

Electronic Supplementary Information

**An ultrastable olefin-linked covalent organic framework for photocatalytic
decarboxylative alkylations under highly acidic conditions**

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Table of Contents

I. General information.....	S2
II. Preparation of 2D-COFs and alkyl NHPI Esters	S3
III. Structure characterizations and photocatalytic properties of 2D-COFs	S6
IV. Optimization of the reaction conditions	S11
V. General procedures for visible-light-driven decarboxylative alkylations of heterocycles	S14
VI. Recycling experiments	S15
VII. Application and scale up experiment.....	S18
VIII. Radical intermediate capture experiment	S18
IX. Calculation of Apparent Quantum Efficiency (A.Q.E.)	S19
X. <i>In situ</i> ESR spectra	S20
XI. Characterization data of compounds 3-58	S21
XII. References	S39
XIII. The ¹ H and ¹³ C NMR spectra of compounds 3-58	S40

I. General information

¹H NMR spectra were recorded on Bruker 400 or 500 MHz spectrometer with CDCl₃ and DMSO-d₆ as the solvent; ¹³C NMR spectra were recorded on Bruker 101 or 126 MHz spectrometer with CDCl₃ as the solvent. Chemical shifts were reported in parts per million (δ) with TMS (0 ppm) as the internal standard. The peak patterns are indicated as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br s = broad singlet. The coupling constants (J) are reported in Hertz (Hz). The unknown products were additionally characterized by HRMS. Powder X-ray diffraction (PXRD) patterns of the as-prepared samples were obtained on a powder X-ray diffractometer (Cu K α radiation source, Ultima IV, Rigaku). UV-Vis diffuse reflectance spectra were performed using a Hitachi UH4150 spectrophotometer in the wavelength range of 300-800 nm. FT-IR spectra were collected in the range of 400-4000 cm⁻¹ on Bruker IFS 66 v/s Fourier transform infrared spectrometer. Brunauer-Emmett-Teller (BET) surface area analysis was carried out using a Micromeritics TriStar II 3020 instrument at 77 K. Scanning electron microscopy (SEM) was performed using a ZEISS Gemini 300. The solid phases ¹³C CP/MAS NMR spectra were obtained on a Bruker 400 MHz or Agilent 600 MHz solid state NMR spectrometer. Electron spin resonance spectra (ESR) were collected on a JES-FA200 (JEOL) electron paramagnetic resonance spectrometer under visible-light irradiation.

Electrochemical measurements were carried out on a three-electrode system with CHI660E electrochemical workstation. Indium-tin oxide (ITO) glasses were cleaned by sonication in acetone for 15 min and dried under UV lamp. 5 mg of 2D-COFs powder was mixed with 0.2 mL of DMF and 0.2 mL 5 wt % Nafion to get slurry, which was spreading on the surface of ITO glass and the boundary were protected by Scotch tape. Then put it in the vacuum oven at 100 °C for 2 h. After to the room temperature and removed the Scotch tape. The measurements were carried out in a 0.1 mol L⁻¹ Sodium sulfate, Ag/AgCl electrode (saturated KCl) as reference electrode, a platinum wire as the counter electrode for photocurrent responses, electrochemical impedance spectra and Mott-Schottky (M-S) experiments. Visible-light-irradiation was provided by a 300 W xenon lamp with a $\lambda >$ 400 nm cut-off filter. The potential was measured by using a glassy carbon working electrode, the electrolyte was a 0.1 mol L⁻¹ solution of tetrabutylammonium hexafluorophosphate in acetonitrile. A Pt electrode, and a calomel electrode (SCE) as counter and reference electrode. Scan rate: 100 mV/s.

All starting materials (from energy chemical or bidepharm) and solvents were used as received and

without further purification, unless otherwise specified.

II. Preparation of 2D-COFs and substrates

i. Preparation of 2D-COFs

2D-COF-1,¹ 2D-COF-2,² 2D-COF-3,³ 2D-COF-4⁴ and 2D-COF-5⁵ were synthesized according to the literature and our previously reported methods^{1, 16}.

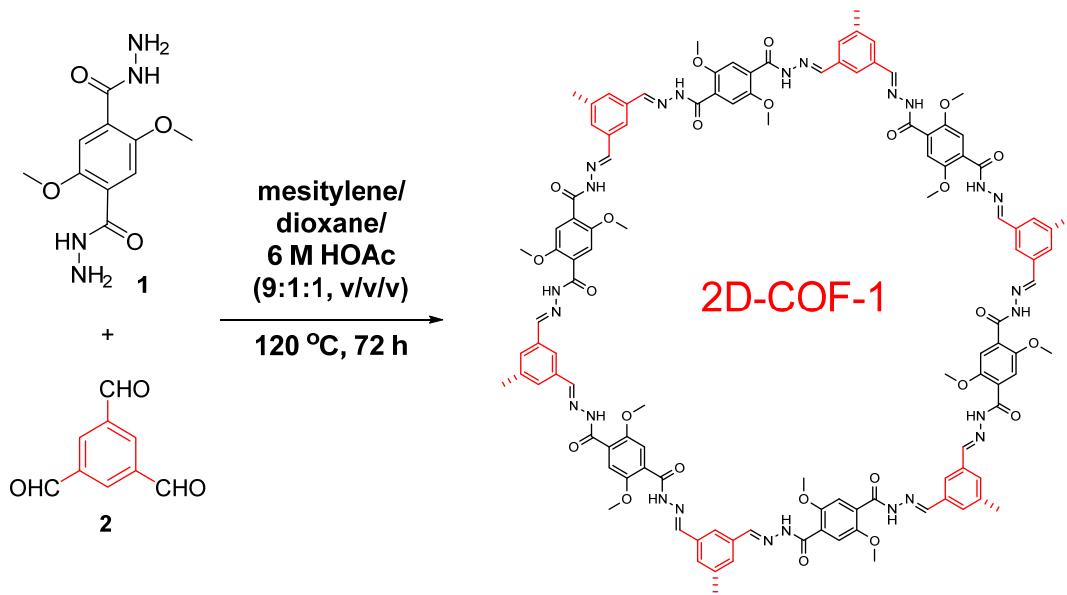


Figure S1. Synthesis of 2D-COF-1.

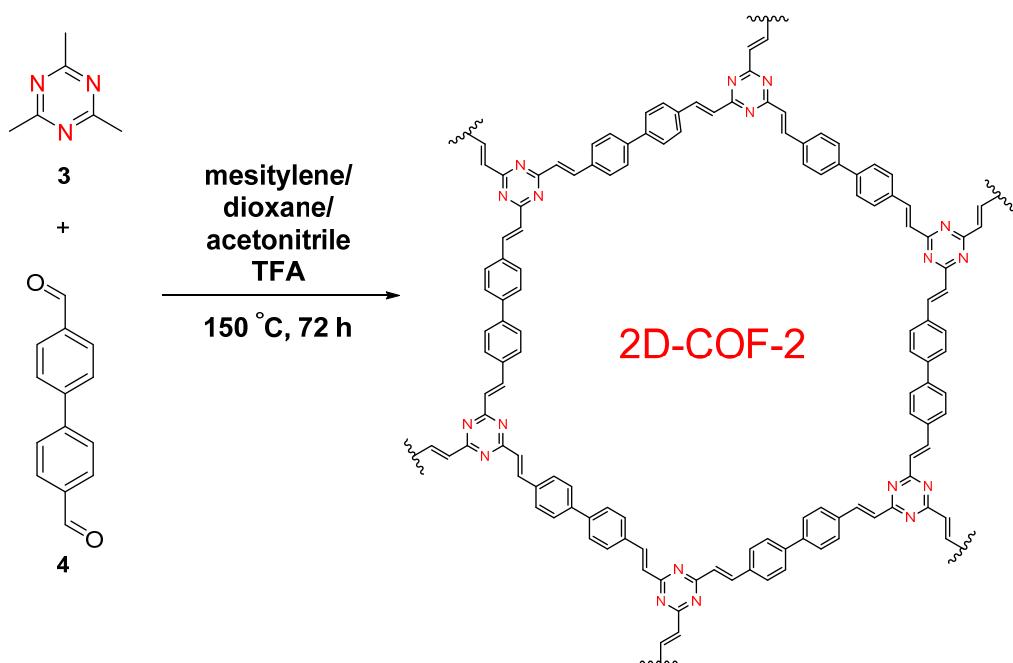


Figure S2. Synthesis of 2D-COF-2.

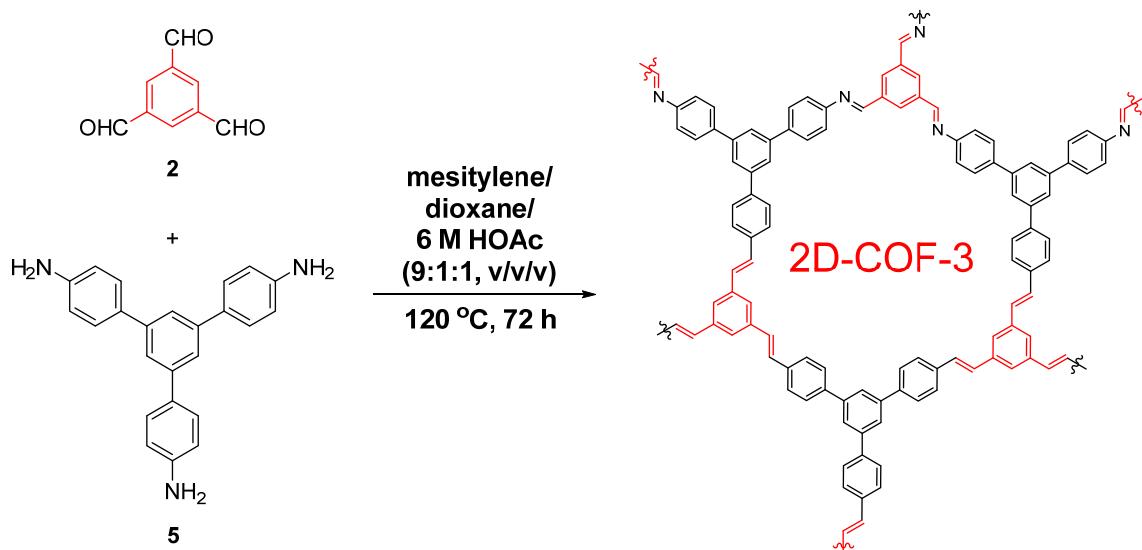


Figure S3. Synthesis of 2D-COF-3.

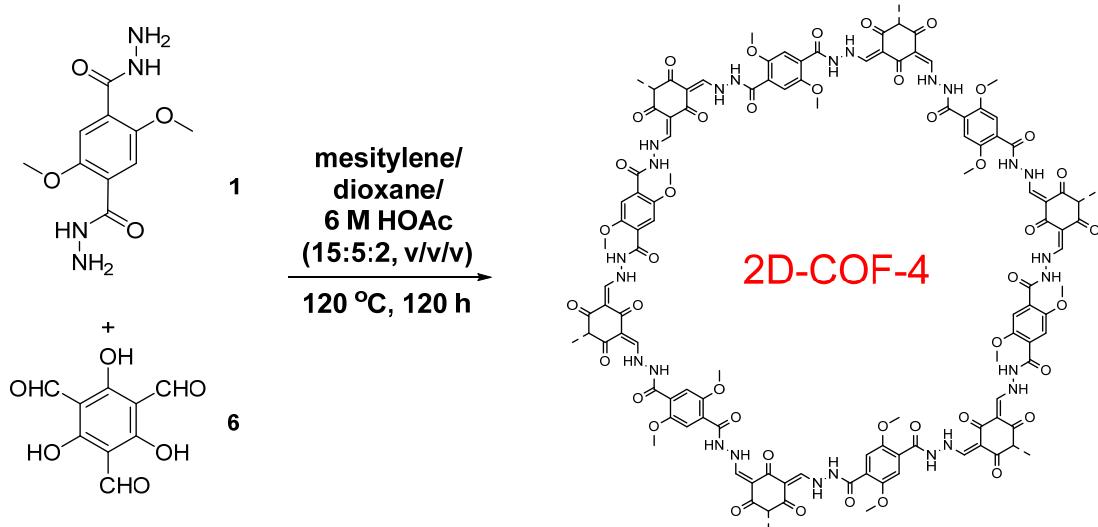


Figure S4. Synthesis of 2D-COF-4.

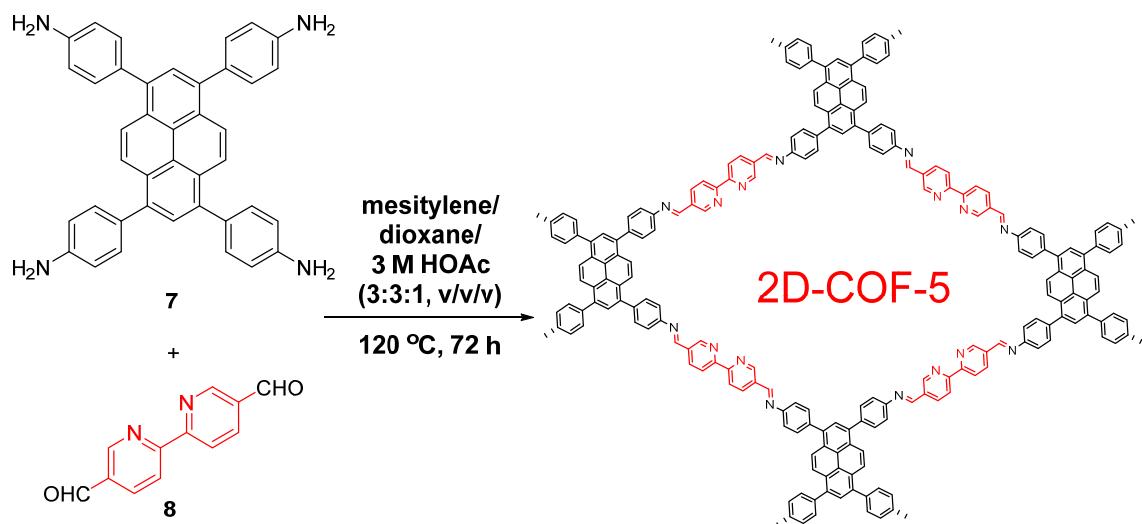
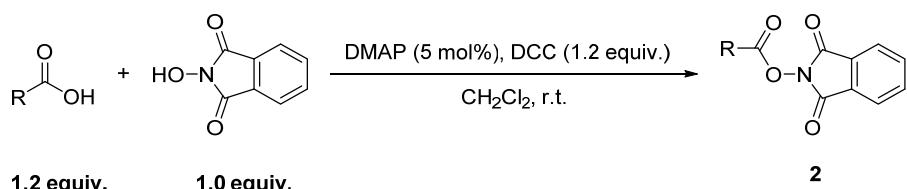


Figure S5. Synthesis of 2D-COF-5.

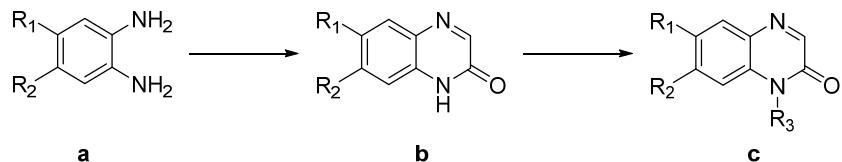
ii. Preparation of alkyl NHPI esters

According to literature reports,⁶ the alkyl NHPI esters can be synthesized by the condensation of *N*-hydroxyphthalimide with the corresponding carboxylic acids.



N-hydroxyphthalimide (5 mmol, 1.0 equiv.) and the corresponding alkyl carboxylic acids (6 mmol, 1.2 equiv.) and 4-dimethylaminopyridine (2.5 mol%) were mixed in a round-bottomed flask with a magnetic stirring bar, then 10 mL CH₂Cl₂ was added. A solution of *N,N'*-dicyclohexylcarbodiimide (6 mmol, 1.2 equiv.) in CH₂Cl₂ was added slowly. The reaction mixture was allowed to stir at room temperature for 0.5-3 hours. The white solid was removed by filtration and the filtrate was concentrated under reduced pressure. The crude product was purified by chromatography on a silica gel column.

iii. Preparation of quinoxalin-2(1*H*)-ones⁷



O-arylenediamine **a** (10 mmol, 1 equiv.) and ethanol (10 mL) were mixed in a round-bottomed flask with a magnetic stirring bar, and ethyl 2-oxoacetate (11 mmol, 1.1 equiv.) was added. The reaction mixture was stirred at reflux for 1 h, then maintained overnight at room temperature. The solid was filtered and washed with ethanol, then dried to give quinoxalinone **b**. Quinoxalinone **b** (1 equiv.), DMF (5 mL), and potassium carbonate (1.2 equiv.) were mixed in another round-bottomed flask with a magnetic stirring bar, then the corresponding halogenoalcano (1.6 equiv.) was added slowly. The reaction mixture was stirred overnight at room temperature. The liquid was extracted with ethyl acetate. The organic layers were washed with a saturated solution of NH₄Cl then brine, dried over MgSO₄, filtered and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel to afford the desired product **c**.

III. Structure characterizations and photocatalytic properties of 2D-COFs

In our previous^{1, 16} and newly published online work⁸, some chemical structure characterizations and photocatalytic property measurements of 2D-COF-1, 2D-COF-3, and 2D-COF-5 have been already performed and reported, so they are not repeated here.

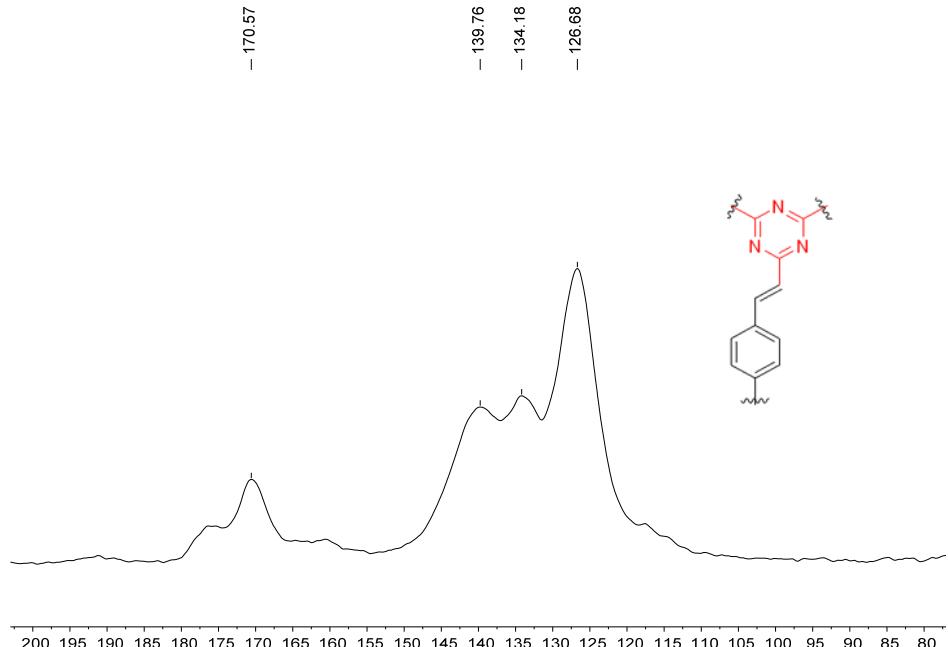


Figure S6. ¹³C CP/MAS NMR spectra of 2D-COF-2.

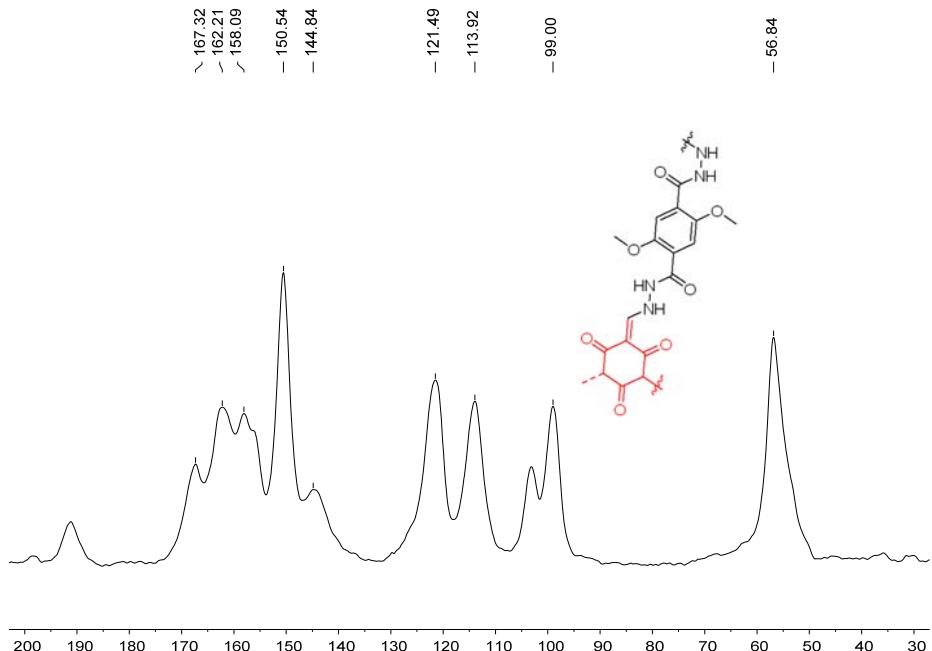


Figure S7. ¹³C CP/MAS NMR spectra of 2D-COF-4.

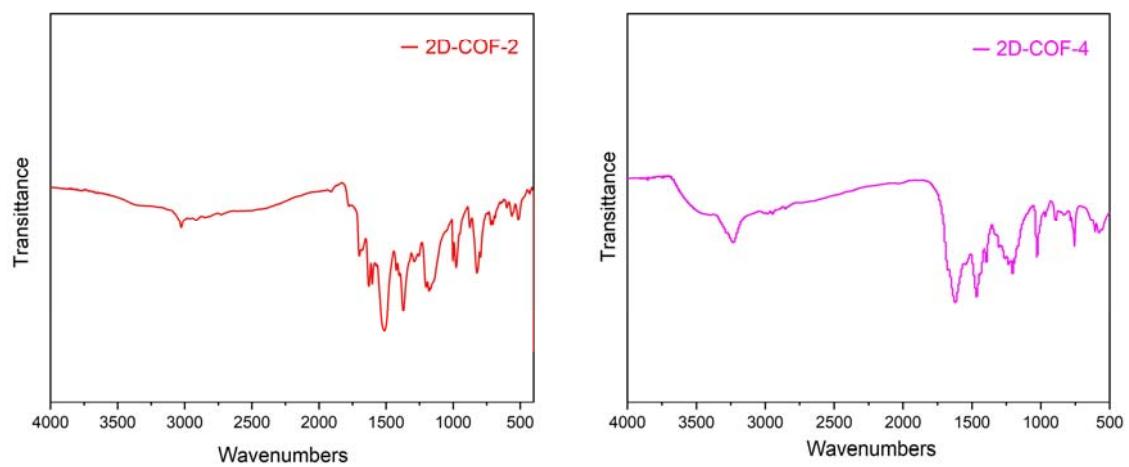


Figure S8. IR spectra of 2D-COF-2 and 2D-COF-4.

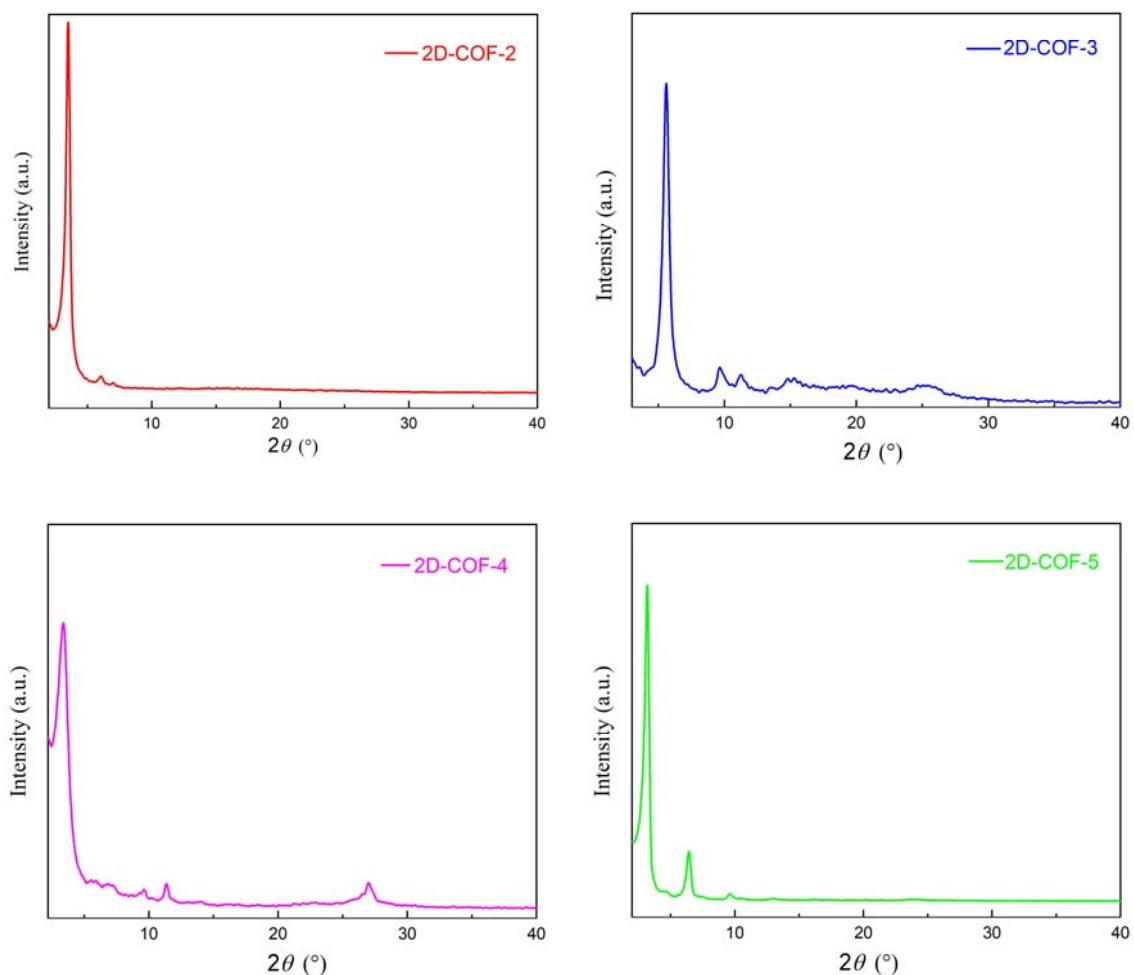


Figure S9. Powder X-ray diffraction of 2D-COFs.

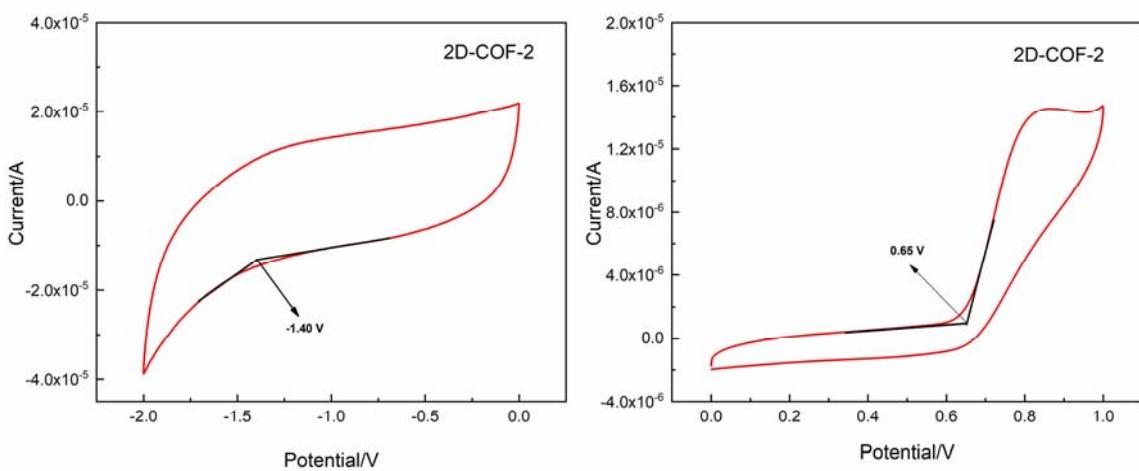


Figure S10. Cyclic voltammetry of 2D-COF-2 at a scan rate of 100 mV/s.

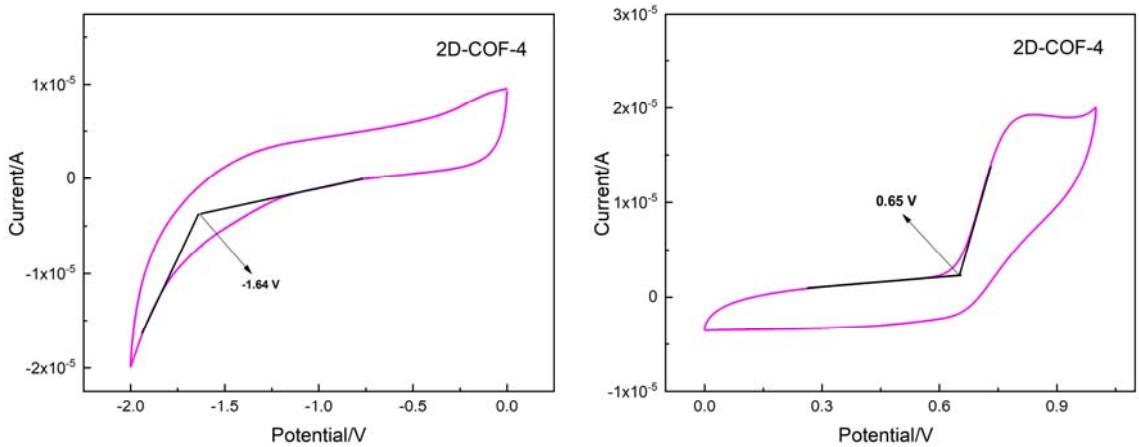


Figure S11. Cyclic voltammetry of 2D-COF-4 at a scan rate of 100 mV/s.

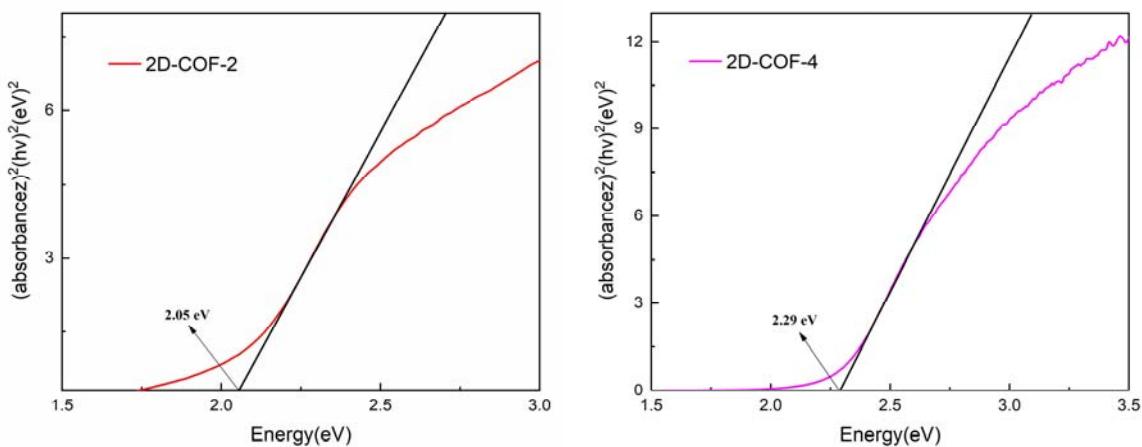


Figure S12. Direct Kubelka-Munk plot for 2D-COF-2 and 2D-COF-4.

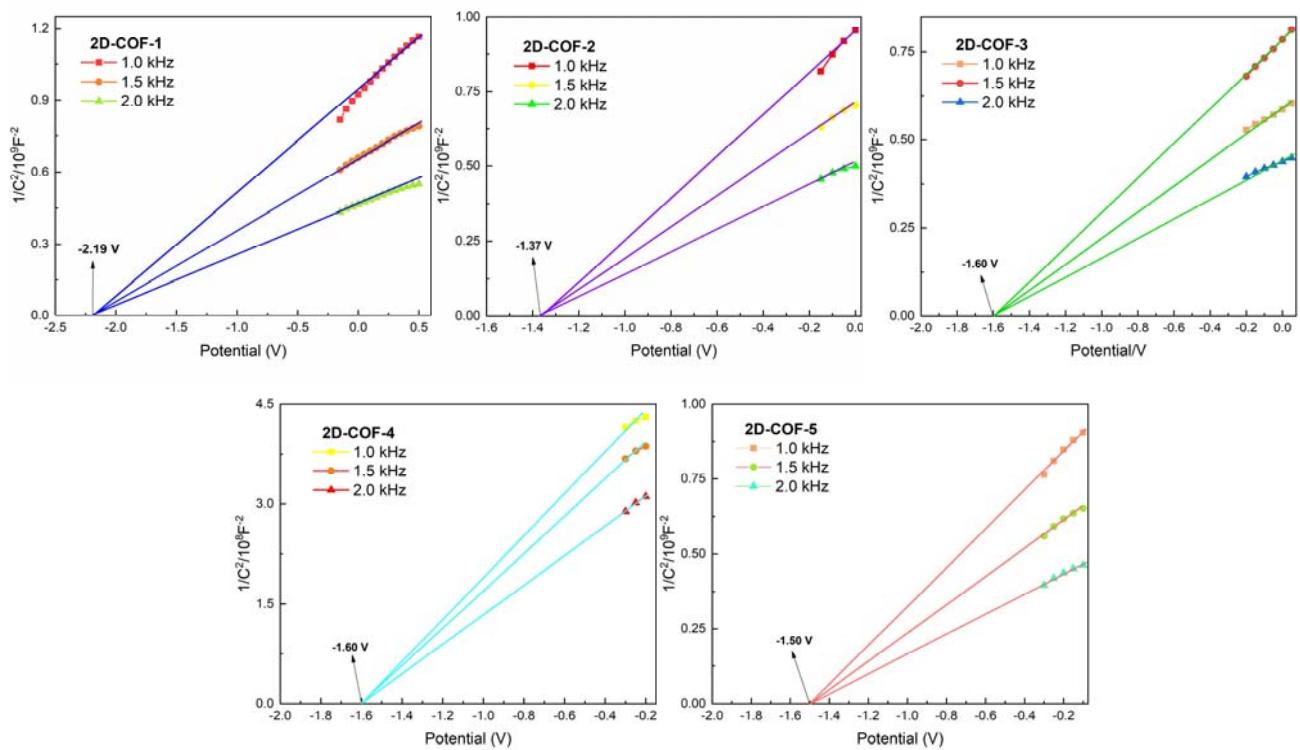


Figure S13. Mott-Schottky (M-S) plot for 2D-COFs measured in $0.1 \text{ mol L}^{-1} \text{Na}_2\text{SO}_4$ (pH 7.1) with Ag/AgCl (+0.199 V vs. NHE) as the reference electrode under dark.

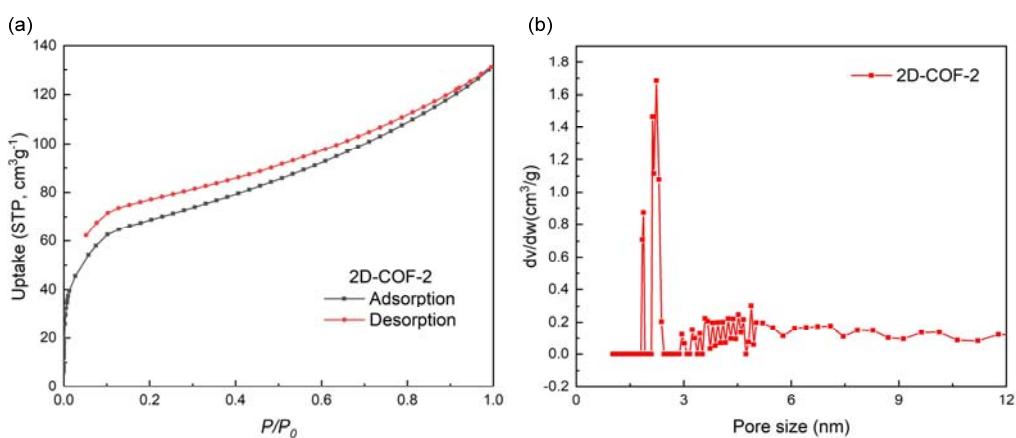


Figure S14. (a) N_2 adsorption and desorption isotherms of 2D-COF-2 measured at 77 K. (b) Pore size distributions of 2D-COF-2 derived from N_2 sorption isotherm measured at 77 K.

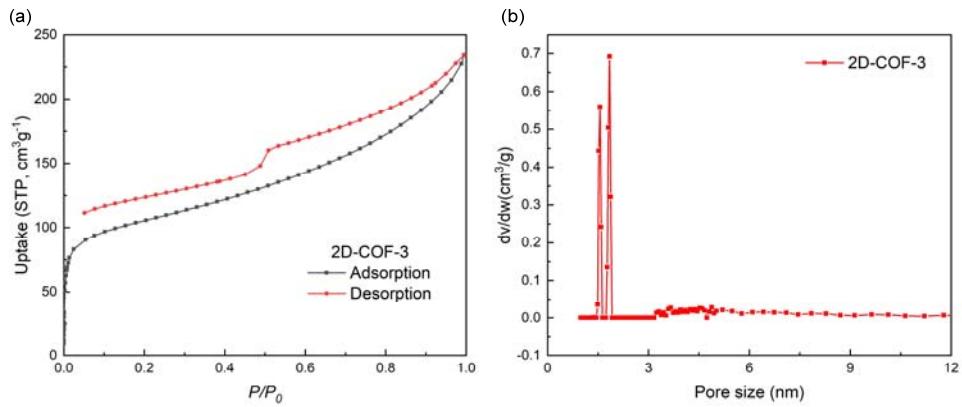


Figure S15. (a) N_2 adsorption and desorption isotherms of 2D-COF-3 measured at 77 K. (b) Pore size distributions of 2D-COF-3 derived from N_2 sorption isotherm measured at 77 K.

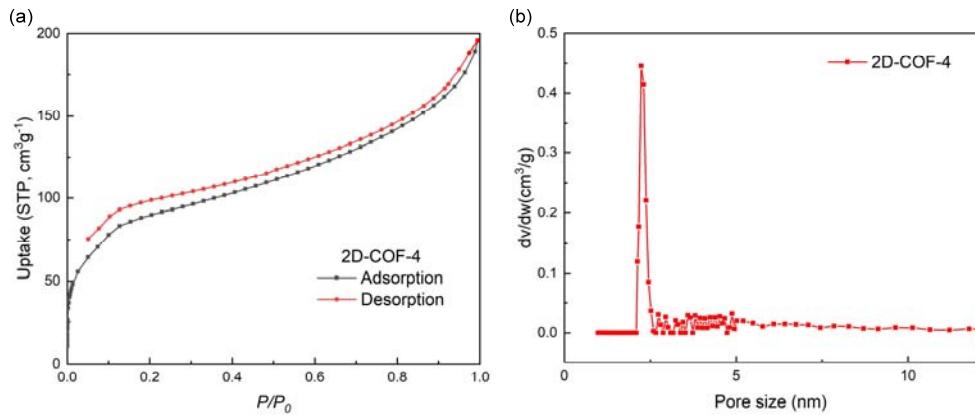


Figure S16. (a) N_2 adsorption and desorption isotherms of 2D-COF-4 measured at 77 K. (b) Pore size distributions of 2D-COF-4 derived from N_2 sorption isotherm measured at 77 K.

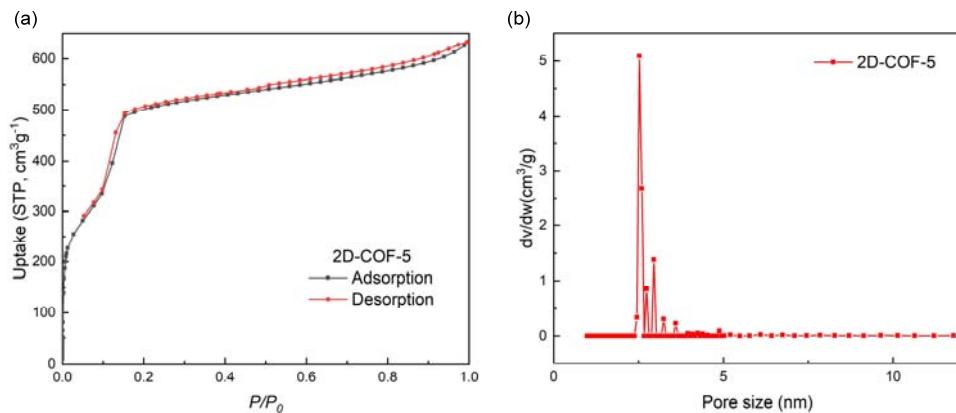
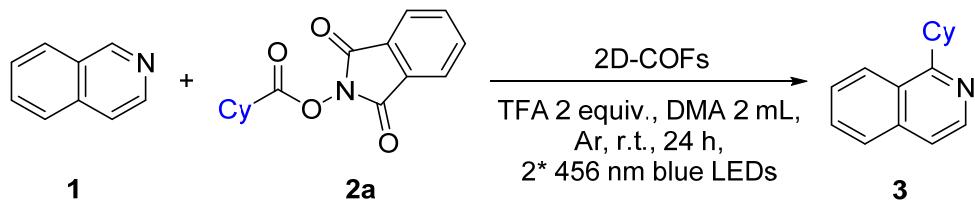


Figure S17. (a) N_2 adsorption and desorption isotherms of 2D-COF-5 measured at 77 K. (b) Pore size distributions of 2D-COF-5 derived from N_2 sorption isotherm measured at 77 K.

IV. Optimization of the reaction conditions

Table S1. Screening of photocatalysts^a.



Entry	Change from the standard reaction conditions	Yields (%) ^b
1	2D-COF-1	69
2	2D-COF-2	85
3	2D-COF-3	78
4	2D-COF-4	59
5	2D-COF-5	50

^a Standard reaction conditions: **1** (0.1 mmol, 12.9 mg), **2a** (0.2 mmol, 54.6 mg), 2D-COFs (2D-COF-1 (4 mg), 2D-COF-2 (2 mg), 2D-COF-3 (3 mg), 2D-COF-4 (4 mg), 2D-COF-5 (4 mg)), TFA (0.2 mmol, 22.8 mg) in 2 mL DMA, 2* 456 nm blue LED, Ar, r.t., 24 h. ^b Isolated yield.

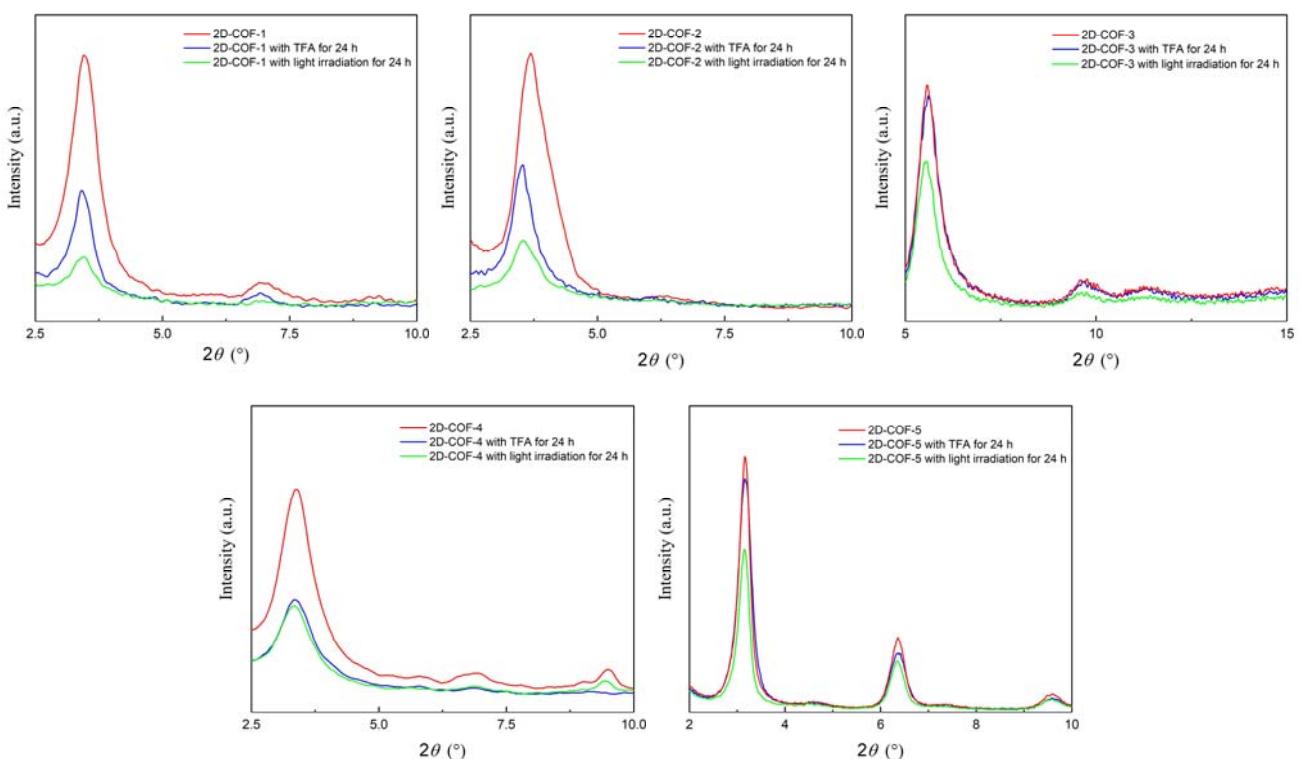


Figure S18. PXRD patterns of newly synthesized 2D-COFs (red), PXRD patterns of recycled 2D-COFs (30 mg) after the treatment with TFA (3 mmol) in DMA (5 mL) for 24 h (blue), PXRD patterns of recycled 2D-COFs (30 mg) after the treatment with blue LED irradiation (2*40 W blue LEDs 456 nm) in DMA (5 mL) for 24 h (green).

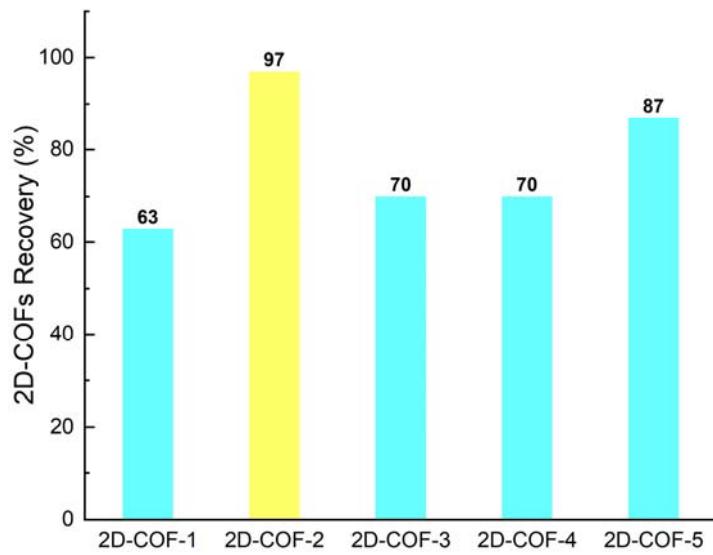
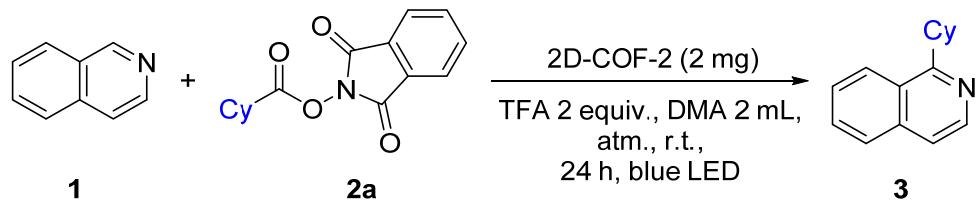


Figure S19. The recovery rate of 2D-COFs (30 mg) under standard reaction conditions.

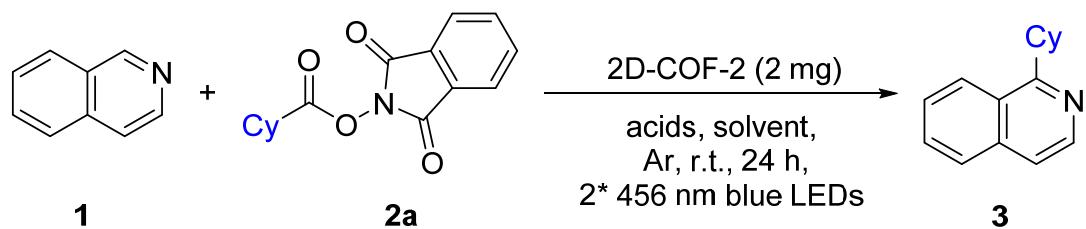
Table S2. Control experiments^a.



Entry	Change from standard conditions	Yields (%) ^b
1	under oxygen atmosphere	N.R.
2	under air atmosphere	N.R.
3	without light	N.R.
4	390 nm blue LED, 40 W	64%
5	427 nm blue LED, 40 W	49%
6	467 nm blue LED, 40 W	trace

^a Standard reaction conditions: **1** (0.1 mmol, 12.9 mg), **2a** (0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), TFA (0.2 mmol, 22.8 mg) in 2 mL DMA, 2* 456 nm blue LED, 40 W, Ar, r.t., 24 h. ^b Isolated yield.

Table S3. Screening of different additives and solvents^a.

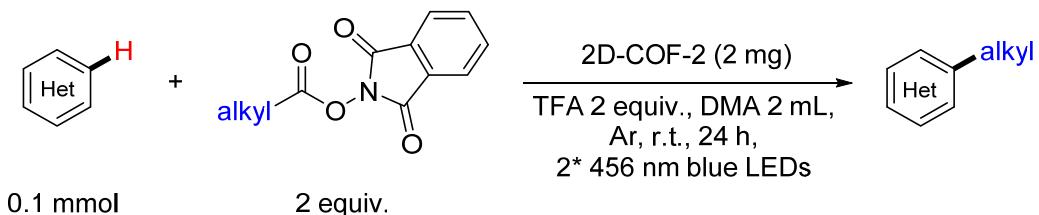


Entry	Additive	Solvent	Yields (%) ^b
1	TFA	DMA	85
2	TFA	DMF	43
3	TFA	DMSO	N.R.
4	TFA	Acetone	N.R.
5	TFA	EtOH	76
6	TFA	DIPE	N.R.
7	TFA	EA	N.R.
8	TFA	DCM	N.R.
9	TFA	CHCl ₃	N.R.
10	TFA	CH ₃ OH	63
11	TFA	H ₂ O	Trace
12	TFA	MeCN	N.R.
13	TfOH	DMA	62
14	CH ₃ COOH	DMA	5
15	Adamantoic acid	DMA	69
16	-	DMA	44

^a Reaction conditions: **1**(0.1 mmol, 12.9 mg), **2a** (0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), additive (0.2 mmol, 2 equiv.), solvent 2 mL, Ar, r.t., 24 h, 2*40 W blue LED (456 nm). ^b Isolated yield.

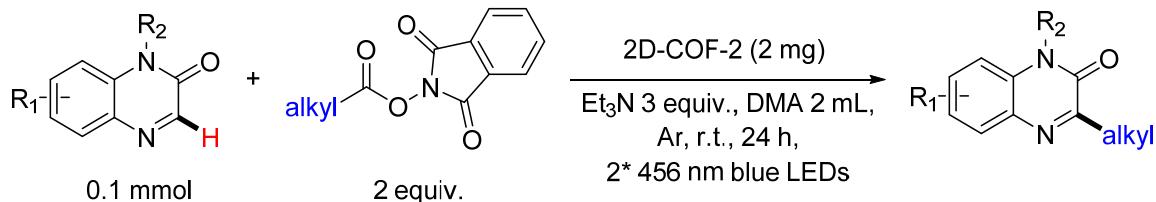
V. General procedures for visible-light-driven decarboxylative alkylations of heterocycles

i. General procedure A



To a 10 mL glass tube was charged with heterocycles (0.1 mmol, 1 equiv.), alkyl NHPI ester (0.2 mmol, 2 equiv.), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMA (2 mL) and TFA (0.2 mmol, 2 equiv.) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, the remaining mixture was slowly added to a saturated aq solution of Na₂CO₃ (10 mL). The aqueous layer was extracted with ethyl acetate (5 mL × 3). The organic layers were washed with saturated brine then dried (MgSO₄), filtered, and concentrated under reduced pressure. the remaining mixture was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product.

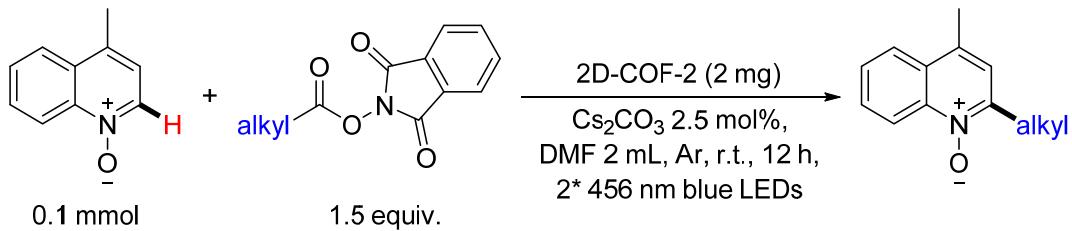
ii. General procedure B



To a 10 mL glass tube was charged with quinoxalin-2(1H)-ones (0.1 mmol, 1 equiv.), alkyl NHPI ester (0.2 mmol, 2 equiv.), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMA (2 mL) and Et₃N (0.3 mmol, 3 equiv.) were added *via* a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, the remaining mixture was quenched with water and extracted with ethyl acetate (5 mL × 3). The organic layers were washed with saturated

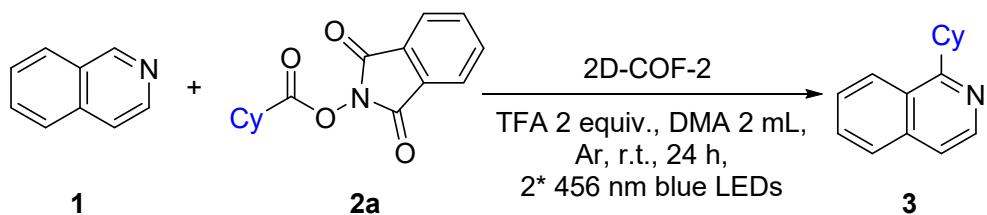
brine then dried (MgSO_4), filtered, and concentrated under reduced pressure. The remaining mixture was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product.

iii. General procedure C



To a 10 mL glass tube was charged with *N*-oxides (0.1 mmol, 1 equiv.), alkyl NHPI ester (0.15 mmol, 1.5 equiv.), Cs_2CO_3 (2.5 mol%), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMF (2 mL) was added *via* a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm^2 , and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 12 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, the remaining mixture was quenched with water and extracted with ethyl acetate (5 mL \times 3). The organic layers were washed with saturated brine then dried (MgSO_4), filtered, and concentrated under reduced pressure. The residue was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product.

VI. Recycling experiments



To a 10 mL glass tube was charged with Isoquinoline (**1**, 0.1 mmol, 12.9 mg), cyclohexyl NHPI ester (**2a**, 0.2 mmol, 54.6 mg), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMA (2 mL) and TFA (0.2 mmol, 22.8 mg) were added *via* a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm^2 , and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, and washed with plenty of ethyl acetate,

ethanol and water. Then the powder was dried at 120 °C under vacuum for 6 h to yield the recovered 2D-COF-2. In addition, the remaining mixture was slowly added to a saturated aq solution of Na₂CO₃ (10 mL). The aqueous layer was extracted with ethyl acetate (5 mL × 3). The organic layers were washed with saturated brine then dried (MgSO₄), filtered, and concentrated under reduced pressure. The residue was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product **3**.

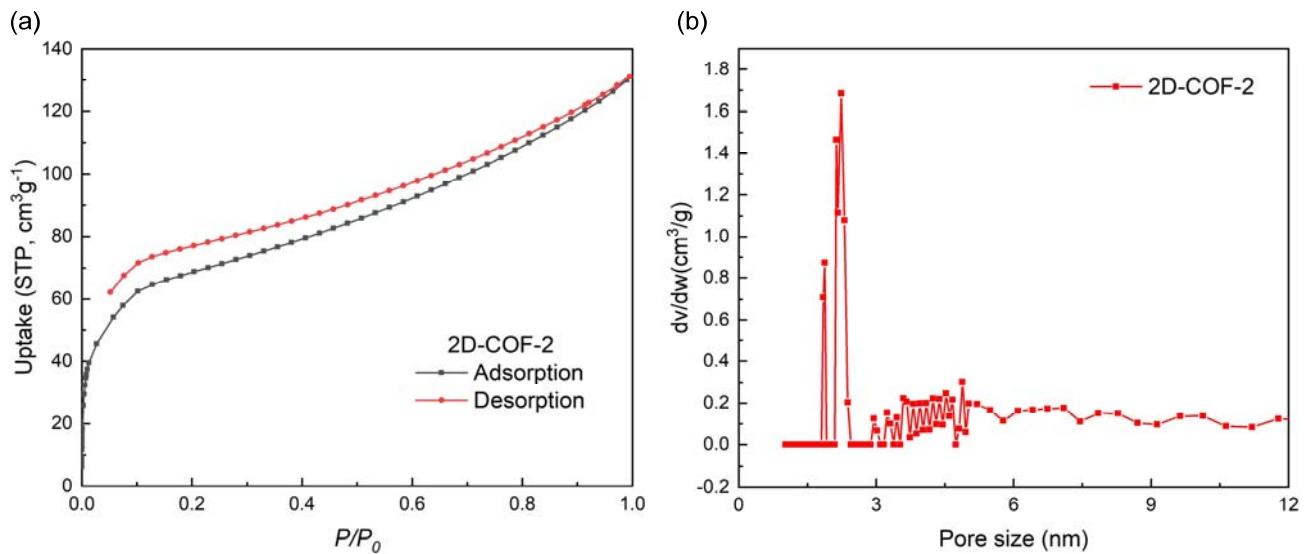


Figure S20. (a) N_2 sorption isotherm of 2D-COF-2. (b) Pore size distributions of 2D-COF-2.

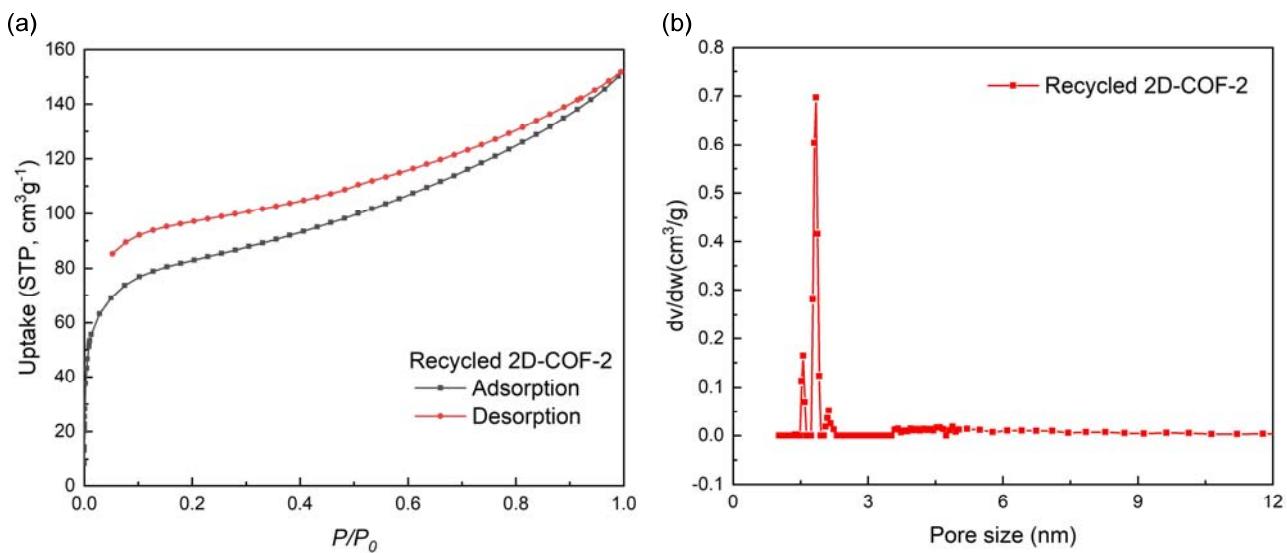
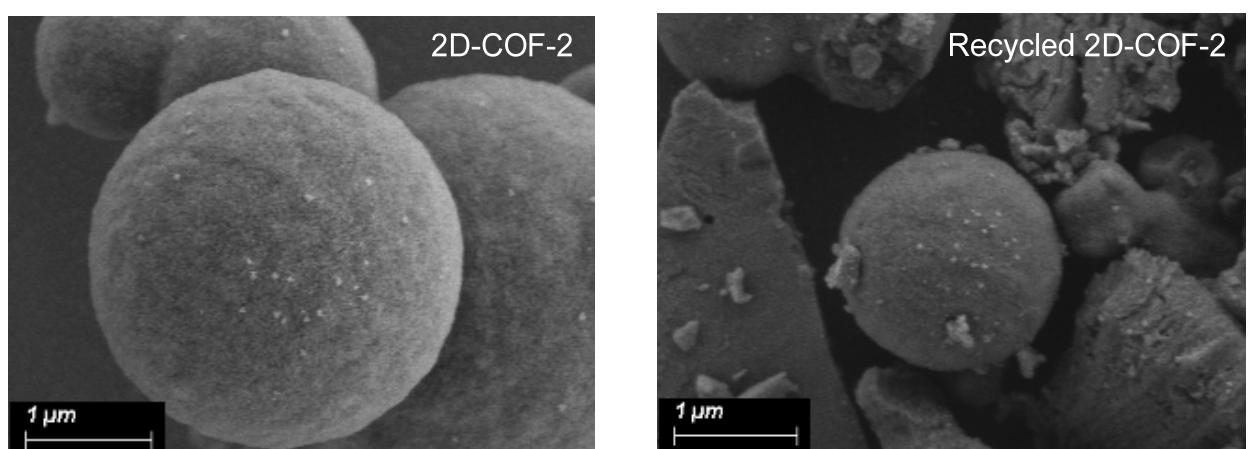
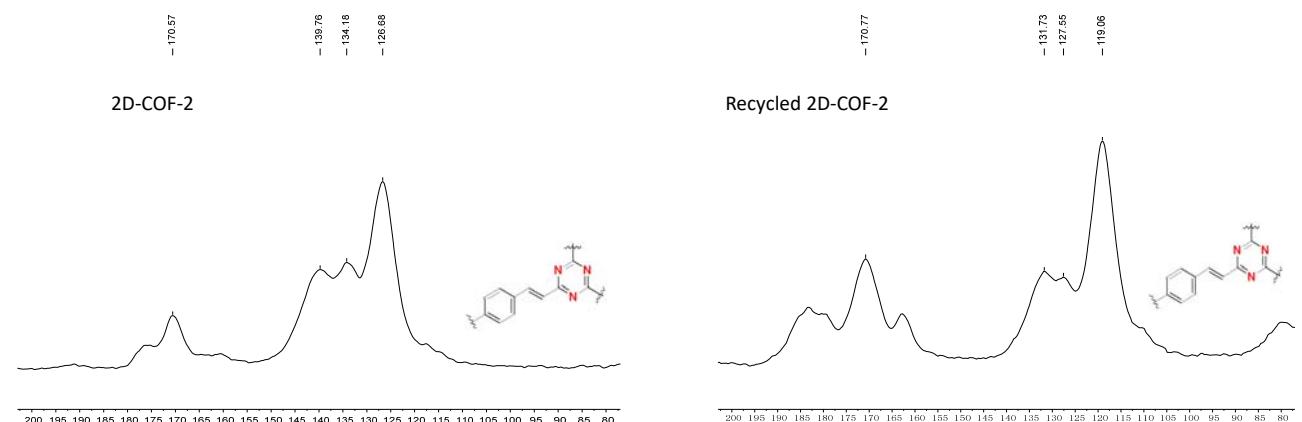
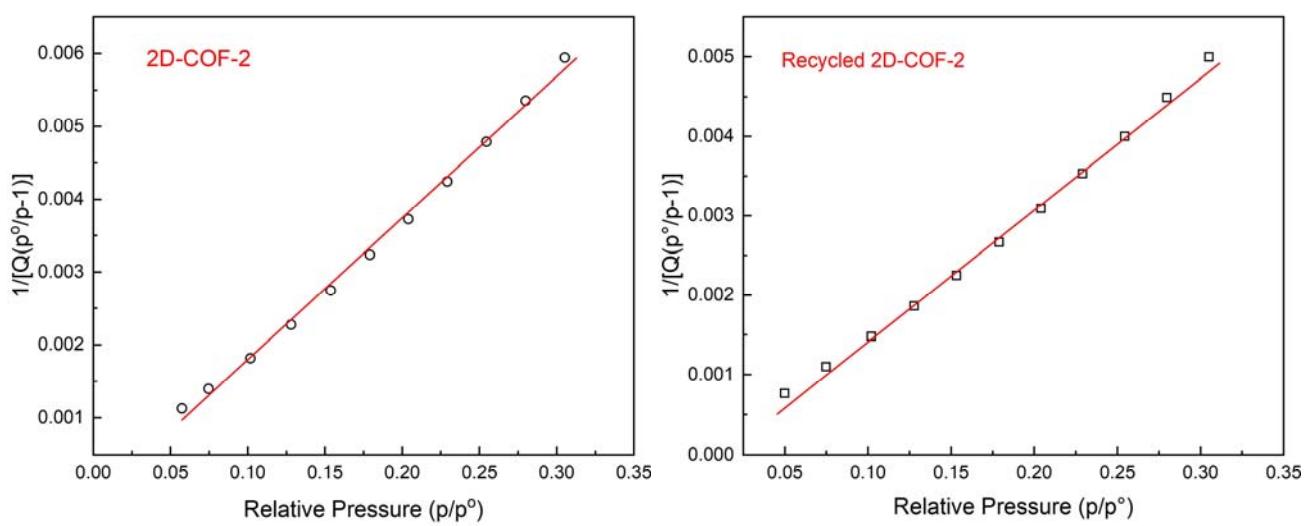
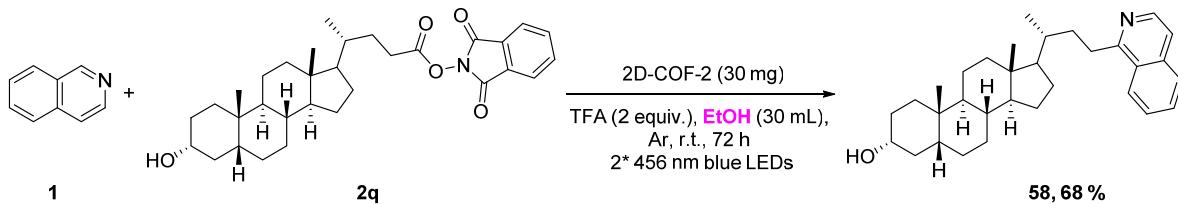


Figure S21. (a) N_2 sorption isotherm of 2D-COF-2 after recycling. (b) Pore size distributions of 2D-COF-2 after recycling.

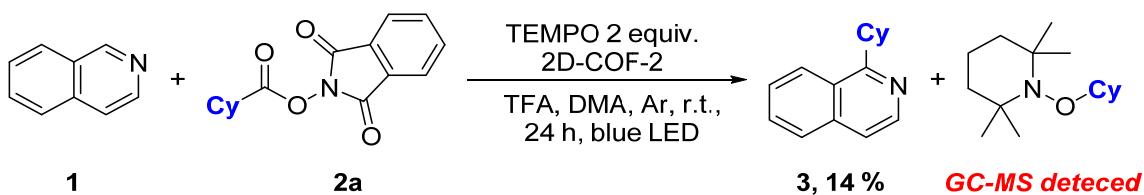


VII. Application and scale up experiment



Isoquinoline (**1**, 0.5 g, 3.88 mmol), alkyl NHPI ester (**2q**, 2 equiv., 7.76 mmol), 2D-COF-2 (30 mg) were added into a reaction flask (50 mL) equipped with a magnetic stir bar. Then, the flask was evacuated and filled with argon for three cycles. TFA (7.76 mmol, 2 equiv.) and EtOH (30 mL) were added *via* a gastight syringe under argon atmosphere. The reaction mixture was stirred (1500 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 72 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, then Potassium carbonate (3.88 mmol, 0.535 g) was added into the remaining mixture for neutralization, the solvent was removed under reduced pressure and the residue was on silica gel (petroleum ether and ethyl acetate) to afford the desired product **58** (0.31 g, 68 %).

VIII. Radical intermediate capture experiment



Isoquinoline (**1**, 0.1 mmol, 12.9 mg), cyclohexyl NHPI ester (**2a**, 0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO, 0.2 mmol, 31.2 mg) were placed in a glass tube equipped with a stirring bar, the tube was evacuated and filled with argon for three cycles. TFA (0.2 mmol, 22.8 mg) and DMA (2 mL) were added *via* a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. The reaction was greatly suppressed. The photocatalyst 2D-COF-2 was removed by centrifugation, the above solution was detected by GC-MS. The cyclohexyl radical adduct 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine was observed in GC-MS spectra. The desired product **3** was isolated in 14 % yield.

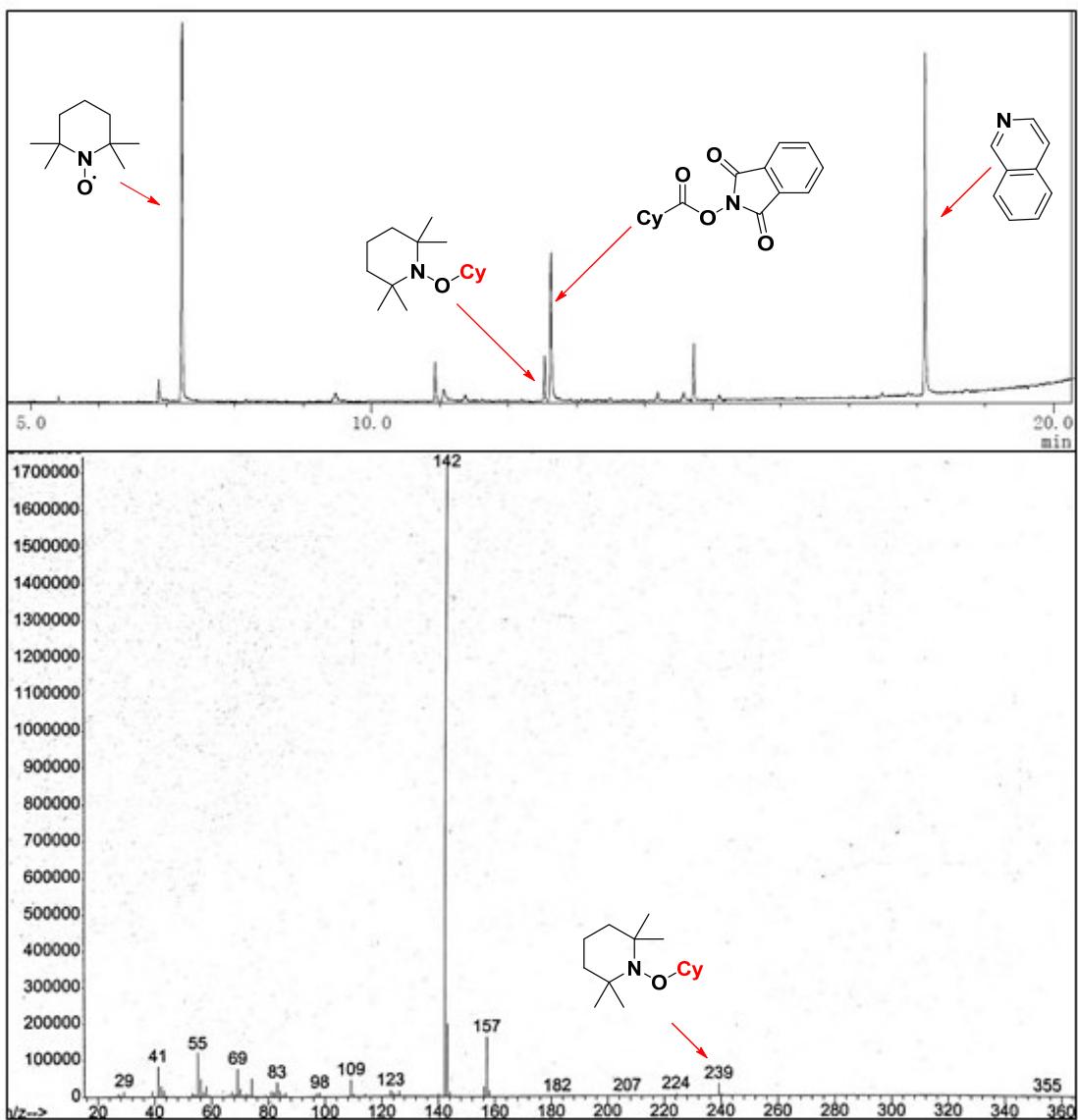


Figure S25. GC-MS spectra of the model reaction in the presence of 2 equiv. TEMPO

IX. Calculation of Apparent Quantum Efficiency (A.Q.E.)

In principle, it takes one photon to generate one radical to form one target product. The energy of one photon (E_{photon}) with wavelength of λ_{inc} (nm) is calculated using eq.1.

$$E_{\text{photon}} = \frac{hc}{\lambda_{\text{inc}}} = \frac{6.63 \times 10^{-34} \text{ J}\cdot\text{s} \times 3 \times 10^8 \text{ ms}^{-1}}{456 \times 10^{-9} \text{ m}} = 4.36 \times 10^{-19} \text{ J} \quad (\text{eq.1})$$

Where h (J·s) is Planck's constant, c (m·s⁻¹) is the speed of light and λ_{inc} (m) is the wavelength of the incident light, 456 nm. And the total energy of the incident monochromatic light (E_{total}) is calculated using eq.2.

$$E_{\text{total}} = PSt \quad (\text{eq.2})$$

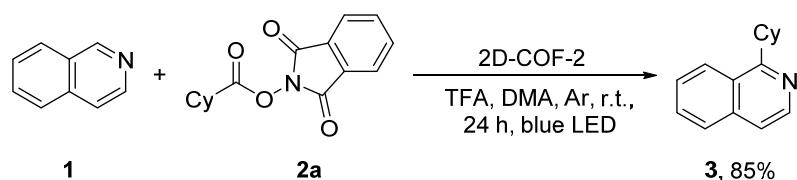
Where P ($\text{W}\cdot\text{cm}^{-2}$) is the power density of the incident light, S (cm^2) is the irradiation area, t (s) is the photoreaction time. The total number of incident photons can be obtained through eq.3.

$$\text{Number of incident photons} = \frac{E_{\text{total}}}{E_{\text{photon}}} \quad (\text{eq.3})$$

The apparent quantum efficiency (A.Q.E.) is defined as eq.4.

$$\text{A.Q.E.(%)} = \frac{\text{Number of product}}{\text{Number of incident photons}} \times 100\% \quad (\text{eq.4})$$

A.Q.E% of the reaction between **1** and **2a**.



$$E_{\text{total}} = PSt = 0.003 \text{ W} / \text{cm}^2 \times 2 \text{ cm}^{-2} \times 24 \times 3600 \text{ s} = 518.4 \text{ J}$$

$$\text{Number of incident photons} = \frac{E_{\text{total}}}{E_{\text{photon}}} = \frac{518.4 \text{ J}}{4.36 \times 10^{-19} \text{ J}} = 1.19 \times 10^{21} = 1.97 \text{ mmol}$$

$$\text{A.Q.E.(%)} = \frac{\text{Number of product}}{\text{Number of incident photons}} \times 100 \% = \frac{0.085 \text{ mmol}}{1.97 \text{ mmol}} \times 100 \% = 4.3 \%$$

X. *In situ* ESR spectra

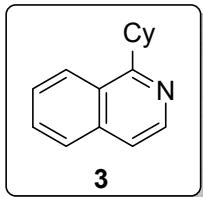
A glass tube was charged with cyclohexyl NHPI ester **2a** (0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), TFA (0.2 mmol, 22.8 mg) and 2 mL DMA in the dark room. The mixture was degassed by bubbling with N_2 for 5 minutes. Under nitrogen atmosphere, 50 μL of DMPO was added. The formed mixture (50-100 μL) was transferred into the ESR capillary and sealed quickly and subsequently. The ESR spectra was firstly recorded under dark. Secondly, it was recorded with the light irradiation from a 300 W Xenon lamp ($\lambda > 400 \text{ nm}$) for 2 minutes.

Finally, another glass tube was charged with cyclohexyl NHPI ester **2a** (0.2 mmol, 54.6 mg), isoquinoline **1** (0.1 mmol, 12.9 mg), 2D-COF-2 (2 mg), TFA (0.2 mmol, 22.8 mg) and 2 mL DMA in the dark room. The mixture was degassed by bubbling with N_2 for 5 minutes. Under nitrogen atmosphere, 50 μL of DMPO was added. The formed mixture (50-100 μL) was transferred into the ESR capillary and sealed quickly and subsequently. The third ESR spectra was then recorded with the light irradiation from a 300 W Xenon lamp ($\lambda > 400 \text{ nm}$) for 2 minutes.

ESR conditions: frequency (9.057 GHz), power (0.998 mW), modulation width (0.1 mT), center field (322.500 mT), sweep width (5 mT), sweep time (1 min), time constant (0.03 s).

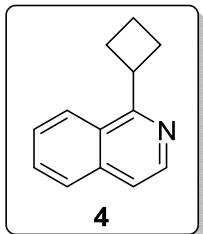
XI. Characterization data of compounds 3-58.

1-cyclohexylisoquinoline (3)⁹



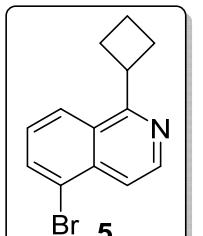
Following the general procedure A, **3** was obtained in 85% yield (18 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.44 (d, *J* = 5.7 Hz, 1H), 8.13 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 7.7 Hz, 1H), 7.55-7.43 (m, 2H), 7.36 (d, *J* = 5.7 Hz, 1H), 3.50 (tt, *J* = 11.5, 3.2 Hz, 1H), 1.97-1.73 (m, 7H), 1.54-1.29 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 165.45, 141.78, 136.19, 129.31, 127.35, 126.61, 126.09, 124.50, 118.70, 41.39, 32.47, 26.76, 26.16.

1-cyclobutylisoquinoline (4)¹⁰



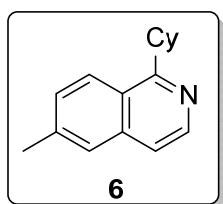
Following the general procedure A, **4** was obtained in 82% yield (15 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 8.49 (d, *J* = 5.7 Hz, 1H), 8.08-8.04 (m, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.64 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H), 7.56 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 7.49 (d, *J* = 5.7 Hz, 1H), 4.37 (p, *J* = 8.7 Hz, 1H), 2.69-2.58 (m, 2H), 2.50 (m, 2H), 2.25-2.12 (m, 1H), 2.01-1.92 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 163.60, 141.92, 136.24, 129.77, 127.42, 126.87, 126.47, 125.34, 119.13, 39.46, 27.85, 18.69.

5-bromo-1-cyclobutylisoquinoline (5)¹¹



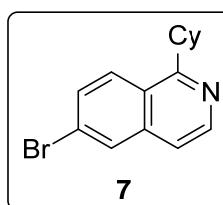
Following the general procedure A, **5** was obtained in 52% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 8.63-8.52 (m, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 7.97-7.83 (m, 2H), 7.60-7.36 (m, 1H), 4.35 (p, *J* = 8.6 Hz, 1H), 2.67-2.57 (m, 2H), 2.50 (m, 2H), 2.24-2.14 (m, 1H), 1.96 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 163.99, 143.29, 135.42, 133.57, 127.60, 127.20, 125.05, 122.49, 117.96, 39.55, 27.93, 18.64.

1-cyclohexyl-6-methylisoquinoline (6)¹⁰



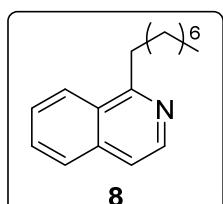
Following the general procedure A, **6** was obtained in 80% yield (18 mg). Eluent (petroleum ether: ethyl acetate, 10:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 8.43 (d, *J* = 5.7 Hz, 1H), 8.11 (d, *J* = 8.7 Hz, 1H), 7.57 (s, 1H), 7.42-7.37 (m, 2H), 3.52 (tt, *J* = 11.7, 3.2 Hz, 1H), 2.53 (s, 3H), 1.99-1.90 (m, 4H), 1.86-1.76 (m, 3H), 1.53 (qt, *J* = 12.8, 3.7 Hz, 2H), 1.40 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 165.51, 142.17, 139.83, 136.83, 129.13, 126.59, 124.78, 124.71, 118.56, 41.63, 32.70, 27.03, 26.39, 21.91.

6-bromo-1-cyclohexylisoquinoline (7)¹⁰



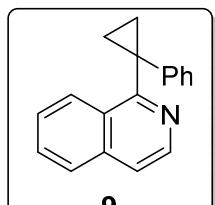
Following the general procedure A, **7** was obtained in 73% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 10:1). white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.49 (d, *J* = 5.7 Hz, 1H), 8.08 (d, *J* = 9.1 Hz, 1H), 7.97 (d, *J* = 2.0 Hz, 1H), 7.64 (dd, *J* = 9.0, 2.0 Hz, 1H), 7.38 (d, *J* = 5.7 Hz, 1H), 3.49 (tt, *J* = 11.7, 3.2 Hz, 1H), 1.98-1.89 (m, 4H), 1.86-1.75 (m, 3H), 1.51 (m, 2H), 1.39 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.11, 143.18, 137.72, 130.41, 129.76, 126.72, 124.81, 124.37, 117.95, 41.77, 32.71, 26.94, 26.31.

1-octylisoquinoline (8)¹²



Following the general procedure A, **8** was obtained in 54% yield (13 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 8.43 (d, *J* = 5.7 Hz, 1H), 8.18-8.13 (m, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.65 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H), 7.58 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.49 (d, *J* = 5.7 Hz, 1H), 3.31-3.26 (m, 2H), 1.89-1.82 (m, 2H), 1.52-1.24 (m, 10H), 0.90-0.85 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 162.60, 142.05, 136.38, 129.85, 127.50, 127.04, 127.03, 125.51, 119.23, 35.76, 31.95, 30.03, 29.99, 29.36, 22.80, 14.24.

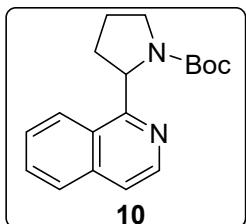
1-(1-phenylcyclopropyl) isoquinoline (9)¹⁰



Following the general procedure A, **9** was obtained in 71% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.54 (d, *J* = 5.7 Hz, 1H), 8.31 (d, *J* = 8.5 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.63-7.58 (m, 2H), 7.47 (ddd, *J* = 8.2, 7.0, 1.1 Hz, 1H), 7.21-7.17 (m, 2H), 7.10 (m, 3H), 1.60

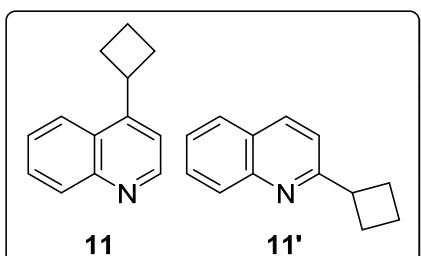
(m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.44, 144.68, 142.00, 136.85, 129.84, 128.39, 127.99, 127.36, 127.06, 127.03, 126.09, 125.72, 120.33, 30.09, 16.75.

tert-butyl 2-(isoquinolin-1-yl)pyrrolidine-1-carboxylate (10**)¹³**



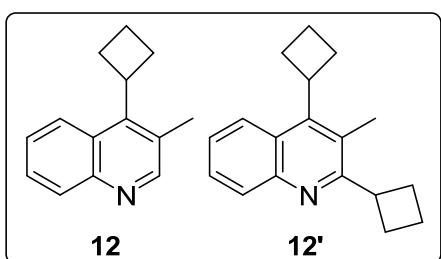
Following the general procedure A, **10** was obtained in 67% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 8.44 (m, 1H), 8.23-8.15 (m, 1H), 7.81 (m, 1H), 7.68-7.48 (m, 3H), 5.88-5.58 (m, 1H), 3.86 (m, 1H), 3.76-3.57 (m, 1H), 2.48 (m, 1H), 2.11-1.92 (m, 3H), 1.44 (s, 3H), 0.91 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.23, 161.85, 154.48, 154.46, 141.91, 136.33, 129.75, 127.60, 127.03, 124.39, 124.15, 119.84, 119.63, 79.28, 78.84, 59.59, 58.75, 47.45, 47.18, 34.02, 33.01, 28.64, 28.04, 24.17, 23.89.

4-cyclobutylquinoline:2-cyclobutylquinoline (11**, **11'**)¹¹**



Following the general procedure A, **11** and **11'** was obtained in 71% yield (13 mg, **11:11'**=7:4). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. ^1H NMR (500 MHz, CDCl_3) δ 8.07 (dd, J = 8.2, 5.8 Hz, 2H), 7.86 (d, J = 8.3 Hz, 1H), 7.77 (d, J = 8.0 Hz, 1H), 7.70-7.62 (m, 2H), 7.49-7.43 (m, 2H), 7.35 (d, J = 8.5 Hz, 1H), 7.20 (s, 1H), 4.12 (p, J = 8.8 Hz, 1H), 3.87 (h, J = 8.6 Hz, 2H), 2.58-2.52 (m, 2H), 2.51-2.44 (m, 6H), 2.32 (pd, J = 9.2, 2.4 Hz, 2H), 2.24-2.17 (m, 1H), 2.17-2.08 (m, 2H), 2.00-1.91 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 165.21, 164.90, 151.17, 148.01, 147.94, 136.28, 129.83, 129.37, 129.19, 128.84, 127.57, 126.93, 125.75, 125.72, 125.29, 123.93, 119.72, 116.08, 43.11, 42.92, 37.49, 28.93, 28.41, 18.81, 18.52.

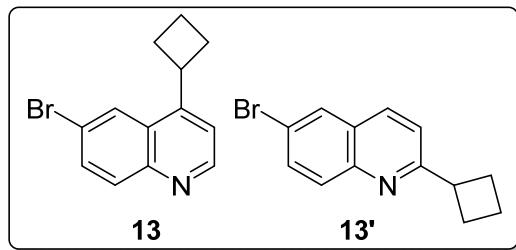
4-cyclobutyl-3-methylquinoline:2,4-dicyclobutyl-3-methylquinoline (12**, **12'**)**



Following the general procedure A, **12** and **12'** was obtained in 60% yield (14 mg, **12:12'**=2:5). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 8.09-8.03 (m, 1H), 8.00-7.96 (m, 1H), 7.58 (m, 1H), 7.41 (m, 1H), 4.26 (p, J = 9.2 Hz, 1H), 3.98-3.86 (m, 1H), 2.72 (m, 2H), 2.61 (m, 3H),

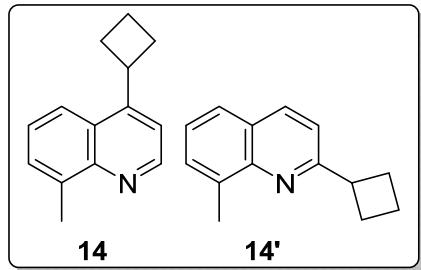
2.52-2.45 (m, 2H), 2.41 (qd, $J = 5.9, 3.6$ Hz, 4H), 2.28 (s, 3H), 2.18-2.05 (m, 3H), 1.96-1.87 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.38, 147.99, 146.56, 135.43, 129.95, 129.02, 128.19, 127.29, 126.91, 126.69, 126.30, 125.67, 124.49, 124.45, 41.04, 40.52, 38.76, 31.98, 27.14, 26.86, 19.30, 18.99, 18.22, 18.15, 16.33.

6-bromo-4-cyclobutylquinoline:6-bromo-2-cyclobutylquinoline (**13**, **13'**)



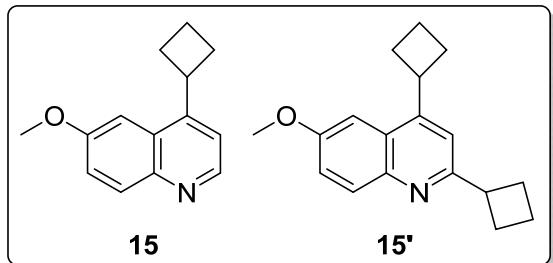
Following the general procedure A, **13** and **13'** was obtained in 68% yield (18 mg, **13:13'**=1.78:1). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 7.99-7.96 (m, 2H), 7.94-7.90 (m, 2H), 7.71 (m, 2H), 7.35 (d, $J = 8.5$ Hz, 1H), 7.19 (s, 1H), 4.04 (p, $J = 9.1$ Hz, 1H), 3.83 (h, $J = 8.4$ Hz, 2H), 2.54 (m, 2H), 2.49-2.42 (m, 7H), 2.30 (pd, $J = 8.8, 8.3, 2.1$ Hz, 2H), 2.25-2.07 (m, 3H), 1.95 (m, 3H), 1.37-1.22 (m, 1H), 0.87 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 165.69, 165.39, 150.41, 146.66, 146.55, 135.25, 132.76, 132.19, 131.57, 130.97, 129.60, 128.06, 127.00, 126.39, 120.68, 119.42, 119.22, 116.98, 43.01, 42.83, 37.23, 28.90, 28.30, 28.29, 18.77, 18.48, 18.47.

4-cyclobutyl-8-methylquinoline:2-cyclobutyl-8-methylquinoline(**14**, **14'**)



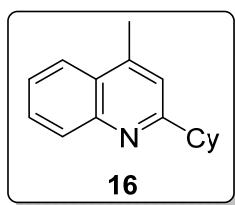
Following the general procedure A, **14** and **14'** was obtained in 66% yield (13 mg, **14:14'**=1.85: 1). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 8.00 (d, $J = 8.4$ Hz, 1H), 7.70 (d, $J = 8.3$ Hz, 1H), 7.59 (d, $J = 8.1$ Hz, 1H), 7.50 (dd, $J = 14.0, 7.0$ Hz, 2H), 7.37-7.30 (m, 2H), 7.26 (d, $J = 15.5$ Hz, 1H), 7.12 (d, $J = 1.0$ Hz, 1H), 4.10 (p, $J = 8.8$ Hz, 1H), 3.84 (h, $J = 8.4$ Hz, 2H), 2.83 (s, 5H), 2.57-2.48 (m, 6H), 2.42 (m, 4H), 2.29 (pd, $J = 9.1, 2.4$ Hz, 2H), 2.21-2.05 (m, 3H), 2.02-1.88 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.78, 163.37, 150.97, 146.93, 146.85, 137.71, 137.16, 136.26, 129.37, 128.95, 126.74, 125.49, 125.36, 124.80, 121.78, 119.70, 116.10, 43.25, 43.06, 37.66, 29.01, 28.37, 18.78, 18.54, 18.53, 17.92.

4-cyclobutyl-6-methoxyquinoline:2,4-dicyclobutyl-6-methoxyquinoline (15, 15')



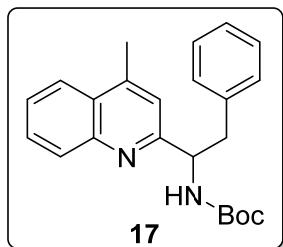
Following the general procedure A, **15** and **15'** was obtained in 43% yield (11 mg, **15:15'**=1:5). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ¹H NMR (500 MHz, CDCl₃) δ 7.97 (m, 1H), 7.34-7.28 (m, 1H), 7.16 (s, 1H), 7.11 (d, *J* = 2.7 Hz, 1H), 4.05 (p, *J* = 8.7 Hz, 1H), 3.92 (s, 3H), 3.83 (m 1H), 2.55 (m, 2H), 2.49-2.40 (m, 4H), 2.32 (pd, *J* = 9.2, 2.3 Hz, 2H), 2.24-2.15 (m, 1H), 2.14-2.06 (m, 1H), 1.99-1.90 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 162.75, 162.46, 157.29, 156.92, 149.78, 143.84, 135.15, 131.19, 130.58, 127.73, 126.41, 121.81, 120.57, 119.93, 116.28, 105.33, 102.77, 55.57, 42.95, 42.75, 37.62, 28.71, 28.51, 18.78, 18.50, 18.48.

2-cyclohexyl-4-methylquinoline (16)⁹



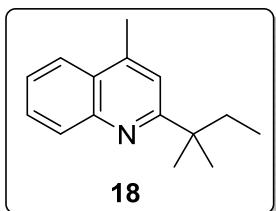
Following the general procedure A, **16** was obtained in 89% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 20:1). Colorless oil. ¹H NMR (500 MHz, CDCl₃) δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 1H), 7.68-7.64 (m, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.16 (s, 1H), 2.87 (tt, *J* = 12.1, 3.4 Hz, 1H), 2.68 (s, 3H), 2.04-1.99 (m, 2H), 1.89 (dt, *J* = 12.9, 3.0 Hz, 2H), 1.79 (d, *J* = 14.0 Hz, 1H), 1.63 (qd, *J* = 12.5, 3.1 Hz, 2H), 1.47 (m, 2H), 1.35 (m, *J* = 12.8, 3.5 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 166.65, 147.79, 144.30, 129.66, 129.03, 127.17, 125.47, 123.68, 120.37, 47.76, 32.97, 26.71, 26.28, 18.97.

tert-butyl (1-(4-methylquinolin-2-yl)-2-phenylethyl)carbamate (17)¹⁴



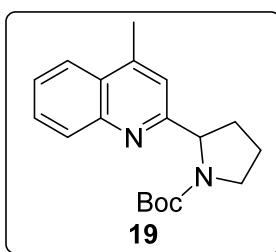
Following the general procedure A, **17** was obtained in 94% yield (34 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, *J* = 8.0 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.72-7.66 (m, 1H), 7.56-7.51 (m, 1H), 7.17 (s, 3H), 7.01 (d, *J* = 4.6 Hz, 2H), 6.81 (s, 1H), 6.16 (d, *J* = 7.2 Hz, 1H), 5.11 (q, *J* = 7.2 Hz, 1H), 3.31 (dd, *J* = 13.2, 5.6 Hz, 1H), 3.17 (dd, *J* = 13.2, 7.7 Hz, 1H), 2.58 (s, 3H), 1.46 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 159.47, 155.44, 147.43, 144.30, 137.65, 129.79, 129.20, 128.20, 127.45, 126.41, 126.08, 123.80, 121.38, 79.34, 57.06, 42.83, 28.56, 18.73.

4-methyl-2-(*tert*-pentyl)quinoline (18**)¹⁰**



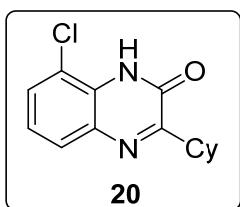
Following the general procedure A, **18** was obtained in 80% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 8.3 Hz, 1H), 7.96-7.92 (m, 1H), 7.69-7.63 (m, 1H), 7.53-7.46 (m, 1H), 7.30 (s, 1H), 2.69 (s, 3H), 1.85 (q, *J* = 7.5 Hz, 2H), 1.43 (s, 6H), 0.74 (t, *J* = 7.5 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 168.18, 147.49, 143.48, 130.10, 128.74, 126.61, 125.48, 123.53, 119.53, 41.32, 35.98, 27.48, 19.13, 9.39.

***tert*-butyl 2-(4-methylquinolin-2-yl)pyrrolidine-1-carboxylate (**19**)¹⁰**



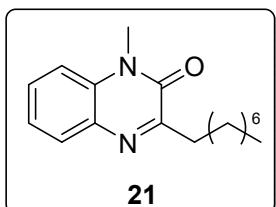
Following the general procedure A, **19** was obtained in 80% yield (28 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, *J* = 8.4 Hz, 1H), 7.93 (dd, *J* = 16.9, 8.3 Hz, 1H), 7.65 (m, 1H), 7.49 (m, 1H), 7.15-7.12 (m, 1H), 5.14-4.95 (m, 1H), 3.71 (t, *J* = 6.7 Hz, 2H), 2.67 (s, 3H), 2.43 (m, 1H), 2.05-1.87 (m, 3H), 1.46 (s, 3H), 1.10 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 163.98, 154.80, 147.43, 144.66, 134.21, 129.53, 129.25, 127.14, 125.78, 123.71, 118.32, 79.46, 63.67, 47.37, 34.69, 28.21, 23.71, 18.93.

8-chloro-3-cyclohexylquinoxalin-2(1*H*)-one (20**)**



Following the general procedure A, **20** was obtained in 88% yield (23 mg). Eluent (petroleum ether: ethyl acetate, 5:1). White solid. ¹H NMR (400 MHz, DMSO-d₆) δ 11.91 (s, 1H), 7.69 (d, *J* = 7.9 Hz, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.28 (t, *J* = 8.0 Hz, 1H), 3.22-3.14 (m, 1H), 1.84 (dd, *J* = 28.2, 11.8 Hz, 4H), 1.71 (d, *J* = 12.5 Hz, 1H), 1.50-1.31 (m, 4H), 1.27-1.19 (m, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 166.64, 154.18, 133.53, 129.04, 127.94, 127.86, 123.71, 118.50, 40.39, 30.57, 26.36, 26.23. HR-MS (ESI)[M+H]⁺ m/z calcd for C₁₄H₁₆N₂OCl 263.0946, found 263.0933.

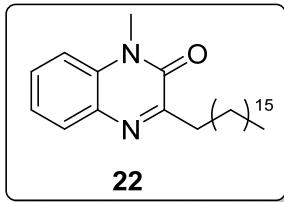
1-methyl-3-octylquinoxalin-2(1*H*)-one (21**)**



Following the general procedure B, **21** was obtained in 92% yield (25 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.52 (ddd, *J* = 8.6, 7.4, 1.5 Hz, 1H),

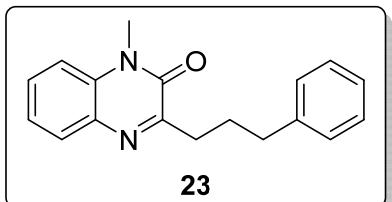
7.36-7.28 (m, 2H), 3.70 (s, 3H), 2.96-2.91 (m, 2H), 1.82-1.74 (m, 2H), 1.48-1.21 (m, 10H), 0.89-0.86 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.56, 155.06, 133.23, 132.86, 129.73, 129.62, 123.66, 113.68, 34.56, 31.92, 29.72, 29.30, 29.17, 27.03, 22.79, 14.24. HR-MS (ESI)[M+H] $^+$ m/z calcd for $\text{C}_{17}\text{H}_{25}\text{N}_2\text{O}$ 273.1961, found 273.1954.

3-heptadecyl-1-methylquinoxalin-2(1*H*)-one (**22**)



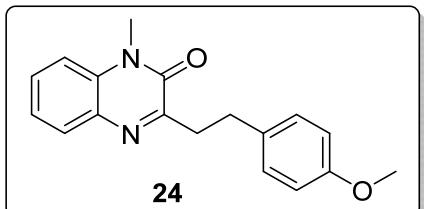
Following the general procedure B, **22** was obtained in 82% yield (33 mg). Eluent (petroleum ether: ethyl acetate, 3:1). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.82 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.53-7.47 (m, 1H), 7.34-7.29 (m, 1H), 7.29-7.26 (m, 1H), 3.69 (s, 3H), 2.96-2.89 (m, 2H), 1.81-1.73 (m, 2H), 1.24 (s, 30H), 0.87 (t, $J = 6.7$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.51, 155.01, 133.21, 132.86, 129.72, 129.57, 123.60, 113.63, 34.53, 32.05, 29.83, 29.49, 29.13, 27.00, 22.82, 14.25. HR-MS (ESI)[M+H] $^+$ m/z calcd for $\text{C}_{26}\text{H}_{42}\text{N}_2\text{O}$ 399.3375, found 399.3374.

1-methyl-3-(3-phenylpropyl)quinoxalin-2(1*H*)-one (**23**)¹⁴



Following the general procedure B, **23** was obtained in 73% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 5:1). White solid. ^1H NMR (400 MHz, CDCl_3) δ 7.83 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.52 (ddd, $J = 8.6, 7.5, 1.5$ Hz, 1H), 7.36-7.31 (m, 1H), 7.29-7.24 (m, 5H), 7.16 (dt, $J = 6.9, 2.5$ Hz, 1H), 3.68 (s, 3H), 3.03-2.98 (m, 2H), 2.80-2.75 (m, 2H), 2.19-2.11 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.91, 154.99, 142.25, 134.39, 133.19, 132.82, 129.75, 128.66, 128.37, 125.83, 123.66, 113.66, 35.82, 33.99, 29.15, 28.41.

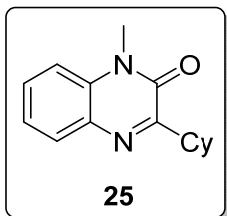
3-(4-methoxyphenethyl)-1-methylquinoxalin-2(1*H*)-one (**24**)



Following the general procedure B, **24** was obtained in 93% yield (27 mg). Eluent (petroleum ether: ethyl acetate, 5:1). White solid. ^1H NMR (400 MHz, CDCl_3) δ 7.92 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.62-7.57 (m, 1H), 7.44-7.39 (m, 1H), 7.38-7.34 (m, 1H), 7.30 (s, 2H), 6.92-6.88 (m, 2H), 3.85 (s, 3H), 3.77 (s, 3H), 3.33-3.28 (m, 2H), 3.14 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3)

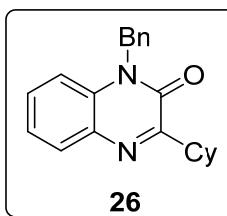
δ 160.25, 157.95, 154.95, 133.82, 133.23, 132.81, 129.79, 129.77, 129.62, 123.68, 113.87, 113.69, 55.36, 36.39, 31.81, 29.15. HR-MS (ESI)[M+H]⁺ m/z calcd for C₁₈H₁₉N₂O₂ 295.1441, found 295.1437.

3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one (**25**)¹⁶



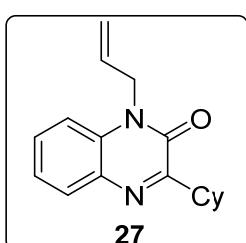
Following the general procedure A, **25** was obtained in 91% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.79 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.49-7.43 (m, 1H), 7.31-7.26 (m, 1H), 7.25-7.22 (m, 1H), 3.65 (s, 3H), 3.30 (tt, *J* = 11.5, 3.2 Hz, 1H), 1.95-1.80 (m, 4H), 1.73 (d, *J* = 12.6 Hz, 1H), 1.55 (m, 2H), 1.42 (tt, *J* = 12.7, 3.0 Hz, 2H), 1.27 (qt, *J* = 12.1, 3.2 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 164.36, 154.63, 132.99, 132.95, 129.86, 129.46, 123.47, 113.54, 40.87, 30.62, 29.14, 26.42, 26.26.

1-benzyl-3-cyclohexylquinoxalin-2(1*H*)-one (**26**)¹⁶



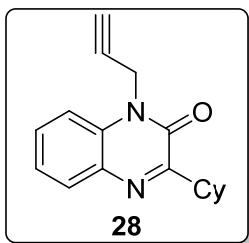
Following the general procedure A, **26** was obtained in 92% yield (29 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.38-7.34 (m, 1H), 7.32-7.26 (m, 3H), 7.25-7.20 (m, 4H), 5.48 (s, 2H), 3.39 (t, *J* = 11.5 Hz, 1H), 2.03-1.76 (m, 5H), 1.63-1.44 (m, 4H), 1.33 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 164.52, 154.71, 135.62, 133.29, 132.31, 130.01, 129.48, 129.01, 127.74, 127.05, 123.56, 114.36, 46.05, 40.95, 30.71, 26.47, 26.30.

1-allyl-3-cyclohexylquinoxalin-2(1*H*)-one (**27**)¹⁷



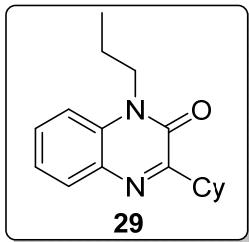
Following the general procedure A, **27** was obtained in 75% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.44 (ddd, *J* = 8.6, 7.4, 1.5 Hz, 1H), 7.30-7.22 (m, 2H), 5.91 (ddt, *J* = 17.3, 10.4, 5.2 Hz, 1H), 5.26-5.11 (m, 2H), 4.87 (dt, *J* = 5.2, 1.7 Hz, 2H), 3.33 (tt, *J* = 11.6, 3.3 Hz, 1H), 1.98-1.91 (m, 2H), 1.84 (dt, *J* = 12.6, 3.0 Hz, 2H), 1.78-1.71 (m, 1H), 1.55 (qd, *J* = 12.2, 2.5 Hz, 2H), 1.44 (qt, *J* = 12.8, 2.9 Hz, 2H), 1.29 (qt, *J* = 12.4, 3.3 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 164.45, 154.19, 133.20, 132.19, 130.95, 130.00, 129.40, 123.49, 118.11, 114.11, 44.66, 40.87, 30.67, 26.45, 26.29.

3-cyclohexyl-1-(prop-2-yn-1-yl)quinoxalin-2(1H)-one (28)¹⁷



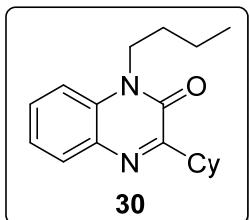
Following the general procedure A, **28** was obtained in 86% yield (23 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.84 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.53 (td, *J* = 7.9, 7.2, 1.4 Hz, 1H), 7.45-7.41 (m, 1H), 7.37-7.32 (m, 1H), 5.04 (d, *J* = 2.5 Hz, 2H), 3.33 (tt, *J* = 11.5, 3.2 Hz, 1H), 2.28 (t, *J* = 2.5 Hz, 1H), 1.96 (d, *J* = 11.1 Hz, 2H), 1.86 (dt, *J* = 12.6, 2.9 Hz, 2H), 1.79-1.73 (m, 1H), 1.57 (m, 2H), 1.45 (m, 2H), 1.31 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 164.30, 153.62, 133.21, 131.47, 130.05, 129.60, 123.90, 114.07, 73.15, 40.96, 31.58, 30.66, 26.42, 26.26.

3-cyclohexyl-1-propylquinoxalin-2(1H)-one (29)¹⁷



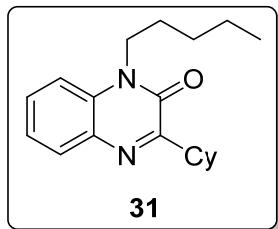
Following the general procedure A, **29** was obtained in 63% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.81 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.49-7.42 (m, 1H), 7.30-7.23 (m, 2H), 4.21-4.15 (m, 2H), 3.31 (tt, *J* = 11.5, 3.2 Hz, 1H), 1.93 (d, *J* = 11.6 Hz, 2H), 1.88-1.81 (m, 2H), 1.76 (m, 3H), 1.55 (m, 2H), 1.49-1.38 (m, 2H), 1.28 (m, 1H), 1.02 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 164.40, 154.37, 133.28, 132.16, 130.13, 129.37, 123.27, 113.60, 43.91, 40.86, 30.66, 26.47, 26.30, 20.78, 11.54.

1-butyl-3-cyclohexylquinoxalin-2(1H)-one (30)



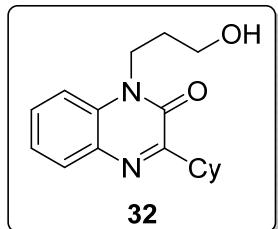
Following the general procedure A, **30** was obtained in 85% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.83-7.79 (m, 1H), 7.49-7.43 (m, 1H), 7.29-7.23 (m, 2H), 4.24-4.18 (m, 2H), 3.31 (tt, *J* = 11.5, 3.1 Hz, 1H), 1.93 (d, *J* = 12.7 Hz, 2H), 1.87-1.81 (m, 2H), 1.77-1.68 (m, 3H), 1.60-1.47 (m, 3H), 1.44 (m, 3H), 1.33-1.23 (m, 1H), 0.98 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 164.35, 154.33, 133.29, 132.13, 130.11, 129.37, 123.25, 113.56, 42.22, 40.84, 30.65, 29.45, 26.46, 26.29, 20.43, 13.89. HR-MS (ESI)[M+H]⁺ m/z calcd for C₁₈H₂₅N 285.1691, found 285.1953.

3-cyclohexyl-1-pentylquinoxalin-2(1*H*)-one (31**)**



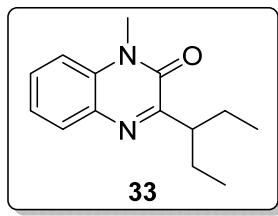
Following the general procedure A, **31** was obtained in 81% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.81 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.49-7.43 (m, 1H), 7.30-7.23 (m, 2H), 4.23-4.17 (m, 2H), 3.32 (tt, *J* = 11.5, 3.2 Hz, 1H), 1.94 (d, *J* = 11.3 Hz, 2H), 1.88-1.81 (m, 2H), 1.73 (m, 3H), 1.56 (m, 2H), 1.51-1.43 (m, 2H), 1.40 (m, 4H), 1.30 (m, 1H), 0.90 (t, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 164.35, 154.29, 133.28, 132.13, 130.10, 129.36, 123.22, 113.54, 42.43, 40.83, 30.64, 29.25, 27.07, 26.45, 26.28, 22.49, 14.08. HR-MS (ESI)[M+H]⁺ m/z calcd for C₁₉H₂₇N₂O 299.2118, found 299.2114.

3-cyclohexyl-1-(3-hydroxypropyl)quinoxalin-2(1*H*)-one (32**)**



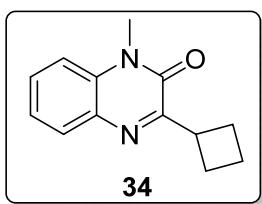
Following the general procedure A, **32** was obtained in 80% yield (23 mg). Eluent (petroleum ether: ethyl acetate, 5:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 7.87 (d, *J* = 8.0 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 4.44 (t, *J* = 6.1 Hz, 2H), 3.68 (t, *J* = 6.8 Hz, 1H), 3.52 (q, *J* = 5.9 Hz, 2H), 3.32 (m, 1H), 2.05-1.99 (m, 2H), 1.98-1.92 (m, 2H), 1.86 (d, *J* = 12.6 Hz, 2H), 1.76 (m, 1H), 1.56 (m, 2H), 1.51-1.40 (m, 2H), 1.35-1.27 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 163.89, 155.35, 133.62, 131.64, 130.32, 129.77, 123.95, 113.69, 58.23, 50.10, 40.88, 39.00, 30.61, 30.16, 26.41, 26.25. HR-MS (ESI)[M+H]⁺ m/z calcd for C₁₇H₂₃N₂O₂ 287.1754, found 287.1757.

1-methyl-3-(pentan-3-yl)quinoxalin-2(1*H*)-one (33**)¹⁷**



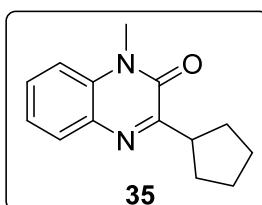
Following the general procedure B, **33** was obtained in 61% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (500 MHz, CDCl₃) δ 7.87-7.83 (m, 1H), 7.53-7.49 (m, 1H), 7.35-7.28 (m, 2H), 3.70 (s, 3H), 3.35 (m, 1H), 1.87 (m, 2H), 1.73-1.67 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 163.99, 155.23, 132.98, 132.97, 129.98, 129.56, 123.49, 113.59, 44.79, 29.26, 25.89, 12.12.

3-cyclobutyl-1-methylquinoxalin-2(1*H*)-one (34**)¹⁸**



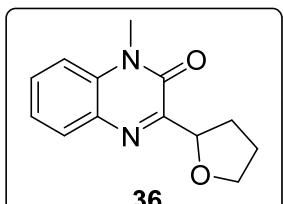
Following the general procedure B, **34** was obtained in 81% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (500 MHz, CDCl₃) δ 7.86 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.50 (m, 1H), 7.34-7.30 (m, 1H), 7.28-7.25 (m, 1H), 4.10-4.01 (m, 1H), 3.67 (s, 3H), 2.47-2.40 (m, 2H), 2.37 (m, 2H), 2.10 (m, 1H), 1.89 (m, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 162.38, 154.71, 133.11, 132.98, 129.88, 129.50, 123.55, 113.59, 38.48, 28.97, 26.45, 18.37.

3-cyclopentyl-1-methylquinoxalin-2(1*H*)-one (35**)¹⁶**



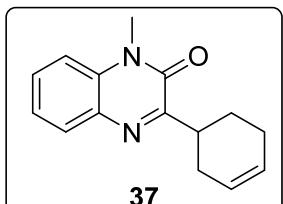
Following the general procedure B, **35** was obtained in 92% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (500 MHz, CDCl₃) δ 7.81 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.51-7.46 (m, 1H), 7.33-7.26 (m, 2H), 3.74-3.69 (m, 1H), 3.69 (s, 3H), 2.06 (m, 2H), 1.96-1.88 (m, 2H), 1.84-1.78 (m, 2H), 1.71 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 163.85, 155.11, 133.10, 132.85, 129.87, 129.41, 123.48, 113.53, 42.85, 30.97, 29.14, 26.07.

1-methyl-3-(tetrahydrofuran-2-yl)quinoxalin-2(1*H*)-one (36**)¹⁹**



Following the general procedure B, **36** was obtained in 91% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.94 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.53 (ddd, *J* = 8.6, 7.4, 1.5 Hz, 1H), 7.35-7.28 (m, 2H), 5.37 (m, 1H), 4.25-4.19 (m, 1H), 4.03-3.97 (m, 1H), 3.68 (s, 3H), 2.53-2.45 (m, 1H), 2.06-2.00 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 159.55, 154.17, 133.26, 132.60, 130.55, 130.26, 123.76, 113.64, 77.71, 69.25, 30.59, 28.93, 25.74.

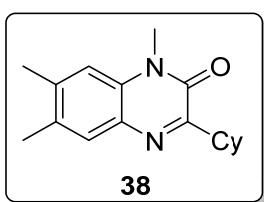
3-(cyclohex-3-en-1-yl)-1-methylquinoxalin-2(1*H*)-one (37**)²⁰**



Following the general procedure B, **37** was obtained in 91% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, Chloroform-d) δ 7.83 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.51 (ddd, *J* = 8.6, 7.4, 1.5 Hz, 1H), 7.35-7.27 (m, 2H), 5.83-5.73 (m, 2H), 3.70 (s, 3H), 3.58 (m, 1H),

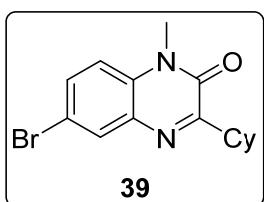
2.46 (m, 1H), 2.34-2.16 (m, 3H), 2.08-2.01 (m, 1H), 1.78 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.80, 154.72, 133.00, 132.94, 129.95, 129.65, 126.65, 126.43, 123.57, 113.61, 37.00, 29.19, 28.98, 26.98, 25.72.

3-cyclohexyl-1,6,7-trimethylquinoxalin-2(1*H*)-one (38**)¹⁶**



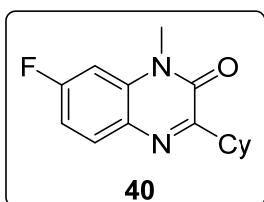
Following the general procedure A, **38** was obtained in 79% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.59 (s, 1H), 7.03 (s, 1H), 3.66 (s, 3H), 3.31 (tt, J = 11.5, 3.2 Hz, 1H), 2.39 (s, 3H), 2.33 (s, 3H), 1.93-1.70 (m, 5H), 1.57-1.43 (m, 4H), 1.29 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.14, 154.74, 139.09, 132.31, 131.40, 130.93, 130.01, 114.18, 40.76, 30.70, 29.08, 26.48, 26.31, 20.57, 19.21.

6-bromo-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one (39**)**



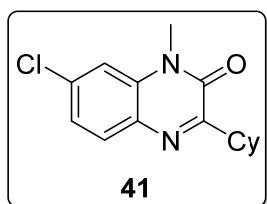
Following the general procedure A, **39** was obtained in 75% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ^1H NMR (500 MHz, CDCl_3) δ 7.66 (d, J = 8.7 Hz, 1H), 7.41 (m, 2H), 3.64 (s, 3H), 3.30 (tt, J = 11.5, 3.2 Hz, 1H), 1.95-1.83 (m, 4H), 1.76 (d, J = 12.9 Hz, 1H), 1.53 (m, 2H), 1.44 (m, 2H), 1.29 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 164.79, 154.29, 134.06, 131.87, 131.12, 126.70, 123.38, 116.61, 40.98, 30.58, 29.30, 26.39, 26.26. HR-MS (ESI)[M+H]⁺ m/z calcd for $\text{C}_{15}\text{H}_{18}\text{N}_2\text{OBr}$ 321.0597, found 321.0589.

3-cyclohexyl-7-fluoro-1-methylquinoxalin-2(1*H*)-one (40**)**



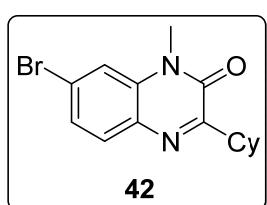
Following the general procedure A, **40** was obtained in 92% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.51 (d, J = 8.9 Hz, 1H), 7.22 (m, 2H), 3.67 (d, J = 3.0 Hz, 3H), 3.32 (m, 1H), 1.93 (d, J = 12.0 Hz, 2H), 1.88-1.81 (m, 2H), 1.78-1.72 (m, 1H), 1.55-1.41 (m, 4H), 1.29 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.95, 158.74 (d, J = 242.9 Hz), 154.28, 133.61 (d, J = 11.3 Hz), 129.63, 117.09 (d, J = 23.8 Hz), 115.33 (d, J = 22.3 Hz), 114.58 (d, J = 8.8 Hz), 40.98, 30.60, 29.42, 26.37, 26.24. HR-MS (ESI)[M+H]⁺ m/z calcd for $\text{C}_{15}\text{H}_{18}\text{N}_2\text{OF}$ 261.1398, found 261.1390.

7-chloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one (41**)¹⁷**



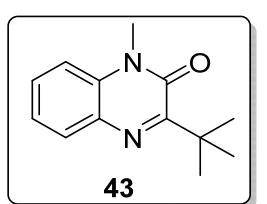
Following the general procedure A, **41** was obtained in 87% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (s, 1H), 7.44 (dd, J = 8.9, 2.4 Hz, 1H), 7.19 (d, J = 8.9 Hz, 1H), 3.67 (s, 3H), 3.32 (m, 1H), 1.96-1.90 (m, 2H), 1.89-1.82 (m, 2H), 1.76 (m, 1H), 1.50 (d, J = 8.9 Hz, 2H), 1.48-1.39 (m, 2H), 1.30 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 165.84, 154.32, 133.55, 131.69, 129.43, 129.28, 128.80, 114.70, 40.95, 30.60, 29.36, 26.37, 26.24.

7-bromo-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one (42**)**



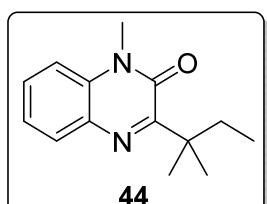
Following the general procedure A, **42** was obtained in 75% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.99 (m, 1H), 7.63-7.53 (m, 1H), 7.17-7.12 (m, 1H), 3.66 (s, 3H), 3.36-3.31 (m, 1H), 1.93 (d, J = 11.9 Hz, 2H), 1.86 (d, J = 12.3 Hz, 2H), 1.76 (d, J = 12.9 Hz, 1H), 1.50 (m, 4H), 1.32-1.26 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 165.84, 154.34, 133.89, 132.36, 132.21, 132.16, 116.08, 115.03, 40.96, 30.64, 29.35, 26.39, 26.26. HR-MS (ESI)[M+H]⁺ m/z calcd for C₁₅H₁₈N₂OBr 321.0597, found 321.0592.

3-(*tert*-butyl)-1-methylquinoxalin-2(1*H*)-one (43**)¹⁶**



Following the general procedure A, **43** was obtained in 65% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (dd, J = 8.0, 1.3 Hz, 1H), 7.52-7.47 (m, 1H), 7.31 (m, 1H), 7.26 (dd, J = 8.4, 1.0 Hz, 1H), 3.67 (s, 3H), 1.49 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 165.41, 153.86, 133.45, 132.31, 130.24, 129.63, 123.29, 113.37, 39.60, 28.88, 28.01.

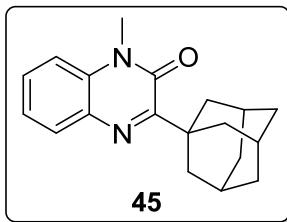
1-methyl-3-(*tert*-pentyl)quinoxalin-2(1*H*)-one (44**)²¹**



Following the general procedure A, **44** was obtained in 97% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.84 (dd, J = 8.0, 1.4 Hz, 1H), 7.50 (m, 1H), 7.33-7.29 (m, 1H), 7.28-7.25 (m, 1H), 3.67 (s, 3H), 2.04 (q, J = 7.5 Hz, 2H), 1.43 (s, 6H), 0.75 (t, J =

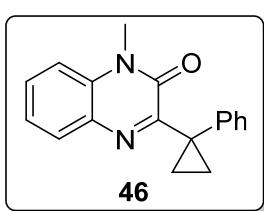
7.5 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.88, 153.89, 133.36, 132.36, 130.27, 129.59, 123.26, 113.38, 43.21, 32.47, 28.89, 25.98, 9.66.

3-((3*r*,5*r*,7*r*)-adamantan-1-yl)-1-methylquinoxalin-2(1*H*)-one (45)¹⁵



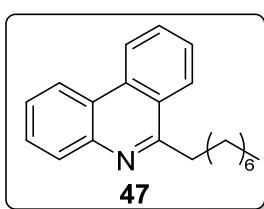
Following the general procedure A, **45** was obtained in 71% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.82 (dd, J = 8.0, 1.3 Hz, 1H), 7.52-7.46 (m, 1H), 7.33-7.28 (m, 1H), 7.26 (m, 1H), 3.66 (s, 3H), 2.24 (d, J = 2.7 Hz, 6H), 2.11 (s, 3H), 1.82 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.88, 153.76, 133.16, 132.54, 130.21, 129.59, 123.29, 113.36, 42.09, 38.95, 37.14, 28.78, 28.69.

1-methyl-3-(1-phenylcyclopropyl)quinoxalin-2(1*H*)-one (46)



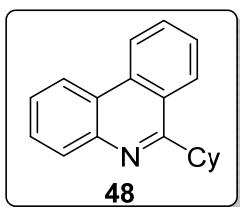
Following the general procedure A, **46** was obtained in 73% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.89 (dd, J = 8.0, 1.3 Hz, 1H), 7.51 (m, 1H), 7.48-7.45 (m, 2H), 7.35-7.31 (m, 1H), 7.28-7.24 (m, 3H), 7.20-7.15 (m, 1H), 3.61 (s, 3H), 1.50-1.47 (m, 2H), 1.38-1.35 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.49, 154.55, 141.98, 133.72, 132.59, 130.21, 130.08, 128.66, 128.26, 126.56, 123.55, 113.58, 30.83, 29.10, 13.81. HR-MS (ESI)[M+H]⁺ m/z calcd for $\text{C}_{18}\text{H}_{17}\text{N}_2$ 277.1335, found 277.1330.

6-octylphenanthridine (47)



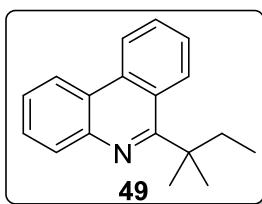
Following the general procedure A, **47** was obtained in 72% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 8.63 (d, J = 8.2 Hz, 1H), 8.55-8.52 (m, 1H), 8.25 (d, J = 8.1 Hz, 1H), 8.13 (dd, J = 8.2, 1.1 Hz, 1H), 7.82 (m, 1H), 7.73-7.66 (m, 2H), 7.61 (m, 1H), 3.39-3.34 (m, 2H), 1.92 (m, 2H), 1.58-1.36 (m, 5H), 1.31 (m, 5H), 0.89 (t, J = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.63, 143.88, 133.05, 130.35, 129.68, 128.67, 127.31, 126.48, 126.35, 125.34, 123.75, 122.58, 122.01, 36.67, 31.96, 30.13, 29.84, 29.38, 22.80, 14.24. HR-MS (ESI)[M+H]⁺ m/z calcd for $\text{C}_{21}\text{H}_{26}\text{N}$ 292.2060, found 292.2043.

6-cyclohexylphenanthridine (48)⁹



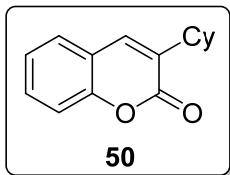
Following the general procedure A, **48** was obtained in 93% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 20:1). Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.66-8.63 (m, 1H), 8.54 (dd, J = 8.2, 1.3 Hz, 1H), 8.34-8.30 (m, 1H), 8.16 (dd, J = 8.2, 1.0 Hz, 1H), 7.81 (m, 1H), 7.70 (m, 2H), 7.61 (m, 1H), 3.63 (tt, J = 11.2, 3.2 Hz, 1H), 2.13-2.07 (m, 2H), 2.02-1.94 (m, 4H), 1.90-1.83 (m, 1H), 1.59 (m, 2H), 1.45 (qt, J = 12.9, 3.3 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 165.38, 144.00, 133.11, 130.05, 130.01, 128.48, 127.16, 126.22, 125.71, 124.83, 123.45, 122.68, 121.92, 42.11, 32.42, 27.01, 26.45.

6-(*tert*-pentyl)phenanthridine (49)²²



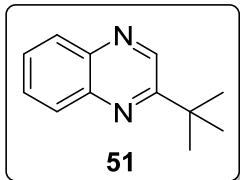
Following the general procedure A, **49** was obtained in 68% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 20:1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 8.70 (d, J = 8.0 Hz, 1H), 8.64 (d, J = 8.4 Hz, 1H), 8.55-8.51 (m, 1H), 8.14 (dd, J = 8.0, 1.2 Hz, 1H), 7.79 (m, 1H), 7.73-7.68 (m, 1H), 7.66-7.59 (m, 2H), 2.23 (m), 1.70 (d, J = 3.6 Hz, 6H), 0.75 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 165.82, 143.10, 133.92, 130.48, 129.37, 128.42, 127.77, 126.52, 126.16, 124.92, 123.44, 123.08, 121.72, 44.14, 35.65, 29.37, 9.69.

3-cyclohexyl-2*H*-chromen-2-one (50)¹⁰



Following the general procedure A, **50** was obtained in 61% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.48-7.42 (m, 3H), 7.30 (d, J = 8.1 Hz, 1H), 7.24 (m, 1H), 2.78 (m, 1H), 2.03-1.93 (m, 2H), 1.90-1.74 (m, 3H), 1.45 (qt, J = 12.7, 3.2 Hz, 2H), 1.28 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.66, 152.83, 136.42, 135.00, 130.53, 127.41, 124.27, 119.79, 116.41, 38.31, 32.24, 26.63, 26.29.

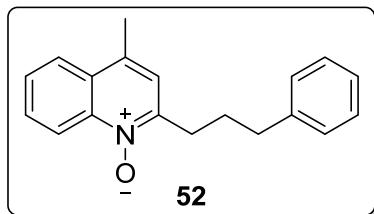
2-(*tert*-butyl)quinoxaline (51)²³



Following the general procedure A, **51** was obtained in 65% yield (12 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 8.99 (s, 1H), 8.09-8.03 (m, 2H), 7.75-7.67 (m, 2H), 1.51 (s, 9H). ¹³C NMR

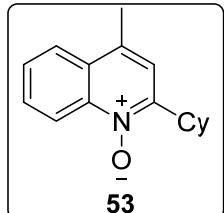
(101 MHz, CDCl₃) δ 163.83, 143.57, 141.75, 140.90, 129.78, 129.42, 129.02, 37.39, 29.89.

4-methyl-2-(3-phenylpropyl)quinoline 1-oxide (52)



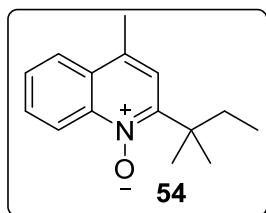
Following the general procedure C, **52** was obtained in 76% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 2:1). Pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 8.81 (d, J = 8.7, 1.2 Hz, 1H), 7.90 (d, J = 8.3, 1.3 Hz, 1H), 7.73 (m, 1H), 7.60 (m, 1H), 7.27 (d, J = 7.7 Hz, 1H), 7.25-7.20 (m, 3H), 7.18-7.13 (m, 1H), 7.06 (s, 1H), 3.16-3.10 (m, 2H), 2.78 (t, J = 7.7 Hz, 2H), 2.60 (s, 3H), 2.15 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 148.24, 141.85, 141.31, 133.56, 130.12, 128.69, 128.62, 128.50, 127.65, 126.05, 124.67, 122.65, 120.39, 35.97, 31.29, 27.89, 18.43. HR-MS (ESI)[M+H]⁺ m/z calcd for C₁₉H₂₀NO 278.1539, found 278.1544.

2-cyclohexyl-4-methylquinoline 1-oxide (53)²⁴



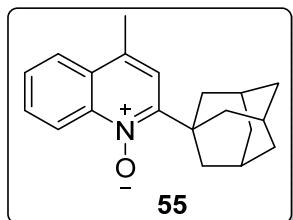
Following the general procedure C, **53** was obtained in 80% yield (19 mg). Eluent (petroleum ether: ethyl acetate, 4:1). Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.84 (d, J = 8.7 Hz, 1H), 7.90 (d, J = 8.3 Hz, 1H), 7.73 (m, 1H), 7.61-7.56 (m, 1H), 7.13 (s, 1H), 3.83 (m, 1H), 2.63 (s, 3H), 2.08 (d, J = 11.7 Hz, 2H), 1.84 (m, 3H), 1.55 (m, 2H), 1.46-1.29 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 152.37, 141.19, 133.79, 130.06, 128.27, 127.53, 124.53, 120.64, 120.06, 37.85, 30.71, 26.51, 26.40, 18.61.

4-methyl-2-(tert-pentyl)quinoline 1-oxide (54)²⁴



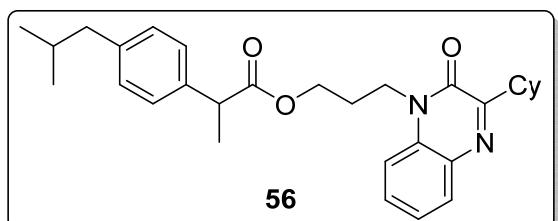
Following the general procedure C, **54** was obtained in 85% yield (19 mg). Eluent (petroleum ether: ethyl acetate, 4:1). Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.84 (d, J = 8.8 Hz, 1H), 7.91 (d, J = 8.3 Hz, 1H), 7.72 (t, J = 7.8 Hz, 1H), 7.60 (m, 1H), 7.22 (s, 1H), 2.64 (s, 3H), 2.31-2.21 (m, 2H), 1.59-1.52 (m, 6H), 0.70-0.63 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 152.53, 142.55, 132.73, 129.99, 128.68, 127.70, 124.47, 121.92, 120.55, 40.62, 30.48, 25.96, 18.75, 9.89.

2-((3*r*,5*r*,7*r*)-adamantan-1-yl)-4-methylquinoline 1-oxide (55)²⁴



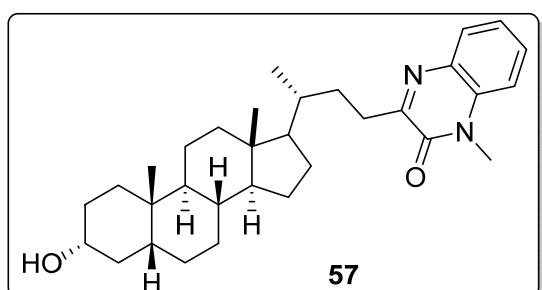
Following the general procedure C, **55** was obtained in 76% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 2:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 8.85 (d, *J* = 8.8 Hz, 1H), 7.91 (d, *J* = 8.2 Hz, 1H), 7.74 (t, *J* = 7.8 Hz, 1H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.22 (s, 1H), 2.64 (s, 3H), 2.44 (m, 6H), 2.15 (s, 3H), 1.90-1.78 (m, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 153.12, 142.84, 133.27, 130.08, 128.45, 127.67, 124.46, 120.79, 120.52, 39.02, 37.27, 37.06, 28.70, 18.79.

3-(3-cyclohexyl-2-oxoquinoxalin-1(2*H*)-yl)propyl 2-(4-isobutylphenyl)propanoate (56)



Following the general procedure A, **56** was obtained in 71% yield (34 mg). Eluent (petroleum ether: ethyl acetate, 3:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.39-7.34 (m, 1H), 7.30-7.23 (m, 3H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 1H), 4.23 (m, 1H), 4.19-4.15 (m, 2H), 3.73 (q, *J* = 7.1 Hz, 1H), 3.31 (tt, *J* = 11.5, 3.1 Hz, 1H), 2.41 (d, *J* = 7.2 Hz, 2H), 2.04 (m, 2H), 1.93 (d, *J* = 12.6 Hz, 2H), 1.89-1.83 (m, 2H), 1.80-1.74 (m, 2H), 1.58 (d, *J* = 12.3 Hz, 1H), 1.51 (d, *J* = 7.2 Hz, 3H), 1.43 (m, 2H), 1.36-1.20 (m, 2H), 0.83 (dd, *J* = 6.6, 1.5 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 174.65, 164.24, 154.27, 140.81, 137.80, 133.17, 131.94, 130.12, 129.57, 129.52, 127.27, 123.40, 113.25, 62.29, 45.26, 45.10, 40.75, 39.47, 30.63, 30.26, 26.54, 26.42, 26.24, 22.43, 18.43. HR-MS(ESI)[M+H]⁺ m/z calcd for C₃₀H₃₉N₂O₃ 475.2955, found 475.2930.

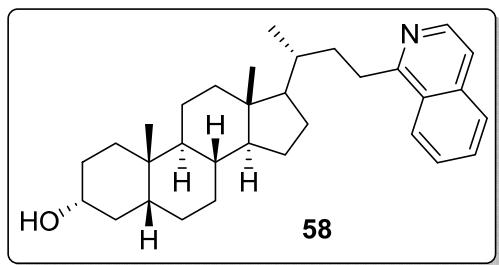
3-((3*R*)-3-((3*R*,5*R*,8*R*,9*S*,10*S*,13*R*,14*S*)-3-hydroxy-10,13-dimethylhexadecahydro-1*H*-cyclopenta[a]phenanthren-17-yl)butyl)-1-methylquinoxalin-2(1*H*)-one (57)



Following the general procedure B, **57** was obtained in 78% yield (38 mg). Eluent (petroleum ether: ethyl acetate, 2:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.53-7.47 (m, 1H), 7.34-7.30 (m, 1H), 7.28 (d, *J* = 8.4 Hz, 1H), 3.69 (s, 3H), 3.61 (m, 1H), 3.00 (m, 1H), 2.80 (m, 1H), 2.02-1.96 (m, 1H), 1.94-1.83

(m, 3H), 1.75 (m, 3H), 1.69-1.63 (m, 2H), 1.58-1.53 (m, 2H), 1.52-1.45 (m, 2H), 1.36 (m, 7H), 1.25-1.15 (m, 4H), 1.04 (d, J = 6.3 Hz, 6H), 0.91 (s, 3H), 0.65 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.03, 155.01, 133.19, 132.88, 129.68, 129.53, 123.60, 113.64, 71.97, 56.61, 56.16, 42.89, 42.24, 40.56, 40.30, 36.59, 36.04, 35.99, 35.49, 34.70, 33.02, 31.37, 30.69, 29.15, 28.38, 27.34, 26.56, 24.38, 23.52, 20.96, 18.75, 12.20. HR-MS (ESI)[M+H] $^+$ m/z calcd for $\text{C}_{32}\text{H}_{47}\text{N}_2\text{O}_2$ 491.3632, found 491.3622.

(3*R*,5*R*,8*R*,9*S*,10*S*,13*R*,14*S*)-17-((*R*)-4-(isoquinolin-1-yl)butan-2-yl)-10,13-dimethylhexadecahydro-1*H*-cyclopenta[a]phenanthren-3-ol (58)²⁵



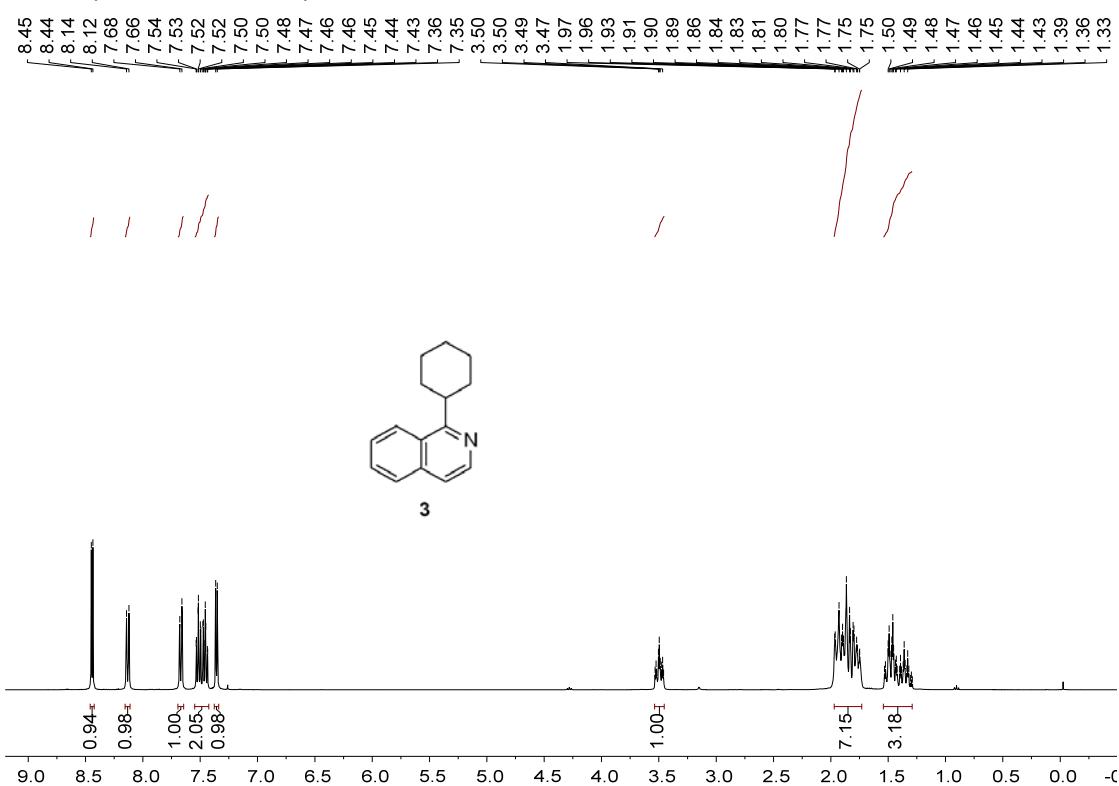
Following the general procedure A, **58** was obtained in 74% yield (34 mg). Eluent (petroleum ether: ethyl acetate, 2:1). White crystal. ^1H NMR (400 MHz, CDCl_3) δ 8.41 (d, J = 5.7 Hz, 1H), 8.13 (d, J = 8.3 Hz, 1H), 7.80 (d, J = 8.1 Hz, 1H), 7.62 (m, 2H), 7.48 (d, J = 5.7 Hz, 1H), 3.62 (m, 1H), 3.38 (td, J = 12.5, 11.7, 3.8 Hz, 1H), 3.19-3.09 (m, 1H), 2.01 (d, J = 10.6 Hz, 1H), 1.98-1.82 (m, 4H), 1.77 (s, 2H), 1.59 (m, 6H), 1.38 (m, 6H), 1.29-1.19 (m, 5H), 1.13 (s, 3H), 1.08-0.97 (m, 3H), 0.91 (s, 3H), 0.67 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.13, 141.99, 136.42, 129.89, 127.53, 127.08, 126.97, 125.46, 119.23, 71.93, 56.61, 56.16, 42.91, 42.23, 40.55, 40.33, 36.59, 36.56, 36.33, 35.97, 35.49, 34.70, 32.72, 30.69, 28.50, 27.33, 26.55, 24.38, 23.52, 20.97, 18.93, 12.19.

XII. References

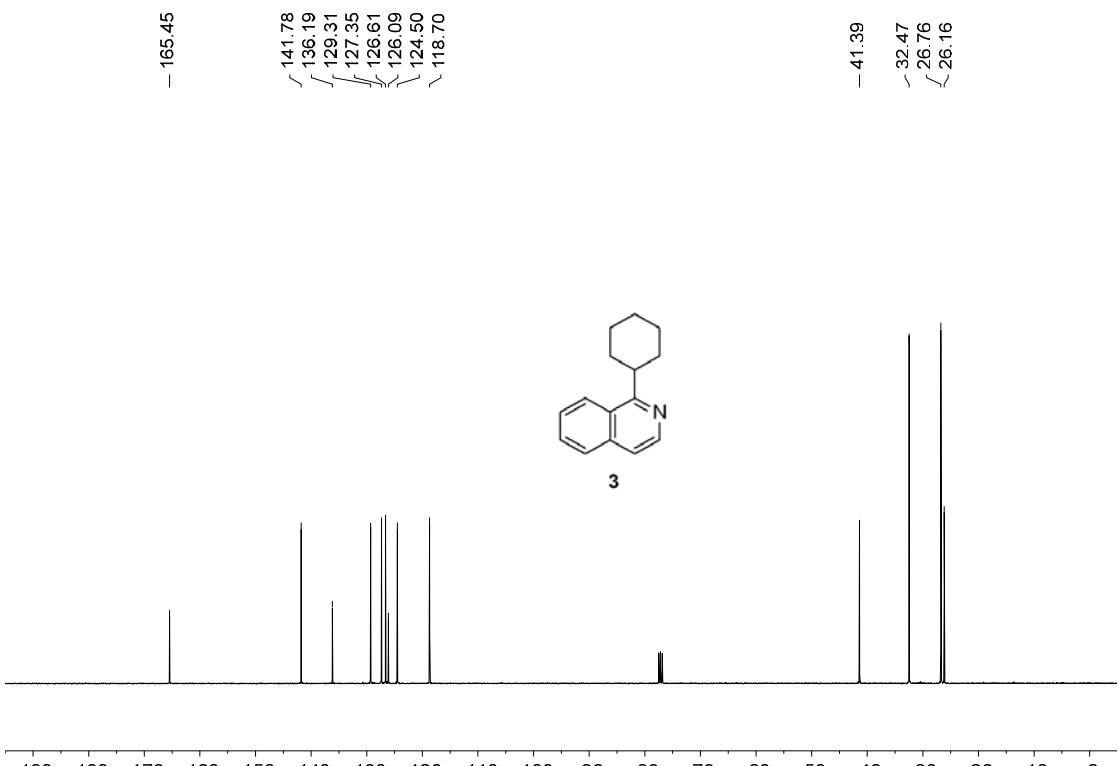
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XIII. The ^1H and ^{13}C NMR spectra of compounds 3-58.

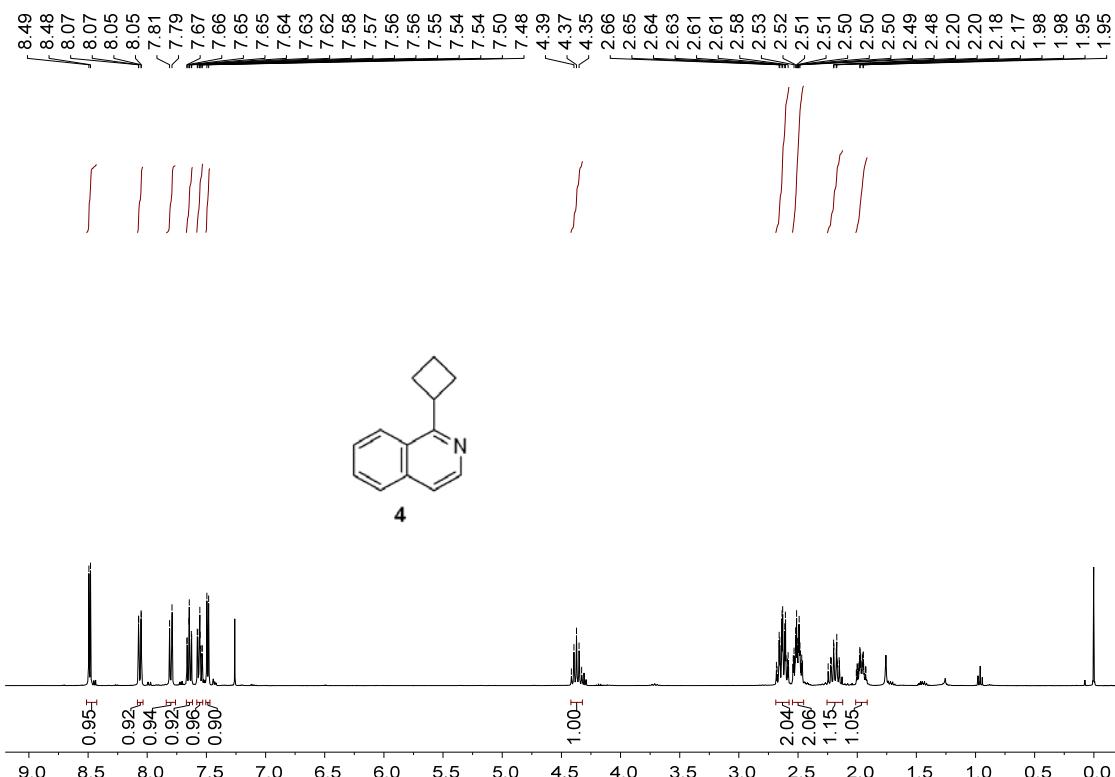
^1H NMR for **3** (400 MHz, CDCl_3)



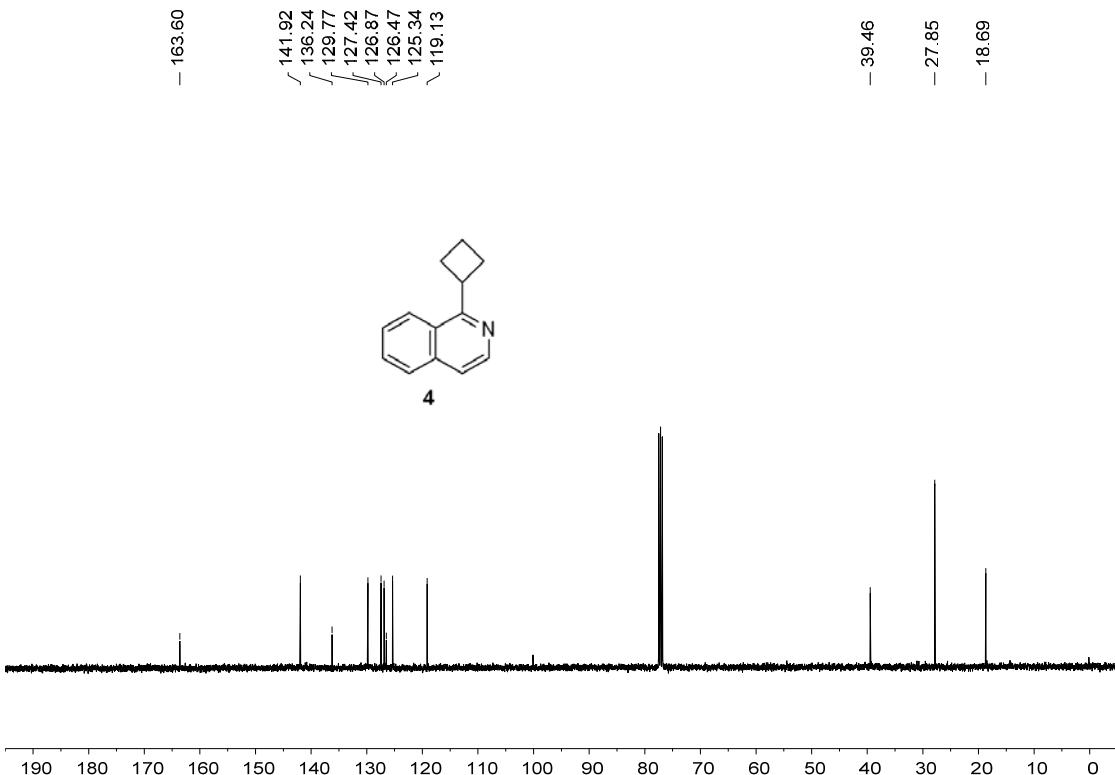
^{13}C NMR for **3** (101 MHz, CDCl_3)



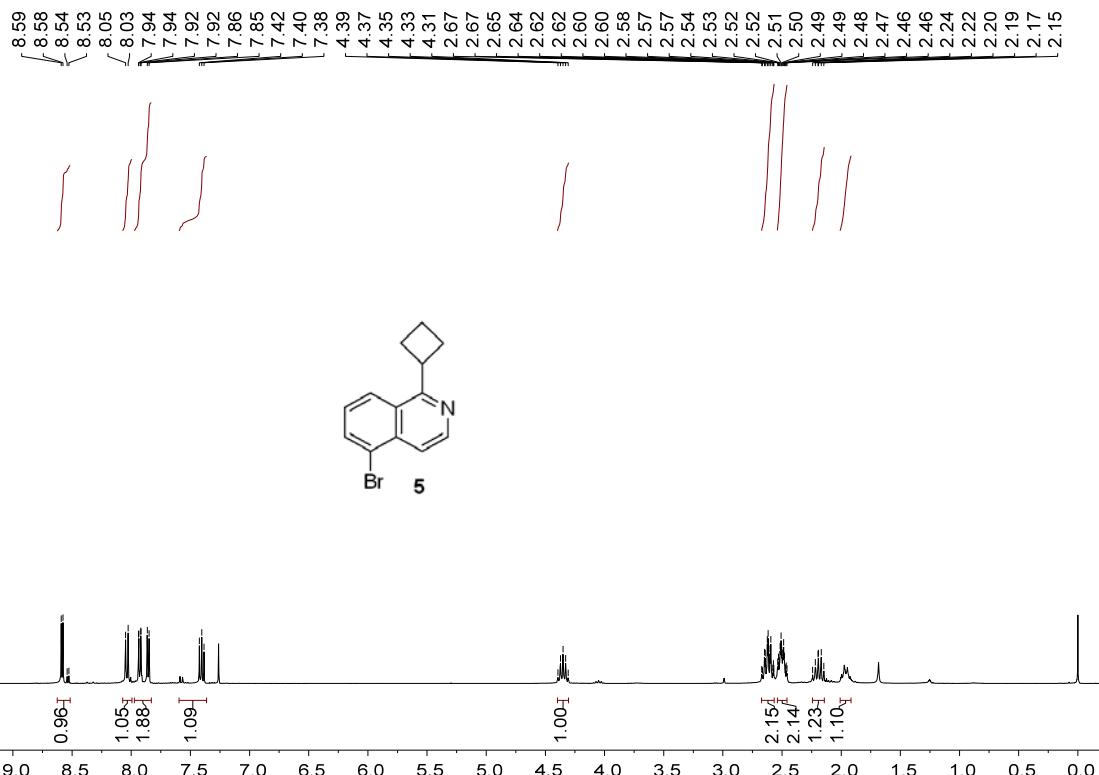
¹H NMR for **4** (400 MHz, CDCl₃)



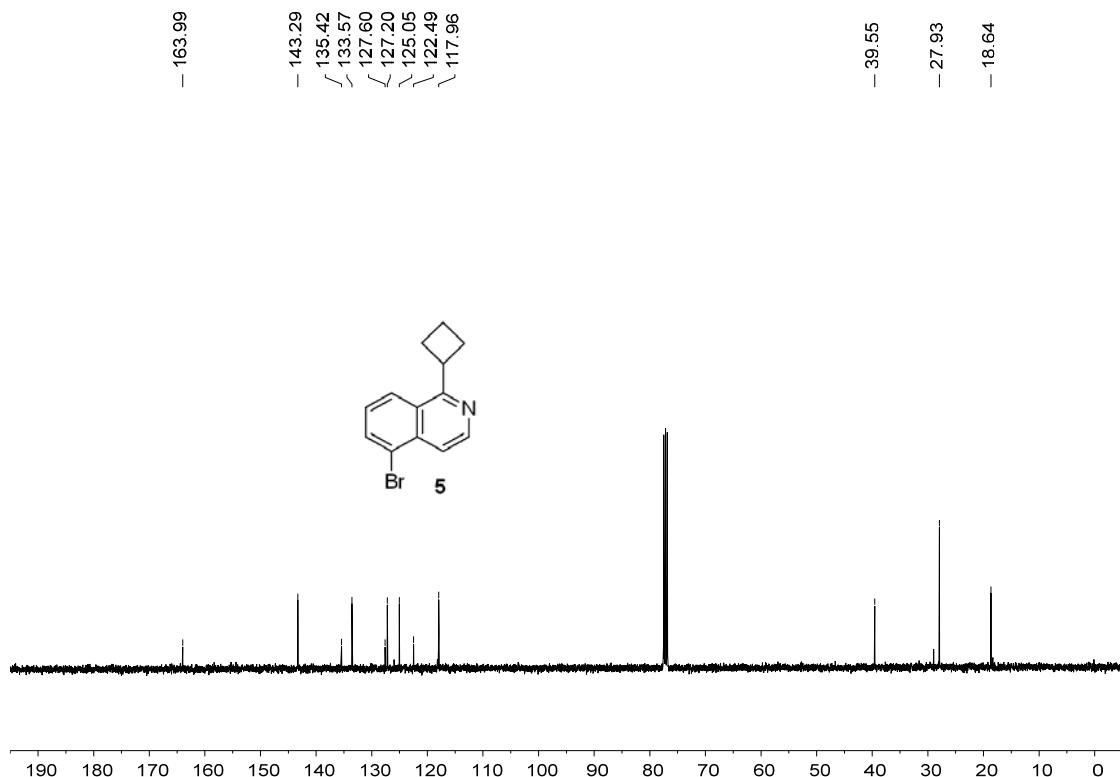
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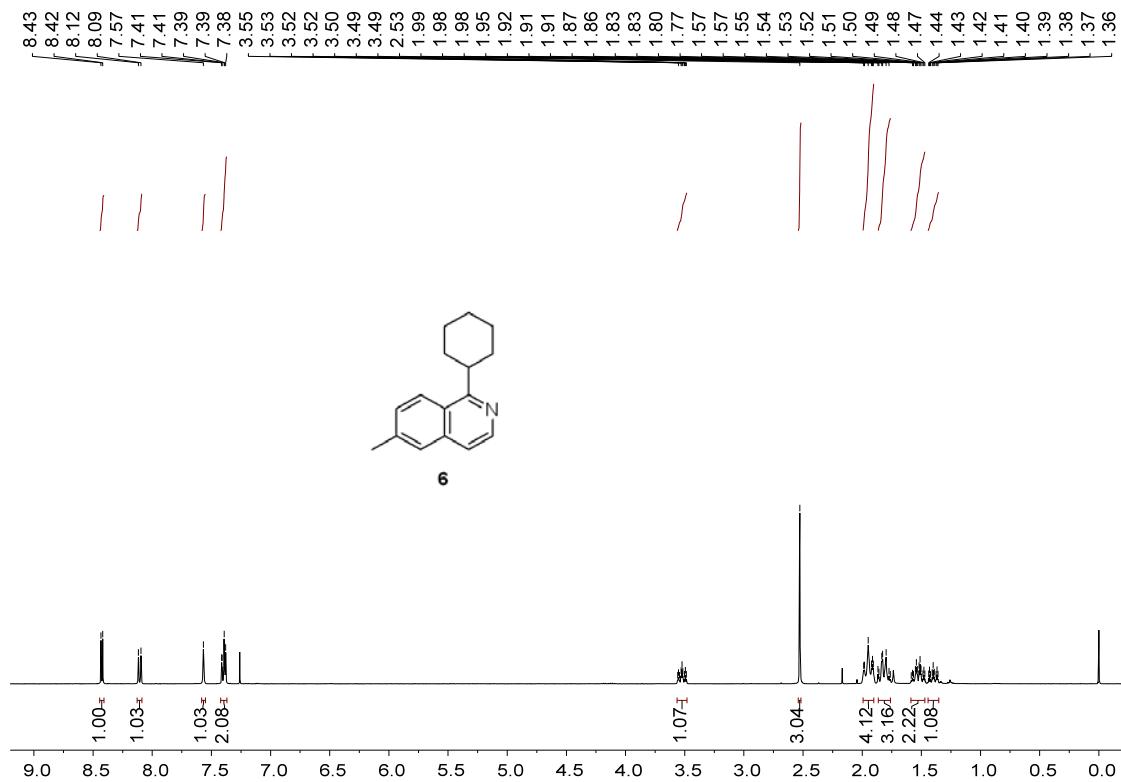
¹H NMR for **5** (400 MHz, CDCl₃)



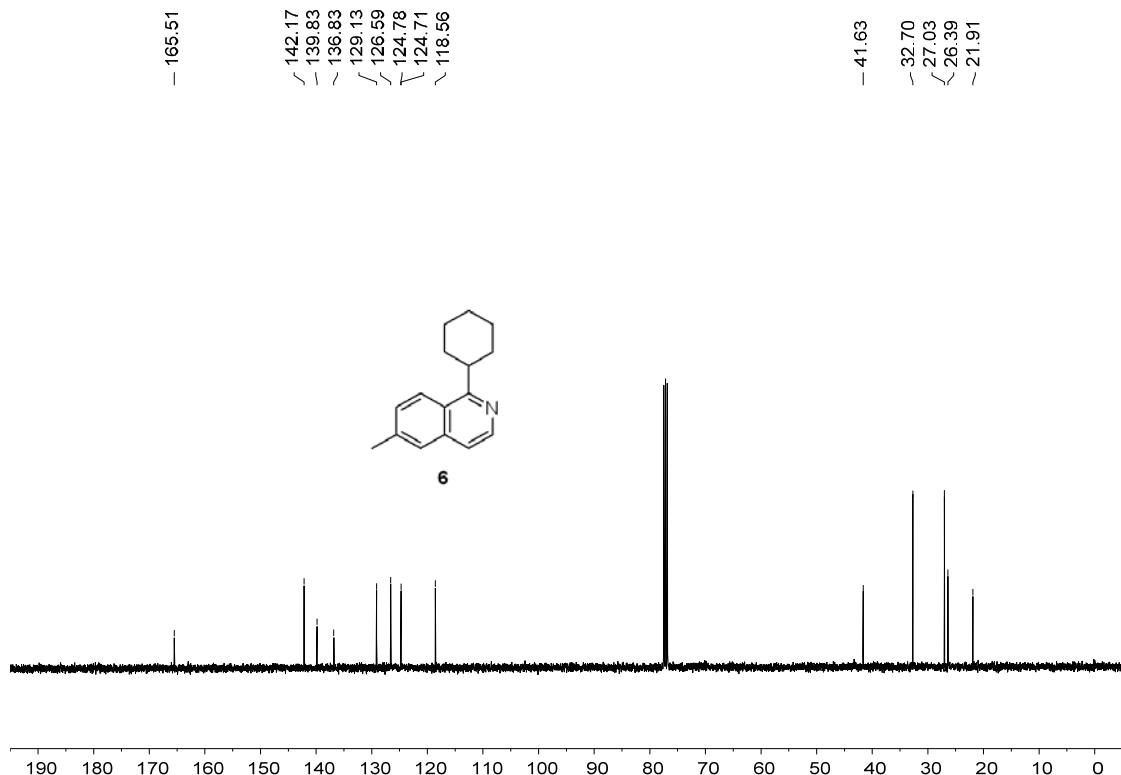
¹³C NMR for **5** (101 MHz, CDCl₃)



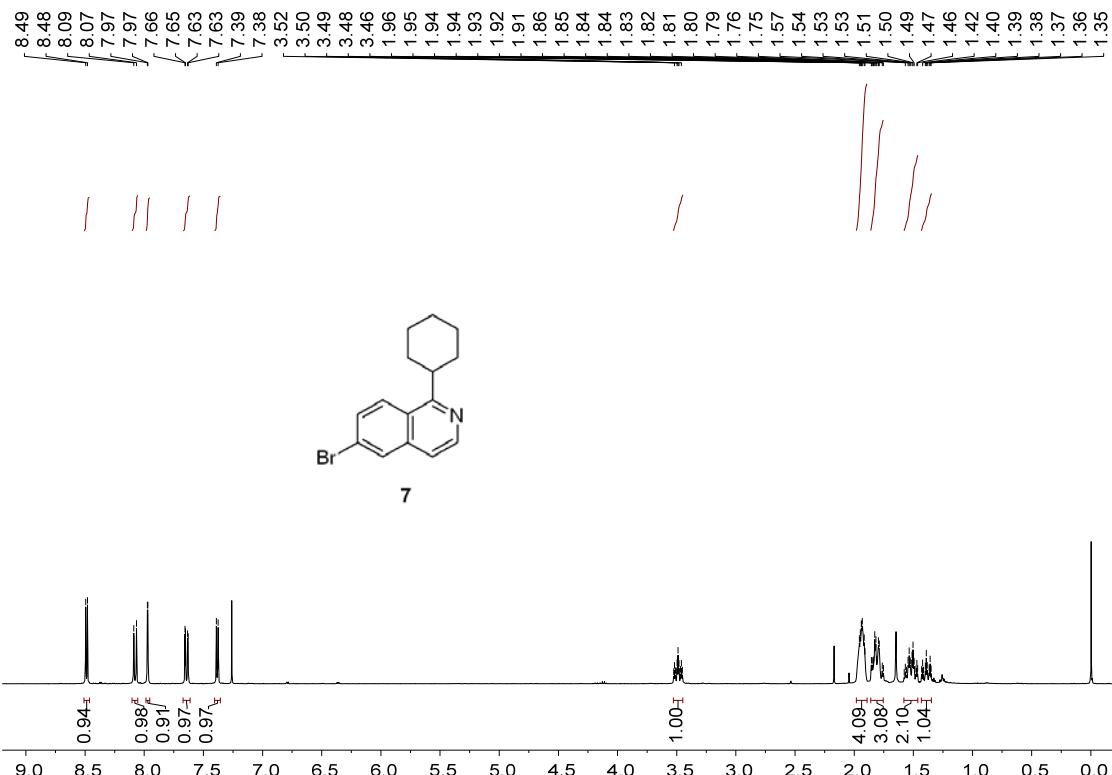
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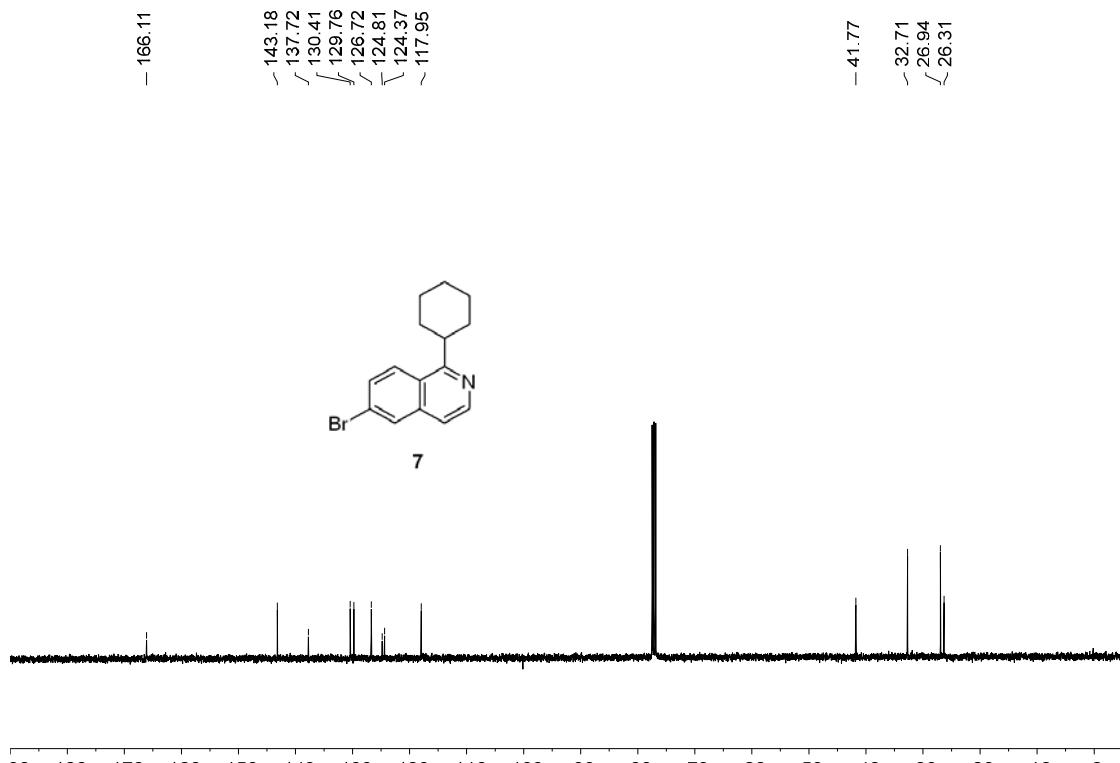
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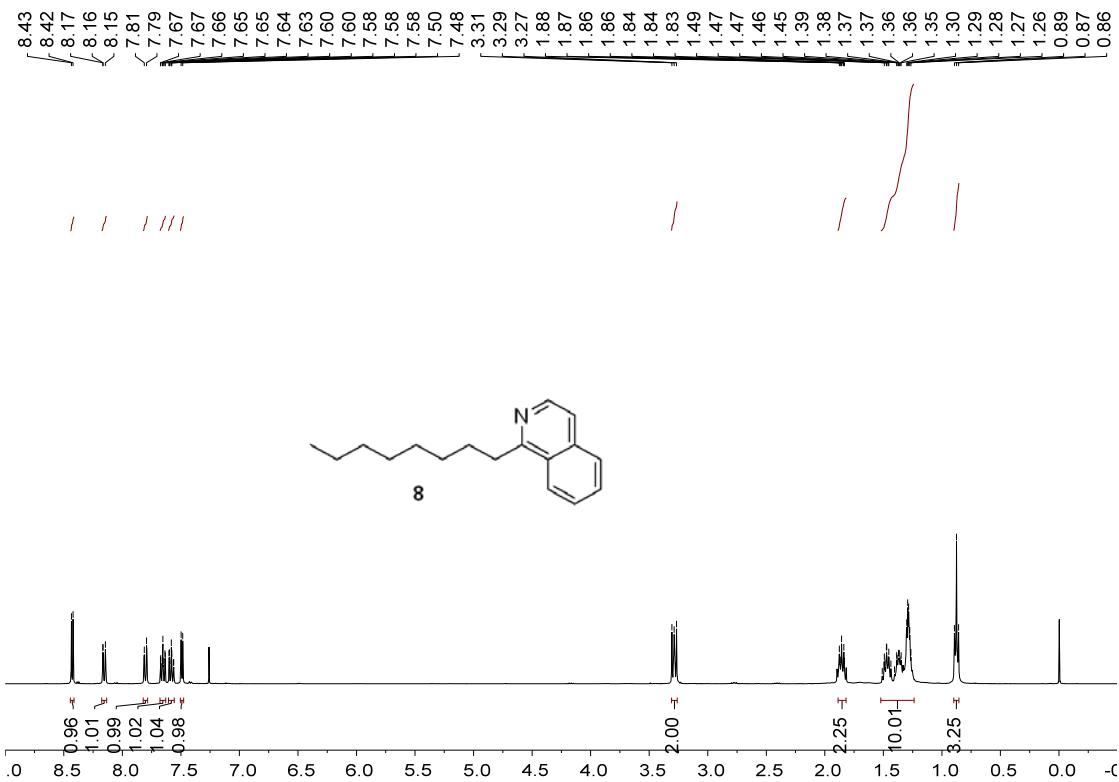
¹H NMR for **7** (400 MHz, CDCl₃)



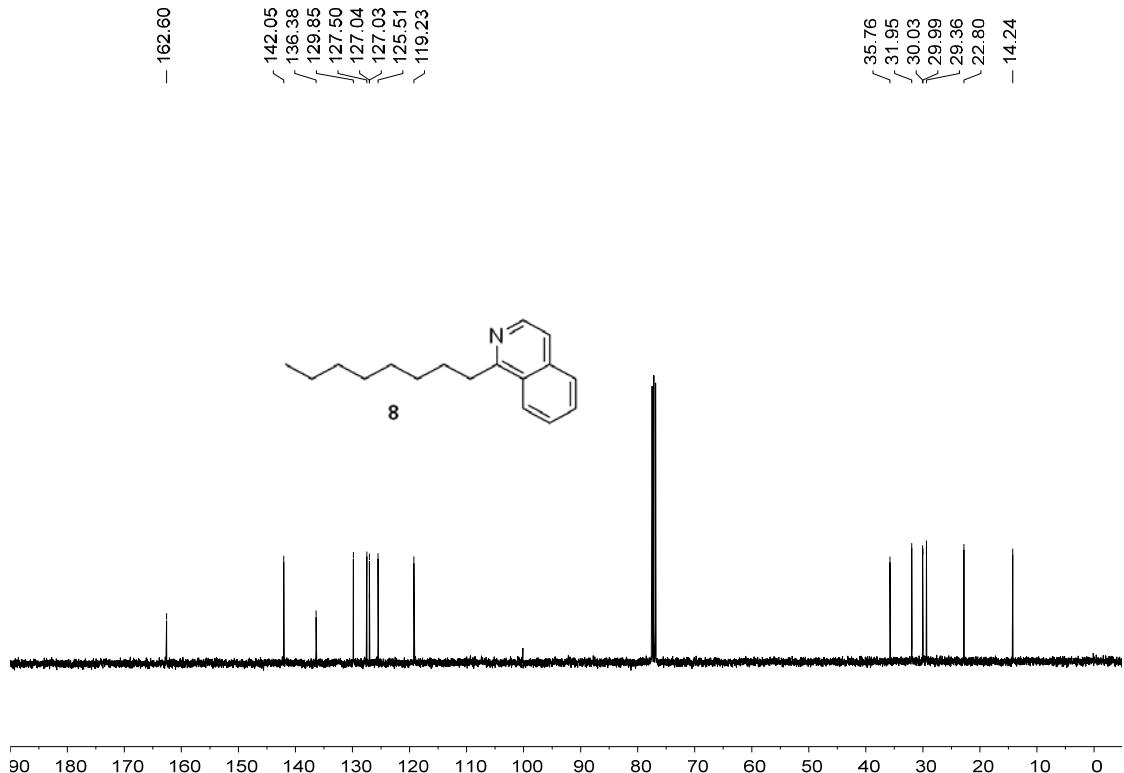
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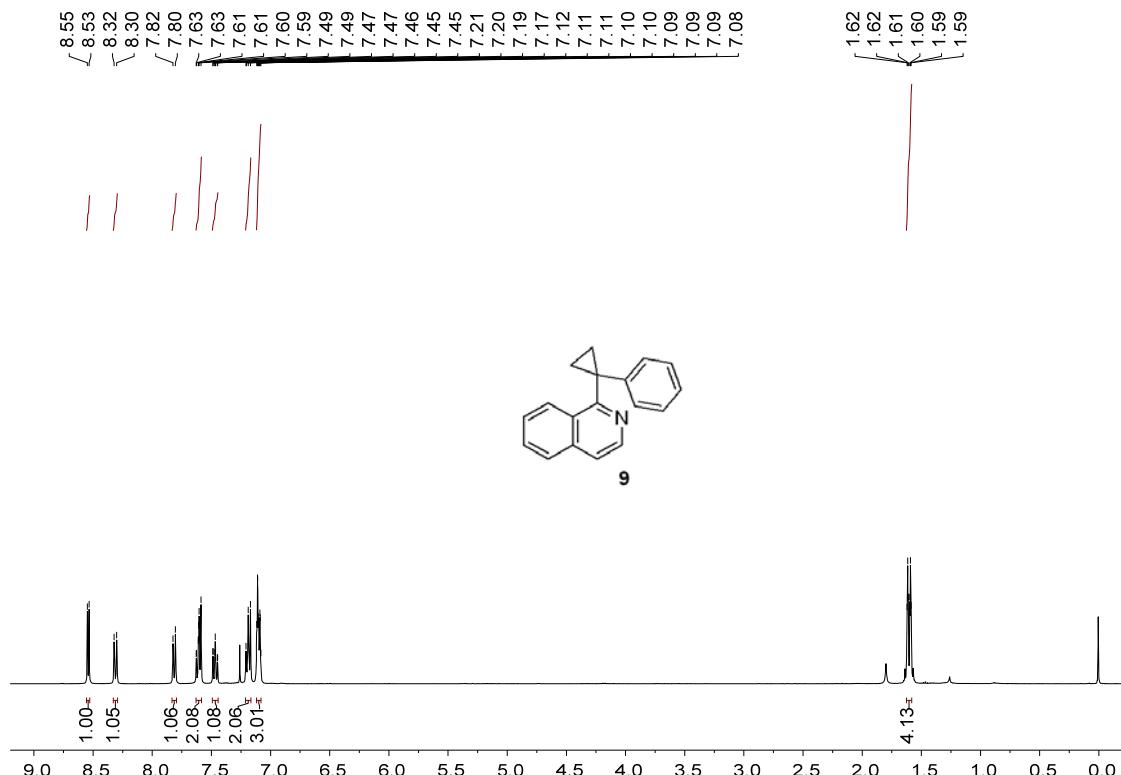
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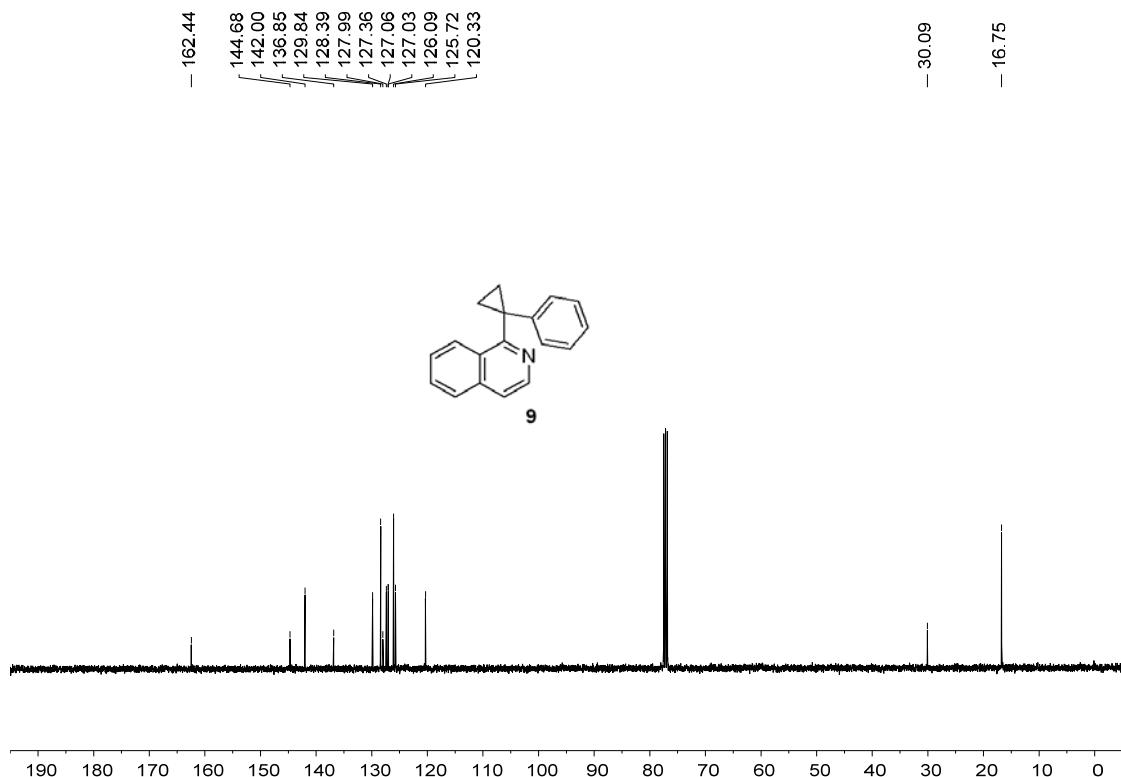
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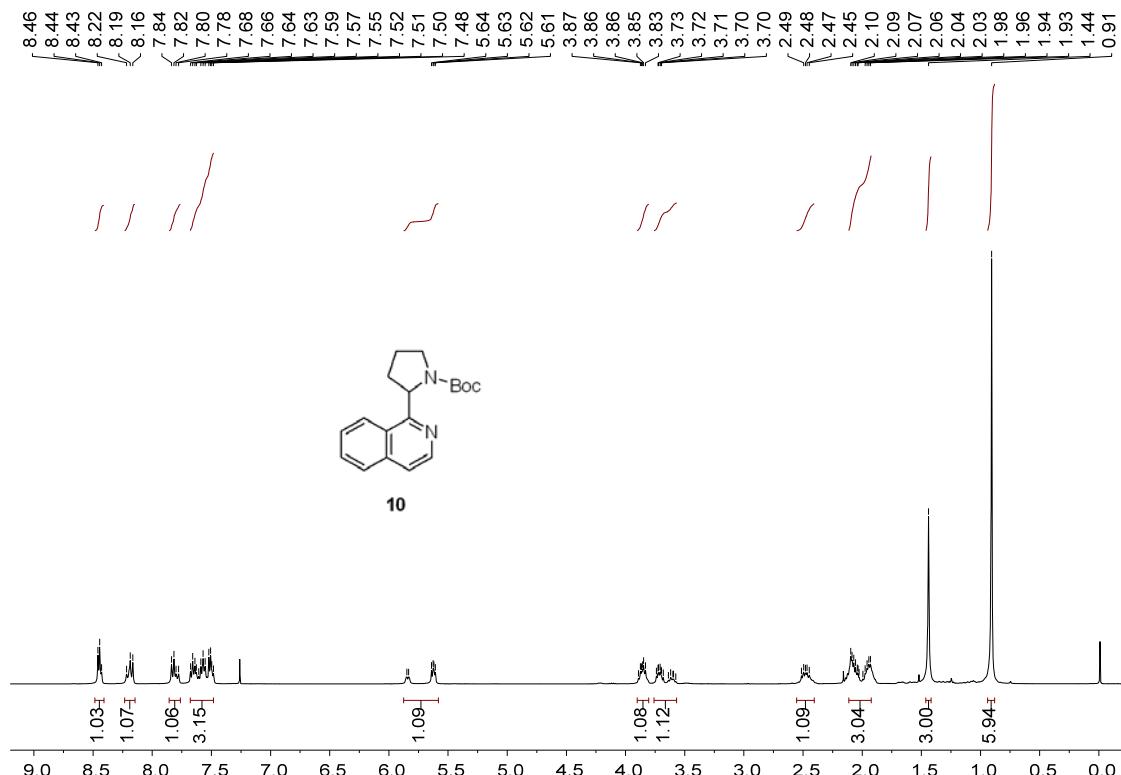
¹H NMR for **9** (400 MHz, CDCl₃)



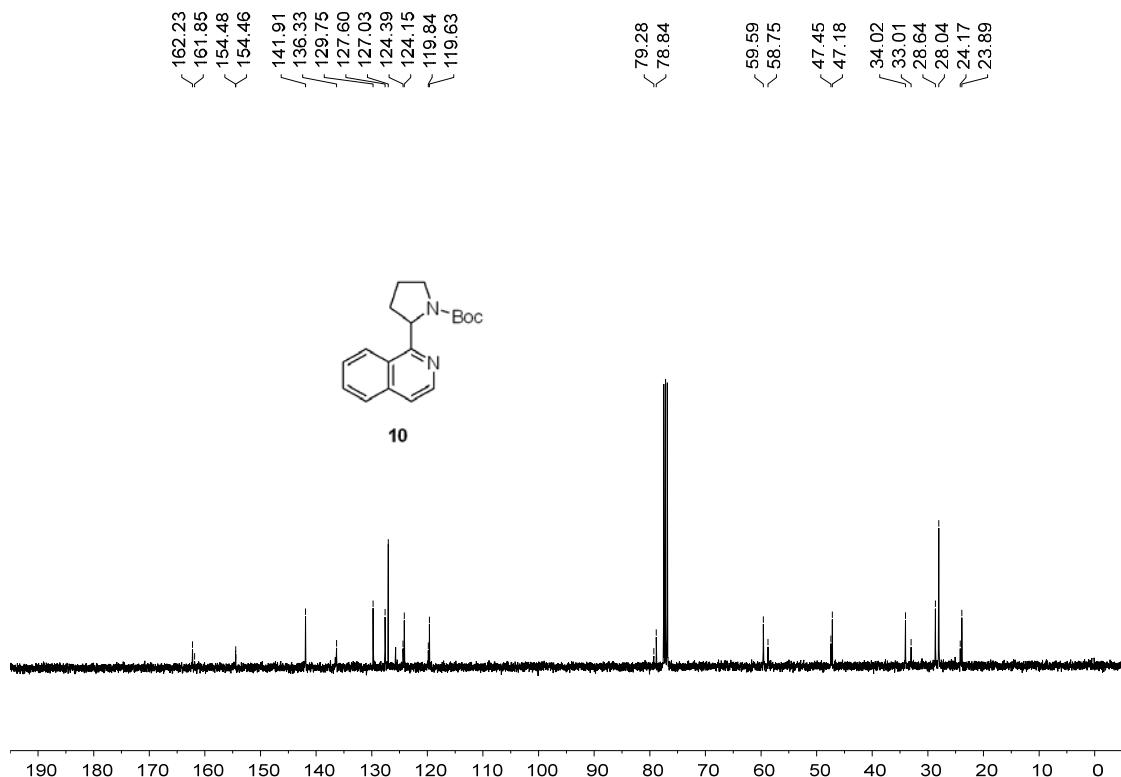
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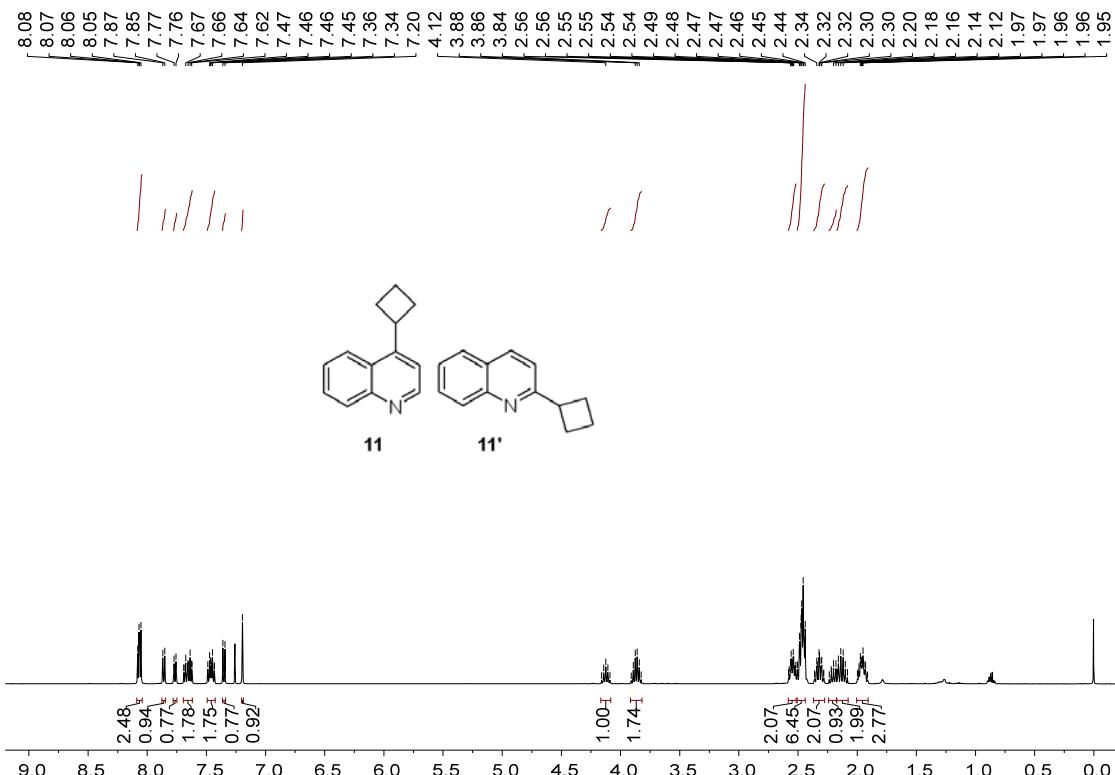
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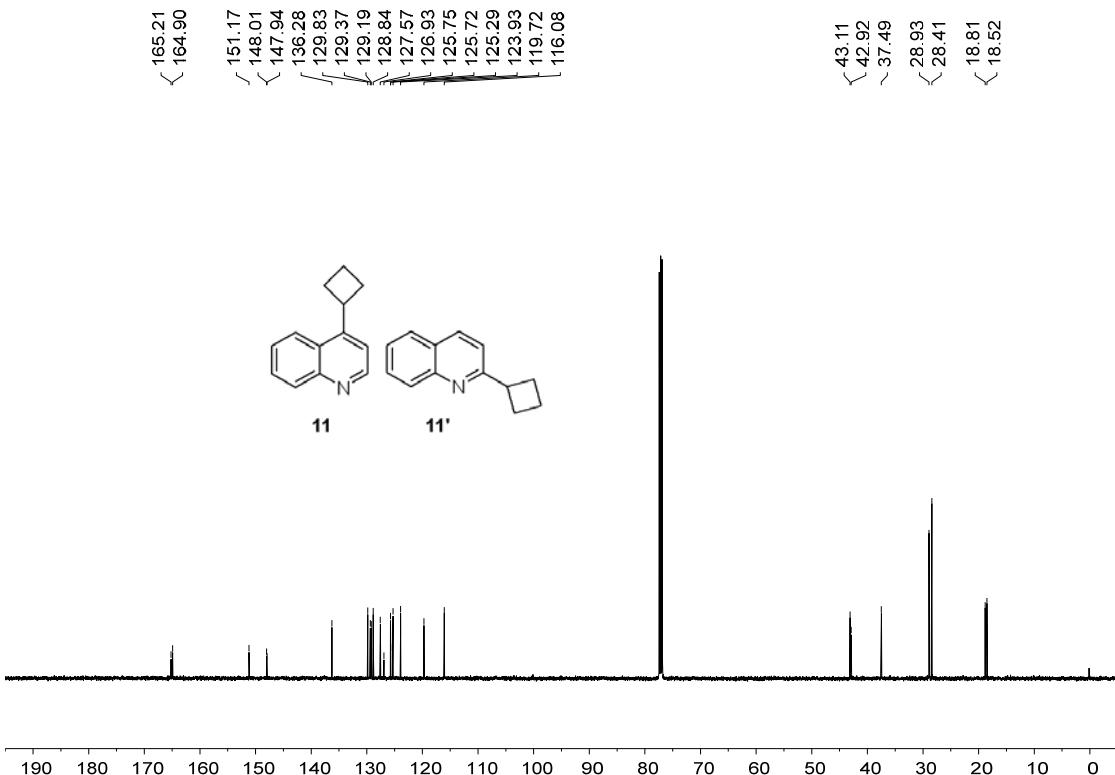
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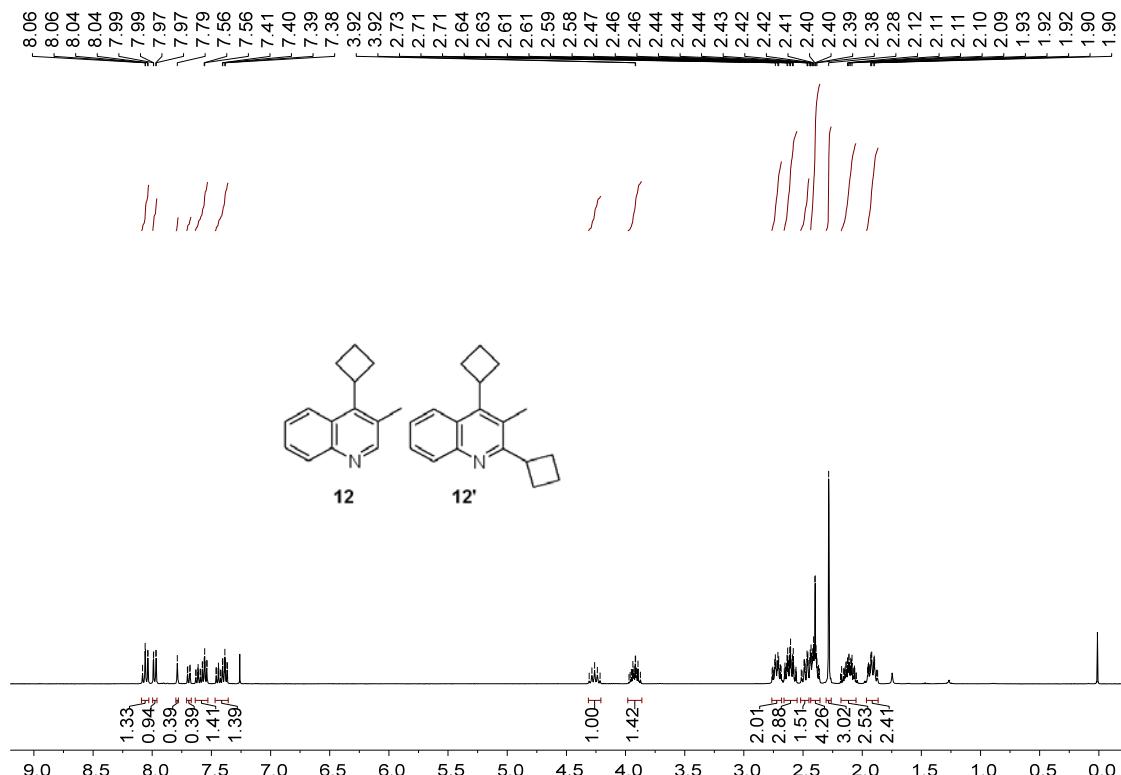
¹H NMR for **11** (500 MHz, CDCl₃)



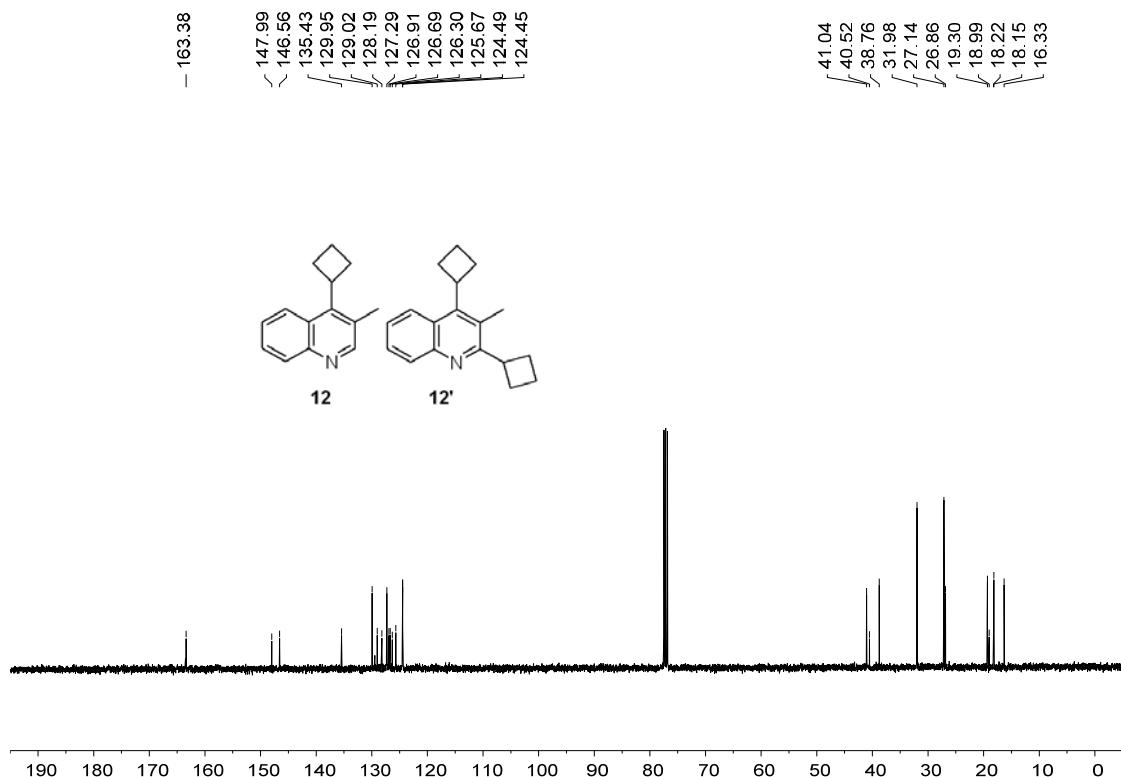
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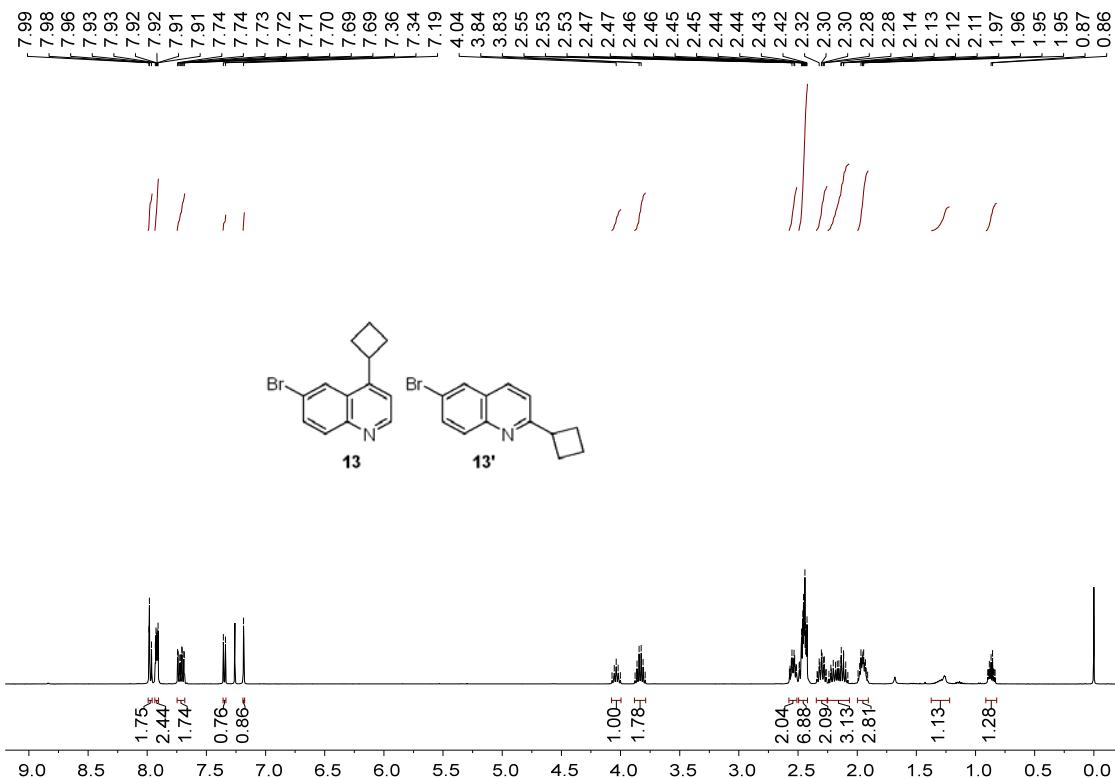
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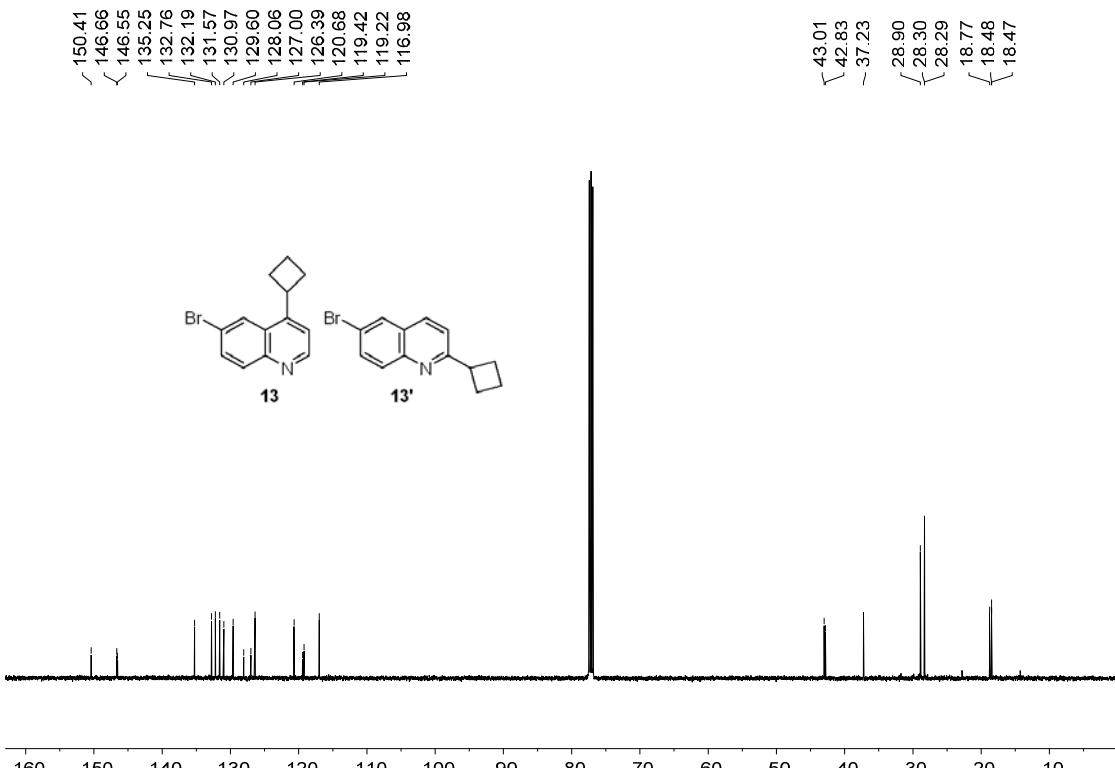
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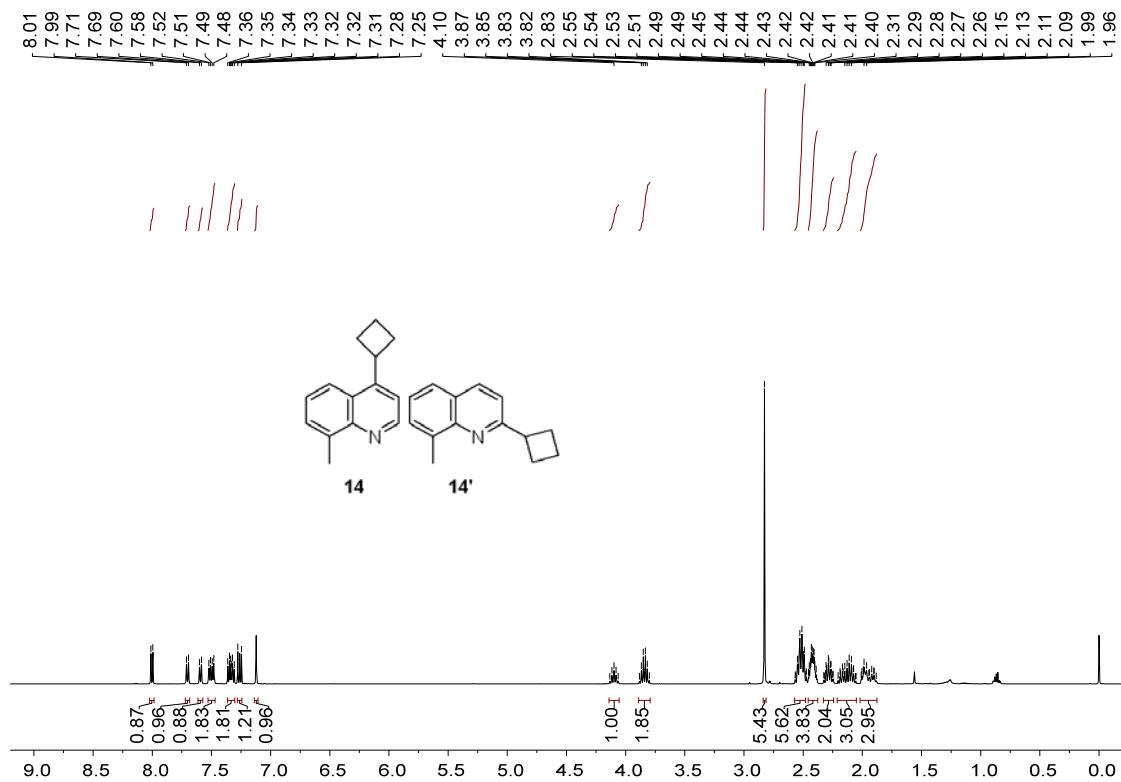
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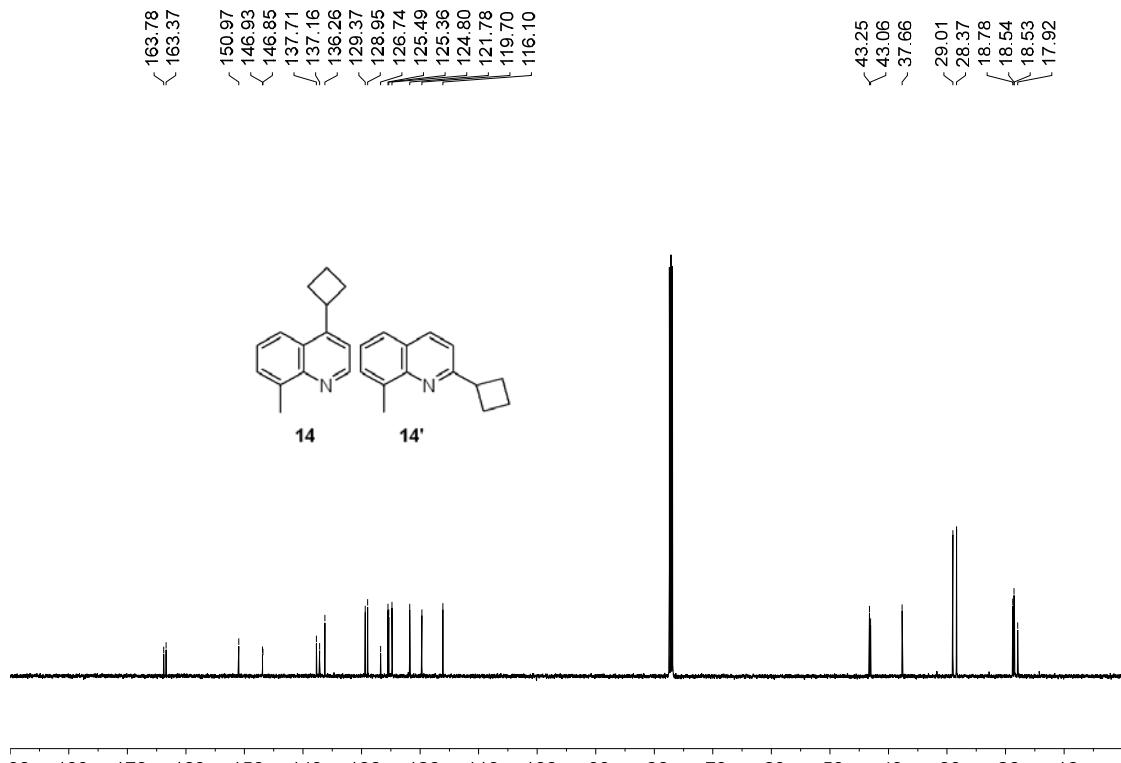
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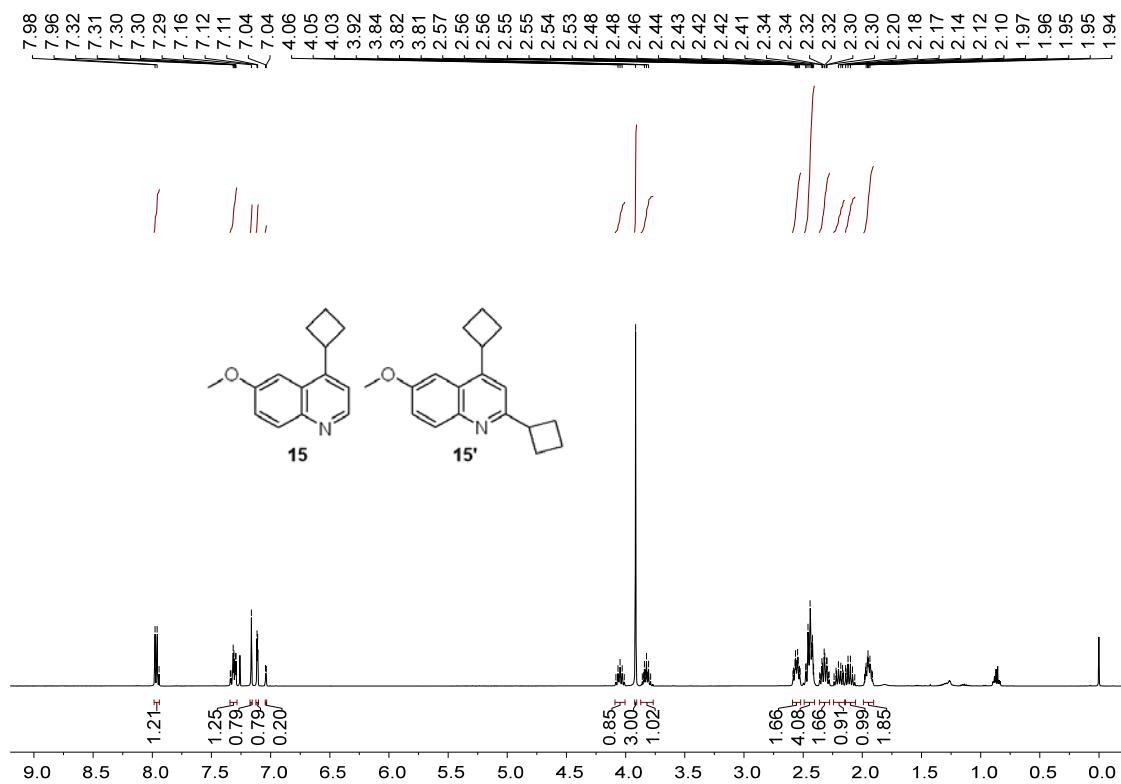
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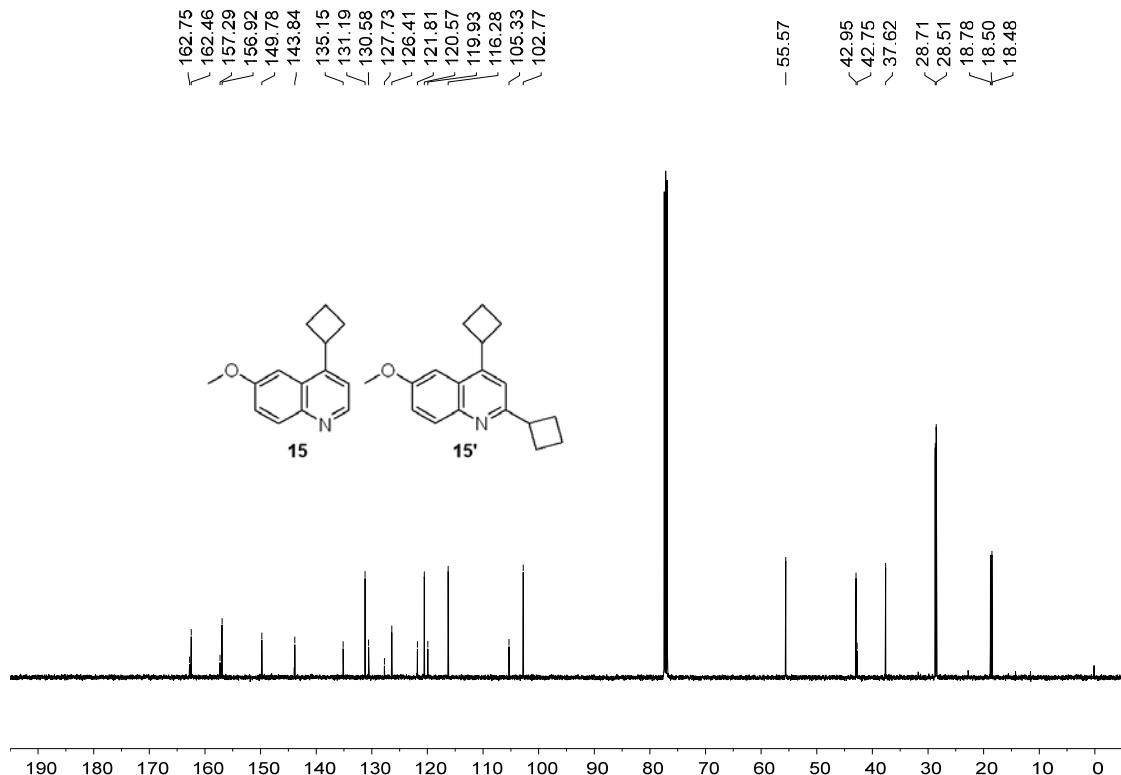
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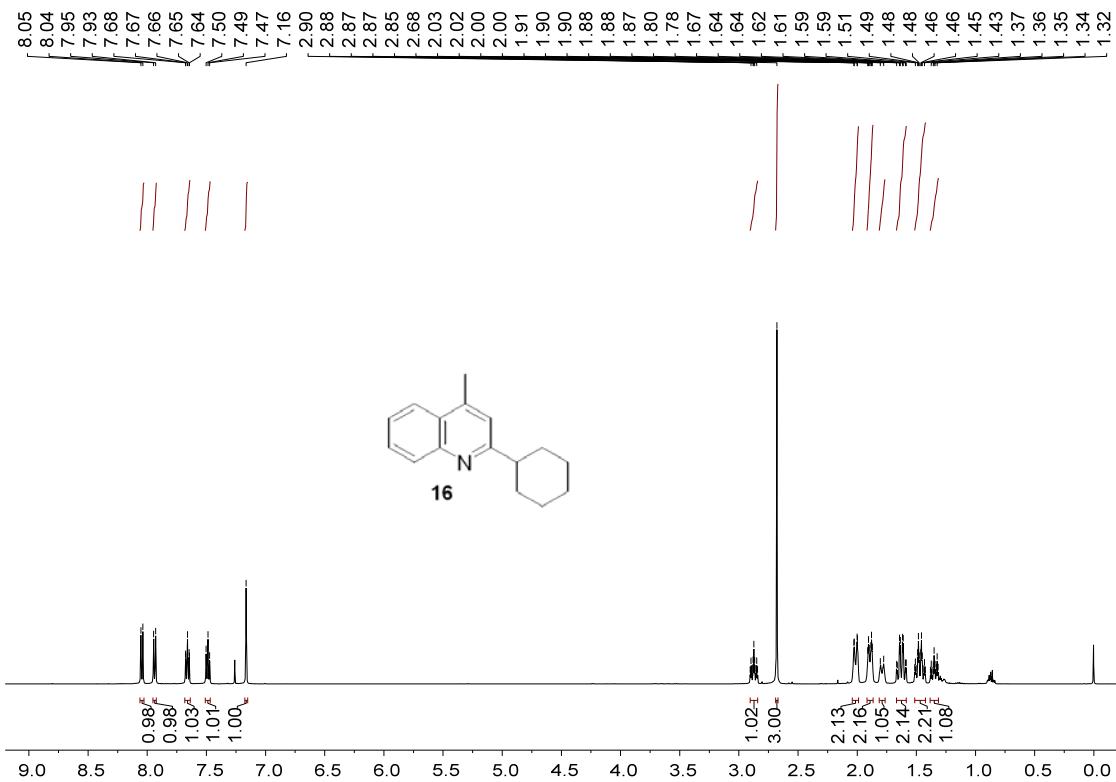
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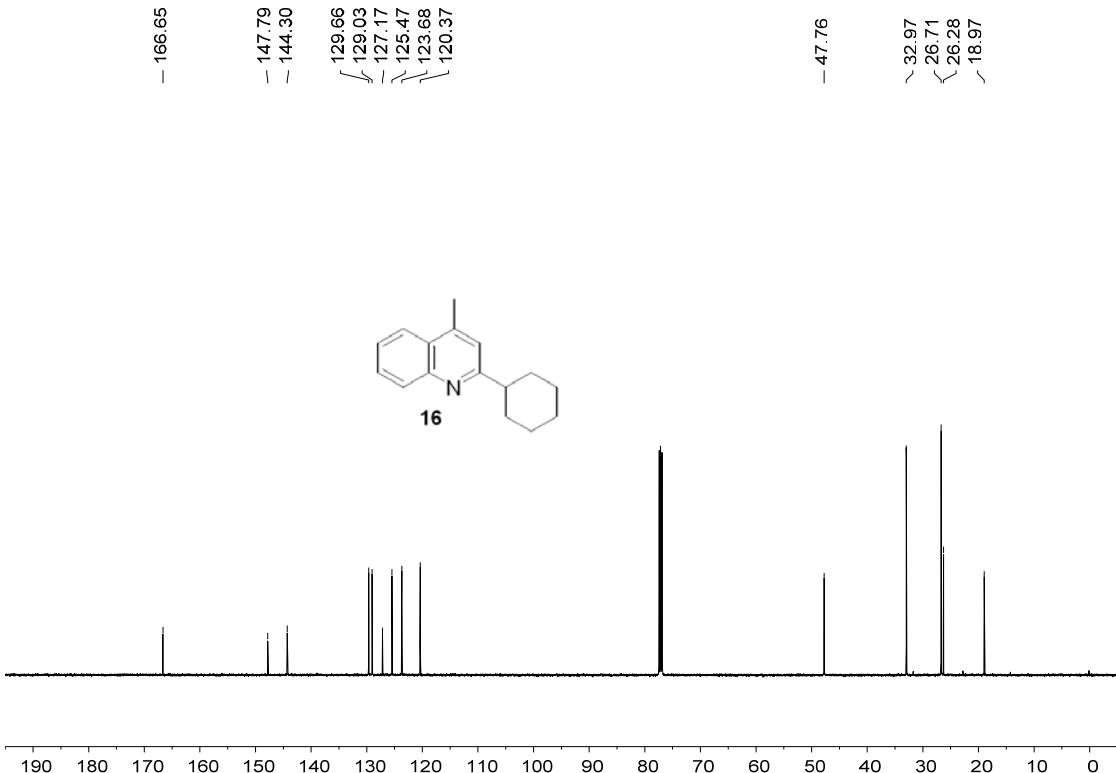
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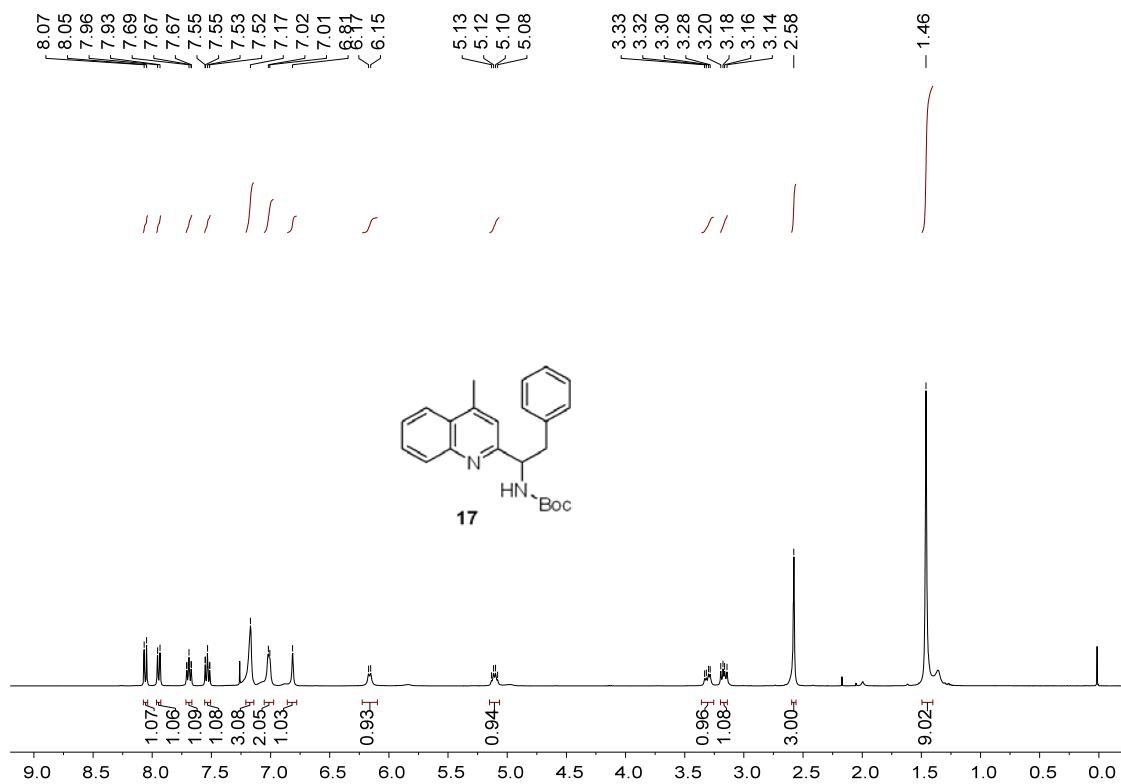
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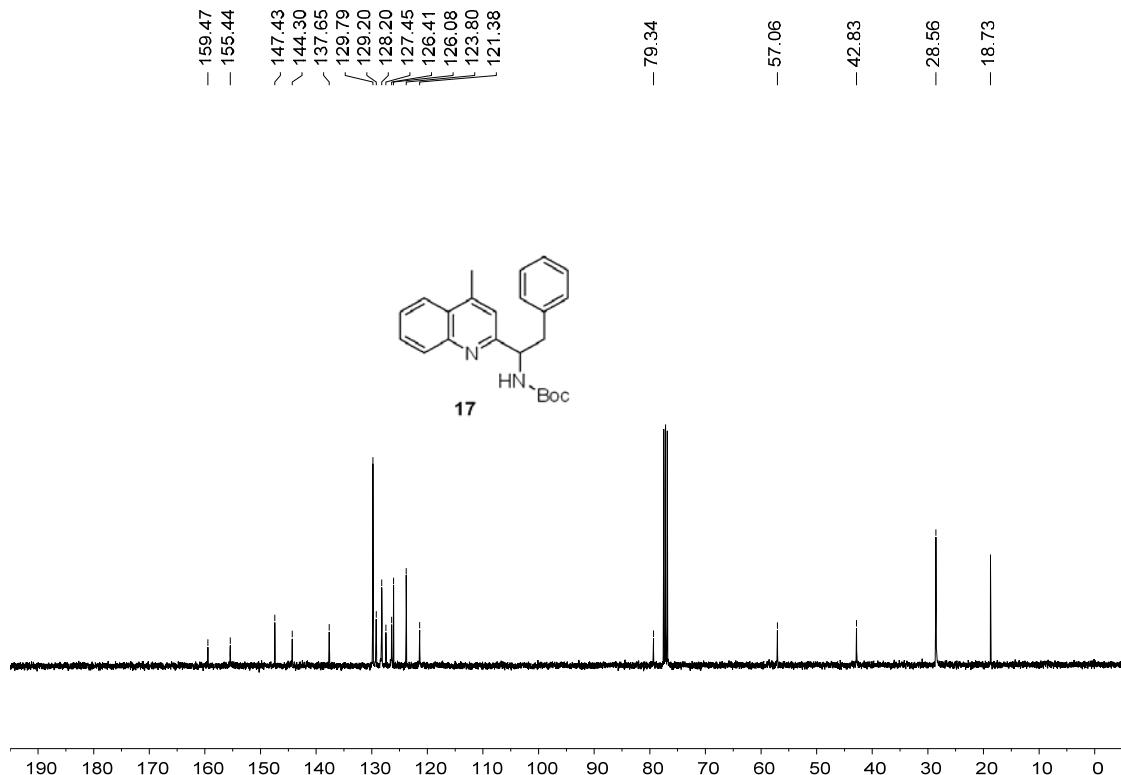
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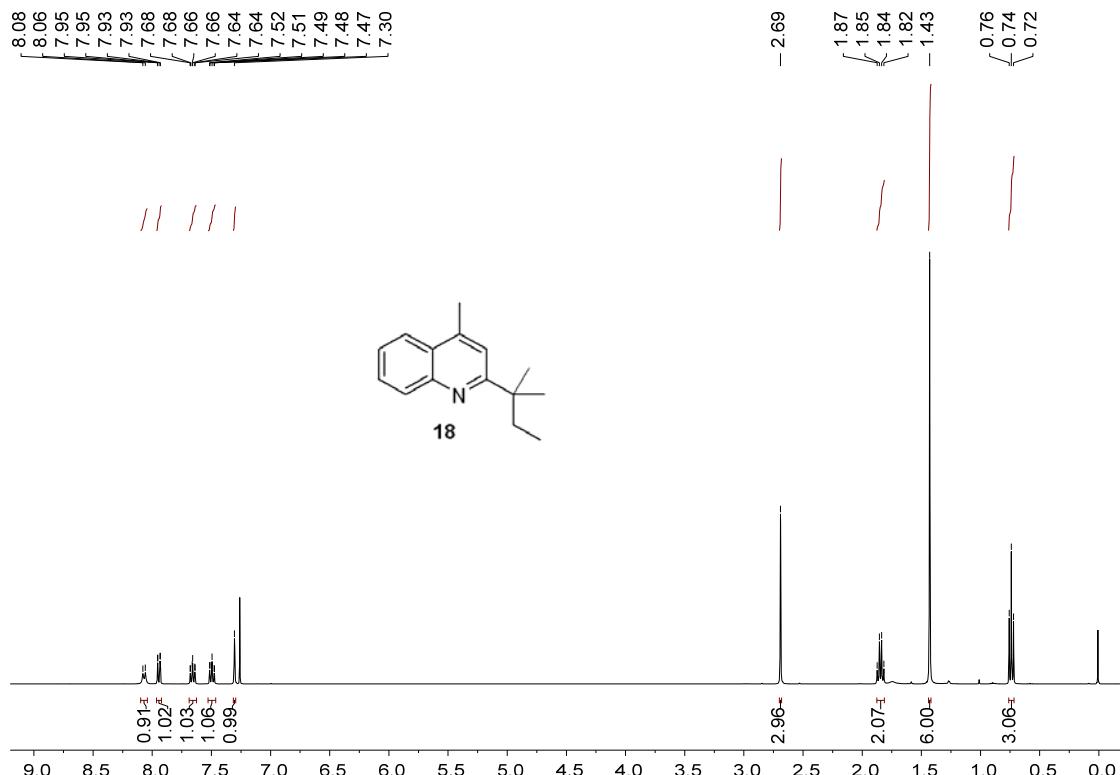
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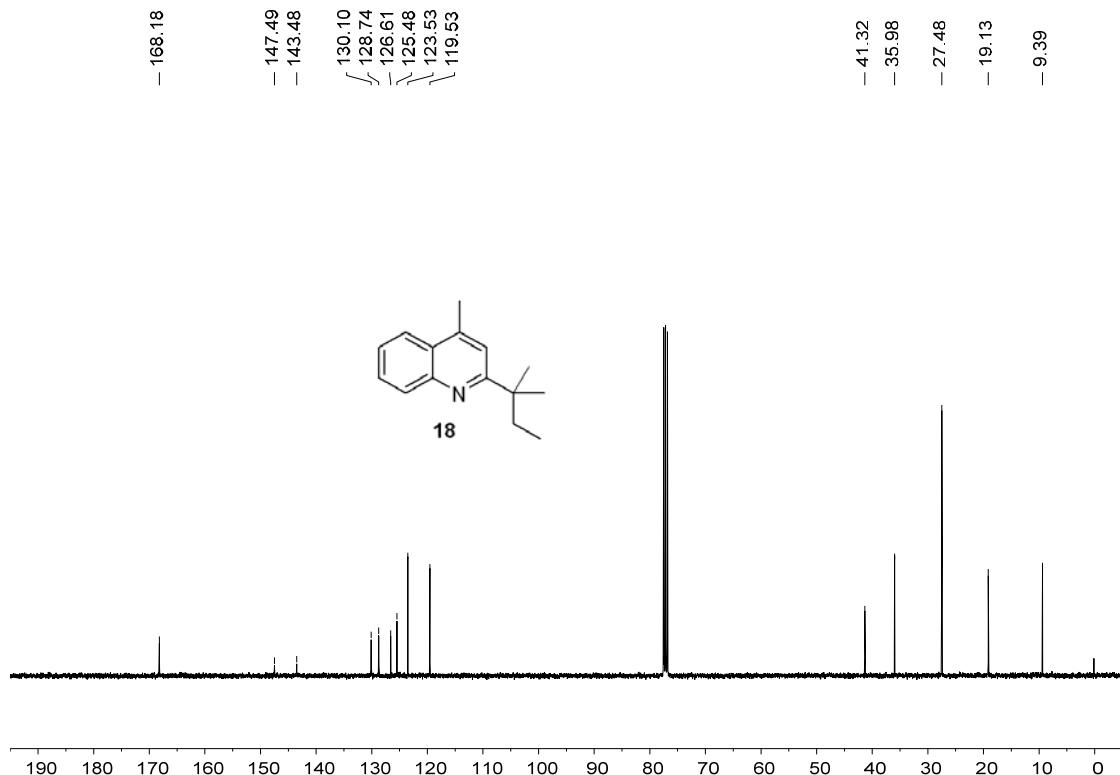
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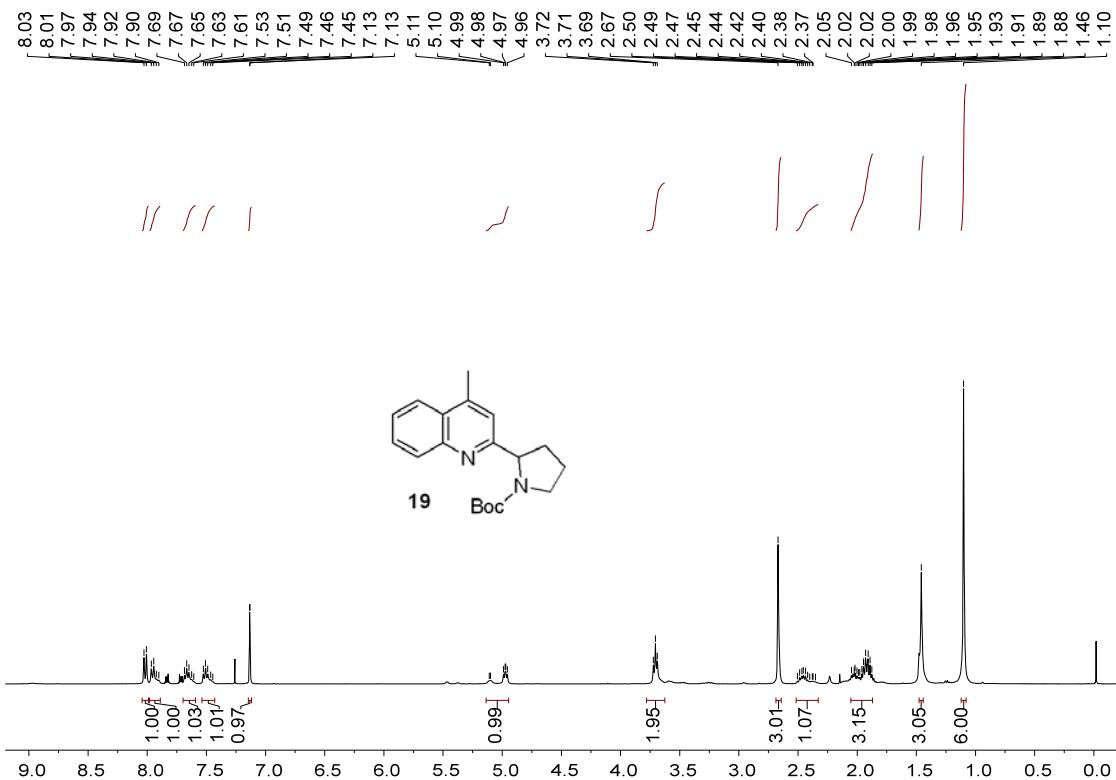
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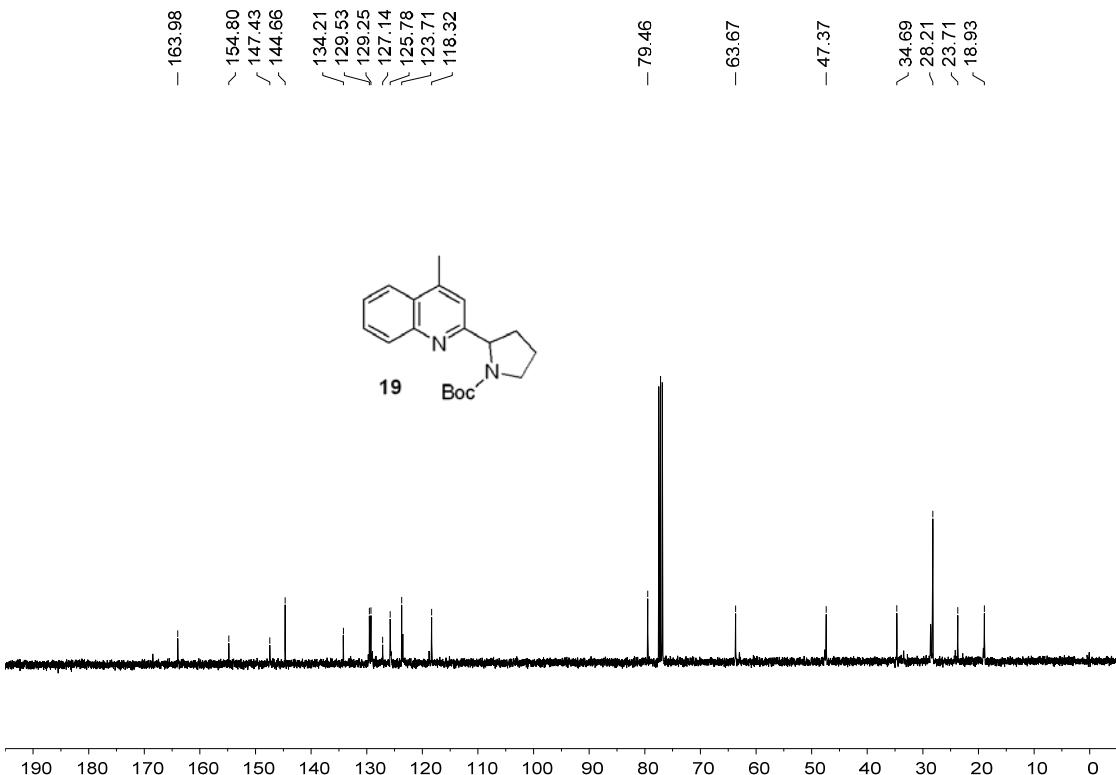
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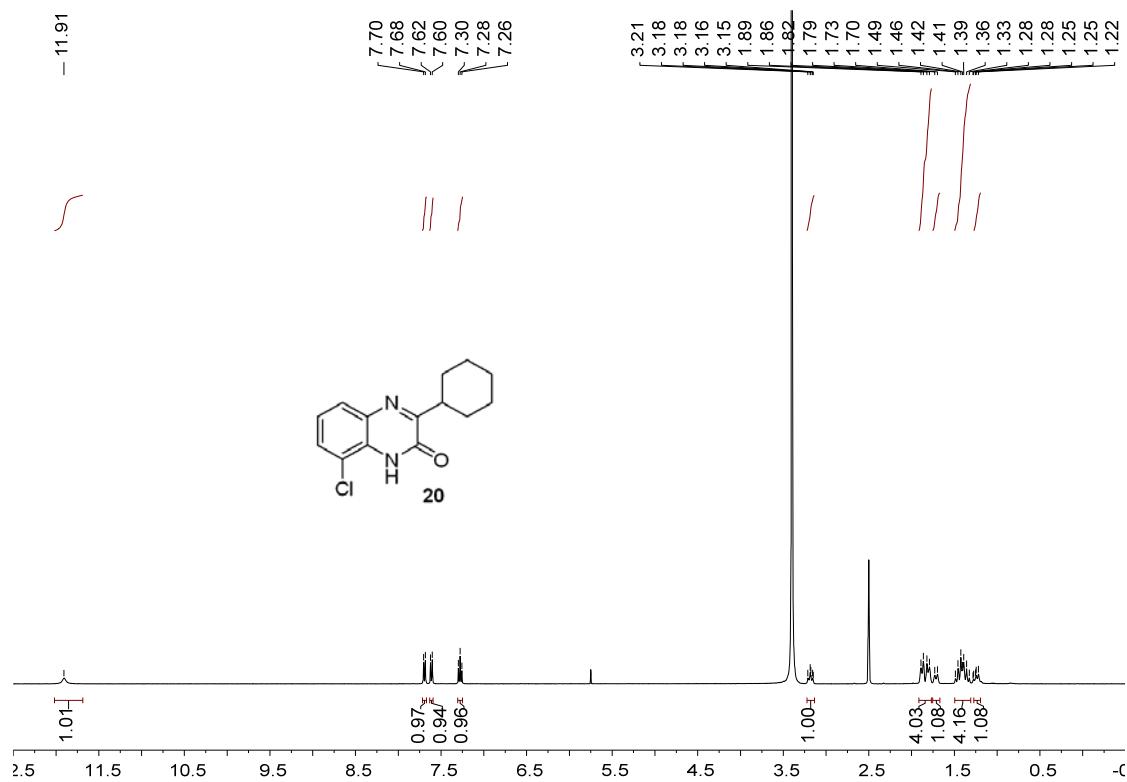
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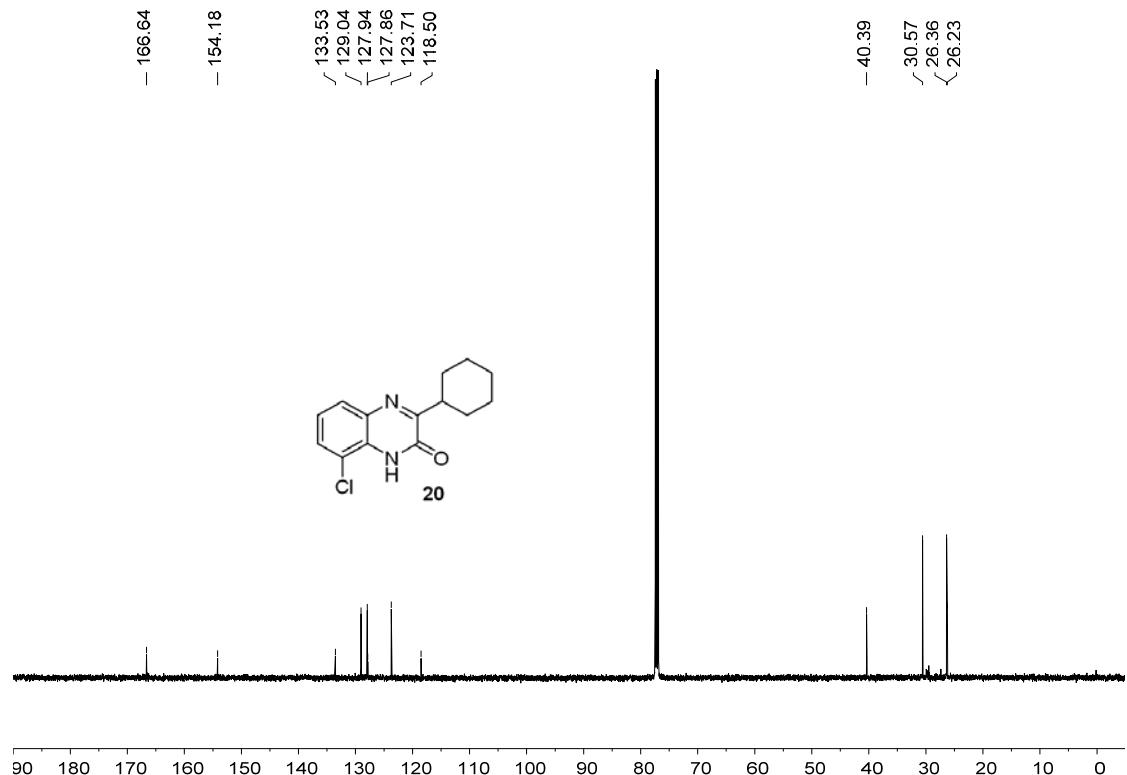
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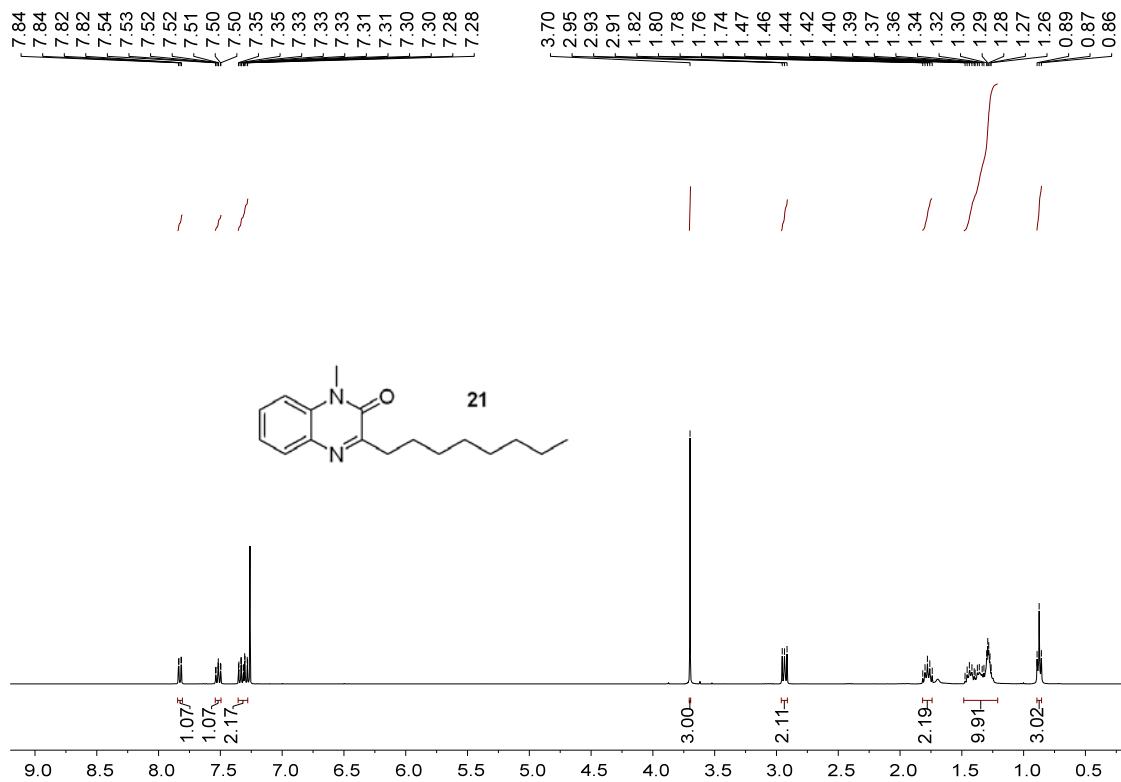
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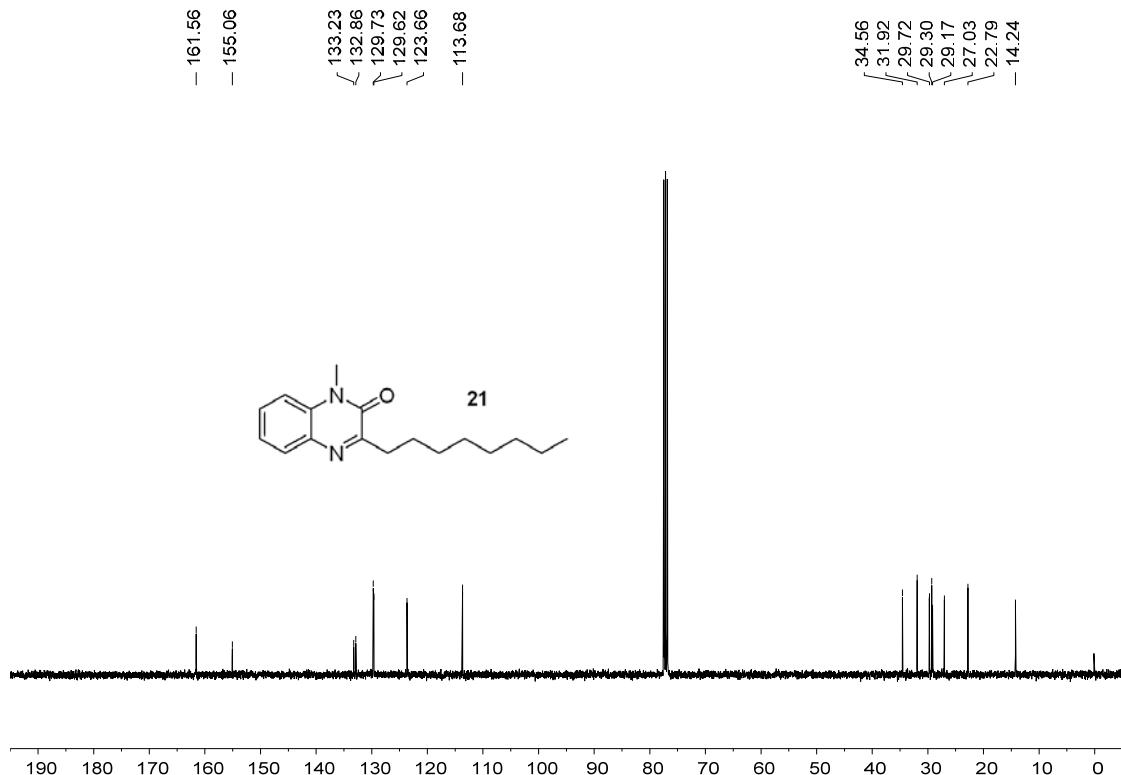
¹³C NMR for **20** (126 MHz, CDCl₃)



¹H NMR for **21** (400 MHz, CDCl₃)



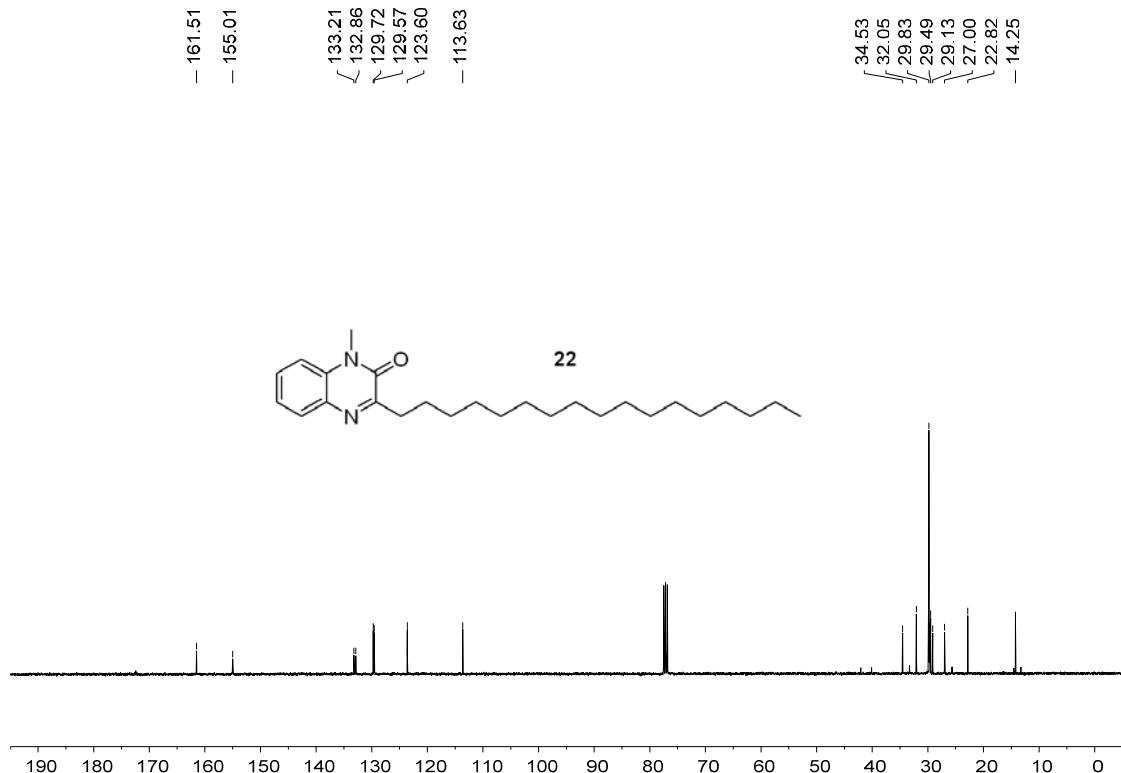
¹³C NMR for **21** (101 MHz, CDCl₃)



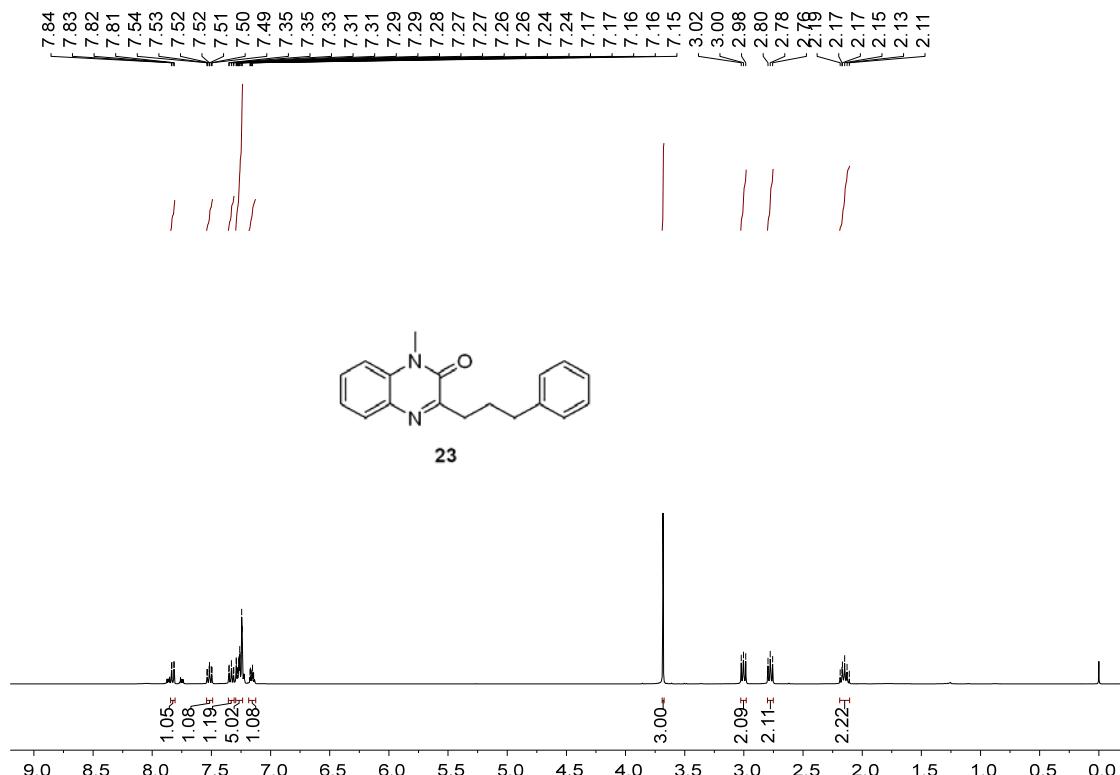
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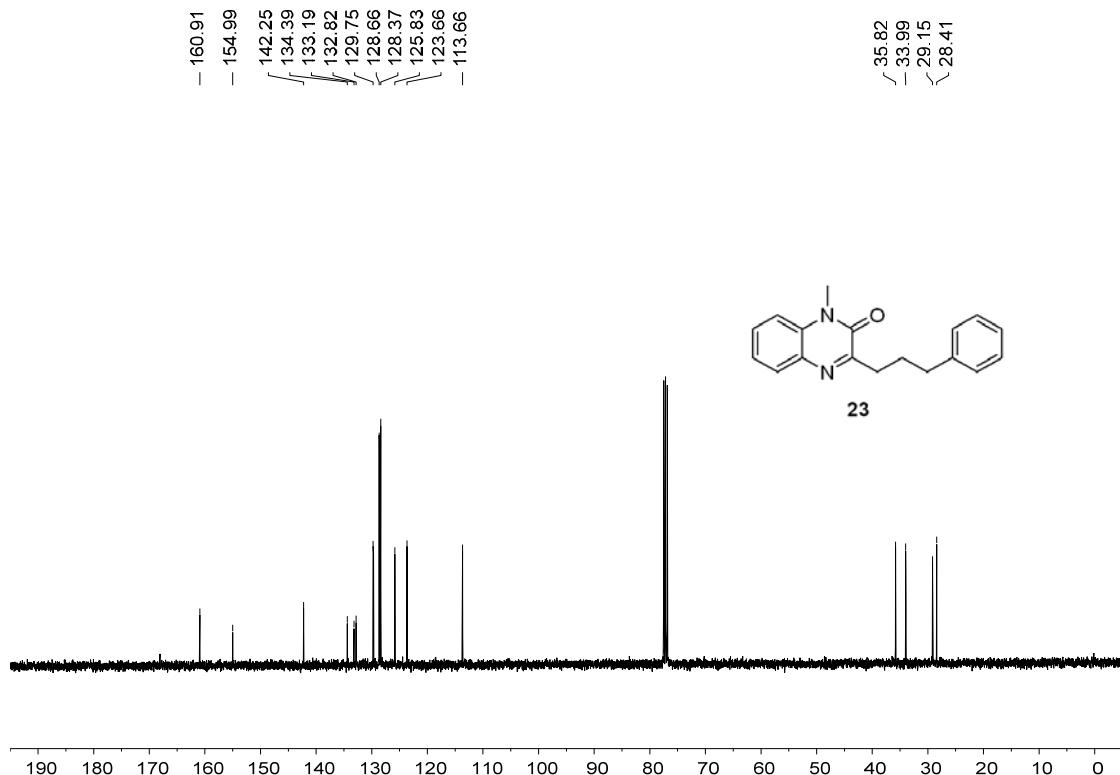
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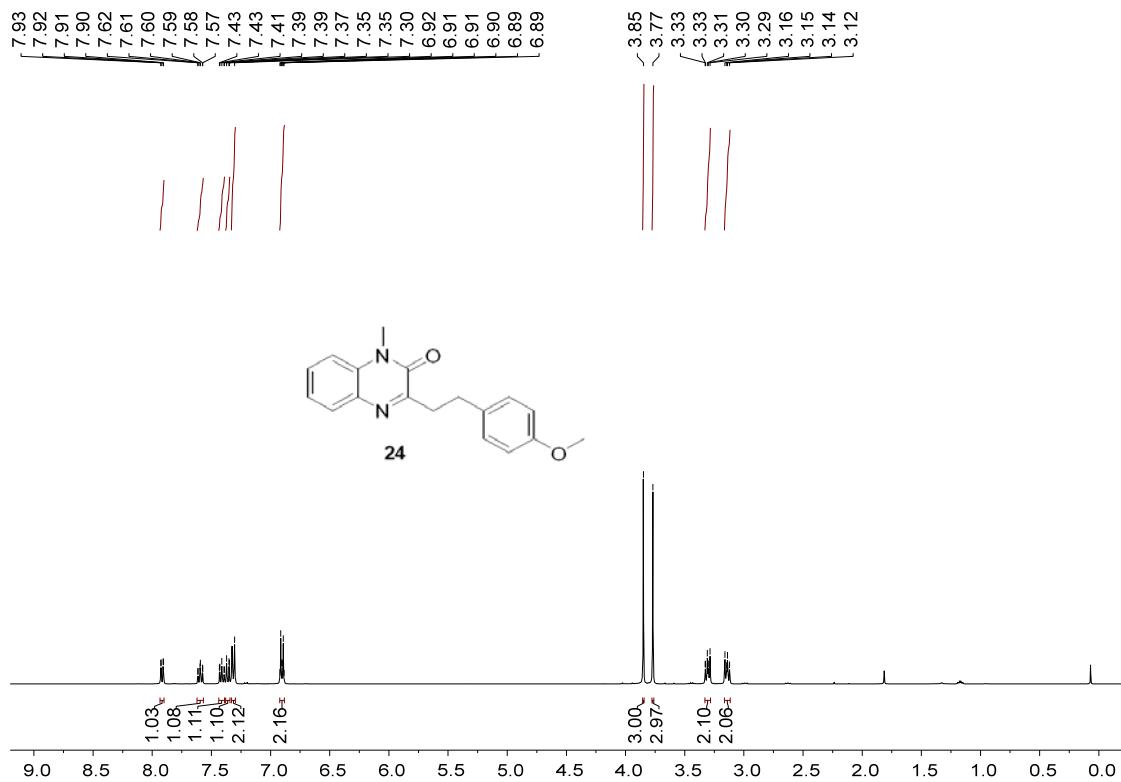
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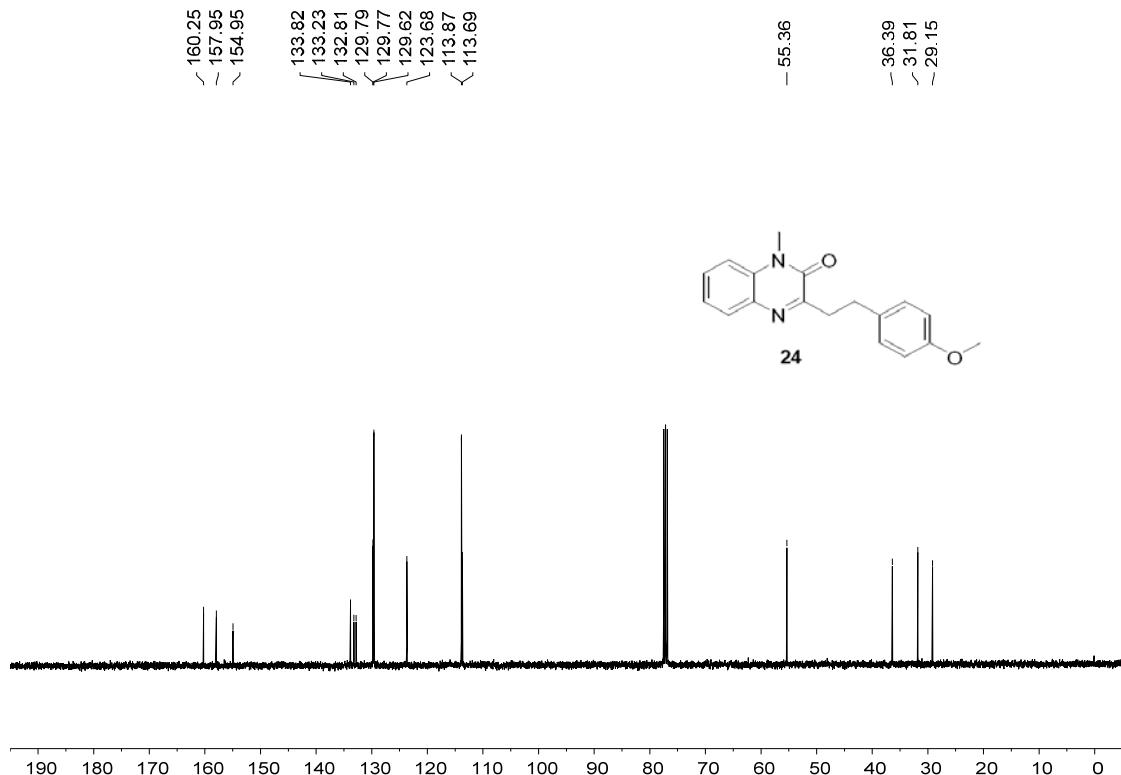
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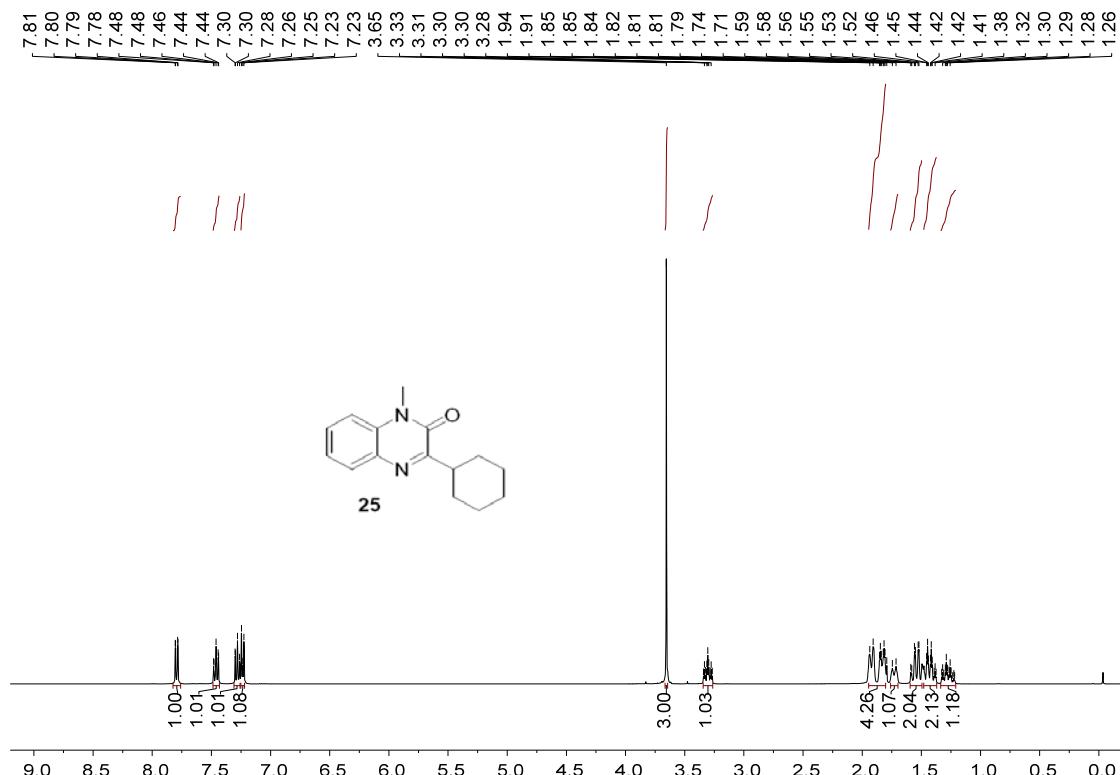
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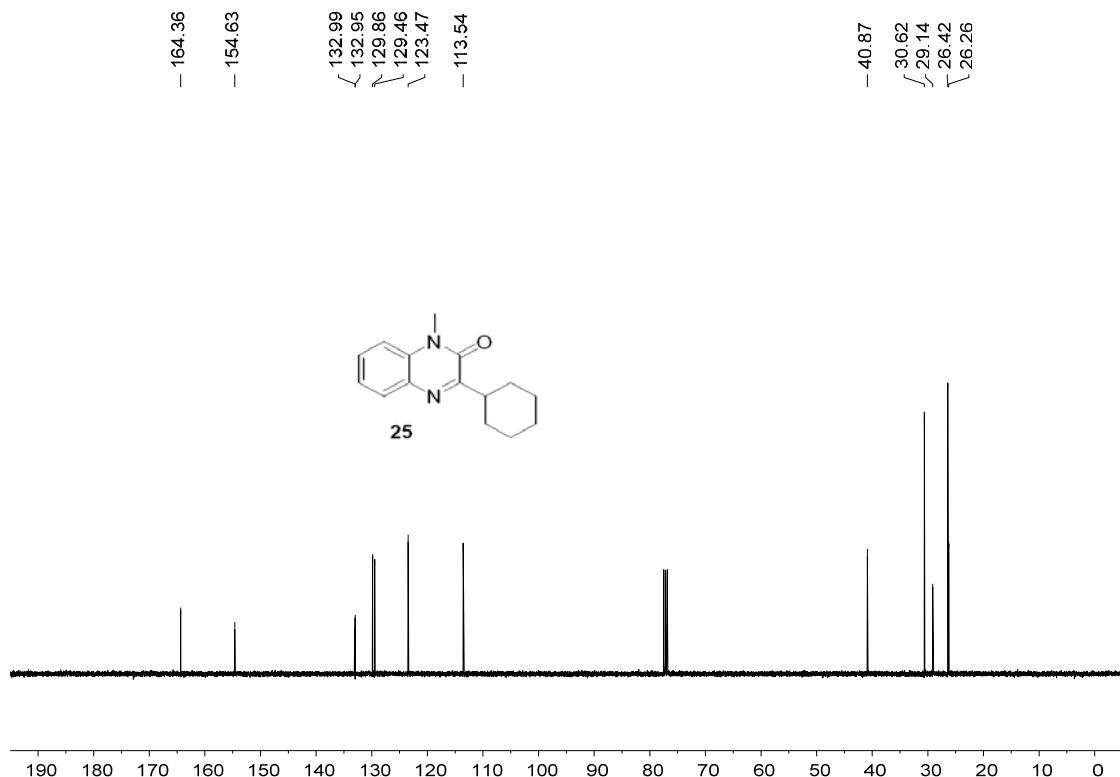
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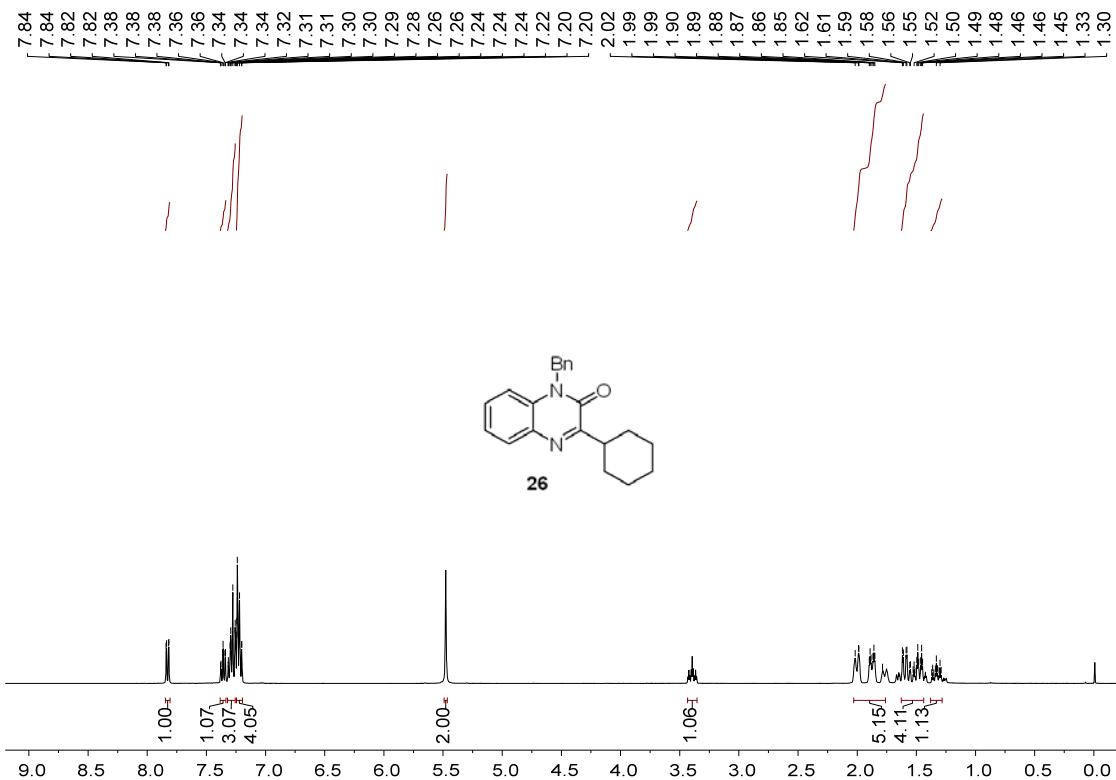
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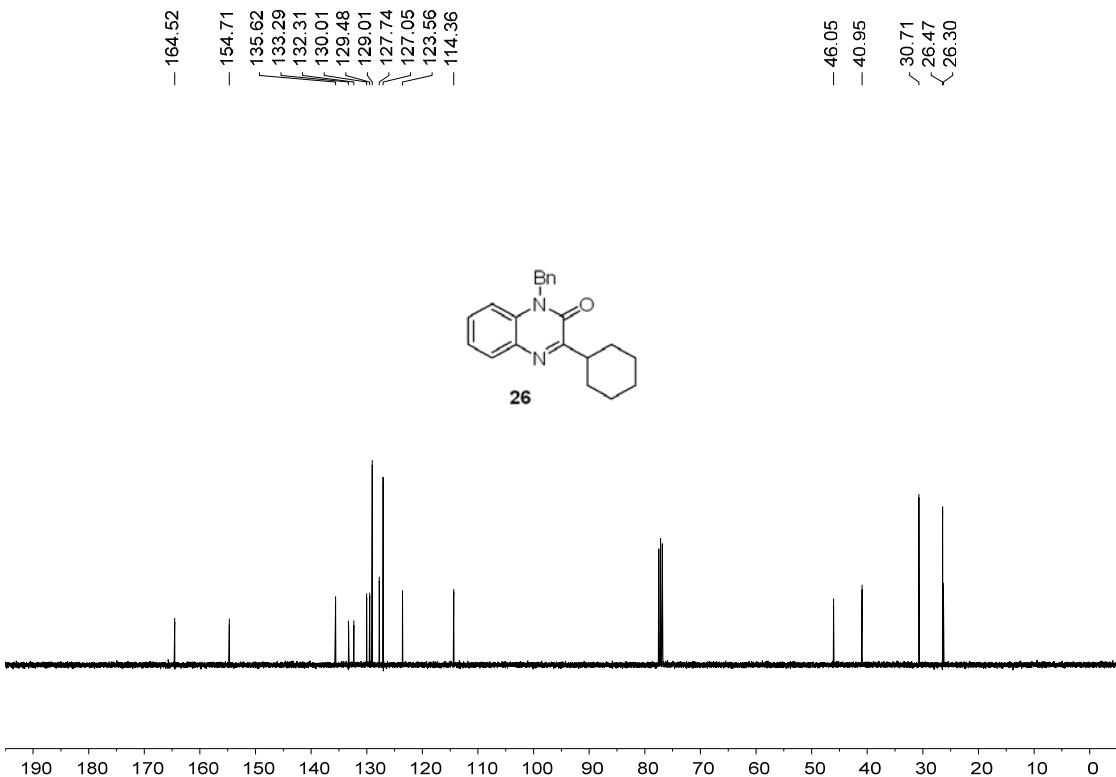
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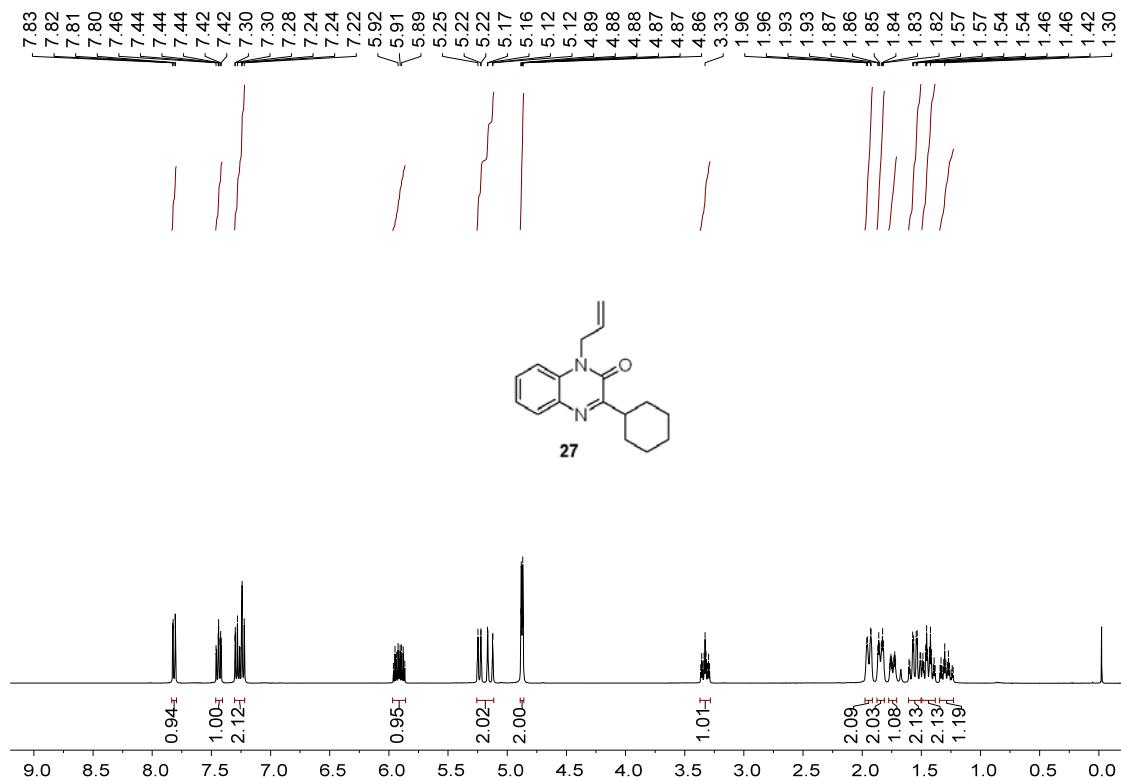
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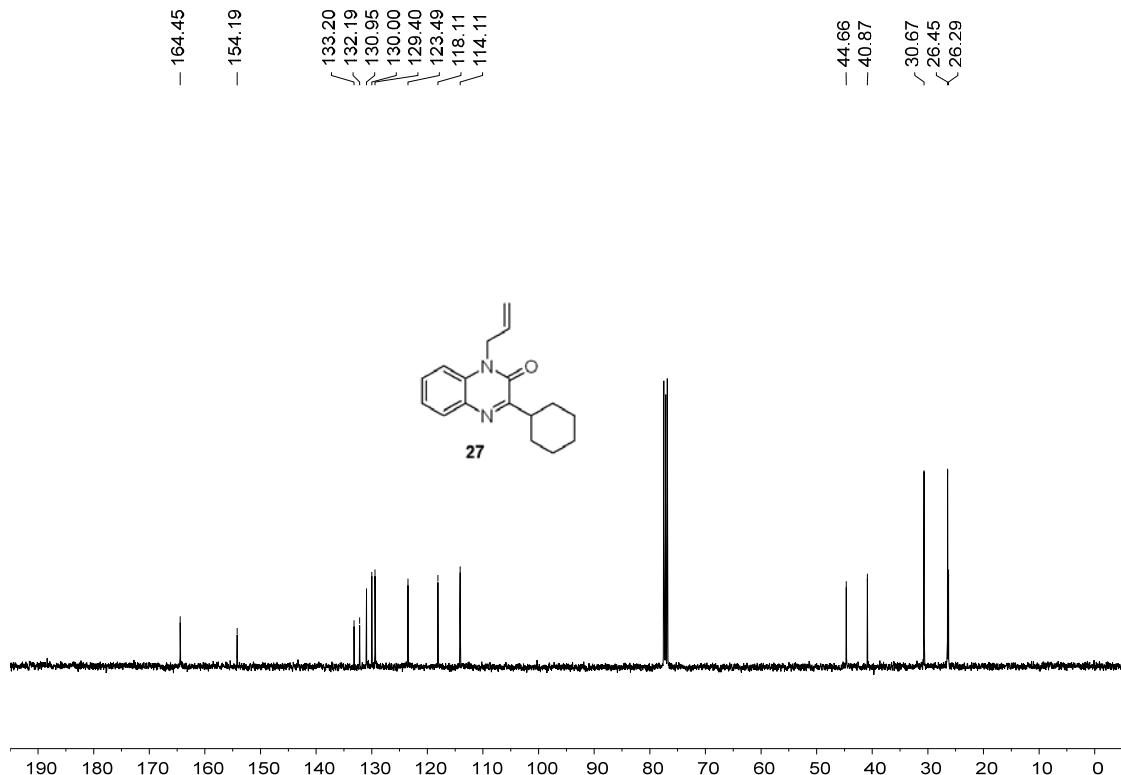
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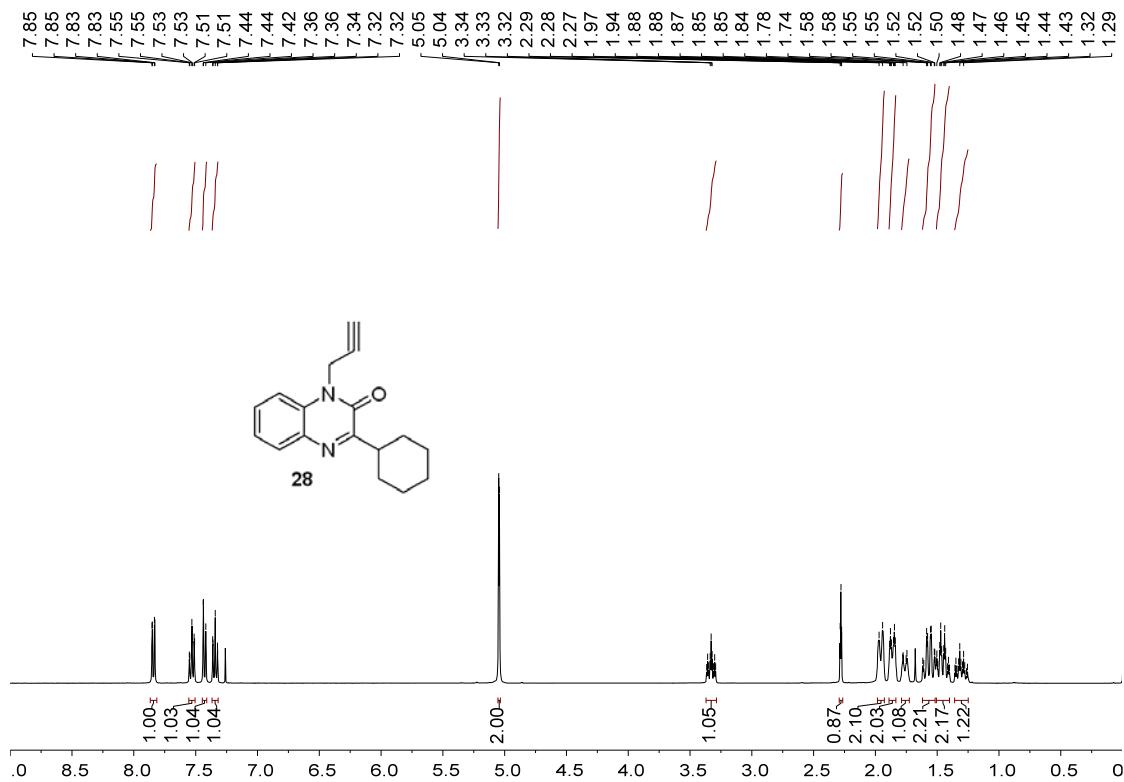
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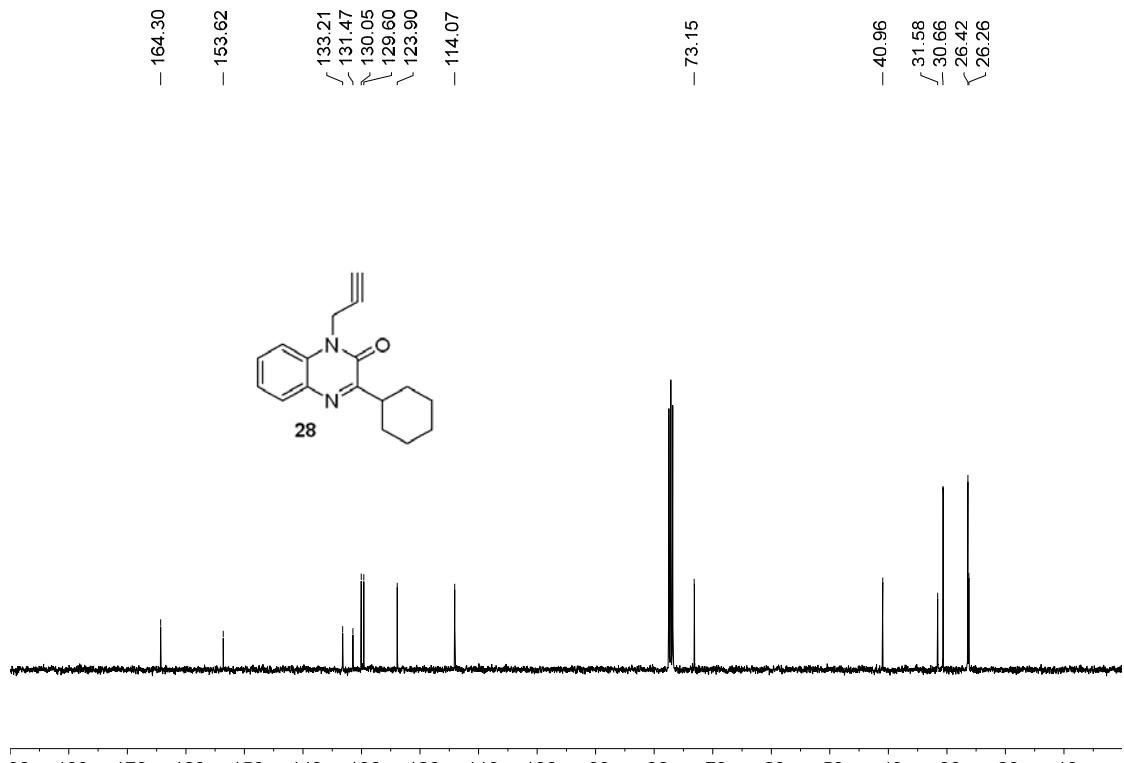
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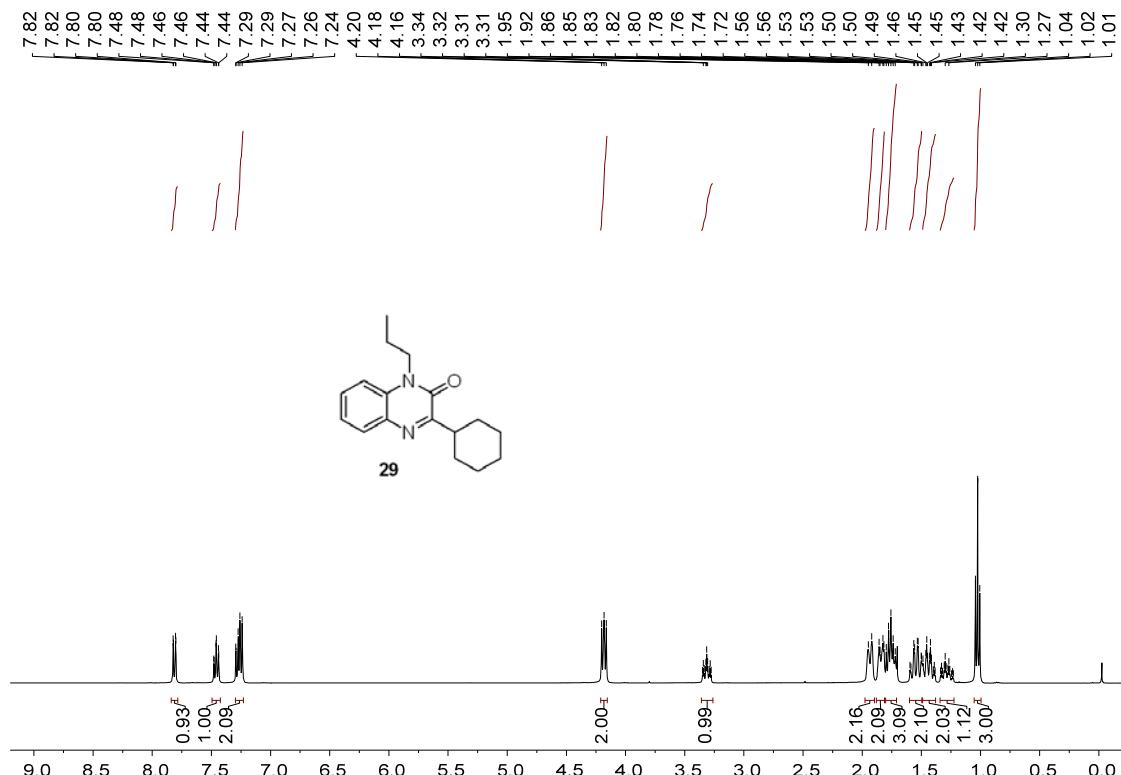
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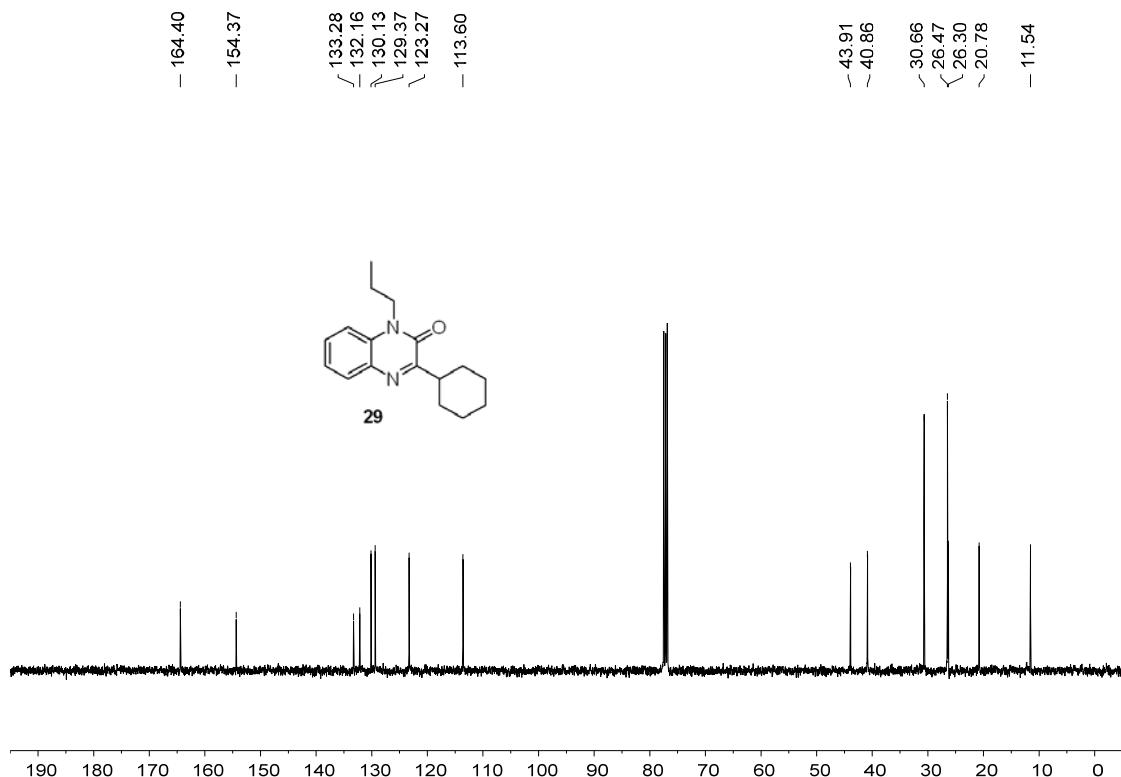
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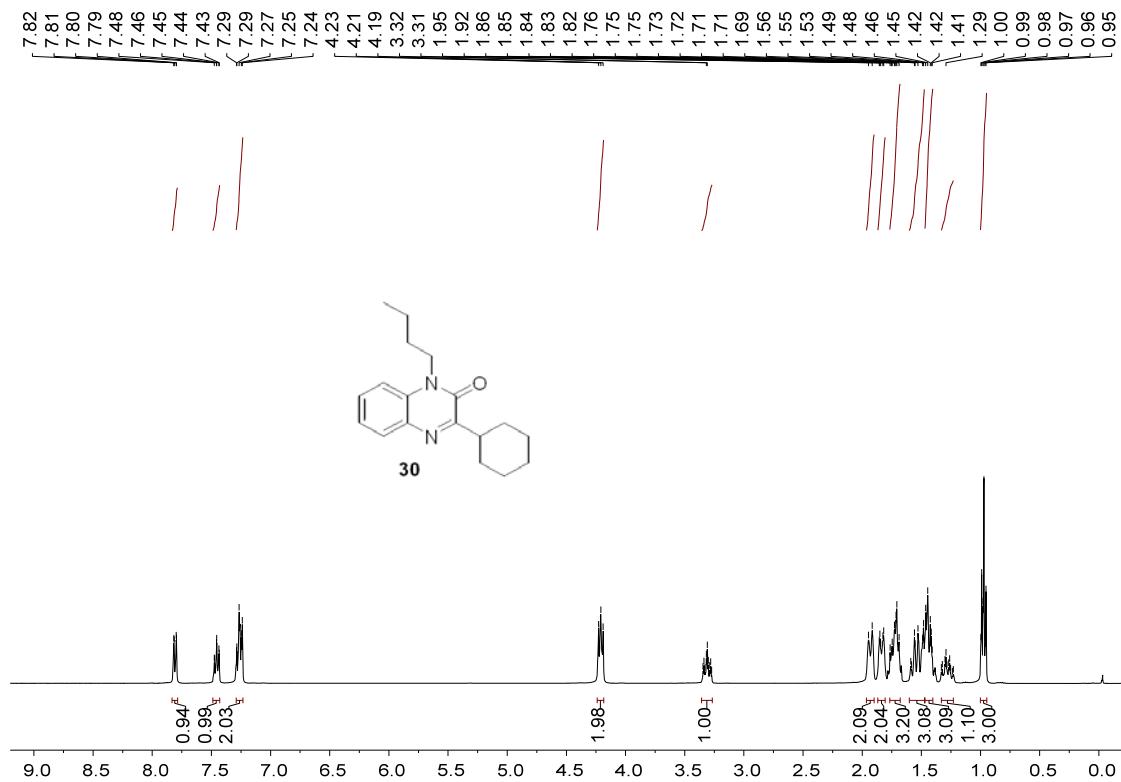
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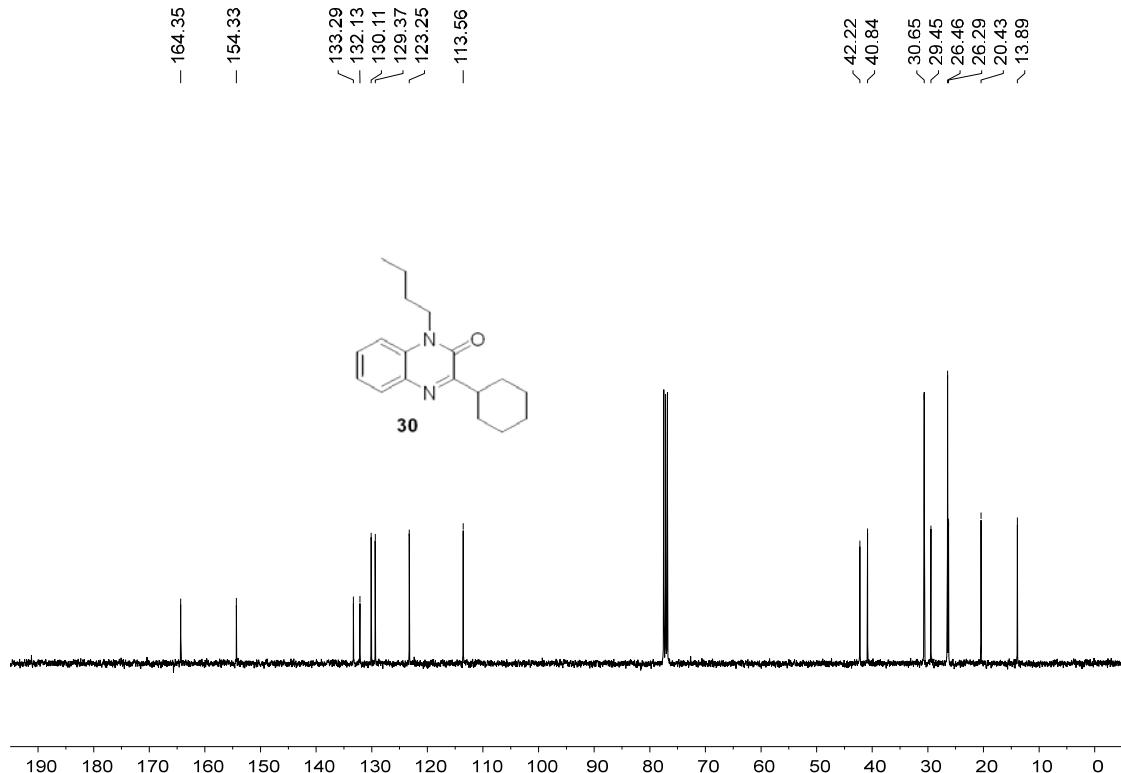
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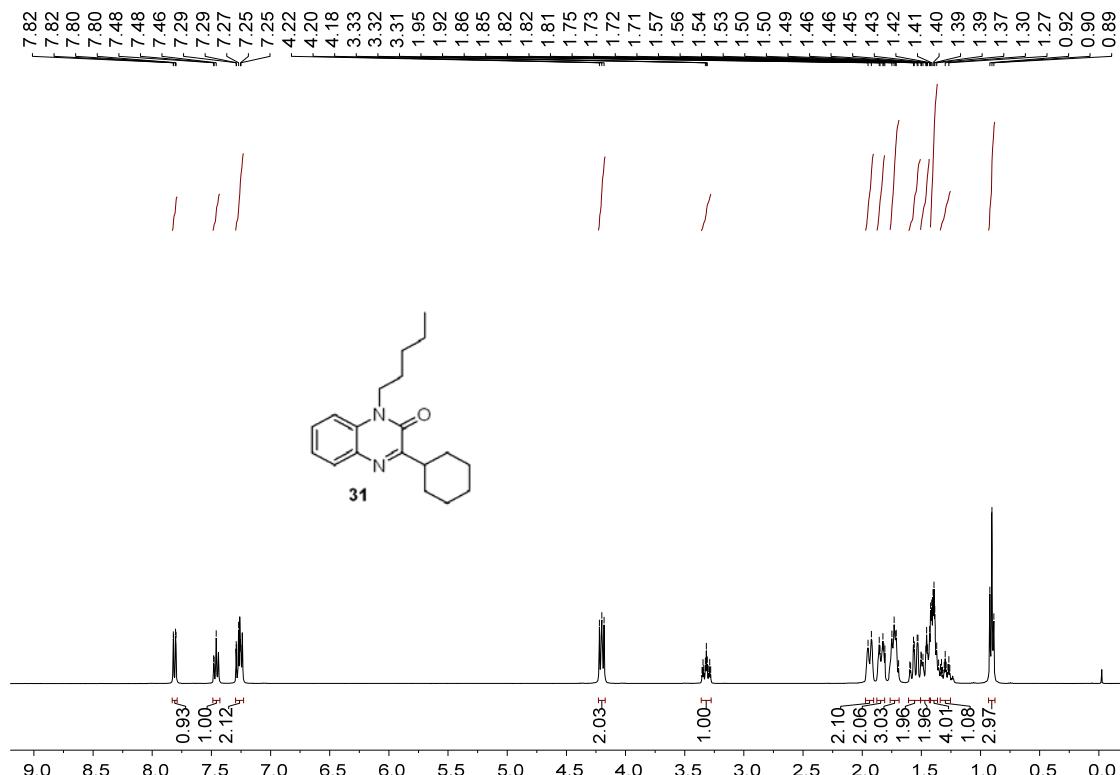
¹H NMR for **30** (400 MHz, CDCl₃)



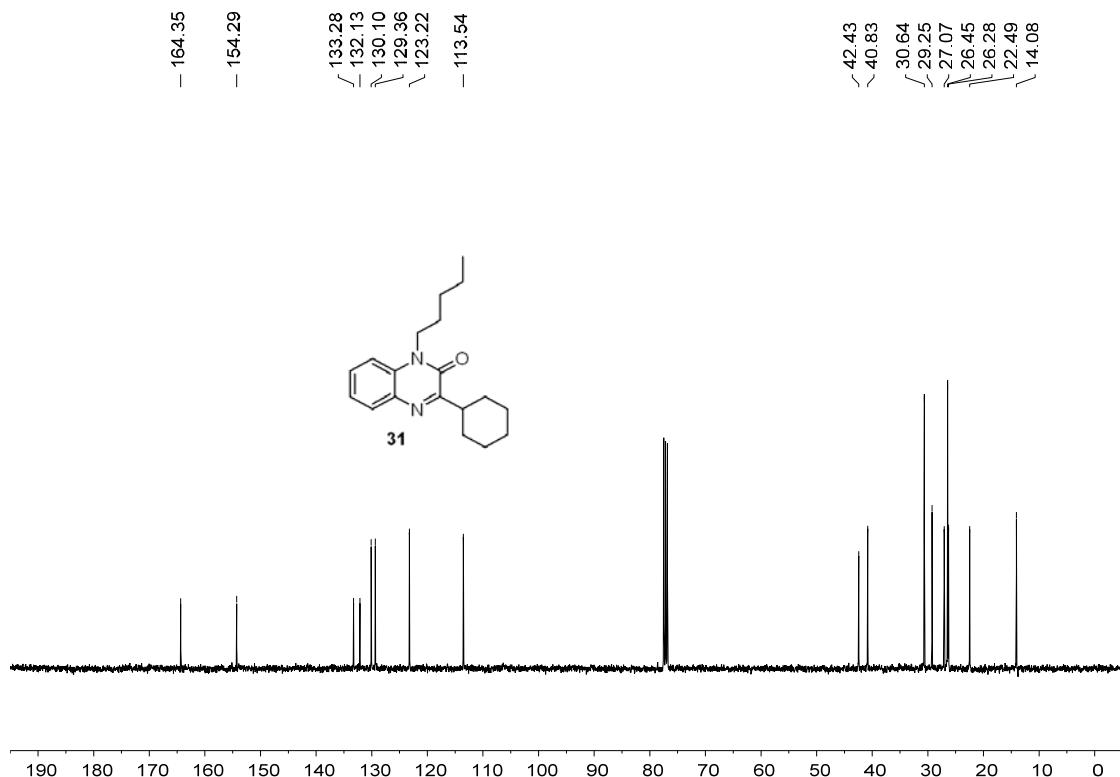
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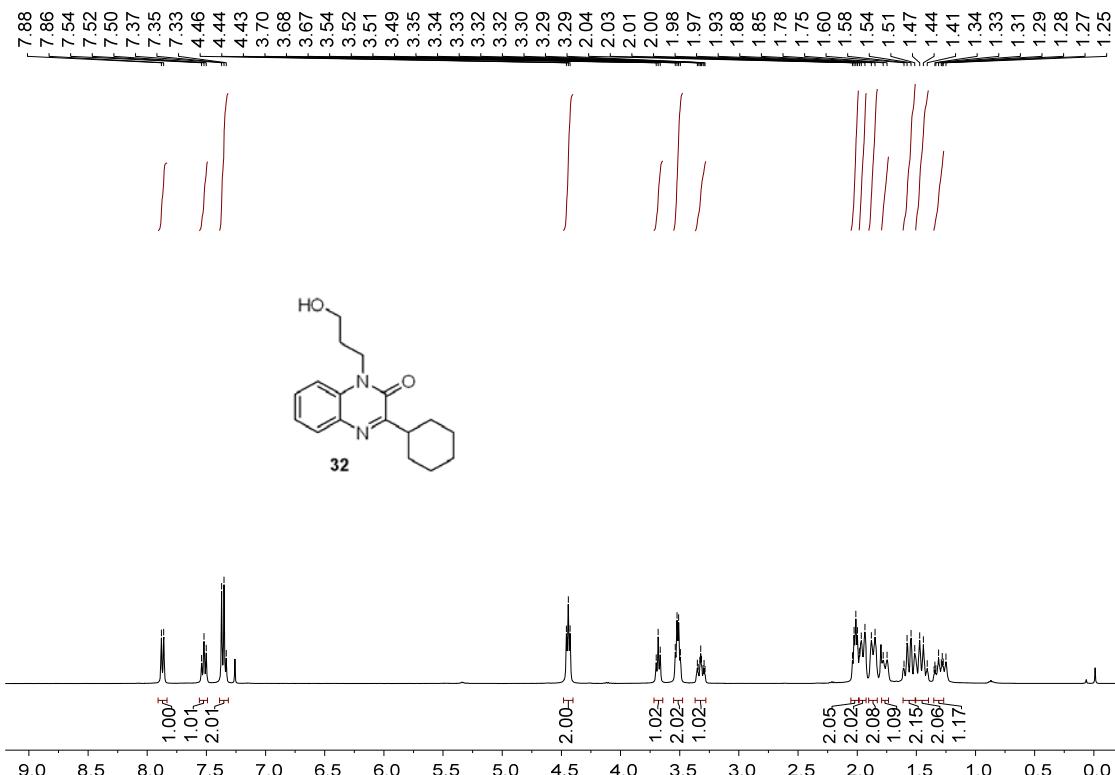
¹H NMR for **31** (400 MHz, CDCl₃)



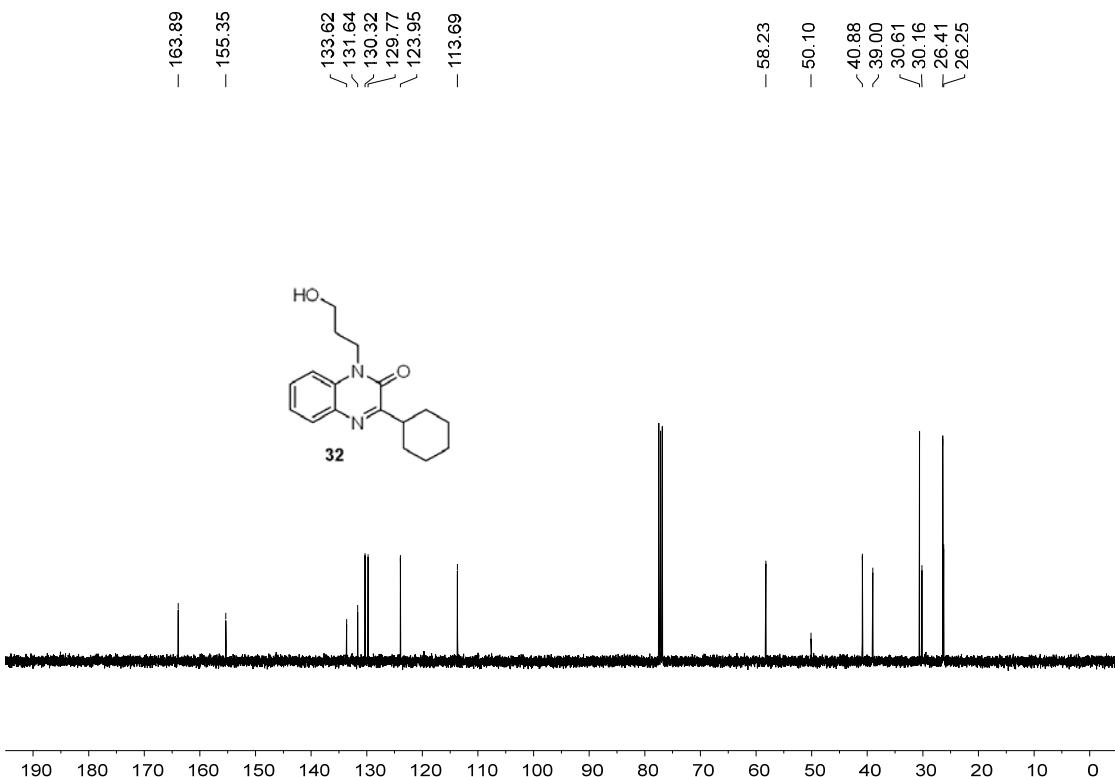
¹³C NMR for **31** (101 MHz, CDCl₃)



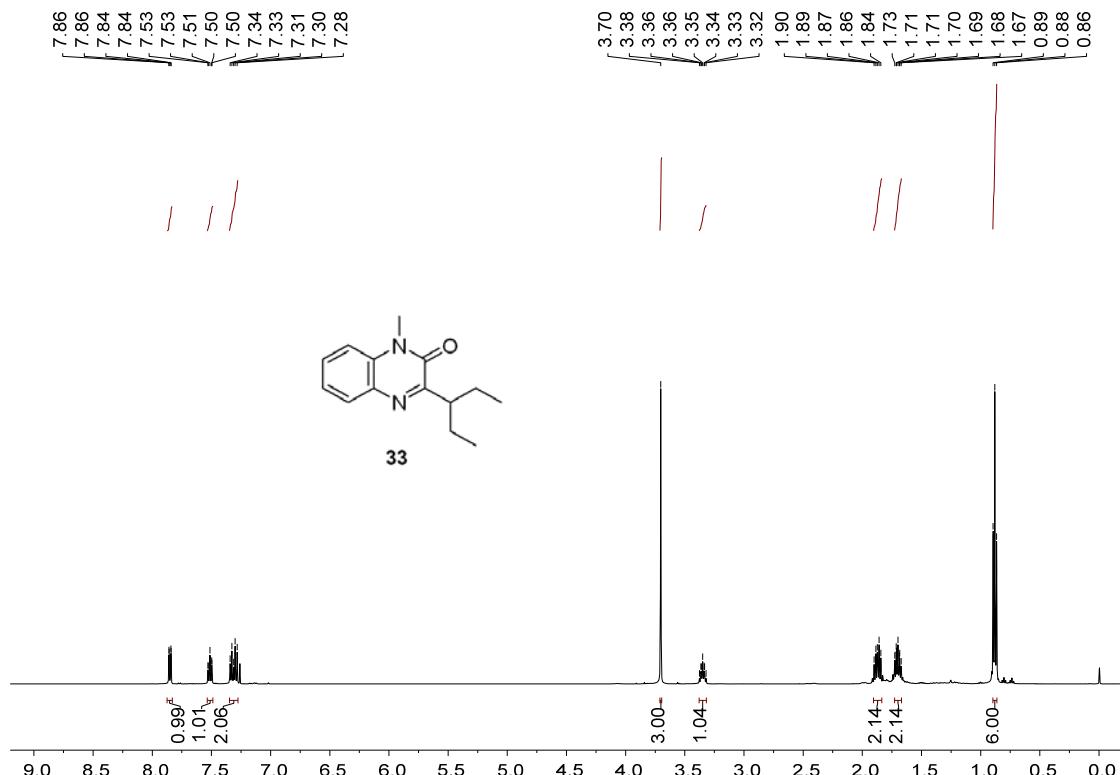
¹H NMR for **32** (400 MHz, CDCl₃)



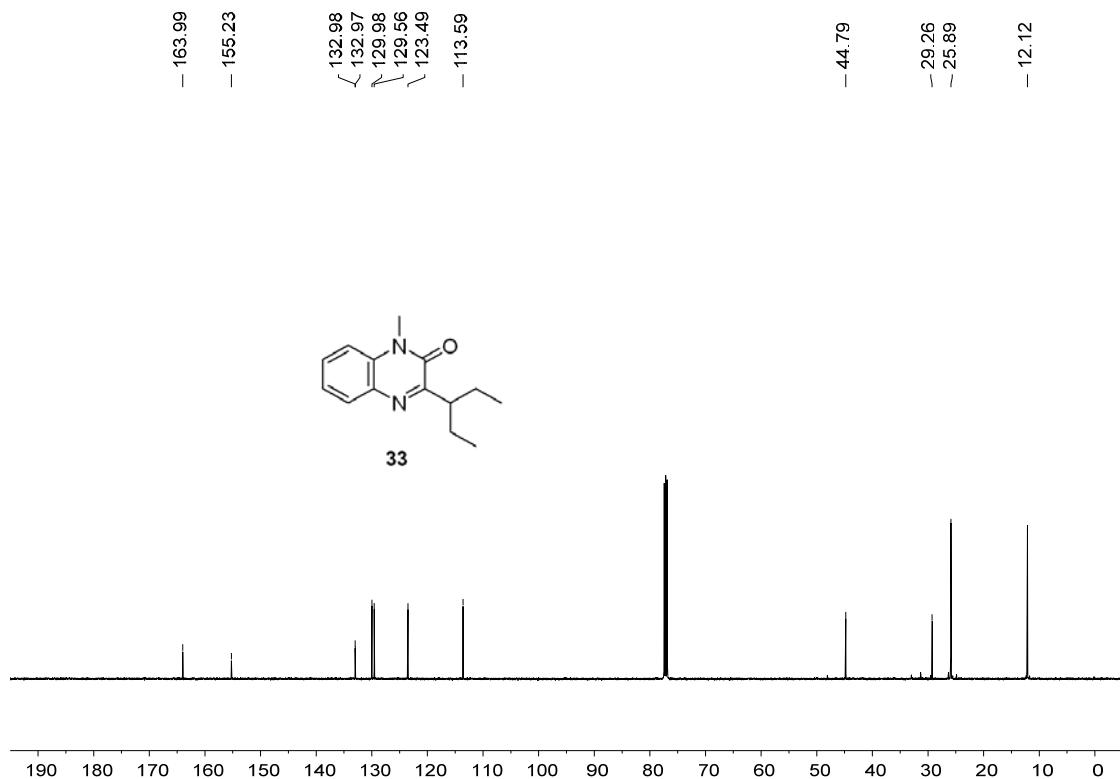
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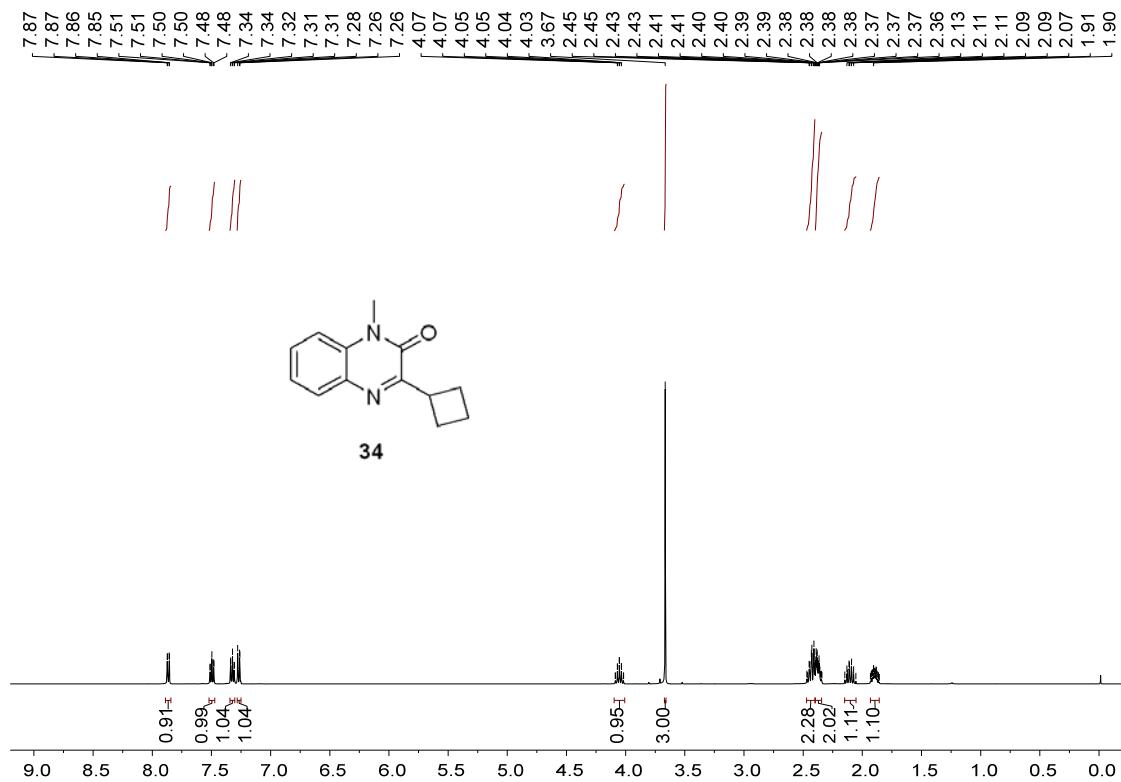
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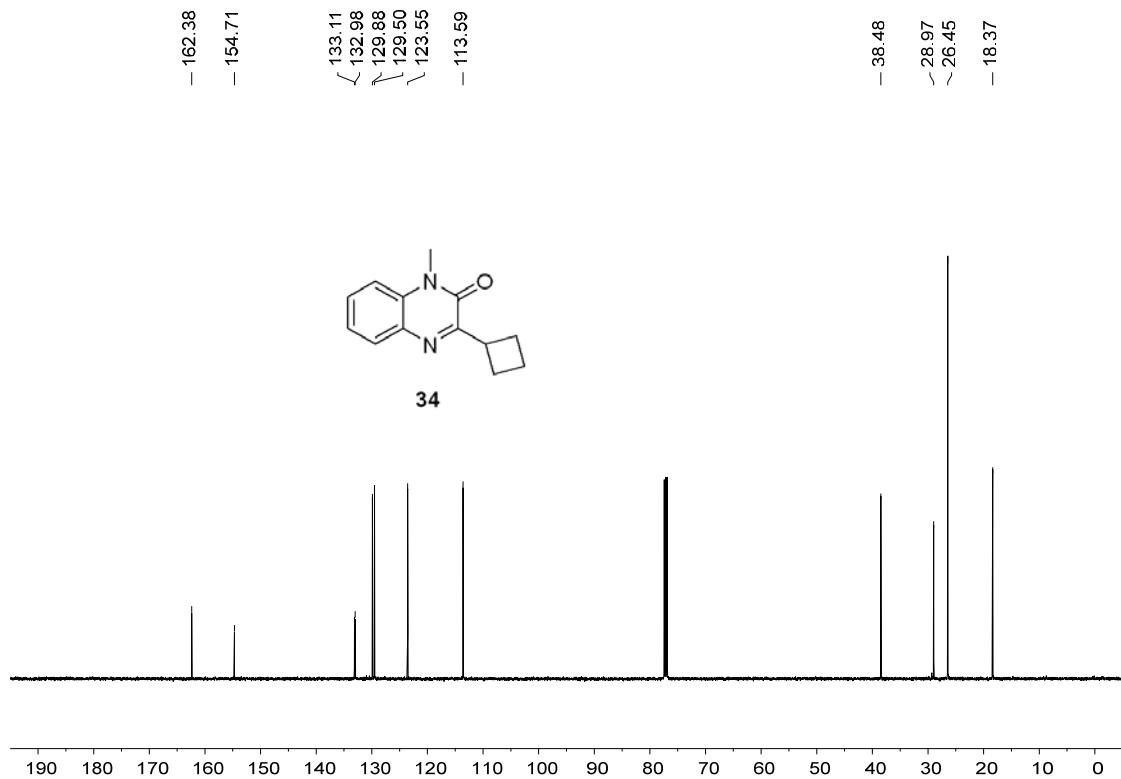
¹³C NMR for **33** (126 MHz, CDCl₃)



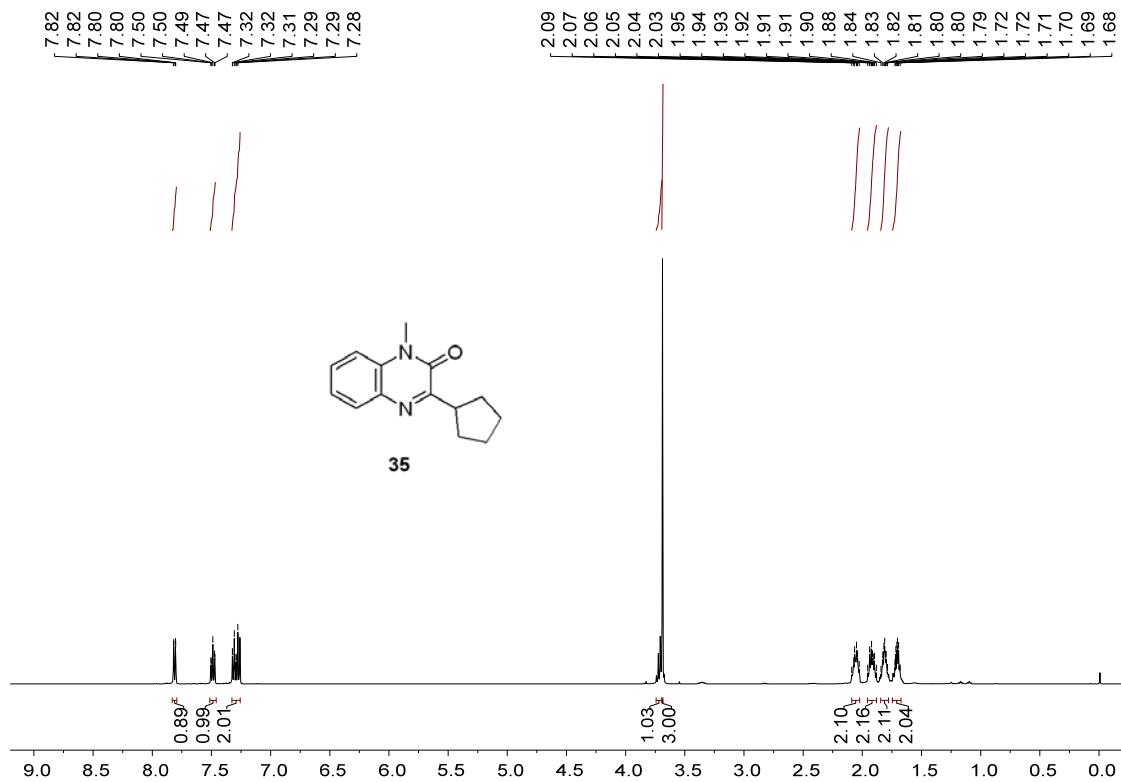
¹H NMR for **34** (500 MHz, CDCl₃)



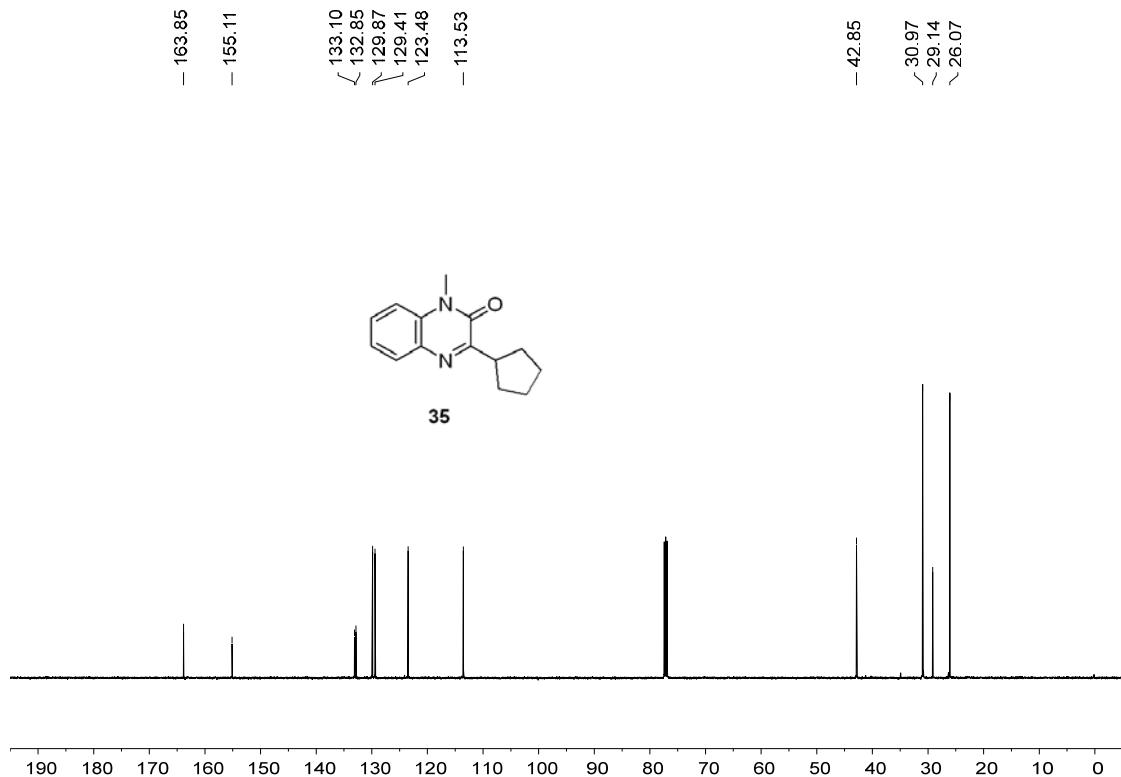
¹³C NMR for **34** (126 MHz, CDCl₃)



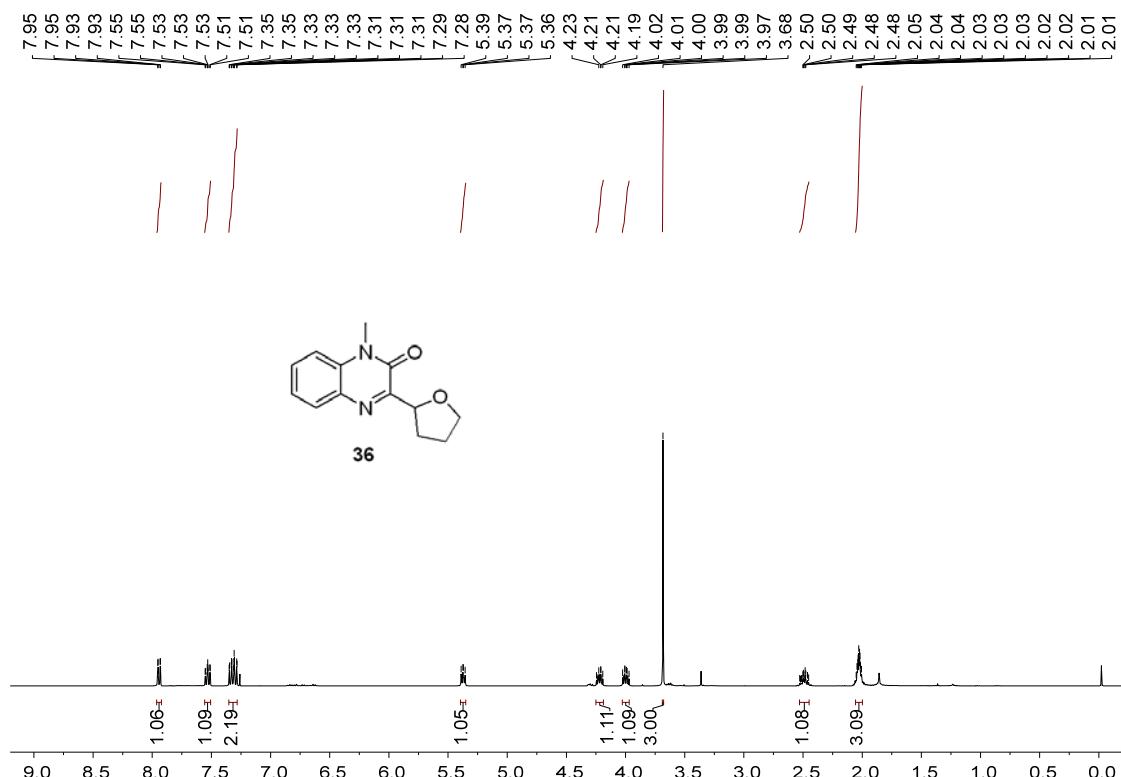
¹H NMR for **35** (500 MHz, CDCl₃)



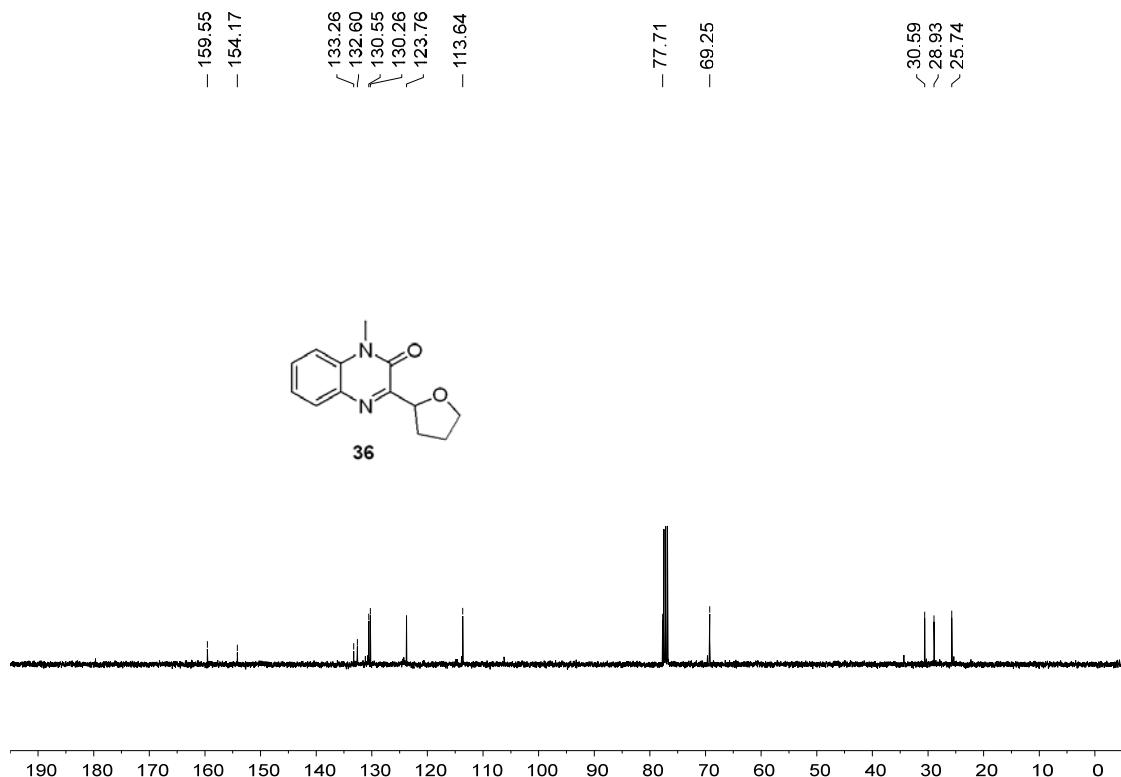
¹³C NMR for **35** (126 MHz, CDCl₃)



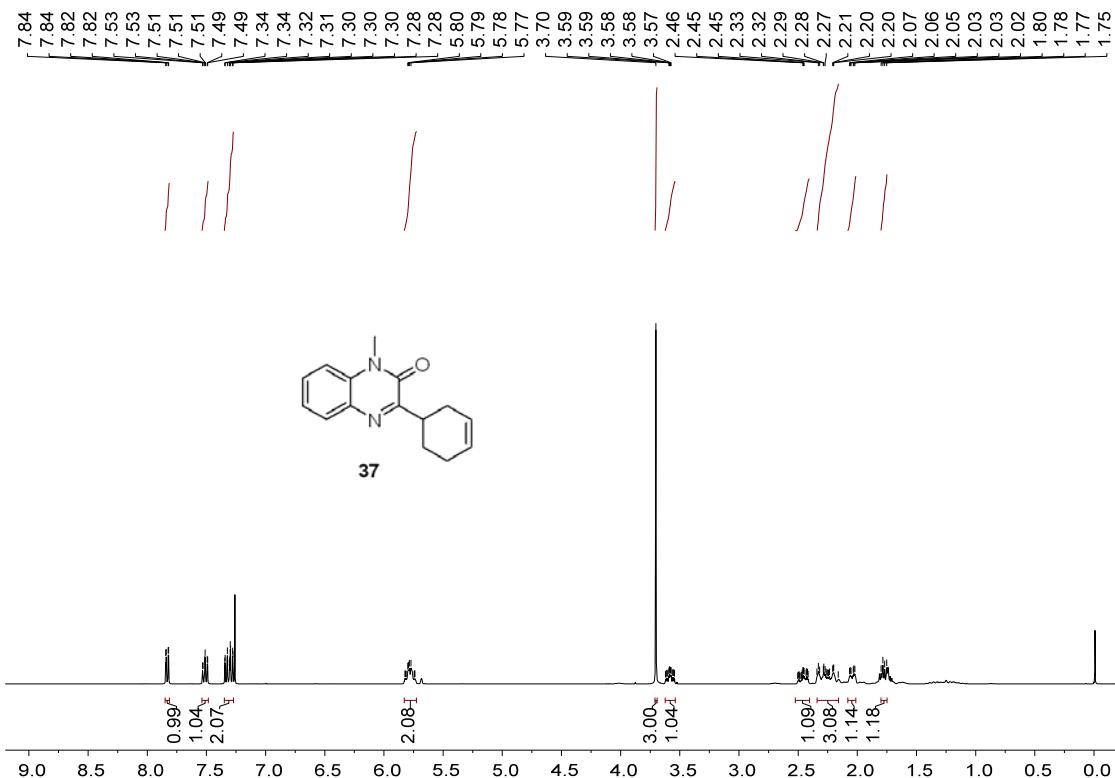
¹H NMR for **36** (400 MHz, CDCl₃)



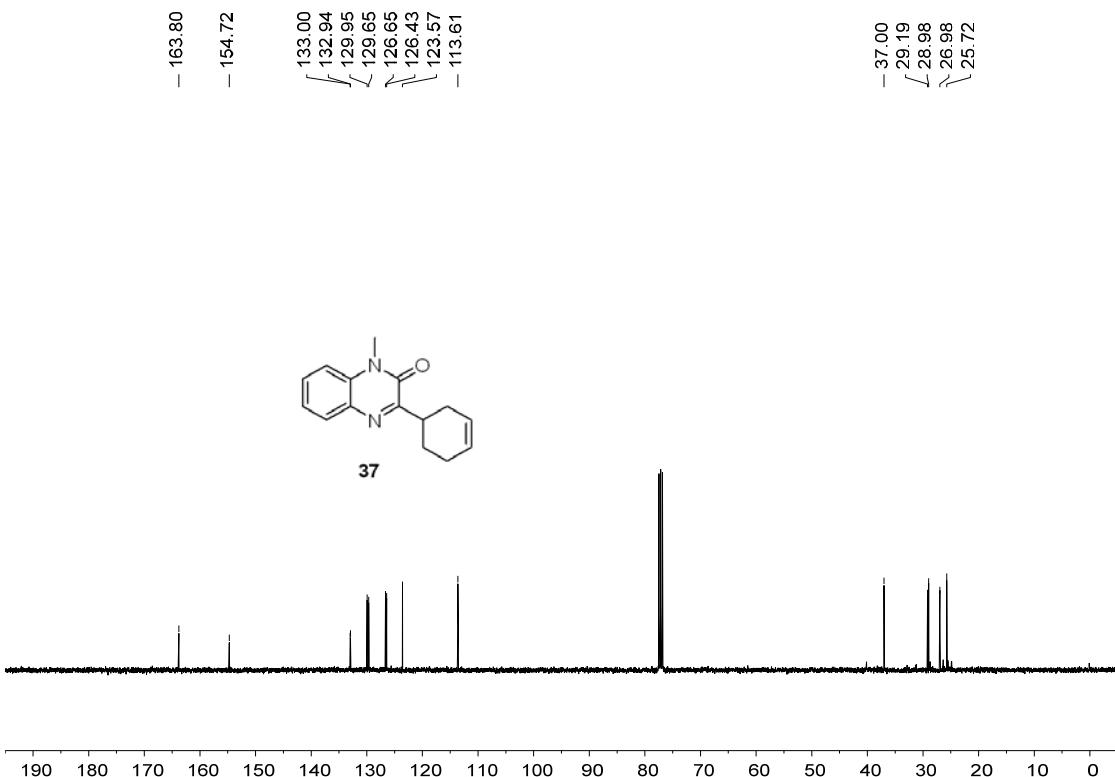
¹³C NMR for **36** (101 MHz, CDCl₃)



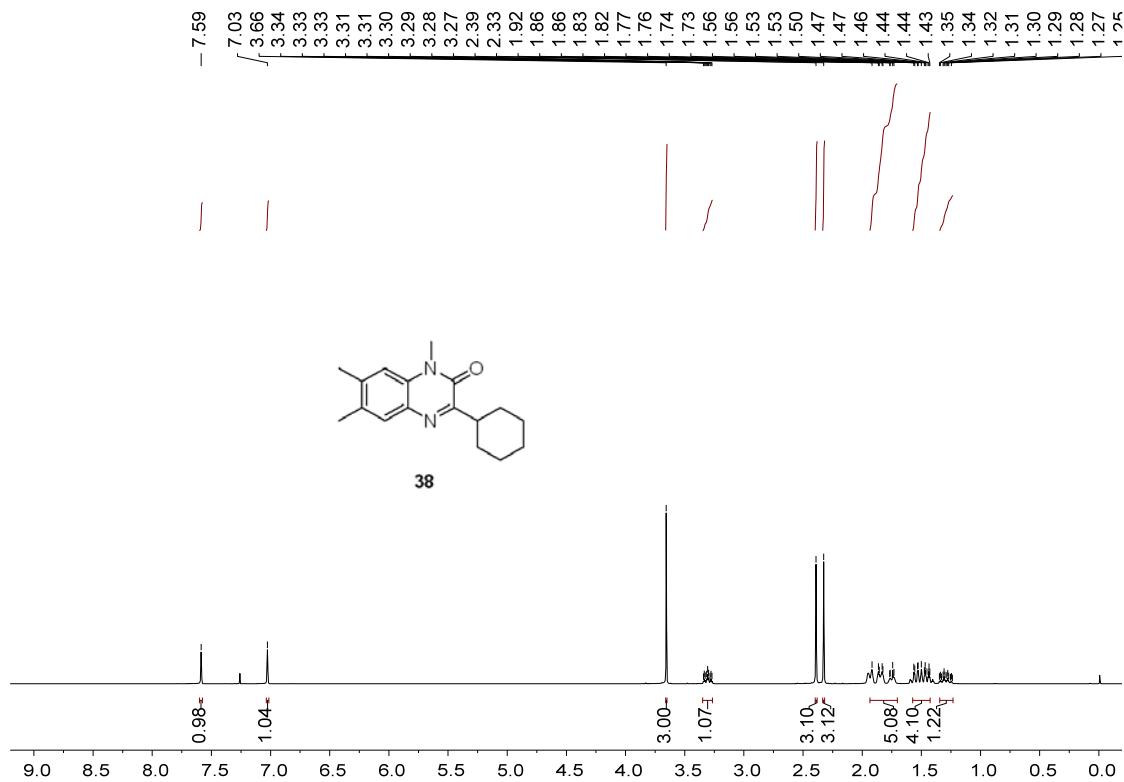
¹H NMR for **37** (400 MHz, CDCl₃)



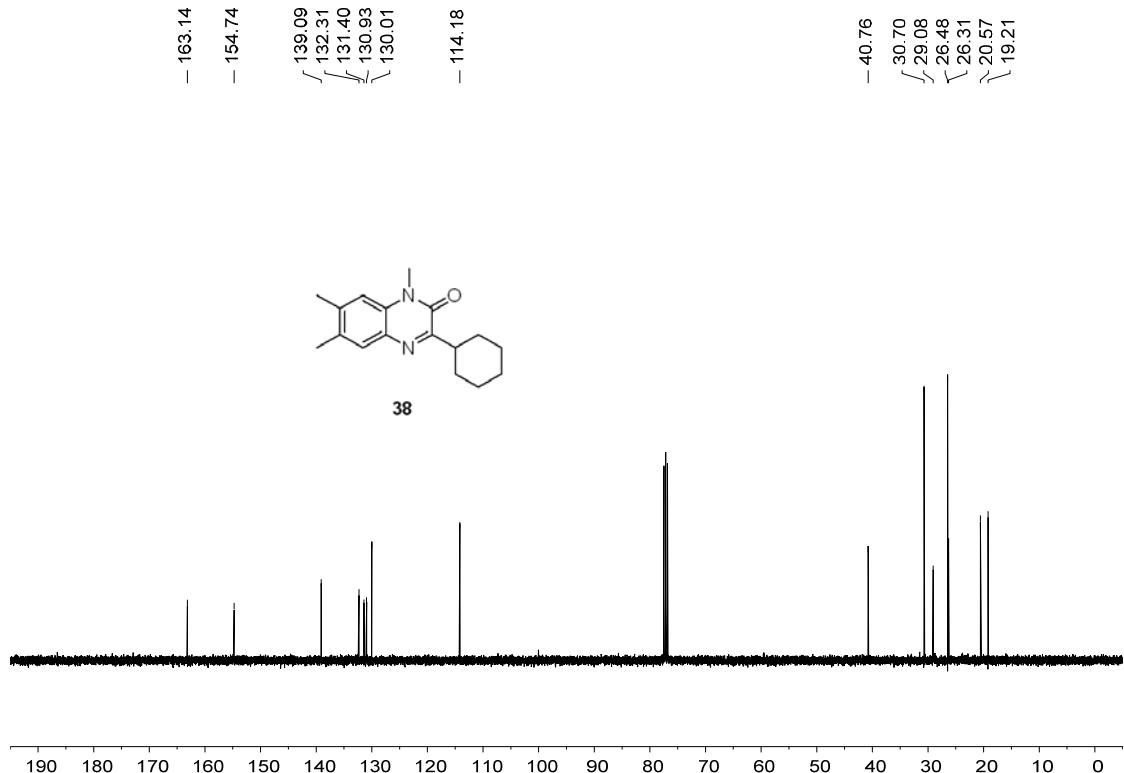
¹³C NMR for **37** (101 MHz, CDCl₃)



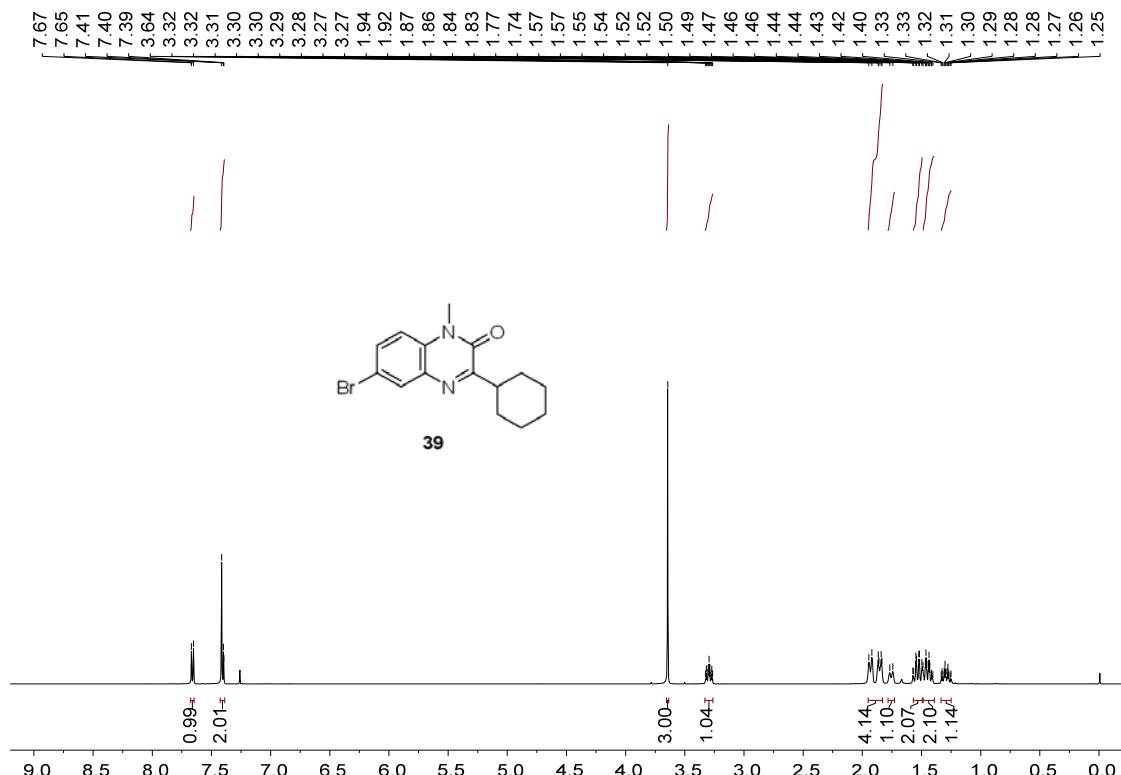
¹H NMR for **38** (400 MHz, CDCl₃)



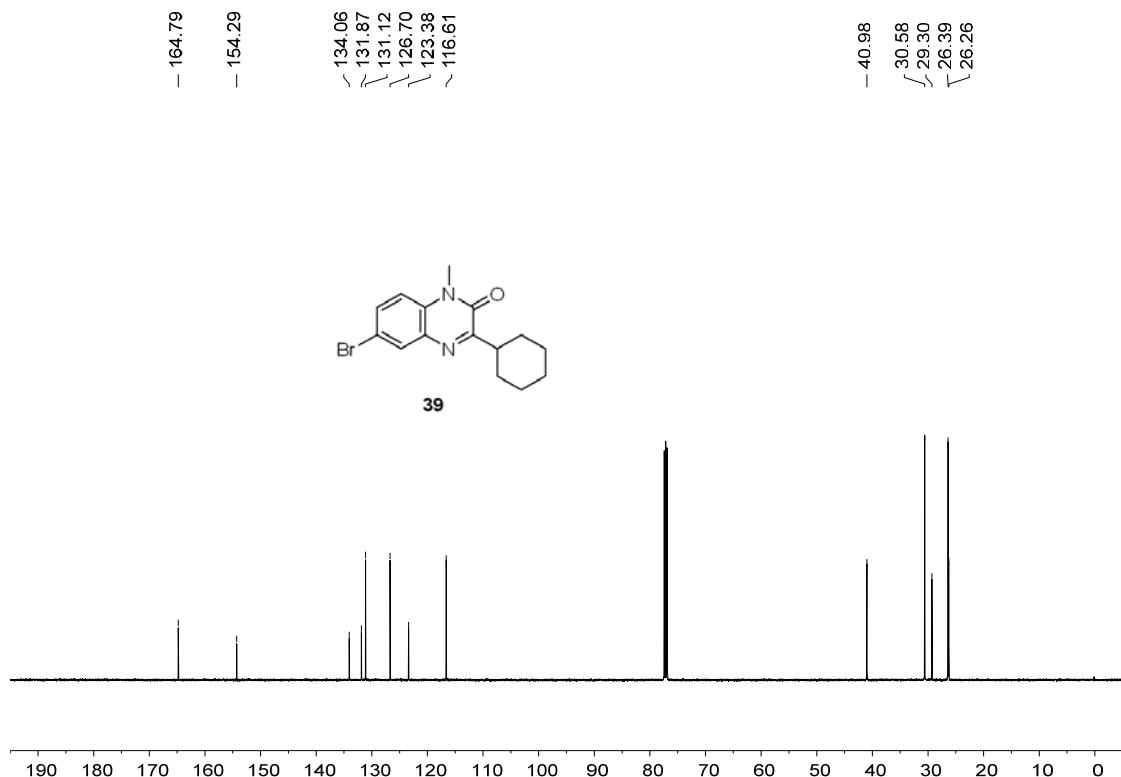
¹³C NMR for **38** (101 MHz, CDCl₃)



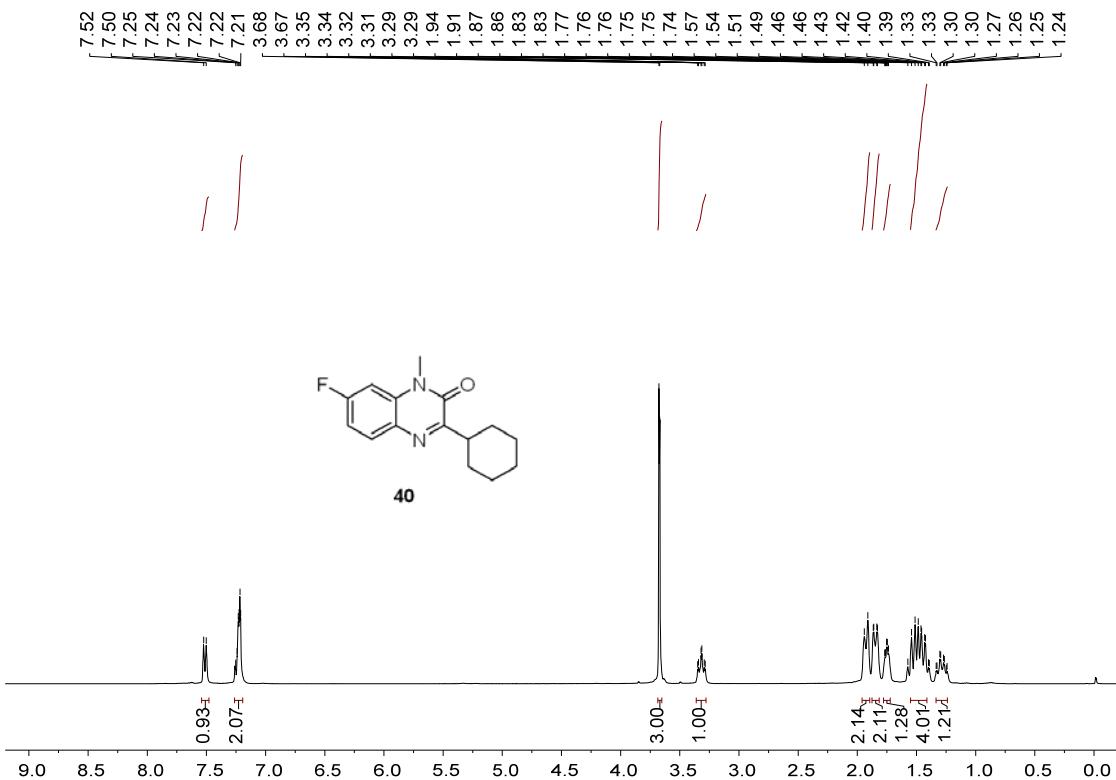
¹H NMR for **39** (500 MHz, CDCl₃)



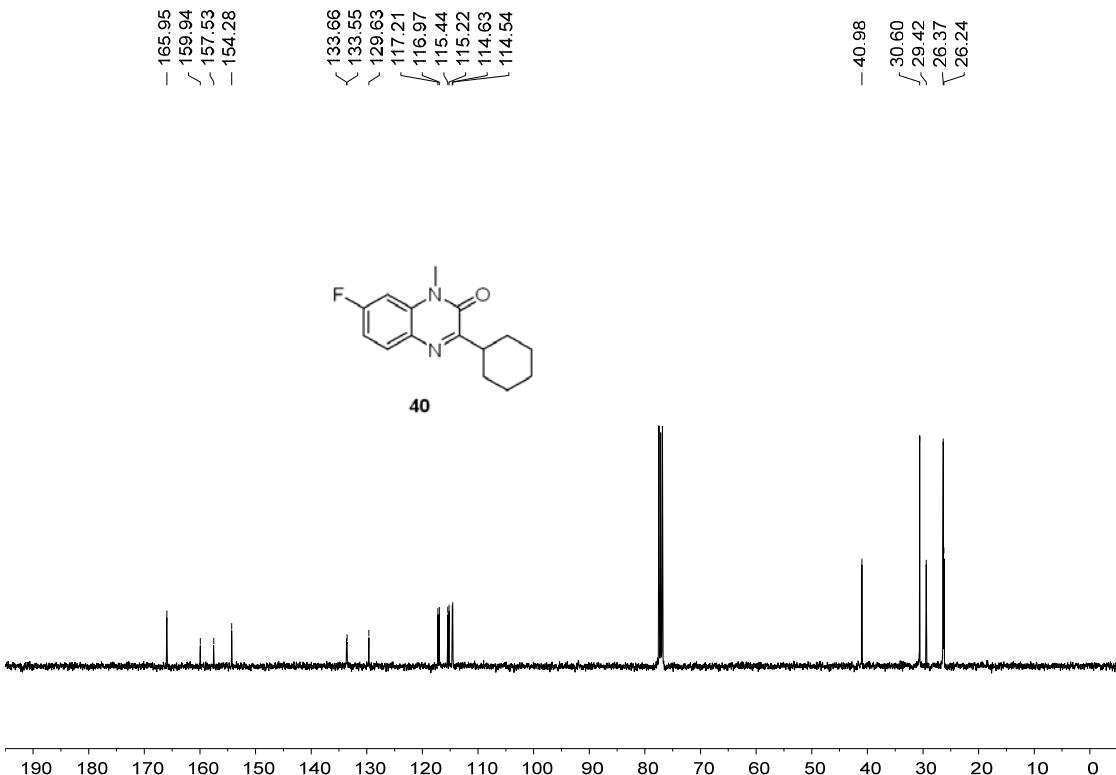
¹³C NMR for **39** (126 MHz, CDCl₃)



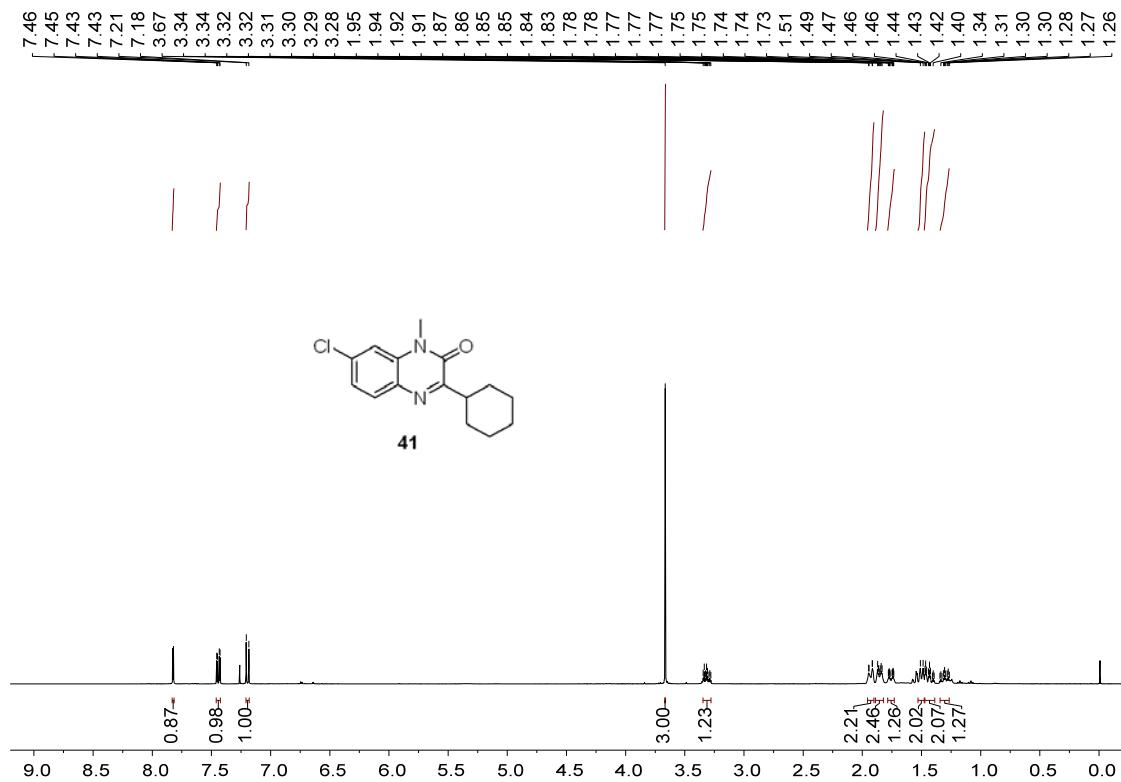
¹H NMR for **40** (400 MHz, CDCl₃)



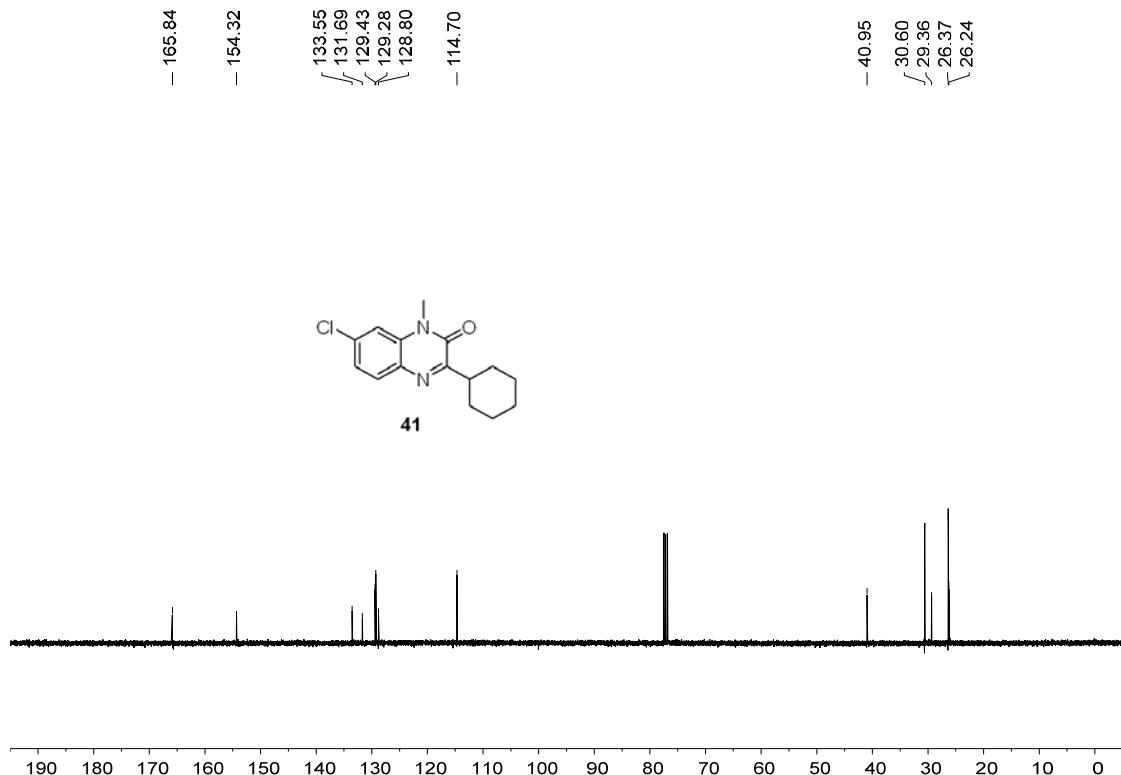
¹³C NMR for **40** (101 MHz, CDCl₃)



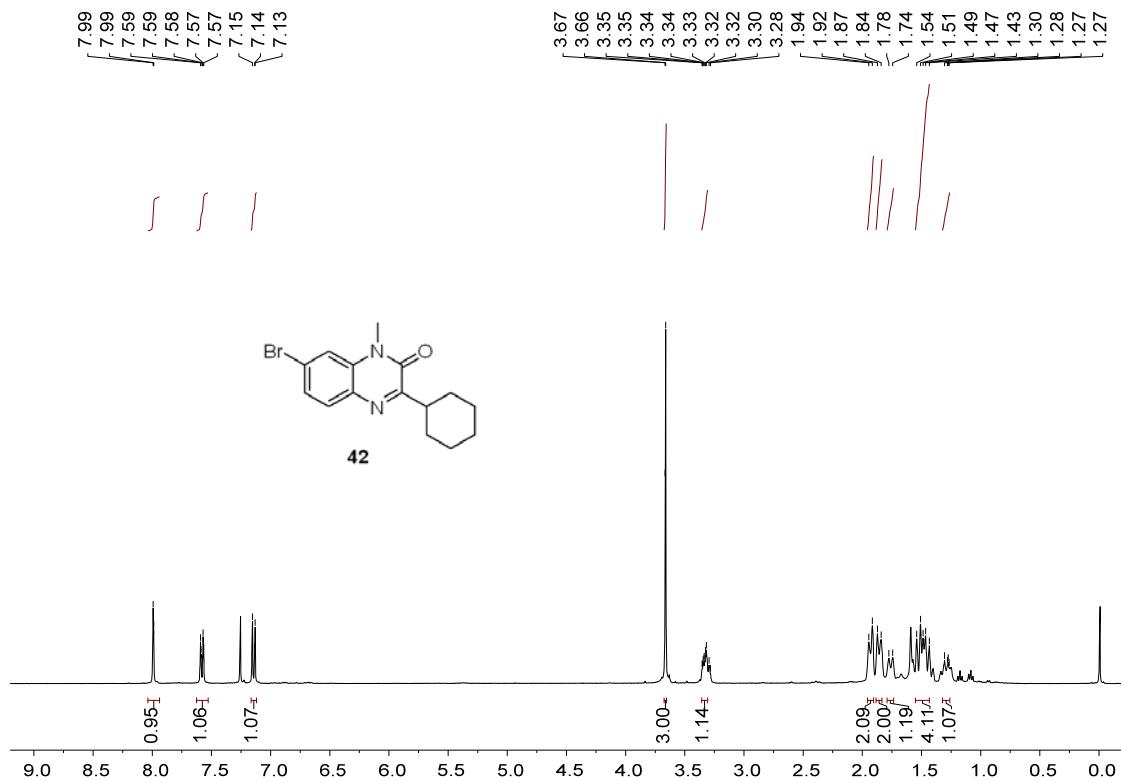
¹H NMR for **41** (400 MHz, CDCl₃)



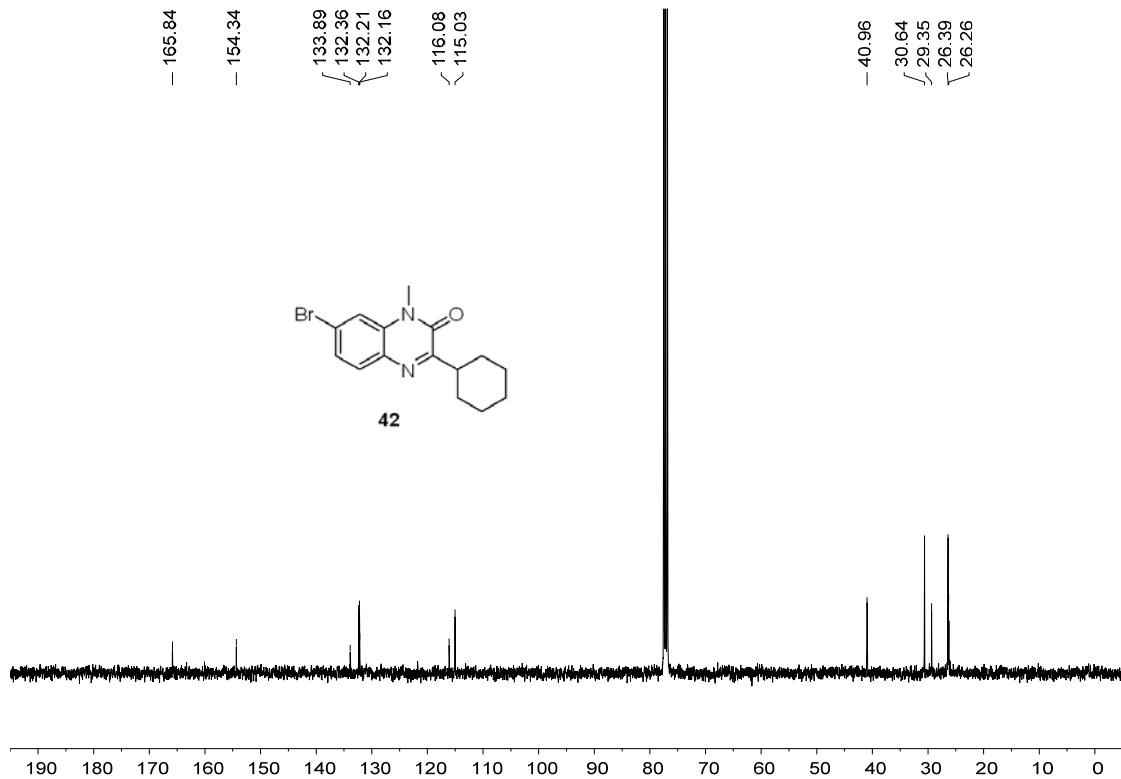
¹³C NMR for **41** (101 MHz, CDCl₃)



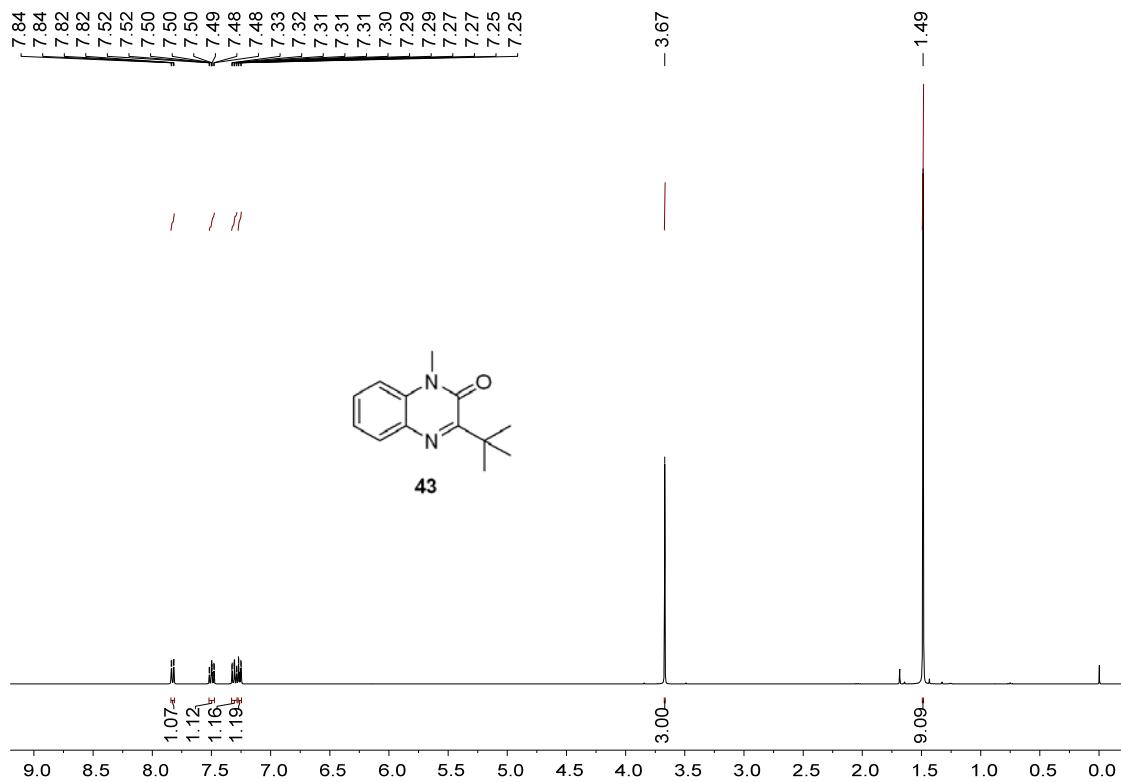
¹H NMR for **42** (400 MHz, CDCl₃)



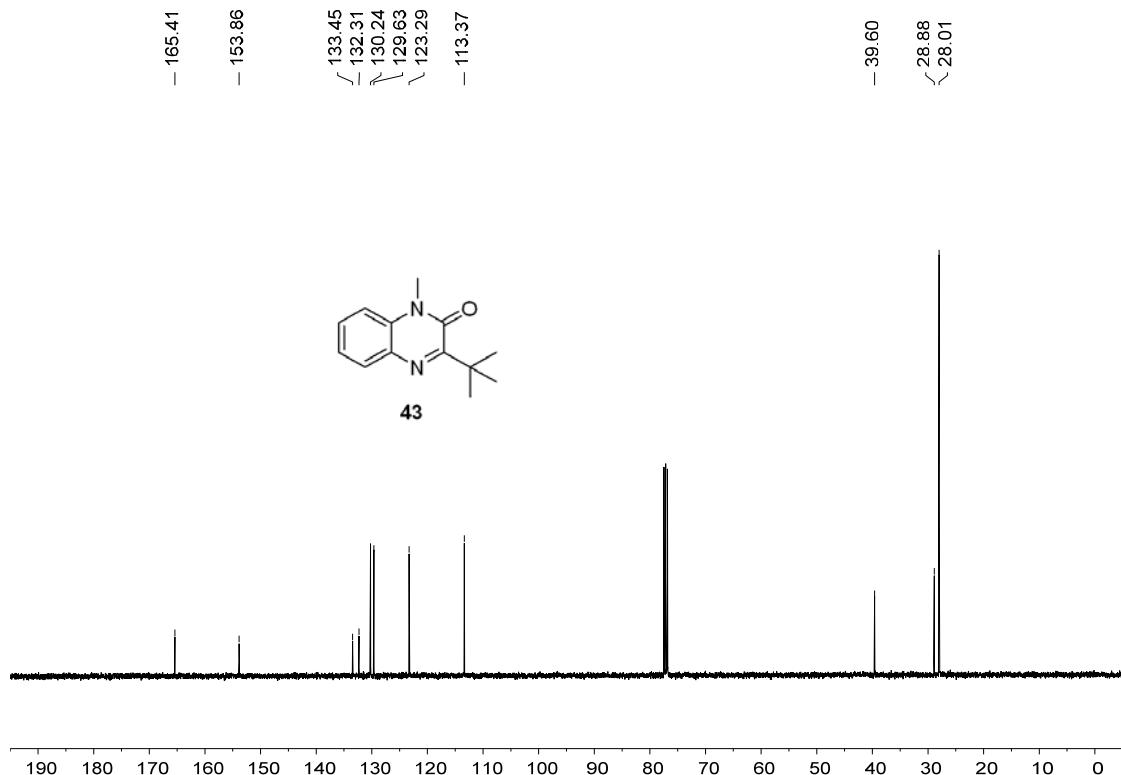
¹³C NMR for **42** (101 MHz, CDCl₃)



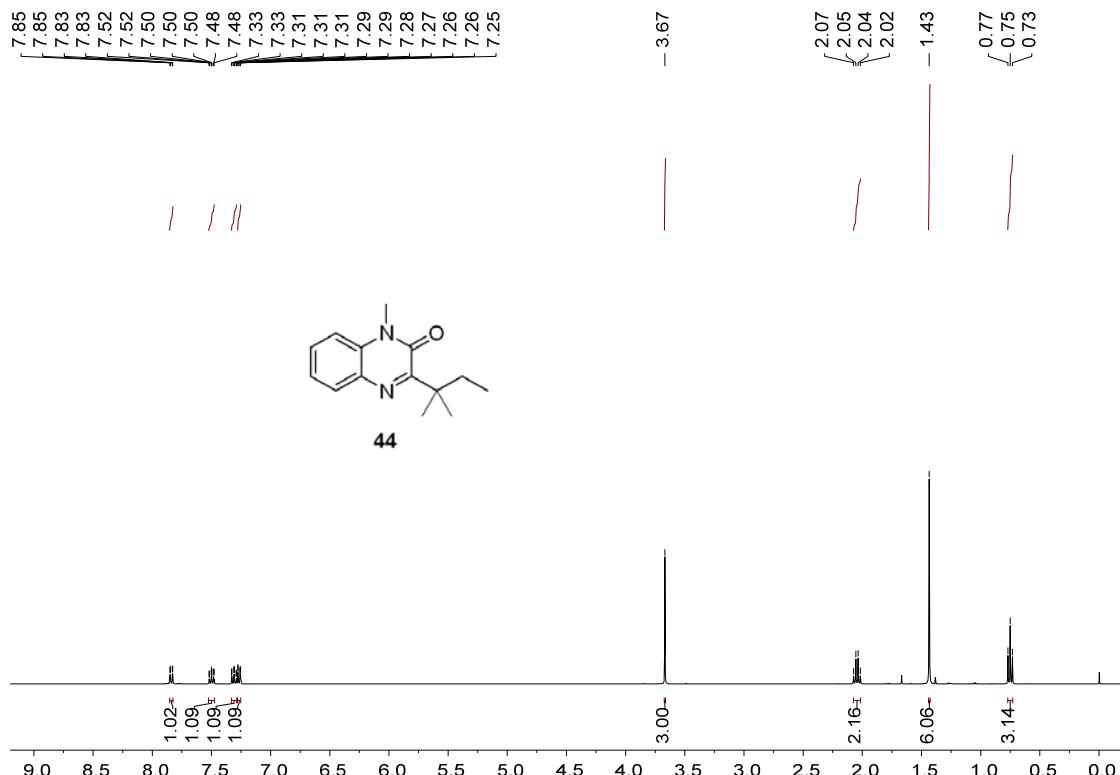
¹H NMR for **43** (400 MHz, CDCl₃)



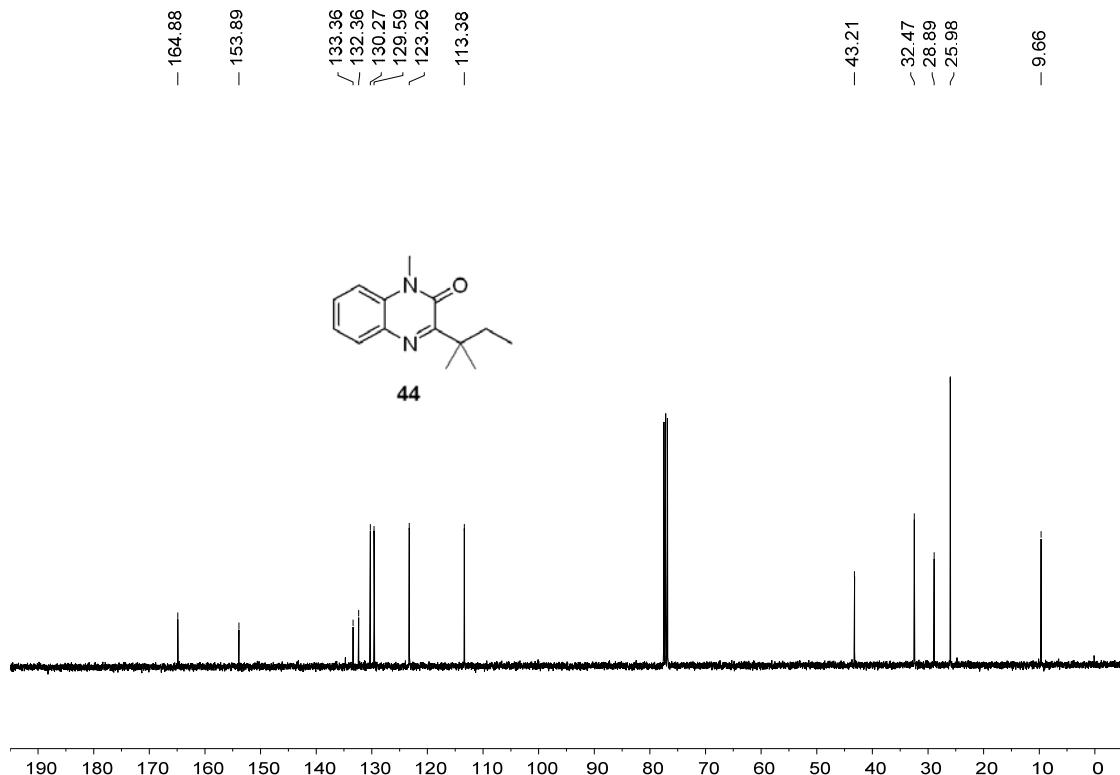
¹³C NMR for **43** (101 MHz, CDCl₃)



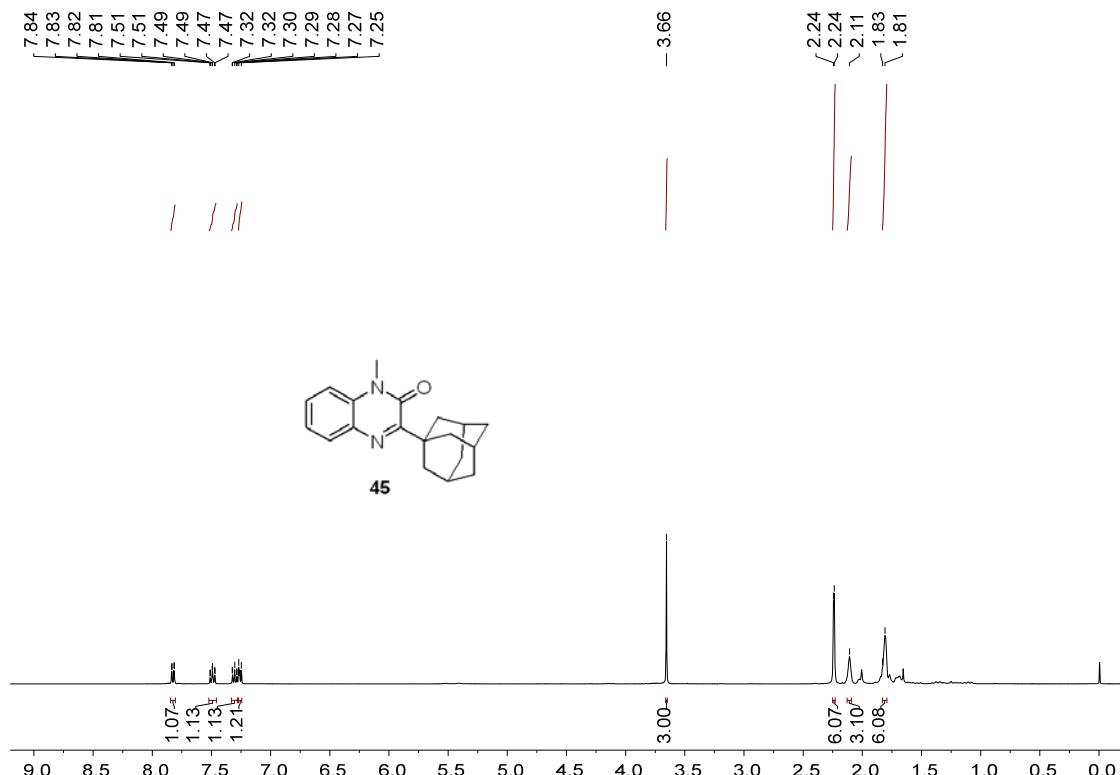
¹H NMR for **44** (400 MHz, CDCl₃)



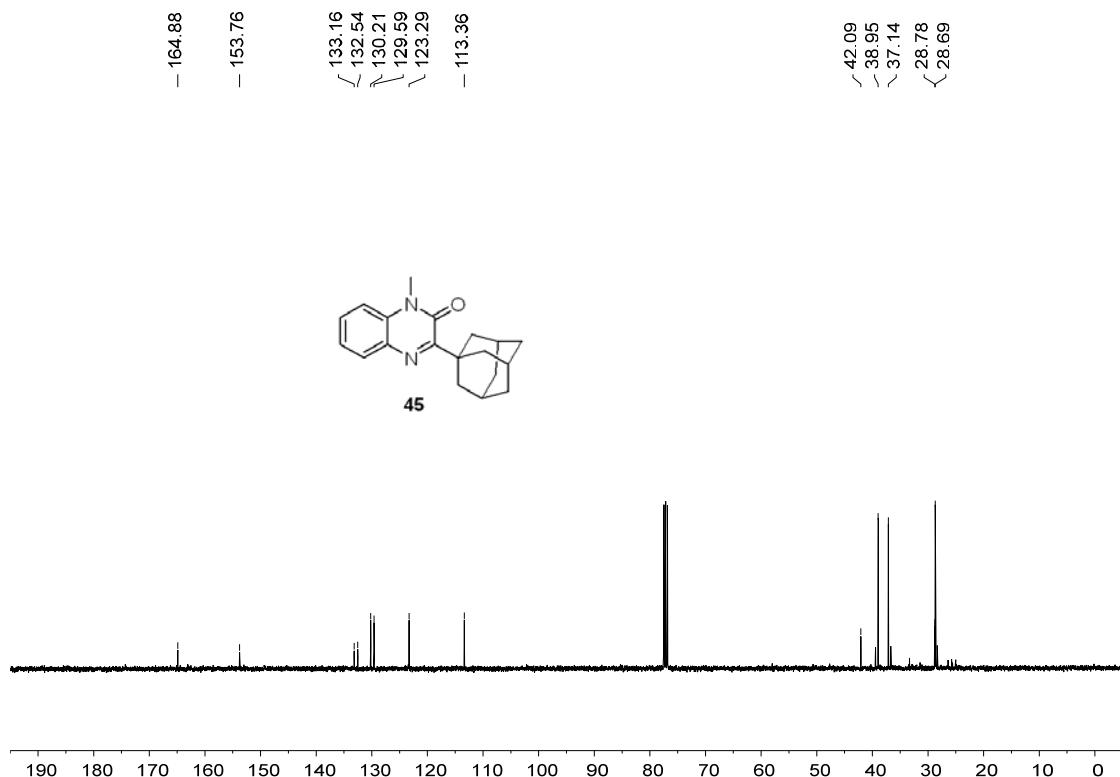
¹³C NMR for **44** (101 MHz, CDCl₃)



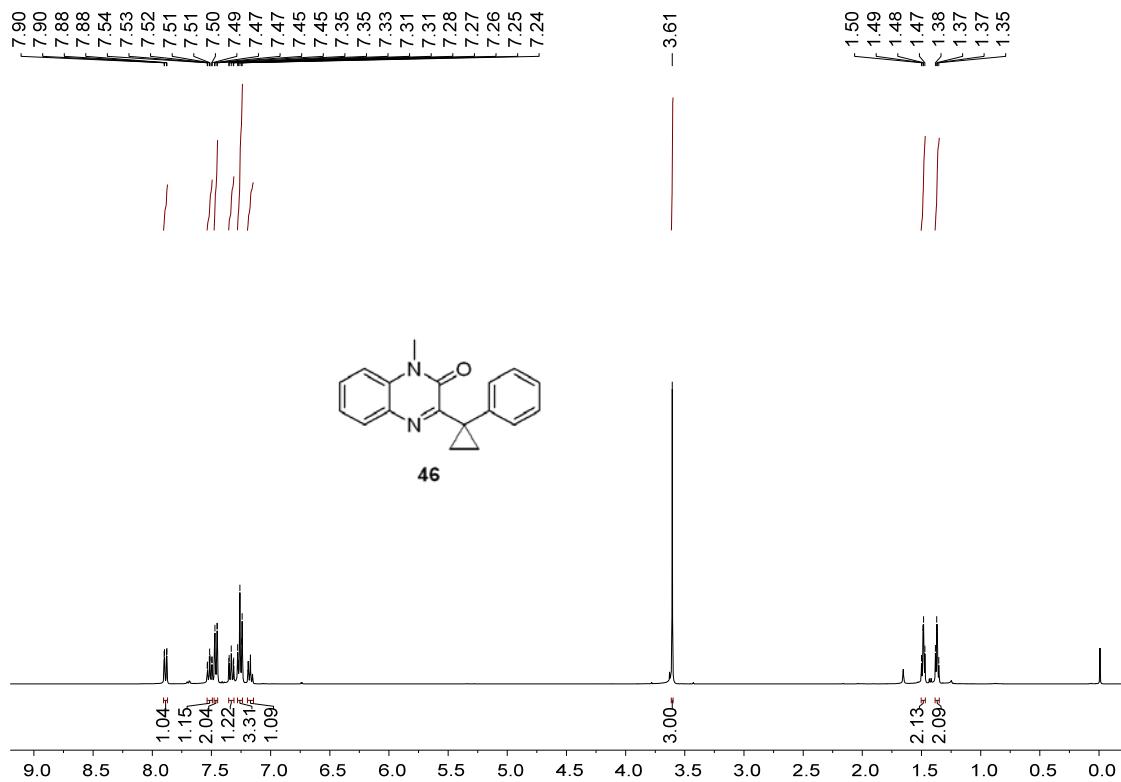
¹H NMR for **45** (400 MHz, CDCl₃)



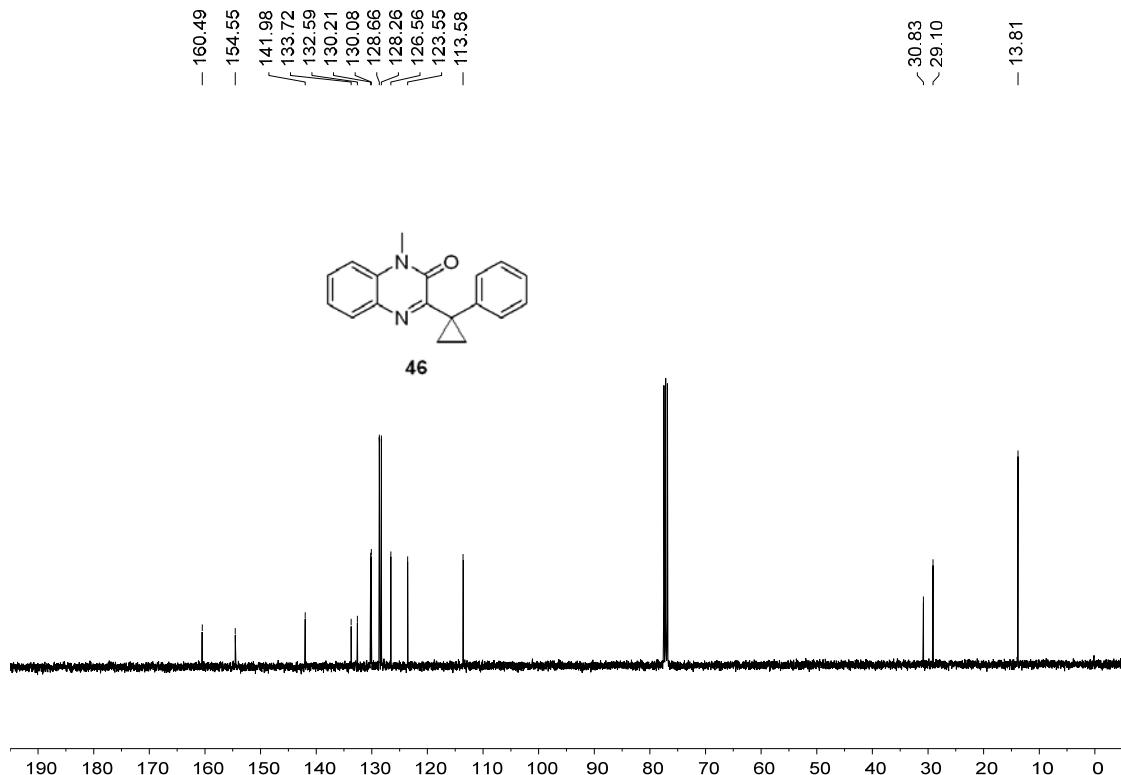
¹³C NMR for **45** (101 MHz, CDCl₃)



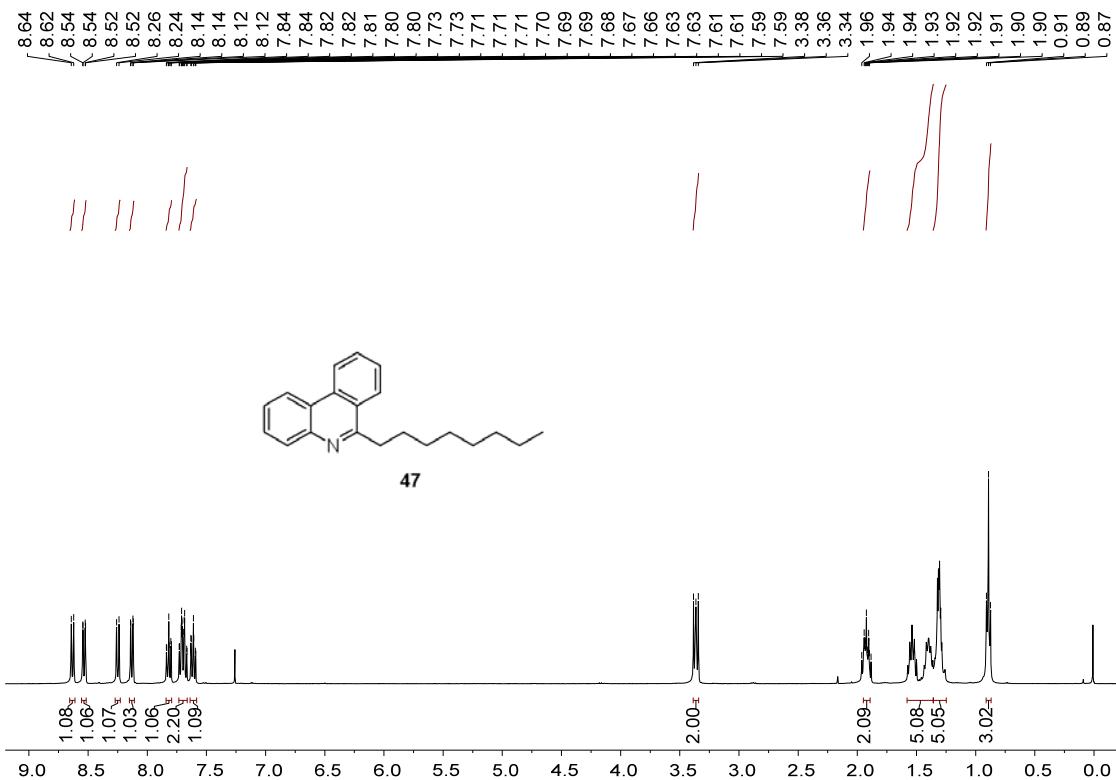
¹H NMR for **46** (400 MHz, CDCl₃)



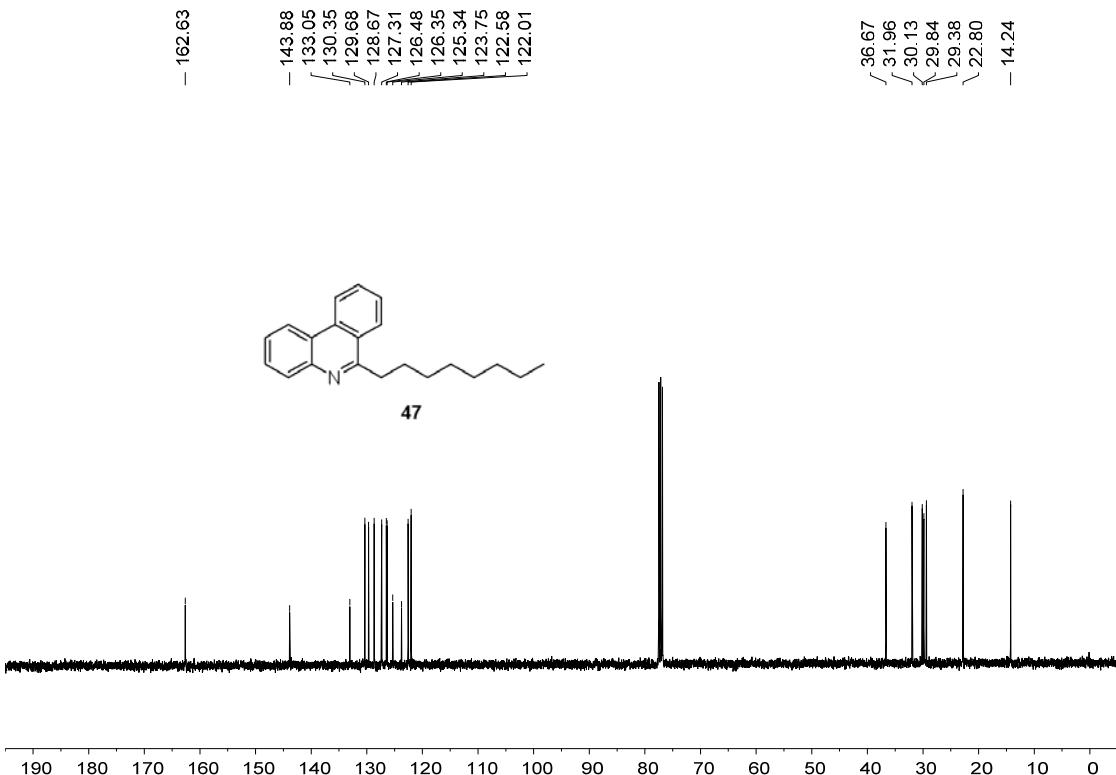
¹³C NMR for **46** (101 MHz, CDCl₃)



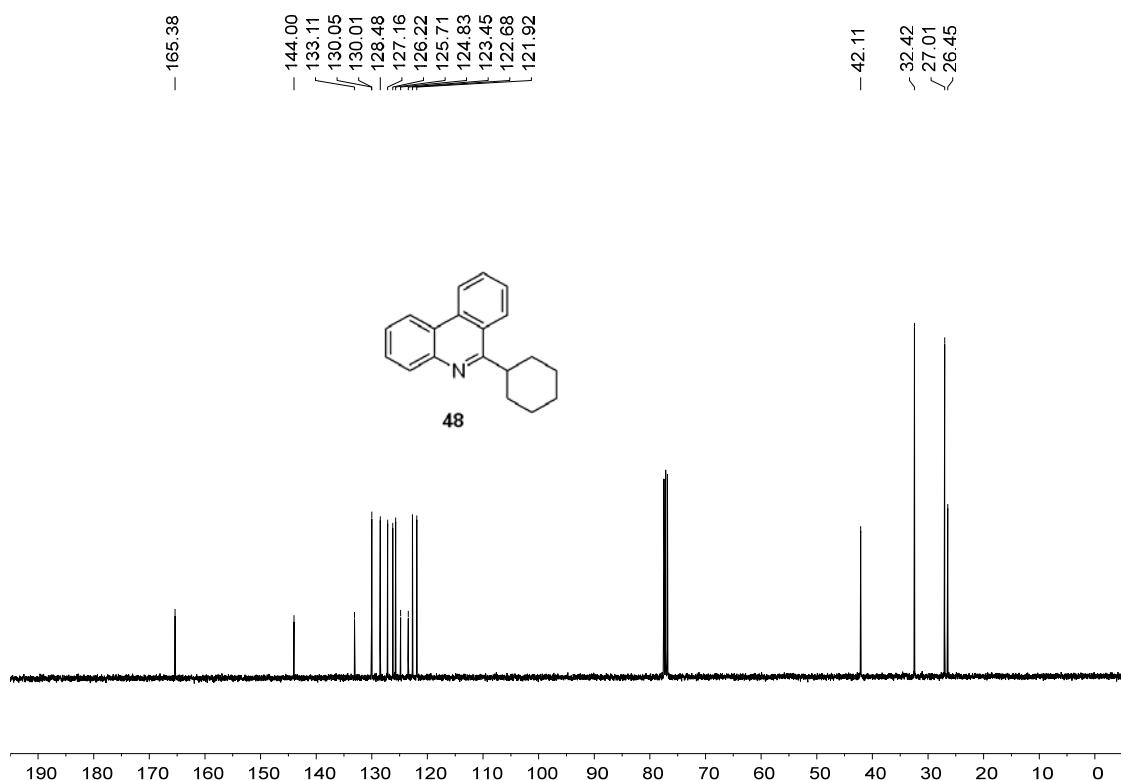
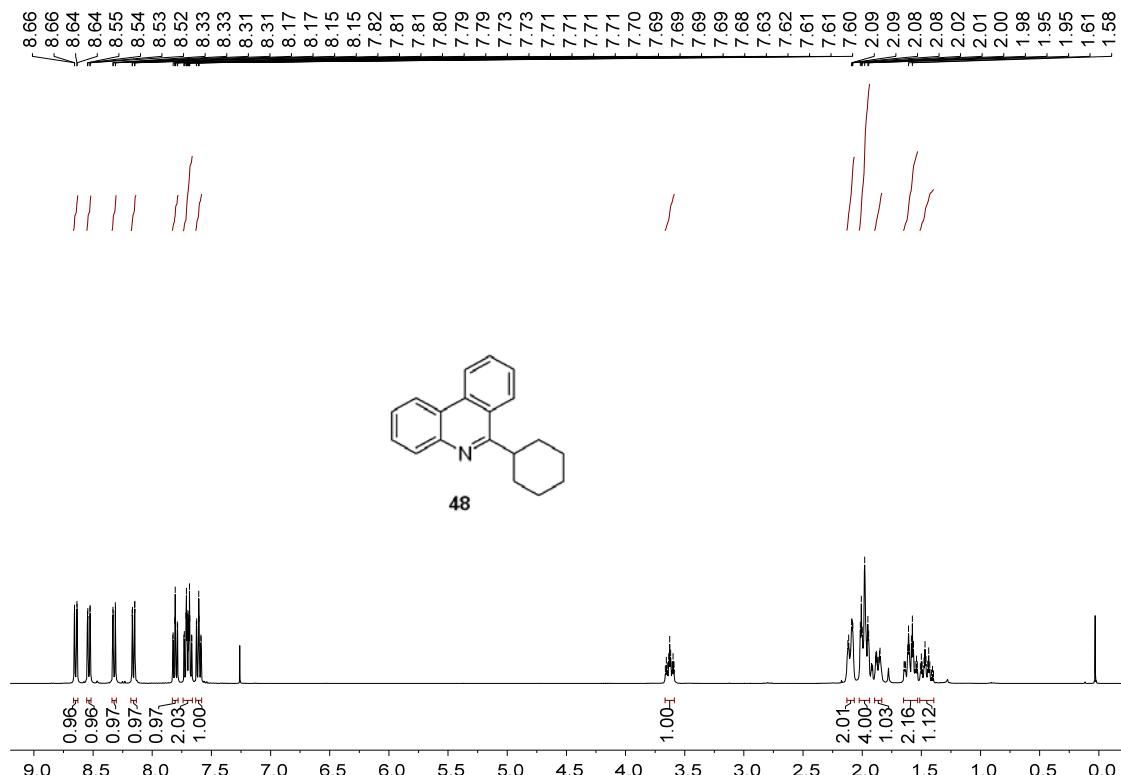
¹H NMR for **47** (400 MHz, CDCl₃)



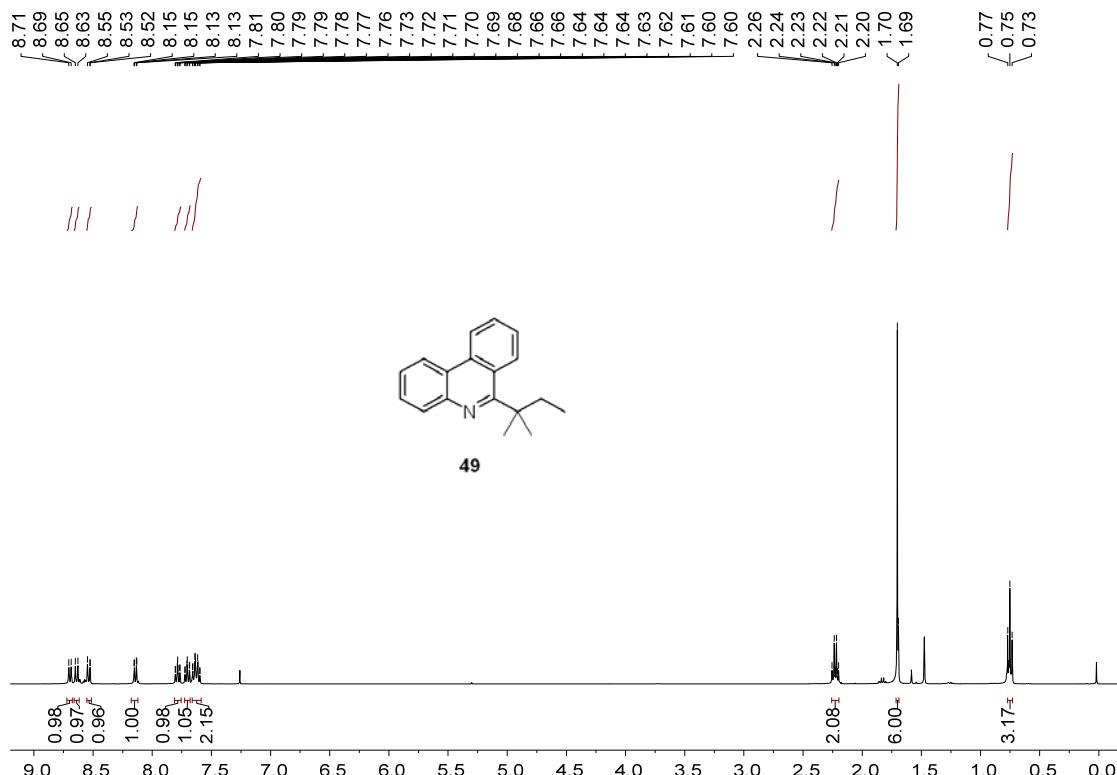
¹³C NMR for **47** (101 MHz, CDCl₃)



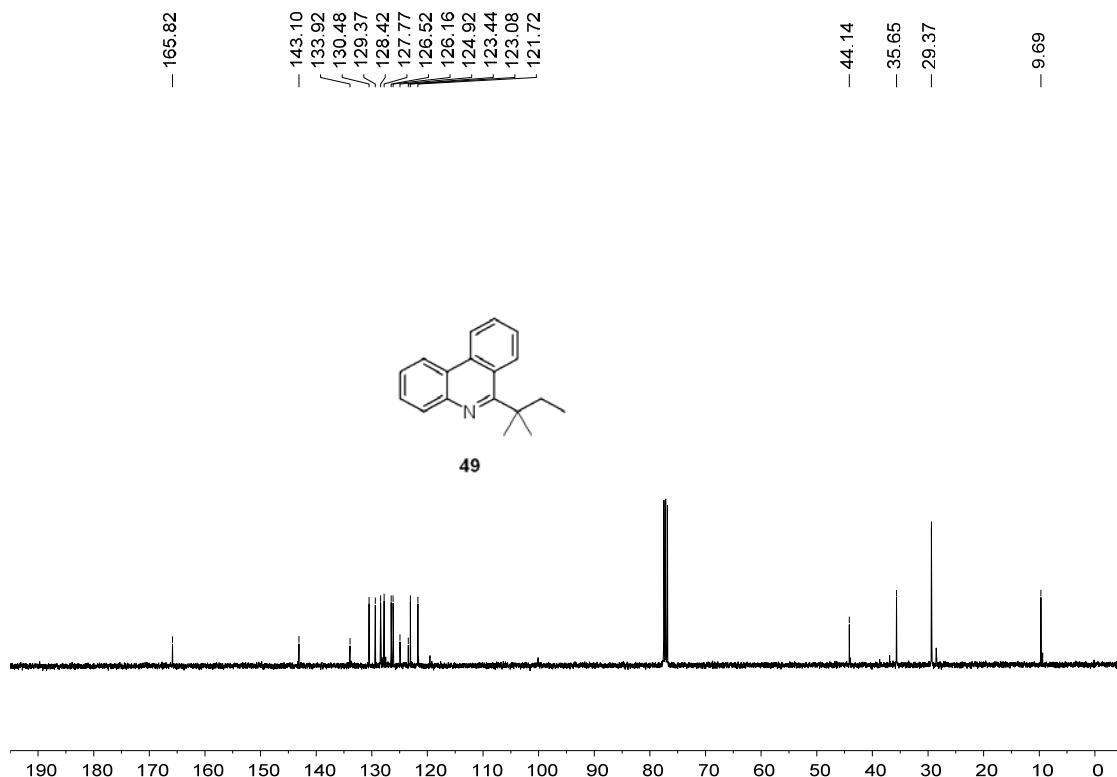
¹H NMR for **48** (400 MHz, CDCl₃)



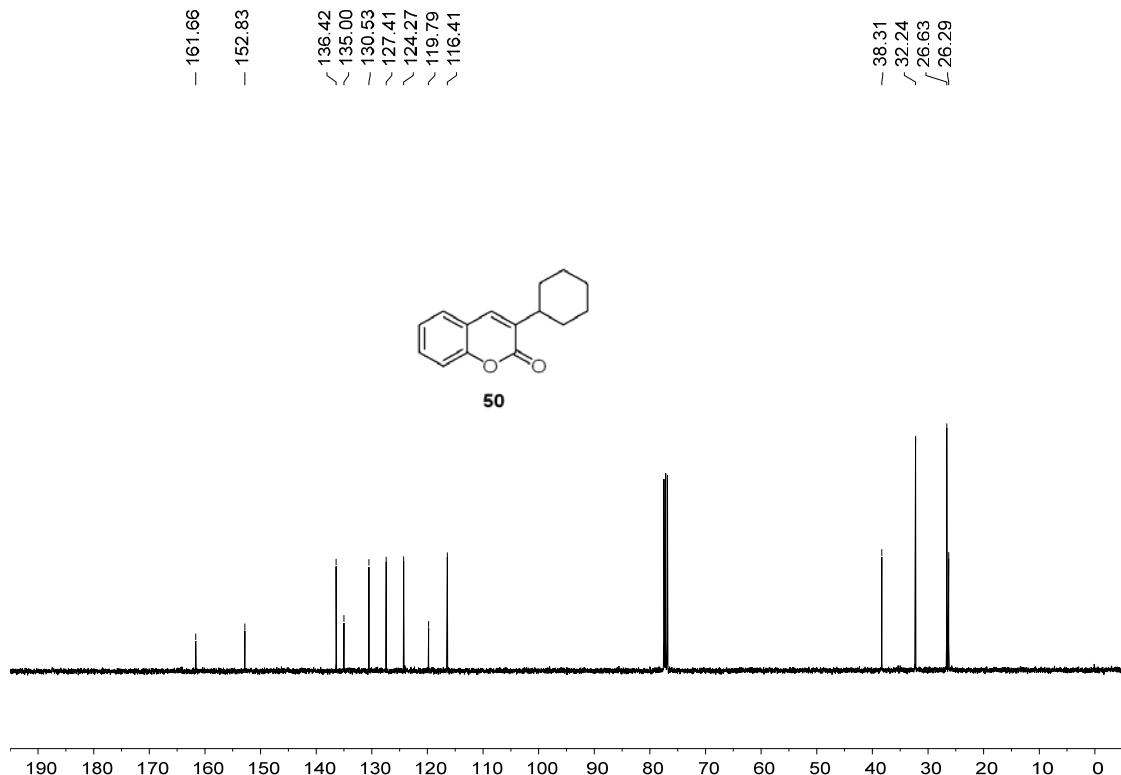
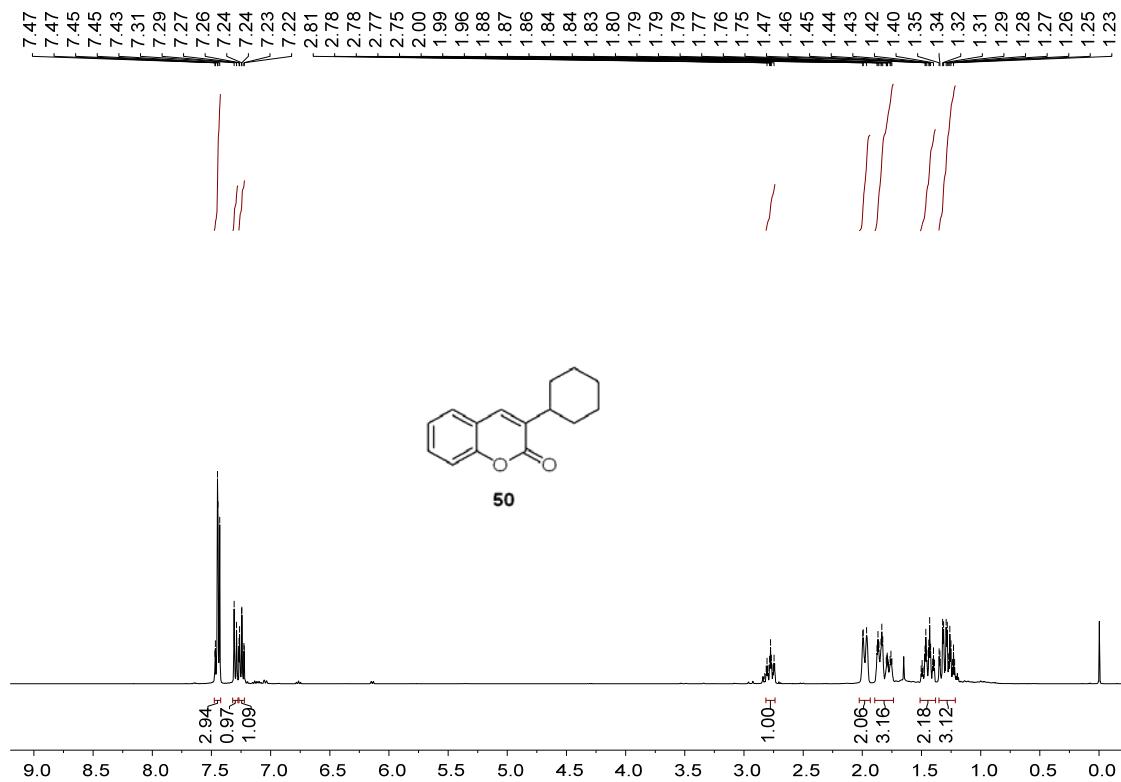
¹H NMR for **49** (400 MHz, CDCl₃)



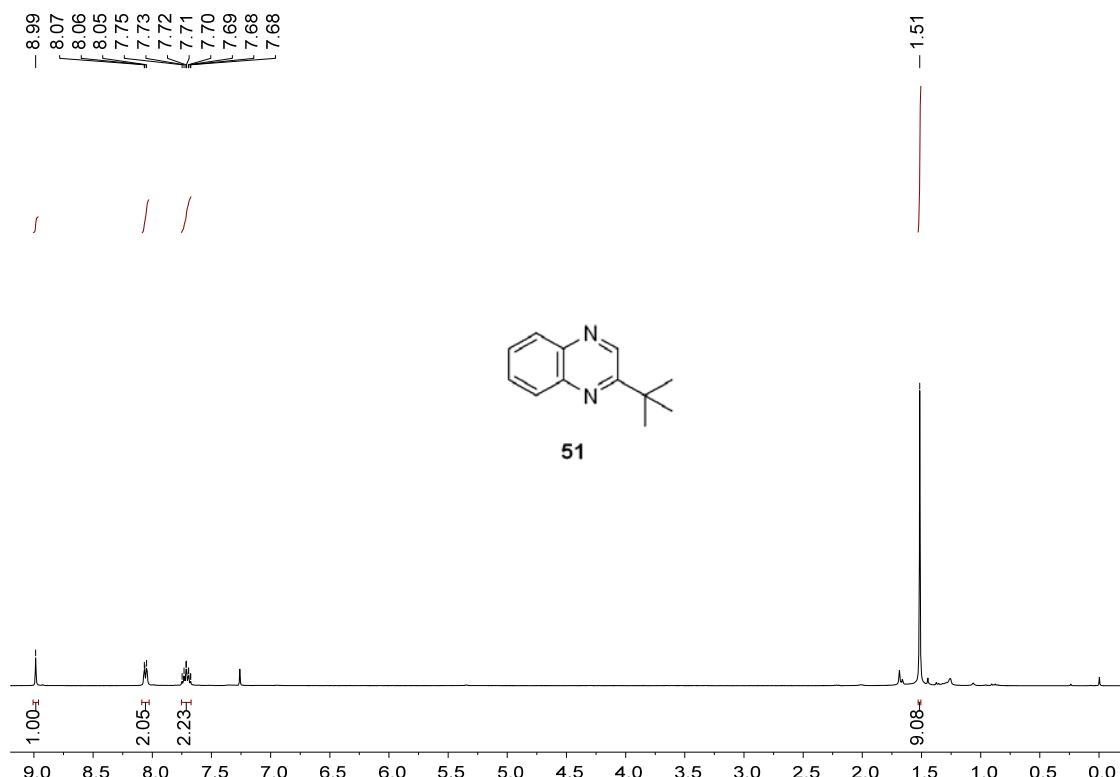
¹³C NMR for **49** (101 MHz, CDCl₃)



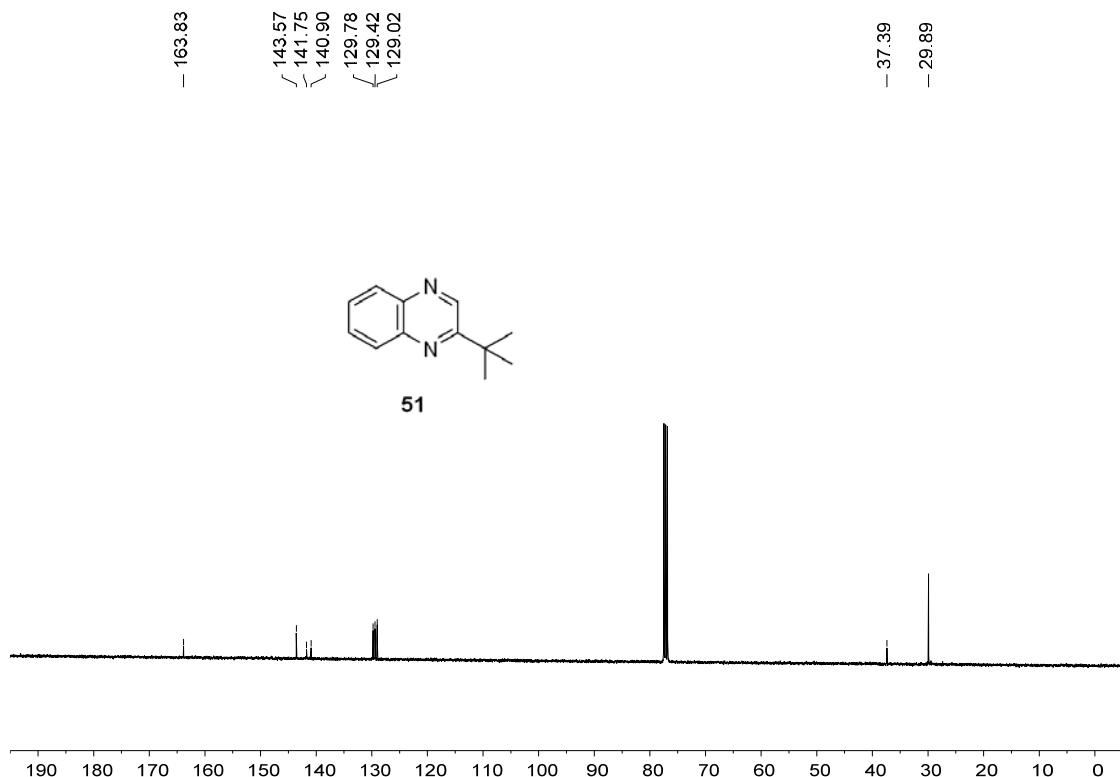
¹H NMR for **50** (400 MHz, CDCl₃)



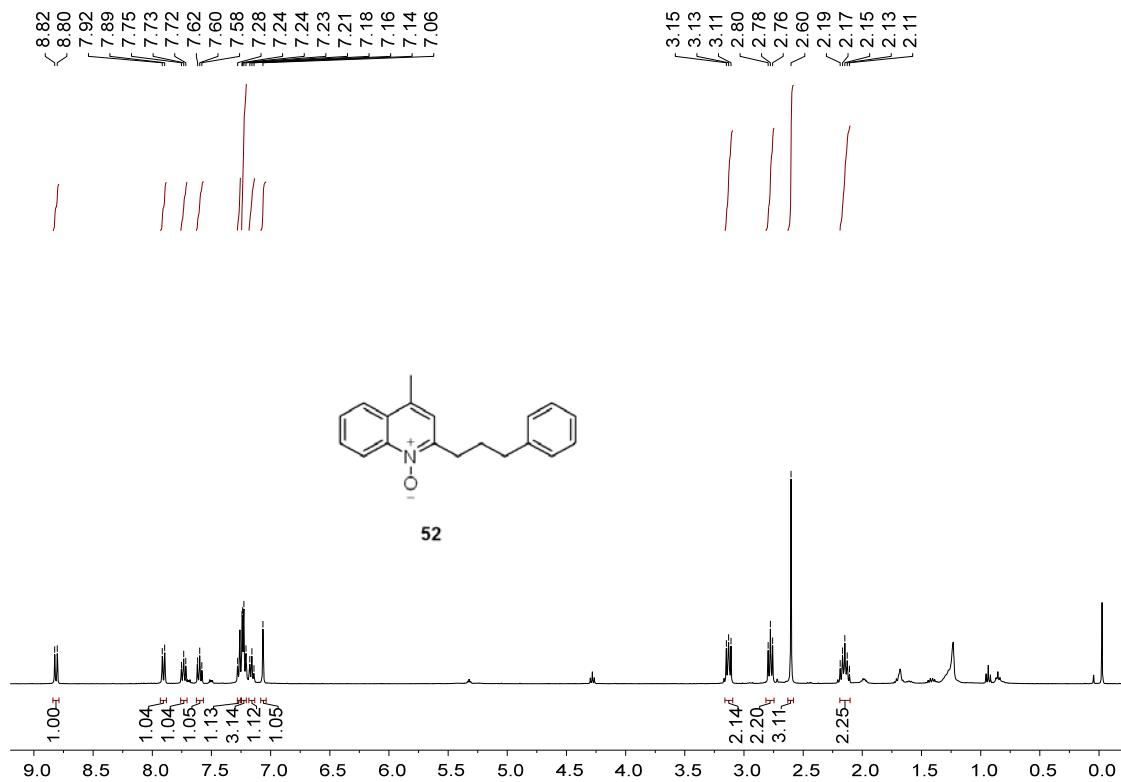
¹H NMR for **51** (400 MHz, CDCl₃)



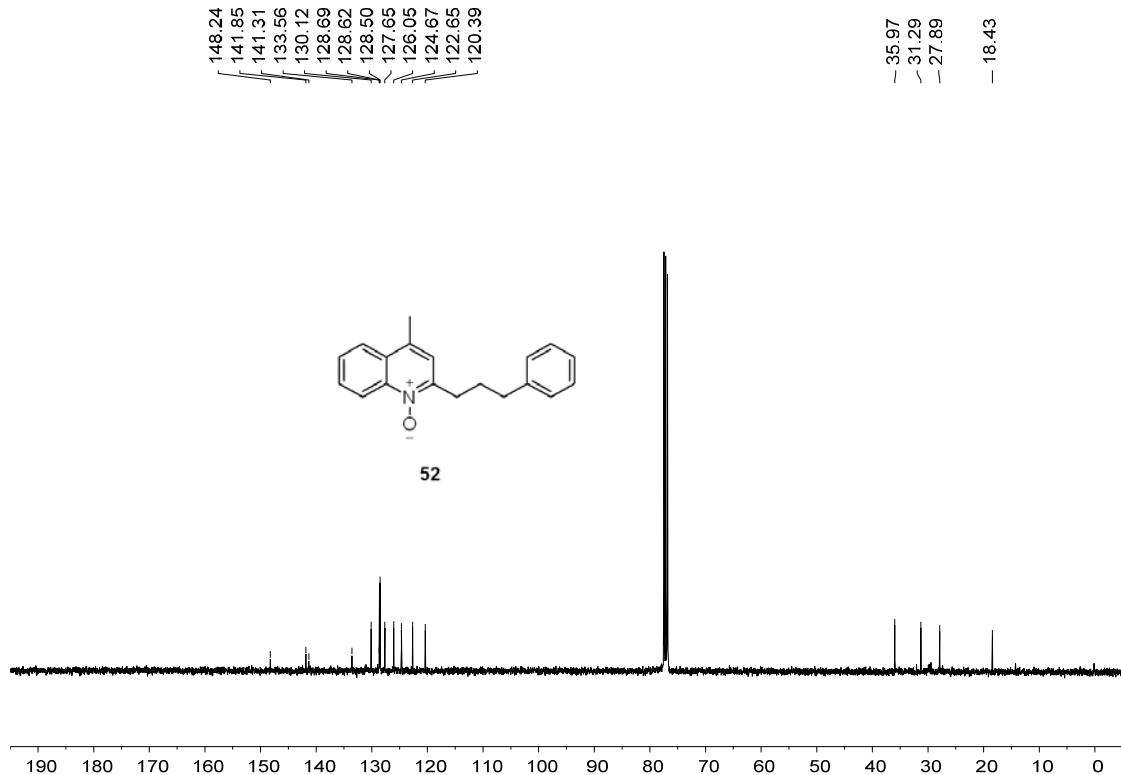
¹³C NMR for **51** (101 MHz, CDCl₃)



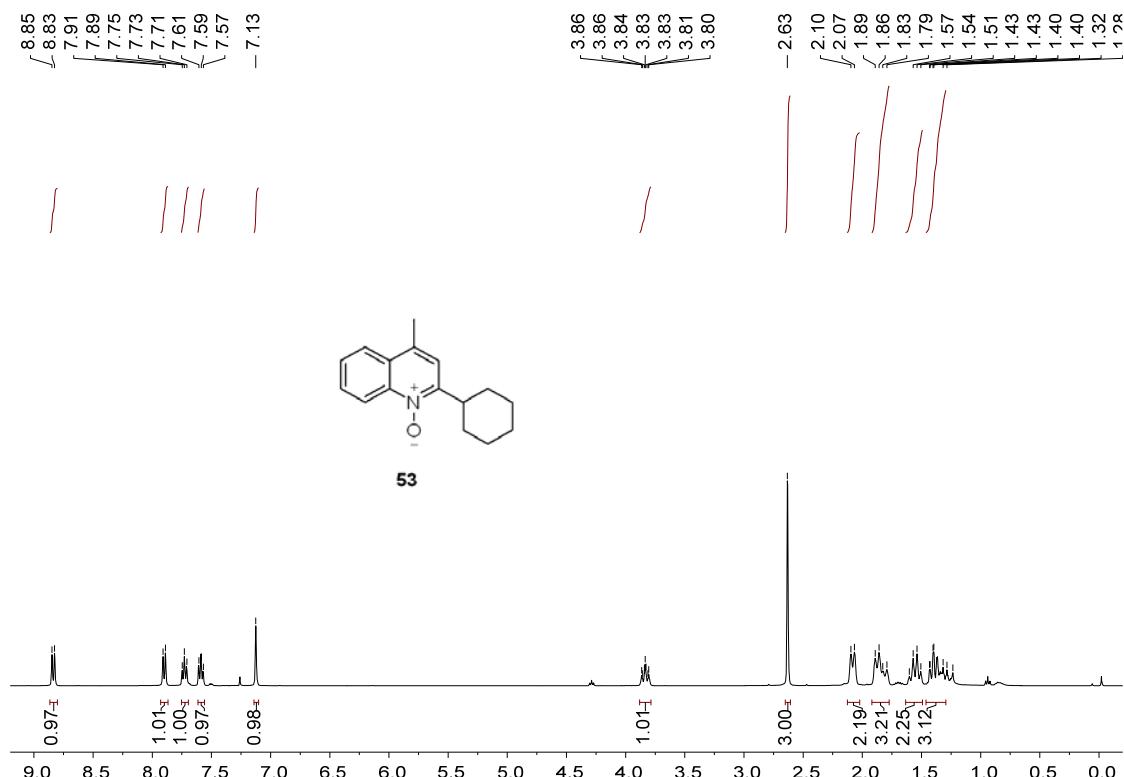
¹H NMR for **52** (400 MHz, CDCl₃)



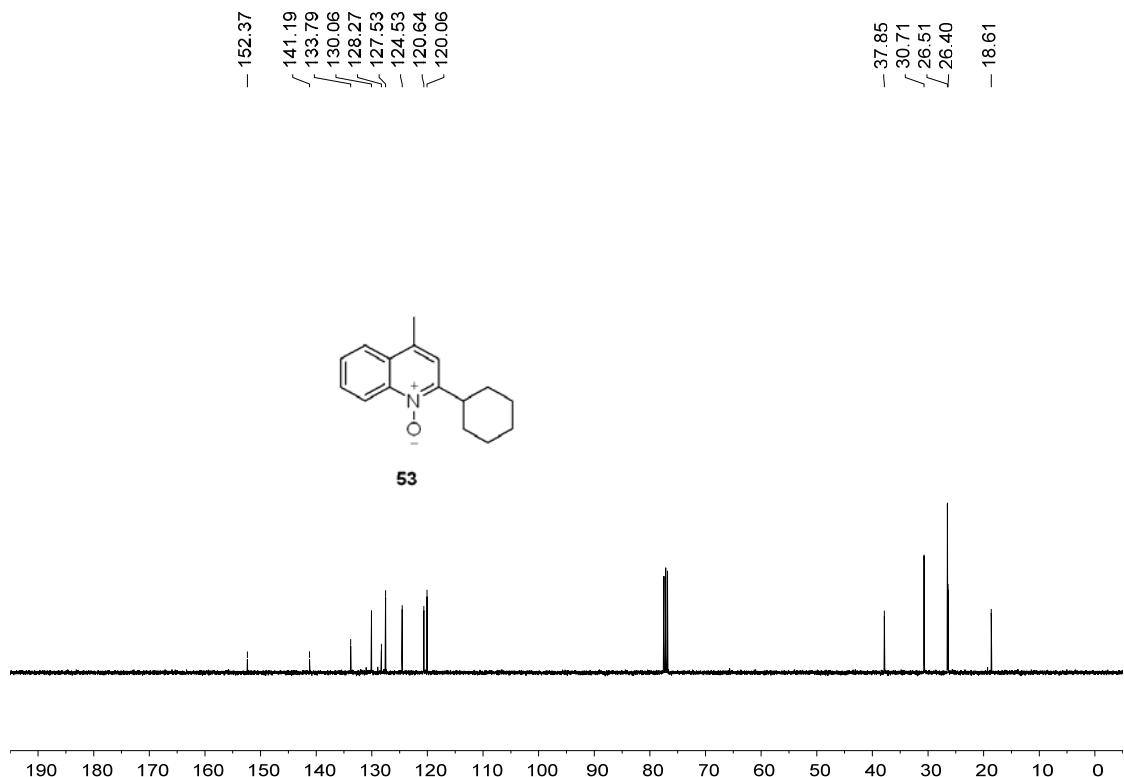
¹³C NMR for **52** (101 MHz, CDCl₃)



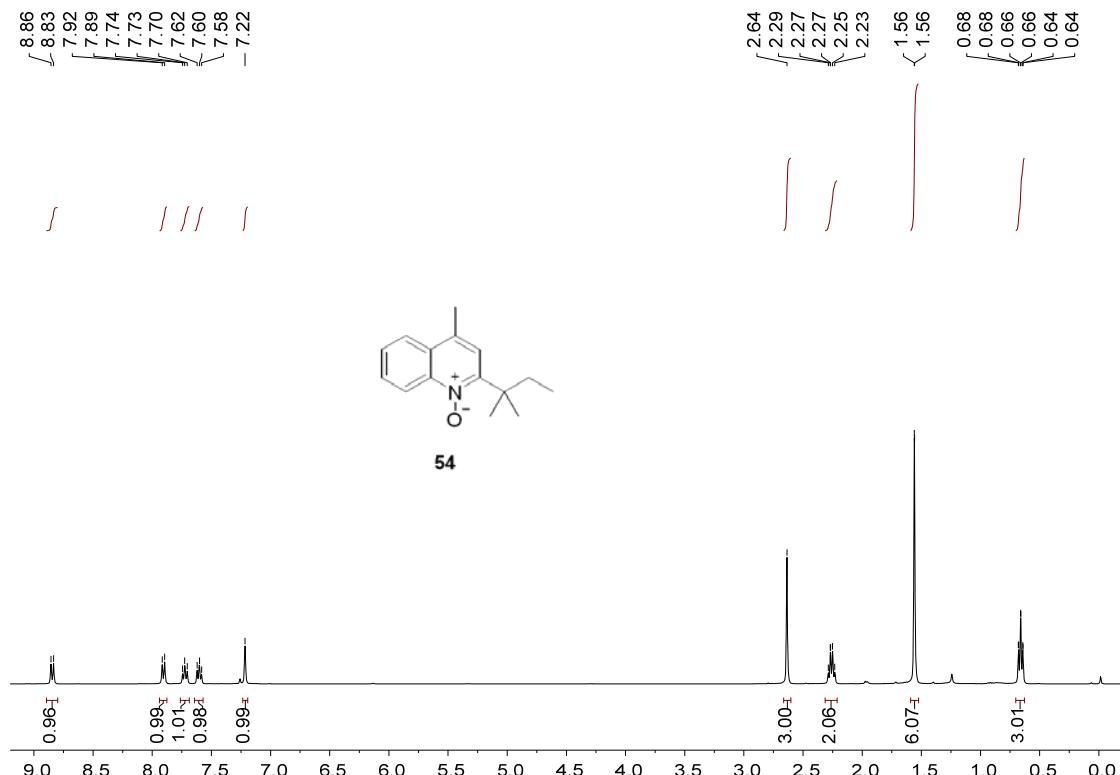
¹H NMR for **53** (400 MHz, CDCl₃)



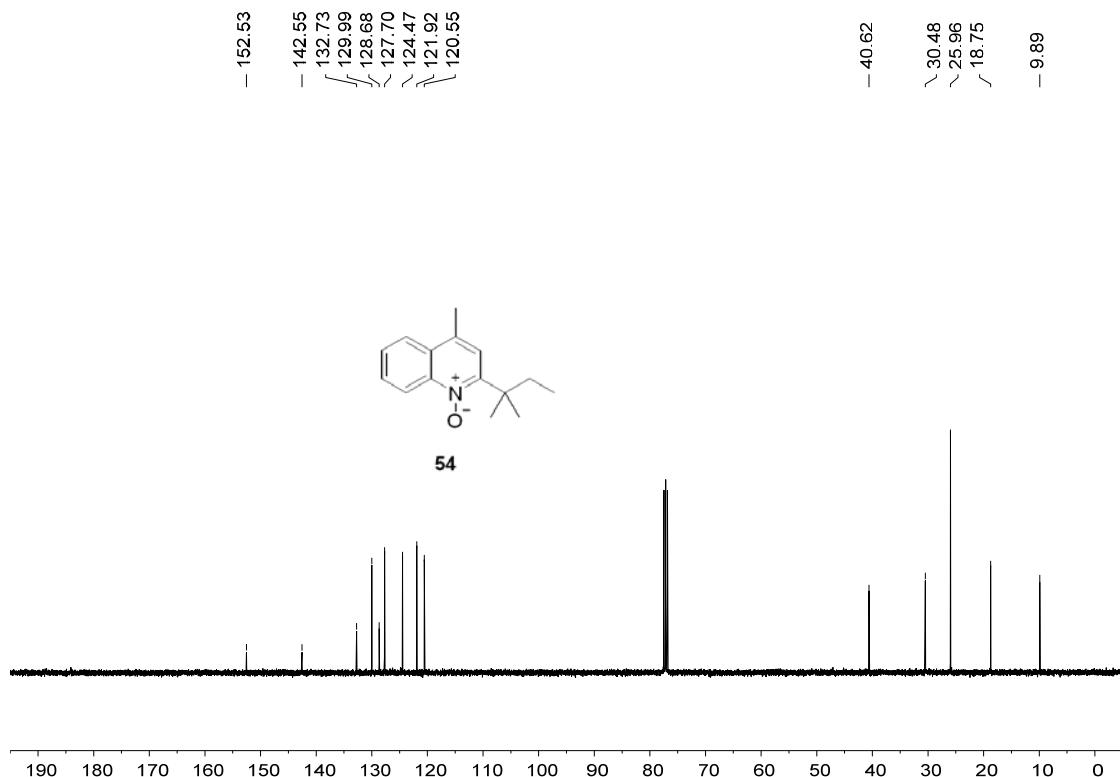
¹³C NMR for **53** (101 MHz, CDCl₃)



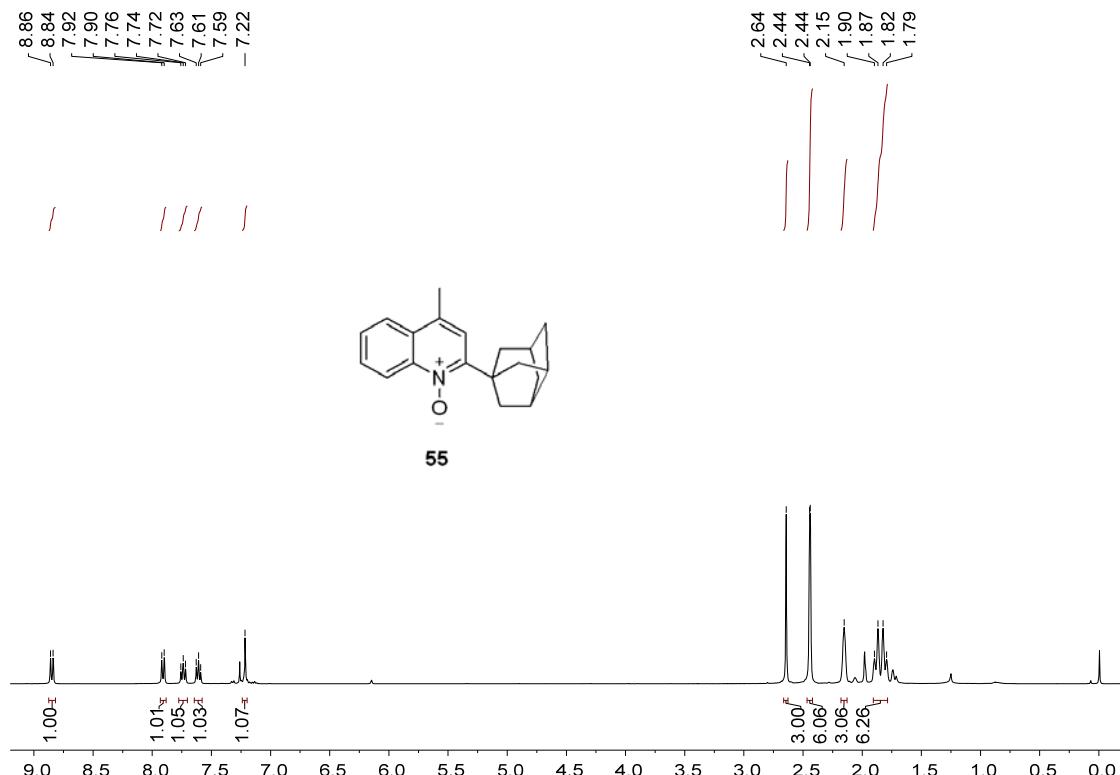
¹H NMR for **54** (400 MHz, CDCl₃)



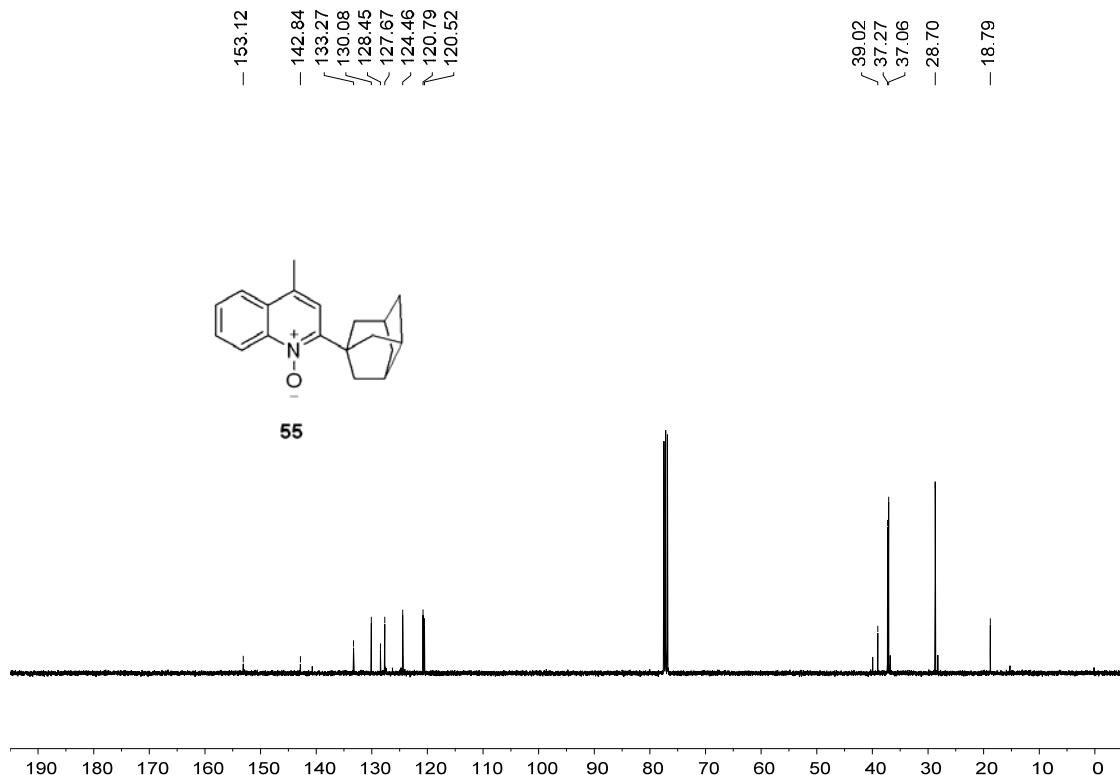
¹³C NMR for **54** (101 MHz, CDCl₃)



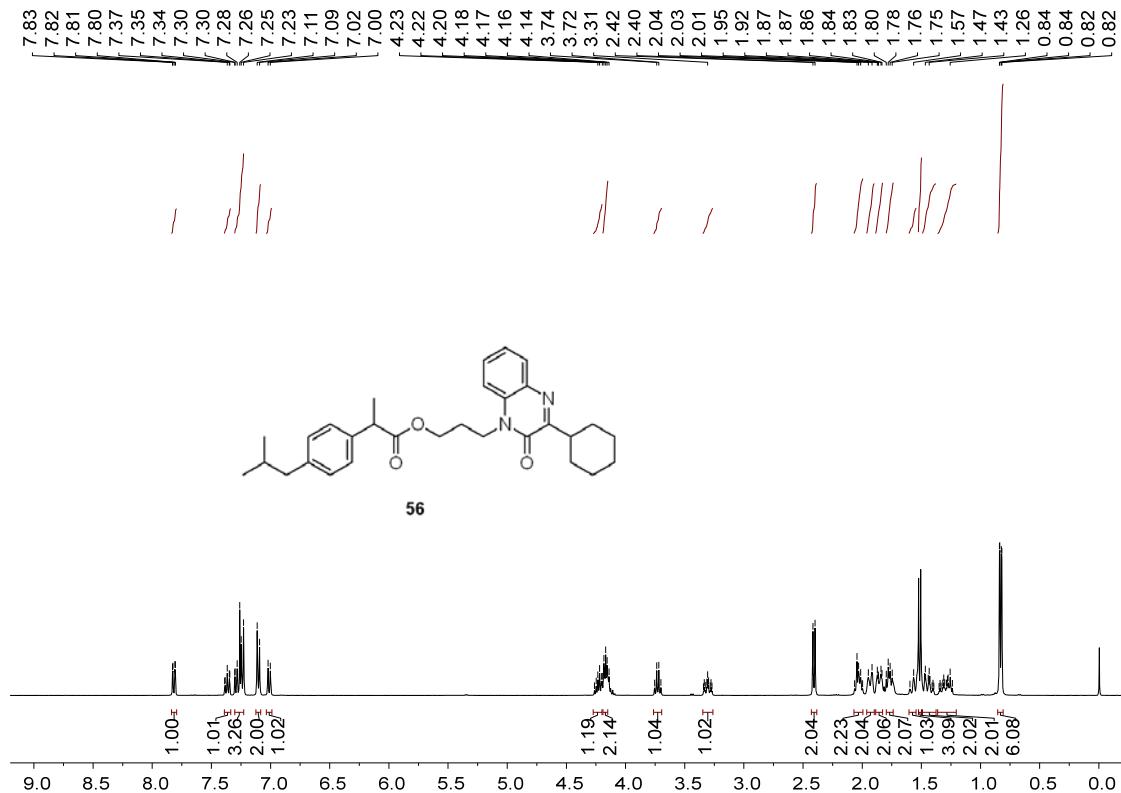
¹H NMR for **55** (400 MHz, CDCl₃)



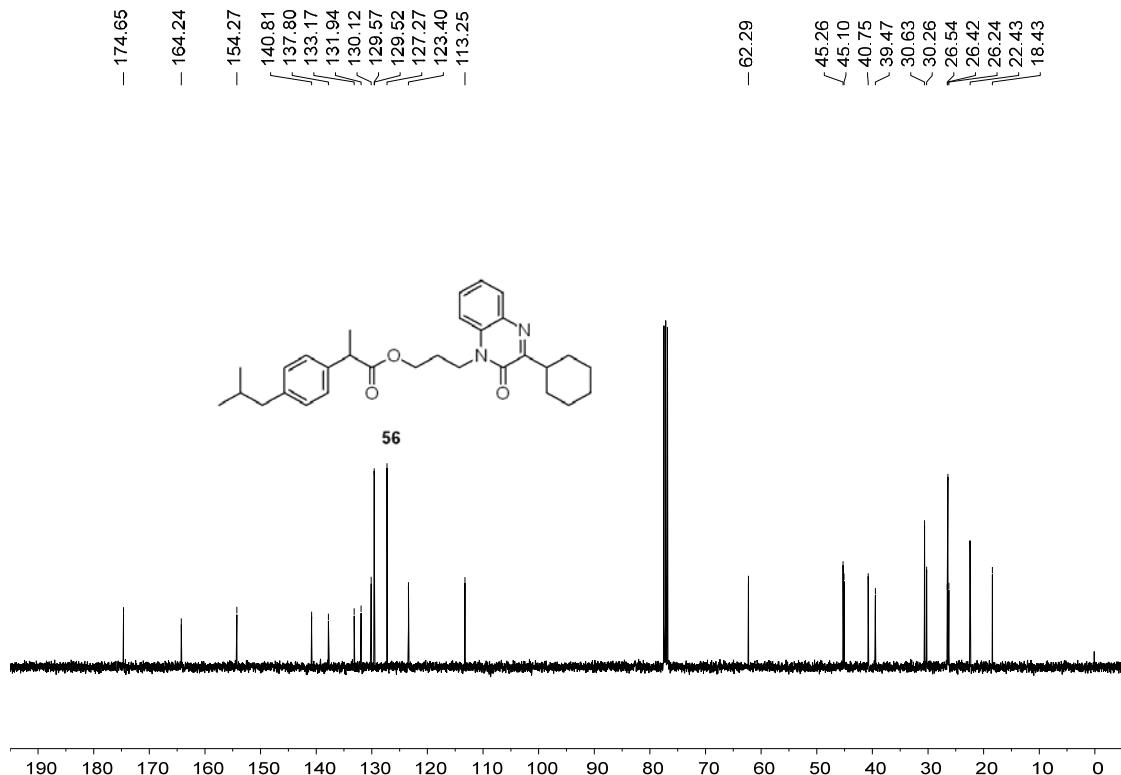
¹³C NMR for **55** (101 MHz, CDCl₃)



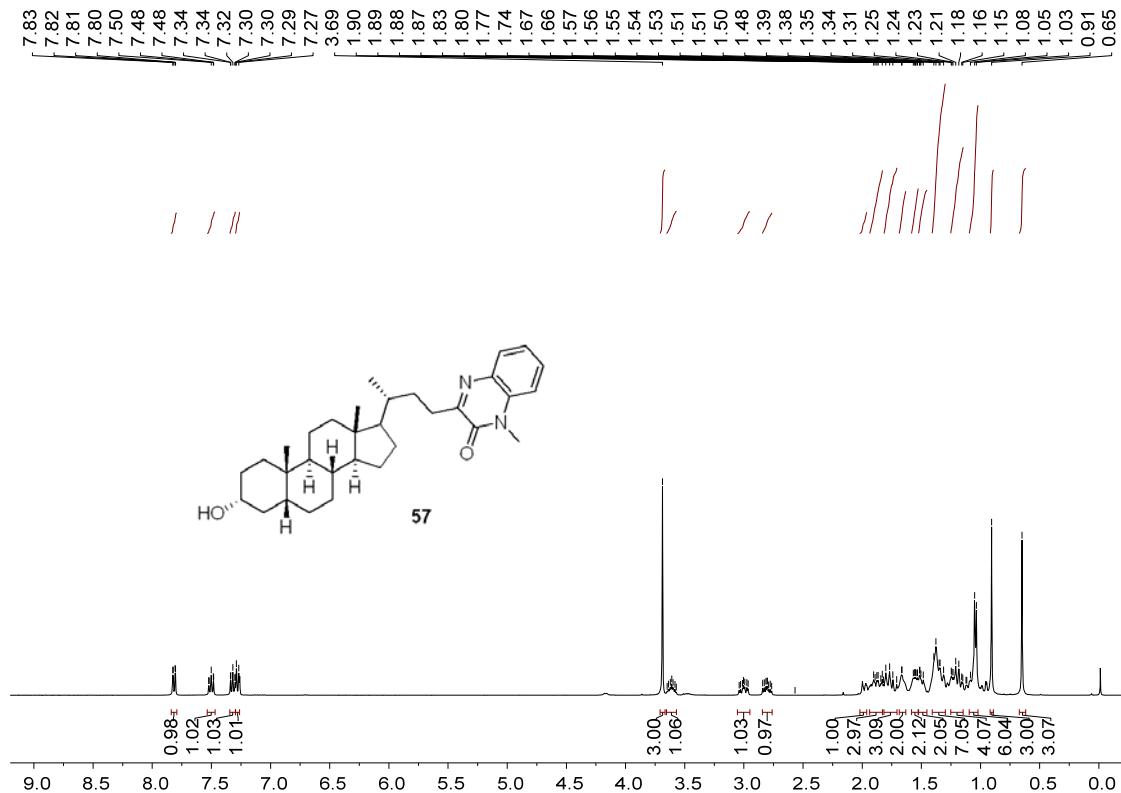
¹H NMR for **56** (400 MHz, CDCl₃)



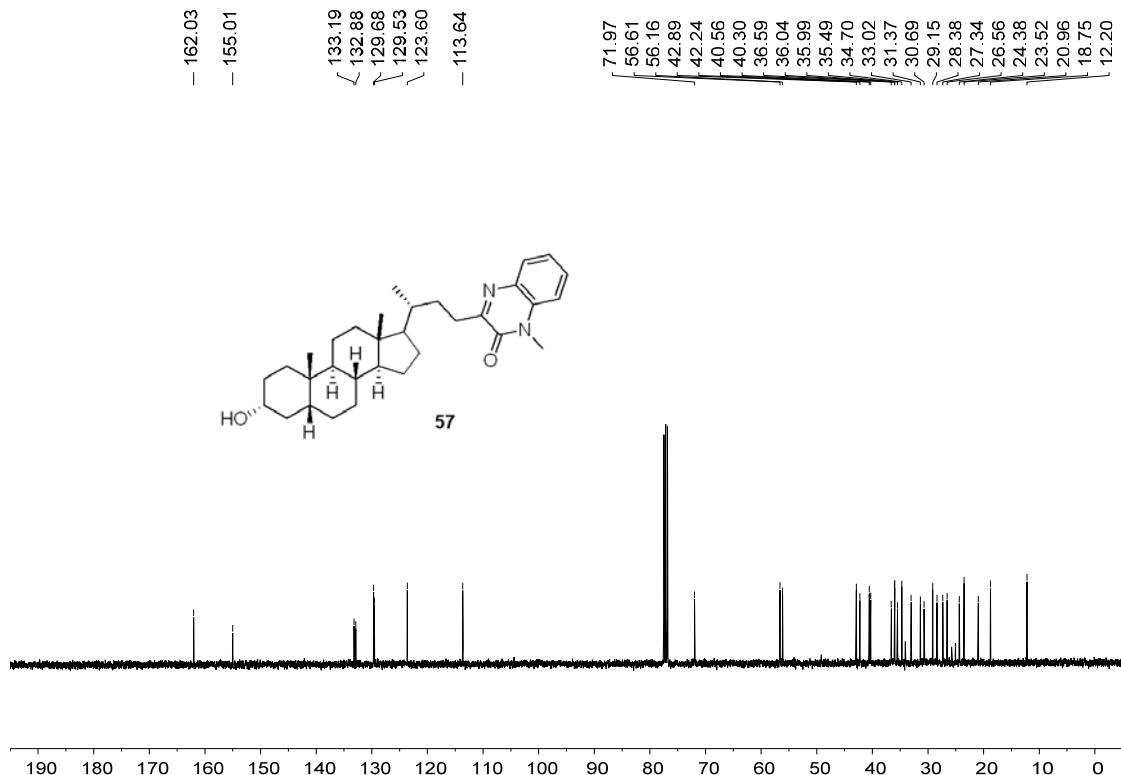
¹³C NMR for **56** (101 MHz, CDCl₃)



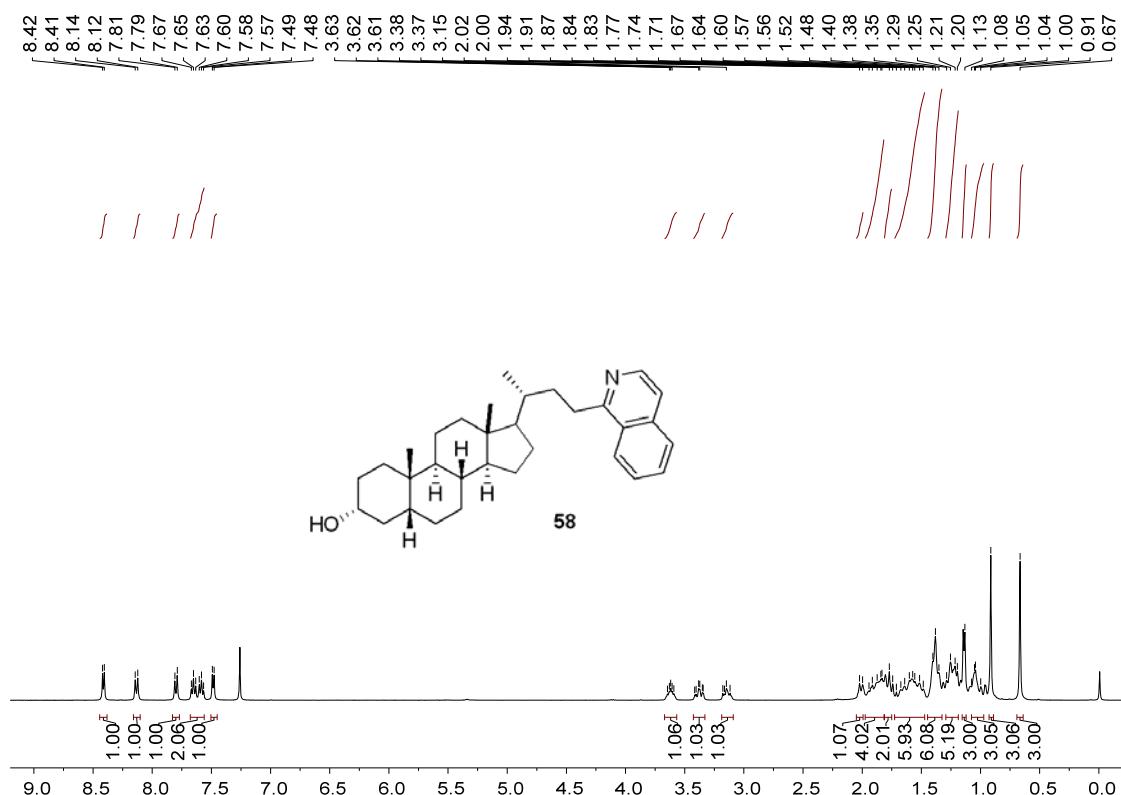
¹H NMR for **57** (400 MHz, CDCl₃)



¹³C NMR for **57** (101 MHz, CDCl₃)



¹H NMR for **58** (400 MHz, CDCl₃)



¹³C NMR for **58** (101 MHz, CDCl₃)

