Supporting Information

Photocatalytic hydrogen atom transfer and aryl migration strategy for the arylalkylation of activated alkenes with 1-(*o*-iodoaryl)-alkan-1-ones

Yan-Lin Chu,[†] Liang Zeng,[†] Shuang Wu,*[,†] Jie Yang,[†] Ming Hu,*[,†] and Jin-Heng Li*[,†,‡,§]

[†]State Key Laboratory Base of Eco-Chemical Engineering, College of Chemical Engineering, Qingdao University of Science and Technology, Qingdao 266042, China

[‡]State Key Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 730000, China

[§]School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang, Henan 453007, China

E-mail: shuangwuchem@163.com, huming0731@163.com, jhli@hnu.edu.cn

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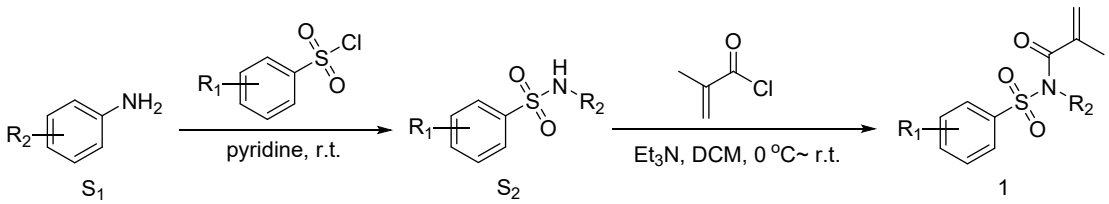
(A) General Experimental Procedures

(a) General Information:

^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on a Bruker 400, 500 MHz advance spectrometer at room temperature in $CDCl_3$ with tetramethylsilane as internal standard. High-resolution mass spectra (HRMS) was recorded on an electrospray ionization (ESI) apparatus using time-of-flight (TOF) mass spectrometry. All products were identified by ^{1}H and ^{13}C NMR, HRMS; High-resolution mass spectra (HRMS) were recorded on an electrospray ionization (ESI) apparatus using time-of-flight (TOF) mass spectrometry; Cyclic voltammograms were obtained on a CHI 605E potentiostat. Unless otherwise noted, all reactions were carried out using standard Schlenk techniques. Column chromatography was performed on silica gel (300-400 mesh) using petroleum ether/ethyl acetate. The light source were used 18W Blue LEDs (manufacturer: liang yuan lighting, model: LY-PD001, wavelength range: 450-460 nm, λ max = 455 nm), with wrap in foil, less than 5cm from the light source to the irradiation vessel. Unless otherwise noted, all reactions were carried out using standard Schlenk techniques, and the starting materials and solvents were commercially available and were used without further purification.

(b) General Procedure for Synthesis of N-phenyl-N-(phenylsulfonyl)methacrylamide (1)

Substrates 1 were prepared by the following procedure (method A)



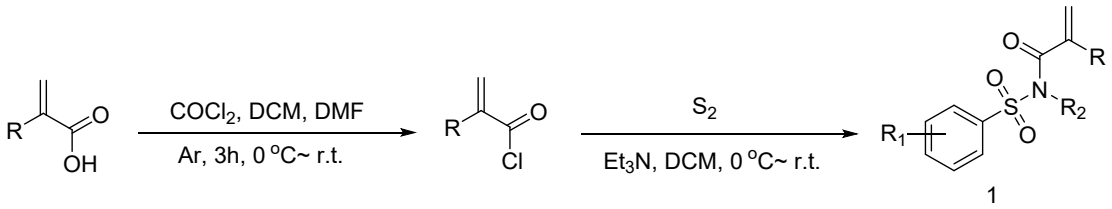
Following the modified procedure by Nevado et al[1].

(i) In a dry 50 mL round-bottom flask, a mixture of aniline S1 (10 mmol) in 20 mL of pyridine were stirred at room temperature. The benzene sulfochloride (11 mmol, 1.1 equiv.) was added slowly. The reaction was monitored by TLC. Then the reaction mixture was poured into water and the aqueous layer was extracted with

CH_2Cl_2 (3×15 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography to provide amide S2.

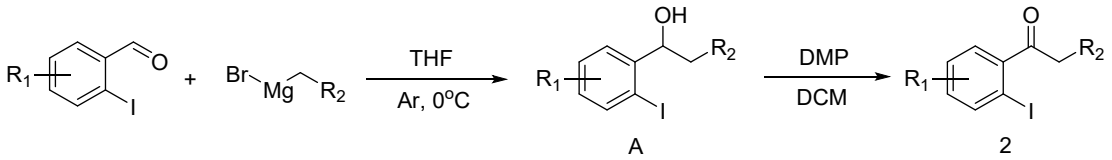
(ii) In a dry 100 mL round-bottom flask, a mixture of N-phenylbenzenesulfonamide S2 (10 mmol) and Et_3N (30 mmol) in 20 mL of DCM were stirred in ice bath. The methacryloyl chloride (12 mmol, 1.2 equiv.) was added slowly by a dropping funnel. The reaction was monitored by TLC. Then the reaction was quenched by H_2O (20 mL), and the aqueous layer was extracted with CH_2Cl_2 (3×20 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography to provide amide product 1.

Substrates 1 were prepared by the following procedure (method B)[2].



To a 50 mL round-bottom flask equipped with a magnetic stir-bar was added solution of acrylic acid (5.0 mmol, 1 equiv.) in dichloromethane (DCM, 20 mL),followed by dropwise addition of oxalyl chloride (10.0 mmol, 2 equiv.) and 2 drops of DMF under argon atmosphere. The mixture was stirred at room temperature for 6 hours before removing all volatiles under reduced pressure. The crude product was used for step (ii) without purification.

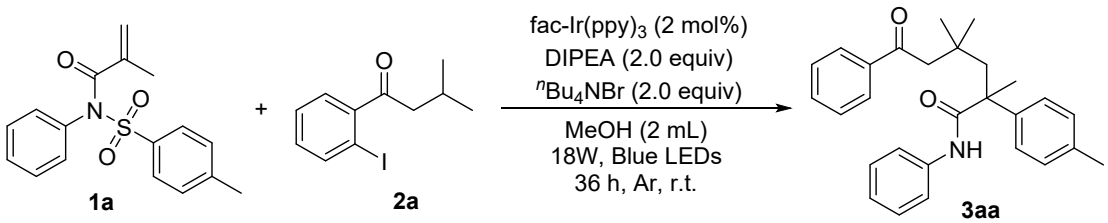
(c) General Procedure for Synthesis of 1-(o-iodoaryl)alkan-1-ones (2) [3-4] :



Following a modified literature procedure[3] , to a flame-dried three-neck flask were added 2-Iodobenzaldehyde (50 mmol) and THF (50 mL), cool to 0 ℃ under a

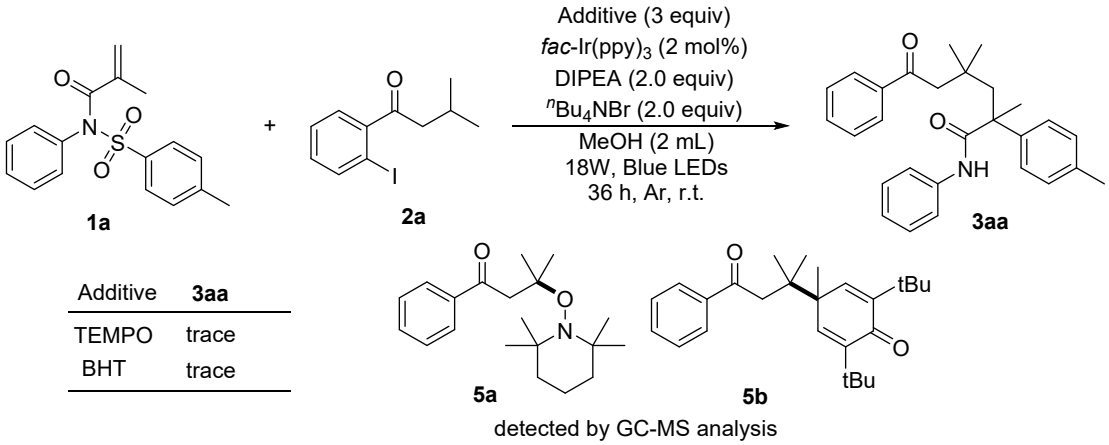
nitrogen atmosphere, then corresponding grignard reagent (1.5 equiv) added in dropwise, the resulting mixture was stirred at this temperature for 1.5 - 3 h, and quenched with a saturated aqueous solution of NH_4Cl. After extraction with ethyl acetate, the organic layer was washed with brine (50 mL) and dried over anhydrous Na_2SO_4, purified by column chromatography on silica gel to give desired secondary benzylic alcohol **A**. To a solution of **A** (1.0 equiv) in DCM (0.1 M) was added DMP (1.5 equiv) under 0 ℃, then the resulted reaction mixture was stirred at room temperature. Upon completion, the reaction mixture was filtered and washed with EtOAc. The resulting mixture was concentrated, and the residue was purified by flash column chromatography on silica gel (eluted with petroleumether/ethyl acetate) to afford 1-(*o*-iodoaryl)alkan-1-ones **2**.

(d) General Procedure for Photoredox Hydrogen Atom Transfer and Aryl Migration Strategy for the Arylalkylation of Activated Alkenes.



To a Schlenk tube were added **1a** (0.20 mmol), **2a** (0.30 mmol, 1.5 equiv), *fac*-$Ir(ppy)_3$ (2 mol %), DIPEA (2 equiv), $^nBu_4NBr$ (2 equiv), MeOH (2 mL, 0.1 M), Then the tube was charged with argon three times, and was stirred irradiated with a 18 W blue LED lamp (at approximately 5.0 cm away from the light source) with cooled by a fan at room temperature for 36 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was concentrated in vacuum, diluted in diethyl ether, and washed with saturated brine. The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum. The resulting residue was purified by silicagel column chromatography (PE/EA = 35:1: to 25:1) to provide **3aa** in 71% isolated yield.

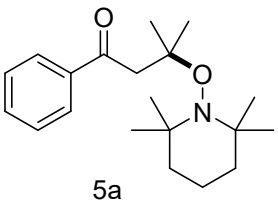
(e) Control Experiments



Additive	3aa
TEMPO	trace
BHT	trace

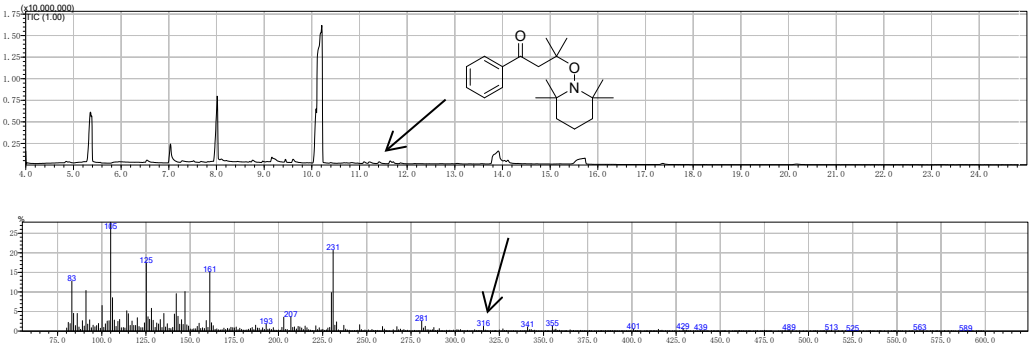
detected by GC-MS analysis

To a Schlenk tube were added **1a** (0.20 mmol), **2a** (0.30 mmol, 1.5 equiv), *fac*-Ir(ppy)$_3$ (2 mol %), DIPEA (2 equiv), ^{n}Bu$_4$NBr (2 equiv), additive TEMPO or BHT (3equiv), MeOH (2 mL, 0.1 M), Then the tube was charged with argon three times, and was stirred irradiated with a 18 W blue LED lamp (at approximately 5.0 cm away from the light source) with cooled by a fan at room temperature for 36 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was concentrated in vacuum, diluted in diethyl ether, and washed with saturated brine. The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum. No desired product **3aa** was observed, and the product **5a** (m/z = 317) and **5b** (m/z = 380.3) were detected by GC-MS analysis of the crude mixture.



5a

Chemical Formula: $C_{20}H_{31}NO_2$
Molecular Weight: 317

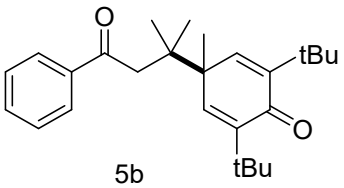


[MS Spectrum]

# of Peaks 487

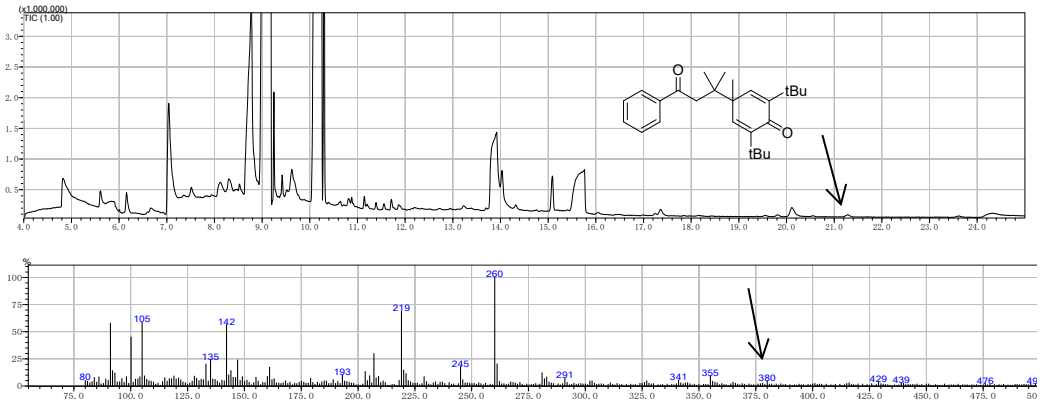
Raw Spectrum 24.840 (scan : 4169)
Background No Background Spectrum
Base Peak m/z 142.15 (Inten : 1,570)
Event# 1
m/z Absolute Intensity Relative Intensity

299.00	86	5.48	309.00	24	1.53	320.00	3	0.19
300.00	44	2.80	310.00	14	0.89	321.00	18	1.15
301.00	46	2.93	311.00	68	4.33	322.00	2	0.13
302.00	36	2.29	312.00	30	1.91	323.00	58	3.69
303.00	127	8.09	313.00	24	1.53	324.00	8	0.51
304.00	22	1.40	314.00	38	2.42	325.00	100	6.37
305.00	55	3.50	315.00	94	5.99	326.00	18	1.15
306.00	3	0.19	**317.00**	**44**	**2.80**	327.00	100	6.37
307.00	63	4.01	318.00	19	1.21	328.00	42	2.68
308.00	10	0.64	319.00	46	2.93	329.00	113	7.20



5b

Chemical Formula: $C_{26}H_{36}O_2$
Molecular Weight: 380



[MS Spectrum]
# of Peaks 518
Raw Spectrum 24.625 (scan : 4126)
Background No Background Spectrum
Base Peak m/z 91.05 (Inten : 14,382)
Event# 1
m/z Absolute Intensity Relative Intensity

369.10	97	0.67	379.10	54	0.38	389.10	86	0.60
370.10	68	0.47	**380.10**	**44**	**0.31**	390.10	19	0.13
371.10	94	0.65	381.10	78	0.54	391.10	49	0.34
372.10	26	0.18	382.10	52	0.36	392.10	38	0.26
373.10	103	0.72	383.10	79	0.55	393.10	49	0.34

374.10	30	0.21	384.10	60	0.42	394.10	63	0.44
375.10	46	0.32	385.10	89	0.62	395.10	36	0.25
376.10	33	0.23	386.10	76	0.53	396.10	10	0.07
377.10	138	0.96	387.10	111	0.77	397.10	49	0.34
378.10	94	0.65	388.10	52	0.36	398.10	18	0.13

(f) Light on-off Experiments

Three parallel reactions were performed between **1a** (0.20 mmol), **2a** (0.30 mmol) according to the General Procedure. The resulting residue was purfied by silica gel column chromatography (PE/EA = 35:1 to 25:1) to afford the **3aa**. The white area indicates the light irradiation, while the grey area indicates time in the dark. The results demonstrate that the visible light is crucial.

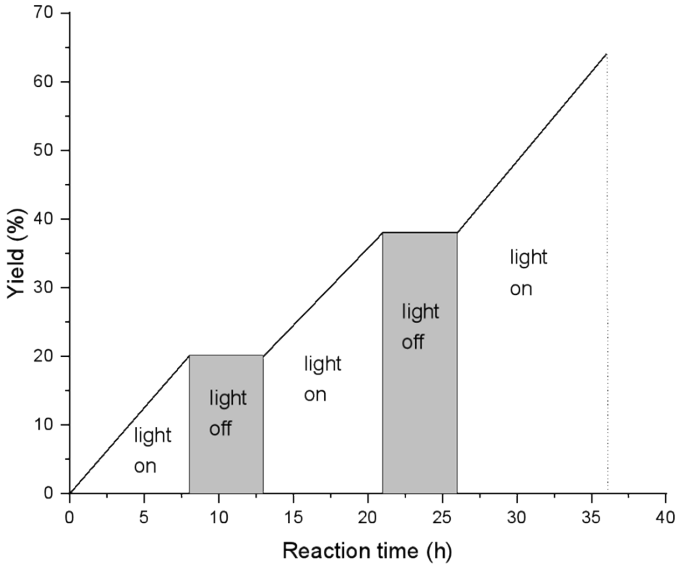


Figure S1: Light on-off experiments plot

(g) Stern-Volmer Fluorescence Quenching Experiments

Fluorescence spectra samples for the quenching experiments were prepared in a glass cuvette with a septum screw cap. $Ir(ppy)_3$ was irradiated at 500 nm and the emission intensity at 515 nm was observed. In a typical experiment, the emission spectrum of a 1.0×10^{-5} M solution of $Ir(ppy)_3$ in MeOH was collected. In Figure S2, a stock solution of DIPEA (12.9 mg, 0.1 mmol) in 1 mL of MeOH was prepared. Then, different amounts of this stock solution were added to a solution of the photocatalyst in MeOH (1.0×10^{-5} M). The obviously sharp decrease of $Ir(ppy)_3$

luminescence was observed, suggesting that the mechanism might operate via a canonical photoredox cycle consisting of a reductive quenching with DIPEA.

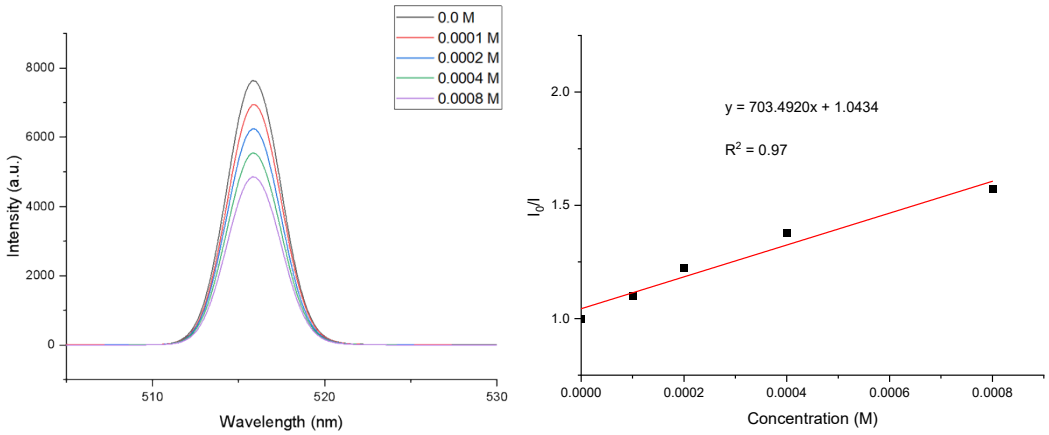


Figure S2. Stern-Volmer fluorescence quenching experiments of $Ir(ppy)_3$ with DIPEA.

In Figure S3, a stock solution of Bu_4NBr (32.2 mg, 0.1 mmol) in 1 mL of MeOH was prepared. Then, different amounts of this stock solution were added to a solution of the photocatalyst in MeOH (1.0×10^{-5} M). The results show that slight decrease of $Ir(ppy)_3$ luminescence was observed.

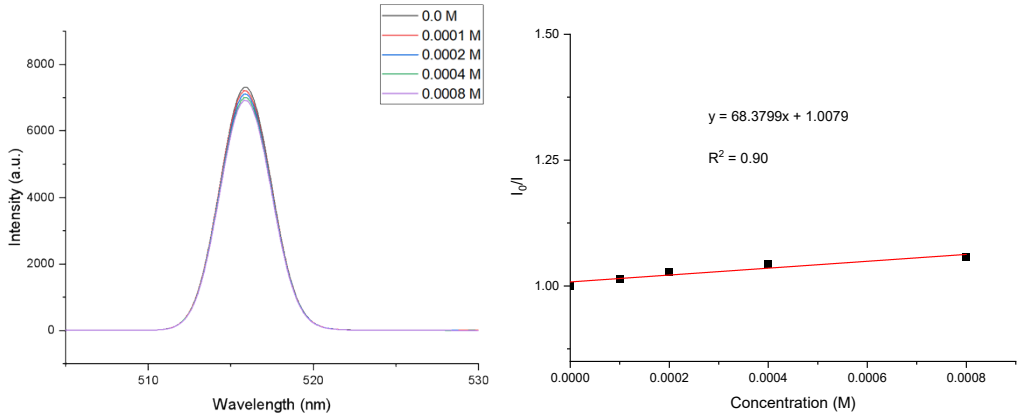


Figure S3. Stern-Volmer fluorescence quenching experiments of $Ir(ppy)_3$ with Bu_4NBr.

In Figure S4, a stock solution of *N*-phenyl-*N*-(phenylsulfonyl)methacrylamide (**1a**) (31.5 mg, 0.1 mmol) in 1 mL of MeOH was prepared. Then, different amounts of this stock solution were added to a solution of the photocatalyst in MeOH (1.0×10^{-5} M). The results show that the luminescence of $Ir(ppy)_3$ is slightly diminished.

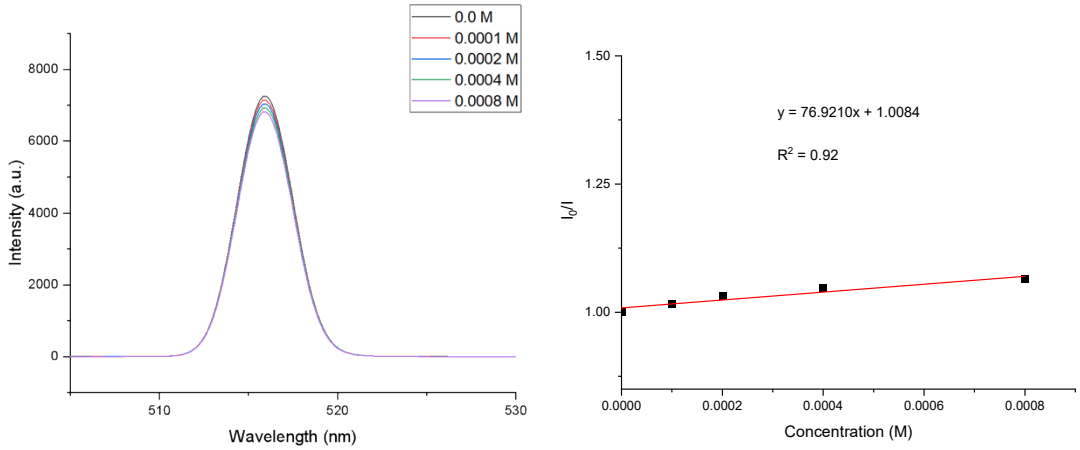


Figure S4. Stern-Volmer fluorescence quenching experiments of $Ir(ppy)_3$ with **1a**.

In Figure S5, a stock solution of 1-(2-iodophenyl)-3-methylbutan-1-one **2a** (28.8 mg, 0.1 mmol) in 1 mL of MeOH was prepared. Then, different amounts of this stock solution were added to a solution of the photocatalyst in MeOH (1.0×10^{-5} M). The results show that slight decrease of $Ir(ppy)_3$ luminescence was observed.

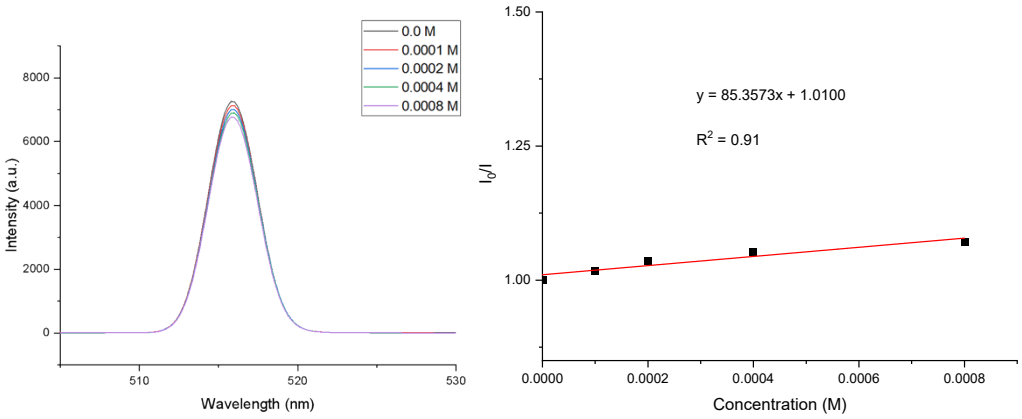


Figure S5. Stern-Volmer fluorescence quenching experiments of $Ir(ppy)_3$ with **2a**.

Fluorescence spectra samples for the quenching experiments were prepared in a glass cuvette with a septum screw cap. $[Ir(dtbbpy)(ppy)_2]PF_6$ was irradiated at 540 nm and the emission intensity at 550 nm was observed. In a typical experiment, the emission spectrum of a 1.0×10^{-5} M solution of $[Ir(dtbbpy)(ppy)_2]PF_6$ in DCM was collected. In Figure S6, a stock solution of *N*-methyl-*N*-tosylmethacrylamide (**1i**) (25.3 mg, 0.1 mmol) in 1 mL of DCM was prepared. Then, different amounts of this stock solution were added to a solution of the photocatalyst in DCM (1.0×10^{-5} M). The results show that the luminescence of $[Ir(dtbbpy)(ppy)_2]PF_6$ is slightly diminished.

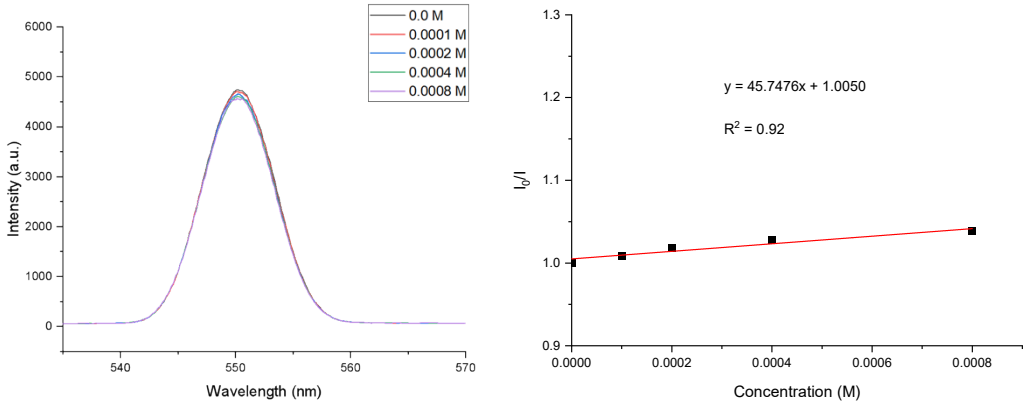


Figure S6: Stern-Volmer fluorescence quenching experiments of $[Ir(dtbbpy)(ppy)_2]PF_6$ with **1i**.

In Figure S7, a stock solution of 1-(2-iodophenyl)-3-methylbutan-1-one **2a** (28.8 mg, 0.1 mmol) in 1 mL of DCM was prepared. Then, different amounts of this stock solution were added to a solution of the photocatalyst in DCM (1.0×10^{-5} M). The results show that slight decrease of $[Ir(dtbbpy)(ppy)_2]PF_6$ luminescence was observed.

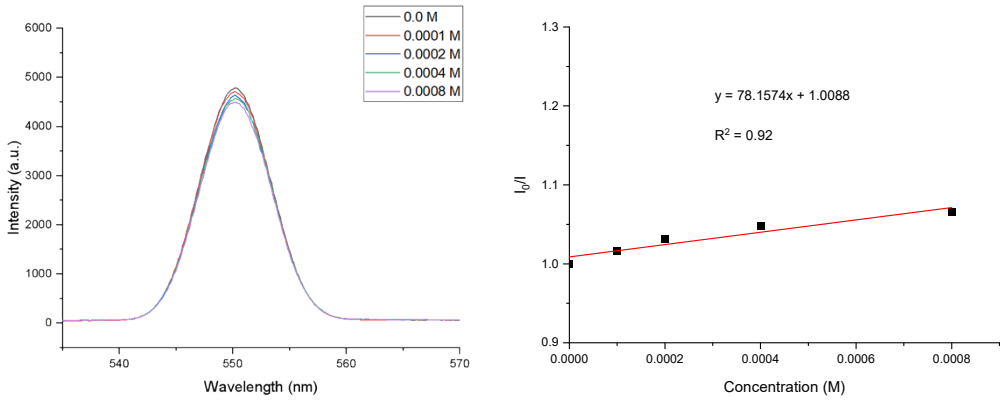


Figure S7: Stern-Volmer fluorescence quenching experiments of $[Ir(dtbbpy)(ppy)_2]PF_6$ with **2a**.

In Figure S8, a stock solution of K_3PO_4 (21.2 mg, 0.1 mmol) in 1 mL of DCM was prepared (Using ultrasound to help dissolve, and the supernatant was used as a stock solution for fluorescence quenching experiments.). Then, different amounts of this stock solution were added to a solution of the photocatalyst in DCM (1.0×10^{-5} M). The results show that slight decrease of $[Ir(dtbbpy)(ppy)_2]PF_6$ luminescence was observed.

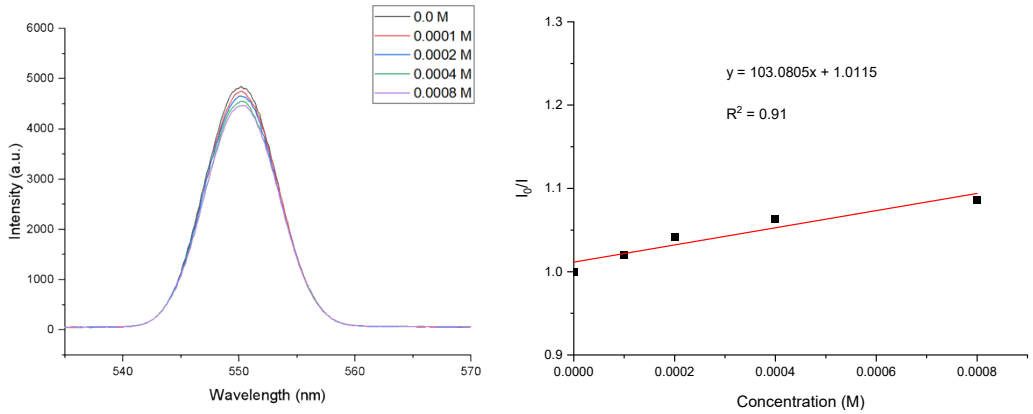
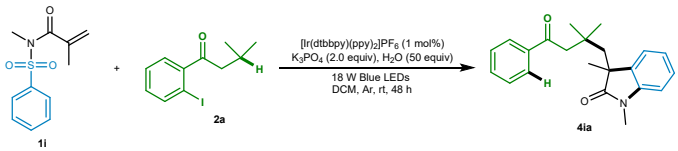


Figure S8: Stern-Volmer fluorescence quenching experiments of $[Ir(dtbbpy)(ppy)_2]PF_6$ with K_3PO_4.

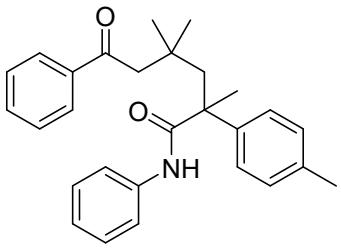
(h) Optimization of Reaction Conditions



Entry	Variation from the standard conditions	Yield (%)[b]
1	none	67
2	*fac*-Ir(ppy)$_3$ instead of $[Ir(dtbbpy)(ppy)_2]PF_6$	50
3	4-DPAIPN instead of $[Ir(dtbbpy)(ppy)_2]PF_6$	52
4	4-CZIPN instead of $[Ir(dtbbpy)(ppy)_2]PF_6$	31
5	without $[Ir(dtbbpy)(ppy)_2]PF_6$	trace
6	without K_3PO_4	12
7	without H_2O	60
8	Na_2CO_3 instead of K_3PO_4	31
9	$NaHCO_3$ instead of K_3PO_4r	29
10	Na_2HPO_4 instead of K_3PO_4	46
11	DIPEA instead of K_3PO_4	trace
12	MeCN instead of DCM	40
13	DMSO instead of DCM	45
14	MeOH instead of DCM	trace
15	DMF instead of DCM	39
16	36 W instead of 18 W	61
17	40 °C instead of r.t.	54
18	24 h instead of 48 h	38
19[c]	without light (in dark)	0

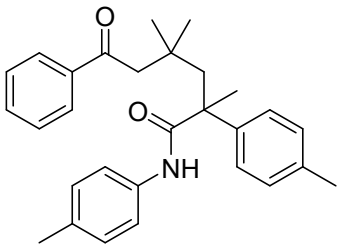
[a]Standard reaction conditions: **1i** (0.2 mmol), **2a** (0.3 mmol), $Ir(ppy)_2(dtbbpy)PF_6$ (1 mol%), K_3PO_4 (2 equiv), H_2O (50 equiv), DCM (2 mL), 18 W blue LEDs, argon, room temperature, and 48 h.

(B) Analytical data



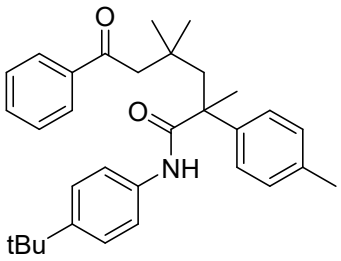
2,4,4-trimethyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide (3aa)

56.8 mg, 71% yield; Rf = 0.4 (PE/EA = 10 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.80 (m, 2H), 7.51 (t, *J* = 7.2 Hz, 1H), 7.46 (m, 2H), 7.41 - 7.33 (m, 5H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.15 - 7.11 (m, 2H), 7.03 (t, *J* = 7.6 Hz, 1H), 2.83 (d, *J* = 4.0 Hz, 2H), 2.68 (d, *J* = 15.2 Hz, 1H), 2.36 (d, *J* = 15.2 Hz, 1H), 2.30 (s, 3H), 1.76 (s, 3H), 1.13 (d, *J* = 15.2 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.1, 175.4, 142.4, 138.1, 138.1, 136.8, 132.7, 129.5, 128.8, 128.4, 127.9, 126.6, 123.9, 119.6, 51.3, 49.7, 47.7, 34.6, 30.7, 28.6, 26.0, 20.9; HRMS *m/z* (ESI) calcd for $C_{28}H_{31}NO_2$ ([M+Na]$^+$) 436.2247, found 4436.2246.



2,4,4-trimethyl-6-oxo-6-phenyl-N,2-di-p-tolylhexanamide (3ba)

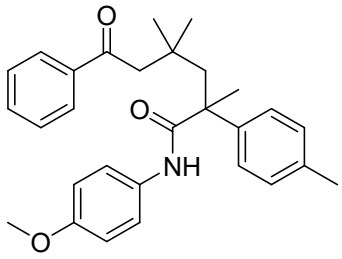
60.6 mg, 71% yield; Rf = 0.5 (PE/EA = 10 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.79 (m, 2H), 7.53 - 7.48 (m, 1H), 7.40 - 7.36 (m, 2H), 7.35 - 7.32 (m, 4H), 7.29 (s, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 2.83 (d, *J* = 3.6 Hz, 2H), 2.66 (d, *J* = 15.2 Hz, 1H), 2.35 (d, *J* = 15.2 Hz, 1H), 2.30 (s, 3H), 2.26 (s, 3H), 1.75 (s, 3H), 1.12 (d, *J* = 15.6 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.1, 175.3, 142.6, 138.1, 136.7, 135.6, 133.4, 132.7, 129.4, 129.3, 128.3, 127.9, 126.6, 119.7, 51.3, 49.6, 47.8, 34.7, 30.6, 28.7, 26.0, 20.9, 20.8; HRMS *m/z* (ESI) calcd for $C_{29}H_{33}NO_2$ ([M+Na]$^+$) 450.2404, found 450.2404.



N-(4-(tert-butyl)phenyl)-2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide (3ca)

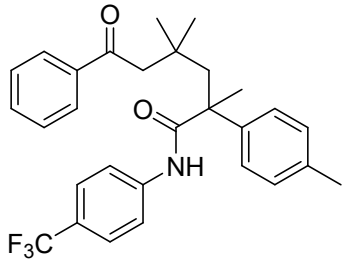
63.8 mg, 68% yield; Rf = 0.6 (PE/EA = 10 : 1); White solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.79 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.40 - 7.32 (m, 7H), 7.26 - 7.23 (m, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 2.90 - 2.78 (m, 2H), 2.69 (d, *J* = 14.8 Hz,

1H), 2.34 (d, *J* = 15.2 Hz, 1H), 2.29 (s, 3H), 1.75 (s, 3H), 1.26 (s, 9H), 1.14 (d, *J* = 14.4 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.1, 175.3, 146.7, 142.7, 138.1, 136.7, 135.5, 132.7, 129.4, 128.3, 127.9, 126.5, 125.6, 119.3, 51.2, 49.6, 47.7, 34.6, 34.2, 31.3, 31.3, 30.7, 28.6, 26.0, 20.9; HRMS *m/z* (ESI) calcd for $C_{32}H_{39}NO_2$ ([M+Na]$^+$) 492.2873, found 492.2868.



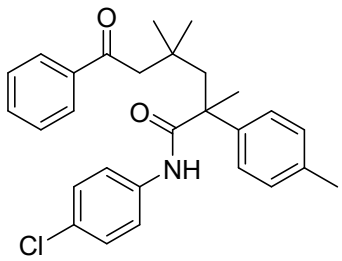
N-(4-methoxyphenyl)-2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide (3da)

46.1 mg, 52% yield; Rf = 0.3 (PE/EA = 5 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.79 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.40 - 7.30 (m, 7H), 7.13 (d, *J* = 8.0 Hz, 2H), 6.80 - 6.75 (m, 2H), 3.75 (s, 3H), 2.88 - 2.78 (m, 2H), 2.67 (d, *J* = 15.2 Hz, 1H), 2.34 (d, *J* = 15.2 Hz, 1H), 2.30 (s, 3H), 1.75 (s, 3H), 1.13 (d, *J* = 15.2 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.1, 175.2, 156.1, 142.6, 138.1, 136.7, 132.7, 131.3, 129.4, 128.3, 127.9, 126.6, 121.4, 113.9, 55.4, 51.1, 49.7, 47.8, 34.6, 30.6, 28.6, 26.0, 20.9; HRMS *m/z* (ESI) calcd for $C_{29}H_{33}NO_3$ ([M+Na]$^+$) 466.2353, found 466.2347.



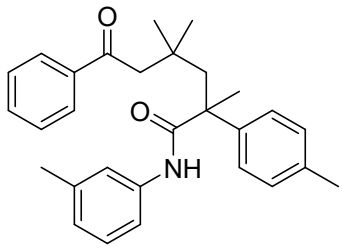
2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)-N-(4-(trifluoromethyl)phenyl)hexanamide (3ea)

54.8 mg, 57% yield; Rf = 0.5 (PE/EA = 10 : 1); Yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.80 (m, 3H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.48 (m, 3H), 7.41 - 7.36 (m, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.16 - 7.11 (m, 2H), 2.97 - 2.77 (m, 3H), 2.29 (d, *J* = 14.0 Hz, 4H), 1.75 (s, 3H), 1.15 (d, *J* = 1.6 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.2, 175.8, 142.5, 141.2, 137.8, 136.9, 133.0, 129.5, 128.4, 127.8, 126.3, 126.0 (q, *J* = 4.0 Hz), 125.6, 125.3, 119.2, 51.4, 49.7, 46.8, 34.4, 31.5, 27.9, 26.2, 20.9; ^{19}F NMR (376 MHz, Chloroform-*d*) δ (ppm) -61.96; HRMS *m/z* (ESI) calcd for $C_{29}H_{30}F_3NO_2$ ([M+Na]$^+$) 504.5490, found 504.5494.



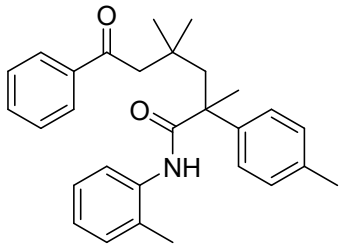
N-(4-chlorophenyl)-2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide (3fa)

59.0 mg, 66% yield; Rf = 0.5 (PE/EA = 10 : 1); White solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.80 (m, 2H), 7.55 - 7.50 (m, 2H), 7.45 - 7.43 (m, 2H), 7.41 - 7.37 (m, 2H), 7.34 - 7.31 (m, 2H), 7.19 - 7.17 (m, 2H), 7.15 - 7.12 (m, 2H), 2.85 (d, *J* = 9.6 Hz, 2H), 2.74 (d, *J* = 15.2 Hz, 1H), 2.29 (d, *J* = 6.8 Hz, 4H), 1.74 (s, 3H), 1.14 (d, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.0, 175.5, 142.5, 137.9, 136.9, 136.8, 132.9, 129.5, 128.7, 128.4, 127.9, 126.4, 120.9, 51.3, 49.7, 47.2, 34.5, 31.1, 28.1, 26.1, 20.9; HRMS *m/z* (ESI) calcd for $C_{28}H_{30}ClNO_2$ ([M+Na]$^+$) 470.1863, found 470.1856.



2,4,4-trimethyl-6-oxo-6-phenyl-N-(m-tolyl)-2-(p-tolyl)hexanamide (3ga)

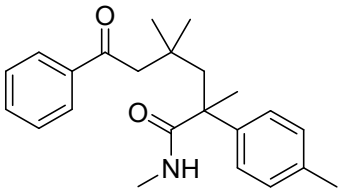
50.4 mg, 59% yield; Rf = 0.5 (PE/EA = 10 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.79 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.41 - 7.37 (m, 2H), 7.37 - 7.27 (m, 5H), 7.14 - 7.09 (m, 3H), 6.85 - 6.82 (m, 1H), 2.88 - 2.78 (m, 2H), 2.68 (d, *J* = 15.2 Hz, 1H), 2.36 (d, *J* = 14.8 Hz, 1H), 2.30 (s, 3H), 2.27 (s, 3H), 1.75 (s, 3H), 1.13 (d, *J* = 16.0 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.0, 175.4, 142.5, 138.7, 138.1, 138.0, 136.8, 132.7, 129.4, 128.6, 128.3, 127.9, 126.9, 124.7, 120.2, 116.6, 51.3, 49.5, 47.8, 34.7, 30.6, 28.7, 26.0, 21.4, 20.9; HRMS *m/z* (ESI) calcd for $C_{29}H_{33}NO_2$ ([M+Na]$^+$) 450.2404, found 450.2401.



2,4,4-trimethyl-6-oxo-6-phenyl-N-(o-tolyl)-2-(p-tolyl)hexanamide (3ha)

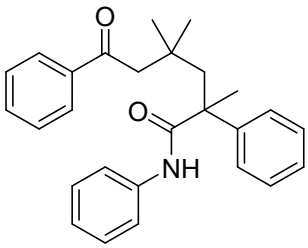
57.2 mg, 67% yield; Rf = 0.5 (PE/EA = 10 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.87 (d, *J* = 8.4 Hz, 1H), 7.78 - 7.69 (m, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.42 - 7.34 (m, 4H), 7.18 - 7.10 (m, 3H), 7.03 (d, *J* = 7.2 Hz, 1H), 6.99 - 6.82 (m, 2H), 2.82 - 2.71 (m, 2H), 2.60 (d, *J* = 14.8 Hz, 1H), 2.50 (d, *J* = 14.8 Hz, 1H), 2.30 (s, 3H), 1.81 (d, *J* = 6.8 Hz, 6H), 1.10 (d, *J* = 34.8 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.7, 175.7, 141.6, 138.2, 137.0, 136.0, 132.5, 130.2, 129.5, 128.3, 127.8,127.7, 127.1, 126.6, 124.3, 121.5, 51.6, 49.5, 48.0, 34.8, 29.9, 29.6, 25.3,

20.9, 17.0; HRMS *m/z* (ESI) calcd for $C_{29}H_{33}NO_2$ ([M+Na]$^+$) 450.2404, found 450.2399.



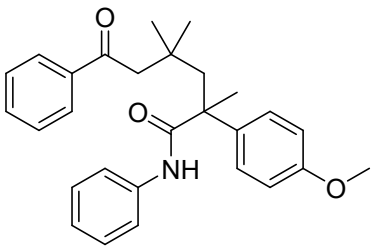
N,2,4,4-tetramethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide (3ia)

47.1 mg, 67% yield; Rf = 0.3 (PE/EA = 5 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.80 (m, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.43 - 7.39 (m, 2H), 7.28 (d, *J* = 2.0 Hz, 1H), 7.25 (s, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 5.50 (s, 1H), 2.82 - 2.70 (m, 2H), 2.67 (d, *J* = 4.8 Hz, 3H), 2.51 (d, *J* = 14.8 Hz, 1H), 2.32 - 2.27 (m, 4H), 1.68 (s, 3H), 1.07 (d, *J* = 21.6 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.1, 178.0, 142.6, 138.3, 136.5, 132.7, 129.2, 128.4, 127.9, 126.8, 50.4, 49.5, 48.4, 34.7, 30.0, 29.3, 26.6, 25.5, 20.8; HRMS *m/z* (ESI) calcd for $C_{23}H_{29}NO_2$ ([M+Na]$^+$) 374.2091, found 374.2098.



2,4,4-trimethyl-6-oxo-N,2,6-triphenylhexanamide (3ja)

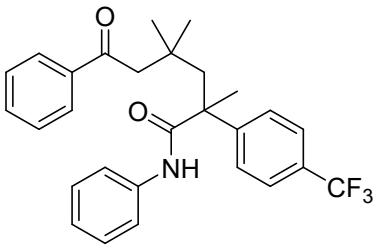
47.9 mg, 60% yield; Rf = 0.5 (PE/EA = 10 : 1); White solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.82 (m, 2H), 7.54 - 7.46 (m, 6H), 7.41 - 7.32 (m, 4H), 7.26 (s, 1H), 7.25 - 7.22 (m, 2H), 7.05 - 7.01 (m, 1H), 2.88 (d, *J* = 4.0 Hz, 2H), 2.73 (d, *J* = 15.2 Hz, 1H), 2.35 (d, *J* = 14.8 Hz, 1H), 1.78 (s, 3H), 1.14 (d, *J* = 12.4 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.2, 175.1, 145.7, 138.1, 138.1, 132.8, 128.8, 128.8, 128.4, 127.9, 127.1, 126.6, 123.9, 119.7, 51.6, 49.9, 47.7, 34.6, 30.7, 28.3, 26.0; HRMS *m/z* (ESI) calcd for $C_{27}H_{29}NO_2$ ([M+Na]$^+$) 422.2091, found 422.2088.



2-(4-methoxyphenyl)-2,4,4-trimethyl-6-oxo-N,6-diphenylhexanamide (3ka)

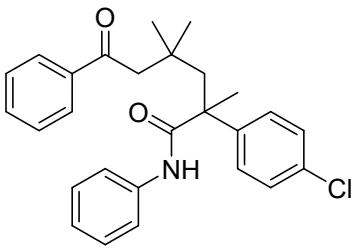
53.2 mg, 62% yield; Rf = 0.5 (PE/EA = 10 : 1); Yellow solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.83 - 7.79 (m, 2H), 7.51 (s, 1H), 7.45 (d, *J* = 7.6 Hz, 2H), 7.41 - 7.36 (m, 5H), 7.26 - 7.22 (m, 2H), 7.03 (t, *J* = 7.6 Hz, 1H), 6.88 - 6.83 (m, 2H), 3.76 (s, 3H), 2.84 (d, *J* = 4.0 Hz, 2H), 2.67 (d, *J* = 15.2 Hz, 1H), 2.35 (d, *J* = 15.2 Hz, 1H), 1.76 (s, 3H), 1.12 (d, *J* = 17.2 Hz, 6H); ^{13}C NMR (100 MHz,

Chloroform-*d*) δ (ppm) 200.1, 175.6, 158.5, 138.1, 137.3, 132.8, 128.8, 128.4, 127.9, 127.9, 123.9, 119.6, 114.0, 55.2, 51.0, 49.7, 47.7, 34.6, 30.6, 28.7, 26.0; HRMS *m/z* (ESI) calcd for $C_{28}H_{31}NO_3$ ([M+Na]$^+$) 452.2196, found 452.2190.



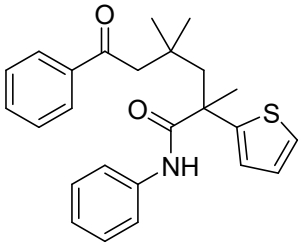
2,4,4-trimethyl-6-oxo-N,6-diphenyl-2-(4-(trifluoromethyl)phenyl)hexanamide (3la)

51.4 mg, 55% yield; Rf = 0.5 (PE/EA = 10 : 1); Yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.90 - 7.80 (m, 3H), 7.62 - 7.51 (m, 7H), 7.41 (t, *J* = 8.0 Hz, 2H), 7.31 - 7.26 (m, 2H), 7.06 (t, *J* = 7.6 Hz, 1H), 3.02 - 2.89 (m, 2H), 2.81 (d, *J* = 15.2 Hz, 1H), 2.30 (d, *J* = 15.2 Hz, 1H), 1.78 (s, 3H), 1.18 (d, *J* = 2.8 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.3, 174.0, 150.3, 150.3, 137.9, 137.8, 133.1, 129.3, 128.9, 128.5, 127.9, 126.7, 125.5 (q, *J* = 3.8 Hz), 124.2, 119.9, 51.6, 50.1, 47.0, 34.4, 31.4, 27.6, 26.1; ^{19}F NMR (376 MHz, Chloroform-*d*) δ (ppm) -62.43; HRMS *m/z* (ESI) calcd for $C_{28}H_{28}F_3NO_2$ ([M+Na]$^+$) 490.5220, found 490.5218.



2-(4-chlorophenyl)-2,4,4-trimethyl-6-oxo-N,6-diphenylhexanamide (3ma)

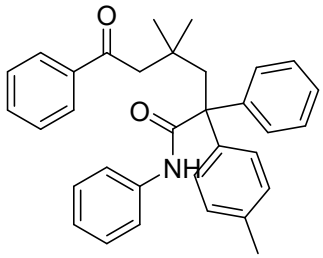
48.5 mg, 56% yield; Rf = 0.5 (PE/EA = 10 : 1); White solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.84 (m, 2H), 7.62 (s, 1H), 7.55 - 7.50 (m, 3H), 7.41 (m, 4H), 7.31 - 7.28 (m, 2H), 7.27 - 7.24 (m, 2H), 7.05 (t, *J* = 7.2 Hz, 1H), 2.92 (d, *J* = 4.4 Hz, 2H), 2.74 (d, *J* = 14.8 Hz, 1H), 2.29 (d, *J* = 14.8 Hz, 1H), 1.75 (s, 3H), 1.15 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.2, 174.5, 144.5, 138.0, 137.9, 133.0, 132.9, 128.9, 128.8, 128.5, 127.9, 127.9, 124.1, 119.8, 51.2, 50.0, 47.2, 34.5, 31.0, 28.0, 26.0; HRMS *m/z* (ESI) calcd for $C_{27}H_{28}ClNO_2$ ([M+Na]$^+$) 456.1701, found 456.1704.



2,4,4-trimethyl-6-oxo-N,6-diphenyl-2-(thiophen-2-yl)hexanamide (3na)

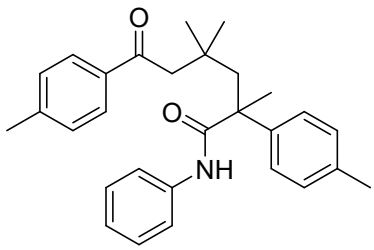
55.1 mg, 68% yield; Rf = 0.5 (PE/EA = 10 : 1); Orange oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.94 - 7.85 (m,

3H), 7.54 - 7.50 (m, 3H), 7.43 - 7.38 (m, 2H), 7.27 (d, *J* = 7.6 Hz, 1H), 7.23 (m, 1H), 7.10 (m, 1H), 7.07 - 7.02 (m, 1H), 6.97 (m, 1H), 2.96 (d, *J* = 0.8 Hz, 2H), 2.87 (d, *J* = 14.8 Hz, 1H), 2.29 (d, *J* = 14.8 Hz, 1H), 1.86 (s, 3H), 1.16 (s, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.2, 173.5, 151.1, 138.0, 138.0, 132.9, 128.8, 128.4, 127.9, 127.0, 124.5, 124.4, 124.1, 119.8, 50.1, 49.7, 49.3, 34.7, 30.4, 27.8, 26.9; HRMS *m/z* (ESI) calcd for $C_{25}H_{27}NO_2S$ ([M+Na]$^+$) 428.1655, found 428.1660.



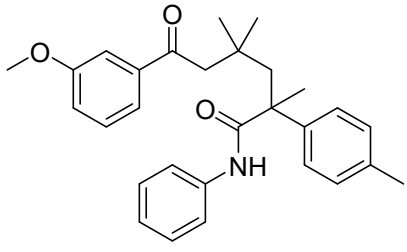
4,4-dimethyl-6-oxo-N,2,6-triphenyl-2-(p-tolyl)hexanamide (3oa)

39.0 mg, 41% yield; Rf = 0.5 (PE/EA = 10 : 1); White solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 8.13 (s, 1H), 7.83 (m, 2H), 7.56 - 7.51 (m, 5H), 7.43 - 7.39 (m, 4H), 7.27 (d, *J* = 2 Hz, 1H), 7.25 (d, *J* = 8.4 Hz, 3H), 7.18 (t, *J* = 7.2 Hz, 1H), 7.06 - 7.01 (m, 3H), 3.06 - 2.94 (m, 2H), 2.76 (s, 2H), 2.26 (s, 3H), 0.89 (d, *J* = 14.0 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.6, 172.3, 145.2, 141.9, 138.3, 137.9, 136.4, 132.9, 129.2, 129.0, 128.8, 128.8, 128.4, 128.0, 127.9, 126.6, 123.9, 120.0, 58.9, 50.1, 46.7, 34.1, 29.0, 28.5, 20.9; HRMS *m/z* (ESI) calcd for $C_{33}H_{33}NO_2$ ([M+Na]$^+$) 498.2404, found 498.2405.



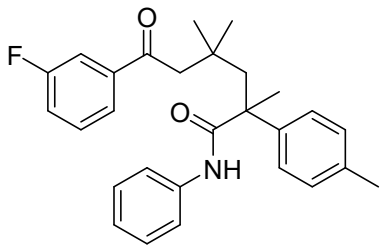
2,4,4-trimethyl-6-oxo-N-phenyl-2,6-di-p-tolylhexanamide (3ab)

44.4 mg, 52% yield; Rf = 0.5 (PE/EA = 10 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.71 (d, *J* = 8.4 Hz, 2H), 7.47 (m, 3H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.25 - 7.22 (m, 2H), 7.20 - 7.17 (m, 2H), 7.15 - 7.12 (m, 2H), 7.03 (t, *J* = 7.2 Hz, 1H), 2.82 (d, *J* = 2.8 Hz, 2H), 2.69 (d, *J* = 15.2 Hz, 1H), 2.39 (s, 3H), 2.35 (s, 1H), 2.31 (s, 3H), 1.75 (s, 3H), 1.12 (d, *J* = 13.2 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.8, 175.4, 143.5, 142.6, 138.2, 136.7, 135.7, 129.4, 129.1, 128.8, 128.1, 126.5, 123.8, 119.7, 51.3, 49.7, 47.7, 34.7, 30.7, 28.4, 25.9, 21.6, 20.9; HRMS *m/z* (ESI) calcd for $C_{29}H_{33}NO_2$ ([M+Na]$^+$) 450.2404, found 450.2402.



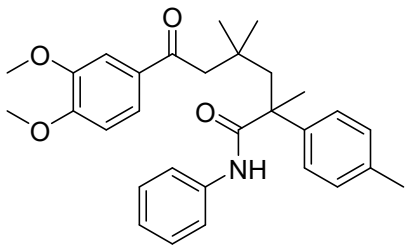
6-(3-methoxyphenyl)-2,4,4-trimethyl-6-oxo-N-phenyl-2-(p-tolyl)hexanamide (3ac)

52.2 mg, 59% yield; Rf = 0.3 (PE/EA = 5 : 1); Brown oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.44 (m, 2H), 7.37 - 7.29 (m, 6H), 7.25 - 7.21 (m, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.06 - 7.00 (m, 2H), 3.82 (s, 3H), 2.87 - 2.76 (m, 2H), 2.67 (d, *J* = 14.8 Hz, 1H), 2.36 (d, *J* = 14.8 Hz, 1H), 2.30 (s, 3H), 1.76 (s, 3H), 1.12 (d, *J* = 15.6 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.9, 175.5, 159.6, 142.4, 139.5, 138.1, 136.8, 129.5, 129.3, 128.8, 126.6, 123.9, 120.6, 119.6, 119.1, 112.2, 55.4, 51.3, 49.7, 47.7, 34.7, 30.5, 28.7, 25.9, 20.9; HRMS *m/z* (ESI) calcd for $C_{29}H_{33}NO_3$ ([M+Na]$^+$) 466.2353, found 466.2349.



6-(3-fluorophenyl)-2,4,4-trimethyl-6-oxo-N-phenyl-2-(p-tolyl)hexanamide (3ad)

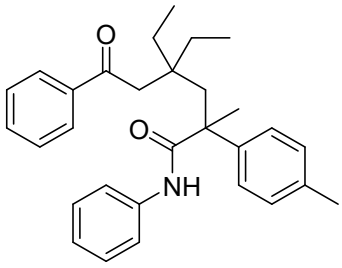
47.4 mg, 55% yield; Rf = 0.5 (PE/EA = 10 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.55 - 7.52 (m, 1H), 7.43 - 7.39 (m, 3H), 7.35 - 7.32 (m, 3H), 7.25 - 7.17 (m, 4H), 7.15 - 7.12 (m, 2H), 7.02 (t, *J* = 7.6 Hz, 1H), 2.82 - 2.70 (m, 2H), 2.65 (d, *J* = 15.2 Hz, 1H), 2.37 (d, *J* = 14.8 Hz, 1H), 2.30 (s, 3H), 1.76 (s, 3H), 1.13 (d, *J* = 18.0 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 198.5 (d, *J* = 2.1 Hz), 175.5, 162.6 (d, *J* = 246.2 Hz), 142.1, 140.1 (d, *J* = 6.0 Hz), 138.0, 136.9, 130.0 (d, *J* = 7.6 Hz), 129.5, 128.8, 126.7, 123.9, 123.6 (d, *J* = 3.2 Hz), 119.7, 119.6, 114.6 (d, *J* = 22.2 Hz), 51.3, 49.4, 47.7, 34.6, 30.4, 29.0, 25.9, 20.8; ^{19}F NMR (376 MHz, Chloroform-*d*) δ (ppm) -112.02; HRMS *m/z* (ESI) calcd for $C_{28}H_{30}FNO_2$ ([M+Na]$^+$) 454.2153, found 454.2152.



6-(3,4-dimethoxyphenyl)-2,4,4-trimethyl-6-oxo-N-phenyl-2-(p-tolyl)hexanamide (3ae)

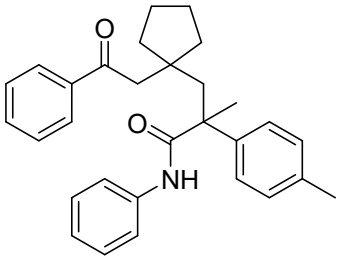
53.9 mg, 57% yield; Rf = 0.3 (PE/EA = 3 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.46 - 7.41 (m, 4H), 7.38 - 7.33 (m, 3H), 7.23 (t, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.80 (d, *J* = 8.8 Hz, 1H), 3.91 (d, *J* = 11.6 Hz, 6H), 2.90 - 2.77 (m, 2H), 2.71 (d, *J* = 14.8 Hz, 1H), 2.34 (d, *J* = 15.2 Hz, 1H), 2.31 (s, 3H), 1.76 (s, 3H), 1.12 (d, *J* = 16.8 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 198.7, 175.4, 153.0, 148.8, 142.6, 138.1,

136.7, 131.4, 129.4, 128.7, 126.5, 123.8, 122.7, 119.6, 109.9, 109.7, 56.0, 55.8, 51.2, 49.1, 47.8, 34.8, 30.7, 28.6, 26.0, 20.9; HRMS *m/z* (ESI) calcd for $C_{30}H_{35}NO_4$ ([M+Na]$^+$) 496.2458, found 496.2453.



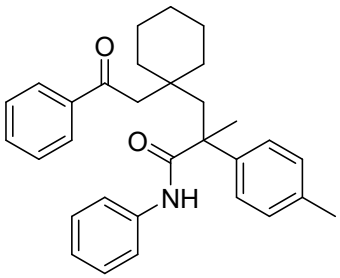
4,4-diethyl-2-methyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide (3af)

54.6 mg, 62% yield; Rf = 0.6 (PE/EA = 10 : 1); White solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.70 (m, 2H), 7.49 - 7.45 (m, 1H), 7.40 - 7.32 (m, 6H), 7.19 (m, 3H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.02 - 6.96 (m, 1H), 2.86 - 2.64 (m, 3H), 2.44 (d, *J* = 15.2 Hz, 1H), 2.28 (s, 3H), 1.74 (s, 3H), 1.66 - 1.54 (m, 4H), 0.79 (m, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.9, 175.9, 142.9, 138.0, 138.0, 136.8, 132.5, 129.5, 128.7, 128.2, 127.6, 126.7, 123.8, 119.5, 51.2, 43.0, 40.6, 39.9, 29.6, 28.7, 25.8, 20.9, 8.0, 7.9; HRMS *m/z* (ESI) calcd for $C_{30}H_{35}NO_2$ ([M+Na]$^+$) 464.2560, found 464.2553.



2-methyl-3-(1-(2-oxo-2-phenylethyl)cyclopentyl)-N-phenyl-2-(p-tolyl)propanamide (3ag)

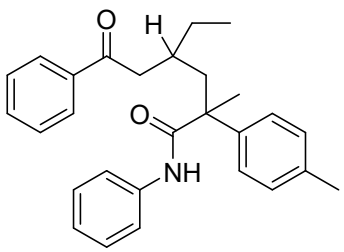
43.9 mg, 50% yield; Rf = 0.5 (PE/EA = 10 : 1); White solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.73 - 7.69 (m, 2H), 7.50 - 7.39 (m, 4H), 7.32 (m, 4H), 7.21 - 7.16 (m, 2H), 7.04 - 6.96 (m, 3H), 3.02 - 2.78 (m, 3H), 2.58 (d, *J* = 15.2 Hz, 1H), 2.18 (s, 3H), 1.89 - 1.76 (m, 2H), 1.70 (s, 3H), 1.68 - 1.51 (m, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.0, 175.6, 142.5, 138.2, 137.5, 136.7, 132.6, 129.4, 128.6, 128.2, 127.7, 126.6, 123.7, 119.5, 51.5, 45.1, 44.7, 44.6, 40.7, 38.6, 25.7, 23.9, 23.5, 20.8; HRMS *m/z* (ESI) calcd for $C_{30}H_{33}NO_2$ ([M+Na]$^+$) 462.2404, found 462.2398.



2-methyl-3-(1-(2-oxo-2-phenylethyl)cyclohexyl)-N-phenyl-2-(p-tolyl)propanamide (3ah)

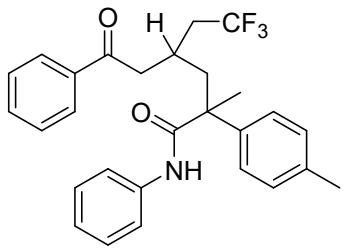
50.7 mg, 56% yield; Rf = 0.5 (PE/EA = 10 : 1); Yellow solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.78 (m, 2H), 7.51 - 7.43 (m, 4H), 7.38 - 7.30 (m, 4H), 7.22 - 7.18 (m, 2H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.99 (t, *J* = 7.2 Hz, 1H), 3.20 - 3.11 (m, 1H),

2.88 - 2.79 (m, 2H), 2.42 (d, *J* = 15.2 Hz, 1H), 2.24 (s, 3H), 1.95 - 1.85 (m, 1H), 1.74 (s, 3H), 1.65 (m, 2H), 1.58 - 1.40 (m, 6H), 1.28 - 1.25 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.3, 175.7, 143.1, 138.1, 138.1, 136.6, 132.6, 129.4, 128.7, 128.3, 127.7, 126.5, 123.7, 119.6, 51.2, 38.0, 37.3, 35.6, 26.7, 26.0, 22.0, 21.9, 20.8; HRMS *m/z* (ESI) calcd for $C_{31}H_{35}NO_2$ ([M+Na]$^+$) 476.2560, found 476.2561.



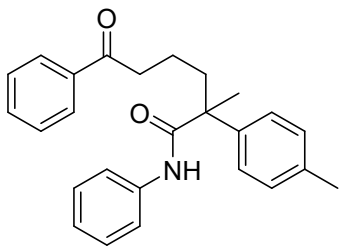
4-ethyl-2-methyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide (3ai)

42.1 mg, 51% yield; Rf = 0.6 (PE/EA = 10 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.84 (m, 2H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.40 - 7.36 (m, 4H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.25 - 7.20 (m, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.10 (s, 1H), 7.03 (t, *J* = 7.6 Hz, 1H), 2.93 (d, *J* = 6.0 Hz, 2H), 2.33 (s, 3H), 2.25 - 2.15 (m, 2H), 2.09 (m, 1H), 1.68 (s, 3H), 1.59 (s, 1H), 1.21 (m, 1H), 0.76 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.3, 175.3, 141.2, 138.0, 137.2, 137.0, 132.8, 129.5, 128.8, 128.4, 128.0, 126.7, 124.0, 119.7, 51.3, 43.9, 42.5, 31.8, 28.1, 24.4, 20.9, 10.6; HRMS *m/z* (ESI) calcd for $C_{28}H_{31}NO_2$ ([M+Na]$^+$) 436.2247, found 436.2245.



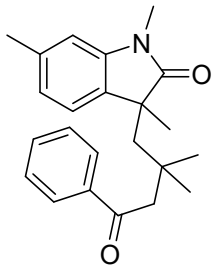
6,6,6-trifluoro-2-methyl-4-(2-oxo-2-phenylethyl)-N-phenyl-2-(p-tolyl)hexanamide (3aj)

45.7 mg, 49% yield; Rf = 0.6 (PE/EA = 10 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.81 (m, 2H), 7.54 - 7.50 (m, 1H), 7.39 (t, *J* = 8.0 Hz, 2H), 7.33 - 7.28 (m, 4H), 7.24 - 7.18 (m, 4H), 7.03 (t, *J* = 7.2 Hz, 1H), 6.91 (s, 1H), 3.08 (m, 2H), 2.62 - 2.48 (m, 1H), 2.34 (s, 3H), 2.29 (d, *J* = 5.2 Hz, 1H), 2.23 (d, *J* = 6.0 Hz, 1H), 2.12 (m, 1H), 1.91 (m, 1H), 1.72 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 198.7, 175.1, 139.9, 137.7, 137.5, 136.9, 133.1, 129.8, 128.8, 128.5, 127.8, 126.7, 124.2, 119.7, 51.0, 43.1, 42.7, 37.7 (q, *J* = 26.7 Hz), 25.3, 25.3, 23.7, 20.9; ^{19}F NMR (376 MHz, Chloroform-*d*) δ (ppm) -62.53; HRMS *m/z* (ESI) calcd for $C_{28}H_{28}F_3NO_3$ ([M+Na]$^+$) 490.1964, found 490.1959.



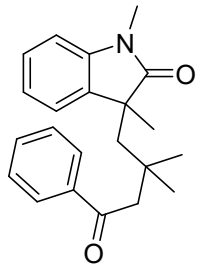
2-methyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide (3ak)

30.8 mg, 40% yield; Rf = 0.5 (PE/EA = 10 : 1); Yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.92 (m, 2H), 7.57 - 7.51 (m, 1H), 7.48 - 7.42 (m, 4H), 7.32 - 7.27 (m, 4H), 7.25 (s, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.06 (t, *J* = 7.2 Hz, 1H), 3.00 (m, 2H), 2.35 (s, 3H), 2.23 - 2.03 (m, 2H), 1.83 - 1.71 (m, 1H), 1.67 (s, 3H), 1.60 (s, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.1, 175.20, 140.4, 138.1, 136.9, 136.8, 133.0, 129.6, 128.8, 128.6, 128.0, 126.7, 124.0, 119.8, 51.2, 38.5, 23.8, 21.0, 19.1; HRMS *m/z* (ESI) calcd for $C_{26}H_{27}NO_2$ ($[M+Na]^+$) 408.1934, found 408.1936.



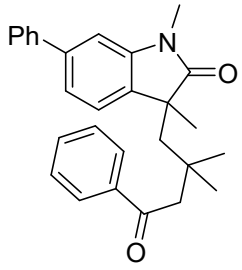
3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one (4ia)

46.8 mg, 67% yield; Rf = 0.5 (PE/EA = 5 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.68 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.39 - 7.35 (m, 2H), 7.04 (d, *J* = 7.2 Hz, 1H), 6.68 - 6.63 (m, 2H), 3.21 (s, 3H), 2.62 (d, *J* = 16.0 Hz, 1H), 2.42 (d, *J* = 14.8 Hz, 1H), 2.35 (d, *J* = 16.4 Hz, 1H), 2.32 (s, 3H), 2.18 (d, *J* = 14.8 Hz, 1H), 1.29 (s, 3H), 0.90 (s, 3H), 0.75 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.7, 181.4, 142.7, 138.2, 137.7, 132.6, 130.8, 128.2, 127.8, 123.6, 122.9, 109.1, 48.5, 48.3, 47.0, 34.6, 29.4, 28.3, 28.2, 26.3, 21.7; HRMS *m/z* (ESI) calcd for $C_{23}H_{27}NO_2$ ([M+Na]$^+$) 372.1934, found 372.1940.



3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethylindolin-2-one (4pa)

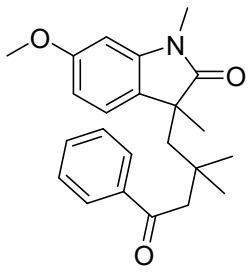
46.9 mg, 70% yield; Rf = 0.5 (PE/EA = 5 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.69 (m, 2H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.40 - 7.35 (m, 2H), 7.23 - 7.17 (m, 2H), 6.88 - 6.83 (m, 2H), 3.23 (s, 3H), 2.63 (d, *J* = 16.0 Hz, 1H), 2.46 (d, *J* = 14.4 Hz, 1H), 2.35 (d, *J* = 16.0 Hz, 1H), 2.22 (d, *J* = 14.8 Hz, 1H), 1.32 (s, 3H), 0.89 (s, 3H), 0.74 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.7, 181.1, 142.6, 138.2, 133.8, 132.6, 128.3, 127.8, 127.6, 123.8, 122.3, 108.1, 48.6, 48.2, 47.3, 34.6, 29.3, 28.3, 28.2, 26.3; HRMS *m/z* (ESI) calcd for $C_{22}H_{25}NO_2$ ([M+Na]$^+$) 358.1783, found 358.1793.



3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethyl-6-phenylindolin-2-one (4qa)

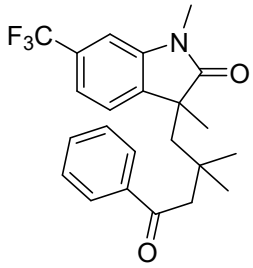
44.4 mg, 54% yield; Rf = 0.4 (PE/EA = 5 : 1); Yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.63 (m, 2H), 7.47 - 7.39 (m, 3H), 7.38 - 7.30 (m, 4H), 7.28 - 7.24 (m, 3H), 6.93 (m, 1H), 3.27 (s, 3H), 2.67 (d, *J* = 16.4 Hz, 1H), 2.58 (d, *J* = 14.8 Hz, 1H), 2.42 (d, *J* = 16.4 Hz, 1H), 2.25 (d, *J* = 14.8 Hz, 1H), 1.37 (s, 3H), 0.93 (s, 3H), 0.80 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.4, 181.1, 142.0, 140.5, 137.9, 135.6, 134.4, 132.6, 128.6, 128.2, 127.7, 126.8, 126.6, 126.5, 122.7, 108.4, 48.5, 48.1, 47.5,

34.5, 29.4, 28.4, 28.3, 26.4; HRMS *m/z* (ESI) calcd for $C_{28}H_{29}NO_2$ ([M+Na]$^+$) 434.2091, found 434.2098.



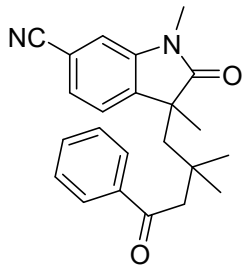
3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-6-methoxy-1,3-dimethylindolin-2-one (4ra)

44.6 mg, 61% yield; Rf = 0.3 (PE/EA = 4 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.71 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 8.0 Hz, 2H), 6.79 - 6.68 (m, 3H), 3.53 (s, 3H), 3.21 (s, 3H), 2.61 (d, *J* = 16.4 Hz, 1H), 2.50 (d, *J* = 14.8 Hz, 1H), 2.37 (d, *J* = 16.8 Hz, 1H), 2.19 (d, *J* = 14.4 Hz, 1H), 1.31 (s, 3H), 0.93 (s, 3H), 0.77 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.5, 180.7, 155.7, 138.0, 136.1, 135.1, 132.6, 128.2, 127.8, 111.9, 111.1, 108.4, 55.4, 48.3, 47.8, 47.7, 34.4, 29.4, 28.5, 28.2, 26.3; HRMS *m/z* (ESI) calcd for $C_{23}H_{27}NO_3$ ([M+Na]$^+$) 388.1889, found 388.1891.



3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethyl-6-(trifluoromethyl)indolin-2-one (4sa)

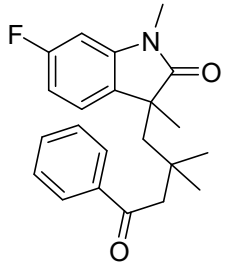
40.3 mg, 50% yield; Rf = 0.5 (PE/EA = 5 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.72 (m, 2H), 7.53 - 7.46 (m, 3H), 7.38 (t, *J* = 8.0 Hz, 2H), 6.93 (d, *J* = 8.0 Hz, 1H), 3.26 (s, 3H), 2.65 (d, *J* = 16.4 Hz, 1H), 2.50 (d, *J* = 14.4 Hz, 1H), 2.38 (d, *J* = 16.4 Hz, 1H), 2.27 (d, *J* = 14.8 Hz, 1H), 1.35 (s, 3H), 0.86 (s, 3H), 0.75 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.2, 180.9, 145.6, 137.9, 134.5, 132.8, 128.4, 127.7, 125.5 (q, *J* = 4.0 Hz), 124.7, 124.4, 120.8 (q, *J* = 4.0 Hz), 107.9, 48.8, 48.3, 47.3, 34.6, 29.1, 28.3, 28.2, 26.5; ^{19}F NMR (376 MHz, Chloroform-*d*) δ (ppm) -61.42; HRMS *m/z* (ESI) calcd for $C_{23}H_{24}F_3NO_2$ ([M+Na]$^+$) 426.1651, found 426.1659.



3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethyl-2-oxoindoline-6-carbonitrile (4ta)

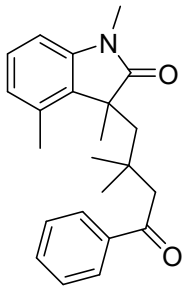
35.3 mg, 49% yield; Rf = 0.3 (PE/EA = 5 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.76 (m, 2H), 7.56 - 7.49 (m, 2H), 7.45 - 7.40 (m, 3H), 6.90 (d, *J* = 8.0 Hz, 1H), 3.26 (s, 3H), 2.66 (d, *J* = 16.8 Hz, 1H), 2.54 (d, *J* = 14.4 Hz, 1H), 2.36 (d, *J* = 16.8 Hz, 1H), 2.25 (d, *J* = 14.8 Hz, 1H), 1.33 (s, 3H), 0.88 (s, 3H), 0.74 (s, 3H); ^{13}C NMR (100

MHz, Chloroform-*d*) δ (ppm) 199.1, 180.6, 146.4, 137.8, 135.0, 133.0, 128.5, 127.7, 127.0, 118.8, 108.5, 105.5, 48.7, 47.7, 47.1, 34.4, 29.2, 28.7, 28.2, 26.5; HRMS *m/z* (ESI) calcd for $C_{23}H_{24}N_2O_2$ ([M+Na]$^+$) 383.1730, found 383.1736.



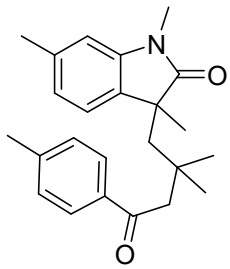
3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-6-fluoro-1,3-dimethylindolin-2-one (4ua)

38.1 mg, 54% yield; Rf = 0.5 (PE/EA = 5 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.74 (m, 2H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 2H), 6.96 - 6.86 (m, 2H), 6.77 (m, 1H), 3.22 (s, 3H), 2.67 (d, *J* = 16.4 Hz, 1H), 2.47 - 2.37 (m, 2H), 2.23 (d, *J* = 14.8 Hz, 1H), 1.31 (s, 3H), 0.89 (s, 3H), 0.77 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.4, 180.6 (d, *J* = 1.5 Hz), 159.1 (d, *J* = 239.8 Hz), 138.5 (d, *J* = 2.0 Hz), 138.0, 135.6 (d, *J* = 7.9 Hz), 132.7, 128.3, 127.7, 113.8 (d, *J* = 23.5 Hz), 112.0 (d, *J* = 24.5 Hz), 108.5 (d, *J* = 8.3 Hz), 48.6, 48.0, 47.7 (d, *J* = 2.0 Hz), 34.5, 29.2, 28.5, 28.2, 26.4; ^{19}F NMR (376 MHz, Chloroform-*d*) δ (ppm) -120.61; HRMS *m/z* (ESI) calcd for $C_{22}H_{24}FNO_2$ ([M+Na]$^+$) 376.1683, found 376.1686.



3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3,4-trimethylindolin-2-one (4va)

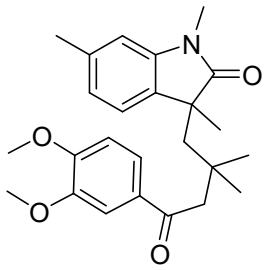
27.9 mg, 40% yield; Rf = 0.5 (PE/EA = 5 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.69 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.38 (t, *J* = 8.0 Hz, 2H), 7.04 - 6.99 (m, 1H), 6.93 (d, *J* = 7.6 Hz, 1H), 6.75 (t, *J* = 7.6 Hz, 1H), 3.51 (s, 3H), 2.68 - 2.57 (m, 4H), 2.43 - 2.33 (m, 2H), 2.20 (d, *J* = 14.4 Hz, 1H), 1.29 (s, 3H), 0.89 (s, 3H), 0.74 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.8, 181.8, 140.4, 138.3, 134.4, 132.6, 131.3, 128.3, 127.9, 122.2, 121.8, 119.8, 48.7, 48.6, 46.6, 34.6, 29.6, 29.3, 28.7, 28.1, 19.1; HRMS *m/z* (ESI) calcd for $C_{23}H_{27}NO_2$ ([M+Na]$^+$) 372.1934, found 372.1945.



3-(2,2-dimethyl-4-oxo-4-(p-tolyl)butyl)-1,3,6-trimethylindolin-2-one (4ib)

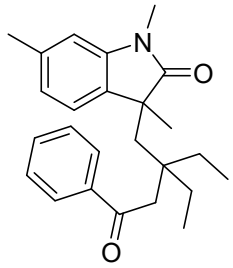
42.8 mg, 59% yield; Rf = 0.5 (PE/EA = 5 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.59 (d, *J* = 8.4 Hz, 2H), 7.20 - 7.13 (m, 2H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.70 - 6.63 (m, 2H),

3.21 (s, 3H), 2.60 (d, *J* = 16.0 Hz, 1H), 2.40 (d, *J* = 13.2 Hz, 4H), 2.35 - 2.30 (m, 4H), 2.18 (d, *J* = 14.4 Hz, 1H), 1.29 (s, 3H), 0.88 (s, 3H), 0.73 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.4, 181.4, 143.2, 142.7, 137.7, 135.8, 130.8, 128.9, 128.0, 123.6, 122.8, 109.0, 48.5, 48.4, 47.0, 34.6, 29.3, 28.3, 28.2, 26.2, 21.7, 21.5; HRMS *m/z* (ESI) calcd for $C_{24}H_{29}NO_2$ ([M+Na]$^+$) 386.2091, found 386.2099.



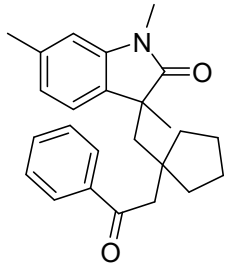
3-(4-(3,4-dimethoxyphenyl)-2,2-dimethyl-4-oxobutyl)-1,3,6-trimethylindolin-2-one (4ie)

49.9 mg, 61% yield; Rf = 0.3 (PE/EA = 2 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.41 (d, *J* = 2.0 Hz, 1H), 7.25 (m, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.78 (d, *J* = 8.4 Hz, 1H), 6.69 (d, *J* = 6.0 Hz, 2H), 3.93 (d, *J* = 7.6 Hz, 6H), 3.22 (s, 3H), 2.61 (d, *J* = 16.0 Hz, 1H), 2.39 (d, *J* = 14.8 Hz, 1H), 2.37 - 2.33 (m, 4H), 2.19 (d, *J* = 14.4 Hz, 1H), 1.29 (s, 3H), 0.87 (s, 3H), 0.74 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 198.3, 181.4, 152.8, 148.8, 142.7, 137.6, 131.5, 130.8, 123.7, 122.8, 122.6, 109.9, 109.5, 109.0, 56.0, 55.9, 48.4, 48.1, 47.1, 34.7, 29.2, 28.4, 28.3, 26.3, 21.7; HRMS *m/z* (ESI) calcd for $C_{25}H_{31}NO_4$ ([M+Na]$^+$) 432.2145, found 432.2146.



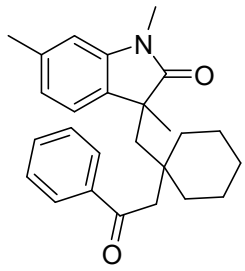
3-(2,2-diethyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one (4if)

43.7 mg, 58% yield; Rf = 0.5 (PE/EA = 5 : 1); Yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.69 - 7.61 (m, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.36 (t, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 7.6 Hz, 1H), 6.63 (s, 1H), 6.46 (d, *J* = 7.6 Hz, 1H), 3.19 (s, 3H), 2.67 - 2.37 (m, 2H), 2.29 - 2.14 (m, 5H), 1.49 - 1.31 (m, 2H), 1.29 (s, 3H), 1.25 - 1.20 (m, 1H), 1.05 (m, 1H), 0.72 (m, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.4, 181.3, 142.5, 138.2, 137.5, 132.4, 131.1, 128.1, 127.5, 123.4, 122.8, 108.9, 46.7, 43.0, 41.9, 39.5, 28.7, 28.6, 28.5, 26.2, 21.6, 7.8, 7.6; HRMS *m/z* (ESI) calcd for $C_{25}H_{31}NO_2$ ([M+Na]$^+$) 400.2247, found 400.2248.



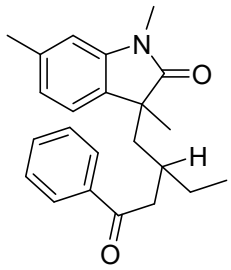
1,3,6-trimethyl-3-((1-(2-oxo-2-phenylethyl)cyclopentyl)methyl)indolin-2-one (4ig)

41.3 mg, 55% yield; Rf = 0.5 (PE/EA = 5 : 1); Yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.60 (d, *J* = 7.2 Hz, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 2H), 6.83 (d, *J* = 7.6 Hz, 1H), 6.61 (s, 1H), 6.25 (d, *J* = 7.6 Hz, 1H), 3.18 (s, 3H), 2.81 (d, *J* = 14.4 Hz, 1H), 2.49 (d, *J* = 18.8 Hz, 1H), 2.28 - 2.09 (m, 5H), 1.66 - 1.39 (m, 6H), 1.28 (s, 3H), 1.27 - 1.10 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.3, 181.4, 142.5, 137.8, 137.4, 132.3, 131.0, 128.0, 127.5, 123.2, 123.0, 108.7, 47.4, 45.0, 44.6, 44.3, 40.0, 38.2, 27.5, 26.2, 23.9, 23.3, 21.5; HRMS *m/z* (ESI) calcd for $C_{25}H_{29}NO_2$ ([M+Na]$^+$) 398.2091, found 398.2095.



1,3,6-trimethyl-3-((1-(2-oxo-2-phenylethyl)cyclohexyl)methyl)indolin-2-one (4ih)

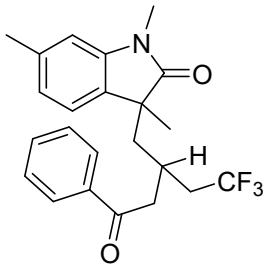
40.5 mg, 52% yield; Rf = 0.5 (PE/EA = 5 : 1); Yellow solid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.69 - 7.62 (m, 2H), 7.54 - 7.46 (m, 1H), 7.36 (t, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 7.6 Hz, 1H), 6.63 (s, 1H), 6.32 (d, *J* = 7.2 Hz, 1H), 3.18 (s, 3H), 2.76 (m, 2H), 2.29 - 2.10 (m, 5H), 1.53 - 1.20 (m, 13H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.6, 181.6, 142.4, 138.3, 137.4, 132.4, 131.0, 128.1, 127.6, 123.3, 122.9, 108.9, 46.7, 36.8, 36.7, 36.5, 28.4, 26.3, 26.0, 21.7, 21.6, 21.5; HRMS *m/z* (ESI) calcd for $C_{26}H_{31}NO_2$ ([M+Na]$^+$) 412.2247, found 412.2250.



3-(2-ethyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one (4ii)

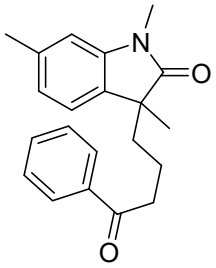
41.9 mg, 60% yield; Rf = 0.6 (PE/EA = 5 : 1); Yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.72 - 7.68 (m, 2.00H), 7.66 - 7.62 (m, 0.98H), 7.53 - 7.47 (t, *J* = 7.6 Hz, 1.55H), 7.36 (t, *J* = 7.6 Hz, 3.03H), 7.07 (t, *J* = 3.6 Hz, 0.98H), 7.05 (s, 0.43H), 6.89 (d, *J* = 7.2 Hz, 0.94H), 6.79 (d, *J* = 7.6 Hz, 0.42H), 6.69 (s, 0.97H), 6.63 (s, 0.43H), 3.20 (s, 1.50H), 3.11 (s, 3.00H), 2.80 (m, 0.96H), 2.70 - 2.56 (m, 1.92H), 2.41 (s, 3.00H), 2.31 (s, 1.51H), 2.07 (m, 0.52H), 2.01 - 1.94 (m, 2.01H), 1.84 (m, 0.50H), 1.35 (s, 3.18H), 1.31 (s, 1.58H), 1.28 (s, 0.51H), 1.25 (s, 1.08H), 1.19 - 1.13 (m, 2.00H), 1.11 - 1.06 (m, 1.02H), 0.76 (t, *J* = 7.2 Hz, 1.50H), 0.71 (t, *J* = 7.2 Hz, 3.00H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.8, 181.2, 143.3, 143.2, 137.9, 137.8, 137.0, 137.0,

132.7, 132.7, 130.6, 130.6, 128.4, 128.3, 128.0, 127.9, 123.1, 123.0, 123.0, 122.6, 109.2, 109.0, 47.8, 47.7, 43.4, 43.2, 41.8, 41.7, 32.8, 32.4, 28.0, 27.0, 26.2, 26.1, 25.9, 25.8, 21.8, 21.7, 10.5, 10.3; HRMS *m/z* (ESI) calcd for $C_{23}H_{27}NO_2$ ($[M+Na]^+$) 372.1939, found 372.1944.



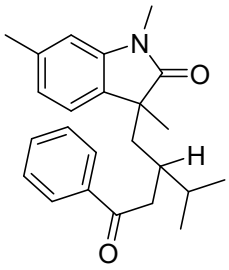
1,3,6-trimethyl-3-(4,4,4-trifluoro-2-(2-oxo-2-phenylethyl)butyl)indolin-2-one (4ij)

44.3 mg, 55% yield; Rf = 0.6 (PE/EA = 5 : 1); Colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.75 (m, 2H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.43 - 7.38 (m, 2H), 7.08 (d, *J* = 7.6 Hz, 1H), 6.94 - 6.90 (m, 1H), 6.70 - 6.68 (m, 1H), 3.10 (s, 3H), 2.97 - 2.80 (m, 2H), 2.40 (s, 3H), 2.21 - 2.08 (m, 3H), 1.98 (m, 2H), 1.34 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 198.2, 180.7, 143.1, 138.3, 136.7, 133.1, 129.7, 128.5, 127.8, 123.5, 122.7, 109.3, 47.7, 41.8, 41.6, 37.7 (q, *J* = 26.9 Hz), 26.2 (q, *J* = 2.1 Hz), 26.1, 25.9, 21.8; ^{19}F NMR (376 MHz, Chloroform-*d*) δ (ppm) -62.71; HRMS *m/z* (ESI) calcd for $C_{23}H_{24}F_3NO_2$ ($[M+Na]^+$) 426.1651, found 426.1659.



1,3,6-trimethyl-3-(4-oxo-4-phenylbutyl)indolin-2-one (4ik)

26.3 mg, 41% yield; Rf = 0.5 (PE/EA = 5 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.90 - 7.81 (m, 2H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 2H), 7.08 (d, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 7.6 Hz, 1H), 6.67 (s, 1H), 3.20 (s, 3H), 2.96 - 2.76 (m, 2H), 2.38 (s, 3H), 1.99 - 1.83 (m, 2H), 1.35 (s, 4H), 1.25 (s, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 199.6, 180.9, 143.3, 137.8, 136.8, 132.9, 130.7, 128.5, 127.9, 123.1, 122.4, 109.0, 48.2, 38.4, 37.8, 26.1, 24.0, 21.8, 19.3; HRMS *m/z* (ESI) calcd for $C_{21}H_{23}NO_2$ ($[M+Na]^+$) 344.1621, found 344.1617.



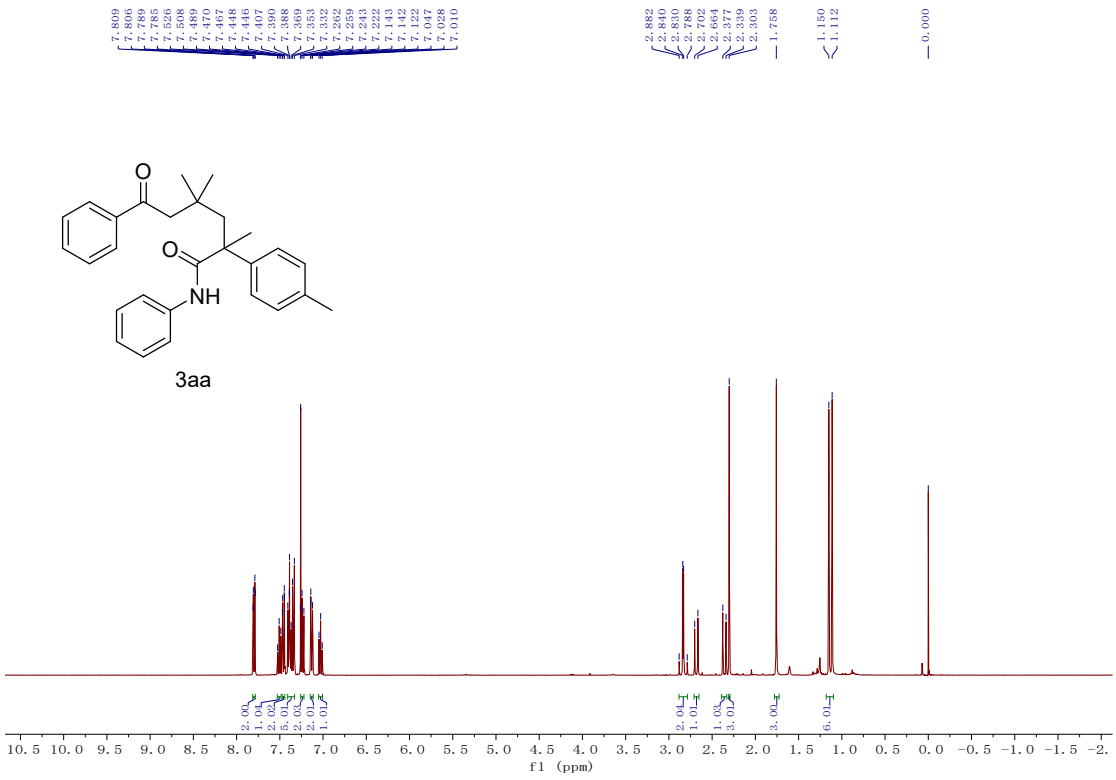
3-(2-isopropyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one (4il)

36.3 mg, 50% yield; Rf = 0.5 (PE/EA = 5 : 1); Light yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ (ppm) 7.93 (m, 2H), 7.58 - 7.53 (m, 1H), 7.45 (m, 2H), 7.07 (d, *J* = 7.2 Hz, 1H), 6.81 (m, 1H), 6.67 (s, 1H), 3.17 (s, 3H), 2.88 (m, 2H), 2.38 (s, 3H), 2.21 (d, *J* = 14.8 Hz, 1H), 1.87

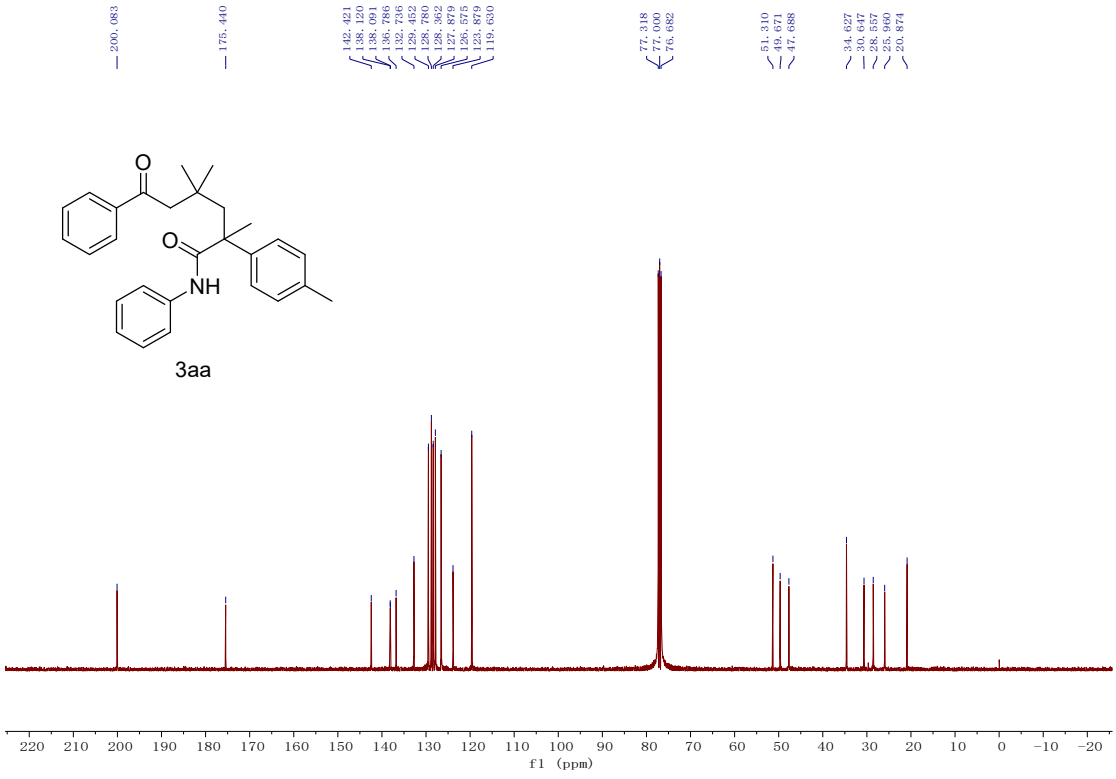
(d, J = 14.4 Hz, 1H), 1.38 (m, 2H), 1.28 (s, 3H), 0.63 (d, J = 4.8 Hz, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ (ppm) 200.8, 181.2, 142.8, 137.7, 137.1, 132.8, 130.9, 128.5, 128.0, 123.5, 122.6, 109.2, 48.4, 47.0, 37.3, 34.0, 33.7, 28.6, 28.5, 27.5, 26.2, 21.8; HRMS *m/z* (ESI) calcd for $C_{24}H_{29}NO_2$ ($[M+Na]^+$) 386.2091, found 386.2094.

(C) Spectra (NMR Spectra)

2,4,4-trimethyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide

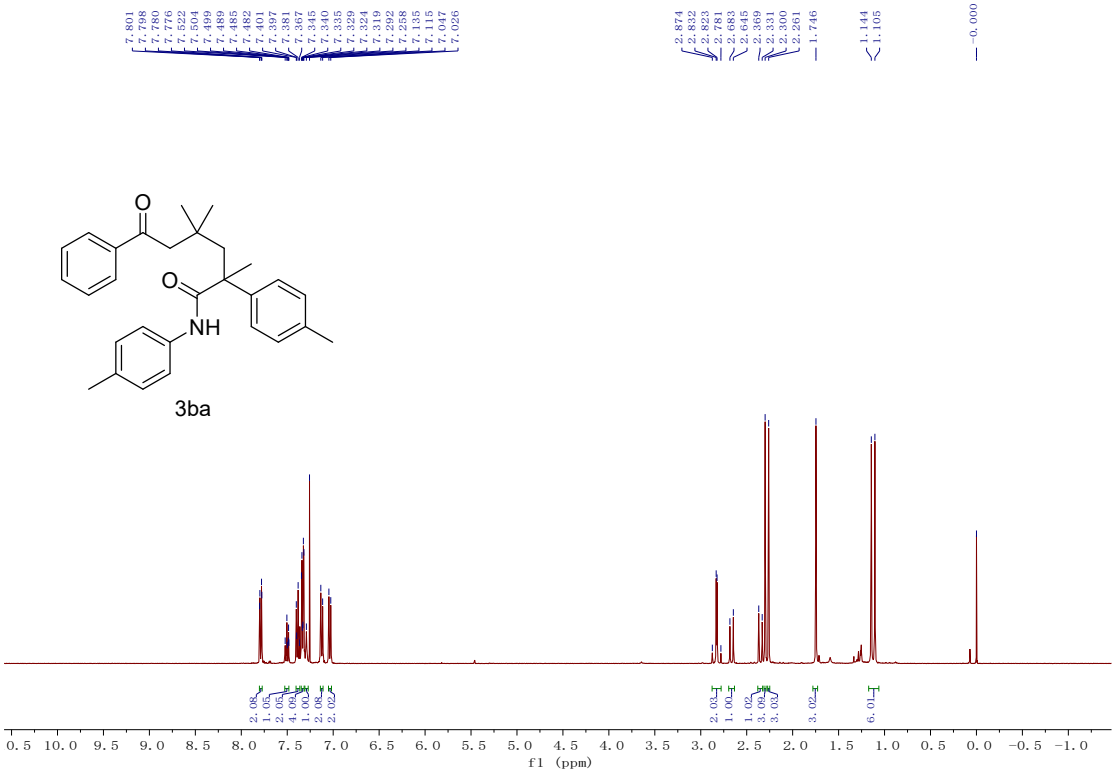


^{1}H NMR (400 MHz, $CDCl_3$)

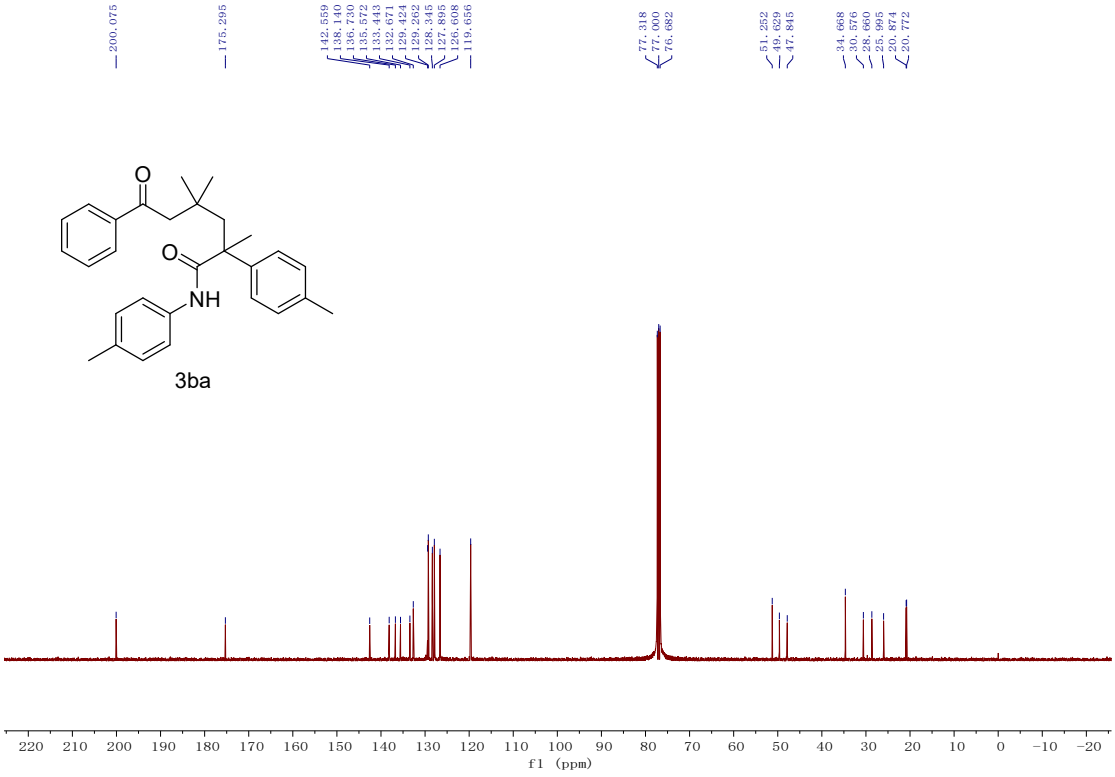


^{13}C NMR (100 MHz, $CDCl_3$)

2,4,4-trimethyl-6-oxo-6-phenyl-N,2-di-p-tolylhexanamide

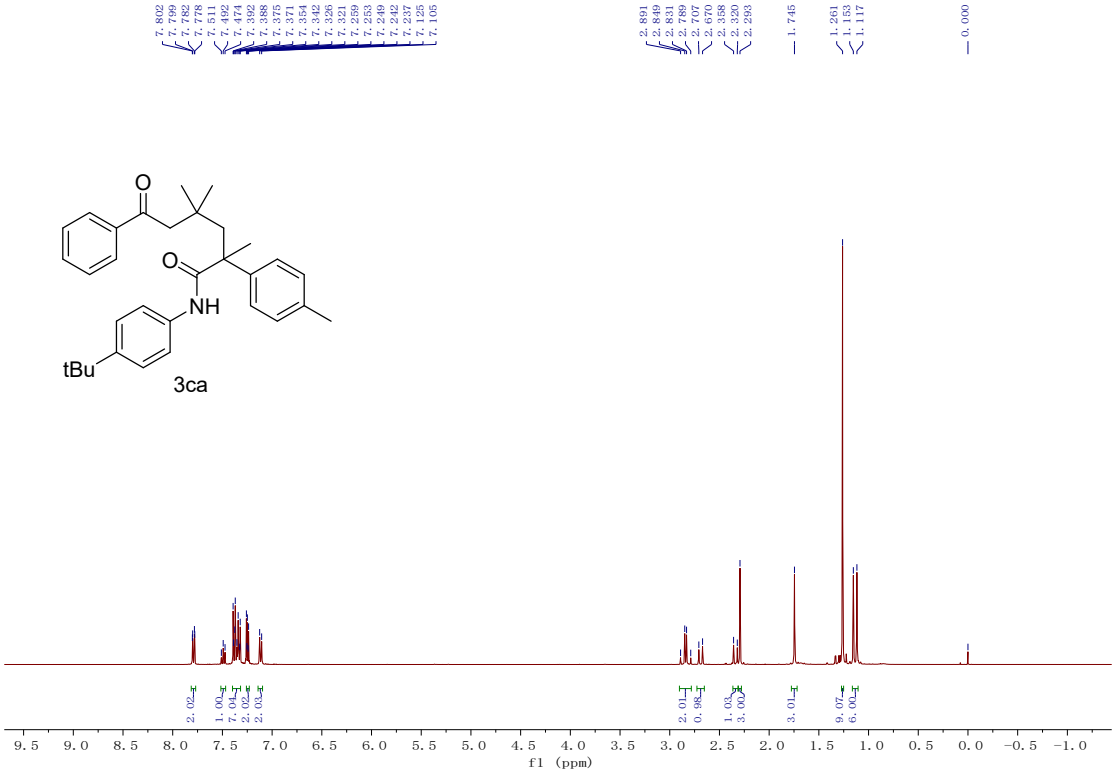


^{1}H NMR (400 MHz, $CDCl_3$)

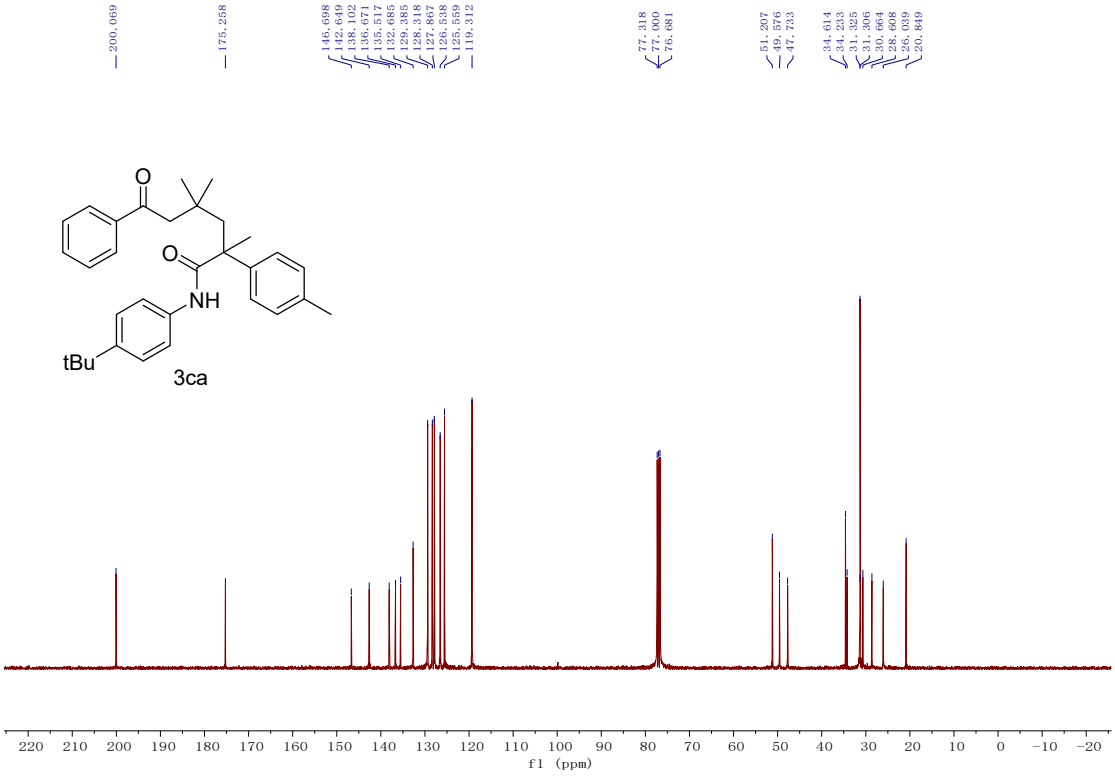


^{13}C NMR (100 MHz, $CDCl_3$)

N-(4-(tert-butyl)phenyl)-2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide

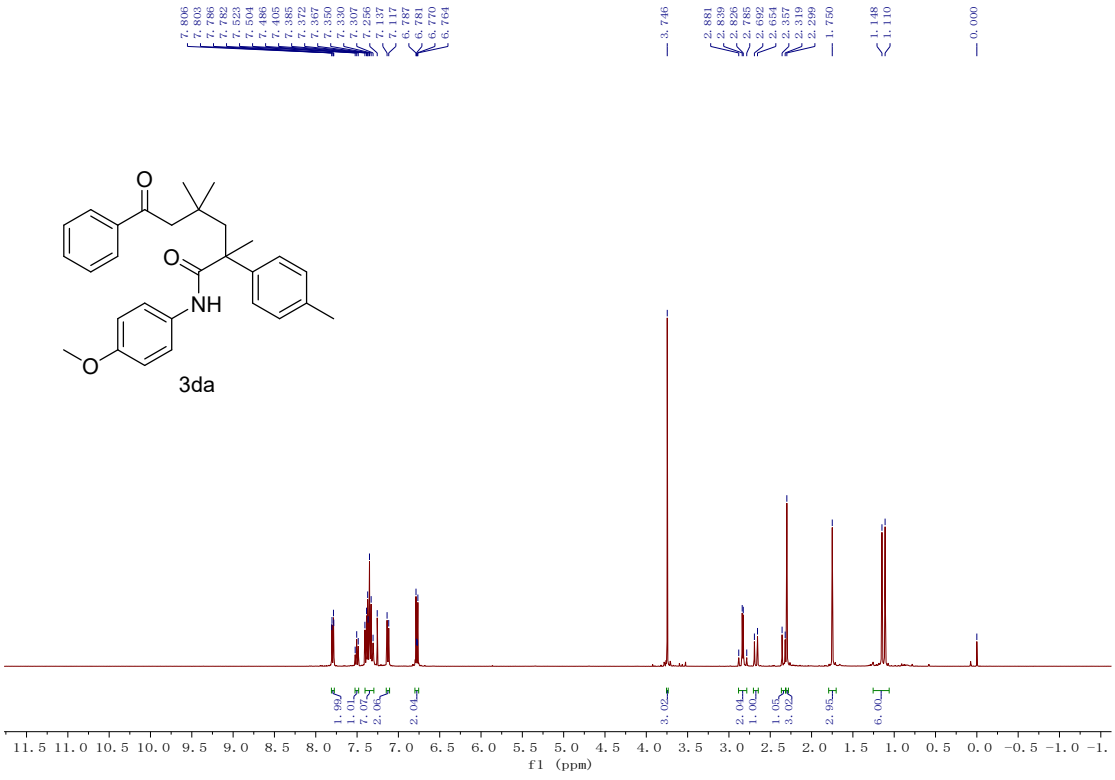


^{1}H NMR (400 MHz, $CDCl_3$)

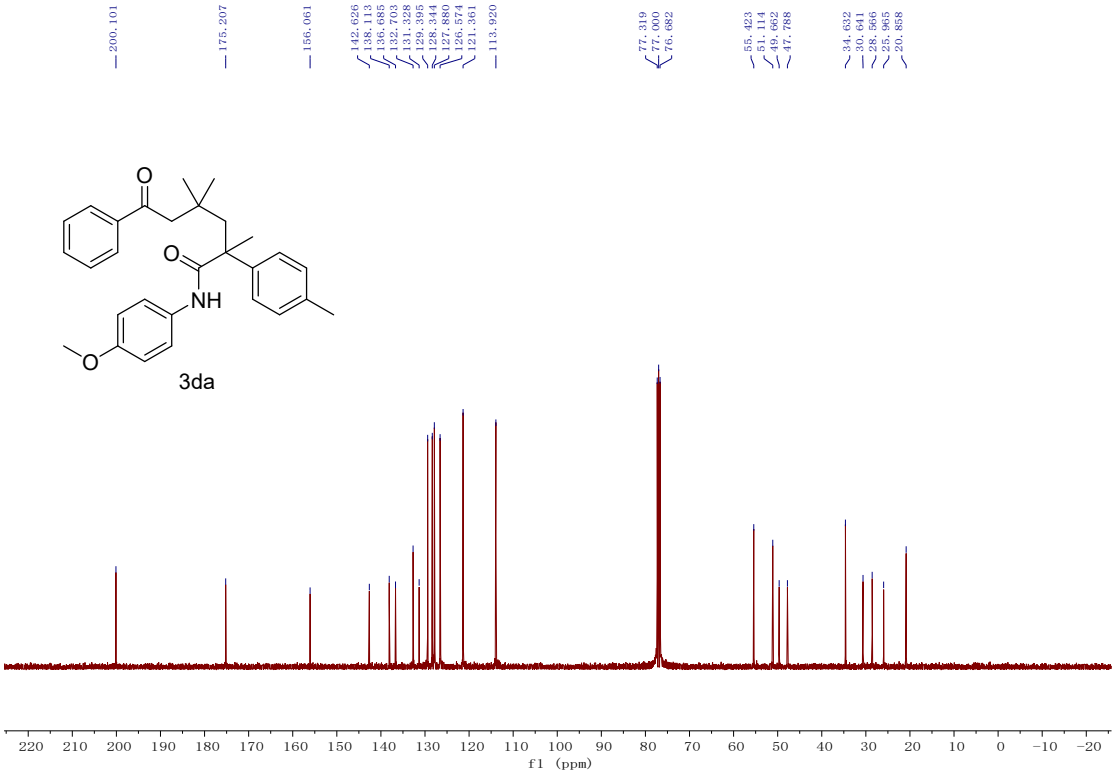


^{13}C NMR (100 MHz, $CDCl_3$)

N-(4-methoxyphenyl)-2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide

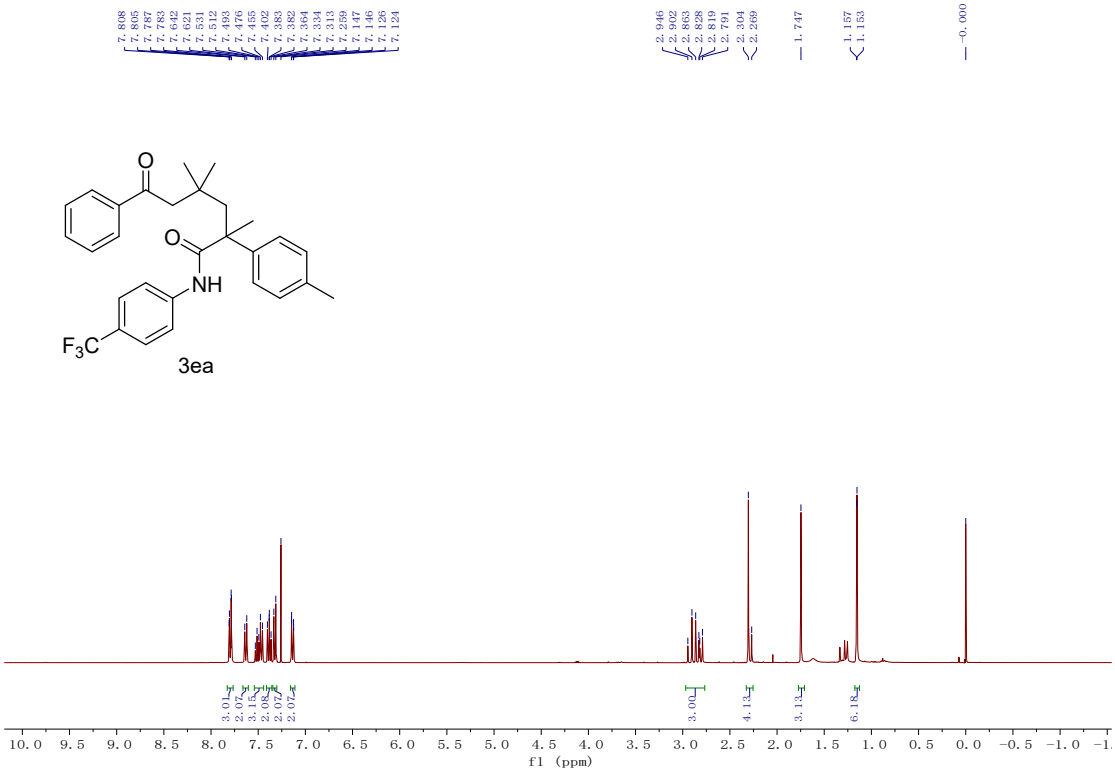


^{1}H NMR (400 MHz, $CDCl_3$)

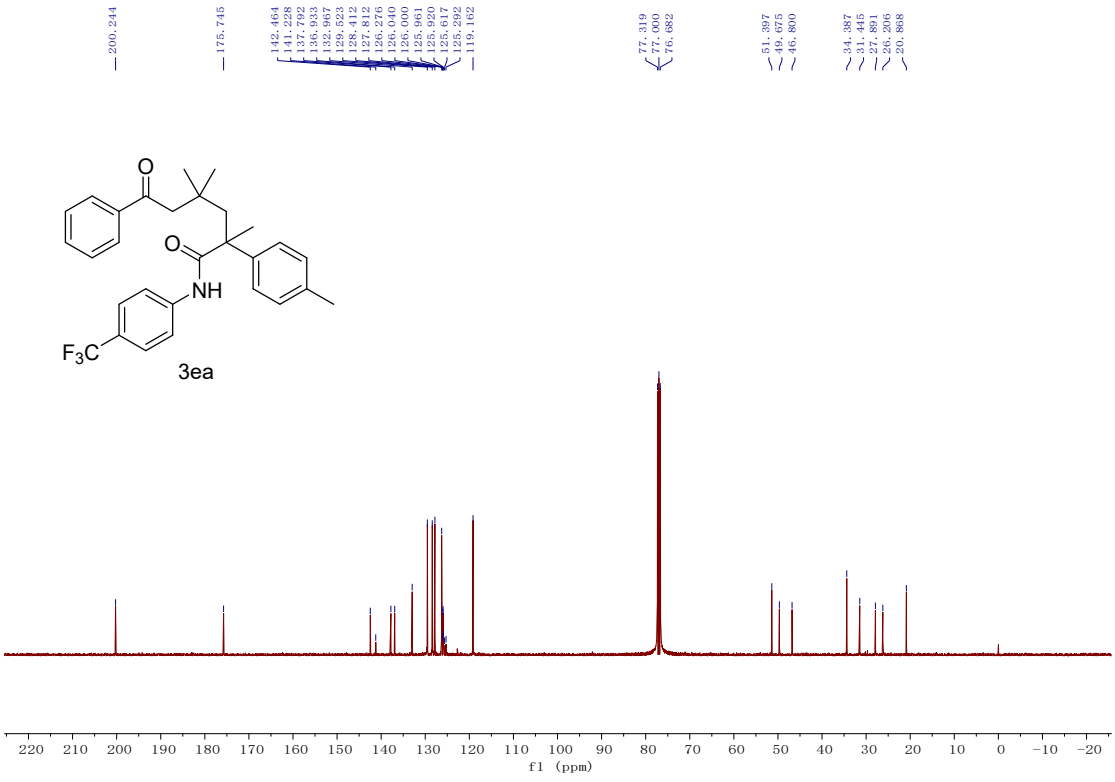


^{13}C NMR (100 MHz, $CDCl_3$)

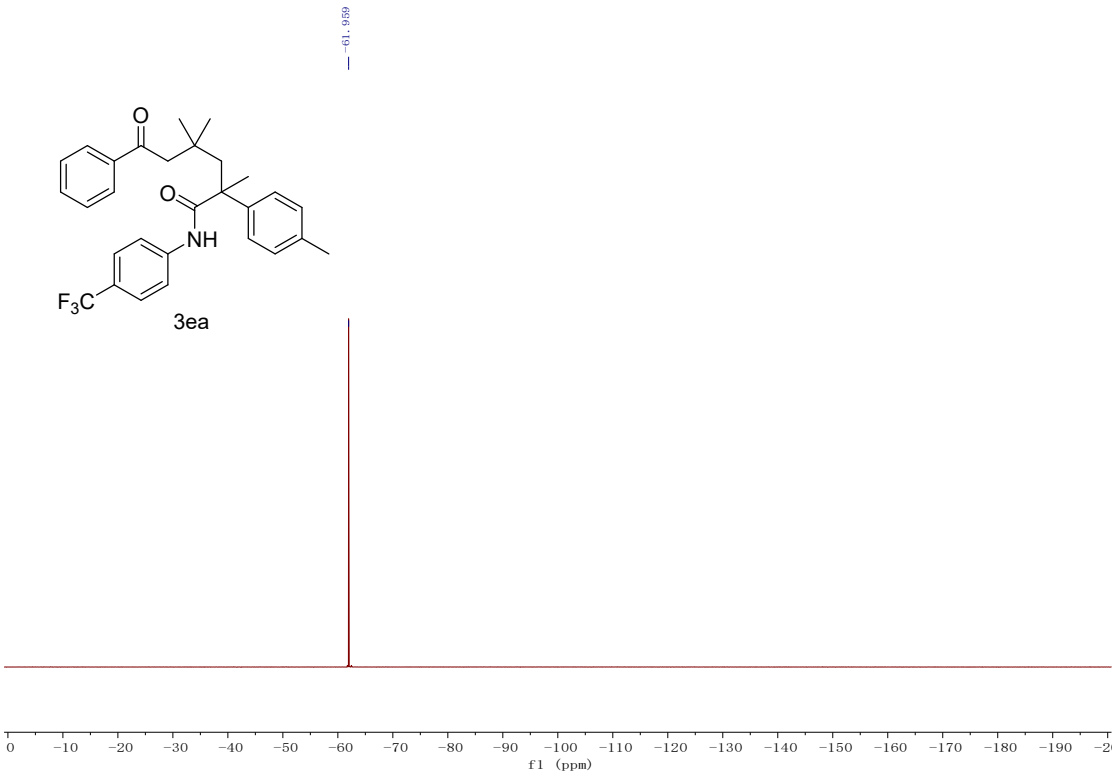
2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)-N-(4-(trifluoromethyl)phenyl)hexanamide



^{1}H NMR (400 MHz, $CDCl_3$)

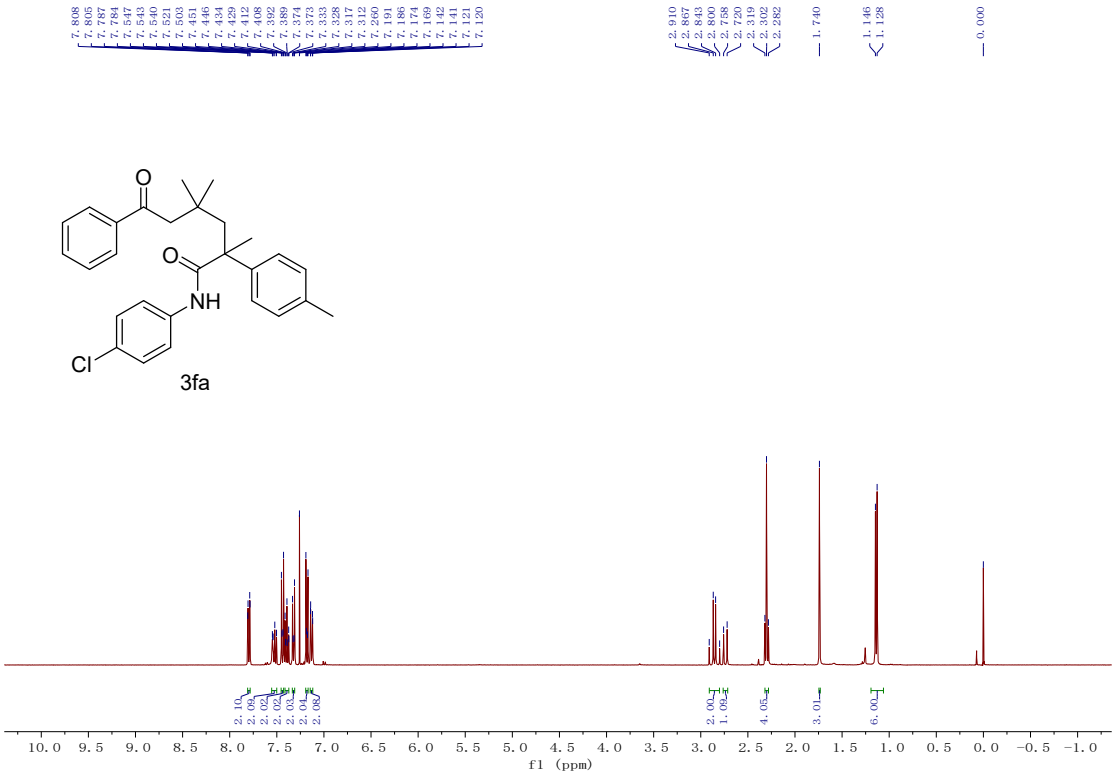


^{13}C NMR (100 MHz, $CDCl_3$)

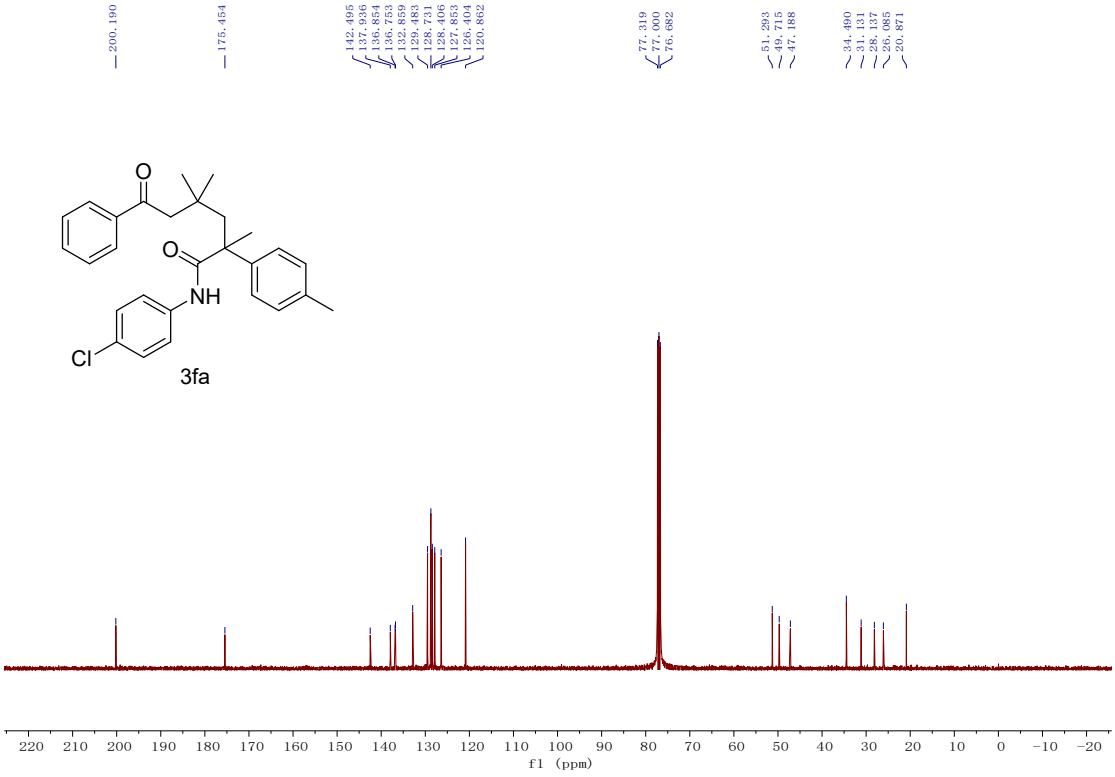


^{19}F NMR (376 MHz, $CDCl_3$)

N-(4-chlorophenyl)-2,4,4-trimethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide

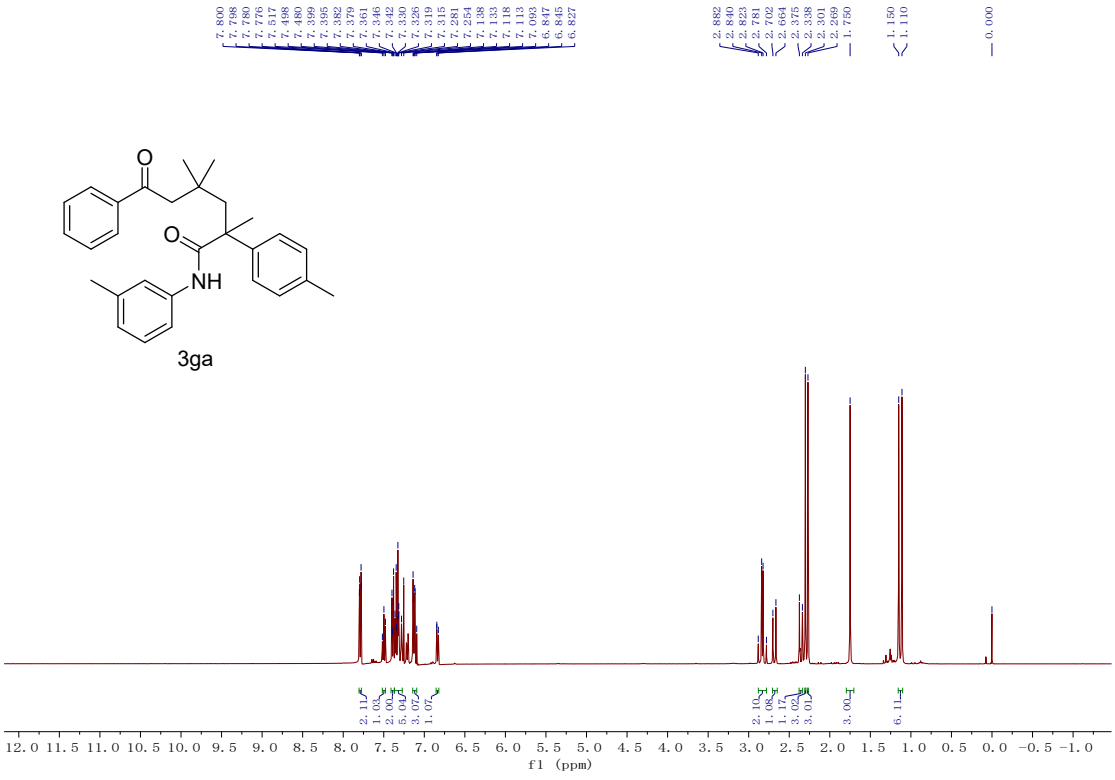


^{1}H NMR (400 MHz, $CDCl_3$)

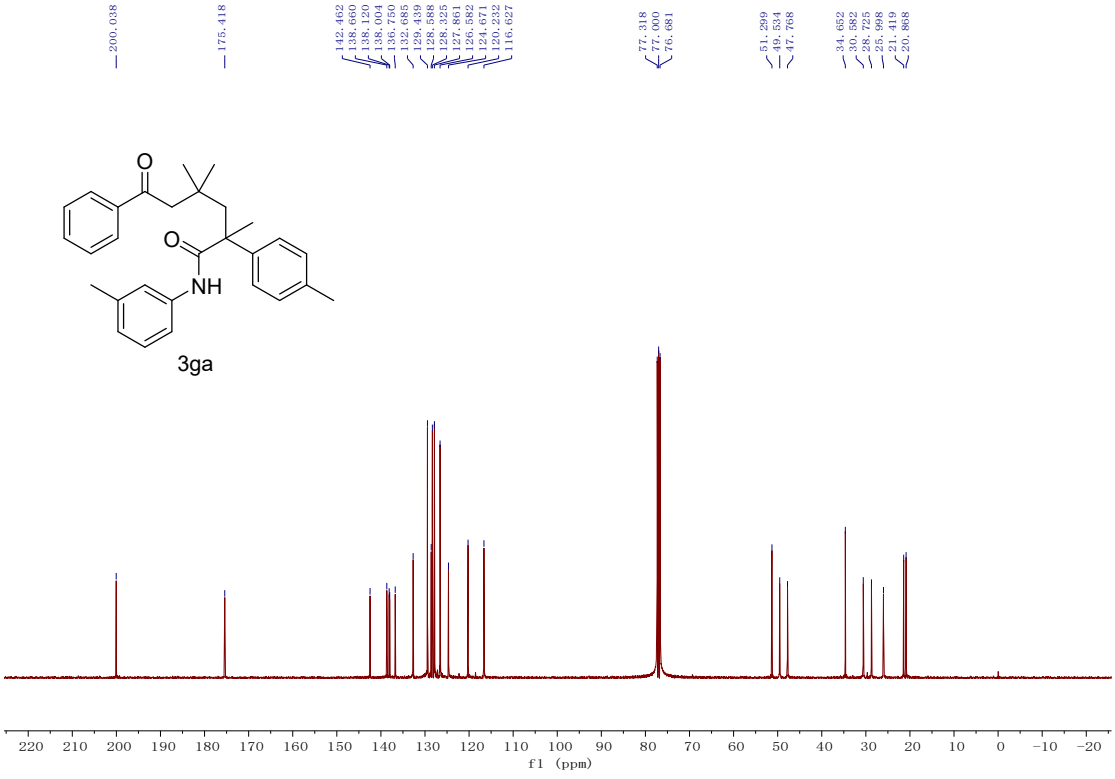


^{13}C NMR (100 MHz, $CDCl_3$)

2,4,4-trimethyl-6-oxo-6-phenyl-N-(m-tolyl)-2-(p-tolyl)hexanamide

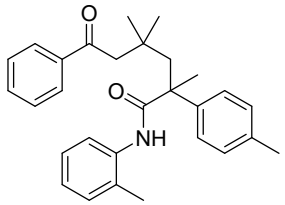


^{1}H NMR (400 MHz, $CDCl_3$)

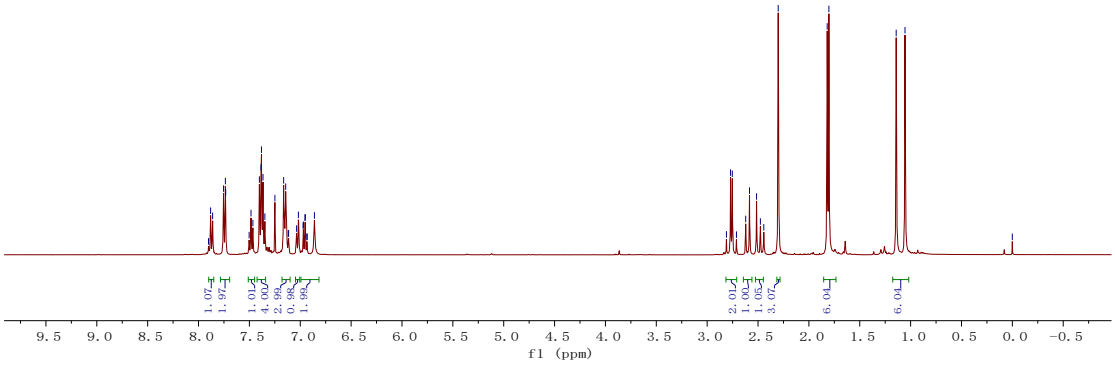


^{13}C NMR (100 MHz, $CDCl_3$)

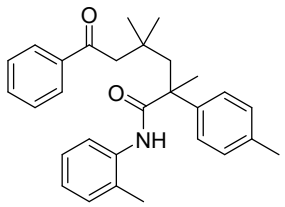
2,4,4-trimethyl-6-oxo-6-phenyl-N-(o-tolyl)-2-(p-tolyl)hexanamide



3ha



^{1}H NMR (400 MHz, $CDCl_3$)



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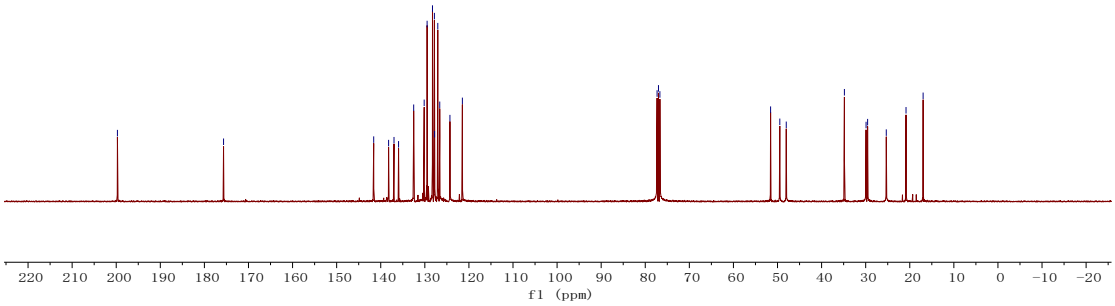
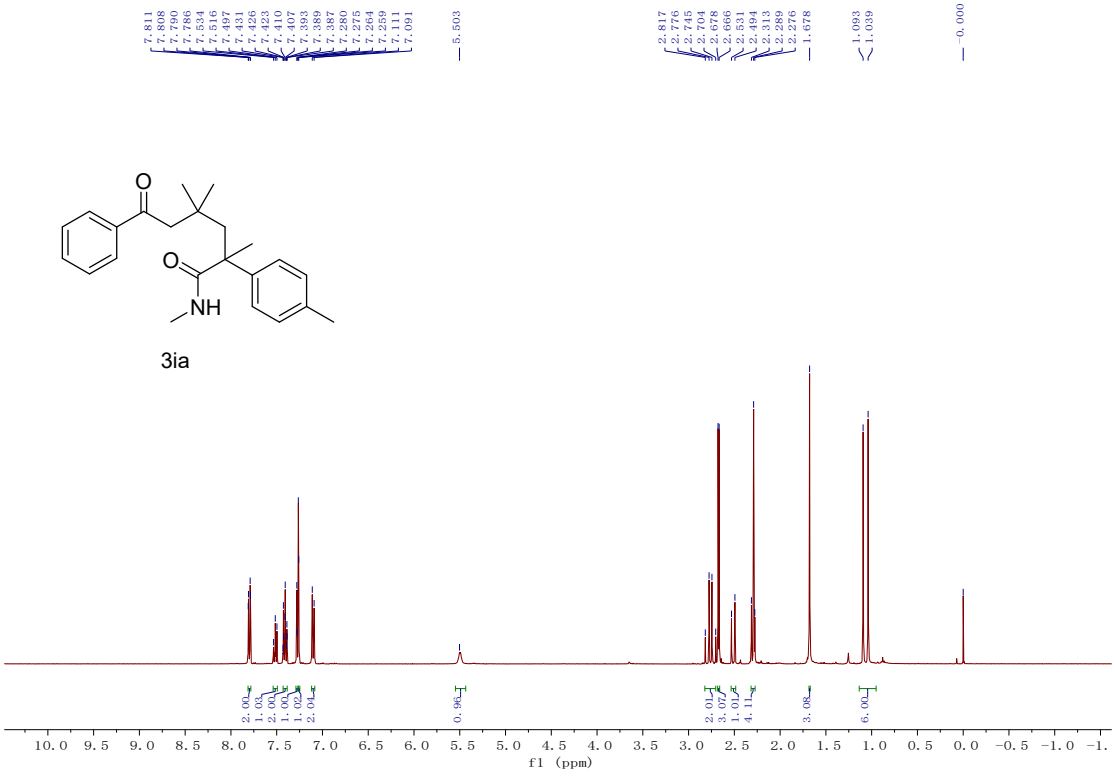
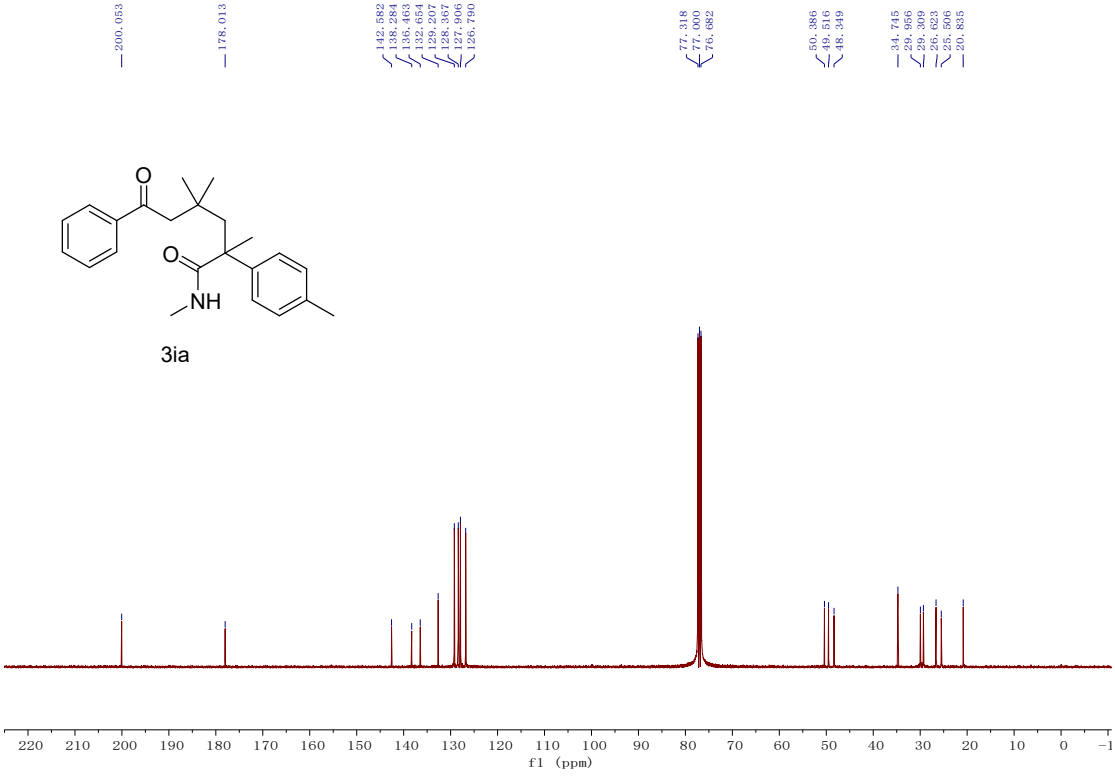


^{13}C NMR (100 MHz, $CDCl_3$)

N,2,4,4-tetramethyl-6-oxo-6-phenyl-2-(p-tolyl)hexanamide

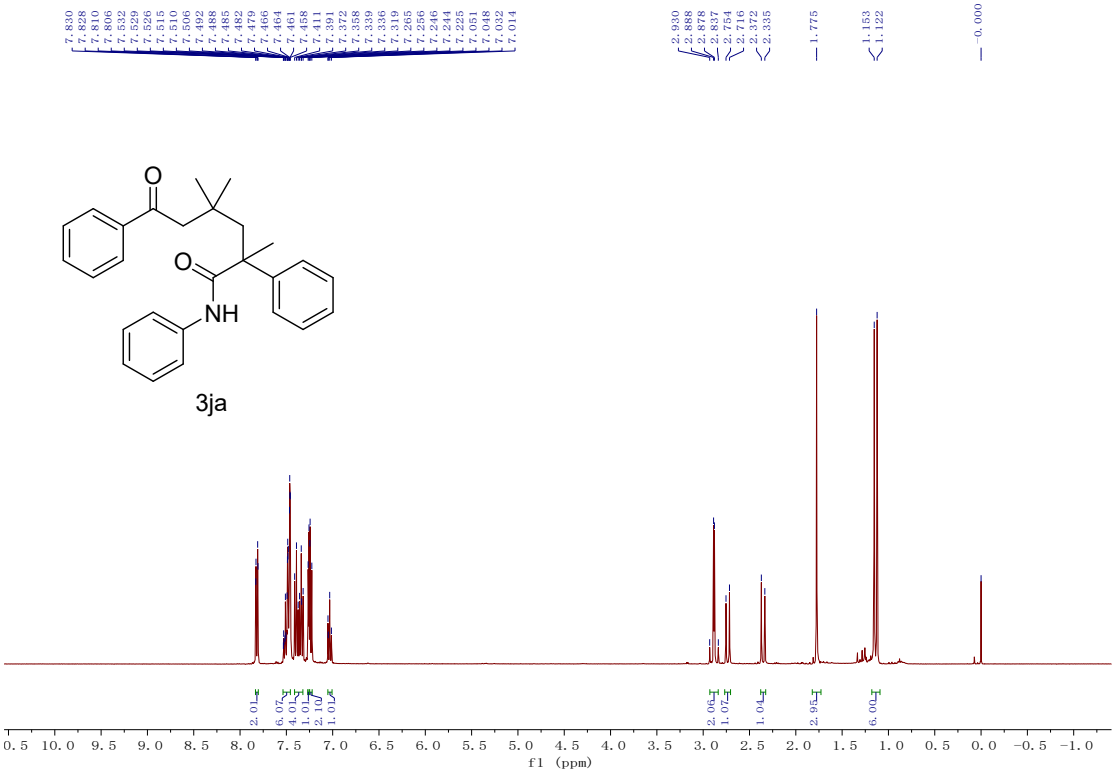


^{1}H NMR (400 MHz, $CDCl_3$)

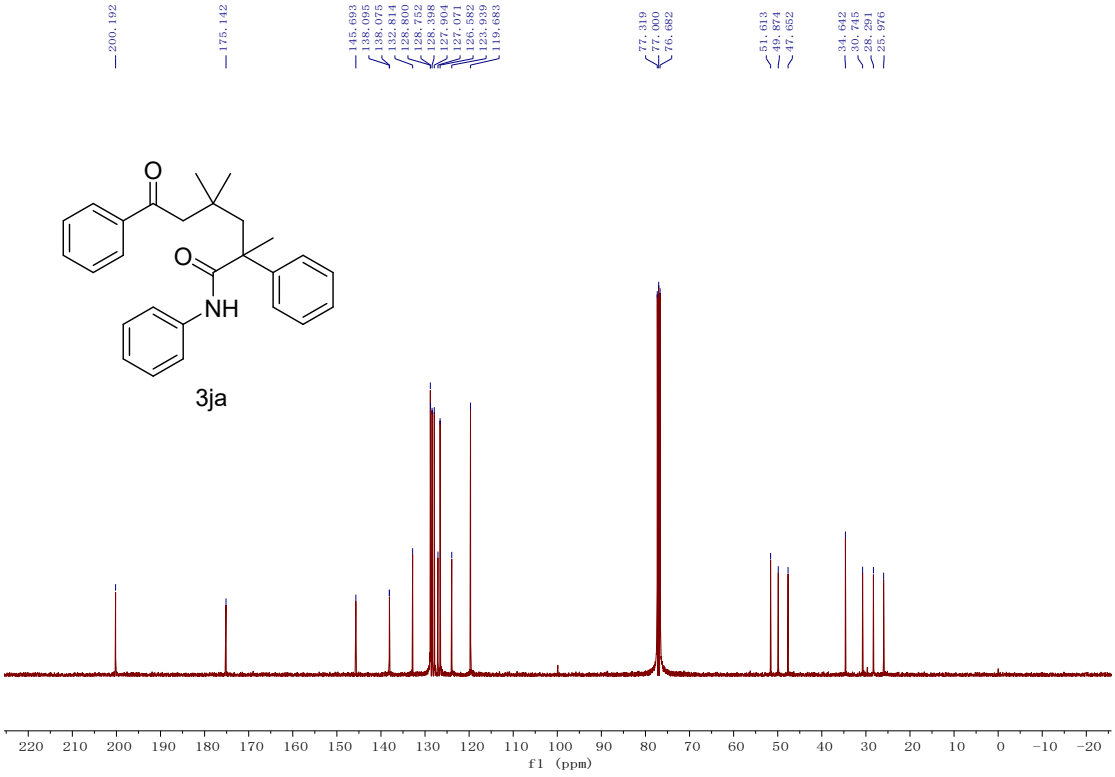


^{13}C NMR (100 MHz, $CDCl_3$)

2,4,4-trimethyl-6-oxo-N,2,6-triphenylhexanamide

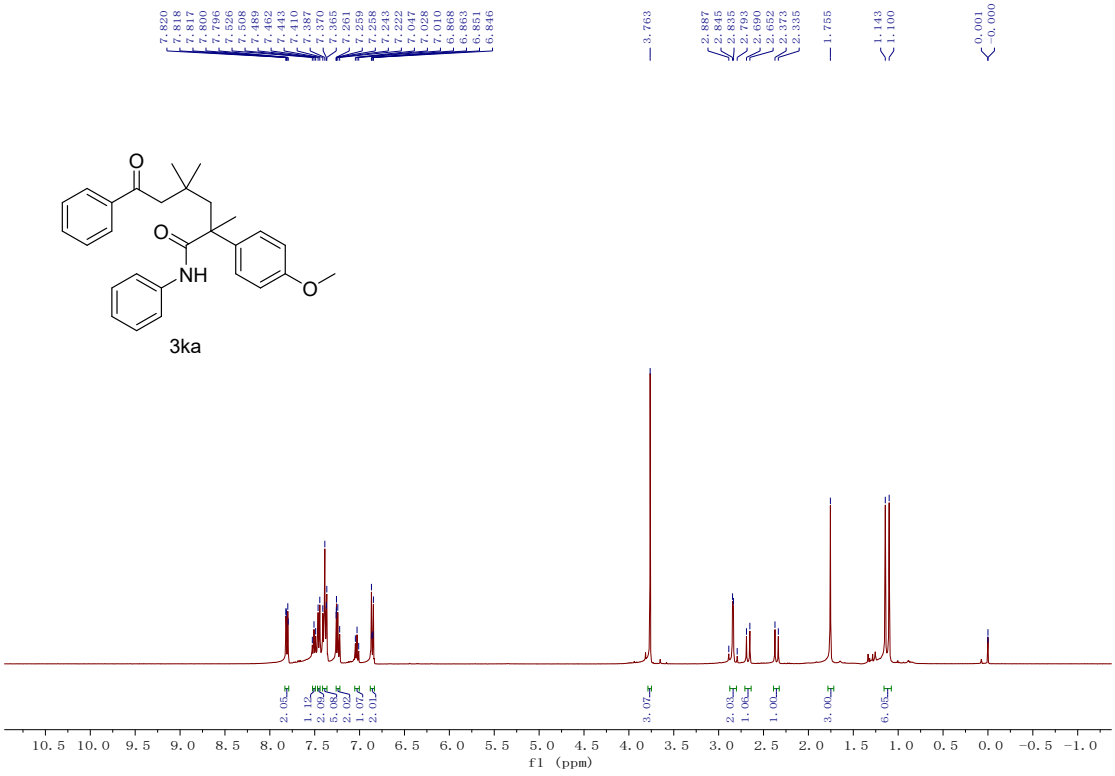


^{1}H NMR (400 MHz, $CDCl_3$)

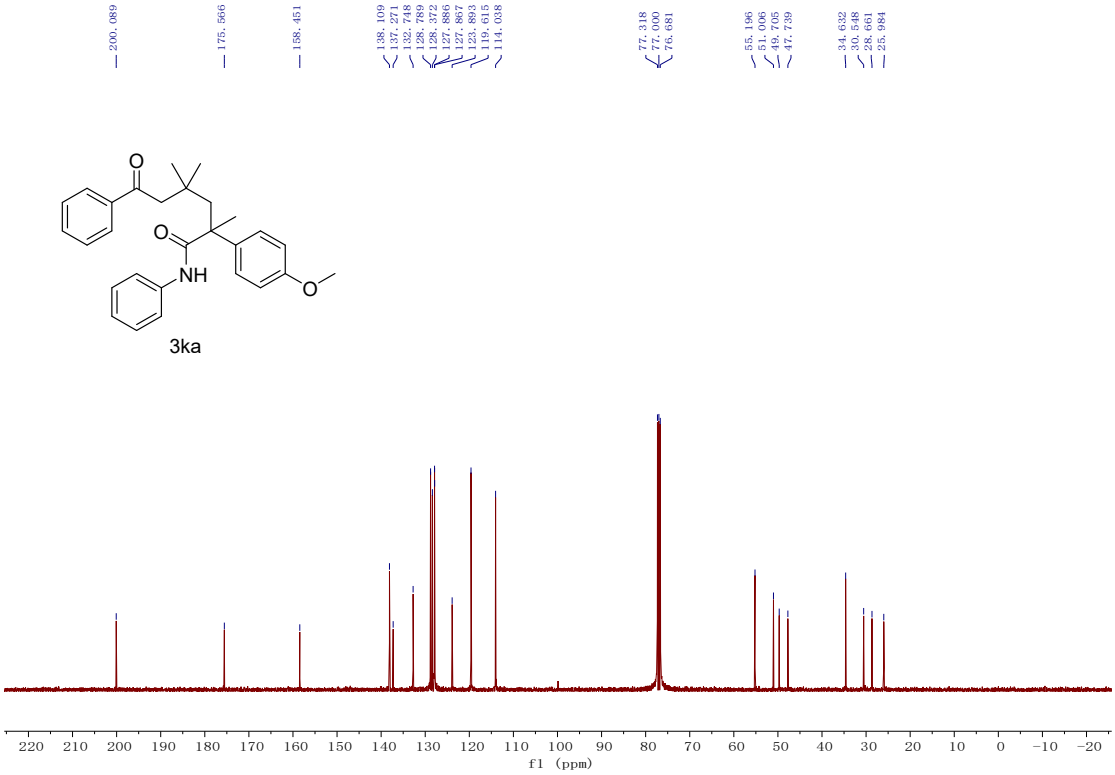


^{13}C NMR (100 MHz, $CDCl_3$)

2-(4-methoxyphenyl)-2,4,4-trimethyl-6-oxo-N,6-diphenylhexanamide

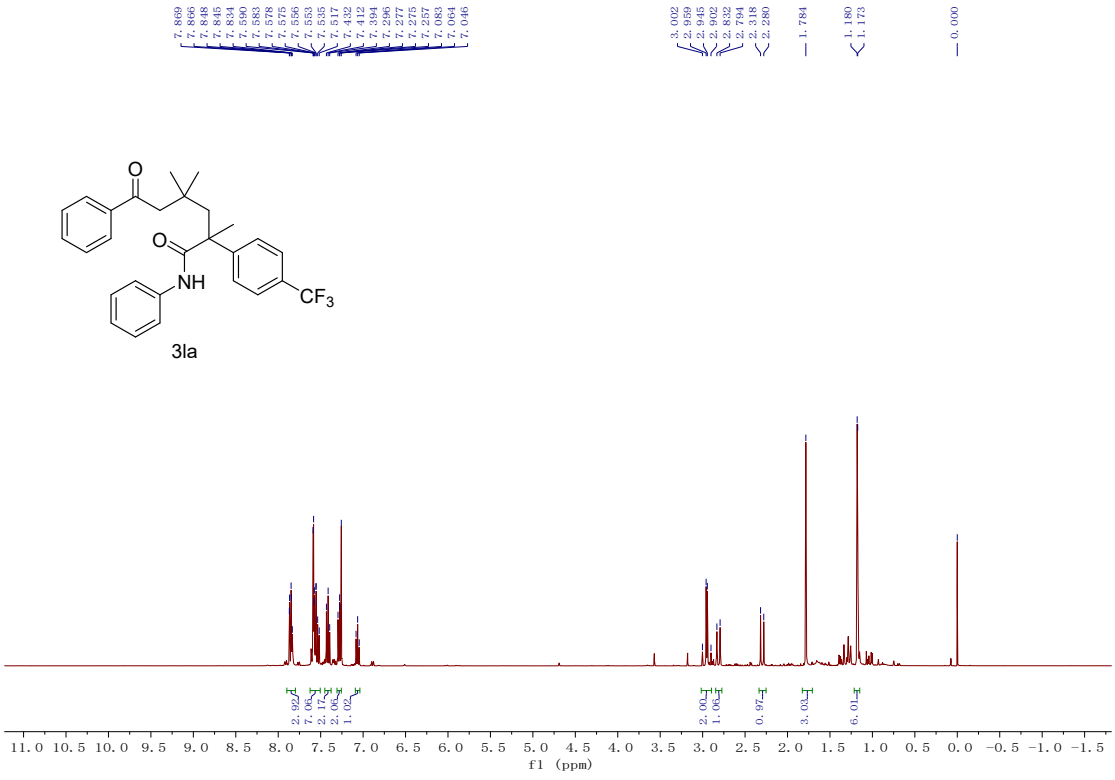


^{1}H NMR (400 MHz, $CDCl_3$)

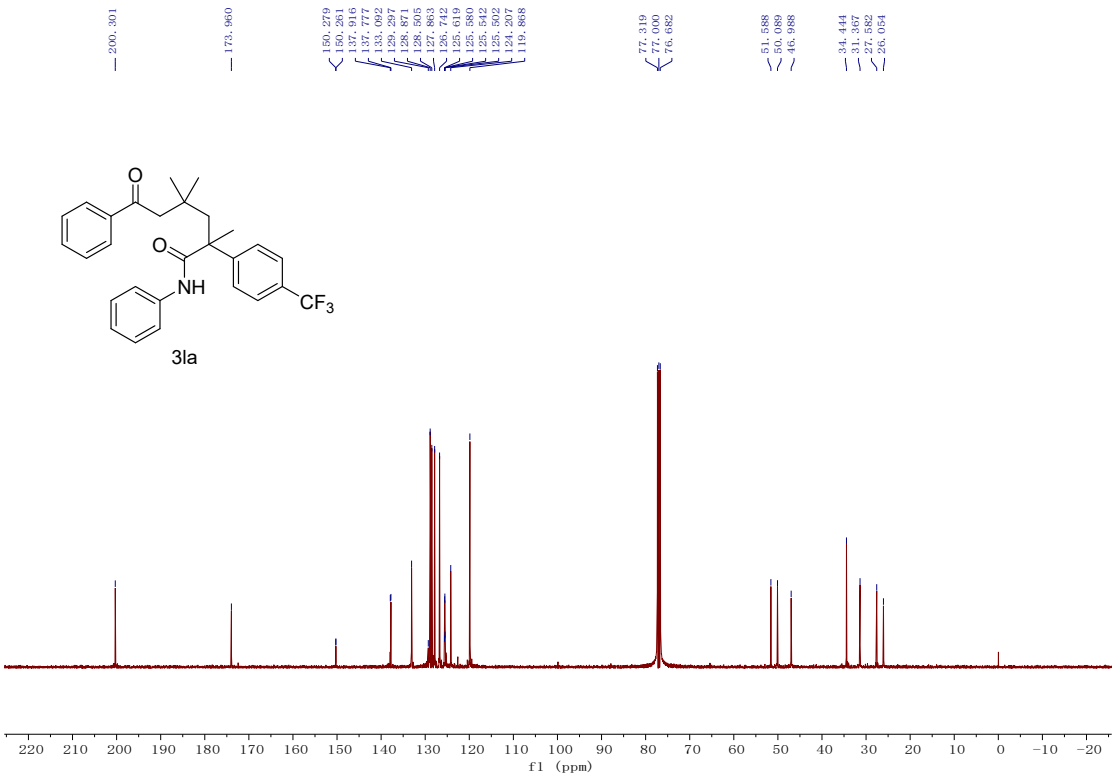


^{13}C NMR (100 MHz, $CDCl_3$)

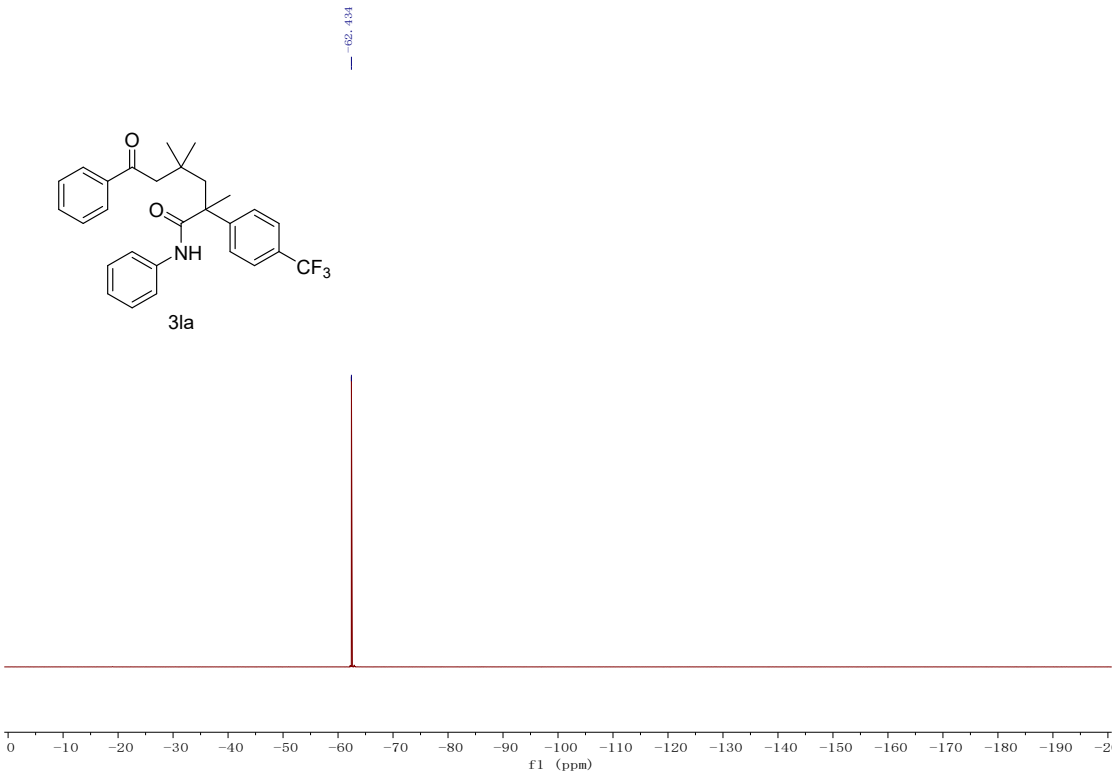
2,4,4-trimethyl-6-oxo-N,6-diphenyl-2-(4-(trifluoromethyl)phenyl)hexanamide



^{1}H NMR (400 MHz, $CDCl_3$)

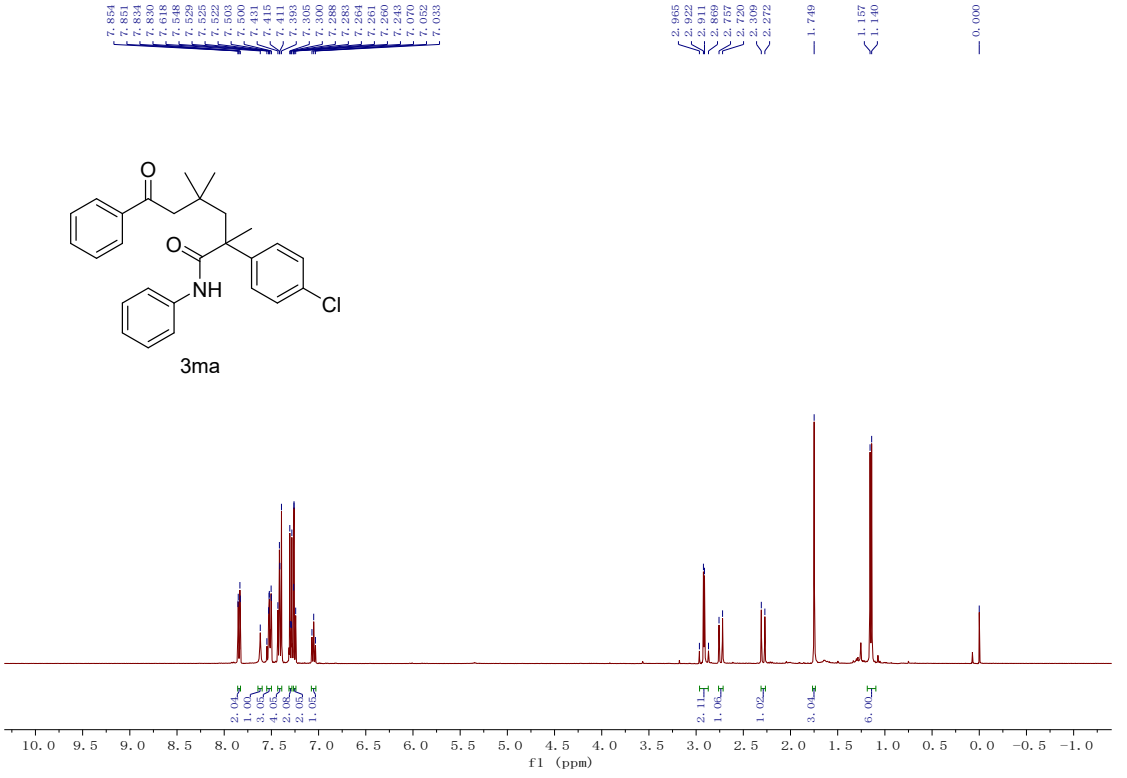


^{13}C NMR (100 MHz, $CDCl_3$)

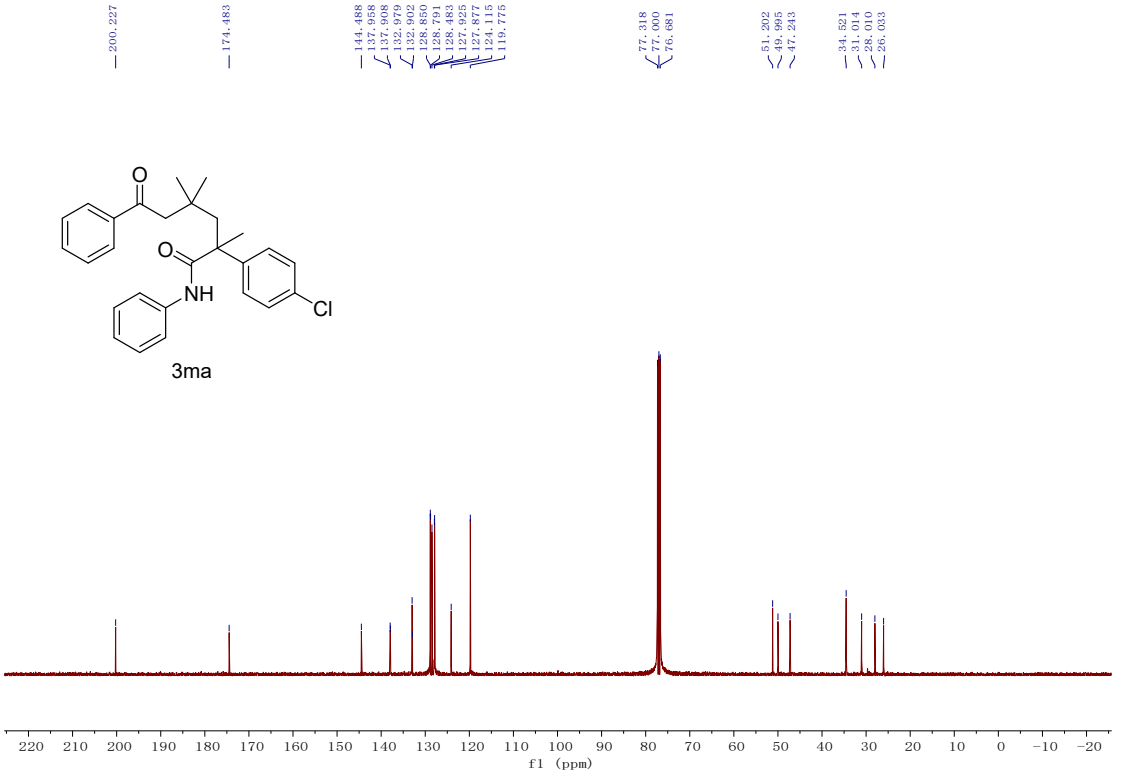


^{19}F NMR (376 MHz, $CDCl_3$)

2-(4-chlorophenyl)-2,4,4-trimethyl-6-oxo-N,6-diphenylhexanamide

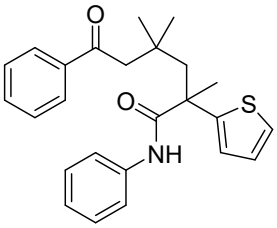


^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

2,4,4-trimethyl-6-oxo-N,6-diphenyl-2-(thiophen-2-yl)hexanamide



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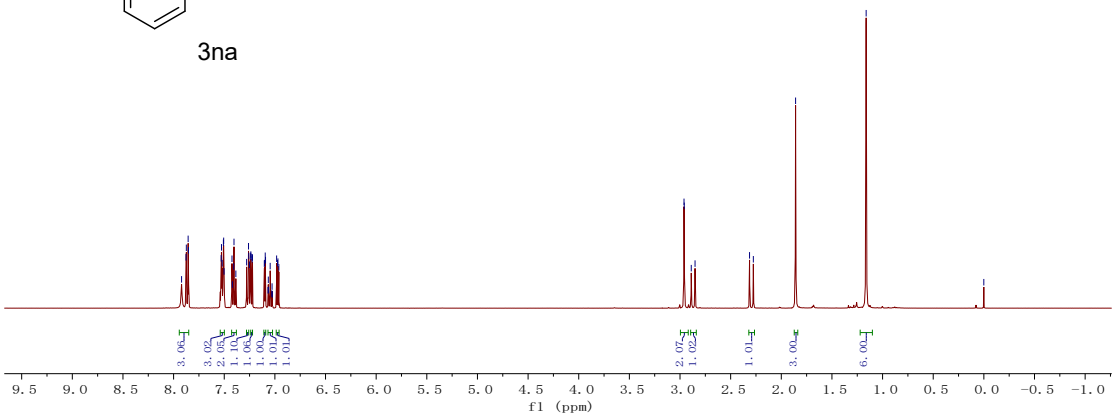
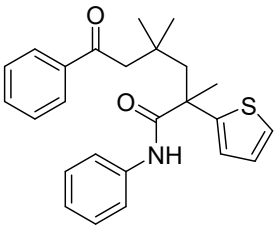


^{1}H NMR (400 MHz, $CDCl_3$)



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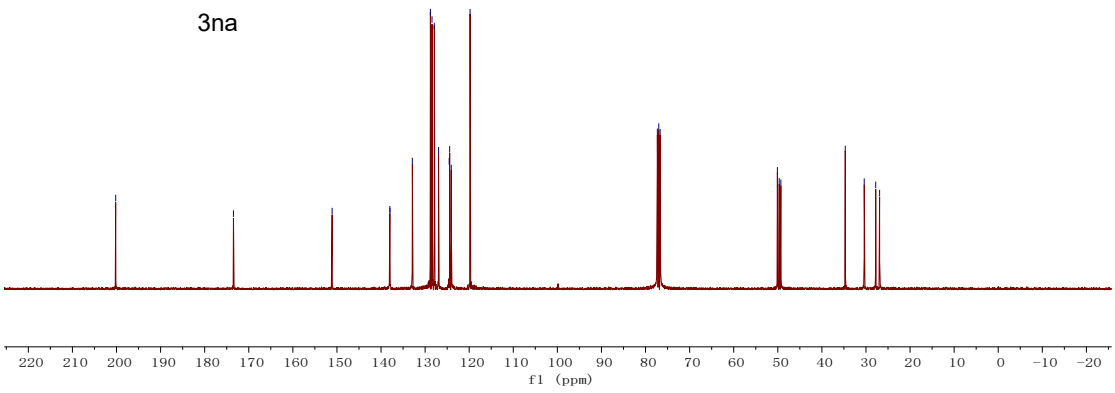
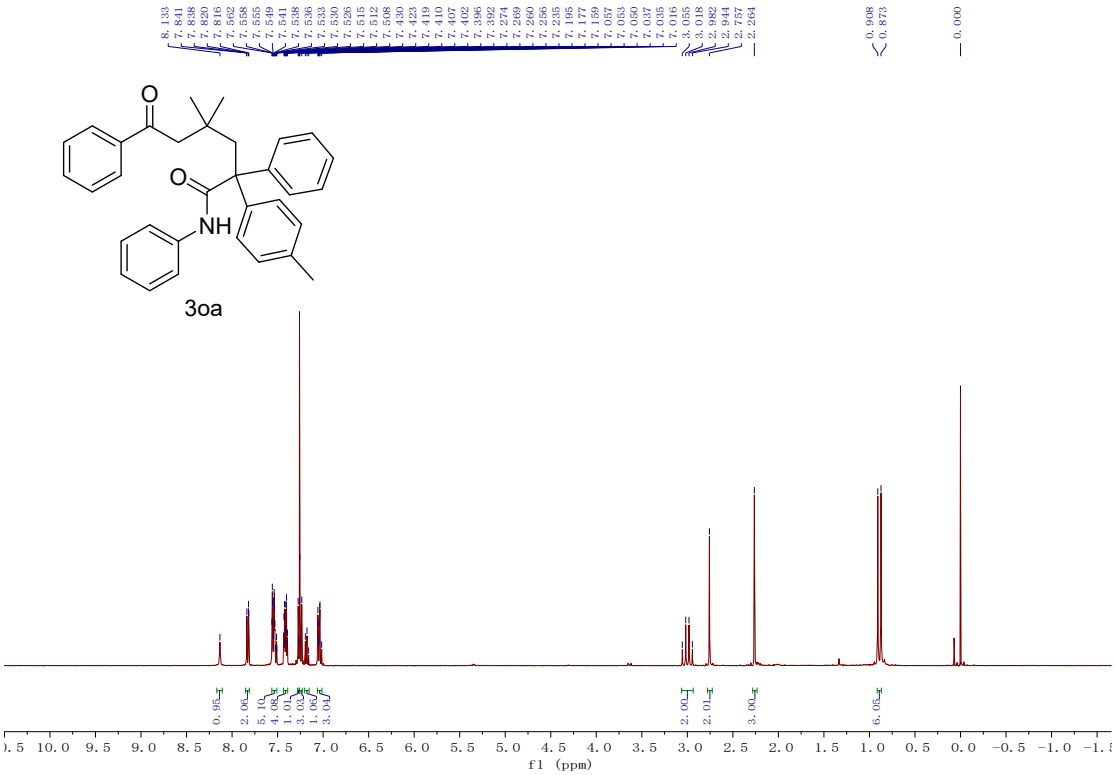
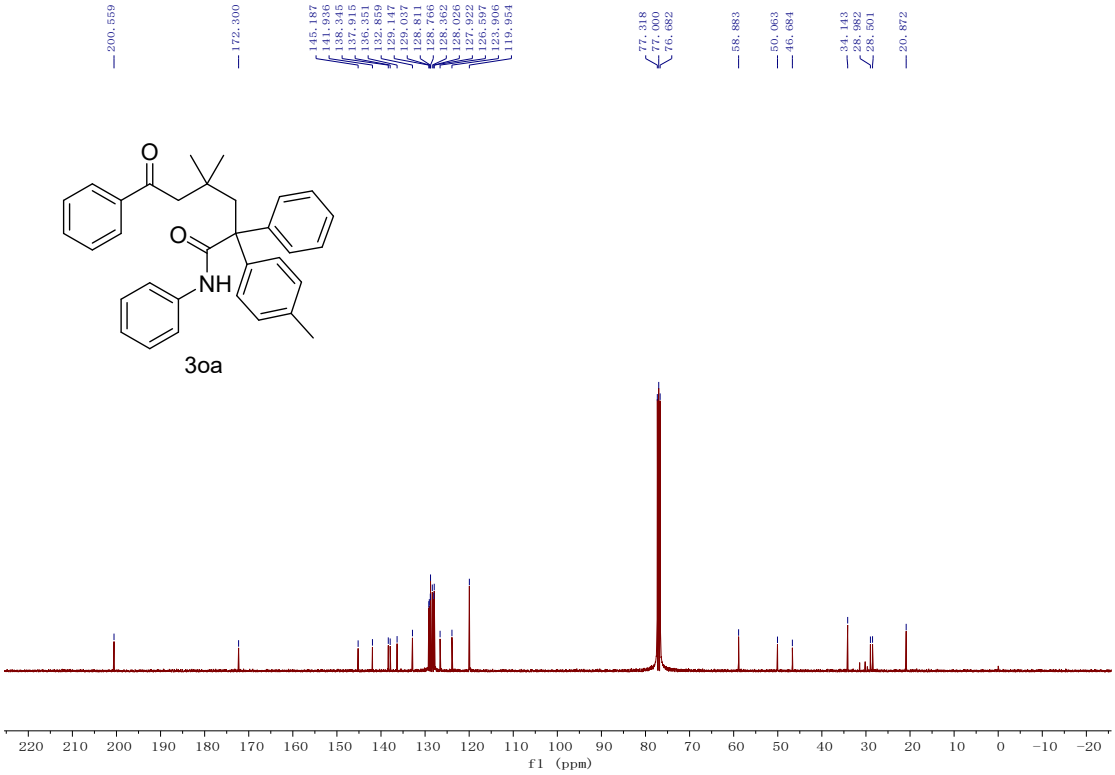


^{13}C NMR (100 MHz, $CDCl_3$)

4,4-dimethyl-6-oxo-N,2,6-triphenyl-2-(p-tolyl)hexanamide

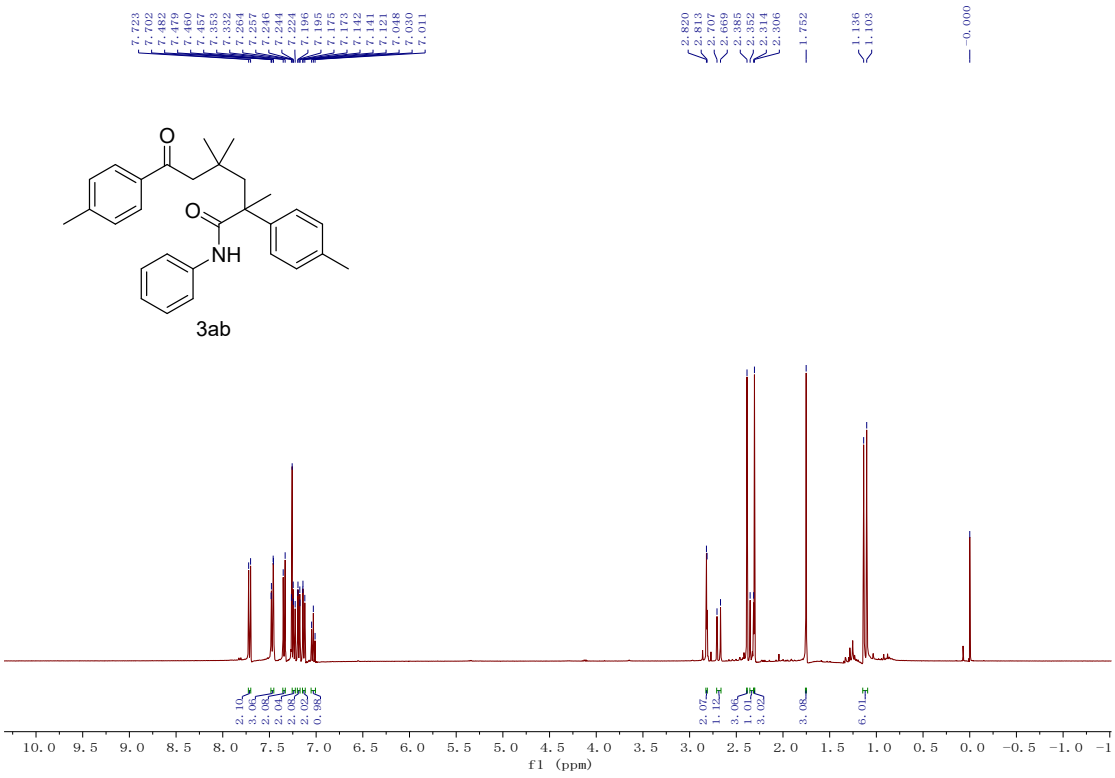


^{1}H NMR (400 MHz, $CDCl_3$)

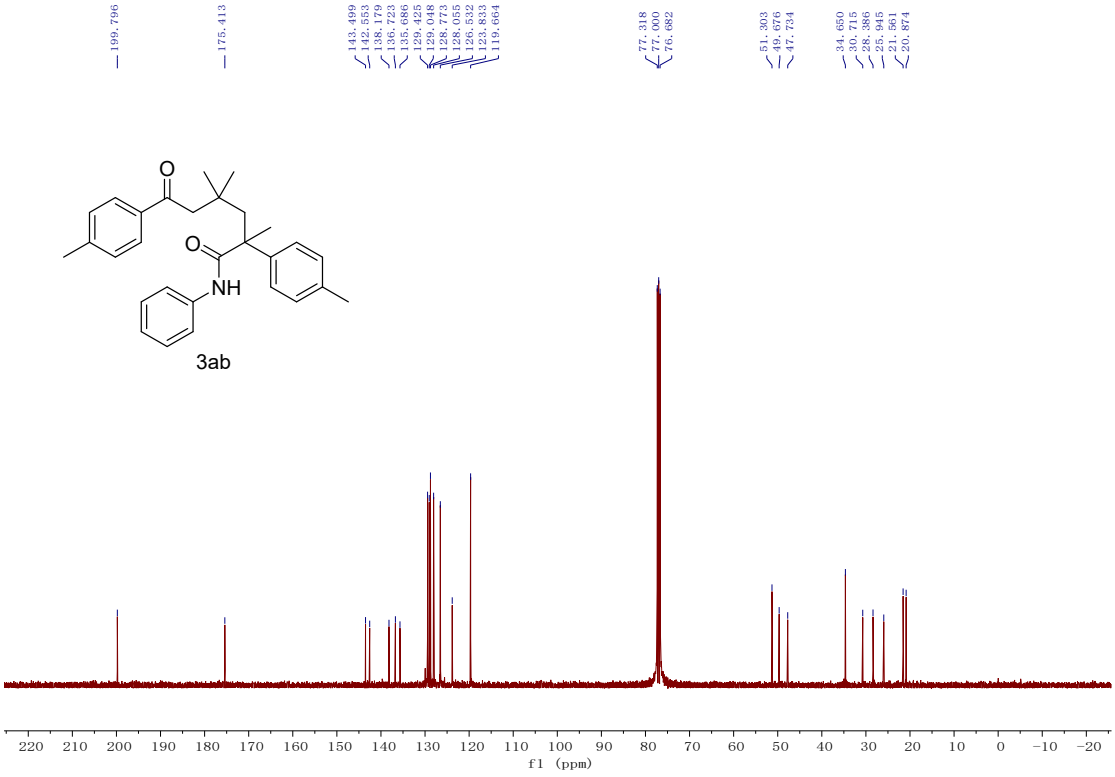


^{13}C NMR (100 MHz, $CDCl_3$)

2,4,4-trimethyl-6-oxo-N-phenyl-2,6-di-p-tolylhexanamide

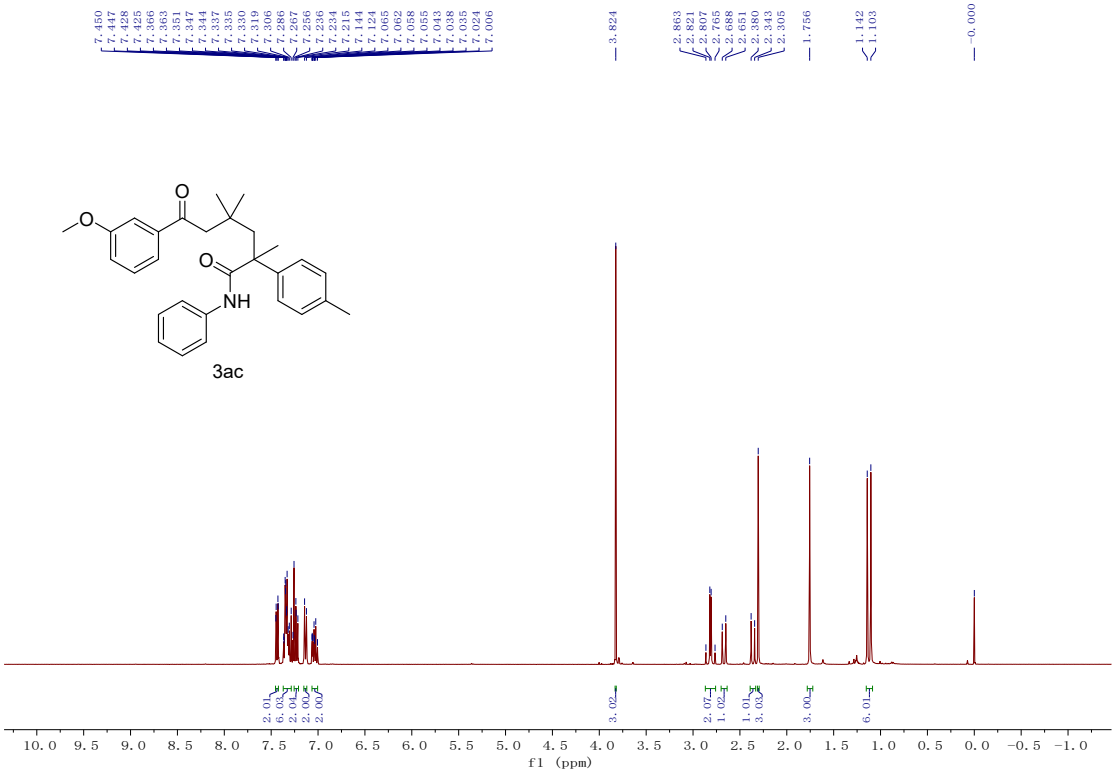


^{1}H NMR (400 MHz, $CDCl_3$)

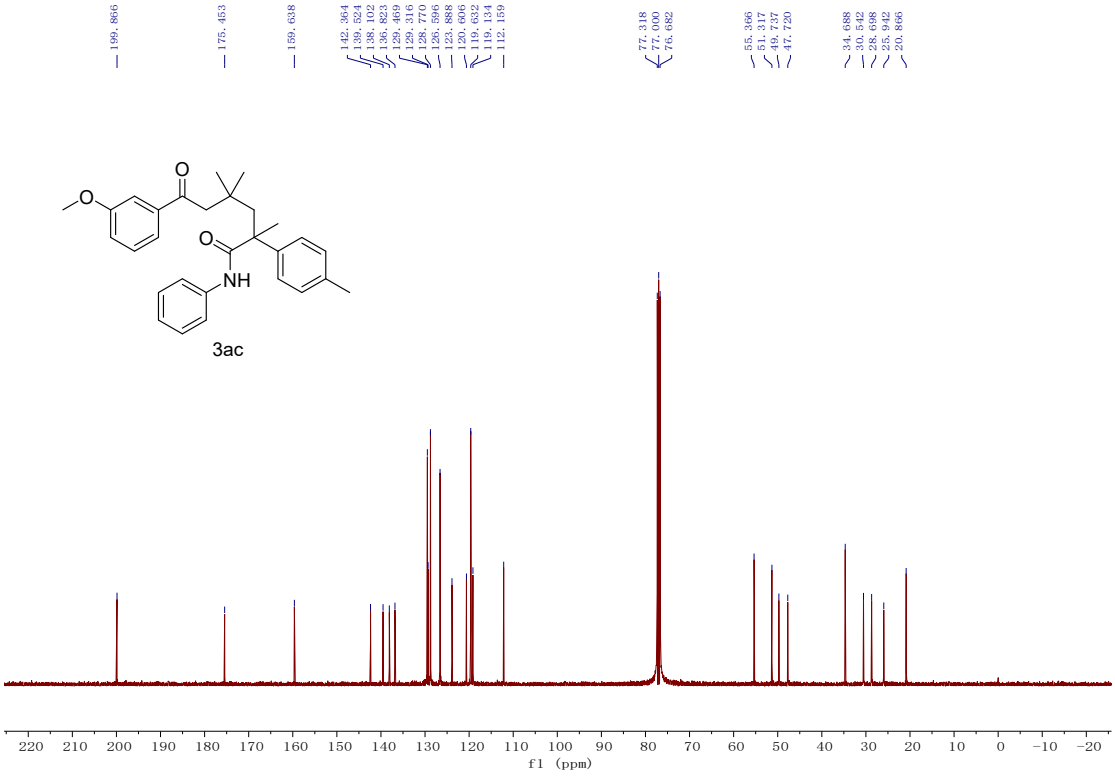


^{13}C NMR (100 MHz, $CDCl_3$)

6-(3-methoxyphenyl)-2,4,4-trimethyl-6-oxo-N-phenyl-2-(p-tolyl)hexanamide

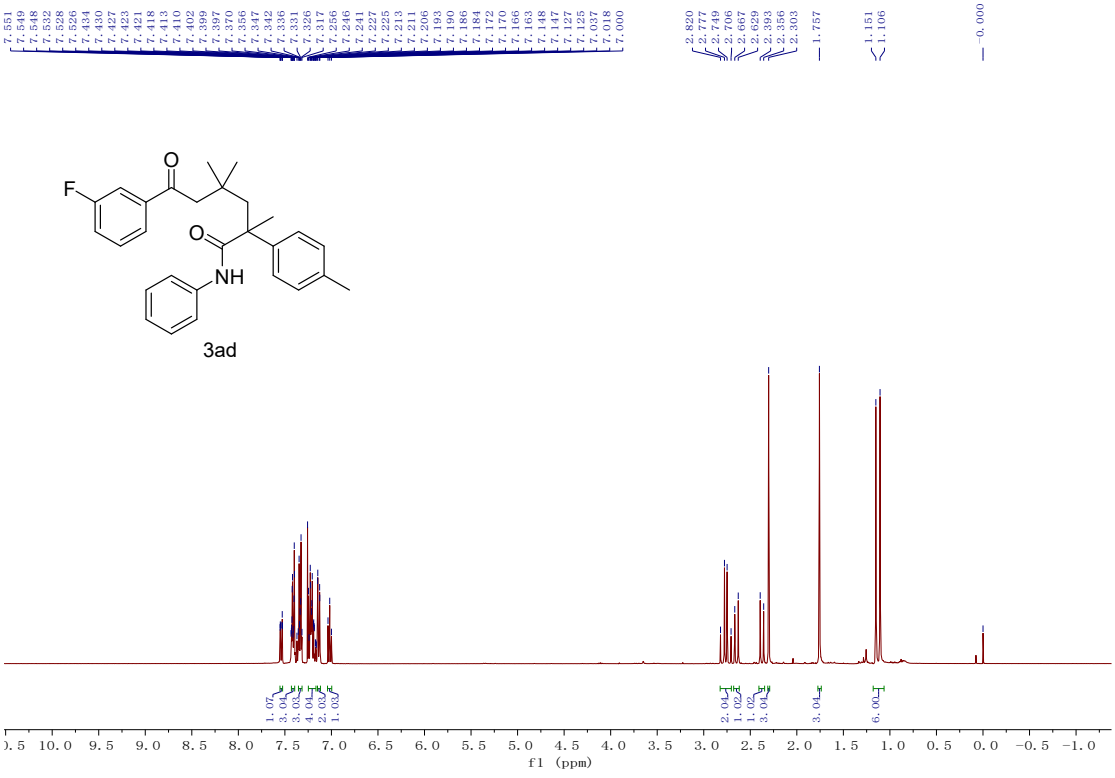


^{1}H NMR (400 MHz, $CDCl_3$)

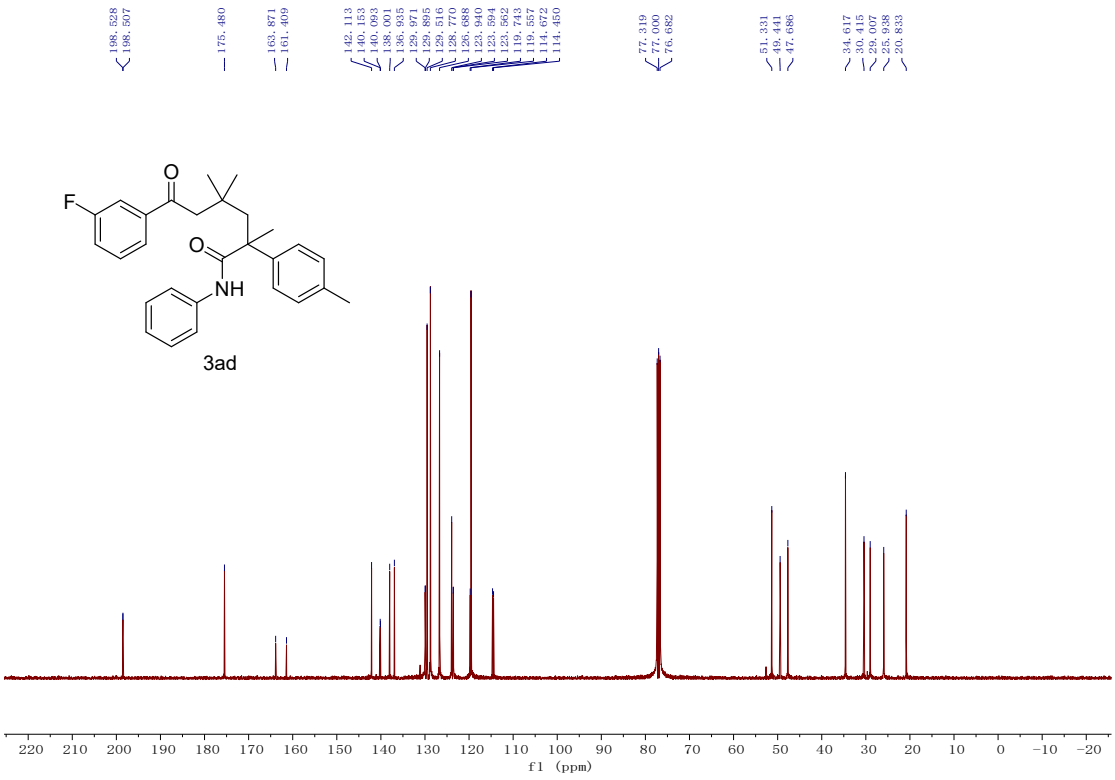


^{13}C NMR (100 MHz, $CDCl_3$)

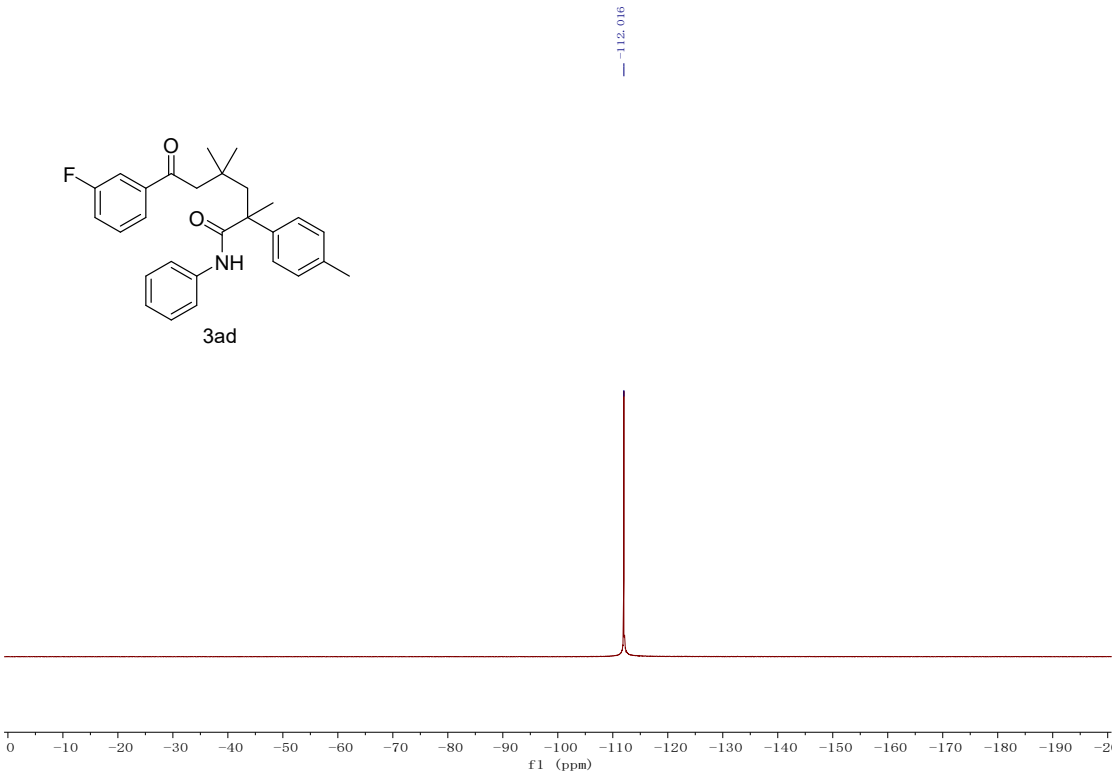
6-(3-fluorophenyl)-2,4,4-trimethyl-6-oxo-N-phenyl-2-(p-tolyl)hexanamide



^{1}H NMR (400 MHz, $CDCl_3$)

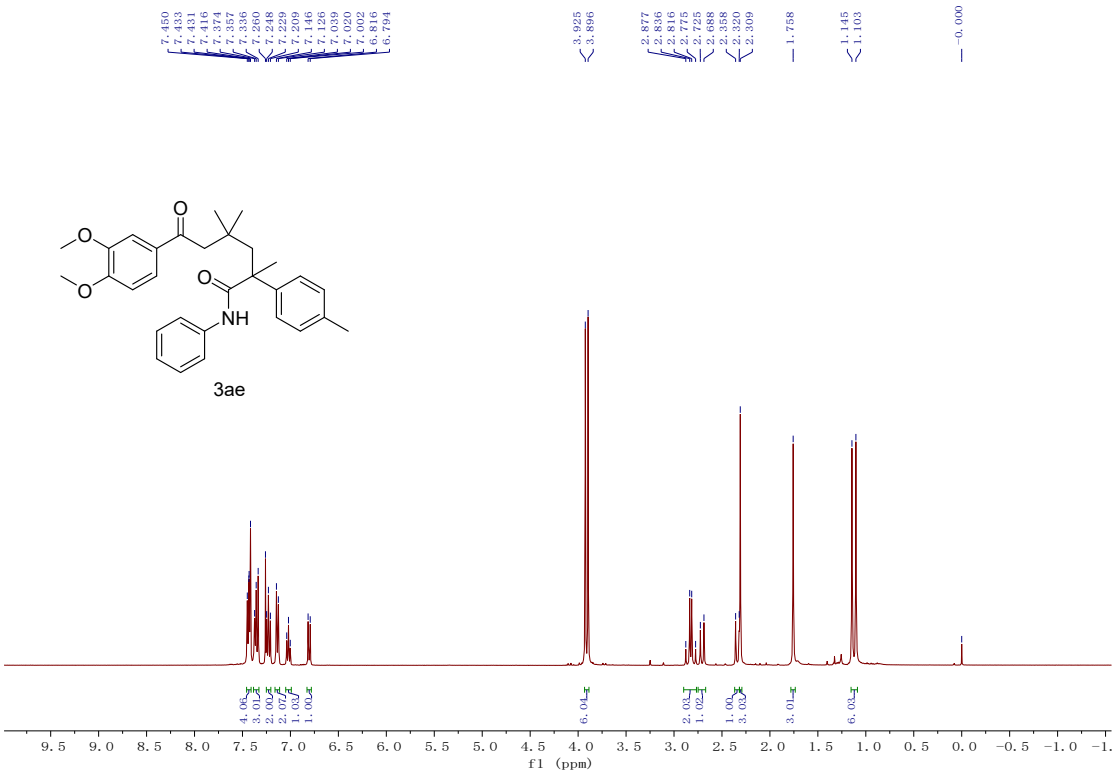


^{13}C NMR (100 MHz, $CDCl_3$)

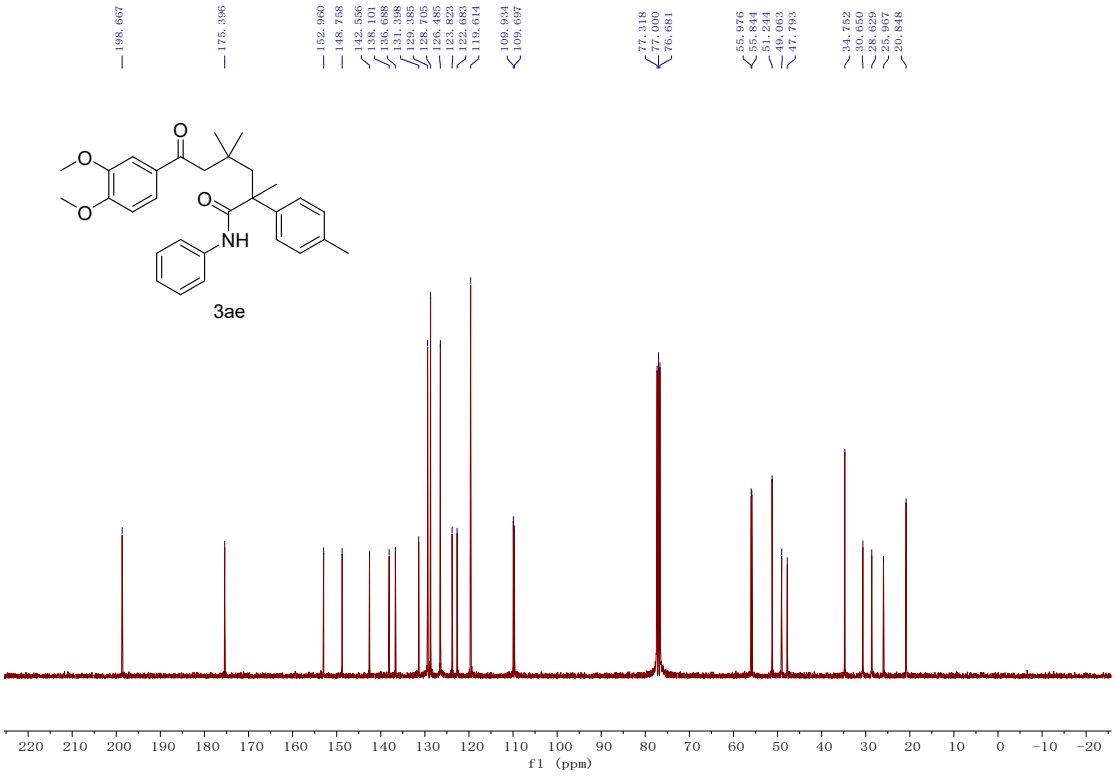


^{19}F NMR (376 MHz, $CDCl_3$)

6-(3,4-dimethoxyphenyl)-2,4,4-trimethyl-6-oxo-N-phenyl-2-(p-tolyl)hexanamide

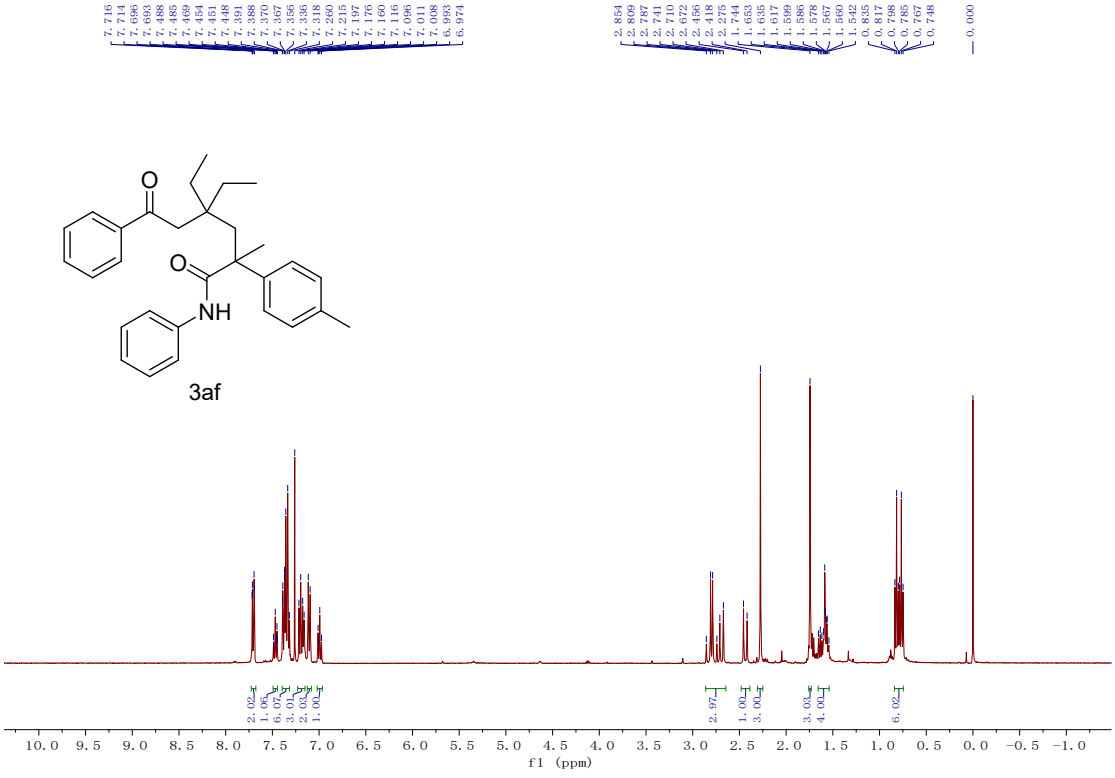


^{1}H NMR (400 MHz, $CDCl_3$)

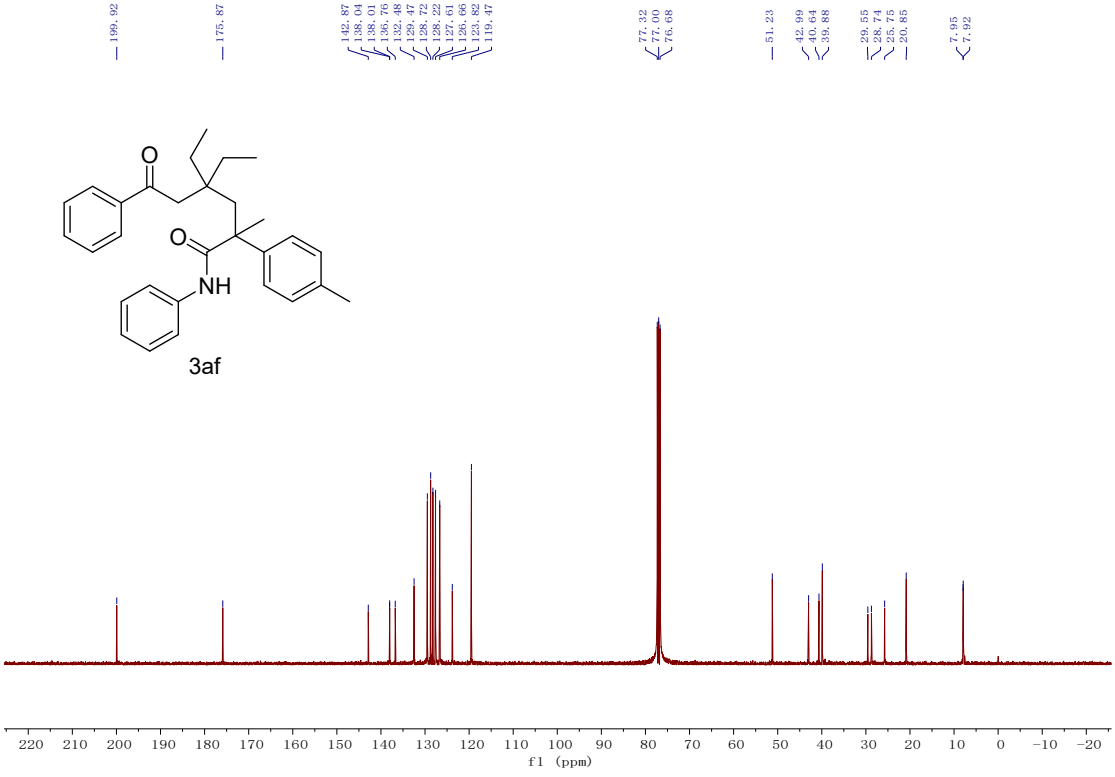


^{13}C NMR (100 MHz, $CDCl_3$)

4,4-diethyl-2-methyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide

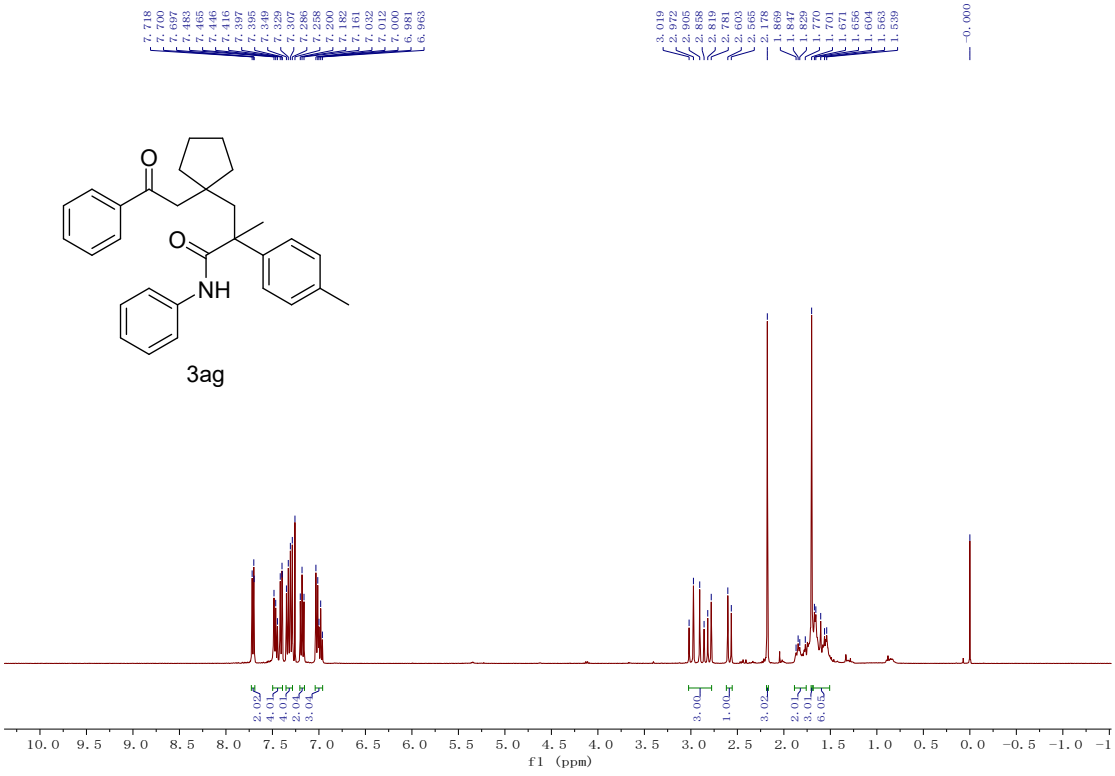


^{1}H NMR (400 MHz, $CDCl_3$)

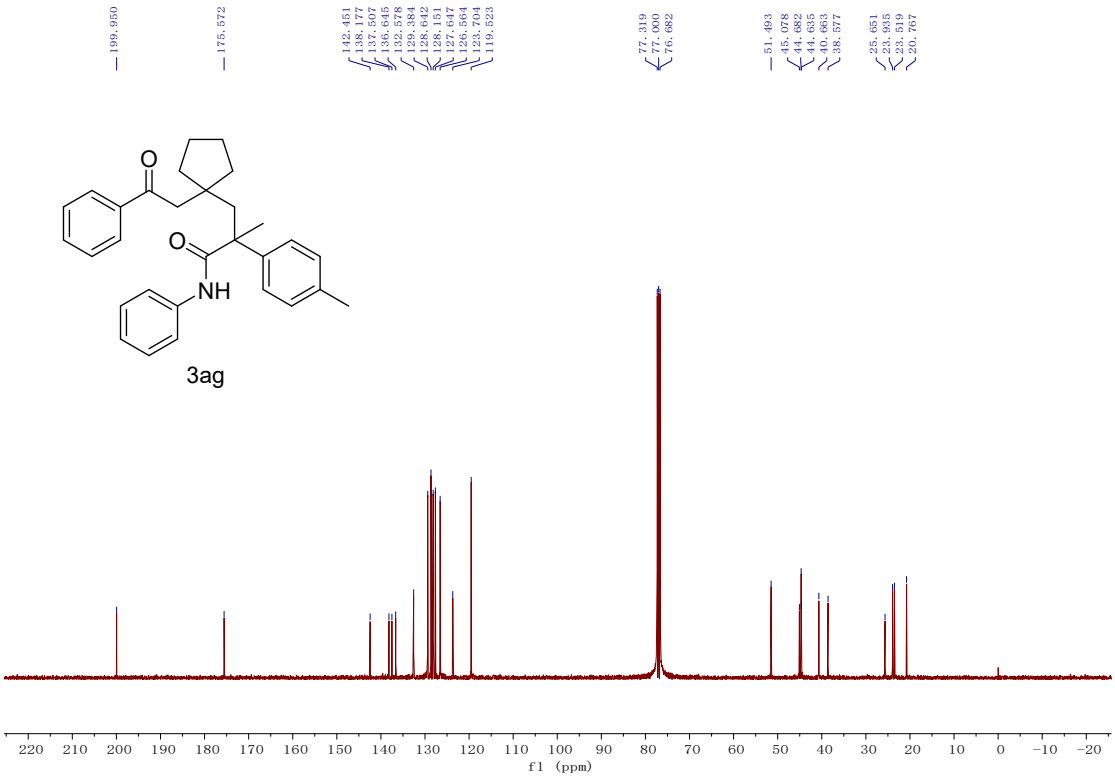


^{13}C NMR (100 MHz, $CDCl_3$)

2-methyl-3-(1-(2-oxo-2-phenylethyl)cyclopentyl)-N-phenyl-2-(p-tolyl) propanamide

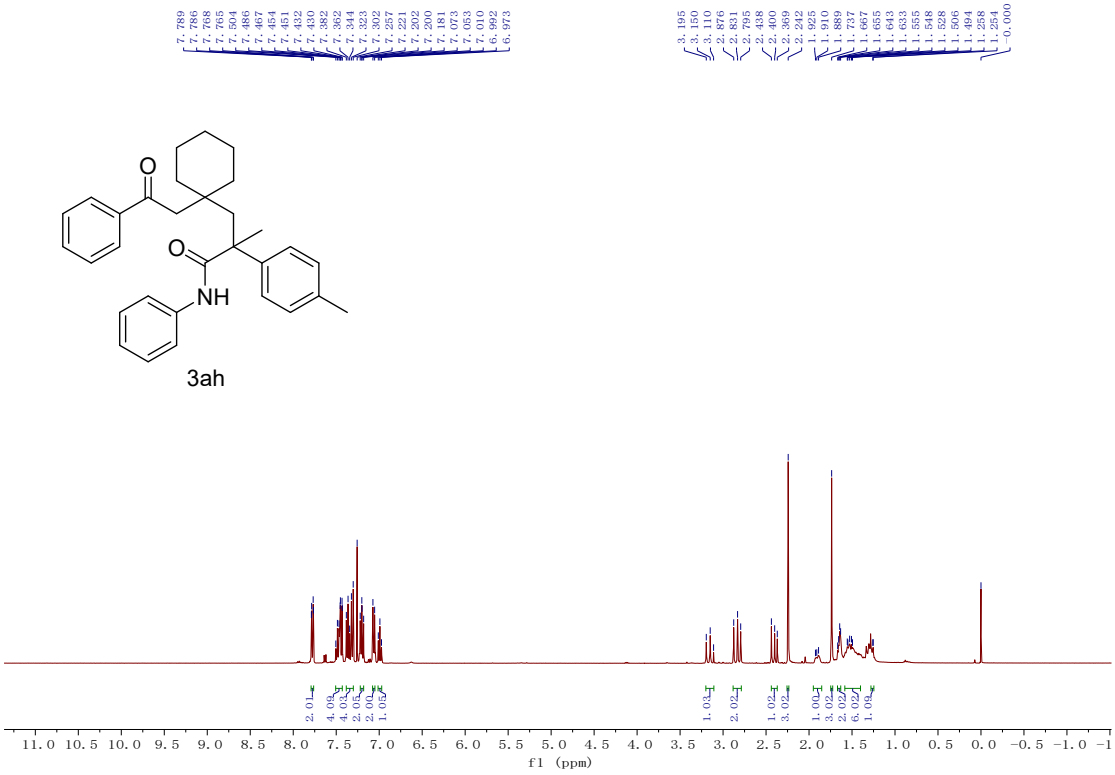


^{1}H NMR (400 MHz, $CDCl_3$)

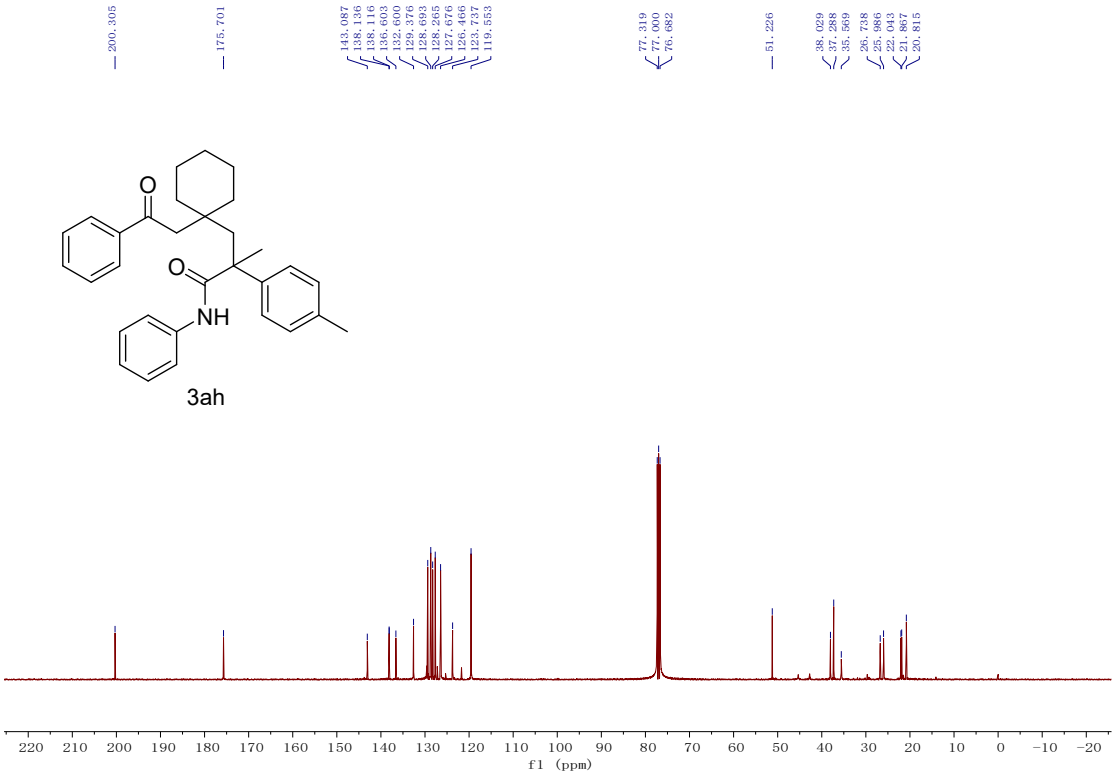


^{13}C NMR (100 MHz, $CDCl_3$)

2-methyl-3-(1-(2-oxo-2-phenylethyl)cyclohexyl)-N-phenyl-2-(p-tolyl) propanamide

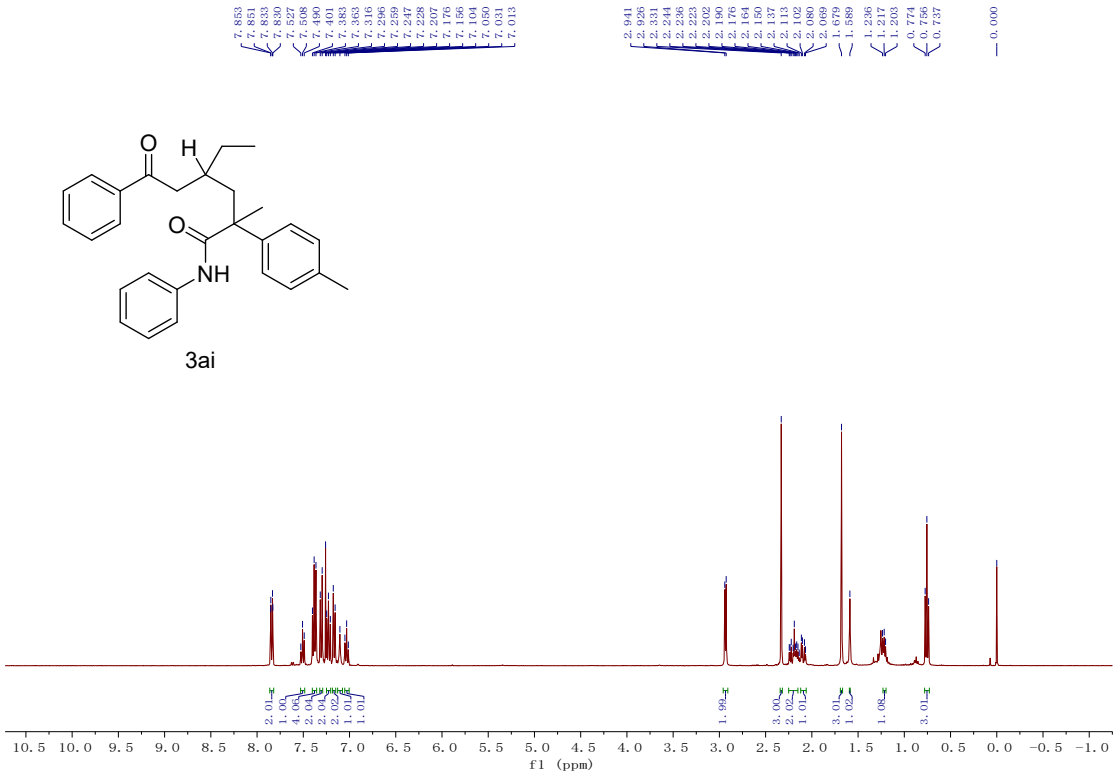


^{1}H NMR (400 MHz, $CDCl_3$)

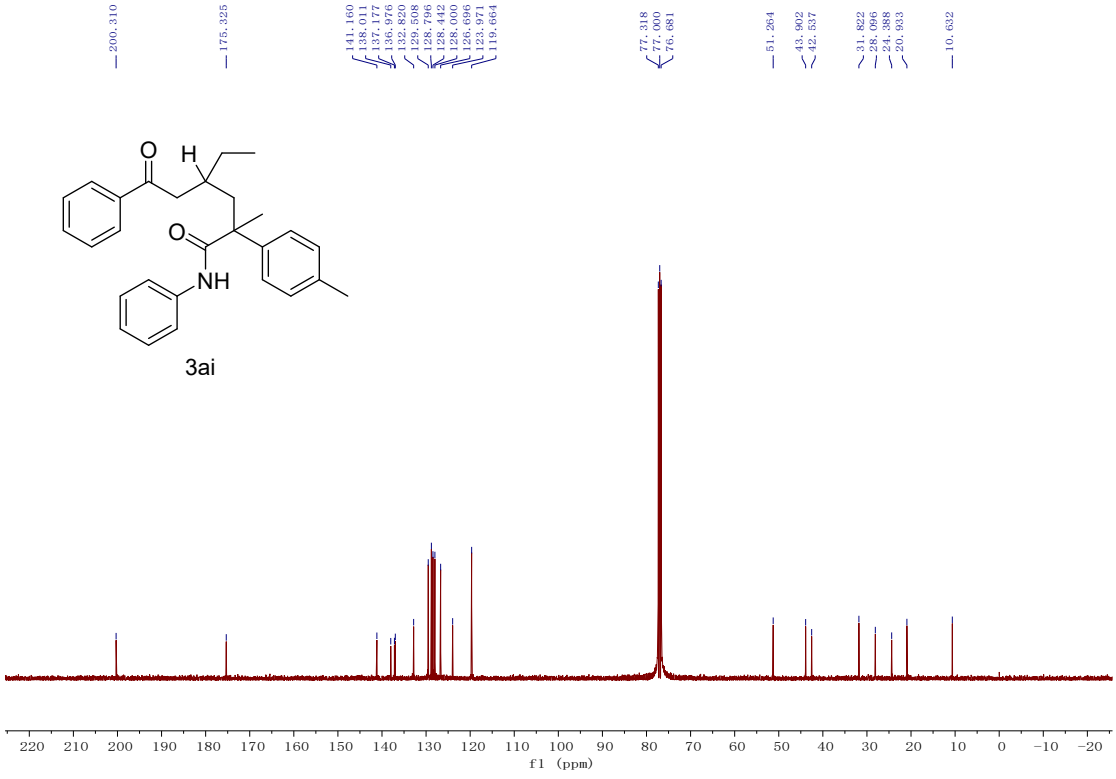


^{13}C NMR (100 MHz, $CDCl_3$)

4-ethyl-2-methyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide

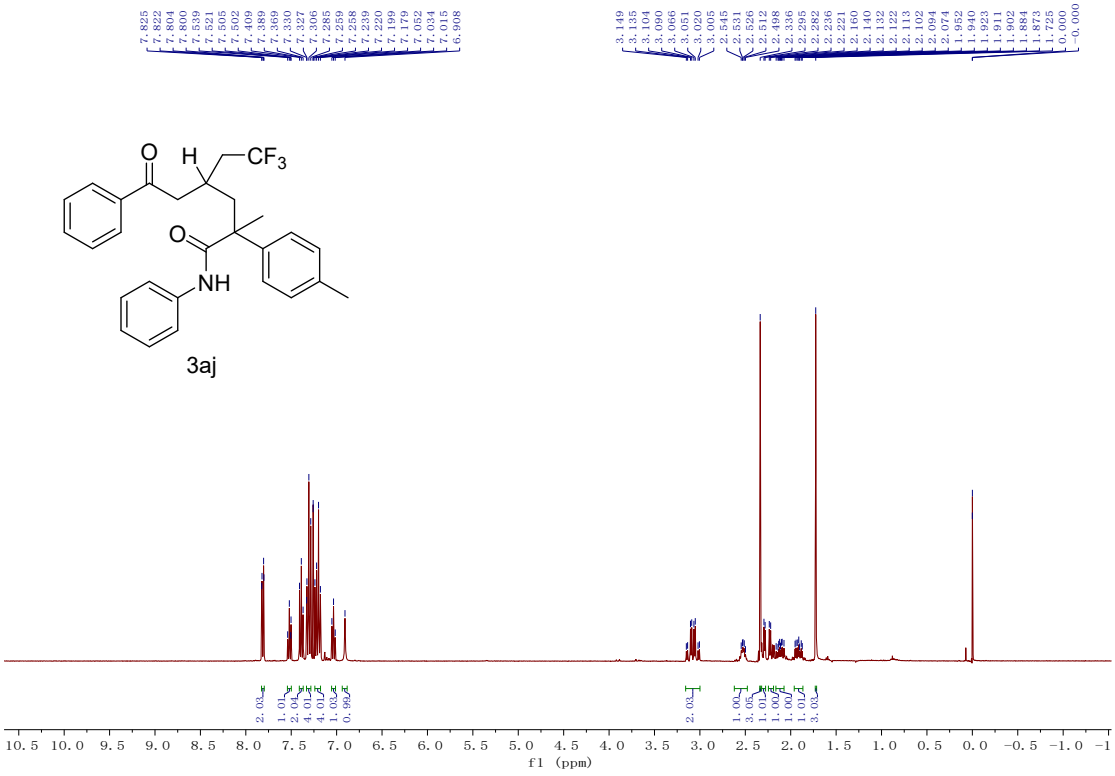


^{1}H NMR (400 MHz, $CDCl_3$)

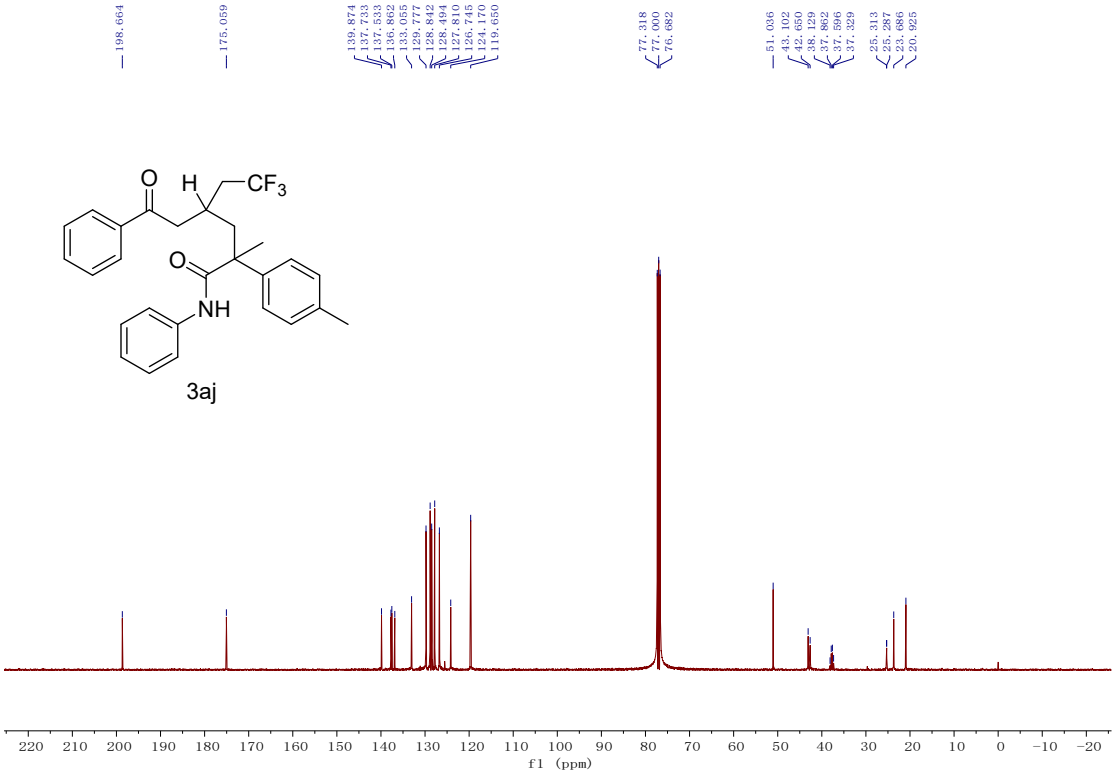


^{13}C NMR (100 MHz, $CDCl_3$)

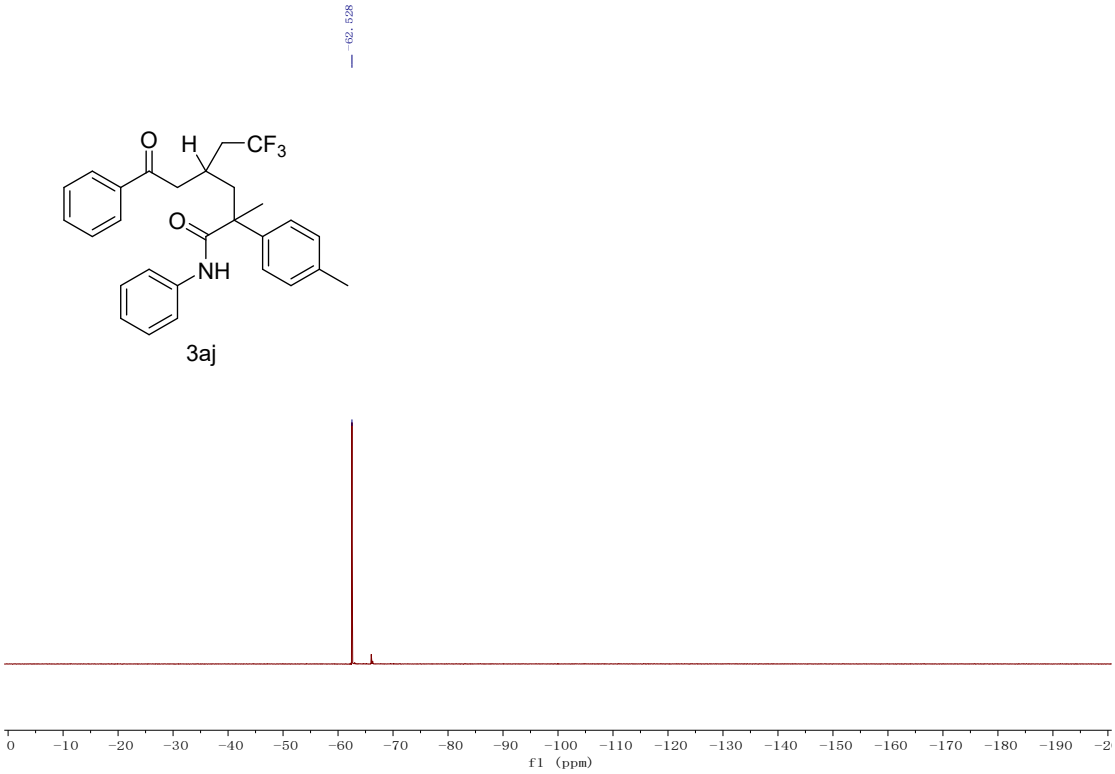
6,6,6-trifluoro-2-methyl-4-(2-oxo-2-phenylethyl)-N-phenyl-2-(p-tolyl)hexanamide



^{1}H NMR (400 MHz, $CDCl_3$)

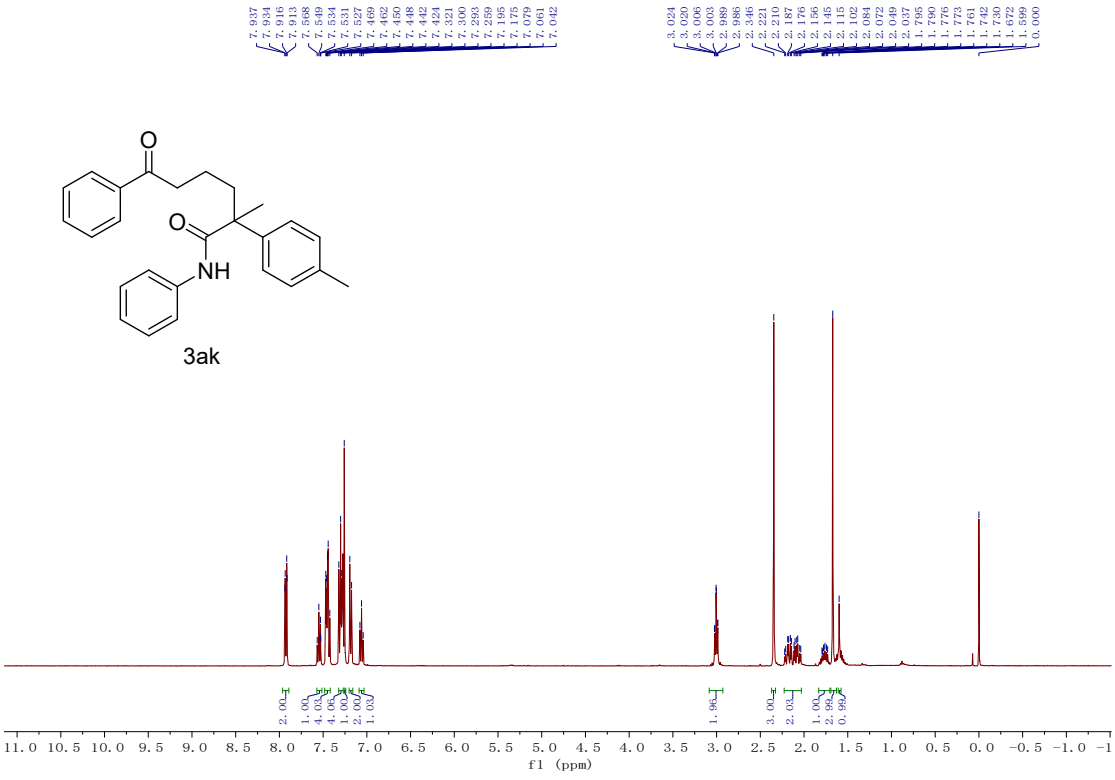


^{13}C NMR (100 MHz, $CDCl_3$)

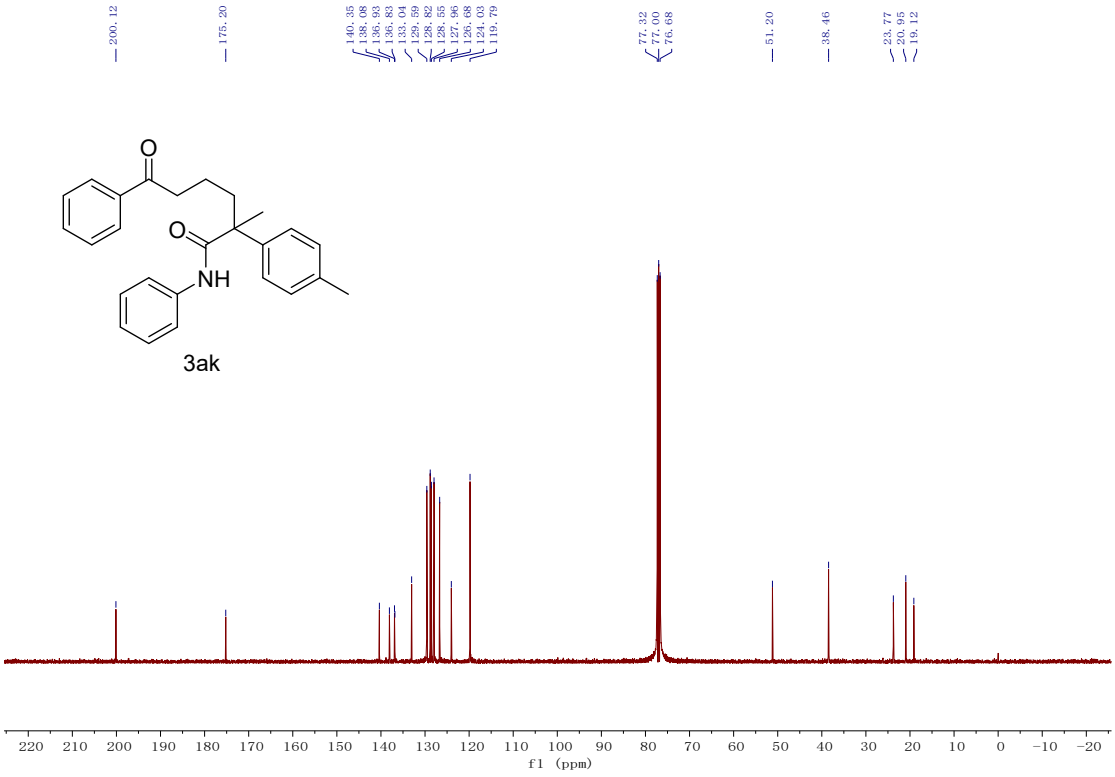


^{19}F NMR (376 MHz, $CDCl_3$)

2-methyl-6-oxo-N,6-diphenyl-2-(p-tolyl)hexanamide

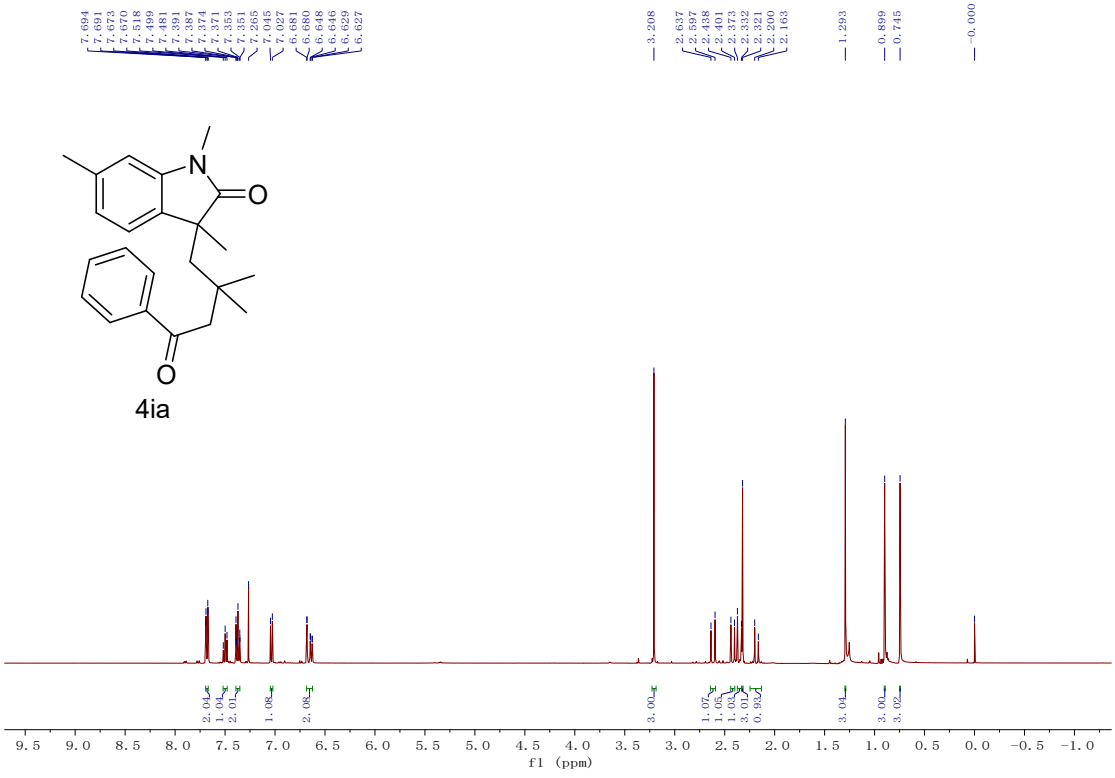


^{1}H NMR (400 MHz, $CDCl_3$)

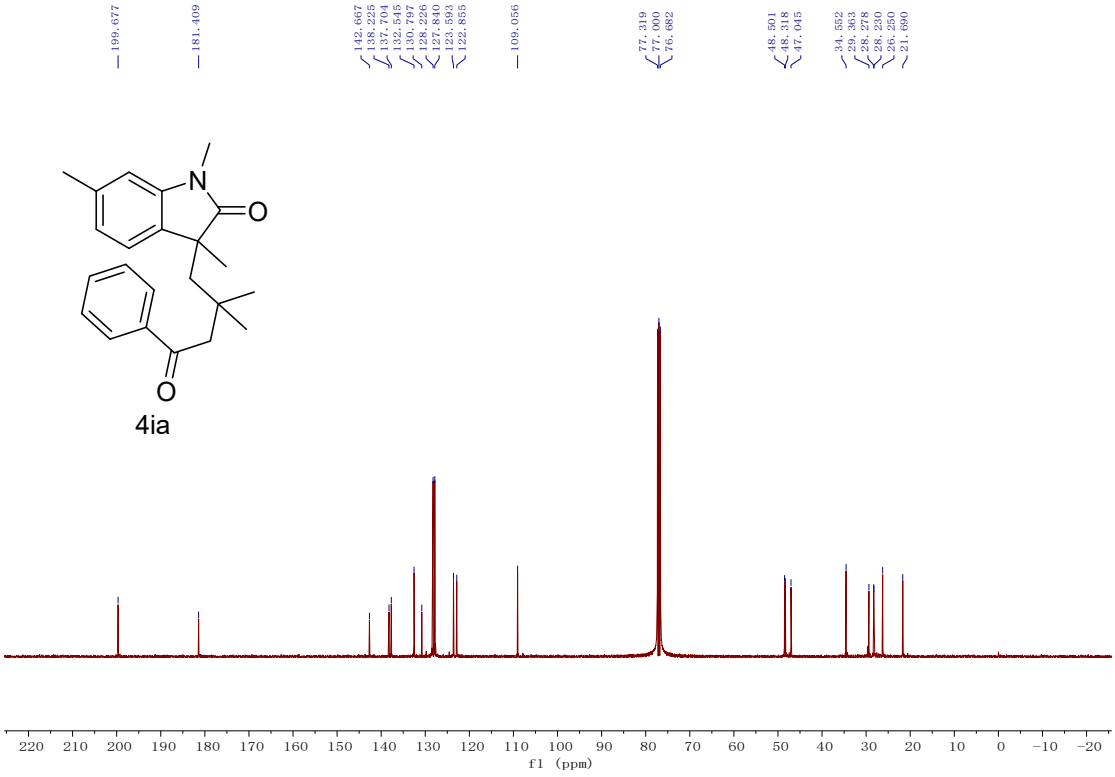


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one

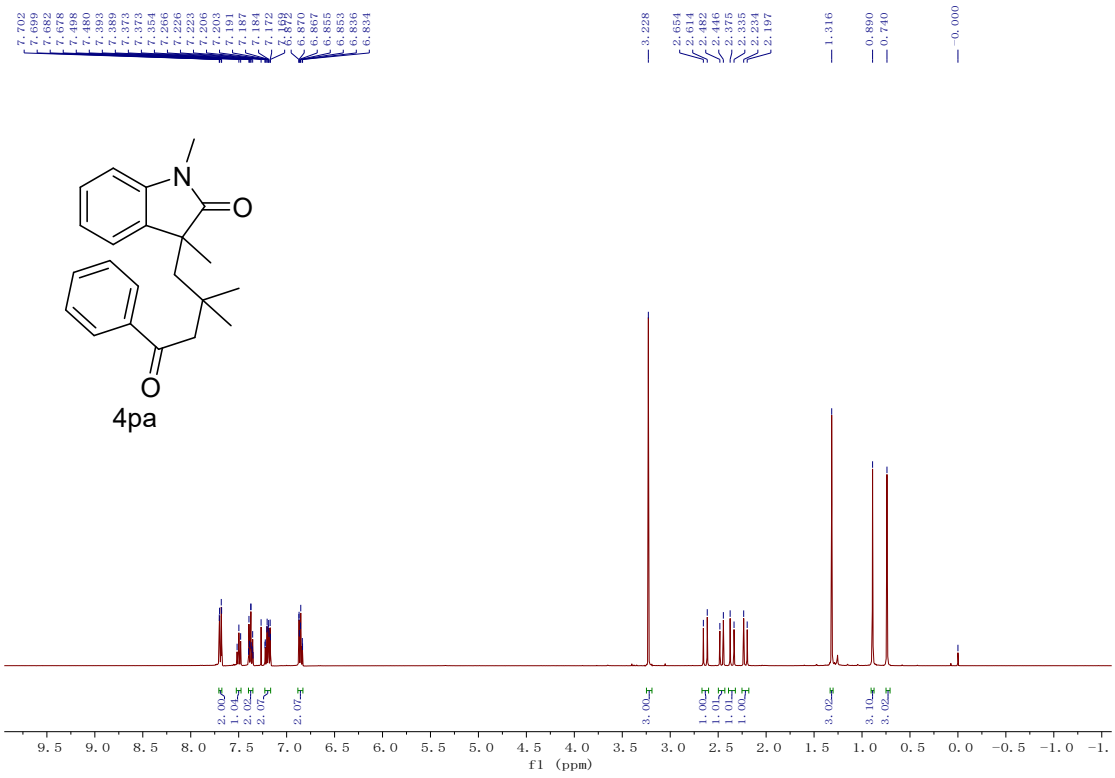


^{1}H NMR (400 MHz, $CDCl_3$)

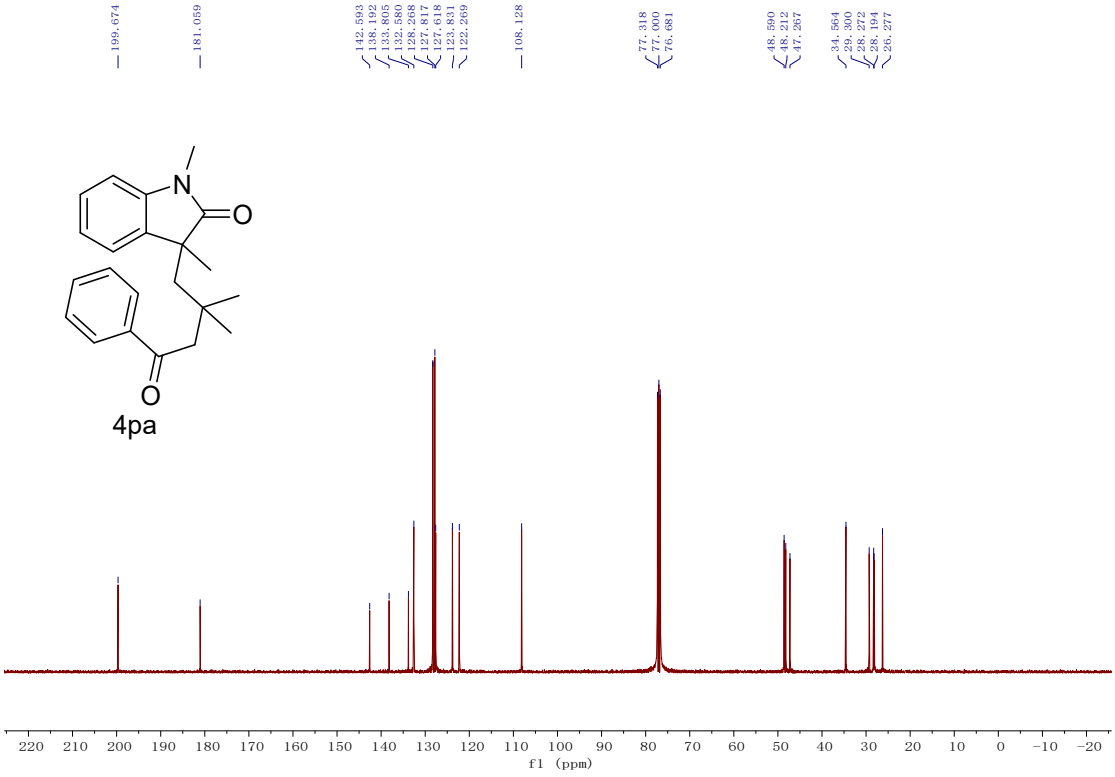


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethylindolin-2-one

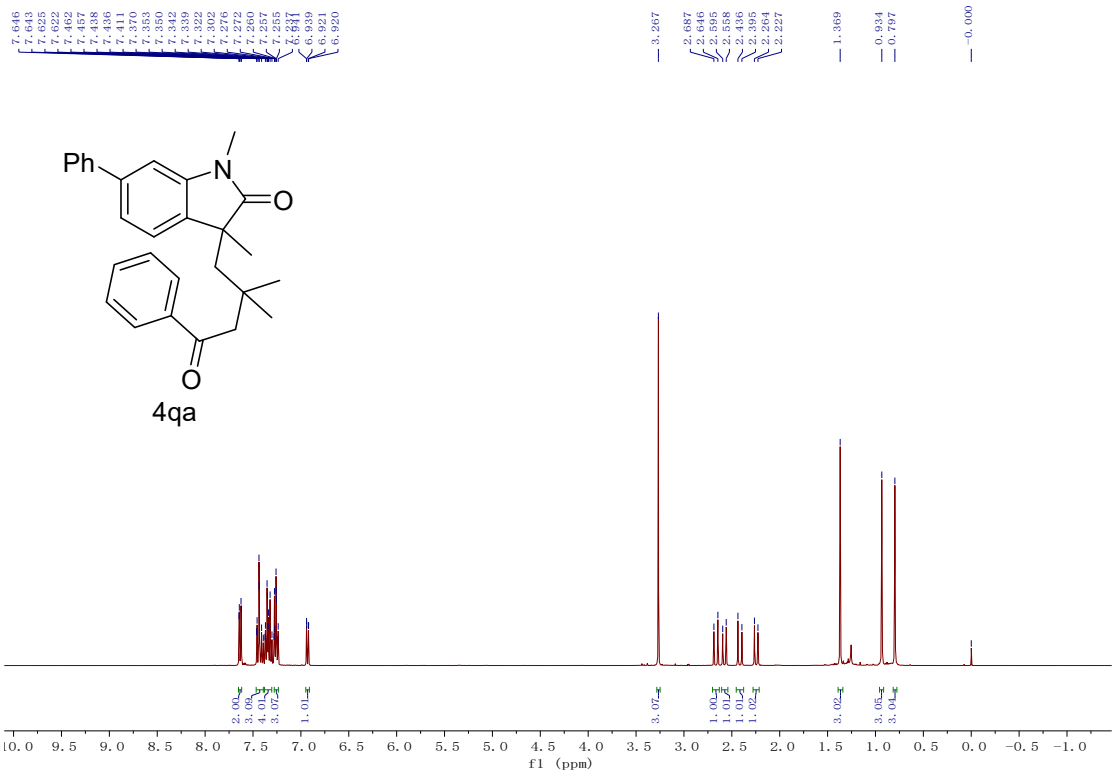


^{1}H NMR (400 MHz, $CDCl_3$)

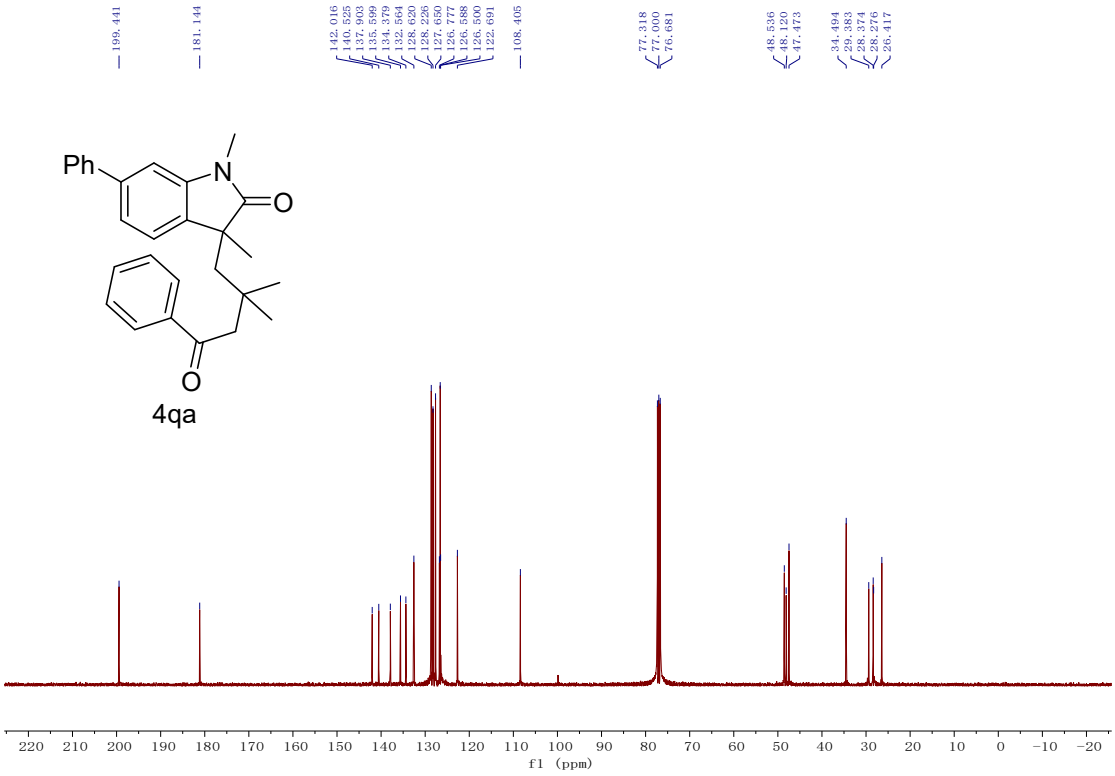


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethyl-6-phenylindolin-2-one

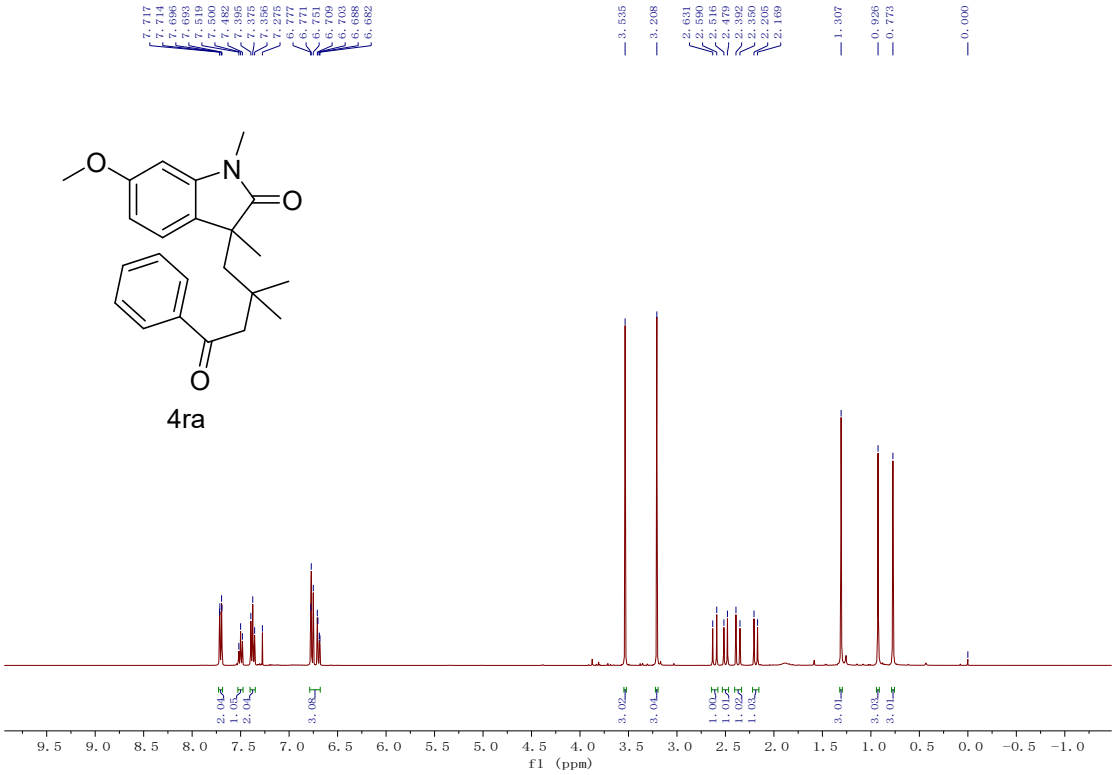


^{1}H NMR (400 MHz, $CDCl_3$)

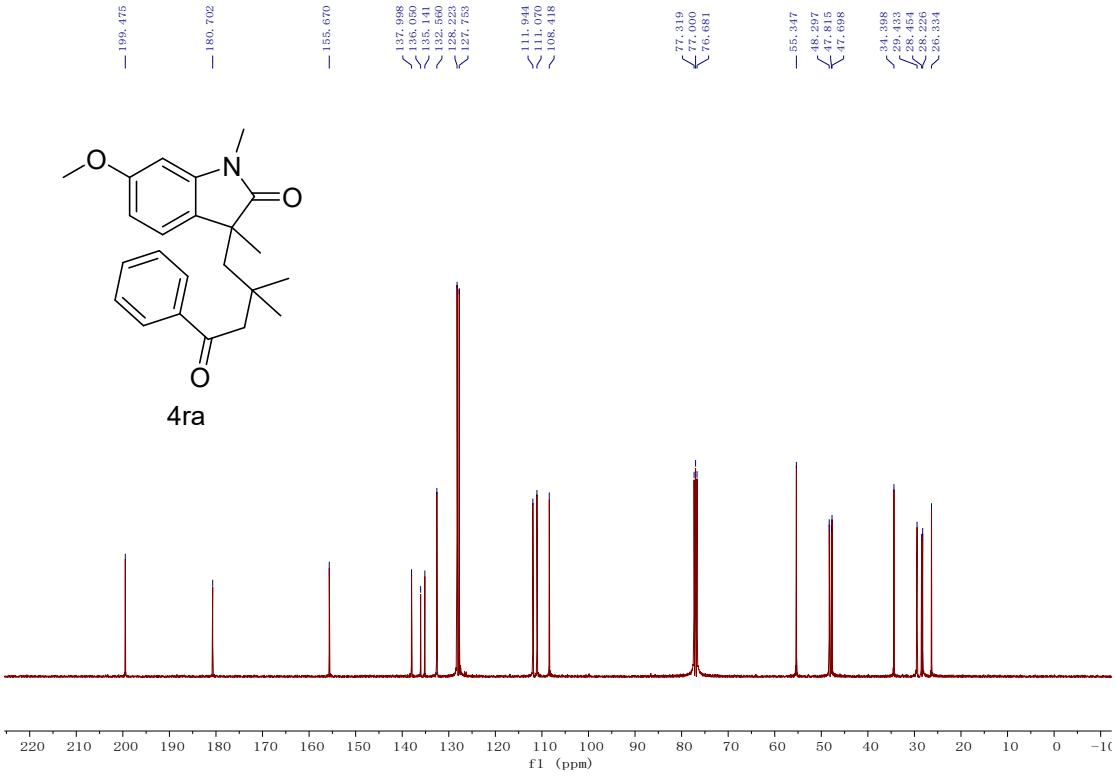


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-6-methoxy-1,3-dimethylindolin-2-one

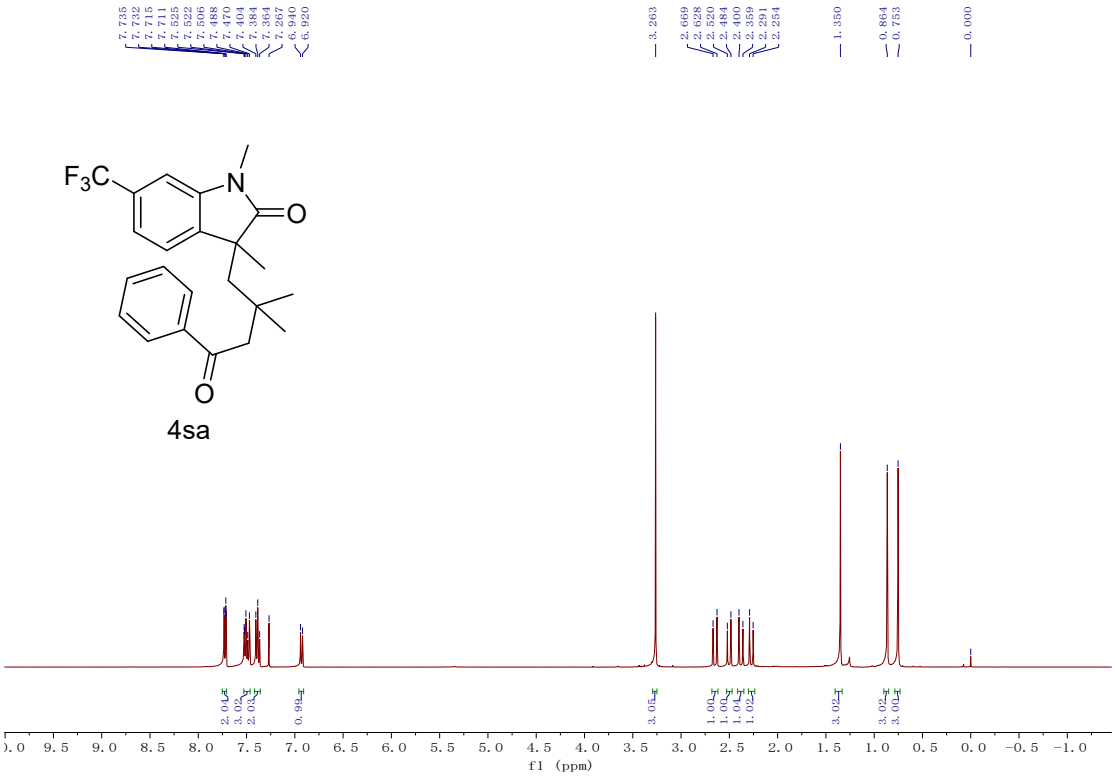


^{1}H NMR (400 MHz, $CDCl_3$)

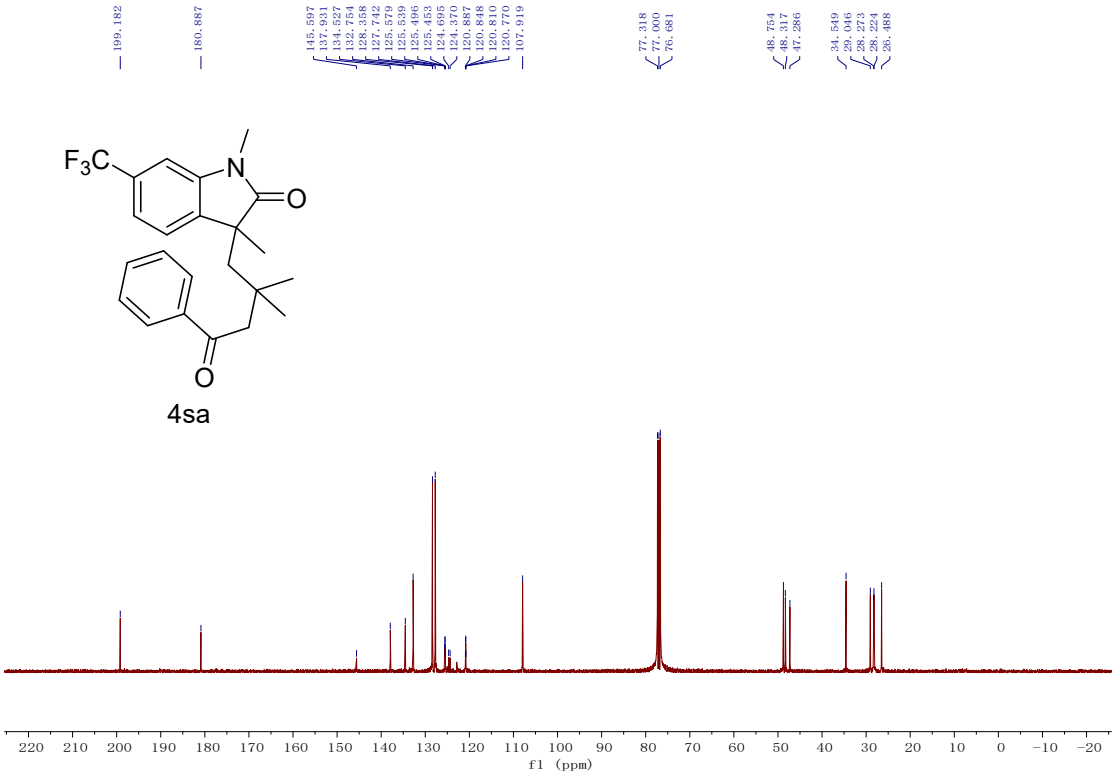


^{13}C NMR (100 MHz, $CDCl_3$)

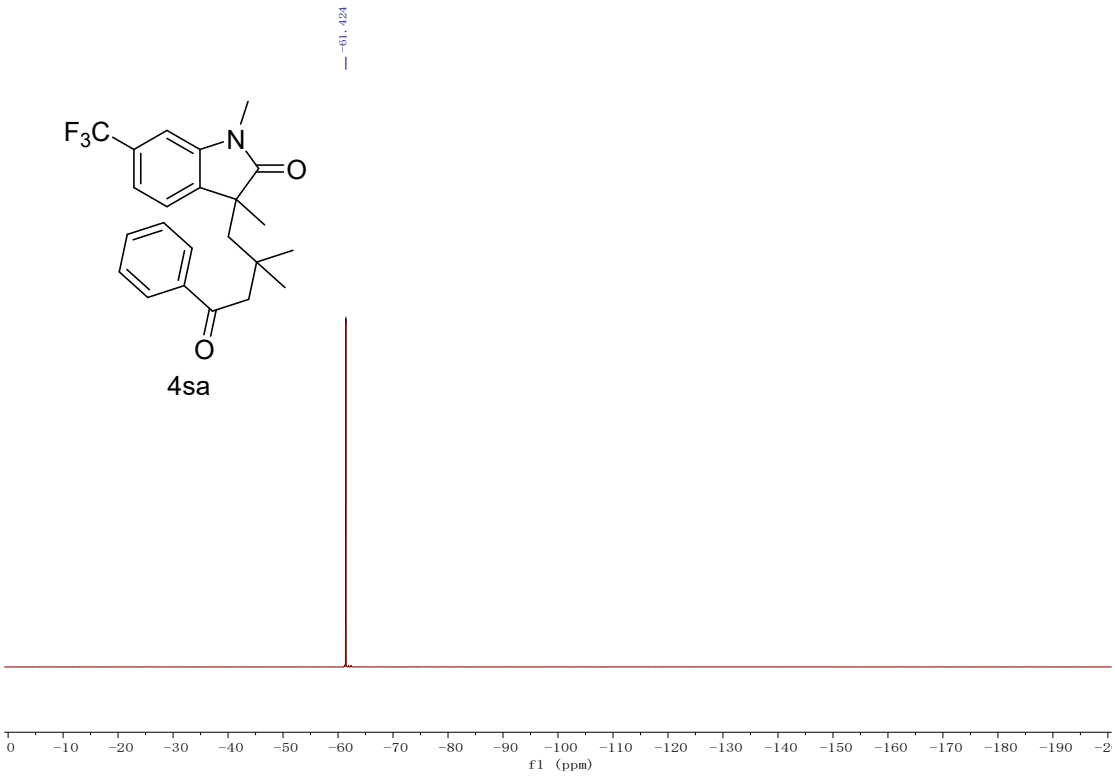
3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethyl-6-(trifluoromethyl)indolin-2-one



^{1}H NMR (400 MHz, $CDCl_3$)

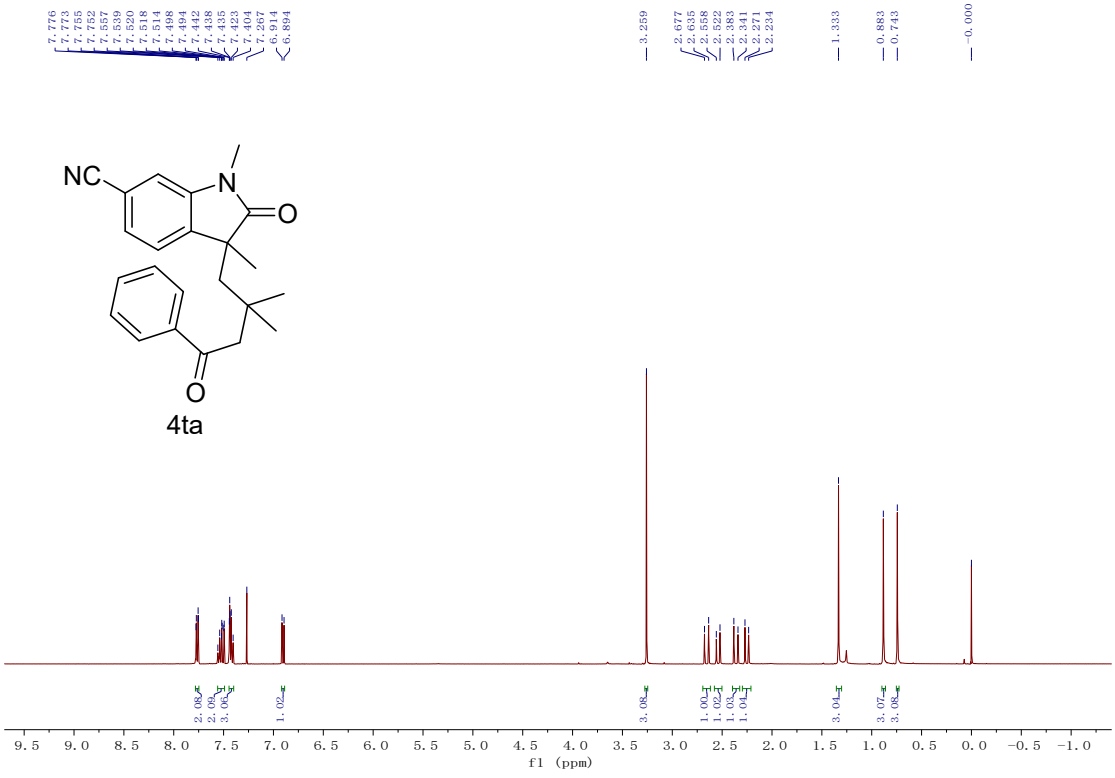


^{13}C NMR (100 MHz, $CDCl_3$)

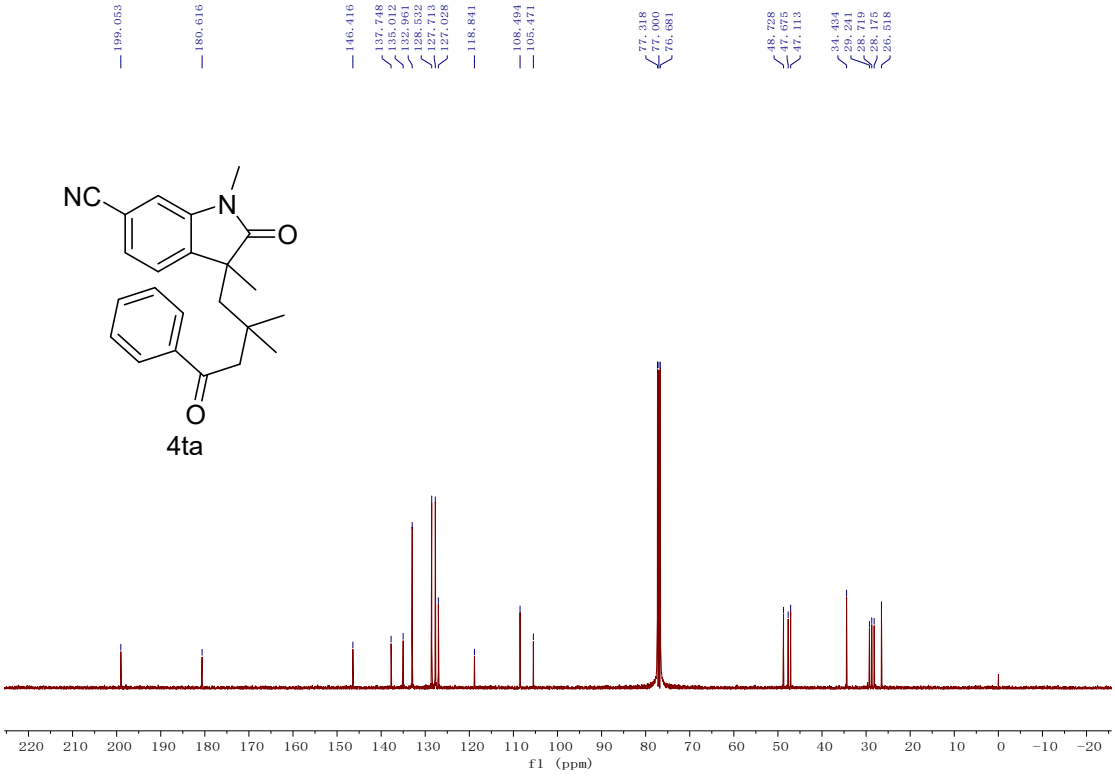


^{19}F NMR (376 MHz, $CDCl_3$)

3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3-dimethyl-2-oxoindoline-6-carbonitrile

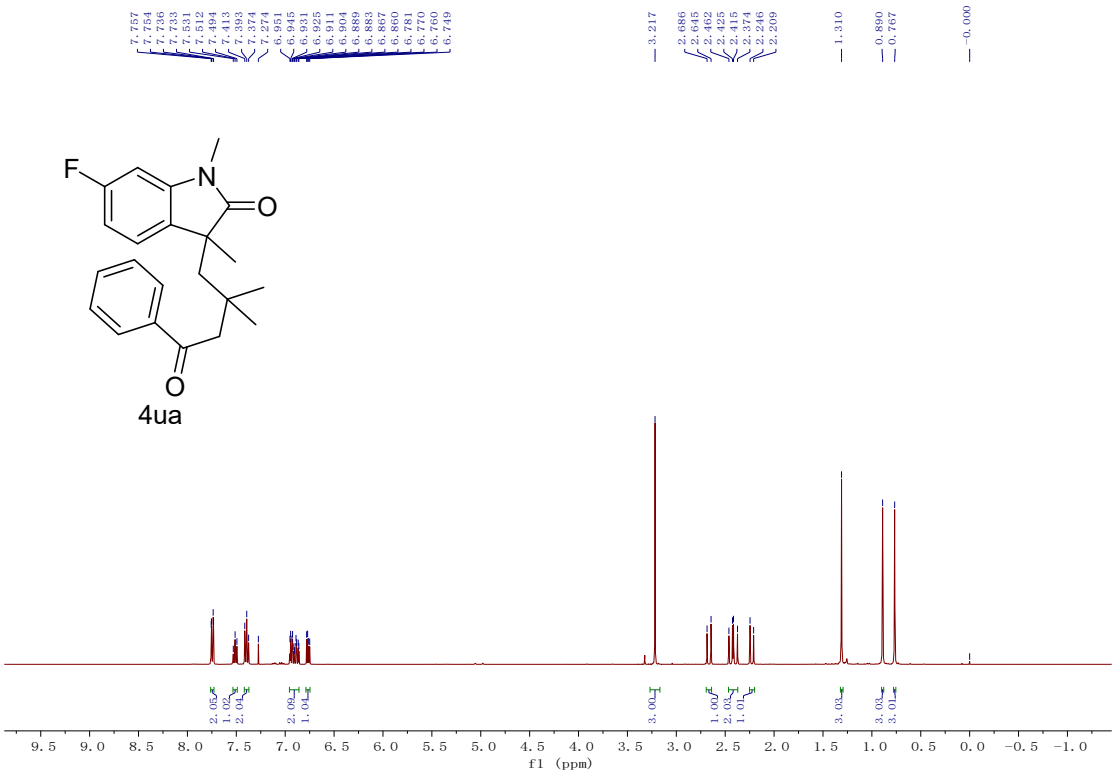


^{1}H NMR (400 MHz, $CDCl_3$)

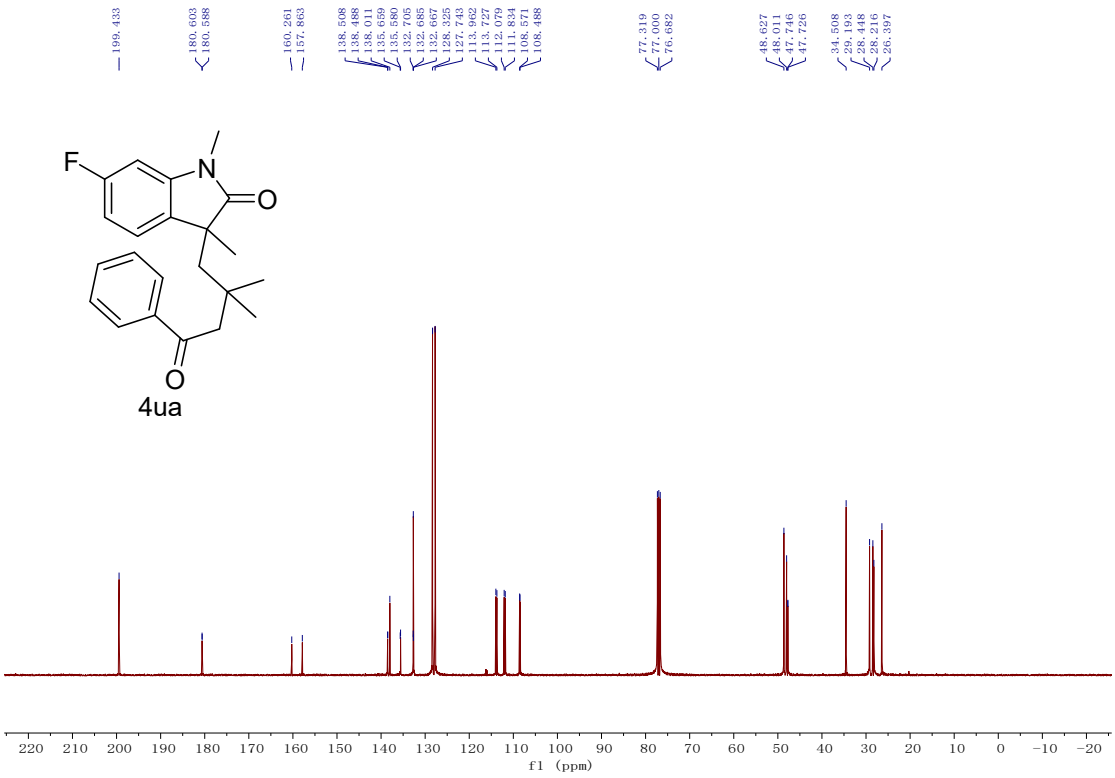


^{13}C NMR (100 MHz, $CDCl_3$)

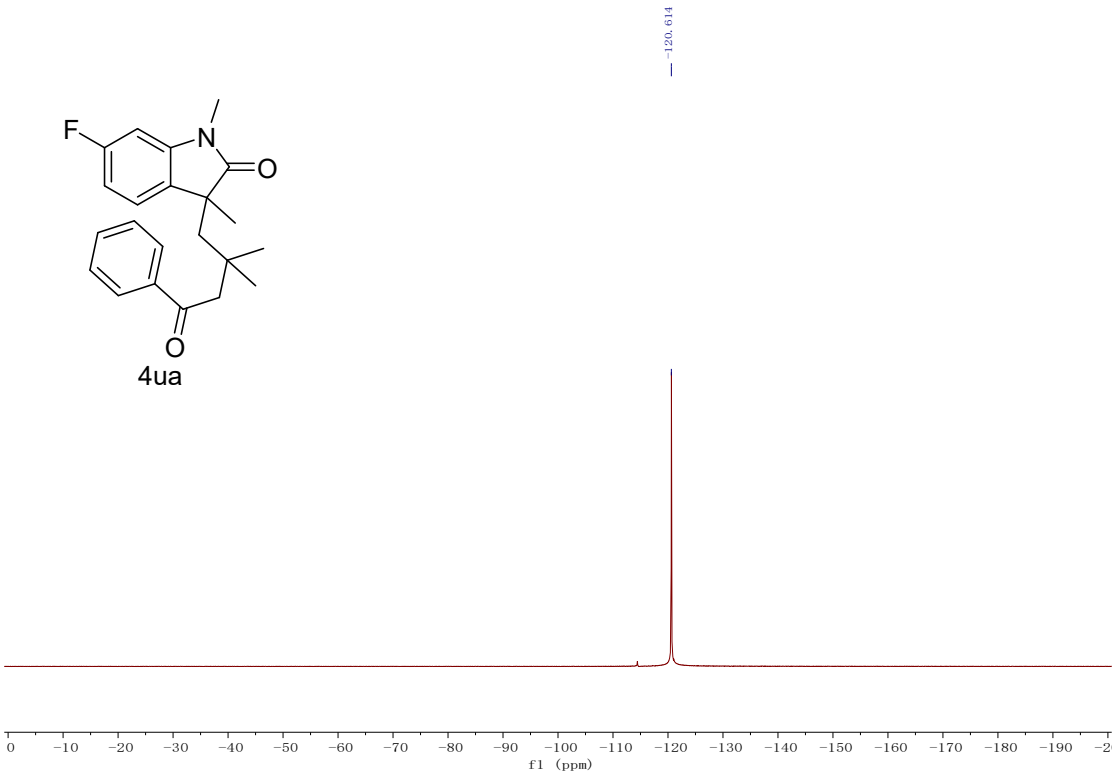
3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-6-fluoro-1,3-dimethylindolin-2-one



^{1}H NMR (400 MHz, $CDCl_3$)

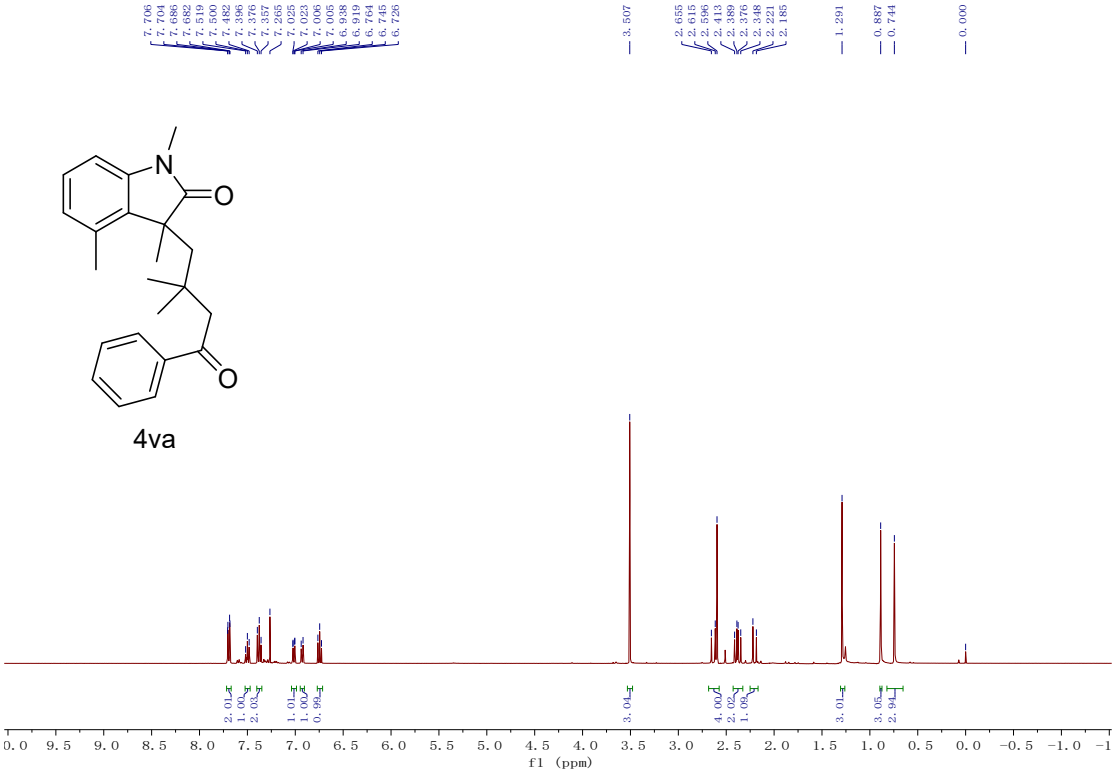


^{13}C NMR (100 MHz, $CDCl_3$)

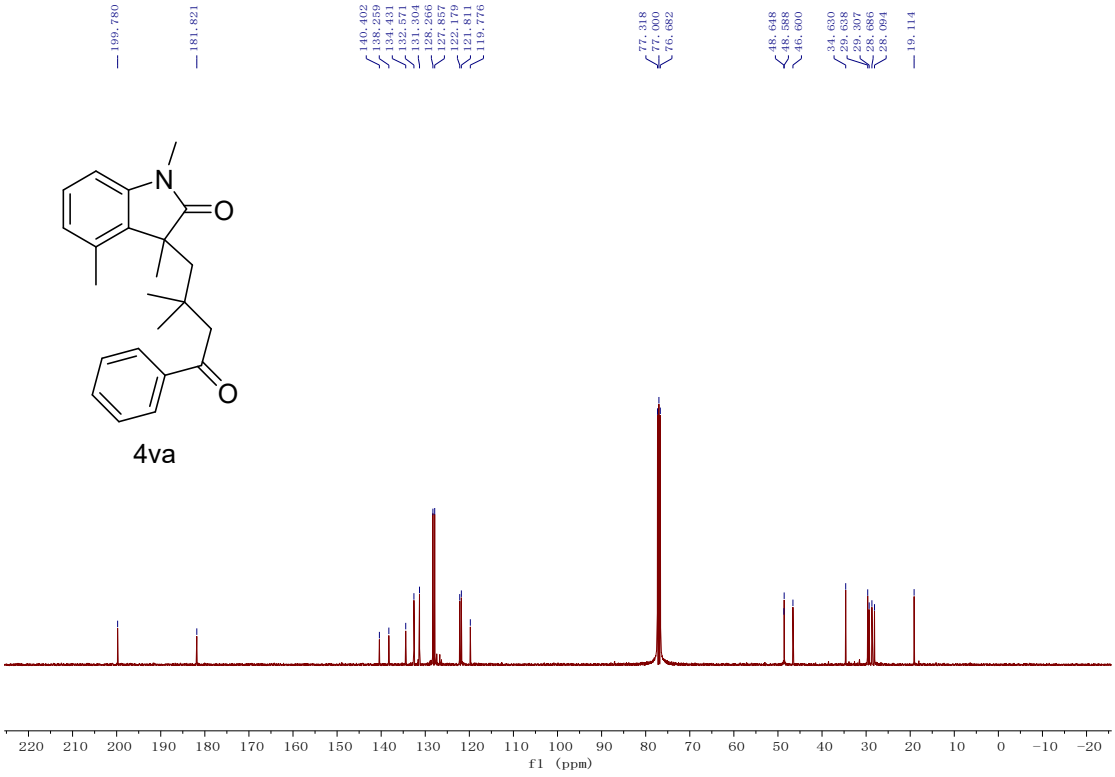


^{19}F NMR (376 MHz, $CDCl_3$)

3-(2,2-dimethyl-4-oxo-4-phenylbutyl)-1,3,4-trimethylindolin-2-one

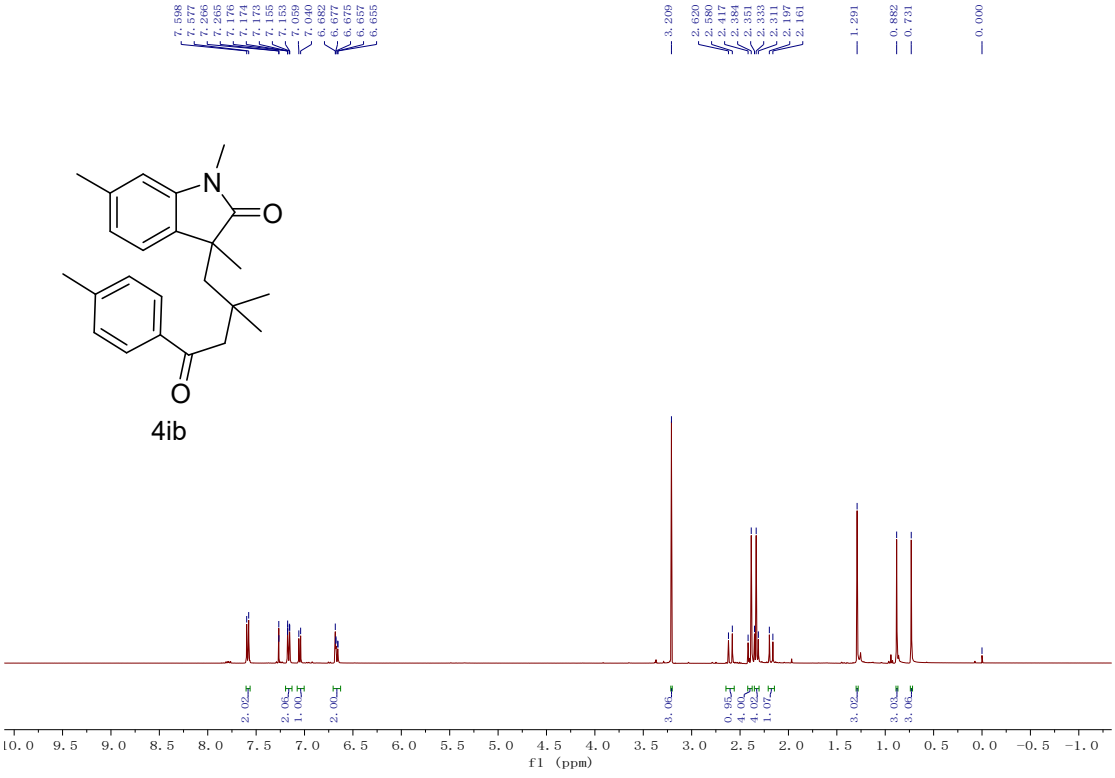


^{1}H NMR (400 MHz, $CDCl_3$)

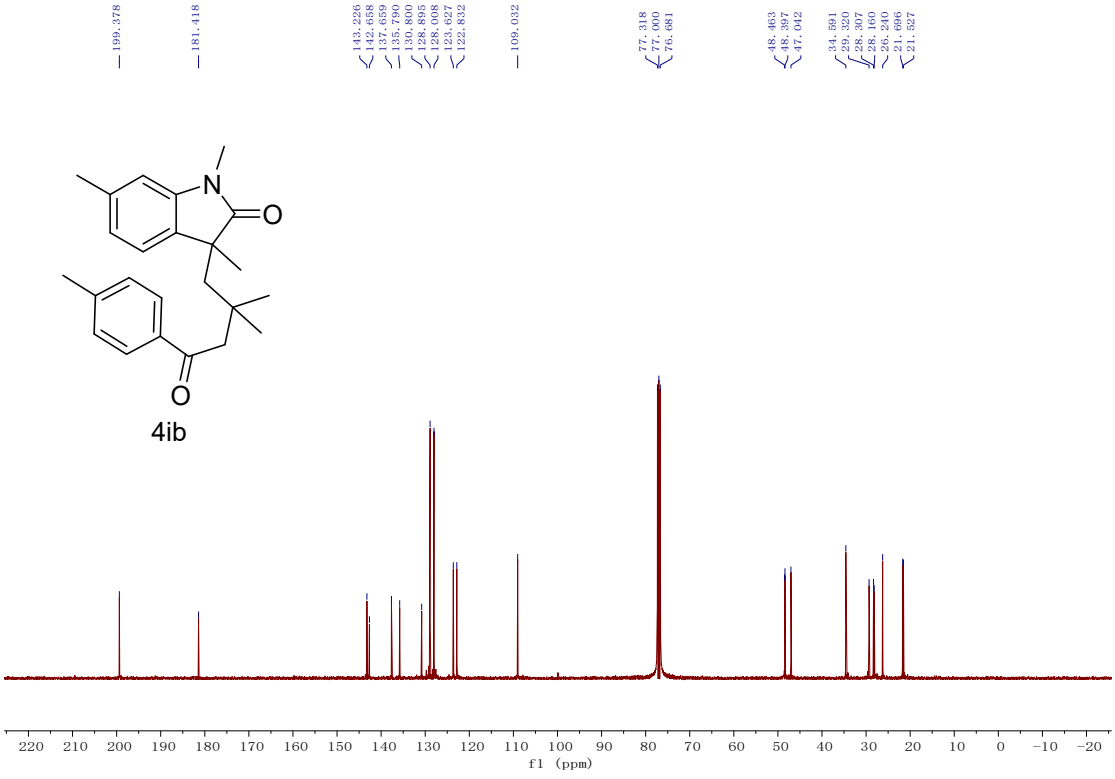


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2,2-dimethyl-4-oxo-4-(p-tolyl)butyl)-1,3,6-trimethylindolin-2-one

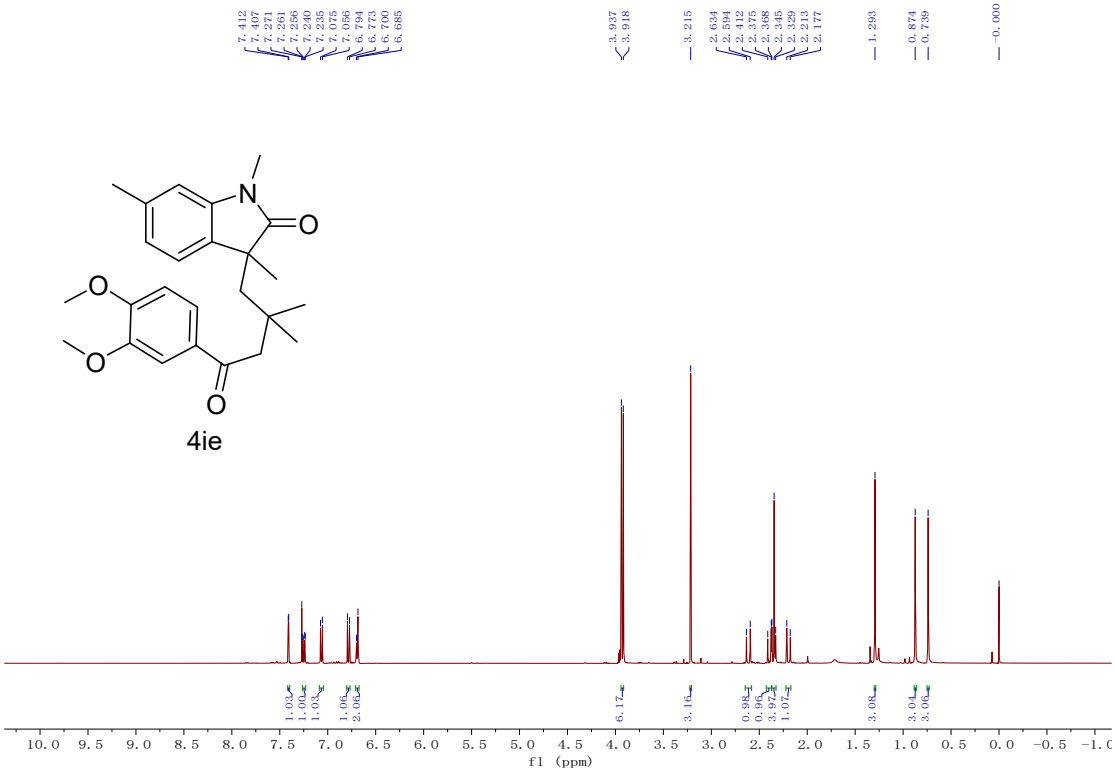


^{1}H NMR (400 MHz, $CDCl_3$)

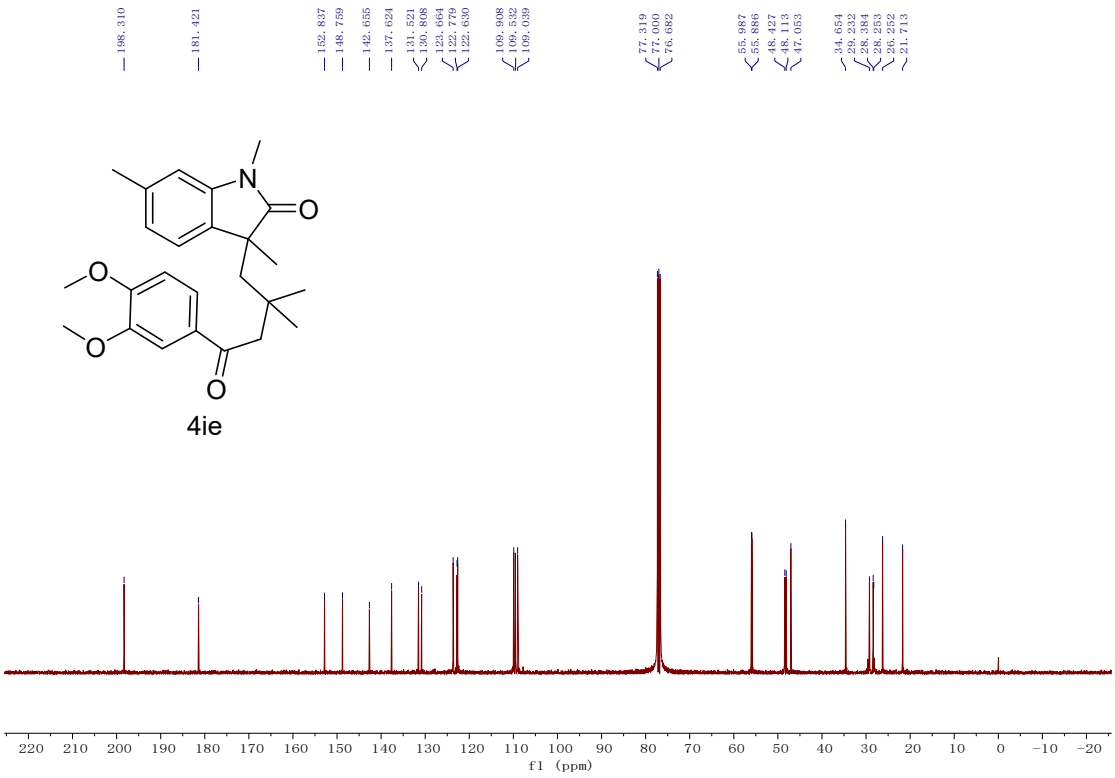


^{13}C NMR (100 MHz, $CDCl_3$)

3-(4-(3,4-dimethoxyphenyl)-2,2-dimethyl-4-oxobutyl)-1,3,6-trimethylindolin-2-one

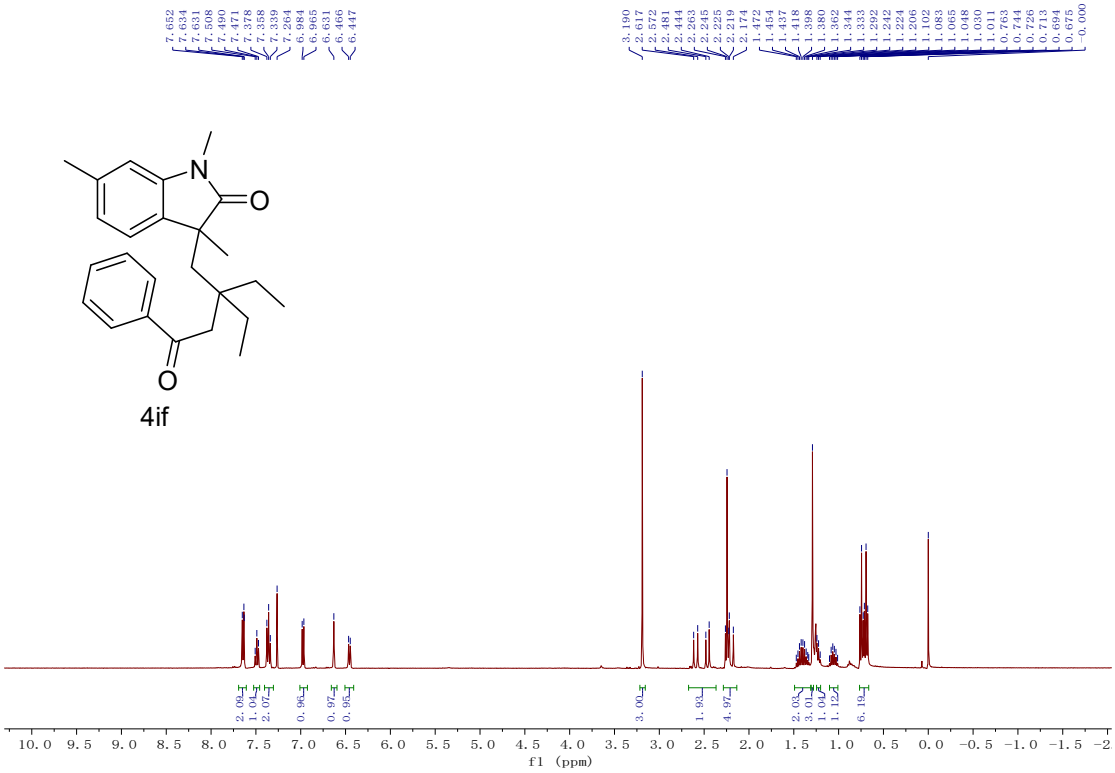


^{1}H NMR (400 MHz, $CDCl_3$)

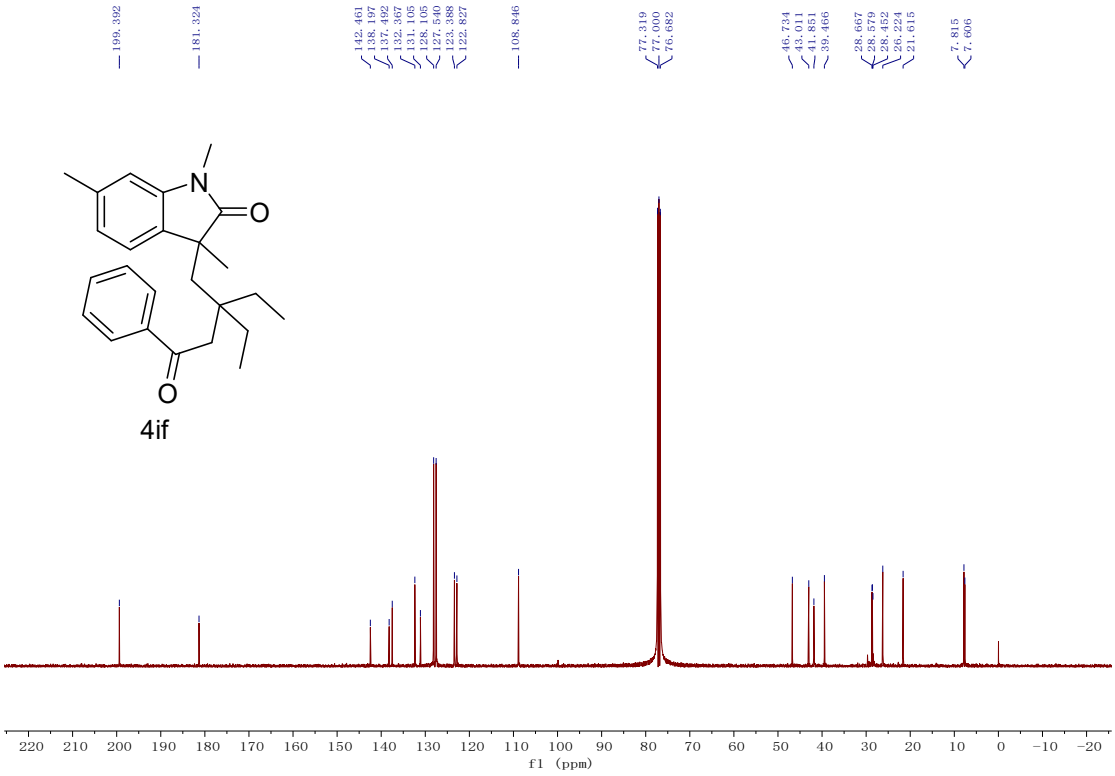


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2,2-diethyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one

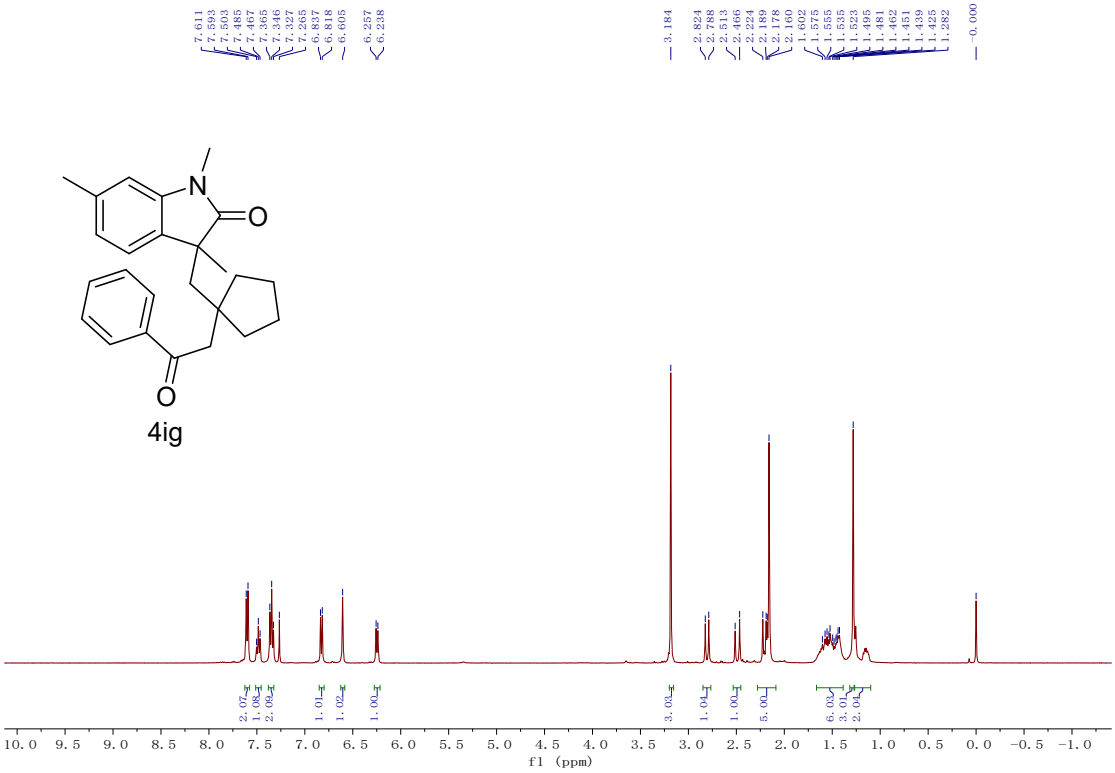


^{1}H NMR (400 MHz, $CDCl_3$)

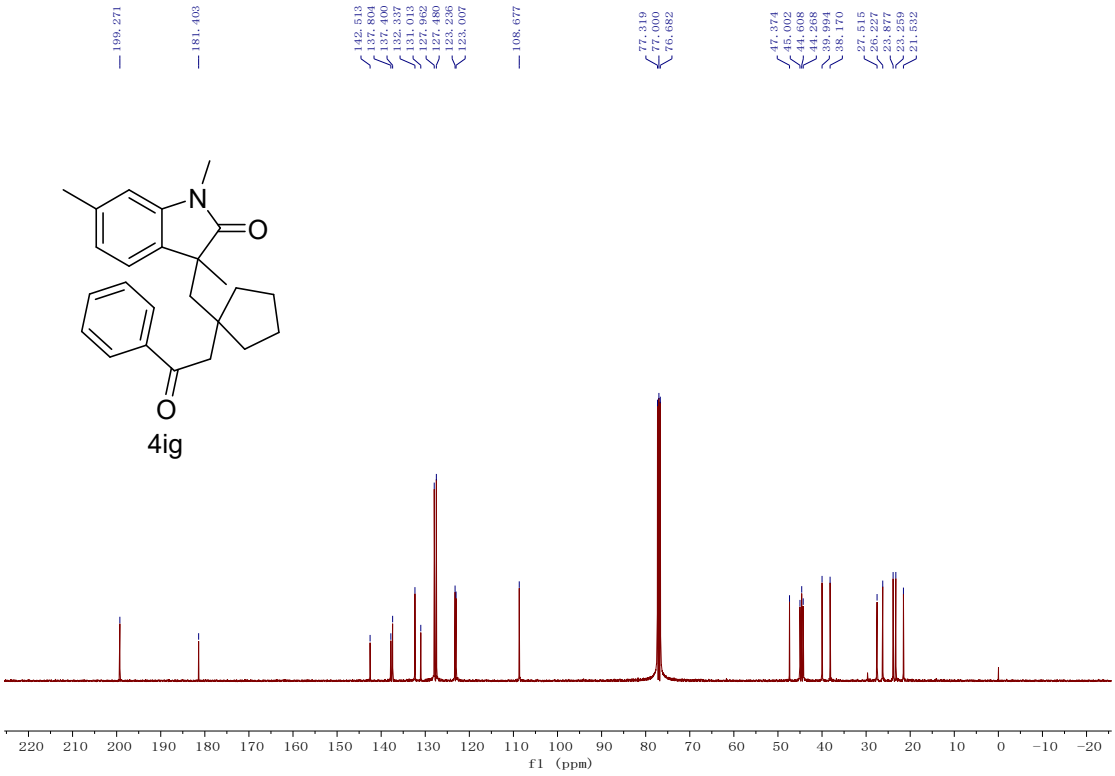


^{13}C NMR (100 MHz, $CDCl_3$)

1,3,6-trimethyl-3-((1-(2-oxo-2-phenylethyl)cyclopentyl)methyl)indolin-2-one

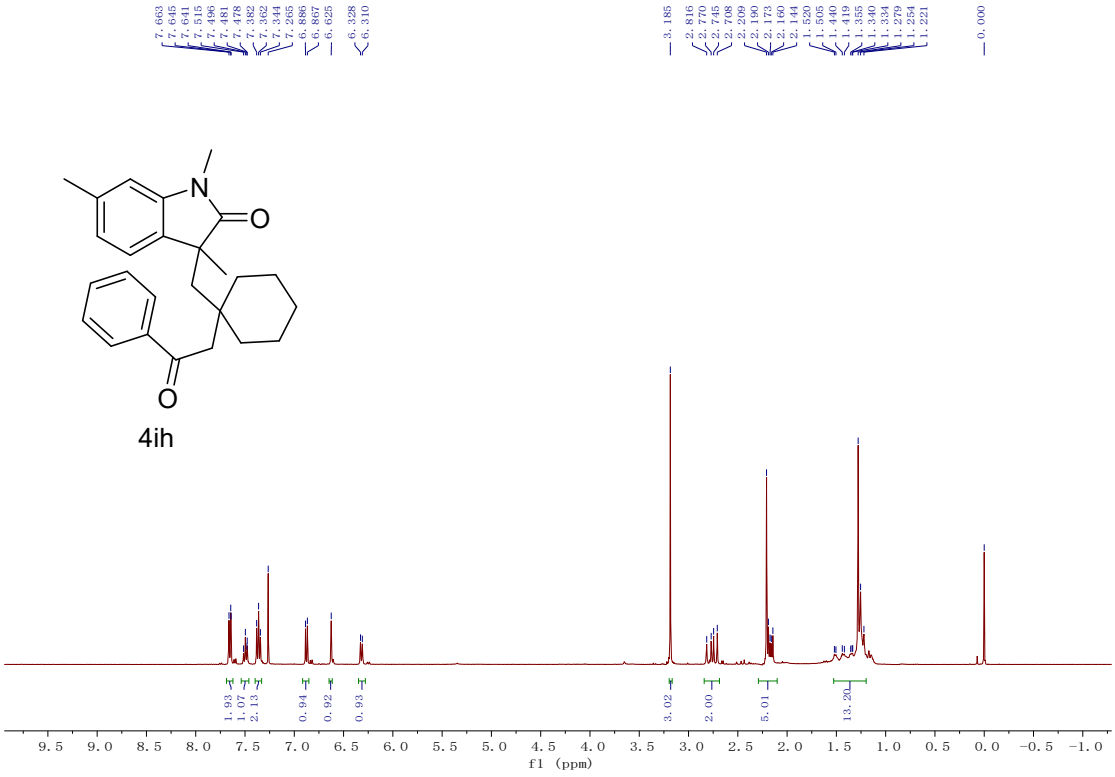


^{1}H NMR (400 MHz, $CDCl_3$)

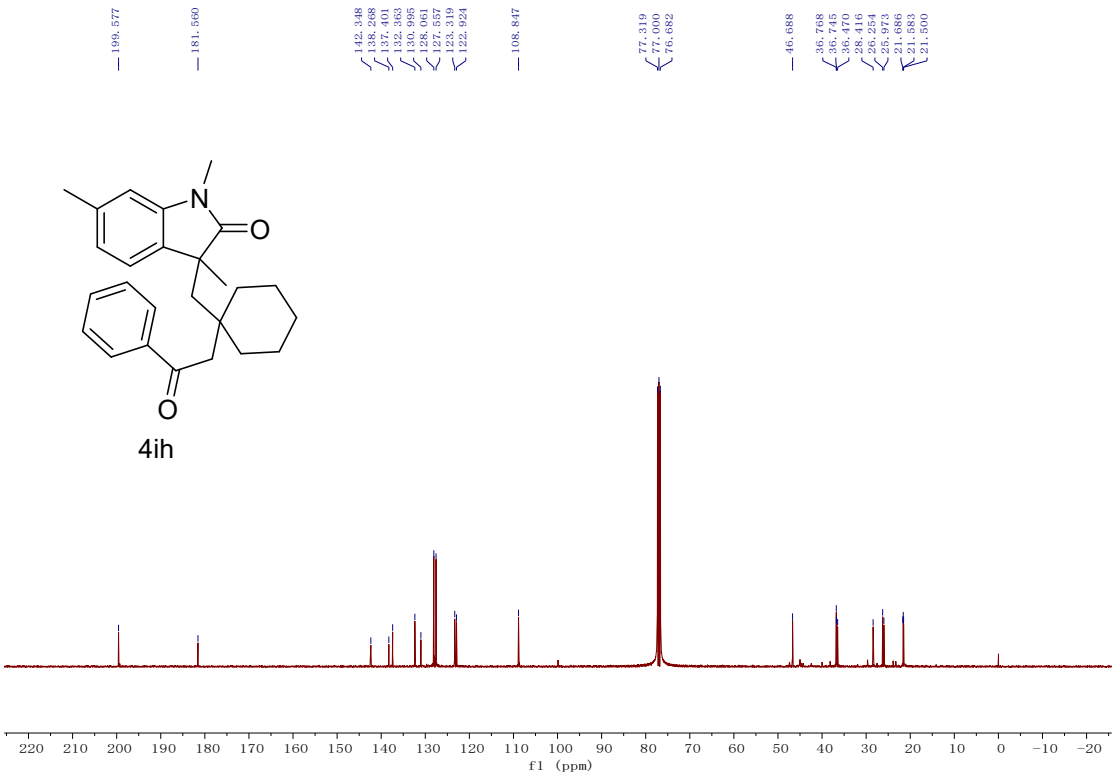


^{13}C NMR (100 MHz, $CDCl_3$)

1,3,6-trimethyl-3-((1-(2-oxo-2-phenylethyl)cyclohexyl)methyl)indolin-2-one

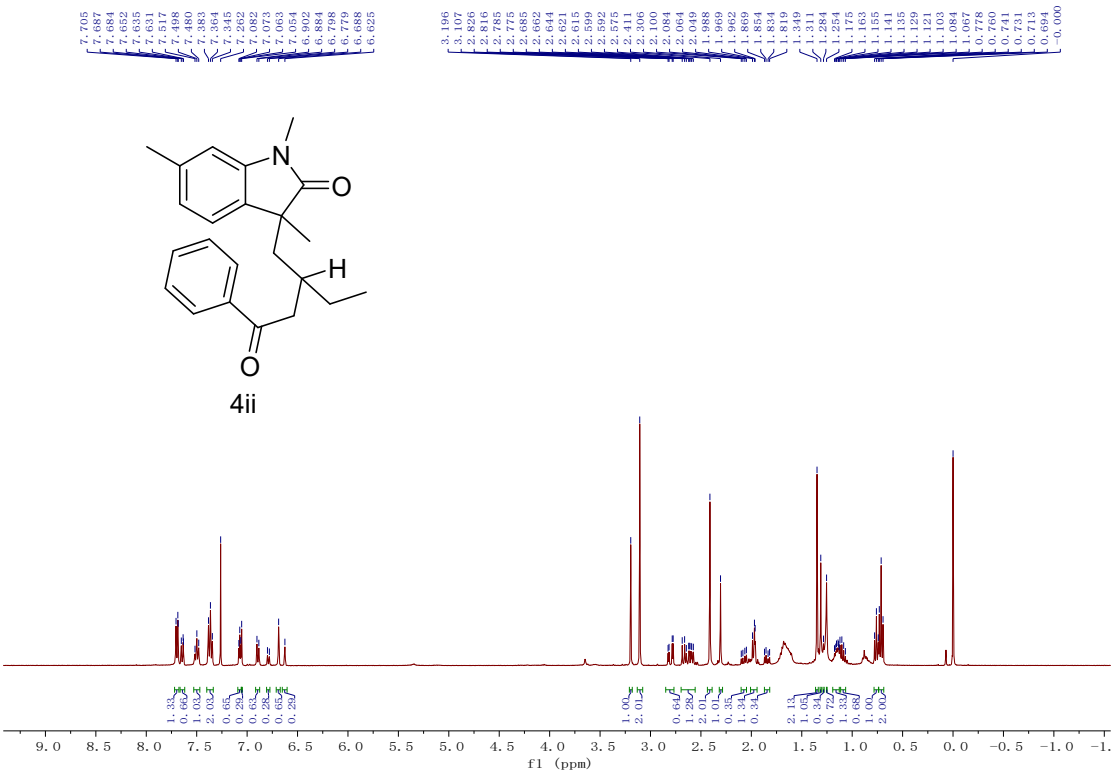


^{1}H NMR (400 MHz, $CDCl_3$)

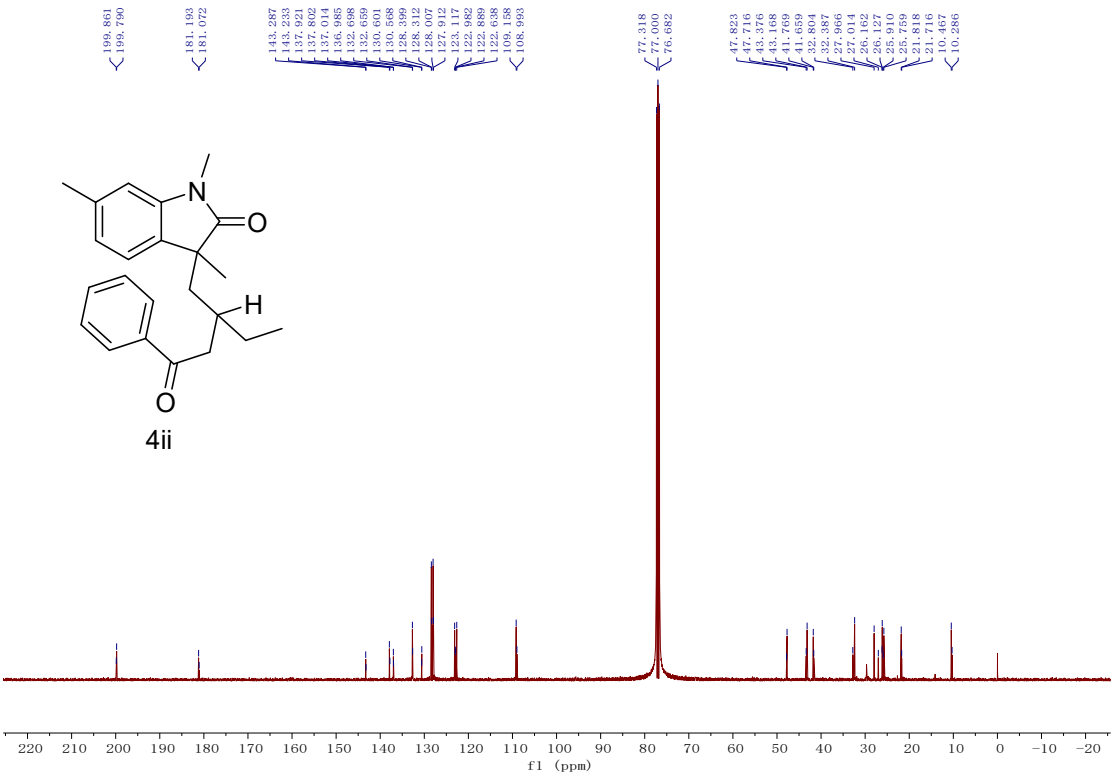


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2-ethyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one

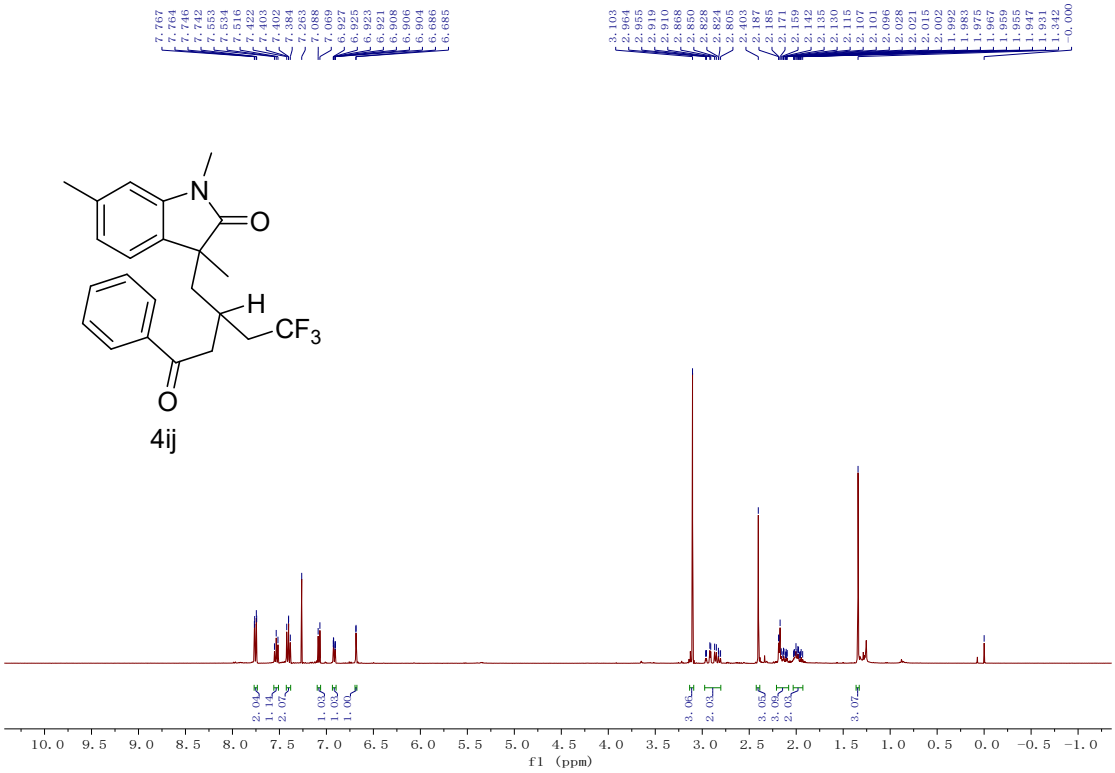


^{1}H NMR (400 MHz, $CDCl_3$)

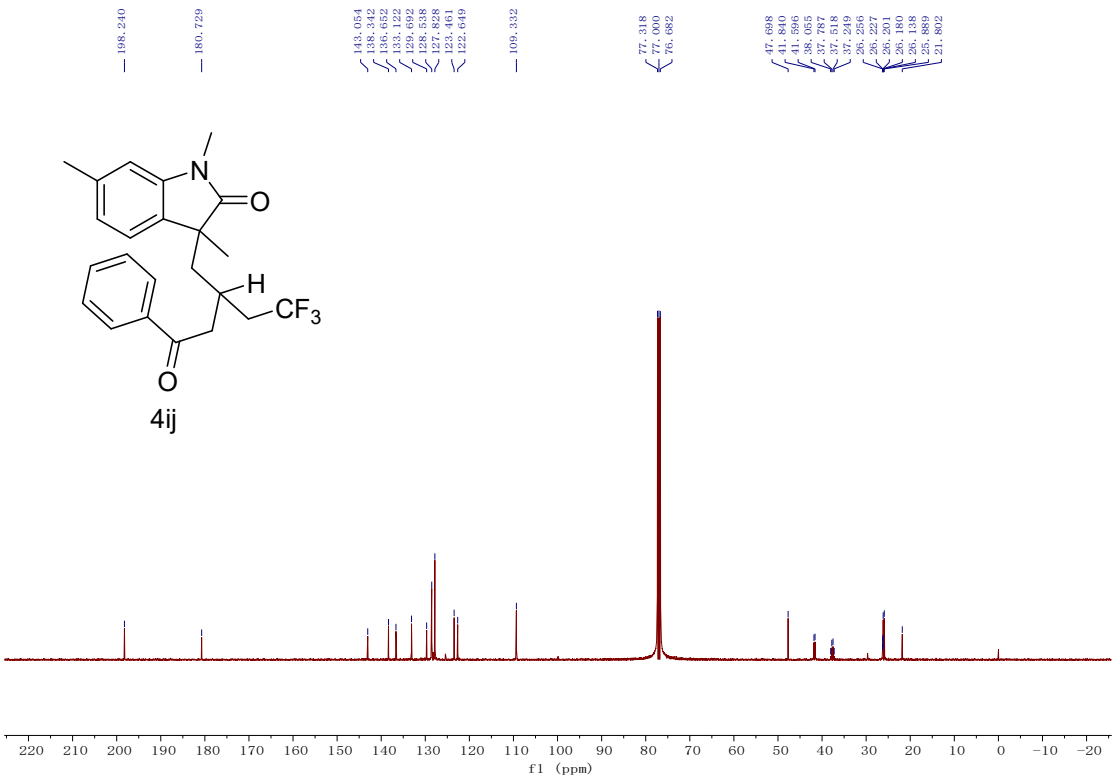


^{13}C NMR (100 MHz, $CDCl_3$)

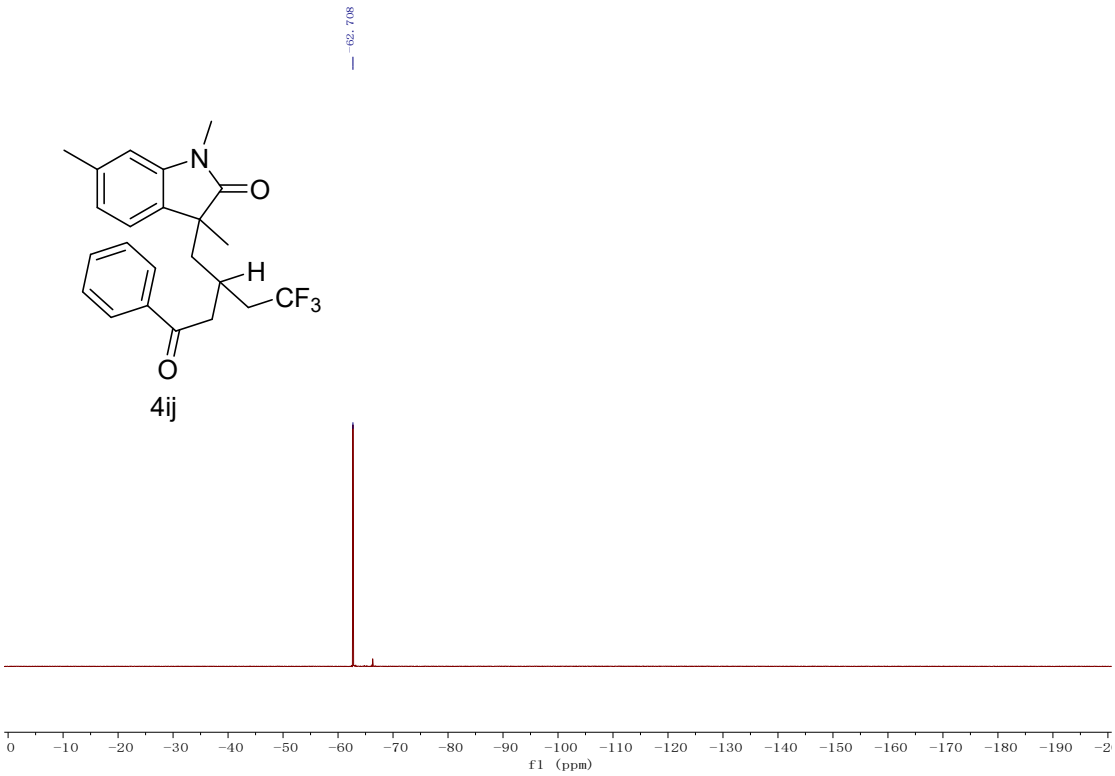
1,3,6-trimethyl-3-(4,4,4-trifluoro-2-(2-oxo-2-phenylethyl)butyl)indolin-2-one



^{1}H NMR (400 MHz, $CDCl_3$)

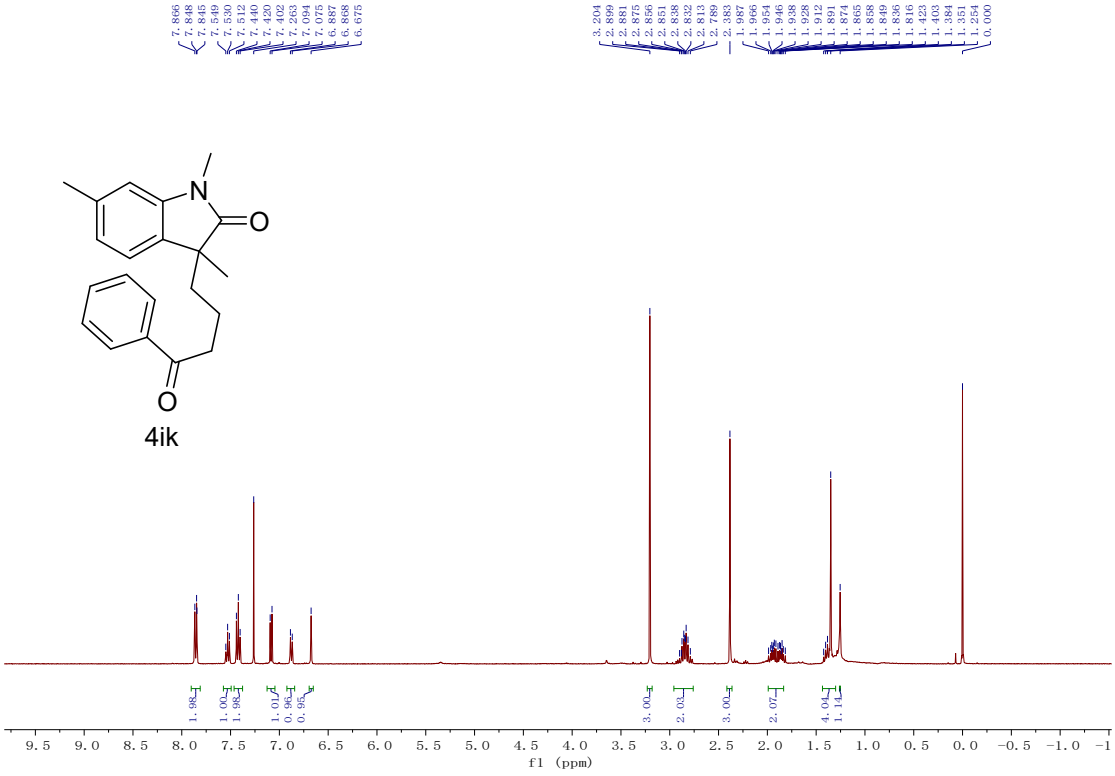


^{13}C NMR (100 MHz, $CDCl_3$)

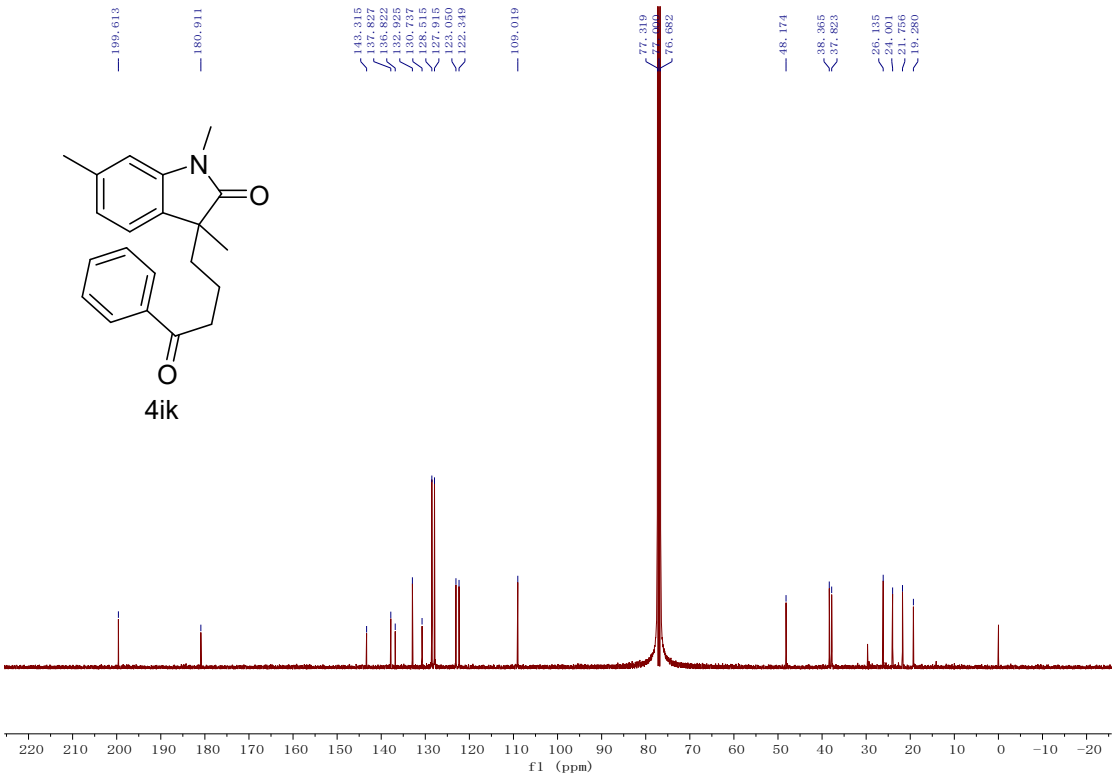


^{19}F NMR (376 MHz, $CDCl_3$)

1,3,6-trimethyl-3-(4-oxo-4-phenylbutyl)indolin-2-one

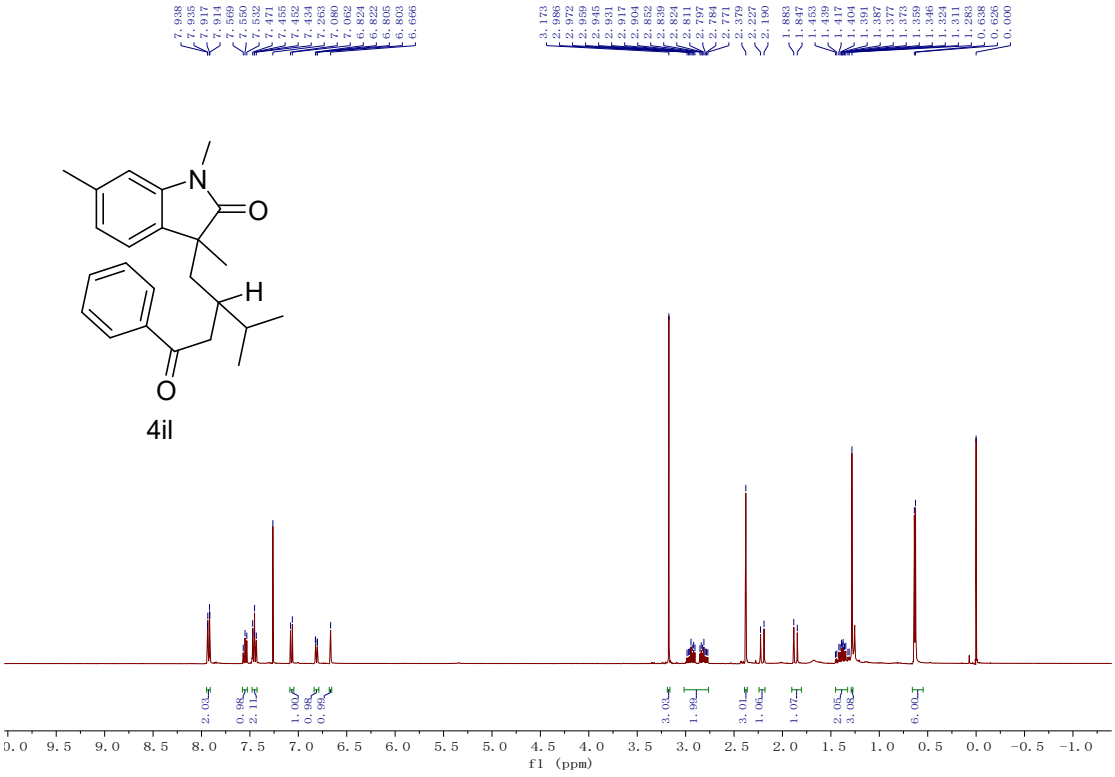


^{1}H NMR (400 MHz, $CDCl_3$)

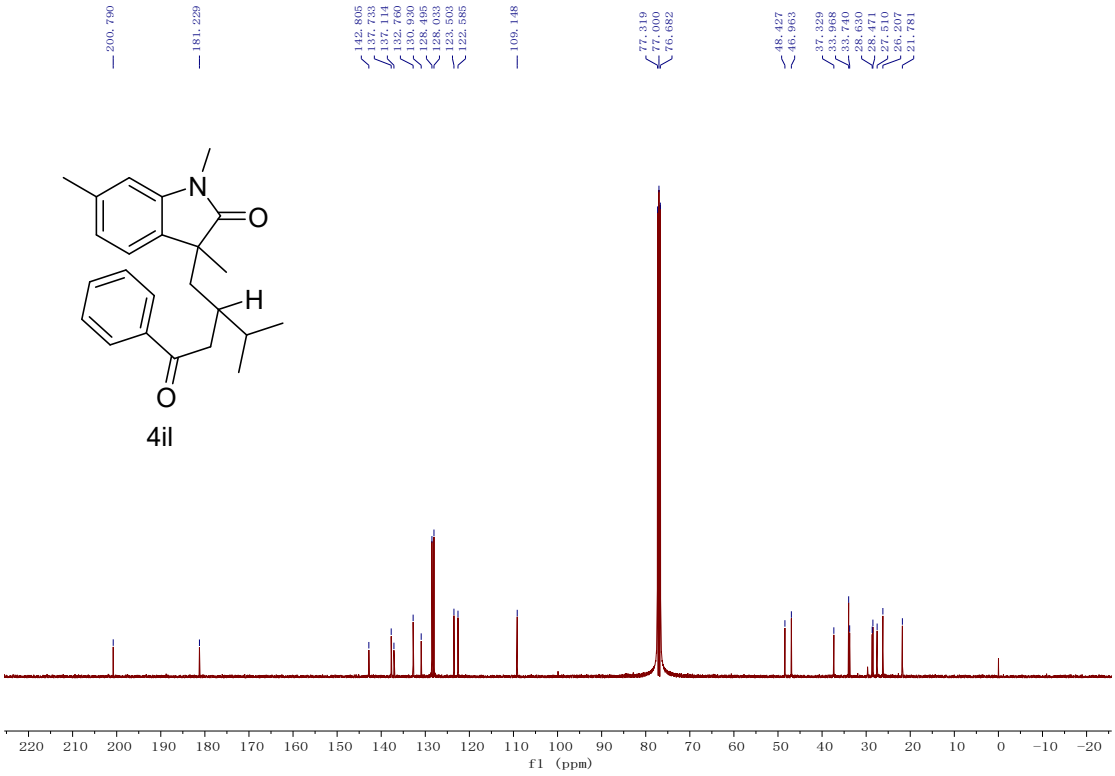


^{13}C NMR (100 MHz, $CDCl_3$)

3-(2-isopropyl-4-oxo-4-phenylbutyl)-1,3,6-trimethylindolin-2-one



^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

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2 Ryu, J.; Kwak, J.; Shin, K.; Lee, D.; Chang, S. Ir(III)-Catalyzed Mild C-H Amidation of Arenes and Alkenes: An Efficient Usage of Acyl Azides as the Nitrogen Source. *J. Am. Chem. Soc*. **2013**, *135*, 12861-12868.

3 Gao D.; Jiao, L. *Angew. Chem. Int. Ed*. **2022**, *61*, e202116024.

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