Supporting Information

Photoredox-catalyzed alkylarylation of activated alkenes via a ring-opening/Truce-Smiles rearrangement cascade

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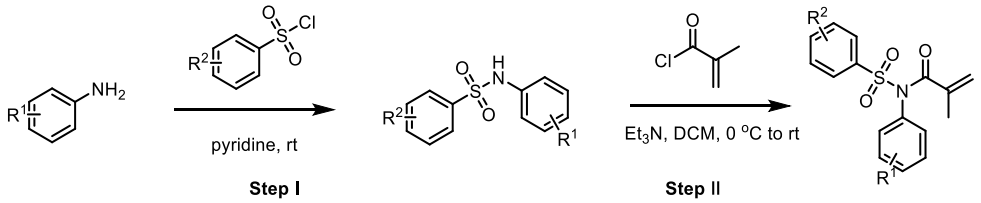
1. General Information

The experiments were conducted using an oven-dried Schlenk-tube. Photoinduced reactions were performed in an oven-dried Schlenk-tube with Wattecs Blue LEDs Irradiation Parallel Reactor. Unless otherwise specified, all reagents were procured from commercial sources and utilized without additional purification. ^{1}H and ^{13}C NMR spectra were collected using a Bruker 400 MHz (100 MHz for ^{13}C NMR) spectrometer at room temperature. Chemical shifts were reported in parts per million (ppm) relative to tetramethylsilane (TMS) with the solvent resonance serving as the internal standard ($CDCl_3$: ^{1}H NMR: δ = 7.26; ^{13}C NMR: δ = 77.0). Coupling constants were denoted in Hertz (Hz) with respective multiplet type abbreviations: s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), td (triplet of doublets), and m (multiplet) HRMS were obtained on a WATERS I-Class VION IMS Q-Tof. Analytical TLC: aluminum backed plates pre-coated (0.25 mm) with Merck Silica Gel 60F-254. Compounds were visualized by exposure to UV-light or by dipping the plates in 2,4-dinitrophenylhydrazine stain followed by heating.

2. Starting Materials

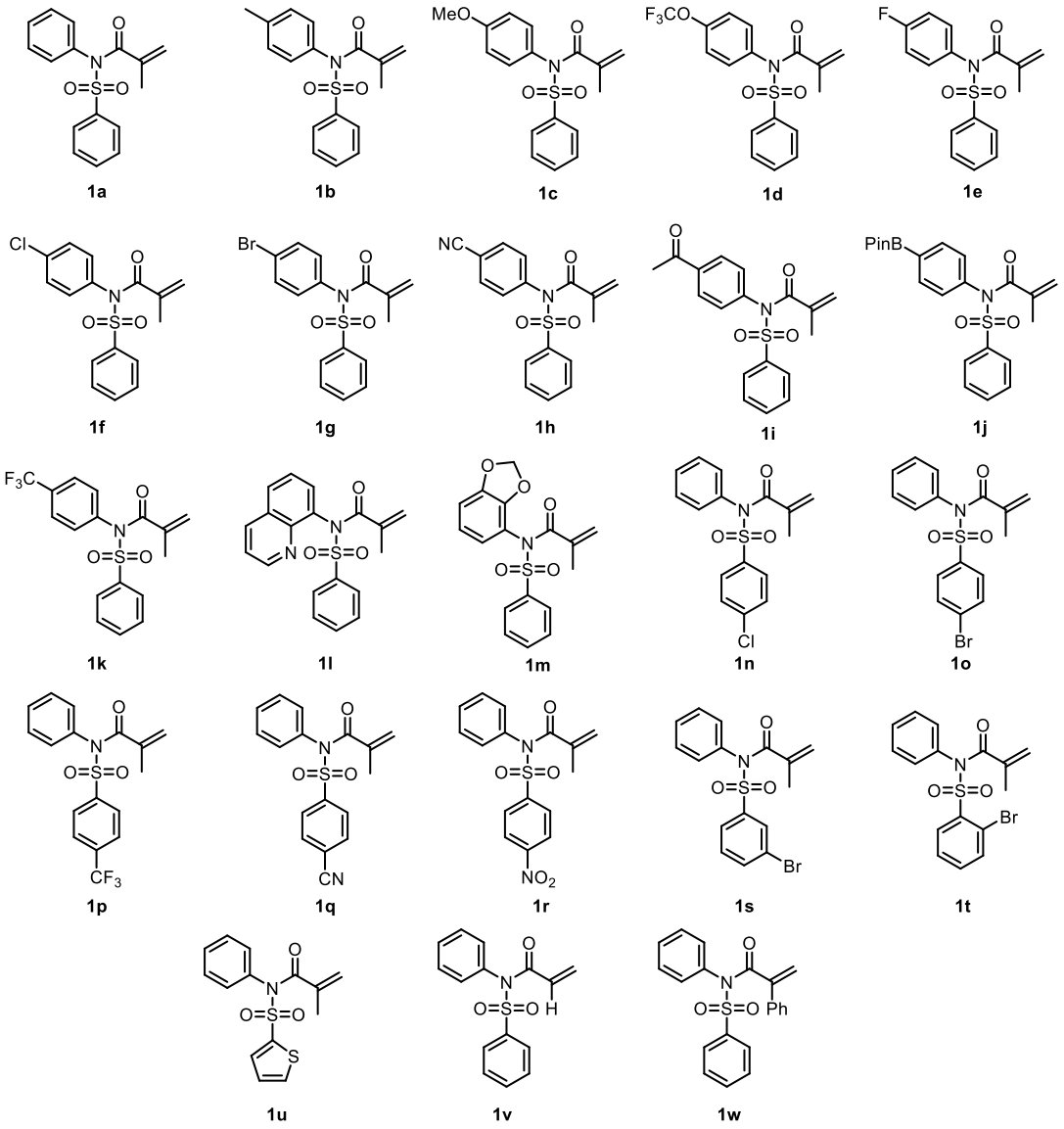
The *N*-aryl-*N*-(arylsulfonyl) acrylamides **1** and cycloalkyl hydroperoxides **2** were prepared according to the literature. The NMR spectra of the know compounds were in full accordance with the data in the literatures.

2.1 General Procedure for the Synthesis of *N*-Aryl-*N*-(arylsulfonyl)methacrylamides 1[1]



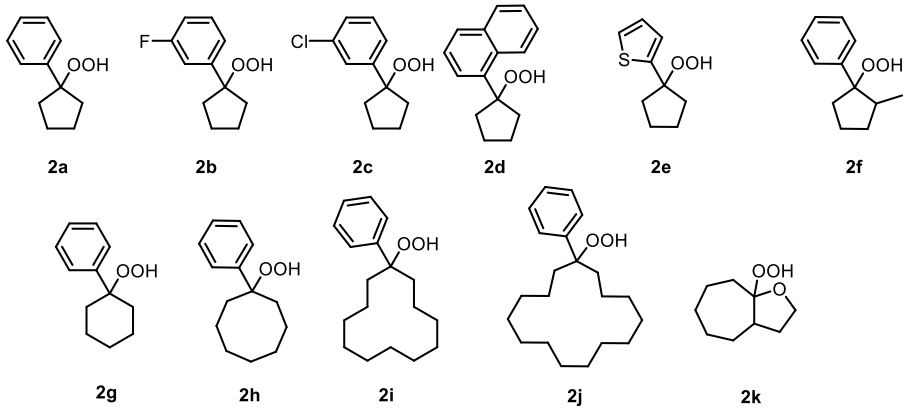
Step I: In a dry 50 mL round-bottom flask, a solution of arylamine (10 mmol) in 20 mL of pyridine was subjected to stirring at room temperature. The benzene sulfonyl chloride (11 mmol, 1.1 equiv.) was added slowly. The progress of the reaction was monitored by thin-layer chromatography (TLC). Then the reaction mixture was poured into water and the aqueous layer was extracted with DCM (3 × 15 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude products were purified by flash column chromatography to provide *N*-phenylbenzenesulfonamide.

Step II: In a dry 100 mL round-bottom flask, a mixture of *N*-phenylbenzenesulfonamide (10 mmol) and Et_3N (30 mmol) in 20 mL of DCM were stirred in an ice bath. Subsequently, methacryloyl chloride (12 mmol, 1.2 equiv.) was cautiously added via a dropping funnel. The progress of the reaction was monitored by thin-layer chromatography (TLC). Then the reaction was quenched by H_2O (20 mL), and the aqueous layer was extracted with DCM (3 × 15 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography to provide amide products.

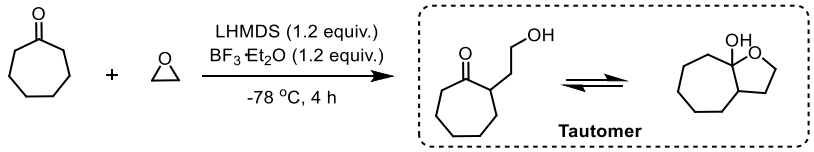


2.2 General Procedure for the Synthesis of Cycloalkyl Hydroperoxides 2a-2j.

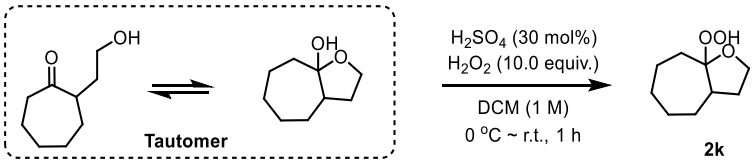
The cycloalkyl hydroperoxides **2a-2j** were prepared according to the literature[2]. The NMR spectra of the known compounds were in full accordance with the data in the literature



2.3 General Procedure for the Synthesis of Hemiketal Hydroperoxide 2k.[3]



To a stirred solution of LiHMDS (1.0 M in THF, 1.2 equiv.) in THF at -78 °C was added cycloheptanone (10 mmol, 1.0 equiv.) over 5 mins. After 1 h, epoxide (3.0 M in THF, 2.0 equiv.) was added to the reaction solution. After 1 h, the $BF_3{\cdot}Et_2O$ (1.2 equiv.) was added very slowly. The reaction mixture was stirred at -78 °C for 2 h and the reaction was quenched by the addition of saturated aqueous NH_4Cl solution at -78 °C. Layers were separated and the aqueous layer was extracted with EtOAc. The combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc) to obtain the tautomer of α-hydroxyalkylketones and lactols.



Based on the amount of the tautomer of α-hydroxyalkylketones and lactols: To a reaction flask was added a solution of H_2O_2 (30% wt in H_2O, 10.0 equiv.), and conc. H_2SO_4 (30 mol %). Then a solution of the tautomer of α-hydroxyalkylketones and lactols (1.0 equiv.) in DCM (1.0 M) at 0 °C was added. The reaction mixture was stirred for 1 h at room temperature. The aqueous layer was extracted with DCM, the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc) to obtain the hemiketal hydroperoxides **2k**.

2.4 Notes for Hydroperoxides

These α-hydroxyalkyl ketones readily tautomerize to the lactols in solution, and the ratio is easily changed under acid or basic conditions, concentration, solvent, temperature and other conditions. This property was also described in these articles: *Tetrahedron*, 1969, **25**, 3157; *Tetrahedron*, 1987, **43**, 3371; *Angew. Chem., Int. Ed*., 2021, **60**, 5370. All hydroperoxides were heated and concentrated by vacuo at below 30 °C and were stored under -20 ℃. We have never experienced a safety problem with these materials. Hydroperoxides are bicyclic fused compounds with an inseparable mixture of *cis-trans* isomers, we have tried our best to get cleaner ^{1}H NMR and ^{13}C NMR spectra of hydroperoxides.

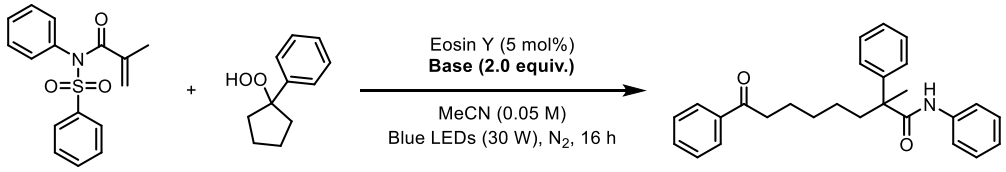
3. Detailed Optimization of Reaction Conditions

3.1 General Procedure for the Reaction of Acrylamides 1a and Hydroperoxide 2a.

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with *N*-phenyl-*N*-(phenysulfonyl)acrylamide **1a** (0.20 mmol, 1.0 equiv.), photocatalyst (0.01mmol, 0.05 equiv.). Then, the tube were evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cyclopentyl hydroperoxide **2a** (0.40 mmol, 2.0 equiv.) in solvent (0.05 M) were added by a syringe. The reaction mixture was stirred under the irradiation of a 30 W Blue LED (λ = 460−470 nm) for a specified time. After that, the resulting mixture were quenched with H_2O and extracted with EtOAc (3 x 10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and concentrated in vacuo. Purification of the crude product by flash chromatography on silica gel (petroleum ether/EtOAc = 15:1 to 10:1) furnishes the desired product **3aa** as colorless oil.

3.2 Optimization of the Reaction of Acrylamides 1a and Hydroperoxides 2a.

Base[a]

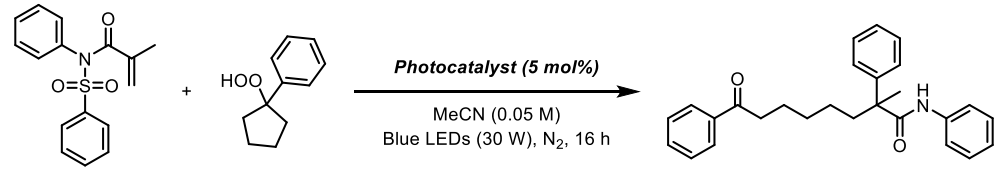


1a 2a 3aa

Entry	Base	Yield (**3aa**, %)[a]
1	**No base**	**35**
2	K_2HPO_4	12
3	KH_2PO_4	10
4	K_3PO_4	9
5	K_2CO_3	7
6	Na_2CO_3	trace
7	TEA	16
8	DABCO	20
9	DIPEA	6
10	DMAP	7

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Eosin Y (5 mol%), Base (2.0 equiv.), MeCN (0.05 M), Blue LEDs (30 W), rt, for 16 h, under N_2. Yield of isolated product.

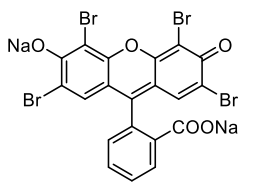
Photocatalyst (PC)[a]



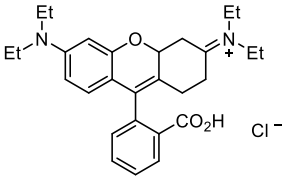
1a 2a 3aa

Entry	Photocatalyst	Yield (**3aa**, %)[a]
1	Eosin Y	35
2	**Rhodamine B**	**42**
3	Fluorescein	20
4	4CzIPN	35
5	Riboflavin	25
6	Erythrosine B	trace
7	PQ	20
8	*fac*-Ir(ppy)$_3$	35

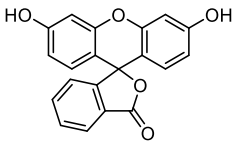
[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), photocatalyst (5 mol%), MeCN (0.05 M), Blue LEDs (30 W), rt, for 16 h, under N_2. Yield of isolated product.



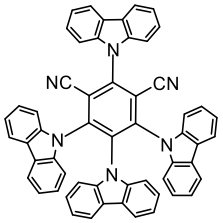
Eosin Y



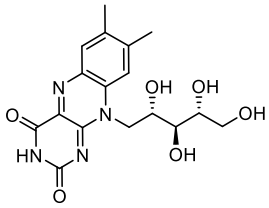
Rhodamine B



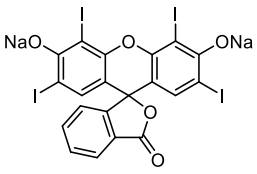
Fluorescein



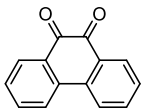
4CzIPN



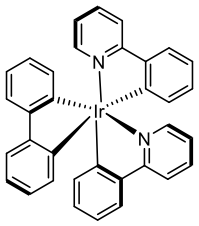
Riboflavin



Erythrosin B

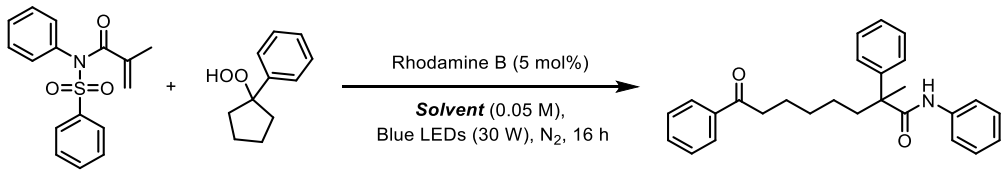


PQ



fac-Ir(ppy)$_3$

Solvent[a]

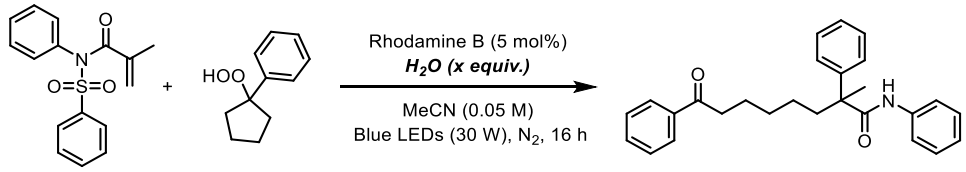


1a **2a** **3aa**

Entry	Solvent	Yield (**3aa**, %)[a]
1	**CH_3CN**	**42**
2	1,4-dioxane	35
3	MeOH	36
4	EtOH	39
5	DMF	18
6	DCE	16
7	$PhCF_3$	25
8	EtOAc	28
9	THF	30
10	toluene	trace

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Rhodamine B (5 mol%), Solvent (0.05 M), Blue LEDs (30 W), rt, for 16 h, under N_2. Yield of isolated product.

Amount of H_2O[a]

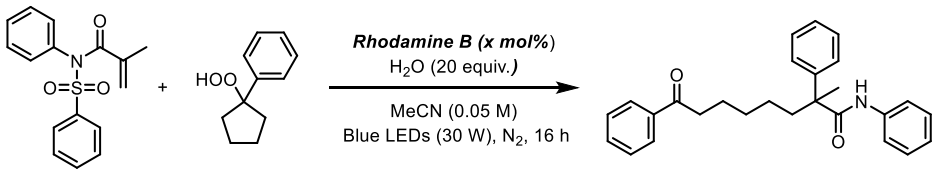


1a **2a** **3aa**

Entry	H_2O (x equiv.)	Yield (**3aa**, %)[a]
1	0	42
2	10	64
3	**20**	**67**
4	30	66
5	40	63

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Rhodamine B (5 mol%), H_2O (x equiv.), MeCN (0.05 M), Blue LEDs (30 W), rt, for 16 h, under N_2. Yield of isolated product.

Loading amount of PC[a]

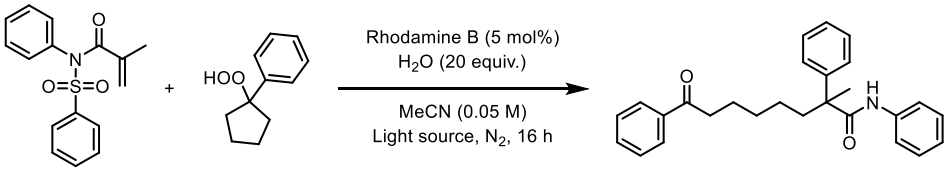


1a **2a** **3aa**

Entry	Rhodamine B (x mol%)	Yield (**3aa**, %)[a]
1	1	53
2	2.5	59
3	**5**	**67**
4	7.5	67
5	10	63

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Rhodamine B (x mol%), H_2O (20 equiv.), MeCN (0.05 M), Blue LEDs (30 W), rt, for 16 h, under N_2. Yield of isolated product.

Light source[a]

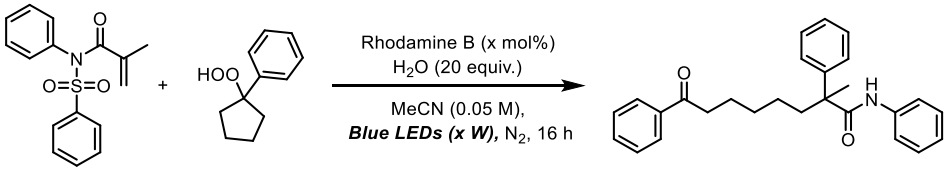


1a 2a 3aa

Entry	Light source	Yield (**3aa**, %)[a]
1	**Blue LEDs (30 W, 460-470 nm)**	**67**
2	Purple LEDs (30 W, 390 nm)	58
3	White LEDs (15 W, 390-780 nm)	trace

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Rhodamine B (5 mol%), H_2O (20 equiv.), MeCN (0.05 M), Blue LEDs (x W), rt, for 16 h, under N_2. Yield of isolated product.

Wattage of Blue LEDs[a]

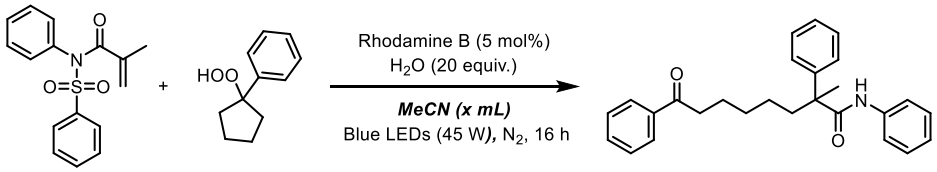


1a 2a 3aa

Entry	Blue LEDs (x W)	Yield (**3aa**, %)[a]
1	10	53
2	20	58
3	30	67
4	**45**	**75**
5	50	74

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Rhodamine B (5 mol%), H_2O (20 equiv.), MeCN (0.05 M), Blue LEDs (x W), rt, for 16 h, under N_2. Yield of isolated product.

Concentration[a]

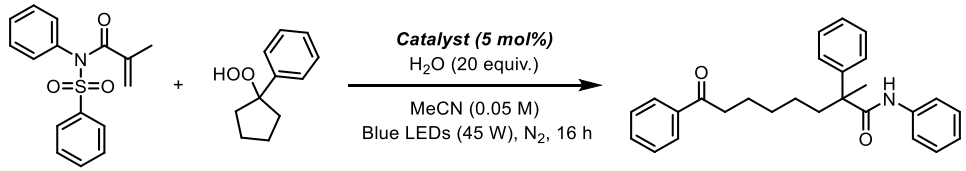


1a 2a 3aa

Entry	Concentration (x M)	Yield (**3aa**, %)[a]
1	0.20	51
2	0.10	58
3	**0.05**	**75**
4	0.025	60

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Rhodamine B (5 mol%), H_2O (20 equiv.), MeCN (x M), Blue LEDs (45 W), rt, for 16 h, under N_2. Yield of isolated product.

Others[a]

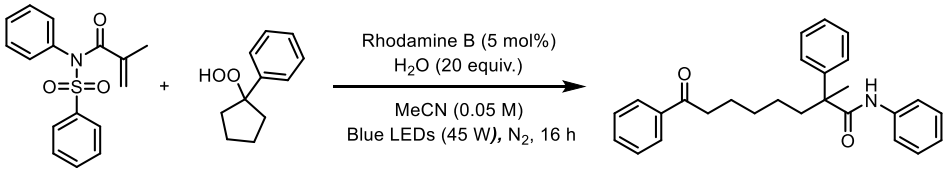


1a 2a 3aa

Entry	Catalyst	Yield (**3aa**, %)[a]
1	**Rhodamine B**	**75**
2	4CzIPN	61
3	Eosin Y	53
4	$Fe(OTf)_2$	46[b]
5	CuOTf	54[b]

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Catalyst (5 mol%), H_2O (20 equiv.), MeCN (0.05 M), Blue LEDs (45 W), rt, for 16 h, under N_2. Yield of isolated product. [b]80 °C, without irradiation.

Control experiments[a]



1a **2a** **3aa**

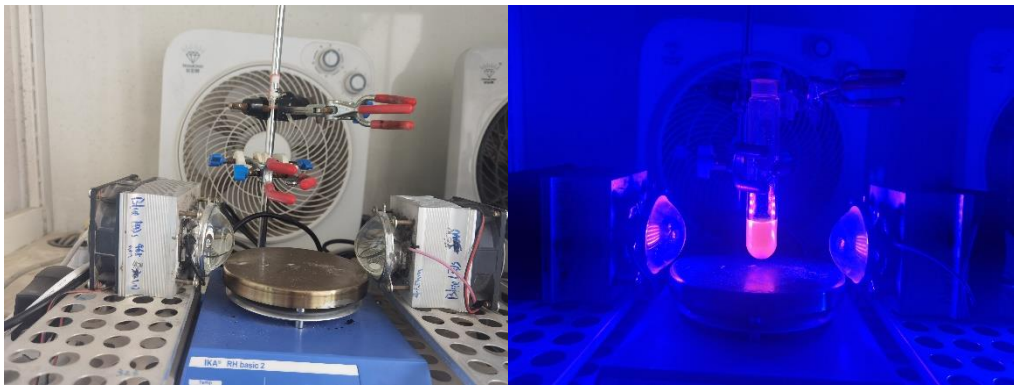
Entry	Deviation from standard condition	Yield (**3aa**, %)[a]
1	None	75
2	Without Water	56
3	Without PC	trace
4	Without Light	N.R
5	Dry MeCN	38
6	molecular sieves 4Å (30 mg)	49
7	Air instead of N_2	N.D
8	O_2 instead of N_2	N.D

[a]Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), Rhodamine B (5 mol%), H_2O (20 equiv.), MeCN (0.05 M), Blue LEDs (45 W), rt, for 16 h, under N_2. Yield of isolated product.

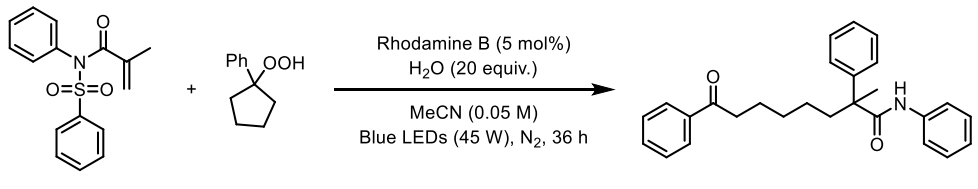
4. Representative Procedure for the Reaction of Acrylamides and Cycloalkyl Hydroperoxides.

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with *N*-aryl-*N*-(arylsulfonyl)acrylamides **1** (0.20 mmol, 1.0 equiv.), Rhodamine B (0.01mmol, 0.05 equiv.). Then, the tube were evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cycloalkyl hydroperoxides **2** (0.40 mmol, 2.0 equiv.) and H_2O (4.0 mmol, 20 equiv.) in MeCN (0.05 M) were added by a syringe. The reaction mixture was stirred under the irradiation of a 45 W Blue LED (λ = 460−470 nm) for a 16 h. After that, the resulting mixture were quenched with H_2O and extracted with EtOAc (3 x 10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and concentrated in vacuo. Purification of the crude product by flash chromatography on silica gel (petroleum ether/EtOAc = 15:1 to 10:1) furnishes the desired products **3**.

The Visible-Light Photoredox Catalysis Experimental Setup (photographed by author Li-Na Guo)



5. Large-Scale Synthesis of 3aa

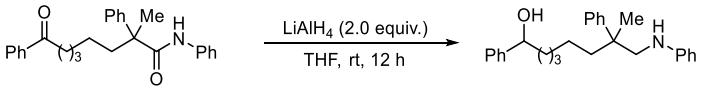


1a 2.0 mmol + **2a** 4.0 mmol → **3aa** 64%, 511.0 mg

A 100 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with *N*-phenyl-*N*-(phenysulfonyl)acrylamide **1a** (2.0 mmol, 1.0 equiv.), Rhodamine B (0.1 mmol, 0.05 equiv.). Then, the tube was evacuated and backfilled with nitrogen (three times). Then, a solution of cyclopentyl hydroperoxide **2a** (4.0 mmol, 2.0 equiv.) and H_2O (40 mmol, 20 equiv.) in MeCN (40 mL) were added by a syringe. The reaction mixture was stirred under the irradiation of a 45 W blue LED (λ = 460−470 nm; distance app. 1.0 cm from the bulb) for 36 h. After that, the resulting mixture was quenched with H_2O and extracted with EtOAc (3 × 20 mL). The combined organic phase was washed with brine (30 mL), dried over Na_2SO_4, and concentrated in vacuo. Purification of the crude product by flash chromatography on silica gel (petroleum ether/EtOAc: 15:1 to 10:1) furnishes the desired product **3aa** (64%, 511.0 mg).

6. Derivatizations of 3aa

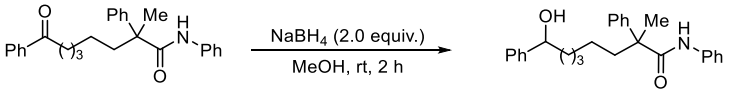
6.1 Reduction of 3aa with $LiAlH_4$



3aa **5aa**

An oven-dried 10 mL Schlenk tube equipped with a stirring bar was charged with **3aa** (79.8 mg, 0.2 mmol, 1.0 equiv.) and then the tube was evacuated and backfilled with nitrogen for three times at 0 ^{o}C. Then, THF (2.0 mL) was added by syringe under nitrogen. Subsequently, $LiAlH_4$ was added in batches under nitrogen. The reaction mixture was stirred under 25 ^{o}C for 12 h. After quenching with saturated aqueous NH_4Cl (5.0 mL), the solution was extracted with DCM (3 × 10 mL). The combined organic layer was washed with saturated brine, respectively. The combined organic layer was dried over anhydrous Na_2SO_4 and the solvent was removed under reduced pressure. Followed by work-up procedure, and flash column chromatography (petroleum ether/ EtOAc = 20:1) afforded product **5aa** as colorless oil.

6.2 Reduction of 3aa with $NaBH_4$

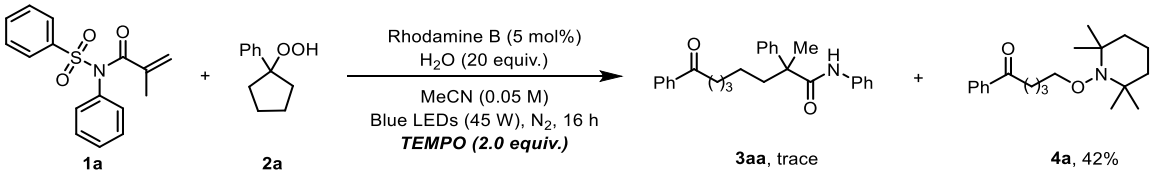


3aa **6aa**

To a solution of compound **3aa** (79.8 mg, 0.2 mmol, 1.0 equiv.) in MeOH (2.0 mL) was added $NaBH_4$ (9.0 mg, 0.40 mmol, 2.0 equiv.) at 25 ^{o}C. Then, the reaction was stirred at 25 ^{o}C for 2 h. After quenching with saturated aqueous NH_4Cl (5.0 mL), the solution was extracted with DCM (3 × 10 mL). The combined organic layer was washed with saturated brine, respectively. The combined organic layer was dried over anhydrous Na_2SO_4 and the solvent was removed under reduced pressure. Followed by work-up procedure, and flash column chromatography (petroleum ether/ EtOAc = 20:1) afforded product **6aa** as colorless oil.

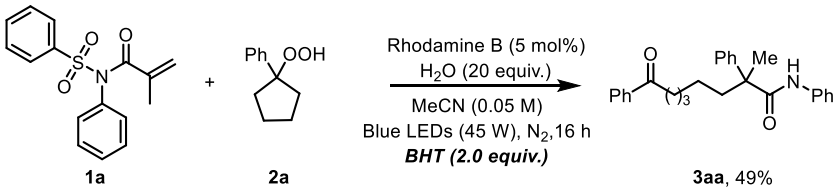
7. Mechanistic Investigation

7.1 TEMPO-Trapping Experiment



A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with *N*-phenyl-*N*-(phenysulfonyl)acrylamide **1a** (0.2 mmol, 1.0 equiv.), Rhodamine B (0.01mmol, 0.05 equiv.), TEMPO (0.4 mmol, 2.0 equiv.). Then, the tube was evacuated and backfilled with nitrogen (three times). Then, a solution of cyclopentyl hydroperoxide **2a** (0.4 mmol, 2.0 equiv.) and H_2O (4.0 mmol, 20 equiv.) in MeCN (0.05 M) were added by a syringe. The reaction mixture was stirred under the irradiation of 45 W Blue LEDs (λ = 460−470 nm; distance app. 1.0 cm from the bulb) for 16 h. After that, the resulting mixture was quenched with H_2O and extracted with EtOAc (3 × 10 mL). The combined organic phase was washed with brine (30 mL), dried over Na_2SO_4. After that, the residue was concentrated and purified by flash column chromatography on silica gel to afford the corresponding alkyl-TEMPO adduct **4a** in 42% yield and no product **3aa** was detected. This result indicated that a radical pathway might be involved in this transformation.

7.2 BHT-Inhibiting Experiment



A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with *N*-phenyl-*N*-(phenysulfonyl)acrylamide **1a** (0.2 mmol, 1.0 equiv.), Rhodamine B (0.01mmol, 0.05 equiv.), BHT (0.4 mmol, 2.0 equiv.). Then, the tube was evacuated and backfilled with nitrogen (three times). Then, a solution of cyclopentyl hydroperoxide **2a** (0.4 mmol, 2.0 equiv.) and H_2O (4.0 mmol, 20 equiv.) in MeCN (0.05 M) were added by a syringe. The reaction mixture was stirred under the irradiation of 45 W Blue LEDs (λ = 460−470 nm; distance app. 1.0 cm from the bulb) for 16 h. After that, the resulting mixture was quenched with H_2O and extracted with EtOAc (3 × 10 mL). The combined organic phase was washed with brine (30 mL), dried over Na_2SO_4. After that, the residue was concentrated and purified by flash column chromatography on silica gel to afford the product **3aa** with yield of 49%. This result indicated that a radical pathway might be involved in this transformation.

7.3 Stern-Volmer Fluorescence Quenching Experiments

To a solution of Rhodamine B in anhydrous, N_2-saturated MeCN (5×10^{-4} mol/L) in a quartz cuvette, different amounts of *N*-phenyl-*N*-(phenysulfonyl)acrylamide **1a** and cyclopentyl hydroperoxide **2a** were added, respectively, and the resulting changes in fluorescence intensity

(concentration of **1a** and **2a**: 5×10^{-2} mol/L, 1×10^{-1} mol/L, 1.5×10^{-1} mol/L, 2×10^{-1} mol/L were collected. The emission intensity at 606 nm was collected with excited wavelength of 450 nm. The results are shown in **Figure S1** and **S2**

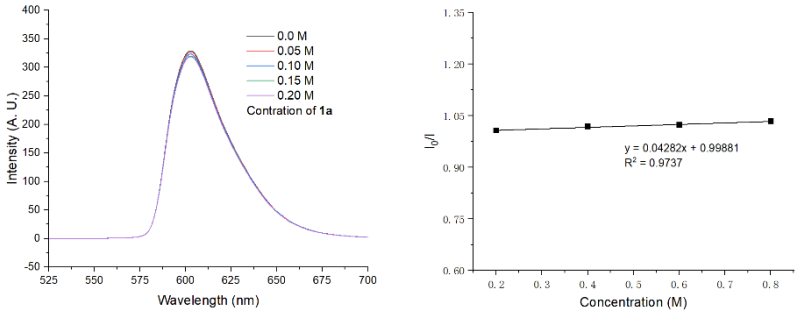


Figure S1. (a) The fluorescence emission spectra of Rhodamine B with different concentration of **1a** added. (b) The Stern–Volmer emission quenching studies of **1a**. $\mathbf{I_0}$ is the inherent fluorescence intensity of Rhodamine B. **I** is the fluorescence intensity of Rhodamine B in the presence of **1a**.

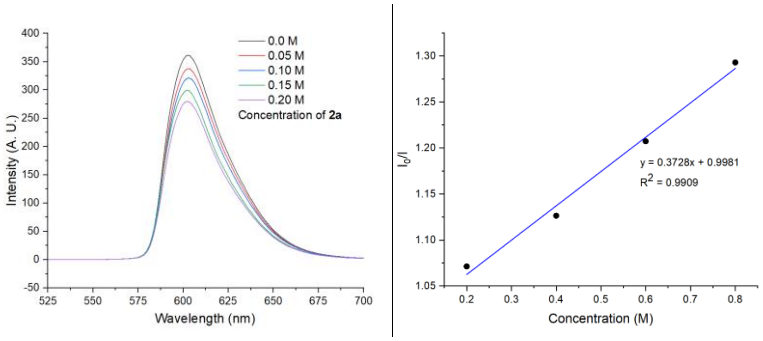


Figure S2. (a) The fluorescence emission spectra of Rhodamine B with different concentration of **2a** added. (b) The Stern–Volmer emission quenching studies of **2a**. $\mathbf{I_0}$ is the inherent fluorescence intensity of Rhodamine B. **I** is the fluorescence intensity of Rhodamine B in the presence of **2a**. Thus, Stern−Volmer fluorescence quenching studies indicated that the cyclopentyl peroxide **2a** interacts with the excited state of Rhodamine B rather than **1a**.

7.4 Light On-Off Experiments

To further examine the impact of light, we conducted experiments under alternating periods of irradiation and darkness. The yield of **3aa** was determined by crude ^{1}H NMR spectra using 1,3,5-trimethoxybenzene as an internal standard. The results are shown in **Figure S3**.

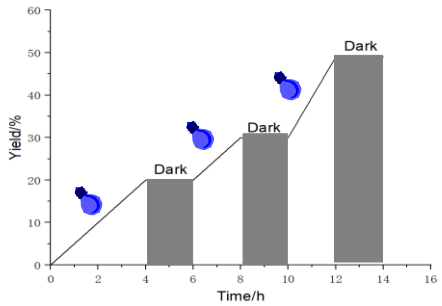


Figure S3. Light On-Off Experiments

The results of light on-off experiments indicated that the reaction proceeded only under the irradiation of light. Thus, the reaction maybe proceeds via a catalytic process rather than a radical chain process.

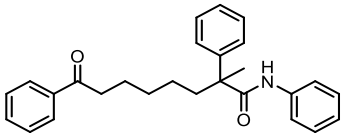
8. References

1. Z. Ni, X. Huang and Y. Pan, *Org. Lett*. 2016, **18**, 2612.

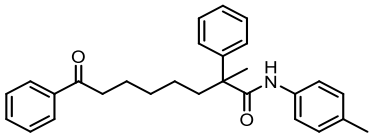
2. S. Liu, M. Bai, P.-F. Xu, Q.-X. Sun, X.-H. Duan and L.-N. Guo, *Chem. Commun*. 2021, **57**, 8652.

3. S. Liu, P. Ma, L. Zhang, S. Shen, H.-J. Miao, L. Liu, K. N. Houk, X.-H. Duan and L.-N. Guo, *Chem. Sci*., 2023, **14**, 5220.

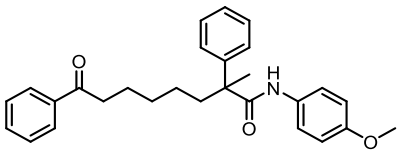
9. Characterization of Products



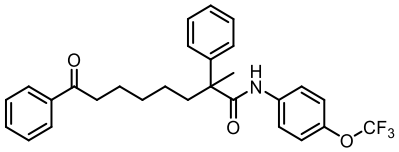
2-Methyl-8-oxo-*N*,2,8-triphenyloctanamide (3aa): colorless oil (75%, 59.8 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:15); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.91 (m, 2H), 7.57-7.53 (m, 1H), 7.47-7.43 (m, 2H), 7.40-7.39 (m, 4H), 7.37 -7.34 (m, 2H), 7.33-7.30 (m, 1H), 7.28-7.24 (m, 2H), 7.07-7.04 (m, 1H), 6.80 (s, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.15-2.02 (m, 2H), 1.75-1.68 (m, 2H), 1.63 (s, 3H), 1.45-1.22 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.2, 143.5, 137.9, 137.0, 132.9, 128.92, 128.85, 128.5, 128.0, 127.3, 126.9, 124.1, 119.7, 51.5, 38.7, 38.4, 29.8, 24.2, 24.1, 23.9 ppm; HRMS (ESI) calcd for $C_{27}H_{30}NO_2$ $[M+H]^+$ 400.2271, found 400.2280.



2-Methyl-8-oxo-2,8-diphenyl-N-(p-tolyl)octanamide (3ba): colorless oil (72%, 59.5 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.91 (m, 2H), 7.56-7.53 (m, 1H), 7.46-7.38 (m, 6H), 7.32-7.29 (m, 1H), 7.27-7.23 (m, 2H), 7.06 (d, *J* = 8.4 Hz, 2H), 6.77 (s, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.27 (s, 3H), 2.13-2.04 (m, 2H), 1.75-1.69 (m, 2H), 1.62 (s, 3H), 1.44-1.16 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.0, 143.7, 136.9, 135.3, 133.7, 132.9, 129.3, 128.9, 128.5, 128.0, 127.2, 126.9, 119.7, 51.4, 38.7, 38.4, 29.7, 24.2, 24.0, 23.9, 20.8 ppm; HRMS (ESI) calcd for $C_{28}H_{32}NO_2$ $[M+H]^+$ 414.2428, found 414.2425.

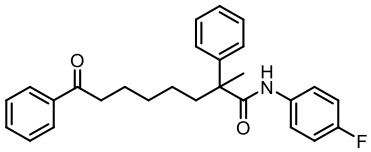


***N*-(4-Methoxyphenyl)-2-methyl-8-oxo-2,8-diphenyloctanamide (3ca):** colorless oil (40%, 34.3 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.91 (m, 2H), 7.56-7.52 (m, 1H), 7.46-7.42 (m, 2H), 7.39-7.36 (m, 4H), 7.33-7.29 (m, 1H), 7.26-7.24 (m, 2H), 6.80-6.78 (m, 2H), 6.73 (s, 1H), 3.75 (s, 3H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.13-2.02 (m, 2H), 1.73-1.67 (m, 2H), 1.62 (s, 3H), 1.42-1.17 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.1, 156.3, 143.7, 137.0, 132.9, 131.0, 128.9, 128.5, 128.0, 127.2, 126.9, 121.6, 114.0, 55.4, 51.3, 38.8, 38.4, 29.7, 24.2, 24.1, 23.9 ppm; HRMS (ESI) calcd for $C_{28}H_{32}NO_3$ $[M+H]^+$ 430.2377, found 430.2389.

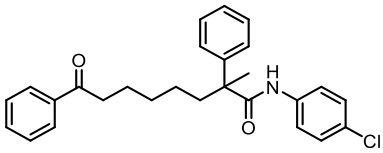


2-Methyl-8-oxo-2,8-diphenyl-*N*-(4-(trifluoromethoxy)phenyl)octanamide (3da): colorless oil (51%, 49.3 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.91 (m, 2H), 7.56-7.53 (m, 2H), 7.46-7.42 (m, 2H), 7.40-7.37 (m, 5H), 7.33-7.30 (m, 1H), 7.11 (d, *J* = 8.8 Hz, 2H), 6.86 (s, 1H), 2.92 (t, *J* = 6.8 Hz, 2H), 2.13-2.02 (m, 2H), 1.75-1.69 (m, 2H),1.63 (s, 3H), 1.44-1.36 (m, 2H), 1.30-1.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.3, 145.1, 143.3,

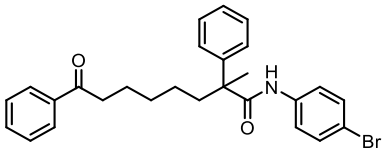
137.0, 136.6, 132.9, 129.0, 128.5, 128.0, 127.4, 126.8, 121.6, 120.4 (q, *J* = 255.5 Hz), 120.8, 51.5, 38.7, 38.4, 29.7, 24.2, 24.0, 23.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -59.13 ppm; HRMS (ESI) calcd for $C_{28}H_{29}F_3NO_3$ $[M+H]^+$ 484.2094, found 484.2093.



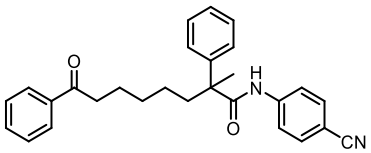
***N*-(4-Fluorophenyl)-2-methyl-8-oxo-2,8-diphenyloctanamide (3ea)**: colorless oil (64%, 53.4 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.91 (m, 2H), 7.56-7.53 (m, 1H), 7.46-7.42 (m, 2H), 7.40 -7.38 (m, 3H), 7.33-7.28 (m, 3H), 6.96-7.92 (m, 2H), 6.79 (s, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 2.13-2.02 (m, 2H), 1.75-1.69 (m, 2H), 1.63 (s, 3H), 1.42-1.36 (m, 2H), 1.29-1.11 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.2, 159.2 (d, *J* = 241.9 Hz), 143.5, 137.0, 133.9 (d, *J* = 3.0 Hz), 132.9, 128.9, 128.5, 128.0, 127.3, 126.8, 121.6 (d, *J* = 7.7 Hz), 115.4 (d, *J* = 22.3 Hz), 51.4, 38.7, 38.4, 29.7, 24.2, 24.0, 23.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -119.78 ppm; HRMS (ESI) calcd for $C_{27}H_{29}FNO_2$ $[M+H]^+$ 418.2177, found 418.2185



***N*-(4-Chlorophenyl)-2-methyl-8-oxo-2,8-diphenyloctanamide (3fa):** colorless oil (67%, 58.0); R_f = 0.2 (EtOAc/petroleum ether = 1:15); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.90 (m, 2H), 7.57-7.53 (m, 1H), 7.46-7.42 (m, 2H), 7.40-7.36 (m, 3H), 7.32 -7.29 (m, 3H), 7.22-7.19 (m, 2H), 6.80 (s, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 2.14-2.00 (m, 2H), 1.74-1.67 (m, 2H), 1.62 (s, 3H), 1.40-1.12 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.2, 143.3, 137.0, 136.5, 132.9, 129.02, 128.99, 128.8, 128.5, 128.0, 127.4, 126.8, 120.9, 51.5, 38.7, 38.3, 29.7, 24.2, 24.0, 23.8 ppm; HRMS (ESI) calcd for $C_{27}H_{29}Cl^{35}NO_2$ $[M+H]^+$ 434.1881, found 434.1895; HRMS (ESI) calcd for $C_{27}H_{29}Cl^{37}NO_2$ $[M+H]^+$ 436.1881, found 436.1877.

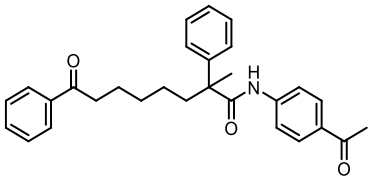


***N*-(4-Bromophenyl)-2-methyl-8-oxo-2,8-diphenyloctanamide (3ga):** colorless oil (56%, 53.4 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:15); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.90 (m, 2H), 7.56-7.53 (m, 1H), 7.46-7.42 (m, 2H), 7.39 -7.34 (m, 6H), 7.32-7.29 (m, 1H), 2.27-7.24 (m, 2H), 6.80 (s, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 2.12-2.00 (m, 2H), 1.74-1.67 (m, 2H), 1.62 (s, 3H), 1.43-1.36 (m, 2H), 1.28-1.13 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.2, 143.3, 137.0, 132.9, 131.8, 129.0, 128.6, 128.5, 128.0, 127.4, 126.8, 121.2, 116.6, 51.5, 38.6, 38.3, 29.7, 24.2, 24.0, 23.8 ppm; HRMS (ESI) calcd for $C_{27}H_{29}Br^{79}NO_2$ $[M+H]^+$ 478.1376, found 478.1389; HRMS (ESI) calcd for $C_{27}H_{29}Br^{81}NO_2$ $[M+H]^+$ 480.1376, found 480.1374.

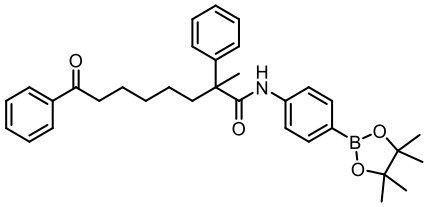


***N*-(4-Cyanophenyl)-2-methyl-8-oxo-2,8-diphenyloctanamide** (**3ha**): colorless oil (65%, 55.1 mg); R_f

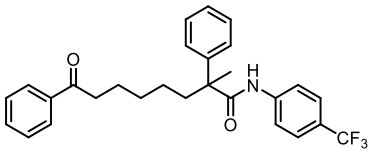
= 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92-7.89 (m, 2H), 7.56-7.53 (m, 2H), 7.51-7.50 (m, 3H), 7.47-7.44 (m, 2H), 7.42-7.35 (m, 4H), 7.33-7.29 (m, 1H), 7.05 (s, 1H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.12-2.03 (m, 2H), 1.73-1.65 (m, 2H), 1.62 (s, 3H), 1.41-1.33 (m, 2H), 1.28-1.14 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 175.6, 142.8, 141.9, 136.9, 133.1, 132.9, 129.1, 128.5, 127.9, 127.6, 126.7, 119.4, 118.8, 106.8, 51.7, 38.5, 38.3, 29.6, 24.1, 23.9, 23.6 ppm; HRMS (ESI) calcd for $C_{28}H_{29}N_2O_2$ $[M+H]^+$ 425.2224, found 425.2224.



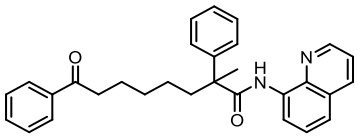
***N*-(4-Acetylphenyl)-2-methyl-8-oxo-2,8-diphenyloctanamide (3ia):** colorless oil (57%, 50.3 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.91-7.89 (m, 2H), 7.85 (d, *J* = 8.8 Hz, 2H), 7.55-7.52 (m, 1H), 7.48-7.43 (m, 4H), 7.41-7.37 (m, 4H), 7.32-7.29 (m, 1H), 7.09 (s, 1H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.52 (s, 3H), 2.13-2.04 (m, 2H), 1.73-1.68 (m, 2H), 1.63 (s, 3H), 1.41-1.33 (m, 2H), 1.27-1.15 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 196.9, 175.5, 143.0, 142.3, 136.9, 132.9, 132.6, 129.5, 129.0, 128.5, 127.9, 127.4, 126.7, 118.7, 51.7, 38.6, 38.3, 29.6, 26.3, 24.1, 23.9, 23.7 ppm; HRMS (ESI) calcd for $C_{29}H_{32}NO_3$ $[M+H]^+$ 442.2377, found 442.2380.



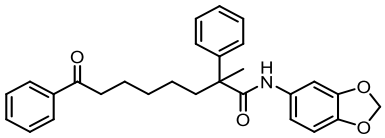
2-Methyl-8-oxo-2,8-diphenyl-*N*-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)octan-amide (3ja): colorless oil (36%, 37.8 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92-7.90 (m, 2H), 7.70 (d, *J* = 8.0 Hz, 2H), 7.56-7.52 (m, 1H), 7.46-7.42 (m, 2H), 7.39-7.36 (m, 6H), 7.32-7.29 (m, 1H), 6.83 (s, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.14-2.00 (m, 2H), 1.74-1.69 (m, 2H), 1.62 (s, 3H), 1.40-1.36 (m, 2H), 1.32 (s, 12H), 1.25-1.11 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.2, 143.4, 140.6, 137.0, 135.7, 132.9, 129.0, 128.54, 128.52, 128.0, 127.4, 126.9, 118.4, 83.7, 51.6, 38.7, 38.4, 29.7, 24.8, 24.2, 24.1, 23.9 ppm; HRMS (ESI) calcd for $C_{33}H_{41}BNO_4$ $[M+H]^+$ 526.3123, found 526.3129.



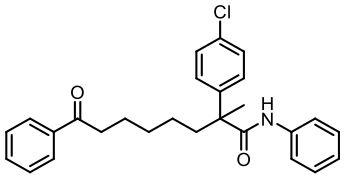
2-Methyl-8-oxo-2,8-diphenyl-*N*-(4-(trifluoromethyl)phenyl)octanamide (3ka): colorless oil (65%, 60.7 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.90 (m, 2H), 7.57-7.53 (m, 1H), 7.52-7.48 (m, 4H), 7.46-7.42 (m, 2H), 7.40-7.37 (m, 4H), 7.34-7.30 (m, 1H), 6.94 (s, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.11-2.05 (m, 2H), 1.75-1.69 (m, 2H), 1.64 (s, 3H), 1.43-1.36 (m, 2H), 1.34-1.29 (m, 1H), 1.22-1.13 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.5, 143.1, 140.9, 136.9, 132.9, 129.1, 128.5, 128.0, 127.5, 126.8, 126.1 (q, *J* = 3.7 Hz), 125.8 (q, *J* = 32.8 Hz), 124.0 (q, *J* = 269.4 Hz), 51.7, 38.6, 38.3, 29.7, 24.2, 24.0, 23.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -62.97 ppm; HRMS (ESI) calcd for $C_{28}H_{29}F_3NO_2$ $[M+H]^+$ 468.2145, found 468.2153.



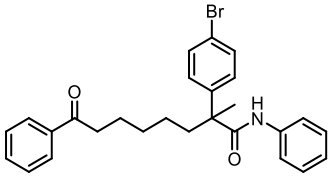
2-Methyl-8-oxo-2,8-diphenyl-*N*-(quinolin-8-yl)octanamide (3la): colorless oil (44%, 39.6 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.86 (s, 1H), 8.77-8.75 (m, 1H), 8.61-8.59 (m, 1H), 8.09-8.06 (m, 1H), 7.91-7.89 (m, 2H), 7.55-7.49 (m, 4H), 7.45-7.33 (m, 6H), 7.30-7.28 (m, 1H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.30-2.24 (m, 1H), 2.18-2.10 (m, 1H), 1.76 (s, 3H), 1.75-1.69 (m, 2H), 1.48-1.39 (m, 2H), 1.33-1.25 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.3, 148.1, 144.0, 138.6, 137.0, 136.1, 134.6, 132.8, 128.7, 128.5, 128.0, 127.8, 127.3, 126.9, 126.7, 121.4, 121.2, 116.0, 51.9, 38.9, 38.4, 29.8, 24.4, 24.1, 23.6 ppm; HRMS (ESI) calcd for $C_{30}H_{31}N_2O_2$ $[M+H]^+$ 451.2380, found 451.2382.



***N*-(Benzo[d][1,3]dioxol-5-yl)-2-methyl-8-oxo-2,8-diphenyloctanamide (3ma):** colorless oil (39%, 34.6 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.91 (m, 2H), 7.56-7.53 (m, 1H), 7.48-7.42 (m, 3H), 7.39-7.37 (m, 3H), 7.32-7.28 (m, 1H), 7.11 (d, *J* = 2.4 Hz, 1H), 6.71 (s, 1H), 6.67-6.64 (m, 1H), 6.58-6.55 (m, 1H), 5.9 (s, 2H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.12-2.04 (m, 2H), 1.75-1.69 (m, 2H),1.61 (s, 3H), 1.44-1.36 (m, 2H), 1.29-1.12 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 175.1, 147.7, 144.1, 143.6, 137.0, 132.9, 132.1, 128.9, 128.5, 128.0, 127.3, 126.9, 112.9, 107.9, 102.7, 101.2, 51.3, 38.7, 38.4, 29.7, 24.2, 24.1, 23.9 ppm; HRMS (ESI) calcd for $C_{28}H_{30}NO_4$ $[M+H]^+$ 444.2169, found 444.2171.

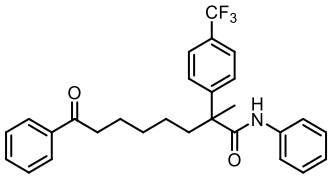


2-(4-Chlorophenyl)-2-methyl-8-oxo-*N*,8-diphenyloctanamide (3na): colorless oil (60%, 51.9 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.92 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.39-7.32 (m, 6H), 7.30 -7.27 (m, 2H), 7.10-7.06 (m, 1H), 6.81 (s, 1H), 2.93 (t, *J* = 7.2 Hz, 2H), 2.13-1.98 (m, 2H), 1.76-1.69 (m, 2H), 1.62 (s, 3H), 1.45-1.37 (m, 2H), 1.30-1.17 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 174.5, 142.2, 137.7, 136.9, 133.2, 132.9, 129.0, 128.9, 128.5, 128.2, 128.0, 124.3, 119.7, 51.1, 38.8, 38.3, 29.7, 24.2, 24.0, 23.8 ppm; HRMS (ESI) calcd for $C_{27}H_{29}Cl^{35}NO_2$ $[M+H]^+$ 434.1881, found 434.1880, HRMS (ESI) calcd for $C_{27}H_{29}Cl^{37}NO_2$ $[M+H]^+$ 436.1881, found 436.1861.

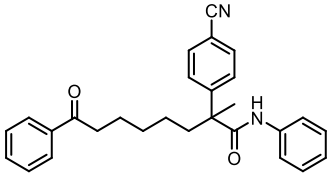


2-(4-Bromophenyl)-2-methyl-8-oxo-*N*,8-diphenyloctanamide (3oa): colorless oil (62%, 59.1 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:15); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.92 (m, 2H), 7.56 (t, *J* =

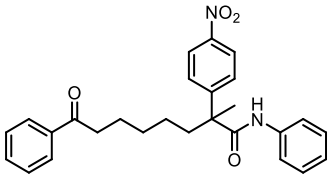
7.6 Hz, 1H), 7.53-7.51 (m, 2H), 7.48-7.44 (m, 2H), 7.39 -7.36 (m, 2H), 7.30-7.27 (m, 4H), 7.10-7.06 (m, 1H), 6.77 (s, 1H), 2.93 (t, *J* = 7.6 Hz, 2H), 2.12-1.98 (m, 2H), 1.76-1.69 (m, 2H), 1.61 (s, 3H), 1.43-1.39 (m, 2H), 1.35-1.29 (m, 1H), 1.23-1.15 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 174.5, 142.9, 137.8, 137.1, 133.0, 132.1, 129.1, 128.73, 128.67, 128.1, 124.4, 121.5, 119.9, 51.3, 38.9, 38.5, 29.8, 24.3, 24.1, 23.9 ppm; HRMS (ESI) calcd for $C_{27}H_{29}Br^{79}NO_2$ $[M+H]^+$ 478.1376, found 478.1386; HRMS (ESI) calcd for $C_{27}H_{29}Br^{81}NO_2$ $[M+H]^+$ 480.1376, found 480.1370.



2-Methyl-8-oxo-*N*,8-diphenyl-2-(4-(trifluoromethyl)phenyl)octanamide (3pa): colorless oil (58%, 54.1 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:7); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92-7.90 (m, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.56-7.50 (m, 3H), 7.45-7.41 (m, 2H), 7.38-7.36 (m, 2H), 7.28-7.24 (m, 2H), 7.09-7.05 (m, 1H), 6.80 (s, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 2.14-1.99 (m, 2H), 1.73-1.67 (m, 2H), 1.64 (s, 3H), 1.43-1.36 (m, 2H), 1.28-1.17 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 173.9, 148.0, 137.6, 136.9, 132.9, 129.3 (q, *J* = 32.4 Hz), 128.9, 128.5, 127.9, 125.7 (q, *J* = 3.9 Hz), 123.9 (q, *J* = 270.4 Hz), 124.4, 119.9, 51.5, 38.8, 38.3, 29.6, 24.2, 23.9, 23.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -64.13 ppm; HRMS (ESI) calcd for $C_{28}H_{29}F_3NO_2$ $[M+H]^+$ 468.2145, found 468.2154.

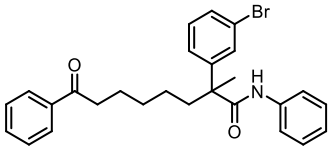


2-(4-Cyanophenyl)-2-methyl-8-oxo-*N*,8-diphenyloctanamide (3qa): colorless oil (50%, 42.4 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.90 (m, 2H), 7.66-7.64 (m, 2H), 7.57-7.53 (m, 1H), 7.52-7.49 (m, 2H), 7.47-7.43 (m, 2H), 7.40-7.38 (m, 2H), 7.30-7.26 (m, 2H), 7.11-7.07 (m, 1H), 6.88 (s, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.15-1.97 (m, 2H), 1.73-1.67 (m, 2H), 1.64 (s, 3H), 1.44-1.37 (m, 2H), 1.32-1.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.2, 173.3, 149.5, 137.5, 136.9, 133.0, 132.5, 128.9, 128.5, 128.0, 127.5, 124.5, 119.9, 118.4, 111.1, 51.7, 38.8, 38.2, 29.6, 24.3, 23.9, 23.5 ppm; HRMS (ESI) calcd for $C_{28}H_{29}N_2O_2$ $[M+H]^+$ 425.2224, found 425.2225.

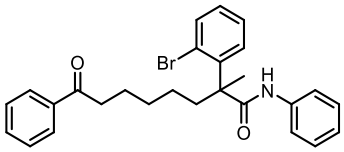


2-Methyl-2-(4-nitrophenyl)-8-oxo-*N*,8-diphenyloctanamide (3ra): colorless oil (43%, 38.2 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:8); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.23-8.20 (m, 2H), 7.93-7.90 (m, 2H), 7.58-7.53 (m, 3H), 7.46-7.42 (m, 2H), 7.40 -7.38 (m, 2H), 7.30-7.26 (m, 2H), 7.11-7.08 (m, 1H), 6.84 (s, 1H), 2.93 (t, *J* = 7.2 Hz, 2H), 2.18-2.02 (m, 2H), 1.76-1.70 (m, 2H), 1.68 (s, 3H), 1.44-1.38 (m, 2H), 1.27-1.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.2, 173.1, 151.5, 146.9, 137.4, 136.9, 133.0, 129.0, 128.6, 128.0, 127.6, 124.7, 124.0, 119.9, 51.8, 39.0, 38.2, 29.6, 24.3, 23.9, 23.6 ppm;

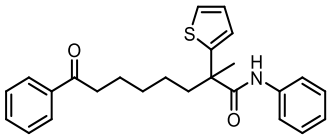
HRMS (ESI) calcd for $C_{27}H_{29}N_2O_4$ $[M+H]^+$ 445.2122, found 445.2121.



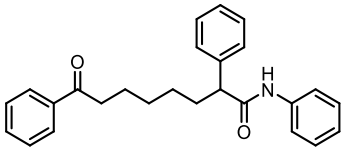
2-(3-Bromophenyl)-2-methyl-8-oxo-*N*,8-diphenyloctanamide (3sa): colorless oil (43%, 41.0 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.60-7.56 (m, 2H), 7.50-7.45 (m, 3H), 7.43-7.41 (m, 2H), 7.36 -7.28 (m, 4H), 7.13-7.09 (m, 1H), 6.88 (s, 1H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.15-2.00 (m, 2H), 1.79-1.72 (m, 2H), 1.65 (s, 3H), 1.48-1.41 (m, 2H), 1.35-1.19 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 200.3, 174.1, 146.3, 137.7, 136.9, 132.9, 130.42, 130.40, 129.7, 128.9, 128.5, 128.0, 125.6, 124.3, 123.1, 119.9, 51.4, 38.7, 38.3, 29.6, 24.2, 24.0, 23.7 ppm; HRMS (ESI) calcd for $C_{27}H_{29}Br^{79}NO_2$ $[M+H]^+$ 478.1376, found 478.1379, HRMS (ESI) calcd for $C_{27}H_{29}Br^{81}NO_2$ $[M+H]^+$ 480.1376, found 480.1366.



2-(2-Bromophenyl)-2-methyl-8-oxo-*N*,8-diphenyloctanamide (3ta): colorless oil (31%, 29.5 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.90 (m, 2H), 7.65-7.62 (m, 1H), 7.56-7.49 (m, 2H), 7.46-7.36 (m, 5H), 7.30-7.26 (m, 2H), 7.21-7.18 (m, 1H), 7.09-7.06 (m, 1H), 6.78 (s, 1H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.52-2.44 (m, 1H), 2.08-2.01 (m, 1H), 1.71-1.66 (m, 5H), 1.41-1.25 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 174.4, 141.6, 138.0, 136.9, 135.2, 132.9, 129.4, 129.1, 128.8, 128.5, 128.0, 127.7, 124.5, 124.2, 120.3, 52.5, 38.4, 36.1, 29.7, 24.5, 24.3, 24.1 ppm; HRMS (ESI) calcd for $C_{27}H_{29}Br^{79}NO_2$ $[M+H]^+$ 478.1376, found 478.1386; HRMS (ESI) calcd for $C_{27}H_{29}Br^{81}NO_2$ $[M+H]^+$ 480.1376, found 480.1368.

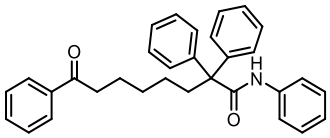


2-Methyl-8-oxo-*N*,8-diphenyl-2-(thiophen-2-yl)octanamide (3ua): colorless oil (39%, 31.6 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.92 (m, 2H), 7.57-7.53 (m, 1H), 7.47-7.43 (m, 2H), 7.39-7.36 (m, 2H), 7.33-7.31 (m, 1H), 7.29-7.25 (m, 2H), 7.21 (s, 1H), 7.09-7.04 (m, 3H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.25-2.17 (m, 1H), 2.08-2.00 (m, 1H), 1.78-1.71 (m, 5H), 1.46-1.26 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 173.8, 148.4, 137.7, 136.9, 132.9, 128.9, 128.5, 128.0, 127.2, 125.32, 125.29, 124.3, 119.7, 49.9, 40.1, 38.4, 29.6, 24.8, 24.3, 24.1 ppm; HRMS (ESI) calcd for $C_{25}H_{28}NO_2S$ $[M+H]^+$ 406.1835, found 406.1839.

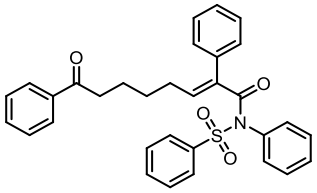


8-Oxo-*N*,2,8-triphenyloctanamide (3va): colorless oil (60%, 46.2 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.76-7.54 (m, 2H), 7.47-7.43 (m, 4H), 7.37-7.35 (m, 4H),7.31-7.29 (m, 1H), 7.25 (s, 1H), 7.15-7.12 (m, 1H), 7.08-7.05 (m, 1H), 3.50 (t, *J* = 7.6 Hz, 1H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.31-2.23 (m, 1H), 1.89-1.81 (m, 1H), 1.77-1.70 (m, 2H),

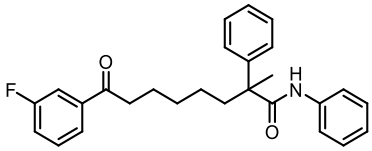
1.46-1.39 (m, 2H), 1.29-1.24 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 171.7, 139.6, 137.9, 136.9, 132.9, 129.0, 128.9, 128.5, 128.02, 127.96, 127.5, 124.2, 119.7, 54.1, 38.3, 33.0, 28.9, 27.4, 24.0 ppm; HRMS (ESI) calcd for $C_{26}H_{28}NO_2$ $[M+H]^+$ 386.2115, found 386.2126.



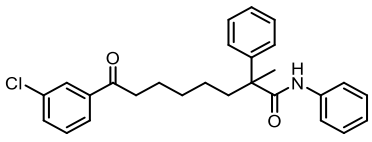
8-Oxo-*N*,2,2,8-tetraphenyloctanamide (3wa): colorless oil (46%, 42.4 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.91 (m, 2H), 7.57-7.52 (m, 1H), 7.46-7.42 (m, 2H), 7.40-7.35 (m, 10H), 7.32-7.24 (m, 4H), 7.16 (s, 1H), 7.09-7.05 (m, 1H), 2.89 (t, *J* = 7.2 Hz, 2H), 2.51-2.47 (m, 2H), 1.73-1.65 (m, 2H), 1.45-1.38 (m, 2H), 1.31-1.24 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 172.6, 143.0, 137.8, 137.1, 132.9, 128.94, 128.87, 128.6, 128.5, 128.1, 127.2, 124.3, 119.8, 61.7, 38.9, 38.5, 29.9, 25.3, 24.1 ppm; HRMS (ESI) calcd for $C_{32}H_{32}NO_2$ $[M+H]^+$ 462.2428, found 462.2424.



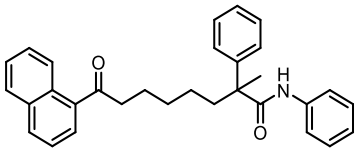
(*E*)-8-Oxo-*N*,2,8-triphenyl-*N*-(phenylsulfonyl)oct-2-enamide (3wa'): colorless oil (44%, 46.0 mg); R_f = 0.1 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.09-8.07 (m, 2H), 7.98-7.96 (m, 2H), 7.69-7.65 (m, 1H), 7.59-7.55 (m, 3H), 7.49-7.46 (m, 2H), 7.30-7.26 (m, 1H), 7.21-7.11 (m, 5H), 6.79-6.76 (m, 4H), 5.65 (t, *J* = 8.0 Hz, 1H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.14-2.09 (m, 2H), 1.73-1.66 (m, 2H), 1.50-1.43 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 168.8, 138.8, 137.6, 136.9, 136.0, 135.1, 134.5, 133.9, 133.0, 130.0, 129.5, 129.1, 128.7, 128.57, 128.55, 128.4, 128.0, 127.8, 125.6, 38.0, 30.0, 28.4, 23.7 ppm; HRMS (ESI) calcd for $C_{32}H_{29}NO_4SK$ $[M+K]^+$ 562.1449, found 562.1458.



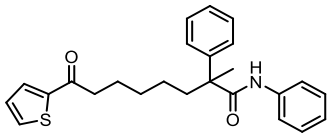
8-(3-Fluorophenyl)-2-methyl-8-oxo-*N*,2-diphenyloctanamide (3ab): colorless oil (68%, 56.7 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.71-7.68 (m, 1H), 7.62-7.58 (m, 1H), 7.45-7.41 (m, 1H), 7.40-7.39 (m, 4H), 7.36-7.34 (m, 2H), 7.33-7.29 (m, 1H), 7.27-7.23 (m, 3H), 7.07-7.03 (m, 1H), 6.81 (s, 1H), 2.89 (t, *J* = 7.2 Hz, 2H), 2.15-2.02 (m, 2H), 1.74-1.69 (m, 2H), 1.63 (s, 3H), 1.43-1.36 (m, 2H), 1.27-1.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.0, 175.2, 162.8 (d, *J* = 246.3 Hz), 143.5, 139.1 (d, *J* = 5.9 Hz), 137.9, 130.2 (d, *J* = 7.6 Hz), 128.93, 128.85, 127.3, 126.9, 124.1, 123.7 (d, *J* = 3.0 Hz), 119.9 (d, *J* = 21.4 Hz), 119.7, 114.7 (d, *J* = 22.0 Hz), 51.5, 38.7, 38.5, 29.7, 24.2, 23.88, 23.85; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -113.53 ppm; HRMS (ESI) calcd for $C_{27}H_{29}FNO_2$ $[M+H]^+$ 418.2177, found 418.2187.



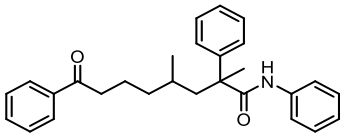
8-(3-Chlorophenyl)-2-methyl-8-oxo-*N*,2-diphenyloctanamide (3ac): colorless oil (42%, 36.4 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89-7.88 (m, 1H), 7.80-7.77 (m, 1H), 7.53-7.50 (m, 1H), 7.41-7.38 (m, 5H), 7.36-7.35 (m, 1H), 7.35-7.34 (m, 1H), 7.33-7.29 (m, 1H), 7.27-7.23 (m, 2H), 7.07-7.03 (m, 1H), 6.80 (s, 1H), 2.88 (t, *J* = 7.2 Hz, 2H), 2.11-2.04 (m, 2H), 1.72-1.67 (m, 2H), 1.63 (s, 3H), 1.40-1.33 (m, 2H), 1.29-1.22 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.0, 175.2, 143.5, 138.5, 137.9, 134.9, 132.8, 129.9, 128.94, 128.87, 128.1, 127.3, 126.9, 126.1, 124.1, 119.7, 51.5, 38.7, 38.5, 29.6, 24.2, 23.9, 23.8 ppm; HRMS (ESI) calcd for $C_{27}H_{29}Cl^{35}NO_2$ $[M+H]^+$ 434.1881, found 434.1888, HRMS (ESI) calcd for $C_{27}H_{29}Cl^{37}NO_2$ $[M+H]^+$ 436.1881, found 436.1879.



2-Methyl-8-(naphthalen-1-yl)-8-oxo-*N*,2-diphenyloctanamide (3ad): colorless oil (39%, 35.0 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (d, *J* = 8.4 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 7.6 Hz, 1H), 7.79 (d, *J* = 7.2 Hz, 1H), 7.59-7.46 (m, 3H), 7.40-7.38 (m, 4H), 7.36-7.34 (m, 2H), 7.32-7.29 (m, 1H), 7.27-7.23 (m, 2H), 7.07-7.03 (m, 1H), 6.78 (s, 1H), 3.00 (t, *J* = 7.2 Hz, 2H), 2.14-2.01 (m, 2H), 1.80-1.73 (m, 2H), 1.63 (s, 3H), 1.47-1.39 (m, 2H), 1.29-1.15 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 205.0, 175.2, 143.5, 137.9, 136.4, 133.9, 132.3, 130.1, 128.94, 128.87, 128.4, 127.8, 127.3, 127.1, 126.9, 126.4, 125.7, 124.4, 124.1, 119.7, 51.5, 42.1, 38.7, 29.8, 24.5, 24.2, 23.9 ppm; HRMS (ESI) calcd for $C_{31}H_{32}NO_2$ $[M+H]^+$ 450.2428, found 450.2418.

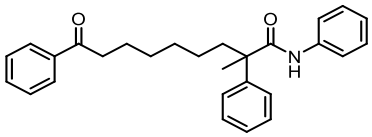


2-Methyl-8-oxo-*N*,2-diphenyl-8-(thiophen-2-yl)octanamide (3ae): colorless oil (42%, 34.0 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.66 (d, *J* = 3.6 Hz, 1H), 7.61 (d, *J* = 5.2 Hz, 1H), 7.40-7.39 (m, 3H), 7.37-7.34 (m, 2H), 7.32-7.29 (m, 1H), 7.28-7.24 (m, 3H), 7.12-7.09 (m, 1H), 7.07-7.04 (m, 1H), 6.79 (s, 1H), 2.84 (t, *J* = 7.2 Hz, 2H), 2.13-2.01 (m, 2H), 1.74-1.68 (m, 2H), 1.63 (s, 3H), 1.44-1.36 (m, 2H), 1.33-1.28 (m, 1H), 1.20-1.14 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.4, 175.2, 144.4, 143.5, 137.9, 133.4, 131.7, 128.94, 128.87, 128.0, 127.3, 126.9, 124.1, 119.6, 51.5, 39.2, 38.7, 29.7, 24.5, 24.2, 23.9 ppm; HRMS (ESI) calcd for $C_{25}H_{28}NO_2S$ $[M+H]^+$ 406.1835, found 406.1824.

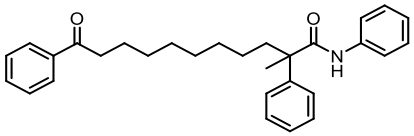


2,4-Dimethyl-8-oxo-*N*,2,8-triphenyloctanamide (3af): colorless oil (62%, 51.2 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10, d.r. = 1:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.91-7.87 (m, 2H), 7.57-7.52 (m, 1H), 7.47-7.40 (m, 4H), 7.38-7.34 (m, 4H), 7.30-7.21 (m, 3H), 7.06-7.01 (m, 1H), 6.82 (s, 1H), 2.88-2.71 (m, 2H), 2.24-1.93 (m, 2H), 1.72-1.61 (m, 5H), 1.56-1.50 (m, 1H), 1.30-1.32 (m, 2H), 0.93 (d, *J* = 6.8 Hz, 1.5H), 0.70 (d, *J* = 6.4 Hz, 1.5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.44, 200.38, 175.4, 144.1, 144.0, 138.0, 137.9, 137.0, 132.9, 128.91, 128.89, 128.87, 128.5, 128.0, 127.4, 127.1, 124.12, 124.09, 119.70, 119.67, 51.73, 51.66, 45.6, 38.7, 38.5, 38.42, 38.35, 29.1, 29.0, 24.34, 24.29,

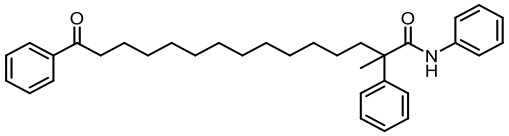
21.7, 21.6, 21.5 ppm; HRMS (ESI) calcd for $C_{28}H_{32}NO_2$ $[M+H]^+$ 414.2428, found 414.2440.



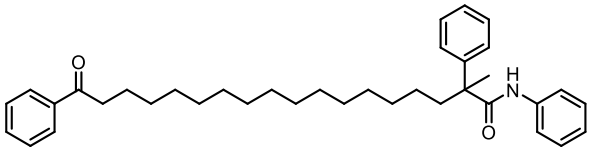
2-Methyl-9-oxo-*N*,2,9-triphenylnonanamide (3ag): colorless oil (23%, 19.0 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:15); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.92 (m, 2H), 7.57-7.53 (m, 1H), 7.47-7.43 (m, 2H), 7.40-7.39 (m, 4H), 7.37-7.34 (m, 2H), 7.32-7.29 (m, 1H), 7.27-7.24 (m, 2H), 7.07-7.03 (m, 1H), 6.80 (s, 1H), 2.93 (t, *J* = 7.2 Hz, 2H), 2.13-2.01 (m, 2H), 1.71-1.67 (m, 2H), 1.62 (s, 3H), 1.38-1.34 (m, 2H), 1.27-1.25 (m, 1H), 1.18-1.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 175.3, 143.6, 137.9, 137.0, 132.9, 128.92, 128.86, 128.5, 128.0, 127.3, 126.9, 124.1, 119.7, 51.5, 38.8, 38.5, 29.9, 29.1, 24.2, 23.9 ppm; HRMS (ESI) calcd for $C_{28}H_{32}NO_2$ $[M+H]^+$ 414.2428, found 414.2432.



2-Methyl-11-oxo-*N*,2,11-triphenylundecanamide (3ah): colorless oil (35%, 30.9 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.94 (m, 2H), 7.57-7.53 (m, 1H), 7.47-7.43 (m, 2H), 7.40-7.39 (m, 4H), 7.37-7.34 (m, 2H), 7.32-7.29 (m, 1H), 7.28-7.24 (m, 2H), 7.07-7.03 (m, 1H), 6.80 (s, 1H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.13-2.03 (m, 2H), 1.72-1.66 (m, 2H), 1.62 (s, 3H), 1.34-1.26 (m, 9H), 1.15-1.08 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 175.3, 143.7, 137.9, 137.0, 132.8, 128.89, 128.85, 128.5, 128.0, 127.2, 126.9, 124.1, 119.7, 51.5, 38.8, 38.6, 30.1, 29.38, 29.35, 29.3, 29.2, 24.3, 23.9 ppm; HRMS (ESI) calcd for $C_{30}H_{36}NO_2$ $[M+H]^+$ 442.2741, found 442.2745.

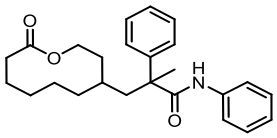


2-Methyl-15-oxo-*N*,2,15-triphenylpentadecanamide (3ai): colorless oil (78%, 77.5 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.57-7.53 (m, 1H), 7.47-7.44 (m, 2H), 7.42-7.35 (m, 6H), 7.32-7.30 (m, 1H), 7.28-7.24 (m, 2H), 7.07-7.03 (m, 1H), 6.83 (s, 1H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.14-2.03 (m, 2H), 1.74-1.69 (m, 2H), 1.63 (s, 3H), 1.35-1.12 (m, 18H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 175.2, 143.8, 137.9, 137.0, 132.8, 128.84, 128.79, 128.5, 128.0, 127.2, 126.8, 124.0, 119.6, 51.5, 38.8, 38.6, 30.1, 29.5, 29.40, 29.37, 29.3, 24.32, 24.30, 23.9 ppm; HRMS (ESI) calcd for $C_{34}H_{44}NO_2$ $[M+H]^+$ 498.3367, found 498.3368.

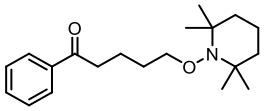


2-Methyl-18-oxo-*N*,2,18-triphenyloctadecanamide (3aj): colorless oil (55%, 59.3 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.57-7.53 (m, 1H), 7.48-7.44 (m, 2H), 7.41-7.38 (m, 4H), 7.37-7.35 (m, 2H), 7.33-7.30 (m, 1H), 7.28-7.24 (m, 2H), 7.07-7.04 (m, 1H), 6.81 (s, 1H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.13-2.01 (m, 2H), 1.75-1.63 (m, 2H), 1.63 (s, 3H), 1.35-1.23 (m, 24H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 175.3, 143.8, 138.0, 137.1, 132.8,

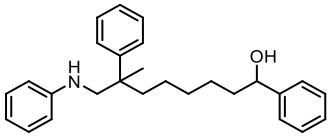
128.91, 128.87, 128.5, 128.1, 127.2, 126.9, 124.1, 119.7, 51.6, 38.9, 38.7, 30.2, 29.64, 29.61, 29.51, 29.48, 29.45, 29.39, 24.41, 24.37, 24.0 ppm; HRMS (ESI) calcd for $C_{37}H_{50}NO_2$ $[M+H]^+$ 540.3836, found 540.3851.



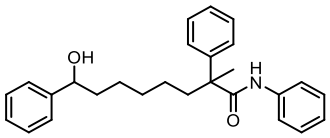
2-Methyl-3-(10-oxooxecan-4-yl)-*N*,2-diphenylpropanamide (3ak): colorless oil (71%, 55.8 mg,); R_f = 0.2 (EtOAc/petroleum ether = 1:10, d.r. = 1:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.40-7.38 (m, 4H), 7.34-7.31 (m, 3H), 7.28-7.24 (m, 2H), 7.08-7.04 (m, 1H), 6.82 (s, 0.5H), 6.80 (s, 0.5H), 4.32-3.97 (m, 2H), 2.30-2.26 (m, 2H), 2.16-2.08 (m, 1H), 2.06-1.99 (m, 1H), 1.77-1.70 (m, 3H), 1.66 (s, 1.5H), 1.65 (s, 1.5H), 1.51-1.08 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.3, 175.1, 173.8, 143.6, 143.3, 137.80, 137.76, 128.89, 128.85, 127.5, 127.4, 127.1, 126.9, 124.18, 124.15, 119.7, 63.7, 63.6, 51.7, 44.3, 44.1, 34.8, 32.6, 32.5, 31.9, 31.7, 30.8, 30.5, 27.3, 23.94, 23.89, 20.83, 20.75, 20.54, 20.52 ppm; HRMS (ESI) calcd for $C_{25}H_{32}NO_3$ $[M+H]^+$ 394.2377, found 394.2379.



1-Phenyl-5-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)pentan-1-one (4a): colorless oil (42%, 26.6 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.57-7.53 (m, 1H), 7.48-7.44 (m, 2H), 3.78 (t, *J* = 6.4 Hz, 2H), 3.01 (t, *J* = 7.6 Hz, 2H), 1.88-1.80 (m, 2H), 1.66-1.59 (m, 2H), 1.45-1.42 (m, 4H), 1.32-1.25 (m, 2H), 1.15 (s, 6H), 1.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 137.0, 132.9, 128.6, 128.0, 59.6, 39.5, 38.6, 33.1 28.4, 21.5, 20.1, 17.1 ppm; HRMS (ESI) calcd for $C_{20}H_{32}NO_2$ $[M+H]^+$ 318.2428, found 318.2433.



***N*-(2-Methyl-2,8-diphenyloctyl)aniline (5aa):** colorless oil (87%, 67.3 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:20, $d.r._{HPLC}$ = 1:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36-7.22 (m, 11H), 7.15-7.11 (m, 2H), 6.69-6.65 (m, 1H), 6.53 (d, *J* = 8.0 Hz, 2H), 4.63-4.60 (m, 1H), 3.32 (d, *J* = 11.6 Hz, 1H), 3.22 (d, *J* = 11.6 Hz, 1H), 1.81-1.61 (m, 5H), 1.41 (s, 3H), 1.30-1.22 (m, 5H), 1.05-1.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.5, 145.2, 144.8, 129.1, 128.5, 128.4, 127.5, 126.5, 126.1, 125.8, 117.2, 112.9, 74.6, 55.1, 42.0, 41.1, 39.0, 30.1, 25.6, 23.7, 23.0 ppm; HRMS (ESI) calcd for $C_{27}H_{34}NO$ $[M+H]^+$ 388.2635, found 388.2632.

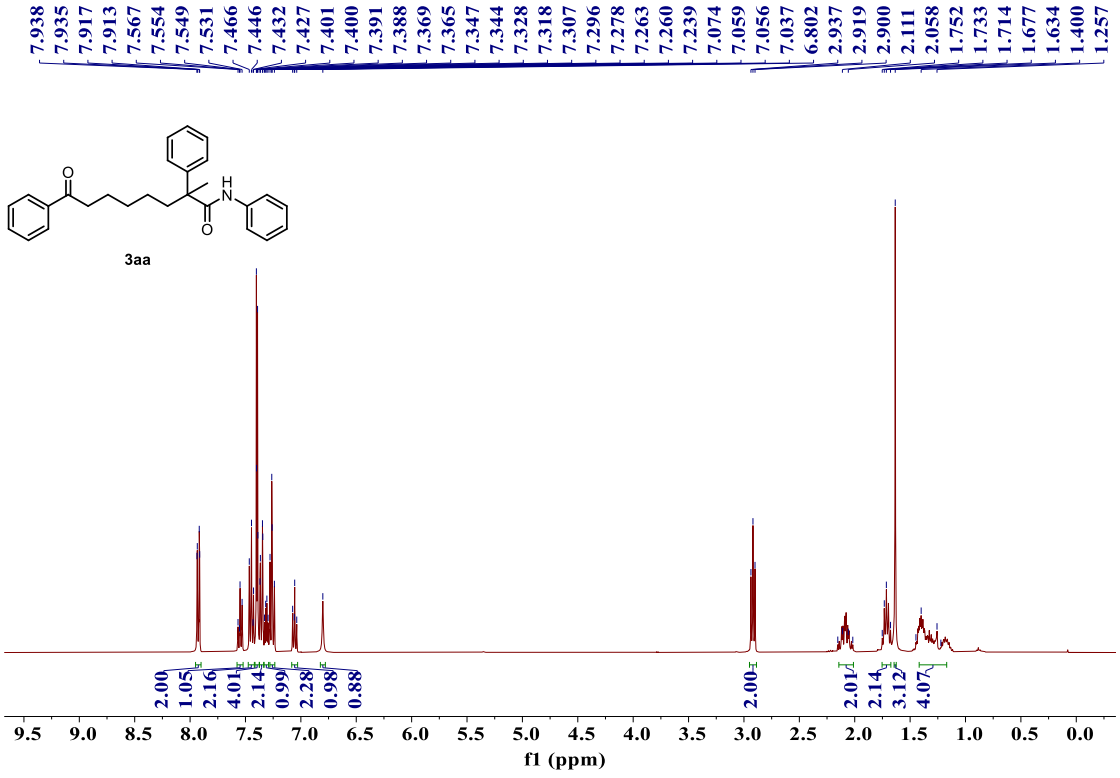


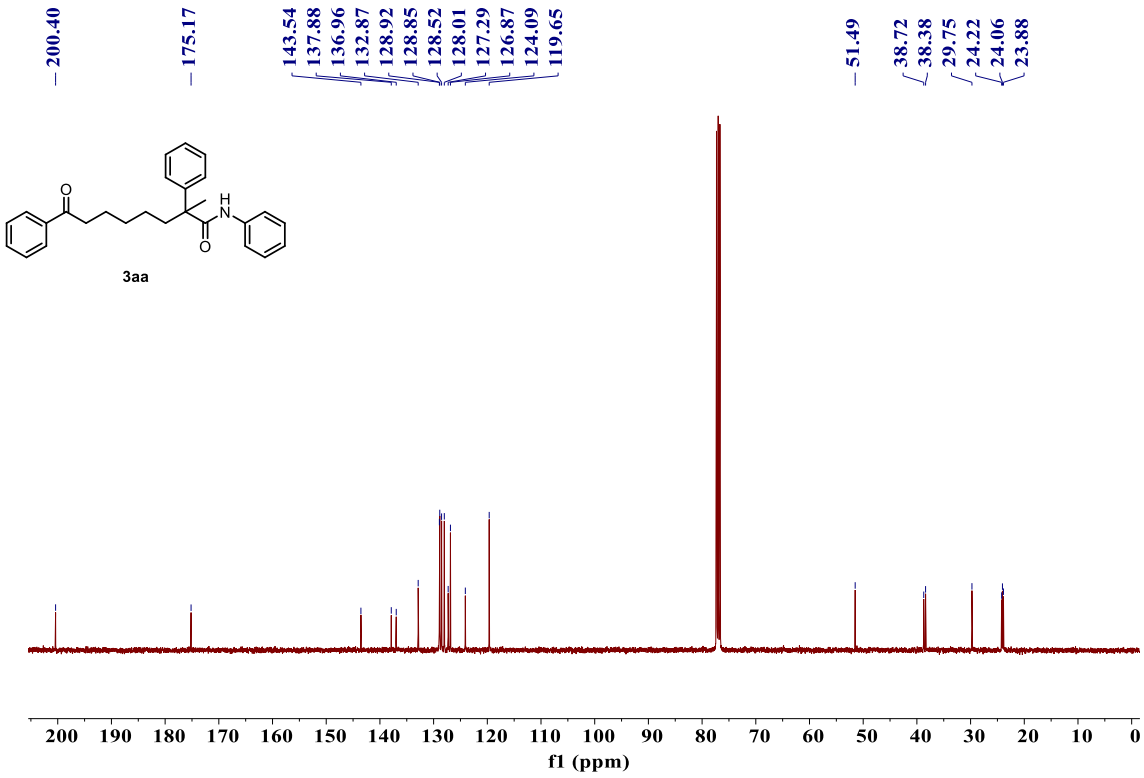
8-Hydroxy-2-methyl-*N*,2,8-triphenyloctanamide (6aa): colorless oil (79%, 63.4 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:20, $d.r._{HPLC}$ = 1:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.42-7.38 (m, 4H), 7.36-7.29 (m, 7H), 7.28-7.24 (m, 3H), 7.08-7.04 (m, 1H), 6.81 (s, 1H), 4.63-4.60 (m, 1H), 2.11-1.99 (m, 3H), 1.77-1.64 (m, 2H), 1.61 (s, 3H), 1.39-1.23 (m, 5H), 1.15-1.06 (m, 1H); ^{13}C NMR (100 MHz,

$CDCl_3$): δ = 175.2, 144.8, 143.6, 137.9, 128.9, 128.8, 128.4, 127.4, 127.2, 126.9, 125.8, 124.1, 119.7, 74.5, 51.5, 38.9, 38.8, 29.9, 25.5, 24.2, 23.8 ppm; HRMS (ESI) calcd for $C_{27}H_{31}NO_2K$ $[M+K]^+$ 440.1986, found 440.1994.

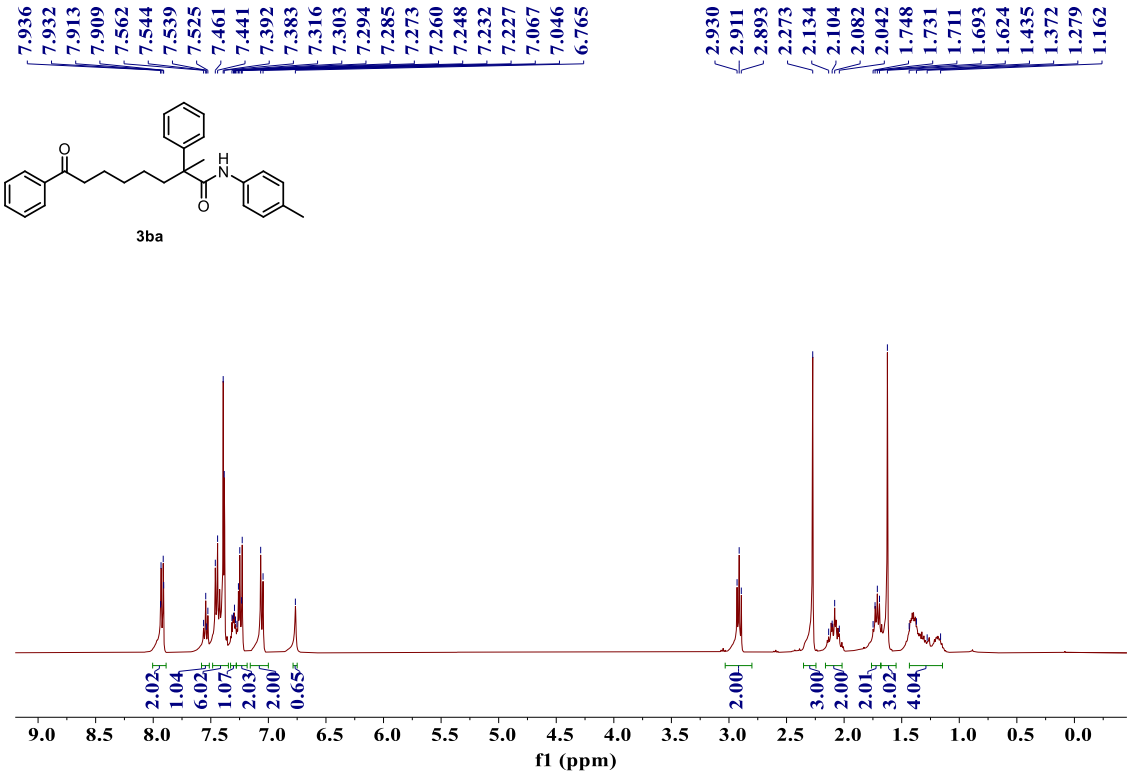
10. ^{1}H, ^{13}C and ^{19}F NMR Spectra of Products

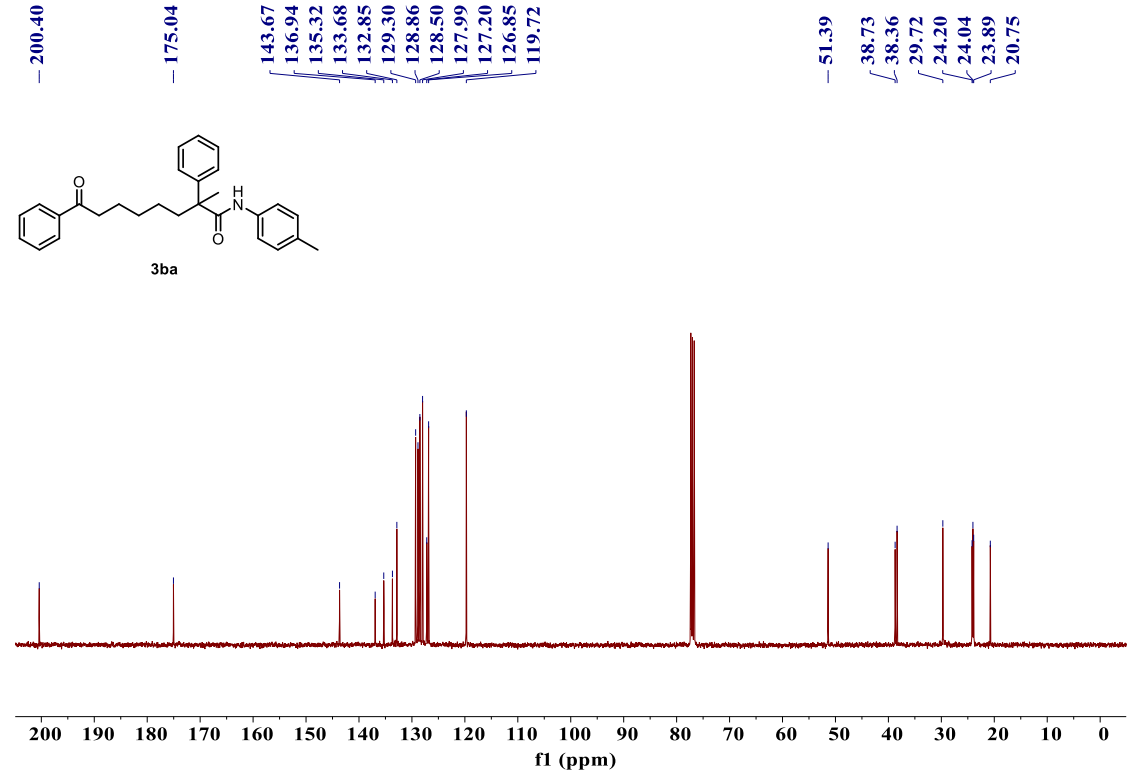
^{1}H and ^{13}C NMR spectra for product 3aa (Chloroform-*d*)



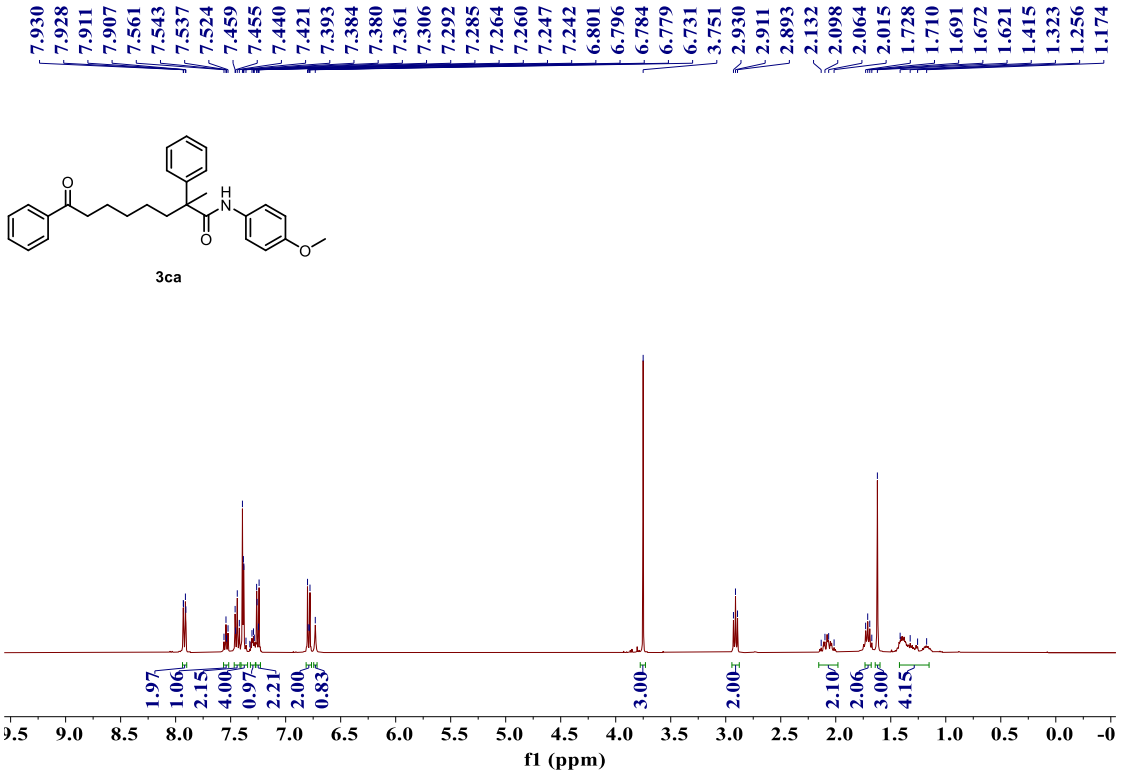


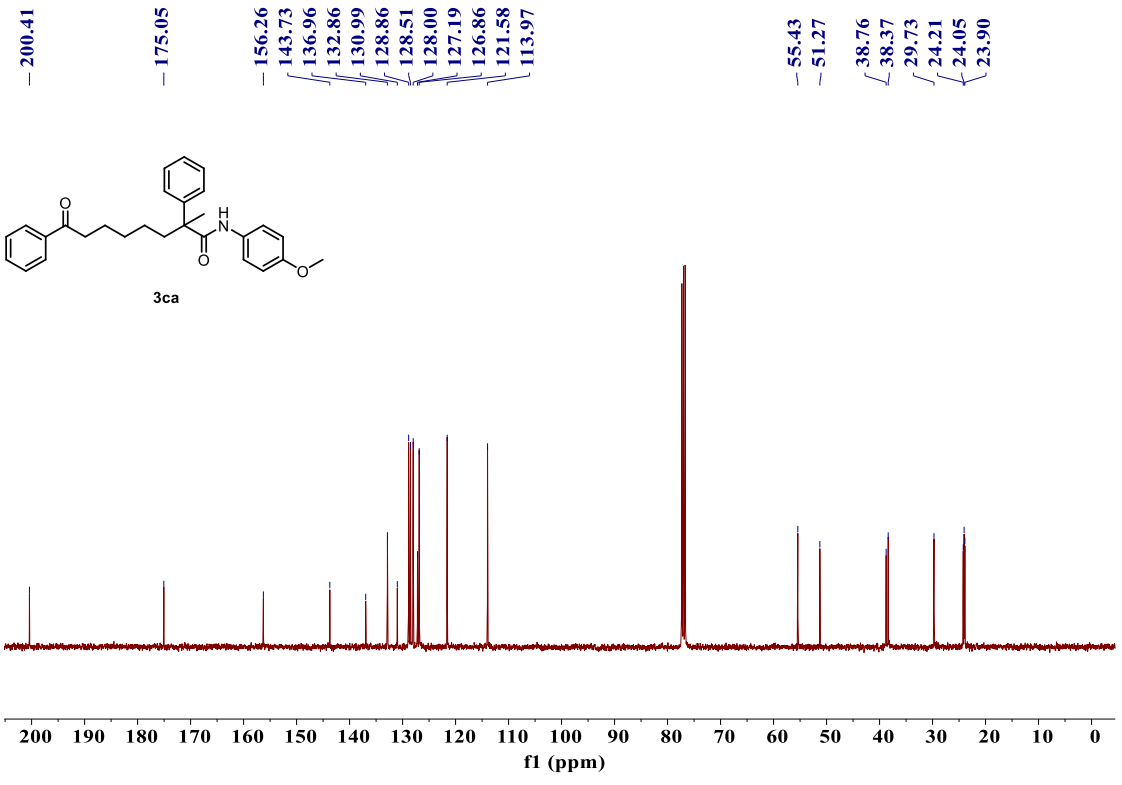
^{1}H and ^{13}C NMR spectra for product 3ba (Chloroform-*d*)



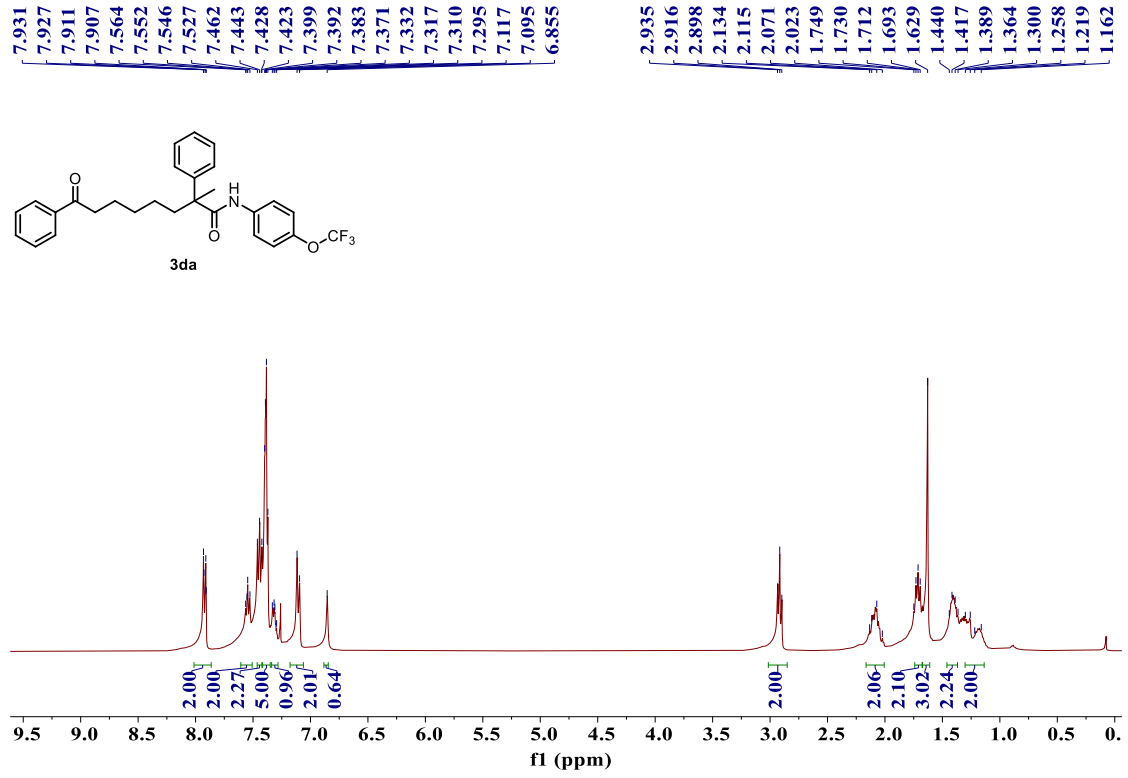


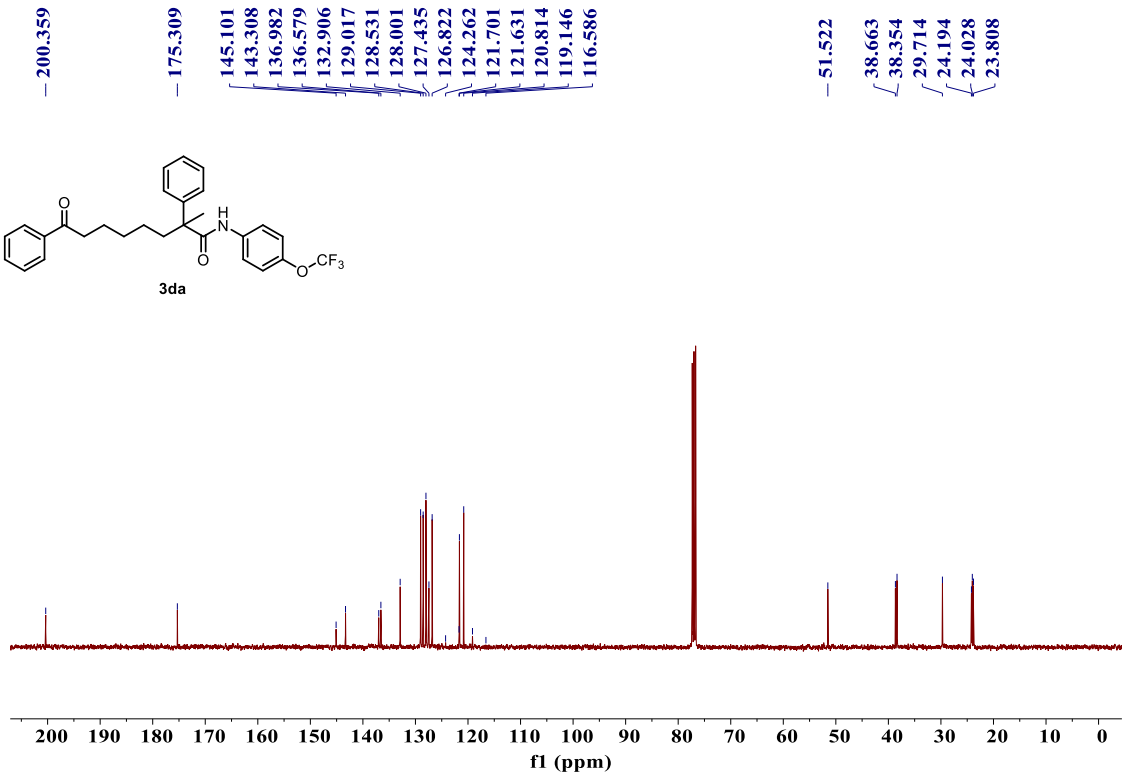
^{1}H and ^{13}C NMR spectra for product 3ca (Chloroform-*d*)

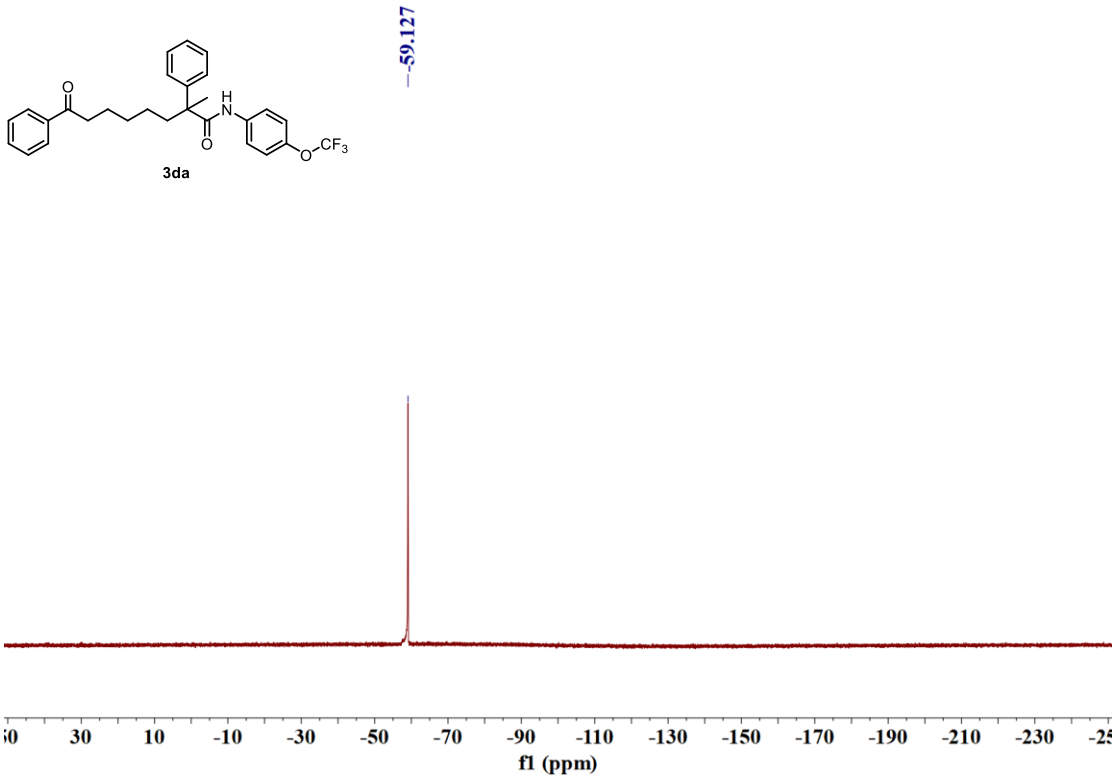




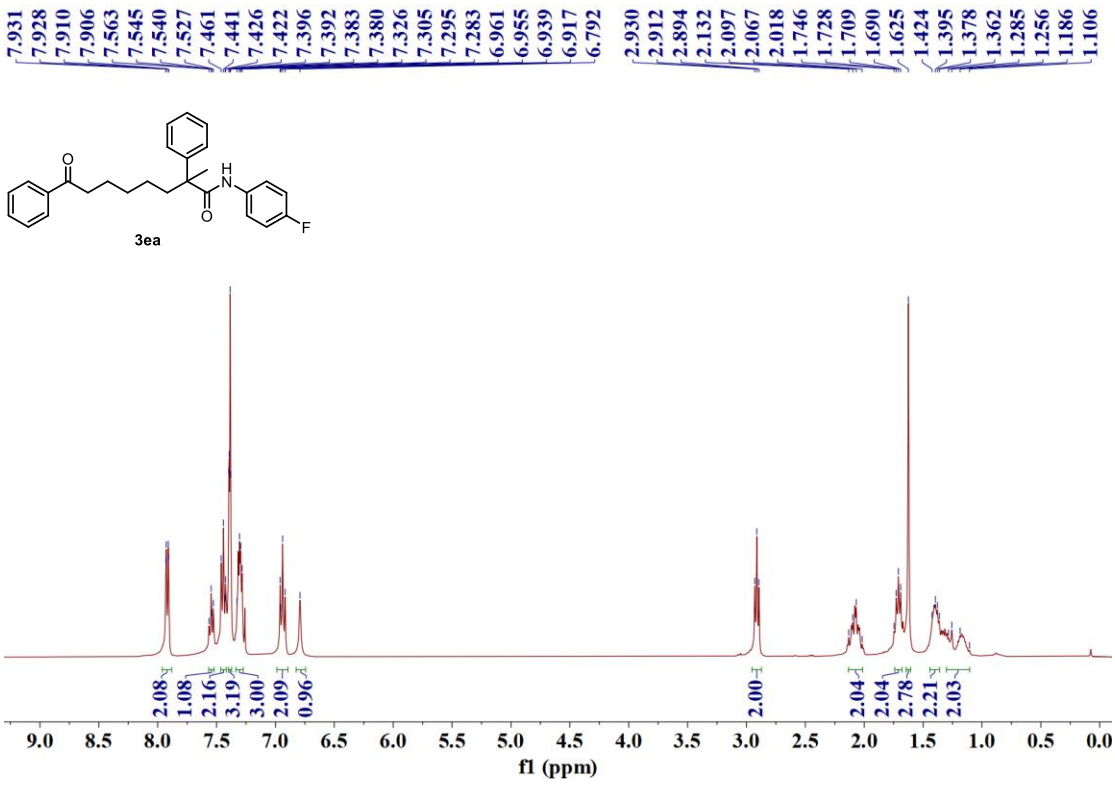
^{1}H, ^{13}C and ^{19}F NMR spectra for product 3da (Chloroform-*d*)

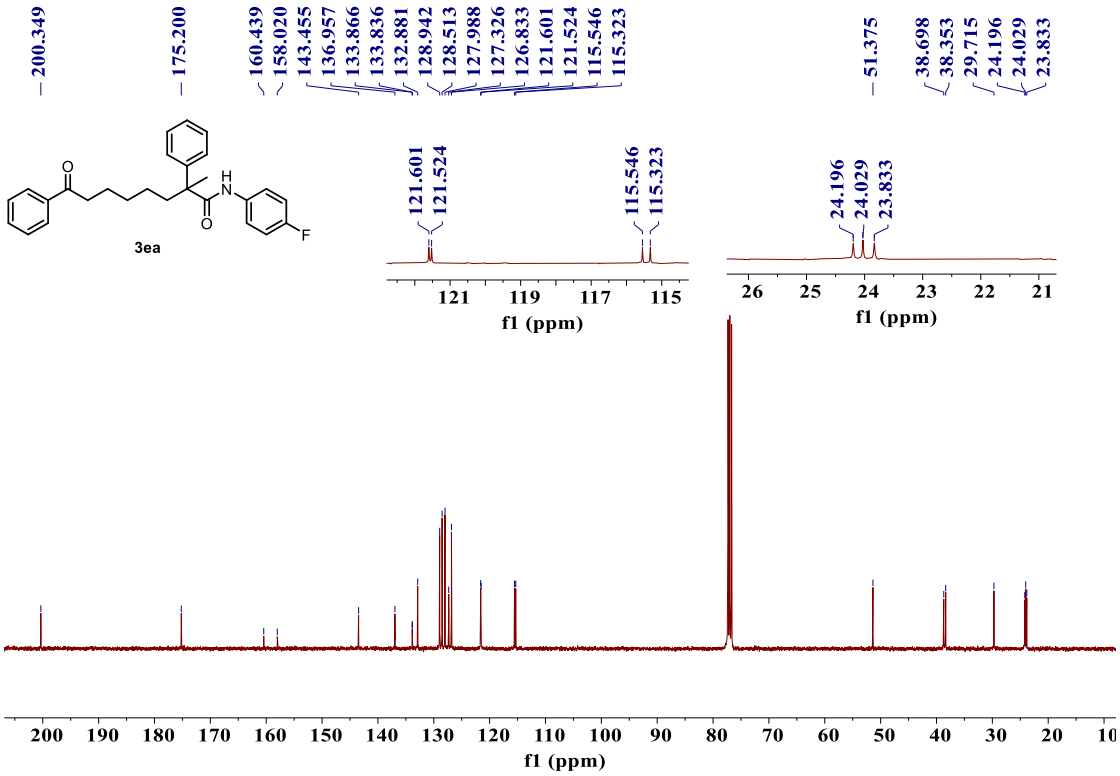


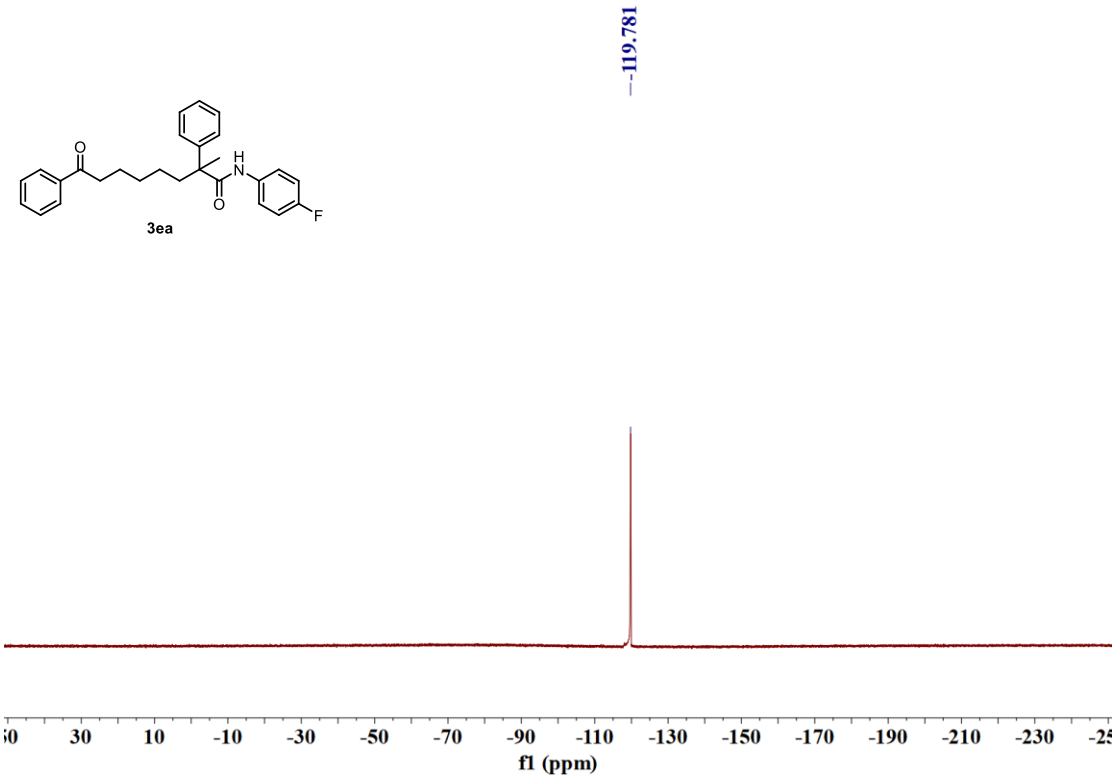




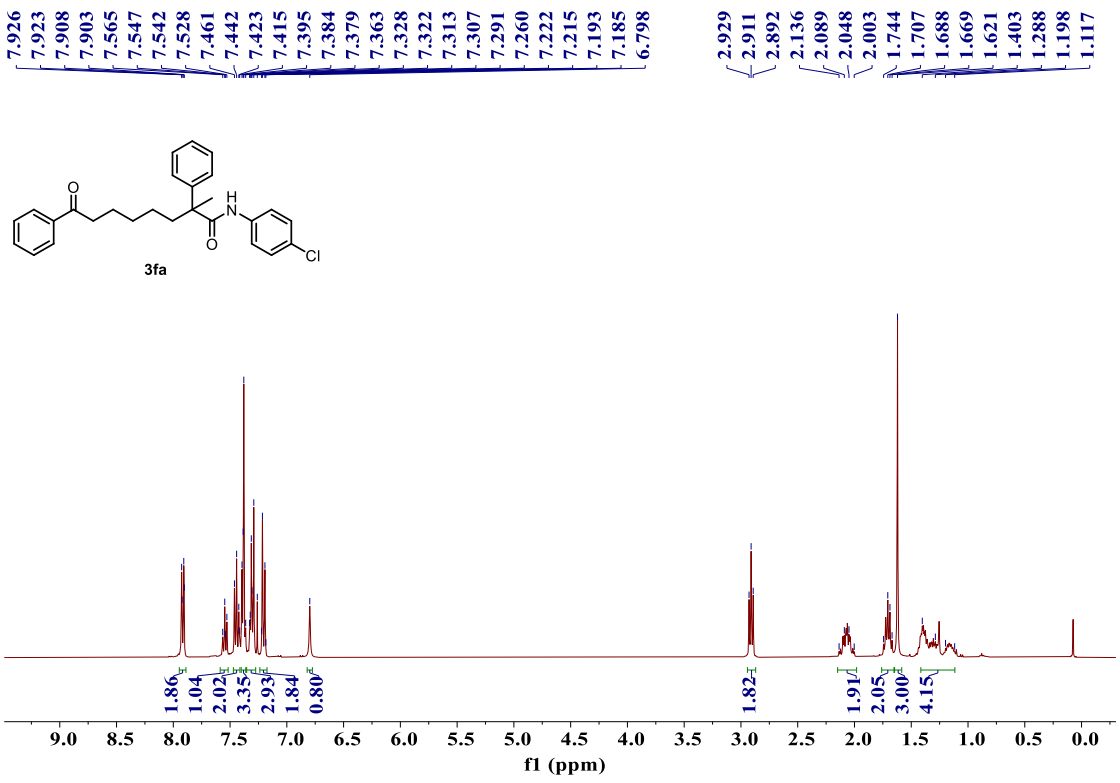
^{1}H, ^{13}C and ^{19}F NMR spectra for product 3ea (Chloroform-*d*)

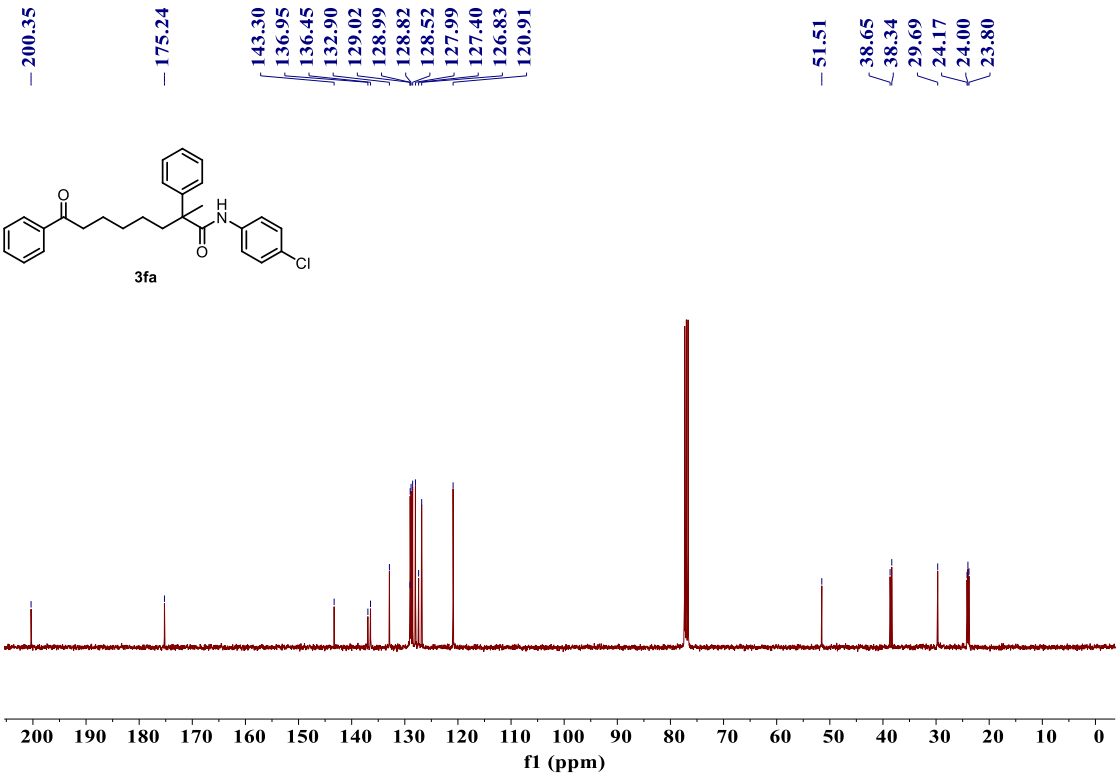



200.349
175.200
160.439
158.020
143.455
136.957
133.866
133.836
132.881
128.942
128.513
127.988
127.326
126.833
121.601
121.524
115.546
115.323
51.375
38.698
38.353
29.715
24.196
24.029
23.833
3ea
f1 (ppm)

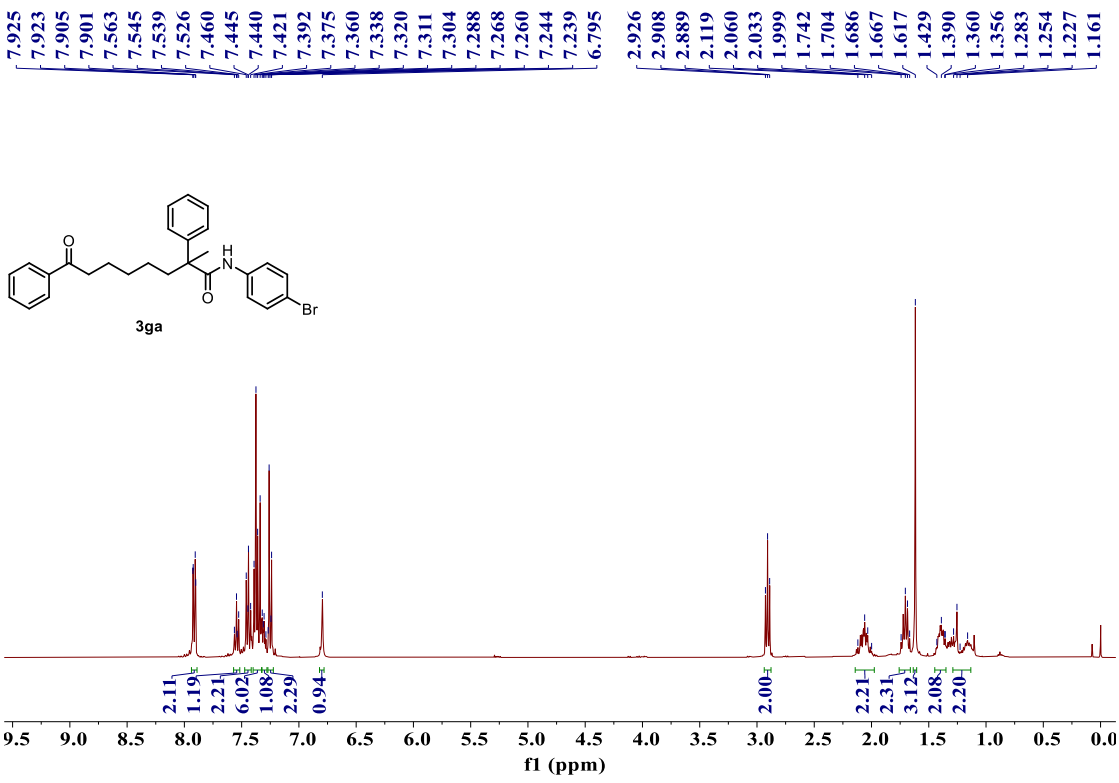

-119.781
3ea
f1 (ppm)

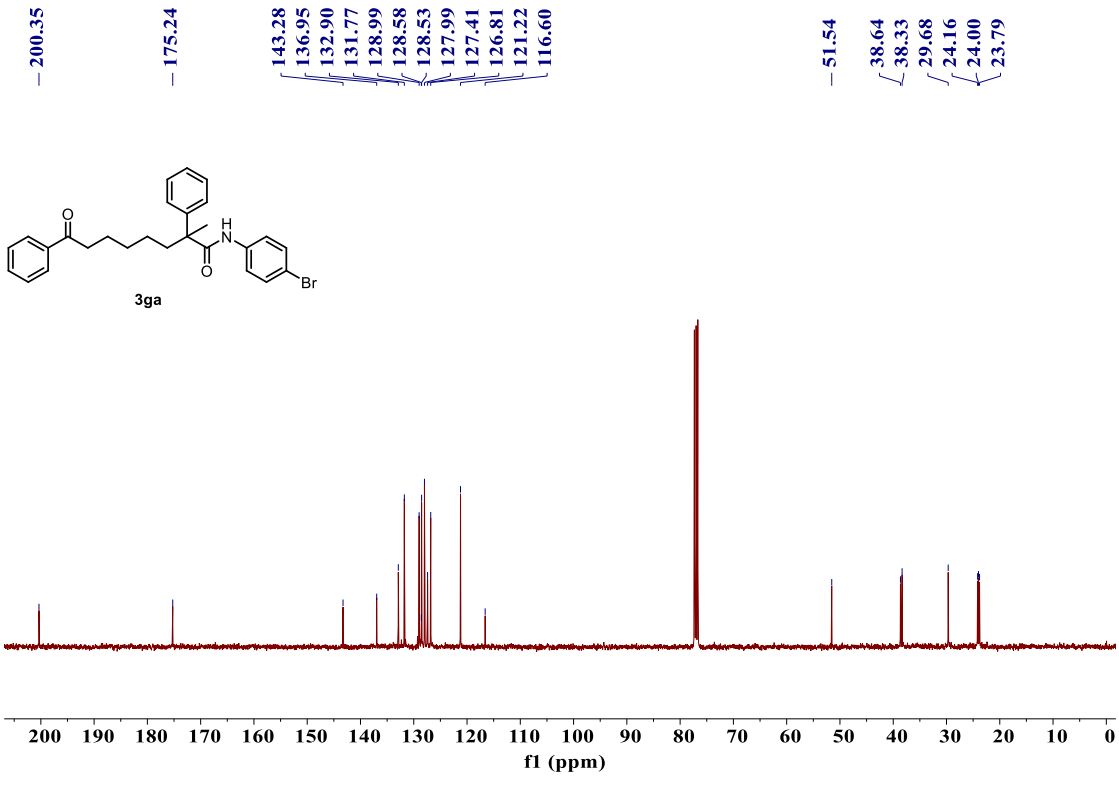
^{1}H and ^{13}C NMR spectra for product 3fa (Chloroform-*d*)



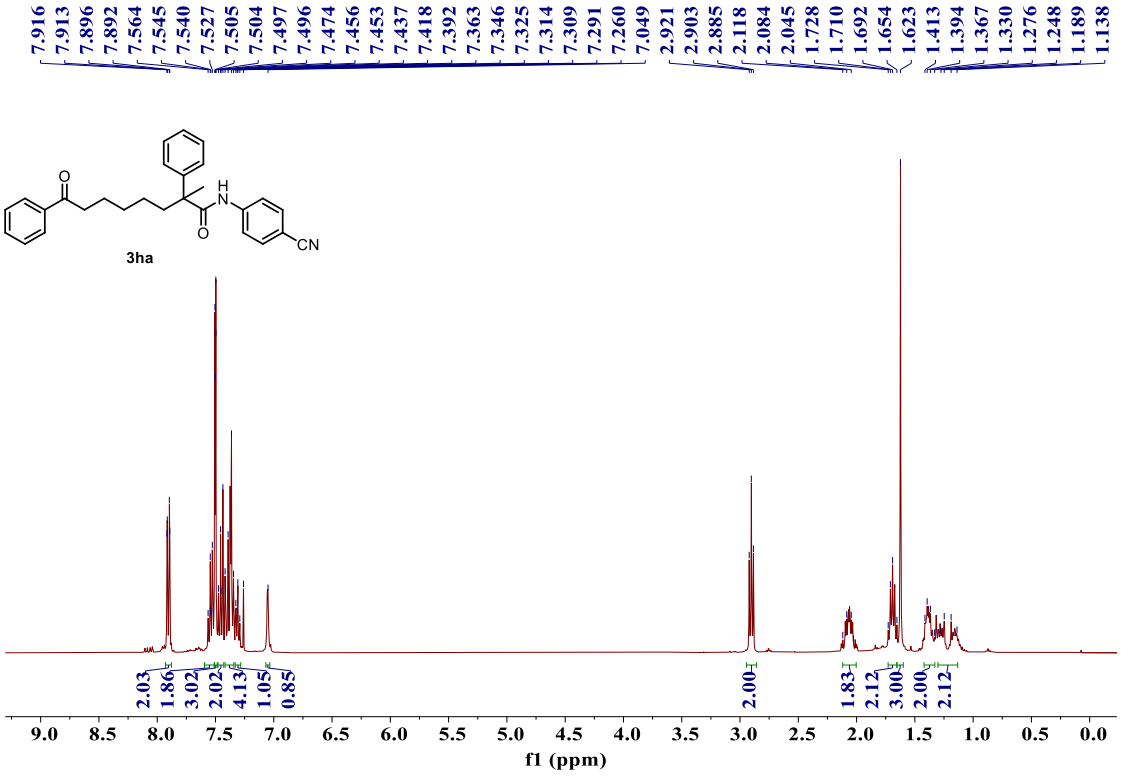


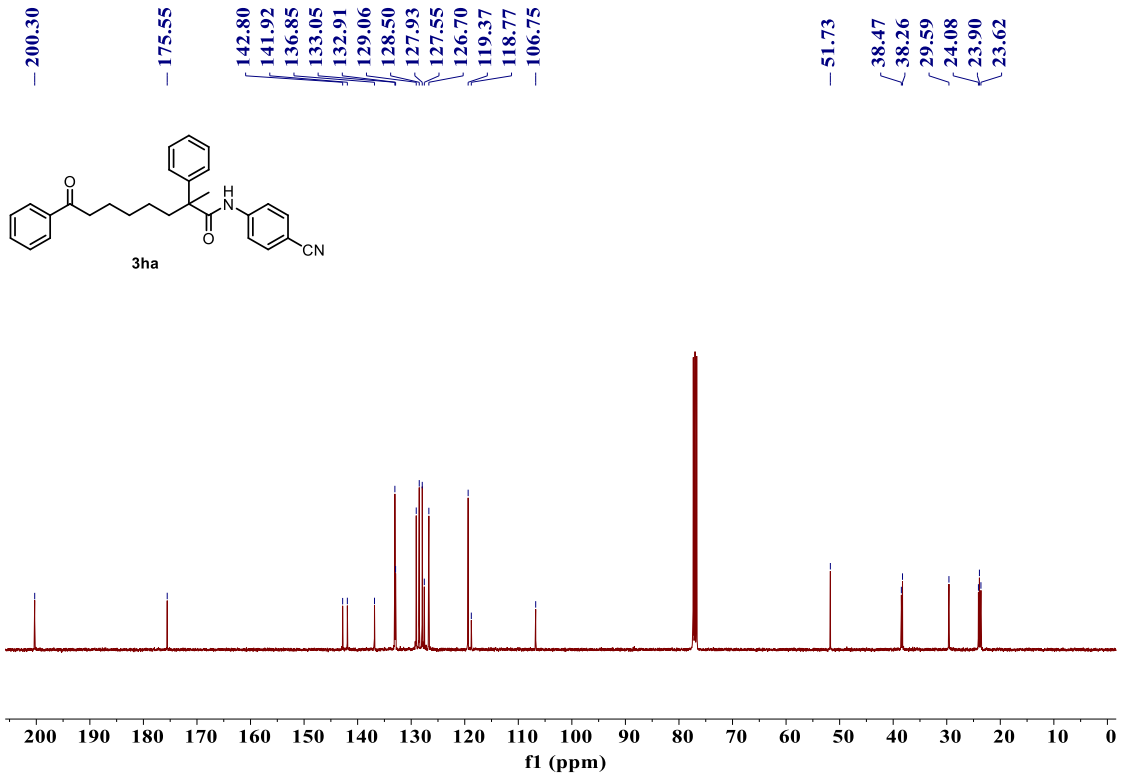
^{1}H and ^{13}C NMR spectra for product 3ga (Chloroform-*d*)



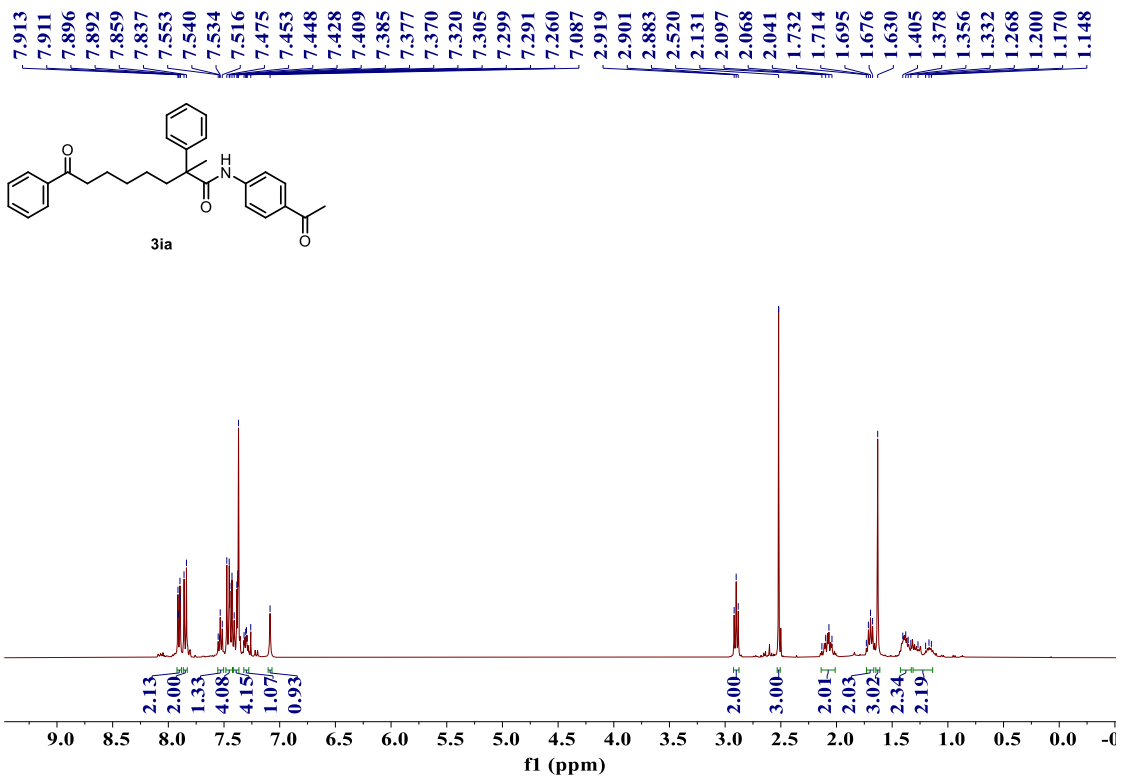


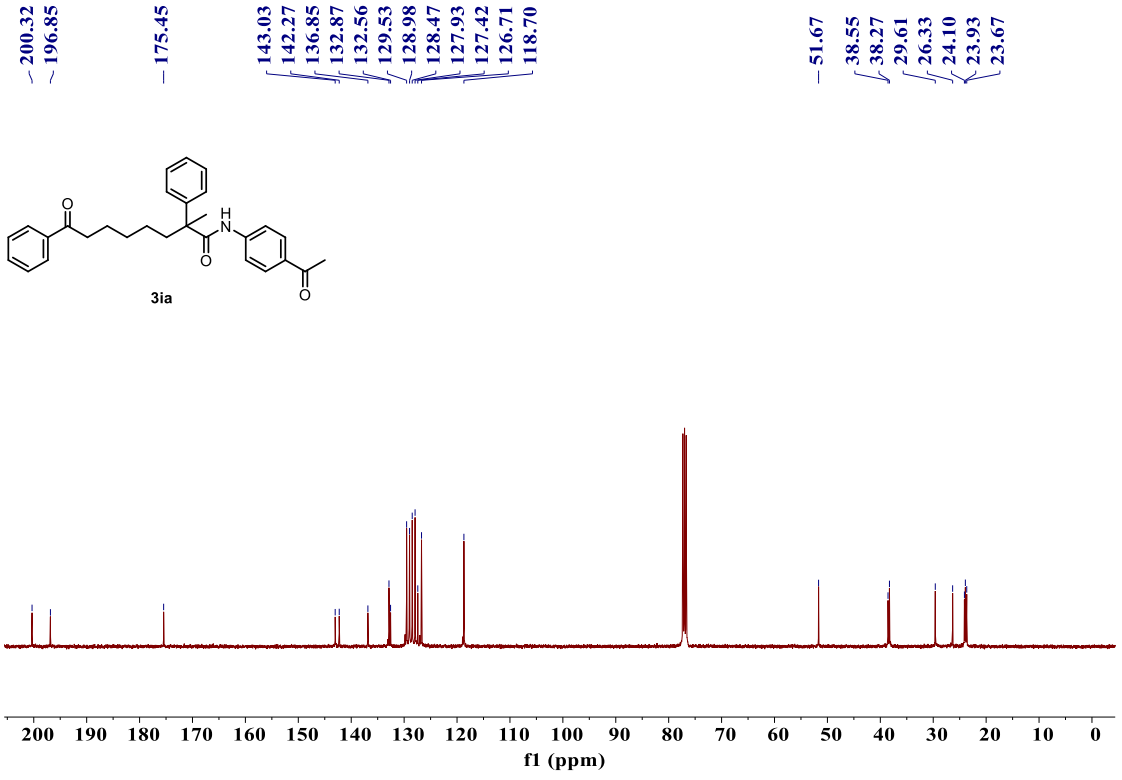
^{1}H and ^{13}C NMR spectra for product 3ha (Chloroform-*d*)



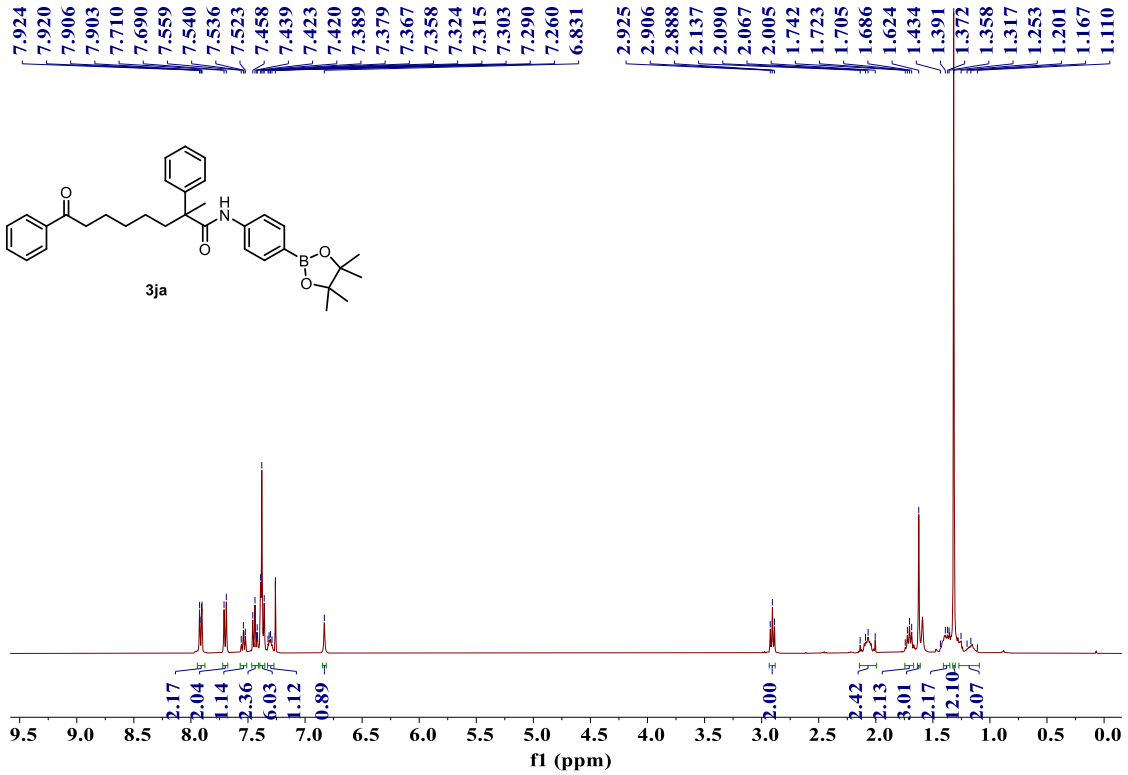


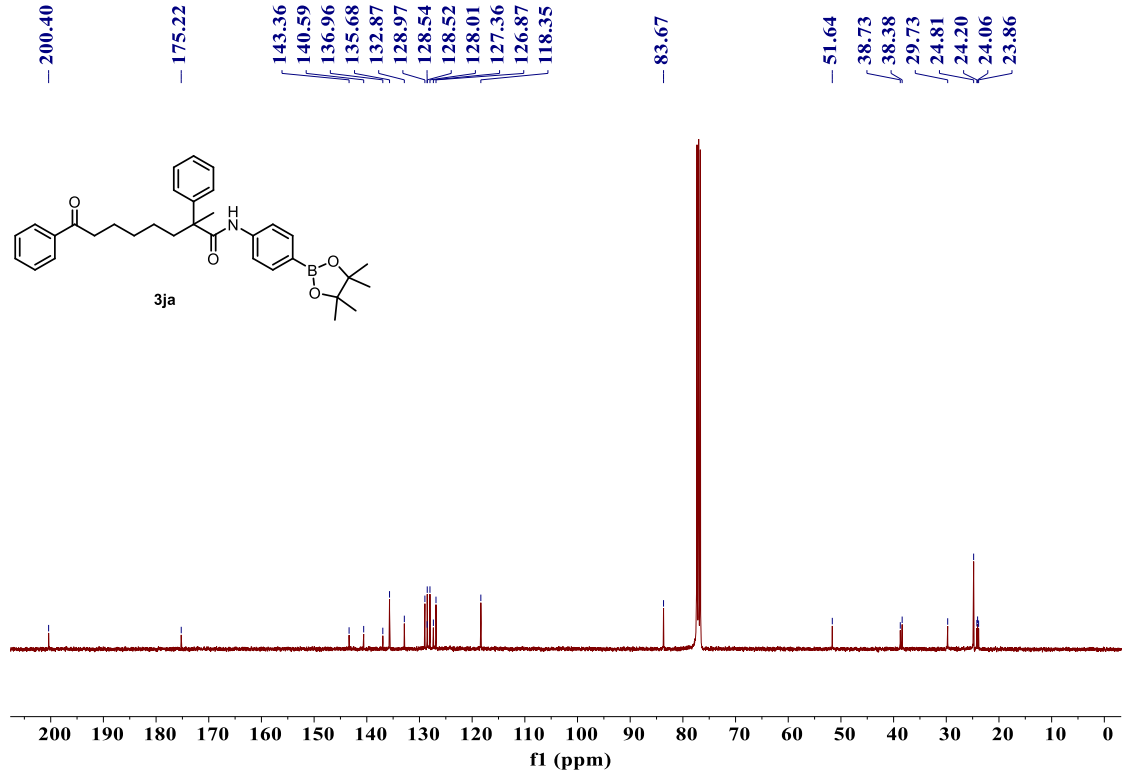
^{1}H and ^{13}C NMR spectra for product 3ia (Chloroform-*d*)



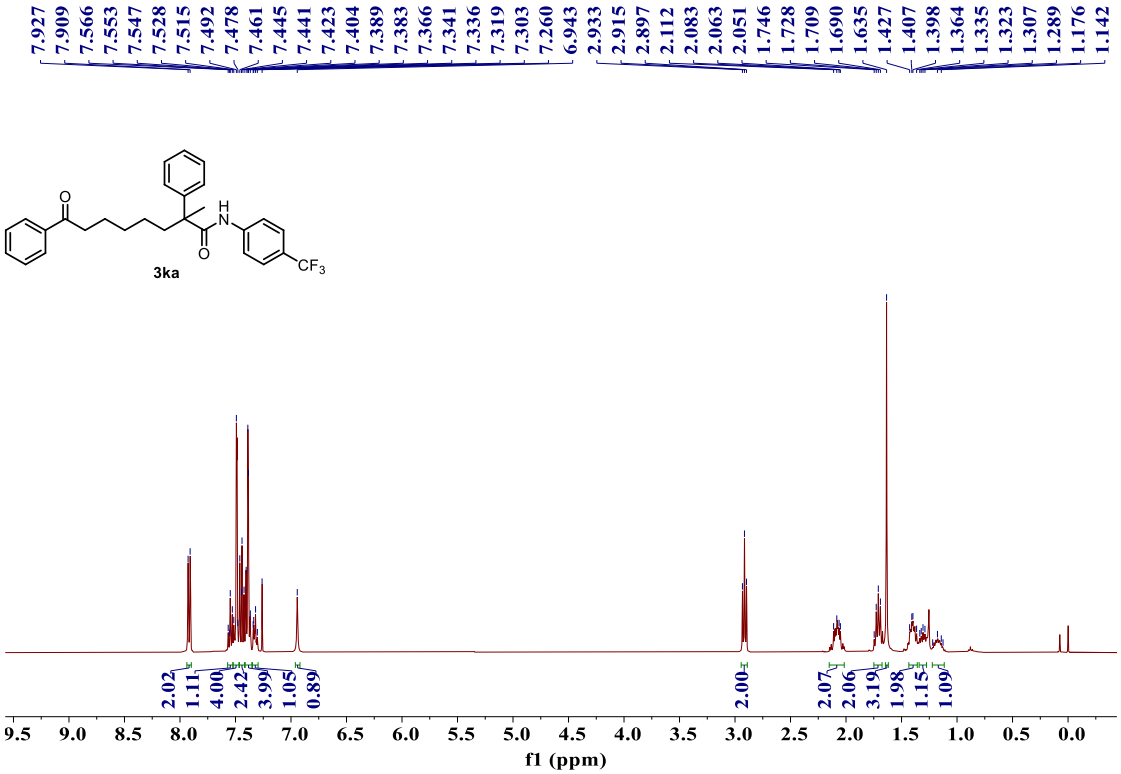


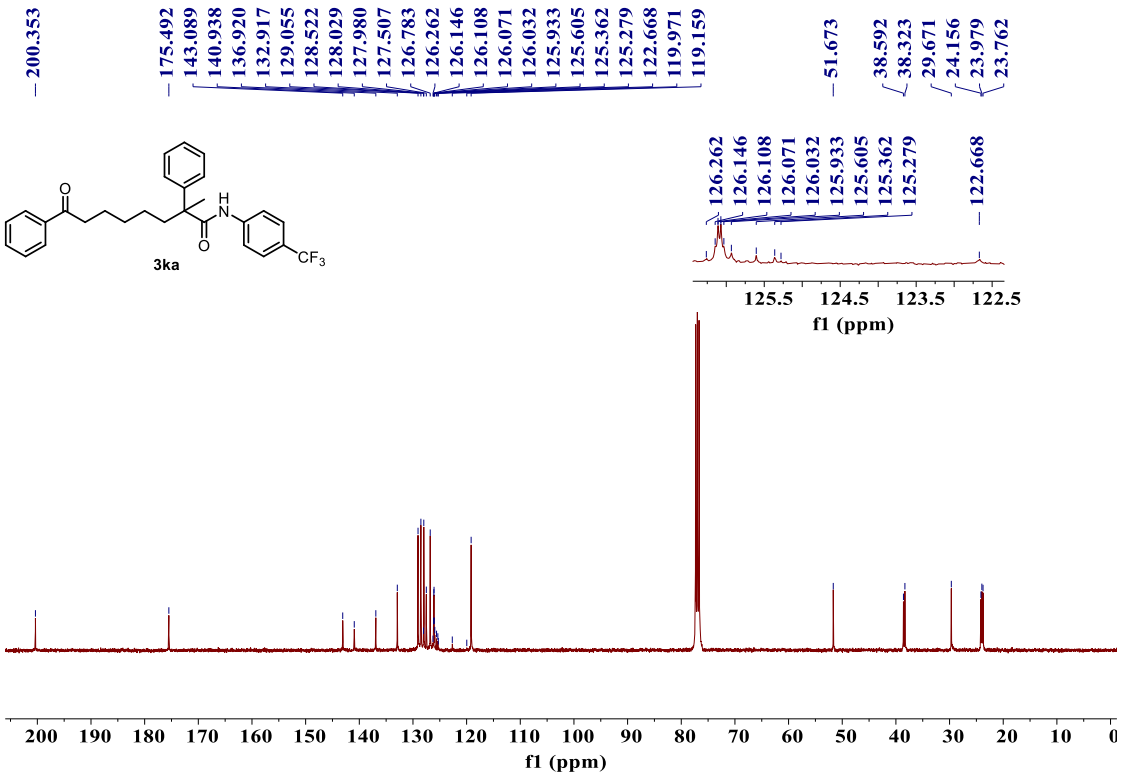
^{1}H and ^{13}C NMR spectra for product 3ja (Chloroform-*d*)

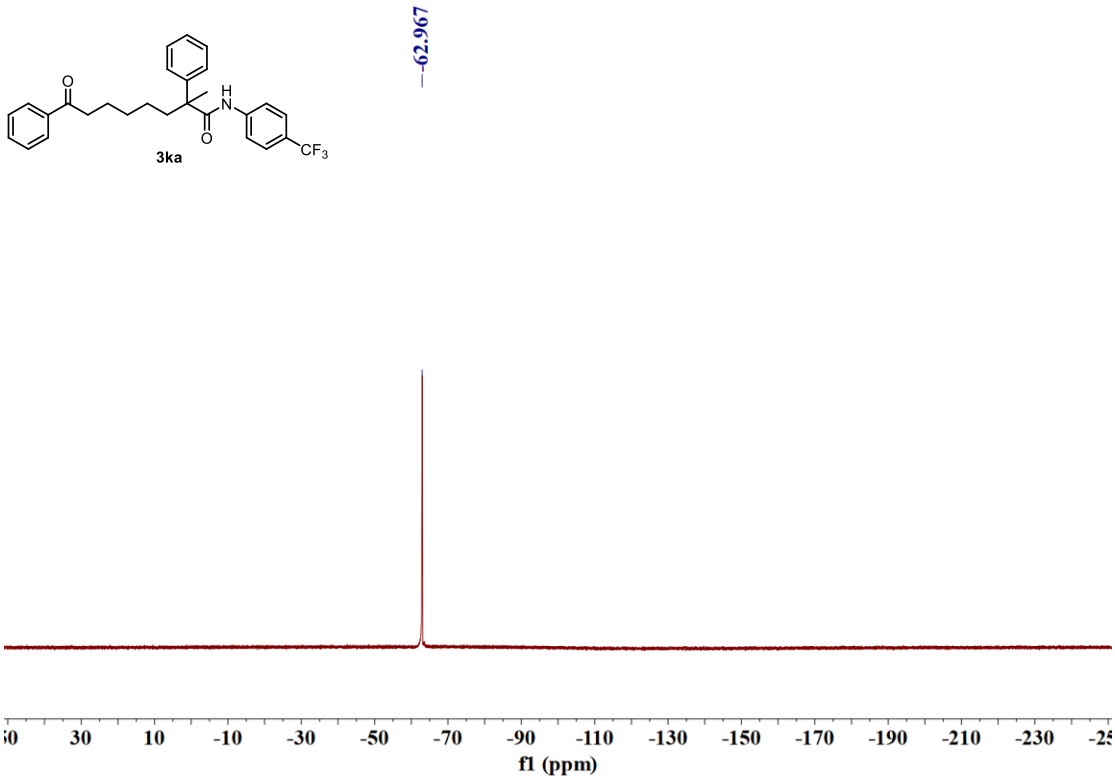




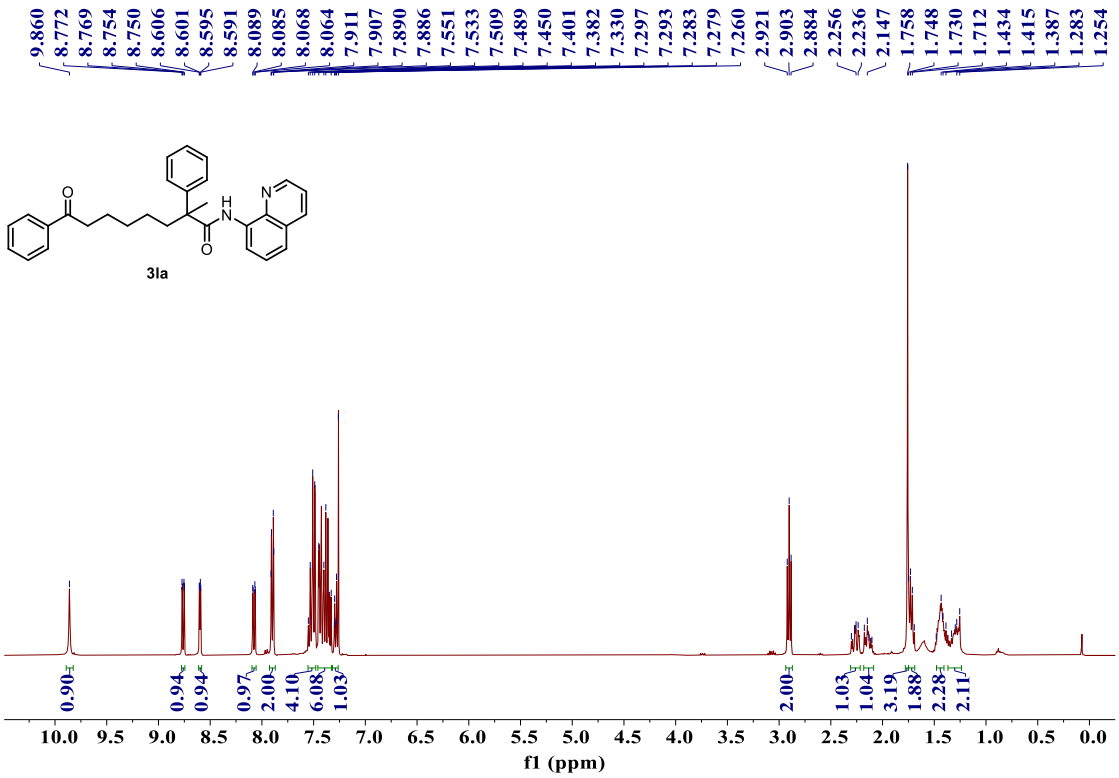
^{1}H, ^{13}C and ^{19}F NMR spectra for product 3ka (Chloroform-*d*)

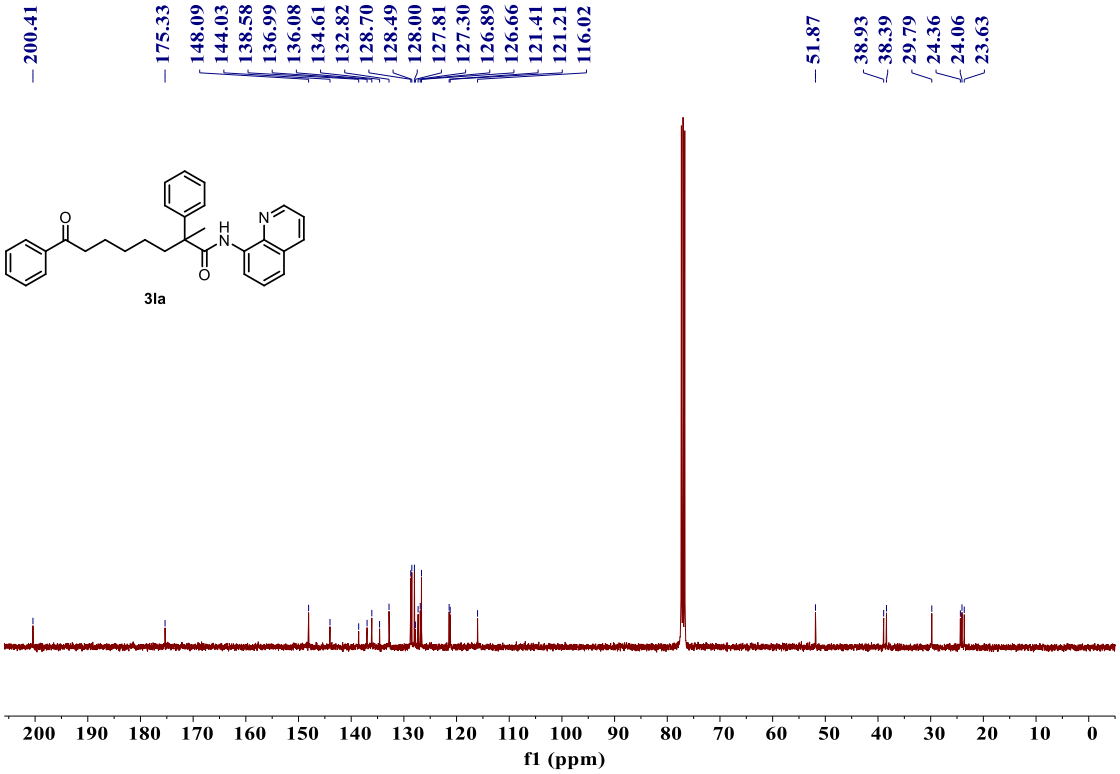




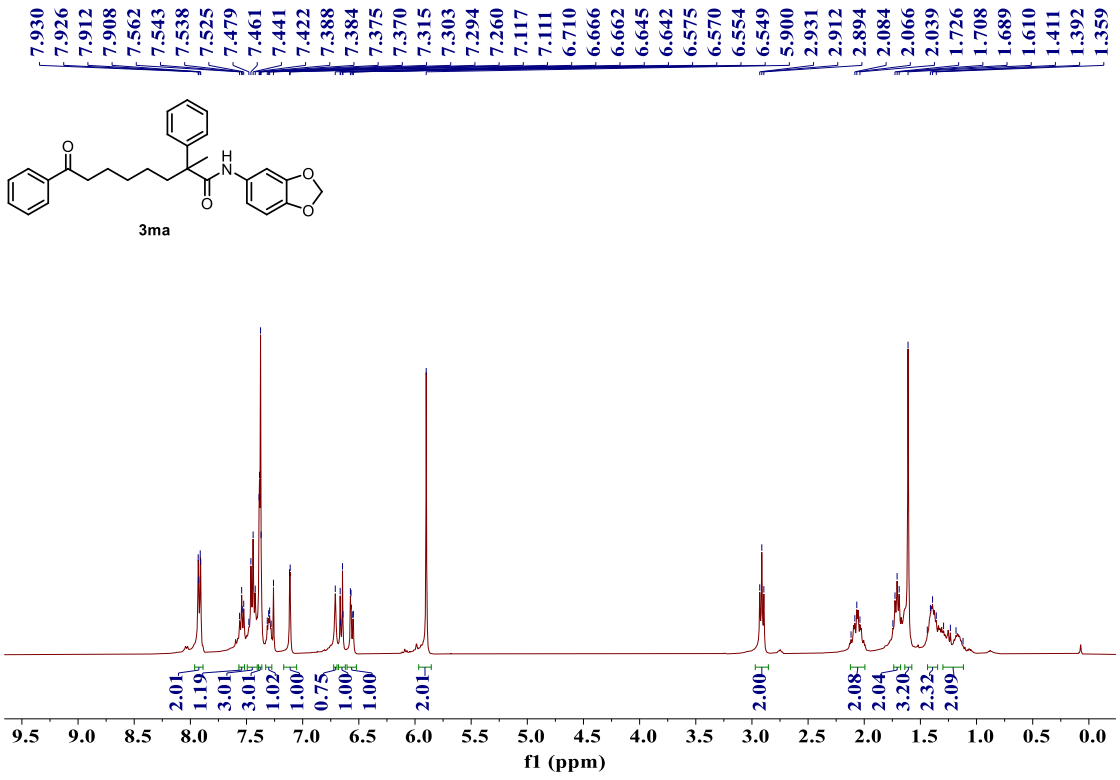


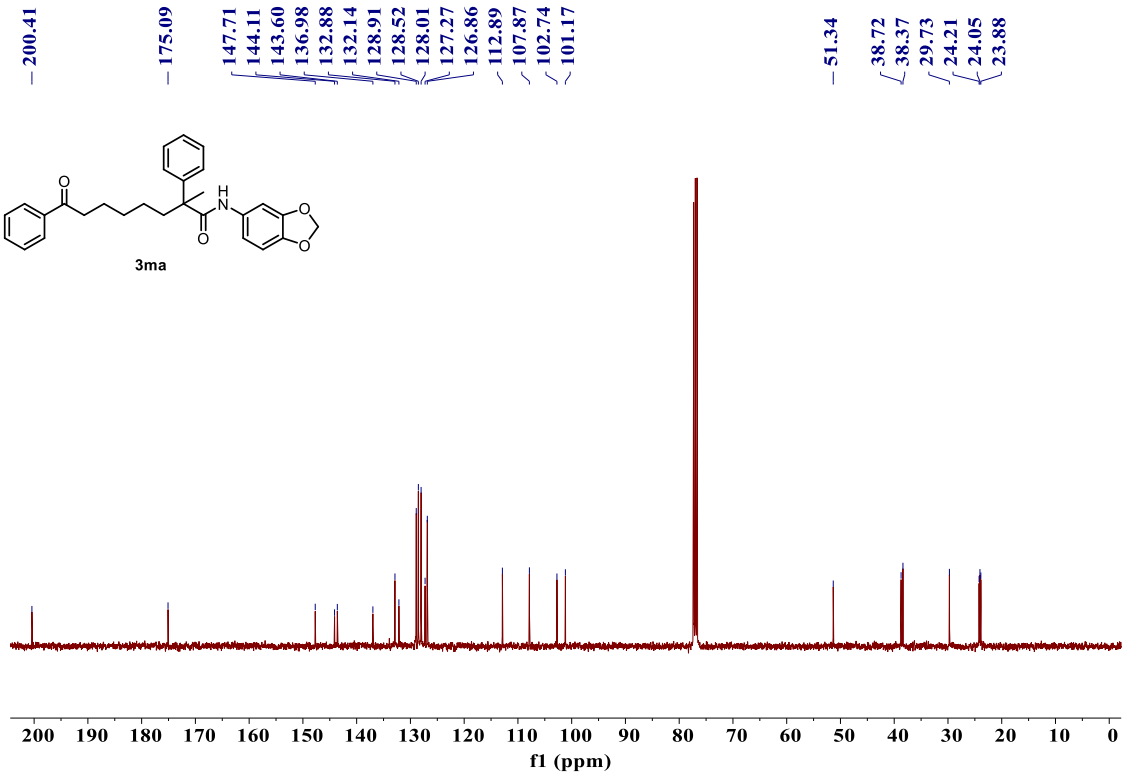
^{1}H and ^{13}C NMR spectra for product 3la (Chloroform-*d*)



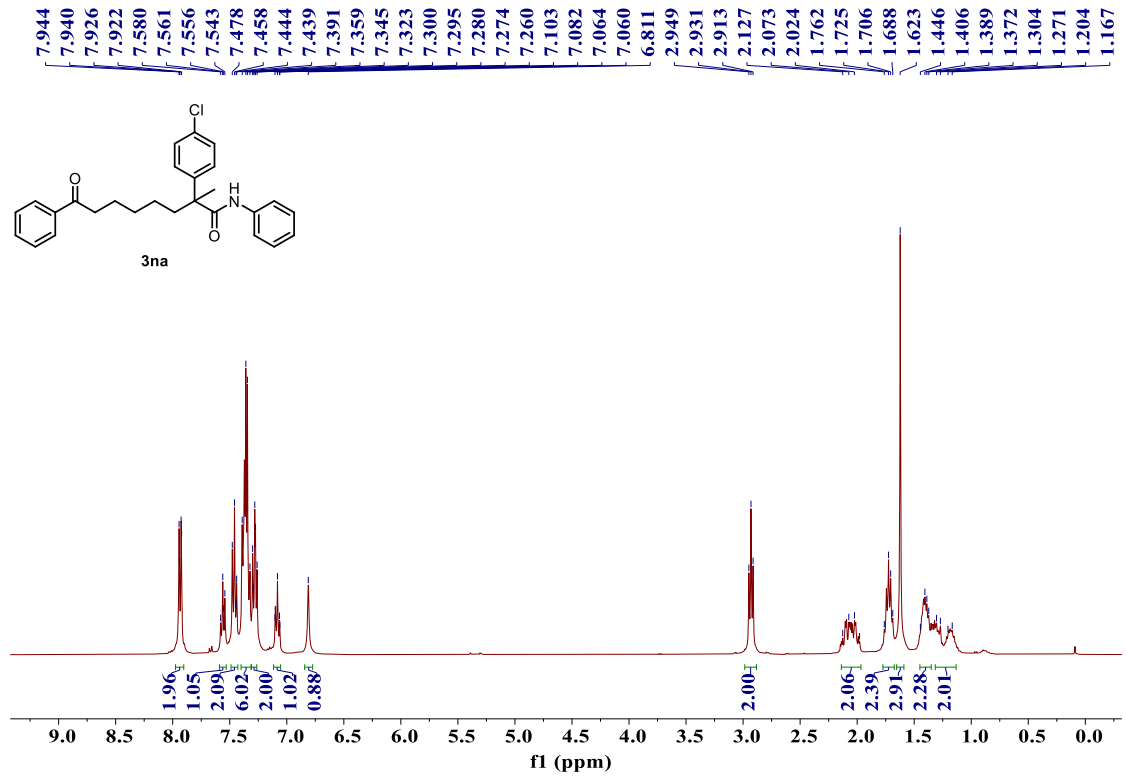


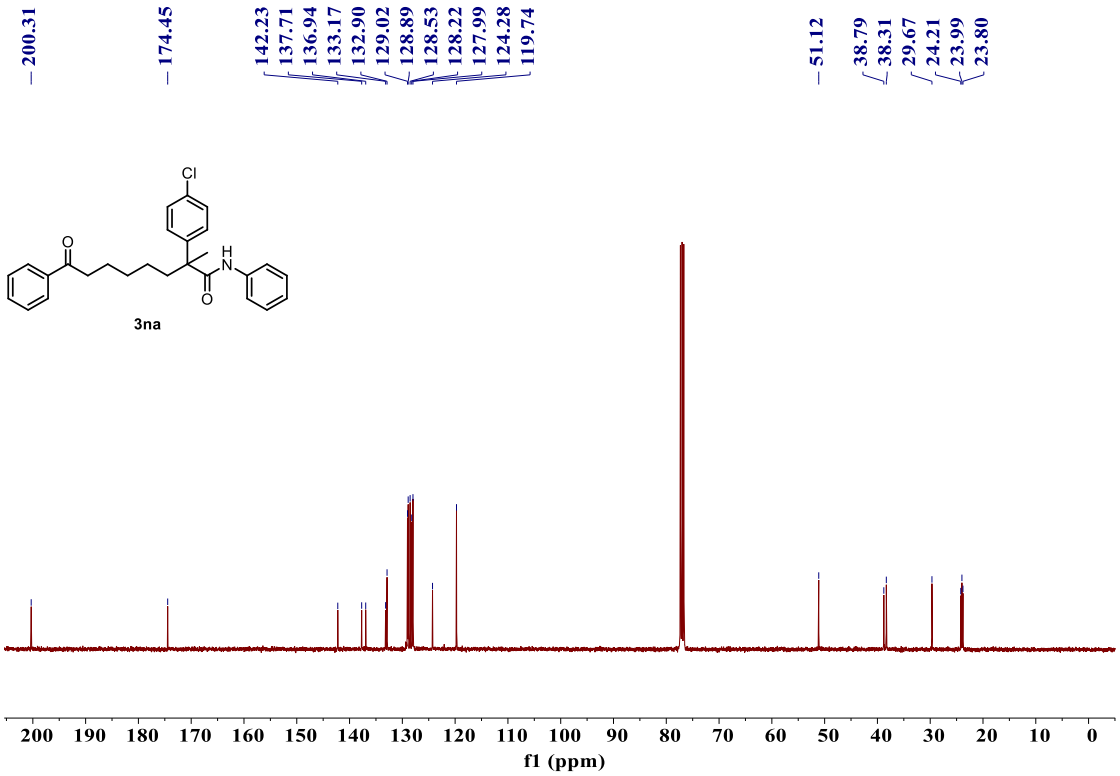
^{1}H and ^{13}C NMR spectra for product 3ma (Chloroform-*d*)



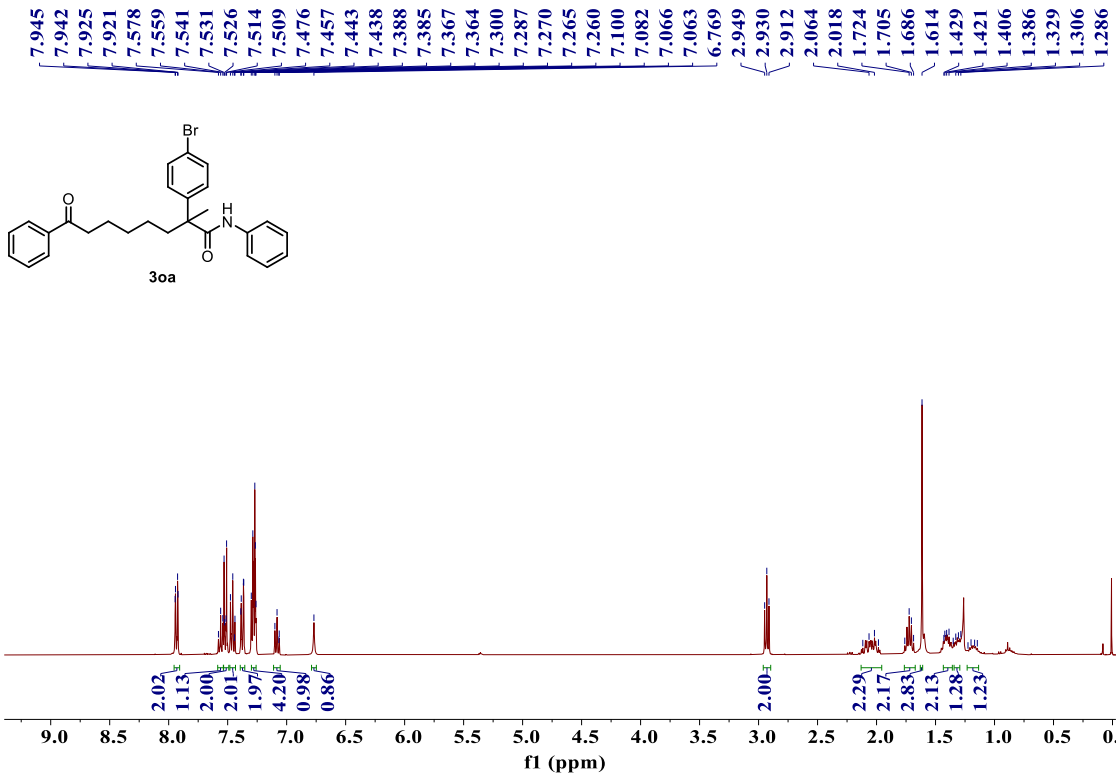


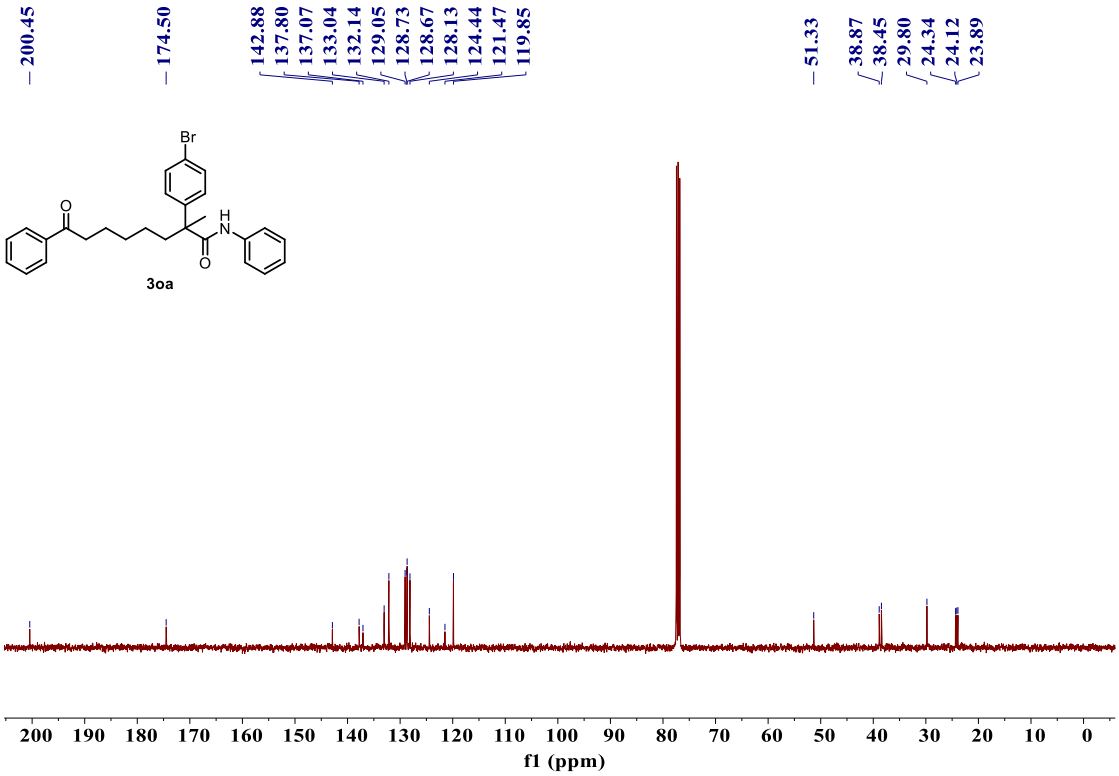
^{1}H and ^{13}C NMR spectra for product 3na (Chloroform-*d*)



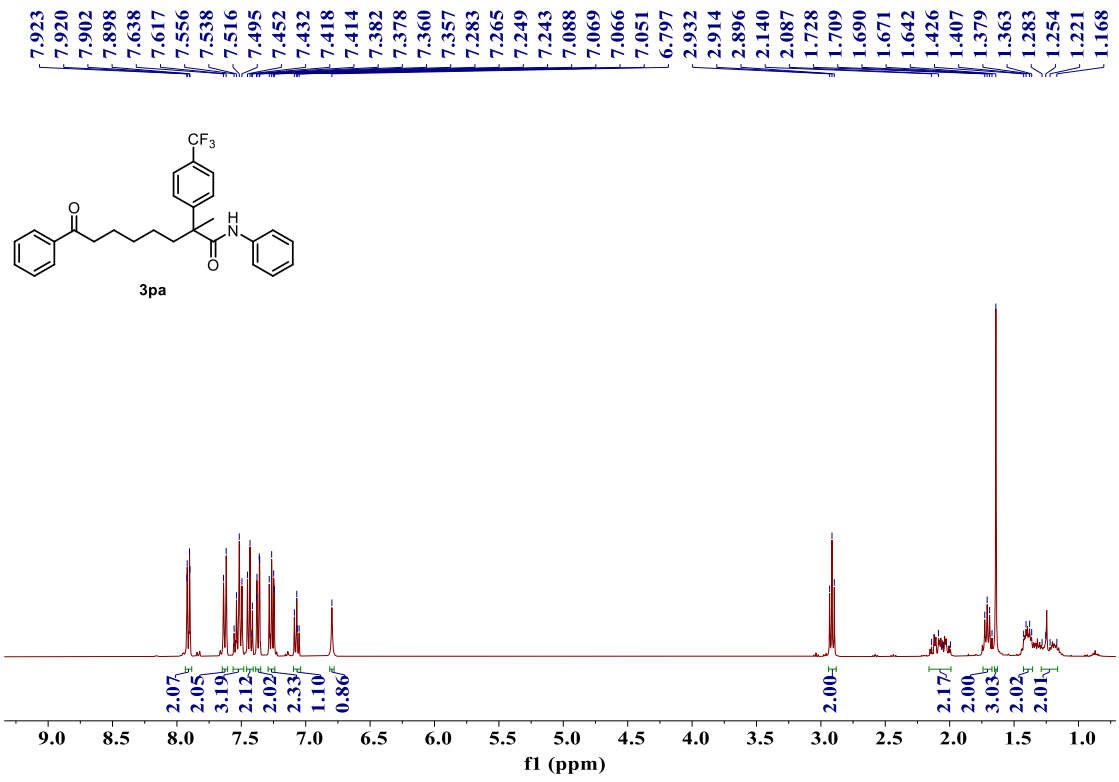


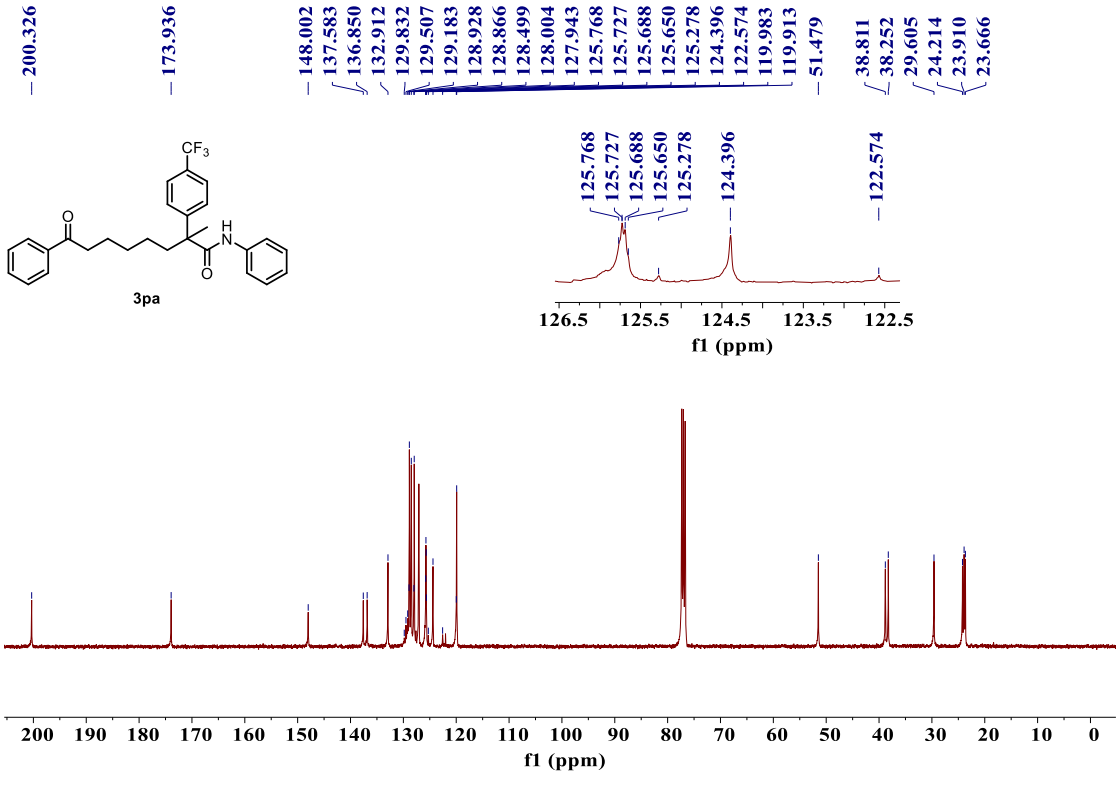
^{1}H and ^{13}C NMR spectra for product 3oa (Chloroform-*d*)





^{1}H, ^{13}C and ^{19}F NMR spectra for product 3pa (Chloroform-*d*)

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148.002
137.583
136.850
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129.832
129.507
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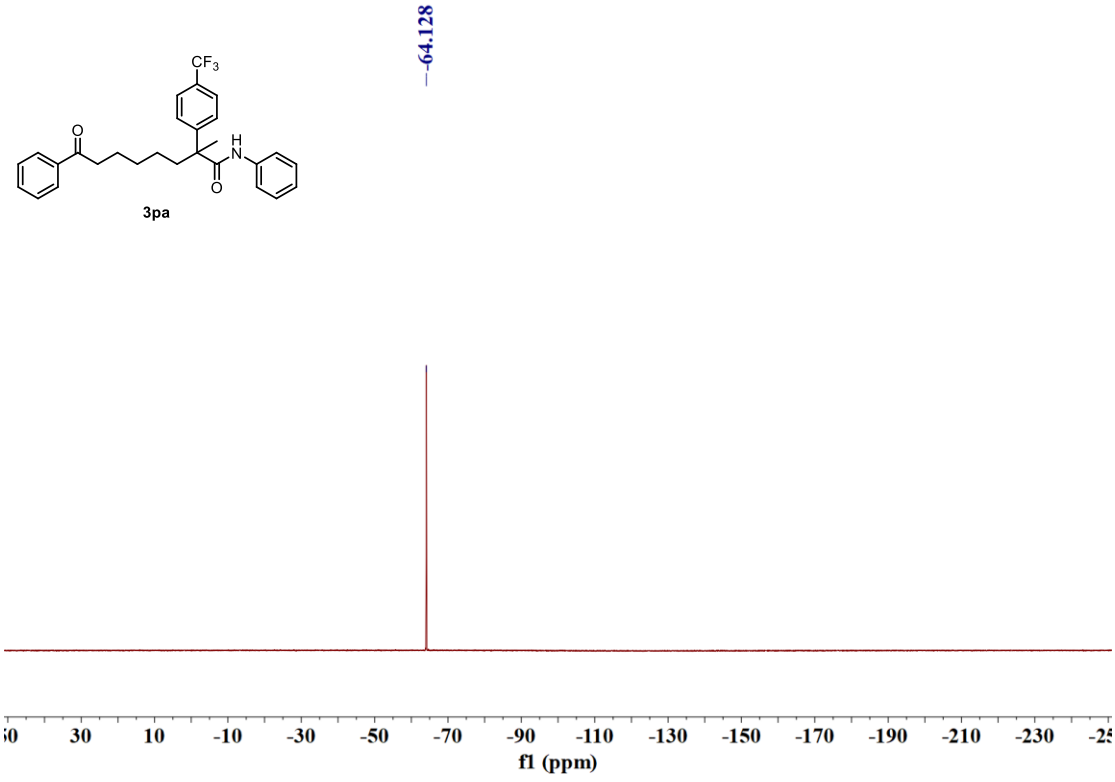
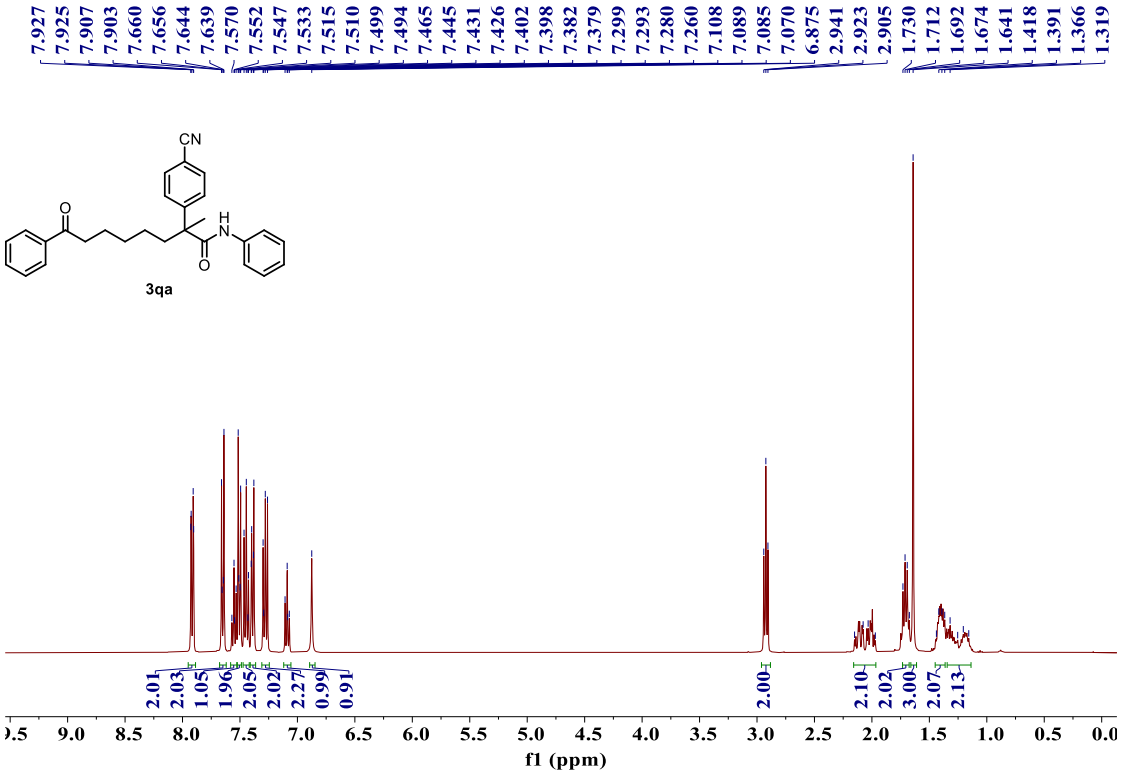
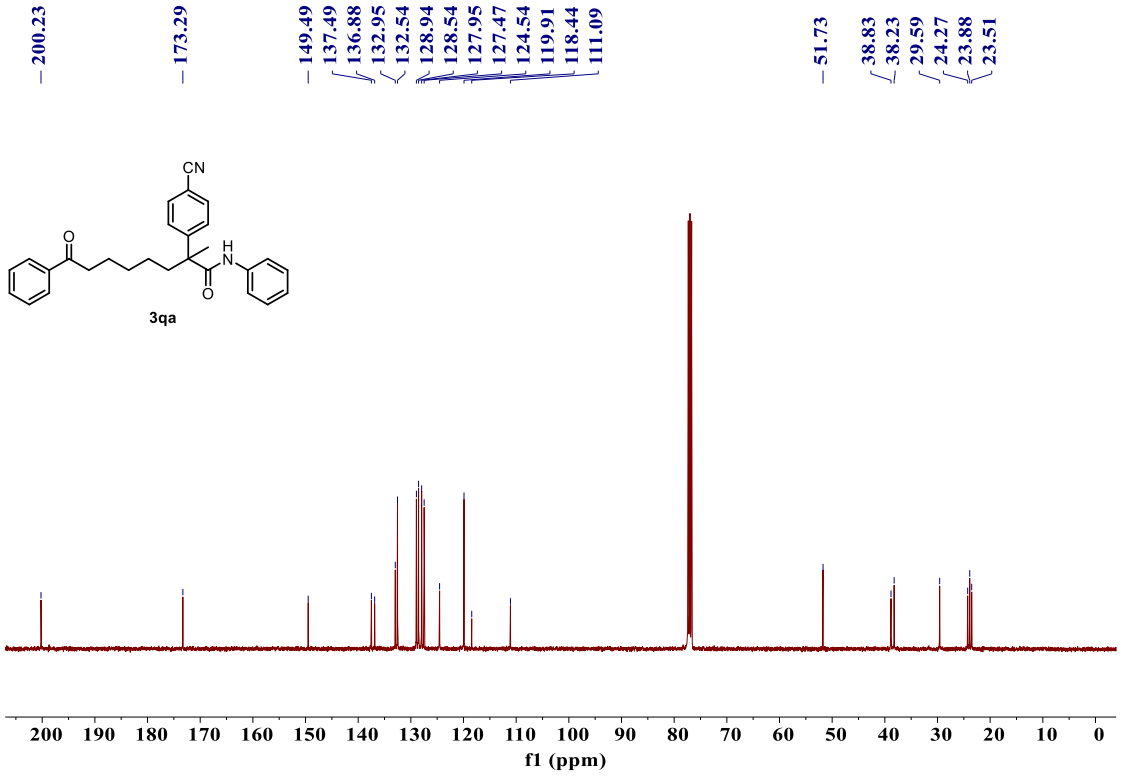
128.499
128.004
127.943
125.768
125.727
125.688
125.650
125.278
124.396
122.574
119.983
119.913
51.479
38.811
38.252
29.605
24.214
23.910
23.666
CF3
O
H
N
O
3pa
125.768
125.727
125.688
125.650
125.278
124.396
122.574
126.5
125.5
124.5
123.5
122.5
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

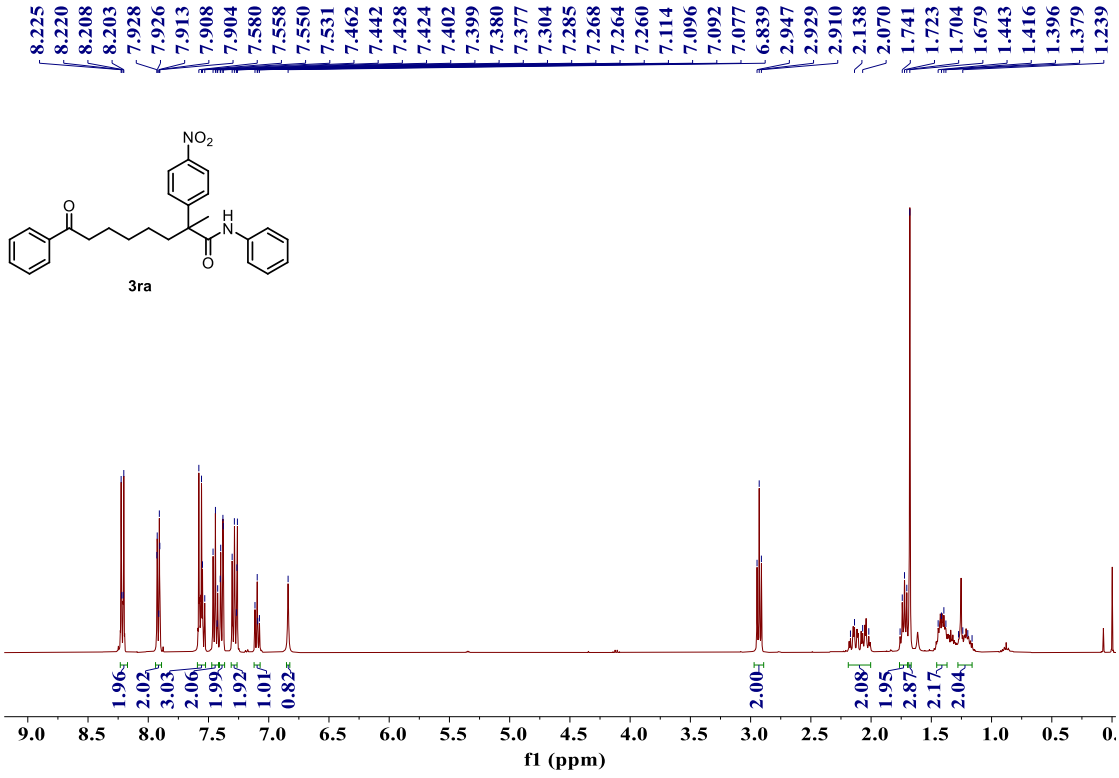

-64.128
CF3
O
H
N
O
3pa
30 10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

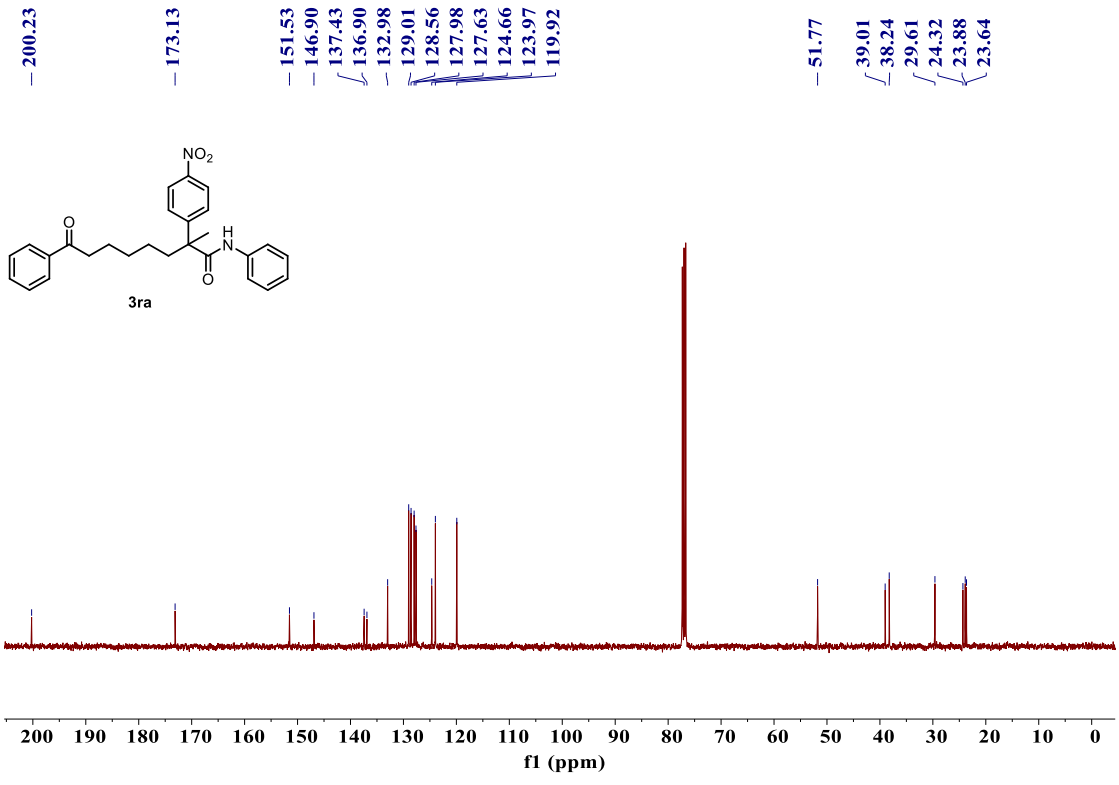
^{1}H and ^{13}C NMR spectra for product 3qa (Chloroform-*d*)



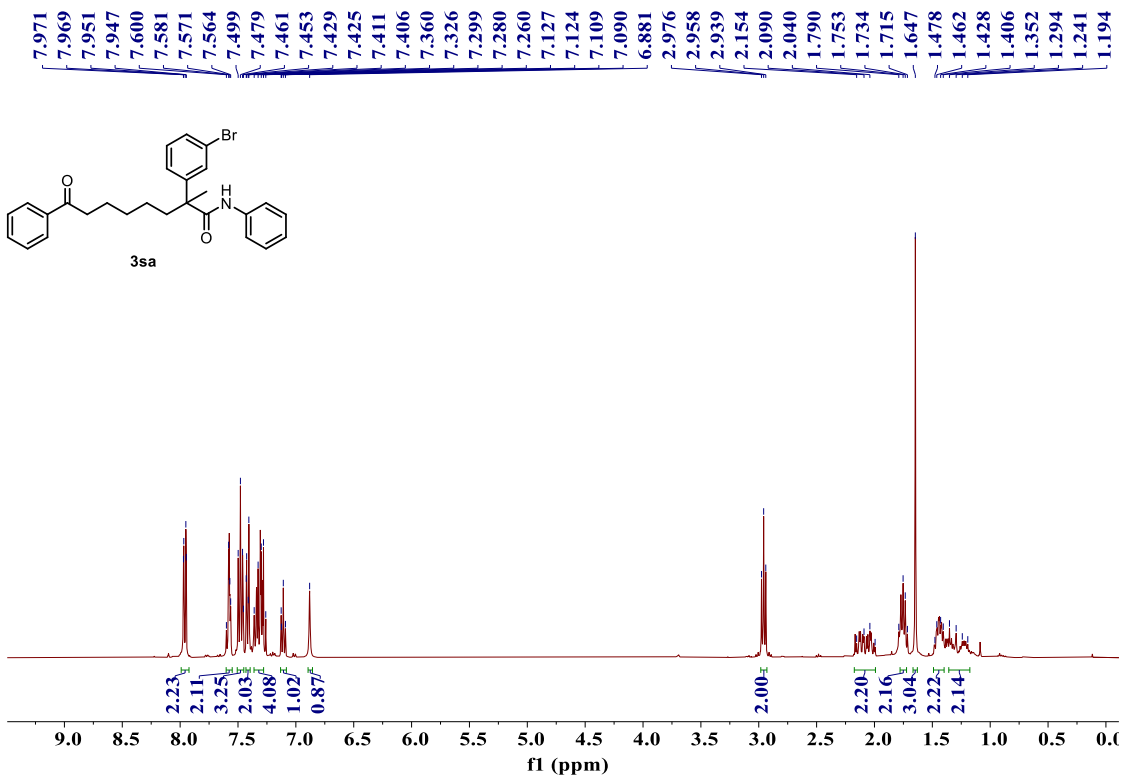


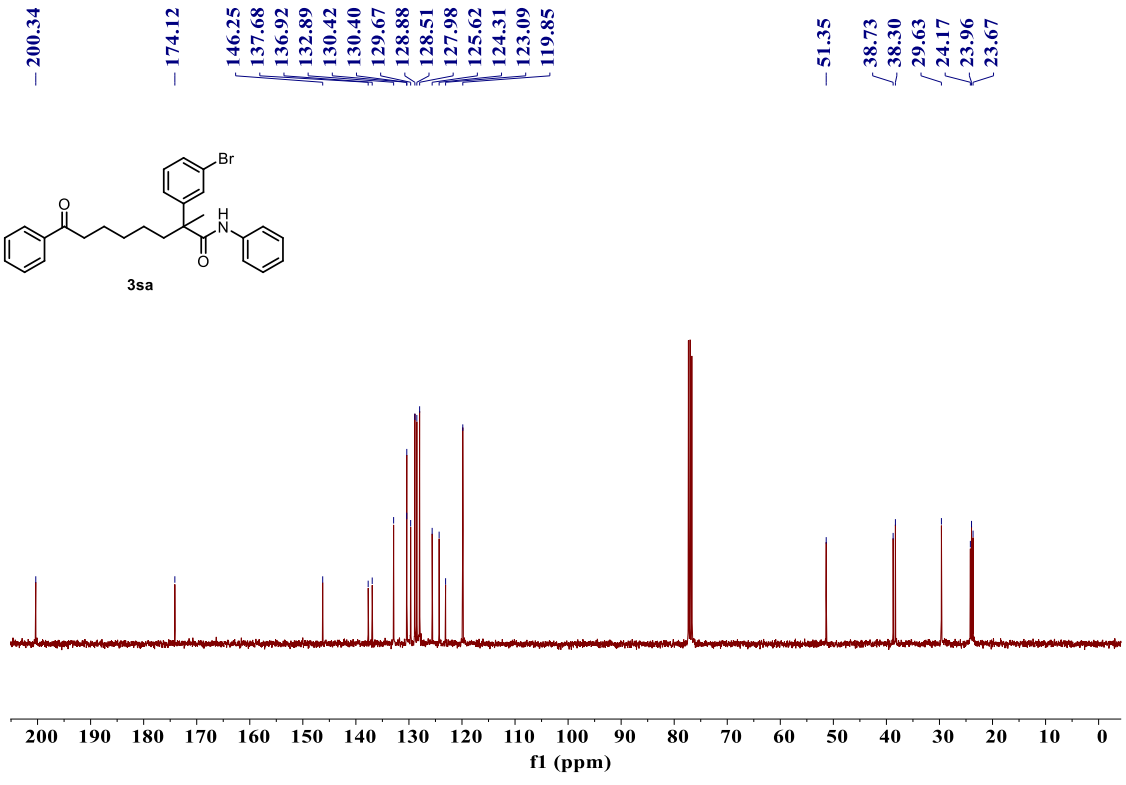
^{1}H and ^{13}C NMR spectra for product 3ra (Chloroform-*d*)



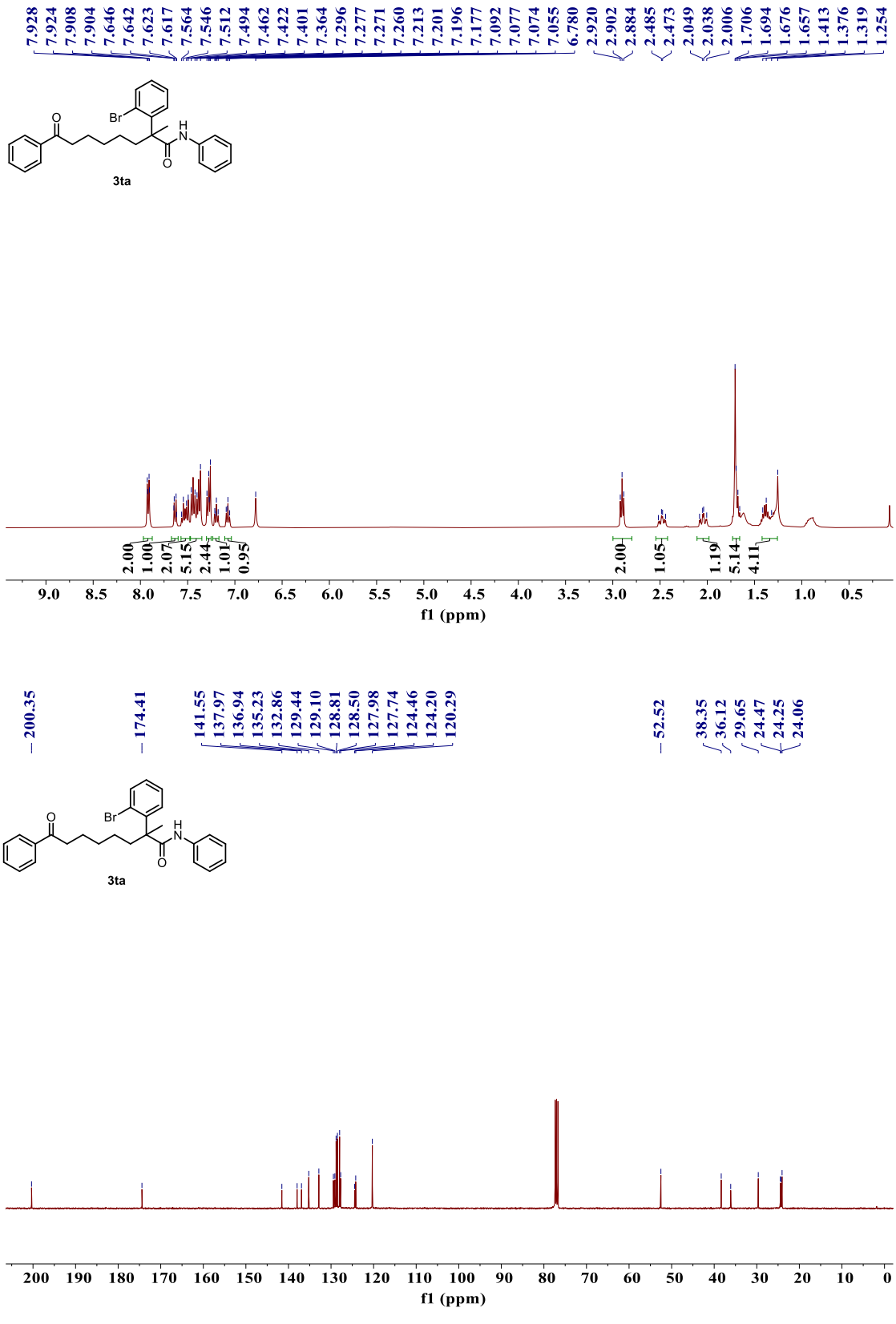


^{1}H and ^{13}C NMR spectra for product 3sa (Chloroform-*d*)

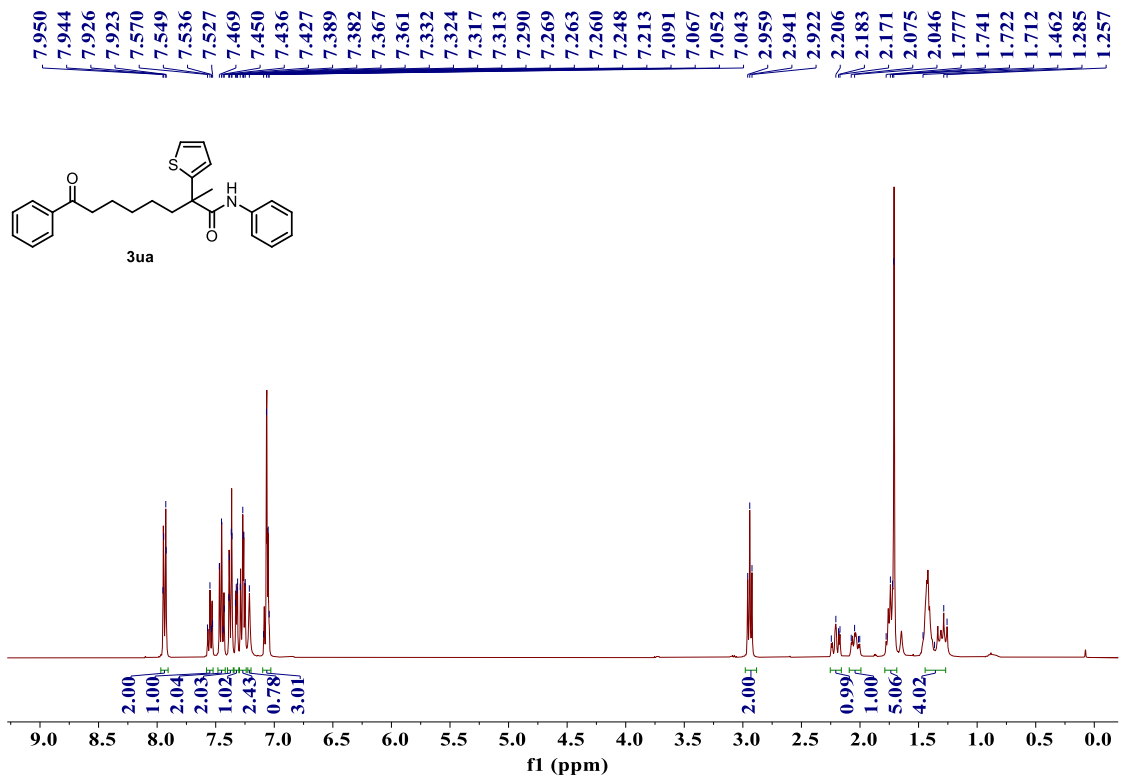


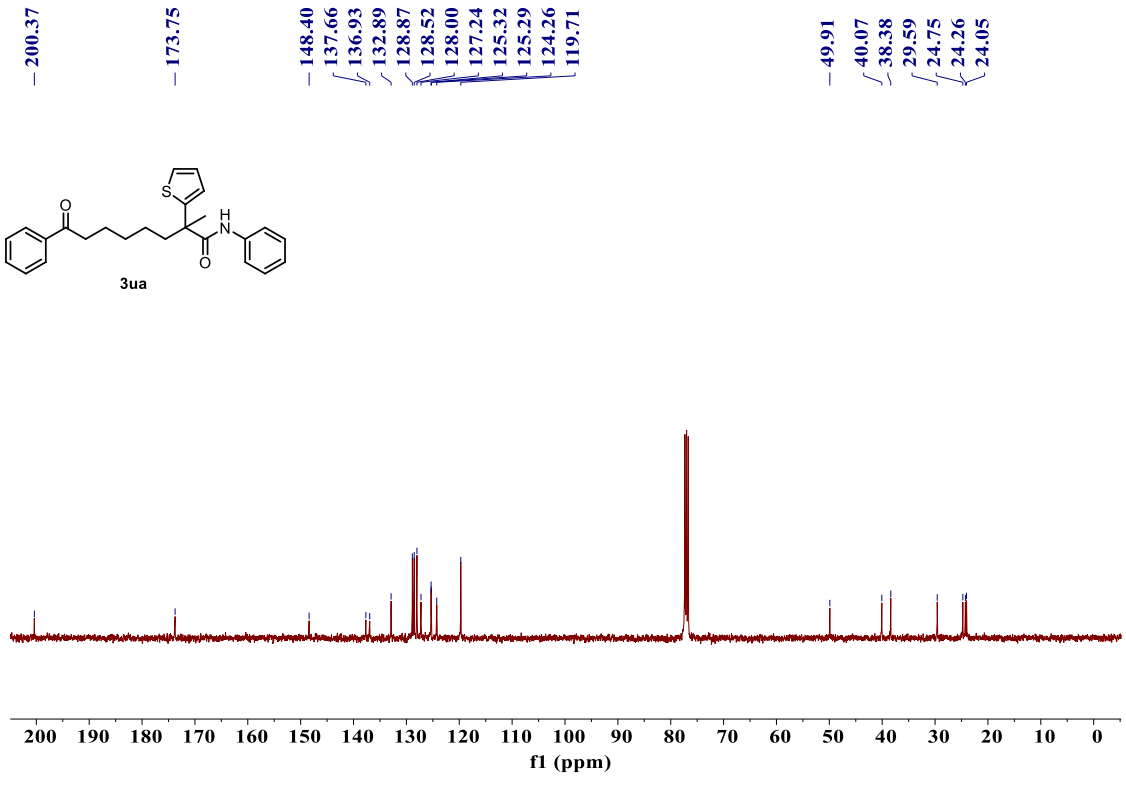


^{1}H and ^{13}C NMR spectra for product 3ta (Chloroform-*d*)

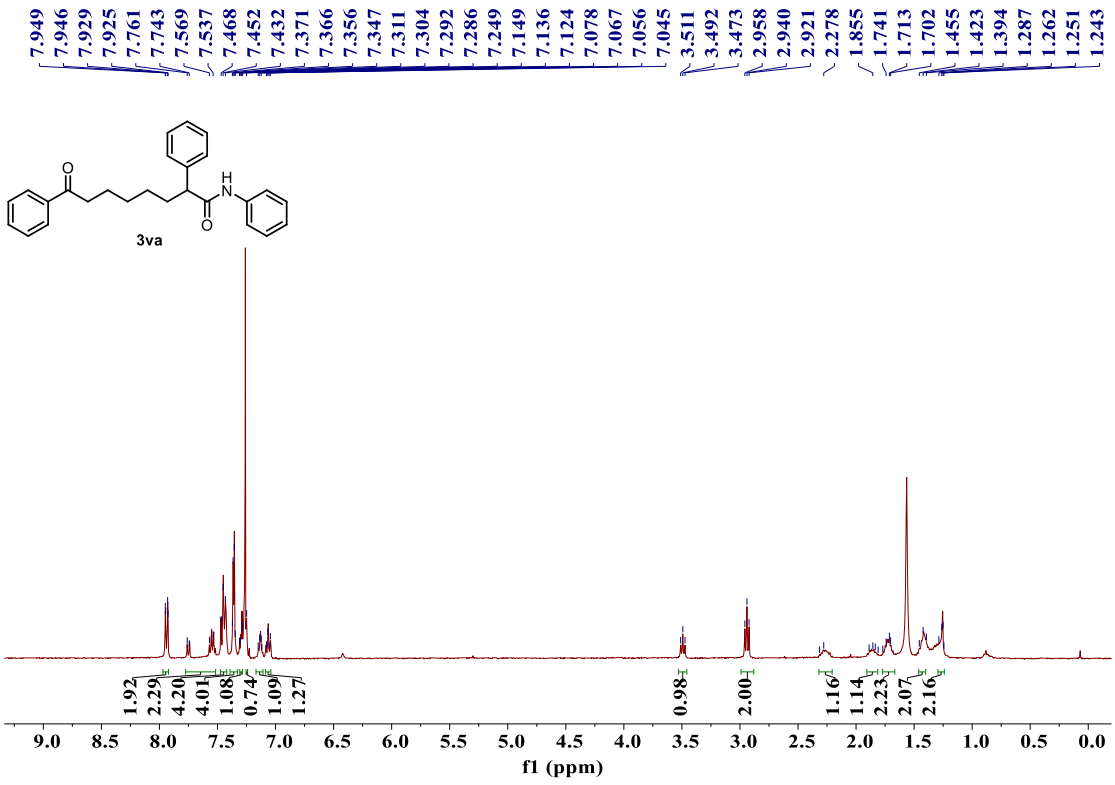


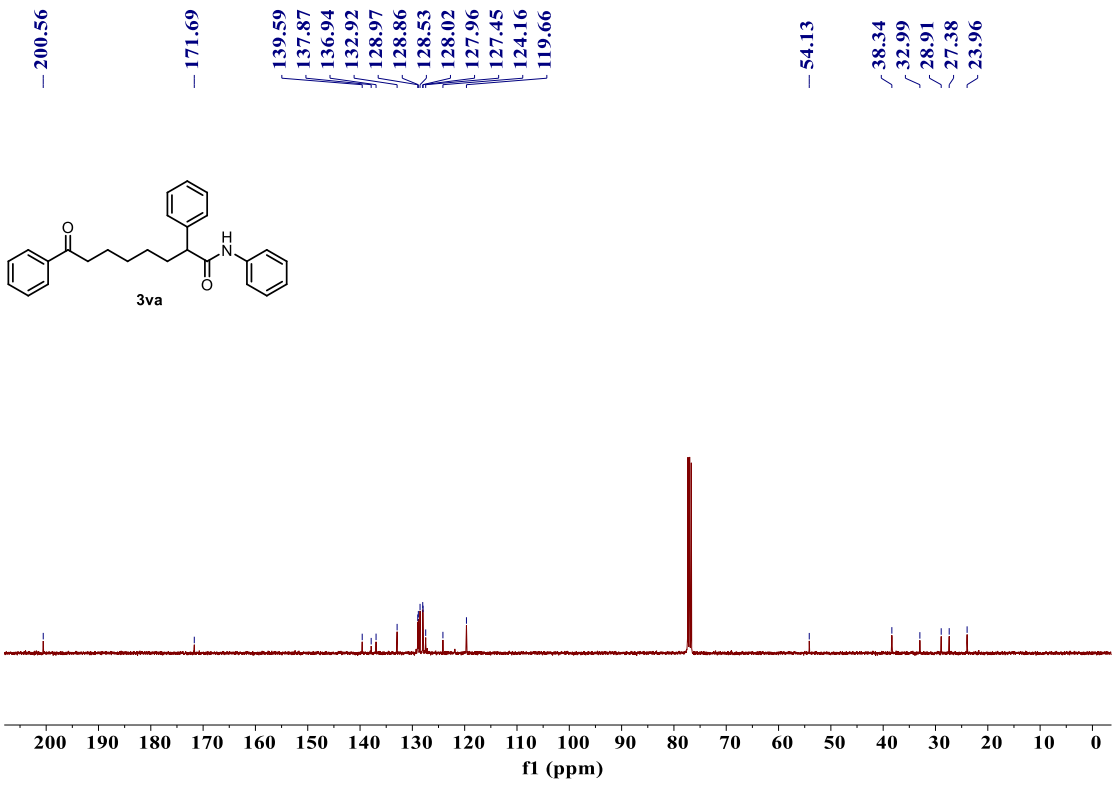
^{1}H and ^{13}C NMR spectra for product 3ua (Chloroform-*d*)



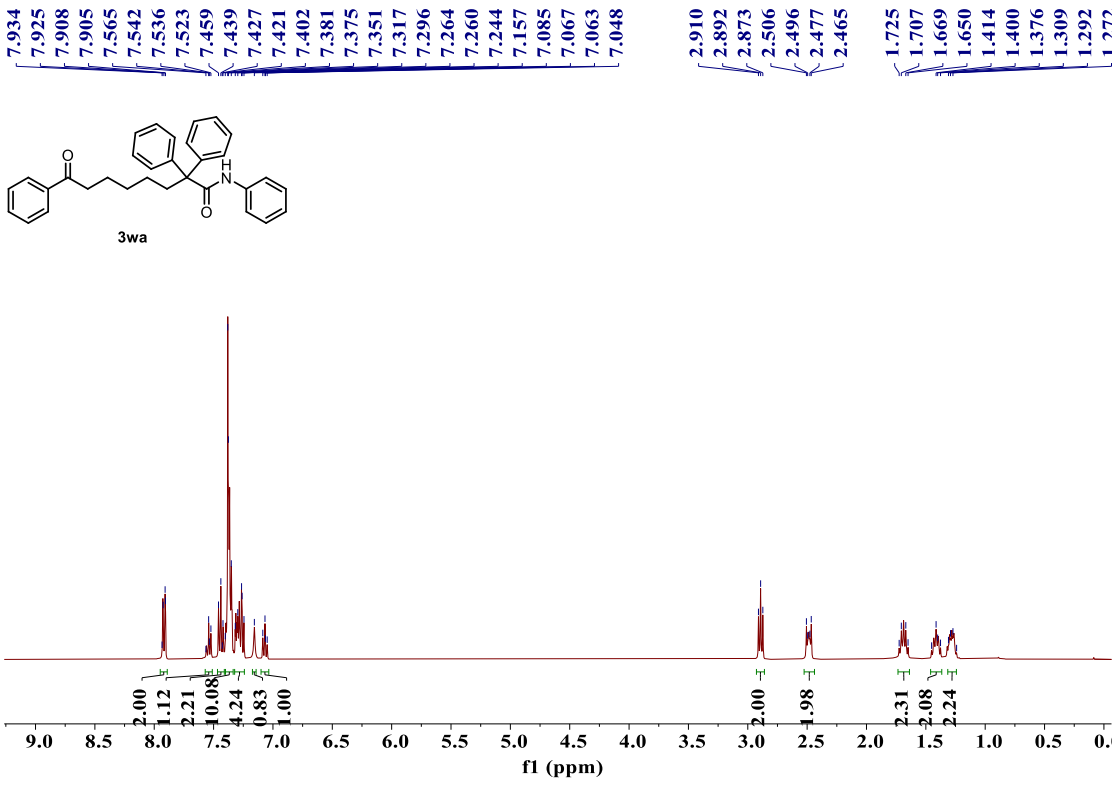


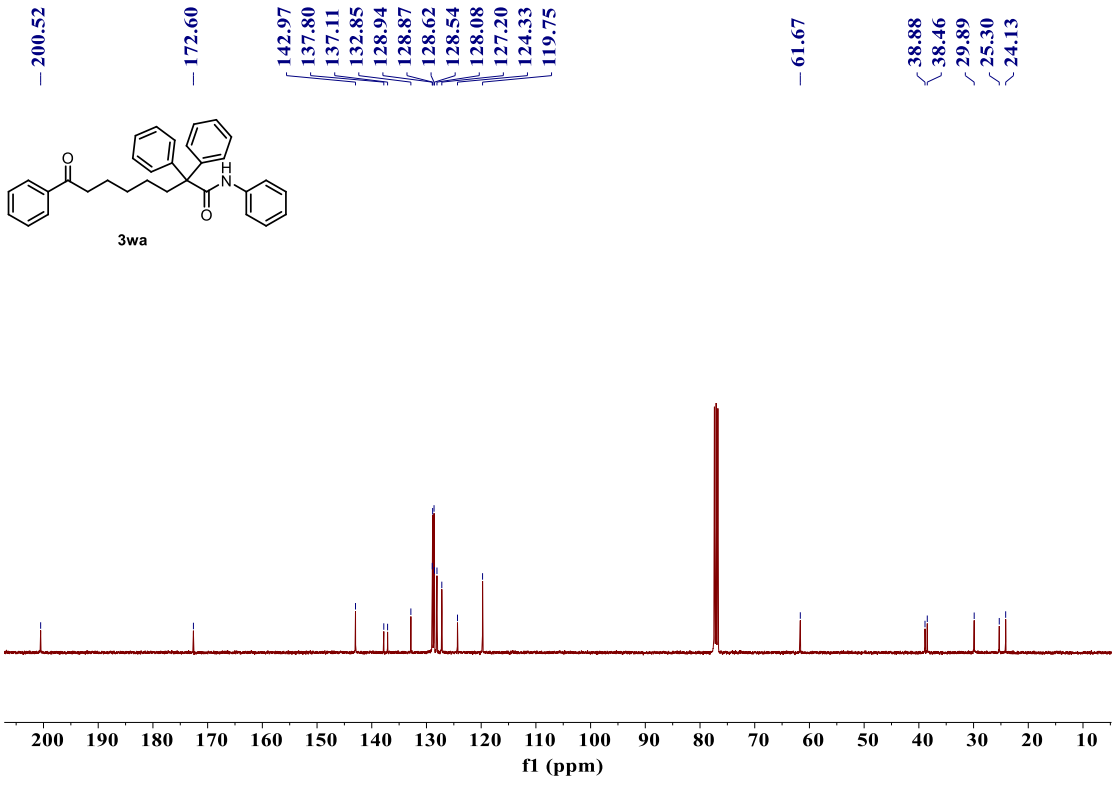
^{1}H and ^{13}C NMR spectra for product 3va (Chloroform-*d*)



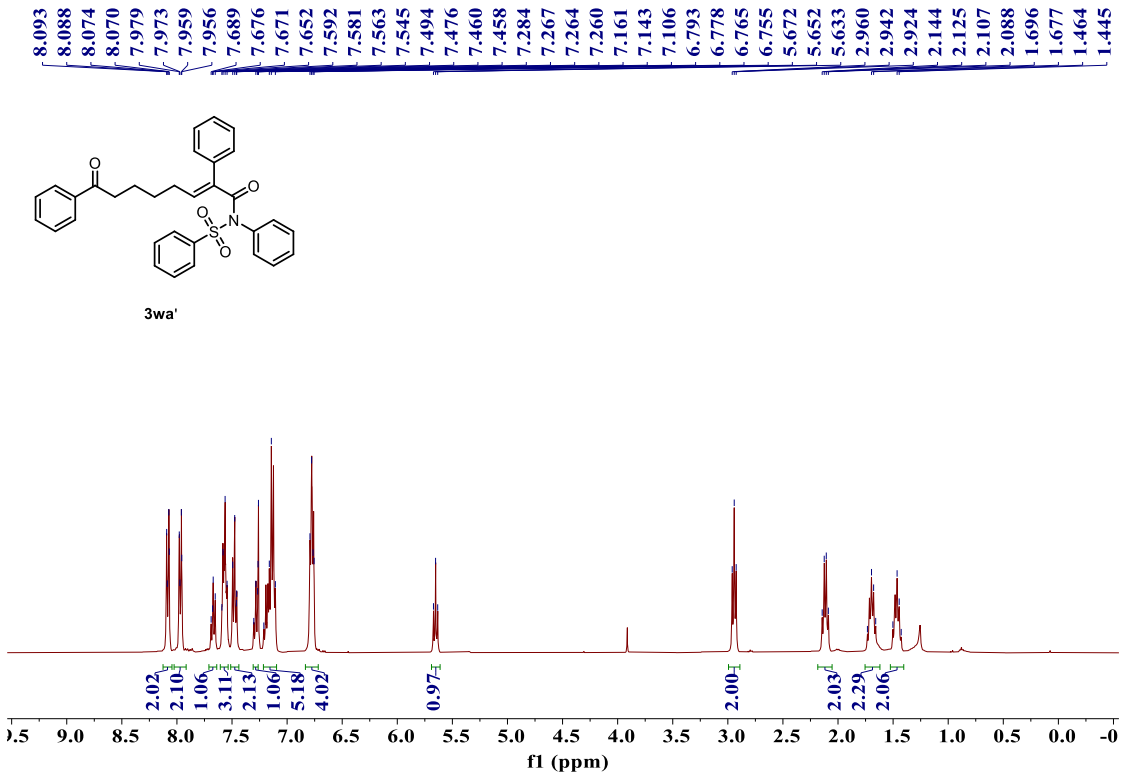


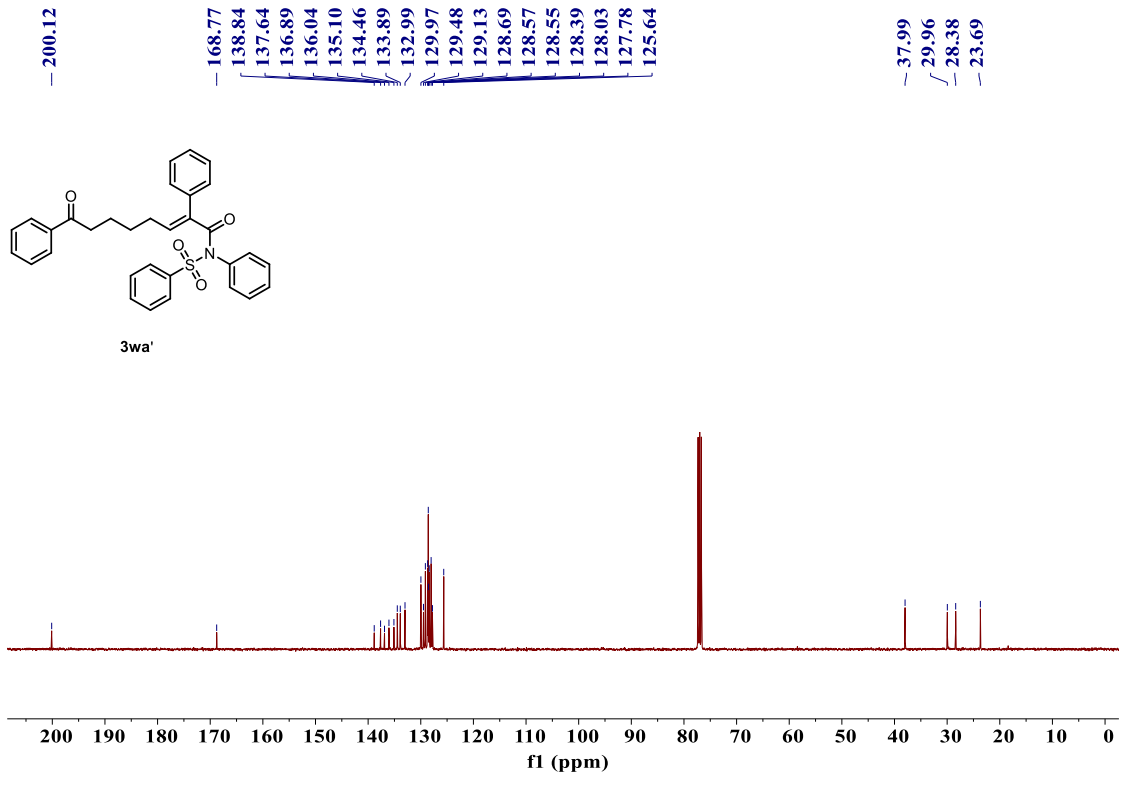
^{1}H and ^{13}C NMR spectra for product 3wa (Chloroform-*d*)



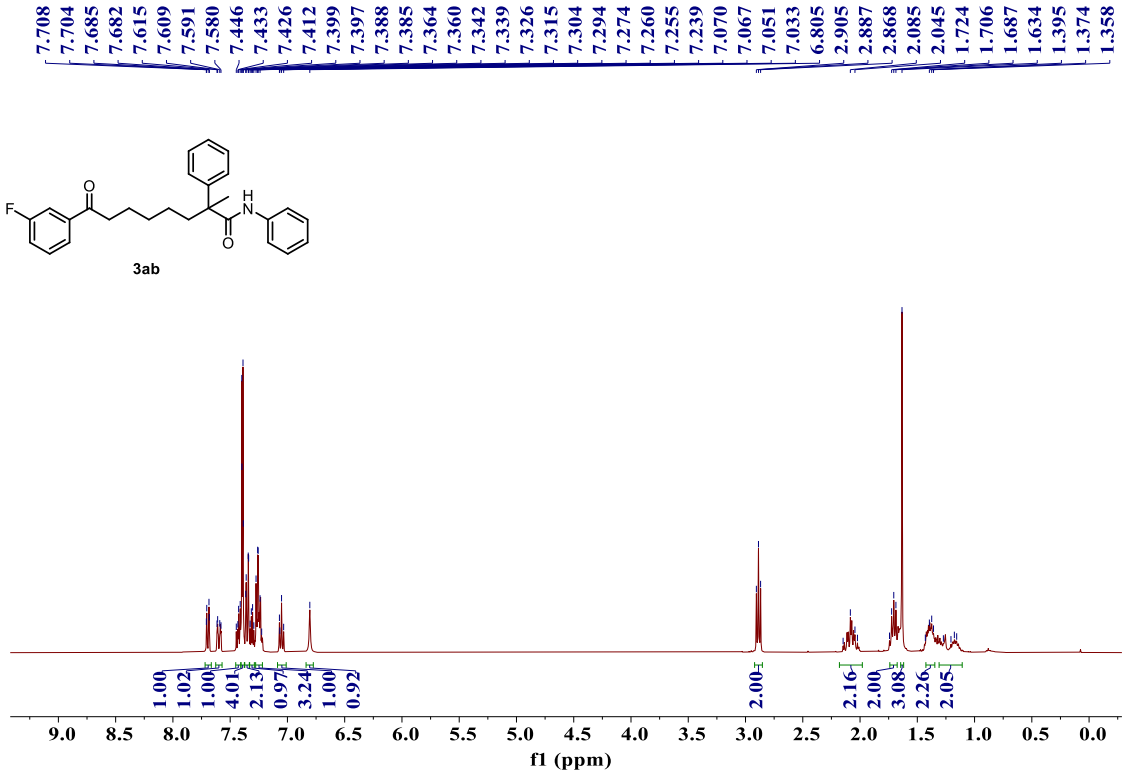


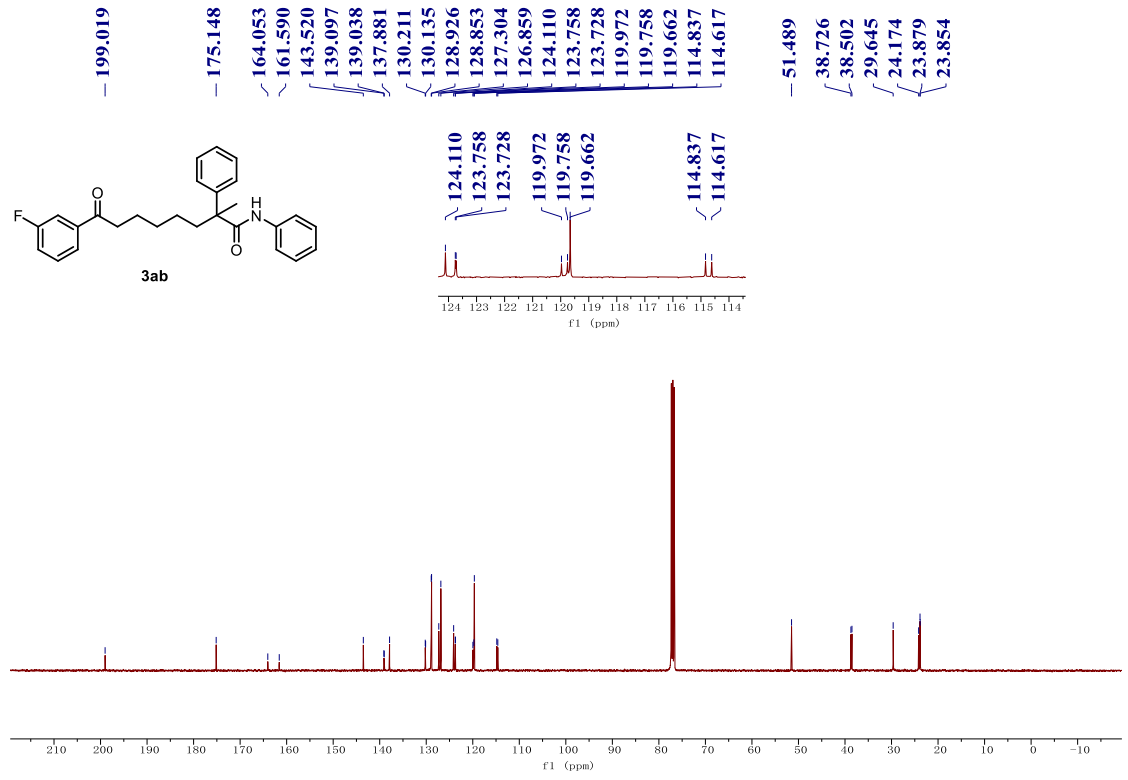
^{1}H and ^{13}C NMR spectra for product 3wa' (Chloroform-*d*)

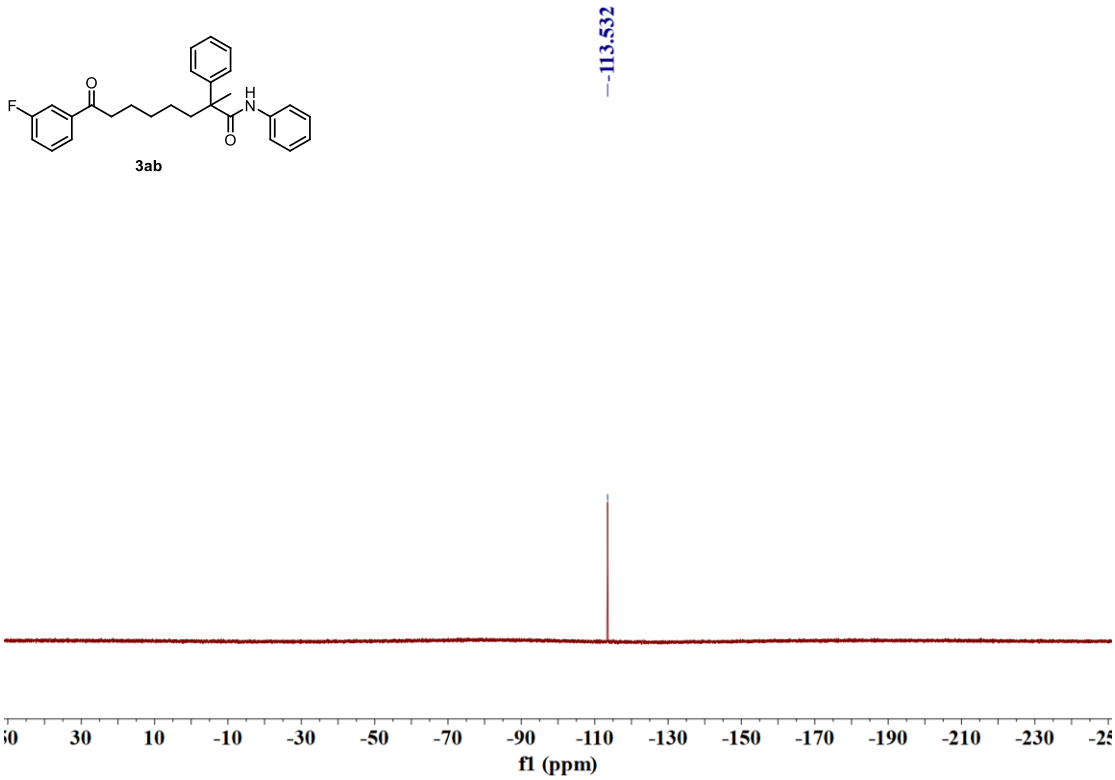




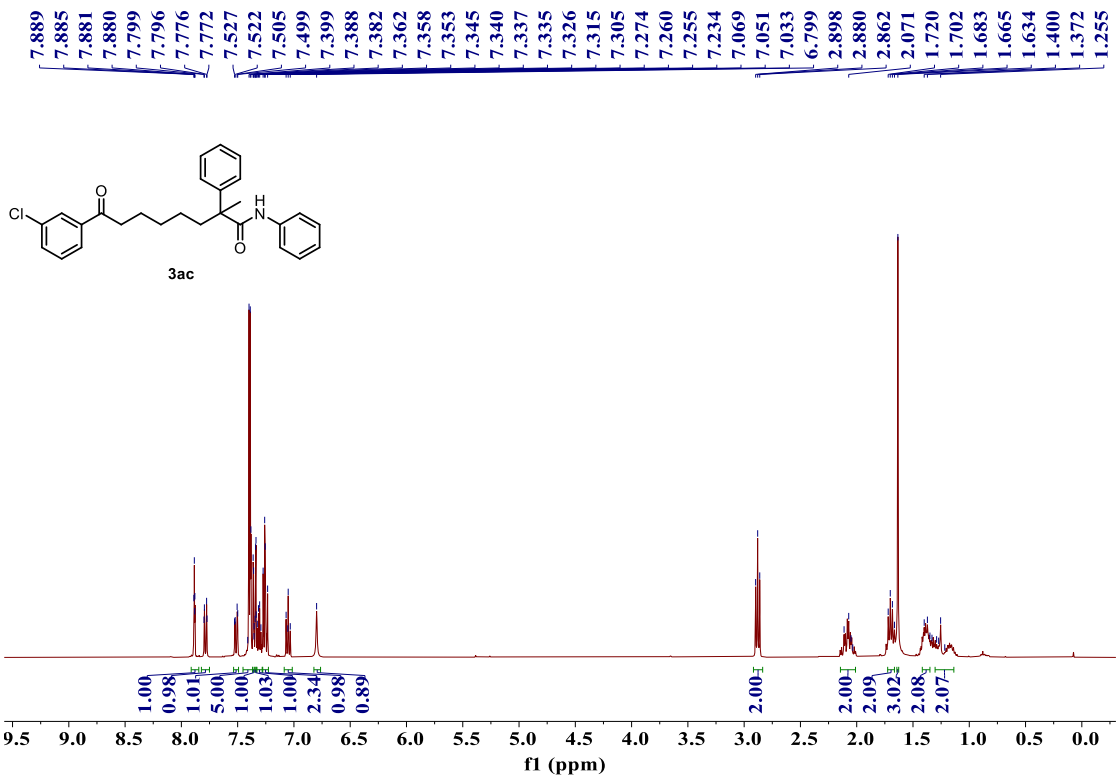
^{1}H,^{13}C and ^{19}F NMR spectra for product 3ab (Chloroform-*d*)

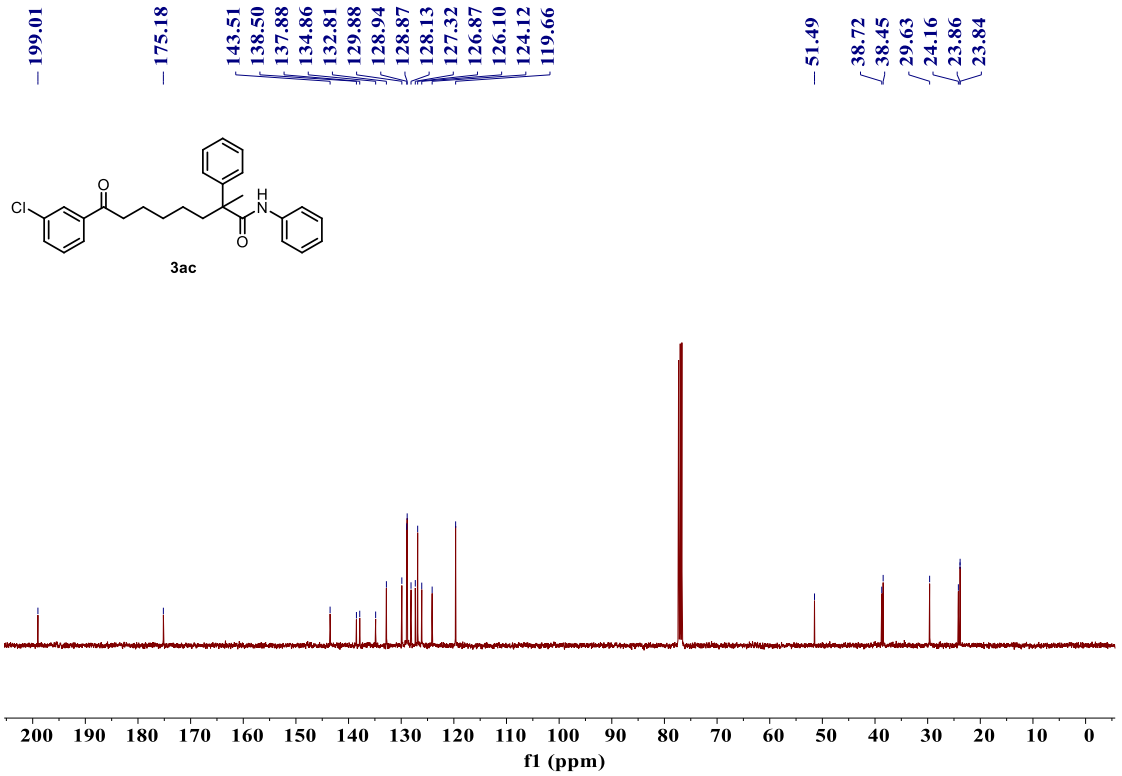




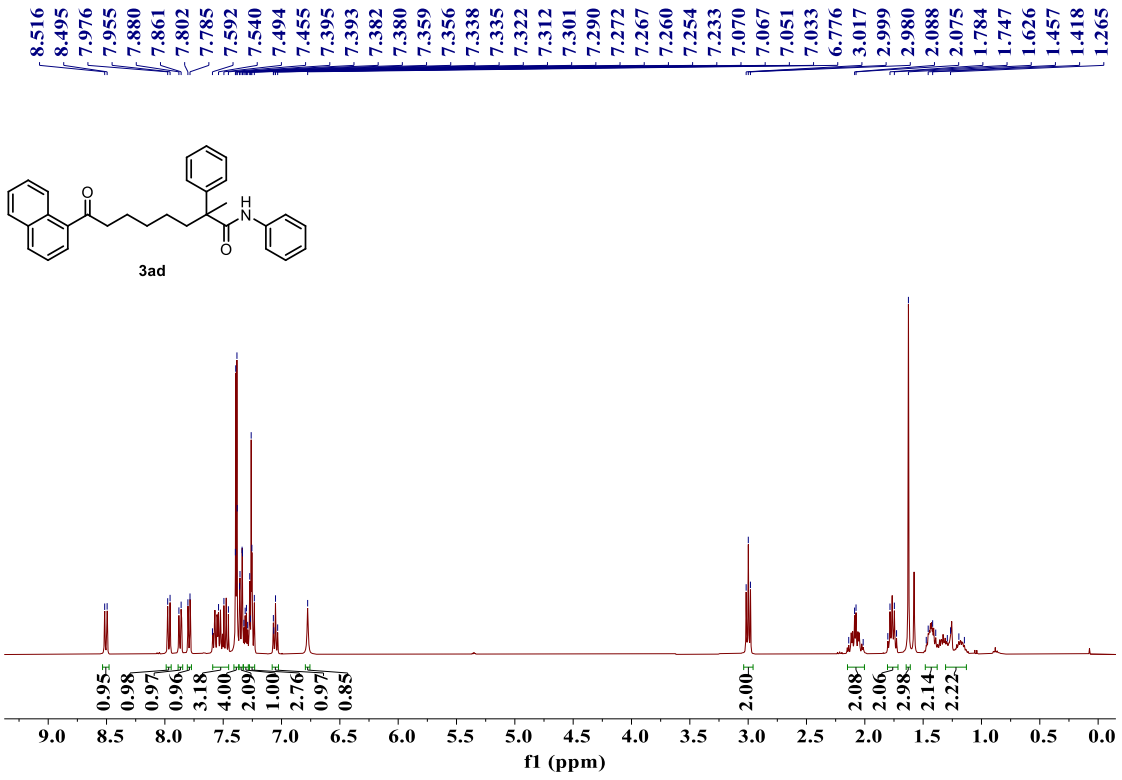


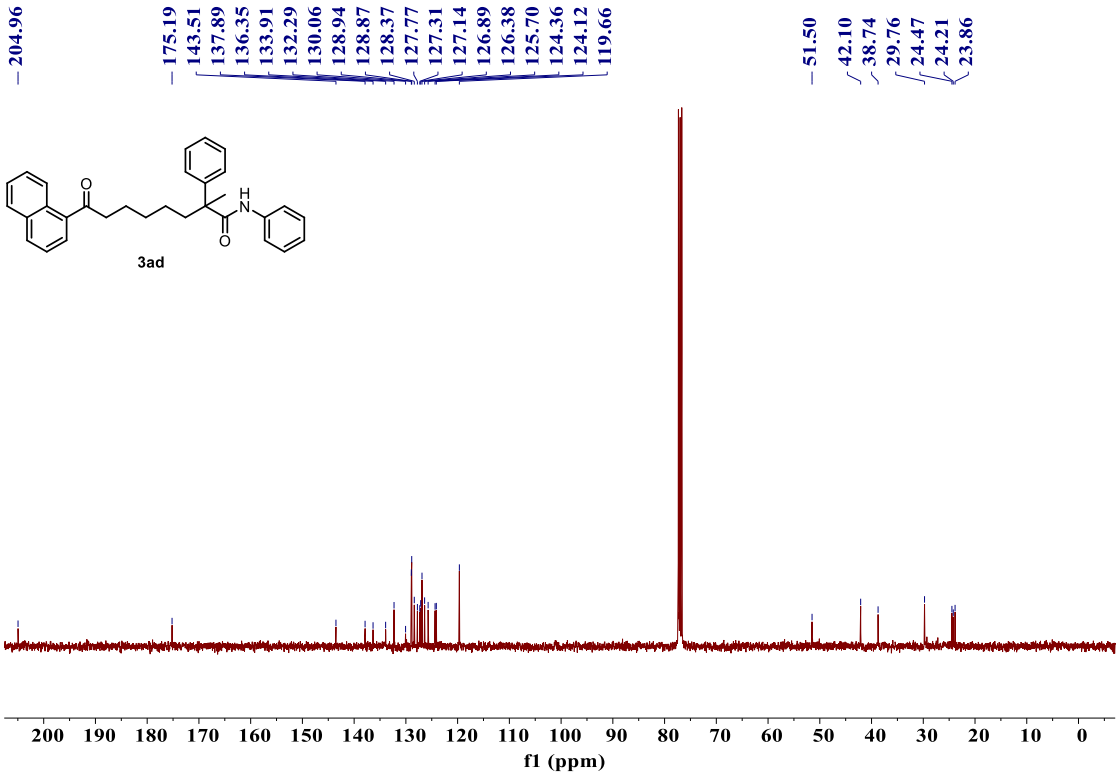
^{1}H and ^{13}C NMR spectra for product 3ac (Chloroform-*d*)



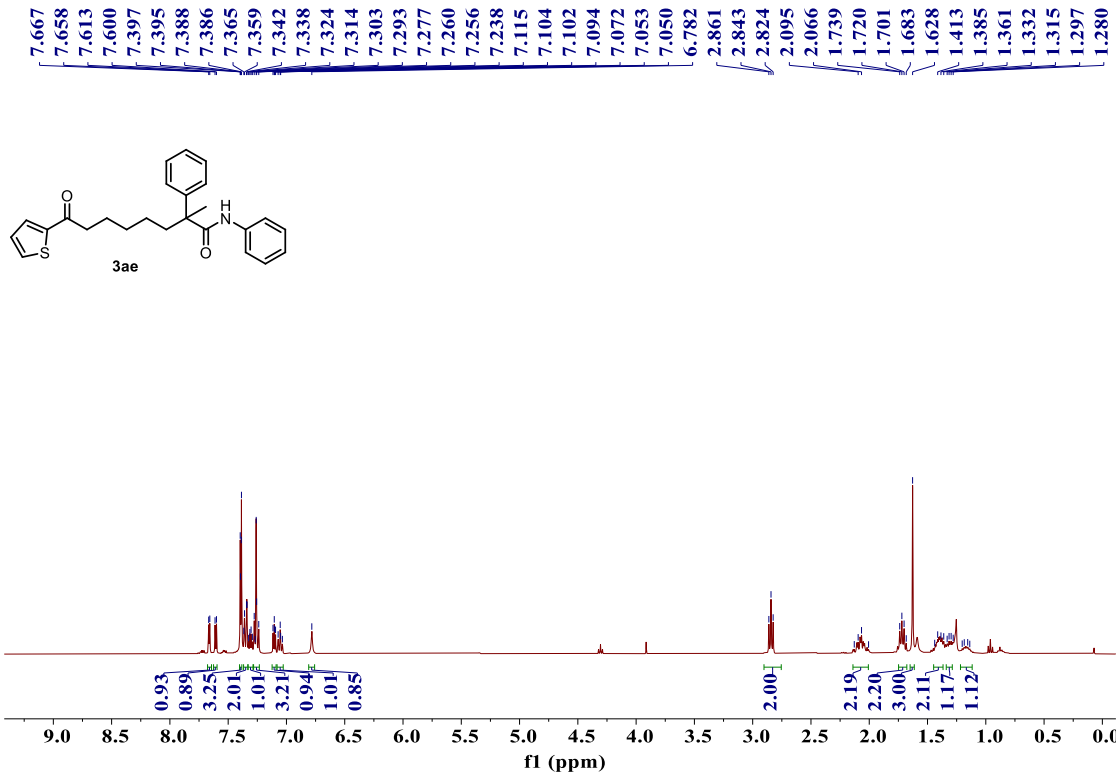


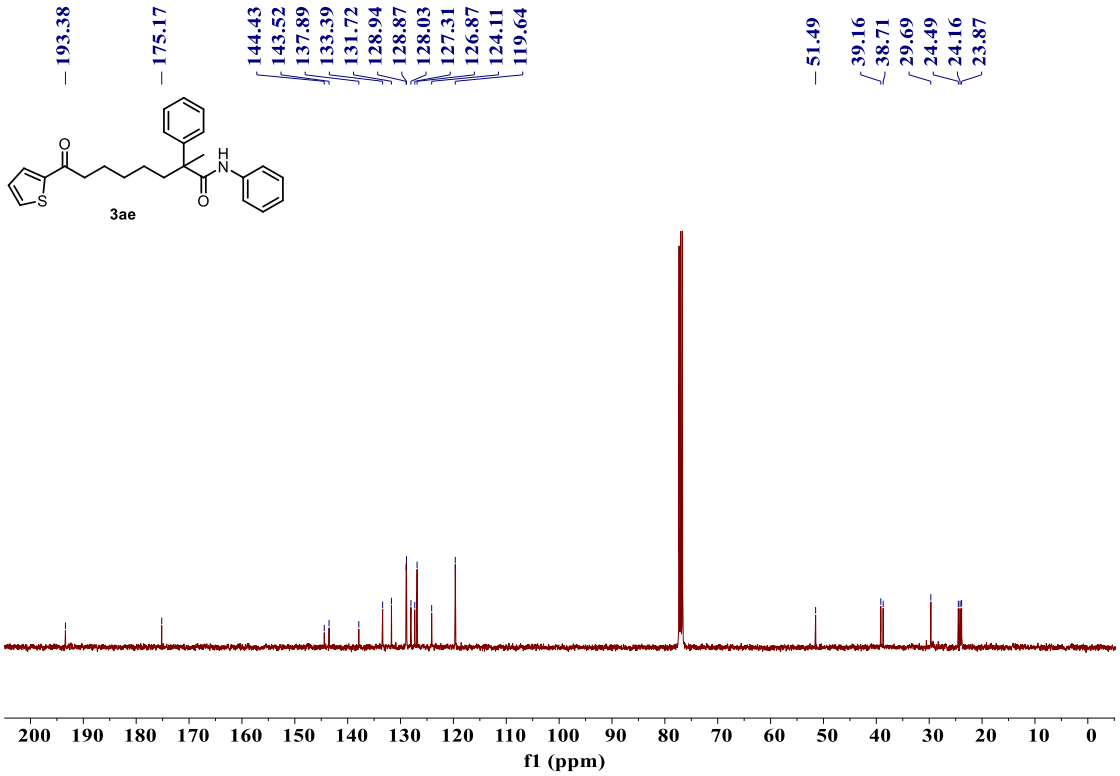
^{1}H and ^{13}C NMR spectra for product 3ad (Chloroform-*d*)



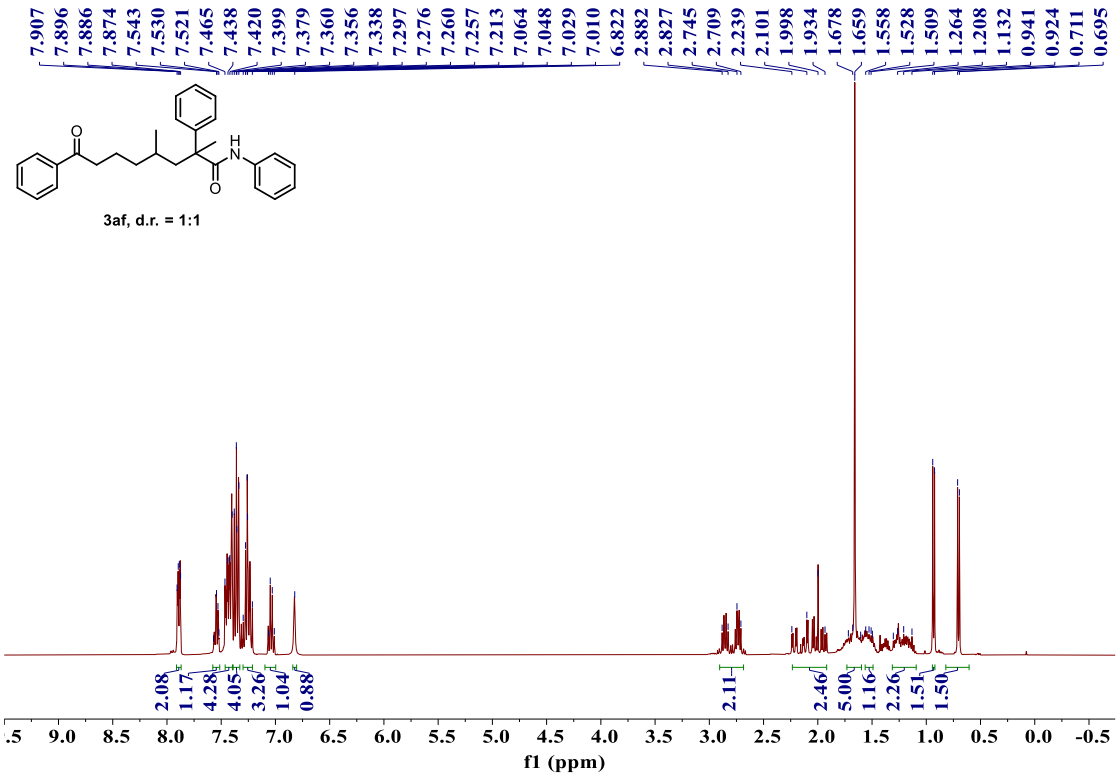


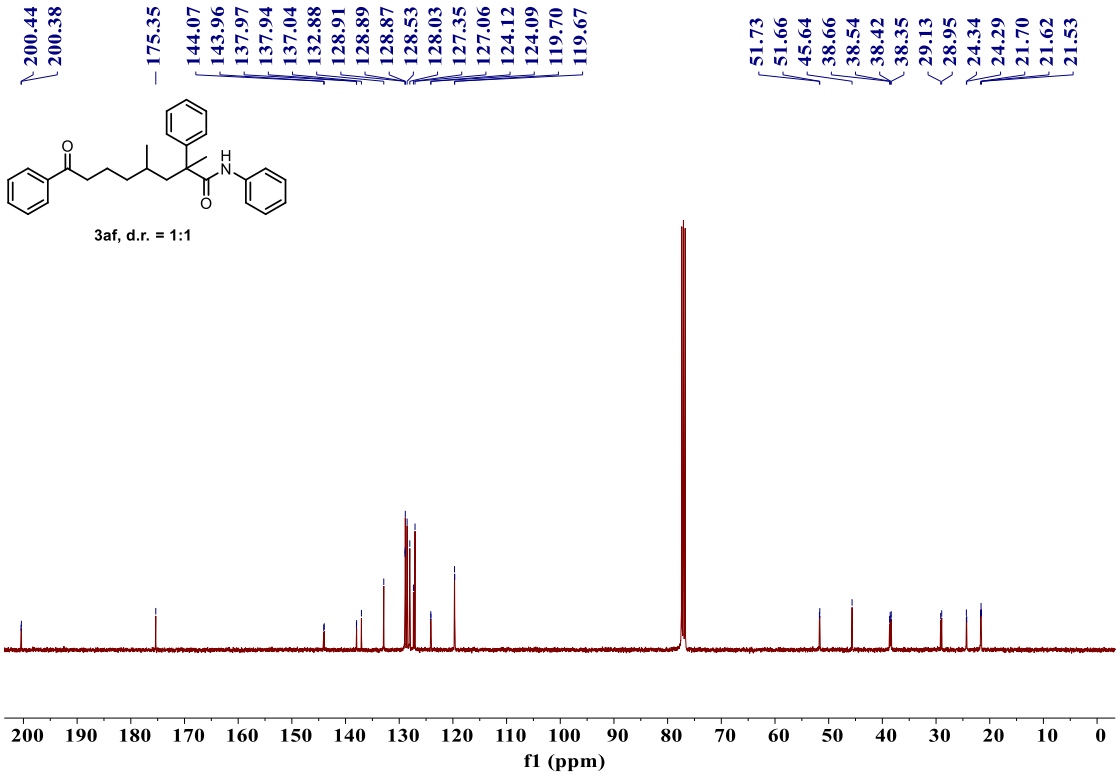
^{1}H and ^{13}C NMR spectra for product 3ae (Chloroform-*d*)



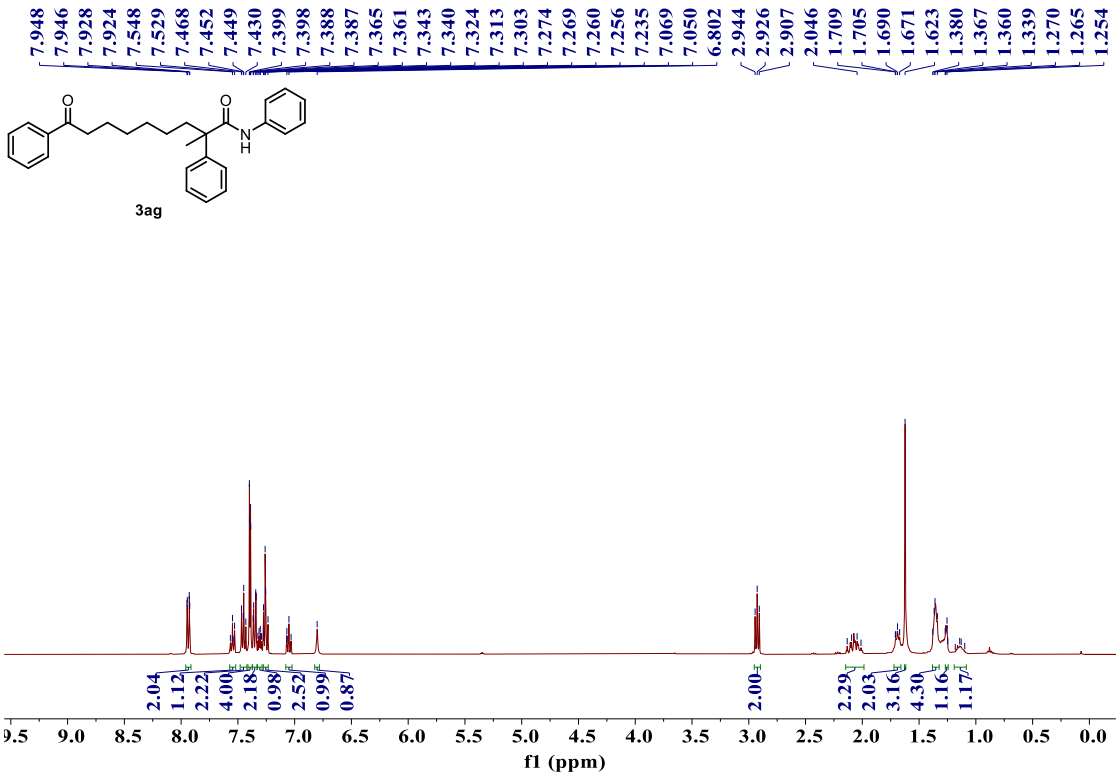


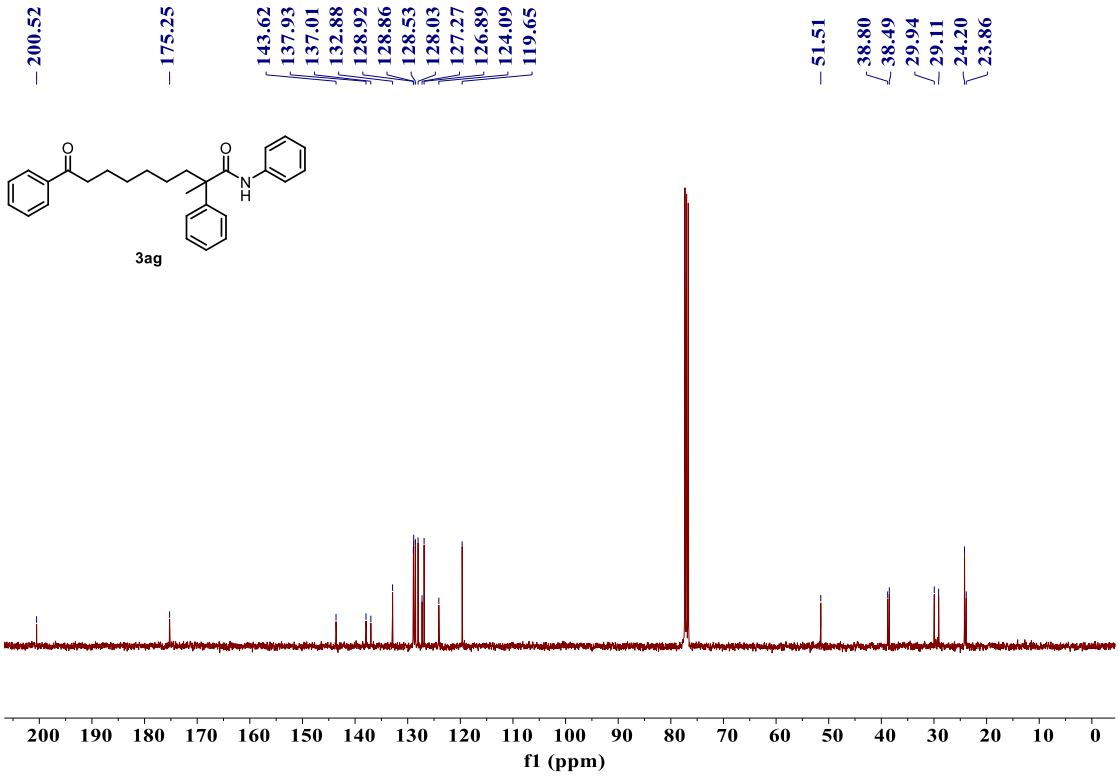
^{1}H and ^{13}C NMR spectra for product 3af (Chloroform-*d*)



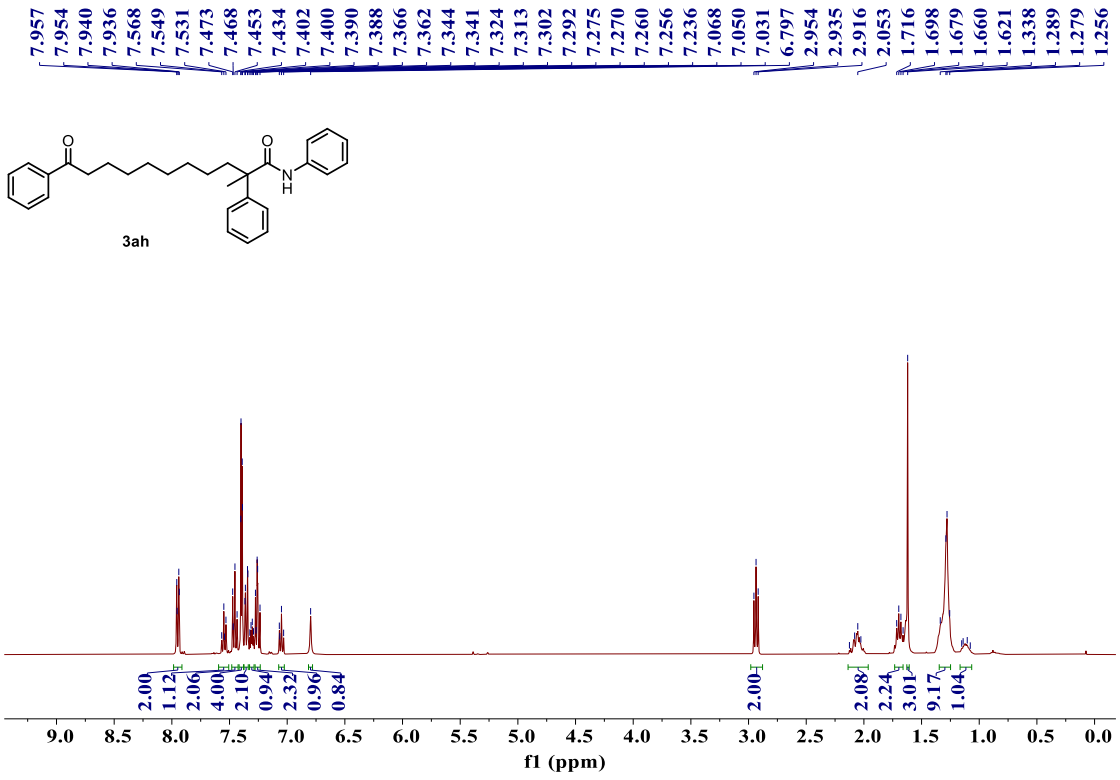


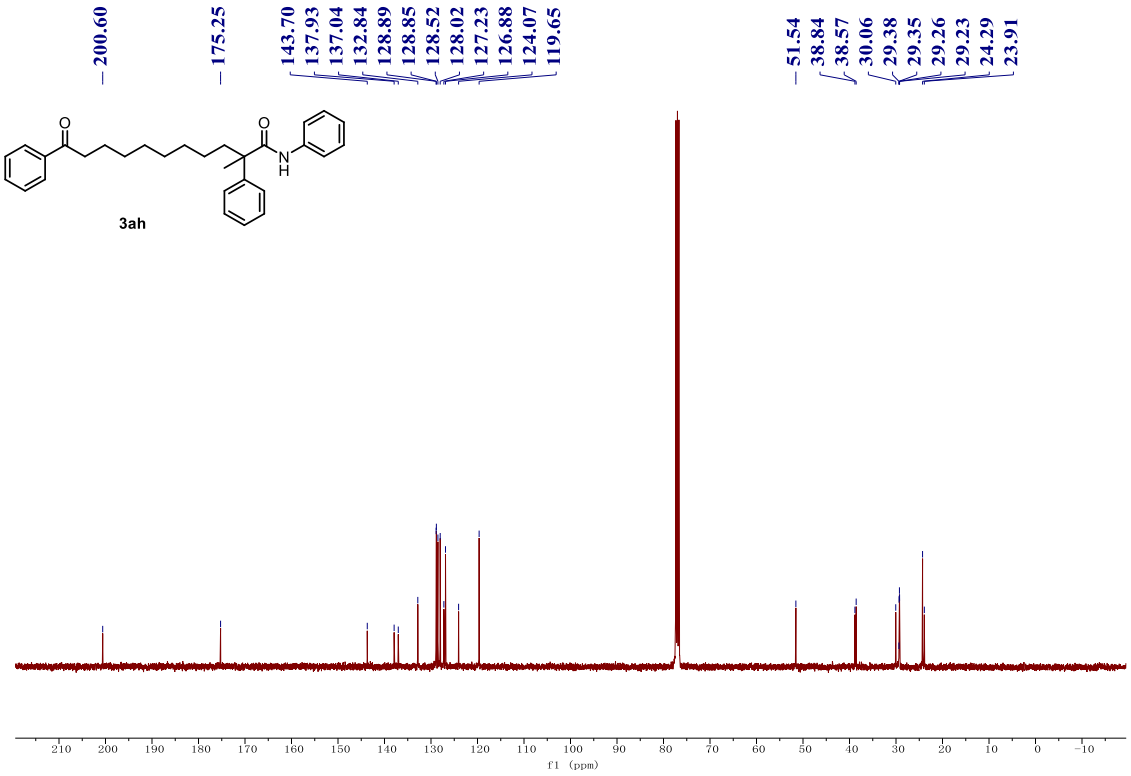
^{1}H and ^{13}C NMR spectra for product 3ag (Chloroform-*d*)



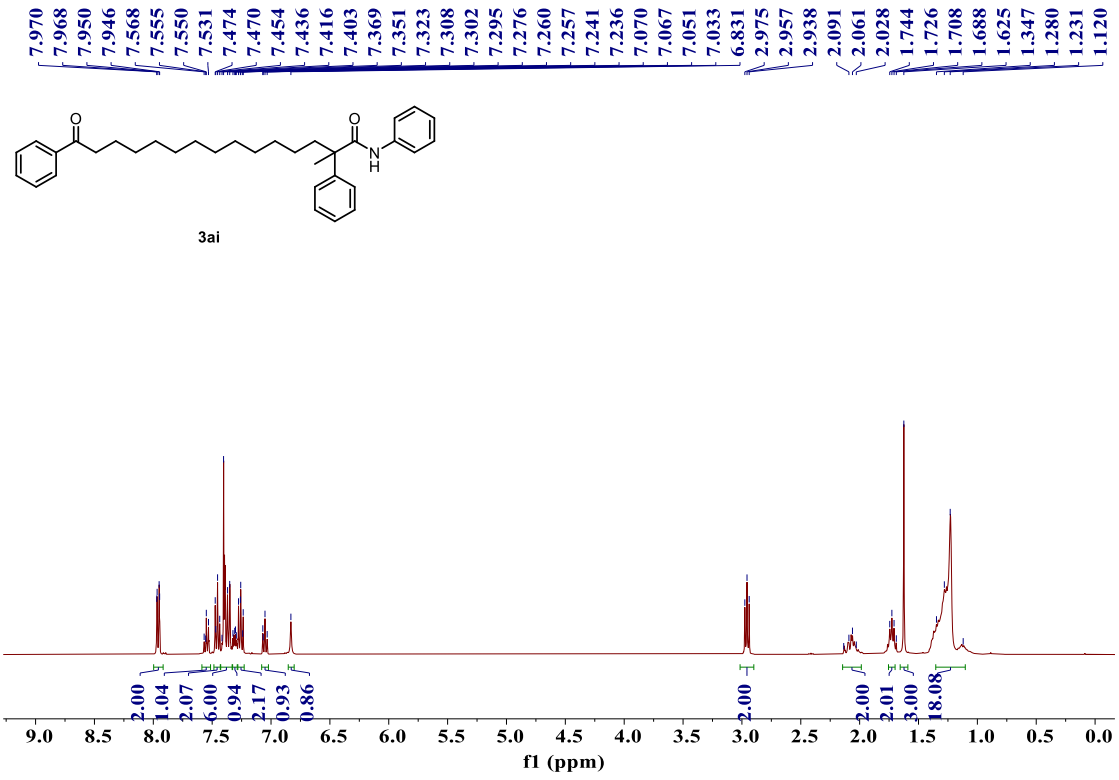


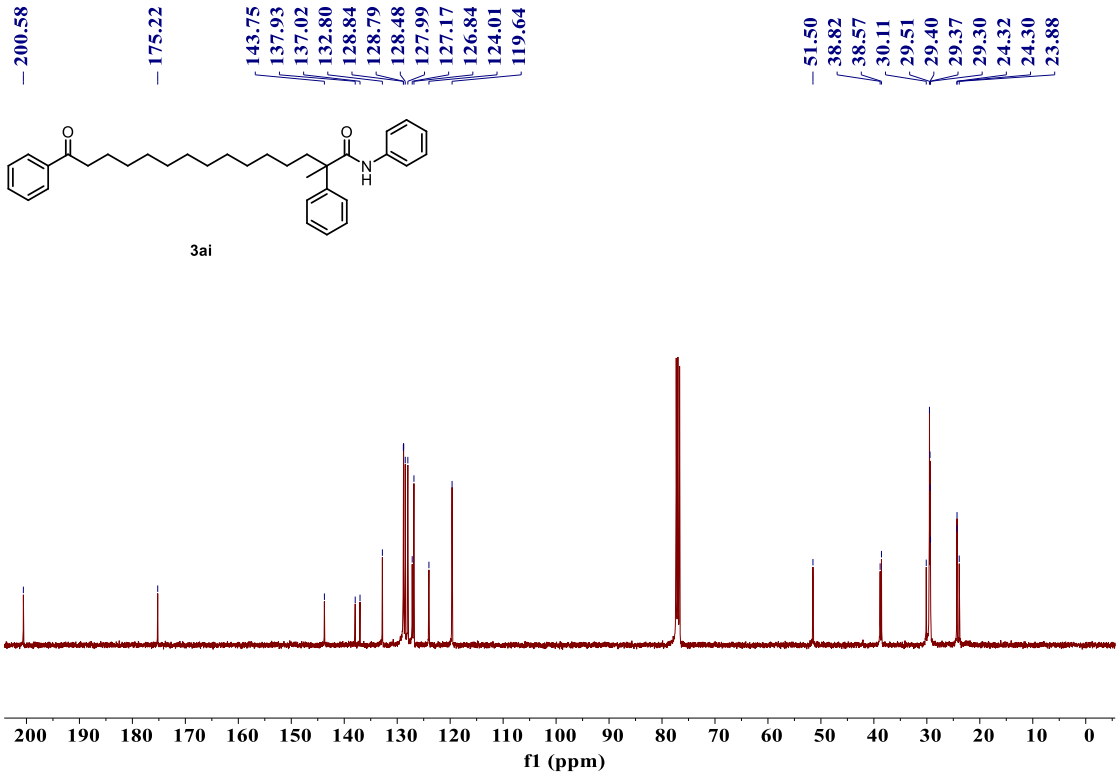
^{1}H and ^{13}C NMR spectra for product 3ah (Chloroform-*d*)



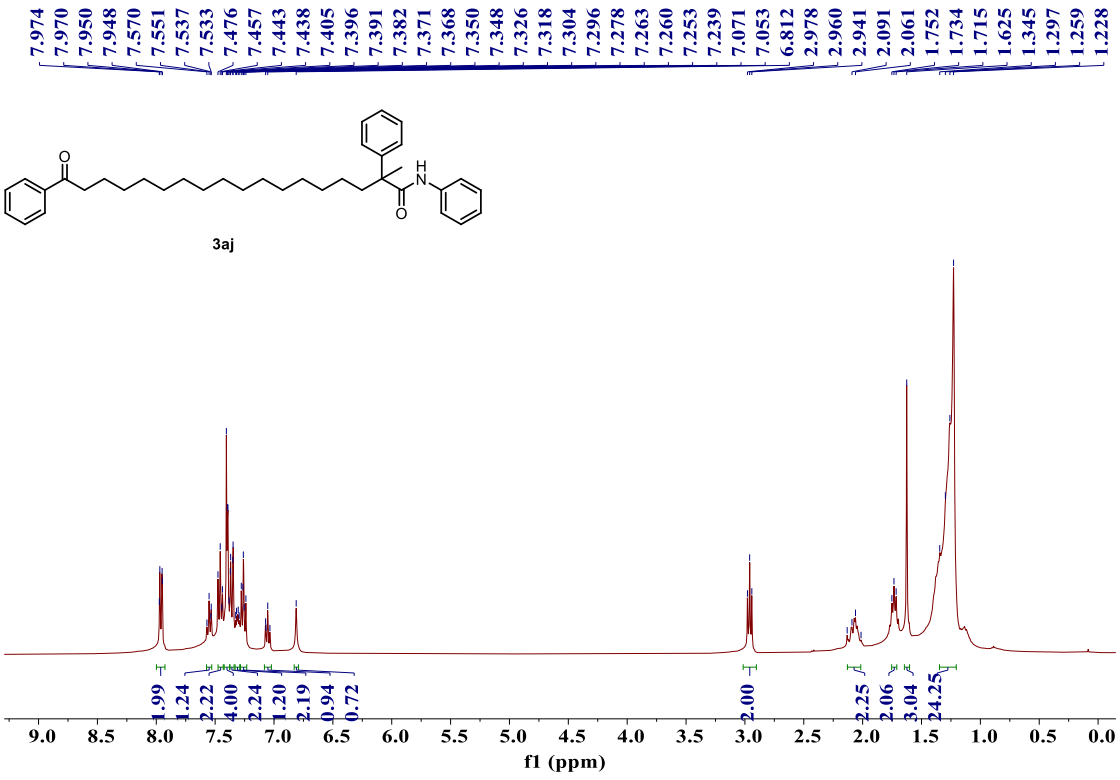


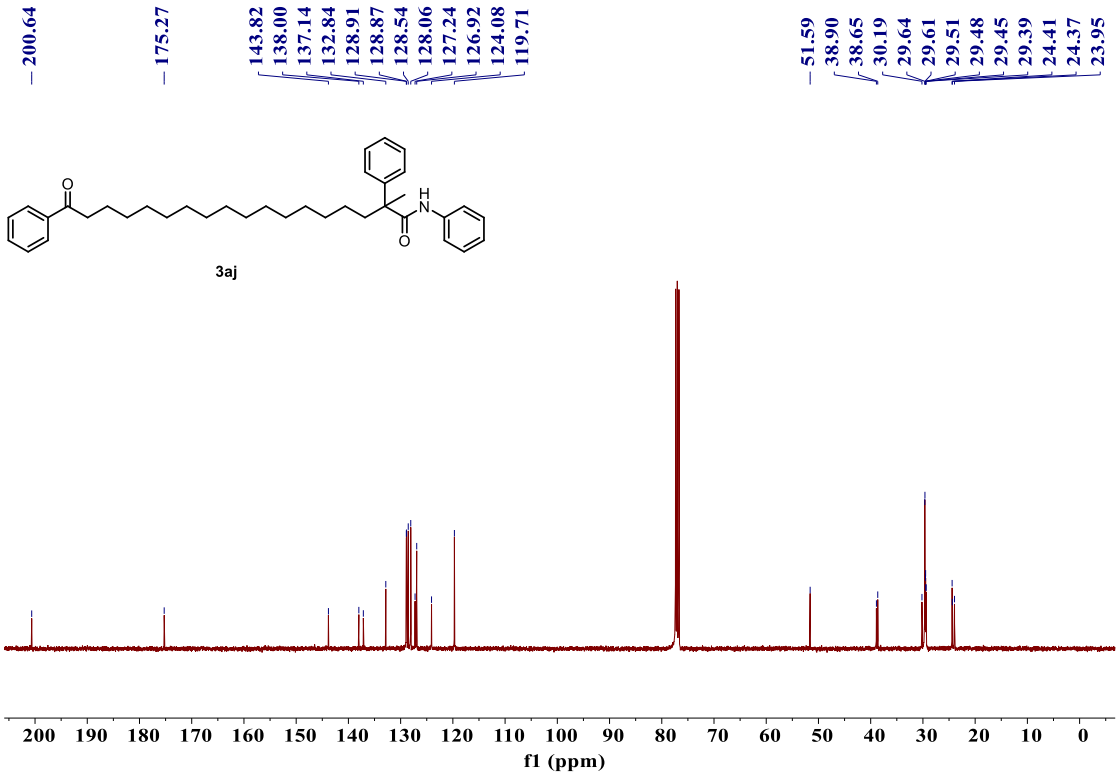
^{1}H and ^{13}C NMR spectra for product 3ai (Chloroform-*d*)



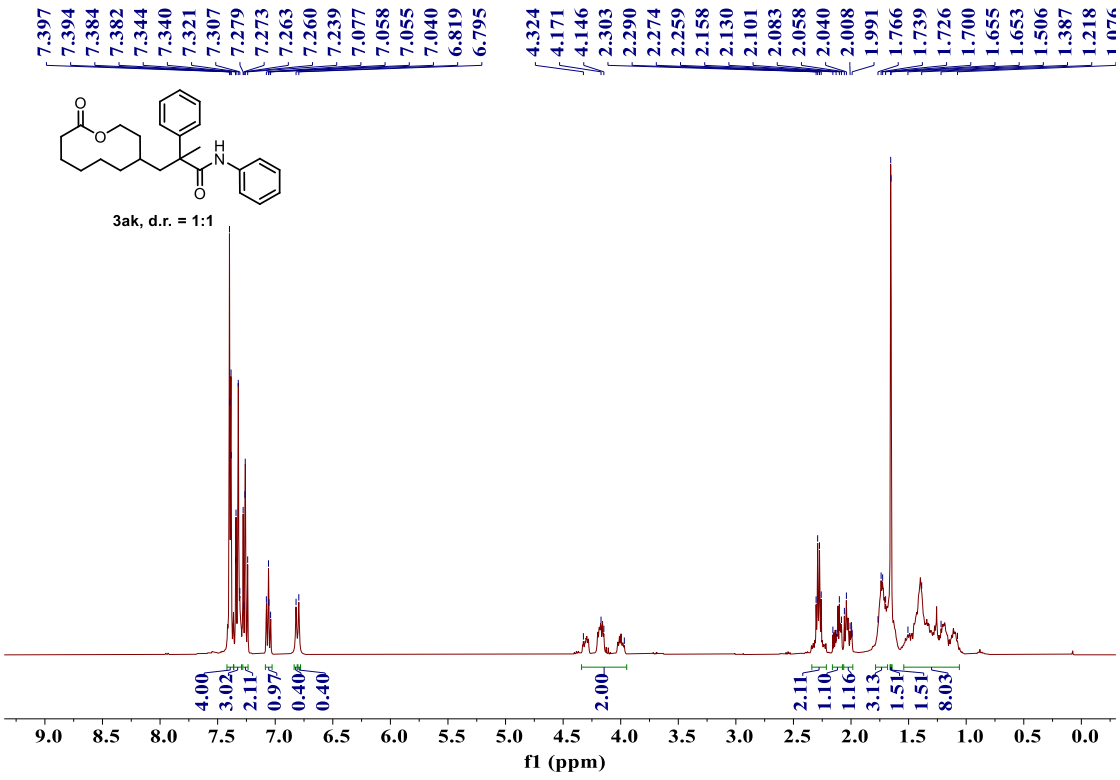


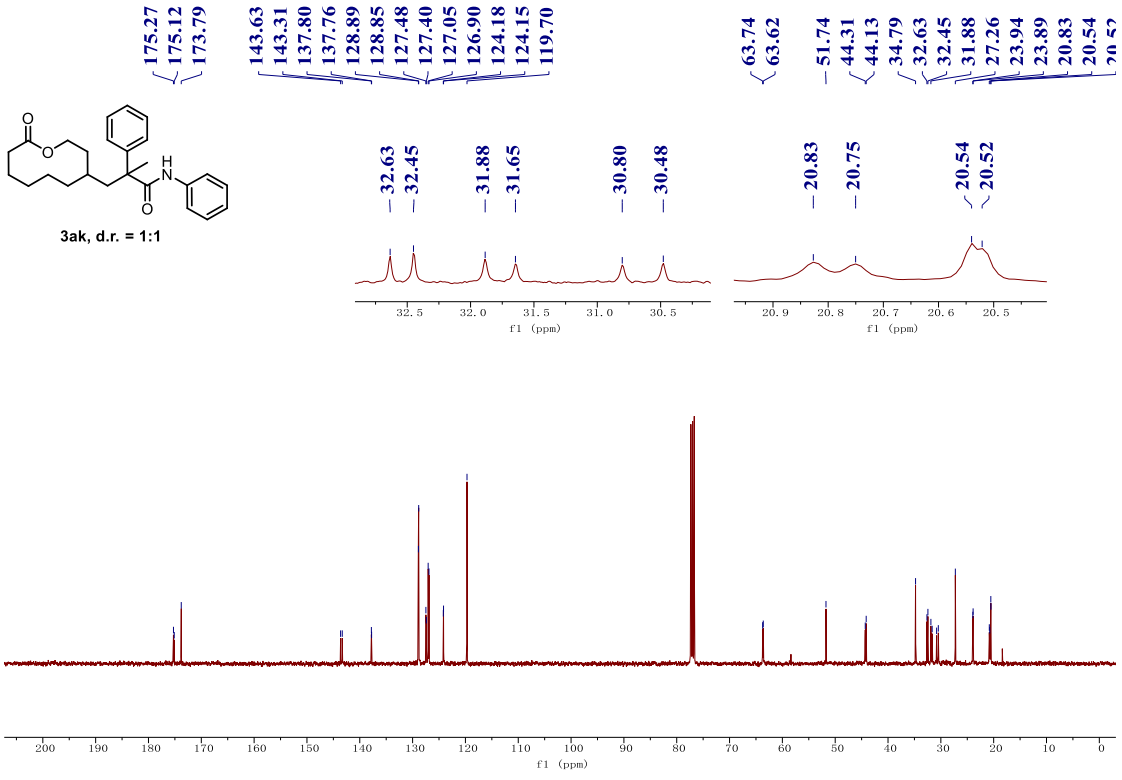
^{1}H and ^{13}C NMR spectra for product 3aj (Chloroform-*d*)



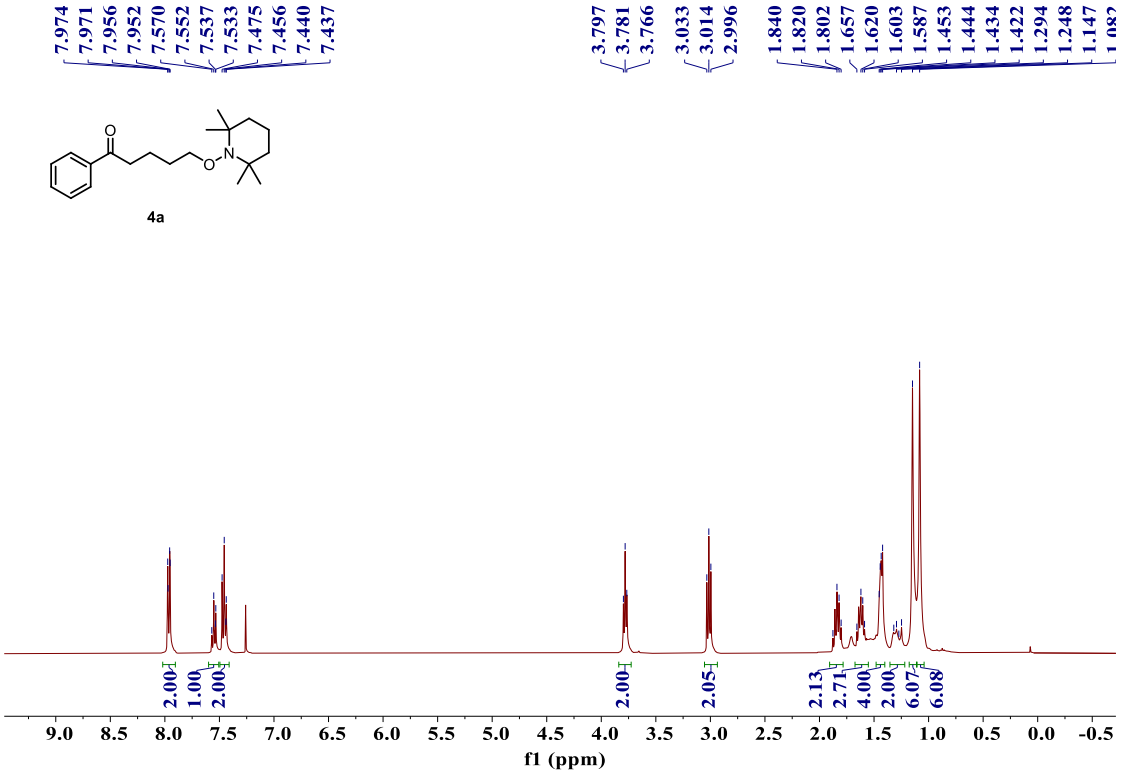


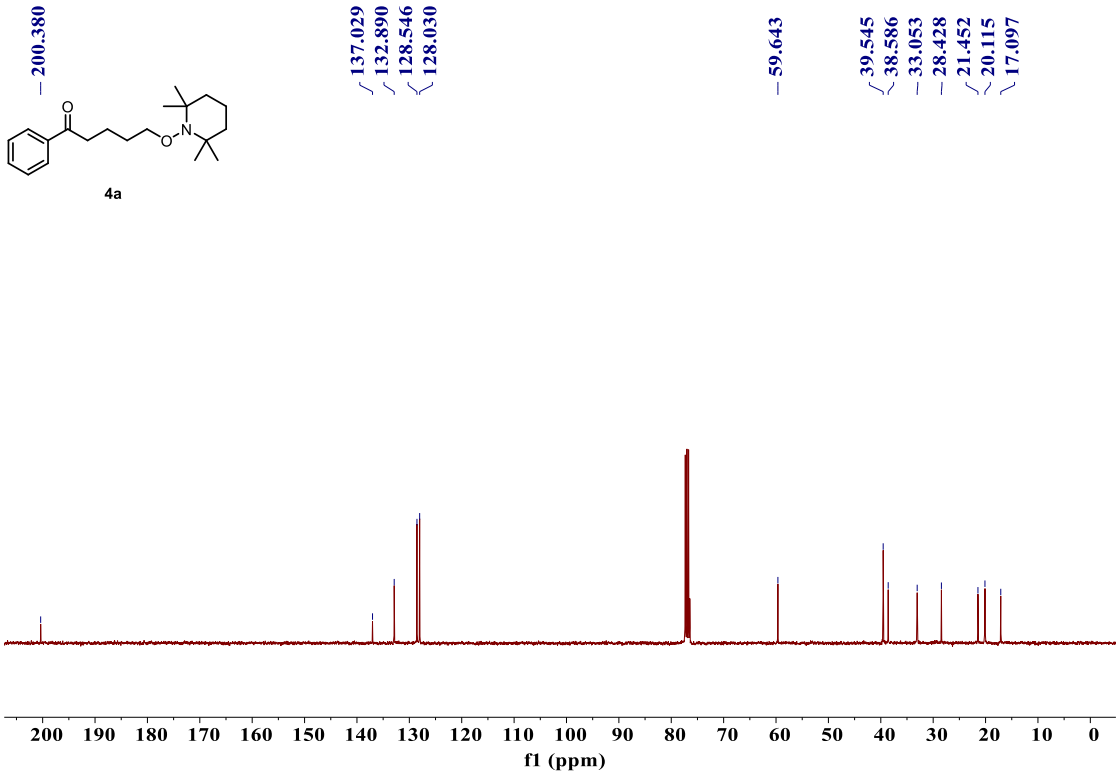
^{1}H and ^{13}C NMR spectra for product 3ak (Chloroform-*d*)



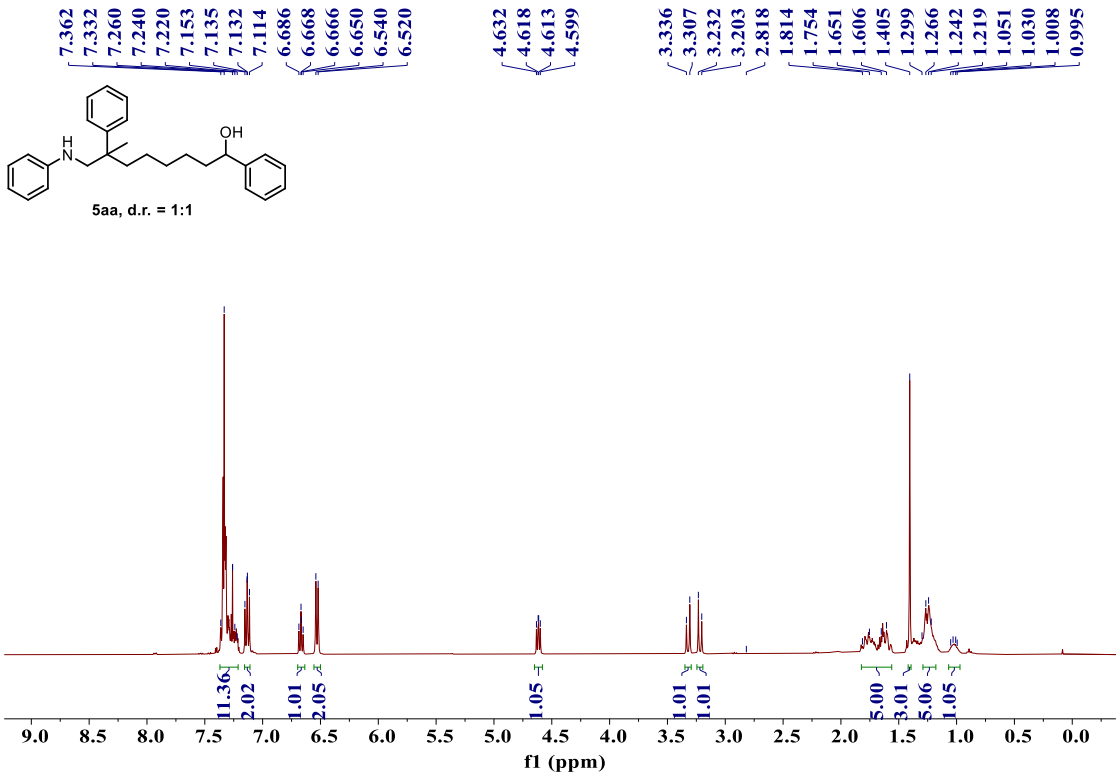


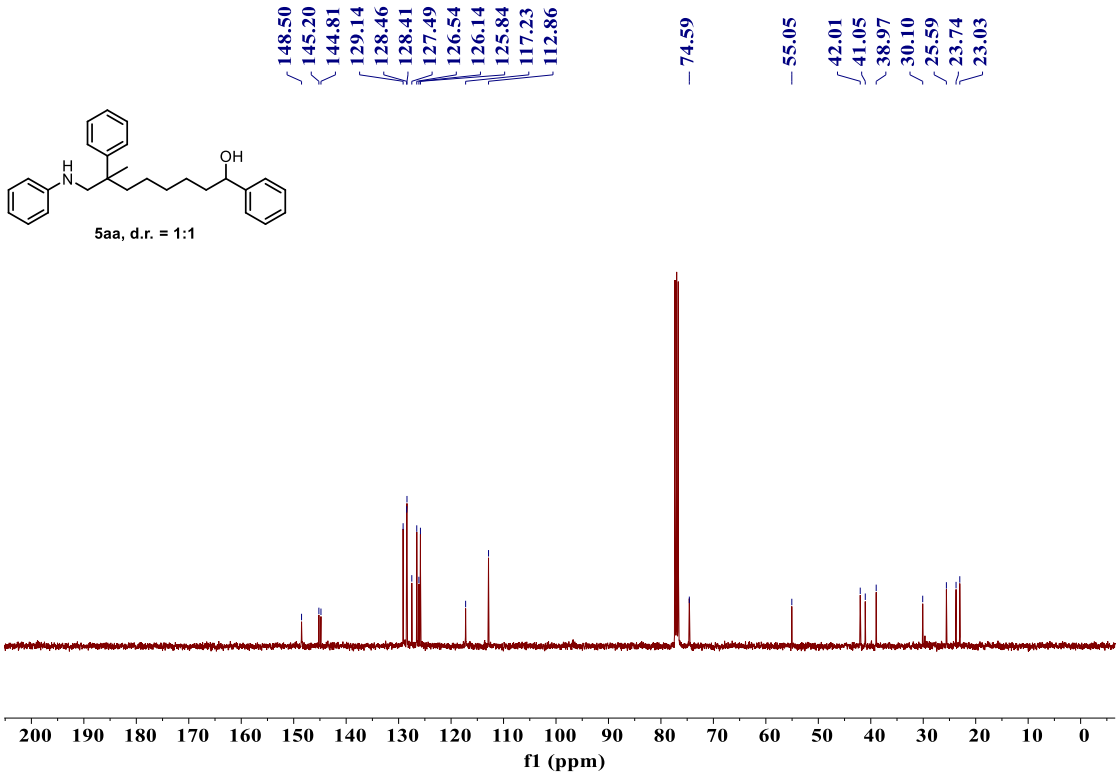
^{1}H and ^{13}C NMR spectra for product 4a (Chloroform-*d*)



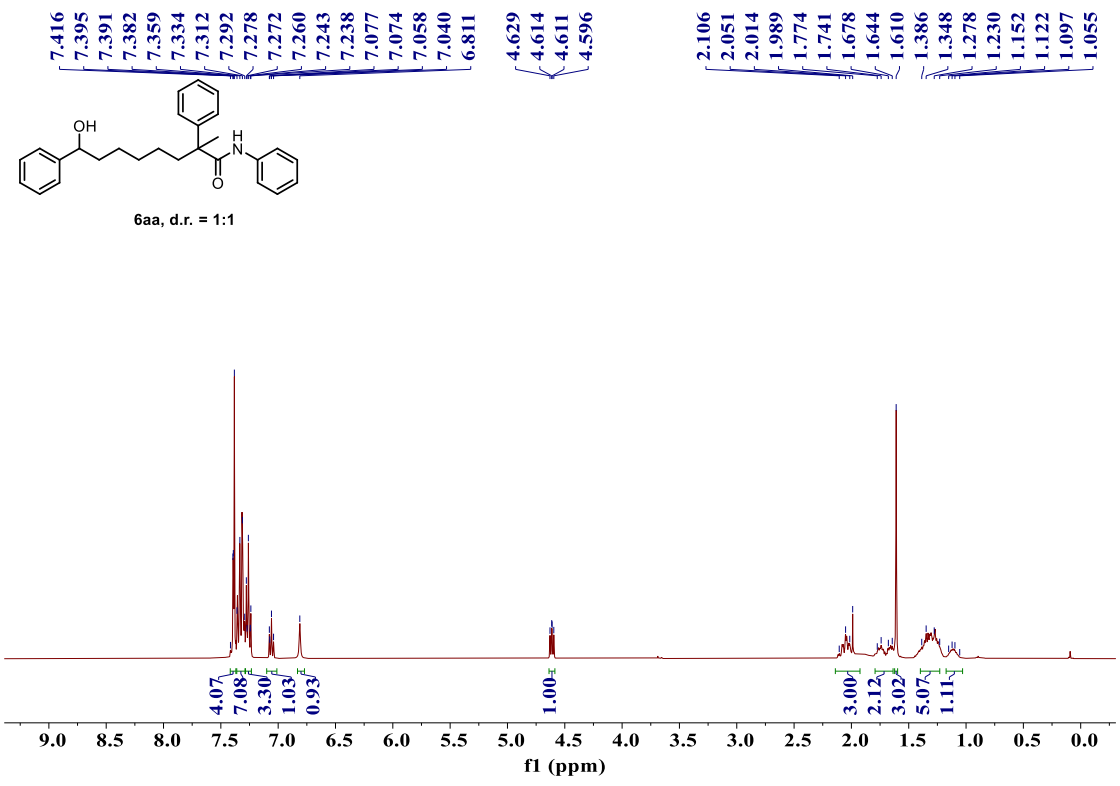


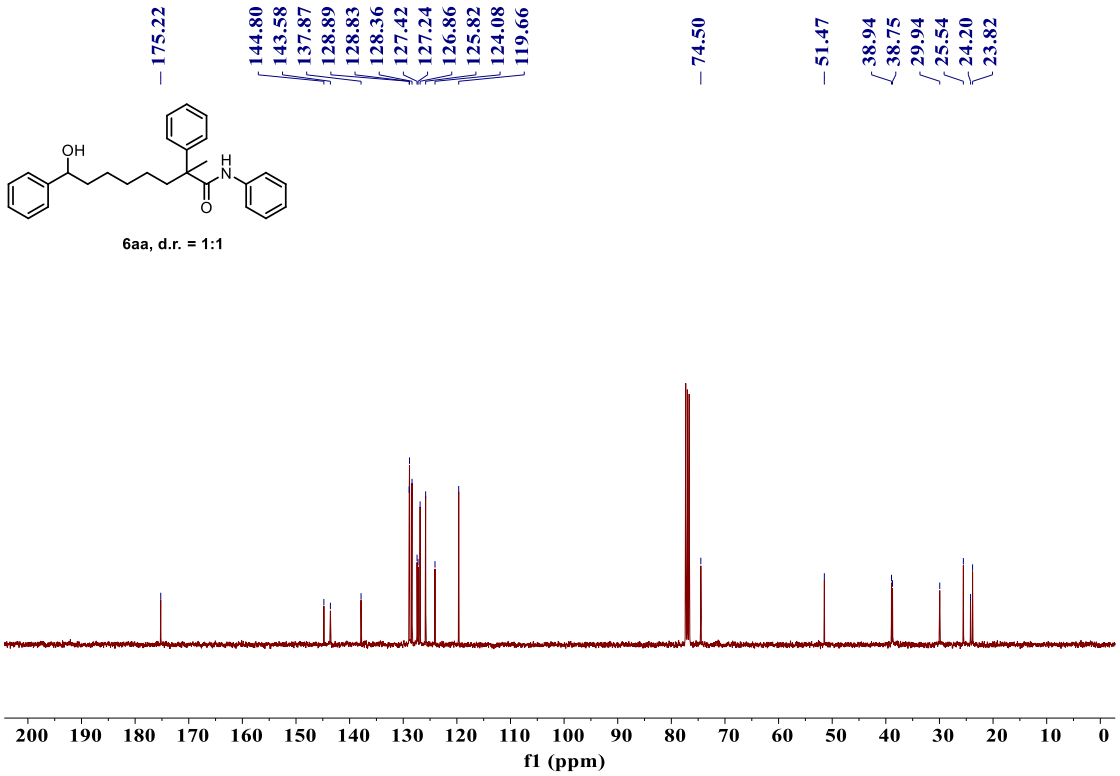
^{1}H and ^{13}C NMR spectra for product 5aa (Chloroform-*d*)





^{1}H and ^{13}C NMR spectra for product 6aa (Chloroform-*d*)

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128.36
127.42
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126.86
125.82
124.08
119.66
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51.47
38.94
38.75
29.94
25.54
24.20
23.82
OH
H
N
O
6aa, d.r. = 1:1
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)