Supporting Information

Alkyl radicals from diacyl peroxides: Metal-/base-/additive-free photocatalytic alkylation of *N*-heteroaromatics

Fukun Cheng,[ab] Lulu Fan,*[b] Qiyan Lv,[ac] Xiaolan Chen*[a], Bing Yu*[a]

[a] Green Catalysis Center, College of Chemistry, Zhengzhou University, Zhengzhou 450001, China. E-mail: bingyu@zzu.edu.cn

[b] College of Chemistry and Chemical Engineering, Henan University of Technology, Zhengzhou 450001, China.

[c] National Engineering Research Center of Low-Carbon Processing and Utilization of Forest Biomass, Nanjing Forestry University, Nanjing 210037, China.

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1. General Information

1.1 Materials and instruments

All reagents were purchased from commercial suppliers and were used directly without further purification unless otherwise stated. TLC was performed on silica gel plates (F254, 200-300 mesh) using UV light (254/366 nm) for detection. Products were purified by column chromatography, which was carried out on 200-300 mesh of silica gel purchased from Qing Dao Hai Yang Chemical Industry Co. All the ^{1}H, ^{13}C, and ^{19}F NMR spectra were recorded on Bruker Avance 400 MHz operating at 400 MHz, 101 MHz, and 376 MHz, respectively. Proton chemical shifts δ were given in ppm using tetramethylsilane as an internal standard. All NMR spectra were recorded in $CDCl_3$ at room temperature (20 ± 3°C). High-resolution mass spectra (HRMS) were taken with a 3000-mass spectrometer, using Waters Q-Tof MS/MS system with the ESI technique. Emission intensities were recorded using an F-4600 FL spectrophotometer. Cyclic voltammetry was performed on the CHI-660E electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd., China).

1.2 The spectrum of our lamp and the visible-light irradiation instrument

The photochemical reaction was carried out under visible light irradiation by a blue LED at 25 ºC. RLH-18 8-position Photo Reaction System manufactured by Beijing Roger Tech Ltd. was used in this system. Eight 10 W blue LEDs were equipped in this Photo reactor. The blue LED's energy peak wavelength is 460 nm, peak width at half-height is 18.4 nm, lirradiance@10 W is 237.57 mW/cm^2. The reaction vessel is borosilicate glass test tube and the distance between it and the lamp is 15 mm, no filter is applied.

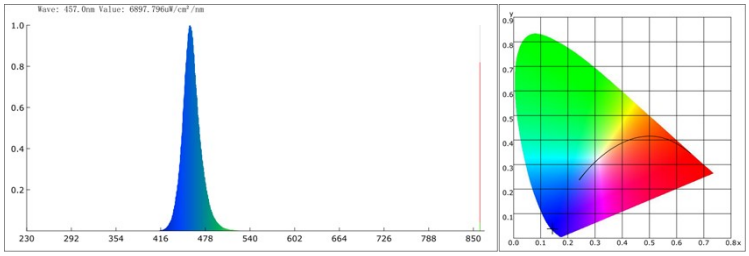


Figure S1. The spectrum of our lamp (blue LED)

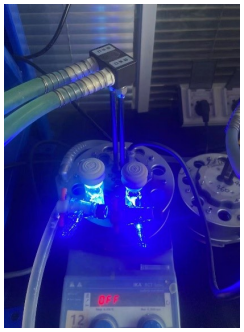
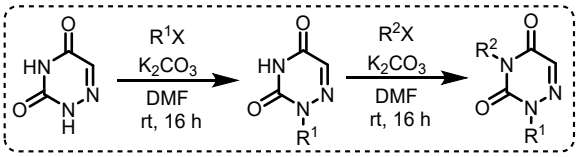


Figure S2. The visible-light irradiation instrument

1.3 General procedure for the synthesis of azauracil substrates[1]

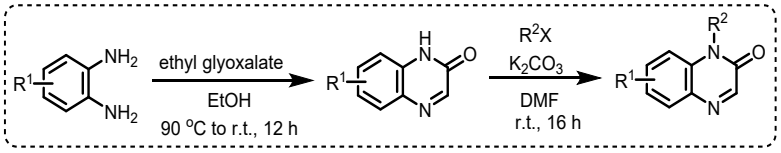


Scheme S1. General experimental procedures for azauracil substrates

Alkyl halides (3.6 mmol, 0.9 equiv.) were added dropwise to a stirring solution of 6-azauracil (4.0 mmol, 1.0 equiv.), K_2CO_3 (2.0 mmol, 0.5 equiv.) in DMF (40 mL). The reaction mixture was allowed to stir at room temperature for 16 h. Then, the mixture was quenched with saturated Na_2CO_3 solution and extracted with DCM three times. The organic layers were combined, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude products were purified through silica gel column chromatography using petroleum ether/ethyl acetate as eluent to give to afford the corresponding *N*-1-alkyl-6-azauracils.

Alkyl halides (2.0 mmol, 1.0 equiv.) were added dropwise to a stirring solution of *N*-1-alkyl-6-azauracils (2.0 mmol, 1.0 equiv.), K_2CO_3 (1.0 mmol, 0.5 equiv.) in DMF (20 mL). The reaction mixture was allowed to stir at room temperature for 16 h. Then, the mixture was quenched with saturated Na_2CO_3 solution and extracted with DCM three times. The organic layers were combined, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude products were purified through silica gel column chromatography using petroleum ether/ethyl acetate as eluent to give to afford the corresponding *N*-1, *N*-3-dialkyl-6-azauracils.

1.4 General procedure for the synthesis of quinoxalinone substrates[2]

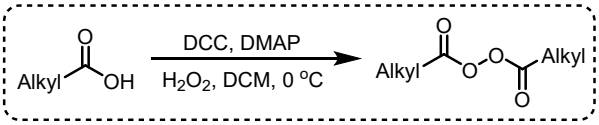


Scheme S2. General experimental procedures for quinoxalinone substrates

Ethyl glyoxalate (1.1 equiv.) was added dropwise to a stirring solution of *o*-arylenediamine (1 equiv.) in ethanol. The reaction mixture was allowed to stir at room temperature for 1 h. The precipitated solid was filtered and washed with ethanol, then dried to give quinoxalinone.

To a suspension of quinoxalinone (1 equiv.) in DMF was added potassium carbonate (1.2 equiv.) and the corresponding halogenoalcane (1.6 equiv.). The mixture was stirred at room temperature overnight. Ethyl acetate and water were added. The aqueous layer was extracted twice with EtOAc. The combined organic layers were washed with a saturated solution of NaCl, dried over $MgSO_4$, filtered, and evaporated under reduced pressure. The residue is purified by flash chromatography over silica gel to afford the desired product *N*-alkyl quinoxalinone.

1.5 General procedure for the synthesis of alkyl diacyl peroxides[3]

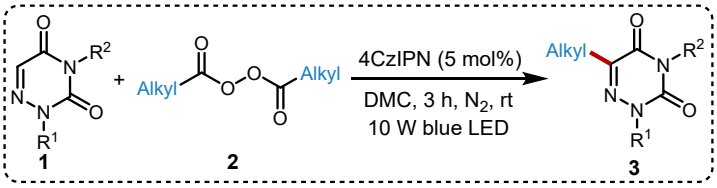


Scheme S3. General experimental procedures for alkyl diacyl peroxides substrates

A solution of DMAP (0.1 equiv.), 30% hydrogen peroxide (1.5 equiv.), and alkyl acid (6 mmol) in DCM was cooled to -10 ºC for about 15 min, then DCC dissolved in DCM was added. After stirring for 2 h at -10 ºC, the solution was filtered through a short pad of silica gel. The combined solution was concentrated on a rotary evaporator under vacuum at 10 – 15 ºC, and purified using DCM as the eluent to afford the diacyl peroxide.

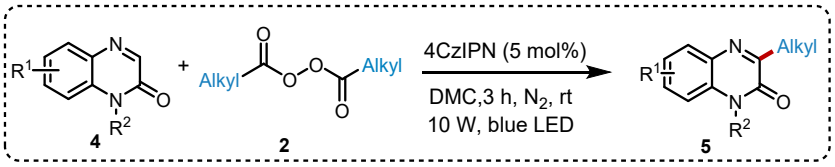
2. Experimental procedures

2.1 General experimental procedures for the desired product



Scheme S4. General experimental procedures for alkylated azauracils

The mixture of azauracil 1 (0.2 mmol), alkyl diacyl peroxides 2 (0.2 mmol), 4CzIPN (5 mol %) and DMC (2.0 mL) were sequentially added in a 25 mL reaction vessel. Then the reaction vessel was irradiated with 10 W blue LED (460 nm) at room temperature under N_2 atmosphere for 3 h. After the reaction, the solvent was evaporated under vacuum. The residue was purified by chromatography on silica gel using petroleum ether/ethyl acetate as eluent to afford the desired product **3**.

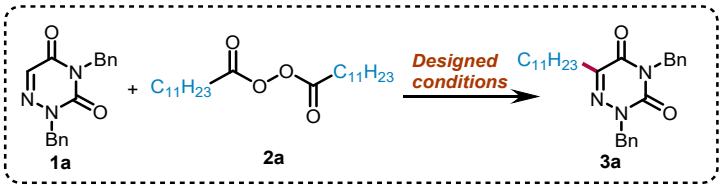


Scheme S5. General experimental procedures for alkylated quinoxalinones

The mixture of quinoxalinone 4 (0.2 mmol), alkyl diacyl peroxides 2 (0.2 mmol), 4CzIPN (5 mol %) and DMC (2.0 mL) was sequentially added in a 25 mL reaction vessel. Then the reaction vessel was irradiated with 10 W blue LED (460 nm) at room temperature under N_2 atmosphere for 3 h. After the reaction, the solvent was evaporated under vacuum. The residue was purified by chromatography on silica gel using petroleum ether/ethyl acetate as eluent to afford the desired product **5**.

2.2 Sensitivity assessment of reaction

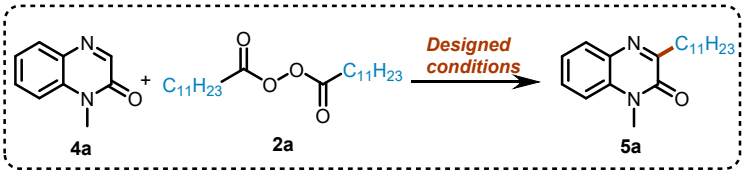
Table S1. Sensitivity assessment of the reaction of **1a** with **2a**



Parameter	Variation		Description	Yield[b]
Concentration (*c*)	High *c*	*c* + 10% *c*	0.9 ml DMC	82%
	Low *c*	*c* - 10% *c*	1.1 mL DMC	80%
H_2O level	High H_2O	+ H_2O; V_{H2O} = 1%V_{rxn}	10 μL H_2O in 1.0 mL DMC	81%
O_2 level	High O_2	Air	Air instead of N_2	40%
Temperature (*T*)	High *T*	*T* + 10 °C	35 °C	83%
	Low *T*	*T* – 10 °C	15 °C	80%
Light intensity (*W*)	Low *W*	*W*/16	0.6 W	0%
Scale	Big scale	n·30	3 mmol of **1a**	85%

[a]Standard conditions: 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione (0.1 mmol), LPO (0.1 mmol), 4CzIPN (5 mol%) and DMC (1.0 mL), 460 nm blue LED (10 W), under N_2 for 3 h, room temperature. [b]The average yield of three parallel reactions.

Table S2. Sensitivity assessment of the reaction of **4a** with **2a**



Parameter	Variation		Description	Yield[b]
Concentration (*c*)	High *c*	*c* + 10% *c*	0.9 mL DMC	79%
	Low *c*	*c* - 10% *c*	1.1 mL DMC	77%
H_2O level	High H_2O	+ H_2O; V_{H2O} = 1%V_{rxn}	10 μL H_2O in 1.0 mL DMC	74%
O_2 level	High O_2	Air	Air instead of N_2	0%
Temperature (*T*)	High *T*	*T* + 10 °C	35 °C	74%
	Low *T*	*T* – 10 °C	15 °C	73%
Light intensity (*W*)	Low *W*	*W*/16	0.6 W	0%
Scale	Big scale	n·30	3 mmol of **4a**	72%

[a]Standard conditions: quinoxalinone (0.1 mmol), LPO (0.1 mmol), 4CzIPN (5 mol %) and DMC (1.0 mL), 460 nm blue LED (10 W), under N_2 for 3 h, room temperature. [b]The average yield of three parallel reactions.

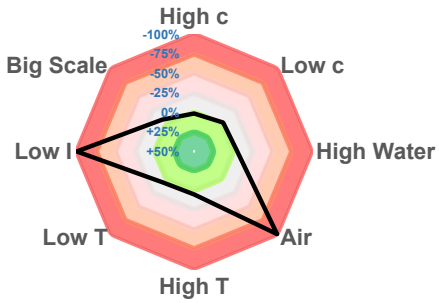
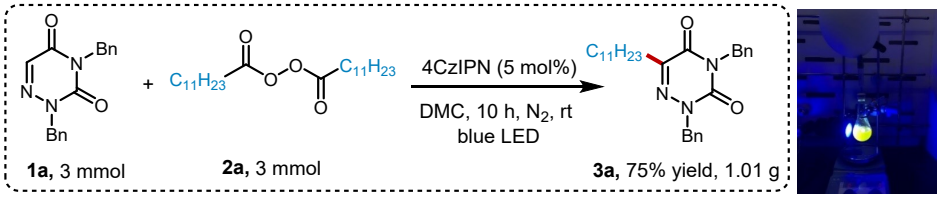


Figure S3. Sensitivity assessment of the reaction of **2a** with **4a**.

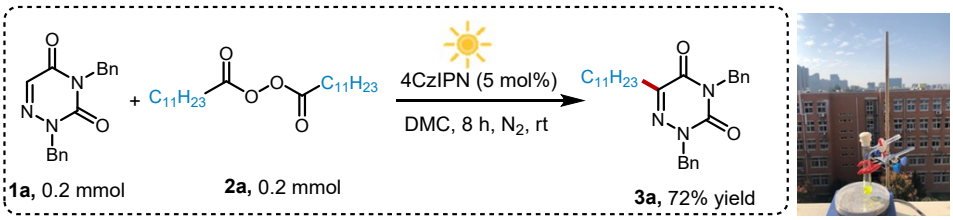
2.3 Gram-scale synthesis



Scheme S6. Gram-scale synthesis of **3a**

The mixture of 2,4-dibenzyl-1,2,4-triazine-3,5 (2H,4H) -dione **1a** (3 mmol), LPO **2a** (3 mmol), 4CzIPN (5 mol%) and DMC (30 mL) were sequentially added in a 100 mL round bottom flask. Then the reaction vessel was irradiated with 10 W blue LED (465 nm) at room temperature under N_2 atmosphere for 10 h. After the reaction, the solvent was evaporated under vacuum. The residue was purified by chromatography on silica gel using petroleum ether/ethyl acetate as eluent to afford the desired product **3a** (1.01 g, 75%).

2.4 Irradiation with natural sunlight



Scheme S7. Synthesis of **3a** under natural sunlight

The mixture of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione **1a** (0.2 mmol), LPO **2a** (0.2 mmol), 4CzIPN (5 mol%) and DMC (2.0 mL) were sequentially added in a 25 mL reaction vessel. Then the reaction system was carried out under sunlight for 8 h (from 9:00 to 17:00. 2022/09/28 in Zhengzhou, Henan province, China. Temperature: 20 ℃ – 25 ℃). After the reaction, the solvent was evaporated under vacuum. The residue was purified by chromatography on silica gel using petroleum ether/ethyl acetate as eluent to afford the desired product **3a** (72%).

2.5 Procedure for emission quenching experiments

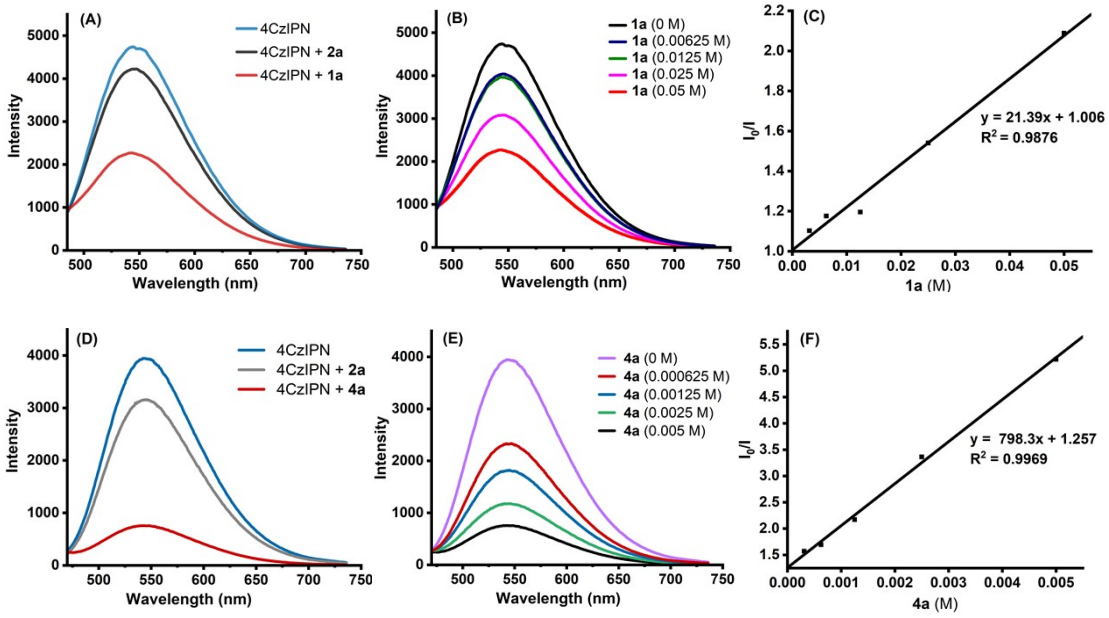


Figure S4. Luminescence quenching study

Emission intensities were recorded using an F-4600 FL Spectrophotometer. First, the emission intensity of 4CzIPN solutions was observed at 550 nm. The solutions were irradiated at 378 nm (Maximum absorption wavelength of 4CzIPN) and fluorescence was measured from 400 nm to 700 nm. In a typical experiment, the emission spectrum of a 5×10^{-5} M solution of 4CzIPN with different concentration of **1a**, **4a** in degassed anhydrous CH_3CN in 10 mm path length quartz cuvette was collected: A) the emission spectra of 5×10^{-5} M solutions of 4CzIPN with reactants (**1a**, **2a**) in degassed anhydrous CH_3CN; B) the emission spectra of a 5×10^{-5} M solution of 4CzIPN with various concentrations of **1a** in degassed anhydrous CH_3CN. C) the linear relationship between I_0/I and the increasing concentration of **1a** (I_0 and I are the fluorescence intensities before and after the increasing the concentration of **1a**, respectively.). D) the emission spectra of 5×10^{-5} M solutions of 4CzIPN with reactants (**2a**, **4a**) in degassed anhydrous CH_3CN; E) the emission spectra of a 5×10^{-5} M solution of 4CzIPN with various concentrations of **4a** in degassed anhydrous CH_3CN. F) the linear relationship between I_0/I and the increasing concentration of **4a** (I_0 and I are the fluorescence intensities before and after the increasing the concentration of **4a**, respectively).

2.6 Procedure for cyclic voltammogram experiments

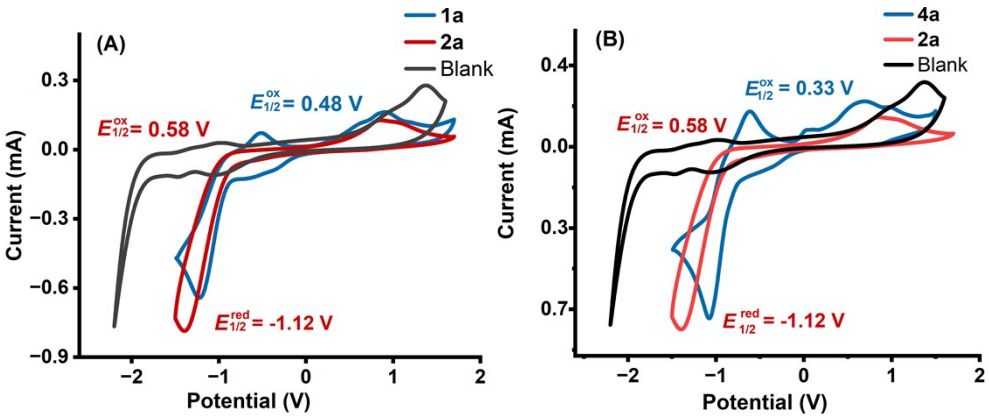


Figure S5. Cyclic voltammogram study

Cyclic voltammetry was performed on the CHI-660E electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd., China). Cyclic voltammograms of 0.1 M tetrabutylammonium hexafluorophosphate (TBAH) and related compounds in CH_3CN using Pt working electrode, Pt wire, and silver chloride electrode (Ag/AgCl) as counter electrode and reference electrode at 100 mV/s scan rate.The relationship between E(Ag/AgCl) and E(SCE) is converted according to the following formula : E(Ag/AgCl) = E(SCE) + 0.042 V).[4]

2.7 Procedure for the fluorescence emission experiments

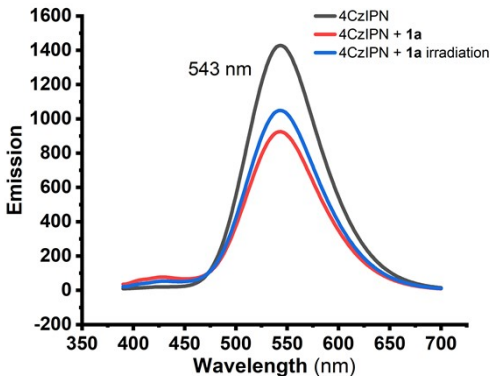
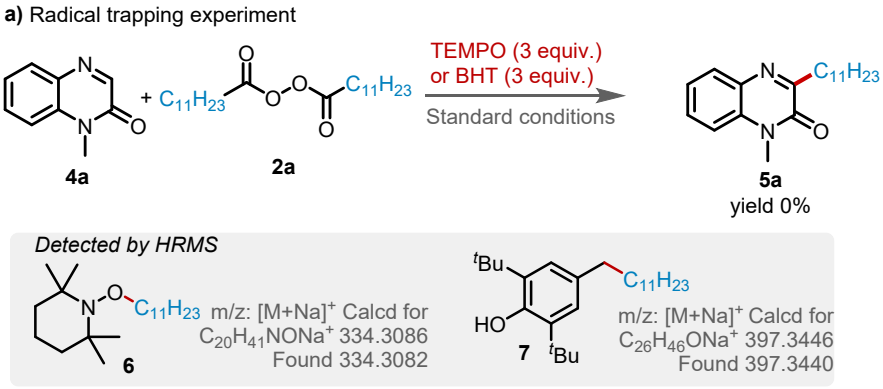


Figure S6. the fluorescence emission experiments

According to the protocol of Section **2.6**, the fluorescence emission experiments of the solution of 4CzIPN (5×10^{-5} M) in anhydrous CH_3CN (Figure S6, grey line), the solution of **1a** (5×10^{-2} M) and 4CzIPN (5×10^{-5} M) in anhydrous CH_3CN (Figure S6, red line), and the solution of **1a** (5×10^{-2} M) and 4CzIPN (5×10^{-5} M) in anhydrous CH_3CN irradiation with 460 nm blue

LED for 10 min before the test (Figure S6, blue line) were conducted. No hypsochromic shift is observed, thus ruling out the consecutive photoinduced electron transfer (ConPET) mechanism.

2.8 Control experiments



Scheme S8. Control experiments

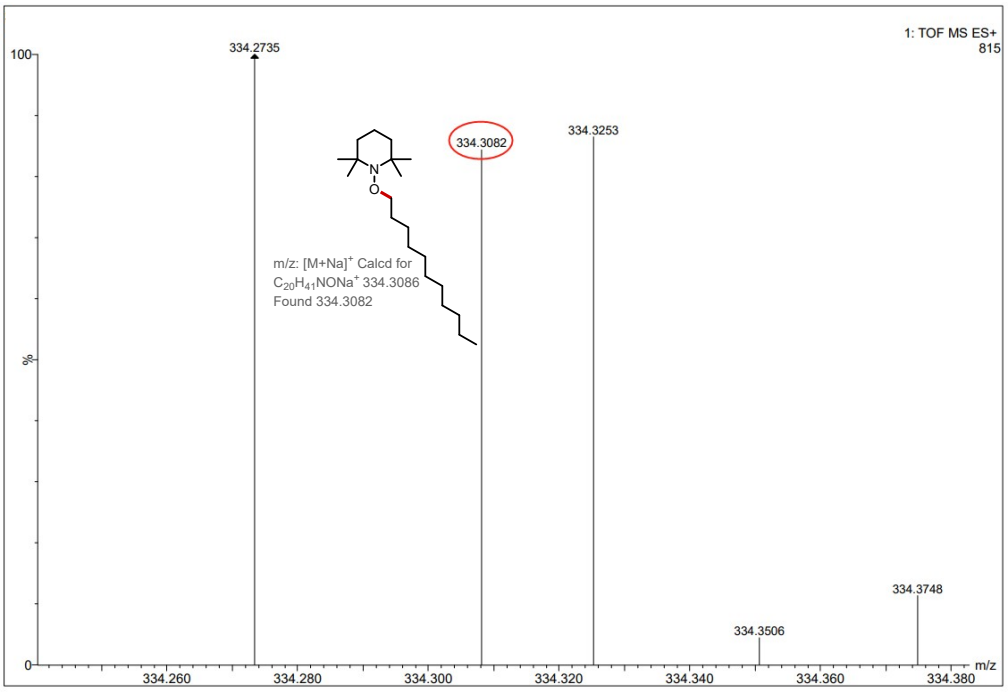


Figure S7. The HRMS analysis of compound **6**

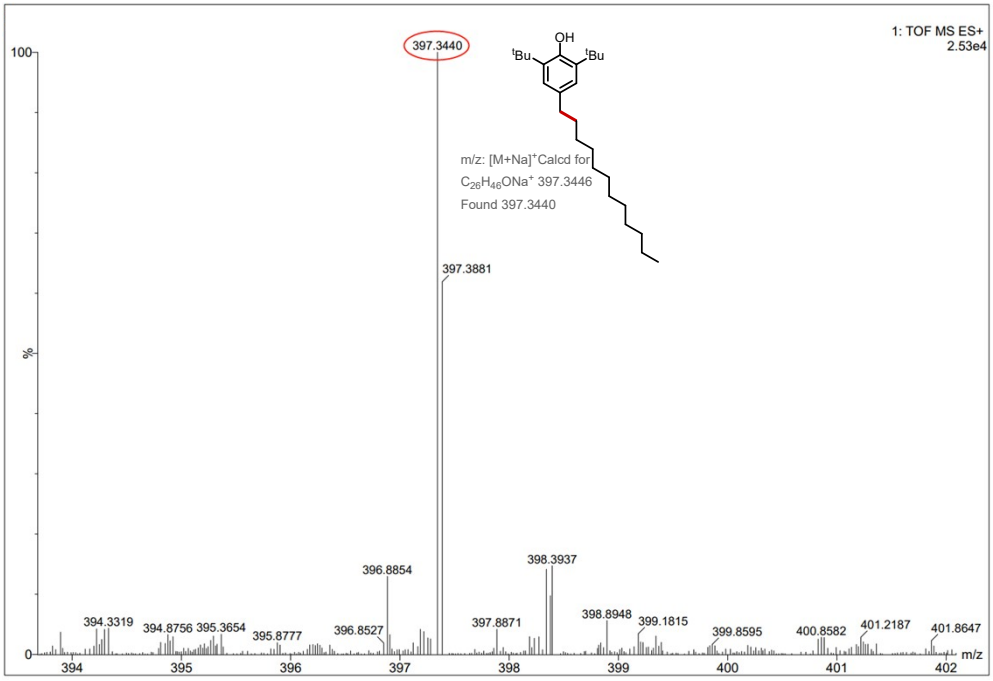


Figure S8. The HRMS analysis of compound **7**

2.9. HRMS spectra of alkyl acid

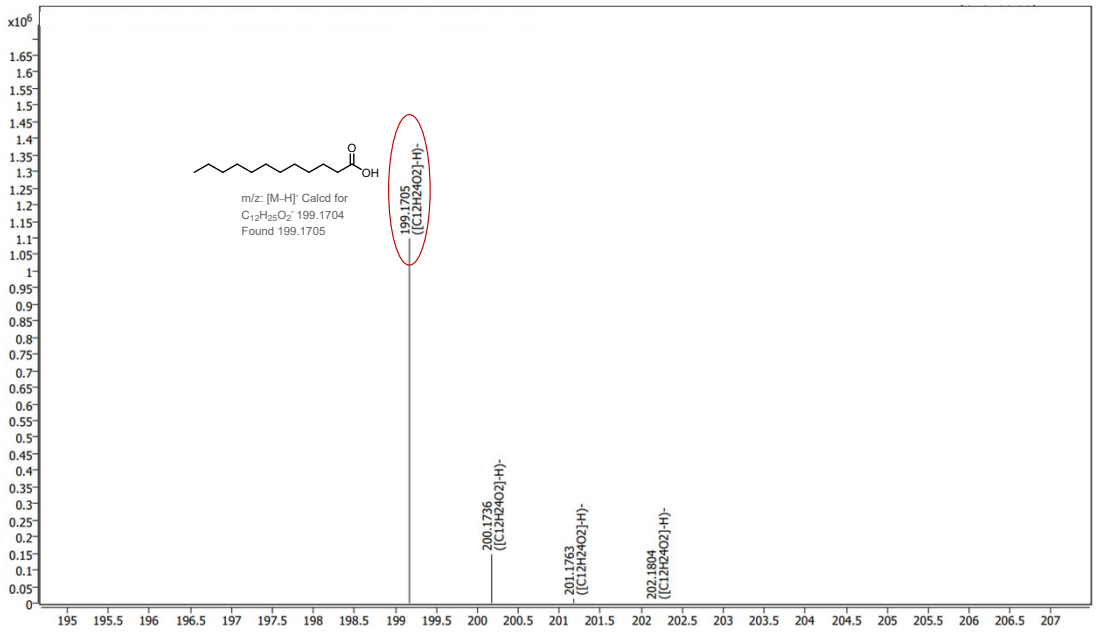
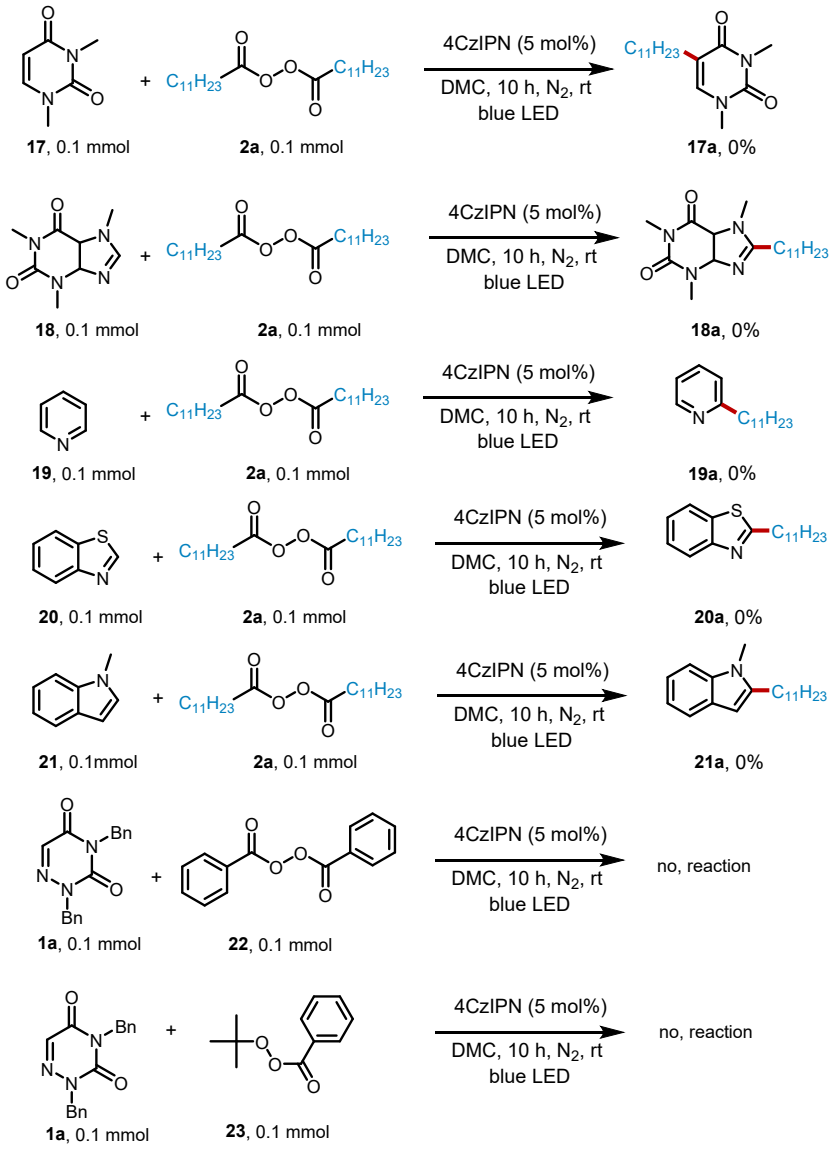


Figure S9. The HRMS analysis of alkyl acid

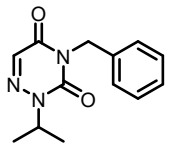
2.11 Ineffective transformations



Scheme S9. Ineffective transformations

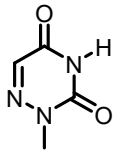
3. Characterization Data for Products

4-benzyl-2-isopropyl-1,2,4-triazine-3,5(2H,4H)-dione (1h)



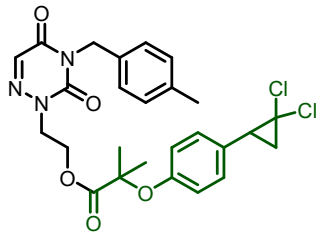
Purification by flash column chromatography (PE:EA, 10:1 v/v). White solid (500.7 mg, 83% yield) mp 80.6 – 82.6 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 – 7.48 (m, 2H), 7.46 (s, 1H), 7.39 – 7.26 (m, 3H), 5.11 (s, 2H), 4.99-4.92 (m, 1H), 1.33 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.7, 148.4, 135.6, 134.0, 129.5, 128.6, 128.1, 50.9, 43.9, 20.6. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{13}H_{15}N_3O_2Na$, 268.1056; Found: 268.1058.

2-methyl-1,2,4-triazine-3,5(2H,4H)-dione (1j)



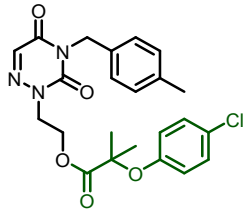
Purification by flash column chromatography (PE:EA, 8:1 v/v). White solid (1200.7 mg, 75% yield). mp 59.5 – 61.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.33 (s, 1H), 7.43 (s, 1H), 3.35 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.2, 149.8, 135.2, 26.4. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_4H_5N_3O_2H$, 128.0455; Found: 128.0459.

2-(4-(4-methylbenzyl)-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (1u)



Purification by flash column chromatography (PE:EA, 20:1 v/v). Colorless oil (500.2 mg, 90% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 8.1 Hz, 2H), 7.23 (s, 1H), 7.14 (d, *J* = 7.8 Hz, 2H), 7.06 (d, *J* = 8.6 Hz, 2H), 6.80 – 6.71 (m, 2H), 5.01 (s, 2H), 4.49 (dd, *J* = 5.7, 4.7 Hz, 2H), 4.23 (dd, *J* = 5.9, 4.5 Hz, 2H), 2.83 (dd, *J* = 10.7, 8.3 Hz, 1H), 2.34 (s, 3H), 1.96 (dd, *J* = 10.7, 7.4 Hz, 1H), 1.78 (dd, *J* = 8.3, 7.4 Hz, 1H), 1.55 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.0, 155.6, 154.8, 148.6, 138.0, 134.5, 132.3, 129.6, 129.4, 129.3, 128.3, 118.5, 62.1, 60.9, 50.4, 43.7, 34.8, 25.9, 25.3, 25.2, 21.2. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{26}H_{27}N_3O_5NaCl_2$, 554.1220; Found: 554.1220.

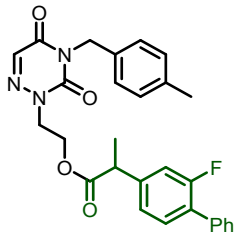
2-(4-(4-methylbenzyl)-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-(4-chlorophenoxy)-2-methylpropanoate (1v)



Purification by flash column chromatography (PE:EA, 30:1 v/v). Colorless oil (350.1 mg, 77%

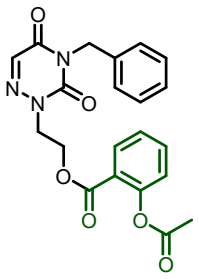
yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (d, *J* = 8.1 Hz, 2H), 7.29 (s, 1H), 7.19 – 7.08 (m, 4H), 6.77 – 6.69 (m, 2H), 5.01 (s, 2H), 4.48 (dd, *J* = 5.7, 4.5 Hz, 2H), 4.23 (dd, *J* = 5.7, 4.5 Hz, 2H), 2.33 (s, 3H), 1.53 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.7, 155.6, 153.9, 148.6, 137.9, 134.5, 132.4, 129.4, 129.3, 129.1, 127.3, 120.3, 62.2, 50.4, 43.7, 25.2, 21.2. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{23}H_{24}N_3O_5NaCl$, 480.1297; Found: 480.1296.

2-(3-(4-methylbenzyl)-2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)ethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (1w)



Purification by flash column chromatography (PE:EA, 20:1 v/v). Colorless oil (500.3 mg, 90% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.62 – 7.54 (m, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.40 (dd, *J* = 11.8, 7.8 Hz, 4H), 7.32 (s, 1H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.14 – 7.03 (m, 2H), 5.06 (d, *J* = 2.2 Hz, 2H), 4.58 – 4.37 (m, 2H), 4.32-4.15 (m, 2H), 3.68 (q, *J* = 7.2 Hz, 1H), 2.35 (s, 3H), 1.51 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.7, 159.7 (d, *J* = 248.5 Hz), 155.7, 148.8, 141.4 (d, *J* = 7.5 Hz), 138.0, 135.4, 134.5, 132.5, 130.8 (d, *J* = 4.0 Hz), 129.4, 129.3, 129.0 (d, *J* = 2.9 Hz), 128.5, 128.0, 127.8, 123.6 (d, *J* = 3.4 Hz), 115.4 (d, *J* = 23.7 Hz), 61.6, 50.6, 44.9, 43.7, 21.2, 18.2. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -117.41. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{28}H_{26}N_3O_4NaF$, 510.1800; Found: 510.1805.

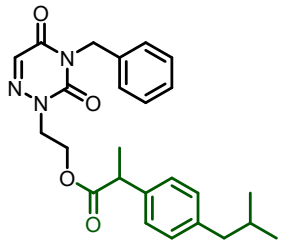
2-(4-benzyl-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-acetoxybenzoate (1x)



Purification by flash column chromatography (PE:EA, 10:1 v/v). Colorless oil (500.3 mg, 90% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.58 (ddd, *J* = 8.1,

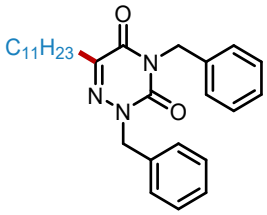
7.4, 1.7 Hz, 1H), 7.51 – 7.46 (m, 2H), 7.40 (s, 1H), 7.30 – 7.24 (m, 4H), 7.11 (dd, *J* = 8.1, 1.2 Hz, 1H), 5.10 (s, 2H), 4.61 (dd, *J* = 5.8, 4.6 Hz, 2H), 4.35 (dd, *J* = 5.7, 4.7 Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.6, 163.9, 155.8, 151.0, 148.9, 135.3, 134.7, 134.2, 131.8, 129.3, 128.6, 128.1, 126.1, 123.9, 122.5, 61.6, 50.7, 44.0, 20.9. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{21}H_{19}N_3O_6Na$, 432.1166; Found: 432.1169.

2-(4-(4-methylbenzyl)-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (1y)



Purification by flash column chromatography (PE:EA, 20:1 v/v). Colorless oil (600.7 mg, 90% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 (dd, *J* = 7.9, 1.7 Hz, 2H), 7.40 – 7.29 (m, 3H), 7.23 (s, 1H), 7.17 – 7.10 (m, 2H), 7.10 – 7.02 (m, 2H), 5.07 (d, *J* = 1.0 Hz, 2H), 4.47 – 4.35 (m, 2H), 4.25 – 4.09 (m, 2H), 3.62 (q, *J* = 7.2 Hz, 1H), 2.47 (d, *J* = 7.2 Hz, 2H), 1.88 – 1.78 (m, 1H), 1.45 (d, *J* = 7.2 Hz, 3H), 0.93 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 155.7, 148.7, 140.7, 137.3, 135.4, 134.3, 129.4, 129.3, 128.6, 128.2, 127.2, 61.2, 50.6, 45.01, 44.95, 43.9, 30.2, 22.4, 18.3. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{25}H_{29}N_3O_4Na$, 458.2050; Found: 458.2056.

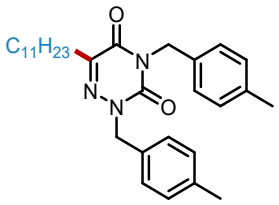
2,4-dibenzyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3a)



Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (69.7 mg, 78% yield), mp 43.1 – 45.1 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 2.0 Hz, 1H), 7.49 (d, *J* = 1.6 Hz, 1H), 7.43 (dd, *J* = 7.9, 1.8 Hz, 2H), 7.40 – 7.29 (m, 6H), 5.13 (s, 2H), 5.11 (s, 2H), 2.66 – 2.58 (m, 2H), 1.63 (d, *J* = 5.6 Hz, 2H), 1.37 – 1.29 (m, 16H), 0.91 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.1, 149.0, 145.8, 135.9, 135.8, 129.4, 128.7,

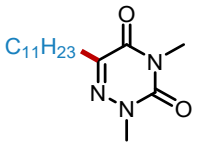
128.6, 128.1, 128.0, 55.2, 44.2, 31.9, 30.3, 29.64, 29.57, 29.37, 29.35, 29.2, 26.2, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{28}H_{37}N_3O_2Na$, 470.2778; Found: 470.2780.

2,4-bis(4-methylbenzyl)-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3b)



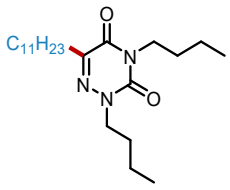
Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (76.1 mg, 80% yield), mp 60.3 – 62.3 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 8.1 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.17 (dd, *J* = 11.7, 7.8 Hz, 4H), 5.08 (s, 2H), 5.08 (s, 2H), 2.68 – 2.59 (m, 2H), 2.38 (s, 3H), 2.36 (s, 3H), 1.65 (t, *J* = 7.3 Hz, 2H), 1.39-1.32 (m, 16H), 0.95 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.1, 149.0, 145.7, 137.9, 137.8, 133.0, 132.9, 129.5, 129.3, 129.2, 128.7, 55.0, 43.9, 32.0, 30.3, 29.7, 29.6, 29.41, 29.39, 29.2, 26.2, 22.7, 21.2, 14.2. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{30}H_{41}N_3O_2Na$, 498.3091; Found: 498.3089.

2,4-dimethyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3c)



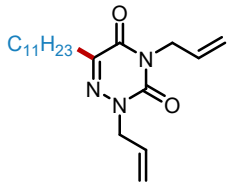
Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (42.5 mg, 72% yield), mp 40.9 – 42.9 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.62 (s, 3H), 3.35 (s, 3H), 2.79 – 2.43 (m, 2H), 1.61 (td, *J* = 8.9, 8.3, 4.6 Hz, 2H), 1.64 – 1.27 (m, 16H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.5, 149.4, 145.2, 39.3, 31.9, 30.4, 29.6, 29.5, 29.3, 29.2, 27.1, 26.5, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{16}H_{29}N_3O_2Na$, 318.2152; Found: 318.2148.

2,4-dibutyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3d)



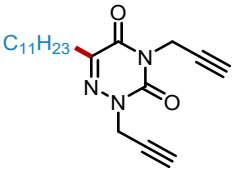
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (61.4 mg, 81% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.96 – 3.90 (m, 4H), 2.64 – 2.53 (m, 2H), 1.72 (td, *J* = 7.1, 3.1 Hz, 2H), 1.65 – 1.58 (m, 4H), 1.41 – 1.26 (m, 20H), 0.95 (td, *J* = 7.4, 4.3 Hz, 6H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.1, 148.9, 145.2, 51.2, 40.7, 31.9, 30.3, 30.3, 29.6, 29.5, 29.3, 29.2, 26.4, 22.7, 20.2, 19.7, 14.1, 13.7. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{22}H_{42}N_3O_2$, 380.3272; Found: 380.3271.

2,4-diallyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3e)



Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (52.9 mg, 75% yield), mp 39.1 – 41.1 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.02 – 5.92 (m, 1H), 5.92 – 5.82 (m, 1H), 5.37 – 5.29 (m, 2H), 5.27 – 5.21 (m, 2H), 4.57 (dd, *J* = 2.9, 1.5 Hz, 2H), 4.56 – 4.53 (m, 2H), 2.68 – 2.58 (m, 2H), 1.65 – 1.61 (m, 2H), 1.41 – 1.19 (m, 16H), 0.90 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.8, 148.6, 145.8, 131.6, 130.5, 119.1, 118.8, 53.9, 42.9, 31.9, 30.3, 29.6, 29.5, 29.33, 29.23, 29.2, 26.4, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{20}H_{34}N_3O_2$, 348.2651; Found: 348.2650.

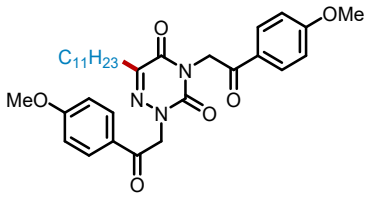
2,4-di(prop-2-yn-1-yl)-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3f)



Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (53.5 mg, 78% yield), mp 34.1 – 36.1 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.76 (d, *J* = 2.50 Hz, 2H), 4.71 (d, *J* = 2.5 Hz, 2H), 2.66 (t, *J* = 7.7 Hz, 2H), 2.37 (s, 1H), 2.24 (s, 1H), 1.66 (t, *J* = 7.5 Hz, 2H),

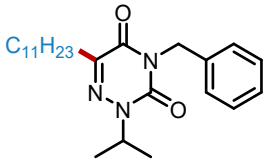
1.43 – 1.18 (m, 16H), 0.90 (t, J = 6.7 Hz, 3H).^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.1, 147.7, 146.5, 73.3, 71.6, 41.2, 31.9, 30.4, 30.0, 29.6, 29.5, 29.33, 29.29, 29.2, 26.2, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{20}H_{30}N_3O_2$, 344.2333; Found: 344.2341.

2,4-bis(2-(4-methoxyphenyl)-2-oxoethyl)-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3g)



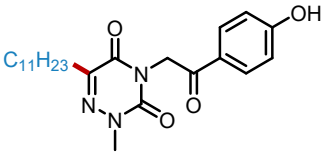
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (85.6 mg, 76% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, J = 7.5 Hz, 2H), 7.96 (d, J = 8.1 Hz, 2H), 6.96 (dd, J = 8.9, 3.6 Hz, 4H), 5.36 (s, 2H), 5.36 (s, 2H), 3.87 (s, 3H), 3.86 (s, 3H), 2.70 – 2.53 (m, 2H), 1.74 – 1.54 (m, 2H), 1.48 – 1.15 (m, 16H), 0.88 (t, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.1, 188.9, 164.2, 164.1, 155.9, 149.2, 146.1, 130.5, 130.4, 127.6, 127.5, 114.1, 114.0, 57.1, 55.53, 55.52,46.2, 31.9, 30.4, 29.6, 29.5, 29.4, 29.3, 29.2, 26.3, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{32}H_{41}N_3O_6Na$, 586.2888; Found: 586.2888.

4-benzyl-2-isopropyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3h)



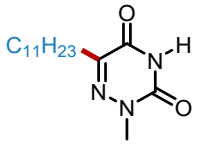
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (61.4 mg, 77% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 (d, J = 1.8 Hz, 1H), 7.52 (d, J = 1.5 Hz, 1H), 7.40 – 7.30 (m, 3H), 5.12 (s, 2H), 4.98 – 4.92 (m, 1H), 3.00 – 2.50 (m, 2H), 1.65 – 1.59 (m, 2H), 1.44 – 1.21 (m, 23H), 0.91 (t, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.9, 148.8, 145.1, 136.0, 129.5, 128.5, 127.9, 50.4, 44.1, 31.9, 30.3, 29.63, 29.61, 29.58, 29.4, 29.3, 29.2, 26.1, 22.7, 20.6, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{24}H_{37}N_3O_2Na$, 422.2778; Found: 422.2776.

4-(2-(4-hydroxyphenyl)-2-oxoethyl)-2-methyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3i)



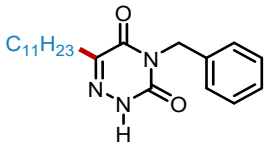
Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (53.9 mg, 65% yield), mp 61.5 – 63.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 5.34 (s, 2H), 3.37 (s, 3H), 2.62 (t, *J* = 7.7 Hz, 2H), 1.60-1.62 (m, 2H), 1.37-1.25 (m, 16H), 0.88 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.5, 162.0, 156.4, 150.1, 146.6, 130.6, 126.7, 115.8, 57.3, 31.9, 30.4, 29.6, 29.5, 29.32, 29.30, 29.2, 27.3, 26.3, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{23}H_{33}N_3O_4Na$, 438.2363; Found: 438.2368.

2-methyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3j)



Purification by flash column chromatography (PE:EA, 50:1 v/v). Yellow solid (38.8 mg, 69% yield), mp 68.1 – 70.1 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.33 (s, 1H), 3.36 (s, 3H), 2.67 – 2.51 (m, 2H), 1.72 – 1.53 (m, 2H), 1.42 – 1.11 (m, 16H), 0.89 (t, *J* = 6.81 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.4, 150.5, 146.4, 31.9, 30.3, 29.63, 29.61, 29.5, 29.3, 29.2, 26.5, 26.2, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_{28}N_3O_2$, 282.2176; Found: 282.2181.

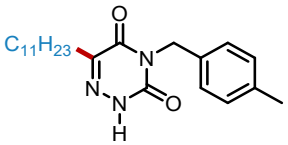
4-benzyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3k)



Purification by flash column chromatography (PE:EA, 50:1 v/v). White solid (50.1 mg, 70% yield), mp 63.2 – 65.2 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.60 (s, 1H), 7.53 – 7.51 (m, 2H), 7.38 – 7.30 (m, 3H), 5.10 (s, 2H), 2.86 – 2.34 (m, 2H), 1.97 – 1.53 (m, 2H), 1.48 – 1.08

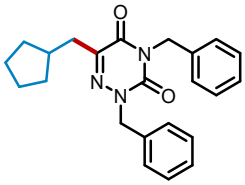
(m, 16H), 0.90 (t, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.1, 146.8, 135.5, 129.4, 128.6, 128.1, 43.6, 31.9, 30.3, 29.6, 29.5, 29.3, 29.2, 26.1, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{21}H_{31}N_3O_2Na$, 380.2308; Found: 380.2307.

4-(4-methylbenzyl)-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3l)



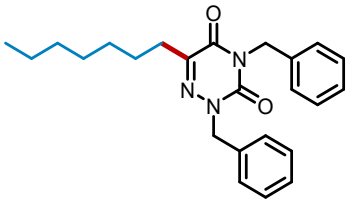
Purification by flash column chromatography (PE:EA, 50:1 v/v). White solid (52.7 mg, 71% yield), mp 57.1 – 59.1 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.04 (s, 1H), 7.42 (s, 1H), 7.40 (s, 1H), 7.16 (s, 1H), 7.14 (s, 1H), 5.07 (s, 2H), 2.68 – 2.52 (m, 2H), 2.34 (s, 3H), 1.63 (t, J = 7.7 Hz, 2H), 1.44 – 1.18 (m, 16H), 0.91 (t, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.1, 150.1, 146.8, 137.9, 132.5, 129.5, 129.3, 43.3, 31.9, 30.3, 29.6, 29.5, 29.3, 29.2, 26.2, 22.7, 21.2, 14.1. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{22}H_{33}N_3O_2Na$, 394.2465; Found: 394.2465.

2,4-dibenzyl-6-(cyclopentylmethyl)-1,2,4-triazine-3,5(2H,4H)-dione (3m)



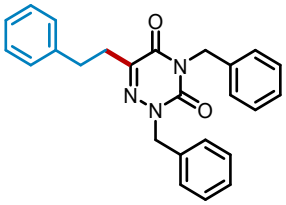
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (45.1 mg, 60% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 – 7.50 (m, 2H), 7.46 – 7.30 (m, 8H), 5.13 (s, 2H), 5.12 (s, 2H), 2.65 (d, J = 7.3 Hz, 2H), 2.33 – 2.25 (m, 1H), 1.85 – 1.50 (m, 8H), 1.27 – 1.17 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.2, 149.0, 145.5, 135.9, 135.8, 129.5, 128.7, 128.6, 128.2, 128.0, 55.2, 44.2, 37.1, 36.2, 32.4, 25.0. HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{23}H_{25}N_3O_2Na$ 398.1839; Found: 398.1843.

2,4-dibenzyl-6-heptyl-1,2,4-triazine-3,5(2H,4H)-dione (3n)



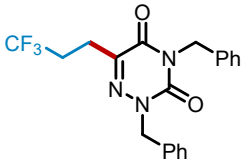
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (50.8 mg, 65% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 – 7.49 (m, 2H), 7.44 (dd, *J* = 8.0, 1.8 Hz, 2H), 7.39 – 7.31 (m, 6H), 5.12 (s, 2H), 5.11 (s, 2H), 2.66 – 2.60 (m, 2H), 1.65 (dd, *J* = 8.8, 6.0 Hz, 2H), 1.37 – 1.28 (m, 8H), 0.93 (q, *J* = 2.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.1, 149.0, 145.8, 135.9, 135.8, 129.4, 128.69, 128.68, 128.6, 128.2, 128.0, 55.2, 44.2, 31.8, 30.3, 29.1, 29.0, 26.2, 22.6, 14.1. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{29}N_3O_2Na$ 414.2152; Found: 414.2155.

2,4-dibenzyl-6-phenethyl-1,2,4-triazine-3,5(2H,4H)-dione (3o)



Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (47.6 mg, 60% yield), mp 61.2 – 63.2 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 (dd, *J* = 7.9, 1.7 Hz, 2H), 7.43 – 7.04 (m, 13H), 5.13 (s, 2H), 5.11 (s, 2H), 3.02 (d, *J* = 2.8 Hz, 2H), 3.01 (d, *J* = 2.8 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.0, 149.0, 144.7, 140.7, 135.8, 135.7, 129.4, 128.8, 128.64, 128.63, 128.56, 128.5, 128.2, 128.1, 126.2, 55.3, 44.2, 32.1, 31.9. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{23}N_3O_2Na$ 420.1683; Found: 420.1683.

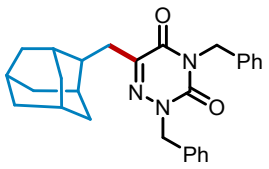
2,4-dibenzyl-6-(3,3,3-trifluoropropyl)-1,2,4-triazine-3,5(2H,4H)-dione (3p)



Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (47.4 mg, 61% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 – 7.47 (m, 2H), 7.46 – 7.31 (m, 8H), 5.13 (s, 2H), 5.12 (s, 2H), 2.96 – 2.89 (m, 2H), 2.59 – 2.40 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.6, 148.8, 142.3, 135.5, 135.4, 129.5, 128.8, 128.7, 128.6, 128.4, 128.2, 126.7 (q, *J* = 276.4

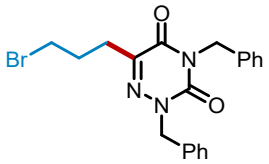
Hz), 55.4, 44.3, 30.0 (q, J = 29.6 Hz), 29.7, 23.4 (q, J = 3.4 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.32. HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{20}H_{18}N_3O_2NaF_3$ 412.1243; Found: 412.1245.

6-(((1R,3S,5r,7r)-adamantan-2-yl)methyl)-2,4-dibenzyl-1,2,4-triazine-3,5(2H,4H)-dione (3q)



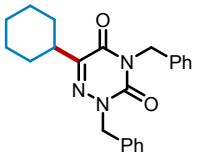
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (50.9 mg, 57% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 – 7.47 (m, 2H), 7.45 – 7.40 (m, 2H), 7.38 – 7.30 (m, 6H), 5.12 (s, 2H), 5.11 (s, 2H), 2.43 (s, 2H), 1.97 – 1.91 (m, 3H), 1.72 – 1.66 (m, 3H), 1.61 – 1.56 (m, 3H), 1.52 (d, J = 2.8 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.6, 148.9, 143.7, 135.9, 135.8, 129.3, 128.7, 128.63, 128.55, 128.1, 128.0, 55.2, 44.3, 42.7, 42.3, 36.8, 34.5, 28.6. HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{28}H_{31}N_3O_2Na$ 464.2308; Found: 464.2310.

2,4-dibenzyl-6-(3-bromopropyl)-1,2,4-triazine-3,5(2H,4H)-dione (3r)



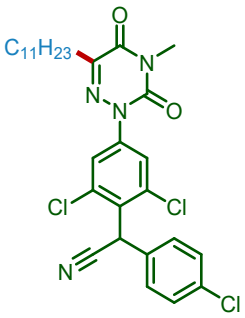
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (49.6 mg, 60% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 – 7.48 (m, 2H), 7.45 – 7.32 (m, 8H), 5.12 (s, 2H), 5.11 (s, 2H), 3.46 (t, J = 6.6 Hz, 2H), 2.81 (t, J = 7.3 Hz, 2H), 2.23 (p, J = 6.8 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.9, 148.9, 144.1, 135.7, 135.6, 129.5, 128.81, 128.78, 128.6, 128.3, 128.1, 55.2, 44.3, 32.6, 28.9, 28.8. HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{20}H_{20}N_3O_2NaBr$ 436.0631; Found: 436.0635.

2,4-dibenzyl-6-cyclohexyl-1,2,4-triazine-3,5(2H,4H)-dione (3s)



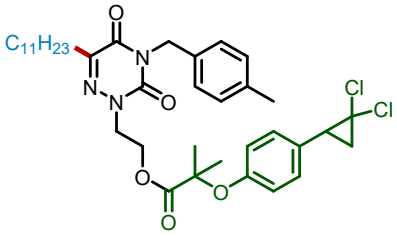
Purification by flash column chromatography (PE:EA, 60:1 v/v). White solid (47.3 mg, 63% yield), mp 71.1– 73.1 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 – 7.50 (m, 2H), 7.46 – 7.42 (m, 2H), 7.40 – 7.30 (m, 6H), 5.11 (s, 2H), 5.11 (s, 2H), 2.94 – 2.87(m, 1H), 1.94 – 1.75 (m, 5H), 1.45 – 1.28 (m, 5H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.7, 149.0, 148.9, 135.93, 135.86, 129.5, 128.8, 128.7, 128.5, 128.1, 128.0, 55.3, 44.2, 38.5, 30.5, 26.1, 26.0. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{25}N_3O_2Na$ 398.1839; Found: 398.1839.

2-(2,6-dimethyl-4-(4-methyl-3,5-dioxo-6-undecyl-4,5-dihydro-1,2,4-triazin-2(3H)-yl)phenyl)-2-(p-tolyl)acetonitrile (3t)



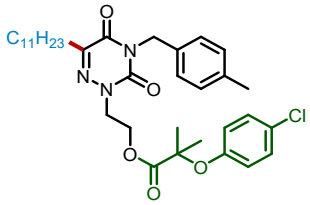
Purification by flash column chromatography (PE:EA, 40:1 v/v). Yellow oil (49.2 mg, 46% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.80 (s, 2H), 7.38 – 7.32 (m, 4H), 6.20 (s, 1H), 3.45 (s, 3H), 2.73 (d, J = 7.7 Hz, 2H), 1.70 (t, J = 7.8 Hz, 2H), 1.29 (d, J = 8.5 Hz, 16H), 0.91 (d, J = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.4, 148.2, 147.6, 141.7, 135.7, 134.3, 130.9, 129.6, 129.2, 128.2, 124.8, 116.3, 37.0, 31.9, 30.6, 29.62, 29.59, 29.5, 29.32, 29.30, 29.2, 27.6, 26.3, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{29}H_{33}Cl_3N_4NaO_2$, 597.1561; Found: 597.1520.

2-(4-(4-methylbenzyl)-3,5-dioxo-6-undecyl-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (3u)



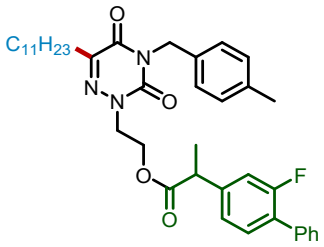
Purification by flash column chromatography (PE:EA, 60:1 v/v). Yellow oil (68.53 mg, 50% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 7.9 Hz, 2H), 7.06 – 7.02 (m, 2H), 6.77 – 6.72 (m, 2H), 4.99 (s, 2H), 4.46 (t, *J* = 5.2 Hz, 2H), 4.23 – 4.16 (m, 2H), 2.80 (dd, *J* = 10.7, 8.3 Hz, 1H), 2.55 – 2.48 (m, 2H), 2.30 (s, 3H), 1.92 (dd, *J* = 10.7, 7.4 Hz, 1H), 1.75 (dd, *J* = 8.4, 7.4 Hz, 1H), 1.61 – 1.54 (m, 2H), 1.51 (s, 6H), 1.26 (s, 16H), 0.89 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.1, 156.0, 154.9, 149.2, 146.2, 138.0, 132.9, 129.7, 129.6, 129.4, 128.4, 118.9, 62.3, 61.0, 50.2, 44.0, 35.0, 32.1, 30.5, 29.8, 29.8, 29.7, 29.50, 29.46, 26.5, 26.0, 25.5, 25.4, 22.8, 21.3, 14.3. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{37}H_{49}N_3O_5NaCl_2$, 708.2941; Found: 708.2947.

2-(4-(4-methylbenzyl)-3,5-dioxo-6-undecyl-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-(4-chlorophenoxy)-2-methylpropanoate (3v)



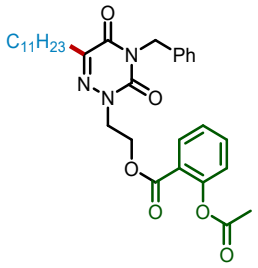
Purification by flash column chromatography (PE:EA, 60:1 v/v). Yellow oil (63.5 mg, 52% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 8.1 Hz, 2H), 7.26 (s, 1H), 7.17 – 7.06 (m, 4H), 6.75 – 6.65 (m, 2H), 4.98 (s, 2H), 4.45 (dd, *J* = 5.7, 4.5 Hz, 2H), 4.20 (dd, *J* = 5.7, 4.5 Hz, 2H), 2.30 (s, 3H), 1.50 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.7, 155.8, 153.9, 149.1, 146.1, 137.8, 129.5, 129.2, 129.1, 127.3, 120.47, 120.46, 62.3, 50.1, 43.9, 31.9, 30.4, 29.7, 29.6, 29.5, 29.4, 29.3, 26.4, 25.2, 22.7, 21.2, 14.2. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{34}H_{46}N_3O_5NaCl$, 634.3018; Found: 634.3021.

2-(4-(4-methylbenzyl)-3,5-dioxo-6-undecyl-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (3w)



Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (65.4 mg, 51% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (dt, *J* = 8.1, 1.5 Hz, 2H), 7.50 – 7.35 (m, 6H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.11 – 7.06 (m, 2H), 5.07 (d, *J* = 2.1 Hz, 2H), 4.56 – 4.15 (m, 4H), 3.67 (q, *J* = 7.2 Hz, 1H), 2.59 (dd, *J* = 8.6, 6.9 Hz, 2H), 2.34 (s, 3H), 1.61 (dd, *J* = 9.8, 5.1 Hz, 2H), 1.51 (d, *J* = 7.2 Hz, 3H), 1.32 (d, *J* = 16.7 Hz, 16H), 0.92 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.7, 159.7 (d, *J* = 248.6 Hz), 155.9, 149.2, 146.1, 141.5 (d, *J* = 7.7 Hz), 137.9, 135.4, 132.8, 130.8 (d, *J* = 4.0 Hz), 129.5, 129.2, 129.0 (d, *J* = 3.0 Hz), 128.5, 127.9 (d, *J* = 13.5 Hz), 127.7, 123.5 (d, *J* = 3.4 Hz), 115.3 (d, *J* = 23.6 Hz), 61.8, 50.0, 44.9, 43.9, 31.9, 30.4, 29.7, 29.6, 29.4, 29.3, 26.4, 22.7, 21.2, 18.3, 14.2. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -117.36. HRMS (ESI-TOF) *m/z*: [M + Na]$^{+}$ Calcd for $C_{39}H_{48}N_3O_4NaF$, 664.3521; Found: 664.3527.

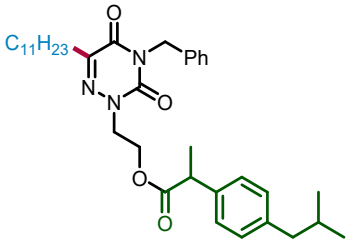
2-(4-benzyl-3,5-dioxo-6-undecyl-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-acetoxybenzoate (3x)



Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (45.1 mg, 40% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.58 (td, *J* = 7.8, 1.7 Hz, 1H), 7.52 – 7.46 (m, 2H), 7.34 – 7.27 (m, 4H), 7.11 (dd, *J* = 8.1, 1.2 Hz, 1H), 5.11 (s, 2H), 4.61 (dd, *J* = 5.8, 4.7 Hz, 2H), 4.34 (dd, *J* = 5.8, 4.7 Hz, 2H), 2.60 – 2.54 (m, 2H), 2.28 (s, 3H), 1.57 – 1.49 (m, 2H), 1.32 – 1.25 (m, 16H), 0.91 (t, *J* = 6.78 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.6, 163.9, 156.0, 151.0, 149.2, 146.2, 135.7, 134.1, 131.8, 129.3, 128.5, 128.0, 126.0, 123.9, 122.6, 61.8, 50.3, 44.2, 31.9, 30.3, 29.64, 29.61, 29.5, 29.4, 29.3, 29.3,

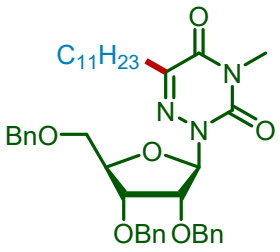
26.2, 22.7, 20.9, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{32}H_{41}N_3O_6Na$, 586.2888; Found: 586.2889.

2-(4-benzyl-3,5-dioxo-6-undecyl-4,5-dihydro-1,2,4-triazin-2(3H)-yl)ethyl 2-(4-isobutylphenyl)propanoate(3y)



Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (58.9 mg, 50% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 – 7.45 (m, 2H), 7.33 – 7.27 (m, 3H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 7.9 Hz, 2H), 5.06 (s, 2H), 4.43 – 4.08 (m, 4H), 3.58 (q, *J* = 7.2 Hz, 1H), 2.57 – 2.50 (m, 2H), 2.42 (d, *J* = 7.2 Hz, 2H), 1.87 – 1.77 (m, 1H), 1.56 (t, *J* = 7.5 Hz, 2H), 1.41 (d, *J* = 7.2 Hz, 3H), 1.28 (d, *J* = 17.5 Hz, 16H), 0.88 (t, *J* = 6.9 Hz, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.2, 155.7, 148.9, 145.7, 140.4, 137.2, 135.5, 129.2, 129.1, 128.3, 127.8, 126.9, 61.2, 49.8, 44.8, 44.7, 43.9, 31.7, 30.1, 30.0, 29.43, 29.40, 29.3, 29.14, 29.05, 26.1, 22.5, 22.2, 18.2, 13.9. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{36}H_{51}N_3O_4Na$, 612.3772; Found: 612.3776.

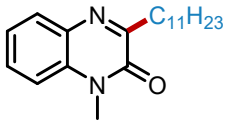
2-((2R,3R,4R,5R)-3,4-bis(benzyloxy)-5-((benzyloxy)methyl)tetrahydrofuran-2-yl)-4-methyl-6-undecyl-1,2,4-triazine-3,5(2H,4H)-dione (3z)



Purification by flash column chromatography (PE:EA, 60:1 v/v). Yellow oil (69.7 mg, 51% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.25 (m, 15H), 6.37 (d, *J* = 3.6 Hz, 1H), 4.65 – 4.54 (m, 4H), 4.50 (d, *J* = 2.2 Hz, 2H), 4.35 (q, *J* = 5.0 Hz, 1H), 4.29 (dd, *J* = 5.3, 3.7 Hz, 1H), 4.19 (t, *J* = 5.5 Hz, 1H), 3.63 – 3.54 (m, 2H), 3.30 (s, 3H), 2.49 – 2.41 (m, 2H), 1.47 – 1.40 (m, 2H), 1.25 (s, 16H), 0.87 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ

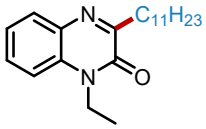
155.8, 149.0, 145.7, 138.0, 137.7, 137.4, 128.43, 128.37, 128.3, 128.1, 128.0, 127.9, 127.6, 127.5, 89.2, 81.2, 78.3, 73.3, 72.4, 72.3, 70.0, 31.9, 30.2, 29.72, 29.67, 29.66, 29.43, 29.38, 29.2, 27.2, 25.8, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{41}H_{53}N_3O_6Na$, 706.3827; Found: 706.3826.

1-methyl-3-undecylquinoxalin-2(1H)-one (5a)[5]



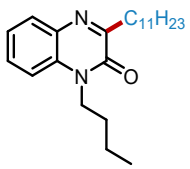
Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (45.8 mg, 73% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.54 – 7.50 (m, 1H), 7.37 – 7.27 (m, 2H), 3.70 (s, 3H), 2.99 – 2.87 (m, 2H), 1.84 – 1.73 (m, 2H), 1.45 – 1.23 (m, 16H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.4, 155.0, 133.1, 132.8, 129.6, 129.5, 123.5, 113.5, 34.4, 31.9, 29.7, 29.62, 29.57, 29.5, 29.4, 29.0, 26.9, 22.7, 14.1.

1-ethyl-3-undecylquienoxalin-2(1H)-one (5b)



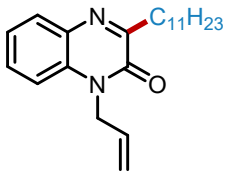
Purification by flash column chromatography (PE:EA, 50:1 v/v). Yellow oil (47.2 mg, 72% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.51 (td, *J* = 7.7, 7.1, 1.6 Hz, 1H), 7.32 (dd, *J* = 8.1, 6.1 Hz, 2H), 4.32 (q, *J* = 7.2 Hz, 2H), 2.98 – 2.90 (m, 2H), 1.78 (q, *J* = 7.7 Hz, 2H), 1.46 – 1.25 (m, 19H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.5 154.4, 133.1, 132.0, 129.9, 129.4, 123.3, 113.4, 37.2, 34.3, 31.9, 29.7, 29.63, 29.58, 29.5, 29.4, 26.9, 22.7, 14.1, 12.4. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{21}H_{33}N_2O$, 329.2587; Found: 329.2589.

1-butyl-3-undecylquinoxalin-2(1H)-one (5c)



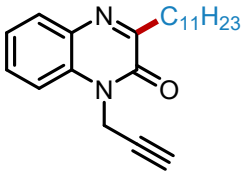
Purification by flash column chromatography (PE:EA, 50:1 v/v). Colorless oil (49.8 mg, 70% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.52 – 7.48 (m, 1H), 7.35 – 7.27 (m, 2H), 4.28 – 4.21 (m, 2H), 2.98 – 2.89 (m, 2H), 1.83 – 1.69 (m, 4H), 1.53 – 1.42 (m, 4H), 1.37 – 1.23 (m, 14H), 1.00 (t, *J* = 7.4 Hz, 3H), 0.90 – 0.85 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.5, 154.6, 133.1, 132.3, 129.9, 129.4, 123.3, 113.6, 42.1, 34.3, 31.9, 29.7, 29.63, 29.58, 29.5, 29.4, 29.3, 26.9, 22.7, 20.3, 14.1, 13.8. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{37}N_2O$, 357.2900; Found: 357.2903.

1-allyl-3-undecylquinoxalin-2(1H)-one (5d)



Purification by flash column chromatography (PE:EA, 40:1 v/v). Colorless oil (47.6 mg, 70% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.52 – 7.48 (m, 1H), 7.37 – 7.28 (m, 2H), 6.01 – 5.91 (m, 1H), 5.28 (d, *J* = 10.4 Hz, 1H), 5.18 (d, *J* = 17.4 Hz, 1H), 4.93 (dd, *J* = 4.4, 2.6 Hz, 2H), 3.00 – 2.92 (m, 2H), 1.85 – 1.77(m, 2H), 1.47 (td, *J* = 8.7, 8.3, 4.3 Hz, 2H), 1.37 – 1.27 (m, 14H), 0.90 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.5, 154.5, 132.9, 132.3, 130.8, 129.7, 129.4, 123.5, 118.0, 114.1, 44.5, 34.3, 31.9, 29.7, 29.63, 29.58, 29.5, 29.4, 26.9, 22.7, 14.1. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{33}N_2O$, 341.2587; Found: 341.2585.

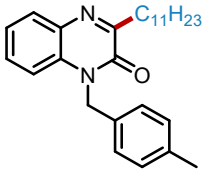
1-(prop-2-yn-1-yl)-3-undecylquinoxalin-2(1H)-one (5e)



Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (46.6 mg, 69% yield), mp 37.5 – 39.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.57 – 7.53 (m, 1H), 7.45 (dd, *J* = 8.4, 1.3 Hz, 1H), 7.36 (td, *J* = 7.6, 1.3 Hz, 1H), 5.06 (d, *J* = 2.6 Hz, 2H), 2.98 – 2.91 (m, 2H), 2.28 (t, *J* = 2.5 Hz, 1H), 1.83 – 1.75 (m, 2H), 1.42 – 1.24 (m, 16H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.3, 153.9, 133.0,

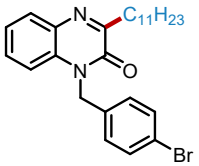
131.6, 129.8, 129.6, 123.9, 114.0, 73.1, 34.3, 31.9, 31.4, 29.7, 29.64, 29.60, 29.57, 29.5, 29.4, 26.8, 22.7, 14.1. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{22}H_{30}N_2ONa$, 361.2250; Found: 361.2256.

1-(4-methylbenzyl)-3-undecylquinoxalin-2(1H)-one (5f)



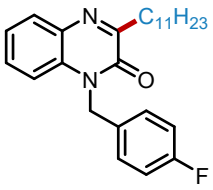
Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (57.4 mg, 71% yield), mp 42.5 – 44.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.42 – 7.34 (m, 1H), 7.30 – 7.24 (m, 2H), 7.15 – 7.09 (m, 4H), 5.45 (s, 2H), 3.04 – 2.95 (m, 2H), 2.30 (s, 3H), 1.87 – 1.78 (m, 2H), 1.47 – 1.24 (m, 16H), 0.90 – 0.86 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.6, 155.0, 137.4, 133.0, 132.5, 132.4, 129.7, 129.6, 129.4, 126.9, 123.5, 114.4, 45.6, 34.4, 31.9, 29.68, 29.66, 29.6, 29.5, 29.4, 26.9, 22.7, 21.1, 14.1. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{27}H_{36}N_2ONa$, 427.2720; Found: 427.2723.

1-(4-bromobenzyl)-3-undecylquinoxalin-2(1H)-one (5g)



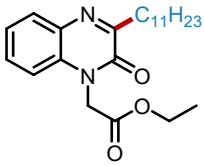
Purification by flash column chromatography (PE:EA, 40:1 v/v). Yellow oil (65.5 mg, 70% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.48 – 7.39 (m, 3H), 7.32 (td, *J* = 7.6, 1.3 Hz, 1H), 7.19 (dd, *J* = 8.4, 1.3 Hz, 1H), 7.14 (d, *J* = 8.5 Hz, 2H), 5.45 (s, 2H), 3.04 – 2.96 (m, 2H), 1.90 – 1.79 (m, 2H), 1.46 – 1.28 (m, 16H), 0.91 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.5, 154.9, 134.4, 133.0, 132.2, 132.1, 129.9, 129.6, 128.7, 123.7, 121.6, 114.1, 45.3, 34.4, 31.9, 29.7, 29.64, 29.63, 29.59, 29.5, 29.4, 26.9, 22.7, 14.1. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{26}H_{34}N_2OBr$, 469.1849; Found: 469.1857.

1-(4-fluorobenzyl)-3-undecylquinoxalin-2(1H)-one (5h)



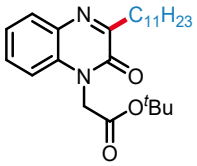
Purification by flash column chromatography (PE:EA, 40:1 v/v). Yellow oil (55.5 mg, 68% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.45 – 7.41 (m, 1H), 7.32 (td, *J* = 7.7, 1.3 Hz, 1H), 7.28 – 7.21 (m, 3H), 7.02 (t, *J* = 8.6 Hz, 2H), 5.47 (s, 2H), 3.08 – 2.95 (m, 2H), 1.88 – 1.81 (m, 2H), 1.53 – 1.44 (m, 2H), 1.42 – 1.26 (m, 14H), 0.90 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 162.2 (d, *J* = 246.4 Hz), 161.5, 154.9, 133.0, 132.3, 131.2 (d, *J* = 3.3 Hz), 129.9, 129.5, 128.7 (d, *J* = 8.1 Hz), 123.7, 115.9 (d, *J* = 21.7 Hz), 114.1, 45.2, 34.4, 31.9, 29.7, 29.64, 29.60, 29.5, 29.4, 26.9, 22.7, 14.1. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -114.4. HRMS (ESI-TOF) m/z: [M + Na]$^{+}$ Calcd for $C_{26}H_{33}N_2ONaF$, 431.2469; Found: 431.2473.

ethyl 2-(2-oxo-3-undecylquinoxalin-1(2H)-yl)acetate (5i)



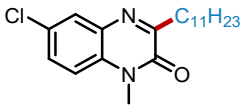
Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (56.3 mg, 73% yield), mp 64.5 – 66.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.50 – 7.45 (m, 1H), 7.36 – 7.30 (m, 1H), 7.05 (d, *J* = 8.3 Hz, 1H), 5.02 (s, 2H), 4.25 (q, *J* = 7.1 Hz, 2H), 2.98 – 2.92 (m, 2H), 1.79 (p, *J* = 7.6 Hz, 2H), 1.48 – 1.41 (m, 2H), 1.36 – 1.23 (m, 17H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.2, 161.2, 154.5, 132.8, 132.2, 130.0, 129.6, 123.8, 113.0, 62.0, 43.5, 34.2, 31.9, 29.7, 29.62, 29.58, 29.56, 29.5, 29.3, 26.7, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: [M + Na]$^{+}$ Calcd for $C_{23}H_{34}N_2O_3Na$, 409.2462; Found: 409.2466.

tert-butyl 2-(2-oxo-3-undecylquinoxalin-1(2H)-yl)acetate (5j)



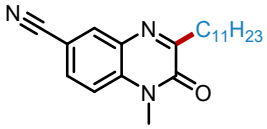
Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (56.3 mg, 68% yield), mp 52.5 – 54.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.53 – 7.48 (m, 1H), 7.35 – 7.30 (m, 2H), 4.29 – 4.22 (m, 2H), 2.98 – 2.93 (m, 2H), 1.83 – 1.73 (m, 4H), 1.53 – 1.40 (m, 4H), 1.43 – 1.19 (m, 16H), 1.02 (t, *J* = 7.4 Hz, 3H), 0.92 – 0.88 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.5, 154.6, 133.0, 132.3, 129.9, 129.4, 123.3, 113.6, 42.1, 34.3, 31.9, 29.7, 29.63, 29.58, 29.5, 29.4, 29.3, 26.9, 22.7, 20.3, 14.1, 13.8. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{25}H_{38}N_2O_3Na$, 437.2775; Found: 437.2778.

6-chloro-1-methyl-3-undecylquinoxalin-2(1H)-one (5k)



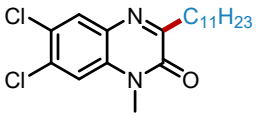
Purfication by flash column chromatography (PE:EA, 40:1 v/v). Yellow solid (49.4 mg, 71% yield), mp 32.1 – 34.1 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.78 – 7.73 (m, 1H), 3.67 (s, 3H), 2.98 – 2.88 (m, 2H), 1.81 – 1.76 (m, 2H), 1.28 (d, *J* = 6.8 Hz, 16H), 0.90 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.5, 154.6, 135.3, 134.0, 131.2, 130.7, 123.9, 113.6, 34.3, 31.9, 29.7, 29.62, 29.60, 29.56, 29.5, 29.3, 29.2, 26.7, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{20}H_{30}N_2OCl$, 349.2041; Found: 349.2038.

1-methyl-2-oxo-3-undecyl-1,2-dihydroquinoxaline-6-carbonitrile (5l)



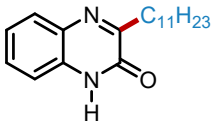
Purification by flash column chromatography (PE:EA, 50:1 v/v). White solid (44.1 mg, 65% yield), mp 56.5 – 58.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (d, *J* = 2.0 Hz, 1H), 7.76 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.38 (d, *J* = 8.7 Hz, 1H), 3.72 (s, 3H), 2.97 – 2.93 (m, 2H), 1.82 – 1.75 (m, 2H), 1.42 – 1.26 (m, 16H), 0.91 – 0.87 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.7, 154.5, 136.4, 133.9, 132.3, 132.1, 118.1, 114.7, 107.0, 34.2, 31.9, 29.64, 29.61, 29.54, 29.46, 29.4, 29.3, 29.3, 26.4, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{21}H_{30}N_3O$, 340.2383; Found: 340.2392.

6,7-dichloro-1-methyl-3-undecylquinoxalin-2(1H)-one (5m)



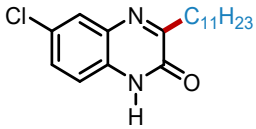
Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (50.4 mg, 66% yield), mp 46.2– 48.2 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.94 (s, 1H), 7.40 (s, 1H), 3.67 (s, 3H), 2.96 – 2.90 (m, 2H), 1.81 – 1.74 (m, 2H), 1.35 – 1.27 (m, 16H), 0.93 – 0.87 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.2, 156.3, 133.3, 129.7, 129.4, 129.3, 128.3, 116.6, 33.5, 31.9, 29.70, 29.67, 29.65, 29.6, 29.49, 29.47, 29.4, 26.6, 22.7, 14.1. HRMS (ESI-TOF) *m/z*: [M + H]$^+$ Calcd for $C_{20}H_{29}N_2OCl_2$, 383.1651; Found: 383.1659.

3-undecylquinoxalin-2(1H)-one (5n)



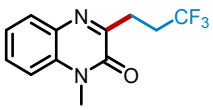
Purification by flash column chromatography (PE:EA, 30:1 v/v). White solid (42.1 mg, 70% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 12.22 (s, 1H), 7.88 – 7.82 (m, 1H), 7.53 – 7.48 (m, 1H), 7.38 – 7.33 (m, 2H), 3.03 – 2.98 (m, 2H), 1.89 – 1.82 (m, 2H), 1.47 – 1.27 (m, 16H), 0.92 – 0.88 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.9, 156.6, 132.9, 130.9, 129.6, 128.7, 124.1, 115.6, 33.6, 31.9, 29.7, 29.64, 29.58, 29.5, 29.4, 26.9, 22.7, 14.1.

6-chloro-3-undecylquinoxalin-2(1H)-one (5o)



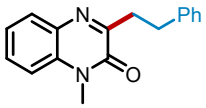
Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (47.4 mg, 71% yield), mp 74.5 – 76.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 12.09 (s, 1H), 7.86 (d, *J* = 2.3 Hz, 1H), 7.46 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.30 – 7.27 (m, 1H), 3.00 – 2.96 (m, 2H), 1.83 (m, 2H), 1.35 – 1.27 (m, 16H), 0.90 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.2, 156.3, 133.3, 129.7, 129.4, 129.3, 128.3, 116.6, 33.5, 31.9, 29.70, 29.67, 29.65, 29.6, 29.49, 29.47, 29.4, 26.6, 22.7, 14.1. HRMS (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{19}H_{28}N_2OCl$, 335.1885; Found: 335.1890.

1-methyl-3-(3,3,3-trifluoropropyl)quinoxalin-2(1H)-one (5p)[6]



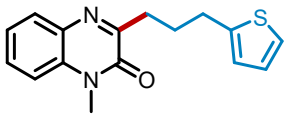
Purification by flash column chromatography (PE:EA, 30:1 v/v). White solid (28.1 mg, 55% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.60 – 7.56 (m, 1H), 7.42 – 7.31 (m, 2H), 3.73 (s, 3H), 3.27 – 3.20 (m, 2H), 2.77 – 2.65 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 154.6, 133.1, 132.5 , 130.1, 129.9, 127.2 (q, *J* = 276.2 Hz). 123.7, 113.7, 30.2 (q, *J* = 29.5 Hz). 29.0, 26.6 (q, *J* = 2.6 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.30.

1-methyl-3-phenethylquinoxalin-2(1H)-one (5q)



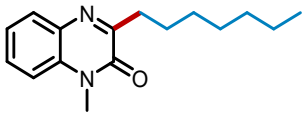
Purification by flash column chromatography (PE:EA, 30:1 v/v). Yellow solid (34.3 mg, 65% yield), mp 68.5 – 70.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.57 – 7.53 (m, 1H), 7.40 – 7.30 (m, 6H), 7.25 – 7.21 (m, 1H), 3.73 (s, 3H), 3.34 – 3.27 (m, 2H), 3.17 (dd, *J* = 9.6, 6.4 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.1, 154.9, 141.7, 133.1, 132.7, 129.7, 128.6, 128.4, 126.0, 123.6, 113.6, 36.0, 32.5, 29.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{17}H_{16}N_2ONa$, 287.1160; Found: 287.1154.

1-methyl-3-(3-(thiophen-2-yl)propyl)quinoxalin-2(1H)-one (5r)



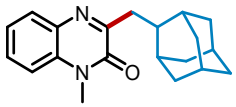
Purification by flash column chromatography (PE:EA, 30:1 v/v). White solid (36.9 mg, 62% yield), mp 130.5 – 132.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.57-7.53 (m, 1H), 7.39 – 7.30 (m, 2H), 7.12 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.93 (dd, *J* = 5.2, 3.4 Hz, 1H), 6.86 (dd, *J* = 3.3, 1.3 Hz, 1H), 3.72 (s, 3H), 3.03 (dt, *J* = 13.6, 7.6 Hz, 4H), 2.27 – 2.19 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 154.9, 145.0, 133.1, 132.7, 129.72, 129.65, 126.7, 124.3, 123.6, 123.0, 113.6, 33.6, 29.7, 29.0, 28.5. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{16}H_{16}N_2ONaS$, 307.0881; Found: 307.0879.

3-heptyl-1-methylquinoxalin-2(1H)-one (5s)



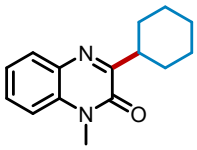
Purification by flash column chromatography (PE:EA, 30:1 v/v). Yellow oil (36.1 mg, 70% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.56 – 7.52 (m, 1H), 7.39 – 7.29 (m, 2H), 3.72 (s, 3H), 3.01 – 2.92 (m, 2H), 1.83 – 1.76 (m, 2H), 1.47 – 1.28 (m, 8H), 0.94 – 0.87 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.4, 154.9, 133.1, 132.8, 129.6, 129.5, 123.5, 113.5, 34.4, 31.8, 29.6, 29.2, 29.0, 26.9, 22.6, 14.1. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{16}H_{22}N_2ONa$, 281.1630; Found: 281.1625.

3-(((1r,3r,5r,7r)-adamantan-2-yl)methyl)-1-methylquinoxalin-2(1H)-one (5t)



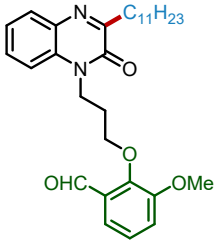
Purification by flash column chromatography (PE:EA, 30:1 v/v). White solid (32.6 mg, 53% yield) mp 101.5 – 103.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (s, 1H), 7.55 (t, *J* = 7.9 Hz, 1H), 7.38 – 7.30 (m, 2H), 3.72 (s, 3H), 2.81 (s, 2H), 1.99 – 1.95 (m, 3H), 1.71 – 1.65 (m, 12H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.6, 133.2, 132.6, 129.8, 129.6, 123.4, 113.5, 46.6, 42.7, 36.9, 35.6, 29.3, 28.9. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{20}H_{24}N_2ONa$, 331.1786; Found: 331.1785.

3-cyclohexyl-1-methylquinoxalin-2(1H)-one (5u)[7]



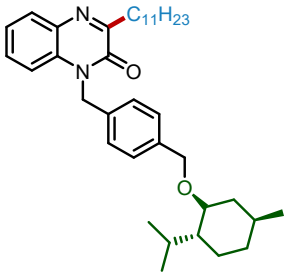
Purification by flash column chromatography (PE:EA, 30:1 v/v). Colorless oil (29.0 mg, 60% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.55 – 7.51 (m, 1H), 7.37 – 7.29 (m, 2H), 3.72 (s, 3H), 3.40 – 3.33 (m, 1H), 2.01 – 1.95 (m, 2H), 1.89 (dt, *J* = 12.8, 3.3 Hz, 2H), 1.81 – 1.76 (m, 1H), 1.62 – 1.46 (m, 4H), 1.33 (dt, *J* = 12.4, 3.6 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 164.3, 154.6, 132.9, 132.9, 129.8, 129.4, 123.4, 113.4, 40.8, 30.5, 29.0, 26.3, 26.2.

3-methoxy-2-(3-(2-oxo-3-undecylquinoxalin-1(2H)-yl)propoxy)benzaldehyde (5v)



Purification by flash column chromatography (PE:EA, 40:1 v/v). Colorless oil (52.9 mg, 50% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.52 (s, 1H), 7.97 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.83 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.64 – 7.59 (m, 1H), 7.57 – 7.53 (m, 1H), 7.45 (t, *J* = 4.7 Hz, 1H), 7.16 (d, *J* = 4.6 Hz, 2H), 4.78 (t, *J* = 6.2 Hz, 2H), 4.38 (t, *J* = 6.2 Hz, 2H), 3.85 (s, 3H), 3.00 – 2.95 (m, 2H), 2.44 – 2.38 (m, 2H), 1.80 (t, *J* = 7.6 Hz, 2H), 1.42 – 1.27 (m, 16H), 0.90 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.0, 155.9, 153.0, 151.7, 151.5, 139.8, 138.6, 130.0, 128.8, 128.2, 126.8, 126.3, 124.1, 119.4, 118.1, 71.7, 63.0, 56.0, 33.7, 31.9, 29.7, 29.6, 29.54, 29.46, 29.3, 27.5, 22.7, 14.1. HRMS (ESI-TOF) m/z: [M + Na]$^{+}$ Calcd for $C_{30}H_{40}N_2O_4Na$, 515.2880; Found: 515.2884.

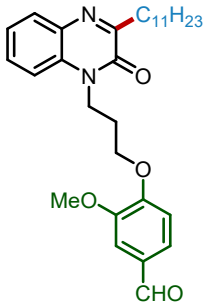
1-(4-((((1R,2R,5S)-2-isopropyl-5-methylcyclohexyl)oxy)methyl)benzyl)-3-undecylquinoxalin-2(1H)-one (5w)



Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (61.4 mg, 55% yield), mp 41.8 – 43.8 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 7.9 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 16.6 Hz, 3H), 7.23 (d, *J* = 8.0 Hz, 3H), 5.51 (s, 2H), 4.63 (d, *J* = 11.5 Hz, 1H), 4.36 (d, *J* = 11.5 Hz, 1H), 3.16 (td, *J* = 10.6, 4.1 Hz, 1H), 3.02 (t, *J* = 7.8 Hz, 2H), 2.32 – 2.24 (m, 1H), 2.18 (d, *J* = 12.4 Hz, 1H), 1.89 – 1.81 (m, 2H), 1.68 (s, 3H), 1.48 (q, *J* = 7.4 Hz, 2H), 1.34 – 1.24 (m, 15H), 0.92 (dd, *J* = 19.3, 7.3 Hz, 12H), 0.69 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.5, 155.0, 138.7, 134.5, 133.0, 132.4, 129.7, 129.4, 128.4, 126.9, 123.5, 114.4, 70.0, 48.3, 45.7, 40.3, 34.6, 34.4, 31.9, 31.6, 29.70, 29.66,

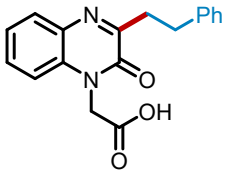
29.64, 29.59, 29.5, 29.4, 26.9, 25.5, 23.2, 22.7, 22.3, 21.0, 16.0, 14.1. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{37}H_{55}N_2O_2$, 559.4258; Found: 559.4264.

3-methoxy-4-(3-(2-oxo-3-undecylquinoxalin-1(2H)-yl)propoxy)benzaldehyde (5x)



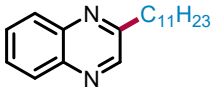
Purification by flash column chromatography (PE:EA, 40:1 v/v). White solid (44.3 mg, 45% yield), mp 56.2 – 58.2 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.52 (s, 1H), 7.97 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.83 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.64 – 7.59 (m, 1H), 7.57 – 7.53 (m, 1H), 7.45 (t, *J* = 4.7 Hz, 1H), 7.16 (d, *J* = 4.6 Hz, 2H), 4.78 (t, *J* = 6.2 Hz, 2H), 4.38 (t, *J* = 6.2 Hz, 2H), 3.85 (s, 3H), 3.00 – 2.95 (m, 2H), 2.44 – 2.38 (m, 2H), 1.80 (t, *J* = 7.61 Hz, 2H), 1.42 – 1.27 (m, 16H), 0.90 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.8, 155.8, 153.9, 151.4, 150.0, 139.7, 138.7, 130.2, 128.8, 128.2, 126.7, 126.6, 126.4, 111.5, 109.4, 66.0, 62.9, 56.0, 33.7, 31.9, 29.65, 29.62, 29.56, 29.5, 29.4, 29.3, 28.7, 27.6, 22.7, 14.1. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{30}H_{40}N_2O_4Na$, 515.2880; Found: 515.2880.

2-(2-oxo-3-phenethylquinoxalin-1(2H)-yl)acetic acid (5y)[8]



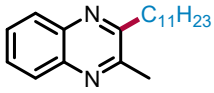
Purify by flash column chromatography (CH_2Cl_2:MeOH, 40:2 v/v). Yellow solid (24.1 mg, 40% yield), ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.83 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.58 (td, *J* = 7.8, 7.2, 1.5 Hz, 1H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 1H), 7.29 (q, *J* = 4.3, 3.3 Hz, 4H), 7.20 – 7.16 (m, 1H), 5.02 (s, 2H), 3.17 – 3.12 (m, 2H), 3.08 – 3.04 (m, 2H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 169.3, 159.5, 154.3, 141.9, 132.8, 132.3, 130.4, 129.5, 128.9, 128.8, 126.4, 124.0, 115.0, 44.1, 35.6, 32.0.

2-undecylquinoxaline (7a)



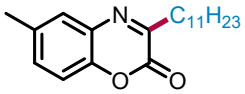
Purification by flash column chromatography (PE:EA, 50:1 v/v), Clorless oil (34.1 mg, 60%, yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.76 (s, 1H), 8.11 – 8.05 (m, 2H), 7.79 – 7.70 (m, 2H), 3.07 – 3.00 (m, 2H), 1.91 – 1.82 (m, 2H), 1.47 – 1.26 (m, 16H), 0.89 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.7, 145.9, 142.2, 141.2, 129.9, 129.2, 128.9, 36.6, 31.9, 29.61, 29.60, 29.57, 29.51, 29.46, 29.4, 29.3, 22.7, 14.1. HRMS (ESI-TOF) m/z: [M + H]$^{+}$ Calcd for $C_{19}H_{29}N_2$, 285.2325; Found: 285.2336.

2-methyl-3-undecylquinoxaline (7b)



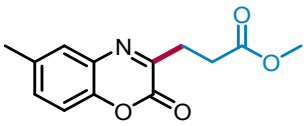
Purification by flash column chromatography (PE:EA, 50:1 v/v), Yellow liquid (26.8 mg, 45% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.03 – 7.98 (m, 2H), 7.67 (dd, *J* = 6.4, 3.4 Hz, 2H), 3.04 – 2.97 (m, 2H), 2.78 (s, 3H), 1.87 – 1.80 (m, 2H), 1.43 – 1.28 (m, 16H), 0.89 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.0, 153.1, 141.2, 140.9, 128.8, 128.7, 128.5, 128.3, 36.0, 31.9, 29.7, 29.64, 29.62, 29.57, 29.5, 29.3, 28.2, 22.8, 22.7, 14.1. HRMS (ESI-TOF) m/z: [M + H]$^{+}$ Calcd for $C_{20}H_{31}N_2$, 299.2487; Found: 299.2487.

6-methyl-3-undecyl-2H-benzo[b][1,4]oxazin-2-one (7c)



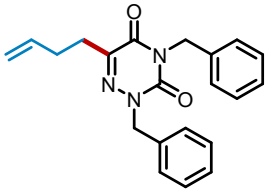
Purification by flash column chromatography (PE:EA, 50:1 v/v). Yellow liquid (34.6 mg, 55% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 2.1 Hz, 1H), 7.29 – 7.26 (m, 1H), 7.18 (d, *J* = 8.4 Hz, 1H), 2.93 – 2.87 (m, 2H), 2.45 (s, 3H), 1.83 – 1.75 (m, 2H), 1.46 – 1.28 (m, 16H), 0.90 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.0, 153.1, 141.2, 140.9, 128.8, 128.7, 128.5, 128.3, 36.0, 31.9, 29.7, 29.64, 29.62, 29.57, 29.5, 29.3, 28.2, 22.8, 22.7, 14.1. HRMS (ESI-TOF) m/z: [M + H]$^{+}$ Calcd for $C_{20}H_{30}NO_2$, 316.2271; Found: 316.2273.

methyl 3-(6-methyl-2-oxo-2H-benzo[b][1,4]oxazin-3-yl)propanoate (7d)[9]



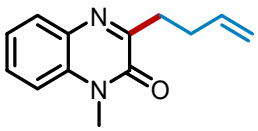
Purification by flash column chromatography (PE:EA, 50:1 v/v). White solid (22.2 mg, 40% yield). mp 380.5 – 382.5 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.49 (m, 1H), 7.30 – 7.26 (m, 1H), 7.17 (d, *J* = 8.4 Hz, 1H), 3.72 (s, 3H), 3.23 (t, *J* = 6.9 Hz, 2H), 2.87 (t, *J* = 6.9 Hz, 2H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.9, 155.6, 153.1, 144.3, 135.3, 131.6, 130.7, 128.8, 115.9, 51.8, 29.5, 28.5, 20.8.

2,4-dibenzyl-6-(but-3-en-1-yl)-1,2,4-triazine-3,5(2H,4H)-dione (9)



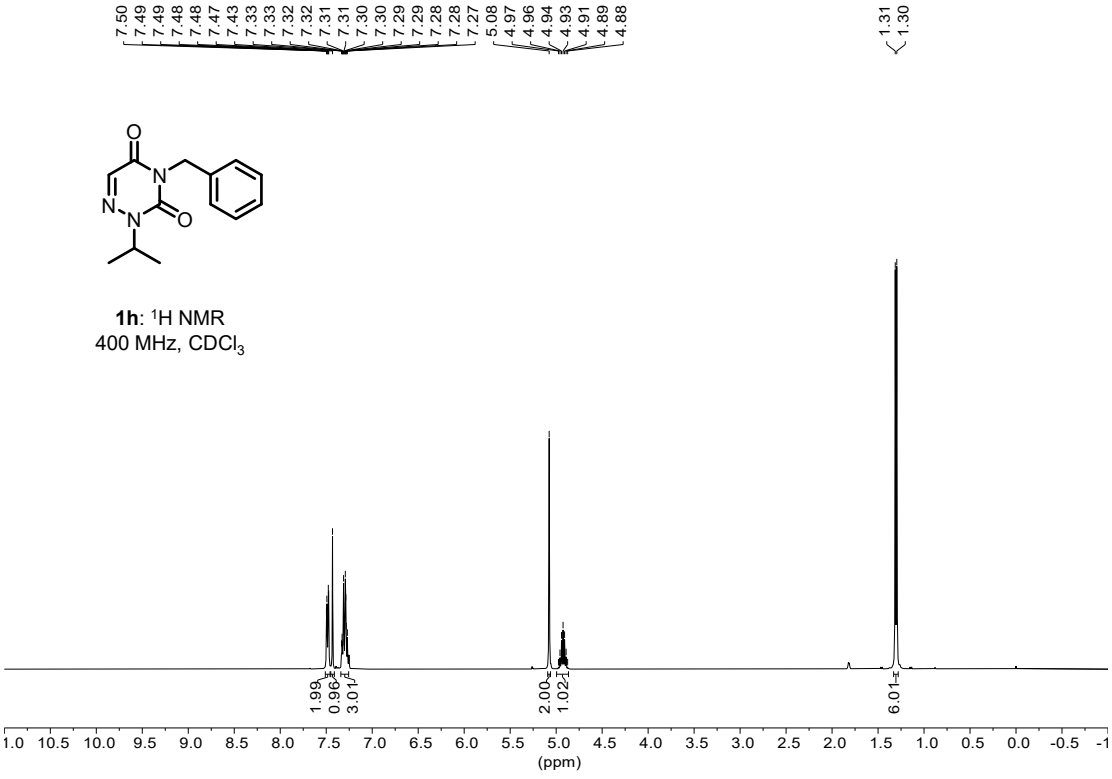
Purification by flash column chromatography (PE:EA, 60:1 v/v). Colorless oil (43.1 mg, 62% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 – 7.47 (m, 2H), 7.45 – 7.30 (m, 8H), 5.86 – 5.76 (m, 1H), 5.11 (d, *J* = 2.4 Hz, 4H), 5.09 – 4.97 (m, 2H), 2.75 (dd, *J* = 8.4, 6.7 Hz, 2H), 2.51 – 2.31 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.0, 149.0, 144.9, 137.0, 135.8, 135.7, 129.4, 128.7, 128.6, 128.2, 128.0, 115.7, 55.2, 44.2, 30.1, 29.6. HRMS (ESI-TOF) m/z: [M + Na]$^{+}$ Calcd for $C_{21}H_{21}N_3O_2Na$ 370.1526; Found: 370.1528.

3-(but-3-en-1-yl)-1-methylquinoxalin-2(1H)-one (10)[10]

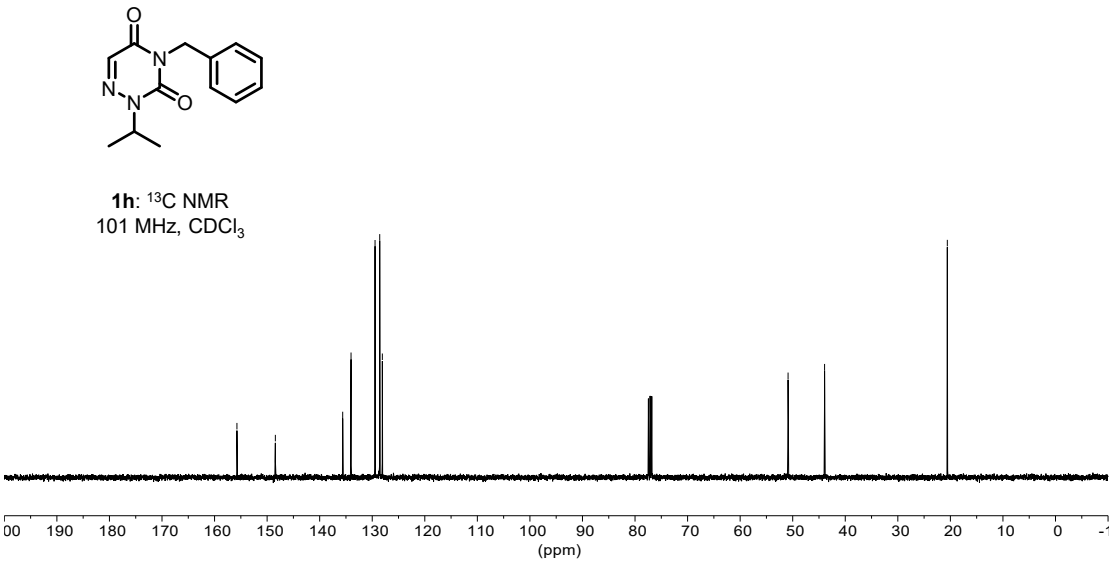


Purification by flash column chromatography (PE:EA, 30:1 v/v). White solid (26.1 mg, 61% yield), mp 45.5 – 48.5 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.56-7.52 (m, 1H), 7.39 – 7.29 (m, 2H), 6.03 – 5.92 (m, 1H), 5.13 (dd, *J* = 17.1, 1.7 Hz, 1H), 5.01 (dt, *J* = 10.4, 1.6 Hz, 1H), 3.71 (s, 3H), 3.11 – 3.02 (m, 2H), 2.62 – 2.57 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.3, 154.9, 137.8, 133.1, 132.7, 129.7, 129.6, 123.6, 115.2, 113.6, 33.5, 30.6, 29.0.

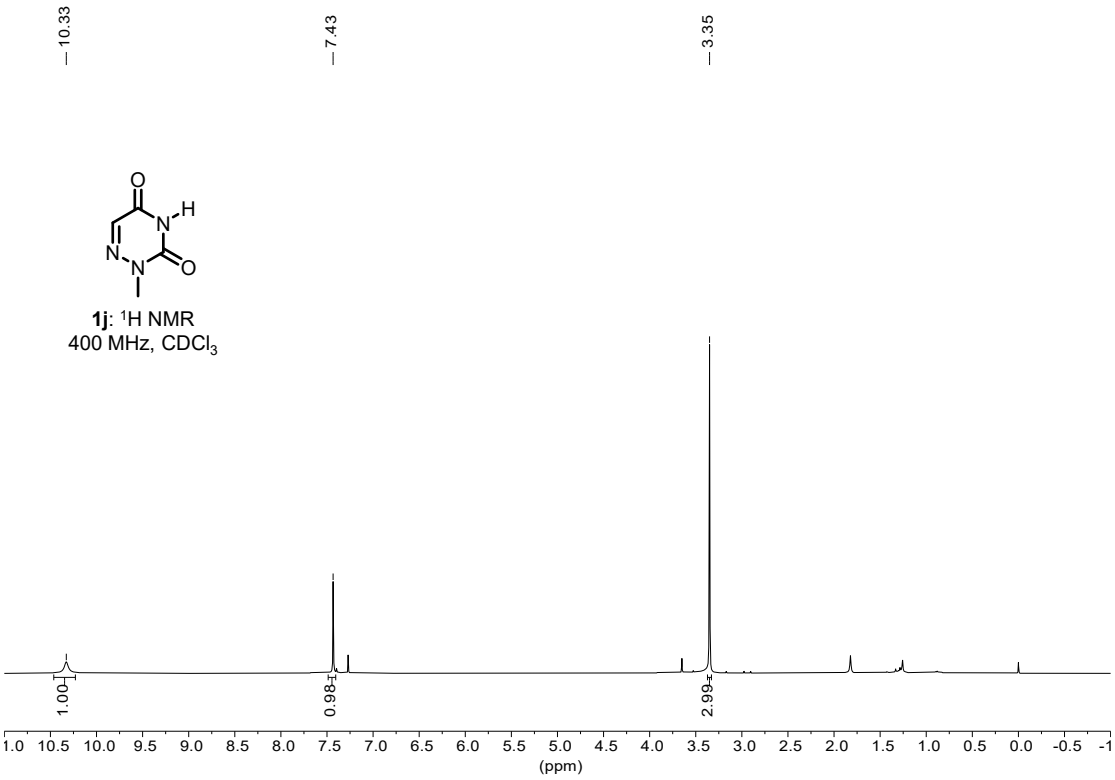
4. NMR Copies of Products



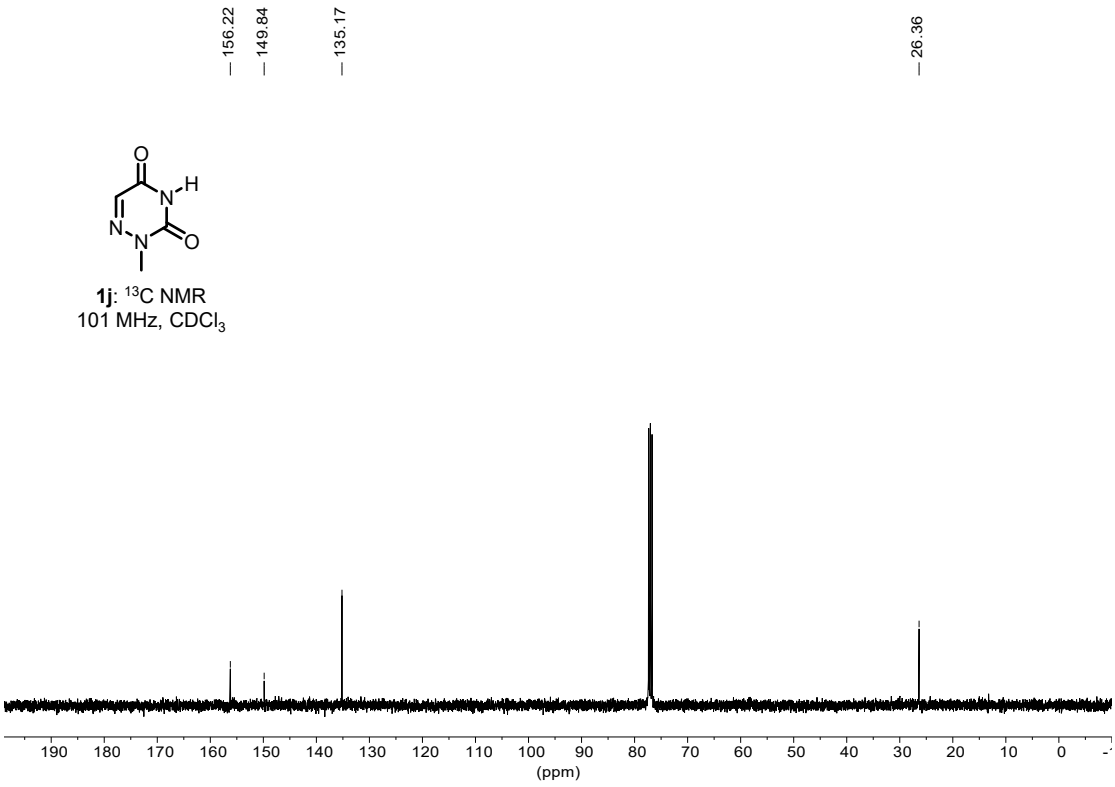
1h: ^{1}H NMR
400 MHz, $CDCl_3$



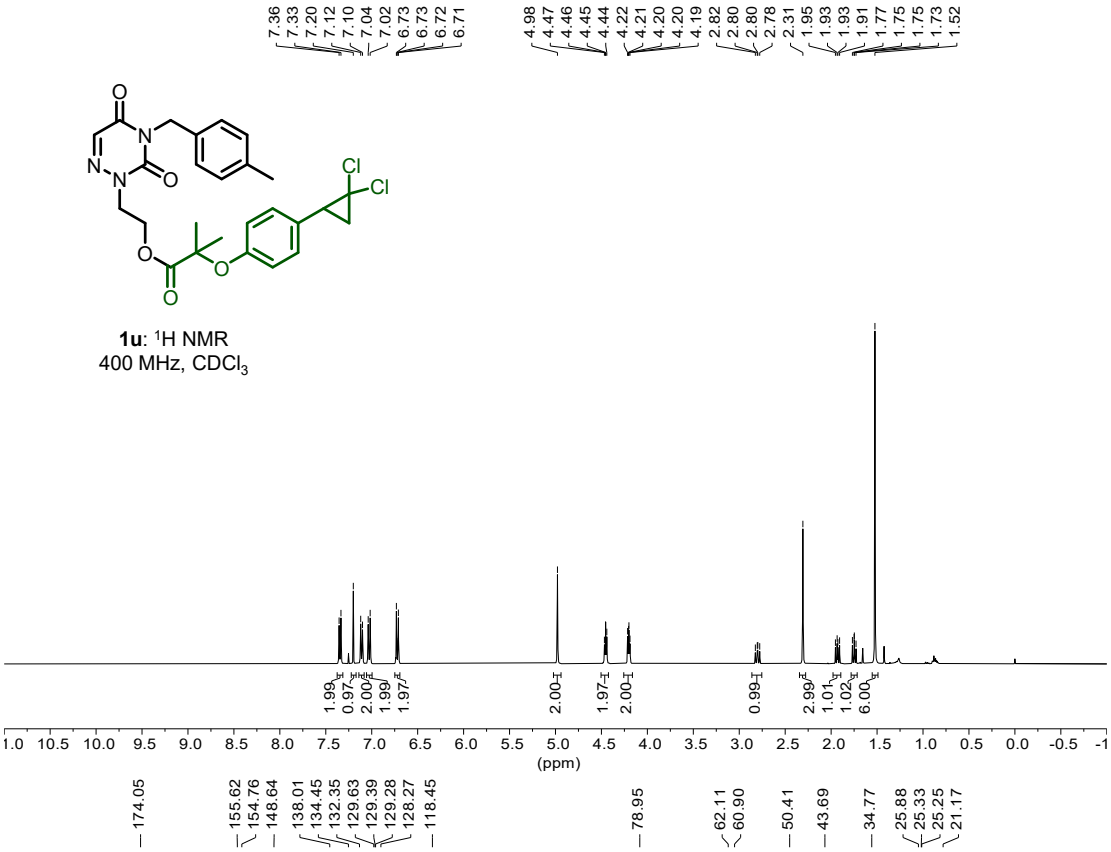
1h: ^{13}C NMR
101 MHz, $CDCl_3$



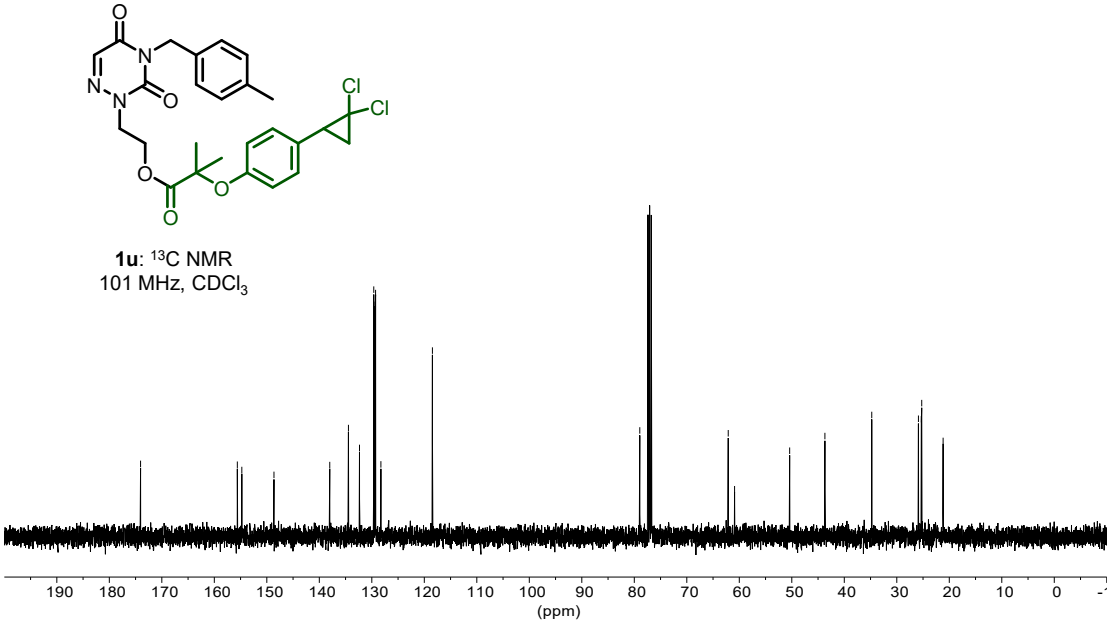
1j: ^{1}H NMR
400 MHz, $CDCl_3$



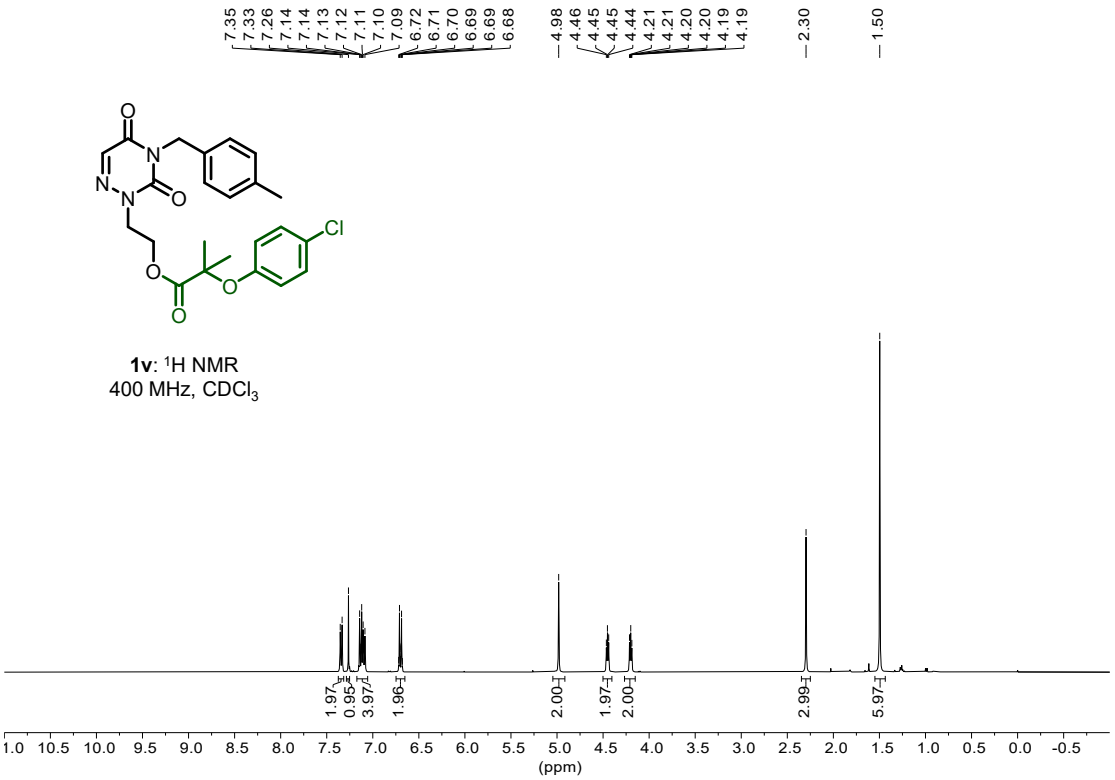
1j: ^{13}C NMR
101 MHz, $CDCl_3$



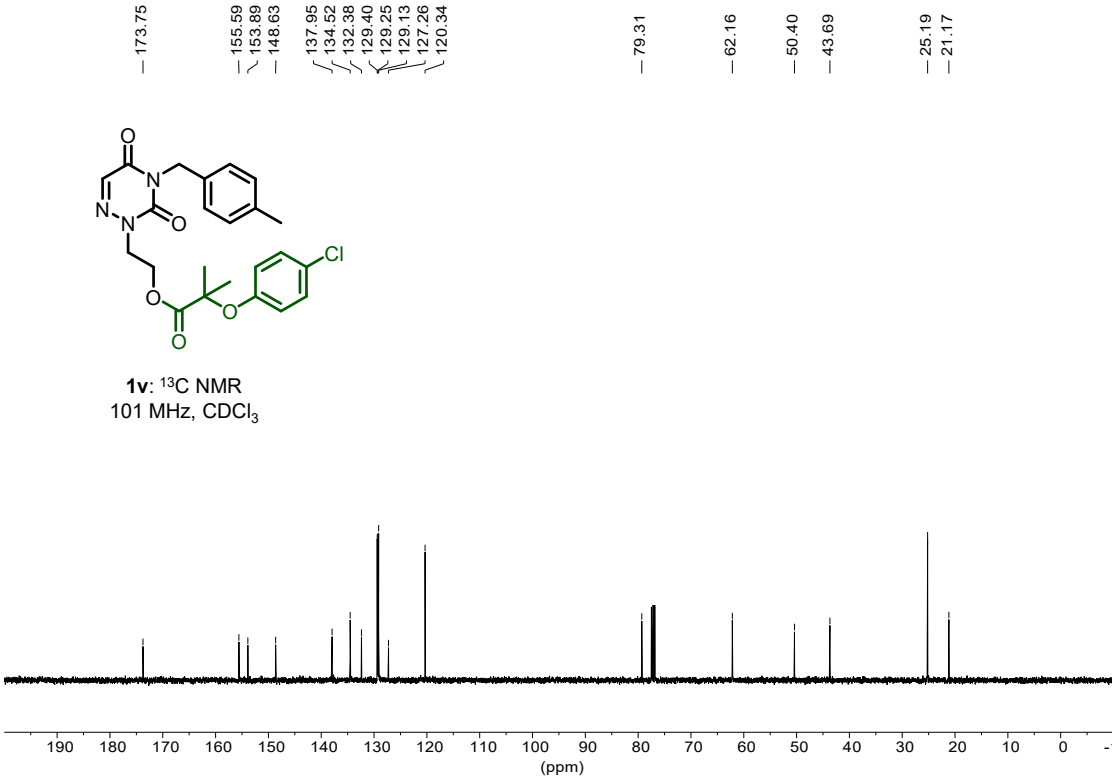
1u: ^{1}H NMR
400 MHz, $CDCl_3$



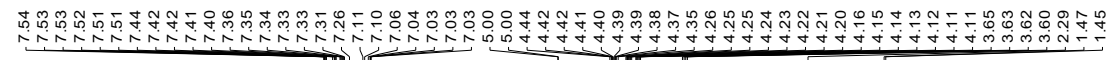
1u: ^{13}C NMR
101 MHz, $CDCl_3$

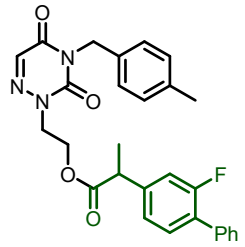


1v: ^{1}H NMR
400 MHz, $CDCl_3$

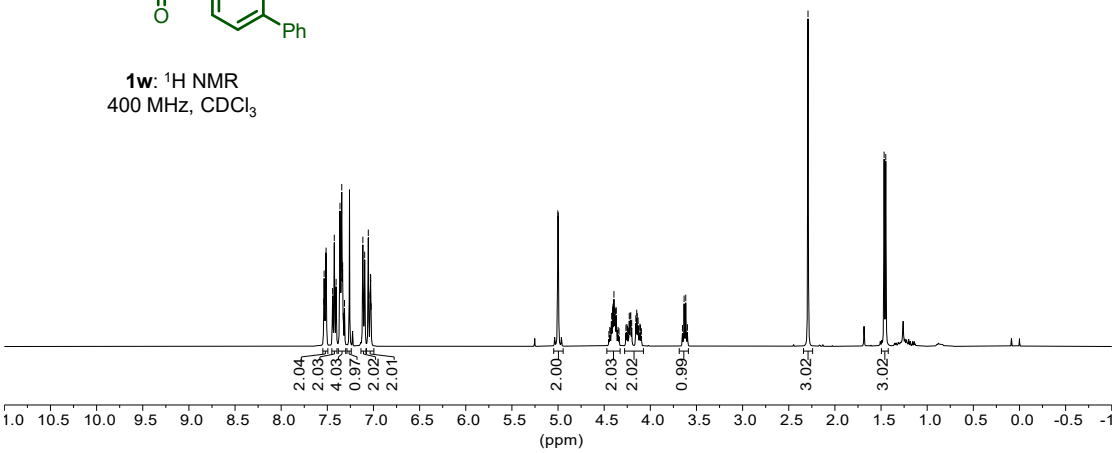


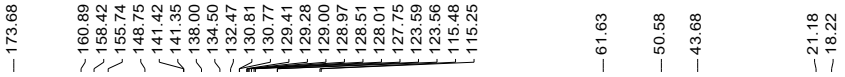
1v: ^{13}C NMR
101 MHz, $CDCl_3$

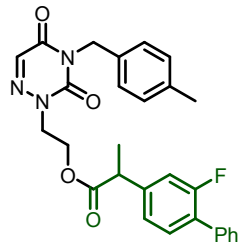




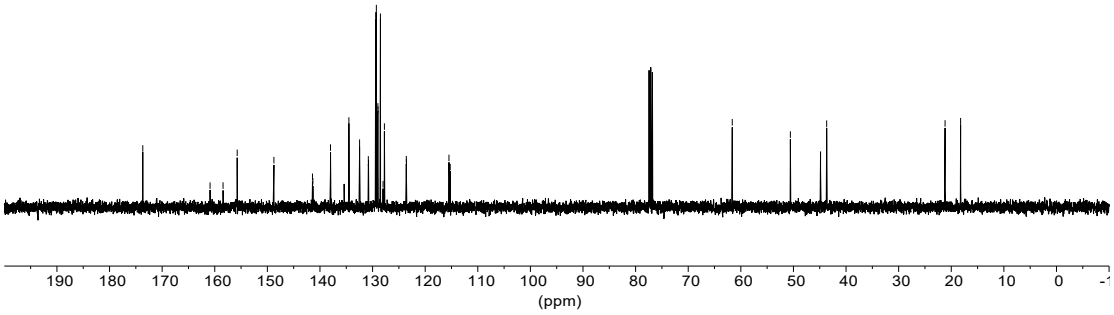
1w: ^{1}H NMR
400 MHz, $CDCl_3$

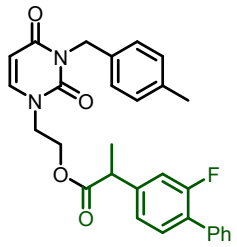




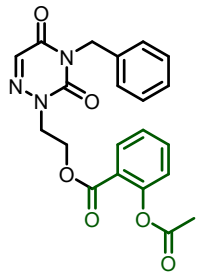


1w: ^{13}C NMR
101 MHz, $CDCl_3$

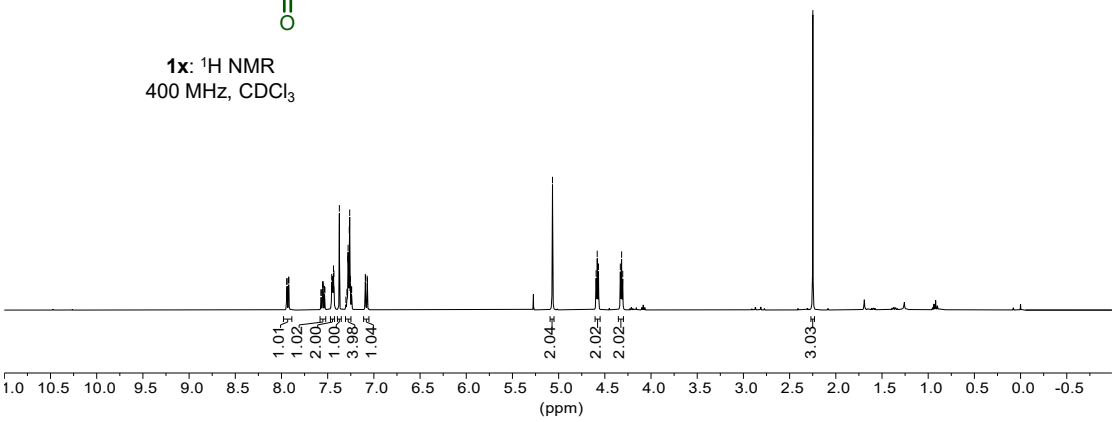


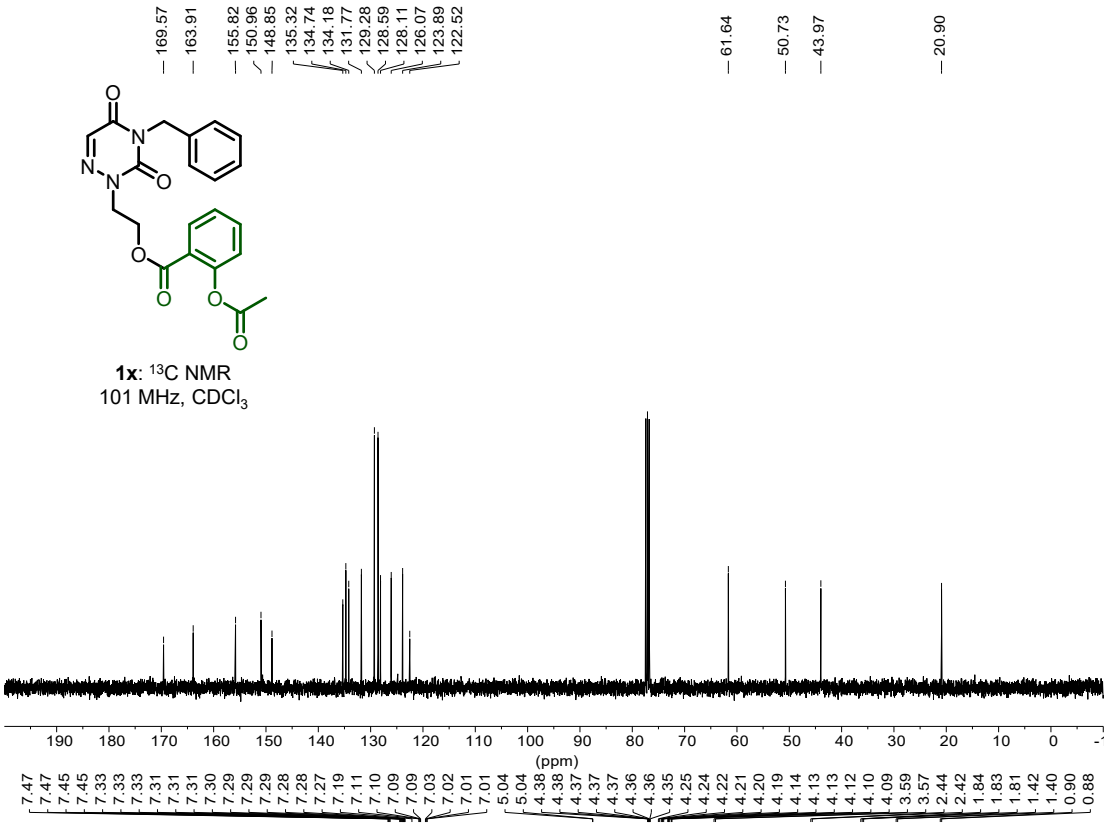


1w: ^{19}F NMR
376 MHz, $CDCl_3$

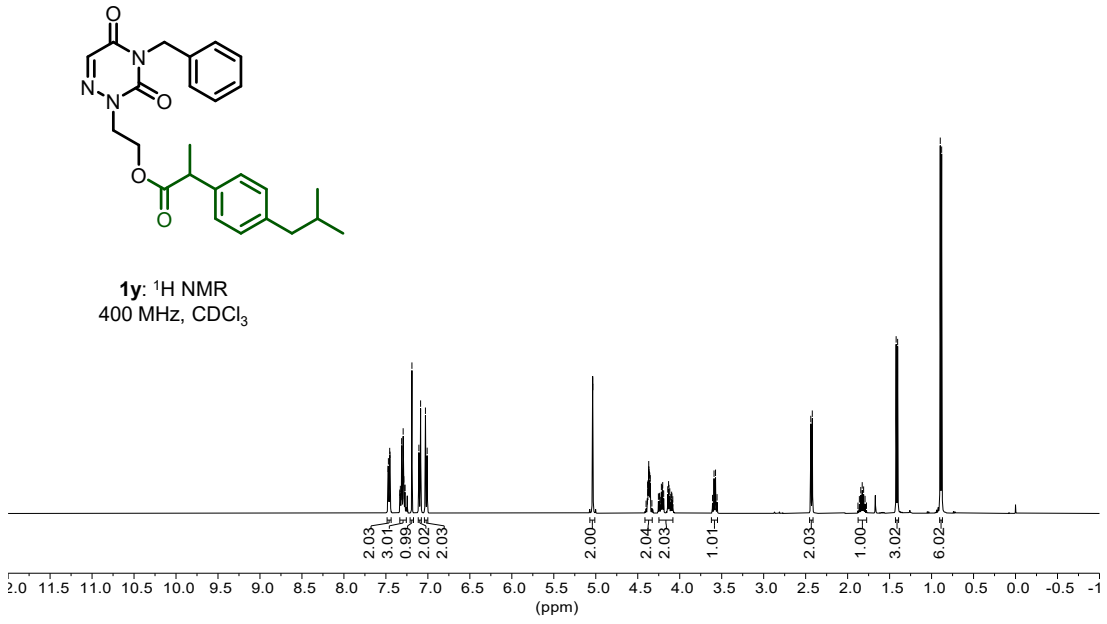


1x: ^{1}H NMR
400 MHz, $CDCl_3$

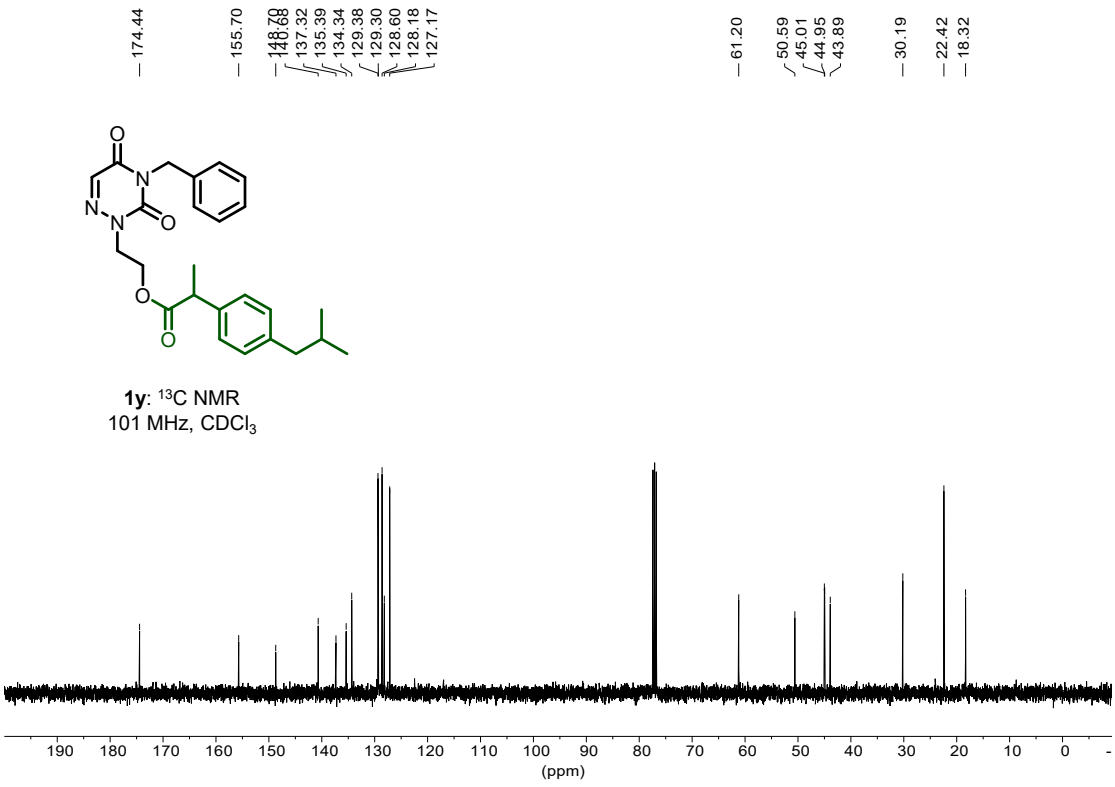




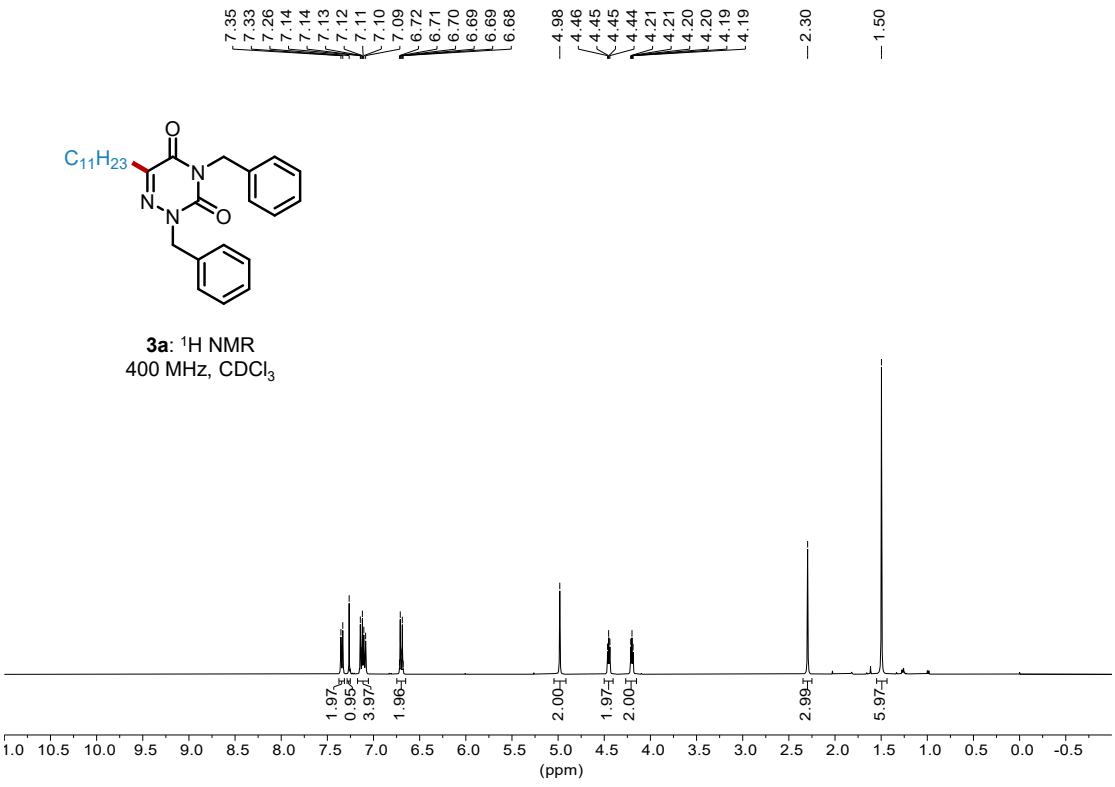
1x: ^{13}C NMR
101 MHz, $CDCl_3$



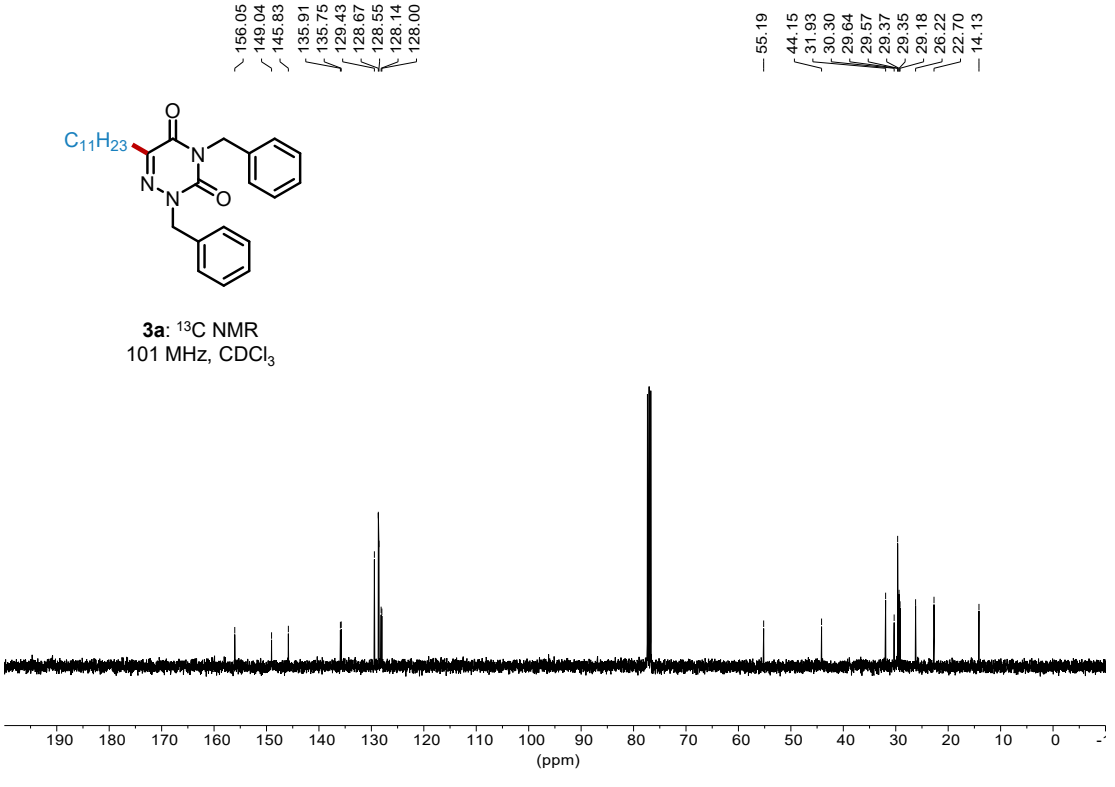
1y: ^{1}H NMR
400 MHz, $CDCl_3$



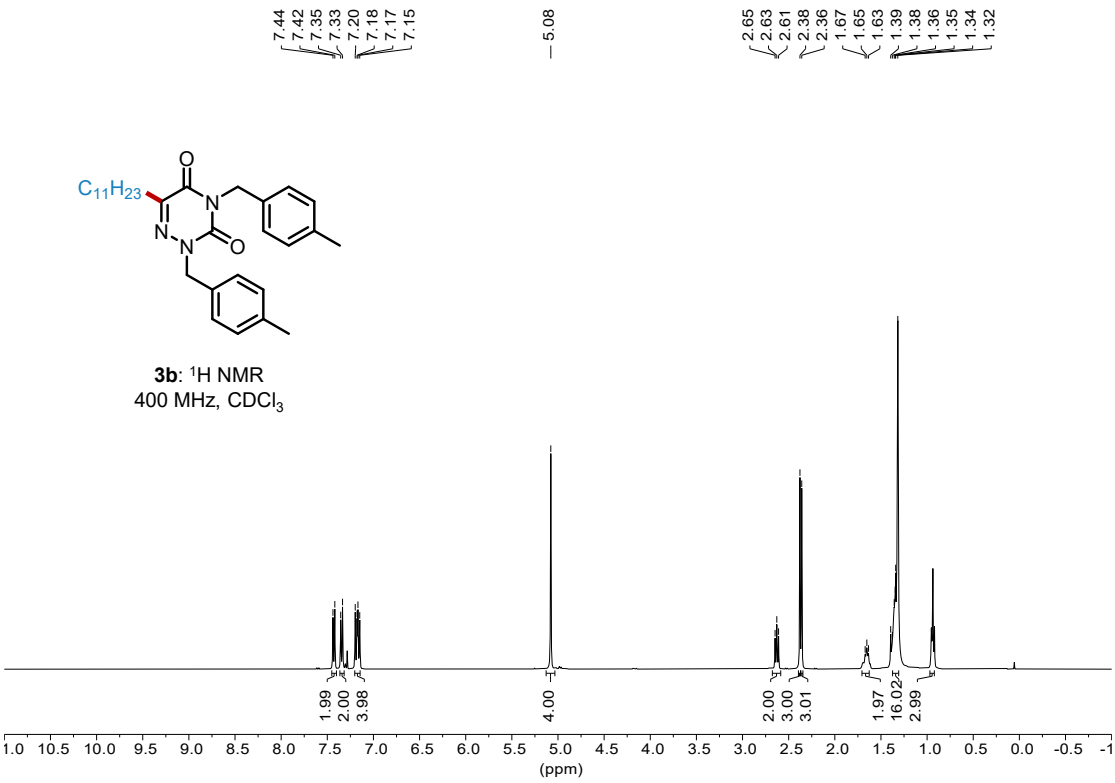
1y: ^{13}C NMR
101 MHz, $CDCl_3$



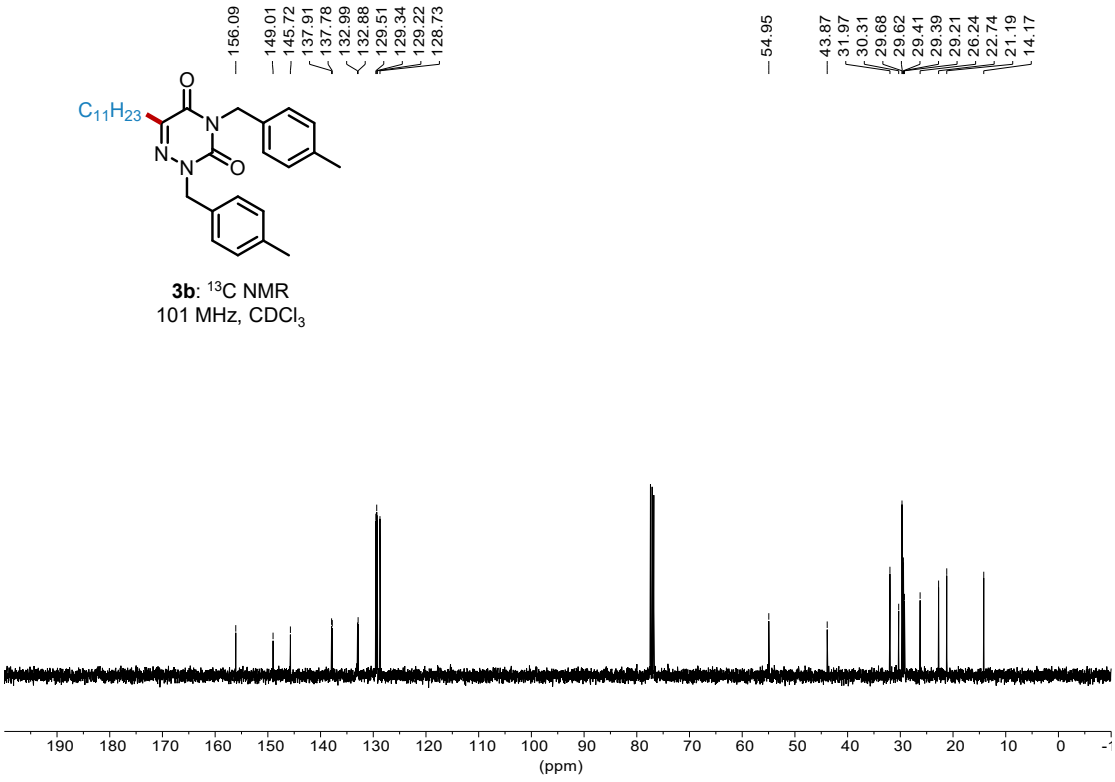
3a: ^{1}H NMR
400 MHz, $CDCl_3$



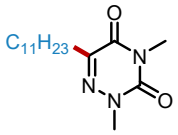
3a: ^{13}C NMR
101 MHz, $CDCl_3$



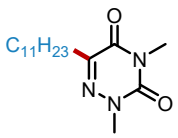
3b: ^{1}H NMR
400 MHz, $CDCl_3$



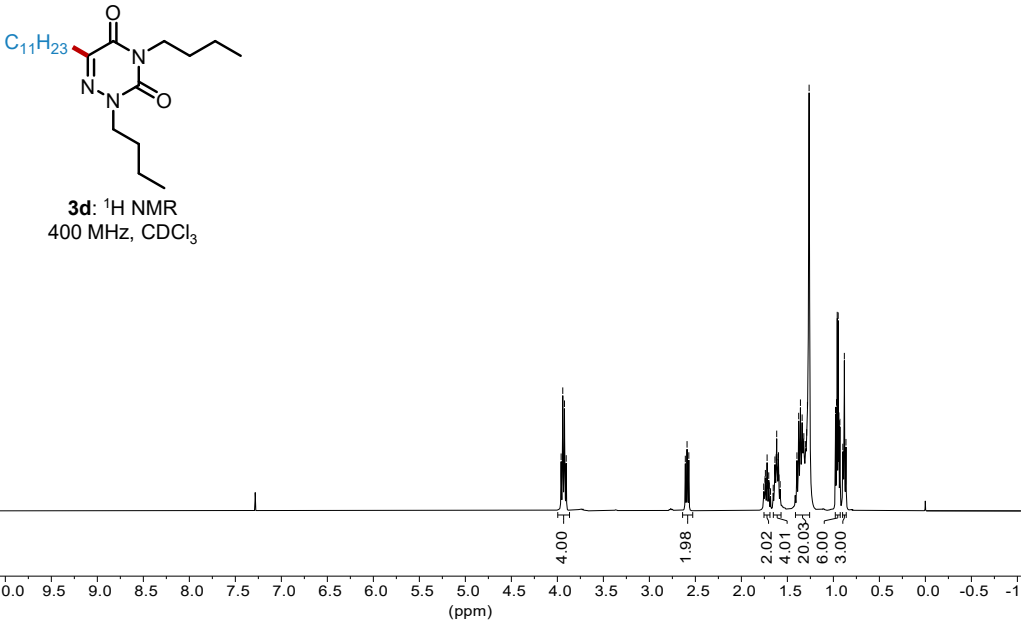
3b: ^{13}C NMR
101 MHz, $CDCl_3$



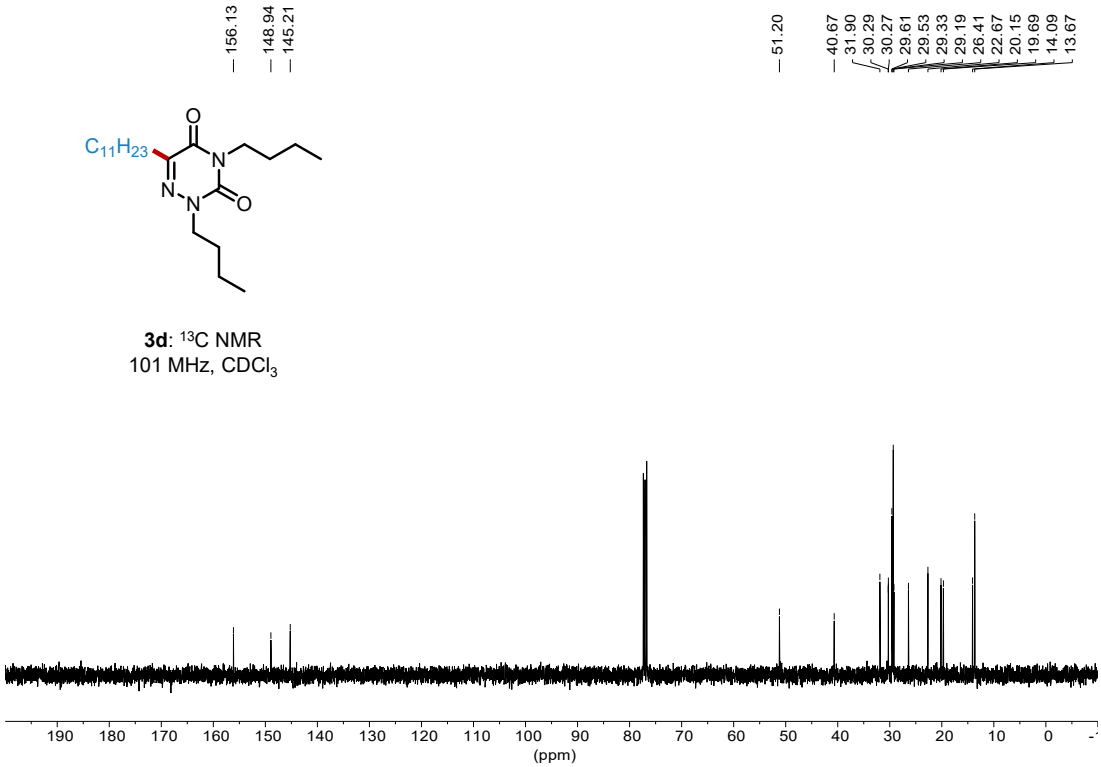
3c: ^{1}H NMR
400 MHz, $CDCl_3$



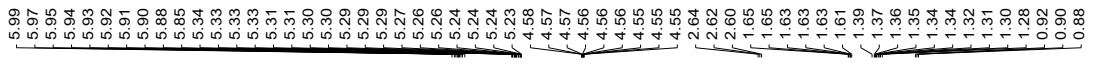
3c: ^{13}C NMR
101 MHz, $CDCl_3$

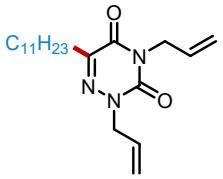


3d: ^{1}H NMR
400 MHz, $CDCl_3$

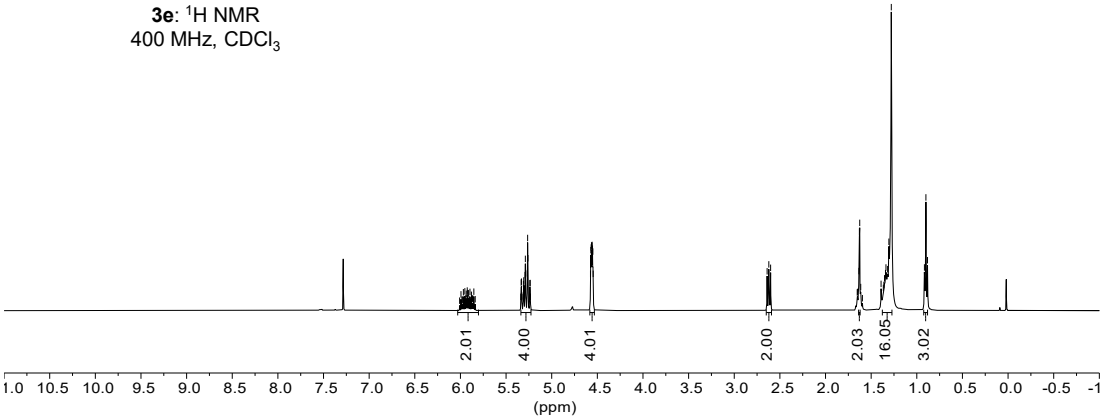


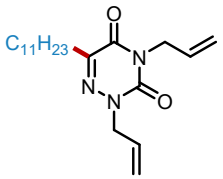
3d: ^{13}C NMR
101 MHz, $CDCl_3$



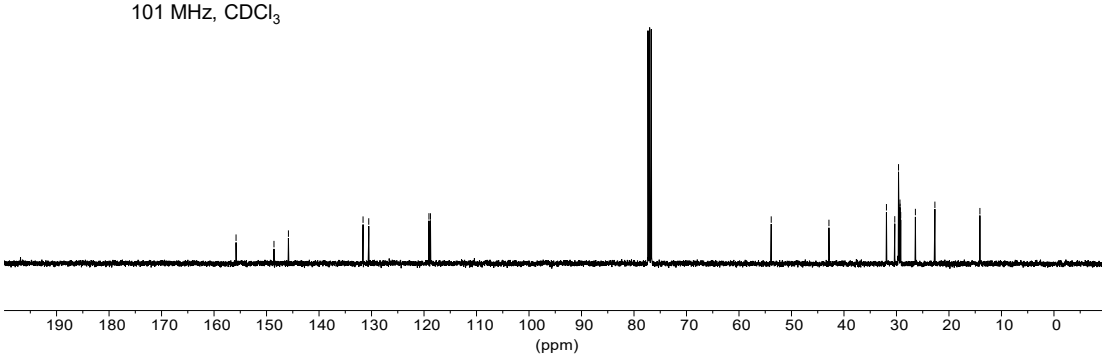


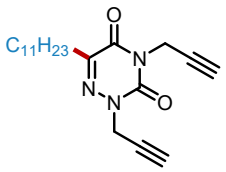
3e: ^{1}H NMR
400 MHz, $CDCl_3$



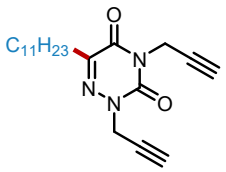


3e: ^{13}C NMR
101 MHz, $CDCl_3$

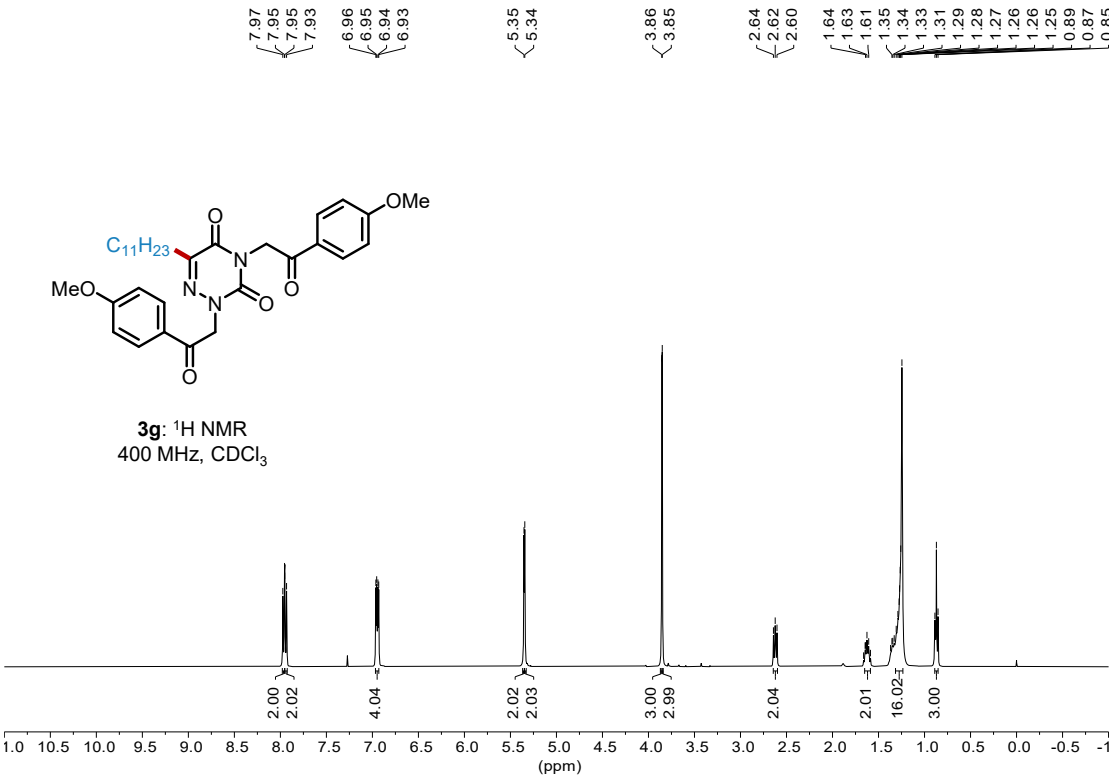




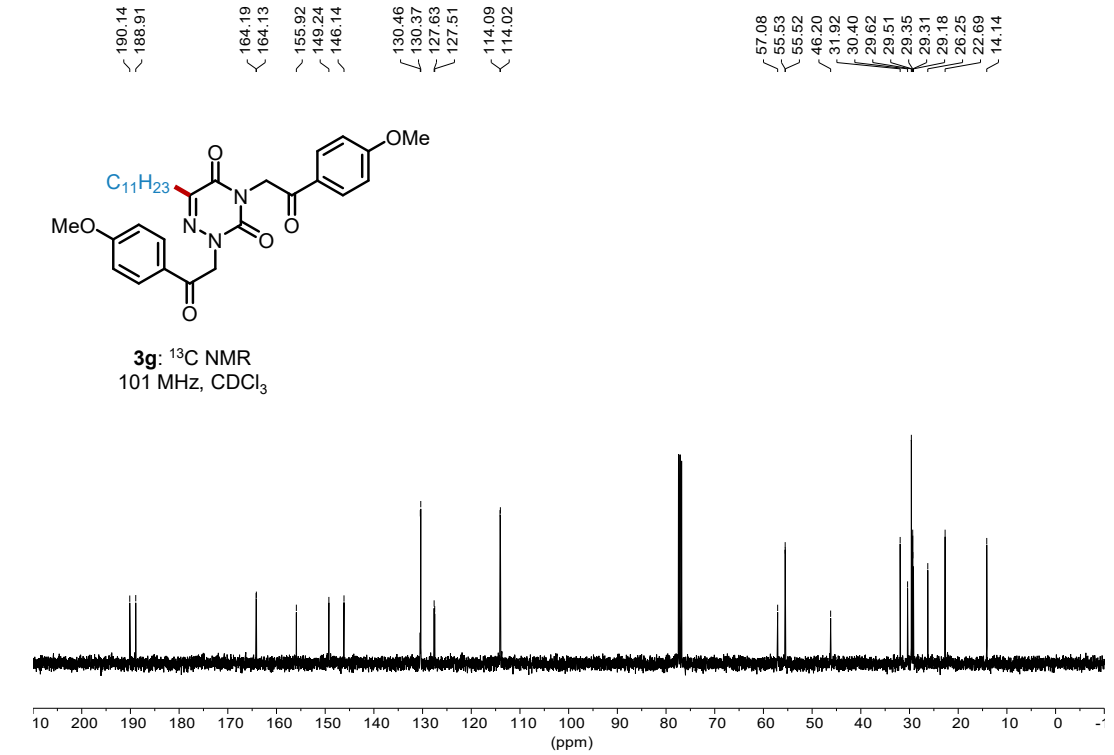
3f: ^{1}H NMR
400 MHz, $CDCl_3$



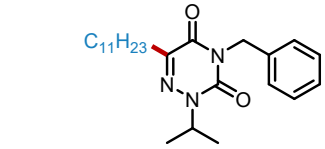
3f: ^{13}C NMR
101 MHz, $CDCl_3$



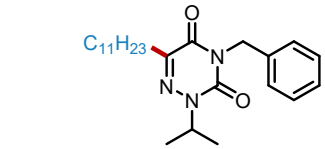
3g: ^{1}H NMR
400 MHz, $CDCl_3$



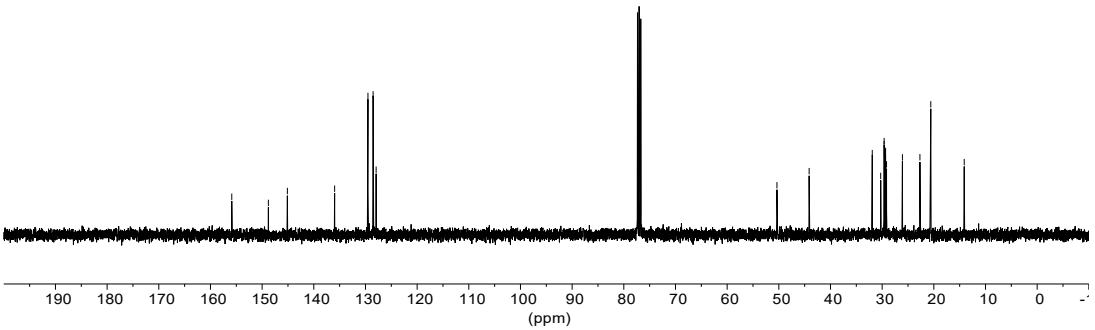
3g: ^{13}C NMR
101 MHz, $CDCl_3$

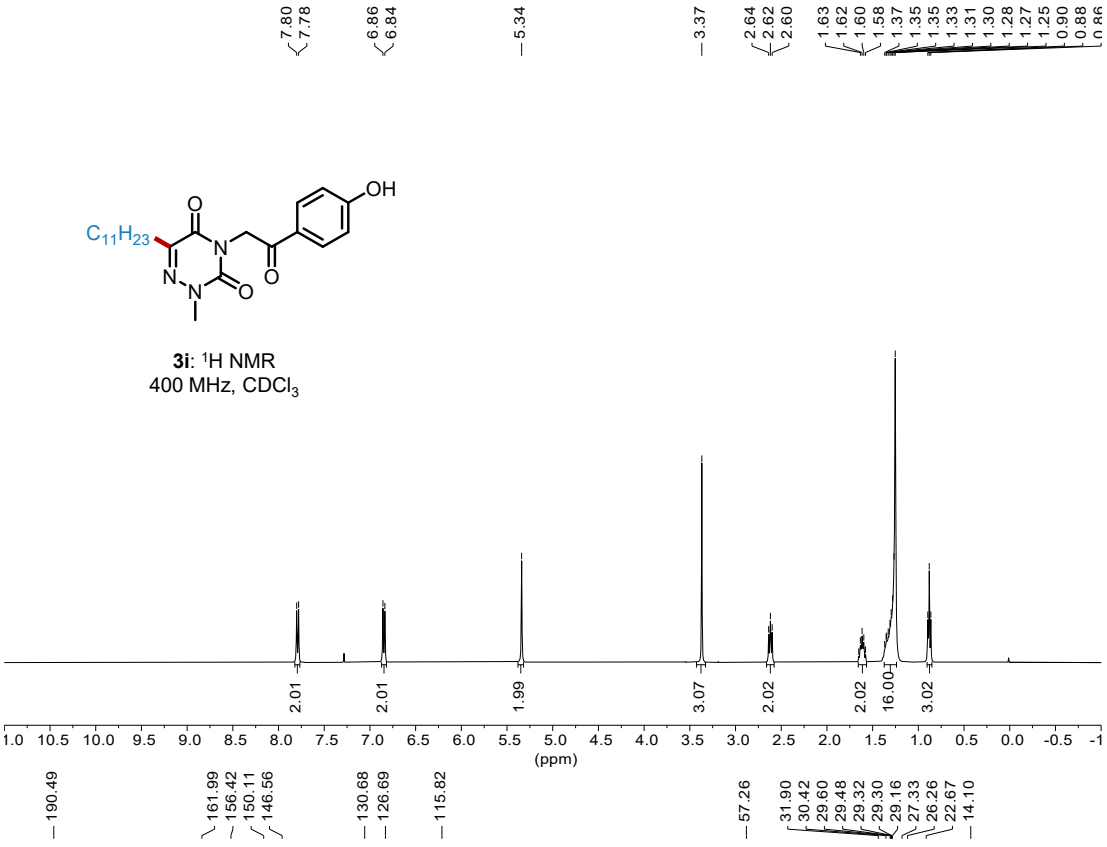


3h： ^{1}H NMR
400 MHz, $CDCl_3$

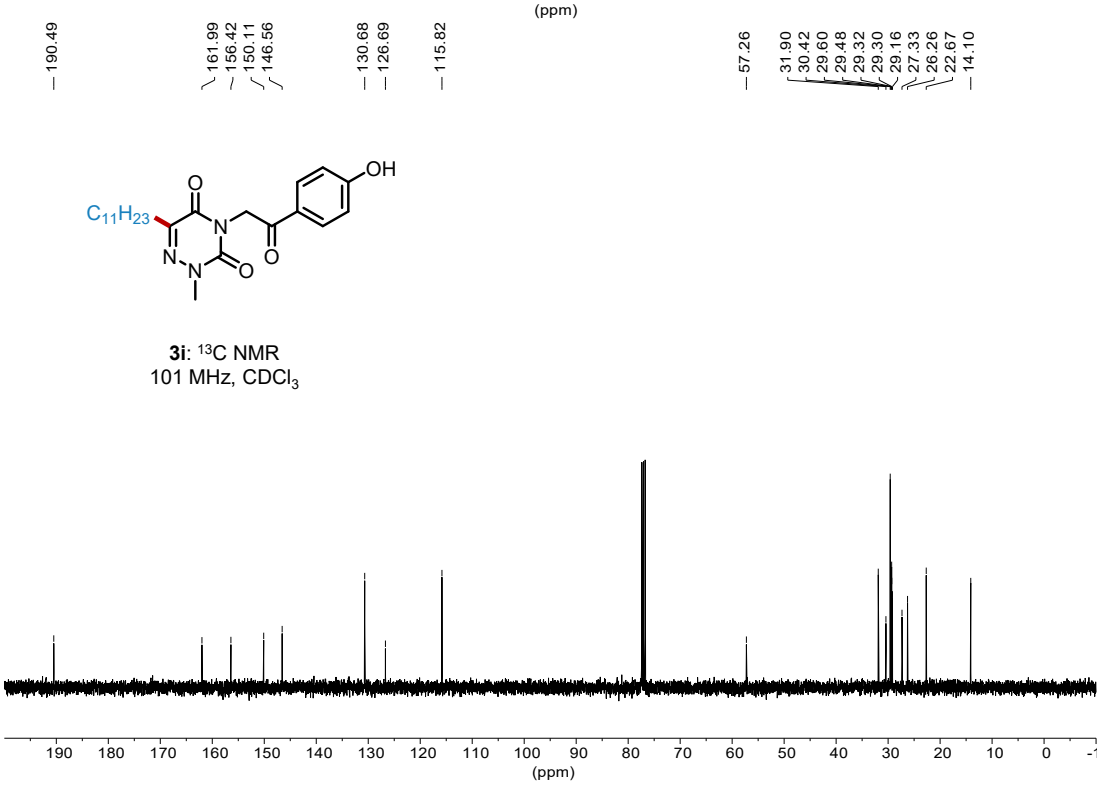


3h: ^{13}C NMR
101 MHz, $CDCl_3$

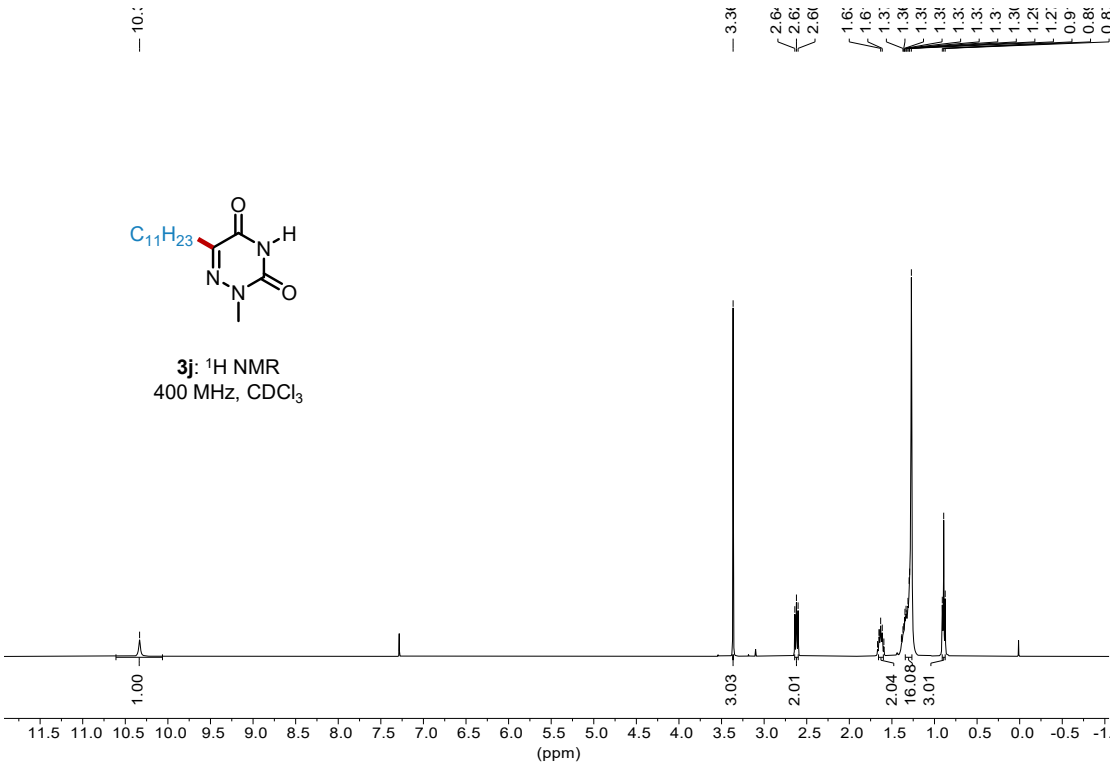




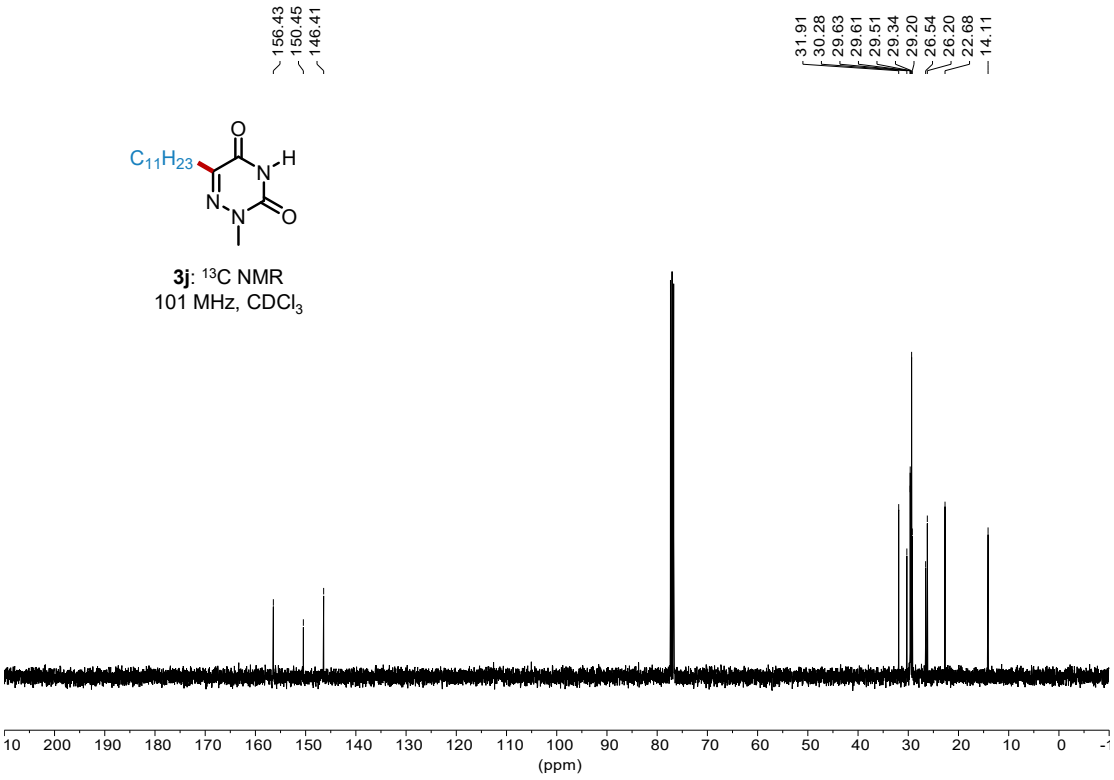
3i: ^{1}H NMR
400 MHz, $CDCl_3$



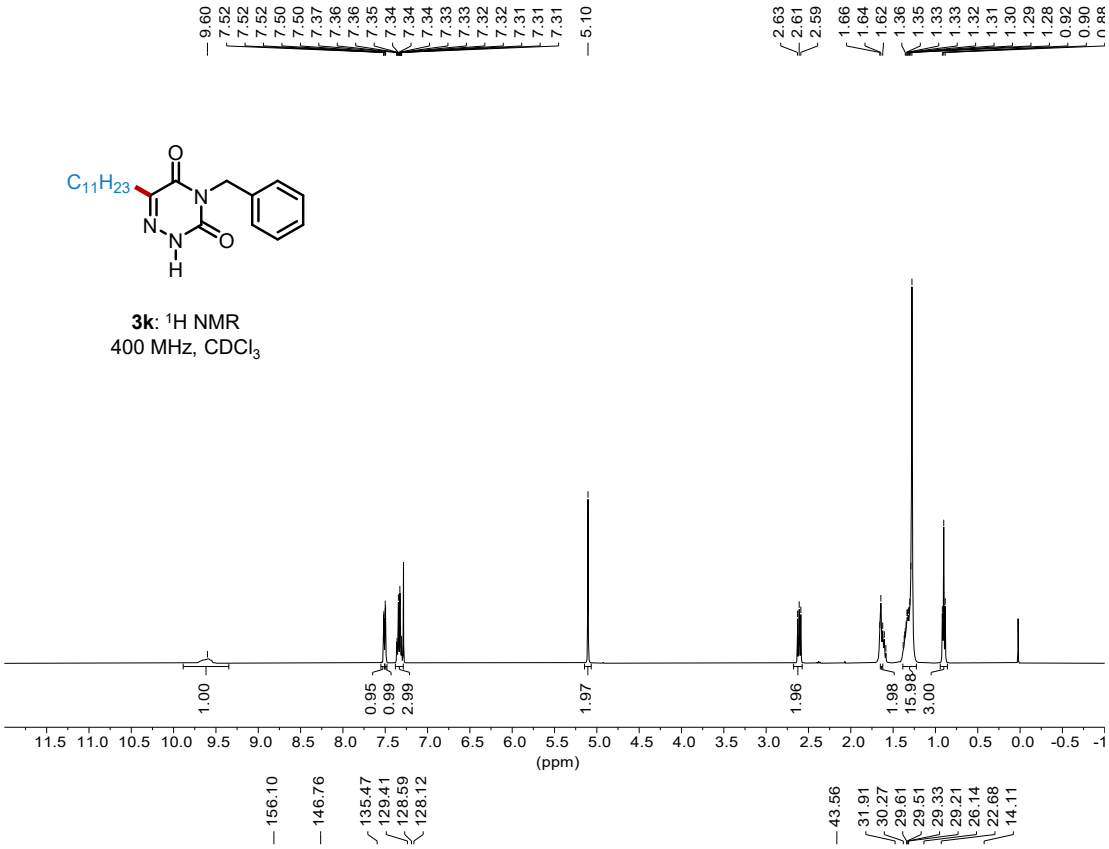
3i: ^{13}C NMR
101 MHz, $CDCl_3$



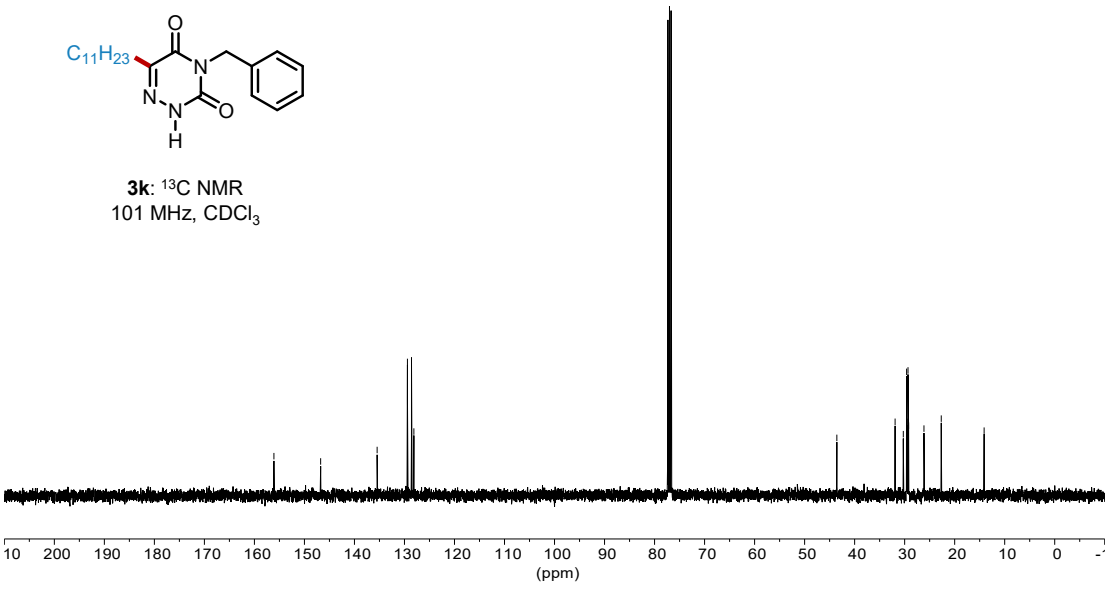
3j: ^{1}H NMR
400 MHz, $CDCl_3$



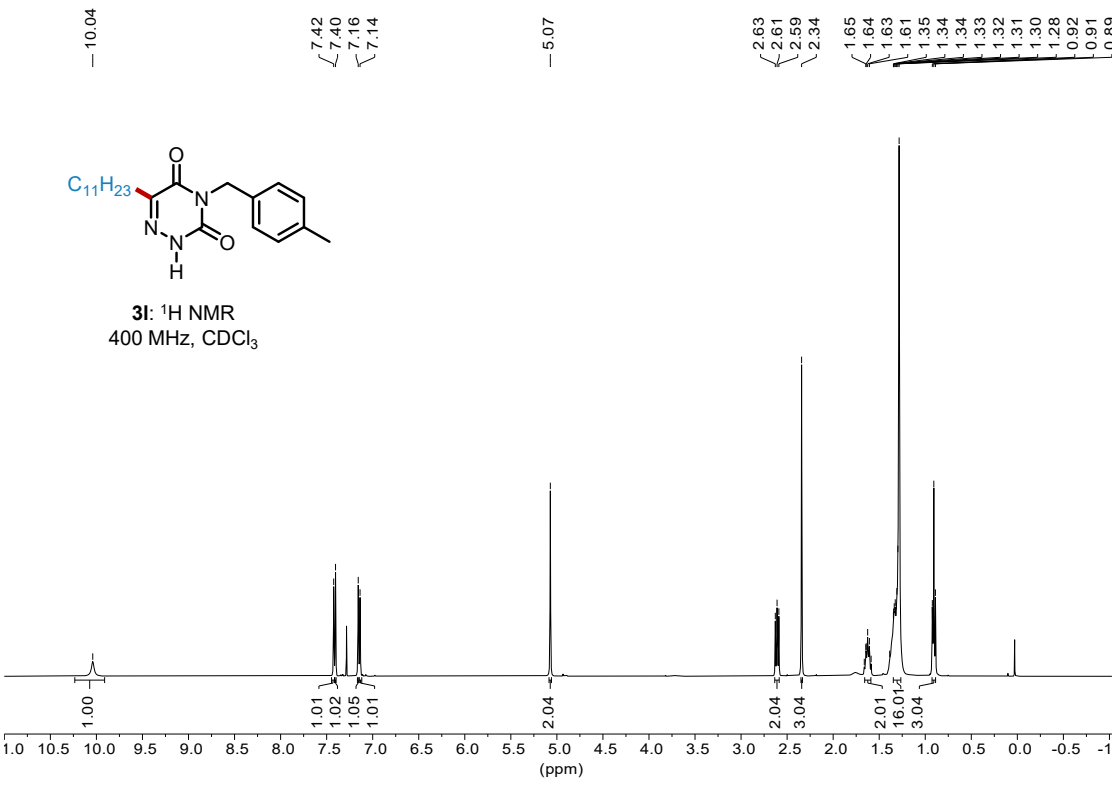
3j: ^{13}C NMR
101 MHz, $CDCl_3$



3k: ^{1}H NMR
400 MHz, $CDCl_3$



3k: ^{13}C NMR
101 MHz, $CDCl_3$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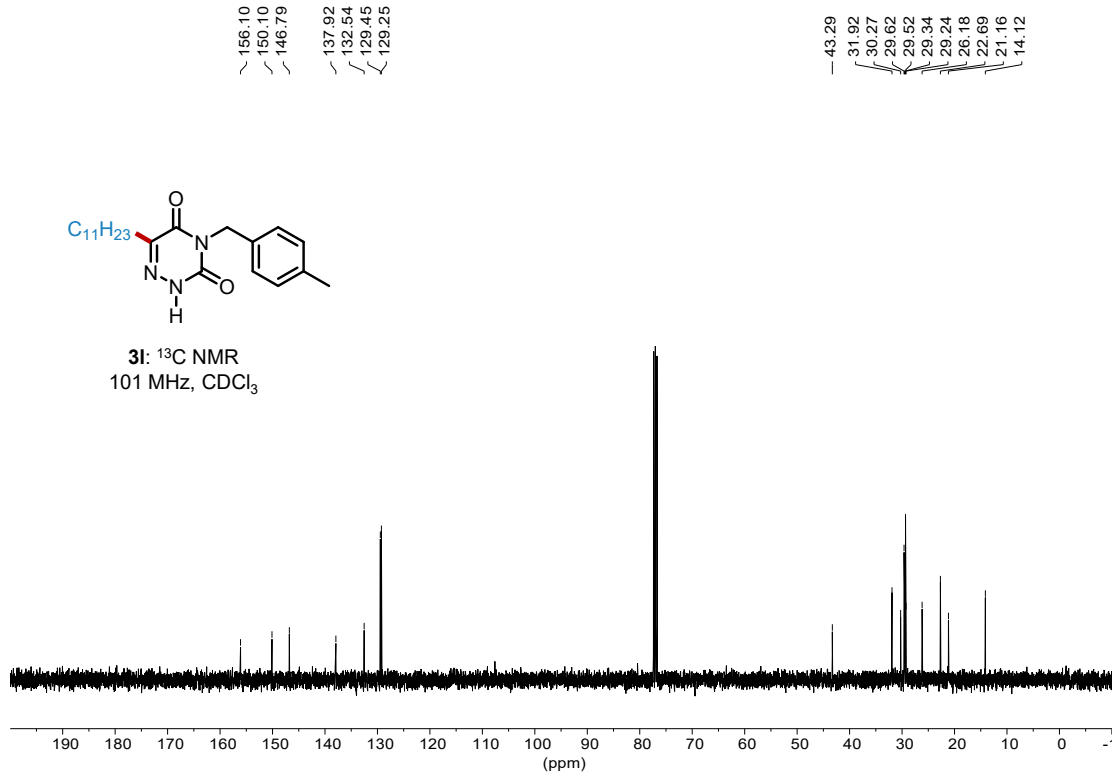
7.42
7.40
7.16
7.14
5.07
2.63
2.61
2.59
2.34
1.65
1.64
1.63
1.61
1.35
1.34
1.34
1.33
1.32
1.31
1.30
1.28
0.92
0.91
C11H23
3I: 1H NMR
400 MHz, CDCl3
1.00
1.01
1.02
1.05
1.01
2.04
2.04
3.04
2.01
16.01
3.04
(ppm)


156.10
150.10
146.79
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132.54
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43.29
31.92
30.27
29.62
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26.18
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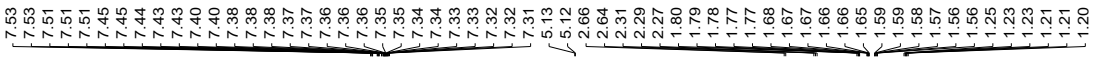
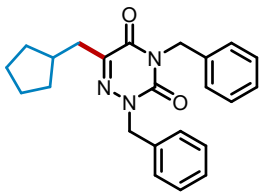
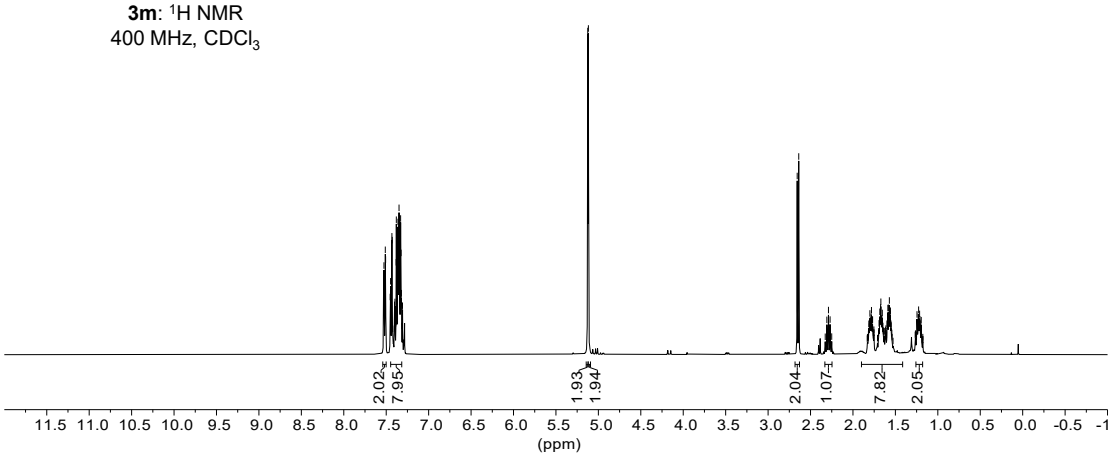
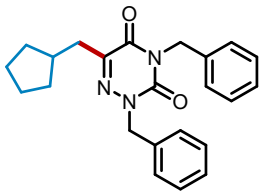
21.16
14.12
C11H23
3I: 13C NMR
101 MHz, CDCl3
(ppm)



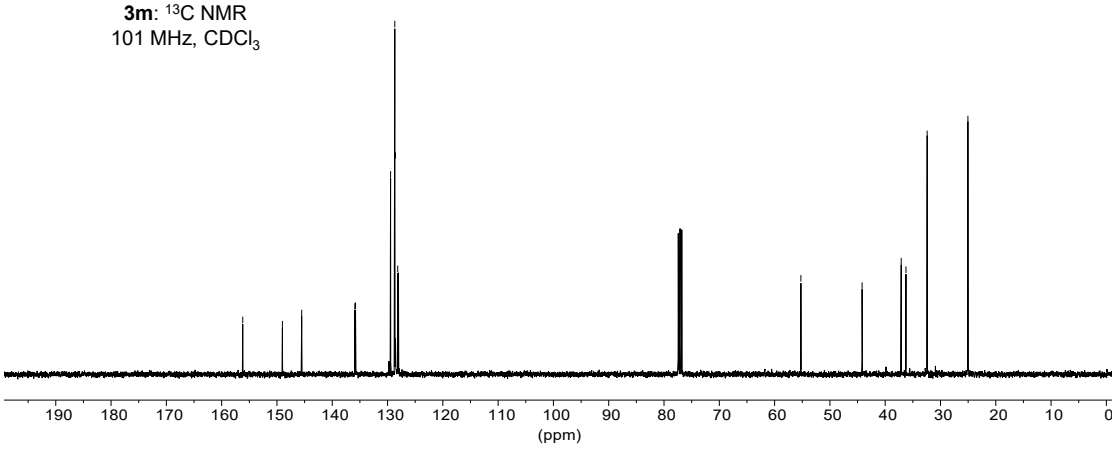


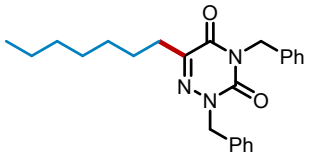
3m: ^{1}H NMR
400 MHz, $CDCl_3$



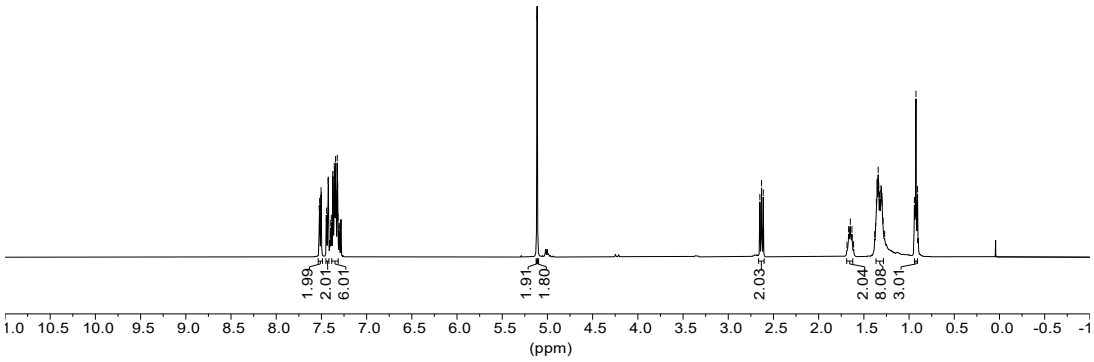


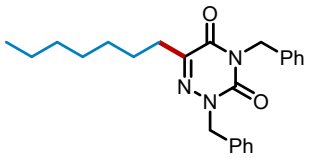
3m: ^{13}C NMR
101 MHz, $CDCl_3$



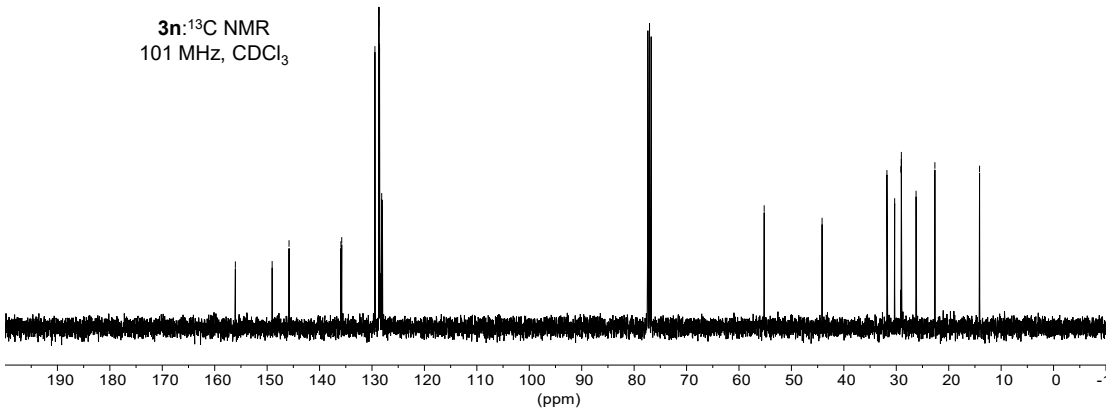


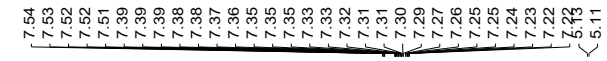
3n: ^{1}H NMR
400 MHz, $CDCl_3$

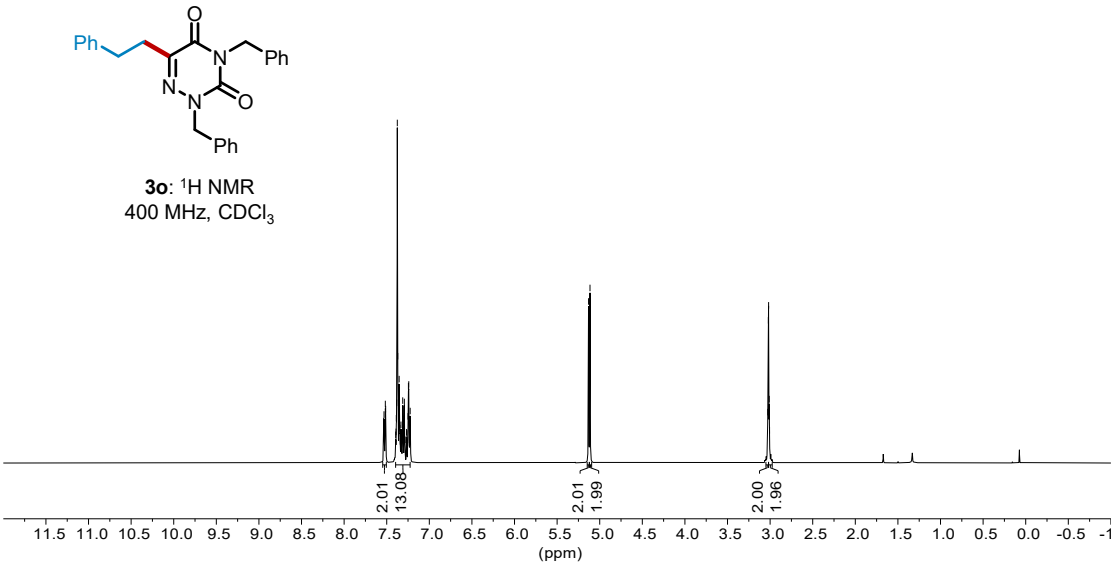




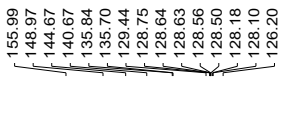
3n:^{13}C NMR
101 MHz, $CDCl_3$

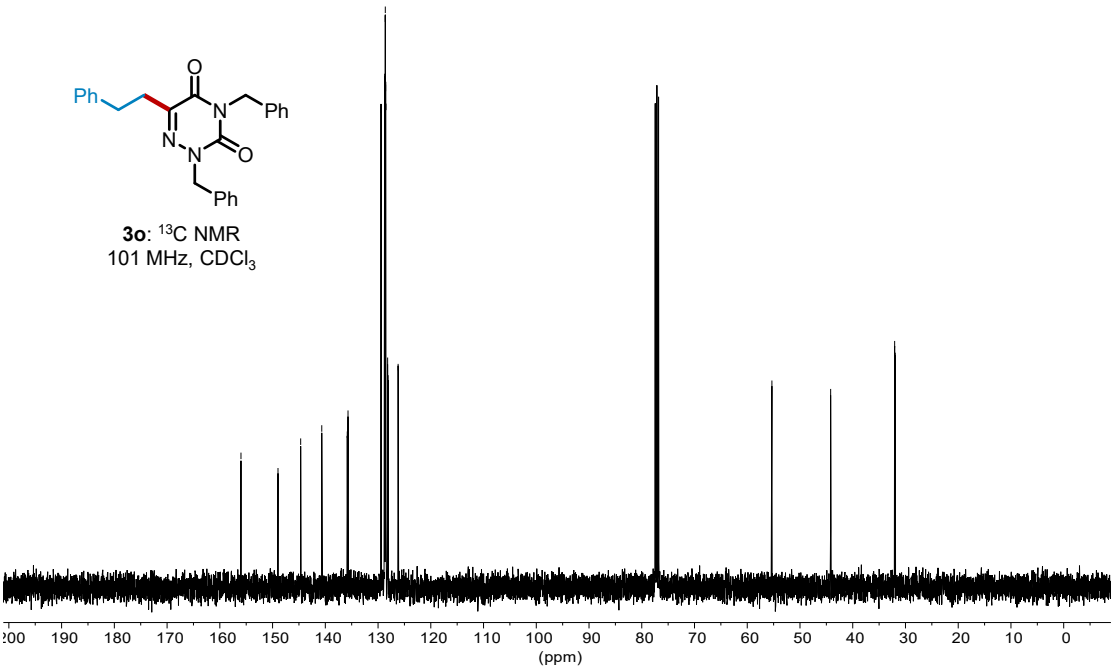




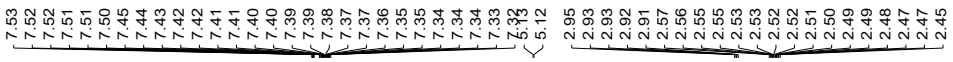


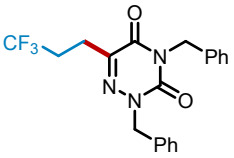
3o: ^{1}H NMR
400 MHz, $CDCl_3$



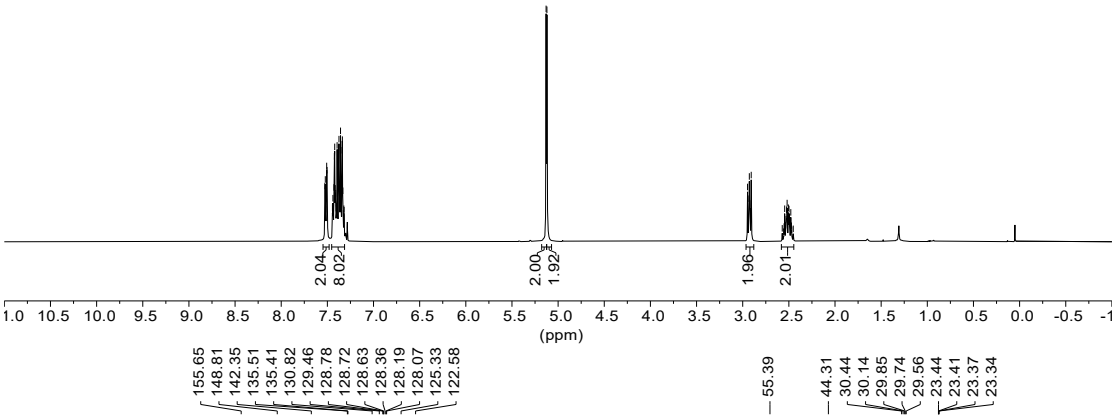


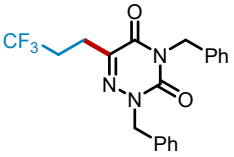
3o: ^{13}C NMR
101 MHz, $CDCl_3$



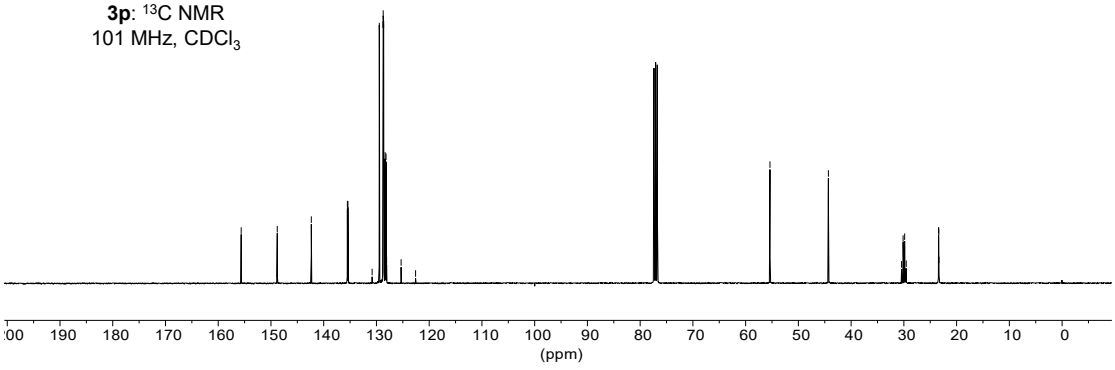


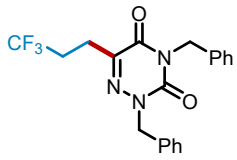
3p: ^{1}H NMR
400 MHz, $CDCl_3$



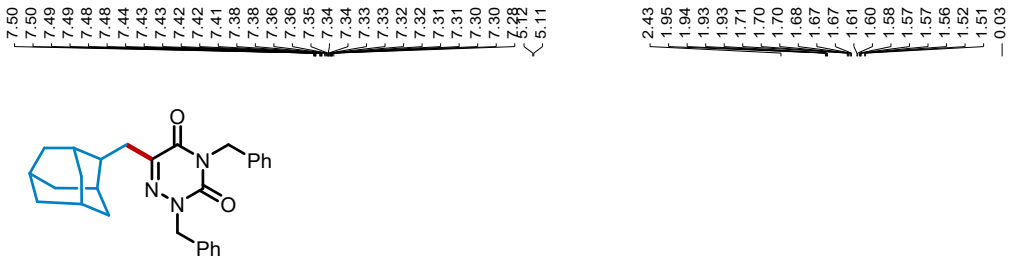


3p: ^{13}C NMR
101 MHz, $CDCl_3$

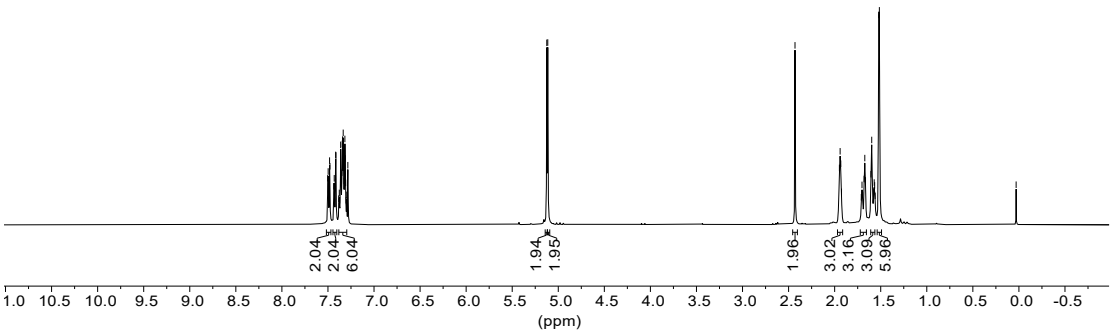


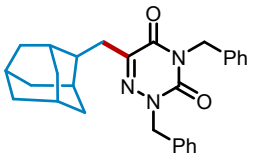


3p: ^{19}F NMR
376 MHz, $CDCl_3$

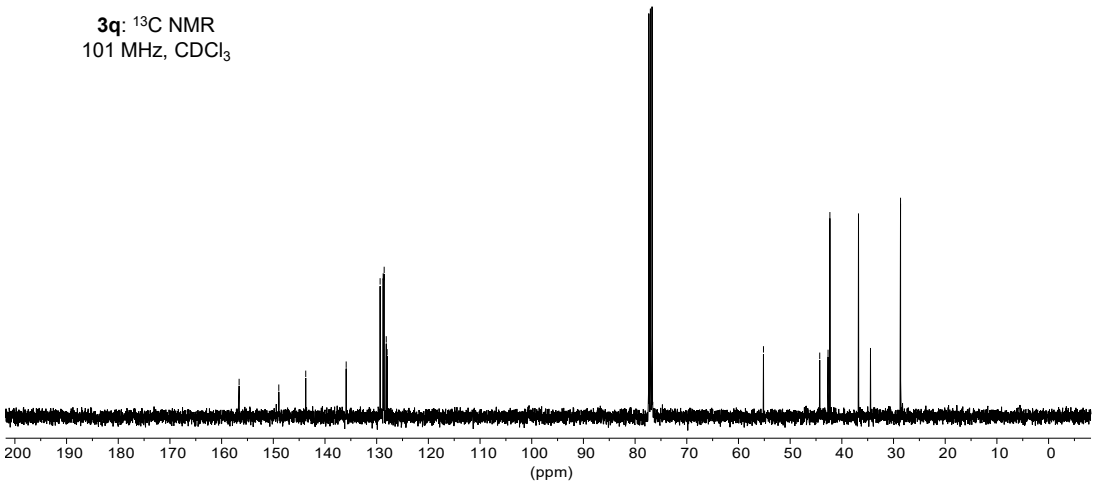


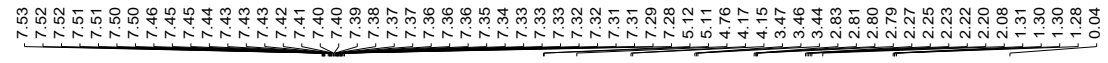
3q: ^{1}H NMR
400 MHz, $CDCl_3$

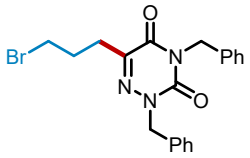




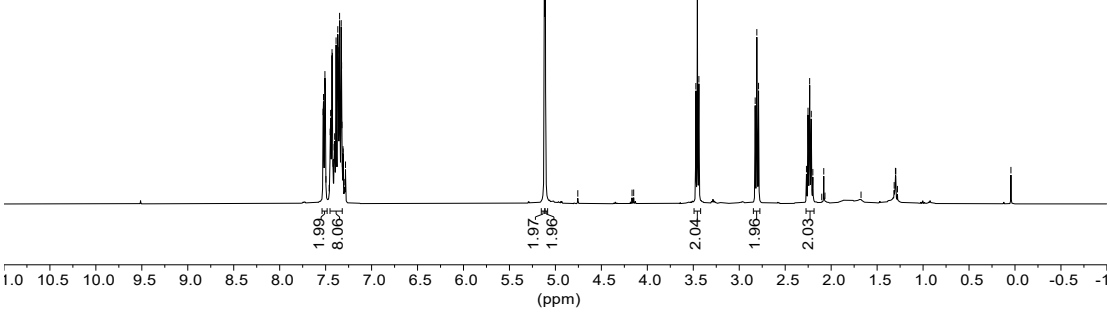
3q: ^{13}C NMR
101 MHz, $CDCl_3$

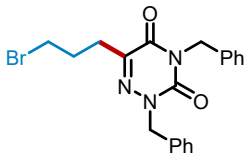




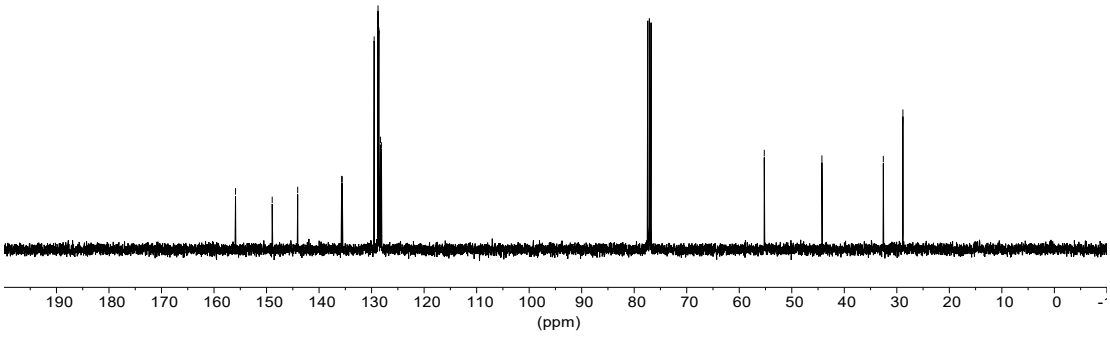


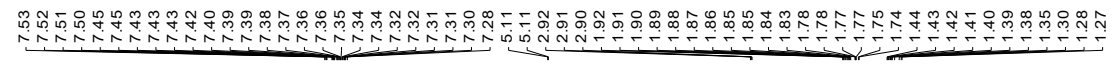
3r: ^{1}H NMR
400 MHz, $CDCl_3$

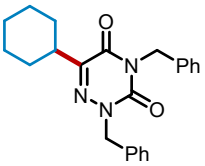




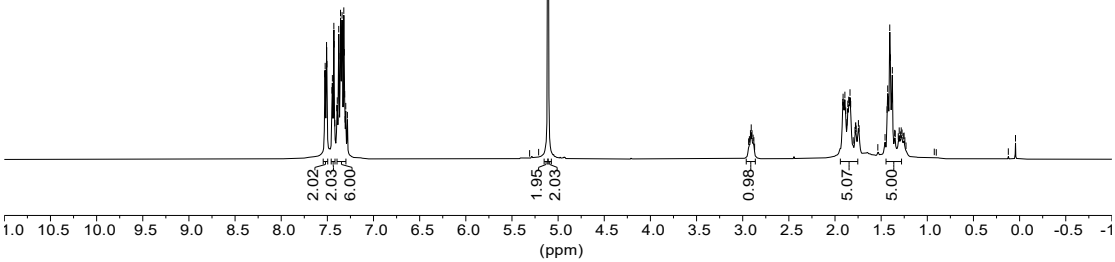
3r: ^{13}C NMR
101 MHz, $CDCl_3$

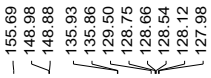


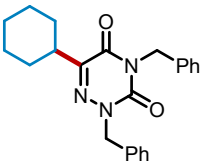




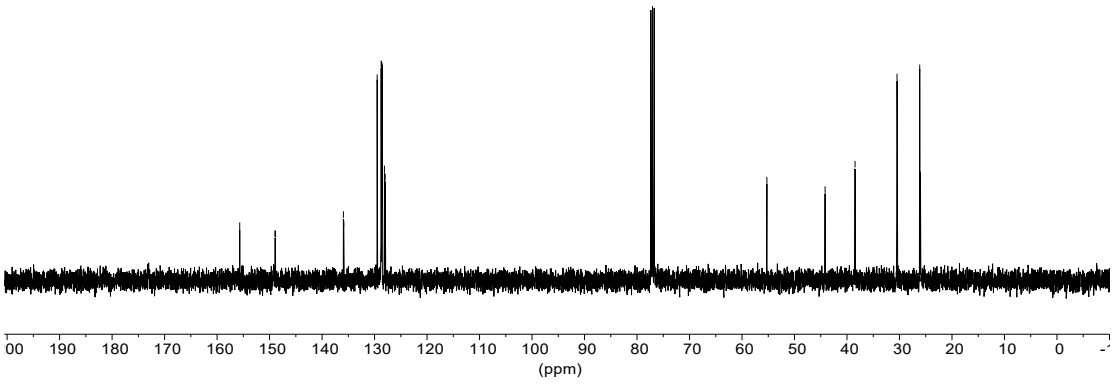
3s ^{1}H NMR
400 MHz, $CDCl_3$

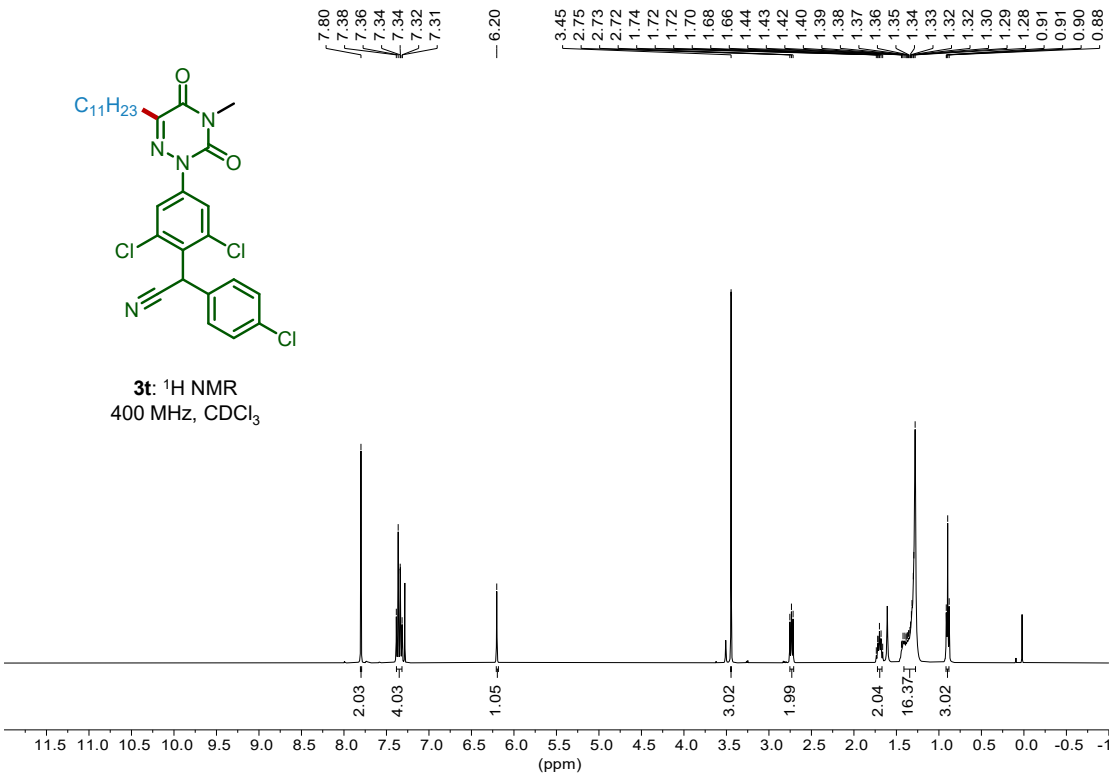




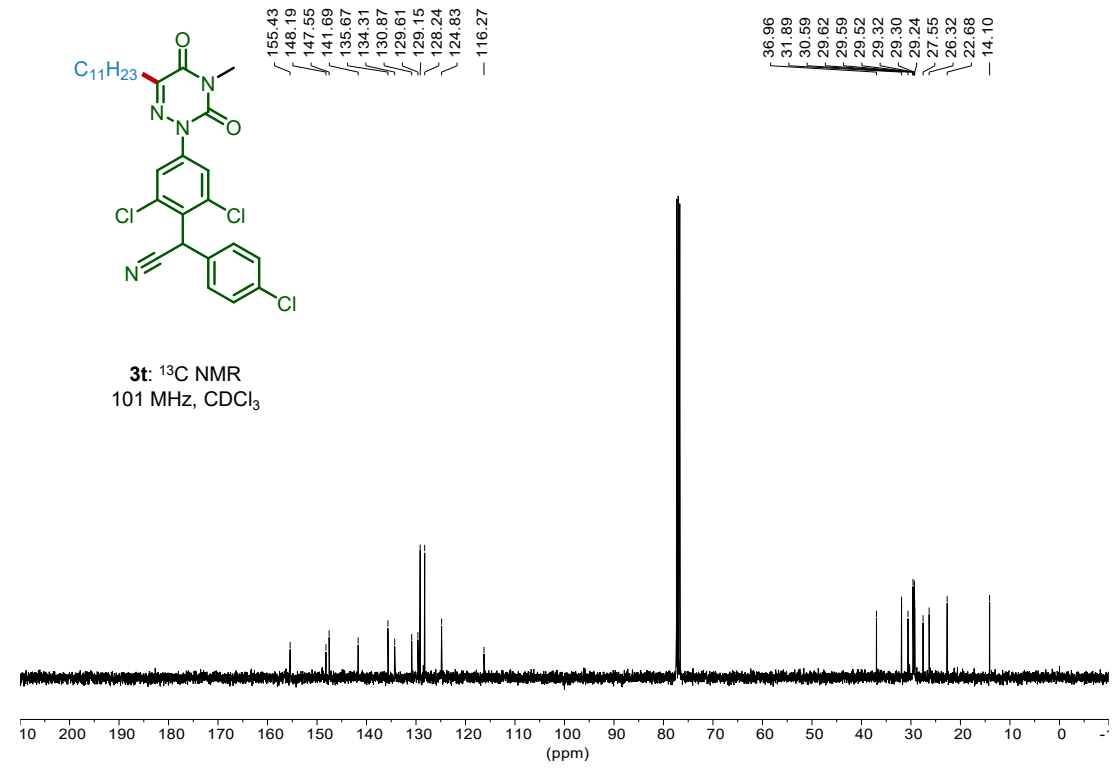


3s: ^{13}C NMR
101 MHz, $CDCl_3$

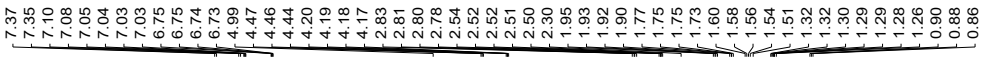


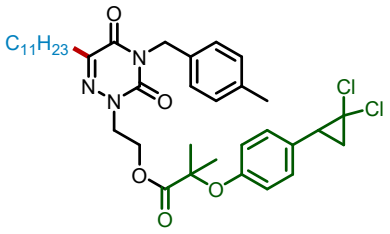


3t: ^{1}H NMR
400 MHz, $CDCl_3$

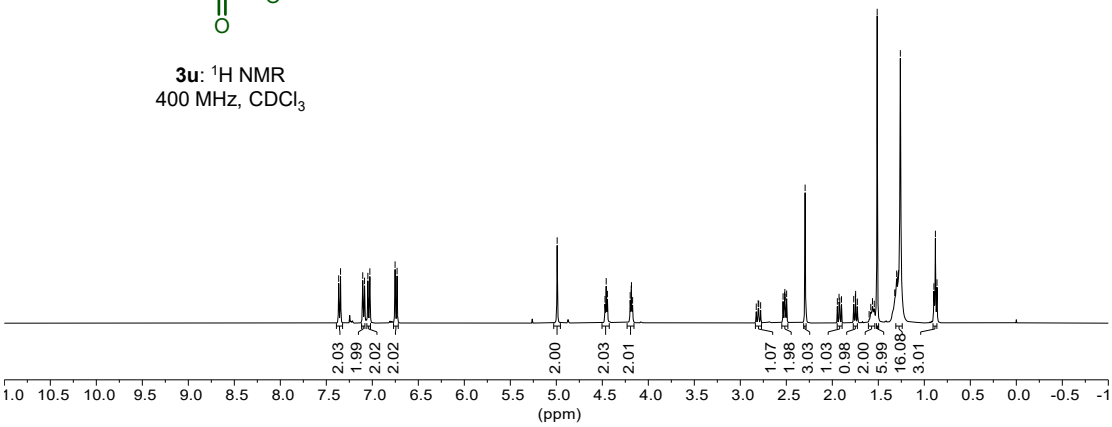


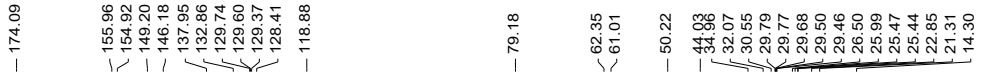
3t: ^{13}C NMR
101 MHz, $CDCl_3$

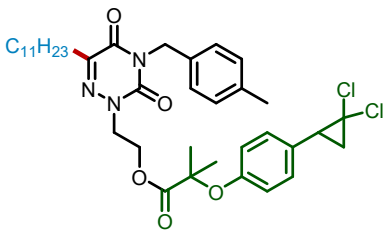




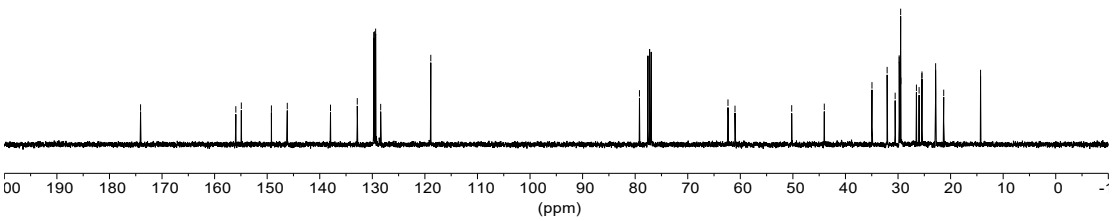
3u: ^{1}H NMR
400 MHz, $CDCl_3$

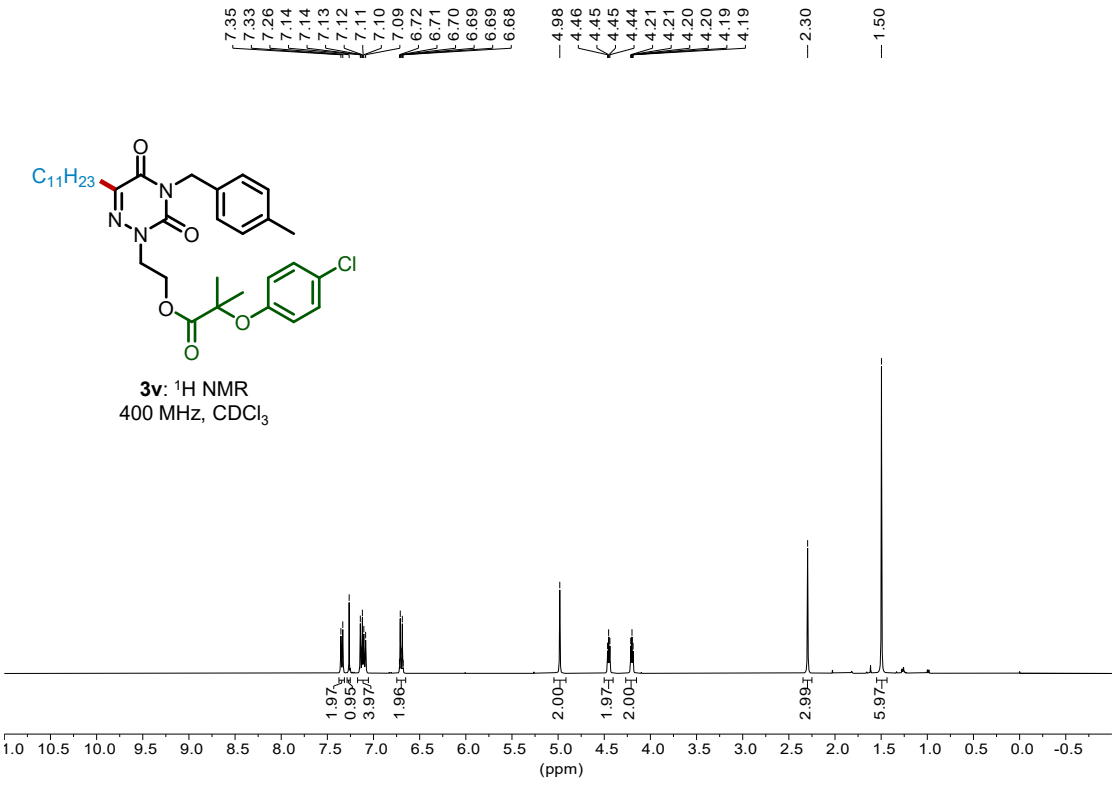




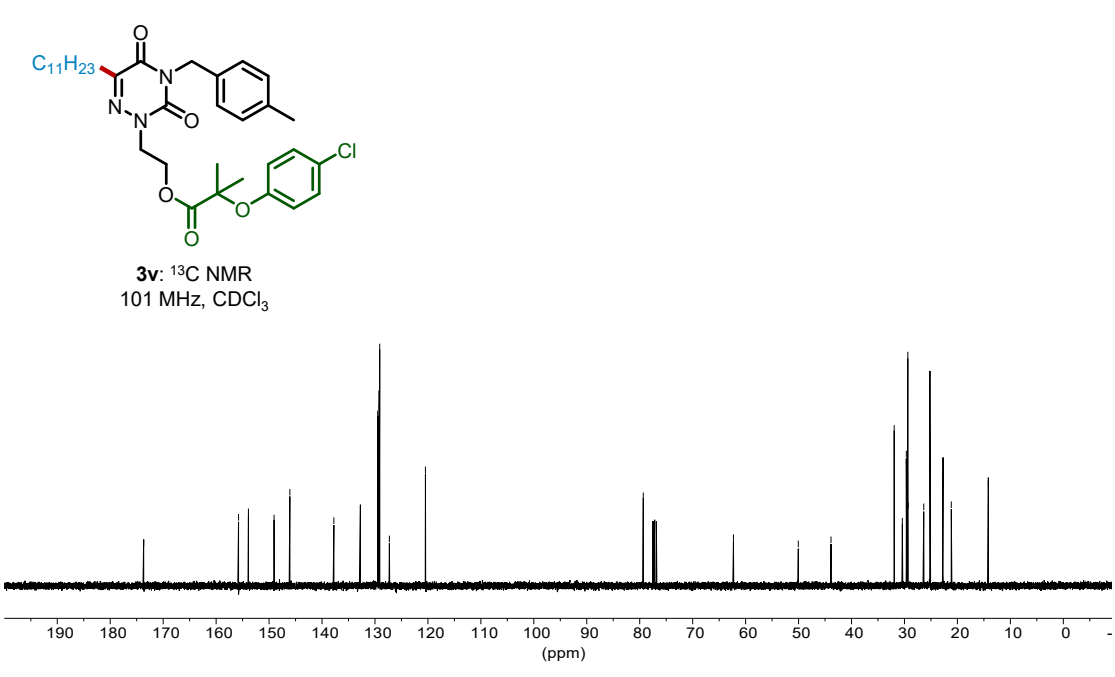


3u: ^{13}C NMR
101 MHz, $CDCl_3$

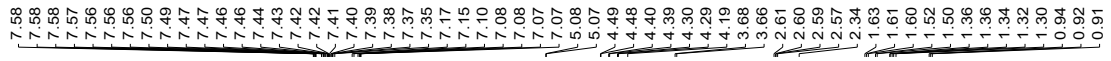


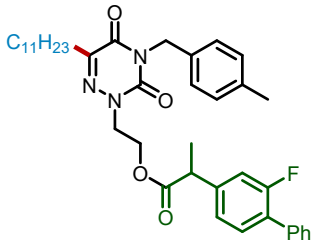


3v: ^{1}H NMR
400 MHz, $CDCl_3$

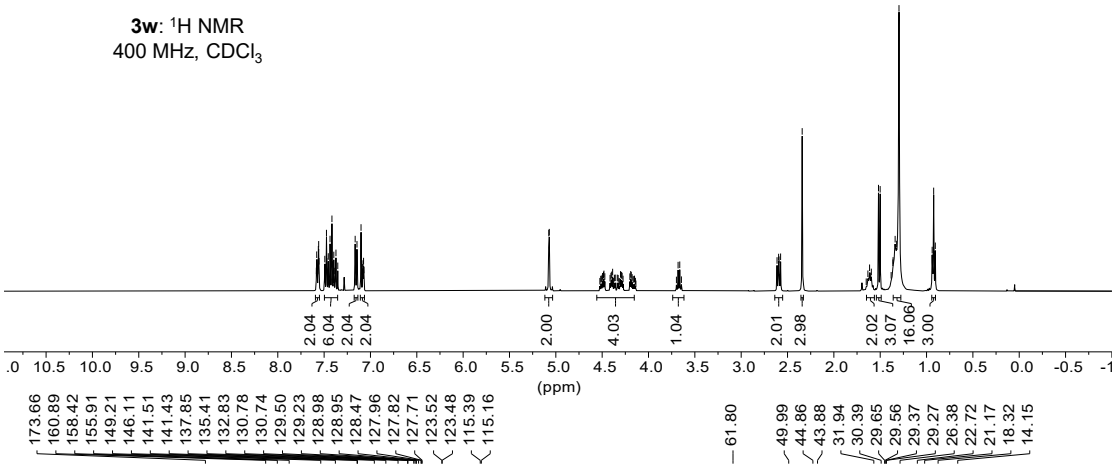


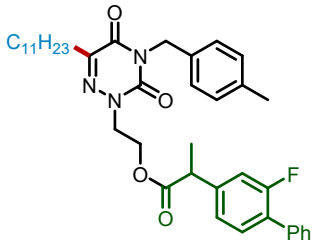
3v: ^{13}C NMR
101 MHz, $CDCl_3$



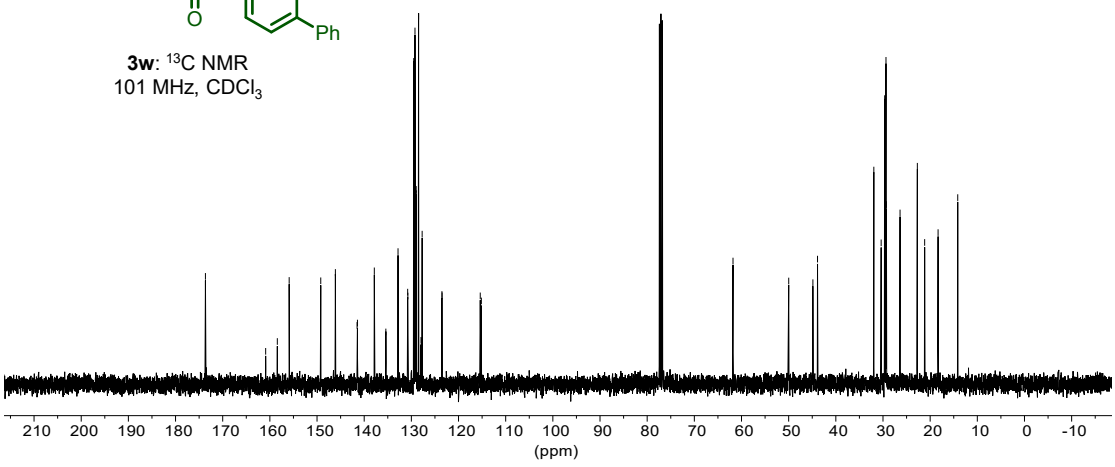


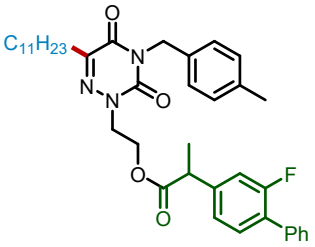
3w: ^{1}H NMR
400 MHz, $CDCl_3$



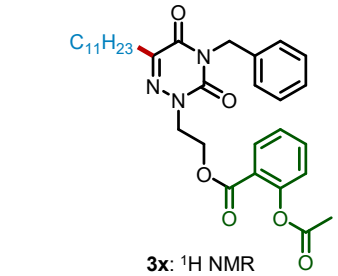


3w: ^{13}C NMR
101 MHz, $CDCl_3$

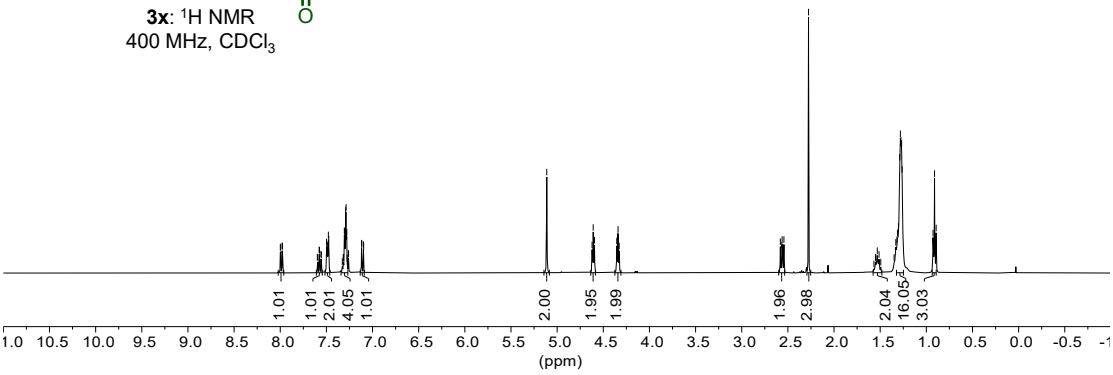


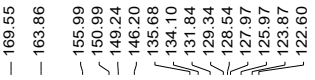


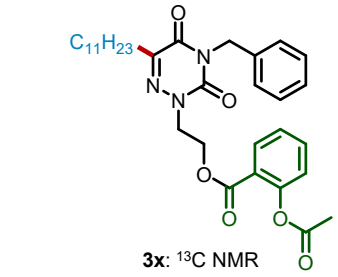
3w: ^{19}F NMR
376 MHz, $CDCl_3$



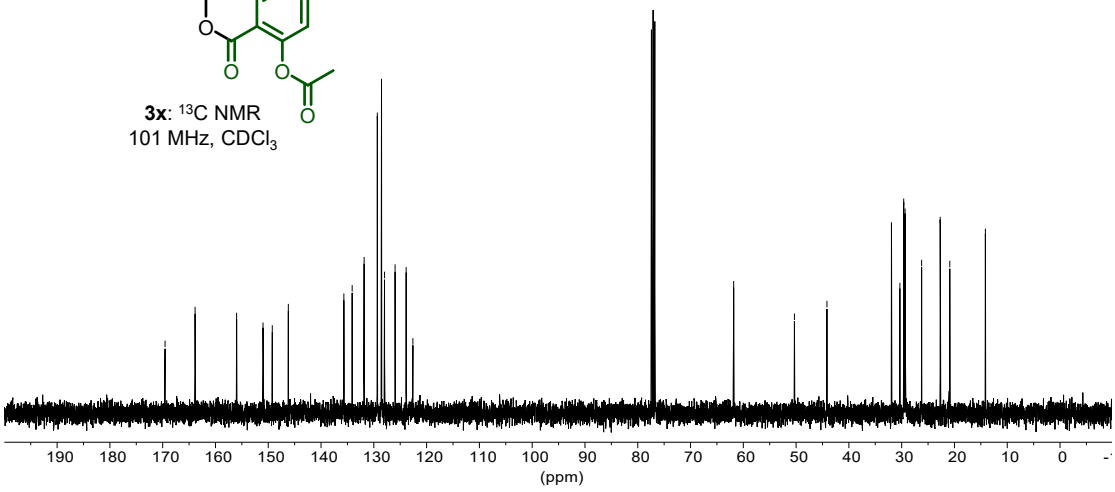
3x: ^{1}H NMR
400 MHz, $CDCl_3$

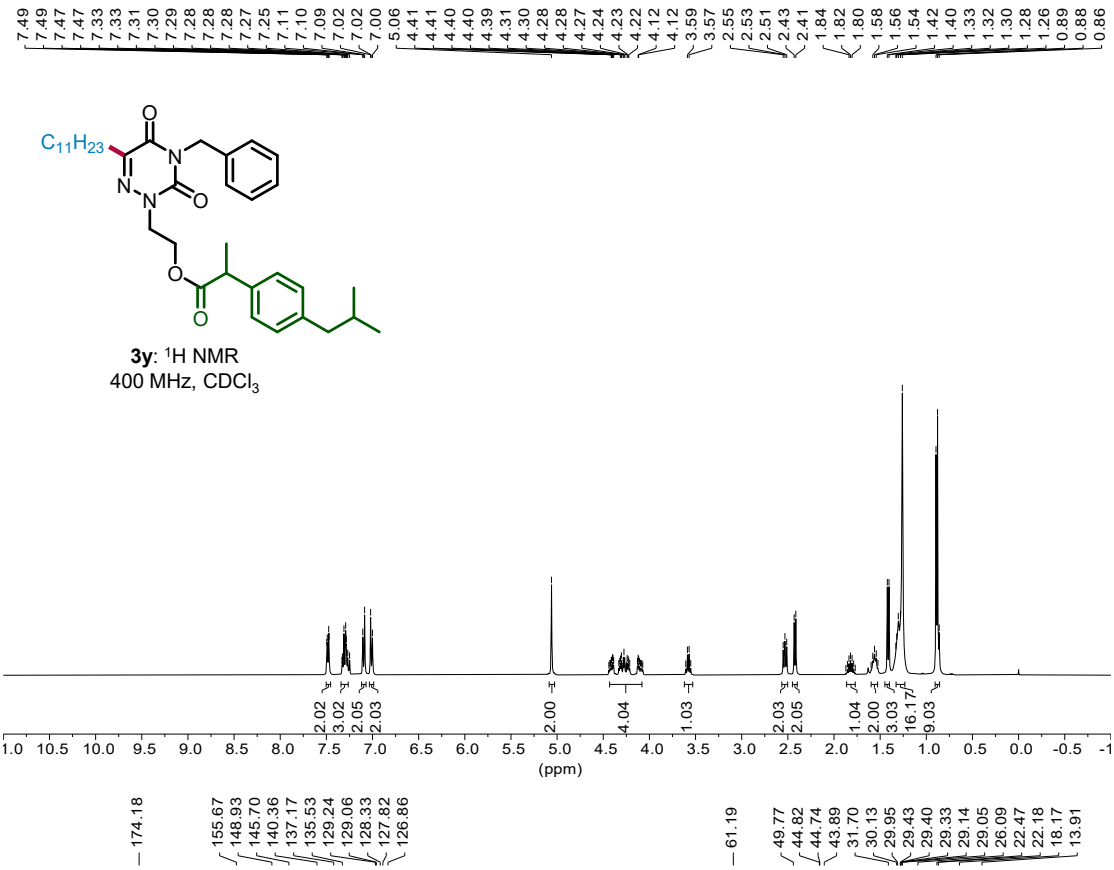




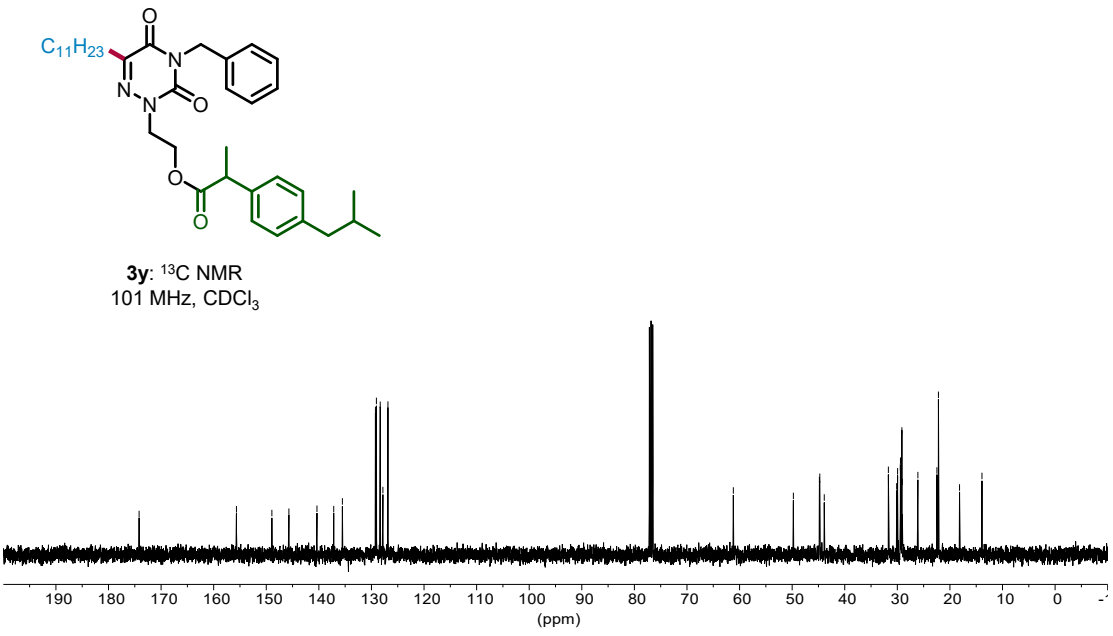


3x: ^{13}C NMR
101 MHz, $CDCl_3$

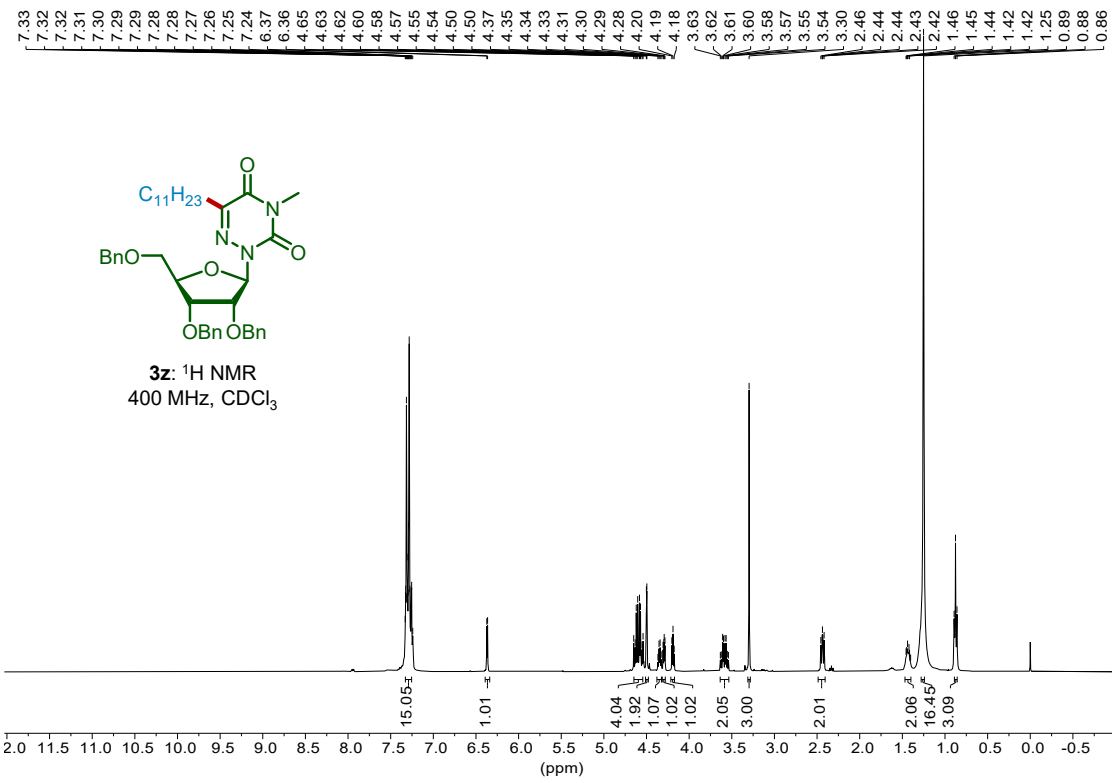




3y: ^{1}H NMR
400 MHz, $CDCl_3$



3y: ^{13}C NMR
101 MHz, $CDCl_3$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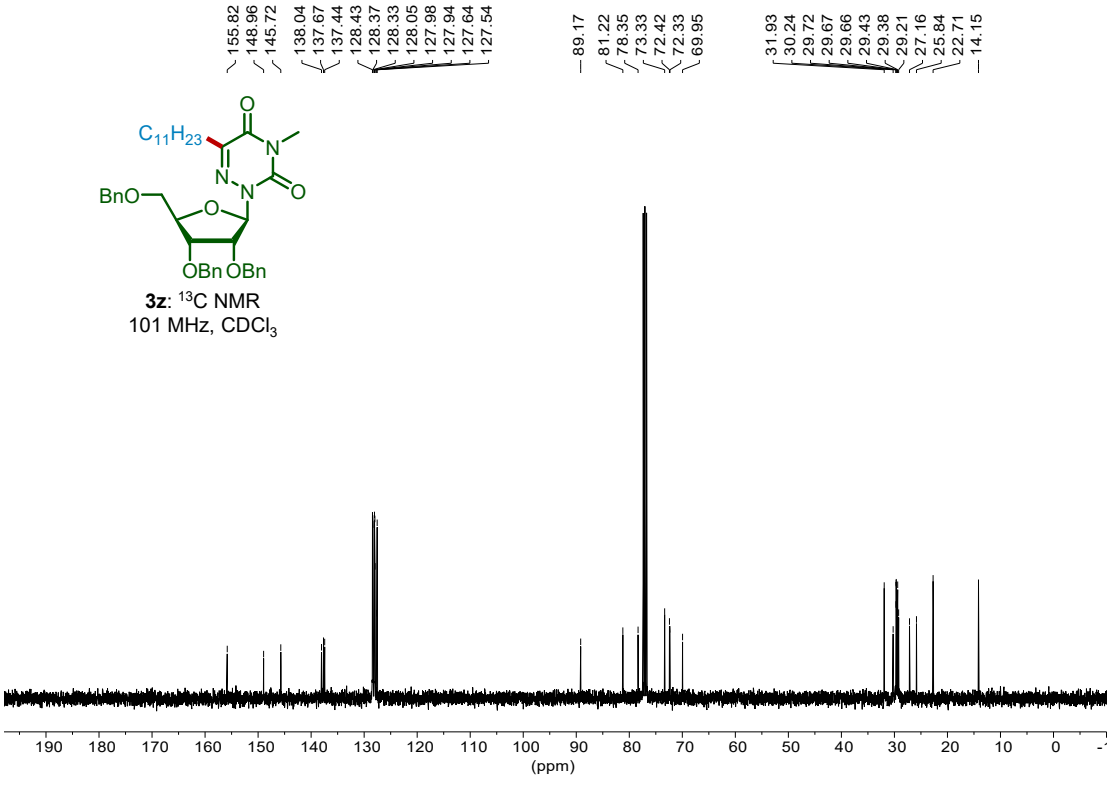
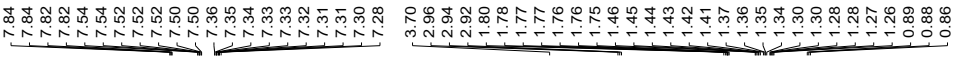
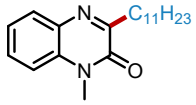
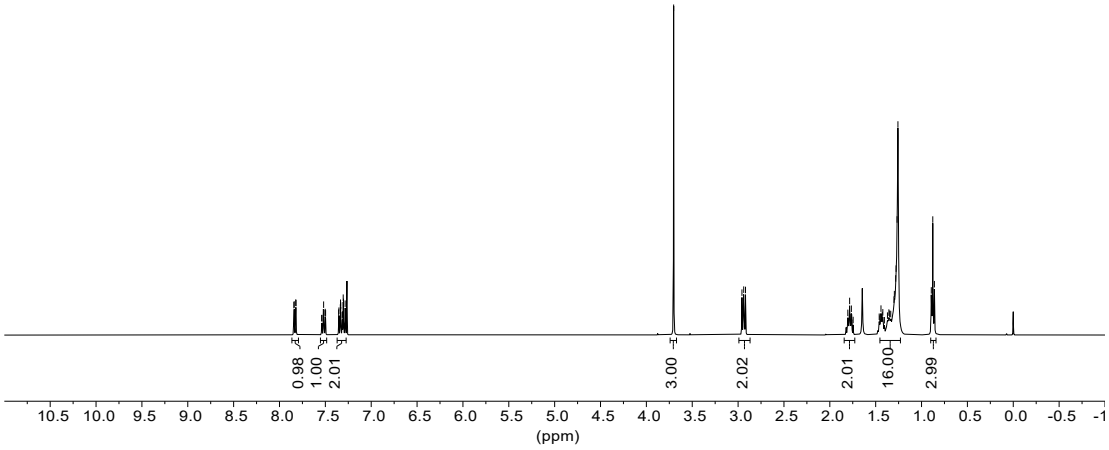
C11H23
BnO
OBn OBn
3z: 1H NMR
400 MHz, CDCl3
15.05 1.01 4.04 1.92 1.07 1.02 1.02 2.05 3.00 2.01 2.06 16.45 3.09
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
(ppm)

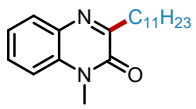

155.82 148.96 145.72 138.04 137.67 137.44 128.43 128.37 128.33 128.05 127.98 127.94 127.64 127.54
89.17 81.22 78.35 73.33 72.42 72.33 69.95
31.93 30.24 29.72 29.67 29.66 29.43 29.38 29.21 27.16 25.84 22.71 14.15
C11H23
BnO
OBn OBn
3z: 13C NMR
101 MHz, CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)



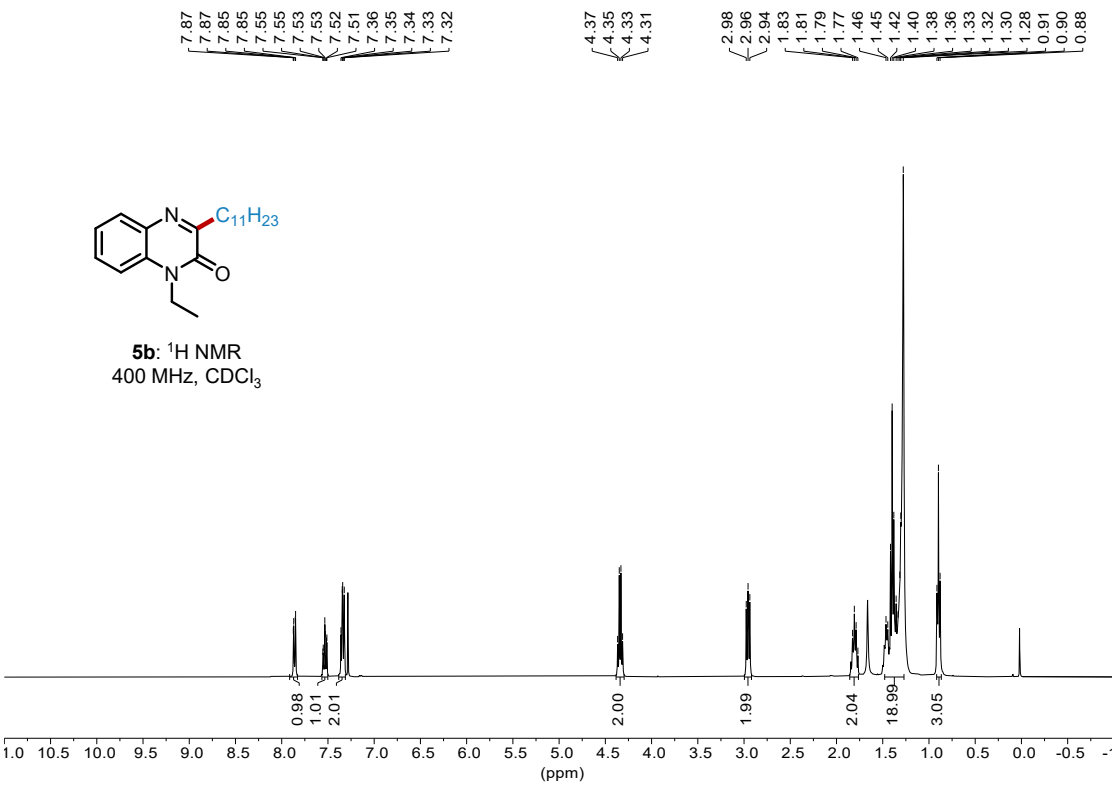


5a: ^{1}H NMR
400 MHz, $CDCl_3$

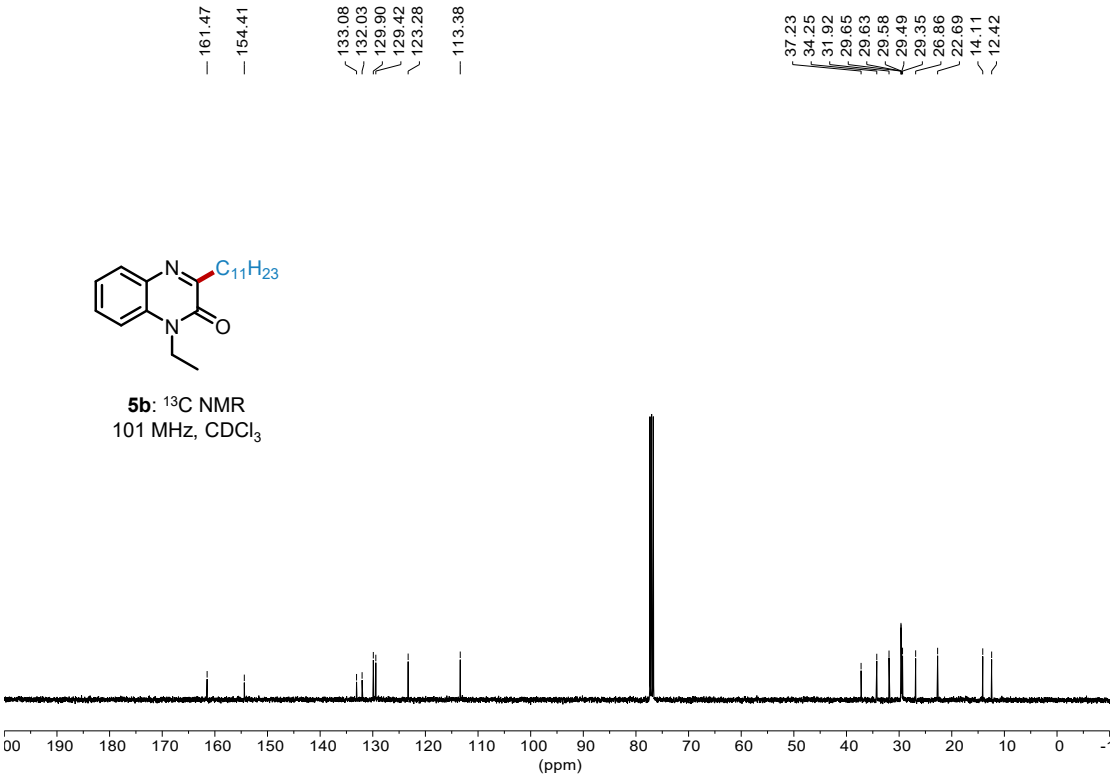




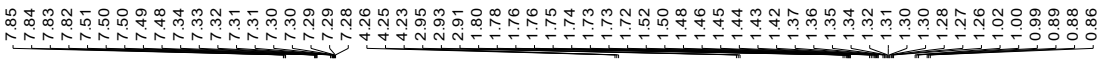
5a: ^{13}C NMR
101 MHz, $CDCl_3$

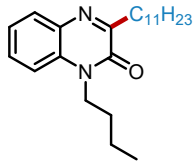


5b: ^{1}H NMR
400 MHz, $CDCl_3$

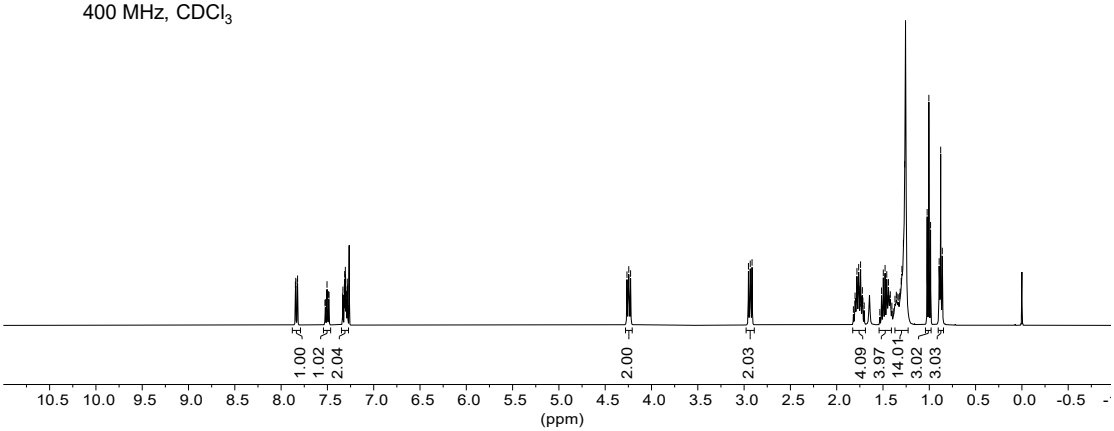


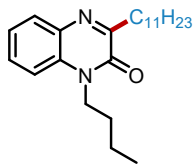
5b: ^{13}C NMR
101 MHz, $CDCl_3$



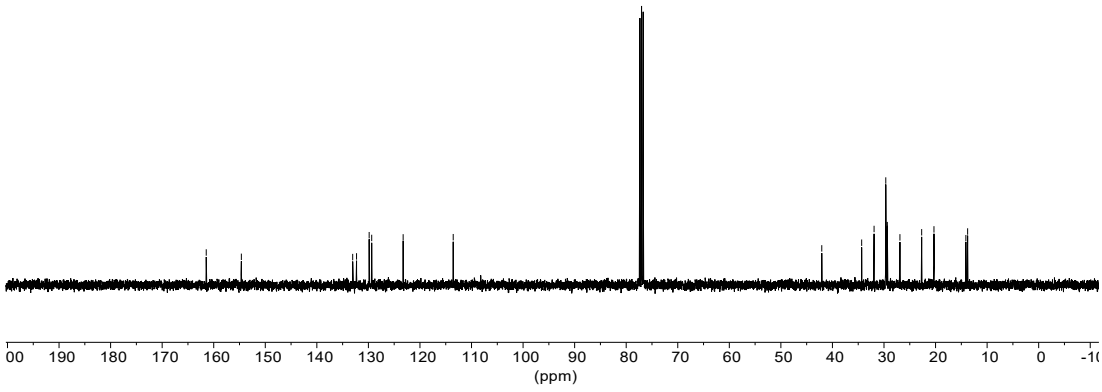


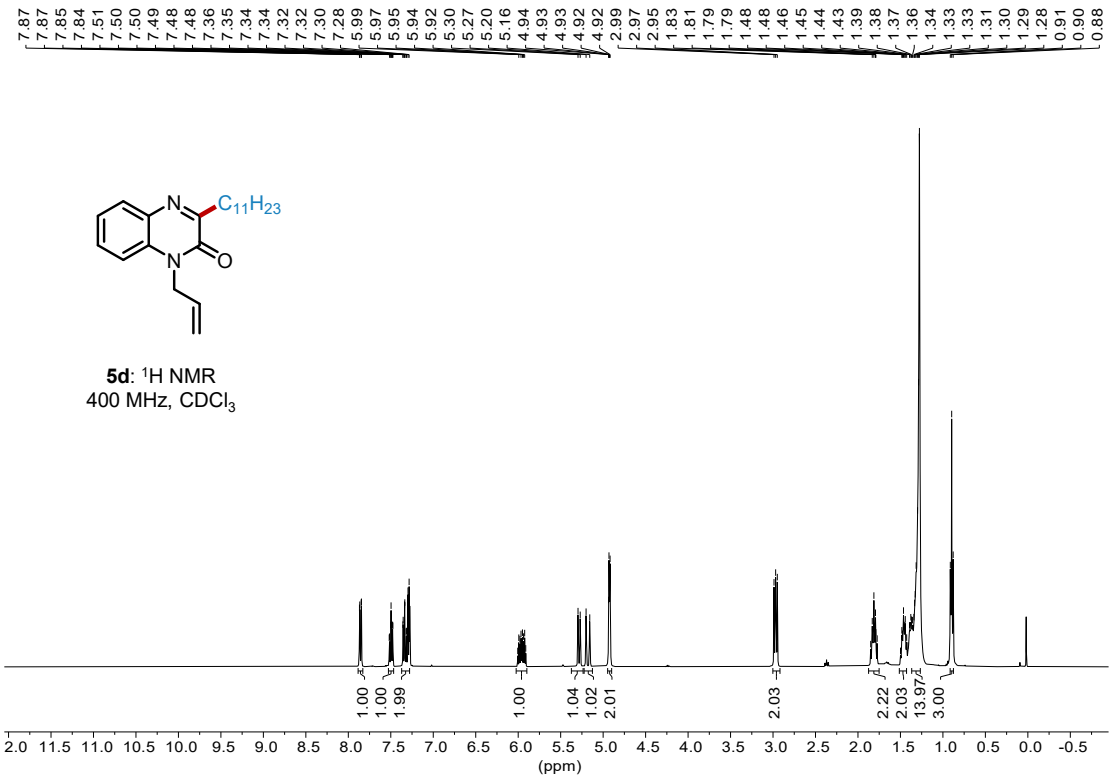
5c: ^{1}H NMR
400 MHz, $CDCl_3$



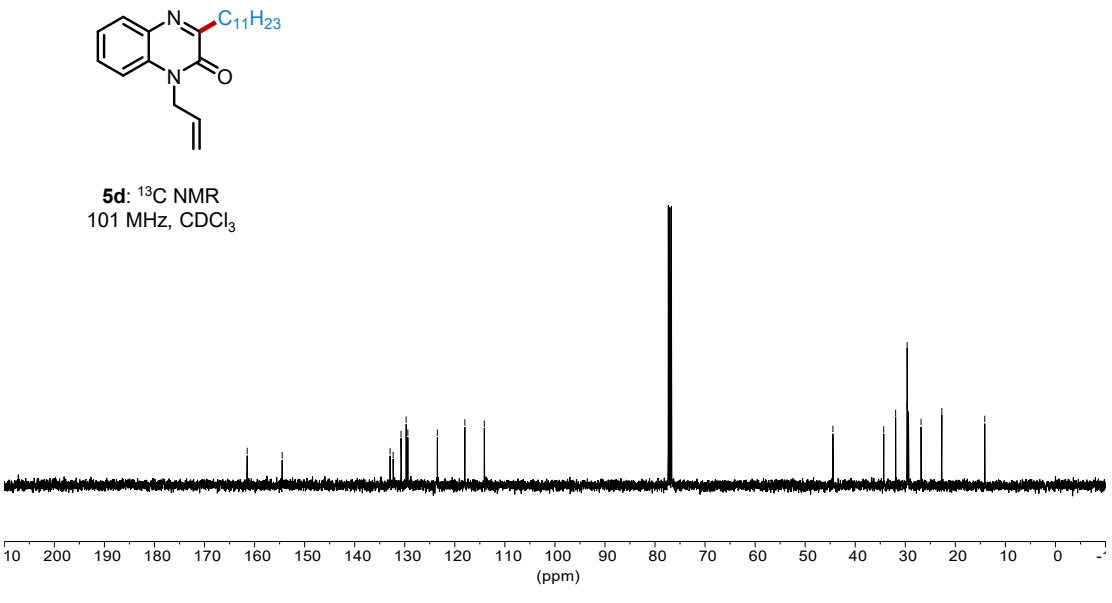


5c: ^{13}C NMR
101 MHz, $CDCl_3$

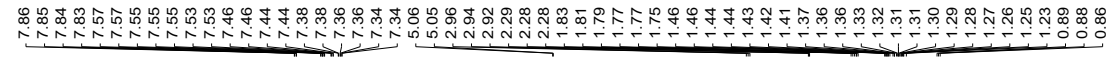


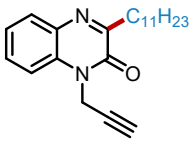


5d: ^{1}H NMR
400 MHz, $CDCl_3$

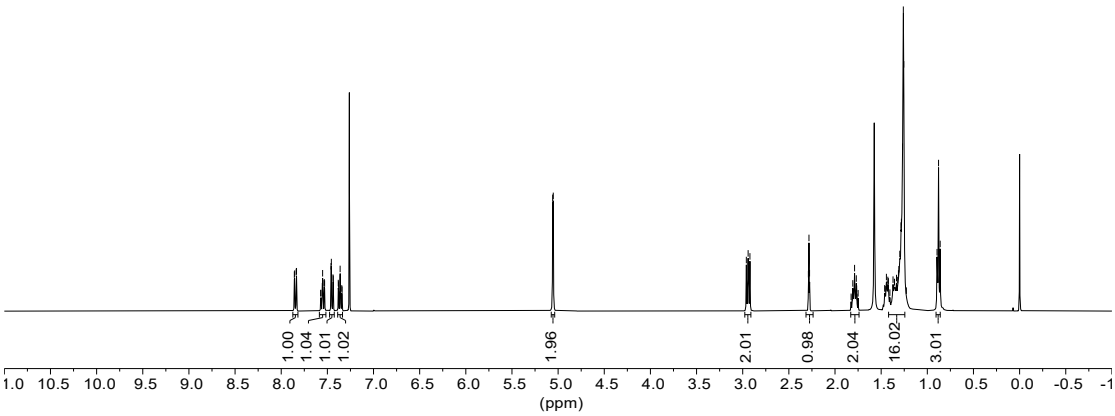


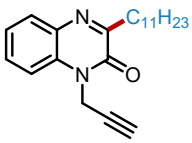
5d: ^{13}C NMR
101 MHz, $CDCl_3$



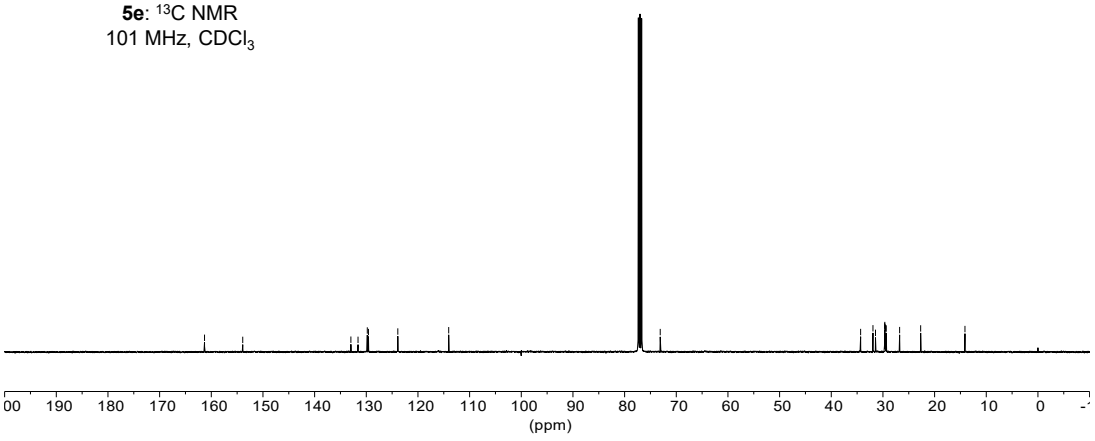


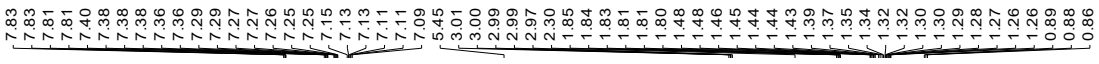
5e: ^{1}H NMR
400 MHz, $CDCl_3$

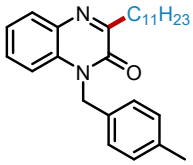




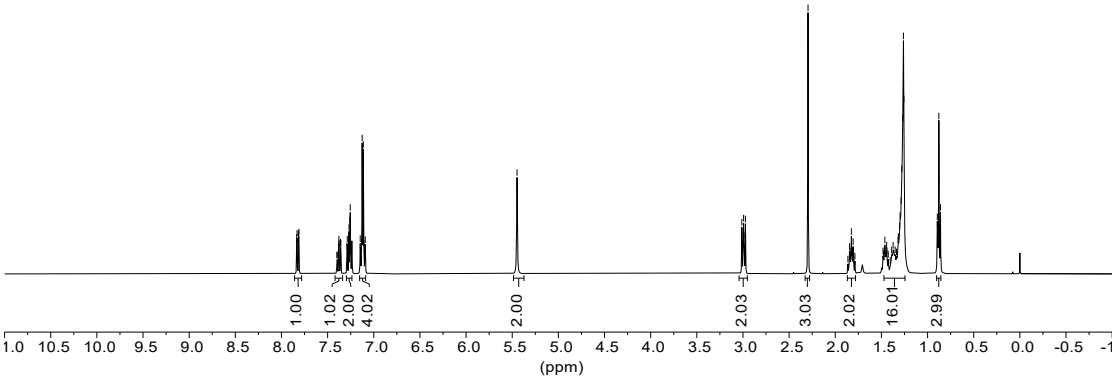
5e: ^{13}C NMR
101 MHz, $CDCl_3$

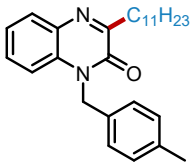




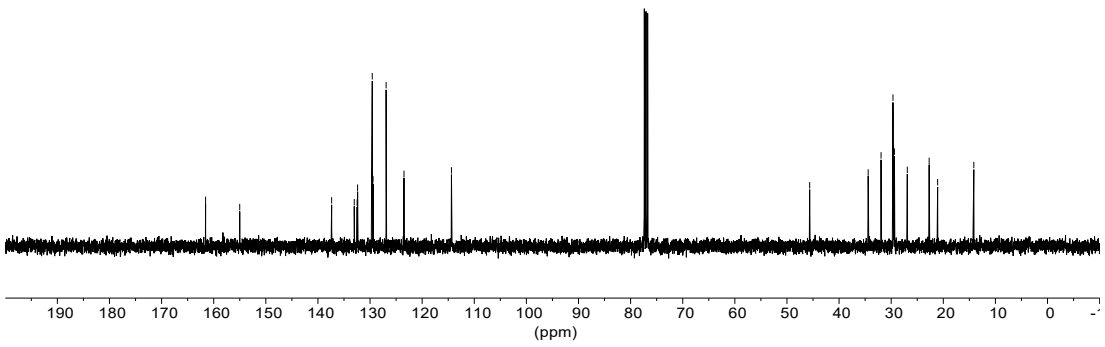


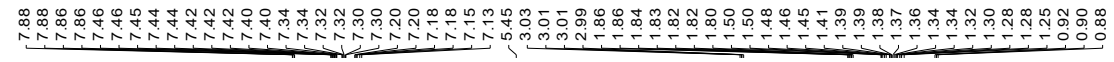
5f: ^{1}H NMR
400 MHz, $CDCl_3$

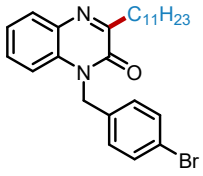




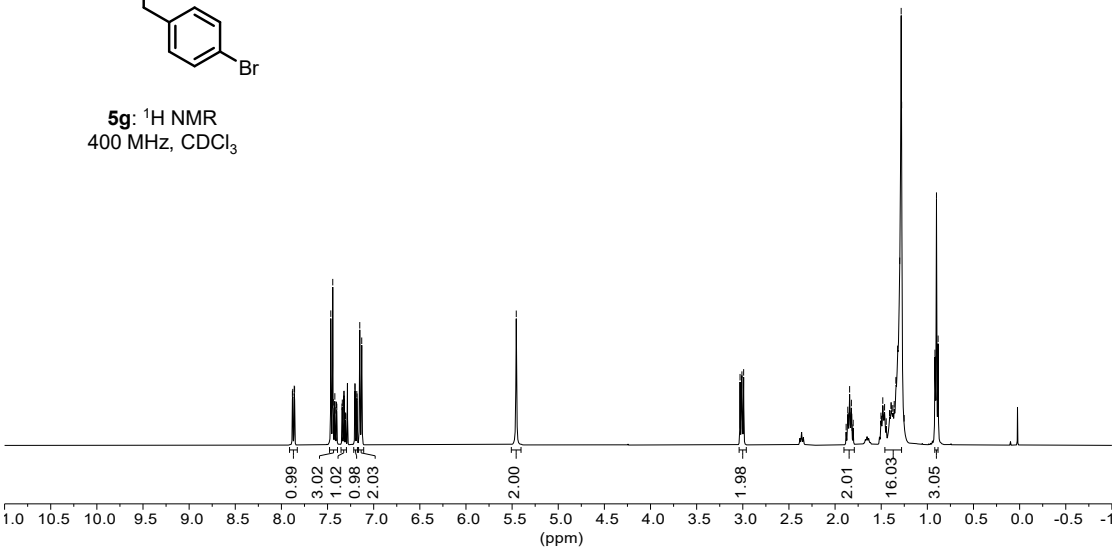
5f: ^{13}C NMR
101 MHz, $CDCl_3$

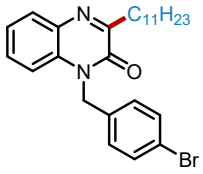




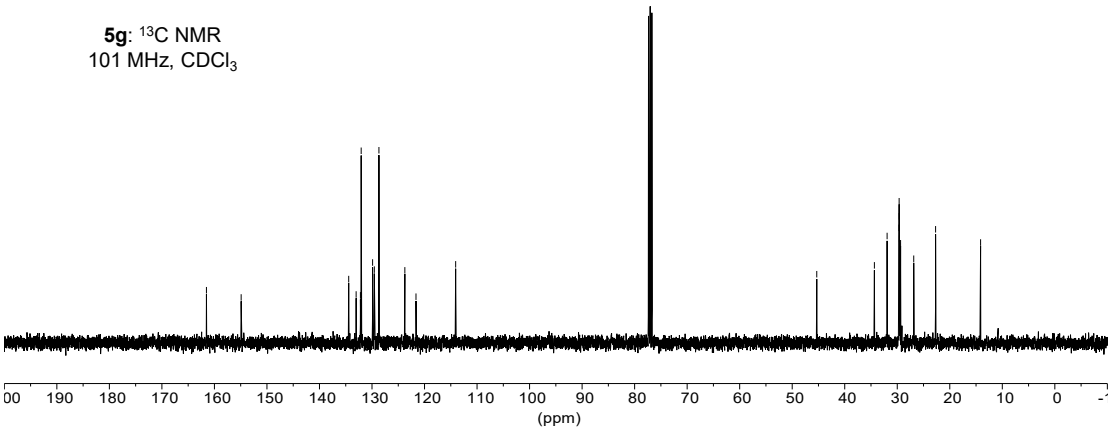


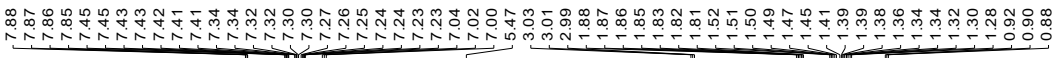
5g: ^{1}H NMR
400 MHz, $CDCl_3$

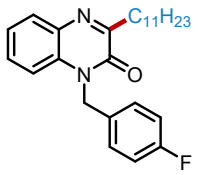




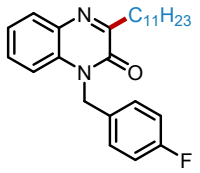
5g: ^{13}C NMR
101 MHz, $CDCl_3$



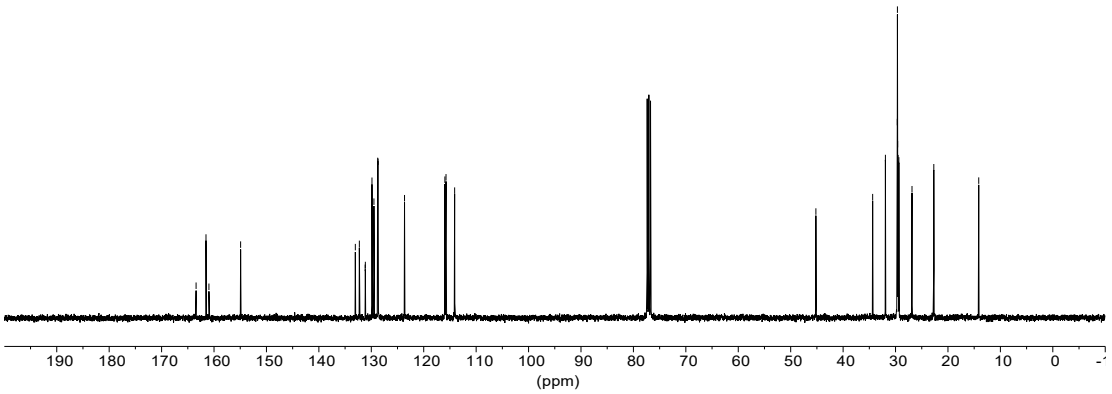


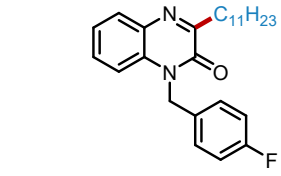


5h: ^{1}H NMR
400 MHz, $CDCl_3$

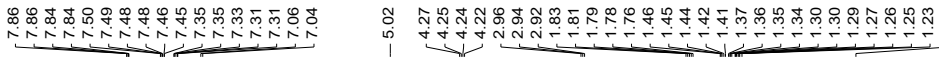


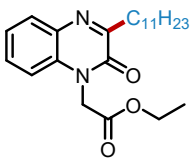
5h: ^{13}C NMR
101 MHz, $CDCl_3$



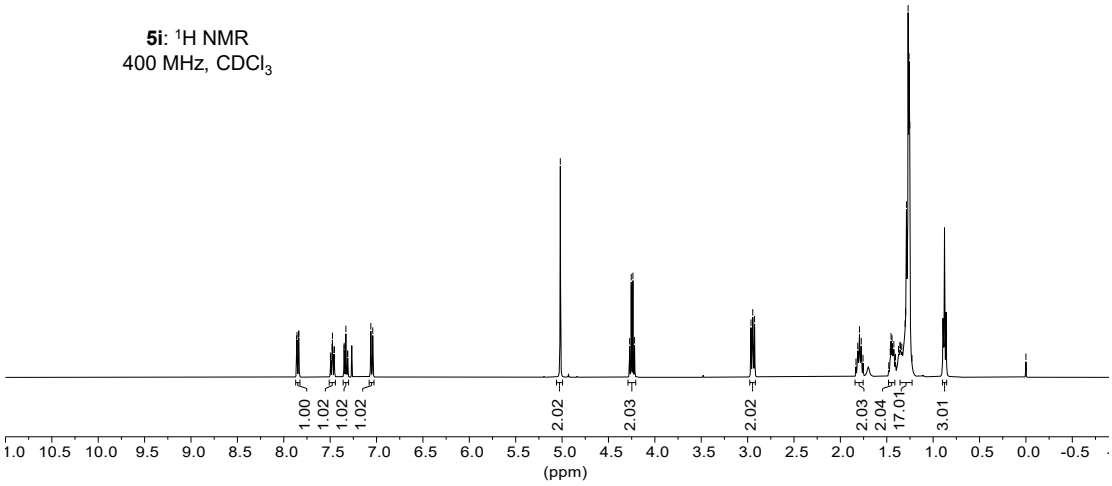


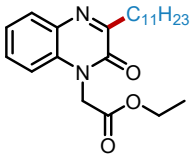
5h: ^{19}F NMR
376 MHz, $CDCl_3$



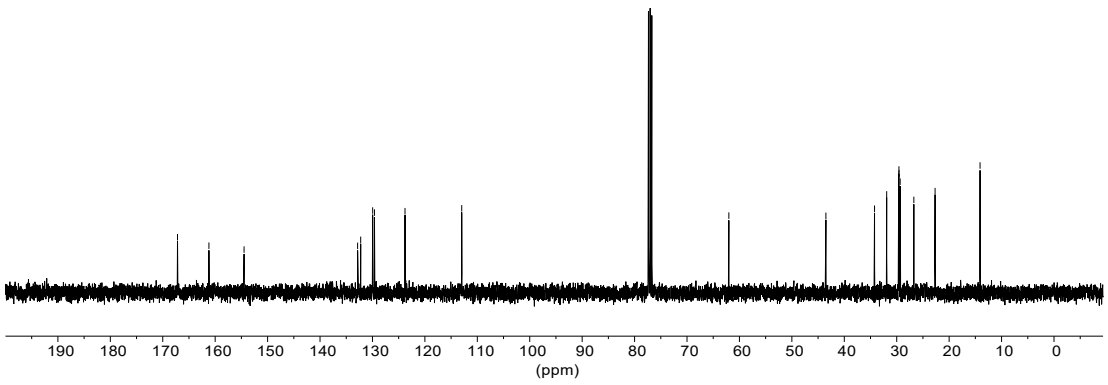


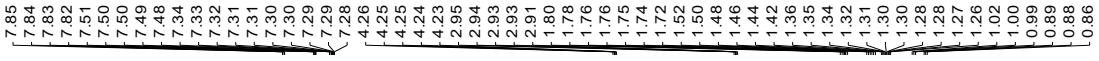
5i: ^{1}H NMR
400 MHz, $CDCl_3$

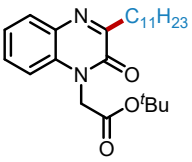




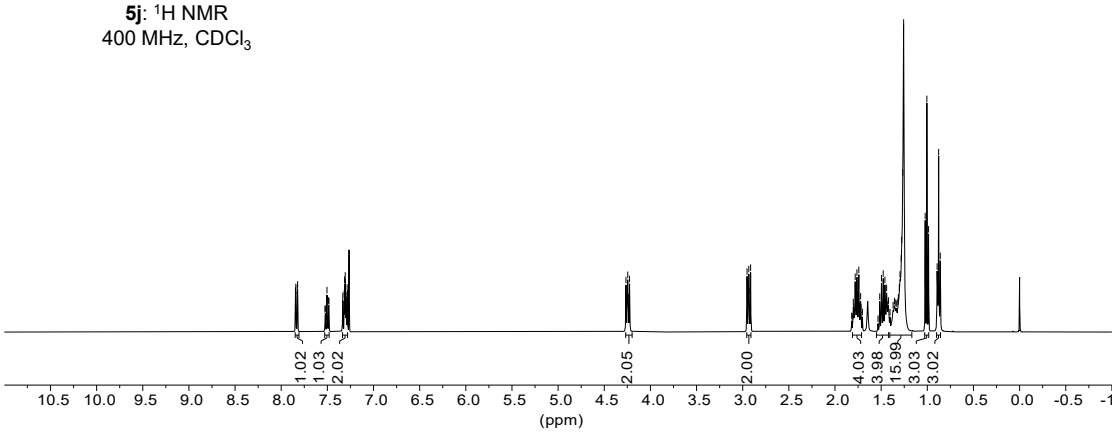
5i: ^{13}C NMR
101 MHz, $CDCl_3$

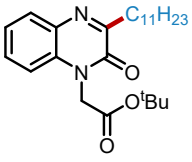




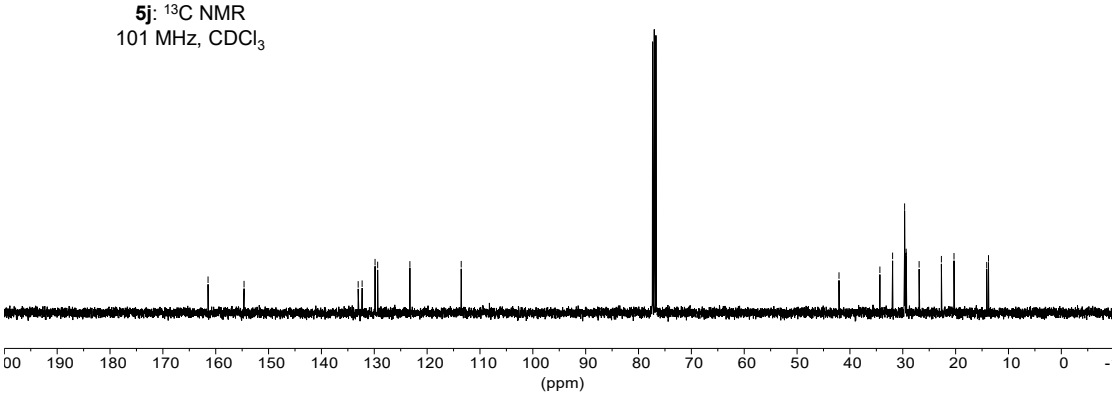


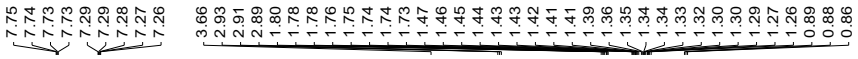
5j: ^{1}H NMR
400 MHz, $CDCl_3$

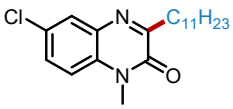




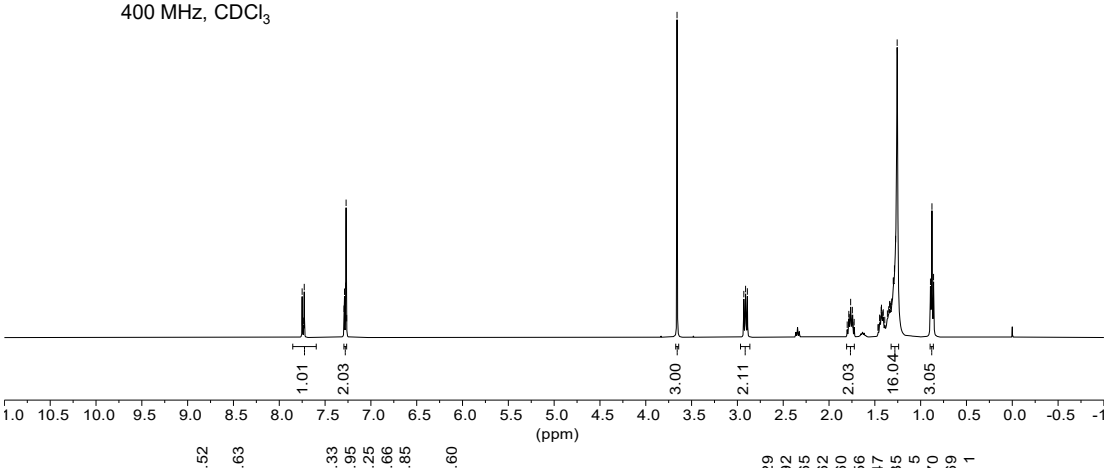
5j: ^{13}C NMR
101 MHz, $CDCl_3$

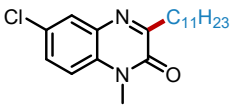




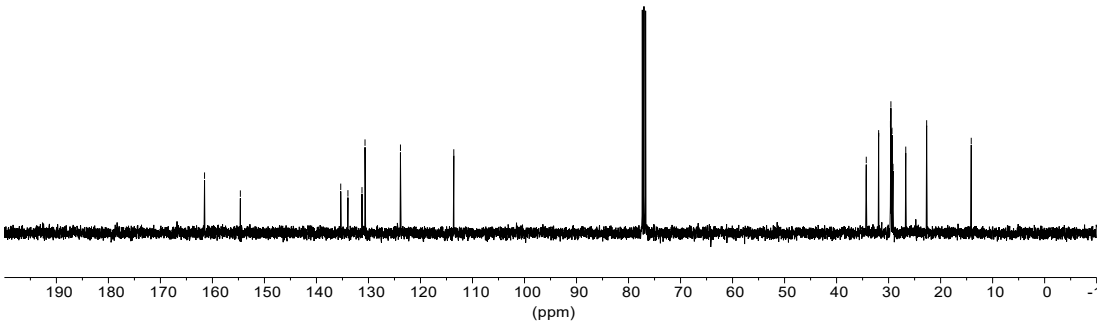


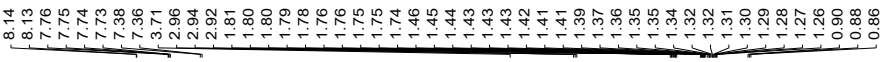
5k: ^{1}H NMR
400 MHz, $CDCl_3$

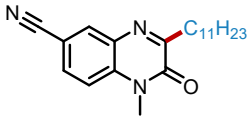




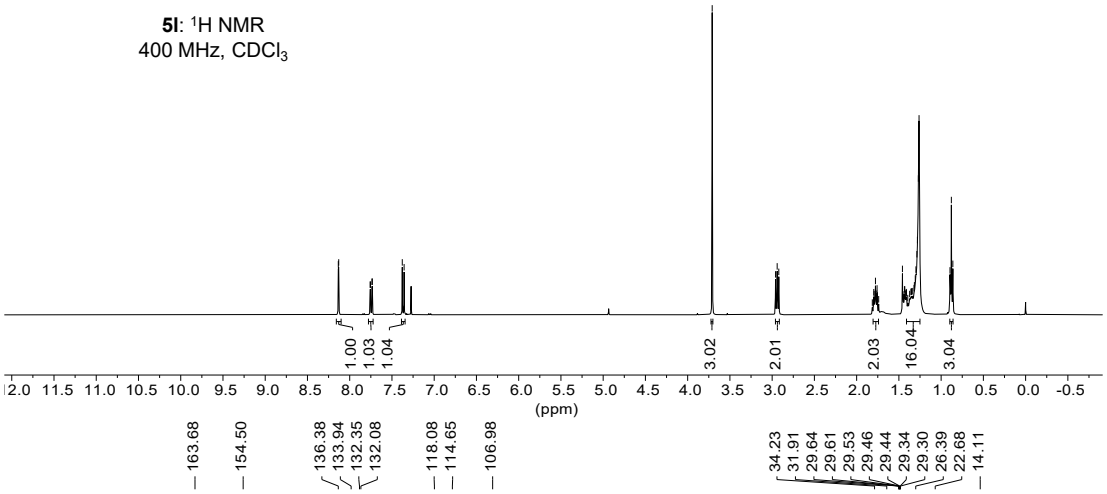
5k: ^{13}C NMR
101 MHz, $CDCl_3$

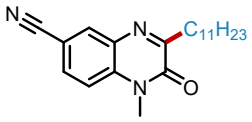




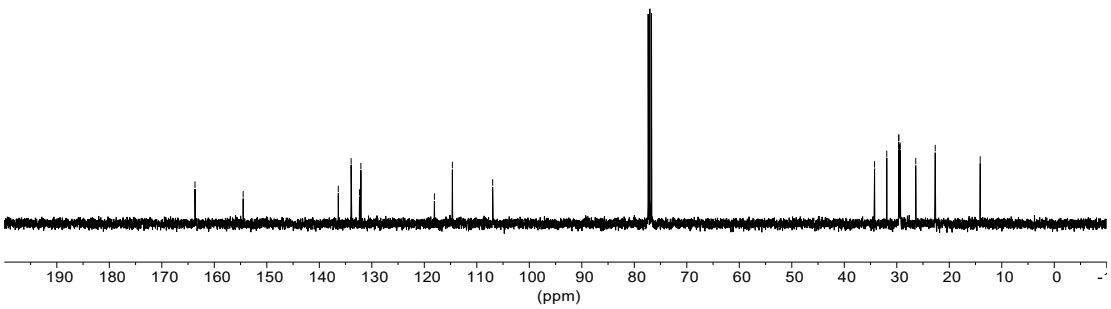


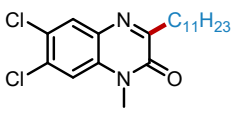
5l: ^{1}H NMR
400 MHz, $CDCl_3$



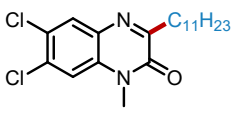


5l: ^{13}C NMR
101 MHz, $CDCl_3$

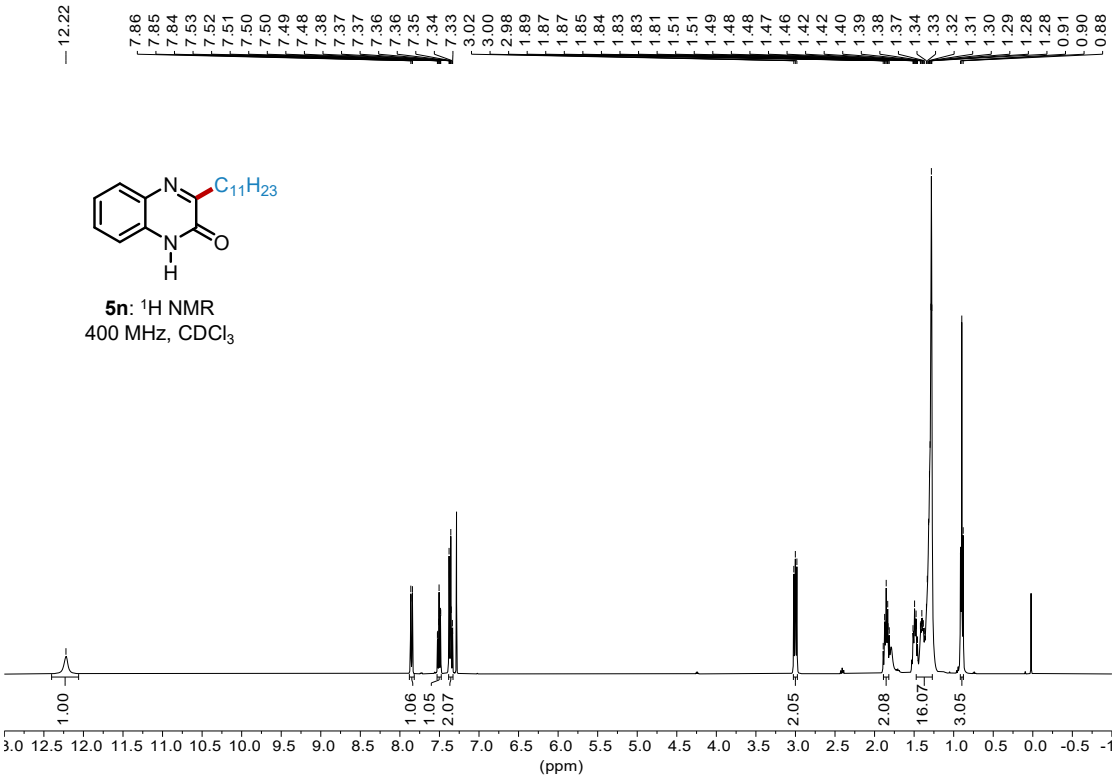




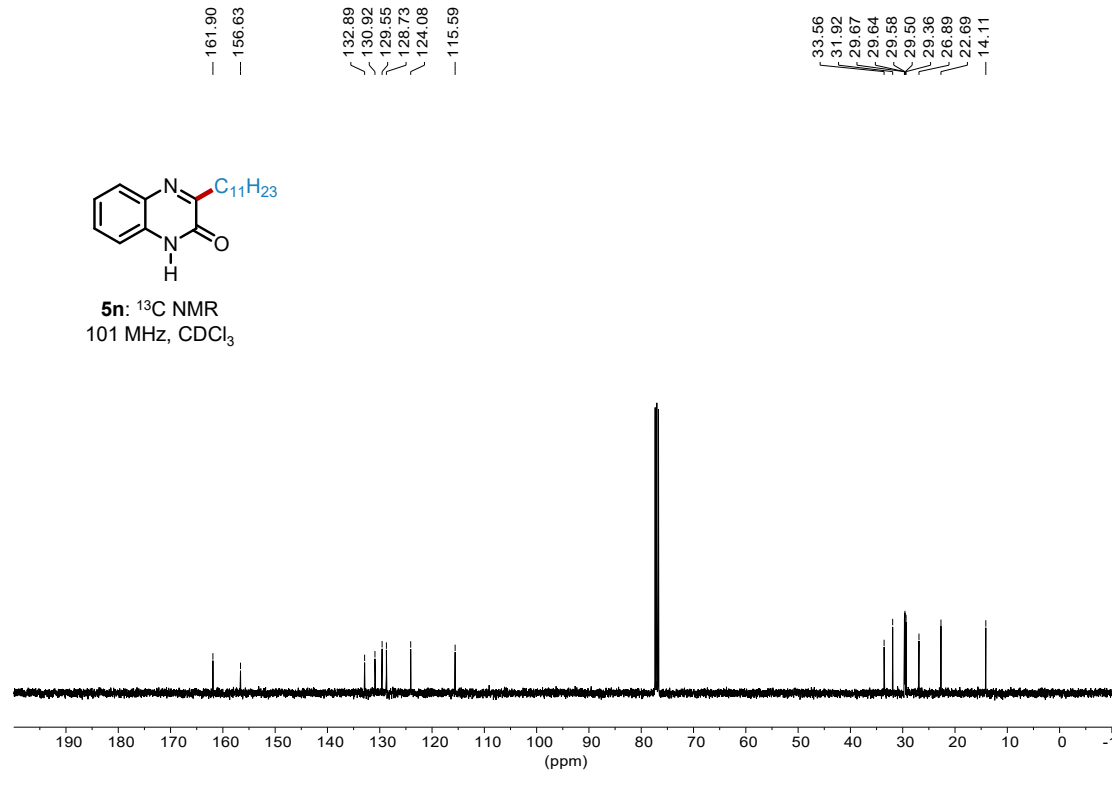
5m: ^{1}H NMR
400 MHz, $CDCl_3$



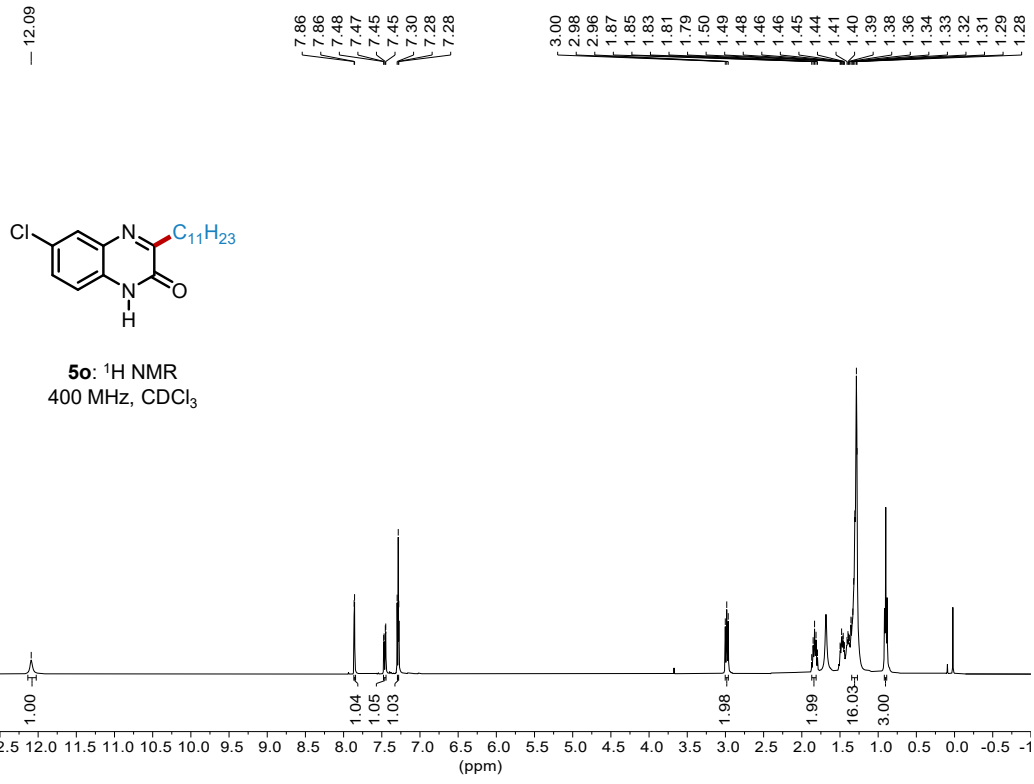
5m: ^{13}C NMR
101 MHz, $CDCl_3$



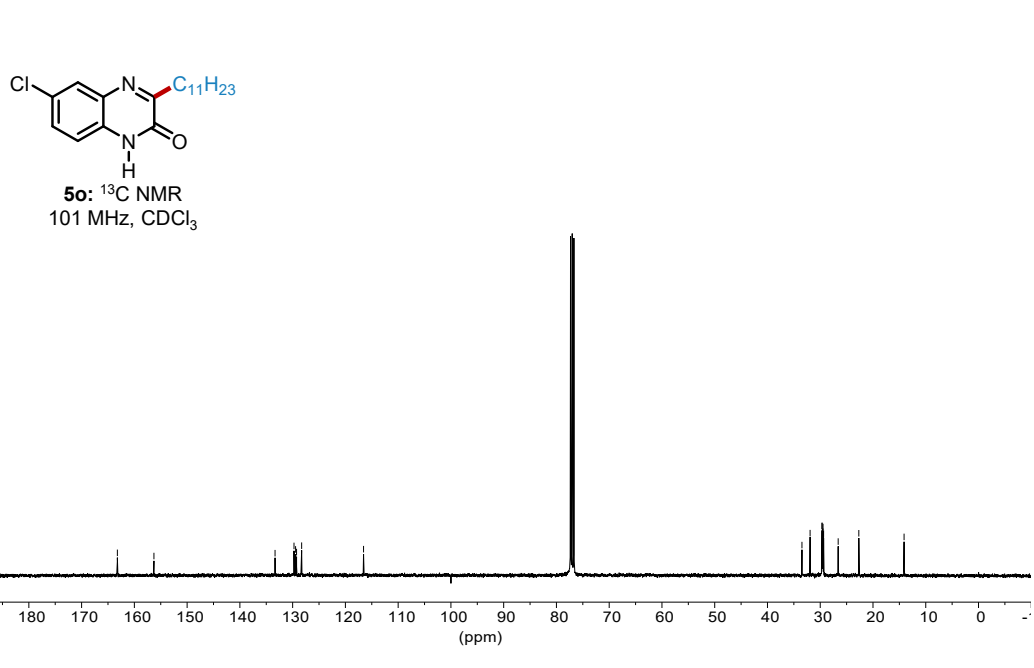
5n: ^{1}H NMR
400 MHz, $CDCl_3$



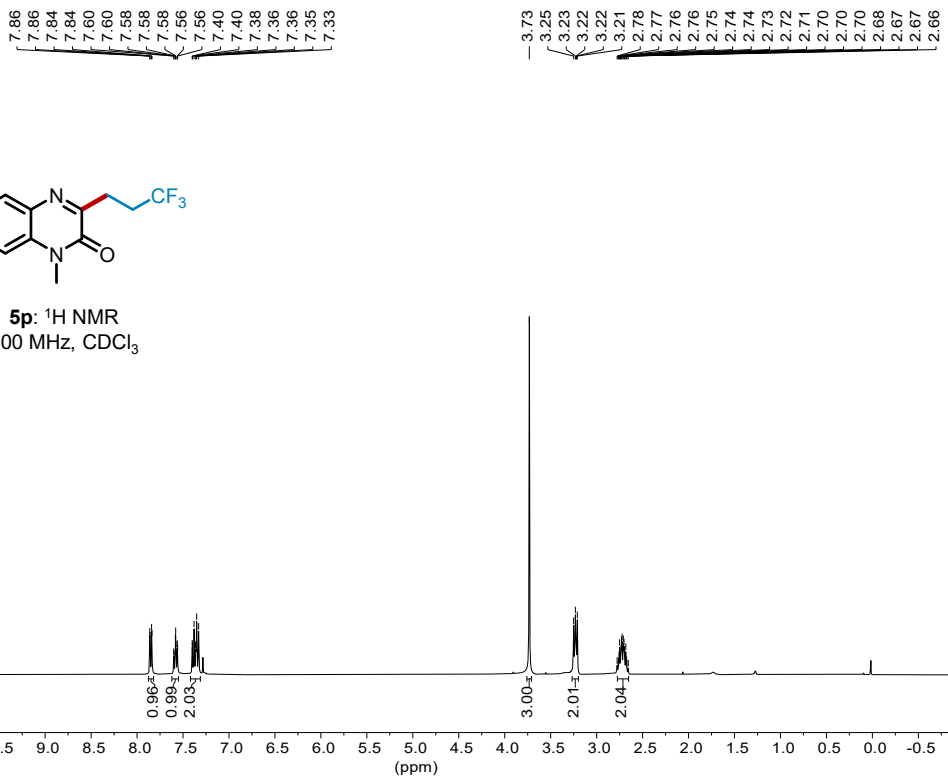
5n: ^{13}C NMR
101 MHz, $CDCl_3$



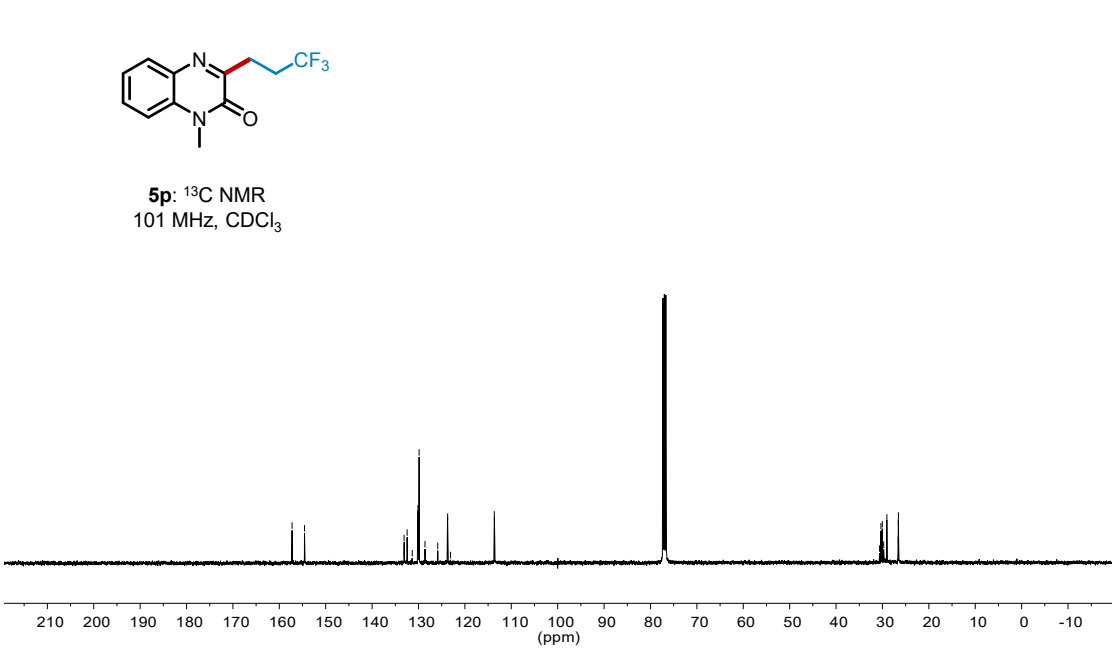
5o: ^{1}H NMR
400 MHz, $CDCl_3$



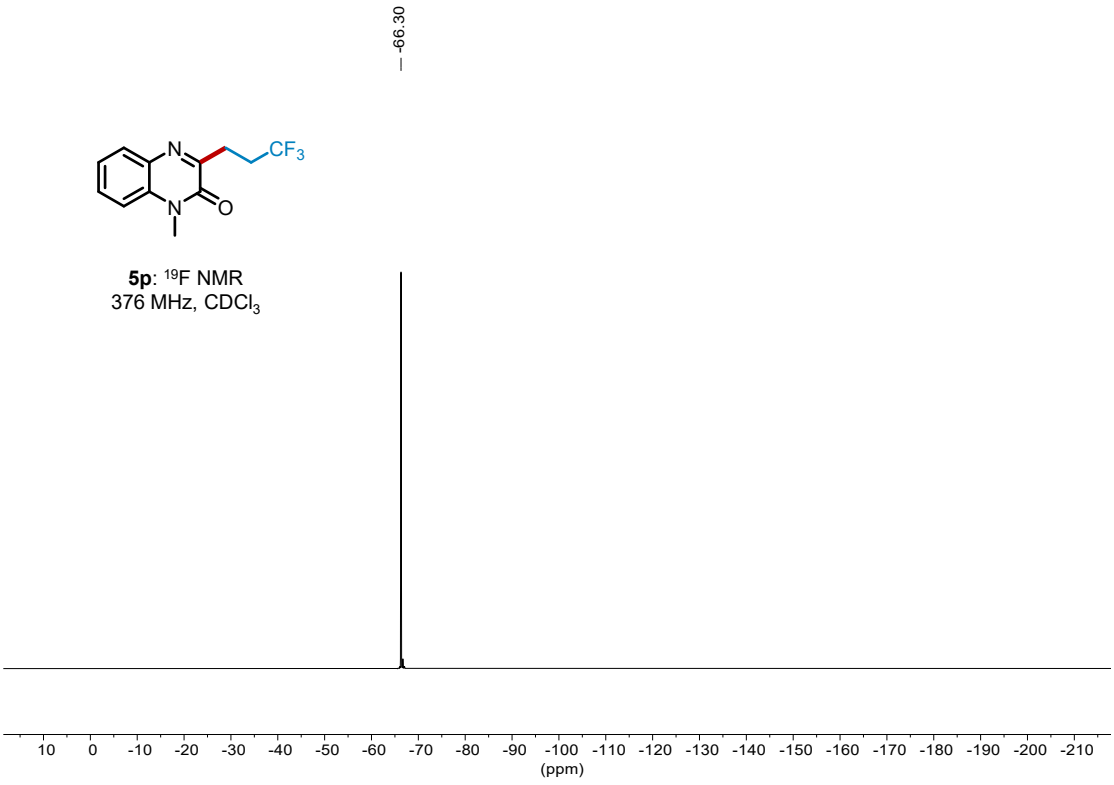
5o: ^{13}C NMR
101 MHz, $CDCl_3$



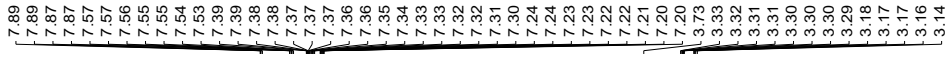
5p: ^{1}H NMR
400 MHz, $CDCl_3$

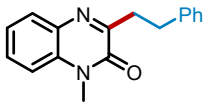


5p: ^{13}C NMR
101 MHz, $CDCl_3$

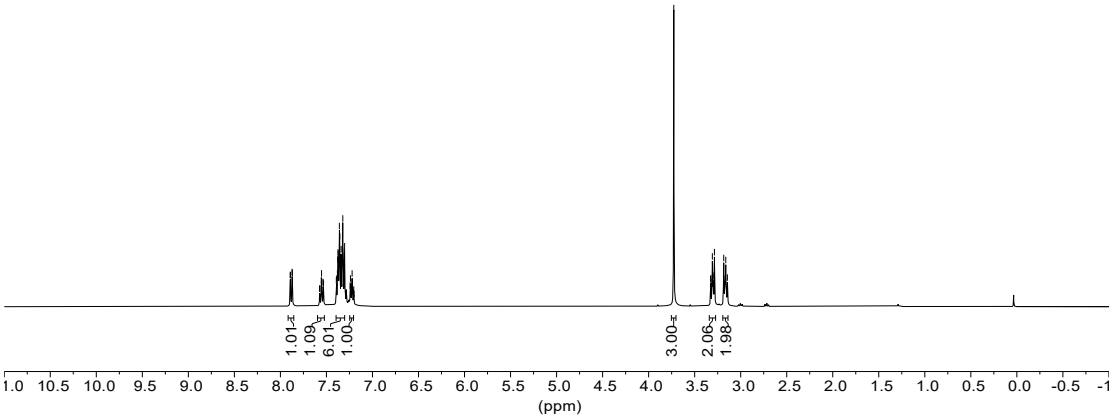


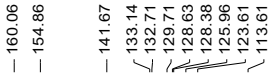
5p: ^{19}F NMR
376 MHz, $CDCl_3$

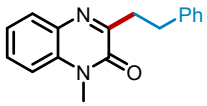




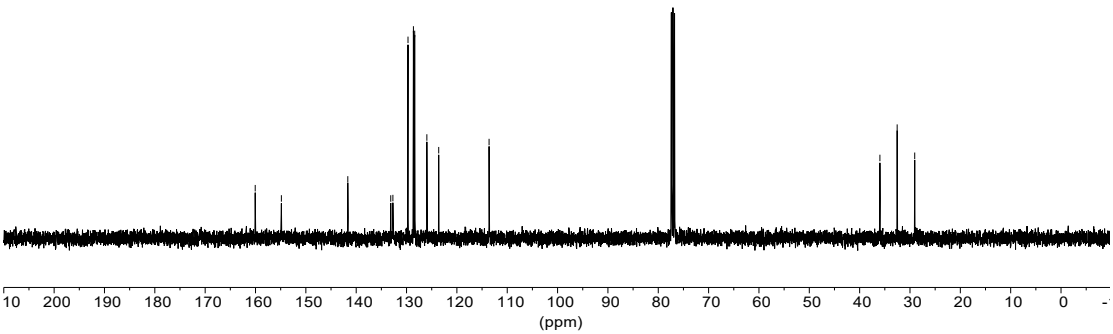
5q: ^{1}H NMR
400 MHz, $CDCl_3$

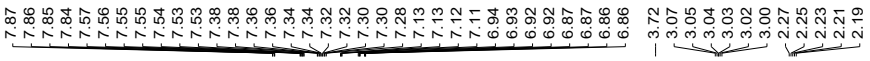


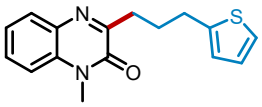




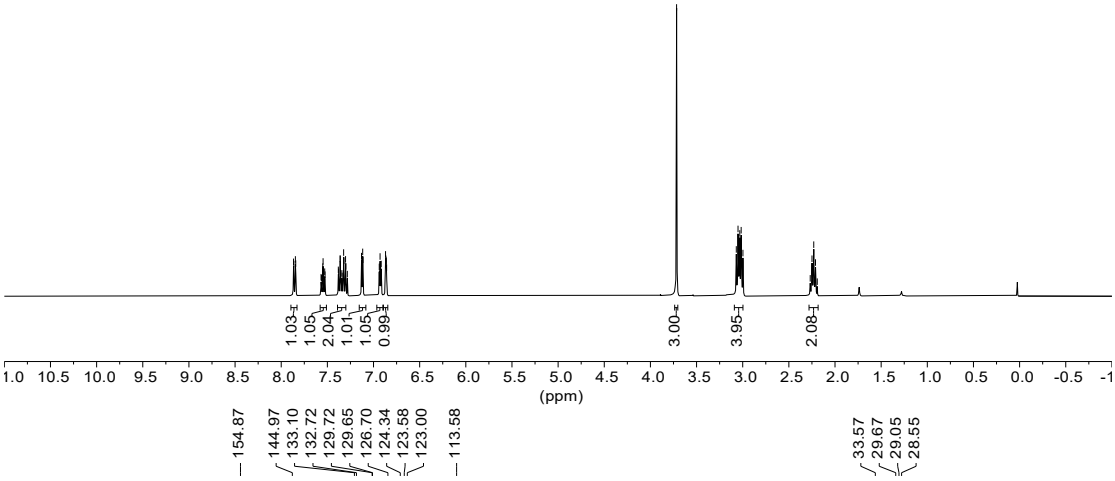
5q: ^{13}C NMR
101 MHz, $CDCl_3$

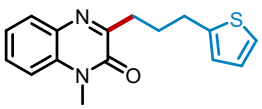




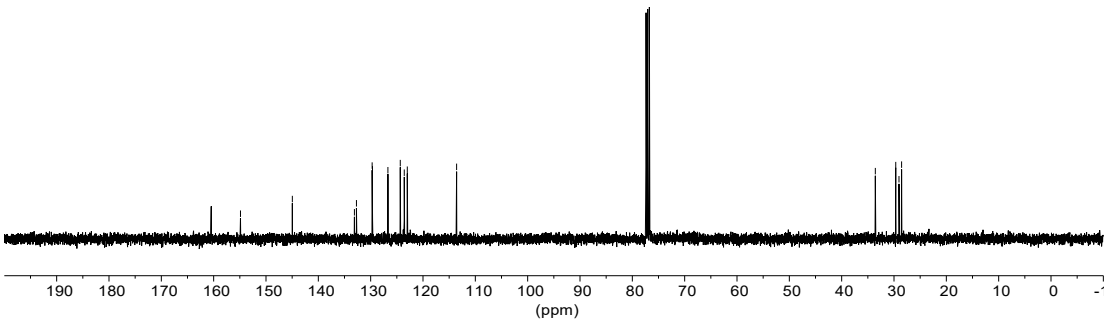


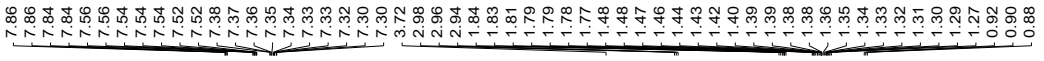
5r: ^{1}H NMR
400 MHz, $CDCl_3$

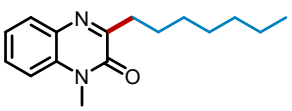




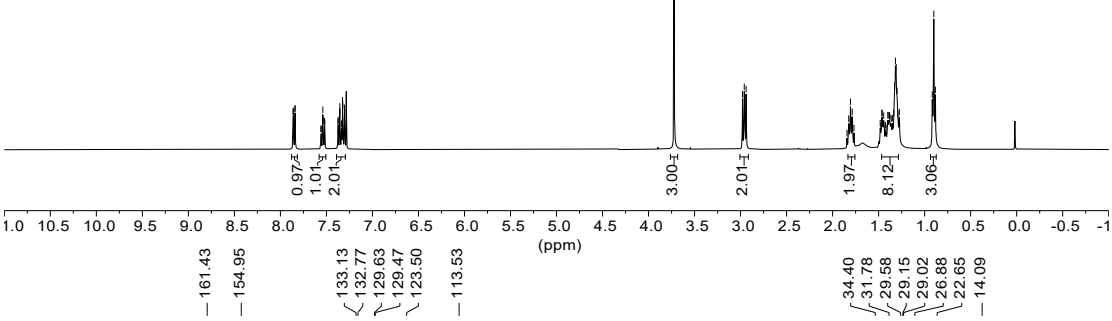
5r: ^{13}C NMR
101 MHz, $CDCl_3$

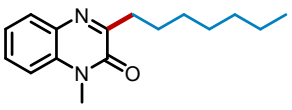




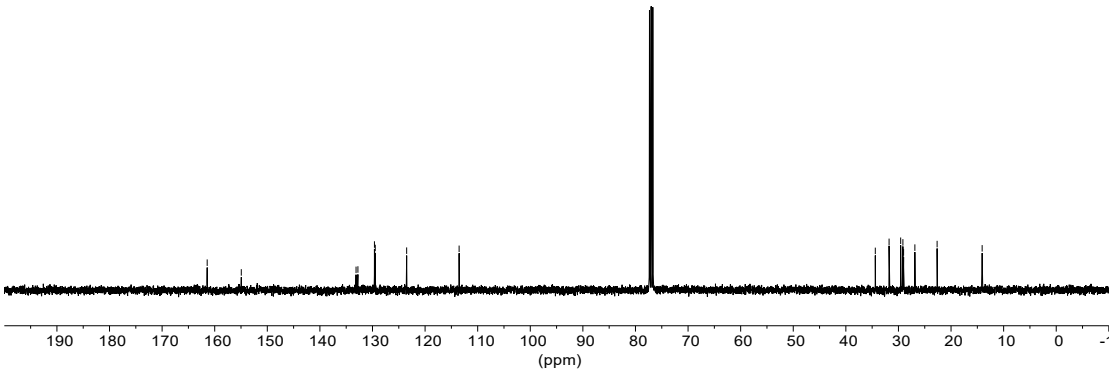


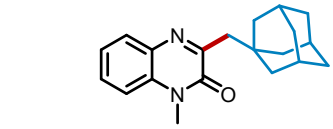
5s: ^{1}H NMR
400 MHz, $CDCl_3$



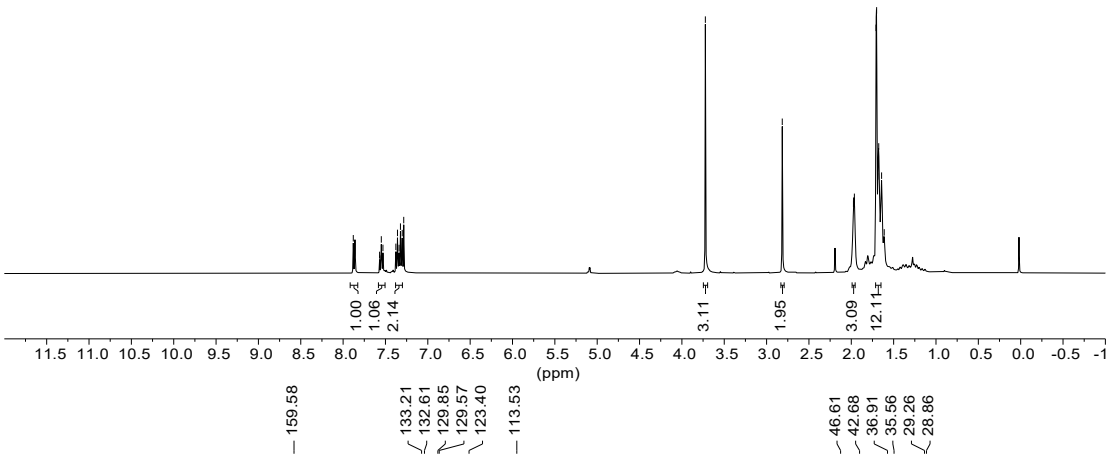


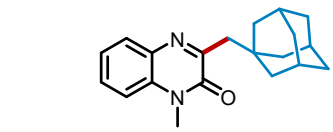
5s: ^{13}C NMR
101 MHz, $CDCl_3$



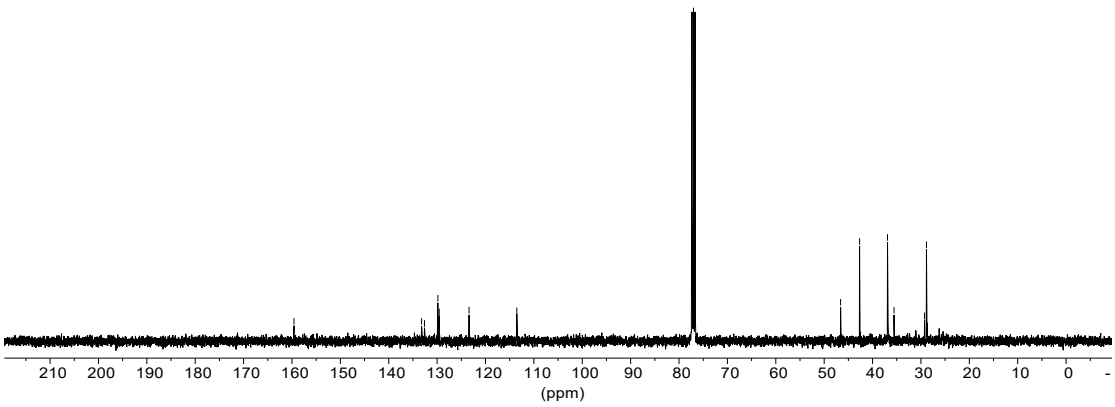


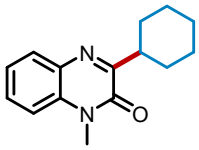
5t: ^{1}H NMR
400 MHz, $CDCl_3$



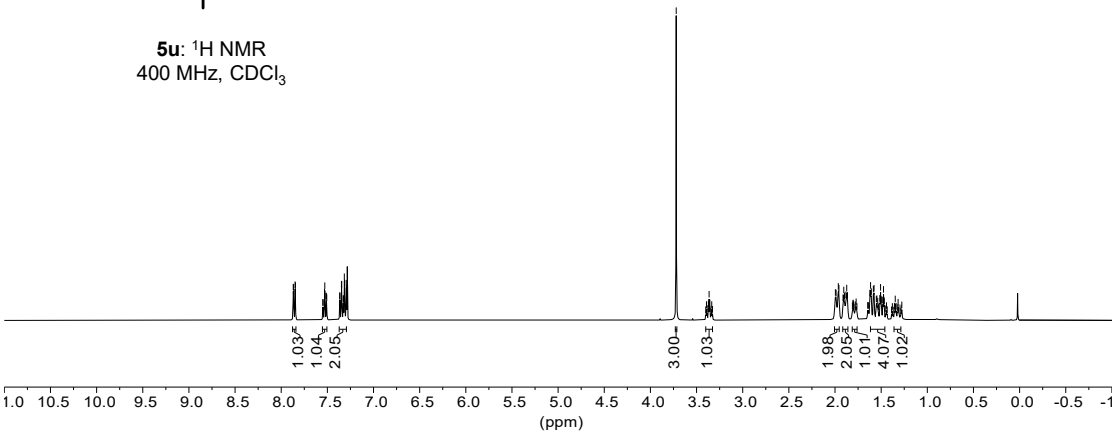


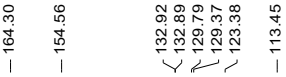
5t: ^{13}C NMR
101 MHz, $CDCl_3$

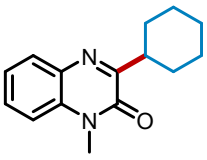




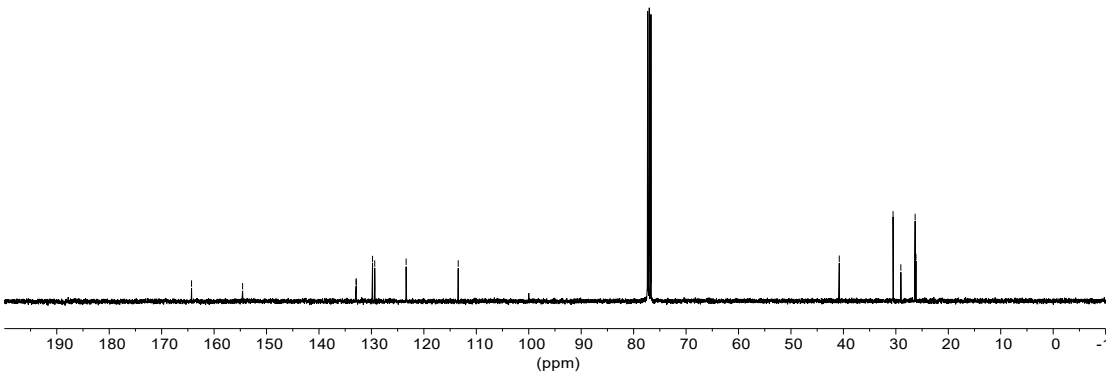
5u: ^{1}H NMR
400 MHz, $CDCl_3$

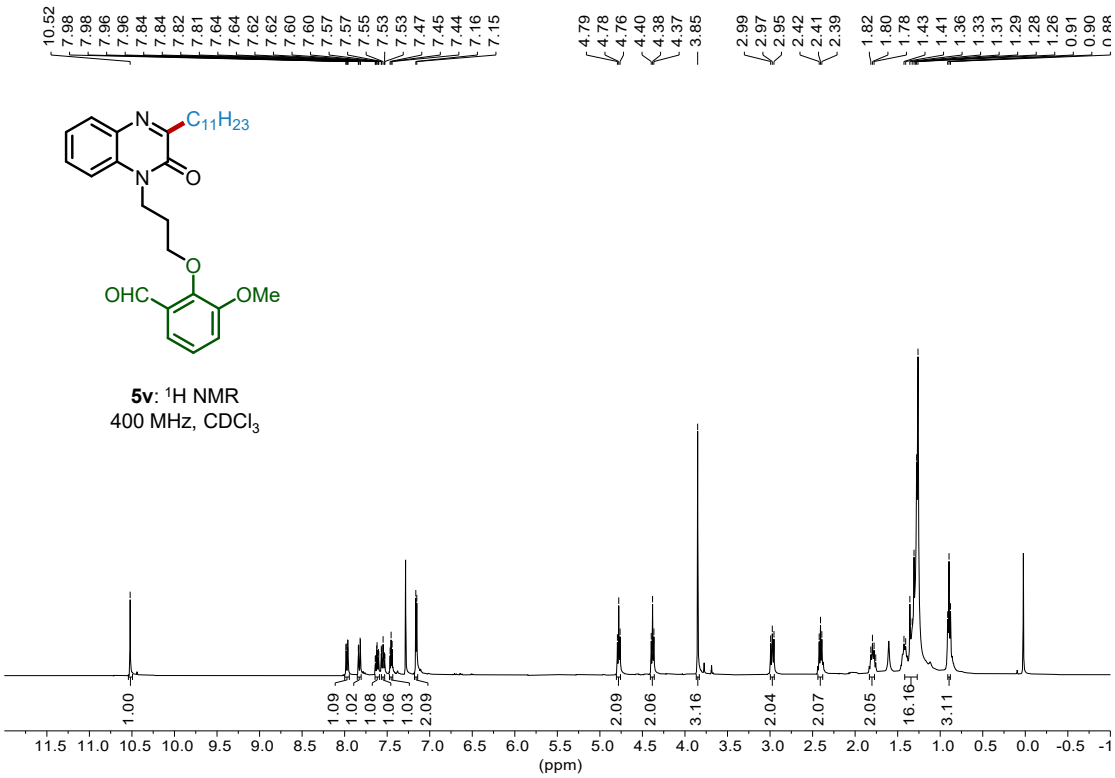




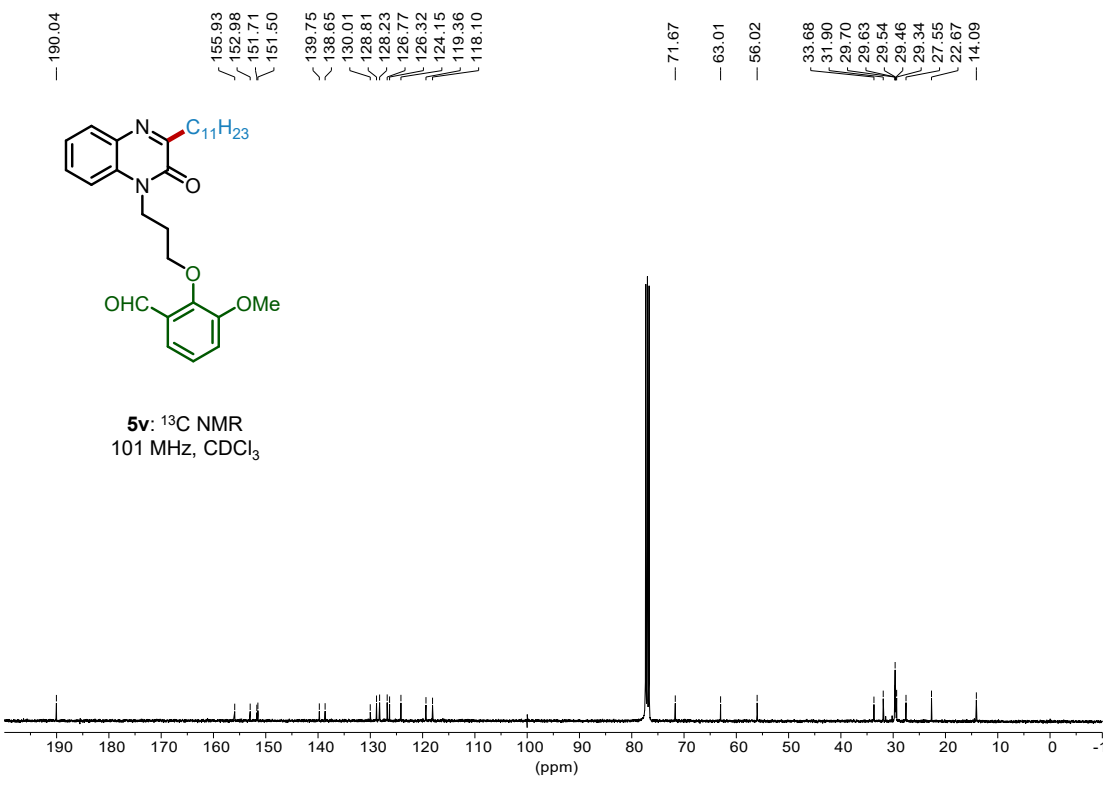


5u: ^{13}C NMR
101 MHz, $CDCl_3$

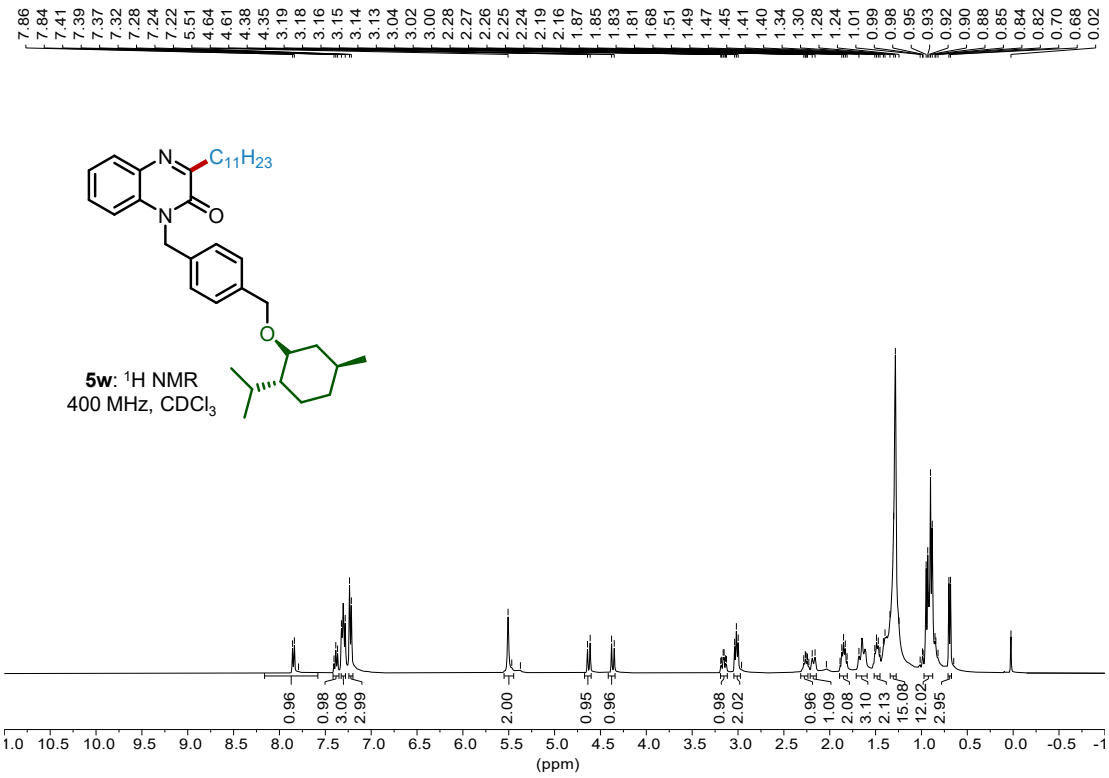


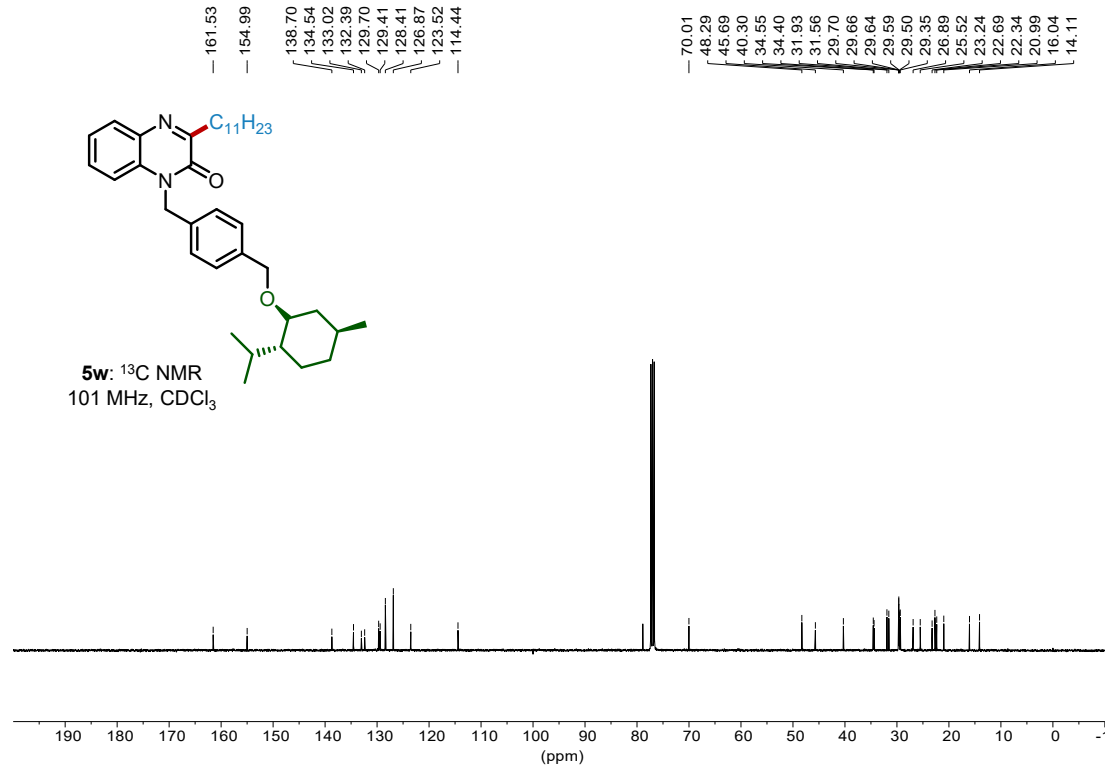


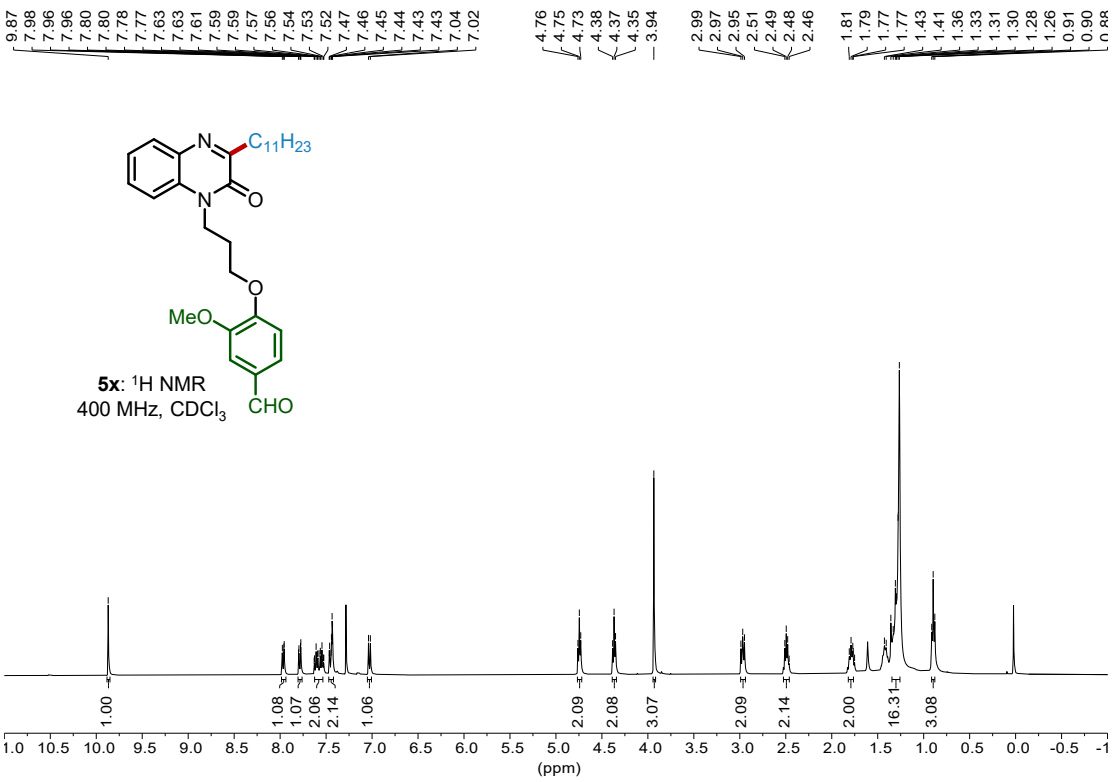
5v: ^{1}H NMR
400 MHz, $CDCl_3$

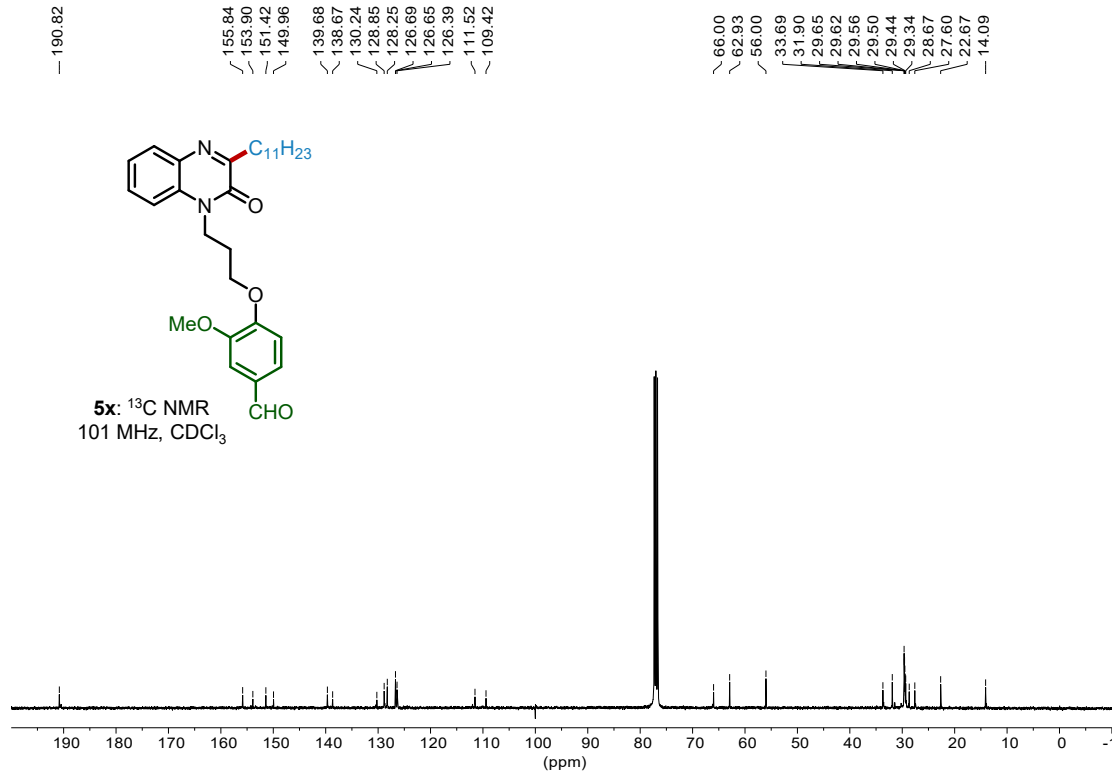


5v: ^{13}C NMR
101 MHz, $CDCl_3$


7.86 7.84 7.41 7.39 7.37 7.32 7.28 7.24 7.22 5.51 4.64 4.61 4.38 4.35 3.19 3.18 3.16 3.15 3.14 3.13 3.04 3.02 3.00 2.28 2.27 2.26 2.25 2.24 2.19 2.16 1.87 1.85 1.83 1.81 1.68 1.51 1.49 1.47 1.45 1.41 1.40 1.34 1.30 1.28 1.24 1.01 0.99 0.98 0.95 0.93 0.92 0.90 0.88 0.85 0.84 0.82 0.70 0.68 0.02
N
C11H23
N
O
O
5w: 1H NMR
400 MHz, CDCl3
0.96 0.98 3.08 2.99 2.00 0.95 0.96 0.98 2.02 0.96 1.09 2.08 3.10 2.13 15.08 12.02 2.95
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
(ppm)


161.53 154.99 138.70 134.54 133.02 132.39 129.70 129.41 128.41 126.87 123.52 114.44
70.01 48.29 45.69 40.30 34.55 34.40 31.93 31.56 29.70 29.66 29.64 29.59 29.50 29.35 26.89 25.52 23.24 22.69 22.34 20.99 16.04 14.11
N
C11H23
N
O
O
5w: 13C NMR
101 MHz, CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)


9.87
7.98
7.96
7.96
7.80
7.80
7.78
7.77
7.63
7.63
7.61
7.59
7.59
7.57
7.56
7.54
7.53
7.52
7.47
7.46
7.45
7.44
7.43
7.43
7.04
7.02
4.76
4.75
4.73
4.38
4.37
4.35
3.94
2.99
2.97
2.95
2.51
2.49
2.48
2.46
1.81
1.79
1.77
1.77
1.43
1.41
1.36
1.33
1.31
1.30
1.28
1.26
0.91
0.90
C11H23
N
N
O
O
MeO
CHO
5x: 1H NMR
400 MHz, CDCl3
1.00
1.08
1.07
2.06
2.14
1.06
2.09
2.08
3.07
2.09
2.14
2.00
16.31
3.08
(ppm)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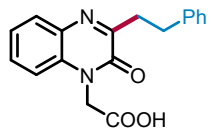
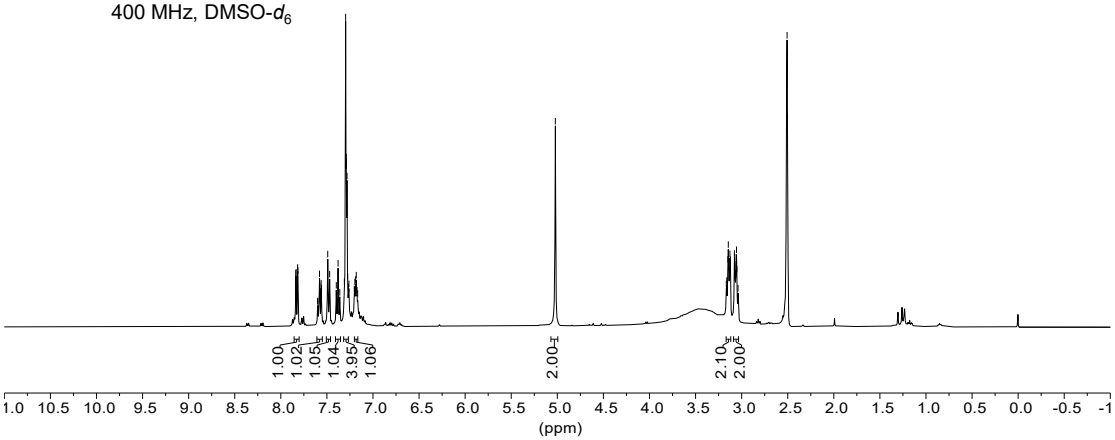
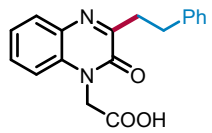
155.84
153.90
151.42
149.96
139.68
138.67
130.24
128.85
128.25
126.69
126.65
126.39
111.52
109.42
66.00
62.93
56.00
33.69
31.90
29.65
29.62
29.56
29.50
29.44
29.34
28.67
27.60
22.67
14.09
C11H23
N
N
O
O
MeO
CHO
5x: 13C NMR
101 MHz, CDCl3
(ppm)

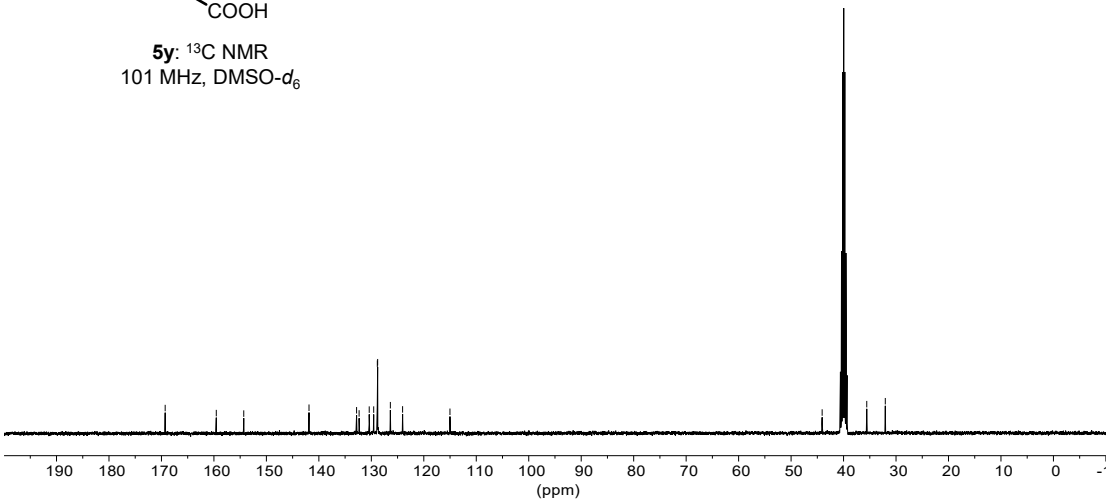


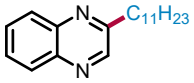
5y: ^{1}H NMR
400 MHz, DMSO-d_6



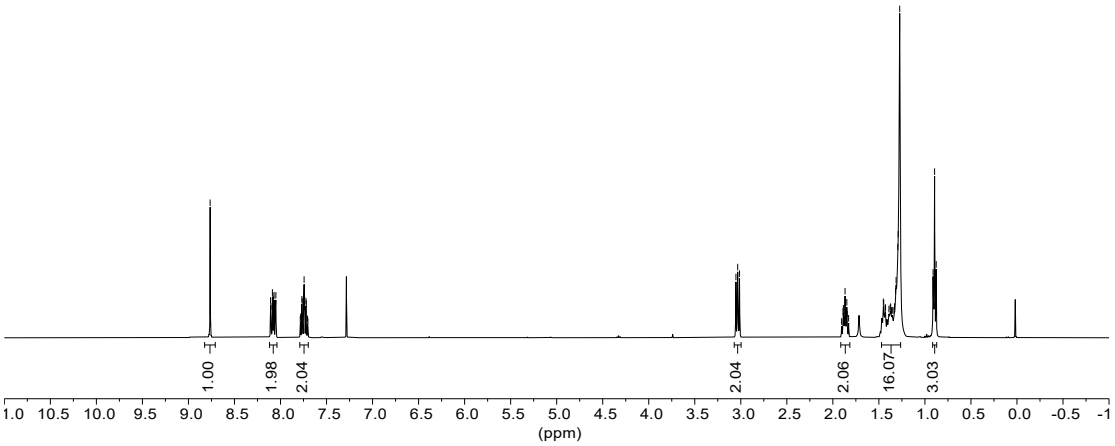


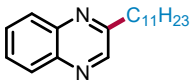
5y: ^{13}C NMR
101 MHz, DMSO-d_6



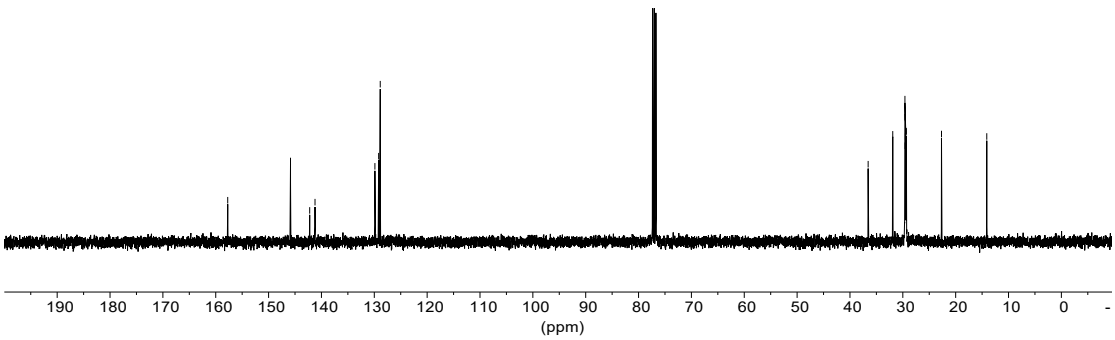


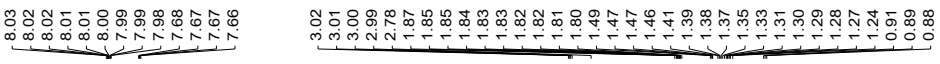
7a: ^{1}H NMR
400 MHz, $CDCl_3$

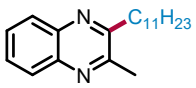




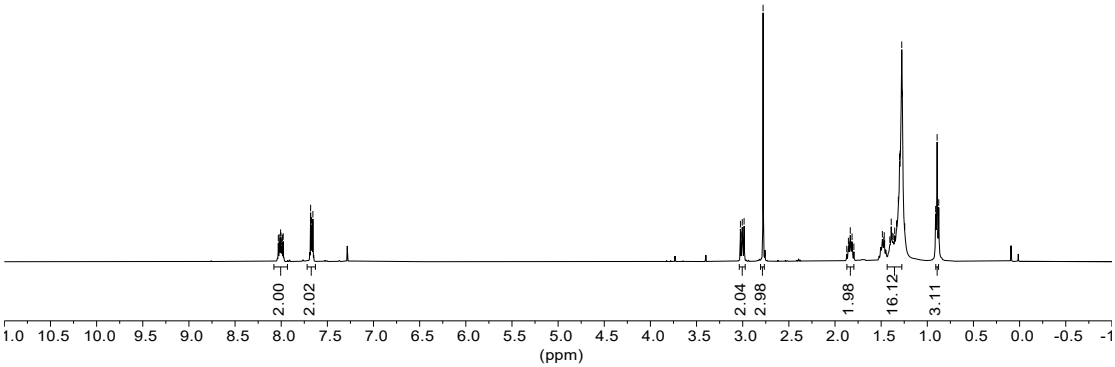
7a: ^{13}C NMR
101 MHz, $CDCl_3$

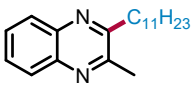




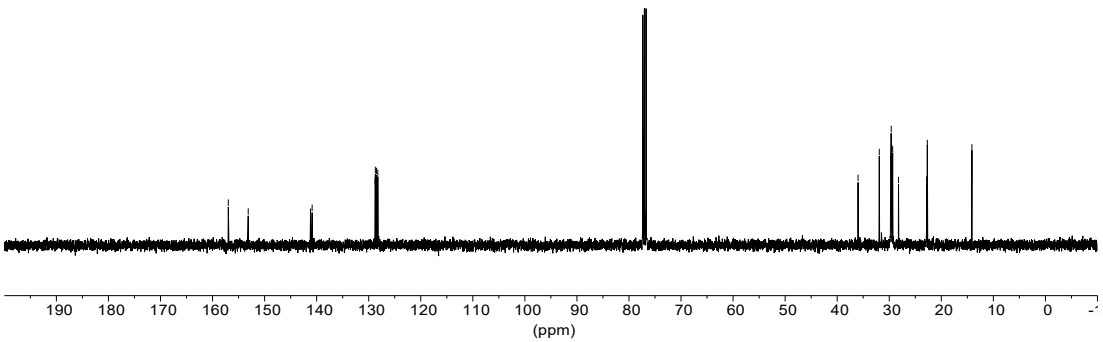


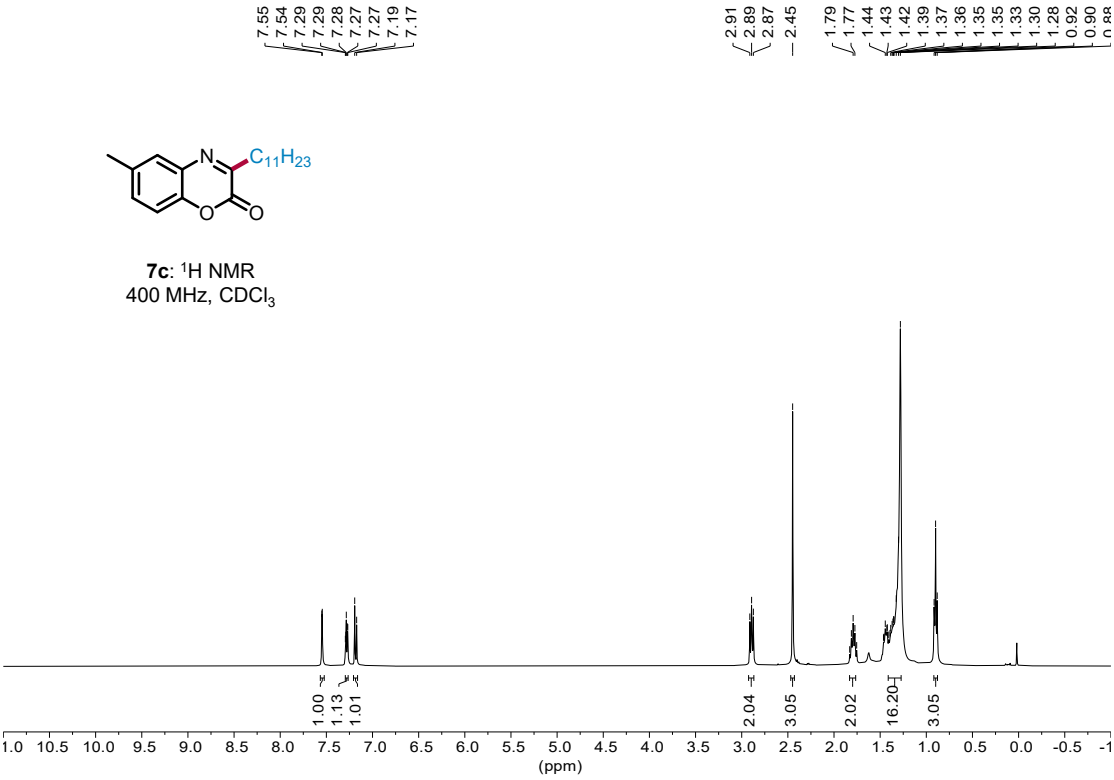
7b: ^{1}H NMR
400 MHz, $CDCl_3$



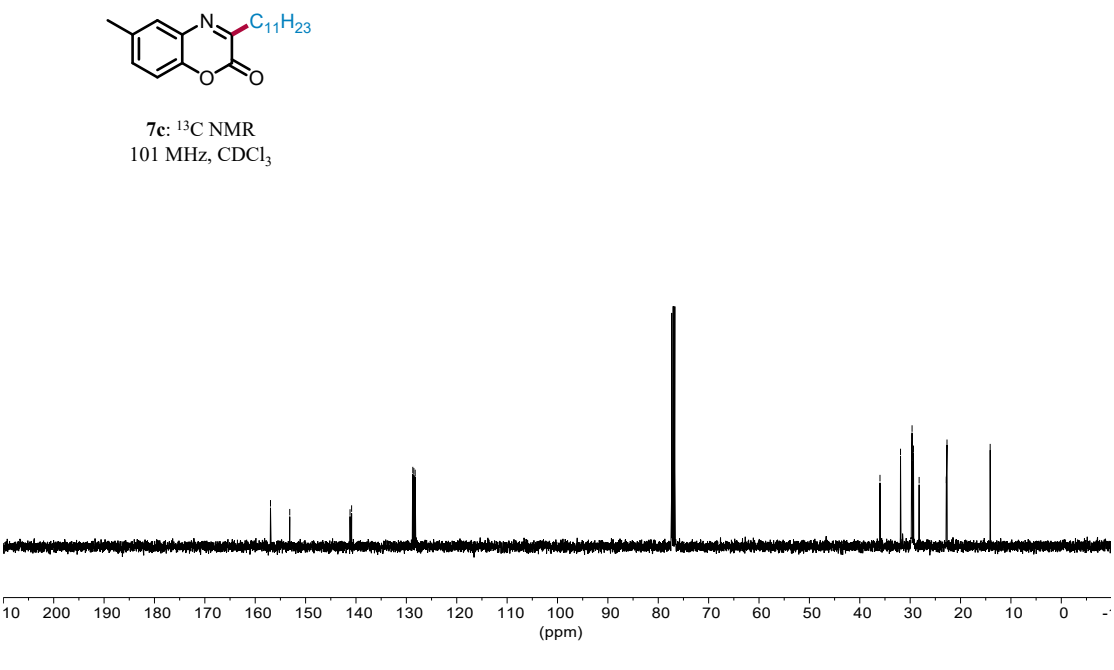


7b: ^{1}H NMR
400 MHz, $CDCl_3$

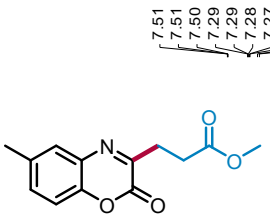




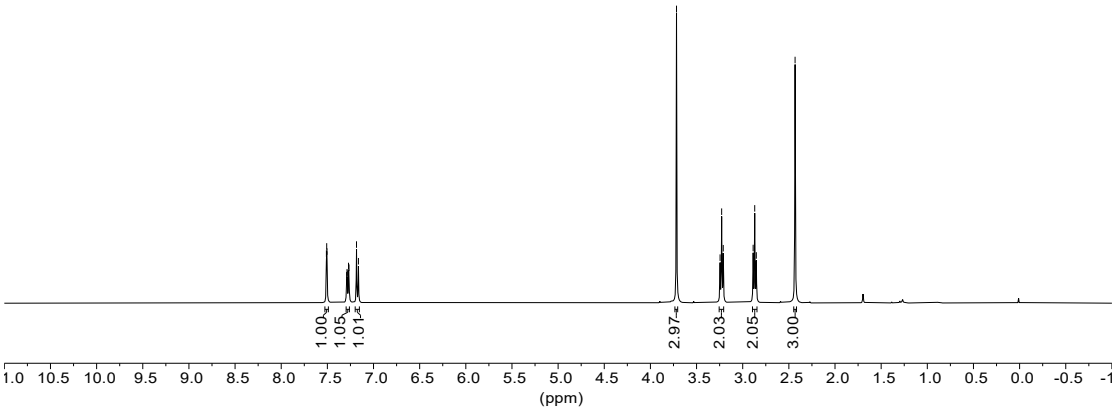
7c: ^{1}H NMR
400 MHz, $CDCl_3$

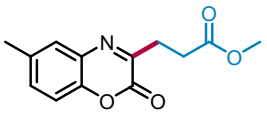


7c: ^{13}C NMR
101 MHz, $CDCl_3$

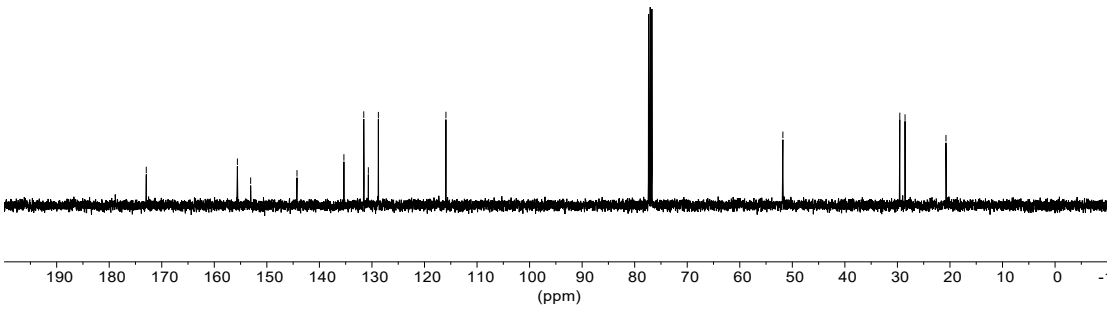


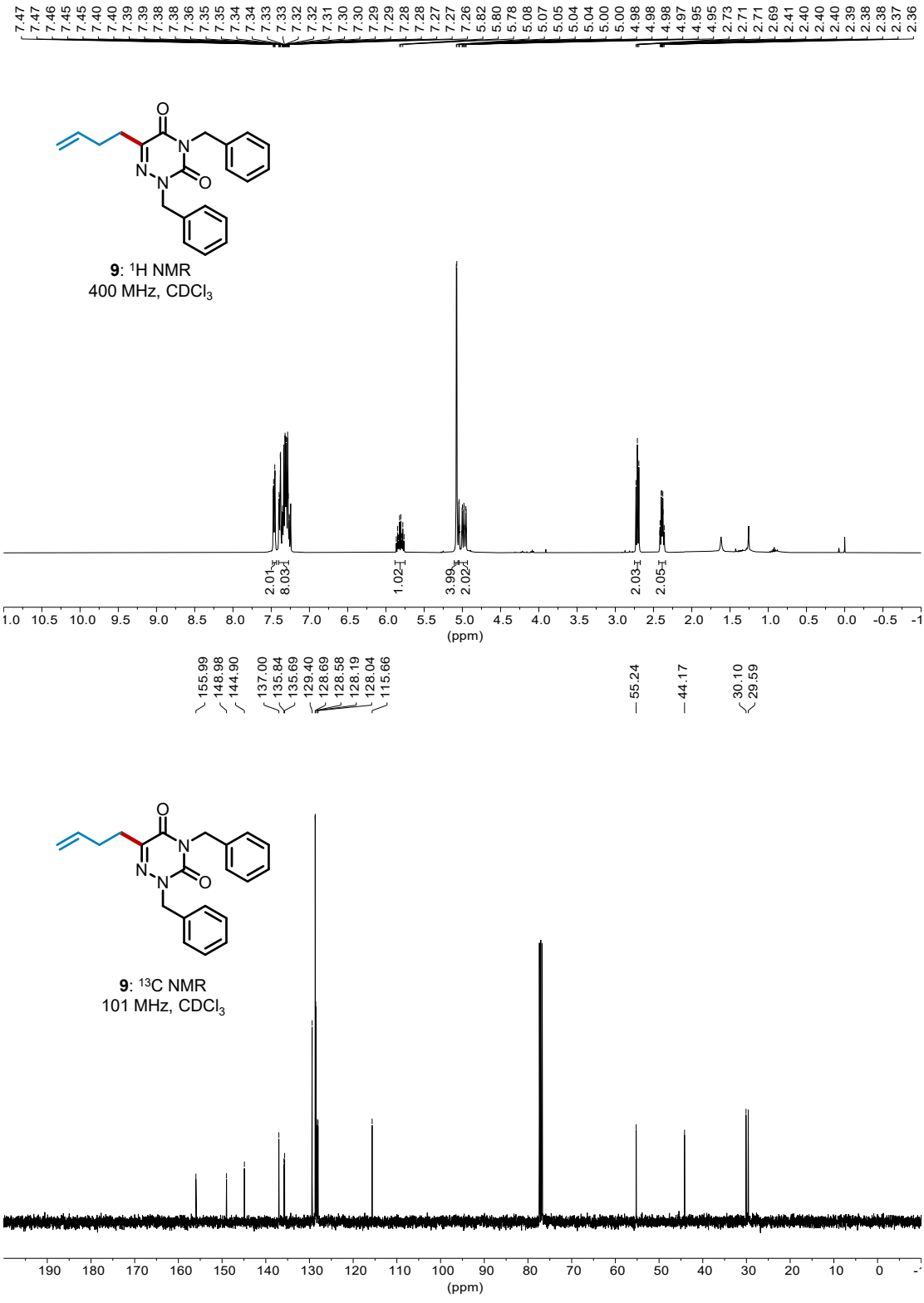
7d: ^{1}H NMR
400 MHz, $CDCl_3$





7d: ^{13}C NMR
101 MHz, $CDCl_3$

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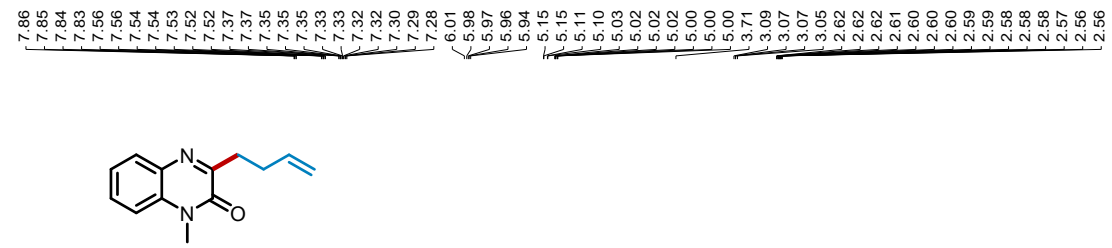
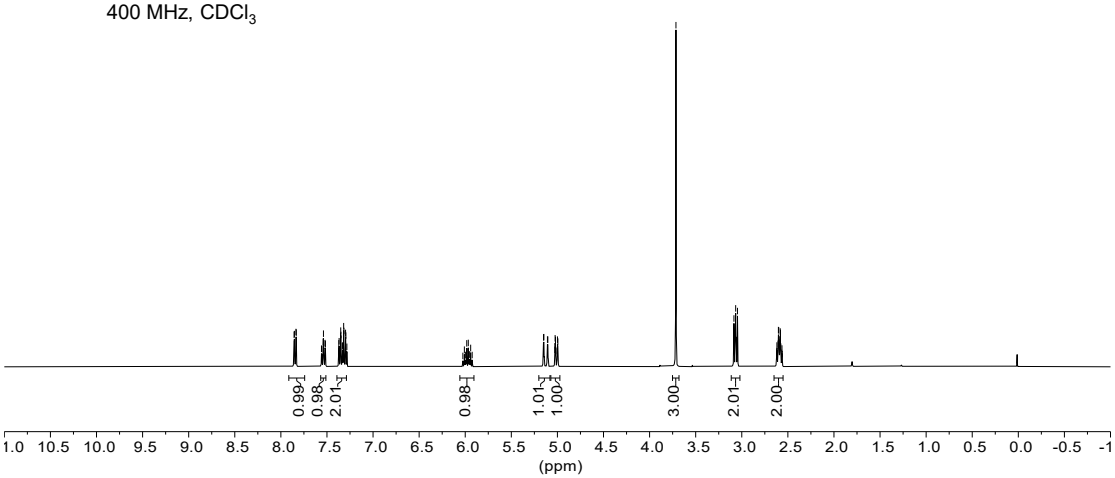
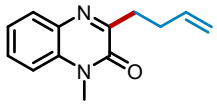
O
N
N
N
O
9: 1H NMR
400 MHz, CDCl3
2.01
8.03
1.02
3.99
2.02
2.03
2.05
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
(ppm)
155.99
148.98
144.90
137.00
135.84
135.69
129.40
128.69
128.58
128.19
128.04
115.66
55.24
44.17
30.10
29.59
O
N
N
N
O
9: 13C NMR
101 MHz, CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -
(ppm)

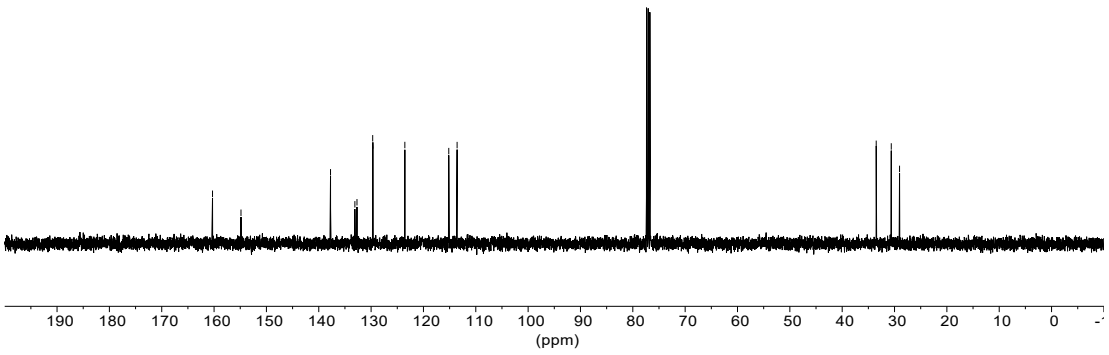


10: ^{1}H NMR
400 MHz, $CDCl_3$





10: ^{13}C NMR
101 MHz, $CDCl_3$



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