Supporting Information

Catalyst-free synthesis of α,α-disubstituted carboxylic acid derivatives in ambient conditions via Wolff rearrangement

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1. General Information

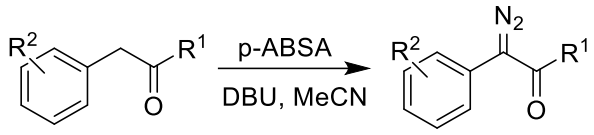
Unless otherwise noted, all air- and moisture-sensitive manipulations were carried out with standard Schlenk techniques under nitrogen or in a glove box under nitrogen. All reactions were carried out under a nitrogen atmosphere; materials obtained from commercial suppliers were used directly without further purification. Solvents were distilled following standard procedures before use. Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed on silica gel 60 (particle size 300-400 mesh ASTM, purchased from Yantai, China) and eluted with petroleum ether/ethyl acetate.

Trichloromethane ($CHCl_3$), dichloromethane, dichloroethane and ethyl acetate were freshly distilled from CaH_2; tetrahydrofuran (THF), toluene and ether were dried with sodium benzophenone and distilled before use.

^1H NMR spectra were recorded on a BRUKER 500 (500 MHz) or BRUKER 600 (600 MHz) spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with tetramethylsilane (TMS: 0 ppm) with the solvent resonance as the internal standard. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. ^{13}C NMR spectra were recorded on a BRUKER 500 (125 MHz) or BRUKER 600 (600 MHz) spectrometer in $CDCl_3$ with complete proton decoupling. Chemical shifts are reported in ppm with the deuterium solvent as the internal standard (e.g. $CDCl_3$: 77.0 ppm)..

2. General procedure for the Synthesis of diazo Compound

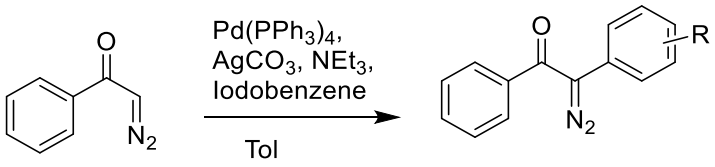
Method A



5

At 0°C, use 20 mL of acetonitrile to dissolve 10 mmol of compound **5** and 15 mmol of p-ABSA, and then add 15 mmol of DBU. until ketone was completely consumed determined by TLC analysis. The mixture was quenched with 100mL water, extracted with ether (20mL×3), and then the mixture was concentrated at 30°C. The mixture was concentrated under vaccum and the residue was purified by column chromatography on silica gel (PE/EA = 50:1 to 10:1) to afford the desired product.

Method B



4 **1**

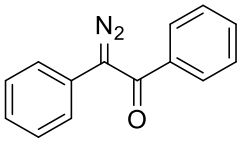
Dissolve 1.69 mmol of iodobenzene, 5% of tetrakistriphenylphosphine palladium, 0.65 mmol of silver carbonate, and 1.69 mmol of triethylamine with toluene in the reaction tube, and then dissolve 1.3 mmol of diazo **5** In 1ml of toluene, slowly drip into the reaction mixture, monitor the reaction by TLC, wait until the conversion of diazo **6** is complete, evaporate the solvent, and purify by column chromatography on silica gel (PE/EA = 50:1 to 10:1) to afford the desired product.

Method C

6

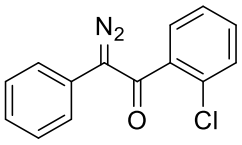
At room temperature, use 10 mL of acetonitrile to dissolve 5 mmol of compound **6** and 7.5 mmol of p-ABSA, and then add 7.5 mmol of DBU. until ketone was completely consumed determined by TLC analysis. The mixture was quenched with 50mL water, extracted with ether (20mL×3), and then the mixture was concentrated at 30°C. The mixture was concentrated under vaccum and the residue was purified by column chromatography on silica gel (PE/EA = 50:1 to 10:1) to afford the desired product.

1) 2-diazo-1,2-diphenylethan-1-one (1a)[1]



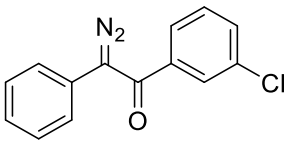
1a (Use method A to synthesis)**,** yellow solid, 7%, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.9 Hz, 2H), 7.54-7.45(m, 3H), 7.44-7.37(m, 4H), 7.29-7.24(m, 1H). ^{1}H NMR spectrum is consistent with literature reports.

2) 1-(2-chlorophenyl)-2-diazo-2-phenylethan-1-one (1b)



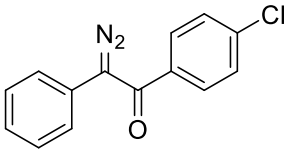
1b (Use method A to synthesis)**,** yellow oil, 76%, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90-7.35 (m, 8H), 7.30-7.25(m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 186.6, 137.8, 131.4, 130.6, 130.0, 129.0, 128.6, 127.3, 127.1, 125.3, 125.0, 75.5.

3) 1-(3-chlorophenyl)-2-diazo-2-phenylethan-1-one (1c)



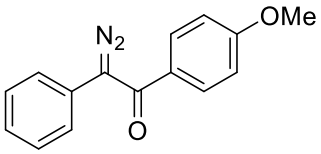
1c (Use method A to synthesis)**,** yellow solid, 70%, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 1.4 Hz, 1H), 7.53 – 7.37 (m, 6H), 7.34 (t, *J* = 7.8 Hz, 1H), 7.31 – 7.25 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 186.6, 139.4, 134.7, 131.6, 129.7, 129.1, 127.9, 127.3, 126.2, 125.7, 125.6, 73.4.

4) 1-(4-chlorophenyl)-2-diazo-2-phenylethan-1-one (1d)[1]



1d (Use method A to synthesis)**,** yellow solid, 24%, ^{1}H NMR (500 MHz, CDCl3) δ 7.59 – 7.50 (m, 2H), 7.48-7.33(m, 6H), 7.29 – 7.24 (m, 1H). ^{1}H NMR spectrum is consistent with literature reports.

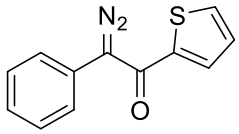
5) 2-diazo-1-(4-methoxyphenyl)-2-phenylethan-1-one (1e)[1]



1e (Use method A to synthesis)**,** yellow solid, 27%, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.1 Hz, 2H), 7.47-7.42 (m, 2H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.29-7.22 (m, 1H), 6.89 (d, *J* = 8.2 Hz, 2H), 3.84 (s, 3H). ^{1}H NMR spectrum is consistent with

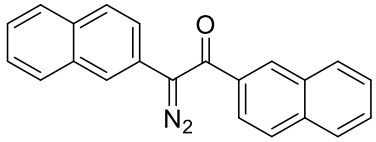
literature reports.

6) 2-diazo-2-phenyl-1-(thiophen-2-yl)ethan-1-one (1f)[2]



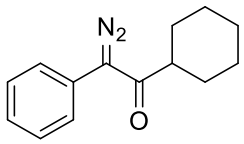
1f (Use method A to synthesis)**,** yellow solid, 50% , ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 5.0 Hz, 1H), 7.52 (d, *J* = 7.6 Hz, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 7.30 (d, *J* = 3.7 Hz, 1H), 7.02 (t, *J* = 4.3 Hz, 1H). NMR H spectrum is consistent with literature reports.

7) 2-diazo-1,2-di(naphthalen-2-yl)ethan-1-one (1g)



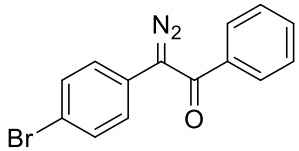
1g (Use method A to synthesis)**,** yellow solid, 43%, ^{1}H NMR (500 MHz, CDCl3) δ 8.21 (s, 1H), 8.07 (s, 1H), 7.92 – 7.81 (m, 6H), 7.74 (m, 7.27-7.20, 1H), 7.63 – 7.48 (m, 5H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 188.2, 135.2, 134.7, 133.5, 132.3, 132.0, 129.0, 128.7, 128.4, 128.4, 127.9, 127.8, 127.7, 127.6, 126.8, 126.6, 126.2, 124.8, 124.3, 123.8, 123.4, 73.7.

8) 1-cyclohexyl-2-diazo-2-phenylethan-1-one (1h)



1h (Use method A to synthesis)**,** yellow oil, 24%, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.5 Hz, 2H), 7.46-7.39 (m, 2H), 7.30 – 7.25 (m, 1H), 2.74 – 2.66 (m, 1H), 1.92-1.80 (m, 4H), 1.65 – 1.51 (m, 2H), 1.38 – 1.18 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ196.3, 128.9, 126.8, 125.9, 125.7, 70.8, 46.7, 28.9, 25.6, 25.6.

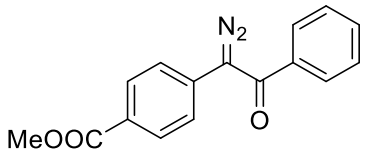
9) 2-(4-bromophenyl)-2-diazo-1-phenylethan-1-one (1i)[1]



1i (Use method A to synthesis)**,** yellow solid, 86%, ^{1}H NMR (500 MHz, $CDCl_3$) δ

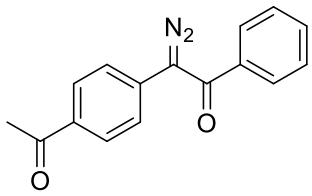
7.64 – 7.59 (m, 2H), 7.56 – 7.50 (m, 3H), 7.47 – 7.42 (m, 2H), 7.40 – 7.35 (m, 2H). ^{1}H NMR spectrum is consistent with literature reports.

10) ethyl 4-(1-diazo-2-oxo-2-phenylethyl)benzoate (1j)[3]



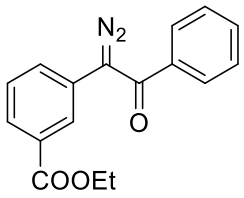
1j (Use method B to synthesis)**,** yellow solid , 71%, ^{1}H NMR (500 MHz, CDCl3) δ 8.06 (dd, J = 8.4, 1.4 Hz, 2H), 7.65 – 7.61 (m, 2H), 7.58 (d, J = 8.0 Hz, 2H), 7.53 (td, J = 7.6, 1.3 Hz, 1H), 7.48 – 7.42 (m, 2H), 3.92 (s, 3H). ^{1}H NMR spectrum is consistent with literature reports.

11) 2-(4-acetylphenyl)-2-diazo-1-phenylethan-1-one (1k)



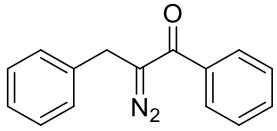
1k (Use method B to synthesis)**,** yellow solid, 66%, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (t, *J* = 8.1 Hz, 2H), 7.68 – 7.58 (m, 4H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 2.59 (s, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 197.1, 187.9, 137.6, 135.0, 132.1, 131.6, 129.0, 128.7, 127.6, 124.8, 73.3, 26.5. HRMS (ESI) m/z calculated for $C_{16}H_{12}N_2NaO_2$, $[M+Na]^+$ = 287.0796, found 287.0788.

12) ethyl 3-(1-diazo-2-oxo-2-phenylethyl)benzoate (1l)



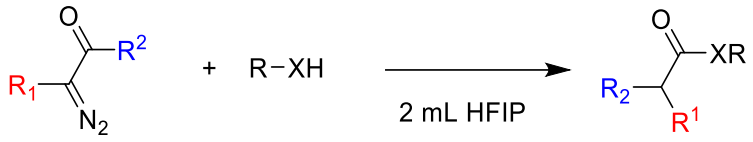
1l (Use method B to synthesis), yellow solid, 65%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.93 (d, *J* = 7.7 Hz, 1H), 7.78 (d, *J* = 7.7 Hz, 1H), 7.63 (d, *J* = 7.9 Hz, 2H), 7.47 (m, 7.55-7.41, 4H), 4.38 (q, *J* = 6.7 Hz, 2H), 1.39 (t, *J* = 7.1, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ188.1, 166.1, 137.6, 131.9, 131.3, 130.4, 129.1, 128.6, 127.9, 127.6, 126.8, 126.2, 72.6, 61.2, 14.3. HRMS (ESI) m/z calculated for $C_{17}H_{14}N_2NaO_3$, $[M+Na]^+$ = 317.0902, found 317.0897.

13) 2-diazo-1,3-diphenylpropan-1-one (1m)[4]



1m (Use method C to synthesis), yellow oil, 20%, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 – 7.62 (m, 2H), 7.54 – 7.49 (m, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.41 – 7.36 (m, 2H), 7.35-7.29 (m, 3H), 3.89 (s, 2H). ^{1}H NMR spectrum is consistent with literature reports.

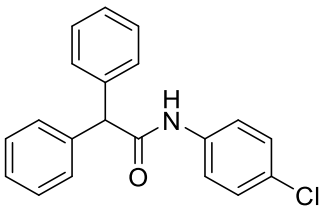
3. General procedure for wolff rearrangement reaction



1 **2** **3**

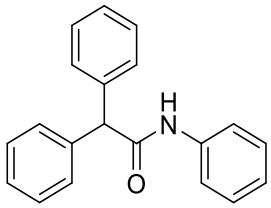
In a 4 mL glass bottle, aniline **2** (0.2 mmol, 1 equivalent) was dissolved in 2 mL of hexafluoroisopropanol, and 1.2 equiv diazo compound **1** was added directly to the mixture at once. The resulting mixture was then continuously stirred at room temperature for 10 min-6 hours, until aniline **2** was completely consumed determined by TLC analysis. The mixture was concentrated under vaccum and the residue was purified by column chromatography on silica gel (PE/EA = 50:1 to 5:1) to afford the desired product.

14) N-(4-chlorophenyl)-2,2-diphenylacetamide (3a)[1]



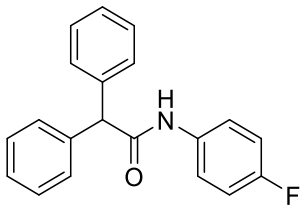
60.1mg, 93%, white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.21 (m, 15H), 5.07 (s, 1H). ^{1}H NMR spectrum is consistent with literature reports.

15) N,2,2-triphenylacetamide (3b)[5]



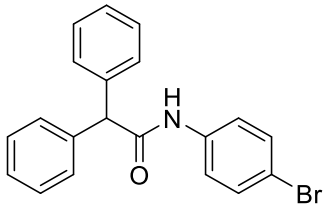
56.0 mg, 98%, white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.1 Hz, 3H), 7.37 – 7.23 (m, 12H), 7.08 (t, *J* = 7.2 Hz, 1H), 5.07 (s, 1H). ^{1}H NMR spectrum is consistent with literature reports.

16) N-(4-fluorophenyl)-2,2-diphenylacetamide (3c)[5]



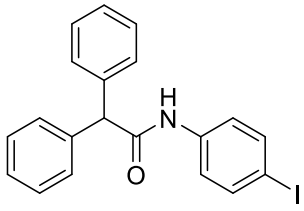
59.9 mg, 98%, white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (s,1H), 7.43 – 7.28 (m, 12H), 6.95 (t, *J* = 8.1 Hz, 2H), 5.06 (s, 1H). ^{1}H NMR spectrum is consistent with literature reports.

17) N-(4-bromophenyl)-2,2-diphenylacetamide (3d)[5]



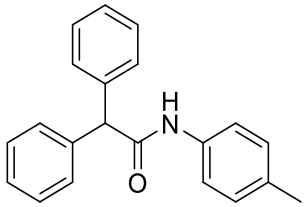
68.6 mg, 94%, white solid. ^{1}H NMR (500 MHz, CDCl3) δ 7.41-7.28 (m, 14H), 5.06 (s, 1H). ^{1}H NMR spectrum is consistent with literature reports.

18) N-(4-iodophenyl)-2,2-diphenylacetamide (3e)[5]



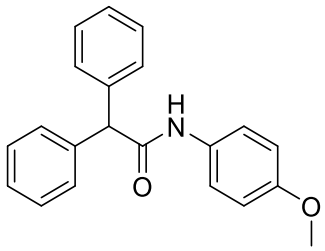
80.0 mg, 97%, white solid, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.5 Hz, 2H), 7.40 – 7.27 (m, 11H), 7.24 (d, *J* = 8.5 Hz, 2H), 5.06 (s, 1H). ^{1}H NMR spectrum is consistent with literature reports.

19) 2,2-diphenyl-N-(p-tolyl)acetamide (3f)[5]



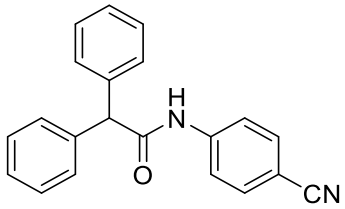
57.2 mg, 95%, white solid, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.38 – 7.27 (m, 12H), 7.08 (d, *J* = 8.2 Hz, 2H), 5.07 (s, 1H), 2.30 (s, 3H). ^{1}H NMR spectrum is consistent with literature reports.

20) N-(4-methoxyphenyl)-2,2-diphenylacetamide (3g)[5]



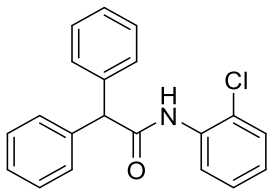
60.5 mg, 95%, white solid, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 12H), 7.22 (s, 1H), 6.86 – 6.79 (m, 2H), 5.07 (s, 1H), 3.77 (d, *J* = 6.2 Hz, 3H). ^{1}H NMR spectrum is consistent with literature reports.

21) N-(4-cyanophenyl)-2,2-diphenylacetamide (3h)[5]



54.3 mg, 87%, white solid, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (s, 1H), 7.57 (dd, *J* = 22.3, 8.6 Hz, 4H), 7.41-7.27 (m, 10H), 5.09 (s, 1H). ^{1}H NMR spectrum is consistent with literature reports.

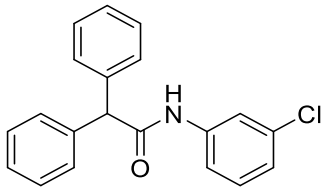
22) N-(2-chlorophenyl)-2,2-diphenylacetamide (3i)[6]



60.5 mg, 94%, white solid, ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.2 Hz, 1H),

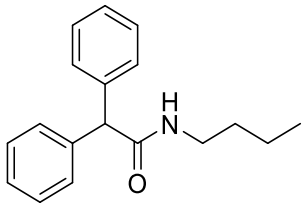
7.89 (s, 1H), 7.41 – 7.23 (m, 12H), 7.02 (t, *J* = 7.6 Hz, 1H), 5.17 (s, 1H). ^{1}H NMR spectrum is consistent with literature reports.

23) N-(3-chlorophenyl)-2,2-diphenylacetamide (3j)



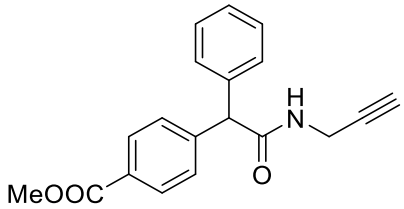
61.4 mg, 98%, white solid, m.p. = 152.6-153.9 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (s, 1H), 7.58 (s, 1H), 7.38 – 7.26 (m, 11H), 7.16 (t, *J* = 8.1 Hz, 1H), 7.06 (d, *J* = 7.9 Hz, 1H), 5.05 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.4, 138.7, 138.7, 134.5, 129.9, 128.9, 128.8, 127.5, 124.5, 119.9, 117.8, 59.8. HRMS (ESI) m/z calculated for $C_{20}H_{16}ClNNaO$, $[M+Na]^+$ = 344.0818, found 388.0813.

24) N-butyl-2,2-diphenylacetamide (3k)[7]



37.5 mg, 70%, colorless oil, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.33 (m, 4H), 7.31-7.24 (m, 6H), 5.71 (s, 1H), 4.94 (s, 1H), 3.29 (q, *J* = 10 Hz, , 2H), 1.50 – 1.44 (m, 2H), 1.34-1.25 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{1}H NMR spectrum is consistent with literature reports.

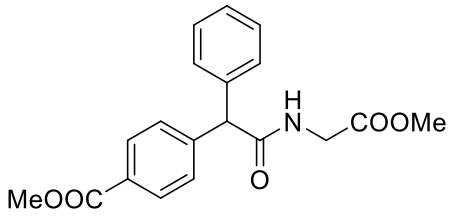
25) methyl 4-(2-oxo-1-phenyl-2-(prop-2-yn-1-ylamino)ethyl)benzoate (3l)



50.8 mg, 83% , white solid, m.p. = 61.8-63.4 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.1 Hz, 2H), 7.36 – 7.31 (m, 4H), 7.29 (d, *J* = 7.1 Hz, 1H), 7.24 (d, *J* = 7.4 Hz, 2H), 6.02 (brs, 1H), 4.96 (s, 1H), 4.12 – 4.01 (m, 2H), 3.89 (s, 3H), 2.21 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) 170.8, 166.7, 144.1, 138.3, 129.9, 129.1, 129.0, 128.9, 128.8,

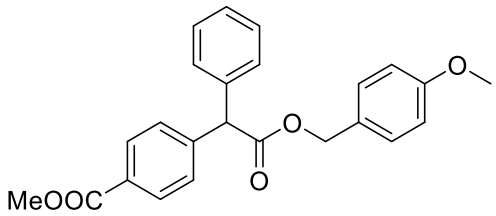
127.7, 79.1, 71.8, 58.5, 52.1, 29.5. HRMS (ESI) m/z calculated for $C_{19}H_{17}NNaO_3$, $[M+Na]^+$ = 330.1101 , found 330.1096.

26) methyl 4-(2-((2-methoxy-2-oxoethyl)amino)-2-oxo-1-phenylethyl)benzoate (3m)



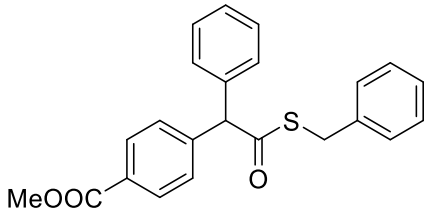
Using glycine methyl ester hydrochloride, 1.2 equiv NEt_3 was added to release glycine methyl ester as nucleophile. 55.8 mg, 82%, white solid, m.p. = 147.3-148 ^oC. ^1H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.0 Hz, 2H), 7.44-7.34 (m, 4H), 7.34-7.25 (m, 3H), 6.24 (brs, 1H), 5.04 (s, 1H), 4.14 – 4.01 (m, 2H), 3.92 (s, 3H), 3.76 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.5, 170.1, 166.7, 144.2, 138.3, 130.0, 129.1, 129.0, 129.0, 128.9 127.7, 58.6, 52.4, 52.1, 41.4. HRMS (ESI) m/z calculated for $C_{19}H_{19}NNaO_5$, $[M+Na]^+$ = 364.1155 , found 364.1153.

27) methyl 4-(2-((4-methoxybenzyl)oxy)-2-oxo-1-phenylethyl)benzoate (3n)



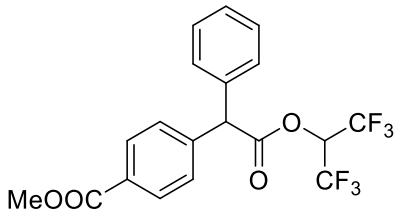
Using 1.5 mL of DCM and 0.5 mL of HFIP as solvent. 40.7 mg, 52% yield, colorless oil, ^1H NMR (500 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.35 – 7.27 (m, 5H), 7.25 (d, *J* = 7.7 Hz, 2H), 6.88 (d, *J* = 7.5 Hz, 2H), 5.19 – 5.12 (m, 2H), 5.11 (s, 1H), 3.92 (s, 3H), 3.83 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 166.8, 159.7, 143.7, 137.9, 130.1, 129.8, 129.1, 128.7, 128.7, 128.6, 127.6, 127.5, 113.9, 67.0, 57.0, 55.3, 52.1. HRMS (ESI) m/z calculated for $C_{24}H_{22}NaO_5$, $[M+Na]^+$ = 413.1359, found 413.1348.

28) methyl 4-(2-(benzylthio)-2-oxo-1-phenylethyl)benzoate (3o)



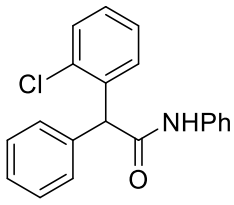
Using 1.5 mL of DCM and 0.5 mL of HFIP as solvent. 54.2 mg, 72% yield, colorless oil, ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.34 – 7.23 (m, 10H), 5.24 (s, 1H), 4.20 – 4.13 (m, 2H), 3.90 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.5, 186.7, 143.1, 137.4, 136.8, 129.9, 129.3, 128.9, 128.8, 128.8, 128.8, 128.6, 127.8, 127.4, 64.7, 52.1, 34.0. HRMS (ESI) m/z calculated for $C_{23}H_{20}NaO_3S$, $[M+Na]^+$ = 399.1025, found 399.1018.

29) methyl 4-(2-((1,1,1,3,3,3-hexafluoropropan-2-yl)oxy)-2-oxo-1-phenylethyl)benzoate (3p)



51.3 mg, 61% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.2 Hz, 2H), 7.39 – 7.34 (m, 5H), 7.27 (d, *J* = 7.4 Hz, 2H), 5.81 (dt, *J* = 12.0, 6.0 Hz, 1H), 5.26 (s, 1H), 3.91 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.0, 166.6, 141.6, 135.9, 130.1, 129.8, 129.0, 128.5, 128.4, 128.2, 120.2 (q, *J* = 281.25 Hz), 66.9 (quint, *J* = 42 Hz), 56.0, 52.2. HRMS (ESI) m/z calculated for $C_{19}H_{14}F_6NaO_4$, $[M+Na]^+$ = 443.0688, found 443.0687.

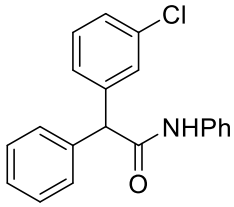
30) 2-(2-chlorophenyl)-N,2-diphenylacetamide (3q)



62.1 mg, 97% yield, white solid, m.p. = 199.9-201 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.0 Hz, 2H), 7.43 – 7.28 (m, 10H), 7.27-7.23 (m, 2H), 7.11 (t, *J* = 7.3 Hz, 1H), 5.50 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ169.2, 137.6, 137.5, 136.9, 134.5,

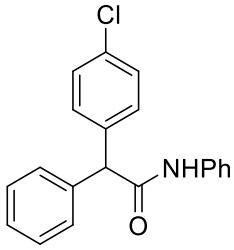
130.4, 129.7, 129.2, 129.0, 129.0, 128.8, 127.8, 127.2, 124.6, 119.8, 56.6. HRMS (ESI) m/z calculated for $C_{20}H_{16}ClNNaO$ $[M+Na]^+$ = 344.0813, found 344.0806.

31) 2-(3-chlorophenyl)-N,2-diphenylacetamide (3r)



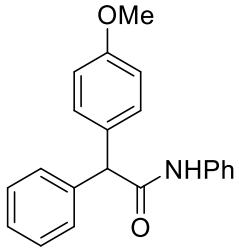
53.0 mg, 82% yield, white solid, m.p. = 152.6-153.4 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (brs, 1H), 7.44 (d, *J* = 7.8 Hz, 2H), 7.40 – 7.24 (m, 10H), 7.22-7.18 (m, 1H), 7.11 (t, *J* = 7.4 Hz, 1H), 5.01 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.5, 141.0, 138.3, 137.4, 134.5, 130.0, 129.1, 129.0, 128.9, 128.8, 127.8, 127.6, 127.1, 124.7, 119.9, 59.3. HRMS (ESI) m/z calculated for $C_{20}H_{16}ClNNaO$ $[M+Na]^+$ = 344.0813, found 344.0811.

32) 2-(4-chlorophenyl)-N,2-diphenylacetamide (3s)



61.6 mg, 96% yield, white solid, m.p. = 178-180 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (brs, 1H), 7.47 (d, *J* = 7.9 Hz, 2H), 7.41 – 7.23 (m, 11H), 7.13 (t, *J* = 7.2 Hz, 1H), 5.04 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.7, 138.6, 137.5, 137.4, 133.3, 130.3, 129.0, 129.0, 128.9, 128.8, 127.7, 124.7, 119.8, 59.1. HRMS (ESI) m/z calculated for $C_{20}H_{16}ClNNaO$ $[M+Na]^+$ = 344.0813, found 344.0806.

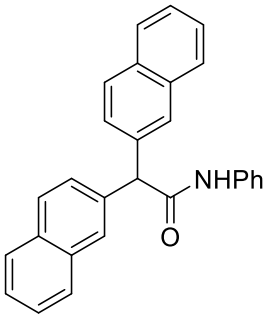
33) 2-(4-methoxyphenyl)-N,2-diphenylacetamide (3t)



60.7 mg, 96% yield, white solid, m.p. = 176-177 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ

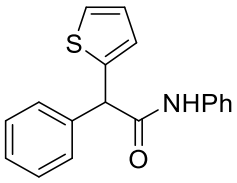
7.52 – 7.44 (m, 3H), 7.41 – 7.35 (m, 2H), 7.35-7.28 (m, 5H), 7.25 (d, *J* = 8.2 Hz, 2H), 7.12 (t, *J* = 7.2 Hz, 1H), 6.91 (d, *J* = 8.2 Hz, 2H), 5.06 (s, 1H), 3.82 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.4, 158.8, 139.4, 137.6, 131.1, 130.0, 128.9, 128.8, 128.8, 127.4, 124.4, 119.7, 114.3, 59.2, 55.3. HRMS (ESI) m/z calculated for $C_{21}H_{19}NNaO_2$ $[M+Na]^+$ = 340.1308, found 340.1311.

34) 2-(4-chlorophenyl)-2-(naphthalen-1-yl)-N-phenylacetamide (3u)



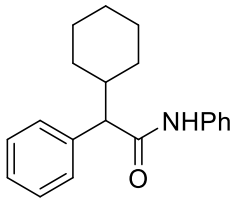
70.6mg, 91% yield, white solid, m.p. = 229-230 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 – 7.73 (m, 8H), 7.51 (s, 8H), 7.37 – 7.23 (m, 3H), 7.13 (t, *J* = 6.5 Hz, 1H), 5.44 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.94, 137.58, 136.38, 133.45, 132.65, 128.98, 128.75, 128.00, 127.83, 127.65, 127.04, 126.39, 126.24, 124.62, 119.85, 60.29. HRMS (ESI) m/z calculated for $C_{28}H_{21}NNaO$, $[M+Na]^+$ =410.1515, found 410.1510;

35) N,2-diphenyl-2-(thiophen-2-yl)acetamide (3v)



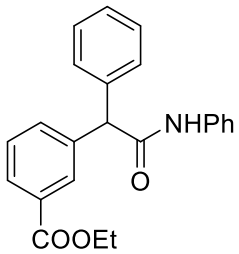
55.1 mg, 94% yield, white solid, m.p. = 141-142 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (brs, 1H), 7.46 (d, *J* = 7.9 Hz, 2H), 7.42 – 7.25 (m, 8H), 7.10 (t, *J* = 7.4 Hz, 1H), 7.02 – 6.95 (m, 2H), 5.26 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.3, 141.6, 139.0, 137.4, 128.9, 128.9, 128.4, 127.8, 126.9, 126.8, 125.8, 124.6, 120.0, 55.0. HRMS (ESI) m/z calculated for $C_{18}H_{15}NNaOS$, $[M+Na]^+$ = 316.0767, found 316.0762.

36) 2-cyclohexyl-N,2-diphenylacetamide (3w)



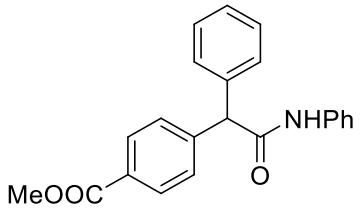
1.5 equiv diazo was used. 57.2 mg, 97% yield, white solid, m.p. = 191-192 ºC , ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.28 (m, 5H), 7.25-7.15 (m, 5H), 6.98 (t, *J* = 7.2 Hz, 1H), 3.00 (d, *J* = 10.1 Hz, 1H), 2.11 (q, *J* = 10.3 Hz, 1H), 1.93 (d, *J* = 11.8 Hz, 1H), 1.69 – 1.50 (m, 3H), 1.32-1.22 (m, 2H), 1.13 – 0.94 (m, 3H), 0.69 (q, *J* = 11.7 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 138.4, 137.8, 128.8, 128.6, 128.3, 127.2, 124.2, 119.8, 61.8, 40.8, 32.2, 30.6, 26.3, 26.1. HRMS (ESI) m/z calculated for $C_{20}H_{23}NNaO$ $[M+Na]^+$ = 316.1672, found 316.1669.

37) ethyl 3-(2-oxo-1-phenyl-2-(phenylamino)ethyl)benzoate (3x)



59.6 mg, 83% yield, yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.97 – 7.89 (m, 2H), 7.54 (d, *J* = 7.7 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 1H), 7.36 – 7.23 (m, 7H), 7.08 (t, *J* = 7.4 Hz, 1H), 5.09 (s, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 1.36 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.8, 166.4, 139.5, 138.6, 137.6, 133.3, 130.8, 130.0, 128.9, 128.8, 128.7, 128.5, 127.6, 124.5, 119.9, 61.0, 59.3, 14.2. HRMS (ESI) m/z calculated for $C_{23}H_{21}NNaO_3$, $[M+Na]^+$ =382.1414, found 382.1410.

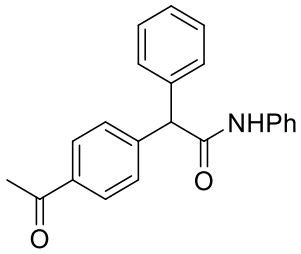
38) methyl 4-(2-oxo-1-phenyl-2-(phenylamino)ethyl)benzoate (3y)



63 mg, 91% yield, white solid, m.p. = 79.6-81.8 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.1 Hz, 2H), 7.78 (brs, 1H), 7.47 (d, *J* = 8.2 Hz, 2H), 7.42-7.35 (m, 4H),

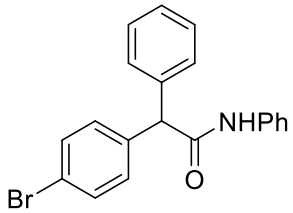
7.33 – 7.24 (m, 5H), 7.12 (t, *J* = 7.3 Hz, 1H), 5.12 (s, 1H), 3.92 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.5, 166.8, 144.2, 138.4, 137.5, 130.0, 129.1, 129.0, 129.0, 128.9, 128.8, 127.7, 124.7, 119.9, 59.6, 52.1. HRMS (ESI) m/z calculated for $C_{22}H_{19}NNaO_3$, $[M+Na]^+$ = 368.1257, found 368.1252.

39) 2-(4-acetylphenyl)-N,2-diphenylacetamide (3z)



Using 1.5 mL of DCM and 0.5 mL of HFIP as solvent. 60.1 mg, 91% yield, yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.9 Hz, 2H), 7.79 (brs, 1H), 7.46 (d, *J* = 8.1 Hz, 2H), 7.42-7.38 (m, 2H), 7.37-7.33 (m, 2H), 7.32 – 7.24 (m, 5H), 7.10 (t, *J* = 7.3 Hz, 1H), 5.10 (s, 1H), 2.56 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.8, 169.4, 144.4, 138.2, 137.5, 136.0, 129.2, 129.0, 128.9, 128.8, 128.7, 127.8, 124.6, 119.9, 59.5, 26.6. HRMS (ESI) m/z calculated for $C_{22}H_{19}NNaO_2$, $[M+Na]^+$ =352.1308, found 352.1299.

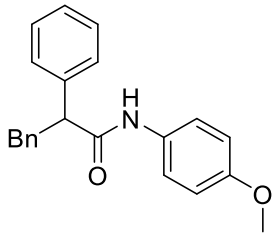
40) 2-(4-bromophenyl)-N,2-diphenylacetamide (3aa)



66.5 mg, 91% yield white solid, m.p. = 158.3-161.2 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ

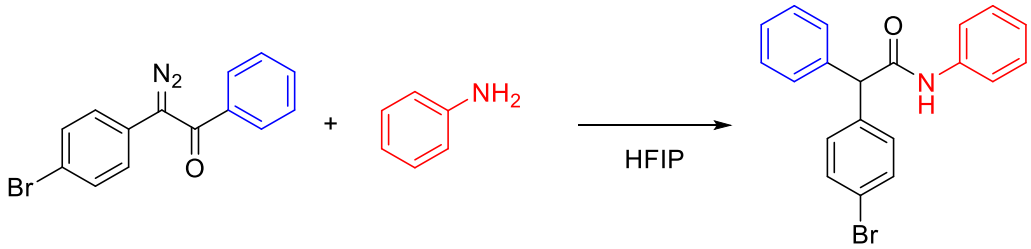
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (s, 1H), 7.45 (dd, *J* = 7.8, 5.9 Hz, 4H), 7.40 – 7.25 (m, 7H), 7.17 (d, *J* = 8.3 Hz, 2H), 7.11 (t, *J* = 7.4 Hz, 1H), 5.00 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) 169.7, 138.5, 138.1, 137.4, 131.8, 130.6, 129.0, 129.0, 128.8, 127.7, 124.7, 121.5, 119.9, 59.1. HRMS (ESI) m/z calculated for $C_{20}H_{16}BrNNaO$, $[M+Na]^+$ =388.0307, found 388.0304;

41) N,2,3-triphenylpropanamide (3ab)



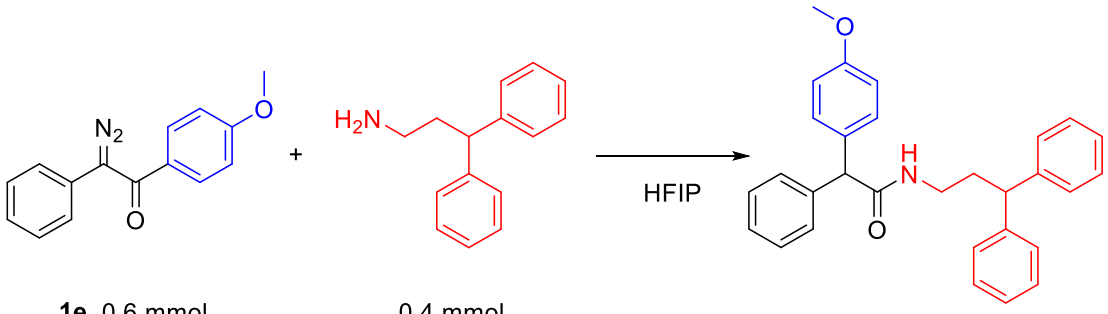
Using 1.5 mL of DCM and 0.5 mL of HFIP as solvent. 24.5 mg, 37% yield, white solid, m.p. = 158-159 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.31 (m, 4H), 7.28-7.20 (m, 5H), 7.19-7.12 (m, 3H), 7.08 (s, 1H), 6.77 (d, *J* = 8.3 Hz, 2H), 3.77 – 3.69 (m, 4H), 3.62 (dd, *J* = 13.3, 7.7 Hz, 1H), 3.05 (dd, *J* = 13.4, 6.6 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.8, 156.4, 139.5, 139.3, 130.7, 129.0, 128.8, 128.3, 128.1, 127.5, 126.2, 121.9, 114.0, 56.3, 55.4, 39.8. HRMS (ESI) m/z calculated for $C_{22}H_{21}NNaO_2$, $[M+Na]^+$ =354.1464, found 354.1461.

4. Scale-Up reaction and synthesis application



6 mmol 5 mmol **3aa**,1.65g, 90%

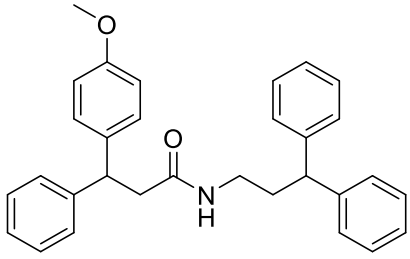
In a 25 mL glass bottle, aniline (5 mmol, 465.7mg, 1 equivalent) was dissolved in 10 mL of hexafluoroisopropanol, and 1.8g diazo **1i** (1.2 equivalent) was added directly to the mixture at once. The resulting mixture was then continuously stirred at room temperature, until aniline was completely consumed determined by TLC analysis. The mixture was concentrated under vaccum and the residue was purified by column chromatography on silica gel (PE/EA =20:1) to afford the **3aa** 1.65g, 90%.



1e, 0.6 mmol 0.4 mmol **3ac**, 86% cannabinoid CB1 receptor

In a 4 mL glass bottle, 3,3-diphenylpropylamine (0.4 mmol, 84.4mg, 1 equivalent) was dissolved in 4 mL of hexafluoroisopropanol, and 151mg diazo **1e** (1.5 equivalent) was added directly to the mixture at once. The resulting mixture was then continuously stirred at room temperature, until 3,3-diphenylpropylamine was completely consumed determined by TLC analysis. The mixture was concentrated under vaccum and the residue was purified by column chromatography on silica gel (PE/EA =8:1) to afford the **3ac** 149mg, 86%.

42) N-(3,3-diphenylpropyl)-3-(4-methoxyphenyl)-3-phenylpropanamide (3ac)[8]



149.0 mg, 86%, (0.4 mmol scale), white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.6 Hz, 2H), 7.41 – 7.33 (m, 7H), 7.33 – 7.25 (m, 8H), 6.99 (d, *J* = 8.7 Hz, 2H), 5.79 (t, *J* = 5.6 Hz, 1H), 4.93 (s, 1H), 3.96 (t, *J* = 7.9 Hz, 1H), 3.91 (s, 3H), 3.40-3.30 (m, 2H), 2.42-2.33 (m, 2H).

5. Reference

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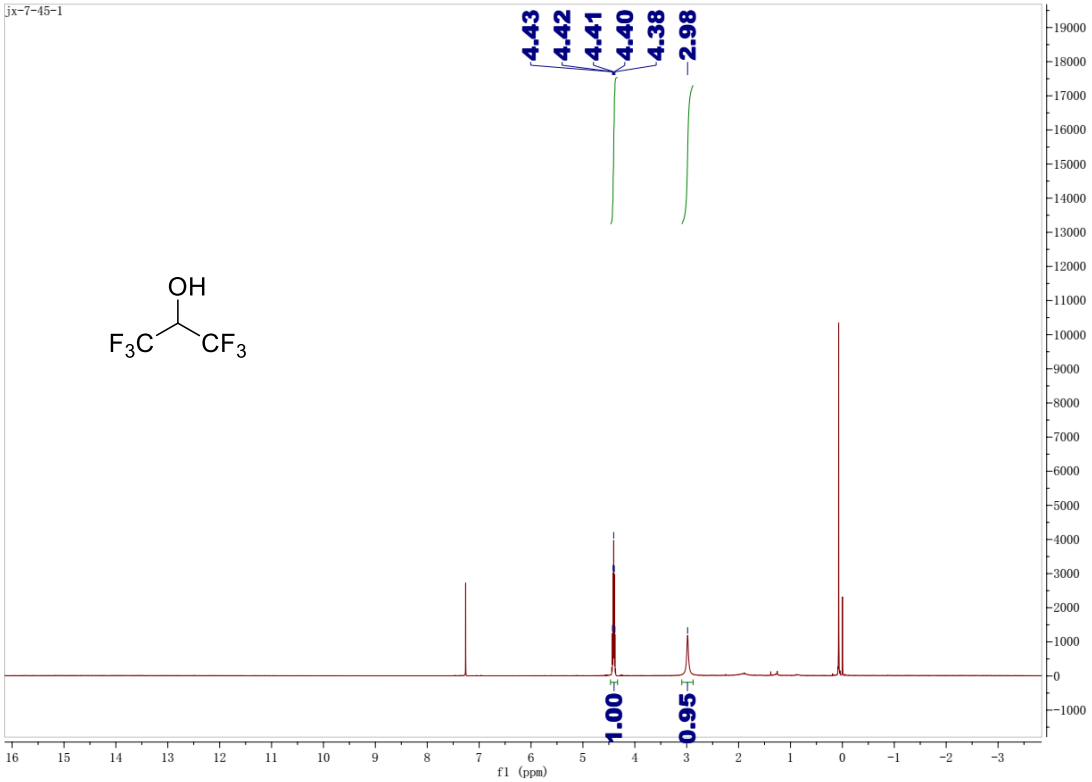
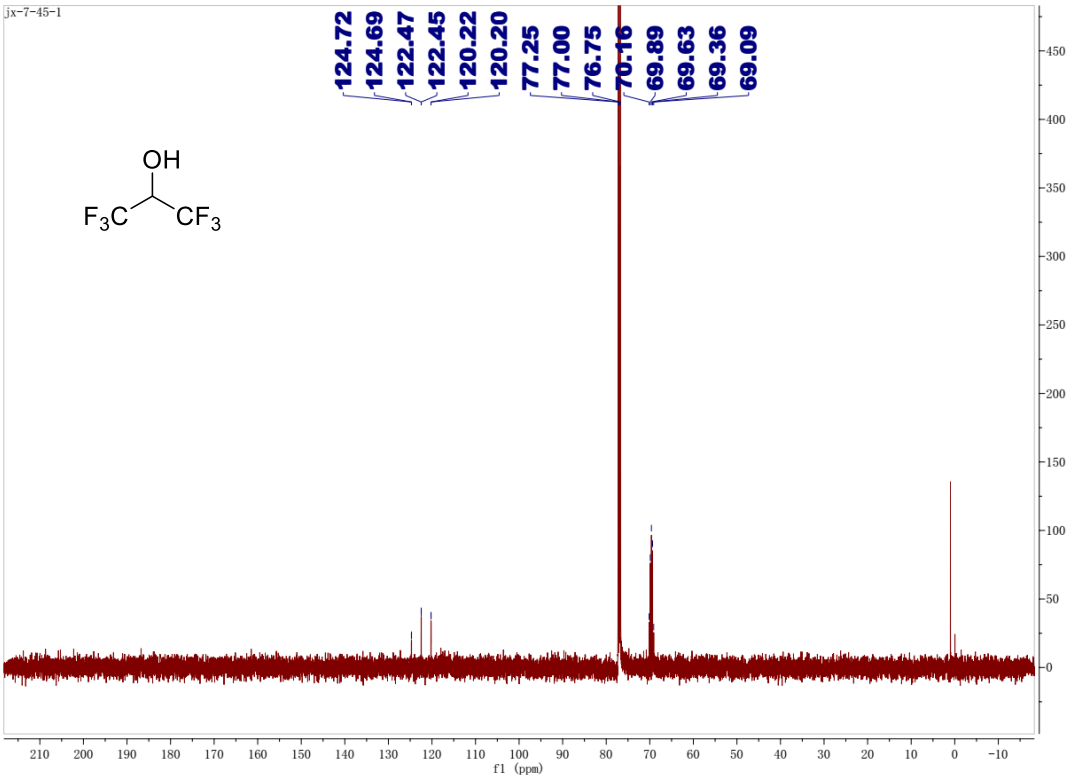
6. NMR titration experiments.

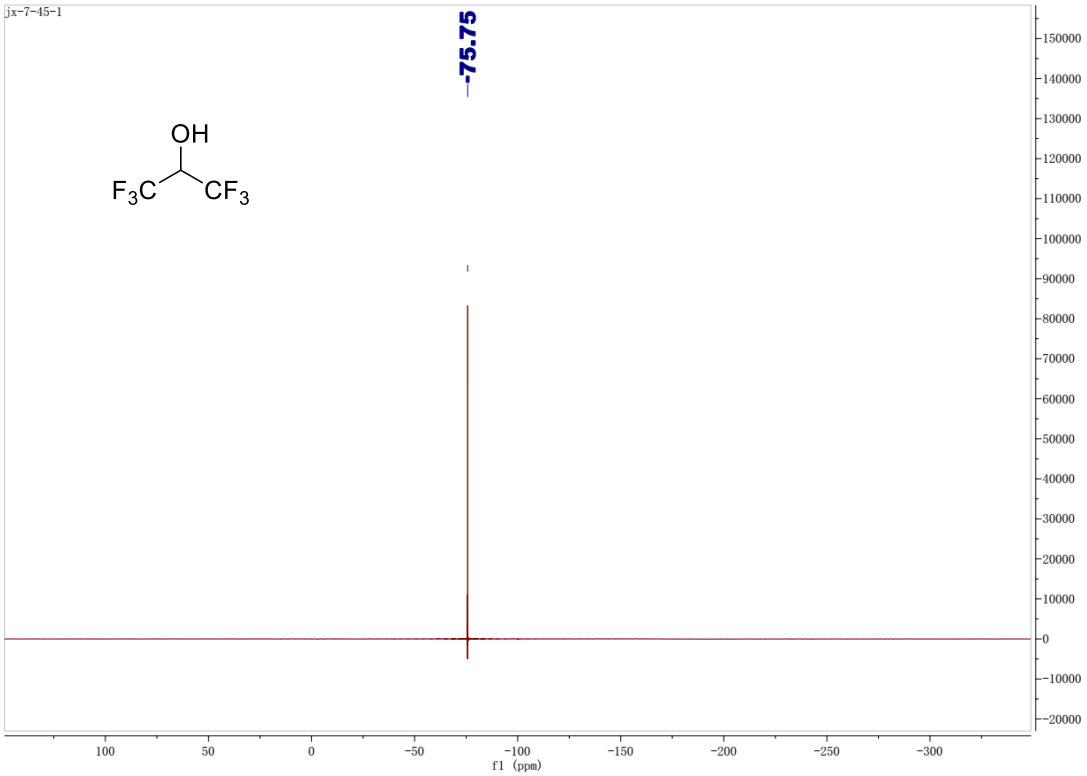
^{1}H NMR of 0.1mmol HFIP in 0.5mL $CDCl_3$



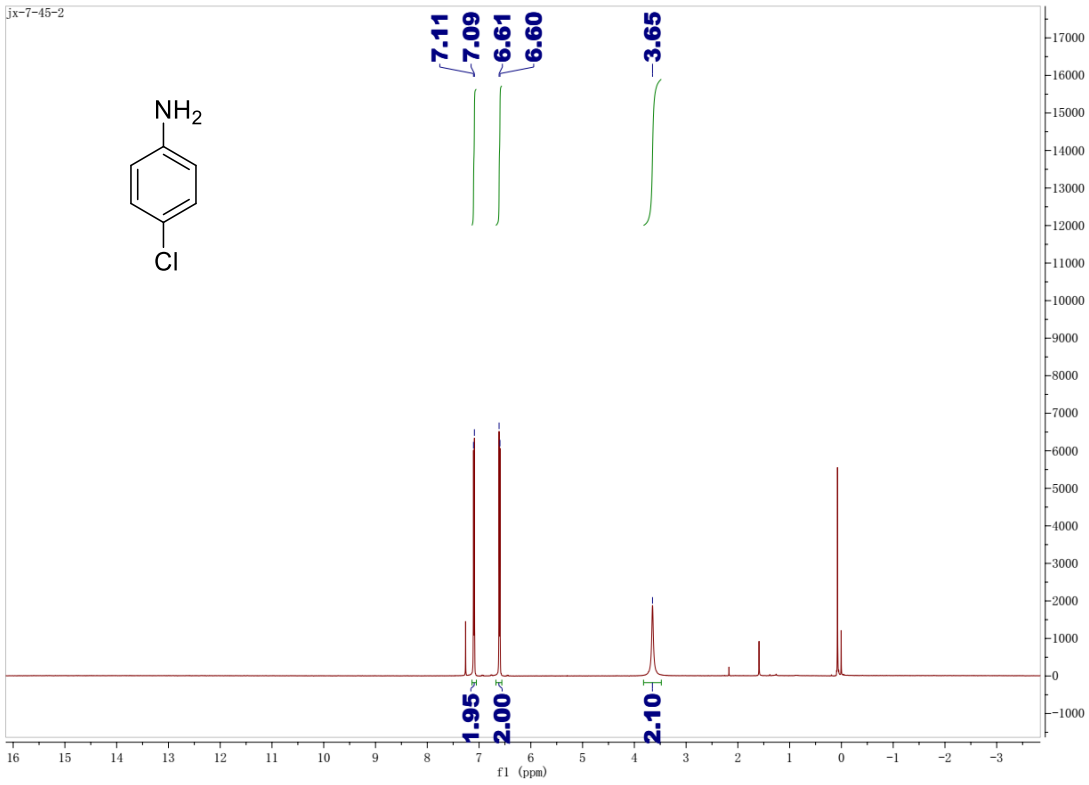
^{13}C NMR of 0.1mmol HFIP in 0.5mL $CDCl_3$



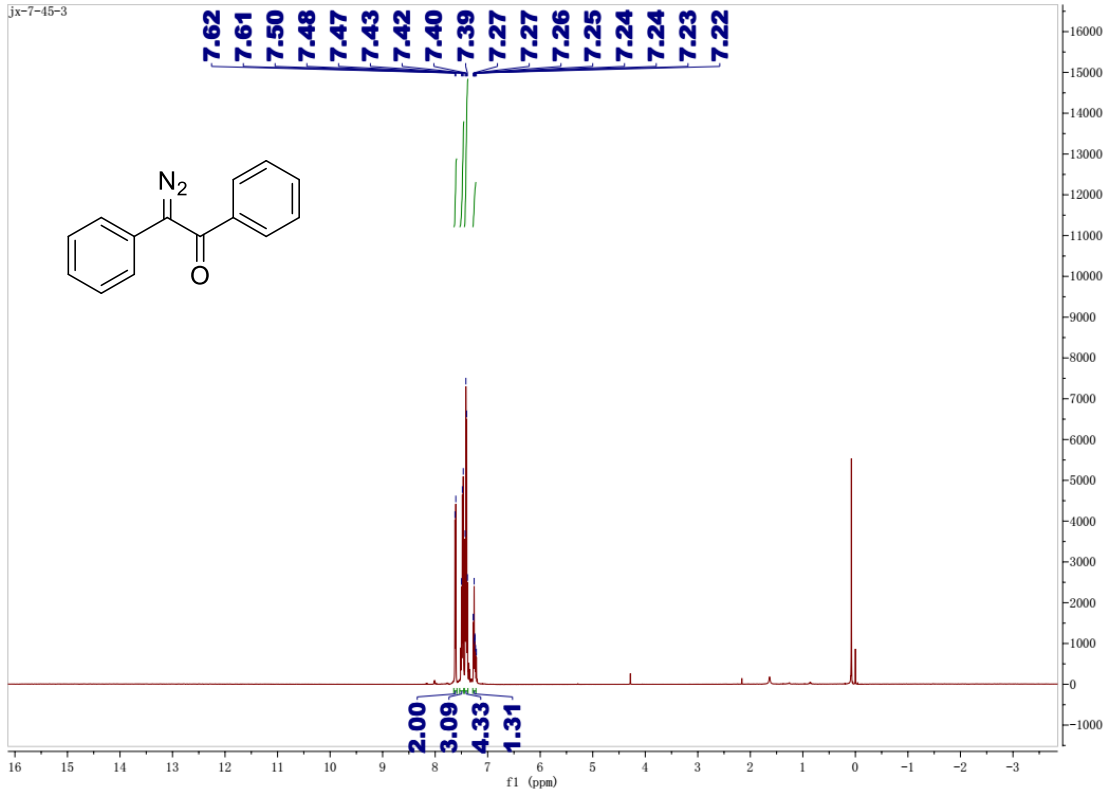
^{19}F NMR of 0.1mmol HFIP in 0.5mL $CDCl_3$



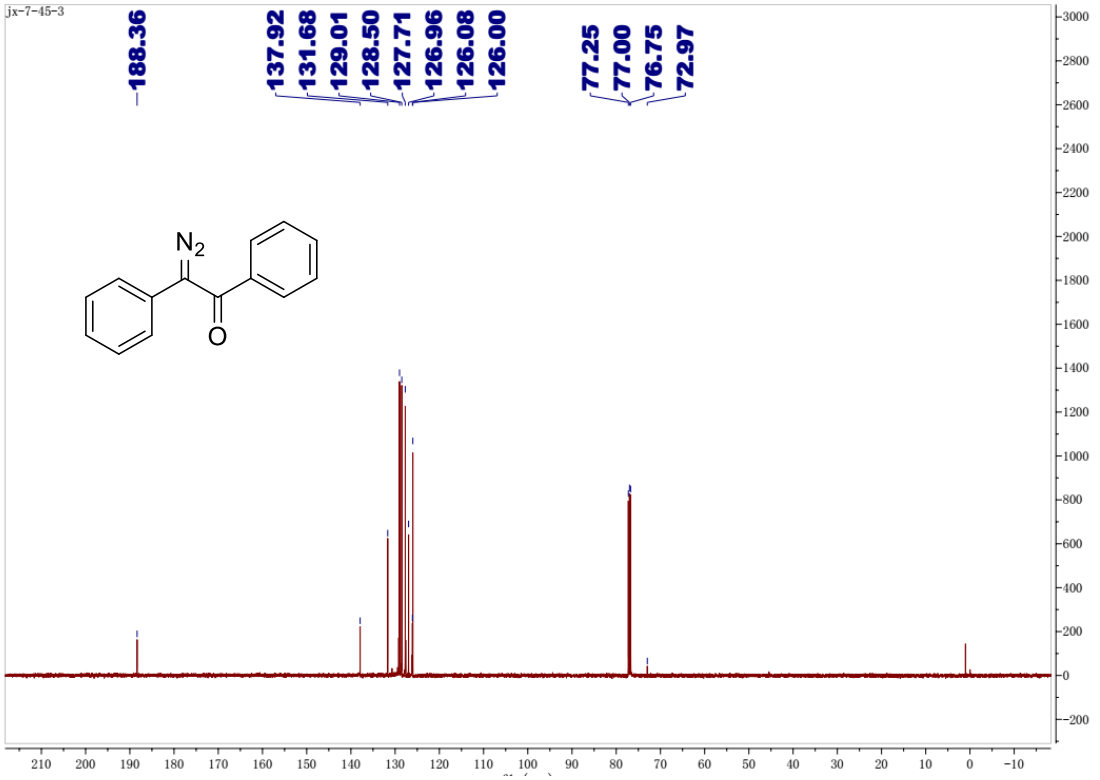
^{1}H NMR of 0.1mmol 4-chloroaniline in 0.5mL $CDCl_3$



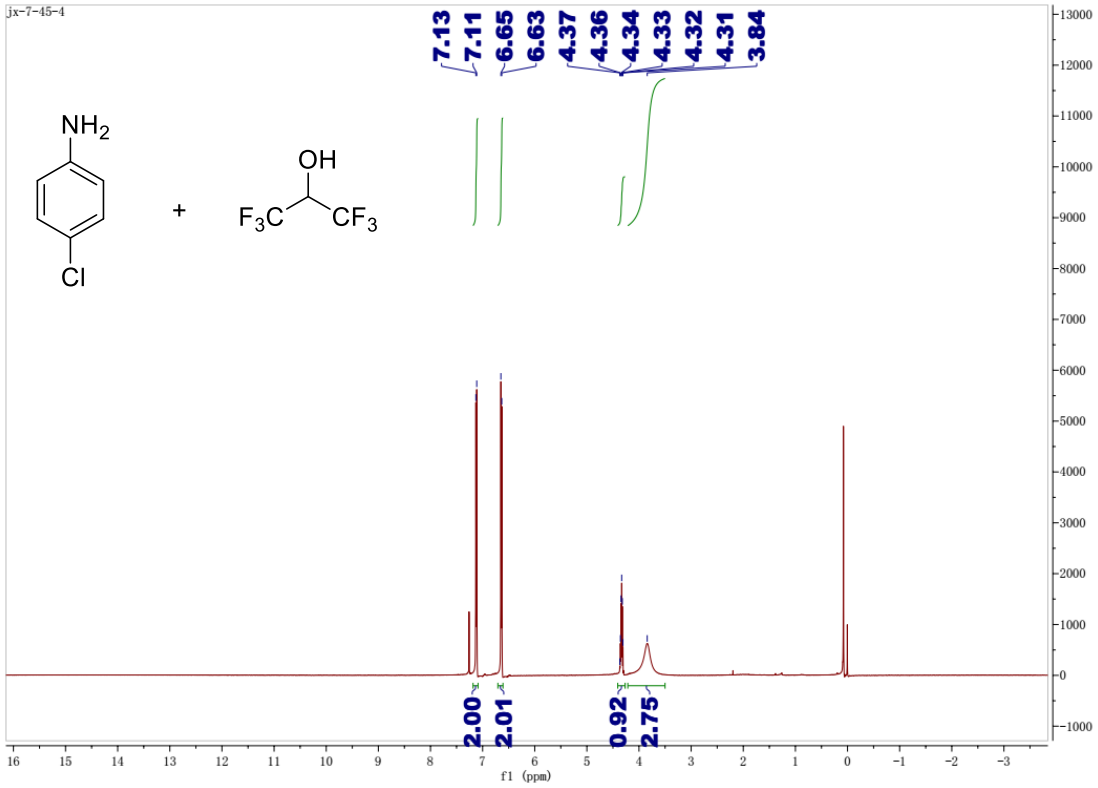
^{1}H NMR of 0.1mmol diazo **1a** in 0.5mL $CDCl_3$



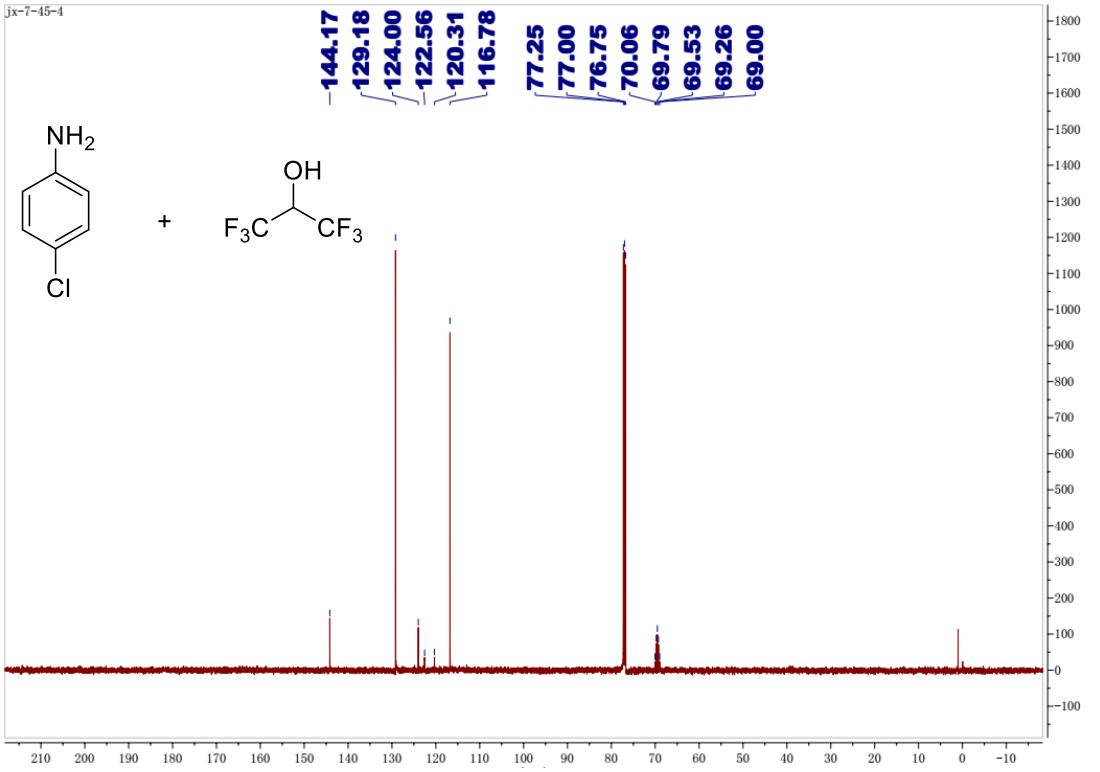
^{13}C NMR of 0.1mmol diazo **1a** in 0.5mL $CDCl_3$



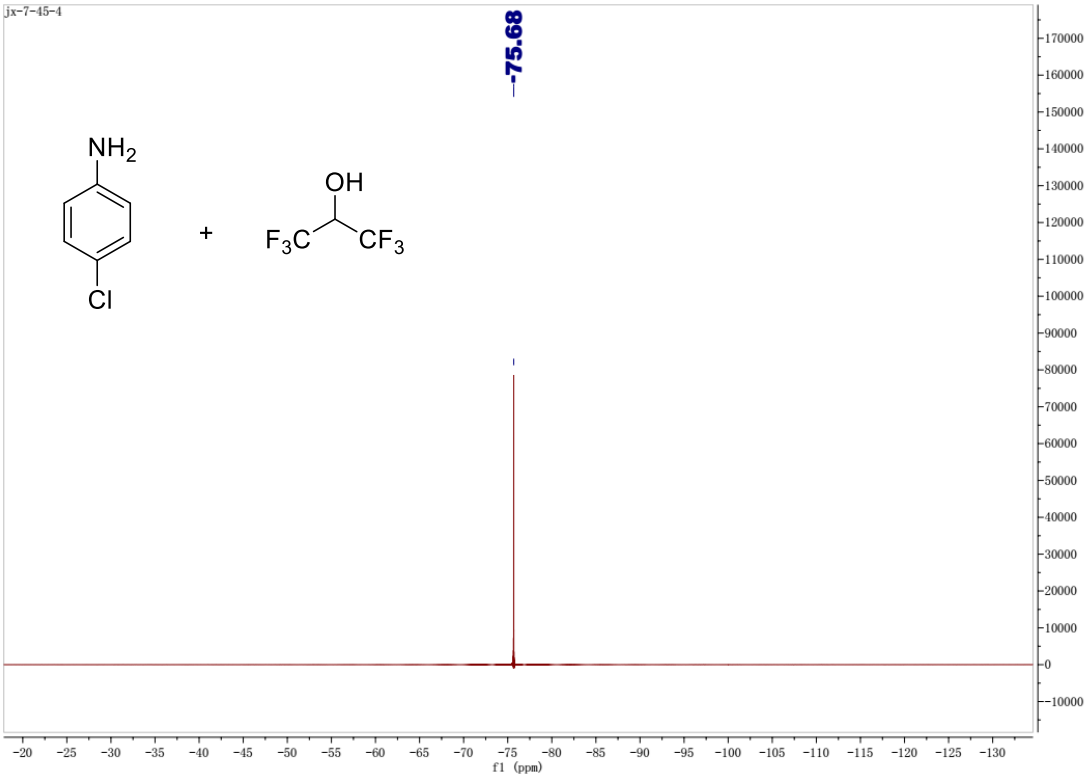
^{1}H NMR of 0.1mmol 4-chloroaniline and 0.1mmol HFIP in 0.5mL $CDCl_3$



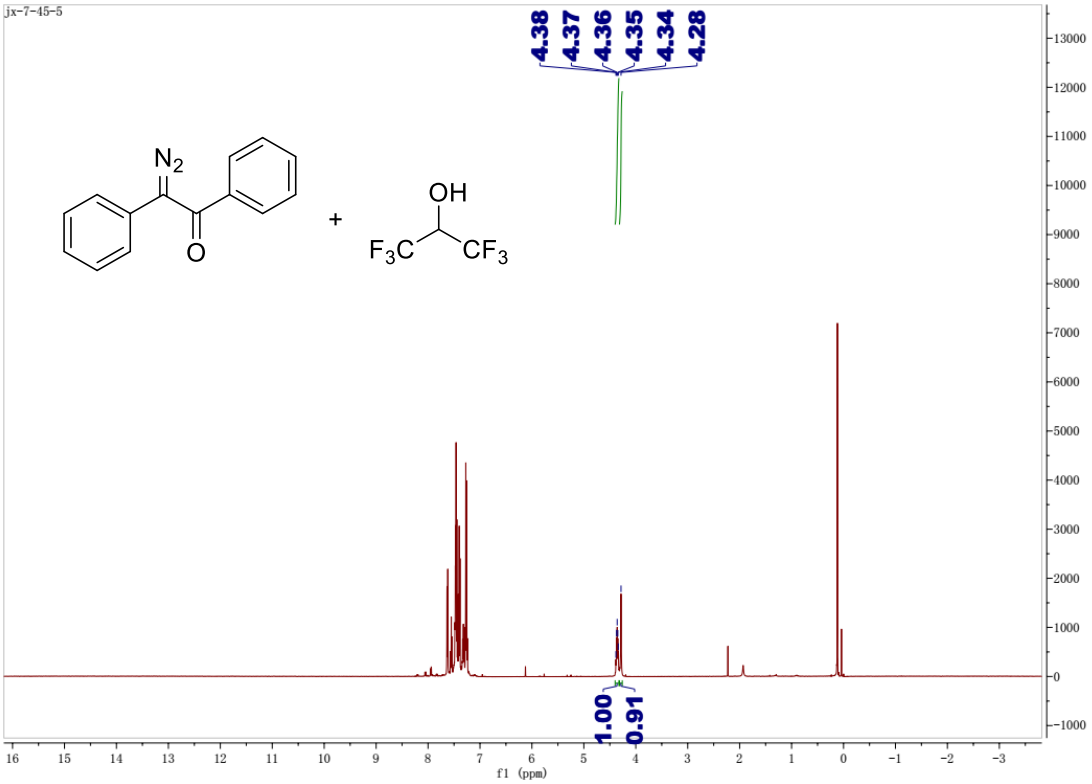
^{13}C NMR of 0.1mmol 4-chloroaniline and 0.1mmol HFIP in 0.5mL $CDCl_3$



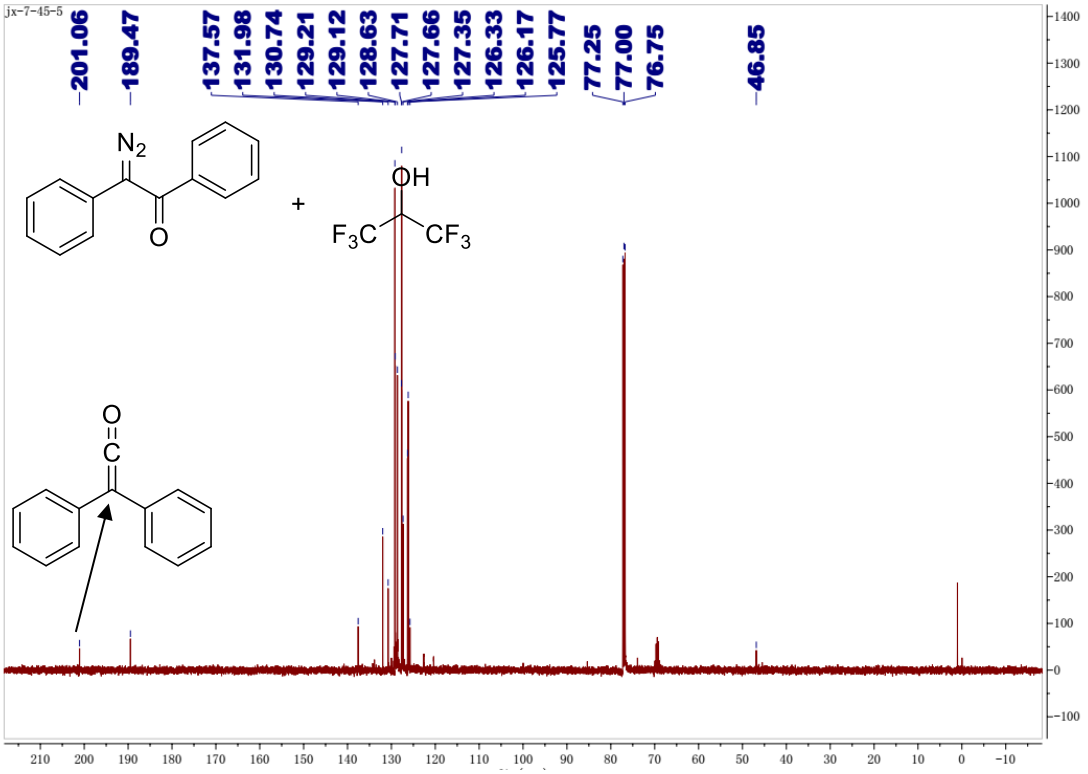
^{19}F NMR of 0.1mmol 4-chloroaniline and 0.1mmol HFIP in 0.5mL $CDCl_3$



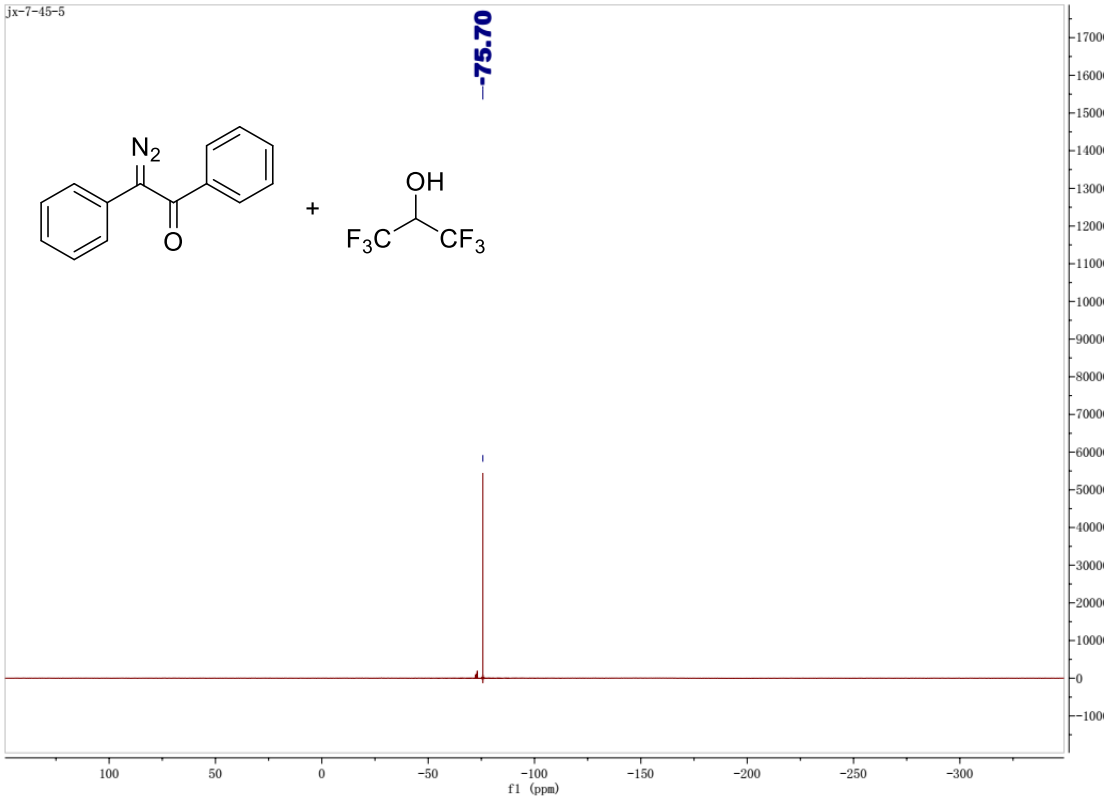
^{1}H NMR of 0.1mmol diazo **1a** and 0.1mmol HFIP in 0.5mL $CDCl_3$



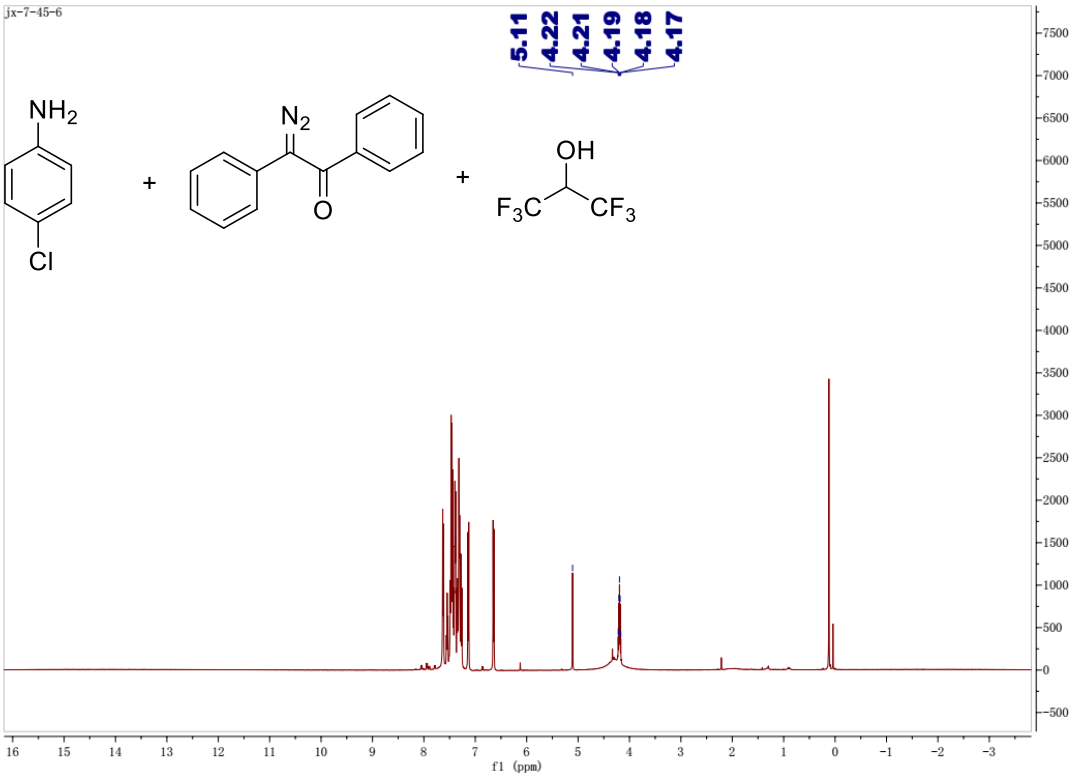
^{13}C NMR of 0.1mmol diazo **1a** and 0.1mmol HFIP in 0.5mL $CDCl_3$



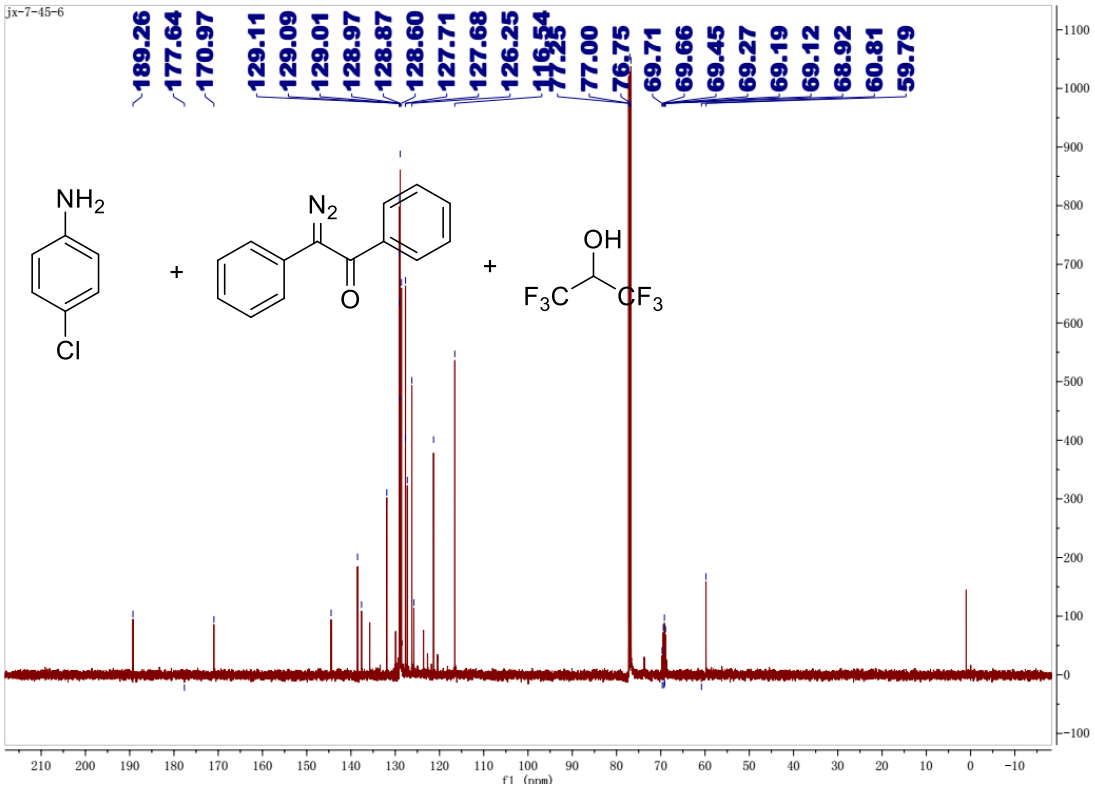
^{19}F NMR of 0.1mmol diazo **1a** and 0.1mmol HFIP in 0.5mL $CDCl_3$



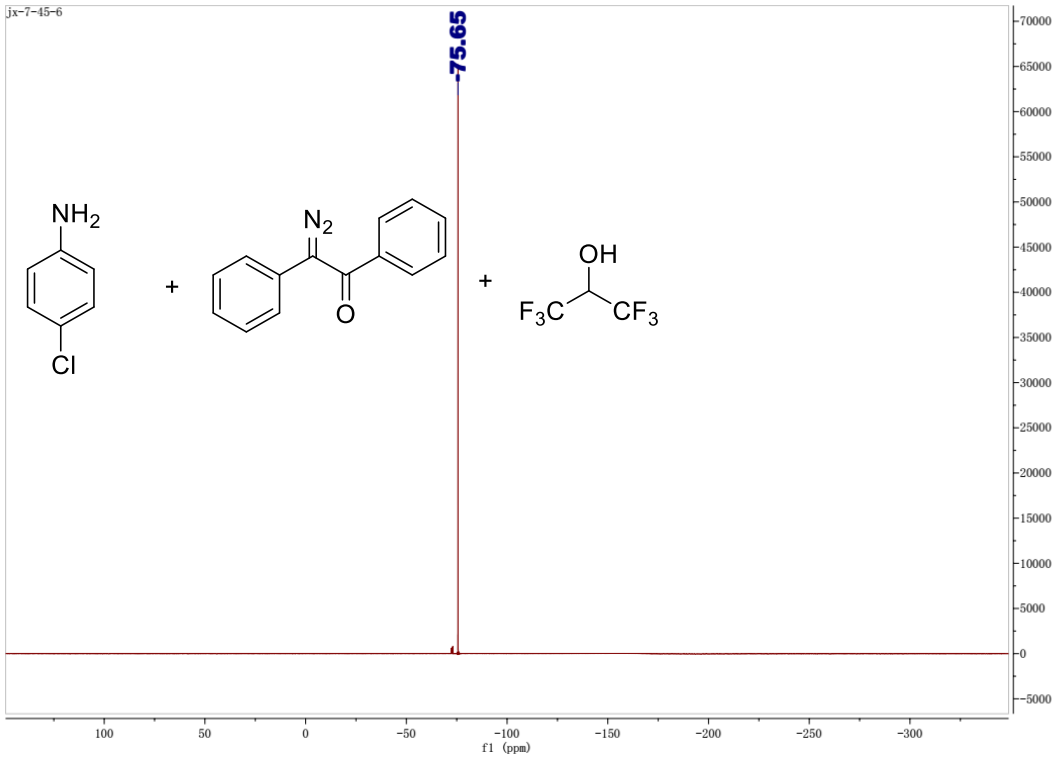
^{1}H NMR of 0.1mmol diazo **1a** , 0.1mmol 4-chloroaniline and 0.1mmol HFIP in 0.5mL $CDCl_3$



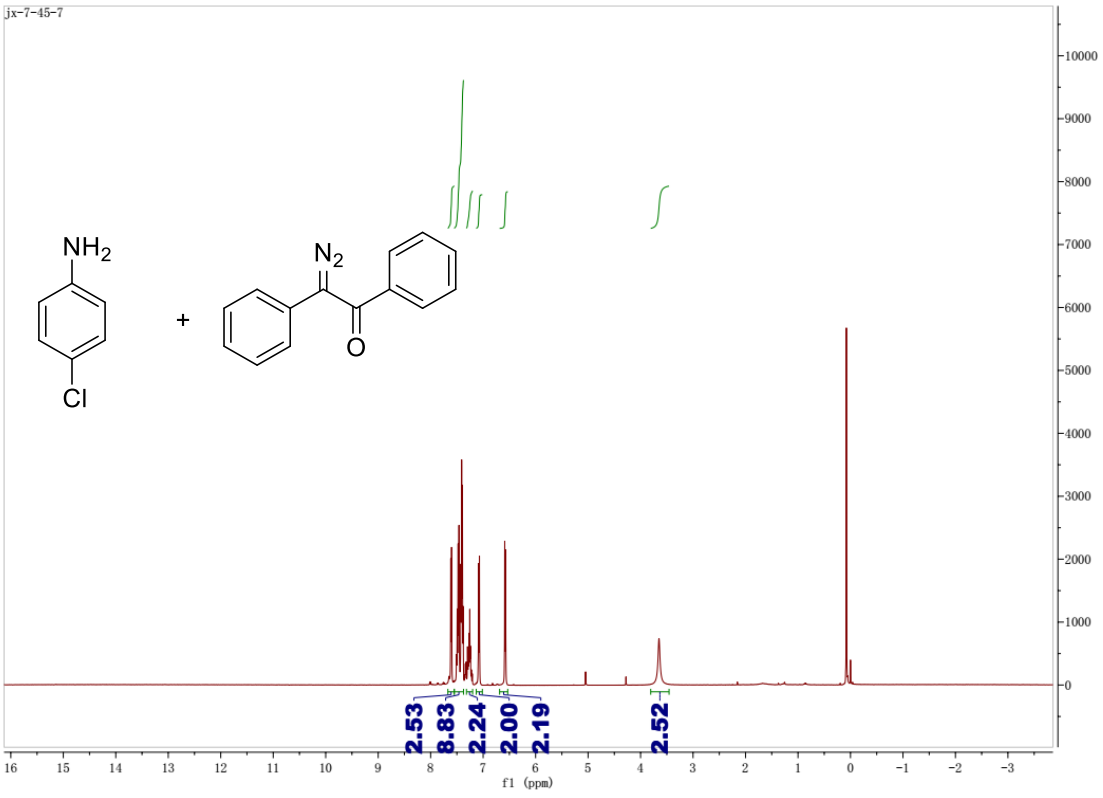
^{13}C NMR of 0.1mmol diazo **1a** , 0.1mmol 4-chloroaniline and 0.1mmol HFIP in 0.5mL $CDCl_3$



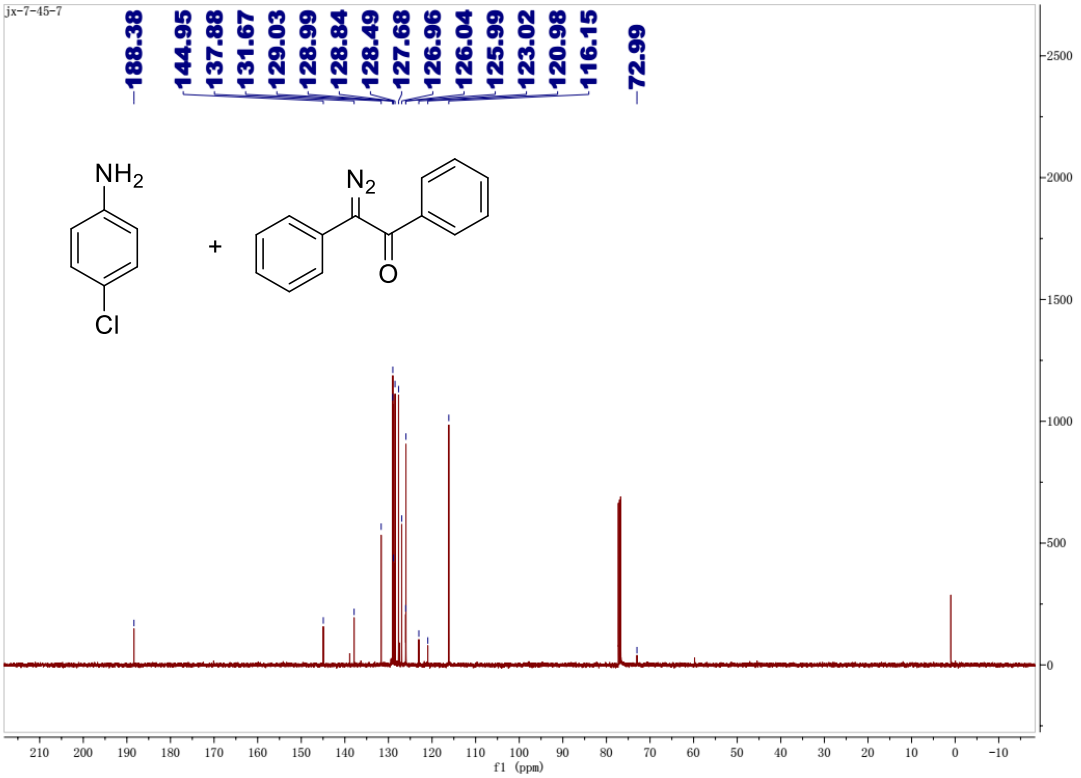
^{19}F NMR of 0.1mmol diazo **1a** , 0.1mmol 4-chloroaniline and 0.1mmol HFIP in 0.5mL $CDCl_3$

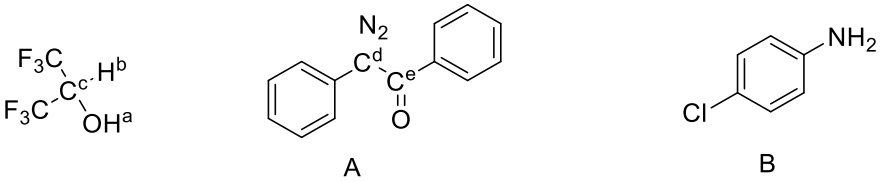


^{1}H NMR of 0.1mmol diazo **1a** and 0.1mmol 4-chloroaniline in 0.5mL $CDCl_3$



^{13}C NMR of 0.1mmol diazo **1a** and 0.1mmol 4-chloroaniline in 0.5mL $CDCl_3$



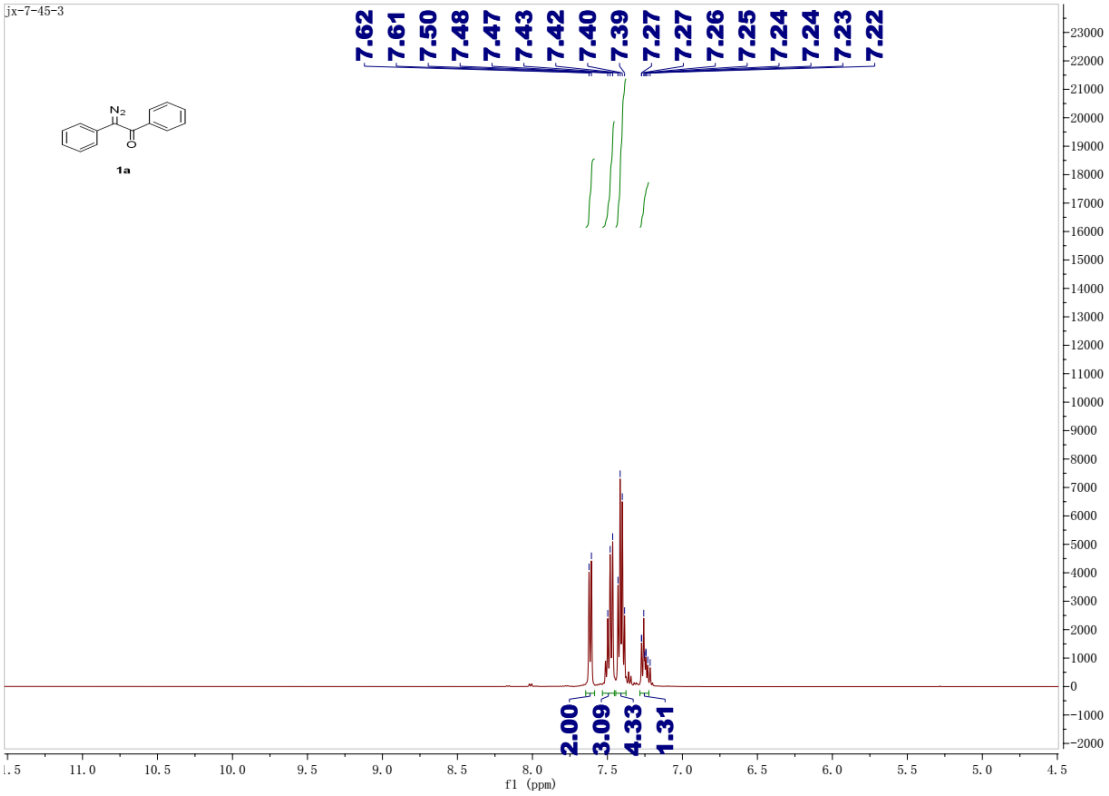


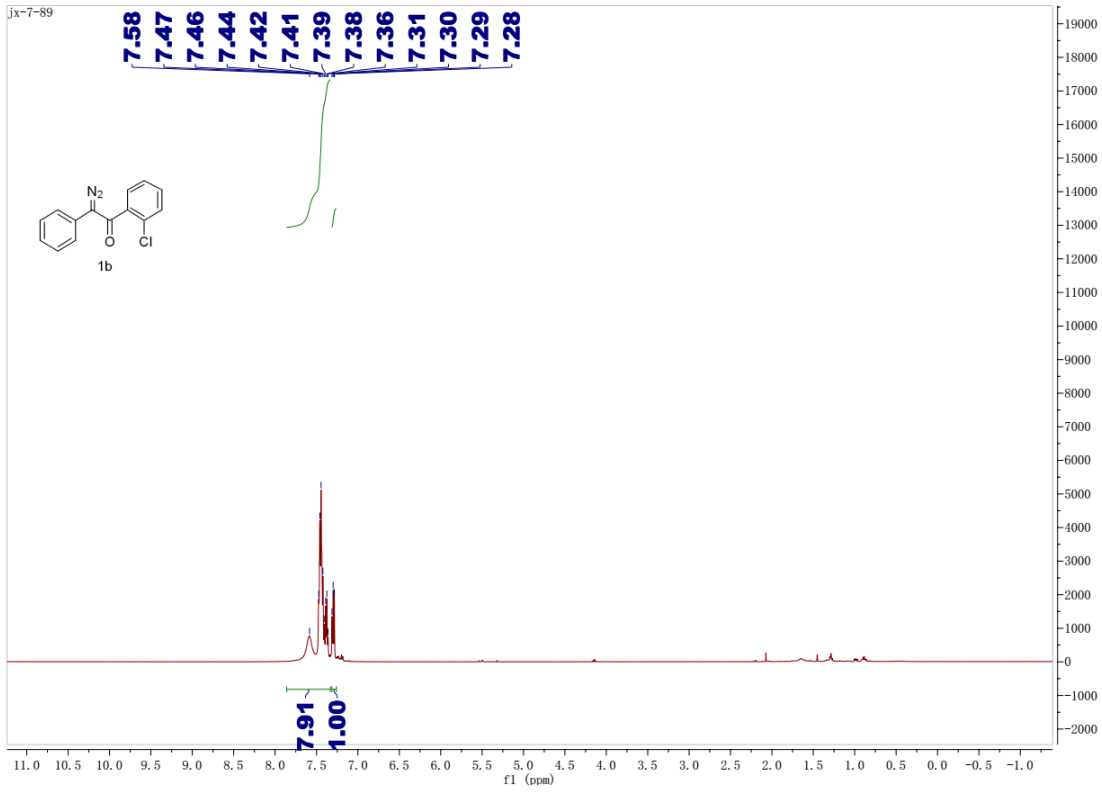
A B

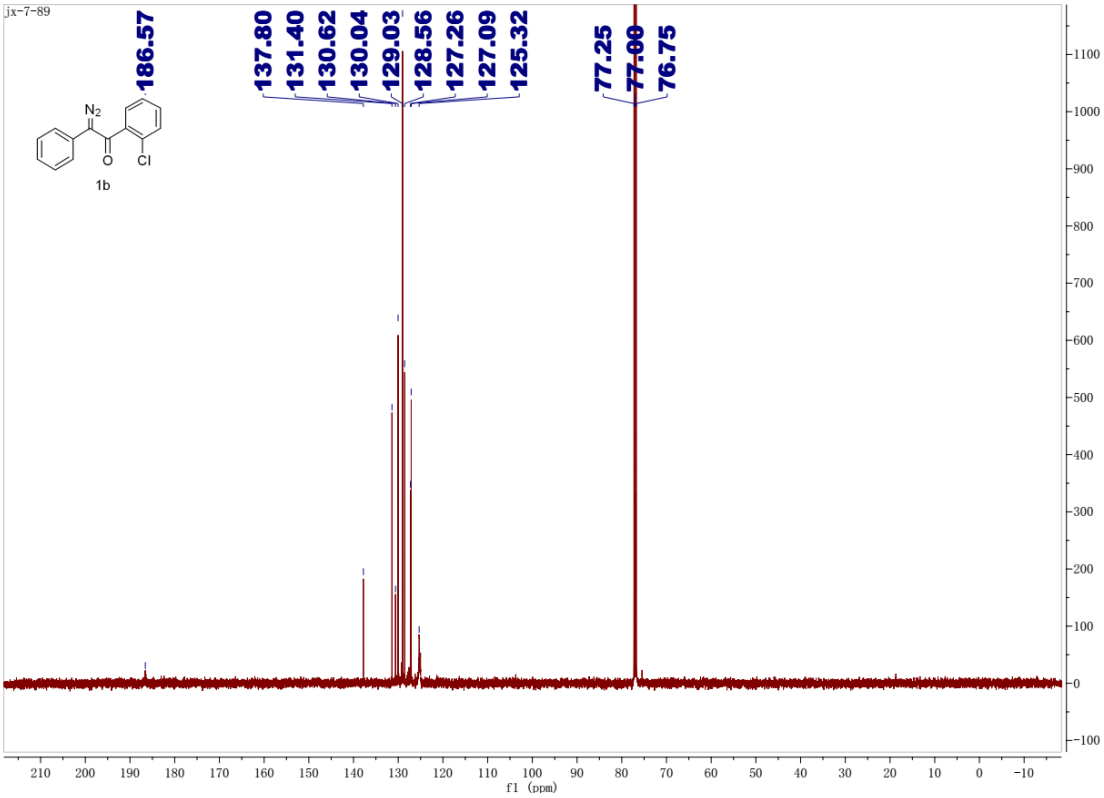
Compound	δ (ppm) H^a	Δδ (ppm) H^a	δ (ppm) H^b	Δδ (ppm) H^b	δ (ppm) F	Δδ (ppm) F
HFIP	2.98	-	4.41	-	77.75	-
HFIP+B	3.84	0.86	4.33	-0.08	75.68	-0.07
HFIP+A	4.26	1.28	4.33	-0.08	75.70	-0.05
HFIP+A+B	-	-	4.17	-0.24	75.65	-0.1

Compound	δ (ppm) C^c	Δδ (ppm) C^c	δ (ppm) C^d	Δδ (ppm) C^c	δ (ppm) C^e	Δδ (ppm) C^e
HFIP	69.63	-	-	-	-	-
A	-	-	72.97	-	188.36	-
HFIP+A	69.42	-0.21	73.92	0.95	189.49	1.13
HFIP+A+B	69.19	-0.44	73.73	0.76	189.26	0.9
A+B	-	-	72.99	0.02	188.38	0.02

7. NMR Spectra of new compound



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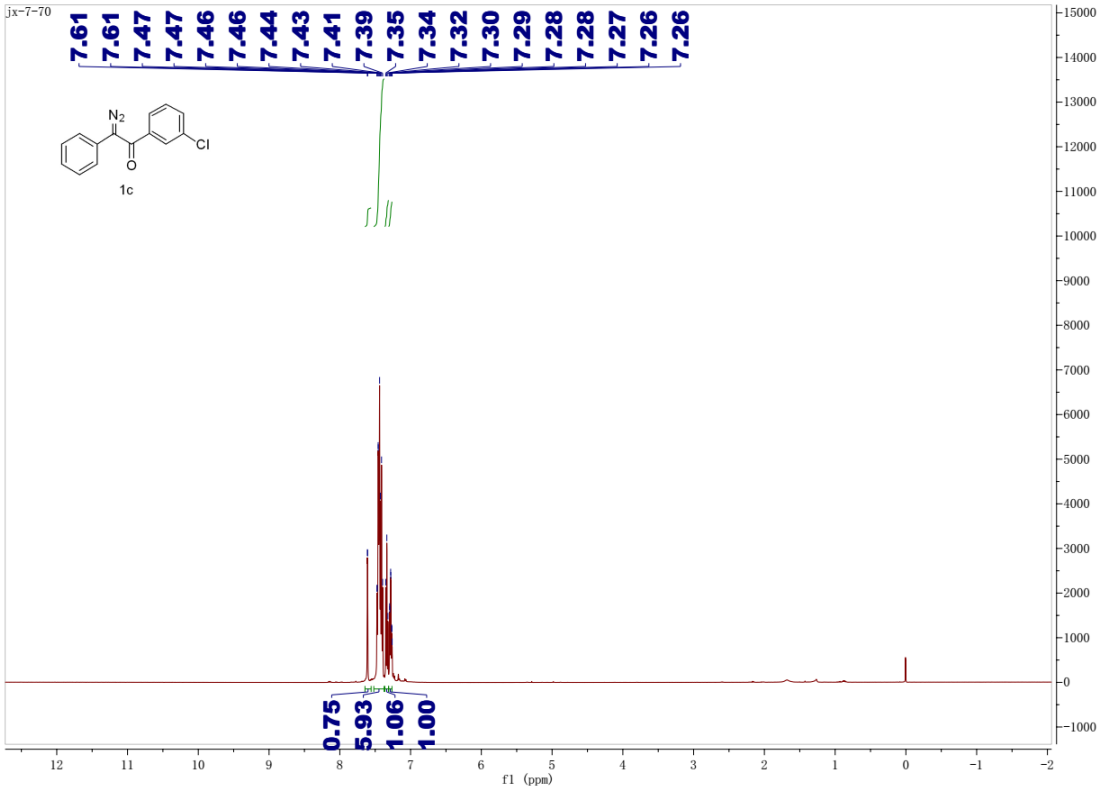
137.80
131.40
130.62
130.04
129.03
128.56
127.26
127.09
125.32
77.25
77.00
76.75
N2
O
Cl
1b
f1 (ppm)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7.34
7.32
7.30
7.29
7.28
7.28
7.27
7.26
7.26
N2
O
Cl
1c
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5.93
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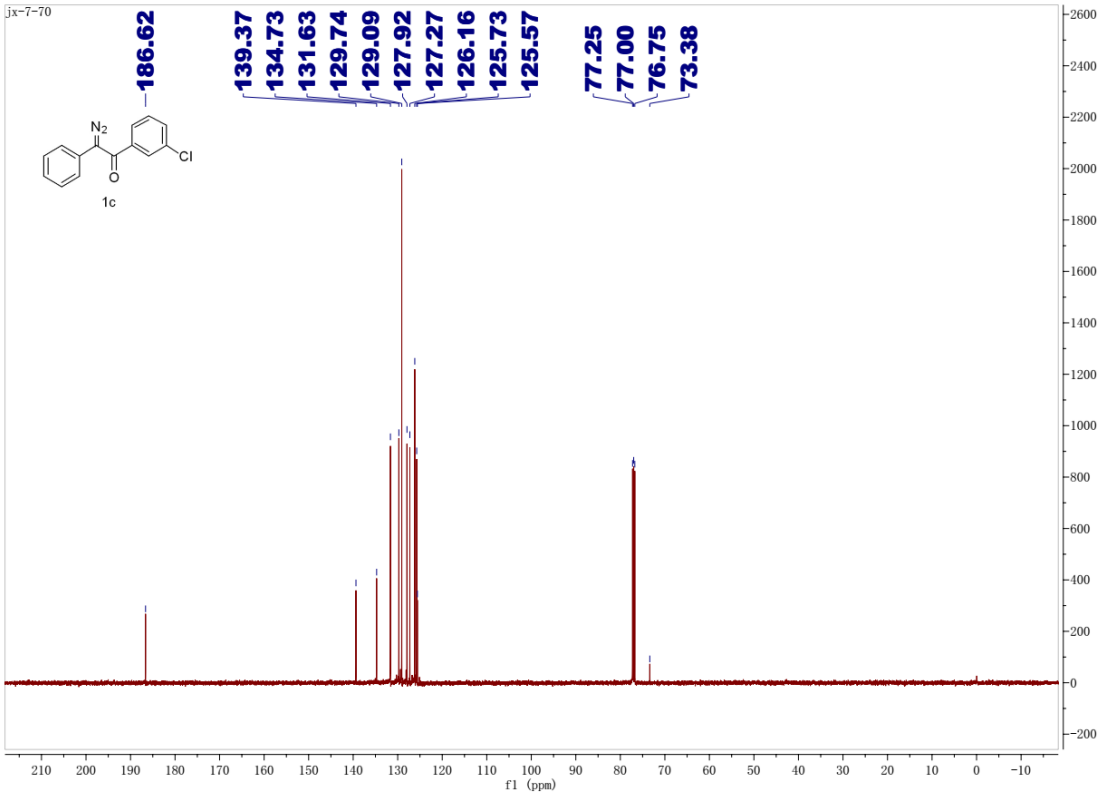
1.00
f1 (ppm)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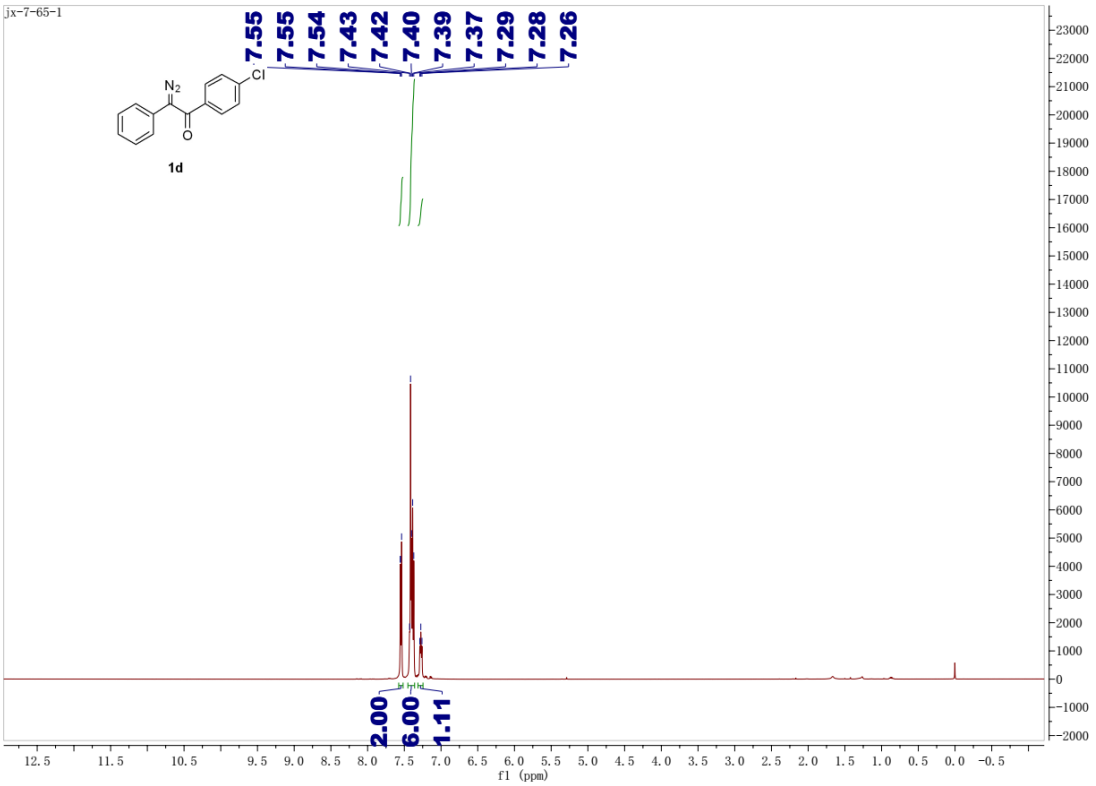
N2
Cl
O
1c
f1 (ppm)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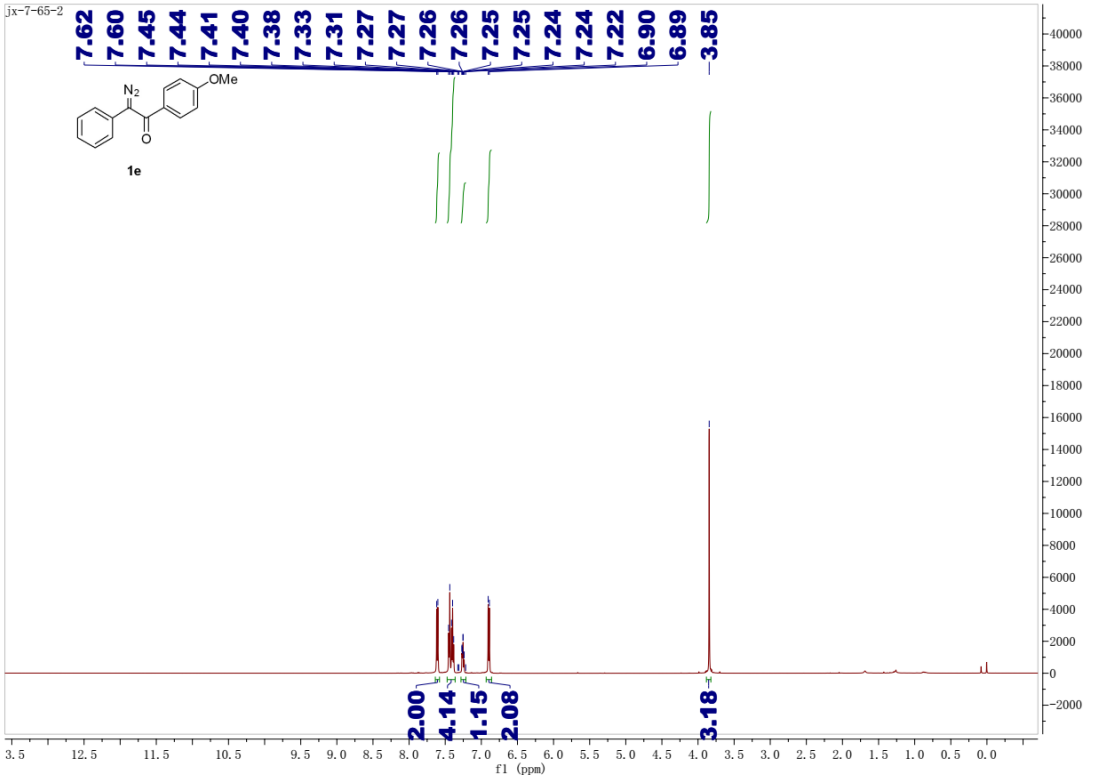
f1 (ppm)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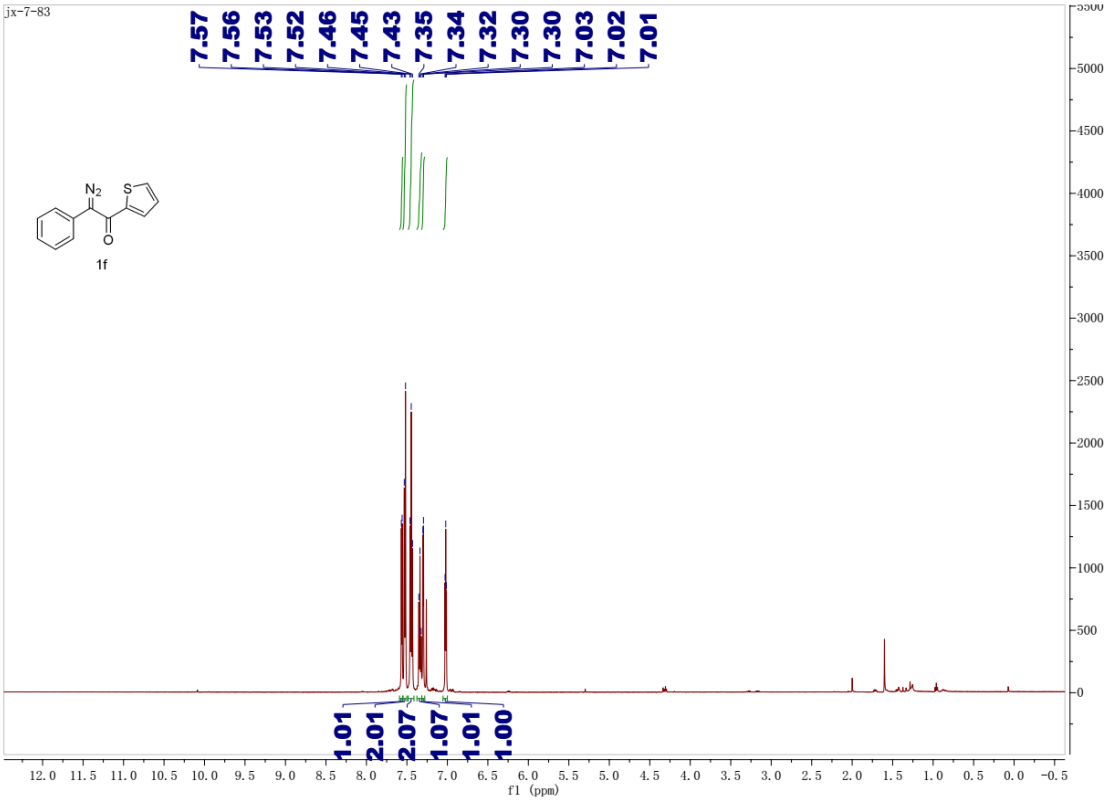
3.18
f1 (ppm)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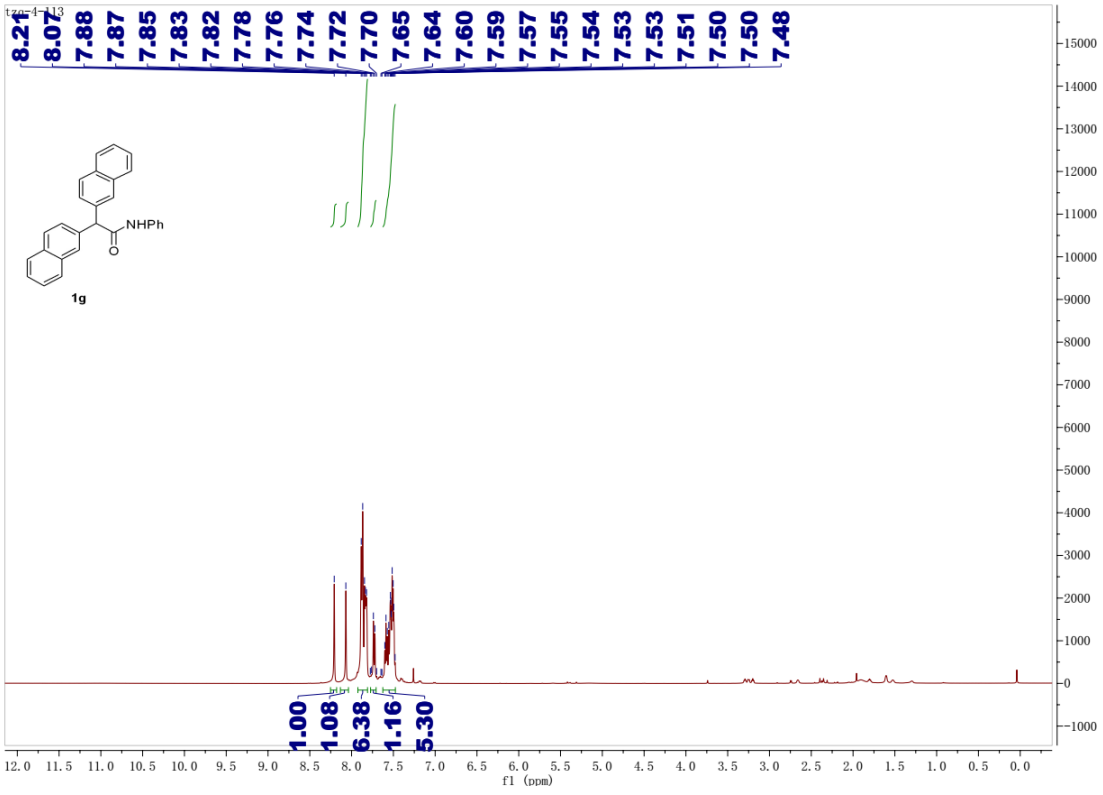
2.07
1.07
1.01
1.00
f1 (ppm)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7.60
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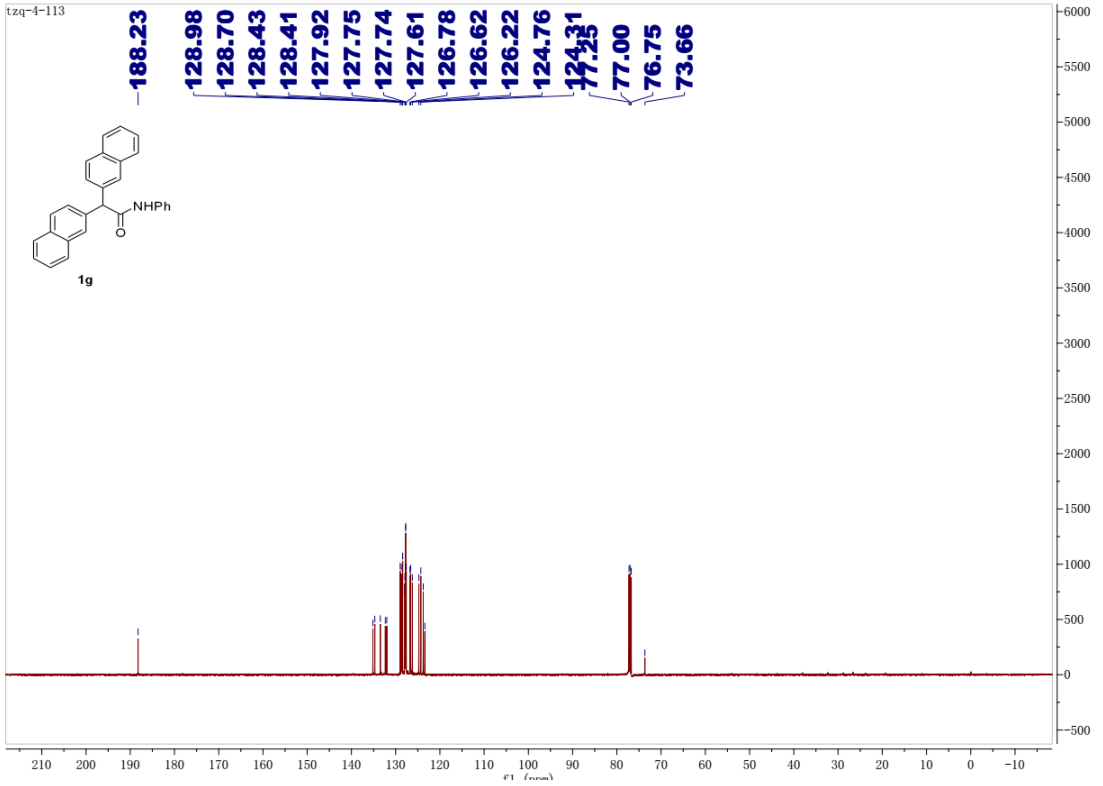
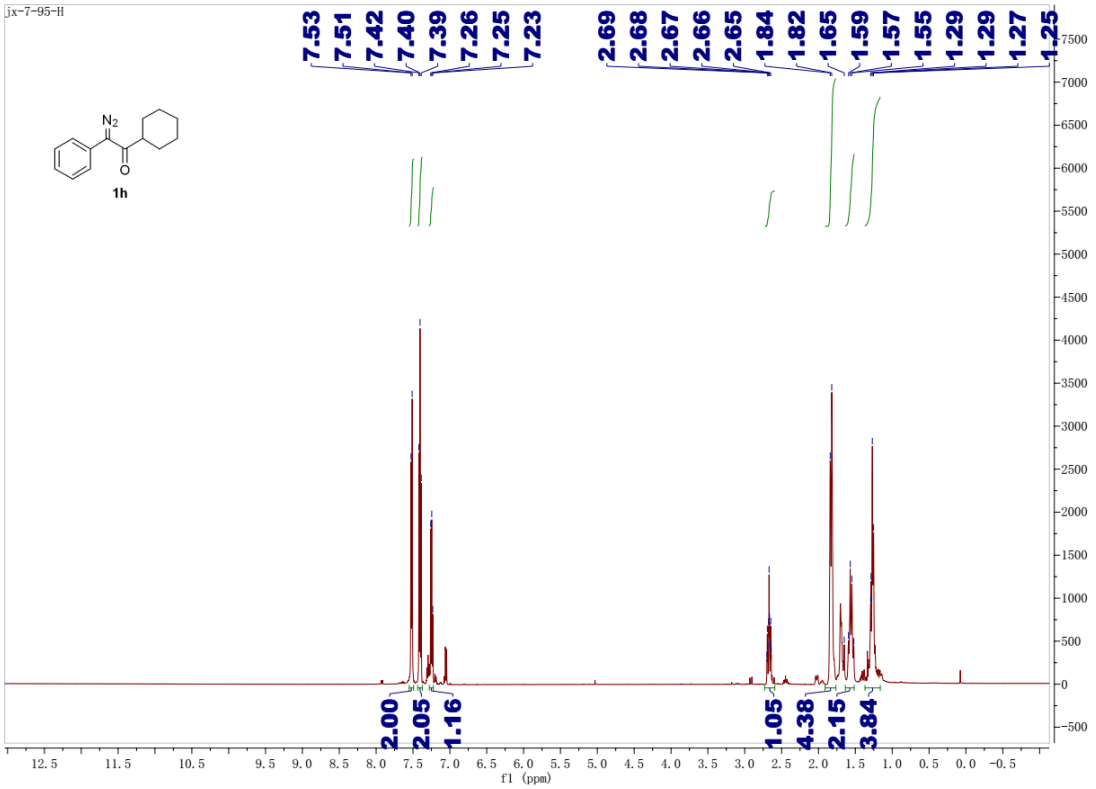
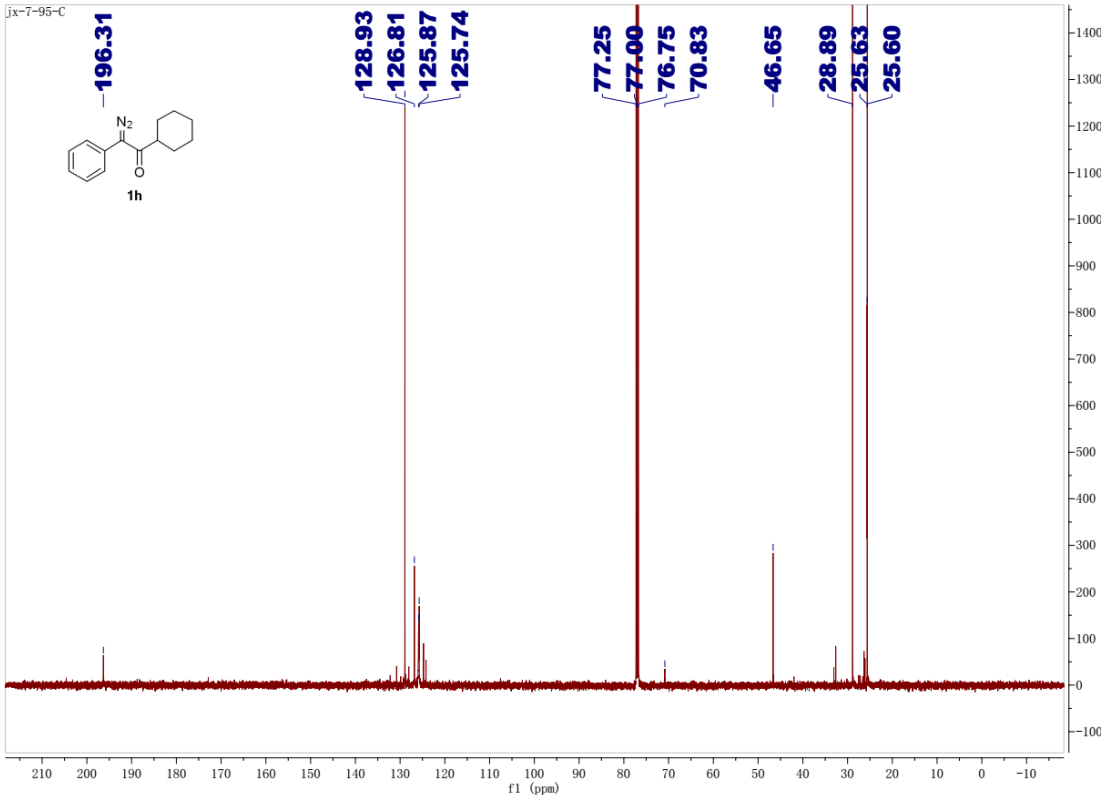
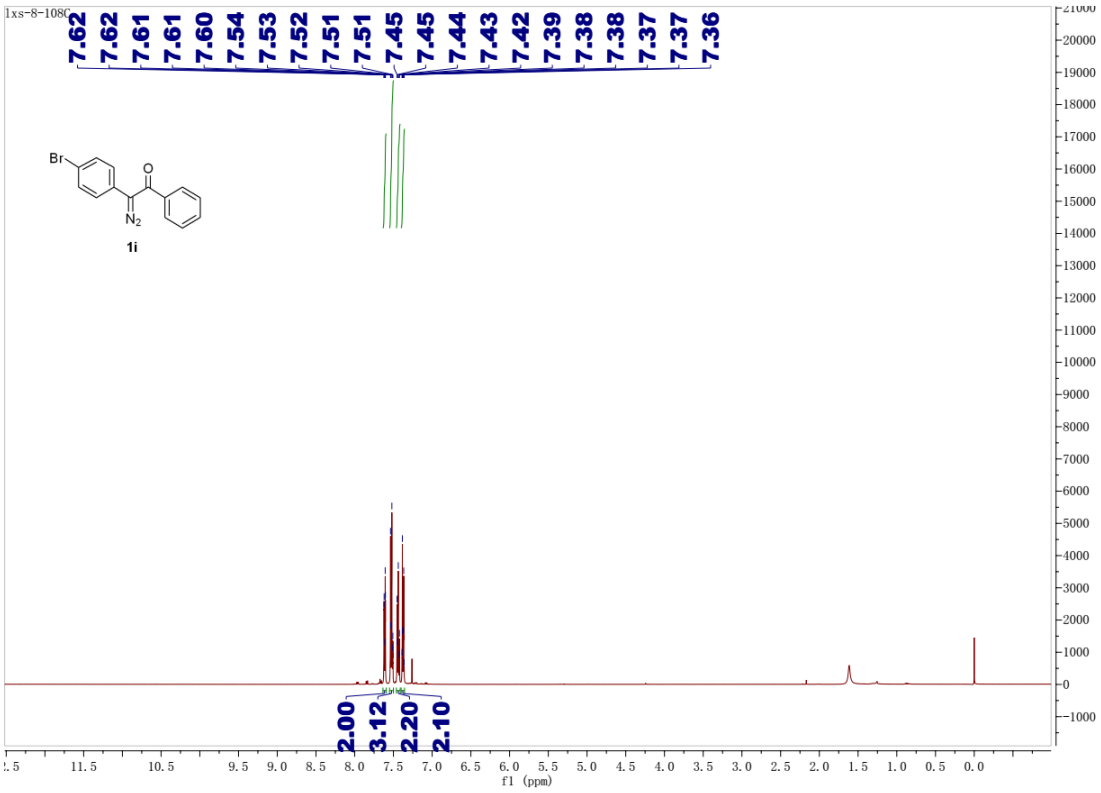
7.55
7.54
7.53
7.53
7.51
7.50
7.50
7.48
NHPh
O
1g
1.00
1.08
6.38
1.16
5.30
f1 (ppm)


tzq-4-113
188.23
128.98
128.70
128.43
128.41
127.92
127.75
127.74
127.61
126.78
126.62
126.22
124.76
124.31
77.25
77.00
76.75
73.66
NHPh
O
1g


jx-7-95-H
7.53
7.51
7.42
7.40
7.39
7.26
7.25
7.23
2.69
2.68
2.67
2.66
2.65
1.84
1.82
1.65
1.59
1.57
1.55
1.29
1.29
1.27
1.25
1h
2.00
2.05
1.16
1.05
4.38
2.15
3.84
f1 (ppm)


jx-7-95-C
196.31
128.93
126.81
125.87
125.74
77.25
77.00
76.75
70.83
46.65
28.89
25.63
25.60
1h
f1 (ppm)


7.62
7.62
7.61
7.61
7.60
7.54
7.53
7.52
7.51
7.51
7.45
7.45
7.44
7.43
7.42
7.39
7.38
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Br
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N2
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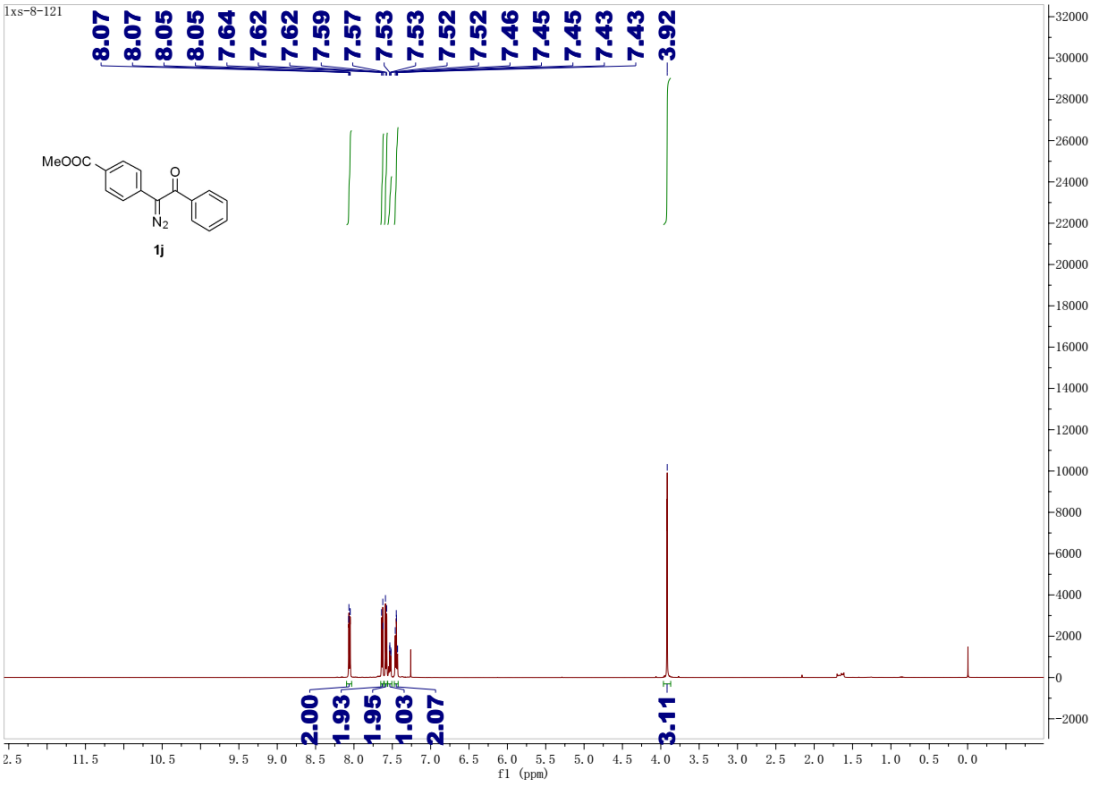
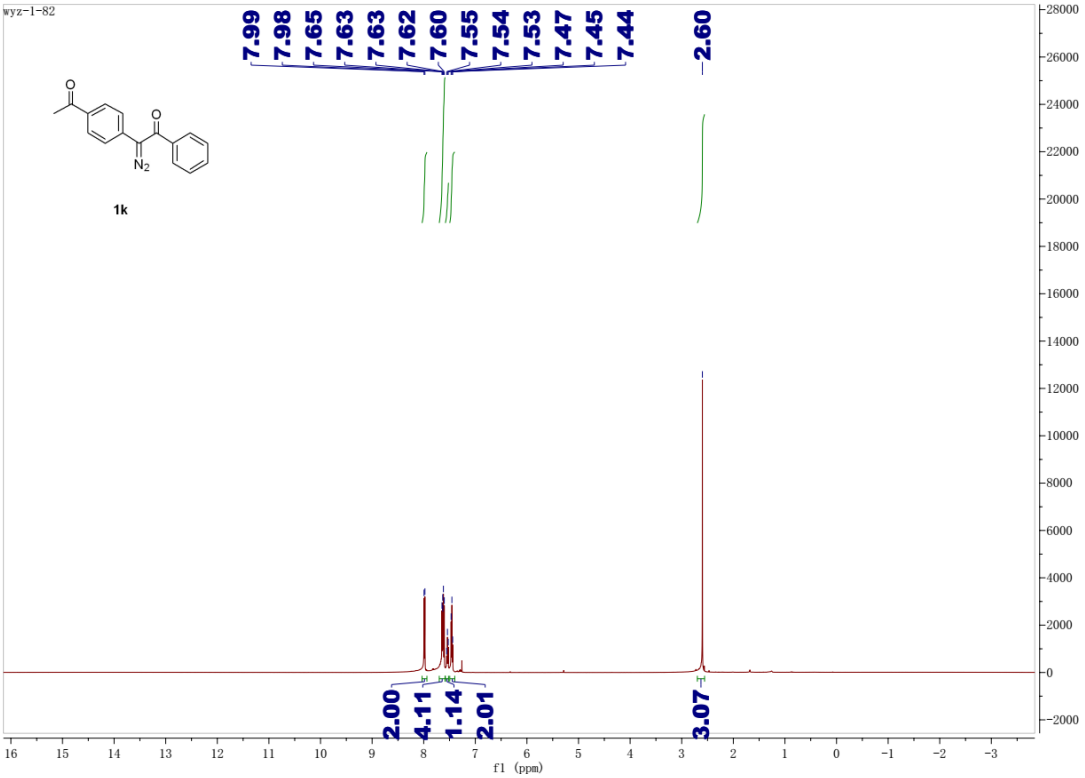
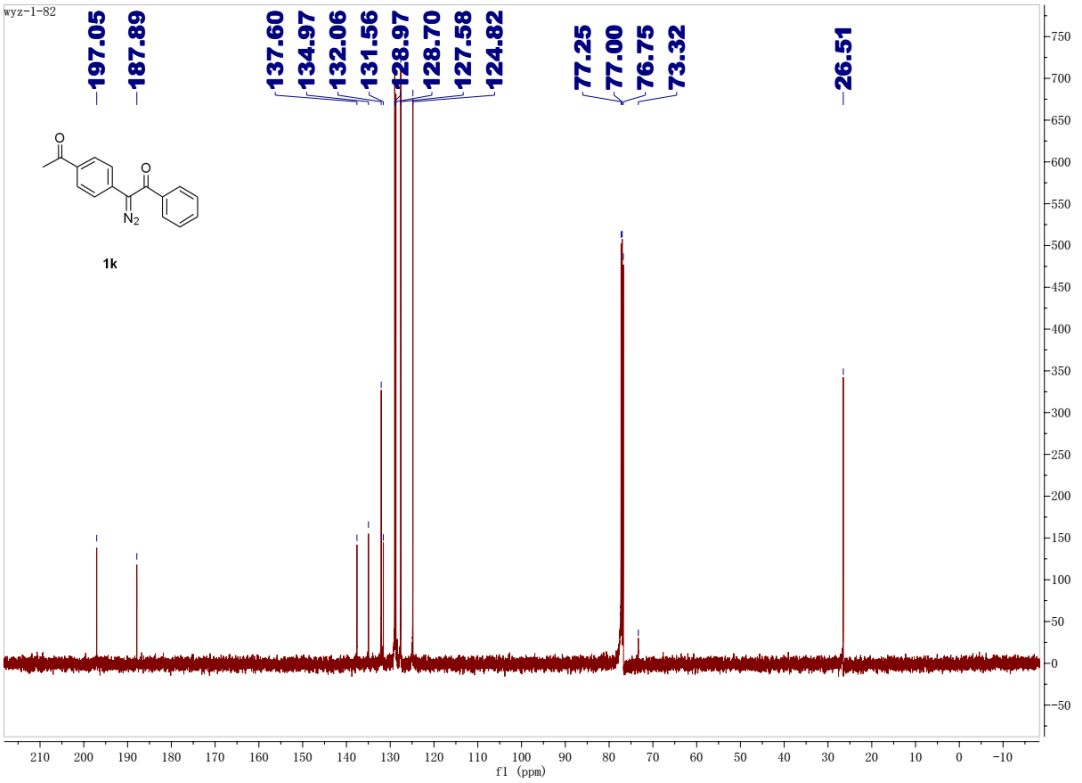
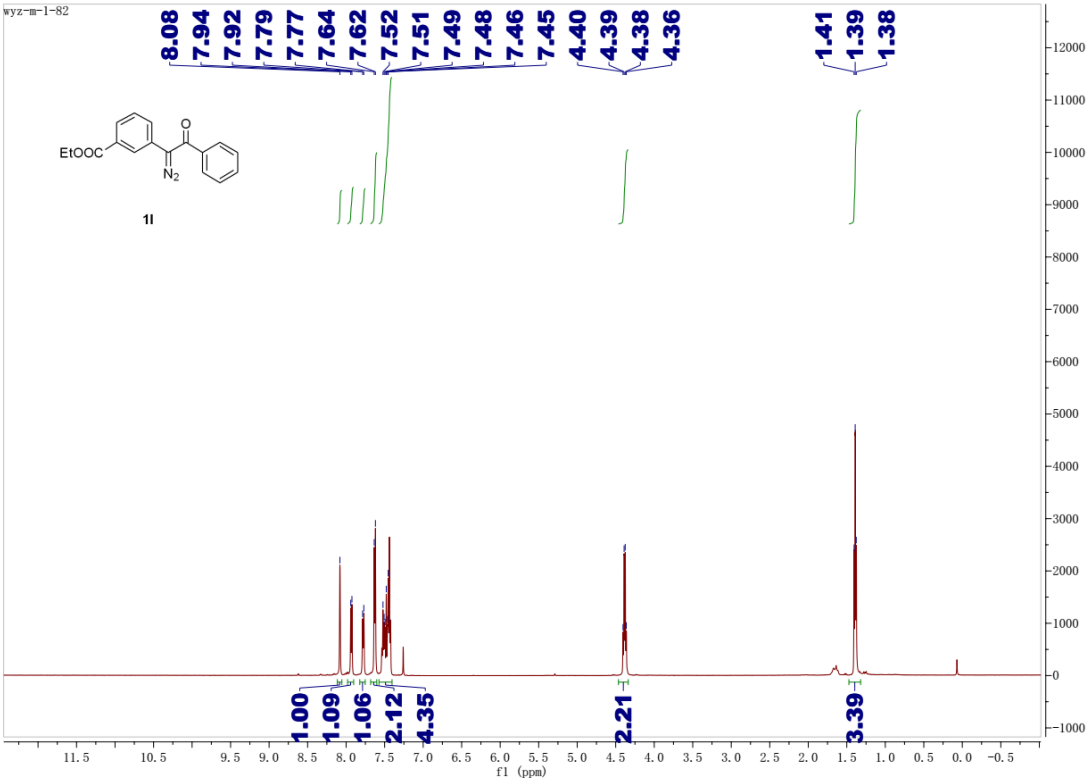
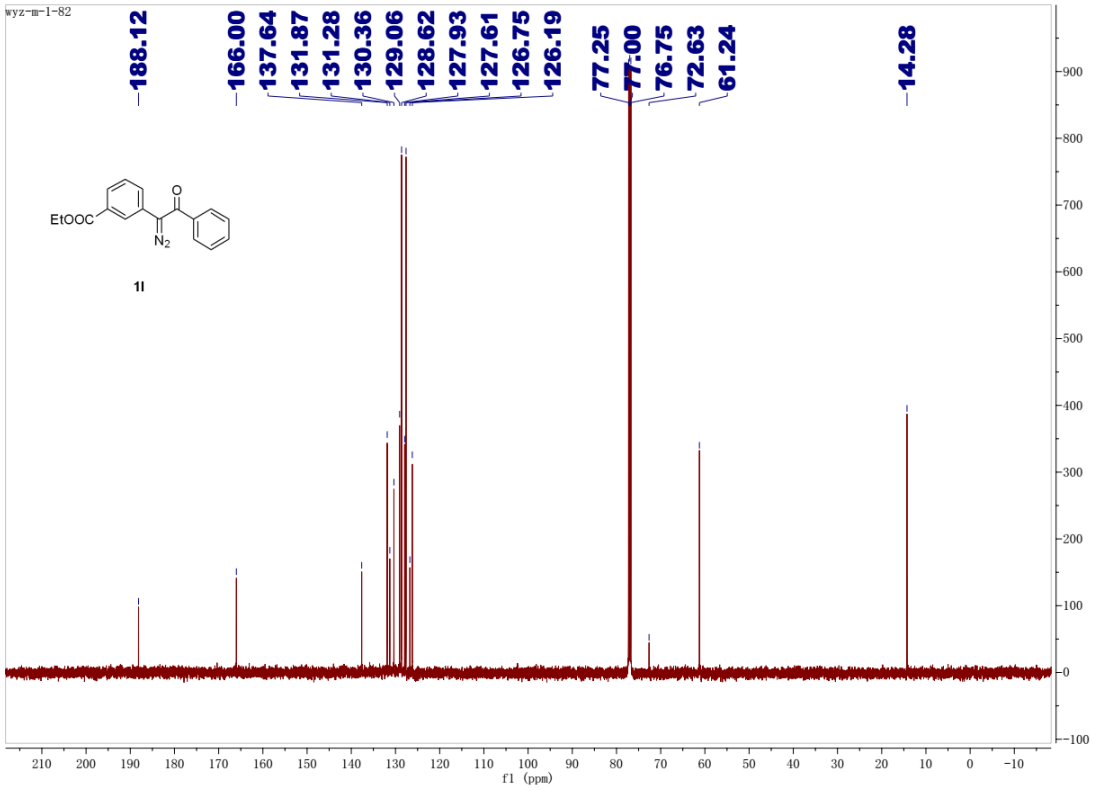
2.00
3.12
2.20
2.10
f1 (ppm)

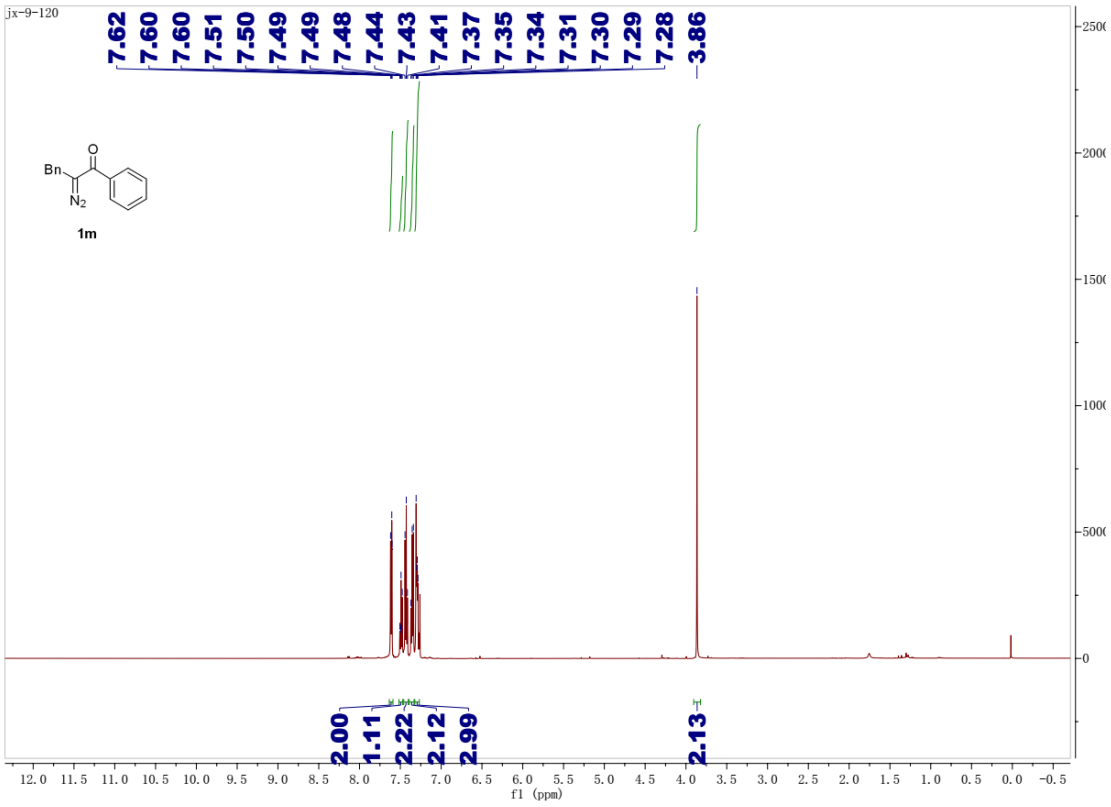

1xs-8-121
8.07
8.07
8.05
8.05
7.64
7.62
7.62
7.59
7.57
7.53
7.53
7.52
7.52
7.46
7.45
7.45
7.43
7.43
3.92
MeOOC
O
N2
1j
2.00
1.93
1.95
1.03
2.07
3.11
f1 (ppm)

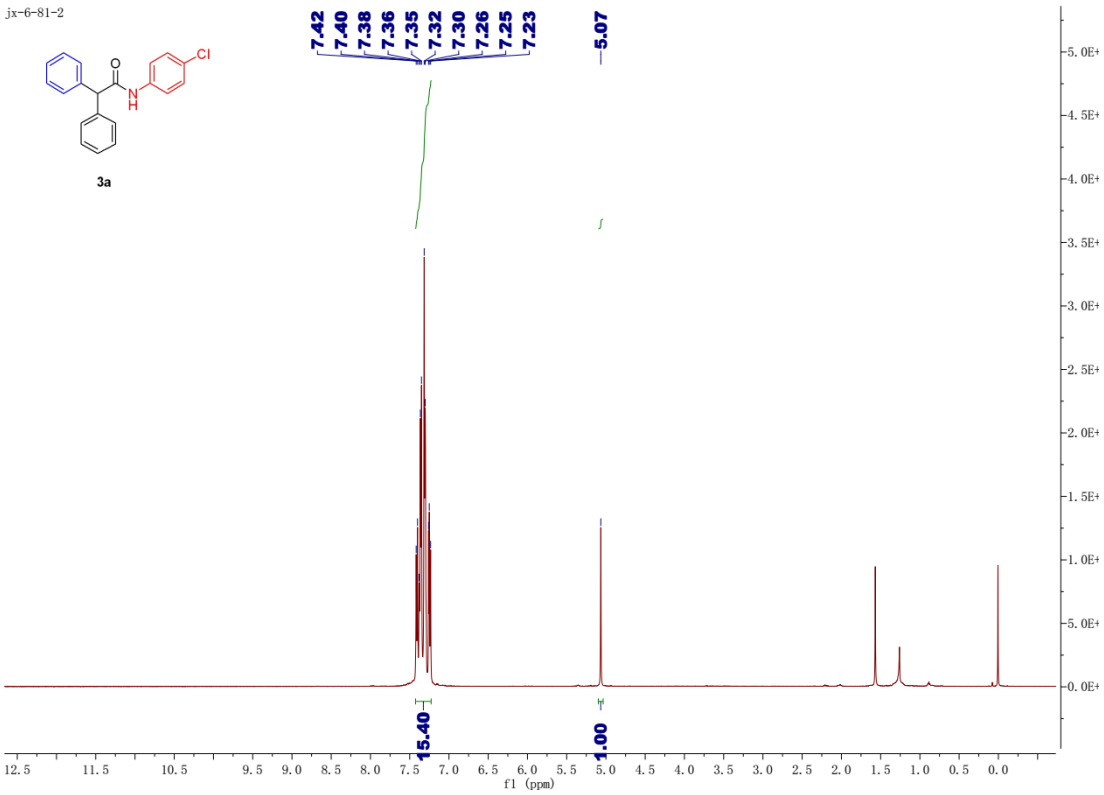

wyz-1-82
7.99
7.98
7.65
7.63
7.63
7.62
7.60
7.55
7.54
7.53
7.47
7.45
7.44
2.60
1k
2.00
4.11
1.14
2.01
3.07
f1 (ppm)

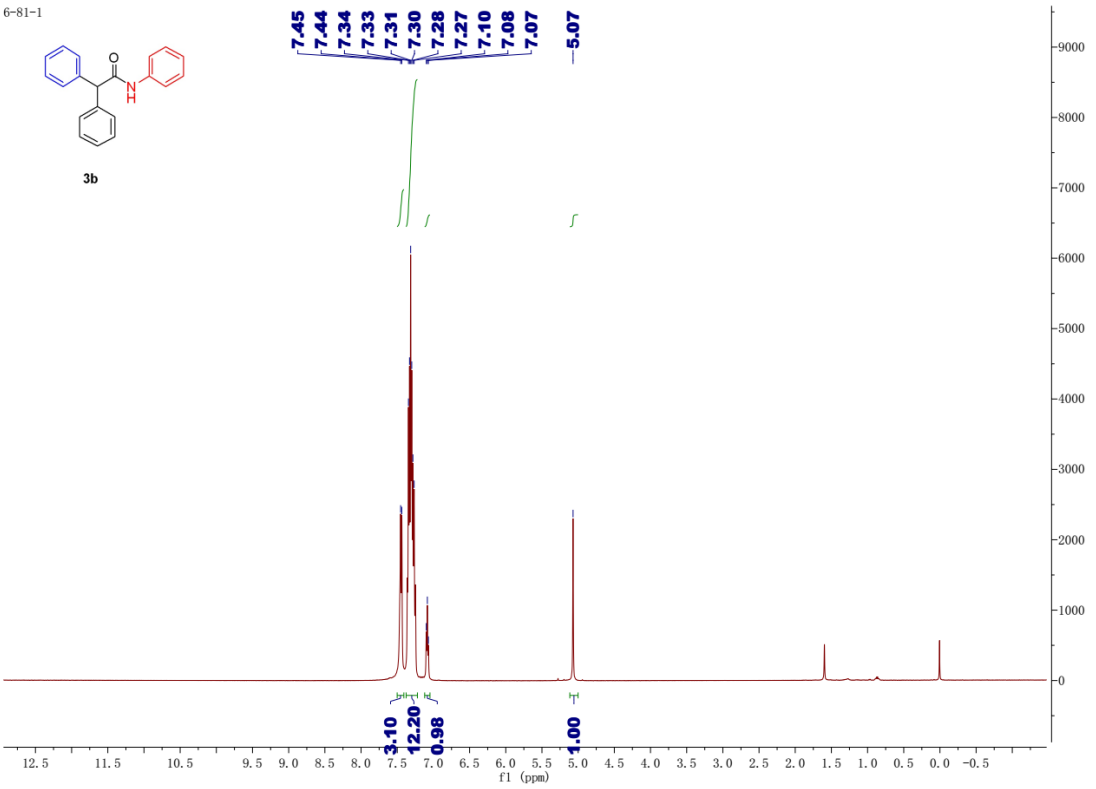

wyz-1-82
197.05
187.89
137.60
134.97
132.06
131.56
128.97
128.70
127.58
124.82
77.25
77.00
76.75
73.32
26.51
1k
f1 (ppm)

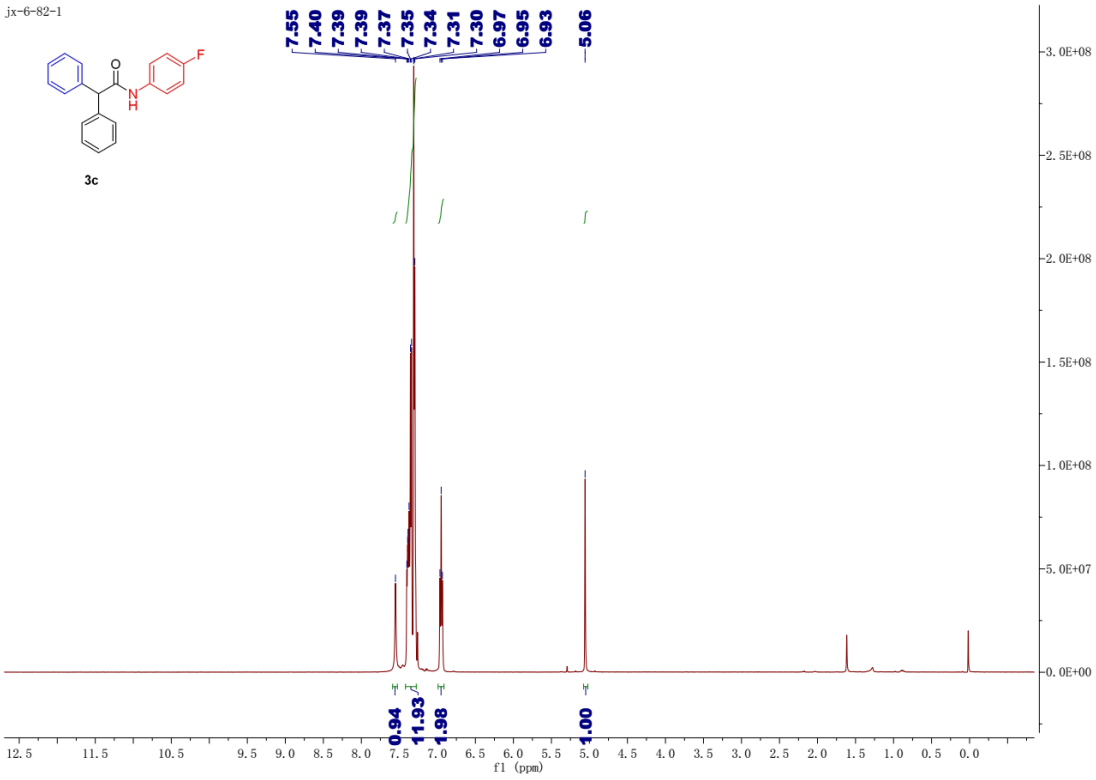

wyz-m-1-82
8.08
7.94
7.92
7.79
7.77
7.64
7.62
7.52
7.51
7.49
7.48
7.46
7.45
4.40
4.39
4.38
4.36
1.41
1.39
1.38
EtOOC
O
N2
1l
1.00
1.09
1.06
2.12
4.35
2.21
3.39
f1 (ppm)

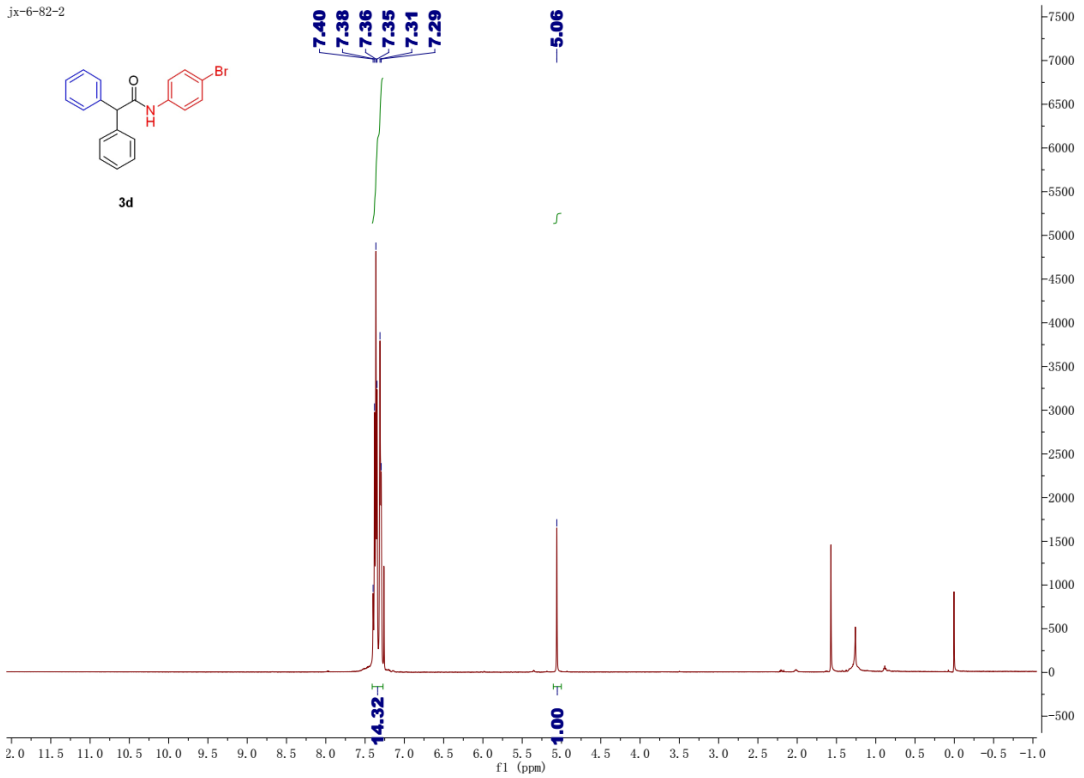

wyz-m-1-82
188.12
166.00
137.64
131.87
131.28
130.36
129.06
128.62
127.93
127.61
126.75
126.19
77.25
77.00
76.75
72.63
61.24
14.28
EtOOC
O
N2
1l
f1 (ppm)

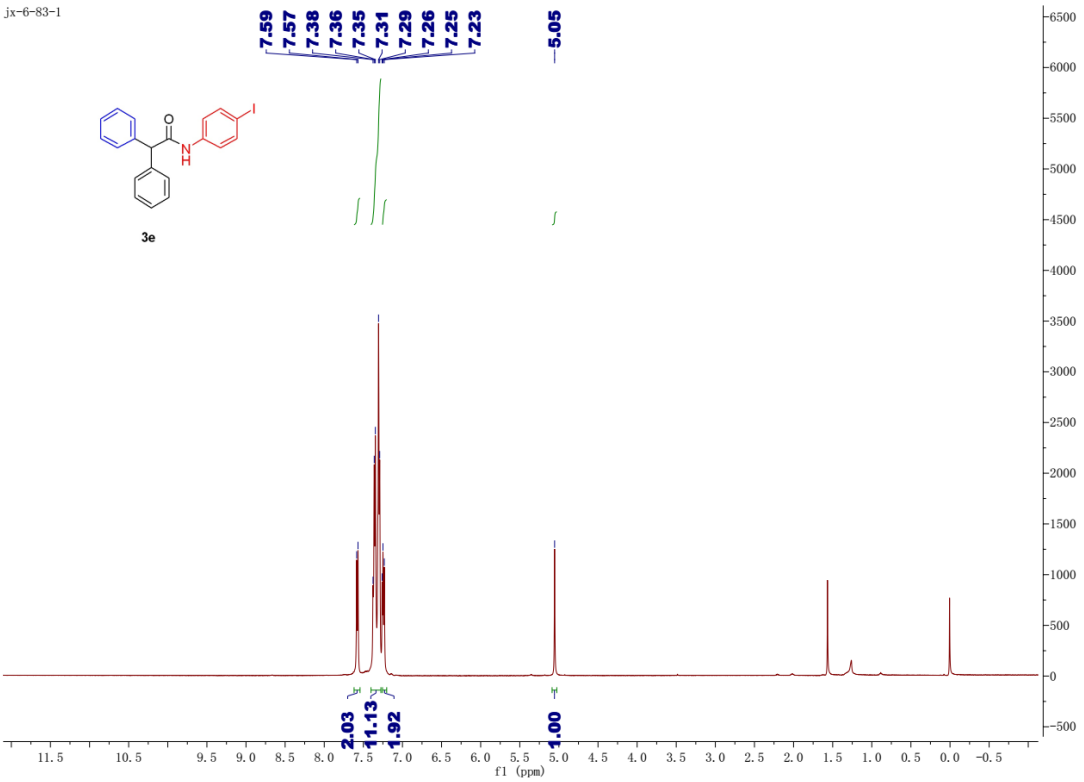

jx-9-120
7.62
7.60
7.60
7.51
7.50
7.49
7.49
7.48
7.44
7.43
7.41
7.37
7.35
7.34
7.31
7.30
7.29
7.28
3.86
Bn
O
N2
1m
2.00
1.11
2.22
2.12
2.99
2.13
f1 (ppm)

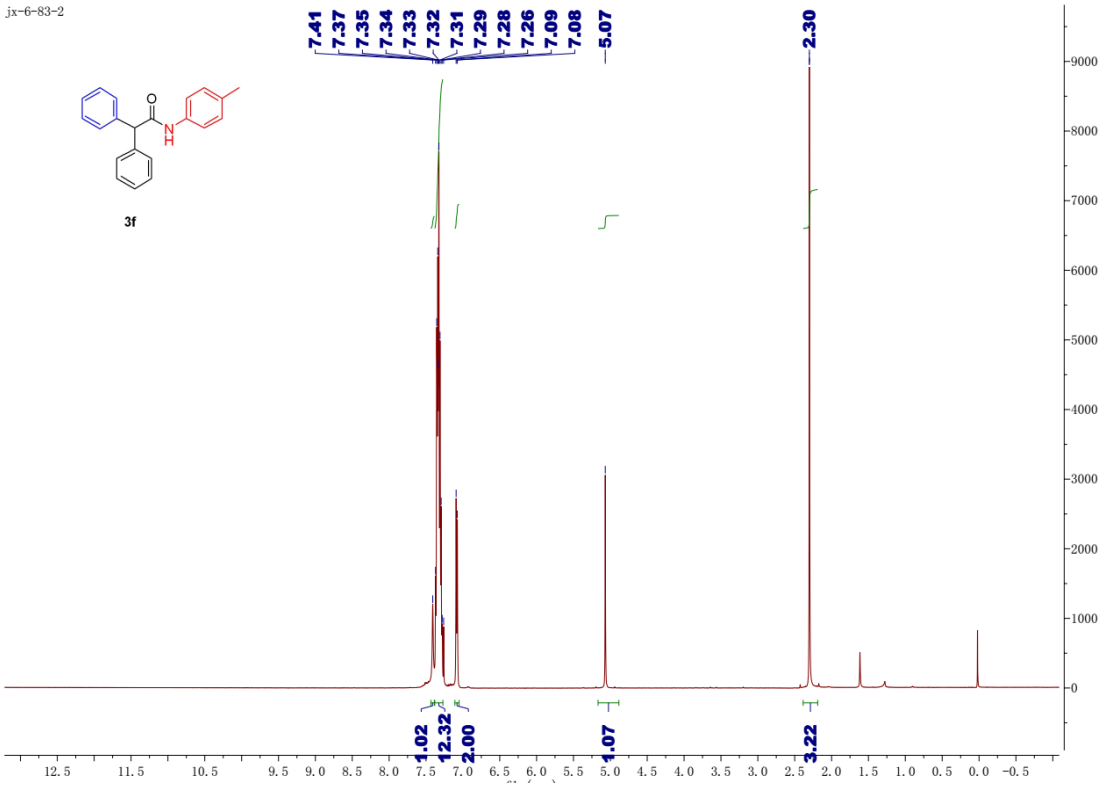

jx-6-81-2
7.42
7.40
7.38
7.36
7.35
7.32
7.30
7.26
7.25
7.23
5.07
O
N
H
Cl
3a
15.40
1.00
f1 (ppm)

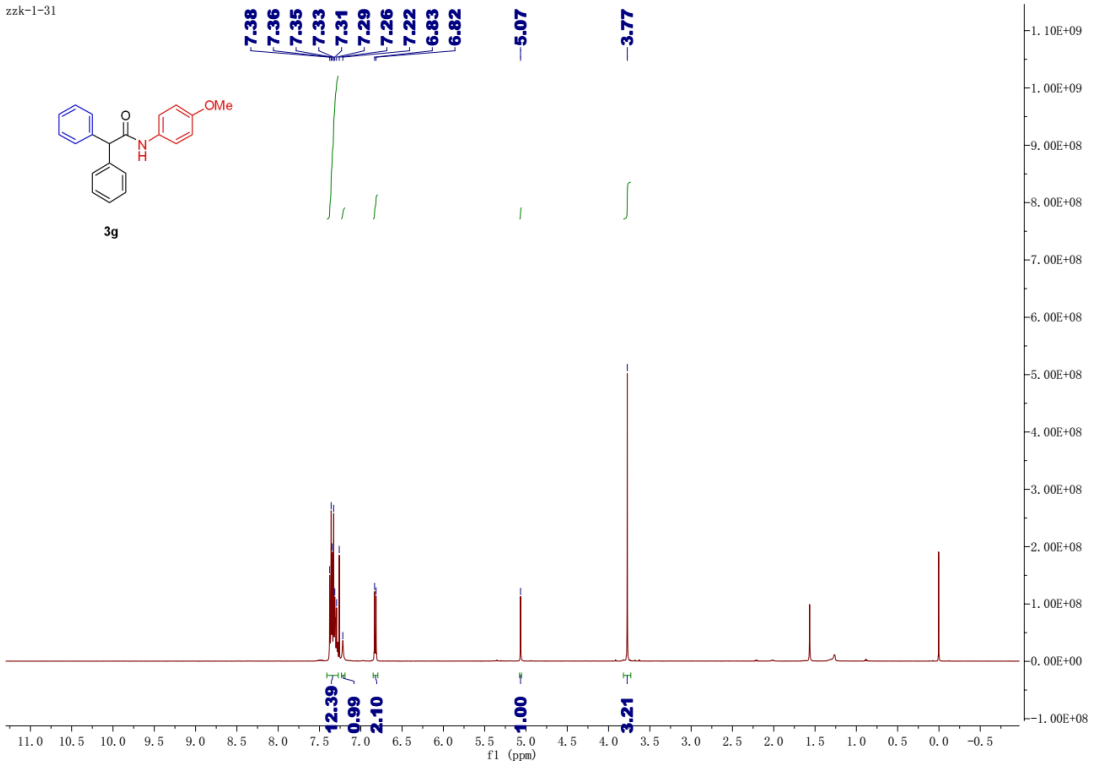

6-81-1
3b
7.45
7.44
7.34
7.33
7.31
7.30
7.28
7.27
7.10
7.08
7.07
5.07
3.10
12.20
0.98
1.00
f1 (ppm)

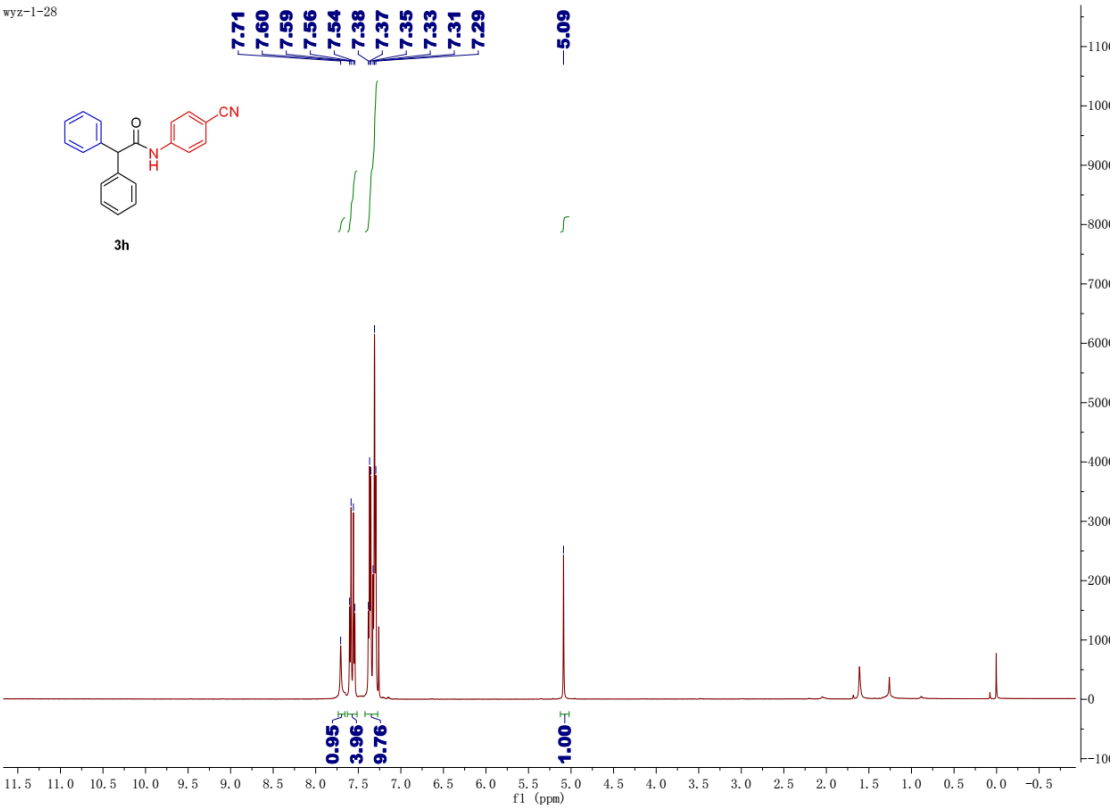

jx-6-82-1
3c
7.55
7.40
7.39
7.39
7.37
7.35
7.34
7.31
7.30
6.97
6.95
6.93
5.06
0.94
11.93
1.98
1.00
f1 (ppm)

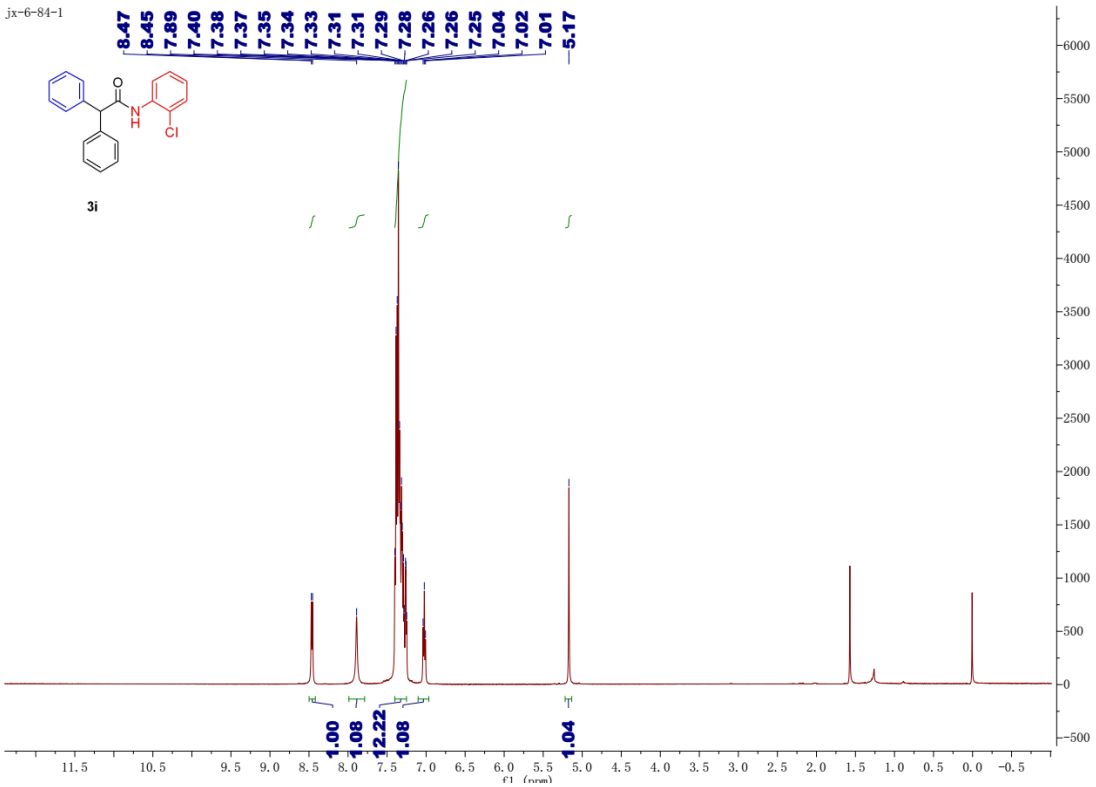

jx-6-82-2
7.40
7.38
7.36
7.35
7.31
7.29
5.06
Br
O
N
H
3d
14.32
1.00
f1 (ppm)

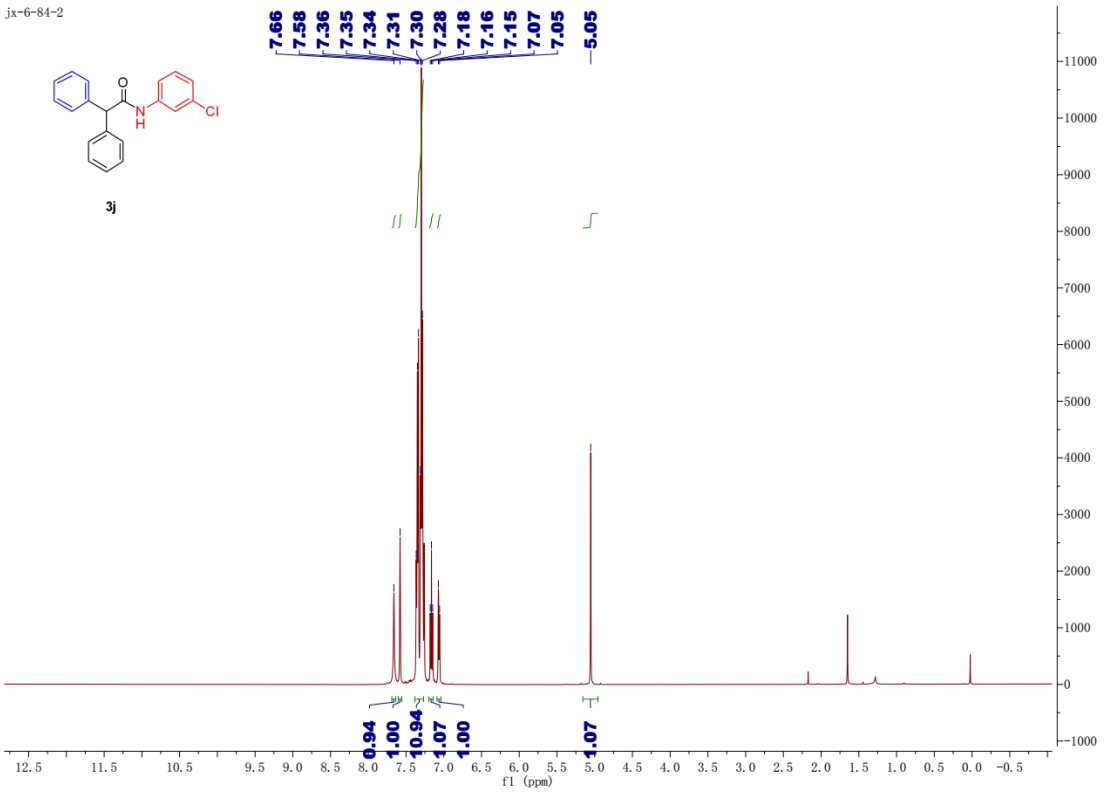

jx-6-83-1
7.59
7.57
7.38
7.36
7.35
7.31
7.29
7.26
7.25
7.23
5.05
I
O
N
H
3e
2.03
1.13
1.92
1.00
f1 (ppm)

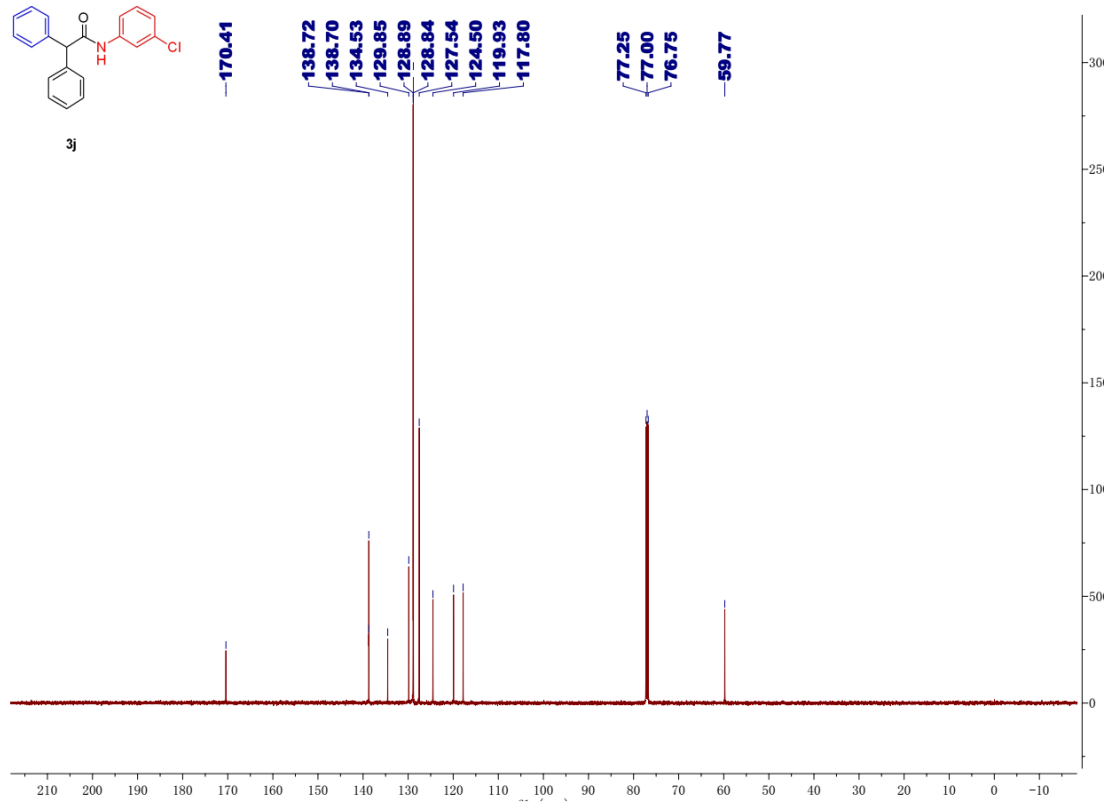

jx-6-83-2
7.41
7.37
7.35
7.34
7.33
7.32
7.31
7.29
7.28
7.26
7.09
7.08
5.07
2.30
3f
1.02
12.32
2.00
1.07
3.22

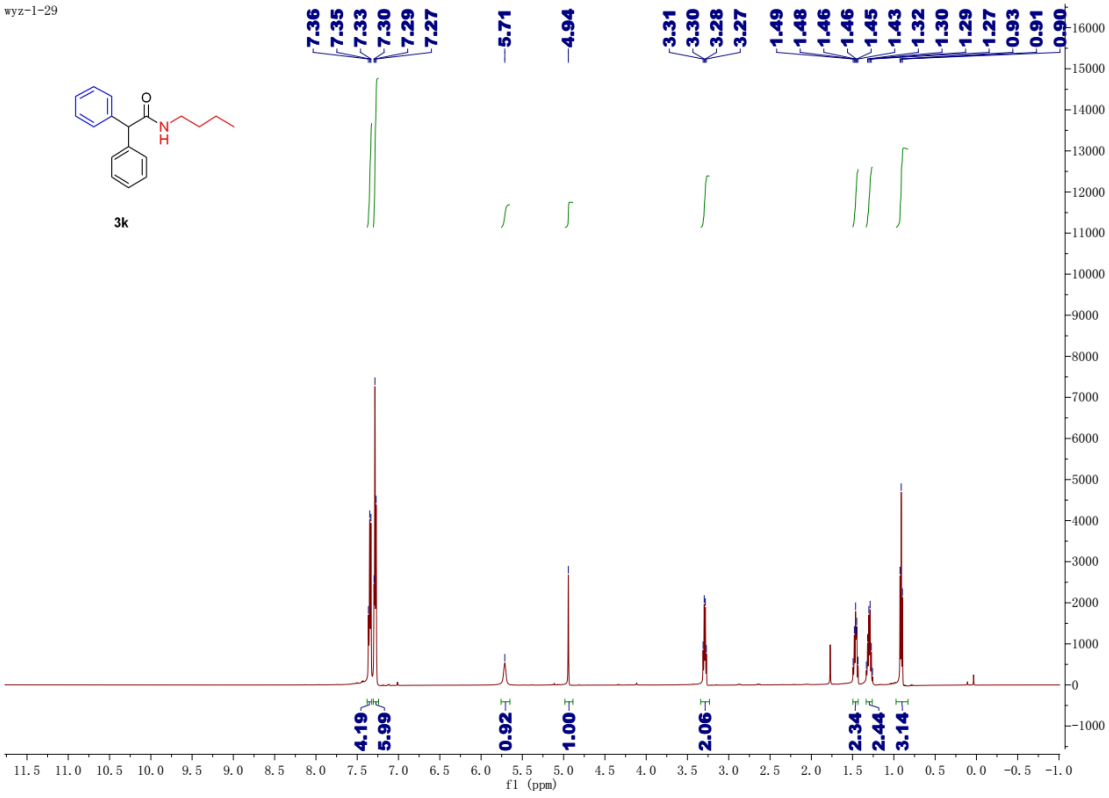

zzk-1-31
7.38
7.36
7.35
7.33
7.31
7.29
7.26
7.22
6.83
6.82
5.07
3.77
OMe
3g
12.39
0.99
2.10
1.00
3.21
f1 (ppm)

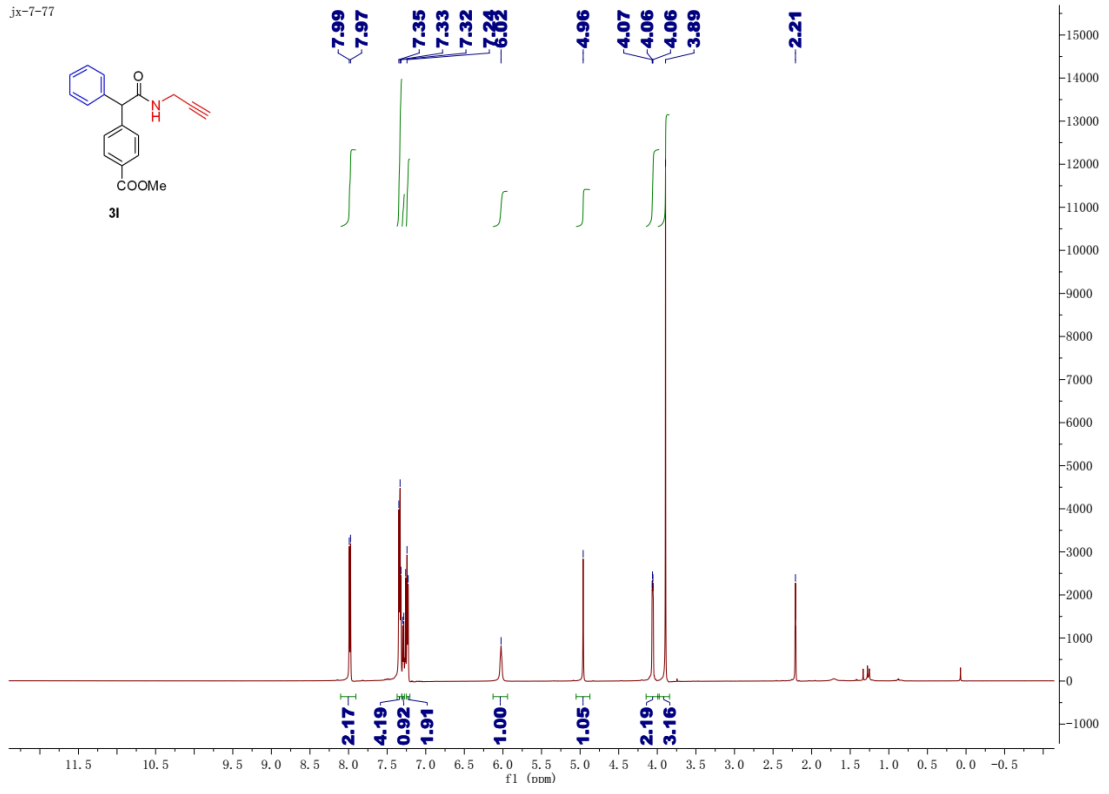

wyz-1-28
7.71
7.60
7.59
7.56
7.54
7.38
7.37
7.35
7.33
7.31
7.29
5.09
CN
O
N
H
3h
0.95
3.96
9.76
1.00
f1 (ppm)

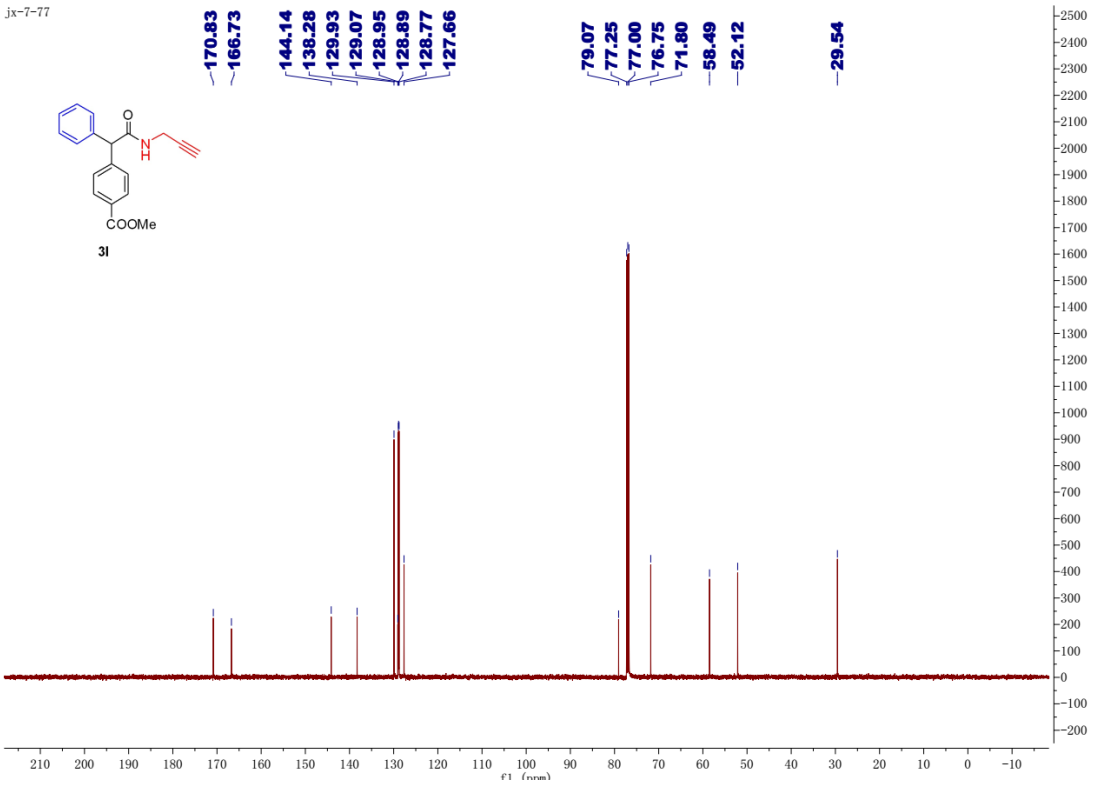

jx-6-84-1
8.47
8.45
7.89
7.40
7.38
7.37
7.35
7.34
7.33
7.31
7.31
7.29
7.28
7.26
7.26
7.25
7.04
7.02
7.01
5.17
O
N
H
Cl
3i
1.00
1.08
12.22
1.08
1.04


jx-6-84-2
3j
7.66
7.58
7.36
7.35
7.34
7.31
7.30
7.28
7.18
7.16
7.15
7.07
7.05
5.05
0.94
1.00
0.94
1.07
1.00
1.07
f1 (ppm)


3j
170.41
138.72
138.70
134.53
129.85
128.89
128.84
127.54
124.50
119.93
117.80
77.25
77.00
76.75
59.77


wyz-1-29
7.36
7.35
7.33
7.30
7.29
7.27
5.71
4.94
3.31
3.30
3.28
3.27
1.49
1.48
1.46
1.46
1.45
1.43
1.32
1.30
1.29
1.27
0.93
0.91
0.90
3k
4.19
5.99
0.92
1.00
2.06
2.34
2.44
3.14
f1 (ppm)


jx-7-77
7.99
7.97
7.35
7.33
7.32
7.24
6.02
4.96
4.07
4.06
4.06
3.89
2.21
COOMe
3l
2.17
4.19
0.92
1.91
1.00
1.05
2.19
3.16
f1 (ppm)


jx-7-77
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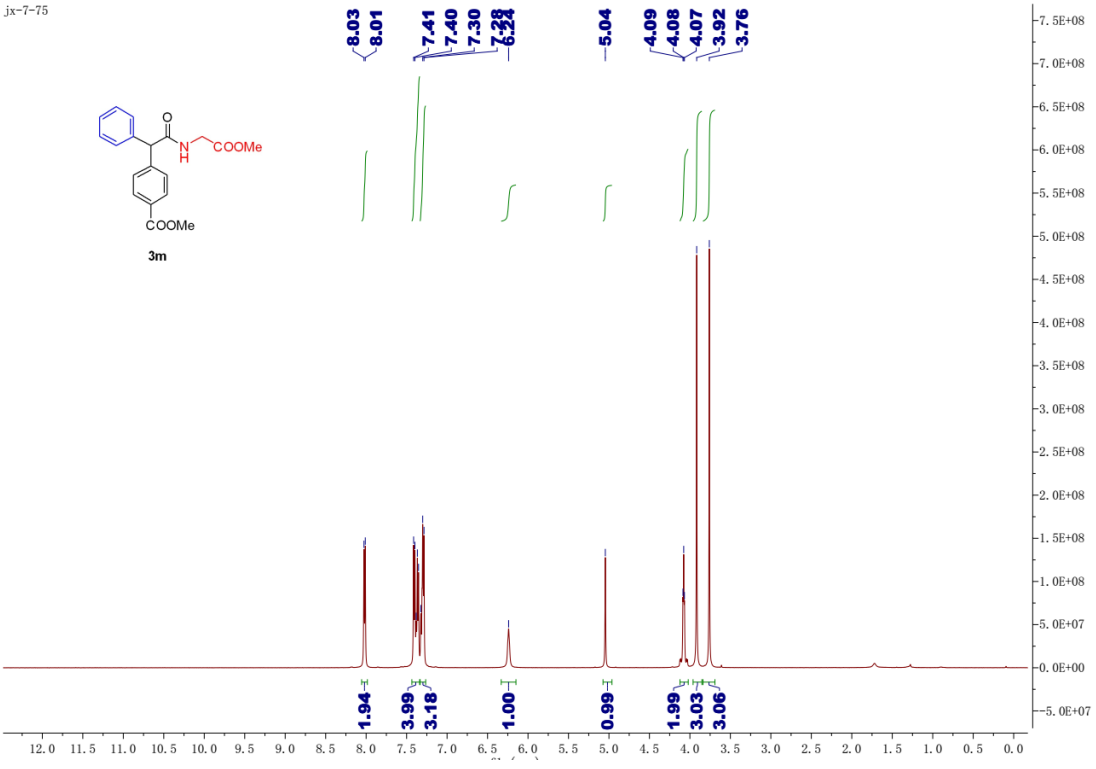
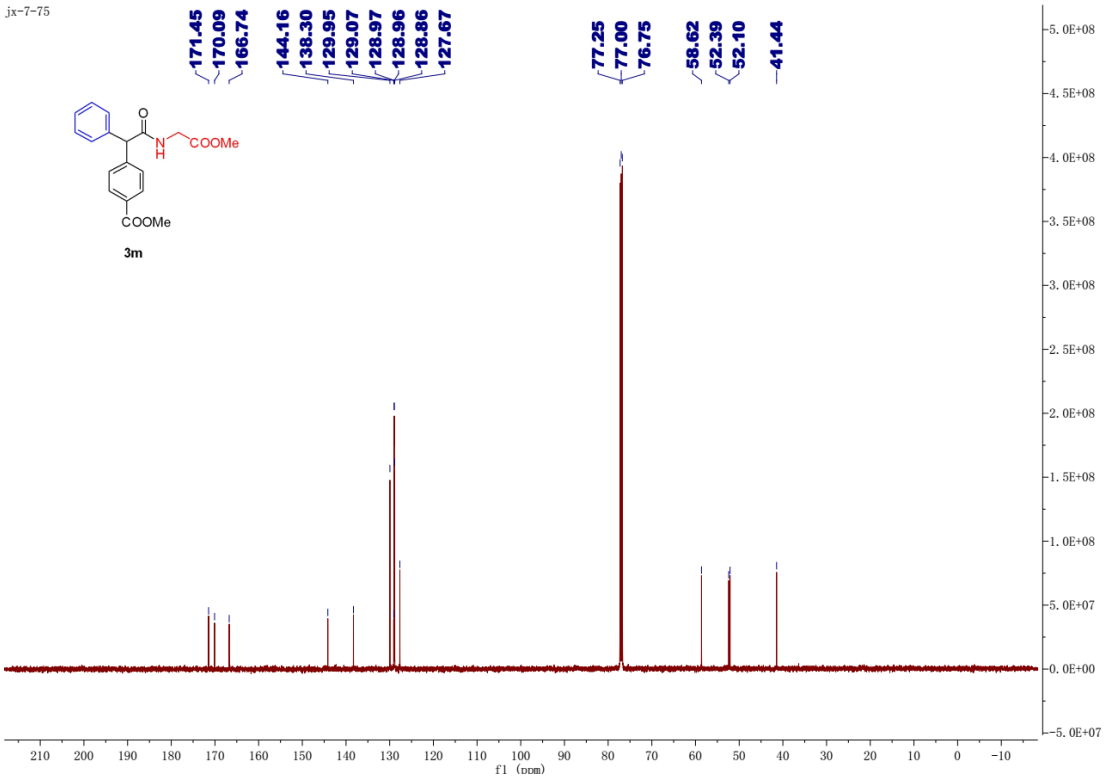
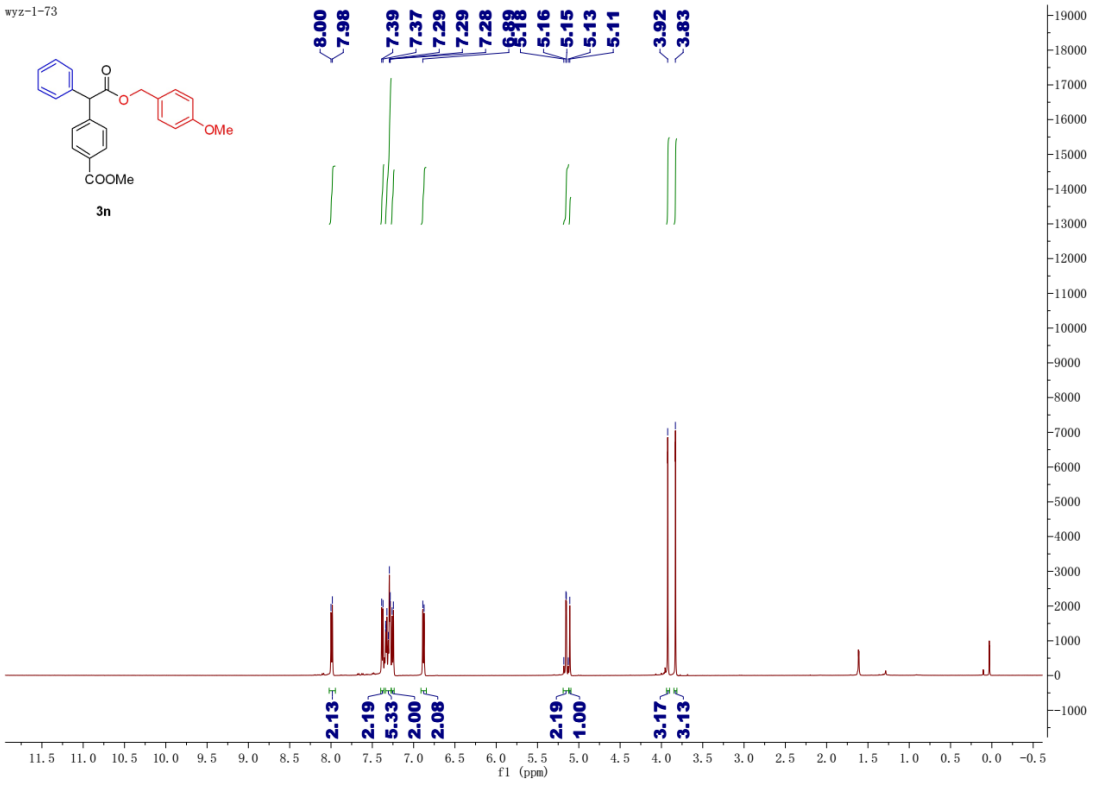
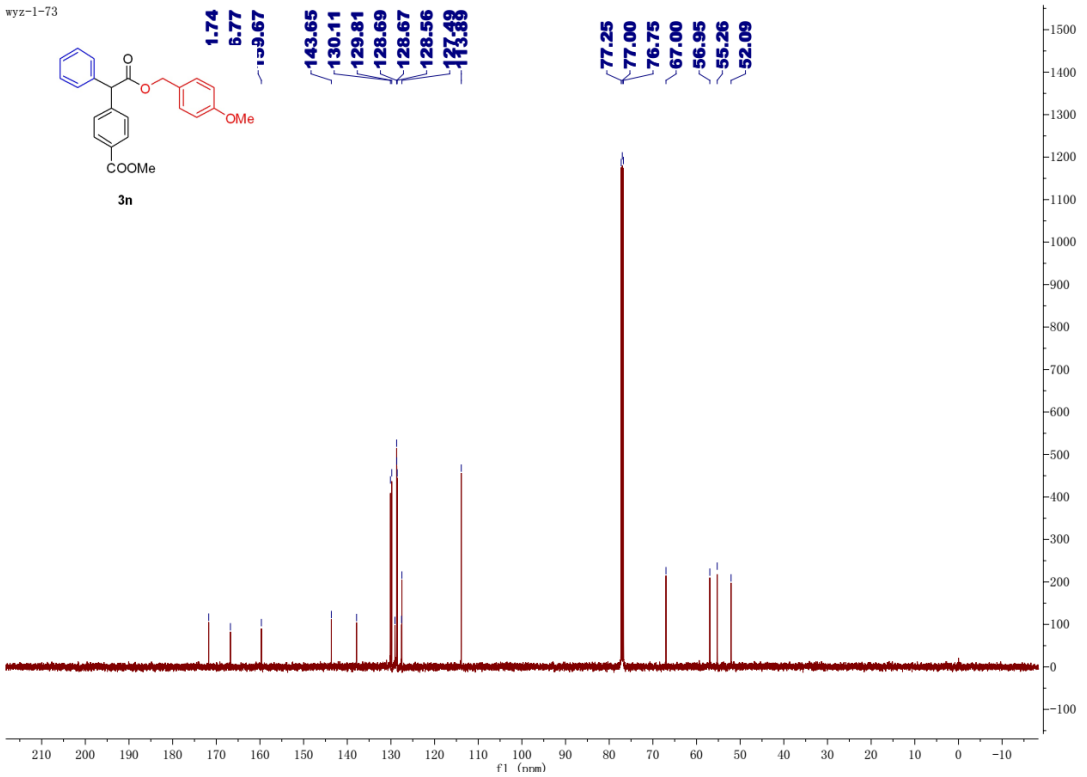
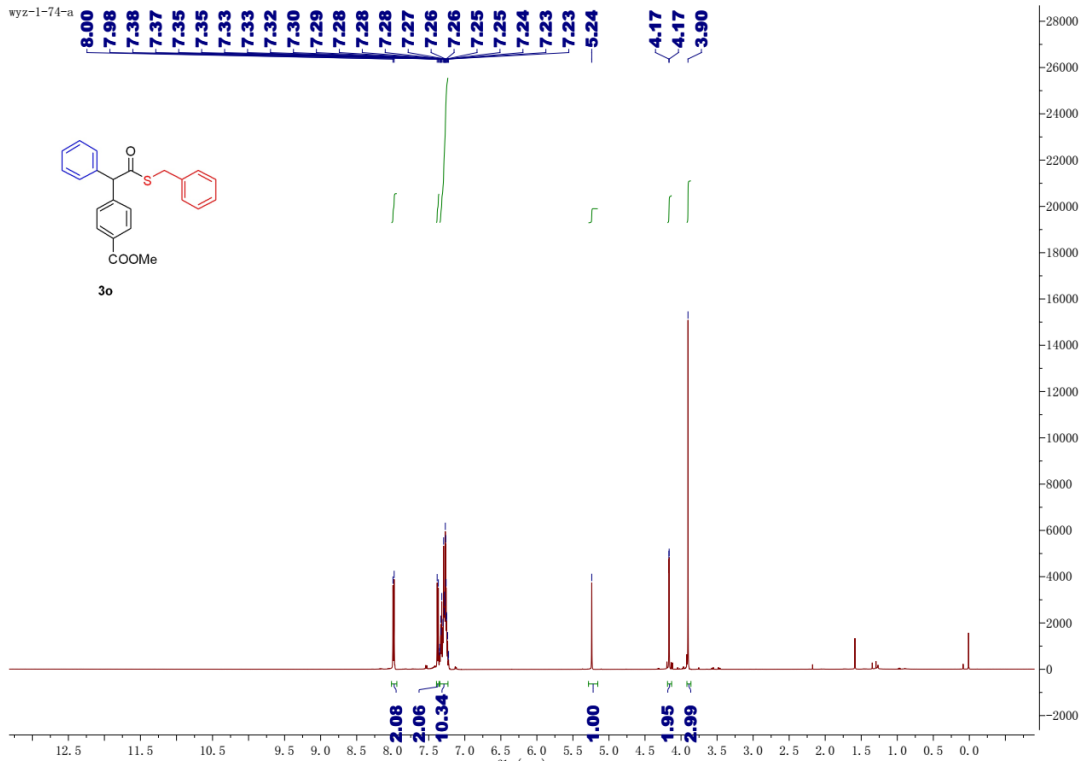
166.73
144.14
138.28
129.93
129.07
128.95
128.89
128.77
127.66
79.07
77.25
77.00
76.75
71.80
58.49
52.12
29.54
COOMe
3l


jx-7-75
8.03
8.01
7.41
7.40
7.30
5.04
4.09
4.08
4.07
3.92
3.76
1.94
3.99
3.18
1.00
0.99
1.99
3.03
3.06
COOMe
COOMe
3m


jx-7-75
171.45
170.09
166.74
144.16
138.30
129.95
129.07
128.97
128.96
128.86
127.67
77.25
77.00
76.75
58.62
52.39
52.10
41.44
COOMe
COOMe
3m
f1 (ppm)


wyz-1-73
8.00
7.98
7.39
7.37
7.29
7.29
7.28
5.16
5.15
5.13
5.11
3.92
3.83
OMe
COOMe
3n
2.13
2.19
5.33
2.00
2.08
2.19
1.00
3.17
3.13
f1 (ppm)


wyz-1-73
1.74
5.77
143.65
130.11
129.81
128.69
128.67
128.56
77.25
77.00
76.75
67.00
56.95
55.26
52.09
OMe
COOMe
3n
f1 (ppm)


wyz-1-74-a
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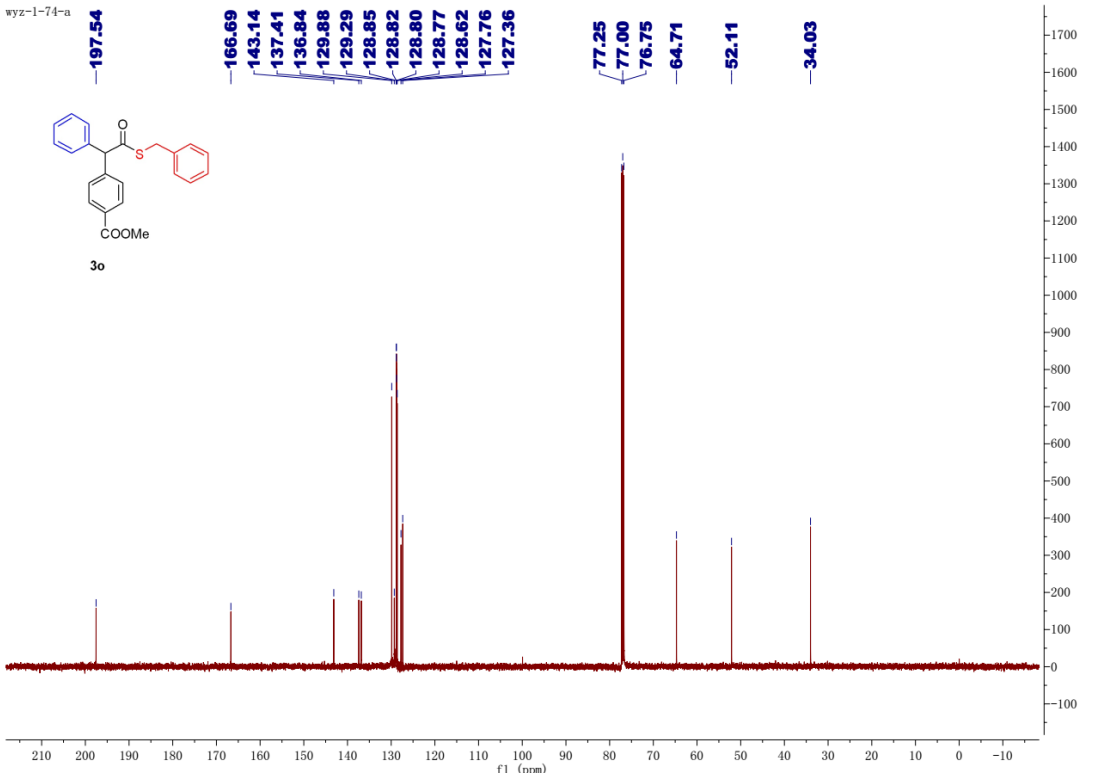
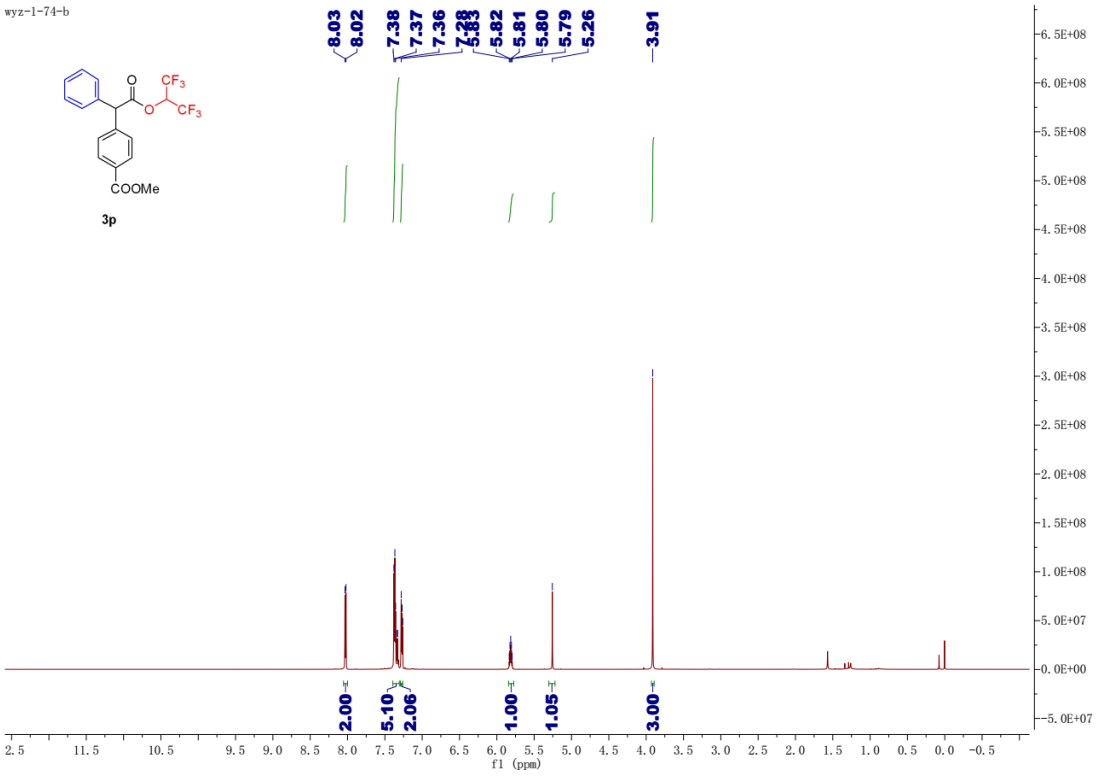
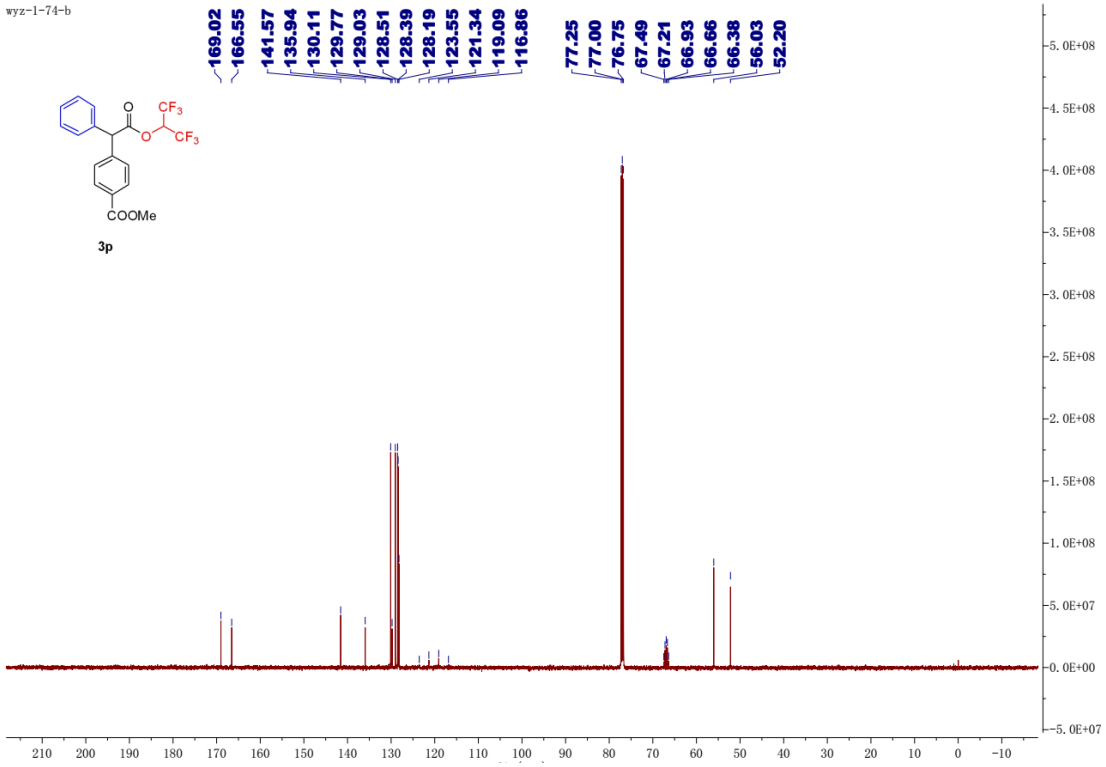
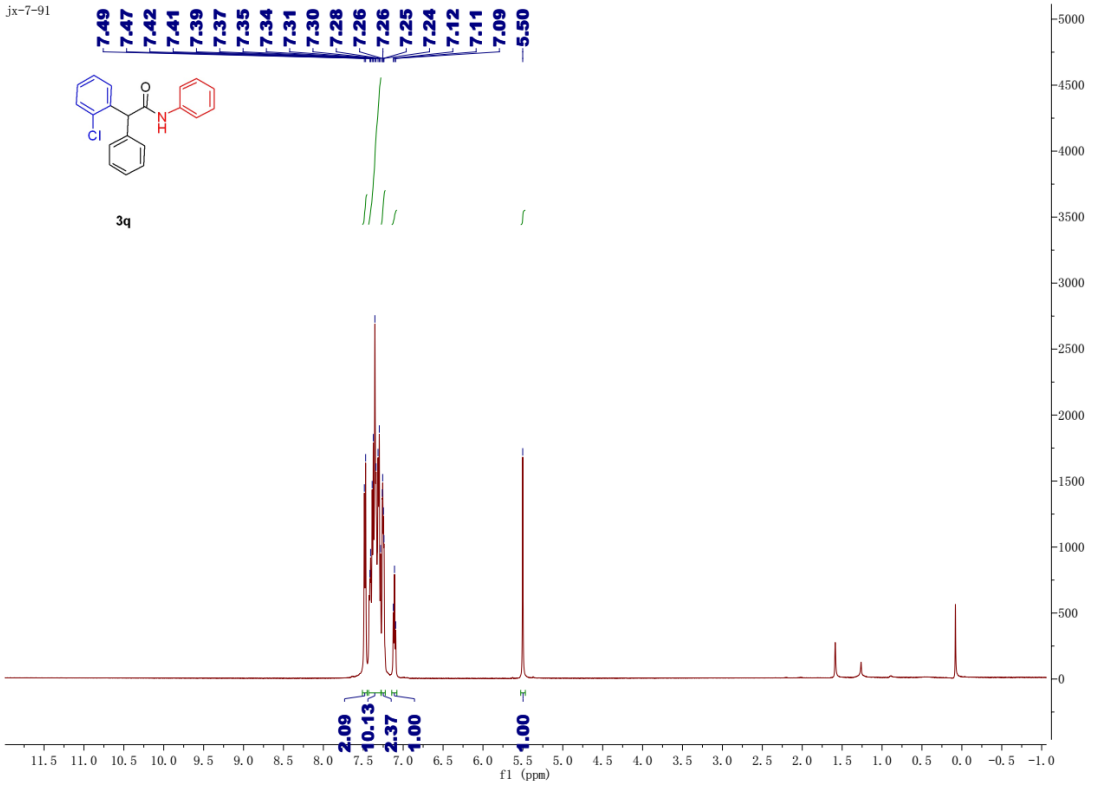
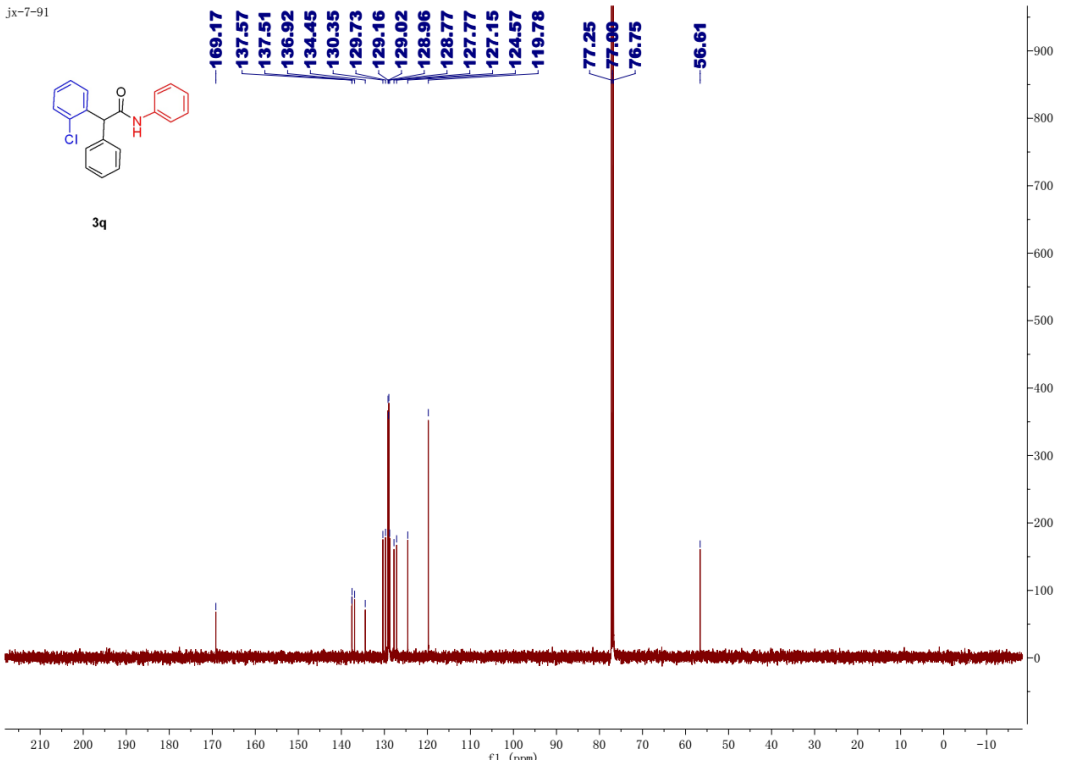
7.98
7.38
7.37
7.35
7.35
7.33
7.33
7.32
7.30
7.29
7.28
7.28
7.28
7.27
7.26
7.26
7.25
7.25
7.24
7.23
7.23
5.24
4.17
4.17
3.90
2.08
2.06
10.34
1.00
1.95
2.99
S
COOMe
3o

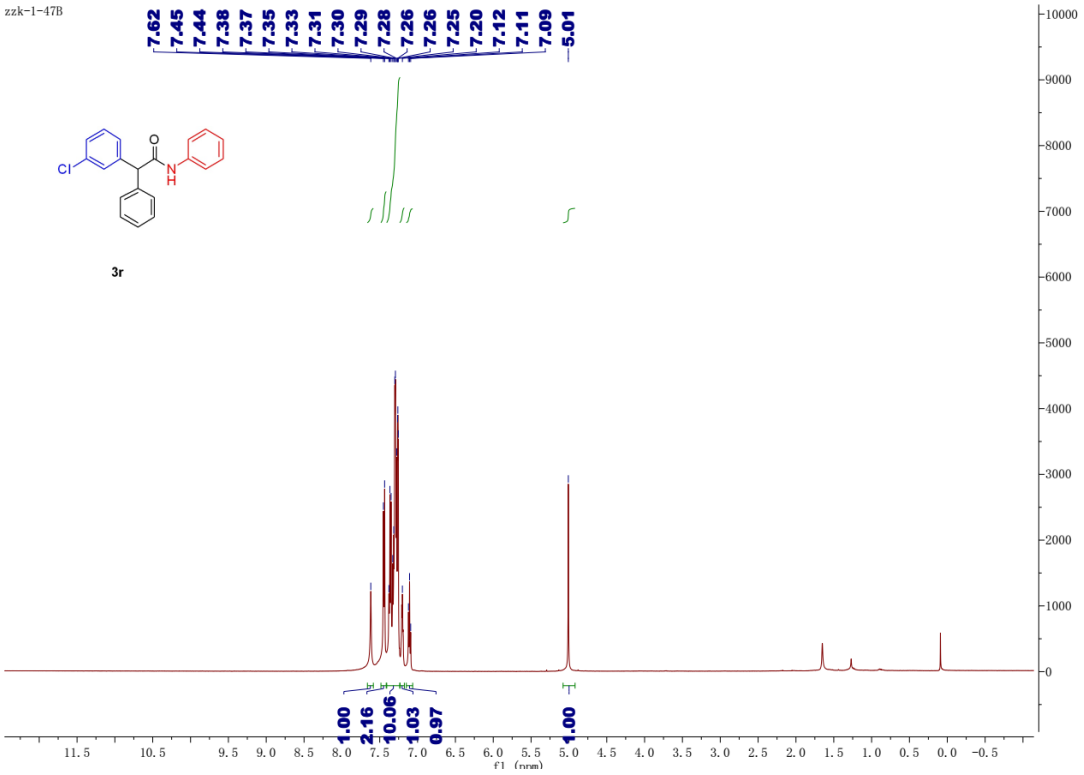

wyz-1-74-a
197.54
166.69
143.14
137.41
136.84
129.88
129.29
128.85
128.82
128.80
128.77
128.62
127.76
127.36
77.25
77.00
76.75
64.71
52.11
34.03
COOMe
3o
f1 (ppm)

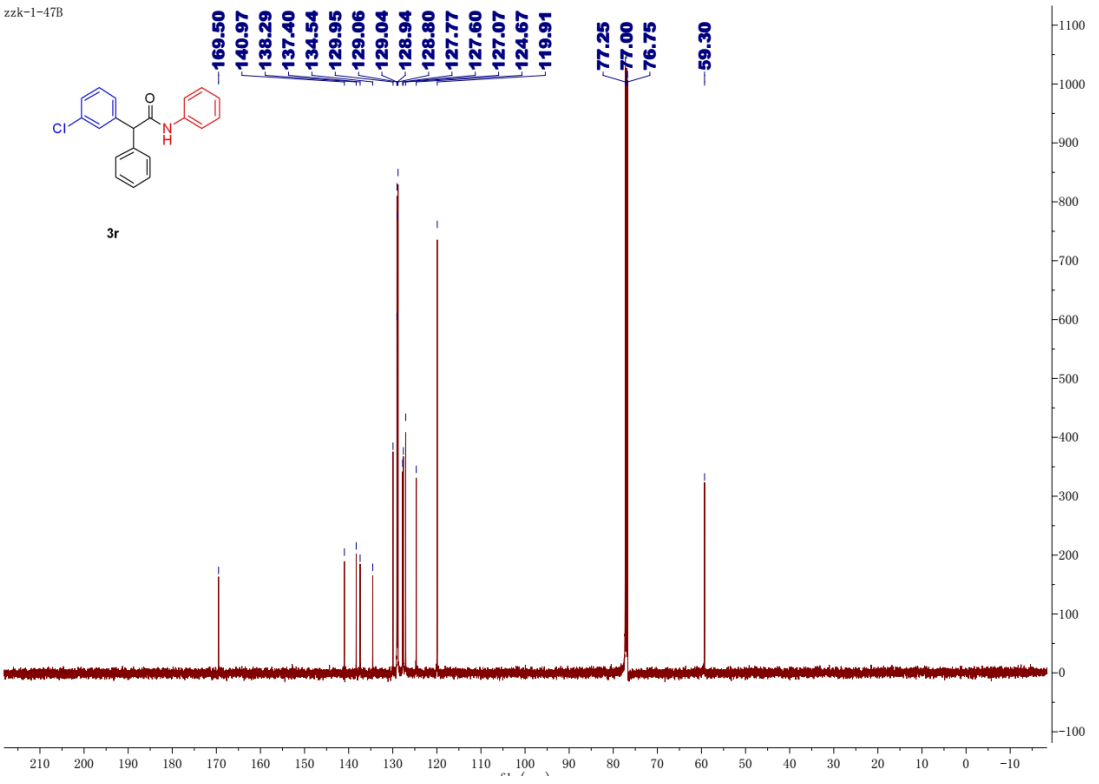

wyz-1-74-b
8.03
8.02
7.38
7.37
7.36
5.82
5.81
5.80
5.79
5.26
3.91
CF3
CF3
COOMe
3p
2.00
5.10
2.06
1.00
1.05
3.00
f1 (ppm)

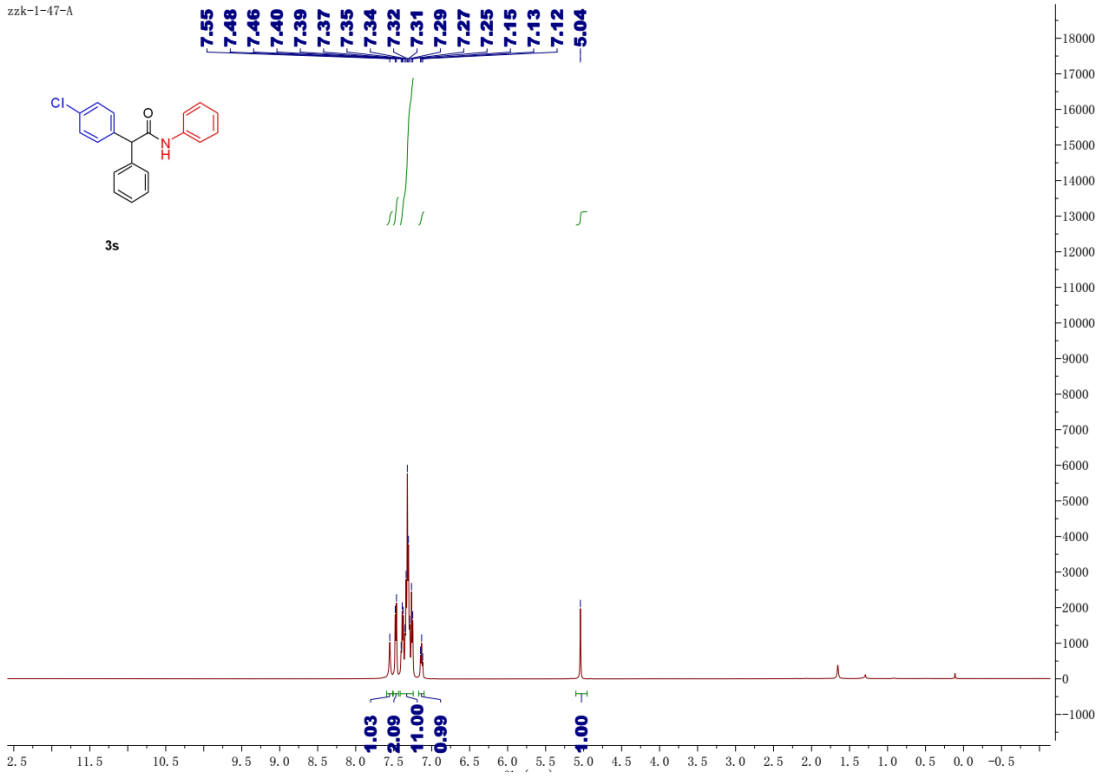

wyz-1-74-b
169.02
166.55
141.57
135.94
130.11
129.77
129.03
128.51
128.39
128.19
123.55
121.34
119.09
116.86
77.25
77.00
76.75
67.49
67.21
66.93
66.66
66.38
56.03
52.20
CF3
CF3
O
COOMe
3p

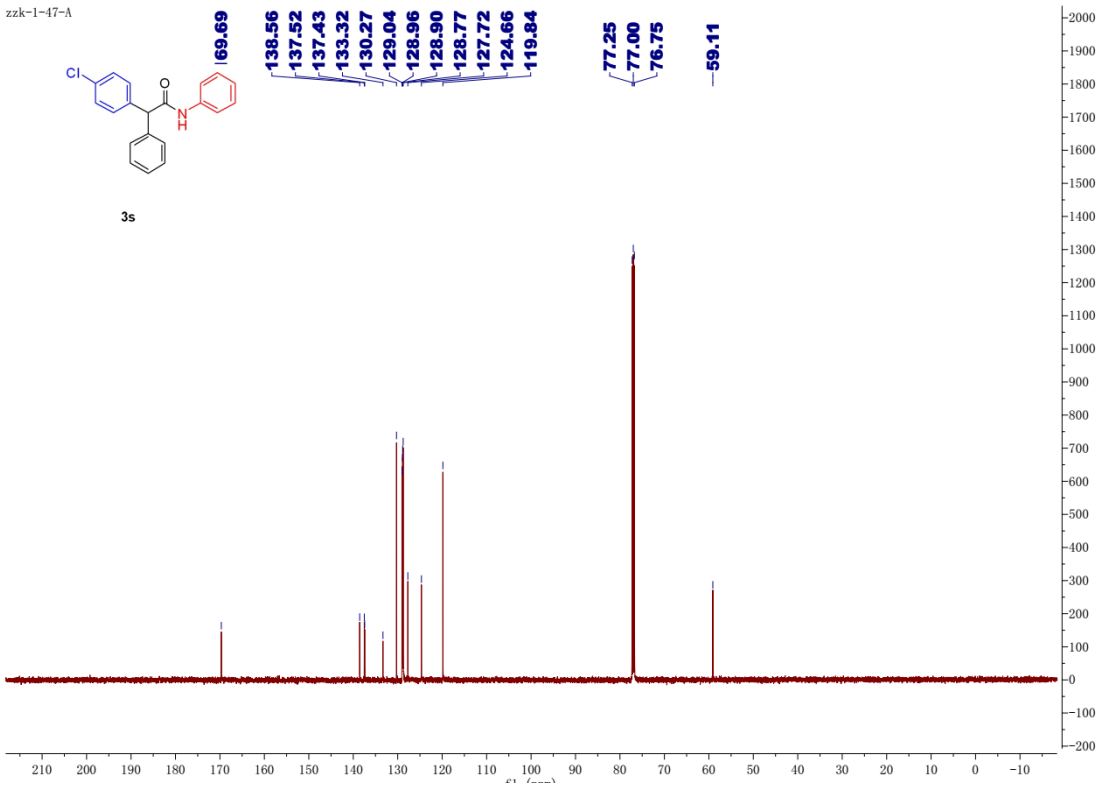

jx-7-91
7.49
7.47
7.42
7.41
7.39
7.37
7.35
7.34
7.31
7.30
7.28
7.26
7.26
7.25
7.24
7.12
7.11
7.09
5.50
Cl
N
H
O
3q
2.09
10.13
2.37
1.00
1.00
f1 (ppm)

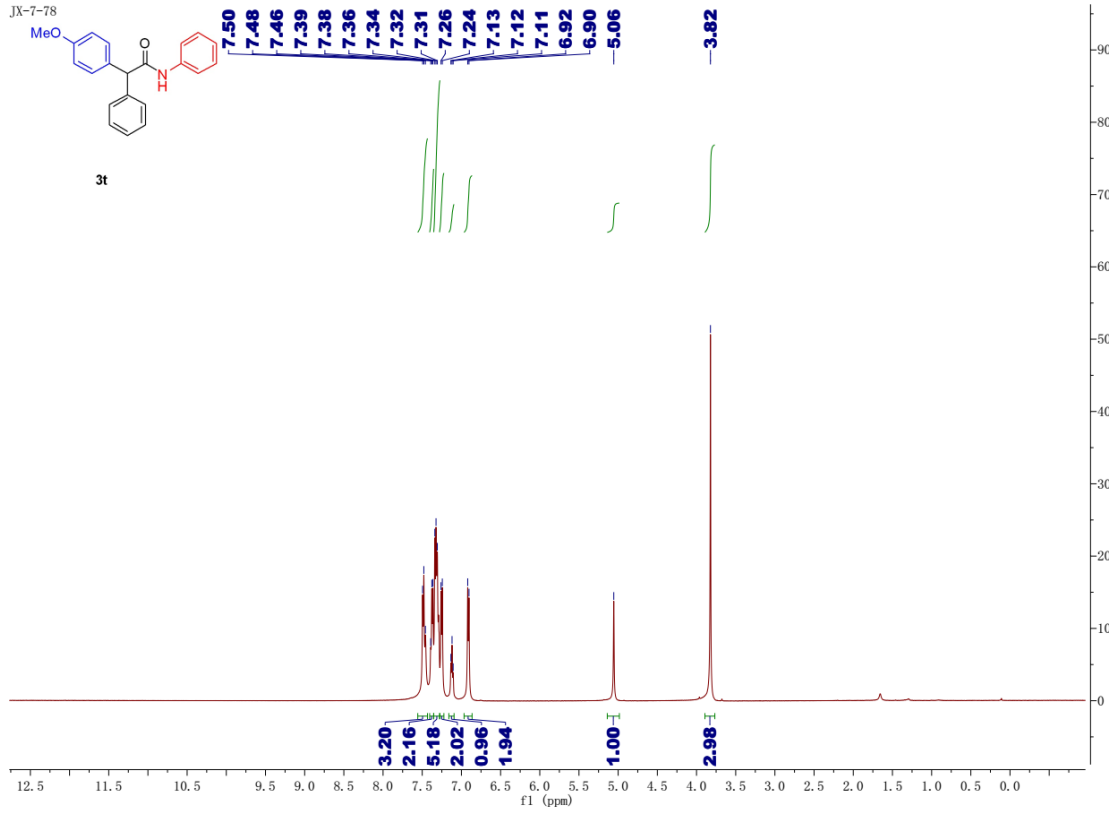

jx-7-91
169.17
137.57
137.51
136.92
134.45
130.35
129.73
129.16
129.02
128.96
128.77
127.77
127.15
124.57
119.78
77.25
77.00
76.75
56.61
Cl
N
H
O
3q
f1 (ppm)

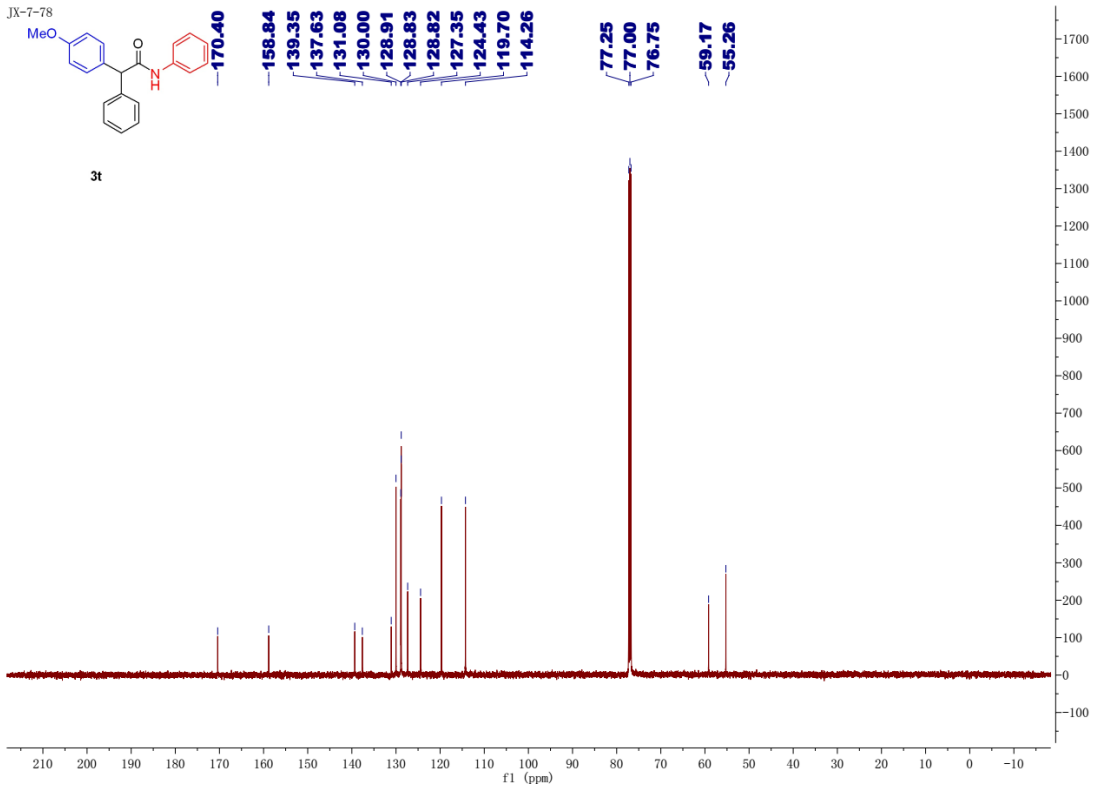

zzk-1-47B
7.62
7.45
7.44
7.38
7.37
7.35
7.33
7.31
7.30
7.29
7.28
7.26
7.26
7.25
7.20
7.12
7.11
7.09
5.01
Cl
N
H
O
3r
1.00
2.16
10.06
1.03
0.97
1.00
f1 (ppm)

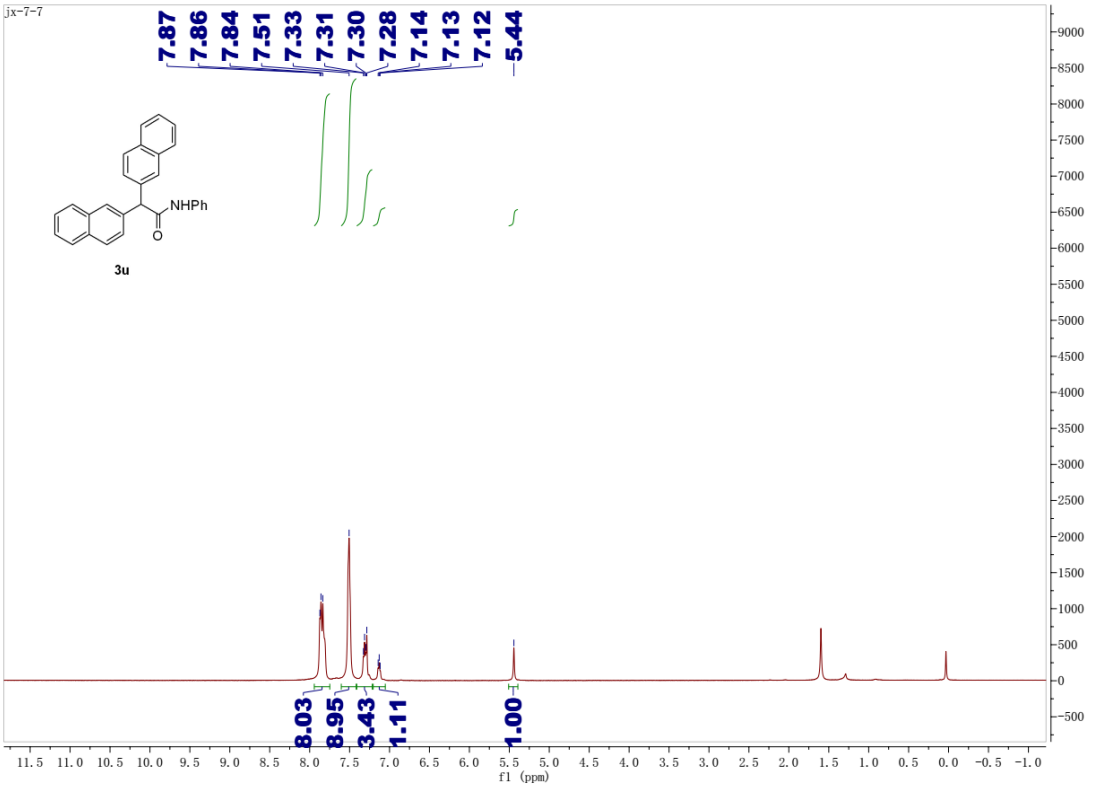

zzk-1-47B
169.50
140.97
138.29
137.40
134.54
129.95
129.06
129.04
128.94
128.80
127.77
127.60
127.07
124.67
119.91
77.25
77.00
76.75
59.30
Cl
O
N
H
3r

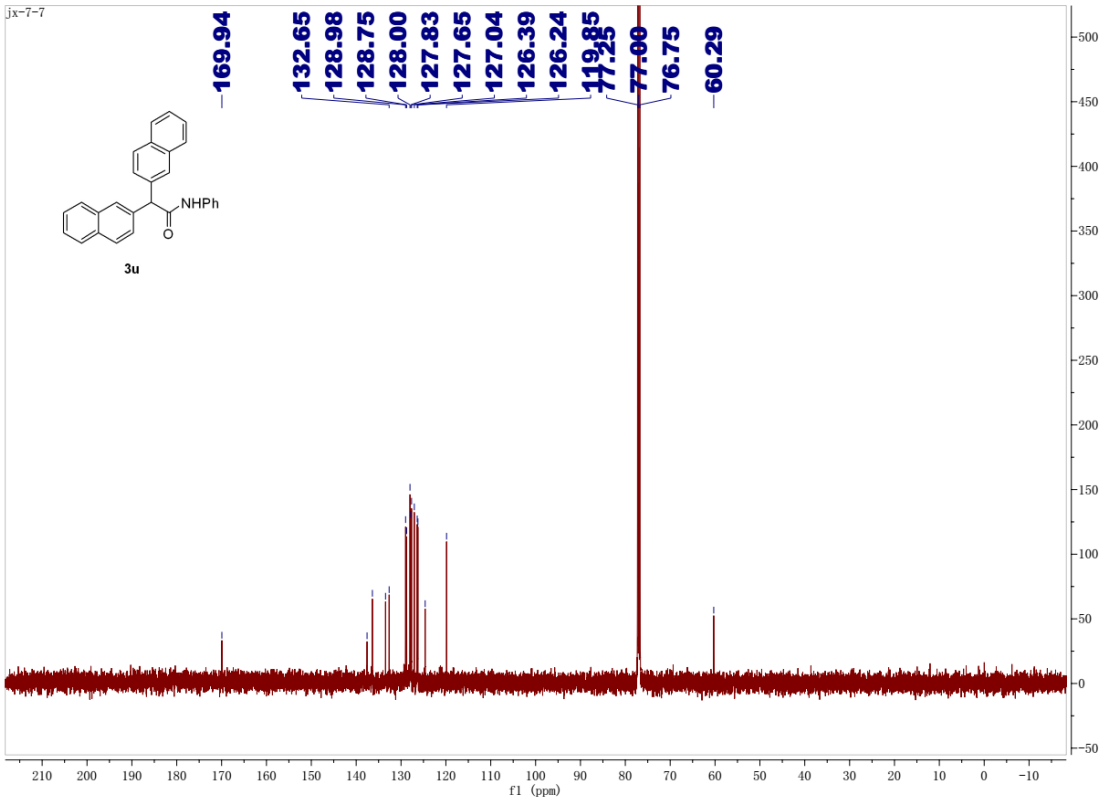

zzk-1-47-A
7.55
7.48
7.46
7.40
7.39
7.37
7.35
7.34
7.32
7.31
7.29
7.27
7.25
7.15
7.13
7.12
5.04
Cl
O
N
H
3s
1.03
2.09
11.00
0.99
1.00

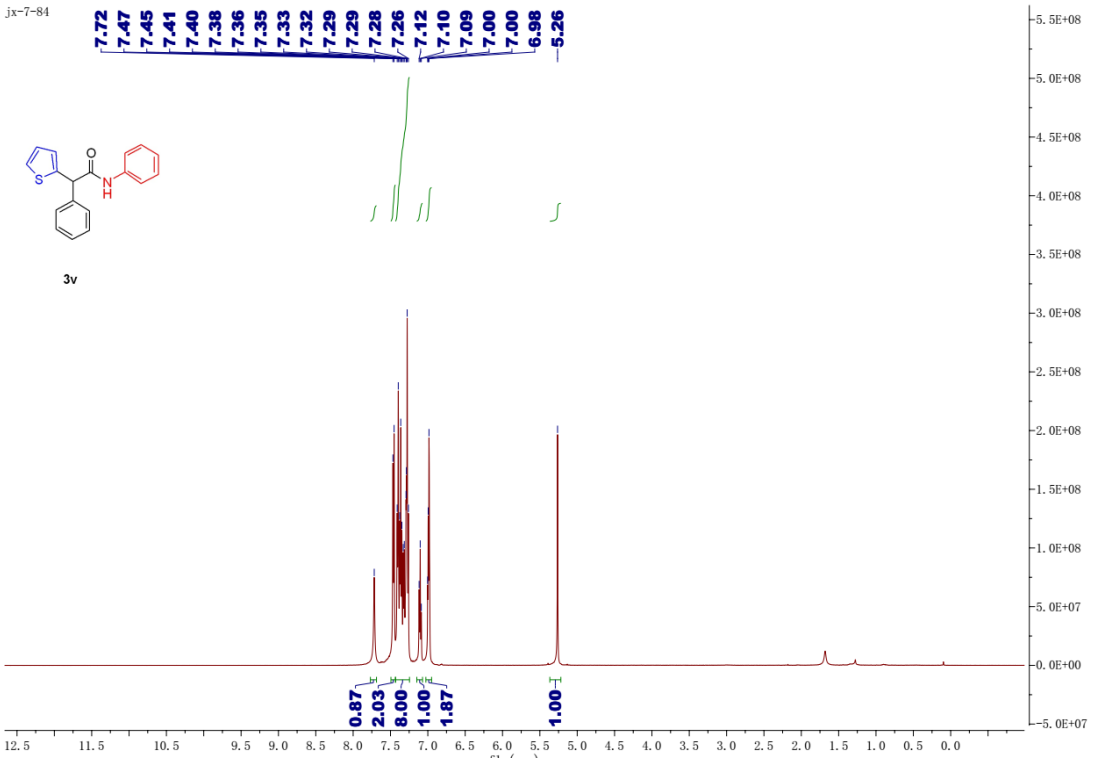

zzk-1-47-A
Cl
O
N
H
3s
169.69
138.56
137.52
137.43
133.32
130.27
129.04
128.96
128.90
128.77
127.72
124.66
119.84
77.25
77.00
76.75
59.11

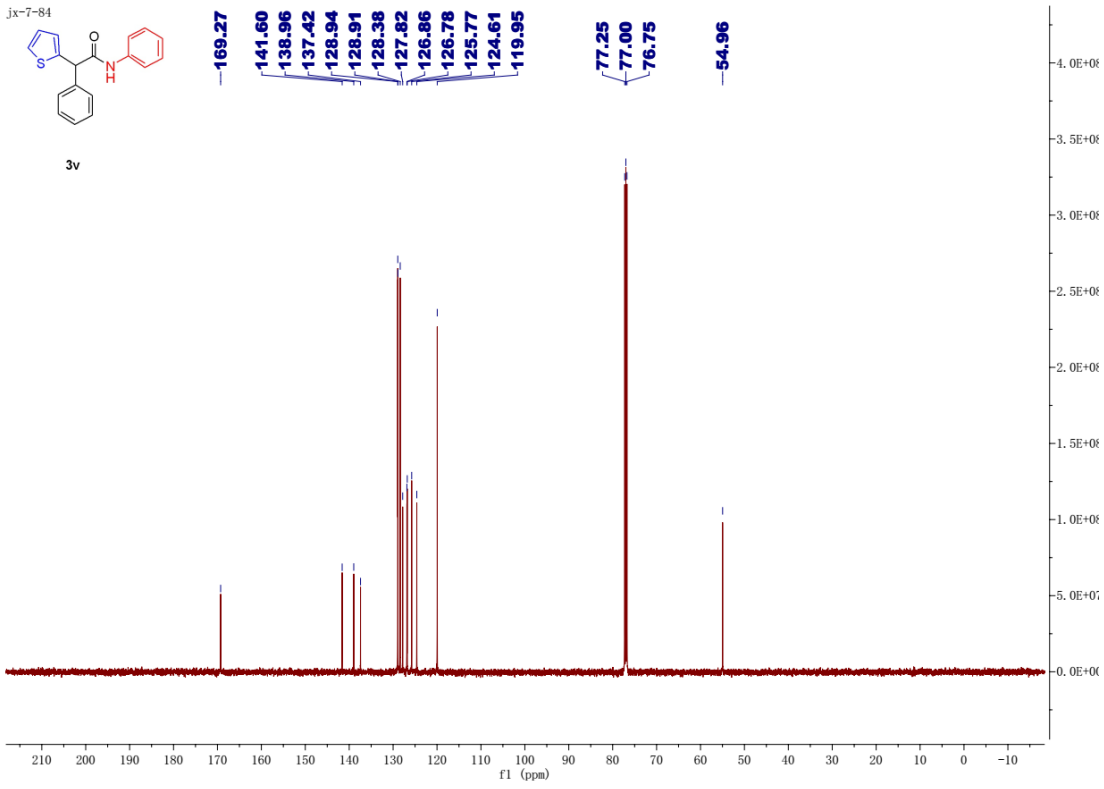

JX-7-78
MeO
O
N
H
3t
7.50
7.48
7.46
7.39
7.38
7.36
7.34
7.32
7.31
7.26
7.24
7.13
7.12
7.11
6.92
6.90
5.06
3.82
3.20
2.16
5.18
2.02
0.96
1.94
1.00
2.98
f1 (ppm)

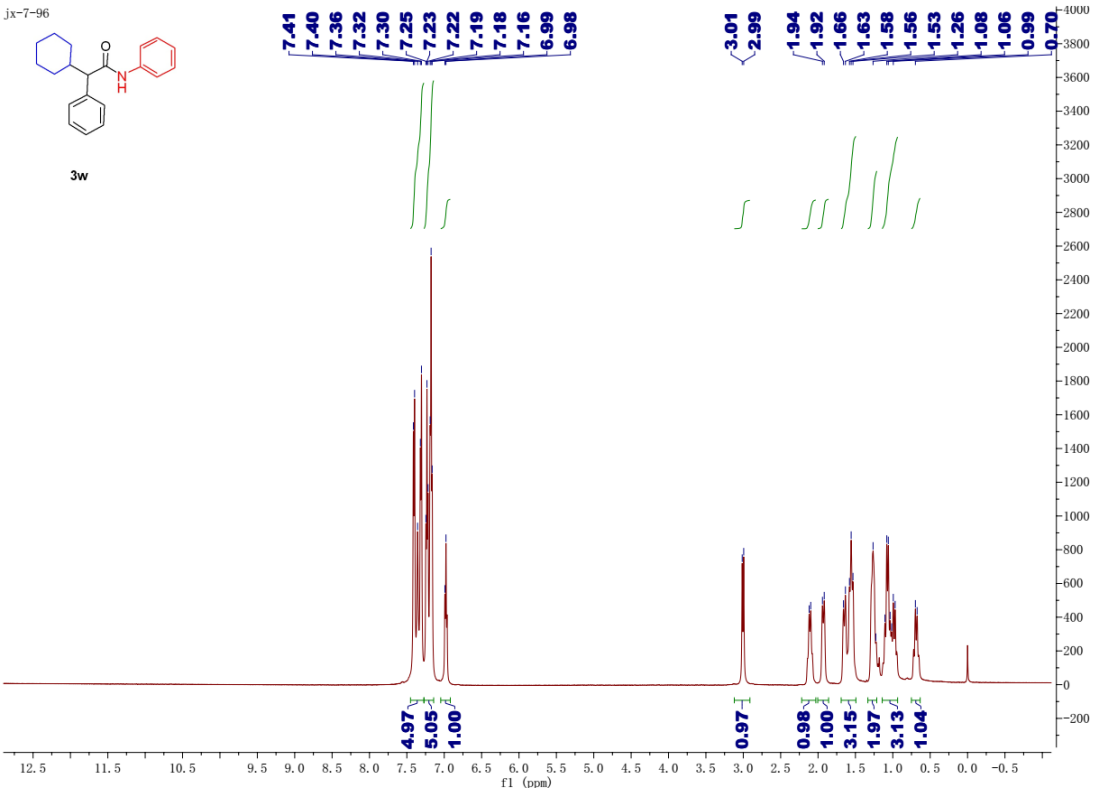

JX-7-78
MeO
O
N
H
3t
170.40
158.84
139.35
137.63
131.08
130.00
128.91
128.83
128.82
127.35
124.43
119.70
114.26
77.25
77.00
76.75
59.17
55.26
f1 (ppm)

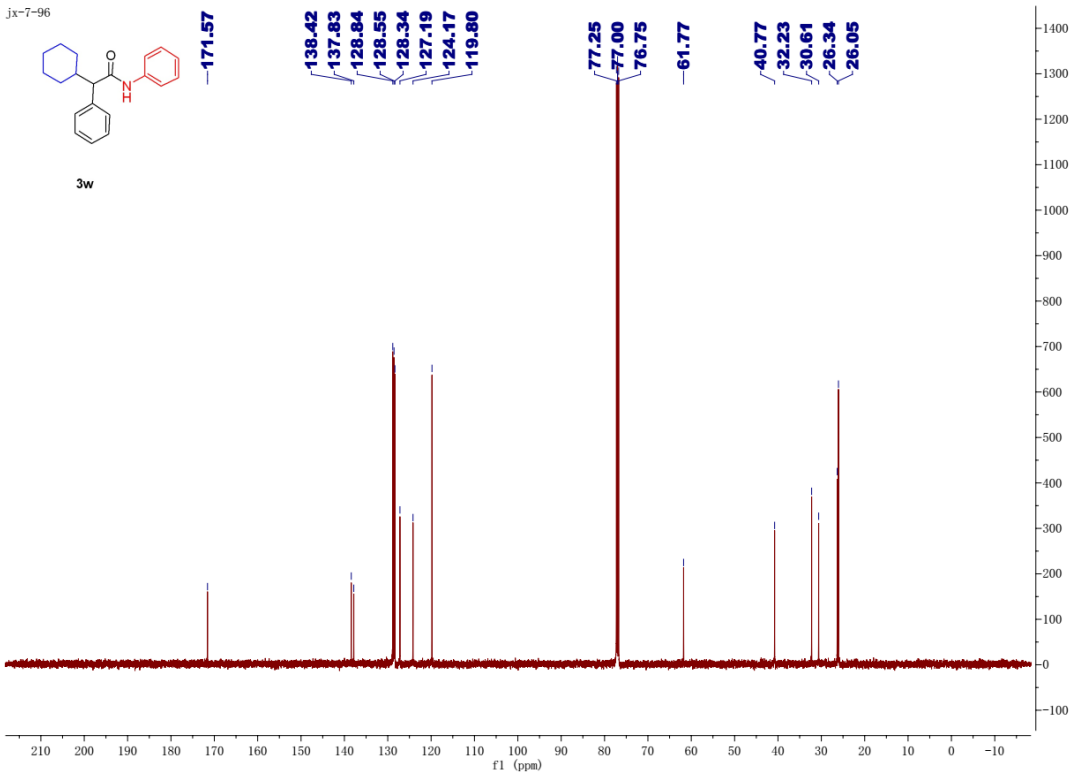

jx-7-7
7.87
7.86
7.84
7.51
7.33
7.31
7.30
7.28
7.14
7.13
7.12
5.44
NHPh
O
3u
8.03
8.95
3.43
1.11
1.00
f1 (ppm)


jx-7-7
169.94
132.65
128.98
128.75
128.00
127.83
127.65
127.04
126.39
126.24
119.85
77.25
77.00
76.75
60.29
NHPh
O
3u
f1 (ppm)


jx-7-84
7.72
7.47
7.45
7.41
7.40
7.38
7.36
7.35
7.33
7.32
7.29
7.29
7.28
7.26
7.12
7.10
7.09
7.00
7.00
6.98
5.26
S
O
N
H
3v
0.87
2.03
8.00
1.00
1.87
1.00


jx-7-84
3v
169.27
141.60
138.96
137.42
128.94
128.91
128.38
127.82
126.86
126.78
125.77
124.61
119.95
77.25
77.00
76.75
54.96
f1 (ppm)


jx-7-96
3w
7.41
7.40
7.36
7.32
7.30
7.25
7.23
7.22
7.19
7.18
7.16
6.99
6.98
3.01
2.99
1.94
1.92
1.66
1.63
1.58
1.56
1.53
1.26
1.08
1.06
0.99
0.70
4.97
5.05
1.00
0.97
0.98
1.00
3.15
1.97
3.13
1.04
f1 (ppm)


jx-7-96
171.57
138.42
137.83
128.84
128.55
128.34
127.19
124.17
119.80
77.25
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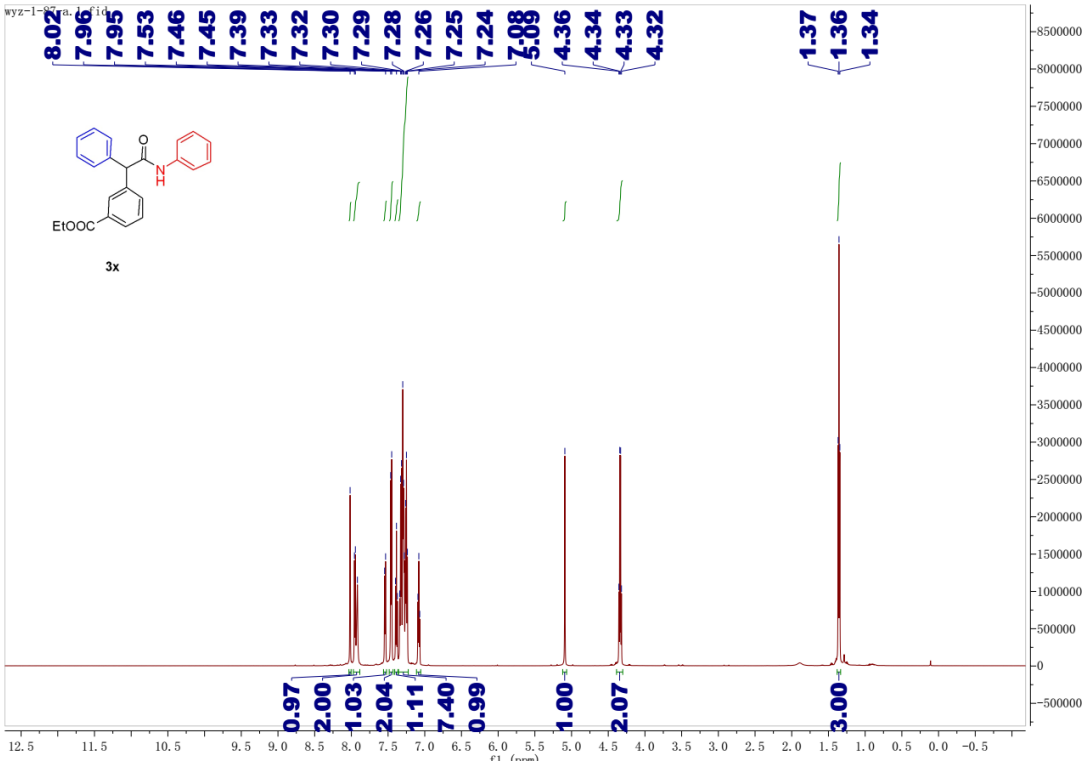
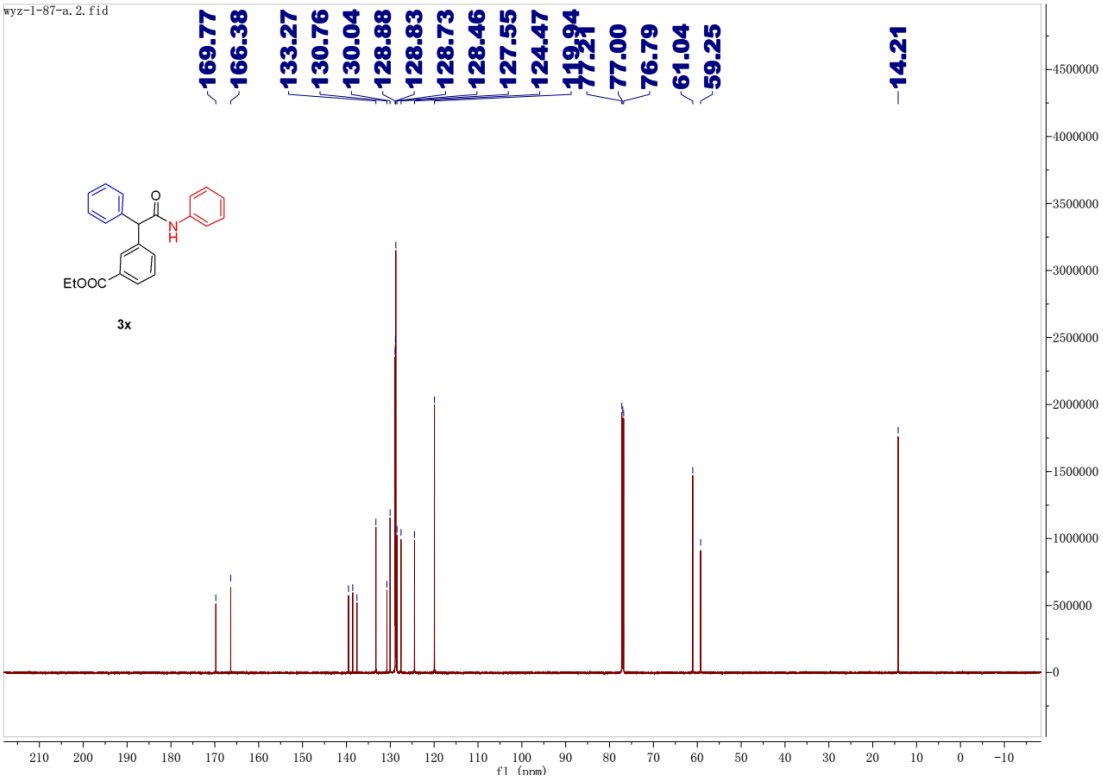
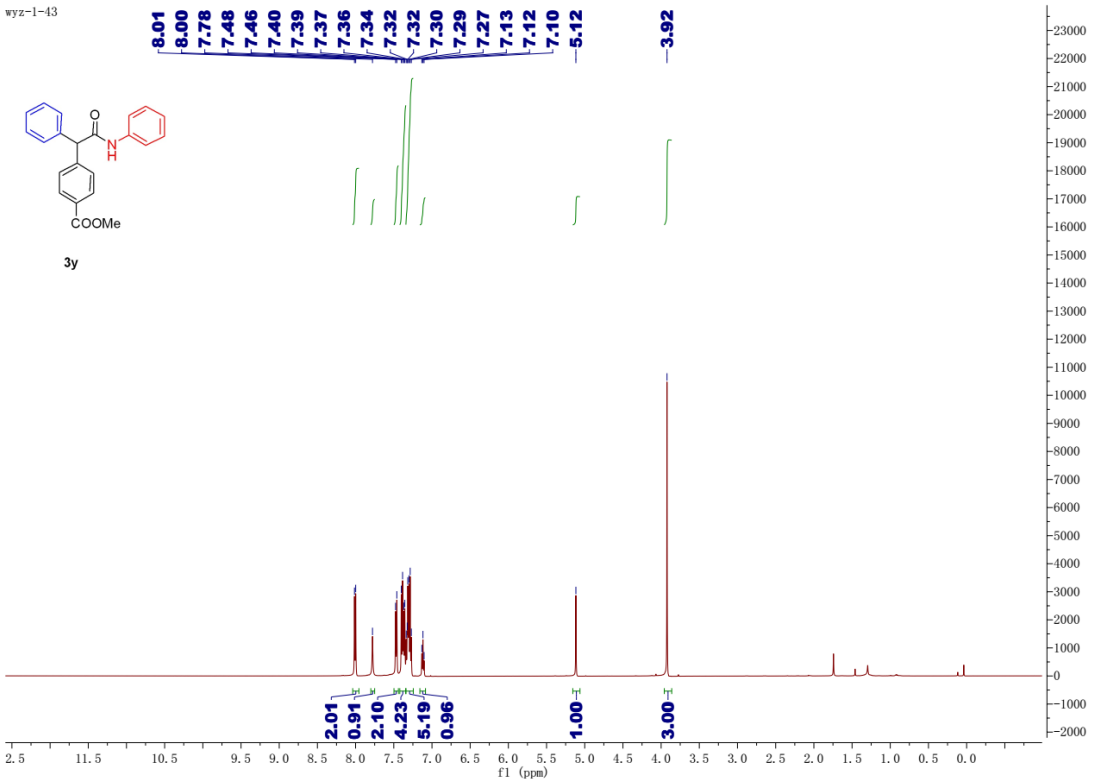
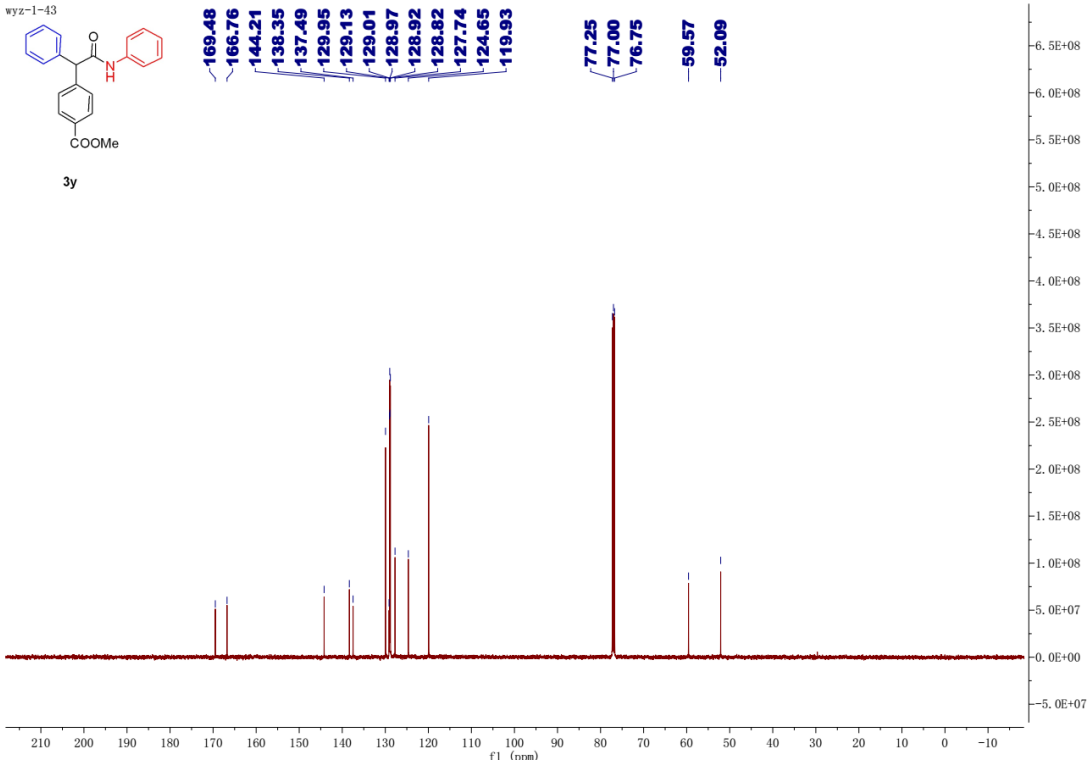
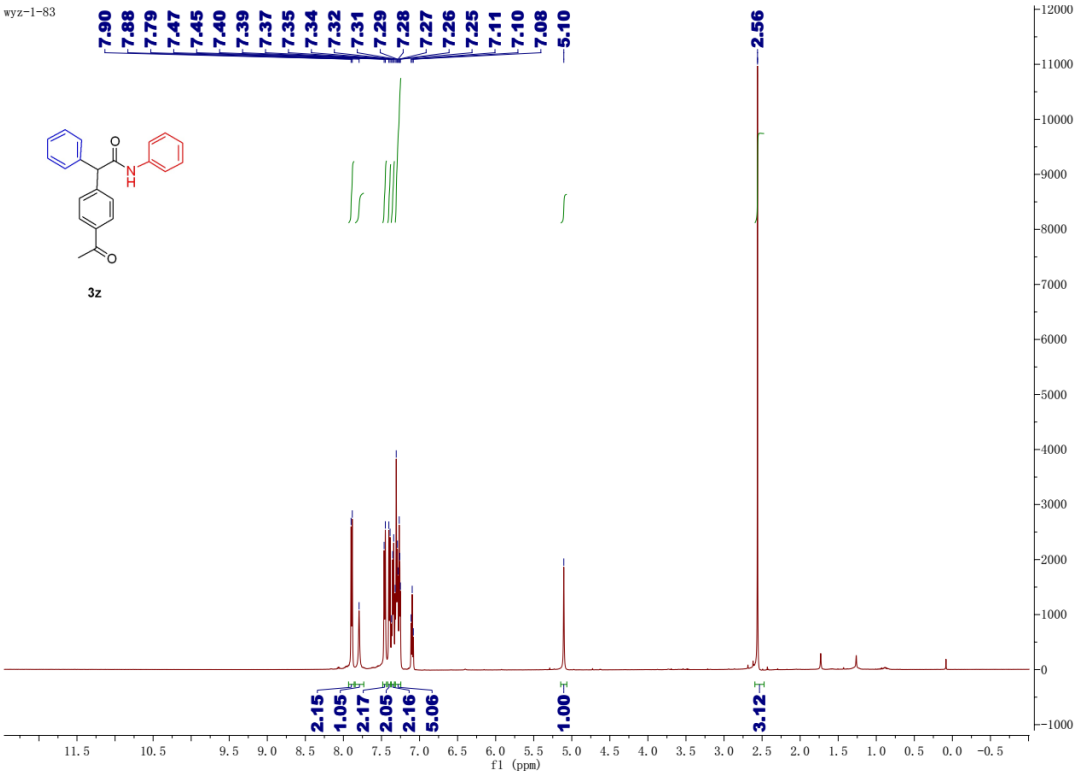
61.77
40.77
32.23
30.61
26.34
26.05
3w
f1 (ppm)

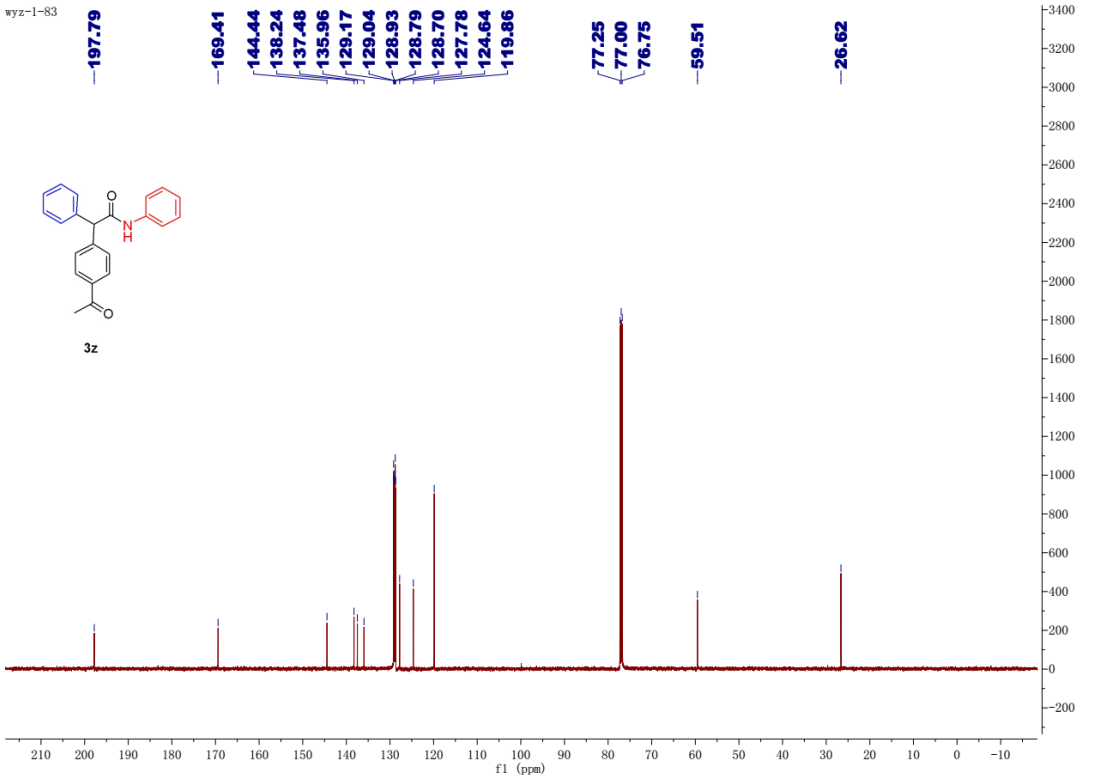

8.02
7.96
7.95
7.53
7.46
7.45
7.39
7.33
7.32
7.30
7.29
7.28
7.26
7.25
7.24
7.08
5.09
4.36
4.34
4.33
4.32
1.37
1.36
1.34
EtOOC
3x
0.97
2.00
1.03
2.04
1.11
7.40
0.99
1.00
2.07
3.00

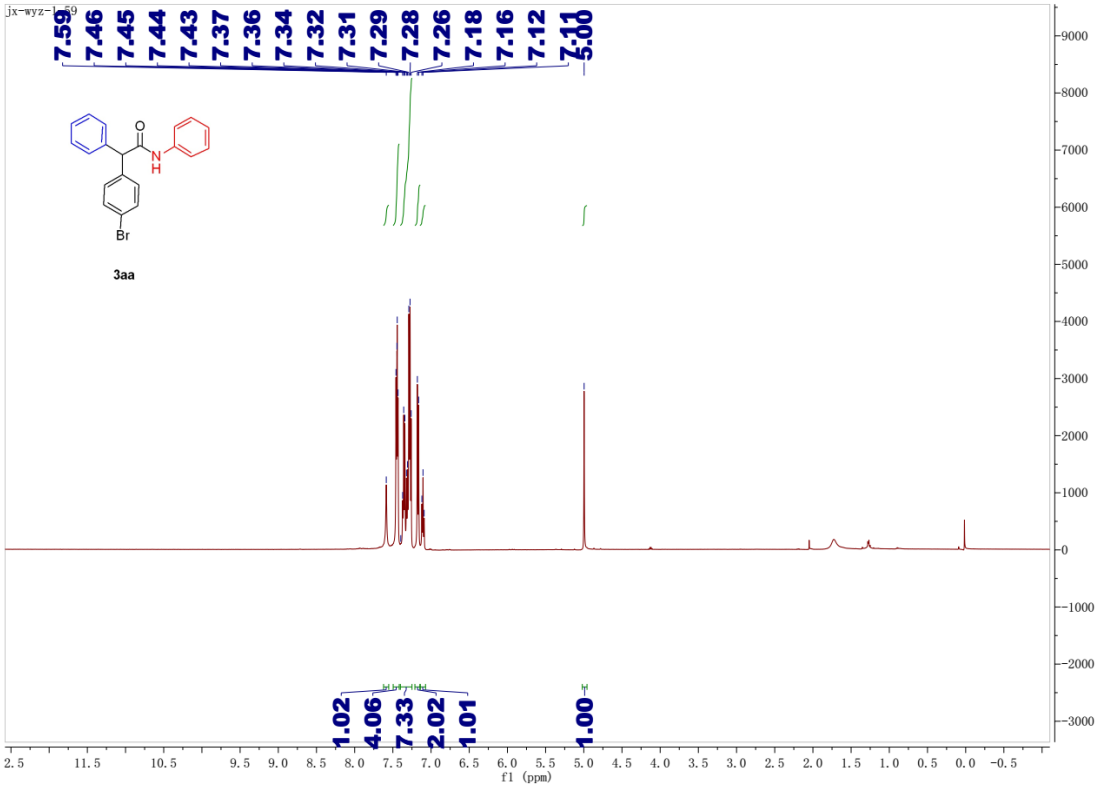

wyz-1-87-a.2.fid
169.77
166.38
133.27
130.76
130.04
128.88
128.83
128.73
128.46
127.55
124.47
119.94
77.21
77.00
76.79
61.04
59.25
14.21
EtOOC
3x
f1 (ppm)

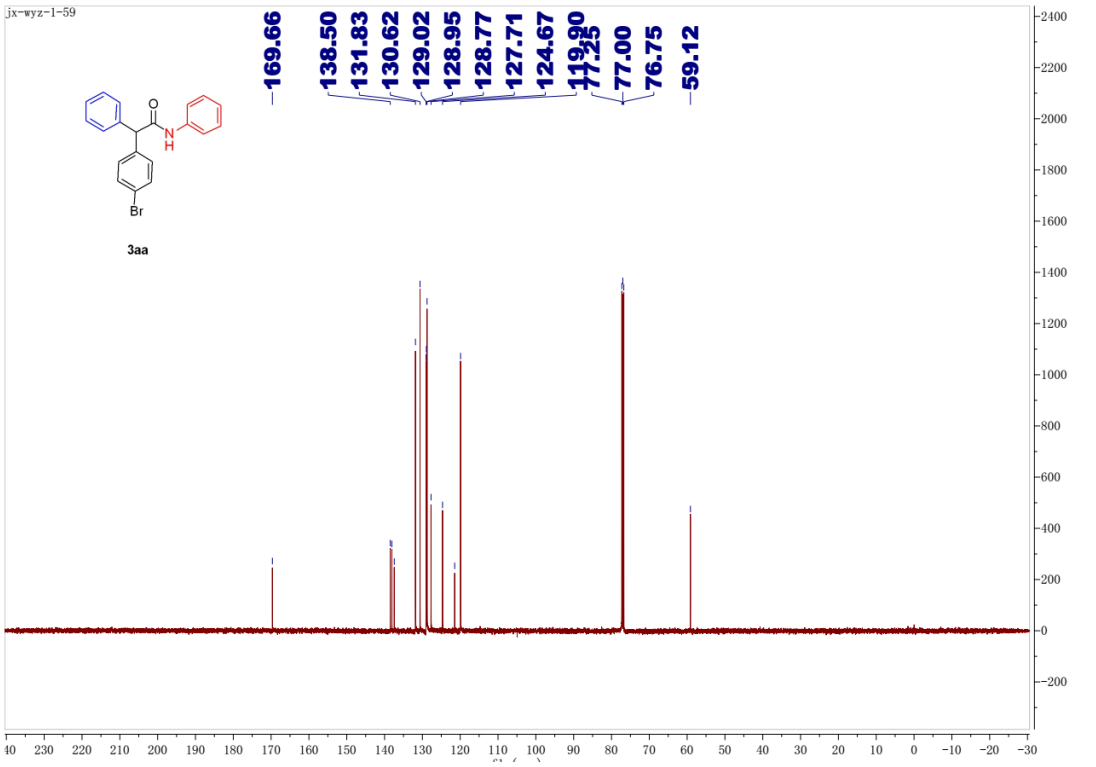

wyz-1-43
8.01
8.00
7.78
7.48
7.46
7.40
7.39
7.37
7.36
7.34
7.32
7.32
7.30
7.29
7.27
7.13
7.12
7.10
5.12
3.92
COOMe
3y
2.01
0.91
2.10
4.23
5.19
0.96
1.00
3.00
f1 (ppm)

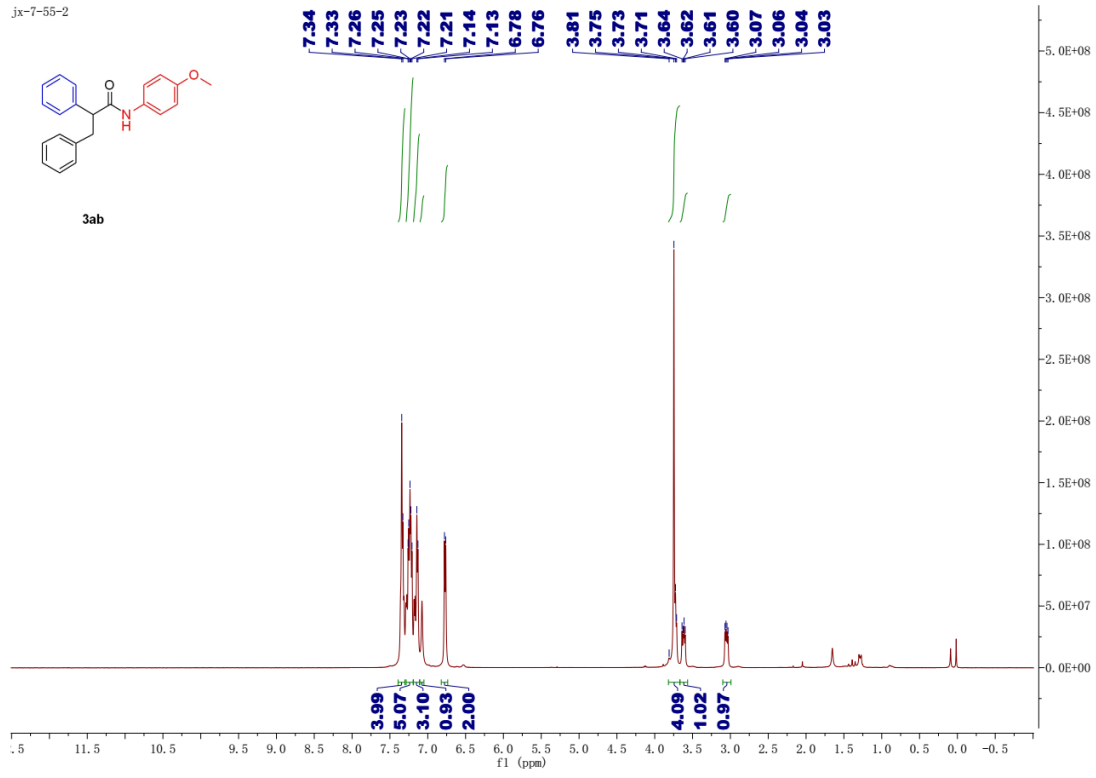

wyz-1-43
169.48
166.76
144.21
138.35
137.49
129.95
129.13
129.01
128.97
128.92
128.82
127.74
124.65
119.93
77.25
77.00
76.75
59.57
52.09
COOMe
3y
f1 (ppm)

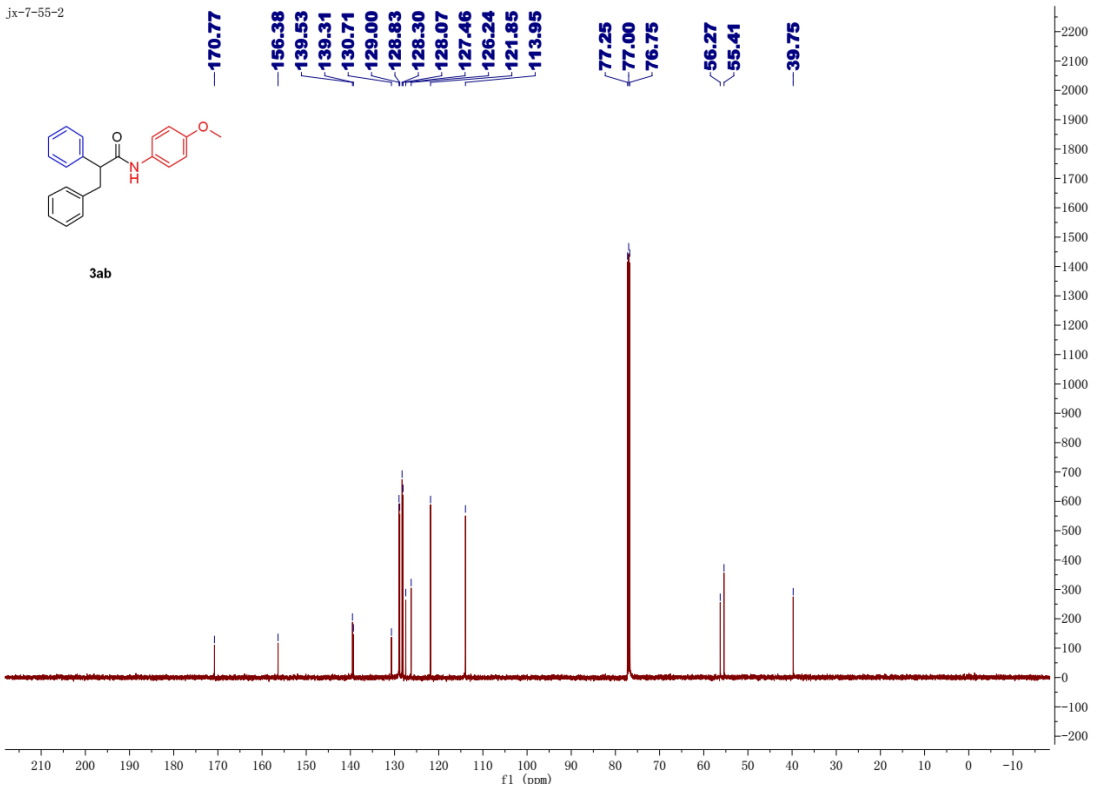

wyz-1-83
7.90
7.88
7.79
7.47
7.45
7.40
7.39
7.37
7.35
7.34
7.32
7.31
7.29
7.28
7.27
7.26
7.25
7.11
7.10
7.08
5.10
2.56
2.15
1.05
2.17
2.05
2.16
5.06
1.00
3.12
3z
f1 (ppm)

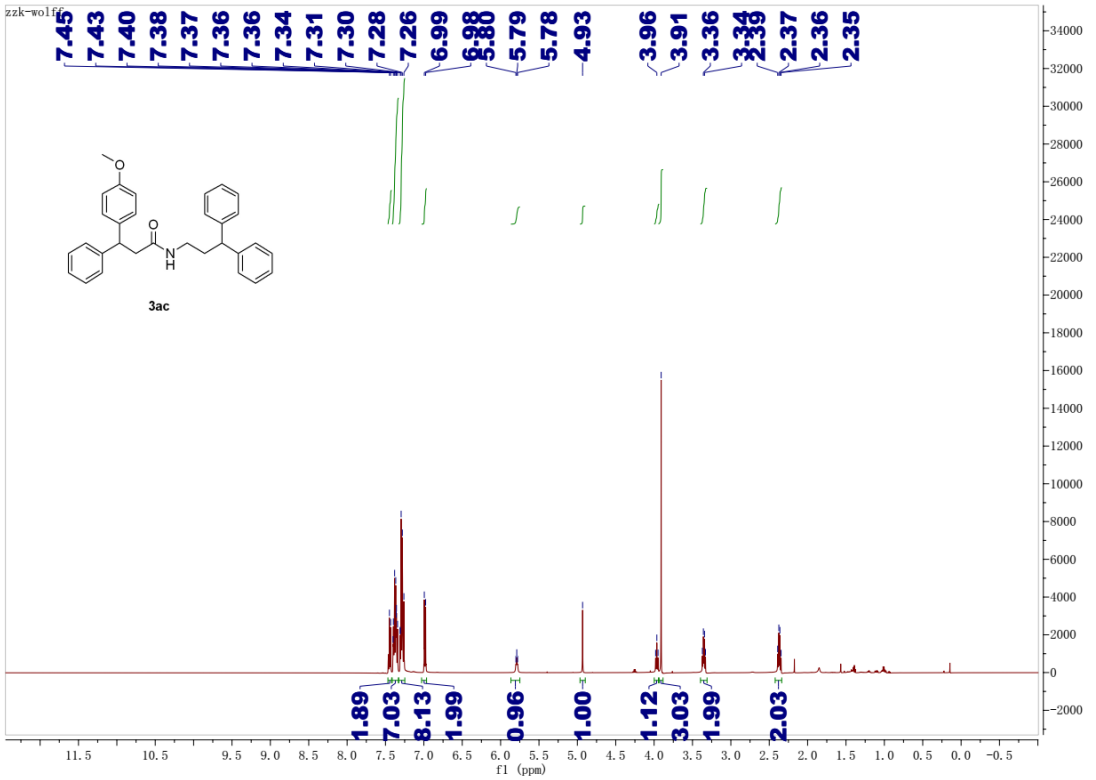

wyz-1-83
197.79
169.41
144.44
138.24
137.48
135.96
129.17
129.04
128.93
128.79
128.70
127.78
124.64
119.86
77.25
77.00
76.75
59.51
26.62
3z
f1 (ppm)


7.59
7.46
7.45
7.44
7.43
7.37
7.36
7.34
7.32
7.31
7.29
7.28
7.26
7.18
7.16
7.12
7.11
5.00
Br
3aa
1.02
4.06
7.33
2.02
1.01
1.00
f1 (ppm)


jx-wyz-1-59
169.66
138.50
131.83
130.62
129.02
128.95
128.77
127.71
124.67
119.90
77.25
77.00
76.75
59.12
Br
3aa


jx-7-55-2
7.34
7.33
7.26
7.25
7.23
7.22
7.21
7.14
7.13
6.78
6.76
3.81
3.75
3.73
3.71
3.64
3.62
3.61
3.60
3.07
3.06
3.04
3.03
3.99
5.07
3.10
0.93
2.00
4.09
1.02
0.97
f1 (ppm)
3ab


jx-7-55-2
170.77
156.38
139.53
139.31
130.71
129.00
128.83
128.30
128.07
127.46
126.24
121.85
113.95
77.25
77.00
76.75
56.27
55.41
39.75
3ab
f1 (ppm)


zzk-wolff
7.45
7.43
7.40
7.38
7.37
7.36
7.36
7.34
7.31
7.30
7.28
7.26
6.99
6.98
6.80
5.79
5.78
4.93
3.96
3.91
3.36
3.34
2.39
2.37
2.36
2.35
3ac
1.89
7.03
8.13
1.99
0.96
1.00
1.12
3.03
1.99
2.03
f1 (ppm)