

Supporting Information

Visible-light-mediated Aerobic Ritter-type C-H Amination of Diarylmethane using DDQ/tert-Butyl Nitrite

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1 General Information

Unless stated otherwise, all reagents were purchased from commercial sources and used without further purification. Thin layer chromatography (TLC) was carried out using Merck TLC silica gel 60 sheet and visualized with ultraviolet light (254/365 nm). Flash column chromatography (FCC) was performed on silica gel (200-300 mesh) as the stationary phase and the solvents employed were of analytical grade. The air, oxygen, nitrogen and helium cylinder were supplied by Hangzhou Jingong Special Gases.

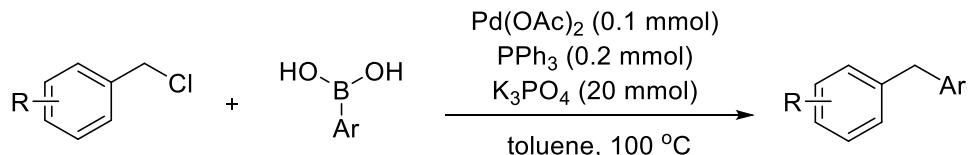
¹H NMR spectra and ¹³C NMR spectra were recorded on a Bruker AVANCE^{III} 500 (500 MHz) spectrometer and Bruker AVANCE^{III} 500 (126 MHz) spectrometer at 25 °C, respectively.

Gas chromatography analysis was carried out using Agilent 7890A with AT•SE-54 GC capillary column (30 m × 250 µm × 0.33 µm) was employed for all the separations using the following conditions: initial column temperature 100 °C; initial hold time 2 min; final temperature 280 °C; hold time 5 min; temperature ramp 15 °C/min; detector temperature: 300 °C, injection temperature 280 °C; injection volume 1 µL; split ratio 30:1; column flow rate 1 mL/min. The effluent was combusted in a H₂/Air flame and detected using an FID (flame ionization detector).

Gas chromatography-mass spectrometry (GC-MS) was carried out using Thermo Fisher Trace ISQ with TG-5MS GC capillary column (60 m × 250 µm × 0.25 µm) was employed for all the separations using the following conditions: the initial column temperature 100 °C, initial held time 2 min; final temperature was increased to 280 °C at 15 °C/min and held for 20 min. Injection temperature 250 °C, ion source temperature 200 °C, EI ionization method with electron energy 70 eV and mass-to-charge ratio: 40-500.

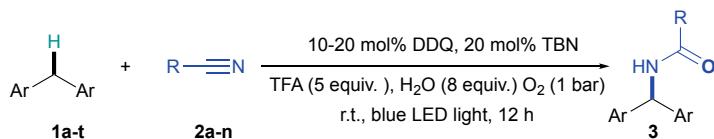
2 Experimental Details

2.1 General procedure for the synthesis of various diarylmethanes



Substituted diarylmethanes (**1b-t**) were synthesised using the method derived from the literature¹: To a 100 mL dry Schlenk flask, 4.2 g of tripotassium phosphate (20 mmol), substituted phenylboronic acid (15 mmol), 52.5 mg of Triphenylphosphine (0.2 mmol), 22.5 mg palladium acetate (0.1 mmol), substituted benzyl chloride (10 mmol) and 35 mL toluene solvent were charged. After nitrogen gas was introduced into the Schlenk bottle as protective gas, the apparatus was placed in a 100 °C oil bath. The reaction was monitored by TLC thin layer chromatography and gas chromatography. After the reaction was completed, reaction was cooled to room temperature. Methyl tert-butyl ether (20 mL), 1 M NaOH aqueous solution (10 mL) and saturated brine (20 mL) were added to the reaction mixture. Then the reaction mixture was transferred to a separating funnel. The aqueous layer was separated and back extracted with methyl tert-butyl ether (20 mL) twice. Then the combined organic layers were then dried over Na₂SO₄, filtered and concentrated under reduced pressure. The crude material was then purified by silica gel flash chromatography using the noted solvent systems.

2.1 General procedure for visible-light-mediated Ritter-type C-H amination using DDQ/tert-butyl nitrite



To a reaction tube DDQ (10-20 mol%, 11-22 mg), diarylmethane (0.5 mmol), trifluoroacetic acid (5.0 equiv., 180 μL), H_2O (8 equiv. 72 μL) and nitrile (2 mL, ~20 equiv.) were added. After the reaction tube was flushed with oxygen, tert-butyl nitrite (20 mol%, 12 μL) was quickly injected via a micro syringe. Then reaction tube was sealed with a tetrafluoroethylene stopper and placed in a blue LED light-emitting device (18 W). After reaction was stirred at room temperature for 12 hours, the reaction mixture was quenched and neutralized with potassium carbonate solution. The reaction mixture was then transferred to a separating funnel, the aqueous layer was separated and back extracted with ethyl acetate (30 mL) twice. The combined organic layers were dried over NaSO_4 , filtered and concentrated under reduced pressure. The crude material was then purified by silica gel flash chromatography using the noted solvent systems.

Note: For nitriles (e.g., 4-chlorobenzonitrile, 4-bromobenzonitrile) that are solid at room temperature, reaction was carried out with 15 equiv. of corresponding nitrile in DCE (2 mL).

Table S1. Effect of DDQ/TBN loadings on aerobic Ritter-type C-H amination

	+			
1a		2a		3aa
Entry	DDQ [mol%]	TBN [mol%]	Yield [%] ^[a]	
1	10	10	61	
2	10	5	52	
3	5	10	46	
4	5	5	41	

Reaction conditions: reaction was performed with **1a** (0.5 mmol), **2a** (2 mL, as reagent/solvent), DDQ (amount as specified), TBN (amount as specified), TFA (5 equiv., 2.5 mmol), and H₂O (4 mmol, 8 equiv.) with blue LED light and oxygen balloon at room temperature for 12 h.

[a] Yields were determined by GC using biphenyl as internal standard.

Table S2. Screening of reaction conditions

	+			
1a		2a		3aa
Entry	Variation from the standard conditions		Yield [%] ^[a]	
1	none		61	
2	40 °C		39	
3	60 °C		40	
4	air (1 bar)		31	
5	50% O ₂ in N ₂ (1 bar)		35	
6	10 mol% isobutyl nitrite		44	
7	10 mol% butyl nitrite		31	
8	LED plain light (15 W)		51	
9	red LED light (15 W)		21	
10	blue LED light (10W)		35	
11	bule LED light (30 W)		59	

Standard conditions: reaction was performed with **1a** (0.5 mmol), **2a** (2 mL, as reagent/solvent), DDQ (10 mol%, 0.10 mmol), TBN (10 mol%, 0.10 mmol), TFA (5 equiv., 2.5 mmol), and H₂O (4 mmol, 8 equiv.) with blue LED light (18 W) and oxygen balloon at room temperature for 12 h.

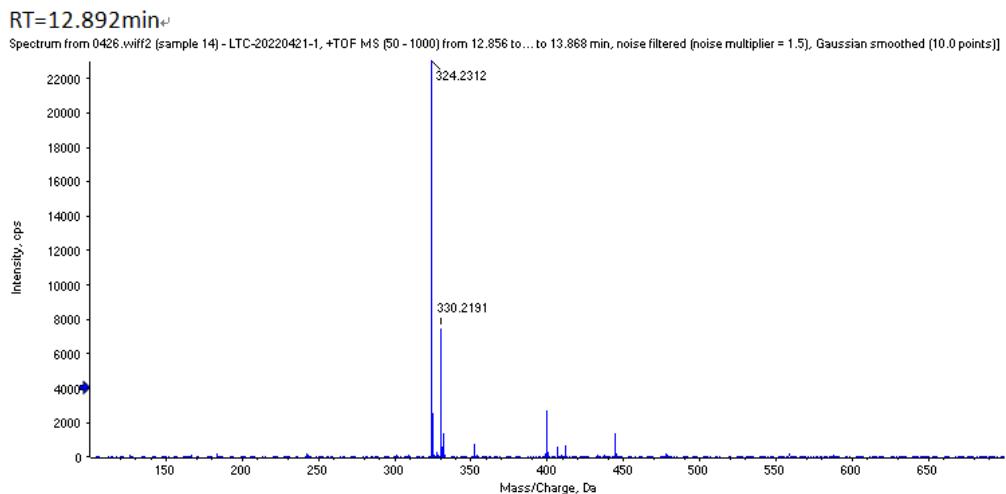
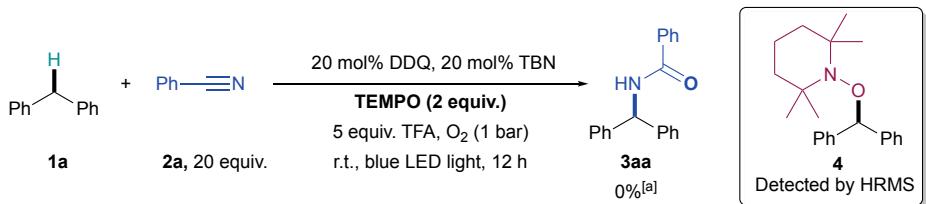


Figure S1. Confirmation of compound **4** by HPLC-MS (ESI). Calcd. for $[M+H]^+$ $C_{22}H_{30}NO$: 324.2322, found: 324.2312, error (ppm): -3.1.

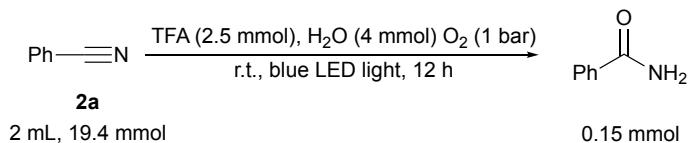
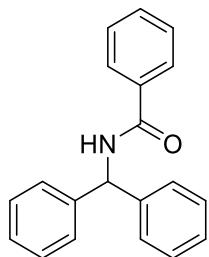
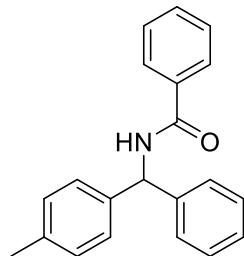


Figure S2. Study of hydrolysis of benzonitrile.

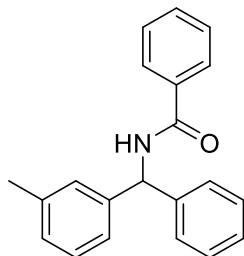
3 Products Characterization data



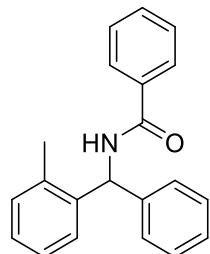
N-benzhydrylbenzamide (3aa): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3aa** as white solid. Yield: 76%, 109 mg. m.p.: 168-169 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.78 (d, *J* = 7.1 Hz, 2H), 7.47 (t, *J* = 8.6 Hz, 1H), 7.39 (t, *J* = 10.0 Hz, 2H), 7.33-7.30 (m, 4H), 7.28-7.22 (m, 6H), 6.72 (d, *J* = 7.5 Hz, 1H), 6.42 (d, *J* = 7.9 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 166.5, 141.5, 134.3, 131.7, 128.8, 128.6, 127.6, 127.5, 127.0, 57.5. NMR data is consistent with literature values.² MS (EI), m/z 287.13 [M⁺, 70%], 105.16 [100%].



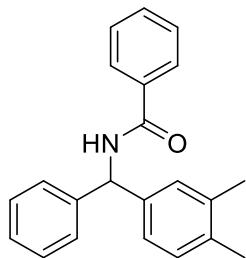
N-(phenyl(p-tolyl)methyl)benzamide (3ba): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3ba** as white solid. Yield: 85%, 128 mg. m.p.: 165-166 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.80 (d, *J* = 7.5 Hz, 2H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.34-7.26 (m, 5H), 7.18-7.13 (m, 4H), 6.74 (d, *J* = 7.1 Hz, 1H), 6.40 (d, *J* = 7.7 Hz, 1H), 2.33 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 166.5, 141.7, 138.6, 137.3, 134.4, 131.6, 129.4, 128.7, 128.6, 127.5, 127.5, 127.4, 127.1, 57.2, 21.1. NMR data is consistent with literature values.³ MS (EI), m/z 301.19 [M⁺, 40%], 105.16 [100%].



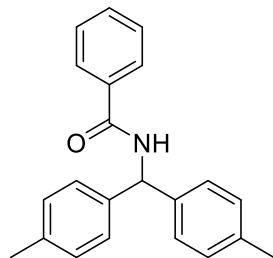
N-(phenyl(m-tolyl)methyl)benzamide (3ca): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3ca** as white solid. Yield: 82%, 123 mg. m.p.: 163-164 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.84 (d, J = 7.4 Hz, 2H), 7.53 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.7 Hz, 2H), 7.35 (t, J = 7.0 Hz, 2H), 7.31-7.28 (m, 3H), 7.22 (d, J = 7.0 Hz, 4H), 6.71 (d, J = 7.4 Hz, 1H), 6.64 (d, J = 7.7 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.5, 141.6, 141.5, 138.4, 134.3, 131.6, 128.7, 128.6, 128.5, 128.4, 128.3, 127.5, 127.1, 124.6, 57.5, 21.5. MS (EI), m/z 301.18 [M^+ , 30%], 105.01 [100%]. HRMS (ESI): m/z [M-H] $^-$ calc. for $[\text{C}_{21}\text{H}_{18}\text{NO}]^-$ 300.1394, found 300.1395.



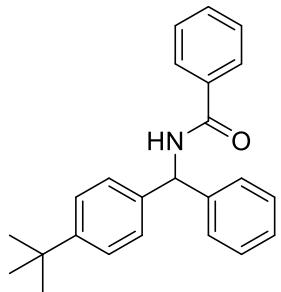
N-(phenyl(o-tolyl)methyl)benzamide (3da): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3da** as white solid. Yield: 80%, 120 mg. m.p.: 163-164 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.78 (d, J = 7.4 Hz, 2H), 7.47 (t, J = 7.4 Hz, 1H), 7.39 (t, J = 7.7 Hz, 2H), 7.30 (t, J = 7.0 Hz, 2H), 7.26-7.22 (m, 3H), 7.17 (d, J = 7.0 Hz, 4H), 6.65 (d, J = 7.4 Hz, 1H), 6.59 (d, J = 7.7 Hz, 1H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.3, 141.0, 139.5, 136.5, 134.3, 131.7, 130.9, 128.7, 128.6, 127.6, 127.5, 127.4, 127.1, 126.8, 126.2, 54.5, 19.5. NMR data is consistent with literature values.³ MS (EI), m/z 301.16 [M^+ , 30%], 105.01 [100%].



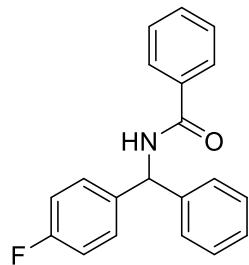
N-((3,4-dimethylphenyl)(phenyl)methyl)benzamide (3ea): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3ea** as white solid. Yield: 64%, 101 mg. m.p.: 150-151 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.81 (d, *J* = 7.1 Hz, 2H), 7.50-7.48 (m, 1H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.34-7.25 (m, 5H), 7.10-7.01 (m, 3H), 6.71 (d, *J* = 7.6 Hz, 1H), 6.37 (d, *J* = 7.8 Hz, 1H), 2.23 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 166.5, 141.7, 139.0, 137.0, 136.0, 134.4, 131.6, 130.0, 128.9, 128.7, 128.6, 127.4, 127.3, 127.1, 124.9, 57.3, 19.9, 19.5. HRMS (ESI): m/z [M-H]⁻ calc. for [C₂₂H₂₀NO]⁻ 314.1550, found 314.1549.



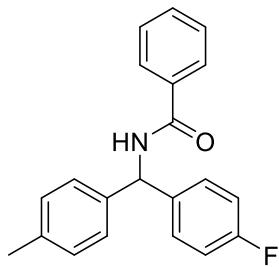
N-(di-p-tolylmethyl)benzamide (3fa): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3fa** as white solid. Yield: 78%, 123 mg. m.p.: 182-183 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.79 (d, *J* = 8.6 Hz, 2H), 7.48 (t, *J* = 6.3 Hz, 1H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.18-7.12 (m, 8H), 6.71 (d, *J* = 7.6 Hz, 1H), 6.36 (d, *J* = 7.8 Hz, 1H), 2.32 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 166.4, 138.8, 137.1, 134.4, 131.6, 129.4, 128.6, 127.4, 127.1, 57.0, 21.0. NMR data is consistent with literature values.⁴ MS (EI), m/z 315.19 [M⁺, 60%], 105.06 [100%].



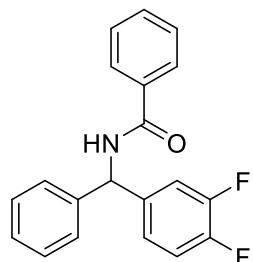
N-((4-(tert-butyl)phenyl)(phenyl)methyl)benzamide (3ga): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3ga** as white solid. Yield: 81%, 138 mg. m.p.: 166-168 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.80 (d, J = 7.1 Hz, 2H), 7.48 (t, J = 6.2 Hz, 1H), 7.40 (t, J = 7.9 Hz, 2H), 7.35-7.32 (m, 6H), 7.26-7.20 (m, 3H), 6.77 (d, J = 7.7 Hz, 1H), 6.42 (d, J = 7.8 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.5, 150.5, 141.6, 138.5, 134.4, 131.6, 128.7, 128.6, 127.4, 127.3, 127.1, 125.7, 57.2, 34.5, 31.3. HRMS (ESI): m/z [M-H] $^-$ calc. for $[\text{C}_{24}\text{H}_{24}\text{NO}]^-$ 342.1863, found 342.1866.



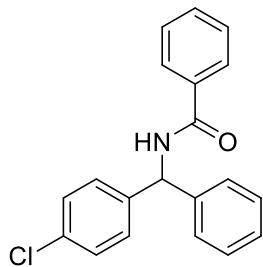
N-((4-fluorophenyl)(phenyl)methyl)benzamide (3ia): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ia** as white solid. Yield: 72%, 109 mg. m.p.: 167-168 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.81 (d, J = 7.2 Hz, 2H), 7.50 (t, J = 7.5 Hz, 1H), 7.41-7.24 (m, 9H), 7.11 (d, J = 7.8 Hz, 1H), 7.01 (t, J = 8.7 Hz, 2H), 6.42 (d, J = 7.9 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.7, 162.1 (d, J = 244.6 Hz), 141.3, 137.4 (d, J = 3.2 Hz), 134.1, 131.7, 129.2 (d, J = 8.1 Hz), 128.8, 128.6, 127.7, 127.5, 127.2, 115.5 (d, J = 21.4 Hz), 56.8. NMR data is consistent with literature values.³ MS (EI), m/z 305.15 [M $^+$, 30%], 105.02 [100%].



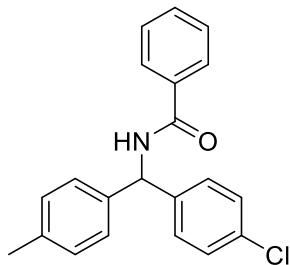
N-((4-fluorophenyl)(p-tolyl)methyl)benzamide (3ja): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ja** as white solid. Yield: 68%, 108 mg. m.p.: 182-183 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.81 (d, J = 7.2 Hz, 2H), 7.52 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.9 Hz, 1H), 7.28-7.26 (m, 2H), 7.17 (s, 4H), 7.02 (t, J = 8.7 Hz, 2H), 6.75 (d, J = 7.5 Hz, 1H), 6.39 (d, J = 7.7 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.5, 162.1 (d, J = 244.6 Hz), 138.4, 137.5, 134.2, 131.7, 129.5, 129.0 (d, J = 8.1 Hz), 128.6, 127.4, 127.1, 115.5 (d, J = 21.3 Hz), 56.6, 21.1. HRMS (ESI): m/z [M-H] $^-$ calc. for $[\text{C}_{21}\text{H}_{17}\text{FNO}]^-$ 318.1300, found 318.1304.



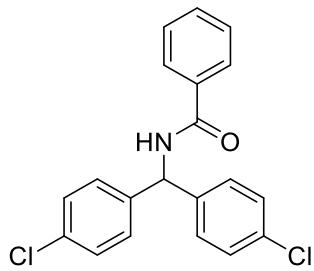
N-((3,4-difluorophenyl)(phenyl)methyl)benzamide (3ka): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ka** as white solid. Yield: 58%, 94 mg. m.p.: 176-178 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.80 (d, J = 7.2 Hz, 2H), 7.52 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.9 Hz, 2H), 7.38-7.30 (m, 3H), 7.25 (t, J = 4.0 Hz, 2H), 7.14-7.03 (m, 3H), 6.76 (d, J = 7.3 Hz, 1H), 6.36 (d, J = 7.6 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.6, 151.0 (dd, J = 93.2, 12.7 Hz), 149.0 (dd, J = 92.6, 12.5 Hz), 140.6, 138.5 (dd, J = 8.3, 4.2 Hz), 133.8, 131.9, 129.0, 128.7, 128.1, 127.5, 127.1, 123.4 (dd, J = 6.3, 3.6 Hz), 117.4 (d, J = 17.2 Hz), 116.4 (d, J = 17.9 Hz), 56.7. HRMS (ESI): m/z [M-H] $^-$ calc. for $[\text{C}_{20}\text{H}_{14}\text{F}_2\text{NO}]^-$ 322.1049, found 322.1049.



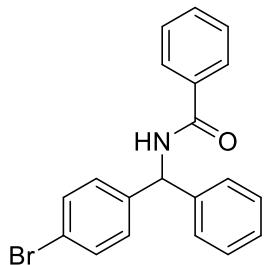
N-((4-chlorophenyl)(phenyl)methyl)benzamide (3la): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3la** as white solid. Yield: 76%, 122 mg. m.p.: 169-170 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.75 (d, J = 7.9 Hz, 2H), 7.46 (t, J = 7.2 Hz, 1H), 7.38-7.22 (m, 9H), 7.18 (d, J = 8.4 Hz, 2H), 6.89 (d, J = 6.4 Hz, 1H), 6.35 (d, J = 7.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.7, 141.1, 140.0, 134.0, 133.3, 131.8, 128.9, 128.8, 128.7, 128.6, 127.8, 127.6, 127.1, 56.9. NMR data is consistent with literature values.⁵ MS (EI), m/z 321.11 [M $^+$, 30%], 105.02 [100%].



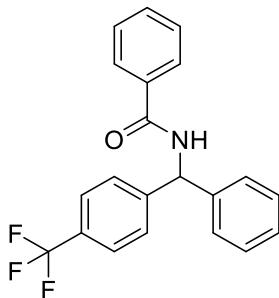
N-((4-chlorophenyl)(p-tolyl)methyl)benzamide (3ma): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ma** as white solid. Yield: 73%, 122 mg. m.p.: 178-180 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.80 (d, J = 7.3 Hz, 2H), 7.51 (t, J = 7.4 Hz, 1H), 7.42 (t, J = 7.8 Hz, 2H), 7.30-7.22 (m, 4H), 7.21 (s, 4H), 6.71 (d, J = 7.4 Hz, 1H), 6.36 (d, J = 7.7 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.5, 140.2, 138.1, 137.6, 134.1, 133.2, 131.8, 129.6, 128.8, 128.7, 128.6, 127.5, 127.1, 56.7, 21.1. MS (EI), m/z 335.17 [M $^+$, 30%], 105.14 [100%]. HRMS (ESI): m/z [M-H] $^-$ calc. for $[\text{C}_{21}\text{H}_{17}\text{ClNO}]^-$ 334.1004, found 334.1008.



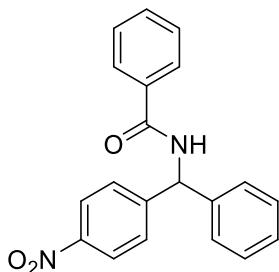
N-(bis(4-chlorophenyl)methyl)benzamide (3na): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3na** as white solid. Yield: 62%, 110 mg. m.p.: 199-200 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.78 (d, J = 7.2 Hz, 2H), 7.52 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.9 Hz, 2H), 7.31 (d, J = 8.5 Hz, 4H), 7.19 (d, J = 8.4 Hz, 4H), 6.73 (d, J = 7.5 Hz, 1H), 6.36 (d, J = 7.7 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.6, 139.5, 133.8, 133.7, 131.9, 129.2, 128.8, 128.7, 127.1, 56.4. NMR data is consistent with literature values.⁶ MS (EI), m/z 355.09 [M^+ , 20%], 105.14 [100%].



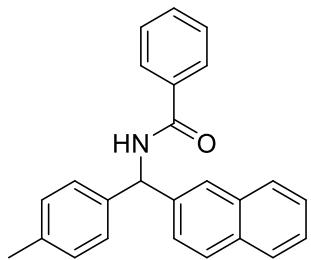
N-((4-bromophenyl)(phenyl)methyl)benzamide (3oa): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3oa** as white solid. Yield: 71%, 129 mg. m.p.: 183-184 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.79 (d, J = 8.2 Hz, 2H), 7.50 (t, J = 7.7 Hz, 1H), 7.45-7.39 (m, 4H), 7.35-7.25 (m, 5H), 7.16 (d, J = 8.4 Hz, 2H), 6.76 (d, J = 7.1 Hz, 1H), 6.37 (d, J = 7.7 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.6, 140.9, 140.5, 134.0, 131.8, 129.2, 128.9, 128.7, 127.9, 127.6, 127.1, 121.5, 57.0. HRMS (ESI): m/z [M-H]⁻ calc. for $[\text{C}_{20}\text{H}_{15}\text{BrNO}]^-$ 364.0343, found 364.0346.



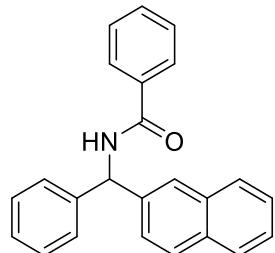
N-(phenyl(4-(trifluoromethyl)phenyl)methyl)benzamide (3pa): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3pa** as white solid. Yield: 66%, 117 mg. m.p.: 190-191 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.80 (d, J = 7.4 Hz, 2H), 7.57 (d, J = 8.2 Hz, 2H), 7.51 (t, J = 7.4 Hz, 1H), 7.43-7.29 (m, 7H), 7.25 (d, J = 6.9 Hz, 2H), 6.84 (d, J = 7.4 Hz, 1H), 6.45 (d, J = 7.6 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.7, 145.4, 140.6, 133.9, 131.9, 129.7 (q, J = 32.2 Hz), 129.0, 128.7, 128.1, 127.7, 127.6, 127.1, 125.7 (q, J = 7.4 Hz), 124.1, (q, J = 270.3 Hz), 57.3. NMR data is consistent with literature values.⁷ HRMS (ESI): m/z [M-H]⁻ calc. for $[\text{C}_{21}\text{H}_{15}\text{F}_3\text{NO}]^-$ 354.1111, found 354.1109.



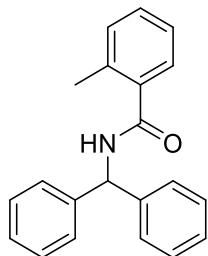
N-((4-nitrophenyl)(phenyl)methyl)benzamide (3qa): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3qa** as white solid. Yield: 62%, 103 mg. m.p.: 172-173 °C. ^1H NMR (500 MHz, CDCl_3) δ 8.14 (d, J = 8.8 Hz, 2H), 7.81 (d, J = 7.2 Hz, 2H), 7.52 (t, J = 7.4 Hz, 1H), 7.47-7.41 (m, 4H), 7.39-7.34 (m, 3H), 7.27-7.25 (m, 2H), 7.01 (d, J = 7.3 Hz, 1H), 6.45 (d, J = 7.3 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.8, 148.8, 147.2, 140.0, 133.6, 132.0, 129.2, 128.7, 128.4, 128.1, 127.8, 127.1, 123.8, 57.4. NMR data is consistent with literature values.⁸ MS (EI), m/z 332.14 [M⁺, 20%], 105.14 [100%].



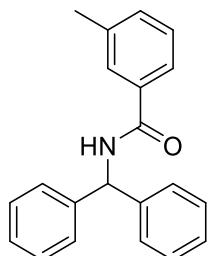
N-(naphthalen-2-yl(p-tolyl)methyl)benzamide (3sa): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3sa** as white solid. Yield: 67%, 118 mg. m.p.: 187-188 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.87-7.78 (m, 6H), 7.53-7.43 (m, 6H), 7.26 (t, J = 10.0 Hz, 3H), 7.18 (d, J = 8.0 Hz, 2H), 6.84 (d, J = 7.7 Hz, 1H), 6.60 (d, J = 7.9 Hz, 1H), 2.37 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 166.5, 139.0, 138.4, 137.4, 134.3, 133.3, 132.8, 131.7, 129.5, 128.6, 128.5, 128.0, 127.7, 127.6, 127.1, 126.3, 126.0, 125.9, 125.7, 57.4, 21.1. HRMS (ESI): m/z [M-H]⁻ calc. for [C₂₅H₂₀NO]⁻ 350.1550, found 350.1546.



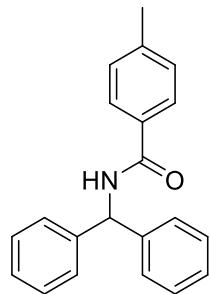
N-(naphthalen-2-yl(phenyl)methyl)benzamide (3ta): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ta** as white solid. Yield: 64%, 108 mg. m.p.: 183-184 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.83-7.74 (m, 6H), 7.51-7.38 (m, 6H), 7.34-7.29 (m, 5H), 6.85 (d, J = 7.5 Hz, 1H), 6.61 (d, J = 7.9 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 166.6, 141.4, 138.8, 134.3, 133.3, 132.8, 131.7, 128.8, 128.6, 128.0, 127.7, 127.1, 126.3, 126.1, 126.0, 125.7, 57.6. HRMS (ESI): m/z [M-H]⁻ calc. for [C₂₄H₁₈NO]⁻ 336.1393, found 336.1397.



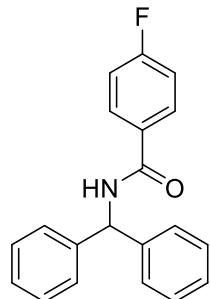
N-benzhydryl-2-methylbenzamide (3ab): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3ab** as white solid. Yield: 79%, 108 mg. m.p.: 176-178 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.83 (d, J = 7.5 Hz, 2H), 7.53 (t, J = 7.3 Hz, 1H), 7.44 (t, J = 7.6 Hz, 2H), 7.38-7.30 (m, 5H), 7.22-7.17 (m, 4H), 6.78 (d, J = 7.1 Hz, 2H), 6.44 (d, J = 7.7 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 169.1, 141.5, 136.4, 136.1, 131.1, 130.1, 128.7, 127.6, 127.4, 126.7, 125.8, 57.3, 19.8. NMR data is consistent with literature values.⁹ MS (EI), m/z 301.14 [M $^+$, 30%], 105.16 [100%].



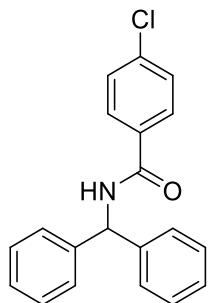
N-benzhydryl-3-methylbenzamide (3ac): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3ac** as white solid. Yield: 80%, 120 mg. m.p.: 157-158 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.60 (d, J = 18.2 Hz, 2H), 7.34-7.25 (m, 12H), 6.74 (d, J = 7.4 Hz, 1H), 6.44 (d, J = 7.9 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.7, 141.5, 138.5, 134.3, 132.4, 128.7, 128.5, 127.8, 127.5, 127.4, 124.0, 57.4, 21.3. NMR data is consistent with literature values.⁹ MS (EI), m/z 301.17 [M $^+$, 40%], 105.12 [100%].



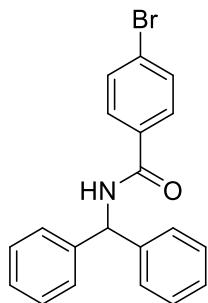
N-benzhydryl-4-methylbenzamide (3ad): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 15) afforded the title product **3ad** as white solid. Yield: 84%, 126 mg. m.p.: 178-179 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.70 (d, J = 8.1 Hz, 2H), 7.34-7.24 (m, 10H), 7.20 (d, J = 8.0 Hz, 2H), 6.75 (d, J = 7.6 Hz, 2H), 6.43 (d, J = 7.9 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.5, 142.1, 141.6, 131.4, 129.3, 128.7, 127.6, 127.1, 57.4, 21.4. NMR data is consistent with literature values.⁹ MS (EI), m/z 301.16 [M^+ , 40%], 105.16 [100%].



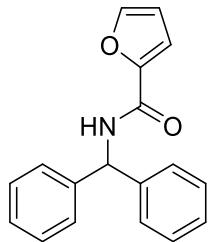
N-benzhydryl-4-fluorobenzamide (3ae): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ae** as white solid. Yield: 72%, 110 mg. m.p.: 215-217 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.83-7.80 (m, 2H), 7.34 (d, J = 7.7 Hz, 4H), 7.30-7.28 (m, 6H), 7.09 (t, J = 8.6 Hz, 2H), 6.67 (d, J = 7.3 Hz, 1H), 6.42 (d, J = 7.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 165.9, 164.6 (d, J = 199.0 Hz), 141.4, 130.4 (d, J = 3.2 Hz), 129.4 (d, J = 8.9 Hz), 128.8, 127.6, 127.5, 115.7 (d, J = 21.8 Hz), 57.6. NMR data is consistent with literature values.⁹ MS (EI), m/z 305.15 [M^+ , 30%], 123.12 [100%].



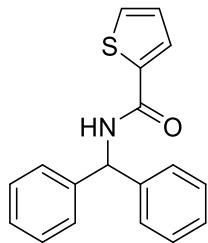
N-benzhydryl-4-chlorobenzamide (**3af**): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3af** as white solid. Yield: 69%, 111 mg. m.p.: 169-170 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.74 (d, J = 8.6 Hz, 2H), 7.40-7.33 (m, 6H), 7.29 (t, J = 5.1 Hz, 6H), 6.69 (d, J = 7.4 Hz, 1H), 6.42 (d, J = 7.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 165.5, 141.3, 138.0, 132.6, 128.9, 128.8, 128.5, 127.7, 127.5, 57.6. NMR data is consistent with literature values.⁶ MS (EI), m/z 139.10 [M $^+$, 40%], 321.14 [100%].



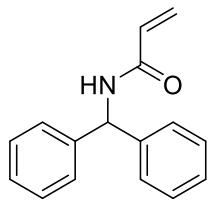
N-benzhydryl-4-bromobenzamide (**3ag**): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ag** as white solid. Yield: 65%, 119 mg. m.p.: 195-196 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.85-7.82 (m, 2H), 7.37 (d, J = 7.7 Hz, 4H), 7.32-7.30 (m, 6H), 7.28 (s, 1H), 7.12 (t, J = 8.6 Hz, 2H), 6.69 (d, J = 7.3 Hz, 1H), 6.45 (d, J = 7.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 165.6, 141.3, 137.9, 132.5, 128.8, 128.7, 128.5, 127.6, 127.4, 57.5. NMR data is consistent with literature values.⁶ MS (EI), m/z 365.08 [M $^+$, 50%], 104.13 [100%].



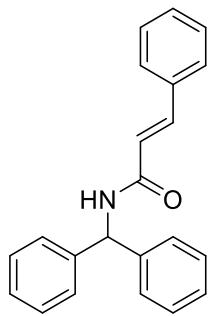
N-benzhydrylfuran-2-carboxamide (3ah): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ah** as white solid. Yield: 51%, 71 mg. m.p.: 163-164 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.41 (d, J = 1.0 Hz, 1H), 7.34-7.32 (m, 4H), 7.29-7.25 (m, 6H), 7.13 (d, J = 4.0 Hz, 1H), 6.98 (d, J = 7.9 Hz, 1H), 6.48-6.47 (m, 1H), 6.42 (d, J = 8.3 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 157.5, 147.8, 144.0, 141.3, 128.7, 127.6, 127.5, 114.8, 112.3, 56.6. NMR data is consistent with literature values.¹⁰ MS (EI), m/z 277.17 [M $^+$, 60%], 95.01 [100%].



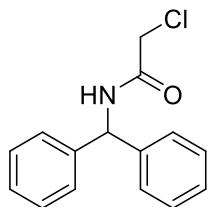
N-benzhydrylthiophene-2-carboxamide (3ai): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ai** as white solid. Yield: 58%, 85 mg. m.p.: 170-171 °C. ^1H NMR (600 MHz, CDCl_3) δ 7.50 (d, J = 3.2 Hz, 1H), 7.43 (d, J = 4.8 Hz, 1H), 7.30 (t, J = 7.7 Hz, 4H), 7.25 (t, J = 7.4 Hz, 6H), 7.01 (t, J = 4.0 Hz, 1H), 6.56 (d, J = 6.3 Hz, 1H), 6.37 (d, J = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, CDCl_3) δ 161.0, 141.3, 138.6, 130.3, 128.8, 128.4, 127.7, 127.6, 127.5, 57.4. NMR data is consistent with literature values.⁹ MS (EI), m/z 293.13 [M $^+$, 40%], 111.03 [100%].



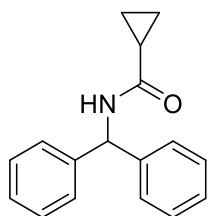
N-benzhydrylacrylamide (3aj): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3aj** as white solid. Yield: 86%, 102 mg. m.p.: 180-181 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.36-7.33 (m, 4H), 7.30-7.25 (m, 6H), 6.35-6.31 (m, 3H), 6.21-6.15 (m, 1H), 5.69-5.66 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 164.6, 141.3, 130.6, 128.7, 127.5, 127.4, 127.1, 57.1. NMR data is consistent with literature values.¹¹ MS (EI), m/z 237.12 [M $^+$, 50%], 104.17 [100%].



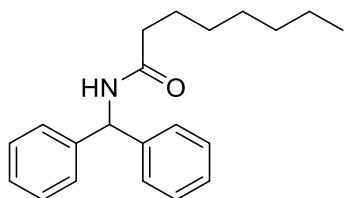
N-benzhydrylcinnamamide (**3ak**): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3ak** as white solid. Yield: 82%, 128 mg. m.p.: 221-222 °C. ¹H NMR (600 MHz, CDCl₃) δ 7.67 (d, *J* = 15.5 Hz, 1H), 7.49 (s, 2H), 7.33 (t, *J* = 7.8 Hz, 7H), 7.26 (t, *J* = 7.4 Hz, 7H), 6.48 (d, *J* = 15.6 Hz, 1H), 6.39 (d, *J* = 7.6 Hz, 1H), 6.27 (d, *J* = 6.3 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 165.0, 141.9, 141.4, 134.7, 129.8, 128.8, 128.7, 127.8, 127.6, 127.5, 120.2, 57.2. NMR data is consistent with literature values.¹¹ MS (EI), m/z 313.17 [M⁺, 40%], 182.16 [100%].



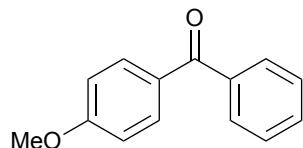
N-benzhydryl-2-chloroacetamide (**3al**): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3al** as white solid. Yield: 78%, 101 mg. m.p.: 131-133 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.35 (t, *J* = 7.1 Hz, 4H), 7.29 (t, *J* = 7.2 Hz, 2H), 7.24 (d, *J* = 7.4 Hz, 5H), 6.25 (d, *J* = 8.3 Hz, 1H), 4.10 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 165.1, 140.7, 128.8, 127.8, 127.3, 57.2, 42.7. NMR data is consistent with literature values.⁷ MS (EI), m/z 259.12 [M⁺, 20%], 224.17 [100%].



N-benzhydrylcyclopropanecarboxamide (3am): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product **3am** as white solid. Yield: 76%, 95 mg. m.p.: 169-170 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.34 (t, J = 7.1 Hz, 5H), 7.28 (t, J = 7.2 Hz, 3H), 7.25 (s, 2H), 6.41 (d, J = 7.3 Hz, 1H), 6.28 (d, J = 8.0 Hz, 1H), 1.46-1.41 (m, 1H), 1.02-0.99 (m, 2H), 0.77-0.73 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 172.7, 141.8, 128.6, 127.5, 127.4, 57.1, 14.8, 7.3. NMR data is consistent with literature values.¹¹ MS (EI), m/z 251.16 [M⁺, 80%], 182.13 [100%].

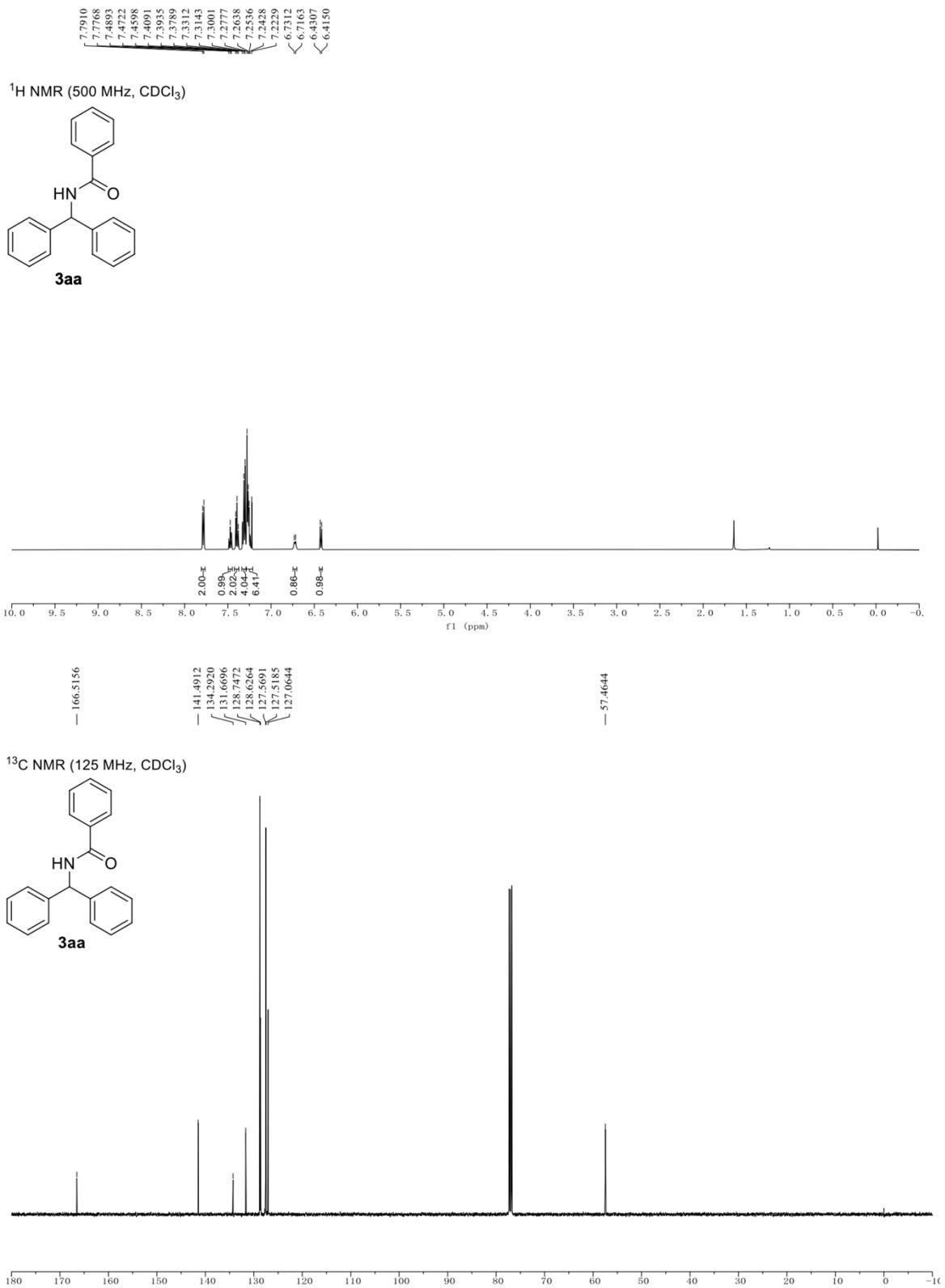


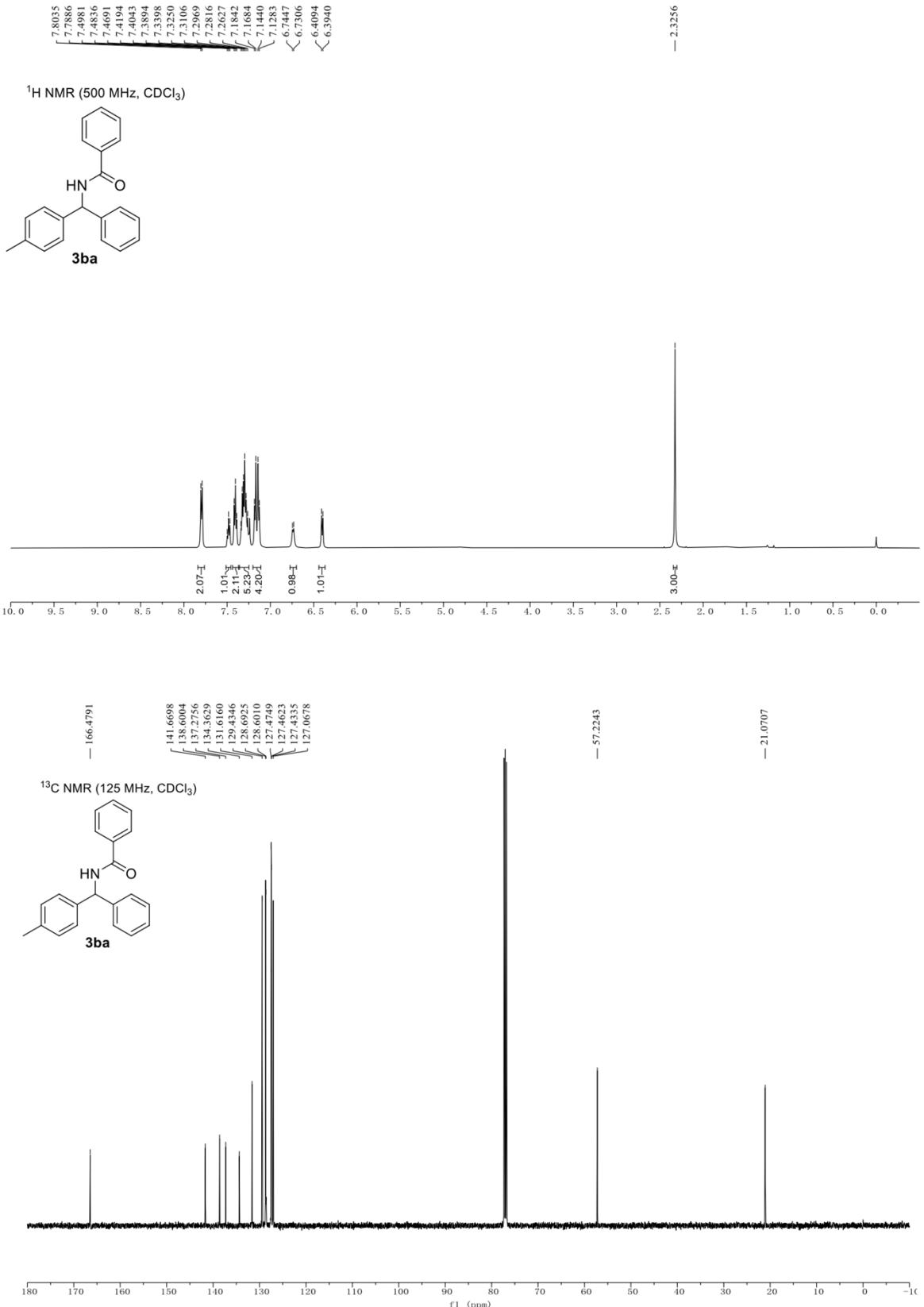
N-benzhydryloctanamide (3an): Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 5) afforded the title product **3an** as white solid. Yield: 71%, 94 mg. m.p.: 106-107 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.32-7.20 (m, 10H), 6.25 (d, J = 7.6 Hz, 2H), 7.22 (t, J = 7.5 Hz, 2H), 1.64 (t, J = 6.5 Hz, 2H), 1.28-1.25 (m, 8H), 0.87 (t, J = 6.7 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 172.3, 141.7, 128.6, 127.5, 127.4, 56.8, 36.8, 31.7, 29.3, 29.0, 25.8, 22.6, 14.1. NMR data is consistent with literature values.¹² MS (EI), m/z 309.17 [M⁺, 30%], 167.17 [100%].



(4-methoxyphenyl)(phenyl)methanone: Column chromatography on silica gel (ethyl acetate : petroleum ether = 1 : 10) afforded the title product as white solid. Yield: 89%, 95 mg. m.p.: 58-63 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, J = 8.8 Hz, 2H), 7.75 (d, J = 6.9 Hz, 2H), 7.56 (t, J = 7.4 Hz, 2H), 7.47 (t, J = 7.5 Hz, 2H), 6.96 (d, J = 8.8 Hz, 2H), 3.88 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 195.7, 163.3, 138.4, 132.7, 132.0, 130.3, 129.8, 128.3, 113.7, 55.6. NMR data is consistent with literature values.¹³ MS (EI), m/z 212.03 [M⁺, 30%], 135.09 [100%].

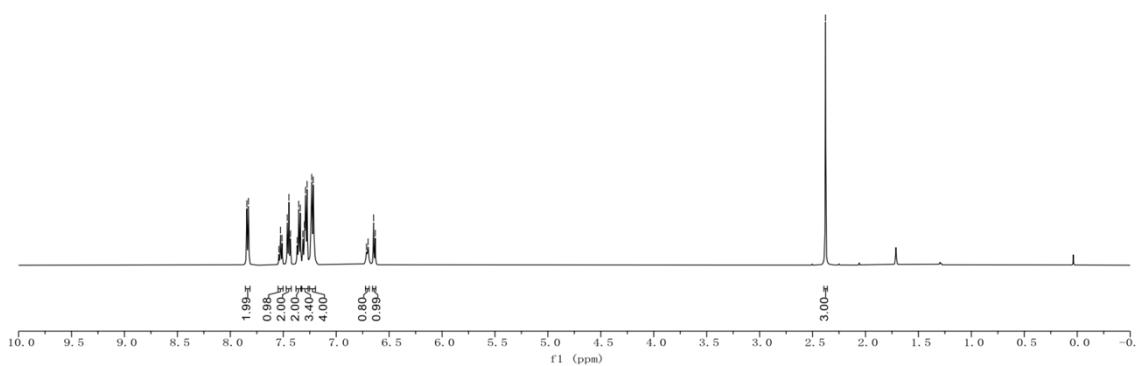
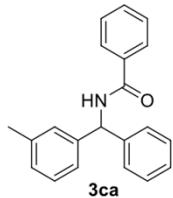
4 NMR data





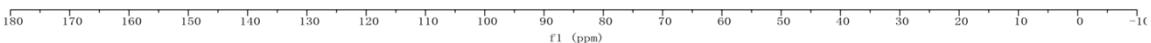
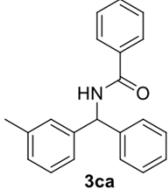
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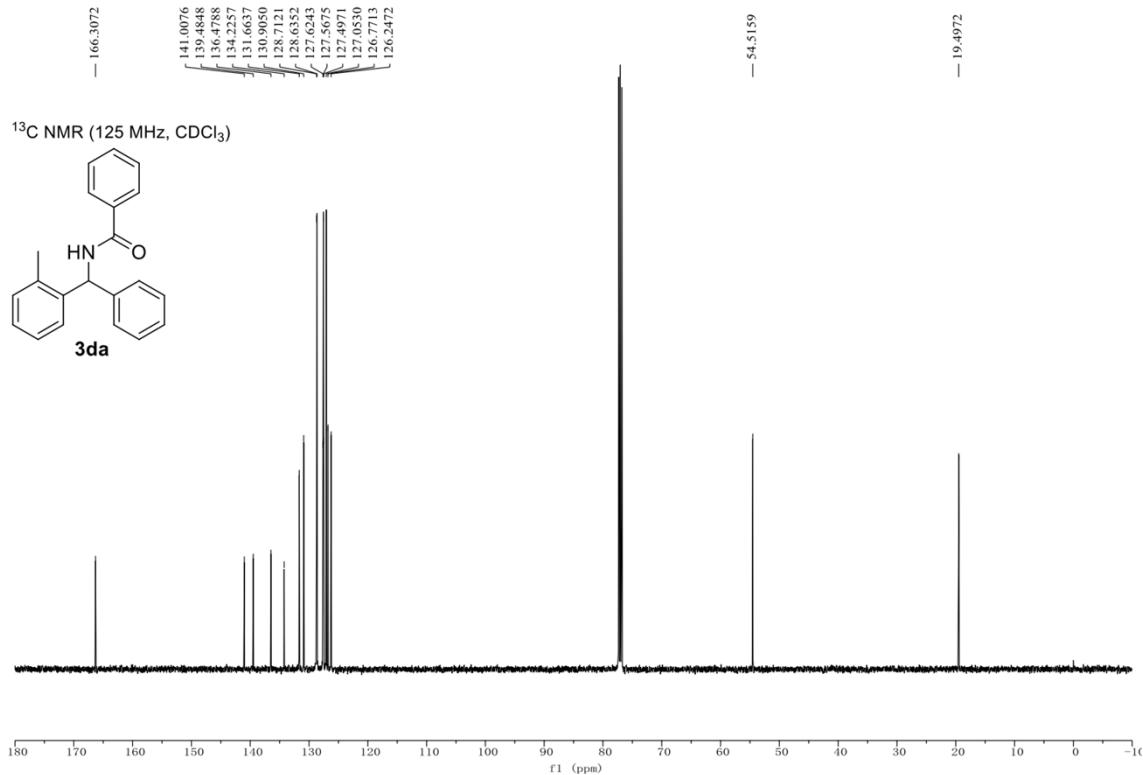
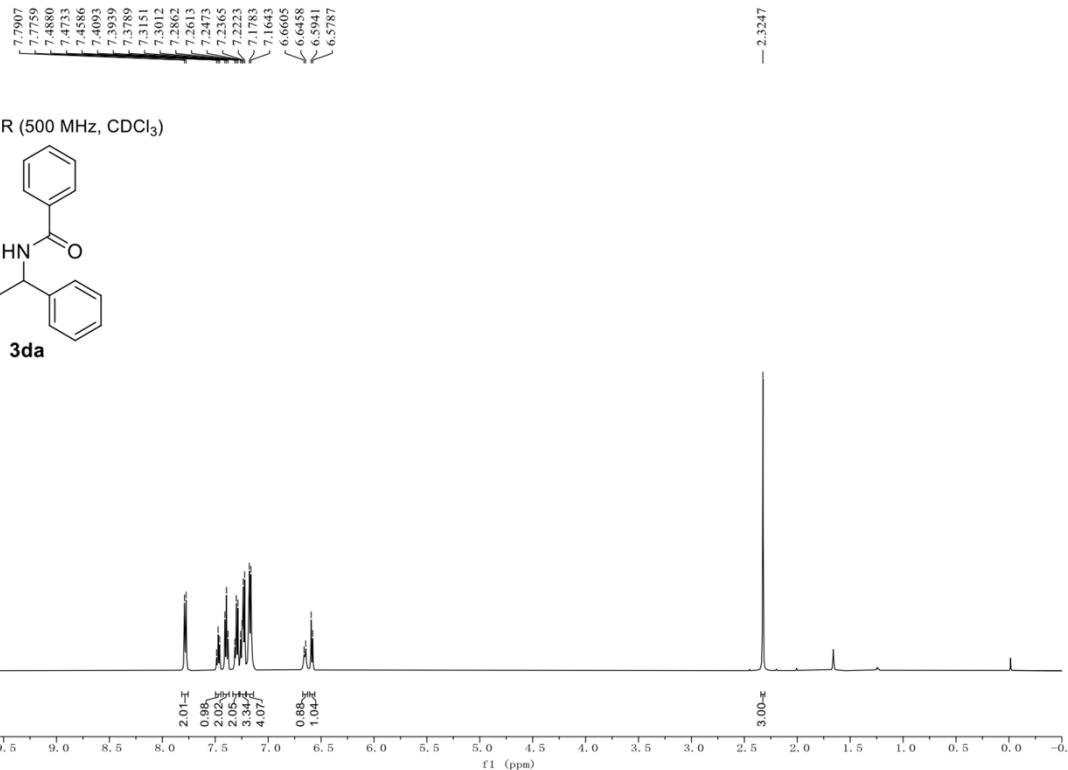
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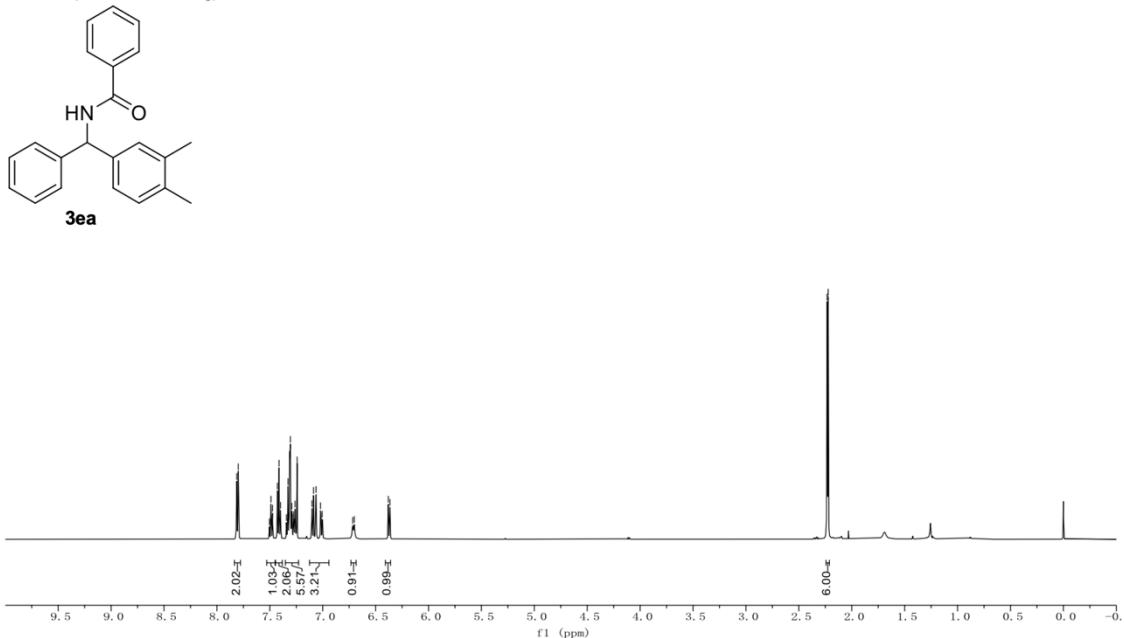
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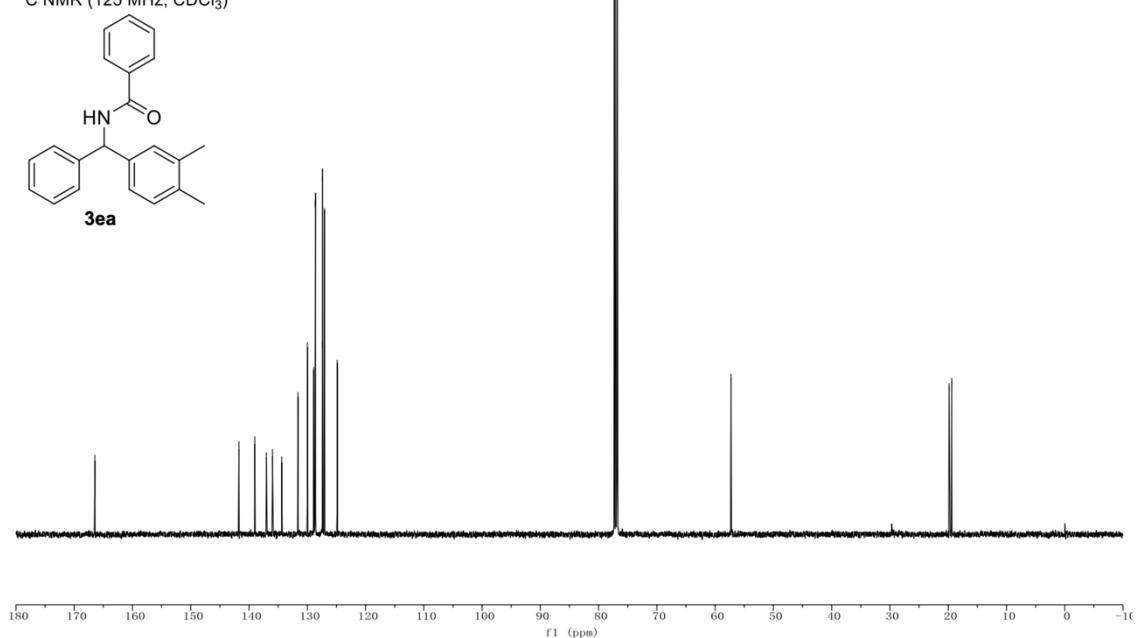


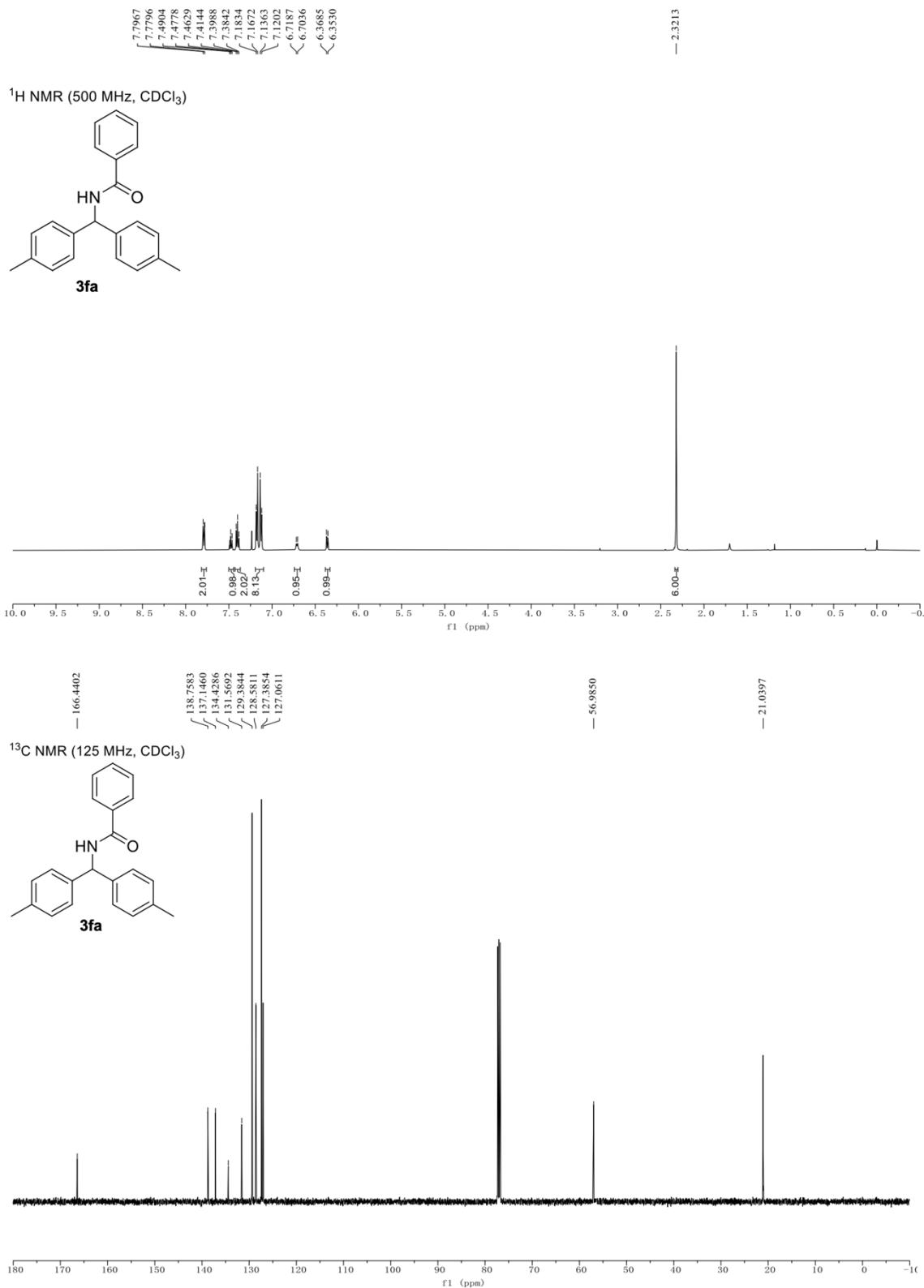
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7.2617
7.2419
7.1621
7.0866
7.0632
7.0211
6.9657
< 6.3647

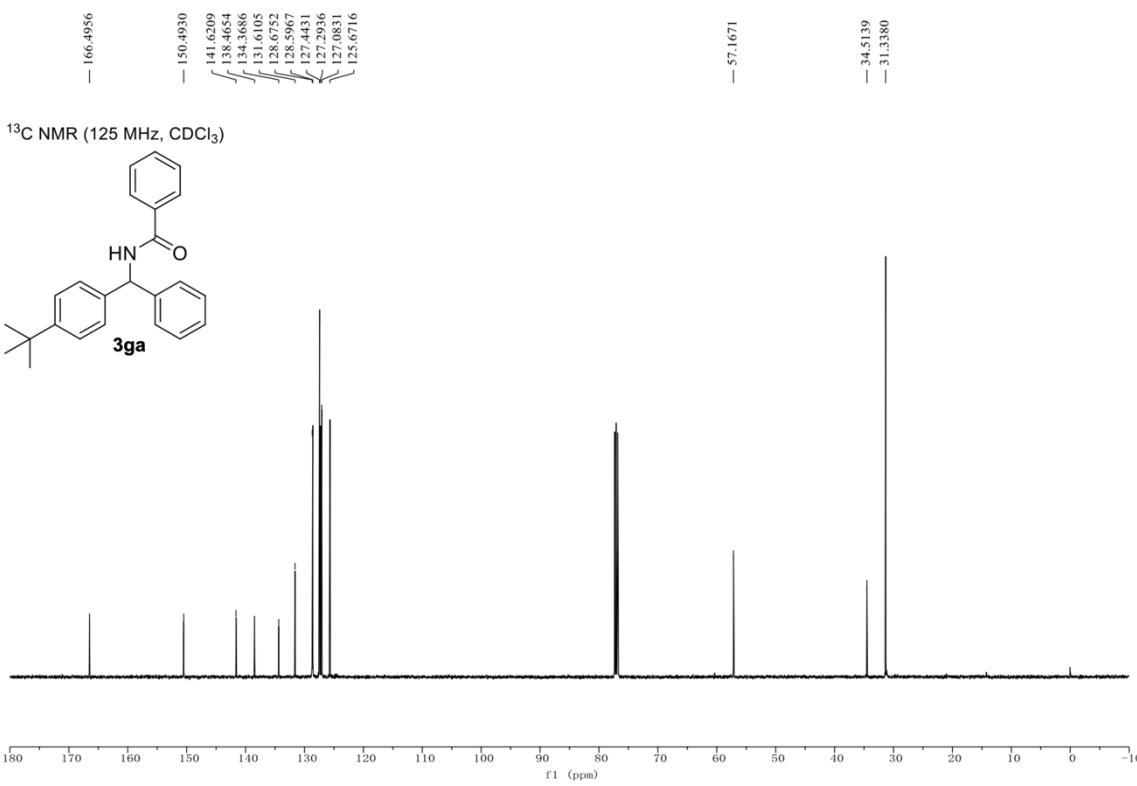
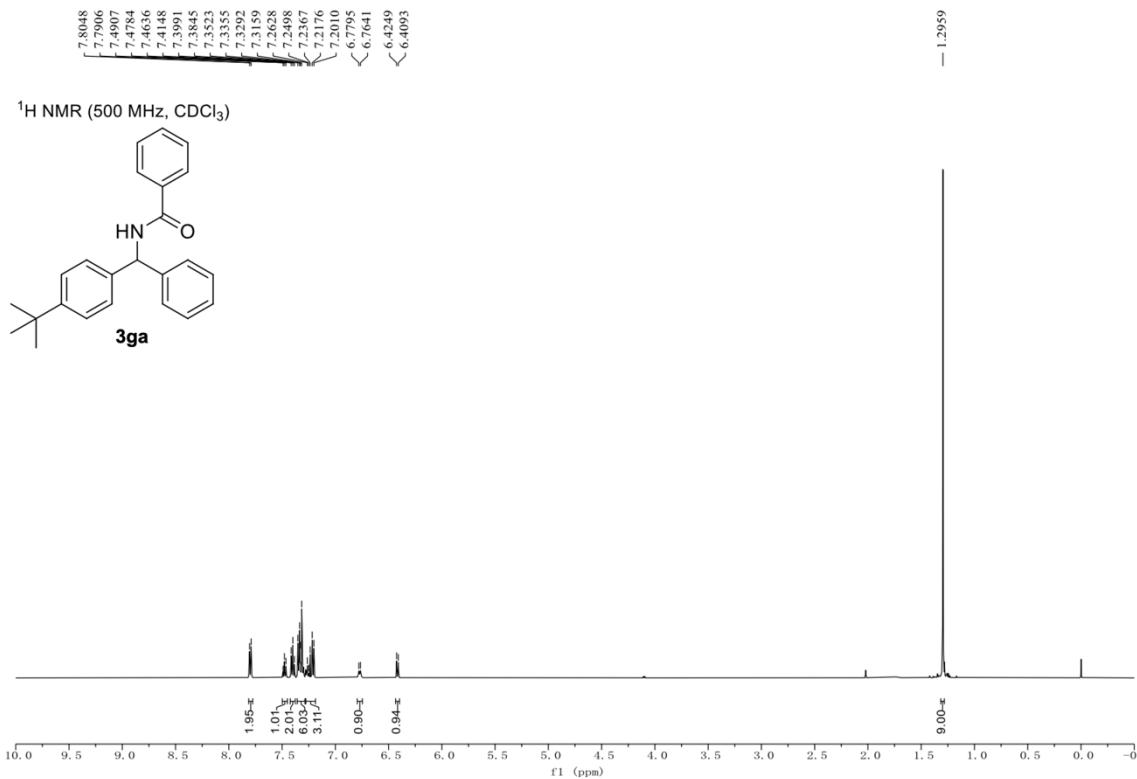
¹H NMR (500 MHz, CDCl₃)



¹³C NMR (125 MHz, CDCl₃)

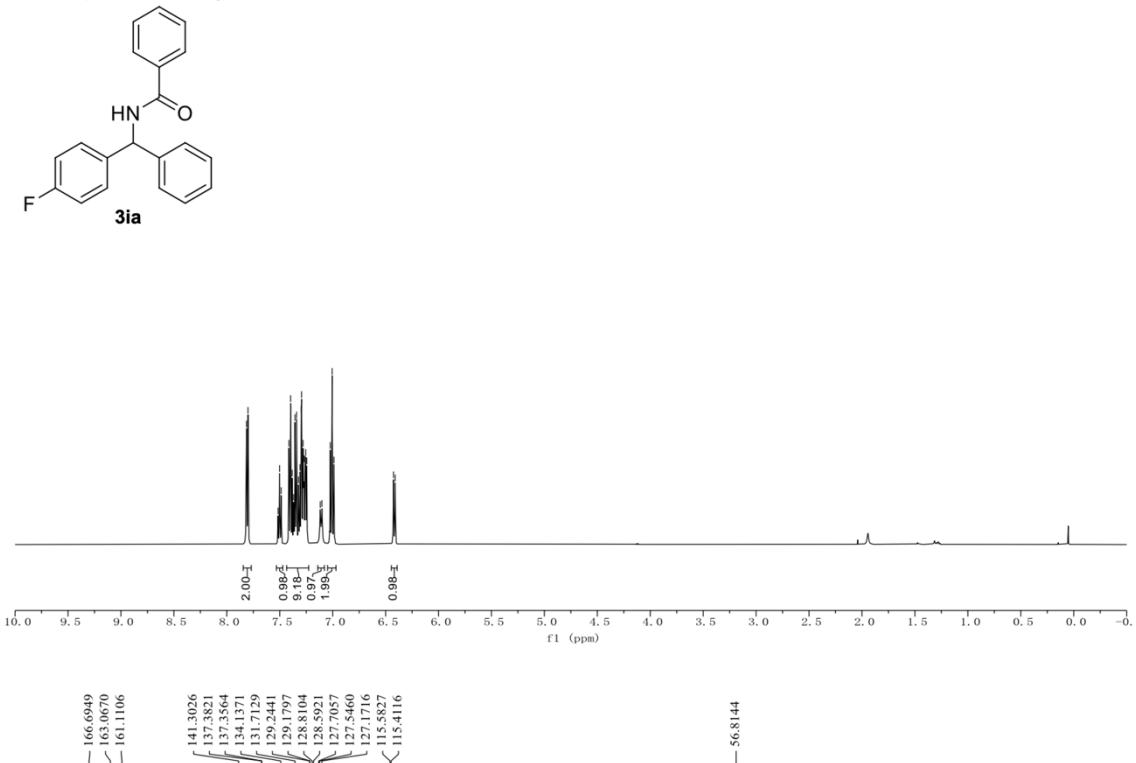




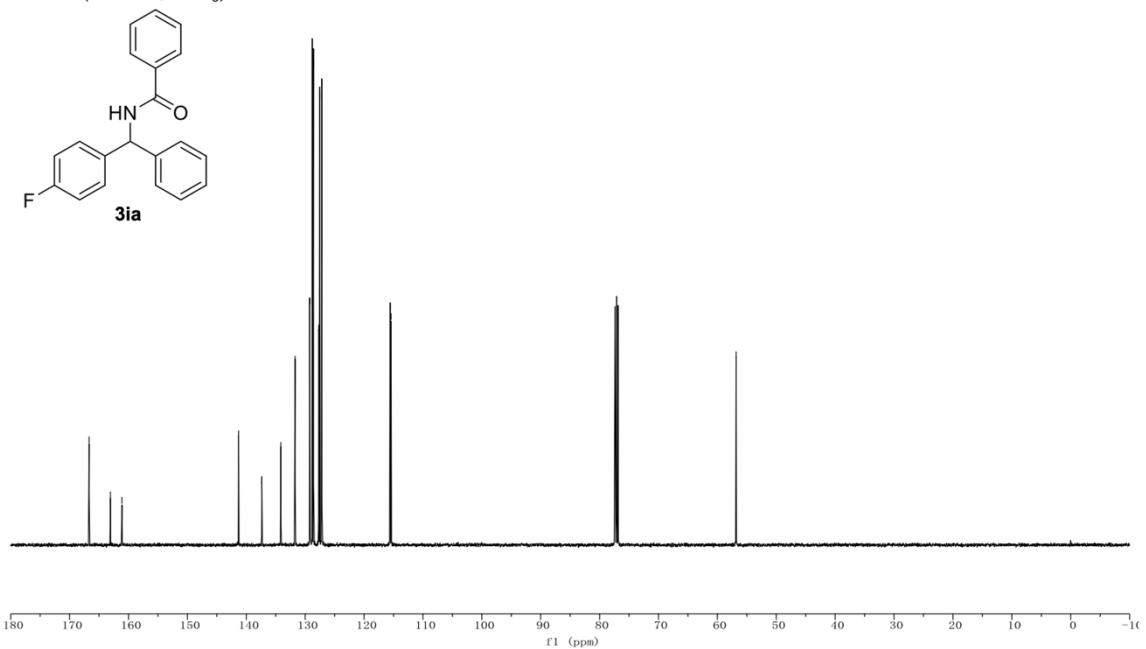


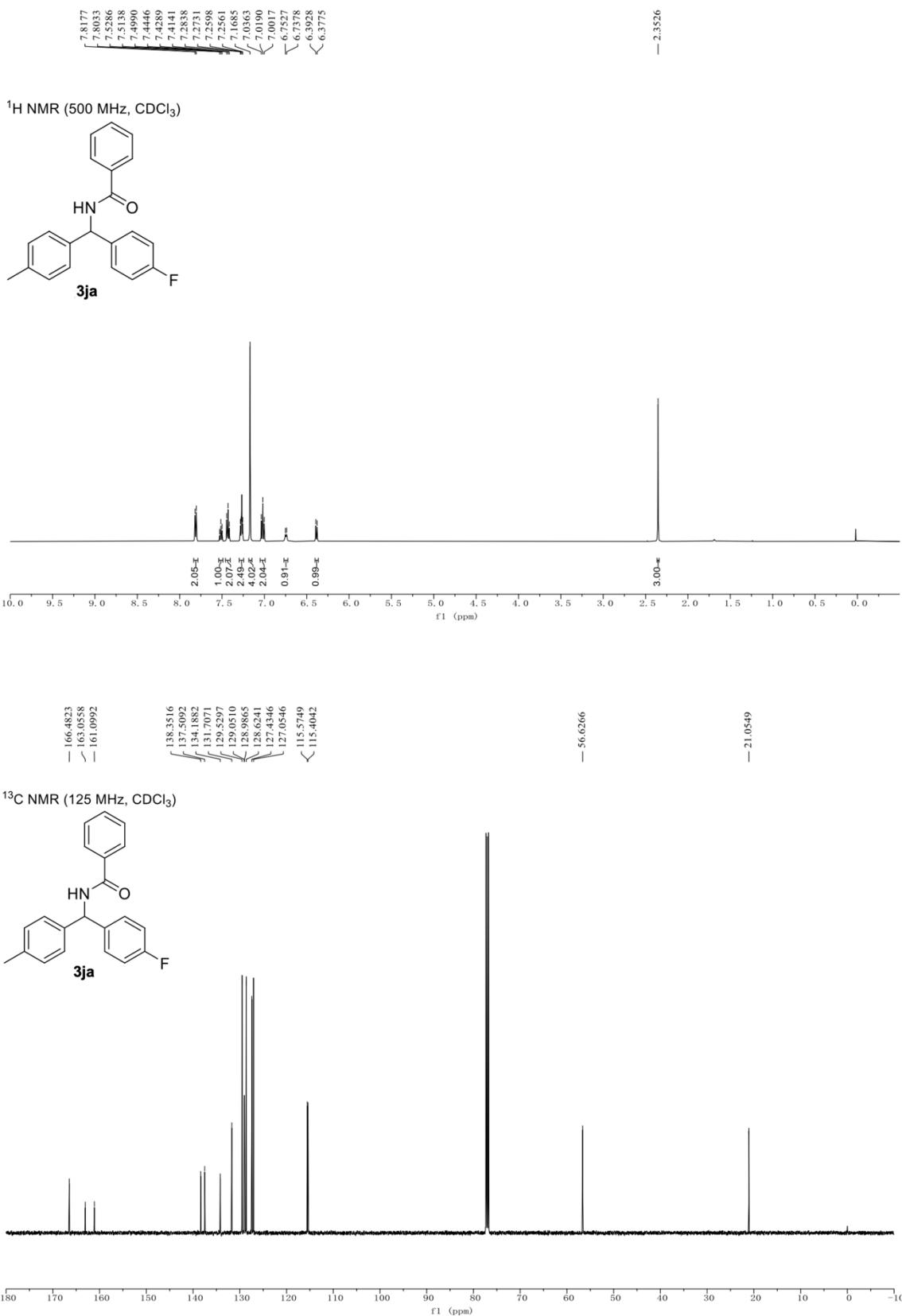


¹H NMR (500 MHz, CDCl₃)



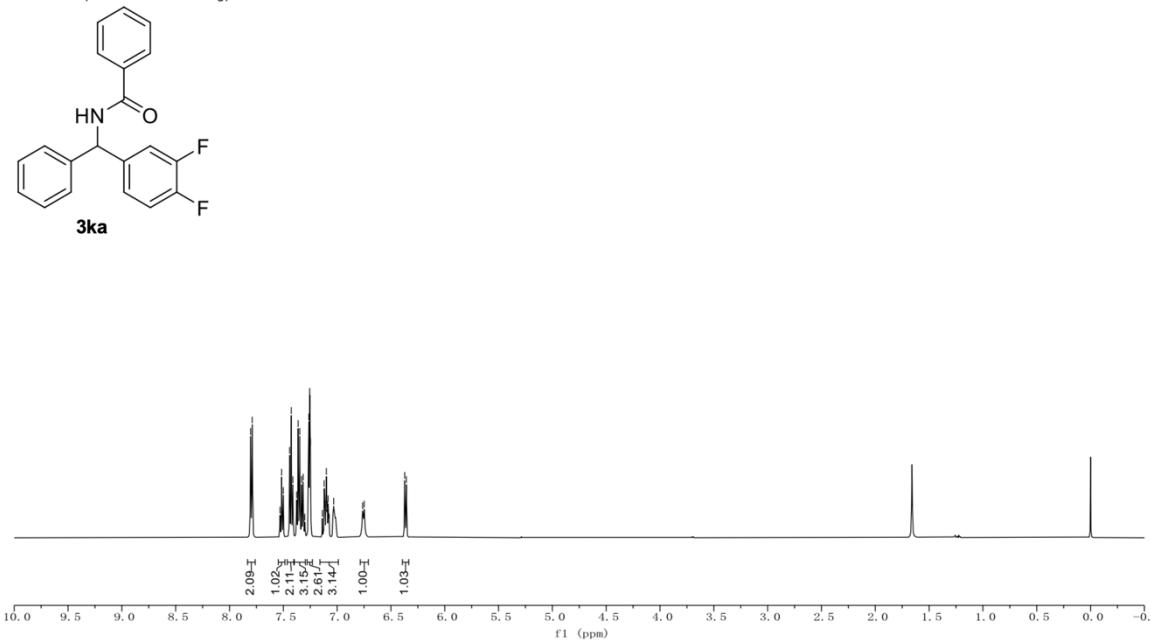
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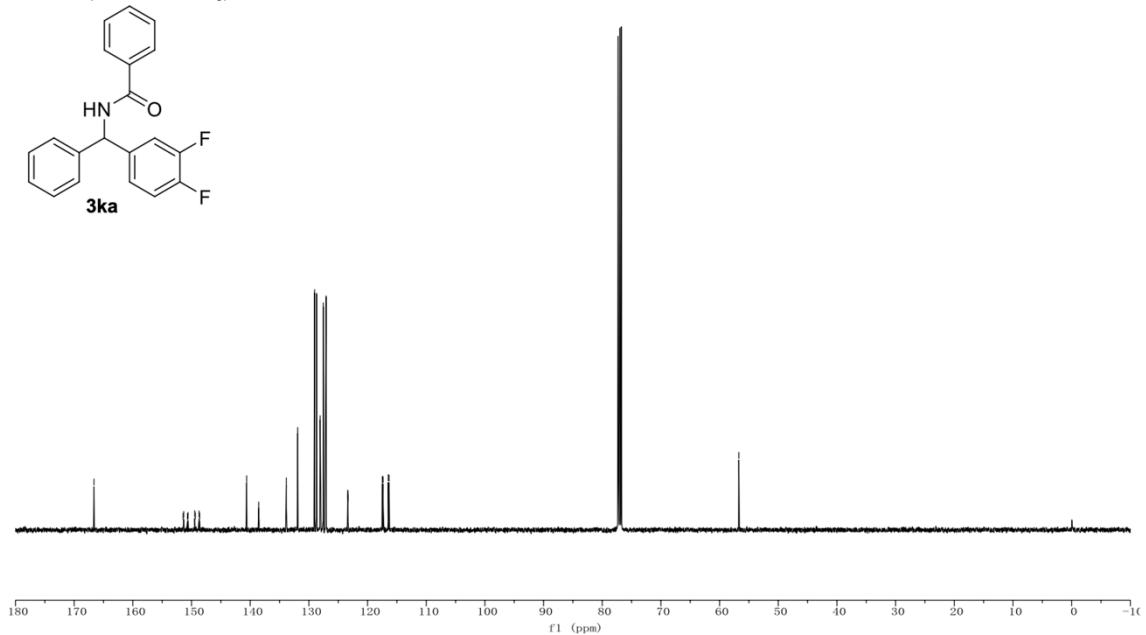




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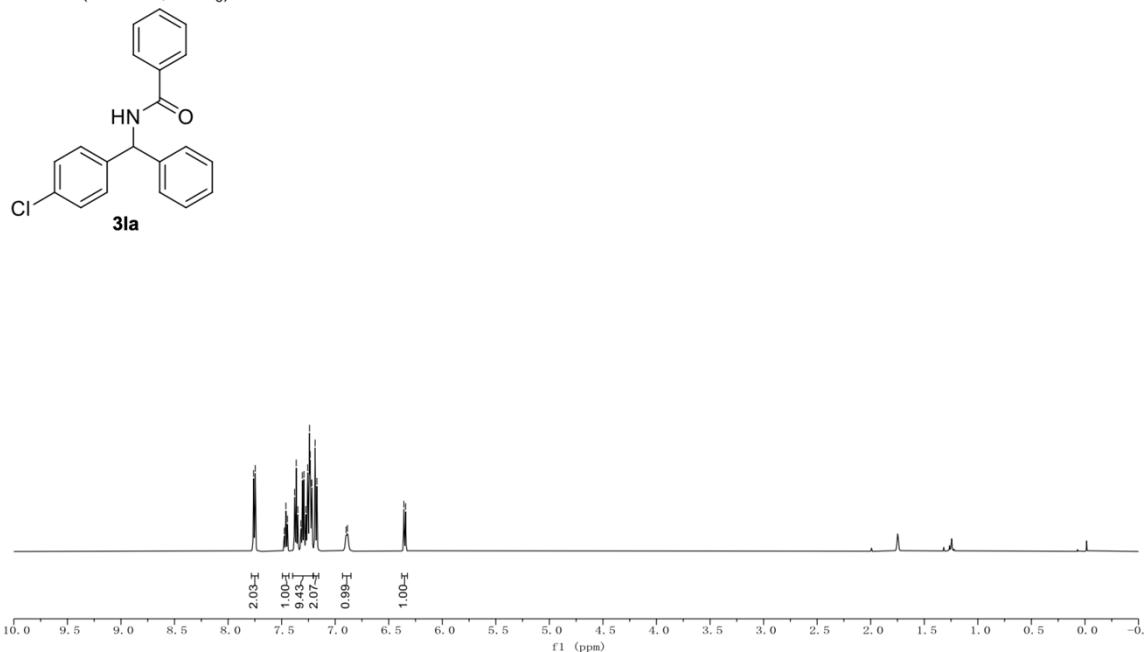


¹³C NMR (125 MHz, CDCl₃)



7.7620
 7.7462
 7.4603
 7.4747
 7.4451
 7.3791
 7.3639
 7.3485
 7.3200
 7.3065
 7.2915
 7.2739
 7.2570
 7.2398
 7.2338
 7.2211
 7.2180
 7.1874
 7.1706
 6.8978
 6.8850
 6.5584
 6.3429

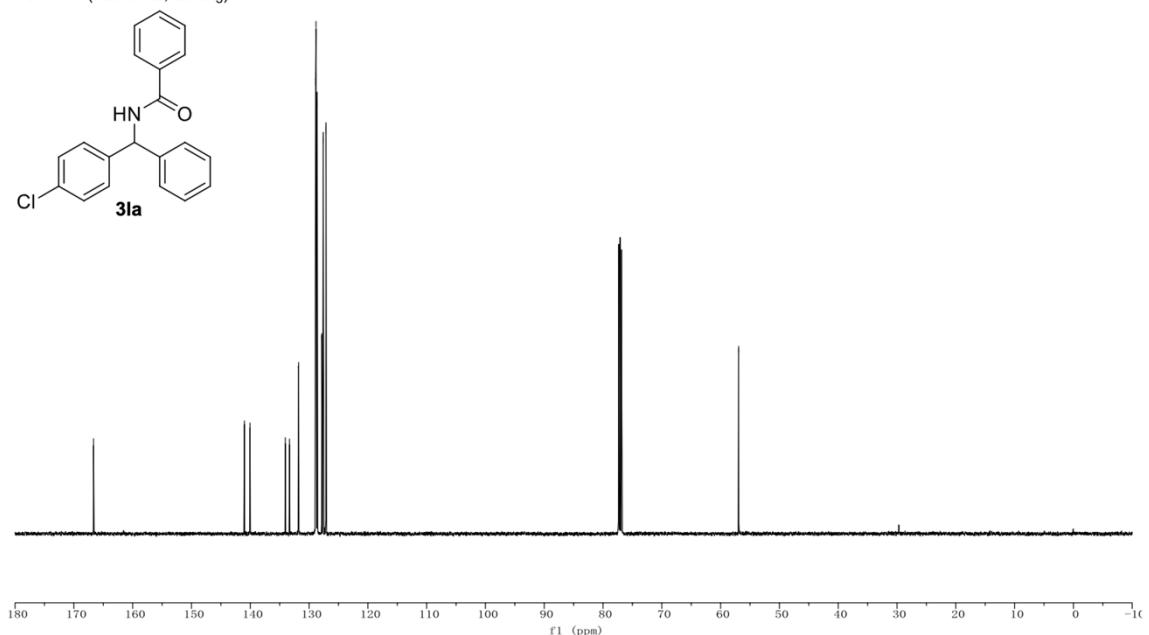
¹H NMR (500 MHz, CDCl₃)



141.0011
 140.0425
 134.0435
 133.3288
 131.7793
 128.8843
 128.8494
 128.8206
 128.6314
 127.8362
 127.5828
 127.1217

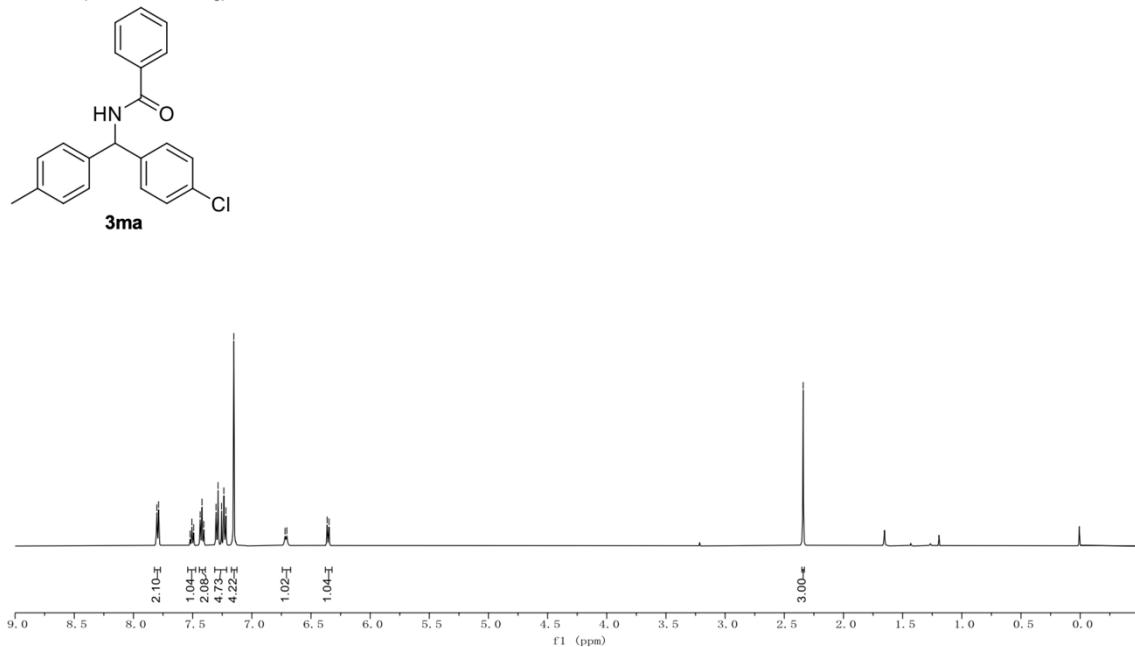
— 166.6575 — 56.9318

¹³C NMR (125 MHz, CDCl₃)

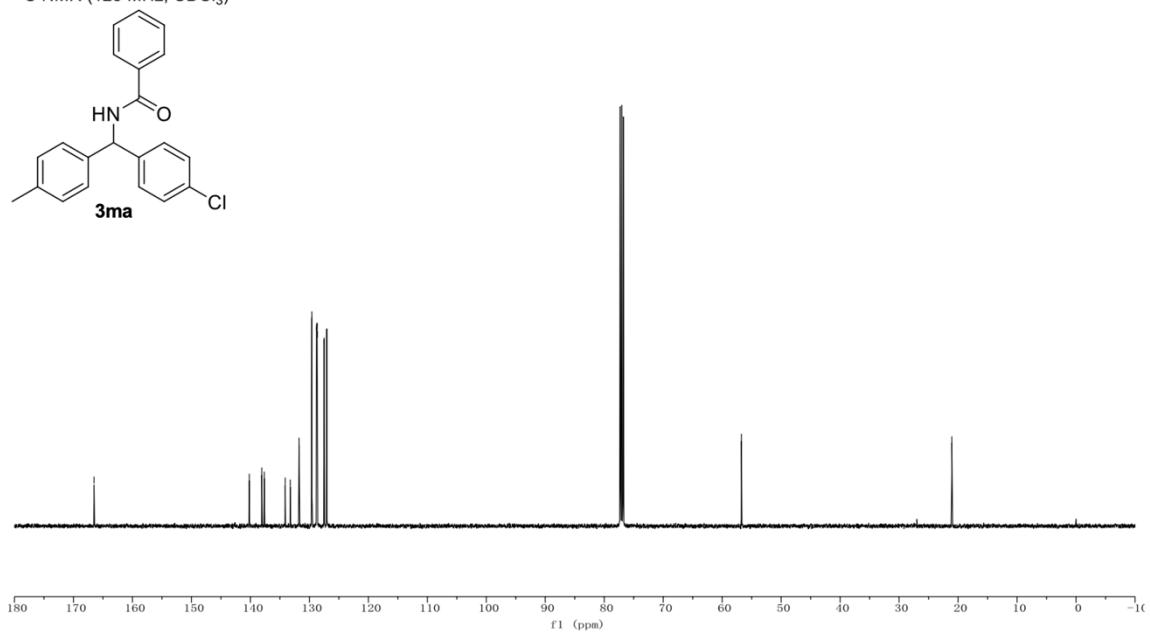


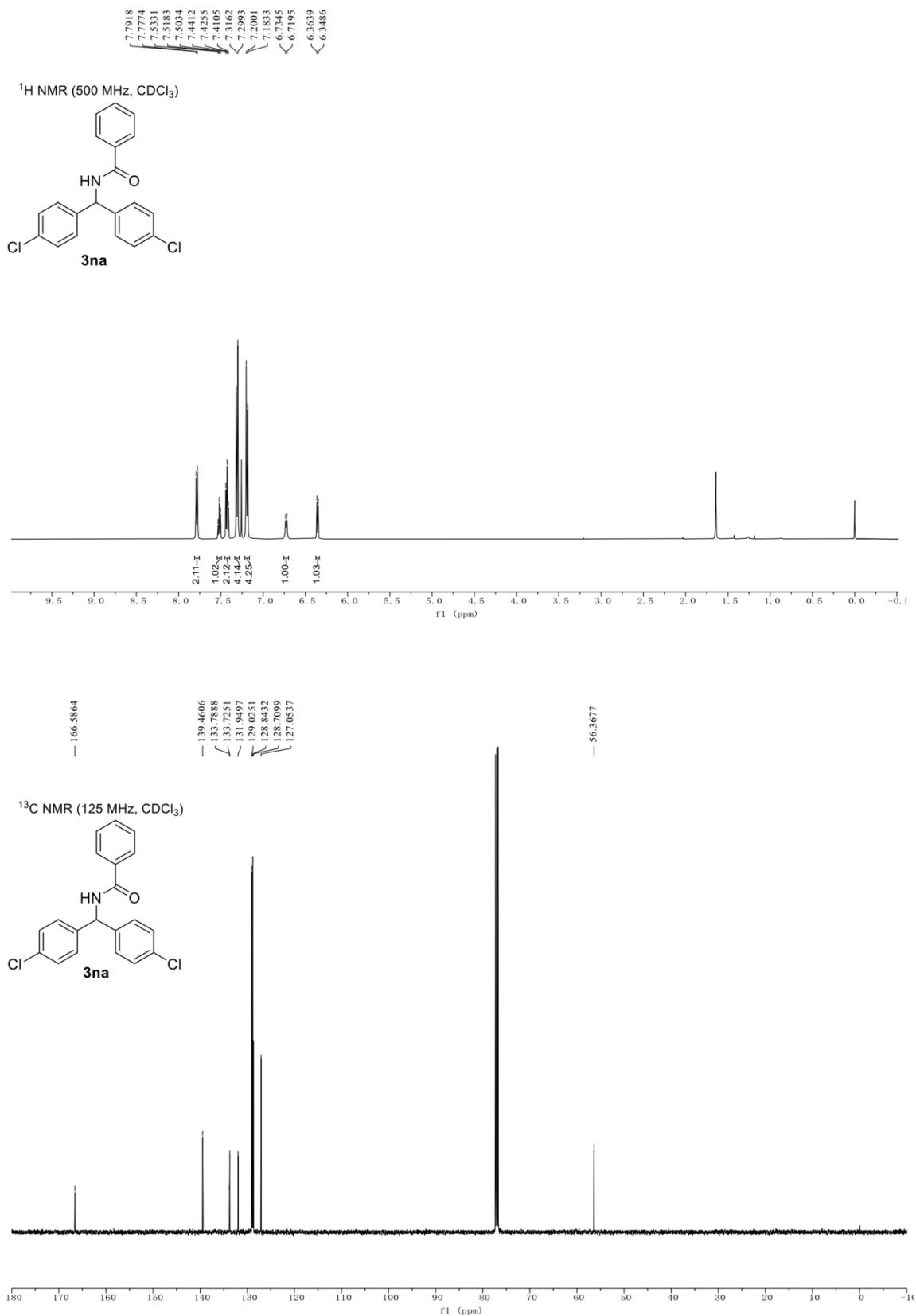
7.8024
 7.7879
 7.5210
 7.5063
 7.4915
 7.4364
 7.4208
 7.4059
 7.3022
 7.2852
 7.2559
 7.2360
 7.2192
 7.1517
 6.7188
 6.7040
 6.3629
 6.3476

¹H NMR (500 MHz, CDCl₃)



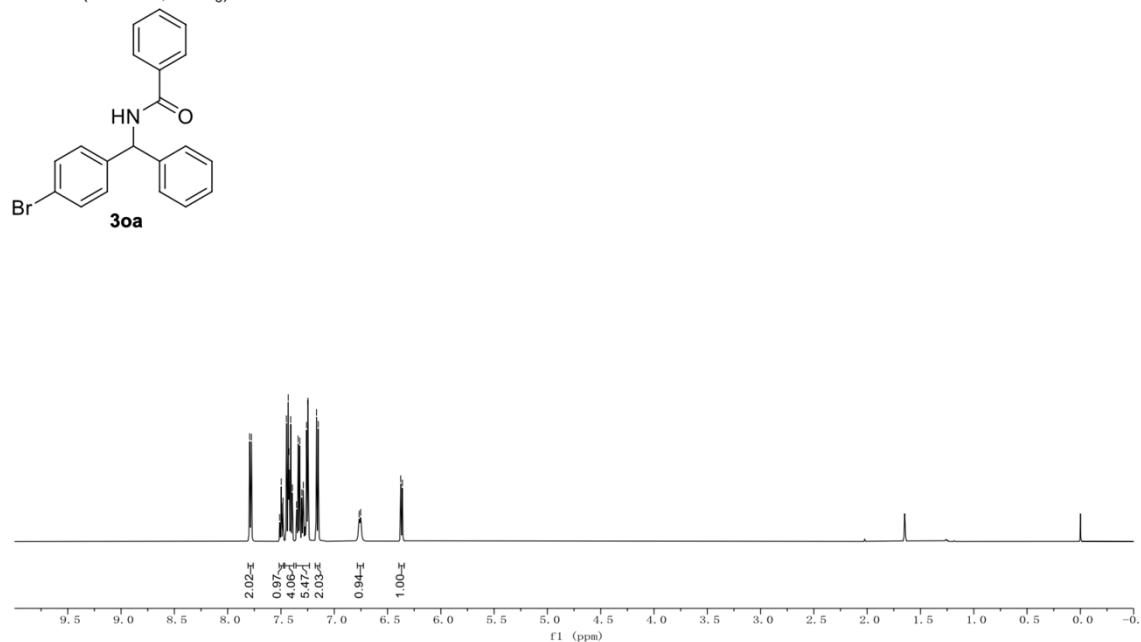
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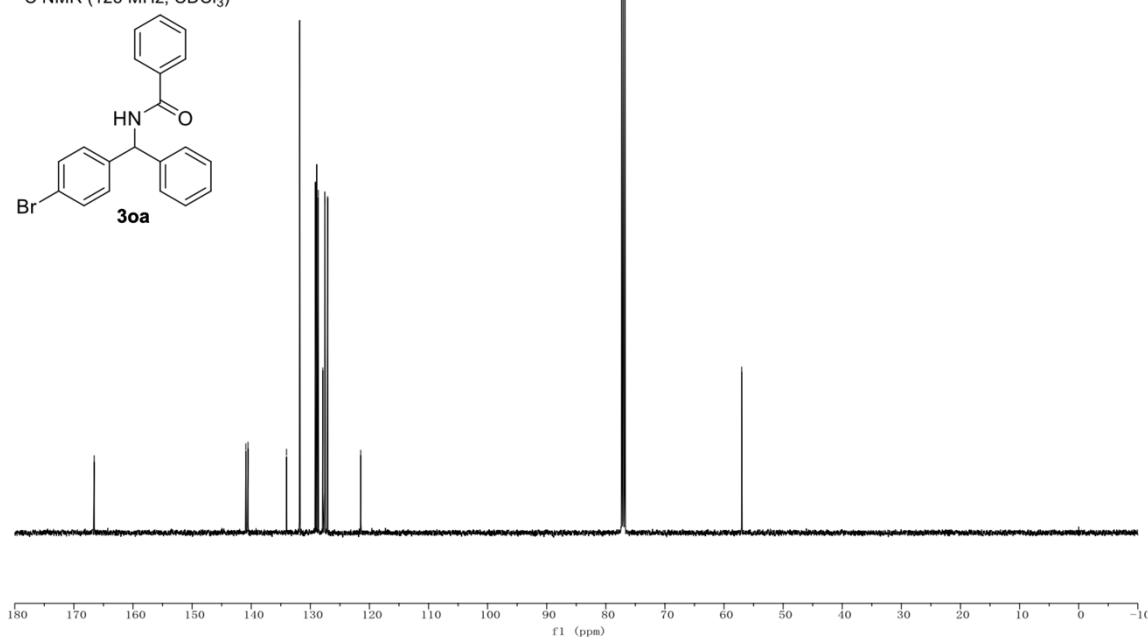


7.7937
7.7774
7.5125
7.4971
7.4812
7.4485
7.4317
7.4244
7.4087
7.3932
7.3532
7.3390
7.3233
7.3047
7.2901
7.2596
7.2470
7.1669
7.1502
6.7666
6.7525
6.3765
6.3611

¹H NMR (500 MHz, CDCl₃)

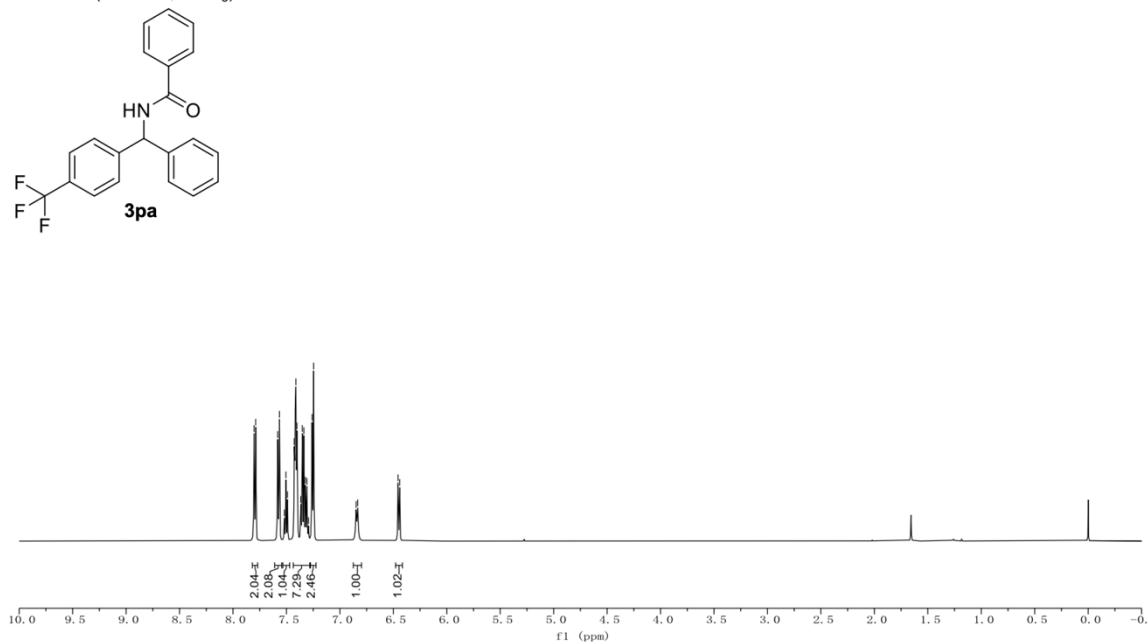


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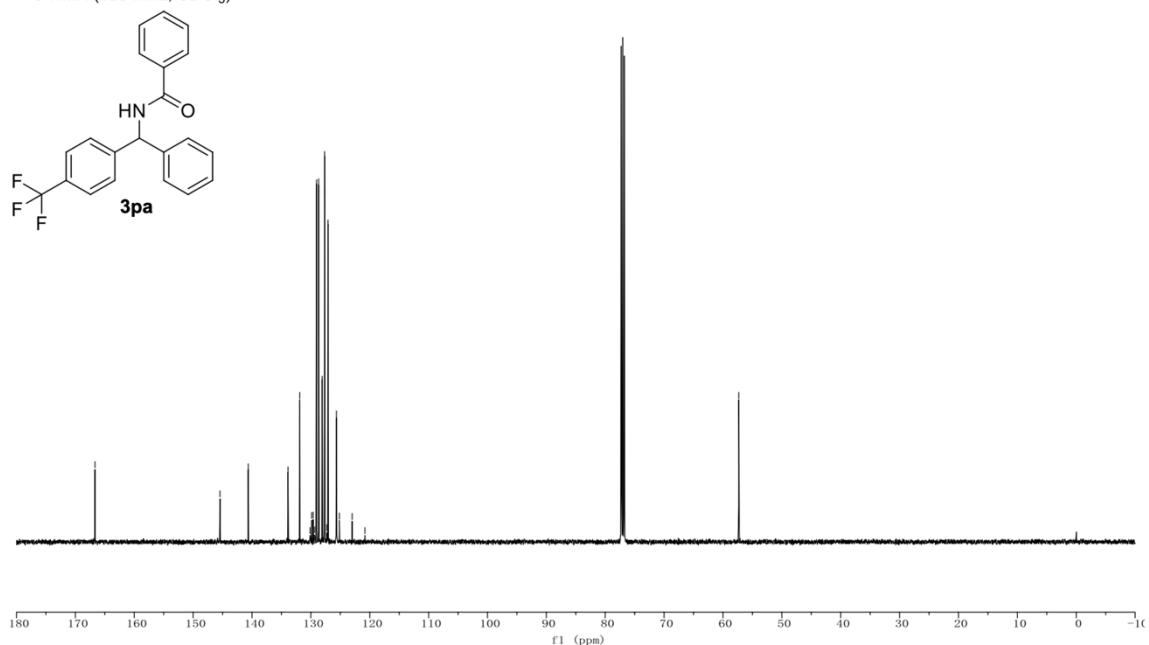


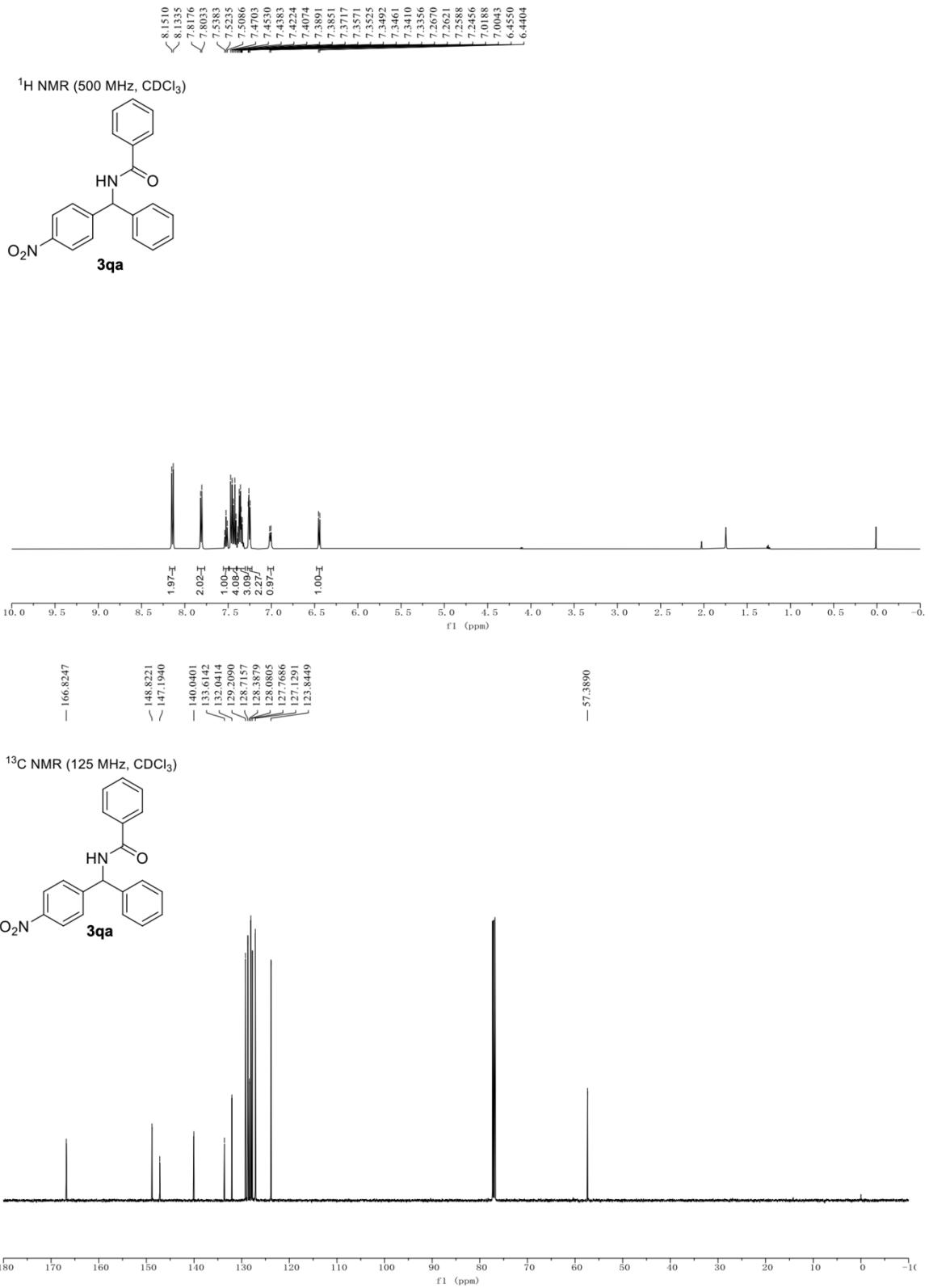


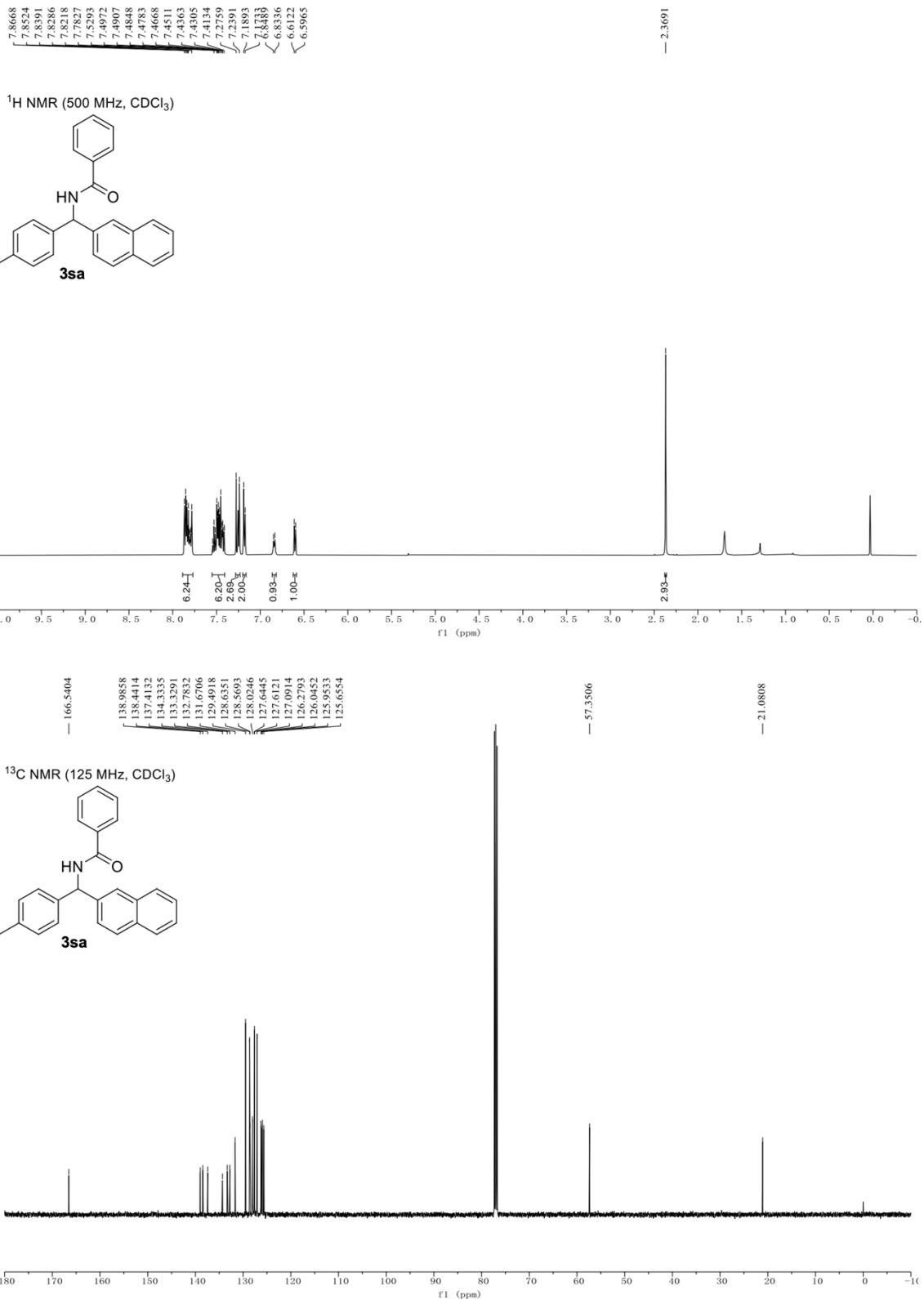
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¹³C NMR (125 MHz, CDCl₃)

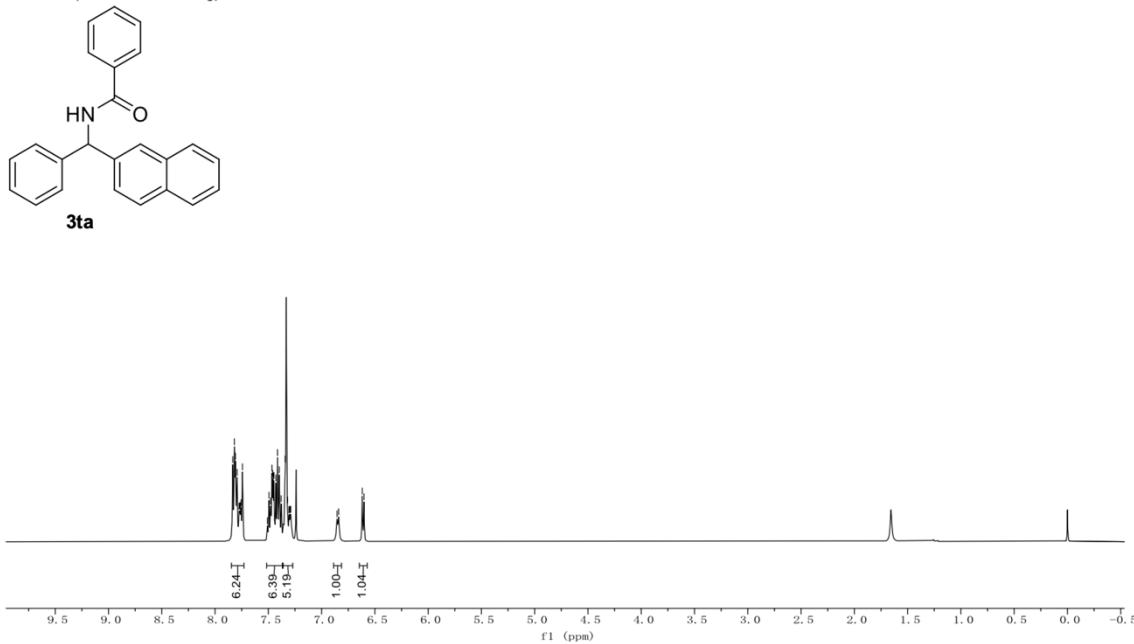




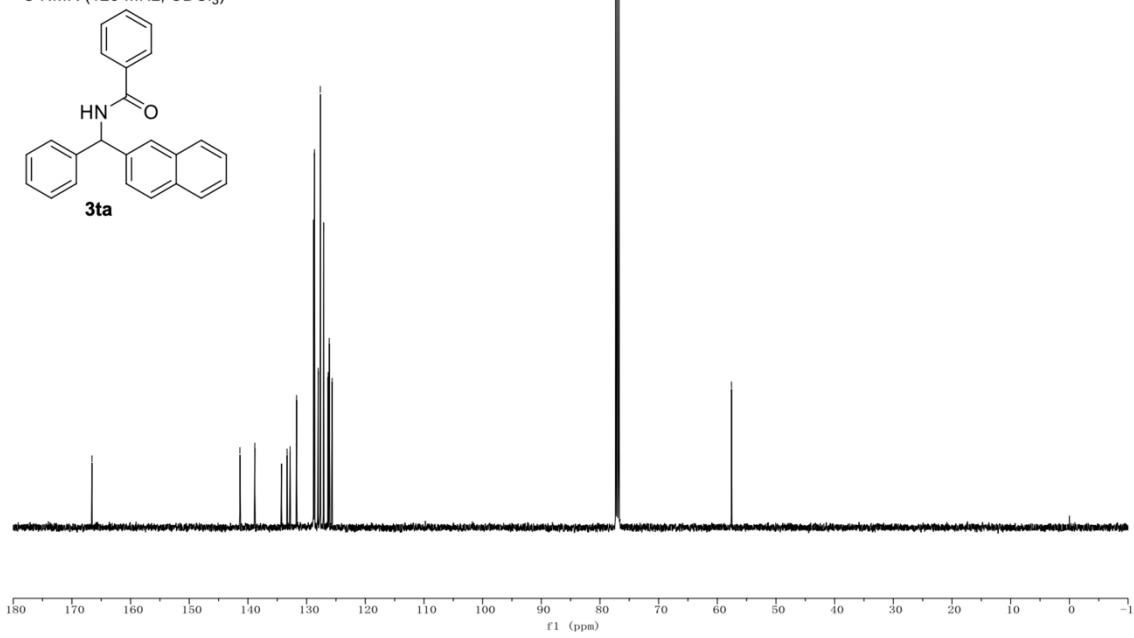


7.8340
 7.8186
 7.8092
 7.7927
 7.7685
 7.7565
 7.7426
 7.4938
 7.4789
 7.4661
 7.4661
 7.4597
 7.4537
 7.4473
 7.4293
 7.4139
 7.3977
 7.3796
 7.3430
 7.3291
 6.8352
 6.8402
 6.6182
 6.6025

¹H NMR (500 MHz, CDCl₃)

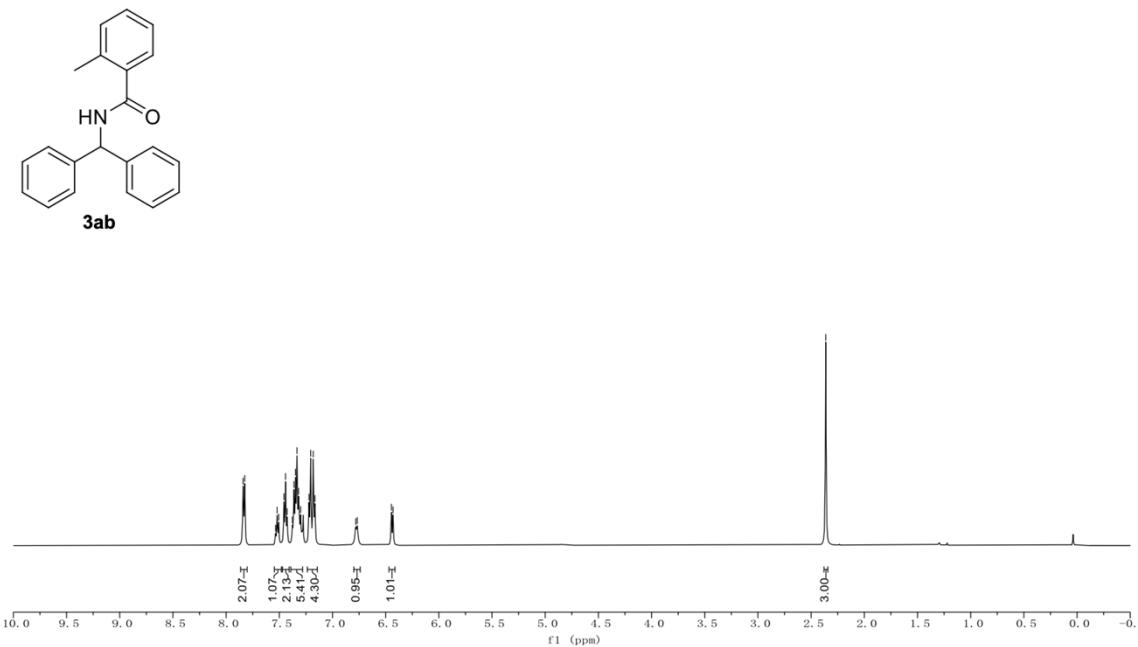


¹³C NMR (125 MHz, CDCl₃)



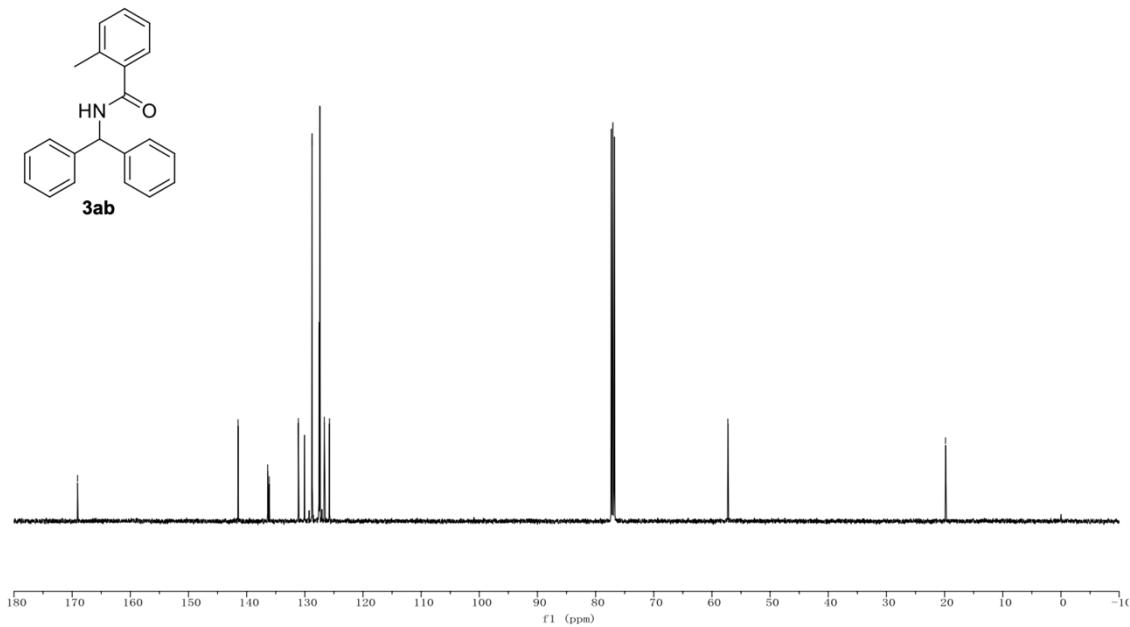
7.8405
 7.8256
 7.5551
 7.5206
 7.5061
 7.4864
 7.4413
 7.4264
 7.3768
 7.3620
 7.3476
 7.3339
 7.3186
 7.2997
 7.2212
 7.2054
 7.1810
 7.1653
 6.7817
 6.7676
 6.4464
 6.4310

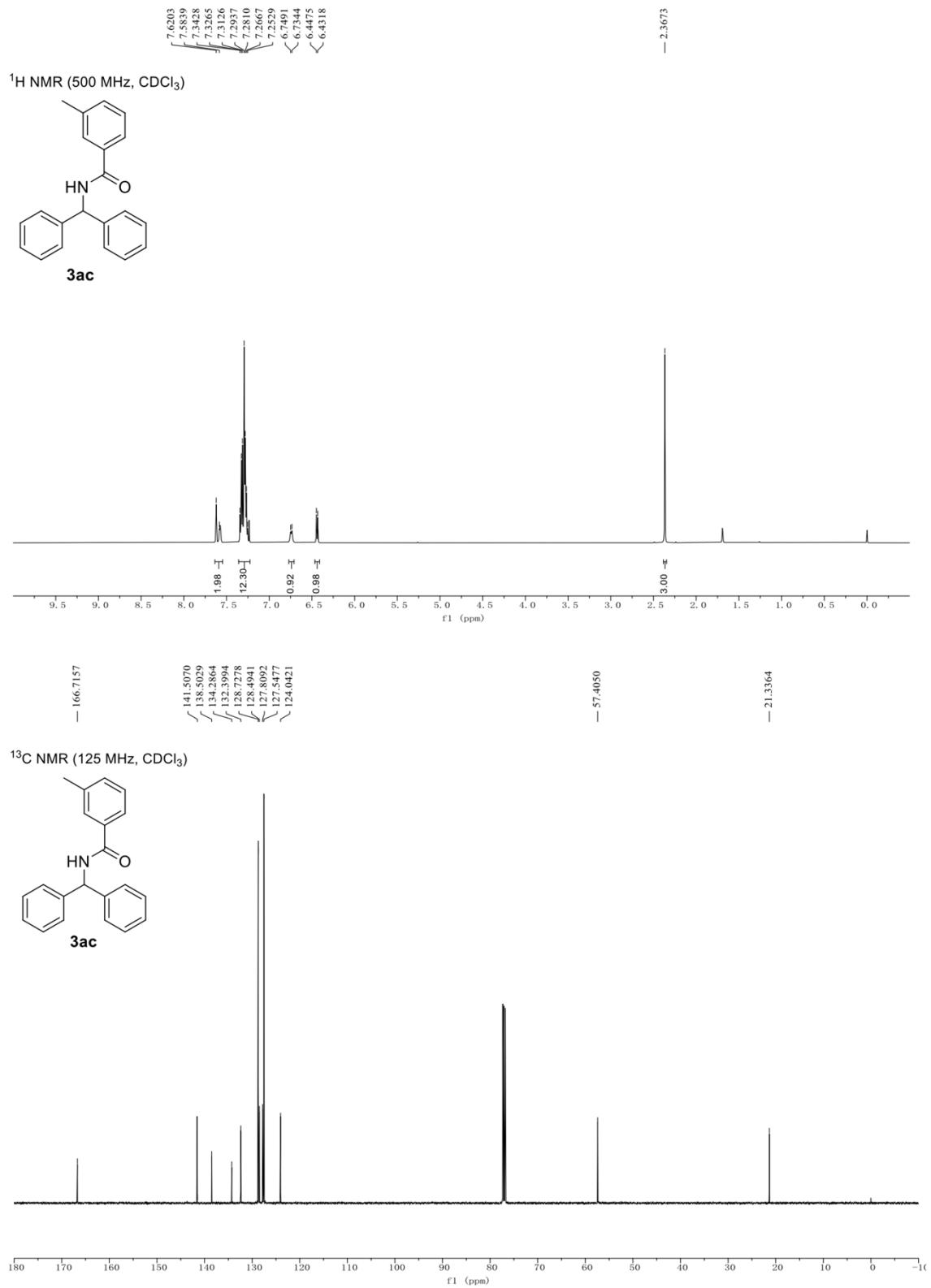
¹H NMR (500 MHz, CDCl₃)

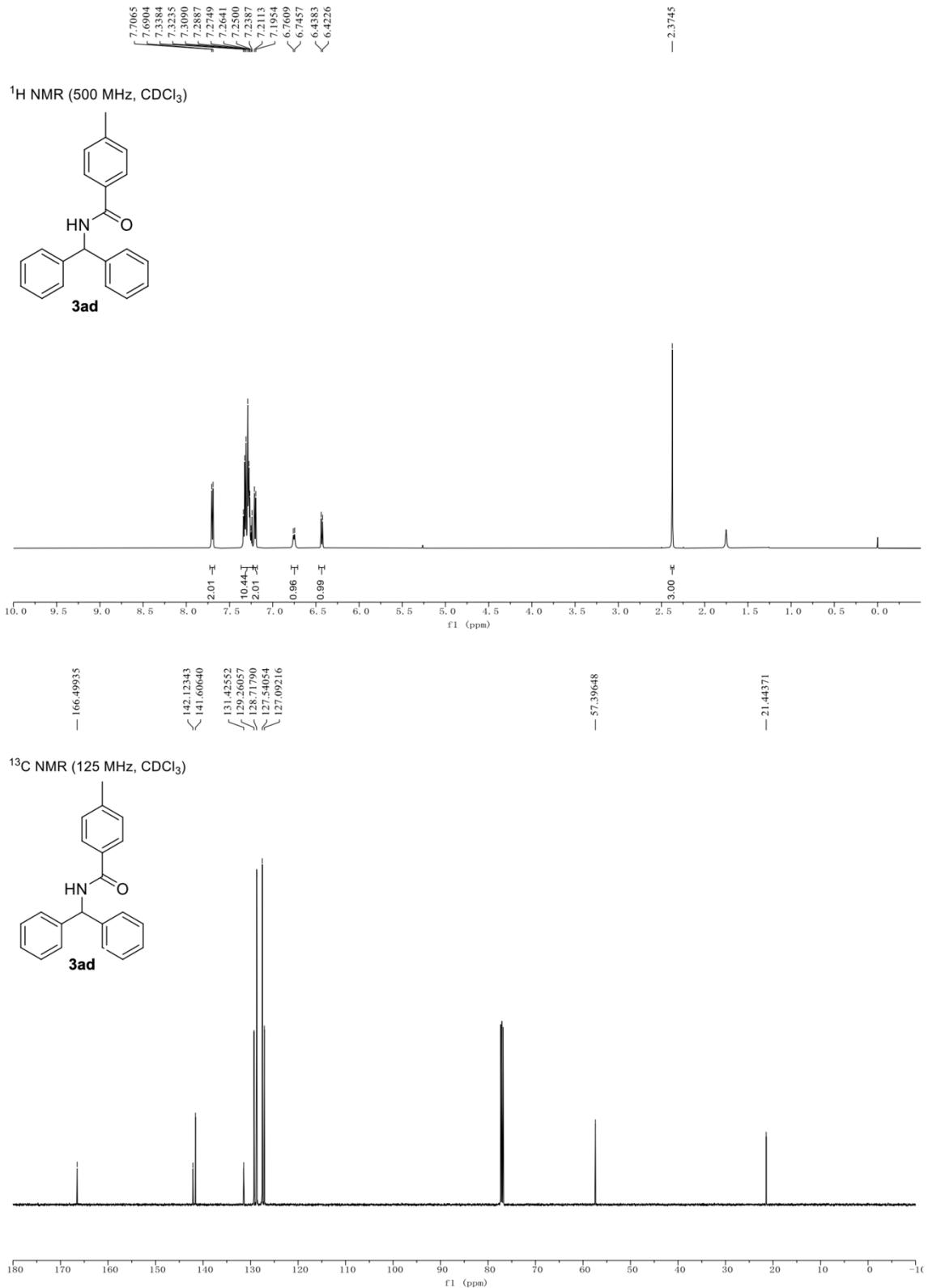


169.0666
 141.4785
 136.3895
 136.1160
 131.1102
 130.0847
 128.7460
 127.5341
 127.4322
 126.6517
 125.7589

¹³C NMR (125 MHz, CDCl₃)

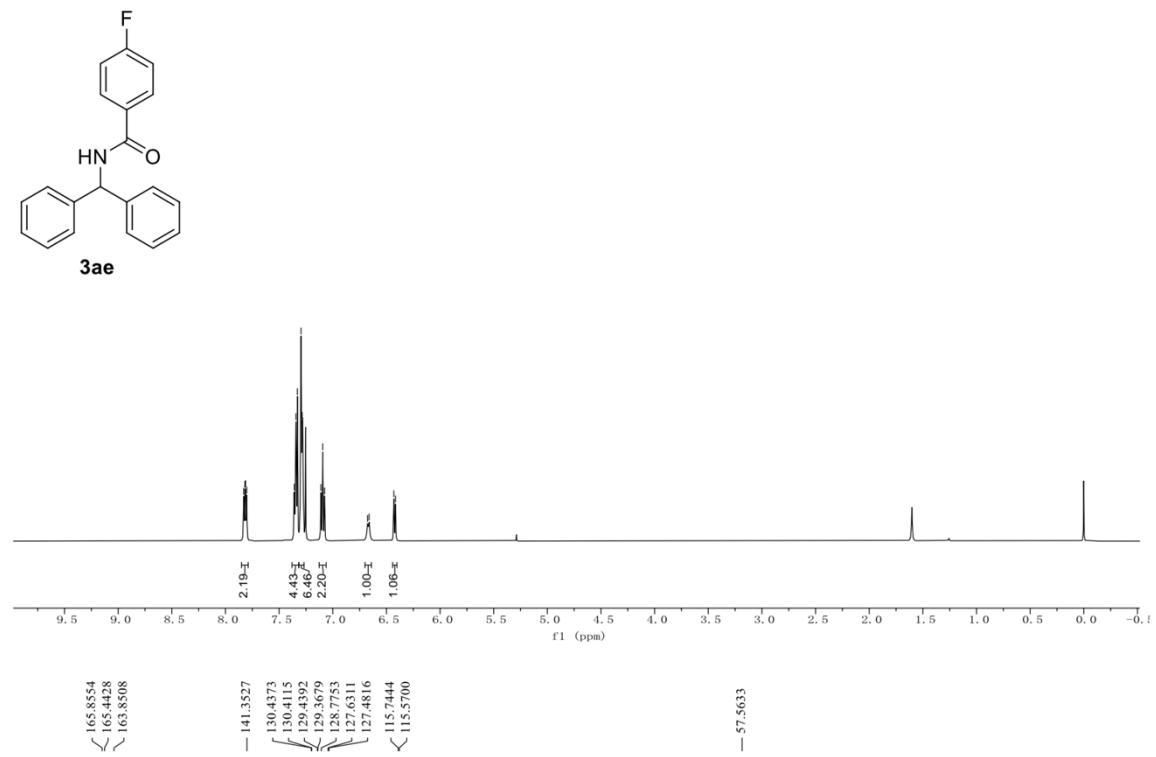




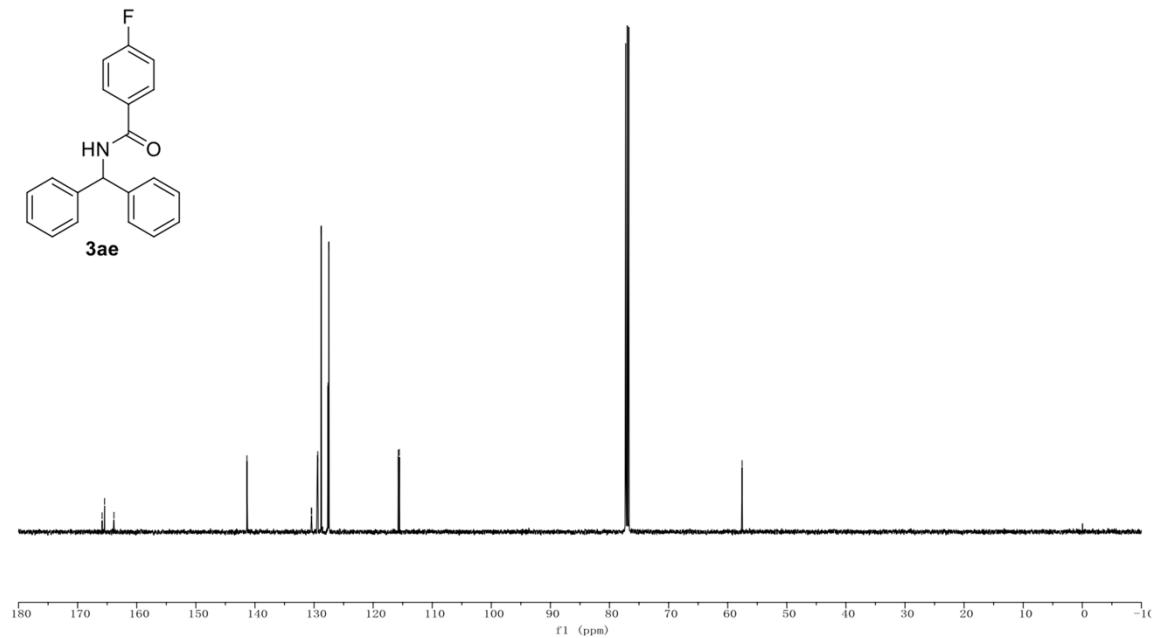




¹H NMR (500 MHz, CDCl₃)

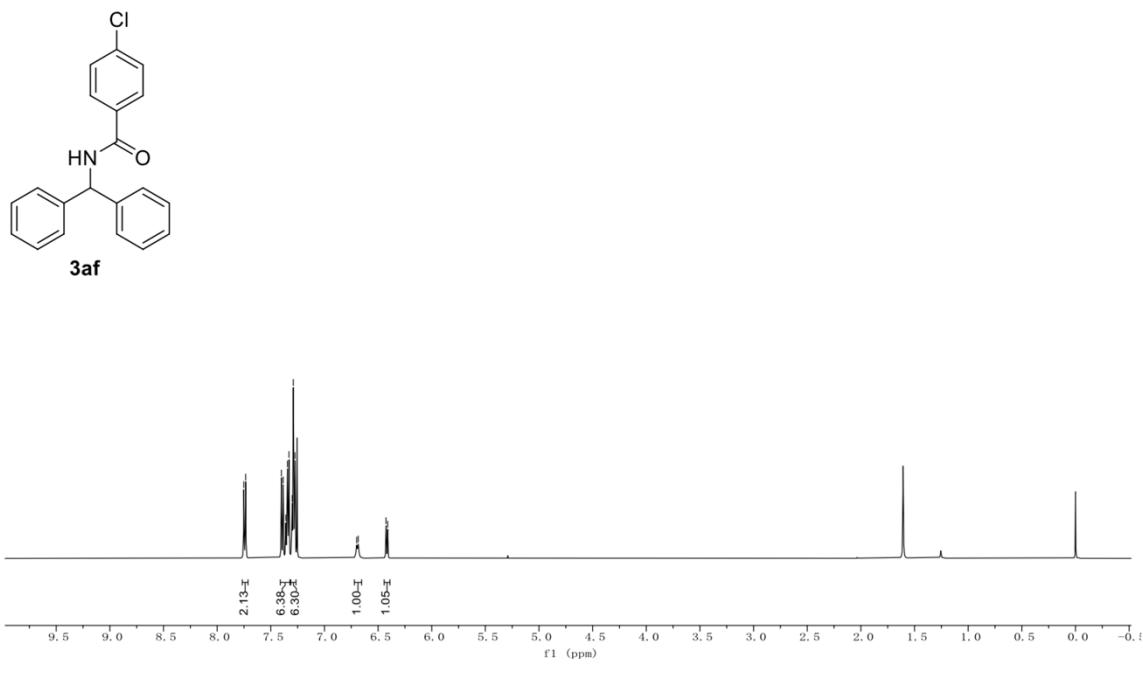


¹³C NMR (125 MHz, CDCl₃)

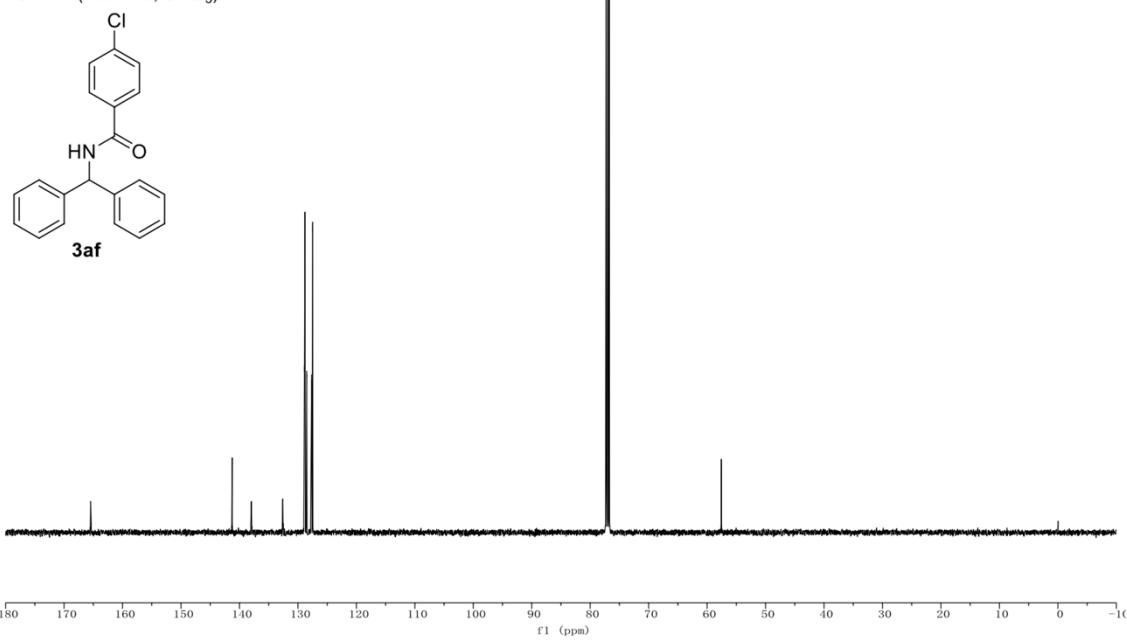




¹H NMR (500 MHz, CDCl_3)

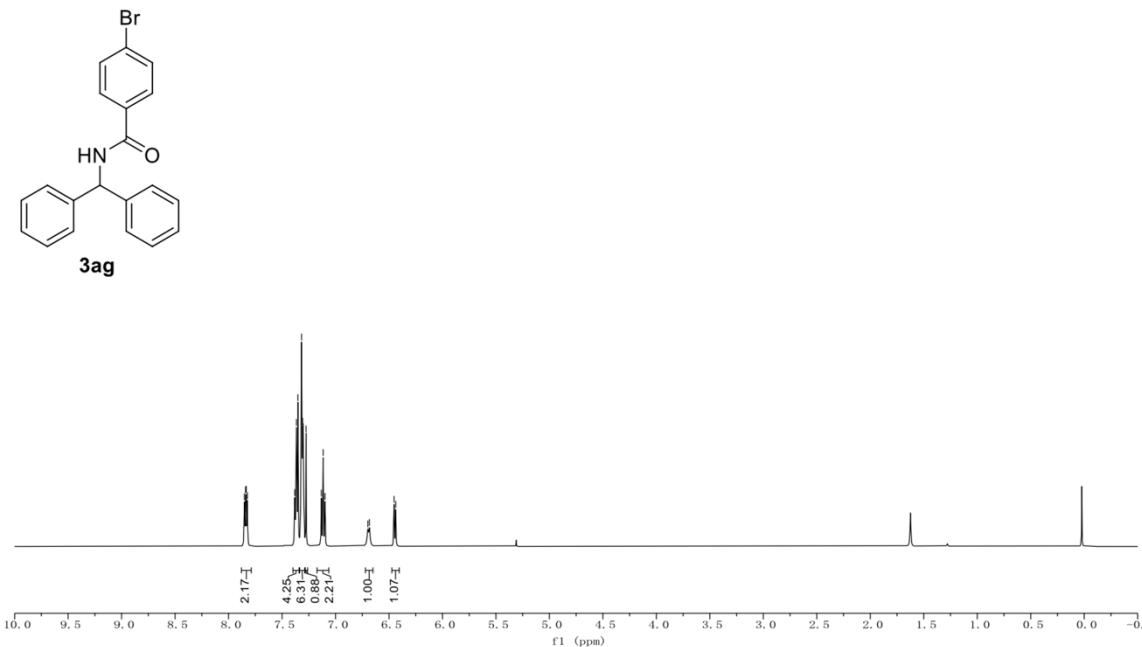


¹³C NMR (125 MHz, CDCl_3)

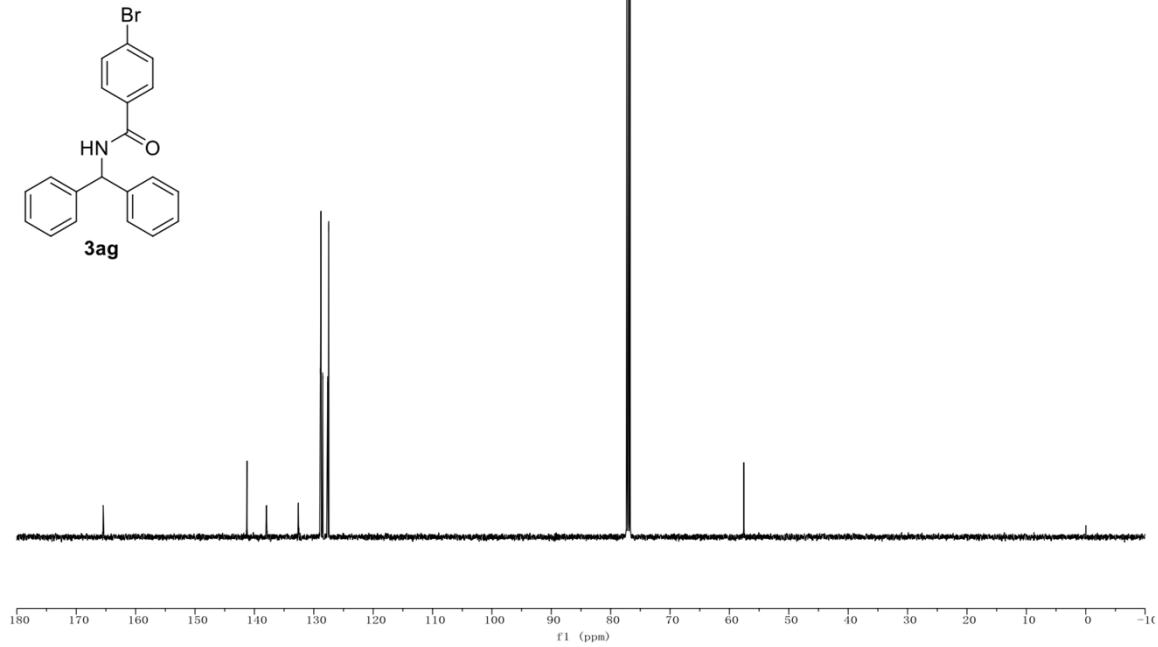


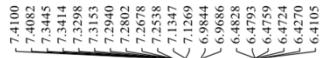


¹H NMR (500 MHz, CDCl₃)

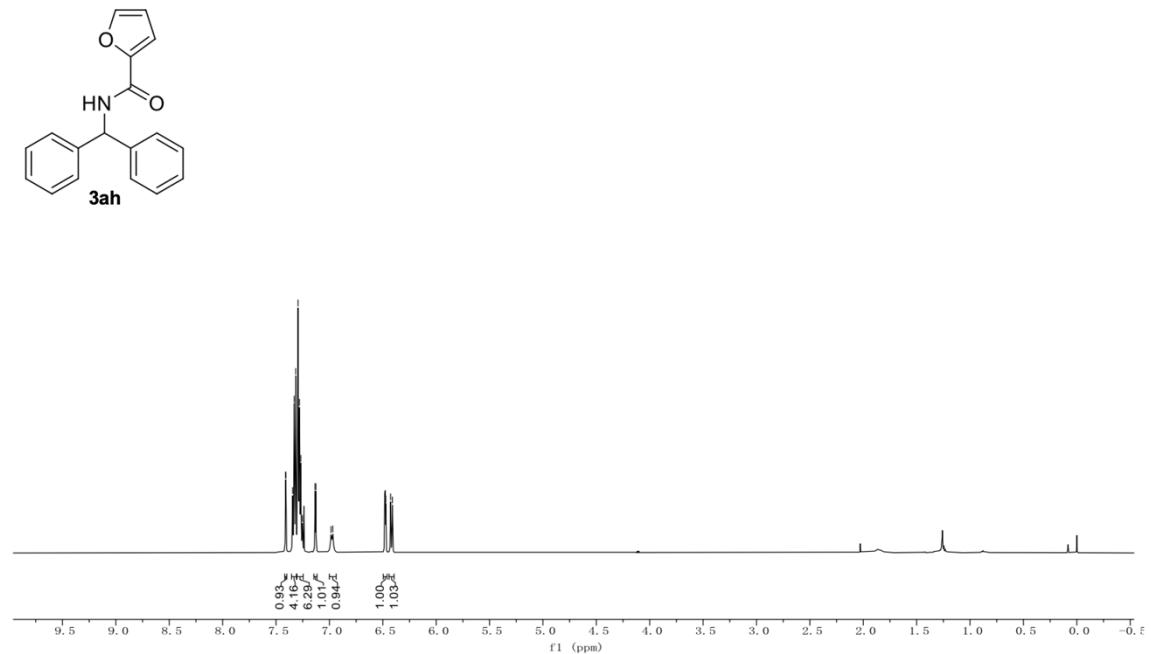


¹³C NMR (125 MHz, CDCl₃)

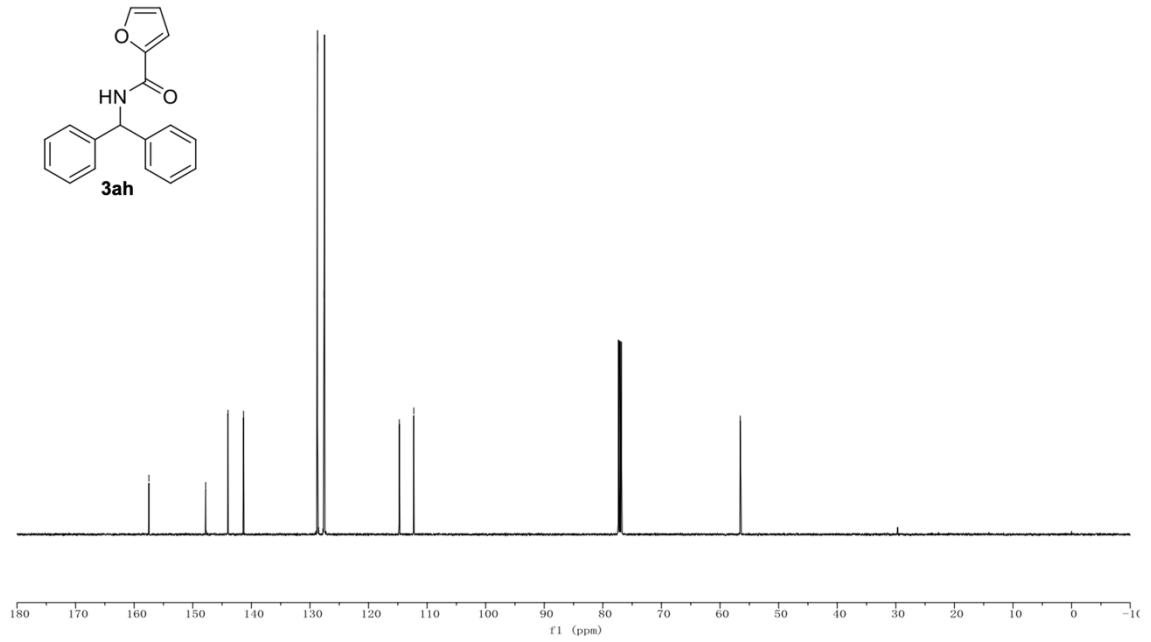




¹H NMR (500 MHz, CDCl₃)

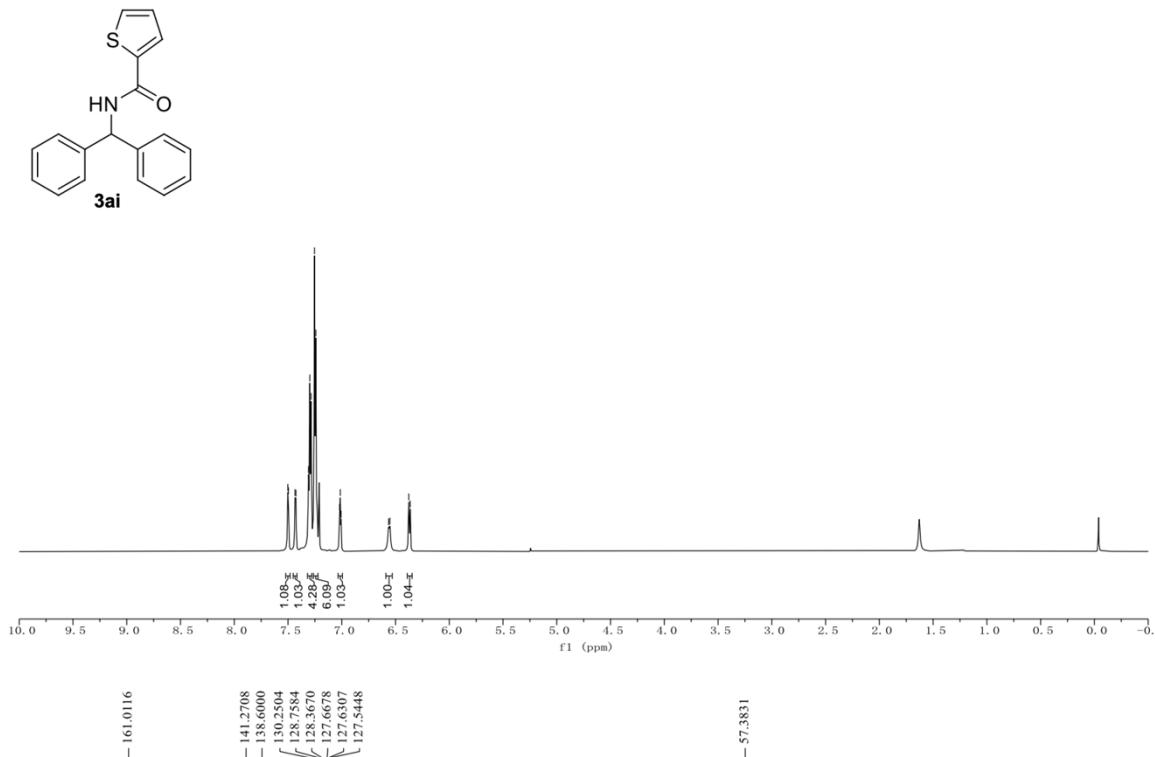


¹³C NMR (125 MHz, CDCl₃)

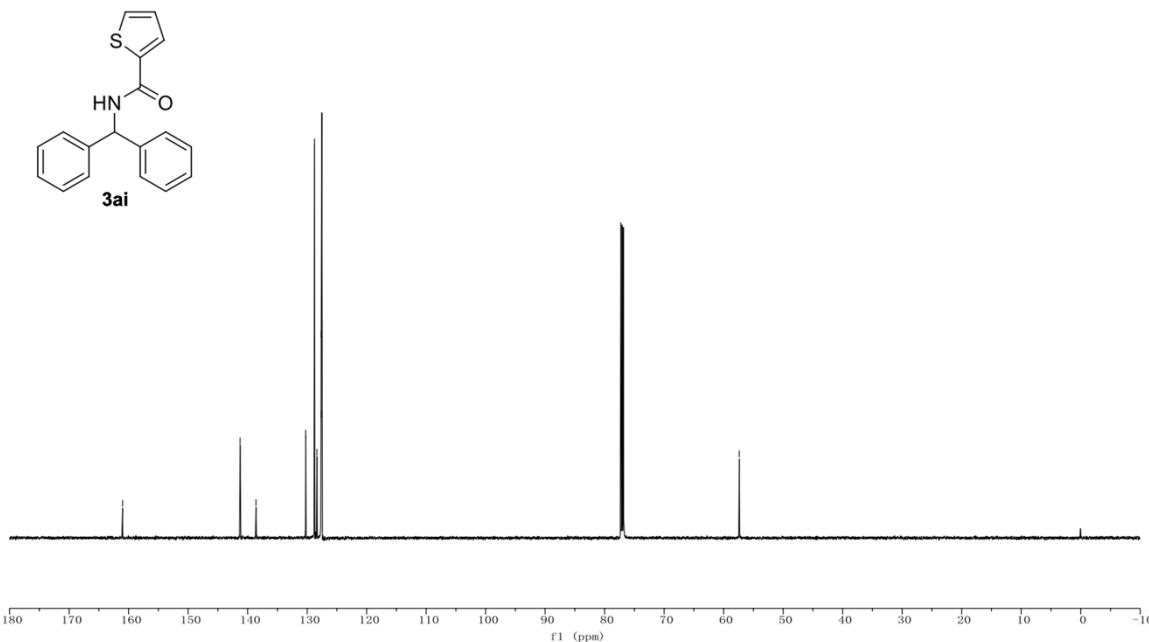




¹H NMR (600 MHz, CDCl₃)

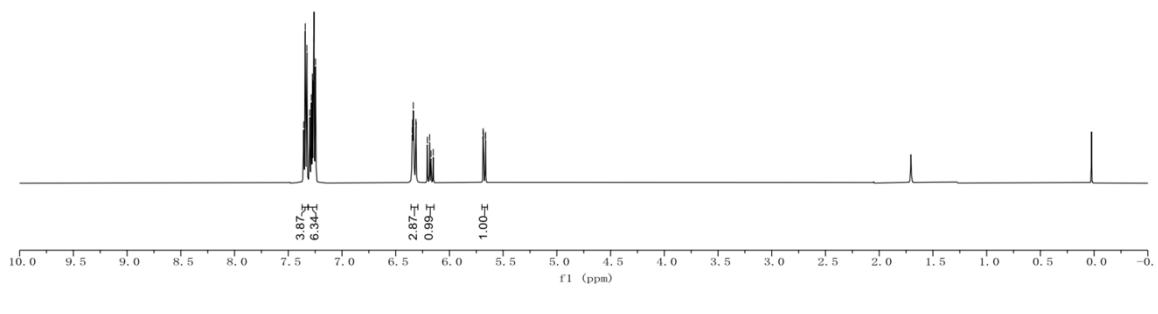


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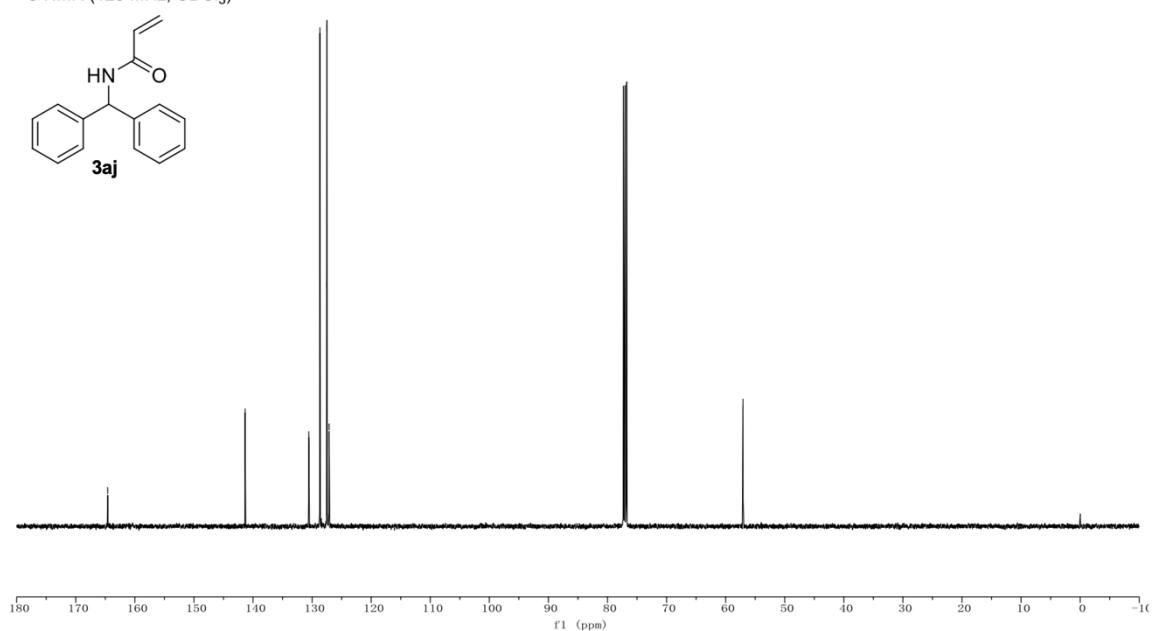




¹H NMR (500 MHz, CDCl₃)

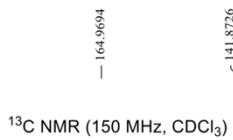
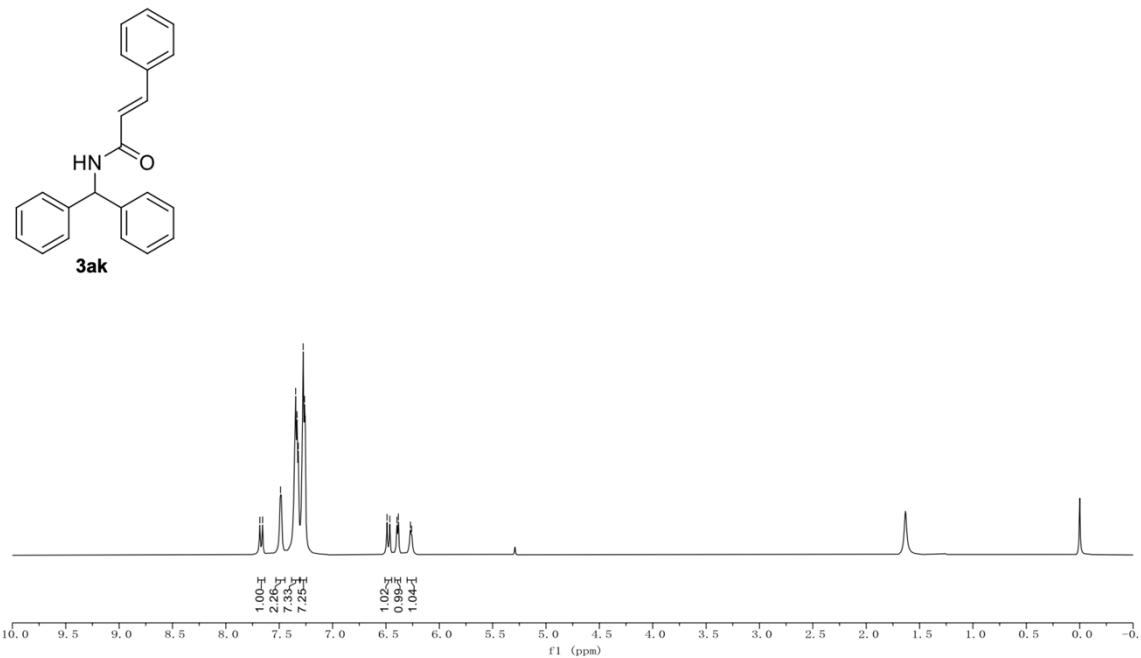


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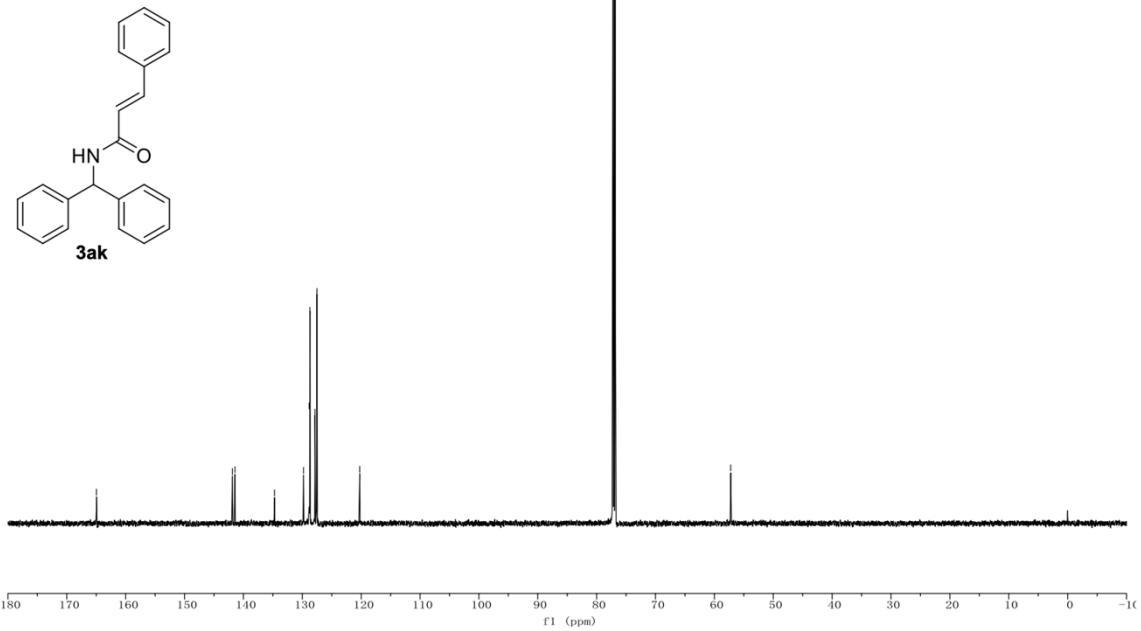


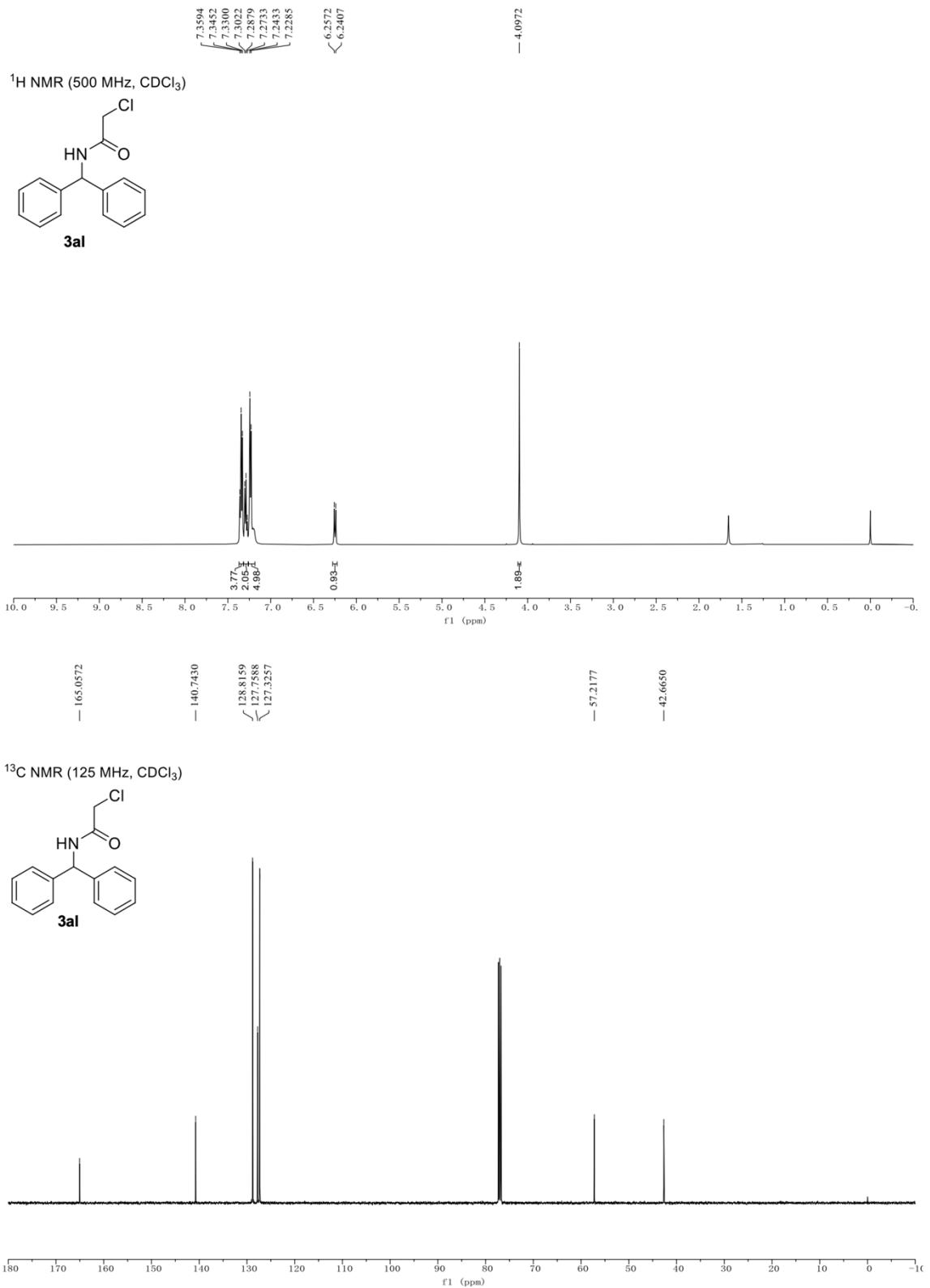


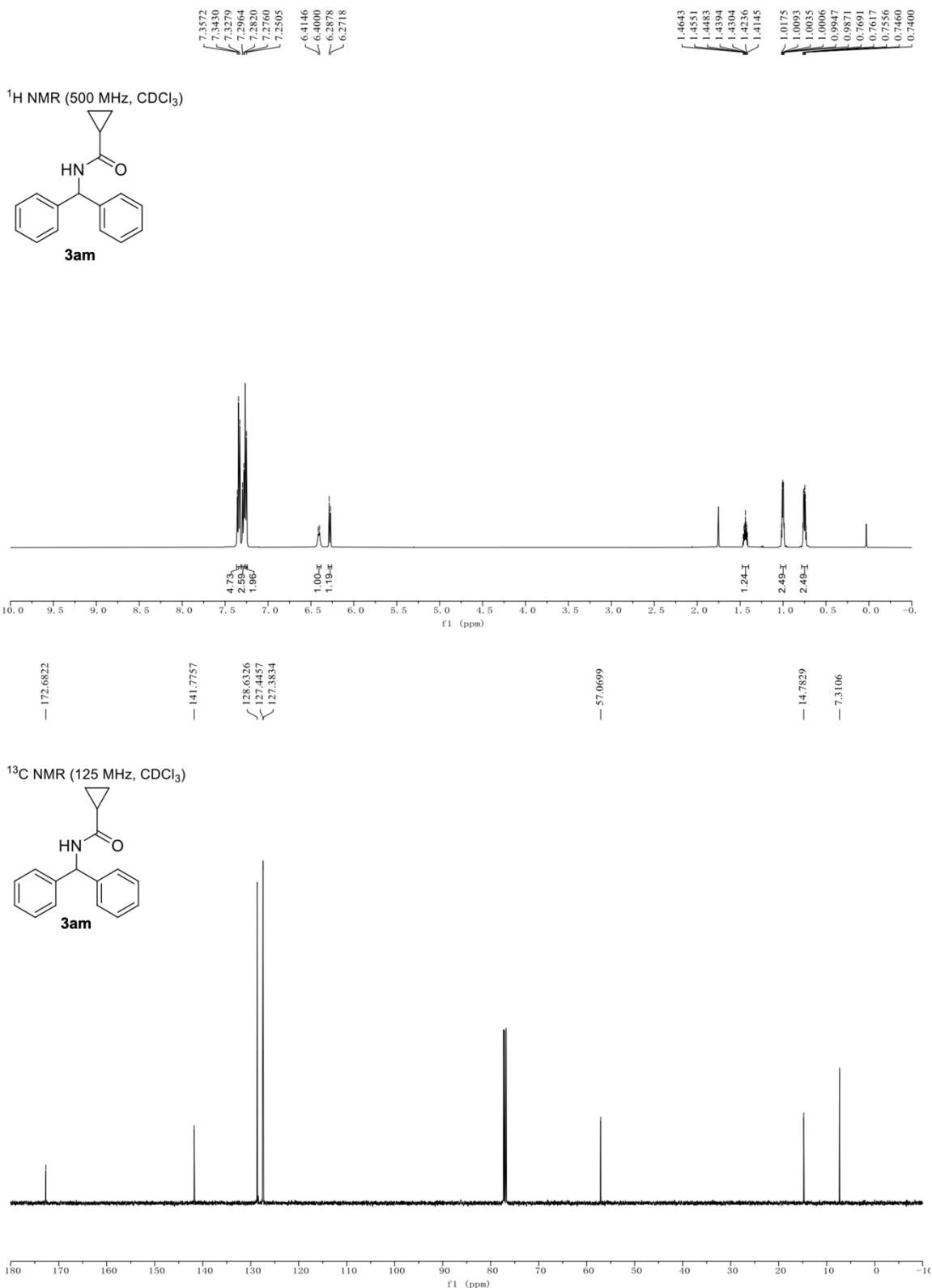
¹H NMR (600 MHz, CDCl₃)

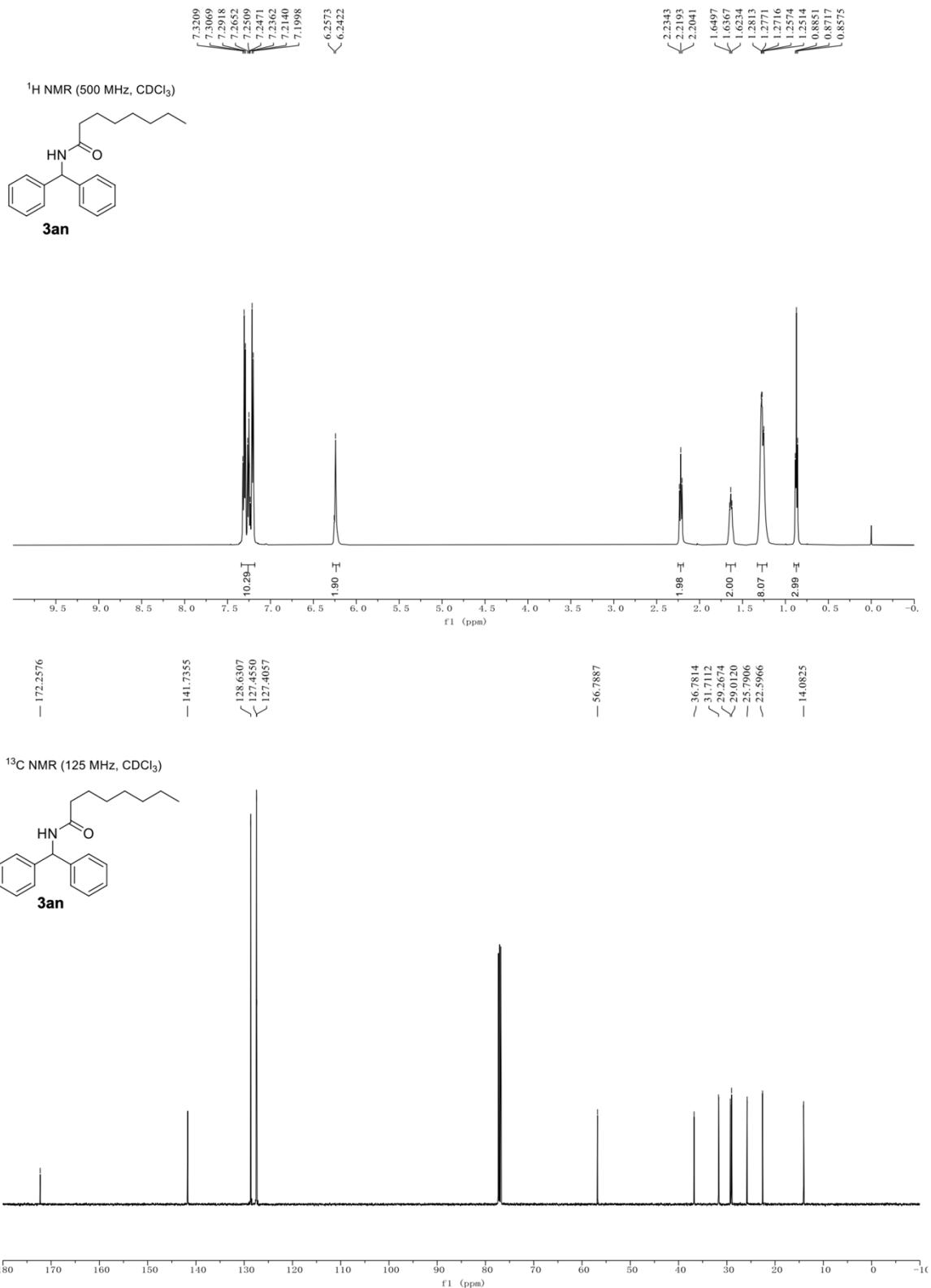


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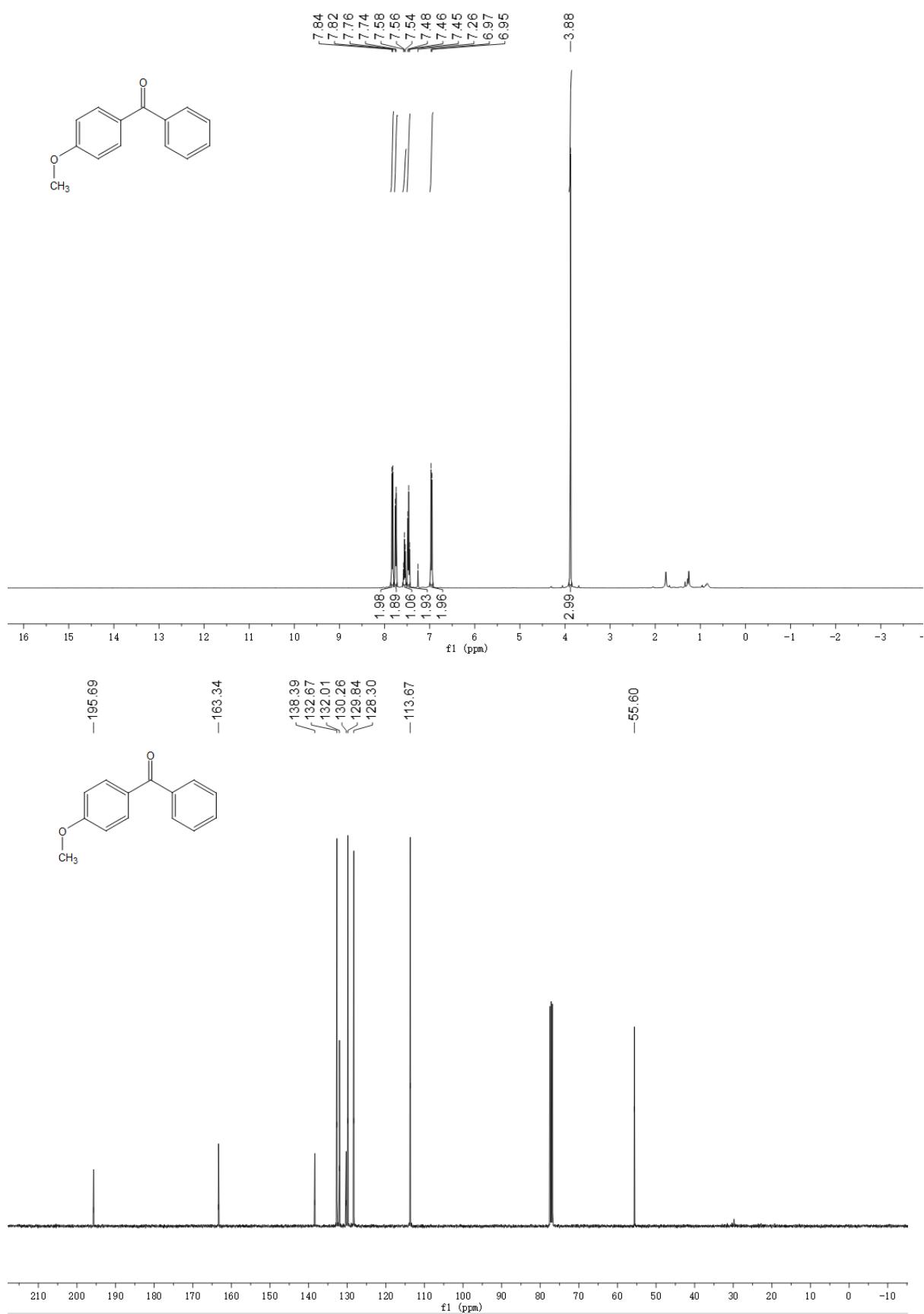








(4-methoxyphenyl)(phenyl)methanone



5 References

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