

Supporting Information

Highly Efficient Markovnikov Hydroaminocarbonylation of Alkenes and Alkynes Catalyzed by a “Soluble” Heterogeneous Pd Catalyst

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1. Materials and Methods

Unless otherwise stated, all reagents and solvents were purchased commercially from Sigma-Aldrich, J&K, Aladdin or Alfa Aesar and used as received without further purification. Analytical thin-layer chromatography (TLC) was carried out with silica gel pre-coated glass plates (TLC-Silica gel GF254, coating thickness: 0.20-0.25 mm, particle size: 10-40 μm) purchased from Xinnuo Chemical (Yantai, China). The TLC was visualized with a UV lamp (254 or 365 nm). Column chromatography was carried out on silica gel (200-300 mesh) purchased from Xinnuo Chemicals (Yantai, China) with technical grade solvents as the eluent. All the yields referred to spectroscopically and chromatographically pure compounds.

The morphology of catalyst was examined by a H-7600 transmission electron microscope (TEM), a Tecnai G2 F30 high-resolution TEM (HRTEM). High resolution mass spectra (HRMS) were recorded on an Agilent 6210 Series 1969A ESI-TOF (time of flight) mass spectrometer using ESI (electrospray ionization) or EI (electron ionization). NMR spectra were recorded from a Bruker DRX-400, or DRX-600, instrument and calibrated using residual non-deuterated solvent (CDCl_3 : $\delta_{\text{H}} = 7.26 \text{ ppm}$, $\delta_{\text{C}} = 77.16 \text{ ppm}$; $\text{DMSO}-d_6$, $\delta_{\text{H}} = 2.50 \text{ ppm}$, $\delta_{\text{C}} = 39.52 \text{ ppm}$) as an internal reference.

2. Catalyst PPOC Solubility Test

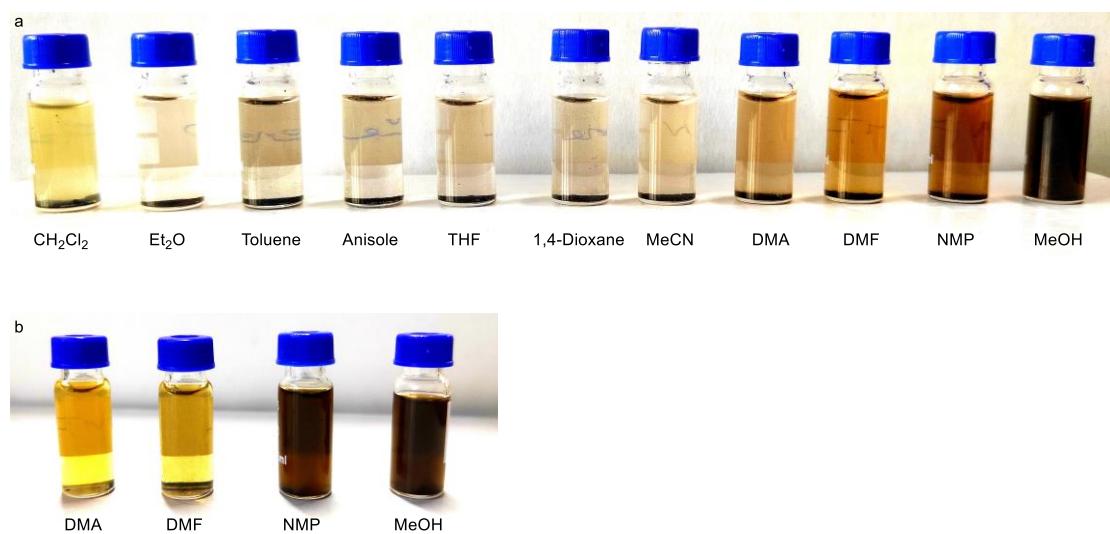


Figure S1. (a) Solubility of catalyst Pd@PPOC in different organic solvents, (b) Solubility of catalyst Pd@PPOC in 60 °C.

3. Optimizing the Reaction Conditions for Hydroaminocarbonylation

Table S1. Optimizing the Reaction Conditions for Hydroaminocarbonylation^a

				Conversion (%)	Yield (%)	3/4
Entry	CO (MPa)	Acid	Solvent			
1	5.0	HCl	THF	30	26	10:1
2	5.0	TsOH	THF	35	23	10:1
3	5.0	B(OH) ₃ /5-ClSA	THF	NR	--	--
4	5.0	HONH ₂ ·HCl	THF	40	12	9:1
5	5.0	HCl	Toluene	NR	--	--
6	5.0	HCl	EtOH	50	40	2:1
7	5.0	HCl	Et ₂ O	36	19	8:1
8	5.0	HCl	anisole	37	7	9:1
9	5.0	HCl	MeCN	87	32	4:1
10	5.0	HCl	Dioxane	47	9	1:3
11	5.0	HCl	DCE	NR	--	--
12	5.0	HCl	DMF	71	46	28:1
13	5.0	HCl	DMA	80	55	31:1
14	5.0	HCl	NMP	100	98	55:1
15 ^b	5.0	HCl	NMP	92	81	74:1
16	2.0	HCl	NMP	98	90	26:1
17 ^c	5.0	HCl	NMP	100	98	66:1
18 ^c	4.0	HCl	NMP	98	96	77:1
19 ^{cd}	4.0	HCl	NMP	100	98	77:1

^aReaction conditions: styrene (0.5 mmol), aniline (0.6 mmol), Acid (0.6 mmol), Pd@PPOC (10 mg), CO (5.0 MPa), Solvent (3 mL), 110 °C, 12 h. ^bPd@PPOC (2 mg); ^cPd@PPOC (4 mg); ^d16 h.

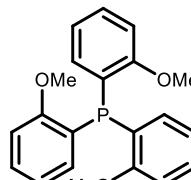
We explored the hydroaminocarbonylation of styrene with aniline using Pd@PPOC as the catalyst in the CO atmosphere and THF as the solvent. The reaction resulted in the formation of only 26% of the amide with a 10/1 branched/linear (b/l) ratio (Table S1, entry 1). With this result in hand, a set of parameters was screened to achieve higher reactivity and regioselectivity, including reaction time, the amount of catalyst loading, the pressure of CO, and the type of acids and solvents. Subsequently, we examined the effect of the acid and observed that other acids, such as TsOH, B(OH)₃/5-ClSA, and HONH₂·HCl, provided unsatisfactory results in terms of reactivity and selectivity (entries 2-4). Different solvents were investigated to improve the reactivity and

selectivity in the reaction (entries 5-11), the yield of the amide increased when EtOH was used, affording the amide in 40% yield but with poor selectivity (entry 6). DMF as the solvent showed amazing selectivity ($b/l = 28:1$) in the formation of the desired product (entry 12). Then the effect of DMA and NMP as solvent were studied, the later gives an excellent yield (98%) and selectivity ($b/l = 55:1$) for the branched amide (entries 13-14). Decreasing the catalyst loading resulted in lower yield (entry 15), while decreasing the pressure of CO led to poor selectivity (entry 16). Considering the high boiling point of NMP, we investigated the effect of combination THF and NMP, affording poor yield and selectivity (entry 17). Decreasing the catalyst loading and the pressure of CO were detrimental to the reaction and result in excellent yield (98%) and selectivity ($b/l = 77:1$) when the reaction time extended from 12 h to 16 h (entry 19).

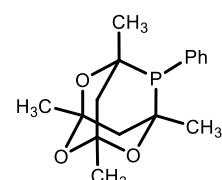
4. Comparison of Turnover Frequency

Table S2. Comparison of Turnover Frequencies of Hydroaminocarbonylation of Olefins and Amines over Different Palladium Catalytic Systems

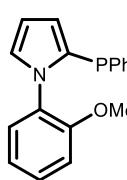
	$\text{R}^1\text{C}\equiv\text{C}$	+	CO	+	R^2NH_2	[Pd]/L, Solvent, Temperature		$\text{R}^1\text{CH}(\text{NHR}^2)\text{C}(=\text{O})\text{H}$
Entry	Cat. (%)	L.	CO (MPa)	Sol.	Temp. (°C)	Y. (%)	b/l	TOF (h ⁻¹)
1 ¹	PdCl ₂ (5.0)	L1	5.0	THF	125	27-98	>99	0.27-0.98
2 ²	PdCl ₂ (5.0)	L2	2.0	Butanone	120	45-96	4.0-67	0.19-0.4
3 ³	PdCl ₂ (1.5)	L3	4.0	THF	125	39-99	1.6-99	1.1-2.75
4 ⁴	Pd(^t Bu ₃ P) ₂ (5.0)	--	2.0	anisole	120	14-98	0.4-20	0.12-0.82
5 ⁵	Pd(^t Bu ₃ P) ₂ (2.0)	--	3.0	amide	120	46-89	3.8-49	0.96-1.85
6 ⁶	Pd(^t Bu ₃ P) ₂ (5.0)	--	3.0	NMP	120	30-89	4.3-49	0.25-0.74
7 ⁷	PdI ₂ (10.0)	L4	5.0	THF	25	56-98	1.4-99	0.08-0.14
8 ⁸	Pd(PPh ₃)Cl ₂ (3.0)	--	4.5	THF	110	48-96	4.9-99	0.67-1.33
9	Pd@PPOC (0.8)	--	4.0	NMP	110	53-96	38->99	5.5-10.0



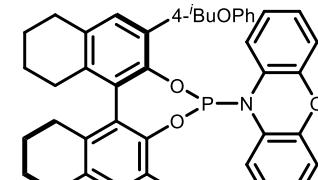
L1



L2



L3



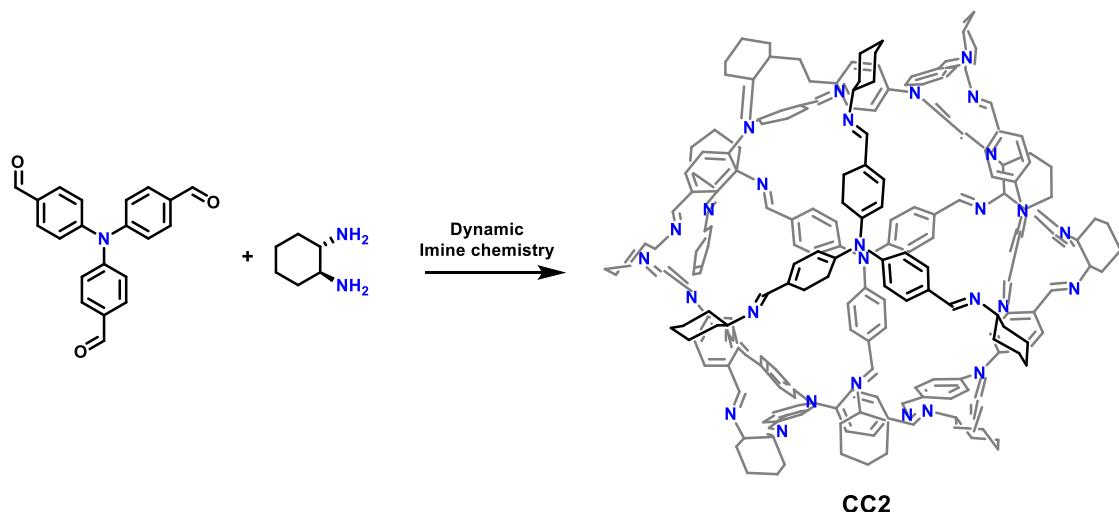
L4

5. Synthesis of Heterogeneous Palladium Catalyst

1) Synthesis of Pd@PPOC Catalyst

To an oven dried 100 mL two neck flask, porous cage PPOC⁹ (100 mg, 0.107 mmol), Pd(OAc)₂ (72.6 mg, 0.324 mmol) and solvent mixture DCM:MeOH (20 mL, 1:1, v/v) was added under N₂. The resulting light yellow colour solution was stirred at room temperature for 4 h. The colour of the solution gradually changed to dark brown indicating the complexation of Pd(OAc)₂ with porous cage. After 4 h, to this solution NaBH₄ (36 eq.) was added portion wise for 10 minutes and the resulting mixture was stirred at room temperature for overnight. During this time the colour of the solution was changed to black, indicating the reduction and deposition of palladium nanoparticles over the cage. The solvent was removed under reduced pressure, the black solid was filtered, washed with water and ethanol dried overnight to get Pd@PPOC as black solid in 61% yield (82 mg). The Pd loading was measured by ICP-AES to be 15 wt%.

2) Synthesis of Pd@CC2 Catalyst



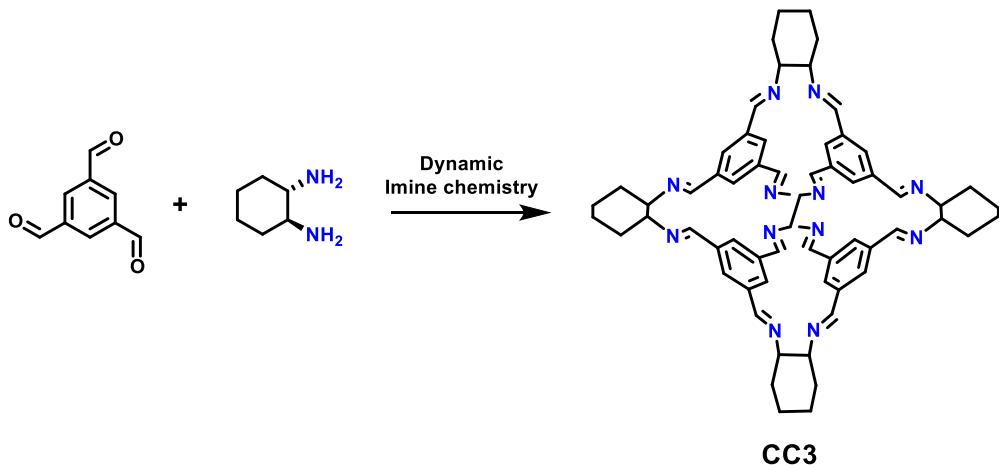
Scheme S1. Synthesis of CC2

Organic porous cage CC2 was synthesized according to a previous literature.¹⁰ In a 250 mL round-bottom flask, 120 mL of a CHCl₃ solution of [(S,S)-1,2-cyclohexanediamine] (31 mg, 0.28 mmol) was added slowly to a stirring solution of tris(4-formylphenyl)amine (100 mg, 0.18 mmol) dissolved in 60 mL of CHCl₃. The resulting

reaction mixture was heated to reflux for 48 h. After completion of the reaction, solvent was removed, and the obtained pale yellow solid was washed with CH₃CN several times with isolated yield of 78%. ¹H NMR (CDCl₃, 400 MHz): δ 8.35 (s, 12H), 8.24 (s, 12H), 7.61–7.55 (m, 48H), 7.08–7.02 (m, 48H), 3.56–3.48 (m, 24H), 1.75 (br, 24H), 1.49 (br, 24H). HRMS (ESI) *m/z* Calcd for C₂₄₀H₂₄₀N₃₂, [M+H]⁺ 1786.9995, found 1787.0017.

Pd@CC2 was prepared following the same synthetic protocol employed for Pd@PPOC. The Pd loading was measured by ICP-AES to be 35 wt%.

3) Synthesis of Pd@CC3 Catalyst



Scheme S2. Synthesis of CC3

Organic porous cage CC3 was synthesized according to a previous literature.¹¹ A typical procedure is as follows: dichloromethane (10 mL) was added slowly onto solid 1,3,5-triformylbenzene (0.5 g, 3.086 mmol) at room temperature. Trifluoroacetic acid (10 μL) was added directly to this solution as a catalyst for imine bond formation. Finally, a solution of (R,R)-1,2-diaminocyclohexane (0.5 g, 4.464 mmol) in dichloromethane (10 mL) was added. The mixture was covered and left to stand for one week. The crystals grew on the sides of the vessel. The crystalline product was removed by centrifugations and washed with 95% methanol/5% dichloromethane, and further dried at 100 °C under vacuum overnight. ¹H NMR (CDCl₃, 400 MHz): δ 8.39 (t, 3H), 8.21 (s, 3H), 8.17 (s, 6H), 8.12 (s, 3H), 7.92 (s, 3H), 7.76 (s, 3H), 7.18 (s, 3H), 3.45–3.36 (m, 12H), 1.88–1.78 (m, 24H), 1.67–1.63 (m, 12H), 1.49–1.44 (m, 12H). HRMS (ESI) *m/z* Calcd for C₇₂H₈₅N₁₂, [M+H]⁺ 1117.7020, found 1117.7028.

Pd@CC3 was prepared following the same synthetic protocol employed for Pd@PPOC. The Pd loading was measured by ICP-AES to be 48 wt%.

4) Synthesis of Pd@PVP Catalyst

Pd@PVP was prepared following the same synthetic protocol employed for Pd@PPOC.

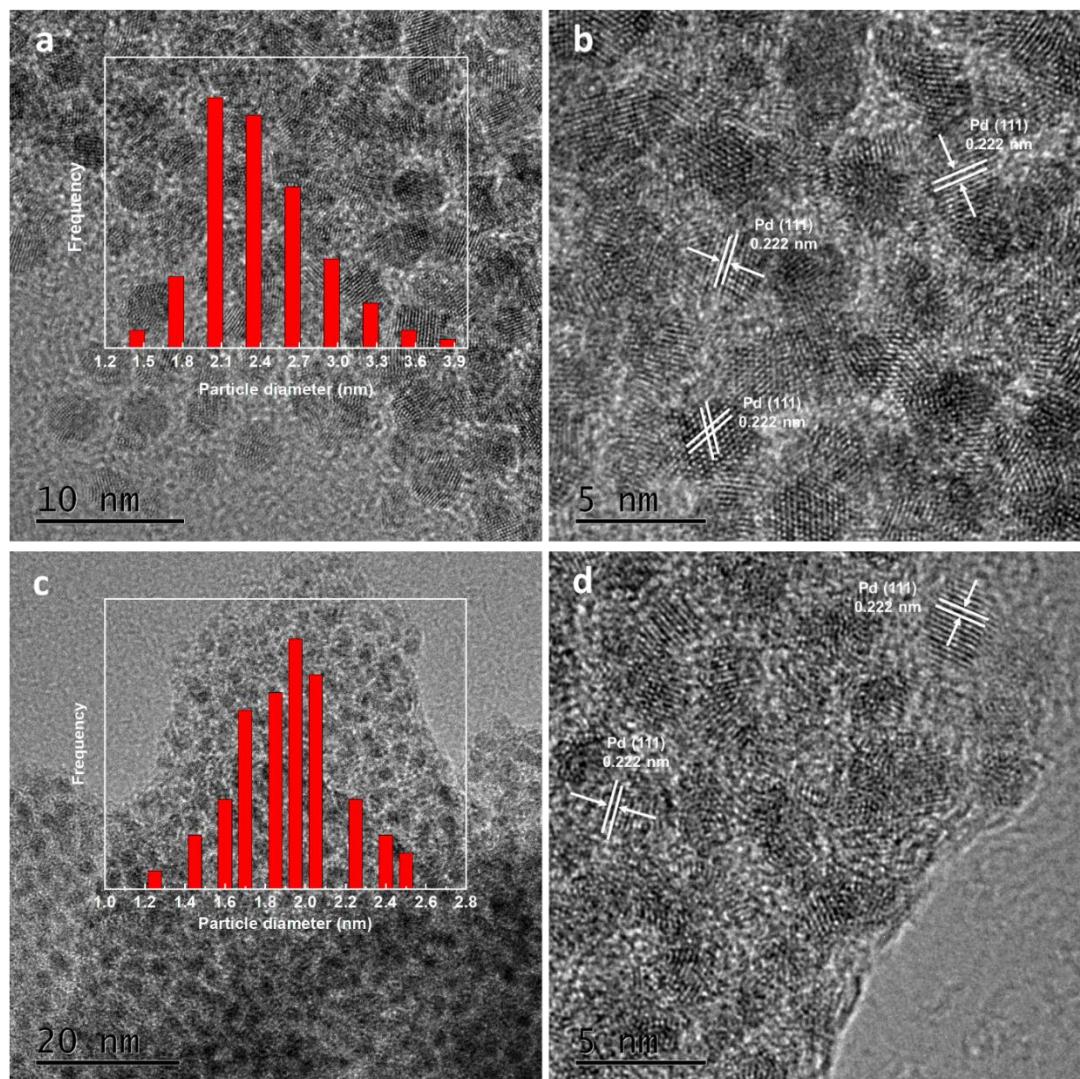


Figure S2. HR-TEM images of Pd@CC3 (a, b), Pd@CC2 (c, d) and particle size distribution (inset), respectively.

6. Results of Stability and Recyclability of Pd@PPOC

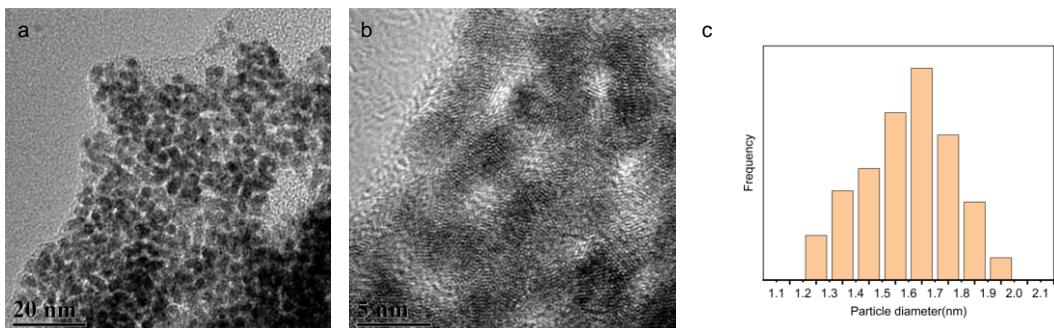
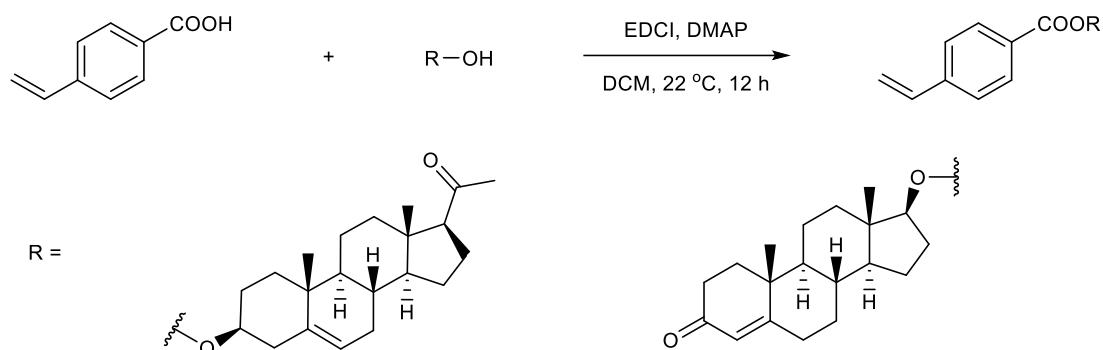


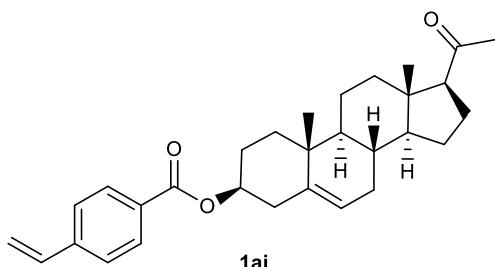
Figure S3. (a,b) HR-TEM images, and (c) size distribution of the catalyst Pd@PPOC after recycles.

We investigated the stability and recyclability of the Pd@PPOC catalyst for the hydroaminocarbonylation reaction of styrene and aniline under the optimized condition. After completion of each run, the catalyst was collected through centrifugation and washed with water and ethanol. After drying under vacuum at 65 °C, the recovered catalyst was used for the next cycle without any reactivation process. The palladium nanocatalyst could be used seven times consecutively with negligible changes in reactivity and selectivity, indicating its excellent recyclability and stability. No obvious aggregation or increase in particle size from the fresh Pd@PPOC was observed, as identified by HR-TEM for the used one, which corroborates the stability of the catalyst.

7. Synthesis of Alkenes 1ai and 1aj.

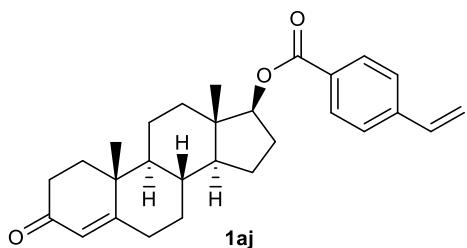


To a solution of alcohol (2 mmol, 1 equiv), 4-vinylbenzoic acid (2.4 mmol, 1.2 equiv) and DMAP (0.2 mmol, 0.1 equiv) in dry CH_2Cl_2 (50 mL) was added EDCI (2.4 mmol, 1.2 equiv). After stirring for 12 h at 22 °C, The resultant mixture was filtered with Celite pad, the filtrate was washed with sat. NaHCO_3 and the organic layers were dried over Na_2SO_4 , filtered, and concentrated. The obtained residue was purified by silica gel column chromatography to afford **1ai-1aj**.



(*3S,8S,9S,10R,13S,14S,17S*)-17-acetyl-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 4-vinylbenzoate ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 8.2$ Hz, 2H), 7.45 (d, $J = 8.2$ Hz, 2H), 6.75 (dd, $J = 17.6, 10.9$ Hz, 1H), 5.86 (d, $J = 17.6$ Hz, 1H), 5.46–5.34 (m, 2H), 4.92–4.81 (m, 1H), 2.55 (t, $J = 8.9$ Hz, 1H), 2.47 (d, $J = 7.7$ Hz, 2H), 2.24–2.10 (m, 4H), 2.09–1.97 (m, 3H), 1.93 (dt, $J = 13.0, 3.1$ Hz, 1H), 1.80–1.42 (m, 8H), 1.30–1.13 (m, 3H), 1.11–1.01 (m, 4H), 0.65 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 209.7, 165.9, 141.9, 139.8, 136.2, 130.04, 129.98, 126.2, 122.6, 116.5, 74.5, 63.8, 57.0, 50.0, 44.1, 38.9, 38.3, 37.2, 36.8, 32.0, 31.9, 31.7, 28.0, 24.6, 23.0, 21.2, 19.5, 13.4. HRMS (ESI) m/z Calcd for $\text{C}_{30}\text{H}_{38}\text{O}_3\text{Na}$ [M+Na] $^+$ 469.27132, found 469.27228.



(8R,9S,10R,13S,14S,17S)-10,13-dimethyl-3-oxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-17-yl 4-vinylbenzoate ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 8.3$ Hz, 2H), 7.46 (d, $J = 8.3$ Hz, 2H), 6.75 (dd, $J = 17.6, 10.9$ Hz, 1H), 5.86 (d, $J = 17.6, 10.9$ Hz, 1H), 5.74 (s, 1H), 5.38 (d, $J = 11.0$ Hz, 1H), 4.85 (t, $J = 7.9$ Hz, 1H), 2.48–2.25 (m, 5H), 2.07–1.99 (m, 1H), 1.92–1.84 (m, 2H), 1.78–1.56 (m, 5H), 1.50–1.36 (m, 2H), 1.32–0.94 (m, 10H). ^{13}C NMR (101 MHz, CDCl_3) δ 199.5, 171.0, 166.4, 142.0, 136.2, 129.95, 129.89, 126.2, 124.1, 116.6, 83.1, 53.9, 50.4, 43.0, 38.8, 36.9, 35.9, 35.6, 34.1, 32.9, 31.7, 27.8, 23.8, 20.7, 17.6, 12.4. HRMS (ESI) m/z Calcd for $\text{C}_{28}\text{H}_{35}\text{O}_3$ [M+H] $^+$ 419.25807, found 419.25885.

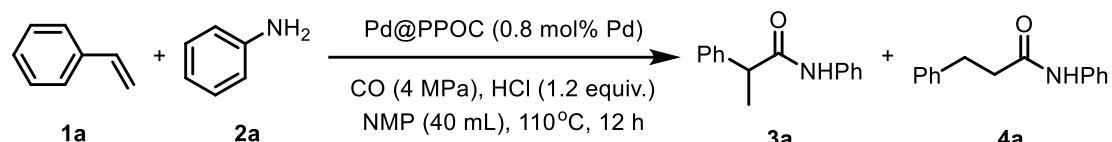
8. General Procedure for Pd@PPOC Catalyzed

Hydroaminocarbonylation of Alkenes and Alkynes

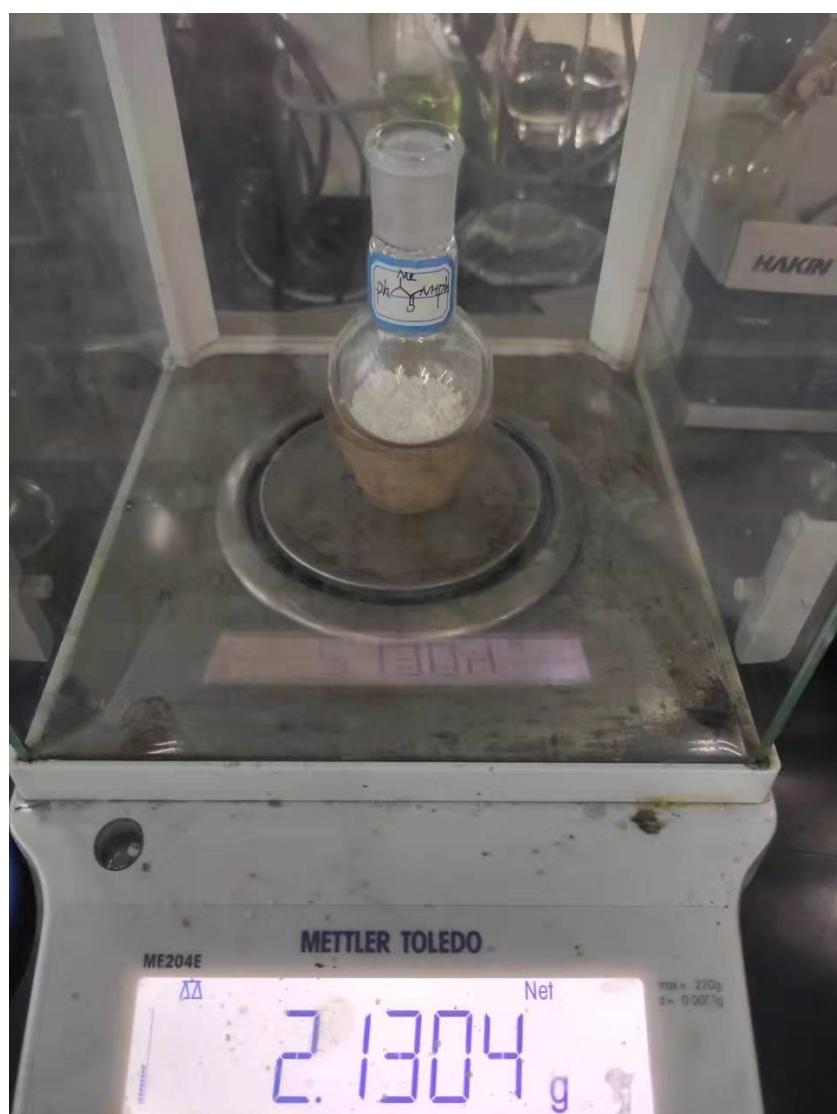
To a 50.0 mL glass vessel was added alkene or alkyne (0.5 mmol, 1.0 equiv), amine (0.6 mmol, 1.2 equiv), concentrated HCl (0.6 mmol, 1.2 equiv), Pd@PPOC (4 mg, 0.8 mol%), and NMP (3.0 mL). The glass vessel was then put into an autoclave. The autoclave was evacuated and backfilled with CO for three times in a well ventilated fume hood, and then pressurized to 40 atm of CO. The reaction mixture in autoclave was stirred at 110 °C for 12 h under 40 atm of CO. The autoclave was cooled to room temperature, and the gas was released slowly in a well-ventilated hood. The ratio of branched and linear amides was determined by NMR analysis of the crude products. The reaction mixture was concentrated and purified by column chromatography on silica gel to afford the desired amide or α,β -unsaturated amide.

9. Gram-Scale Synthesis of 3a

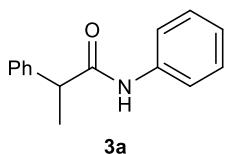
Following the general procedure for Pd@PPOC catalyzed hydroaminocarbonylation of alkenes and alkynes, styrene (1.15 mL, 10 mmol, 1.0 equiv) reacted with aniline (1.09 mL, 12 mmol, 1.2 equiv) in NMP (40 mL) in CO (4.0) for 12 h at 110°C. After work-up as usual, purification by silica gel column chromatography (petroleum ether/EtOAc 4:1) afforded **3a** (2.13 g, 9.5 mmol, 95%).



Scheme S3. Gram-scale synthesis of **3a**

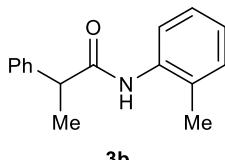


10. NMR Data



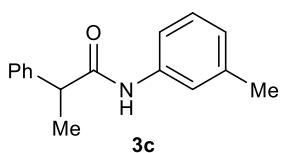
*N, 2-diphenylpropanamide*¹² ¹H NMR (400 MHz, CDCl₃) δ 7.48–7.20 (m, 9H), 7.13–6.97 (m, 2H), 3.72 (q, *J* = 7.1 Hz, 1H), 1.61 (d, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.2, 140.9, 137.8, 129.2, 128.9, 127.7, 127.6, 124.3, 119.7, 48.2, 18.6.



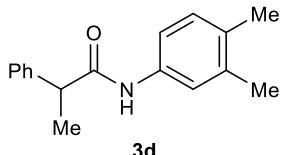
*N-(2-methylphenyl)-2-phenylpropanamide*¹³ ¹H NMR (400 MHz, CDCl₃) δ 7.87 (d, *J* = 7.9 Hz, 1H), 7.37 (d, *J* = 26.1 Hz, 5H), 7.18 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 7.1 Hz, 1H), 7.01 (t, *J* = 7.2 Hz, 1H), 6.90 (s, 1H), 3.79 (q, *J* = 7.0 Hz, 1H), 1.89 (s, 3H), 1.65 (d, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.4, 141.1, 135.8, 130.4, 129.3, 128.3, 128.0, 127.8, 126.8, 124.9, 48.2, 18.1, 17.2.



*N-(3-methylphenyl)-2-phenylpropanamide*¹³ ¹H NMR (400 MHz, CDCl₃) δ 7.42–7.27 (m, 6H), 7.24–7.13 (m, 3H), 6.89 (d, *J* = 7.3 Hz, 1H), 3.71 (q, *J* = 7.1 Hz, 1H), 2.29 (s, 3H), 1.60 (d, *J* = 7.1 Hz, 3H).

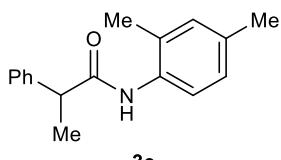
¹³C NMR (101 MHz, CDCl₃) δ 172.4, 141.0, 138.9, 137.8, 129.1, 128.7, 127.7, 127.6, 125.1, 120.4, 116.8, 48.1, 21.5, 18.6.



N-(3,4-dimethylphenyl)-2-phenylpropanamide ¹H NMR (400 MHz, CDCl₃) δ 7.37 (d, *J* = 4.3 Hz, 4H), 7.34–7.27 (m, 1H), 7.25–7.22 (m, 1H), 7.20–7.11 (m, 2H), 7.01 (d, *J* = 8.1 Hz, 1H), 3.70 (q, *J* = 7.1 Hz, 1H), 2.19 (s, 6H), 1.59 (d, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.4, 141.2, 137.2, 135.7, 132.7, 129.9, 129.2, 127.8, 127.6, 121.3, 117.4, 48.1, 19.9, 19.2, 18.7.

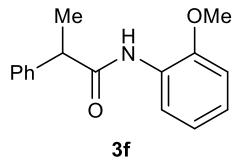
HRMS (ESI) *m/z* Calcd for C₁₇H₂₀ON [M+H]⁺ 254.15394, found 254.15431.



N-(2,4-dimethylphenyl)-2-phenylpropanamide ^1H NMR (400 MHz, CDCl_3) δ 7.66 (d, $J = 8.2$ Hz, 1H), 7.44–7.29 (m, 5H), 6.97 (d, $J = 8.2$ Hz, 1H), 6.90 (s, 1H), 6.81 (s, 1H), 3.77 (q, $J = 7.2$ Hz, 1H), 2.25 (s, 3H), 1.87 (s, 3H), 1.64 (d, $J = 7.2$ Hz, 3H).

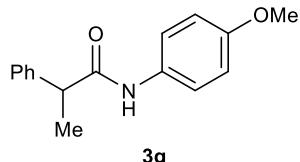
^{13}C NMR (101 MHz, CDCl_3) δ 172.3, 141.1, 134.6, 133.1, 131.0, 129.2, 128.6, 127.9, 127.7, 127.2, 122.5, 48.0, 20.8, 18.1, 17.1.

HRMS (ESI) m/z Calcd for $\text{C}_{17}\text{H}_{20}\text{ON} [\text{M}+\text{H}]^+$ 254.15394, found 254.15430.



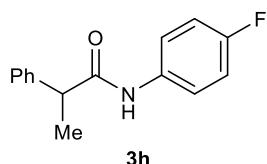
N-(2-methoxyphenyl)-2-phenylpropanamide ^{13}H NMR (400 MHz, CDCl_3) δ 8.36 (d, $J = 7.9$ Hz, 1H), 7.77 (s, 1H), 7.43–7.35 (m, 4H), 7.35–7.26 (m, 1H), 7.03–6.90 (m, 2H), 6.79 (d, $J = 8.0$ Hz, 1H), 3.81–3.69 (m, 4H), 1.62 (d, $J = 7.2$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 172.3, 147.9, 141.1, 129.1, 127.9, 127.5, 123.7, 121.2, 119.6, 110.1, 55.8, 48.6, 18.5.



N-(4-methoxyphenyl)-2-phenylpropanamide ^{13}H NMR (400 MHz, CDCl_3) δ 7.41–7.27 (m, 7H), 7.14 (s, 1H), 6.79 (d, $J = 9.0$ Hz, 2H), 3.75 (s, 3H), 3.70 (q, $J = 7.1$ Hz, 1H), 1.59 (d, $J = 7.2$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 172.4, 156.5, 141.2, 131.1, 129.2, 127.8, 127.6, 121.8, 114.1, 55.6, 47.9, 18.7.



N-(4-fluorophenyl)-2-phenylpropanamide ^7H NMR (400 MHz, CDCl_3) δ 7.44–7.20 (m, 8H), 6.99–6.90 (m, 2H), 3.71 (q, $J = 7.1$ Hz, 1H), 1.59 (d, $J = 7.2$ Hz, 3H).

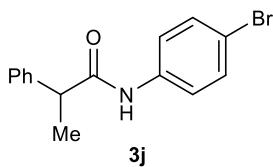
^{13}C NMR (101 MHz, CDCl_3) δ 172.5, 159.4 ($J_{\text{C}-\text{F}} = 244.4$ Hz), 140.9, 133.8 ($J_{\text{C}-\text{F}} = 2.8$ Hz), 129.2, 127.7, 127.6, 121.7 ($J_{\text{C}-\text{F}} = 7.8$ Hz), 115.5 ($J_{\text{C}-\text{F}} = 22.7$ Hz), 47.9, 18.6.

^{19}F NMR (376 MHz, CDCl_3) δ -118.0 – -118.1 (m, 1F).



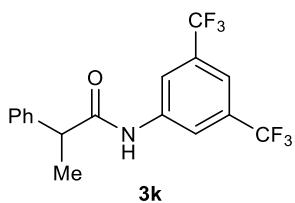
N-(4-chlorophenyl)-2-phenylpropanamide ^{13}H NMR (400 MHz, CDCl_3) δ 7.59 (s, 1H), 7.42–7.28 (m, 7H), 7.19 (d, $J = 8.6$ Hz, 2H), 3.71 (q, $J = 7.0$ Hz, 1H), 1.57 (d, $J = 7.1$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 172.8, 140.8, 136.5, 129.3, 129.2, 128.9, 127.7, 121.3, 47.9, 18.6.



N-(4-bromophenyl)-2-phenylpropanamide¹³ ¹H NMR (400 MHz, CDCl₃) δ 7.37–7.15 (m, 10H), 3.61 (q, J = 7.1 Hz, 1H), 1.48 (d, J = 7.1 Hz, 3H).

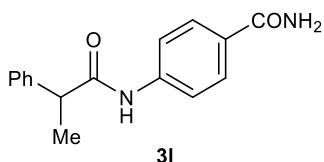
¹³C NMR (101 MHz, CDCl₃) δ 172.6, 140.8, 137.0, 131.9, 129.3, 127.74, 127.72, 121.5, 116.9, 48.1, 18.7.



N-[3,5-Bis(trifluoromethyl)phenyl]-2-phenylpropanamide¹ ¹H NMR (400 MHz, CDCl₃) δ 7.94 (s, 2H), 7.56 (s, 1H), 7.49–7.30 (m, 6H), 3.75 (q, J = 7.1 Hz, 1H), 1.61 (d, J = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 173.0, 140.2, 139.3, 132.4 (q, J_{C-F} = 3.7 Hz), 129.6, 128.2, 127.8, 127.2, 124.5, 121.8, 119.50, 119.47, 119.1, 117.63 (J_{C-F} = 3.7 Hz), 48.3, 18.6.

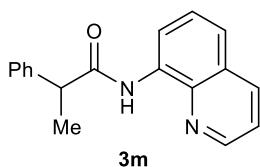
¹⁹F NMR (376 MHz, CDCl₃) δ -63.1 (s, 6F).



N-(4-carbamoylphenyl)-2-phenylpropanamide ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.26 (s, 1H), 7.90–7.78 (m, 3H), 7.65 (d, J = 8.7 Hz, 2H), 7.39 (d, J = 7.3 Hz, 2H), 7.33 (t, J = 7.6 Hz, 2H), 7.28–7.17 (m, 2H), 3.85 (q, J = 6.9 Hz, 1H), 1.42 (d, J = 7.0 Hz, 3H).

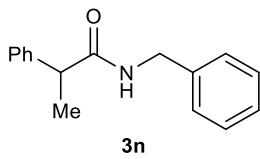
¹³C NMR (101 MHz, DMSO-*d*₆) δ 172.6, 167.3, 141.8, 141.7, 128.7, 128.4, 128.3, 127.3, 126.8, 118.2, 46.0, 18.6.

HRMS (ESI) *m/z* Calcd for C₁₆H₁₇O₂N₂ [M+H]⁺ 269.12845, found 269.12881.



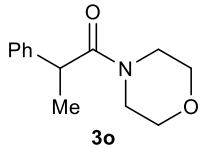
N-(quinolin-8-yl)-2-phenylpropanamide¹⁴ ¹H NMR (400 MHz, CDCl₃) δ 9.90 (s, 1H), 8.78 (d, J = 7.5 Hz, 1H), 8.70 (d, J = 4.2 Hz, 1H), 8.09 (d, J = 8.3 Hz, 1H), 7.54–7.43 (m, 4H), 7.42–7.35 (m, 3H), 7.30 (t, J = 7.3 Hz, 1H), 3.94 (q, J = 7.1 Hz, 1H), 1.70 (d, J = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.8, 148.2, 141.3, 138.6, 136.3, 134.6, 129.1, 128.0, 127.8, 127.44, 127.42, 121.6, 121.5, 116.4, 48.8, 18.7.



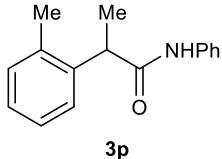
N-benzyl-2-phenylpropanamide¹⁵ ¹H NMR (400 MHz, CDCl₃) δ 7.38–7.20 (m, 8H), 7.14 (d, *J* = 6.6 Hz, 2H), 5.72 (s, 1H), 4.47–4.31 (m, 2H), 3.60 (q, *J* = 7.2 Hz, 1H), 1.56 (d, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 174.2, 141.4, 138.4, 129.1, 128.7, 127.8, 127.6, 127.5, 127.4, 47.3, 43.7, 18.7.



I-morpholino-2-phenylpropan-1-one¹⁶ ¹H NMR (400 MHz, CDCl₃) δ 7.28–7.21 (m, 2H), 7.19–7.11 (m, 3H), 3.82–3.67 (m, 2H), 3.57 (t, *J* = 10.4 Hz, 1H), 3.49–3.36 (m, 3H), 3.35–3.26 (m, 1H), 3.27–3.18 (m, 1H), 3.09–2.97 (m, 1H), 1.38 (d, *J* = 6.9 Hz, 3H).

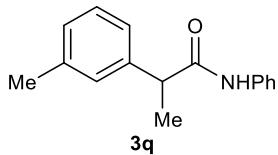
¹³C NMR (101 MHz, CDCl₃) δ 172.2, 141.9, 129.0, 127.2, 126.9, 66.8, 66.3, 46.0, 43.2, 42.4, 20.7.



N-phenyl-2-(2-methylphenyl)propanamide ¹H NMR (400 MHz, CDCl₃) δ 7.37 (t, *J* = 6.2 Hz, 3H), 7.30–7.19 (m, 5H), 7.06 (t, *J* = 7.3 Hz, 1H), 6.95 (s, 1H), 3.93 (q, *J* = 7.2 Hz, 1H), 2.37 (s, 3H), 1.60 (d, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.8, 138.9, 137.9, 136.6, 131.2, 129.0, 127.7, 127.15, 127.11, 124.4, 119.9, 44.8, 19.7, 17.8.

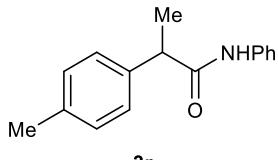
HRMS (ESI) *m/z* Calcd for C₁₆H₁₈ON [M+H]⁺ 240.13829, found 240.13875.



N-phenyl-2-(3-methylphenyl)propanamide ¹H NMR (400 MHz, CDCl₃) δ 7.42 (d, *J* = 7.9 Hz, 2H), 7.30–7.19 (m, 4H), 7.18–7.01 (m, 4H), 3.67 (q, *J* = 7.1 Hz, 1H), 2.35 (s, 3H), 1.57 (d, *J* = 7.1 Hz, 3H).

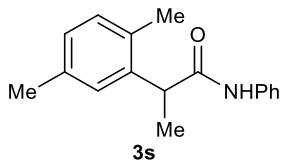
¹³C NMR (101 MHz, CDCl₃) δ 172.6, 141.0, 139.0, 138.0, 129.1, 129.0, 128.5, 128.4, 124.8, 124.3, 119.8, 48.1, 21.6, 18.6.

HRMS (ESI) *m/z* Calcd for C₁₆H₁₈ON [M+H]⁺ 240.13829, found 240.13873.



N-phenyl-2-(4-methylphenyl)propanamide¹³ ¹H NMR (400 MHz, CDCl₃) δ 7.41 (d, *J* = 7.9 Hz, 2H), 7.29–7.21 (m, 4H), 7.17 (d, *J* = 7.9 Hz, 3H), 7.05 (t, *J* = 7.3 Hz, 1H), 3.68 (q, *J* = 7.1 Hz, 1H), 2.35 (s, 3H), 1.57 (d, *J* = 7.1 Hz, 3H).

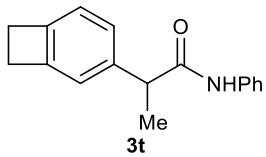
¹³C NMR (101 MHz, CDCl₃) δ 172.7, 138.02, 137.99, 137.4, 129.9, 129.0, 127.7, 124.3, 119.8, 47.8, 21.2, 18.7.



N-phenyl-2-(2,5-dimethylphenyl)propanamide ¹H NMR (400 MHz, CDCl₃) δ 7.38 (d, *J* = 7.9 Hz, 2H), 7.24 (t, *J* = 7.8 Hz, 2H), 7.16 (s, 1H), 7.14–6.98 (m, 4H), 3.89 (q, *J* = 7.1 Hz, 1H), 2.32 (d, *J* = 4.2 Hz, 6H), 1.57 (d, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.9, 138.7, 137.9, 136.5, 133.2, 131.0, 128.9, 128.3, 127.8, 124.2, 119.9, 44.6, 21.2, 19.2, 17.7.

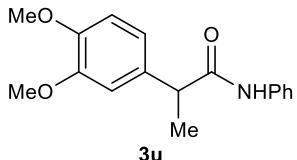
HRMS (ESI) *m/z* Calcd for C₁₇H₂₀ON [M+H]⁺ 254.15394, found 254.15444.



N-phenyl-2-(bicyclo[4.2.0]octa-1,3,5-trien-1-yl)propanamide ¹H NMR (400 MHz, CDCl₃) δ 7.43 (d, *J* = 7.9 Hz, 2H), 7.37 (s, 1H), 7.24 (t, *J* = 7.9 Hz, 2H), 7.16 (d, *J* = 7.5 Hz, 1H), 7.08–7.01 (m, 3H), 3.67 (q, *J* = 7.1 Hz, 1H), 3.15 (s, 4H), 1.56 (d, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.9, 146.7, 145.1, 139.7, 138.1, 128.9, 126.5, 124.2, 123.2, 121.9, 119.8, 48.6, 29.5, 29.4, 18.9.

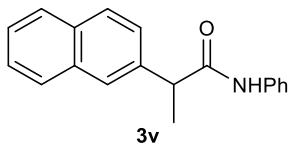
HRMS (ESI) *m/z* Calcd for C₁₇H₁₈ON [M+H]⁺ 252.13829, found 252.13872.



N-phenyl-2-(3,4-dimethoxyphenyl)propanamide ¹H NMR (400 MHz, CDCl₃) δ 7.54–7.39 (m, 3H), 7.25 (t, *J* = 7.9 Hz, 2H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.92–6.79 (m, 3H), 3.84 (d, *J* = 7.0 Hz, 6H), 3.67 (q, *J* = 7.1 Hz, 1H), 1.57 (d, *J* = 7.1 Hz, 3H).

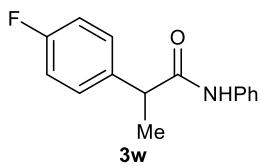
¹³C NMR (101 MHz, CDCl₃) δ 172.9, 149.5, 148.5, 138.1, 133.6, 129.0, 124.3, 119.94, 119.85, 111.6, 110.7, 56.02, 56.00, 47.7, 18.8.

HRMS (ESI) *m/z* Calcd for C₁₇H₂₀O₃N [M+H]⁺ 286.14470, found 286.14429.



N-phenyl-2-(naphthalen-2-yl)propanamide ¹H NMR (400 MHz, CDCl₃) δ 7.94–7.74 (m, 4H), 7.57–7.35 (m, 5H), 7.31–7.12 (m, 3H), 7.05 (t, *J* = 7.2 Hz, 1H), 3.88 (q, *J* = 7.0 Hz, 1H), 1.68 (d, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.3, 138.5, 137.9, 133.7, 132.8, 129.1, 129.0, 127.90, 127.85, 126.6, 126.3, 125.7, 124.4, 119.8, 48.3, 18.7.

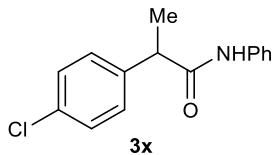


N-phenyl-2-(4-fluorophenyl)propanamide ^1H NMR (400 MHz, CDCl_3) δ 7.43 (d, $J = 7.9$ Hz, 2H), 7.37–7.21 (m, 5H), 7.12–6.98 (m, 3H), 3.68 (q, $J = 7.1$ Hz, 1H), 1.56 (d, $J = 7.1$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 172.4, 162.2 ($J_{\text{C}-\text{F}} = 247.3$ Hz), 137.9, 136.8, 129.3 ($J_{\text{C}-\text{F}} = 8.0$ Hz), 129.1, 124.5, 119.9, 116.0 ($J_{\text{C}-\text{F}} = 21.4$ Hz), 47.4, 18.9.

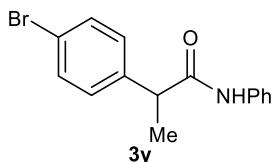
^{19}F NMR (376 MHz, CDCl_3) δ -114.8 – -114.9 (m, 1F).

HRMS (ESI) m/z Calcd for $\text{C}_{15}\text{H}_{15}\text{ONF}$ [$\text{M}+\text{H}]^+$ 244.11322, found 244.11372.



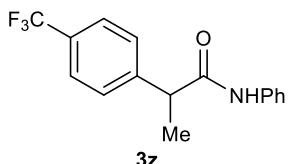
N-phenyl-2-(4-chlorophenyl)propanamide ^{13}H NMR (400 MHz, CDCl_3) δ 7.42 (d, $J = 8.0$ Hz, 2H), 7.37–7.18 (m, 7H), 7.08 (t, $J = 7.4$ Hz, 1H), 3.67 (q, $J = 7.1$ Hz, 1H), 1.55 (d, $J = 7.1$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 172.0, 139.5, 137.8, 133.5, 129.3, 129.11, 129.08, 124.6, 120.0, 47.5, 18.9.



N-phenyl-2-(4-bromophenyl)propanamide ^{13}H NMR (400 MHz, CDCl_3) δ 7.45 (d, $J = 7.5$ Hz, 4H), 7.35–7.13 (m, 5H), 7.08 (t, $J = 6.6$ Hz, 1H), 3.65 (d, $J = 6.8$ Hz, 1H), 1.56 (d, $J = 6.7$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 171.8, 140.0, 137.8, 132.3, 129.5, 129.1, 124.6, 121.6, 119.9, 47.7, 18.8.

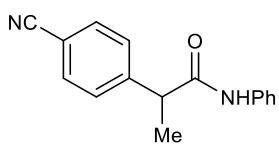


N-phenyl-2-(4-trifluoromethylphenyl)propanamide ^1H NMR (400 MHz, CDCl_3) δ 7.61 (d, $J = 8.1$ Hz, 2H), 7.46 (d, $J = 8.0$ Hz, 4H), 7.33–7.17 (m, 3H), 7.09 (t, $J = 7.4$ Hz, 1H), 3.75 (q, $J = 7.0$ Hz, 1H), 1.59 (d, $J = 7.1$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 171.4, 145.0 ($J_{\text{C}-\text{F}} = 0.9$ Hz), 137.7, 129.9 ($J_{\text{C}-\text{F}} = 32.7$ Hz), 129.1, 128.1, 126.1 (q, $J_{\text{C}-\text{F}} = 3.7$ Hz), 124.7, 124.1 (d, $J_{\text{C}-\text{F}} = 273.1$ Hz), 120.0, 48.1, 19.0.

^{19}F NMR (376 MHz, CDCl_3) δ -62.6 (s, 3F).

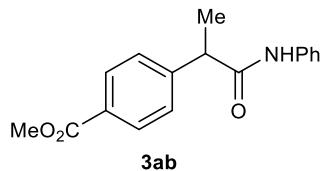
HRMS (ESI) m/z Calcd for $\text{C}_{16}\text{H}_{15}\text{ONF}_3$ [$\text{M}+\text{H}]^+$ 294.11003, found 294.11057.



N-phenyl-2-(4-cyanophenyl)propanamide ^1H NMR (400 MHz, CDCl_3) δ 7.82 (s, 1H), 7.57 (d, $J = 8.3$ Hz, 2H), 7.51–7.40 (m, 4H), 7.26 (t, $J = 7.9$ Hz, 2H), 7.09 (t, $J = 7.4$ Hz, 1H), 3.74 (q, $J = 7.0$ Hz, 1H), 1.55 (d, $J = 7.0$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 171.3, 146.6, 137.7, 132.6, 129.0, 128.5, 124.8, 120.2, 118.8, 111.1, 47.9, 18.9.

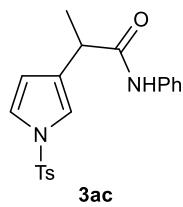
HRMS (ESI) m/z Calcd for $\text{C}_{16}\text{H}_{15}\text{ON}_2$ [$\text{M}+\text{H}]^+$ 251.11789, found 251.11839.



N-phenyl-2-(4-methoxycarbonylphenyl)propanamide ^1H NMR (400 MHz, CDCl_3) δ 8.01 (d, $J = 8.1$ Hz, 2H), 7.43 (d, $J = 7.8$ Hz, 4H), 7.36 (s, 1H), 7.26 (t, $J = 7.8$ Hz, 2H), 7.07 (t, $J = 7.3$ Hz, 1H), 3.91 (s, 3H), 3.76 (q, $J = 7.0$ Hz, 1H), 1.58 (d, $J = 7.0$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 171.6, 166.9, 146.3, 137.8, 130.4, 129.4, 129.1, 127.8, 124.6, 120.0, 52.3, 48.1, 18.8.

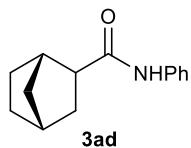
HRMS (ESI) m/z Calcd for $\text{C}_{17}\text{H}_{18}\text{O}_3\text{N}$ [$\text{M}+\text{H}]^+$ 284.12812, found 284.12860.



N-phenyl-2-(1-tosyl-1*H*-pyrrol-3-yl)propanamide ^1H NMR (400 MHz, CDCl_3) δ 7.76 (d, $J = 8.3$ Hz, 2H), 7.35 (d, $J = 7.7$ Hz, 2H), 7.31 (s, 1H), 7.30–7.23 (m, 3H), 7.21–7.16 (m, 2H), 7.13 (s, 1H), 7.07 (t, $J = 7.3$ Hz, 1H), 6.32–6.27 (m, 1H), 3.59 (q, $J = 7.2$ Hz, 1H), 2.41 (s, 3H), 1.50 (d, $J = 7.2$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 172.0, 145.4, 137.8, 135.9, 130.3, 129.0, 128.7, 127.0, 124.5, 122.1, 119.8, 118.1, 113.3, 40.8, 21.8, 18.2.

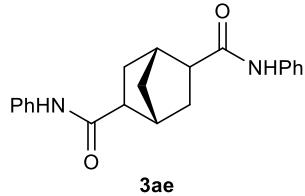
HRMS (ESI) m/z Calcd for $\text{C}_{20}\text{H}_{21}\text{O}_3\text{N}_2\text{S}$ [$\text{M}+\text{H}]^+$ 369.12674, found 369.12720.



N-phenylbicyclo[2.2.1]heptane-2-carboxamide ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J = 7.9$ Hz, 2H), 7.42 (s, 1H), 7.29 (t, $J = 7.9$ Hz, 2H), 7.07 (t, $J = 7.4$ Hz, 1H), 2.50 (s, 1H), 2.37–2.22 (m, 2H), 2.02–1.93 (m, 1H), 1.70–1.43 (m, 4H), 1.30–1.13 (m, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 174.3, 138.4, 129.0, 124.0, 119.8, 49.1, 41.8, 36.6, 36.1, 34.3, 29.9, 28.8.

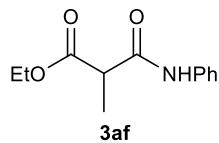
HRMS (ESI) m/z Calcd for $\text{C}_{14}\text{H}_{18}\text{ON}$ [$\text{M}+\text{H}]^+$ 216.13829, found 216.13887.



*N*2,*N*5-diphenylbicyclo[2.2.1]heptane-2,5-dicarboxamide ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.82 (s, 2H), 7.62–7.53 (m, 4H), 7.24 (t, *J* = 7.7 Hz, 4H), 6.97 (t, *J* = 7.3 Hz, 2H), 2.50–2.36 (m, 4H), 1.96–1.87 (m, 1H), 1.86–1.73 (m, 1H), 1.51–1.36 (m, 4H).

^{13}C NMR (101 MHz, DMSO) δ 173.5, 173.2, 139.6, 139.5, 128.7, 128.6, 122.9, 122.8, 119.1, 119.0, 47.3, 46.8, 41.5, 34.4, 33.6, 33.2, 33.1.

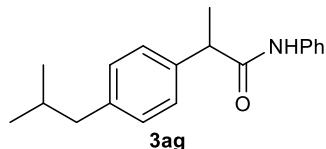
HRMS (ESI) *m/z* Calcd for C₂₁H₂₃O₂N₂ [M+H]⁺ 335.17540, found 335.17587.



ethyl 2-methyl-3-(phenylamino)-3-oxopropanoate ^1H NMR (400 MHz, DMSO-*d*₆) δ 10.18 (s, 1H), 7.58 (d, *J* = 7.3 Hz, 2H), 7.31 (t, *J* = 6.5 Hz, 2H), 7.07 (d, *J* = 6.8 Hz, 1H), 4.10 (s, 2H), 3.69–3.50 (m, 1H), 1.35–1.14 (m, 6H).

^{13}C NMR (101 MHz, DMSO-*d*₆) δ 170.3, 168.1, 138.9, 128.7, 123.5, 119.2, 60.6, 46.8, 14.0, 13.9.

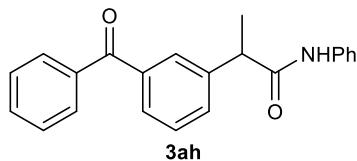
HRMS (ESI) *m/z* Calcd for C₁₂H₁₆O₃N [M+H]⁺ 222.11247, found 222.11282.



N-phenyl-2-[4-(2-methylpropyl)phenyl]propanamide ^1H NMR (400 MHz, CDCl₃) δ 7.41 (d, *J* = 7.9 Hz, 2H), 7.31–7.23 (m, 4H), 7.15 (d, *J* = 7.9 Hz, 2H), 7.12–7.02 (m, 2H), 3.69 (q, *J* = 7.1 Hz, 1H), 2.47 (d, *J* = 7.2 Hz, 2H), 1.87 (dp, *J* = 13.5, 6.7 Hz, 1H), 1.59 (d, *J* = 7.1 Hz, 3H), 0.91 (d, *J* = 6.6 Hz, 6H).

^{13}C NMR (101 MHz, CDCl₃) δ 172.7, 141.2, 138.2, 138.0, 130.0, 129.0, 127.6, 124.3, 119.8, 47.9, 45.1, 30.3, 22.5, 18.6.

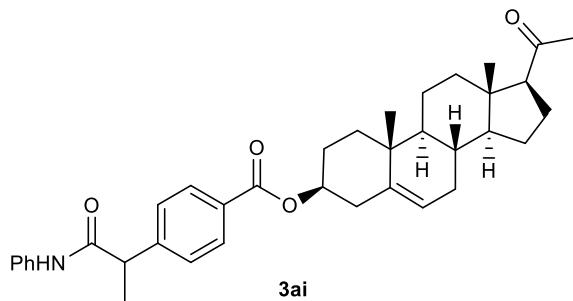
HRMS (ESI) *m/z* Calcd for C₁₉H₂₄ON [M+H]⁺ 282.18524, found 282.18570.



N-phenyl-2-(3-benzoylphenyl)propanamide ^1H NMR (400 MHz, CDCl₃) δ 7.87–7.72 (m, 3H), 7.63 (t, *J* = 8.0 Hz, 3H), 7.54–7.38 (m, 6H), 7.26 (d, *J* = 6.3 Hz, 4H), 7.07 (t, *J* = 7.3 Hz, 1H), 3.77 (q, *J* = 7.0 Hz, 1H), 1.59 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, CDCl₃) δ 196.8, 171.9, 141.7, 138.2, 137.9, 137.4, 132.8, 131.6, 130.2, 129.5, 129.3, 129.1, 128.5, 124.5, 120.0, 48.0, 18.9.

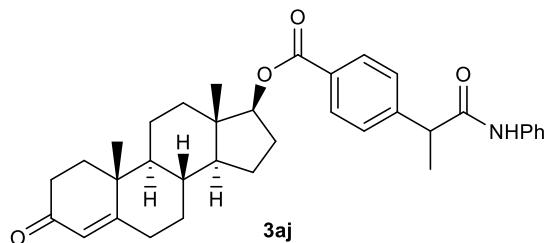
HRMS (ESI) *m/z* Calcd for C₂₂H₂₀O₂N [M+H]⁺ 330.14886, found 330.14920.



N-phenyl-2-(4-(3-*O*-pregnenolnoyl)carbonylphenyl)propanamide ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 8.2$ Hz, 2H), 7.62 (s, 1H), 7.43 (t, $J = 6.9$ Hz, 4H), 7.28–7.22 (m, 2H), 7.05 (t, $J = 7.3$ Hz, 1H), 5.39 (s, 1H), 4.91–4.79 (m, 1H), 3.76 (q, $J = 6.9$ Hz, 1H), 2.54 (t, $J = 8.8$ Hz, 1H), 2.45 (d, $J = 7.5$ Hz, 2H), 2.23–2.10 (m, 4H), 2.08–1.86 (m, 4H), 1.80–1.39 (m, 11H), 1.28–1.14 (m, 3H), 1.08–0.99 (m, 4H), 0.63 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 209.7, 171.5, 165.7, 146.0, 139.6, 137.8, 130.3, 130.0, 128.9, 127.6, 124.4, 122.5, 119.8, 74.6, 63.7, 56.8, 49.9, 48.0, 44.0, 38.8, 38.2, 37.0, 36.7, 31.84, 31.80, 31.6, 27.9, 24.5, 22.9, 21.1, 19.4, 18.7, 13.3.

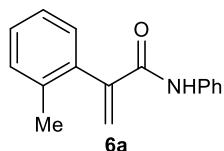
HRMS (ESI) m/z Calcd for $\text{C}_{37}\text{H}_{46}\text{O}_4\text{N} [\text{M}+\text{H}]^+$ 568.34214, found 568.34332.



N-phenyl-2-(4-(17-*O*-testosteronyl)carbonylphenyl)propanamide ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, $J = 8.3$ Hz, 2H), 7.44 (d, $J = 8.1$ Hz, 4H), 7.36–7.23 (m, 3H), 7.07 (t, $J = 7.3$ Hz, 1H), 5.73 (s, 1H), 4.88–4.82 (m, 1H), 3.77 (q, $J = 7.1$ Hz, 1H), 2.53–2.21 (m, 5H), 2.05–1.97 (m, 1H), 1.91–1.83 (m, 2H), 1.78–1.53 (m, 8H), 1.48–1.36 (m, 2H), 1.30–0.92 (m, 10H).

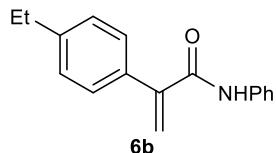
^{13}C NMR (101 MHz, CDCl_3) δ 199.7, 171.5, 171.2, 166.2, 146.3, 137.9, 130.4, 129.9, 129.0, 127.8, 124.5, 124.0, 119.9, 83.2, 53.8, 50.4, 48.2, 43.0, 38.8, 36.8, 35.8, 35.5, 34.0, 32.9, 31.6, 27.8, 23.7, 20.7, 18.9, 17.5, 12.4.

HRMS (ESI) m/z Calcd for $\text{C}_{35}\text{H}_{42}\text{O}_4\text{N} [\text{M}+\text{Na}]^+$ 540.31084, found 540.31186.



N-phenyl-2-(2-methylphenyl)acrylamide¹⁷ ^1H NMR (400 MHz, CDCl_3) δ 7.43 (d, $J = 7.9$ Hz, 2H), 7.39–7.24 (m, 6H), 7.20–7.04 (m, 2H), 6.61 (s, 1H), 5.58 (s, 1H), 2.29 (s, 3H).

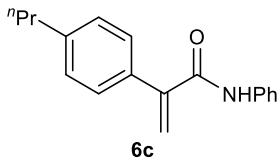
^{13}C NMR (101 MHz, CDCl_3) δ 164.2, 144.1, 137.7, 136.9, 136.6, 130.8, 130.1, 129.2, 129.0, 126.6, 126.3, 124.7, 120.1, 19.9.



N-phenyl-2-(4-ethylphenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J = 8.0$ Hz, 2H), 7.43 (s, 1H), 7.37 (d, $J = 8.0$ Hz, 2H), 7.32 (t, $J = 7.9$ Hz, 2H), 7.29–7.23 (m, 2H), 7.12 (t, $J = 7.4$ Hz, 1H), 6.26 (s, 1H), 5.70 (s, 1H), 2.70 (q, $J = 7.6$ Hz, 2H), 1.28 (t, $J = 7.6$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.5, 145.3, 145.1, 137.8, 134.1, 129.1, 128.6, 128.4, 124.7, 123.0, 120.1, 28.7, 15.6.

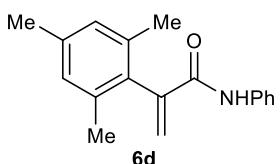
HRMS (ESI) m/z Calcd for $\text{C}_{17}\text{H}_{18}\text{ON} [\text{M}+\text{H}]^+$ 252.13829, found 252.13885.



N-phenyl-2-(4-propylphenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J = 7.9$ Hz, 2H), 7.43 (s, 1H), 7.39–7.29 (m, 4H), 7.24 (d, $J = 8.0$ Hz, 2H), 7.12 (t, $J = 7.4$ Hz, 1H), 6.25 (s, 1H), 5.70 (s, 1H), 2.63 (t, $J = 7.6$ Hz, 2H), 1.74–1.64 (m, 2H), 0.98 (t, $J = 7.3$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.5, 145.1, 143.8, 137.8, 134.1, 129.17, 129.11, 128.3, 124.7, 122.9, 120.0, 37.9, 24.6, 14.0.

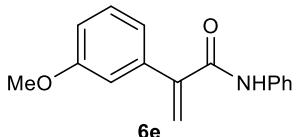
HRMS (ESI) m/z Calcd for $\text{C}_{18}\text{H}_{20}\text{ON} [\text{M}+\text{H}]^+$ 266.15394, found 266.15445.



N-phenyl-2-(2,4,6-trimethylphenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.42 (d, $J = 7.8$ Hz, 2H), 7.27 (t, $J = 7.9$ Hz, 2H), 7.14 (s, 1H), 7.08 (t, $J = 7.4$ Hz, 1H), 6.97 (s, 2H), 6.79 (s, 1H), 5.51 (s, 1H), 2.34 (s, 3H), 2.21 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 163.8, 141.8, 138.5, 137.7, 137.0, 132.8, 129.0, 127.2, 124.6, 120.2, 21.2, 20.4.

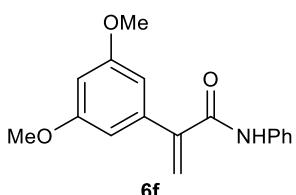
HRMS (ESI) m/z Calcd for $\text{C}_{18}\text{H}_{20}\text{ON} [\text{M}+\text{H}]^+$ 266.15394, found 266.15439.



N-phenyl-2-(3-methoxyphenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J = 7.9$ Hz, 2H), 7.45 (s, 1H), 7.38–7.29 (m, 3H), 7.12 (t, $J = 7.4$ Hz, 1H), 7.02 (d, $J = 7.6$ Hz, 1H), 7.00–6.93 (m, 2H), 6.31 (s, 1H), 5.72 (d, $J = 1.2$ Hz, 1H), 3.84 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.0, 160.0, 145.0, 138.2, 137.8, 130.2, 129.1, 124.7, 123.9, 120.7, 120.1, 114.5, 114.1, 55.5.

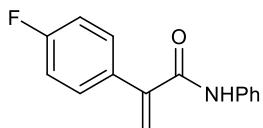
HRMS (ESI) m/z Calcd for $\text{C}_{16}\text{H}_{16}\text{O}_2\text{N} [\text{M}+\text{H}]^+$ 254.11756, found 254.11797.



N-phenyl-2-(3,5-dimethoxyphenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J = 7.8$ Hz, 2H), 7.46 (s, 1H), 7.32 (t, $J = 7.9$ Hz, 2H), 7.12 (t, $J = 7.4$ Hz, 1H), 6.57 (d, $J = 2.2$ Hz, 2H), 6.51 (t, $J = 2.2$ Hz, 1H), 6.34 (s, 1H), 5.71 (s, 1H), 3.82 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 164.7, 161.3, 144.9, 138.9, 137.8, 129.1, 124.7, 124.3, 120.1, 106.6, 100.9, 55.6.

HRMS (ESI) m/z Calcd for $\text{C}_{17}\text{H}_{16}\text{O}_3\text{N} [\text{M}-\text{H}]^-$ 282.11357, found 282.11392.

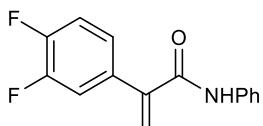


6g

N-phenyl-2-(4-fluorophenyl)acrylamide ^{18}H NMR (400 MHz, CDCl_3) δ 7.53 (d, $J = 7.9$ Hz, 2H), 7.48–7.41 (m, 2H), 7.41–7.29 (m, 3H), 7.18–7.08 (m, 3H), 6.21 (s, 1H), 5.72 (s, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.2, 163.1 ($J_{\text{C}-\text{F}} = 249.9$ Hz), 144.3, 137.5, 132.6 ($J_{\text{C}-\text{F}} = 3.5$ Hz), 130.0 ($J_{\text{C}-\text{F}} = 8.3$ Hz), 129.1, 124.7, 122.7, 119.9, 115.9 ($J_{\text{C}-\text{F}} = 21.7$ Hz).

^{19}F NMR (376 MHz, CDCl_3) δ -112.3 – -112.4 (m, 1F).



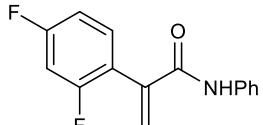
6h

N-phenyl-2-(3,4-difluorophenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.54 (d, $J = 7.9$ Hz, 2H), 7.45 (s, 1H), 7.38–7.28 (m, 3H), 7.23–7.12 (m, 3H), 6.14 (s, 1H), 5.75 (s, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.1, 150.7 (dd, $J_{\text{C}-\text{F}} = 251.0, 39.1$ Hz), 150.6 (dd, $J_{\text{C}-\text{F}} = 250.9, 37.8$ Hz), 143.8, 137.5, 133.5 (dd, $J_{\text{C}-\text{F}} = 10.1, 1.7$ Hz), 129.2, 125.0, 124.4 (dd, $J_{\text{C}-\text{F}} = 9.9, 2.6$ Hz), 122.6, 120.2, 117.6 (dd, $J_{\text{C}-\text{F}} = 48.7, 48.1$ Hz).

^{19}F NMR (376 MHz, CDCl_3) δ -136.3 – -137.0 (m, 2F).

HRMS (ESI) m/z Calcd for $\text{C}_{15}\text{H}_{10}\text{ONF}_2$ [M-H] $^+$ 258.07359, found 258.07407.



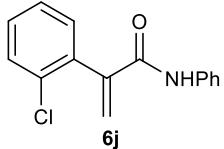
6i

N-phenyl-2-(2,4-difluorophenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.51 (d, $J = 7.9$ Hz, 2H), 7.43–7.29 (m, 4H), 7.13 (t, $J = 7.4$ Hz, 1H), 7.00–6.86 (m, 2H), 6.37 (s, 1H), 5.77 (s, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 164.6, 163.5 (dd, $J_{\text{C}-\text{F}} = 252.7, 12.1$ Hz), 160.3 (dd, $J_{\text{C}-\text{F}} = 253.6, 12.7$ Hz), 139.3, 137.6, 132.1 (dd, $J_{\text{C}-\text{F}} = 9.7, 9.6$ Hz), 129.2, 125.7, 124.9, 120.9 (dd, $J_{\text{C}-\text{F}} = 18.9, 10.9$ Hz), 120.3, 112.1 (dd, $J_{\text{C}-\text{F}} = 21.3, 3.8$ Hz), 105.05 (dd, $J_{\text{C}-\text{F}} = 25.7$ Hz).

^{19}F NMR (376 MHz, CDCl_3) δ -108.0—-108.3 (m, 2F).

HRMS (ESI) m/z Calcd for $\text{C}_{15}\text{H}_{12}\text{ONF}_2$ [M+H] $^+$ 260.08815, found 260.08893.

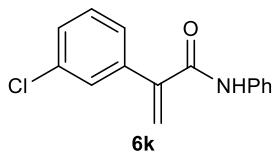


6j

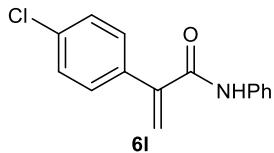
N-phenyl-2-(2-chlorophenyl)acrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.48 (d, $J = 8.0$ Hz, 3H), 7.42–7.27 (m, 5H), 7.20 (s, 1H), 7.11 (t, $J = 7.4$ Hz, 1H), 6.54 (s, 1H), 5.68 (s, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 163.8, 143.1, 137.7, 136.1, 133.7, 131.7, 130.5, 130.1, 129.1, 127.6, 126.8, 124.8, 120.4.

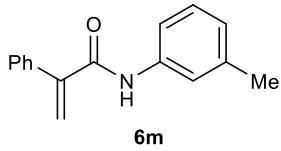
HRMS (ESI) m/z Calcd for $\text{C}_{15}\text{H}_{13}\text{ONCl}$ [M+H] $^+$ 258.06802, found 258.06854.



*N-phenyl-2-(3-chlorophenyl)acrylamide*¹⁹ ¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, *J* = 7.8 Hz, 2H), 7.46 (s, 2H), 7.40–7.29 (m, 5H), 7.14 (t, *J* = 7.3 Hz, 1H), 6.21 (s, 1H), 5.75 (s, 1H).
¹³C NMR (101 MHz, CDCl₃) δ 165.1, 144.3, 138.4, 137.6, 135.0, 130.2, 129.2, 129.1, 128.4, 126.4, 124.9, 123.4, 120.2.

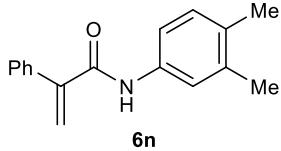


*N-phenyl-2-(4-chlorophenyl)acrylamide*²⁰ ¹H NMR (400 MHz, CDCl₃) δ 7.52–7.46 (m, 3H), 7.39 (s, 4H), 7.32 (t, *J* = 7.8 Hz, 2H), 7.13 (t, *J* = 7.4 Hz, 1H), 6.18 (s, 1H), 5.73 (s, 1H).
¹³C NMR (101 MHz, CDCl₃) δ 165.3, 144.4, 137.6, 135.05, 135.01, 129.5, 129.21, 129.16, 124.9, 122.8, 120.1.

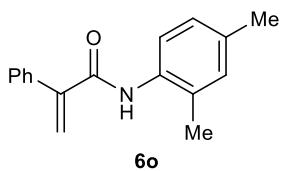


N-(3-methylphenyl)-2-phenylacrylamide ¹H NMR (400 MHz, CDCl₃) δ 7.48–7.41 (m, 5H), 7.40–7.27 (m, 3H), 7.20 (t, *J* = 7.8 Hz, 1H), 6.94 (d, *J* = 7.5 Hz, 1H), 6.29 (s, 1H), 5.72 (s, 1H), 2.33 (s, 3H).
¹³C NMR (101 MHz, CDCl₃) δ 165.3, 145.3, 139.1, 137.7, 136.8, 129.1, 128.96, 128.95, 128.4, 125.5, 123.4, 120.7, 117.1, 21.6.

HRMS (ESI) *m/z* Calcd for C₁₆H₁₆ON [M+H]⁺ 238.12264, found 238.12315.



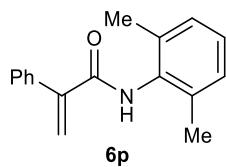
*N-(3,4-dimethylphenyl)-2-phenylacrylamide*²¹ ¹H NMR (400 MHz, CDCl₃) δ 7.46–7.37 (m, 5H), 7.34 (s, 1H), 7.29 (s, 1H), 7.27–7.22 (m, 1H), 7.05 (d, *J* = 8.1 Hz, 1H), 6.25 (s, 1H), 5.69 (s, 1H), 2.22 (s, 6H).
¹³C NMR (101 MHz, CDCl₃) δ 165.1, 145.2, 137.3, 136.8, 135.4, 133.0, 120.0, 128.9, 128.8, 128.3, 123.1, 121.3, 117.5, 19.9, 19.2.



N-(2,4-dimethylphenyl)-2-phenylacrylamide ¹H NMR (400 MHz, CDCl₃) δ 7.86 (d, *J* = 8.2 Hz, 1H), 7.52–7.37 (m, 5H), 7.20 (s, 1H), 7.03 (d, *J* = 8.1 Hz, 1H), 6.96 (s, 1H), 6.36 (s, 1H), 5.71 (s, 1H), 2.28 (s, 3H), 2.00 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 164.9, 145.1, 137.2, 134.9, 133.2, 131.2, 129.0, 128.9, 128.66, 128.59, 127.5, 123.7, 122.5, 21.0, 17.6.

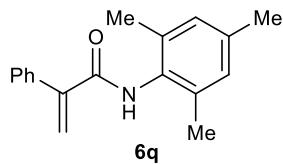
HRMS (ESI) *m/z* Calcd for C₁₇H₁₈ON [M+H]⁺ 252.13829, found 252.13878.



N-(2,6-dimethylphenyl)-2-phenylacrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.51 (d, $J = 6.7$ Hz, 2H), 7.47–7.36 (m, 3H), 7.12–7.04 (m, 3H), 6.91 (s, 1H), 6.30 (s, 1H), 5.72 (s, 1H), 2.23 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.4, 144.8, 137.1, 135.4, 133.7, 128.9, 128.8, 128.27, 128.24, 127.5, 123.0, 18.6.

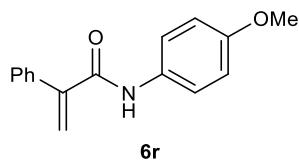
HRMS (ESI) m/z Calcd for $\text{C}_{17}\text{H}_{18}\text{ON} [\text{M}+\text{H}]^+$ 252.13829, found 252.13875.



N-(2,4,6-trimethylphenyl)-2-phenylacrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.55–7.49 (m, 2H), 7.47–7.37 (m, 3H), 6.89 (s, 2H), 6.85 (s, 1H), 6.29 (s, 1H), 5.71 (s, 1H), 2.27 (s, 3H), 2.20 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.8, 144.9, 137.30, 137.23, 135.2, 131.1, 129.1, 129.0, 128.8, 128.4, 122.9, 21.1, 18.6.

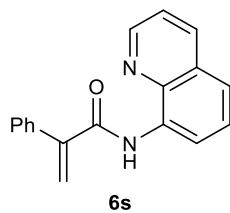
HRMS (ESI) m/z Calcd for $\text{C}_{18}\text{H}_{20}\text{ON} [\text{M}+\text{H}]^+$ 266.15394, found 266.15442.



N-(4-methoxyphenyl)-2-phenylacrylamide ^1H NMR (400 MHz, CDCl_3) δ 7.53–7.29 (m, 8H), 6.85 (d, $J = 9.0$ Hz, 2H), 6.26 (s, 1H), 5.70 (s, 1H), 3.78 (s, 3H).

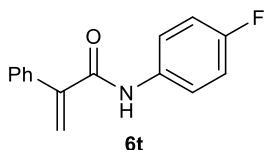
^{13}C NMR (101 MHz, CDCl_3) δ 165.2, 156.7, 145.2, 136.9, 130.9, 129.0, 128.9, 128.4, 123.1, 121.8, 114.2, 55.6.

HRMS (ESI) m/z Calcd for $\text{C}_{16}\text{H}_{16}\text{O}_2\text{N} [\text{M}+\text{H}]^+$ 254.11756, found 254.11800.

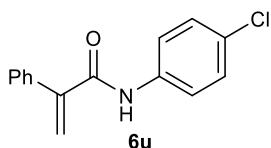


N-(quinolin-8-yl)-2-phenylacrylamide²² ^1H NMR (400 MHz, CDCl_3) δ 10.26 (s, 1H), 8.96–8.85 (m, 1H), 8.64 (d, $J = 4.2$, 1H), 8.13 (d, $J = 8.3$, 1H), 7.60–7.49 (m, 4H), 7.49–7.42 (m, 3H), 7.42–7.38 (m, 1H), 6.33 (s, 1H), 5.83 (s, 1H).

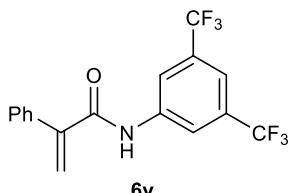
^{13}C NMR (101 MHz, CDCl_3) δ 165.9, 148.4, 146.0, 138.8, 136.9, 136.3, 134.6, 128.8, 128.7, 128.5, 128.0, 127.5, 122.4, 122.0, 121.7, 116.8.



N-(4-fluorophenyl)-2-phenylacrylamide²³ ¹H NMR (400 MHz, CDCl₃) δ 7.55–7.35 (m, 8H), 7.00 (t, *J* = 8.6 Hz, 2H), 6.28 (s, 1H), 5.72 (s, 1H).
¹³C NMR (101 MHz, CDCl₃) δ 165.3, 159.6 (*J*_{C-F} = 244.9 Hz), 145.0, 136.7, 133.8 (*J*_{C-F} = 2.9 Hz), 129.1, 129.0, 128.4, 123.6, 121.9 (*J*_{C-F} = 7.9 Hz), 115.9, 115.7.
¹⁹F NMR (376 MHz, CDCl₃) δ -117.5 – -117.6 (m, 1F).



N-(4-chlorophenyl)-2-phenylacrylamide²¹ ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.42 (s, 1H), 7.76 (d, *J* = 8.8 Hz, 2H), 7.49 (d, *J* = 7.1 Hz, 2H), 7.45–7.31 (m, 5H), 5.97 (s, 1H), 5.76 (s, 1H).
¹³C NMR (101 MHz, DMSO-*d*₆) δ 167.4, 145.1, 138.0, 136.2, 128.6, 128.5, 128.3, 127.2, 126.9, 121.4, 118.0.

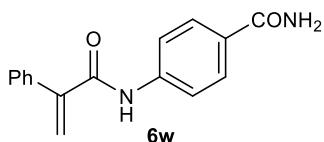


N-[3,5-bis(trifluoromethyl)phenyl]-2-phenylacrylamide ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.86 (s, 1H), 8.45 (s, 2H), 7.79 (s, 1H), 7.51 (d, *J* = 6.9 Hz, 2H), 7.40 (q, *J* = 7.9, 7.0 Hz, 3H), 6.04 (s, 1H), 5.90 (s, 1H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.0, 144.6, 141.0, 135.9, 130.7 (*q*, *J*_{C-F} = 32.9 Hz), 128.49, 128.45, 127.31, 127.1, 124.6, 121.9, 119.63, 119.56, 119.2, 116.4 (*J*_{C-F} = 3.6 Hz).

¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -61.73 (s, 6F).

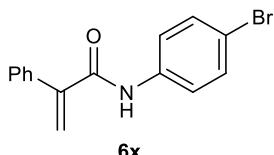
HRMS (ESI) *m/z* Calcd for C₁₇H₁₂ONF₆ [M+H]⁺ 360.08176, found 360.08224.



N-(4-carbamoylphenyl)-2-phenylacrylamide ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.49 (s, 1H), 7.99–7.73 (m, 5H), 7.57–7.26 (m, 6H), 5.98 (s, 1H), 5.77 (s, 1H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 167.6, 167.4, 145.1, 141.7, 136.2, 128.5, 128.4, 128.3, 126.9, 119.0, 118.2.

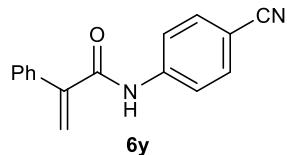
HRMS (ESI) *m/z* Calcd for C₁₆H₁₅O₂N₂ [M+H]⁺ 267.11280, found 267.11340.



N-(4-bromophenyl)-2-phenylacrylamide ^1H NMR (400 MHz, DMSO-*d*₆) δ 10.40 (s, 1H), 7.71 (d, *J* = 8.8 Hz, 2H), 7.55–7.45 (m, 4H), 7.43–7.33 (m, 3H), 5.96 (s, 1H), 5.75 (s, 1H).

^{13}C NMR (101 MHz, DMSO-*d*₆) δ 167.4, 145.0, 138.4, 136.1, 131.4, 128.5, 128.3, 126.8, 121.8, 118.0, 115.3.

HRMS (ESI) *m/z* Calcd for C₁₅H₁₃ONBr [M+H]⁺ 302.01750, found 302.01788.



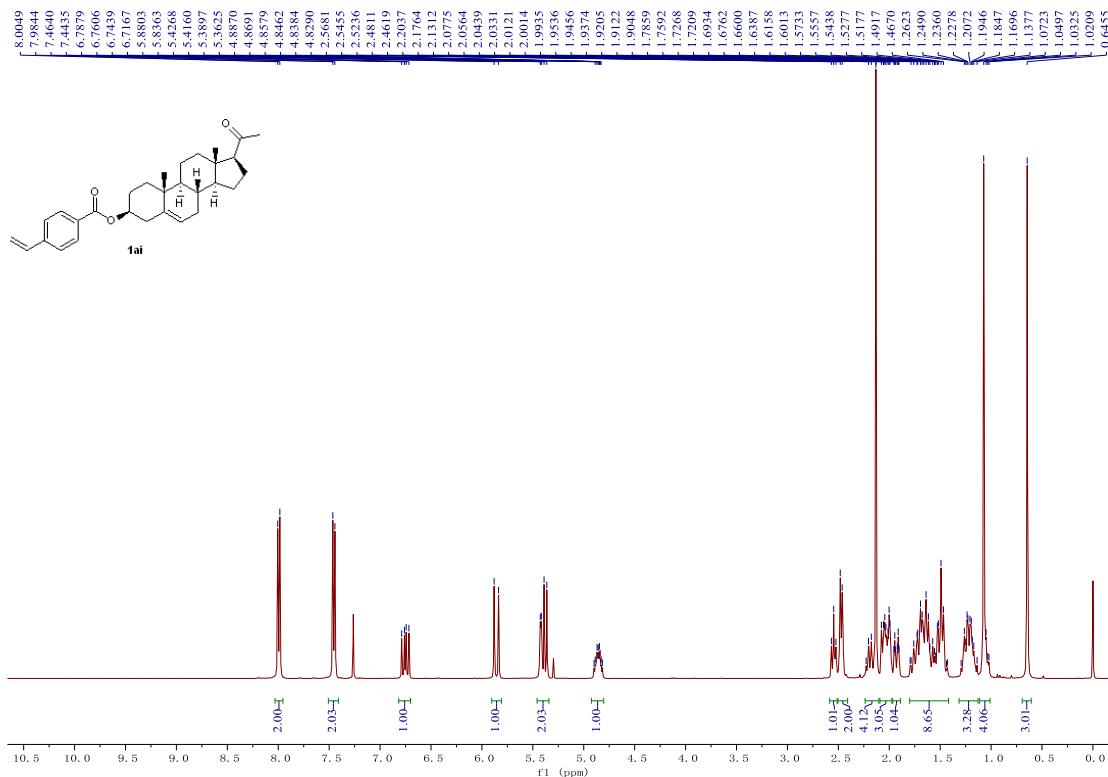
N-(4-cyanophenyl)-2-phenylacrylamide ^1H NMR (400 MHz, DMSO-*d*₆) δ 10.70 (s, 1H), 7.93 (d, *J* = 8.7 Hz, 2H), 7.80 (d, *J* = 8.7 Hz, 2H), 7.49 (d, *J* = 6.9 Hz, 2H), 7.43–7.33 (m, 3H), 6.02 (s, 1H), 5.81 (s, 1H).

^{13}C NMR (101 MHz, DMSO) δ 168.4, 145.3, 143.8, 136.4, 133.6, 129.0, 128.9, 127.3, 120.3, 119.5, 119.2, 105.9.

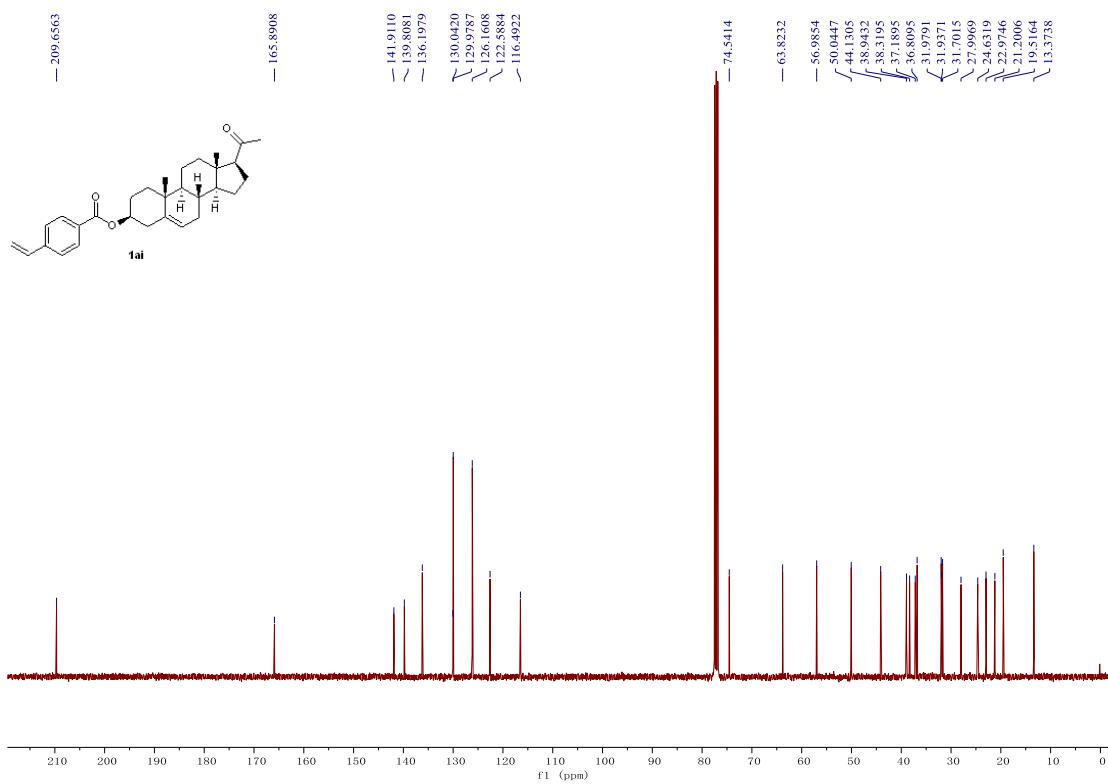
HRMS (ESI) *m/z* Calcd for C₁₆H₁₃ON₂ [M+H]⁺ 249.10224, found 249.10277.

11. NMR Spectra

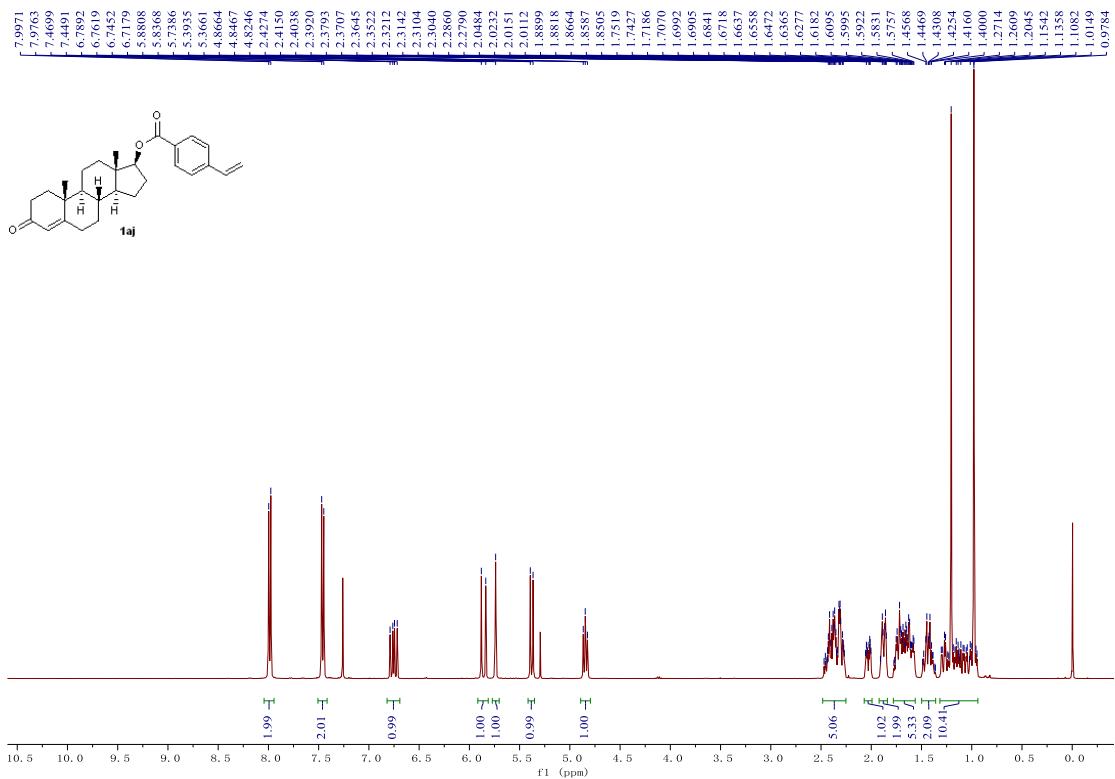
^1H NMR spectrum of **1ai**



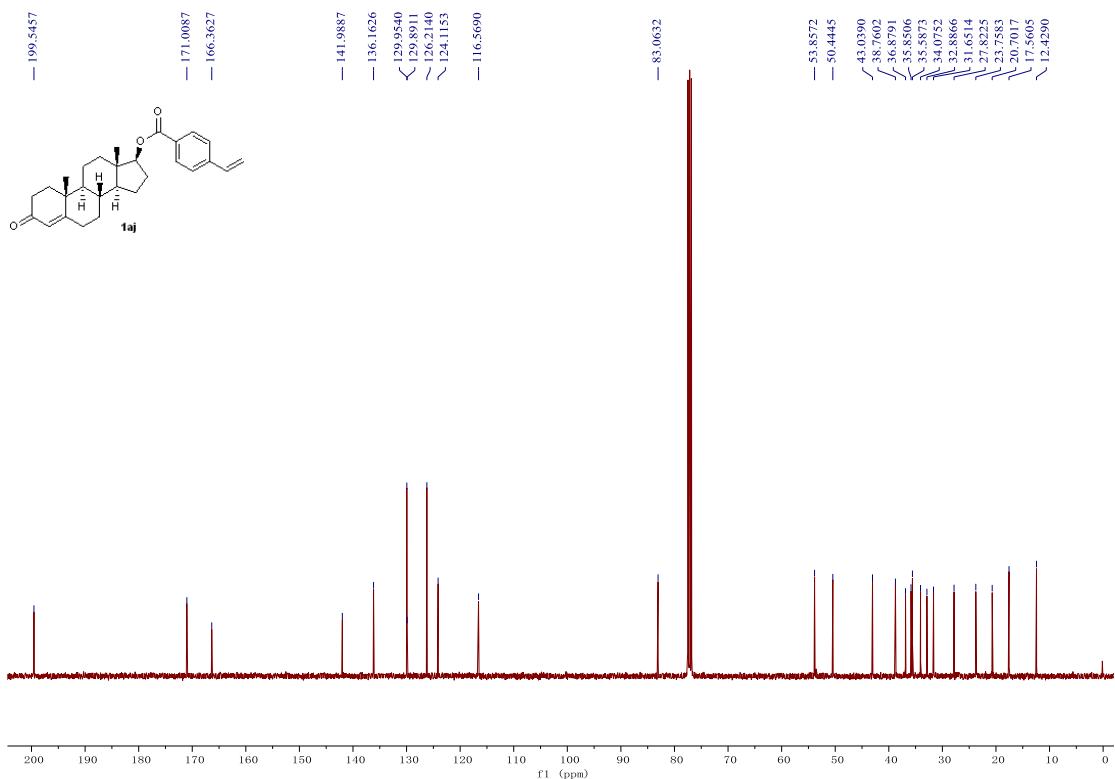
¹³C NMR spectrum of **1ai**



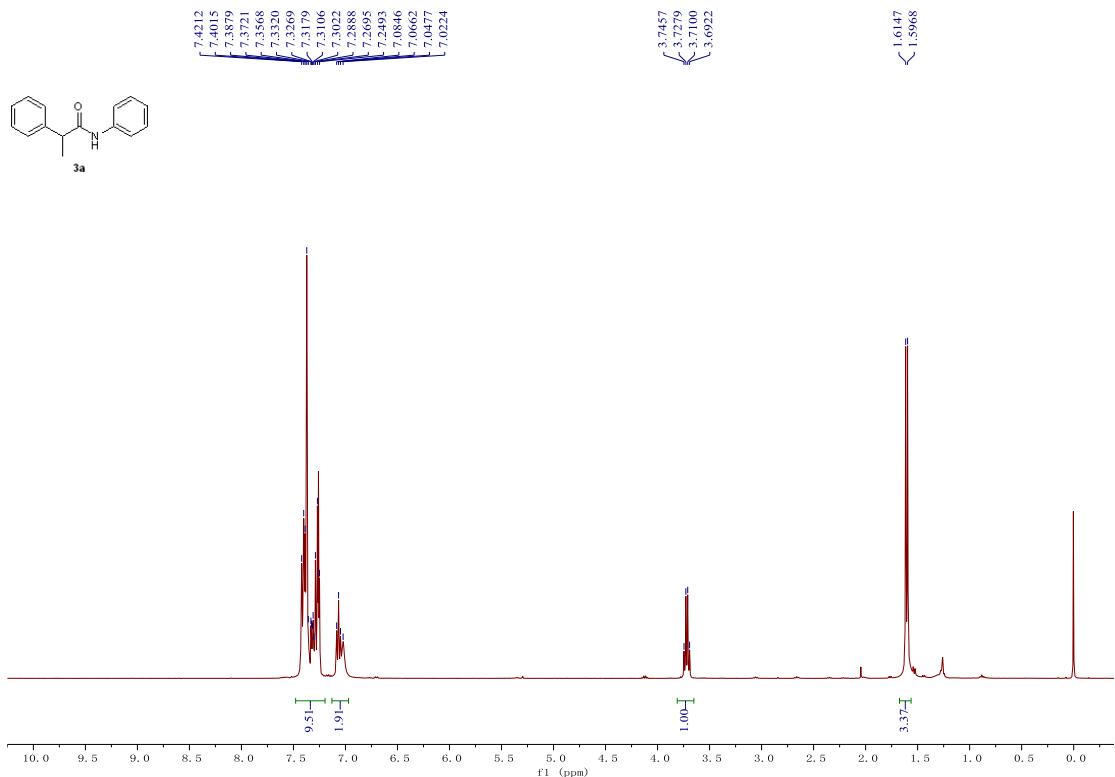
¹H NMR spectrum of **1aj**



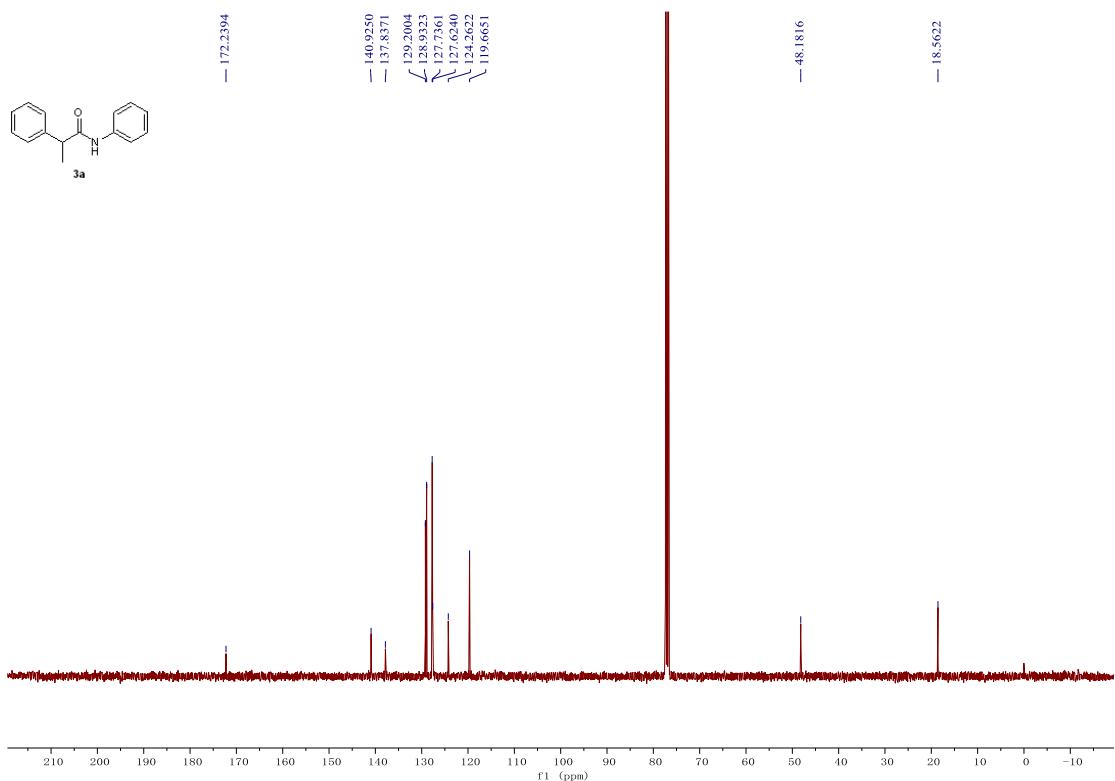
^{13}C NMR spectrum of 1aj



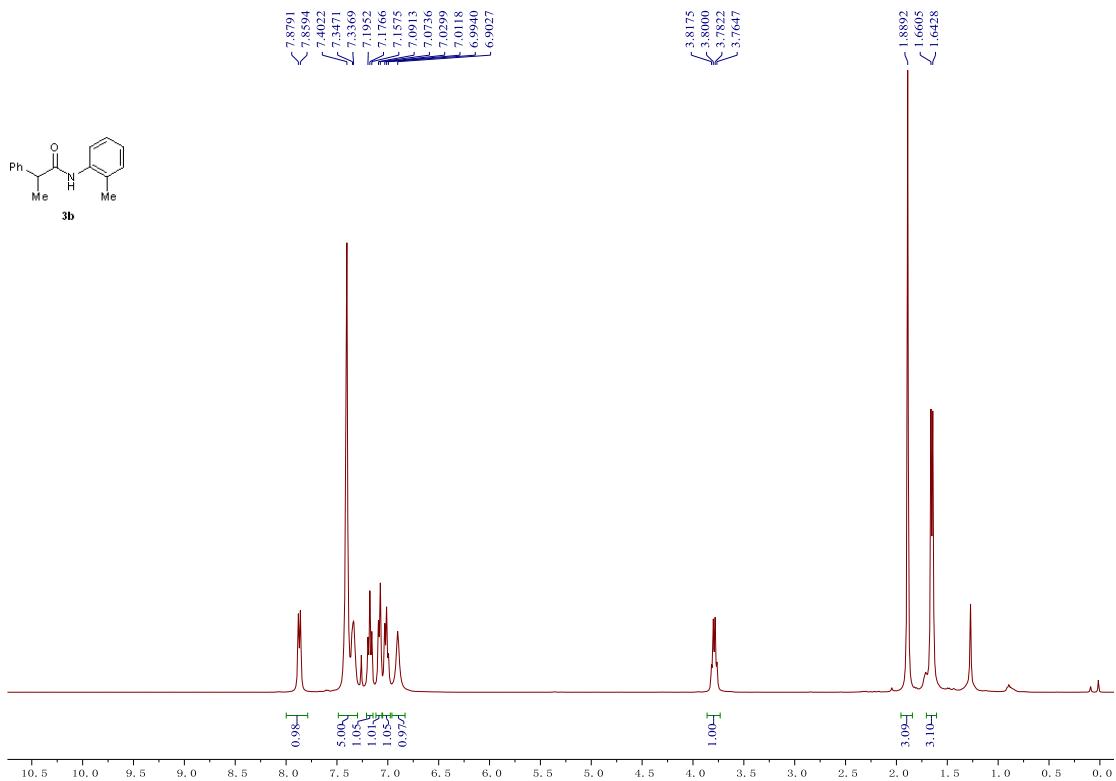
^1H NMR spectrum of 3a



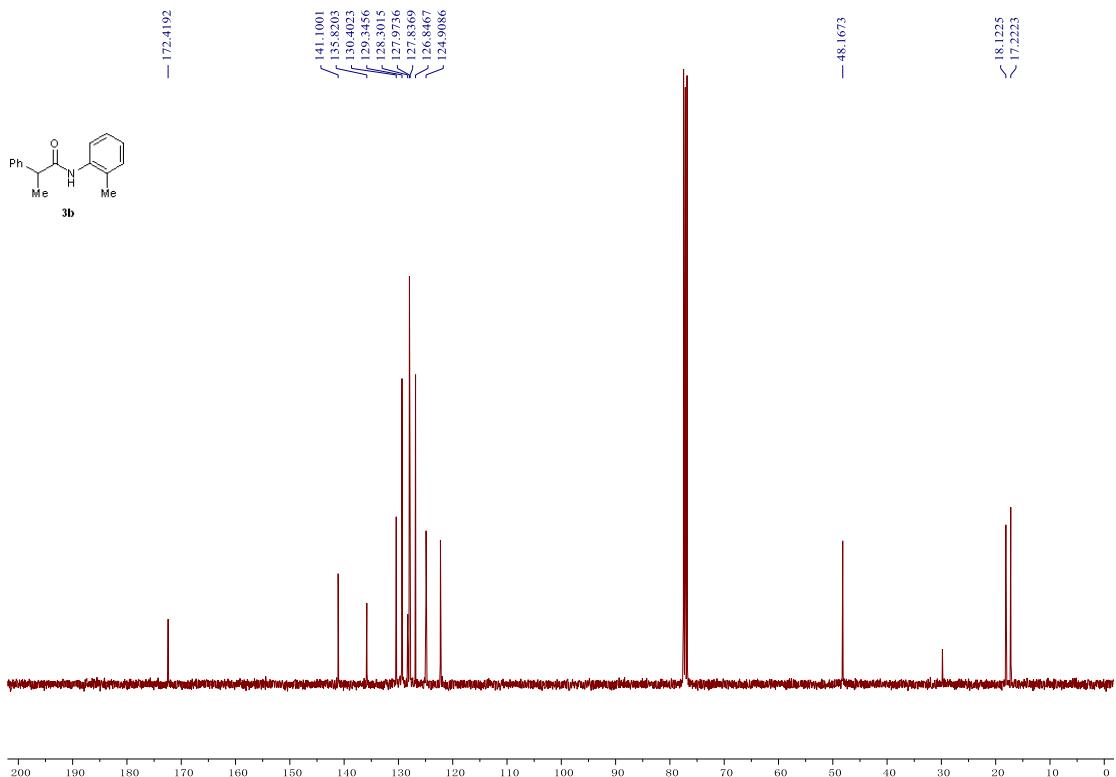
¹H NMR spectrum of **3a**



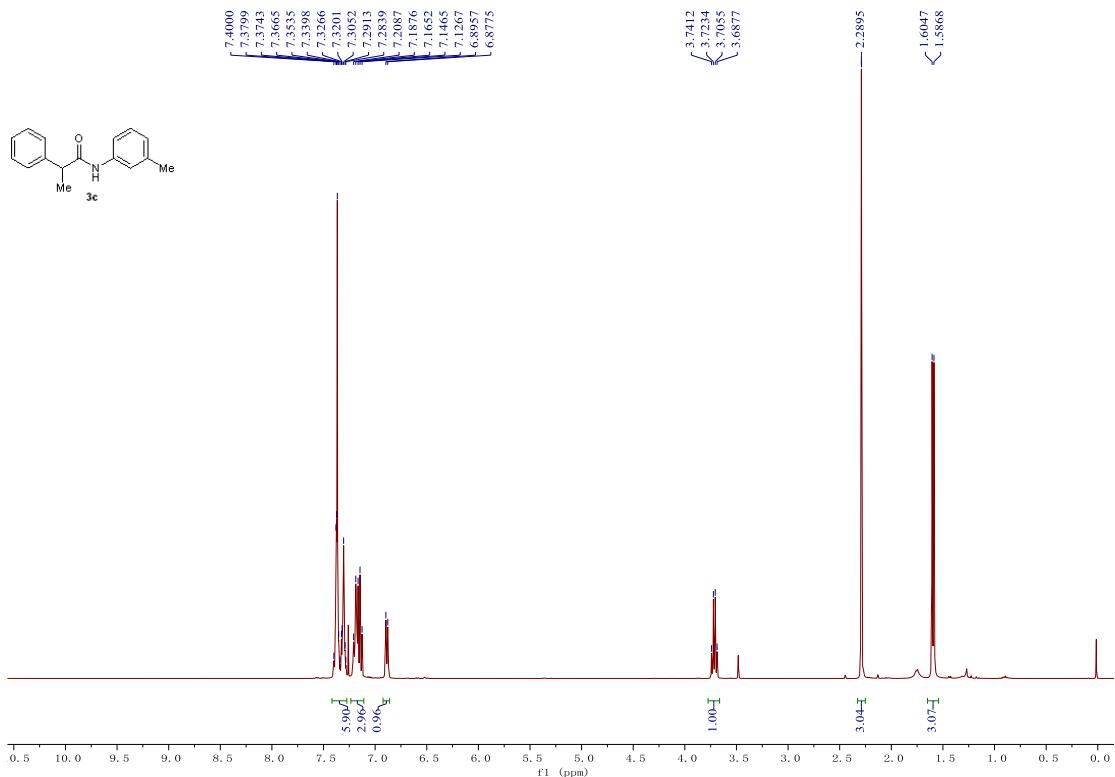
¹H NMR spectrum of **3b**



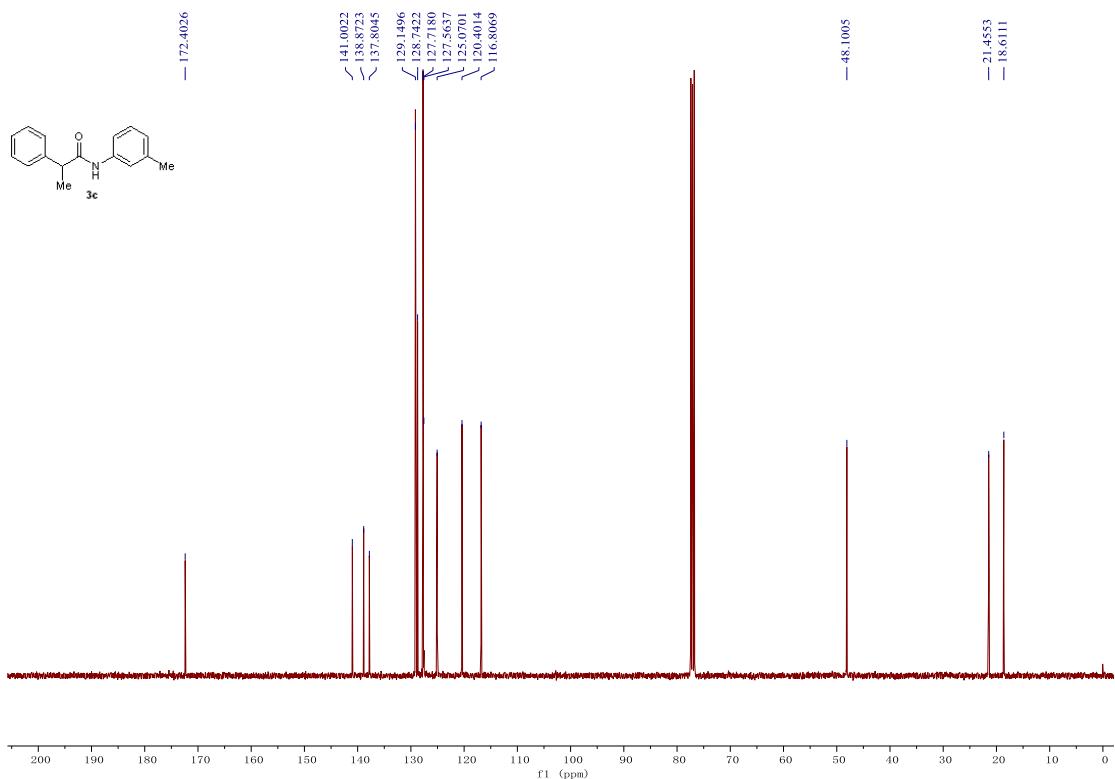
^1H NMR spectrum of **3b**



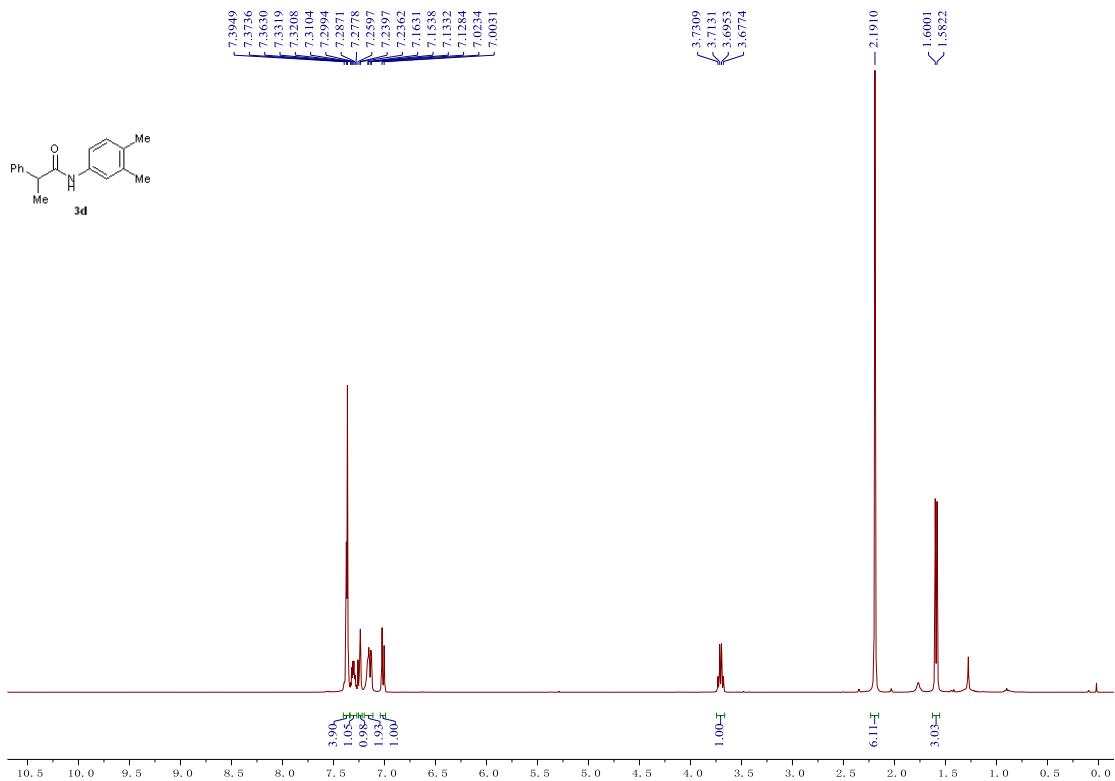
^1H NMR spectrum of **3c**



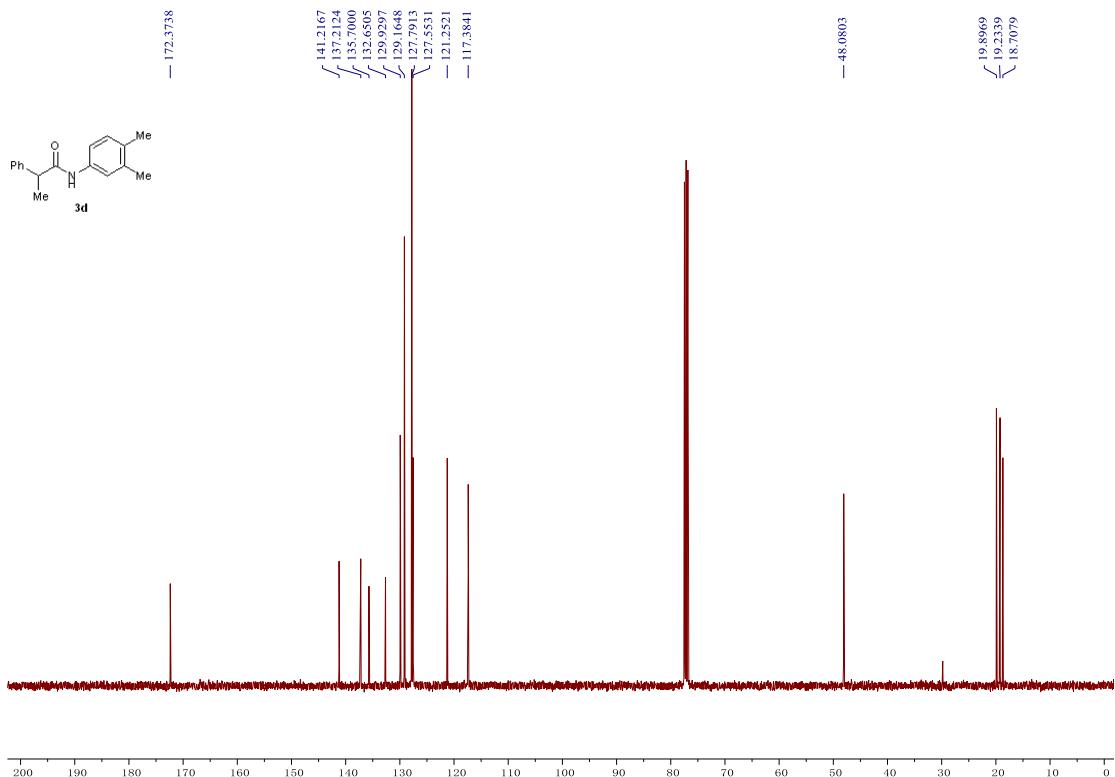
^1H NMR spectrum of **3c**



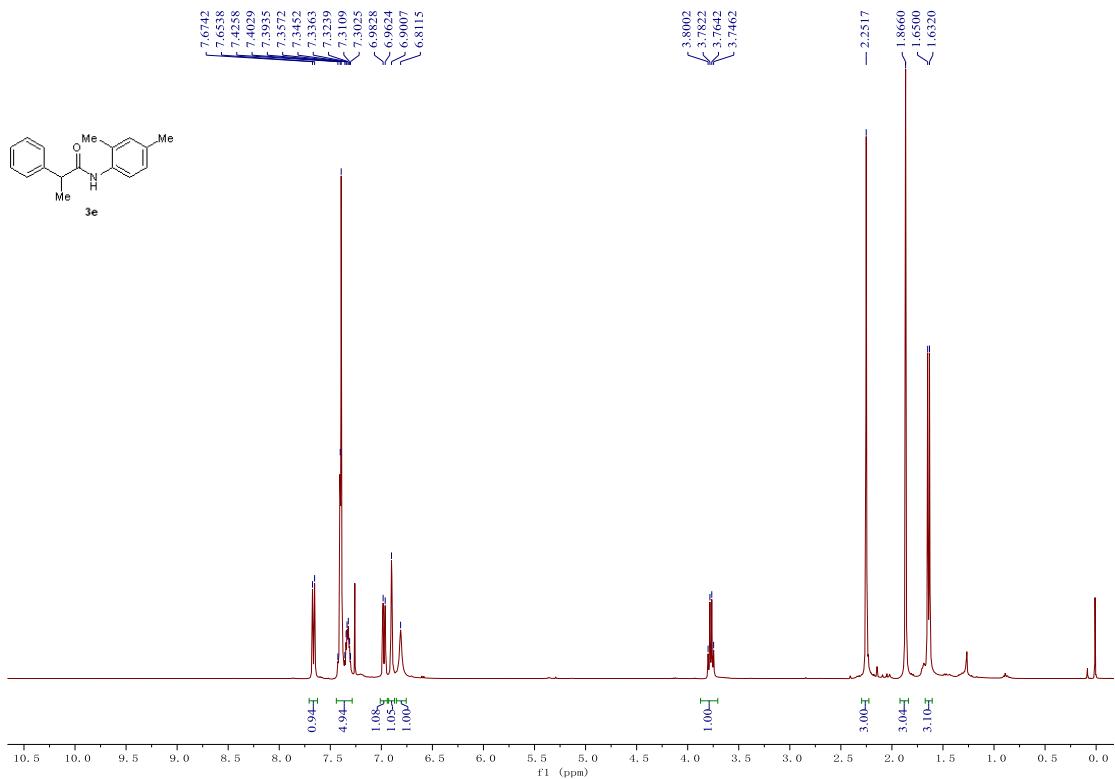
^{13}C NMR spectrum of **3c**



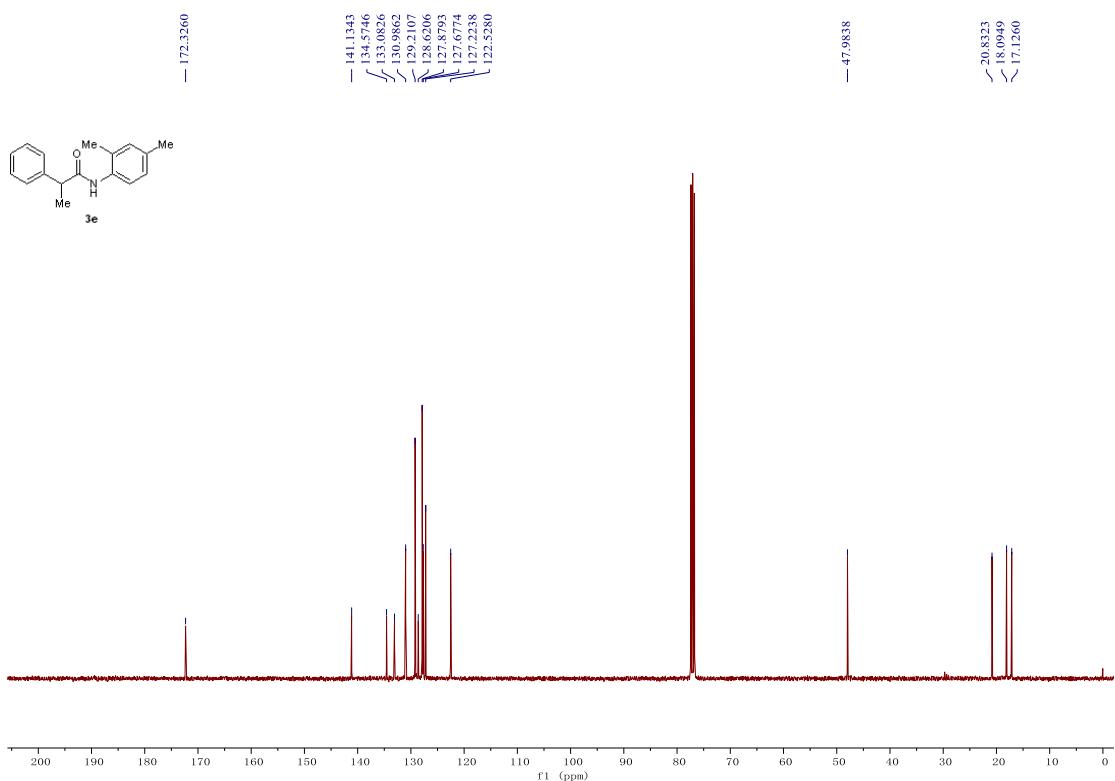
^{13}C NMR spectrum of **3d**



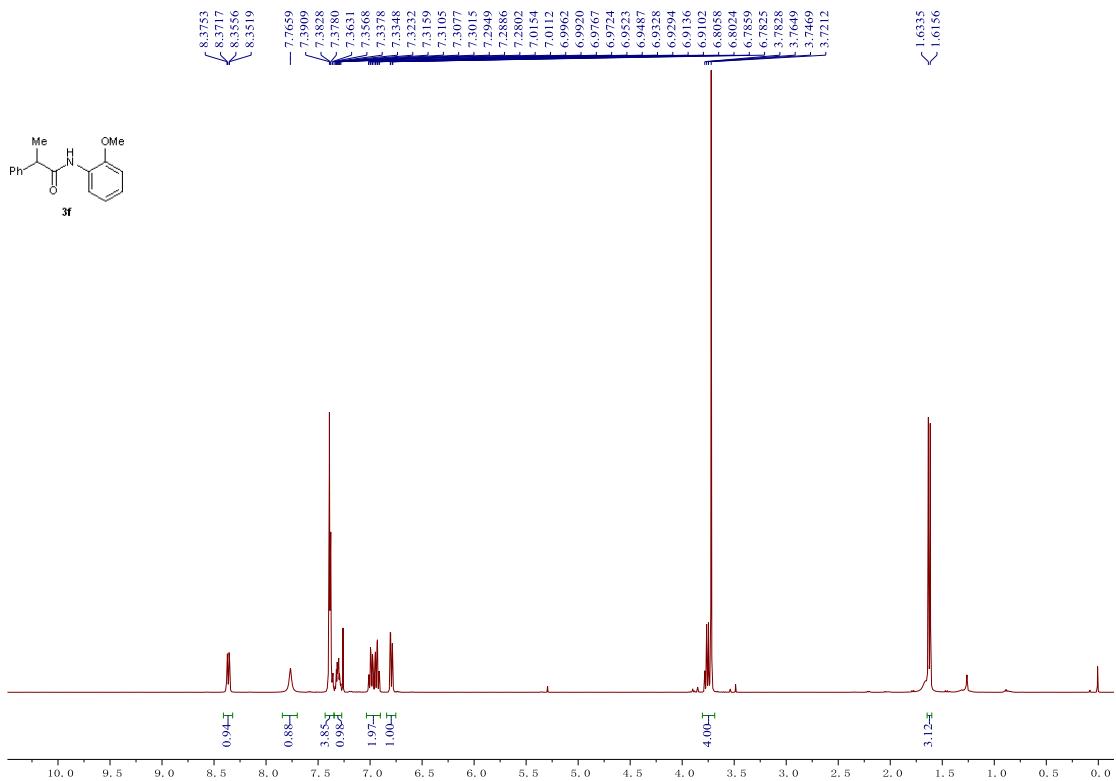
^1H NMR spectrum of **3e**



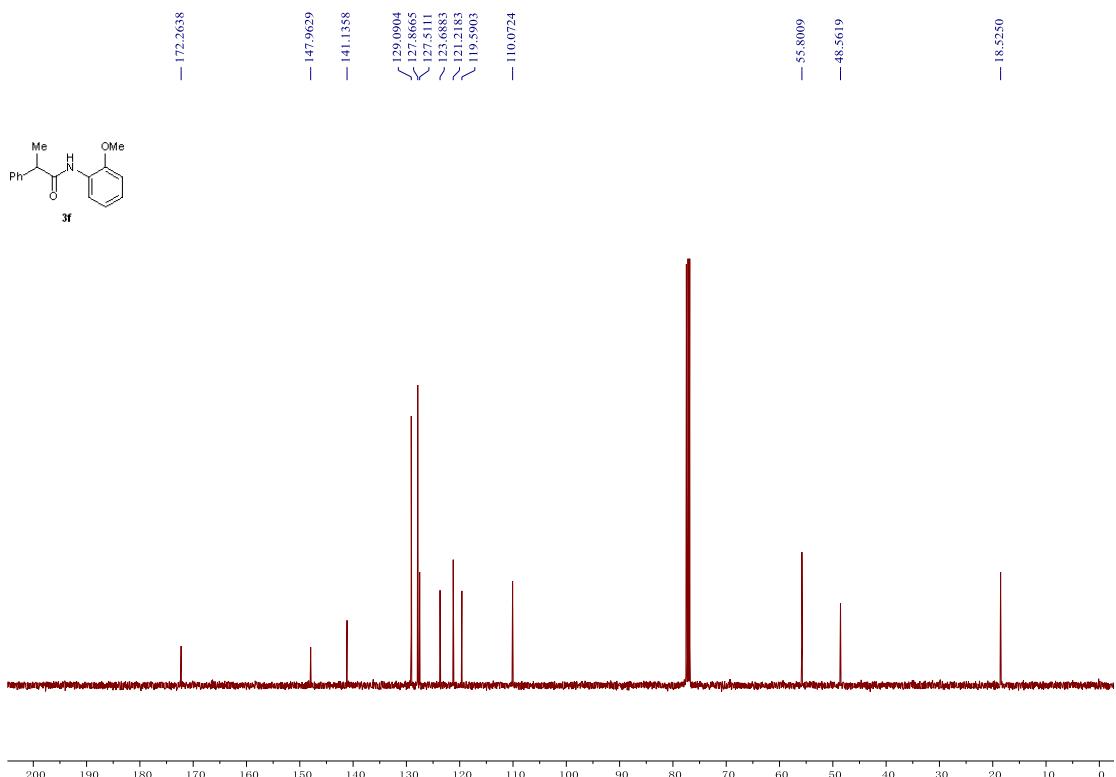
¹³C NMR spectrum of **3e**



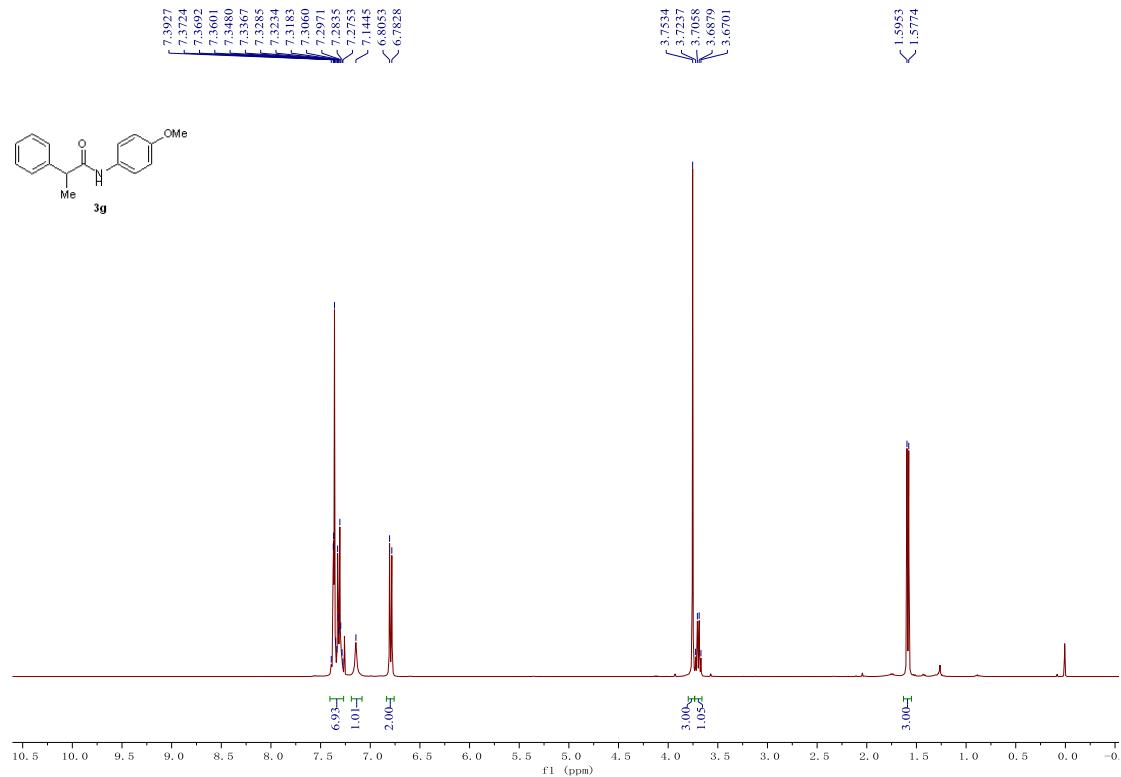
¹H NMR spectrum of **3f**



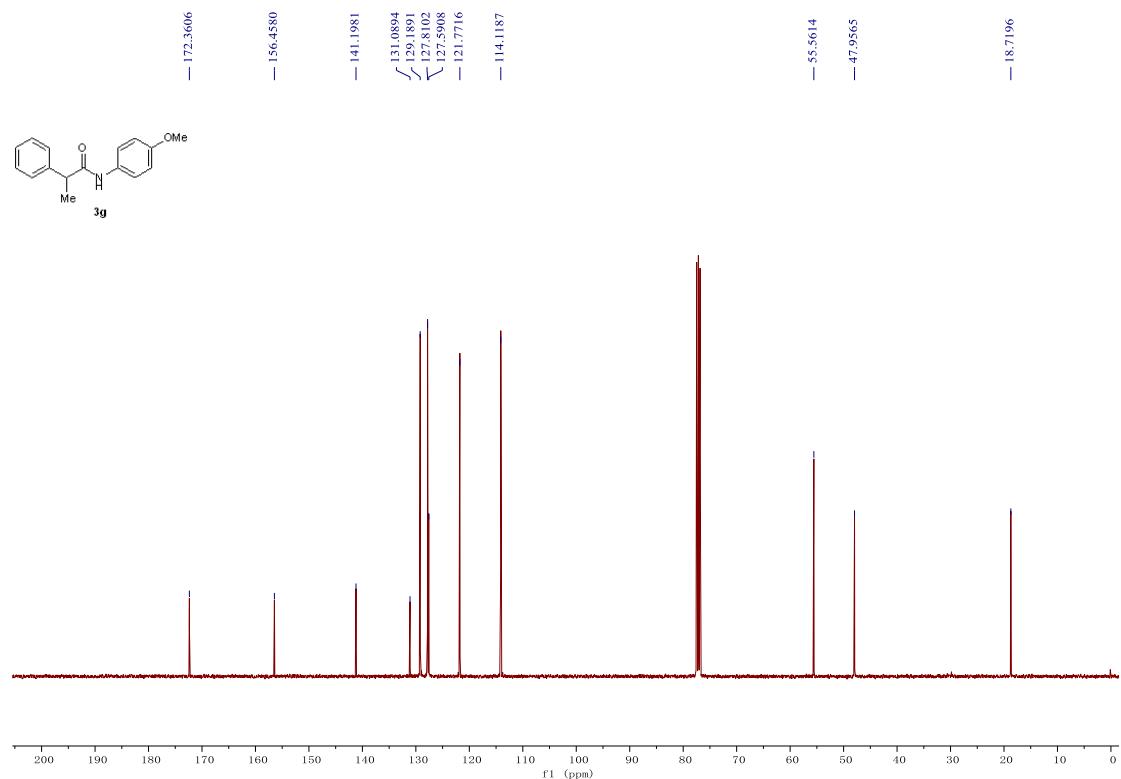
¹³C NMR spectrum of 3f



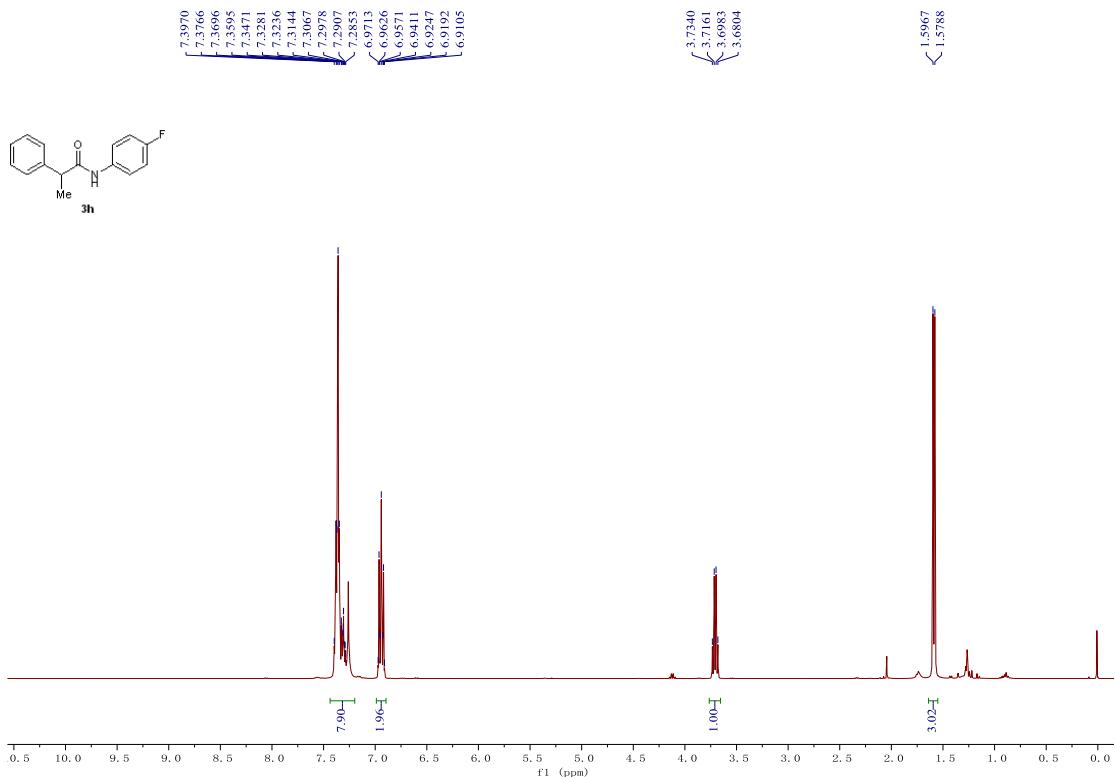
¹H NMR spectrum of 3g



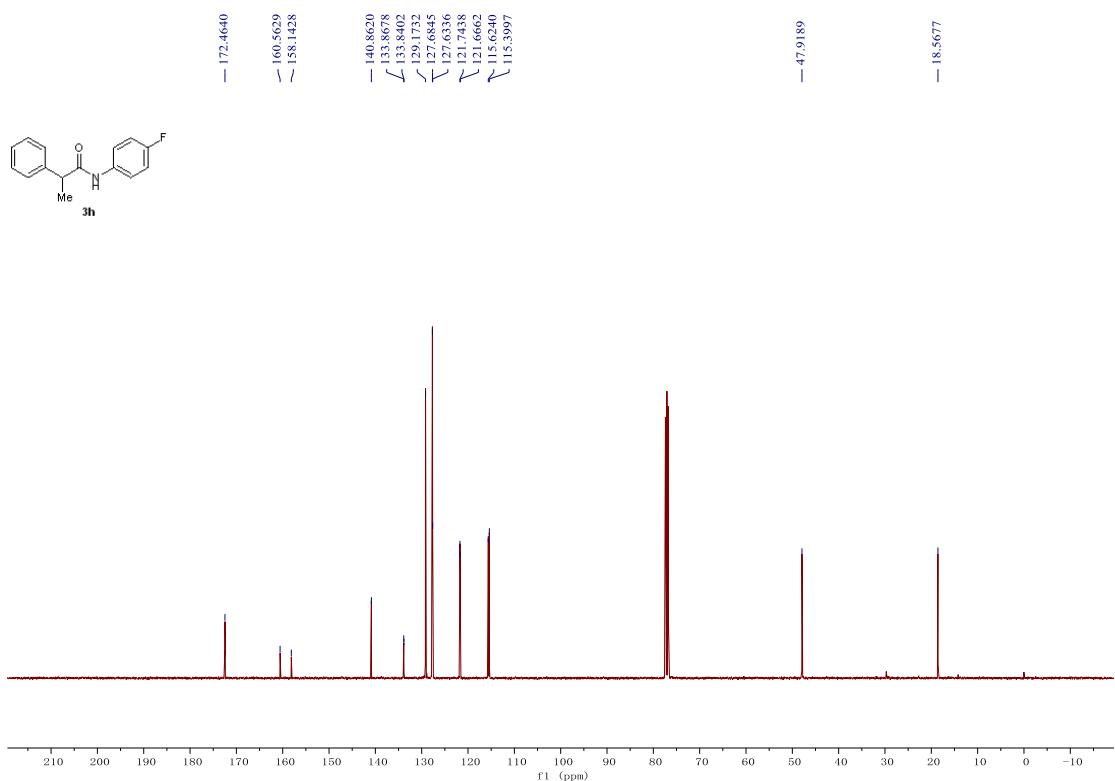
^{13}C NMR spectrum of **3g**



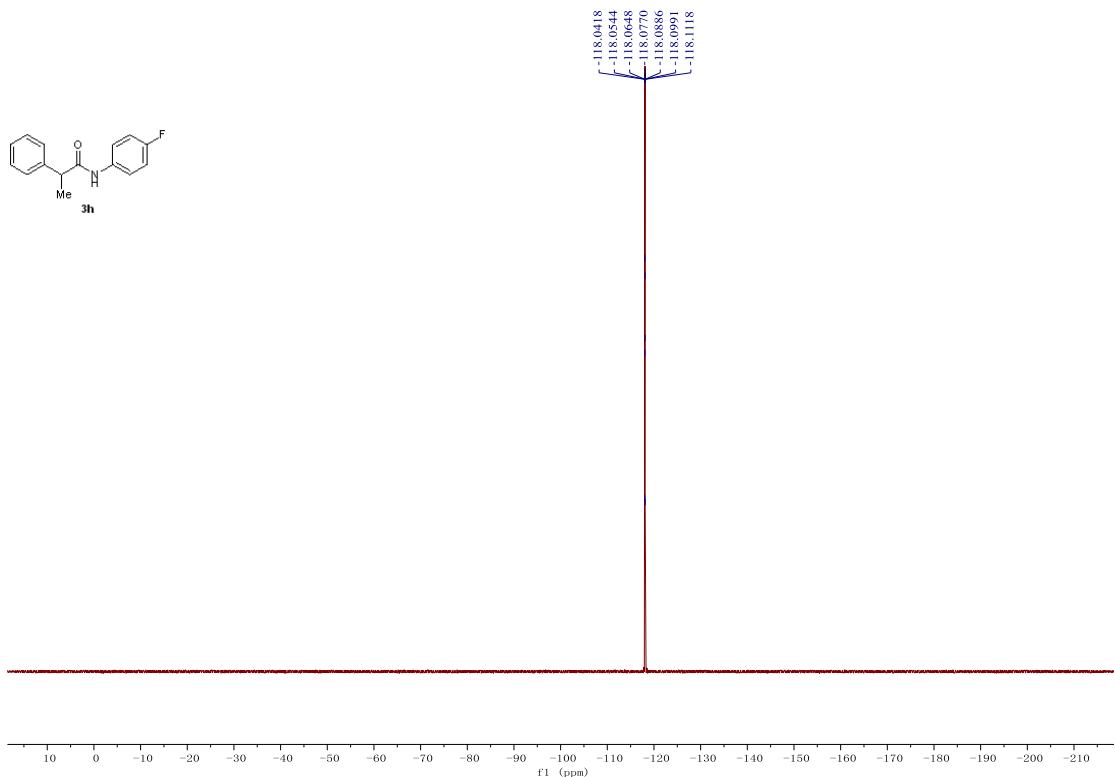
^1H NMR spectrum of **3h**



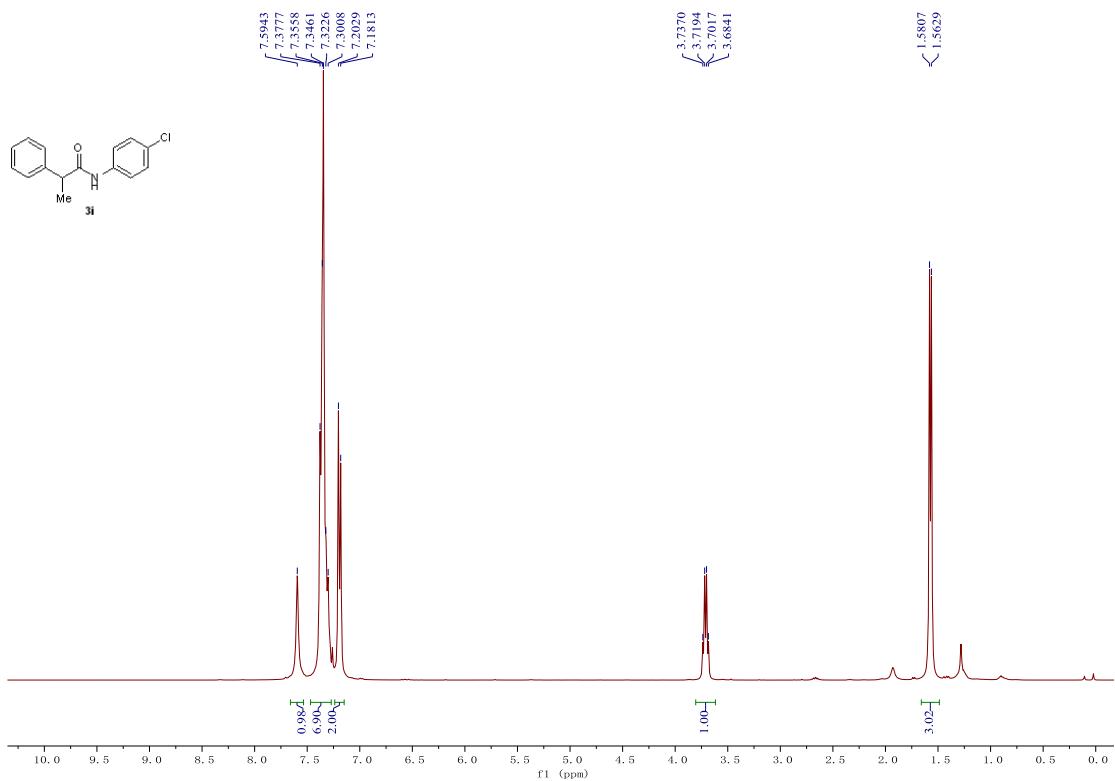
¹³C NMR spectrum of **3h**



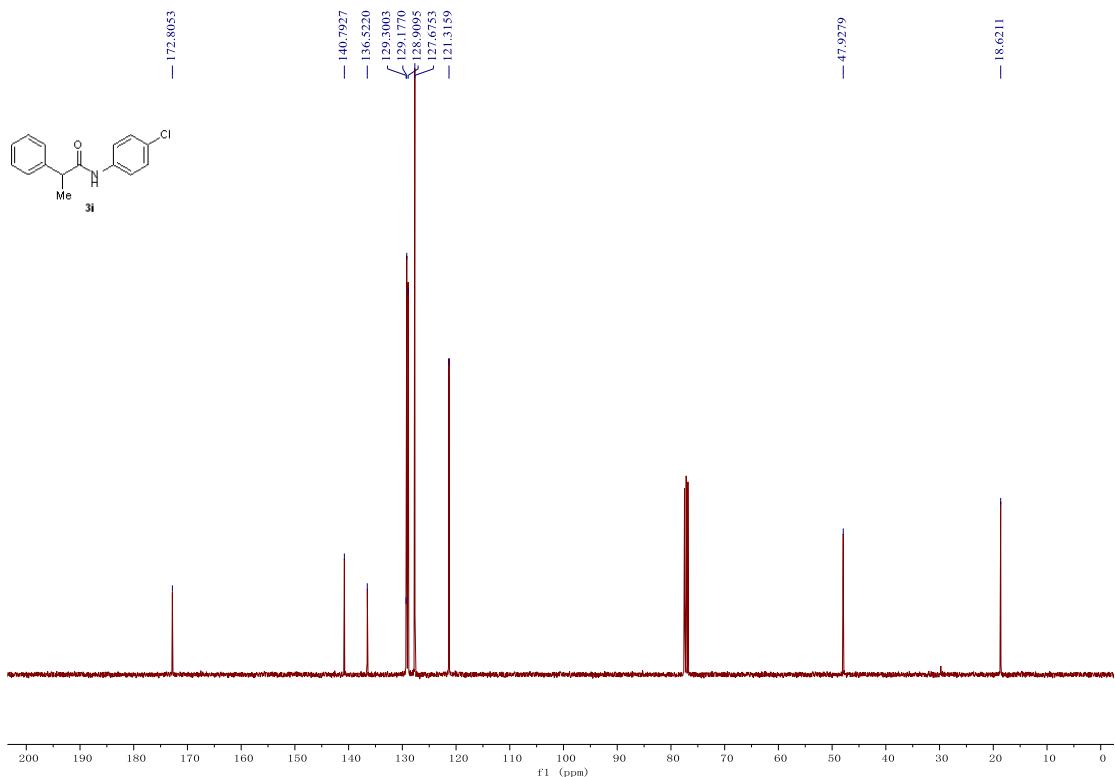
¹⁹F NMR spectrum of **3h**



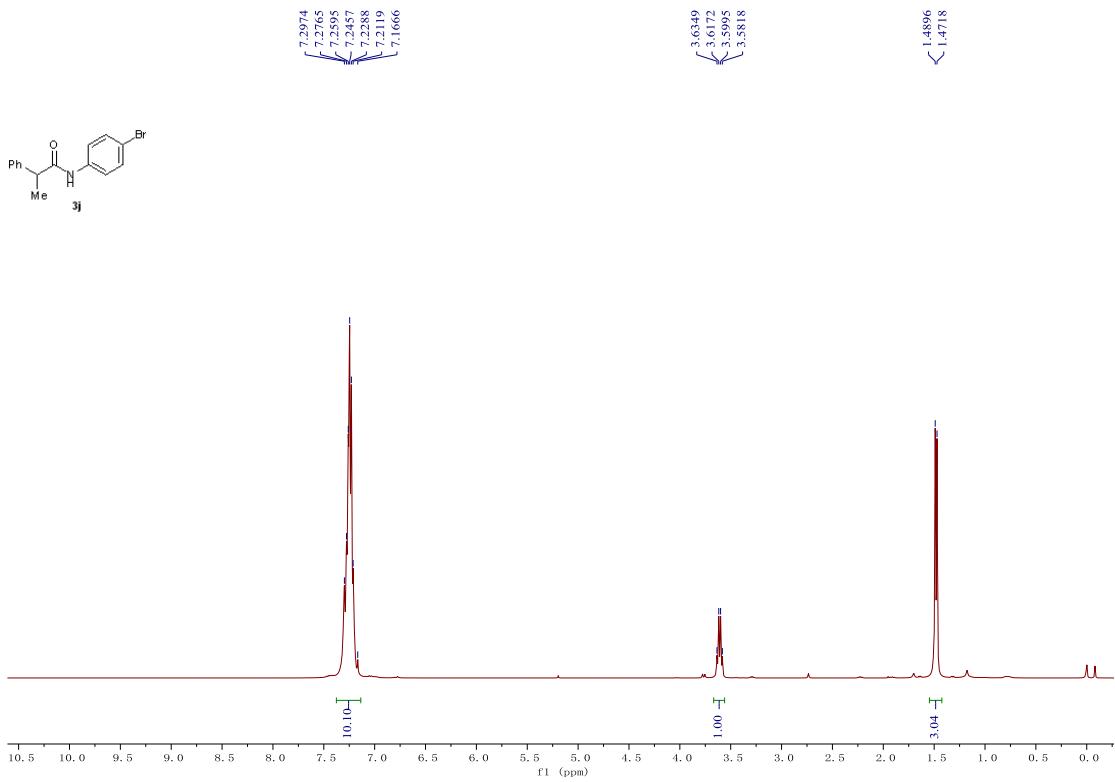
^1H NMR spectrum of **3i**



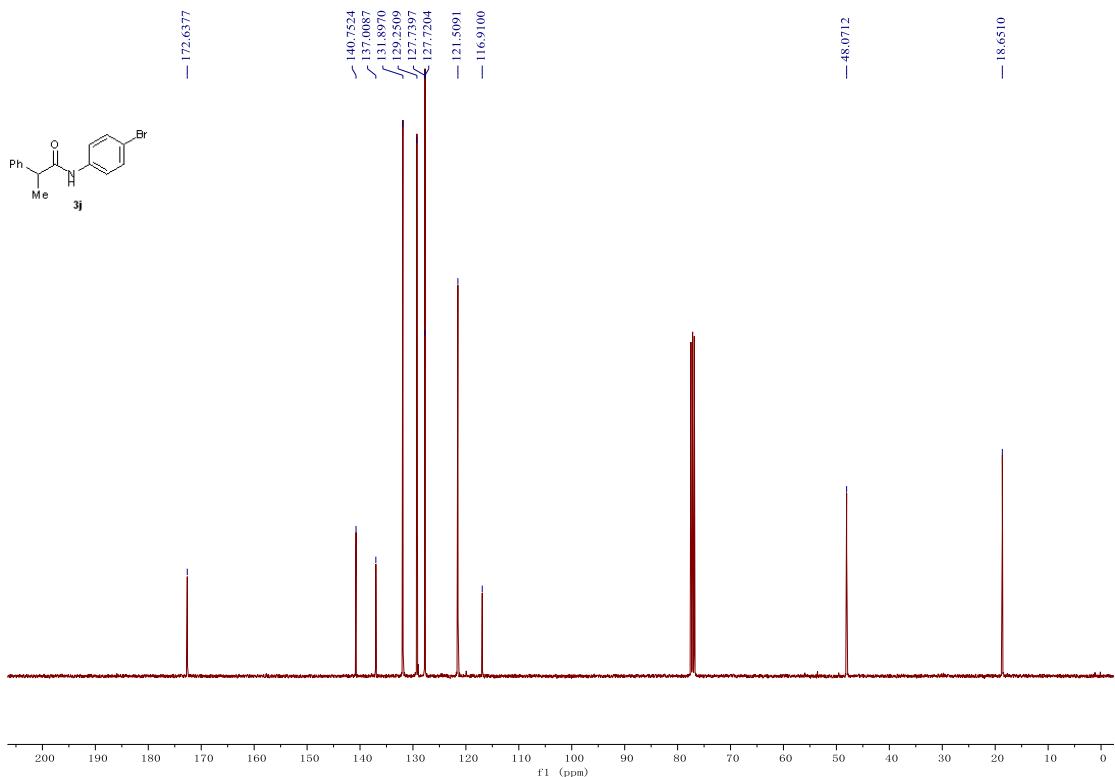
^{13}C NMR spectrum of **3i**



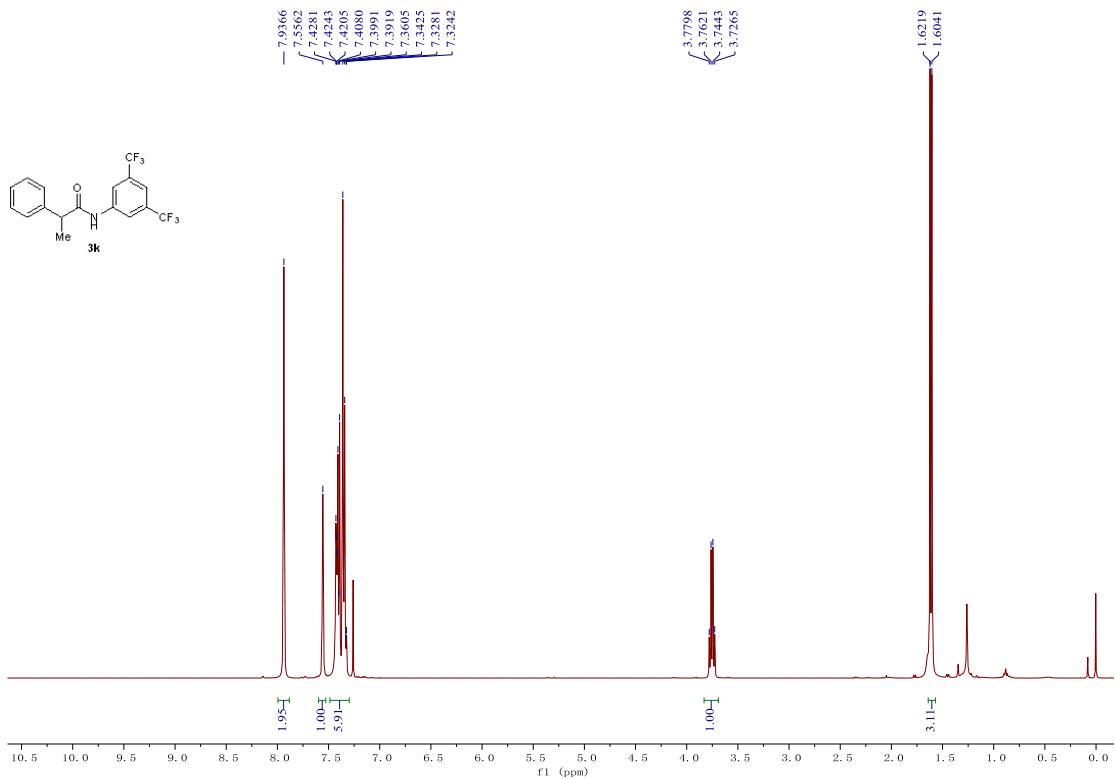
^1H NMR spectrum of **3j**



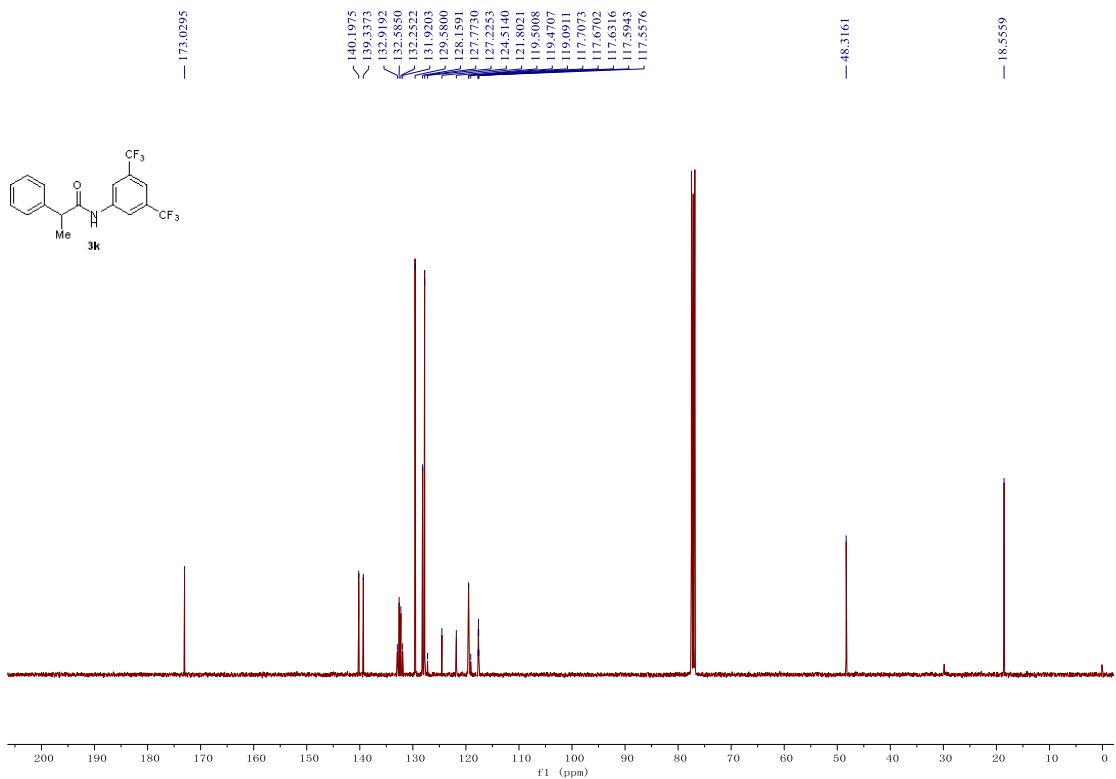
^{13}C NMR spectrum of **3j**



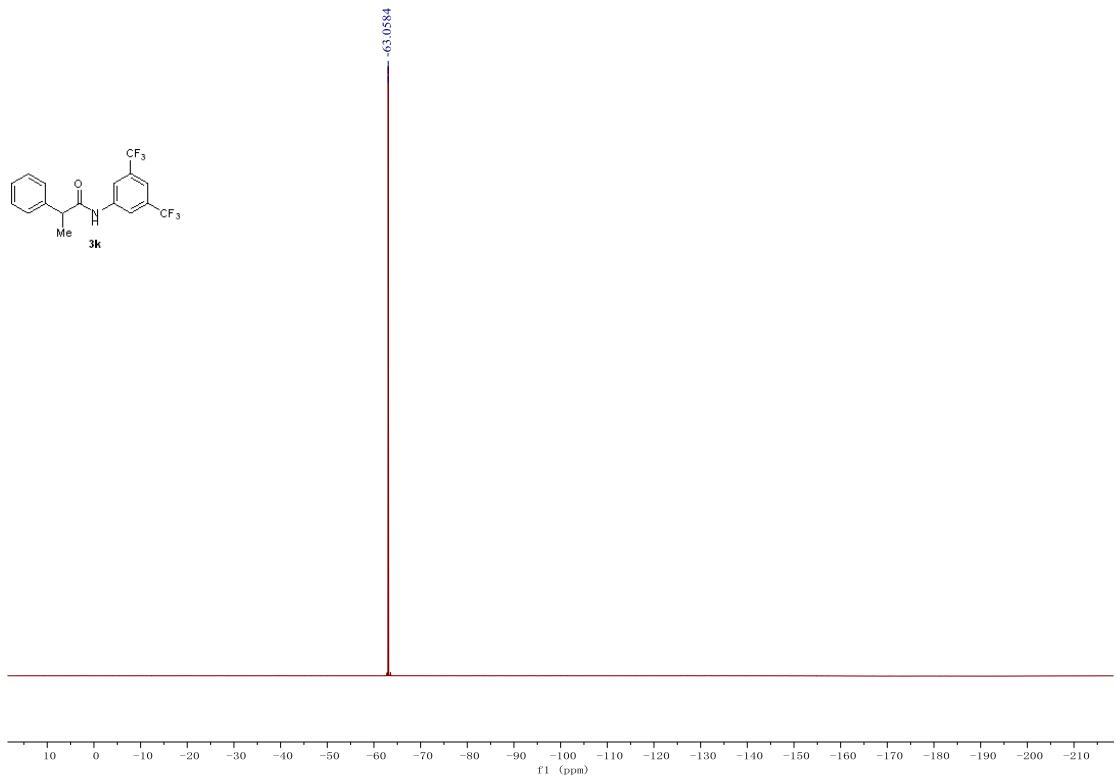
¹H NMR spectrum of **3k**



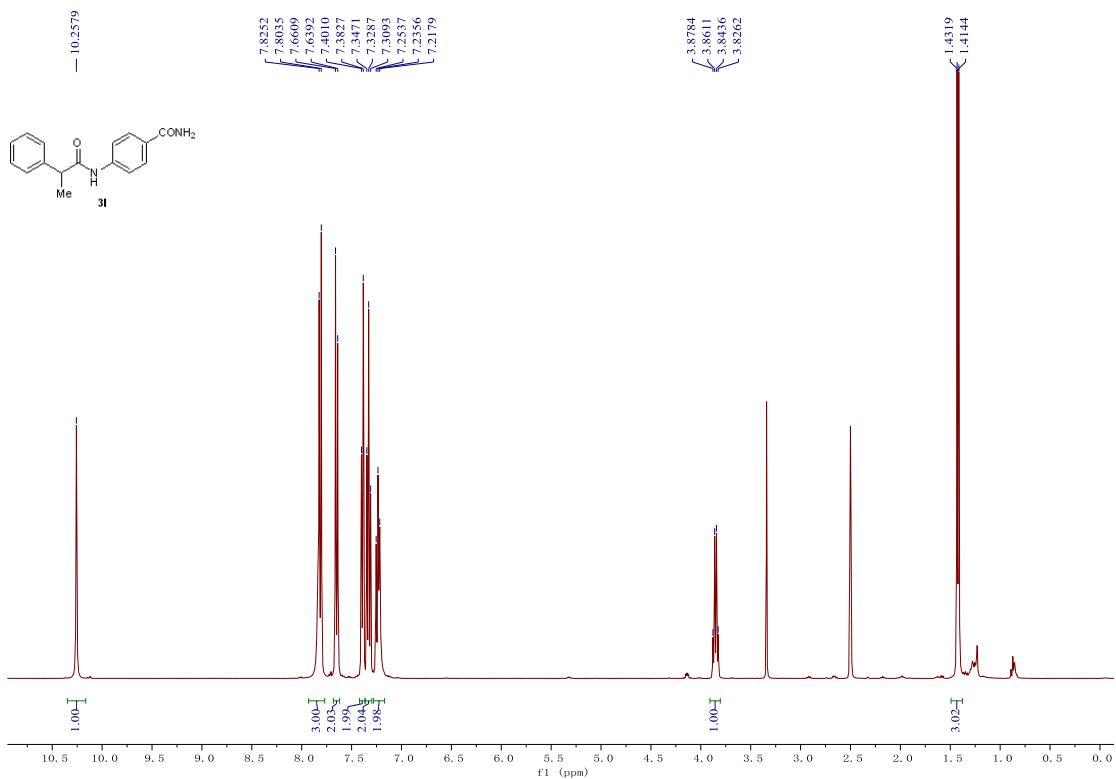
¹³C NMR spectrum of **3k**



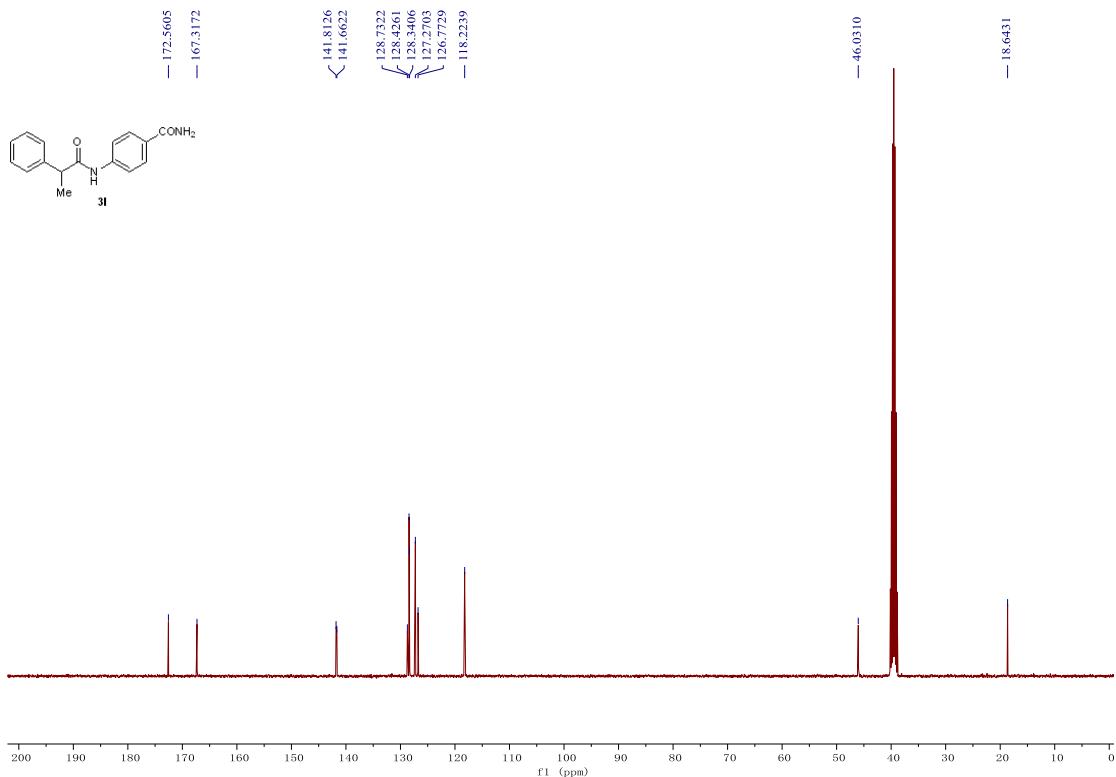
¹⁹F NMR spectrum of **3k**



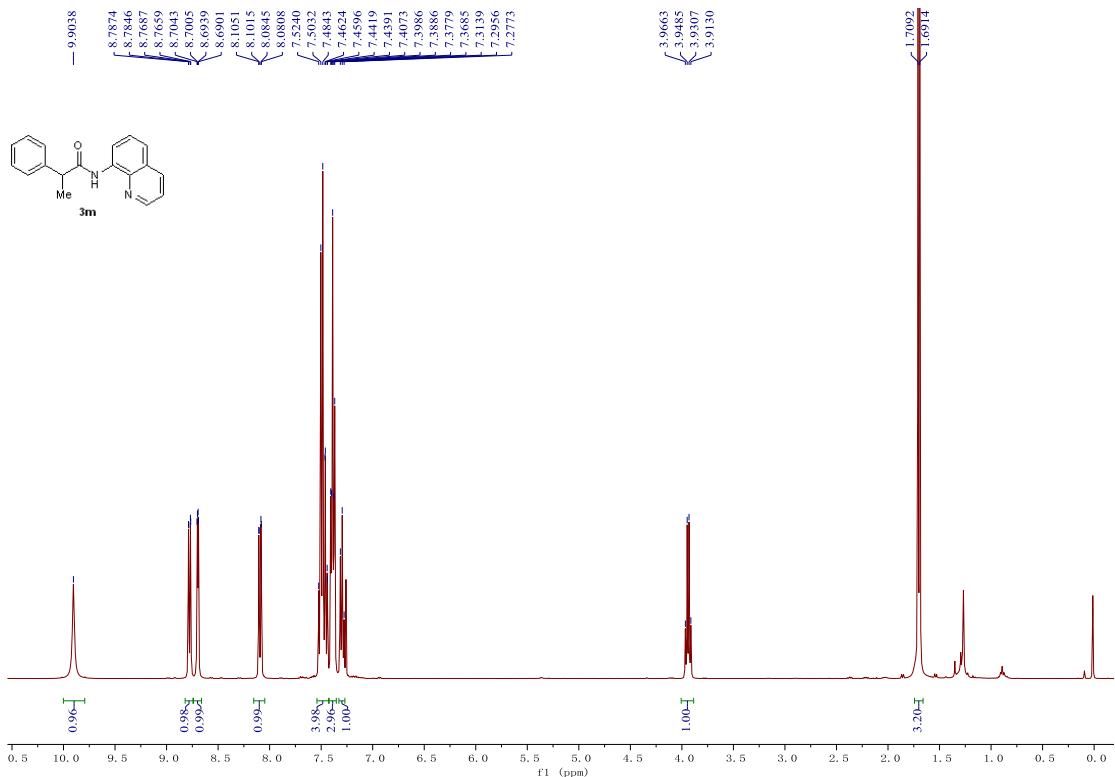
¹H NMR spectrum of **3l**



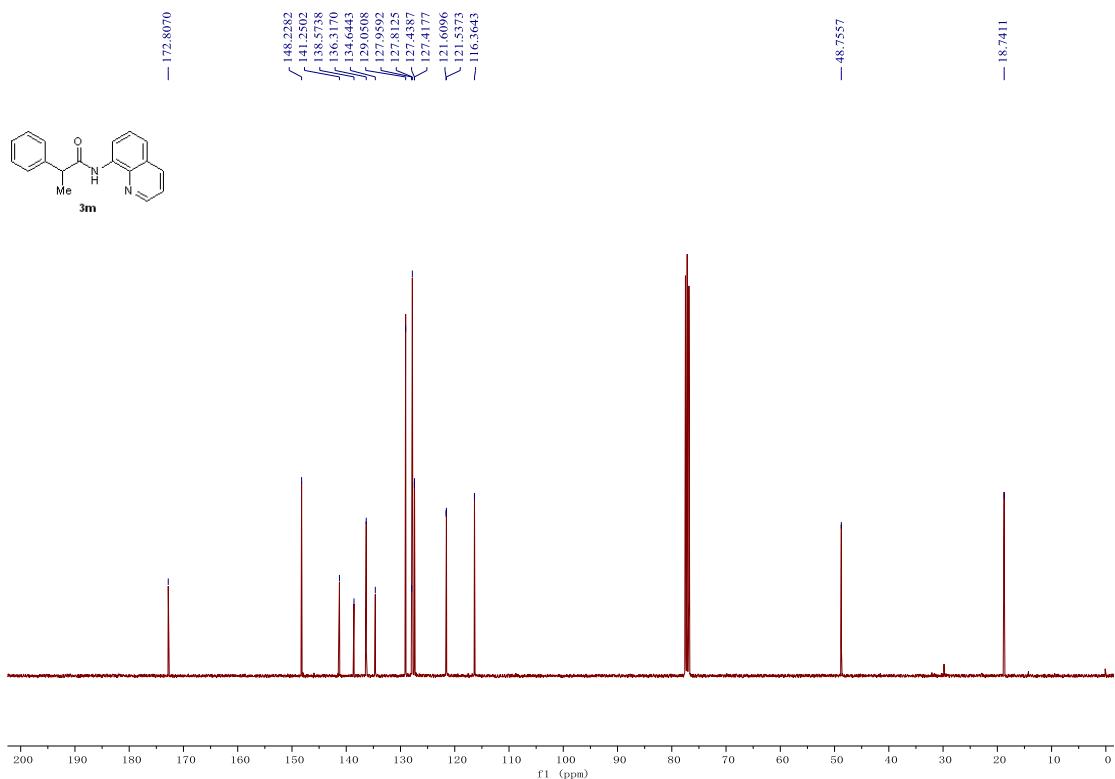
¹³C NMR spectrum of **3l**



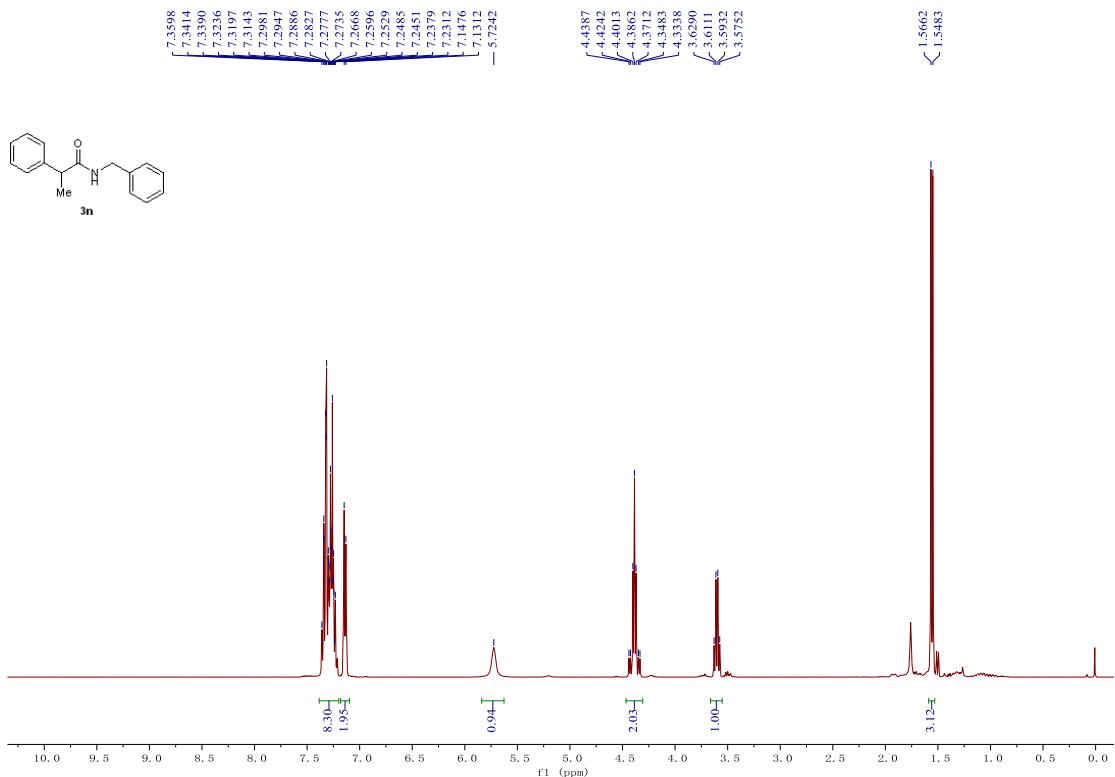
¹H NMR spectrum of **3m**



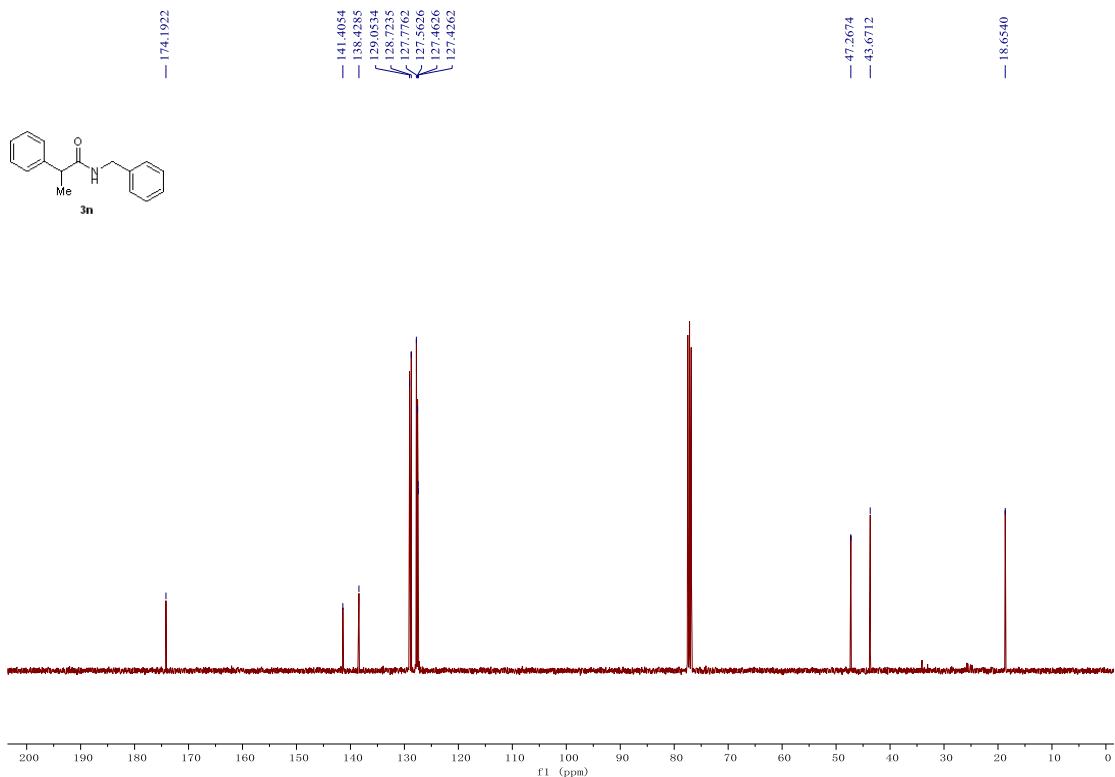
¹³C NMR spectrum of **3m**



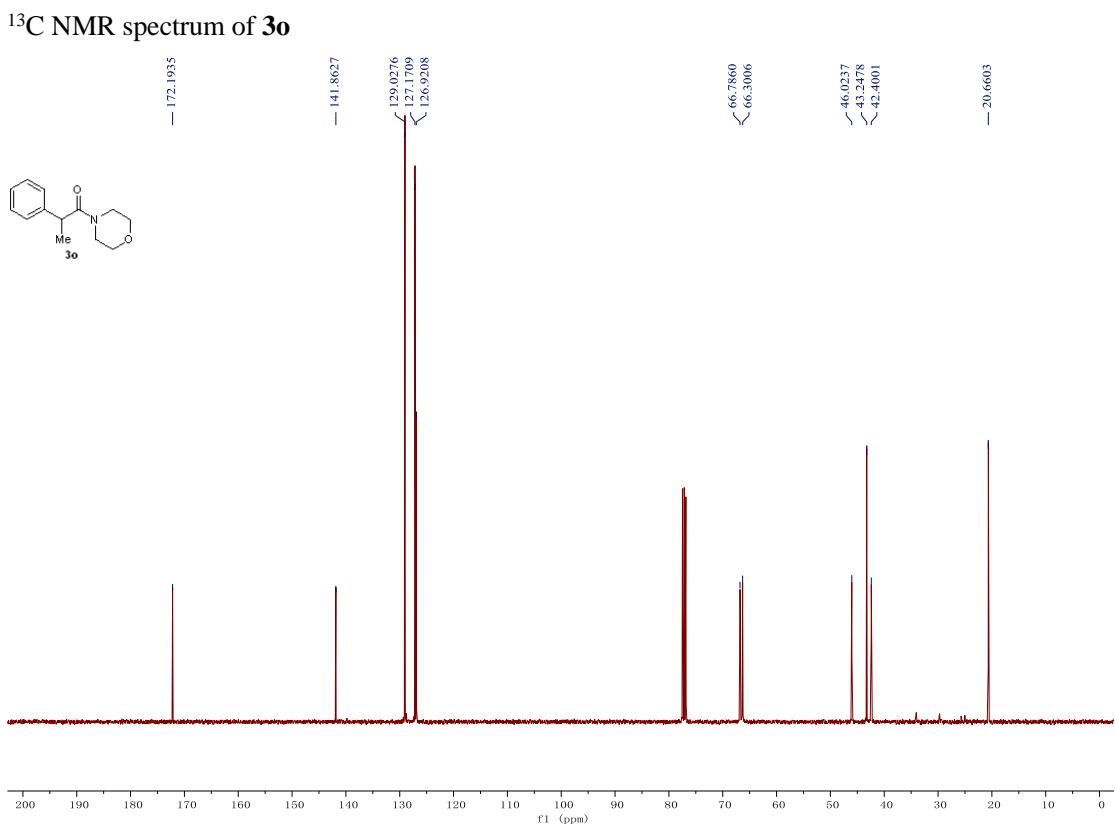
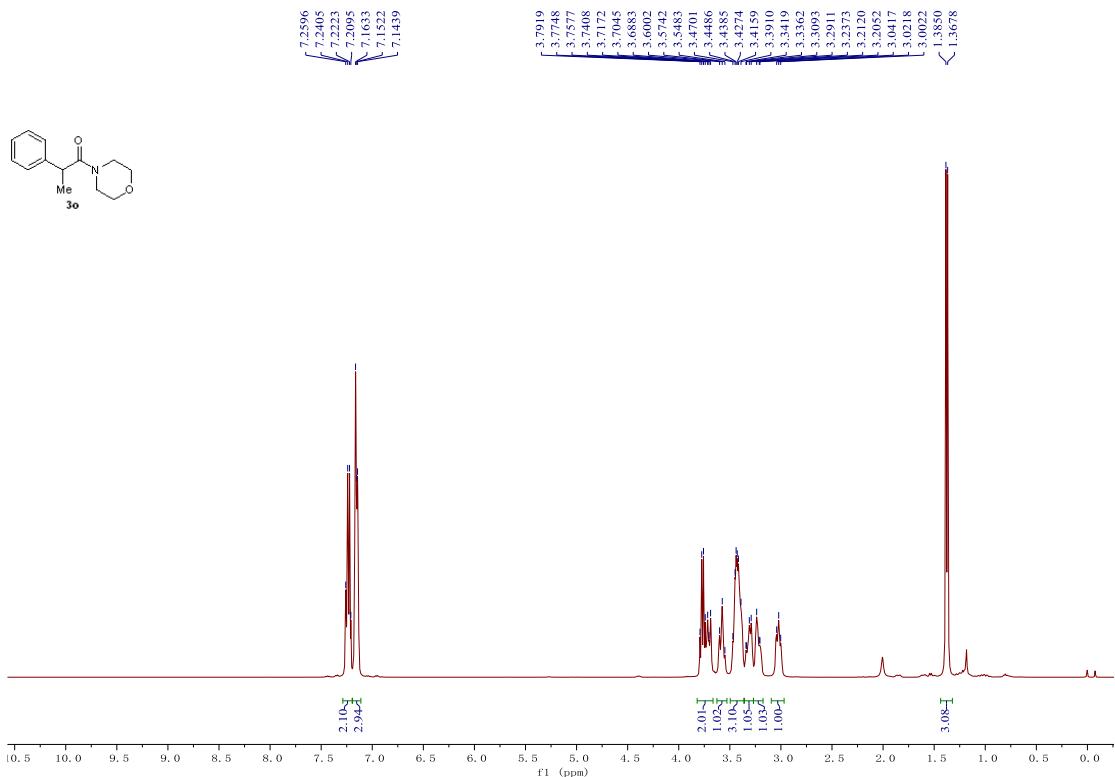
¹H NMR spectrum of **3n**



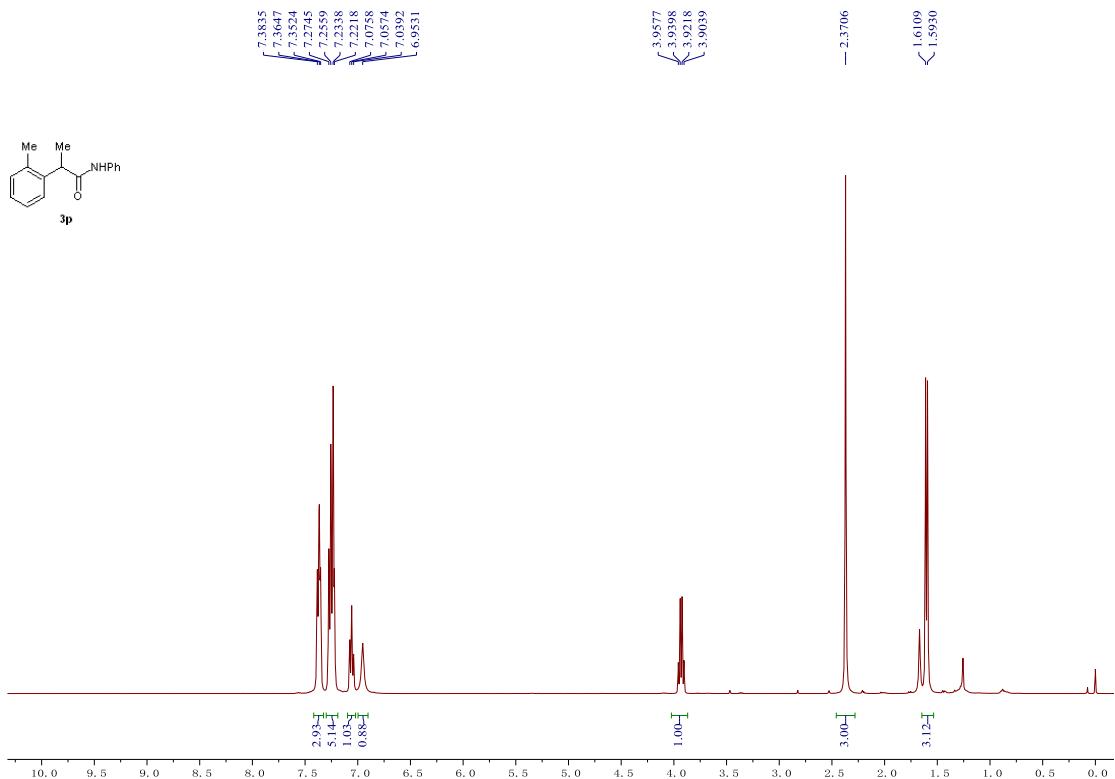
¹³C NMR spectrum of **3n**



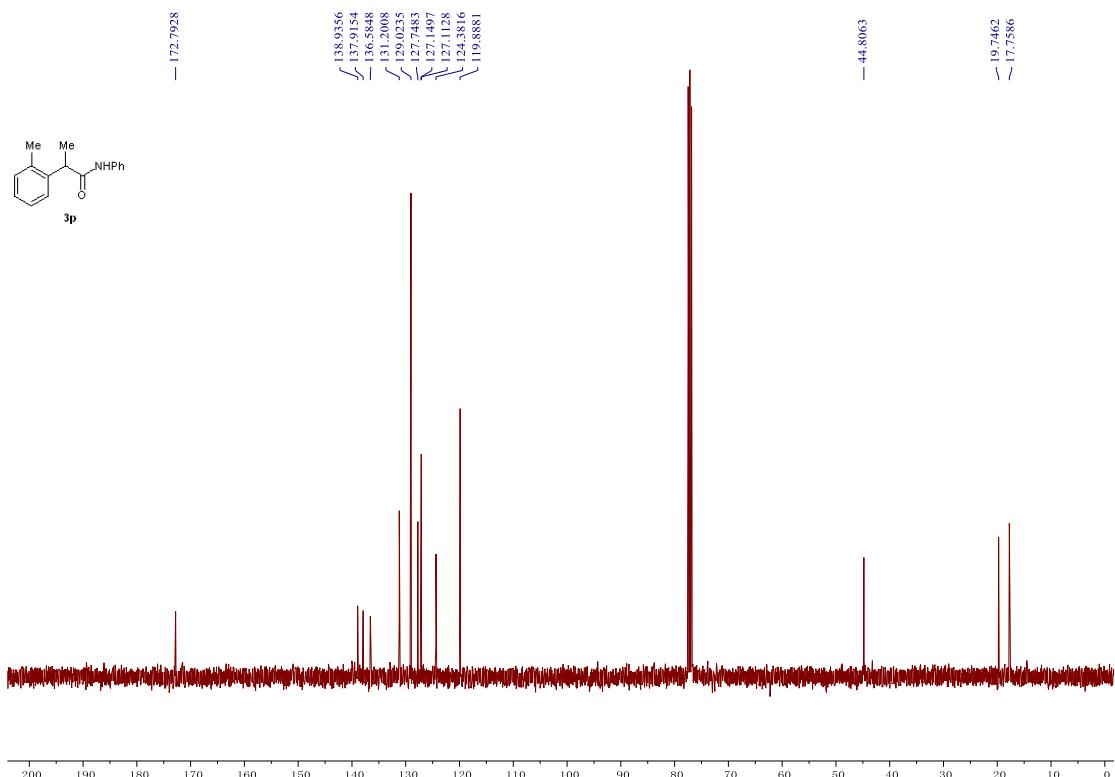
¹H NMR spectrum of **3o**



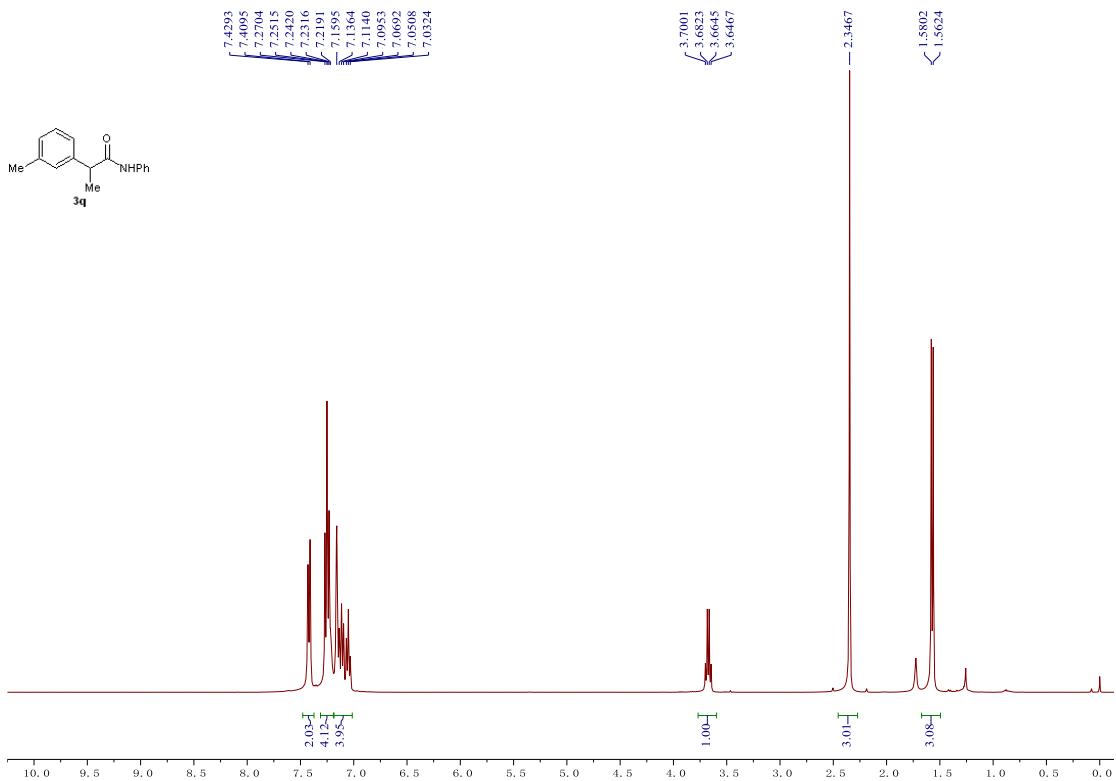
¹H NMR spectrum of **3p**



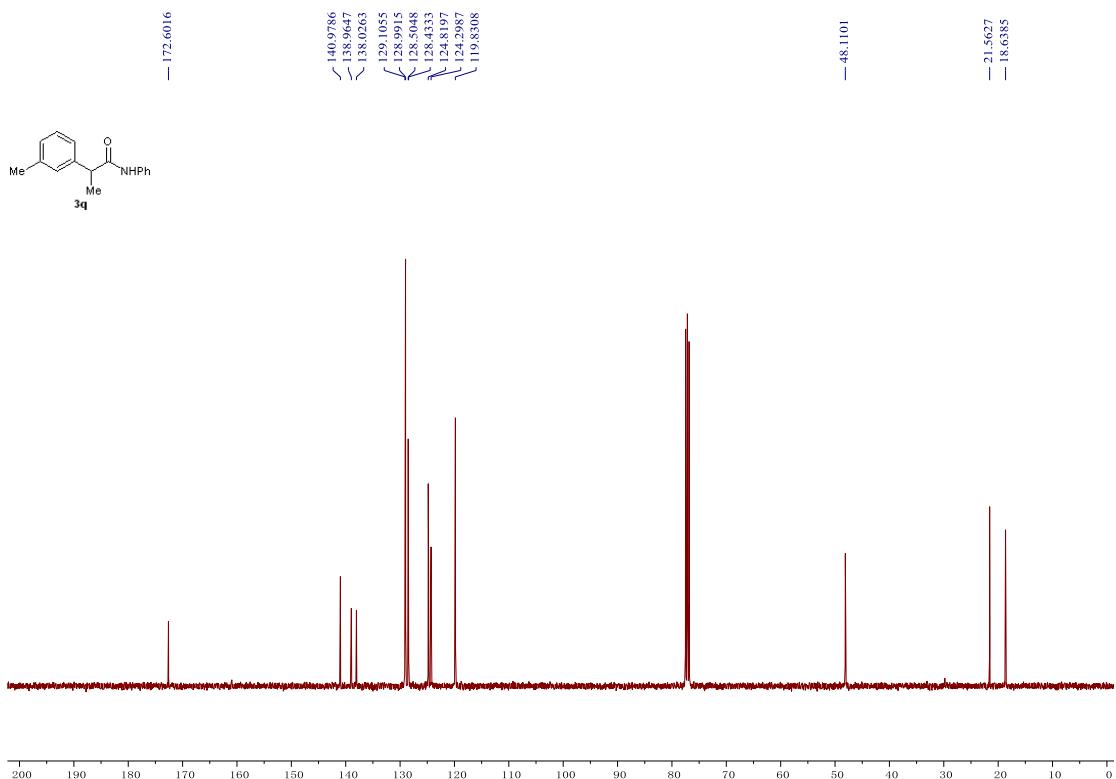
¹³C NMR spectrum of **3p**



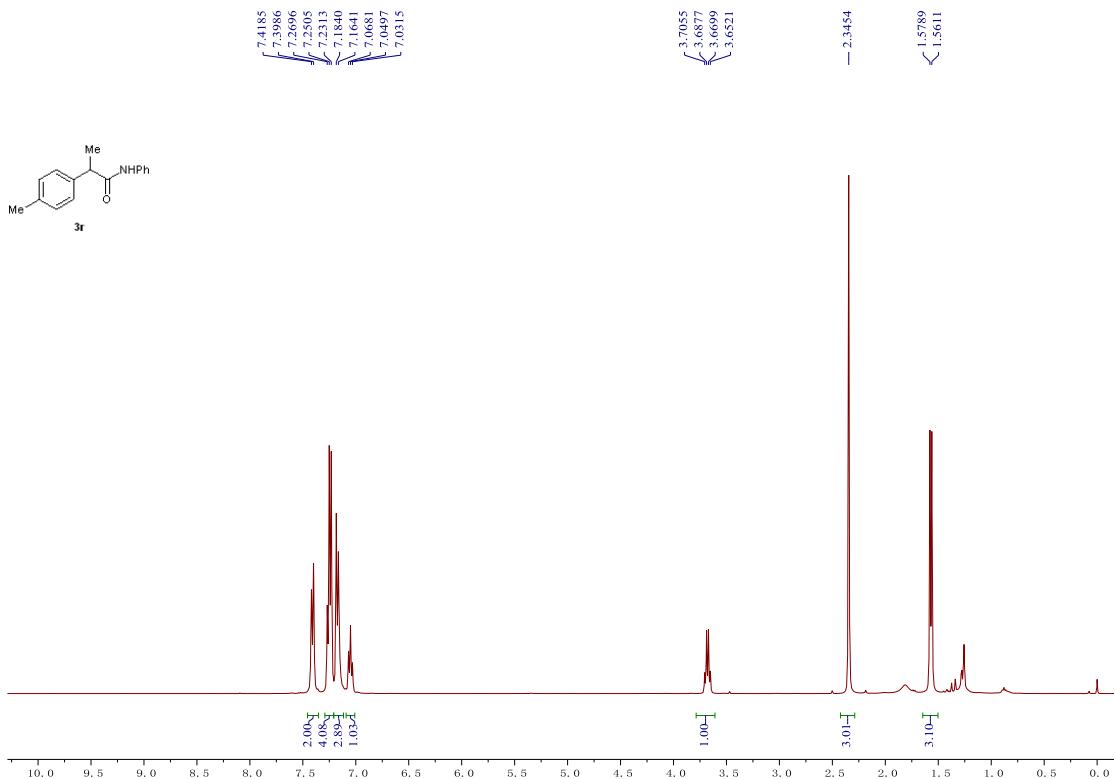
¹H NMR spectrum of **3q**



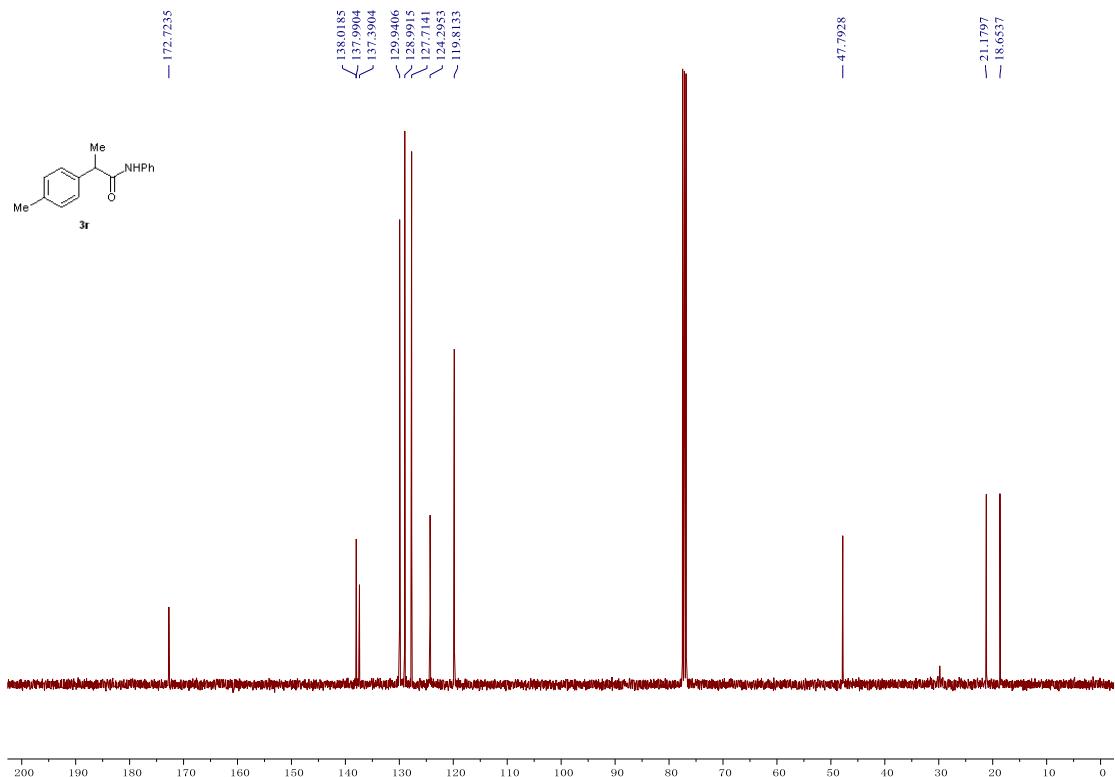
¹³C NMR spectrum of **3q**



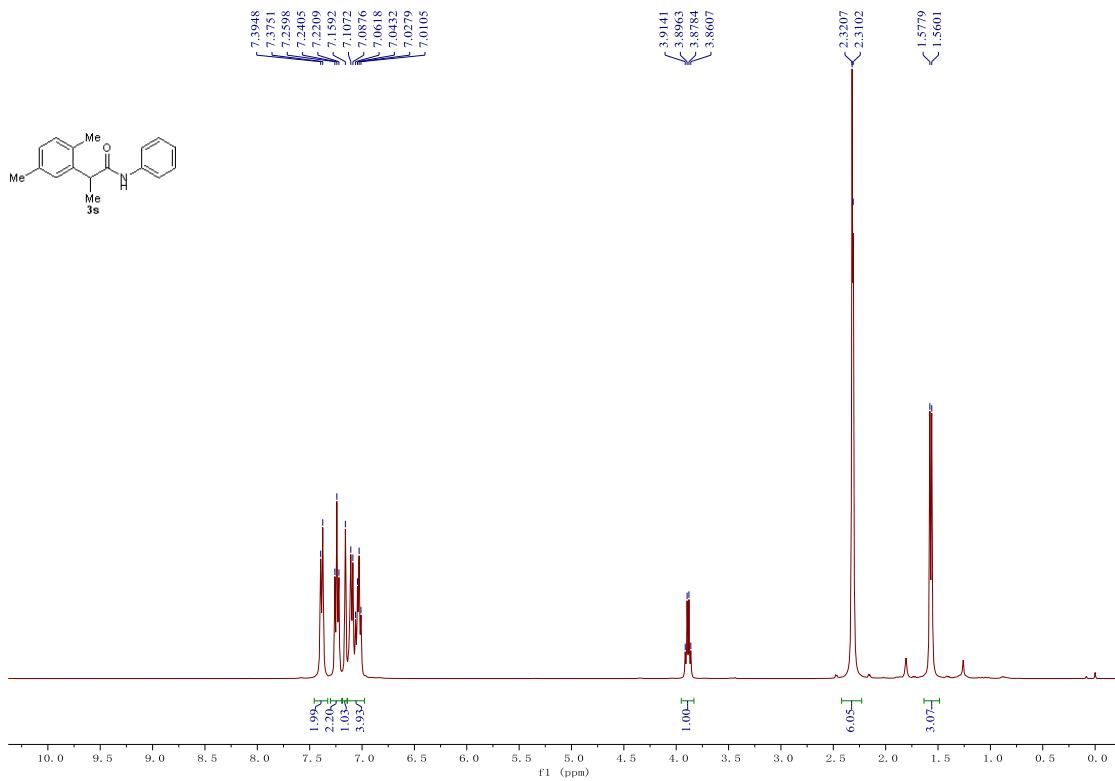
¹H NMR spectrum of **3r**



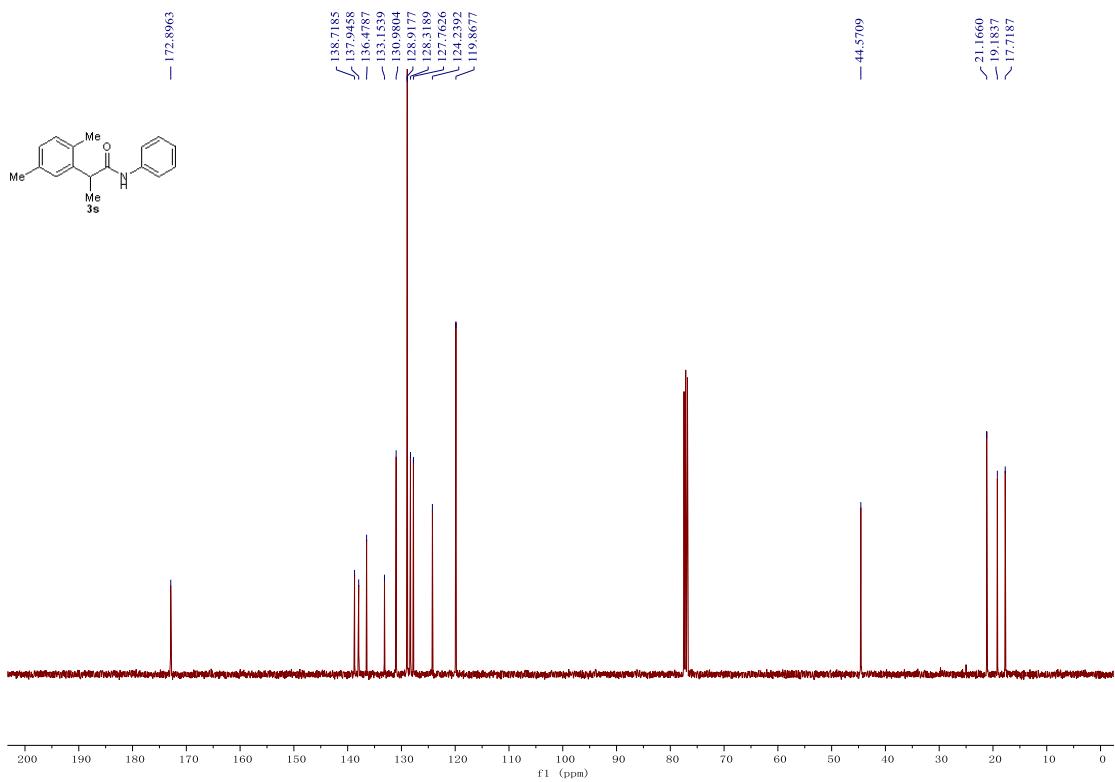
¹³C NMR spectrum of 3r



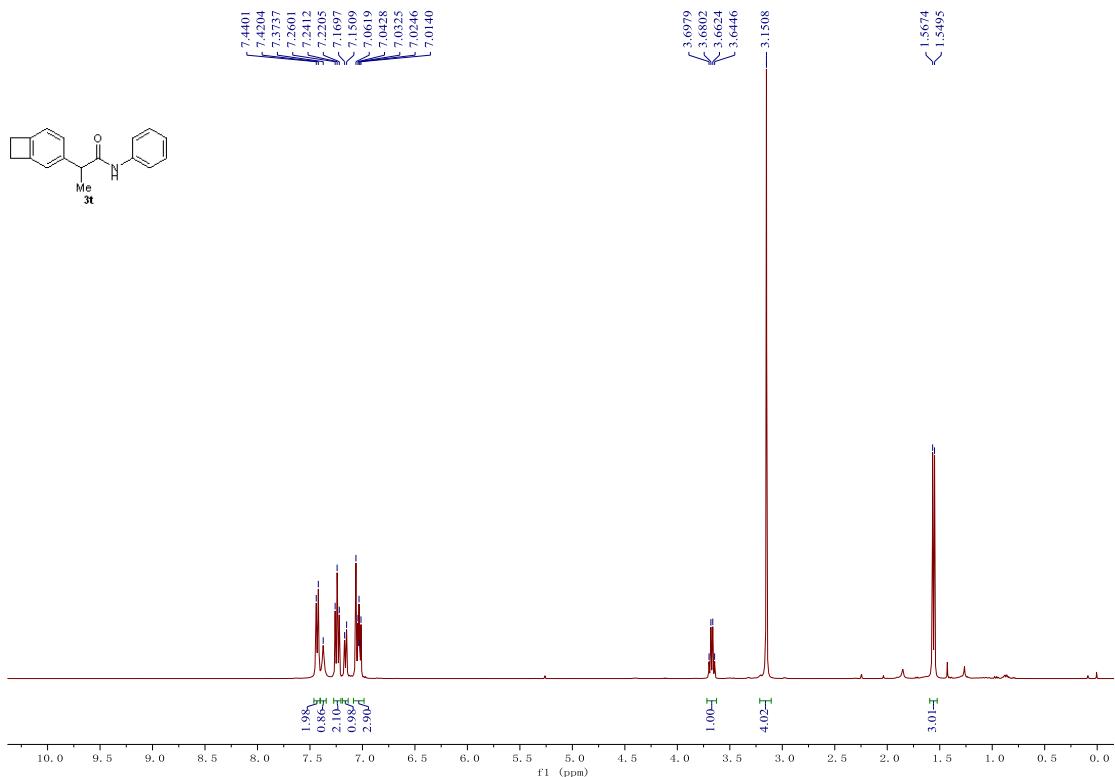
¹H NMR spectrum of 3s



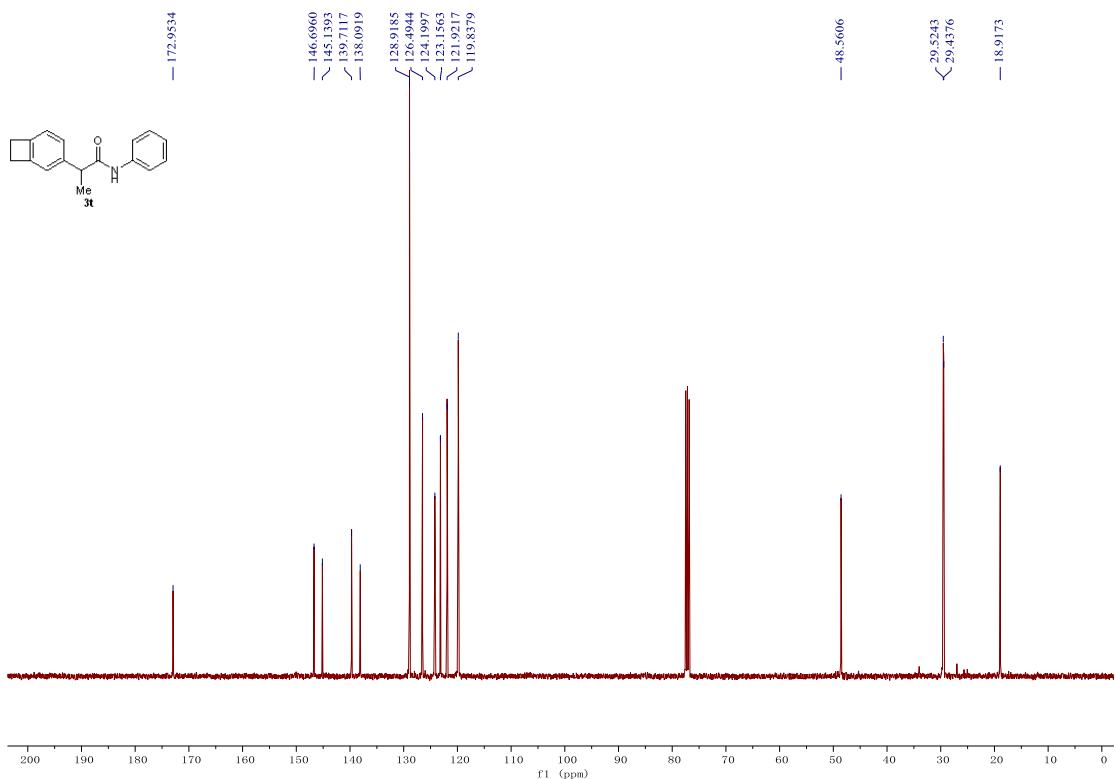
¹H NMR spectrum of **3t**



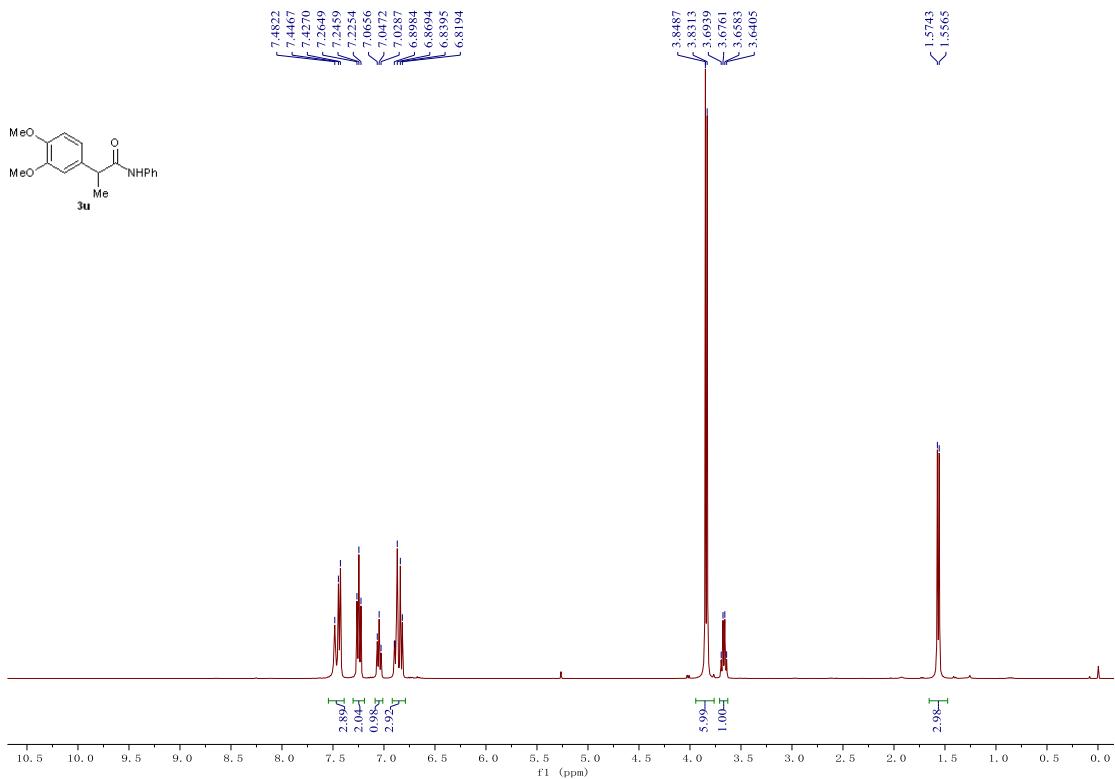
¹H NMR spectrum of **3t**



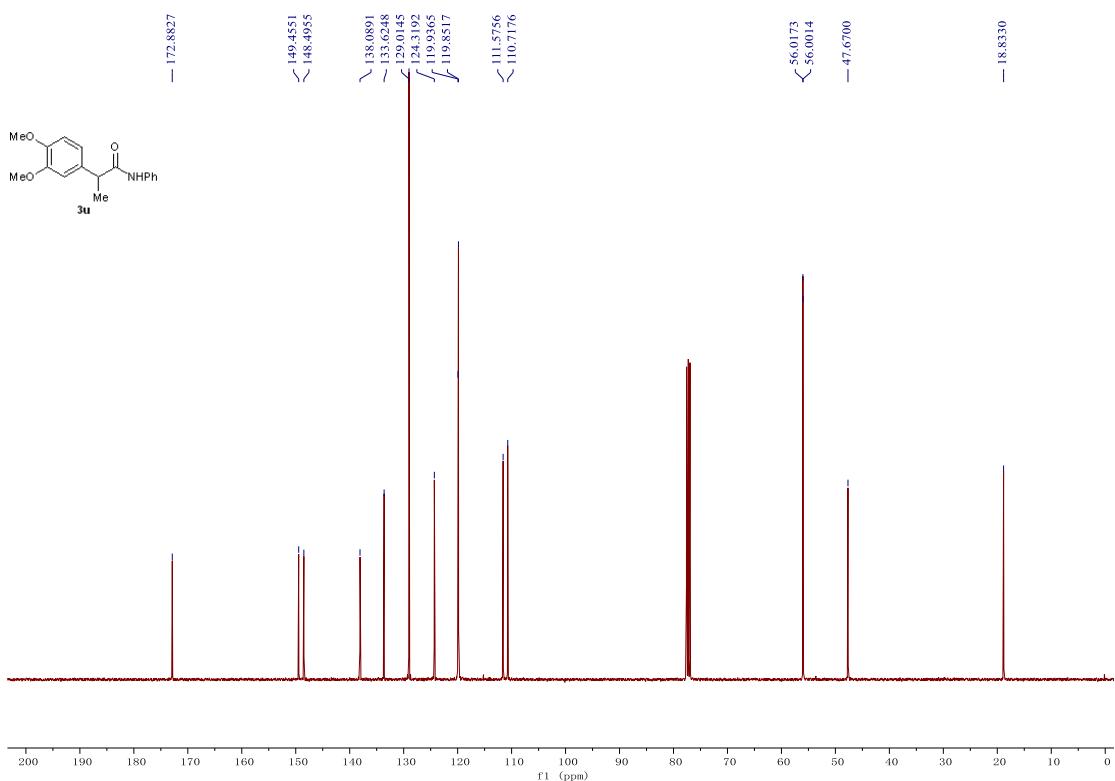
^{13}C NMR spectrum of **3t**



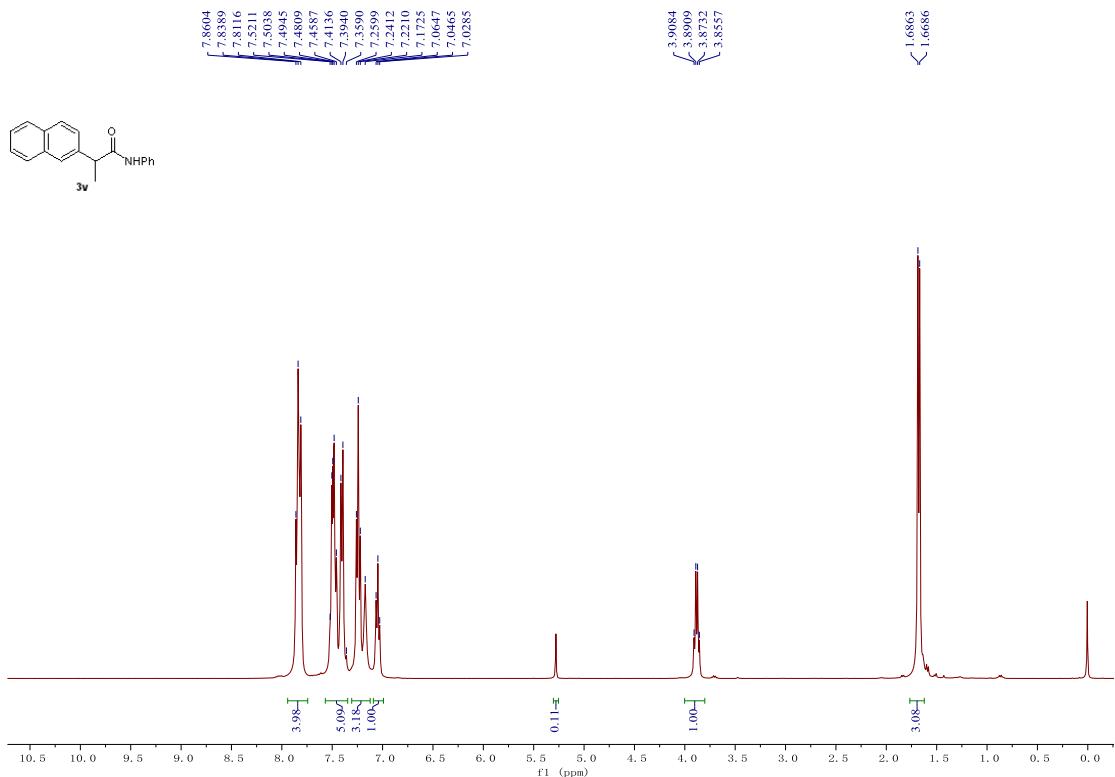
^1H NMR spectrum of **3u**



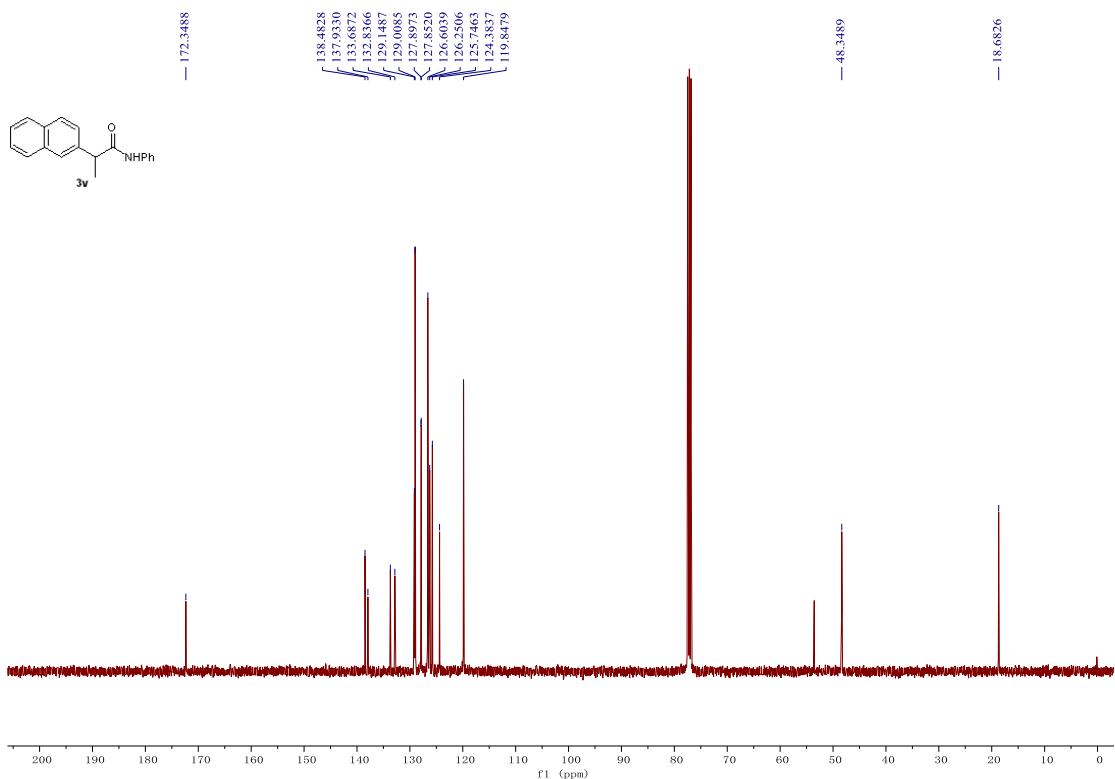
¹³C NMR spectrum of **3u**



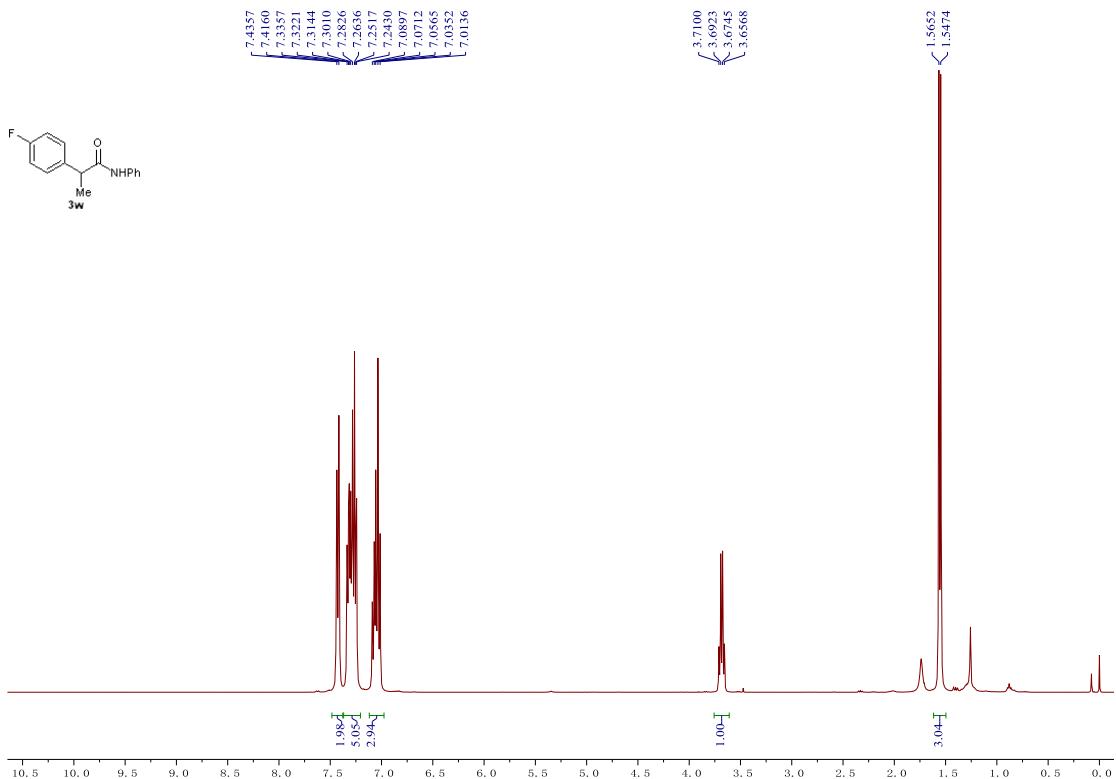
¹H NMR spectrum of **3v**



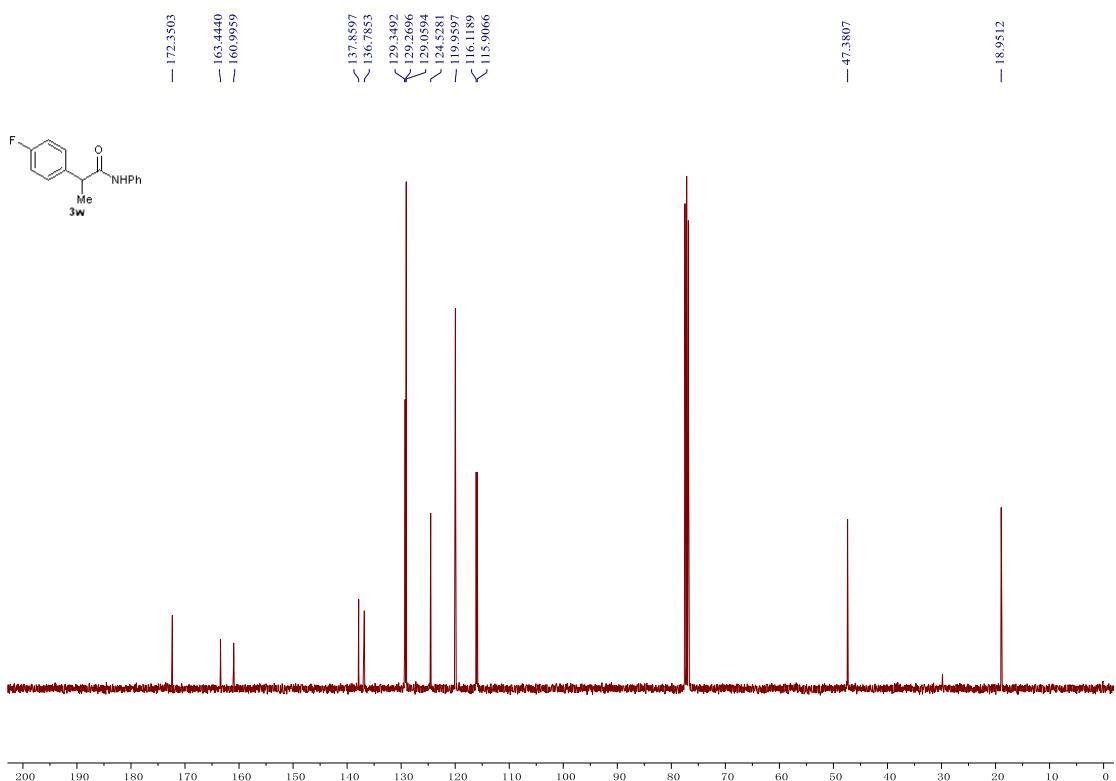
¹³C NMR spectrum of **3w**



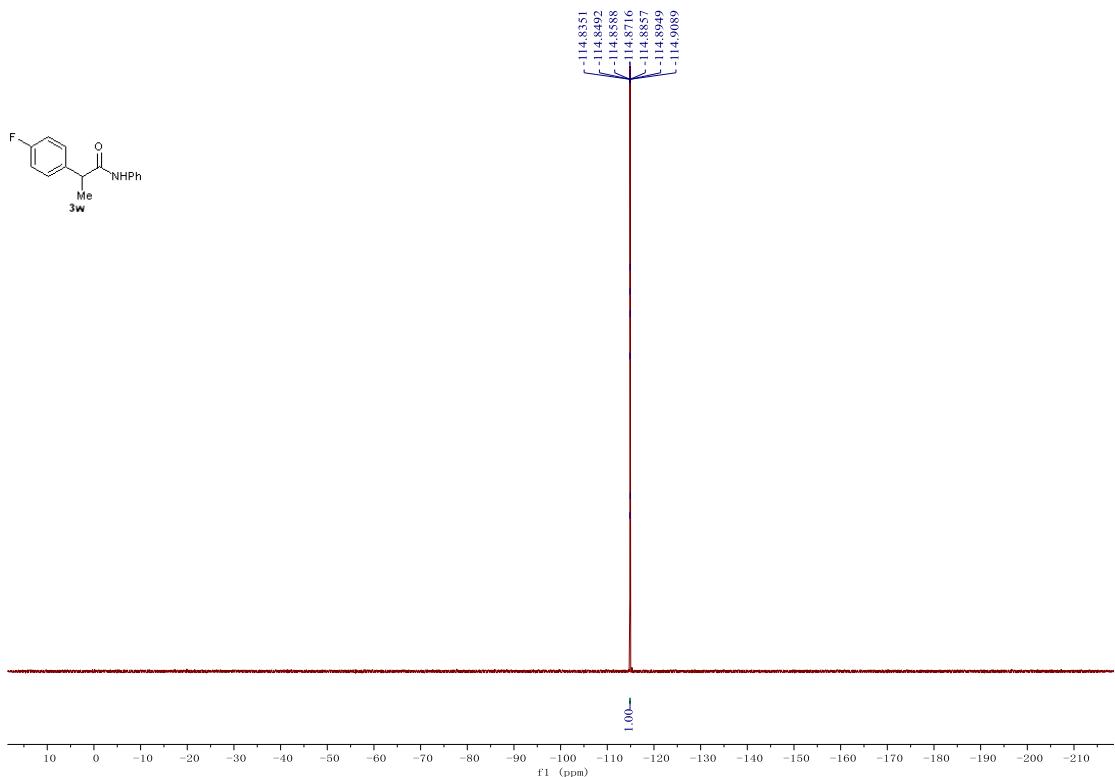
¹H NMR spectrum of **3w**



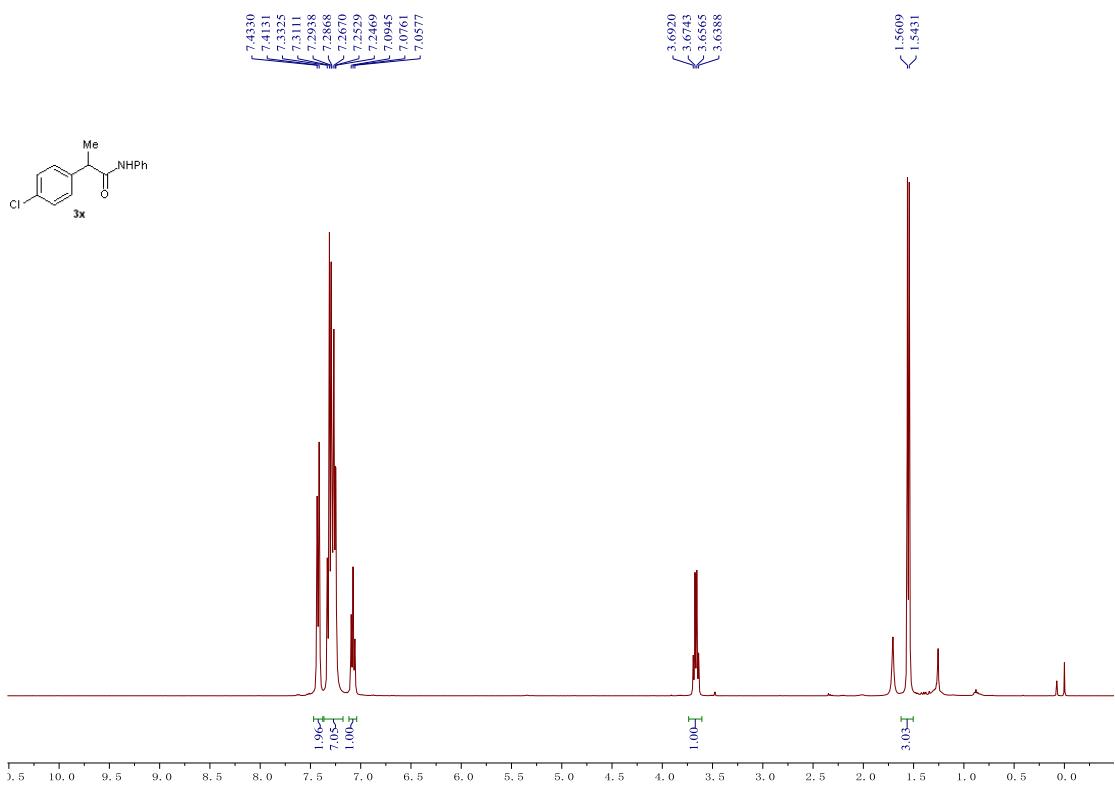
¹³C NMR spectrum of **3w**



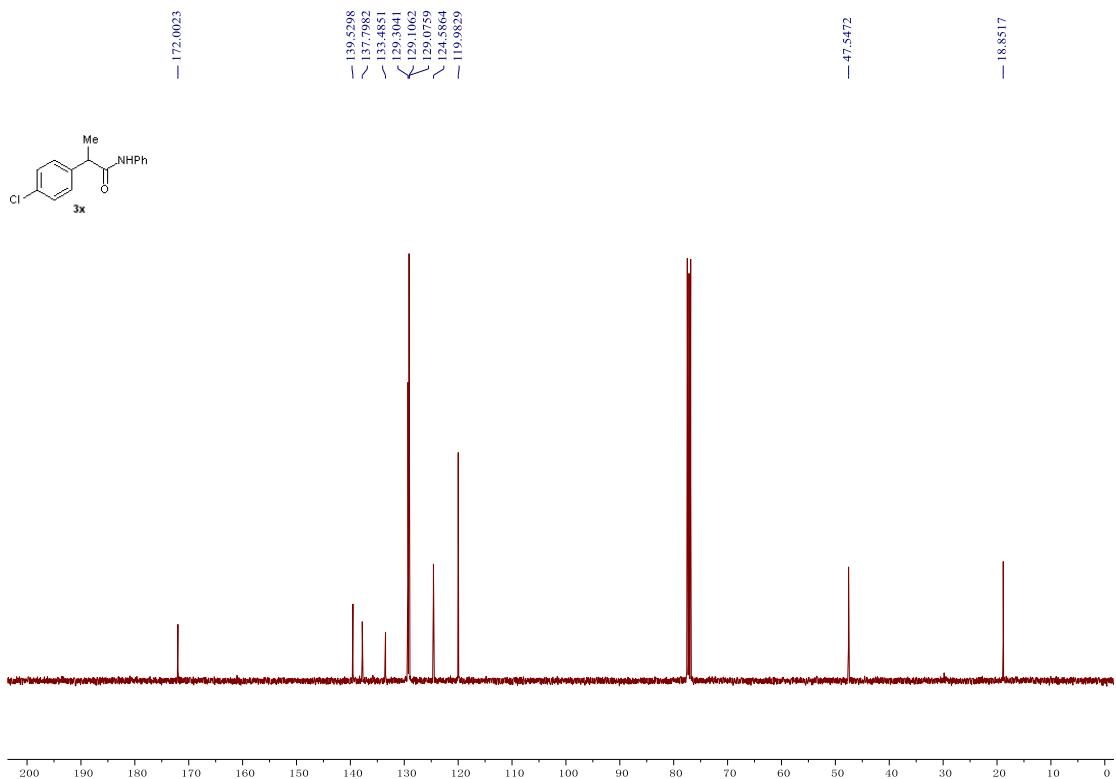
¹⁹F NMR spectrum of **3w**



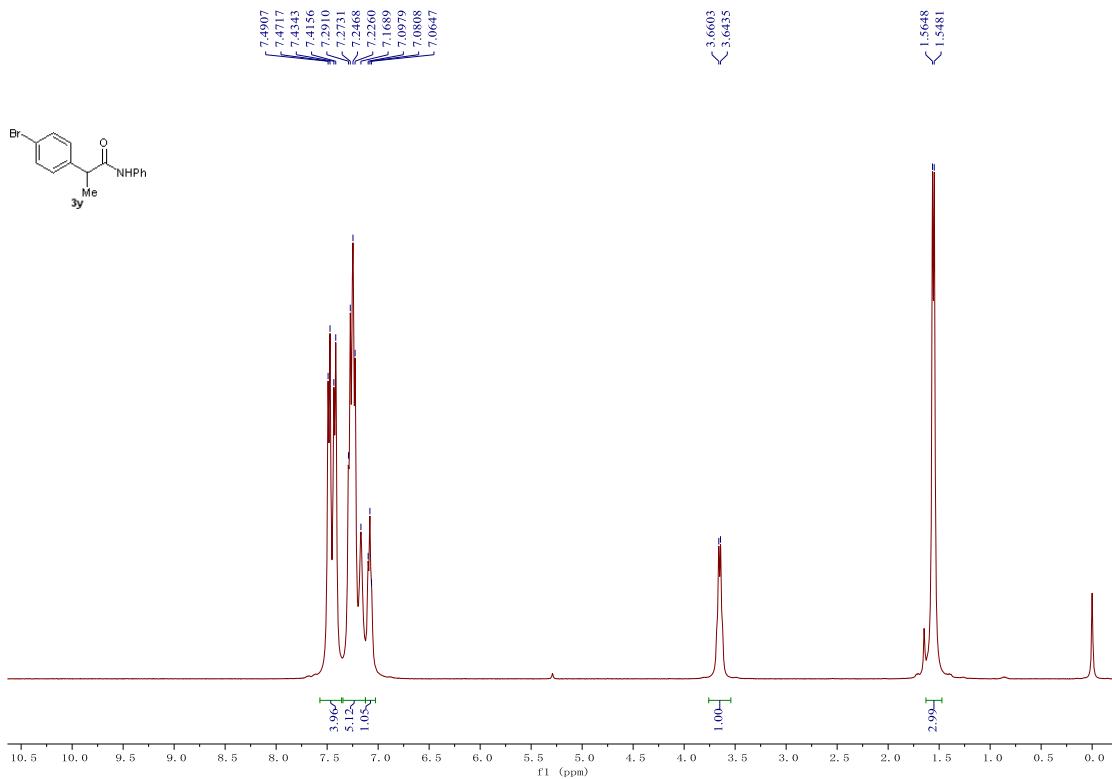
¹H NMR spectrum of **3x**



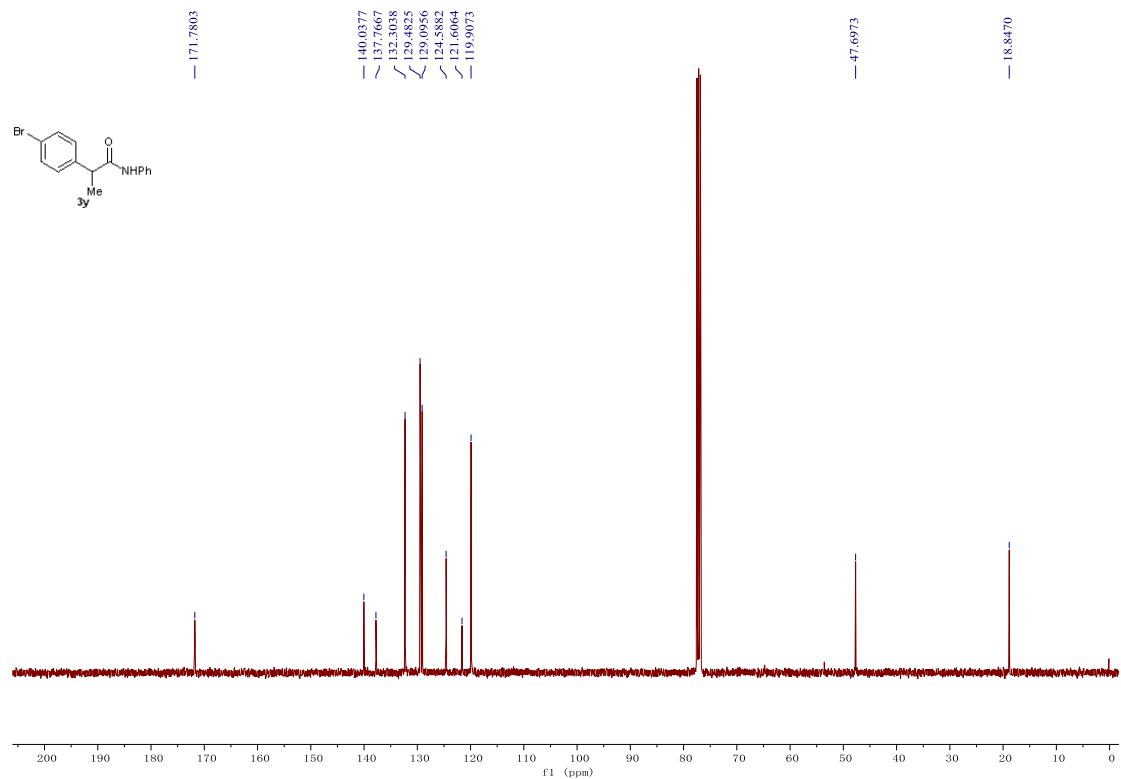
¹³C NMR spectrum of **3x**



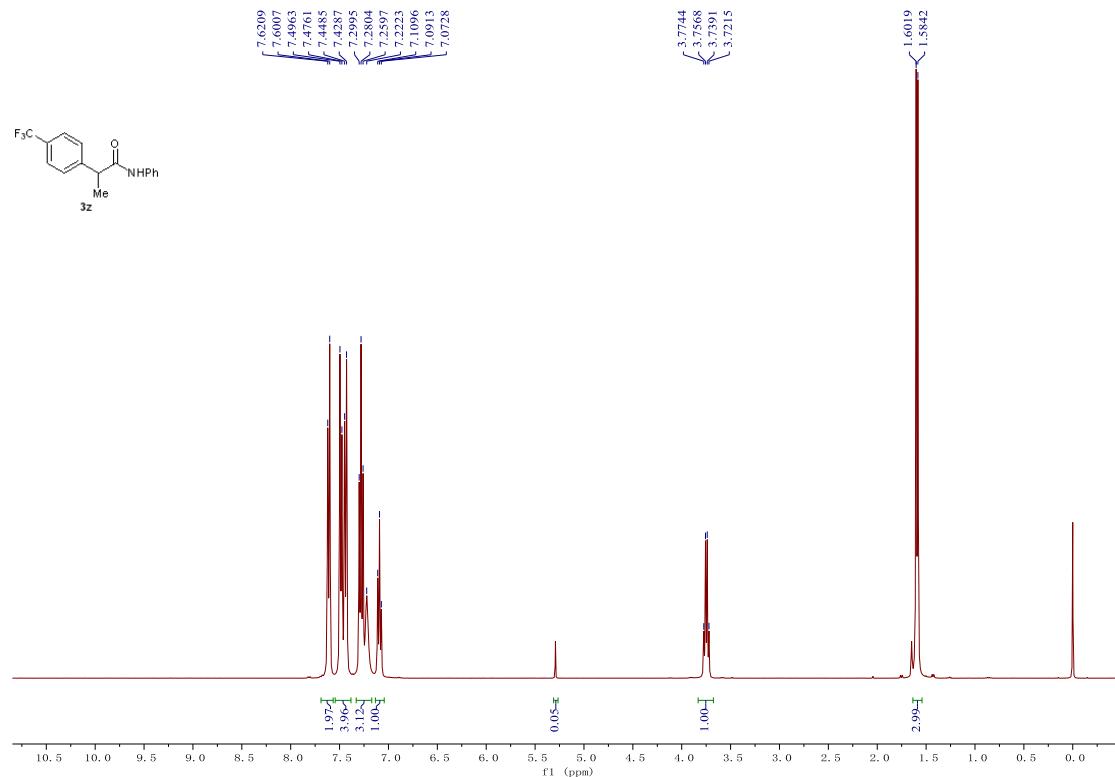
¹H NMR spectrum of **3y**



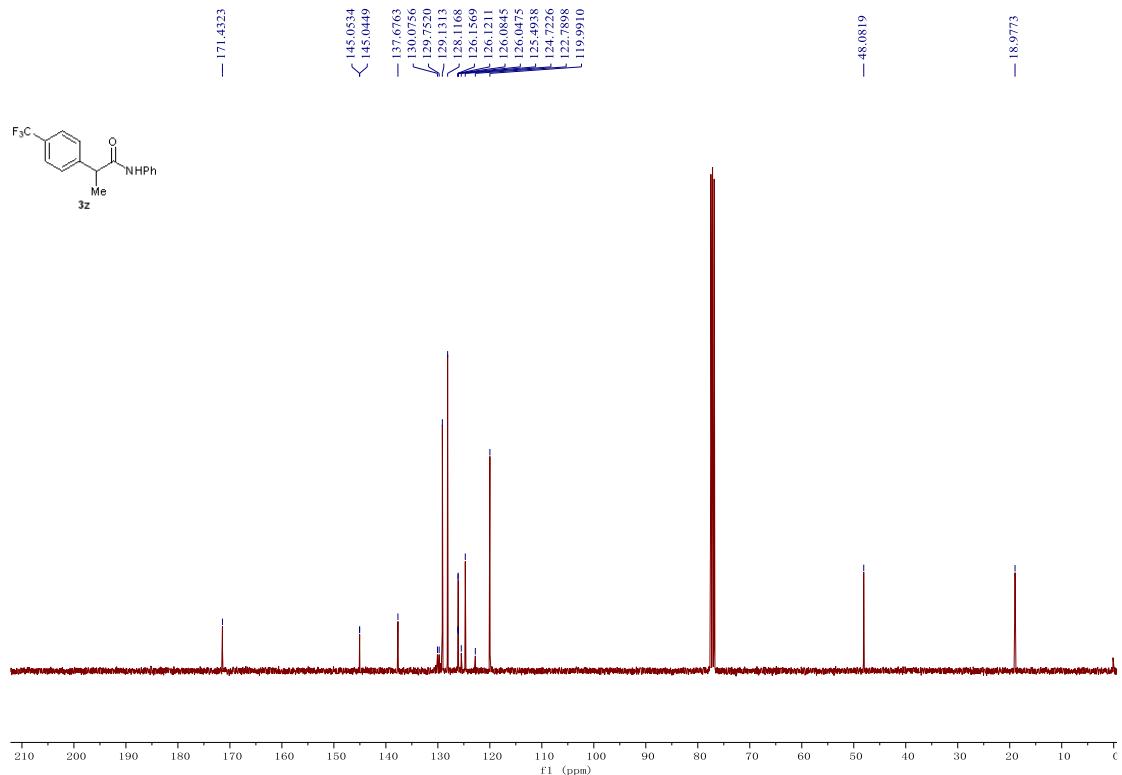
¹³C NMR spectrum of **3y**



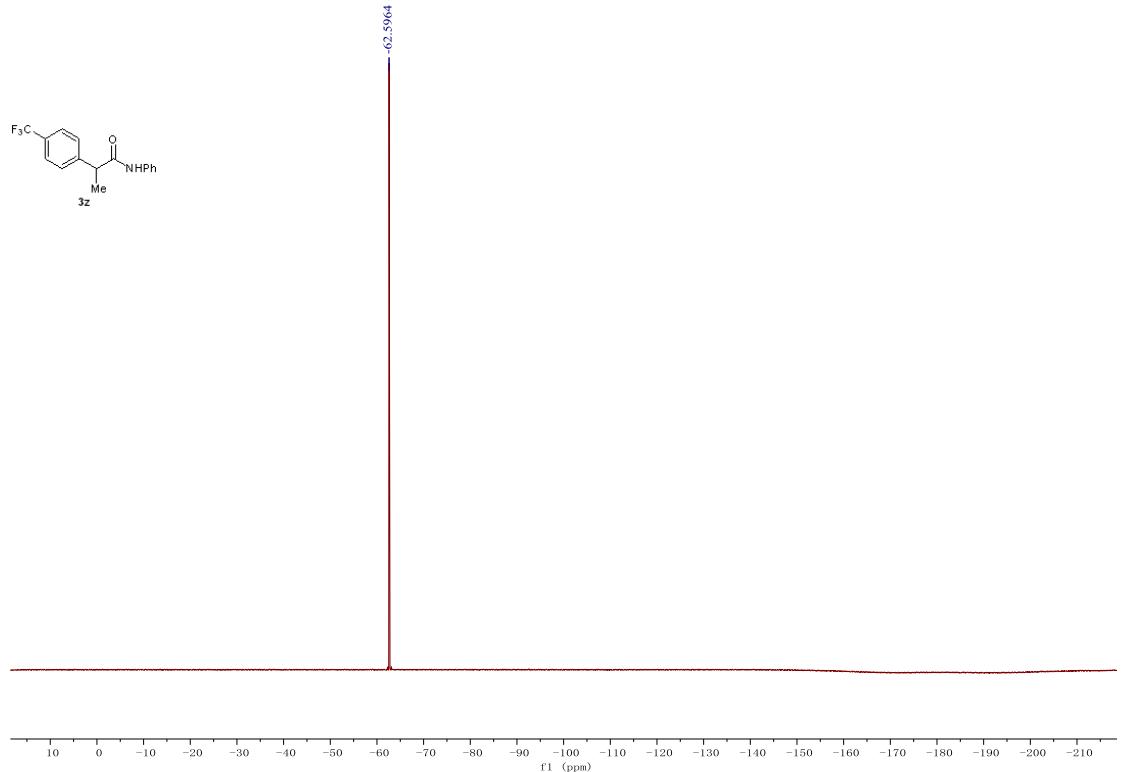
¹H NMR spectrum of **3z**



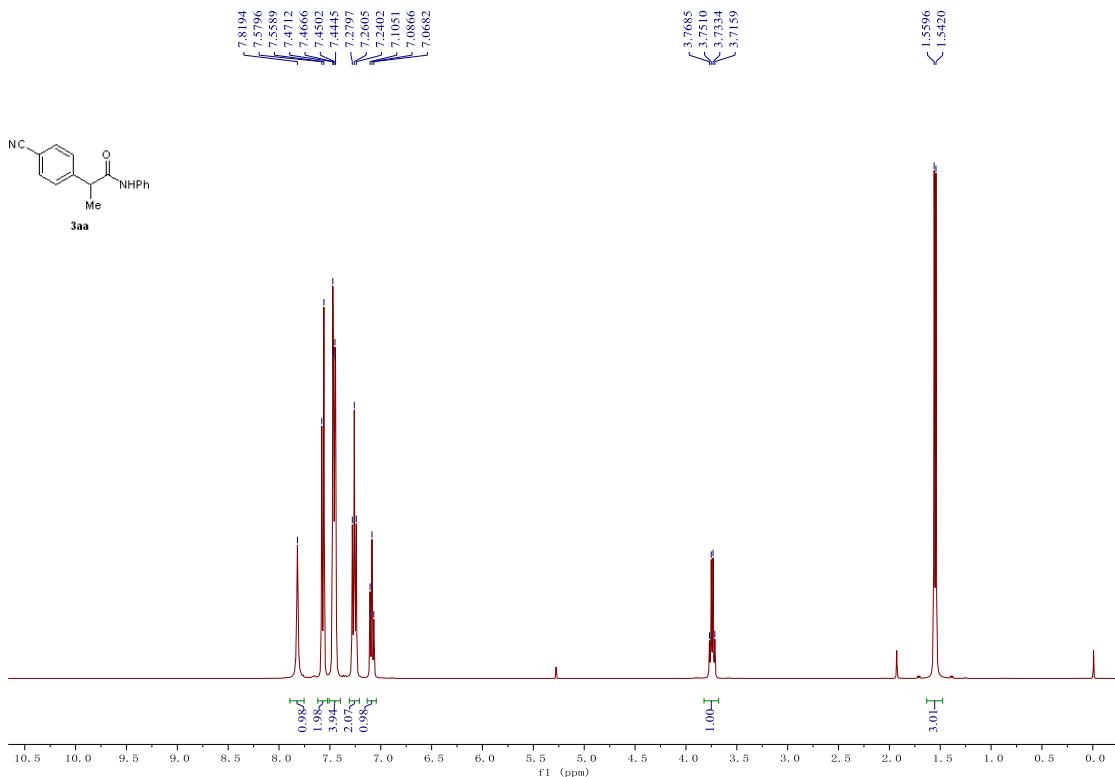
¹³C NMR spectrum of **3z**



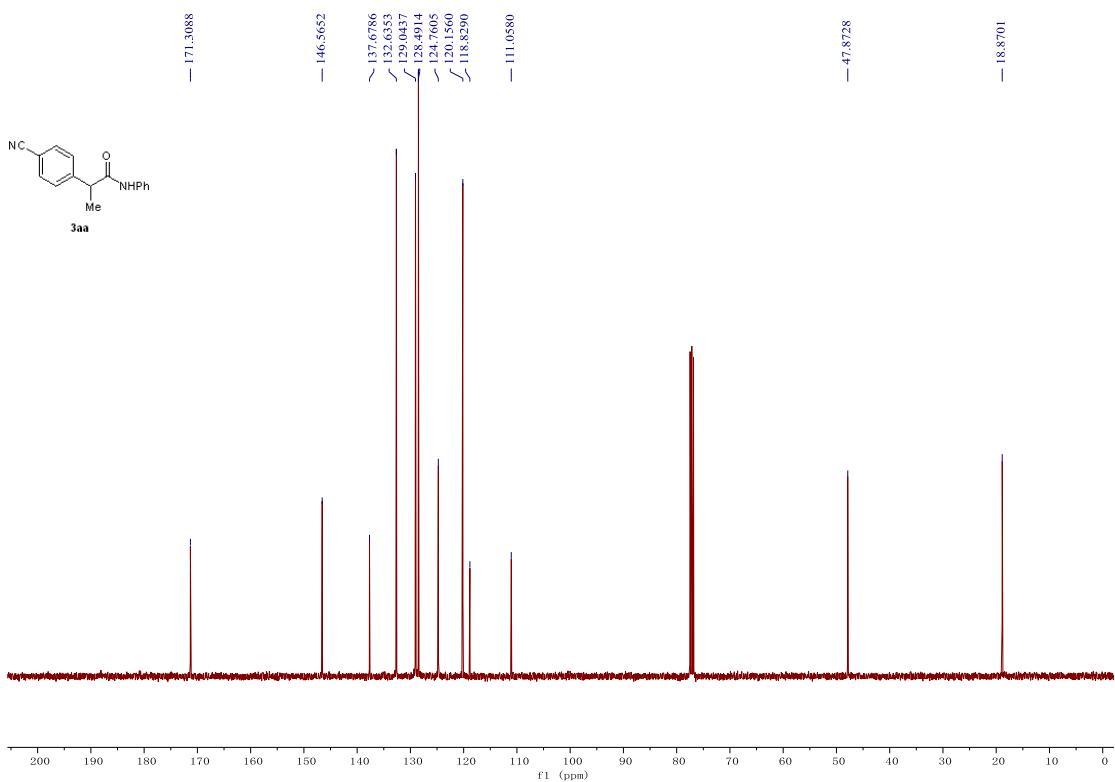
¹⁹F NMR spectrum of 3z



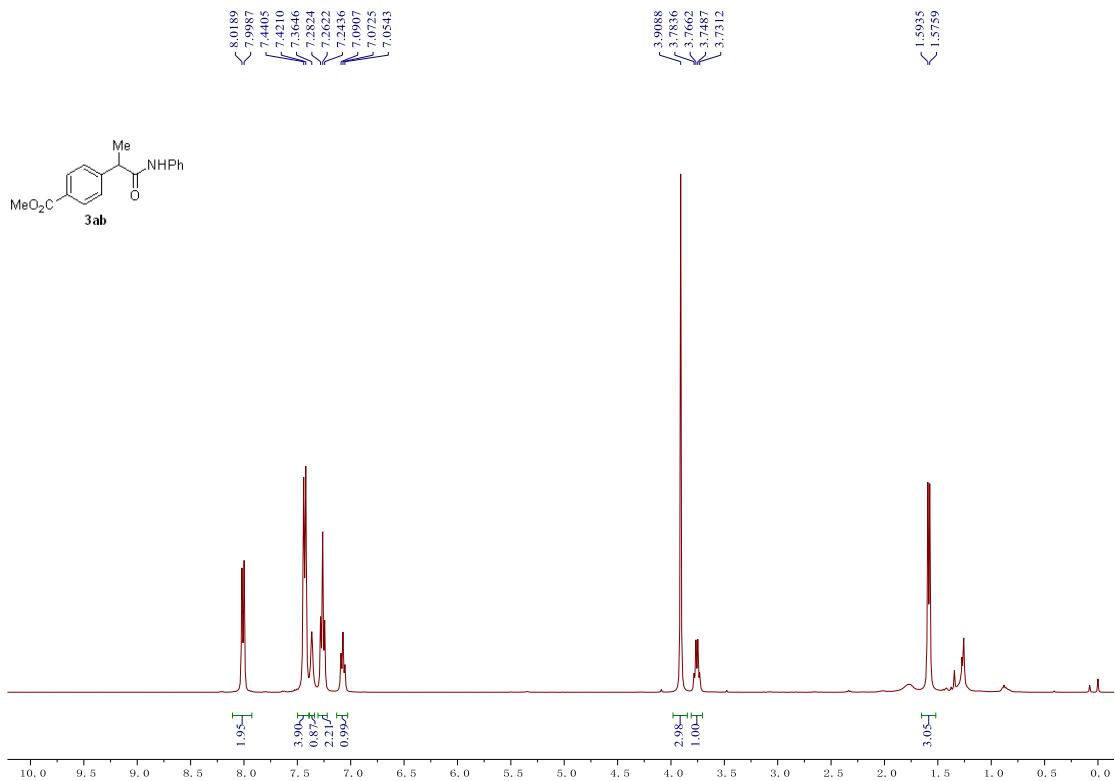
¹H NMR spectrum of **3aa**



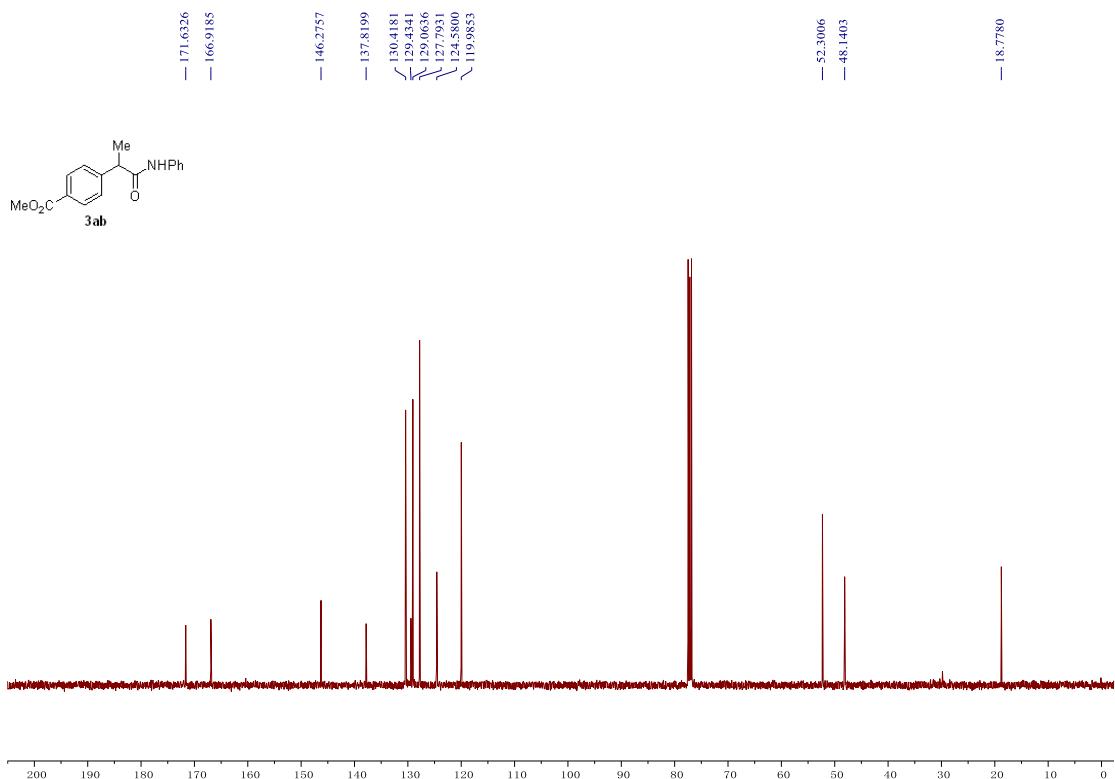
^{13}C NMR spectrum of **3aa**



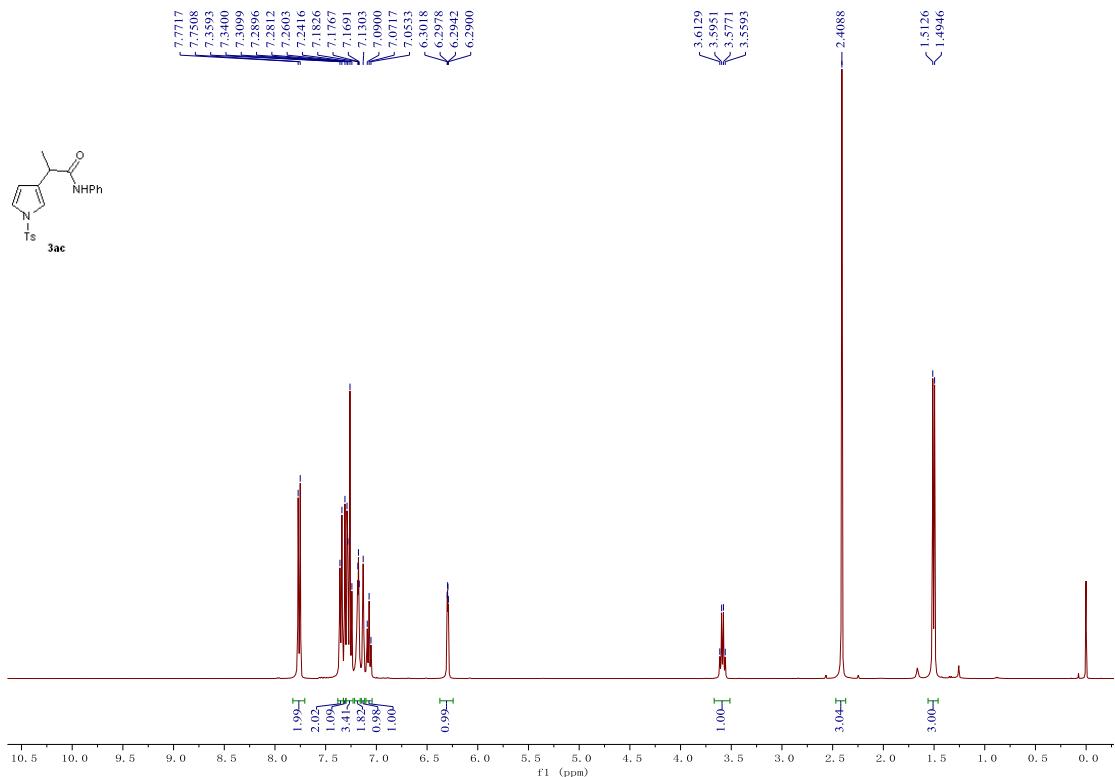
^1H NMR spectrum of **3ab**



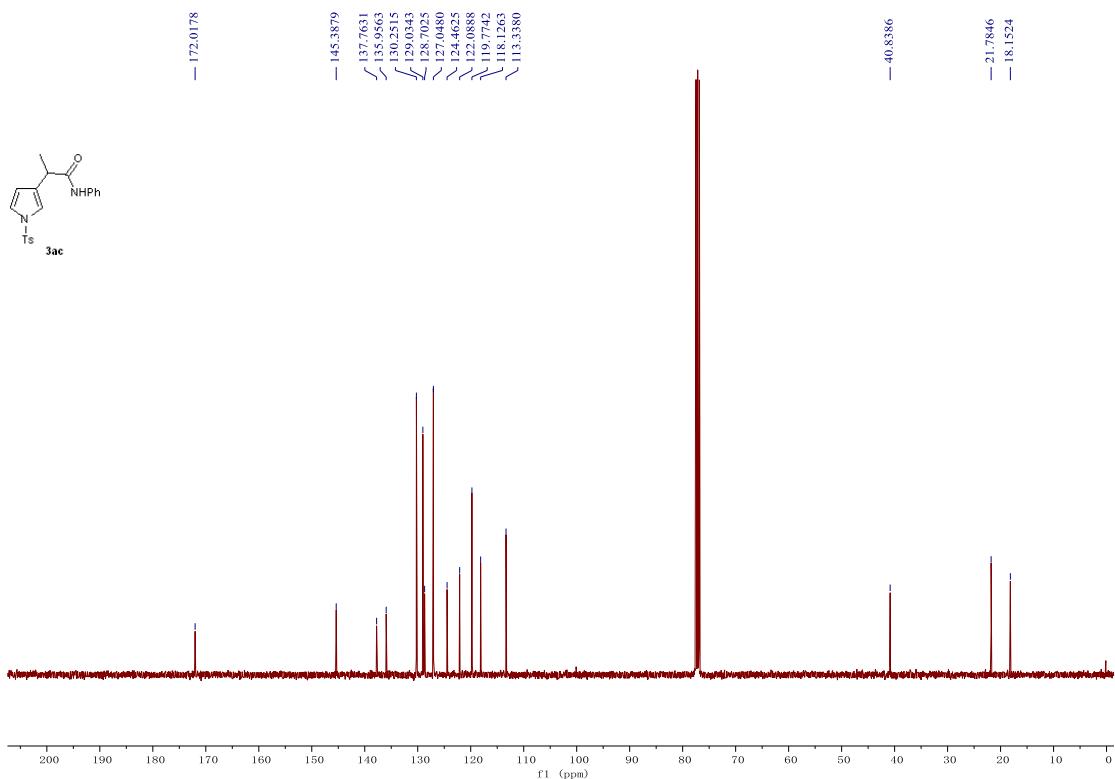
^{13}C NMR spectrum of 3ab



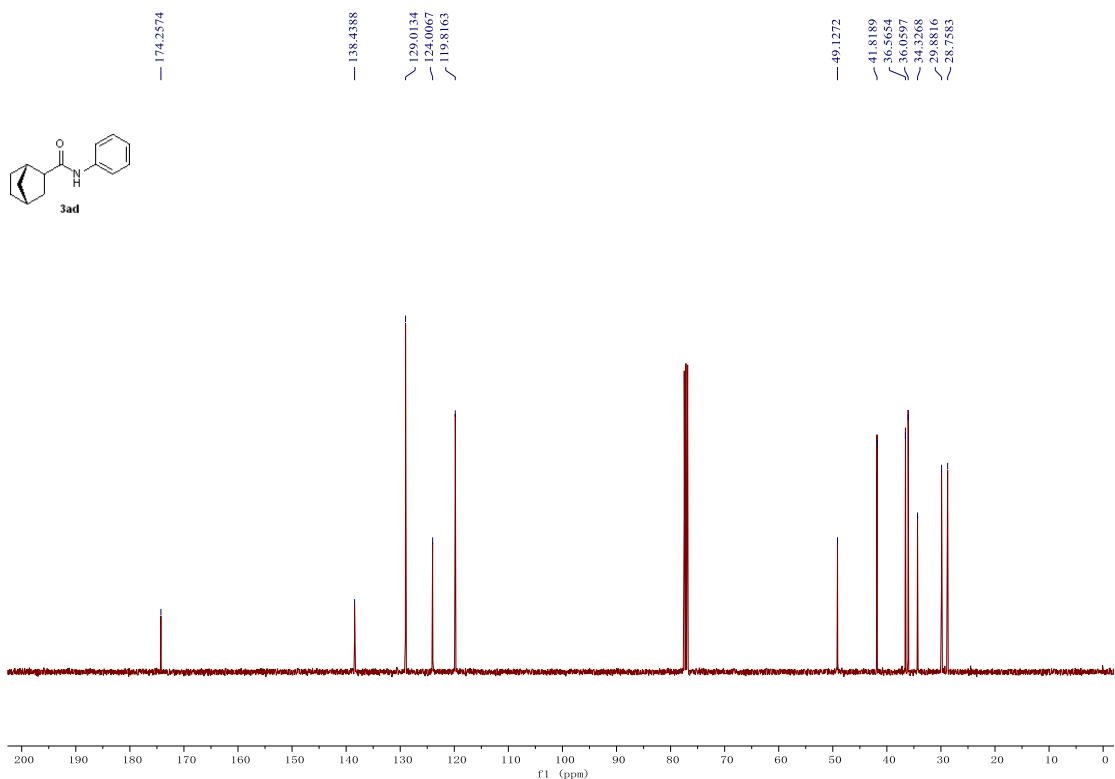
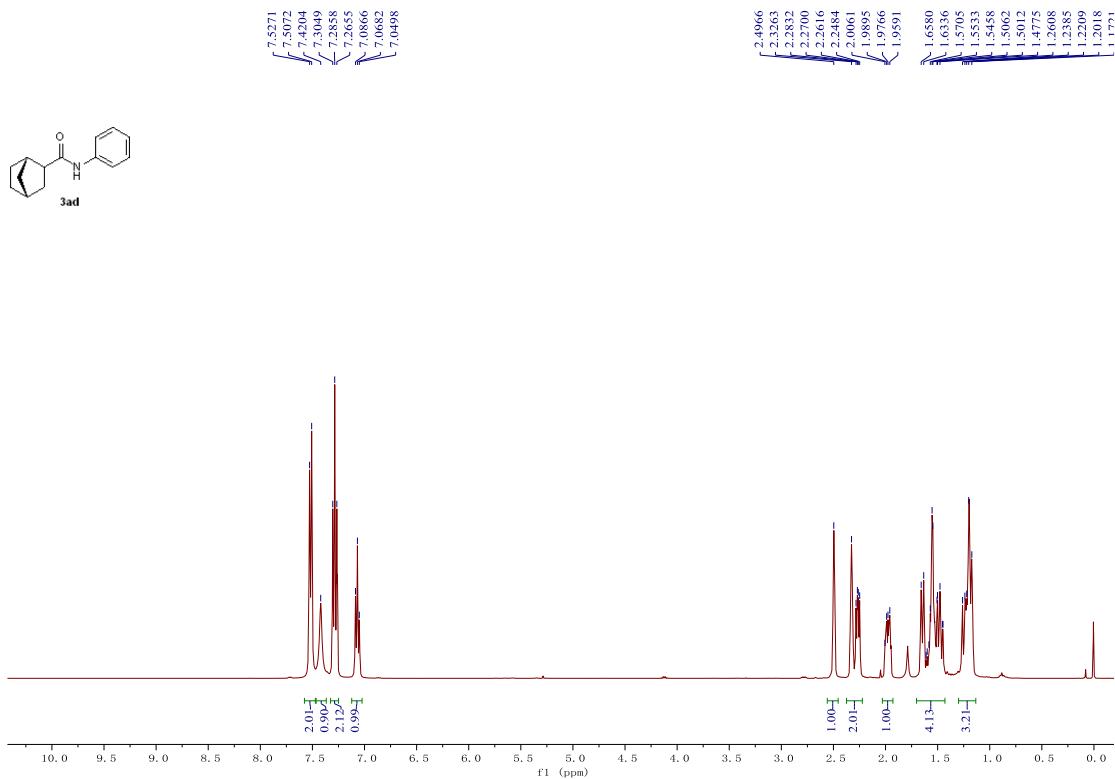
^1H NMR spectrum of 3ac



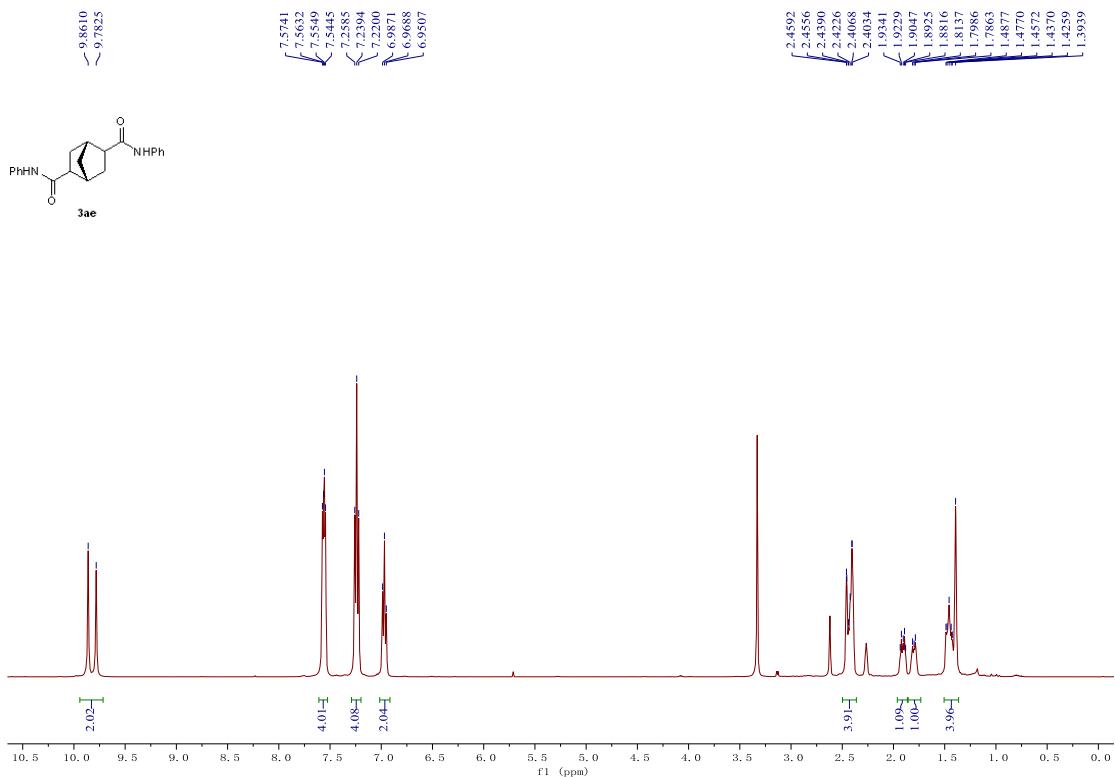
¹³C NMR spectrum of **3ac**



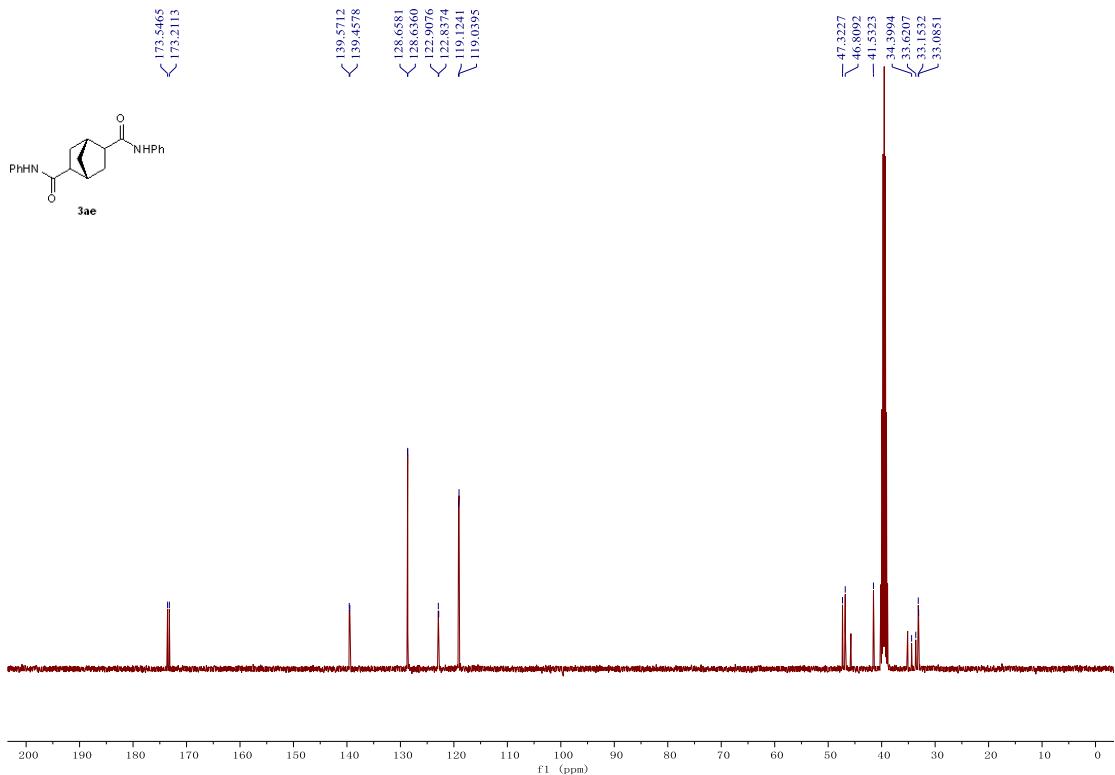
¹H NMR spectrum of **3ad**



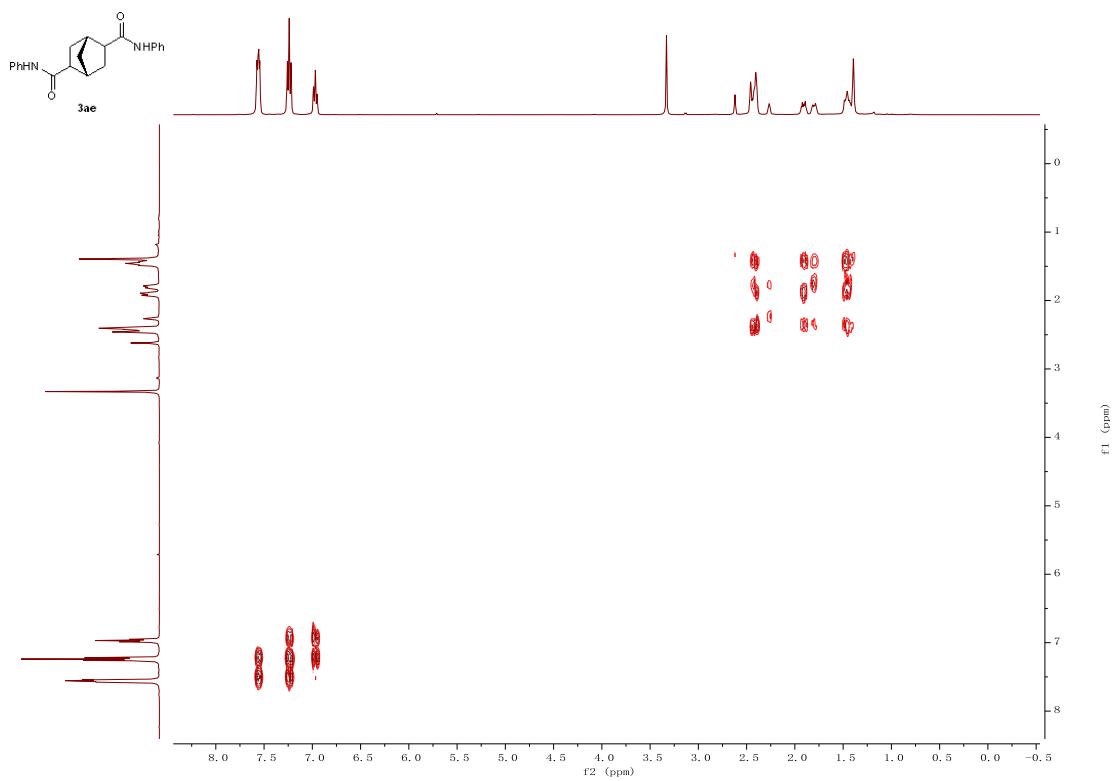
^1H NMR spectrum of **3ae**



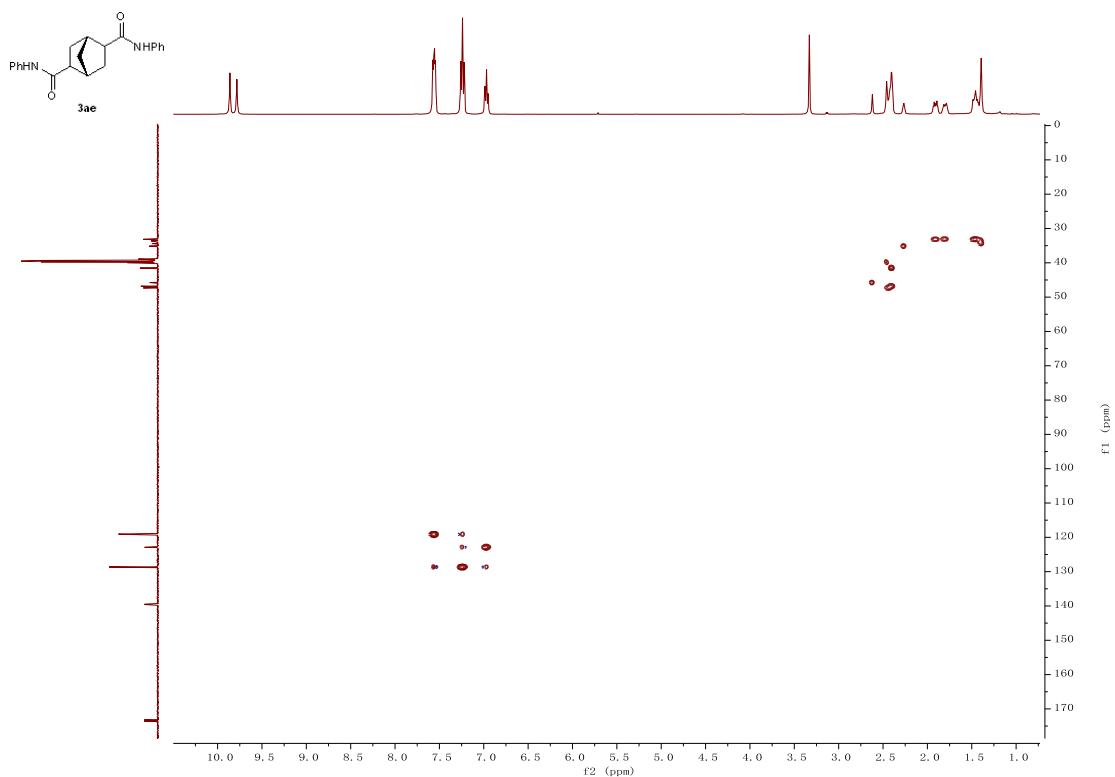
¹³C NMR spectrum of **3ae**



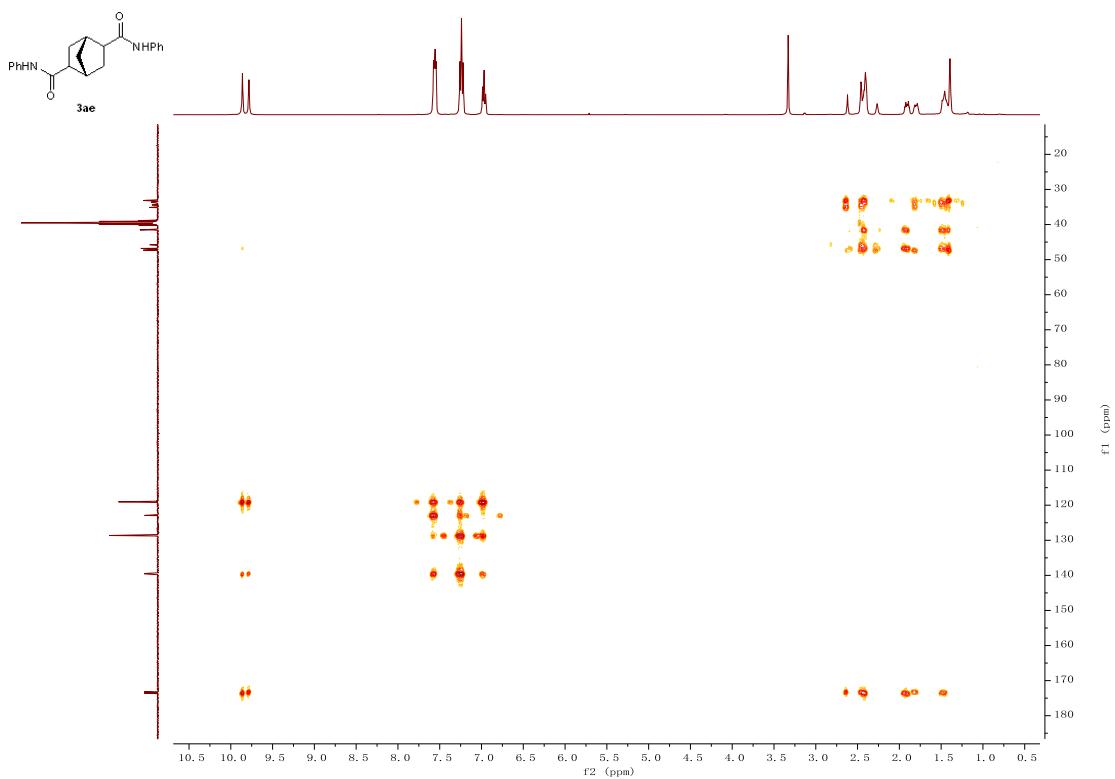
COSY spectrum of **3ae**



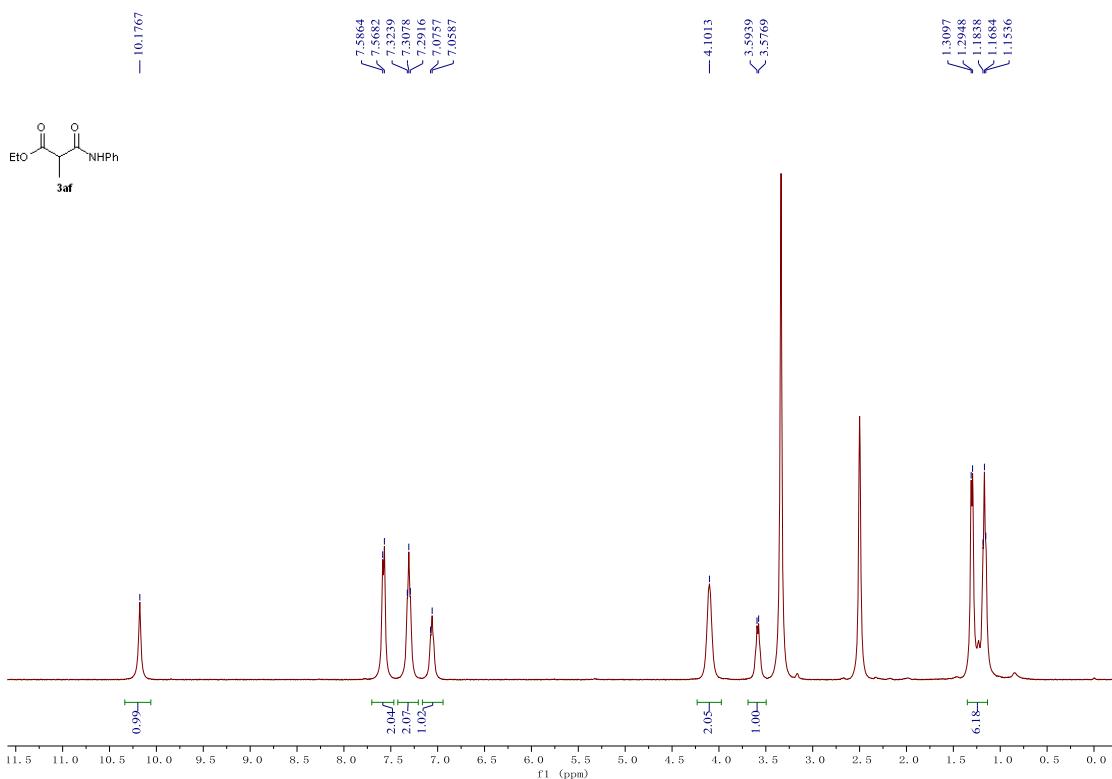
HSQC spectrum of **3ae**



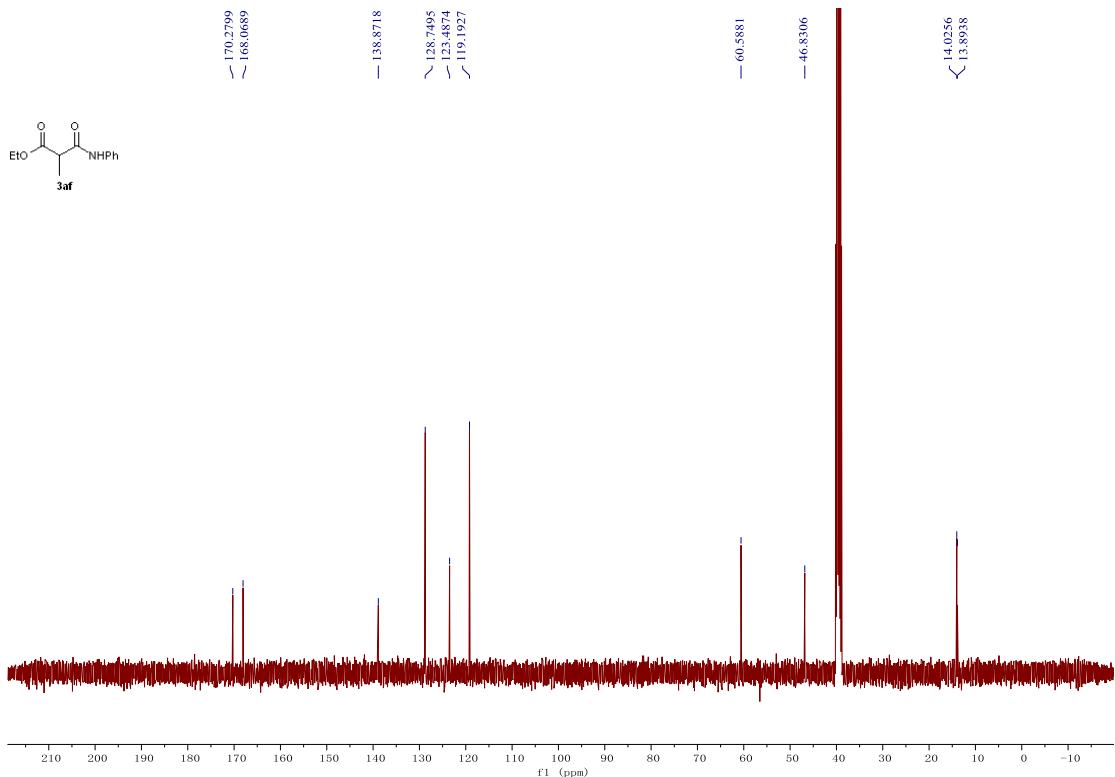
HMBC spectrum of **3ae**



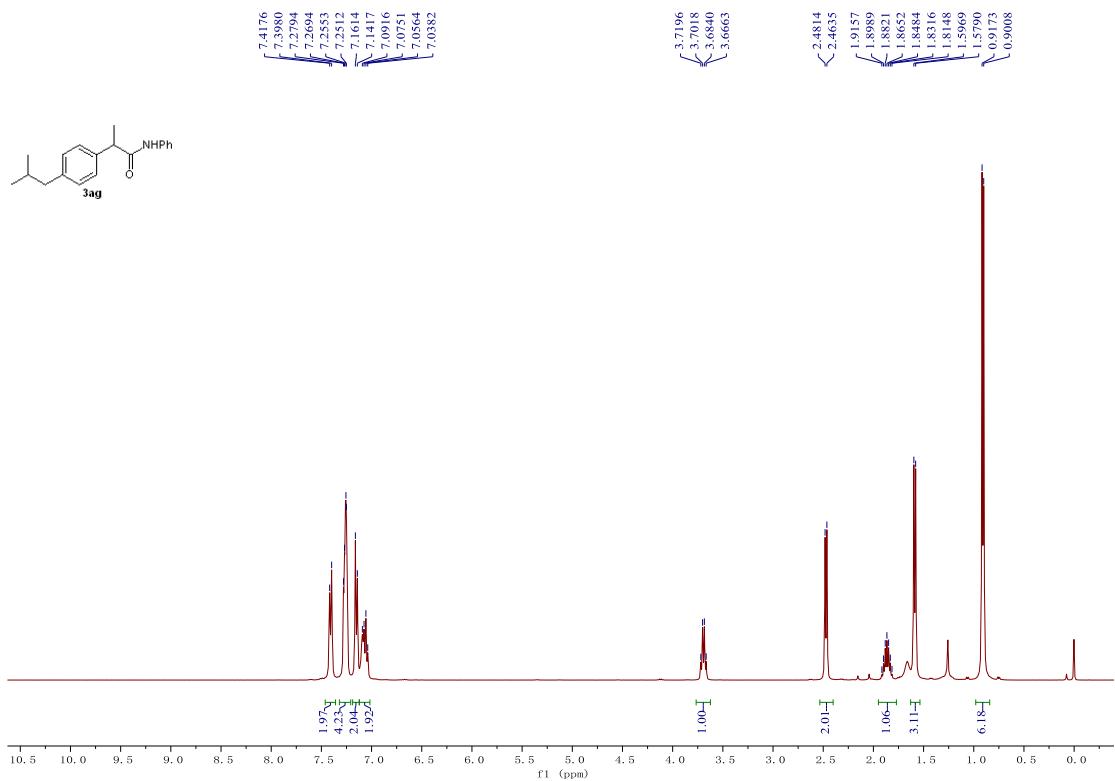
¹H NMR spectrum of 3af



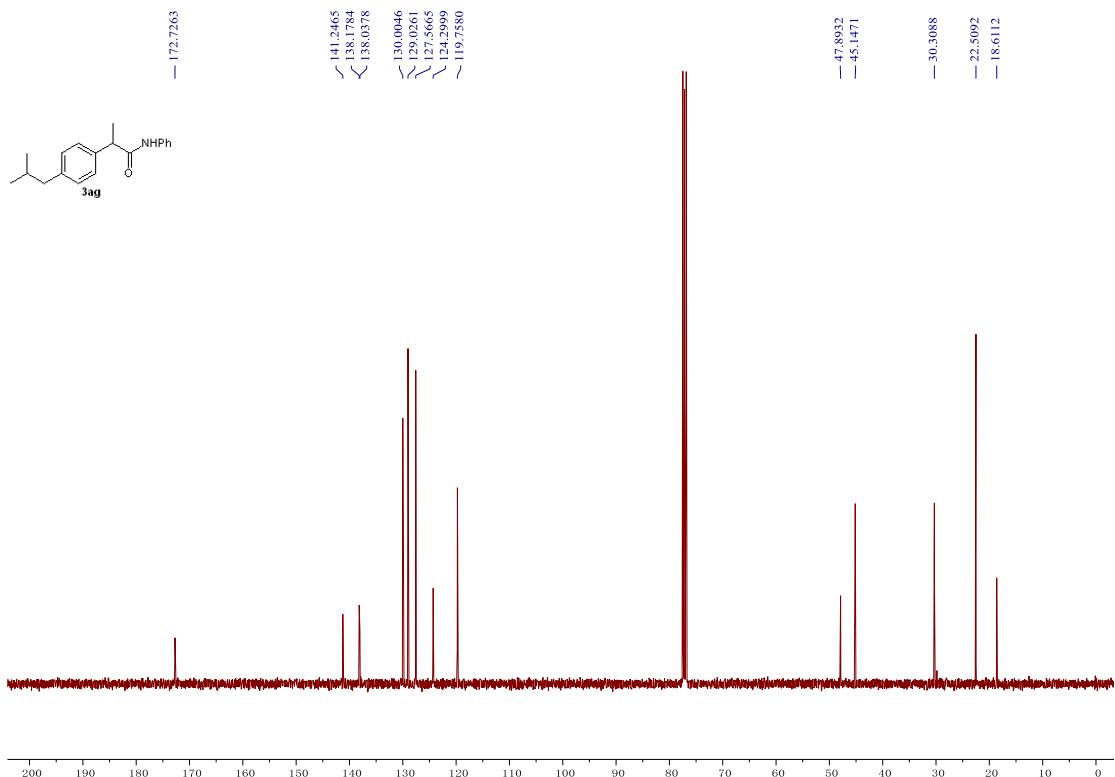
¹³C NMR spectrum of 3af



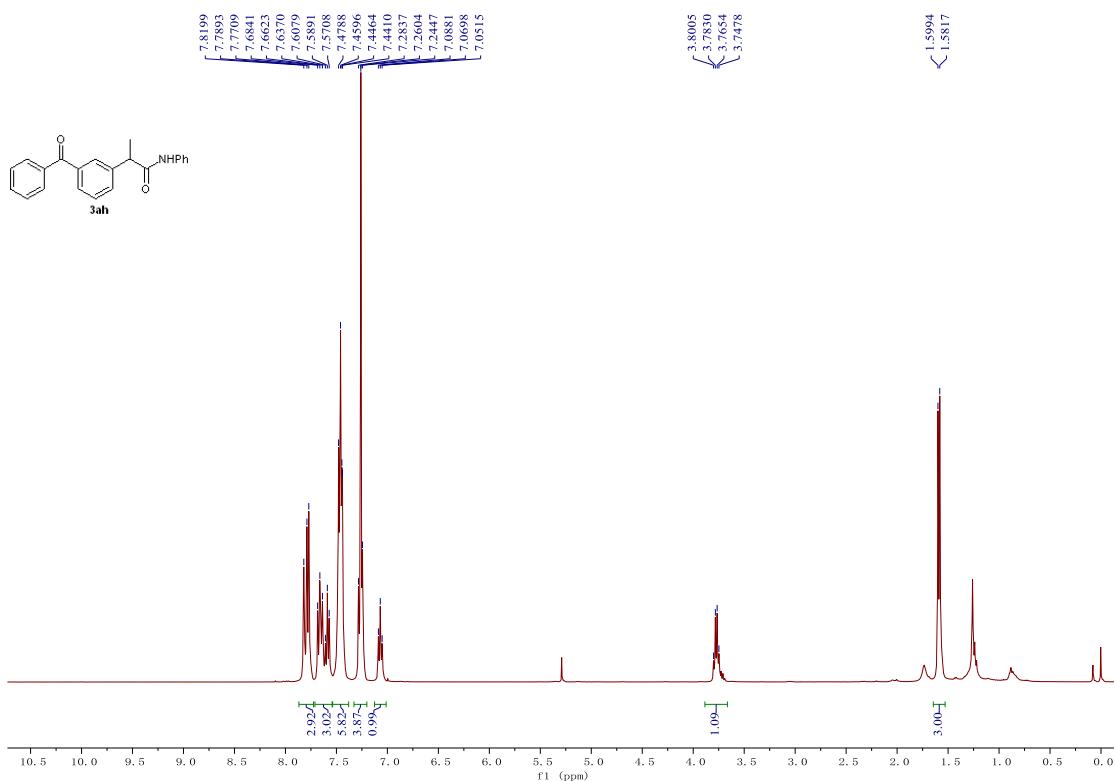
^1H NMR spectrum of **3ag**



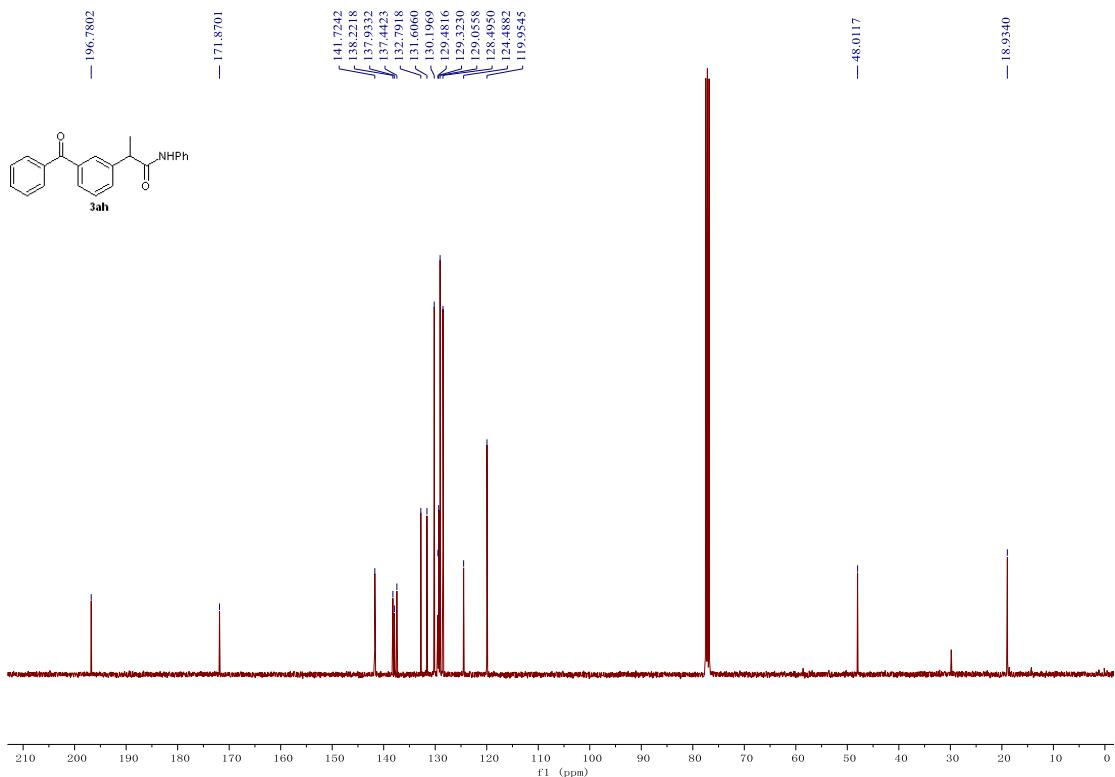
^{13}C NMR spectrum of **3ag**



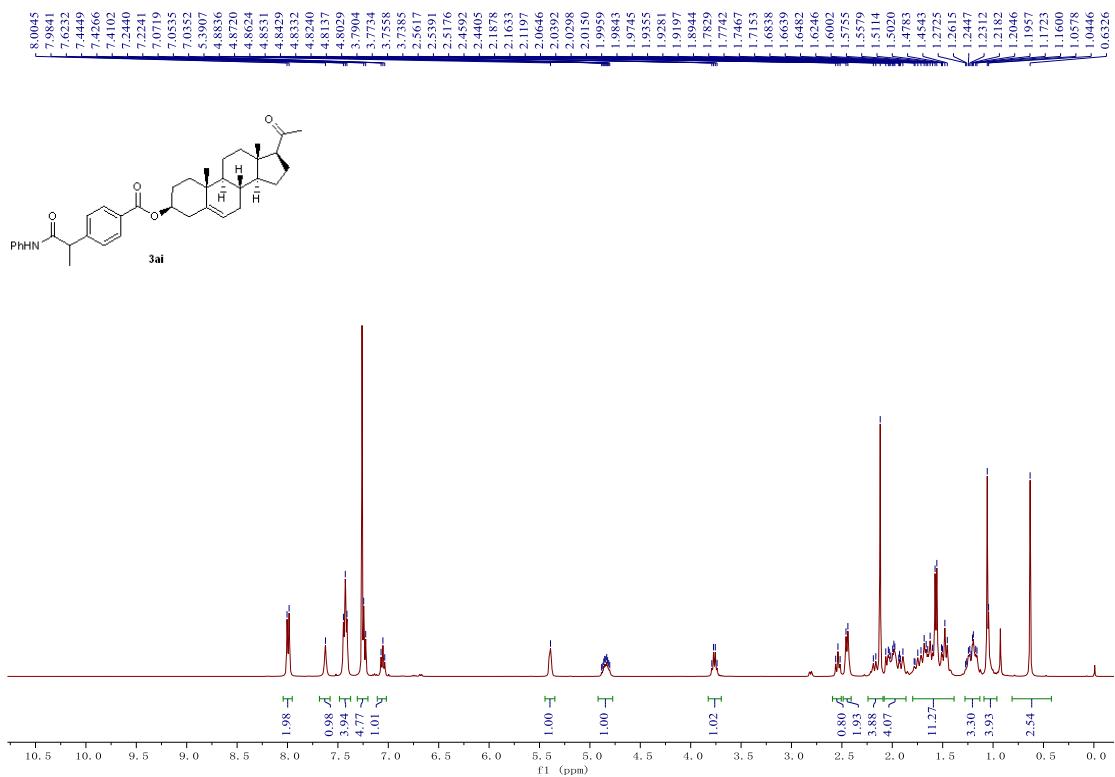
¹H NMR spectrum of **3ah**



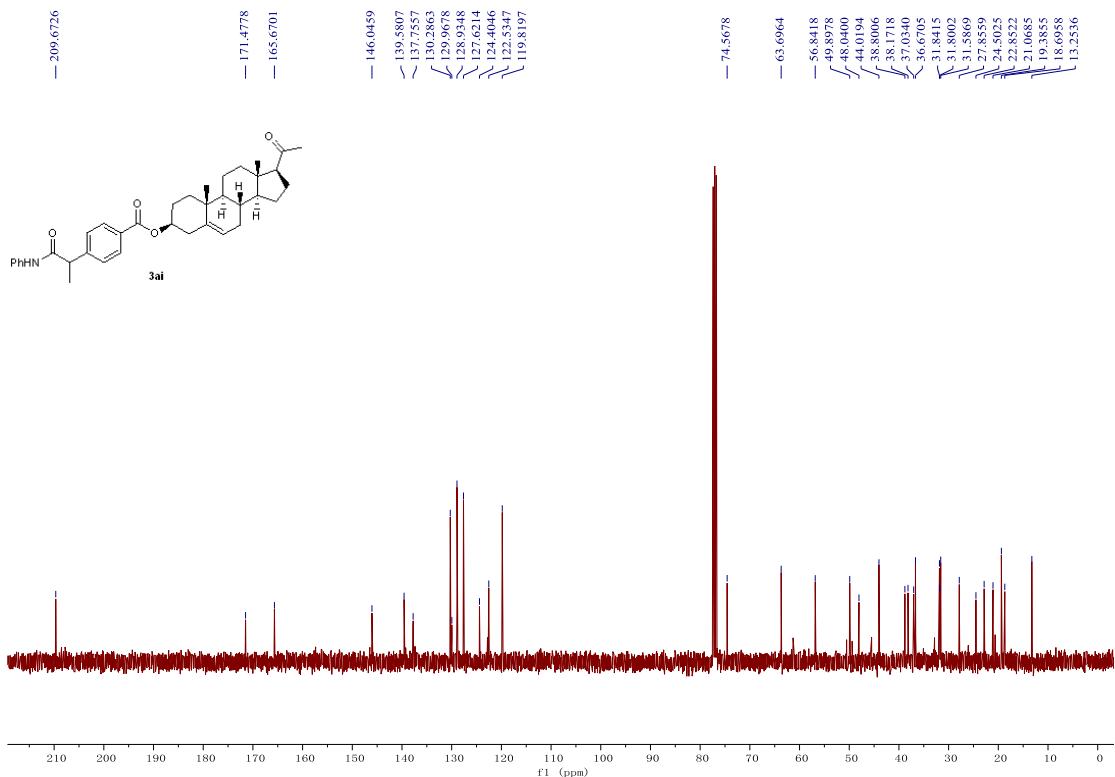
¹³C NMR spectrum of **3ah**



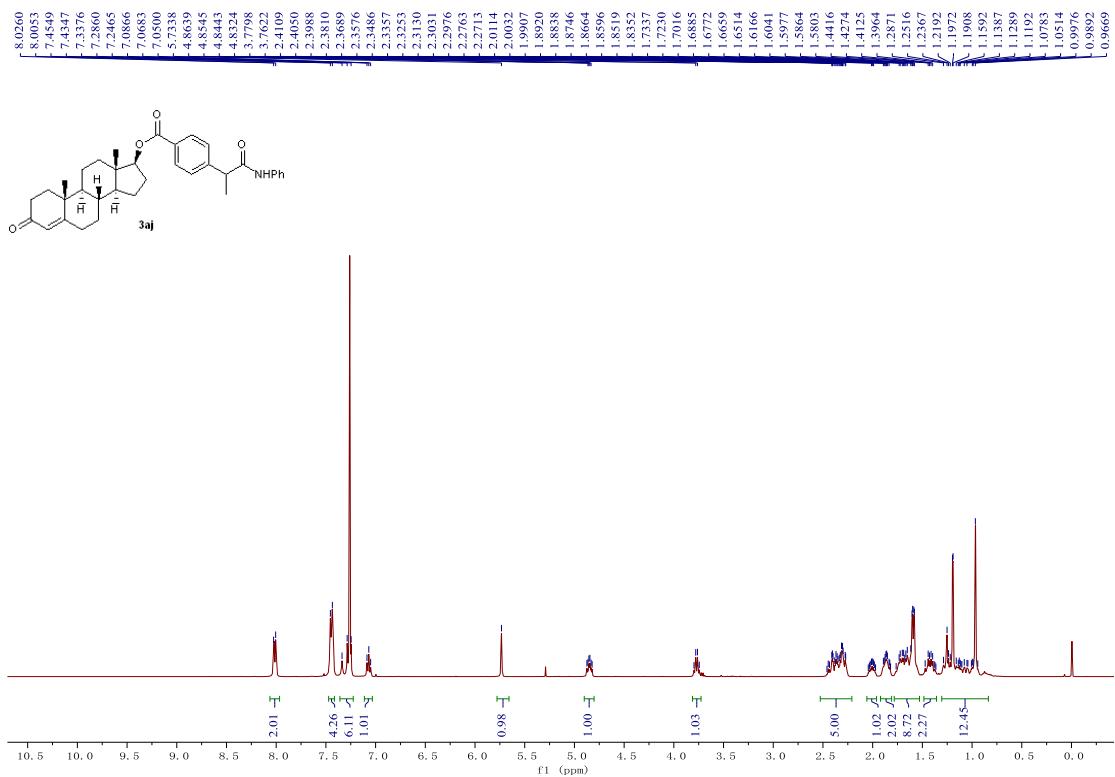
¹H NMR spectrum of **3ai**



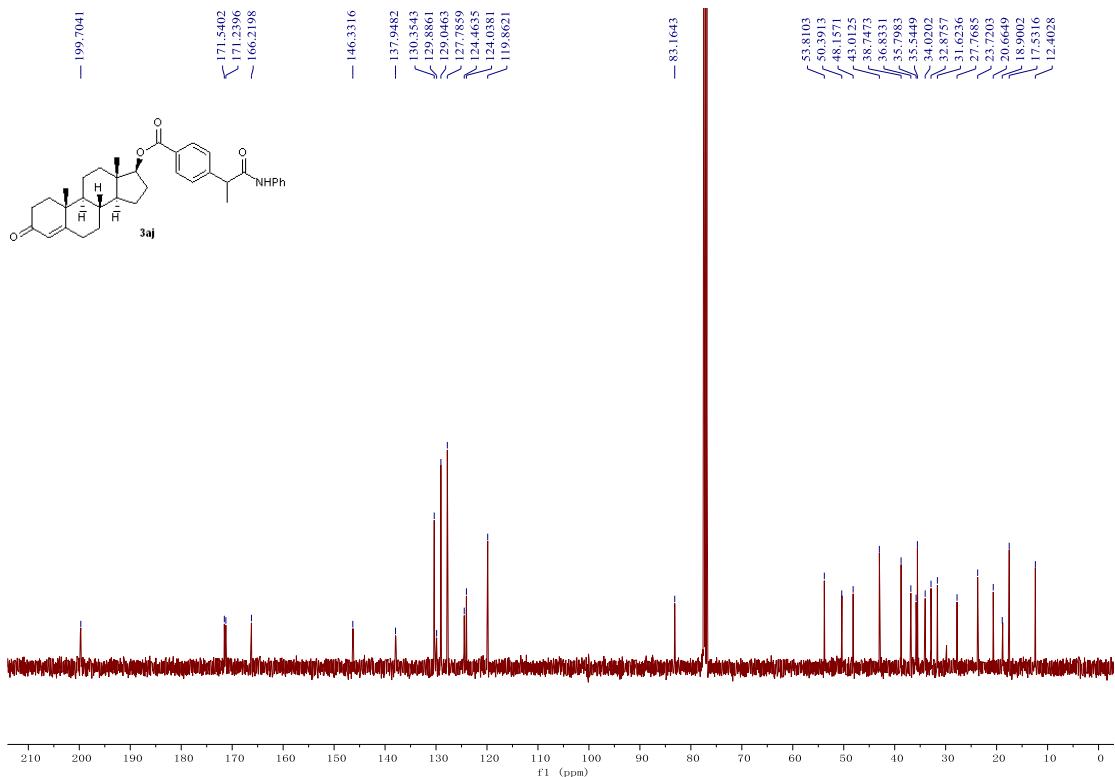
¹³C NMR spectrum of **3ai**



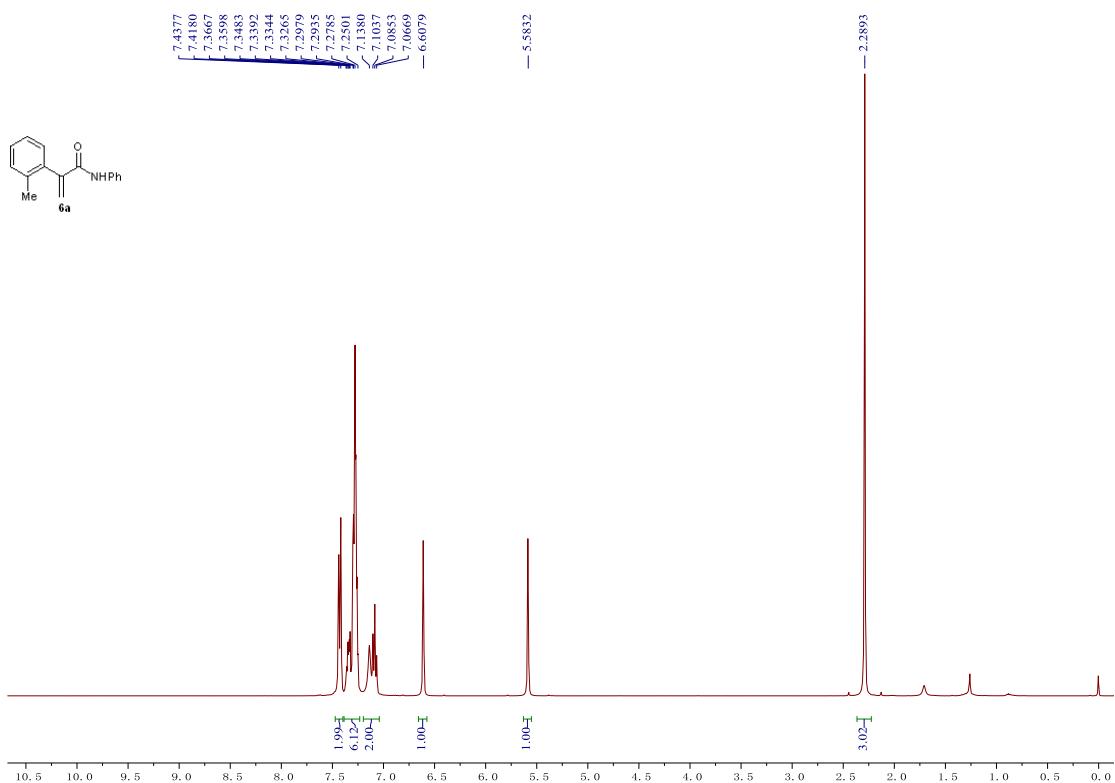
¹H NMR spectrum of 3aj



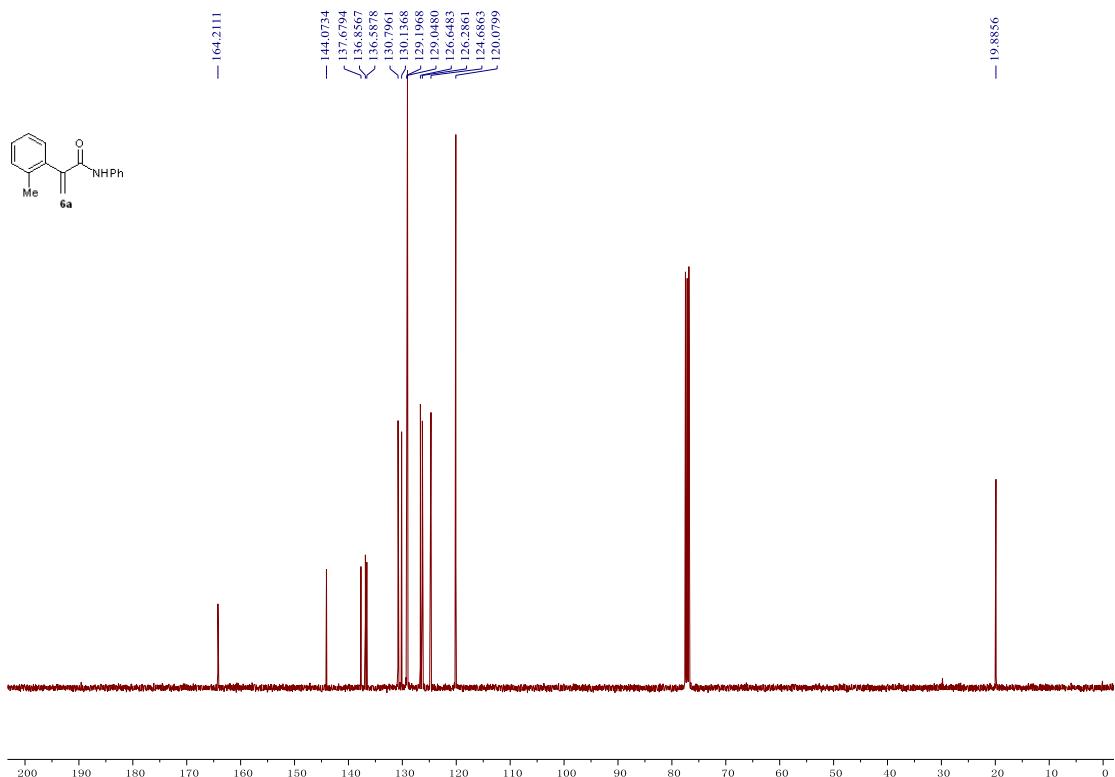
¹³C NMR spectrum of 3aj



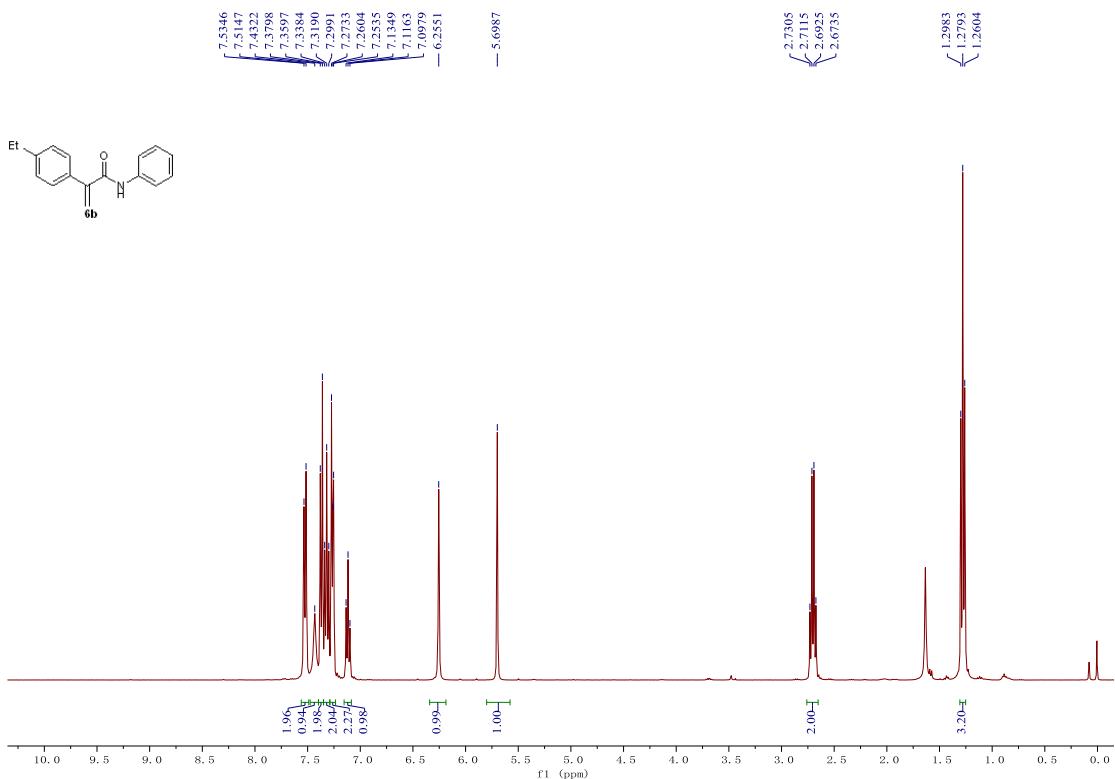
¹H NMR spectrum of 6a



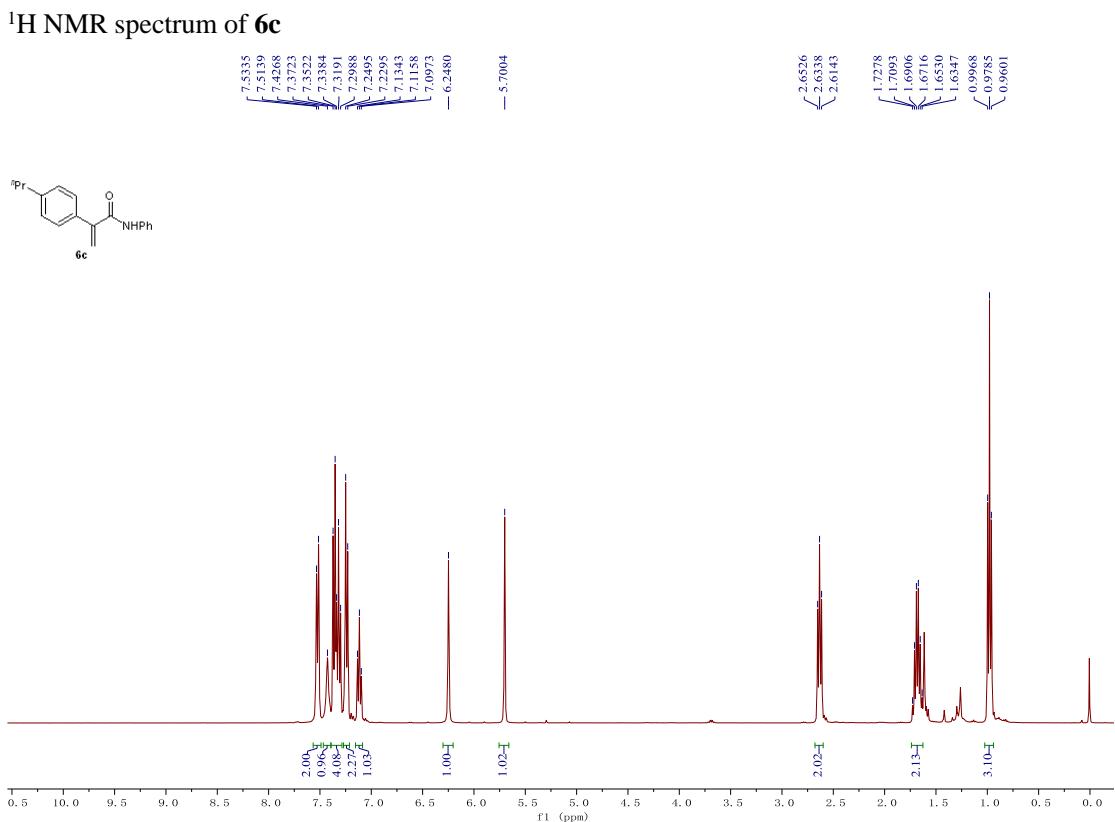
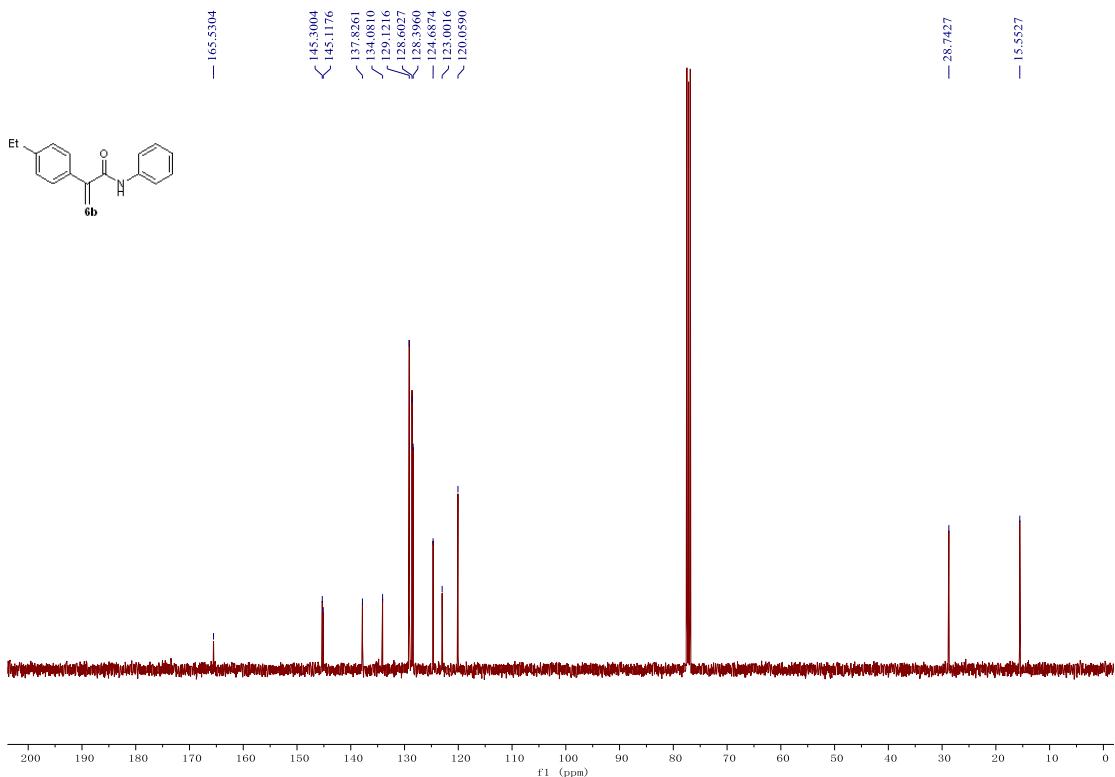
¹³C NMR spectrum of 6a



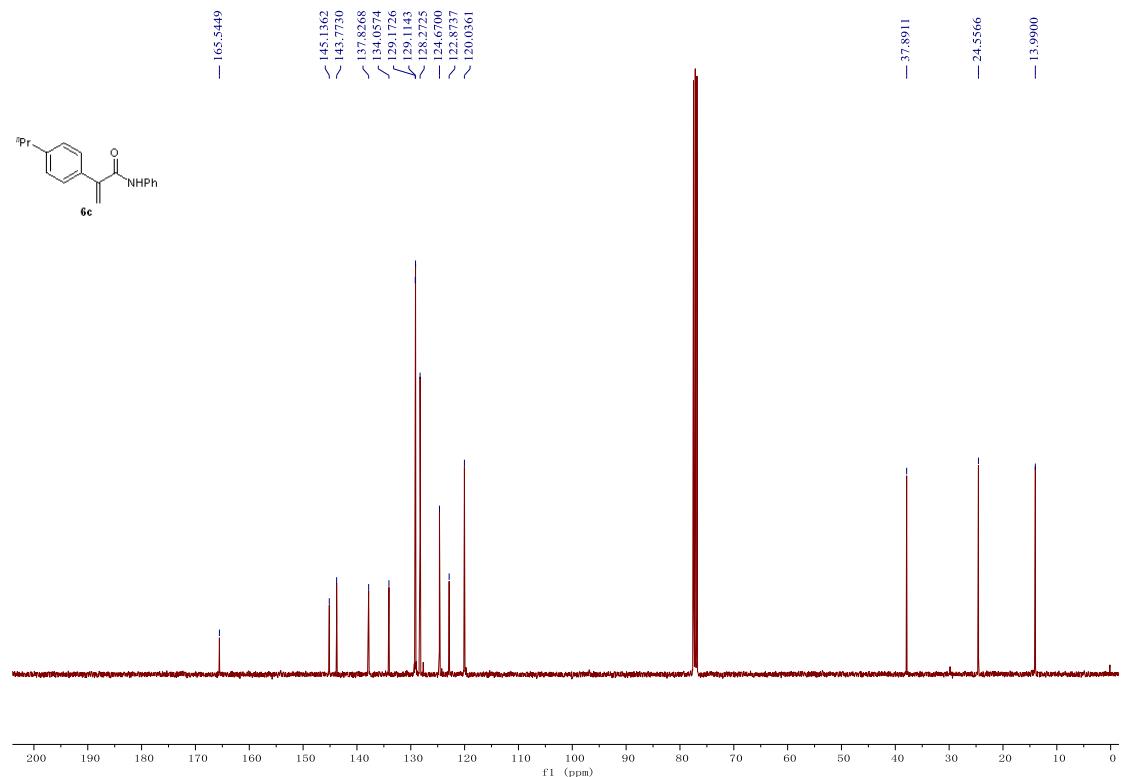
¹H NMR spectrum of **6b**



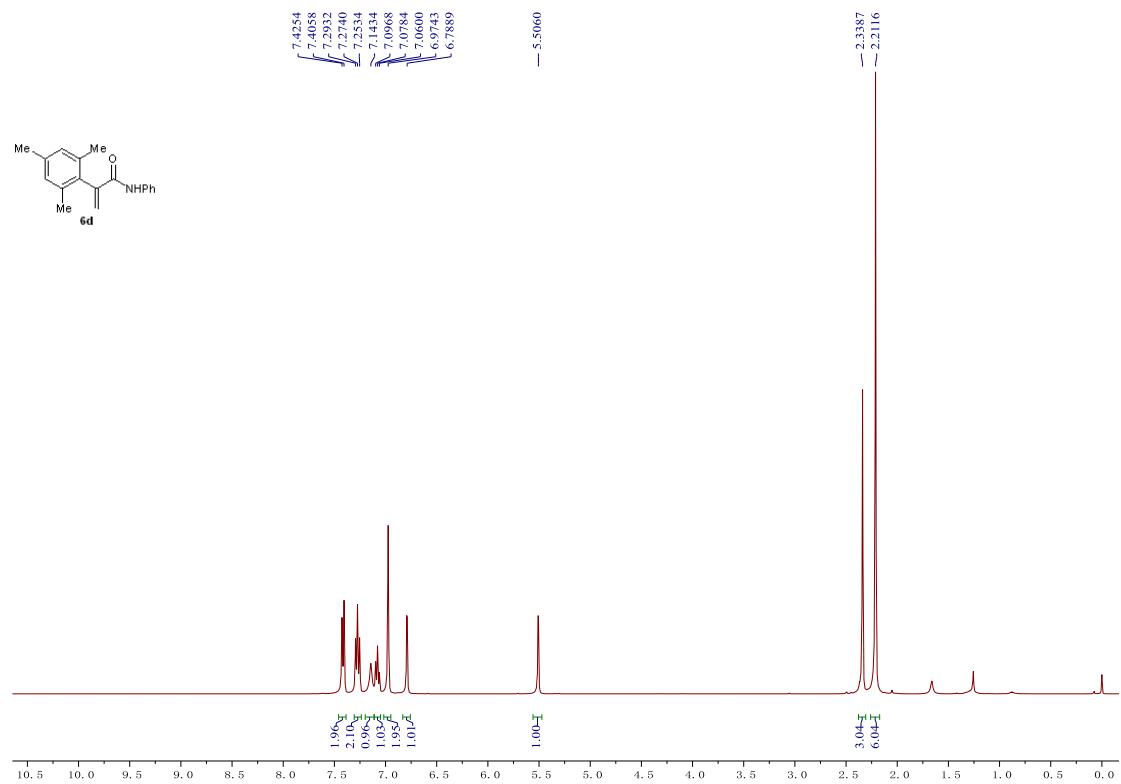
¹³C NMR spectrum of **6b**



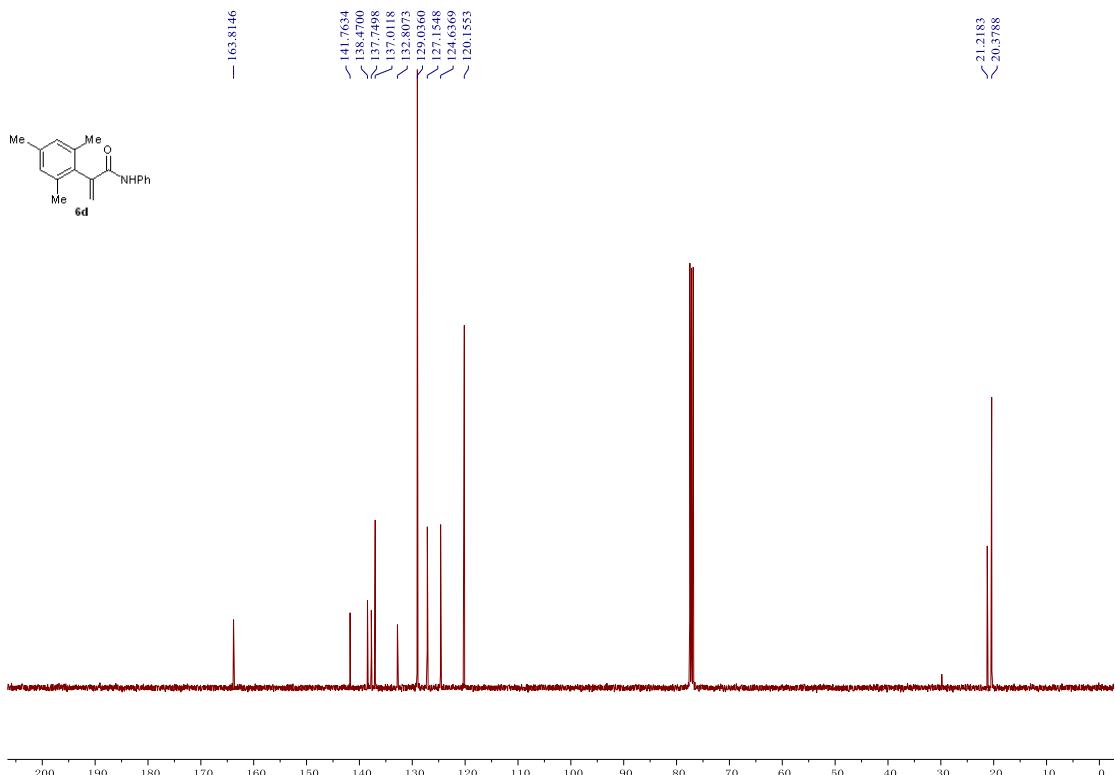
¹³C NMR spectrum of **6c**



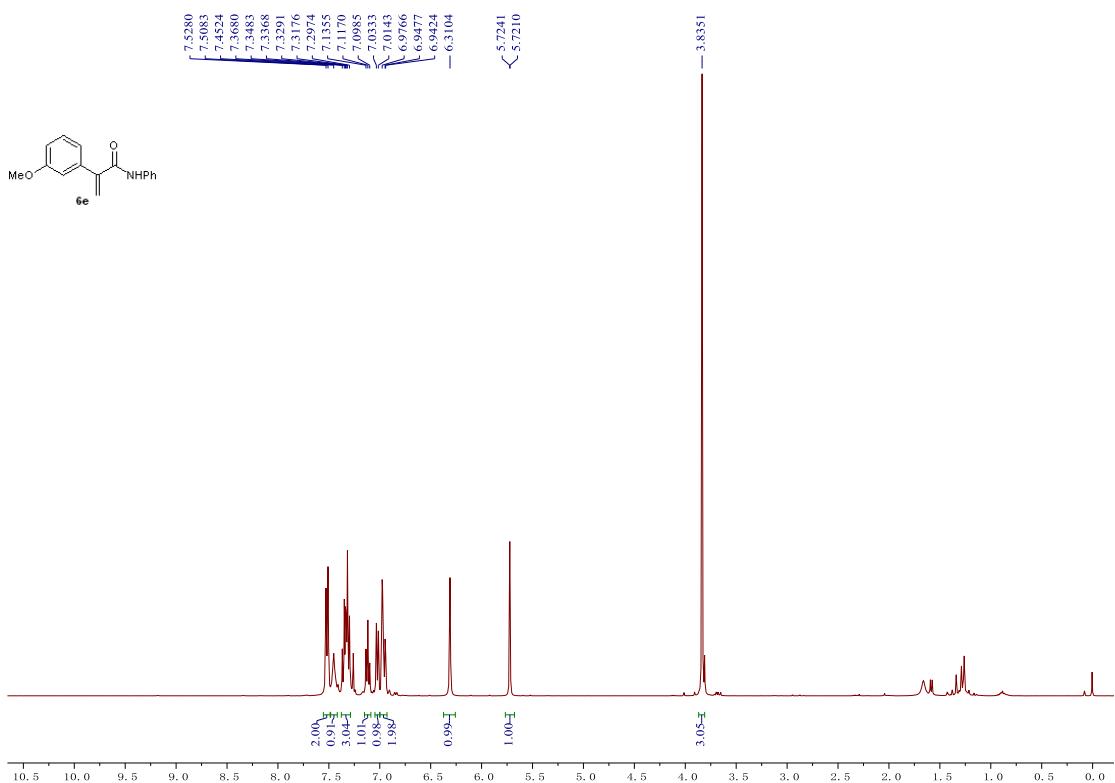
¹H NMR spectrum of **6d**



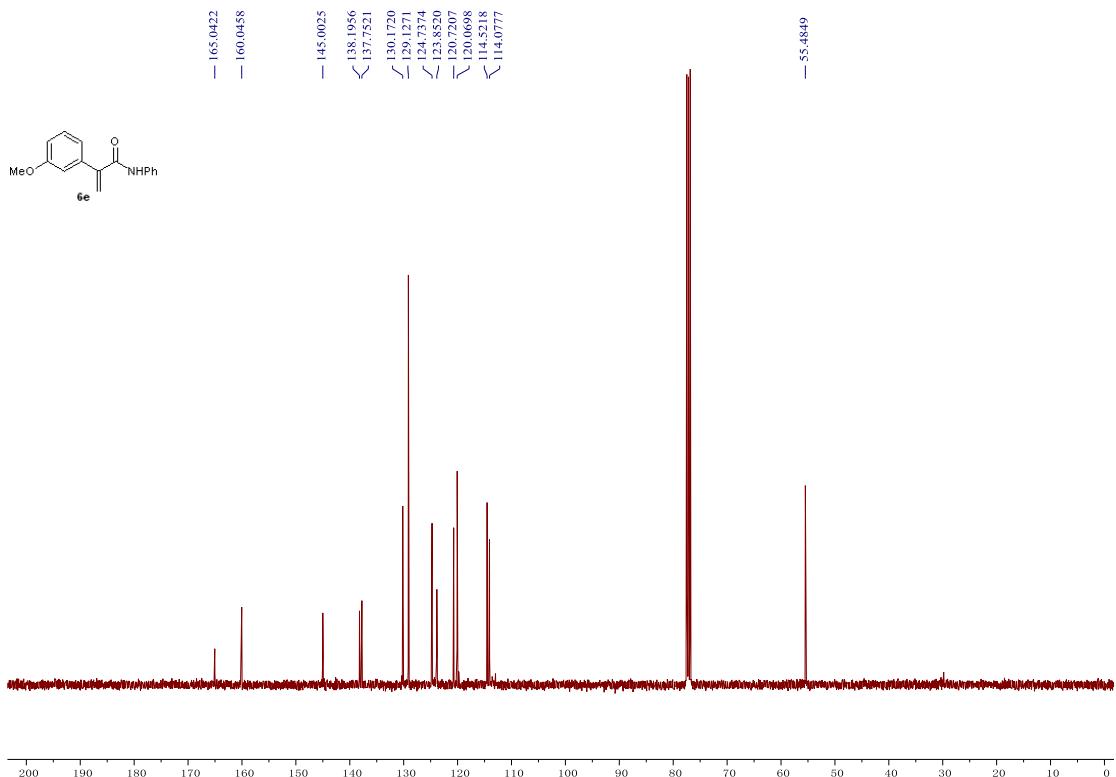
¹³C NMR spectrum of **6d**



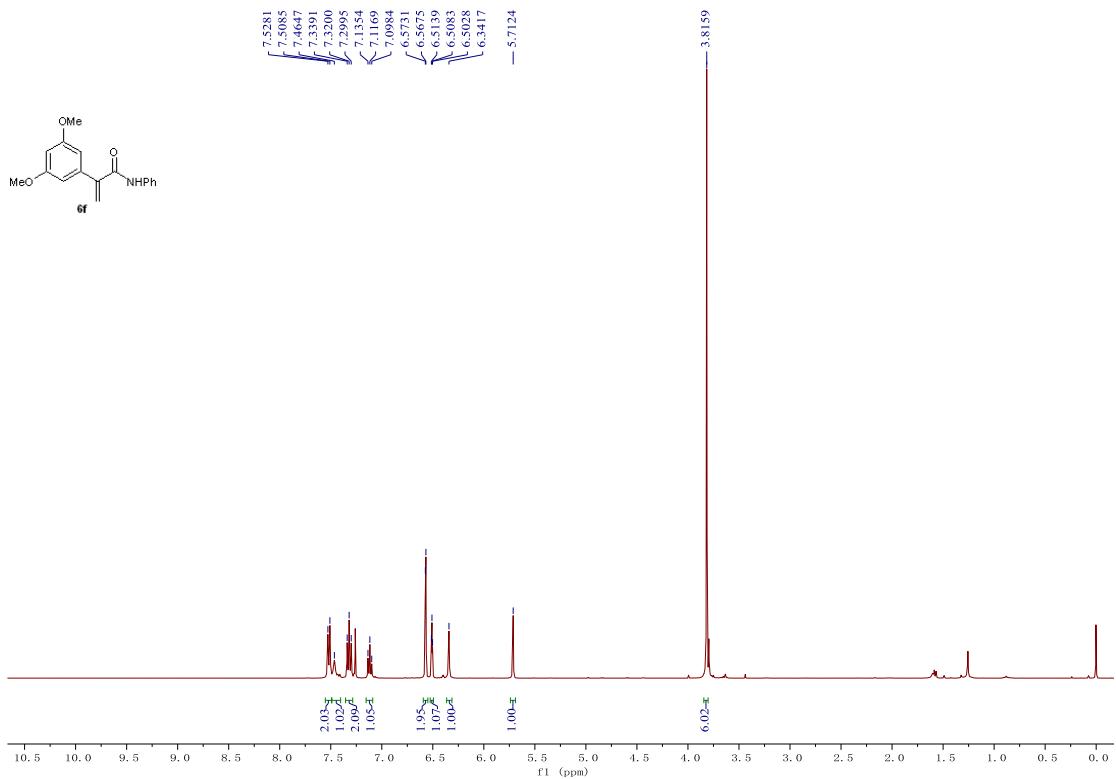
¹H NMR spectrum of **6e**



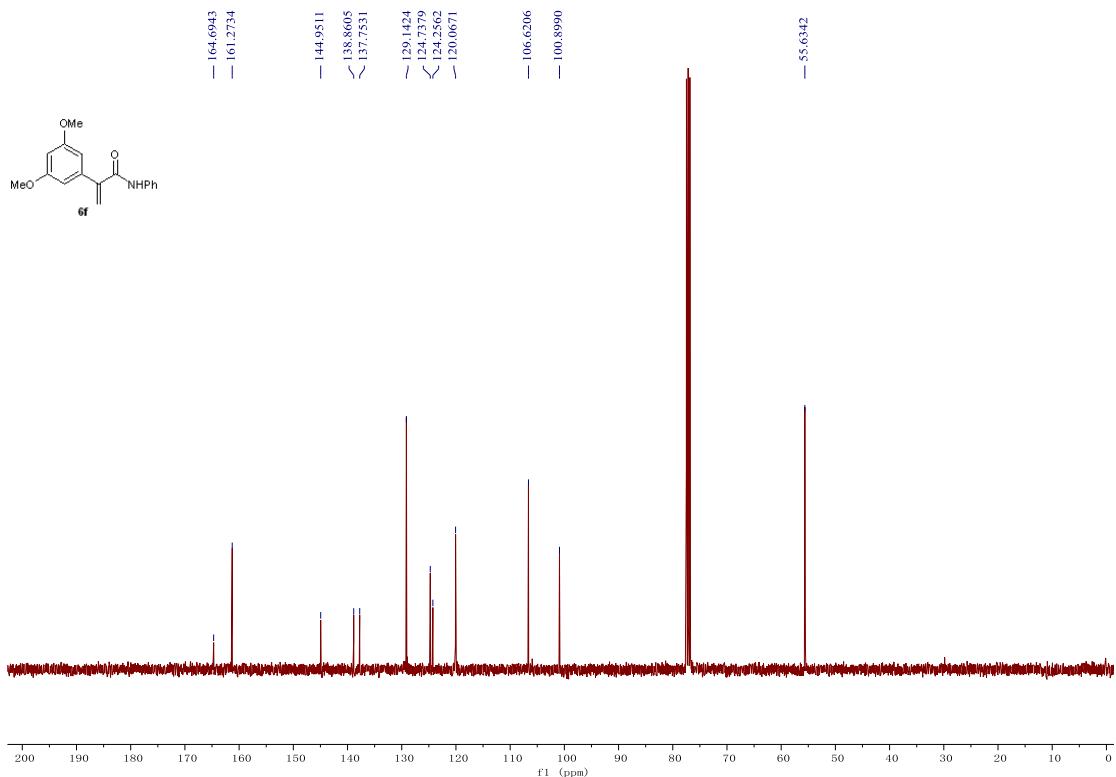
¹³C NMR spectrum of **6e**



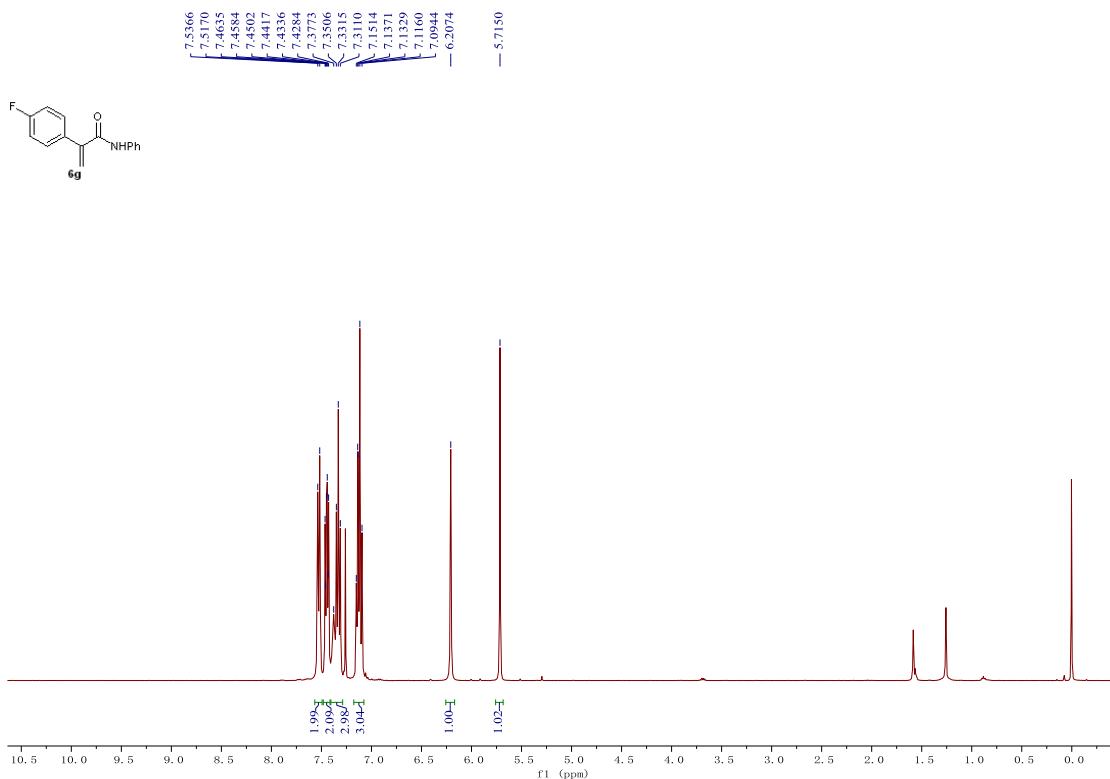
¹H NMR spectrum of **6f**



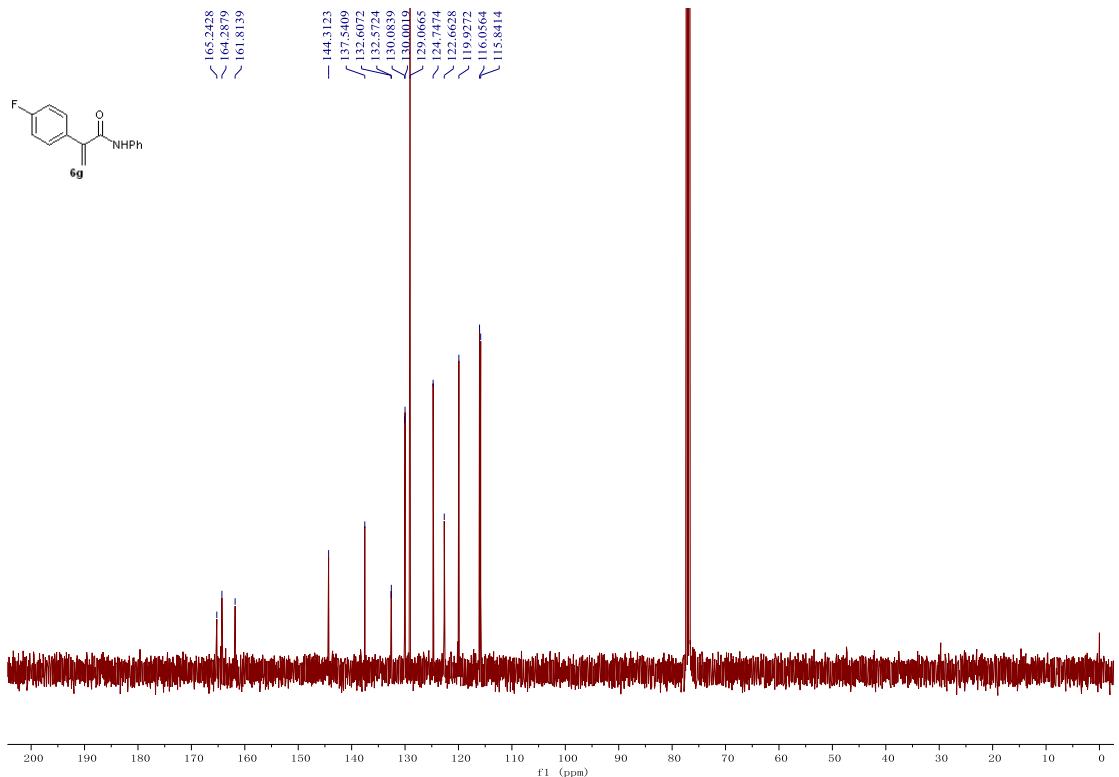
¹³C NMR spectrum of **6f**



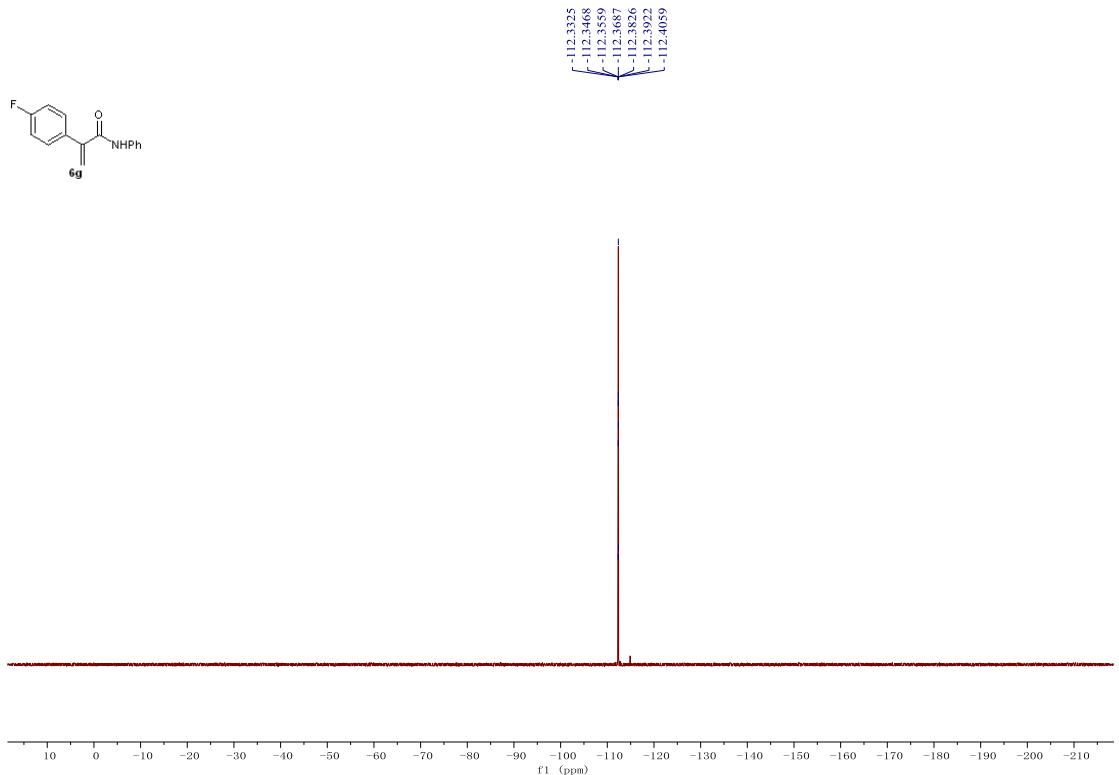
¹H NMR spectrum of **6g**



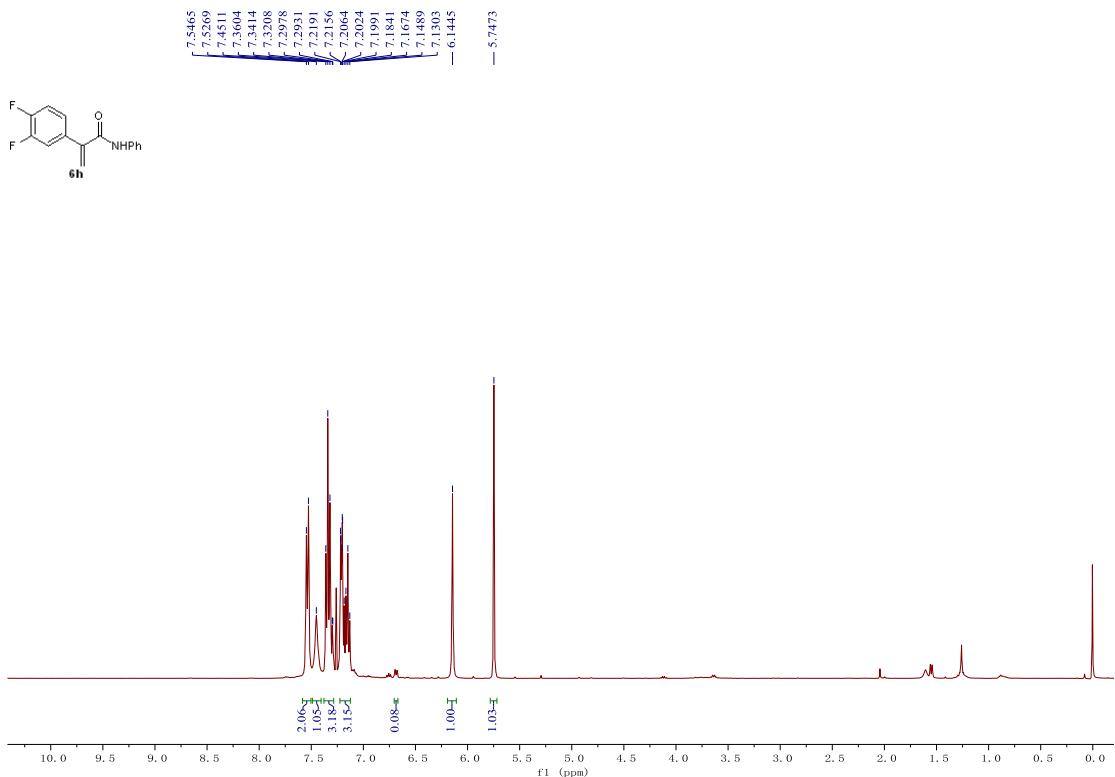
¹³C NMR spectrum of **6g**



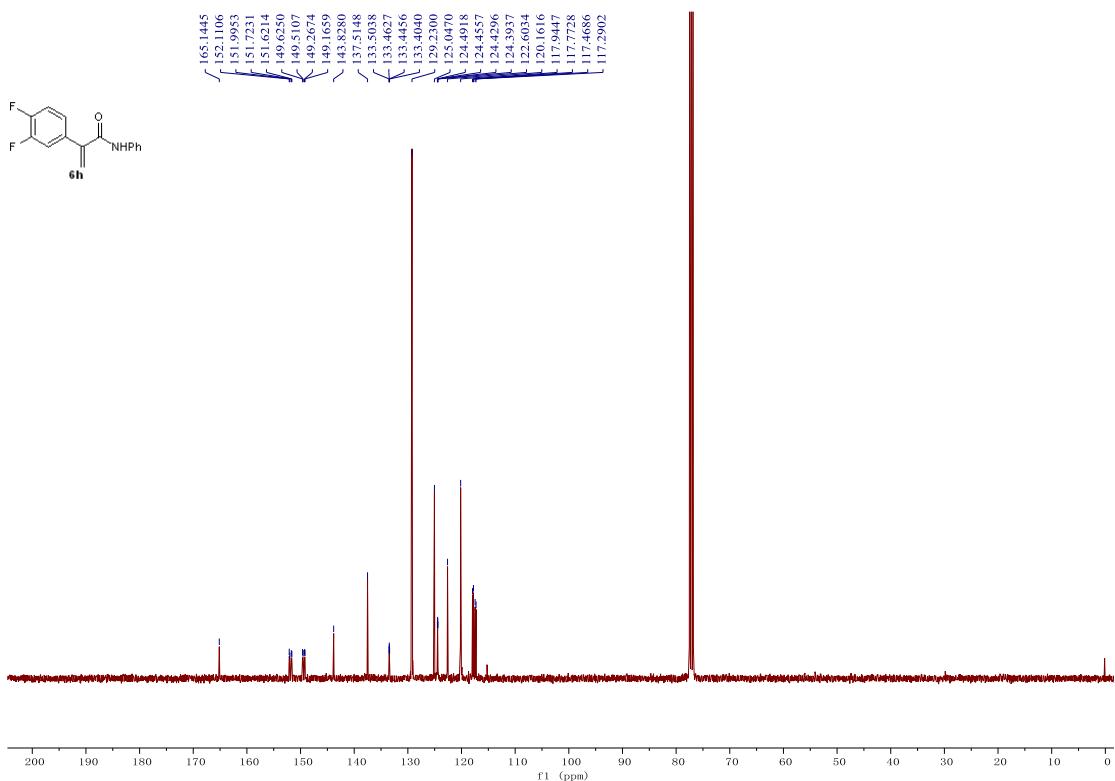
¹⁹F NMR spectrum of **6g**



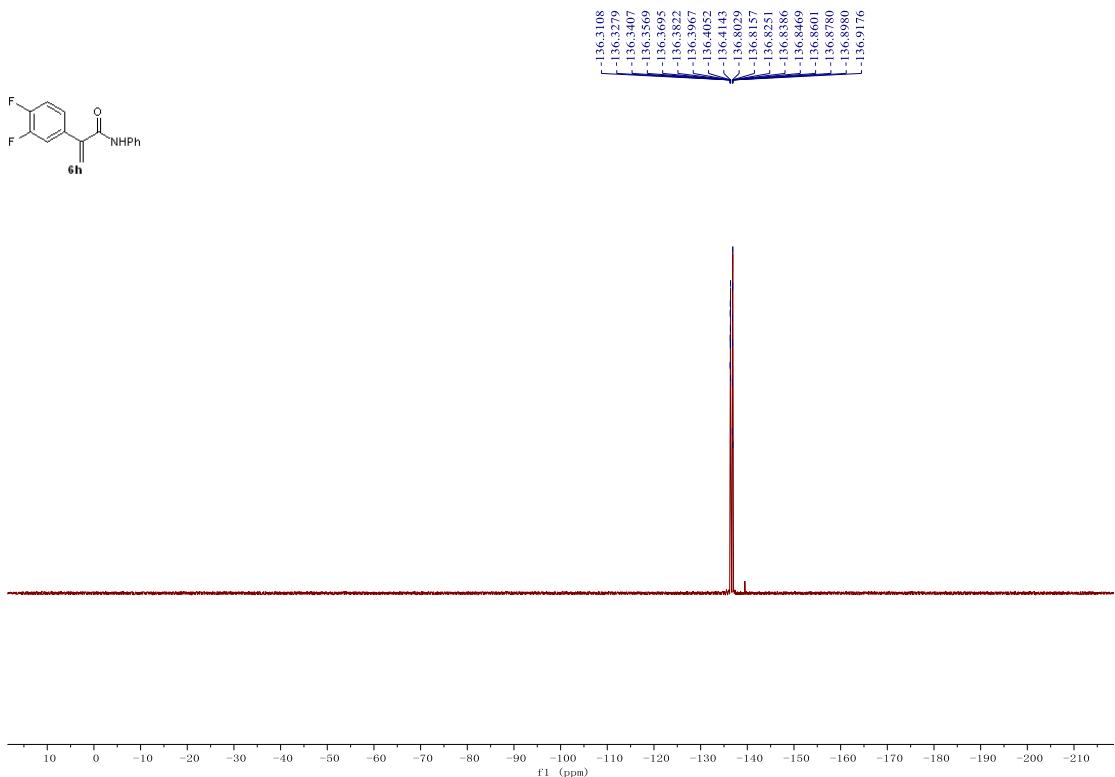
¹H NMR spectrum of **6h**



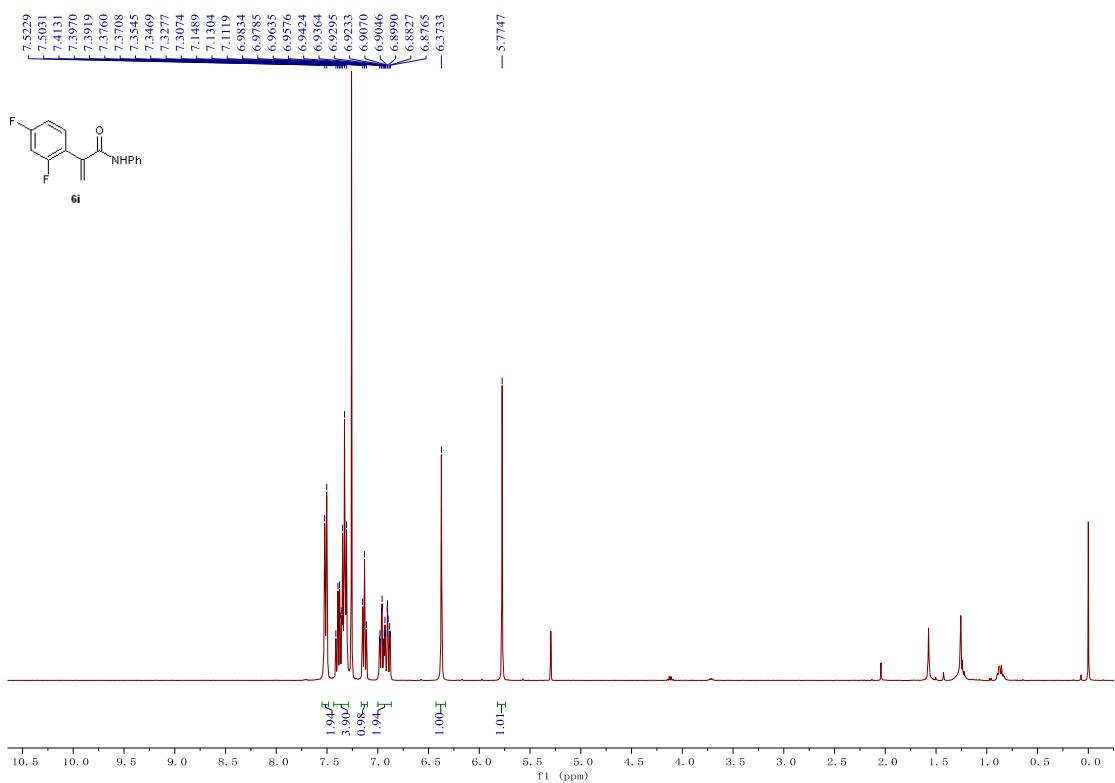
¹³C NMR spectrum of **6h**



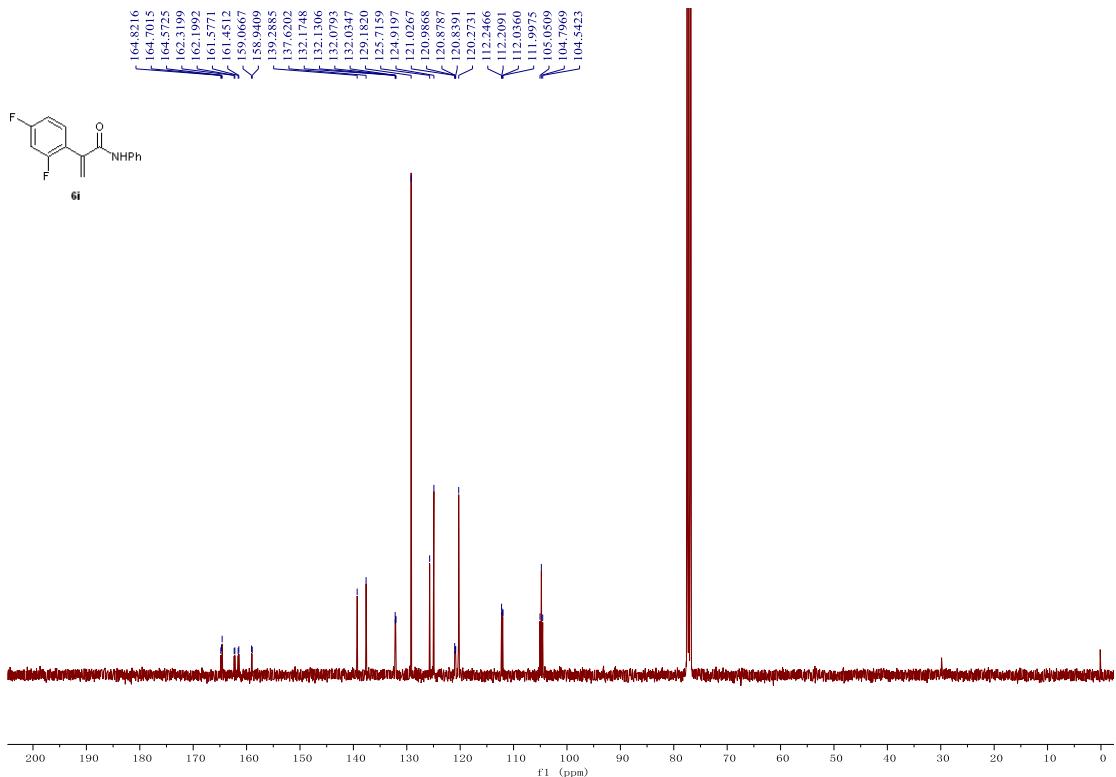
¹⁹F NMR spectrum of **6h**



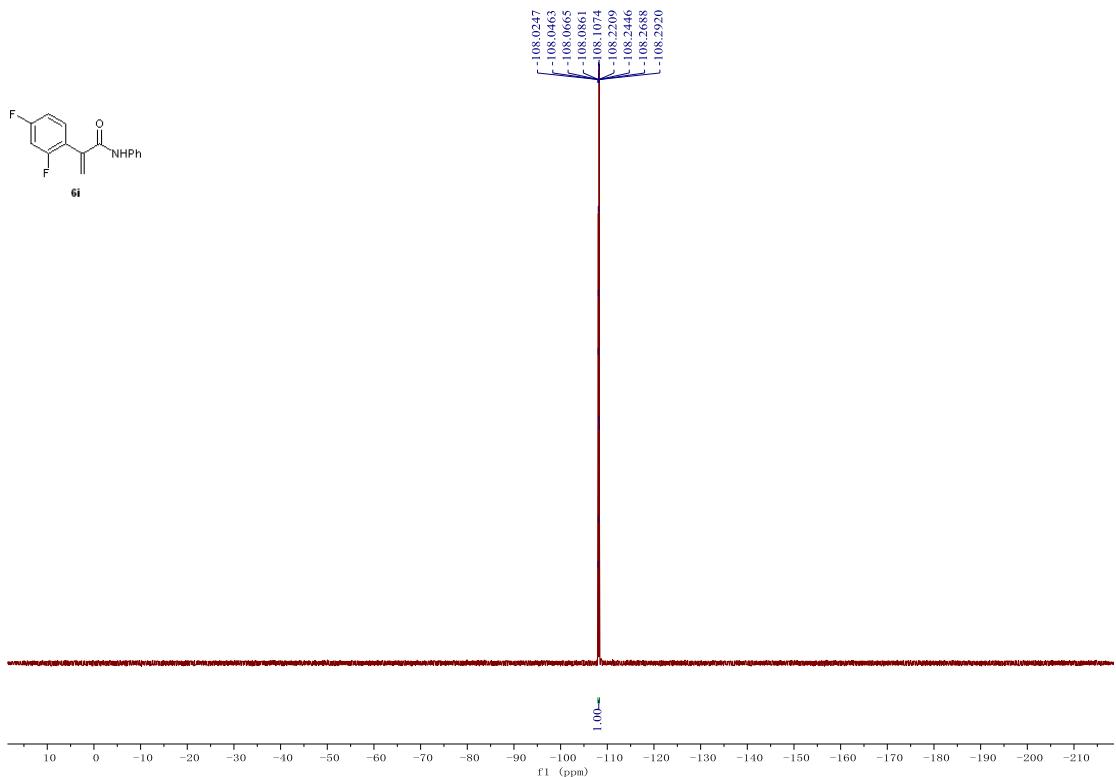
¹H NMR spectrum of **6i**



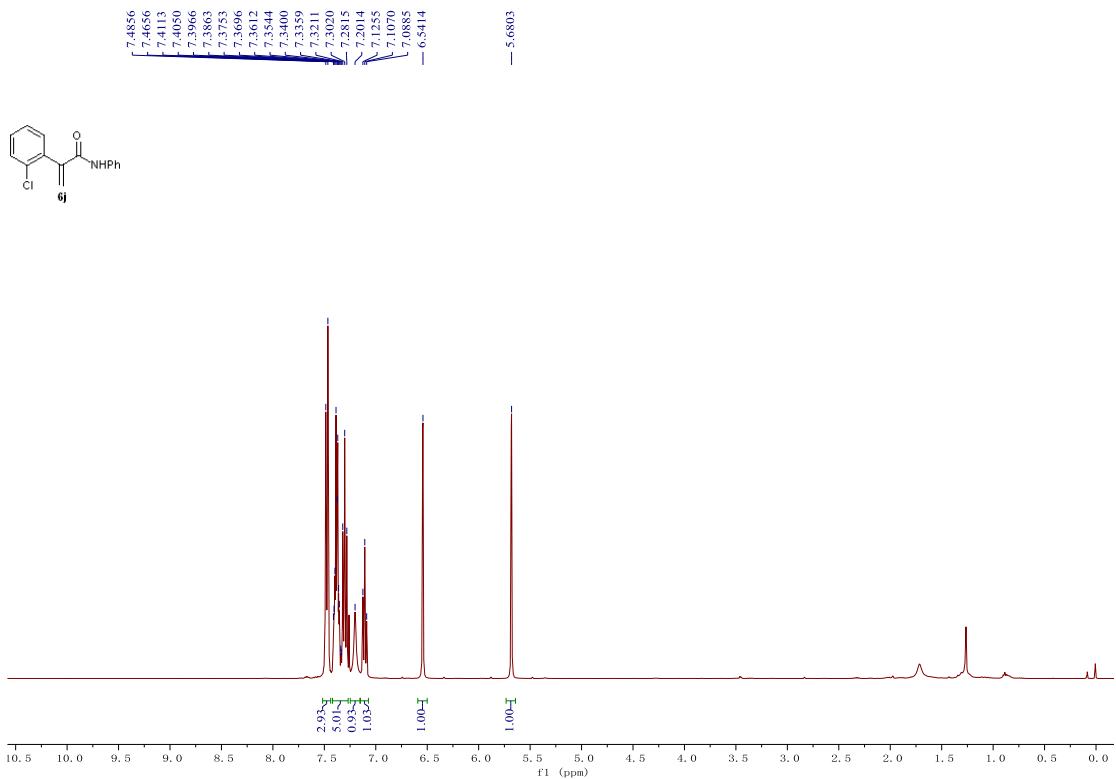
¹³C NMR spectrum of **6i**



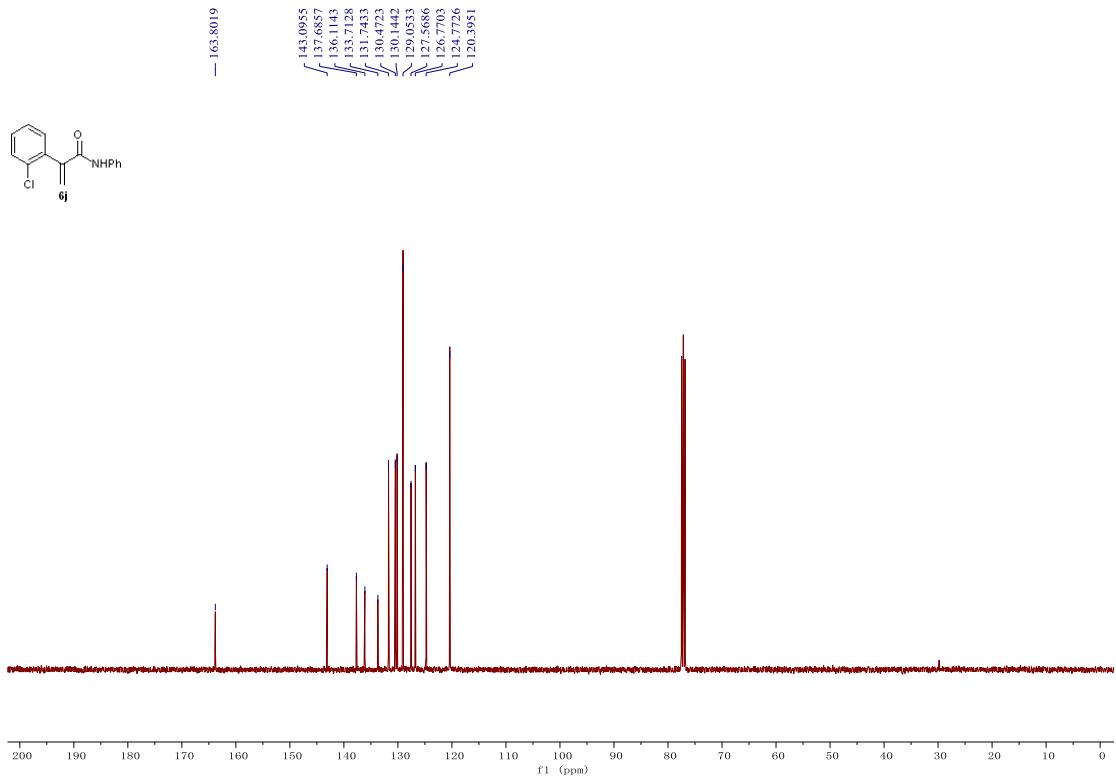
¹⁹F NMR spectrum of **6i**



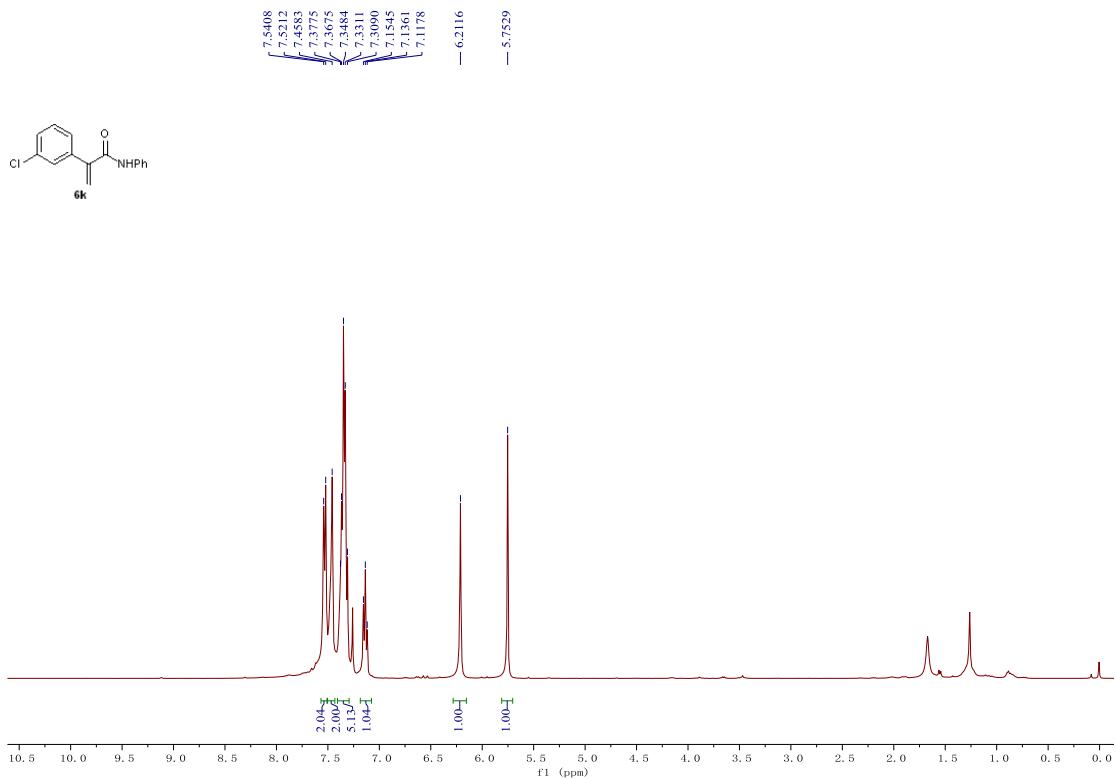
¹H NMR spectrum of **6j**



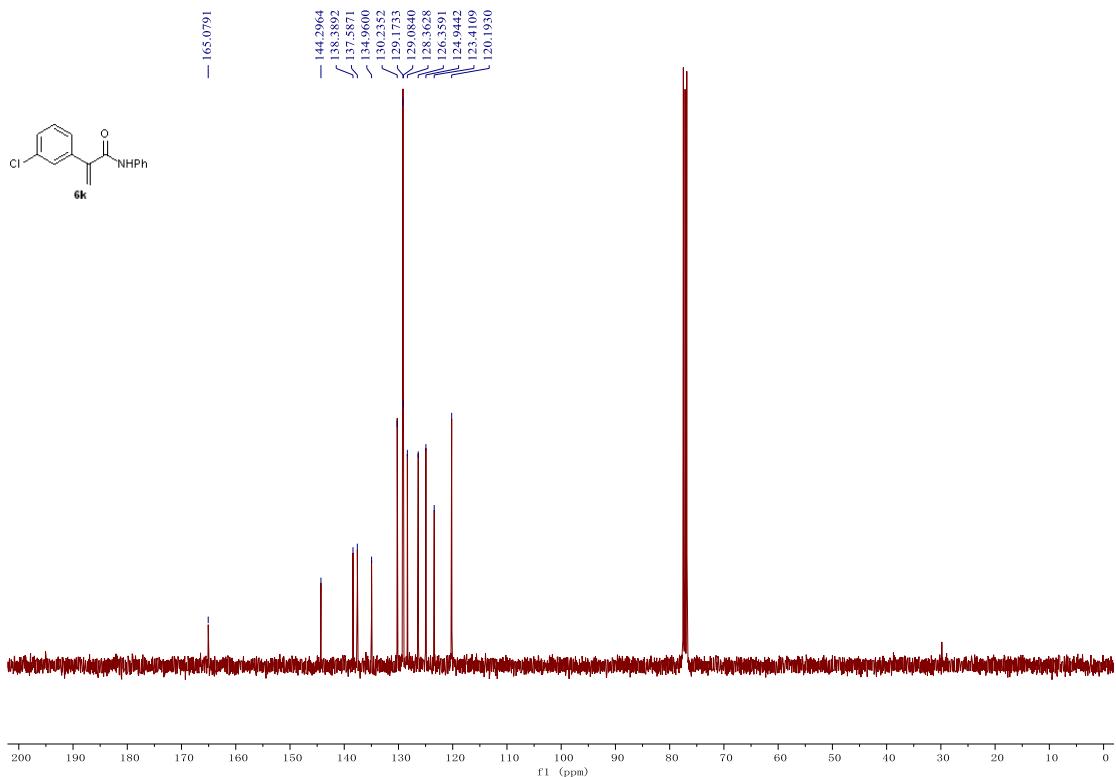
¹³C NMR spectrum of **6j**



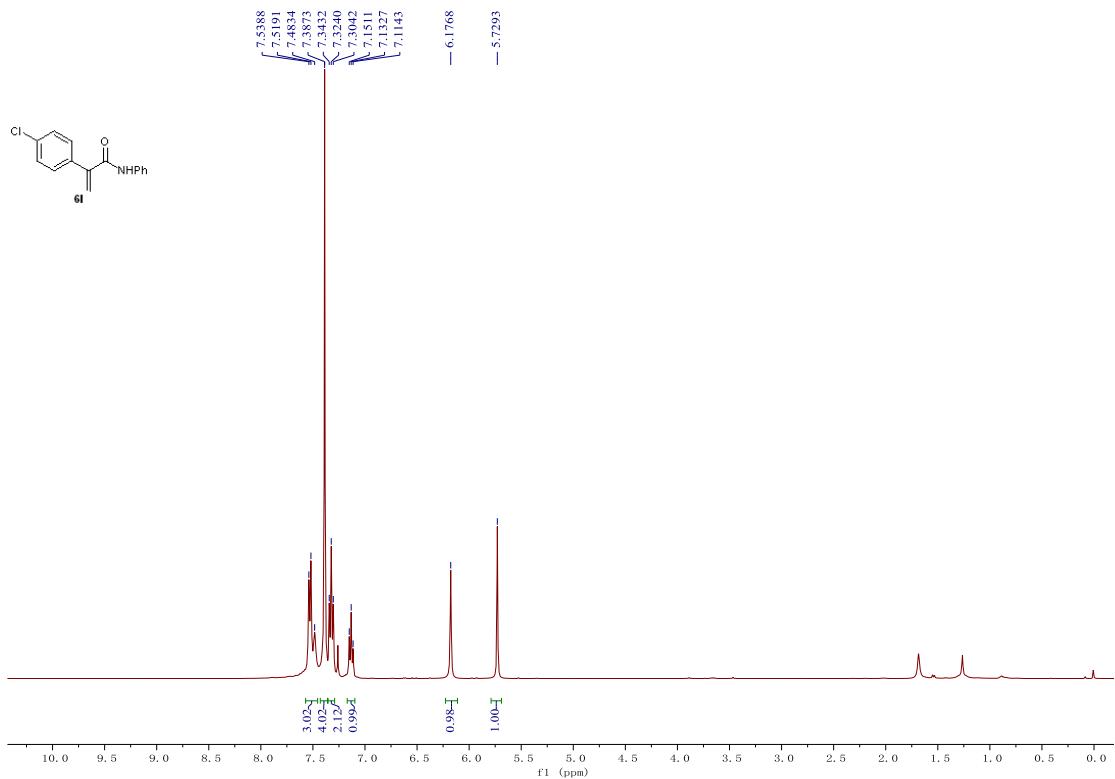
¹H NMR spectrum of **6k**



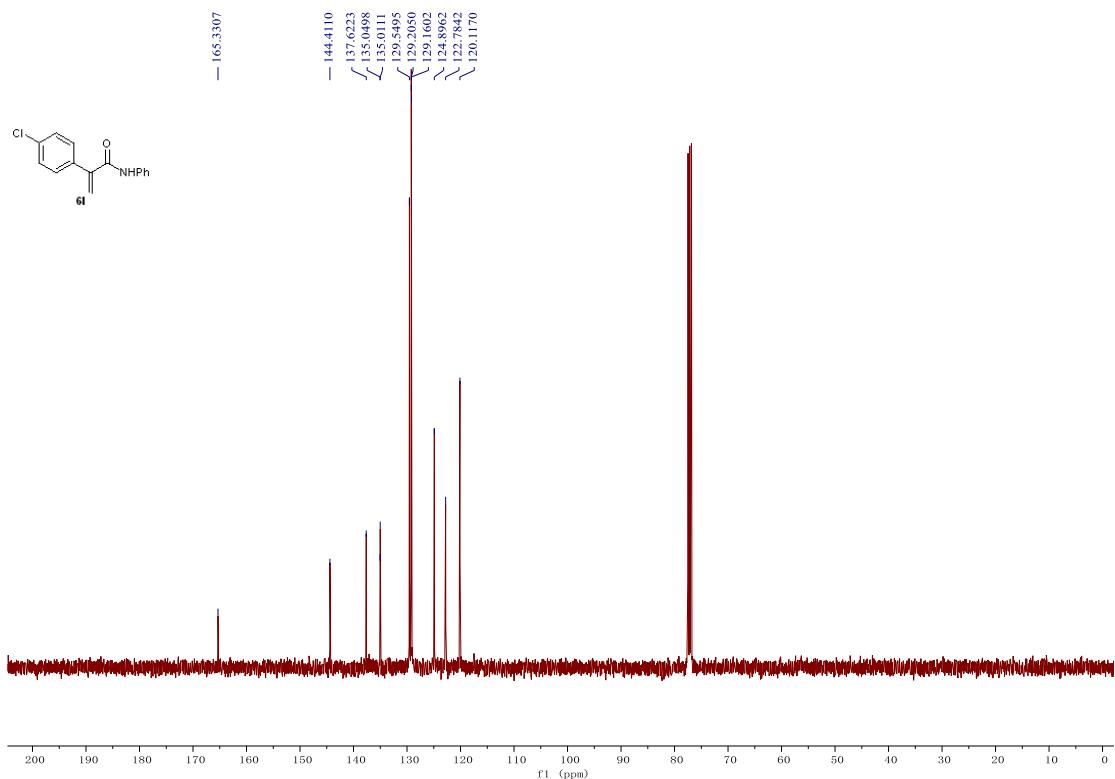
¹³C NMR spectrum of **6k**



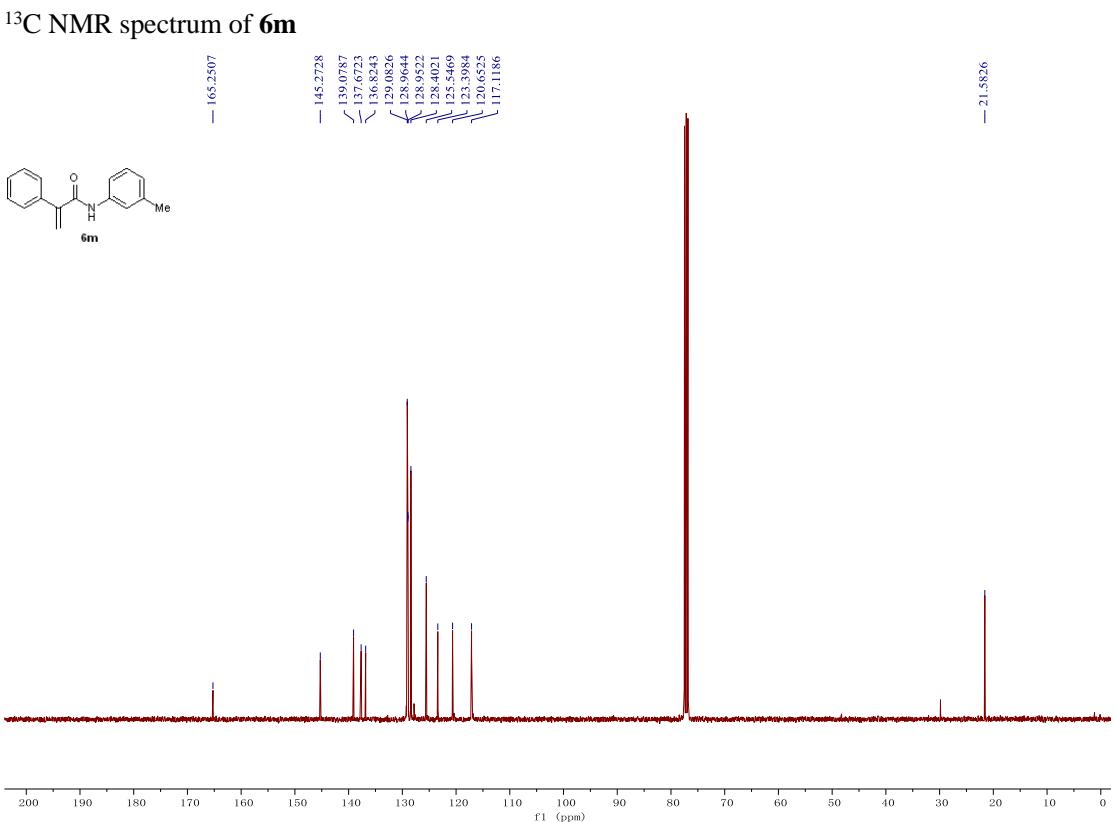
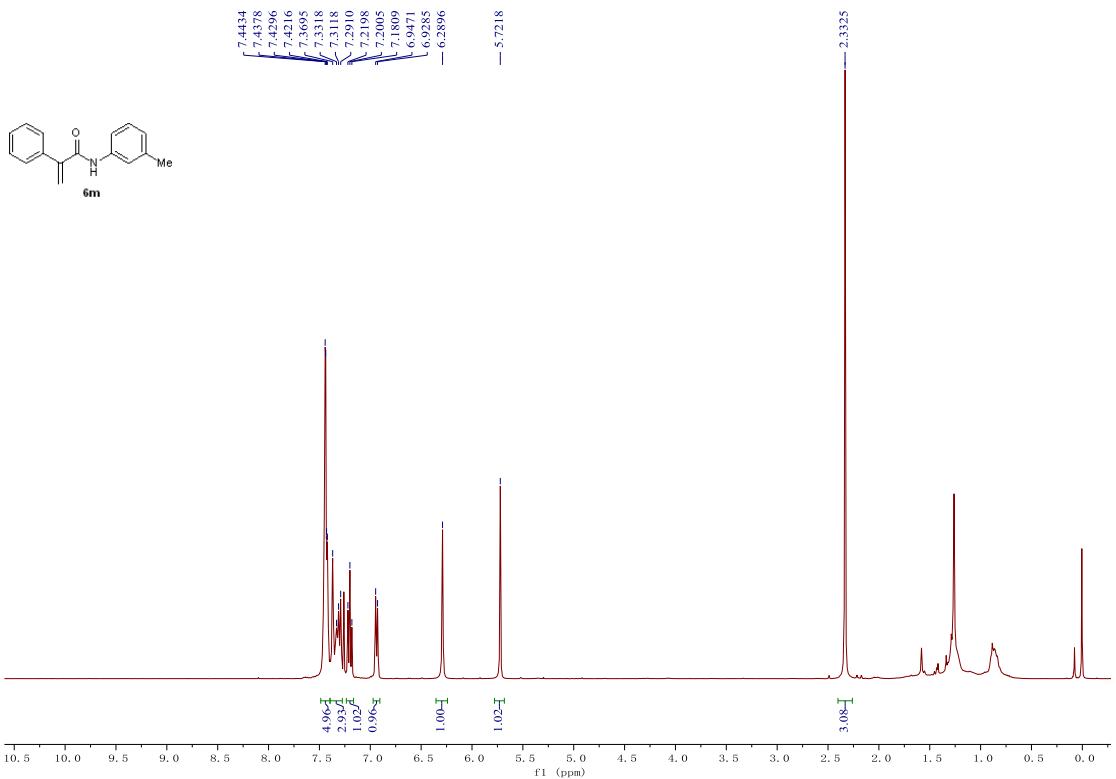
¹H NMR spectrum of **6l**



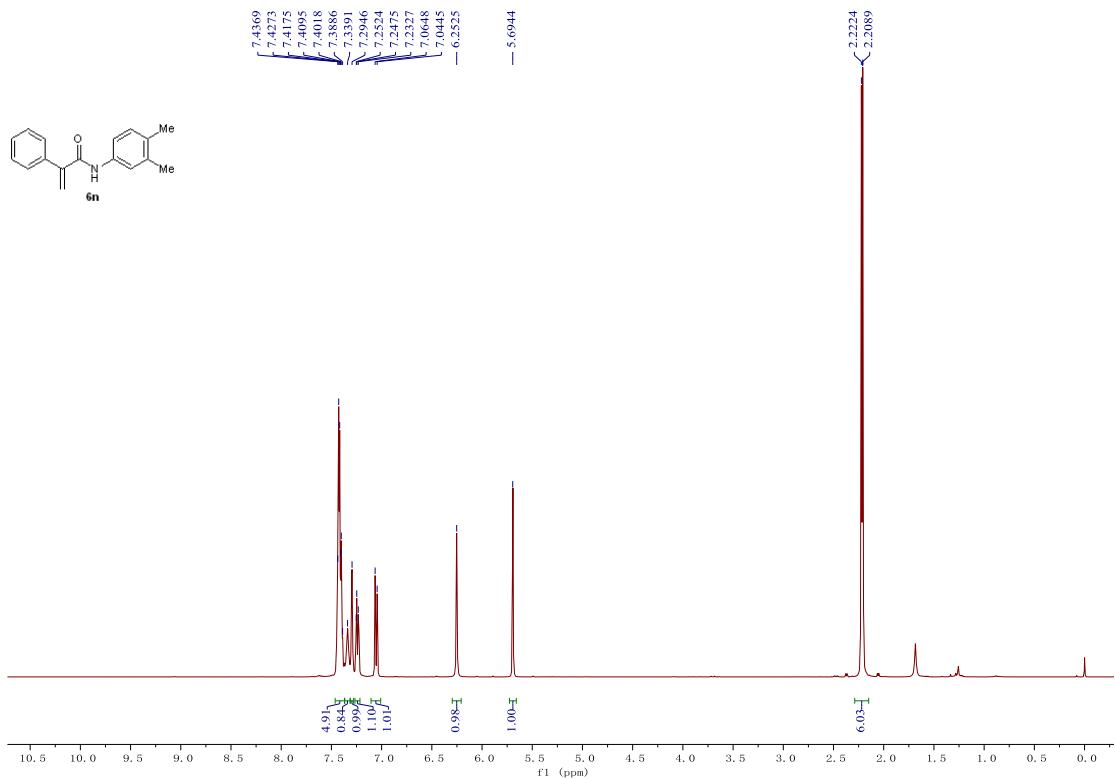
¹³C NMR spectrum of **6l**



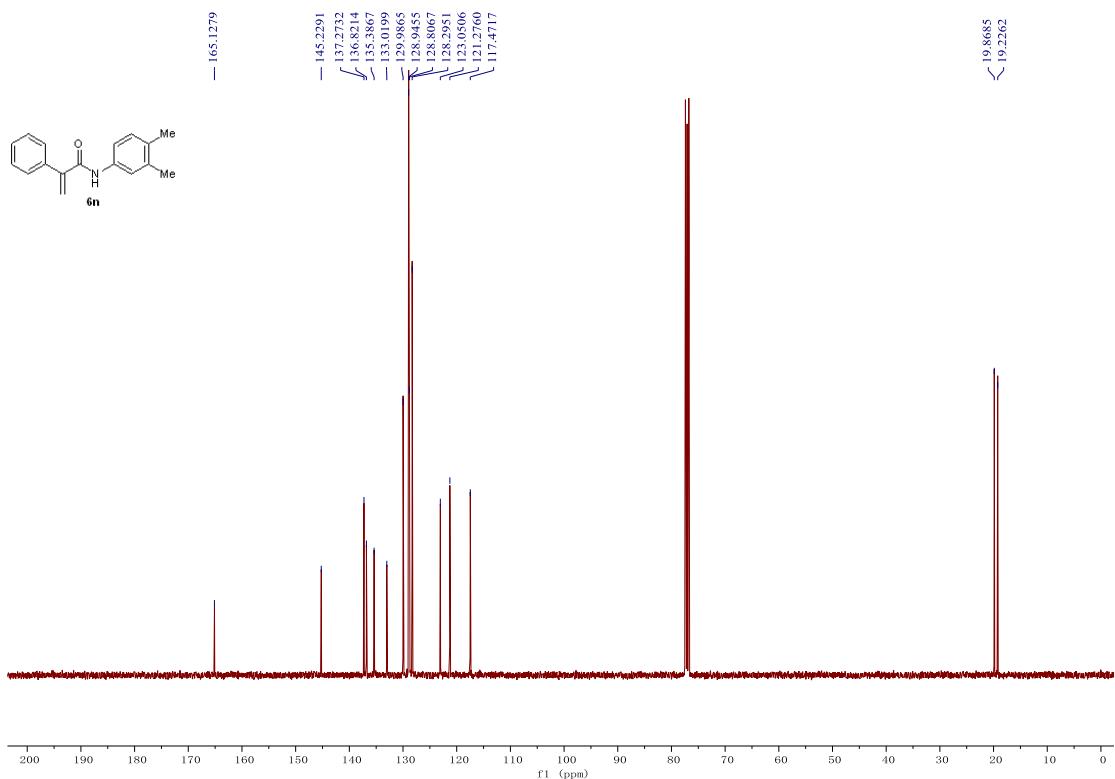
¹H NMR spectrum of **6m**



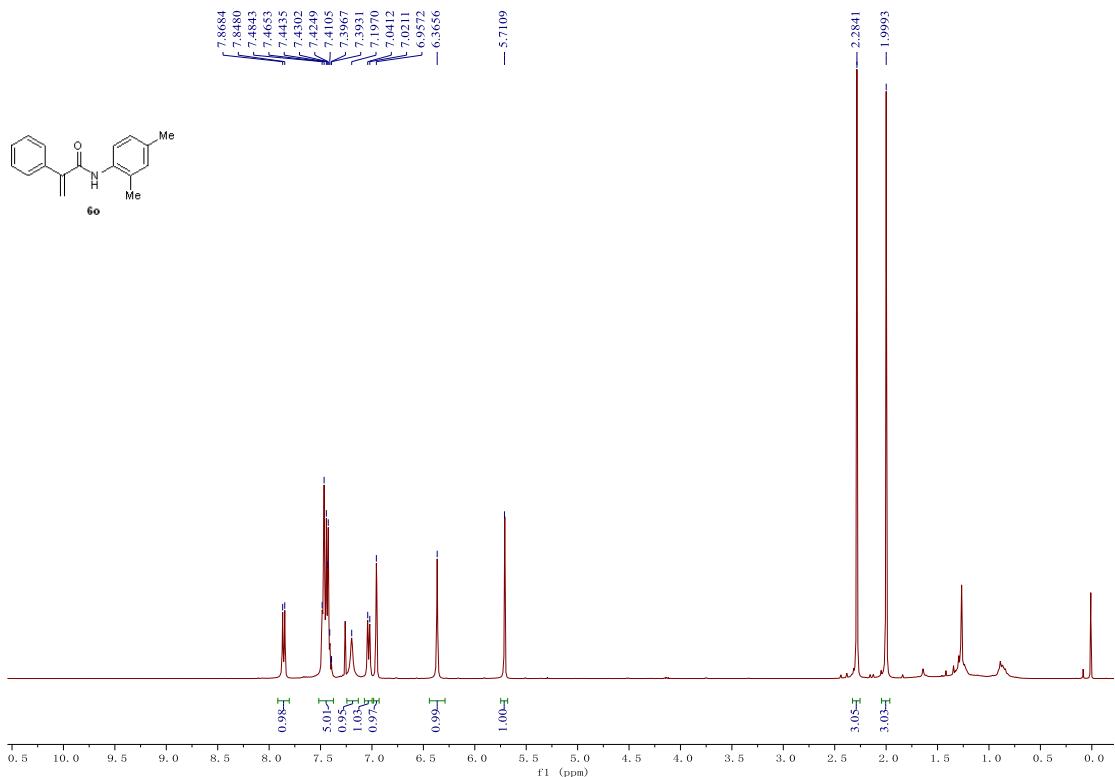
¹H NMR spectrum of **6n**



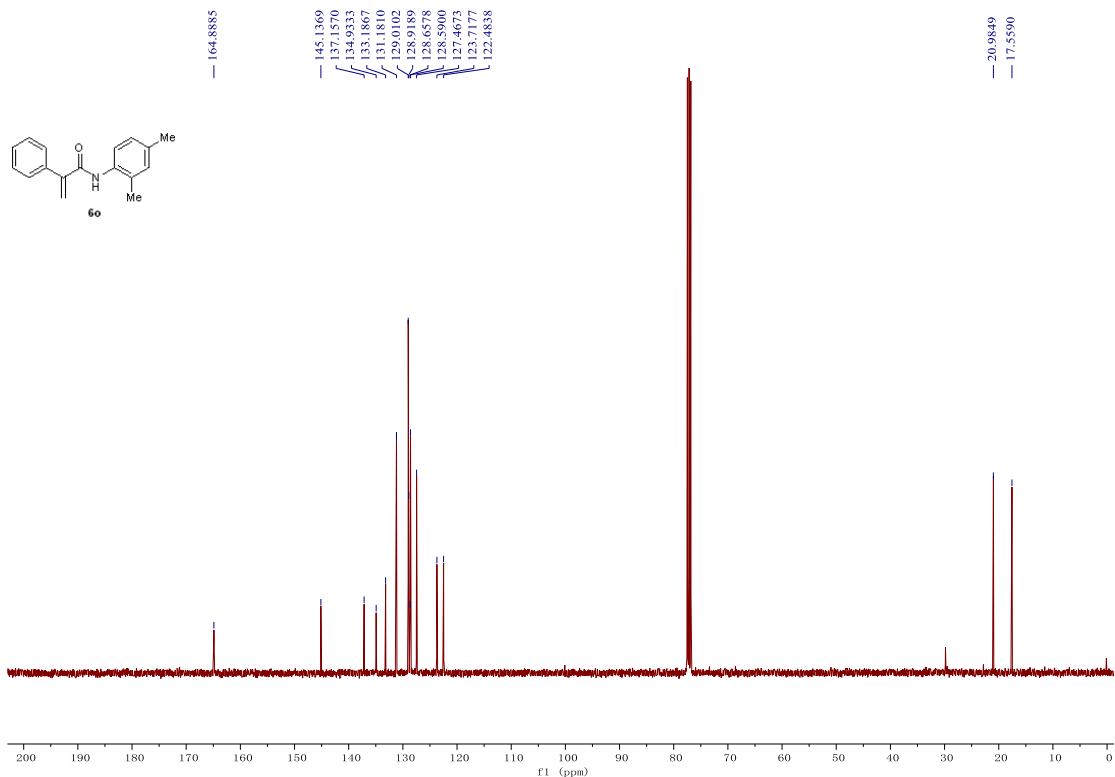
¹³C NMR spectrum of **6n**



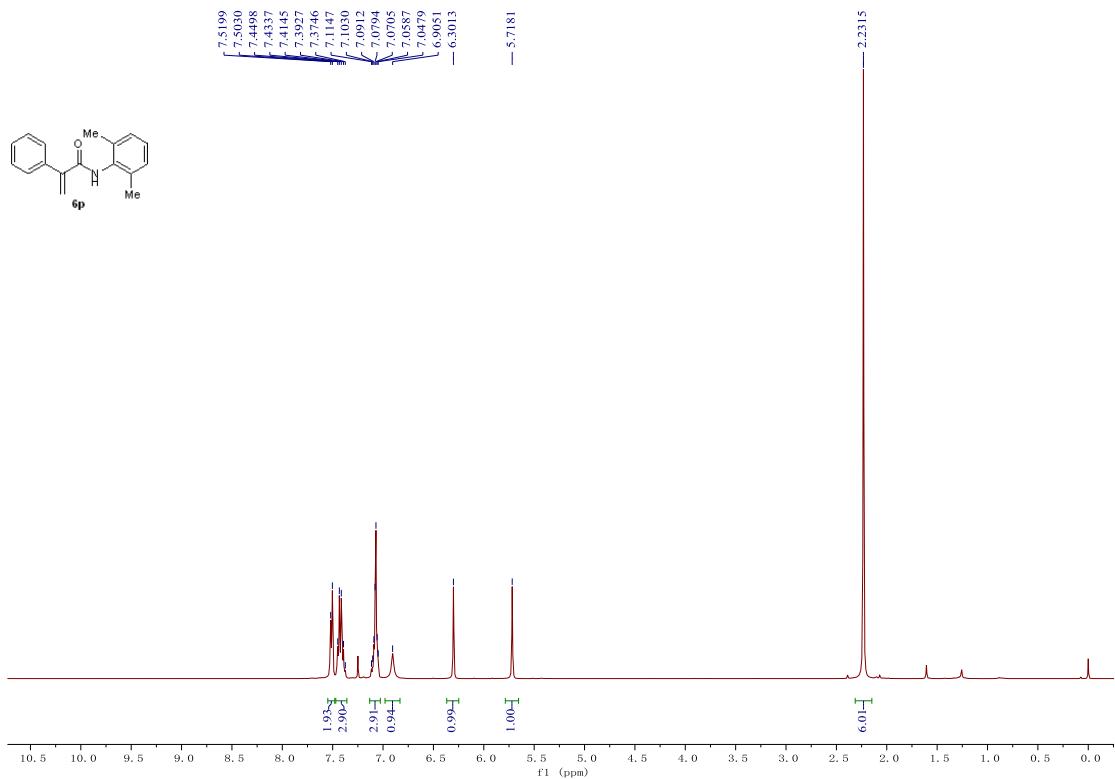
¹H NMR spectrum of **6o**



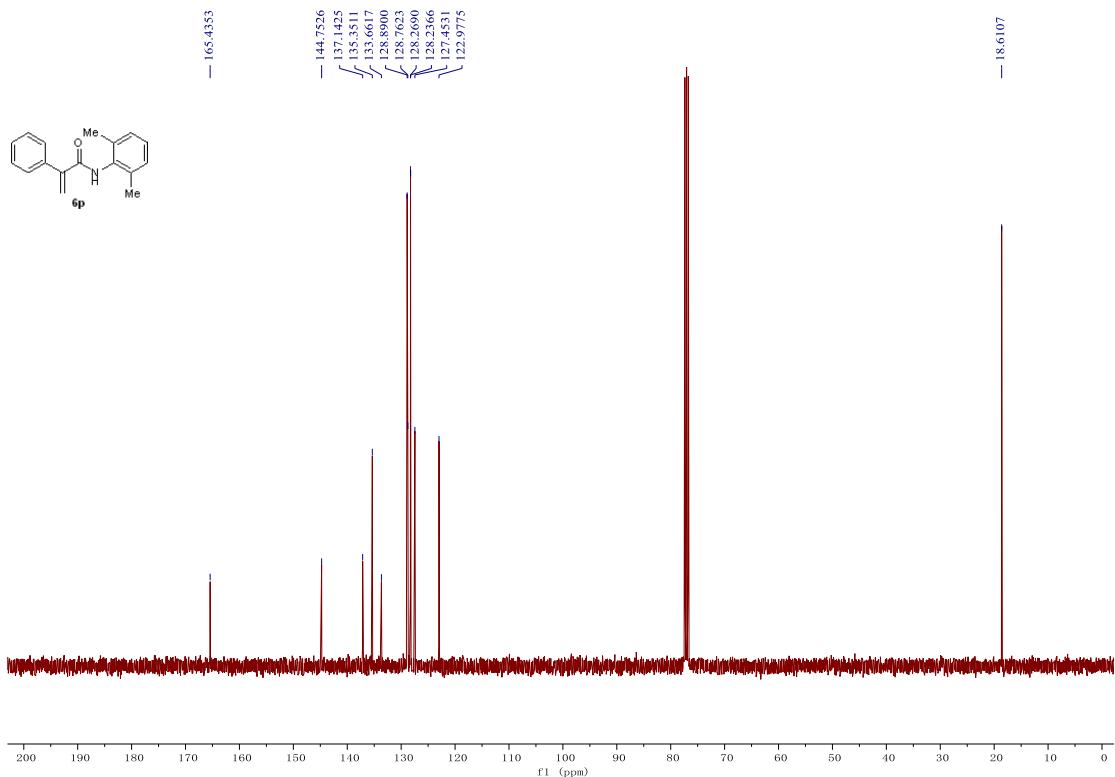
¹³C NMR spectrum of **6o**



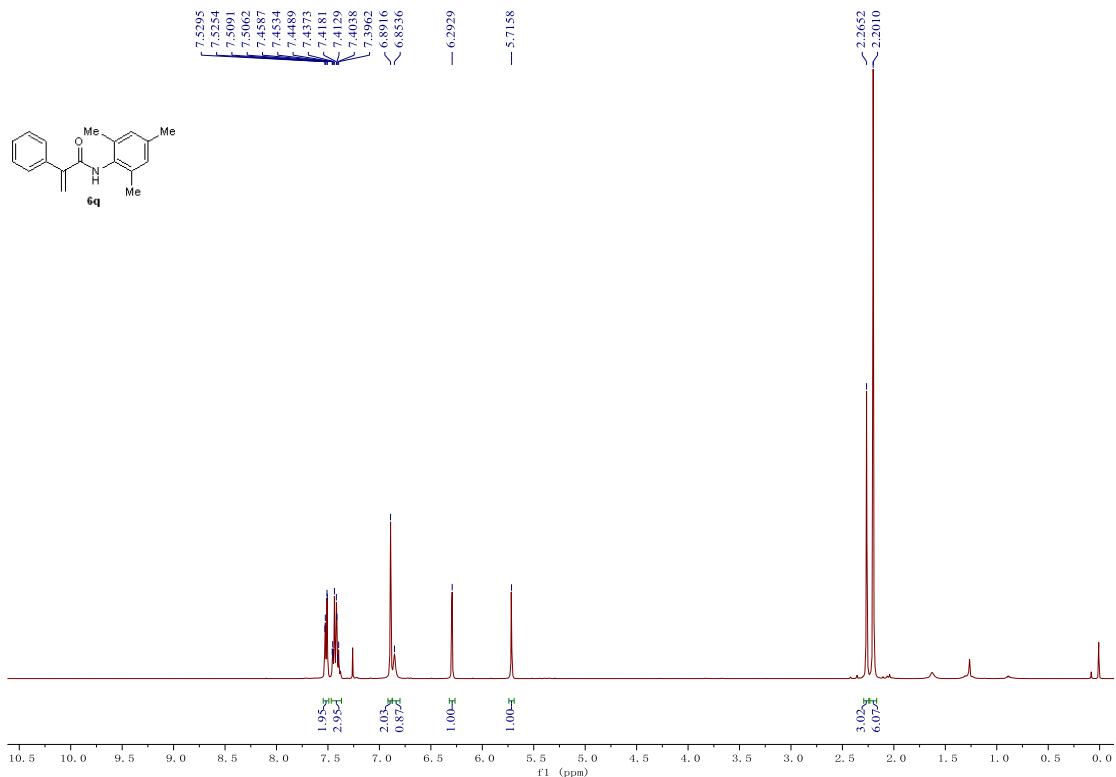
¹H NMR spectrum of **6p**



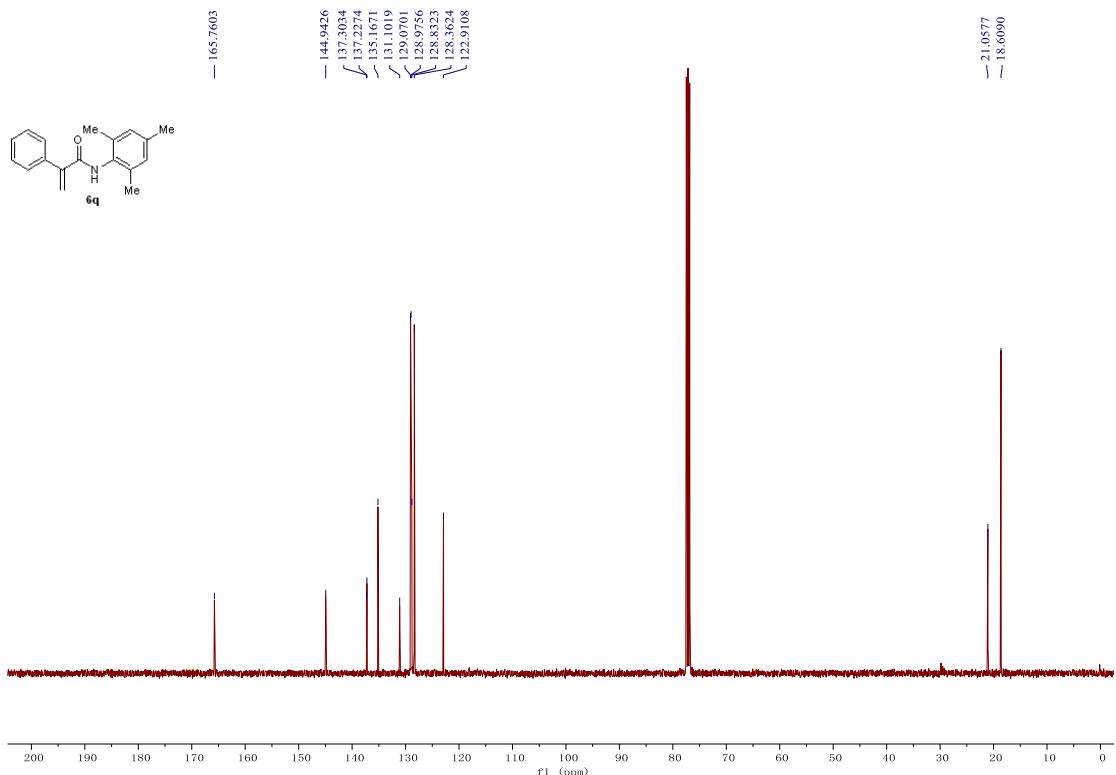
¹H NMR spectrum of **6p**



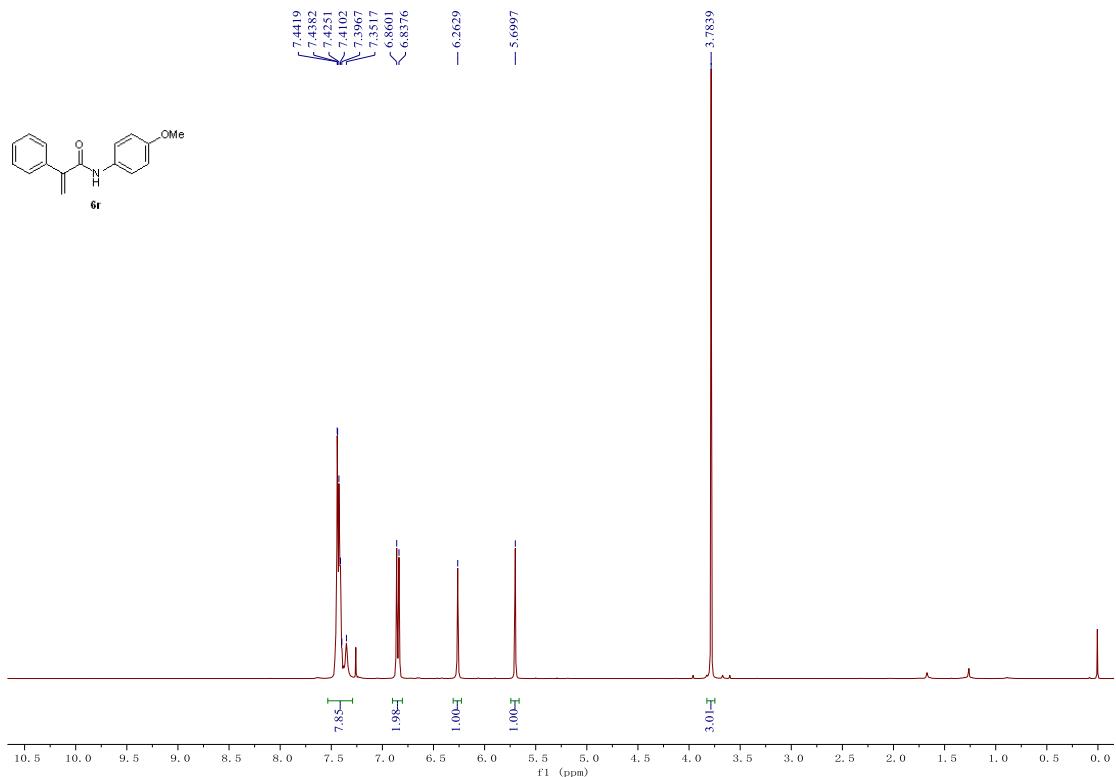
¹H NMR spectrum of **6q**



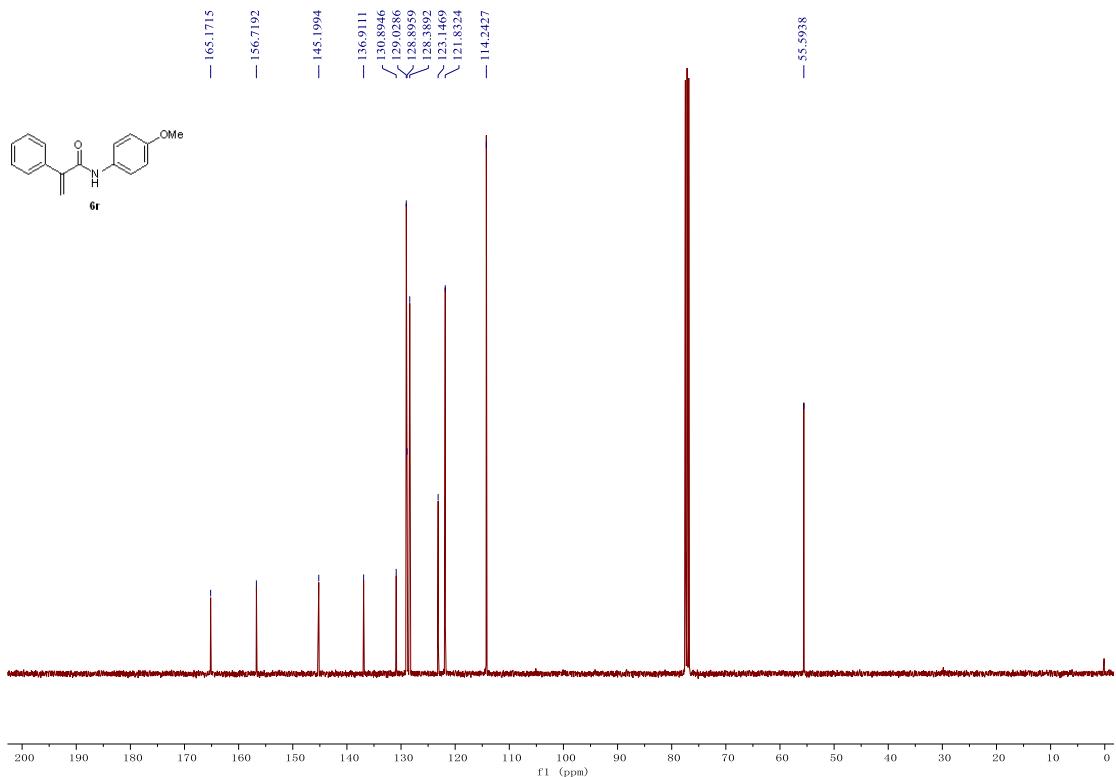
¹³C NMR spectrum of **6q**



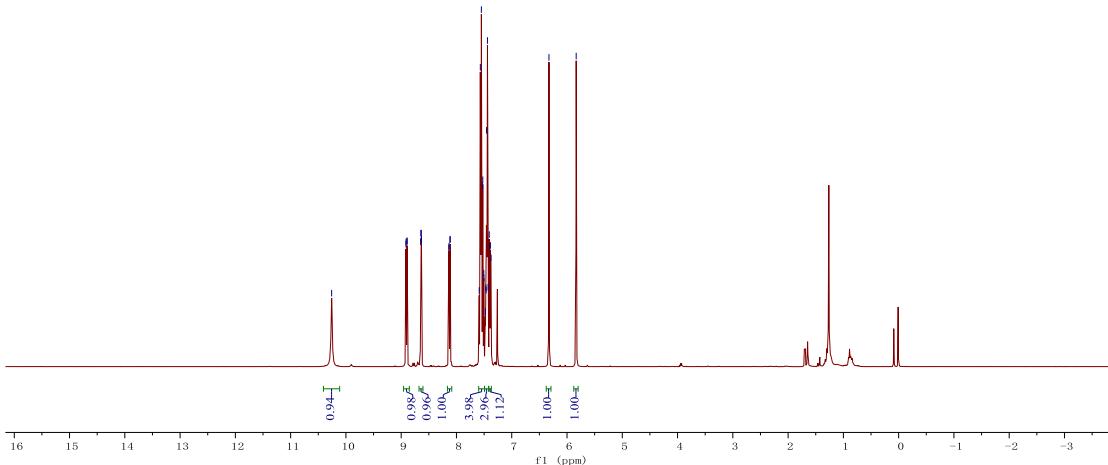
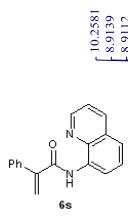
¹H NMR spectrum of **6r**



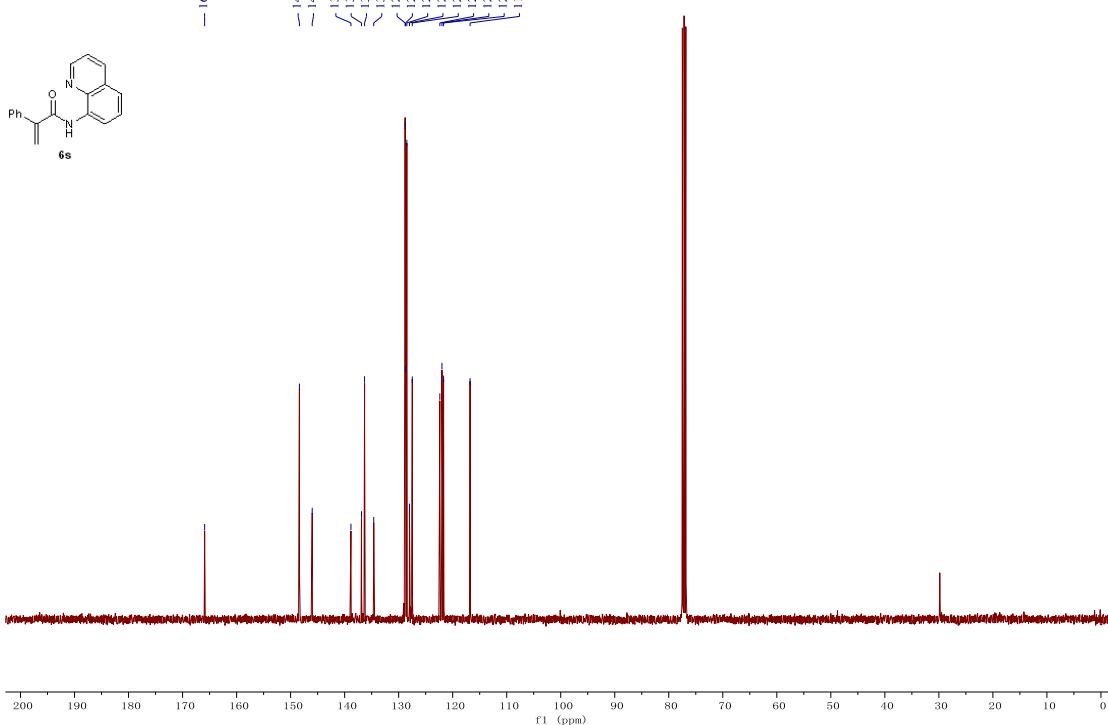
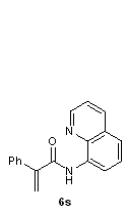
^{13}C NMR spectrum of **6r**



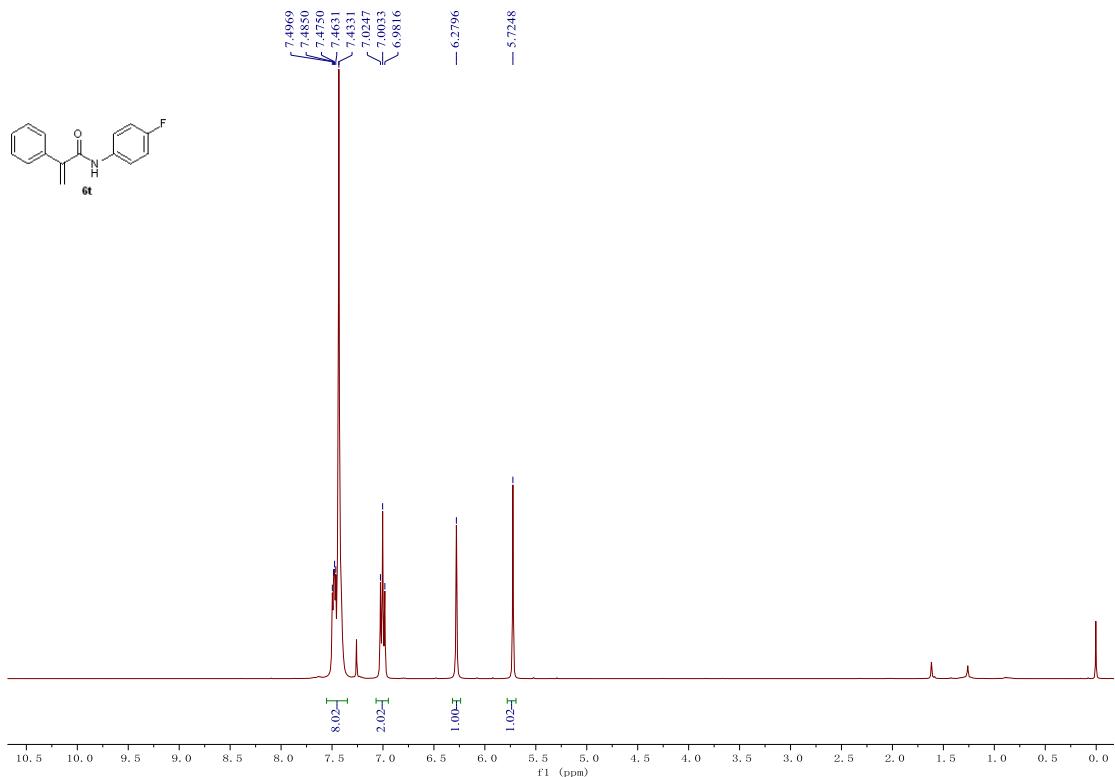
^1H NMR spectrum of **6s**



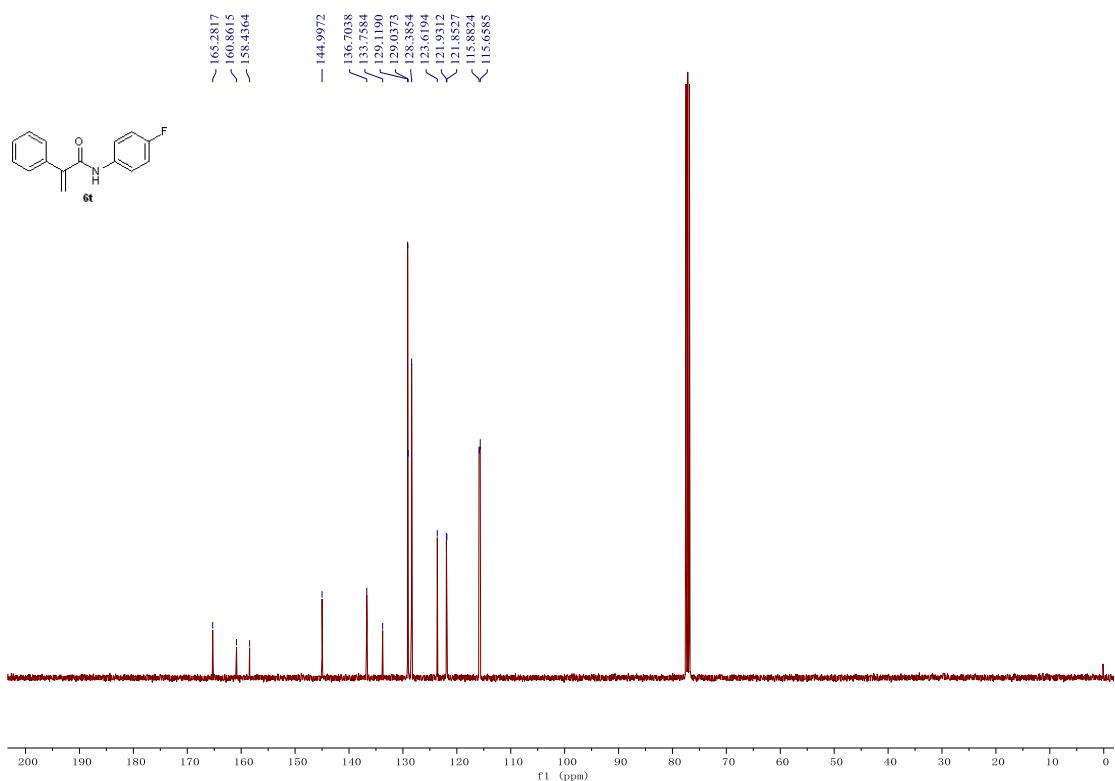
¹³C NMR spectrum of **6s**



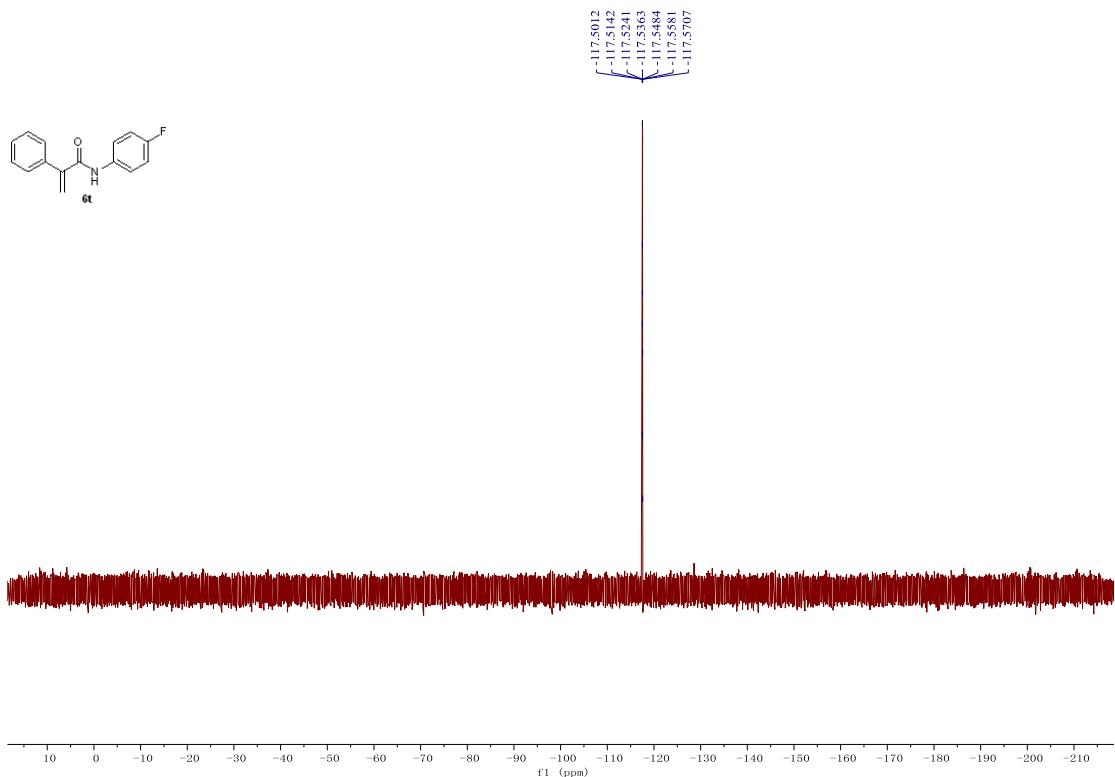
¹H NMR spectrum of **6t**



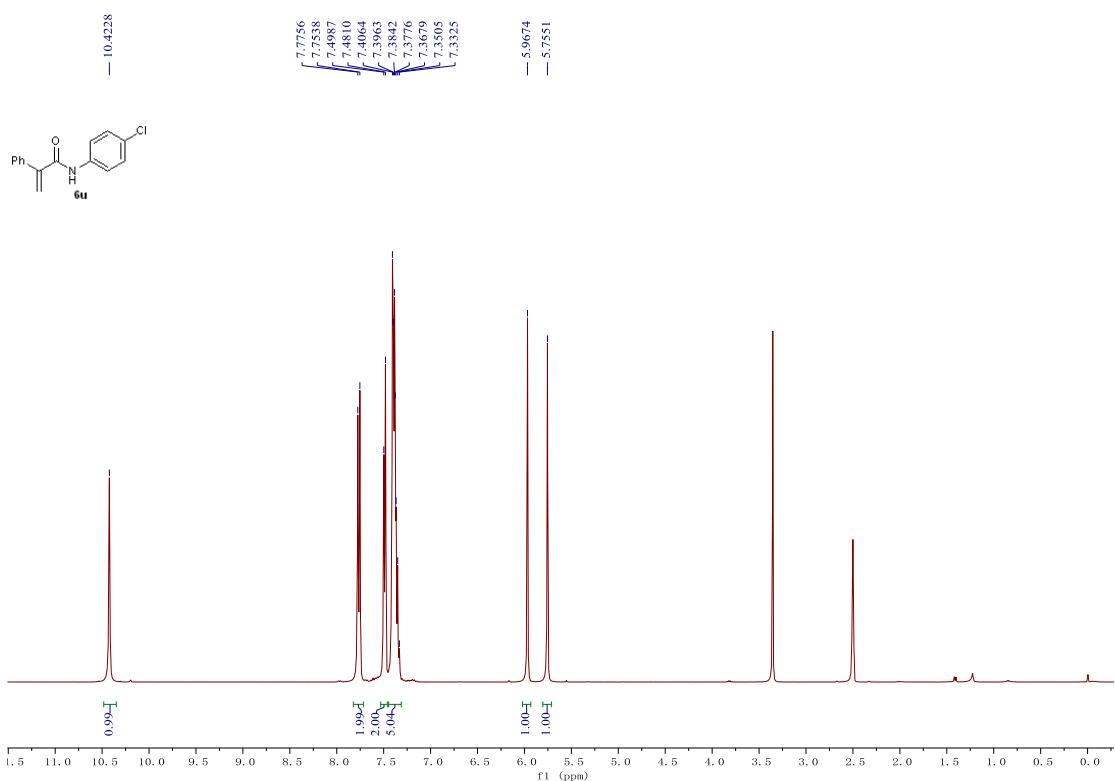
¹³C NMR spectrum of **6t**



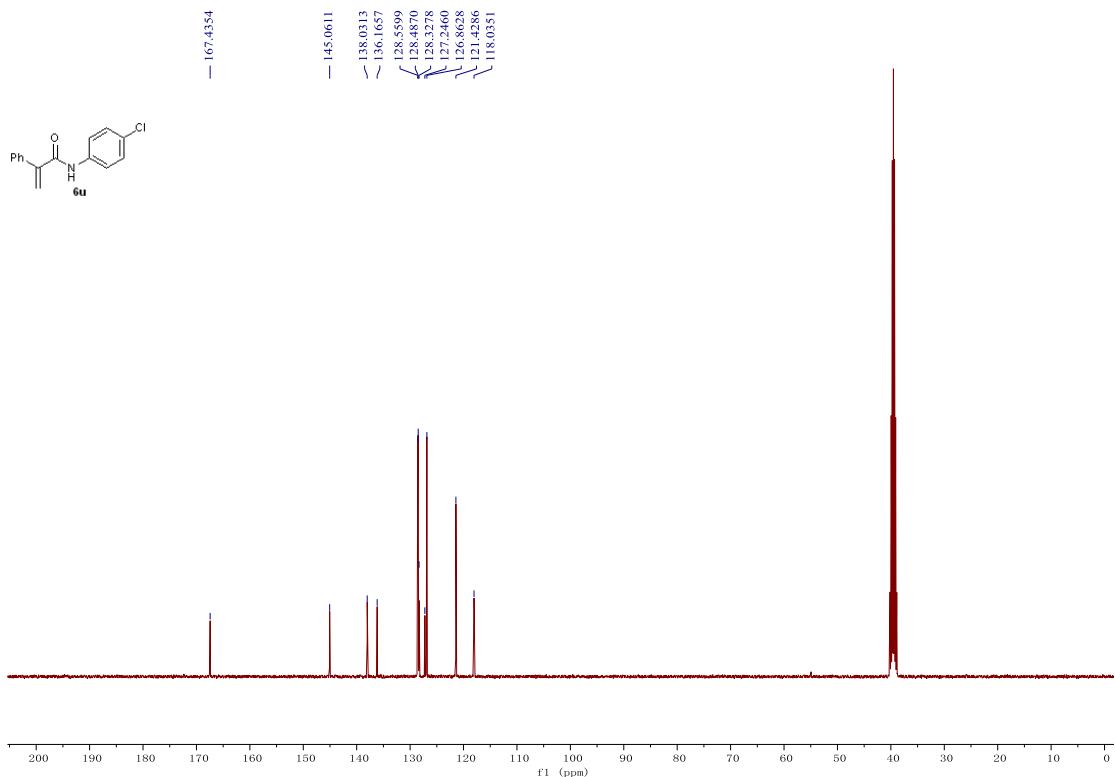
¹⁹F NMR spectrum of **6t**



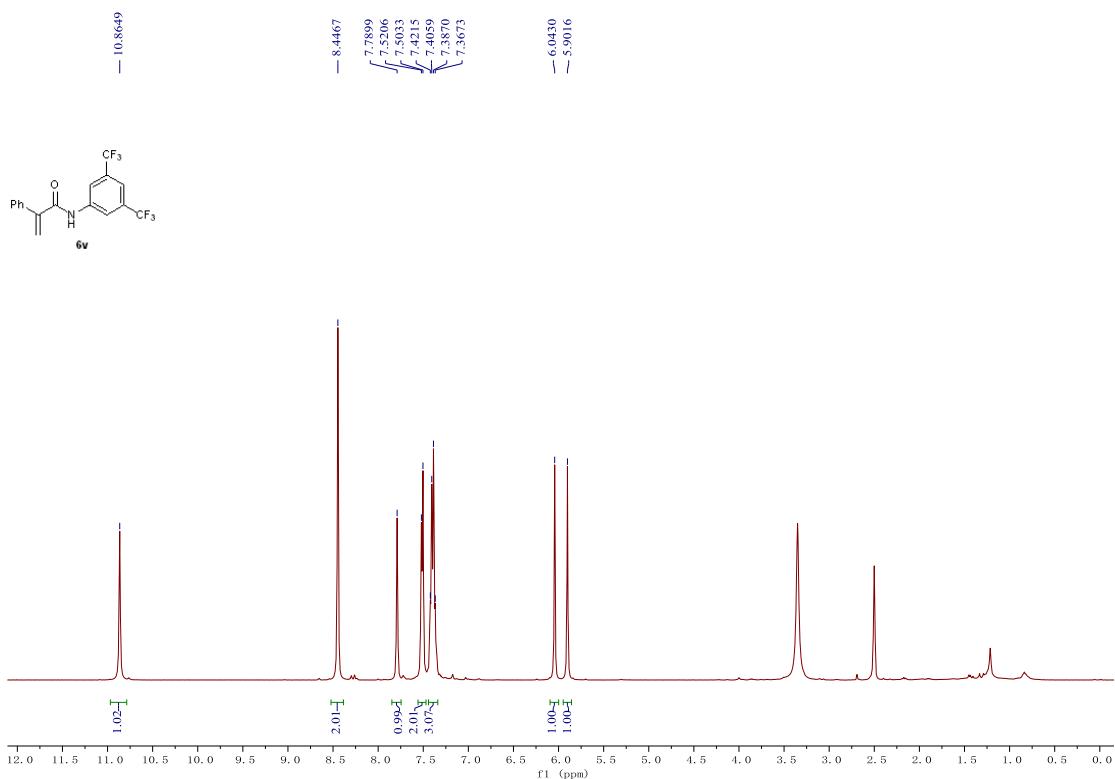
¹H NMR spectrum of **6u**



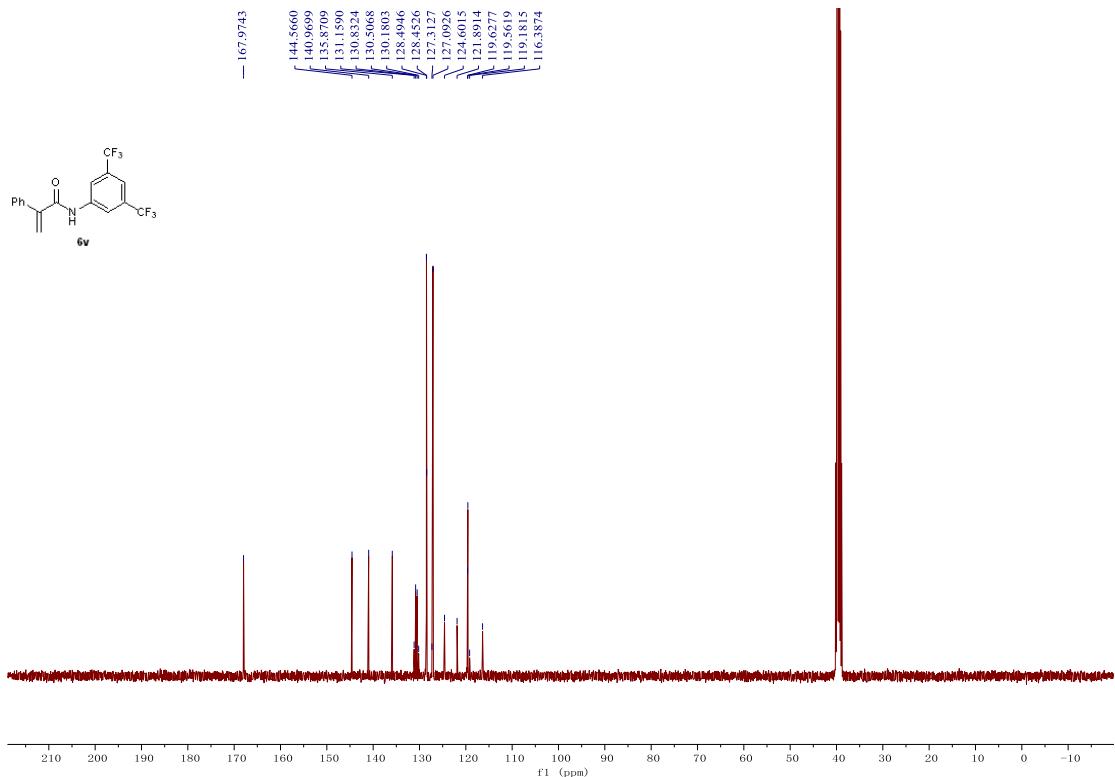
¹³C NMR spectrum of **6u**



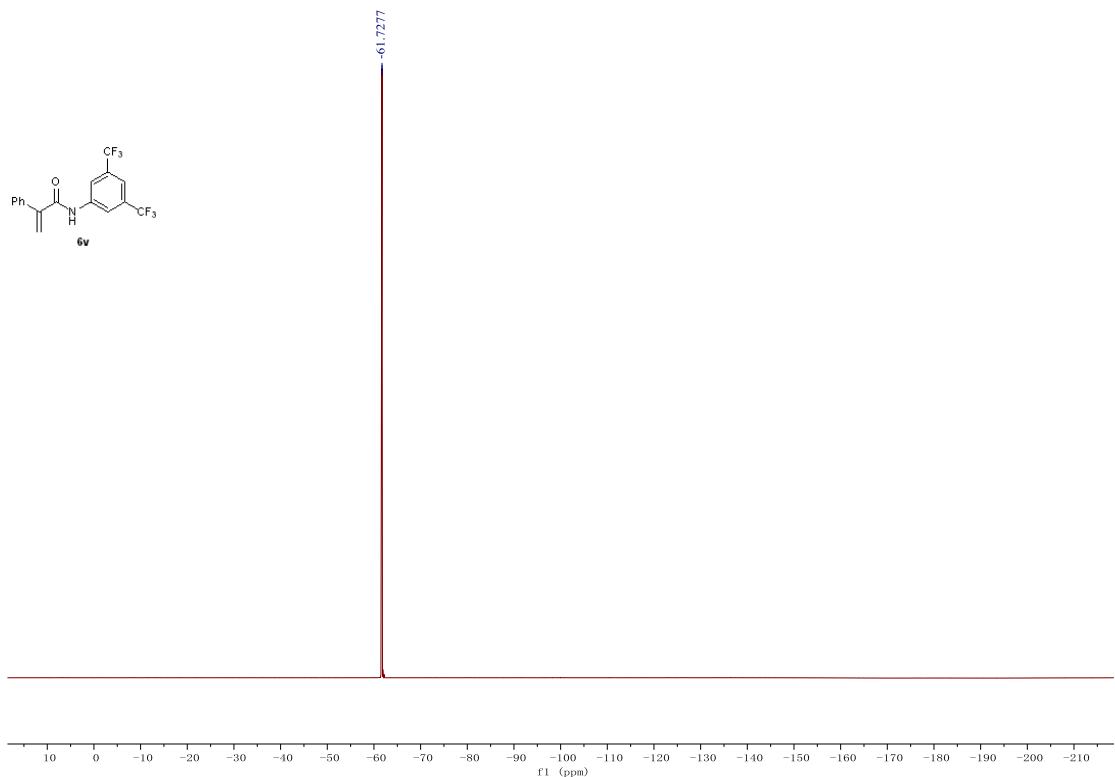
¹H NMR spectrum of **6v**



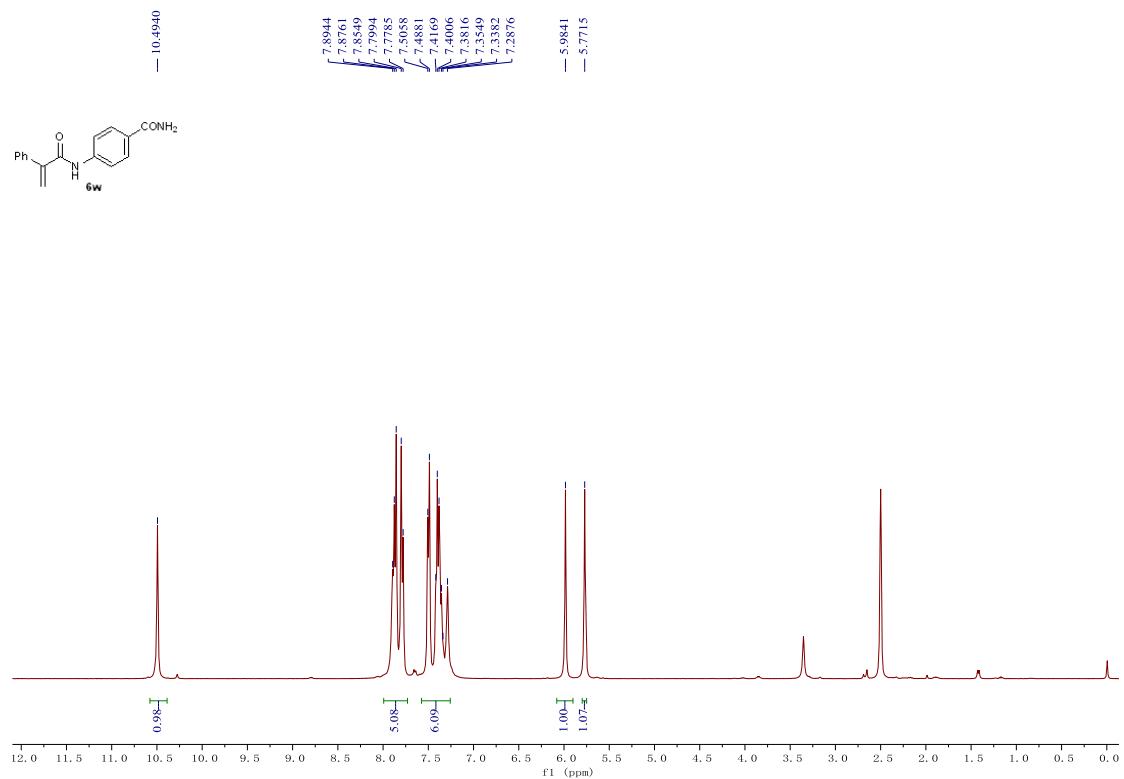
¹³C NMR spectrum of **6v**



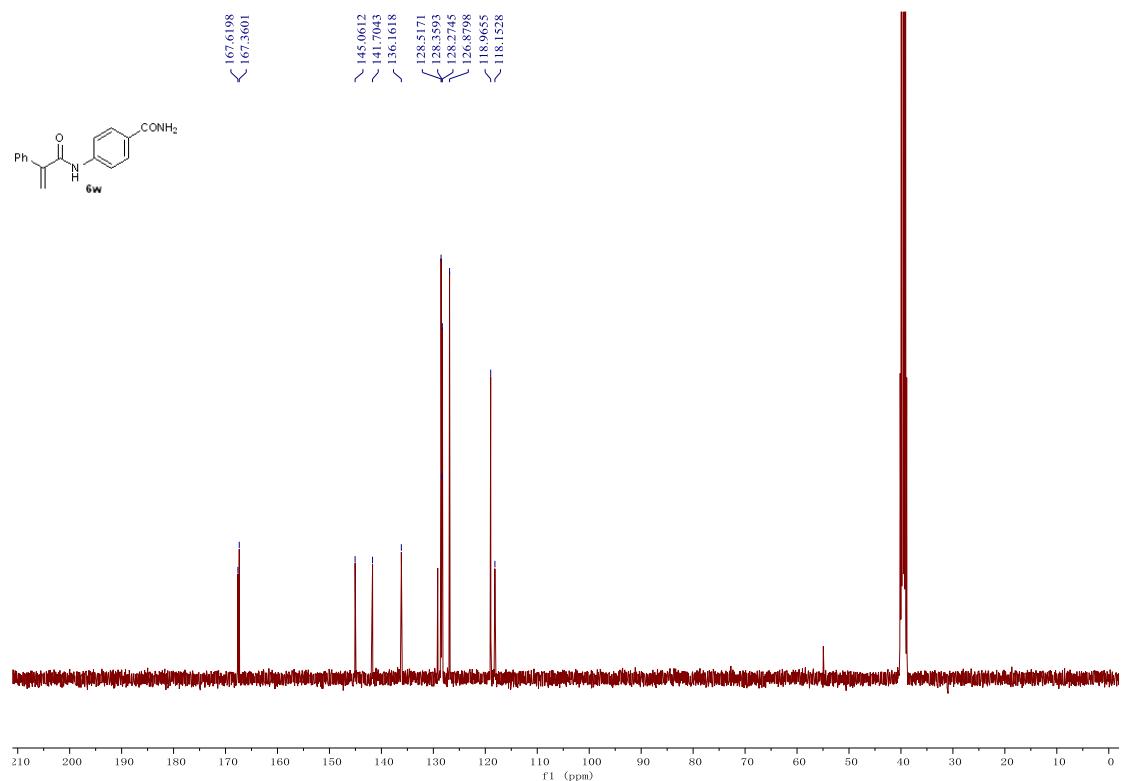
¹⁹F NMR spectrum of **6v**



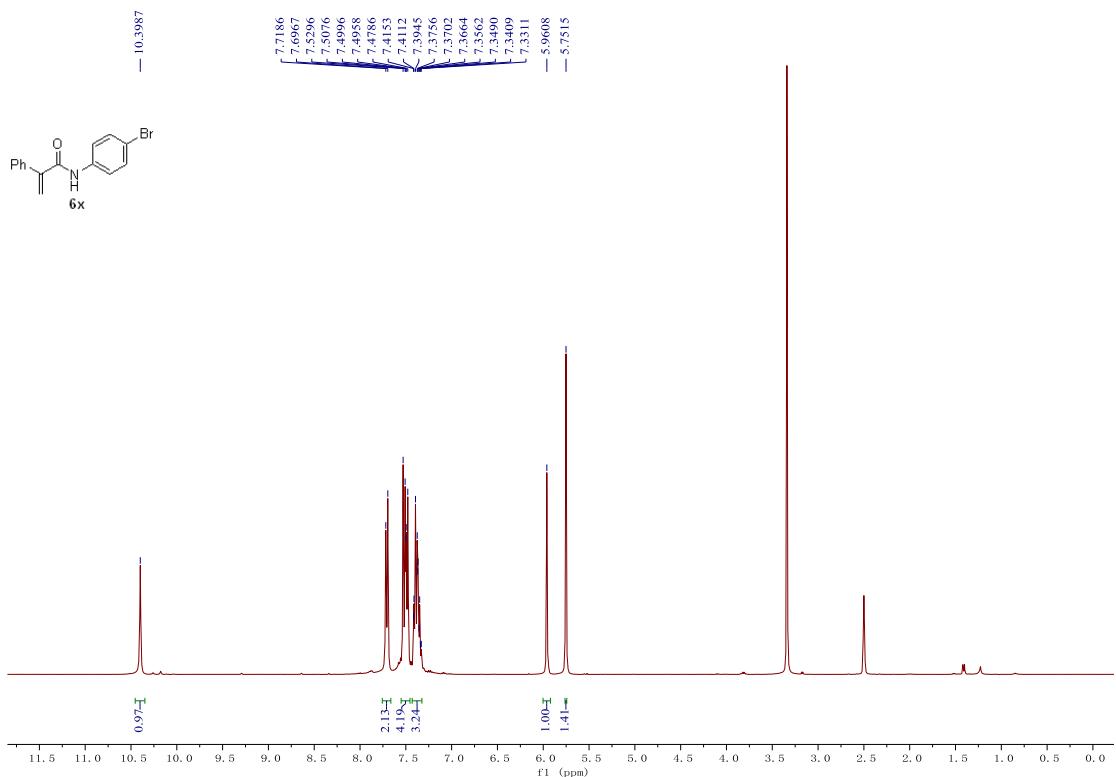
¹H NMR spectrum of **6w**



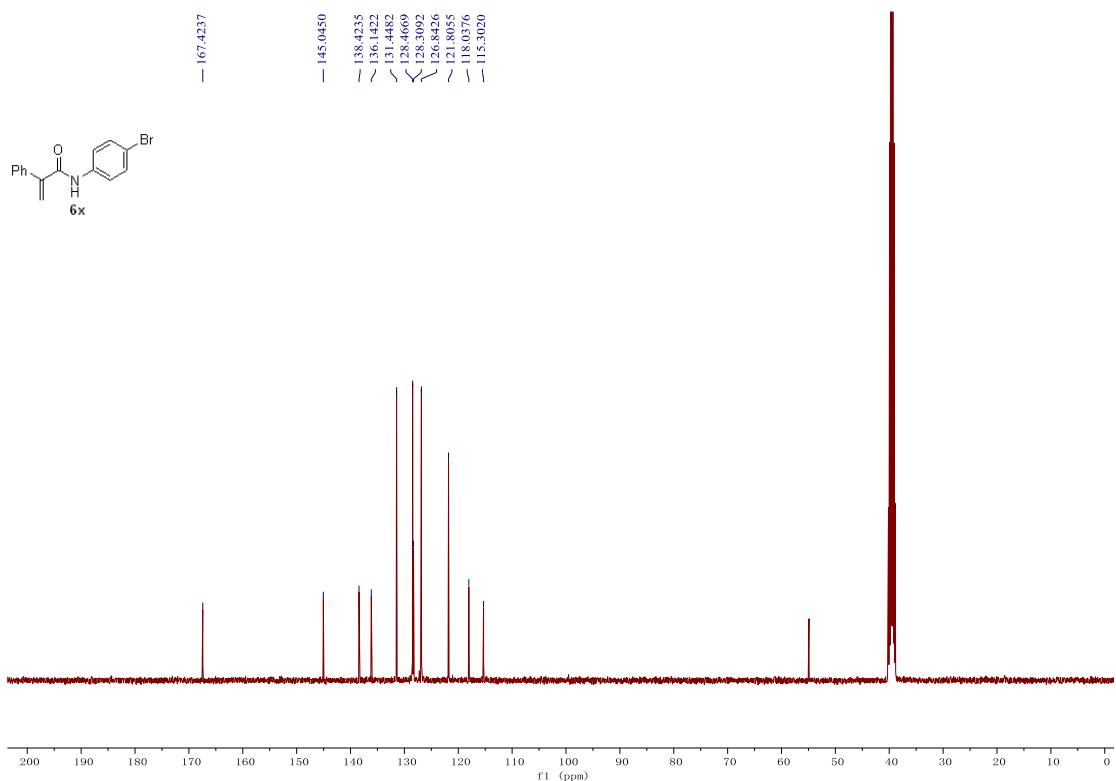
¹³C NMR spectrum of **6w**



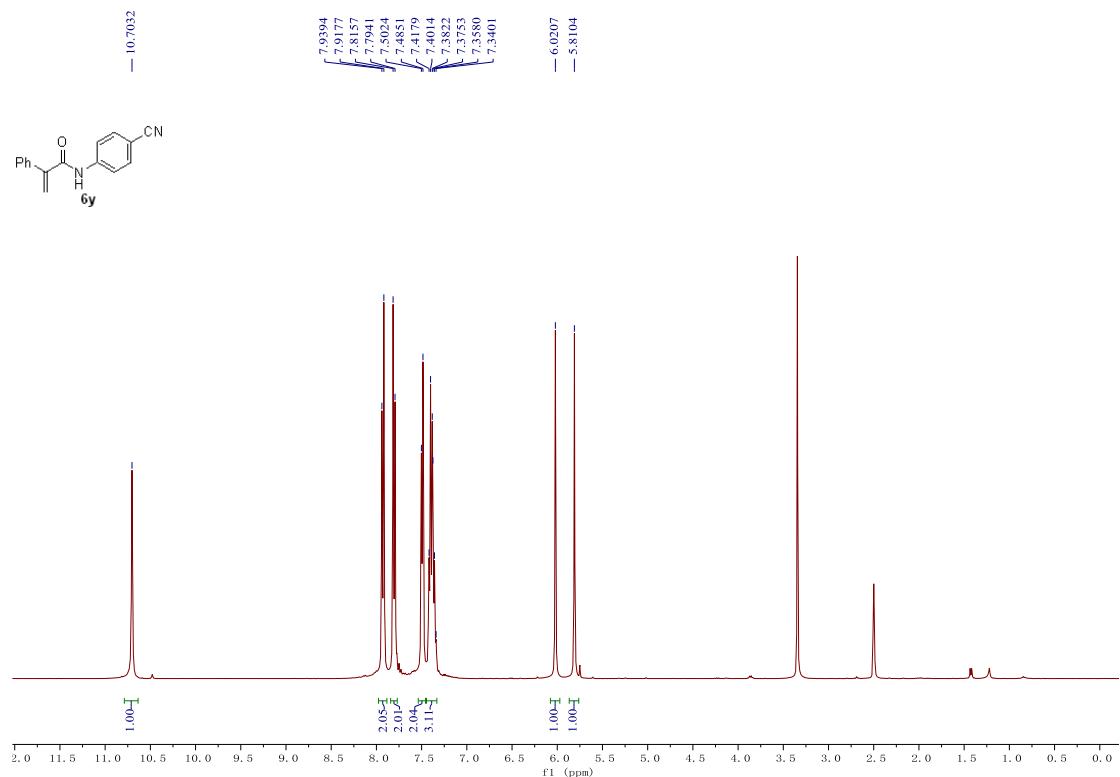
¹H NMR spectrum of **6x**



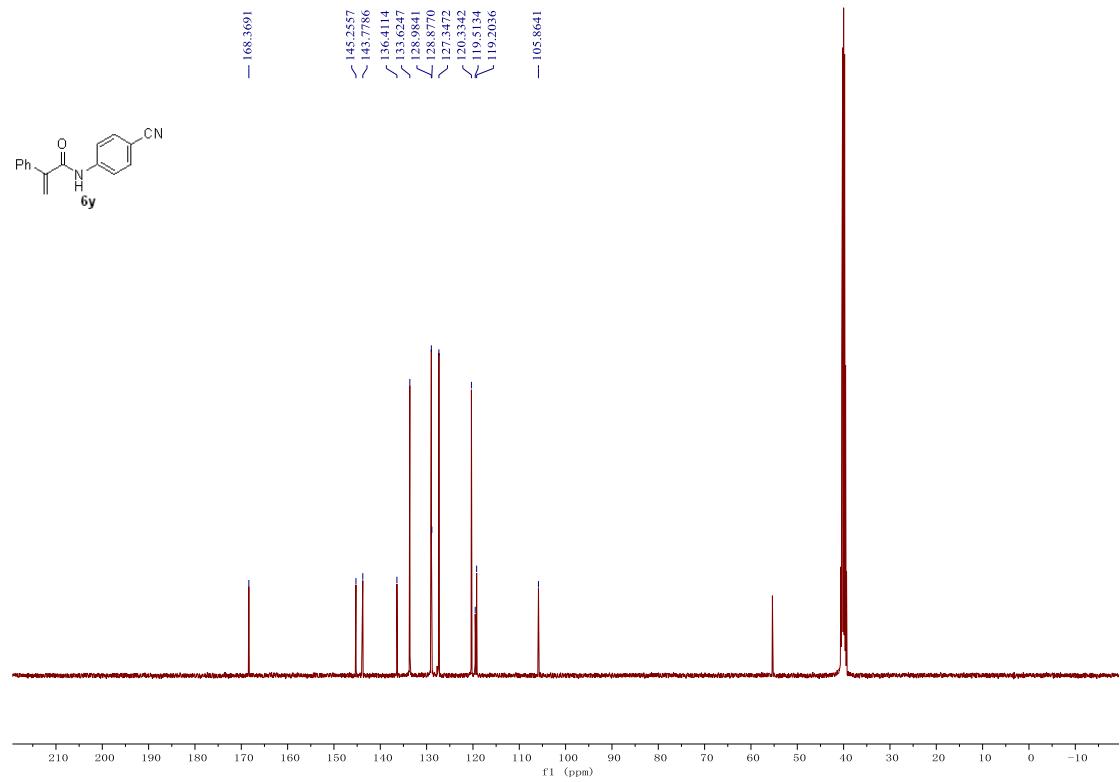
¹³C NMR spectrum of **6x**



¹H NMR spectrum of **6y**



¹³C NMR spectrum of **6y**



12. References

1. H. Liu, N. Yan, P. J. Dyson, *Chem. Commun.* **2014**, *50*, 7848–7851.
2. T. Xu, F. Sha, H. Alper, *J. Am. Chem. Soc.* **2016**, *138*, 6629–6635.
3. J. Liu, H. Li, A. Spannenberg, R. Franke, R. Jackstell, M. Beller, *Angew. Chem. Int. Ed.* **2016**, *55*, 13544–13548.
4. J. Zhu, B. Gao, H. Huang, *Org. Biomol. Chem.* **2017**, *15*, 2910–2913.
5. X. Zhou, G. Zhang, B. Gao, H. Huang, *Org. Lett.* **2018**, *20*, 2208–2212.
6. B. Gao, G. Zhang, X. Zhou, H. Huang, *Chem. Sci.* **2018**, *9*, 380–386.
7. Y.-H. Yao, H.-Y. Yang, M. Chen, F. Wu, X.-X. Xu, Z.-H. Guan, *J. Am. Chem. Soc.* **2021**, *143*, 85–91.
8. H.-Y. Yang, Y.-H. Yao, M. Chen, Z.-H. Ren, Z.-H. Guan, *J. Am. Chem. Soc.* **2021**, *143*, 7298–7305.
9. Z. Wang, C. B. Reddy, X. Zhou, J. J. Ibrahim, Y. Yang, *ACS Appl. Mater. Interfaces* **2020**, *12*, 53141–53149.
10. T. Tozawa, J. T. A. Jones, S. I. Swamy, S. Jiang, D. J. Adams, S. Shakespeare, R. Clowes, D. Bradshaw, T. Hasell, S. Y. Chong, C. Tang, S. Thompson, J. Parker, A. Trewin, J. Bacsa, A. M. Z. Slawin, A. Steiner, A. I. Cooper, *Nat. Mater.* **2009**, *8*, 973–978.
11. B. Mondal, K. Acharyya, P. Howlader, P. S. Mukherjee, *J. Am. Chem. Soc.* **2016**, *138*, 1709–1716.
12. C. Liu, C. He, W. Shi, M. Chen, A. Lei, *Org. Lett.* **2007**, *9*, 5601–5604.
13. W. Ye, J. Mo, T. Zhao, B. Xu, *Chem. Commun.* **2009**, 3246–3248.
14. S. Liu, H. Wang, X. Dai, F. Shi, *Green Chem.* **2018**, *20*, 3457–3462.
15. T. Maki, K. Ishihara, H. Yamamoto, *Org. Lett.* **2006**, *8*, 1431–1434.
16. M. Badioli, R. Ballini, M. Bartolacci, G. Bosica, E. Torregiani, E. Marcantoni, *J. Org. Chem.* **2002**, *67*, 8938–8942.
17. Y. Liu, C.-L. Wang, H.-M. Xia, Z. Wang, Y.-F. Wang, *Org. Biomol. Chem.* **2019**, *17*, 6153–6157.
18. X. Zhao, X. Feng, F. Chen, S. Zhu, F.-L. Qing, L. Chu, *Angew. Chem. Int. Ed.* **2021**, *60*, 26511–26517.
19. L. Wu, F. Wang, P. Chen, G. Liu, *J. Am. Chem. Soc.* **2019**, *141*, 1887–1892.
20. Z.-H. Luan, J.-P. Qu, Y.-B. Kang, *J. Am. Chem. Soc.* **2020**, *142*, 20942–20947.
21. H. Wang, H. Yuan, X. Wang, J. Zhao, D. Wei, F. Shi, *Adv. Synth. Catal.* **2020**, *362*, 2348.
22. Q. Zhao, T. Poisson, X. Pannecoucke, J.-P. Bouillon, T. Basset, *Org. Lett.* **2017**, *19*, 5106–5109.
23. Z. Su, Y. Feng, R. Zou, X. Qiu, J. Wang, C. Tao, *Chem. Commun.* **2020**, *56*, 7483–7486.