Supporting Information

Direct C-H Alkylation of 3,4-Dihydroquinoxaline-2-one with *N*-(acyloxy)phthalimide via Radical-Radical Cross Coupling

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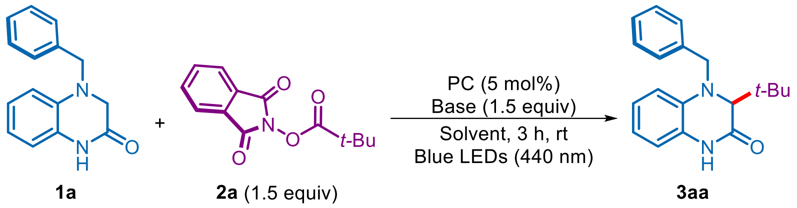
1. Table S1: Complete optimization table:[a]

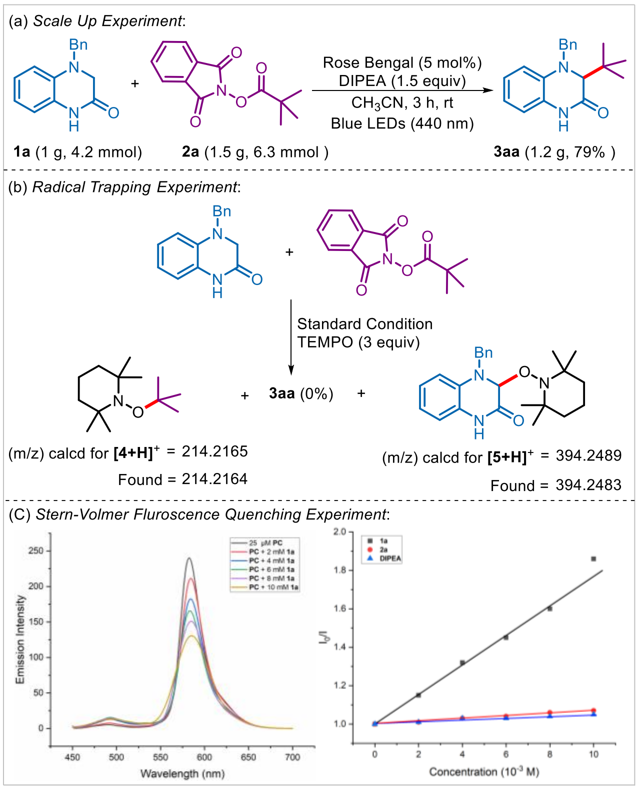


Sl. No	PC	Base	Solvent	Yield (%)[b]
1.	Eosin B	DIPEA	CH_3CN	Trace
2.	Rhodamine B	DIPEA	CH_3CN	N.D
3.	Acridine perchlorate	DIPEA	CH_3CN	N.D
4.	Methylene Blue	DIPEA	CH_3CN	N.D
5.	Fluorescein	DIPEA	CH_3CN	78
6.	$Ru(bpy)_3Cl_2$. $6H_2O$	DIPEA	CH_3CN	85
7.	Eosin Y	DIPEA	CH_3CN	66
8.	Rose bengal	DIPEA	CH_3CN	90
9.	Rose bengal	DIPEA	THF	71
10.	Rose bengal	DIPEA	DMSO	N.D.
11.	Rose bengal	DIPEA	EtOH	75
12.	Rose bengal	DIPEA	DCM	61
13.	Rose bengal	TMEDA	CH_3CN	64
14.	Rose bengal	Et_3N	CH_3CN	46
15.	Rose bengal	Na_2CO_3	CH_3CN	20
16.	Rose bengal	CH_3CO_2Na	CH_3CN	32
17.[c]	Rose bengal	DIPEA	CH_3CN	49
18.[d]	Rose bengal	DIPEA	CH_3CN	76
19.	-	DIPEA	CH_3CN	N.D.
20.	-	-	CH_3CN	N.D.
21.[e]	Rose bengal	DIPEA	CH_3CN	N.D.

22.[f]	Rose bengal	DIPEA	CH_3CN	77
23.[g]	Rose bengal	DIPEA	CH_3CN	76
24.[h]	Rose bengal	DIPEA	CH_3CN	79
25.[i]	Rose bengal	DIPEA	CH_3CN	72

[a]Reaction Conditions: **1a** (0.1 mmol, 1 equiv), **2a** (1.5 equiv), Photoctalyst (PC) (5 mol%), base (1.5 equiv), and solvent (1 mL) under nitrogen atmosphere using blue LEDs (440 nm) for 3 h. [b]Isolated yield. [c]using 1.0 equiv of RAE, [d]using 1.2 equiv of RAE, [e]under dark, [f]with 1.2 equiv of DIPEA, [g]with 427 nm Blue LED, [h]with 456 nm blue LED, [i]with 3 mol% Rose bengal. N.D. = Not Detected.

2. Scheme S1: Scale-Up and Mechanistic Experiments:



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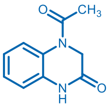
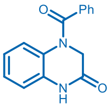
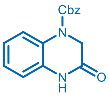
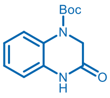
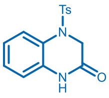










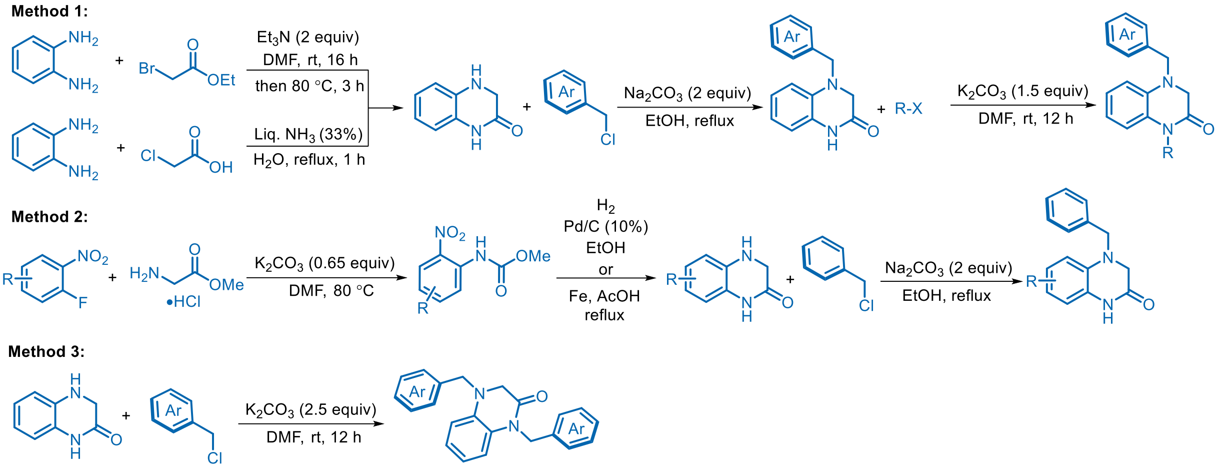
4. Experimental Section

4.1 General Information

Photoredox reactions were performed under an N_2 atmosphere for all the reaction sets using pre-dried glassware and standard Schlenk tubes. All the solvents were obtained from Merck (Emparta grade), dried with calcium hydride, and freshly distilled under Nitrogen. The following starting materials and the reaction components *N*-hydroxyphthalimide, aliphatic carboxylic acids, Benzyl chlorides, *N*,*N*'-Dicyclohexylcarbodiimide (DCC), 4-*N*,*N*-dimethylamino-pyridine (DMAP), K_2CO_3, Na_2CO_3, DIPEA, Et_3N, TMEDA, and TEMPO were obtained from commercial sources and used without further purification. 3,4-dihydroquinoxalin-2(1*H*)-one and phthalimide ester derivatives were synthesized by the synthetic procedures mentioned below. Yields refer to isolated compounds, estimated to be >95% pure as determined by ^1H NMR and ^{13}C NMR. All optimized reactions were conducted under the photo irradiation using 40W Kessil PR160L (Linear Reflector)- 440 nm lamp (Avg. Intensity in 2×4 cm area = 137 mW/cm^2) with 2 cm from the reaction tube made up of borosilicate glass without any filter. Thin layer chromatography (TLC) was performed on Merck pre-coated silica gel 60 F254 aluminum sheets with detection under UV light at 254 nm. Chromatographic separations were carried out on Avra silica gel (100-200 mesh or 230−400 mesh). Nuclear magnetic resonance (NMR) spectroscopy was performed using Bruker 500 MHz spectrometers. Chemical shifts (δ) are provided in ppm if not otherwise specified. HRMS spectra were recorded using Agilent 6500 Q-TOF spectrometer. UV experiment was performed on LABINDIA ANALYTICAL-2000 U UV/VIS Spectrophotometer instrument. The fluorescence experiments were recorded with a “JASCO FP-8300” Scientific Spectrofluorometer.

4.2 Starting Material Preparation

4.2.1 General Procedures (GP1) for the Synthesis of 3,4-dihydroquinoxalin-2(1*H*)-one derivatives[1]



Method 1:

Step-1: In a flask containing DMF (50 mL), *o*-phenylenediamine (5 g, 46.2 mmol) was dissolved, and then ethyl 2-bromoacetate (6.1 mL, 55.0 mmol, 1.2 equiv) and triethyl amine (12.9 mL, 92.5 mmol, 2.0 equiv) were added sequentially at 0°C. The resulting solution was stirred at room temperature for 16 hours, then heated to 80 °C for 3 hours. After removing most of the DMF by rotary evaporation, the residue was partitioned between 10 mL of water and 40 mL of EtOAc. The EtOAc layer was washed with 10 mL of saturated $NaHCO_3$ and 10 mL of brine, dried over Na_2SO_4, and concentrated under vacuum to obtain the crude residue. The residue was triturated with a mixture of CH_2Cl_2 and hexane (1:1 v/v), and the precipitate was filtered and dried under vacuum to yield the product (5.2 g, 76% yield; Rf = 0.31 in 50% EtOAc-hexane) as a beige powder.

or

Step-1: A mixture of *o*-phenylenediamine (10.0 g, 92 mmol), chloroacetic acid (8.7 g, 92 mmol) in aqueous ammonia (33%, 10 mL), and water (80 mL) was heated to reflux temperature for one hour. After cooling, a light brown solid was formed and filtered under reduced pressure. The solid was then dried at 110 °C to obtain an off-white solid. This method was found to be better than the previous one.

Step-2: The substituted quinoxalinone (1 equiv) and Na_2CO_3 (2 equiv) were added to a round-bottomed flask, followed by 96% EtOH (0.33 M) and the corresponding benzyl chloride (1.2 equiv). The resulting mixture was stirred at reflux temperature in an oil bath for 24-48 hours. After this time, the EtOH was removed via rotary evaporation, and the residue was dissolved in EtOAc (100 mL). The organic phase was washed with water (2x50 mL), and the combined aqueous phases were extracted with EtOAc (50 mL). The combined organic phases were dried over MgSO4, and the solvent evaporated. The resulting dark brown residue was washed with cold EtOH several times until a slightly brown solid was obtained. Purifying the product using column chromatography is not recommended, as this may result in partial decomposition.

Step 3: The substituted quinoxalinone (1 equiv) and K_2CO_3 (1.5 equiv) were added to a round-bottomed flask. Then, DMF (1 M) was added, followed by the corresponding benzyl chloride (1.2 equiv). The reaction mixture was stirred at room temperature for 12 h. After this period, DMF was removed by rotary evaporation, and the residue was dissolved in EtOAc. The organic phase was washed with water (2x50 mL), and the combined aqueous phases were extracted with EtOAc (50 mL). The combined organic phases were dried over $MgSO_4$, and the solvent evaporated. The residue was washed with cold EtOH several times until a slightly brown solid was obtained.

Method 2:

Step-1: In a round bottom flask, glycine methyl ester hydrochloride (1.1 equiv), potassium carbonate (0.65 equiv), and DMF (1 M) were placed. The resulting suspension was heated to 80 ^{o}C using an oil bath. At this temperature, the corresponding 2-fluoronitrobenzene (1 equiv) was added, and the resulting mixture was stirred for 4 h at 80 ^{o}C. When the starting material was consumed (as shown by TLC), the reaction mixture was allowed to reach room temperature and was diluted with EtOAc (30 mL). Most of the DMF was extracted with water (5 x 10 mL) and brine (10 mL). The organic phase was dried over $MgSO_4$, and the solvent was removed under reduced pressure. The residue was purified by column chromatography using hexane:dichloromethane mixtures as eluent to afford the corresponding intermediate.

Step-2 (reduction using H_2/Pd): To a mixture of the corresponding intermediate in EtOH (0.25 M) was added Pd/C (10% w/w, 30 mg/mmol), and the resulting mixture was stirred overnight at room temperature with a hydrogen-filled balloon. When the starting material was consumed, the reaction mixture was filtered through a pad of Celite to afford analytically pure dihydroquinoxalinones.

or

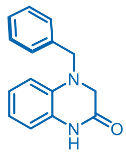
Step-2 (reduction using Fe) [only for the synthesis of quinoxalin-2-one bearing a Br substituent]: In a round-bottomed flask, the proper N-aryl glycinate (1 equiv) was dissolved in glacial acetic acid (0.33 M), and Fe (4 equiv) was added in portions. The resulting suspension was refluxed for 2-3 h and then poured into a 500 mL beaker containing crushed ice. AcOH was neutralized, and solid $NaHCO_3$ was added with continuous stirring until pH 8 was reached. After this, the brown suspension was vacuum filtered through a pad of Celite rinsing with water and EtOAc. The filtrate was transferred to a separation funnel, and the aqueous phase was extracted with EtOAc (x3); the combined organic layers were dried over $MgSO_4$, filtered, and concentrated to afford substituted quinoxalinone, was used in the next step without further purification.

Step-3: Same as step 2 of method 1.

Method-3:

To a solution of 3,4-dihydroquinoxalin-2-one (1 equiv) in DMF (1 M), K_2CO_3 (2.5 equiv) and alkyl bromide (2 equiv) were added successively, and the resulting solution was stirred overnight at room temperature. Then, the reaction mixture was diluted with EtOAc. The organic layer was washed with water (x3) and brine (x2). The organic layer was dried over $MgSO_4$, filtered, and concentrated. The residue was purified by column chromatography to afford 1,4-dialkyl-3,4-dihydroquinoxalin-2(1*H*)-one.

Various dihydroquinoxalinones **1a, 1b, 1c, 1d, 1e, 1f, 1g, 1h, 1i, 1p** and **1q** were prepared according to method 1. **1j, 1k** and **1l** were prepared by method 2. **1m**, and **1n** were prepared by method 3. **1r** has prepared by step 3 (method 1). **1o** has prepared using previous literature.[2]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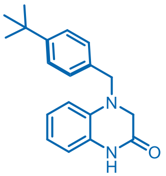
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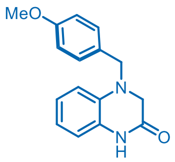
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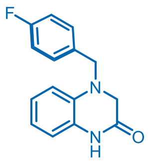
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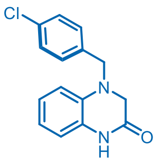
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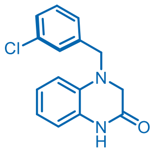
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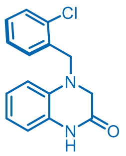
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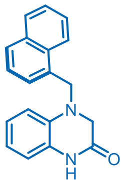
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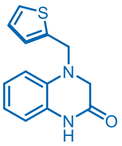
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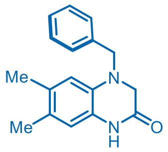
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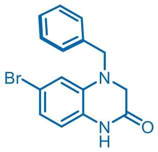
1k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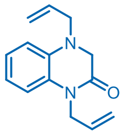
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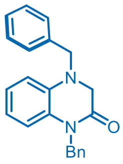
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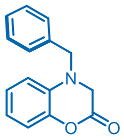
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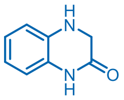
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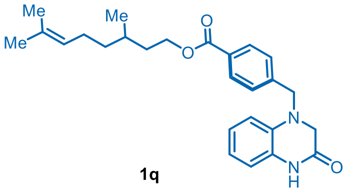
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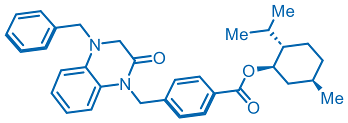
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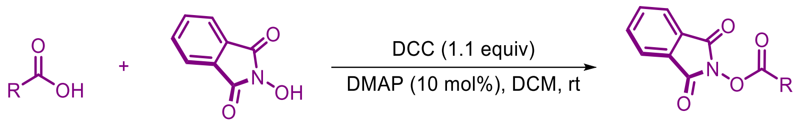
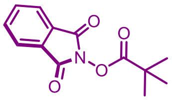
4.2.2 General Procedure (GP2) for the Preparation of NHPI Esters[3]



The corresponding alkyl carboxylic acid (10 mmol, 1.0 equiv), *N*-hydroxyphthalimide (11 mmol, 1.1 equiv), and 4-dimethylaminopyridine (1.0 mmol, 10 mol%) were mixed in a flask with a magnetic stirring bar. Dry DCM (40 mL) was added. Then a solution of N, N´-dicyclohexylcarbodiimide (11.0 mmol, 1.1 equiv) in DCM (15 mL) was added slowly at room temperature. The reaction mixture was monitored by TLC at room temperature. After completed, the white precipitate was filtered off and the solution was concentrated under vacuum. Corresponding redox active esters were purified by column chromatography on silica gel (DCM or Hexane/ethyl acetate as eluent).



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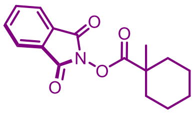

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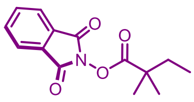

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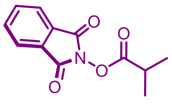

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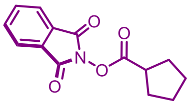

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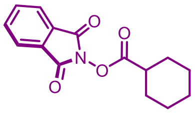

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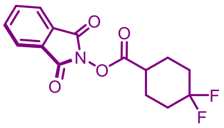

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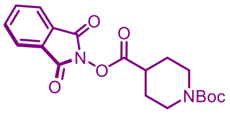

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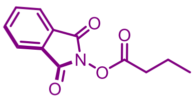

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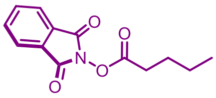

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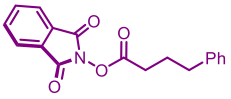

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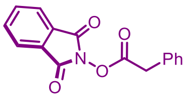

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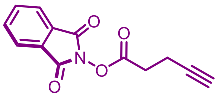

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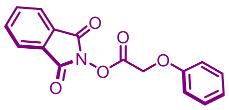

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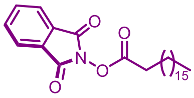

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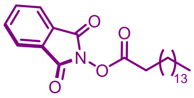

from Palmatic acid

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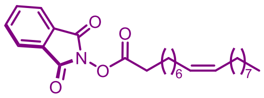

from oleic acid

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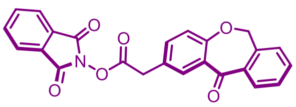

from Isoxepac

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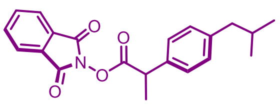

from Ibuprofen

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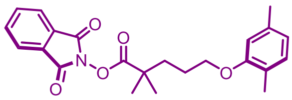
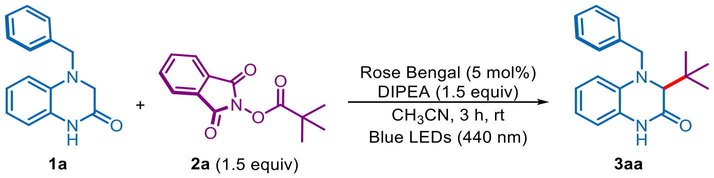


from gemfibrozil

2t

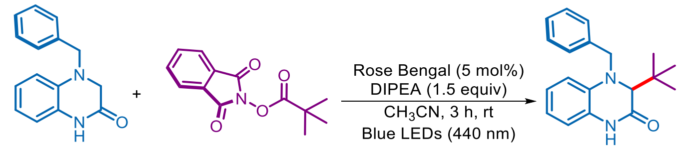
4.3 Representative General Procedure (GP3) for the visible light-mediated photoredox-catalyzed Reaction:

Dihydroquinoxalinone derivative **1** (0.25 mmol, 1 equiv), alkyl NHPI ester **2** (0.375 mmol, 1.5 equiv), rose bengal (5 mol%), and DIPEA (0.375 mmol, 1.5 equiv) were added in a pre-dried 10 ml Schlenk tube under N_2 atmosphere. The tube was degassed and purged with N_2 three times. Then, dry acetonitrile (2.5 mL) was added under the N_2 atmosphere. The resulting reaction mixture was allowed to stir for 3 h at room temperature under the irradiation of 40 W Kessil blue LED (440 nm) lamp. After completion (3 h), the reaction mixture was concentrated under vacuum and purified by silica gel column chromatography using 10-20% ethyl acetate in hexane (for non-polar compounds) and 10-20% acetone in hexane (for polar compounds) to afford the desired product **3aa**.



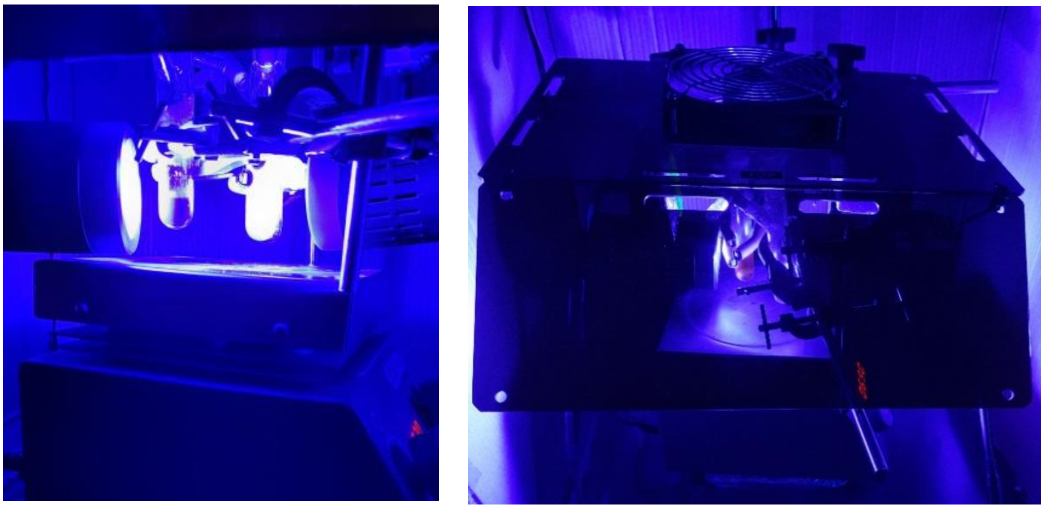
4.4 Gram scale synthesis of 2,4-dibenzyl-6-cyclohexyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione

4-Benzyl-3,4-dihydroquinoxalin-2(1*H*)-one **1a** (1 g, 4.2 mmol, 1 equiv), alkyl NHPI ester **2a** (1.5 g, 6.3 mmol, 1.5 equiv), rose bengal (214 mg, 5 mol%), and DIPEA (1052 µL, 6.3 mmol, 1.5 equiv) were added in a pre-dried 100 ml Schlenk flask under N_2 atmosphere. The flask was degassed and purged with N_2 three times. Then dry acetonitrile (40 mL) was added under the N_2 atmosphere. The resulting reaction mixture was allowed to stir for 3 h at room temperature under the irradiation of 40 W Kessil blue LED (440 nm) lamp. After completion, the reaction mixture was concentrated under vacuum, and purified by silica gel column chromatography using 10 to 20% acetone in hexane to afford the **3aa** as a white solid (1.2 g, 79%).



1a (1 g, 4.2 mmol) **2a** (1.5 g, 6.3 mmol) **3aa** (1.2 g, 79%)

4.5 Image of the photoinduced reaction setup

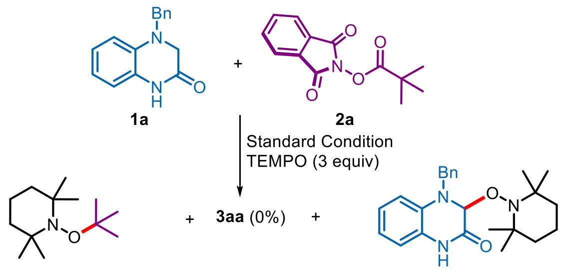


Front view: **Top view:**

Figure S1: Representative pictures of reaction setup.

4.6 Procedure for the Control Experiments

(a)



(m/z) calcd for **[4+H]**$^+$ = 214.2165
Found = 214.2164

(m/z) calcd for **[5+H]**$^+$ = 394.2489
Found = 394.2483

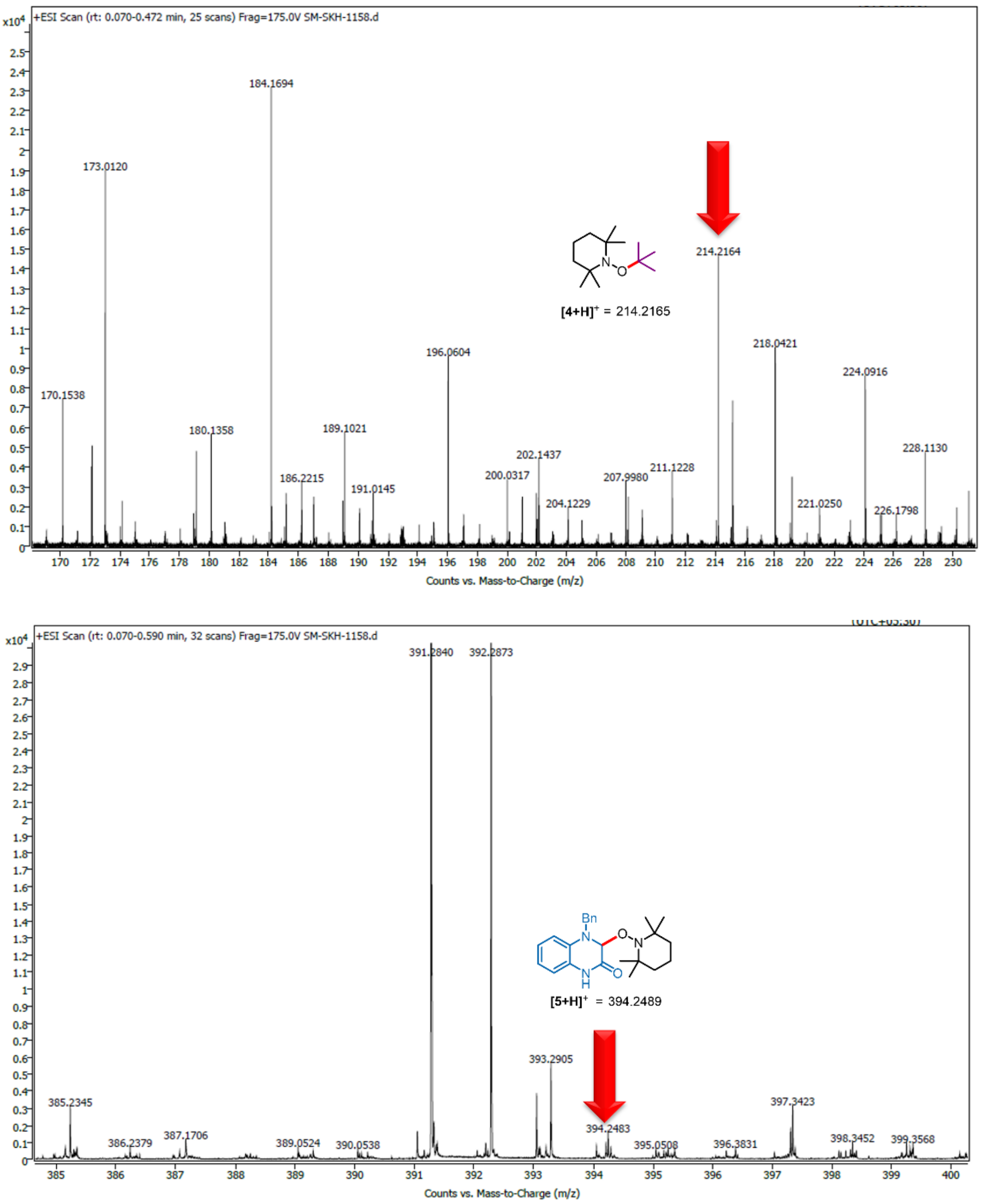
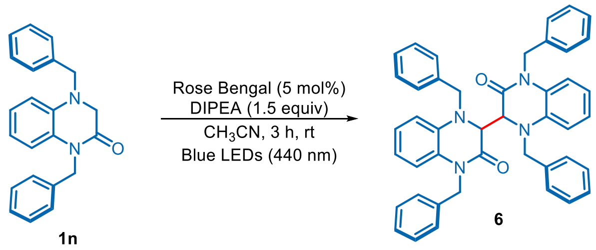


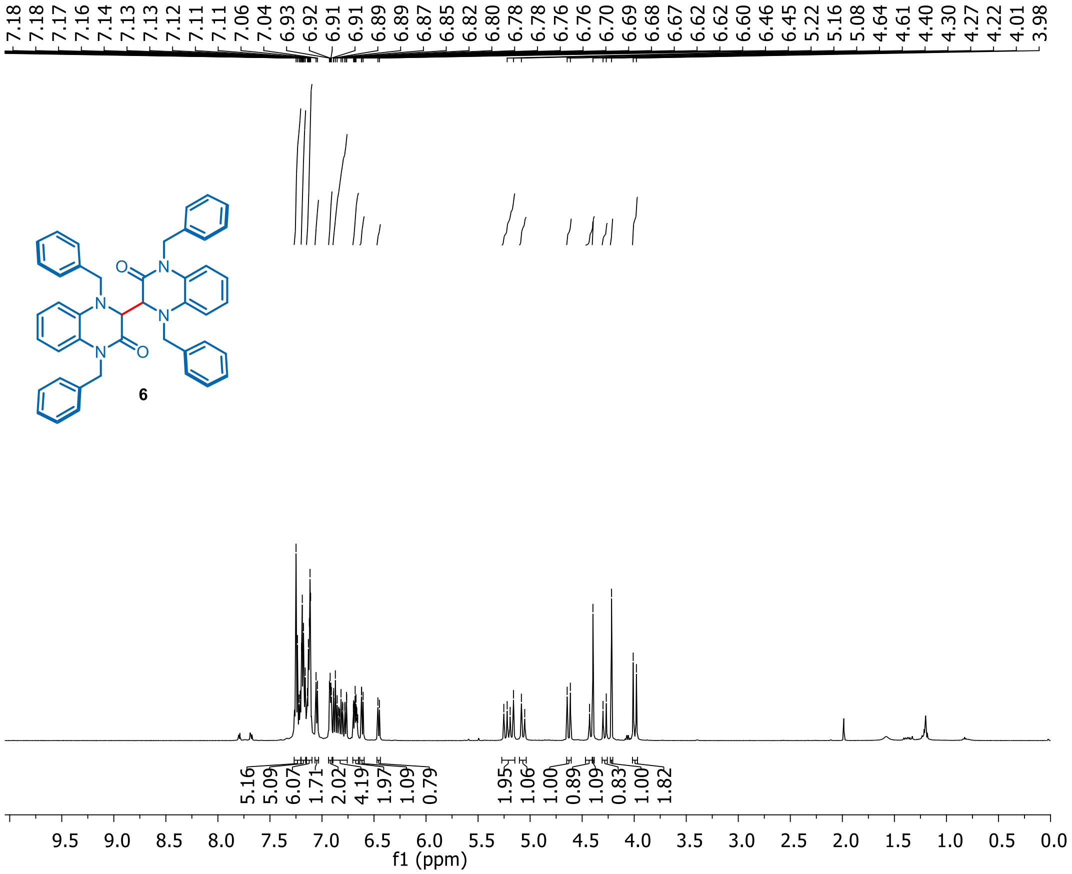
Figure S2: HRMS Data of TEMPO Adduct.

(a) 4-Benzyl-3,4-dihydroquinoxalin-2(1*H*)-one **1a** (0.059 g, 0.25 mmol), alkyl NHPI ester **2a** (0.093 g, 0.375 mmol), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol), 2,2,6,6-Tetramethylpiperidin-1- yloxyl (TEMPO, 0.75 mmol, 3.0 equiv) were added in a pre-dried 10 ml Schlenk tube under N_2 atmosphere. The tube was degassed and purged with N_2 three times. Then dry acetonitrile (2.5 ml) was added under the N_2 atmosphere. Then, the mixture was allowed to stir for 5 h under irradiation of a 40 W Kessil blue LED (440 nm) lamp. After completion, the reaction mixture was concentrated under vacuum and purified by silica gel column chromatography using 10% ethyl acetate in hexane to afford the adduct. HRMS confirmed the formation of the adduct.

(b)



^{1}H NMR spectrum of 6 (500 MHz, CDCl$_3$)



$^{13}C\{^1H\}$ NMR spectrum of 6 (126 MHz, $CDCl_3$)

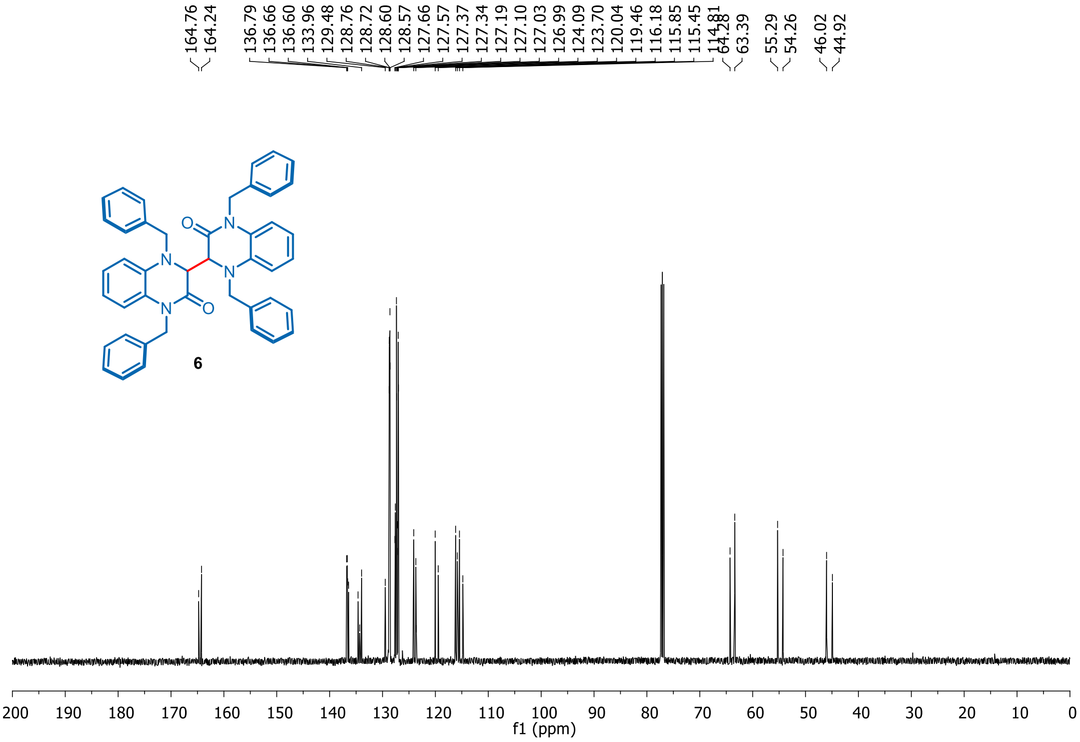


Figure S3: Dimer Adduct **6** without NHPI ester.

(b) 1,4-Dibenzyl-3,4-dihydroquinoxalin-2(1*H*)-one **1n** (0.082 g, 0.25 mmol, 1 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol), were added in a pre-dried 10 ml Schlenk flask under N_2 atmosphere. The flask was degassed and purged with N_2 three times. Then, dry acetonitrile (2.5 mL) was added under the N_2 atmosphere. The resulting reaction mixture was allowed to stir for 3 h at room temperature under the irradiation of 40 W Kessil blue LED (440 nm) lamp. After completion, the reaction mixture was concentrated under vacuum and purified by silica gel column chromatography using 10% ethyl acetate in hexane to afford dimer **6** (0.111 g, 68%) as a white solid. m.p: 148 – 150 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 – 7.20 (m, 5H), 7.20 – 7.16 (m, 5H), 7.15 – 7.10 (m, 6H), 7.05 (d, *J* = 6.6 Hz, 2H), 6.92 (dd, *J* = 6.5, 2.9 Hz, 2H), 6.89 – 6.76 (m, 4H), 6.70 – 6.65 (m, 2H), 6.64 – 6.59 (m, 1H), 6.46 (d, *J* = 7.3 Hz, 1H), 5.26 – 5.15 (m, 2H), 5.07 (d, *J* = 16.0 Hz, 1H), 4.63 (d, *J* = 15.4 Hz, 1H), 4.43 (s, 1H), 4.40 (s, 1H), 4.28 (d, *J* = 15.6 Hz, 1H), 4.22 (s, 1H), 3.99 (d, *J* = 15.4 Hz, 2H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 164.7, 164.2, 136.7, 136.66, 136.60, 136.4, 134.6, 134.3, 133.9, 129.4, 128.76, 128.72, 128.6, 128.5, 127.6, 127.5, 127.37, 127.34, 127.19, 127.10, 127.0, 126.9, 124.0, 123.7, 120.0, 119.4, 116.1, 115.8, 115.4, 114.8, 64.2, 63.3, 55.2, 54.2, 46.0, 44.9. **HRMS-ESI** (*m/z*): calcd for $C_{44}H_{39}N_4O_2$ $[M+H]^+$ 655.3068; found 655.3072.

4.7 Light on-off experiment:

4-Benzyl-3,4-dihydroquinoxalin-2(1*H*)-one **1a** (0.059 g, 0.25 mmol), alkyl NHPI ester **2a** (0.093 g, 0.375 mmol), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol) were added in a pre-dried 10 ml Schlenk tube under N_2 atmosphere. The tube was degassed and purged with N_2 three times. Then dry acetonitrile (3 ml) was added under the N_2 atmosphere. The mixture was irradiated using a 40 W Kessil blue LED (440 nm) lamp and the reaction was placed in light and dark in every alternative 10 min. After every time interval of 10 min, a 0.5 ml reaction aliquot was taken out by a syringe and quenched with water, organic part was taken in ethyl aceteate and NMR was carried out. The NMR yield was determined using mesitylene as an internal standard.

Entry	Time (min)	Light Source	Yield (%)
1	10	On	6
2	20	Off	6
3	30	On	15
4	40	Off	15
5	50	On	25
6	60	Off	25

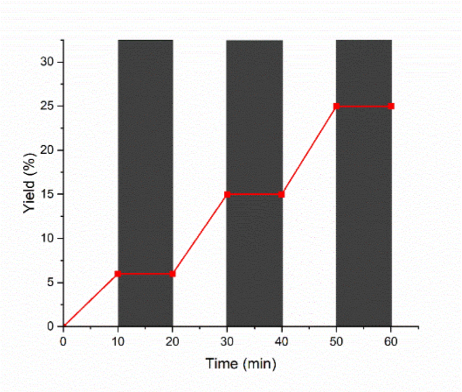


Figure S4: Light on-off Experiment.

4.8 Quantum Yield Calculation[4]

(A) Determination of light intensity of the Blue LED:

0.737 g of potassium ferrioxalate trihydrate was dissolved in 10 mL H_2SO_4 (0.05 M) and stored in the dark. Then, a buffer solution was prepared by dissolving 2.5 g of sodium acetate and 0.5 mL of H_2SO_4 (95-98%) in 50 mL of distilled water.

General Protocol to assess the photon flux of the 440 nm nm blue LEDs: To a 10 mL Schlenk flask containing a stirring bar, 1 mL of the actinometer solution was added. Then, the solution was irradiated for 60 s. Immediately, a 100 µL aliquot was taken and added to a 10 mL volumetric flask containing 15 mg of 1, 10-phenanthroline in 3 mL of the buffer solution. The flask was filled with distilled water. The absorbance of this solution was then measured at 510 nm by UV/Vis spectrophotometry. In a similar manner, this procedure is repeated with the actinometer solution stored in the dark. Using then the Beer's Law, the number of moles of Fe^{2+} produced by light irradiation is obtained by:

$$Fe^{2+} = \frac{v_1 v_3 \Delta A(510\ \text{nm})}{10^3 v_2 l \varepsilon} \qquad \text{..................}1$$

Where:

v_1 = Irradiated volume (1 mL).

v_2 = The aliquot of the irradiated solution taken for the estimation of Fe^{+} ions (0.100 mL).

v_3 = Final volume of the solution after complexation with 1, 10-phenanthroline (10 mL).

ε (510 nm) = Molar extinction coefficient of $[Fe\ (Phen)_3]^{2+}$ complex (11100 L mol^{-1} cm^{-1}).

l = Optical path-length of the cuvette (1 cm).

ΔA (510 nm) = absorbance difference between the irradiated solution and the solution stored in dark.

$$Fe^{2+} = \frac{1\text{ml} \times 10\ \text{ml} \times 3.1509(510\ \text{nm})}{10^3 \times 0.1\text{L} \times 1\text{cm} \times 11100\ \text{L mol}^{-1}\ \text{cm}^{-1}}$$
$$= 2.81 \times 10^{-8}\ \text{mol}$$

The photon flux (F) is obtained by using the following equation:

$$\phi(\lambda) = \frac{mol\ Fe^{2+}}{F(1-10^{-A\lambda})}$$

$$F = 2.81 \times 10^{-8}\ \text{einsteins/s}$$

Where: Φ (λ) = The quantum yield for Fe^{2+} formation at 440 nm is 1.

A (λ) = ferrioxalate actinometer absorbance at 440 nm, which was measured placing 1 mL of the solution in a cuvette of path length 1 cm by UV/Vis spectrophotometry. We obtained an absorbance value of 3.1501. The photon flux (F) is 2.81 x 10^{-8} einsteins/s.

(B) Quantum Yield Calculation:

4-Benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.043 g, 0.25 mmol), 0.059 g, 0.25 mmol), alkyl NHPI ester **2a** (0.093 g, 0.375 mmol), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol) were added in a pre-dried 10 ml Schlenk tube under N_2 atmosphere. The tube was degassed and purged with N_2 three times. Then dry acetonitrile (2.5 ml) was added under the N_2 atmosphere. The mixture was irradiated using a 40 W Kessil blue LED (440 nm) lamp in the optimized condition for 600s and $1.9*10^{-5}$ moles of product were obtained. The Quantum yield was calculated using the following equations;

$$\phi\ (440\ \text{nm}) = \frac{mol\ of\ product}{F(1-10^{-A(440\ \text{nm})})t}$$

$$\phi\ (440\ \text{nm}) = \frac{1.9 \times 10^{-5}\ \text{mol}}{2.81 \times 10^{-8}\ \text{mo} \times 1 \times 600\text{s}}$$

$$= \mathbf{0.113}$$

Where: A (440 nm) = is the absorbance at 440 nm of the photocatalytic reaction which was measured placing 1 mL of the solution in a cuvette of path length 1 cm by UV/Vis spectrophotometry.

t = is the reaction time i.e., 600s.

The quantum yield (Φ) of the reaction is **0.113**.

4.9 Luminescence Quenching Experiment:

(A) Preparation of the Stock Solution: A 0.5 mM solution of the Rose bengal was prepared in a sample vial by dissolving 1.27 mg of the Rose bengal in 10 mL of acetonitrile (spectroscopic grade, purchased from Spectrochem). The freshly prepared solution was used for the spectroscopic measurement. The required amount was taken using a micropipette from the mother solution as an aliquot and it was diluted further by dissolving in 3 mL of acetonitrile in the cuvette. Similarly, 10 mL 0.1 M solution of 1,3-dioxoisoindolin-2-yl pivalate **2a** and DIPEA were prepared by dissolving the requisite amount of each substrate in acetonitrile. Freshly prepared solutions were used for the quenching experiment.

(B) Quenching studies: Fluorescence emission spectra of the Rose bengal in presence of different reactions components (**1a**, **2a**, DIPEA) were recorded and analyzed in detail to estimate the light emission properties of the pure Rose bengal system and their distractions by external interference from the substrates. Emission intensities of Rose bengal were recorded with a “JASCO FP-8300” Scientific Spectrofluorometer using a 10.0 mm quartz cuvette. The Rose bengal exhibits an absorption maximum at 562 nm. The sample solution of Rose bengal with a proper concentration of 25 µM in acetonitrile was excited with a wavelength of 440 nm, and the emission maxima were found to be observed from 550 - 650 nm. To study the quenching behaviour of Rose bengal, different concentration of **1a** was added to the solution and the emission spectra were measured following the aforementioned procedure. The quenching effect **1a** was quite significant on the photocatalyst; the intensity of the emission maxima decreased gradually upon increasing the concentration of **1a**. Some sets of solutions with different concentrations of the **1a** were used; the experiment was repeated and finally, the Stern-Volmer plot was depicted. On the other hand, no significant change in the emission maxima of Rose bengal was observed when alkyl NHPI ester **2a** and DIPEA was used as the quencher. The corresponding Stern-Volmer plot was drawn for all the cases.

(C)

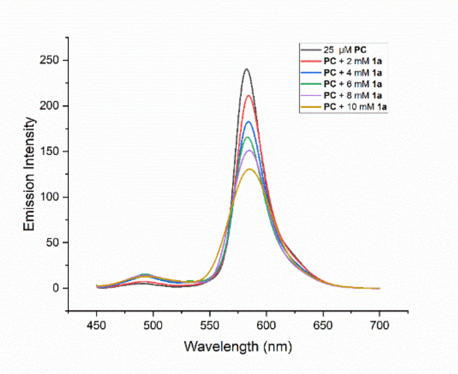


Figure S5: Luminescence spectra of Rose bengal (25 µM) as a function of concentration of 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (**1a)** in CH_3CN with excitation at 440 nm.

(D)

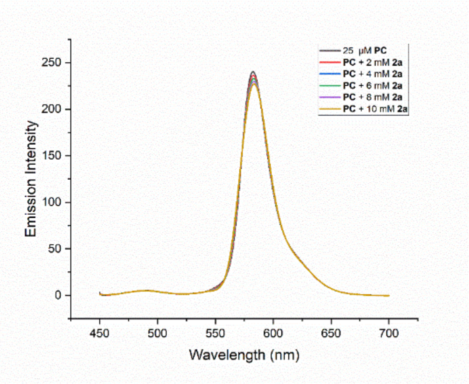


Figure S6: Luminescence spectra of Rose bengal (25 µM) as a function of concentration of NHPI ester **2a** in CH_3CN with excitation at 440 nm.

(E)

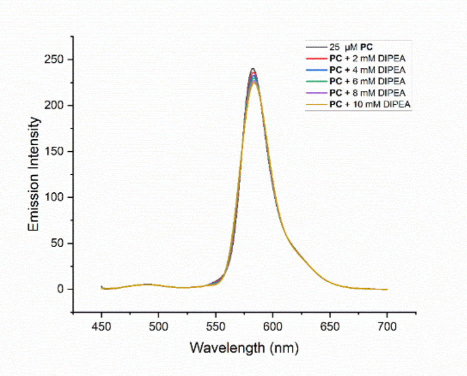


Figure S7: Luminescence spectra of Rose bengal (25 µM) as a function of concentration of DIPEA in CH_3CN with excitation at 440 nm.

(F)

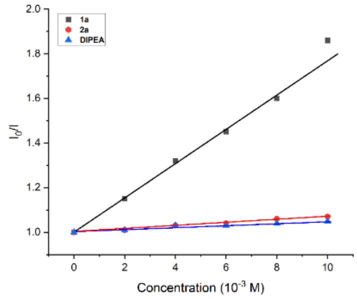
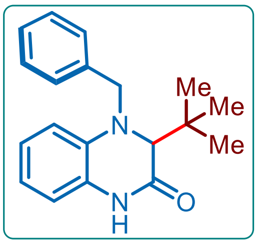


Figure S8: Stern-Volmer Plot.

4.10 Report of NMR Spectra:

4-Benzyl-3-(*tert*-butyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3aa)

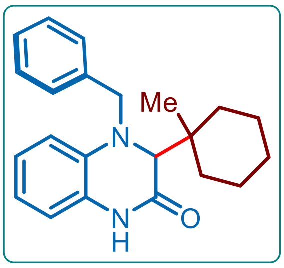


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3aa** (0.066 g, 90% yield) as a white solid. m.p: 130 – 132 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.75 (s, 1H), 7.24 – 7.18 (m, 2H), 7.18 – 7.09 (m, 3H), 6.89 – 6.83 (m, 1H), 6.76 (d, *J* = 8.0 Hz, 1H), 6.67 (d, *J* = 4.1 Hz, 2H), 4.91 (d, *J* = 15.8 Hz, 1H), 4.34 (d, *J* = 15.8 Hz, 1H), 3.67 (s, 1H), 0.93 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.5, 137.5, 134.6, 128.6, 127.6, 127.4, 127.2, 123.9, 118.9, 115.1, 115.0, 71.3, 57.7, 39.5, 27.5. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{23}N_2O$ $[M+H]^+$ 295.1805; found 295.1815.

4-Benzyl-3-(1-methylcyclohexyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ab)

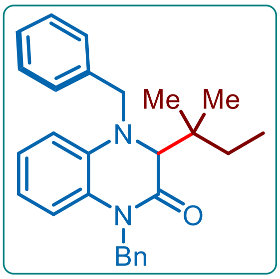


Synthesized according to the GP3 using 1,4-dibenzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.082 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 1-methylcyclohexane-1-carboxylate (0.108 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ab** (0.067 g, 80% yield) as a white solid. m.p: 120 – 122 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.49 (s, 1H), 7.24 – 7.20 (m, 2H), 7.19 – 7.15 (m, 1H), 7.14 (d, *J* = 7.3 Hz, 2H), 6.91 – 6.86 (m, 1H), 6.80 (d, *J* = 8.0 Hz, 1H), 6.73 – 6.66 (m, 2H), 4.96 (d, *J* = 15.8 Hz, 1H), 4.36 (d, *J* = 15.9 Hz, 1H), 3.74 (s, 1H), 1.47 – 1.36 (m, 7H), 1.18 – 1.10 (m, 3H), 0.88 (s, 3H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.2, 137.6, 135.1, 128.6, 127.6, 127.4, 127.2, 123.9, 118.9, 115.3, 115.1, 58.2, 42.5, 34.9, 34.7, 26.0, 21.8, 21.4. **HRMS-ESI** (*m/z*): calcd for $C_{22}H_{27}N_2O$ $[M+H]^+$ 335.2118; found 335.2125.

1,4-Dibenzyl-3-(*tert*-pentyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ac)

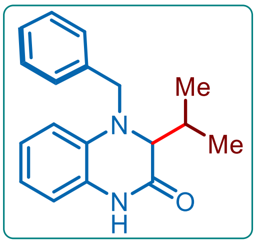


Synthesized according to the GP3 using 1,4-dibenzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.082 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 2,2-dimethylbutanoate (0.098 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ac** (0.085 g, 85% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.32 – 7.27 (m, 2H), 7.26 (s, 1H), 7.26 – 7.19 (m, 5H), 7.15 – 7.11 (m, 2H), 6.90 – 6.85 (m, 2H), 6.84 – 6.80 (m, 1H), 6.70 – 6.66 (m, 1H), 5.20 (q, *J* = 7.5 Hz, 2H), 4.93 (d, *J* = 15.1 Hz, 1H), 4.34 (d, *J* = 15.1 Hz, 1H), 3.93 (s, 1H), 1.48 – 1.34 (m, 2H), 0.86 (t, *J* = 7.5 Hz, 3H), 0.85 (s, 3H), 0.84 (s, 3H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 165.4, 137.6, 137.3, 136.2, 130.8, 128.6, 128.5, 127.45, 127.42, 127.17, 127.14, 123.6, 119.1, 115.9, 115.3, 70.4, 58.0, 46.1, 42.3, 32.2, 24.1, 23.7, 8.2. **HRMS-ESI** (*m/z*): calcd for $C_{27}H_{31}N_2O$ $[M+H]^+$ 399.2431; found 399.2442.

4-Benzyl-3-isopropyl-3,4-dihydroquinoxalin-2(1*H*)-one (3ad)

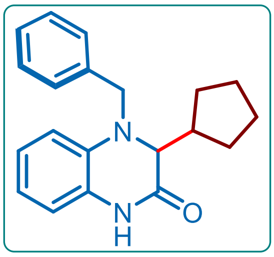


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl isobutyrate (0.087 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ad** (0.048 g, 68% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.57 (s, 1H), 7.31 – 7.27 (m, 2H), 7.25 – 7.21 (m, 3H), 6.93 – 6.87 (m, 1H), 6.76 – 6.72 (m, 3H), 4.77 (d, *J* = 15.4 Hz, 1H), 4.36 (d, *J* = 15.4 Hz, 1H), 3.64 (d, *J* = 7.0 Hz, 1H), 2.04 – 1.95 (m, 1H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.93 (d, *J* = 6.8 Hz, 3H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 167.5, 137.3, 134.3, 128.8, 127.63, 127.60, 126.9, 124.1, 119.0, 115.4, 114.2, 68.2, 55.4, 31.3, 20.4, 18.9. **HRMS-ESI** (*m/z*): calcd for $C_{18}H_{21}N_2O$ $[M+H]^+$ 281.1648; found 281.1658.

4-Benzyl-3-cyclopentyl-3,4-dihydroquinoxalin-2(1*H*)-one (3ae)

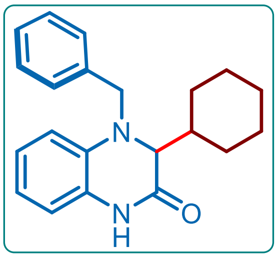


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl cyclopentanecarboxylate (0.097 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ae** (0.049 g, 76% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.46 (s, 1H), 7.32 – 7.27 (m, 4H), 7.26 – 7.21 (m, 1H), 6.94 – 6.90 (m, 1H), 6.81 – 6.76 (m, 2H), 6.73 (d, *J* = 8.0 Hz, 1H), 4.72 (d, *J* = 15.1 Hz, 1H), 4.35 (d, *J* = 15.1 Hz, 1H), 3.70 (d, *J* = 8.6 Hz, 1H), 2.08 – 2.00 (m, 1H), 1.82 – 1.76 (m, 1H), 1.60 – 1.56 (m, 3H), 1.46 – 1.36 (m, 4H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 167.9, 137.1, 134.2, 128.7, 127.6, 127.4, 127.0, 123.9, 119.1, 115.2, 114.5, 66.2, 54.8, 41.7, 31.1, 29.0, 24.5, 24.4. **HRMS-ESI** (*m/z*): calcd for $C_{20}H_{23}N_2O$ [M+H]$^+$ 307.1805; found 307.1810.

4-Benzyl-3-cyclohexyl-3,4-dihydroquinoxalin-2(1*H*)-one (3af)

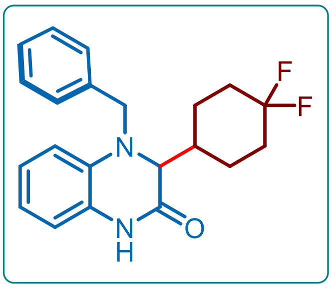


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl cyclohexanecarboxylate (0.102 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3af** (0.062 g, 78% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.92 (s, 1H), 7.30 – 7.26 (m, 2H), 7.26 – 7.20 (m, 3H), 6.92 – 6.88 (m, 1H), 6.79 – 6.71 (m, 3H), 4.78 (d, *J* = 15.4 Hz, 1H), 4.36 (d, *J* = 15.4 Hz, 1H), 3.68 (d, *J* = 6.5 Hz, 1H), 1.74 – 1.64 (m, 5H), 1.59 (s, 1H), 1.15 – 1.07 (m, 5H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 167.2, 137.1, 134.1, 128.6, 127.53, 127.50, 126.8, 123.9, 118.9, 115.3, 114.1, 67.6, 55.4, 40.8, 30.3, 29.3, 26.1, 25.99, 25.96. **HRMS-ESI** (*m/z*): calcd for $C_{21}H_{25}N_2O$ [M+H]$^+$ 321.1961; found 321.1966.

4-Benzyl-3-(4,4-difluorocyclohexyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ag)



Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 4,4-difluorocyclohexane-1-carboxylate (0.116 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ag** (0.069 g, 78% yield) as a white solid. m.p: 115 – 117 °C.

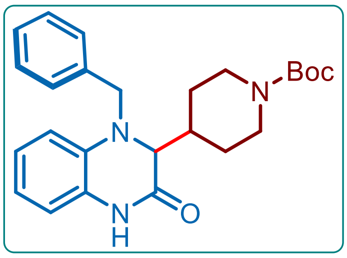
^{1}H NMR (500 MHz, CDCl$_3$) δ 8.92 (s, 1H), 7.32 – 7.27 (m, 2H), 7.25 – 7.21 (m, 3H), 6.96 – 6.93 (m, 1H), 6.81 – 6.78 (m, 3H), 4.76 (d, *J* = 15.3 Hz, 1H), 4.34 (d, *J* = 15.3 Hz, 1H), 3.71 (d, *J* = 7.1 Hz, 1H), 2.08 – 2.01 (m, 2H), 1.83 – 1.65 (m, 4H), 1.64 – 1.52 (m, 2H), 1.51 – 1.47 (m, 1H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.6, 136.9, 133.8, 128.7, 127.7, 127.5, 126.8, 124.2, 119.6, 115.5, 115.1, 66.0, 56.0, 38.6, δ 33.3 (d, *J* = 14.1 Hz), 33.1 (dd, *J* = 14.0, 2.3 Hz), 33.0 (d, *J* = 14.2 Hz), 26.1 (d, *J* = 9.6 Hz), 25.3 (d, *J* = 9.7 Hz).

^{19}F{^{1}H} NMR (471 MHz, CDCl$_3$) δ -91.9 (d, *J* = 235.9 Hz), -102.3 (d, *J* = 232.8 Hz).

HRMS-ESI (*m/z*): calcd for $C_{21}H_{23}F_2N_2O$ [M+H]$^+$ 357.1773; found 357.1771.

Tert-butyl 4-(1-benzyl-3-oxo-1,2,3,4-tetrahydroquinoxalin-2-yl)piperidine-1-carboxylate (3ah)

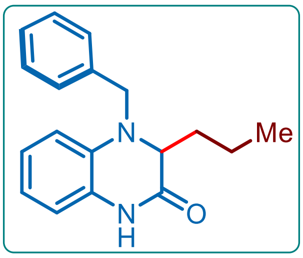


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1-(*tert*-butyl) 4-(1,3-dioxoisoindolin-2-yl) piperidine-1,4-dicarboxylate (0.140 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ah** (0.08 g, 76% yield) as a white solid. m.p: 98 – 100 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.81 (s, 1H), 7.28 (d, *J* = 6.3 Hz, 2H), 7.25 – 7.22 (m, 3H), 6.95 – 6.91 (m, 1H), 6.80 – 6.77 (m, 3H), 4.76 (d, *J* = 15.2 Hz, 1H), 4.34 (d, *J* = 15.2 Hz, 1H), 4.10 – 4.02 (m, 2H), 3.68 (d, *J* = 7.2 Hz, 1H), 2.55 – 2.48 (m, 2H), 1.82 – 1.75 (m, 1H), 1.68 – 1.64 (m, 1H), 1.61 – 1.56 (m, 1H), 1.42 (s, 9H), 1.32 – 1.27 (m, 2H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.5, 154.6, 136.7, 133.7, 128.7, 127.7, 127.6, 126.8, 124.2, 119.6, 115.5, 114.9, 79.5, 66.6, 55.9, 43.6, 43.5, 38.9, 29.2, 28.4, 26.9. **HRMS-ESI** (*m/z*): calcd for $C_{25}H_{31}N_3NaO_3$ $[M+Na]^+$ 444.2258; found 444.2254.

4-Benzyl-3-propyl-3,4-dihydroquinoxalin-2(1*H*)-one (3ai)

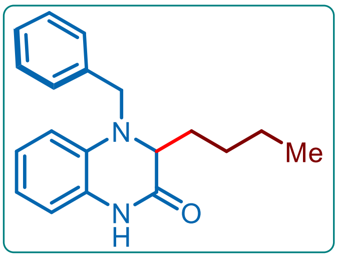


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl butyrate (0.087 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ai** (0.046 g, 66% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 9.10 (s, 1H), 7.37 – 7.26 (m, 5H), 6.94 (dd, *J* = 6.1, 3.9 Hz, 1H), 6.85 – 6.77 (m, 2H), 6.75 – 6.69 (m, 1H), 4.69 (d, *J* = 15.0 Hz, 1H), 4.31 (d, *J* = 15.0 Hz, 1H), 3.98 – 3.83 (m, 1H), 1.69 – 1.57 (m, 2H), 1.45 – 1.39 (m, 2H), 0.90 – 0.87 (m, 3H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 168.8, 137.0, 134.2, 128.7, 127.6, 127.5, 126.3, 124.0, 118.9, 115.3, 113.7, 61.8, 53.1, 31.4, 19.0, 14.0. **HRMS-ESI** (*m/z*): calcd for $C_{18}H_{21}N_2O$ $[M+H]^+$ 281.1648; found 281.1654.

4-Benzyl-3-butyl-3,4-dihydroquinoxalin-2(1*H*)-one (3aj)

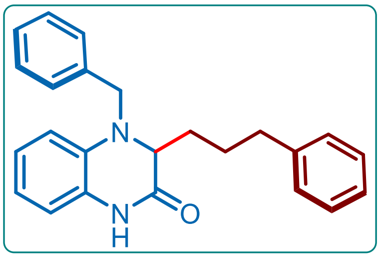


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pentanoate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3aj** (0.047 g, 64% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.54 (s, 1H), 7.26 – 7.23 (m, 3H), 7.19 (s, 2H), 6.87 – 6.82 (m, 1H), 6.69 (d, *J* = 3.9 Hz, 2H), 6.62 (d, *J* = 8.0 Hz, 1H), 4.58 (d, *J* = 15.1 Hz, 1H), 4.20 (d, *J* = 15.1 Hz, 1H), 3.81 – 3.76 (m, 1H), 1.61 – 1.45 (m, 3H), 1.29 – 1.21 (m, 3H), 0.76 (t, *J* = 7.2 Hz, 3H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 168.5, 137.0, 134.2, 128.7, 127.6, 127.5, 126.3, 124.0, 118.9, 115.2, 113.7, 62.0, 53.1, 28.9, 27.7, 22.6, 13.8. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{23}N_2O$ $[M+H]^+$ 295.1805; found 295.1810.

4-Benzyl-3-(3-phenylpropyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ak)

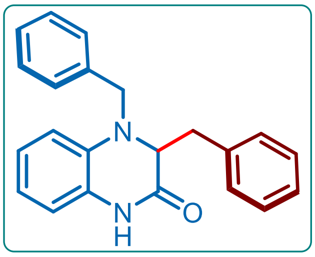


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 4-phenylbutanoate (0.116 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ak** (0.053 g, 60% yield) as a white solid. m.p: 109 – 111 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.75 (s, 1H), 7.23 (dd, *J* = 7.4, 3.9 Hz, 2H), 7.20 (s, 2H), 7.19 – 7.16 (m, 3H), 7.10 (d, *J* = 7.3 Hz, 1H), 7.02 (d, *J* = 7.2 Hz, 2H), 6.87 – 6.83 (m, 1H), 6.71 (d, *J* = 3.8 Hz, 2H), 6.64 (d, *J* = 8.0 Hz, 1H), 4.54 (d, *J* = 15.0 Hz, 1H), 4.14 (d, *J* = 15.0 Hz, 1H), 3.83 – 3.79 (m, 1H), 2.51 – 2.44 (m, 2H), 1.67 – 1.15 (m, 3H), 1.12 – 1.04 (m, 1H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 168.4, 141.7, 136.7, 133.9, 128.7, 128.4, 128.3, 127.69, 127.60, 126.3, 125.8, 124.1, 119.2, 115.4, 113.9, 61.7, 53.2, 35.5, 28.6, 27.1. **HRMS-ESI** (*m/z*): calcd for $C_{24}H_{25}N_2O$ [M+H]$^+$ 357.1961; found 357.1966.

3,4-Dibenzyl-3,4-dihydroquinoxalin-2(1*H*)-one (3al)

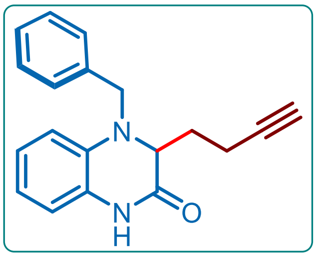


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 2-phenylacetate (0.105 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3al** (0.074 g, 90% yield) as a white solid. m.p: 124 – 126 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 9.11 (s, 1H), 7.27 (dd, *J* = 3.9, 1.6 Hz, 2H), 7.25 – 7.21 (m, 4H), 7.14 – 7.10 (m, 2H), 7.09 – 7.05 (m, 2H), 7.01 – 6.95 (m, 1H), 6.82 – 6.79 (m, 2H), 6.71 (d, *J* = 8.0 Hz, 1H), 4.40 (d, *J* = 15.0 Hz, 1H), 4.17 – 4.13 (m, 1H), 3.76 (d, *J* = 15.0 Hz, 1H), 2.95 – 2.84 (m, 2H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 168.2, 137.4, 136.6, 133.6, 129.6, 128.6, 128.5, 127.7, 127.4, 126.8, 126.4, 124.2, 119.1, 115.5, 114.1, 63.3, 53.4, 36.0. **HRMS-ESI** (*m/z*): calcd for $C_{22}H_{21}N_2O$ [M+H]$^+$ 329.1648; found 329.1653.

4-Benzyl-3-(but-3-yn-1-yl)-3,4-dihydroquinoxalin-2(1*H*)-one (3am)

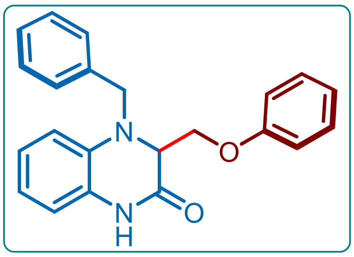


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pent-4-ynoate (0.091 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3am** (0.045 g, 62% yield) as a greenish viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.99 (s, 1H), 7.34 – 7.27 (m, 5H), 6.93 (t, *J* = 7.7 Hz, 1H), 6.80 (t, *J* = 7.5 Hz, 1H), 6.75 (t, *J* = 7.0 Hz, 2H), 4.68 (d, *J* = 15.1 Hz, 1H), 4.32 (d, *J* = 15.1 Hz, 1H), 4.04 – 3.98 (m, 1H), 2.29 – 2.24 (m, 2H), 1.95 (t, *J* = 2.5 Hz, 1H), 1.88 – 1.79 (m, 2H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 167.8, 136.8, 133.6, 128.7, 127.6 (2C), 126.3, 124.2, 119.5, 115.4, 114.5, 82.9, 69.3, 60.7, 53.5, 28.1, 15.1. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{19}N_2O$ [M+H]$^+$ 291.1492; found 291.1496.

4-Benzyl-3-(phenoxymethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3an)

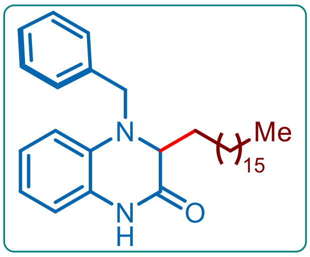


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 2-phenoxyacetate (0.111 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3an** (0.064 g, 74% yield) as a white solid. m.p: 142 – 144 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.94 (s, 1H), 7.34 – 7.28 (m, 4H), 7.25 – 7.19 (m, 3H), 6.93 (t, *J* = 7.4 Hz, 2H), 6.82 – 6.76 (m, 2H), 6.75 – 6.69 (m, 3H), 4.74 (d, *J* = 15.3 Hz, 1H), 4.51 (d, *J* = 15.3 Hz, 1H), 4.39 – 4.35 (m, 1H), 4.24 – 4.18 (m, 1H), 4.17 – 4.12 (m, 1H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.5, 158.0, 136.9, 134.1, 129.4, 128.7, 127.6, 127.5, 125.7, 124.3, 121.2, 118.9, 115.3, 114.5, 113.4, 66.9, 61.8, 53.3. **HRMS-ESI** (*m/z*): calcd for $C_{22}H_{21}N_2O_2$ [M+H]$^+$ 345.1598; found 345.1605.

4-Benzyl-3-heptadecyl-3,4-dihydroquinoxalin-2(1*H*)-one (3ao)

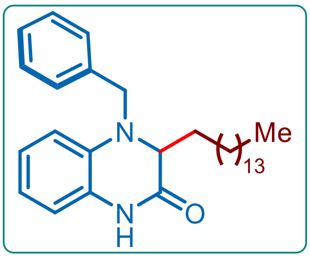


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl stearate (0.161 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ao** (0.079 g, 66% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.61 (s, 1H), 7.27 – 7.23 (m, 3H), 7.22 – 7.17 (m, 2H), 6.88 – 6.83 (m, 1H), 6.74 – 6.67 (m, 3H), 4.57 (d, *J* = 14.9 Hz, 1H), 4.21 (d, *J* = 14.9 Hz, 1H), 3.79 (dd, *J* = 7.9, 5.8 Hz, 1H), 1.66 – 1.57 (m, 1H), 1.53 – 1.44 (m, 1H), 1.29 – 1.21 (m, 5H), 1.18 (s, 24H), 0.81 (t, *J* = 6.9 Hz, 4H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 168.3, 128.7, 127.8, 127.7, 126.5, 124.1, 119.63, 119.6, 115.3, 114.4, 114.3, 62.1, 53.6, 31.9, 29.7, 29.69, 29.67, 29.66, 29.64, 29.61, 29.54, 29.52, 29.46, 29.43, 29.39, 29.38, 29.1, 25.6, 22.7, 14.1. **HRMS-ESI** (*m/z*): calcd for $C_{32}H_{42}N_2O$ $[M+H]^+$ 477.3839; found 477.3854.

4-Benzyl-3-pentadecyl-3,4-dihydroquinoxalin-2(1*H*)-one (3ap)

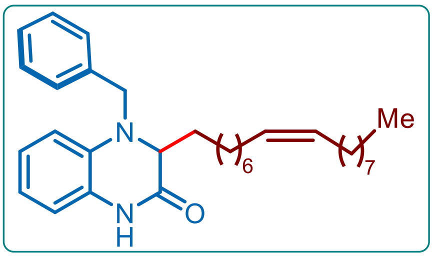


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl palmitate (0.150 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ap** (0.072 g, 64% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.89 (s, 1H), 7.27 – 7.23 (m, 3H), 7.22 – 7.16 (m, 2H), 6.87 – 6.82 (m, 1H), 6.73 – 6.70 (m, 2H), 6.66 (d, *J* = 8.0 Hz, 1H), 4.57 (d, *J* = 15.0 Hz, 1H), 4.21 (d, *J* = 15.0 Hz, 1H), 3.81 – 3.76 (m, 1H), 1.64 – 1.55 (m, 2H), 1.52 – 1.44 (m, 1H), 1.26 – 1.20 (m, 8H), 1.19 (s, 14H), 0.81 (t, *J* = 6.9 Hz, 6H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 168.7, 128.7, 127.7, 127.6, 126.3, 124.1, 119.4, 119.3, 115.4, 114.15, 114.13, 62.0, 53.4, 31.9, 29.7, 29.67, 29.66, 29.63, 29.61, 29.53, 29.52, 29.4, 29.37, 29.30, 29.1, 25.6, 22.7, 14.1. **HRMS-ESI** (*m/z*): calcd for $C_{30}H_{45}N_2O$ $[M+H]^+$ 449.3526; found 449.3537.

(*Z*)-4-Benzyl-3-(heptadec-8-en-1-yl)-3,4-dihydroquinoxalin-2(1*H*)-one (3aq)

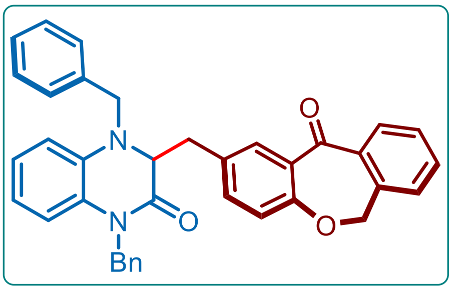


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl oleate (0.160 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3aq** (0.077 g, 65% yield) as viscous liquid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.55 (s, 1H), 7.24 (d, *J* = 4.3 Hz, 3H), 7.21 – 7.17 (m, 2H), 6.86 – 6.81 (m, 1H), 6.71 (dd, *J* = 6.3, 2.7 Hz, 2H), 6.66 (d, *J* = 7.9 Hz, 1H), 5.29 – 5.22 (m, 3H), 4.57 (d, *J* = 15.0 Hz, 1H), 4.20 (d, *J* = 15.0 Hz, 1H), 3.81 – 3.76 (m, 1H), 1.95 – 1.88 (m, 6H), 163 – 1.54 (m, 2H), 1.53 – 1.43 (m, 1H), 1.26 – 1.21 (m, 5H), 1.19 (s, 10H), 0.80 (t, *J* = 6.9 Hz, 6H).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 168.3, 129.9, 129.7, 128.7, 127.7, 127.6, 126.4, 124.1, 119.39, 119.37, 115.3, 114.15, 114.11, 62.0, 53.5, 31.9, 29.78, 29.71, 29.5, 29.4, 29.3, 29.2, 29.19, 29.17, 27.2, 27.19, 27.16, 25.6, 22.6, 14.1. **HRMS-ESI** (*m/z*): calcd for $C_{32}H_{47}N_2O$ $[M+H]^+$ 475.3683; found 475.3700.

1,4-Dibenzyl-3-((11-oxo-6,11-dihydrodibenzo[*b*,*e*]oxepin-2-yl)methyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ar)

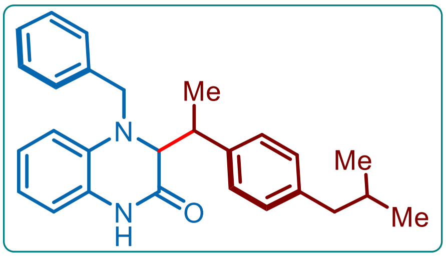


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 2-(11-oxo-6,11-dihydrodibenzo[*b*,*e*]oxepin-2-yl)acetate (0.155 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ar** (0.099 g, 72% yield) as yellow solid. m.p: 150 – 152 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 2.2 Hz, 1H), 7.91 (d, *J* = 7.7 Hz, 1H), 7.60 – 7.55 (m, 1H), 7.52 – 7.46 (m, 1H), 7.37 (d, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 7.0 Hz, 2H), 7.26 – 7.23 (m, 5H), 7.23 – 7.18 (m, 2H), 7.13 (d, *J* = 6.5 Hz, 2H), 6.99 – 6.96 (m, 1H), 6.93 (s, 1H), 6.87 – 6.85 (m, 1H), 6.80 (d, *J* = 7.9 Hz, 1H), 6.76 – 6.70 (m, 1H), 5.39 (d, *J* = 16.1 Hz, 1H), 5.18 (s, 2H), 4.96 (d, *J* = 16.1 Hz, 1H), 4.51 (d, *J* = 14.8 Hz, 1H), 4.31 – 4.26 (m, 1H), 3.98 (d, *J* = 14.8 Hz, 1H), 3.01 – 2.91 (m, 2H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 190.7, 166.5, 160.2, 140.4, 136.7, 136.4, 136.3, 135.6, 132.8, 132.7, 130.8, 129.5, 129.4, 129.2, 128.8, 128.6 (2C), 127.9, 127.8, 127.6, 127.2, 126.5, 125.1, 124.1, 120.8, 119.8, 115.5, 115.0, 73.6, 63.5, 54.1, 45.9, 34.4. **HRMS-ESI** (*m/z*): calcd for $C_{37}H_{31}N_2O_3$ $[M+H]^+$ 551.2329; found 551.2338.

4-Benzyl-3-(1-(4-isobutylphenyl)ethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3as)

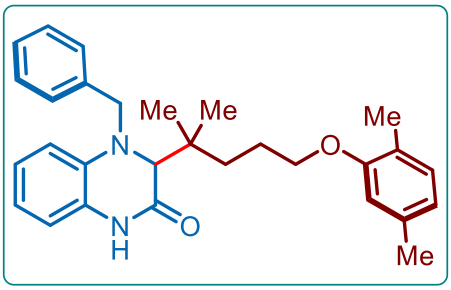


Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 2-(4-isobutylphenyl)propanoate (0.131 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 μL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3as** (0.074 g, 74% yield) as a white solid. m.p: 139 – 141°C.

^1H NMR (500 MHz, $CDCl_3$) δ 8.50 (s, 1H), 7.27 (s, 1H), 7.25 – 7.20 (m, 2H), 7.14 (d, *J* = 7.0 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.87 – 6.82 (m, 1H), 6.66 (d, *J* = 8.0 Hz, 1H), 6.64 – 6.59 (m, 1H), 6.50 (dd, *J* = 7.7, 1.2 Hz, 1H), 4.65 (d, *J* = 15.4 Hz, 1H), 4.10 (d, *J* = 5.0 Hz, 1H), 4.02 (d, *J* = 15.4 Hz, 1H), 3.30 – 3.23 (m, 1H), 2.40 – 2.36 (m, 2H), 1.85 – 1.74 (m, 1H), 1.33 (d, *J* = 7.2 Hz, 3H), 0.87 (dd, *J* = 6.6, 0.8 Hz, 6H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.9, 140.1, 139.0, 136.9, 134.2, 128.8, 128.6, 127.8, 127.4 (2C), 126.1, 123.9, 118.5, 115.0, 113.4, 68.0, 54.2, 45.0, 42.3, 30.1, 22.4, 22.3, 17.0. **HRMS-ESI** (*m/z*): calcd for $C_{27}H_{30}N_2NaO$ $[M+Na]^+$ 421.2250; found 421.2256.

4-Benzyl-3-(5-(2,5-dimethylphenoxy)-2-methylpentan-2-yl)-3,4-dihydroquinoxalin-2(1*H*)-one (3at)



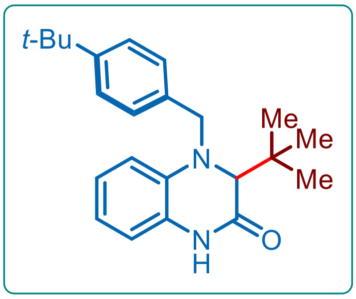
Synthesized according to the GP3 using 4-benzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.059 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (0.148 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 μL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3at** (0.091 g, 82% yield) as viscous liquid.

^1H NMR (500 MHz, $CDCl_3$) δ 8.97 (s, 1H), 7.18 – 7.13 (m, 2H), 7.11 (dd, *J* = 5.2, 1.9 Hz, 1H), 7.08 (dd, *J* = 6.9, 5.5 Hz, 2H), 6.94 (d, *J* = 7.5 Hz, 1H), 6.87 – 6.82 (m, 1H), 6.78 (d, *J* =

7.9 Hz, 1H), 6.65 (d, *J* = 3.9 Hz, 2H), 6.60 (d, *J* = 7.5 Hz, 1H), 6.56 (s, 1H), 4.87 (d, *J* = 15.7 Hz, 1H), 4.29 (d, *J* = 15.7 Hz, 1H), 3.88 – 3.79 (m, 2H), 3.73 (s, 1H), 2.25 (s, 3H), 2.12 (s, 3H), 1.77 – 1.66 (m, 2H), 1.52 – 1.47 (m, 2H), 0.86 (s, 6H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.3, 157.0, 137.4, 136.5, 134.9, 130.3, 128.6, 127.7, 127.5, 127.3, 124.1, 123.5, 120.6, 119.3, 115.8, 115.2, 112.0, 69.8, 68.3, 58.3, 41.8, 36.1, 24.3, 24.1, 24.0, 21.4, 15.9. **HRMS-ESI** (*m/z*): calcd for $C_{29}H_{35}N_2O_2$ [M+H]$^+$ 443.2693; found 443.2711.

3-(*Tert*-butyl)-4-(4-(*tert*-butyl)benzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ba)

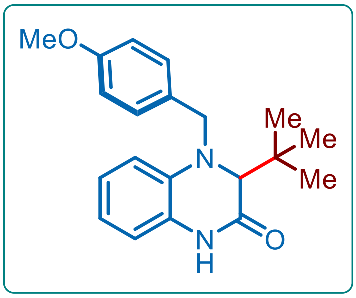


Synthesized according to the GP3 using 4-(4-(*tert*-butyl)benzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.074 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ba** (0.074 g, 85% yield) as a white solid. m.p: 90 – 92 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 9.22 (s, 1H), 7.20 – 7.16 (m, 2H), 7.02 (d, *J* = 8.3 Hz, 2H), 6.85 – 6.81 (m, 1H), 6.73 (d, *J* = 7.9 Hz, 1H), 6.69 – 6.61 (m, 2H), 4.86 (d, *J* = 15.1 Hz, 1H), 4.27 (d, *J* = 15.1 Hz, 1H), 3.64 (s, 1H), 1.18 (s, 9H), 0.89 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.7, 150.2, 134.8, 134.3, 127.3, 126.9, 125.5, 124.0, 118.7, 115.1, 114.8, 70.9, 57.0, 39.6, 34.4, 31.3, 27.5. **HRMS-ESI** (*m/z*): calcd for $C_{23}H_{31}N_2O$ [M+H]$^+$ 351.2431; found 351.2438.

3-(*Tert*-butyl)-4-(4-methoxybenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ca)

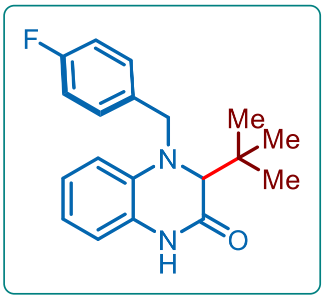


Synthesized according to the GP3 using 4-(4-methoxybenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.067 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ca** (0.062 g, 76% yield) as a white solid. m.p: 95 – 97 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.68 (s, 1H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.94 – 6.90 (m, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 6.78 – 6.75 (m, 2H), 6.74 – 6.68 (m, 2H), 4.85 (d, *J* = 15.3 Hz, 1H), 4.32 (d, *J* = 15.3 Hz, 1H), 3.73 (s, 3H), 3.68 (s, 1H) 0.95 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.2, 159.0, 134.5, 129.0, 128.7, 127.5, 124.0, 119.2, 115.6, 115.0, 114.0, 70.8, 57.5, 55.1, 39.4, 27.5. **HRMS-ESI** (*m/z*): calcd for $C_{20}H_{25}N_2O_2$ $[M+H]^+$ 325.1911; found 325.1910.

3-(*Tert*-butyl)-4-(4-fluorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3da)



Synthesized according to the GP3 using 4-(4-fluorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.064 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 μL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3da** (0.062 g, 80% yield) as a white solid. m.p: 106 – 108 °C.

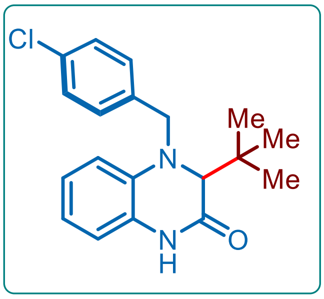
^{1}H NMR (500 MHz, CDCl$_3$) δ 8.78 (s, 1H), 7.07 – 7.02 (m, 2H), 6.86 – 6.81 (m, 3H), 6.71 (d, *J* = 8.0 Hz, 1H), 6.66 – 6.62 (m, 2H), 4.82 (d, *J* = 15.1 Hz, 1H), 4.25 (d, *J* = 15.1 Hz, 1H), 3.60 (s, 1H), 0.88 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.3, 162.1 (d, *J* = 245.4 Hz), 134.3, 133.1 (d, *J* = 3.1 Hz), 128.8 (d, *J* = 8.1 Hz), 127.6, 124.0, 119.2, 115.6, 115.4 (d, *J* = 15.2 Hz), 115.1, 71.4, 57.1, 39.5, 27.5.

^{19}F{^{1}H} NMR (471 MHz, CDCl$_3$) δ -115.0.

HRMS-ESI (*m/z*): calcd for $C_{19}H_{22}FN_2O$ $[M+H]^+$ 313.1711; found 313.1721.

3-(*Tert*-butyl)-4-(4-chlorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ea)

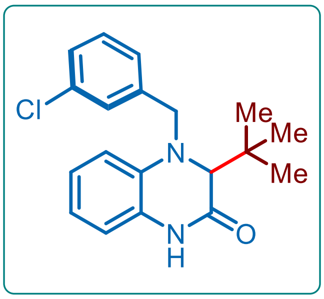


Synthesized according to the GP3 using 4-(4-chlorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.068 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 μL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ea** (0.064 g, 78% yield) as a white solid. m.p: 121 – 123 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.60 (s, 1H), 7.18 (d, *J* = 8.4 Hz, 2H), 7.08 (d, *J* = 8.3 Hz, 2H), 6.93 – 6.86 (m, 1H), 6.76 – 6.70 (m, 3H), 4.90 (d, *J* = 15.1 Hz, 1H), 4.32 (d, *J* = 15.1 Hz, 1H), 3.67 (s, 1H), 0.95 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.2, 136.0, 134.3, 128.8, 128.5, 127.6, 124.0, 123.6, 119.2, 115.15, 115.10, 71.7, 57.1, 39.5, 27.5. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{22}ClN_2O$ [M+H]$^+$ 329.1415; found 329.1418.

3-(*Tert*-butyl)-4-(3-chlorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3fa)

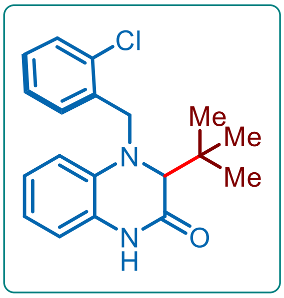


Synthesized according to the GP3 using 4-(3-chlorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.068 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3fa** (0.062 g, 75% yield) as a white solid. m.p: 128 – 130 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.82 (s, 1H), 7.17 – 7.14 (m, 3H), 7.04 – 7.01 (m, 1H), 6.93 – 6.87 (m, 1H), 6.77 – 6.71 (m, 3H), 4.91 (d, *J* = 15.1 Hz, 1H), 4.33 (d, *J* = 15.1 Hz, 1H), 3.68 (s, 1H), 0.96 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.5, 139.8, 134.6, 134.3, 130.1, 127.8, 127.6, 127.5, 125.3, 124.2, 119.4, 115.3, 115.1, 71.8, 57.2, 39.7, 27.7. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{22}ClN_2O$ [M+H]$^+$ 329.1415; found 329.1409.

3-(*Tert*-butyl)-4-(2-chlorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ga)

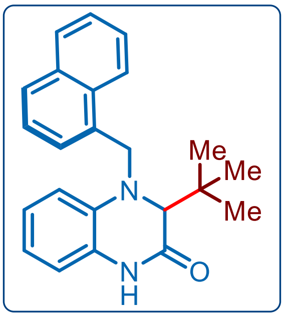


Synthesized according to the GP3 using 4-(2-chlorobenzyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.068 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ga** (0.063 g, 77% yield) as a white solid. m.p: 136 – 138 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.24 (s, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 6.95 – 6.86 (m, 3H), 6.83 (t, *J* = 7.5 Hz, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.66 – 6.59 (m, 2H), 4.82 (d, *J* = 15.7 Hz, 1H), 4.26 (d, *J* = 15.7 Hz, 1H), 3.62 (s, 1H), 0.88 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.3, 138.2, 137.4, 134.8, 128.5, 128.2, 128.0, 127.4, 124.3, 123.9, 118.7, 115.0, 114.9, 71.2, 57.6, 39.5, 27.5. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{22}ClN_2O$ [M+H]$^+$ 329.1415; found 329.1405.

3-(*Tert*-butyl)-4-(naphthalen-1-ylmethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ha)

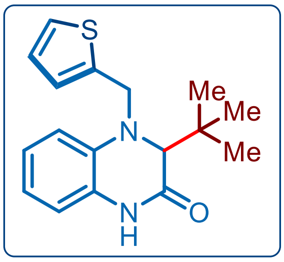


Synthesized according to the GP3 using 4-(naphthalen-1-ylmethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.072 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ha** (0.064 g, 74% yield) as a white solid. m.p: 85 – 87 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.87 (s, 1H), 7.86 – 7.82 (m, 2H), 7.74 (d, *J* = 7.9 Hz, 1H), 7.44 – 7.39 (m, 2H), 7.36 – 7.28 (m, 2H), 6.96 – 6.91 (m, 1H), 6.89 (d, *J* = 7.8 Hz, 1H), 6.77 – 6.71 (m, 2H), 5.41 (d, *J* = 15.1 Hz, 1H), 4.81 (d, *J* = 15.1 Hz, 1H), 3.64 (s, 1H), 0.98 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.4, 135.0, 133.9, 131.9, 131.5, 128.9, 128.4, 127.3, 126.38, 126.32, 125.8, 125.3, 124.1, 122.8, 118.9, 115.2, 114.5, 69.5, 55.0, 39.7, 27.7. **HRMS-ESI** (*m/z*): calcd for $C_{23}H_{25}N_2O$ [M+H]$^+$ 345.1961; found 345.1966.

3-(*Tert*-butyl)-4-(thiophen-2-ylmethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ia)

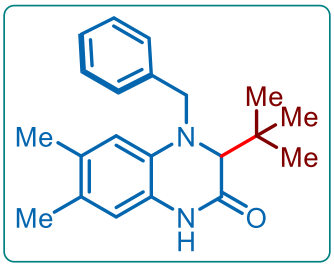


Synthesized according to the GP3 using 4-(thiophen-2-ylmethyl)-3,4-dihydroquinoxalin-2(1*H*)-one (0.061 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ia** (0.051 g, 68% yield) as a white solid. m.p: 148 – 150 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.36 (s, 1H), 7.04 – 7.02 (m, 1H), 5.91 – 6.86 (m, 2H), 6.80 – 6.77 (m, 2H), 6.73 – 6.69 (m, 1H), 6.63 (d, *J* = 7.8 Hz, 1H), 4.99 (d, *J* = 15.1 Hz, 1H), 4.39 (d, *J* = 15.1 Hz, 1H), 3.61 (s, 1H), 0.87 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.2, 140.8, 134.3, 128.1, 126.8, 125.9, 125.2, 124.3, 120.1, 116.4, 115.4, 70.6, 53.6, 39.2, 27.4. **HRMS-ESI** (*m/z*): calcd for $C_{17}H_{21}N_2OS$ [M+H]$^+$ 301.1369; found 301.1388.

4-Benzyl-3-(*tert*-butyl)-6,7-dimethyl-3,4-dihydroquinoxalin-2(1*H*)-one (3ja)

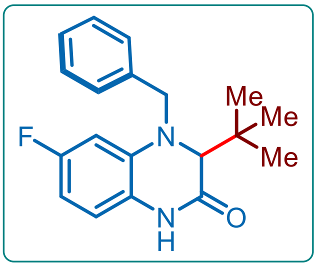


Synthesized according to the GP3 using 4-benzyl-6,7-dimethyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.064 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ja** (0.062 g, 77% yield) as a white solid. m.p: 144 – 146 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.58 (s, 1H), 7.26 – 7.21 (m, 2H), 7.19 (d, *J* = 7.1 Hz, 1H), 7.16 (d, *J* = 7.0 Hz, 2H), 6.68 (s, 1H), 6.47 (s, 1H), 4.86 (d, *J* = 15.1 Hz, 1H), 4.31 (d, *J* = 15.1 Hz, 1H), 3.60 (s, 1H), 2.14 (s, 3H), 2.13 (s, 3H), 0.94 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.2, 137.6, 132.4, 132.0, 128.6 (2C), 127.5, 127.4, 125.5, 117.3, 116.3, 70.9, 58.3, 39.1, 27.5, 19.6, 18.9. **HRMS-ESI** (*m/z*): calcd for $C_{21}H_{27}N_2O$ $[M+H]^+$ 323.2118; found 323.2120.

4-Benzyl-3-(*tert*-butyl)-6-fluoro-3,4-dihydroquinoxalin-2(1*H*)-one (3ka)



Synthesized according to the GP3 using 4-benzyl-6-fluoro-3,4-dihydroquinoxalin-2(1*H*)-one (0.064 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3ka** (0.054 g, 69% yield) as a white solid. m.p: 151 – 153 °C.

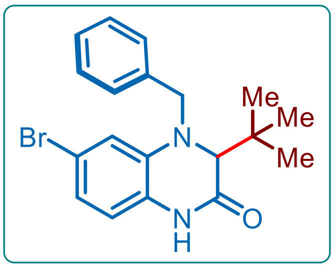
^{1}H NMR (500 MHz, $CDCl_3$) δ 9.17 (s, 1H), 7.26 – 7.21 (m, 2H), 7.21 – 7.18 (m, 1H), 7.14 (d, *J* = 7.2 Hz, 2H), 6.75 – 6.70 (m, 1H), 6.64 – 6.58 (m, 1H), 6.51 – 6.47 (m, 1H), 4.82 (d, *J* = 15.6 Hz, 1H), 4.32 (d, *J* = 15.6 Hz, 1H), 3.64 (s, 1H), 0.94 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.1, 156.6 (d, *J* = 237.6 Hz), 137.4, 130.9 (d, *J* = 2.1 Hz), 128.8 (d, *J* = 10.4 Hz), 128.7, 127.6, 127.5, 116.5 (d, *J* = 8.8 Hz), 109.9 (d, *J* = 22.4 Hz), 102.6 (d, *J* = 26.6 Hz), 71.3, 58.9, 39.3, 27.6.

^{19}F{^{1}H} NMR (471 MHz, $CDCl_3$) δ -124.1.

HRMS-ESI (*m/z*): calcd for $C_{19}H_{25}FN_3O$ $[M+NH_4]^+$ 330.1976; found 330.1956.

4-Benzyl-6-bromo-3-(*tert*-butyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3la)

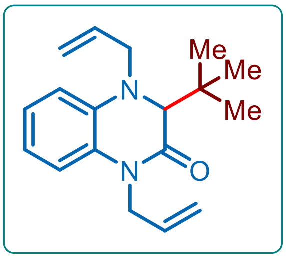


Synthesized according to the GP3 using 4-benzyl-6-bromo-3,4-dihydroquinoxalin-2(1*H*)-one (0.079 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3la** (0.054 g, 58% yield) as a white solid. m.p: 162 – 164 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.94 (s, 1H), 7.19 – 7.10 (m, 3H), 7.06 (d, *J* = 7.2 Hz, 2H), 6.90 (dd, *J* = 8.6, 2.2 Hz, 1H), 6.77 (d, *J* = 2.2 Hz, 1H), 6.55 (d, *J* = 8.6 Hz, 1H), 4.81 (d, *J* = 15.9 Hz, 1H), 4.29 (d, *J* = 15.9 Hz, 1H), 3.64 (s, 1H), 0.89 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.5, 137.0, 133.8, 128.88, 128.86, 127.7, 127.3, 126.7, 117.8, 116.3, 110.6, 71.6, 57.9, 39.8, 27.6. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{22}BrN_2O$ $[M+H]^+$ 373.0910; found 373.0915.

1,4-Diallyl-3-(*tert*-butyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3ma)

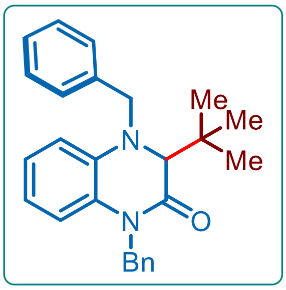


Synthesized according to the GP3 using 1,4-diallyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.057 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ma** (0.055 g, 78% yield) as a viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.00 – 6.94 (m, 1H), 6.92 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.84 – 6.74 (m, 2H), 5.96 – 5.86 (m, 1H), 5.76 – 5.66 (m, 1H), 5.30 – 5.26 (m, 1H), 5.25 – 5.20 (m, 1H), 5.19 – 5.15 (m, 1H), 5.14 – 5.12 (m, 1H), 4.94 – 4.87 (m, 1H), 4.35 – 4.26 (m, 2H), 3.80 – 3.73 (m, 1H), 3.71 (s, 1H), 0.88 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 164.8, 135.9, 133.9, 133.0, 130.7, 123.5, 118.9, 117.8, 117.0, 115.4, 114.8, 70.6, 56.8, 45.2, 39.3, 27.7. **HRMS-ESI** (*m/z*): calcd for $C_{18}H_{25}N_2O$ $[M+H]^+$ 285.1961; found 285.1966.

1,4-Dibenzyl-3-(*tert*-butyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3na)

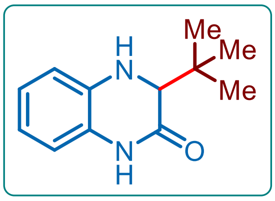


Synthesized according to the GP3 using 1,4-dibenzyl-3,4-dihydroquinoxalin-2(1*H*)-one (0.082 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3na** (0.076 g, 79% yield) as a white solid. m.p: 79 – 81 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.30 (s, 1H), 7.28 (d, J = 4.8 Hz, 3H), 7.25 – 7.20 (m, 4H), 7.16 – 7.13 (m, 2H), 6.90 – 6.85 (m, 2H), 6.80 (dd, J = 8.3, 1.2 Hz, 1H), 6.69 – 6.65 (m, 1H), 5.28 (d, J = 15.1 Hz, 1H), 5.14 (d, J = 15.1 Hz, 1H), 4.94 (d, J = 15.1 Hz, 1H), 4.37 (d, J = 15.1 Hz, 1H), 3.85 (s, 1H), 0.94 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 165.4, 137.5, 137.4, 136.0, 130.6, 128.6, 128.5, 127.4 (2C), 127.1, 127.1, 123.7, 118.9, 115.4, 115.3, 71.9, 57.7, 46.1, 39.5, 27.8. **HRMS-ESI** (*m/z*): calcd for $C_{26}H_{29}N_2O$ [M+H]$^+$ 385.2274; found 385.2282.

3-(*tert*-butyl)-3,4-dihydroquinoxalin-2(1*H*)-one (3oa)

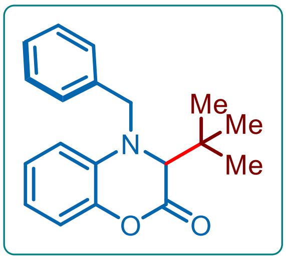


Synthesized according to the GP3 using 3,4-dihydroquinoxalin-2(1*H*)-one (0.037 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (15% ethyl acetate in hexanes as eluent) to afford **3oa** (0.038 g, 74% yield) as yellow solid. m.p: 159-161 °C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.95 (s, 1H), 6.80 – 6.75 (m, 1H), 6.64 – 6.58 (m, 2H), 6.56 (d, *J* = 7.8 Hz, 1H), 3.90 (br s, 1H), 3.63 (s, 1H), 0.94 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.8, 133.5, 125.0, 124.0, 118.4, 115.1, 112.7, 65.2, 37.8, 26.7. **HRMS-ESI** (*m/z*): calcd for $C_{12}H_{17}N_2O$ [M+H]$^+$ 205.1335; found 205.1336.

4-Benzyl-3-(*tert*-butyl)-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (3pa)

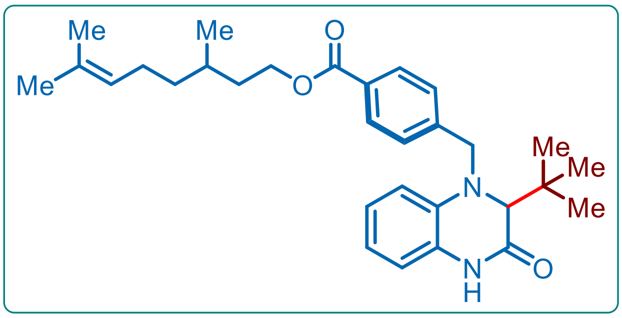


Synthesized according to the GP3 using 4-benzyl-3,4-dihydro-2*H*-benzo[*b*][1,4]oxazin-2-one (0.060 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3pa** (0.052 g, 71% yield) as viscous liquid.

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.31 – 7.26 (m, 2H), 7.25 – 7.22 (m, 1H), 7.15 (d, *J* = 7.3 Hz, 2H), 7.00 (t, *J* = 7.7 Hz, 1H), 6.95 (d, *J* = 8.0 Hz, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 6.80 (t, *J* = 7.6 Hz, 1H), 4.87 (d, *J* = 15.6 Hz, 1H), 4.31 (d, *J* = 15.6 Hz, 1H), 3.77 (s, 1H), 0.93 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 164.1, 143.0, 136.7, 133.0, 128.8, 127.8, 127.4, 125.0, 119.8, 116.3, 116.0, 69.1, 57.4, 39.0, 27.4. **HRMS-ESI** (*m/z*): calcd for $C_{19}H_{22}NO_2$ $[M+H]^+$ 296.1645; found 385.2282.

3,7-Dimethyloct-6-en-1-yl 4-((2-(*tert*-butyl)-3-oxo-3,4-dihydroquinoxalin-1(2*H*)-yl)methyl)benzoate (3qa)

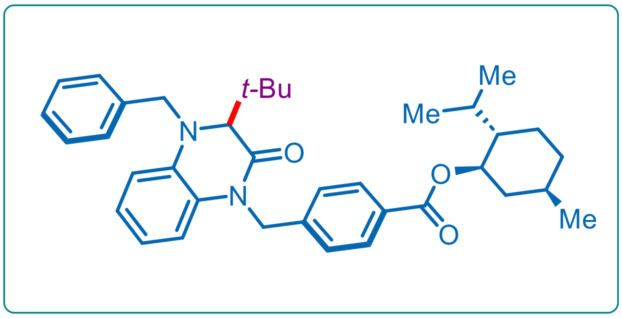


Synthesized according to the GP3 using 3,7-dimethyloct-6-en-1-yl 4-((3-oxo-3,4-dihydroquinoxalin-1(2*H*)-yl)methyl)benzoate (0.105 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10→20% acetone in hexanes as eluent) to afford **3qa** (0.080 g, 76% yield) as a white solid. m.p: 155 – 157 ^{o}C.

^{1}H NMR (500 MHz, CDCl$_3$) δ 8.55 (s, 1H), 7.91 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.2 Hz, 2H), 6.89 – 6.83 (m, 1H), 6.74 – 6.68 (m, 3H), 5.08 (t, *J* = 7.0 Hz, 1H), 5.00 (d, *J* = 16.3 Hz, 1H), 4.41 (d, *J* = 16.3 Hz, 1H), 4.35 – 4.25 (m, 2H), 3.70 (s, 1H), 2.05 – 1.92 (m, 2H), 1.81 – 1.73 (m, 1H), 1.65 (s, 3H), 1.64 – 1.60 (m, 1H), 1.58 (s, 3H), 1.57 – 1.52 (m, 1H), 1.26 (d, *J* = 4.9 Hz, 1H), 1.24 – 1.16 (m, 1H), 0.97 (s, 9H), 0.94 (d, *J* = 6.6 Hz, 3H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 166.3, 166.1, 142.8, 134.1, 131.3, 129.9, 129.7, 127.5, 127.1, 124.5, 124.0, 119.2, 115.0, 114.9, 72.0, 63.4, 57.4, 39.6, 36.9, 35.4, 29.5, 27.5, 25.7, 25.3, 19.4, 17.6. **HRMS-ESI** (*m/z*): calcd for $C_{30}H_{41}N_2O_3$ $[M+H]^+$ 477.3112; found 477.3113.

(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl 4-((4-benzyl-3-(*tert*-butyl)-2-oxo-3,4-dihydroquinoxalin-1(2*H*)-yl)methyl)benzoate (3ra)



Synthesized according to the GP3 using (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 4-((4-benzyl-2-oxo-3,4-dihydroquinoxalin-1(2*H*)-yl)methyl)benzoate (0.128 g, 0.25 mmol, 1.0 equiv), 1,3-dioxoisoindolin-2-yl pivalate (0.093 g, 0.375 mmol, 1.5 equiv), Rose bengal (0.013 g, 5 mol%), and DIPEA (65 µL, 0.375 mmol, 1.5 equiv). The crude mixture was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes as eluent) to afford **3ra** as mixture of diastereoisomers (0.102 g, 72% yield, d.r. = 1:1) as viscous liquid.

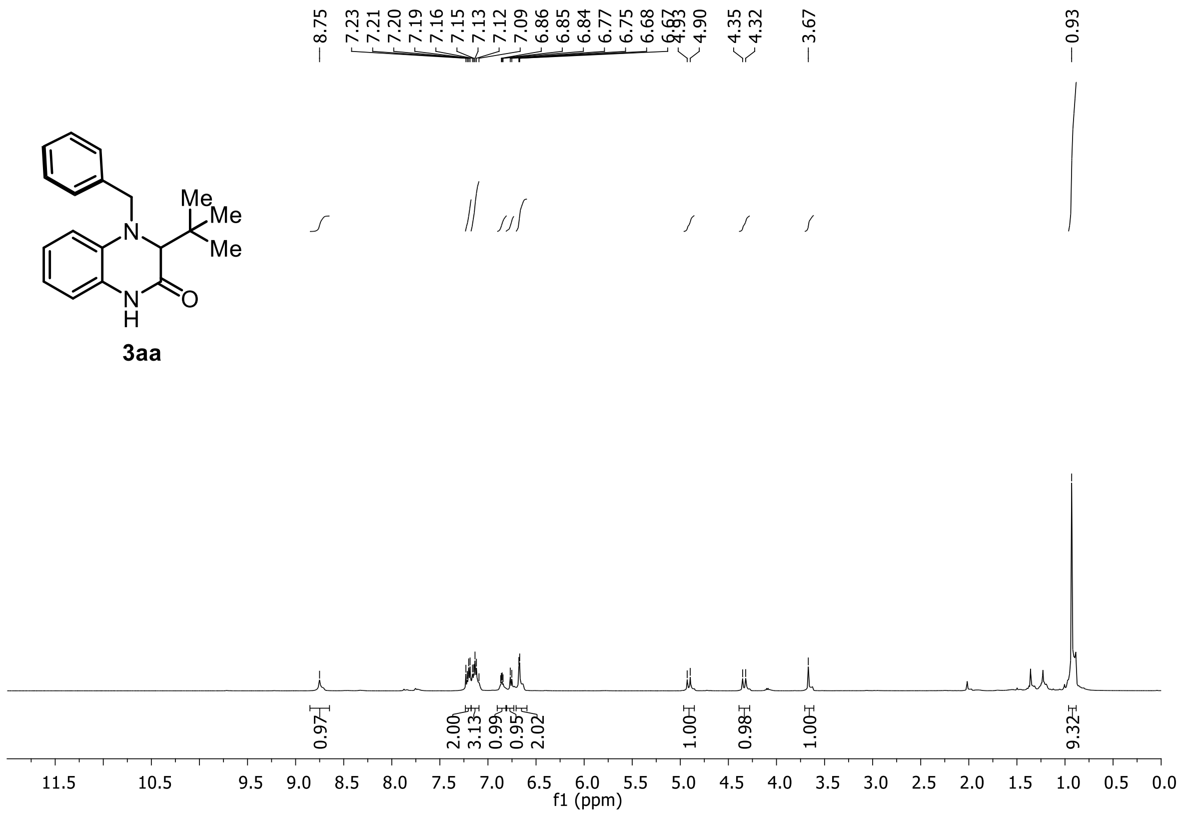
^{1}H NMR (500 MHz, CDCl$_3$) δ 7.97 (dd, *J* = 8.3, 1.6 Hz, 2H), 7.32 – 7.28 (m, 2H), 7.25 – 7.22 (m, 2H), 7.17 – 7.08 (m, 3H), 6.88 (t, *J* = 7.6 Hz, 1H), 6.85 – 6.81 (m, 1H), 6.79 (d, *J* = 8.1 Hz, 1H), 6.69 6.64 (m, 1H), 5.31 – 5.19 (m, 2H), 4.96 – 4.89 (m, 2H), 4.37 (d, *J* = 15.6 Hz, 1H), 3.86 (s, 1H), 2.11 (d, *J* = 12.1 Hz, 1H), 1.99 – 1.92 (m, 1H), 1.73 (d, *J* = 11.3 Hz, 2H), 1.59 – 1.51 (m, 3H), 1.15 – 1.04 (m, 2H), 0.95 (s, 9H), 0.93 – 0.91 (d, *J* = 6.9 Hz, 6H), 0.79 (d, *J* = 6.9 Hz, 3H).

^{13}C{^{1}H} NMR (126 MHz, CDCl$_3$) δ 165.9 (2C), 165.6, 165.5, 142.5, 137.6, 136.17, 136.16, 130.5, 130.4, 130.3, 130.2, 130.1, 129.99, 129.97, 128.76, 128.75, 127.65, 127.63, 127.60, 127.58 (2C), 127.55, 127.1, 127.0, 124.0, 119.20 (2C), 115.8, 115.3, 74.9, 71.9, 57.92, 57.90, 47.4, 46.09, 46.05, 41.1, 39.7, 34.4, 31.5, 29.85, 29.81, 27.96, 27.95, 26.63, 26.61, 23.7, 22.1, 20.9, 16.65, 16.64. **HRMS-ESI** (*m/z*): calcd for $C_{37}H_{47}N_2O_3$ $[M+H]^+$ 567.3581; found 567.3586.

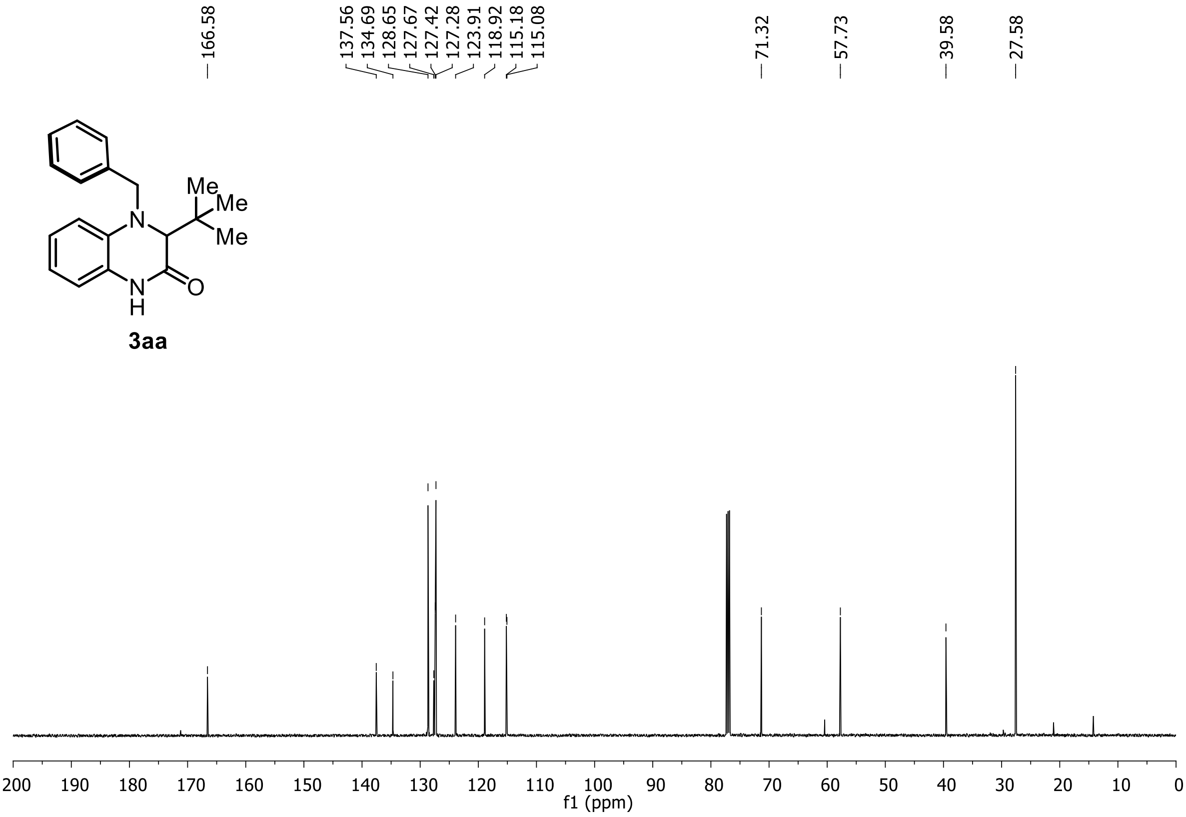
5. References:

1. (a) Y. R. Bonuga et al, *Der Pharma Chemica*, 2013, **5**, 296. (b) P. S. Akula, B.-C. Hong, G.-H. Lee, *RSC Adv.*, 2018, **8**, 19580. (c) J. Rostoll-Berenguer, G. Blay, J. R. Pedro, C. Vila, *Org. Lett.* 2020**, 22**, 8012.
2. C. Huo, J. Dong, Y. Su, J. Tang, F. Chen, *Chem. Commun.* 2016**, 52**, 13341.
3. C. Zheng, Y. Wang, Y. Xu, Z. Chen, G. Chen, S. H. Liang, *Org. Lett.* 2018*,* **20***,* 4824.
4. (a) M.-C. Fu, R. Shang, B. Zhao, B.Wang, Y. Fu, *Science* 2019, **363**, 1429. (b) M. A. Cismesia, T. P Yoon, *Chem. Sci.* 2015, **6**, 5426.

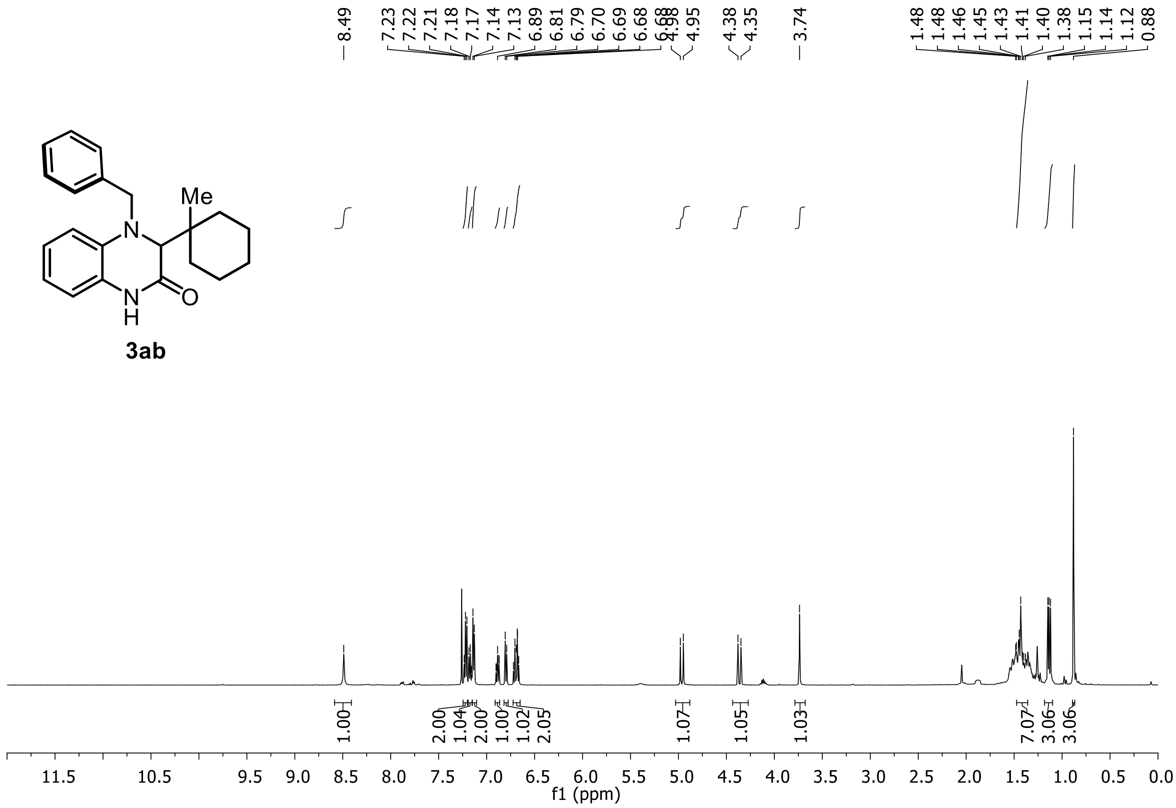
6. ^{1}H NMR spectrum of 3aa (CDCl$_3$, 500 MHz)



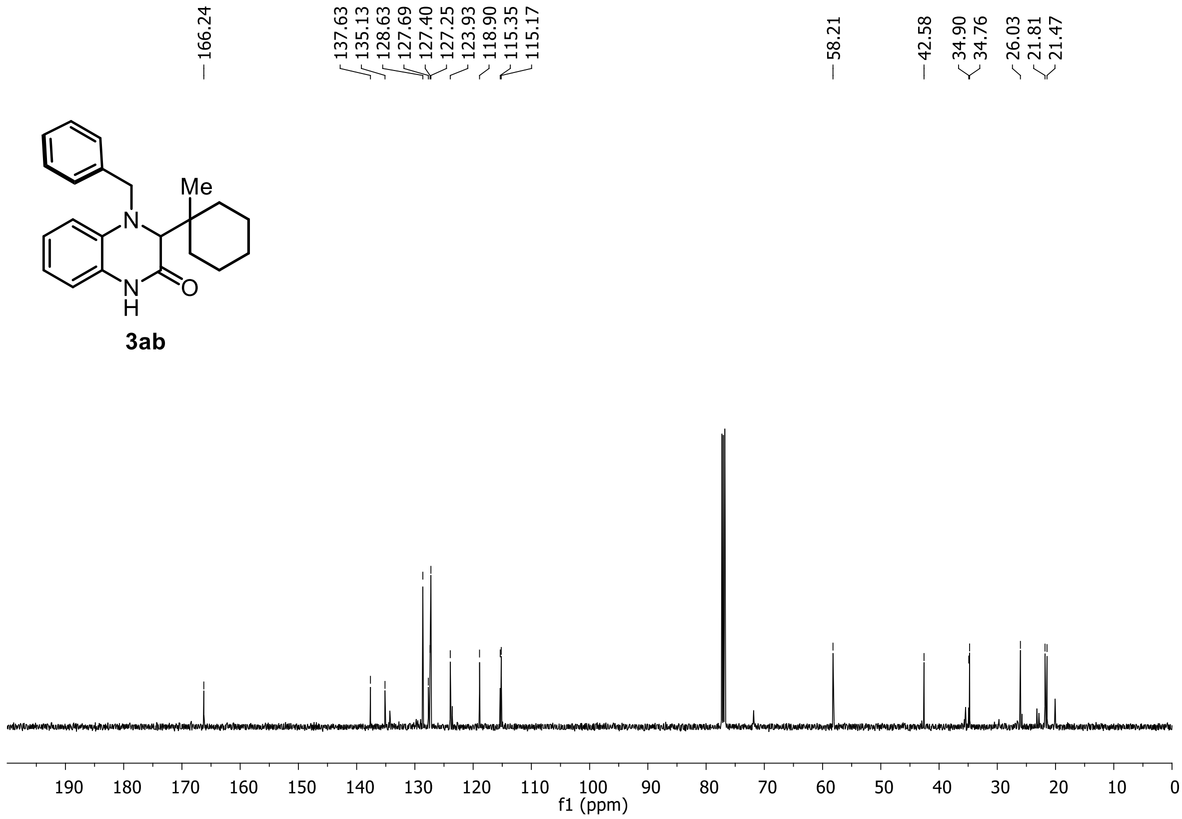
s^{13}C{^{1}H} NMR spectrum of 3aa (CDCl$_3$, 126 MHz)



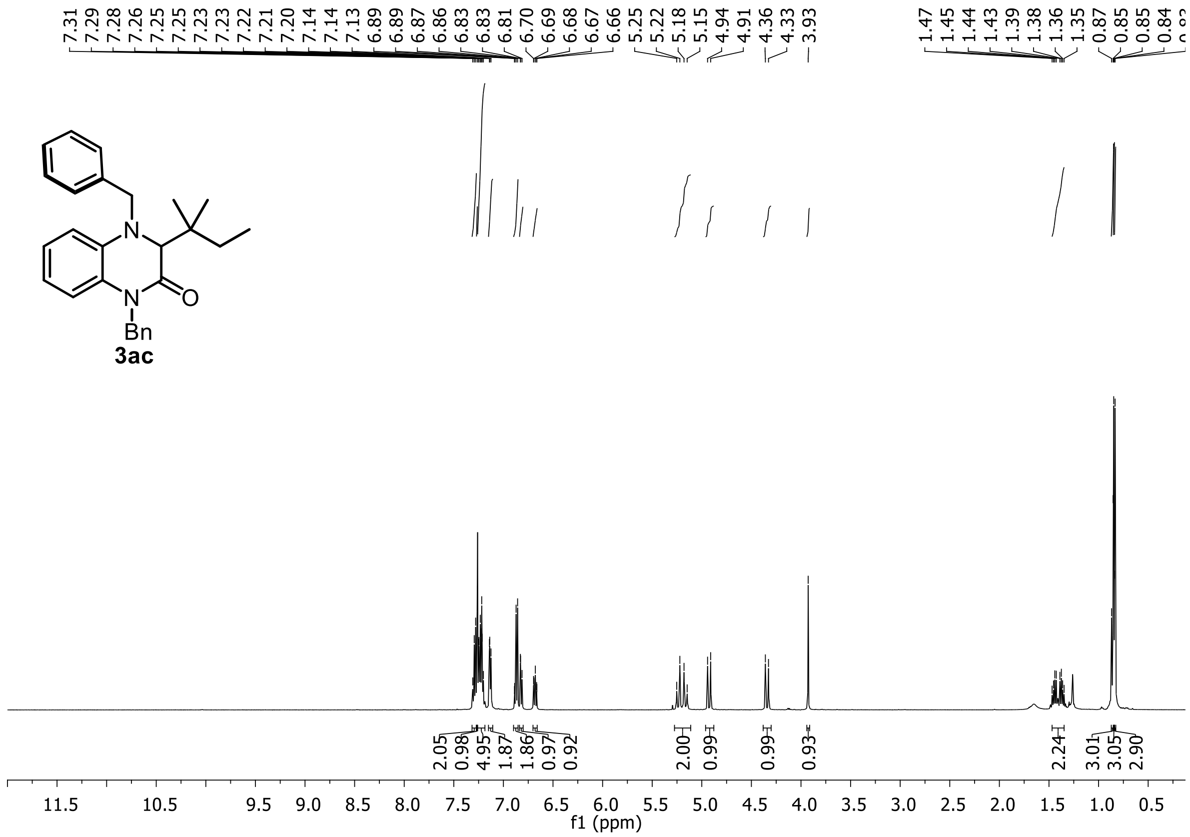
^{1}H NMR spectrum of 3ab (CDCl$_3$, 500 MHz)



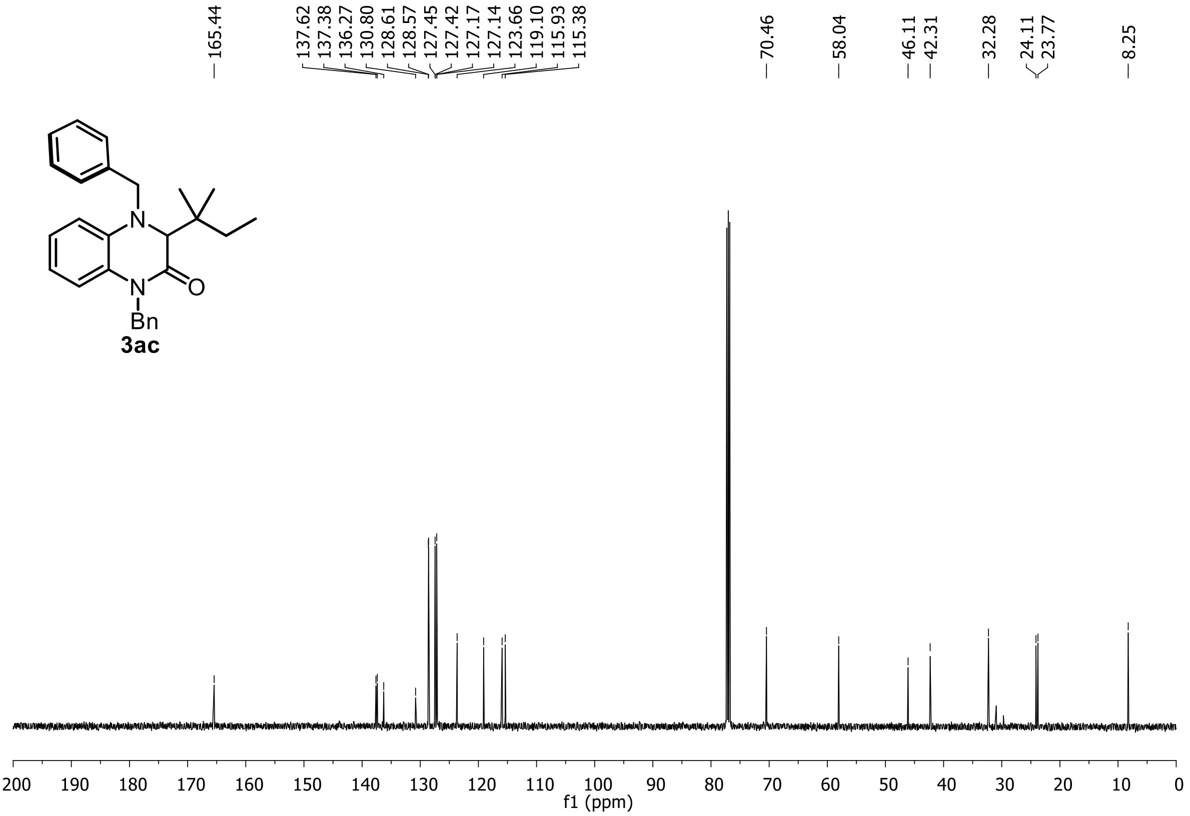
^{13}C{^{1}H} NMR spectrum of 3ab (CDCl$_3$, 126 MHz)



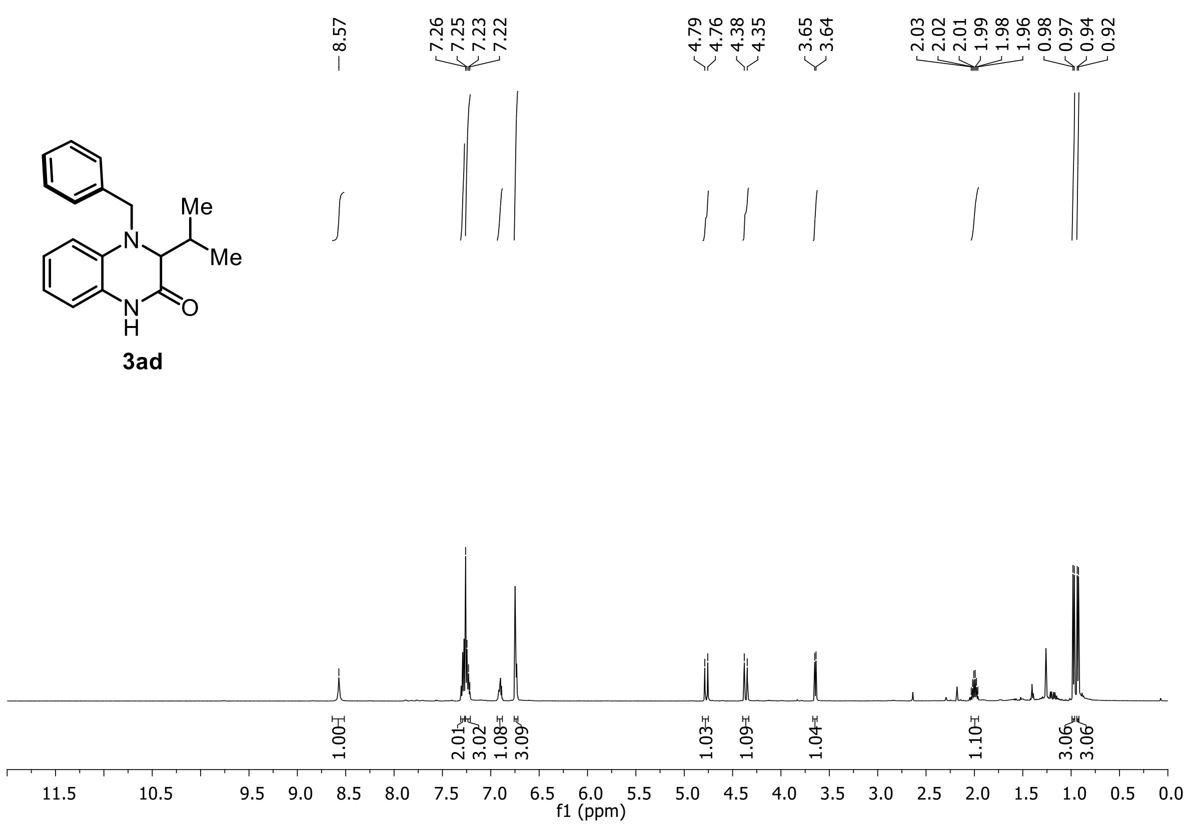
^{1}H NMR spectrum of 3ac (CDCl$_3$, 500 MHz)



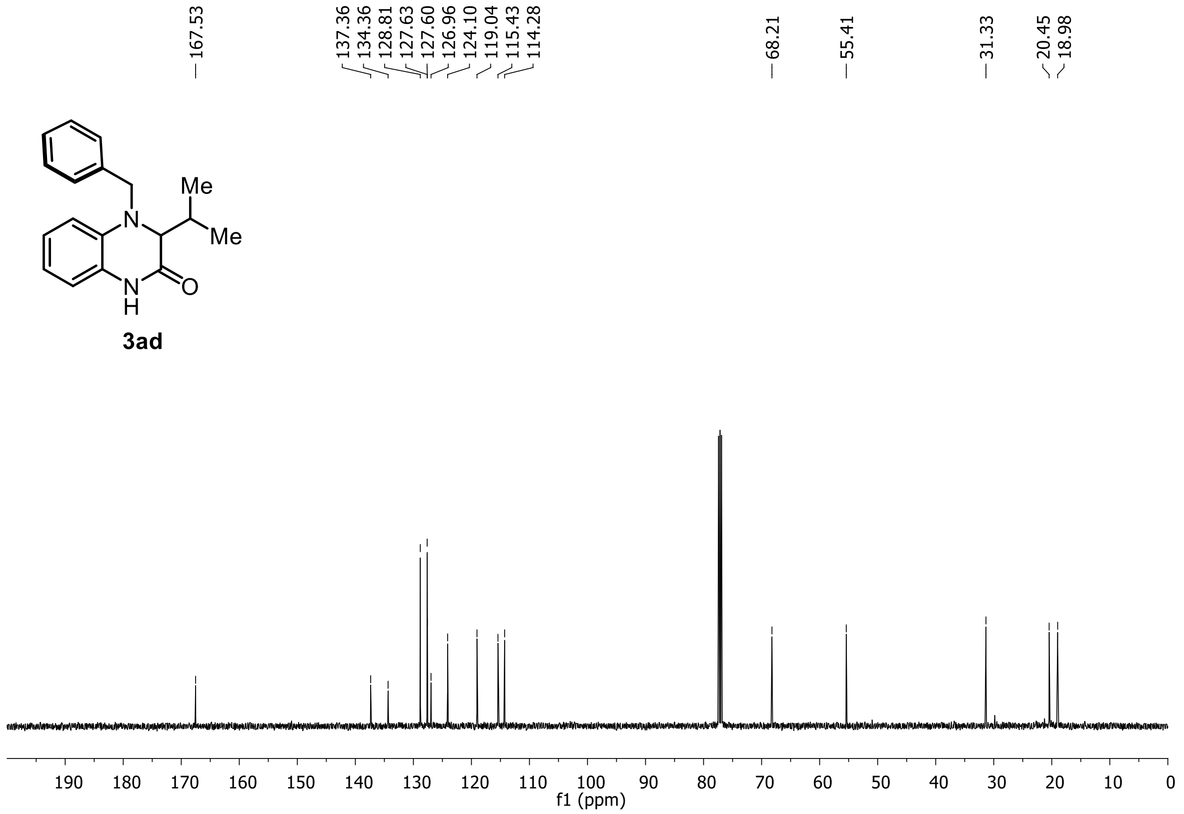
^{13}C{^{1}H} NMR spectrum of 3ac (CDCl$_3$, 126 MHz)



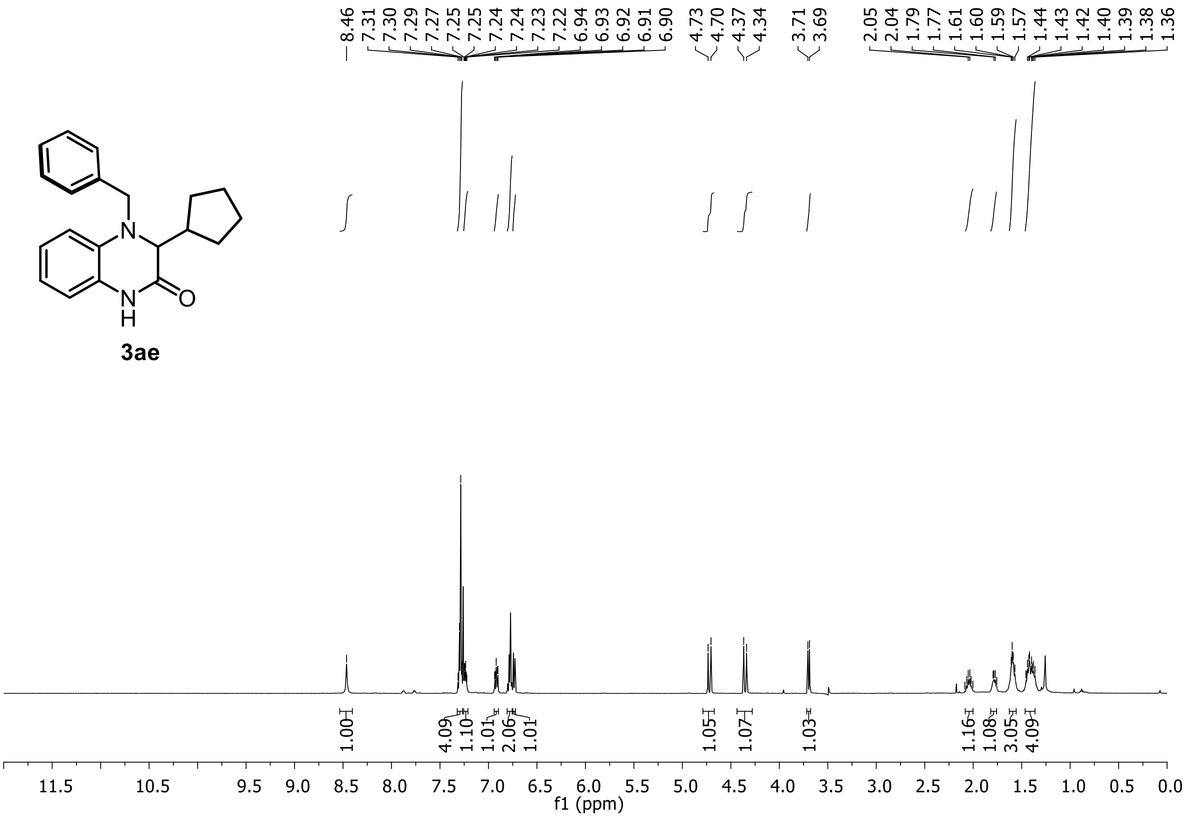
^{1}H NMR spectrum of 3ad (CDCl$_3$, 500 MHz)



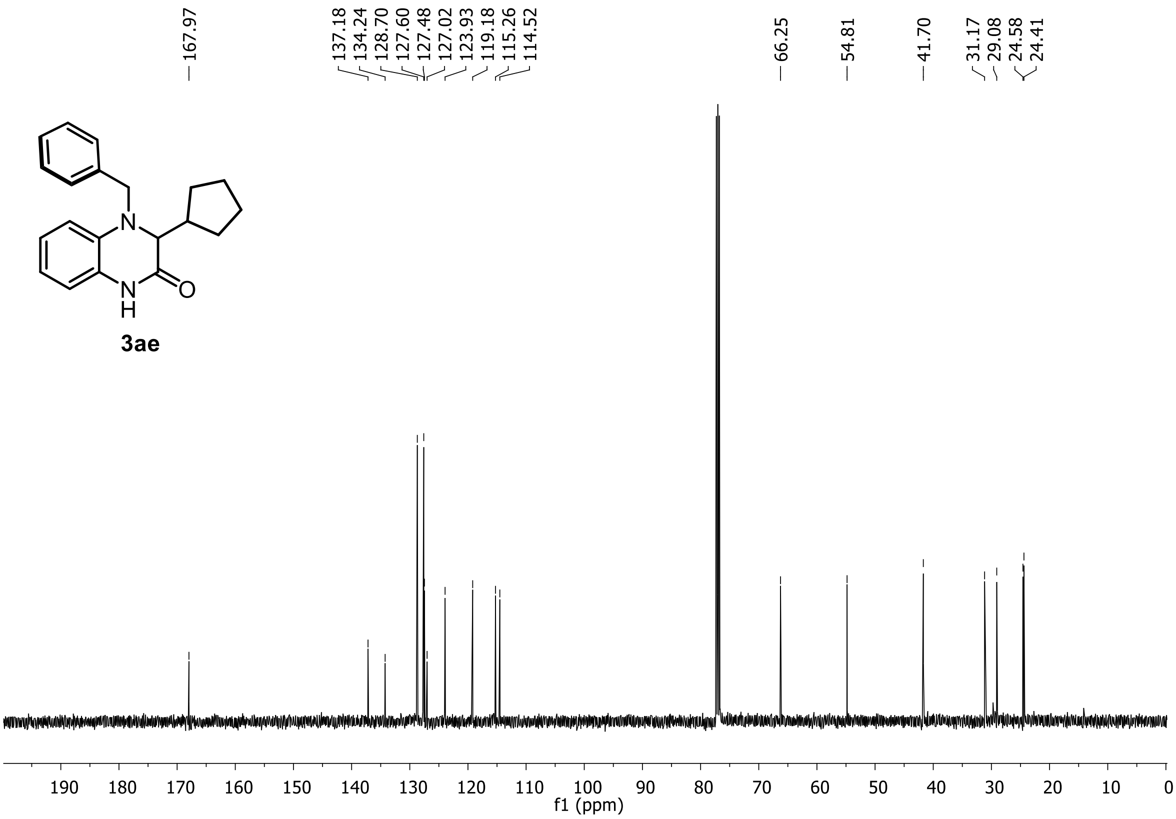
^{13}C{^{1}H} NMR spectrum of 3ad (CDCl$_3$, 126 MHz)



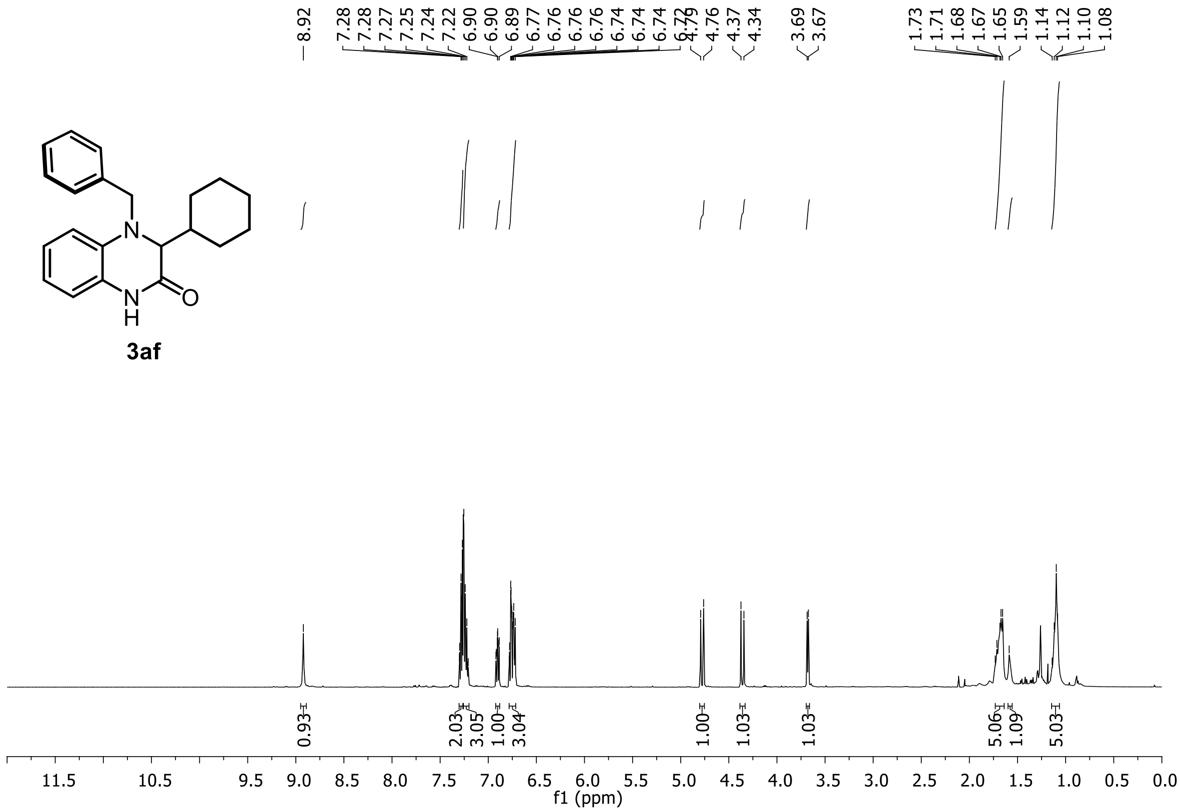
^{1}H NMR spectrum of 3ae (CDCl$_3$, 500 MHz)



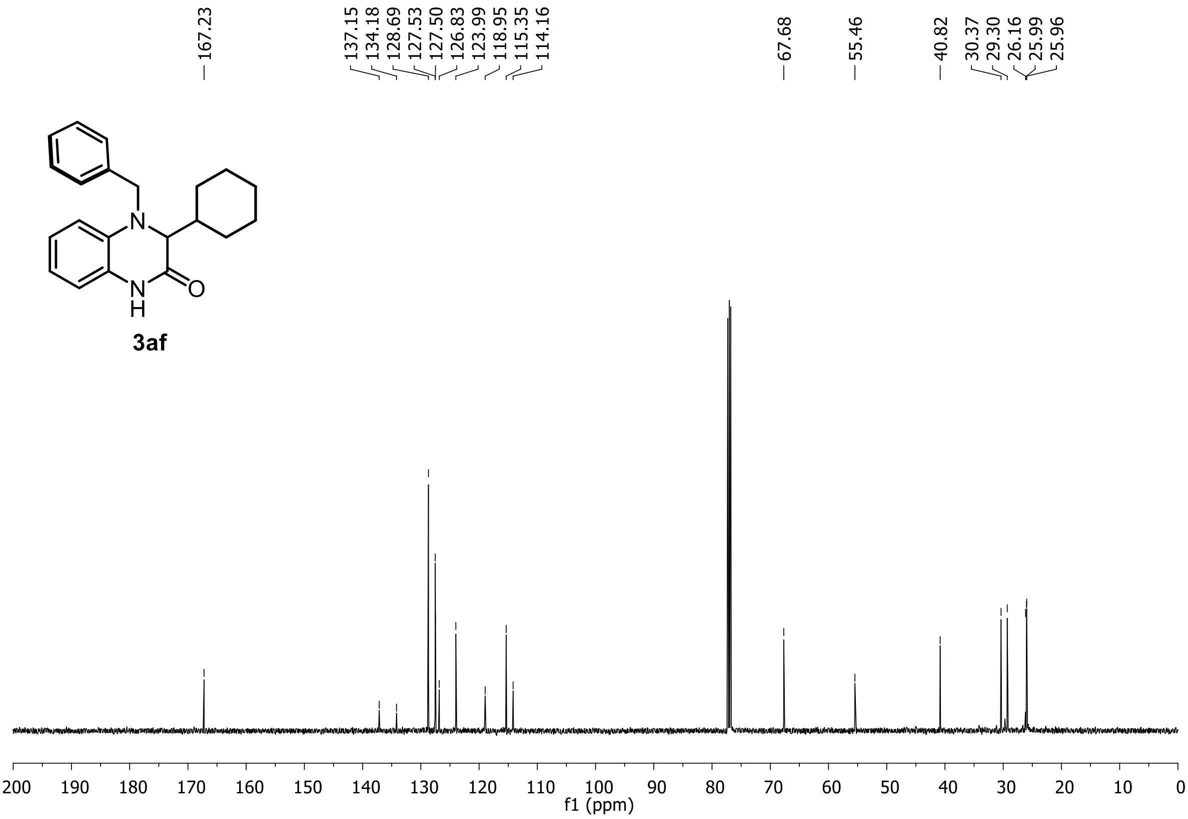
^{13}C{^{1}H} NMR spectrum of 3ae (CDCl$_3$, 126 MHz)



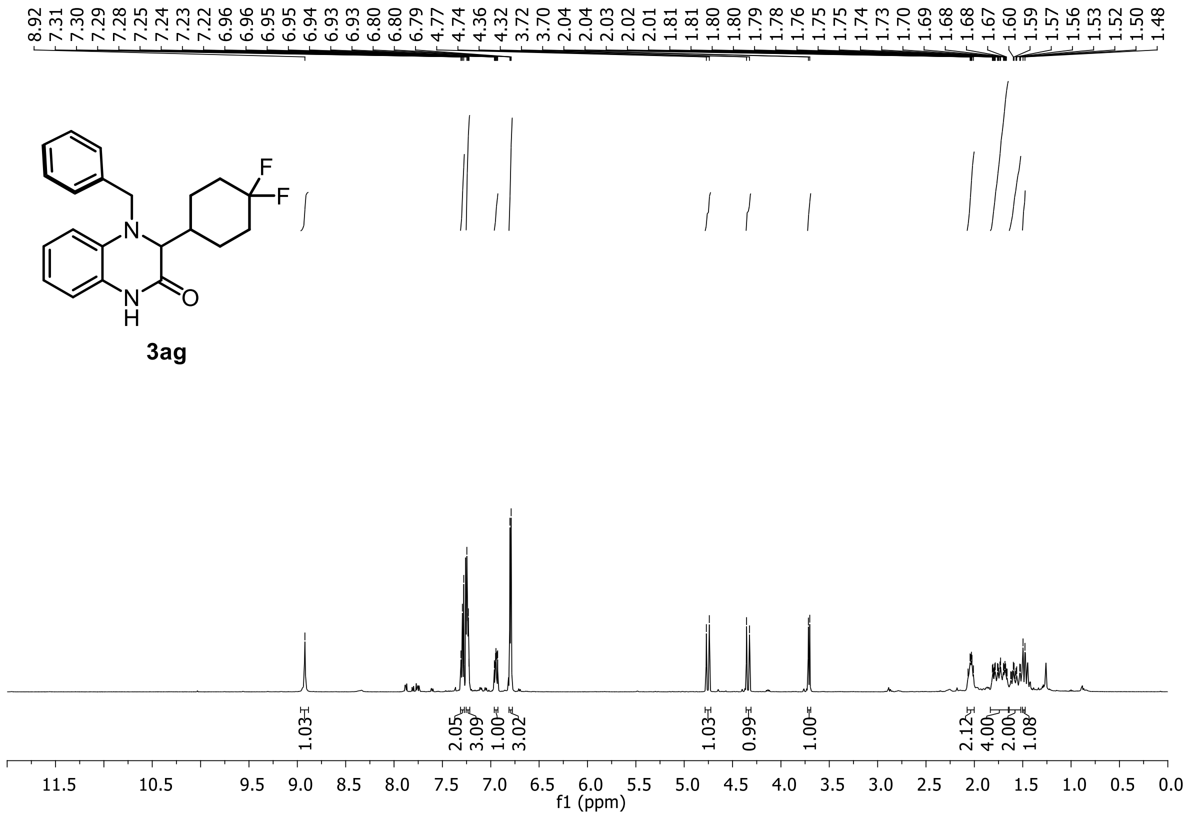
^{1}H NMR spectrum of 3af (CDCl$_3$, 500 MHz)



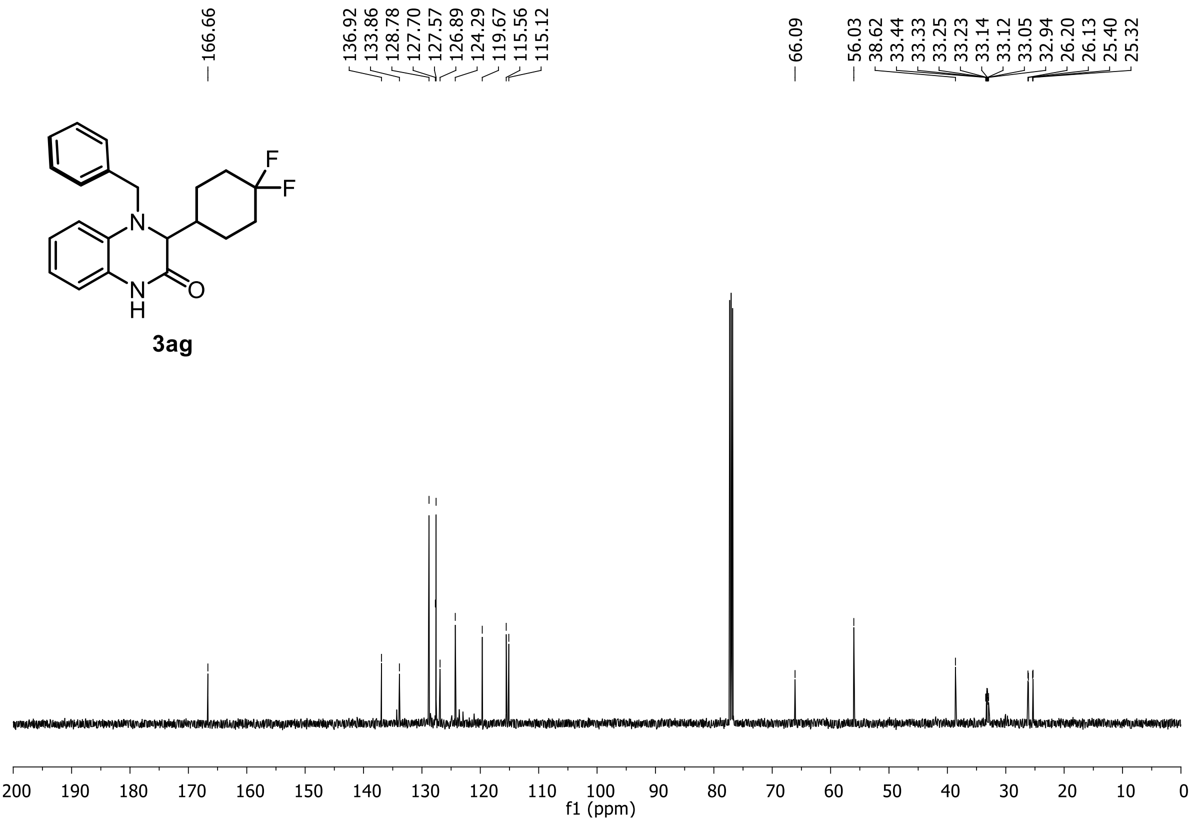
^{13}C{^{1}H} NMR spectrum of 3af (CDCl$_3$, 126 MHz)



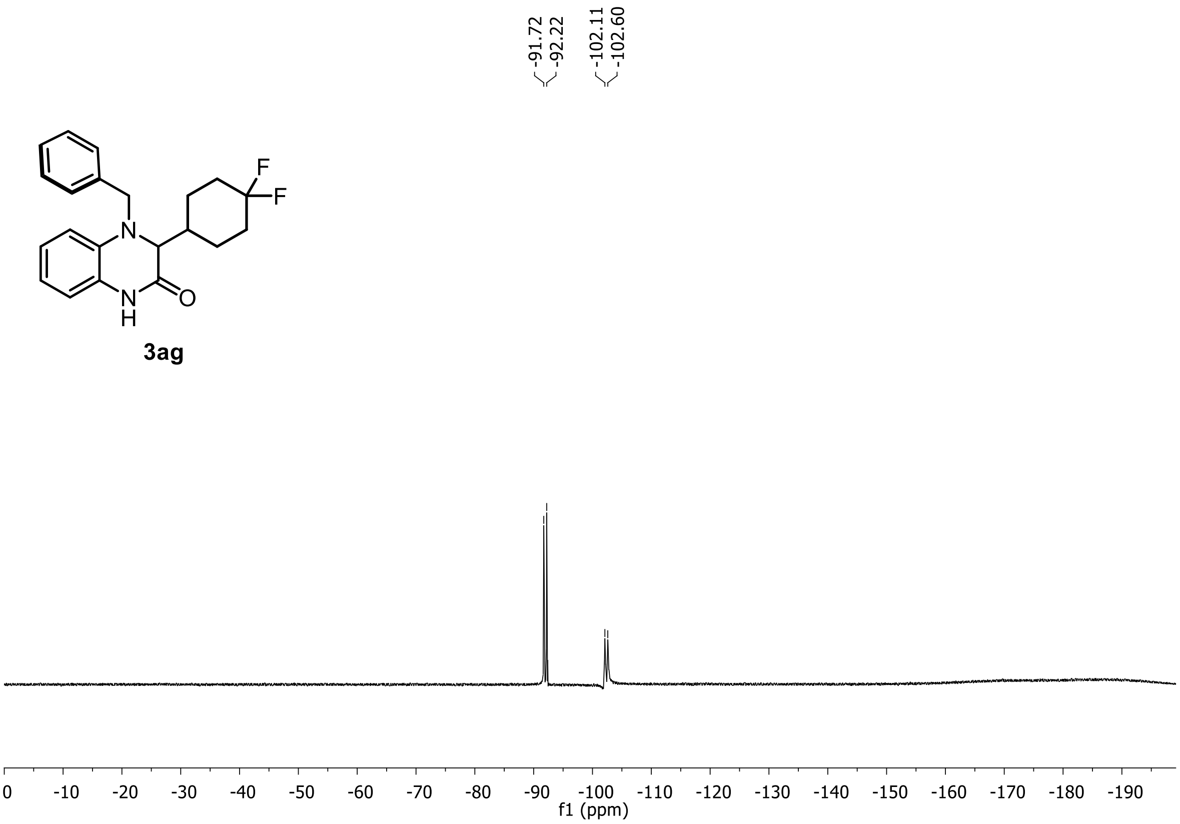
^{1}H NMR spectrum of 3ag (CDCl$_3$, 500 MHz)



^{13}C{^{1}H} NMR spectrum of 3ag (CDCl$_3$, 126 MHz)

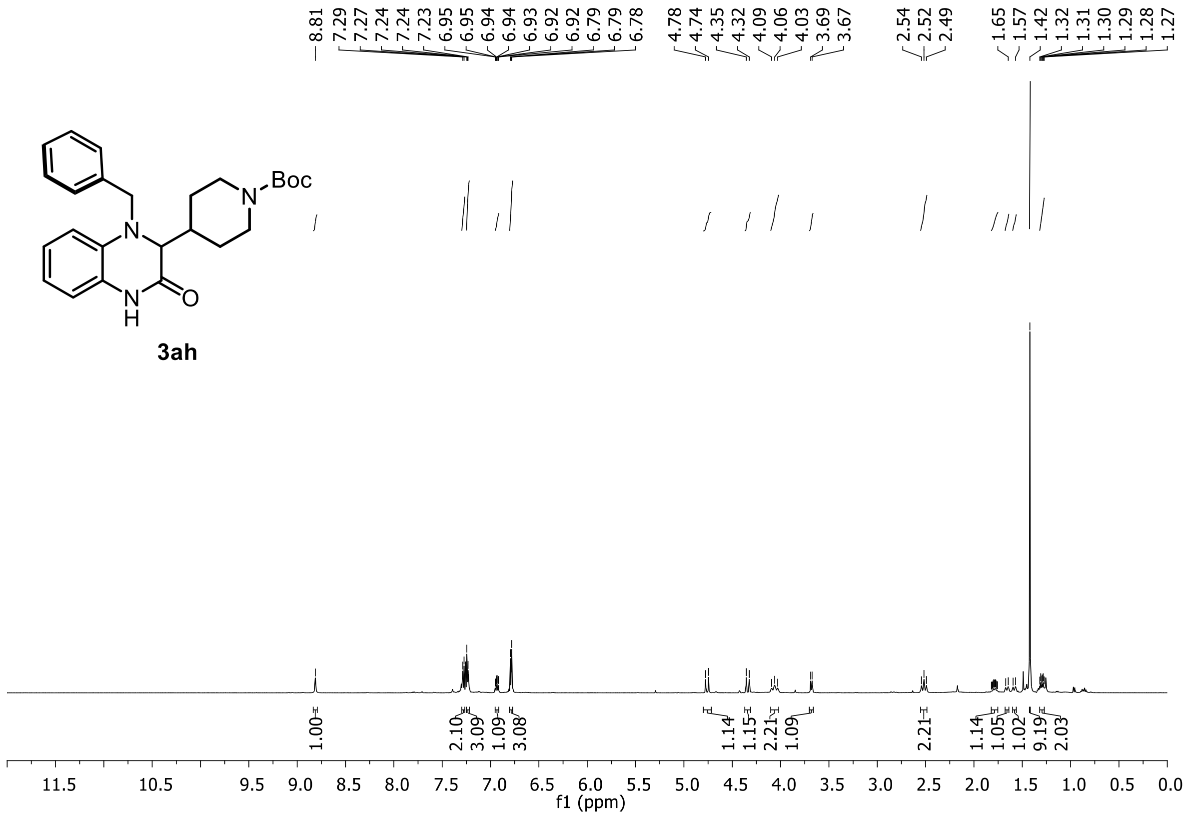


$^{19}F\{^1H\}$ NMR spectrum of 3ag ($CDCl_3$, 471 MHz)

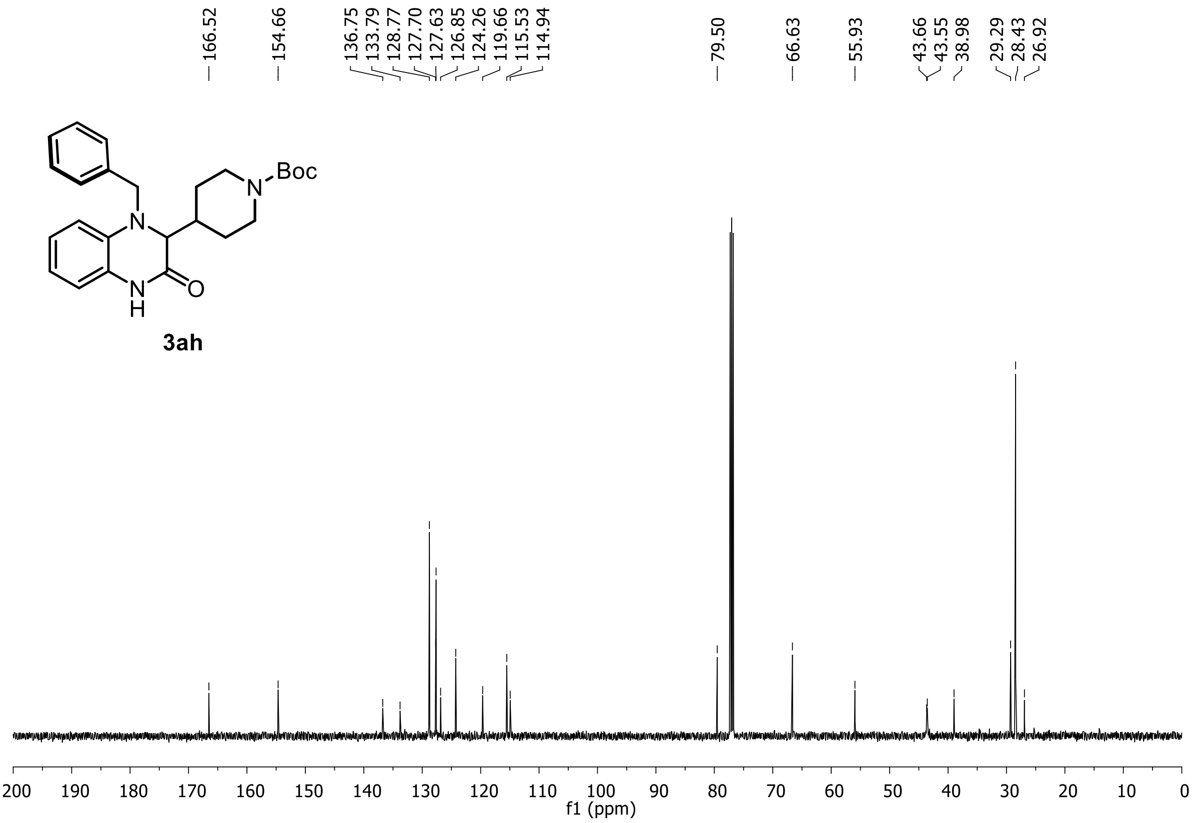


3ag

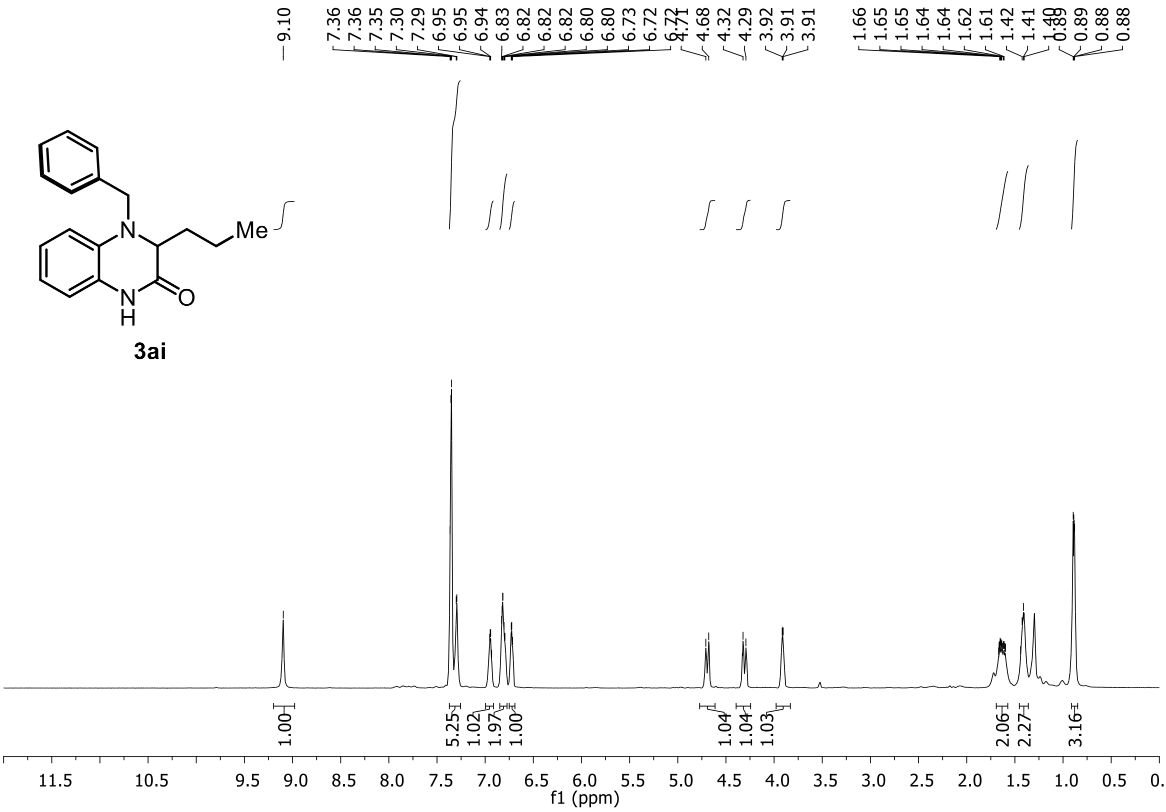
^{1}H NMR spectrum of 3ah ($CDCl_3$, 500 MHz)



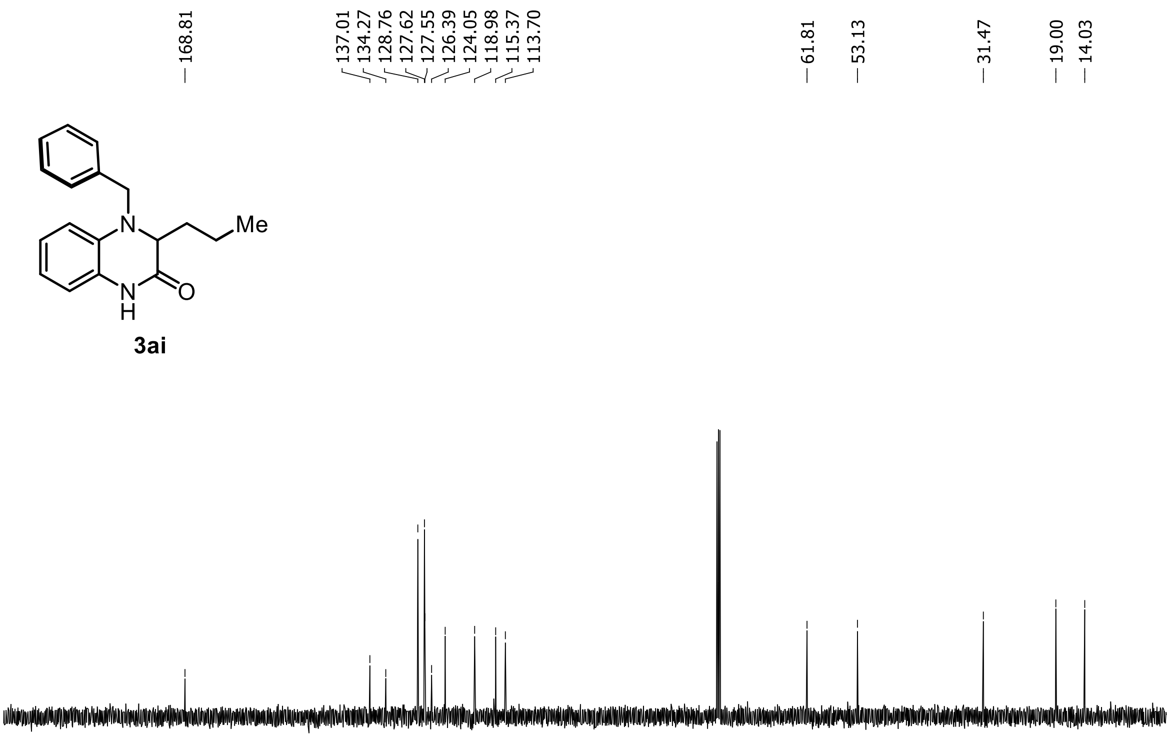
$^{13}C\{^{1}H\}$ NMR spectrum of 3ah ($CDCl_3$, 126 MHz)



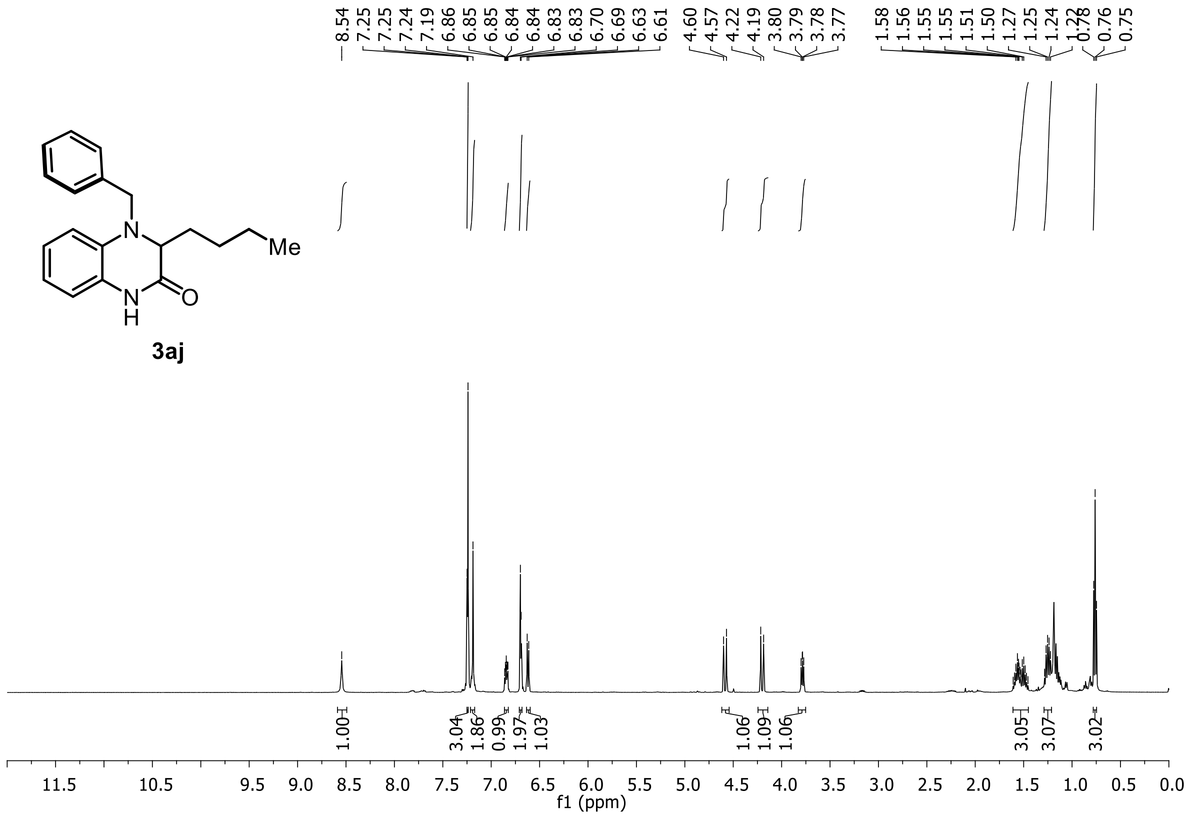
^{1}H NMR spectrum of 3ai (CDCl$_3$, 500 MHz)



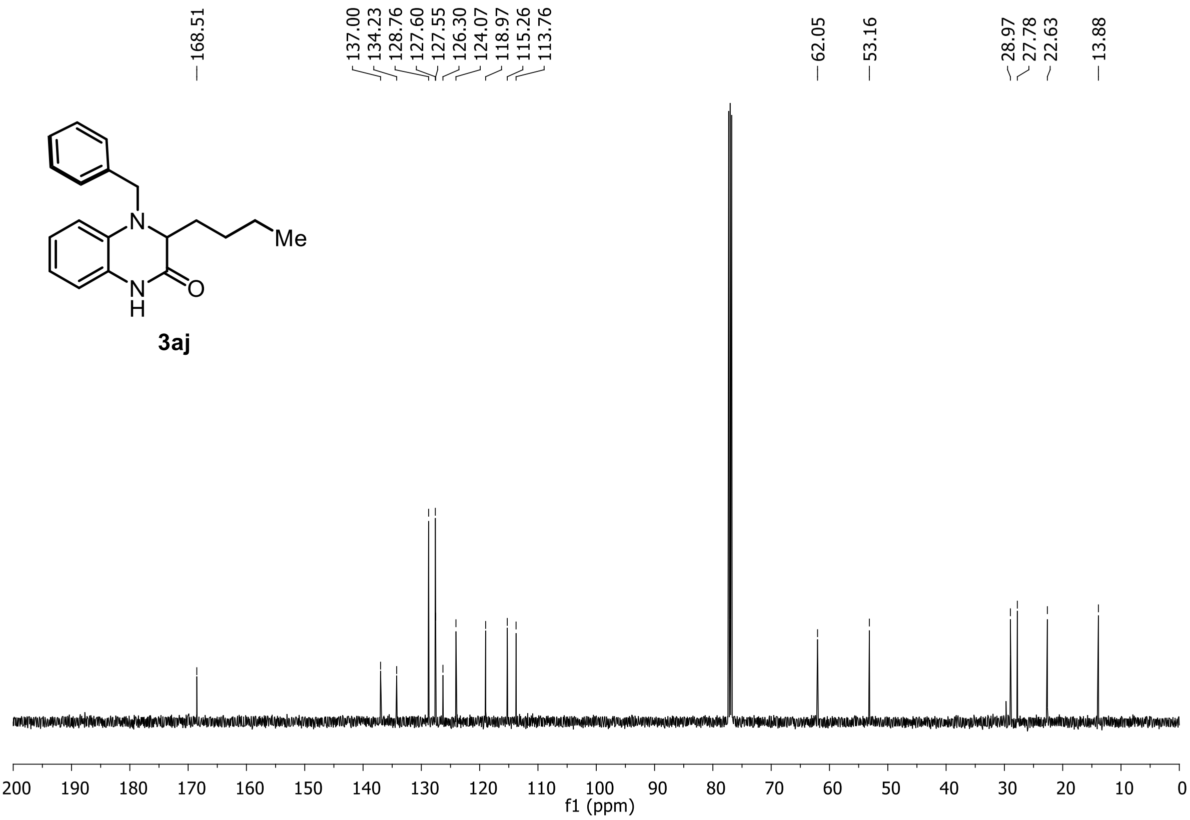
^{13}C{^{1}H} NMR spectrum of 3ai (CDCl$_3$, 126 MHz)



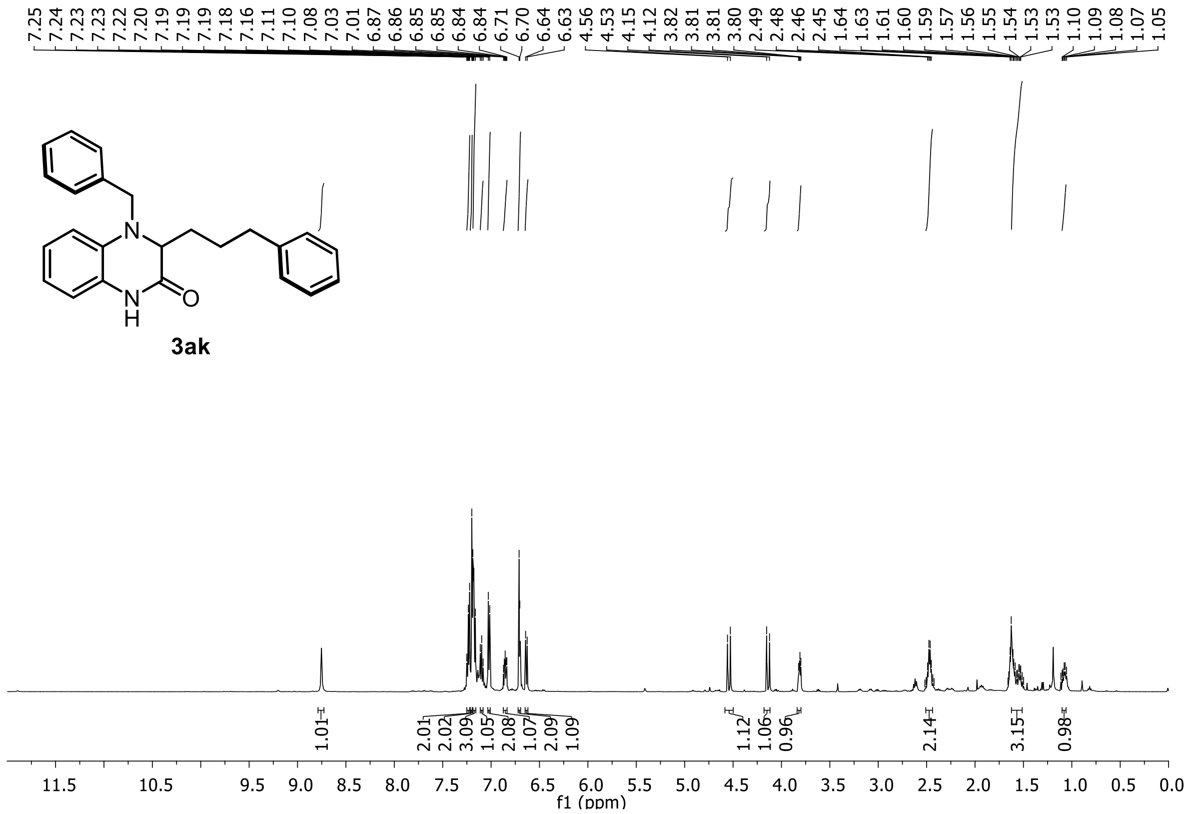
^{1}H NMR spectrum of 3aj ($CDCl_3$, 500 MHz)



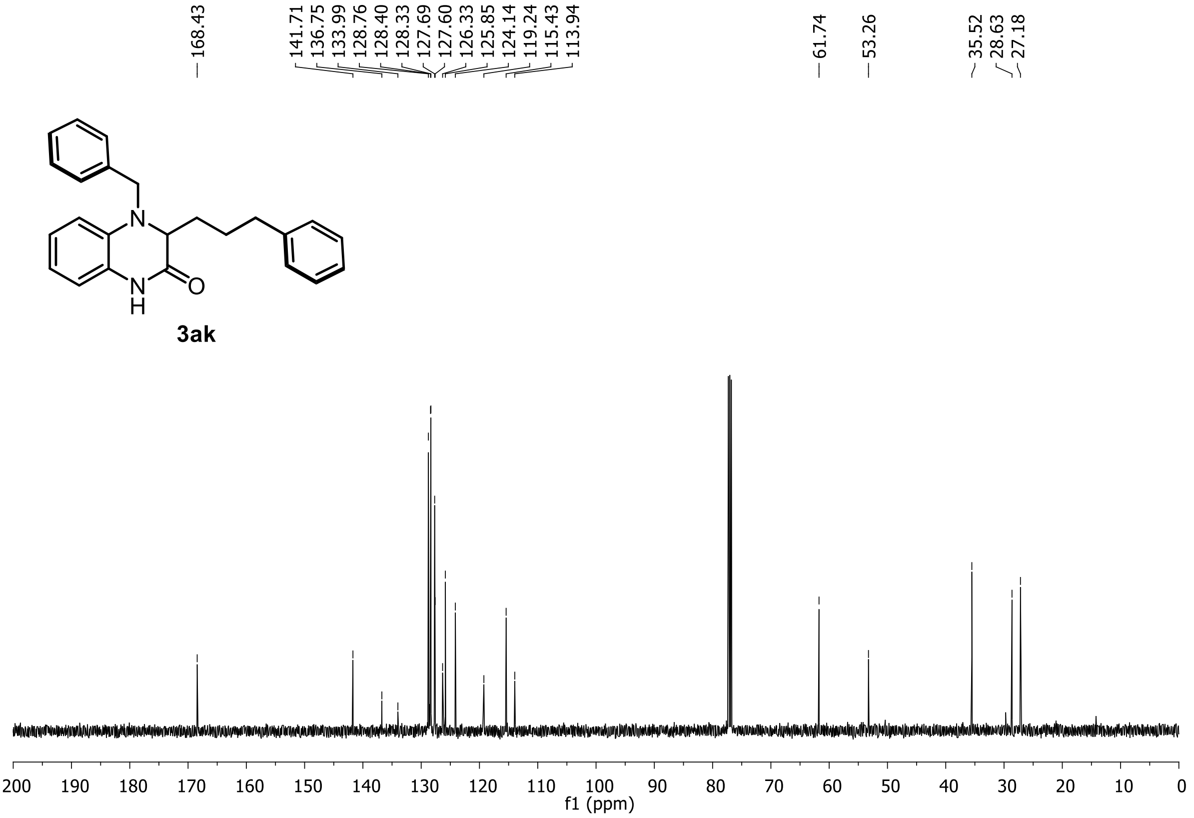
$^{13}C\{^{1}H\}$ NMR spectrum of 3aj ($CDCl_3$, 126 MHz)



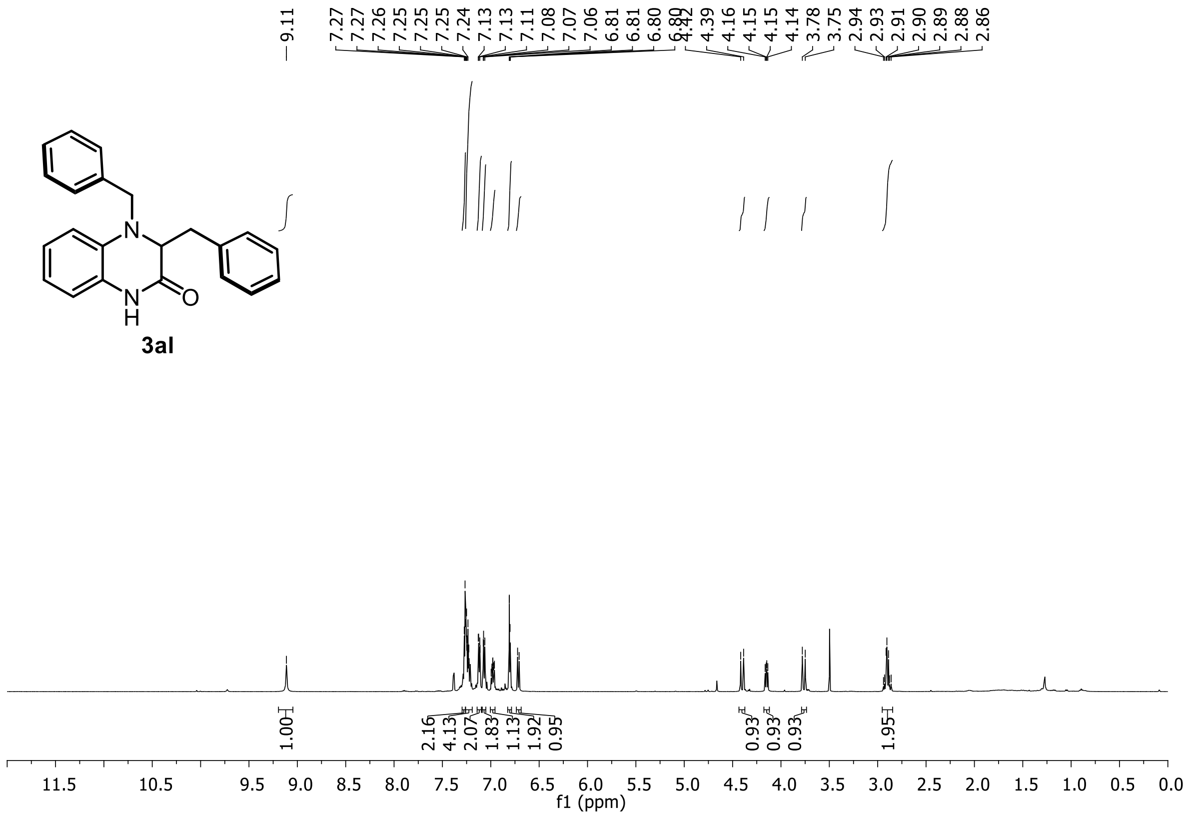
^{1}H NMR spectrum of 3ak ($CDCl_3$, 500 MHz)



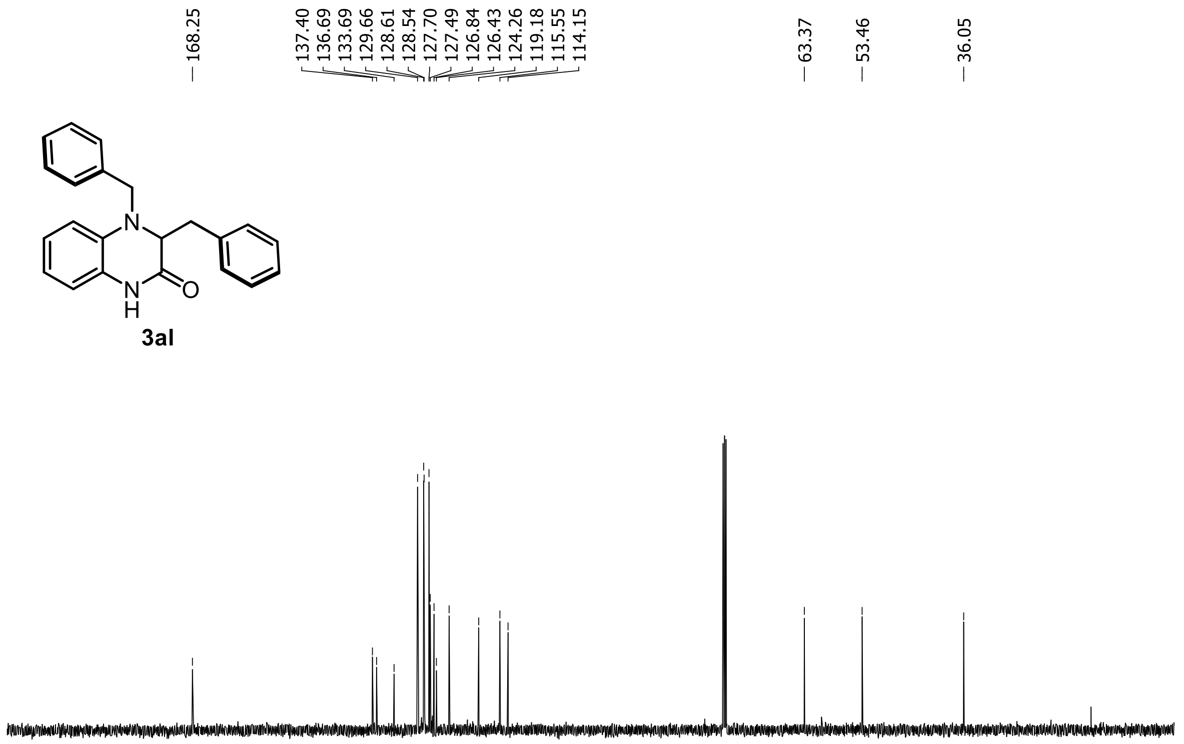
^{13}C{^{1}H} NMR spectrum of 3ak ($CDCl_3$, 126 MHz)



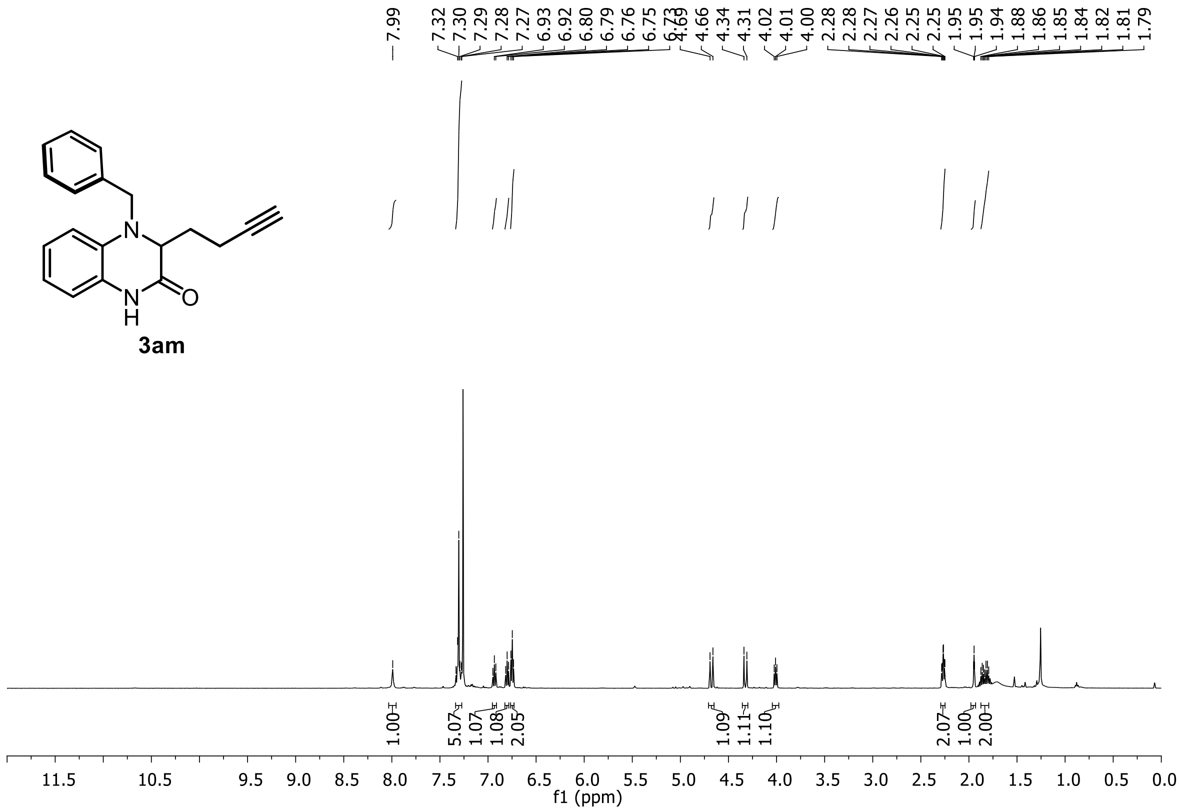
^{1}H NMR spectrum of 3al (CDCl$_3$, 500 MHz)



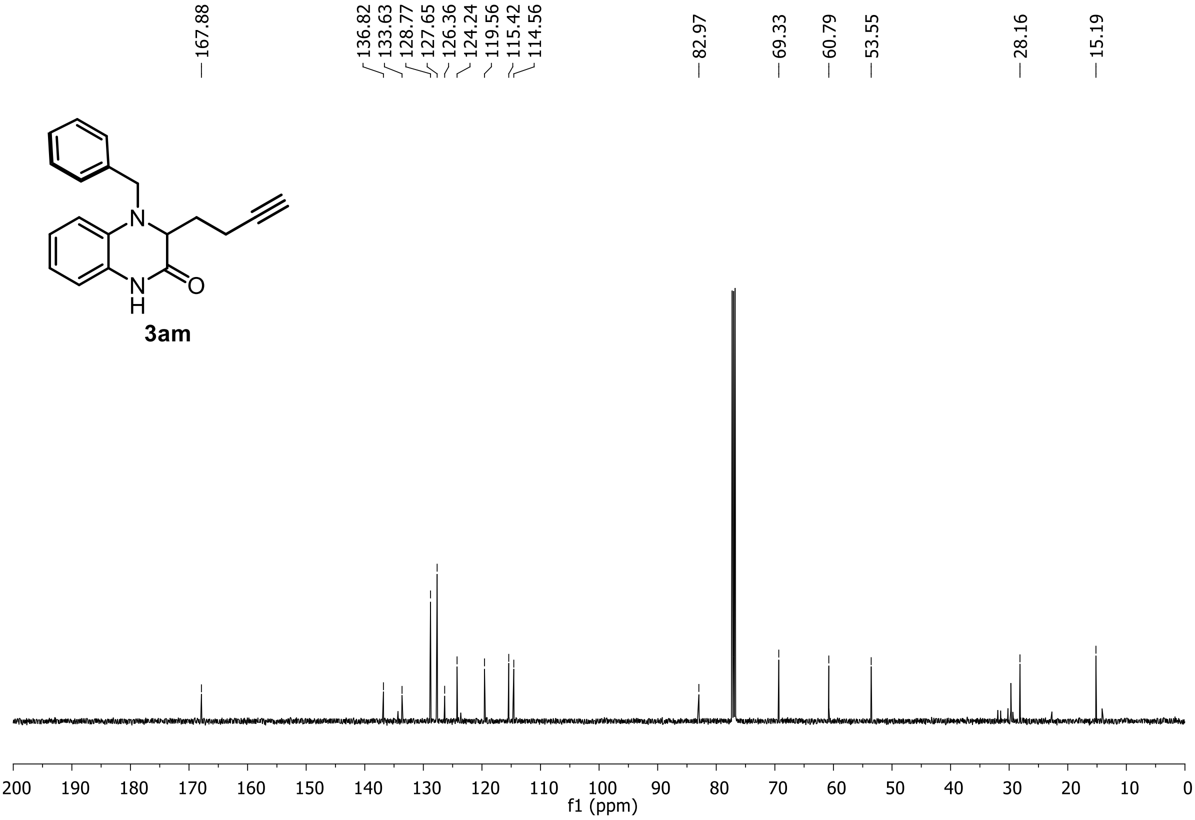
^{13}C{^{1}H} NMR spectrum of 3al (CDCl$_3$, 126 MHz)



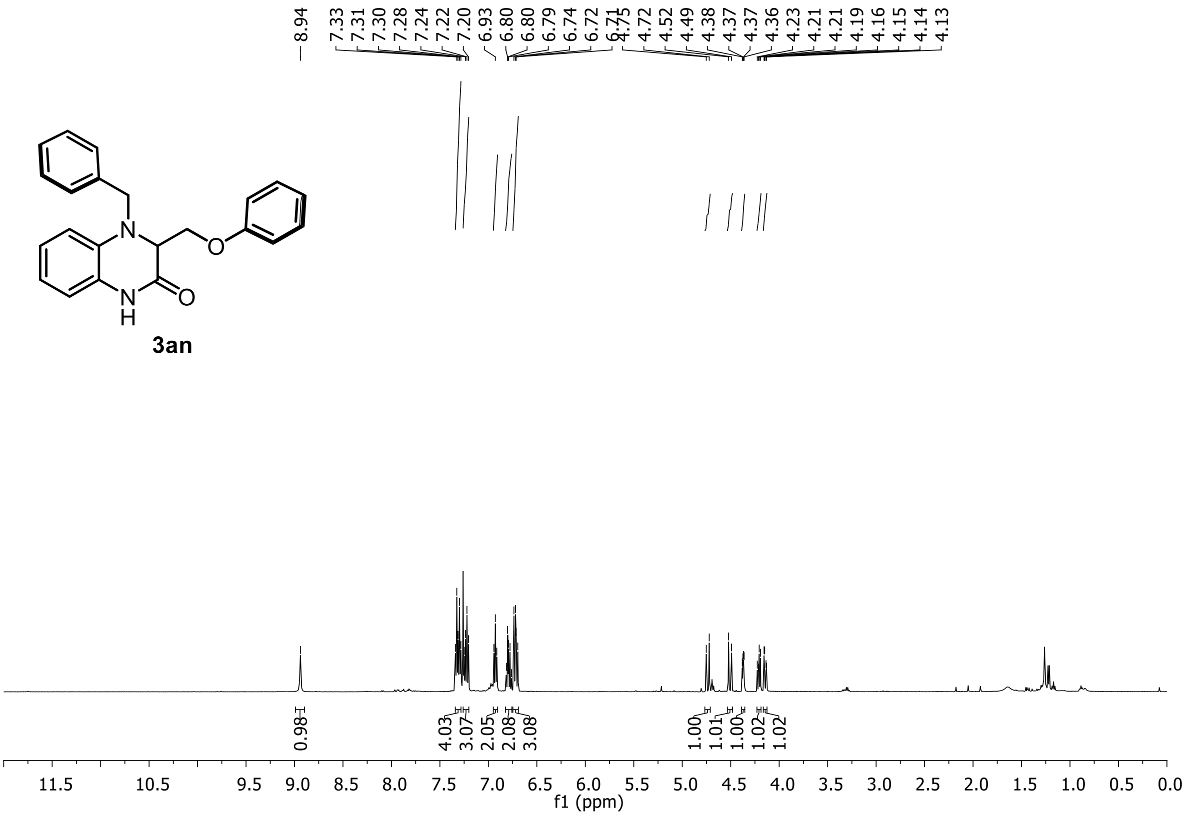
^{1}H NMR spectrum of 3am ($CDCl_3$, 500 MHz)



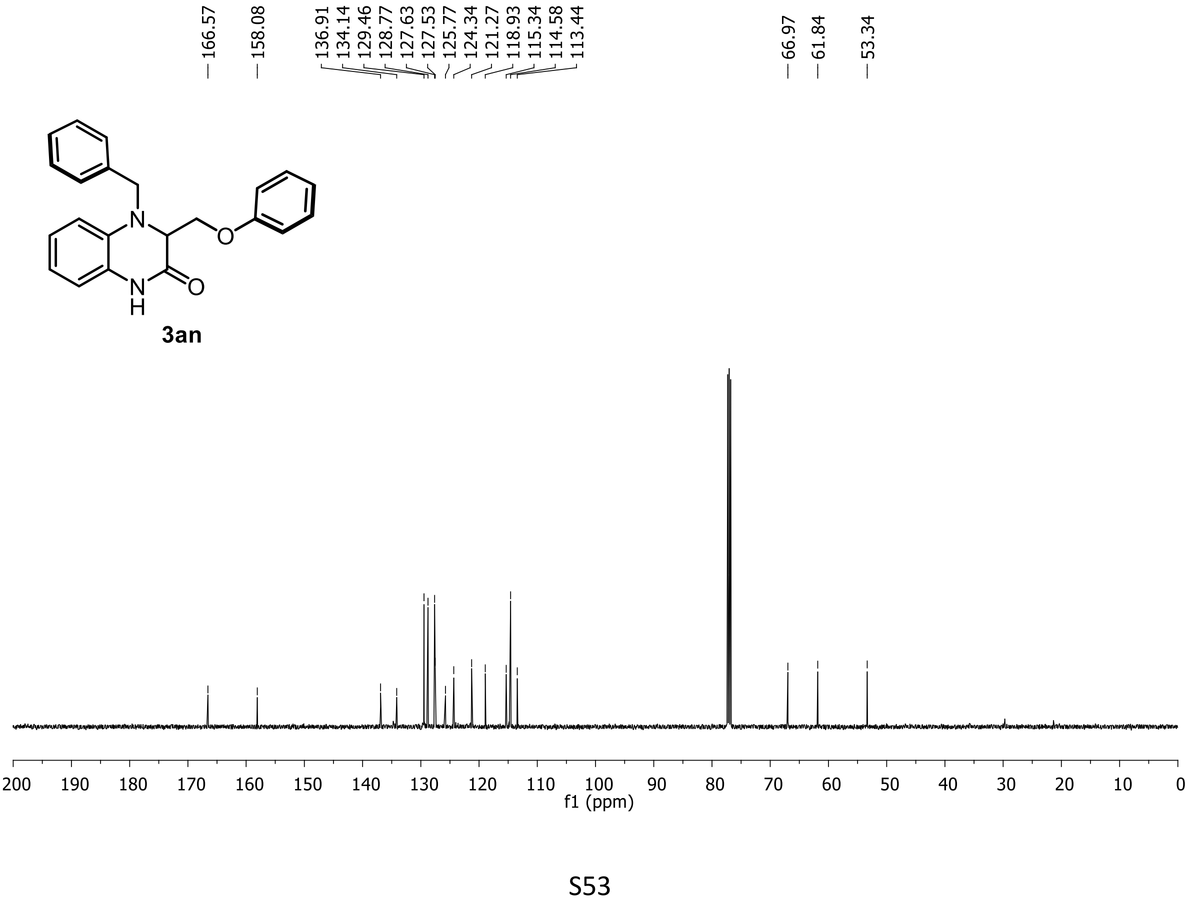
$^{13}C\{^{1}H\}$ NMR spectrum of 3am ($CDCl_3$, 126 MHz)



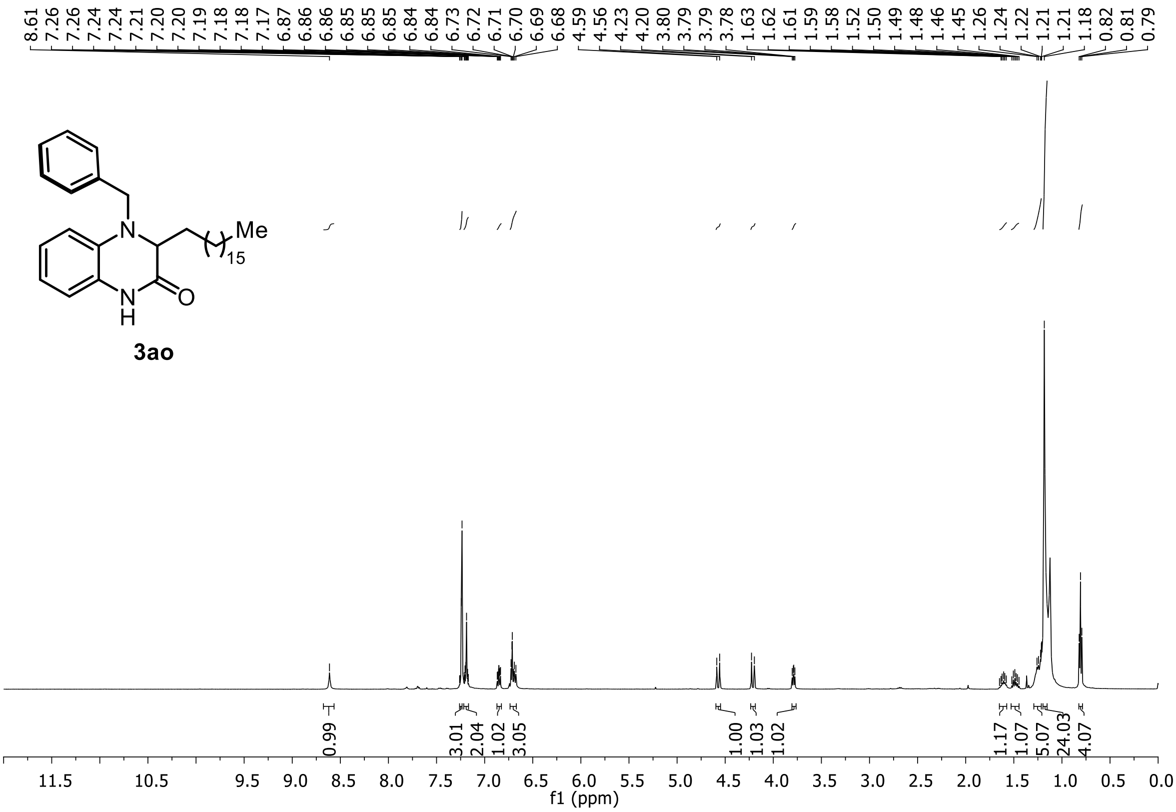
^{1}H NMR spectrum of 3an ($CDCl_3$, 500 MHz)



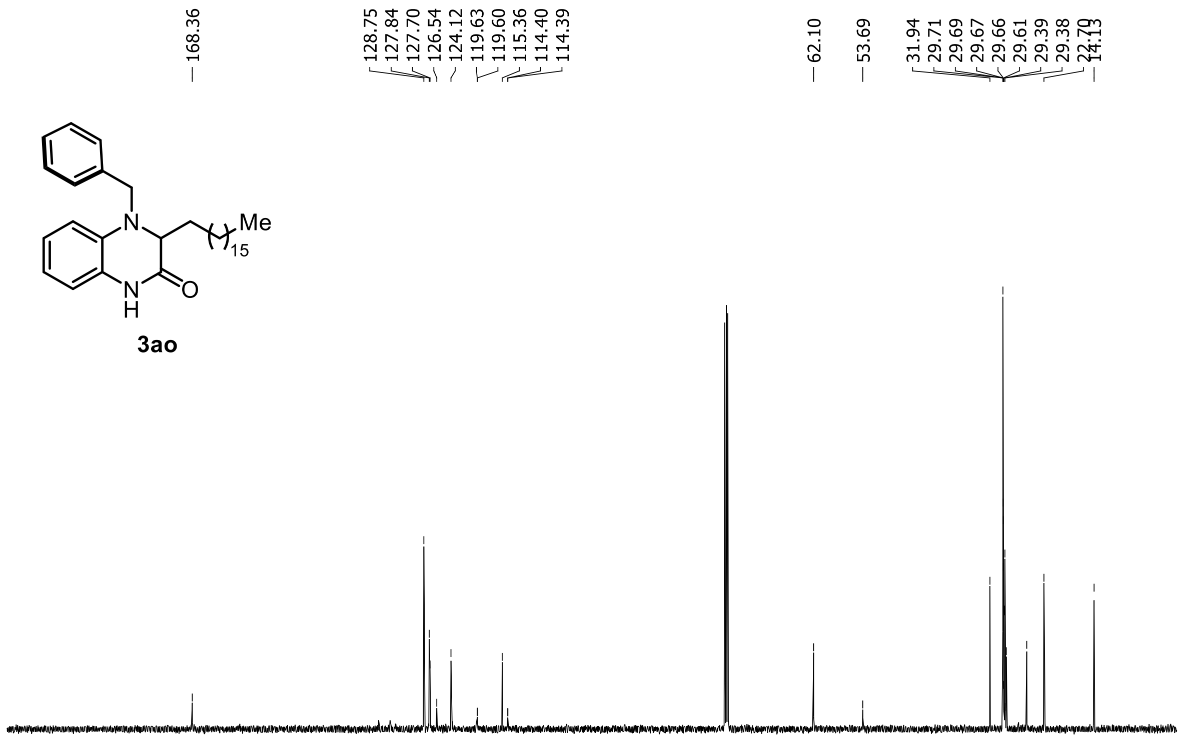
$^{13}C\{^{1}H\}$ NMR spectrum of 3an ($CDCl_3$, 126 MHz)



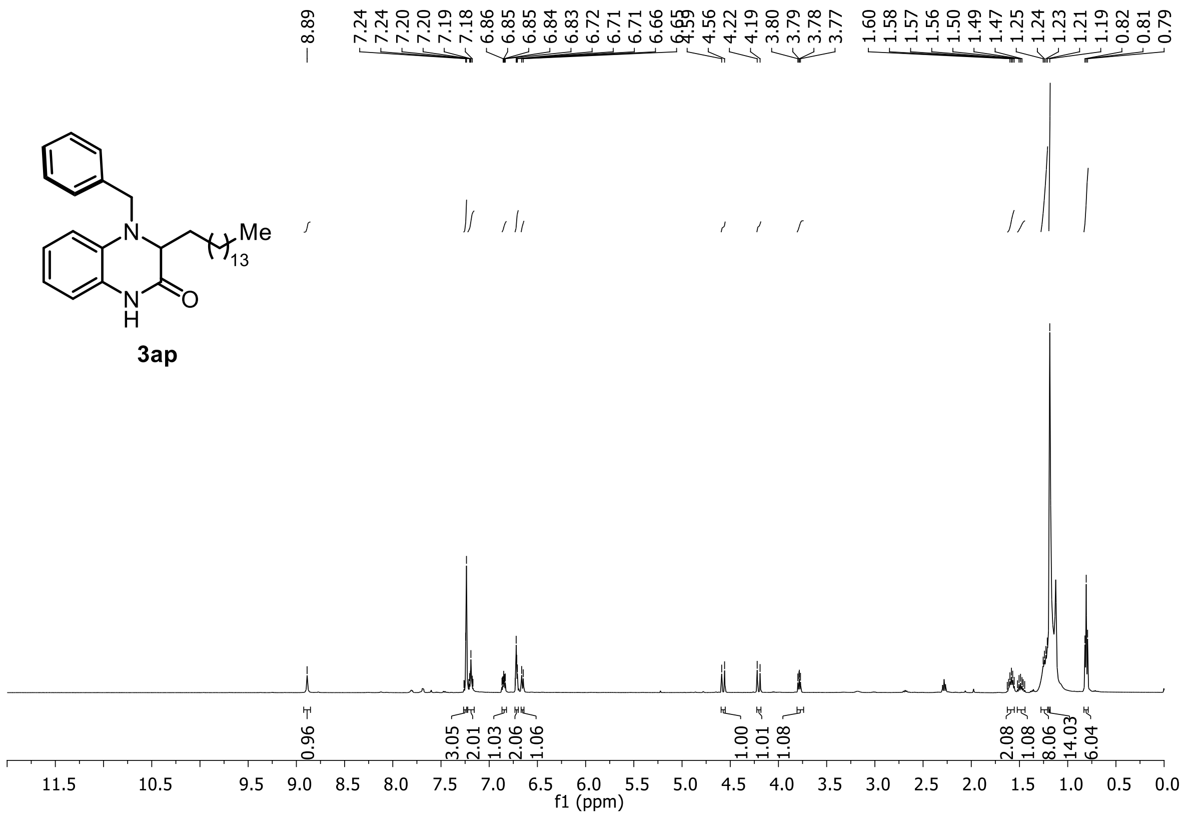
^{1}H NMR spectrum of 3ao ($CDCl_3$, 500 MHz)



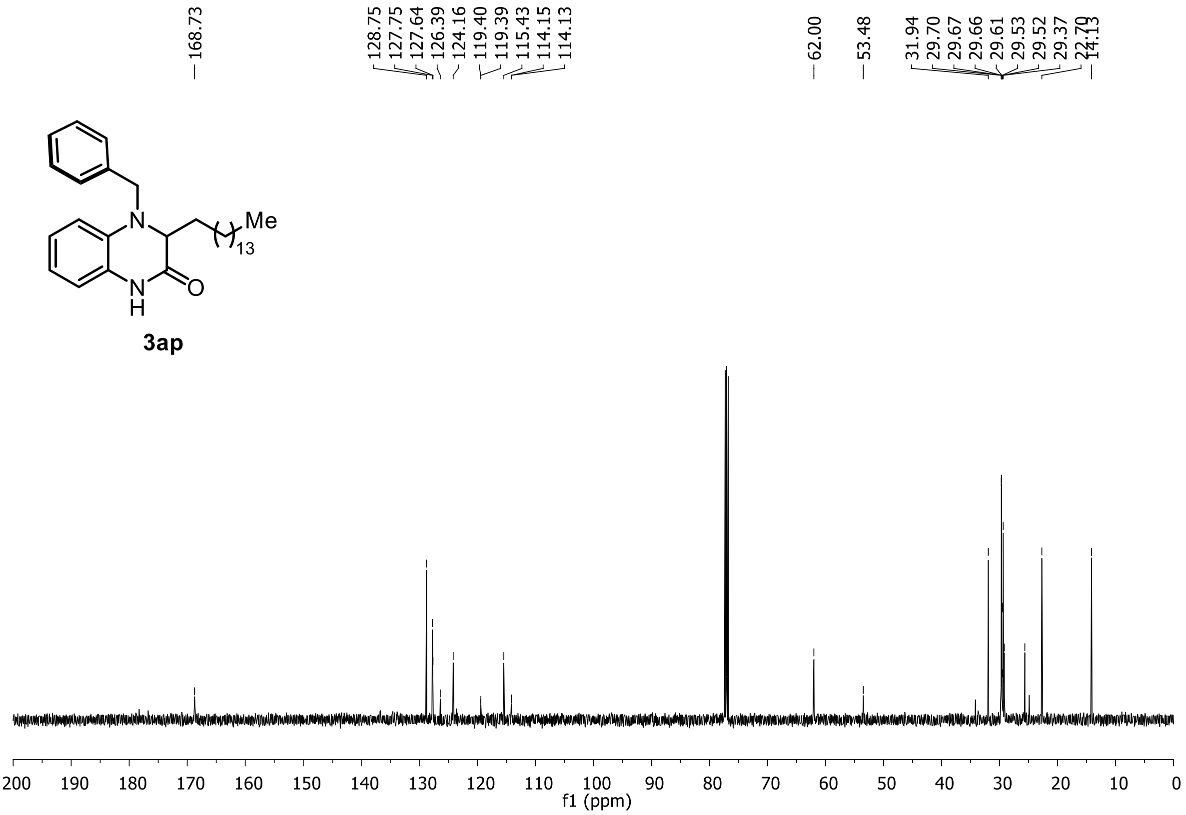
^{13}C{^{1}H} NMR spectrum of 3ao ($CDCl_3$, 126 MHz)



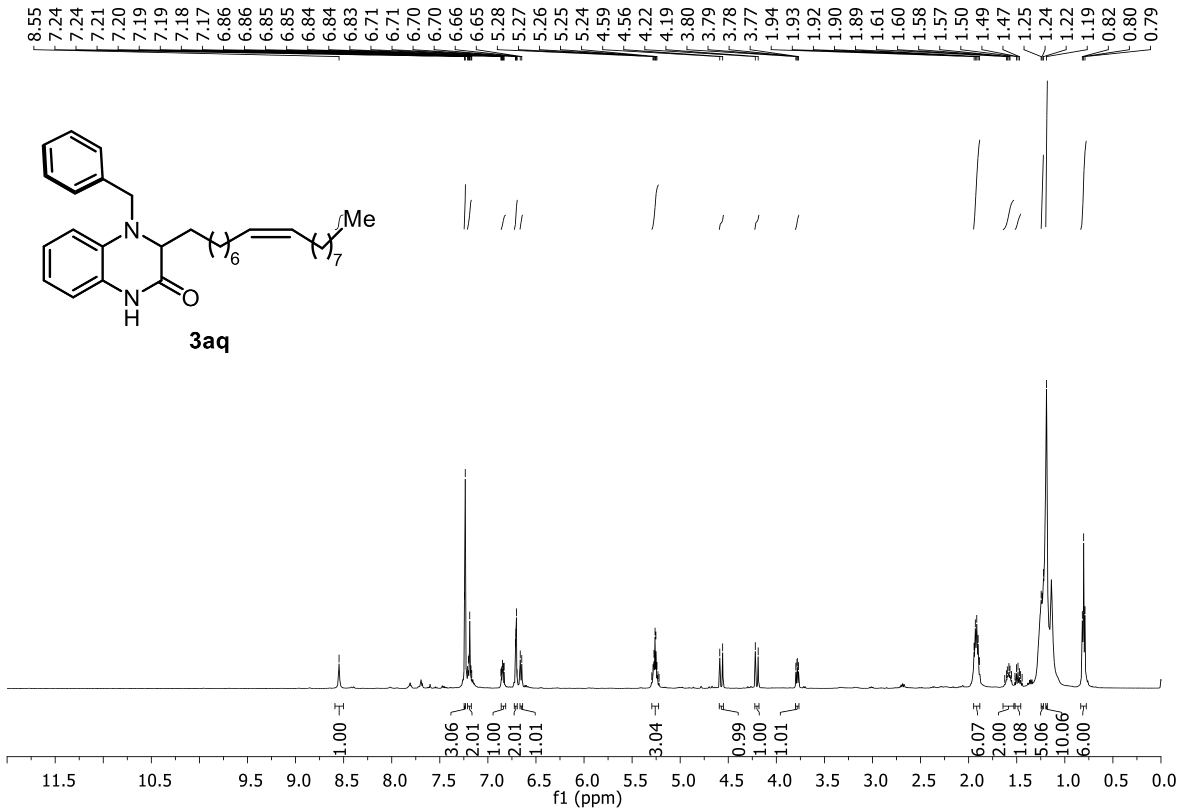
^{1}H NMR spectrum of 3ap (CDCl$_3$, 500 MHz)



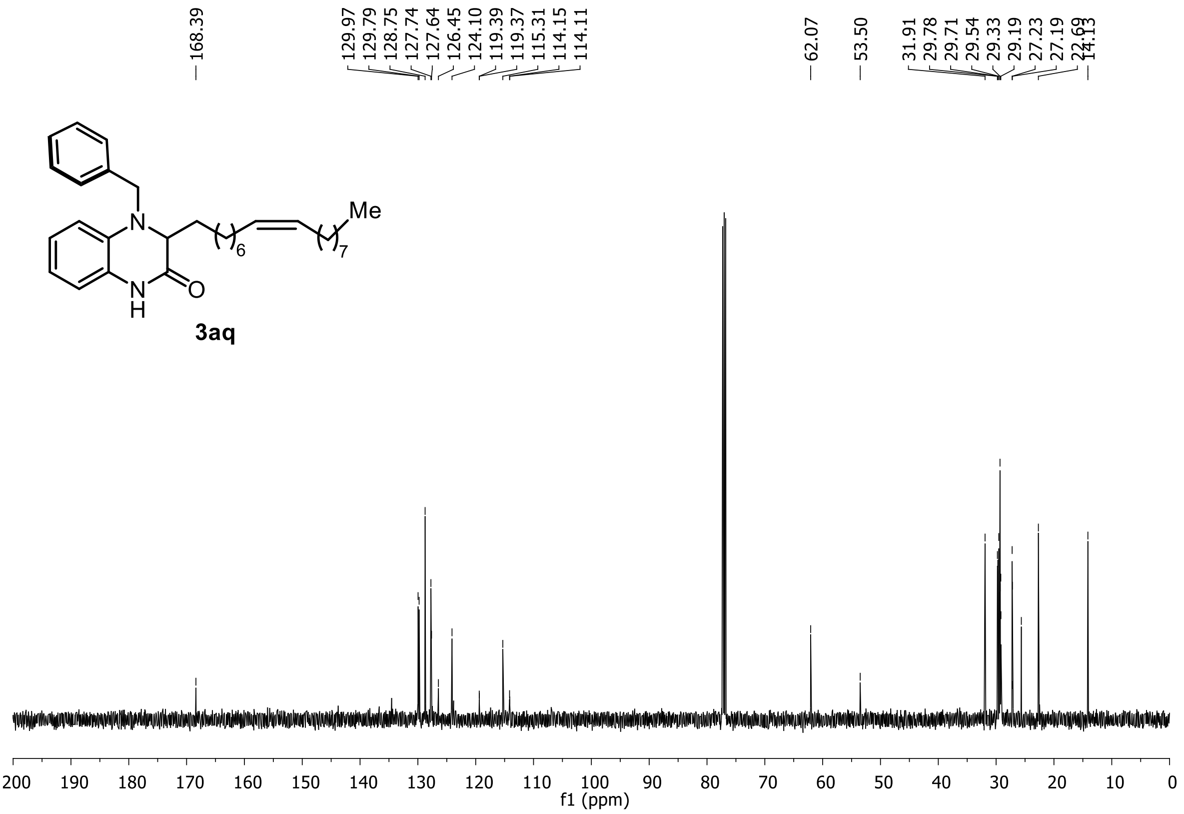
^{13}C{^{1}H} NMR spectrum of 3ap (CDCl$_3$, 126 MHz)



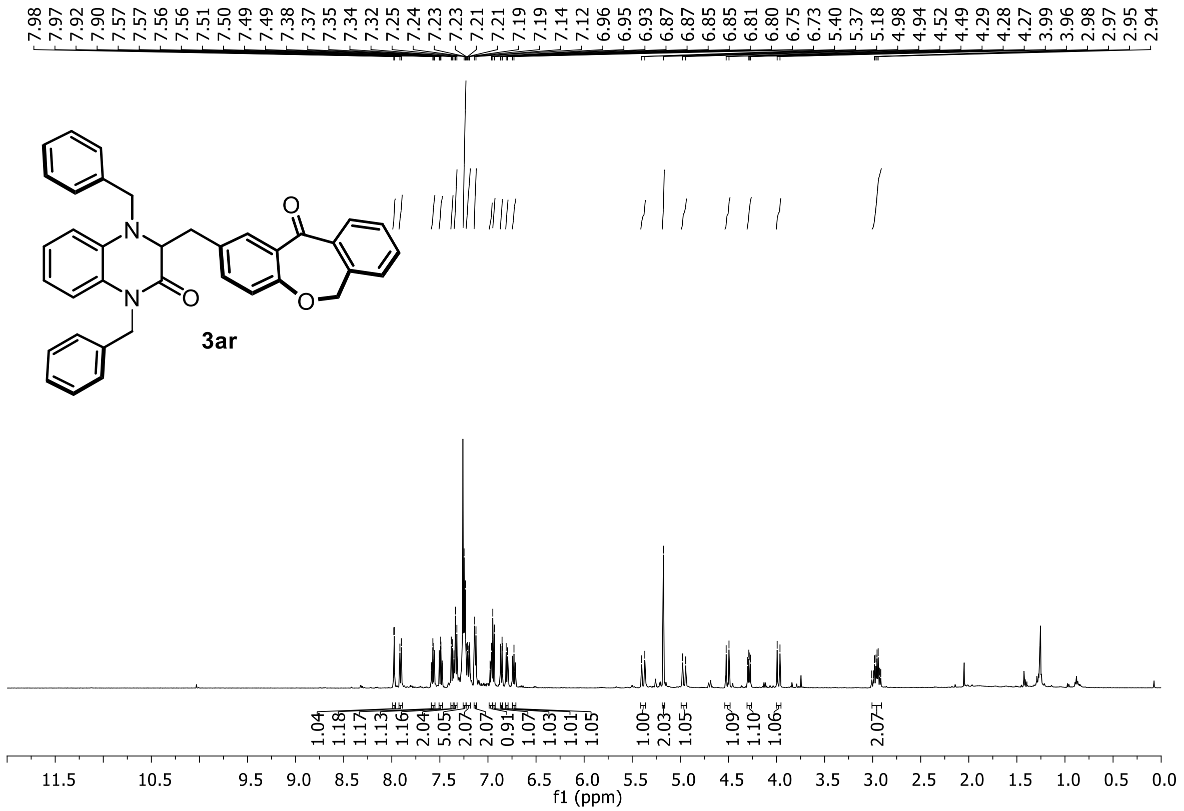
^{1}H NMR spectrum of 3aq ($CDCl_3$, 500 MHz)



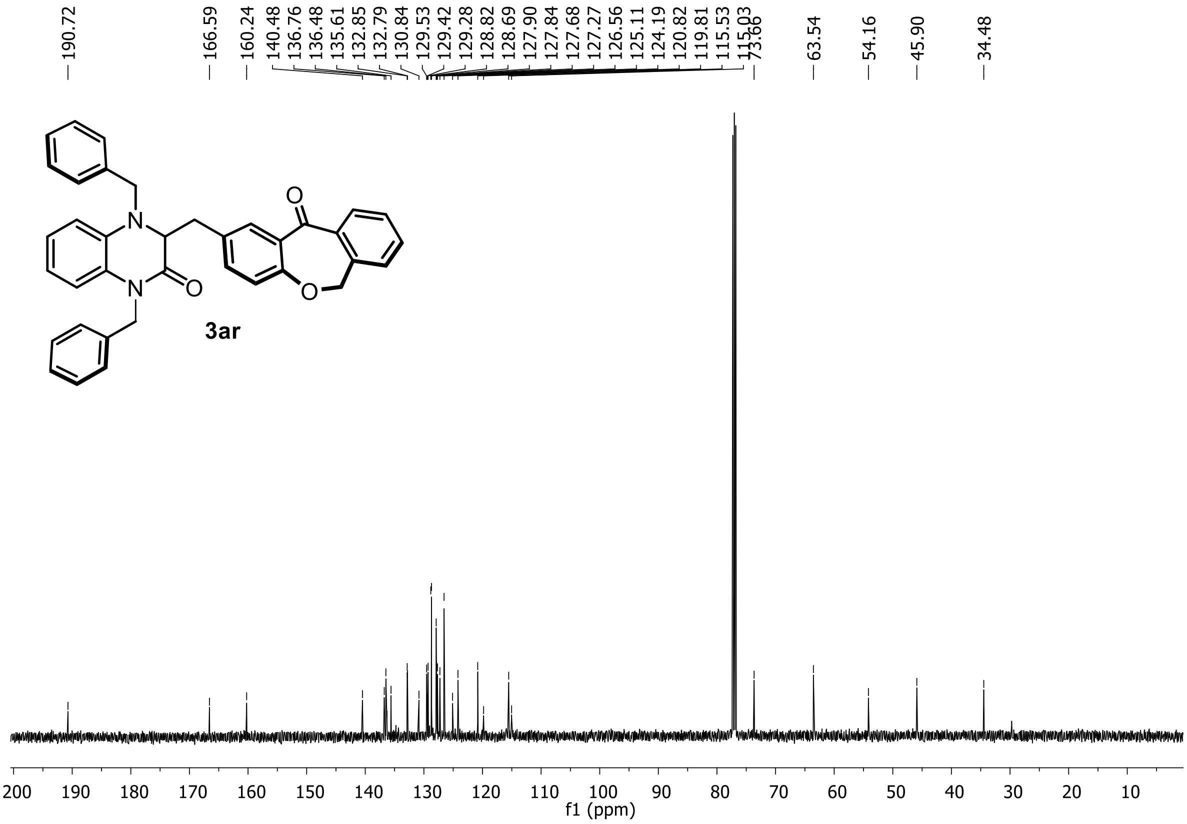
$^{13}C\{^{1}H\}$ NMR spectrum of 3aq ($CDCl_3$, 126 MHz)



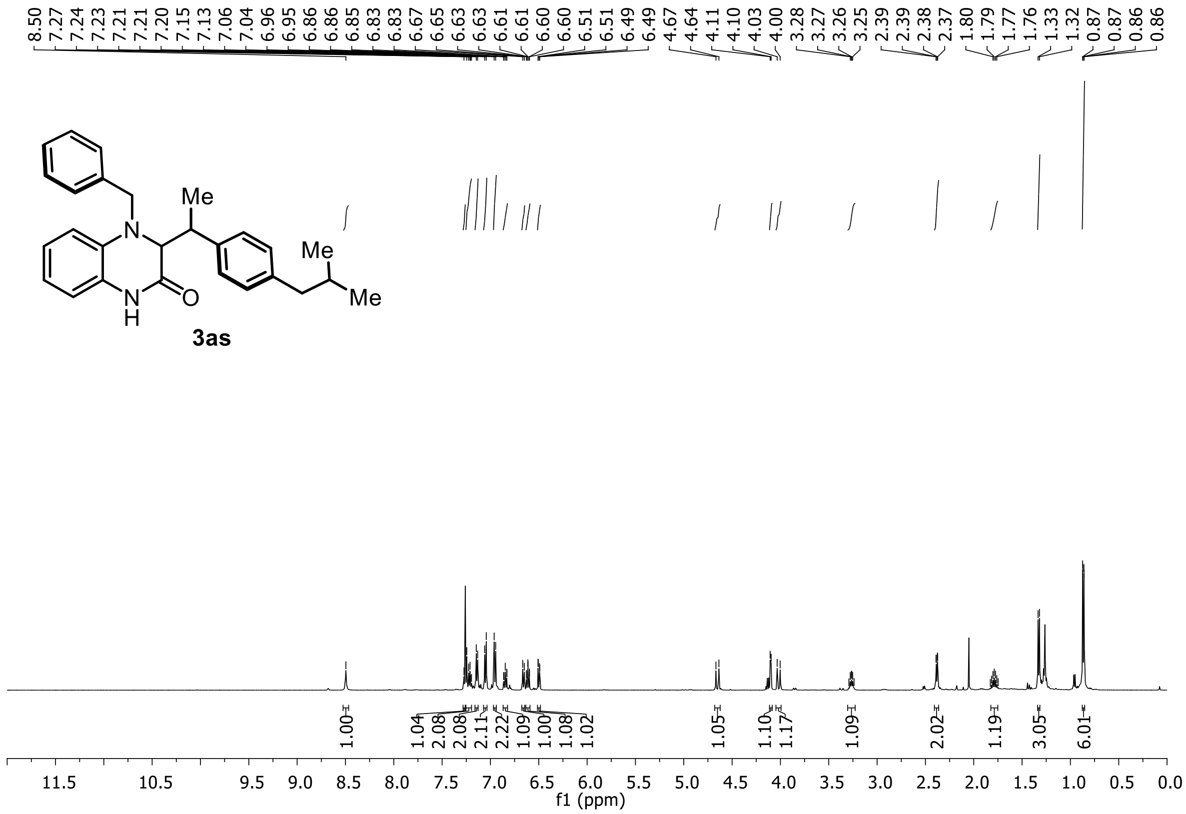
^{1}H NMR spectrum of 3ar ($CDCl_3$, 500 MHz)



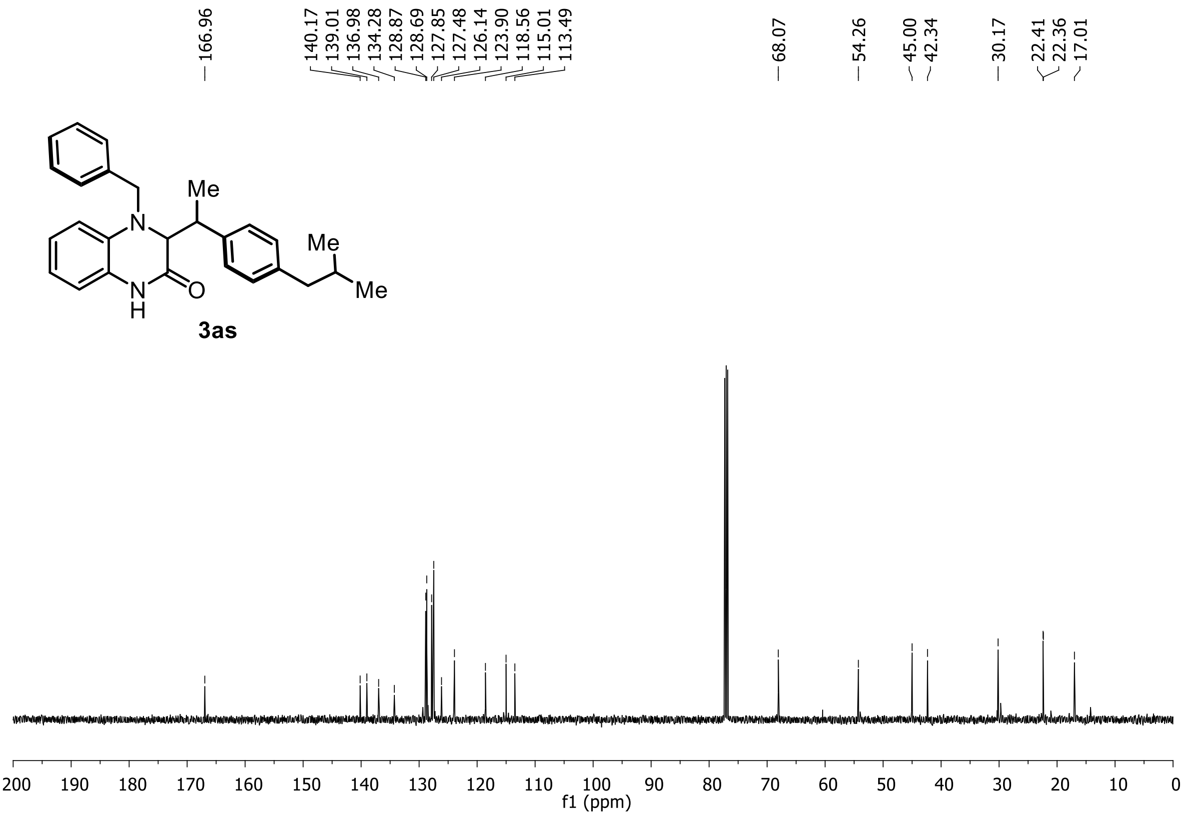
^{13}C{^{1}H} NMR spectrum of 3ar ($CDCl_3$, 126 MHz)



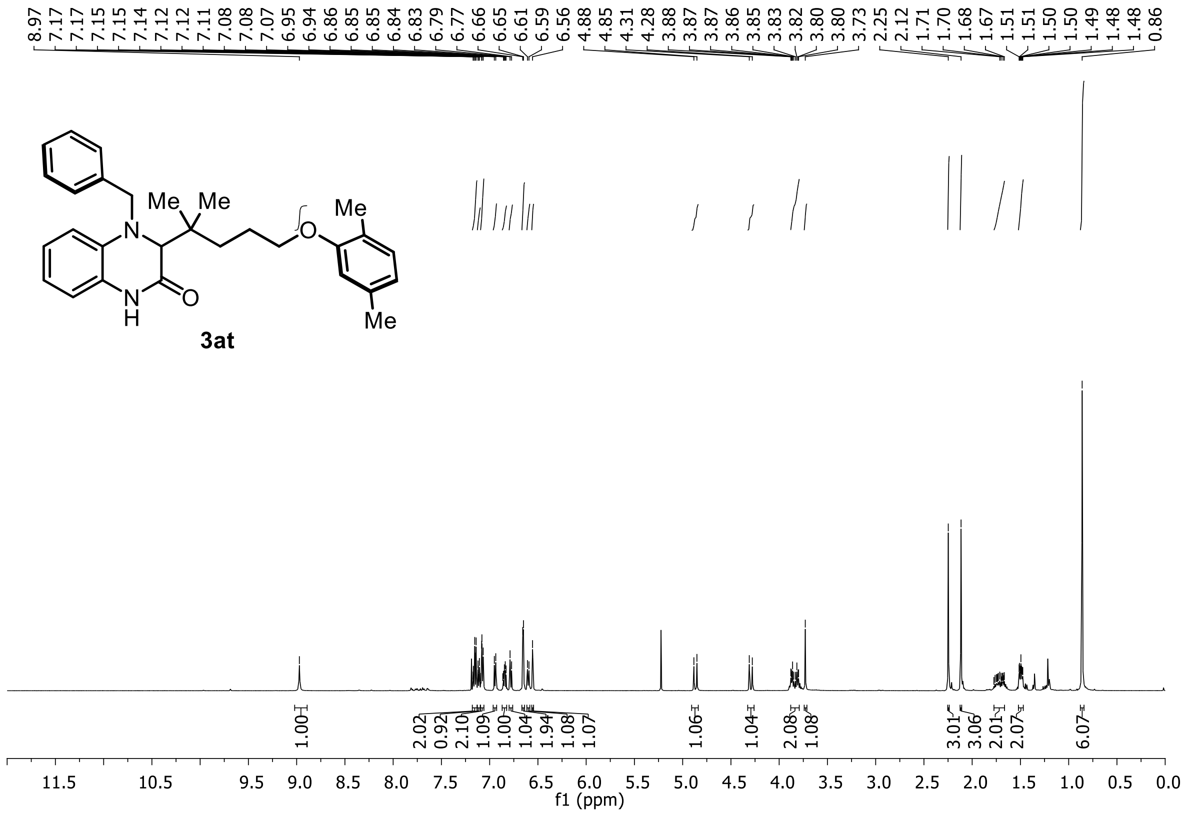
^{1}H NMR spectrum of 3as (CDCl$_3$, 500 MHz)



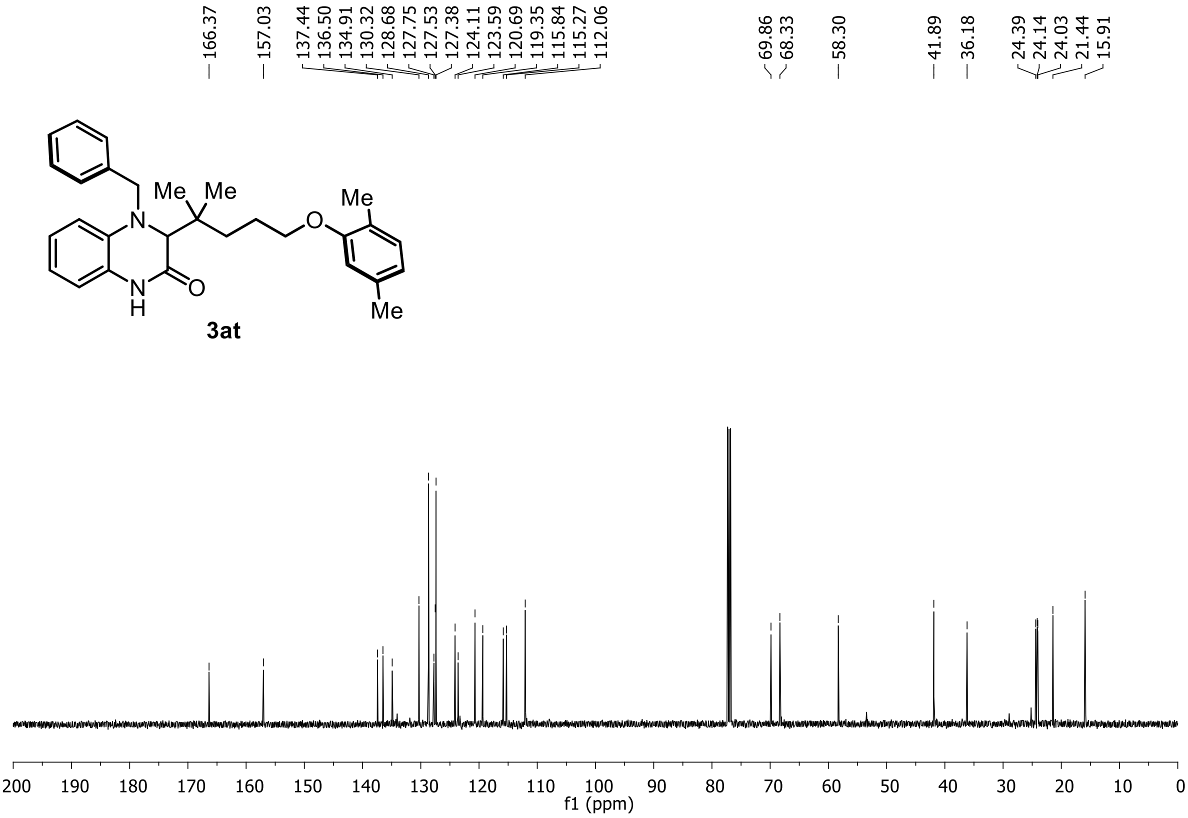
^{13}C{^{1}H} NMR spectrum of 3as (CDCl$_3$, 126 MHz)



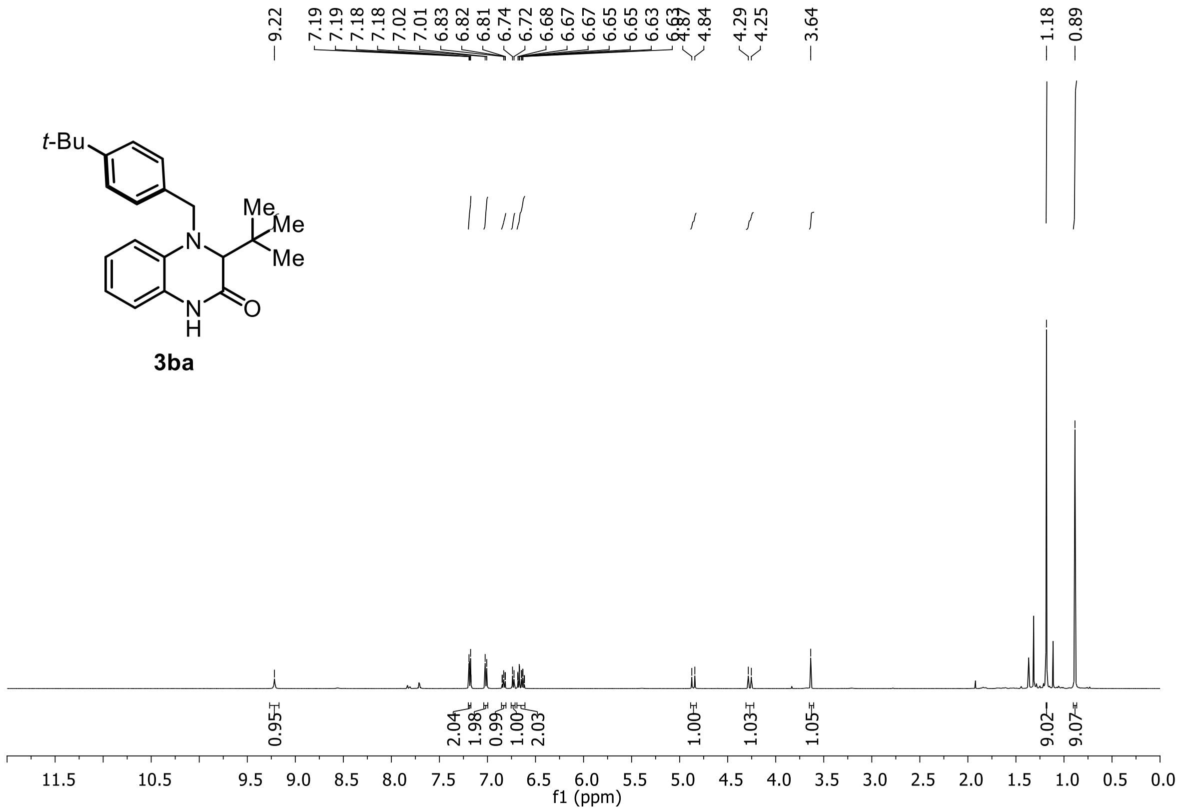
^{1}H NMR spectrum of 3at (CDCl$_3$, 500 MHz)



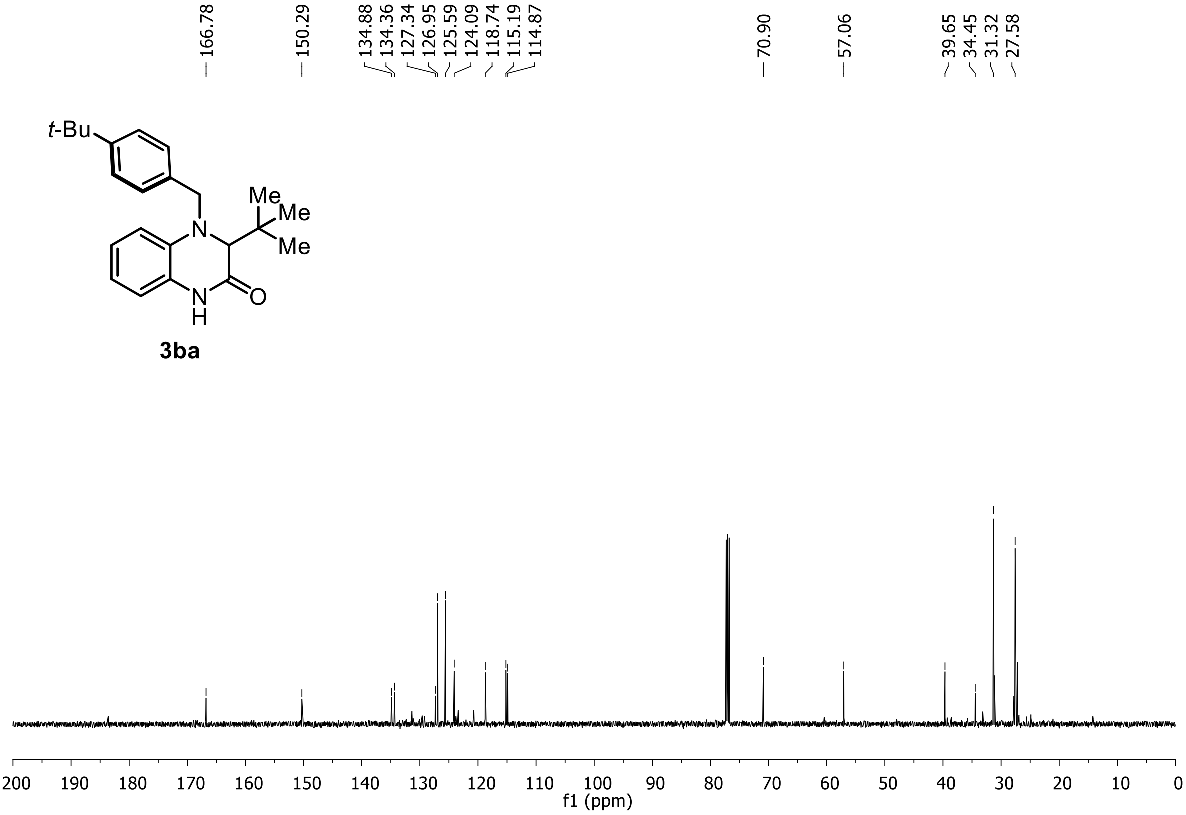
^{13}C{^{1}H} NMR spectrum of 3at (CDCl$_3$, 126 MHz)



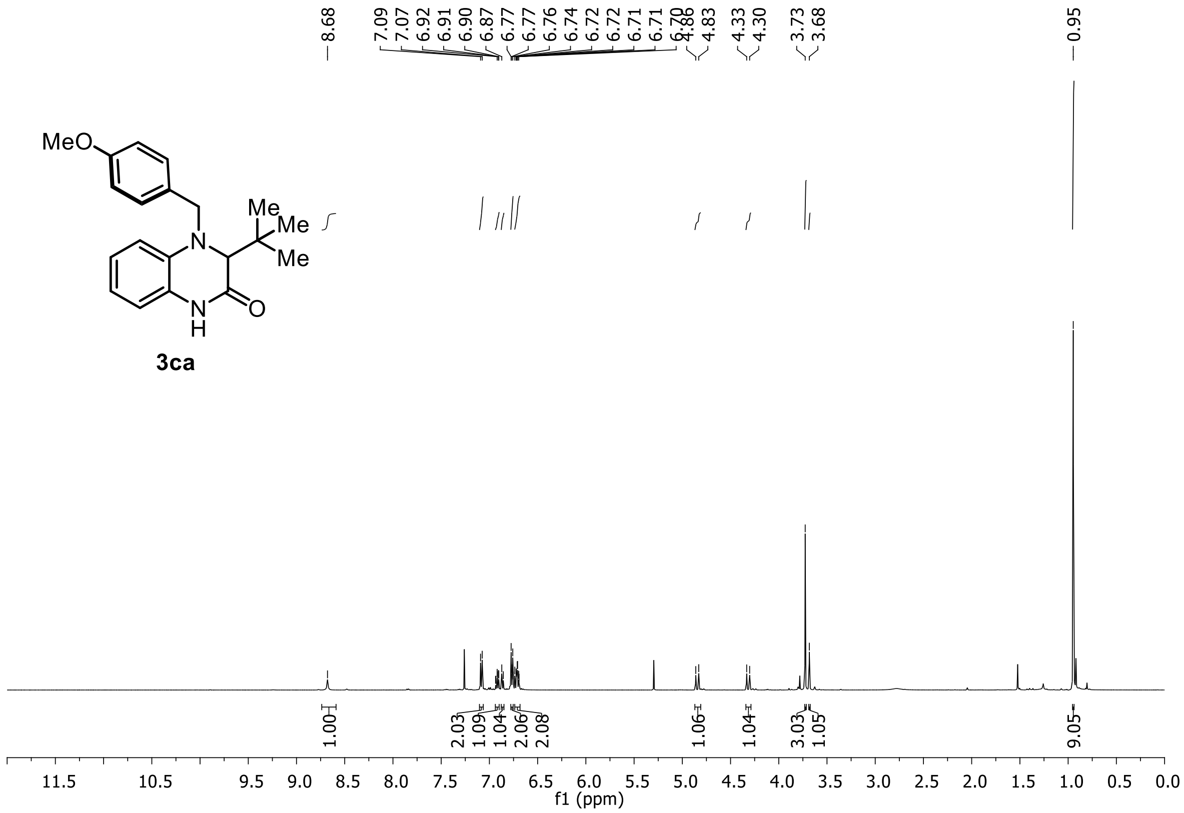
^{1}H NMR spectrum of 3ba (CDCl$_3$, 500 MHz)



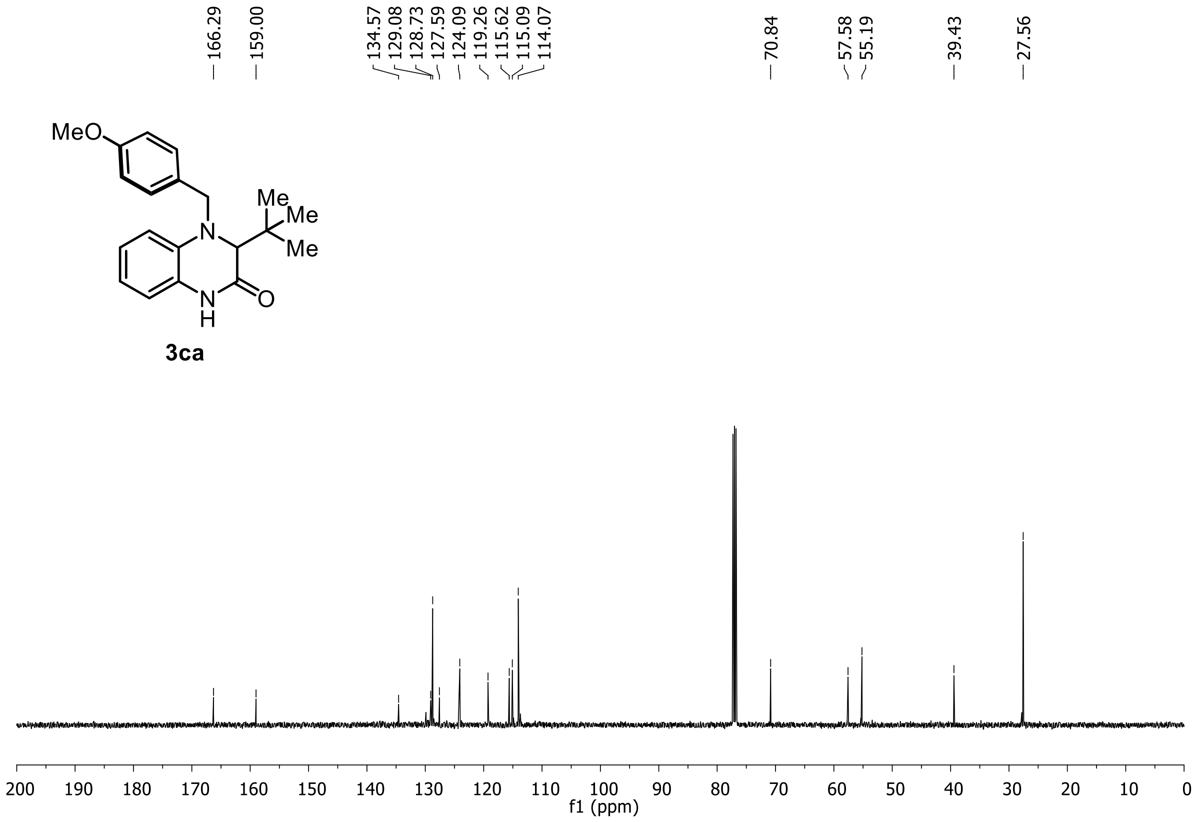
^{13}C{^{1}H} NMR spectrum of 3ba (CDCl$_3$, 126 MHz)



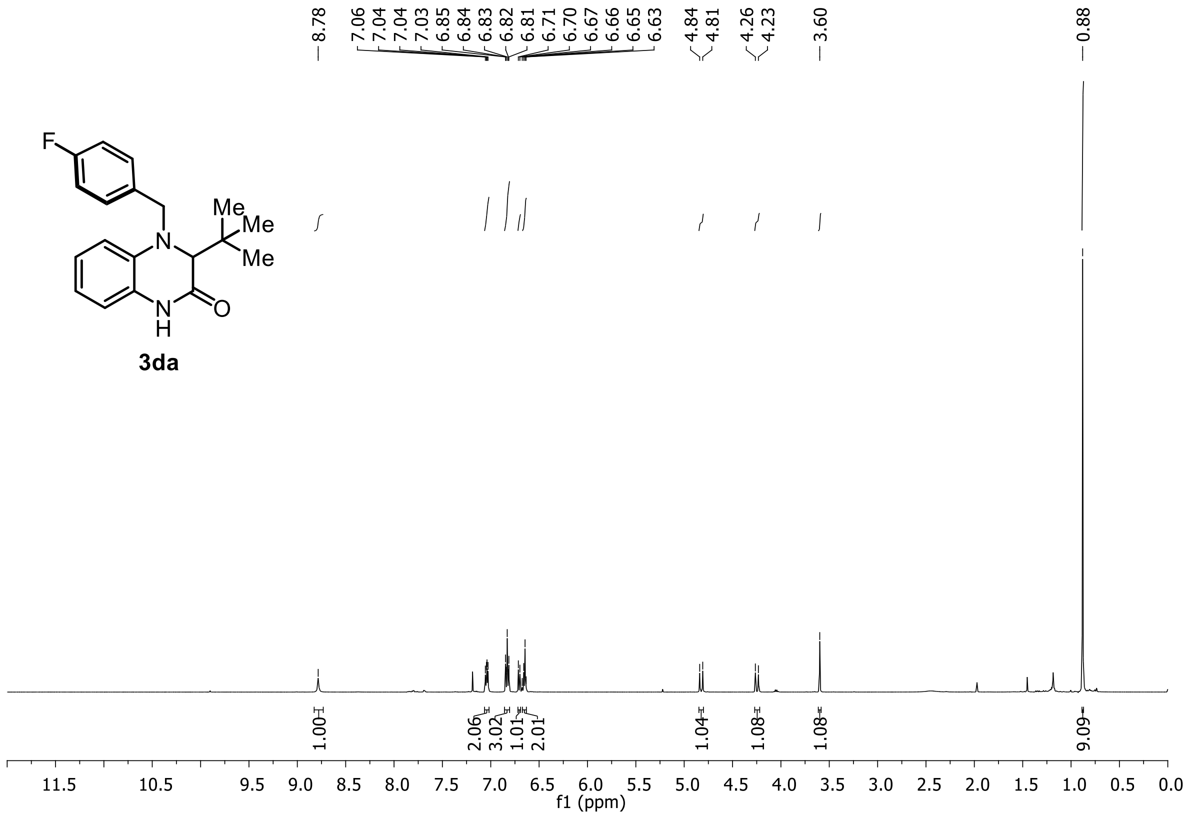
^{1}H NMR spectrum of 3ca ($CDCl_3$, 500 MHz)



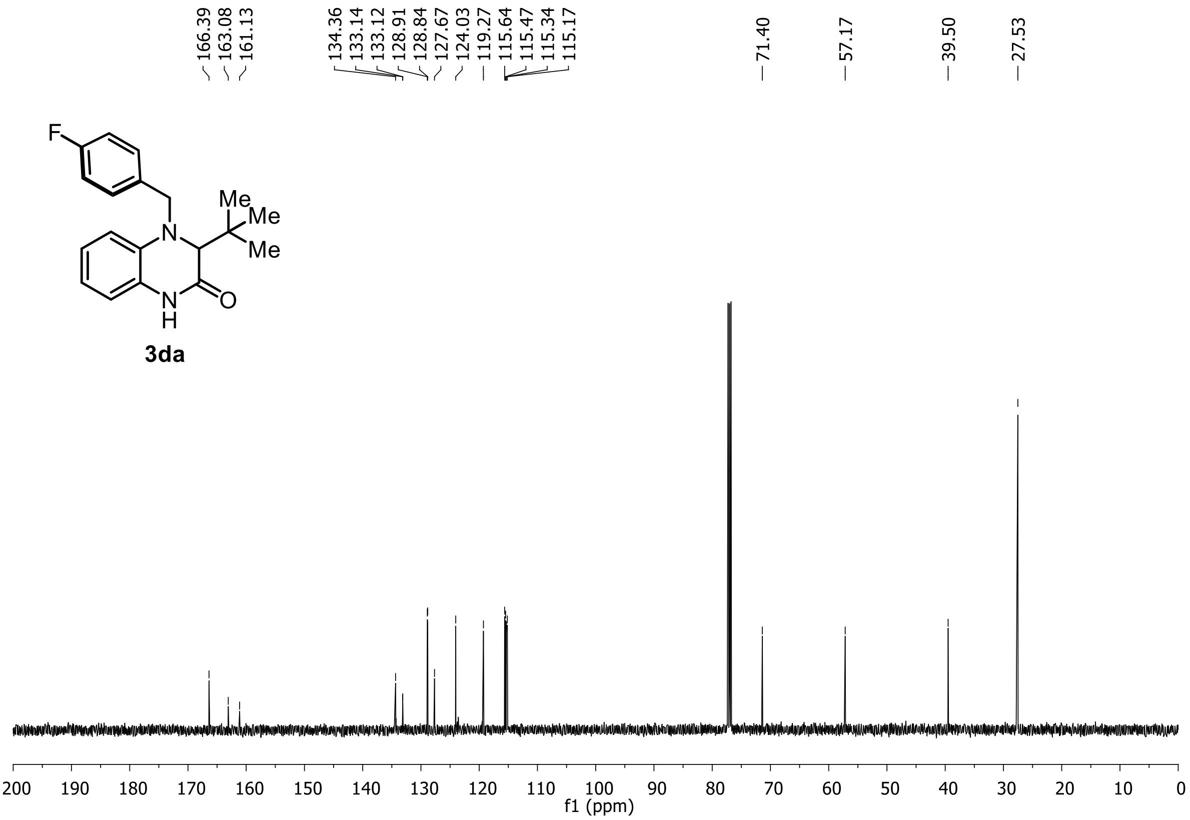
$^{13}C\{^{1}H\}$ NMR spectrum of 3ca ($CDCl_3$, 126 MHz)



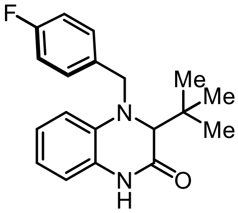
^{1}H NMR spectrum of 3da ($CDCl_3$, 500 MHz)



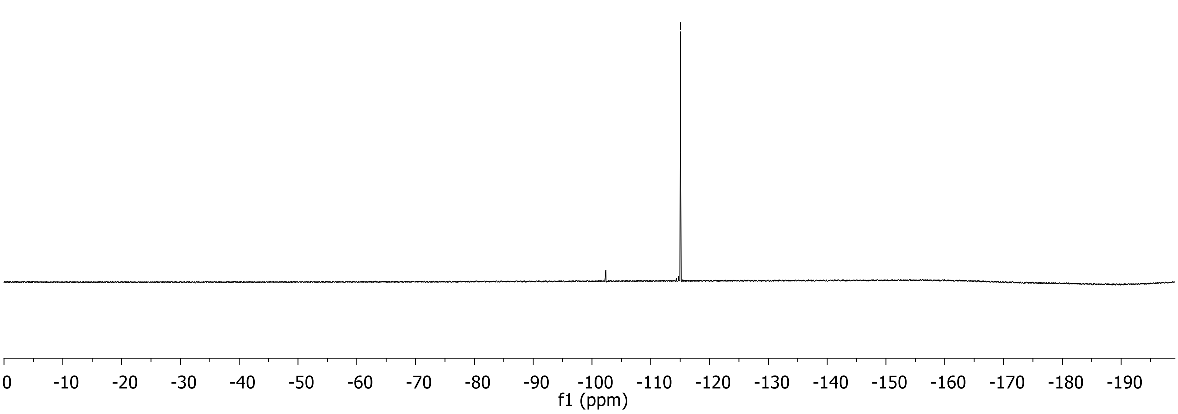
^{13}C{^{1}H} NMR spectrum of 3da ($CDCl_3$, 126 MHz)



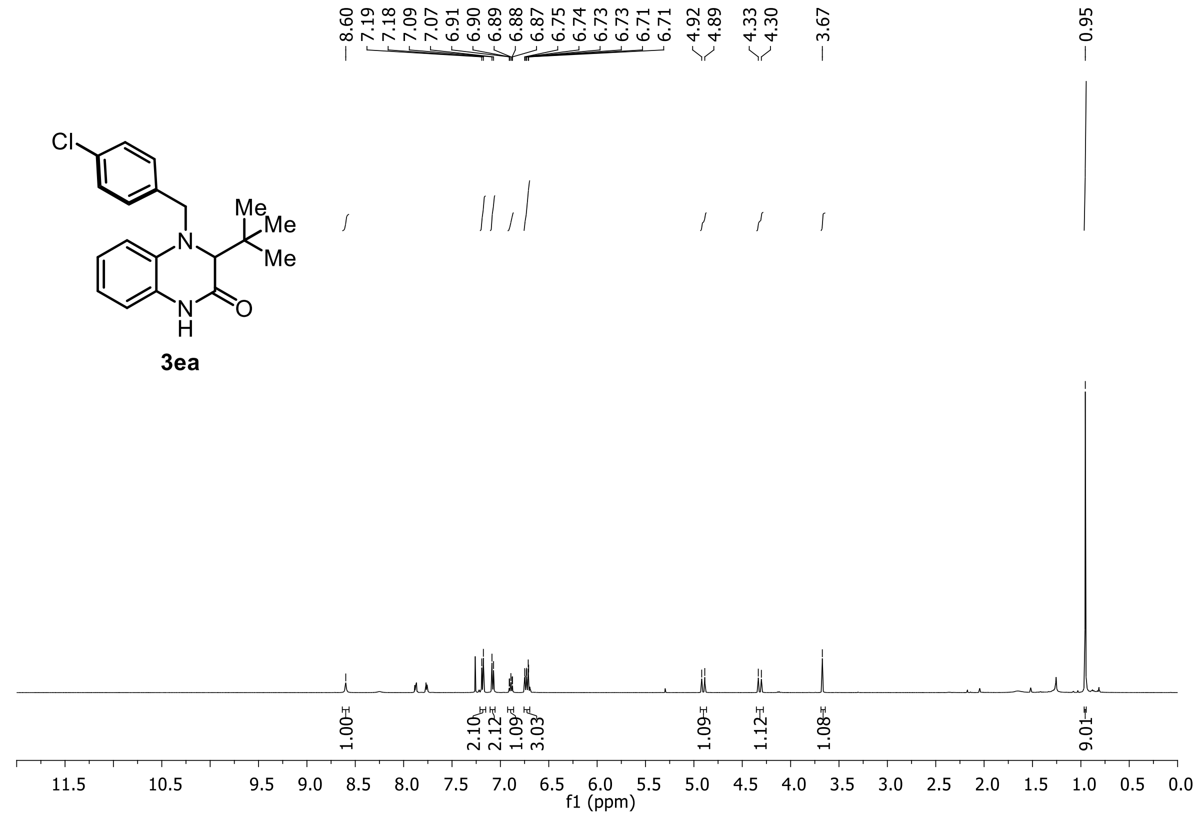
$^{19}F\{^1H\}$ NMR spectrum of 3da ($CDCl_3$, 471 MHz)



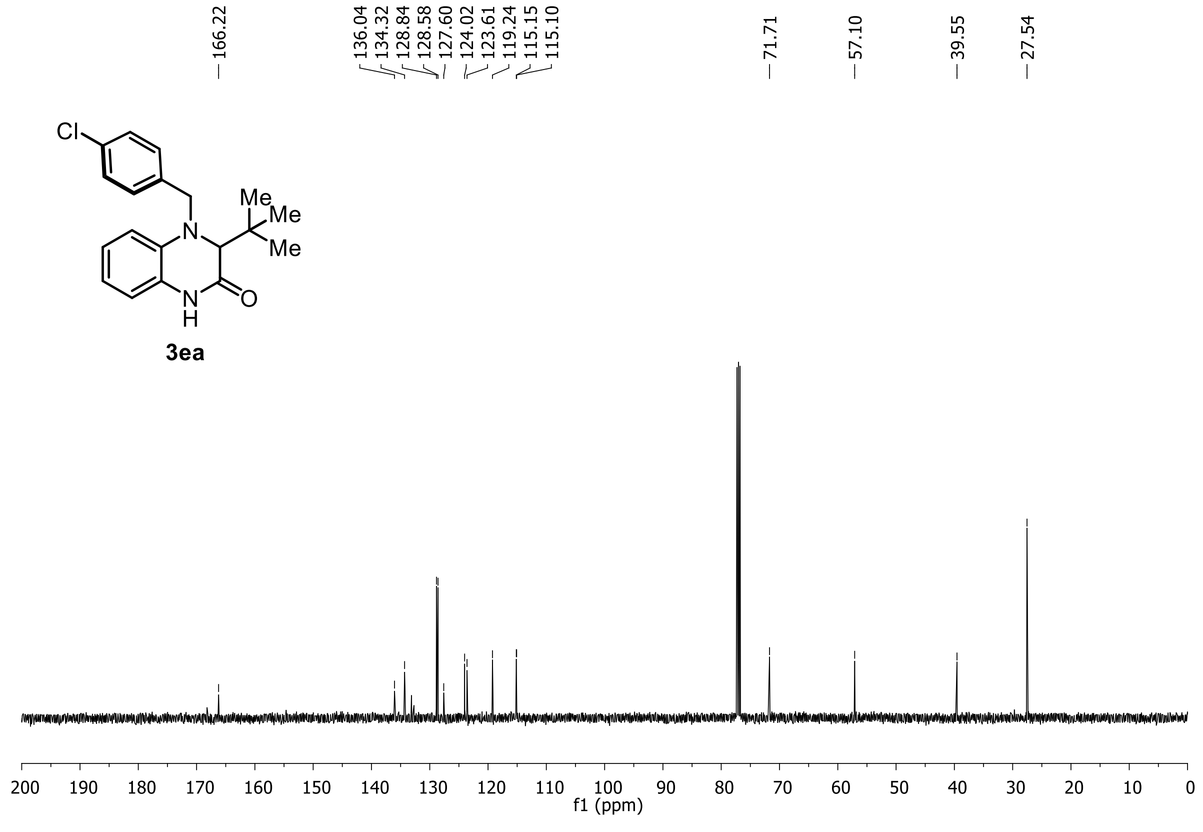
3da



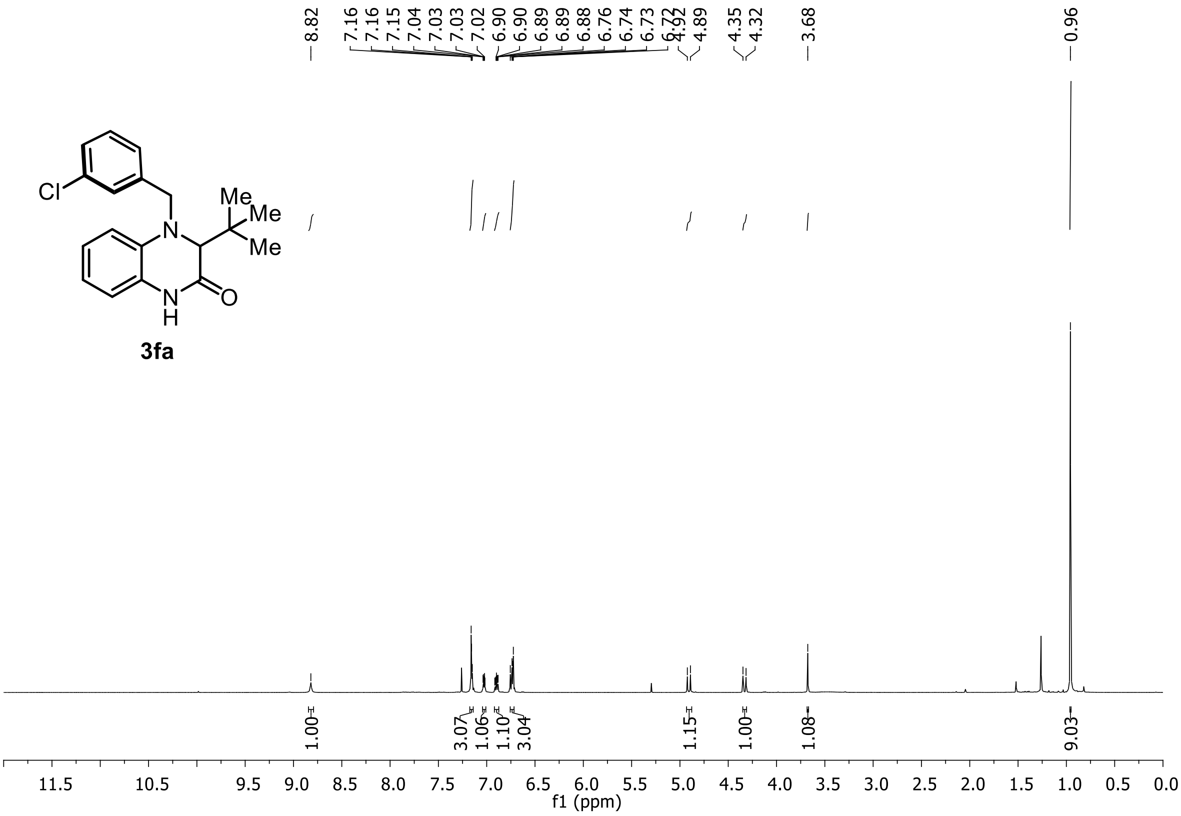
^{1}H NMR spectrum of 3ea ($CDCl_3$, 500 MHz)



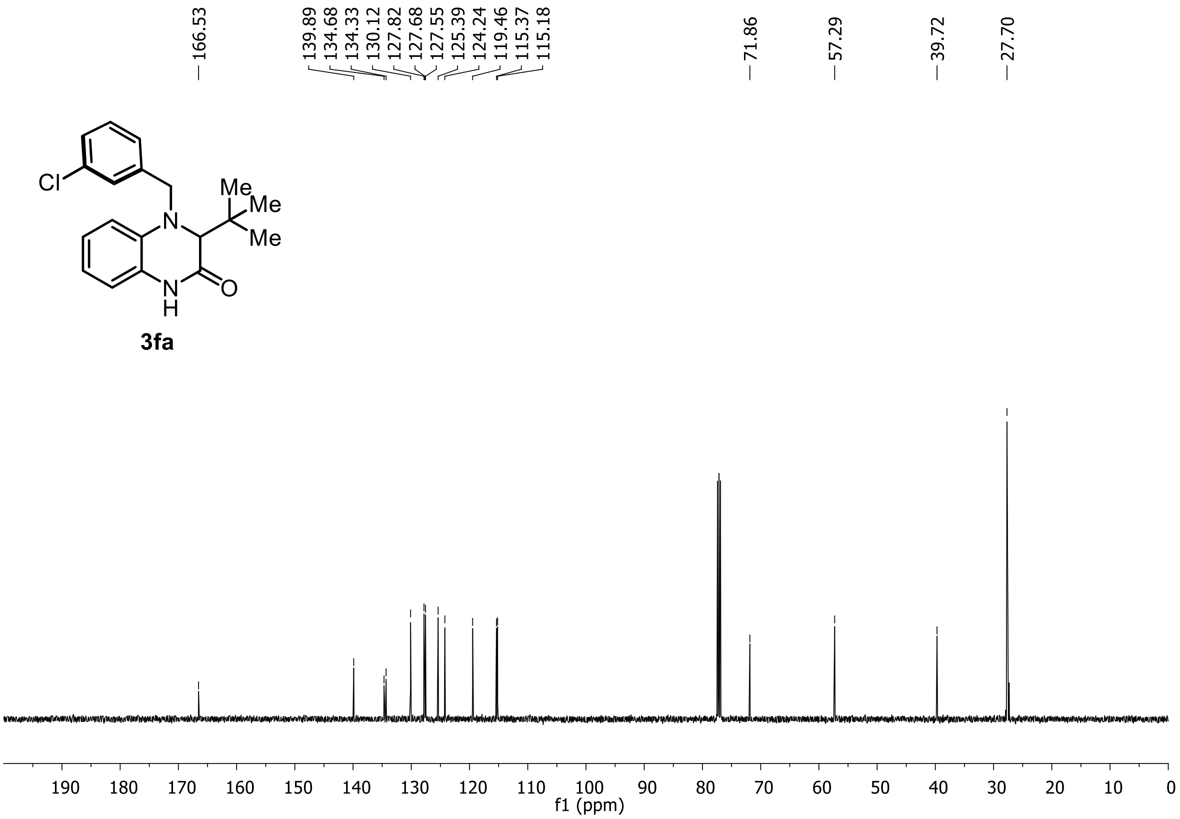
^{13}C{^{1}H} NMR spectrum of 3ea ($CDCl_3$, 126 MHz)



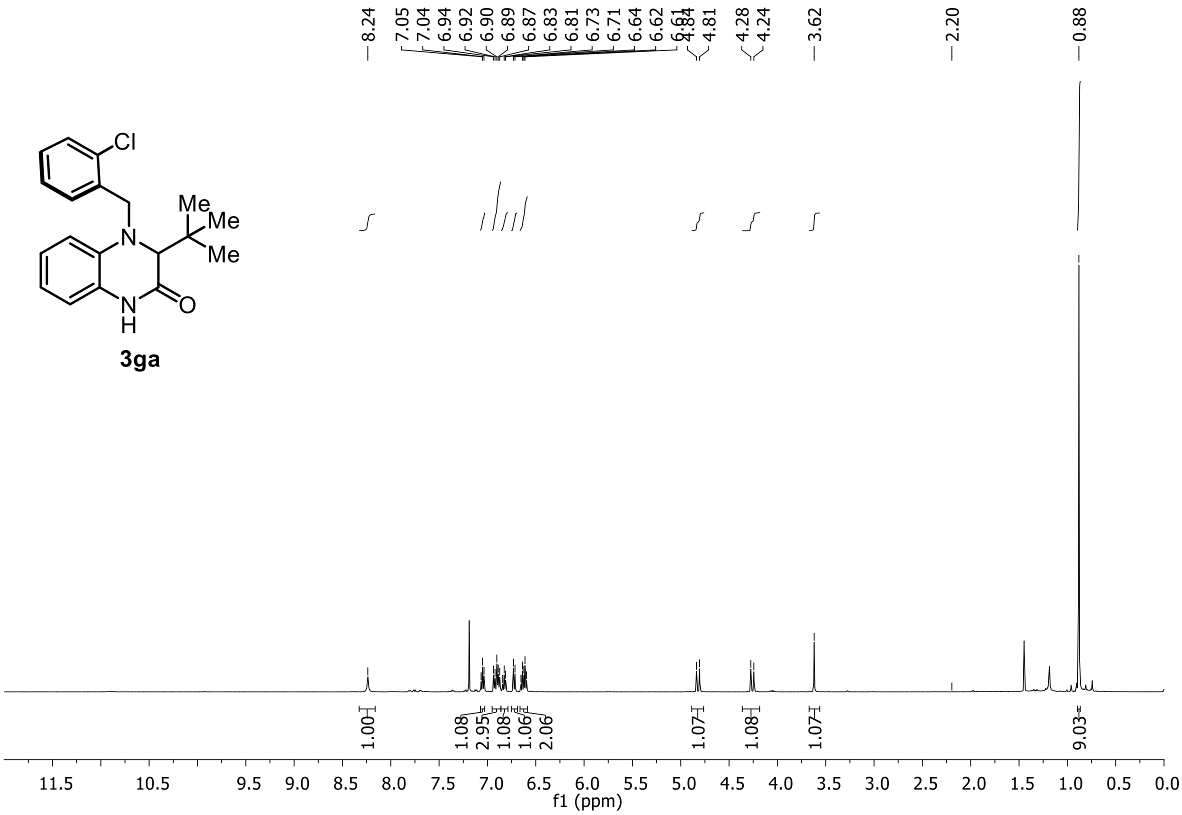
^{1}H NMR spectrum of 3fa (CDCl$_3$, 500 MHz)



^{13}C{^{1}H} NMR spectrum of 3fa (CDCl$_3$, 126 MHz)

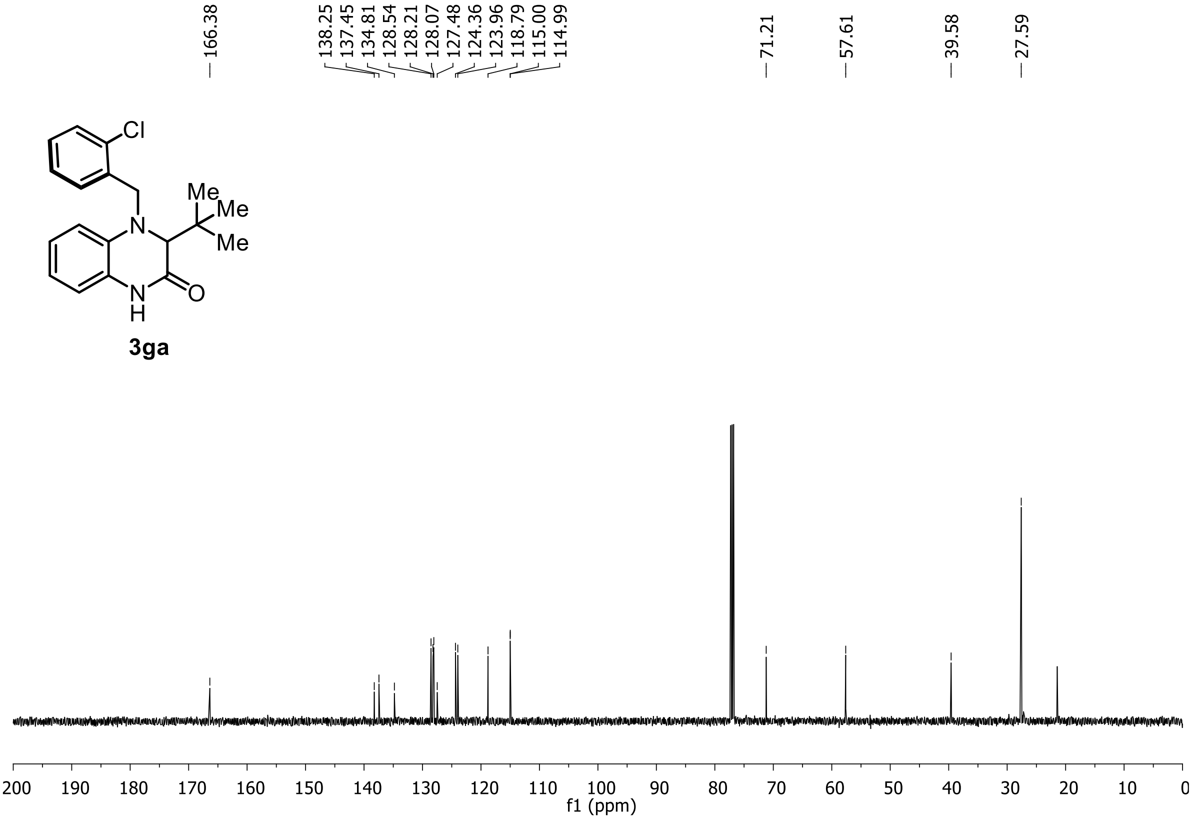


^{1}H NMR spectrum of 3ga ($CDCl_3$, 500 MHz)



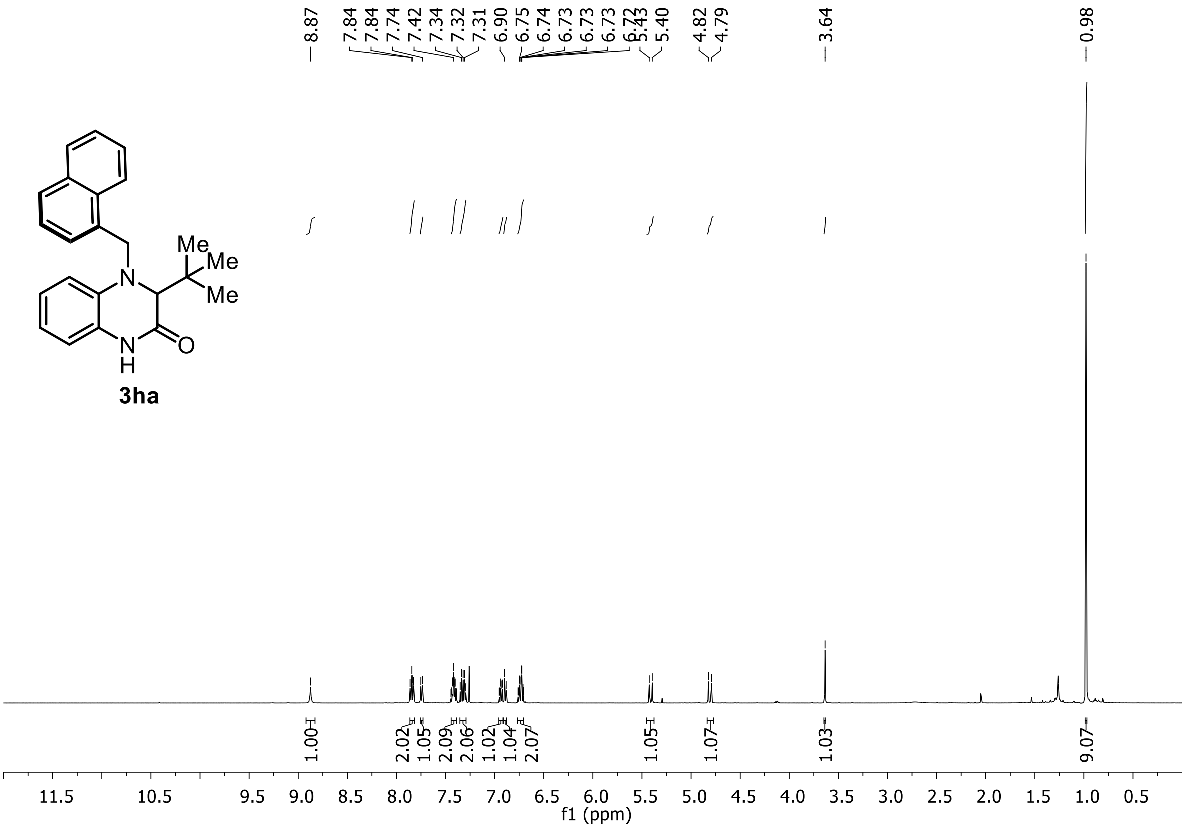
3ga

^{13}C{^{1}H} NMR spectrum of 3ga ($CDCl_3$, 126 MHz)

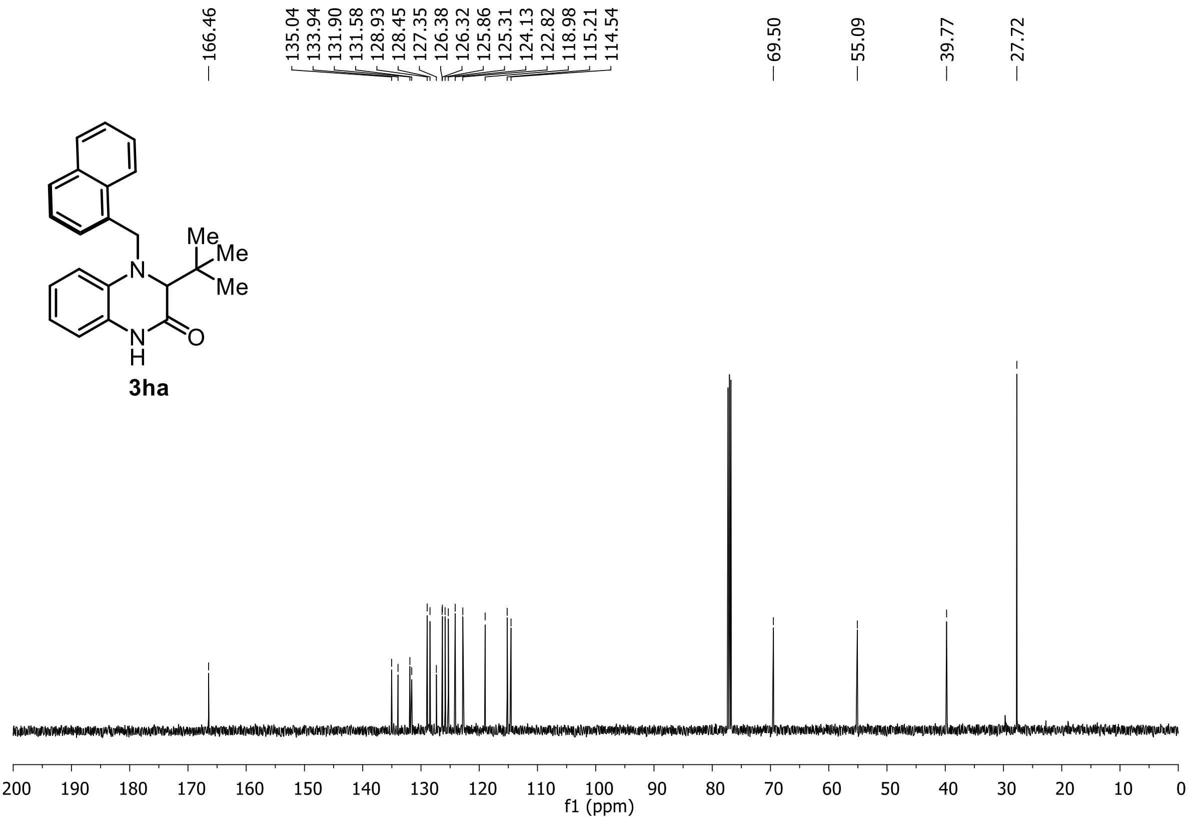


3ga

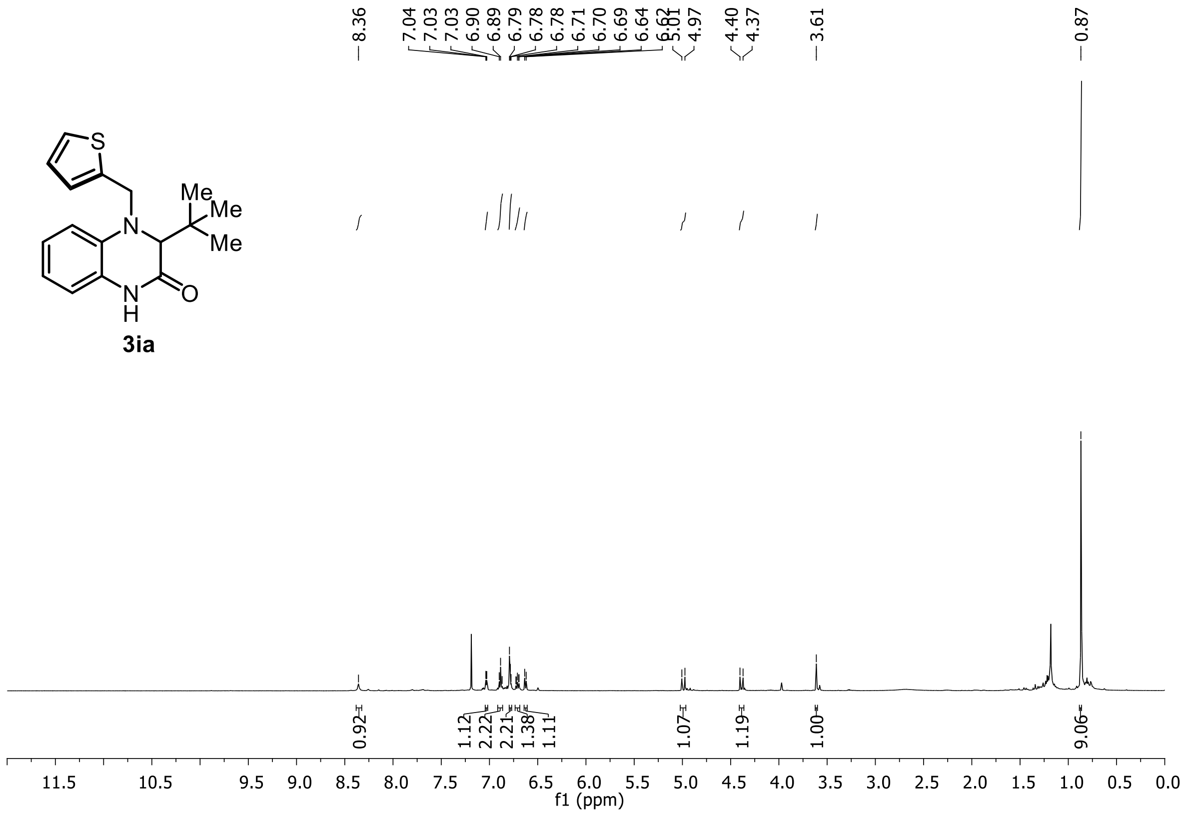
^{1}H NMR spectrum of 3ha ($CDCl_3$, 500 MHz)



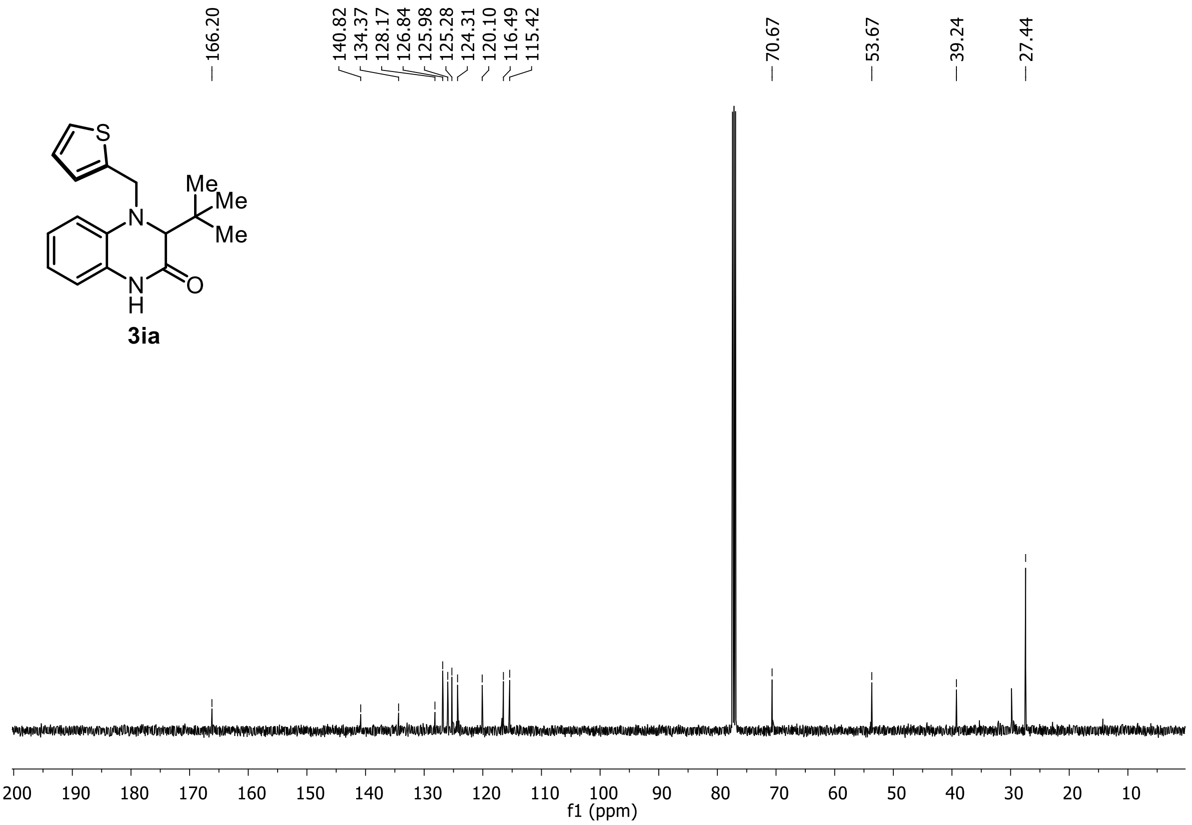
^{13}C{^{1}H} NMR spectrum of 3ha ($CDCl_3$, 126 MHz)



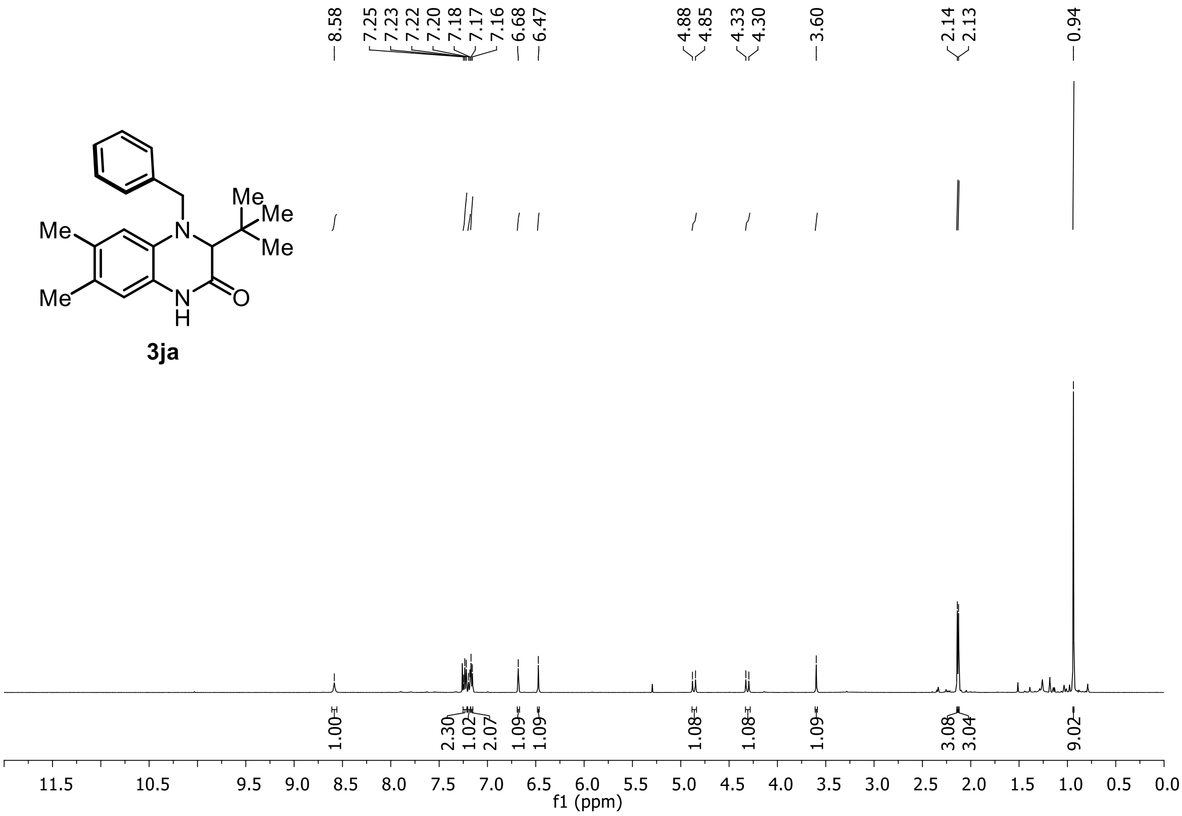
^{1}H NMR spectrum of 3ia (CDCl$_3$, 500 MHz)



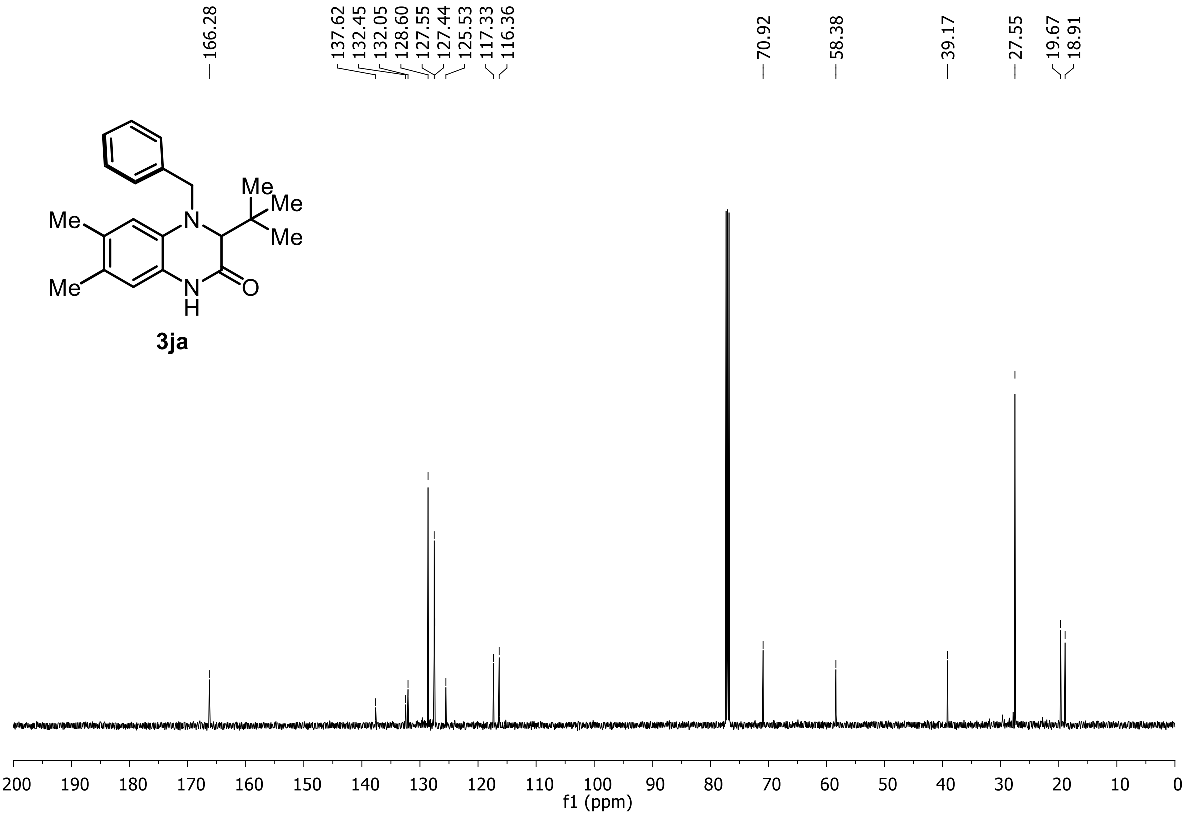
^{13}C{^{1}H} NMR spectrum of 3ia (CDCl$_3$, 126 MHz)



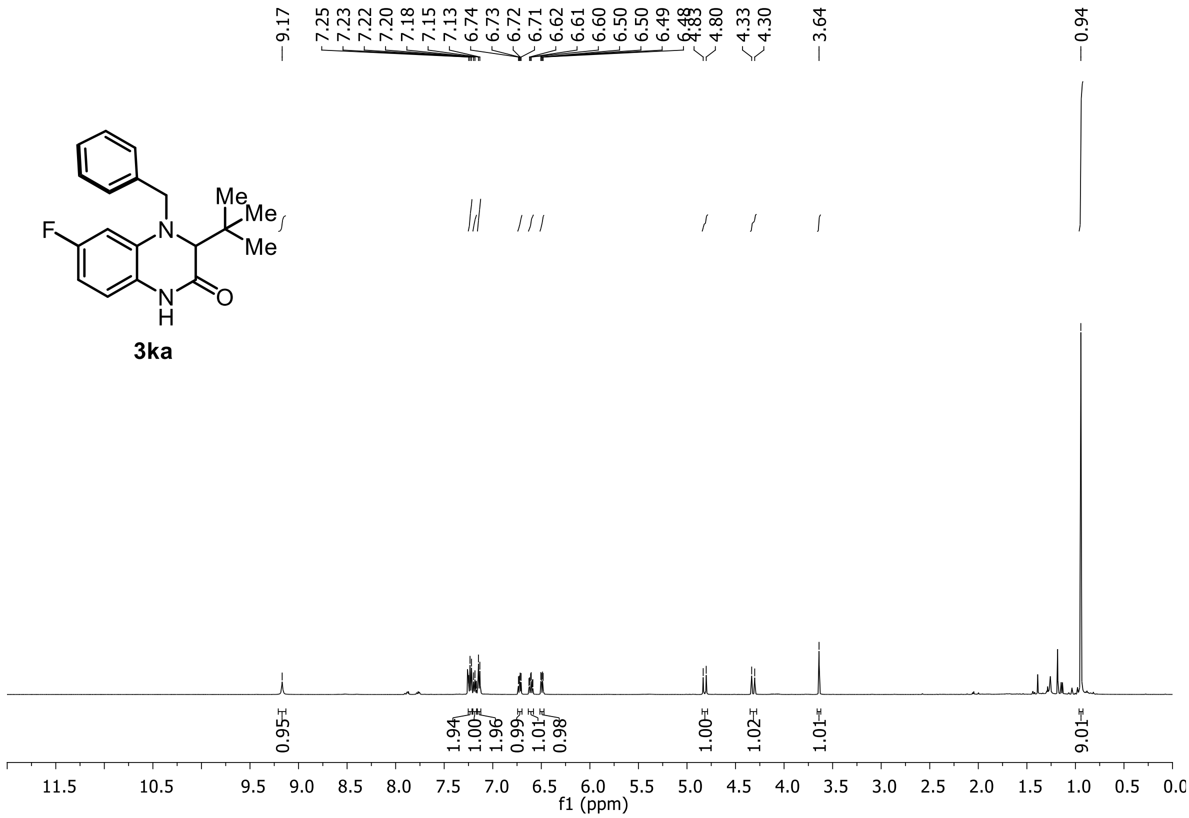
^{1}H NMR spectrum of 3ja (CDCl$_3$, 500 MHz)



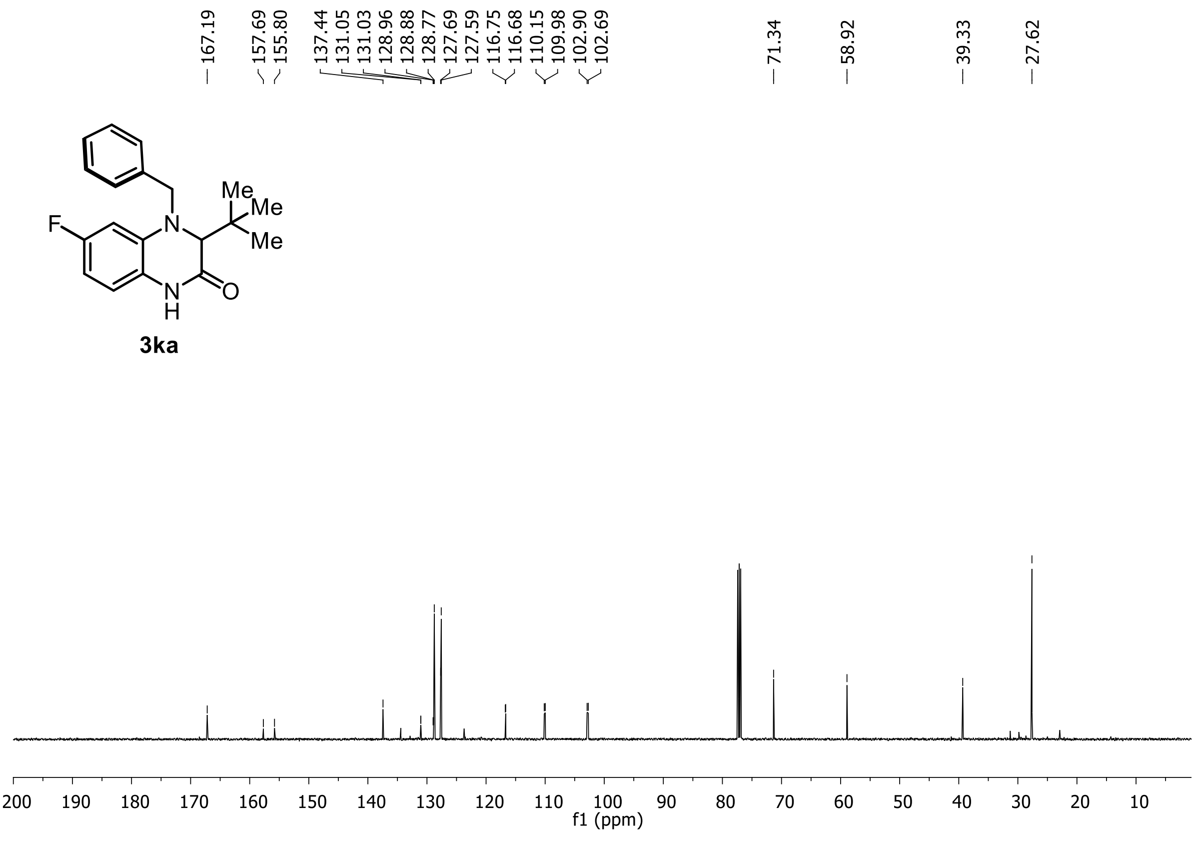
^{13}C{^{1}H} NMR spectrum of 3ja (CDCl$_3$, 126 MHz)



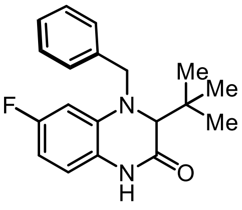
^{1}H NMR spectrum of 3ka ($CDCl_3$, 500 MHz)



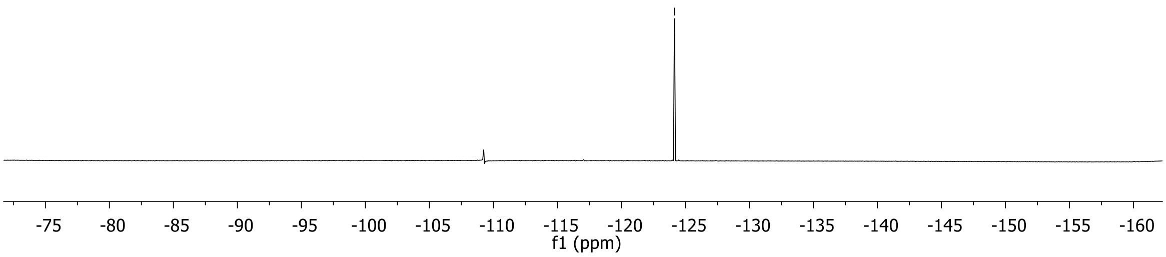
^{13}C{^{1}H} NMR spectrum of 3ka ($CDCl_3$, 126 MHz)



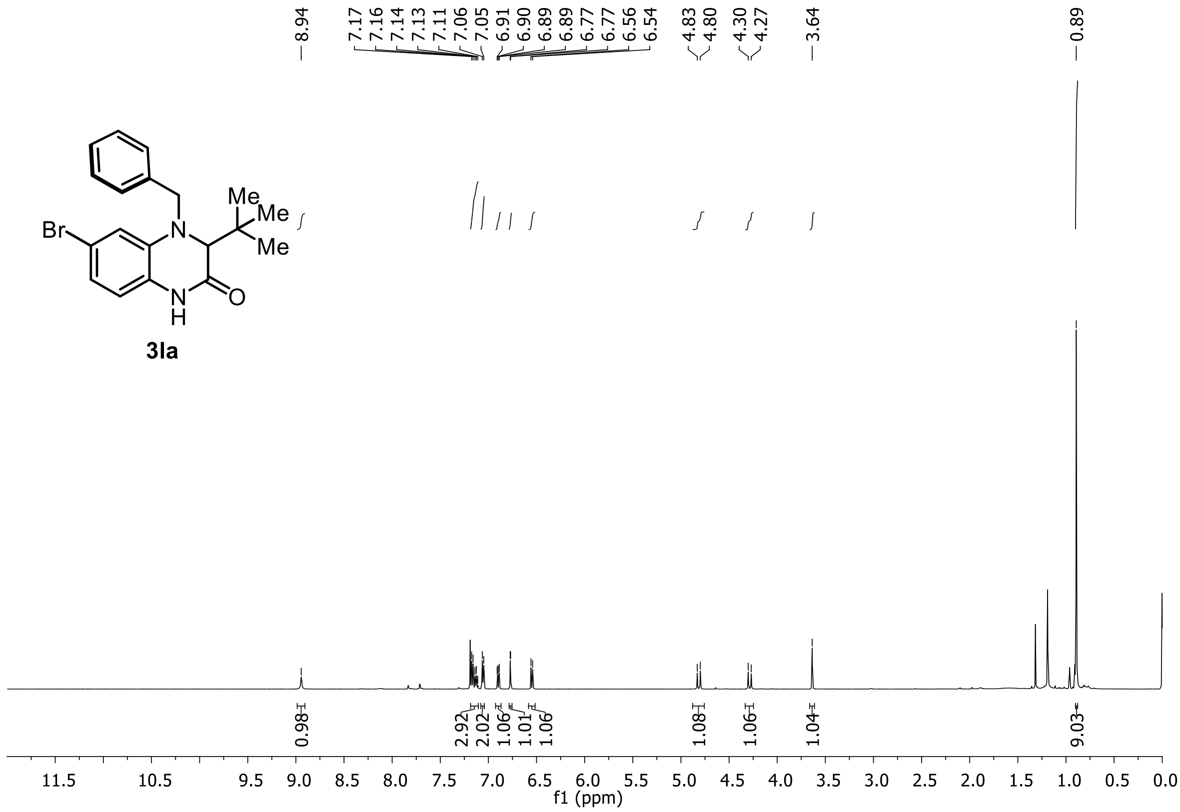
$^{19}F\{^1H\}$ NMR spectrum of 3ka ($CDCl_3$, 471 MHz)



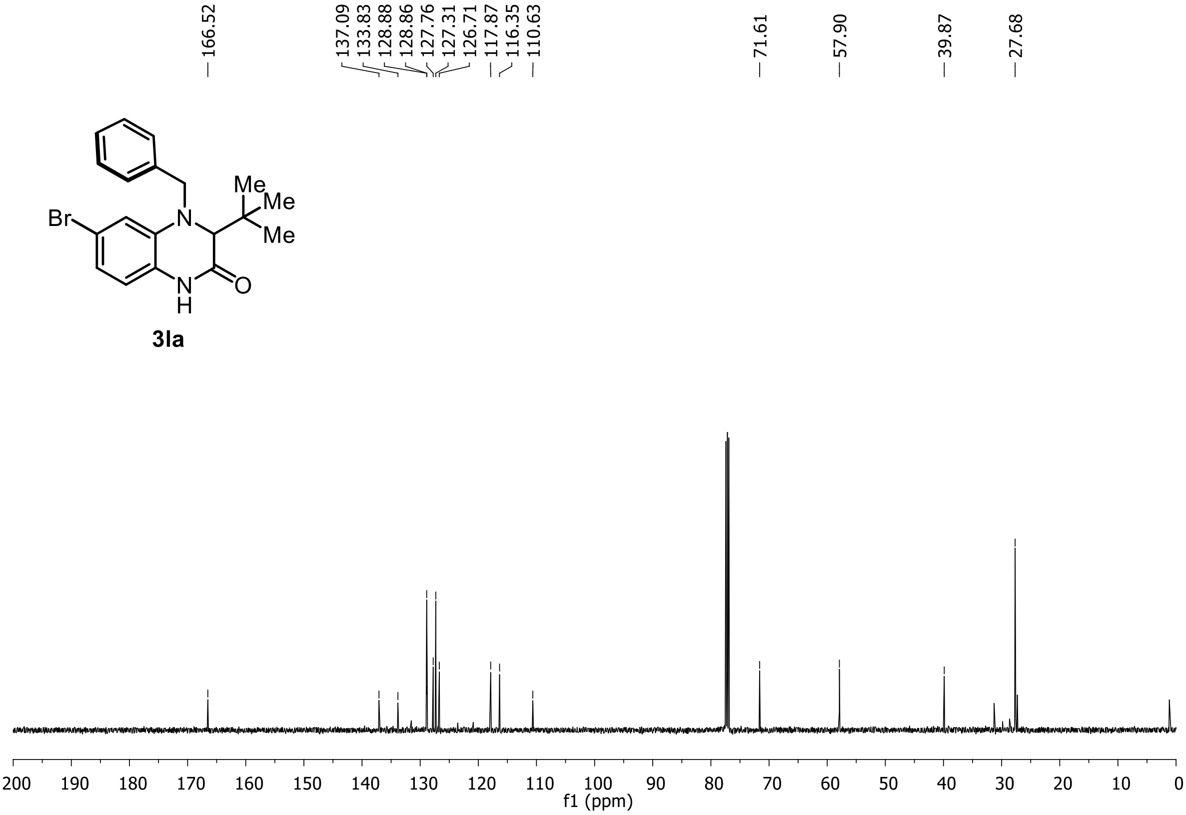
3ka



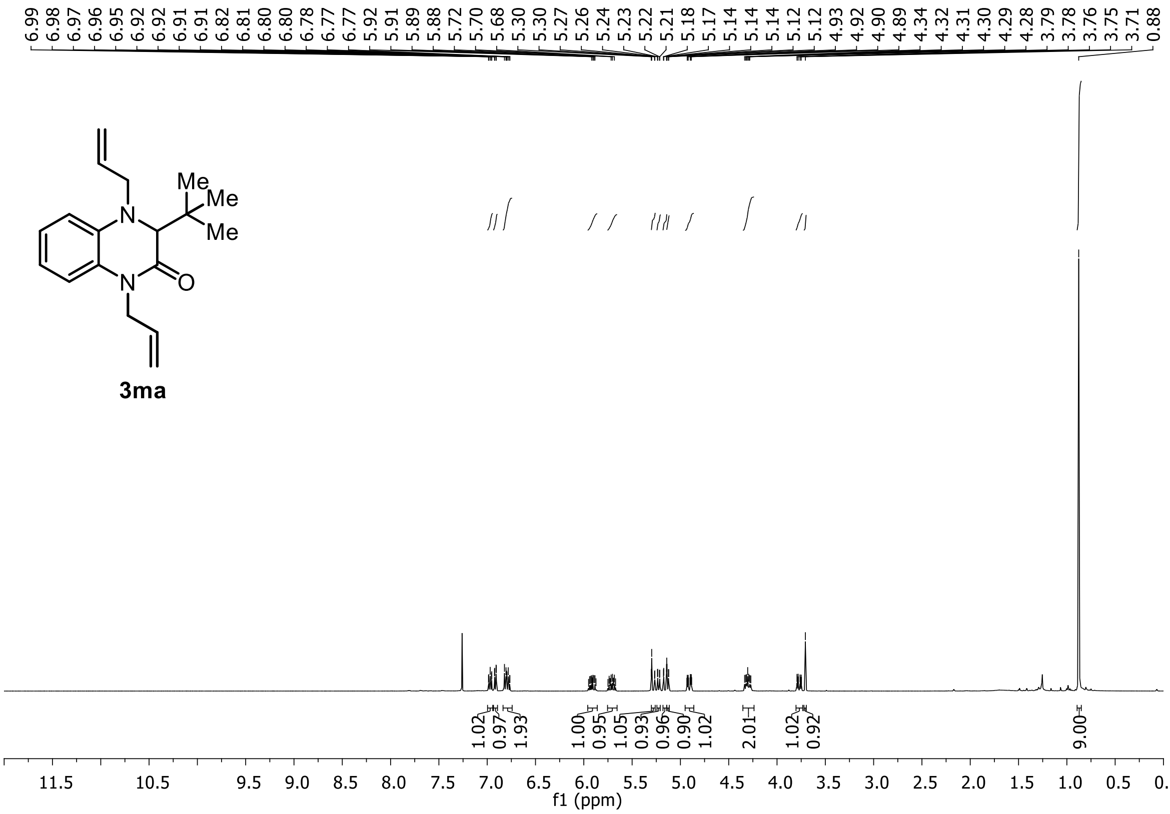
^{1}H NMR spectrum of 3la (CDCl$_3$, 500 MHz)



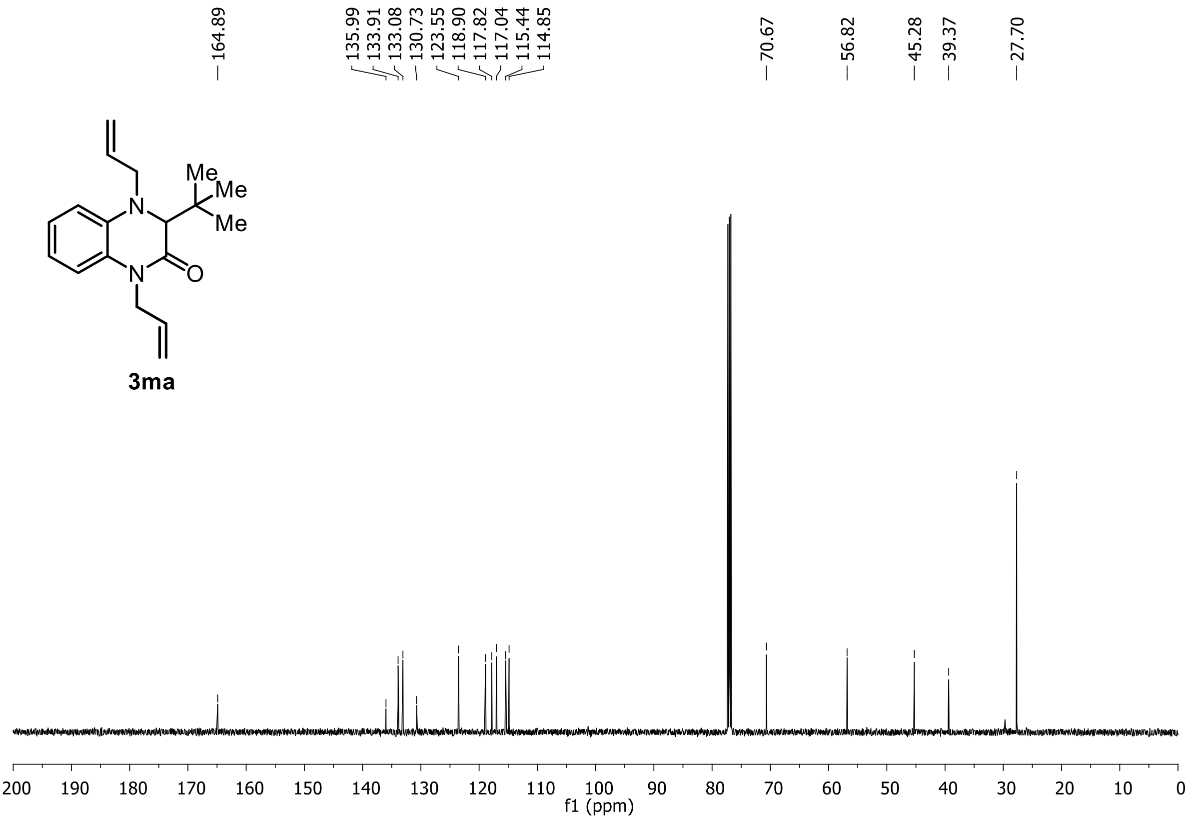
^{13}C{^{1}H} NMR spectrum of 3la (CDCl$_3$, 126 MHz)



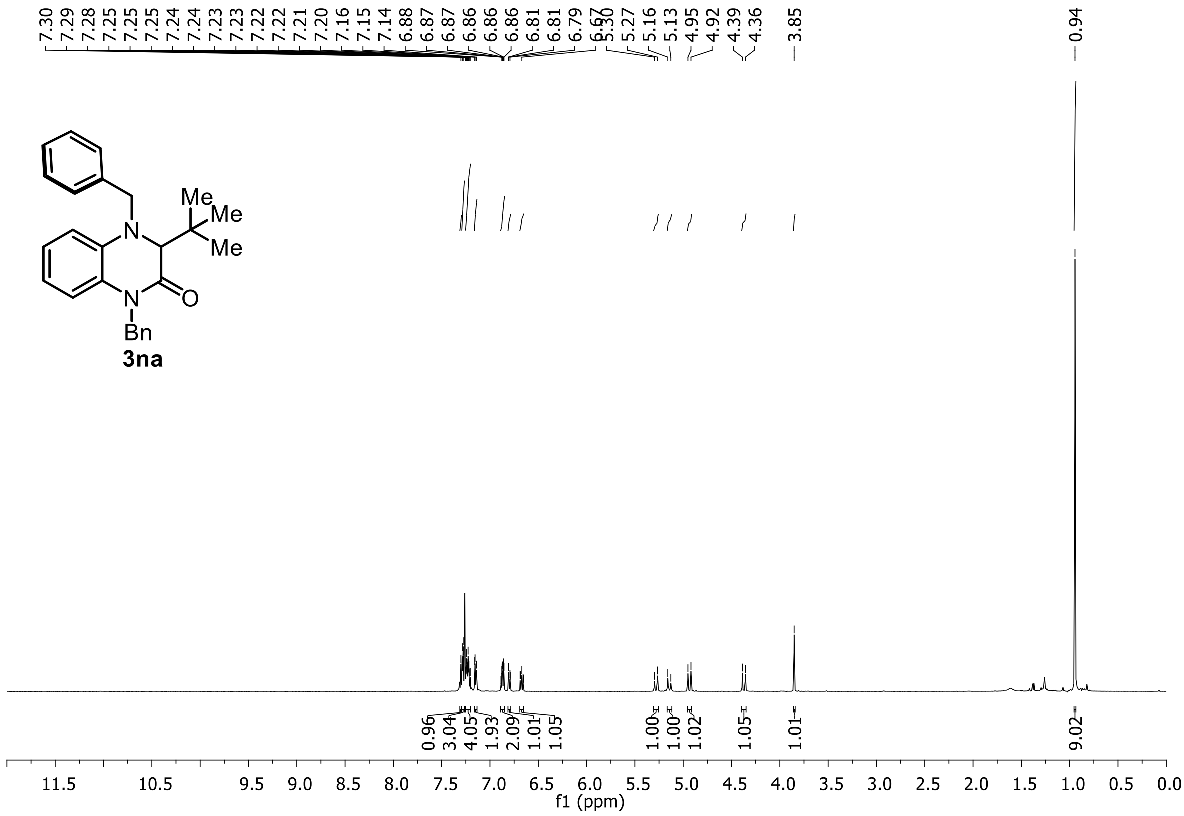
^{1}H NMR spectrum of 3ma (CDCl$_3$, 500 MHz)



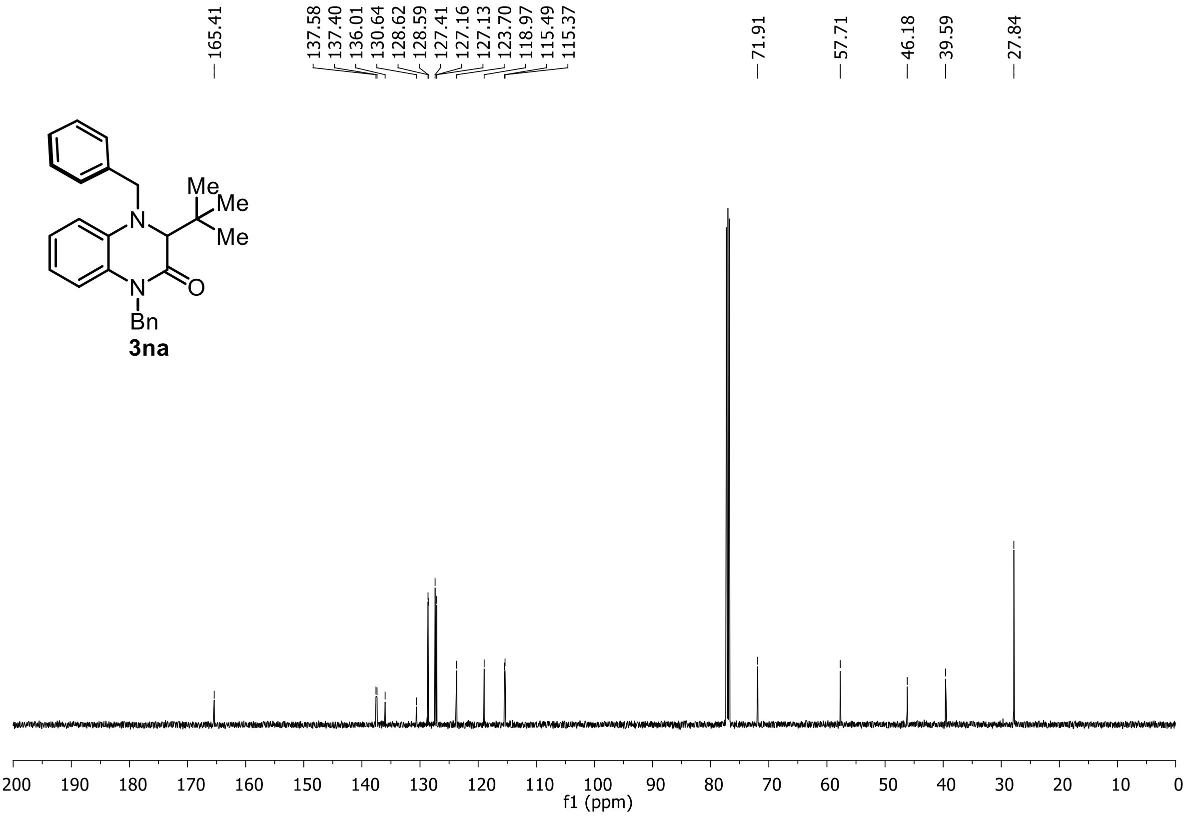
^{13}C{^{1}H} NMR spectrum of 3ma (CDCl$_3$, 126 MHz)



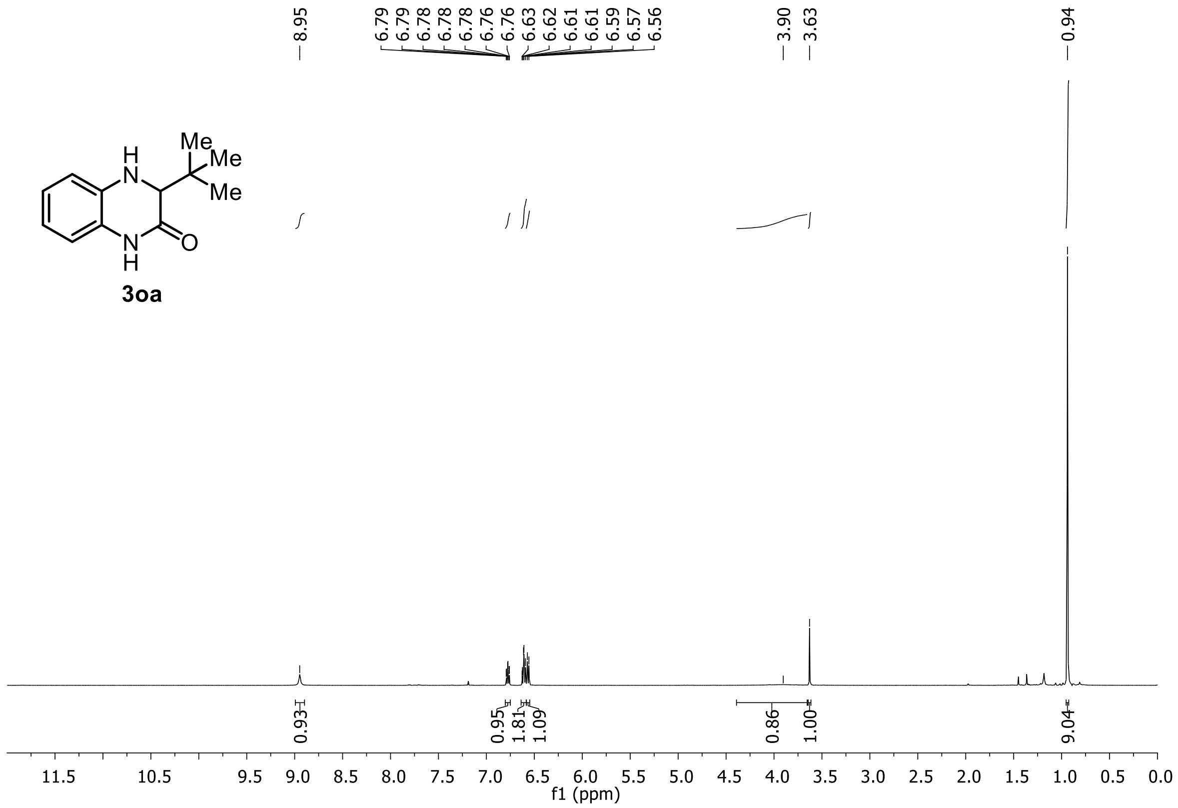
^{1}H NMR spectrum of 3na (CDCl$_3$, 500 MHz)



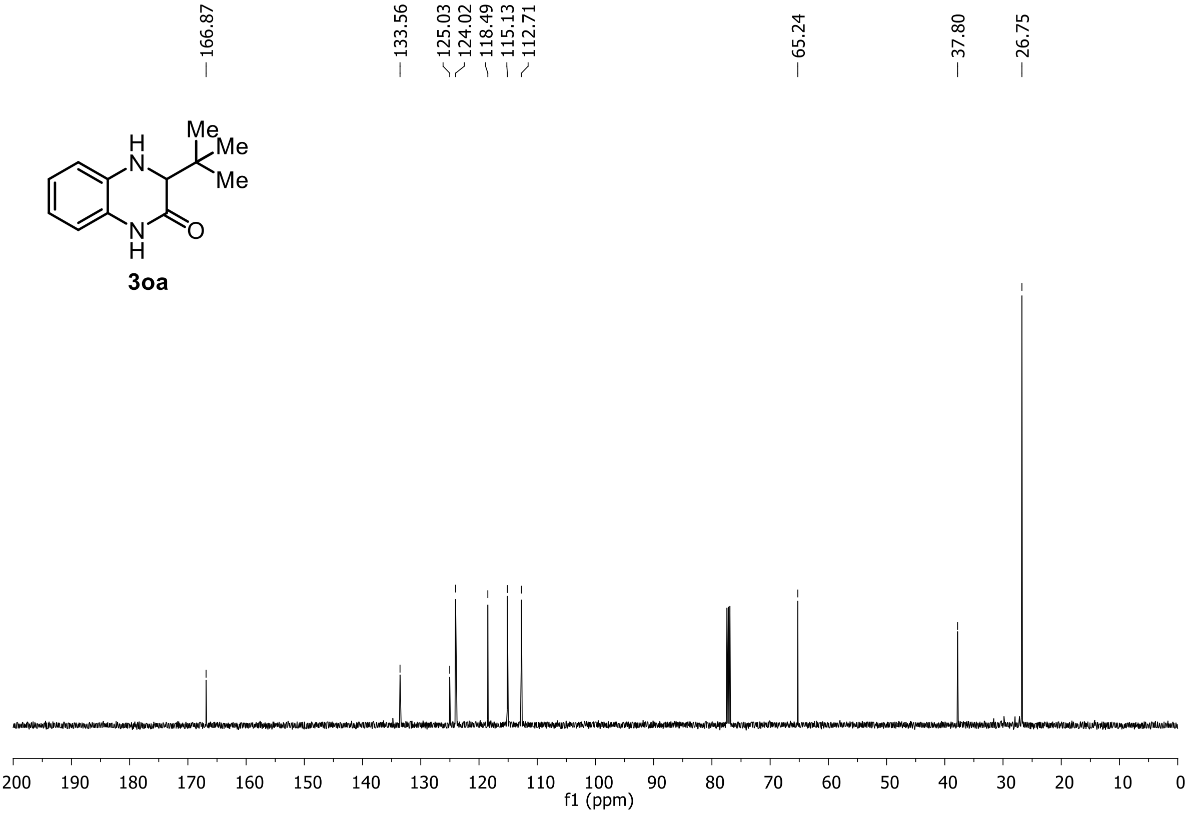
^{13}C{^{1}H} NMR spectrum of 3na (CDCl$_3$, 126 MHz)



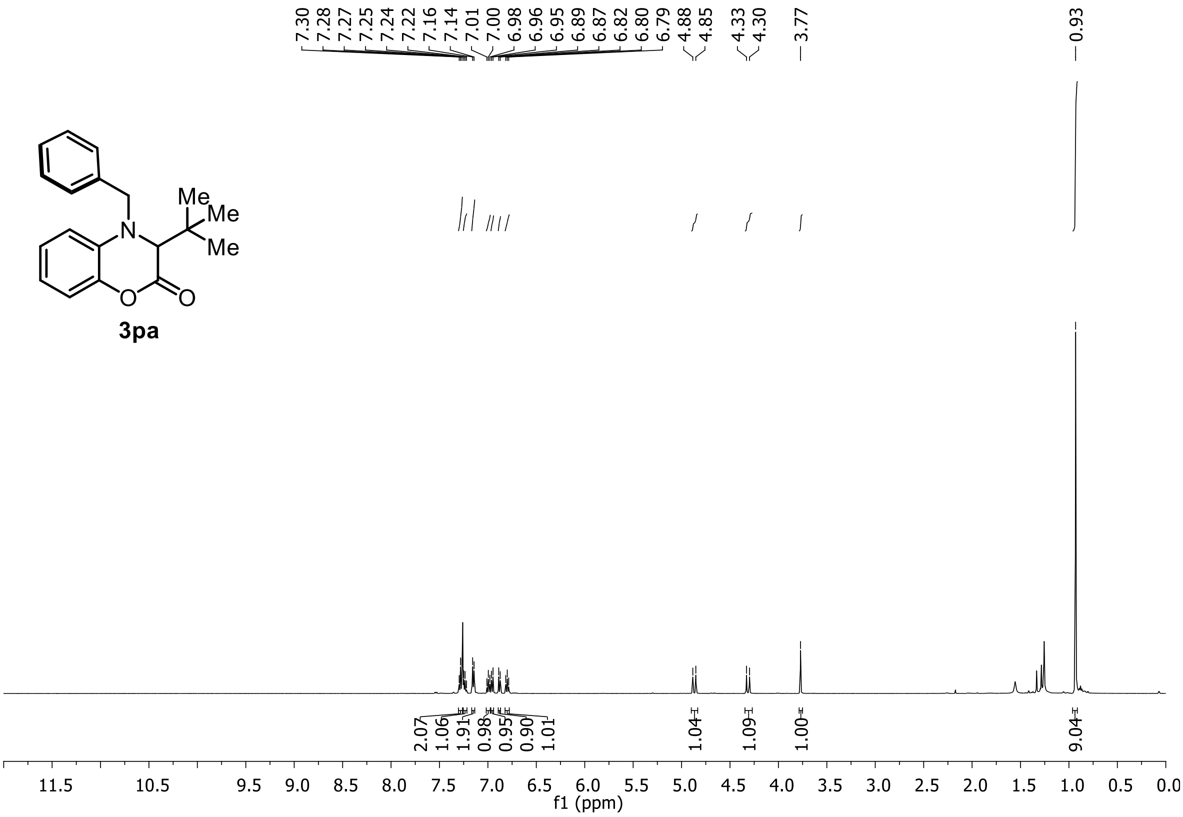
^{1}H NMR spectrum of 3oa ($CDCl_3$, 500 MHz)



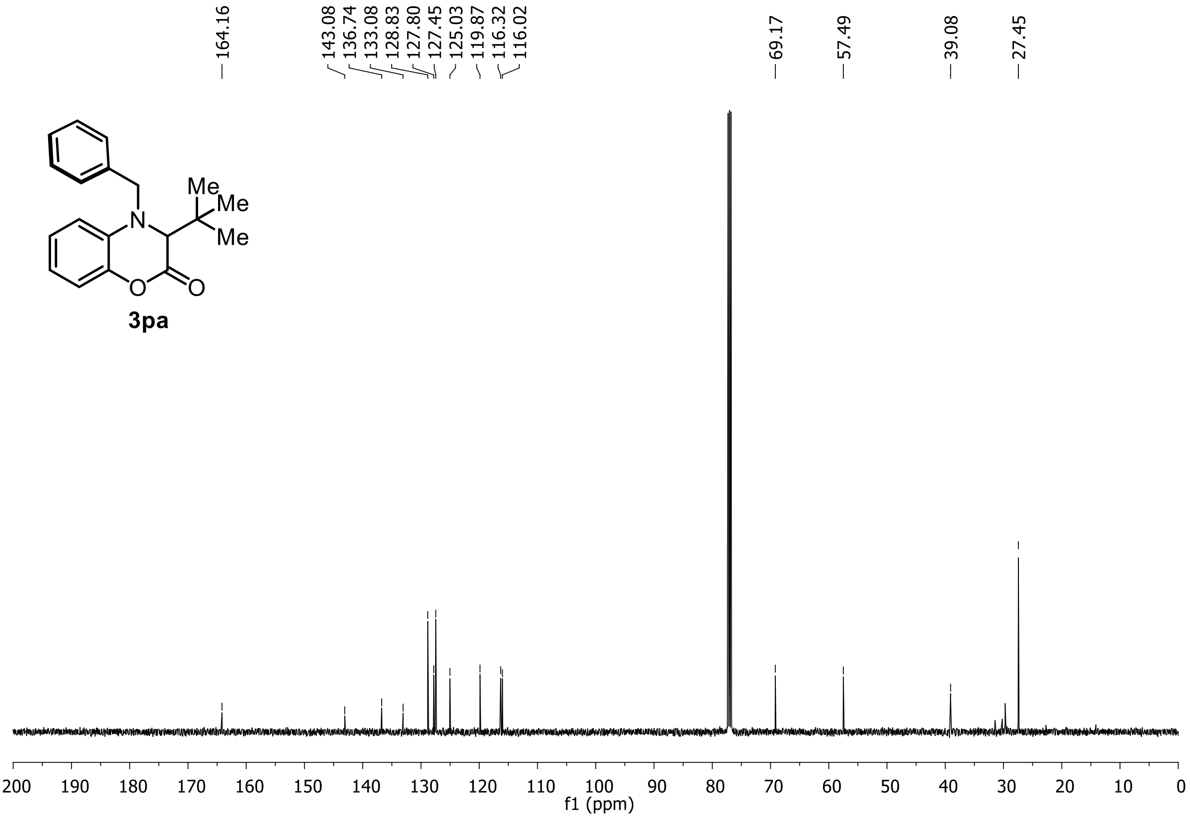
$^{13}C\{^{1}H\}$ NMR spectrum of 3oa ($CDCl_3$, 126 MHz)



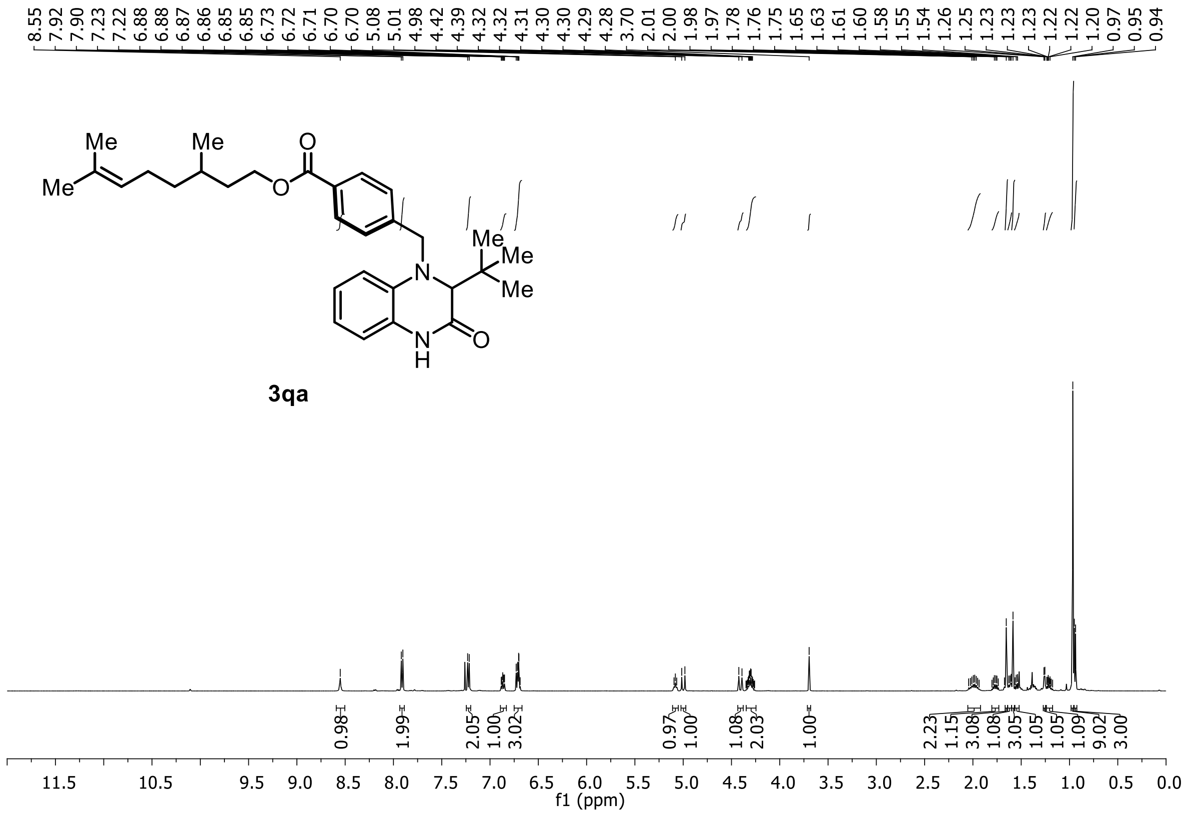
^{1}H NMR spectrum of 3pa (CDCl$_3$, 500 MHz)



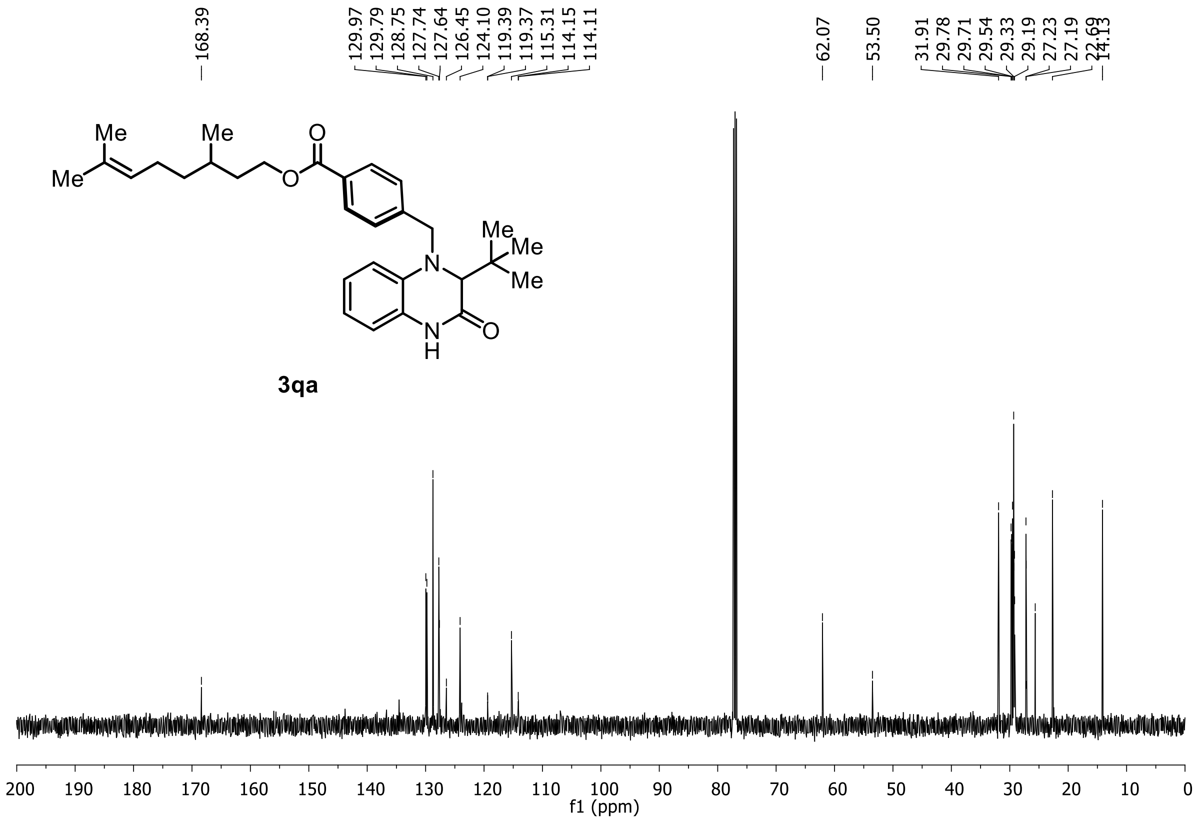
^{13}C{^{1}H} NMR spectrum of 3pa (CDCl$_3$, 126 MHz)



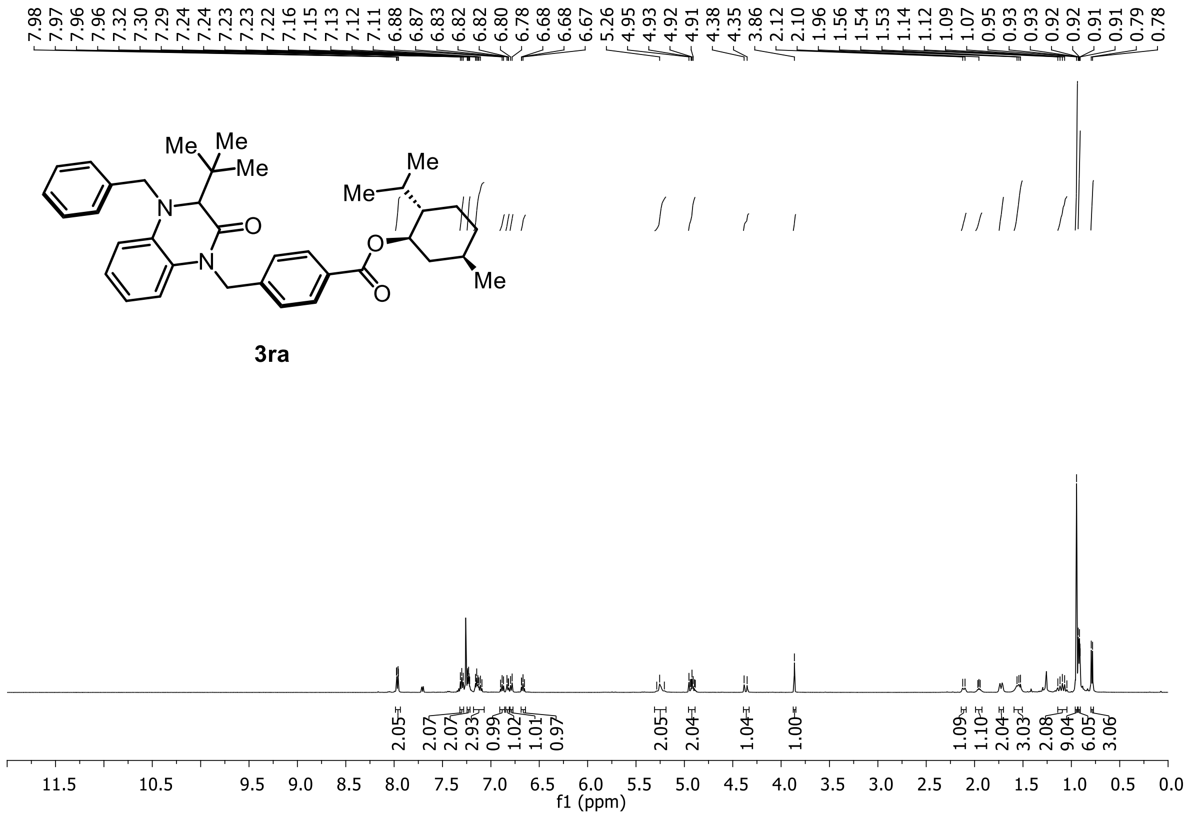
^{1}H NMR spectrum of 3qa (CDCl$_3$, 500 MHz)



^{13}C{^{1}H} NMR spectrum of 3qa (CDCl$_3$, 126 MHz)



^{1}H NMR spectrum of 3ra (CDCl$_3$, 500 MHz)



^{13}C{^{1}H} NMR spectrum of 3ra (CDCl$_3$, 126 MHz)