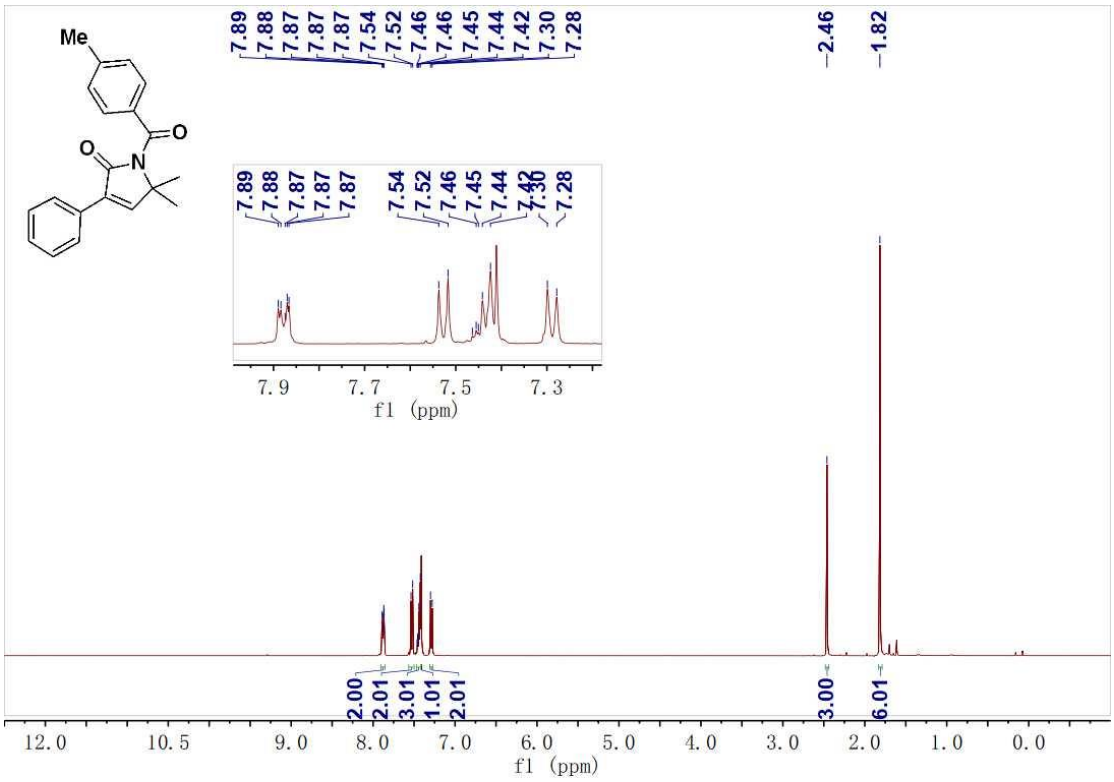
10. Spectra of Products

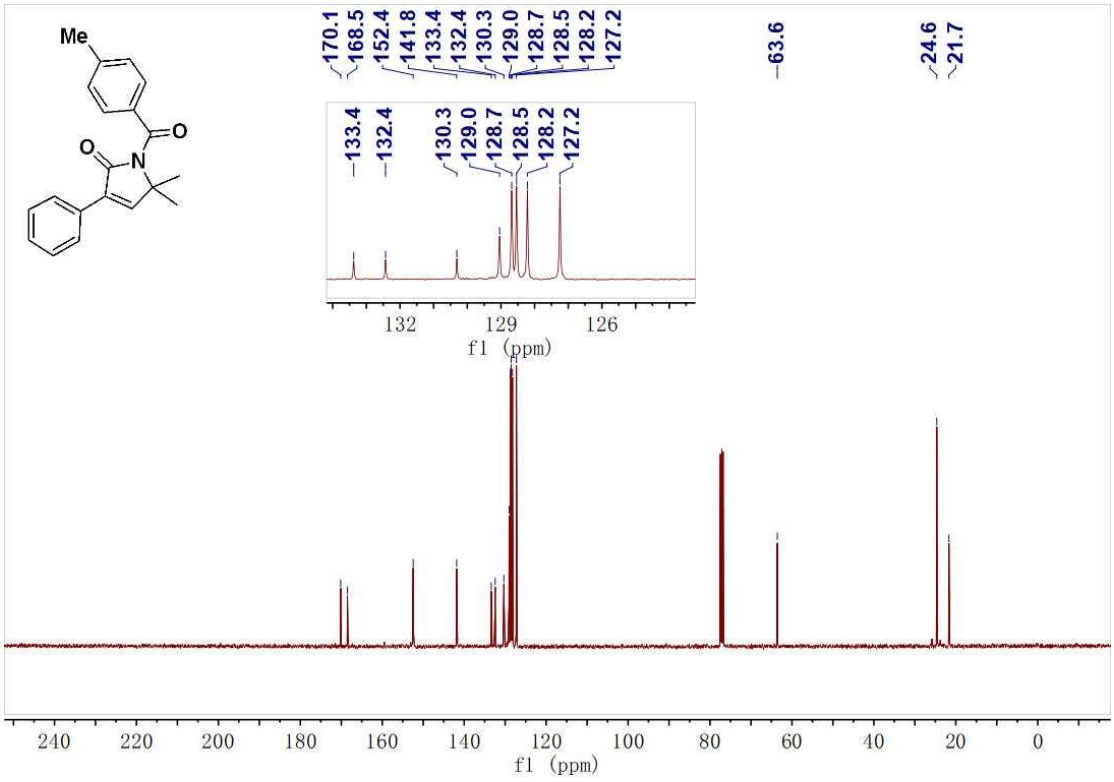
2a



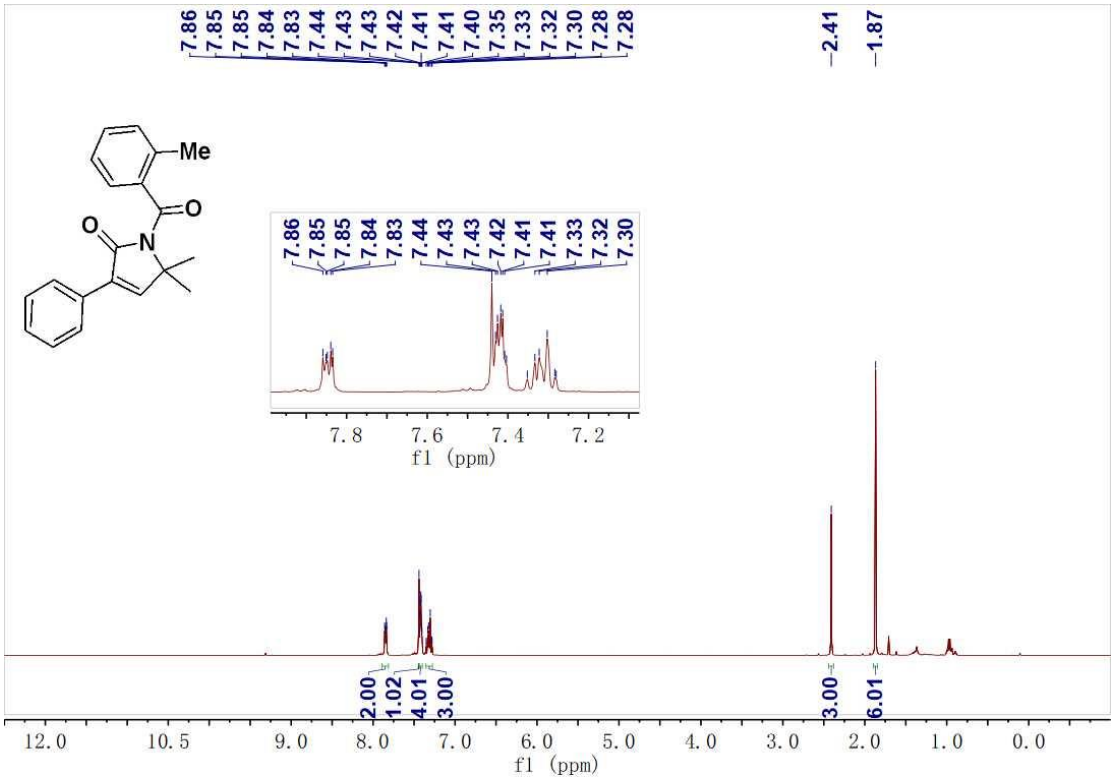


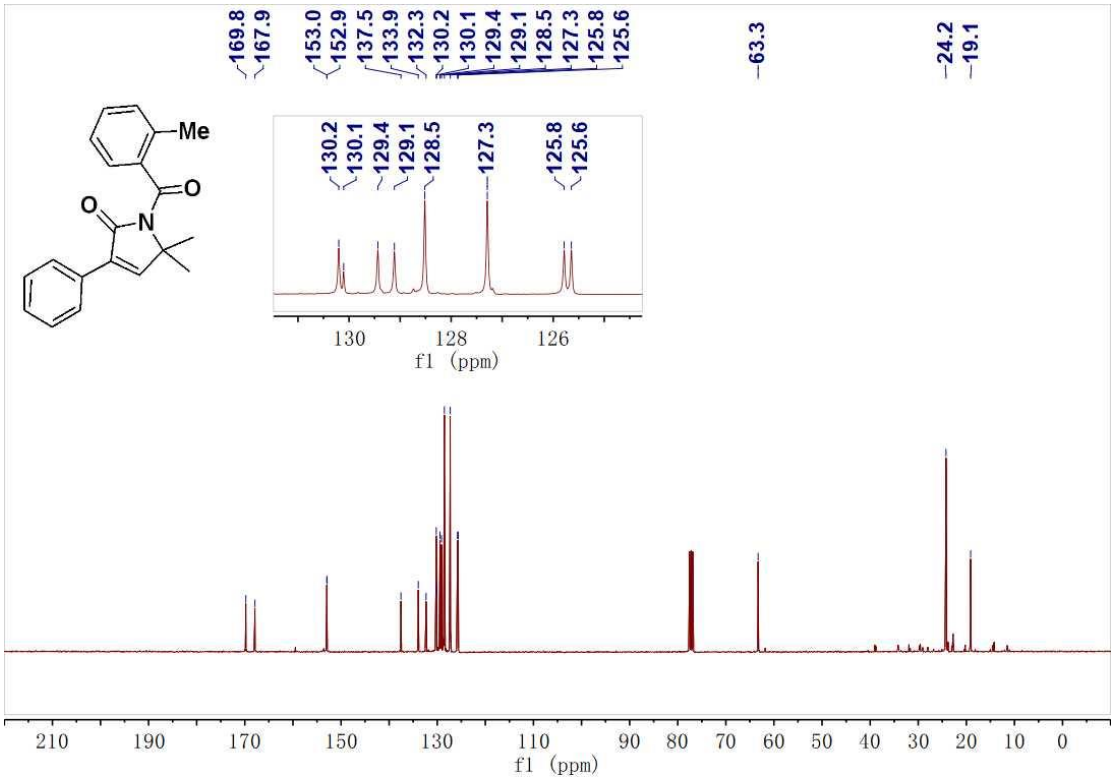
2b


Me
O
O
N
7.89
7.88
7.87
7.87
7.87
7.54
7.52
7.46
7.46
7.45
7.44
7.42
7.30
7.28
2.46
1.82
7.9
7.7
7.5
7.3
f1 (ppm)
2.00
2.01
3.01
1.01
2.01
3.00
6.01
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

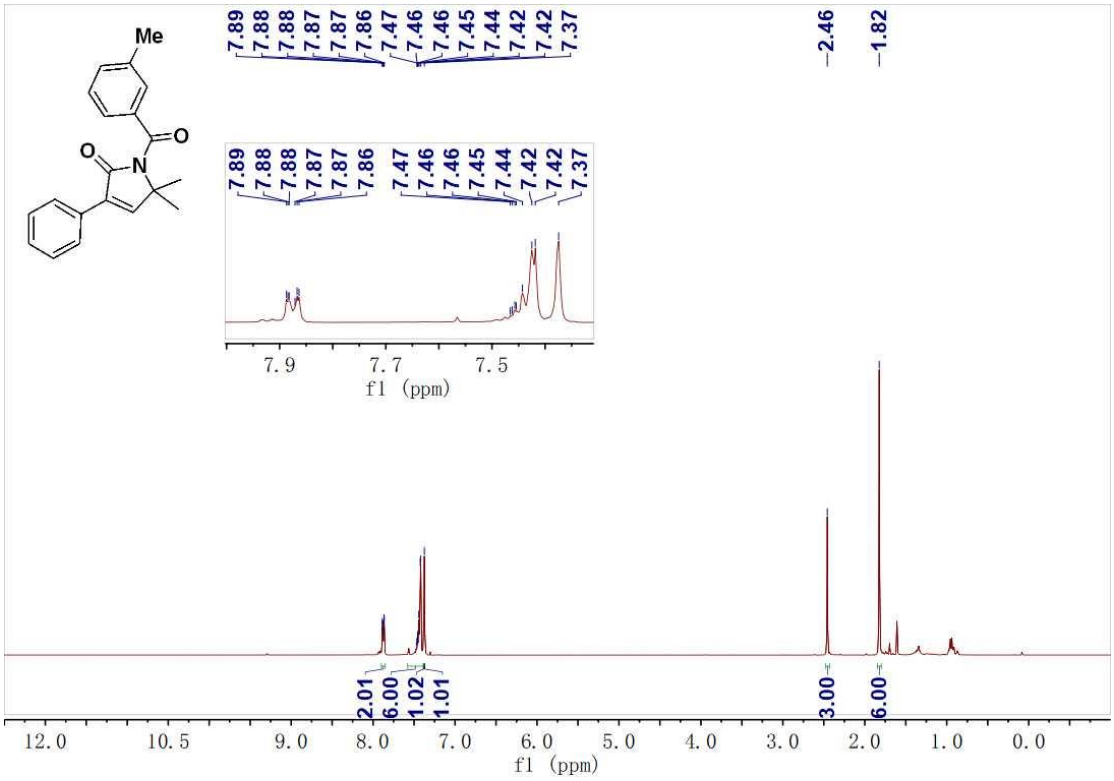

Me
O
O
N
170.1
168.5
152.4
141.8
133.4
132.4
130.3
129.0
128.7
128.5
128.2
127.2
63.6
24.6
21.7
133.4
132.4
130.3
129.0
128.7
128.5
128.2
127.2
132
129
126
f1 (ppm)
240
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

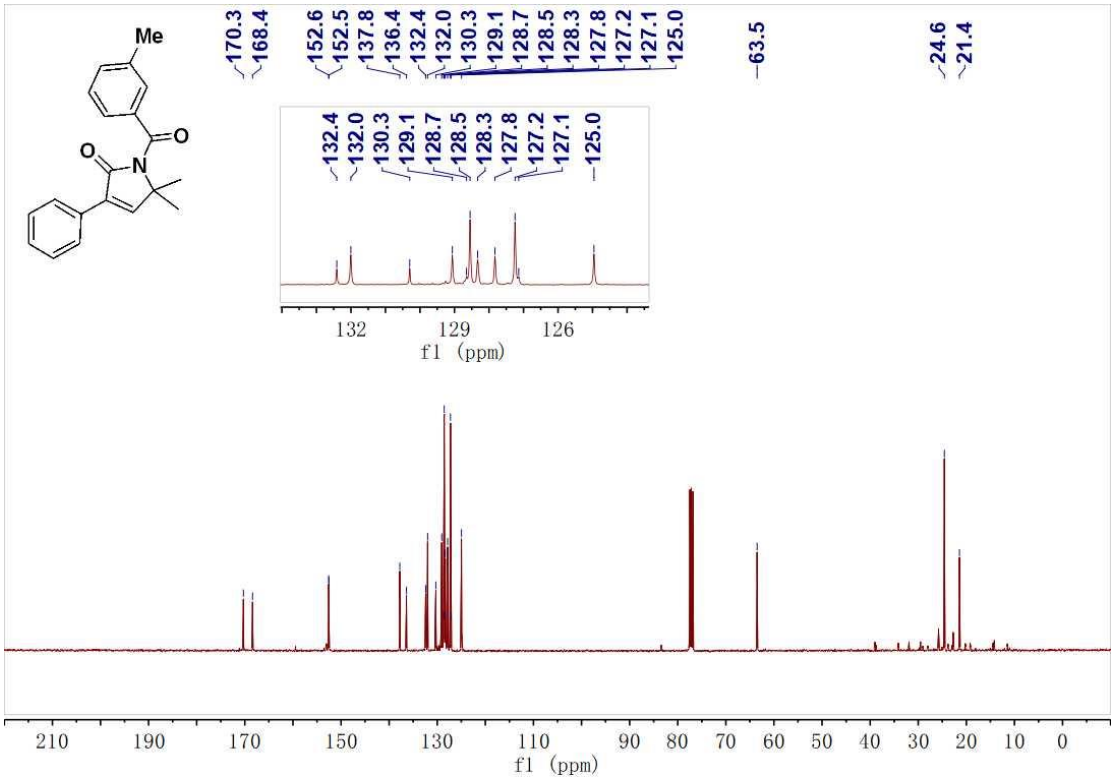
2c


7.86
7.85
7.85
7.84
7.83
7.44
7.43
7.43
7.42
7.41
7.41
7.40
7.35
7.33
7.32
7.30
7.28
7.28
2.41
1.87
Me
O
O
N
7.8
7.6
7.4
7.2
f1 (ppm)
2.00
1.02
4.01
3.00
3.00
6.01
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

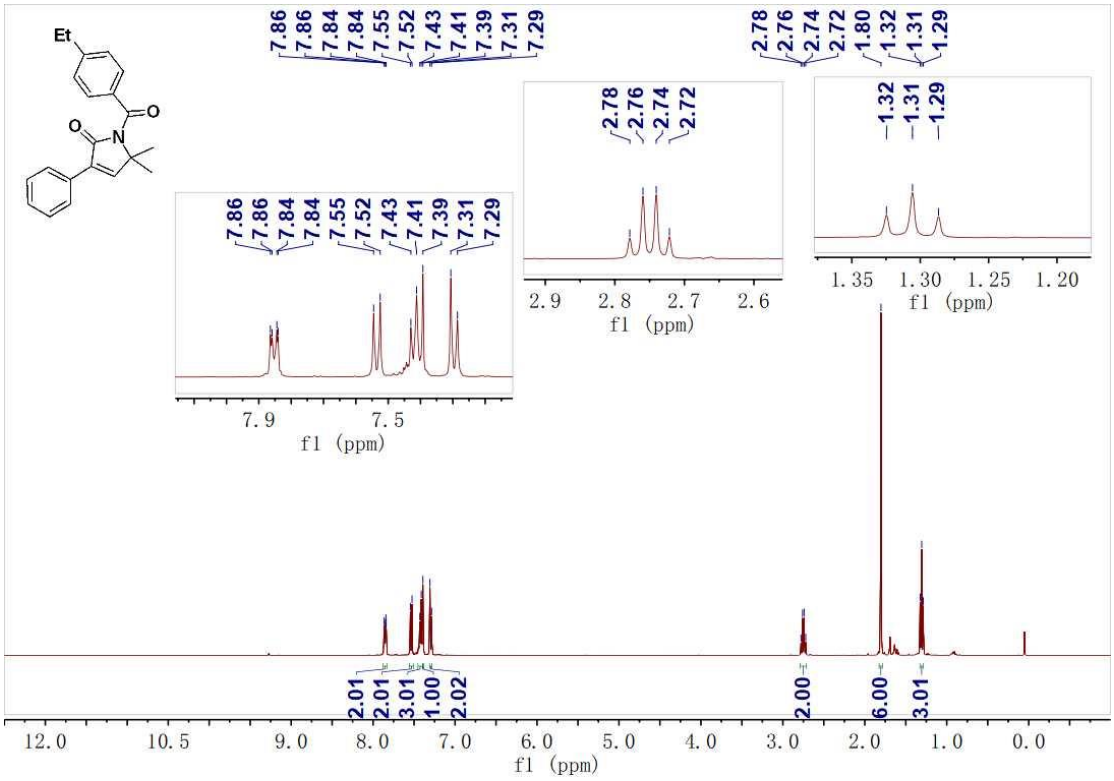

169.8
167.9
153.0
152.9
137.5
133.9
132.3
130.2
130.1
129.4
129.1
128.5
127.3
125.8
125.6
63.3
24.2
19.1
Me
O
O
N
130.2
130.1
129.4
129.1
128.5
127.3
125.8
125.6
130
128
126
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

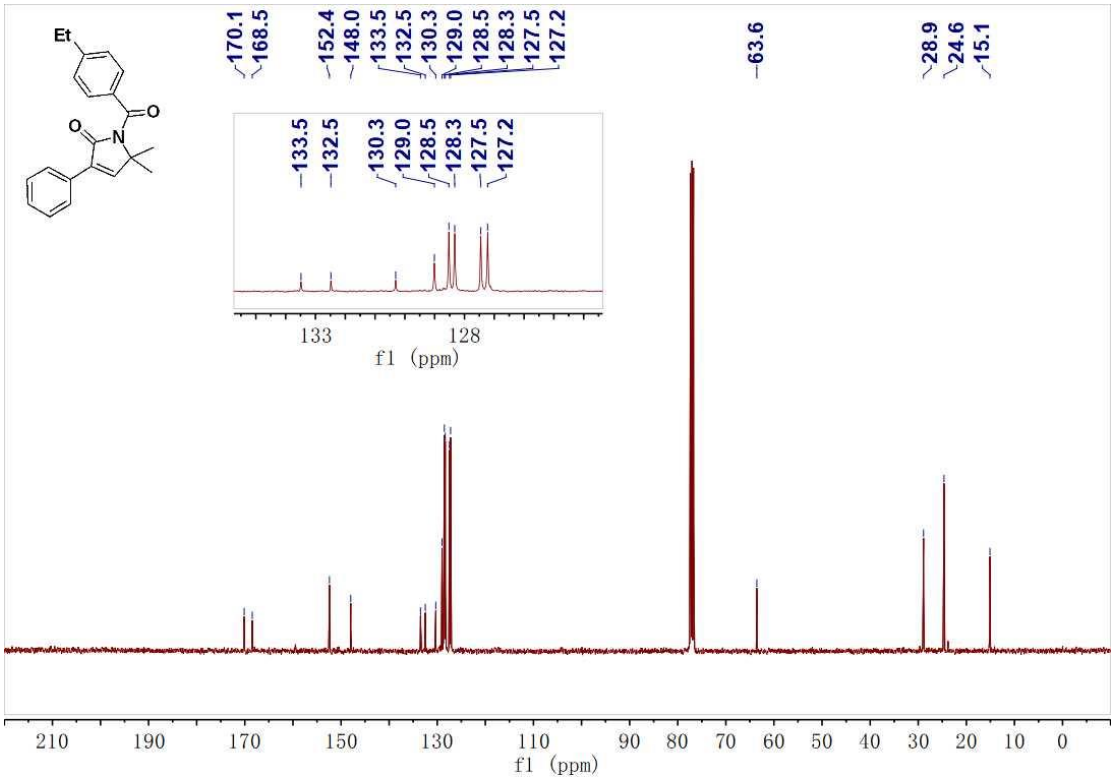
2d


Me
O
O
N
7.89
7.88
7.88
7.87
7.87
7.86
7.47
7.46
7.46
7.45
7.44
7.42
7.42
7.37
2.46
1.82
7.9
7.7
7.5
f1 (ppm)
2.01
6.00
1.02
1.01
3.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

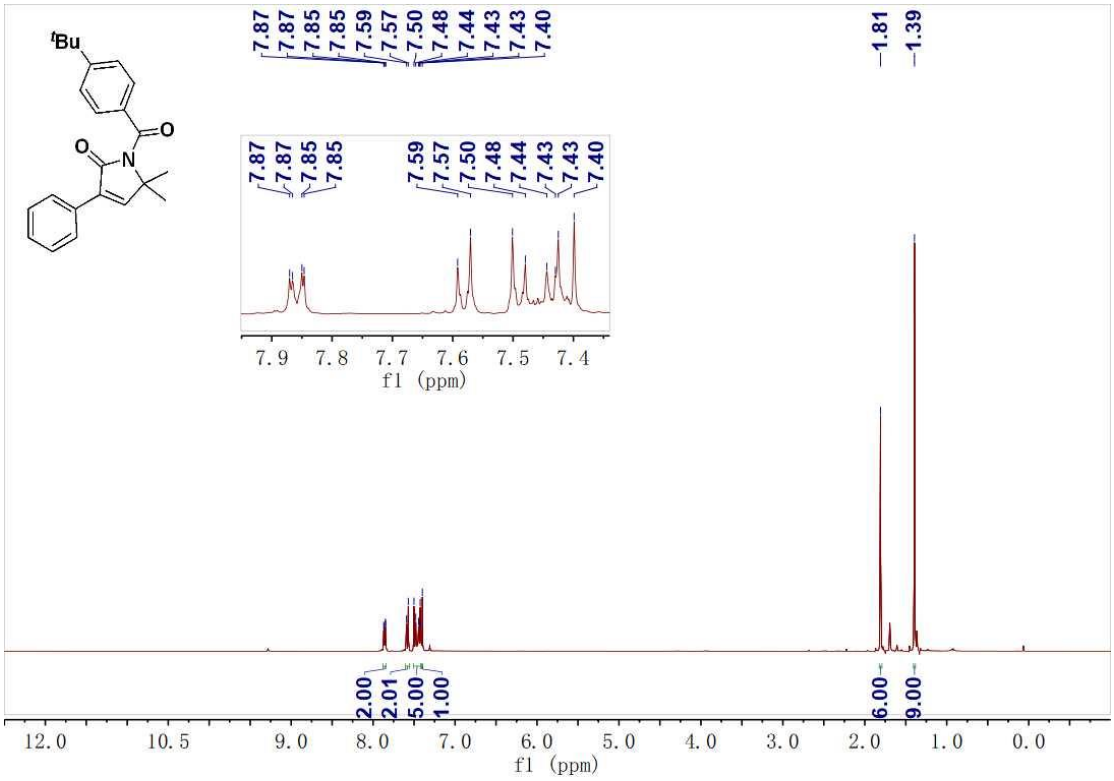

Me
O
O
N
170.3
168.4
152.6
152.5
137.8
136.4
132.4
132.0
130.3
129.1
128.7
128.5
128.3
127.8
127.2
127.1
125.0
63.5
24.6
21.4
132
129
126
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

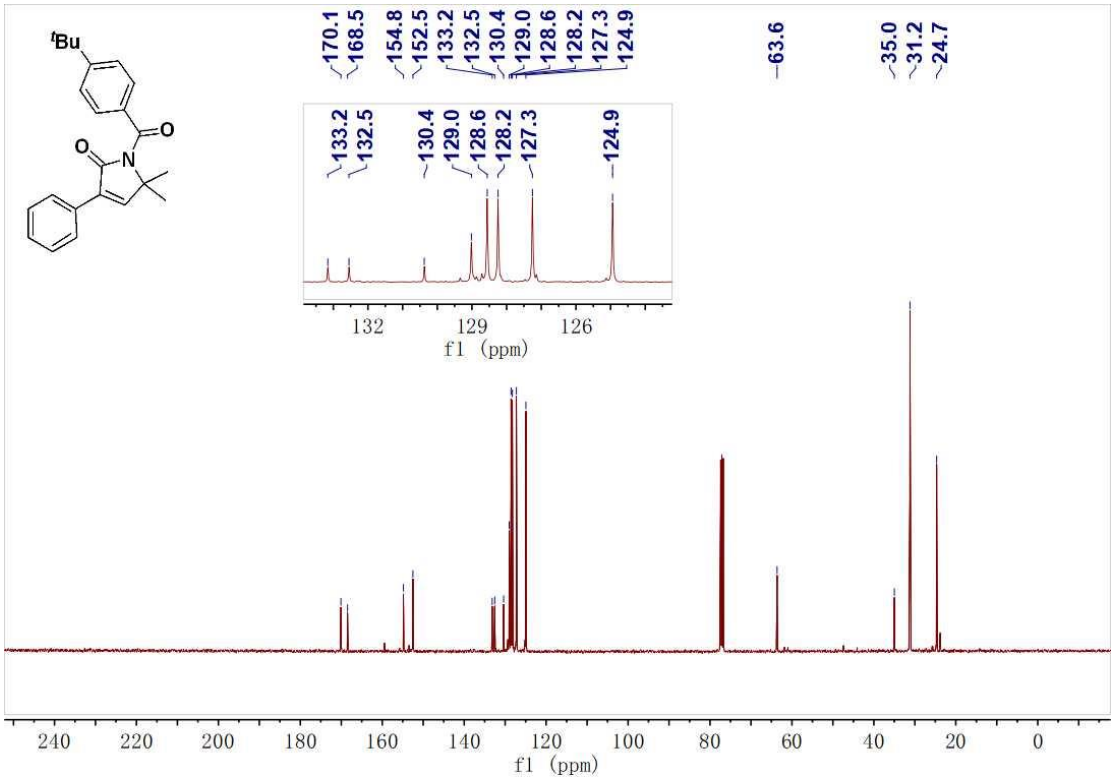
2e


Et
O
O
N
7.86
7.86
7.84
7.84
7.55
7.52
7.43
7.41
7.39
7.31
7.29
2.78
2.76
2.74
2.72
1.80
1.32
1.31
1.29
f1 (ppm)
2.01
2.01
3.01
1.00
2.02
2.00
6.00
3.01

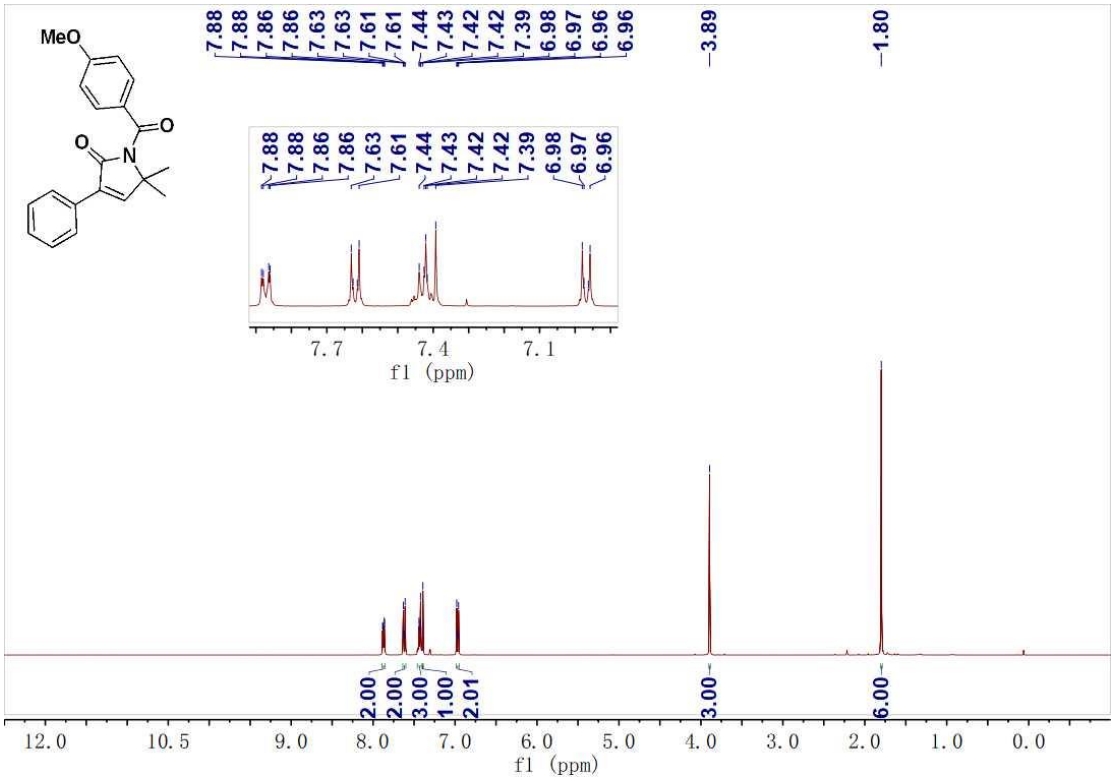

Et
O
O
N
170.1
168.5
152.4
148.0
133.5
132.5
130.3
129.0
128.5
128.3
127.5
127.2
63.6
28.9
24.6
15.1
f1 (ppm)

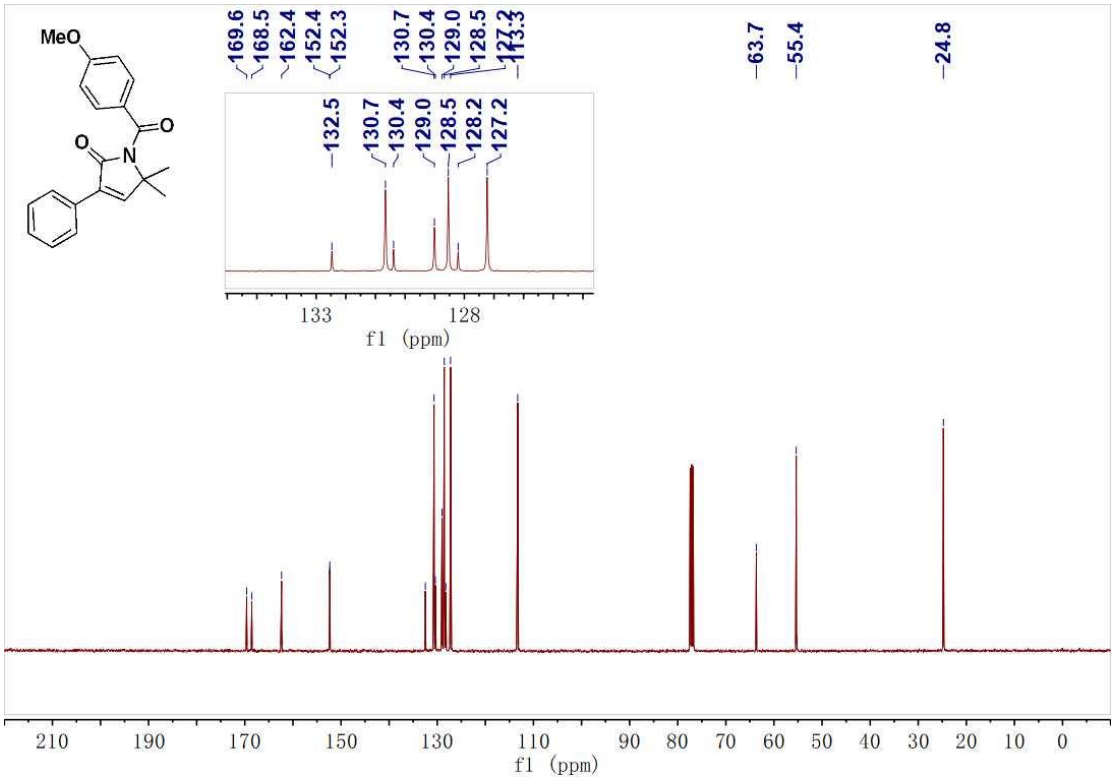
2f


7.87
7.87
7.85
7.85
7.59
7.57
7.50
7.48
7.44
7.43
7.43
7.40
1.81
1.39
2.00
2.01
5.00
1.00
6.00
9.00
f1 (ppm)

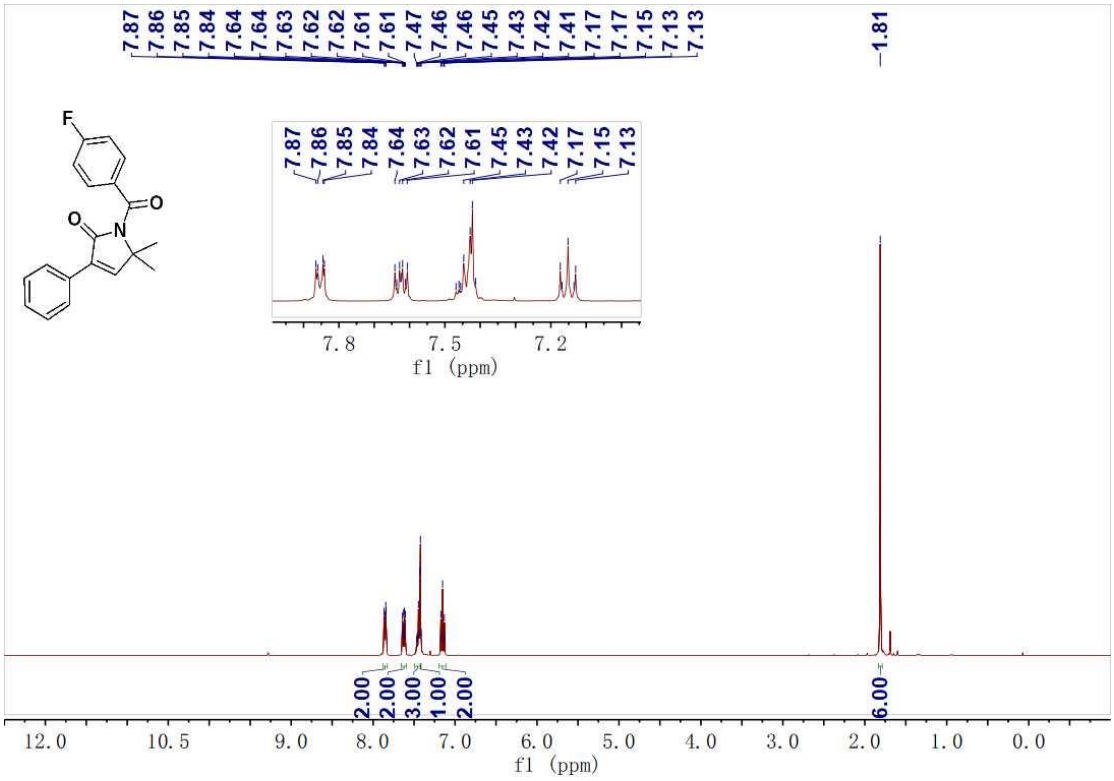

170.1
168.5
154.8
152.5
133.2
132.5
130.4
129.0
128.6
128.2
127.3
124.9
63.6
35.0
31.2
24.7
f1 (ppm)

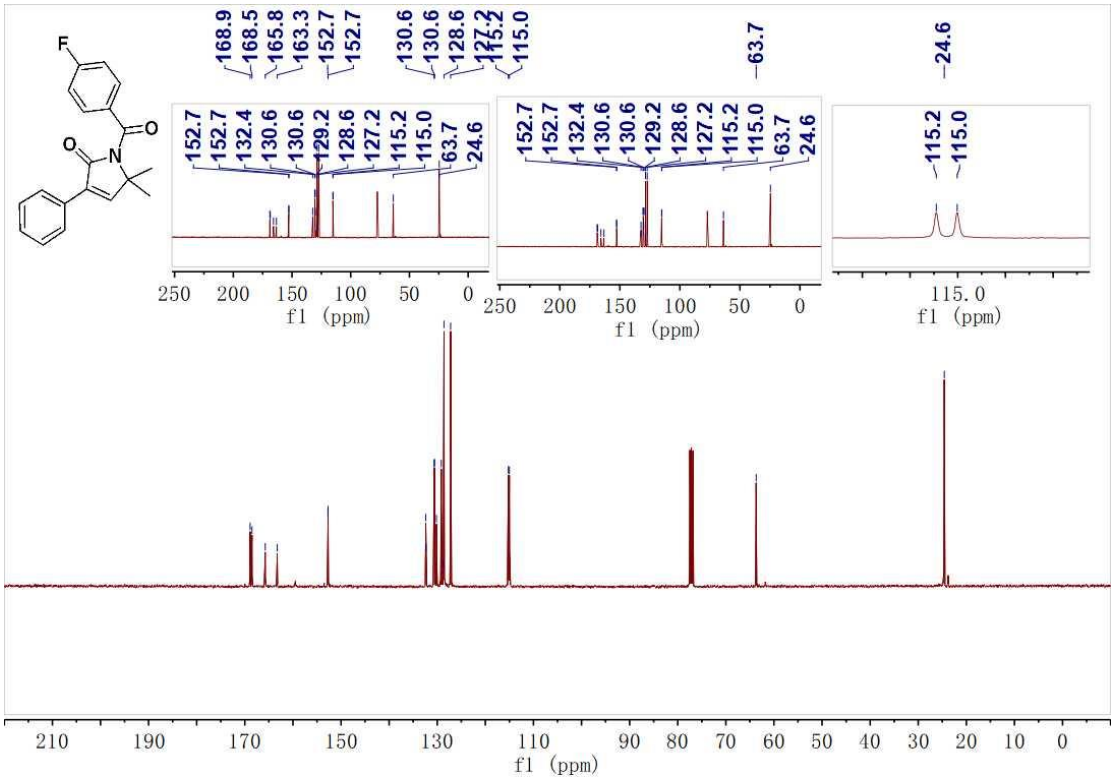
2g


MeO
O
O
N
7.88
7.88
7.86
7.86
7.63
7.63
7.61
7.61
7.44
7.43
7.42
7.42
7.39
6.98
6.97
6.96
6.96
3.89
1.80
7.7
7.4
7.1
f1 (ppm)
2.00
2.00
3.00
1.00
2.01
3.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

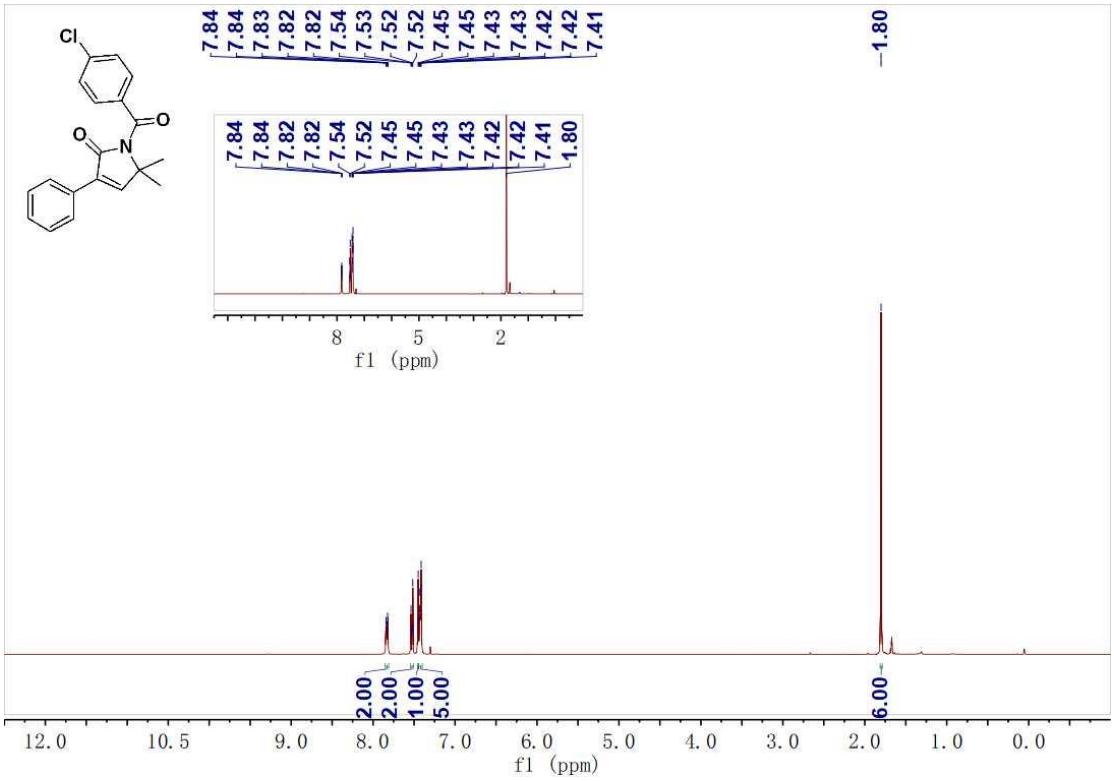

MeO
O
O
N
169.6
168.5
162.4
152.4
152.3
130.7
130.4
129.0
128.5
128.2
127.2
113.3
63.7
55.4
24.8
132.5
130.7
130.4
129.0
128.5
128.2
127.2
133
128
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

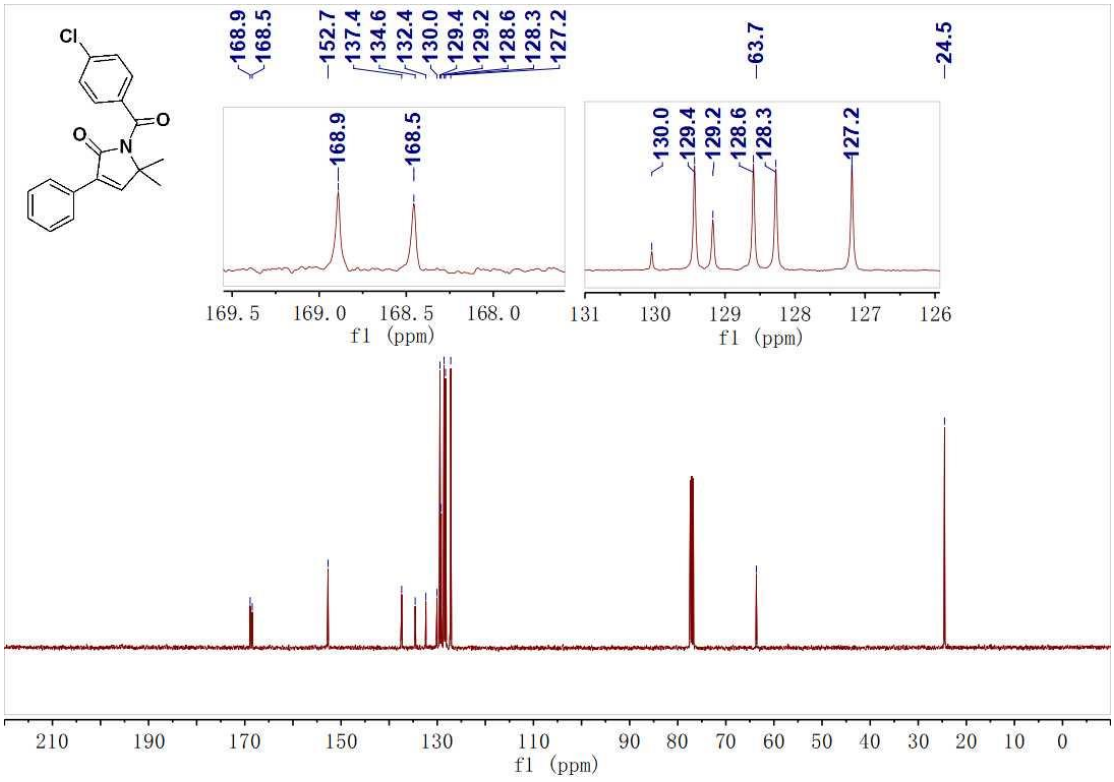
2h


7.87
7.86
7.85
7.84
7.64
7.64
7.63
7.62
7.62
7.61
7.61
7.47
7.46
7.46
7.45
7.43
7.42
7.41
7.17
7.17
7.15
7.13
7.13
1.81
F
O
O
N
7.8
7.5
7.2
f1 (ppm)
2.00
2.00
3.00
1.00
2.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

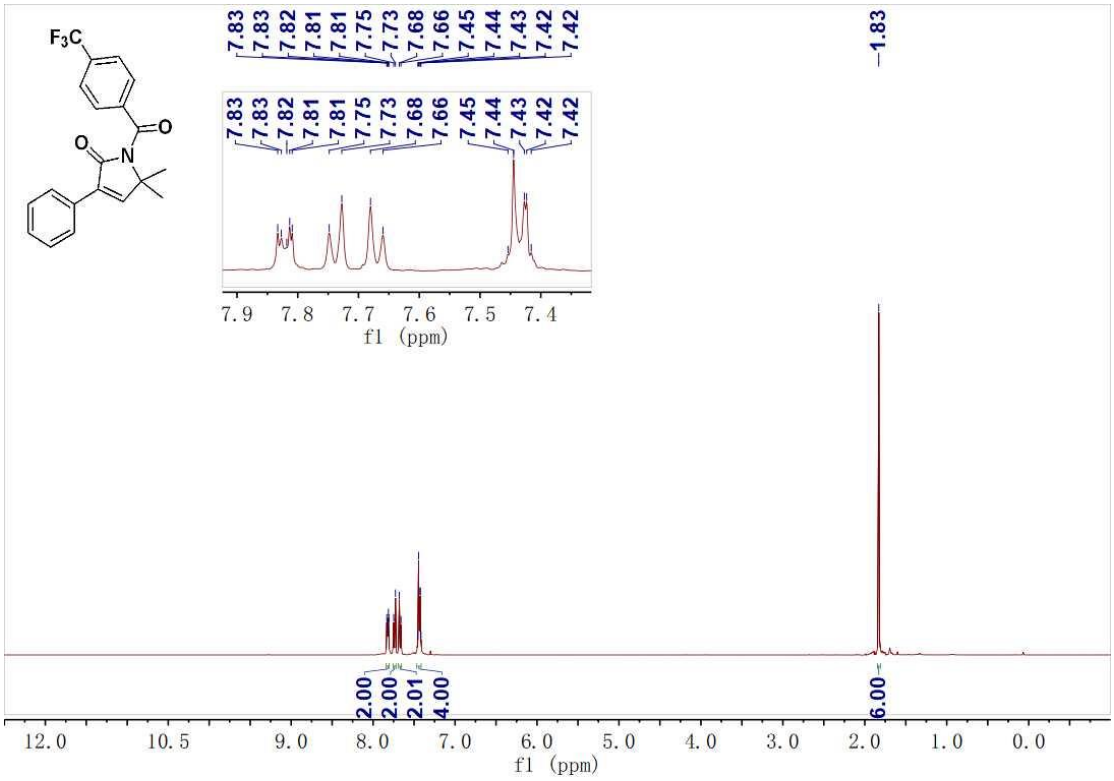

168.9
168.5
165.8
163.3
152.7
152.7
130.6
130.6
128.6
127.2
115.2
115.0
63.7
24.6
F
O
O
N
152.7
152.7
132.4
130.6
130.6
129.2
128.6
127.2
115.2
115.0
63.7
24.6
250
200
150
100
50
0
f1 (ppm)
115.2
115.0
115.0
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

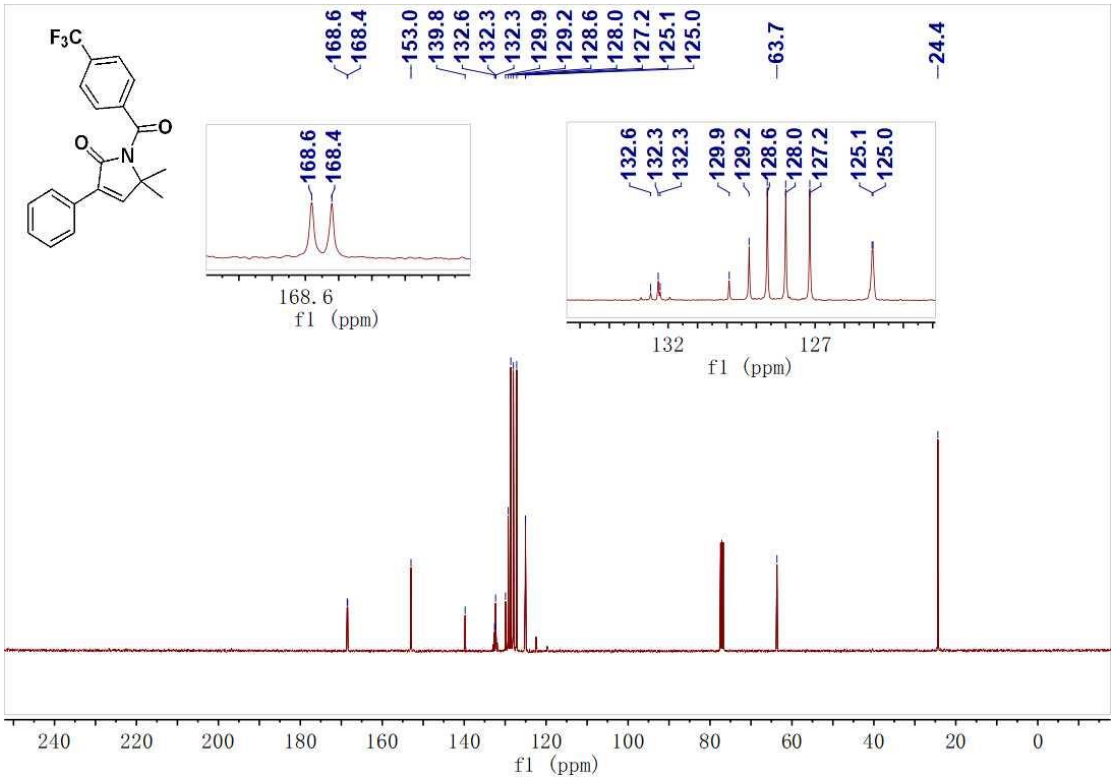
2i


Cl
O
O
N
7.84
7.84
7.83
7.82
7.82
7.54
7.53
7.52
7.52
7.45
7.45
7.43
7.43
7.42
7.42
7.41
1.80
8
5
2
f1 (ppm)
2.00
2.00
1.00
5.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

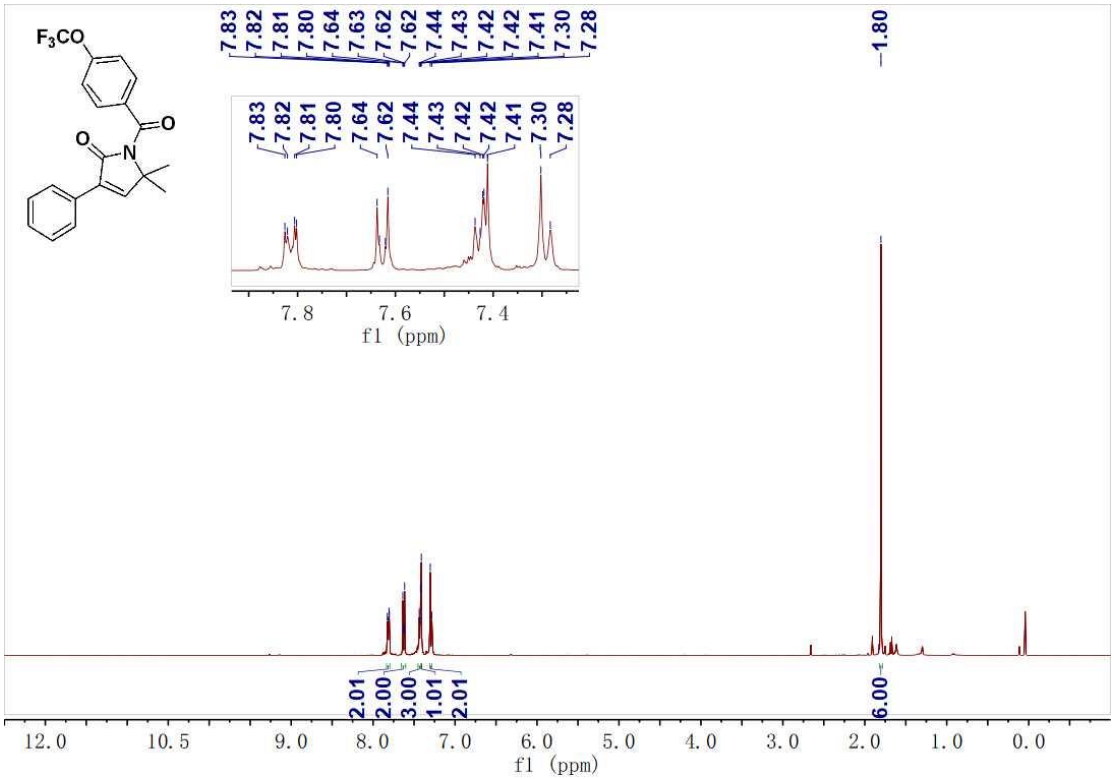

Cl
O
O
N
168.9
168.5
152.7
137.4
134.6
132.4
130.0
129.4
129.2
128.6
128.3
127.2
63.7
24.5
169.5
169.0
168.5
168.0
f1 (ppm)
131
130
129
128
127
126
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

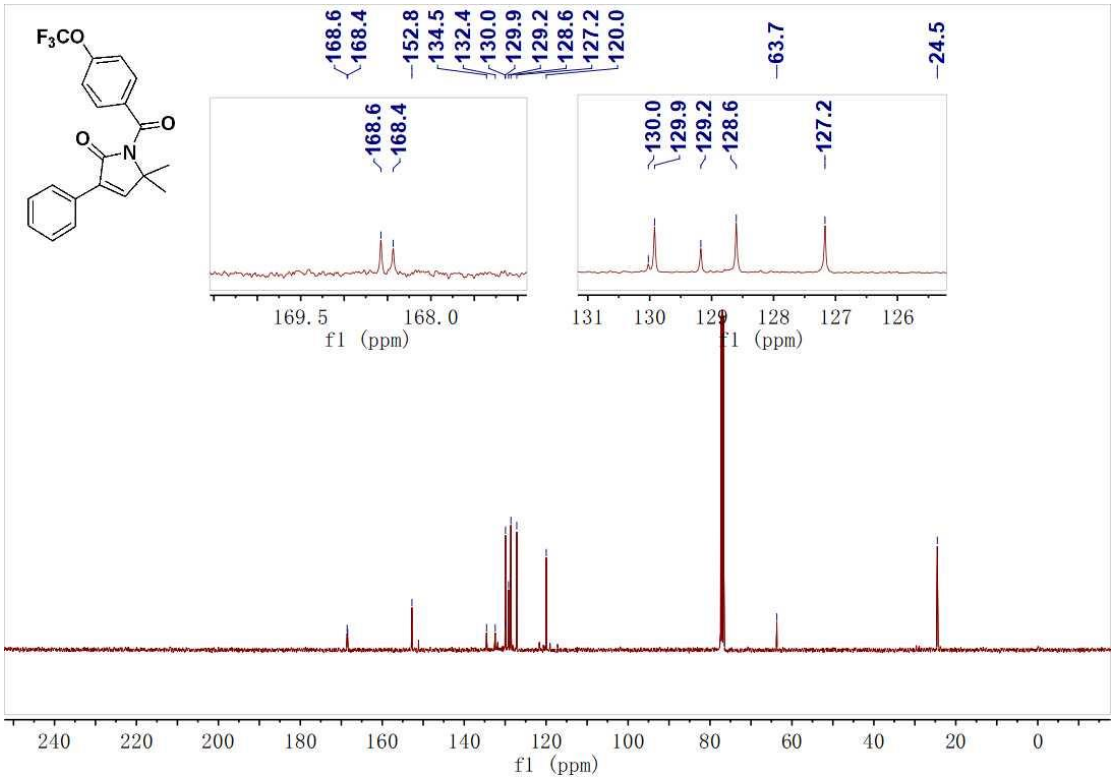
2j


F3C
O
O
N
7.83
7.83
7.82
7.81
7.81
7.75
7.73
7.68
7.66
7.45
7.44
7.43
7.42
7.42
1.83
7.9
7.8
7.7
7.6
7.5
7.4
f1 (ppm)
2.00
2.00
2.01
4.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

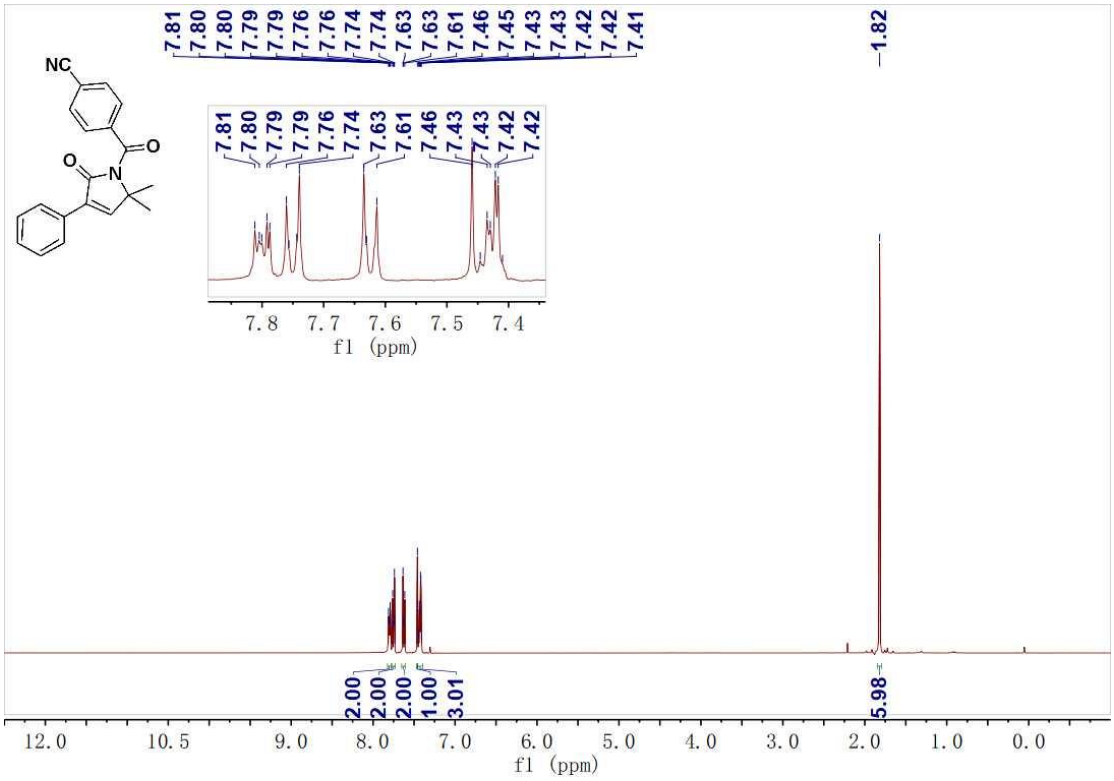

F3C
O
O
N
168.6
168.4
153.0
139.8
132.6
132.3
132.3
129.9
129.2
128.6
128.0
127.2
125.1
125.0
63.7
24.4
168.6
f1 (ppm)
132
127
f1 (ppm)
240
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

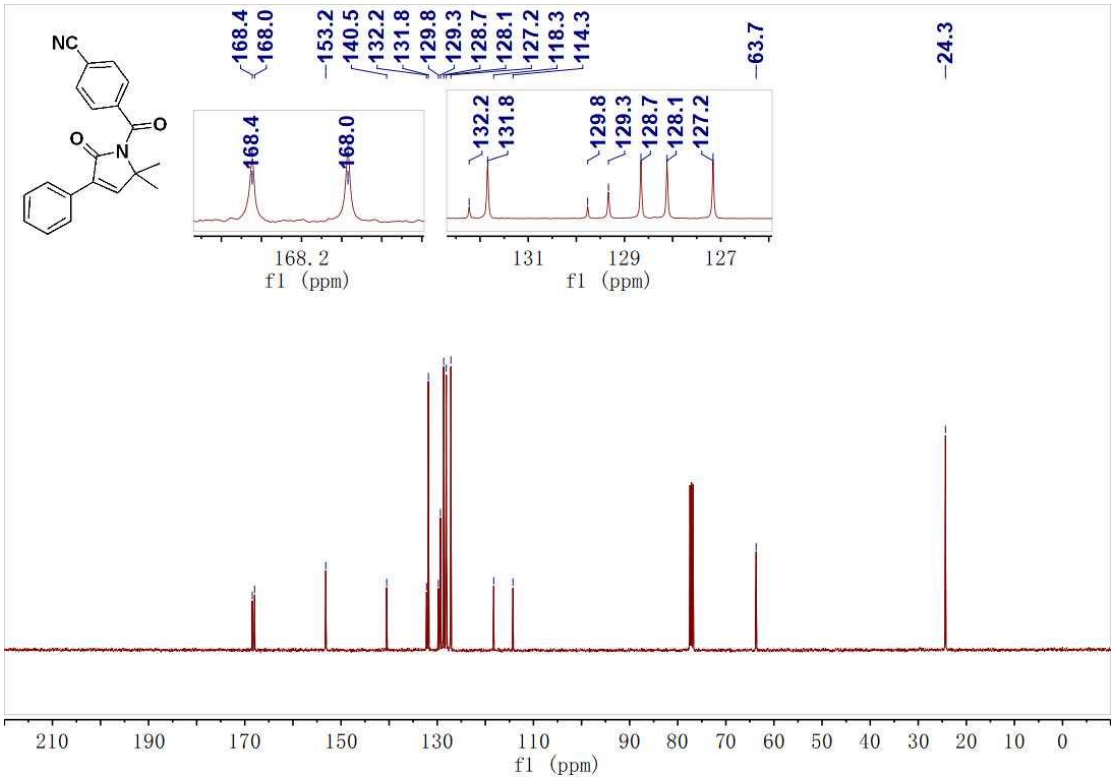
2k


F3CO
O
N
O
7.83
7.82
7.81
7.80
7.64
7.63
7.62
7.62
7.44
7.43
7.42
7.42
7.41
7.30
7.28
1.80
7.8
7.6
7.4
f1 (ppm)
2.01
2.00
3.00
1.01
2.01
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

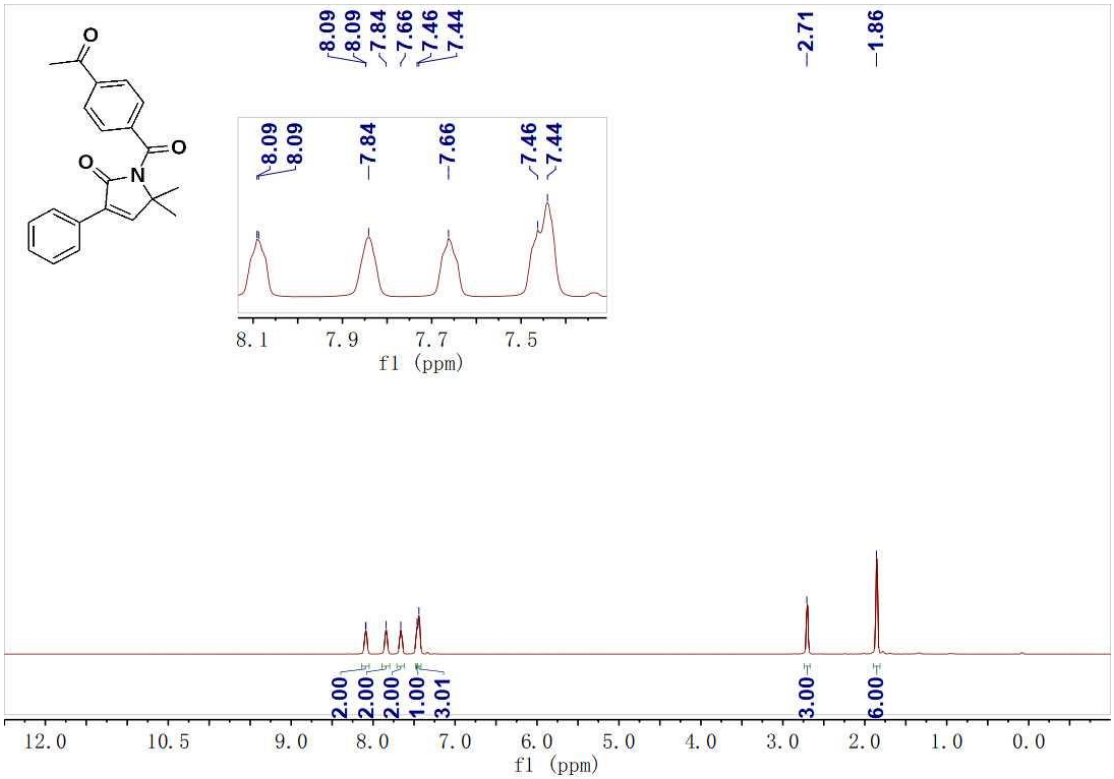

F3CO
O
N
O
168.6
168.4
152.8
134.5
132.4
130.0
129.9
129.2
128.6
127.2
120.0
63.7
24.5
169.5
168.0
f1 (ppm)
131
130
129
128
127
126
f1 (ppm)
240
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

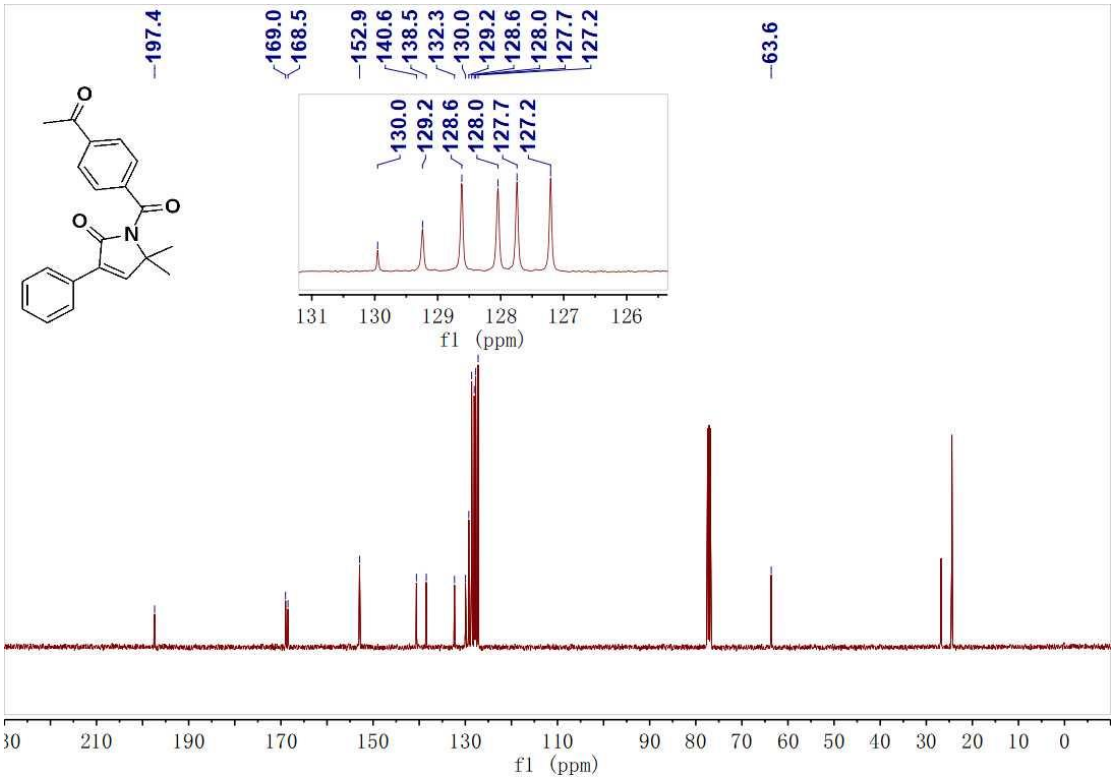
2l


7.81
7.80
7.80
7.79
7.79
7.76
7.76
7.74
7.74
7.63
7.63
7.61
7.46
7.45
7.43
7.43
7.42
7.42
7.41
1.82
NC
O
O
N
7.8
7.7
7.6
7.5
7.4
f1 (ppm)
2.00
2.00
2.00
1.00
3.01
5.98
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

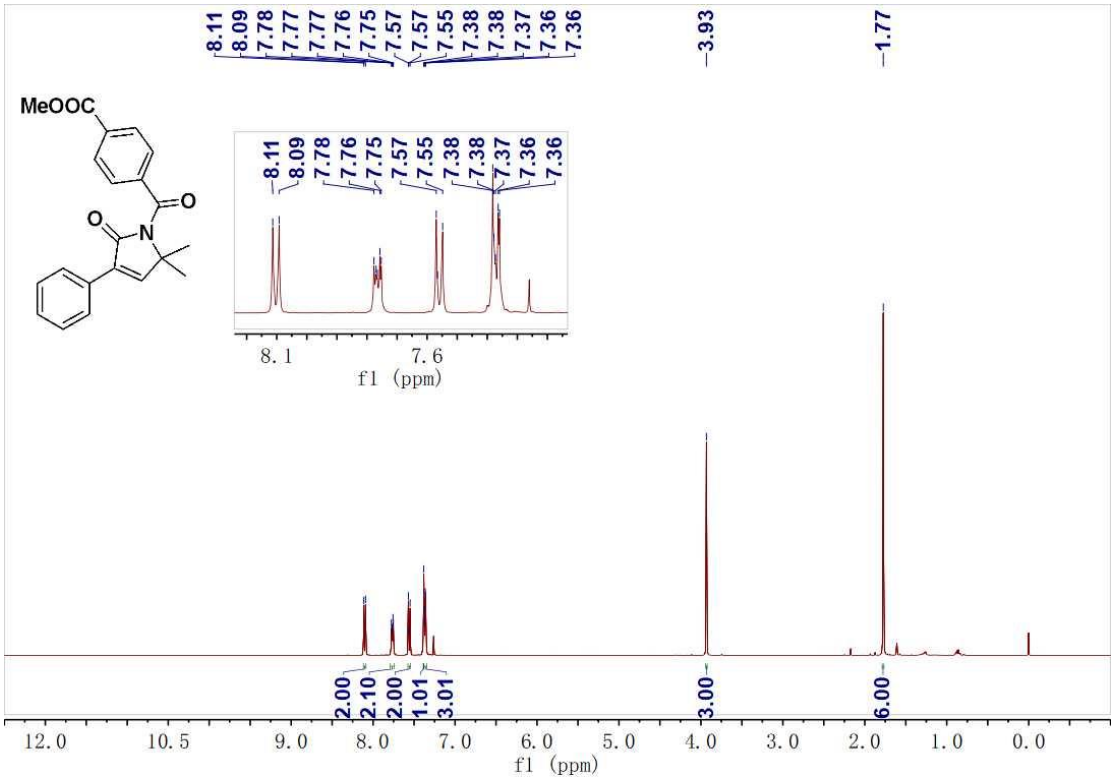

168.4
168.0
153.2
140.5
132.2
131.8
129.8
129.3
128.7
128.1
127.2
118.3
114.3
63.7
24.3
NC
O
O
N
168.2
f1 (ppm)
131
129
127
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

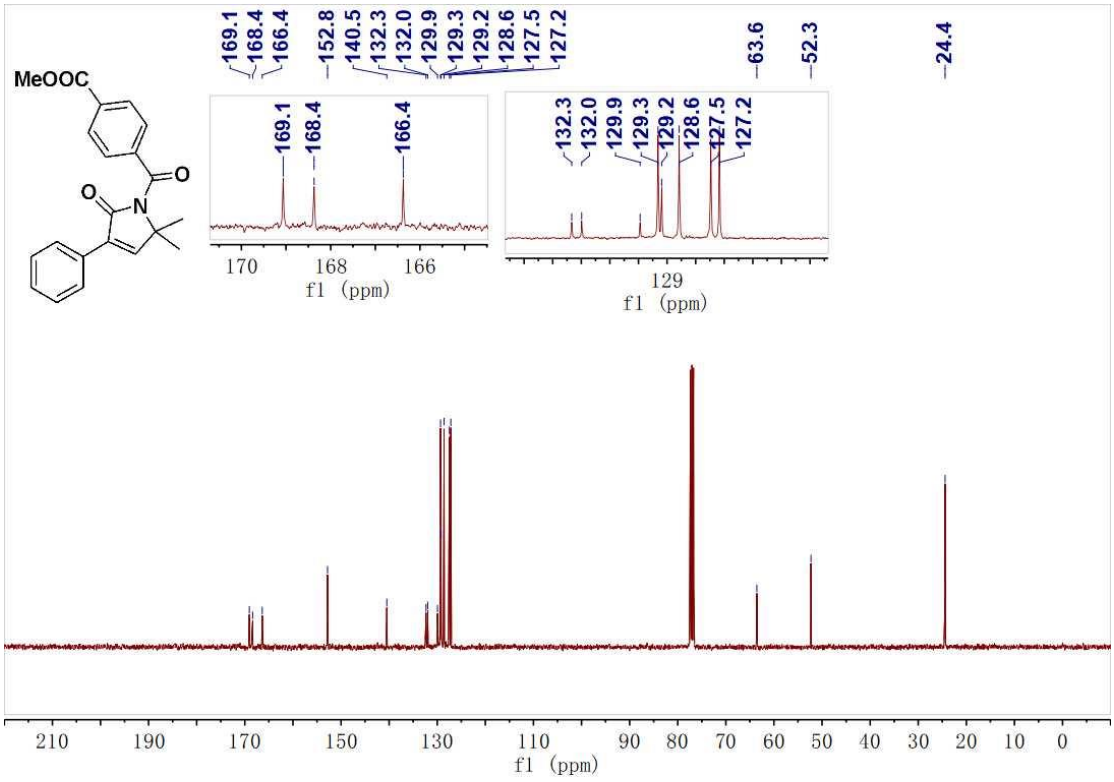
2m


8.09
8.09
7.84
7.66
7.46
7.44
2.71
1.86
f1 (ppm)
2.00
2.00
2.00
1.00
3.01
3.00
6.00

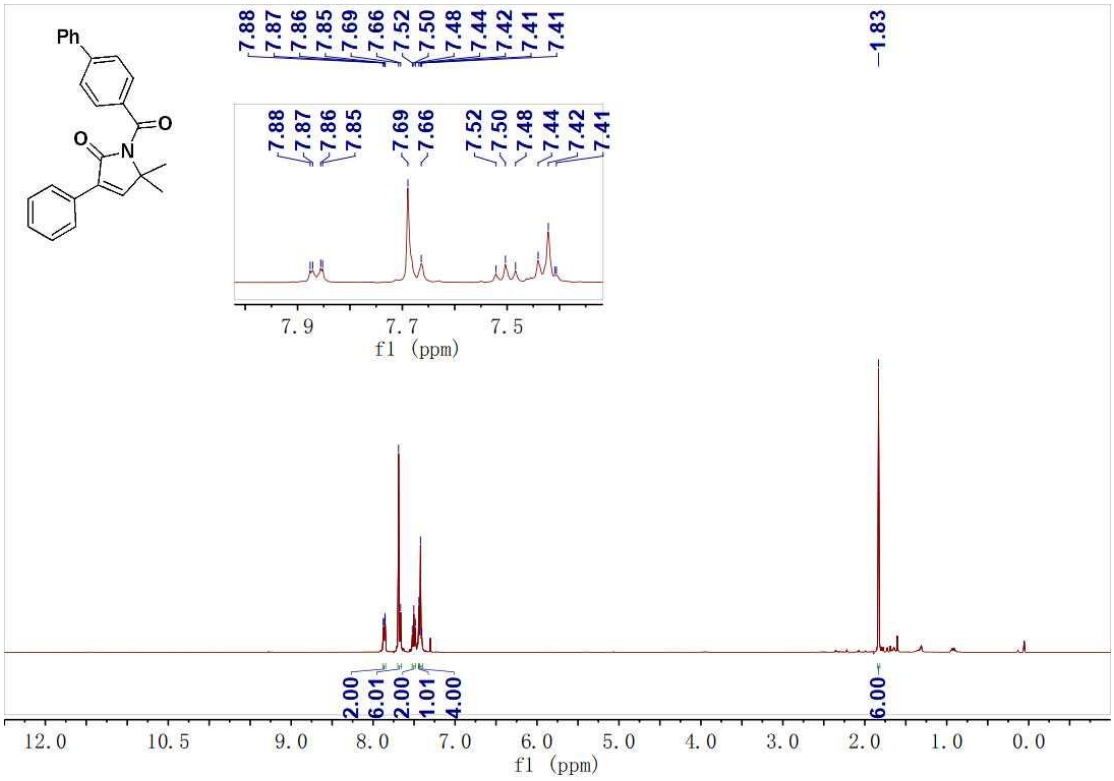

197.4
169.0
168.5
152.9
140.6
138.5
132.3
130.0
129.2
128.6
128.0
127.7
127.2
63.6
f1 (ppm)

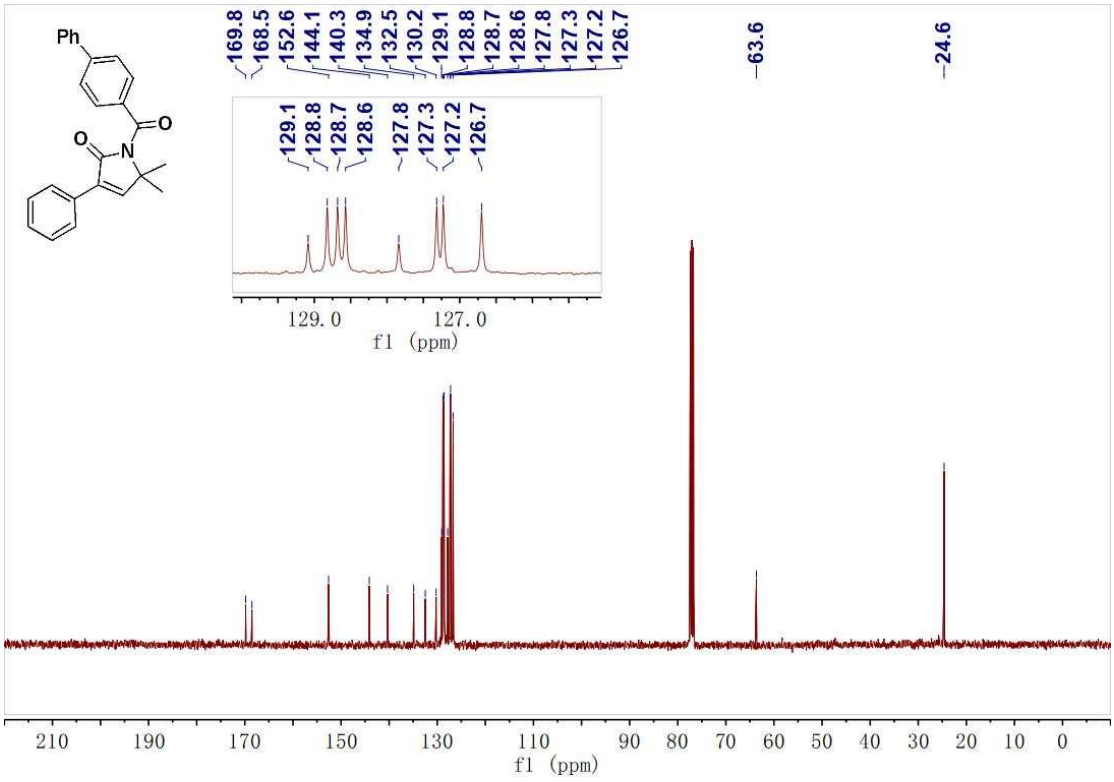
2n


8.11
8.09
7.78
7.77
7.77
7.76
7.75
7.57
7.57
7.55
7.38
7.38
7.37
7.36
7.36
3.93
1.77
MeOOC
O
O
N
8.1
7.6
f1 (ppm)
2.00
2.10
2.00
1.01
3.01
3.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

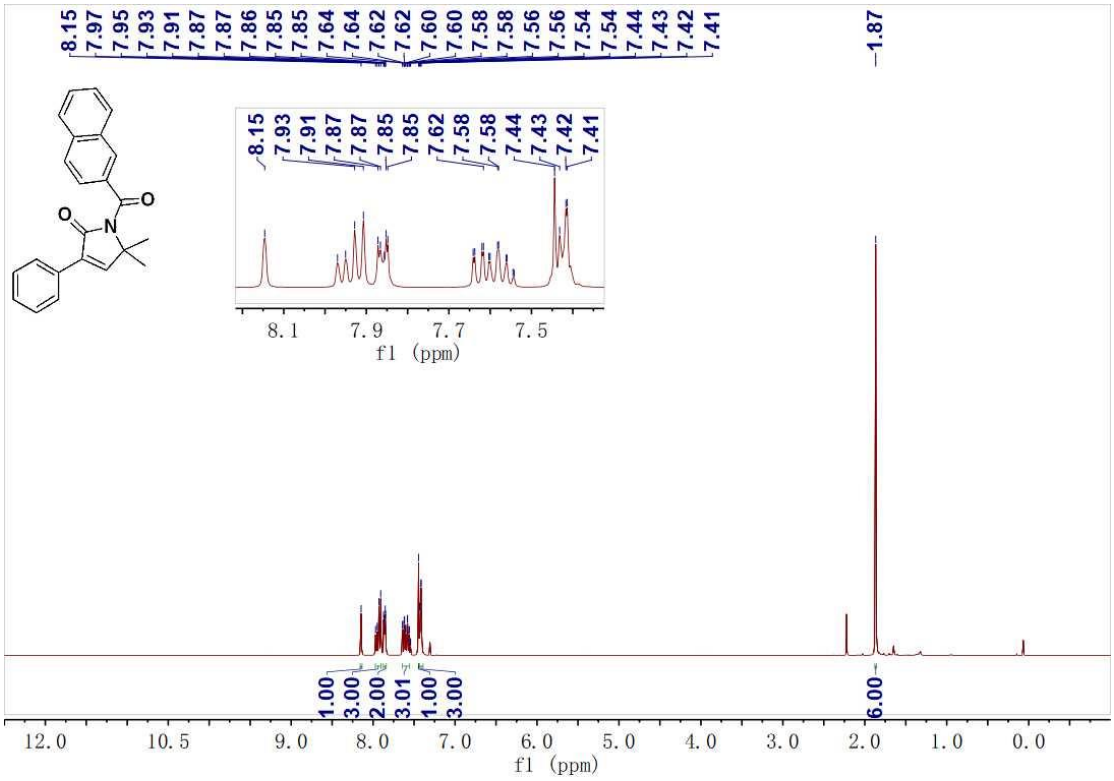

169.1
168.4
166.4
152.8
140.5
132.3
132.0
129.9
129.3
129.2
128.6
127.5
127.2
63.6
52.3
24.4
MeOOC
O
O
N
170
168
166
f1 (ppm)
132.3
132.0
129.9
129.3
129.2
128.6
127.5
127.2
129
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

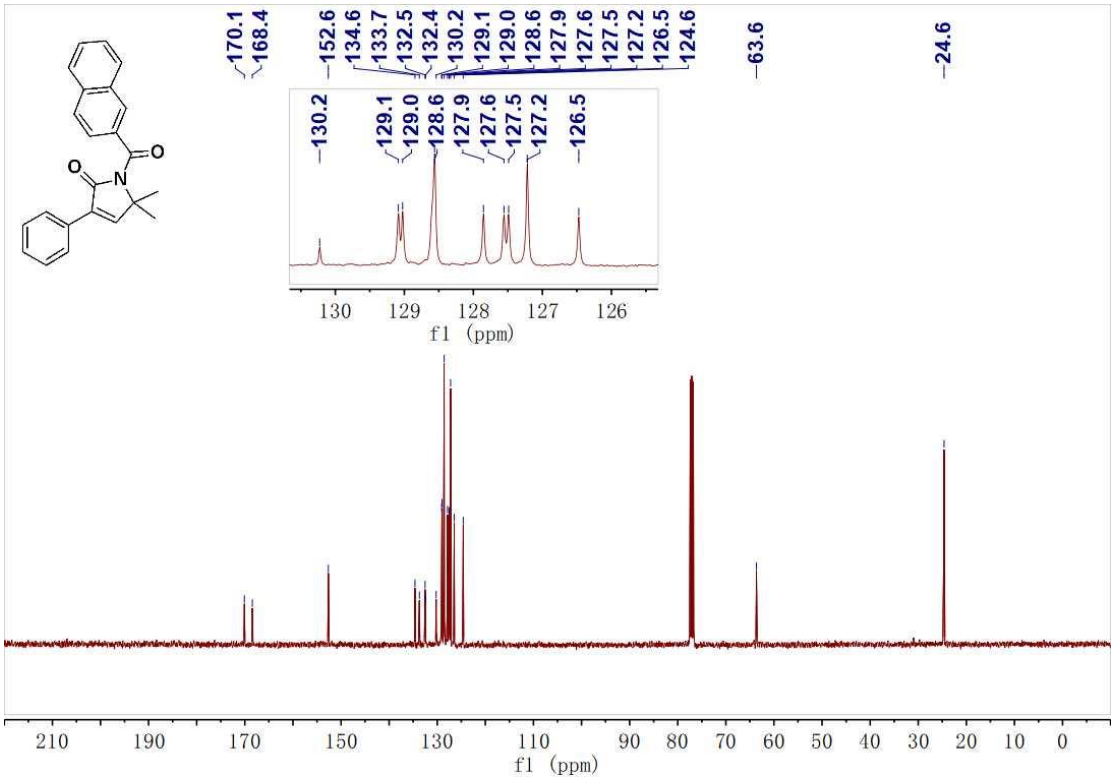
2o


Ph
O
O
N
7.88
7.87
7.86
7.85
7.69
7.66
7.52
7.50
7.48
7.44
7.42
7.41
7.41
1.83
7.9
7.7
7.5
f1 (ppm)
2.00
6.01
2.00
1.01
4.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

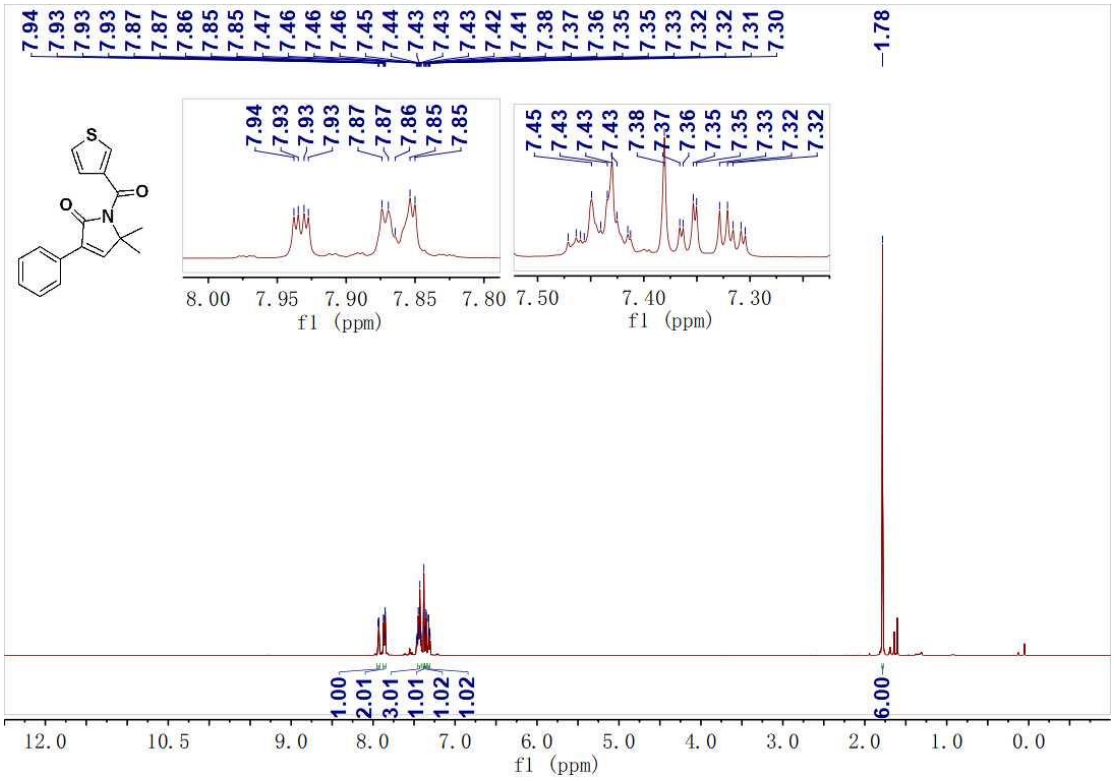

Ph
O
O
N
169.8
168.5
152.6
144.1
140.3
134.9
132.5
130.2
129.1
128.8
128.7
128.6
127.8
127.3
127.2
126.7
63.6
24.6
129.0
127.0
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

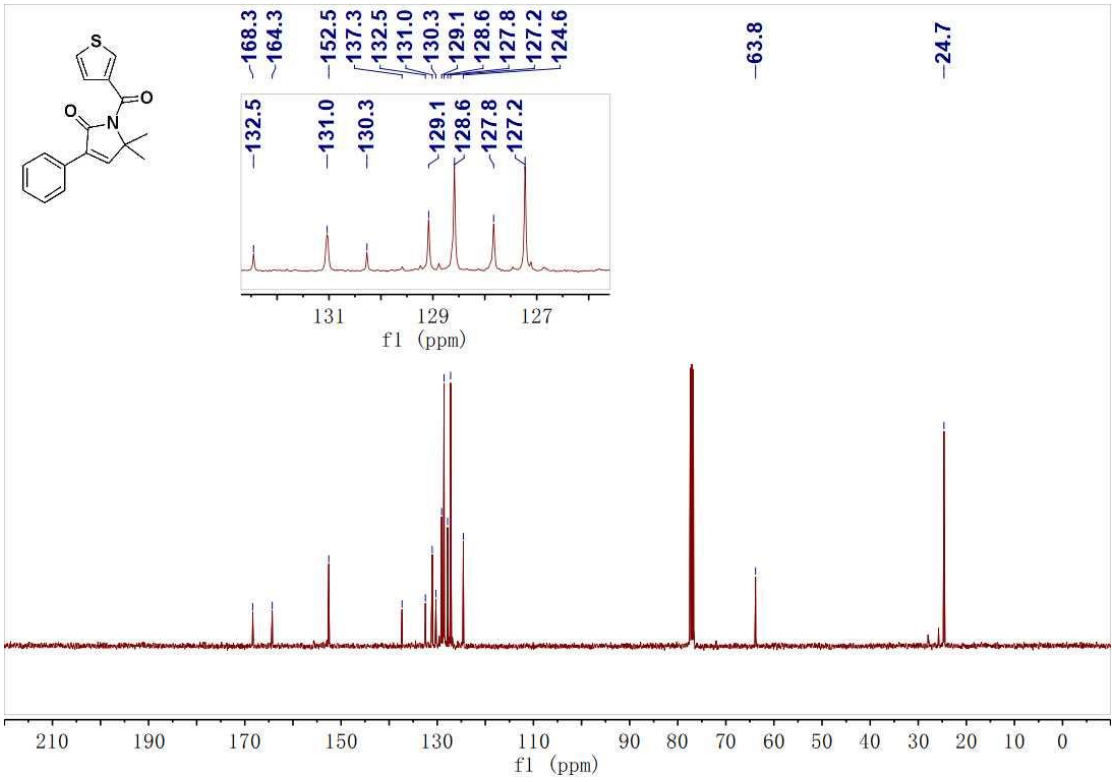
2p


8.15
7.97
7.95
7.93
7.91
7.87
7.87
7.86
7.85
7.85
7.64
7.64
7.62
7.62
7.60
7.60
7.58
7.58
7.56
7.56
7.54
7.54
7.44
7.43
7.42
7.41
1.87
O
O
N
8.1
7.9
7.7
7.5
f1 (ppm)
1.00
3.00
2.00
3.01
1.00
3.00
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

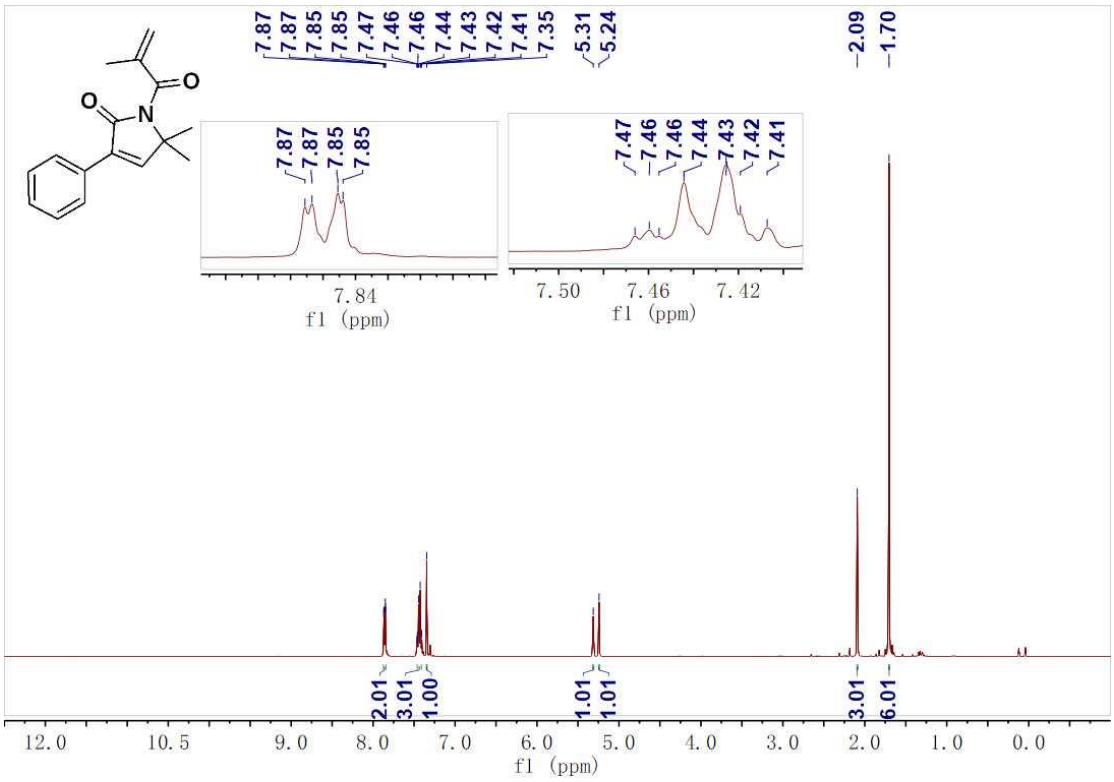

170.1
168.4
152.6
134.6
133.7
132.5
132.4
130.2
129.1
129.0
128.6
127.9
127.6
127.5
127.2
126.5
124.6
63.6
24.6
O
O
N
130
129
128
127
126
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

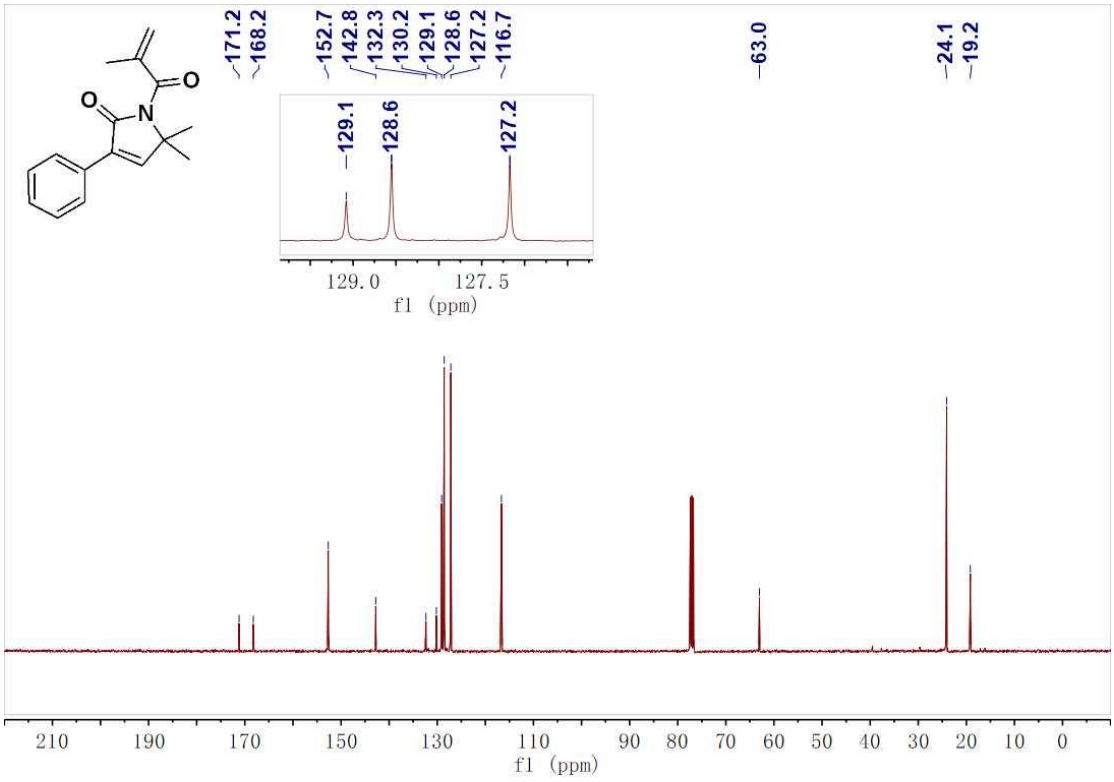
2q


7.94 7.93 7.93 7.87 7.87 7.86 7.85 7.85 7.47 7.46 7.46 7.46 7.45 7.44 7.43 7.43 7.42 7.41 7.38 7.37 7.36 7.35 7.35 7.33 7.32 7.32 7.31 7.30
1.78
1.00 2.01 3.01 1.01 1.02 1.02
6.00
f1 (ppm)

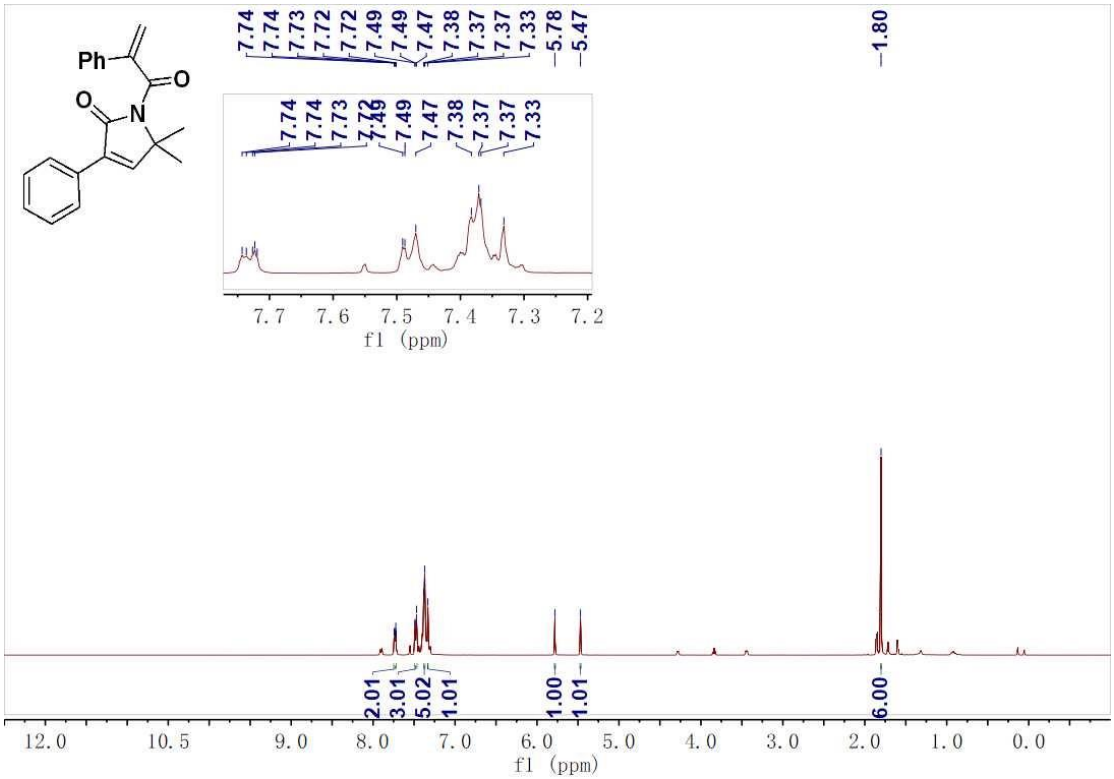

168.3 164.3 152.5 137.3 132.5 131.0 130.3 129.1 128.6 127.8 127.2 124.6
63.8
24.7
f1 (ppm)

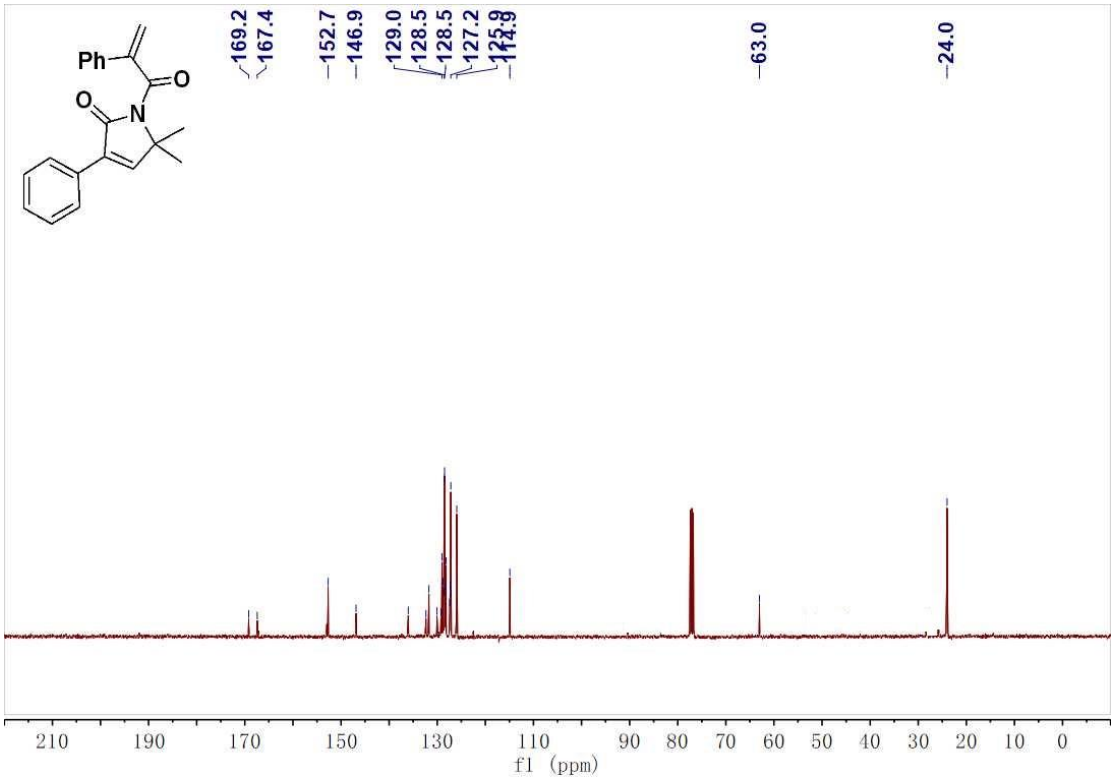
2r


7.87
7.87
7.85
7.85
7.47
7.46
7.46
7.44
7.43
7.42
7.41
7.35
5.31
5.24
2.09
1.70
7.84
f1 (ppm)
7.50
7.46
7.42
f1 (ppm)
2.01
3.01
1.00
1.01
1.01
3.01
6.01
f1 (ppm)

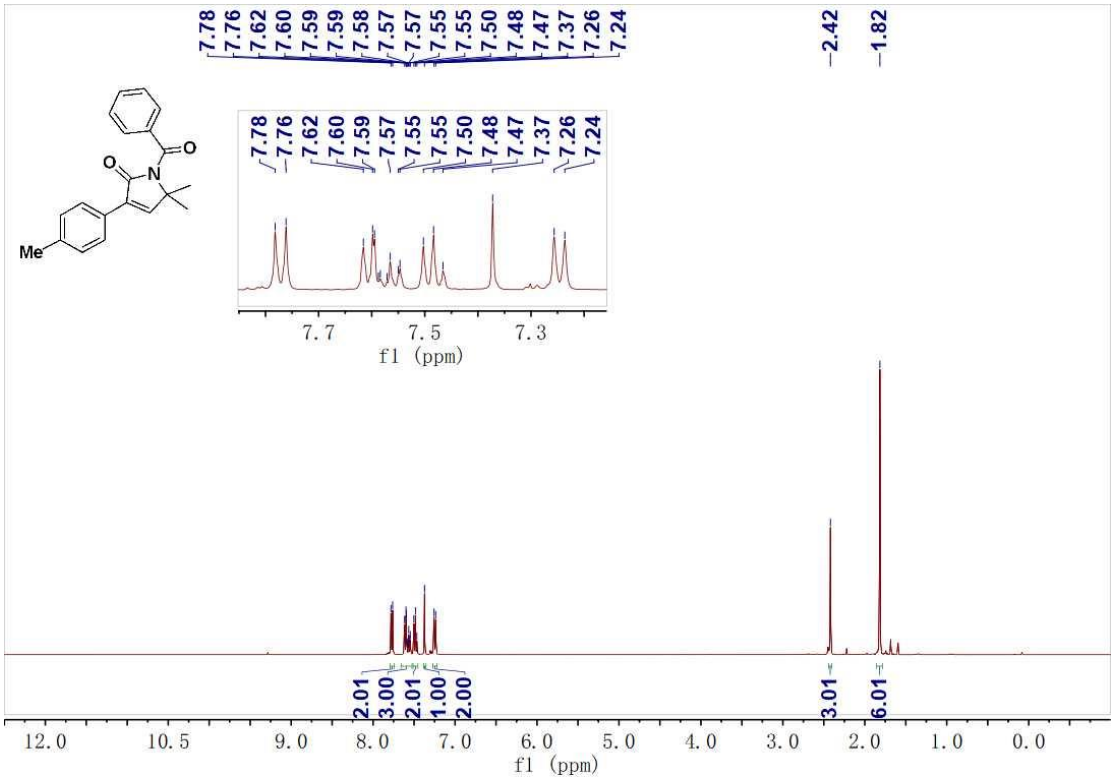

171.2
168.2
152.7
142.8
132.3
130.2
129.1
128.6
127.2
116.7
63.0
24.1
19.2
129.0
127.5
f1 (ppm)
f1 (ppm)

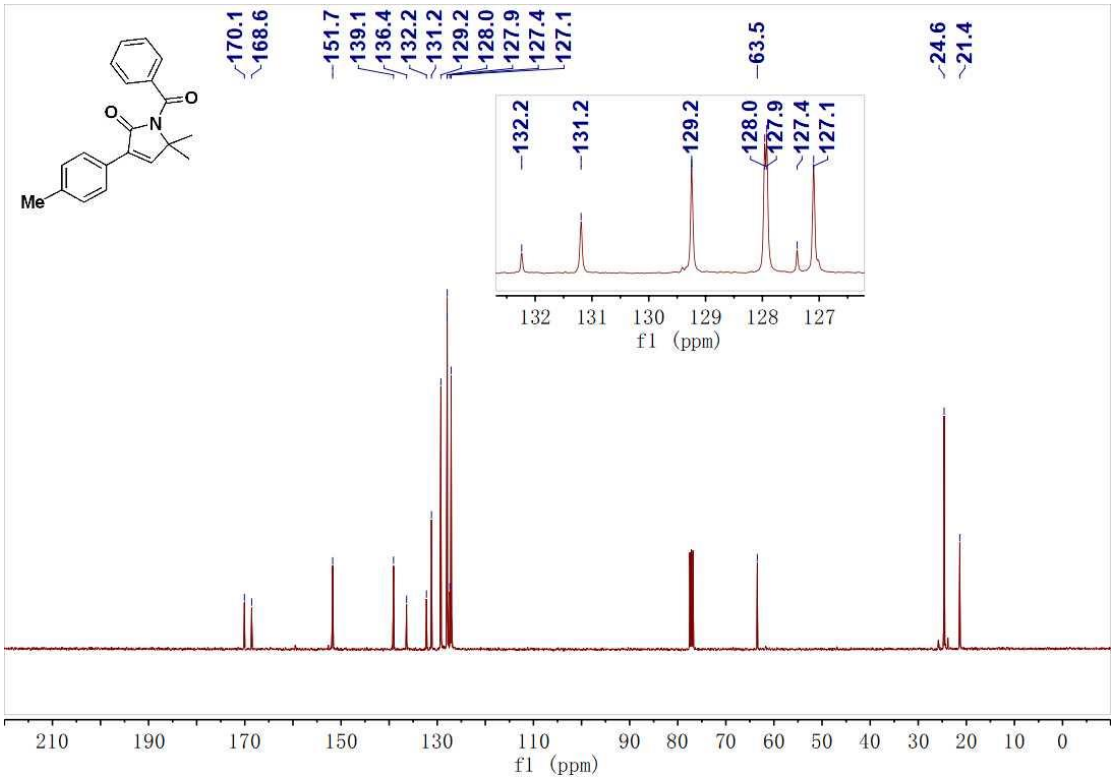
2s


Ph
O
O
N
7.74
7.74
7.73
7.72
7.72
7.49
7.49
7.47
7.38
7.37
7.37
7.33
5.78
5.47
1.80
7.7
7.6
7.5
7.4
7.3
7.2
f1 (ppm)
2.01
3.01
5.02
1.01
1.00
1.01
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

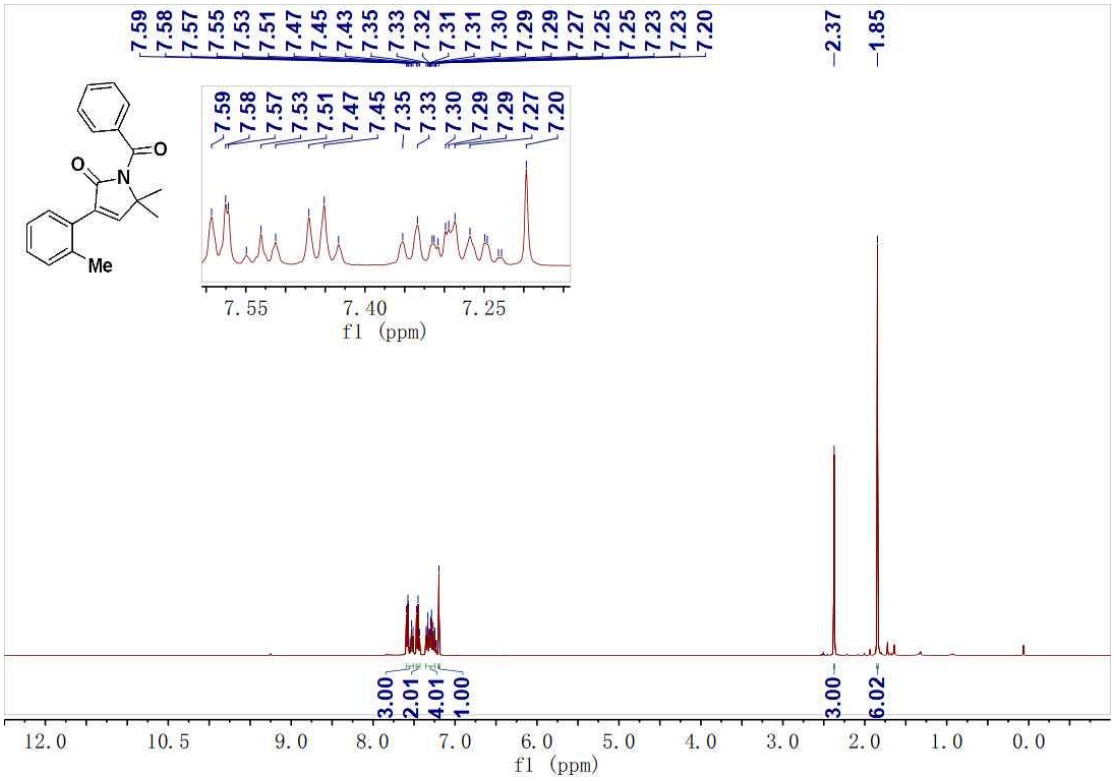

Ph
O
O
N
169.2
167.4
152.7
146.9
129.0
128.5
128.5
127.2
63.0
24.0
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

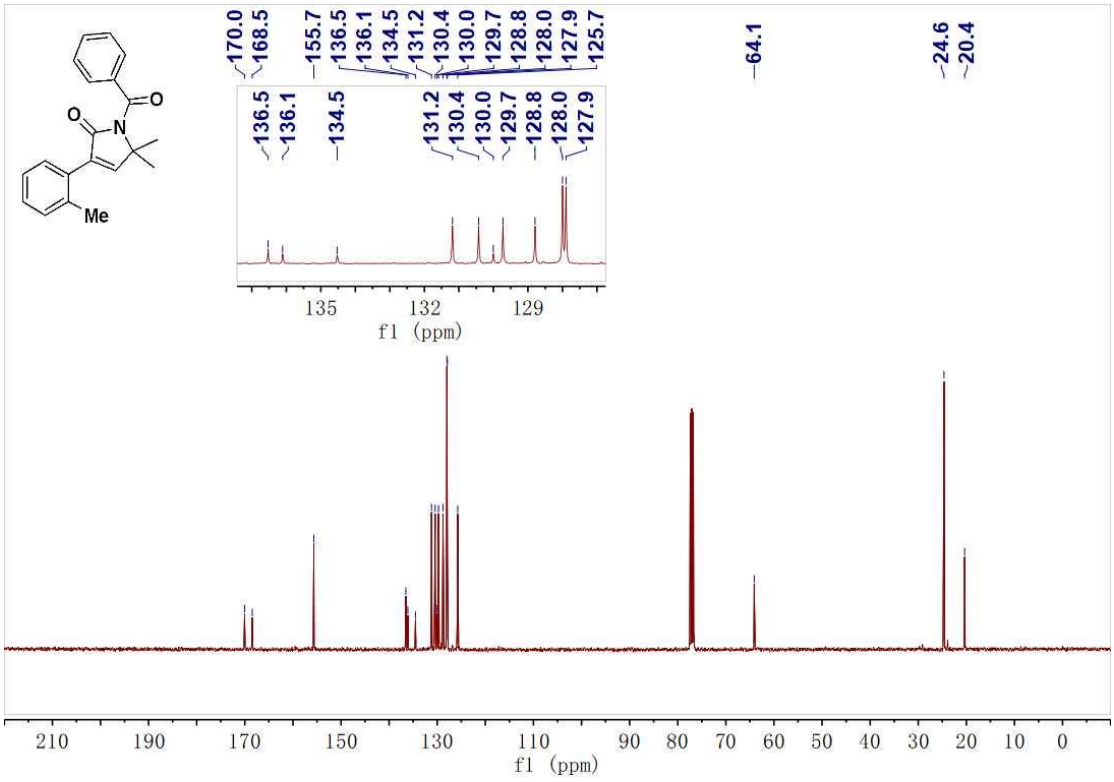
2aa


7.78
7.76
7.62
7.60
7.59
7.59
7.58
7.57
7.57
7.55
7.55
7.50
7.48
7.47
7.37
7.26
7.24
2.42
1.82
Me
O
O
N
7.7
7.5
7.3
f1 (ppm)
2.01
3.00
2.01
1.00
2.00
3.01
6.01
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

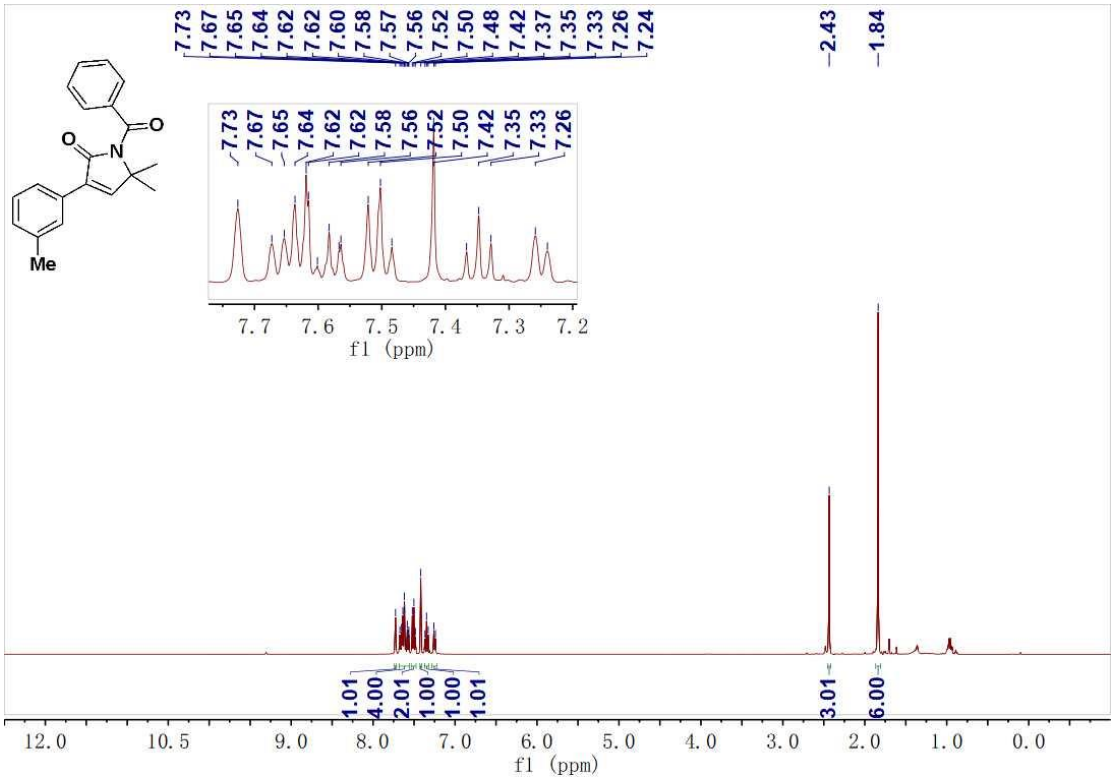

170.1
168.6
151.7
139.1
136.4
132.2
131.2
129.2
128.0
127.9
127.4
127.1
63.5
24.6
21.4
132.2
131.2
129.2
128.0
127.9
127.4
127.1
Me
O
O
N
132
131
130
129
128
127
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

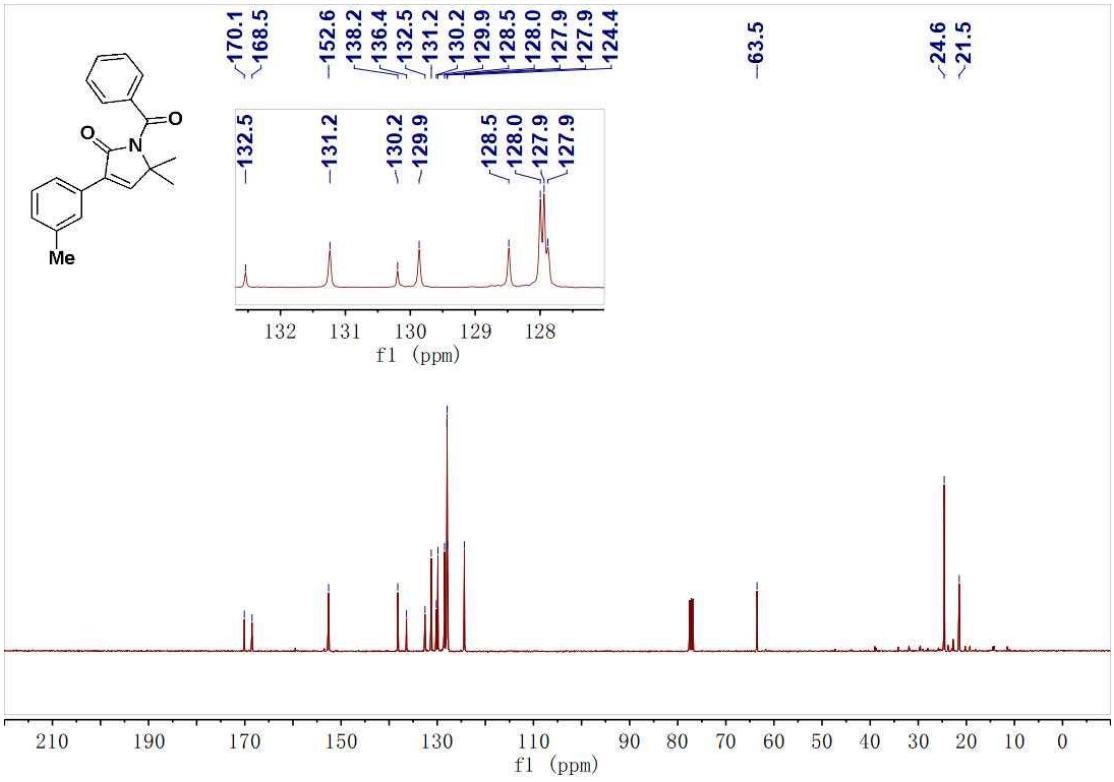
2ab


7.59 7.58 7.57 7.55 7.53 7.51 7.47 7.45 7.43 7.35 7.33 7.32 7.31 7.31 7.30 7.29 7.29 7.27 7.25 7.25 7.23 7.23 7.20
2.37
1.85
O
O
N
Me
7.59 7.58 7.57 7.53 7.51 7.47 7.45 7.35 7.33 7.30 7.29 7.29 7.27 7.20
7.55 7.40 7.25
f1 (ppm)
3.00 2.01 4.01 1.00
3.00 6.02
12.0 10.5 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

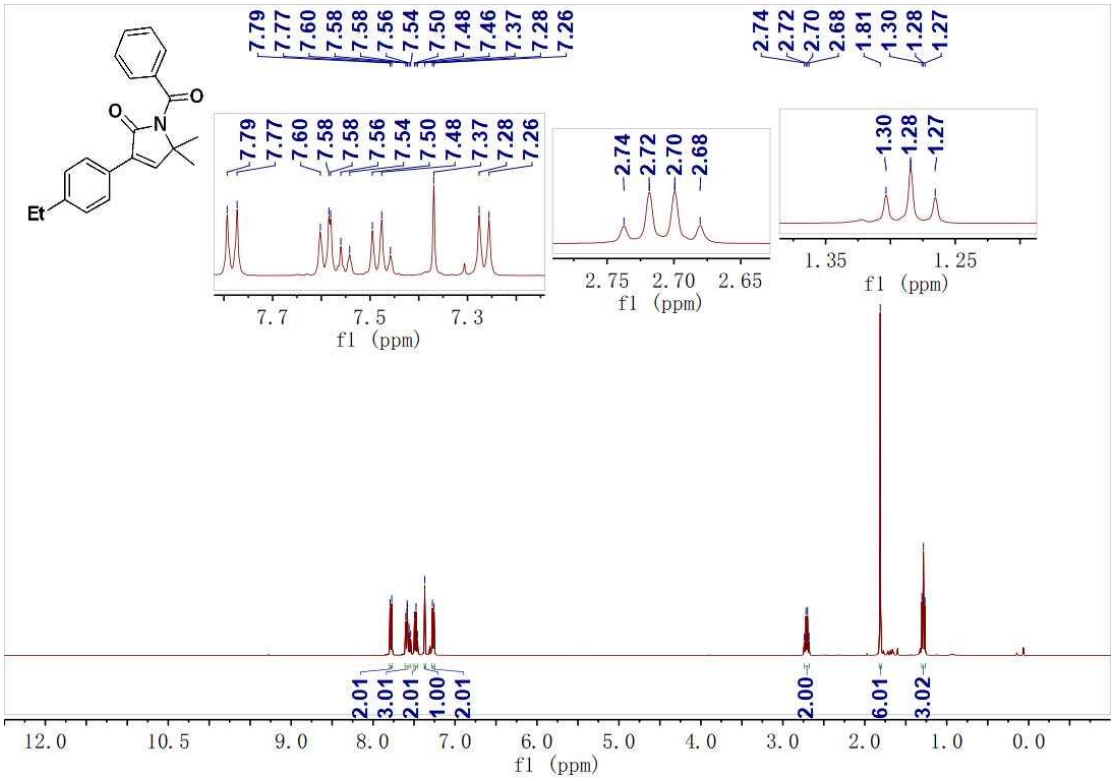

170.0 168.5 155.7 136.5 136.1 134.5 131.2 130.4 130.0 129.7 128.8 128.0 127.9 125.7
64.1
24.6 20.4
O
O
N
Me
136.5 136.1 134.5 131.2 130.4 130.0 129.7 128.8 128.0 127.9
135 132 129
f1 (ppm)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

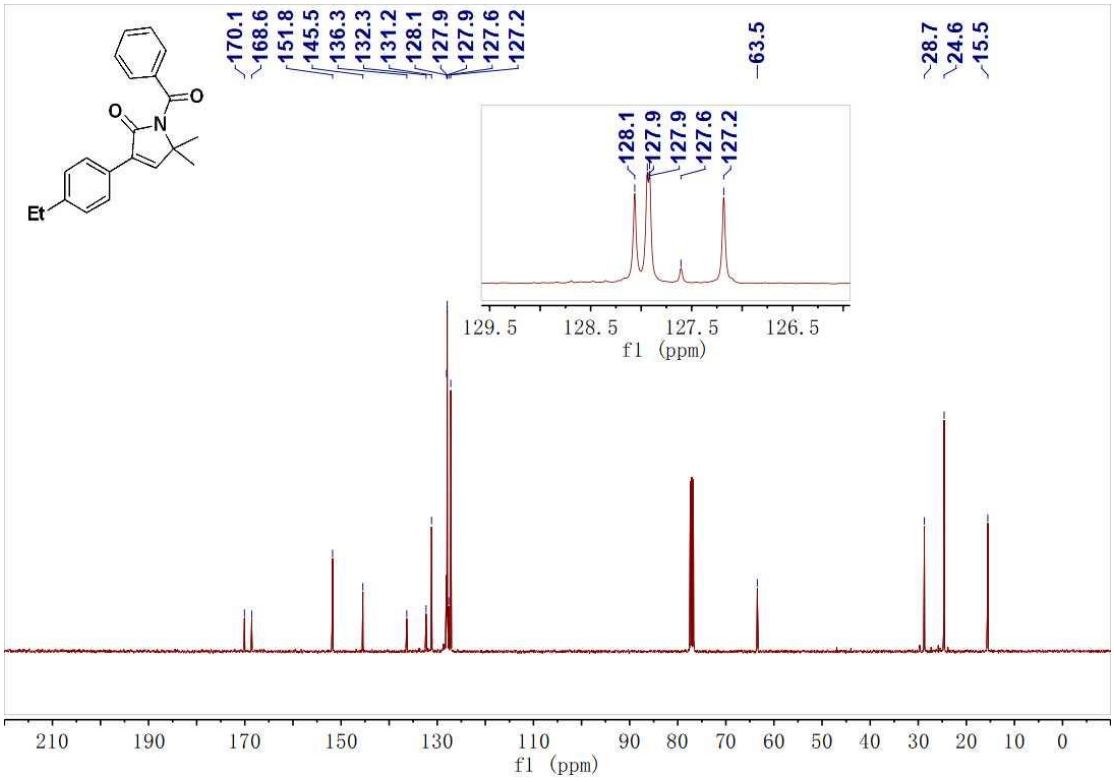
2ac


7.73
7.67
7.65
7.64
7.62
7.62
7.60
7.58
7.57
7.56
7.52
7.50
7.48
7.42
7.37
7.35
7.33
7.26
7.24
2.43
1.84
Me
f1 (ppm)
1.01
4.00
2.01
1.00
1.00
1.01
3.01
6.00

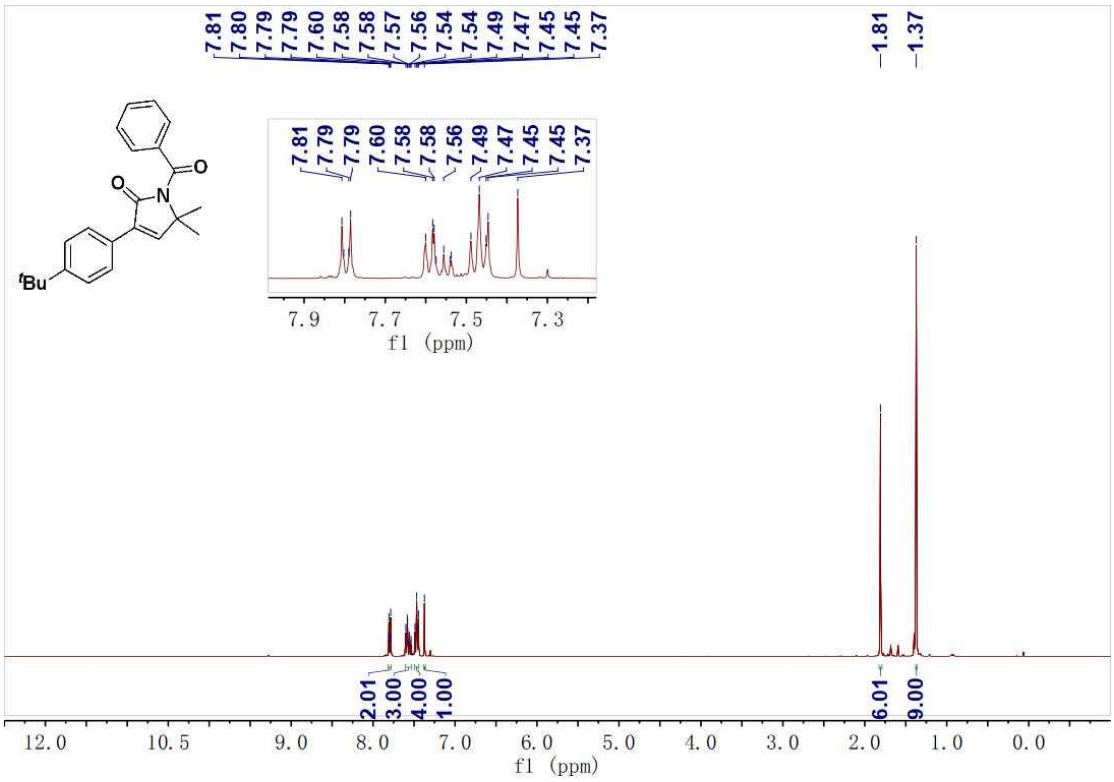

170.1
168.5
152.6
138.2
136.4
132.5
131.2
130.2
129.9
128.5
128.0
127.9
127.9
124.4
63.5
24.6
21.5
Me
f1 (ppm)

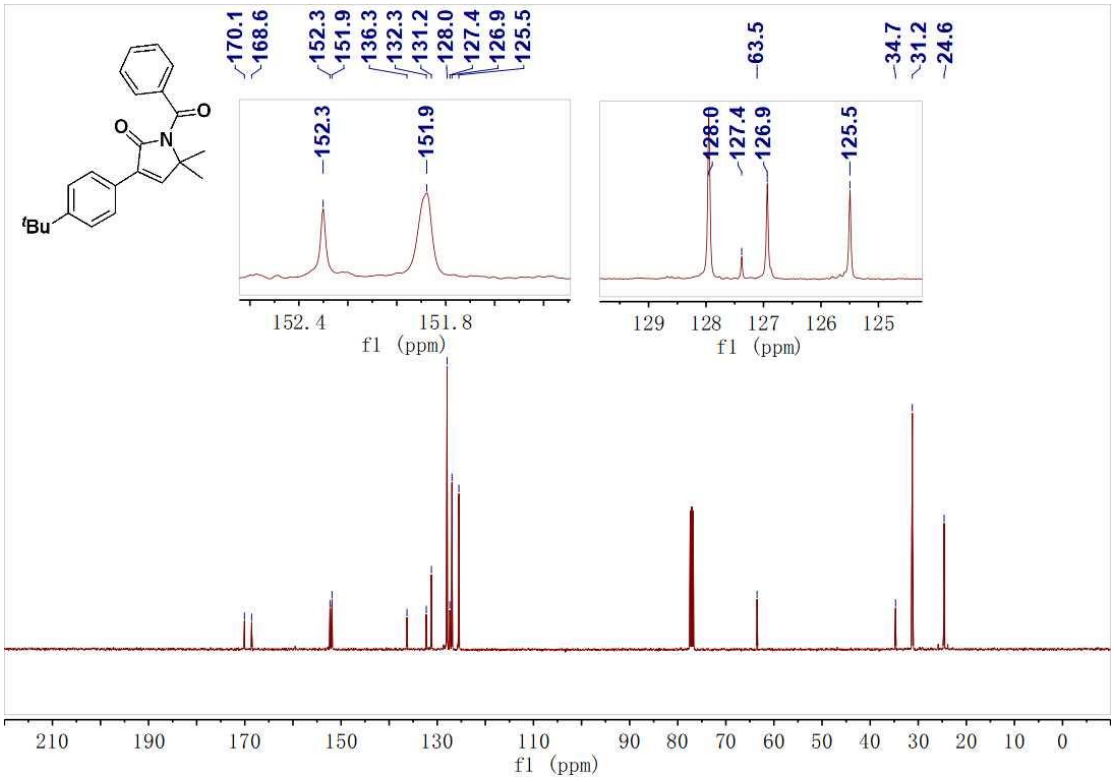
2ad


7.79 7.77 7.60 7.58 7.58 7.56 7.54 7.50 7.48 7.46 7.37 7.28 7.26
2.74 2.72 2.70 2.68 1.81 1.30 1.28 1.27
Et
f1 (ppm)
2.01 3.01 2.01 1.00 2.01 2.00 6.01 3.02

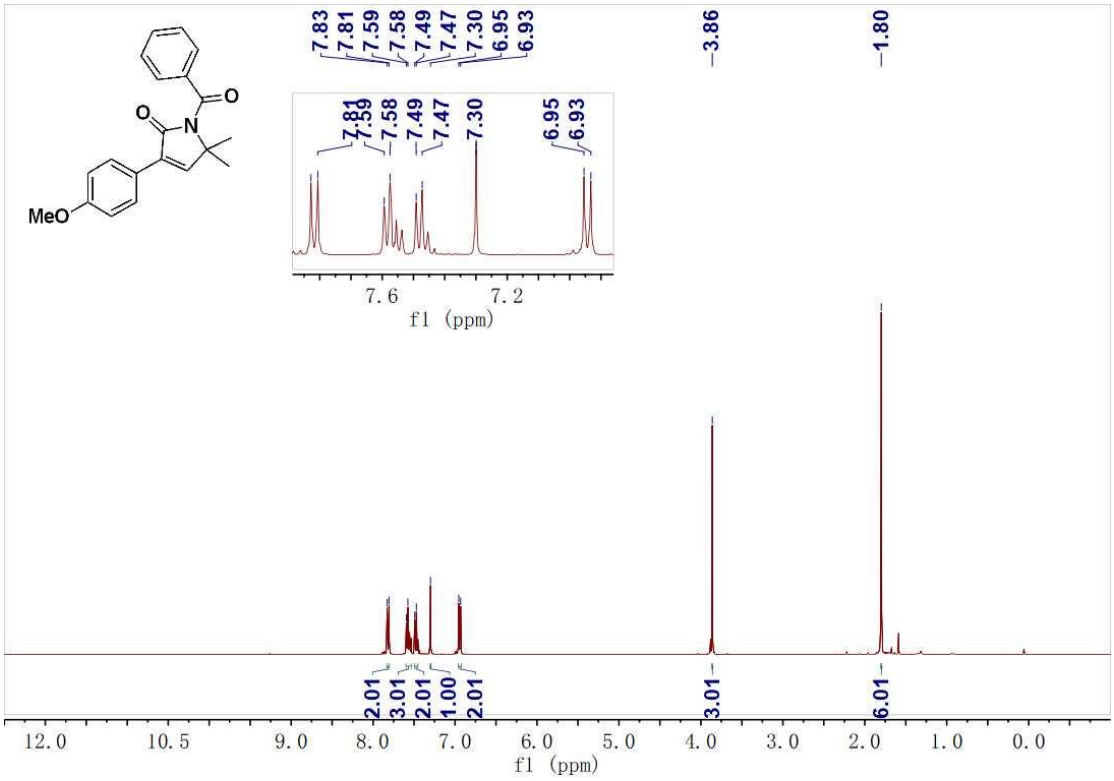

170.1 168.6 151.8 145.5 136.3 132.3 131.2 128.1 127.9 127.9 127.6 127.2
63.5
28.7 24.6 15.5
Et
f1 (ppm)

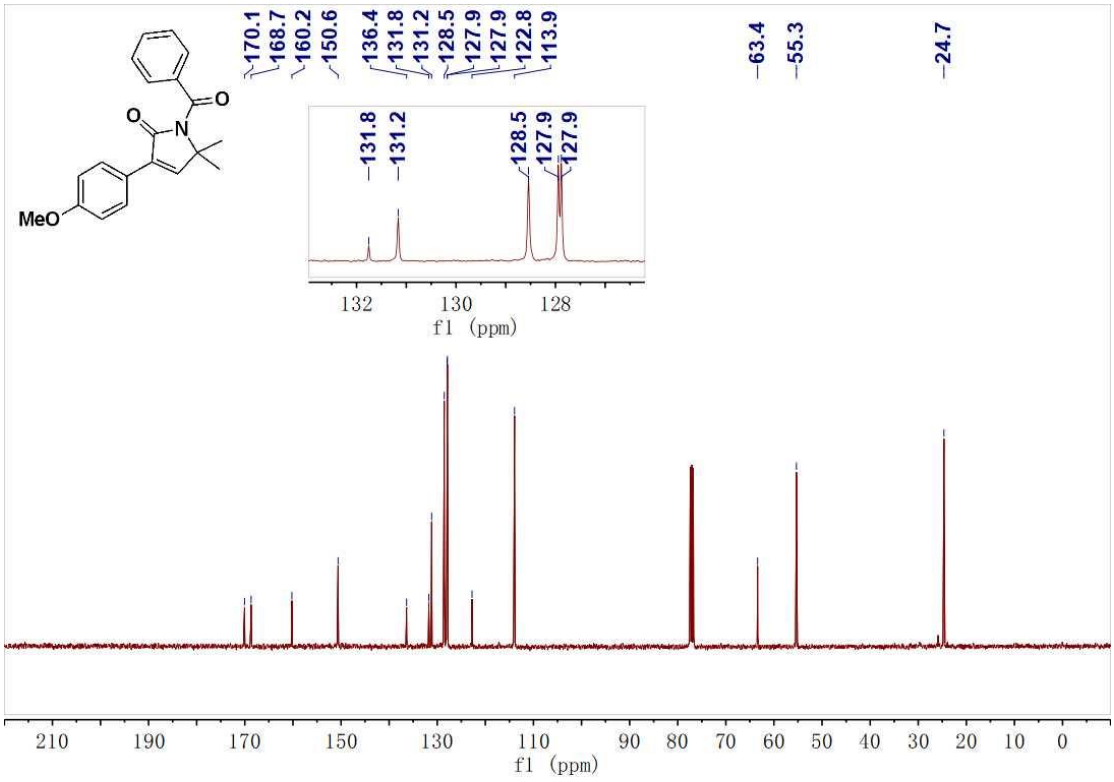
2ae


7.81
7.80
7.79
7.79
7.60
7.58
7.58
7.57
7.56
7.54
7.54
7.49
7.47
7.45
7.45
7.37
1.81
1.37
tBu
f1 (ppm)
2.01
3.00
4.00
1.00
6.01
9.00

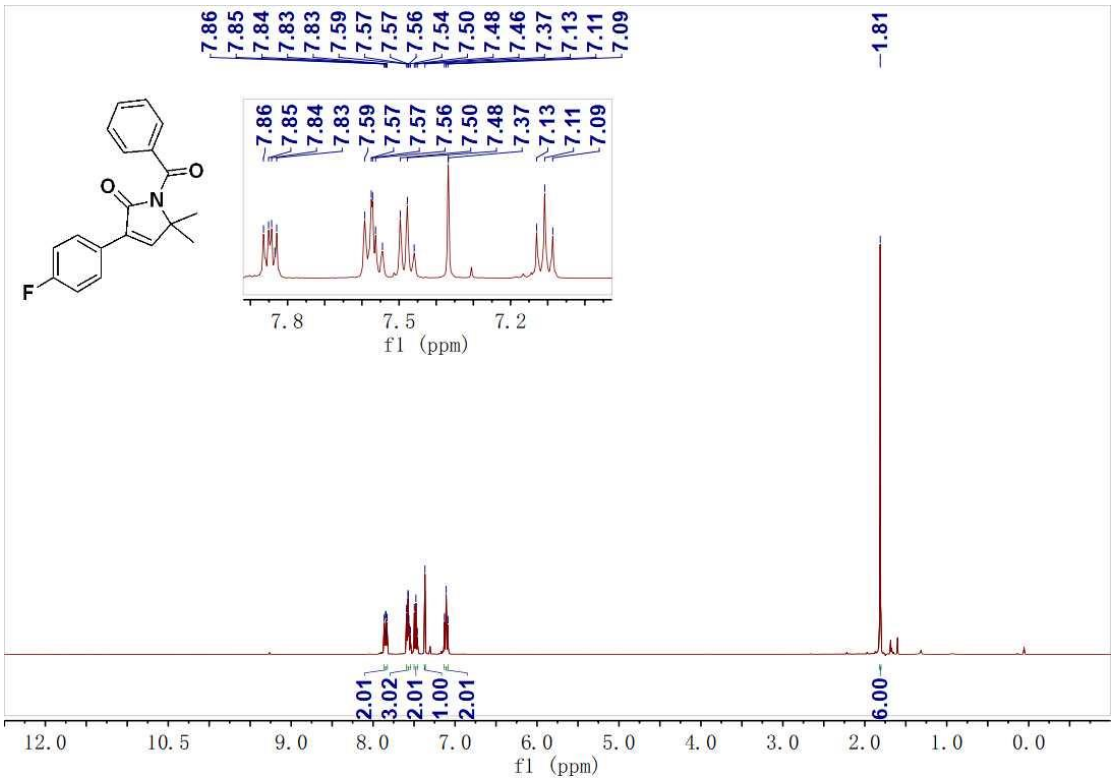

170.1
168.6
152.3
151.9
136.3
132.3
131.2
128.0
127.4
126.9
125.5
63.5
34.7
31.2
24.6
tBu
f1 (ppm)

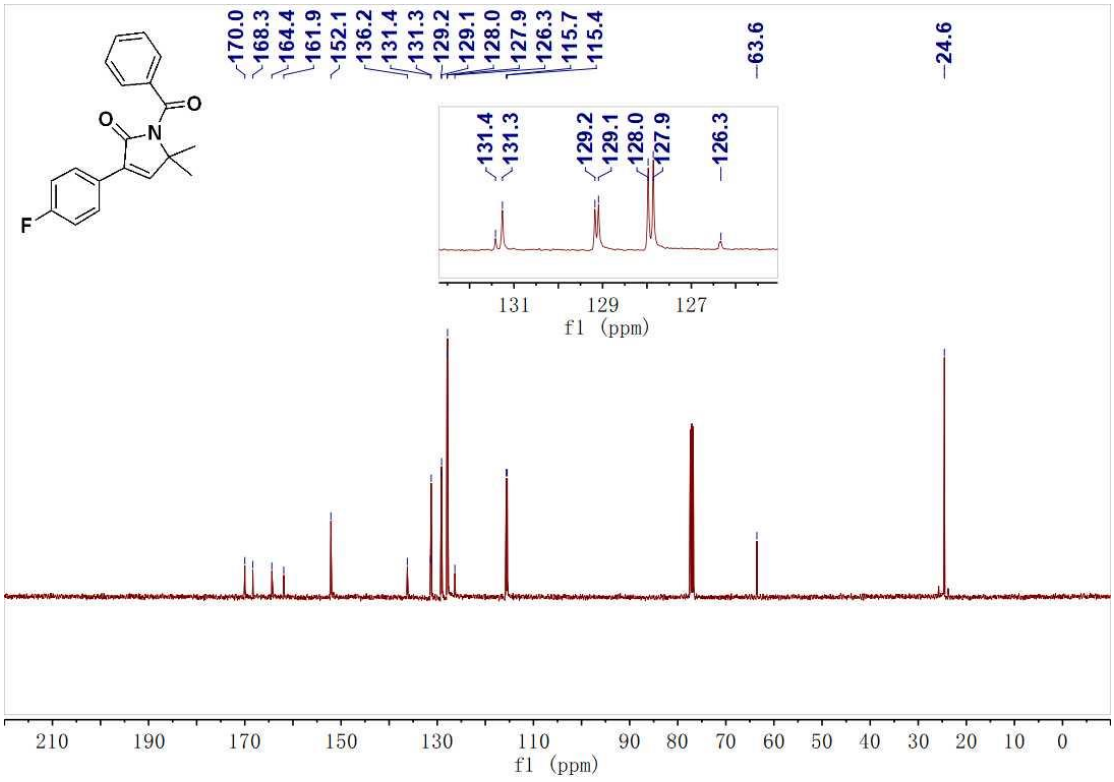
2af


7.83
7.81
7.59
7.58
7.49
7.47
7.30
6.95
6.93
3.86
1.80
MeO
f1 (ppm)
2.01
3.01
2.01
1.00
2.01
3.01
6.01

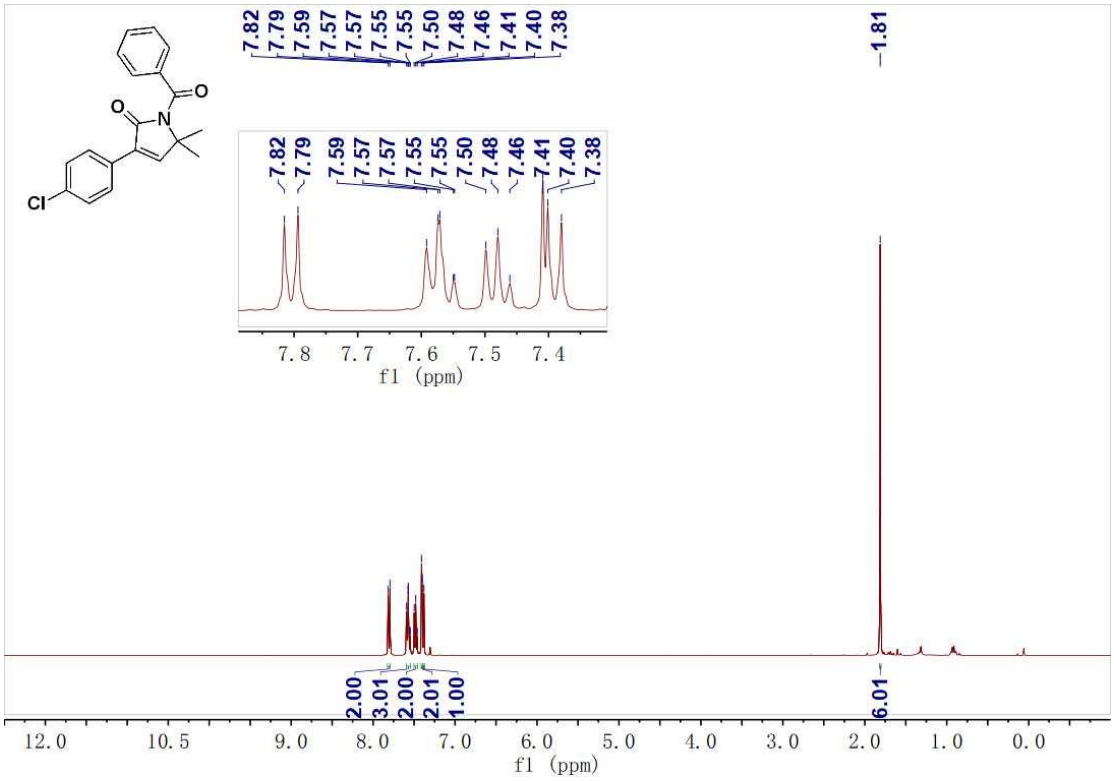

170.1
168.7
160.2
150.6
136.4
131.8
131.2
128.5
127.9
127.9
122.8
113.9
63.4
55.3
24.7
MeO
f1 (ppm)

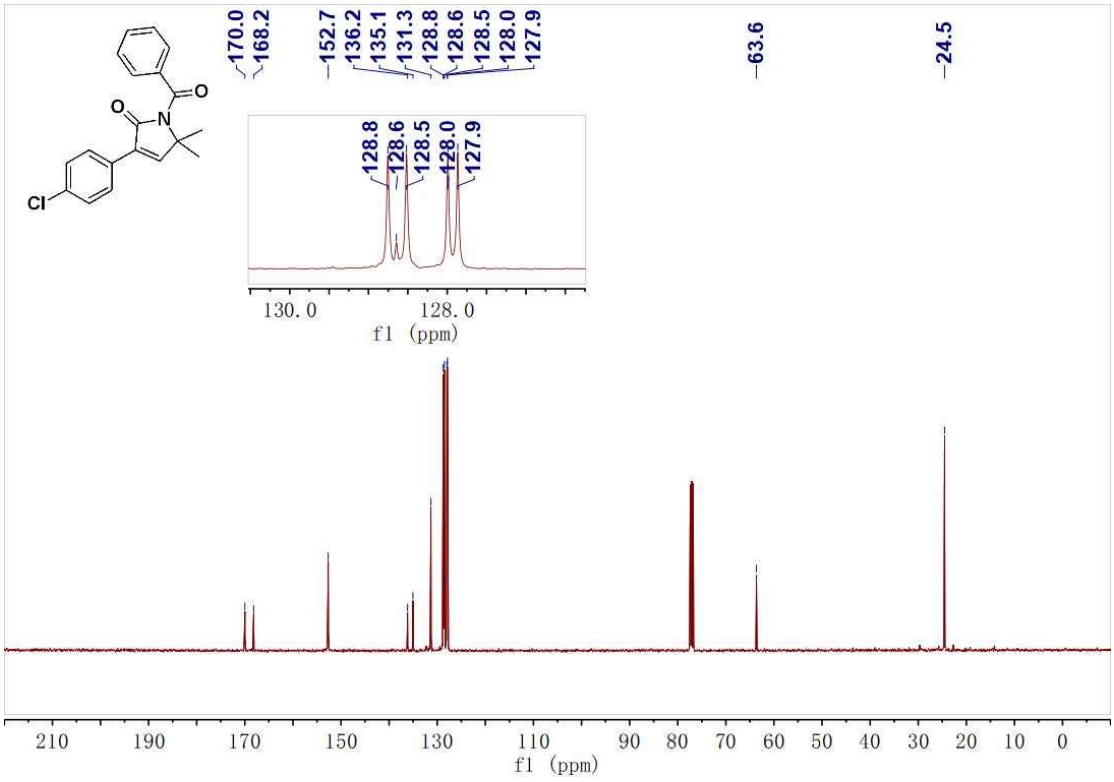
2ag


7.86
7.85
7.84
7.83
7.83
7.59
7.57
7.57
7.56
7.54
7.50
7.48
7.46
7.37
7.13
7.11
7.09
1.81
O
O
N
F
7.8
7.5
7.2
f1 (ppm)
2.01
3.02
2.01
1.00
2.01
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

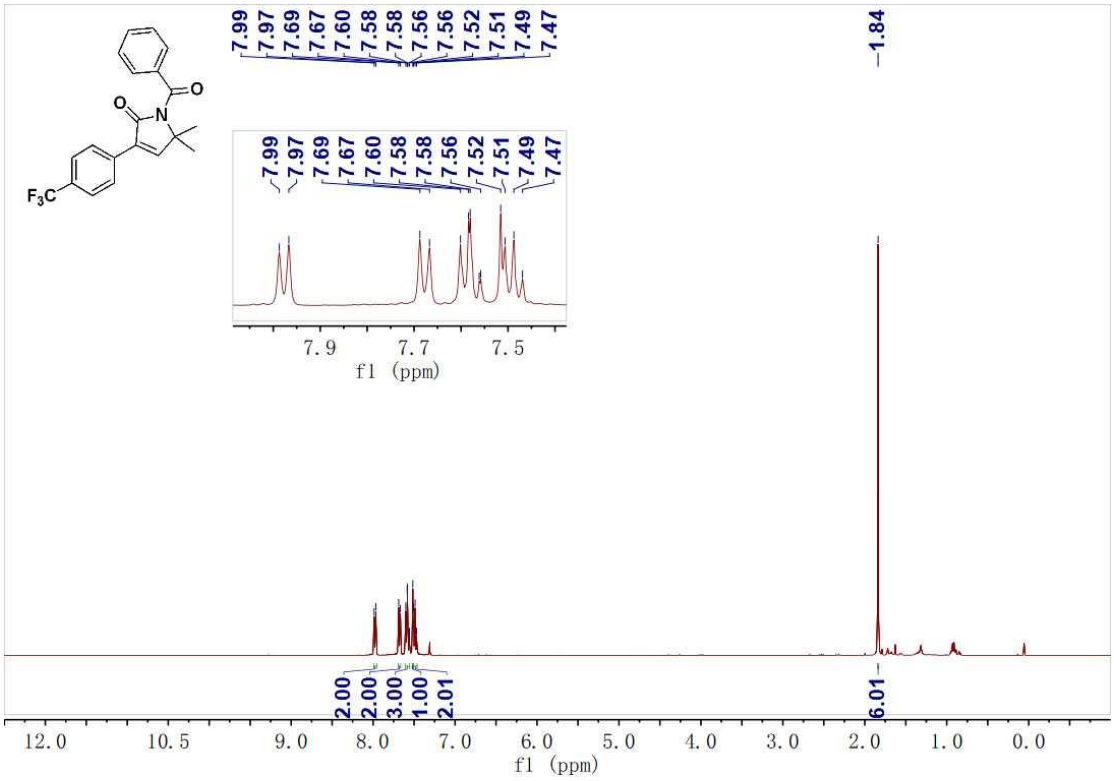

170.0
168.3
164.4
161.9
152.1
136.2
131.4
131.3
129.2
129.1
128.0
127.9
126.3
115.7
115.4
63.6
24.6
O
O
N
F
131.4
131.3
129.2
129.1
128.0
127.9
126.3
131
129
127
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

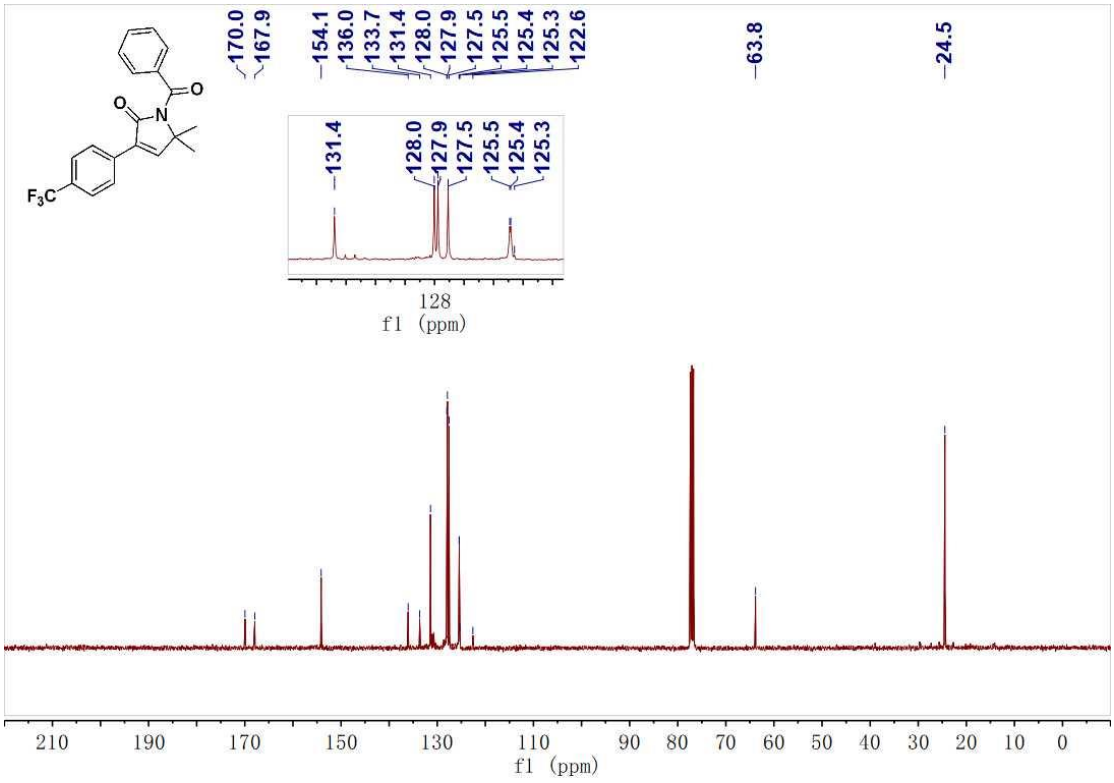
2ah


7.82
7.79
7.59
7.57
7.57
7.55
7.55
7.50
7.48
7.46
7.41
7.40
7.38
1.81
O
N
O
Cl
7.8
7.7
7.6
7.5
7.4
f1 (ppm)
2.00
3.01
2.00
2.01
1.00
6.01
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

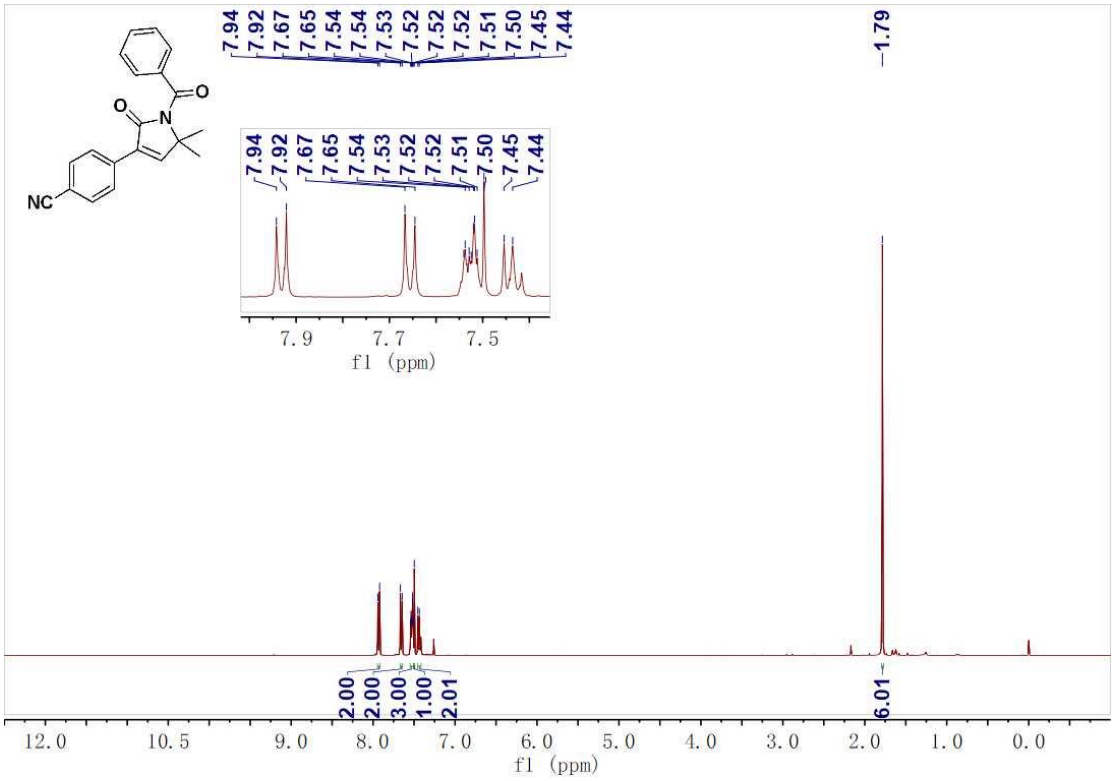

170.0
168.2
152.7
136.2
135.1
131.3
128.8
128.6
128.5
128.0
127.9
63.6
24.5
O
N
O
Cl
130.0
128.0
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

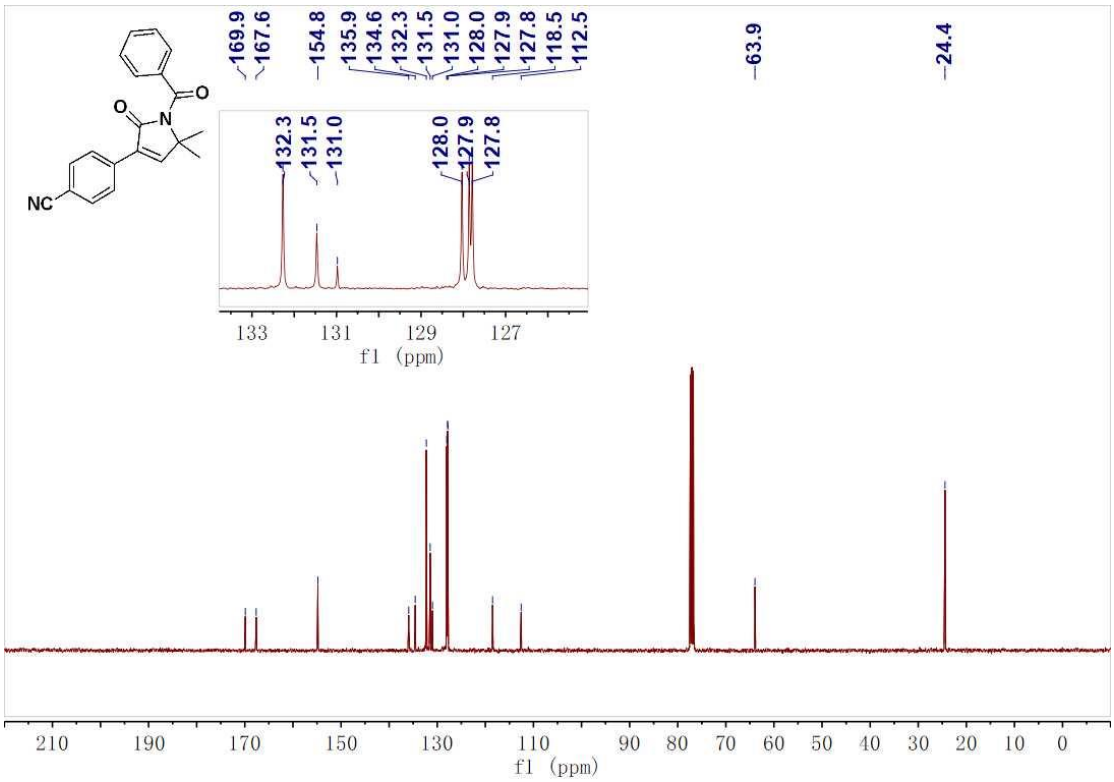
2ai


7.99
7.97
7.69
7.67
7.60
7.58
7.58
7.56
7.56
7.52
7.51
7.49
7.47
1.84
F3C
O
N
7.9
7.7
7.5
f1 (ppm)
2.00
2.00
3.00
1.00
2.01
6.01
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

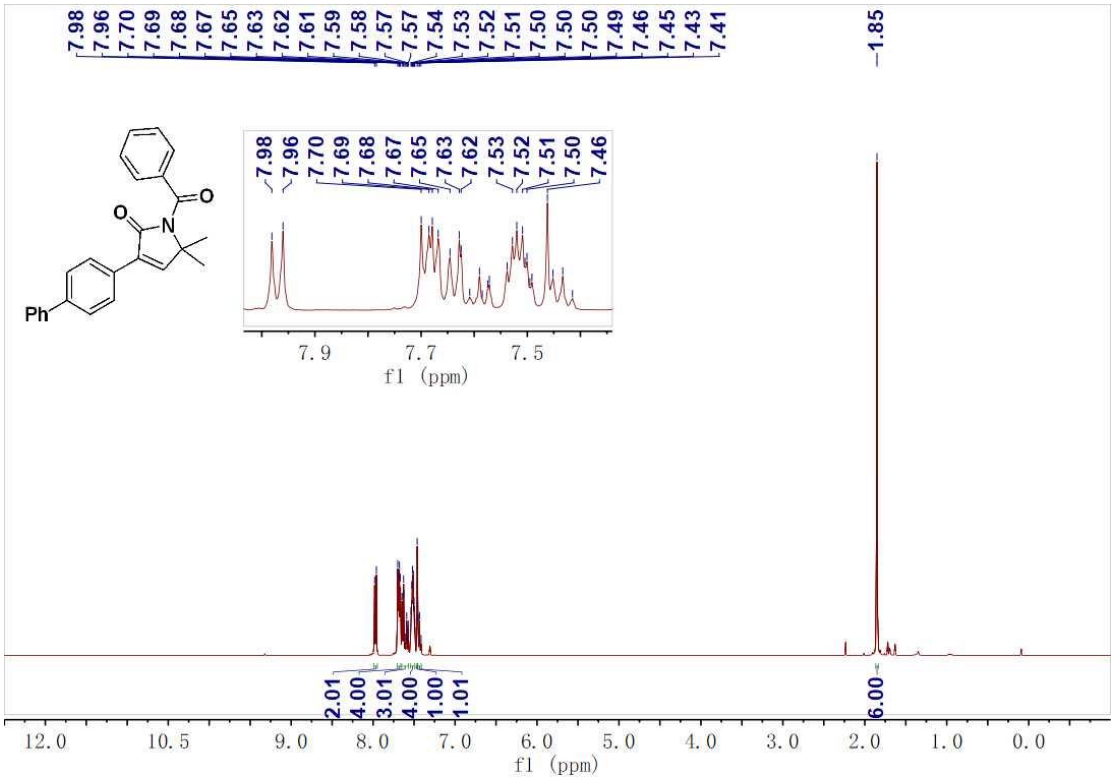

170.0
167.9
154.1
136.0
133.7
131.4
128.0
127.9
127.5
125.5
125.4
125.3
122.6
63.8
24.5
F3C
O
N
131.4
128.0
127.9
127.5
125.5
125.4
125.3
128
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

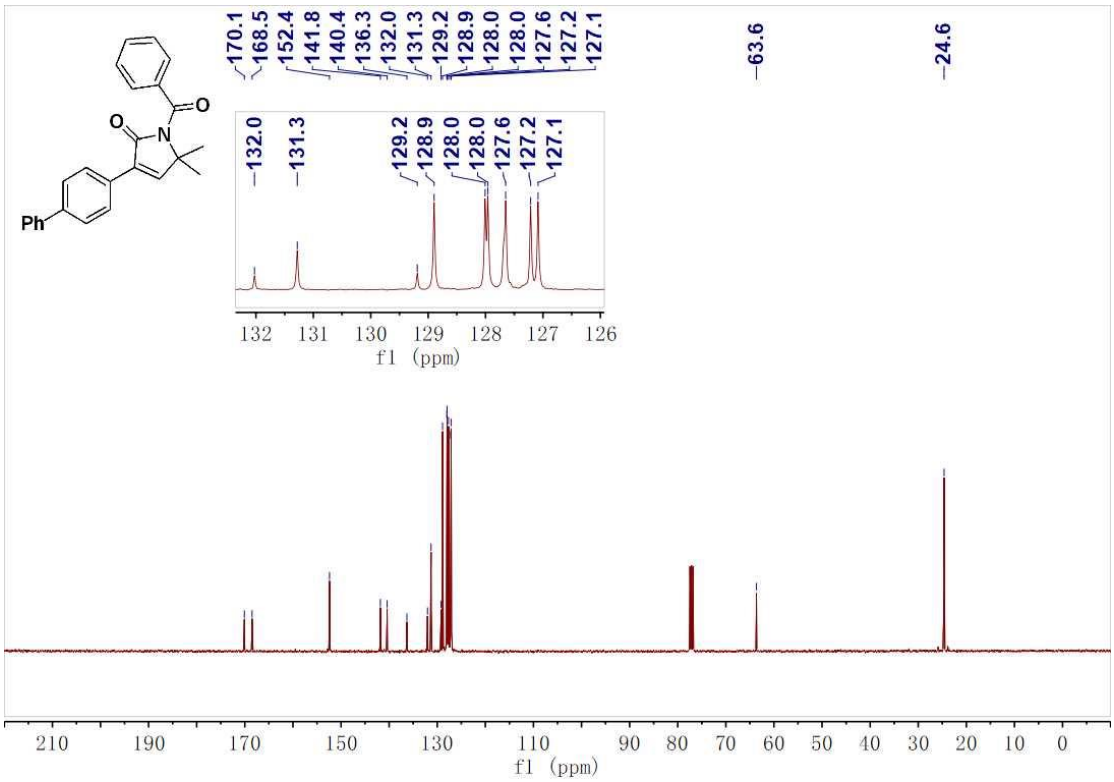
2aj


7.94
7.92
7.67
7.65
7.54
7.54
7.53
7.52
7.52
7.52
7.51
7.50
7.45
7.44
1.79
O
N
O
NC
7.9
7.7
7.5
f1 (ppm)
2.00
2.00
3.00
1.00
2.01
6.01
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

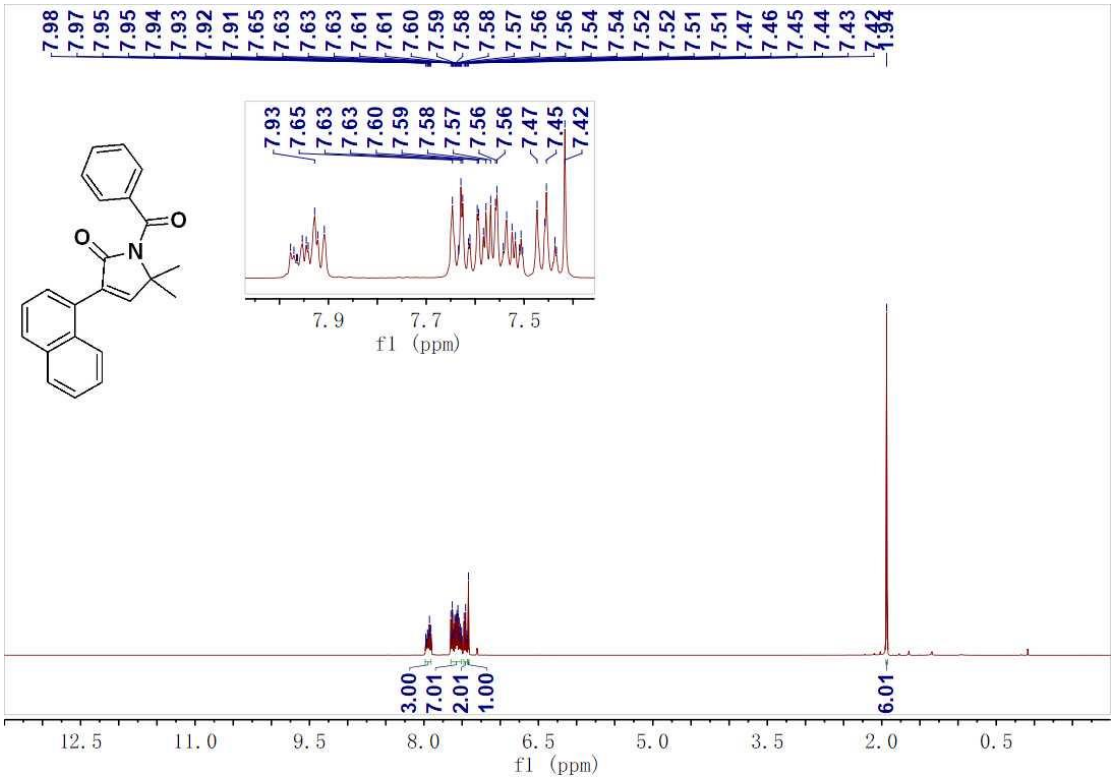

169.9
167.6
154.8
135.9
134.6
132.3
131.5
131.0
128.0
127.9
127.8
118.5
112.5
63.9
24.4
O
N
O
NC
132.3
131.5
131.0
128.0
127.9
127.8
133
131
129
127
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

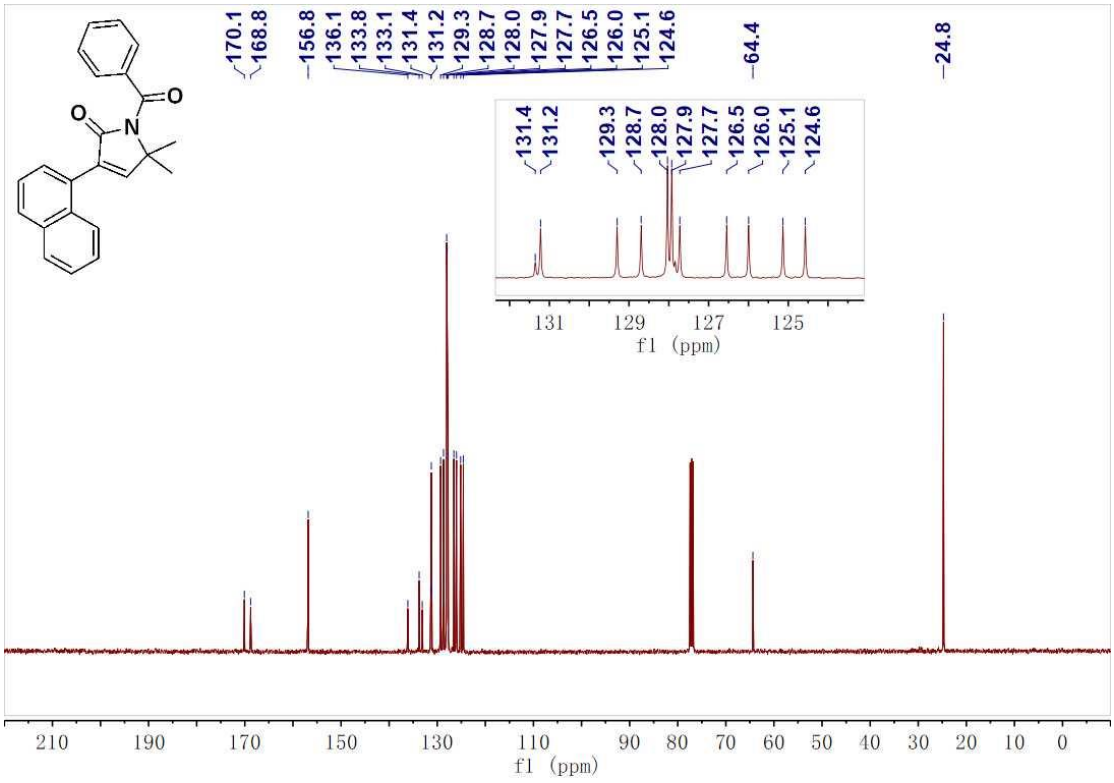
2ak


7.98
7.96
7.70
7.69
7.68
7.67
7.65
7.63
7.62
7.61
7.59
7.58
7.57
7.57
7.54
7.53
7.52
7.51
7.50
7.50
7.50
7.49
7.46
7.45
7.43
7.41
1.85
O
O
N
Ph
7.9
7.7
7.5
f1 (ppm)
2.01
4.00
3.01
4.00
1.00
1.01
6.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

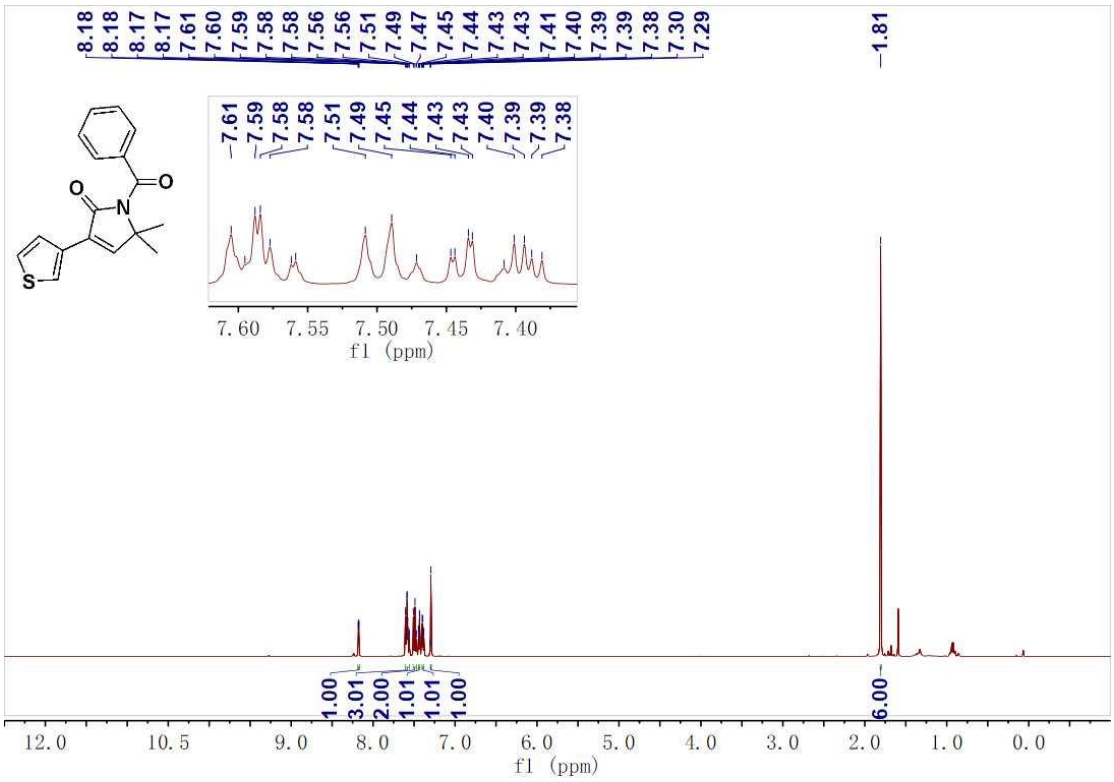

170.1
168.5
152.4
141.8
140.4
136.3
132.0
131.3
129.2
128.9
128.0
128.0
127.6
127.2
127.1
63.6
24.6
O
O
N
Ph
132.0
131.3
129.2
128.9
128.0
128.0
127.6
127.2
127.1
132
131
130
129
128
127
126
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

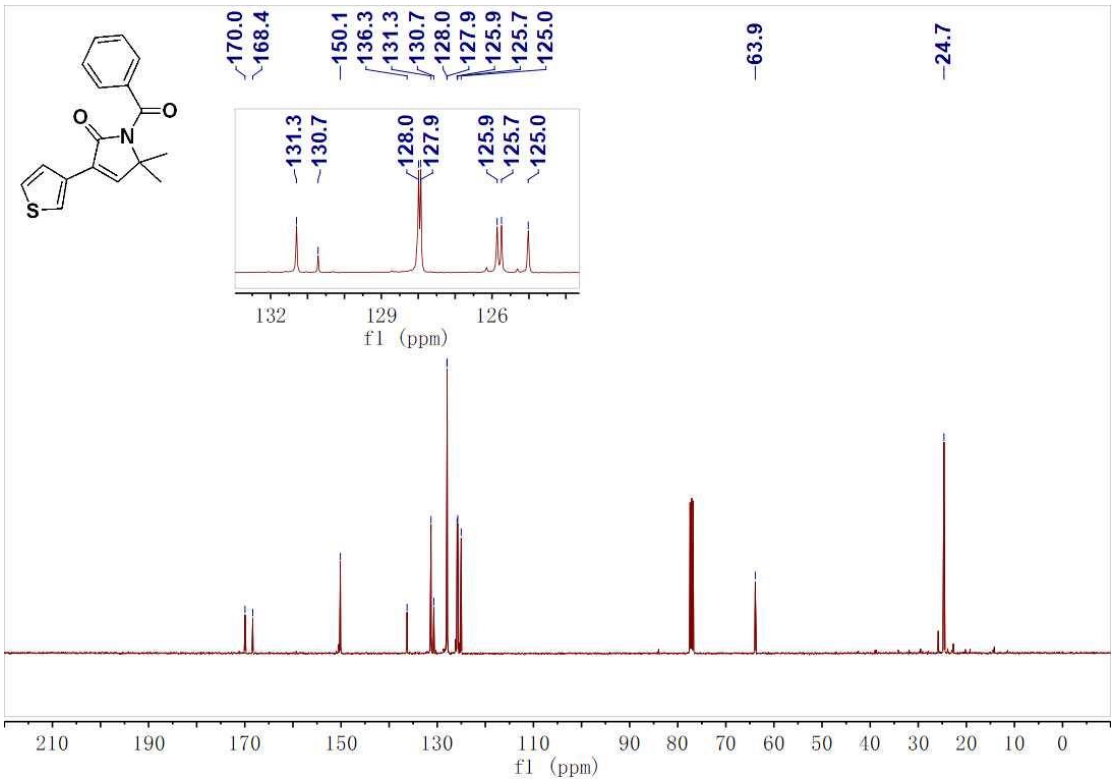
2al


7.98 7.97 7.95 7.95 7.94 7.93 7.92 7.91 7.65 7.63 7.63 7.63 7.61 7.61 7.60 7.59 7.58 7.58 7.57 7.56 7.56 7.54 7.54 7.52 7.51 7.51 7.47 7.46 7.45 7.44 7.43 7.42 1.94
7.93 7.65 7.63 7.63 7.60 7.59 7.58 7.57 7.56 7.56 7.47 7.45 7.42
7.9 7.7 7.5
f1 (ppm)
O
O
N
3.00 7.01 2.01 1.00
6.01
12.5 11.0 9.5 8.0 6.5 5.0 3.5 2.0 0.5
f1 (ppm)

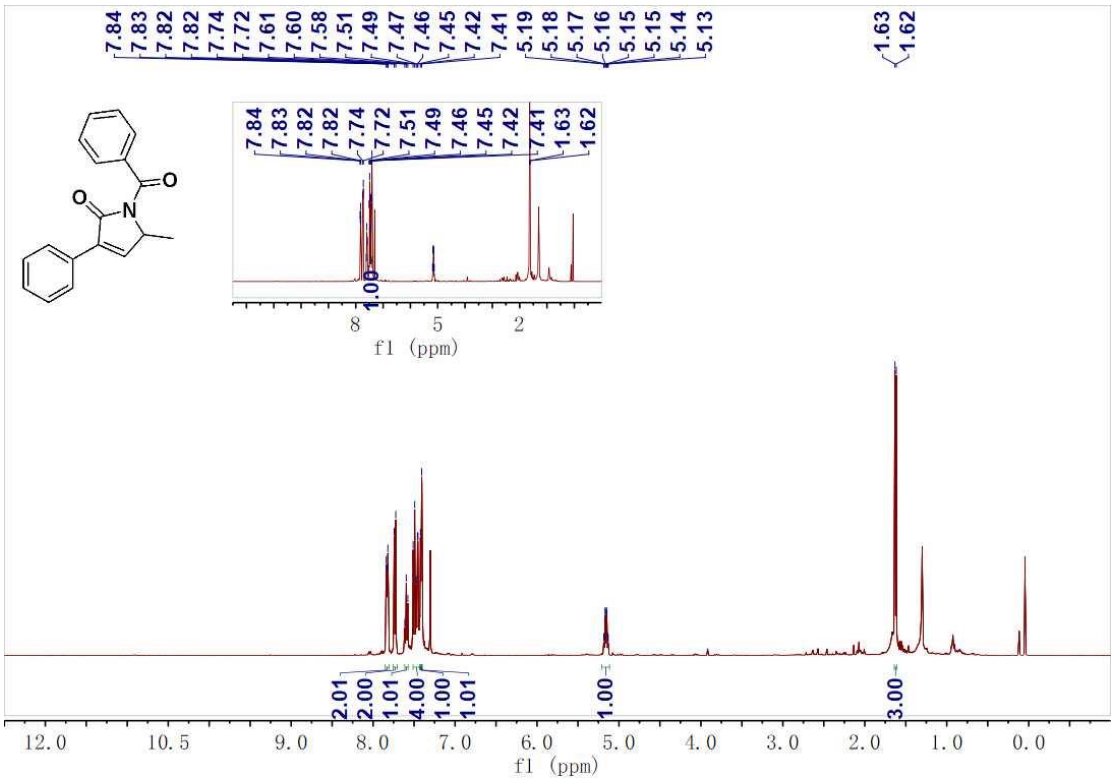

170.1 168.8 156.8 136.1 133.8 133.1 131.4 131.2 129.3 128.7 128.0 127.9 127.7 126.5 126.0 125.1 124.6 64.4 24.8
131.4 131.2 129.3 128.7 128.0 127.9 127.7 126.5 126.0 125.1 124.6
131 129 127 125
f1 (ppm)
O
O
N
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

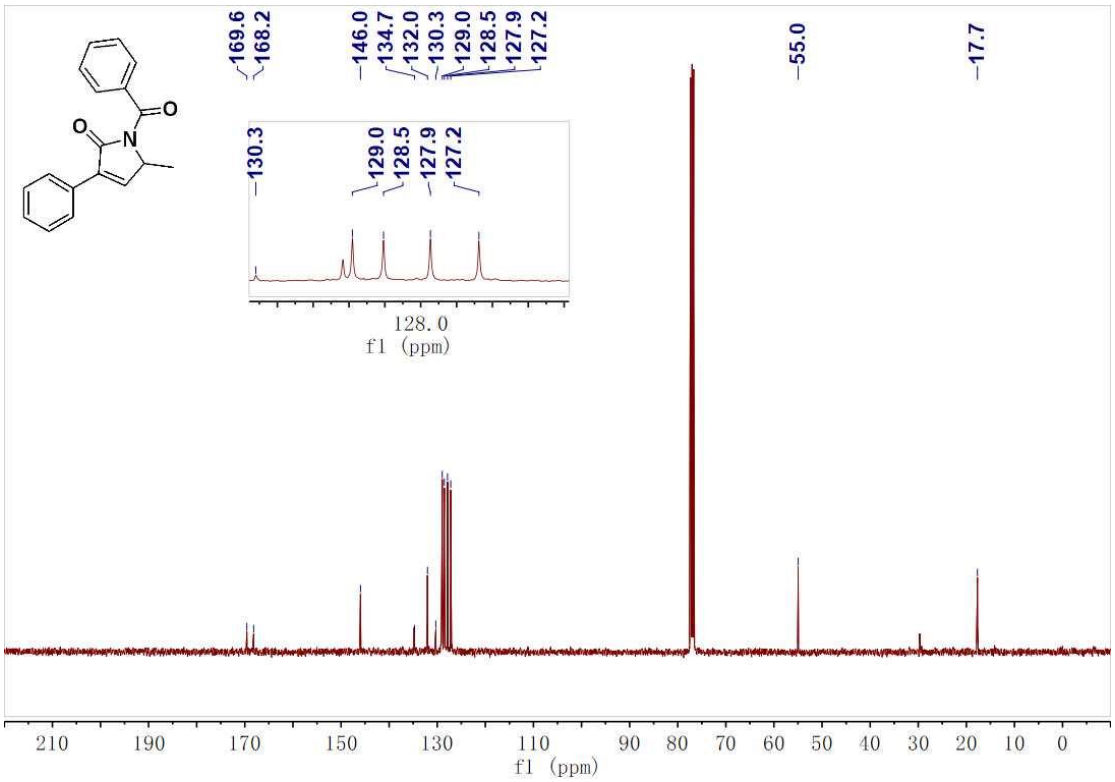
2am


8.18
8.18
8.17
8.17
7.61
7.60
7.59
7.58
7.58
7.56
7.56
7.51
7.49
7.47
7.45
7.44
7.43
7.43
7.41
7.40
7.39
7.39
7.38
7.30
7.29
1.81
7.61
7.59
7.58
7.58
7.51
7.49
7.45
7.44
7.43
7.43
7.40
7.39
7.39
7.38
7.60 7.55 7.50 7.45 7.40
f1 (ppm)
1.00
3.01
2.00
1.01
1.01
1.00
6.00
12.0 10.5 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

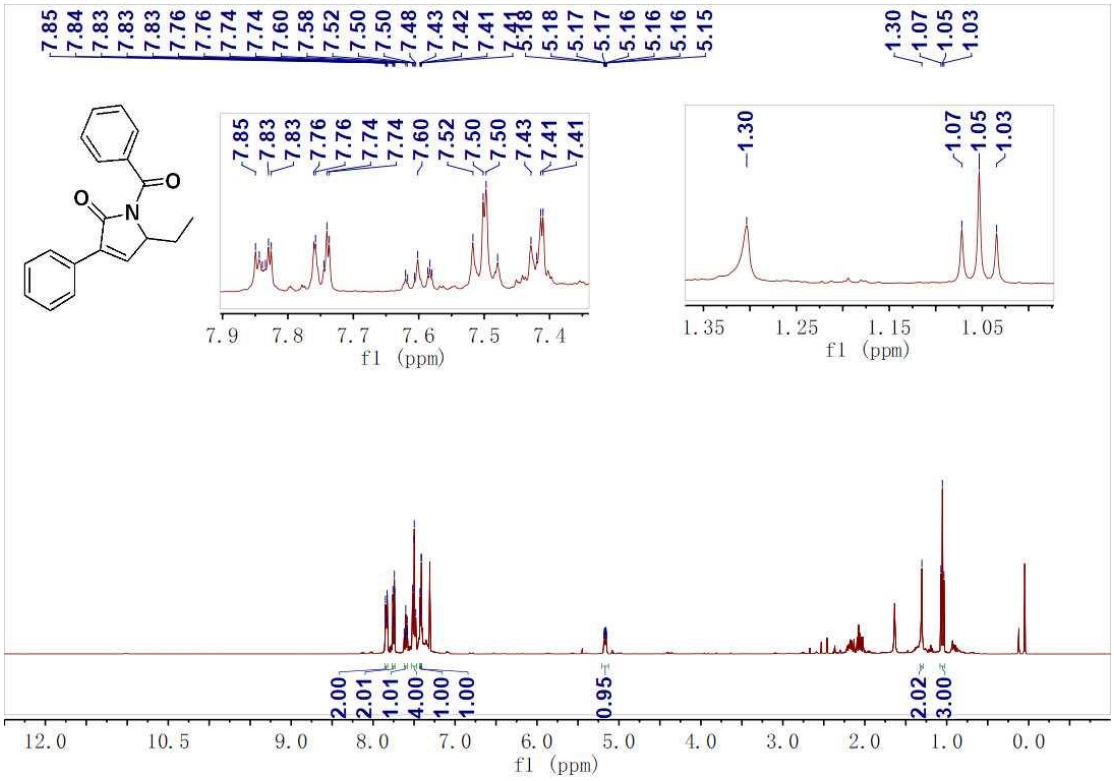

170.0
168.4
150.1
136.3
131.3
130.7
128.0
127.9
125.9
125.7
125.0
63.9
24.7
131.3
130.7
128.0
127.9
125.9
125.7
125.0
132 129 126
f1 (ppm)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

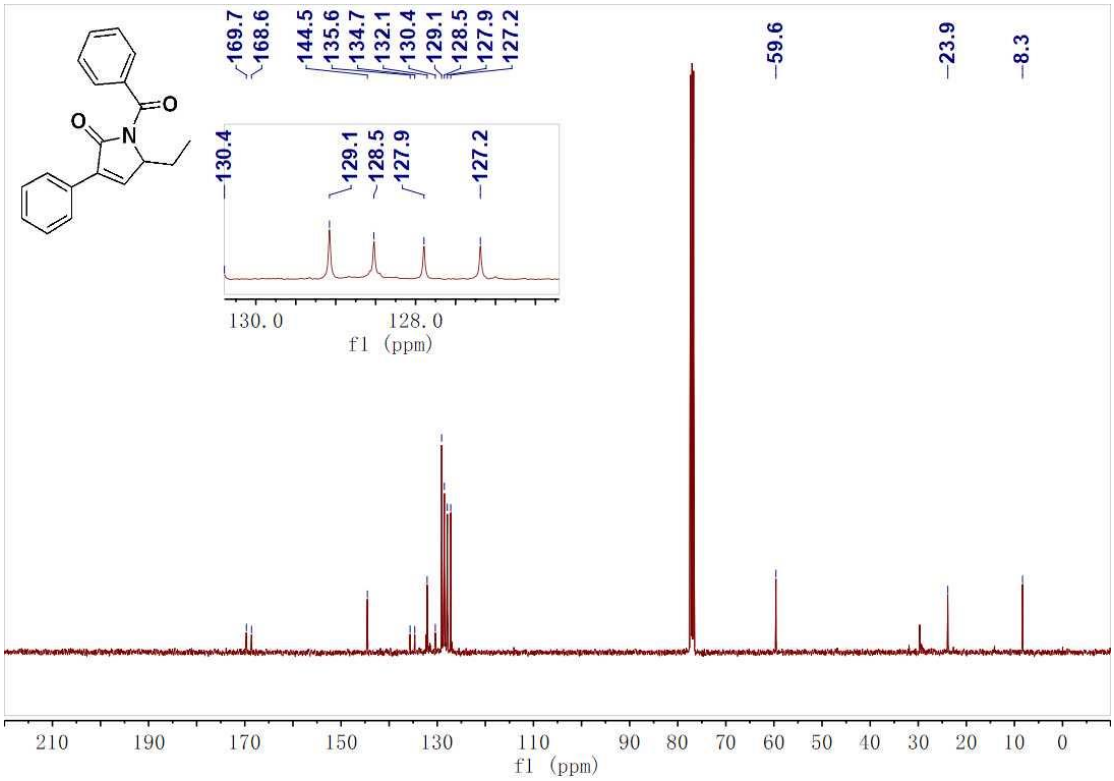
2ap


7.84 7.83 7.82 7.82 7.74 7.72 7.61 7.60 7.58 7.51 7.49 7.47 7.46 7.45 7.42 7.41 5.19 5.18 5.17 5.16 5.15 5.15 5.14 5.13 1.63 1.62
O
O
N
1.00
f1 (ppm)
2.01 2.00 1.01 4.00 1.00 1.01 1.00 3.00
f1 (ppm)


169.6 168.2 146.0 134.7 132.0 130.3 129.0 128.5 127.9 127.2 55.0 17.7
O
O
N
130.3 129.0 128.5 127.9 127.2
f1 (ppm)
f1 (ppm)

2aq


7.85 7.84 7.83 7.83 7.83 7.76 7.76 7.74 7.74 7.60 7.58 7.52 7.50 7.50 7.48 7.43 7.42 7.41 7.41 5.18 5.17 5.17 5.16 5.16 5.15
1.30 1.07 1.05 1.03
7.85 7.83 7.83 7.76 7.76 7.74 7.74 7.60 7.52 7.50 7.50 7.43 7.41 7.41
7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
1.30
1.07 1.05 1.03
1.35 1.25 1.15 1.05
f1 (ppm)
2.00 2.01 1.01 4.00 1.00 1.00 0.95 2.02 3.00
12.0 10.5 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


169.7 168.6 144.5 135.6 134.7 132.1 130.4 129.1 128.5 127.9 127.2
59.6
23.9
8.3
130.4 129.1 128.5 127.9 127.2
130.0 128.0
f1 (ppm)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

11. Spectra of Intermediate 2g’

2g’