Electronic Supplementary Material (ESI) for Green Chemistry.

Metal free, visible-light driven C-H ketoalkylation of glycine derivatives and peptides

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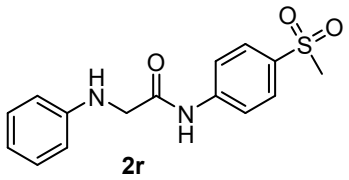
General Information

The reactions were conducted in oven-dried Schlenk-tube. And the photoinduced reactions were carried out in oven-dried Schlenk-tube with Wattecs blue LEDs Irradiation Parallel Reactor. Unless otherwise stated, all reagents were purchased from commercial sources and used without further purification. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz (100 MHz for ^{13}C NMR) spectrometer at ambient temperature. Chemical shift are reported in ppm from TMS with the solvent resonance as internal standard ($CDCl_3$: ^{1}H NMR: $\delta = 7.26$; ^{13}C NMR: $\delta = 77.0$). Coupling constants are reported in Hz with multiplicities denoted as s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), td (triplet of doublets) and m (multiplet). Active hydrogen of products didn't show due to hydrogen deuterium exchange in $CDCl_3$. FT-IR spectra were recorded on a Bruker V 70 spectrometer and only major peaks are reported in cm^{-1}. HRMS were obtained on a WATERS I-Class VION IMS Q-Tof. Melting points were measured using open glass capillaries in a SGW® X-4A apparatus. 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.

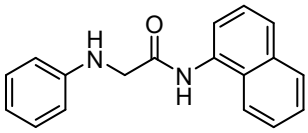
Starting Materials

The cycloalkyl hydroperoxides **1** and glycine derivatives **2** were prepared according to the literature.[1-2] The NMR spectra of the know compounds were in full accordance with the data in the literatures.

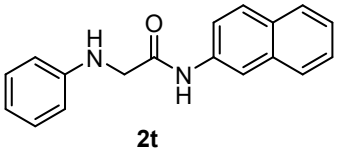
Glycine derivatives



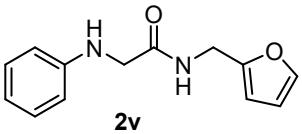
2r



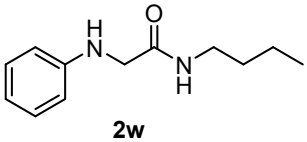
2s



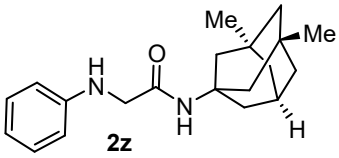
2t



2v

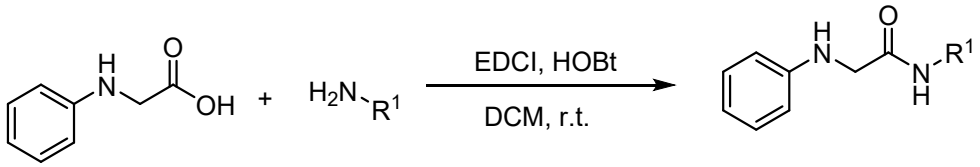


2w



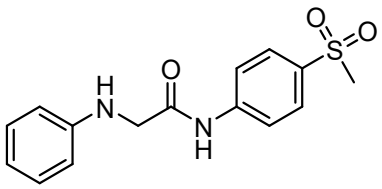
2z

Synthesis of 2r, 2s, 2t, 2v, 2w, 2z



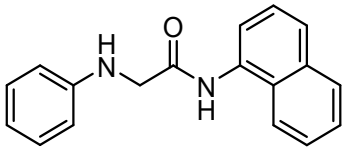
A 50 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with *N*-phenyl glycine (5.0 mmol), EDCI (8.0 mmol), HOBt (7.3 mmol), and corresponding amine (5.0 mmol) were dissolved in 10 mL DCM under N_2, then Et_3N (10 mmol) was added. The reaction mixture stirred overnight. Subsequently, water was added to the Schlenk-tube. The resulting mixture was extracted with DCM (three times), and the combined organic layers were dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography (PE/EA = 2:1 to 1:1). The pure product was obtained.

Characterization of Starting Materials

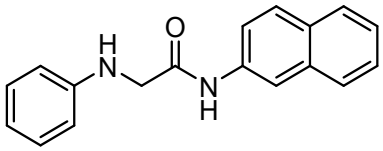


***N*-(4-(Methylsulfonyl)phenyl)-2-(phenylamino)acetamide (2r):** White solid (66%, 1003.2 mg); m.p.: 183-184 °C; R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.88 (s, 1H), 7.88 (d, *J* = 8.8 Hz, 2H), 7.75 (d, *J* = 8.8 Hz, 2H), 7.44-7.40 (m, 2H), 6.91-6.88 (m, 1H), 6.71-6.69 (m, 2H), 3.95 (s, 2H), 3.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.5, 146.7, 142.1, 135.6, 129.7,

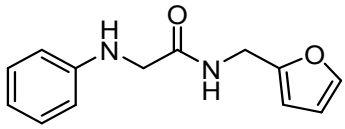
128.7, 120.2, 119.7, 113.7, 50.1, 44.7 ppm; IR (neat): υ $_{max}$ 3328, 3040, 1697, 1595, 1511, 756 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{17}N_2O_3S$ $[M+H]^+$ 305.0954, found 305.0961.



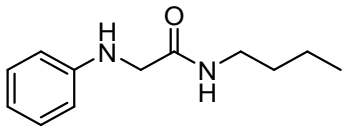
***N*-(Naphthalen-1-yl)-2-(phenylamino)acetamide (2s):** Light yellow solid (62%, 855.6 mg); m.p.: 149-150 °C; R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^1H NMR (400 MHz, $CDCl_3$): δ = 9.17 (s, 1H), 8.06-8.05 (m, 1H), 7.84-7.82 (m, 1H), 7.69-7.66 (m, 1H), 7.53-7.42 (m, 4H), 7.38-7.30 (m, 2H), 6.92-6.88 (m, 1H), 6.82-6.80 (m, 2H), 4.06 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.2, 146.8, 134.0, 131.7, 129.7, 128.7, 126.7, 126.3, 125.9, 125.7, 125.6, 120.2, 120.0, 113.7, 49.9 ppm; IR (neat): υ $_{max}$ 3334, 3054, 1675, 1602, 1501, 1431, 753 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{17}N_2O$ $[M+H]^+$ 277.1335, found 277.1335.



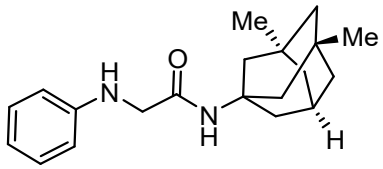
***N*-(Naphthalen-2-yl)-2-(phenylamino)acetamide (2t):** White solid (55%, 759 mg); m.p.: 185-186 °C; R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^1H NMR (400 MHz, $CDCl_3$): δ = 8.75 (s, 1H), 8.22 (s, 1H), 7.80-7.61 (m, 3H), 7.49-7.40 (m, 4H), 7.28-7.24 (m, 1H), 6.90-6.86 (m, 1H), 6.75-6.73 (m, 2H), 3.98 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.0, 147.0, 134.7, 133.8, 130.7, 129.6, 128.8, 127.5, 126.5, 125.1, 119.9, 119.8, 116.6, 113.7, 50.1 ppm; IR (neat): υ $_{max}$ 3331, 3045, 1666, 1601, 1497, 1438, 755 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{17}N_2O$ $[M+H]^+$ 277.1335, found 277.1336.



***N*-(Furan-2-ylmethyl)-2-(phenylamino)acetamide (2v):** White solid (72%, 828 mg); m.p.: 84-85 °C; R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.31-7.30 (m, 1H), 7.23-7.19 (m, 2H), 7.07 (s, 1H), 6.84-6.80 (m, 1H), 6.62-6.60 (m, 2H), 6.29-6.28 (m, 1H), 6.17-6.16 (m, 1H), 4.46 (d, *J* = 5.6 Hz, 2H), 3.82 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.4, 151.0, 147.0, 142.2, 129.4, 119.2, 113.3, 110.3, 107.3, 48.9, 36.1 ppm; IR (neat): υ $_{max}$ 3380, 3040, 1657, 1604, 1509, 754 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{15}N_2O_2$ $[M+H]^+$ 231.1128, found 231.1137.



***N*-Butyl-2-(phenylamino)acetamide (2w):** Light yellow oil (52%, 535.6 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.23-7.20 (m, 2H), 6.84-6.80 (m, 1H), 6.74 (s, 1H), 6.64-6.62 (m, 2H), 3.78 (s, 2H), 3.27 (m, 2H), 1.48-1.23 (m, 4H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.3, 147.0, 129.4, 119.3, 113.4, 49.0, 38.9, 31.6, 20.0, 13.7 ppm; IR (neat): υ $_{max}$ 3315, 3056, 2931, 1654, 1605, 1514, 751 cm^{-1}; HRMS (ESI) calcd for $C_{12}H_{19}N_2O$ $[M+H]^+$ 207.1492, found 207.1501.



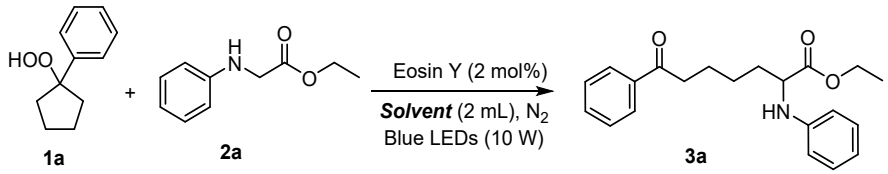
***N*-((3*R*,5*S*,7*r*)-3,5-dimethyladamantan-1-yl)-2-(phenylamino)acetamide (2z):** White solid (75%, 1170 mg); m.p.: 139-140 °C; R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.25-7.21 (m, 2H), 6.88-6.84 (m, 1H), 6.69-6.68 (m, 2H), 6.47 (s, 1H), 3.69 (s, 2H), 2.13-2.11 (m, 1H), 1.81 (s, 2H), 1.62-1.10 (m, 10H), 0.83 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.2, 147.1, 129.4, 119.3, 113.5, 53.2, 50.5, 49.9, 47.4, 42.6, 39.9, 32.4, 30.1, 30.0 ppm; IR (neat): υ $_{max}$ 3349, 3054, 2903, 1659, 1605, 1516, 752 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{29}N_2O$ $[M+H]^+$ 313.2274, found 313.2277.

Detailed Optimization of Reaction Conditions

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with ethyl *N*-phenylglycinate **2a** (0.30 mmol, 1.5 equiv.), photocatalyst, base (See Table S1). Then, the tube was evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cyclopentyl hydroperoxide **1a** (0.20 mmol, 1.0 equiv.) in solvent (2.0 mL) was added by a syringe. The reaction mixture was stirred under the irradiation of a 10 W blue LED (λ = 460−470 nm; distance app. 1.0 cm from the bulb) for a specified time. After that, the resulting mixture was 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: 10:1 to 8:1) furnishes the desired product **3a** as light yellow oil.

Table S1. Optimization of the Reaction of Ethyl *N*-phenylglycinate and Cyclopentyl Hydroperoxide 1a [a]

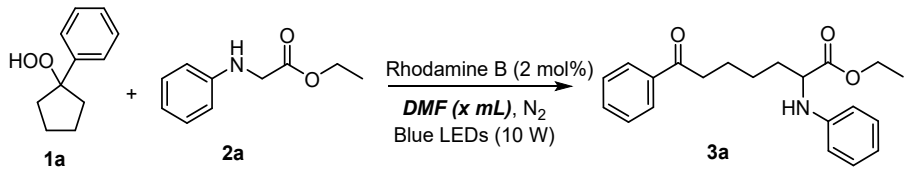
Solvent



Entry	Solvent	Yield (%)[b]
1	CH_3CN	18
2	1,4-dioxane	trace
3	toluene	< 10
4	DMSO	35
5	DMAc	41
6	DCM	23
7	acetone	34
8	NMP	37
9	DMF	42
10	MeOH	trace

[a] Reaction conditions: 2 mol % of Eosin Y, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), Solvent (2.0 mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b] Yield of isolated product.

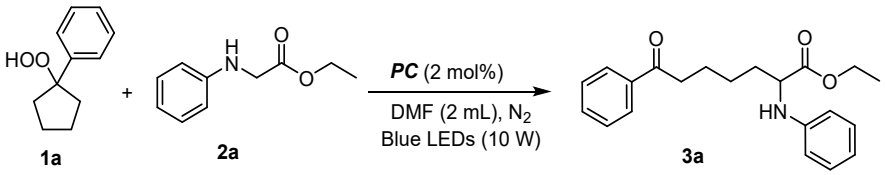
Ratio of Solvent



Entry	DMF (x mL)	Yield (%)[b]
1	1	52
2	2	57
3	3	42
4	4	44

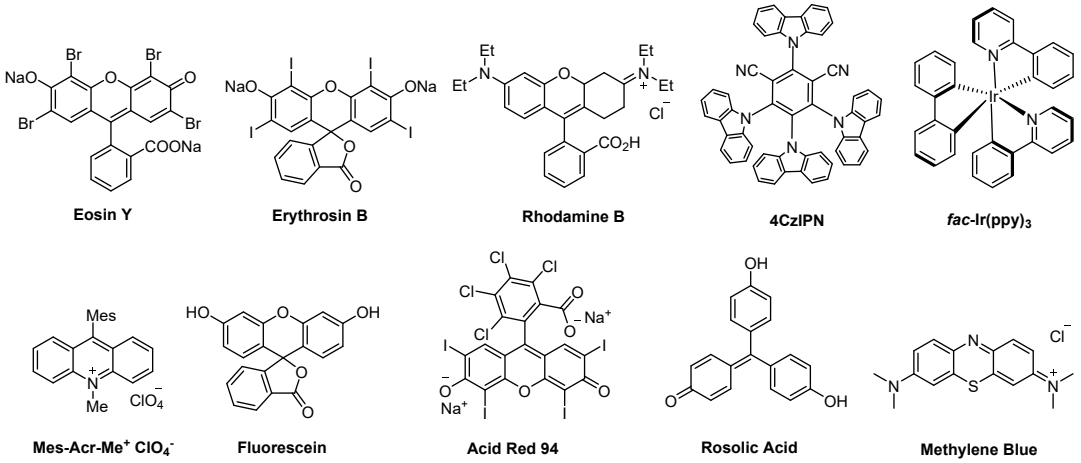
[a] Reaction conditions: 2 mol % of Rhodamine B, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), DMF (x mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b] Yield of isolated product.

Photocatalyst (PC)

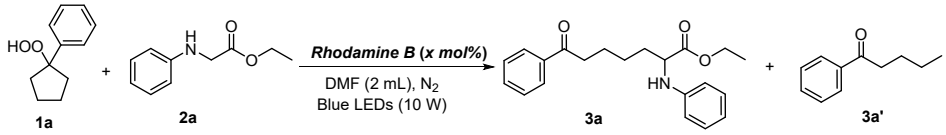


Entry	Photocatalyst	Yield (%)[b]
1	4CzIPN	trace
2	Erythrosine B	33
3	Mes-Acr-Me$^+$ClO$_4^-$	22
4	Fluorescein	14
5	Rhodamine B	57
6	Methylene Blue	42
7	Acid Red 94	< 10
8	Rosolic Acid	24
9	Eosin Y	42
10	*fac*-Ir(ppy)$_3$	< 10

[a] Reaction conditions: 2 mol % of photocatalyst, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), DMF (2.0 mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b] Yield of isolated product.



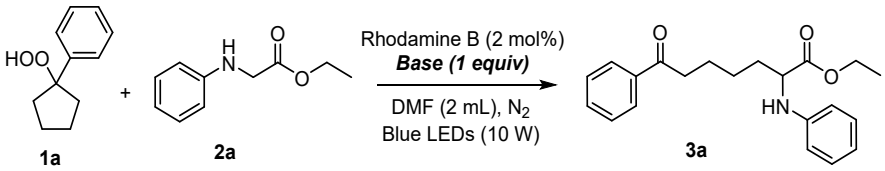
Ratio of Photocatalyst



Entry	Rhodamine B (x mol%)	Yield (%)[b]
1	1	53
2	2	57 (40)[c]
3	4	48
4	6	47

[a] Reaction conditions: x mol % of Rhodamine B, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), DMF (2.0 mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b]Yields of isolated product. [c]Yield of isolated 1-phenylpentan-1-one **3a'**.

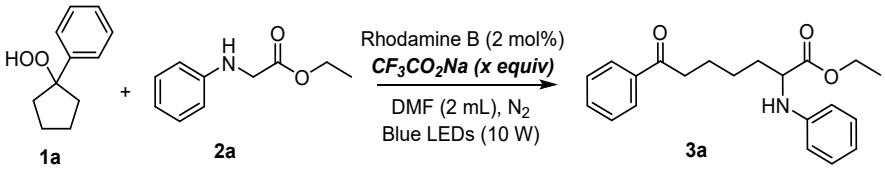
Base



Entry	Base	Yield (%)[b]
1	DABCO	trace
2	Et_3N	< 10
3	2,4,6-Collidine	35
4	K_2CO_3	< 10
5	Cs_2CO_3	trace
6	CF_3CO_2Na	64
7	Na_2CO_3	trace
8	CF_3SO_2Na	33
9	NaOAc	trace

[a] Reaction conditions: 2 mol % of Rhodamine B, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), base (0.2 mmol, 1.0 equiv.), DMF (2 mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b] Yield of isolated product.

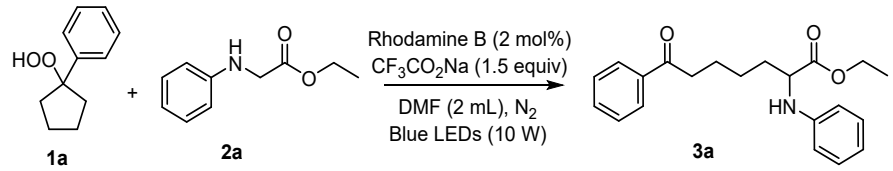
Ratio of Base



Entry	CF_3CO_2Na (x equiv)	Yield (%)[b]
1	1	64
2	1.5	87
3	2	59
4	2.5	47

[a] Reaction conditions: 2 mol % of Rhodamine B, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), CF_3CO_2Na (x equiv.), DMF (2 mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b] Yield of isolated product.

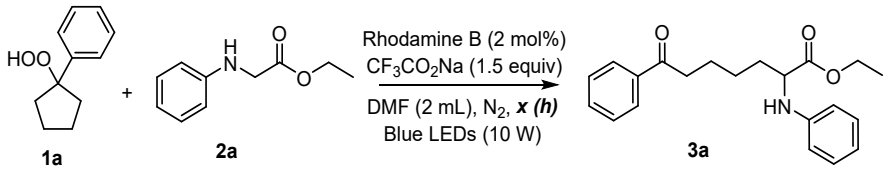
Ratio of 1a:2a



Entry	**1a**:**2a**	Yield (%)[b]
1	1:2	78
2	1:1.5	87
3	1:1	82
4	1.5:1	75
5	2:1	42

[a] Reaction conditions: 2 mol % of Rhodamine B, **1a** : **2a** = x, CF_3CO_2Na (1.5 equiv.), DMF (2.0 mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b] Yield of isolated product.

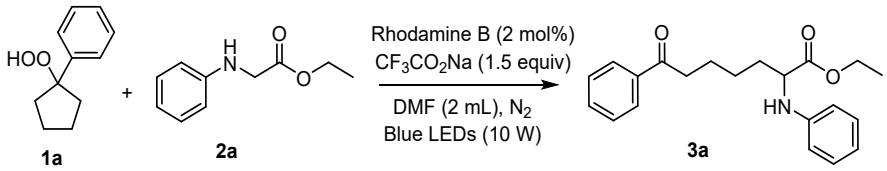
Time



Entry	Time (h)	Yield (%)[b]
1	1	8
2	1.5	74
3	2	77
4	3	84
5	4	88
6	12	91(87)[c]
7	Irradiation 1 h, then no light 12 h	55

[a] Reaction conditions: 2 mol % of Rhodamine B, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), CF_3CO_2Na (1.5 equiv.), DMF (2 mL), Blue LEDs (10 W), r.t., for x h, under N_2. [b] Yields were determined by ^1H NMR using 1,3,5-trimethoxybenzene as an internal standard. [c] Yield of isolated product.

Controlled Experiments



Entry	Deviation from standard conditions	Yield (%)[b]
1	standard	87
2	without Rhodamine B	27
3	without light	n.r.
4	without light	13 [c]
5	without CF_3CO_2Na	57
6	under O_2	0

[a] Reaction conditions: 2 mol % of Rhodamine B, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), DMF (2.0 mL), Blue LEDs (10 W), r.t., for 12 h, under N_2. [b] Yield of isolated product. [c] 80 °C.

Representative Procedure for the Reaction of Cycloalkyl Hydroperoxides and Glycine Derivatives

A 10 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with glycine derivatives **2** (0.30 mmol, 1.5 equiv.), Rhodamine B (1.9 mg, 2 mol %), CF_3CO_2Na (0.30 mmol, 1.5 equiv.). Then, the tube was evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cycloalkyl hydroperoxides **1** (0.20 mmol, 1.0 equiv.) in DMF (2.0 mL) was added by a syringe. The reaction mixture was stirred under the irradiation of a 10 W blue LED (λ = 460−470 nm; distance app. 1.0 cm from the bulb) for a specified time. After that, the resulting mixture was 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: 10:1 to 8:1) furnishes the corresponding products **3**, **4** and **5** in yields listed in Table 2 and Table 3.

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

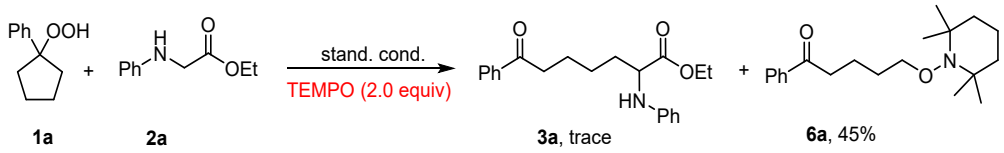
Scale-up Reaction

A 50 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was charged with ethyl *N*-phenylglycinate **2a** (3.0 mmol, 1.5 equiv.), Rhodamine B (19 mg, 2 mol %), CF_3CO_2Na (3.0 mmol, 1.5 equiv.). Then, the tube was evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cyclopentyl hydroperoxide **1a** (2.0 mmol, 1.0 equiv.) in DMF (20 mL) was added by a syringe. The reaction mixture was stirred under the irradiation of a 10 W blue LED (λ = 460−470 nm; distance app. 1.0 cm from the bulb) for 24 h. After that, the resulting mixture was quenched with H_2O and extracted with EtOAc (3 x 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: 10:1 to 8:1) furnishes the desired product **3a** (78%, 529.2 mg).

Mechanistic Investigation

(1) Radical Inhibiting Experiment

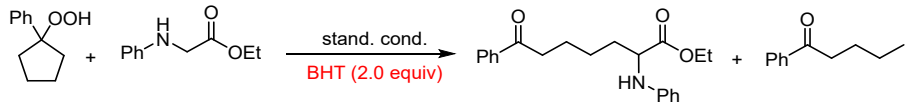
Cyclopentyl hydroperoxide **1a** (0.20 mmol, 1.0 equiv.), ethyl *N*-phenylglycinate **2a** (0.30 mmol, 1.5 equiv.), Rhodamine B (1.9 mg, 2 mol %), CF_3CO_2Na (0.30 mmol, 1.5 equiv.), TEMPO (0.4 mmol, 2.0 equiv.), DMF (2.0 mL) under N_2 for 12 h, with the irradiation of 10 W Blue LEDs.



1a **2a** **3a**, trace **6a**, 45%

When 2.0 equiv of TEMPO was subjected into the reaction of **1a** with **2a** under the standard conditions, only a trace amount of **3a** was observed，along with the TEMPO adduct **6a** isolated in 45% yield. This result indicates that a radical intermediate might be involved in this transformation.

Cyclopentyl hydroperoxide **1a** (0.20 mmol, 1.0 equiv.), ethyl *N*-phenylglycinate **2a** (0.30 mmol, 1.5 equiv.), Rhodamine B (1.9 mg, 2 mol %), CF_3CO_2Na (0.30 mmol, 1.5 equiv.), BHT (0.4 mmol, 2.0 equiv.), DMF (2.0 mL) under N_2 for 12 h, with the irradiation of 10 W Blue LEDs.

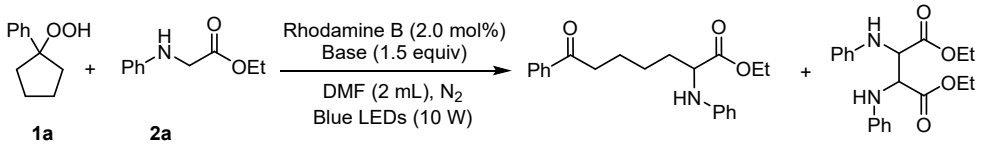


1a **2a** **3a**, 17% **3a'**, 23%

When 2.0 equiv of BHT was added to the reaction of **1a** with **2a** under the standard conditions, the yield of product **3a** was dramatically decreased (17%). This result indicates that a radical pathway might be involved in this transformation.

(2) Control Experiments

a) Cyclopentyl hydroperoxide **1a** (0.20 mmol, 1.0 equiv.), ethyl *N*-phenylglycinate **2a** (0.30 mmol, 1.5 equiv.), Rhodamine B (1.9 mg, 2 mol %), base (0.30 mmol, 1.5 equiv.), DMF (2.0 mL) under N_2 for 12 h, with the irradiation of 10 W Blue LEDs.



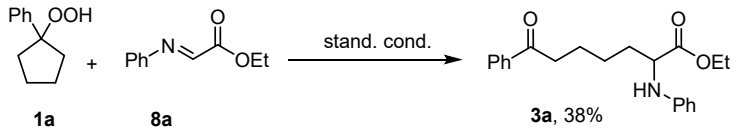
1a **2a**

CF_3CO_2Na	**3a**, 87%	**7a**, < 5%
NaOAc	**3a**, trace	**7a**, 38% (d.r. = 1:1.8)
Na_2CO_3	**3a**, trace	**7a**, 30% (d.r. = 1:1.8)

When CF_3CO_2Na was used as the base, the product **3a** was isolated in 87% yield, along with the homo-coupling product **7a** isolated less than 5% yield. However, using NaOAc or Na_2CO_3 instead of

CF_3CO_2Na led to the formation of **7a** in 38% and 30% yield, respectively. This result indicates that glycine radical was generated in the reaction, and strong base increased the concentration of *α*-carbon radical leading to homo-coulping **7a** formation.

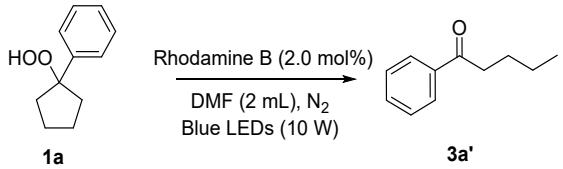
b) Cyclopentyl hydroperoxide **1a** (0.20 mmol, 1.0 equiv.), (*E*)-ethyl-2-(phenylimino)acetate **8a** (0.30 mmol, 1.5 equiv.), Rhodamine B (1.9 mg, 2 mol %), CF_3CO_2Na (0.30 mmol, 1.5 equiv.), DMF (2.0 mL) under N_2 for 12 h, with the irradiation of 10 W Blue LEDs.



1a **8a** **3a**, 38%

When ethyl-2-(phenylimino)acetate **8a** was used instead of **2a** under the standard conditions, the product **3a** was isolated in 38% yield. This result indicates that ethyl-2-(phenylimino)acetate might be as intermediate in this transformation

c) Cyclopentyl hydroperoxide **1a** (0.20 mmol, 1.0 equiv.), Rhodamine B (1.9 mg, 2 mol %), DMF (2.0 mL) under N_2 for 12 h, with the irradiation of 10 W Blue LEDs.



1a **3a'**

Entry	PC	Yield of **3a'** (%)	**1a** left (%)
1	No Rhodamine B	14	74
2	Rhodamine B	83	trace

Irradiation of cyclopentyl hydroperoxide **1a** in DMF with a 10 W blue LED led to the ring-opening by product **3a'** in 14% yield, along with 74% of **1a** recovered. When 2.0 mol% of Rhodamine B was added to the reaction mixture, the **3a'** was isolated in 83 % yield. These results indicate that the existence of Rhodamine B could accelerate the decomposition of cyclopentyl hydroperoxide **1a**.

(3) Stern-Volmer Fluorescence Quenching Experiments

To a solution of Rhodamine B in anhydrous, N_2-saturated DMF (5×10^{-4} mol/L) in a quartz cuvette, different amounts of cyclopentyl hydroperoxide (**1a**) and ethyl *N*-phenylglycinate (**2a**) were added, respectively, and the resulting changes in fluorescence intensity (concentration of **1a** and **2a**: 2.5×10^{-2} mol/L, 5×10^{-2} mol/L, 7.5×10^{-2} mol/L, 10×10^{-2} mol/L, 12.5×10^{-2} 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.[3]

a) b)

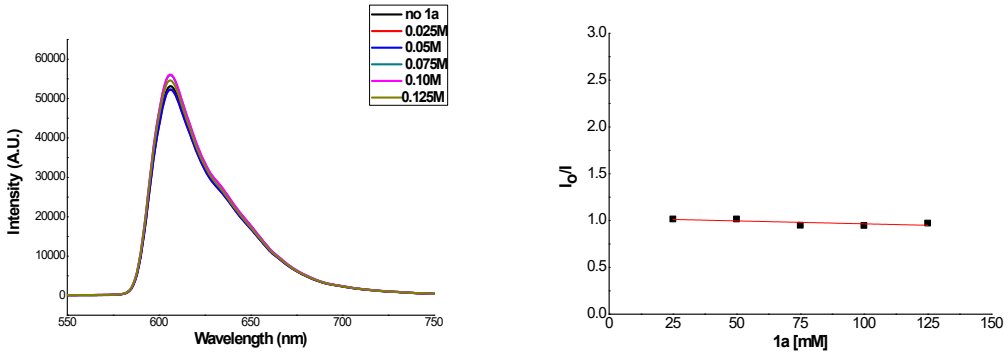


Figure S1. (a) The fluorescence emission spectra of Rhodamine B with different concentration of **1a** added. (b) The Stern–Volmer emission quenching studies of **1a**. I_0 is the inherent fluorescence intensity of Rhodamine B. I is the fluorescence intensity of Rhodamine B in the presence of **1a**.

c) d)

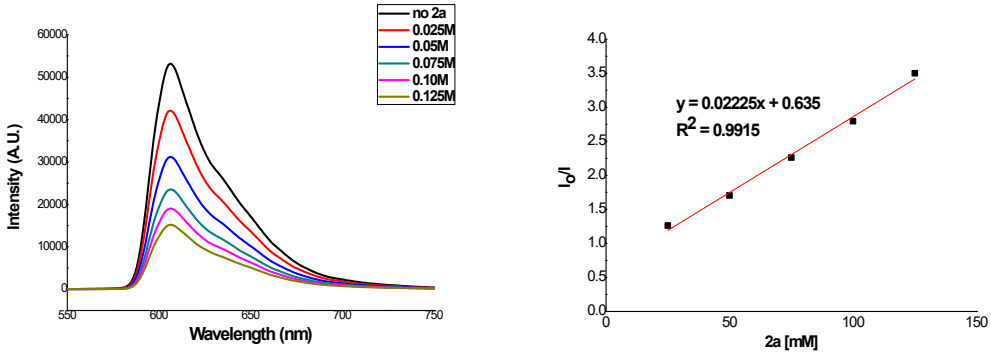
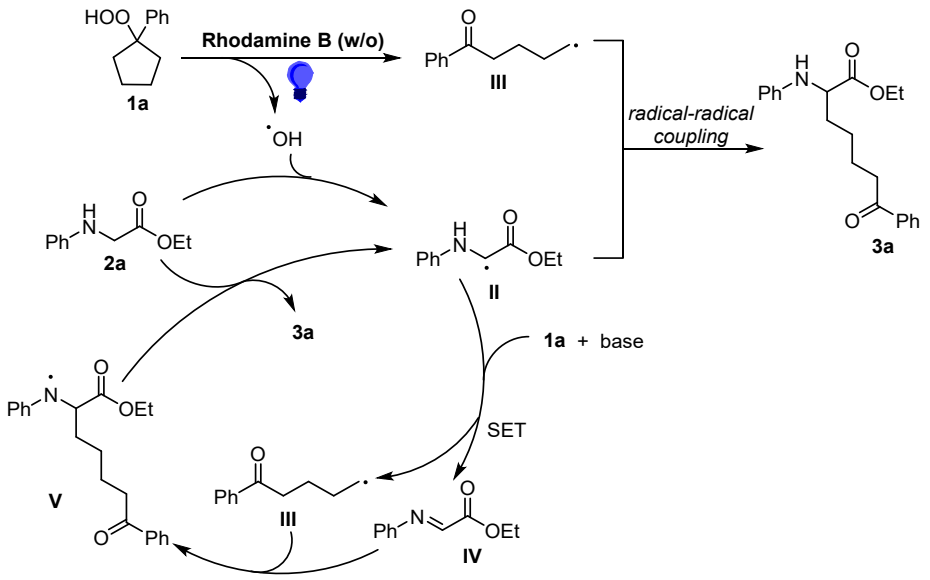


Figure S2. (c) The fluorescence emission spectra of Rhodamine B with different concentration of **2a** added. (d) The Stern–Volmer emission quenching studies of **2a**. I_0 is the inherent fluorescence intensity of Rhodamine B. I is the fluorescence intensity of Rhodamine B in the presence of **2a**.

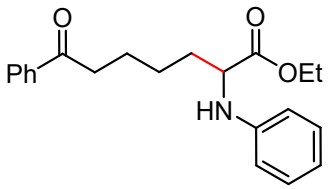
According to the results as well as the corresponding Stern-Volmer plots (Figure S1, Figure S2), the substrate **1a** did not show an obvious quenching effect to the fluorescence intensity of Rhodamine B. While the substrate **2a** showed an obvious quenching effect to the fluorescence intensity of Rhodamine B.

(4) Chain Propagation Mechanism

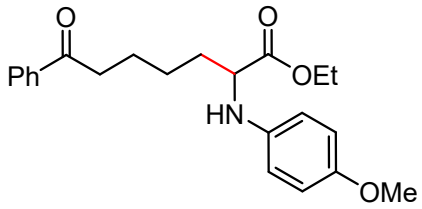


Homolytic cleavage of O-O bond in cyclopentyl hydroperoxide **1a** generates the radical intermediate **III** via a C-C bond cleavage and hydroxide radical in the presence of Rhodamine B (or not) under the blue light irradiation. H-abstraction from ethyl *N*-phenylglycinate **2a** to hydroxide radical affords the radical intermediate **II**. In the presence of cyclopentyl hydroperoxide **1a** and base, intermediate **II** is oxidized further to form the imine intermediate **IV**, which reacts with **III** to generate radical **V**. Finally, radical **V** abstracts hydrogen atom from **2a** to generate product **3a**, along with formation of radical **II**, which participates in the reaction cycle again.

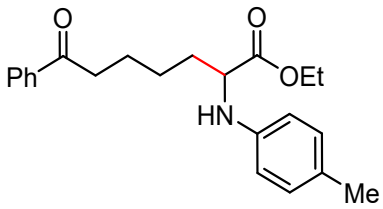
Characterization of Products 3, 4 and 5



Ethyl 7-oxo-7-phenyl-2-(phenylamino)heptanoate (3a): White solid (87%, 59.0 mg); m.p.: 60-61 °C; R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.19-7.15 (m, 2H), 6.75-6.72 (m, 1H), 6.64-6.62 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.07 (t, *J* = 6.4 Hz, 1H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.96-1.75 (m, 4H), 1.57-1.49 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 174.1, 146.8, 136.9, 133.0, 129.3, 128.5, 128.0, 118.2, 113.4, 61.0, 56.5, 38.2, 32.9, 25.3, 23.8, 14.2 ppm; IR (neat): υ $_{max}$ 3382, 3055, 2936, 1730, 1682, 1601, 1505, 750 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{26}NO_3$ $[M+H]^+$ 340.1907, found 340.1910.

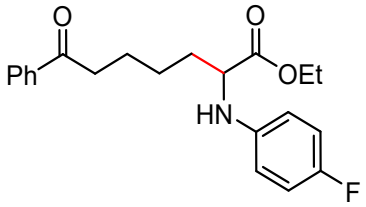


Ethyl 2-((4-methoxyphenyl)amino)-7-oxo-7-phenylheptanoate (3b): Light yellow oil (92%, 67.9 mg); R_f = 0.4 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7.53 (m, 1H), 7.47-7.43 (m, 2H), 6.76 (d, *J* = 8.8 Hz, 2H), 6.60 (d, *J* = 8.8 Hz, 2H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.98 (t, *J* = 6.4 Hz, 1H), 3.73 (s, 3H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.90-1.75 (m, 4H), 1.58-1.51 (m, 2H), 1.22 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 174.4, 152.7, 141.0, 136.9, 132.9, 128.5, 128.0, 115.2, 114.8, 60.9, 57.7, 55.7, 38.2, 33.0, 25.4, 23.9, 14.2 ppm; IR (neat): υ $_{max}$ 3362, 3045, 2964, 1730, 1682, 1611, 1511, 820, 731 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_4$ $[M+H]^+$ 370.2013, found 370.2012.

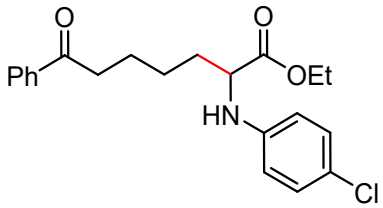


Ethyl 7-oxo-7-phenyl-2-(*p*-tolylamino)heptanoate (3c): Colorless oil (92%, 65.0 mg); R_f = 0.3

(EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.44 (m, 2H), 6.98 (d, *J* = 8.0 Hz, 2H), 6.55 (d, *J* = 8.0 Hz, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.03 (t, *J* = 6.4 Hz, 1H), 2.97 (t, *J* = 7.2 Hz, 2H), 2.23 (s, 3H), 1.92-1.75 (m, 4H), 1.57-1.53 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 174.2, 144.5, 136.9, 132.9, 128.6, 128.0, 127.6, 127.5, 113.7, 61.0, 56.9, 38.2, 32.9, 25.3, 23.9, 20.4, 14.2 ppm; IR (neat): υ $_{max}$ 3380, 3046, 2936, 1730, 1682, 1601, 1517, 860, 771 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_3$ $[M+H]^+$ 354.2064, found 354.2063.

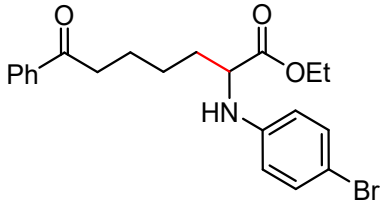


Ethyl 2-((4-fluorophenyl)amino)-7-oxo-7-phenylheptanoate (3d): Light yellow solid (72%, 51.4 mg); m.p.: 83-84 °C; R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.43 (m, 2H), 6.89-6.85 (m, 2H), 6.59-6.56 (m, 2H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.98 (t, *J* = 6.4 Hz, 1H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.92-1.75 (m, 4H), 1.56-1.48 (m, 2H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 174.0, 156.3 (d, *J* = 234.8 Hz), 143.1, 136.9, 133.0, 128.6, 128.0, 115.7 (d, *J* = 22.3 Hz), 114.7 (d, *J* = 7.4 Hz), 61.1, 57.4, 38.2, 32.8, 25.3, 23.8, 14.2 ppm; IR (neat): υ $_{max}$ 3365, 3038, 2942, 1733, 1683, 1599, 1513, 823, 754 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{25}FNO_3$ $[M+H]^+$ 358.1813, found 358.1817.

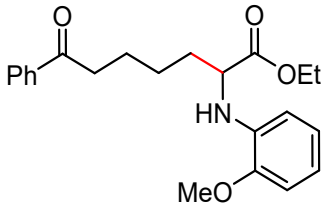


Ethyl 2-((4-chlorophenyl)amino)-7-oxo-7-phenylheptanoate (3e): White solid (65%, 48.5 mg); m.p.: 90-91 °C; R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.47-7.44 (m, 2H), 7.11 (d, *J* = 8.8 Hz, 2H), 6.54 (d, *J* = 8.8 Hz, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.01 (t, *J* = 6.4 Hz, 1H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.93-1.75 (m, 4H), 1.55-1.50 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 173.8, 145.4, 136.9, 133.0, 129.1, 128.6, 128.0, 122.9, 114.6, 61.2, 56.6, 38.2, 32.7, 25.2, 23.8, 14.2 ppm; IR (neat): υ $_{max}$ 3375, 3042, 2939, 1731, 1682, 1599, 1510, 814, 755 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{25}ClNO_3$ $[M+H]^+$ 374.1517,

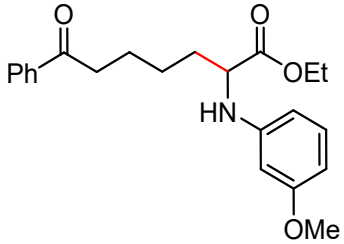
found 374.1520.



Ethyl 2-((4-bromophenyl)amino)-7-oxo-7-phenylheptanoate (3f): White solid (63%, 52.5 mg); m.p.: 97-98 °C; R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.24 (d, *J* = 8.8 Hz, 2H), 6.51 (d, *J* = 8.8 Hz, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.01 (t, *J* = 6.4 Hz, 1H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.91-1.76 (m, 4H), 1.55-1.51 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 173.6, 145.7, 136.9, 133.0, 132.0, 128.6, 128.0, 115.2, 110.2, 61.2, 56.6, 38.2, 32.6, 25.2, 23.8, 14.2 ppm; IR (neat): υ $_{max}$ 3374, 3041, 2939, 1729, 1682, 1596, 1501, 824, 750 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{25}BrNO_3$ $[M+H]^+$ 418.1012, found 418.1016.

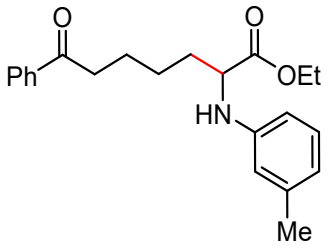


Ethyl 2-((2-methoxyphenyl)amino)-7-oxo-7-phenylheptanoate (3g): Light yellow oil (76%, 56.1 mg); R_f = 0.4 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.43 (m, 2H), 6.85-6.77 (m, 2H), 6.72-6.68 (m, 1H), 6.57-6.55 (m, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.07 (t, *J* = 6.4 Hz, 1H), 3.85 (s, 3H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.94-1.76 (m, 4H), 1.59-1.55 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 174.1, 147.1, 136.9, 136.7, 132.9, 128.5, 128.0, 121.1, 117.5, 110.4, 109.8, 61.0, 56.3, 55.5, 38.3, 32.9, 25.4, 23.9, 14.2 ppm; IR (neat): υ $_{max}$ 3382, 3060, 2939, 1733, 1683, 1600, 1514, 739 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_4$ $[M+H]^+$ 370.2013, found 370.2012.

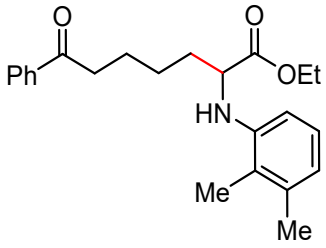


Ethyl 2-((3-methoxyphenyl)amino)-7-oxo-7-phenylheptanoate (3h): Colorless oil (59%, 43.5 mg);

R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.92 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.44 (m, 2H), 7.09-7.05 (m, 1H), 6.32-6.21 (m, 3H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.05 (t, *J* = 6.4 Hz, 1H), 3.75 (s, 3H), 2.97 (t, *J* = 7.2 Hz, 2H), 1.94-1.74 (m, 4H), 1.56-1.50 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 173.9, 160.8, 148.1, 136.9, 133.0, 130.1, 128.6, 128.0, 106.5, 103.7, 99.6, 61.1, 56.6, 55.1, 38.2, 32.8, 25.2, 23.9, 14.2 ppm; IR (neat): υ $_{max}$ 3373, 3038, 2916, 1732, 1679, 1600, 1516, 750, 694 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_4[M+H]^+$ 370.2013, found 370.2014.

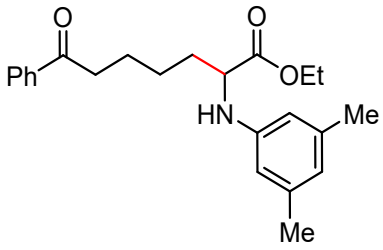


Ethyl 7-oxo-7-phenyl-2-(*m*-tolylamino)heptanoate (3i): Colorless oil (62%, 43.8 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.08-7.04 (m, 1H), 6.57-6.55 (m, 1H), 6.46-6.43 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.07 (t, *J* = 6.4 Hz, 1H), 2.98 (t, *J* = 7.2 Hz, 2H), 2.27 (s, 3H), 1.94-1.76 (m, 4H), 1.57-1.50 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 174.1, 146.8, 139.0, 136.9, 132.9, 129.1, 128.5, 128.0, 119.2, 114.3, 110.5, 61.0, 56.5, 38.2, 32.9, 25.2, 23.8, 21.5, 14.2 ppm; IR (neat): υ $_{max}$ 3386, 3030, 2926, 1731, 1683, 1602, 1516, 757, 692 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_3$ $[M+H]^+$ 354.2064, found 354.2066.

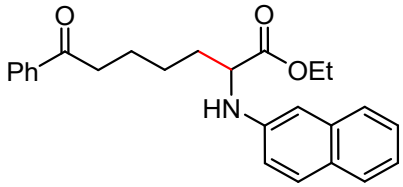


Ethyl 2-((2,3-dimethylphenyl)amino)-7-oxo-7-phenylheptanoate (3j): Light yellow oil (72%, 52.8 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.00-6.96 (m, 1H), 6.63-6.61 (m, 1H), 6.47-6.45 (m, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.10 (t, *J* = 6.4 Hz, 1H), 2.98 (t, *J* = 7.2 Hz, 2H), 2.27 (s, 3H), 2.12 (s, 3H), 1.94-1.76 (m, 4H), 1.59-1.53 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 174.3, 144.6, 136.9, 136.8, 133.0, 128.6, 128.0, 126.1, 121.2, 120.3, 108.9, 61.1, 56.8, 38.2, 33.0, 25.3, 23.9, 20.7, 14.2, 12.6 ppm; IR (neat): υ $_{max}$ 3382, 3060, 2937, 1731, 1683, 1590, 1509, 762, 692 cm^{-1};

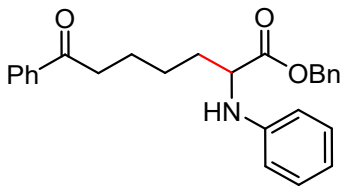
HRMS (ESI) calcd for $C_{23}H_{30}NO_3$ $[M+H]^+$ 368.2220, found 368.2220.



Ethyl 2-((3,5-dimethylphenyl)amino)-7-oxo-7-phenylheptanoate (3k): Colorless oil (68%, 50.6 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 6.40 (s, 1H), 6.28 (s, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.06 (t, *J* = 6.4 Hz, 1H), 2.97 (t, *J* = 7.2 Hz, 2H), 2.22 (s, 6H), 1.95-1.75 (m, 4H), 1.56-1.51 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 174.1, 146.7, 138.9, 136.9, 132.9, 128.6, 128.0, 120.4, 111.6, 61.0, 56.6, 38.2, 32.9, 25.2, 23.9, 21.4, 14.2 ppm; IR (neat): υ $_{max}$ 3378, 3050, 2969, 1733, 1686, 1603, 1500, 755 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{30}NO_3$ $[M+H]^+$ 368.2220, found 368.2226.

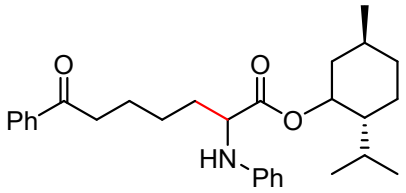


Ethyl 2-(naphthalen-2-ylamino)-7-oxo-7-phenylheptanoate (3l): Light yellow oil (90%, 70.0 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.66-7.53 (m, 4H), 7.43-7.20 (m, 4H), 6.95-6.82 (m, 2H), 4.24-4.17 (m, 3H), 2.99 (t, *J* = 7.2 Hz, 2H), 2.02-1.79 (m, 4H), 1.62-1.54 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 174.0, 144.5, 136.9, 134.9, 132.9, 129.1, 128.5, 128.0, 127.9, 127.6, 126.3, 126.0, 122.3, 118.1, 105.5, 61.1, 56.5, 38.2, 32.7, 25.2, 23.8, 14.2 ppm; IR (neat): υ $_{max}$ 3384, 3057, 2940, 1731, 1682, 1601, 1526, 752, 693 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{28}NO_3$ $[M+H]^+$ 390.2064, found 390.2067.

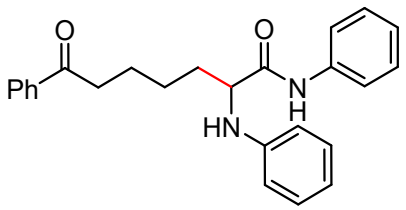


Benzyl 7-oxo-7-phenyl-2-(phenylamino)heptanoate (3m): Light yellow oil (57%, 45.7 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.92 (m, 2H), 7.56-7.54 (m, 1H), 7.47-7.44 (m, 2H), 7.33-7.28 (m, 5H), 7.18-7.14 (m, 2H), 6.77-6.74 (m, 1H), 6.65-6.63 (m, 2H), 5.15 (dd, *J* = 16.8 Hz, 12.0 Hz, 2H), 4.14 (t, *J* = 6.4 Hz, 2H), 2.92 (t, *J* = 7.2 Hz, 2H), 1.95-1.71 (m,

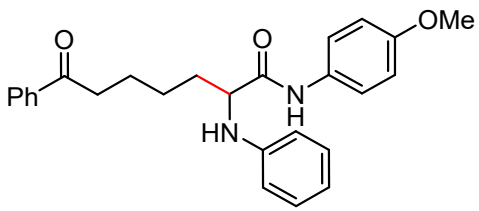
4H), 1.52-1.44 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 173.9, 146.6, 136.9, 135.5, 133.0, 129.3, 128.6, 128.5, 128.3, 128.2, 128.0, 118.6, 113.7, 66.8, 56.7, 38.2, 32.8, 25.2, 23.8 ppm; IR (neat): υ $_{max}$ 3382, 3057, 2929, 1735, 1682, 1601, 1504, 750, 694 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{28}NO_3$ $[M+H]^+$ 402.2064, found 402.2058.



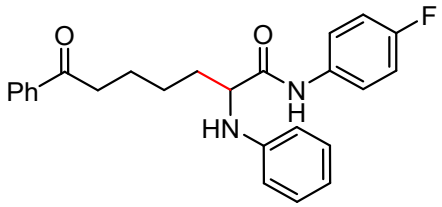
(*2R*,*5S*)-2-isopropyl-5-methylcyclohexyl 7-oxo-7-phenyl-2-(phenylamino)heptanoate (3n): Colorless oil (52%, 46.7 mg, d.r. = 1 : 1.6); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.44 (m, 2H), 7.18-7.14 (m, 2H), 6.75-6.71 (m, 1H), 6.66-6.62 (m, 2H), 4.72-4.62 (m, 1H), 4.08-4.04 (m, 1H), 2.97 (t, *J* = 7.2 Hz, 2H), 1.95-1.32 (m, 12H), 1.06-0.73 (m, 9H), 0.68-0.58 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 199.8, 173.6, 173.5, 146.8, 146.6, 136.9, 133.0, 129.2, 128.0, 118.5, 118.4, 113.9, 113.5, 75.2, 75.1, 57.0, 56.9, 46.8, 40.7, 40.6, 38.3, 38.2, 34.1, 32.8, 31.3, 26.1, 25.6, 25.3, 25.2, 23.9, 22.8, 21.9, 20.8, 20.7, 16.0, 15.6 ppm; IR (neat): υ $_{max}$ 3372, 3056, 2951, 1727, 1685, 1602, 1506, 751, 692 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{40}NO_3$ $[M+H]^+$ 450.3003, found 450.3001.



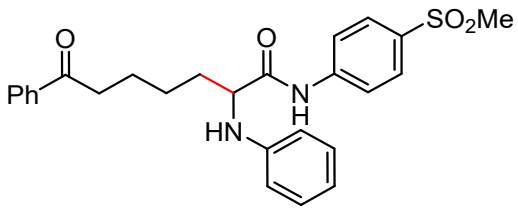
7-Oxo-N,7-diphenyl-2-(phenylamino)heptanamide (3o): White solid (57%, 44.0 mg); m.p.: 136-137 °C; R_f = 0.4 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.76 (s, 1H), 7.95-7.93 (m, 2H), 7.58-7.44 (m, 5H), 7.32-7.20 (m, 4H), 7.11-7.07 (m, 1H), 6.87-6.83 (m, 1H), 6.74-6.72 (m, 2H), 3.82 (dd, *J* = 8.0 Hz, 4.8 Hz, 1H), 3.01 (td, *J* = 7.2 Hz, 1.6 Hz, 2H), 2.14-2.11 (m, 1H), 1.93-1.58 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 171.6, 146.4, 137.4, 136.8, 133.1, 129.5, 128.9, 128.6, 128.0, 124.4, 119.9, 119.8, 114.3, 60.9, 38.0, 33.3, 25.6, 23.7 ppm; IR (neat): υ $_{max}$ 3385, 3053, 2968, 1727, 1676, 1501, 755 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{27}N_2O_2$ $[M+H]^+$ 387.2067, found 387.2071.



***N*-(4-methoxyphenyl)-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3p):** White solid (84%, 69.9 mg); m.p.: 108-109 °C; R_f = 0.3 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.65 (s, 1H), 7.95-7.93 (m, 2H), 7.57-7.41 (m, 5H), 7.23-7.19 (m, 2H), 6.85-6.81 (m, 3H), 6.72-6.69 (m, 2H), 3.81-3.76 (m, 4H), 2.99 (t, *J* = 6.8 Hz, 2H), 2.14-2.09 (m, 1H), 1.91-1.59 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 171.7, 156.4, 146.7, 136.8, 133.0, 130.5, 129.5, 128.6, 128.0, 121.6, 119.5, 114.0, 113.9, 60.5, 55.4, 37.9, 33.3, 25.6, 23.7 ppm; IR (neat): υ $_{max}$ 3376, 3034, 2958, 1727, 1675, 1601, 1511, 754 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{29}N_2O_3$ $[M+H]^+$ 417.2173, found 417.2172.

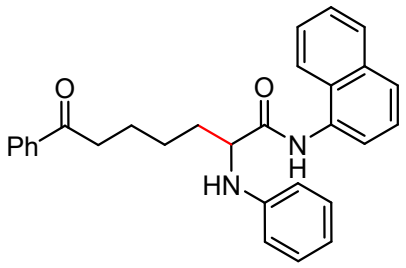


***N*-(4-fluorophenyl)-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3q):** White solid (65%, 52.5 mg); m.p.: 117-118 °C; R_f = 0.3 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.76 (s, 1H), 7.95-7.93 (m, 2H), 7.58-7.44 (m, 5H), 7.24-7.20 (m, 2H), 7.00-6.84 (m, 3H), 6.74-6.72 (m, 2H), 3.81 (dd, *J* = 8.0 Hz, 4.8 Hz, 1H), 3.01 (td, *J* = 6.8 Hz, 1.6 Hz, 2H), 2.17-2.08 (m, 1H), 1.93-1.56 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 171.6, 159.4 (*J* = 242.0 Hz), 136.8, 133.4(*J* = 2.7 Hz), 133.1, 129.6, 128.6, 128.0, 121.6 (*J* = 7.8 Hz), 120.0, 115.6 (*J* = 22.3 Hz), 114.2, 60.7, 37.9, 33.2, 25.6, 23.6 ppm; IR (neat): υ $_{max}$ 3365, 3043, 2939, 1729, 1677, 1603, 1510, 755 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{26}FN_2O_2$ $[M+H]^+$ 405.1973, found 405.1974.

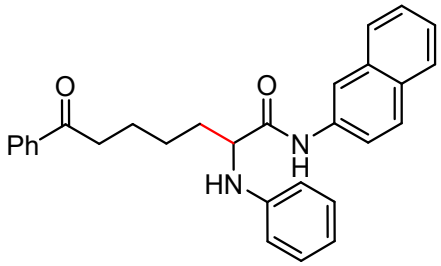


***N*-(4-(methylsulfonyl)phenyl)-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3r):** Colorless oil (42%, 39.2 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.09 (s, 1H), 7.94-7.73 (m, 6H), 7.58-7.43 (m, 3H), 7.23-7.19 (m, 2H), 6.87-6.83 (m, 1H), 6.72-6.68 (m, 2H),

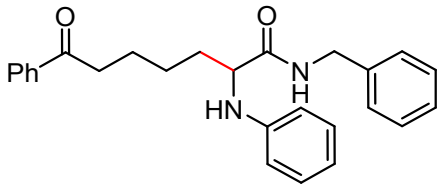
3.84 (dd, J = 8.0 Hz, 4.8 Hz, 1H), 3.05-2.99 (m, 5H), 2.11-2.08 (m, 1H), 1.93-1.57 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 172.5, 146.2, 142.3, 136.7, 135.3, 133.2, 129.6, 128.6, 128.5, 128.0, 120.1, 119.7, 114.1, 60.7, 44.6, 37.9, 33.1, 25.6, 23.5 ppm; IR (neat): υ $_{max}$ 3374, 3048, 2931, 1728, 1680, 1594, 1510, 756 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{29}N_2O_4S$ $[M+H]^+$ 465.1843, found 465.1845.



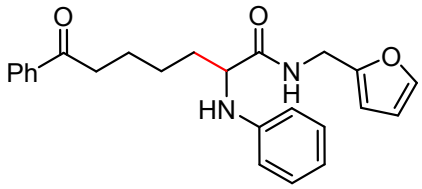
***N*-(naphthalen-1-yl)-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3s):** White solid (50%, 43.6 mg); m.p.: 126-127 ^{o}C; R_f = 0.3 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.32 (s, 1H), 8.03-7.80 (m, 4H), 7.67-7.41 (m, 7H), 7.33-7.24 (m, 3H), 6.91-6.83 (m, 3H), 3.97 (dd, J = 8.0 Hz, 4.8 Hz, 1H), 3.02 (td, J = 6.8 Hz, 1.6 Hz, 2H), 2.26-2.19 (m, 1H), 2.04-1.99 (m, 1H), 1.90-1.82 (m, 2H), 1.71-1.67 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 172.0, 146.6, 136.8, 134.0, 133.1, 131.9, 129.6, 128.6, 128.0, 126.8, 126.2, 125.9, 125.7, 125.4, 120.2, 119.9, 114.1, 60.7, 38.0, 33.5, 25.8, 23.8 ppm; IR (neat): υ $_{max}$ 3365, 3056, 2968, 1726, 1679, 1601, 1526, 755 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{29}N_2O_2$ $[M+H]^+$ 437.2224, found 437.2223.



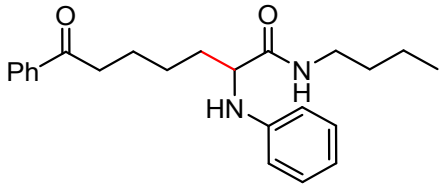
***N*-(naphthalen-2-yl)-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3t):** White solid (53%, 46.2 mg); m.p.: 161-162 ^{o}C; R_f = 0.3 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.97 (s, 1H), 8.24 (s, 1H), 7.95-7.93 (m, 2H), 7.78-7. 74 (m, 3H), 7.53-7.37 (m, 6H), 7.25-7.21 (m, 2H), 6.89-6.85 (m, 1H), 6.79-6.73 (m, 2H), 3.88 (dd, J = 8.0 Hz, 4.8 Hz, 1H), 3.02 (td, J = 6.8 Hz, 1.6 Hz, 2H), 2.22-2.13 (m, 1H), 2.00-1.91 (m, 1H), 1.87-1.80 (m, 2H), 1.66-1.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 171.7, 136.8, 134.8, 133.8, 133.1, 130.7, 129.6, 128.7, 128.6, 128.0, 127.6, 127.5, 126.5, 125.0, 119.8, 116.6, 114.5, 61.1, 37.9, 33.2, 25.6, 23.6 ppm; IR (neat): υ $_{max}$ 3349, 3053, 2941, 1732, 1678, 1601, 754 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{29}N_2O_2$ $[M+H]^+$ 437.2224, found 437.2223.



***N*-benzyl-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3u):** White solid (77%, 61.6 mg); m.p.: 99-100 °C; R_f = 0.3 (EtOAc/petroleum ether = 1:3); ^{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), 7.24-7.15 (m, 7H), 6.83-6.79 (m, 1H), 6.65-6.63 (m, 2H), 4.50 (dd, *J* = 14.8 Hz, 6.0 Hz, 1H), 4.36 (dd, *J* = 14.8 Hz, 5.6 Hz, 1H), 3.78 (dd, *J* = 8.0 Hz, 4.4 Hz, 1H), 2.98 (t, *J* = 6.8 Hz, 2H), 2.11-2.02 (m, 1H), 1.87-1.76 (m, 3H), 1.59-1.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 173.3, 146.6, 138.1, 136.8, 133.0, 129.3, 128.6, 128.5, 128.0, 127.5, 127.3, 119.2, 113.8, 59.7, 43.1, 38.0, 33.3, 25.7, 23.7 ppm; IR (neat): υ $_{max}$ 3339, 3052, 2936, 1731, 1674, 1601, 753 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{29}N_2O_2$ $[M+H]^+$ 401.2224, found 401.2226.

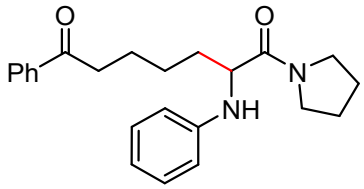


***N*-(furan-2-ylmethyl)-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3v):** Colorless oil (80%, 62.4 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.92 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.44 (m, 2H), 7.20-7.16 (m, 3H), 6.82-6.79 (m, 1H), 6.63-6.61 (m, 2H), 6.25-6.24 (m, 1H), 6.11-6.10 (m, 1H), 4.49 (dd, *J* = 15.6 Hz, 6.0 Hz, 1H), 4.36 (dd, *J* = 15.6 Hz, 5.2 Hz, 1H), 3.75 (dd, *J* = 8.0 Hz, 4.8 Hz, 1H), 2.97 (t, *J* = 6.8 Hz, 2H), 2.08-2.00 (m, 1H), 1.85-1.74 (m, 3H), 1.56-1.49 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 173.3, 151.2, 146.7, 142.0, 136.8, 133.0, 129.3, 128.5, 128.0, 119.1, 113.7, 110.3, 107.1, 59.6, 38.0, 36.1, 33.2, 25.5, 23.7 ppm; IR (neat): υ $_{max}$ 3356, 3057, 2935, 1715, 1676, 1601, 747 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{27}N_2O_3$ $[M+H]^+$ 391.2016, found 391.2021.

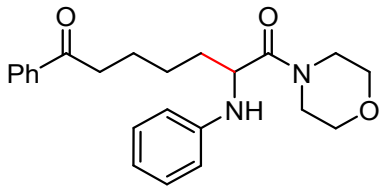


***N*-butyl-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3w):** Colorless oil (49%, 39.1 mg); R_f = 0.1 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7. 54 (m, 1H), 7.47-7.43 (m, 2H), 7.22-7.18 (m, 2H), 6.83-6.79 (m, 1H), 6.65-6.63 (m, 2H), 3.70 (dd, *J* = 8.0 Hz,

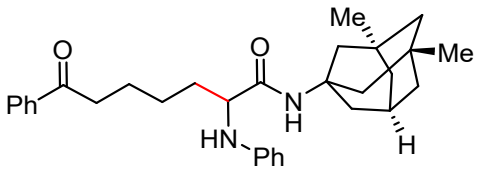
4.8 Hz, 1H), 3.22 (td, *J* = 6.8 Hz, 0.8 Hz, 2H), 2.99 (t, *J* = 7.2 Hz, 2H), 2.08-1.99 (m, 1H), 1.81-1.76 (m, 3H), 1.57-1.53 (m, 2H), 1.44-1.37 (m, 2H), 1.29-1.19 (m, 2H), 0.85 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 173.0, 146.6, 136.8, 133.1, 129.4, 128.6, 128.0, 119.3, 113.9, 59.9, 39.0, 38.0, 33.2, 31.6, 25.6, 23.7, 19.9, 13.6 ppm; IR (neat): υ $_{max}$ 3343, 3051, 2934, 1710, 1678, 1602, 754 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{31}N_2O_2$ $[M+H]^+$ 367.2380, found 367.2383.



7-Phenyl-2-(phenylamino)-1-(pyrrolidin-1-yl)heptane-1,7-dione (3x): Colorless oil (64%, 46.7 mg); R_f = 0.1 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.92 (m, 2H), 7.57-7.53 (m, 1H), 7.47-7.43 (m, 2H), 7.18-7.14 (m, 2H), 6.75-6.71 (m, 1H), 6.68-6.66 (m, 2H), 4.18 (t, *J* = 6.4 Hz, 1H), 3.58-3.41 (m, 4H), 2.97 (t, *J* = 7.2 Hz, 2H), 1.99-1.94 (m, 2H), 1.88-1.73 (m, 6H), 1.54-1.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 171.3, 146.7, 136.9, 133.0, 129.4, 128.6, 128.0, 118.5, 114.2, 55.7, 46.4, 46.0, 38.3, 32.4, 26.1, 25.2, 24.1, 24.0 ppm; IR (neat): υ $_{max}$ 3346, 3054, 2939, 1710, 1681, 1503, 753 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{29}N_2O_2$ $[M+H]^+$ 365.2224, found 365.2222.

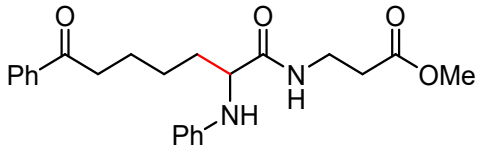


1-Morpholino-7-phenyl-2-(phenylamino)heptane-1,7-dione (3y): Colorless oil (58%, 44.1 mg); R_f = 0.1 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.92 (m, 2H), 7.58-7.54 (m, 1H), 7.47-7.43 (m, 2H), 7.19-7.15 (m, 2H), 6.76-6.73 (m, 1H), 6.67-6.65 (m, 2H), 4.33 (t, *J* = 7.2 Hz, 1H), 3.65-3.56 (m, 8H), 2.97 (t, *J* = 7.2 Hz, 2H), 1.89-1.67 (m, 4H), 1.55-1.48 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 171.5, 146.7, 136.9, 133.0, 129.4, 128.6, 128.0, 118.6, 114.1, 66.9, 66.6, 53.5, 46.0, 42.5, 38.2, 32.7, 25.1, 24.0 ppm; IR (neat): υ $_{max}$ 3341, 3021, 2927, 1720, 1680, 1601, 1503, 751 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{29}N_2O_3$ $[M+H]^+$ 381.2173, found 381.2175.

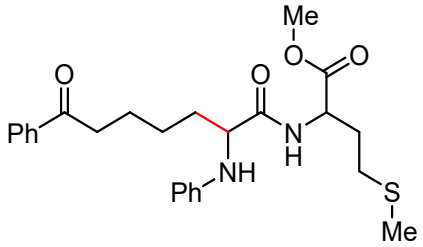


***N*-((*3R*,*5S*,*7r*)-3,5-dimethyladamantan-1-yl)-7-oxo-7-phenyl-2-(phenylamino)heptanamide (3z):**

White solid (67%, 63.4 mg); m.p.: 122-123 °C; R_f = 0.1 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88-7.86 (m, 2H), 7.50-7.46 (m, 1H), 7.40-7.36 (m, 2H), 7.14-7.11 (m, 2H), 6.75-6.55 (m, 1H), 6.57-6.55 (m, 2H), 6.46 (s, 1H), 3.46 (dd, *J* = 7.6 Hz, 4.8 Hz, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.03-2.02 (m, 1H), 1.93-1.86 (m,1H), 1.73-1.45 (m, 11H), 1.28-1.25 (m, 2H), 1.19-1.16 (m, 2H), 1.08-1.00 (m, 2H), 0.74 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 172.2, 146.8, 136.9, 133.0, 129.3, 128.6, 128.0, 119.1, 113.9, 60.5, 53.0, 50.5, 47.3, 47.2, 42.6, 39.8, 38.0, 33.3, 32.3, 30.0, 29.9, 25.5, 23.8 ppm; IR (neat): υ $_{max}$ 3351, 3038, 2901, 1720, 1678, 1601, 1513, 754 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{41}N_2O_2$ $[M+H]^+$ 473.3163, found 473.3165.

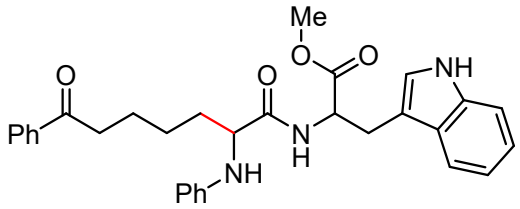


Methyl 3-(7-oxo-7-phenyl-2-(phenylamino)heptanamido)propanoate (4a): Light yellow oil (68%, 53.8 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.92 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.43 (m, 2H), 7.20-7.16 (m, 2H), 6.81-6.77 (m, 1H), 6.61-6.60 (m, 2H), 3.69 (dd, *J* = 7.6 Hz, 4.8 Hz, 1H), 3.57-3.42 (m, 5H), 2.99 (t, *J* = 7.2 Hz, 2H), 2.48 (t, *J* = 6.0 Hz, 2H), 2.04-1.96 (m, 1H), 1.82-1.73 (m, 3H), 1.57-1.52 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.1, 173.3, 172.3, 146.3, 136.8, 133.0, 129.3, 128.6, 128.0, 119.2, 113.7, 59.6, 51.6, 38.0, 34.6, 33.8, 33.1, 25.6, 23.7 ppm; IR (neat): υ $_{max}$ 3358, 3055, 2947, 1733, 1657, 1602, 1508, 754 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{29}N_2O_4$ $[M+H]^+$ 397.2122, found 397.2121.

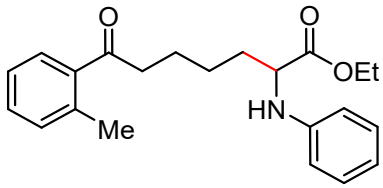


Methyl (7-oxo-7-phenyl-2-(phenylamino)heptanoyl)methioninate (4b): Light yellow oil (57%, 52.1 mg, d.r. = 1:1.4); R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.45-7.37 (m, 2H), 7.22-7.18 (m, 2H), 6.85-6.79 (m, 1H), 6.71-6.64 (m, 2H), 4.74-4.64 (m, 1H), 3.80-3.74 (m, 1H), 3.67 (s, 3H), 2.99 (t, *J* = 7.2 Hz, 2H), 2.47-2.25 (m, 2H), 2.13-1.76 (m, 9H), 1.59-1.54 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 173.6, 173.3, 172.3, 171.7, 146.6, 146.5, 136.8, 133.0, 129.4, 129.3, 128.6, 128.0, 119.3, 119.2, 114.1, 113.5, 59.7, 59.4, 52.4, 52.3, 51.3, 51.0, 38.0, 33.1, 31.2, 30.0, 29.7, 25.5, 23.7, 23.6, 15.4, 15.2 ppm; IR (neat): υ $_{max}$

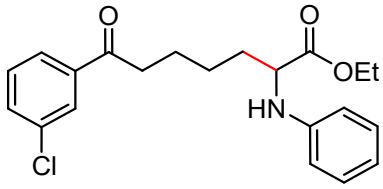
3350, 3055, 2921, 1740, 1673, 1600, 1505, 752 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{33}N_2O_4S$ $[M+H]^+$ 457.2156, found 457.2157.



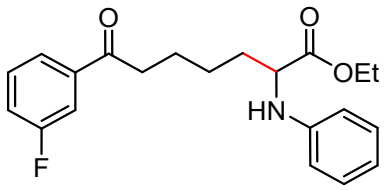
Methyl (7-oxo-7-phenyl-2-(phenylamino)heptanoyl)tryptophanate (4c): Light yellow oil (55%, 56.4 mg, d.r. = 1:1.6); R_f = 0.2 (EtOAc/petroleum ether = 1:2); ^1H NMR (400 MHz, $CDCl_3$): δ = 8.24 (s, 1H), 7.85-7.83 (m, 2H), 7.51-7.21 (m, 5H), 7.12-7.00 (m, 4H), 6.94-6.25 (m, 5H), 4.84-4.83 (m, 1H), 3.60-3.53 (m, 4H), 3.28-3.01 (m, 2H), 2.88-2.80 (m, 2H), 1.89-1.57 (m, 4H), 1.41-1.17 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 200.1, 173.1, 173.0, 172.1, 171.9, 136.8, 136.1, 136.0, 133.1, 133.0, 129.4, 129.2, 128.6, 128.5, 128.1, 128.0, 127.6, 127.1, 123.2, 122.8, 122.1, 122.0, 119.5, 119.4, 118.5, 118.4, 114.3, 113.6, 111.3, 111.1, 110.1, 109.2, 59.8, 59.3, 52.6, 52.3, 52.2, 51.8, 38.0, 33.1, 32.9, 27.6, 27.4, 25.5, 25.3, 23.7 ppm; IR (neat): υ $_{max}$ 3367, 3055, 2926, 1740, 1661, 1601, 1507, 748 cm^{-1}; HRMS (ESI) calcd for $C_{31}H_{34}N_3O_4$ $[M+H]^+$ 512.2544, found 512.2540.



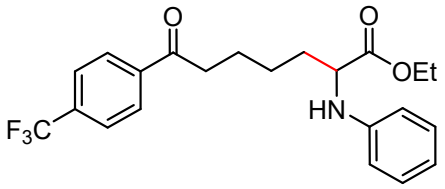
Ethyl 7-oxo-2-(phenylamino)-7-(*o*-tolyl)heptanoate (5a): Light yellow oil (64%, 44.9 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.60-7.58 (m, 1H), 7.38-7.34 (m, 1H), 7.25-7.23 (m, 2H), 7.19-7.15 (m, 2H), 6.75-6.72 (m, 1H), 6.63-6.62 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.07 (t, *J* = 6.4 Hz, 1H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.48 (s, 3H), 1.93-1.72 (m, 4H), 1.55-1.47 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.2, 174.0, 146.8, 138.1, 137.8, 131.9, 131.1, 129.3, 128.2, 125.6, 118.3, 113.5, 61.1, 56.5, 41.2, 32.9, 25.2, 24.0, 21.2, 14.2 ppm; IR (neat): υ $_{max}$ 3382, 3055, 2933, 1731, 1682, 1602, 1505, 750 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_3$ $[M+H]^+$ 354.2064, found 354.2063.



Ethyl 7-(3-chlorophenyl)-7-oxo-2-(phenylamino)heptanoate (5b): Light yellow oil (62%, 46.3 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.90 (s, 1H), 7.81-7.79 (m, 1H), 7.53-7.51 (m, 1H), 7.41-7.38 (m, 1H), 7.19-7.15 (m, 2H), 6.75-6.72 (m, 1H), 6.64-6.62 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.07 (t, *J* = 6.4 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 1.92-1.74 (m, 4H), 1.56-1.49 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.5, 174.0, 146.8, 138.5, 134.9, 132.9, 129.9, 129.3, 128.1, 126.1, 118.4, 113.6, 61.1, 56.5, 38.3, 32.8, 25.2, 23.7, 14.2 ppm; IR (neat): υ $_{max}$ 3386, 3045, 2939, 1732, 1688, 1603, 1507, 791, 690 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{25}ClNO_3$ $[M+H]^+$ 374.1517, found 374.1524.

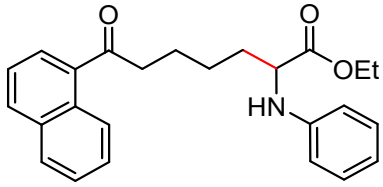


Ethyl 7-(3-fluorophenyl)-7-oxo-2-(phenylamino)heptanoate (5c): Colorless oil (54%, 38.6 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.72-7.70 (m, 1H), 7.63-7.60 (m, 1H), 7.46-7.41 (m, 1H), 7.24-7.23 (m, 1H), 7.19-7.15 (m, 2H), 6.76-6.72 (m, 1H), 6.64-6.62 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.07 (t, *J* = 6.4 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 1.92-1.75 (m, 4H), 1.57-1.49 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.6, 174.0, 162.9 (d, *J* = 246.5 Hz), 146.8, 139.0 (d, *J* = 6.0 Hz), 130.2 (d, *J* = 7.6 Hz), 129.3, 123.7 (d, *J* = 2.9 Hz), 120.0 (d, *J* = 21.3 Hz), 118.4, 114.7 (d, *J* = 22.2 Hz), 113.6, 61.1, 56.6, 38.4, 32.8, 25.2, 23.7, 14.2 ppm; IR (neat): υ $_{max}$ 3384, 3057, 2940, 1732, 1688, 1599, 1506, 796, 690 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{25}FNO_3$ $[M+H]^+$ 358.1813, found 358.1808.

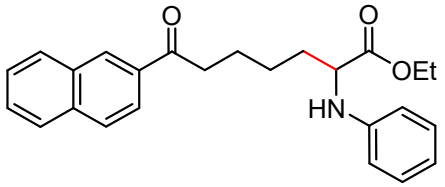


Ethyl 7-oxo-2-(phenylamino)-7-(4-(trifluoromethyl)phenyl)heptanoate (5d): Light yellow oil (47%, 38.3 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.03 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.19-7.15 (m, 2H), 6.76-6.72 (m, 1H), 6.64-6.62 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.08 (t, *J* = 6.4 Hz, 1H), 3.00 (t, *J* = 7.2 Hz, 2H), 1.97-1.78 (m, 4H), 1.58-1.52 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.9, 174.0, 146.7, 139.6, 134.3 (q, *J* = 32.2 Hz), 129.3, 128.3, 125.7 (q, *J* = 3.7 Hz), 123.6 (d, *J* = 270.9 Hz), 118.4, 113.6, 61.1, 56.5, 38.5,

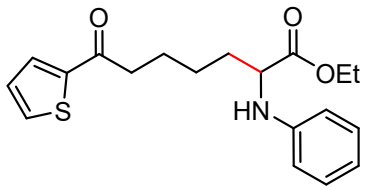
32.8, 25.2, 23.6, 14.2 ppm; IR (neat): υ $_{max}$ 3374, 3051, 2936, 1731, 1690, 1602, 1506, 750 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{25}F_3NO_3$ [M+H]$^+$ 408.1781, found 408.1781.



Ethyl 7-(naphthalen-1-yl)-7-oxo-2-(phenylamino)heptanoate (5e): Light yellow oil (52%, 40.5 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.55-8.53 (m, 1H), 7.99-7.97 (m, 1H), 7.89-7.81 (m, 2H), 7.58-7.47 (m, 3H), 7.19-7.15 (m, 2H), 6.76-6.72 (m, 1H), 6.63-6.61 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.08 (t, *J* = 6.4 Hz, 1H), 3.06 (t, *J* = 7.2 Hz, 2H), 1.95-1.77 (m, 4H), 1.60-1.52 (m, 2H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.4, 174.0, 146.8, 136.2, 133.9, 132.4, 130.1, 129.3, 128.4, 127.8, 127.2, 126.4, 125.7, 124.3, 118.3, 113.5, 61.1, 56.5, 41.9, 32.8, 25.2, 24.3, 14.2 ppm; IR (neat): υ $_{max}$ 3389, 3053, 2937, 1731, 1681, 1602, 1507, 785 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{28}NO_3$ [M+H]$^+$ 390.2064, found 390.2068.

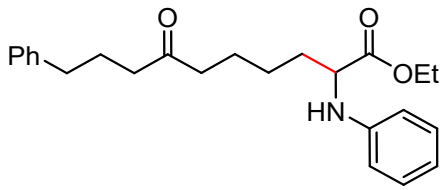


Ethyl 7-(naphthalen-2-yl)-7-oxo-2-(phenylamino)heptanoate (5f): White solid (62%, 48.2 mg); m.p.: 103-104 °C; R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.45 (s, 1H), 8.03-7.87 (m, 4H), 7.62-7.54 (m, 2H), 7.19-7.15 (m, 2H), 6.76-6.72 (m, 1H), 6.65-6.63 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.09 (t, *J* = 6.4 Hz, 1H), 3.12 (t, *J* = 7.2 Hz, 2H), 1.99-1.80 (m, 4H), 1.62-1.54 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 174.0, 146.8, 135.5, 134.3, 132.5, 129.6, 129.5, 129.3, 128.4, 128.3, 127.8, 126.7, 124.8, 118.4, 113.6, 61.1, 56.6, 38.3, 32.9, 25.3, 24.0, 14.2 ppm; IR (neat): υ $_{max}$ 3376, 3052, 2937, 1732, 1681, 1602, 1507, 787 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{28}NO_3$ [M+H]$^+$ 390.2064, found 390.2070.

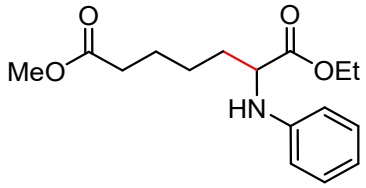


Ethyl 7-oxo-2-(phenylamino)-7-(thiophen-2-yl)heptanoate (5g): Colorless oil (65%, 44.9 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.69-7.61 (m, 2H), 7.18-7.11 (m,

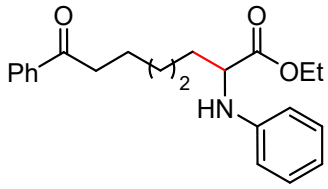
3H), 6.75-6.71 (m, 1H), 6.63-6.61 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.06 (t, *J* = 6.4 Hz, 1H), 2.91 (t, *J* = 7.2 Hz, 2H), 1.91-1.76 (m, 4H), 1.57-1.48 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.9, 174.0, 146.8, 144.3, 133.4, 131.7, 129.3, 128.0, 118.3, 113.5, 61.1, 56.5, 39.0, 32.8, 25.2, 24.2, 14.2 ppm; IR (neat): υ $_{max}$ 3384, 3054, 2936, 1730, 1658, 1602, 1509, 750 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{24}NO_3S$ $[M+H]^+$ 346.1471, found 346.1475.



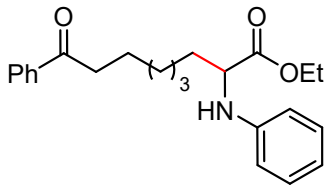
Ethyl 7-oxo-10-phenyl-2-(phenylamino)decanoate (5h): Light yellow oil (75%, 57.2 mg); R_f = 0.2 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.30-7.28 (m, 2H), 7.21-7.15 (m, 5H), 6.75-6.72 (m, 1H), 6.63-6.61 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.04 (t, *J* = 6.4 Hz, 1H), 2.61 (t, *J* = 7.2 Hz, 2H), 2.41-2.36 (m, 4H), 1.94-1.70 (m, 4H), 1.61-1.56 (m, 2H), 1.44-1.39 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 210.5, 174.0, 146.8, 141.6, 129.3, 128.4, 128.3, 125.9, 118.3, 113.5, 61.1, 56.5, 42.4, 41.9, 35.1, 32.8, 25.2, 25.1, 23.3, 14.2 ppm; IR (neat): υ $_{max}$ 3387, 3057, 2938, 1715, 1603, 1505, 750 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{32}NO_3$ $[M+H]^+$ 382.2377, found 382.2375.



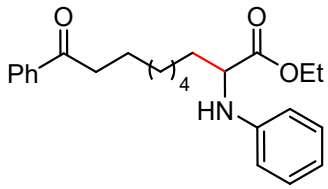
Ethyl 7-oxo-10-phenyl-2-(phenylamino)decanoate (5i): Light yellow oil (60%, 35.2 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.18-7.14 (m, 2H), 6.75-6.71 (m, 1H), 6.63-6.61 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.05 (t, *J* = 6.4 Hz, 1H), 3.66 (s, 3H), 2.32 (t, *J* = 7.2 Hz, 2H), 1.89-1.63 (m, 4H), 1.50-1.43 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 174.0, 173.8, 146.8, 129.3, 118.3, 113.5, 61.0, 56.5, 51.5, 33.7, 32.6, 25.1, 24.6, 14.2 ppm; IR (neat): υ $_{max}$ 3387, 3053, 2948, 1729, 1602, 1505, 750 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{24}NO_4$ $[M+H]^+$ 394.1700, found 394.1705.



Ethyl 8-oxo-8-phenyl-2-(phenylamino)octanoate (5j): Light yellow oil (46%, 32.5 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.19-7.15 (m, 2H), 6.75-6.71 (m, 1H), 6.63-6.61 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.05 (t, *J* = 6.4 Hz, 1H), 2.96 (t, *J* = 7.2 Hz, 2H), 1.91-1.72 (m, 4H), 1.53-1.39 (m, 4H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.2, 174.1, 146.9, 137.0, 132.9, 129.3, 128.6, 128.0, 118.3, 113.5, 61.0, 56.6, 38.3, 32.9, 29.0, 25.4, 24.0, 14.2 ppm; IR (neat): υ $_{max}$ 3385, 3056, 2935, 1732, 1682, 1506, 743, 692 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_3$ $[M+H]^+$ 354.2064, found 354.2064.

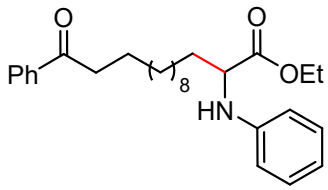


Ethyl 9-oxo-9-phenyl-2-(phenylamino)nonanoate (5k): Light yellow oil (79%, 58.0 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.94 (m, 2H), 7.57-7.53 (m, 1H), 7.48-7.44 (m, 2H), 7.19-7.15 (m, 2H), 6.75-6.71 (m, 1H), 6.63-6.61 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.05 (t, *J* = 6.4 Hz, 1H), 2.96 (t, *J* = 7.2 Hz, 2H), 1.87-1.74 (m, 4H), 1.45-1.39 (m, 6H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 174.1, 146.9, 137.0, 132.9, 129.2, 128.5, 128.0, 118.1, 113.4, 60.9, 56.6, 38.4, 32.9, 29.1, 29.0, 25.3, 24.1, 14.2 ppm; IR (neat): υ $_{max}$ 3383, 3056, 2933, 1731, 1682, 1505, 749, 692 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{30}NO_3$ $[M+H]^+$ 368.2220, found 368.2223

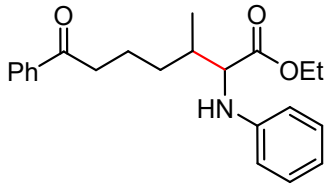


Ethyl 10-oxo-10-phenyl-2-(phenylamino)decanoate (5l): Light yellow oil (57%, 43.4 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.94 (m, 2H), 7.57-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.19-7.14 (m, 2H), 6.74-6.71 (m, 1H), 6.63-6.61 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.04 (t, *J* = 6.4 Hz, 1H), 2.95 (t, *J* = 7.2 Hz, 2H), 1.86-1.71 (m, 4H), 1.42-1.35 (m, 8H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 174.2, 146.9, 137.1, 132.9, 129.3, 128.5, 128.0, 118.2, 113.4, 61.0, 56.6, 38.5, 33.0, 29.2, 29.1, 29.0, 25.5, 24.2, 14.2 ppm; IR (neat): υ $_{max}$ 3375, 3055,

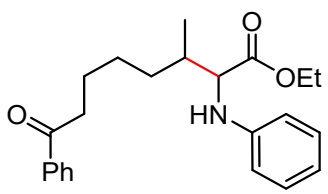
2932, 1733, 1684, 1508, 752, 693 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{32}NO_3$ $[M+H]^+$ 382.2377, found 382.2379.



Ethyl 14-oxo-14-phenyl-2-(phenylamino)tetradecanoate (5m): Light yellow oil (76%, 66.4 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.57-7.53 (m, 1H), 7.48-7.44 (m, 2H), 7.19-7.15 (m, 2H), 6.75-6.71 (m, 1H), 6.64-6.62 (m, 2H), 4.18 (q, *J* = 7.2 Hz, 2H), 4.04 (t, *J* = 6.4 Hz, 1H), 2.96 (t, *J* = 7.2 Hz, 2H), 1.88-1.70 (m, 4H), 1.42-1.23 (m, 19H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 174.2, 146.9, 137.1, 132.8, 129.3, 128.5, 128.0, 118.2, 113.5, 60.9, 56.7, 38.6, 33.0, 29.5 (2C), 29.4 (2C), 29.3 (2C), 25.5, 24.2, 14.2 ppm; IR (neat): υ_{max} 3386, 3056, 2925, 1733, 1684, 1507, 750, 692 cm^{-1}; HRMS (ESI) calcd for $C_{28}H_{40}NO_3$ $[M+H]^+$ 438.3003, found 438.3006.

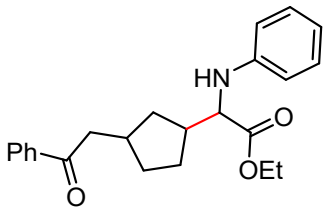


Ethyl 3-methyl-7-oxo-7-phenyl-2-(phenylamino)heptanoate (5n): Light yellow oil (73%, 51.5 mg, d.r. = 1:1); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.43 (m, 2H), 7.19-7.15 (m, 2H), 6.75-6.71 (m, 1H), 6.65-6.63 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 4.02 (d, *J* = 4.8 Hz, 0.5H), 3.94 (d, *J* = 6.0 Hz, 0.5H), 3.01-2.94 (m, 2H), 2.08-1.57 (m, 4H), 1.40-1.33 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.04 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.0, 173.6, 173.4, 147.3, 147.1, 137.0, 133.0, 129.3, 128.6, 128.0, 118.3, 118.2, 113.7, 113.6, 61.4, 60.9 (2C), 38.5, 36.4, 36.1, 32.9, 32.5, 21.9, 21.7, 16.0, 15.3, 14.3 ppm; IR (neat): υ_{max} 3371, 3054, 2935, 1731, 1684, 1507, 786, 693 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{28}NO_3$ $[M+H]^+$ 354.2064, found 354.2066.

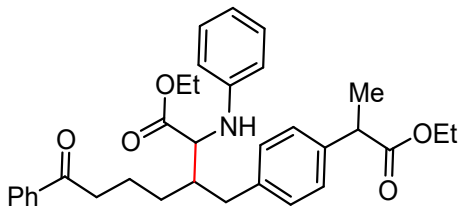


Ethyl 3-methyl-8-oxo-8-phenyl-2-(phenylamino)octanoate (5o): Light yellow oil (70%, 51.4 mg, d.r.

= 1:1); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.93 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.44 (m, 2H), 7.19-7.15 (m, 2H), 6.75-6.71 (m, 1H), 6.65-6.63 (m, 2H), 4.17 (q, *J* = 7.2 Hz, 2H), 3.98 (d, *J* = 4.8 Hz, 0.5H), 3.93 (d, *J* = 6.0 Hz, 0.5H), 3.00-2.95 (m, 2H), 2.05-1.29 (m, 7H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.00 (dd, *J* = 9.2 Hz, 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 173.7, 173.4, 147.4, 147.1, 137.0, 132.9, 129.3, 128.6, 128.0, 118.2, 118.1, 113.7, 113.6, 61.4, 60.9 (2C), 60.8, 38.4, 36.2, 36.0, 33.1, 32.7, 26.9, 26.8, 24.3, 24.2, 15.9, 15.3, 14.3 ppm; IR (neat): υ $_{max}$ 3371, 3056, 2934, 1731, 1683, 1507, 752, 693 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{30}NO_3$ $[M+H]^+$ 368.2220, found 368.2223.



Ethyl 2-(3-(2-oxo-2-phenylethyl)cyclopentyl)-2-(phenylamino)acetate (5p): Light yellow oil (57 %, 41.6 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.44 (m, 2H), 7.18-7.14 (m, 2H), 6.75-6.71 (m, 1H), 6.64-6.62 (m, 2H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.89 (d, *J* = 7.6 Hz, 1H), 3.04-2.99 (m, 2H), 2.49-2.37 (m, 2H), 2.19-1.44 (m, 6H), 1.31-1.20 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =199.9, 199.7, 174.0, 173.9, 173.8, 147.3, 147.28, 147.21, 147.17, 137.1, 133.0, 129.2, 128.6, 128.0, 118.3, 113.6, 113.5, 60.9, 60.7, 44.8, 44.7, 44.6, 44.5, 42.8, 42.7, 41.9, 41.8, 36.5, 35.8, 35.4, 35.0, 34.9, 34.7, 33.0, 32.6, 31.5, 29.4, 29.1, 27.8, 27.6, 14.2 ppm; IR (neat): υ $_{max}$ 3379, 3027, 2950, 1729, 1681, 1506, 750, 693 cm^{-1}; HRMS (ESI) calcd for $C_{23}H_{28}NO_3$ $[M+H]^+$ 366.2064, found 366.2064.



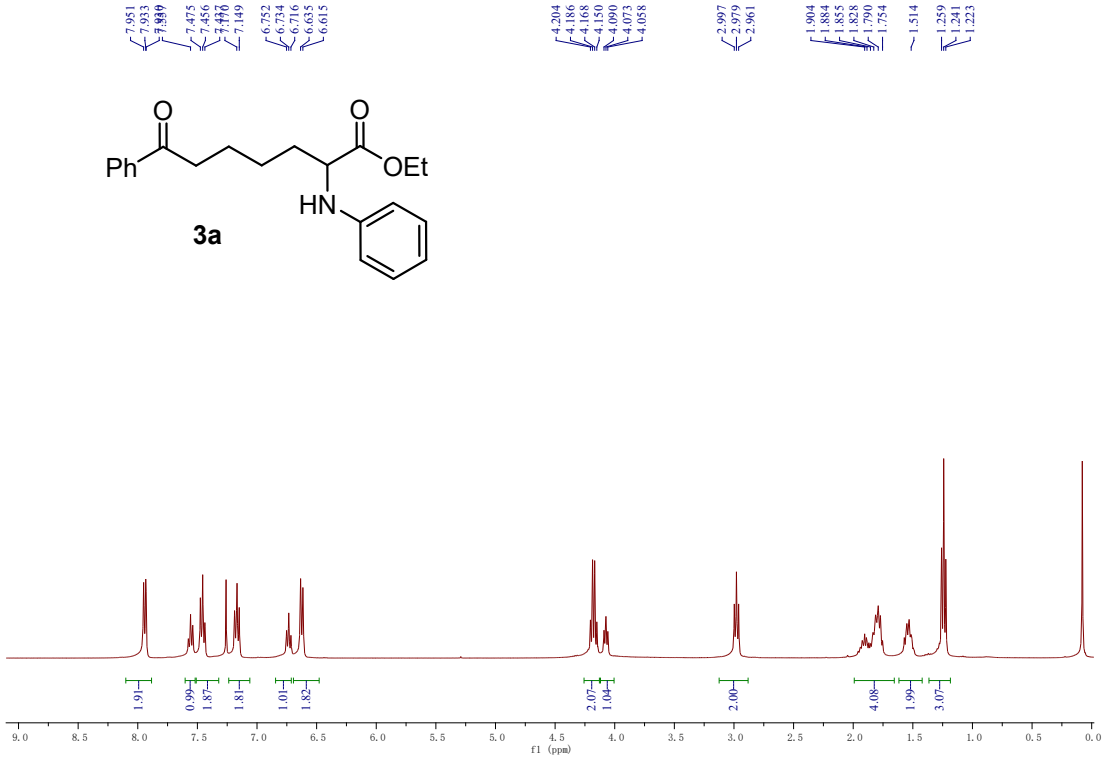
ethyl 3-(4-(1-ethoxy-1-oxopropan-2-yl)benzyl)-7-oxo-7-phenyl-2-(phenylamino)heptanoate (5q): Light yellow oil (52%, 55.0 mg); R_f = 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.89 (m, 2H), 7.58-7.53 (m, 1H), 7.46-7.42 (m, 2H), 7.22-7.05 (m, 6H), 6.75-6.66 (m, 1H), 6.55-6.35 (m, 2H), 4.22-4.06 (m, 5H), 3.74-3.66 (m, 1H), 2.97-2.90 (m, 2H), 2.79-2.67 (m, 2H), 1.89-1.82 (m, 2H), 1.63-1.45 (m, 6H), 1.32-1.19 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =199.9,

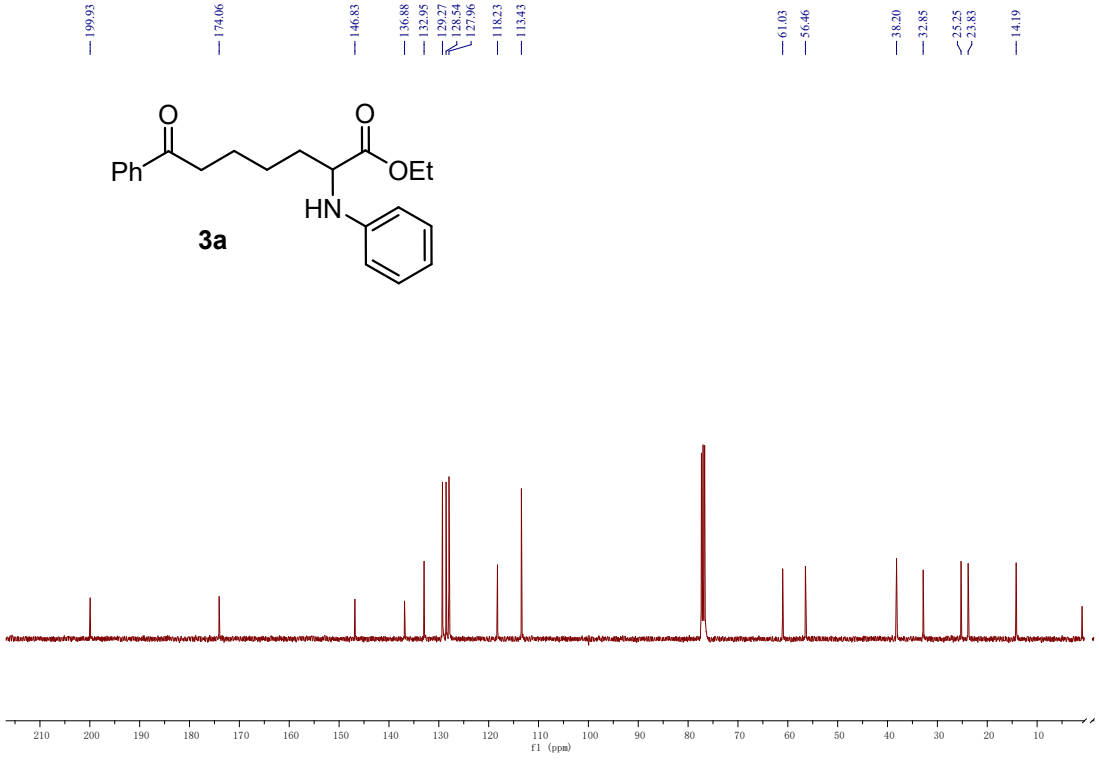
199.8, 174.6, 173.7, 173.0, 147.2, 147.28, 138.7, 138.5,136.9, 133.0, 132.9, 129.7, 129.4, 129.2, 128.6, 128.5, 128.0, 127.5, 127.4, 118.4, 118.1, 113.9, 113.5, 61.2, 61.0, 60.7, 58.3, 57.7, 45.1, 43.4, 42.9, 38.5, 36.7, 36.0, 29.6, 29.5, 22.0, 21.8, 18.5, 14.3, 14.2, 14.1 ppm; IR (neat): υ_{max} 3367, 3055, 2977, 1729, 1685, 1509, 752, 694 cm^{-1}; HRMS (ESI) calcd for $C_{33}H_{40}NO_5$ $[M+H]^+$ 530.2901, found 530.2904.

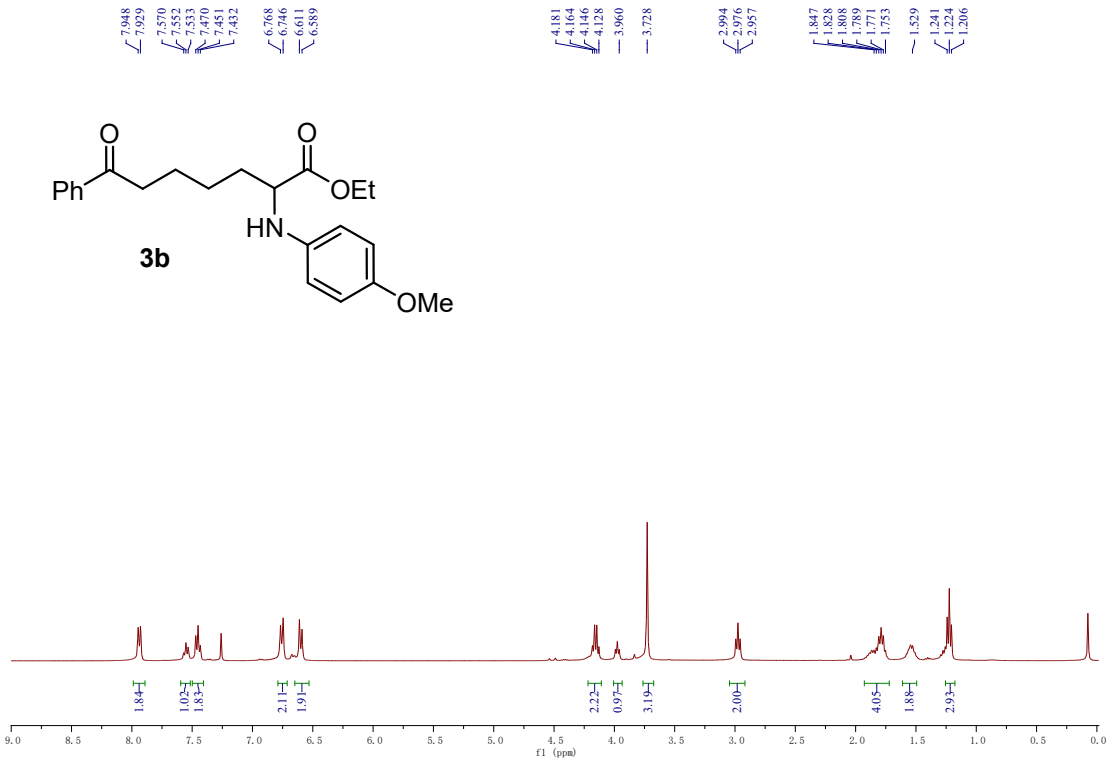
References

1. Q.-X. Sun, H. Chen, S. Liu, X.-Q. Wang, X.-H. Duan and L.-N. Guo, *J. Org. Chem.*, 2021, **86**, 11987.
2. H. Tian, W. Xu, Y. Liu and Q. Wang, *Org. Lett.*, 2020, **22**, 5005.
3. (a) V. K. Soni, H. S. Hwang, Y. K. Moon, S.-W. Park, Y.-M. You and E. J. Cho, *J. Am. Chem. Soc.*, 2019, **141**, 10538. (b) Q. Yu, Y.-T. Zhang and J.-P. Wan, *Green Chem.*, 2019, **21**, 3436.

^{1}H NMR and ^{13}C NMR Spectra of Products 2, 3, 4 and 5



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1.828
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1.241
1.224
1.206
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O
Ph
OEt
HN
3b
OMe
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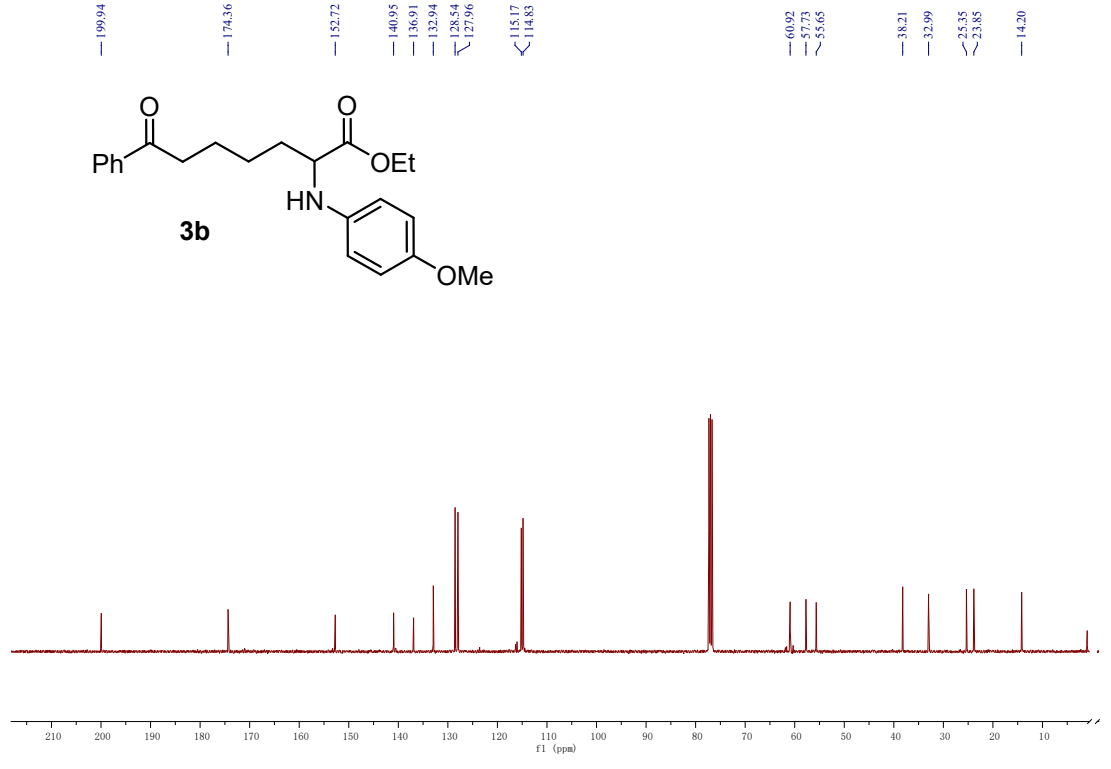
f1 (ppm)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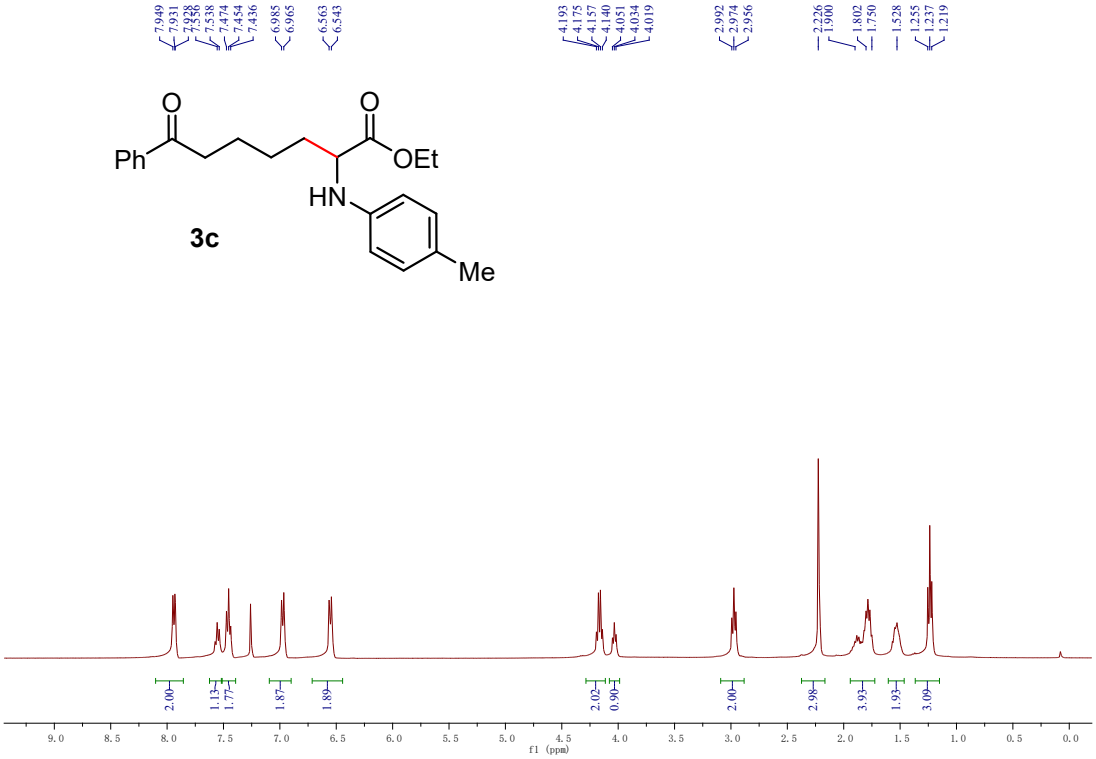
Ph
OEt
HN
3b
OMe
f1 (ppm)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O
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HN
Me
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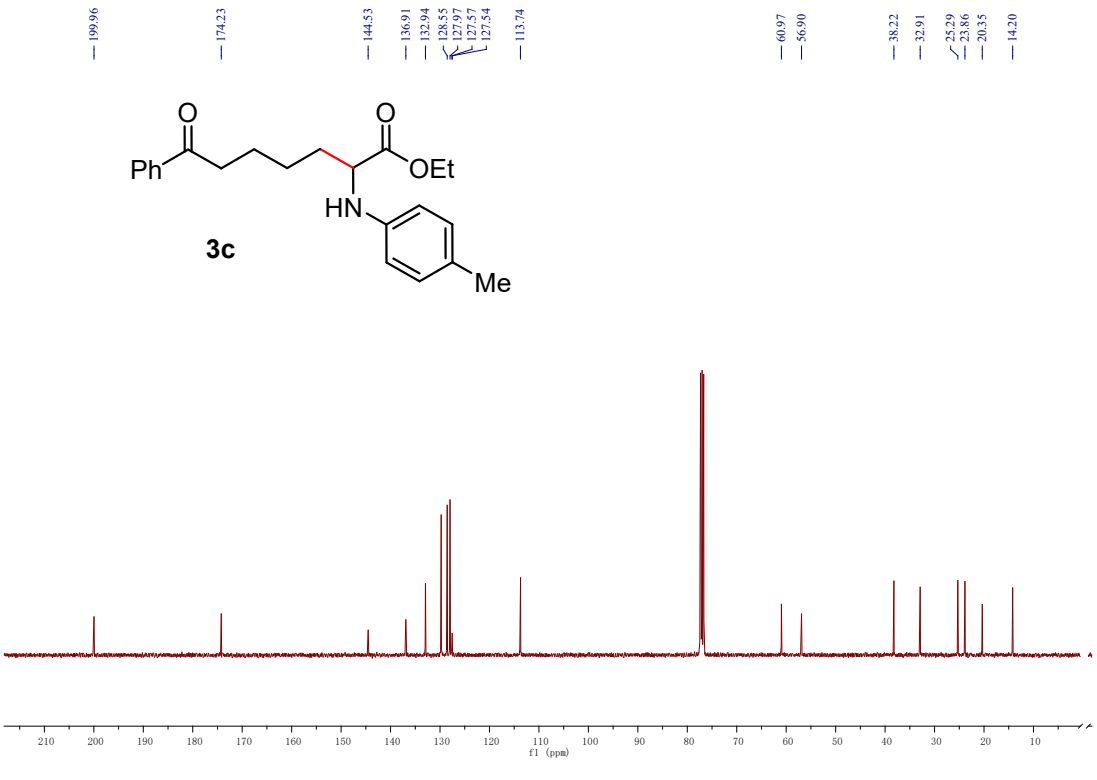
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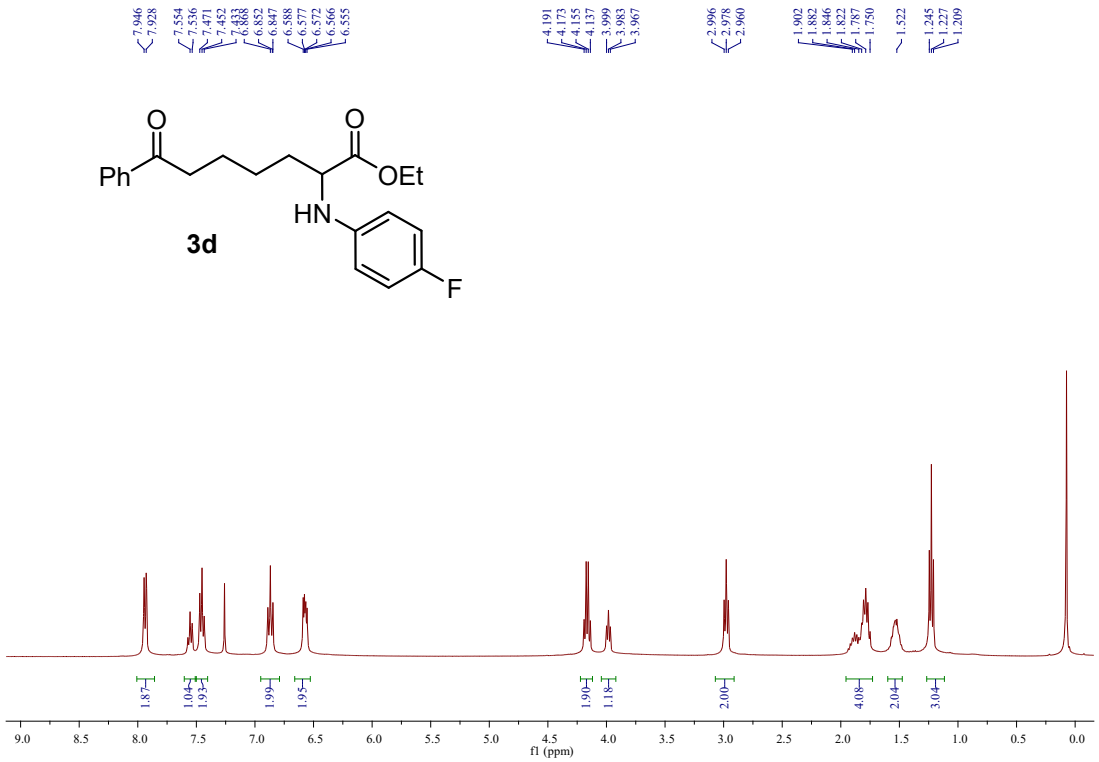
O
Ph
OEt
HN
Me
3c
f1 (ppm)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O
Ph
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HN
F
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8.0
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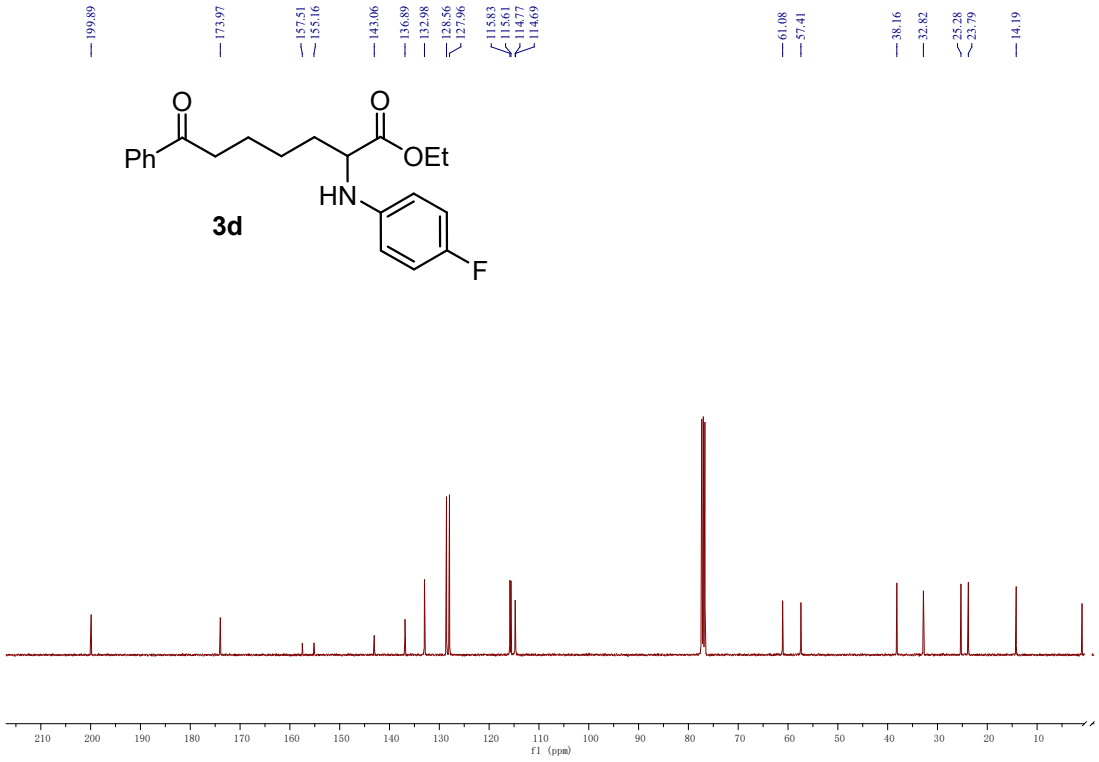
f1 (ppm)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57.41
38.16
32.82
25.28
23.79
14.19
O
O
Ph
OEt
HN
F
3d
210
200
190
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120
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100
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60
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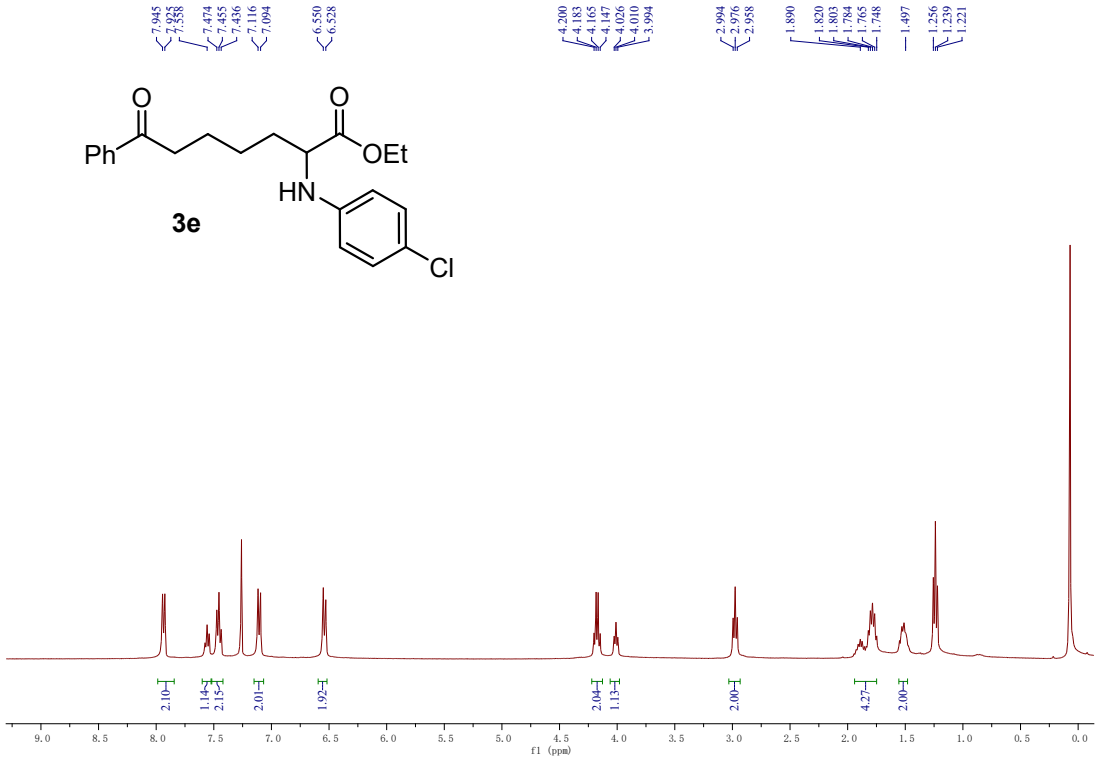
f1 (ppm)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2.958
1.890
1.820
1.803
1.784
1.765
1.748
1.497
1.256
1.239
1.221
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O
Ph
OEt
HN
Cl
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2.00
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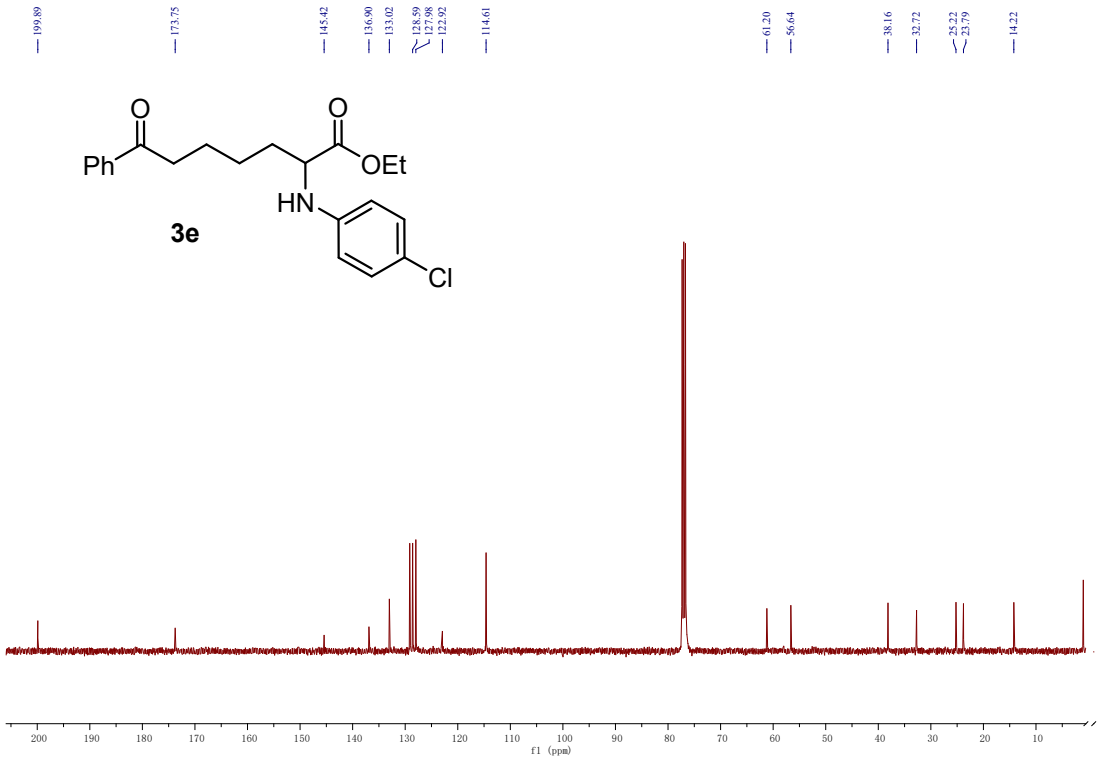
2.00
f1 (ppm)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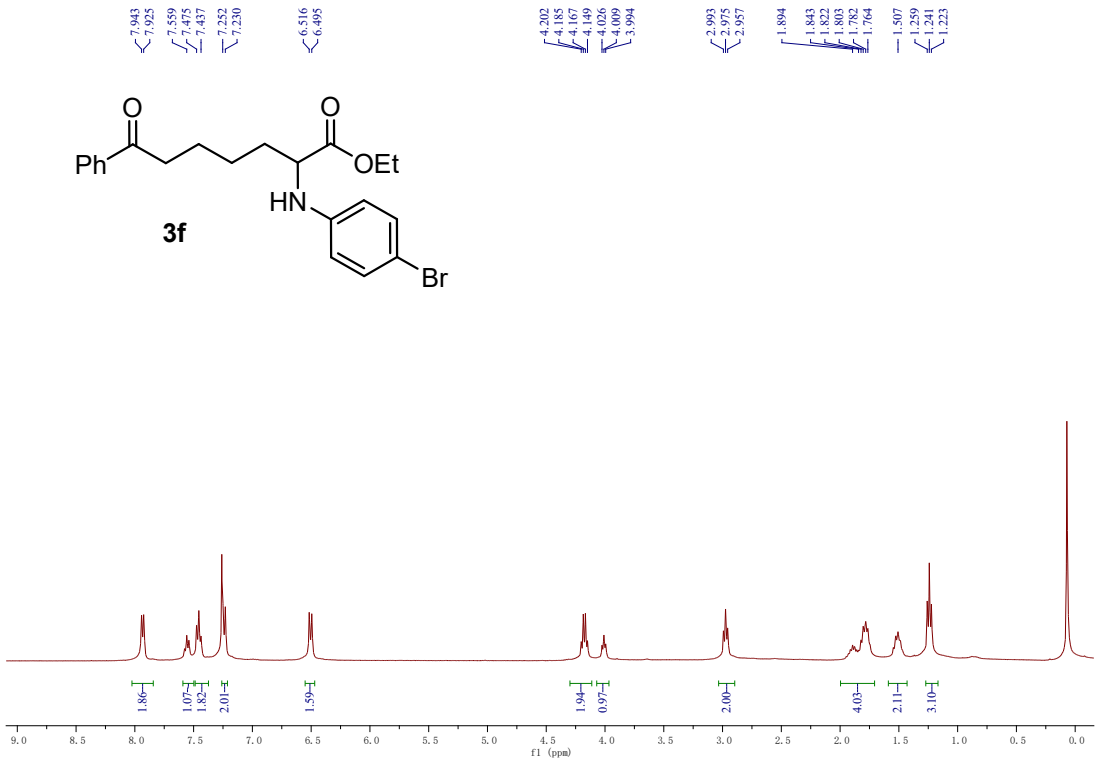
Ph
OEt
HN
Cl
3e
f1 (ppm)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4.009
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1.822
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1.507
1.259
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1.223
O
O
Ph
OEt
HN
Br
3f
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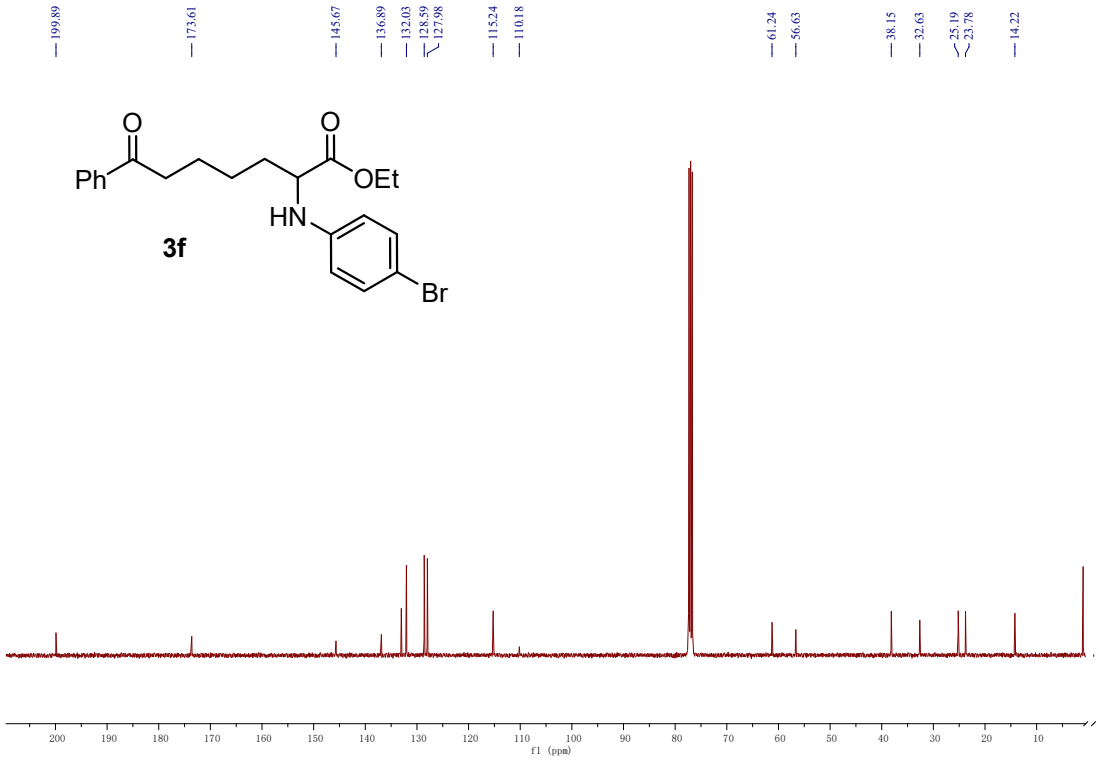
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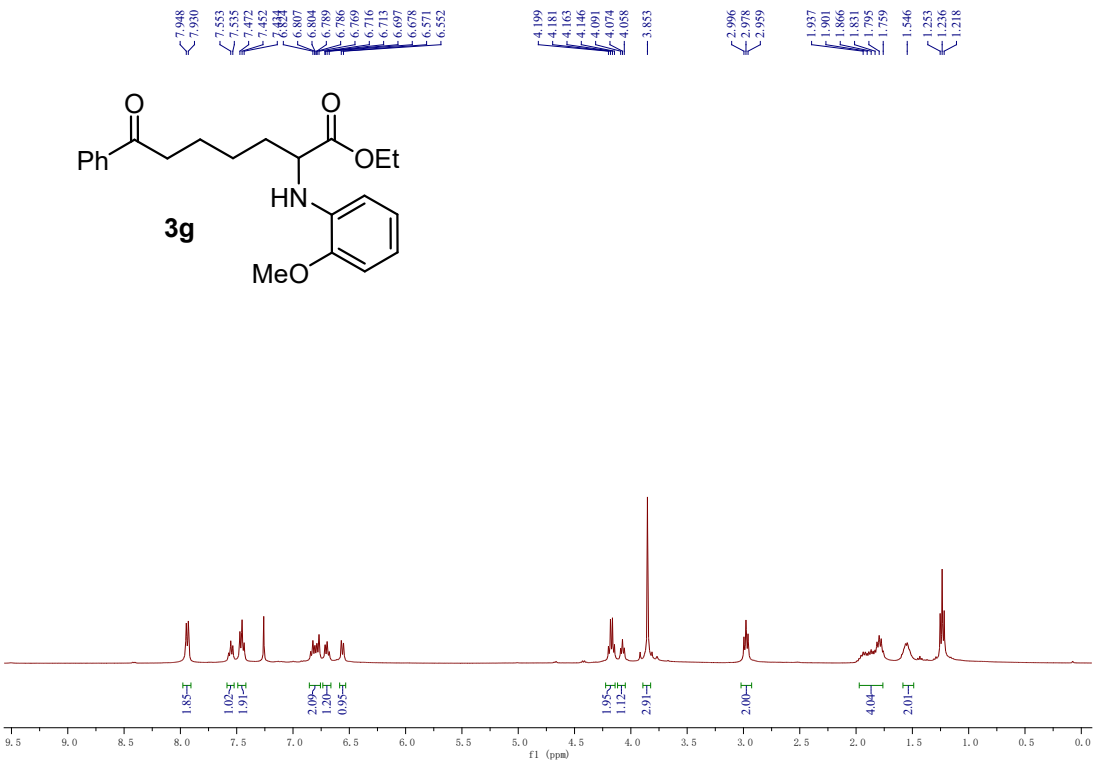
Ph
OEt
HN
Br
3f
f1 (ppm)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4.058
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1.795
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1.253
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1.218
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O
Ph
OEt
HN
MeO
3g
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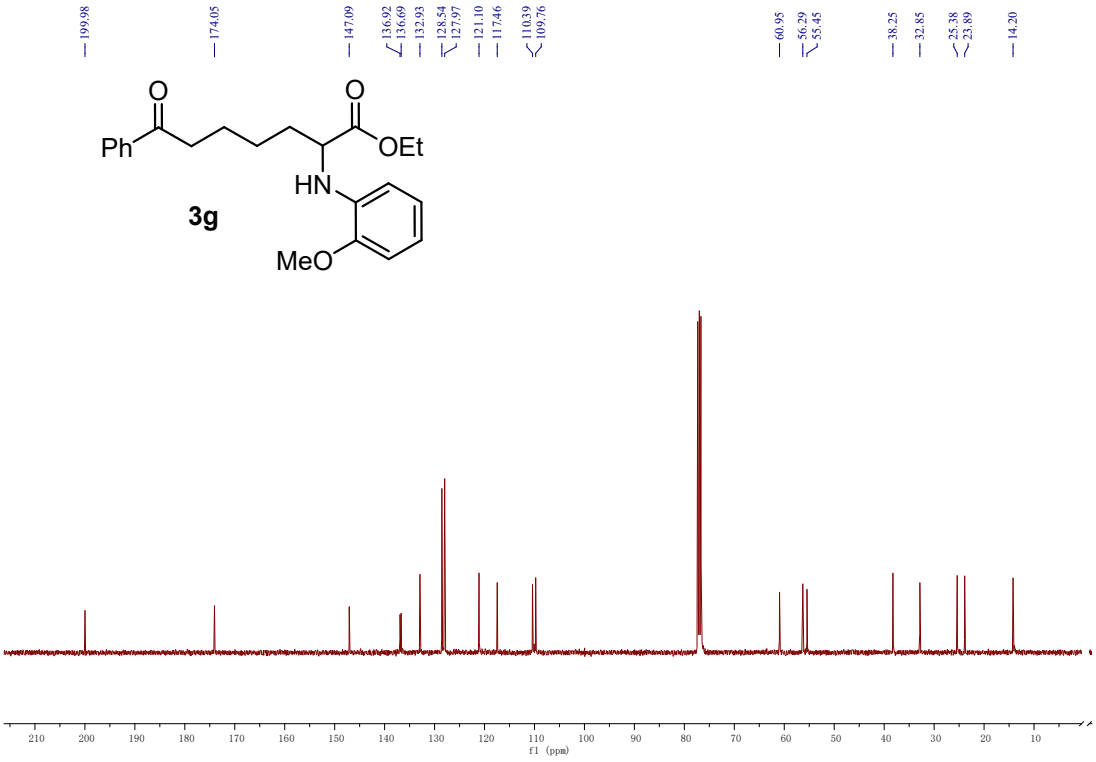
f1 (ppm)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60.95
56.29
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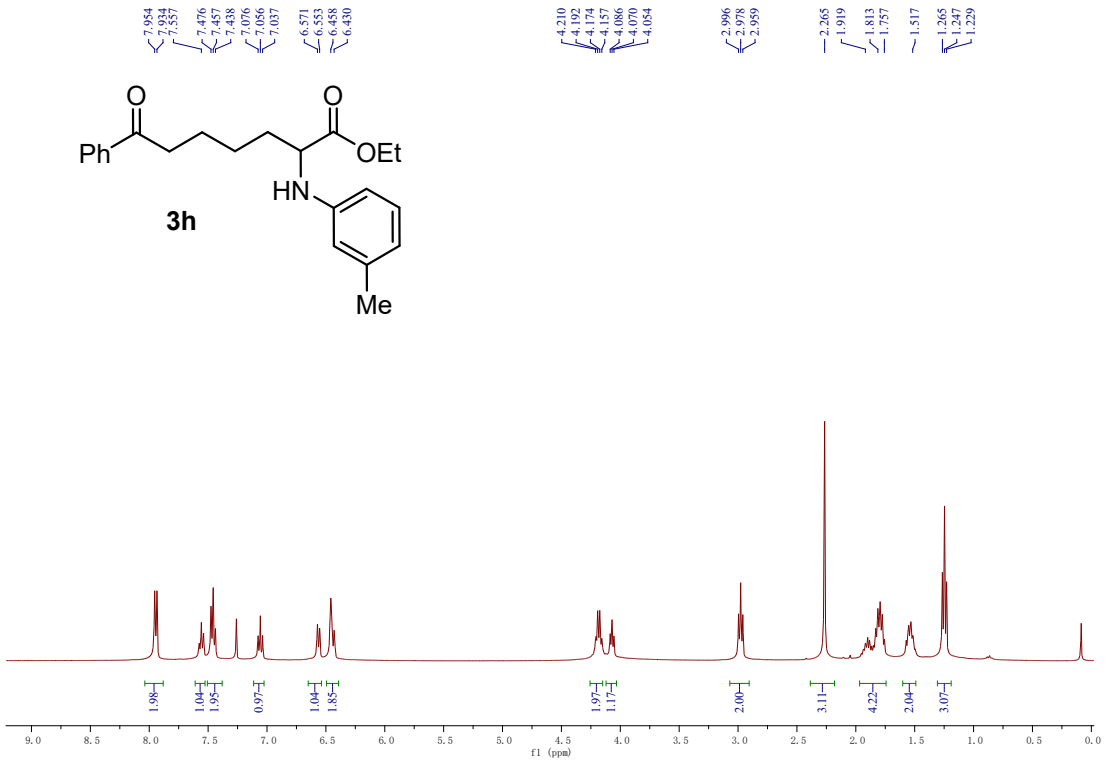
Ph
OEt
HN
MeO
3g
f1 (ppm)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2.959
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1.813
1.757
1.517
1.265
1.247
1.229
O
O
Ph
OEt
HN
3h
Me
1.98
1.04
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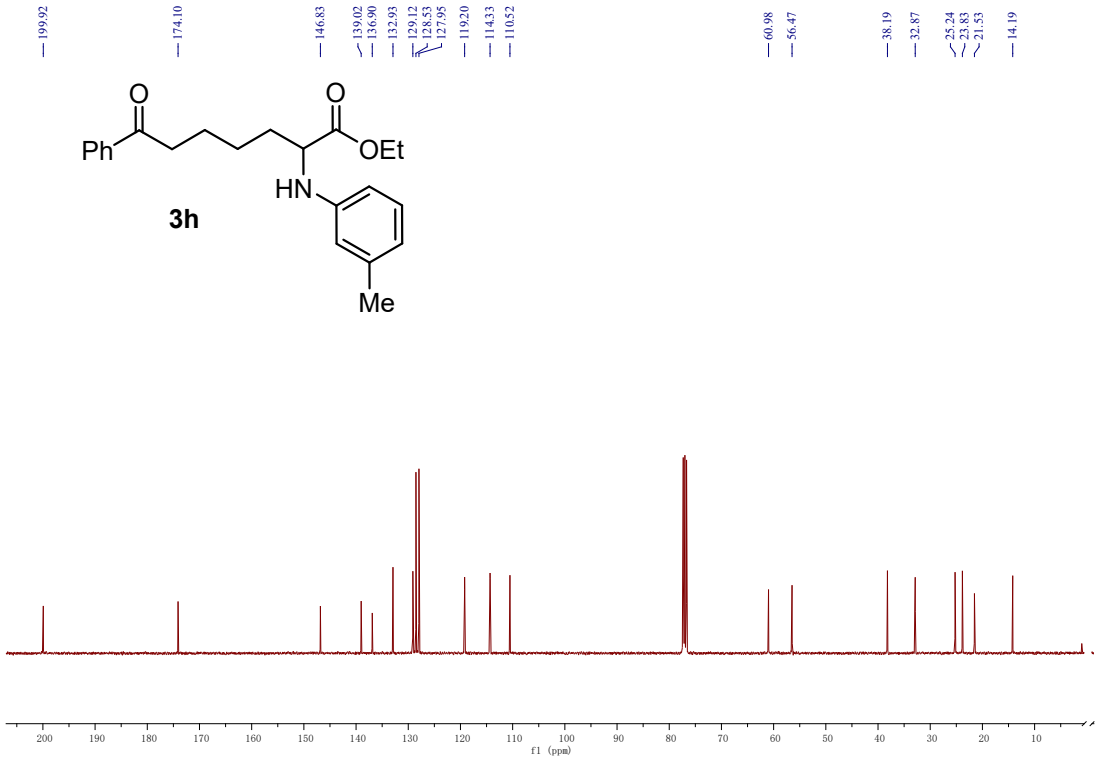
f1 (ppm)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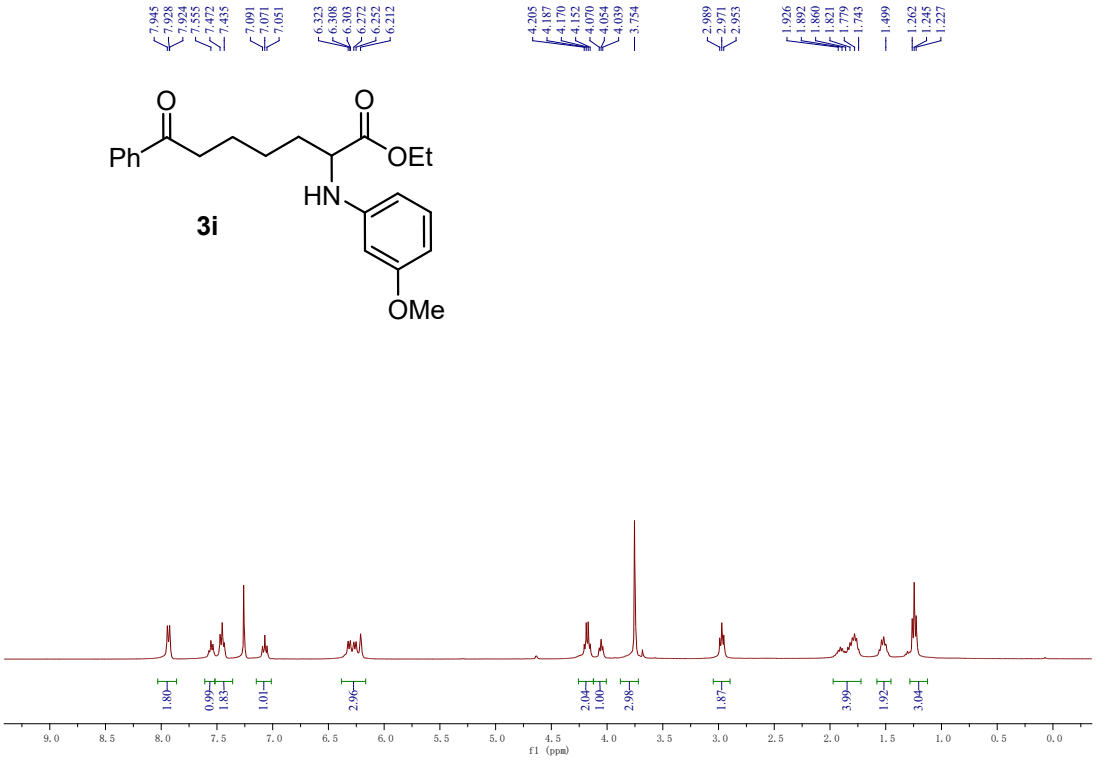
Ph
OEt
HN
3h
Me
f1 (ppm)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1.892
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1.262
1.245
1.227
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O
Ph
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HN
OMe
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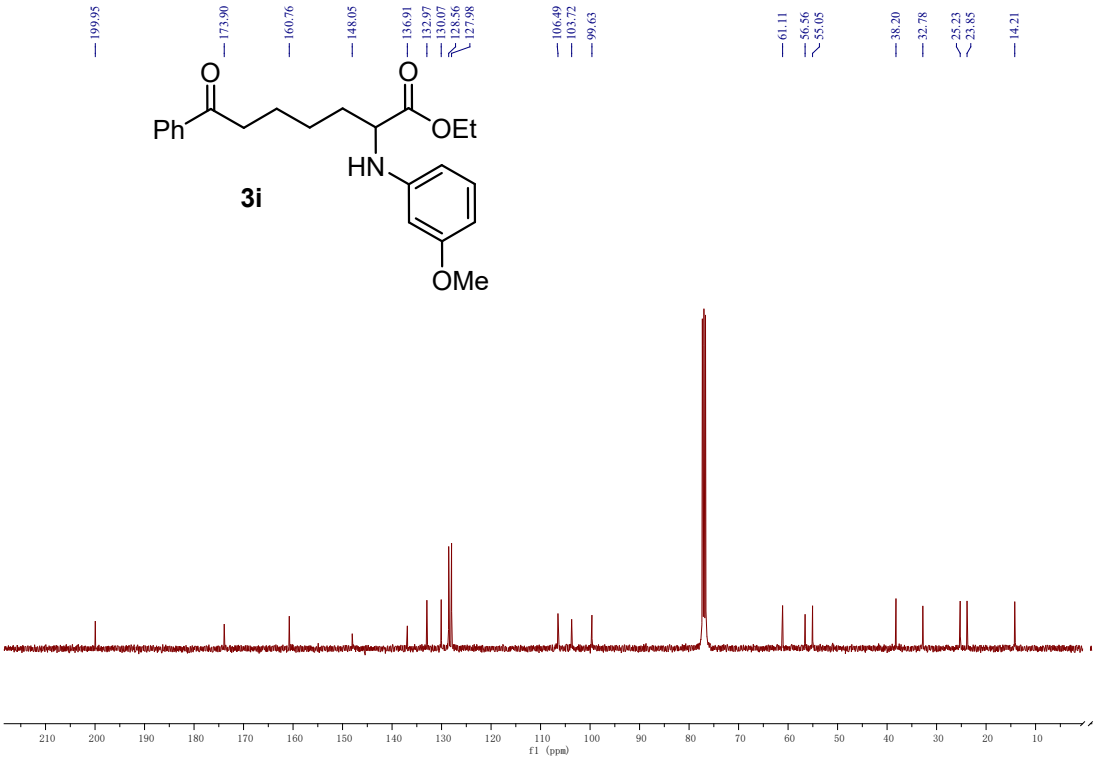
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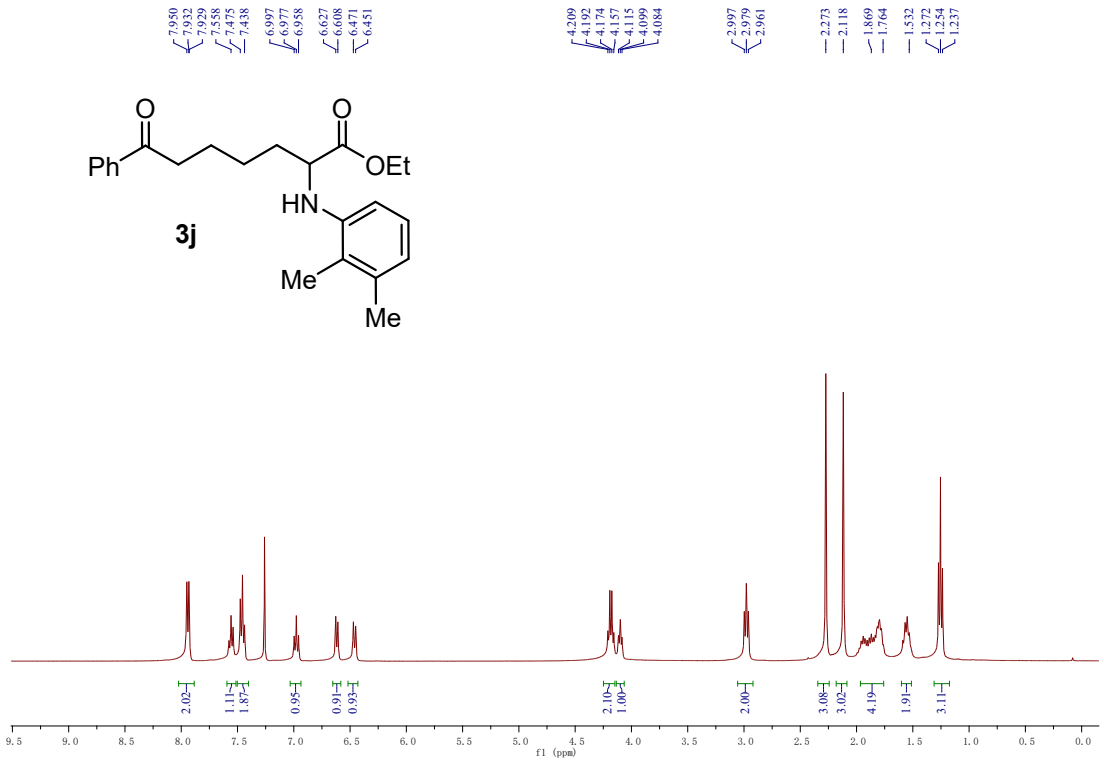
O
Ph
OEt
HN
OMe
3i
f1 (ppm)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Me
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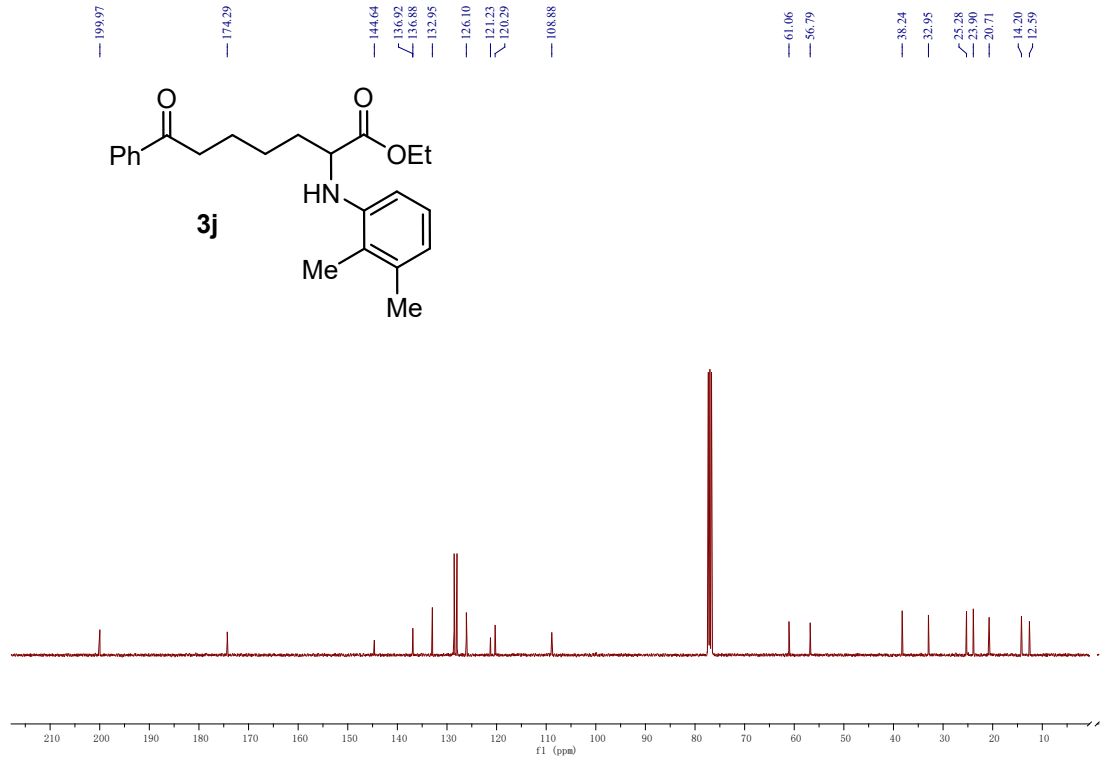
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OEt
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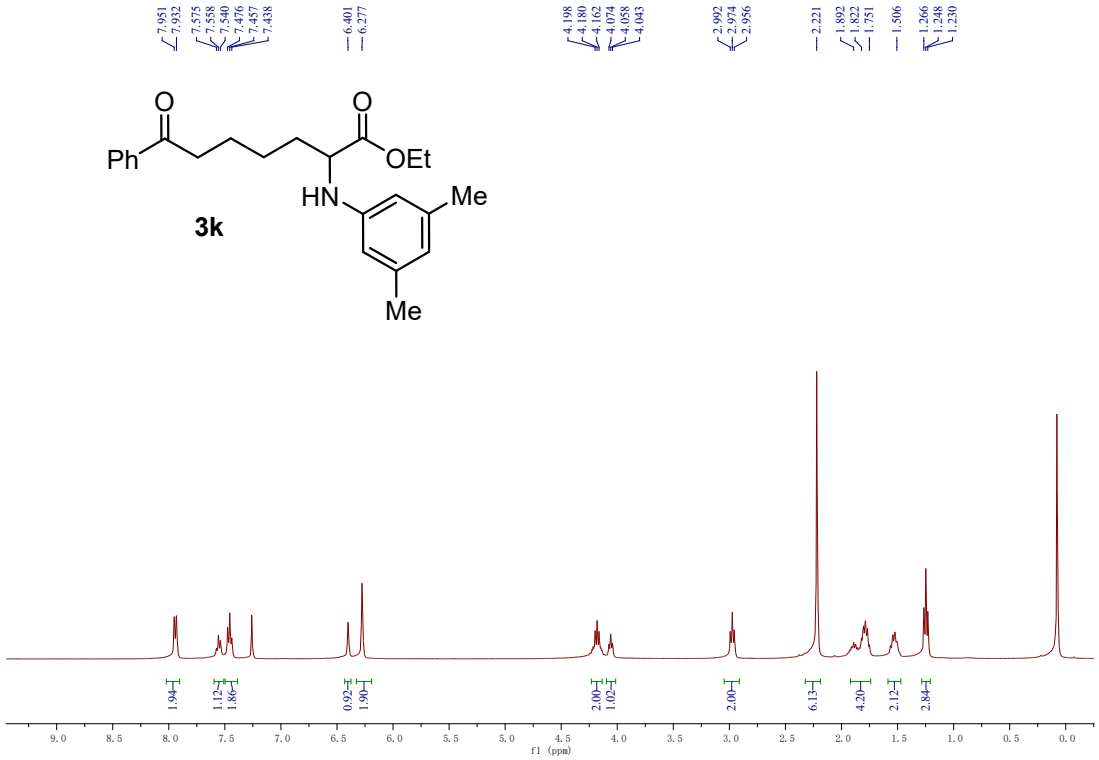
f1 (ppm)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1.266
1.248
1.230
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O
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OEt
HN
Me
Me
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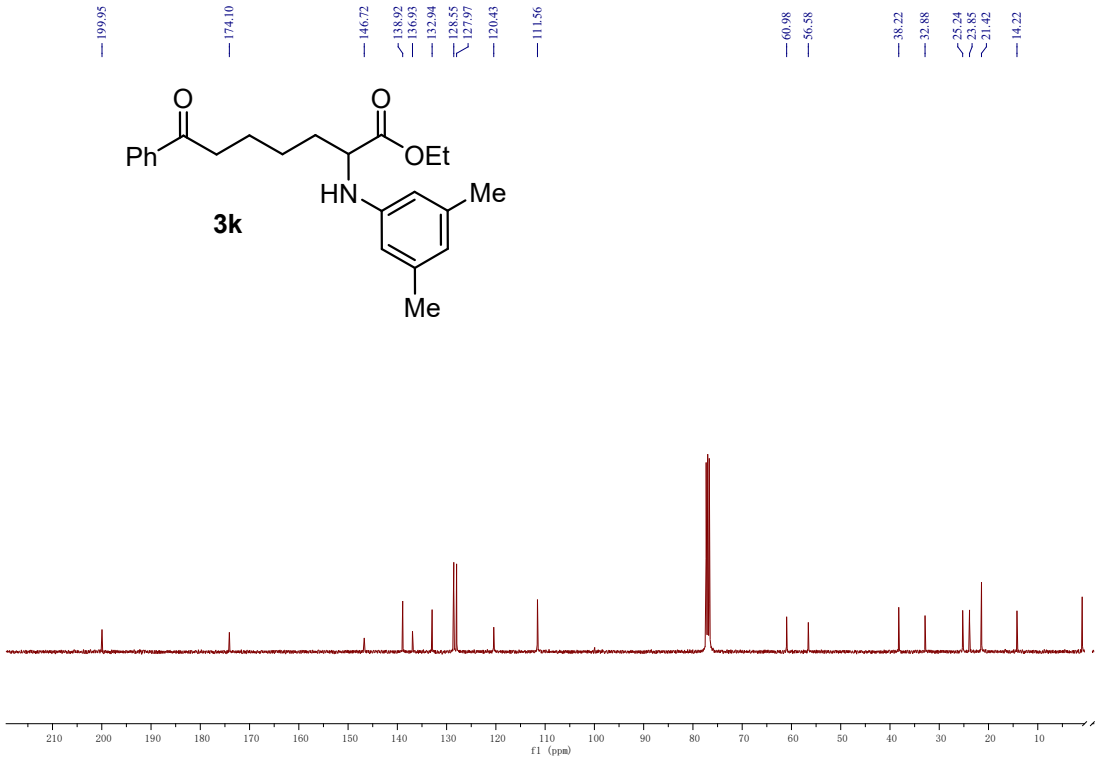
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OEt
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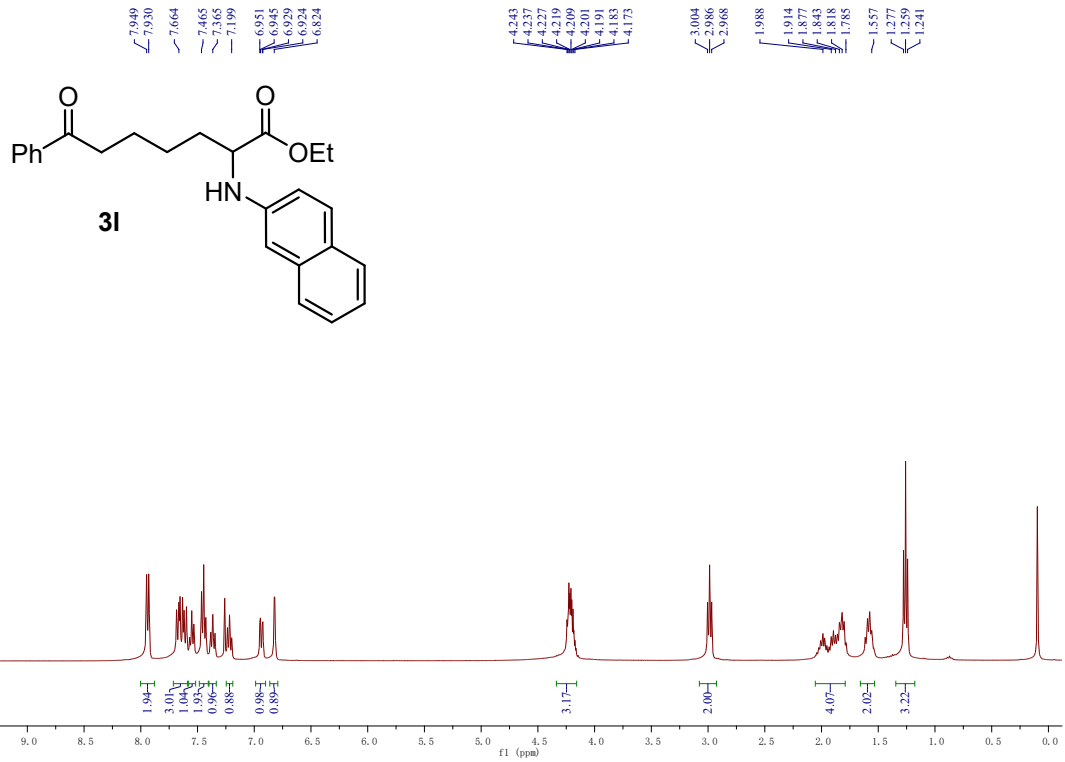
3k
f1 (ppm)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1.259
1.241
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O
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3l
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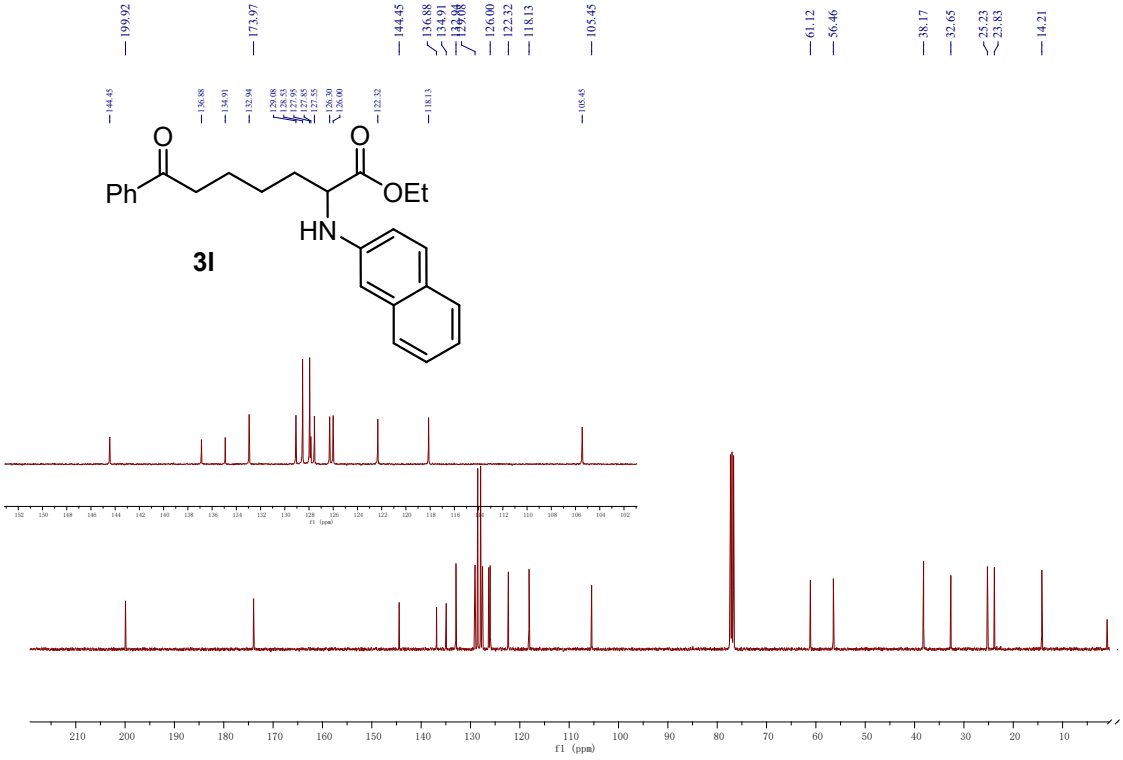
f1 (ppm)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122.32
118.13
105.45
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25.23
23.83
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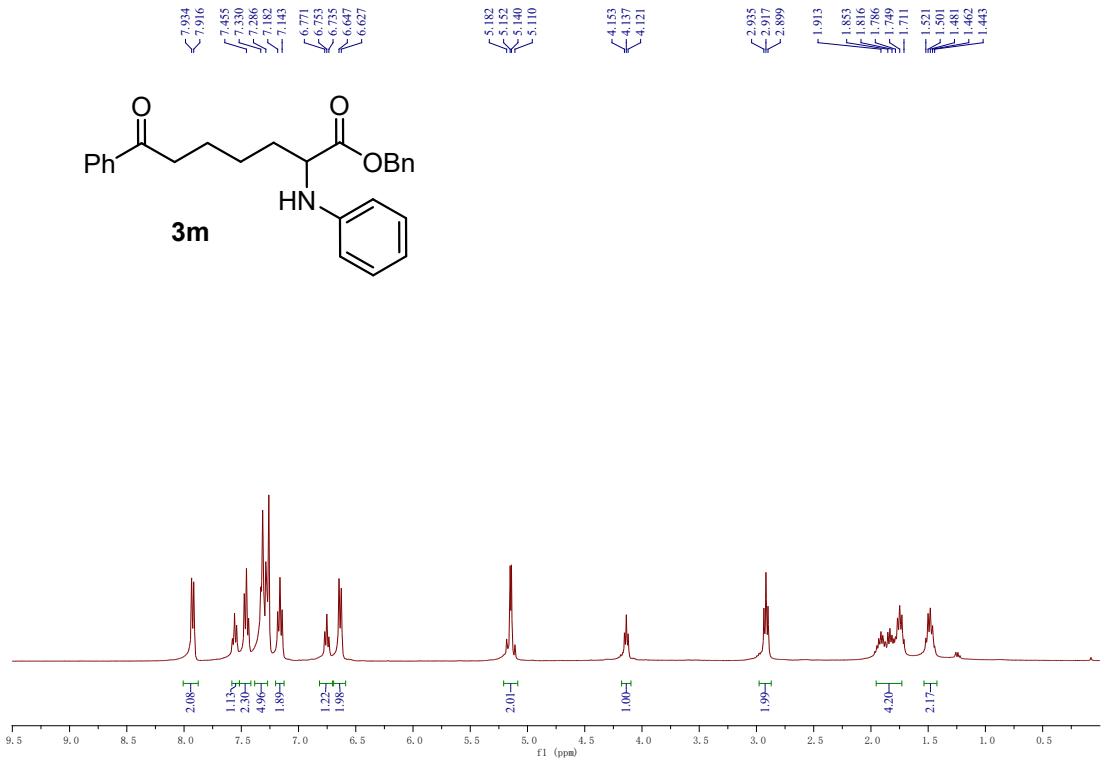
O
Ph
OEt
HN
3l
f1 (ppm)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O
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OBn
HN
3m
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1.00
1.99
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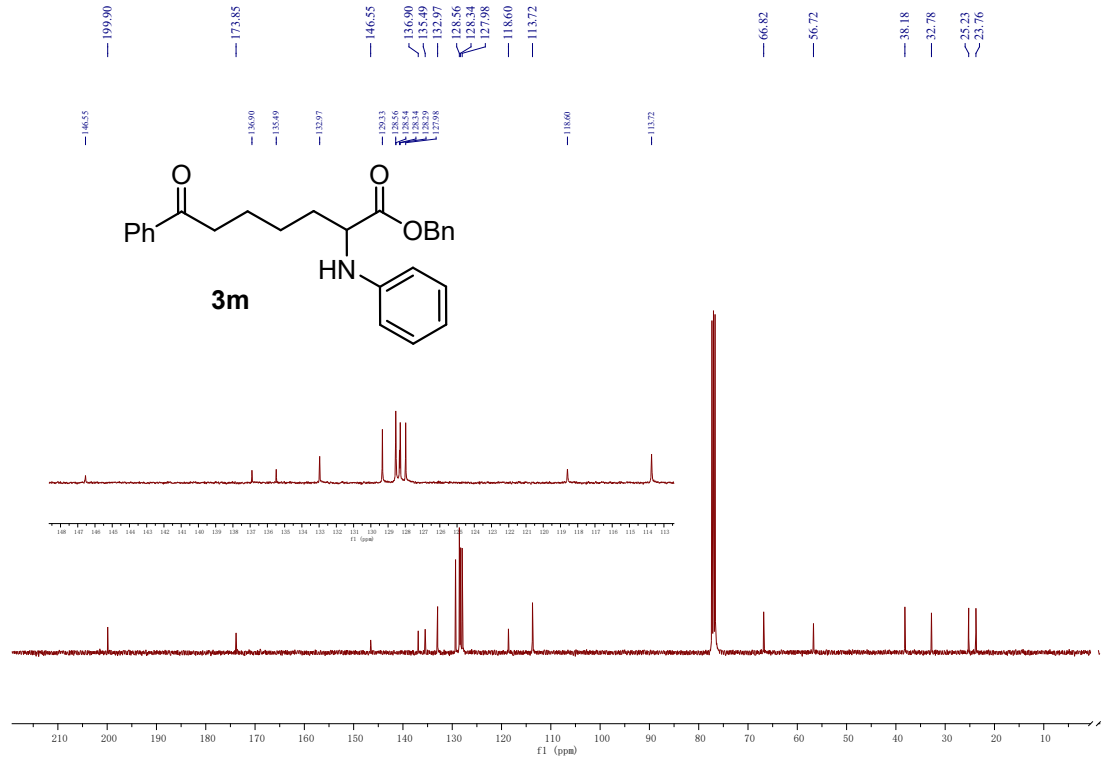
f1 (ppm)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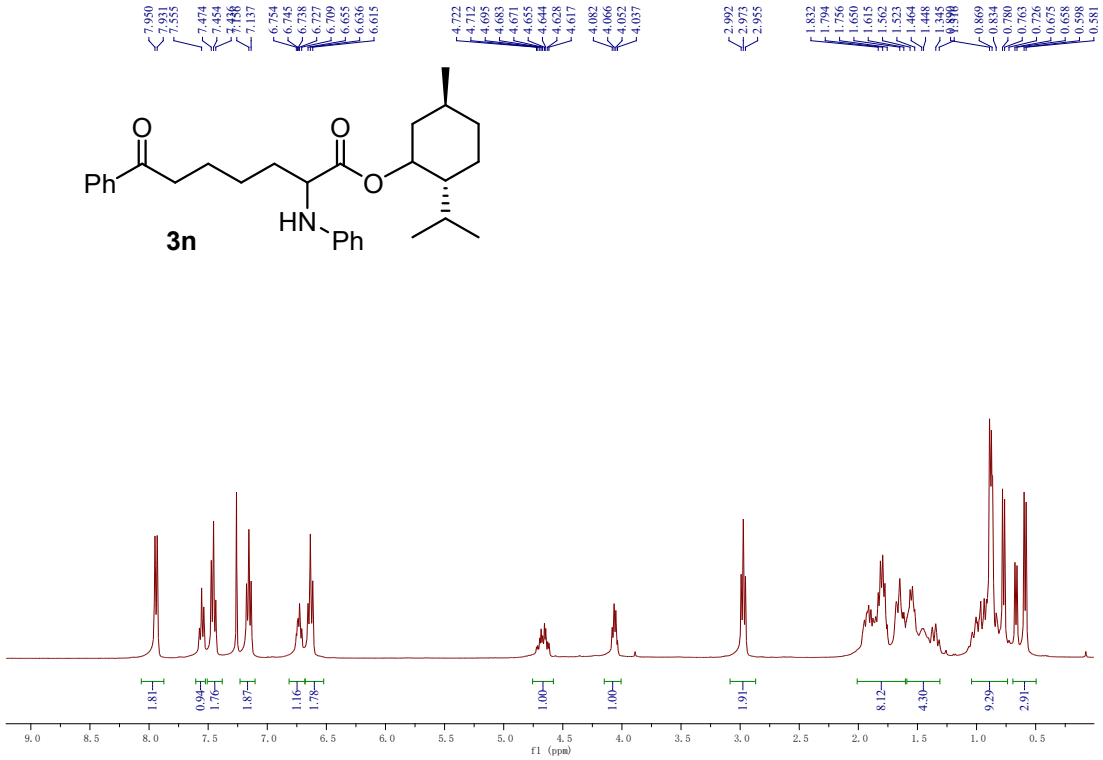
56.72
38.18
32.78
25.23
23.76
O
O
Ph
OBn
HN
3m
f1 (ppm)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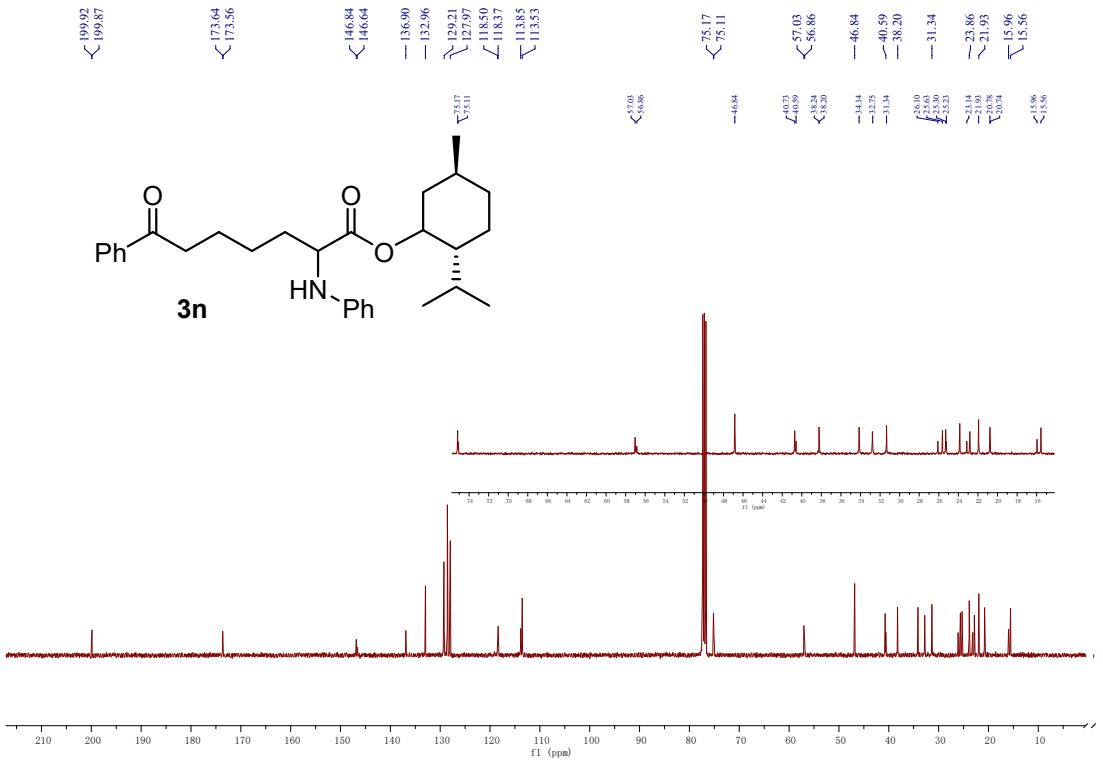
6.727
6.709
6.655
6.636
6.615
4.722
4.712
4.695
4.683
4.671
4.655
4.644
4.628
4.617
4.082
4.066
4.052
4.037
2.992
2.973
2.955
1.832
1.794
1.756
1.650
1.615
1.562
1.523
1.464
1.448
1.345
0.869
0.834
0.780
0.763
0.726
0.675
0.658
0.598
0.581
O
O
Ph
O
HN
Ph
3n
1.81
0.94
1.76
1.87
1.16
1.78
1.00
1.00
1.91
8.12
4.30
9.29
2.91
f1 (ppm)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199.87
173.64
173.56
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146.64
136.90
132.96
129.21
127.97
118.50
118.37
113.85
113.53
75.17
75.11
57.03
56.86
46.84
40.59
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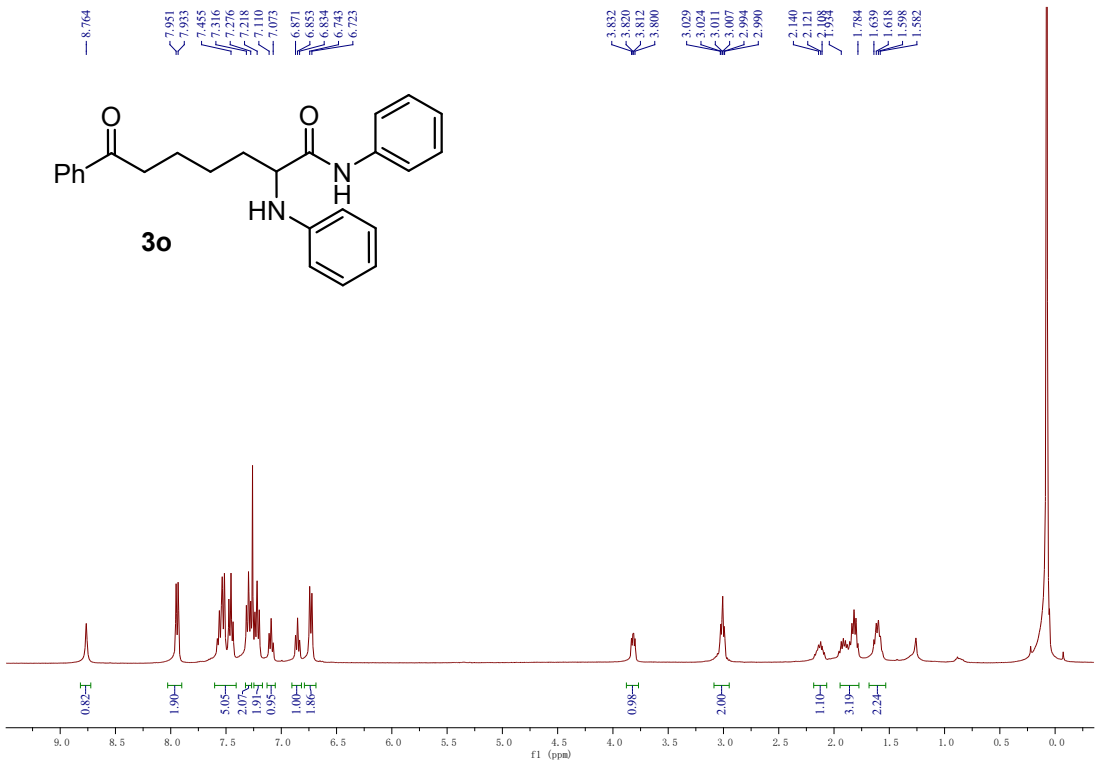
23.86
21.93
15.96
15.56
O
O
Ph
O
HN
Ph
3n
f1 (ppm)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6.834
6.743
6.723
3.832
3.820
3.812
3.800
3.029
3.024
3.011
3.007
2.994
2.990
2.140
2.121
2.108
1.994
1.784
1.639
1.618
1.598
1.582
O
O
Ph
N
H
HN
3o
0.82
1.90
5.05
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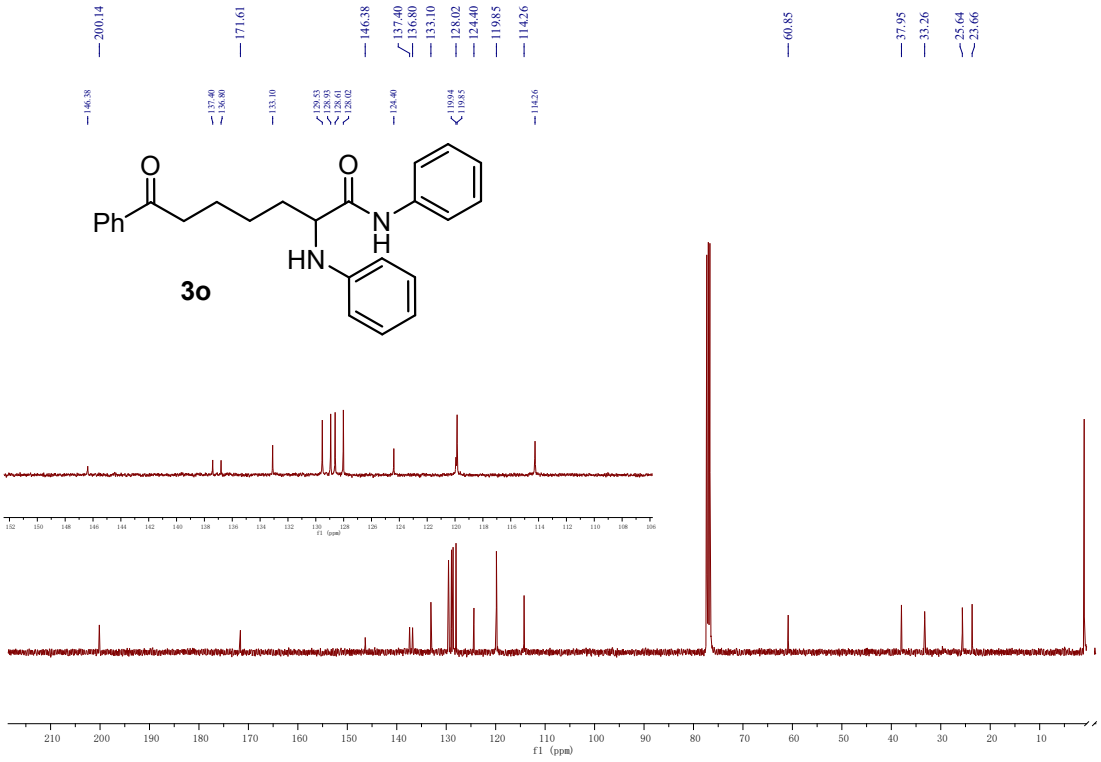
0.95
1.00
1.86
0.98
2.00
1.10
3.19
2.24
f1 (ppm)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146.38
137.40
136.80
133.10
128.02
124.40
119.85
114.26
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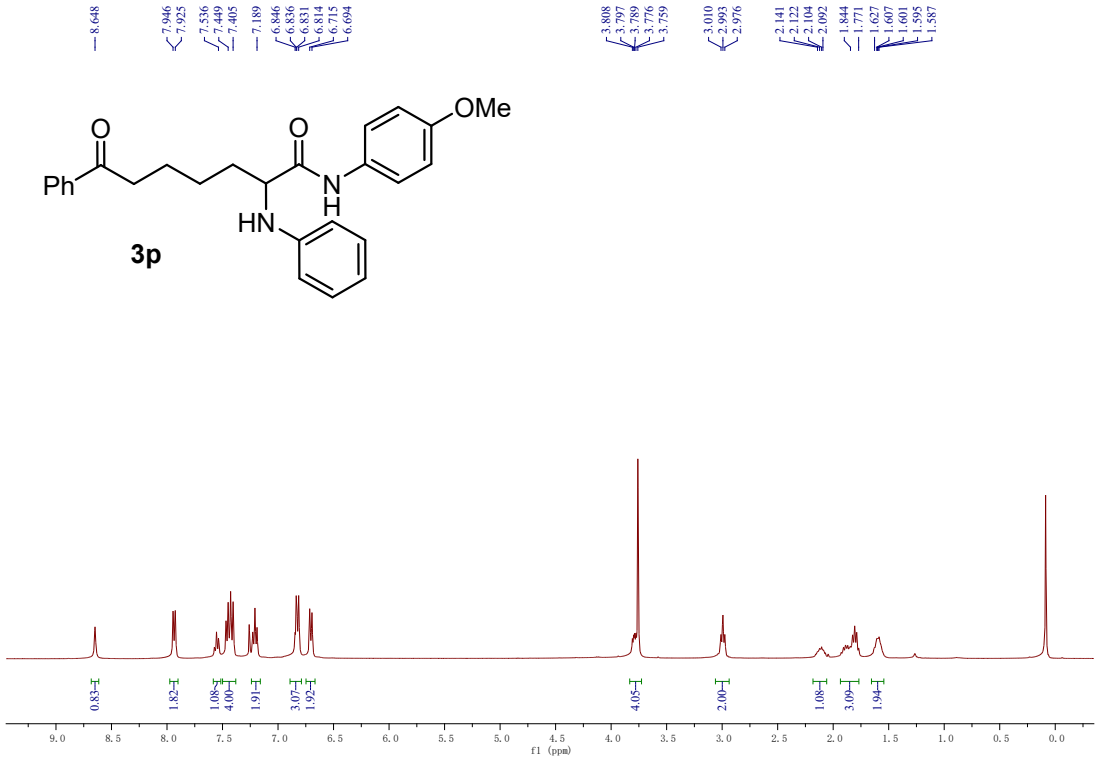
37.95
33.26
25.64
23.66
O
O
Ph
N
H
HN
3o
f1 (ppm)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7.405
7.189
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6.831
6.814
6.715
6.694
3.808
3.797
3.789
3.776
3.759
3.010
2.993
2.976
2.141
2.122
2.104
2.092
1.844
1.771
1.627
1.607
1.601
1.595
1.587
O
O
OMe
Ph
N
H
HN
3p
0.83
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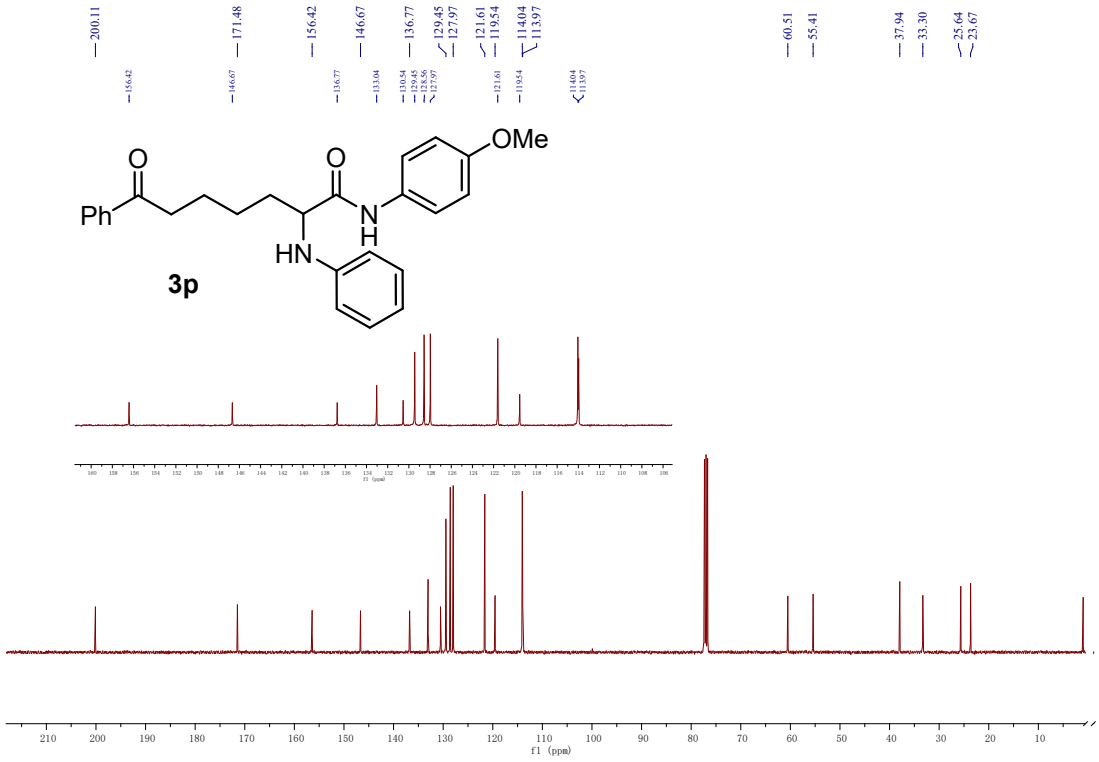
1.91
3.07
1.92
4.05
2.00
1.08
3.09
1.94
f1 (ppm)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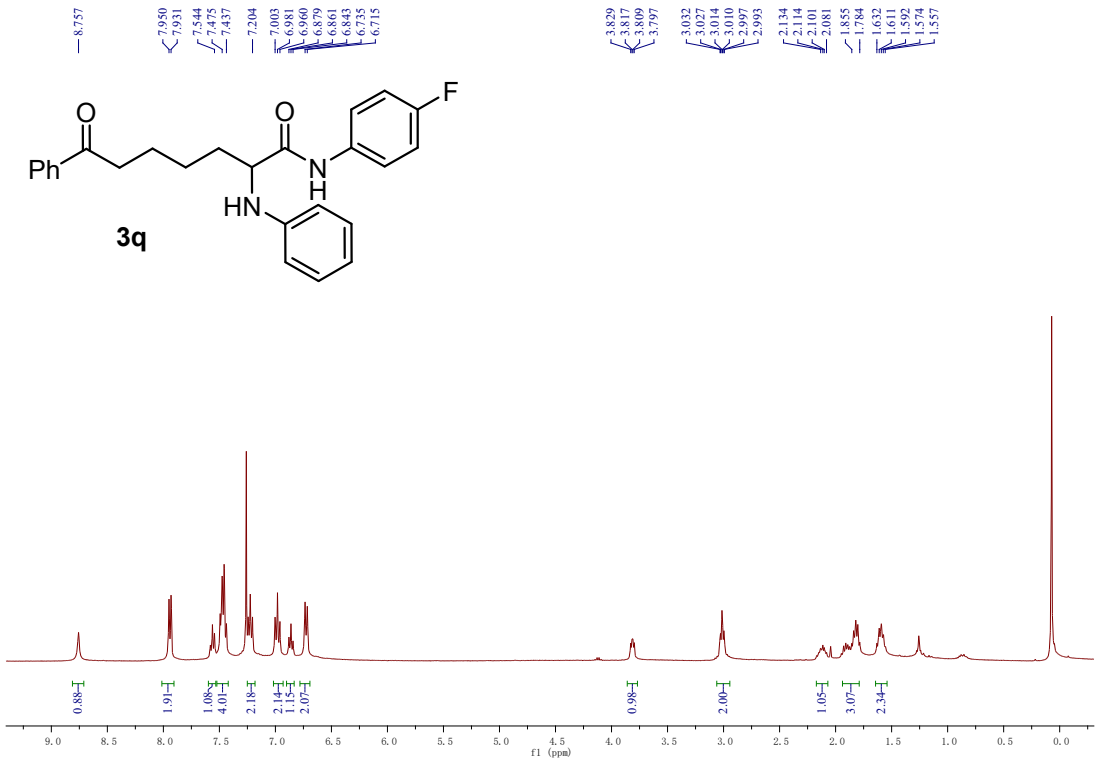
129.45
127.97
121.61
119.54
114.04
113.97
60.51
55.41
37.94
33.30
25.64
23.67
O
O
OMe
Ph
N
H
HN
3p
f1 (ppm)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7.003
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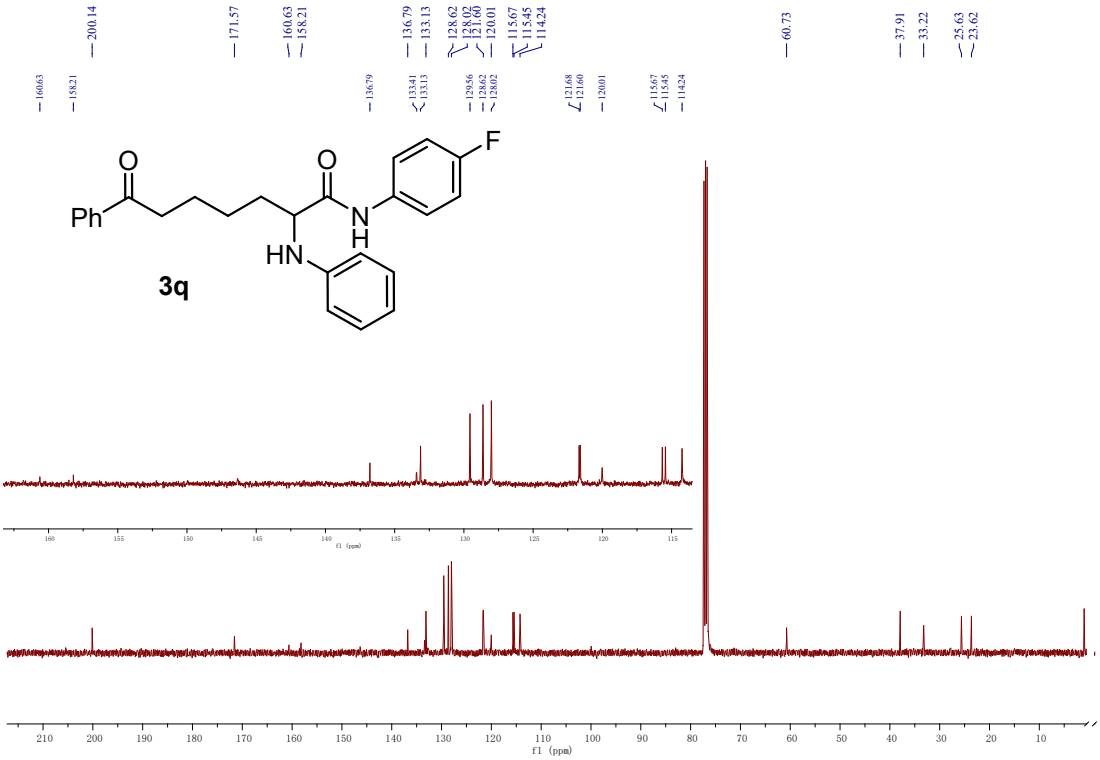
6.960
6.879
6.861
6.843
6.735
6.715
3.829
3.817
3.809
3.797
3.032
3.027
3.014
3.010
2.997
2.993
2.134
2.114
2.101
2.081
1.855
1.784
1.632
1.611
1.592
1.574
1.557
O
O
F
Ph
N
H
HN
3q
0.88
1.91
1.08
4.01
2.18
2.14
1.15
2.07
0.98
2.00
1.05
3.07
2.34
f1 (ppm)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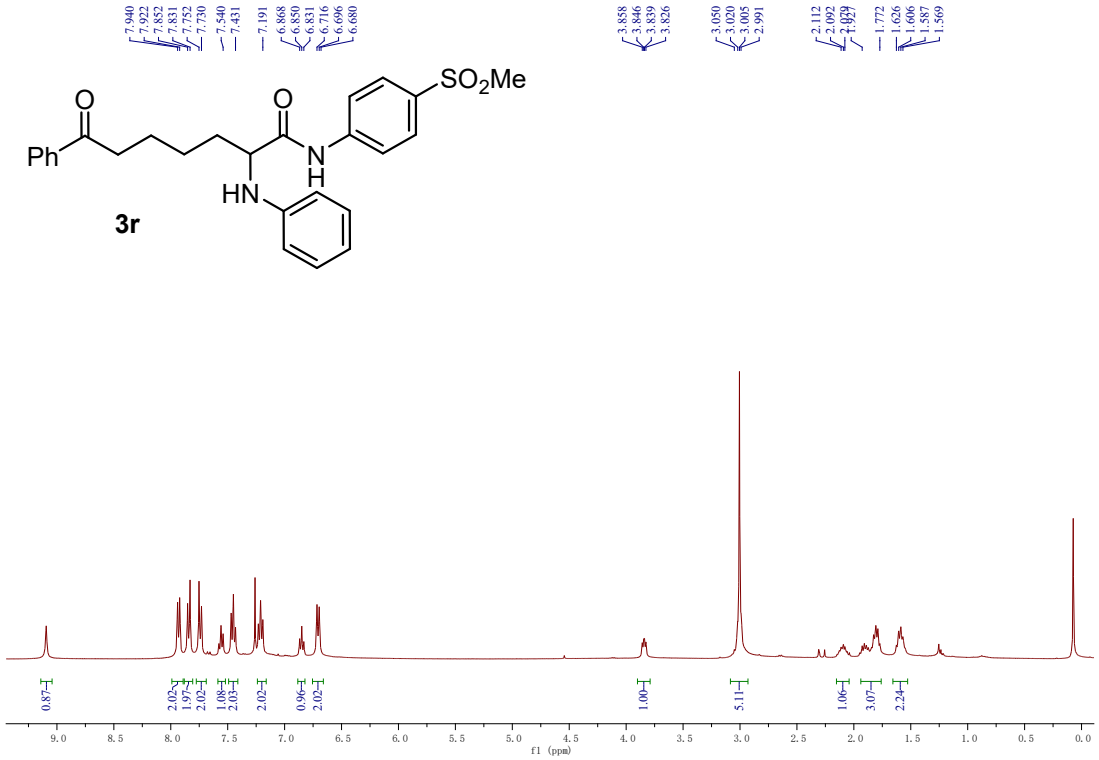
158.21
136.79
133.13
128.62
128.02
121.66
120.01
115.67
115.45
114.24
60.73
37.91
33.22
25.63
23.62
O
O
F
Ph
N
H
HN
3q
f1 (ppm)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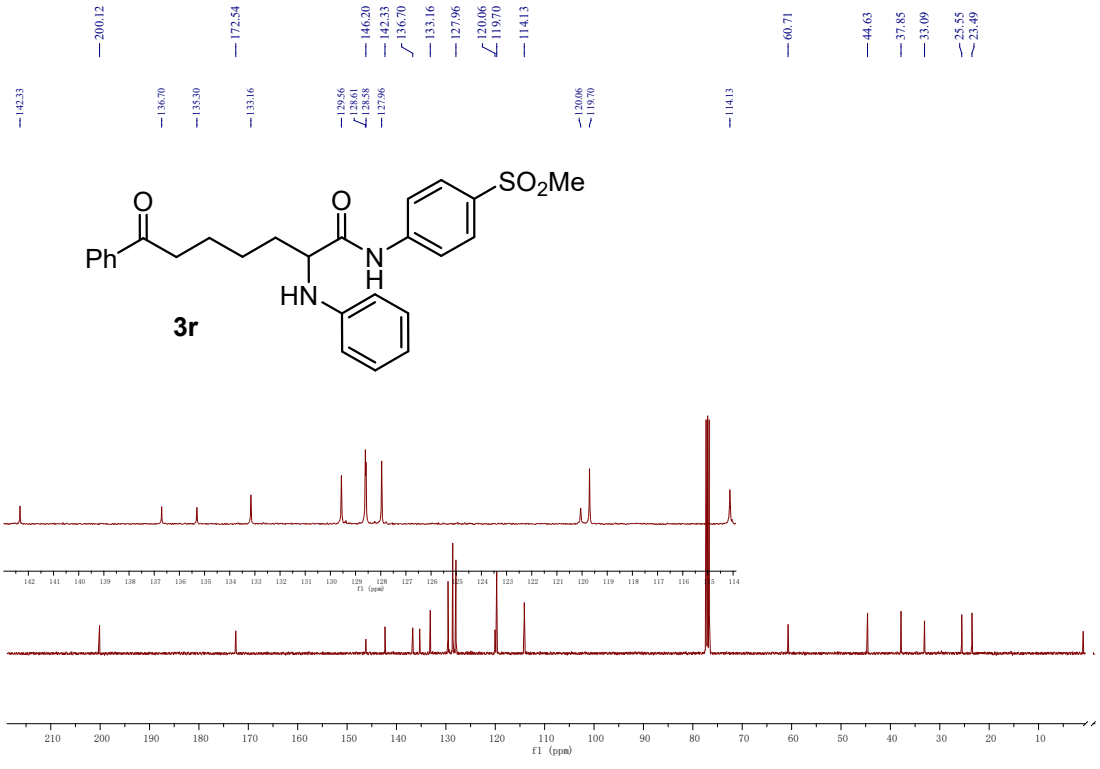
6.850
6.831
6.716
6.696
6.680
3.858
3.846
3.839
3.826
3.050
3.020
3.005
2.991
2.112
2.092
2.079
1.927
1.772
1.626
1.606
1.587
1.569
O
Ph
O
N
H
HN
SO2Me
3r
0.87
2.02
1.97
2.02
1.08
2.03
2.02
0.96
2.02
1.00
5.11
1.06
3.07
2.24
f1 (ppm)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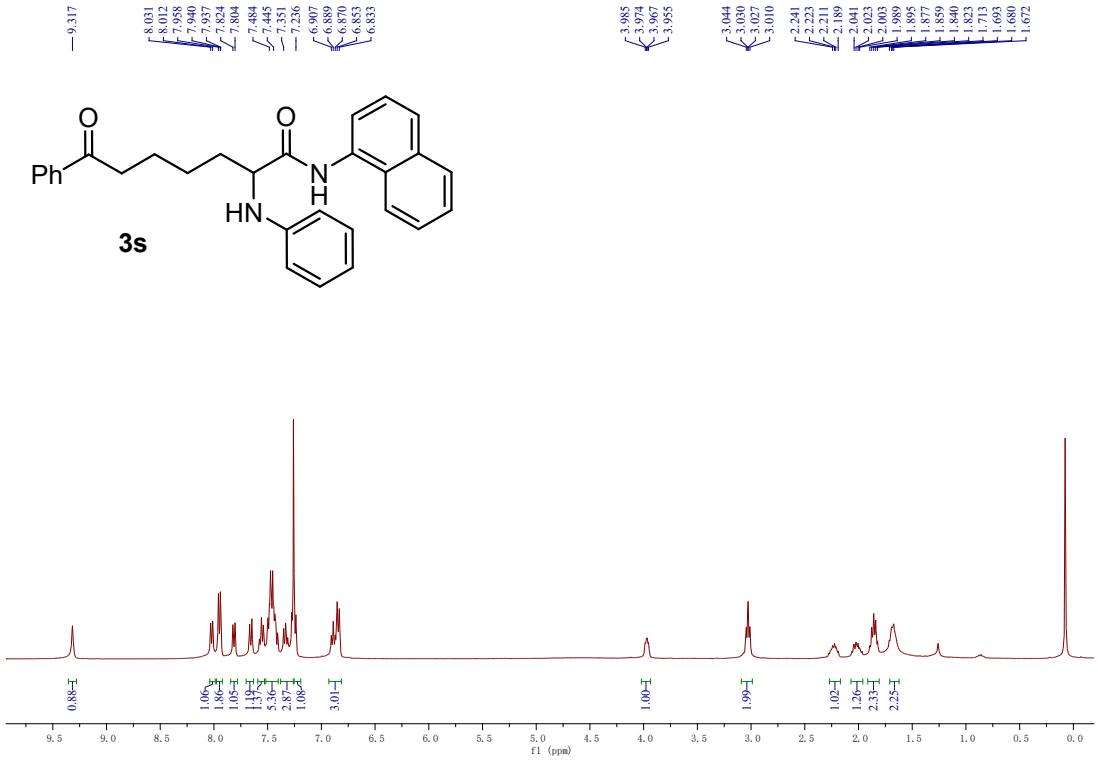
136.70
133.16
127.96
120.06
119.70
114.13
60.71
44.63
37.85
33.09
25.55
23.49
142.33
136.70
135.30
133.16
129.56
128.61
128.58
127.96
120.06
119.70
114.13
O
Ph
O
N
H
HN
SO2Me
3r
f1 (ppm)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7.804
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7.445
7.351
7.236
6.907
6.889
6.870
6.853
6.833
3.985
3.974
3.967
3.955
3.044
3.030
3.027
3.010
2.241
2.223
2.211
2.189
2.041
2.023
2.003
1.989
1.895
1.877
1.859
1.840
1.823
1.713
1.693
1.680
1.672
O
O
Ph
N
H
HN
3s
0.88
1.06
1.86
1.05
1.13
5.36
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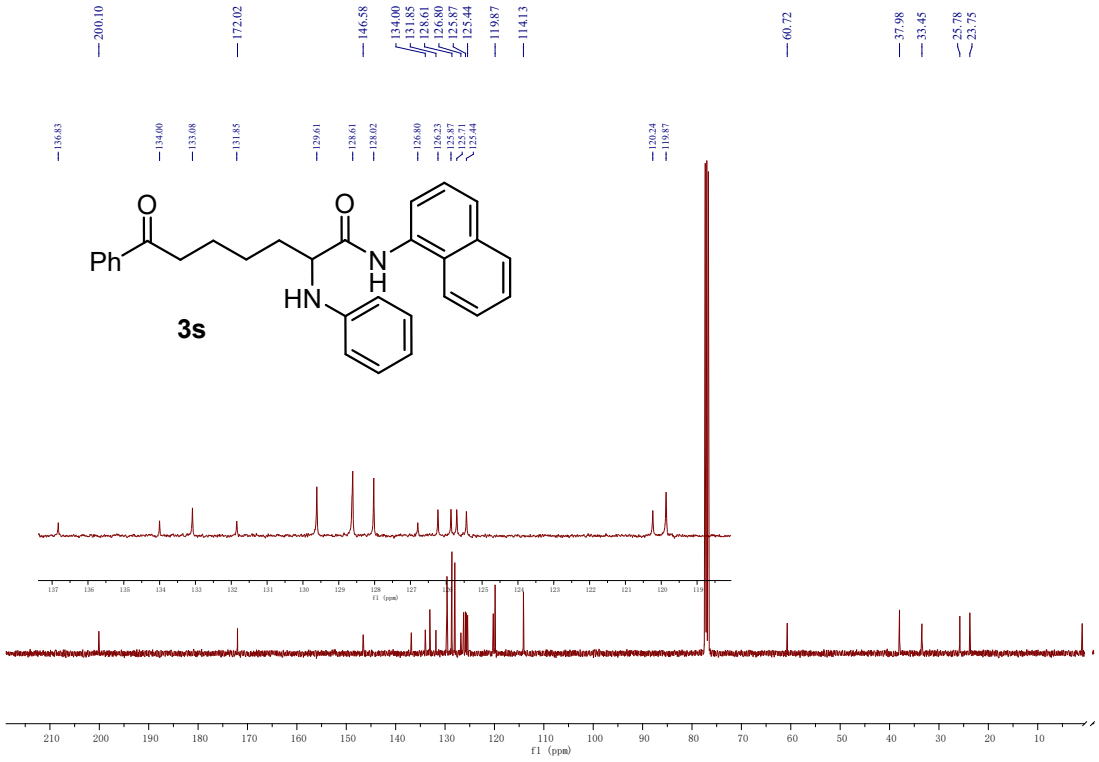
1.00
1.99
1.02
1.26
2.33
2.25
f1 (ppm)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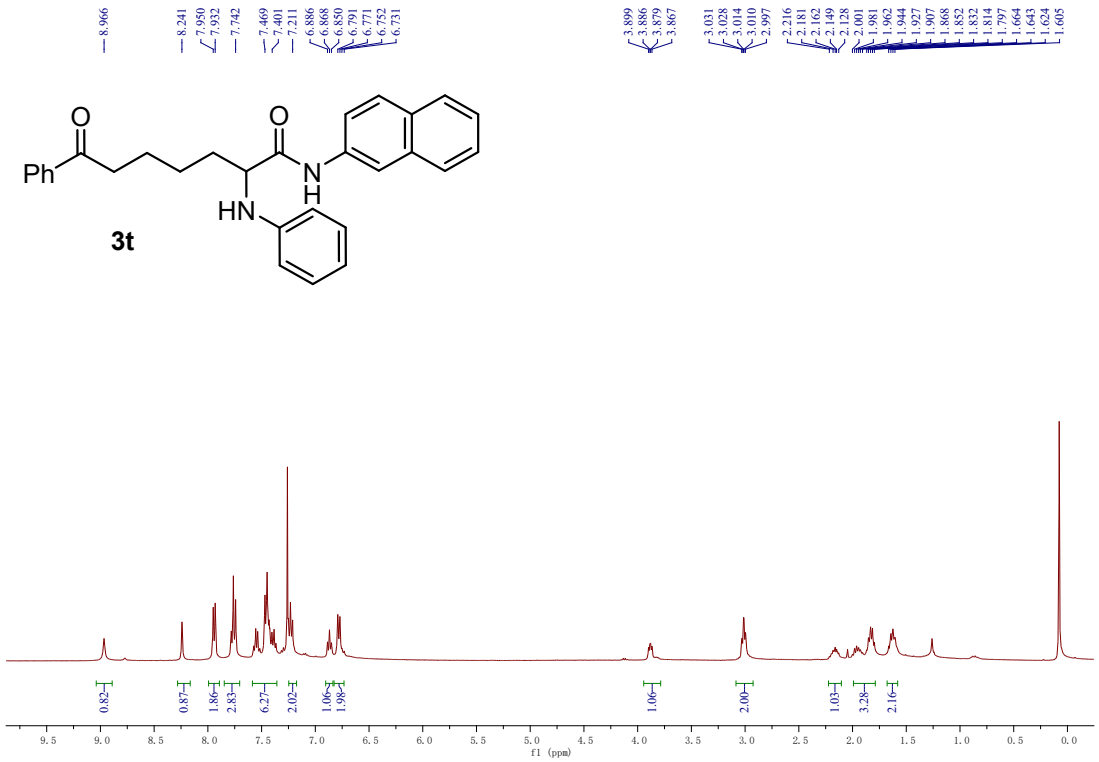
128.61
126.80
125.87
125.44
119.87
114.13
60.72
37.98
33.45
25.78
23.75
136.83
134.00
133.08
131.85
129.61
128.61
126.02
126.80
126.23
125.87
125.71
125.44
120.24
119.87
O
O
Ph
N
H
HN
3s
f1 (ppm)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6.850
6.791
6.771
6.752
6.731
3.899
3.886
3.879
3.867
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3.028
3.014
3.010
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2.162
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2.001
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1.962
1.944
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1.852
1.832
1.814
1.797
1.664
1.643
1.624
1.605
O
O
Ph
N
H
HN
3t
0.82
0.87
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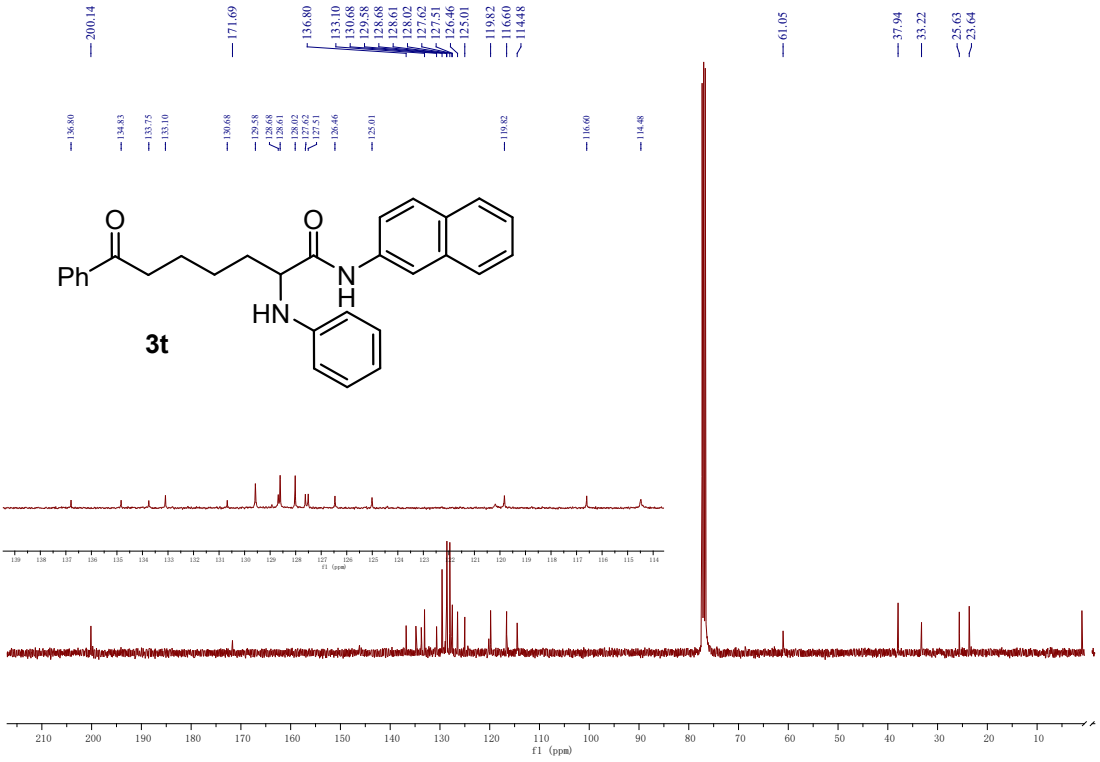
2.02
1.06
1.98
1.06
2.00
1.03
3.28
2.16
f1 (ppm)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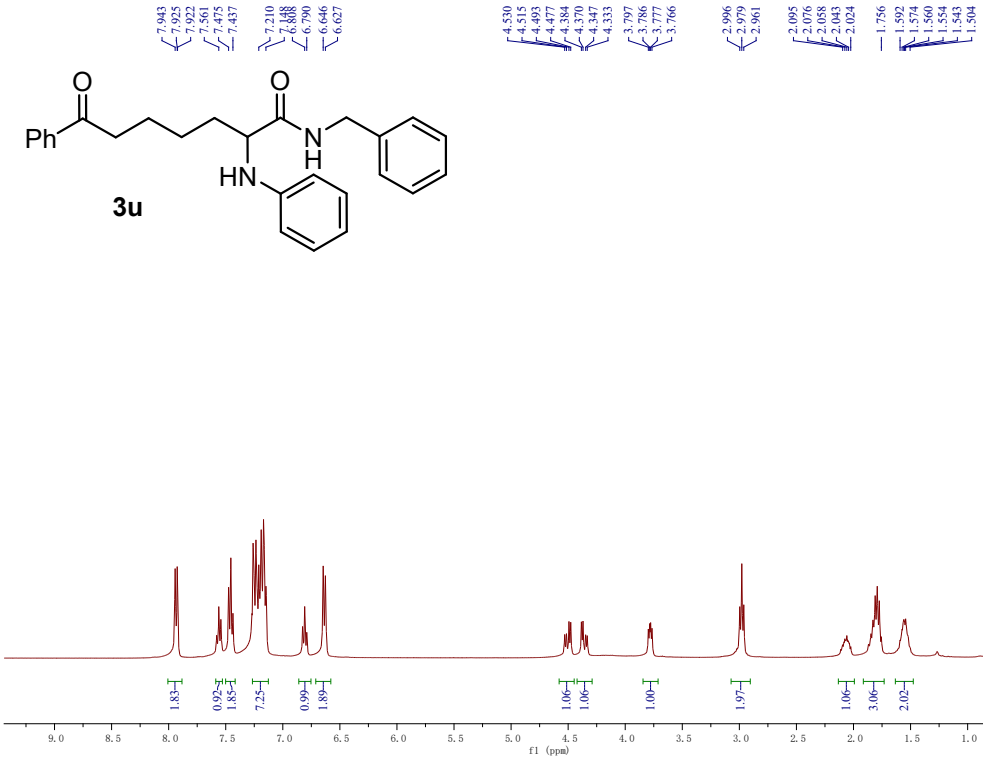
133.10
130.68
129.58
128.68
128.61
128.02
127.62
127.51
126.46
125.01
119.82
116.60
114.48
61.05
37.94
33.22
25.63
23.64
O
O
Ph
N
H
HN
3t
f1 (ppm)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7.148
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6.646
6.627
4.530
4.515
4.493
4.477
4.384
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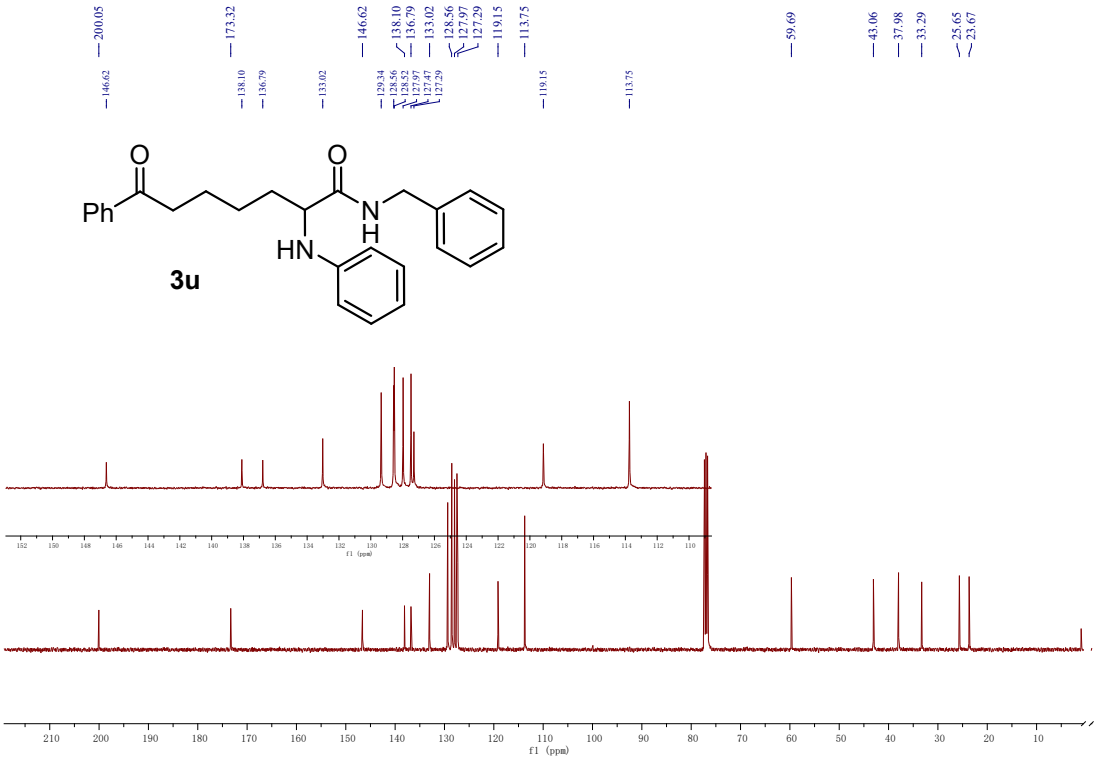
4.347
4.333
3.797
3.786
3.777
3.766
2.996
2.979
2.961
2.095
2.076
2.058
2.043
2.024
1.756
1.592
1.574
1.560
1.554
1.543
1.504
O
O
Ph
N
H
HN
3u
1.83
0.92
1.85
7.25
0.99
1.89
1.06
1.06
1.00
1.97
1.06
3.06
2.02
f1 (ppm)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128.56
128.52
127.97
127.47
127.29
119.15
113.75
59.69
43.06
37.98
33.29
25.65
23.67
O
O
Ph
N
H
HN
3u
f1 (ppm)


7.938
7.920
7.917
7.554
7.469
7.431
7.177
7.159
6.796
6.778
6.625
6.606
6.247
6.239
6.110
6.102
4.520
4.504
4.481
4.465
4.374
4.361
4.335
4.322
3.763
3.751
3.743
3.731
2.989
2.971
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2.000
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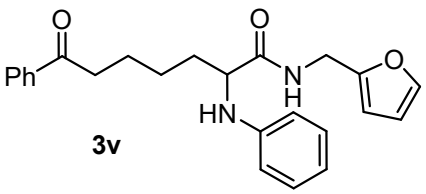
1.538
1.519
1.502
1.486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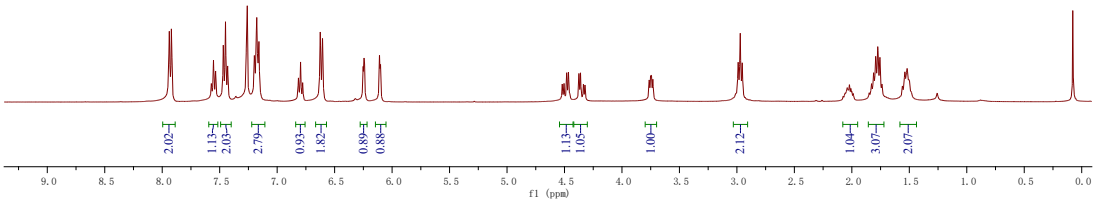
Ph
N
H
O
HN

3v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2.03
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0.88
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1.05
1.00
2.12
1.04
3.07
2.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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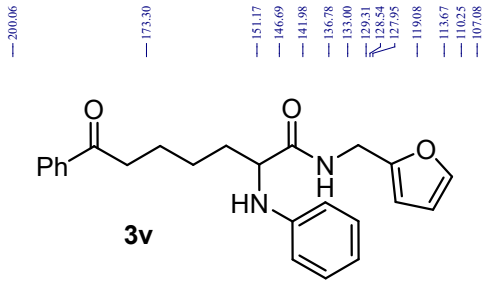
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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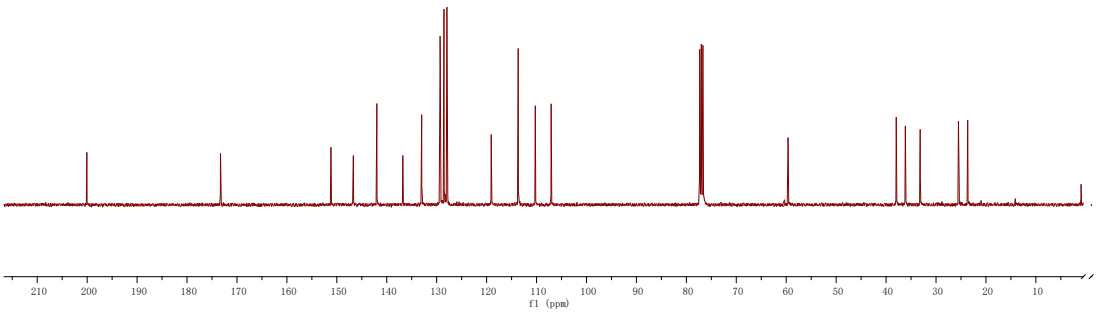
127.95
119.08
113.67
110.25
107.08
59.64
37.97
36.14
33.17
25.51
23.65

O
O
Ph
N
H
O
HN

3v


210
200
190
180
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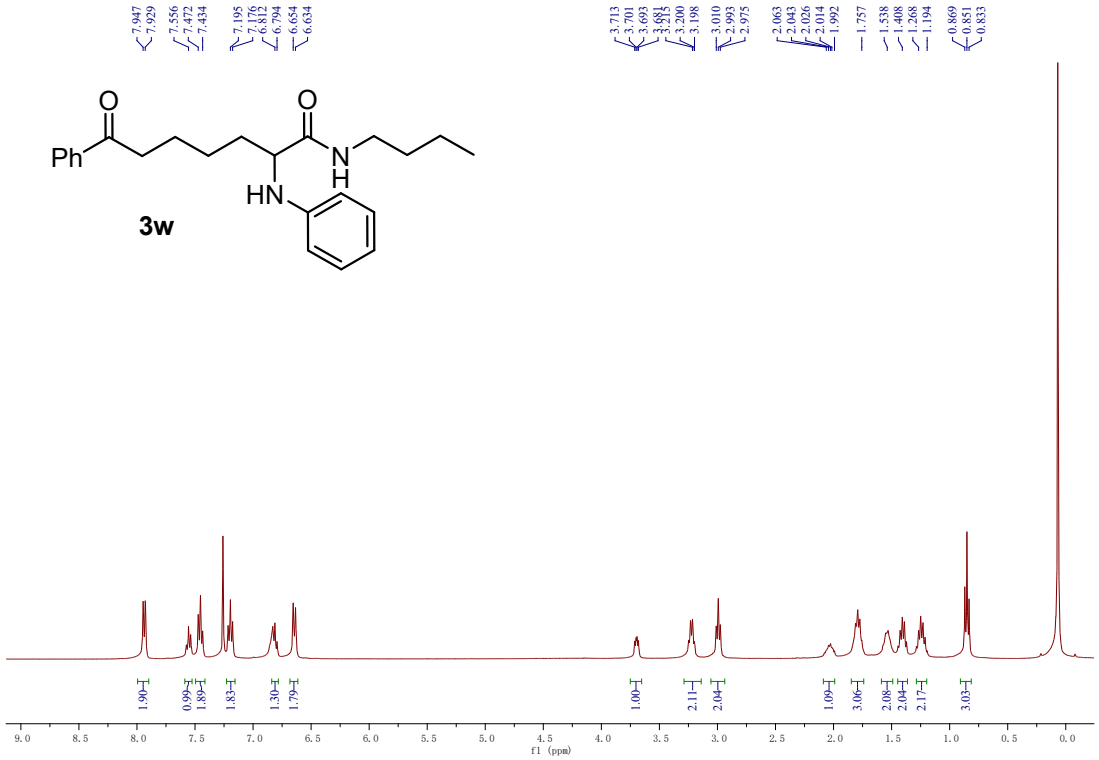
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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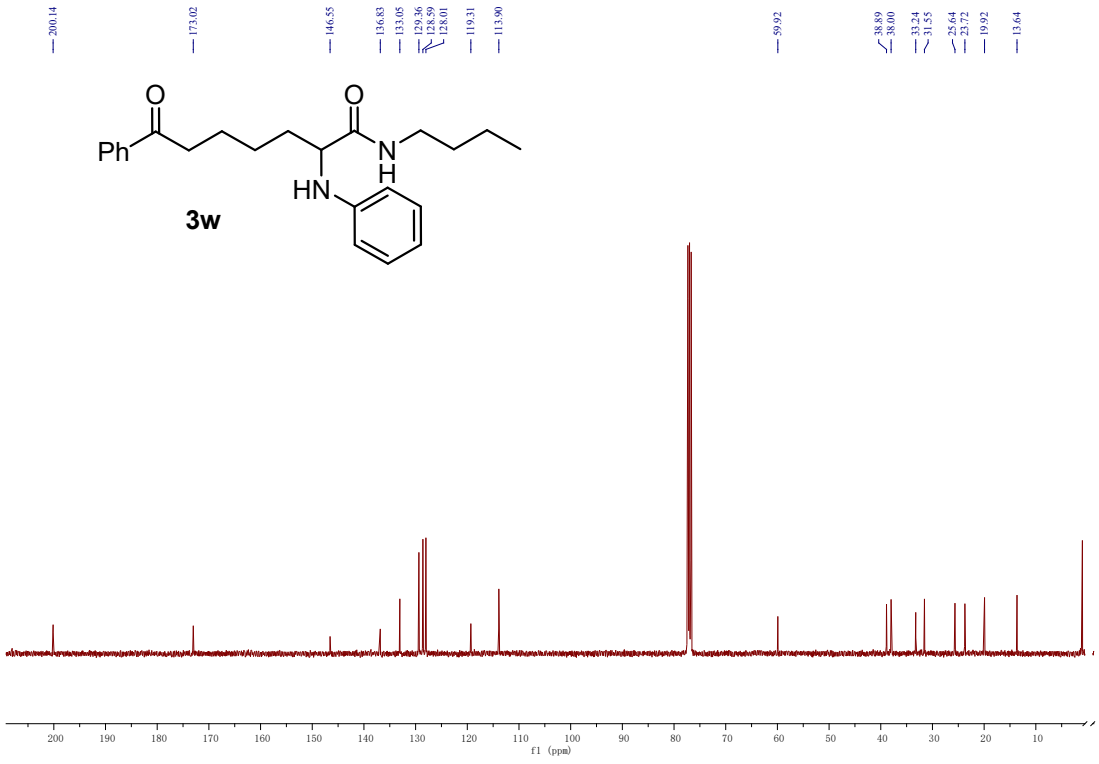
6.634
3.713
3.701
3.693
3.681
3.215
3.200
3.198
3.010
2.993
2.975
2.063
2.043
2.026
2.014
1.992
1.757
1.538
1.408
1.268
1.194
0.869
0.851
0.833
O
O
Ph
N
H
HN
3w
1.90
0.99
1.89
1.83
1.30
1.79
1.00
2.11
2.04
1.09
3.06
2.08
2.04
2.17
3.03
f1 (ppm)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129.36
128.59
128.01
119.31
113.90
59.92
38.89
38.00
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25.64
23.72
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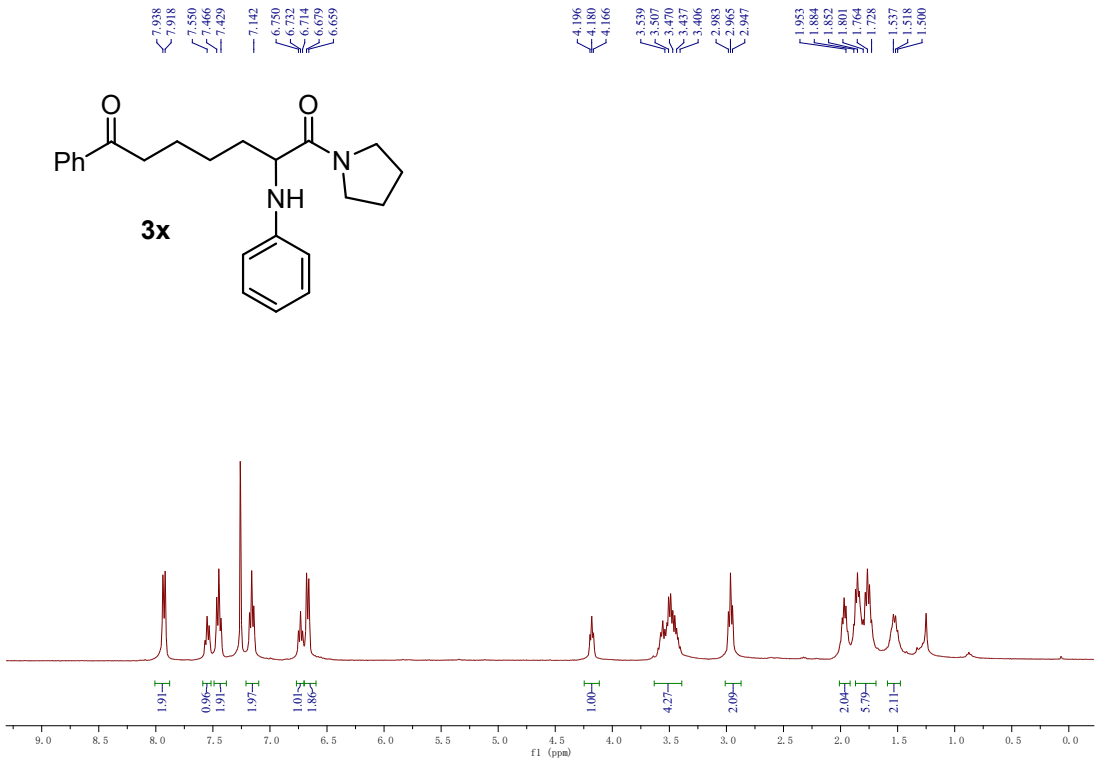
13.64
O
O
Ph
N
H
HN
3w
f1 (ppm)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7.918
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7.466
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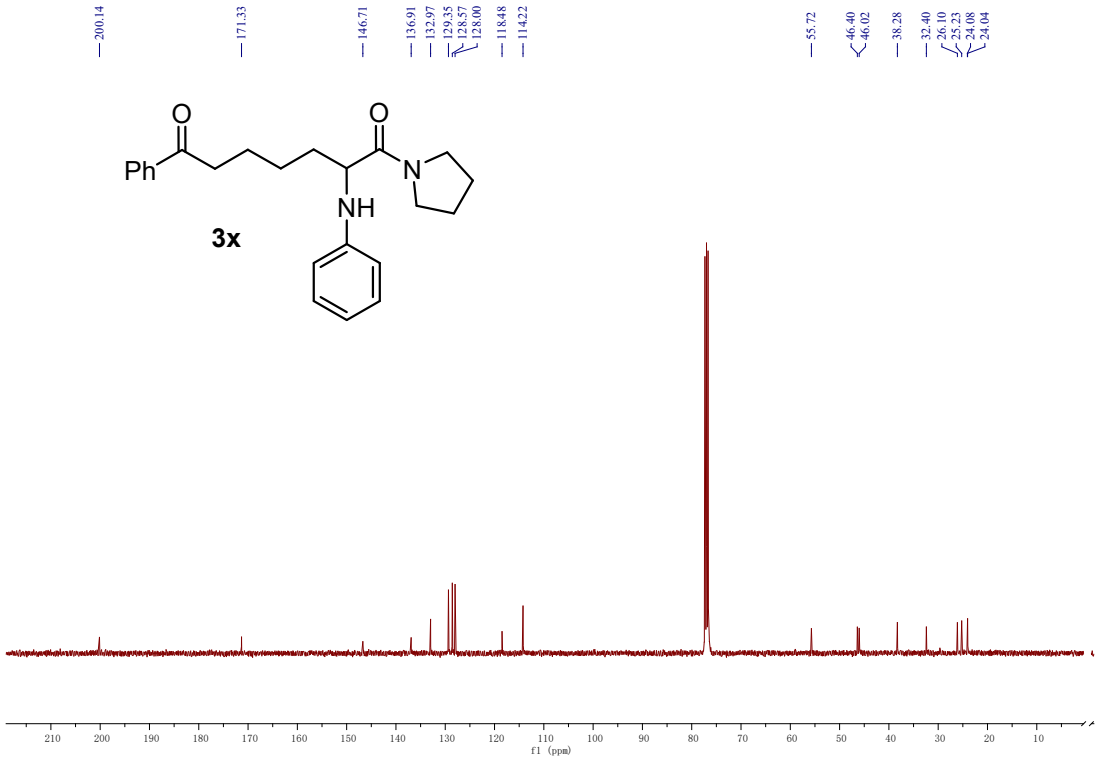
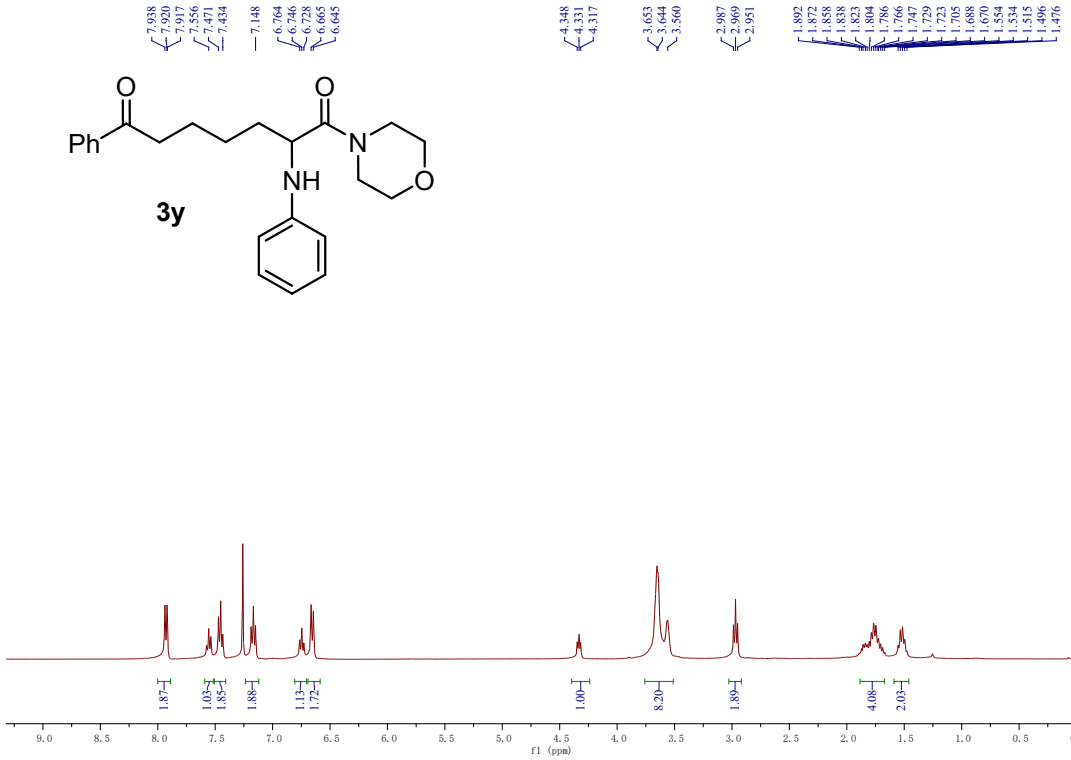
7.142
6.750
6.732
6.714
6.679
6.659
4.196
4.180
4.166
3.539
3.507
3.470
3.437
3.406
2.983
2.965
2.947
1.953
1.884
1.852
1.801
1.764
1.728
1.537
1.518
1.500
O
O
Ph
N
NH
3x
1.91
0.96
1.91
1.97
1.01
1.86
1.00
4.27
2.09
2.04
5.79
2.11
f1 (ppm)


200.14
171.33
146.71
136.91
132.97
129.35
128.57
128.00
118.48
114.22
55.72
46.40
46.02
38.28
32.40
26.10
25.23
24.08
24.04
O
O
Ph
N
NH
3x
f1 (ppm)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4.331
4.317
3.653
3.644
3.560
2.987
2.969
2.951
1.892
1.872
1.858
1.838
1.823
1.804
1.786
1.766
1.747
1.729
1.723
1.705
1.688
1.670
1.554
1.534
1.515
1.496
1.476
O
O
Ph
N
NH
O
3y
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1.88
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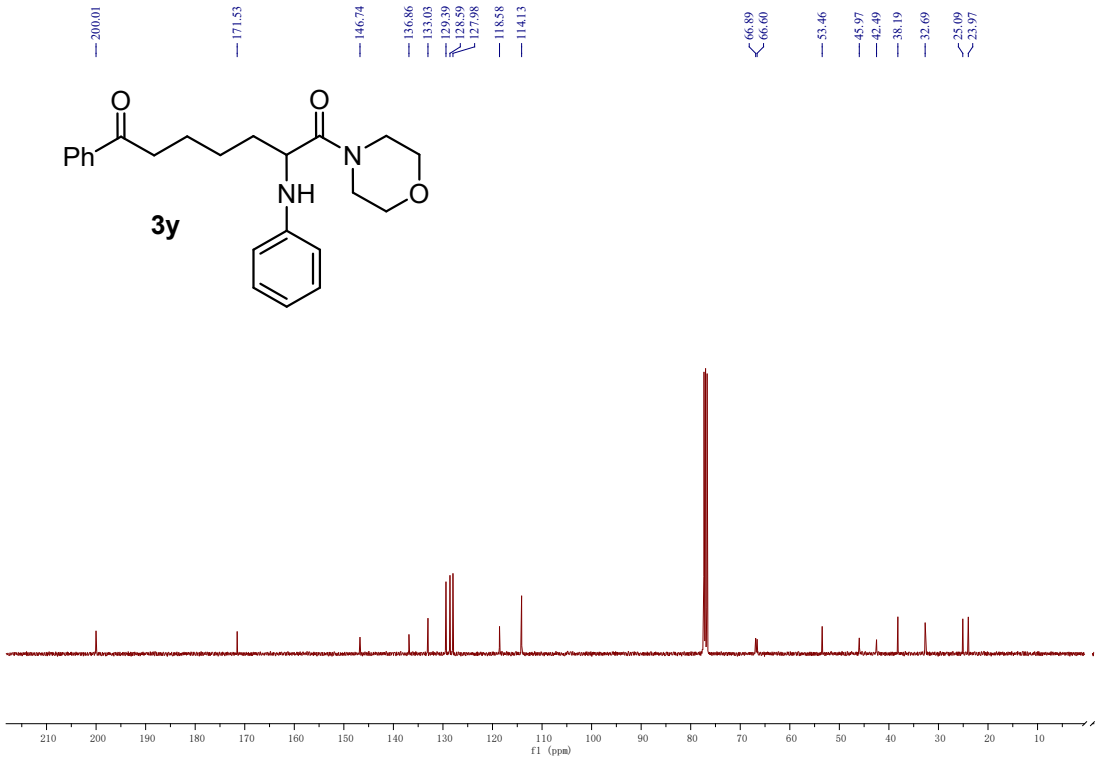
1.89
4.08
2.03
f1 (ppm)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66.60
53.46
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32.69
25.09
23.97
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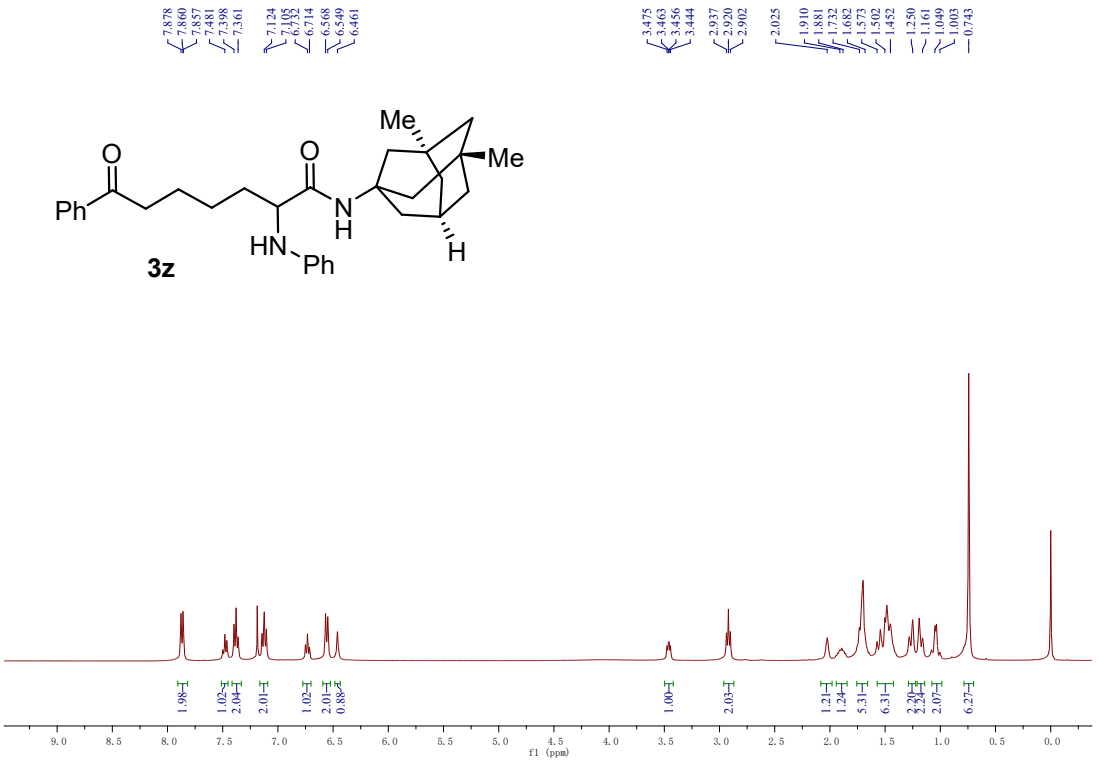
O
Ph
N
NH
O
3y
f1 (ppm)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3.475
3.463
3.456
3.444
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2.920
2.902
2.025
1.910
1.881
1.732
1.682
1.573
1.502
1.452
1.250
1.161
1.049
1.003
0.743
Me
Me
O
O
Ph
N
H
HN
Ph
H
3z
1.98
1.02
2.04
2.01
1.02
2.01
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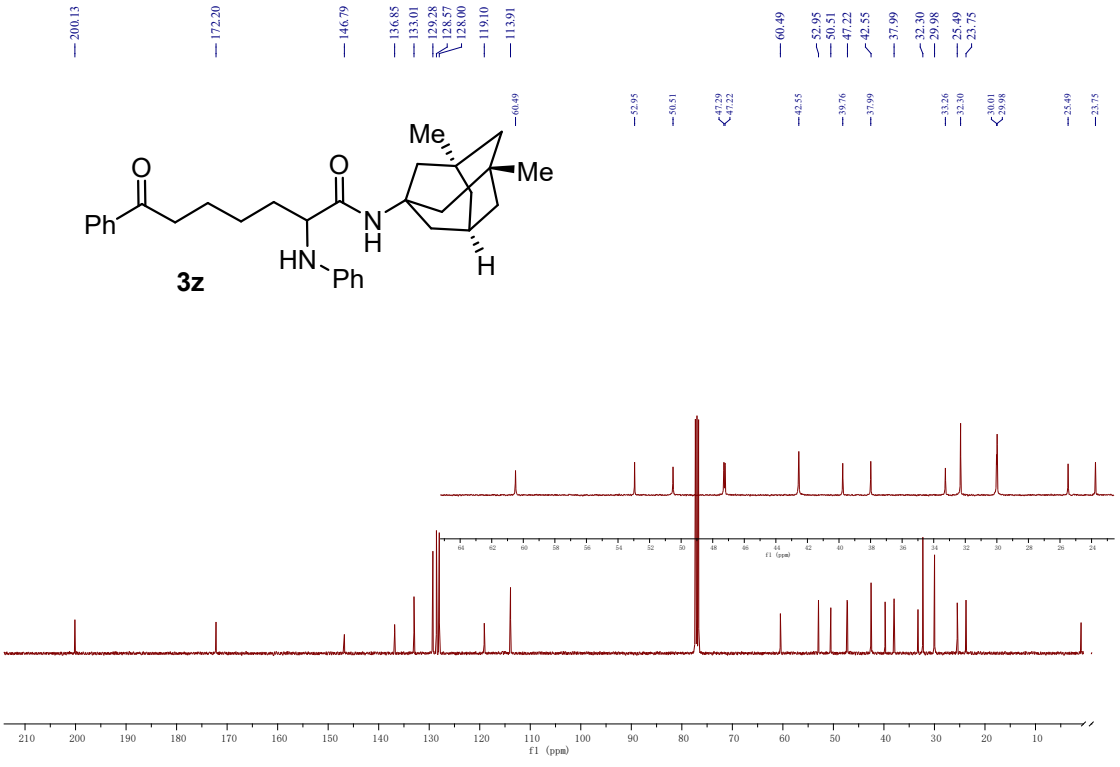
2.24
2.07
6.27
f1 (ppm)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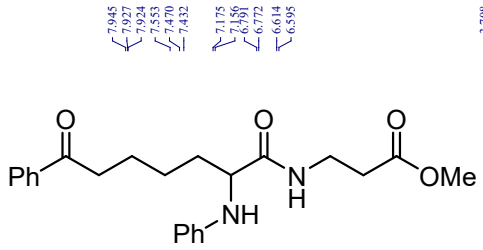
Ph
N
H
HN
Ph
H
3z
f1 (ppm)



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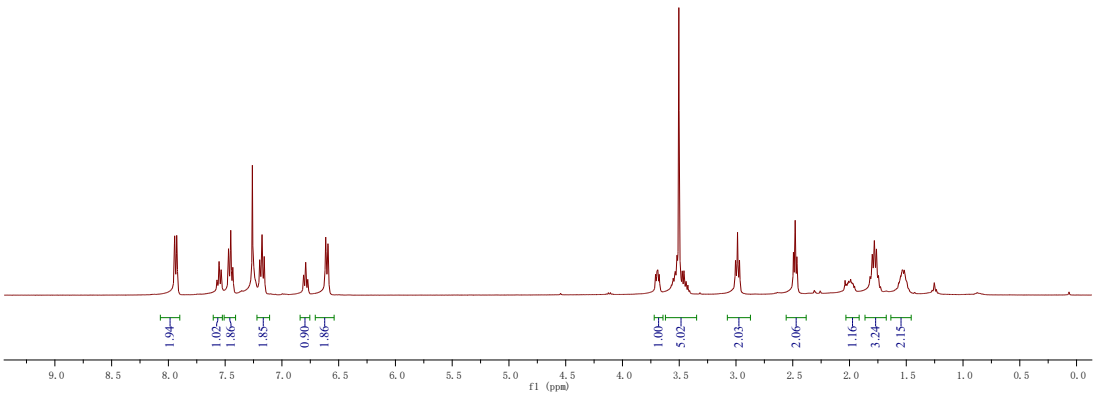

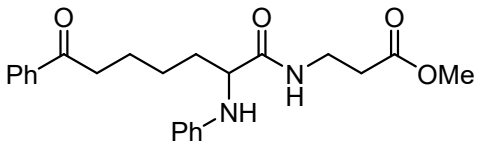

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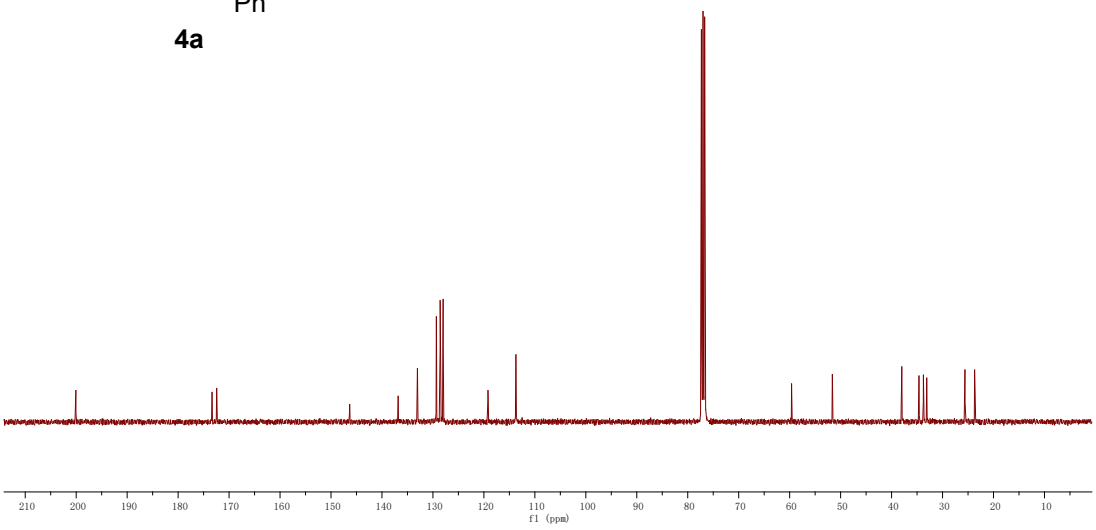

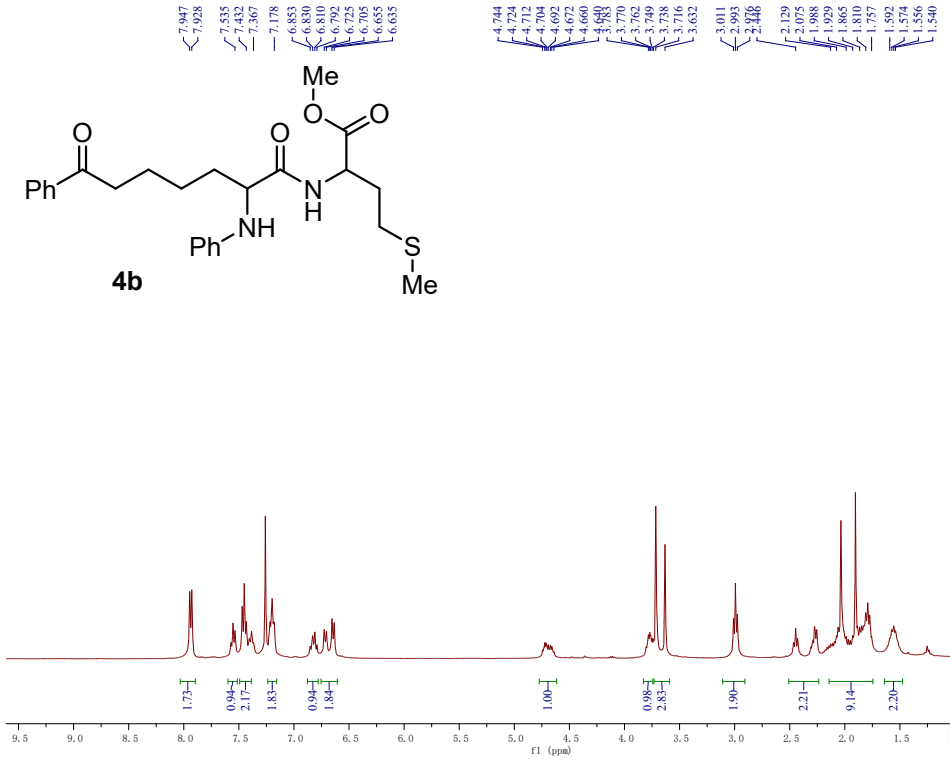
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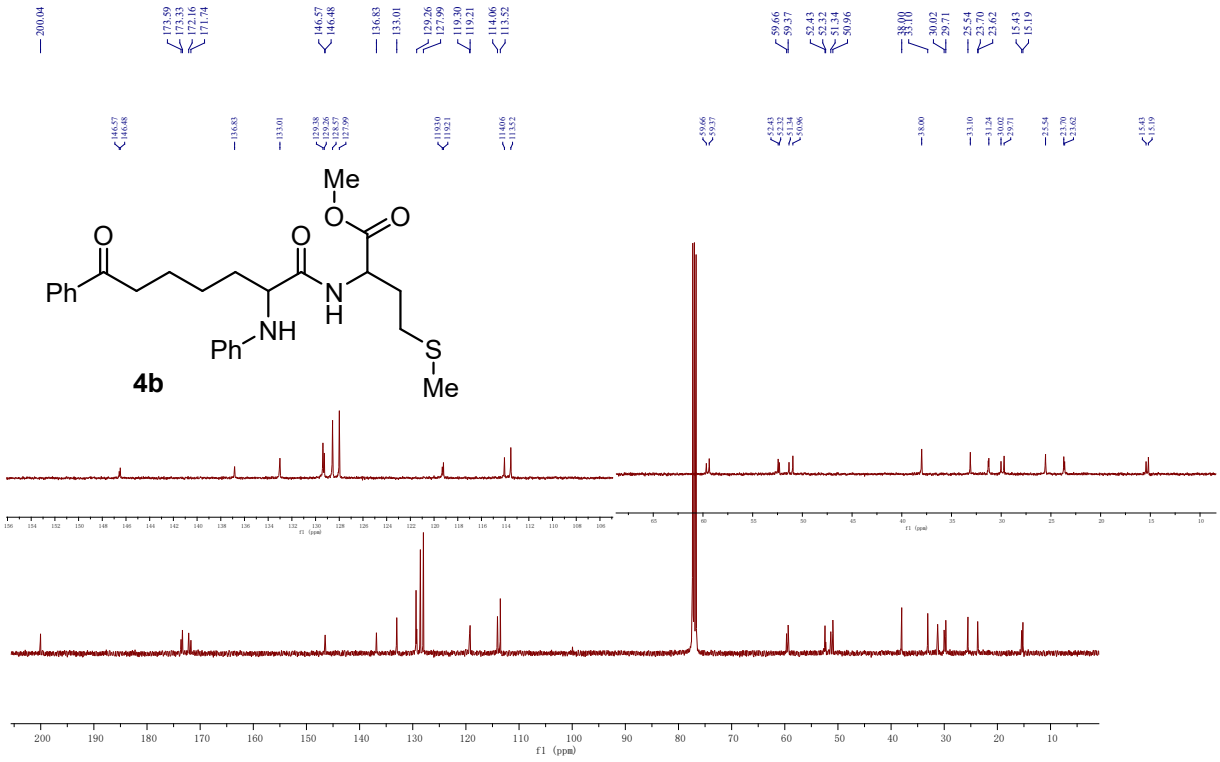
6.792
6.725
6.705
6.655
6.635
4.744
4.724
4.712
4.704
4.692
4.672
4.660
3.770
3.762
3.749
3.738
3.716
3.632
3.011
2.993
2.976
2.129
2.075
2.988
1.929
1.865
1.810
1.757
1.592
1.574
1.556
1.540
Me
O
O
O
Ph
O
N
H
NH
Ph
S
Me
4b
1.73
0.94
2.17
1.83
0.94
1.84
1.00
0.98
2.83
1.90
2.21
9.14
2.20
f1 (ppm)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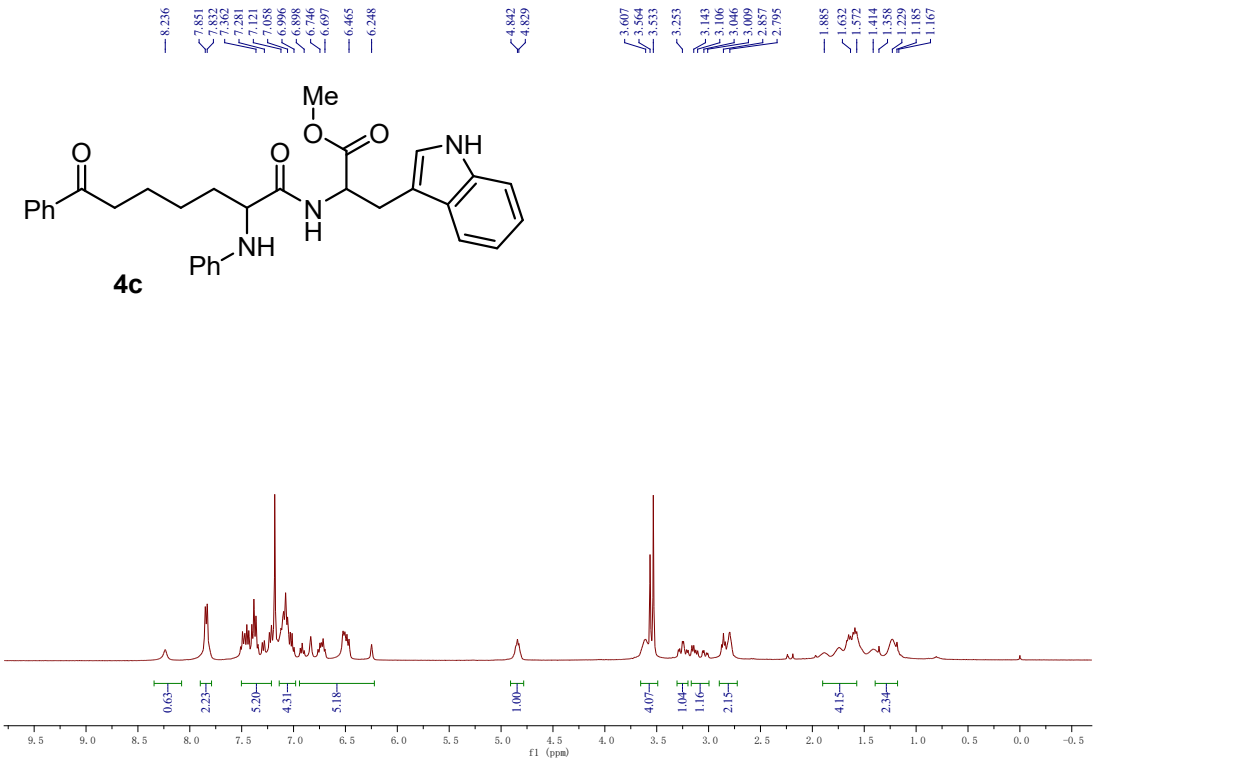
129.26
127.99
119.30
119.21
114.06
113.52
59.66
59.37
52.43
52.32
51.34
50.96
38.00
33.10
30.02
29.71
25.54
23.70
23.62
15.43
15.19
Me
O
O
O
Ph
O
N
H
NH
Ph
S
Me
4b
f1 (ppm)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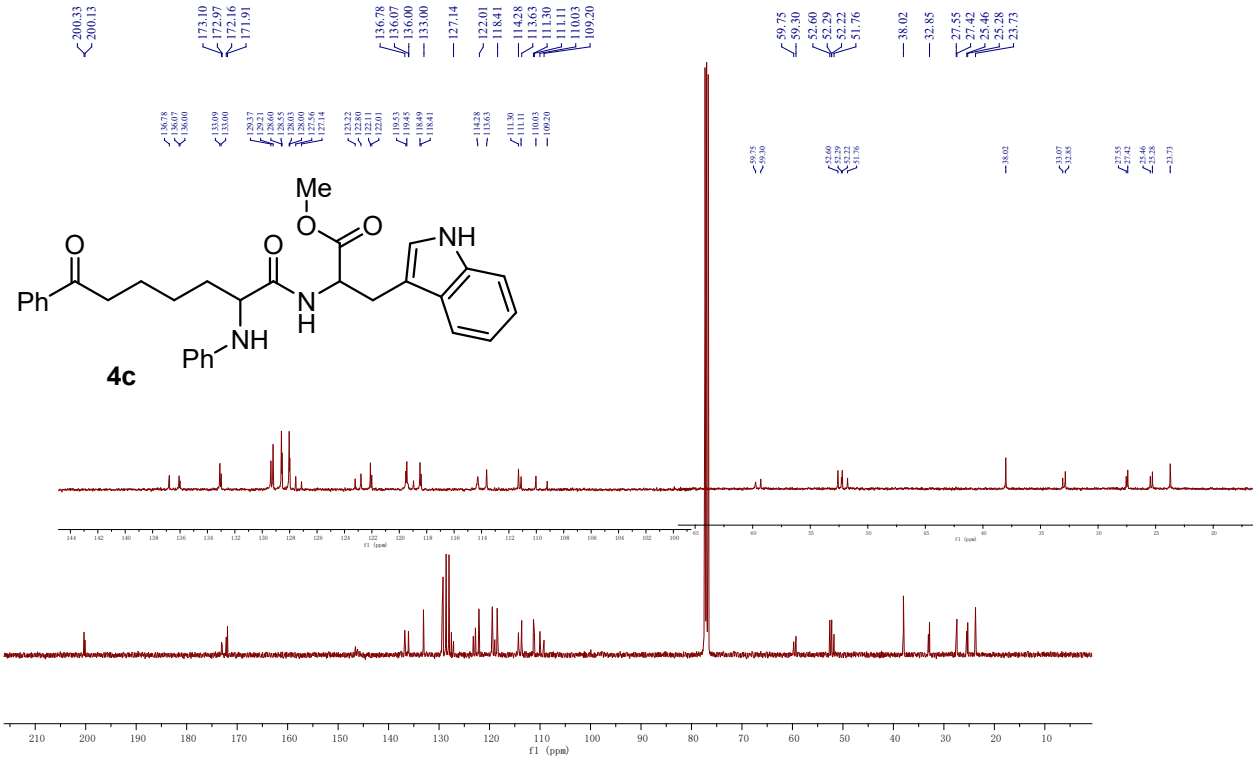
6.248
4.842
4.829
3.607
3.564
3.533
3.253
3.143
3.106
3.046
3.009
2.857
2.795
1.885
1.632
1.572
1.414
1.358
1.229
1.185
1.167
Me
O
O
O
NH
Ph
O
N
H
Ph
NH
4c
0.63
2.23
5.20
4.31
5.18
1.00
4.07
1.04
1.16
2.15
4.15
2.34
f1 (ppm)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136.78
136.07
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133.00
127.14
122.01
118.41
114.28
113.63
111.30
111.11
110.03
109.20
59.75
59.30
52.60
52.29
52.22
51.76
38.02
32.85
27.55
27.42
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23.73
Me
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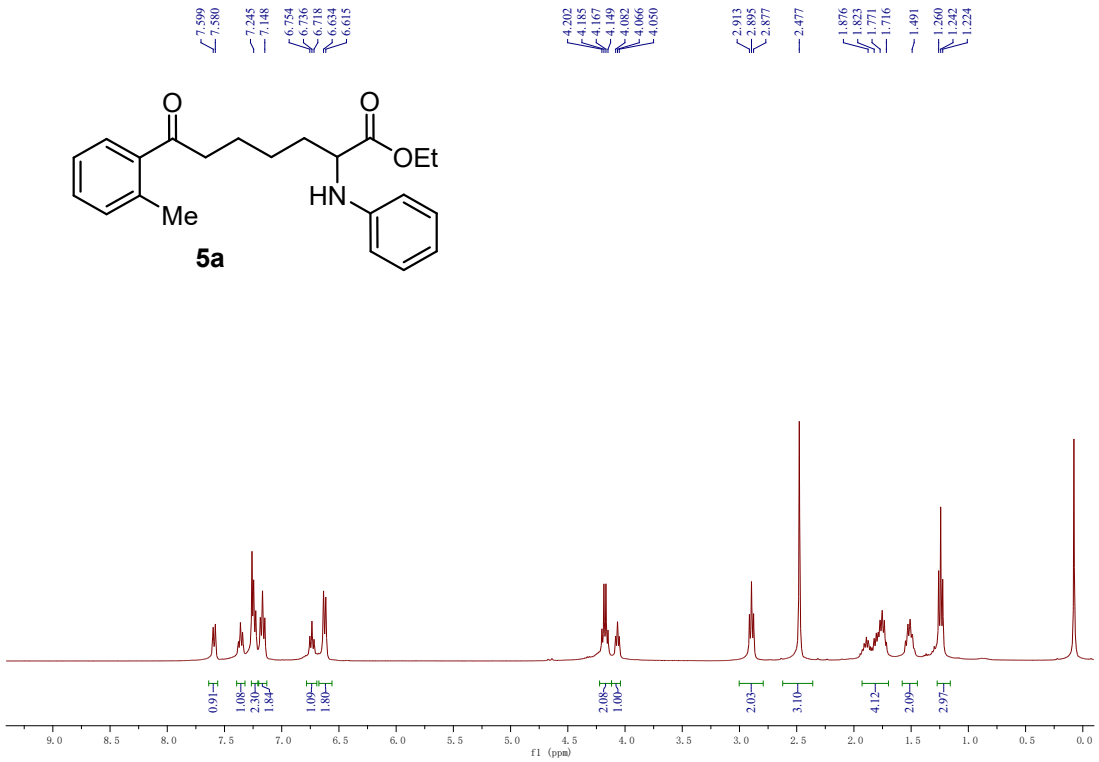
O
O
NH
Ph
O
N
H
Ph
NH
4c
f1 (ppm)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6.718
6.634
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4.167
4.149
4.082
4.066
4.050
2.913
2.895
2.877
2.477
1.876
1.823
1.771
1.716
1.491
1.260
1.242
1.224
O
O
OEt
HN
Me
5a
0.91
1.08
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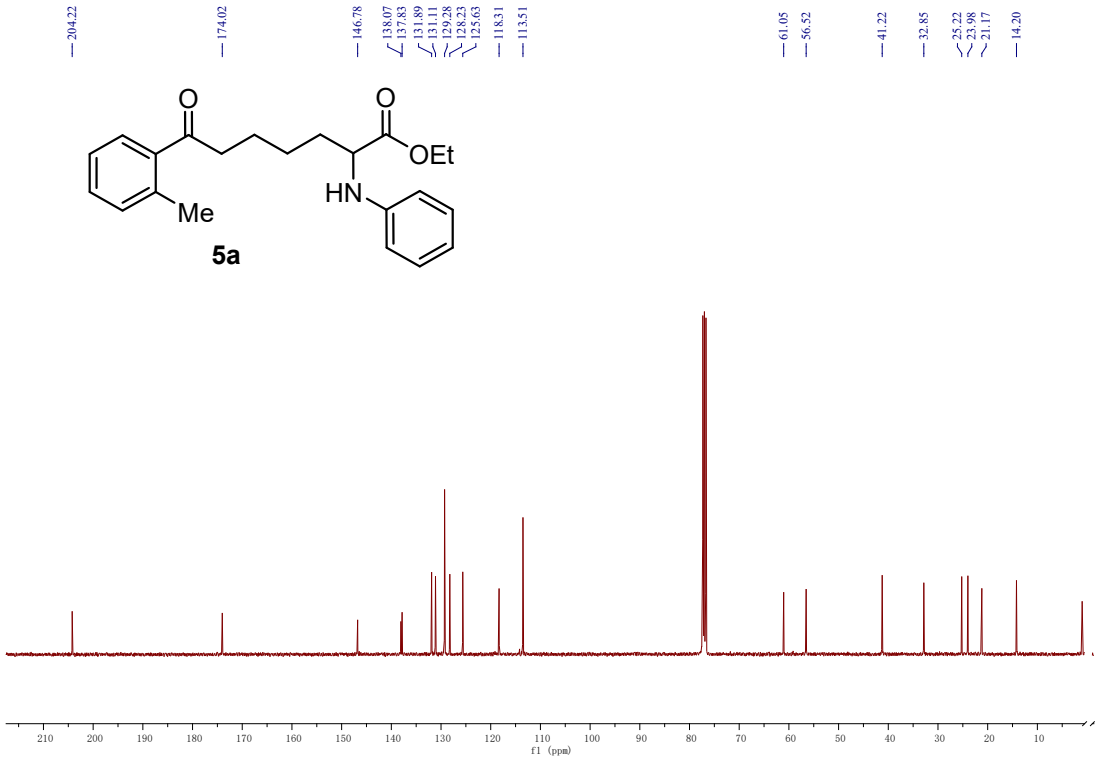
1.84
1.09
1.80
2.08
1.00
2.03
3.10
4.12
2.09
2.97
f1 (ppm)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131.89
131.11
129.28
128.23
125.63
118.31
113.51
61.05
56.52
41.22
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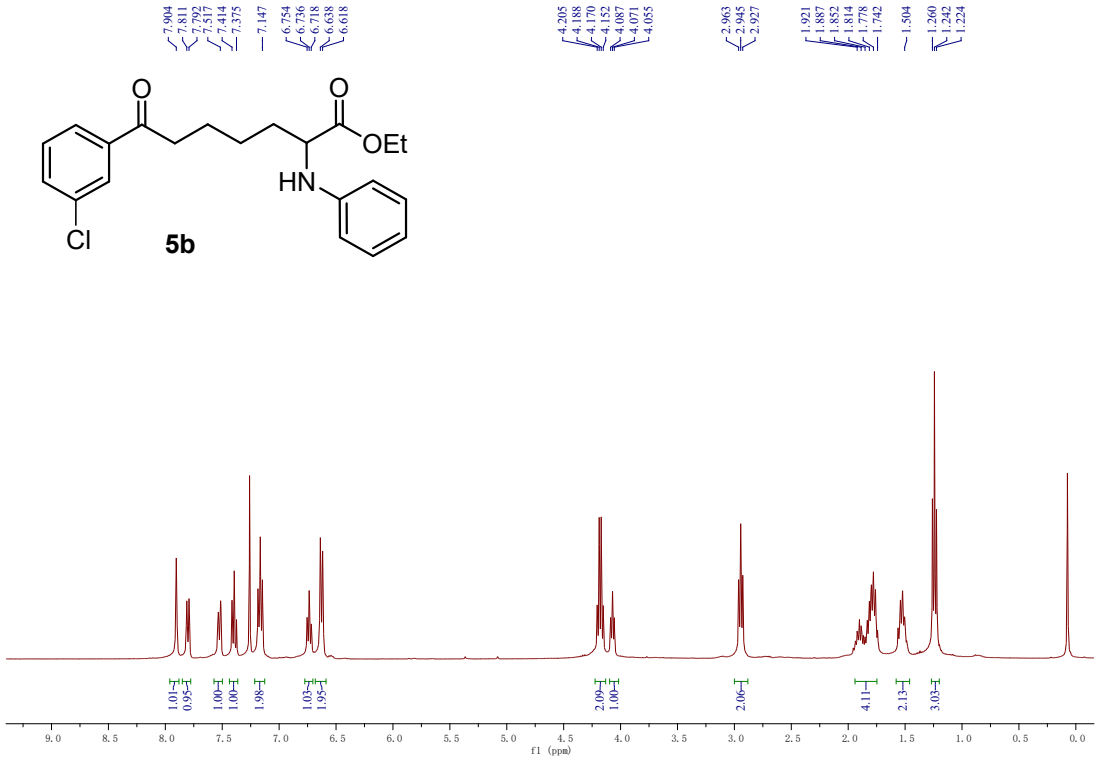
25.22
23.98
21.17
14.20
O
O
OEt
HN
Me
5a
f1 (ppm)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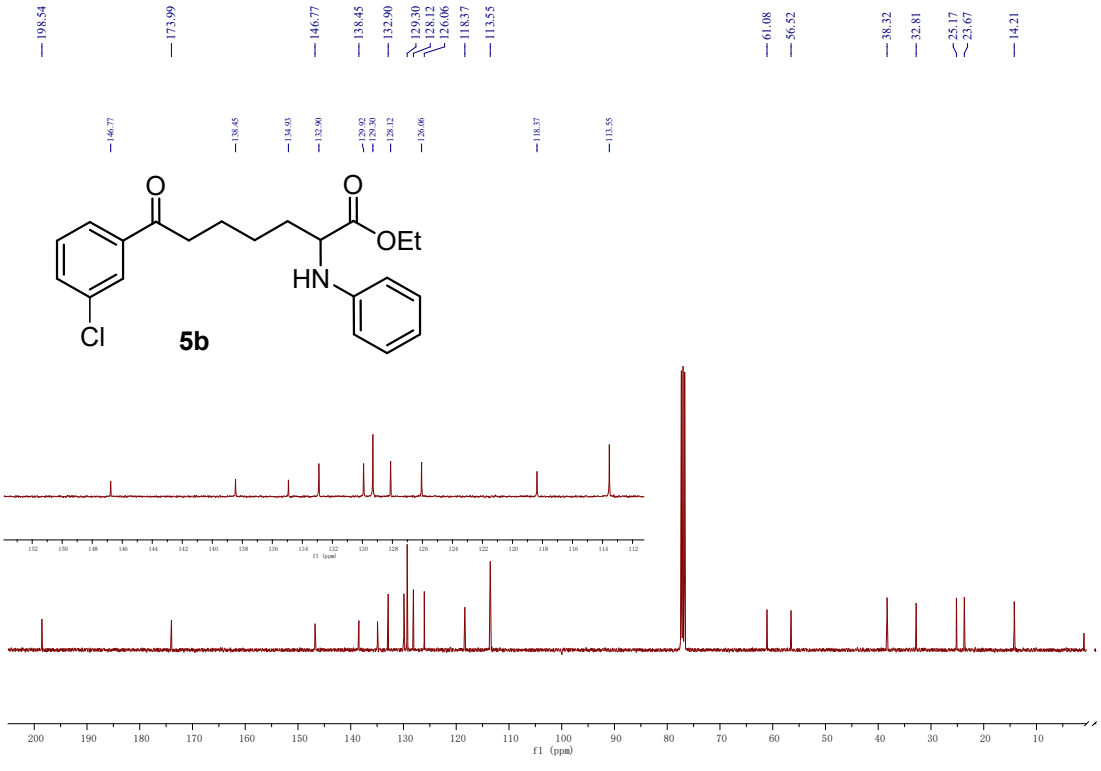
6.754
6.736
6.718
6.638
6.618
4.205
4.188
4.170
4.152
4.087
4.071
4.055
2.963
2.945
2.927
1.921
1.887
1.852
1.814
1.778
1.742
1.504
1.260
1.242
1.224
O
O
OEt
HN
Cl
5b
1.01
0.95
1.00
1.00
1.98
1.03
1.95
2.09
1.00
2.06
4.11
2.13
3.03
f1 (ppm)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126.06
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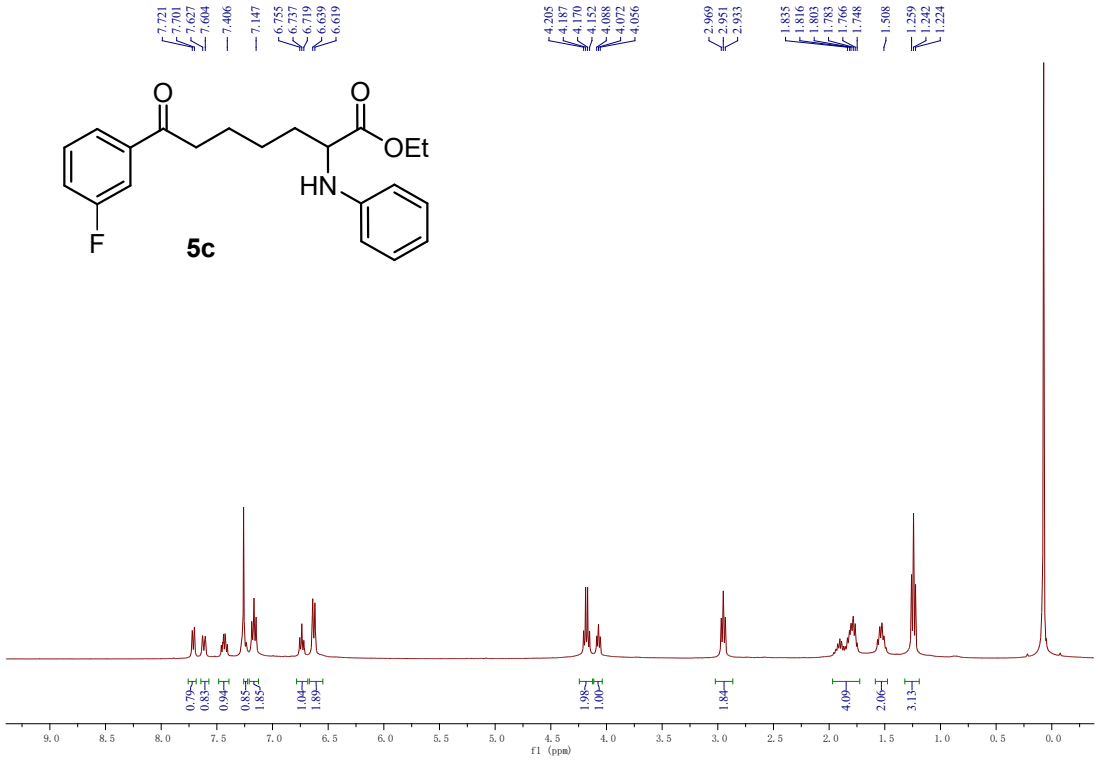
25.17
23.67
14.21
O
O
OEt
HN
Cl
5b
f1 (ppm)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6.639
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4.170
4.152
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4.072
4.056
2.969
2.951
2.933
1.835
1.816
1.803
1.783
1.766
1.748
1.508
1.259
1.242
1.224
O
O
OEt
HN
F
5c
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0.83
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1.98
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4.09
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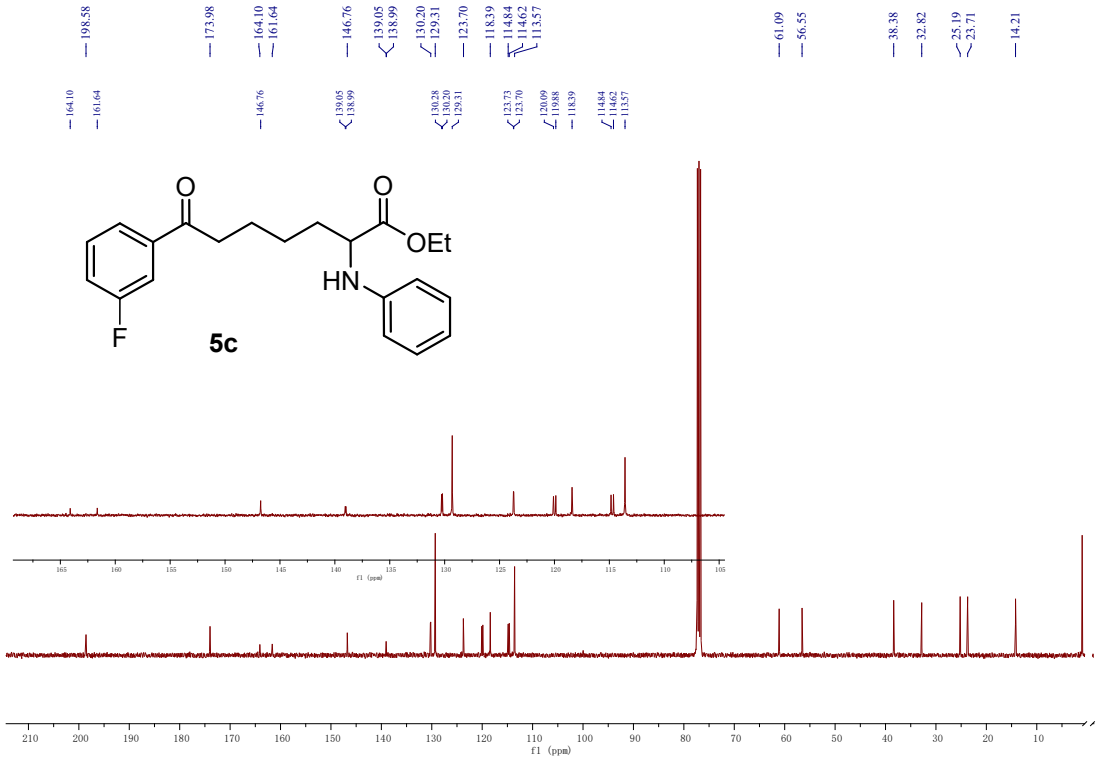
3.13
f1 (ppm)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114.62
113.57
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25.19
23.71
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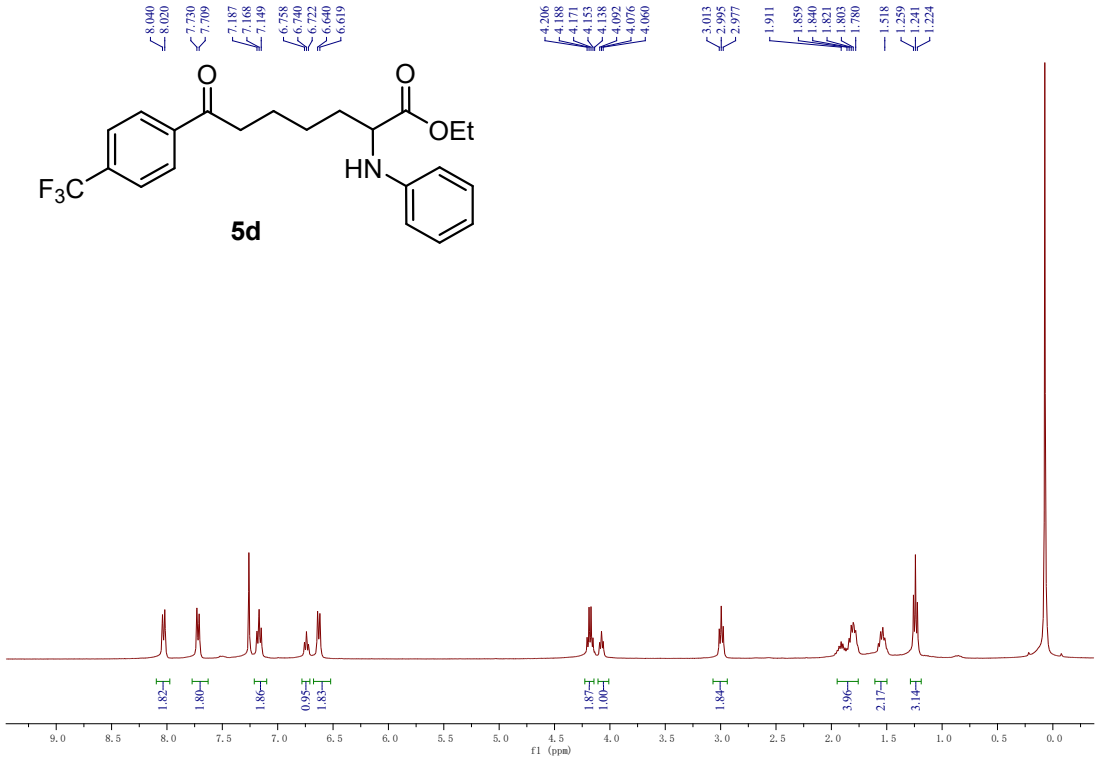
O
O
OEt
HN
F
5c
f1 (ppm)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6.722
6.640
6.619
4.206
4.188
4.171
4.153
4.138
4.092
4.076
4.060
3.013
2.995
2.977
1.911
1.859
1.840
1.821
1.803
1.780
1.518
1.259
1.241
1.224
O
O
OEt
HN
F3C
5d
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1.83
1.87
1.00
1.84
3.96
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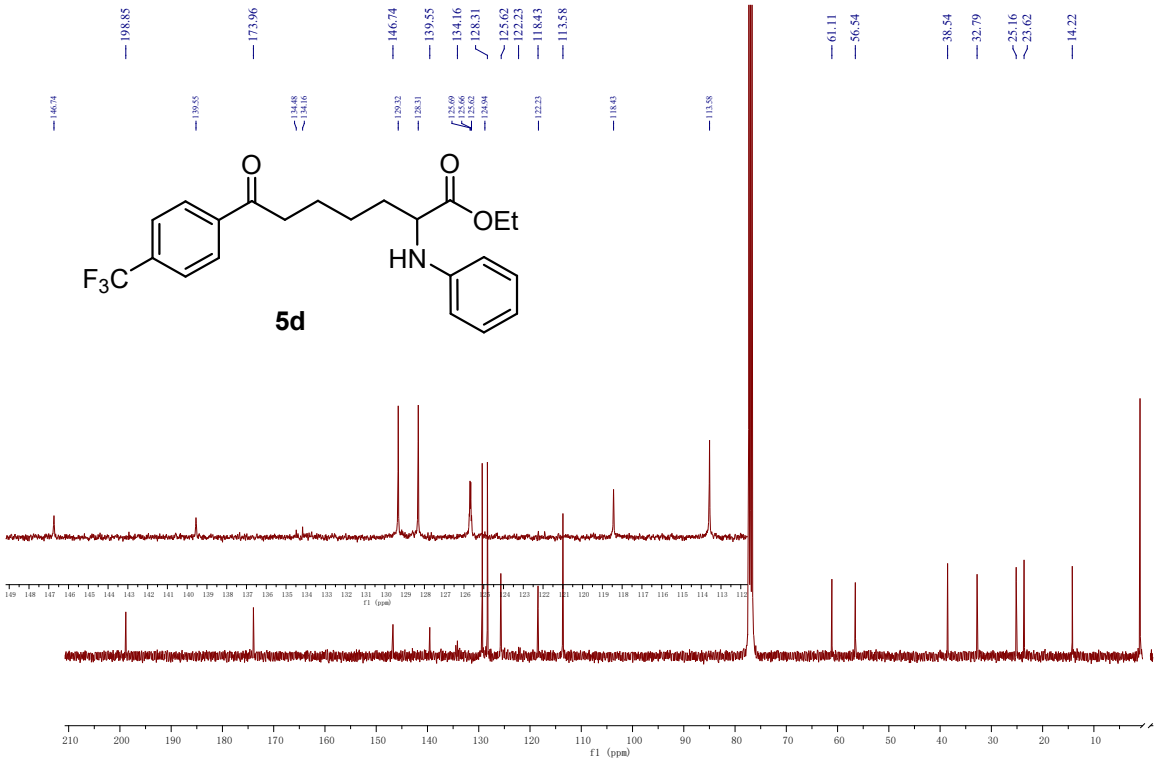
3.14
f1 (ppm)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122.23
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61.11
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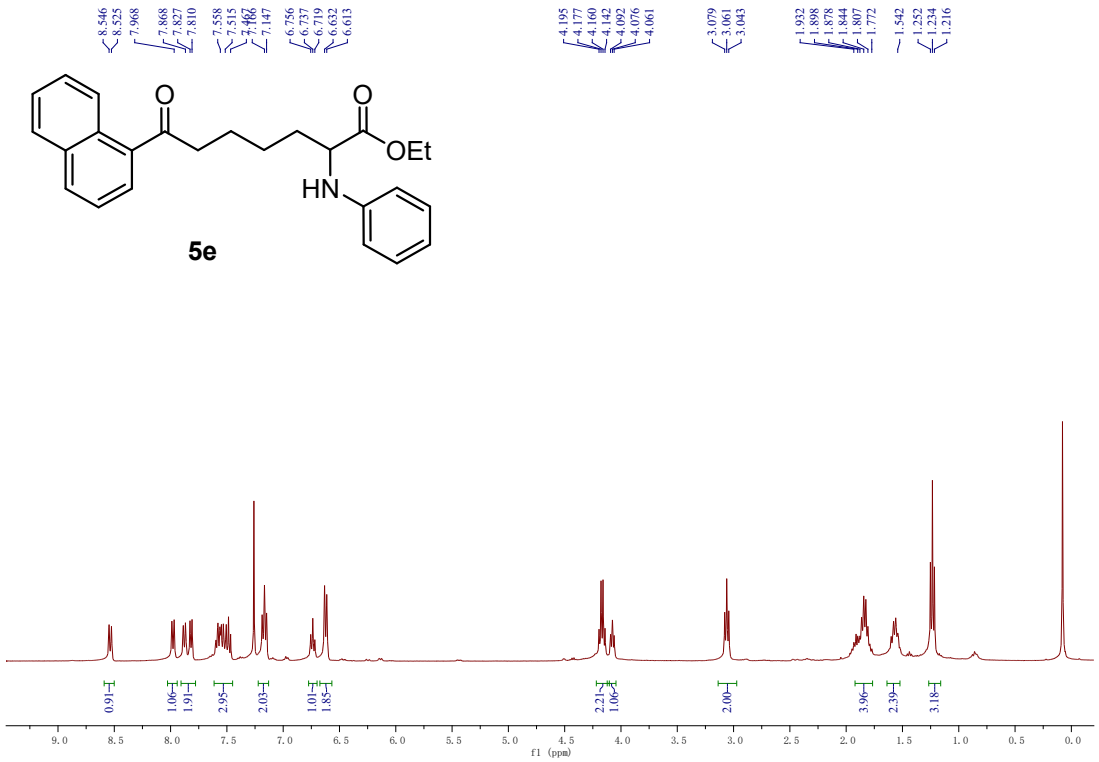
32.79
25.16
23.62
14.22
O
O
OEt
HN
F3C
5d
f1 (ppm)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6.632
6.613
4.195
4.177
4.160
4.142
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4.076
4.061
3.079
3.061
3.043
1.932
1.898
1.878
1.844
1.807
1.772
1.542
1.252
1.234
1.216
O
O
OEt
HN
5e
0.91
1.06
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1.01
1.85
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1.06
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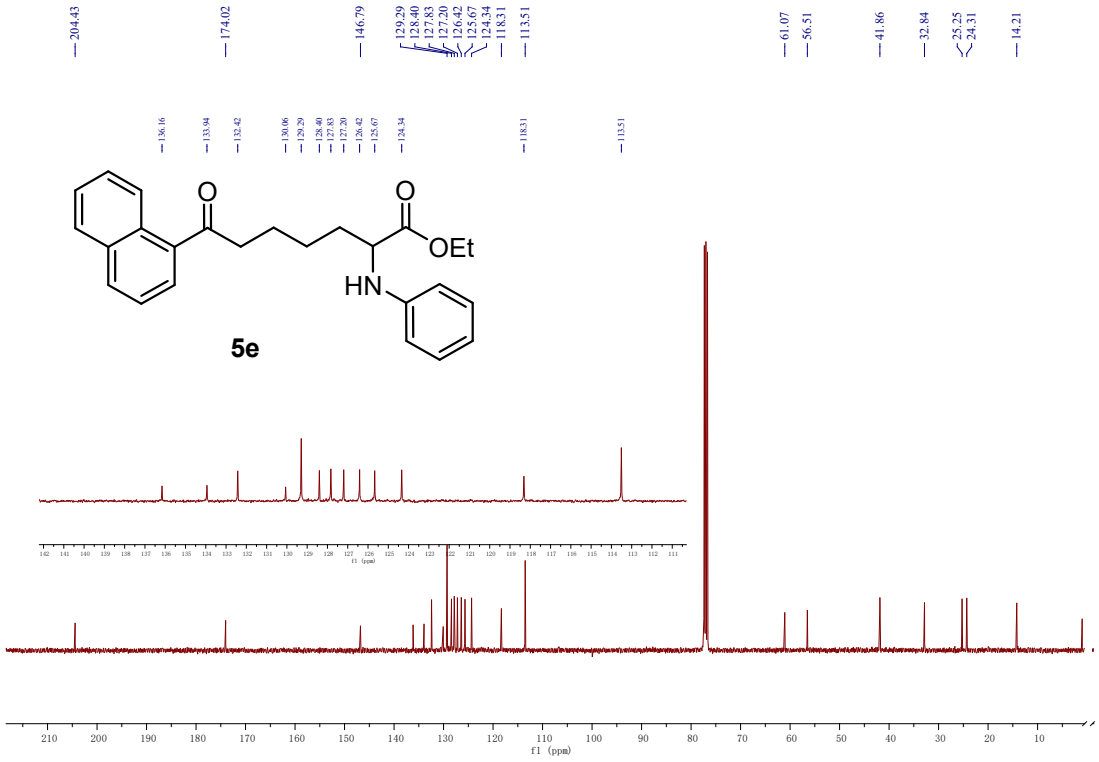
3.96
2.39
3.18
f1 (ppm)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126.42
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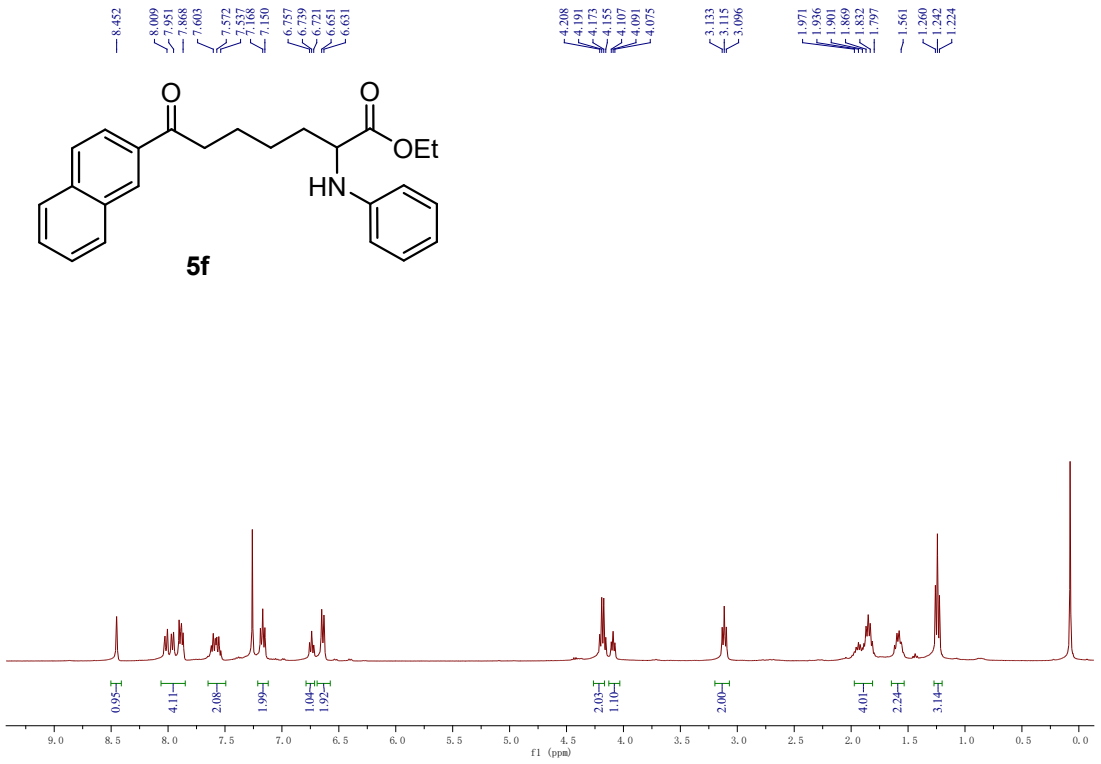
118.31
113.51
61.07
56.51
41.86
32.84
25.25
24.31
14.21
136.16
133.94
132.42
130.06
129.29
128.40
127.83
127.20
126.42
125.67
124.34
118.31
113.51
O
O
OEt
HN
5e
f1 (ppm)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6.631
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4.173
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3.096
1.971
1.936
1.901
1.869
1.832
1.797
1.561
1.260
1.242
1.224
O
O
OEt
HN
5f
0.95
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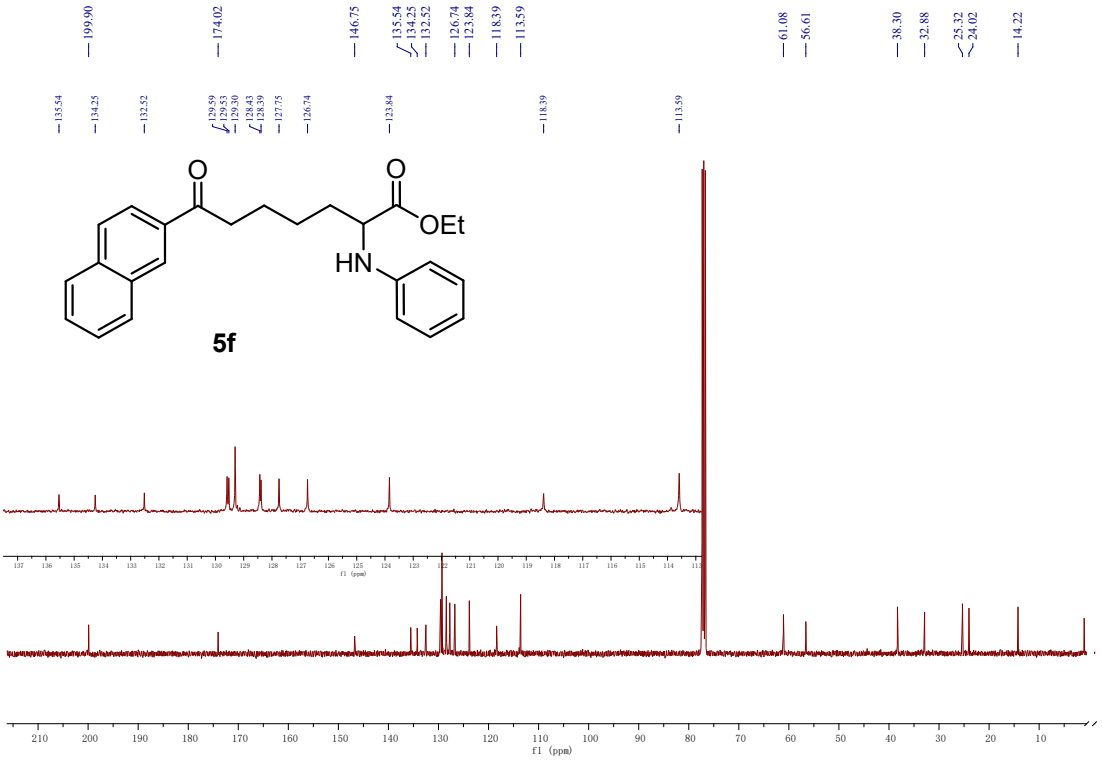
2.03
1.10
2.00
4.01
2.24
3.14
f1 (ppm)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113.59
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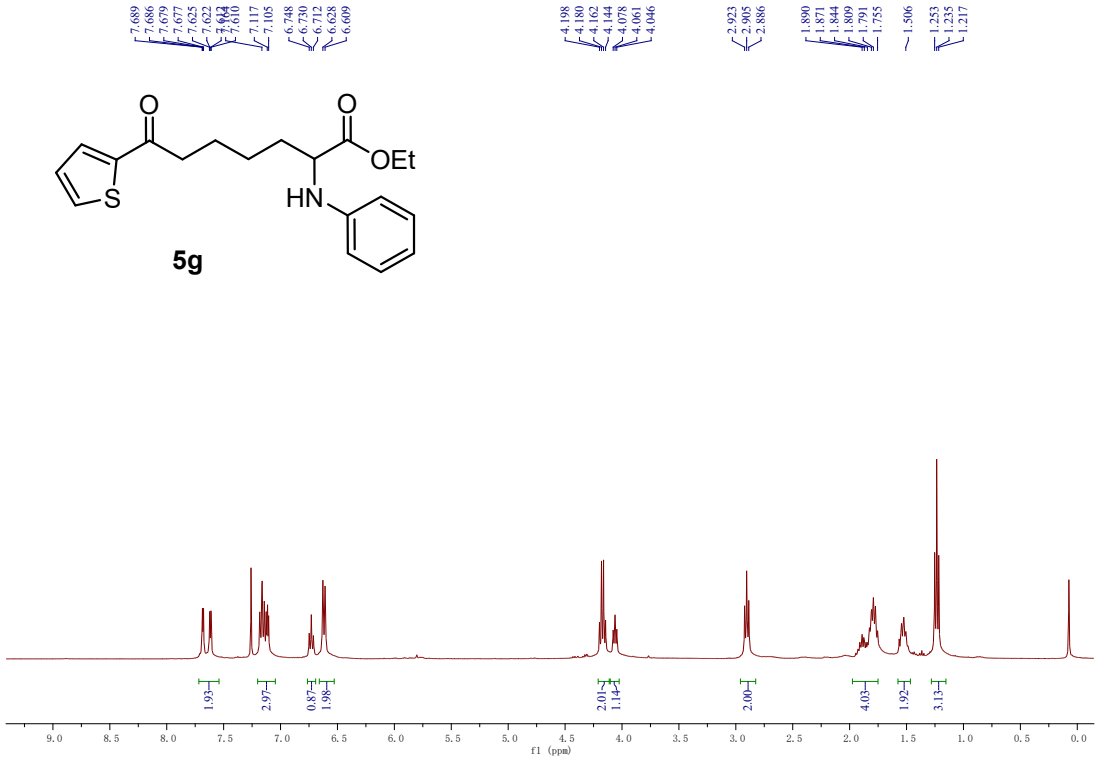
38.30
32.88
25.32
24.02
14.22
135.54
134.25
132.52
129.59
129.53
129.30
128.43
126.39
127.75
126.74
123.84
118.39
113.59
O
O
OEt
HN
5f
f1 (ppm)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6.628
6.609
4.198
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4.162
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2.886
1.890
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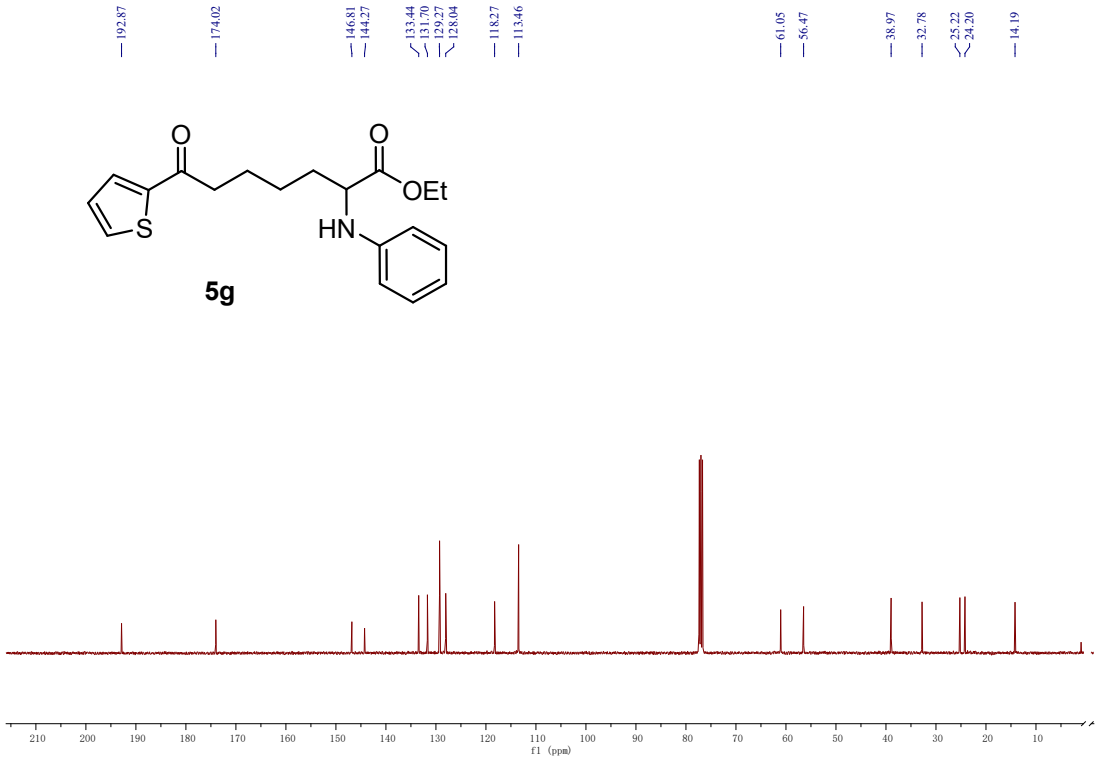
1.844
1.809
1.791
1.755
1.506
1.253
1.235
1.217
O
O
OEt
S
HN
5g
1.93
2.97
0.87
1.98
2.01
1.14
2.00
4.03
1.92
3.13
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.04
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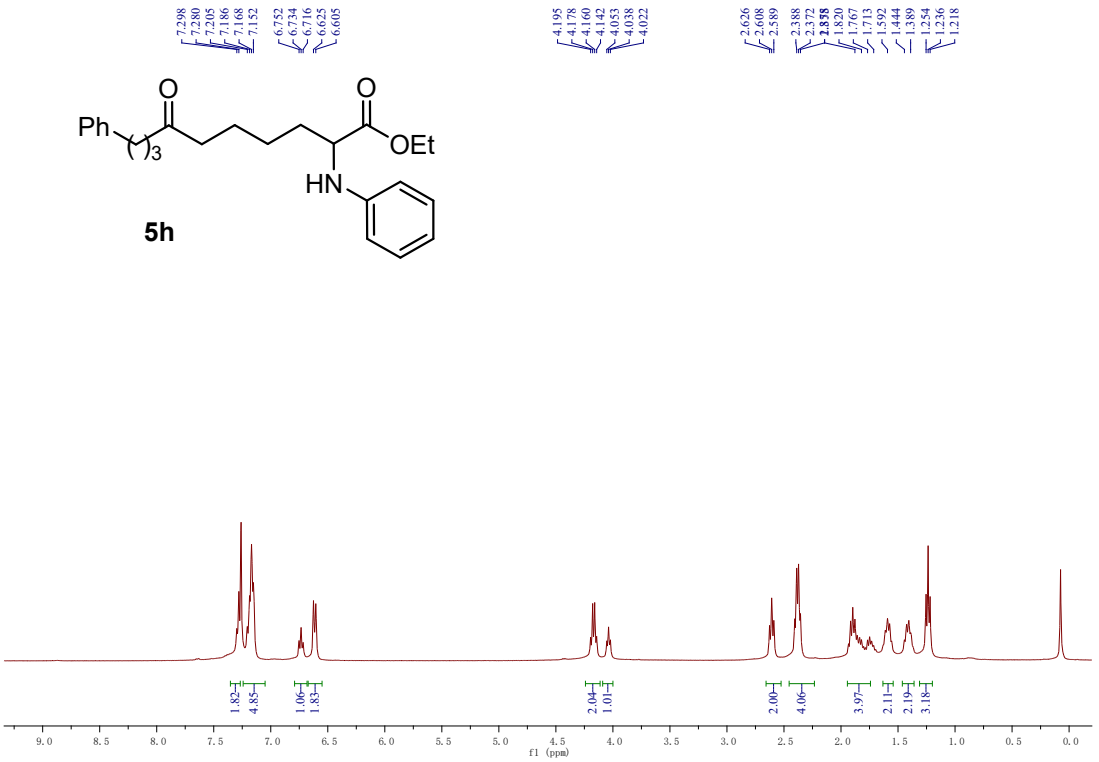
113.46
61.05
56.47
38.97
32.78
25.22
24.20
14.19
O
O
OEt
S
HN
5g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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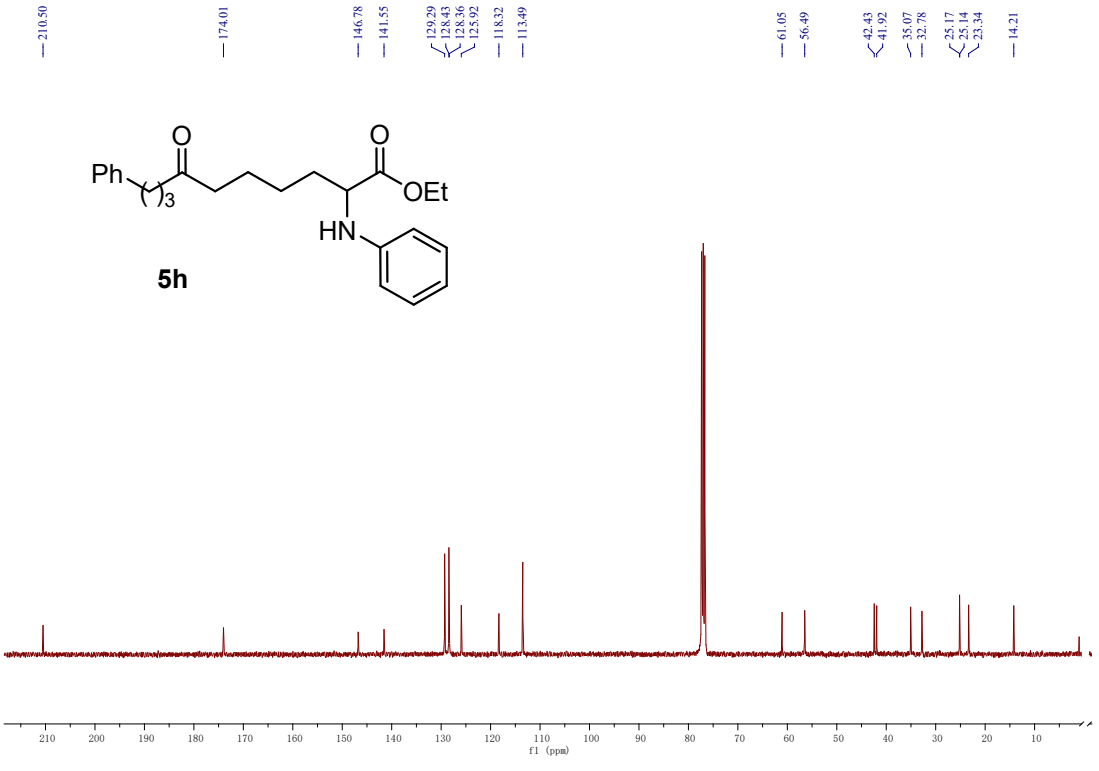
7.186
7.168
7.152
6.752
6.734
6.716
6.625
6.605
4.195
4.178
4.160
4.142
4.053
4.038
4.022
2.626
2.608
2.589
2.388
2.372
2.358
1.820
1.767
1.713
1.592
1.444
1.389
1.254
1.236
1.218
Ph
3
O
O
OEt
HN
5h
1.82
4.85
1.06
1.83
2.04
1.01
2.00
4.06
3.97
2.11
2.19
3.18
f1 (ppm)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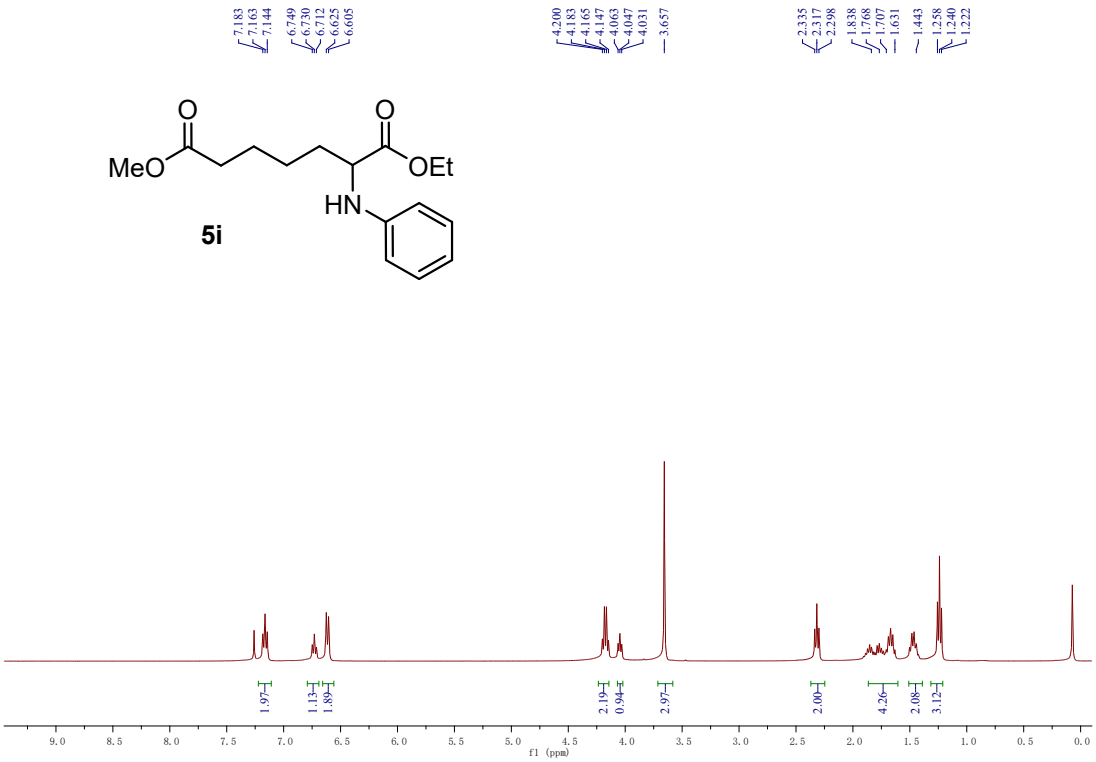
128.43
128.36
125.92
118.32
113.49
61.05
56.49
42.43
41.92
35.07
32.78
25.17
25.14
23.34
14.21
Ph
3
O
O
OEt
HN
5h
f1 (ppm)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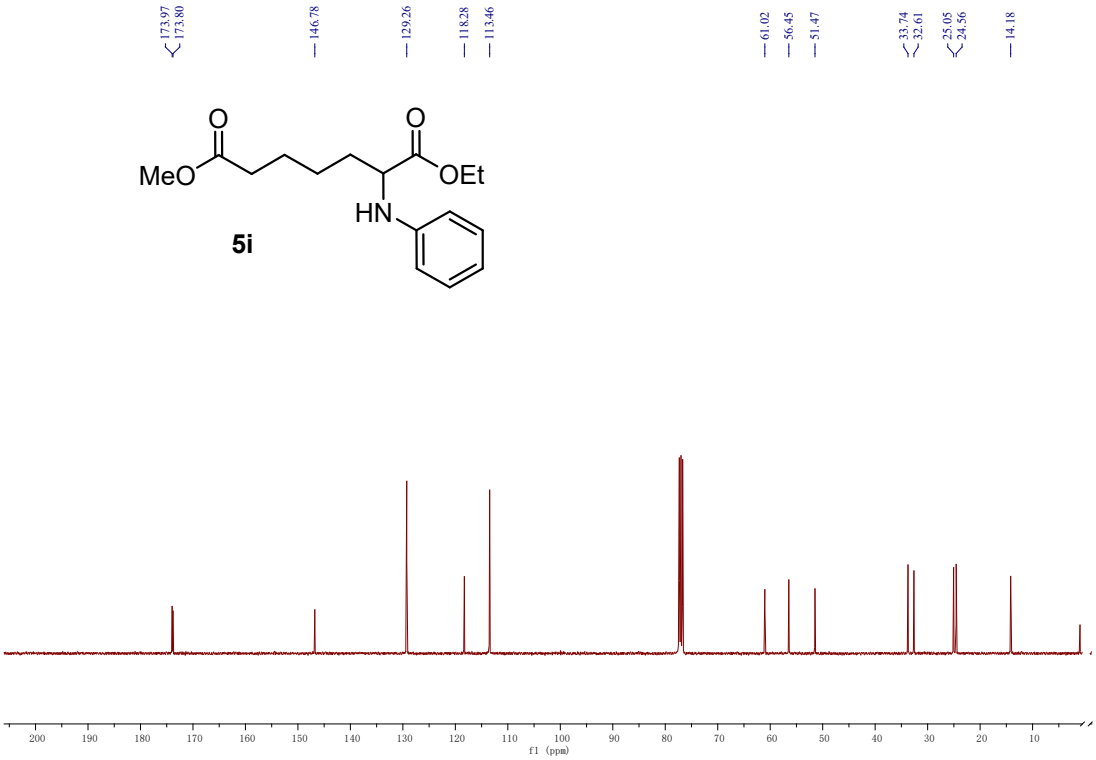
7.163
7.144
6.749
6.730
6.712
6.625
6.605
4.200
4.183
4.165
4.147
4.063
4.047
4.031
3.657
2.335
2.317
2.298
1.838
1.768
1.707
1.631
1.443
1.258
1.240
1.222
O
O
MeO
OEt
HN
5i
1.97
1.13
1.89
2.19
0.94
2.97
2.00
4.26
2.08
3.12
f1 (ppm)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61.02
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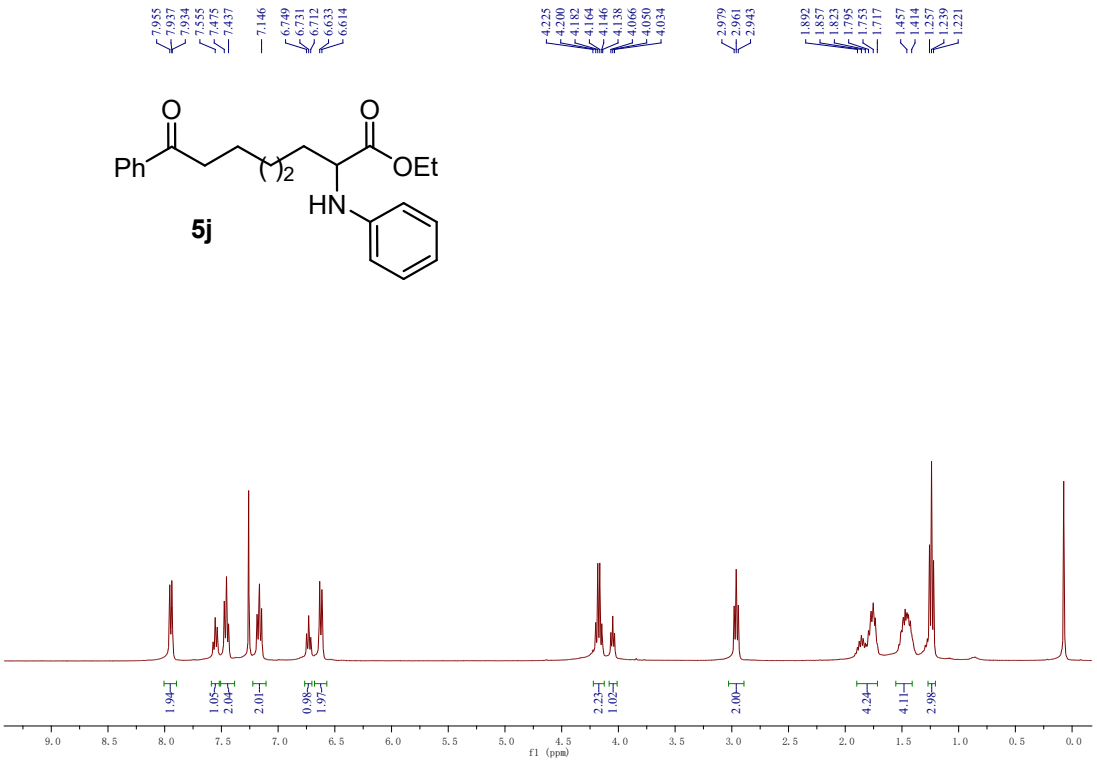
32.61
25.05
24.56
14.18
O
O
MeO
OEt
HN
5i
f1 (ppm)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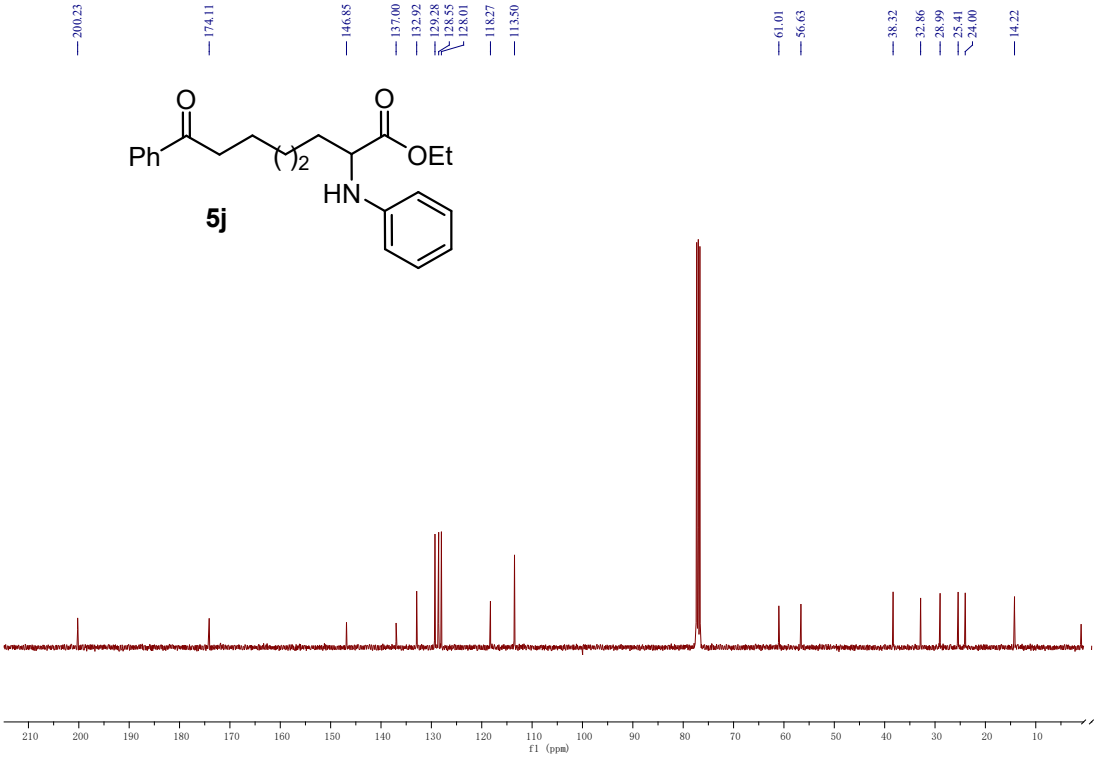
7.937
7.934
7.555
7.475
7.437
7.146
6.749
6.731
6.712
6.633
6.614
4.225
4.200
4.182
4.164
4.146
4.138
4.066
4.050
4.034
2.979
2.961
2.943
1.892
1.857
1.823
1.795
1.753
1.717
1.457
1.414
1.257
1.239
1.221
O
O
Ph
OEt
HN
5j
1.94
1.05
2.04
2.01
0.98
1.97
2.23
1.02
2.00
4.24
4.11
2.98
f1 (ppm)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174.11
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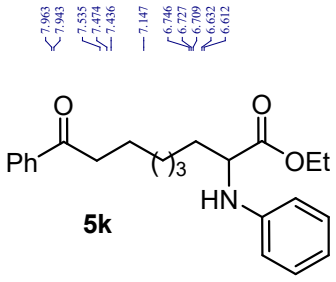
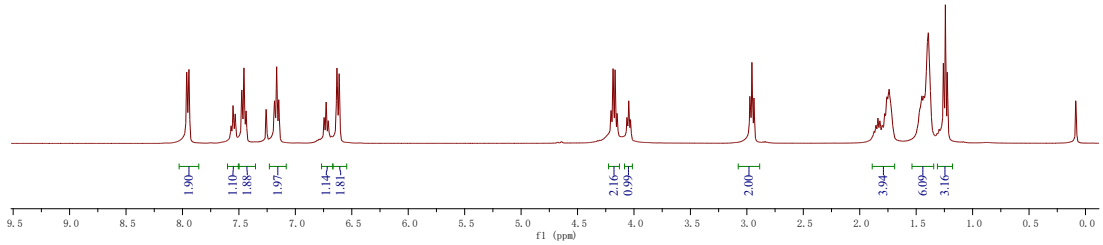
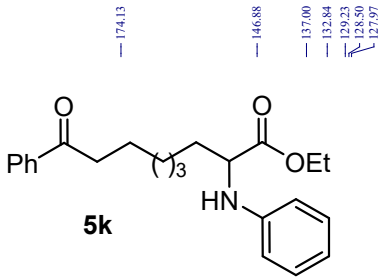
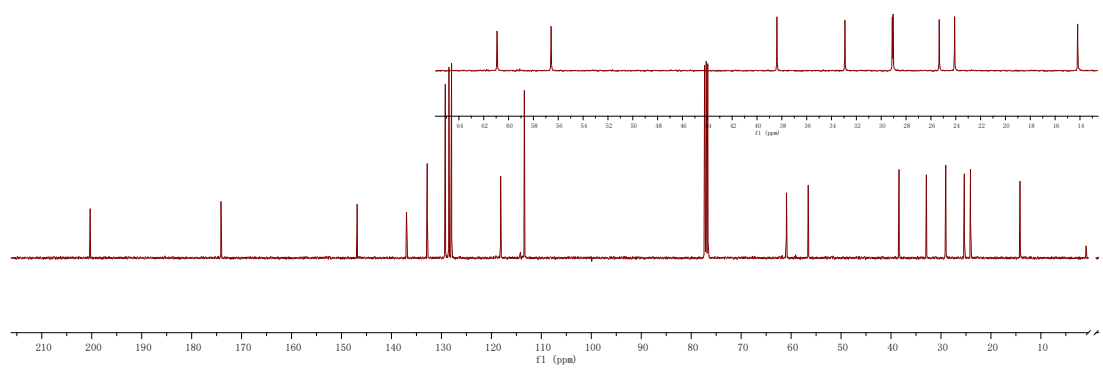
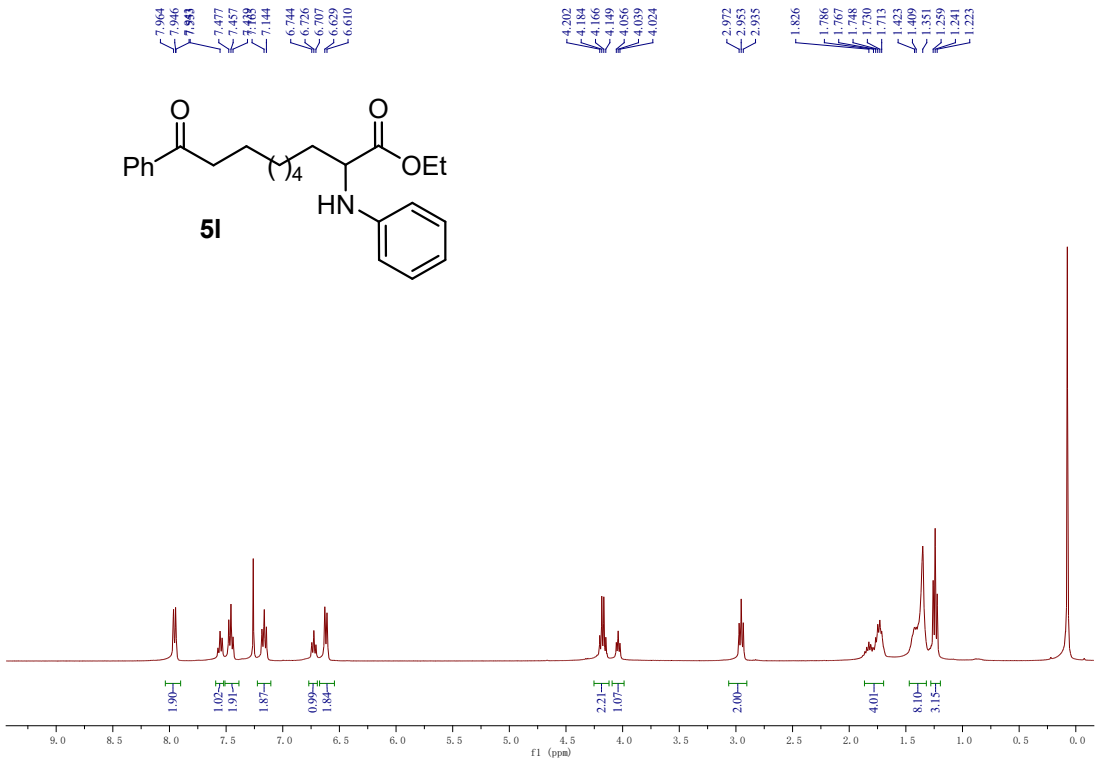
137.00
132.92
129.28
128.55
128.01
118.27
113.50
61.01
56.63
38.32
32.86
28.99
25.41
24.00
14.22
O
O
Ph
OEt
HN
5j
f1 (ppm)


Ph
O
O
OEt
HN
3
5k




Ph
O
O
OEt
HN
3
5k

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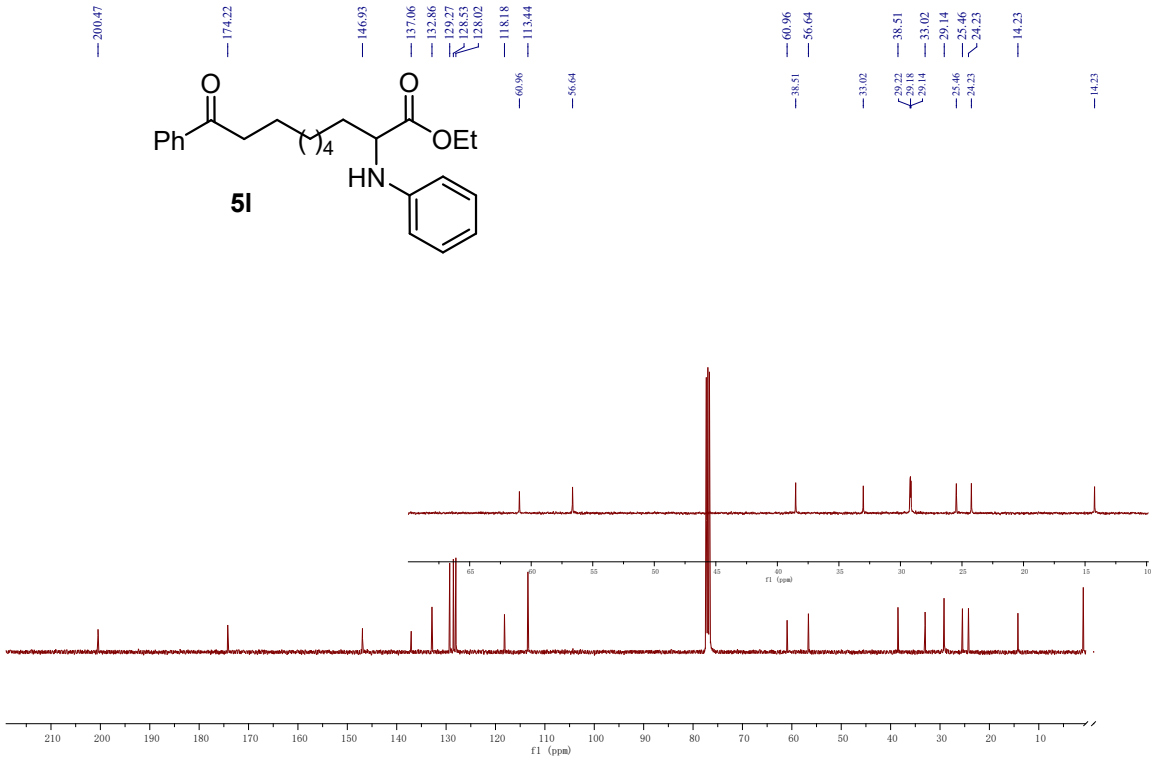
7.943
7.477
7.457
7.144
6.744
6.726
6.707
6.629
6.610
4.202
4.184
4.166
4.149
4.056
4.039
4.024
2.972
2.953
2.935
1.826
1.786
1.767
1.748
1.730
1.713
1.423
1.409
1.351
1.259
1.241
1.223
O
O
Ph
OEt
HN
4
5l
1.90
1.02
1.91
1.87
0.99
1.84
2.21
1.07
2.00
4.01
8.10
3.15
f1 (ppm)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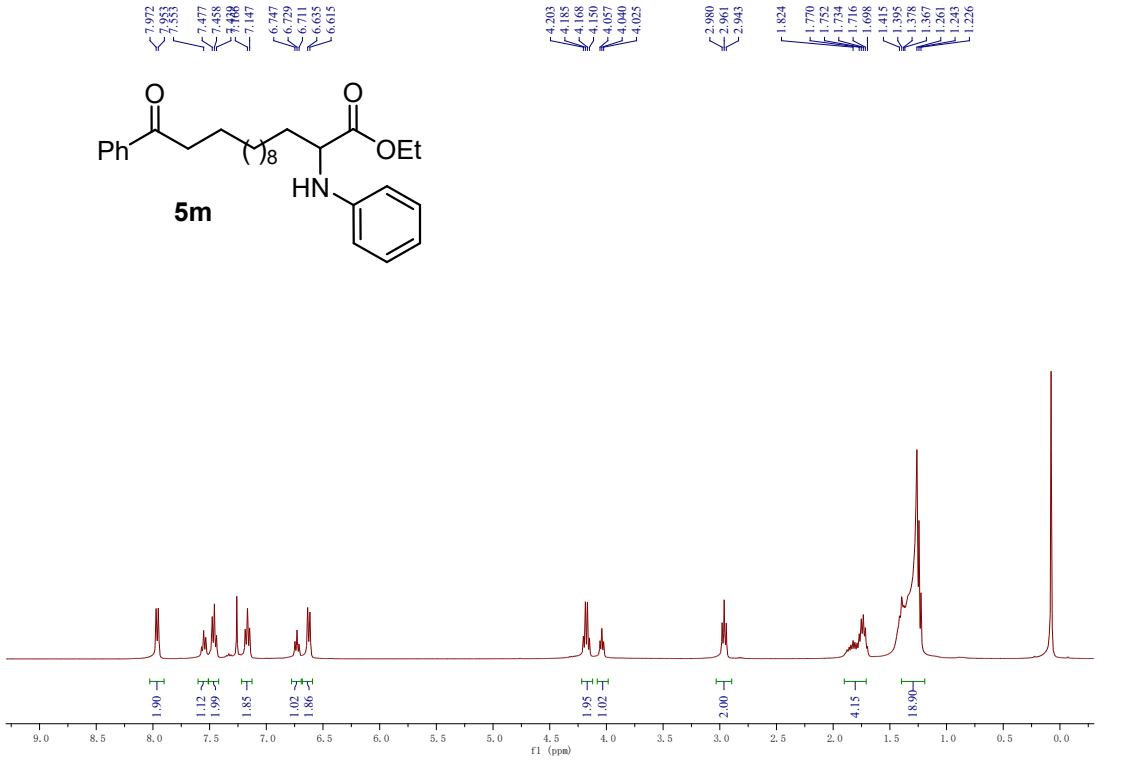
146.93
137.06
132.86
129.27
128.53
128.02
118.18
113.44
60.96
56.64
38.51
33.02
29.22
29.18
29.14
25.46
24.23
14.23
O
O
Ph
OEt
HN
4
5l
f1 (ppm)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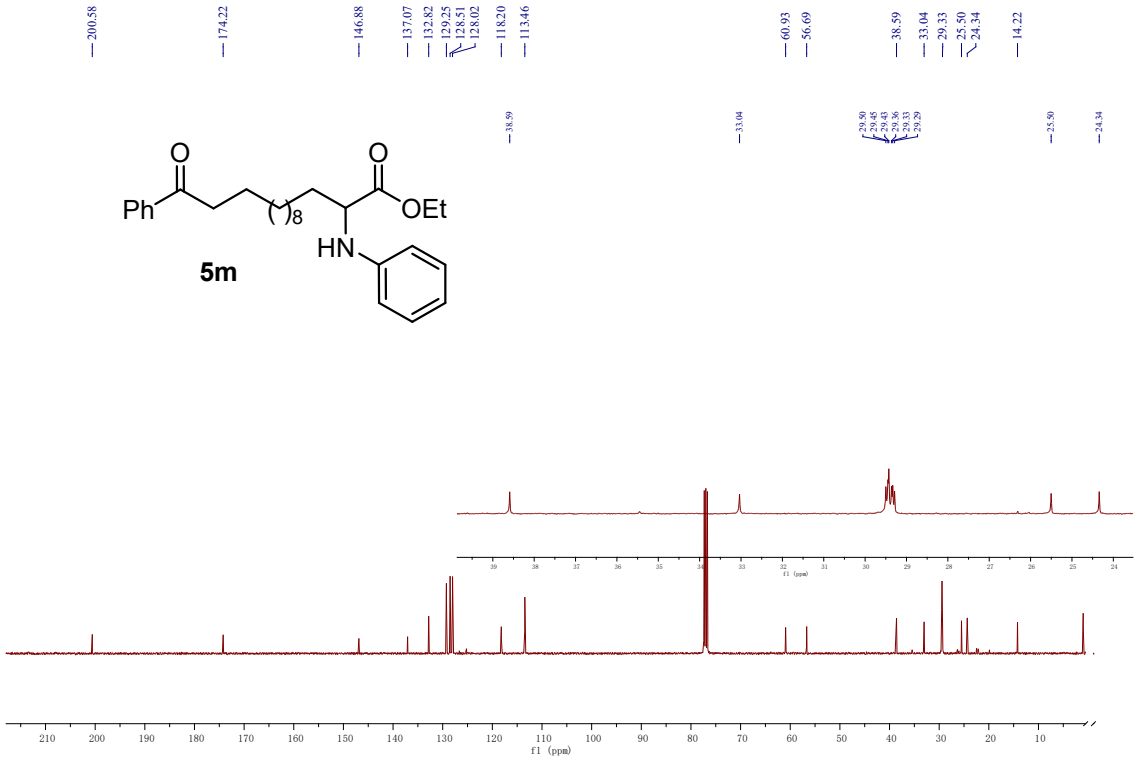
7.953
7.935
7.477
7.458
7.439
7.147
6.747
6.729
6.711
6.635
6.615
4.203
4.185
4.168
4.150
4.057
4.040
4.025
2.980
2.961
2.943
1.824
1.770
1.752
1.734
1.716
1.698
1.415
1.395
1.378
1.367
1.261
1.243
1.226
O
O
Ph
8
OEt
HN
5m
1.90
1.12
1.99
1.85
1.02
1.86
1.95
1.02
2.00
4.15
18.90
f1 (ppm)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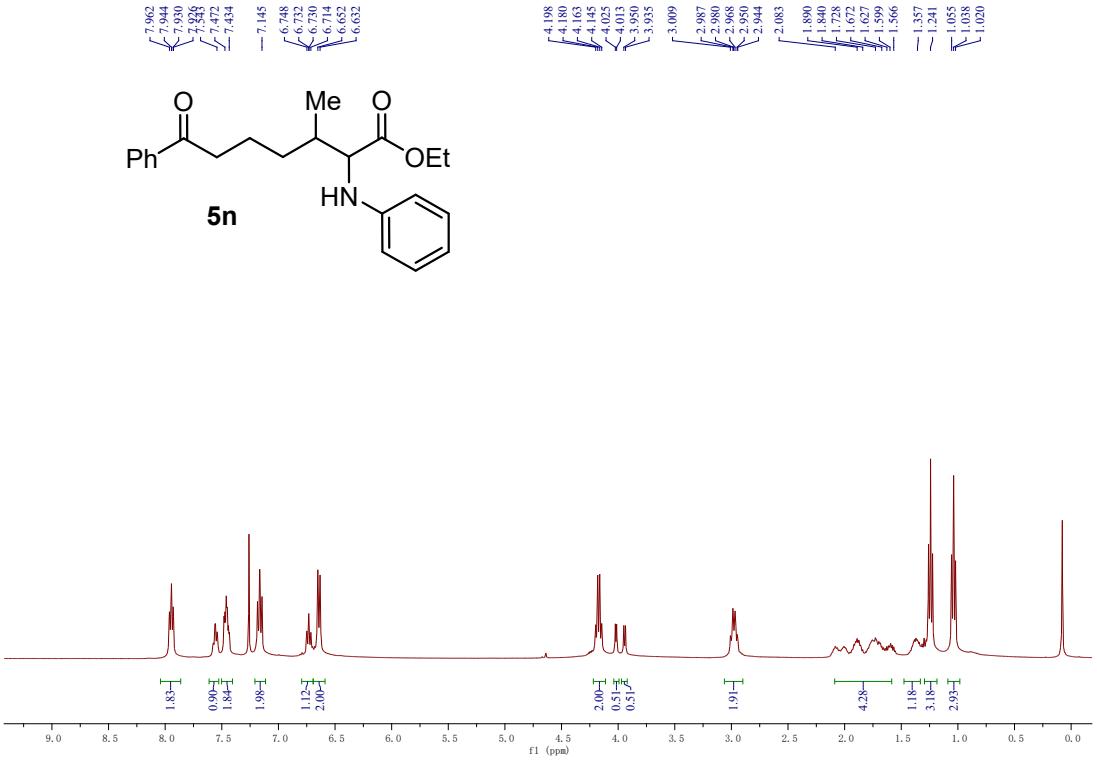
137.07
132.82
129.25
128.51
128.02
118.20
113.46
60.93
56.69
38.59
33.04
29.33
25.50
24.34
14.22
O
O
Ph
8
OEt
HN
5m
f1 (ppm)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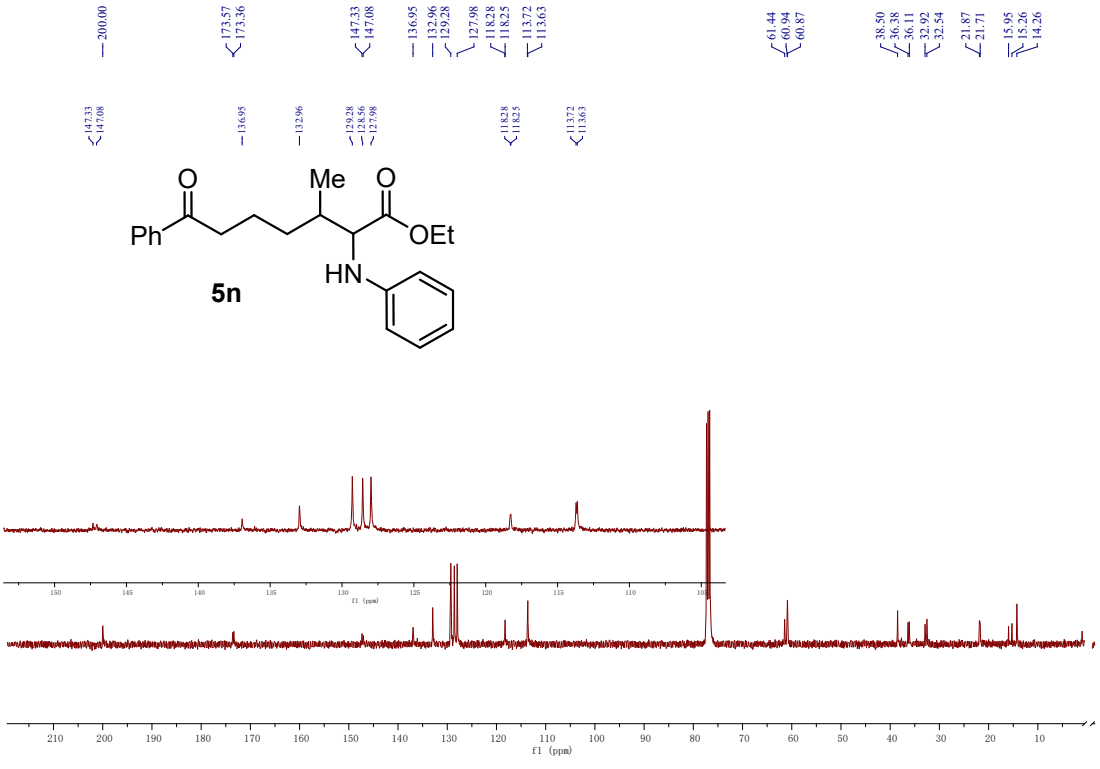
6.730
6.714
6.652
6.632
4.198
4.180
4.163
4.145
4.025
4.013
3.950
3.935
3.009
2.987
2.980
2.968
2.950
2.944
2.083
1.890
1.840
1.728
1.672
1.627
1.599
1.566
1.357
1.241
1.055
1.038
1.020
O
Me
O
Ph
OEt
HN
5n
1.83
0.90
1.84
1.98
1.12
2.00
2.00
0.51
0.51
1.91
4.28
1.18
3.18
2.93
f1 (ppm)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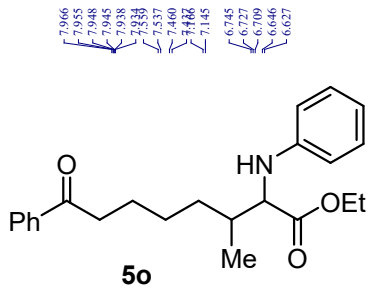
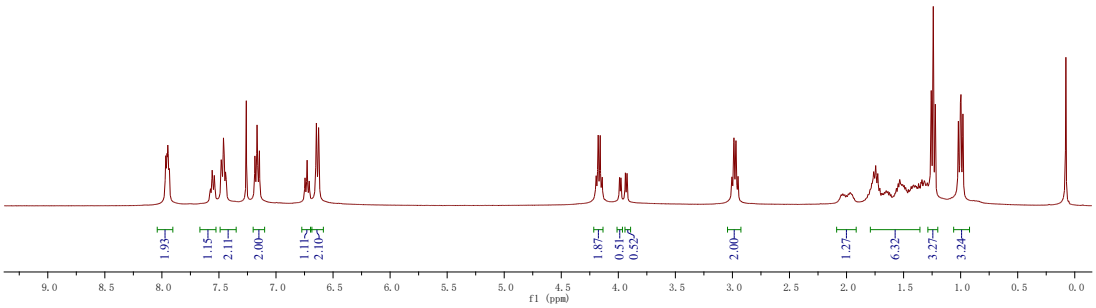
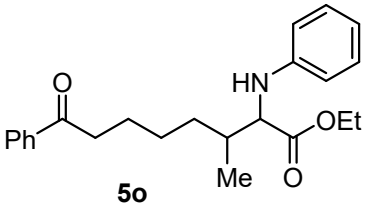
147.08
136.95
132.96
129.28
127.98
118.28
118.25
113.72
113.63
61.44
60.94
60.87
38.50
36.38
36.11
32.92
32.54
21.87
21.71
15.95
15.26
14.26
O
Me
O
Ph
OEt
HN
5n
f1 (ppm)

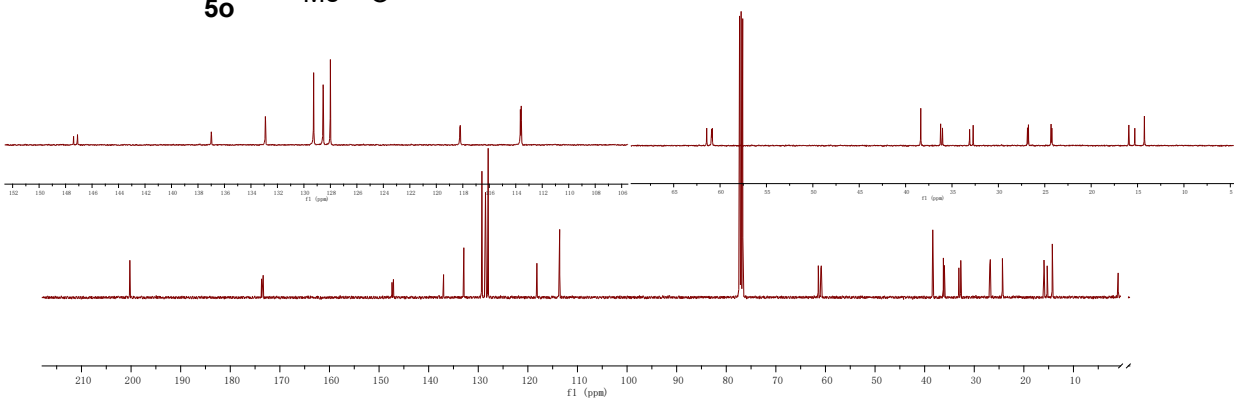


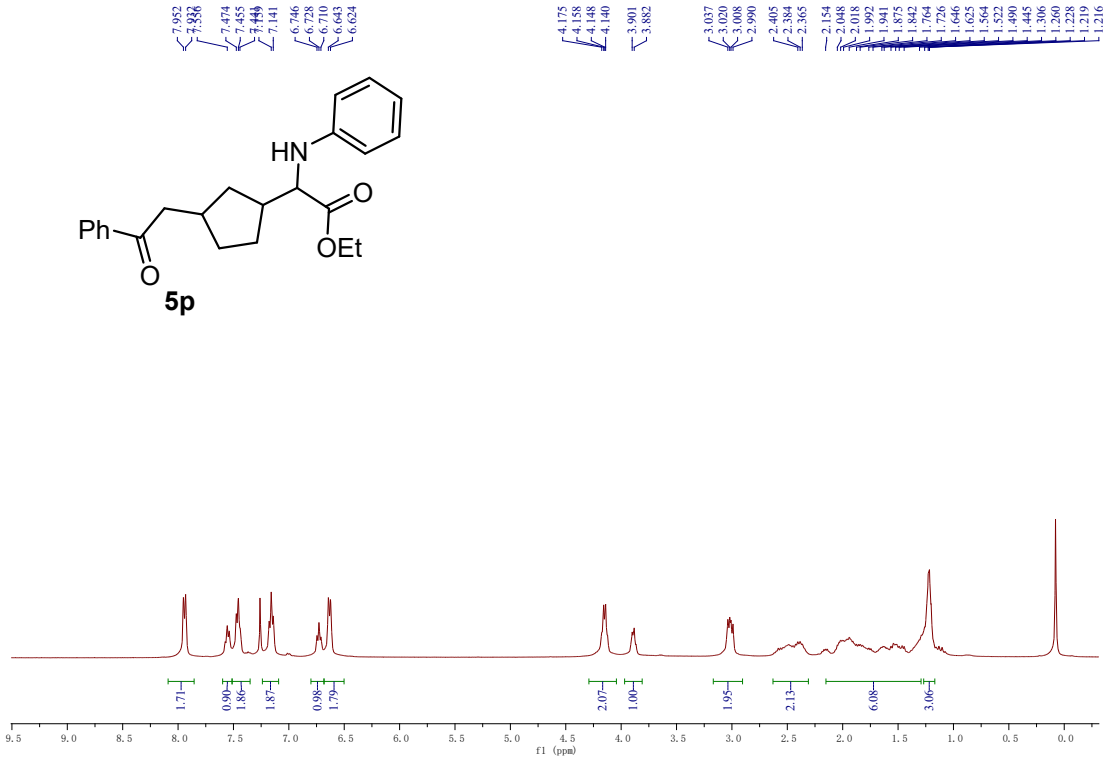
5o

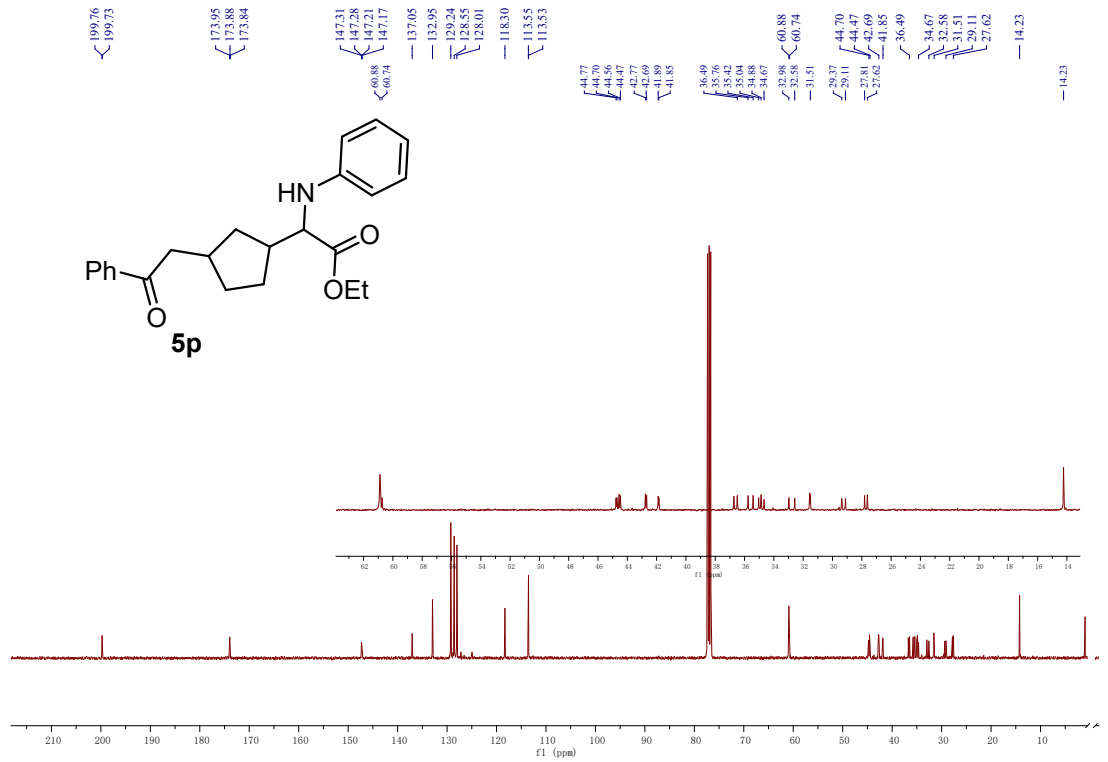


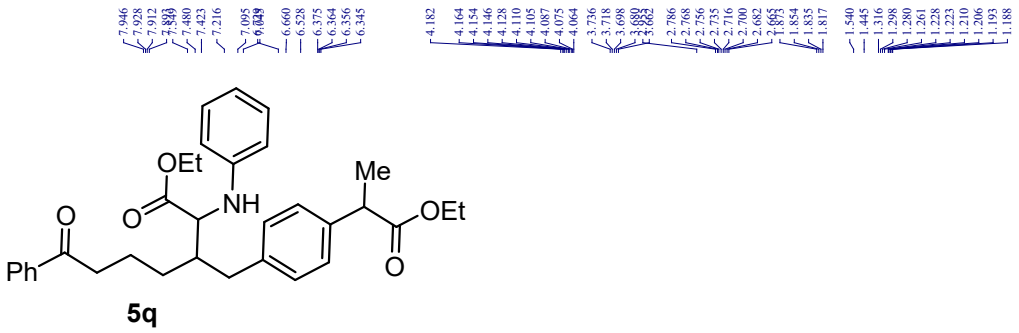


5o

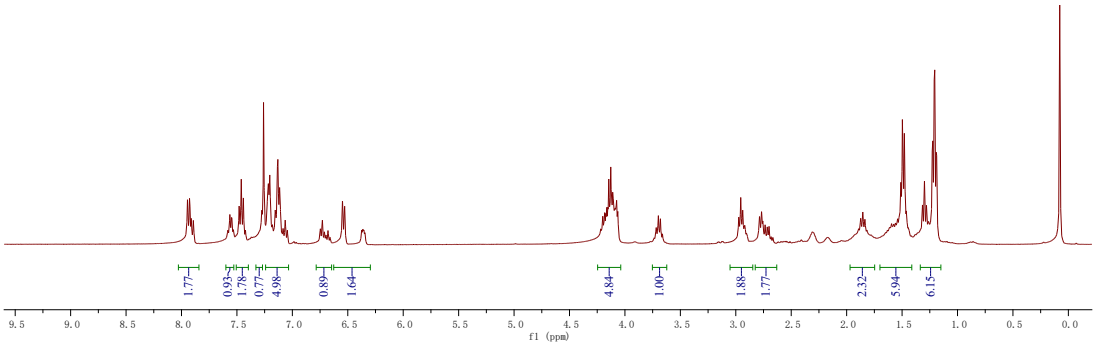


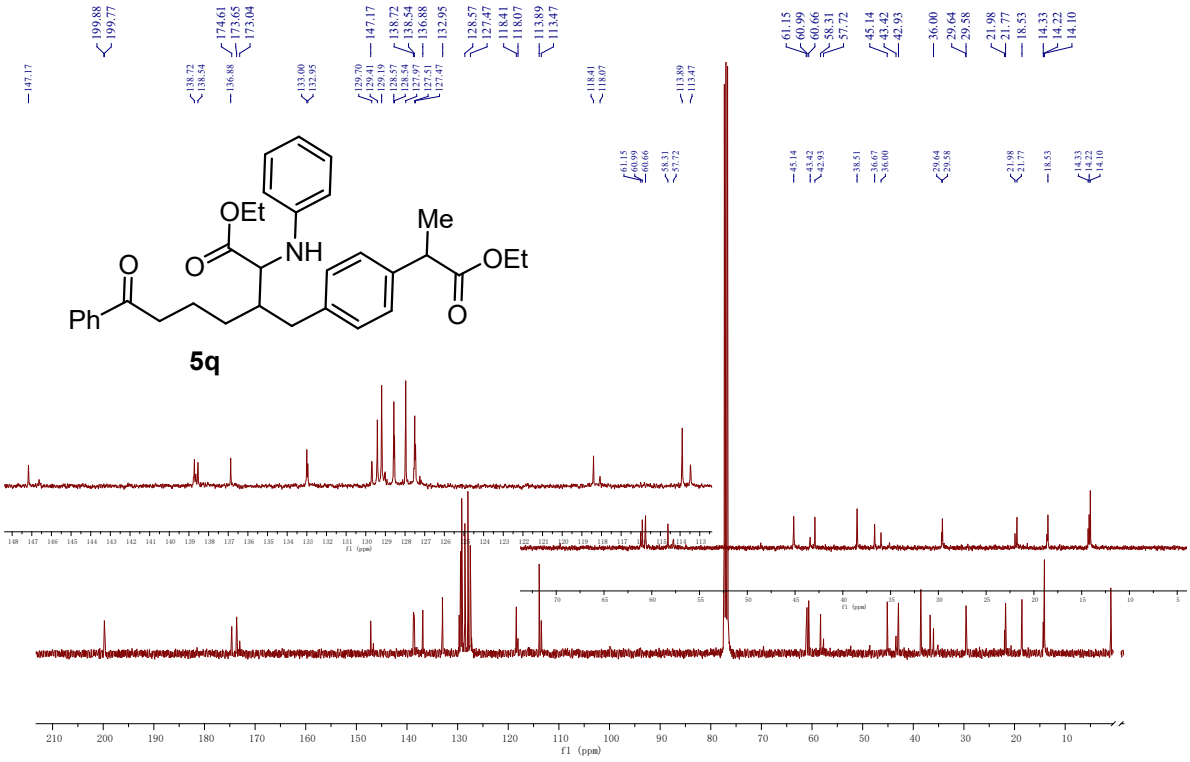

HN
O
Ph
O
OEt
O
5p


HN
O
Ph
O
OEt
O
5p

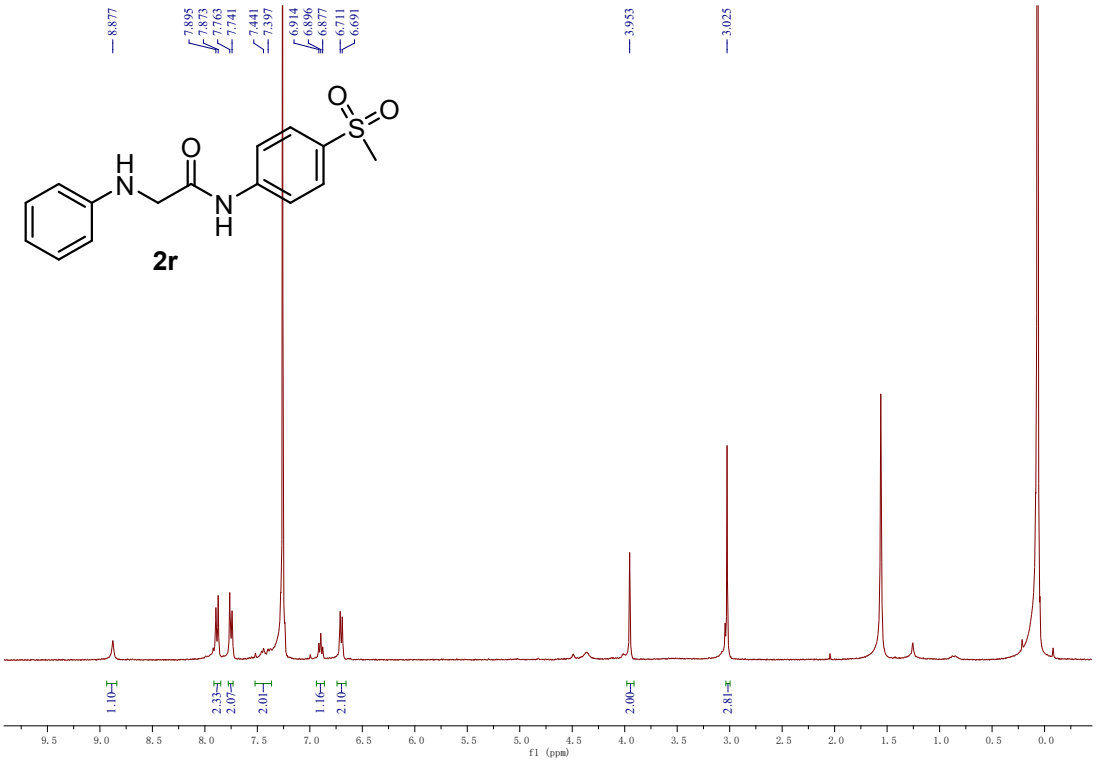


5q





5q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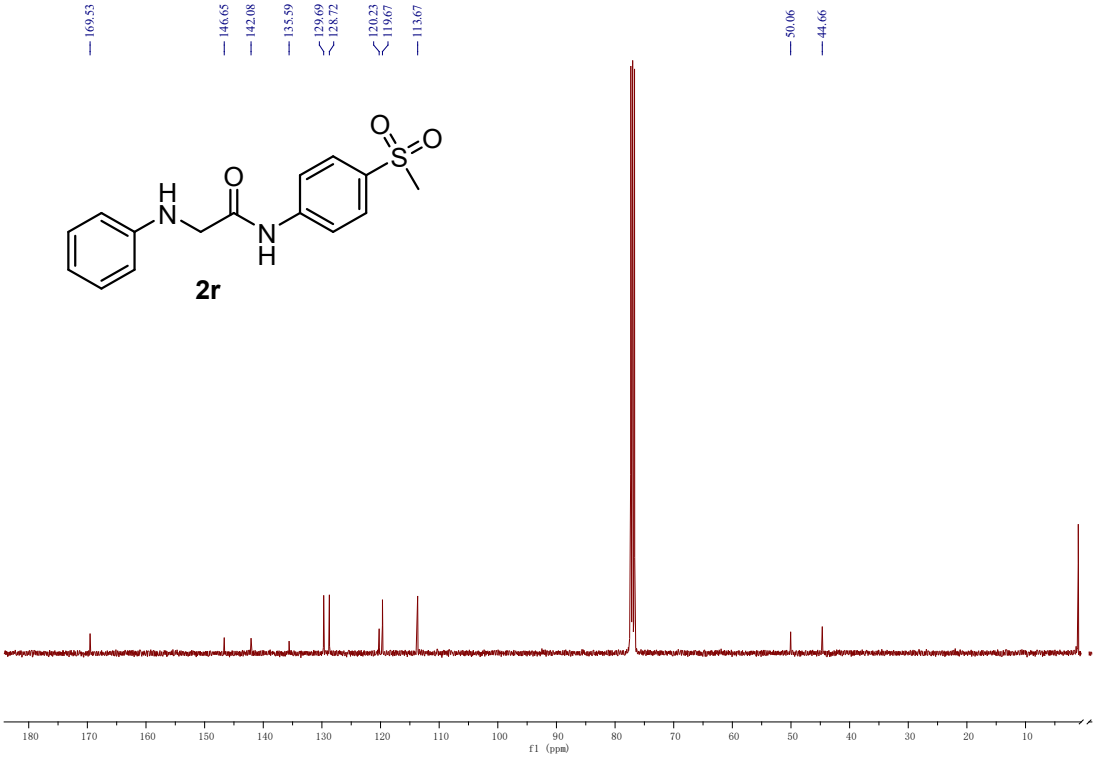
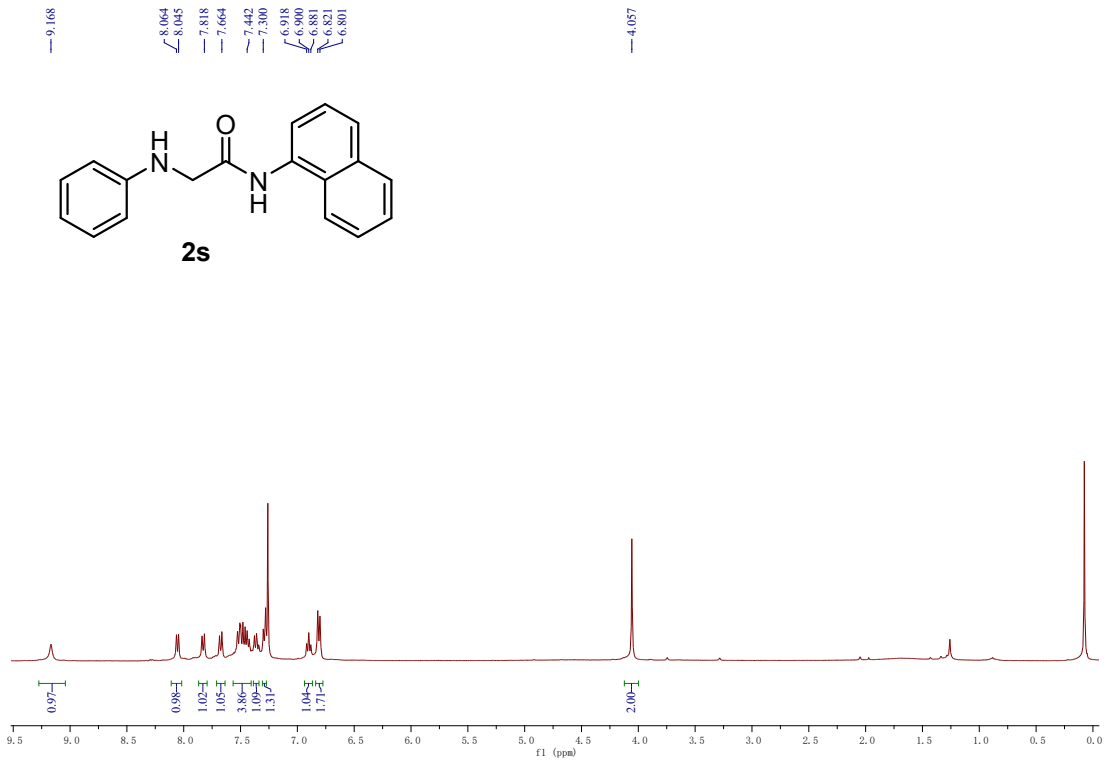
7.741
7.441
7.397
6.914
6.896
6.877
6.711
6.691
3.953
3.025
1.10
2.33
2.07
2.01
1.16
2.10
2.00
2.81
f1 (ppm)
2r


169.53
146.65
142.08
135.59
129.69
128.72
120.23
119.67
113.67
50.06
44.66
f1 (ppm)
2r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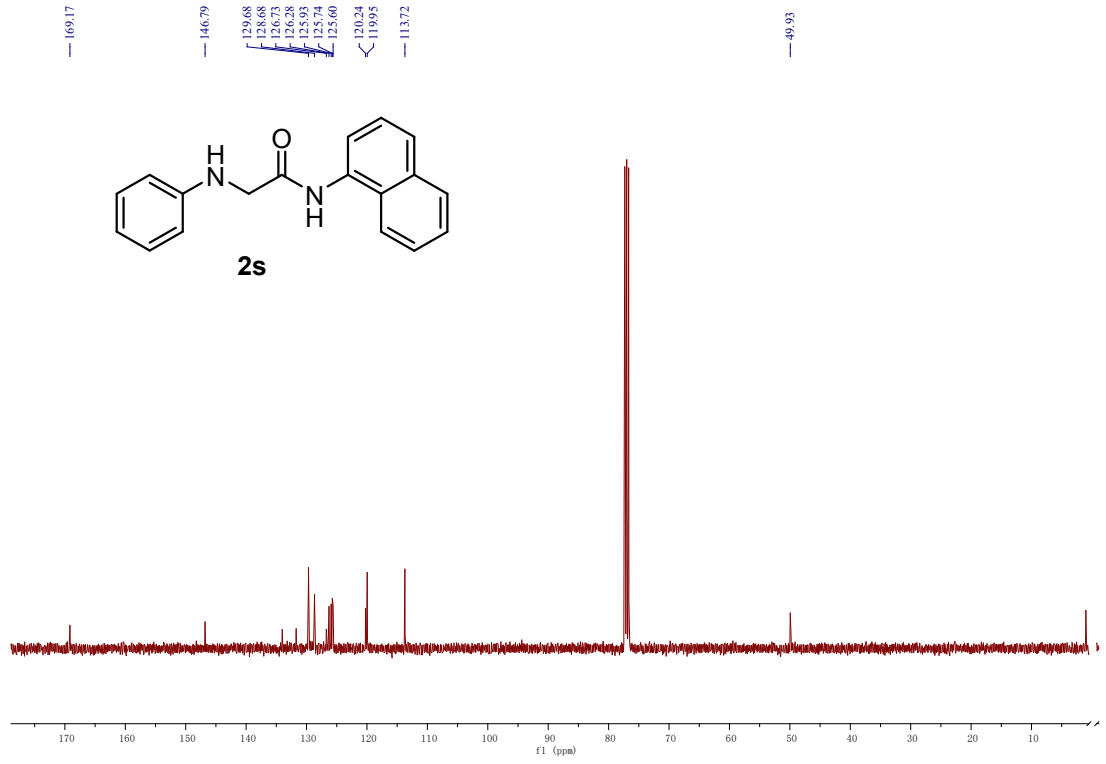
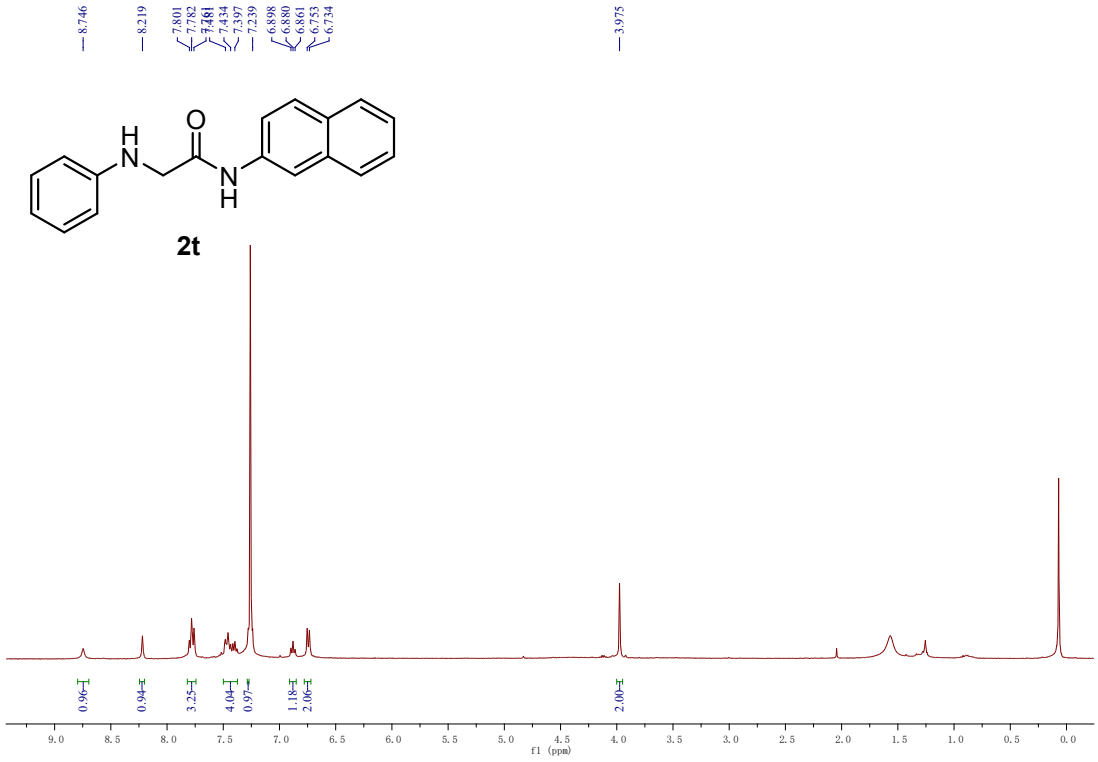
8.045
7.818
7.664
7.442
7.300
6.918
6.900
6.881
6.821
6.801
4.057
2s
0.97
0.98
1.02
1.05
3.86
1.09
1.31
1.04
1.71
2.00
f1 (ppm)


169.17
146.79
129.68
128.68
126.73
126.28
125.93
125.74
125.60
120.24
119.95
113.72
49.93
2s
f1 (ppm)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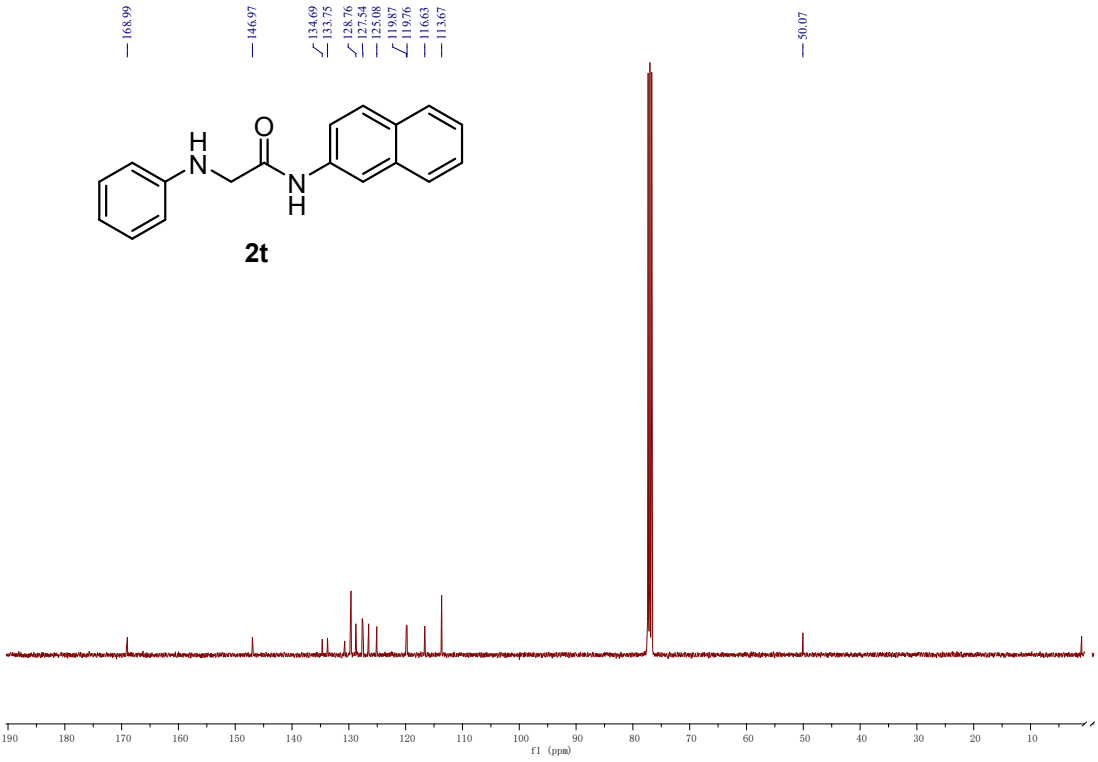
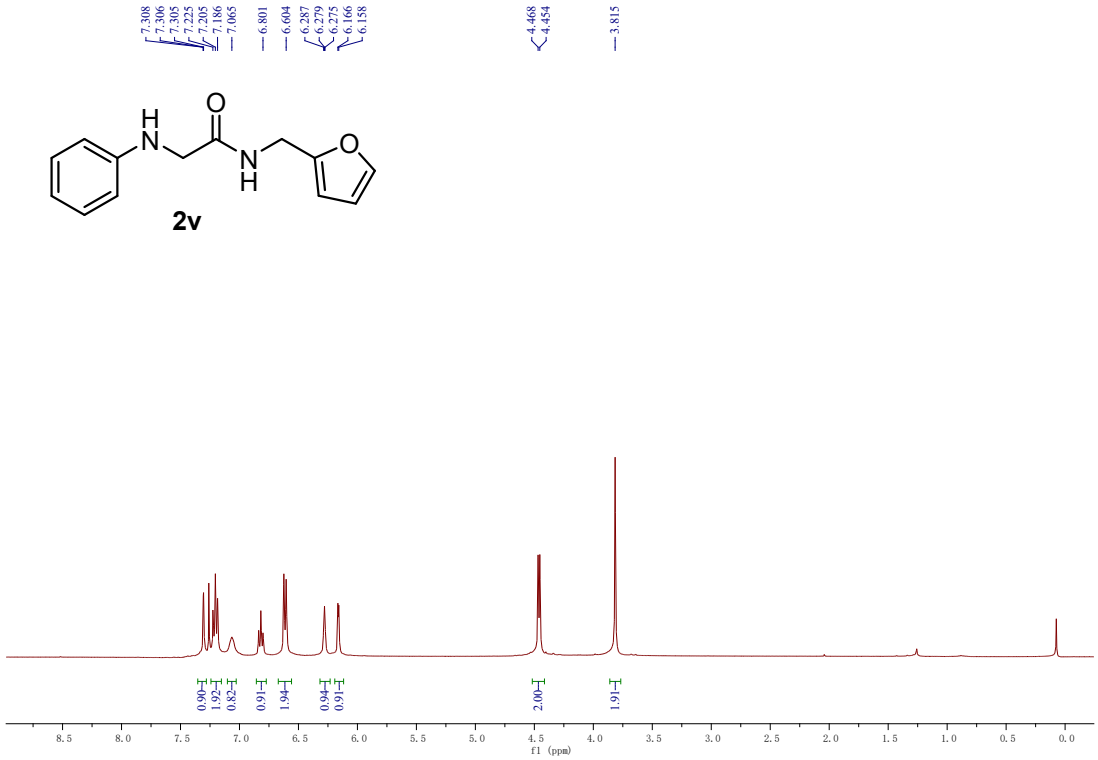
7.801
7.782
7.481
7.434
7.397
7.239
6.898
6.880
6.861
6.753
6.734
3.975
0.96
0.94
3.25
4.04
0.97
1.18
2.06
2.00
f1 (ppm)

2t


168.99
146.97
134.69
133.75
128.76
127.54
125.08
119.87
119.76
116.63
113.67
50.07
f1 (ppm)

2t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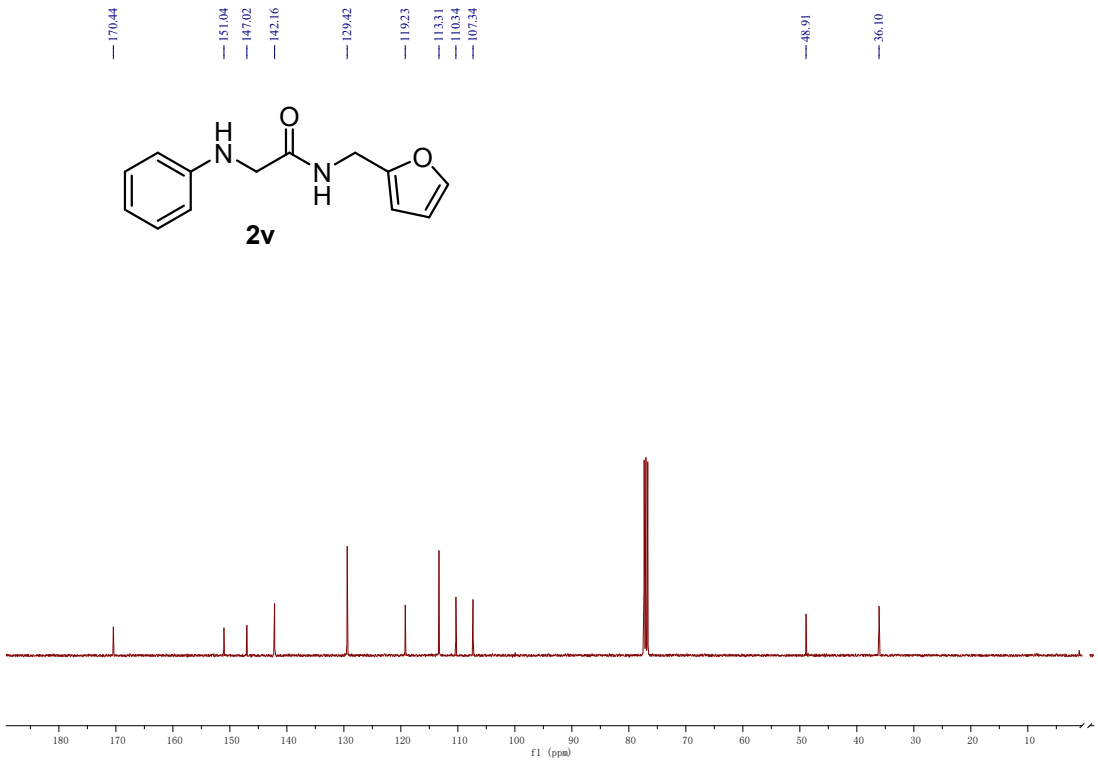
7.305
7.225
7.205
7.186
7.065
6.801
6.604
6.287
6.279
6.275
6.166
6.158
4.468
4.454
3.815
2v
0.90
1.92
0.82
0.91
1.94
0.94
0.91
2.00
1.91
f1 (ppm)


170.44
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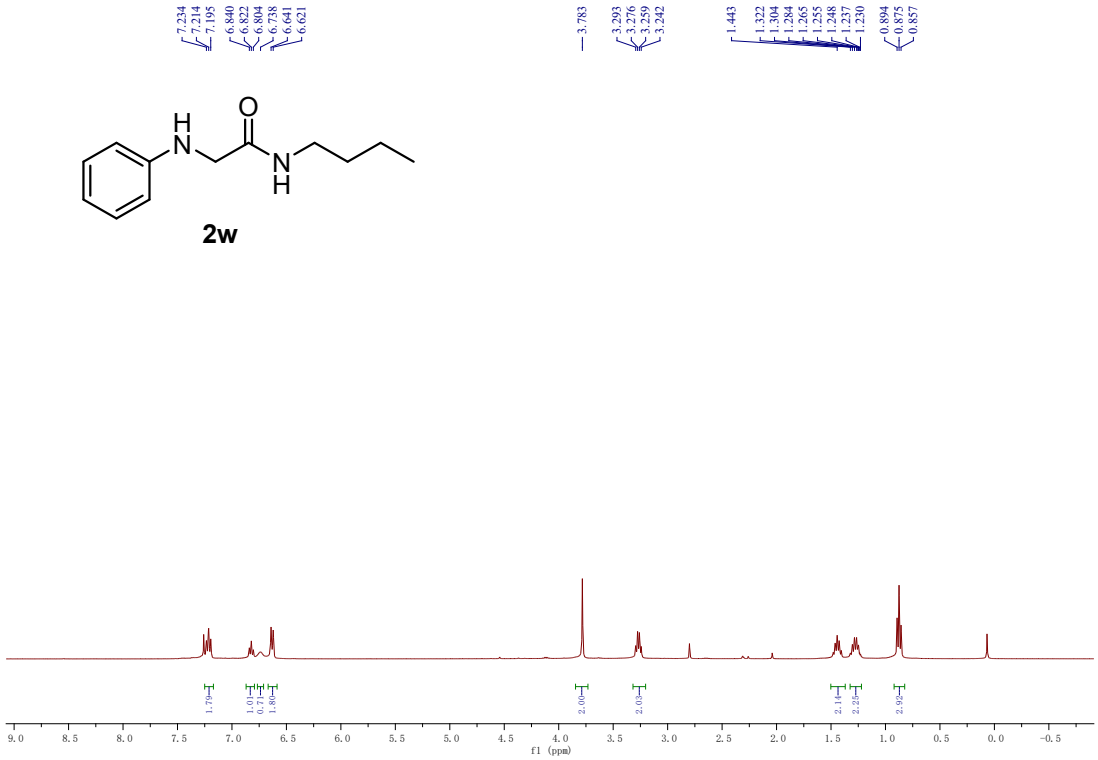
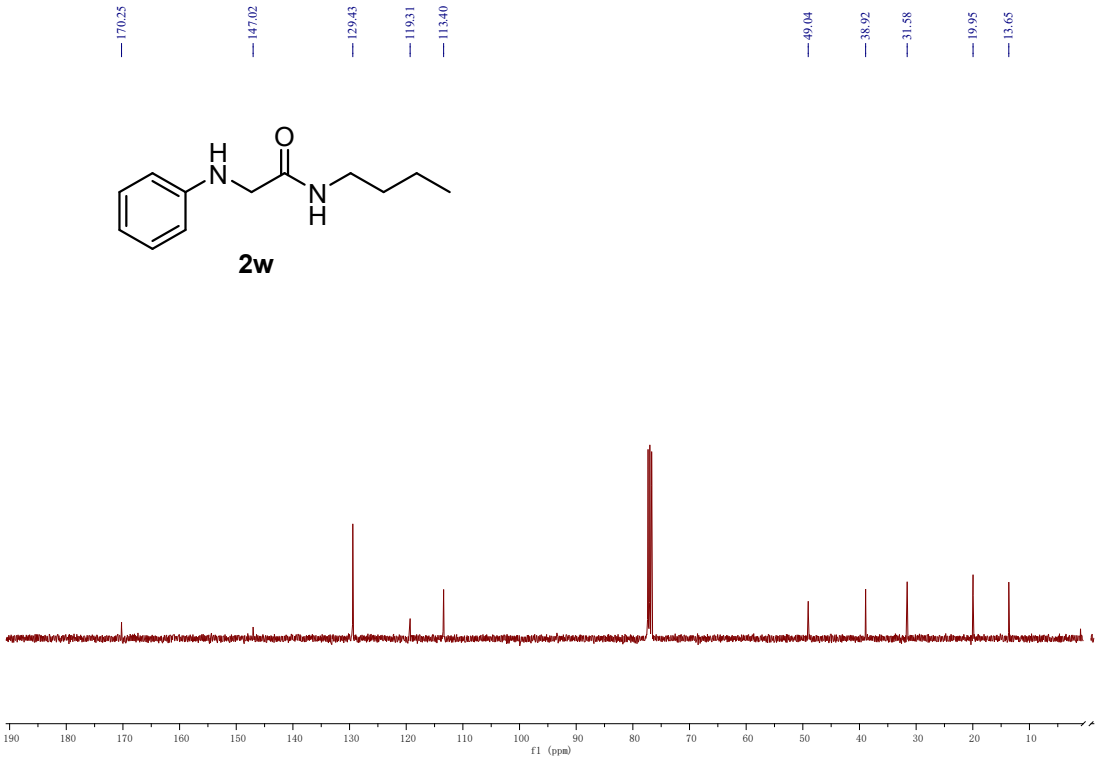
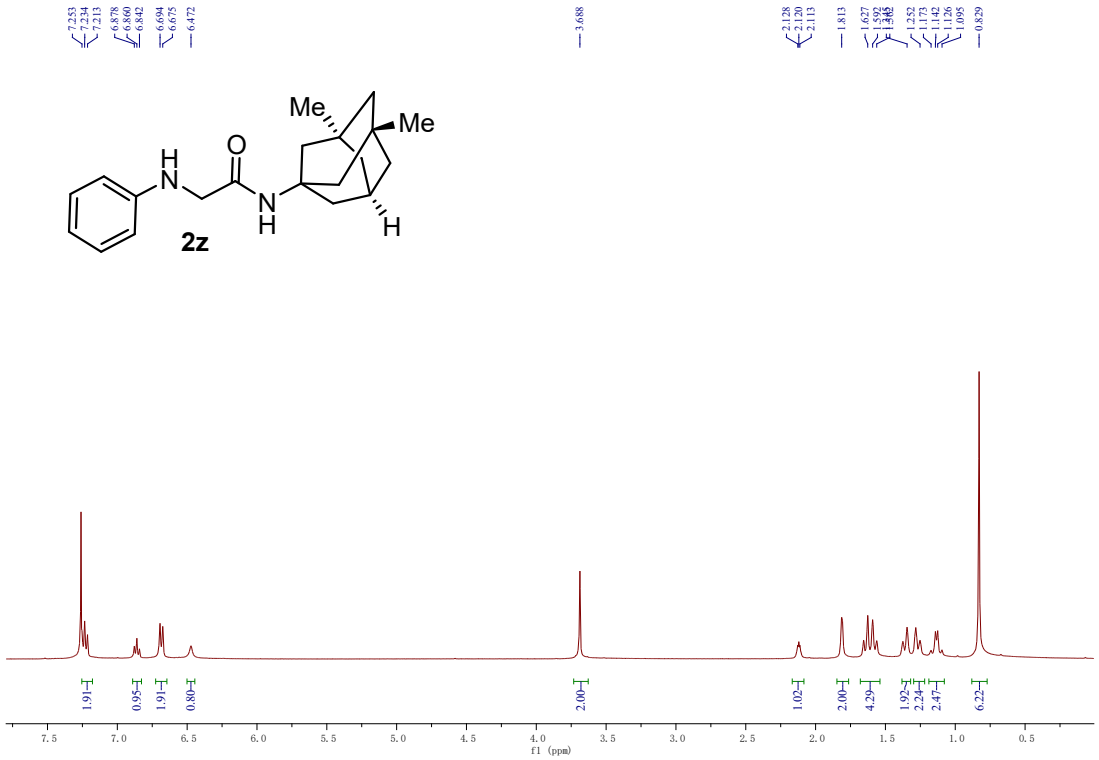
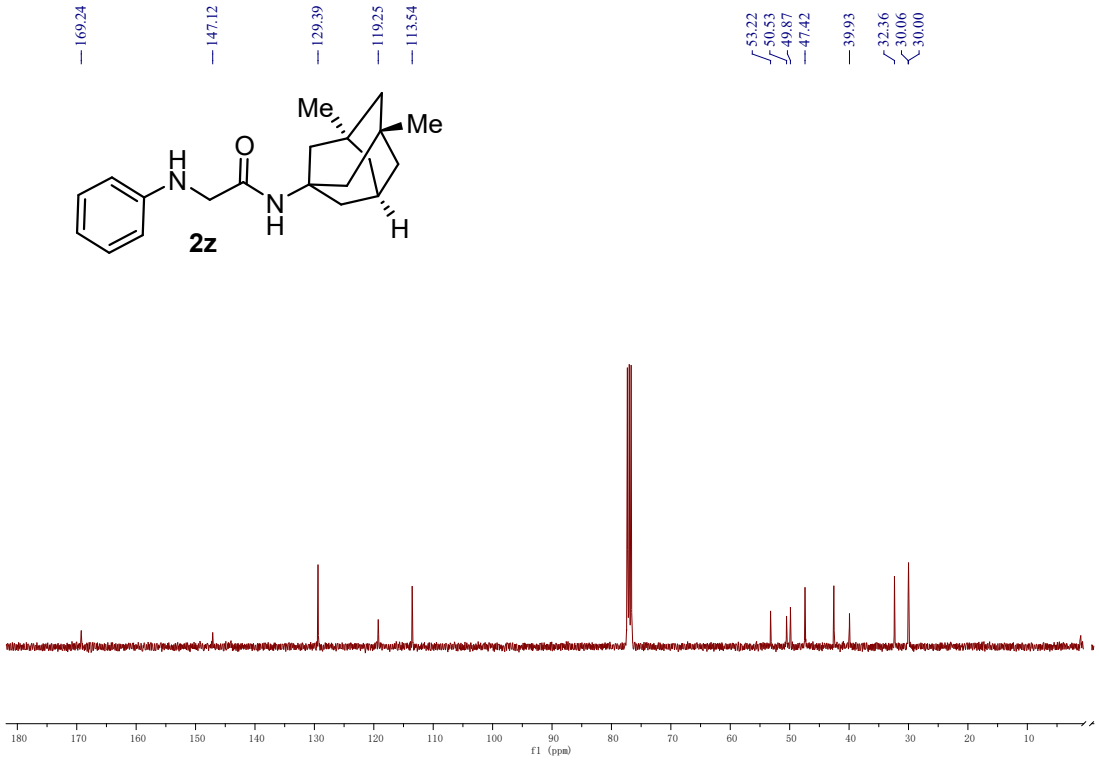
142.16
129.42
119.23
113.31
110.34
107.34
48.91
36.10
2v
f1 (ppm)


7.234
7.214
7.195
6.840
6.822
6.804
6.738
6.641
6.621
3.783
3.293
3.276
3.259
3.242
1.443
1.322
1.304
1.284
1.265
1.255
1.248
1.237
1.230
0.894
0.875
0.857
2w
f1 (ppm)


170.25
147.02
129.43
119.31
113.40
49.04
38.92
31.58
19.95
13.65
2w
f1 (ppm)


7.253
7.234
7.213
6.878
6.860
6.842
6.694
6.675
6.472
3.688
2.128
2.120
2.113
1.813
1.627
1.252
1.173
1.142
1.126
1.095
0.829
Me
Me
H
N
O
N
H
H
2z
1.91
0.95
1.91
0.80
2.00
1.02
2.00
4.29
1.92
2.24
2.47
6.22
f1 (ppm)


169.24
147.12
129.39
119.25
113.54
53.22
50.53
49.87
47.42
39.93
32.36
30.06
30.00
Me
Me
H
N
O
N
H
H
2z
f1 (ppm)