# Supporting Information for

## Photochemical Tandem Reaction of Nitrogen Containing Heterocycles, Bicyclo[1.1.1]pentane, and Difluoroiodane(III) reagents

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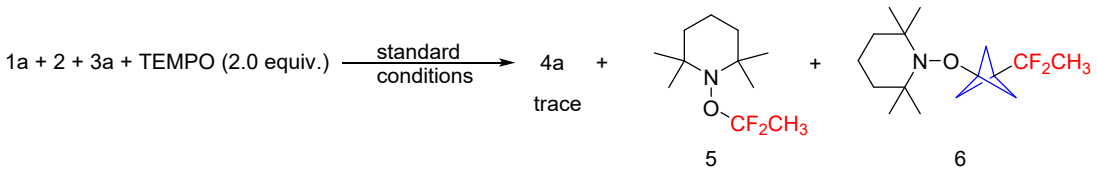
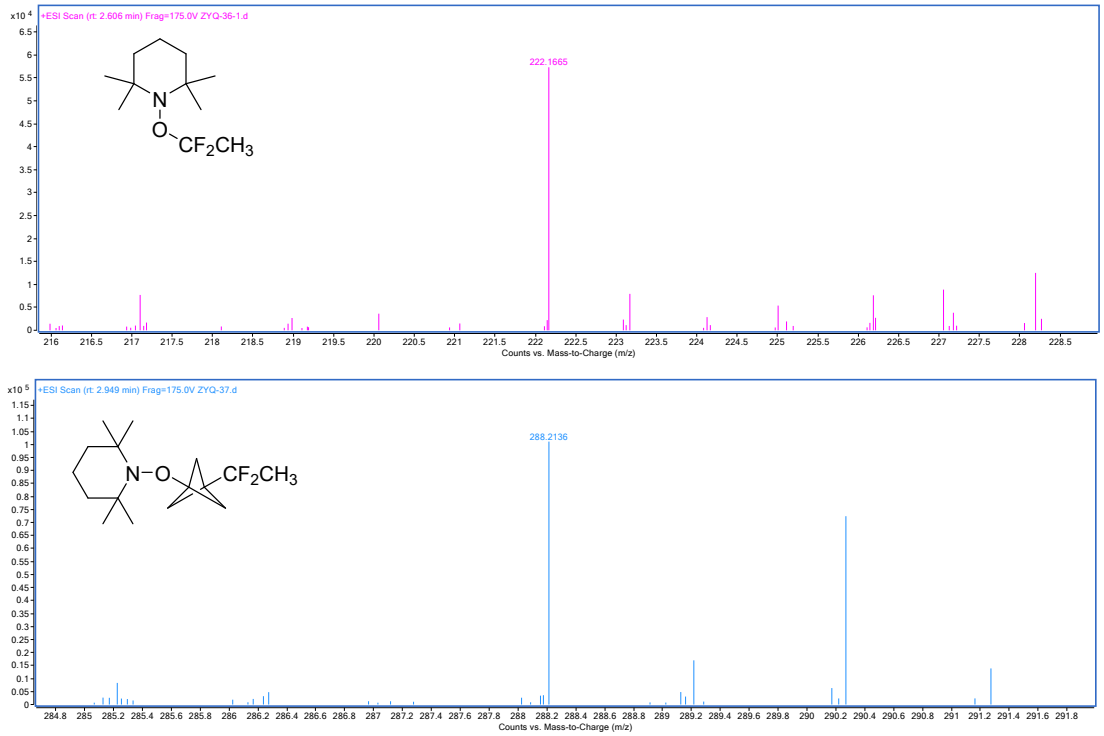
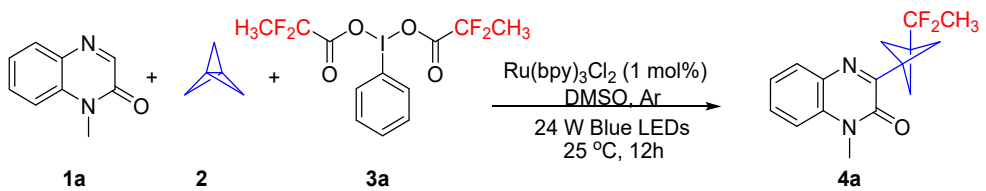
## 1. The radical trapping experiments




To a solution of 1a (0.30 mmol), 2 (0.90 mmol), 3a (0.60 mmol), TEMPO (0.60 mmol) and dry DMSO (1.50 mL). The reaction mixture was evacuated and with pure Ar for three times and stirred under the irradiation of 24 W blue LED at 25 °C for 12 hours. The reaction was completely inhibited, no desired product was obtained and TEMPO-trapped complex 5 (TEMPO-$CF_2CH_3$) and complex 6 (TEMPO-BCP-$CF_2CH_3$) was detected by LC-MS analysis. HRMS ESI (m/z): (5) calcd for 222.1664, found: 222.1665; (6) calcd for 288.2133, found: 288.1236

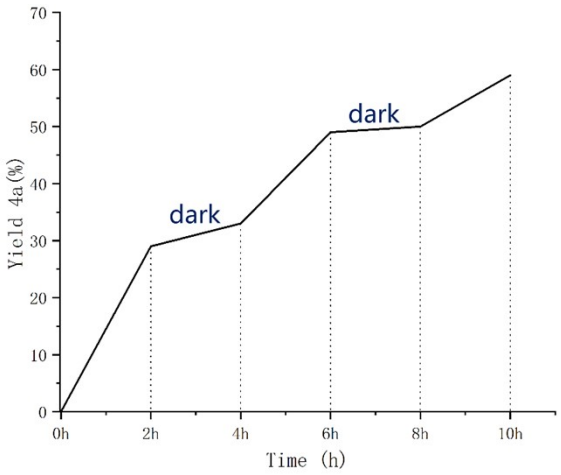



## 2. Light on/off experiment




A frame-dried 10 mL Schlenk tube with a magnetic stirring bar was charged with *N*-methylquinoxalin-2(1*H*)-one (0.30 mmol) (**1a**), phenyl-$\lambda^3$-iodanediyl bis(2,2-

difluoropropanoate) (0.60 mmol) (**3a**), $Ru(bpy)_3Cl_2$ (2 mg, 0.003 mmol) and dry DMSO (1.5 mL) was added via syringe under argon. Then [1.1.1]propellane (1.983 g, 0.90 mmol, 3% in mixed solution of diethoxymethane and n-pentane) (**2**) was added and the reaction mixture was performed using alternating intervals of light and dark with a 24 W blue LEDs (460 nm) at 25 ℃ for 10 hours under argon. The yield of product was determined by $^1H$ NMR (1,3,5-Trimethoxybenzene was used as an internal standard). The reaction profile is shown below (Supplementary figure 1) and indicates that continuous light irradiation is essential to promote the reaction.



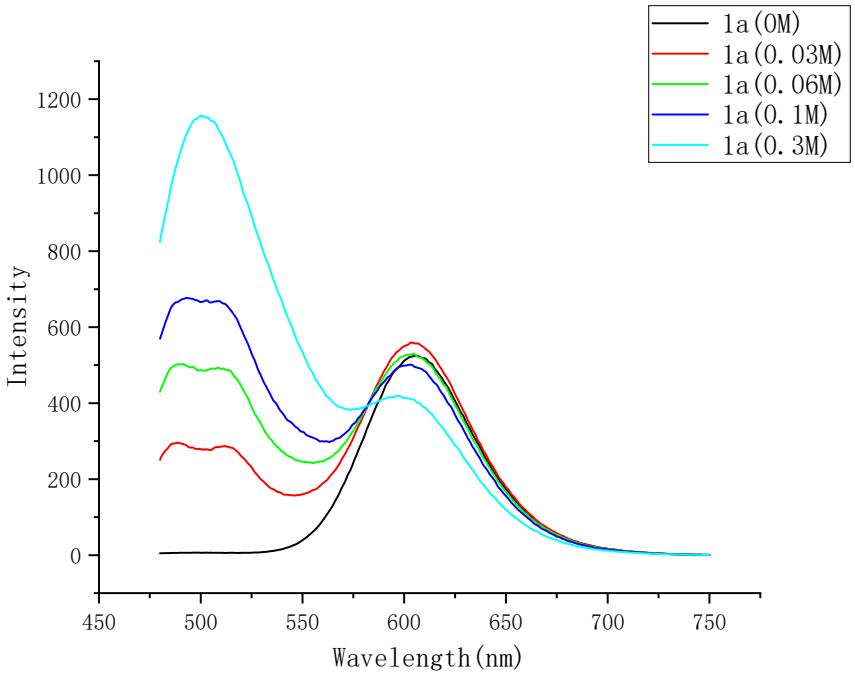

**Supplementary figure 1**: Light on/off experiment.

### 3. Stern-Volmer Luminescence Quenching Analysis

Emission intensities were recorded using an F-75700 FL Spectrophotometer. First, the emission intensity of $Ru(bpy)_3Cl_2$ solutions was observed at 605 nm. The solutions were irradiated at 405 nm (Maximum absorption wavelength of $Ru(bpy)_3Cl_2$) and fluorescence was measured from 450 nm to 750 nm. The solution of $Ru(bpy)_3Cl_2$ (1 mM, 10 mL), **1a** (1 M, 10 mL), **2a** (0.24 M, 10 mL) and **3a** (1 M, 10 mL) were prepared in glovebox.

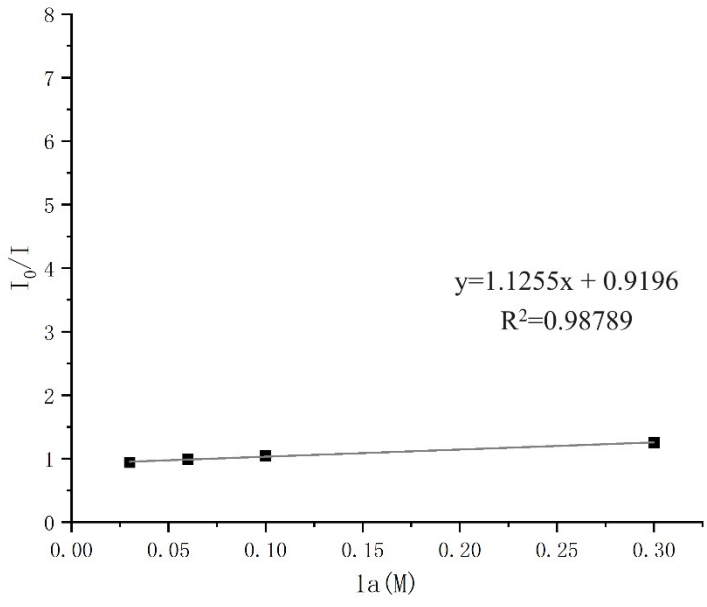
**For Experiment 1:** Constant photocatalyst; Varied quinoxalin-2(1*H*)-ones.

Add 200 μL $Ru(bpy)_3Cl_2$ solution and 0 μL, 20 μL, 60 μL, 120 μL, 200 μL **1a** solution respectively in the quartz cuvette, then diluted the solution to 2 mL.




**Supplementary figure 2**: Fluorescence spectrum of PC + 1a.

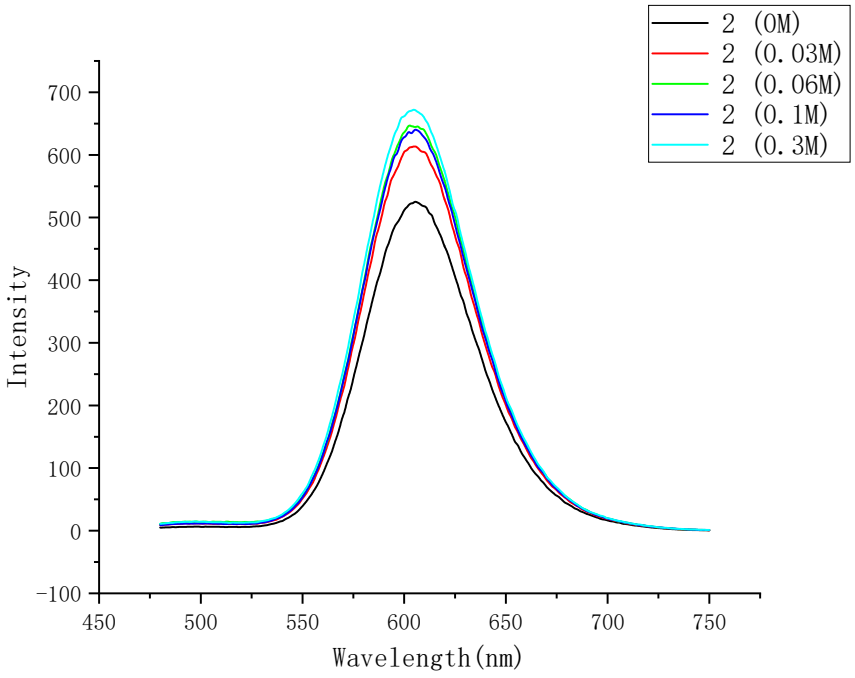
**Comment: 1a** does not react with the excited photocatalyst.




**Supplementary figure 3**: Stern-Volmer Luminescence Quenching Analysis of PC + 1a.

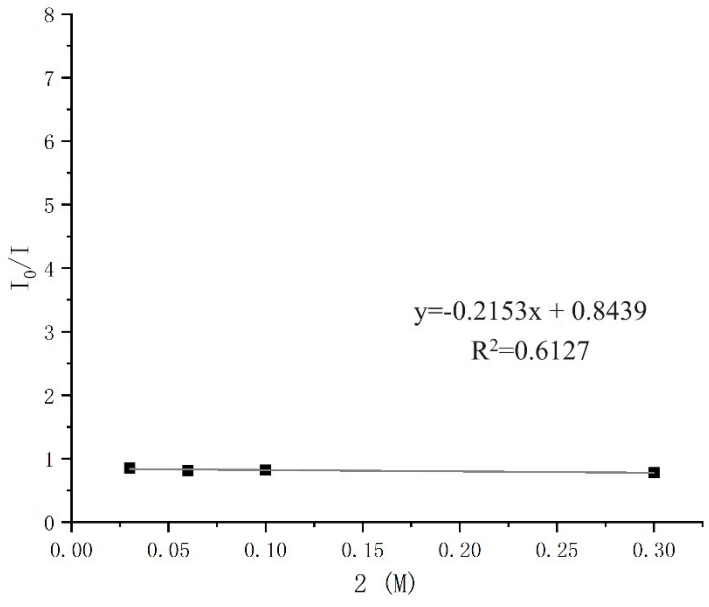
**For Experiment 2:** Constant photocatalyst; Varied [1.1.1]propellane.

Add 200 µL $Ru(bpy)_3Cl_2$ solution and 0 µL, 250 µL, 500 µL, 830 µL, 1670 µL **2** solution respectively in the quartz cuvette, then diluted the solution to 2 mL.




**Supplementary figure 4**: Fluorescence spectrum of PC + 2.

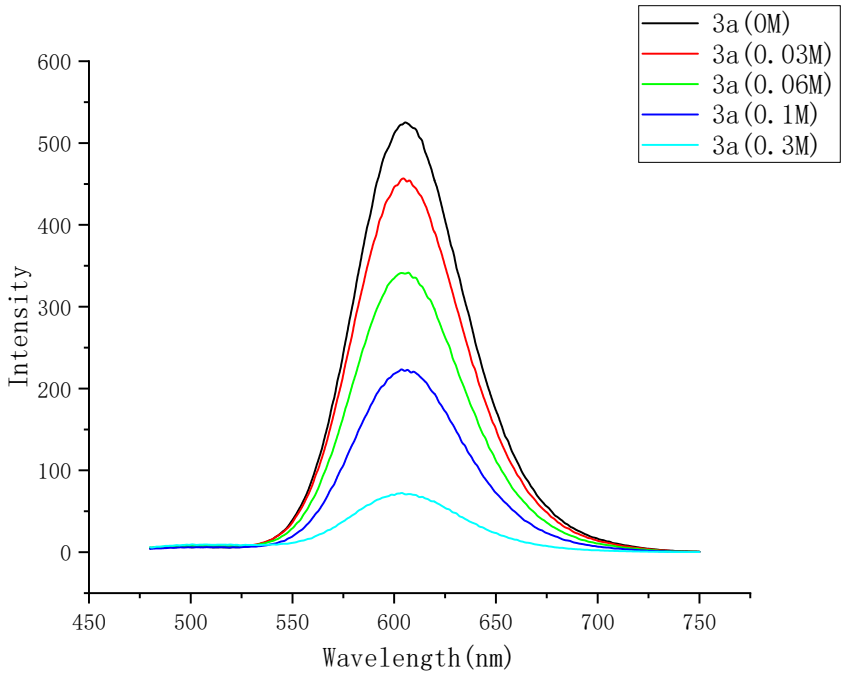
**Comment:** [1.1.1]propellane does not react with the excited photocatalyst.




**Supplementary figure 5**: Stern-Volmer Luminescence Quenching Analysis of PC + 2.

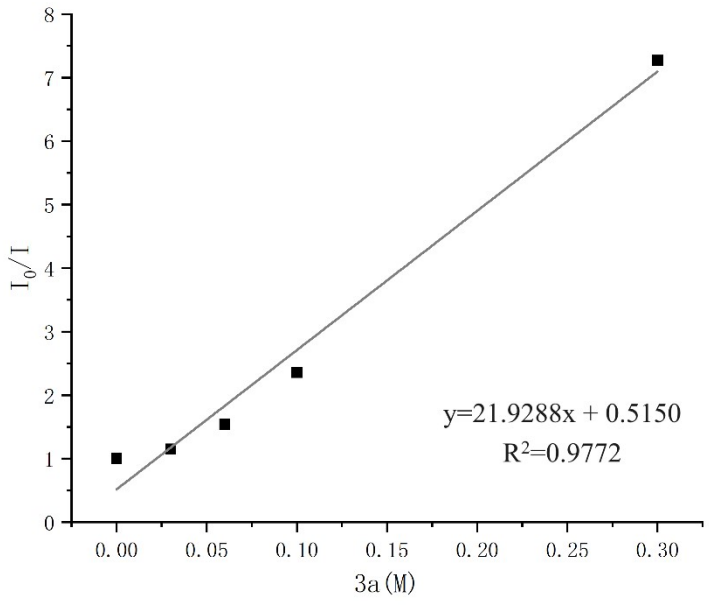
**For Experiment 3:** Constant photocatalyst; Varied hypervalent iodine(III) reagents.

Add 200 μL $Ru(bpy)_3Cl_2$ solution and 0 μL, 20 μL, 60 μL, 120 μL, 200 μL **3a** solution respectively in the quartz cuvette, then diluted the solution to 2 mL.




**Supplementary figure 6**: Fluorescence spectrum of PC + 3a.

**Comment:** Only **3a** could quench the photocatalyst.

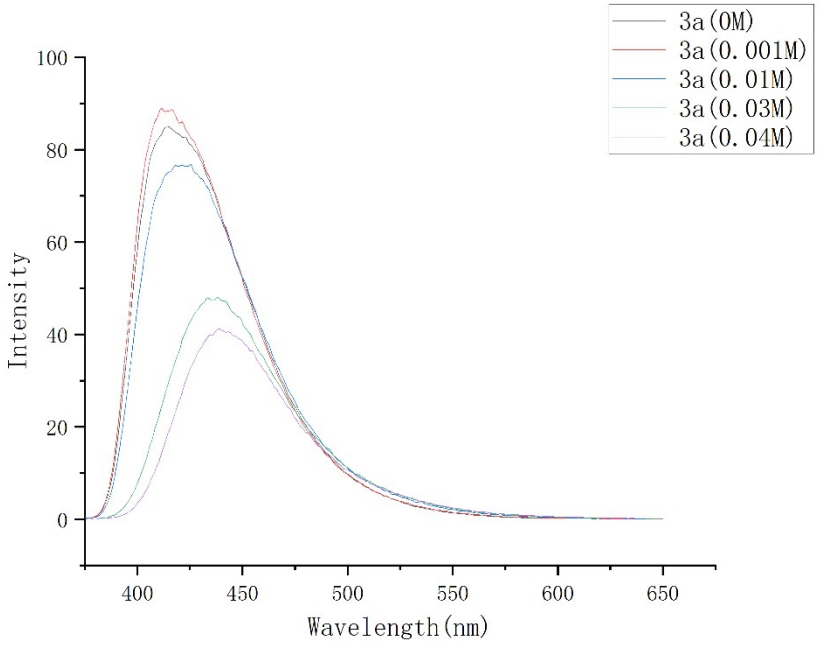



**Supplementary figure 7**: Stern-Volmer Luminescence Quenching Analysis of PC + 3a.

Fluorescence quenching experiment without photocatalyst. First, the emission intensity of **1a** solutions was observed at 414 nm. The solutions were irradiated at 364 nm (Maximum absorption wavelength of **1a**) and fluorescence was measured from 375 nm to 650 nm. The

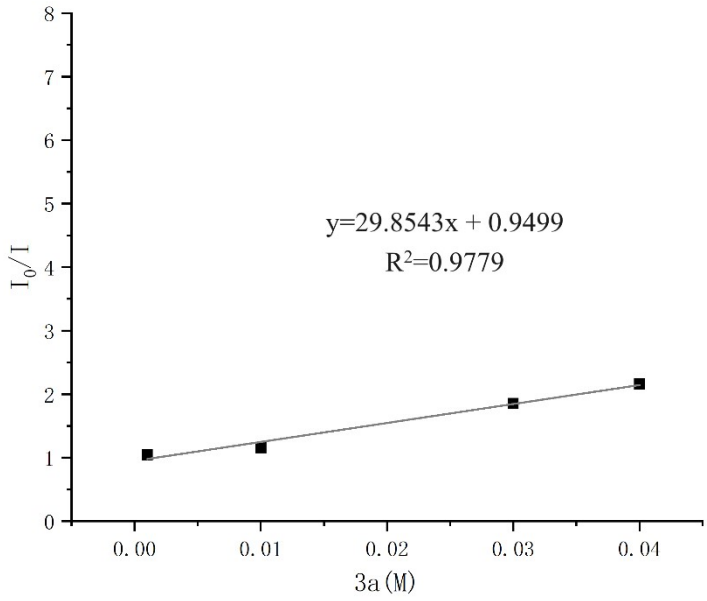
solution of **1a** (1 mM, 10 mL) and **3a** (100 mM, 10 mL) were prepared in glovebox.

**For Experiment 4:** Constant quinoxalin-2(1*H*)-ones; Varied hypervalent iodine(III) reagents. Add 200 μL **1a** solution and 0 μL, 20 μL, 200 μL, 600 μL, 800 μL **3a** solution respectively in the quartz cuvette, then diluted the solution to 2 mL.




**Supplementary figure 8**: Fluorescence spectrum of 1a + 3a.

**Comment: 3a** could quench **1a**.



**Supplementary figure 9**: Stern-Volmer Luminescence Quenching Analysis of 1a + 3a.

## 4. Quantum Yield Experiment

The quantum yield of the reaction is defined as[1]:

$$\Phi(reaction\ at\ 460nm) = \frac{mol\ of\ formed\ product}{mol\ of\ photon\ flux \cdot t \cdot f} \quad (1)$$

Standard ferrioxalate actinometry was used to determine the photon flux of the spectrophotometer using equations 2, 3 and 4. The moles of $Fe^{2+}$ formed are determined spectrophotometrically by development with 1,10phenanthroline (phen) to form the red $[Fe(phen)_3]^{2+}$ moiety ($\lambda$ = 510 nm).[2] The photon flux is defined as shown in equation 2:

$$Photon\ flux = \frac{mol\ Fe^{2+}}{\Phi(Fe^{2+}) \cdot t \cdot f} \quad (2)$$

$$F = 1 - 10^{-A} \quad (3)$$

where $\Phi$ is the quantum yield for the ferrioxalate actinometer (0.845 at $\lambda$ = 460 nm), [3]t is the time (s), f is the fraction of light absorbed at $\lambda$ = 460 nm, and the mol of $Fe^{2+}$ are calculated according to equation 4.

$$mol\ Fe^{2+} = \frac{V \cdot \Delta A}{l \cdot \varepsilon} \quad (4)$$

where V is the total volume of the solution, $\Delta$A is the difference in absorbance between irradiated and nonirradiated solutions, l is the path length (1.0 cm), and $\varepsilon$ is the molar absorptivity at 510 nm (11110 L $mol^{-1}$ $cm^{-1}$).

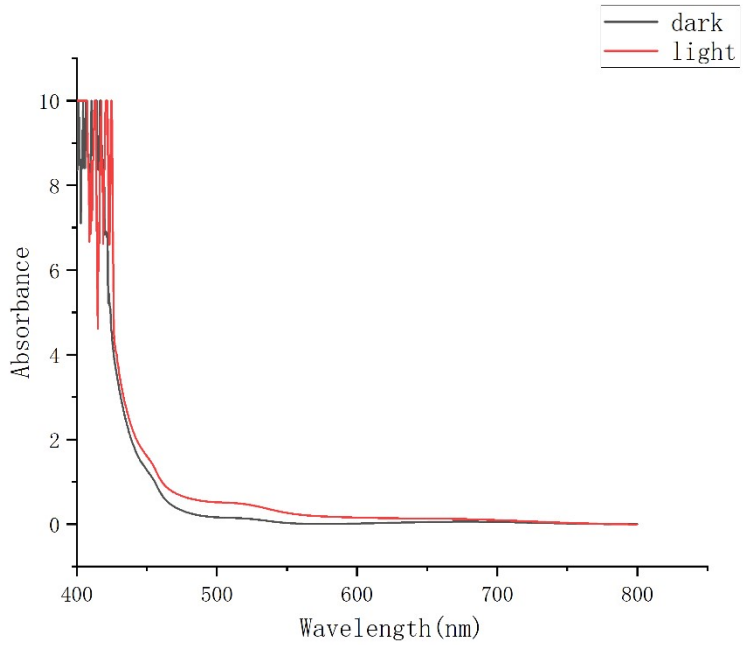
The following solutions were prepared in the dark (flasks were wrapped in aluminum foil) and stored in the dark at room temperature:

**Ferrioxalate solution (0.15 M):** Potassium ferrioxalate hydrate (2.211 g) was added to a flask wrapped in aluminum foil containing $H_2SO_4$ (30 mL, 0.05 M). The flask was stirred for complete solvation of the green solid in complete darkness. It is noteworthy that the solution should not be exposed to any incident light.

**Preparation of buffered solution:** 1,10-Phenanthroline (50 mg) and NaOAc (11.25 g) was added to a flask containing $H_2SO_4$ (50 mL, 0.5 M) and sonicated until completely solvated. The absorbance of the non-irradiated sample.

The buffered solution of phen (700 µL) was added to a ferrioxalate solution (4.0 mL) in a vial that had been covered with aluminum foil and with the lights of the laboratory switched off.

The vial was capped and allowed to rest for 1 h and then transferred to a cuvette. The absorbance of the non-irradiated solution was measured at 510 nm.




**Supplementary figure 10**: Ultraviolet spectroscopic data

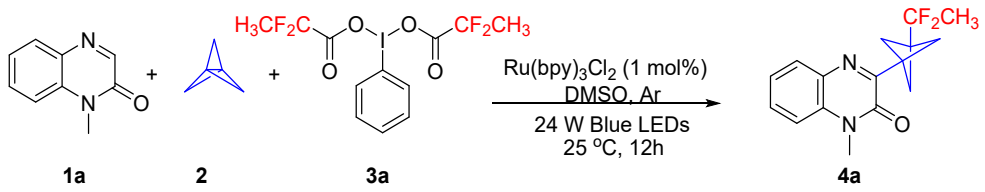
The absorbance of the irradiated sample. In a cuvette equipped with a stir bar was added the ferrioxalate solution (4.0 mL), and the stirred solution was irradiated for 90 s at λ = 460 nm with an excitation slit width = 10.0 nm. After irradiation, the buffered phen solution (700 µL) was added to the cuvette and allowed to rest for 1 h in the dark to allow the ferrous ions to coordinate completely to phen. The absorbance was measured at 510 nm.

$$mol\ Fe^{2+} = \frac{V \cdot \Delta A}{l \cdot \varepsilon} \quad (4)$$

$$mol\ Fe^{2+} = \frac{0.00470\ L \cdot 0.236}{1.0\ cm \cdot 11100\ L \cdot mol^{-1}\ cm^{-1}} = 9.99 \times 10^{-8}\ mol$$

$$Photon\ flux = \frac{mol\ Fe^{2+}}{\Phi(Fe^{2+}) \cdot t \cdot f} \quad (2)$$

$$Photon\ flux = \frac{9.99 \times 10^{-8}\ mol}{0.845 \cdot 90s \cdot 0.294} = 4.468 \times 10^{-8}\ einstein\ s^{-1}$$




**1a** **2** **3a** **4a**

The photoredox transformation was developed using the general procedure by blue LED (λmax = 460 nm) for 2 h (7200 s). The yield of product was determined by $^1$H NMR analysis (1,3,5-Trimethoxybenzene was used as an internal standard). The yield of 4a was determined to be 29% ($8.7\times10^{-5}$ mol). The reaction quantum yield (Φ) was determined using equation 1.

$$\Phi(reaction\ at\ 460nm) = \frac{mol\ of\ formed\ product}{mol\ of\ photon\ flux \cdot t \cdot f} \quad (1)$$

$$\Phi(reaction\ at\ 460nm) = \frac{8.7\ \times\ 10^{-5}\ mol}{4.468\ \times\ 10^{-8}\ einstein\ s^{-1} \cdot 7200\ s \cdot 1} = 0.27$$

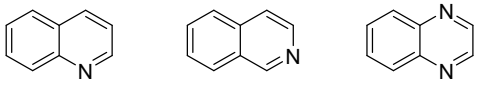
The reaction quantum yield (Φ) was thus determined to be 0.27.

**5. Larger-Scale Experiments**

A frame-dried 200 mL Schlenk flask with a magnetic stirring bar was charged with *N*-methylquinoxalin-2(1*H*)-one (**1a**) (0.80 g, 5.0 mmol), phenyl-λ$^3$-iodanediyl bis(2,2-difluoropropanoate) (**3a**) (4.22 g, 10.0 mmol), $Ru(bpy)_3Cl_2$ (32 mg, 0.05 mmol) and dry DMSO (25.0 mL) was added via syringe under argon. Then [1.1.1]propellane (33.05 g, 15.0 mmol, 3% in mixed solution of diethoxymethane and n-pentane) (2) was added and the reaction mixture was stirred under irradiation by a 24 W blue LEDs at 25 ℃ for 12 hours under argon. The reaction mixture diluted with water (200 mL) and extracted with ethyl acetate (100 mL) for three times. The combined organic phase was dried over anhydrous $Na_2SO_4$ and concentrated to give a residue which was purified by silica gel chromatography, eluting with petroleum ether/ethyl acetate, to give compound **4a** as a light yellow solid (1.15 g, 79%).

**6. Unsuitable substrates**

To expand the substrate scope, we tested some other heteroarenes, such as quinoline, isoquinoline and quinoxaline. Unfortunately, no desired product was obtained.



1ba 1bb 1bc

## 7. References

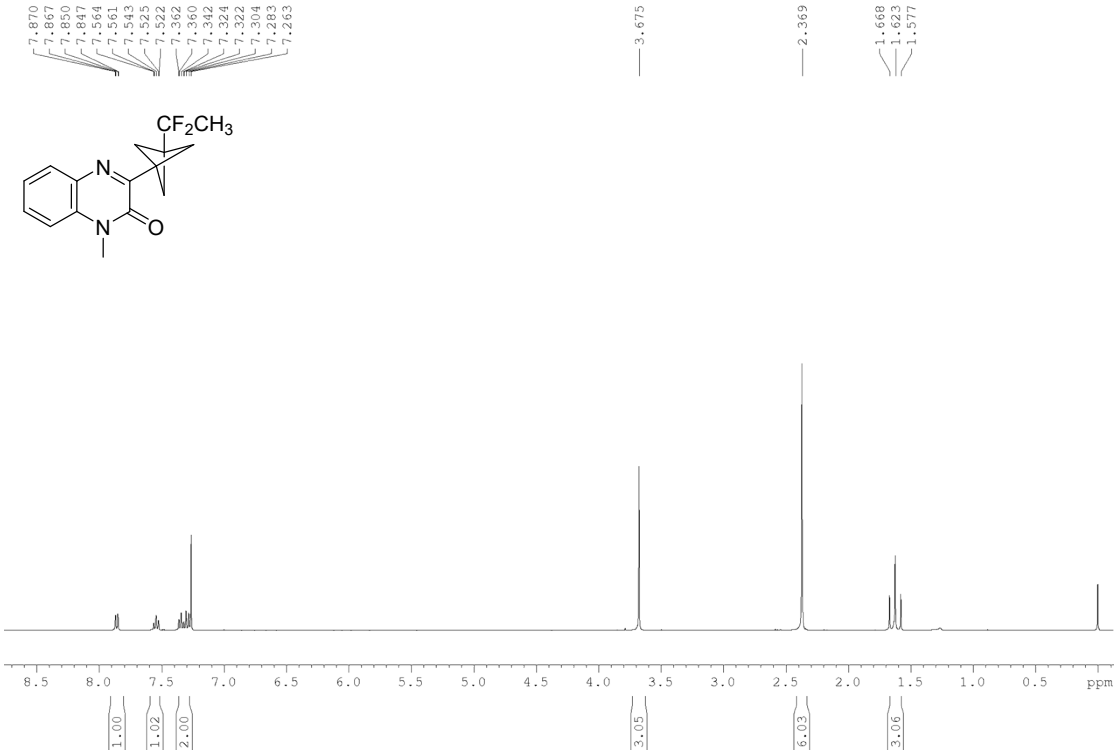
(1) Y. P. Cai, F. Y. Nie, Q. H. Song. *J. Org. Chem*. 2021, **86**, 12419.

(2) M. A. Cismesia, T. P. Yon. *Chem Sci*. 2015, **6**, 5426.

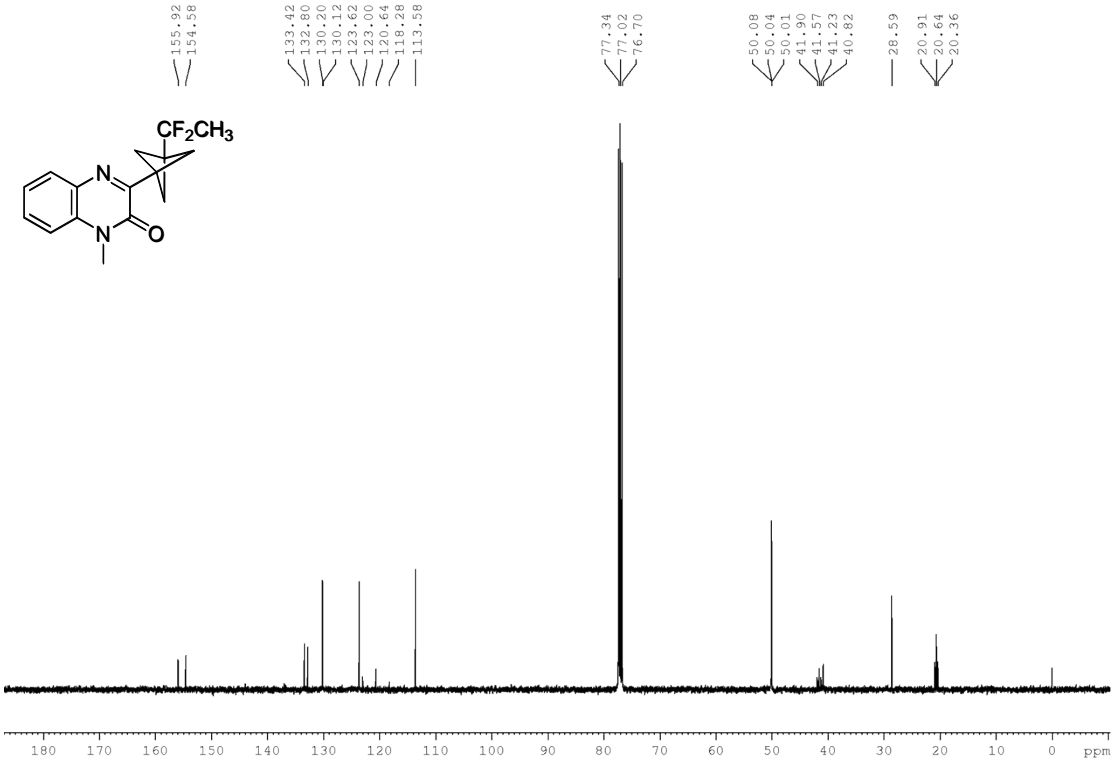
(3) J. N. Demas, W. D. Bowman, E. F. Zalewski, R. A. Velapoldi. *J. Phys. Chem*. 1981, **85**,2766.

## 8. $^{1}H$ NMR, $^{13}C$ NMR and $^{19}F$ NMR spectra

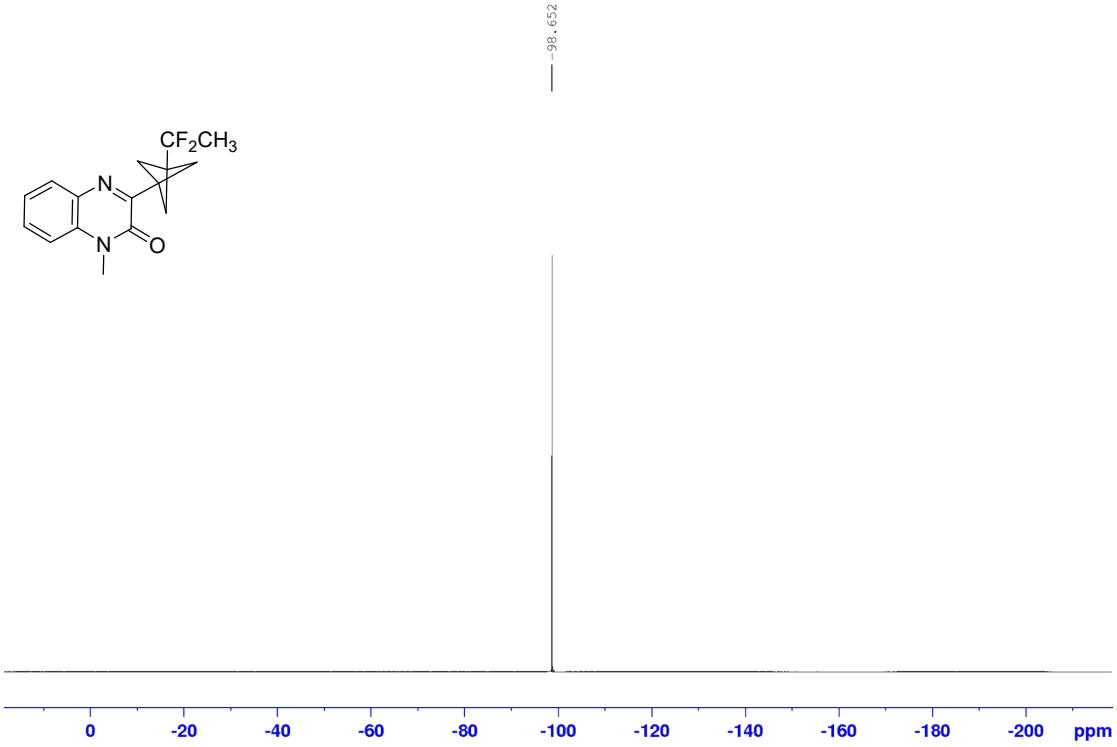
### 4a $^{1}H$ NMR (400 MHz, $CDCl_3$)




### 4a $^{13}C$ NMR (100 MHz, $CDCl_3$)



**4a $^{19}$F NMR (376 MHz, $CDCl_3$)**

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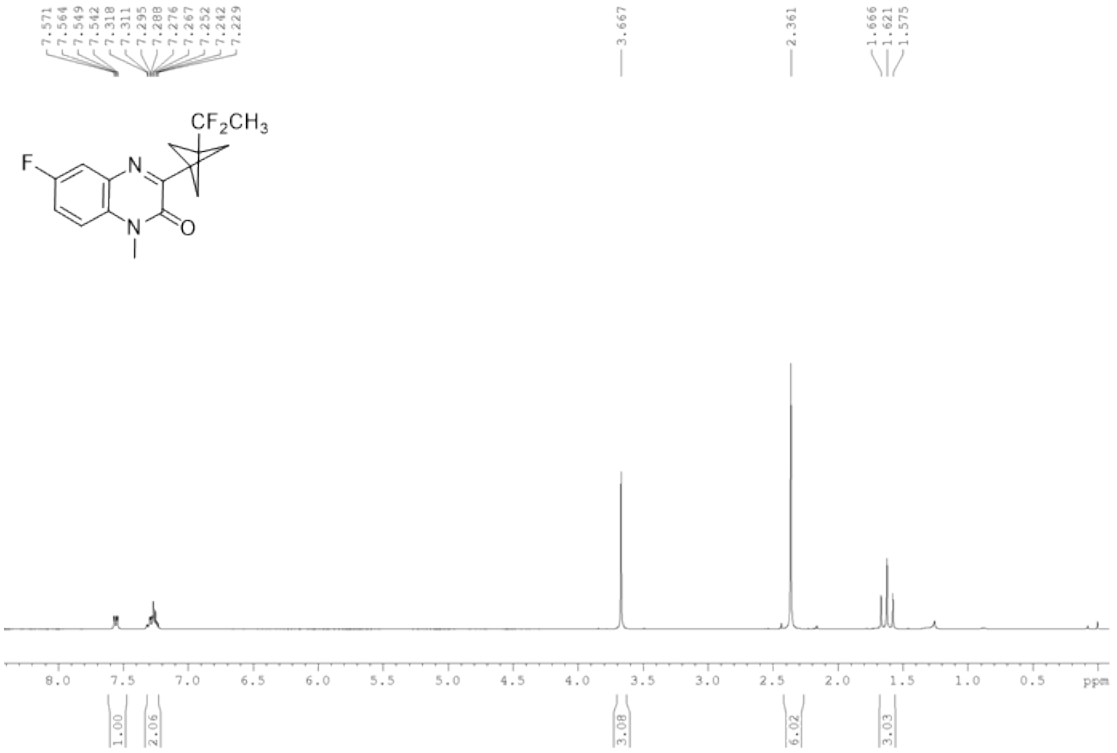
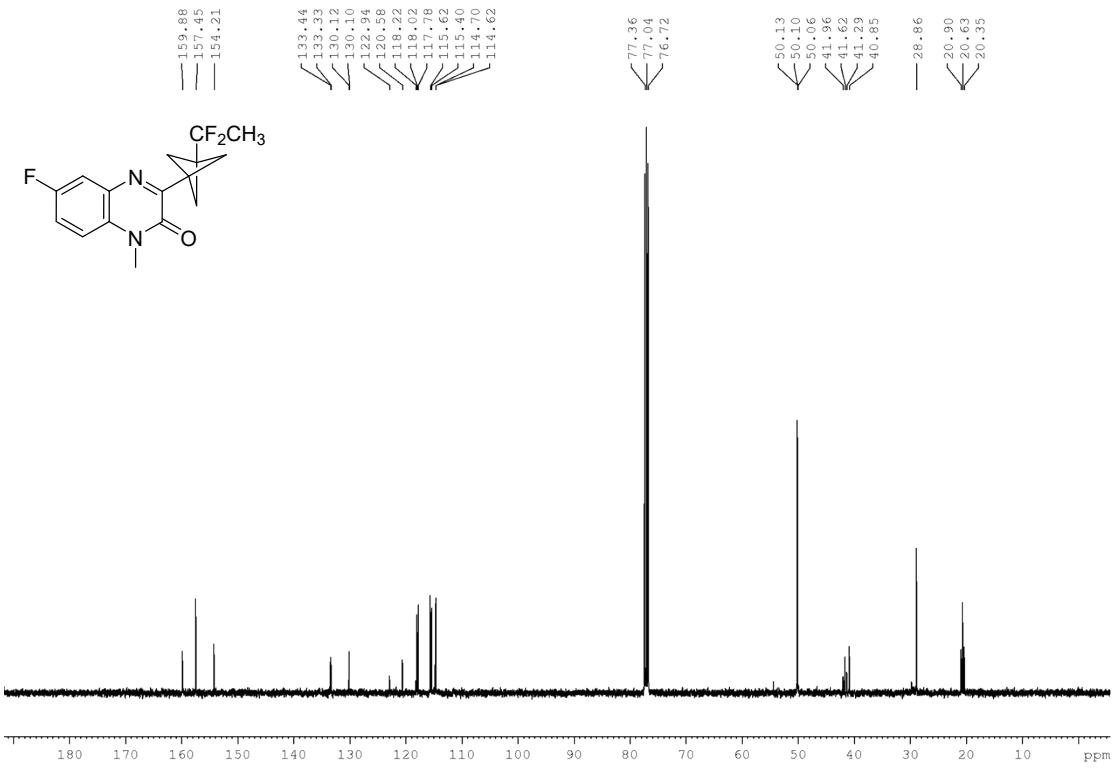
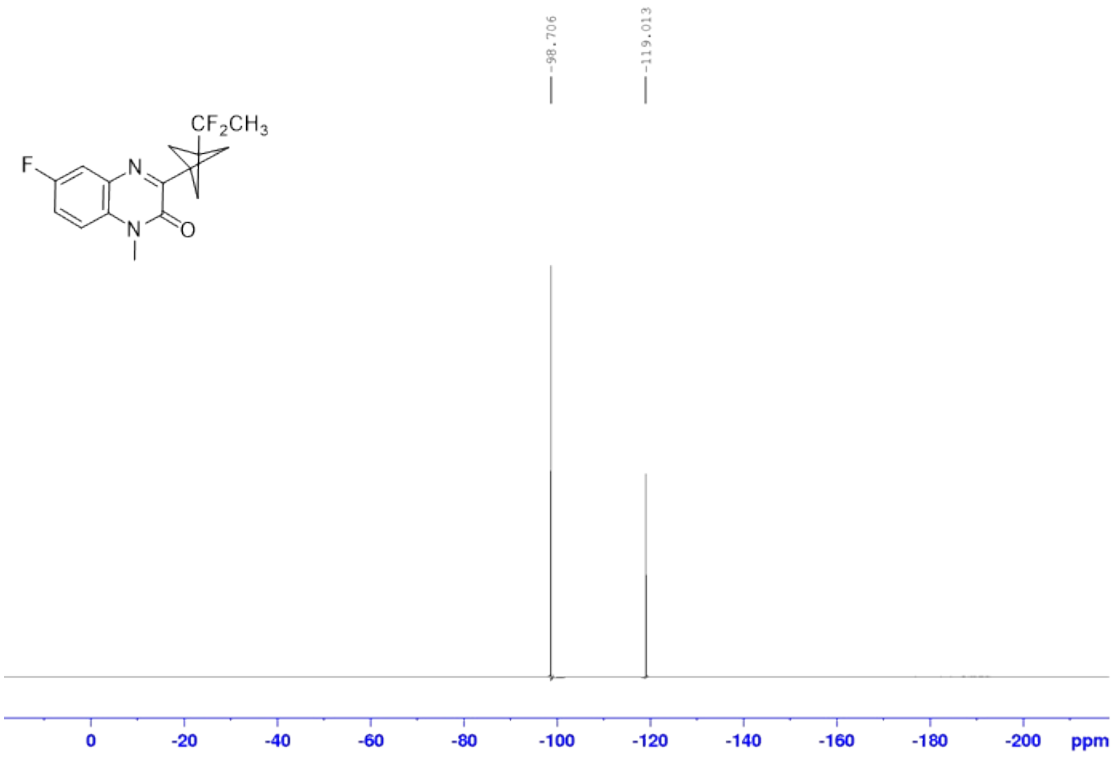


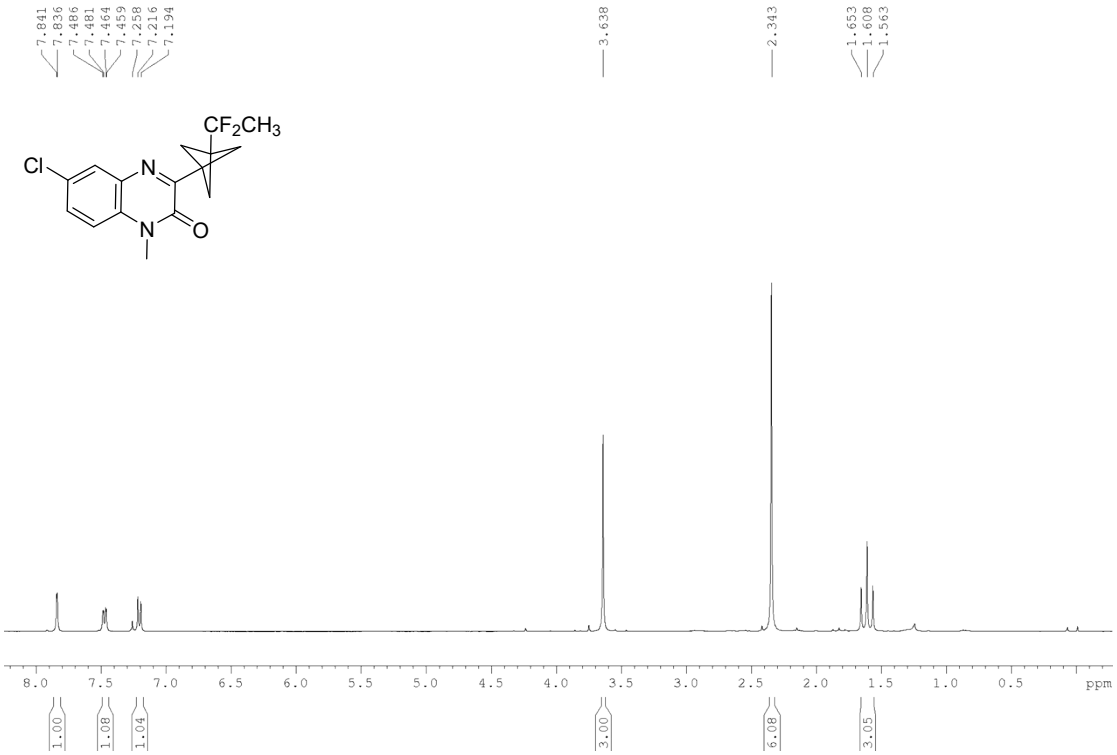

**4b $^{13}$C NMR (100 MHz, $CDCl_3$)**




**4b $^{19}$F NMR (376 MHz, $CDCl_3$)**




**4c $^{1}$H NMR (400 MHz, $CDCl_3$)**

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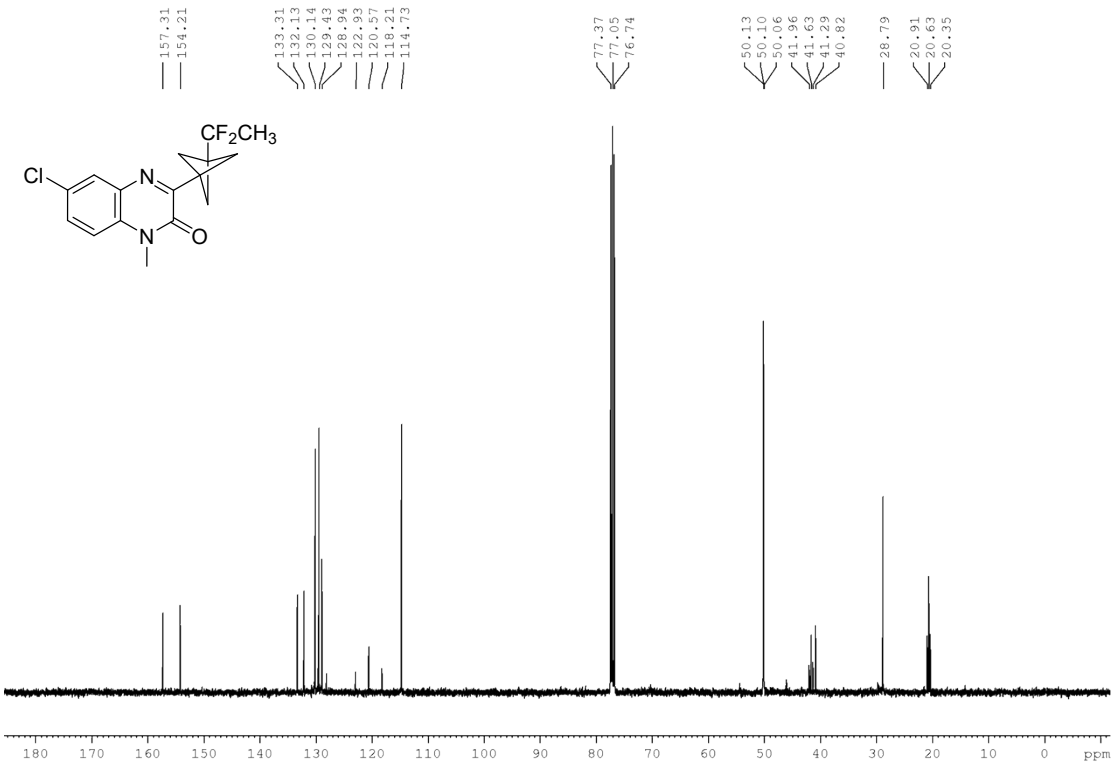
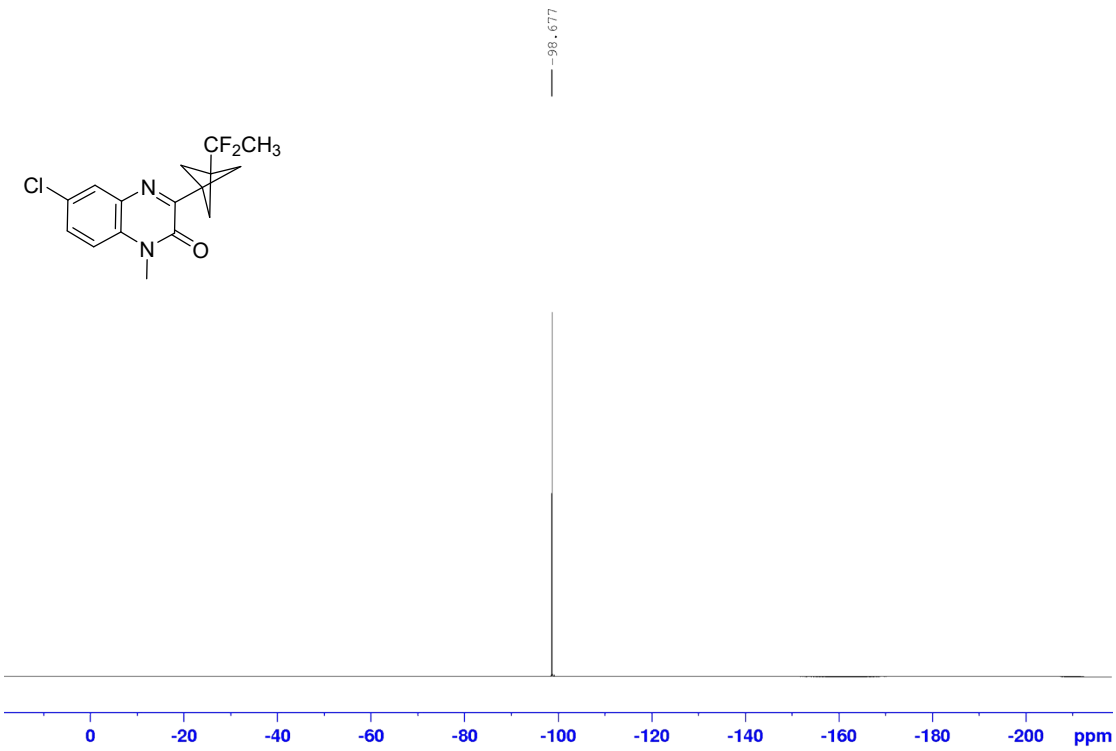
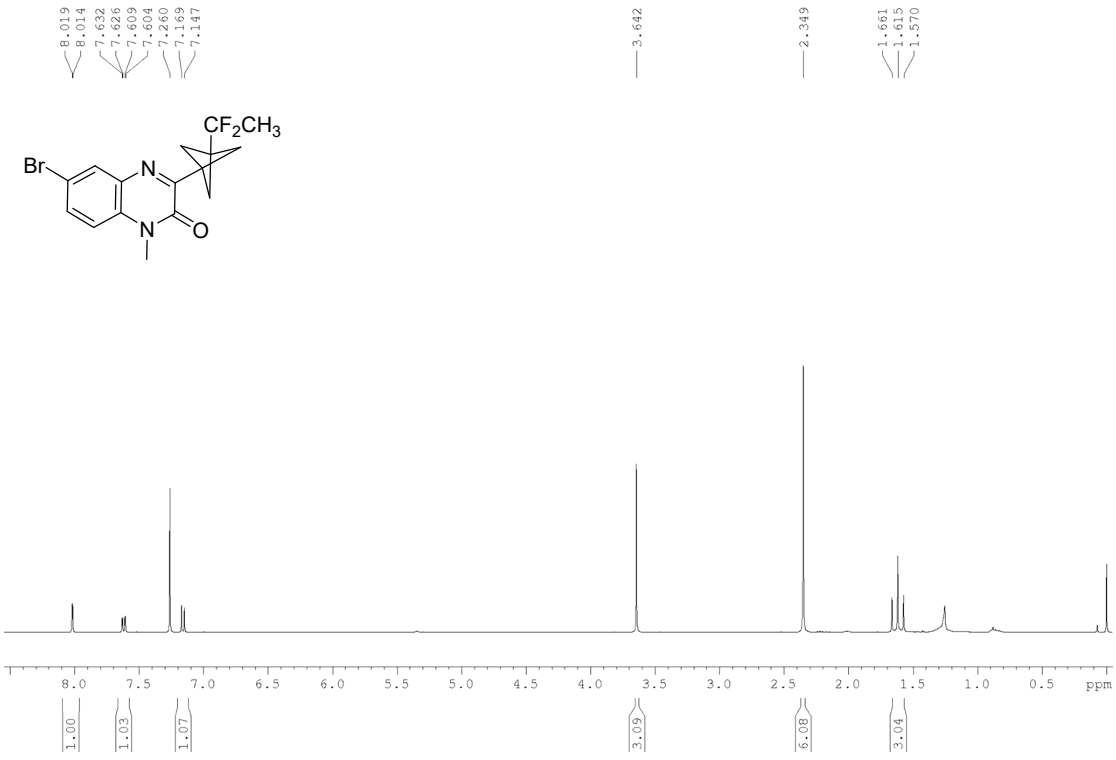


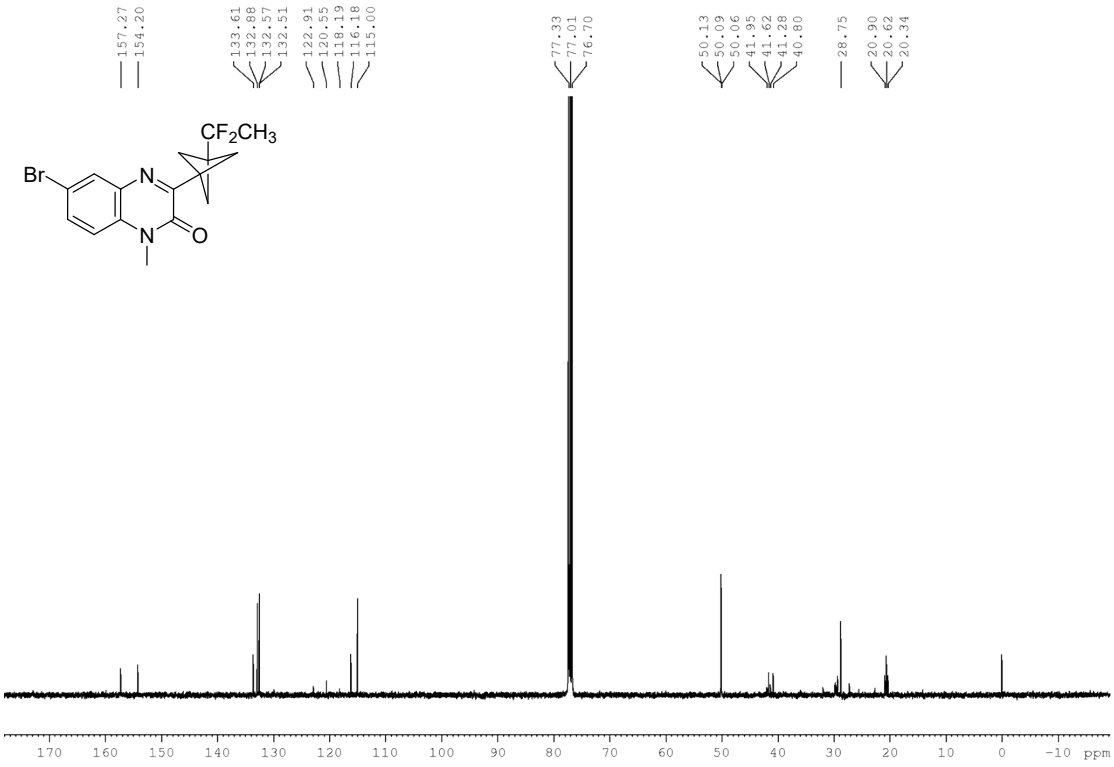

**4c $^{19}$F NMR (376 MHz, $CDCl_3$)**




**4d $^{1}$H NMR (400 MHz, $CDCl_3$)**




**4d $^{13}$C NMR (100 MHz, $CDCl_3$)**

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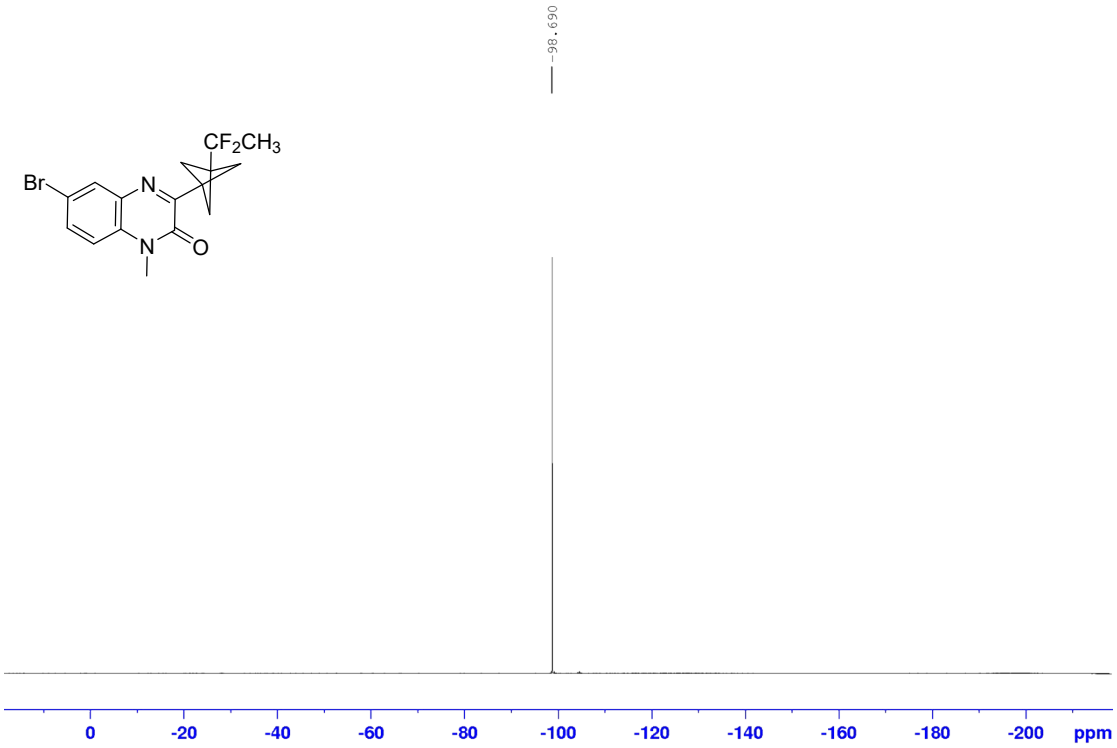


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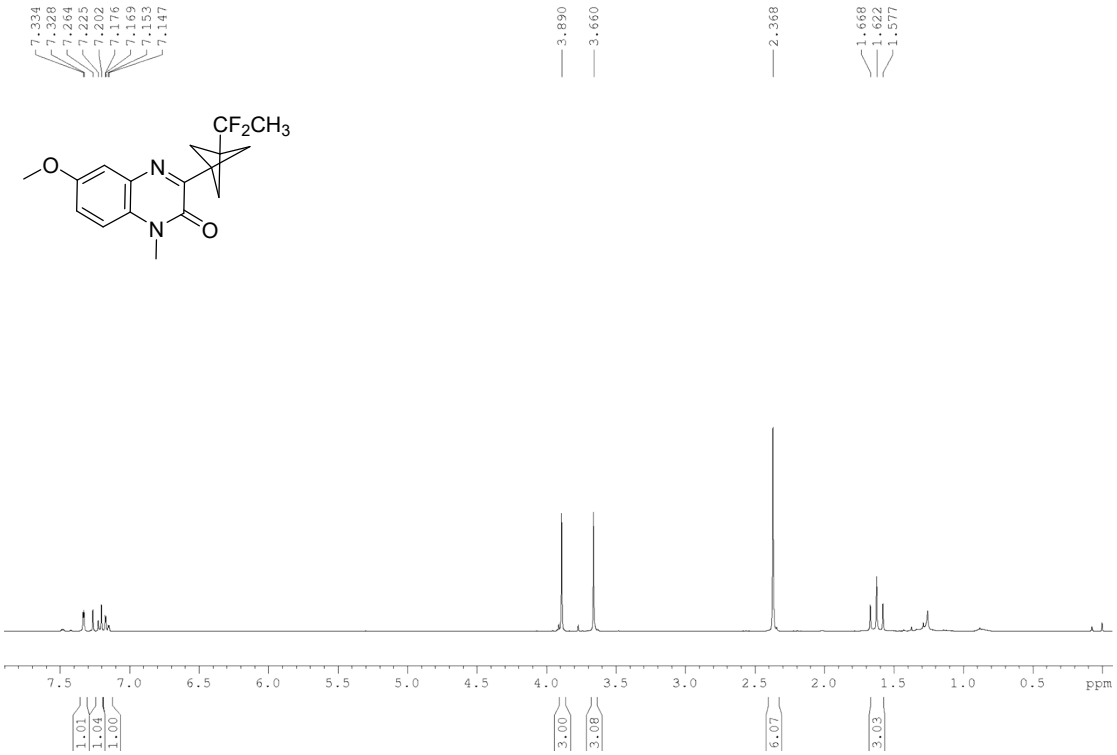


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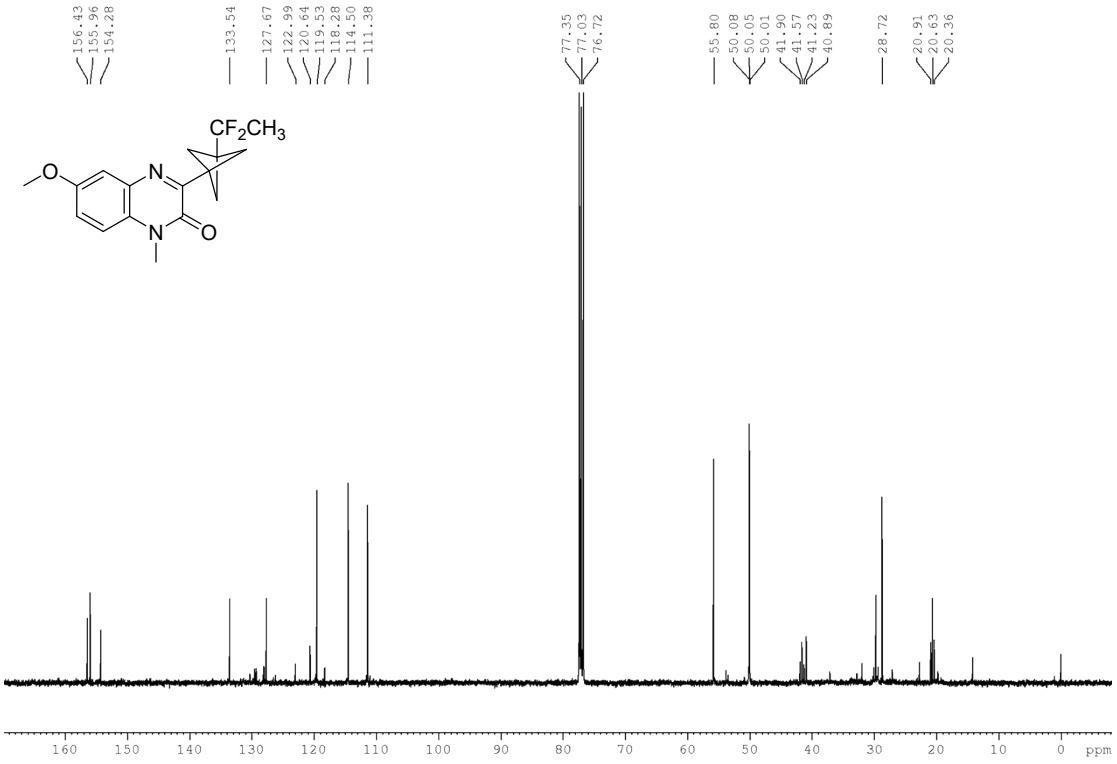


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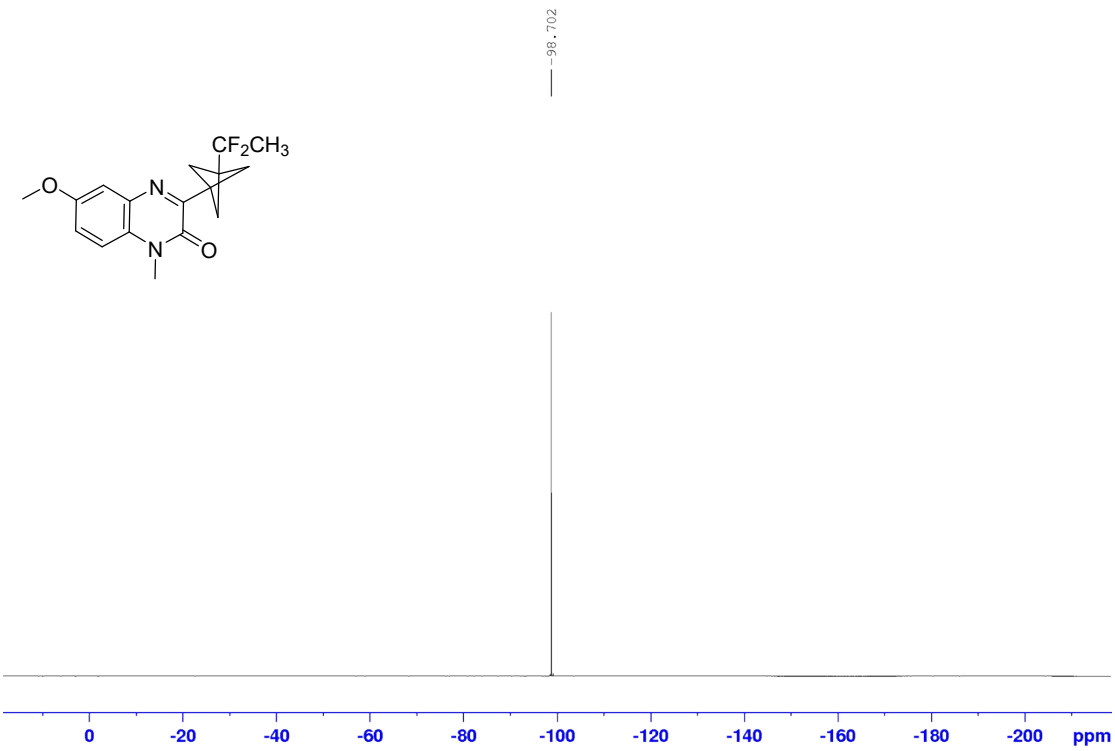
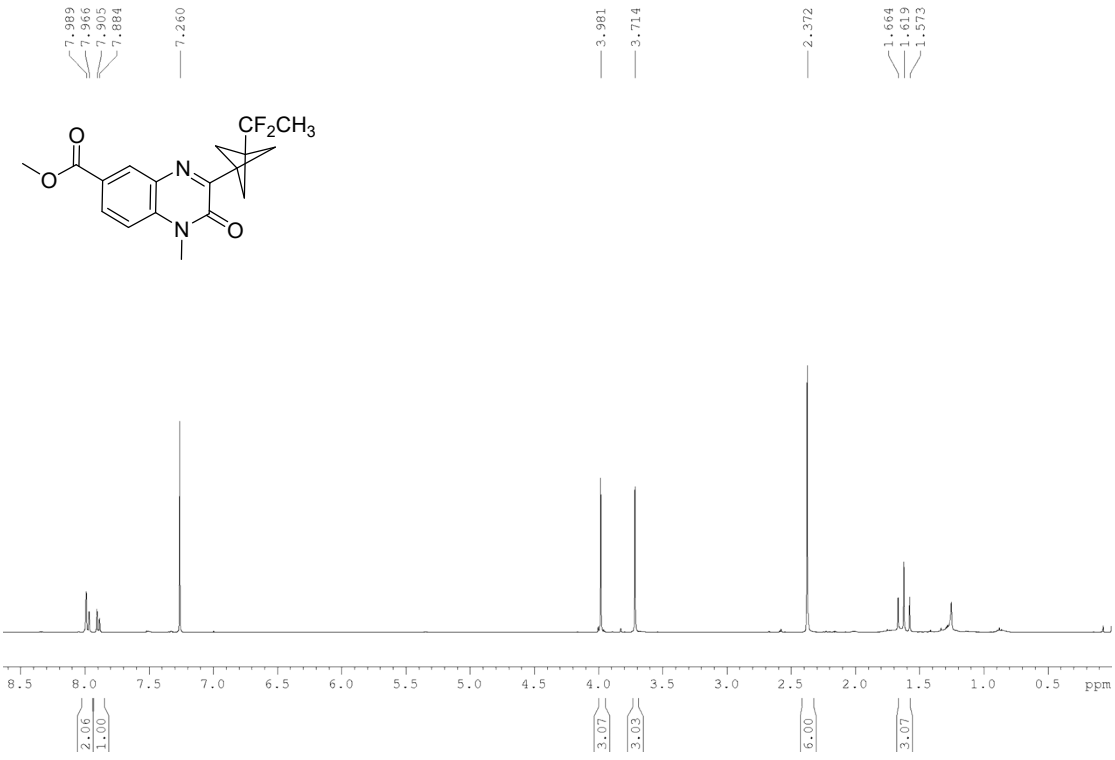
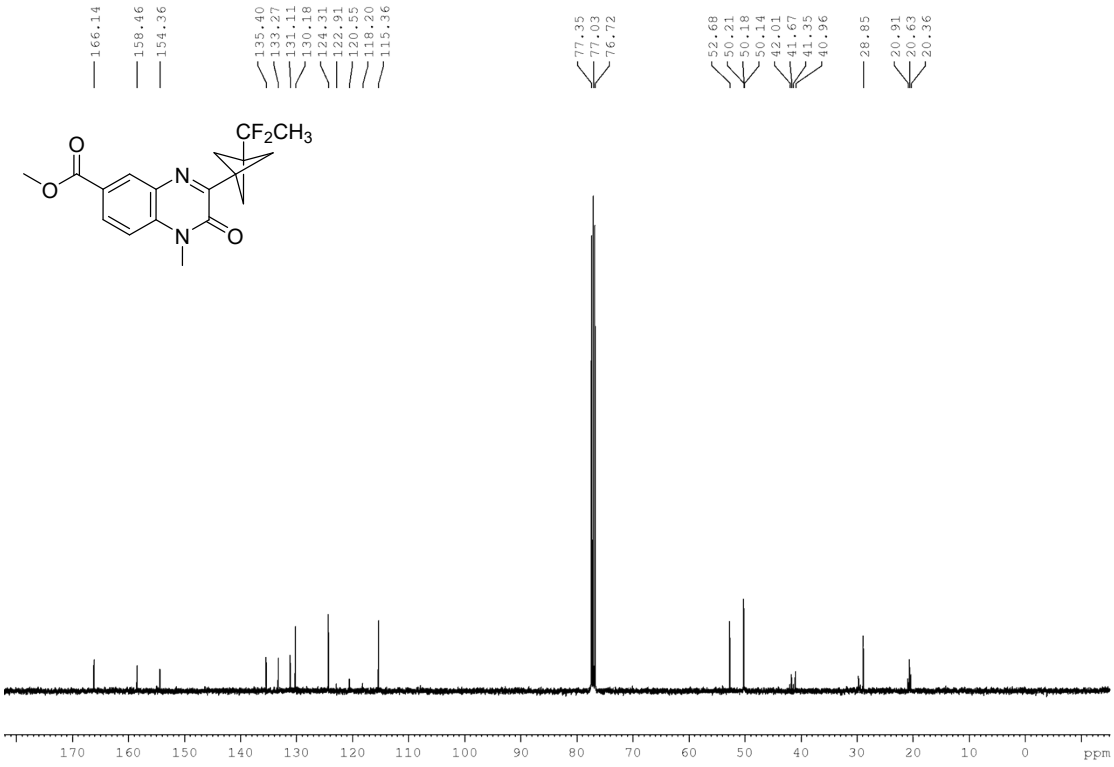


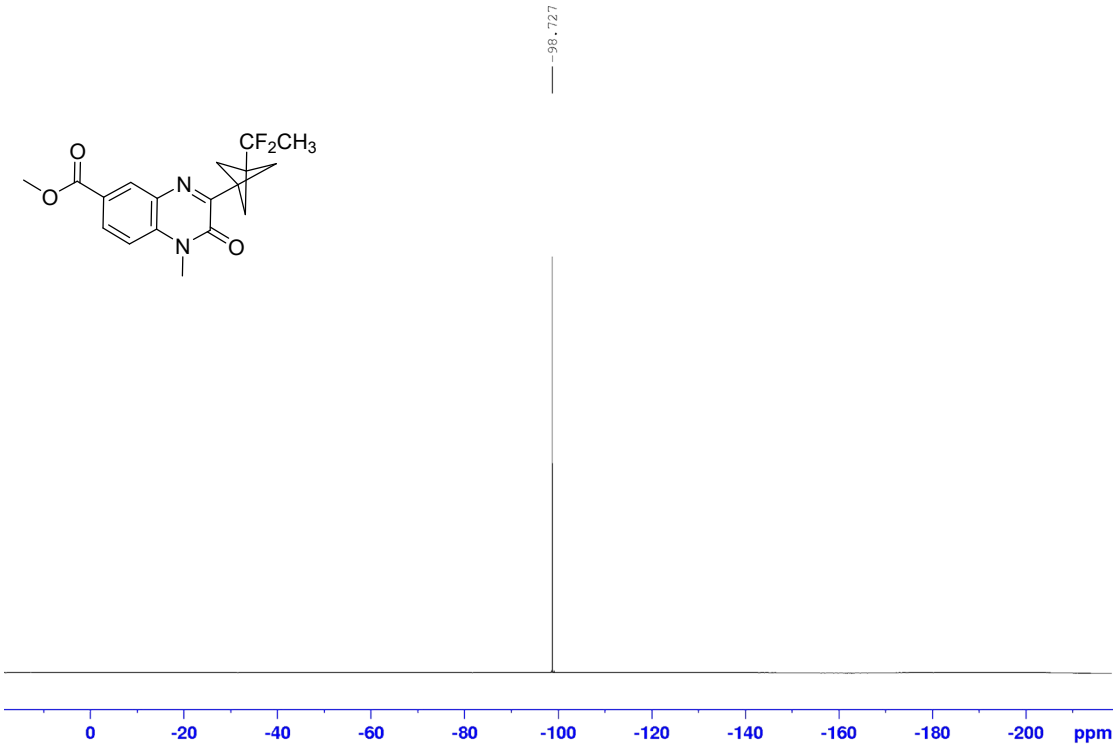

**4f $^{1}$H NMR (400 MHz, $CDCl_3$)**



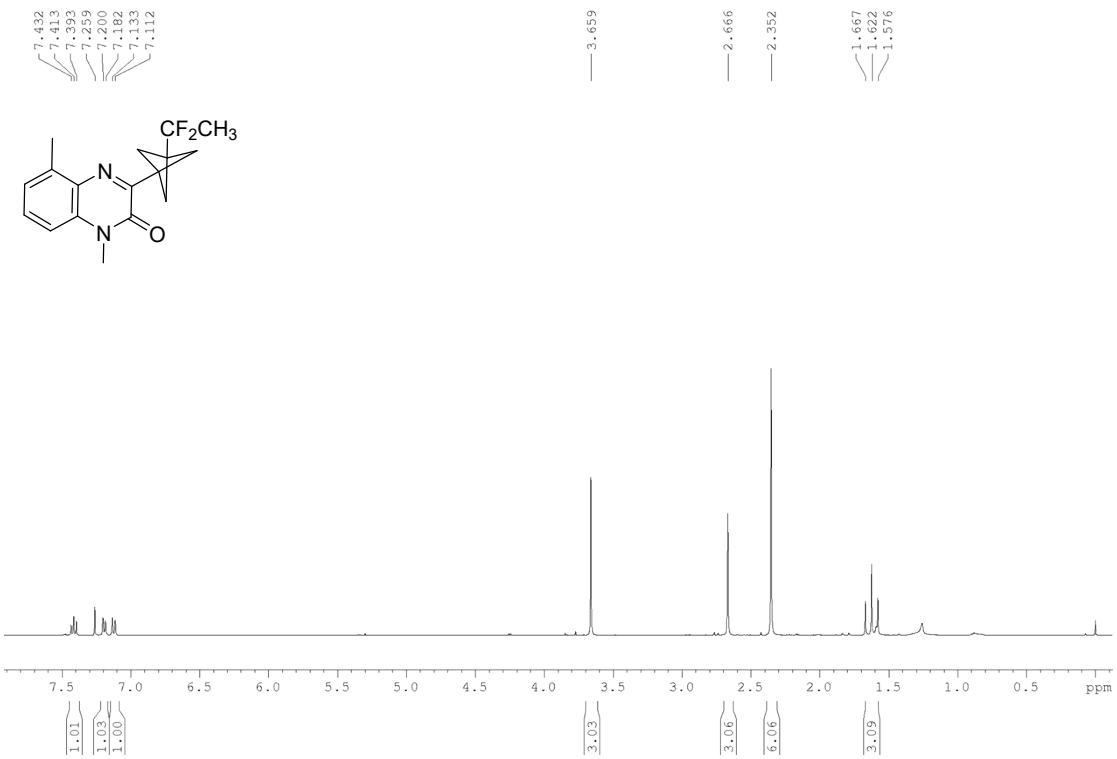

**4f $^{13}$C NMR (100 MHz, $CDCl_3$)**



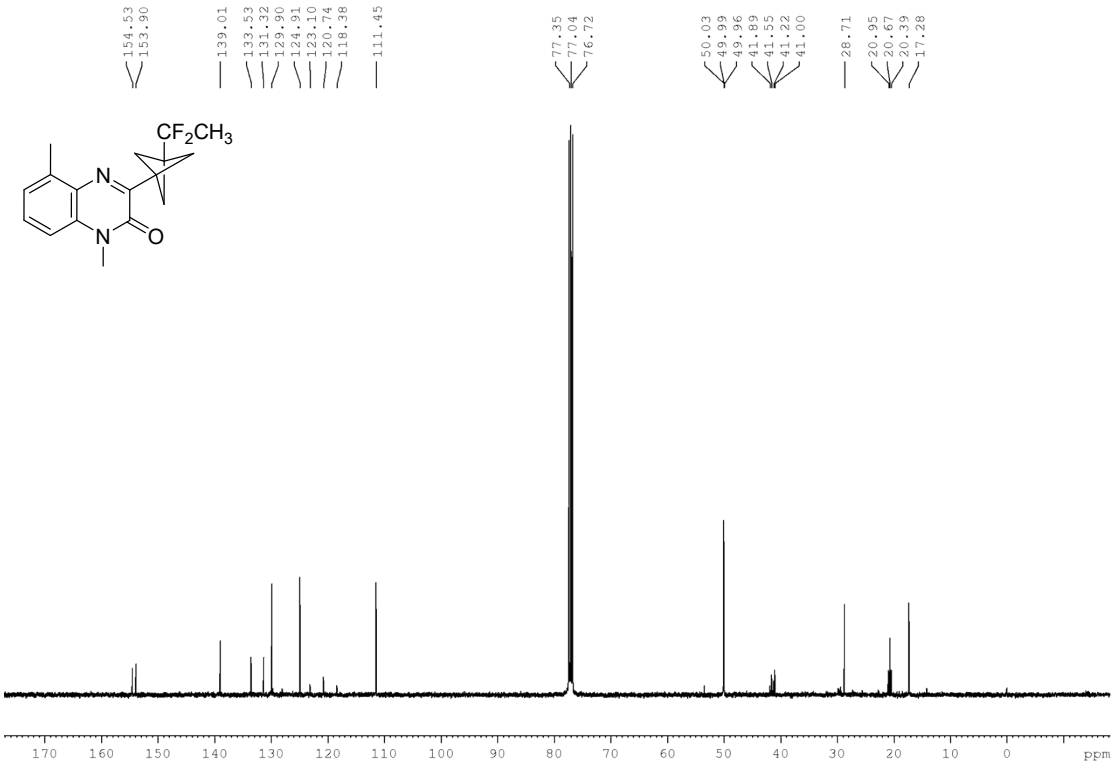

**4f $^{19}$F NMR (376 MHz, $CDCl_3$)**



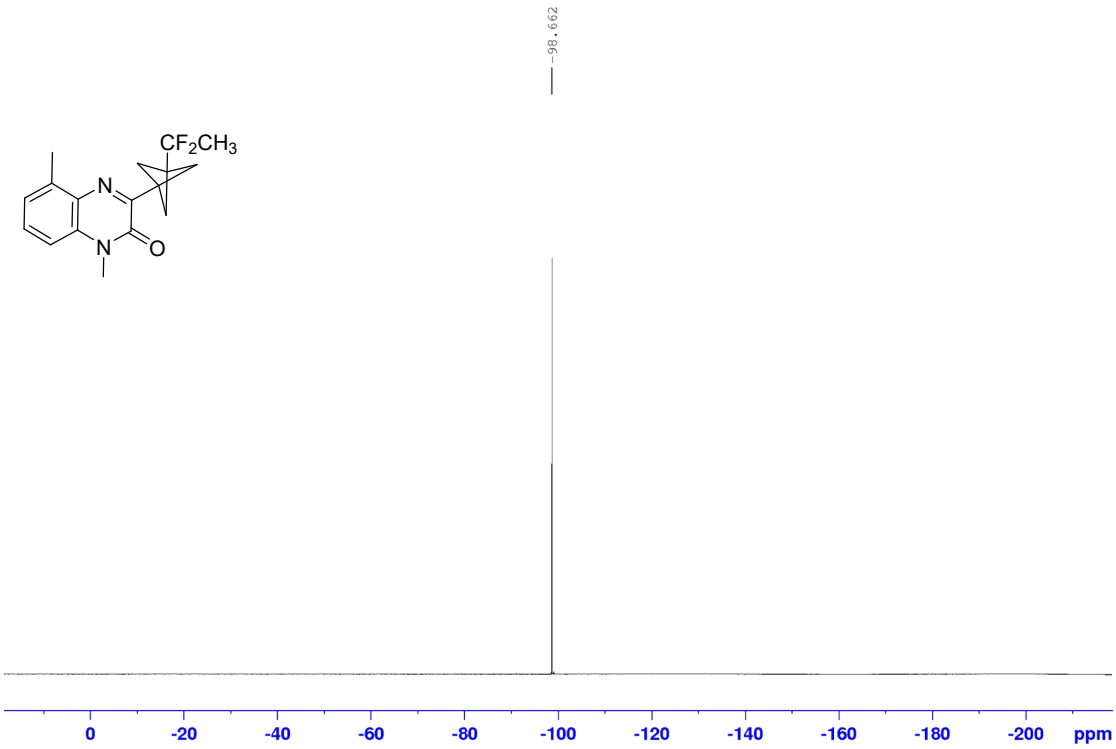

**4g $^{1}$H NMR (400 MHz, $CDCl_3$)**



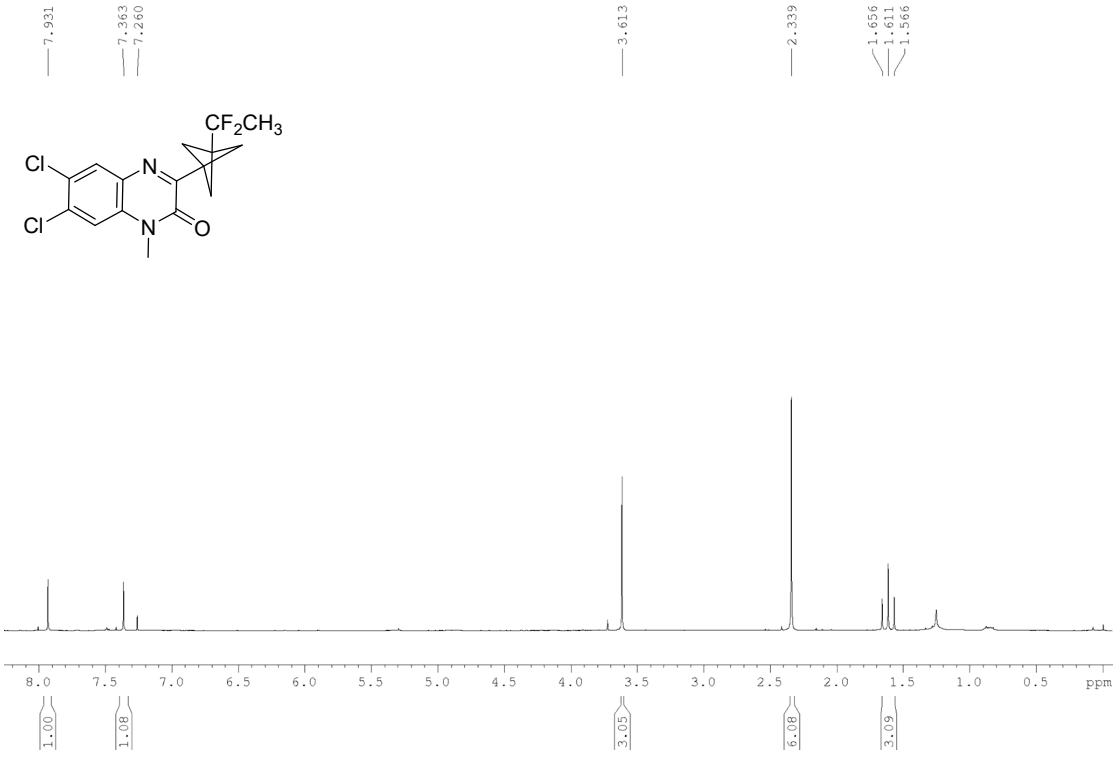

**4g $^{13}$C NMR (100 MHz, $CDCl_3$)**



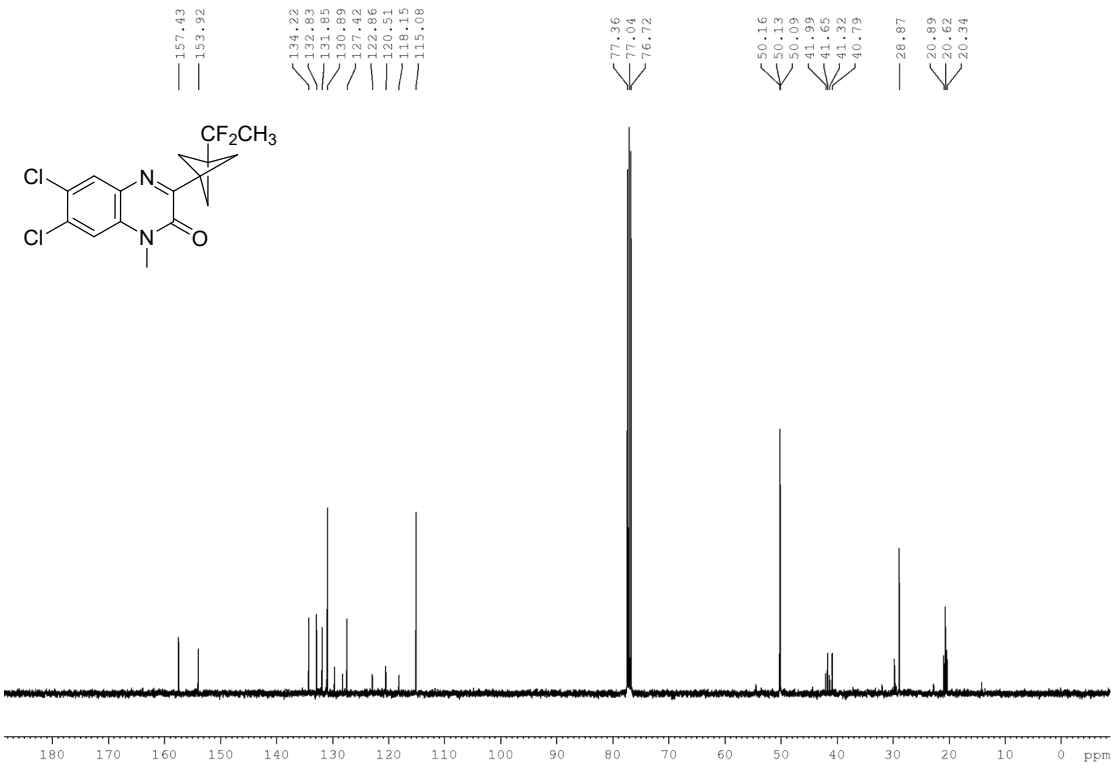

**4g $^{19}$F NMR (376 MHz, $CDCl_3$)**



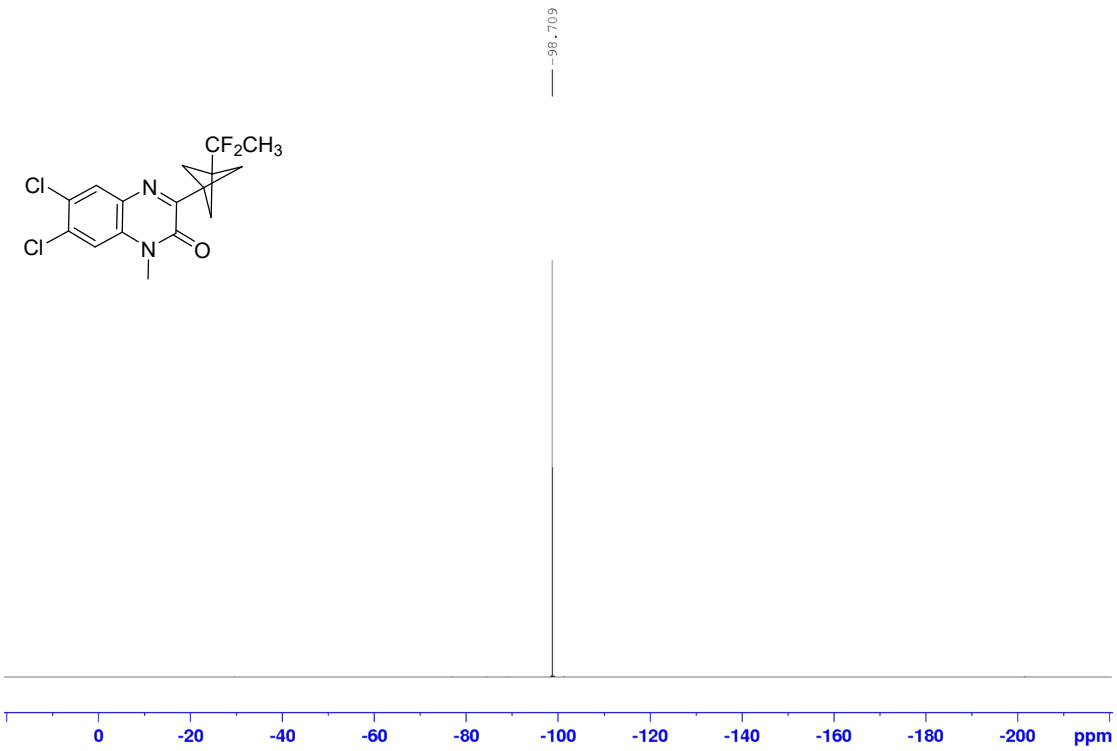

**4h $^{1}$H NMR (400 MHz, $CDCl_3$)**



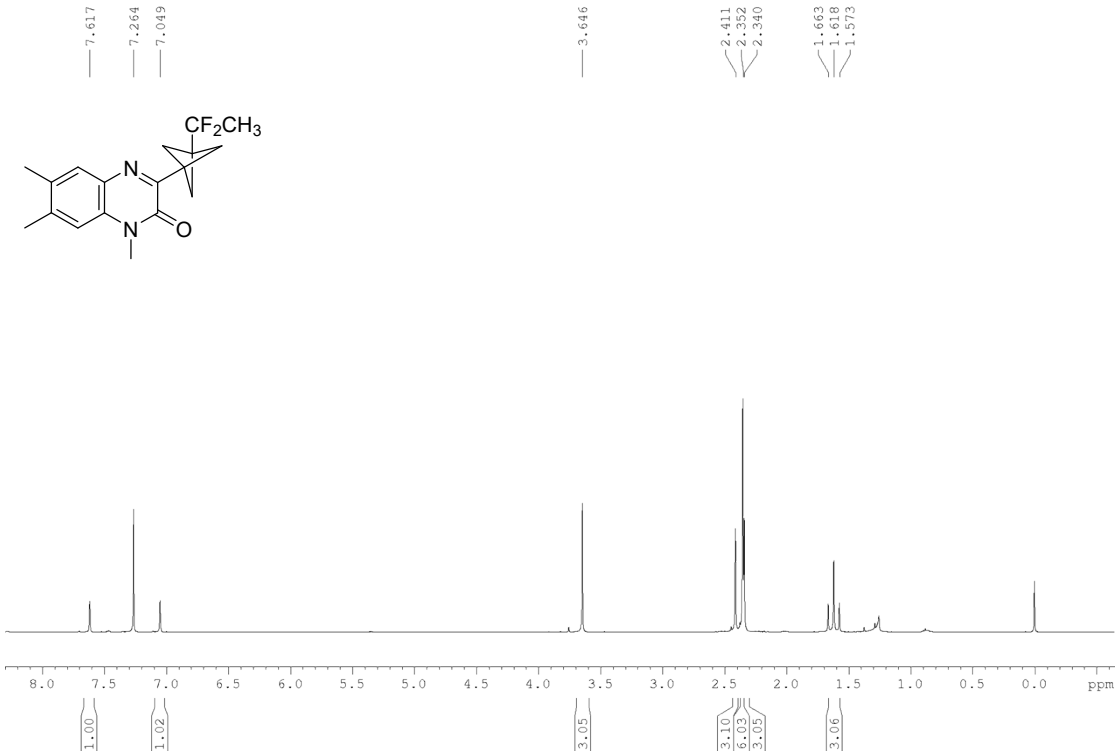

**4h $^{13}$C NMR (100 MHz, $CDCl_3$)**



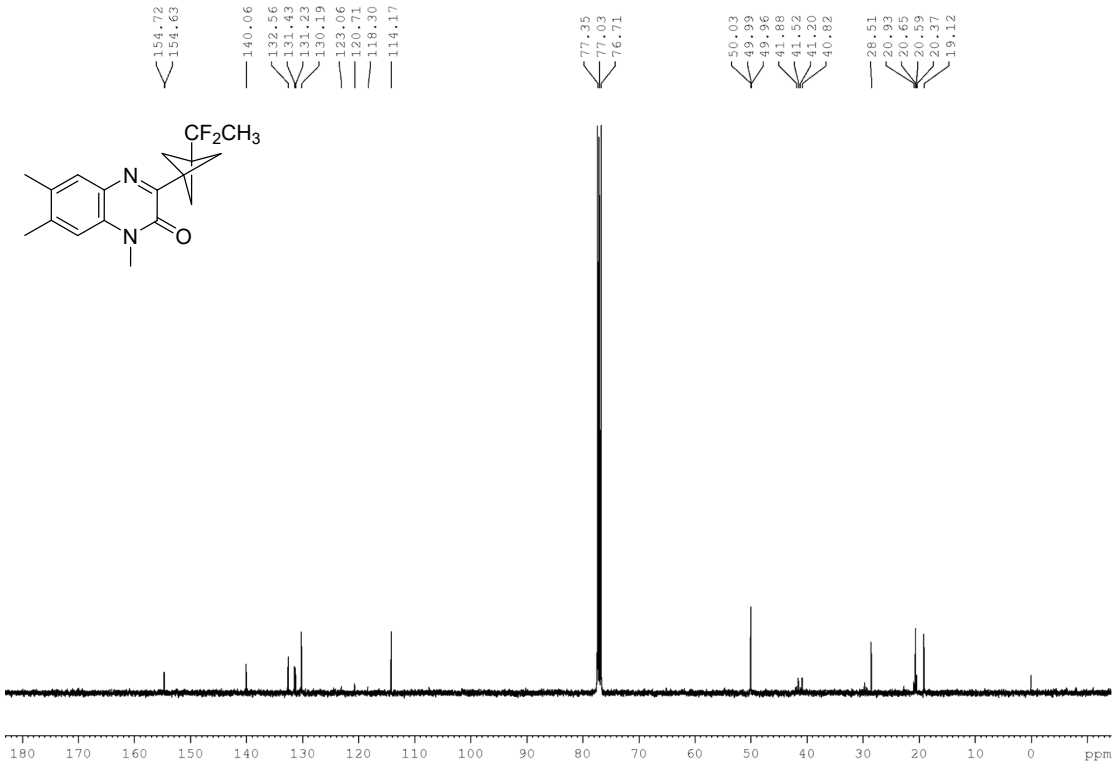

**4h $^{19}$F NMR (376 MHz, $CDCl_3$)**



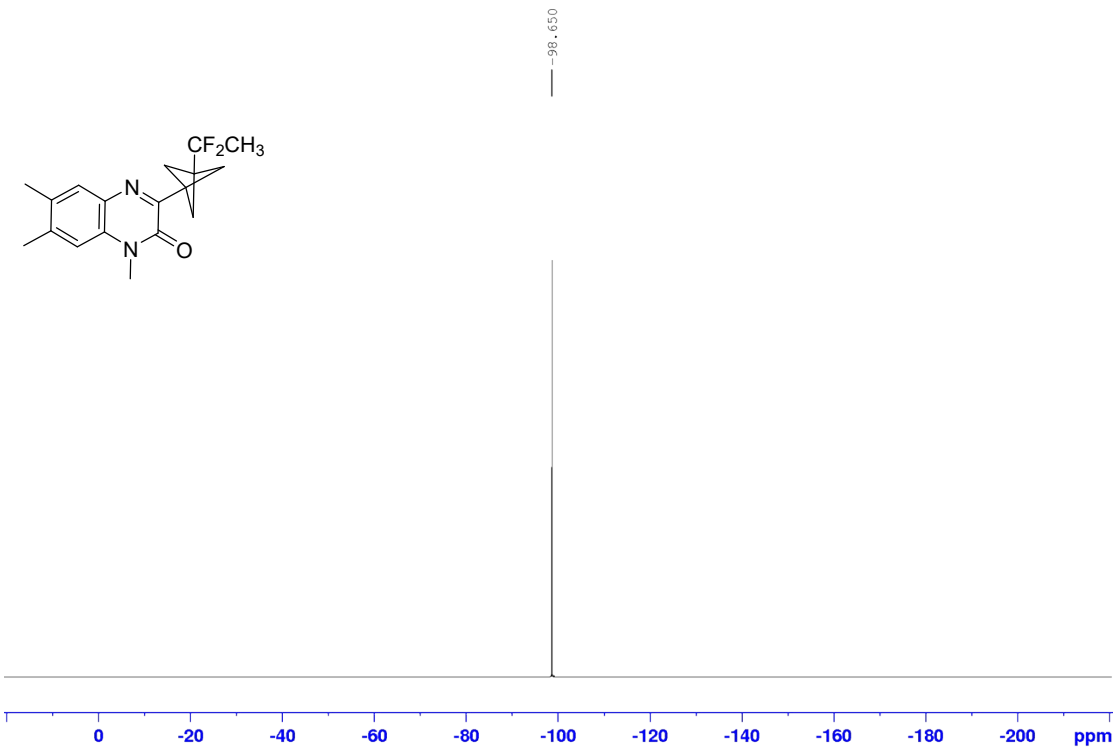

**4i $^{1}$H NMR (400 MHz, $CDCl_3$)**



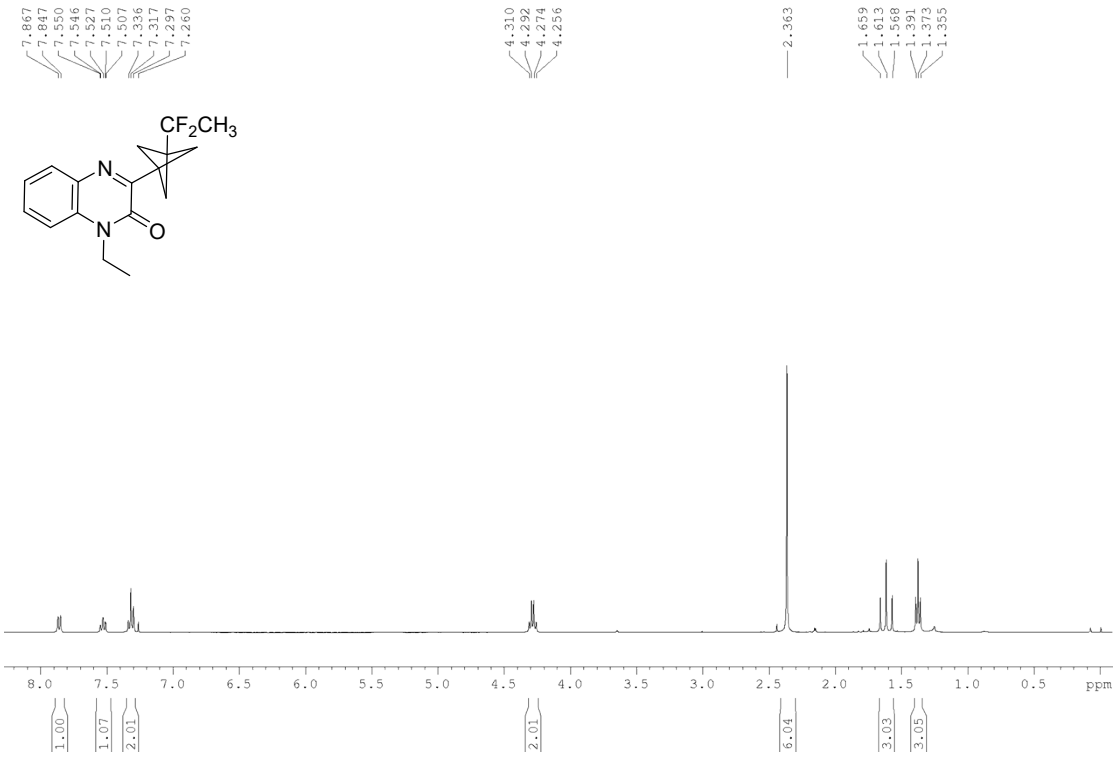

**4i $^{13}$C NMR (100 MHz, $CDCl_3$)**



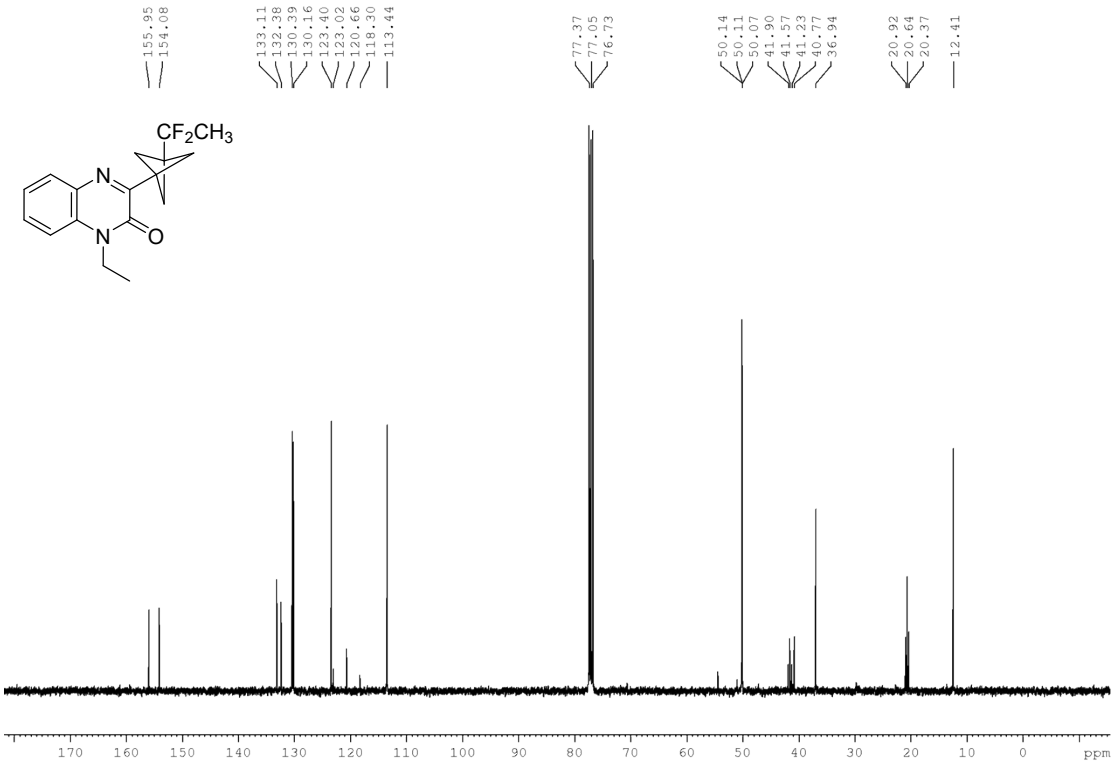

**4i $^{19}$F NMR (376 MHz, $CDCl_3$)**



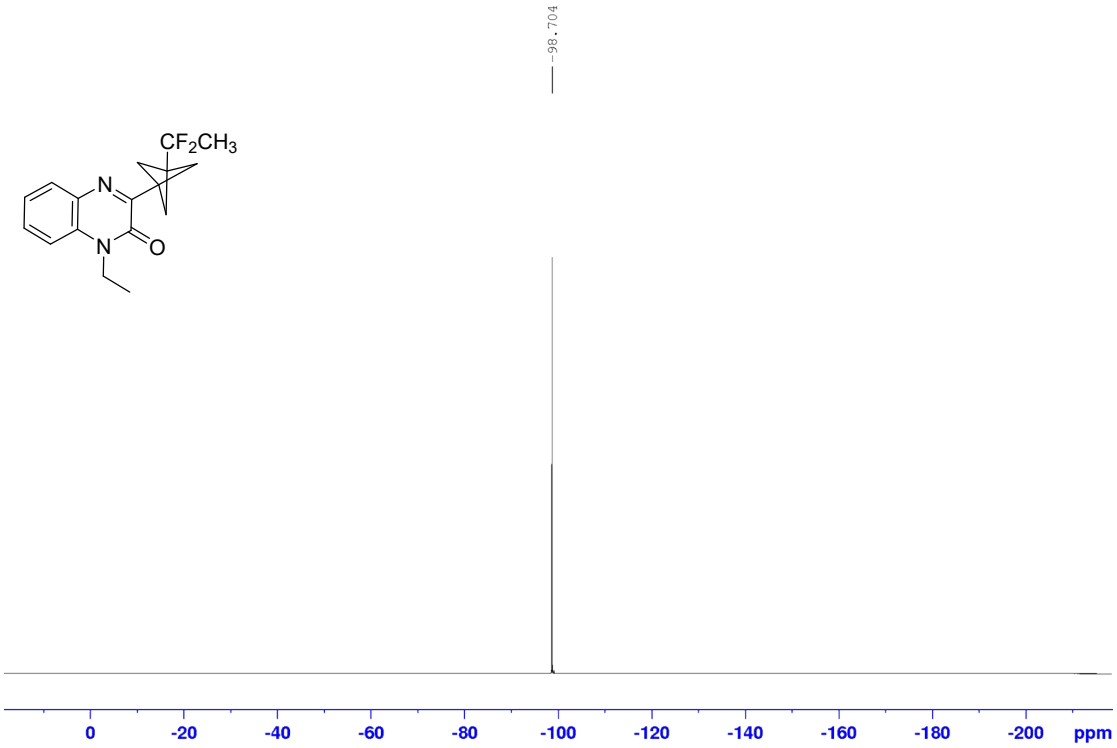

**4j $^{1}$H NMR (400 MHz, $CDCl_3$)**



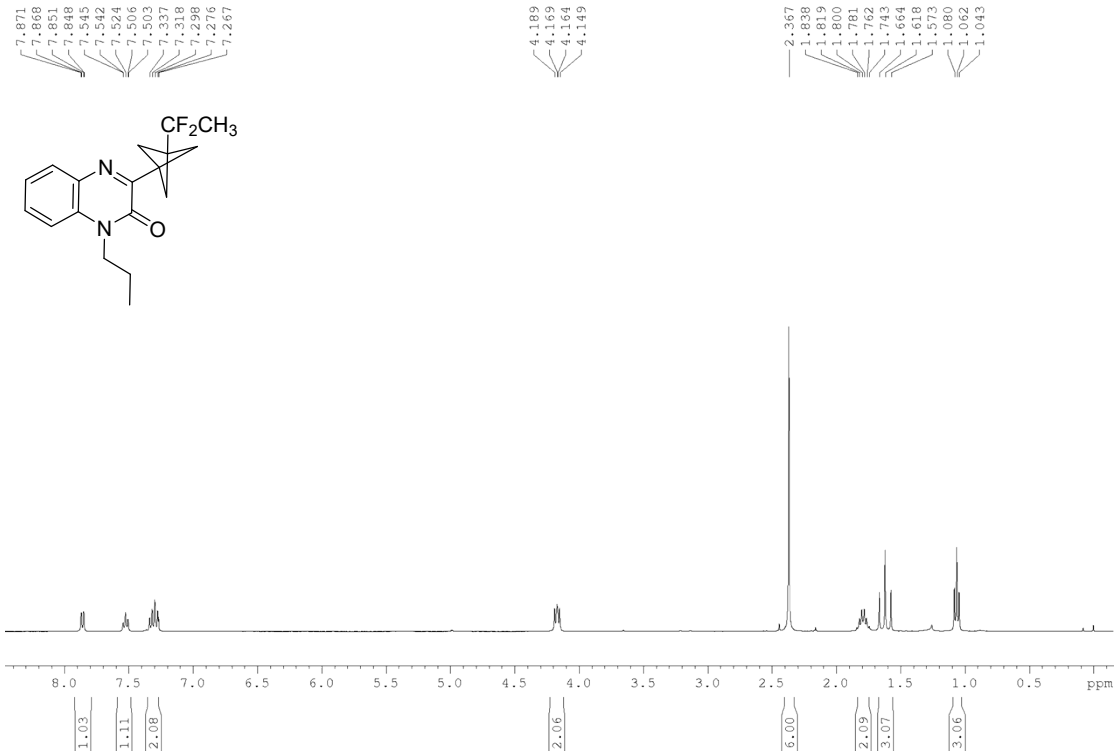

**4j $^{13}$C NMR (100 MHz, $CDCl_3$)**



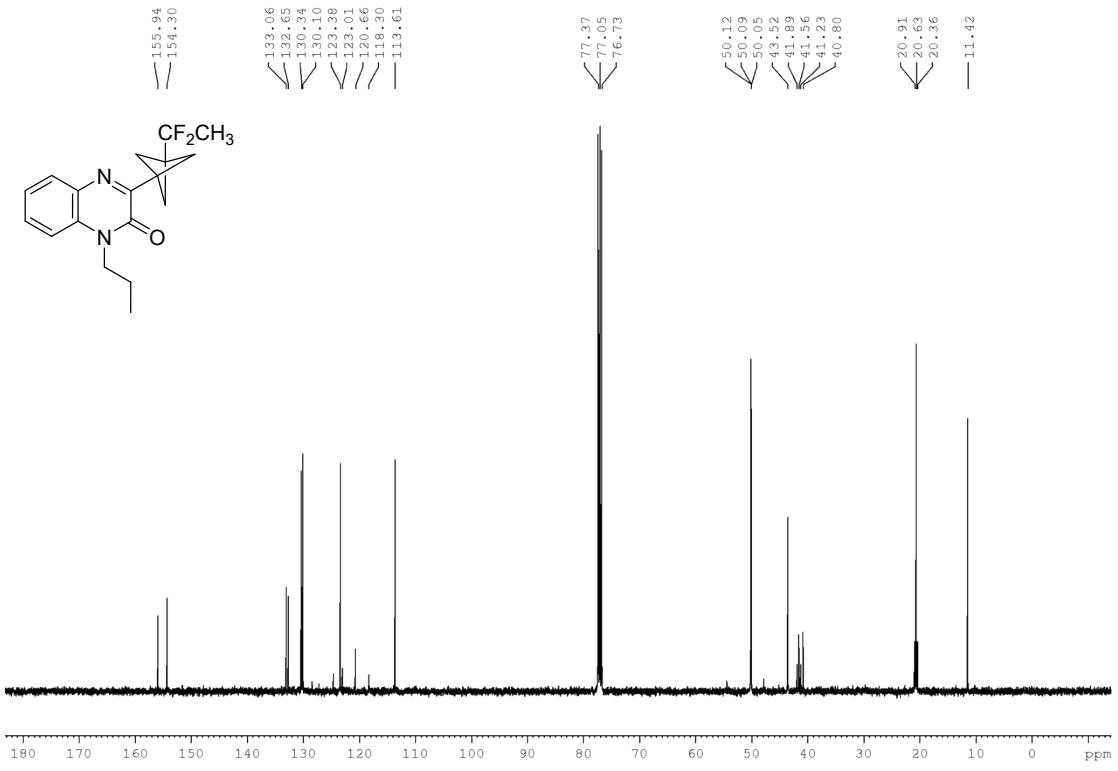

**4j $^{19}$F NMR (376 MHz, $CDCl_3$)**



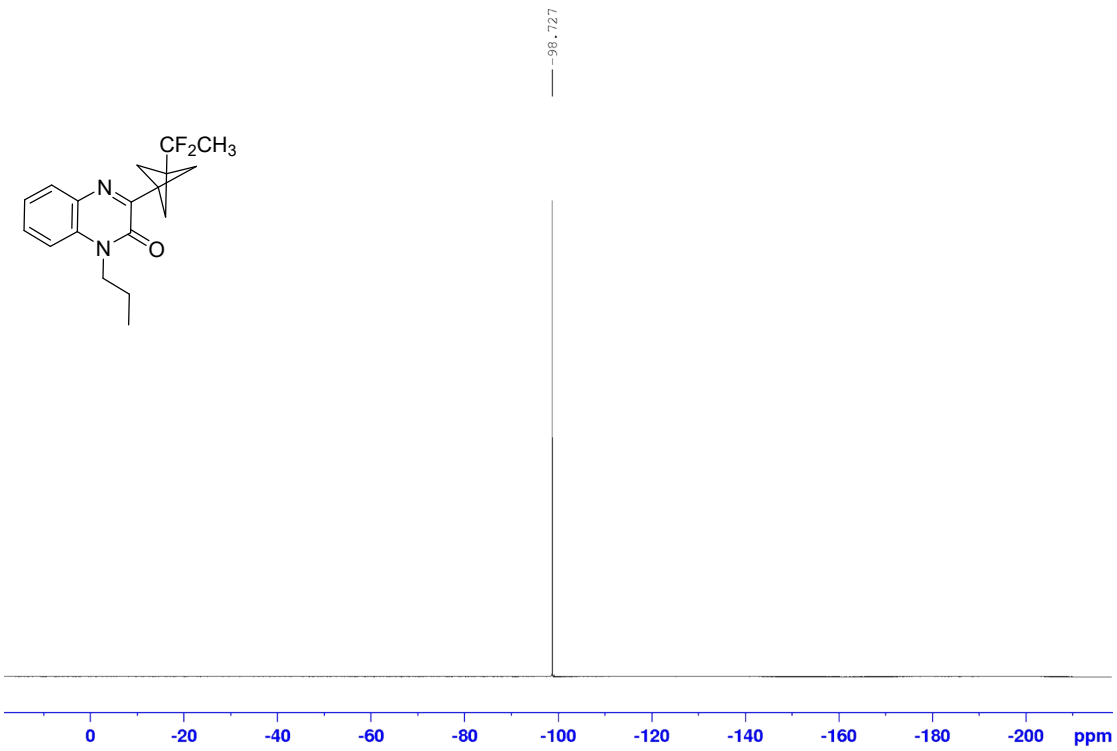

**4k $^{1}$H NMR (400 MHz, $CDCl_3$)**



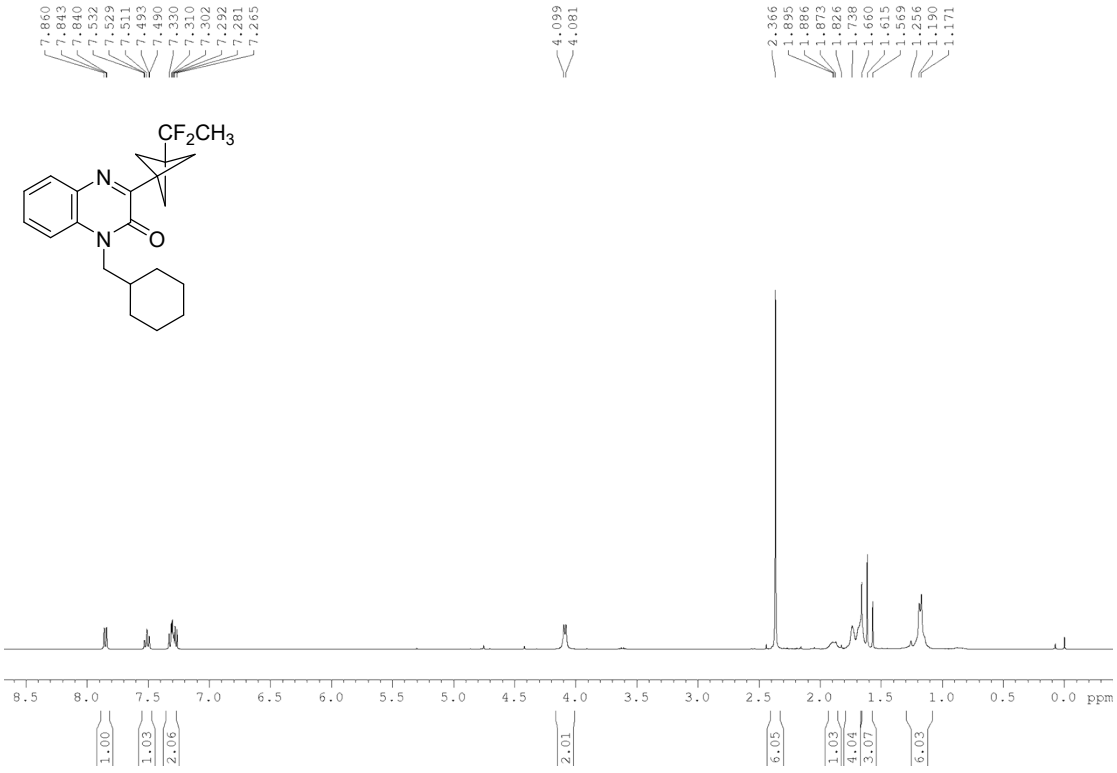

**4k $^{13}$C NMR (100 MHz, $CDCl_3$)**



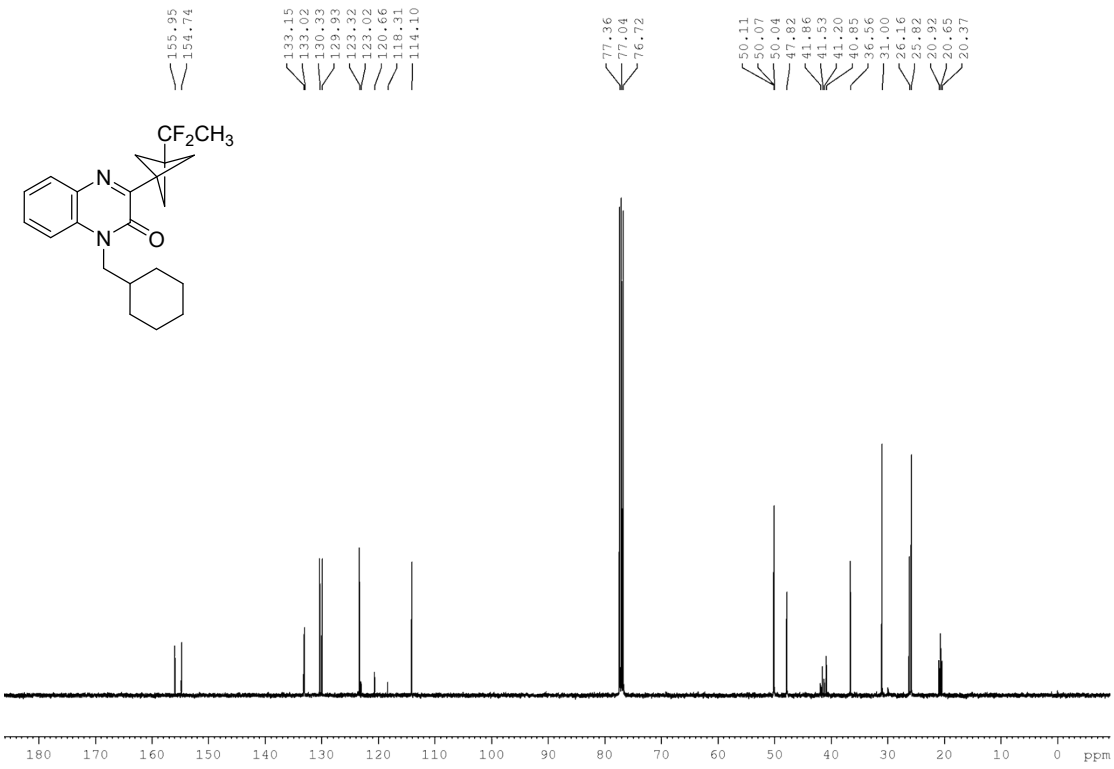

**4k $^{19}$F NMR (376 MHz, $CDCl_3$)**



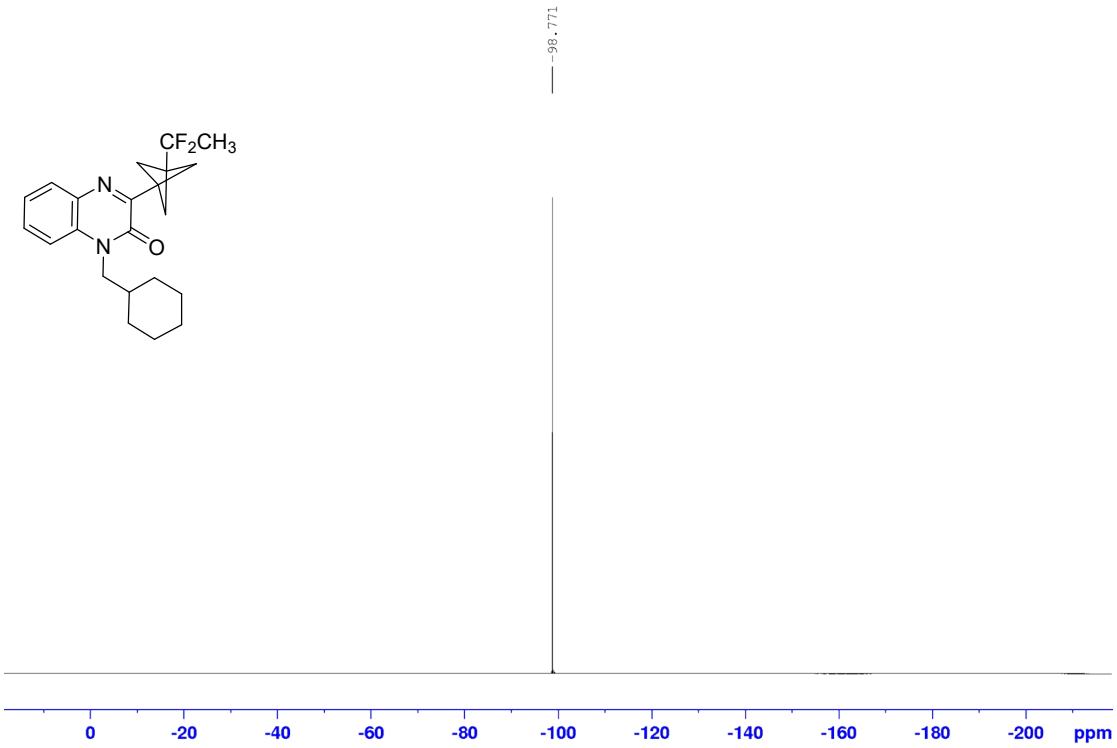

**4l $^{1}$H NMR (400 MHz, $CDCl_3$)**




**4l $^{13}$C NMR (100 MHz, $CDCl_3$)**




**4l $^{19}$F NMR (376 MHz, $CDCl_3$)**

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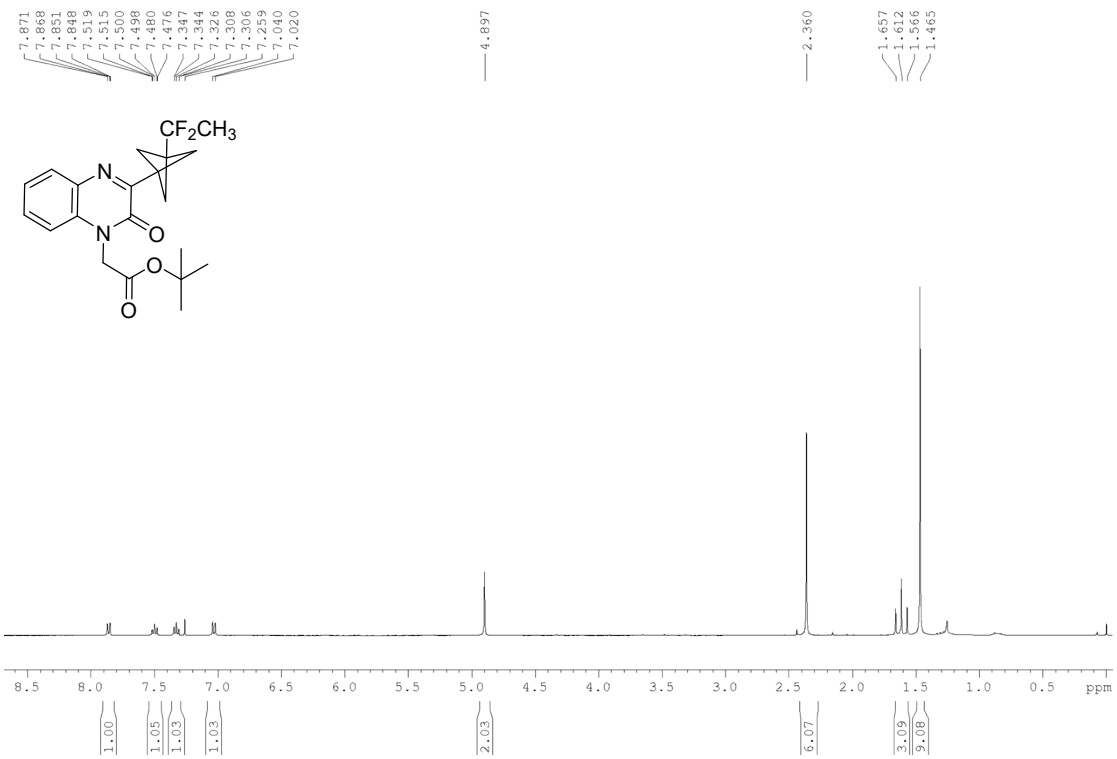
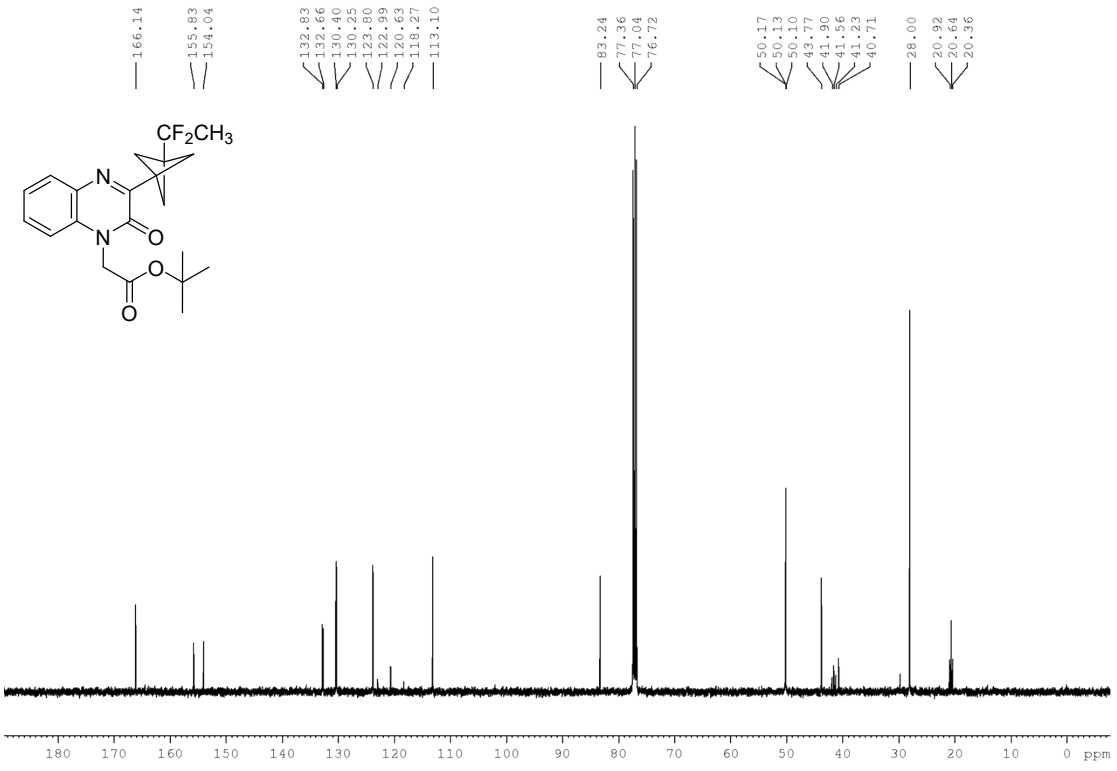
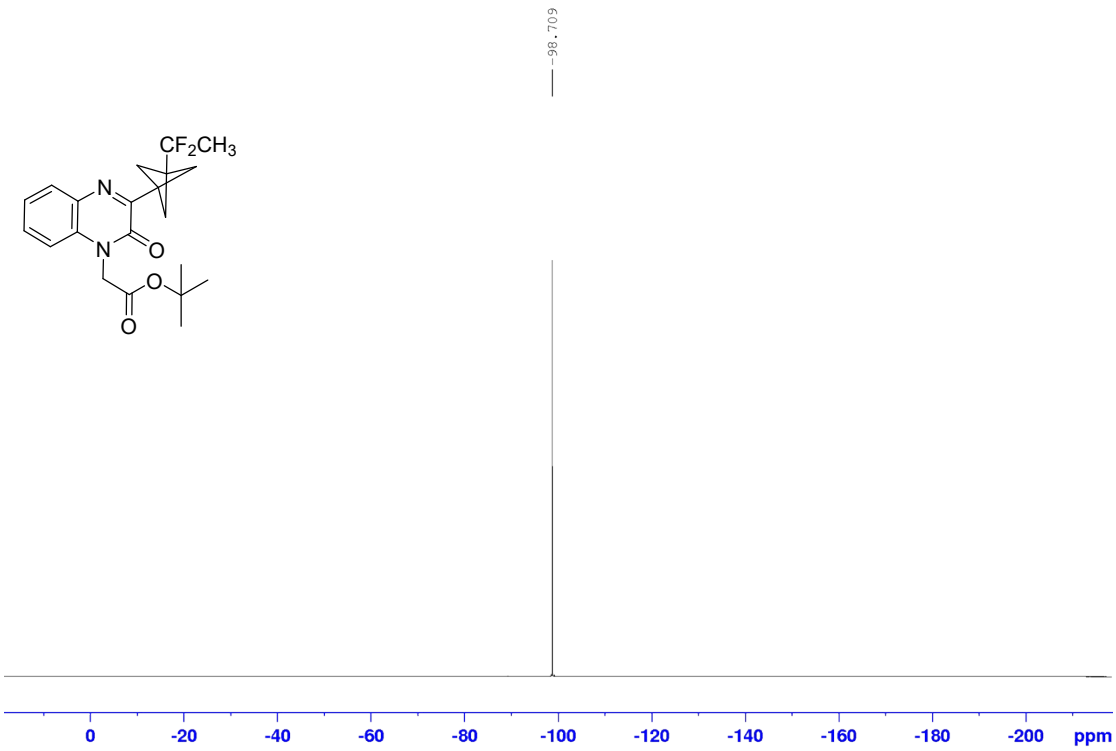


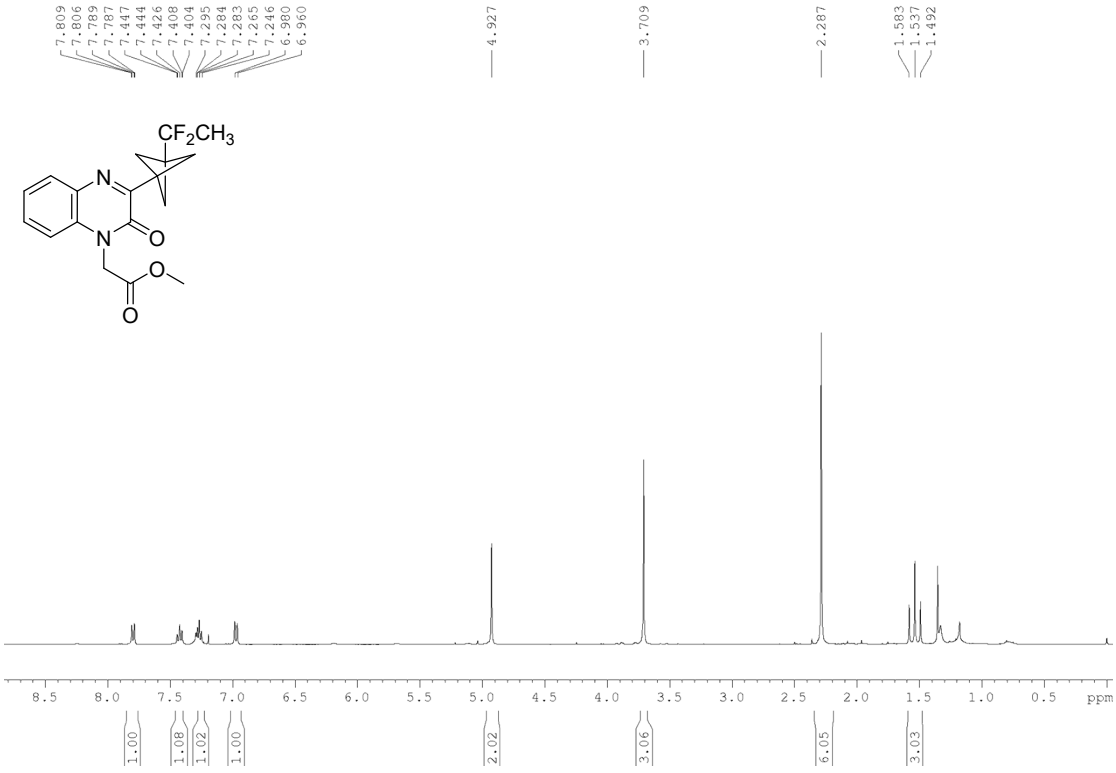

**4m $^{13}$C NMR (100 MHz, $CDCl_3$)**




**4m $^{19}$F NMR (376 MHz, $CDCl_3$)**




**4n $^{1}$H NMR (400 MHz, $CDCl_3$)**

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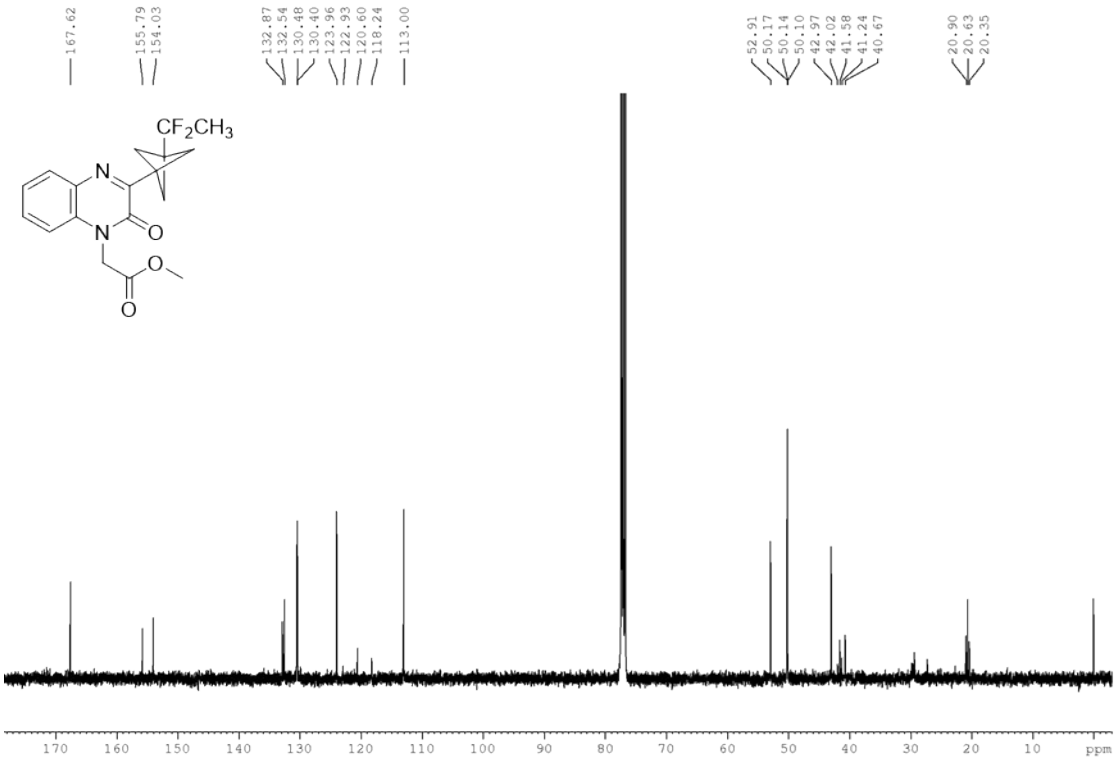


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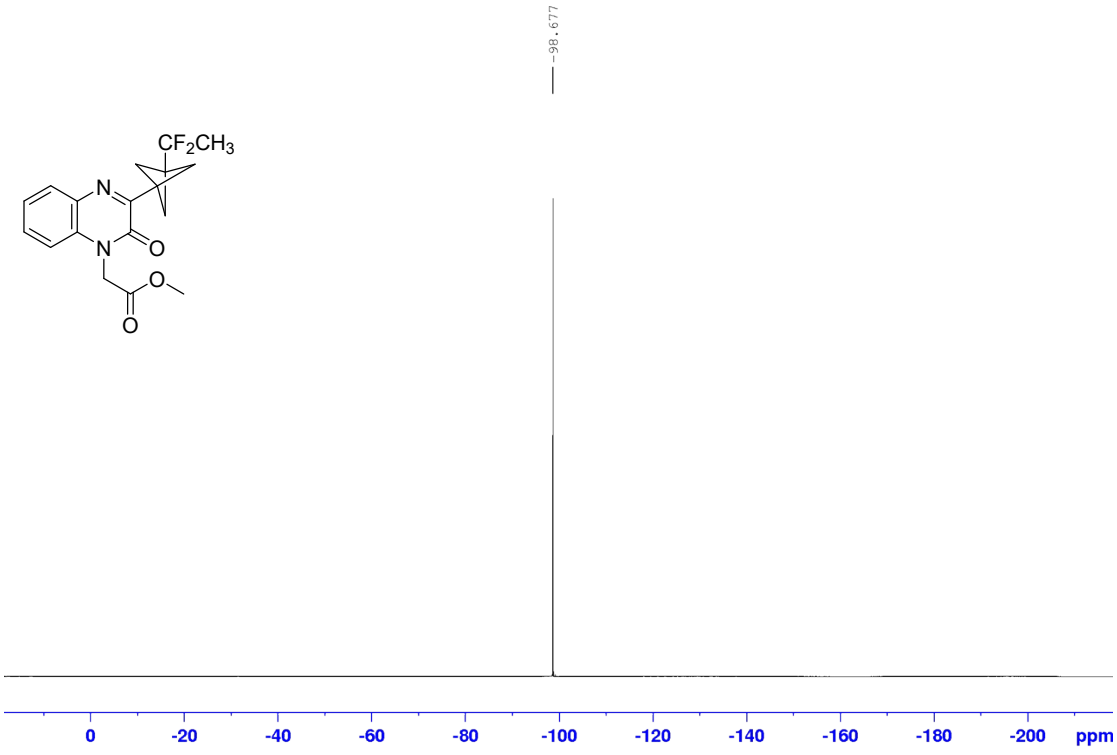


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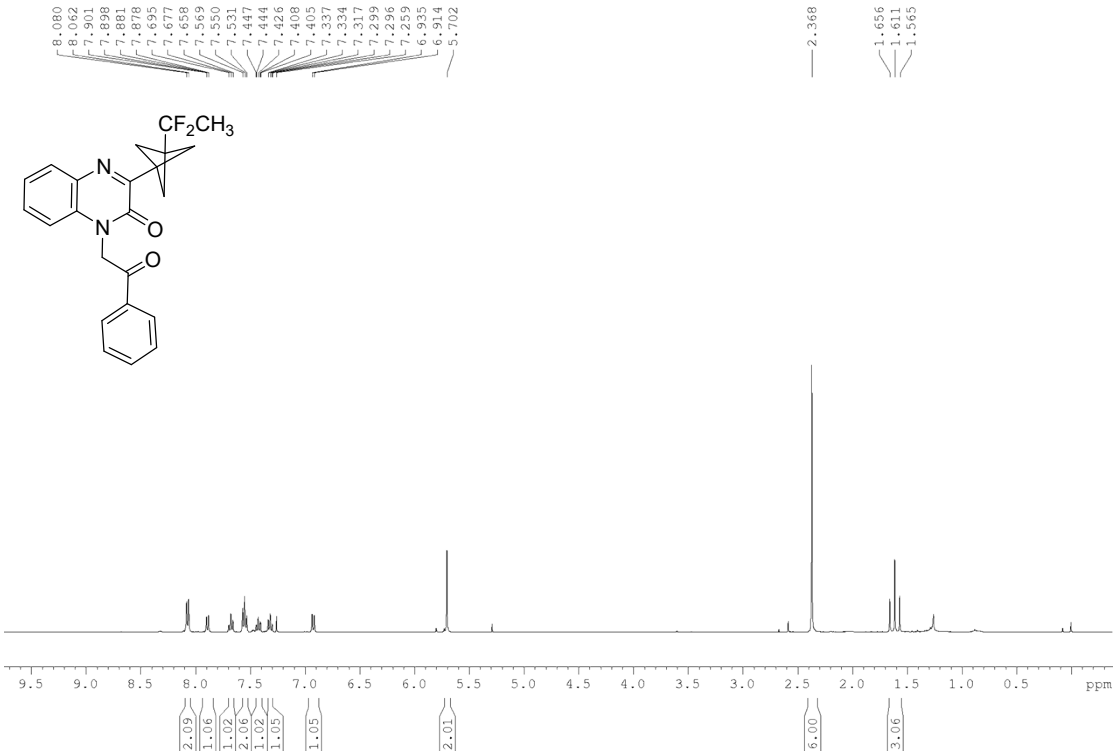


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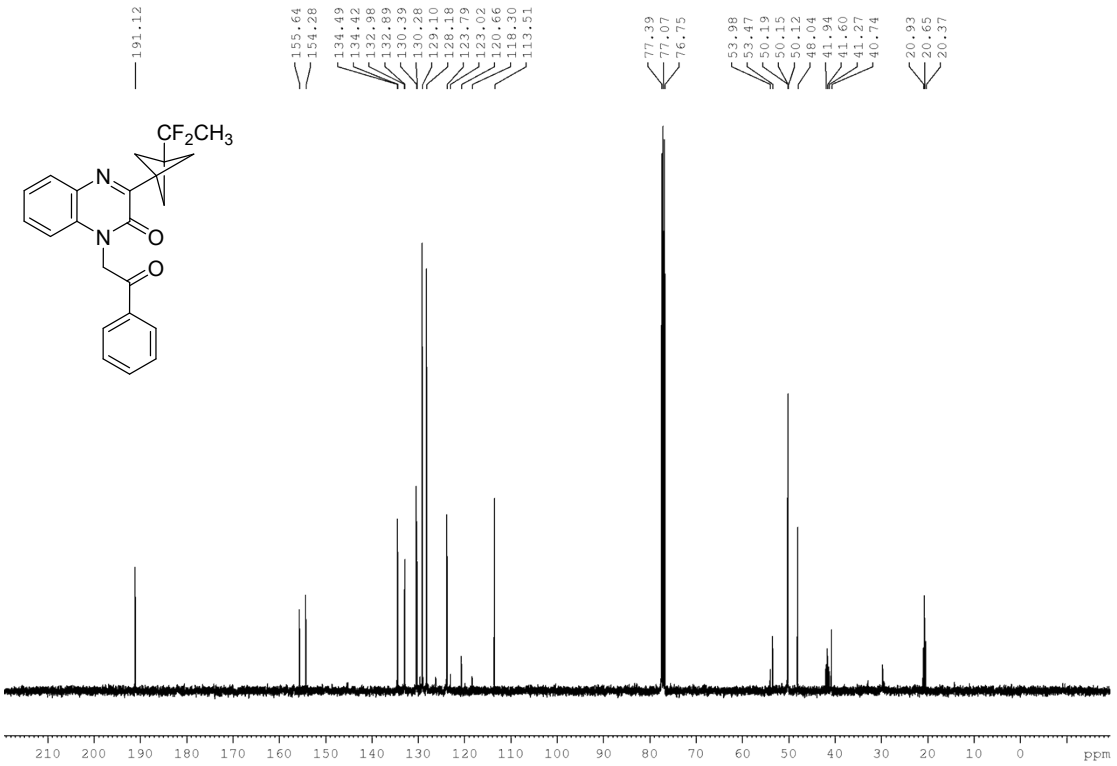
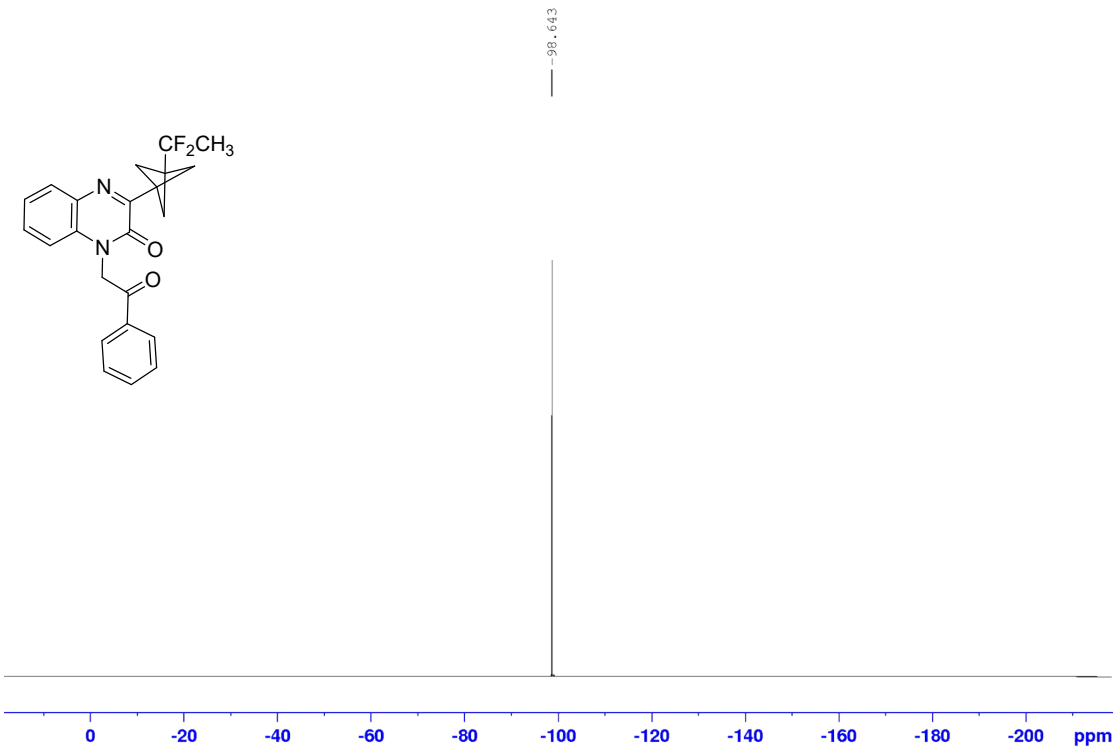
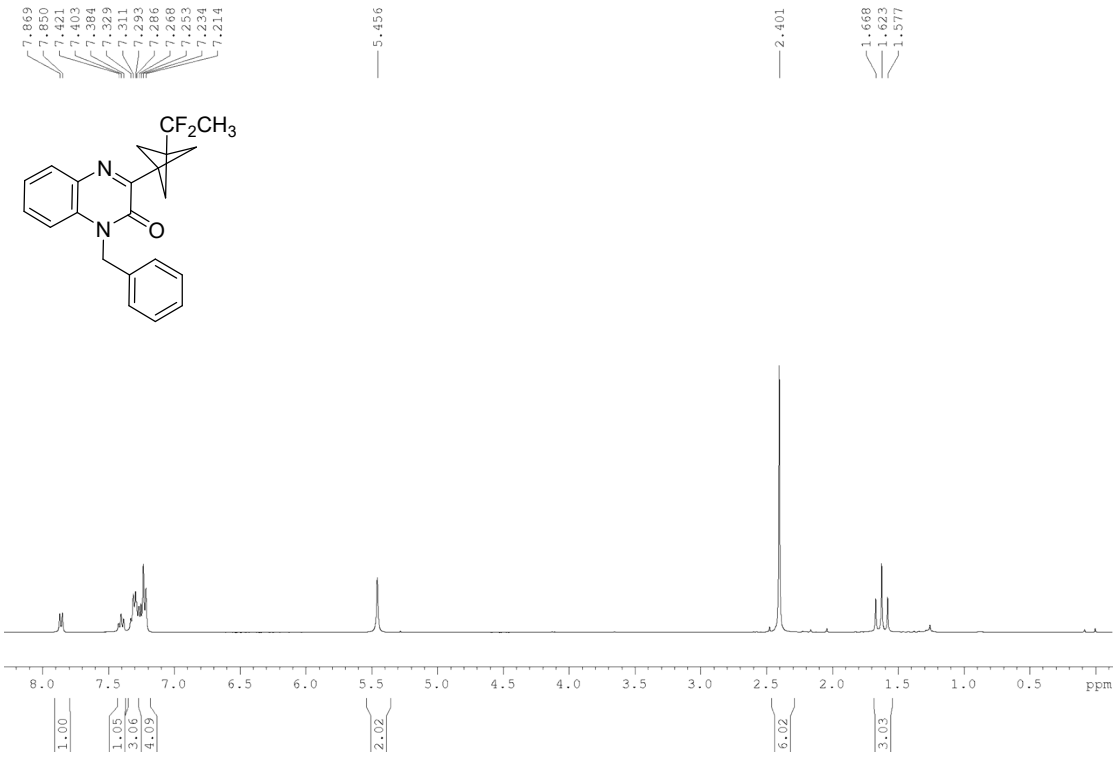


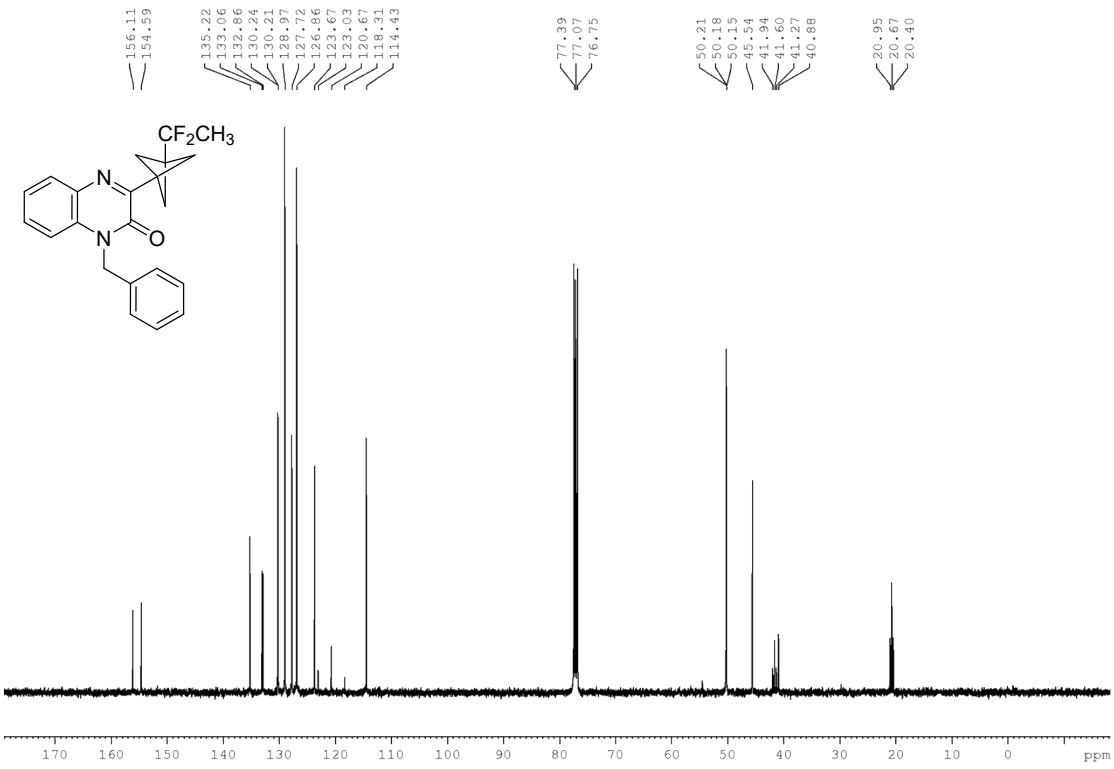

**4o $^{19}$F NMR (376 MHz, $CDCl_3$)**



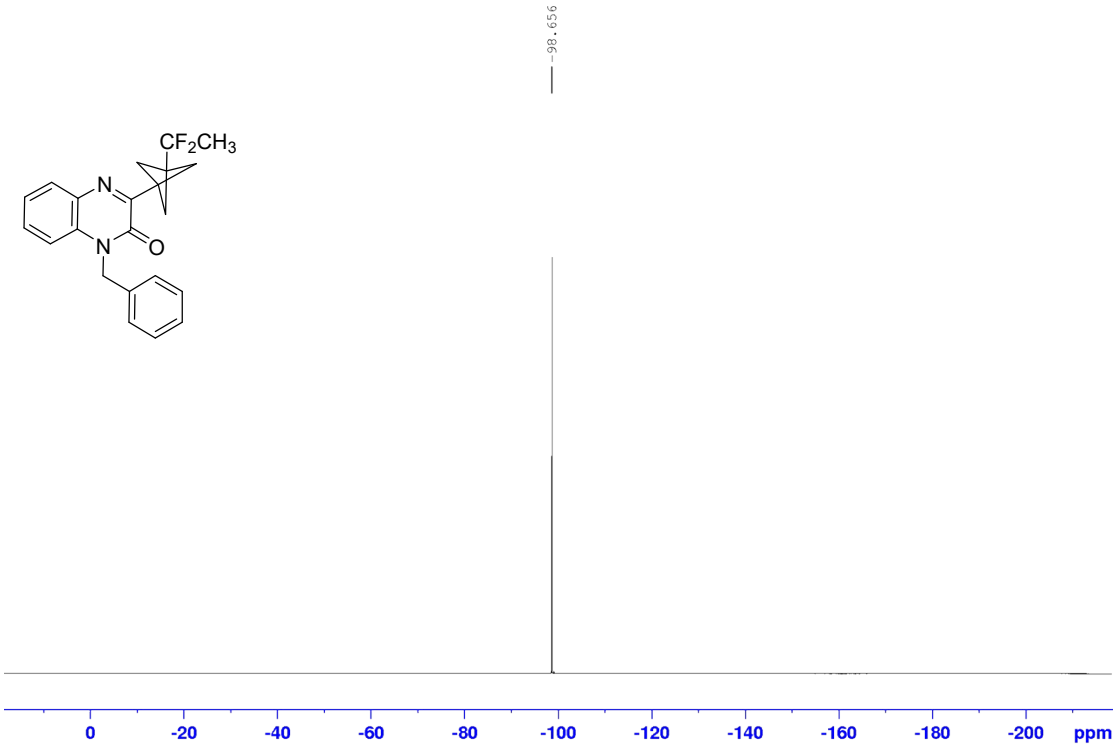

**4p $^{1}$H NMR (400 MHz, $CDCl_3$)**



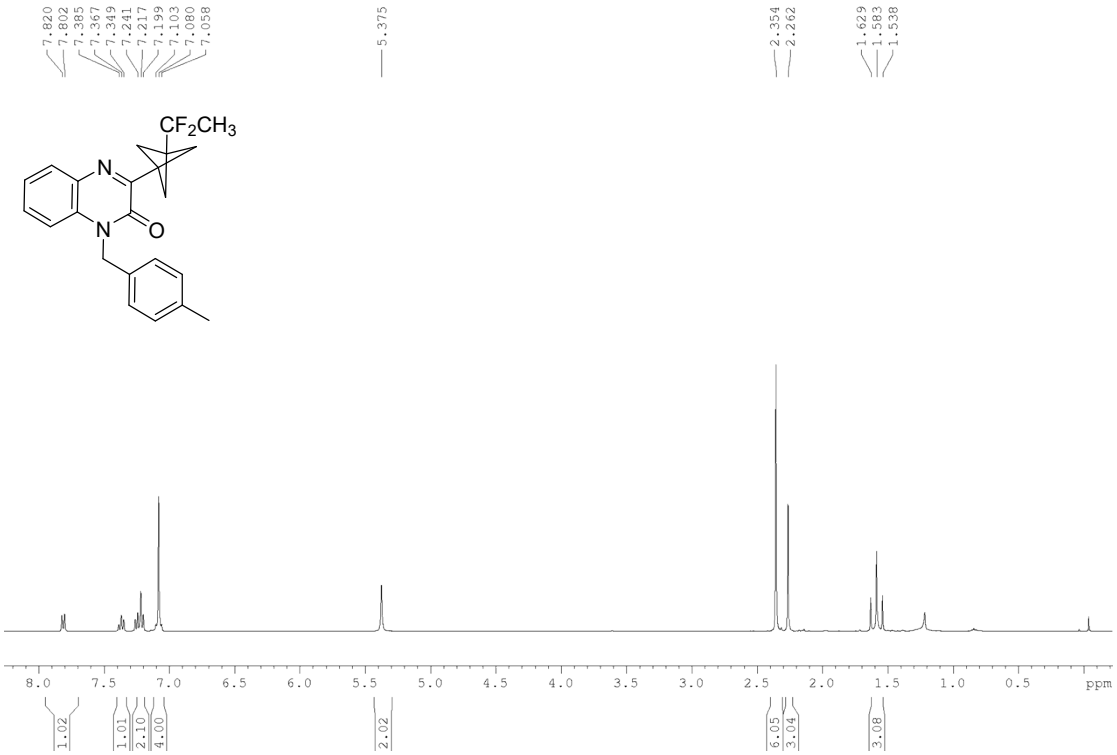

**4p $^{13}$C NMR (100 MHz, $CDCl_3$)**



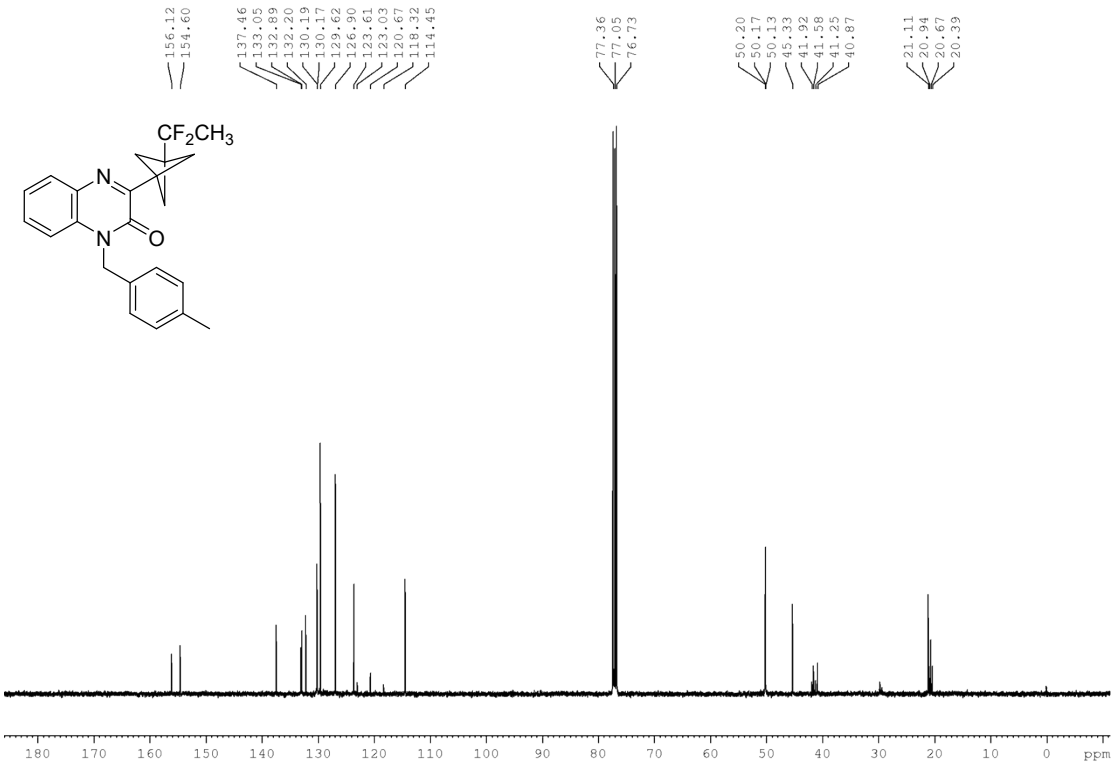

**4p $^{19}$F NMR (376 MHz, $CDCl_3$)**



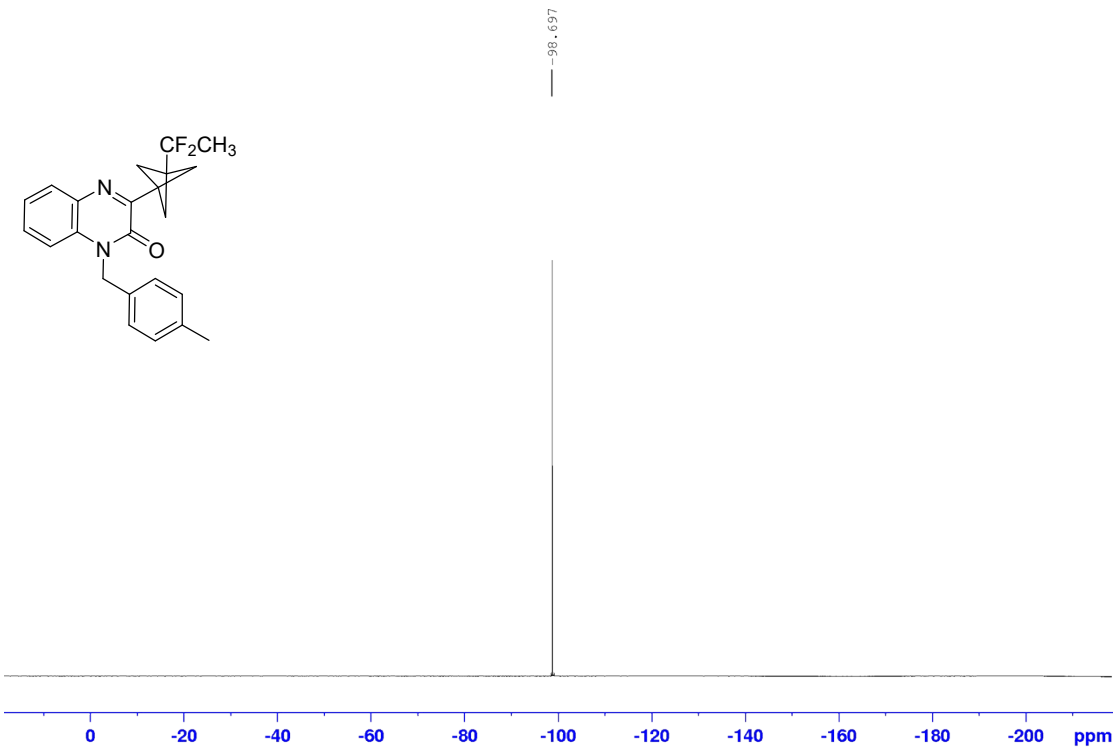

**4q $^{1}$H NMR (400 MHz, $CDCl_3$)**



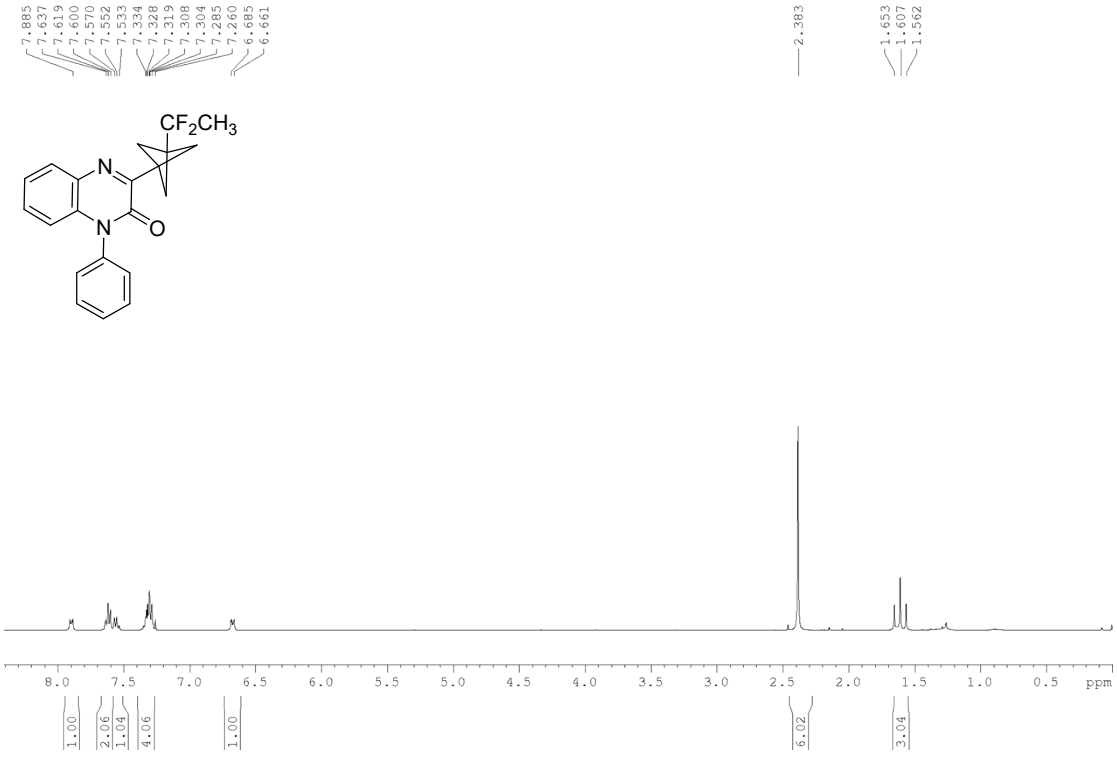

**4q $^{13}$C NMR (100 MHz, $CDCl_3$)**



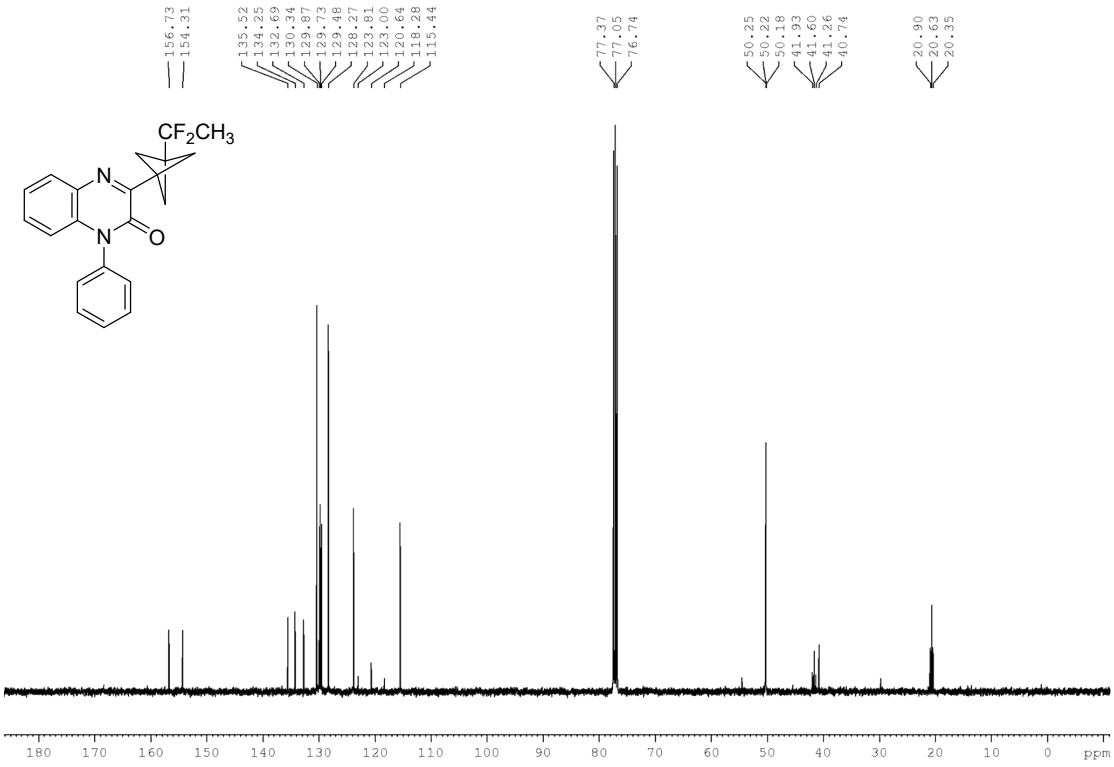

**4q $^{19}$F NMR (376 MHz, $CDCl_3$)**



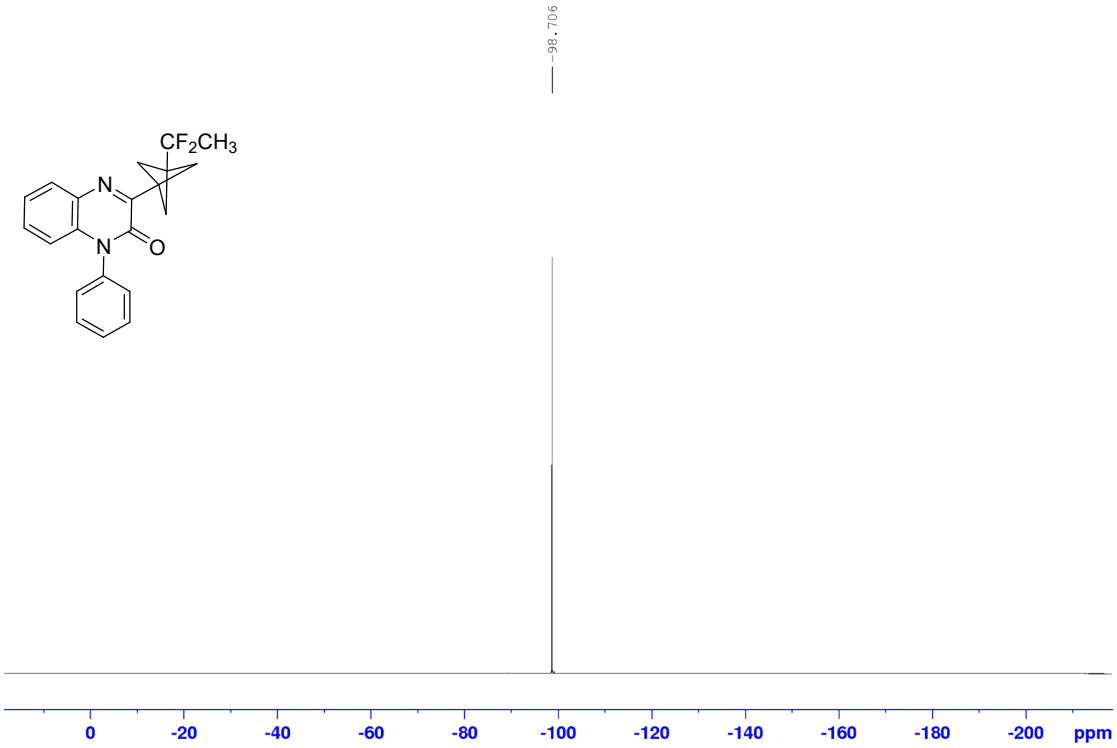

**4r $^{1}$H NMR (400 MHz, $CDCl_3$)**



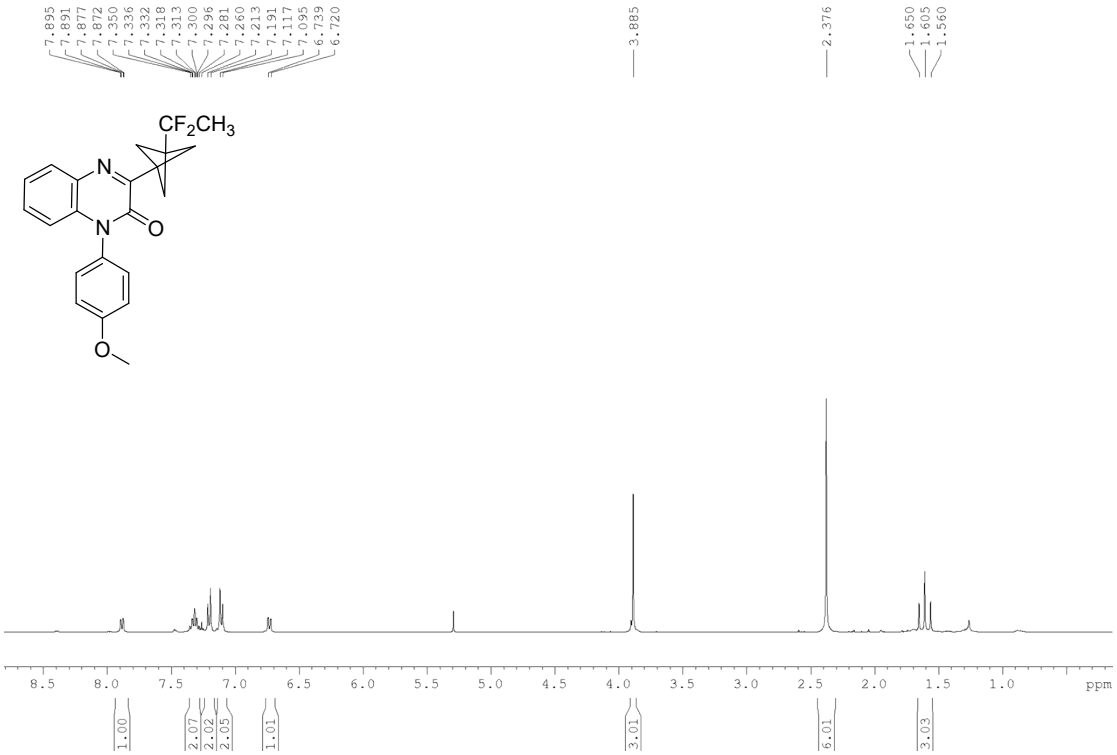

**4r $^{13}$C NMR (100 MHz, $CDCl_3$)**



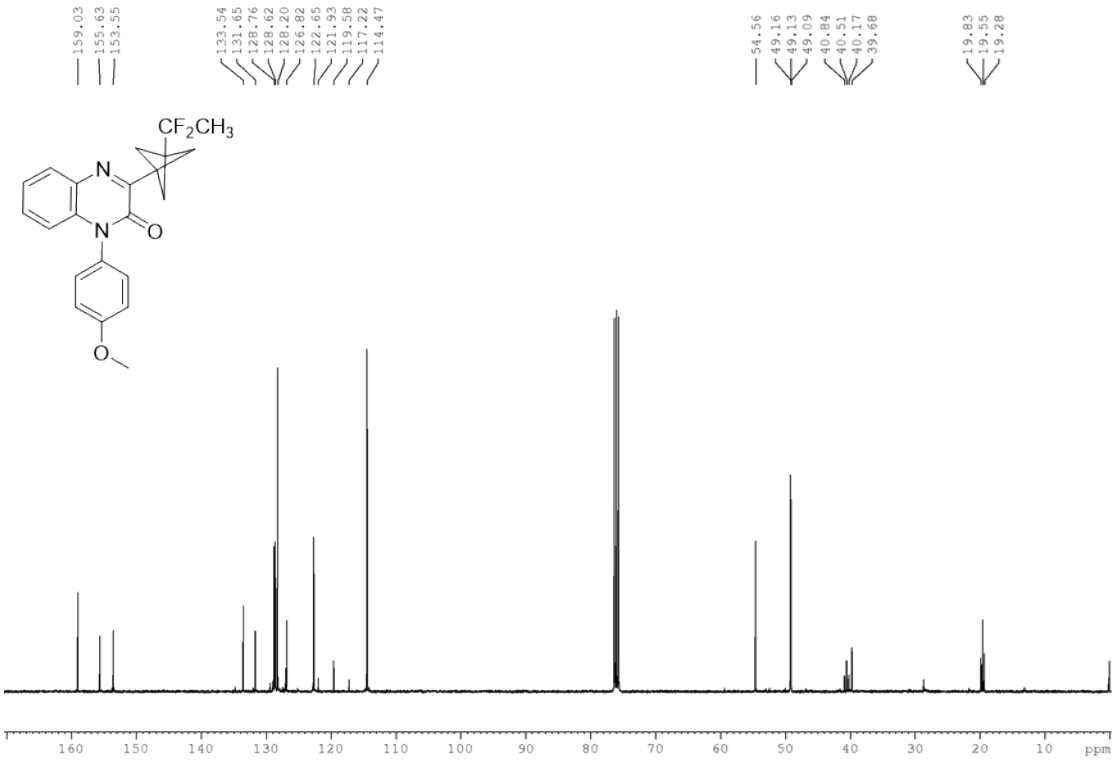

**4r $^{19}$F NMR (376 MHz, $CDCl_3$)**



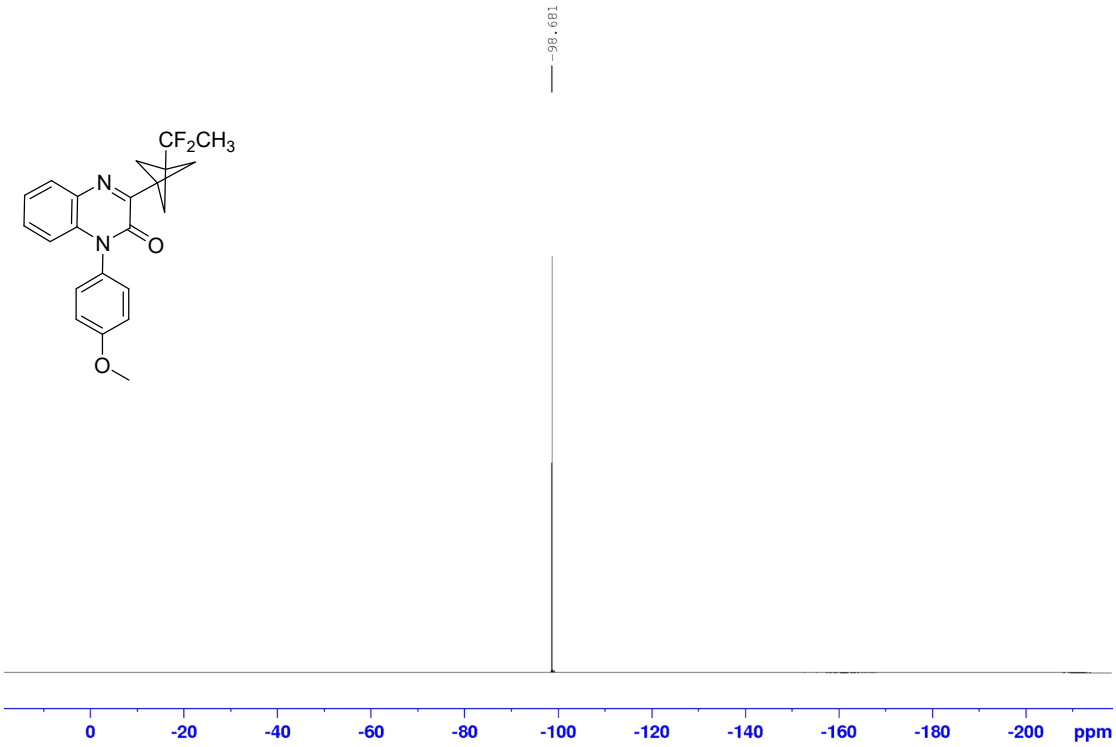

**4s $^{1}$H NMR (400 MHz, $CDCl_3$)**



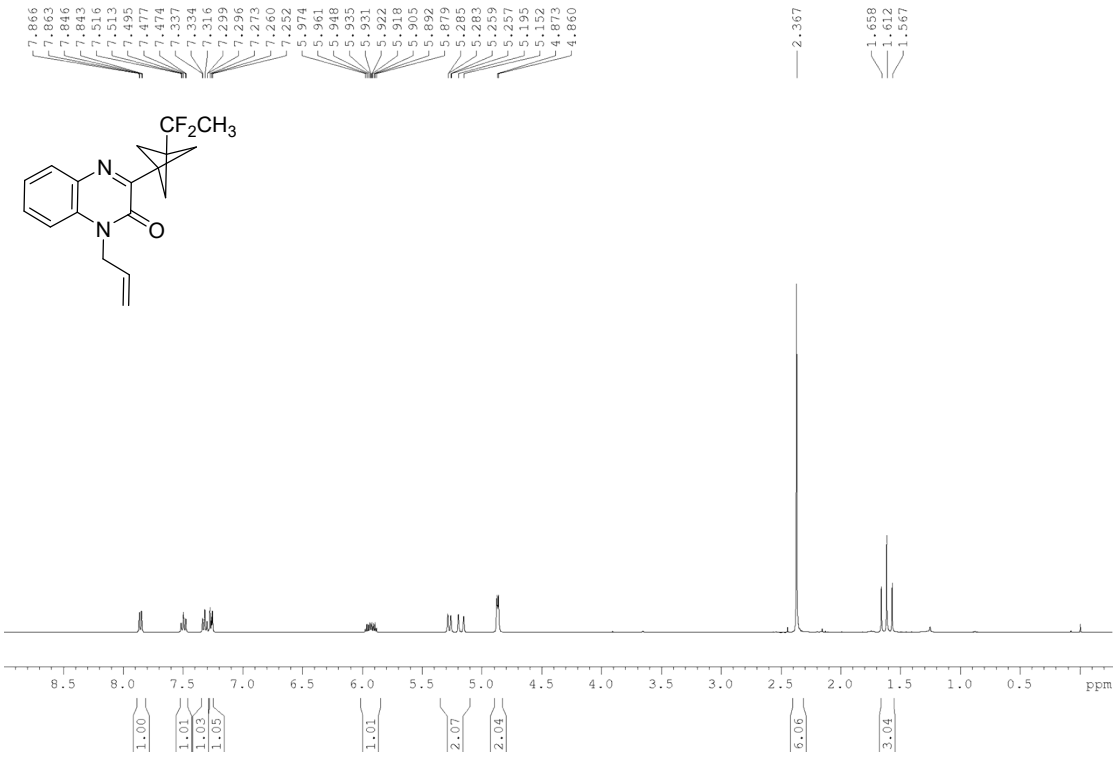

**4s $^{13}$C NMR (100 MHz, $CDCl_3$)**



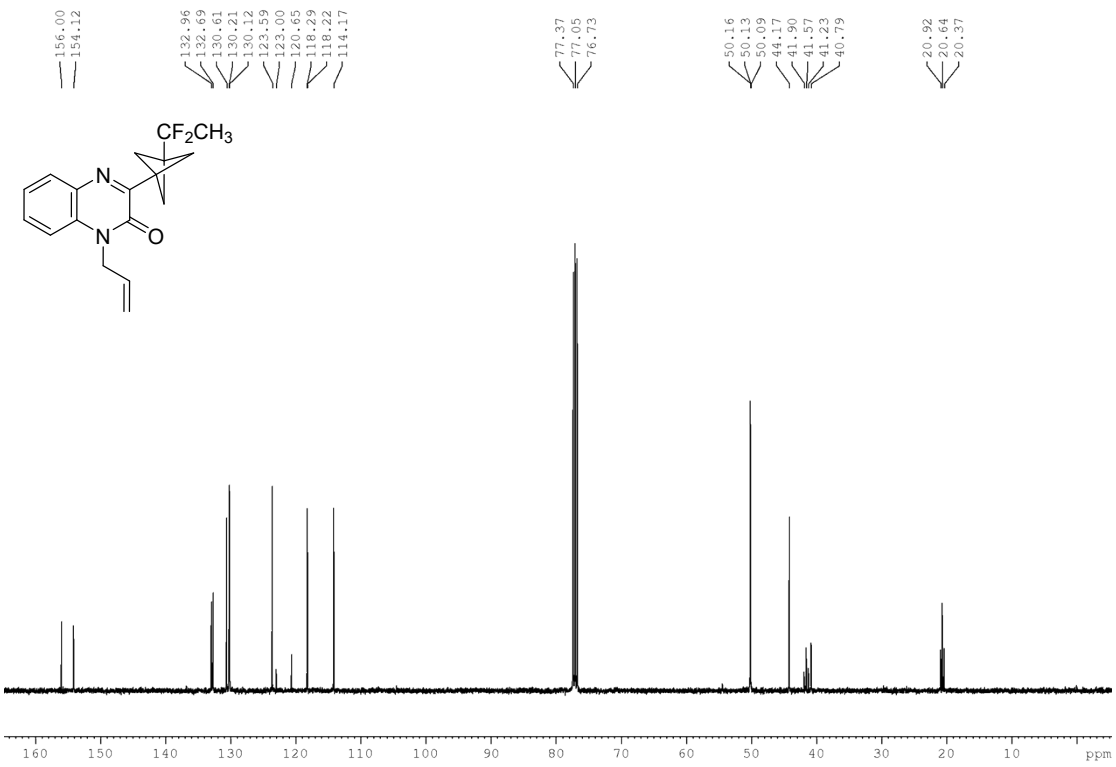

**4s $^{19}$F NMR (376 MHz, $CDCl_3$)**



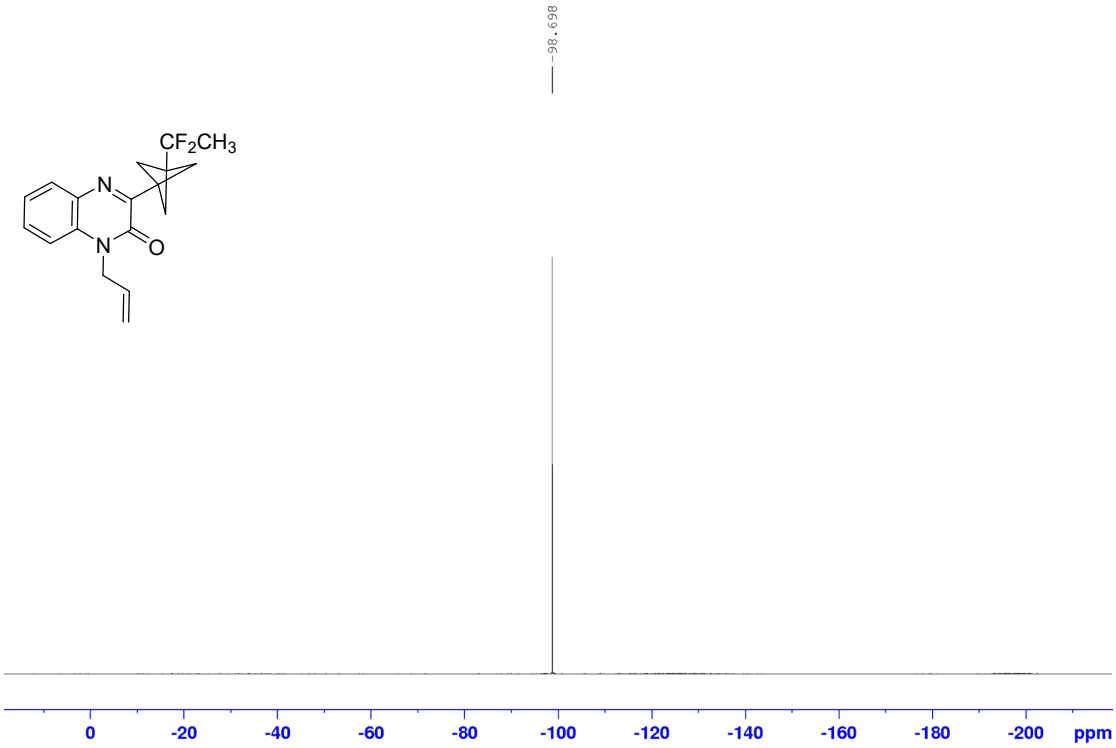

**4t $^{1}$H NMR (400 MHz, $CDCl_3$)**



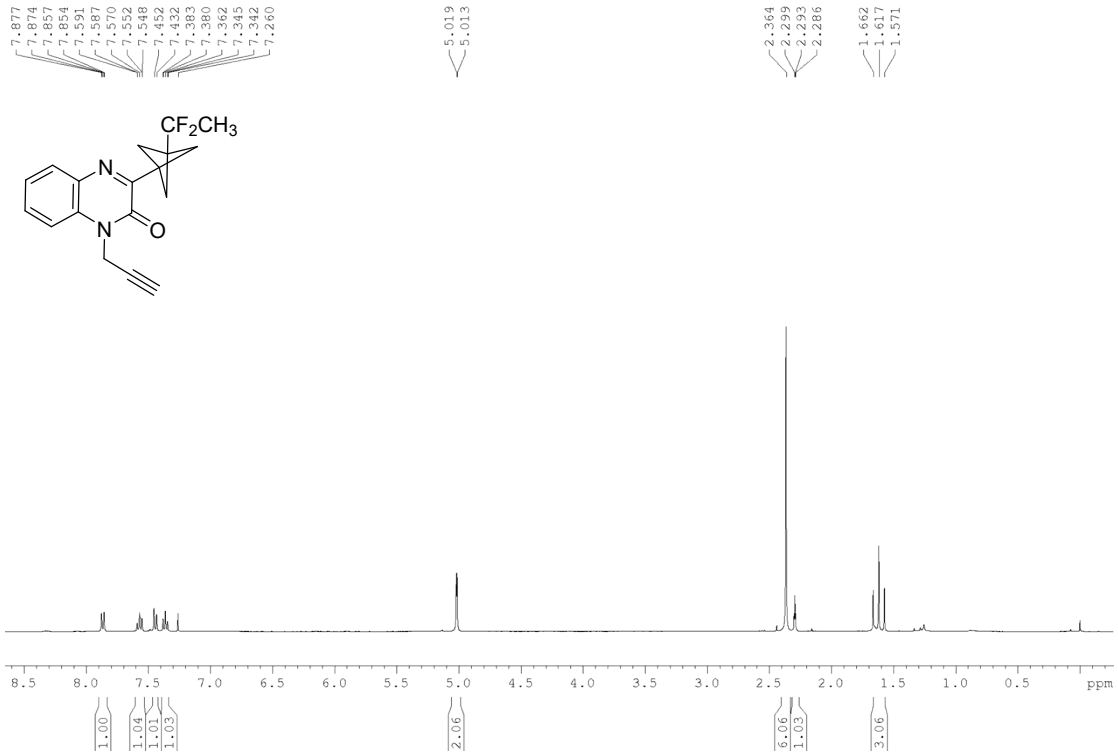

**4t $^{13}$C NMR (100 MHz, $CDCl_3$)**



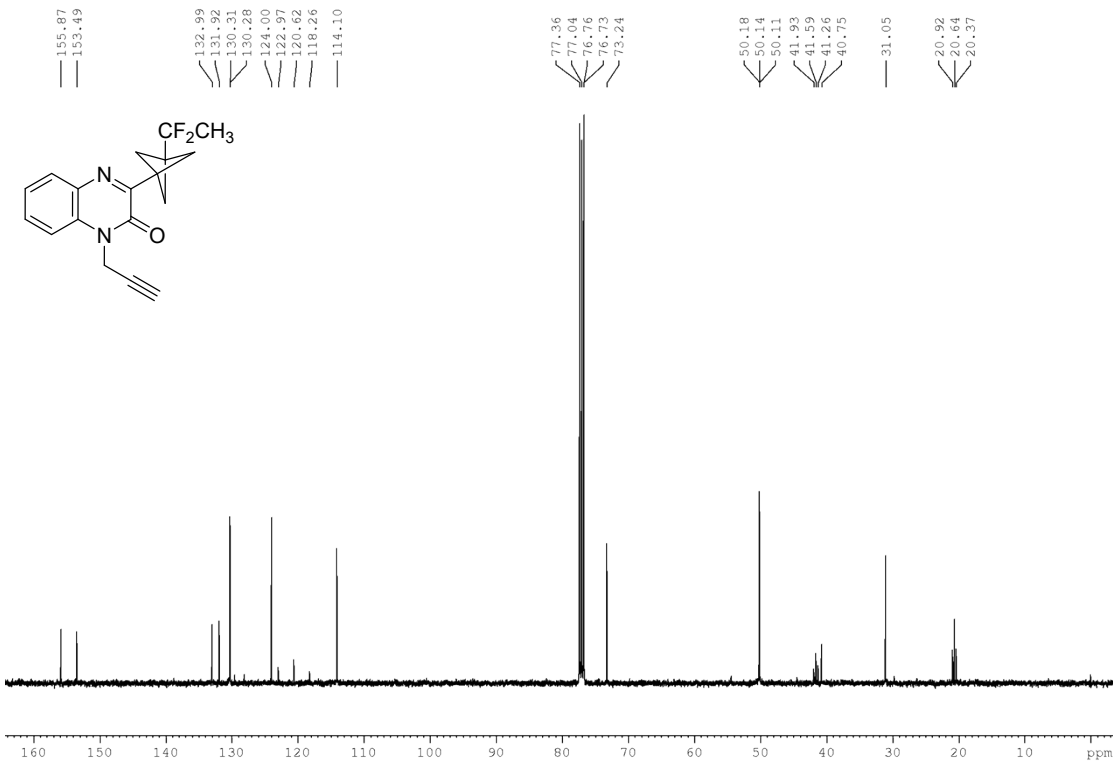

**4t $^{19}$F NMR (376 MHz, $CDCl_3$)**



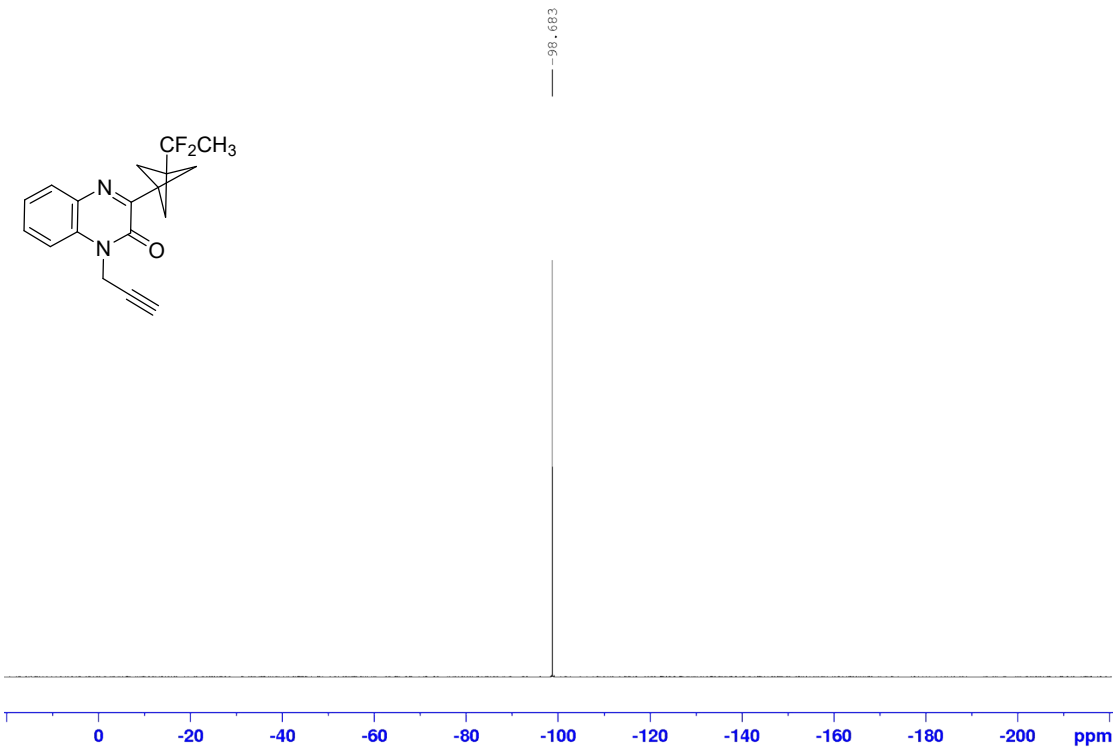

**4u $^{1}$H NMR (400 MHz, $CDCl_3$)**



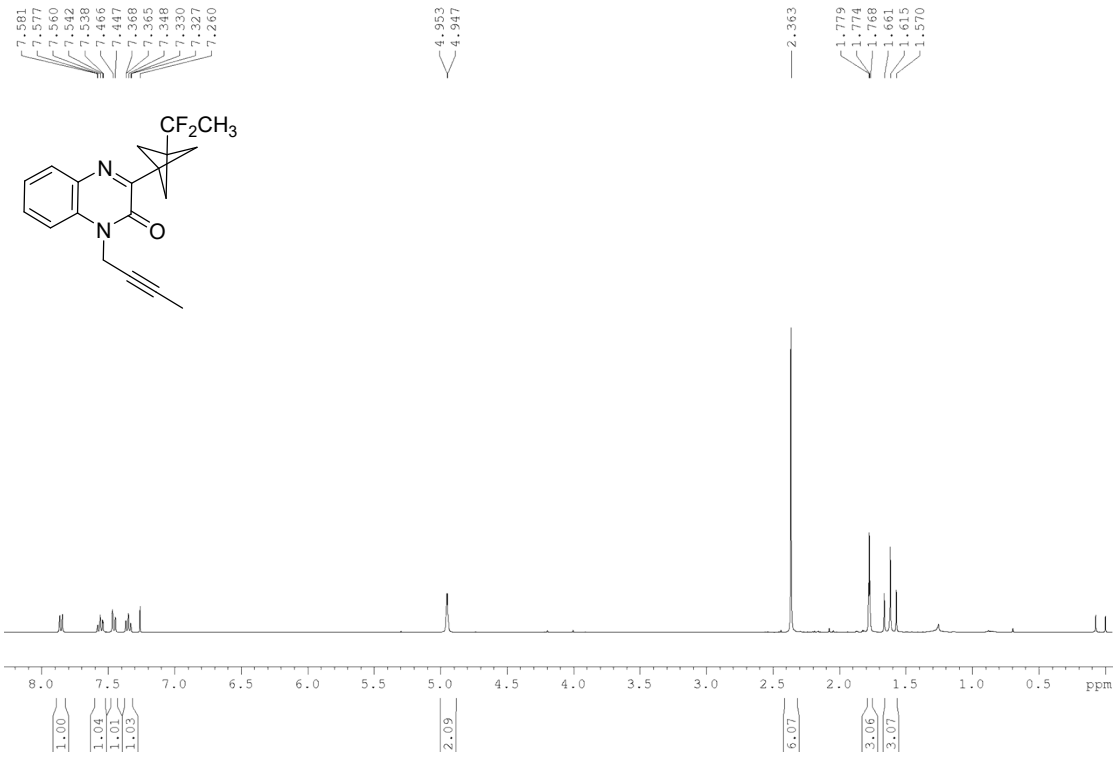

**4u $^{13}$C NMR (100 MHz, $CDCl_3$)**



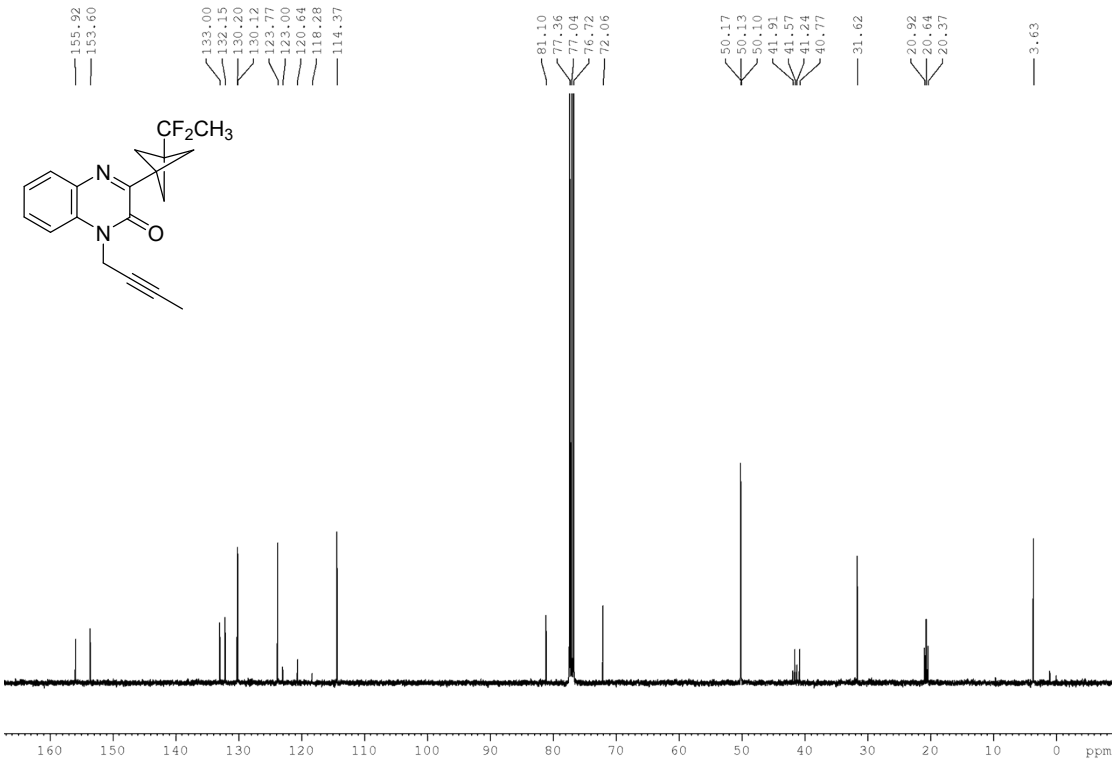

**4u $^{19}$F NMR (376 MHz, $CDCl_3$)**



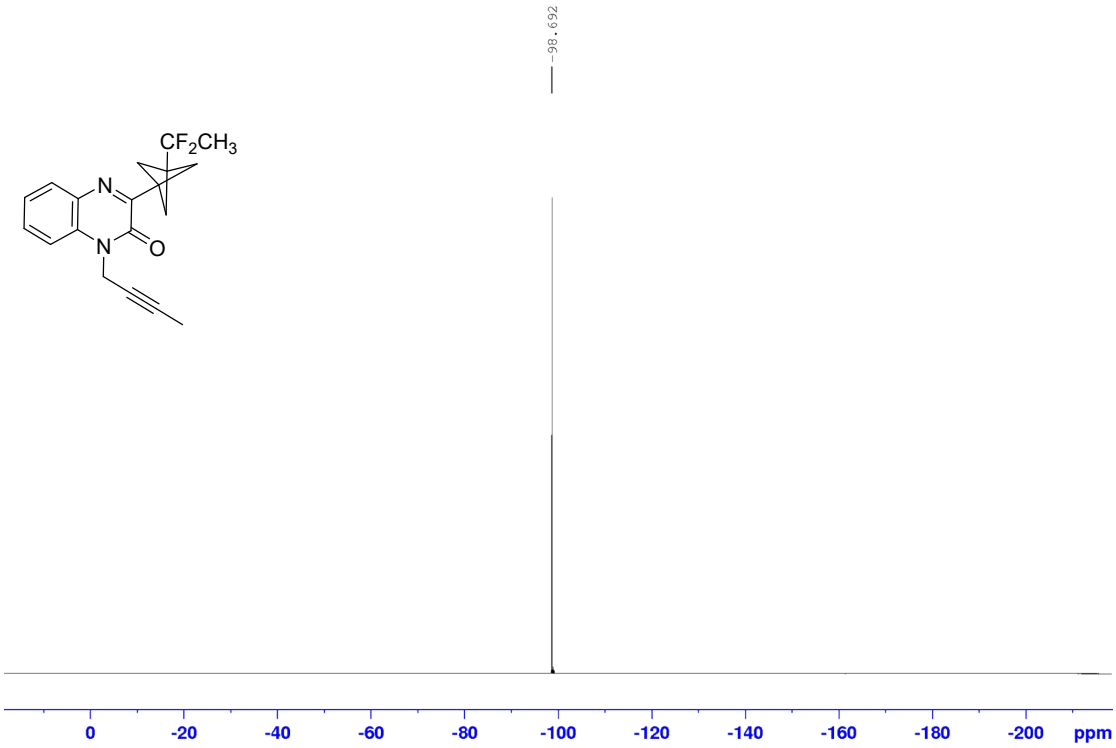

**4v $^{1}$H NMR (400 MHz, $CDCl_3$)**



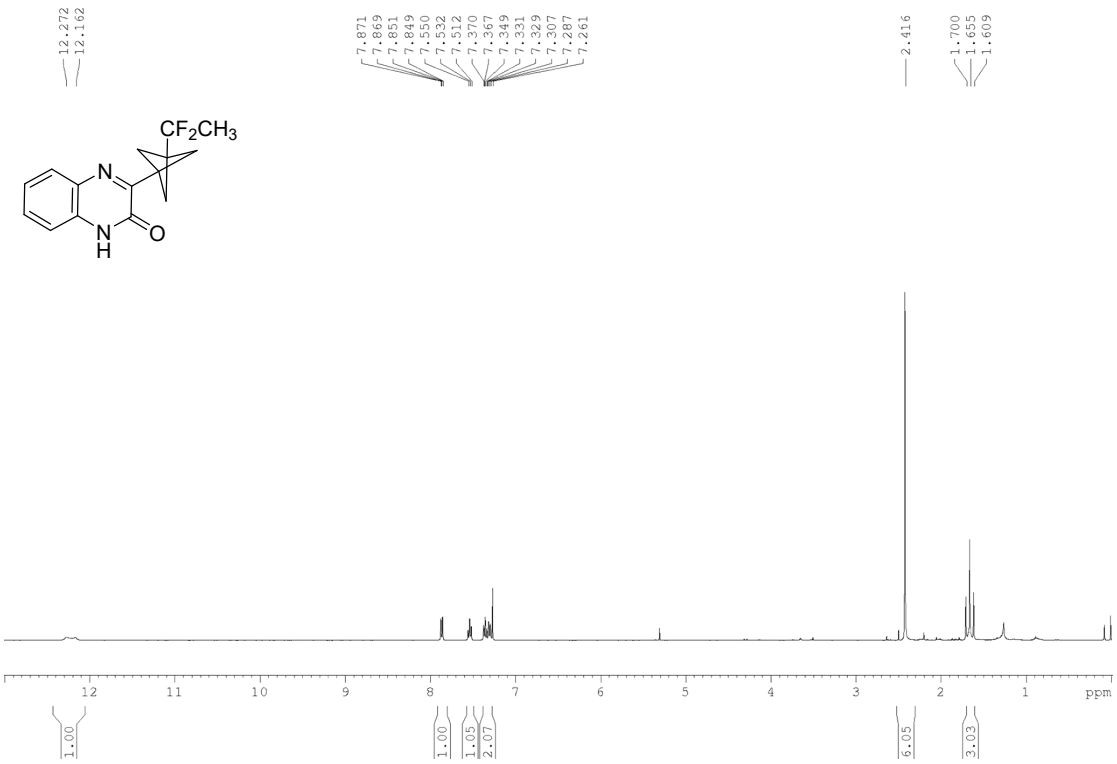

**4v $^{13}$C NMR (100 MHz, $CDCl_3$)**



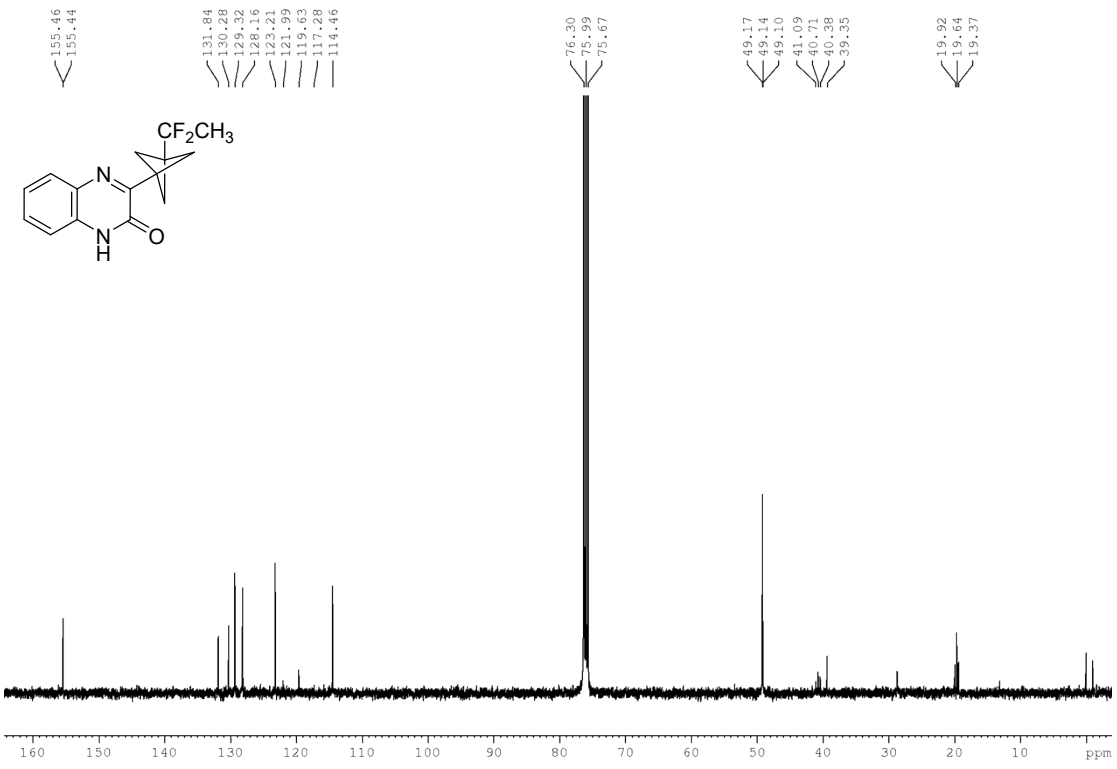

**4v $^{19}$F NMR (376 MHz, $CDCl_3$)**



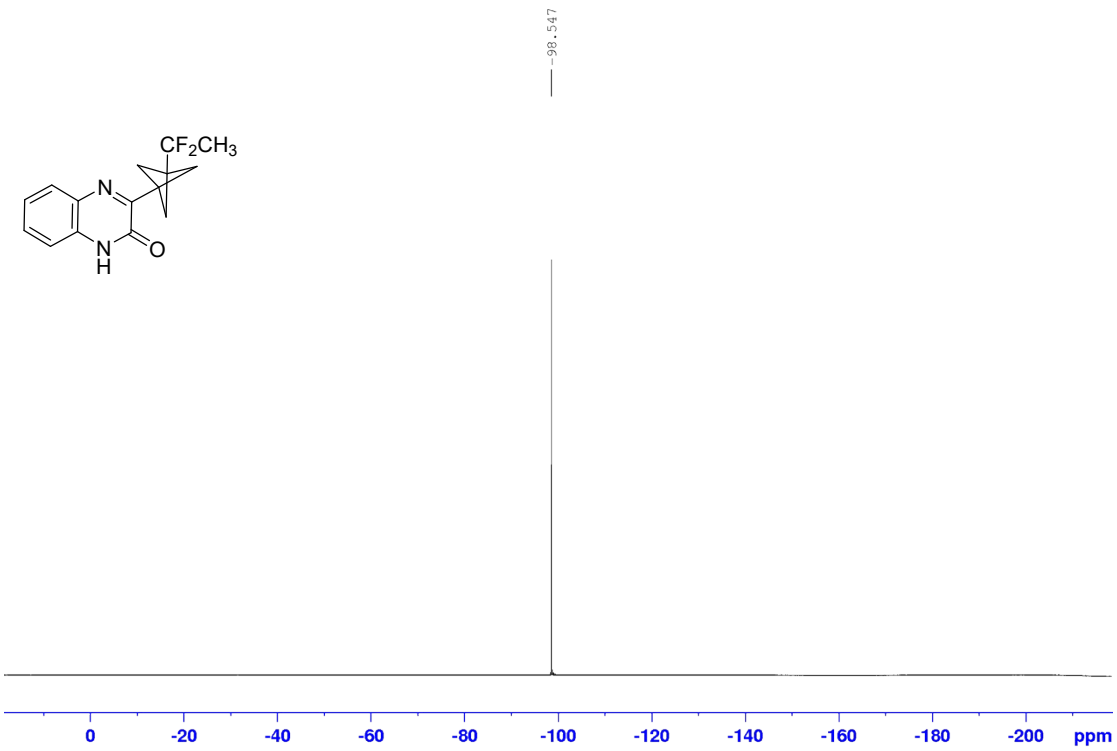

**4w $^{1}$H NMR (400 MHz, $CDCl_3$)**



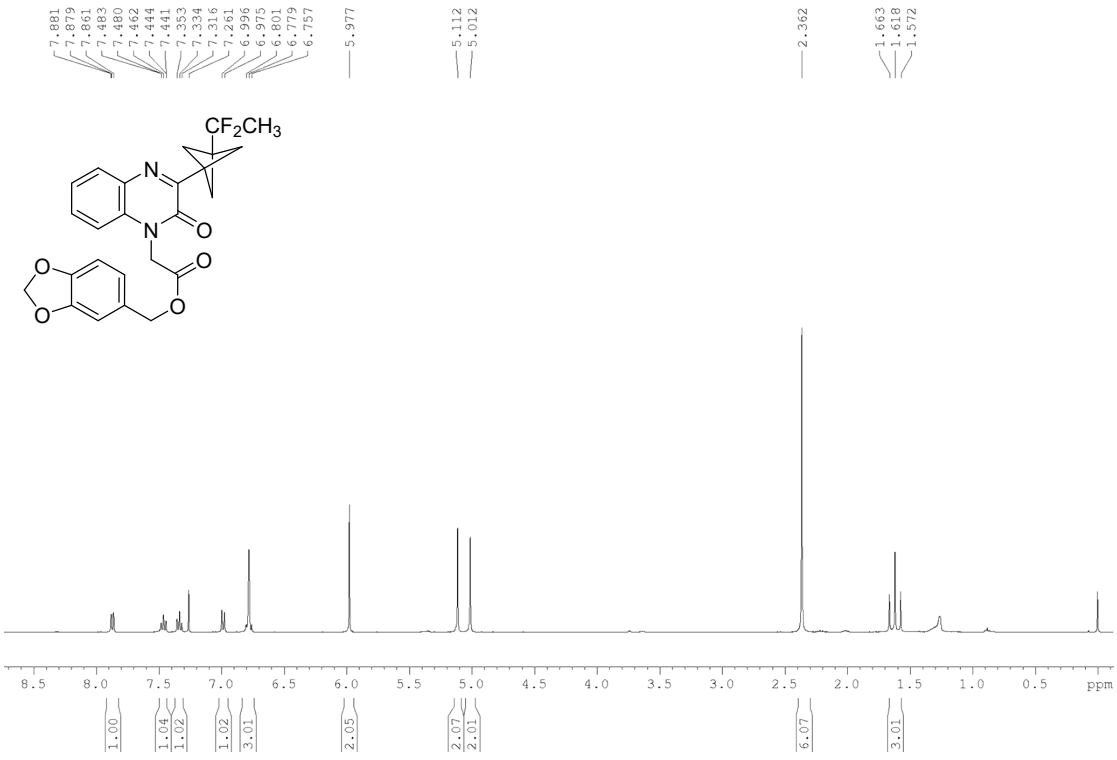

**4w $^{13}$C NMR (100 MHz, $CDCl_3$)**



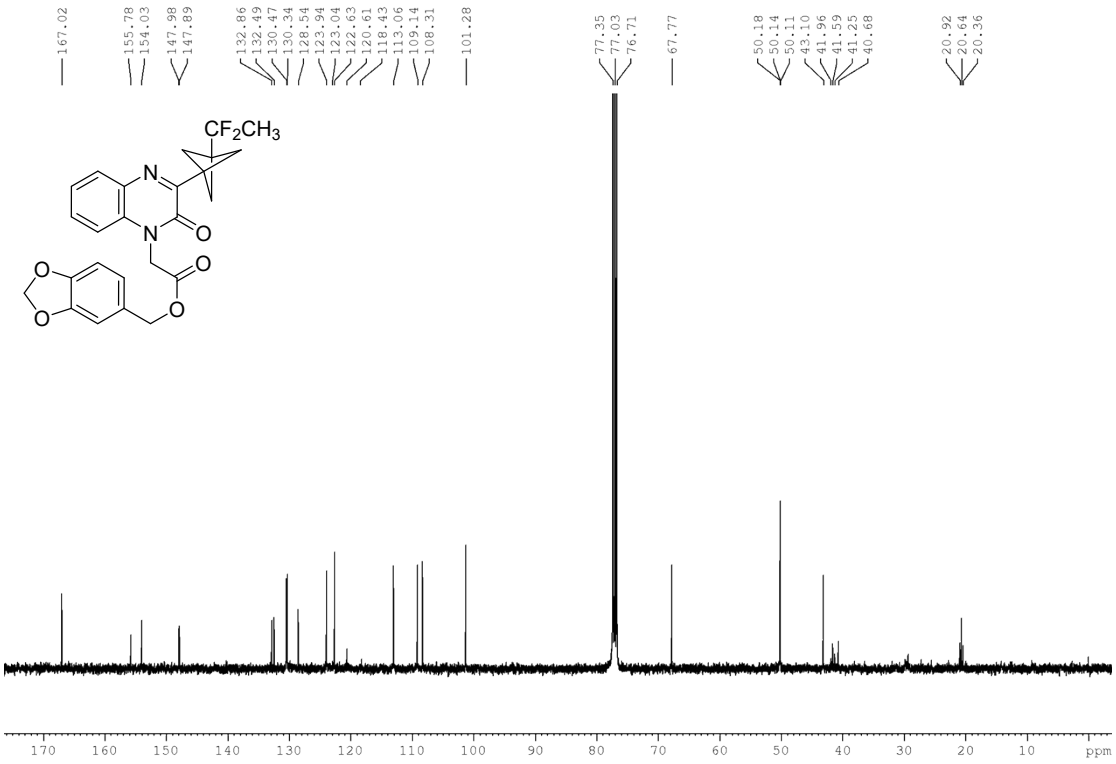

**4w $^{19}$F NMR (376 MHz, $CDCl_3$)**



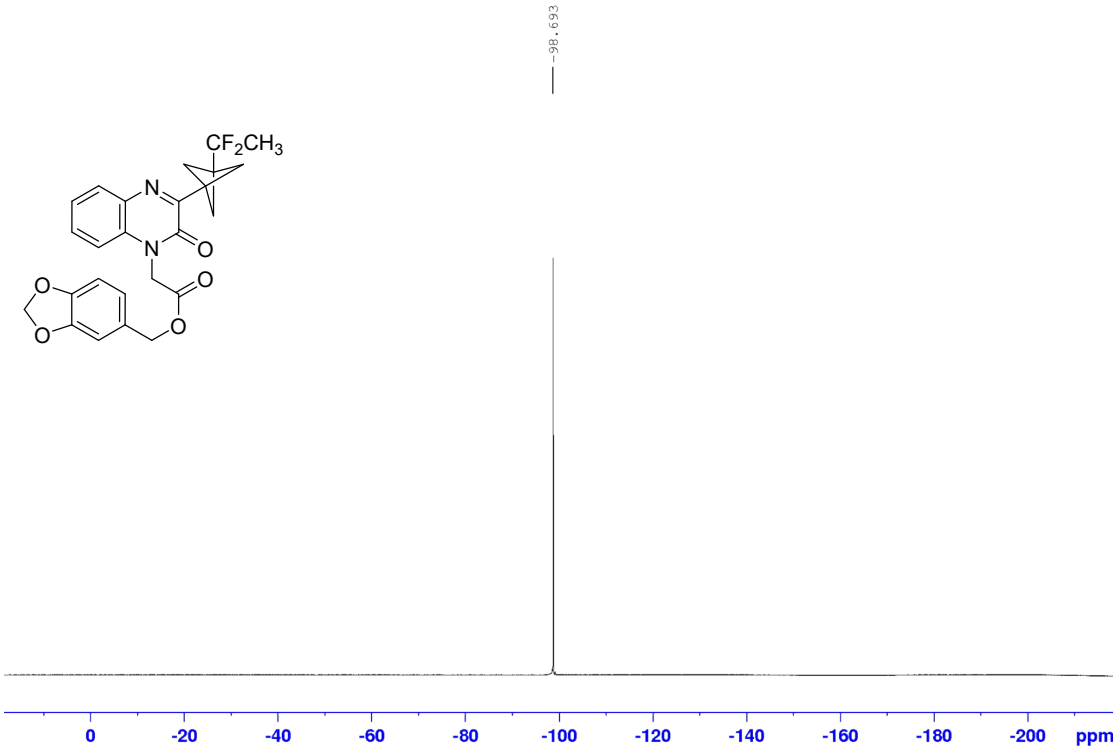

**4x $^{1}$H NMR (400 MHz, $CDCl_3$)**



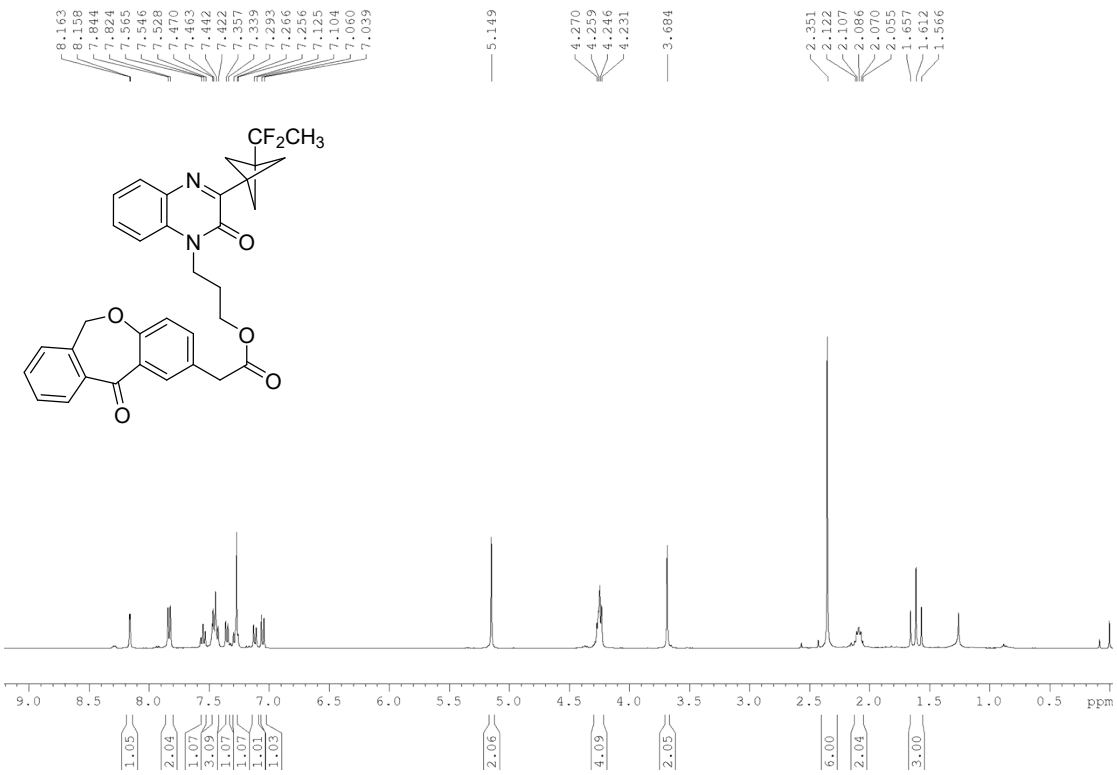

**4x $^{13}$C NMR (100 MHz, $CDCl_3$)**



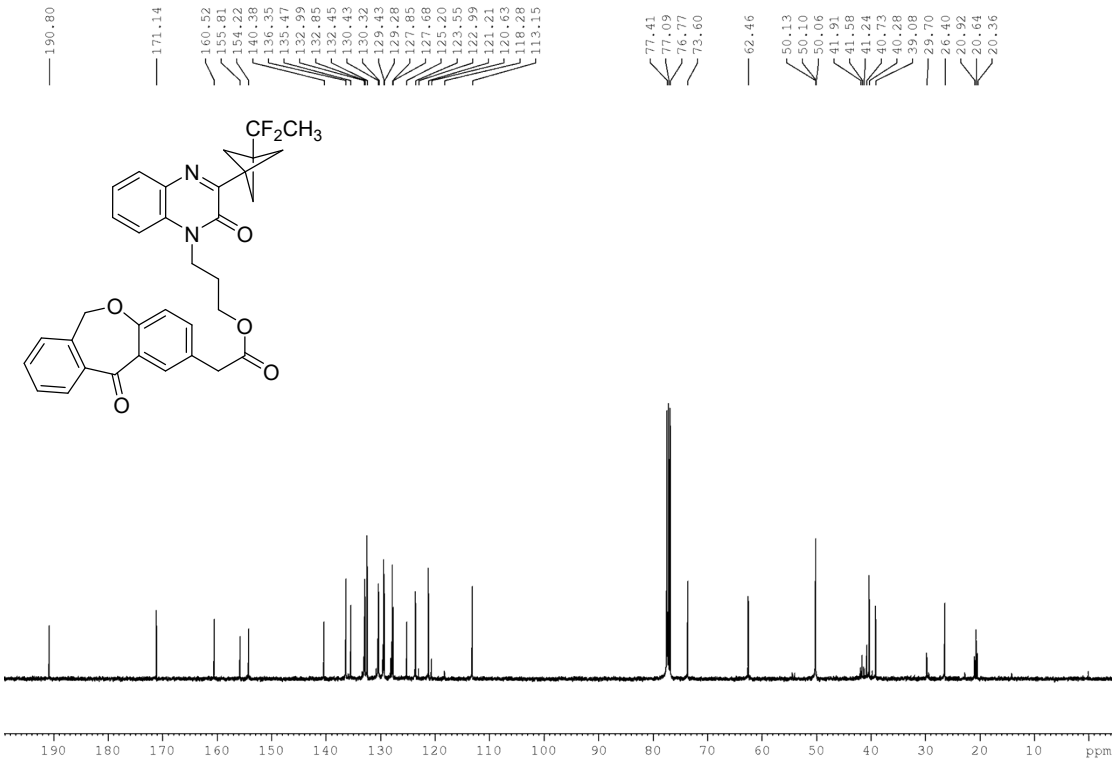

**4x $^{19}$F NMR (376 MHz, $CDCl_3$)**



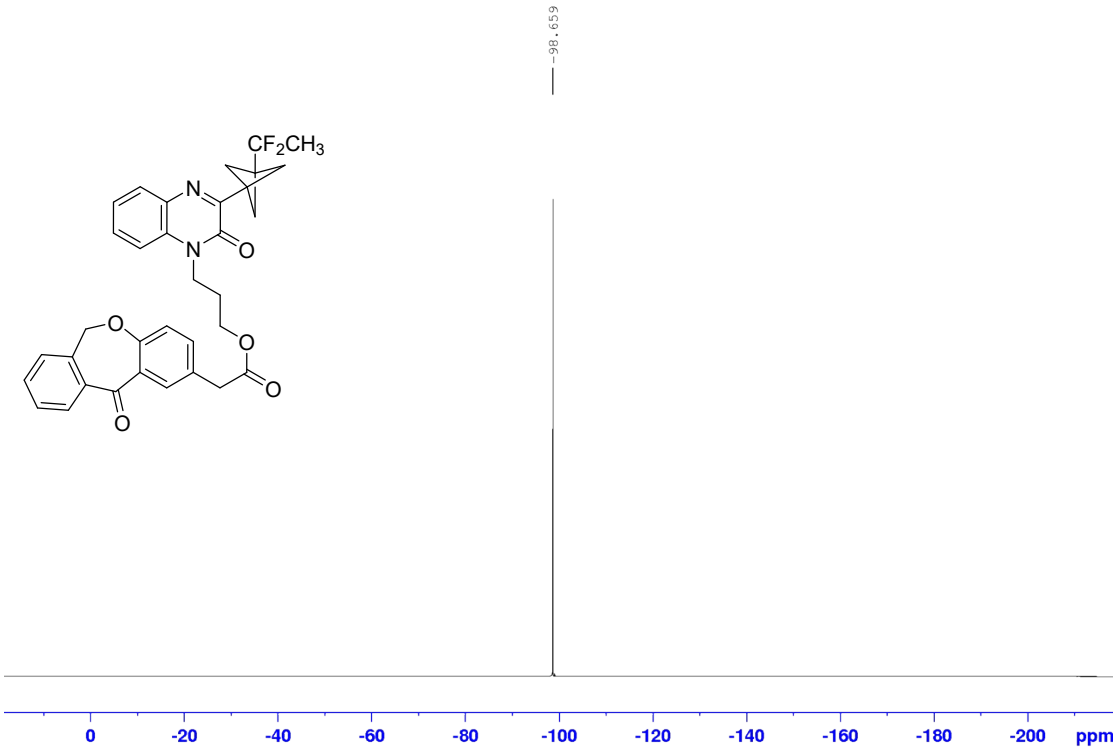

**4y $^{1}$H NMR (400 MHz, $CDCl_3$)**



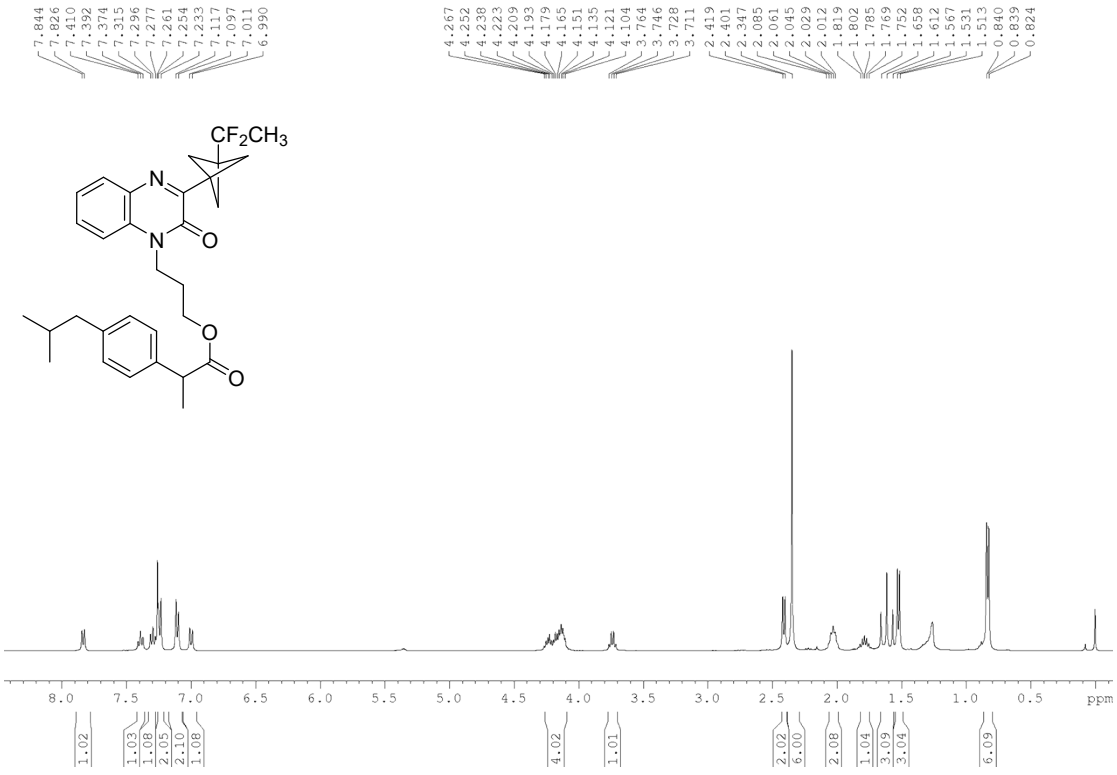

**4y $^{13}$C NMR (100 MHz, $CDCl_3$)**



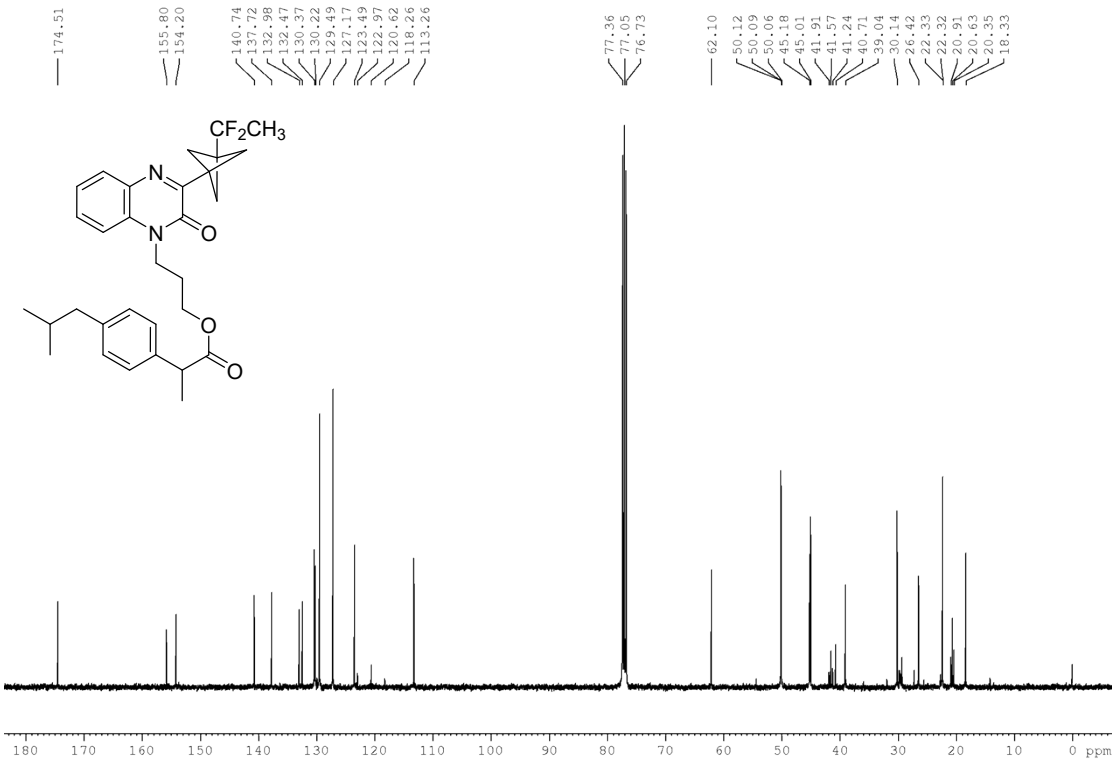

**4y $^{19}$F NMR (376 MHz, $CDCl_3$)**



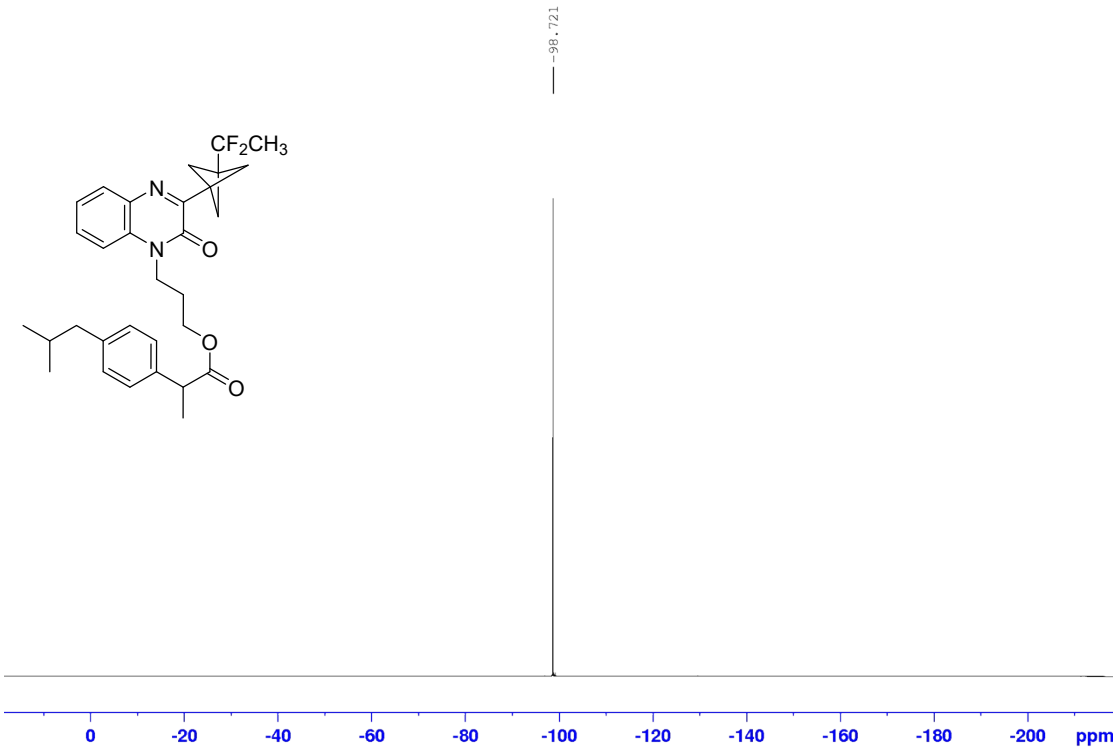

**4z $^{1}$H NMR (400 MHz, $CDCl_3$)**



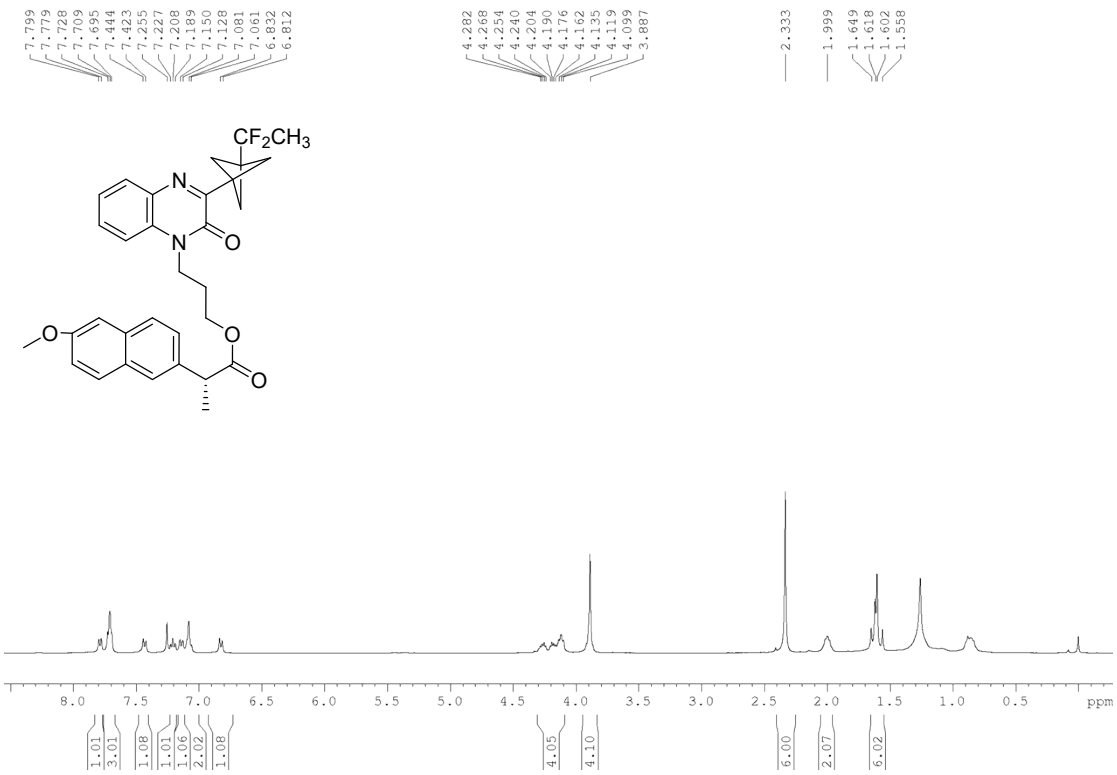

**4z $^{13}$C NMR (100 MHz, $CDCl_3$)**



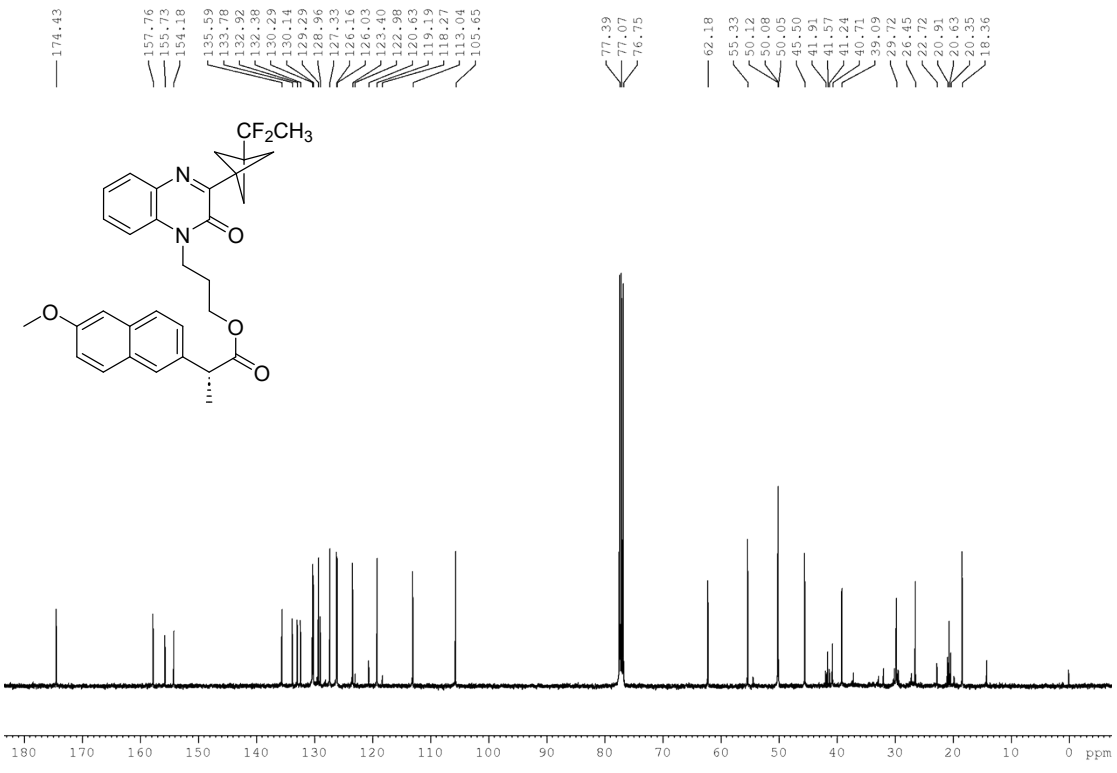

**4z $^{19}$F NMR (376 MHz, $CDCl_3$)**



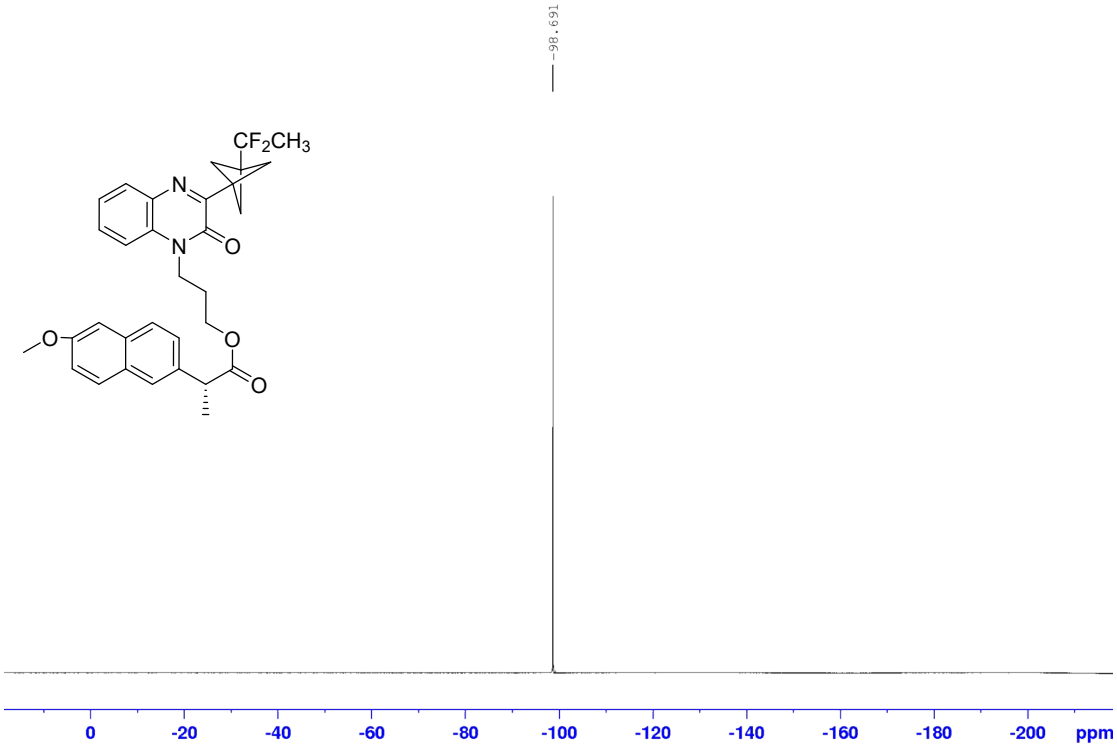

**4aa $^{1}$H NMR (400 MHz, $CDCl_3$)**



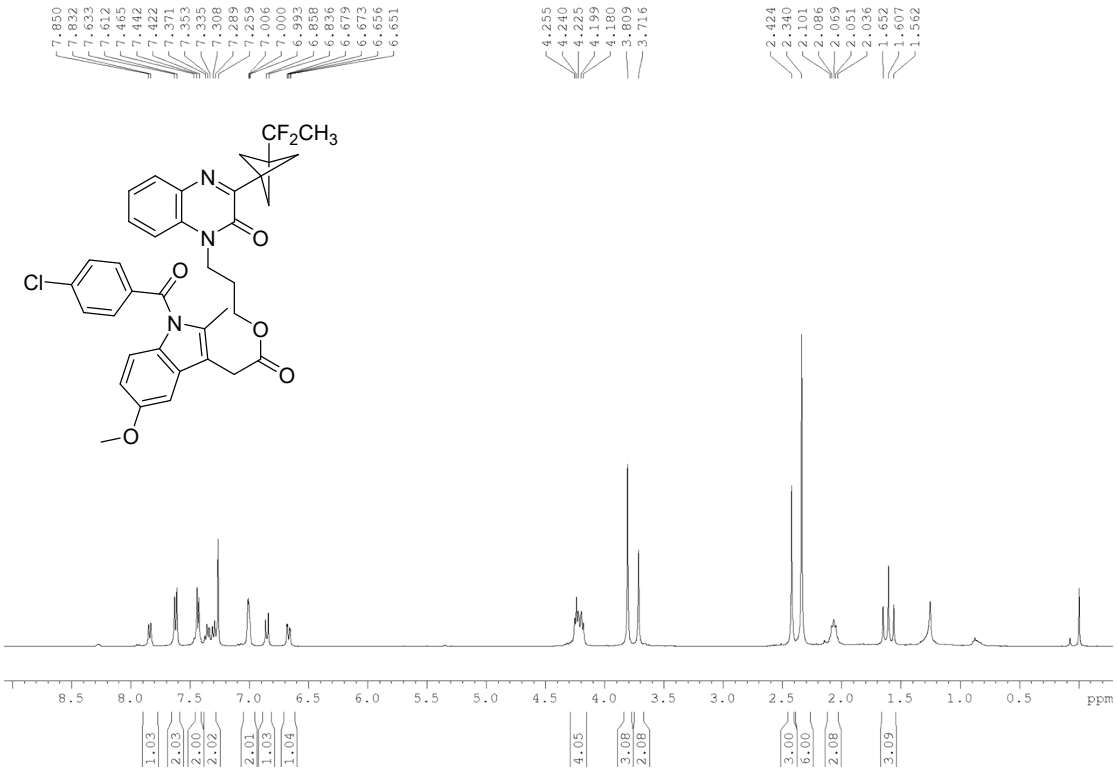

**4aa $^{13}$C NMR (100 MHz, $CDCl_3$)**



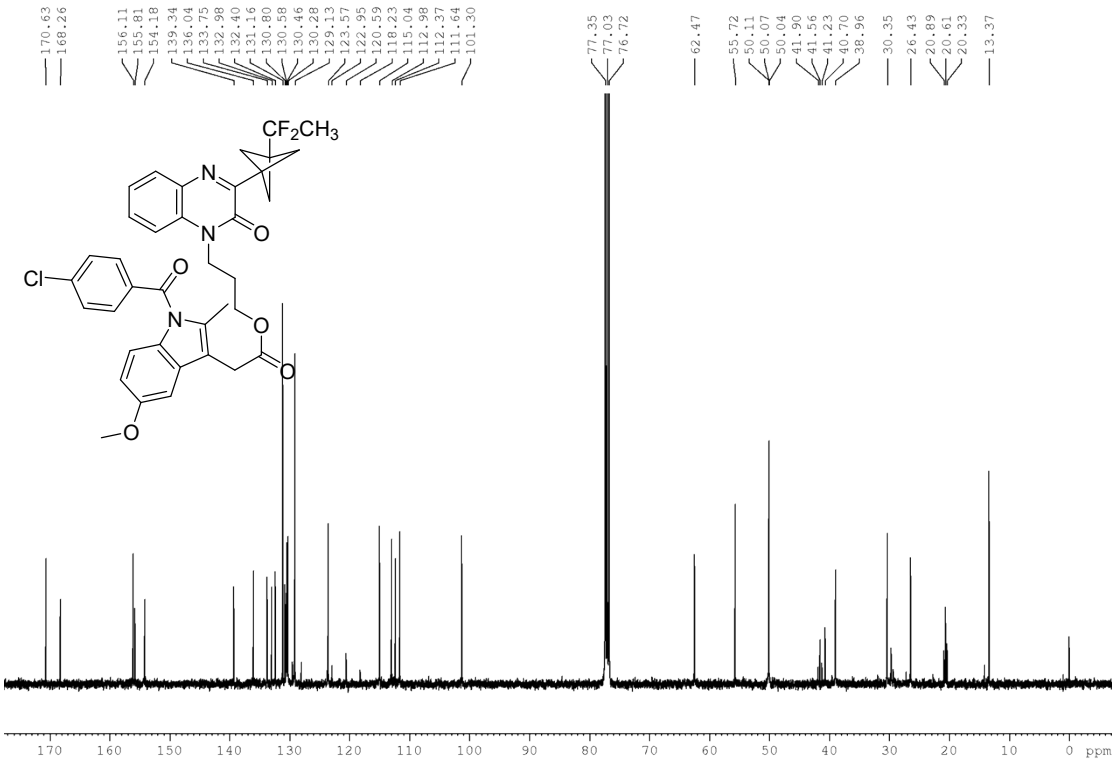

**4aa $^{19}$F NMR (376 MHz, $CDCl_3$)**



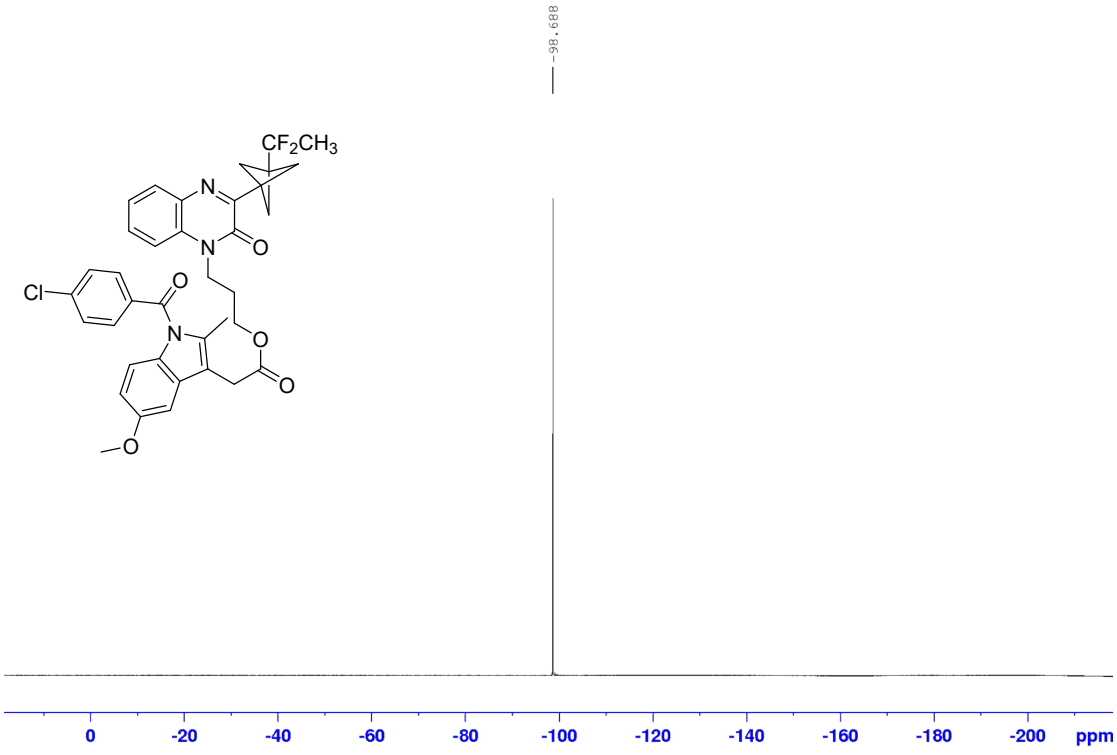

**4ab $^{1}$H NMR (400 MHz, $CDCl_3$)**



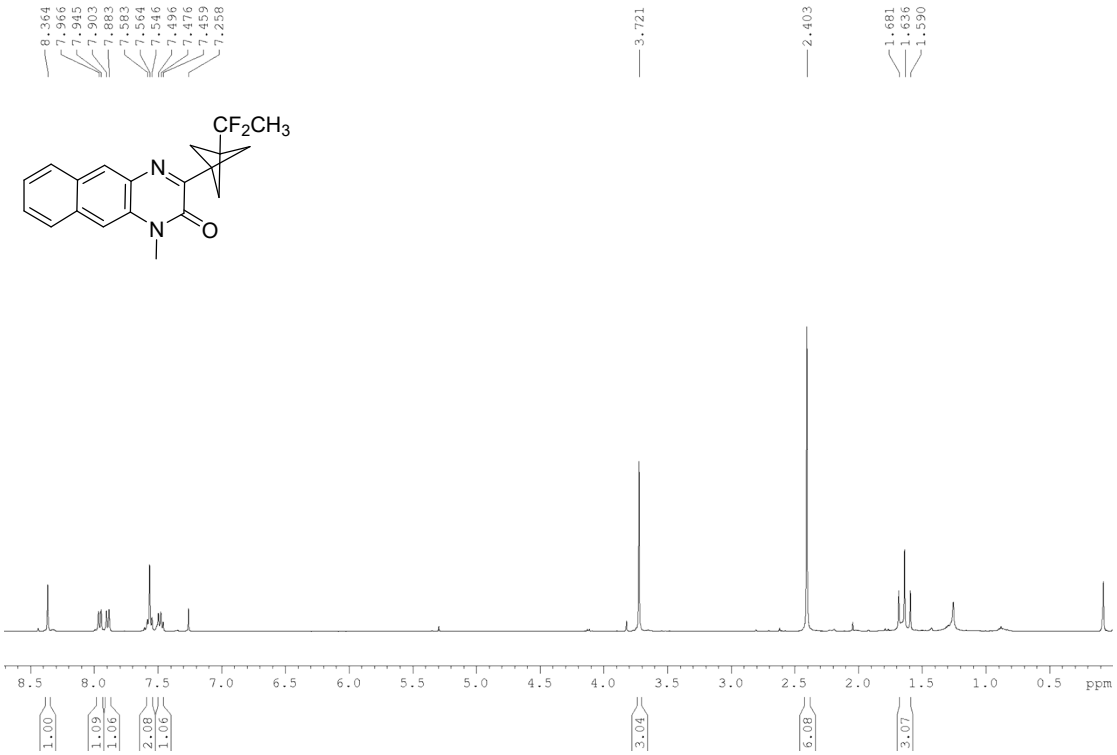

**4ab $^{13}$C NMR (100 MHz, $CDCl_3$)**



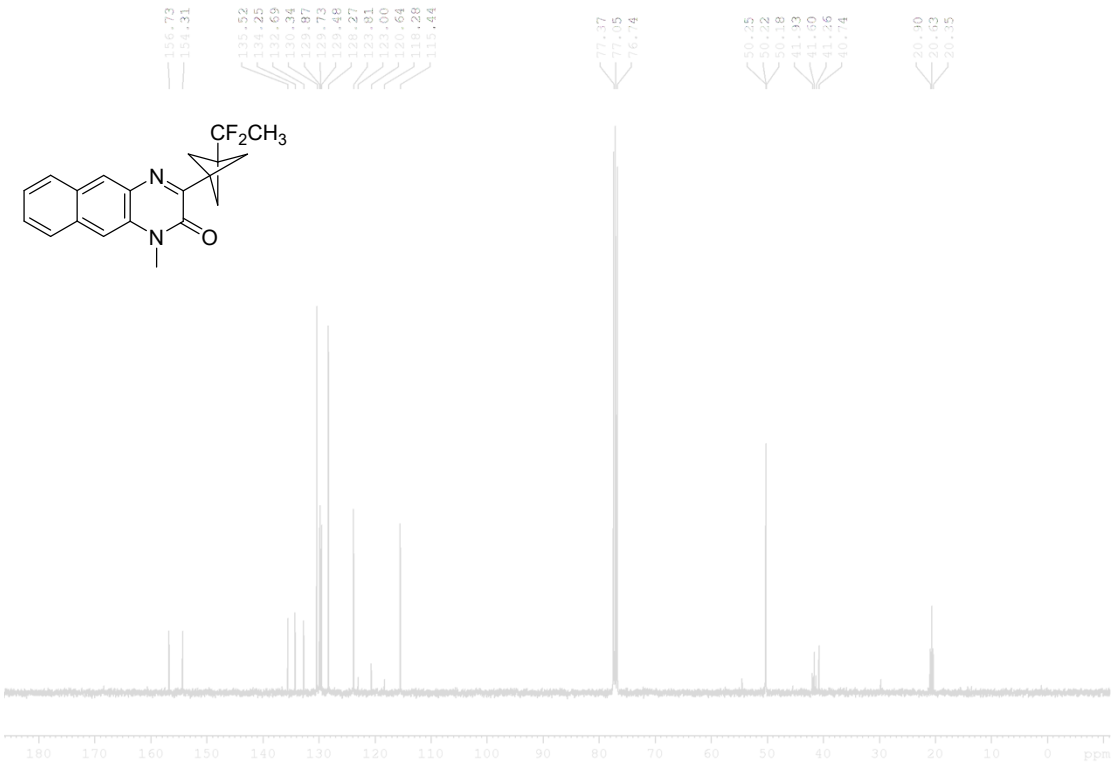

**4ab $^{19}$F NMR (376 MHz, $CDCl_3$)**



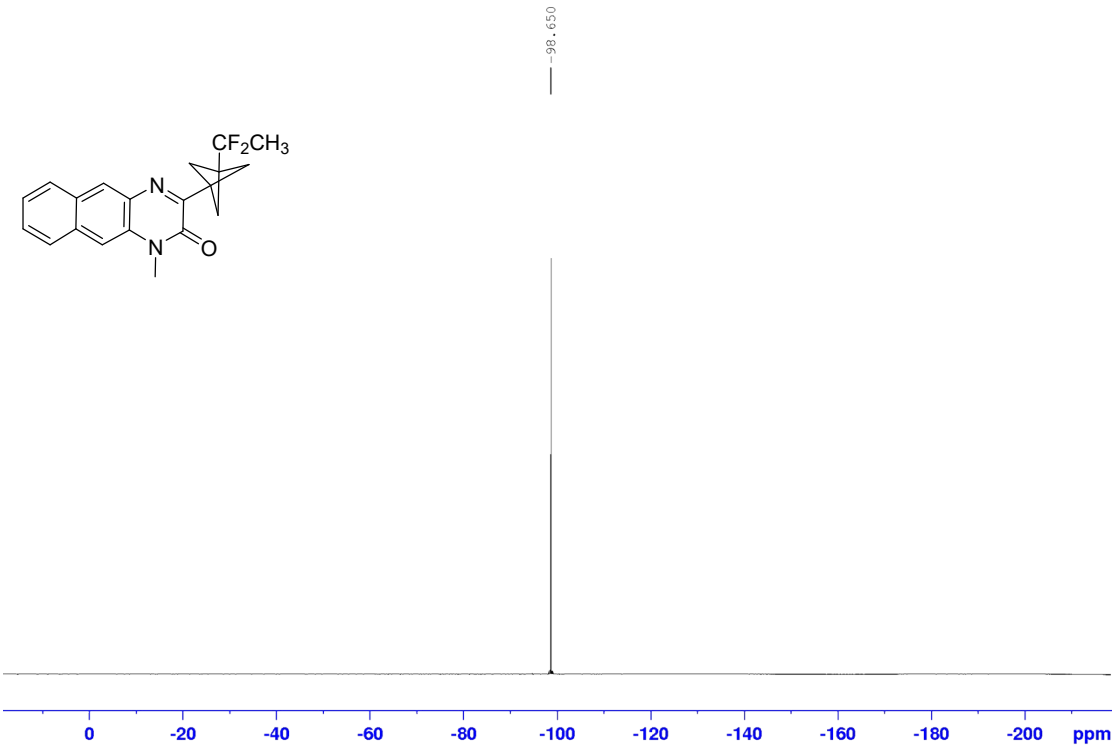

**4ac $^{1}$H NMR (400 MHz, $CDCl_3$)**



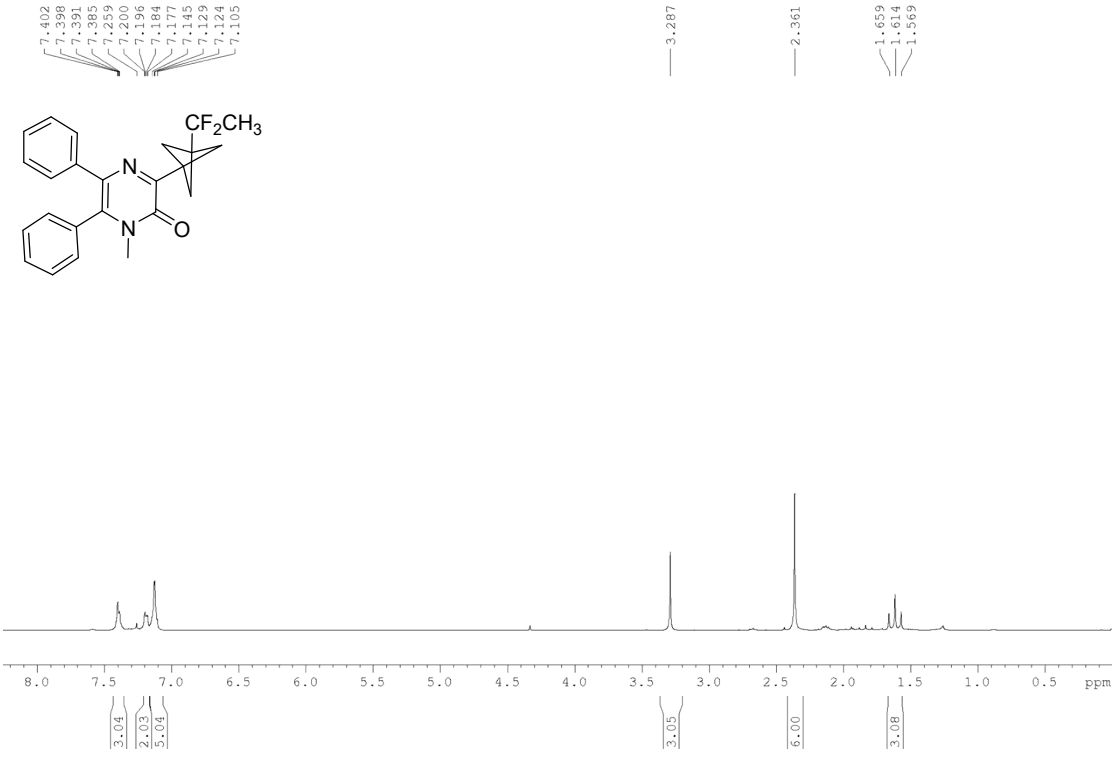

**4ac $^{13}$C NMR (100 MHz, $CDCl_3$)**



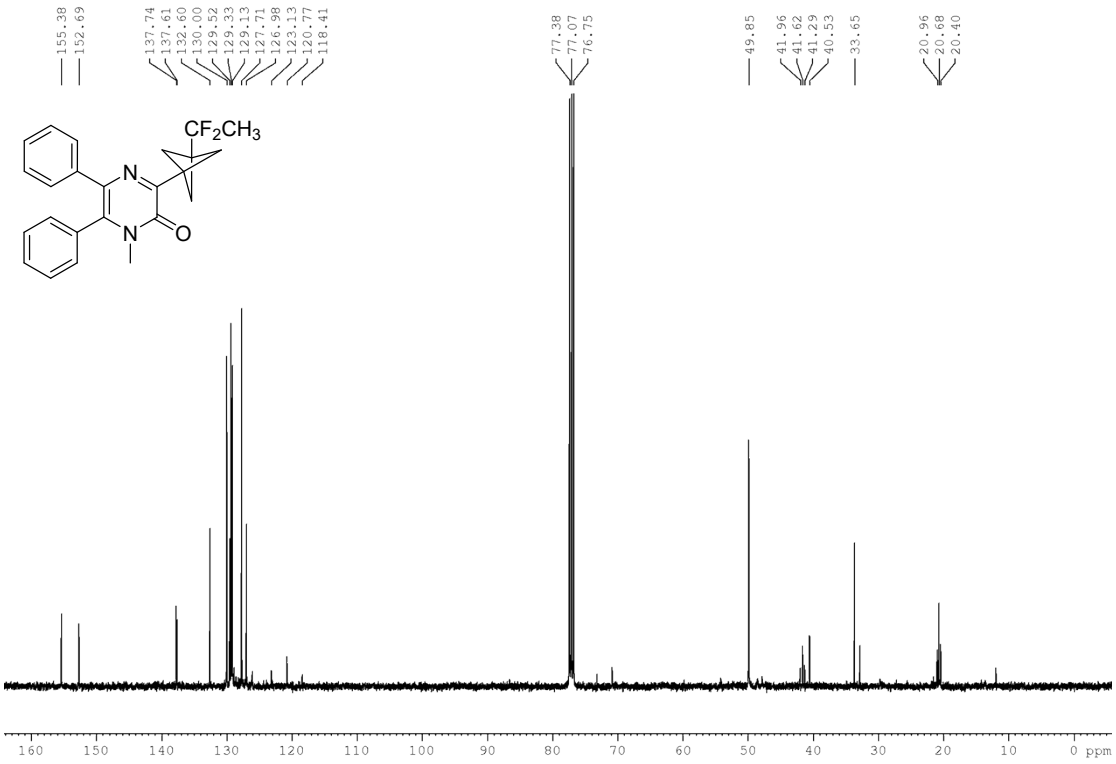

**4ac $^{19}$F NMR (376 MHz, $CDCl_3$)**



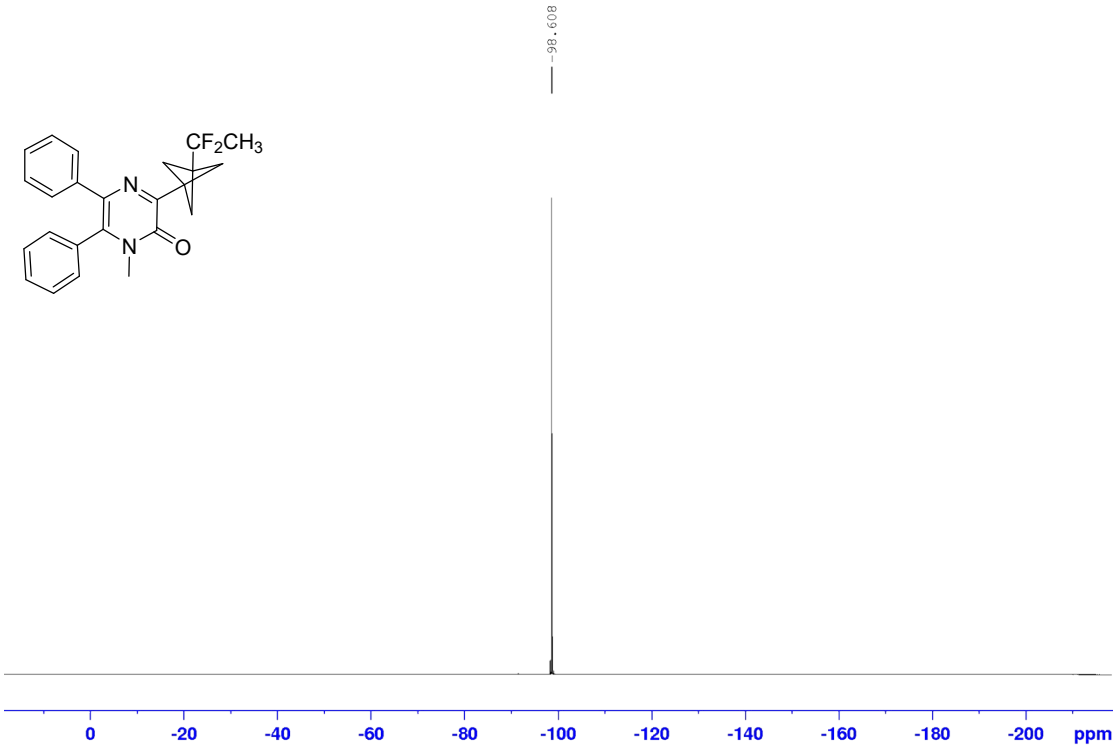

**4ad $^{1}$H NMR (400 MHz, $CDCl_3$)**



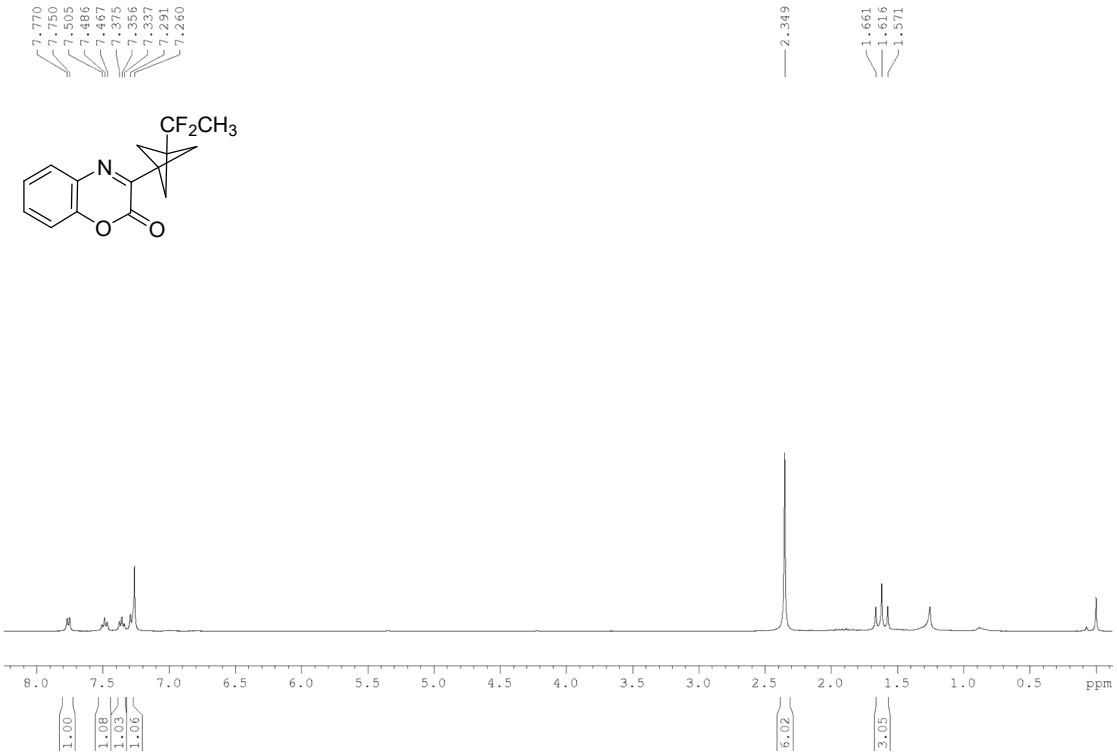

**4ad $^{13}$C NMR (100 MHz, $CDCl_3$)**



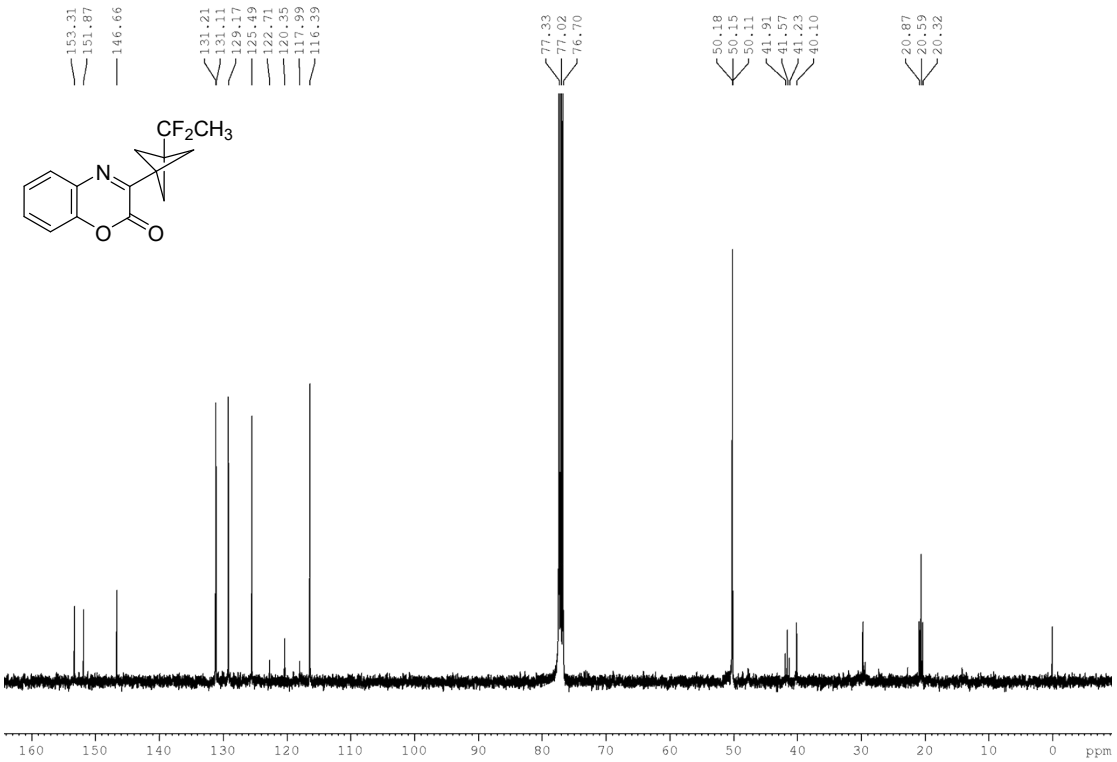

**4ad $^{19}$F NMR (376 MHz, $CDCl_3$)**



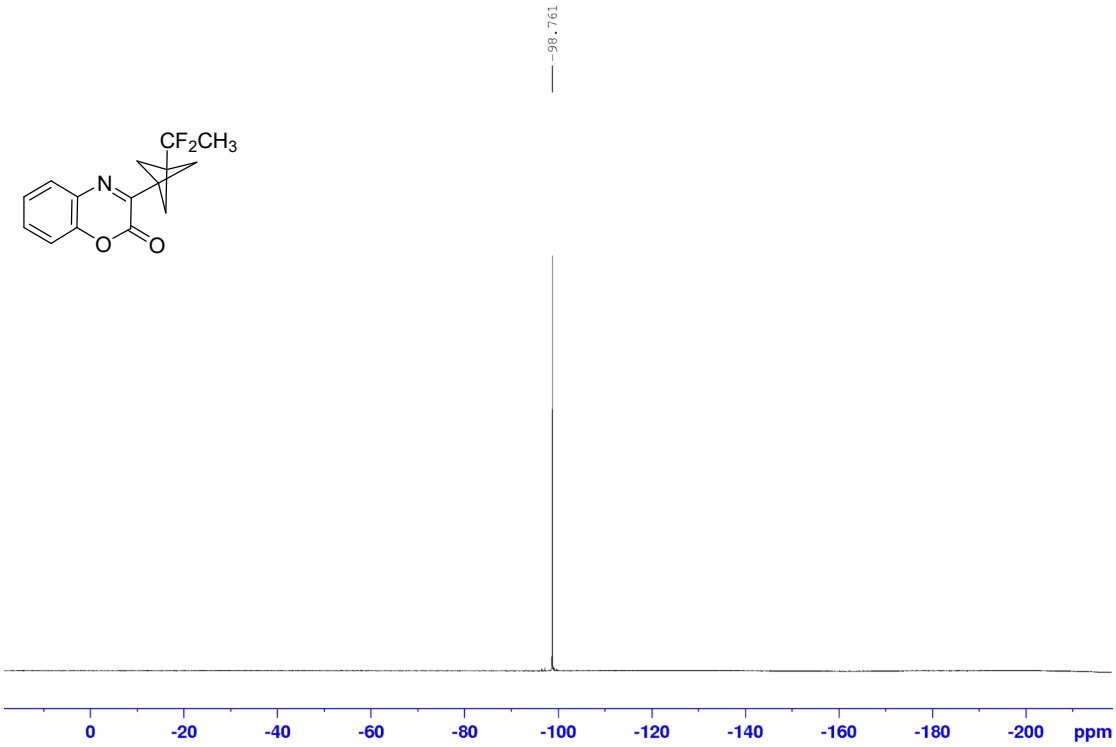

**4ae $^{1}$H NMR (400 MHz, $CDCl_3$)**



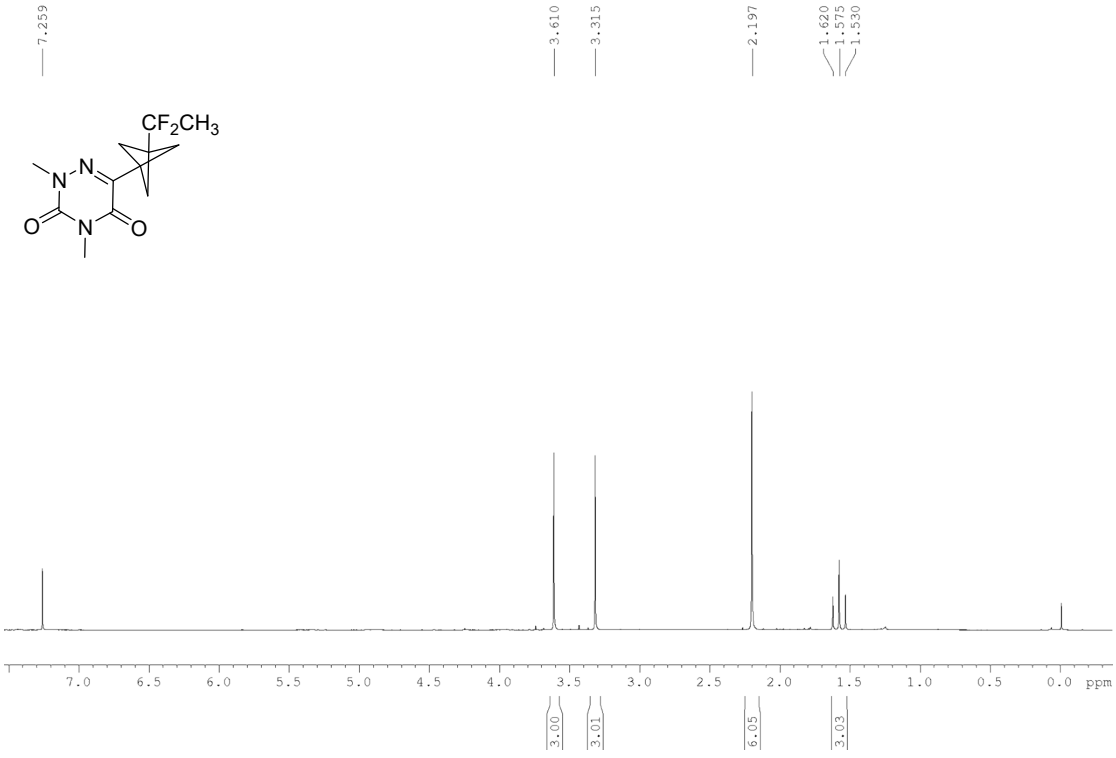

**4ae $^{13}$C NMR (100 MHz, $CDCl_3$)**




**4ae $^{19}$F NMR (376 MHz, $CDCl_3$)**




**4af $^{1}$H NMR (400 MHz, $CDCl_3$)**

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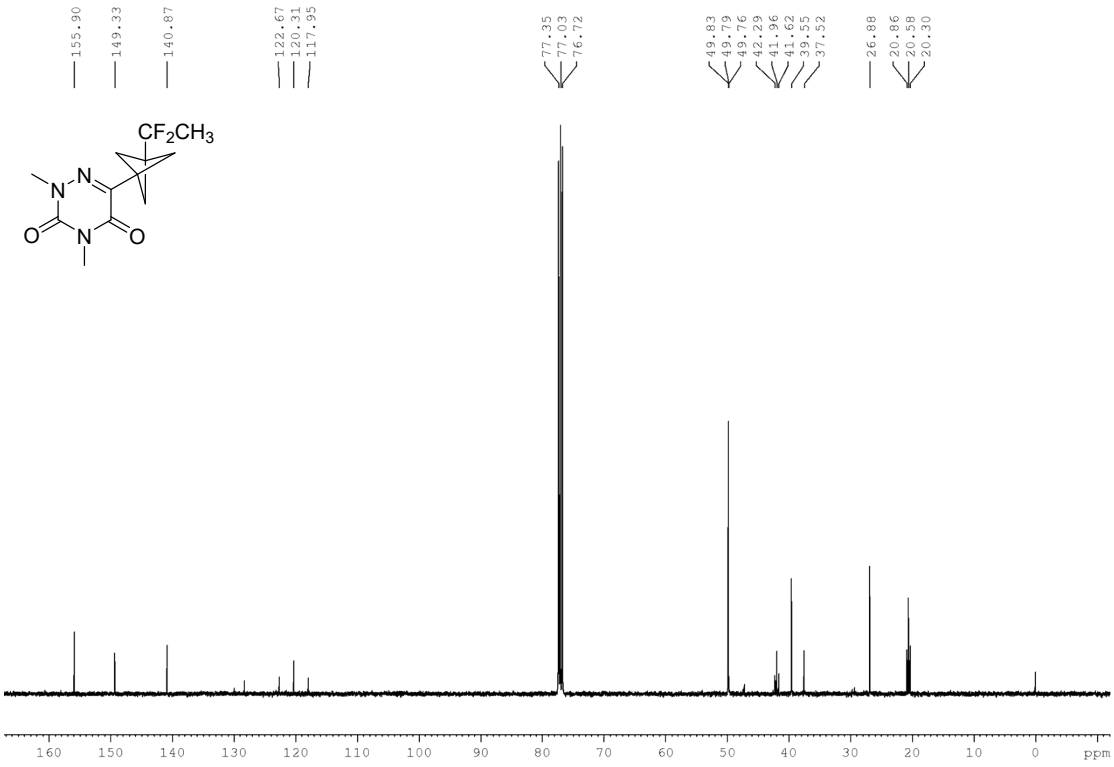
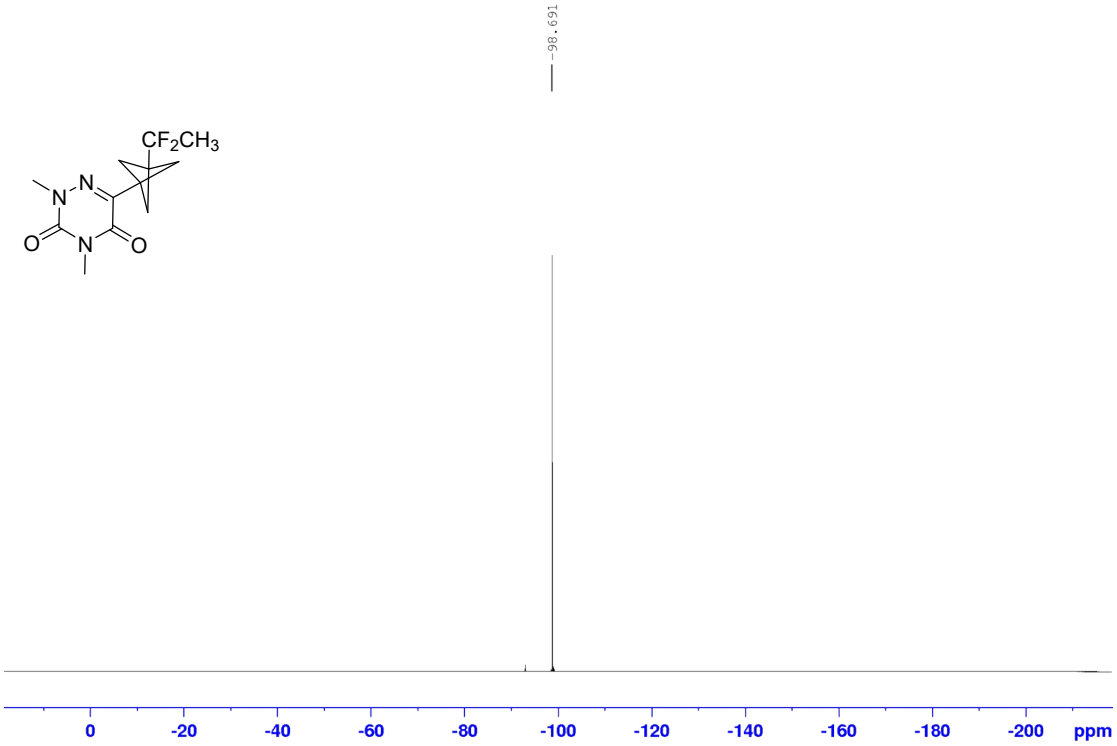



**4af $^{19}$F NMR (376 MHz, $CDCl_3$)**

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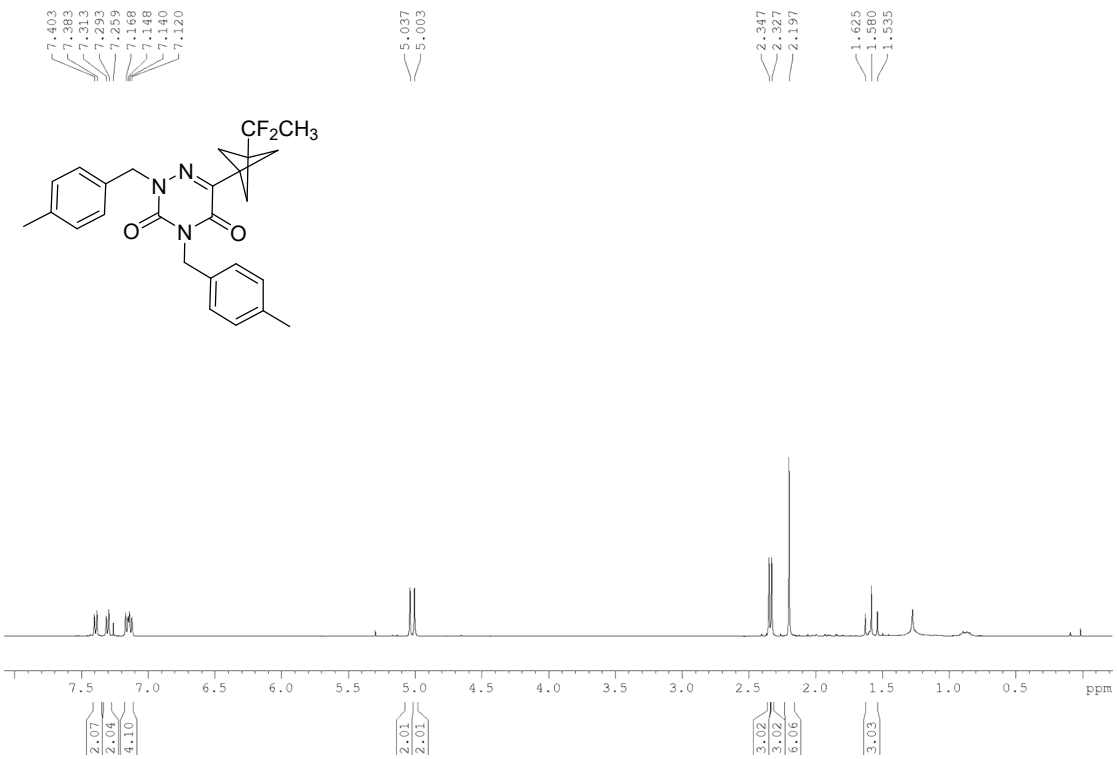
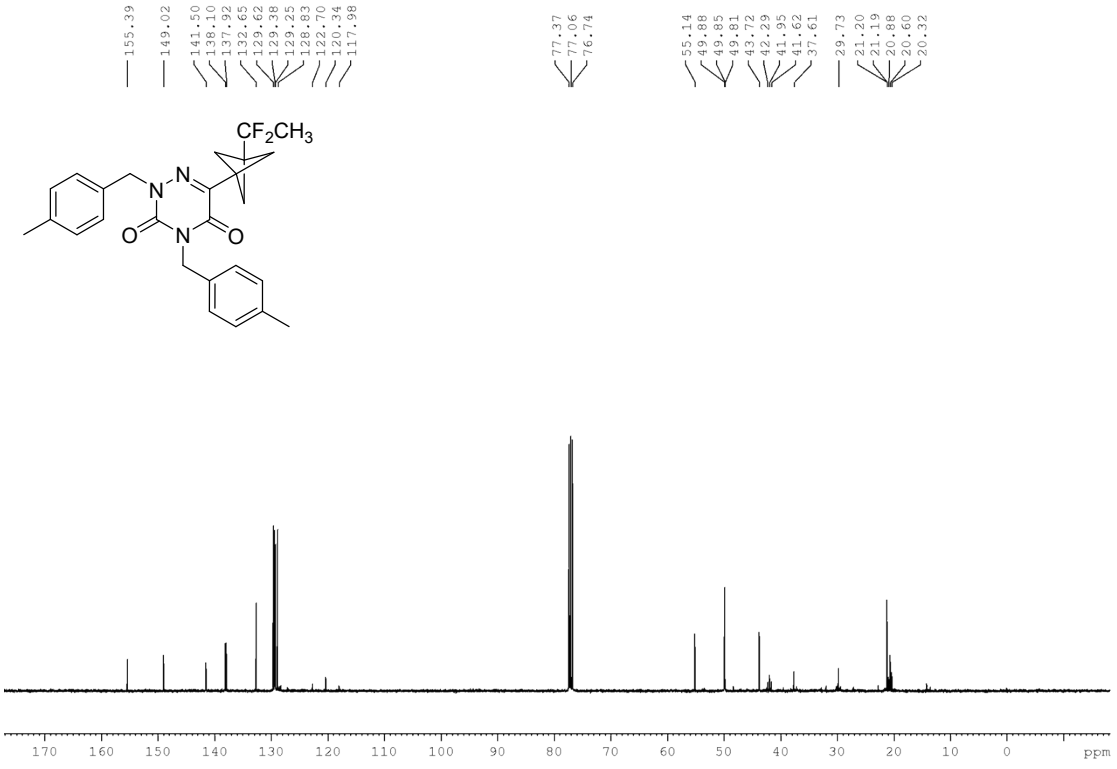
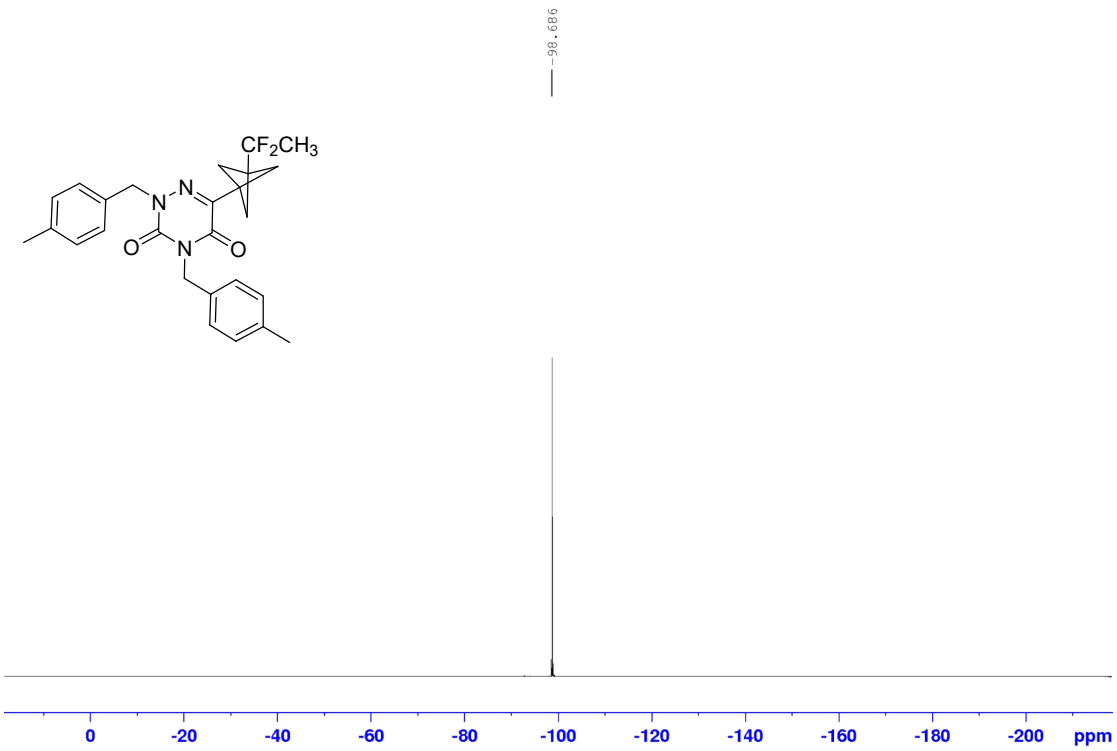


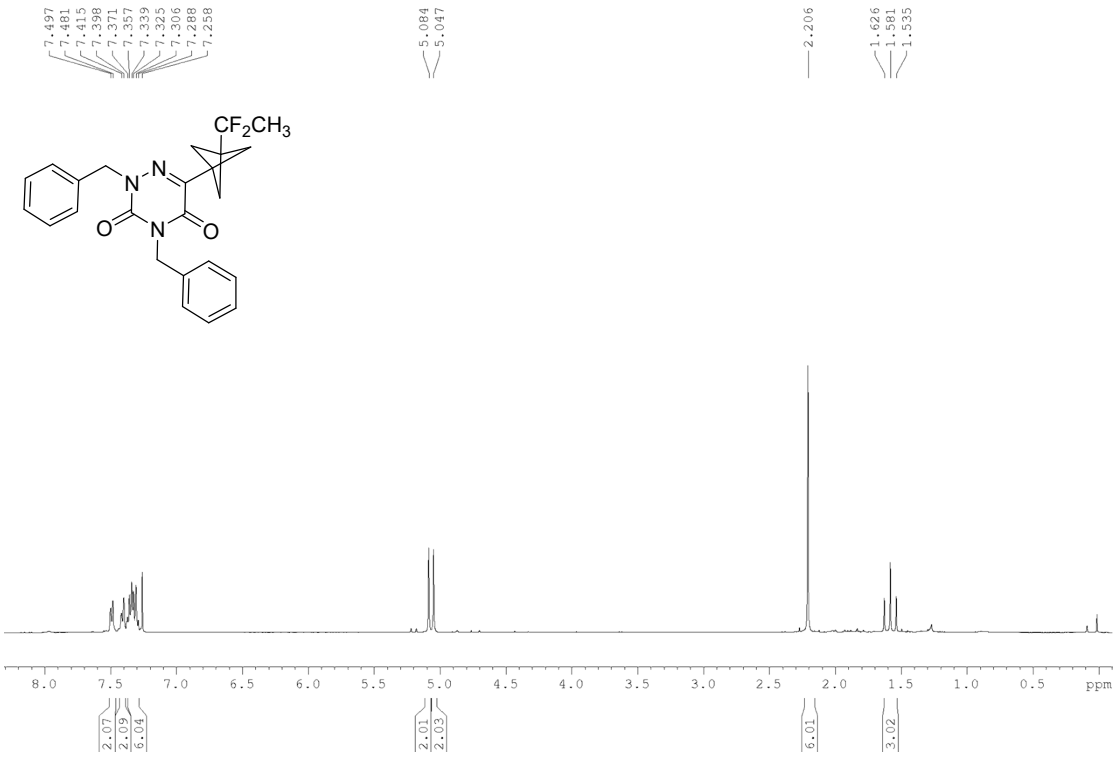

**4ag $^{13}$C NMR (100 MHz, $CDCl_3$)**



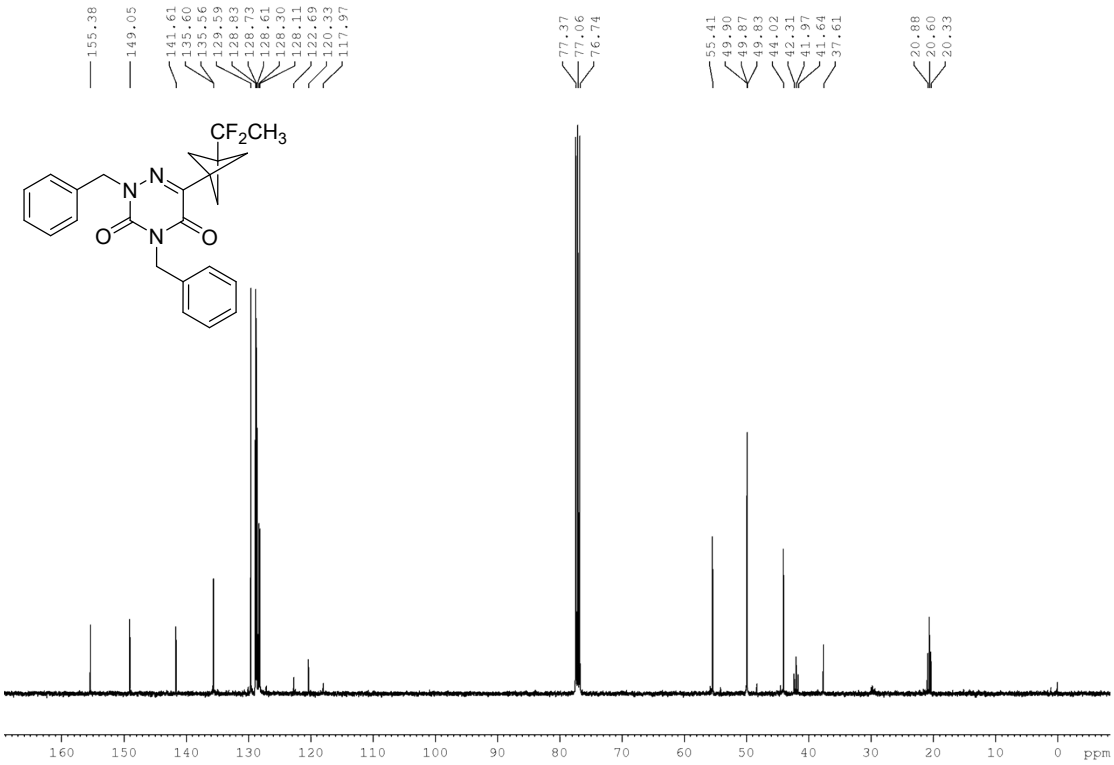

**4ag $^{19}$F NMR (376 MHz, $CDCl_3$)**



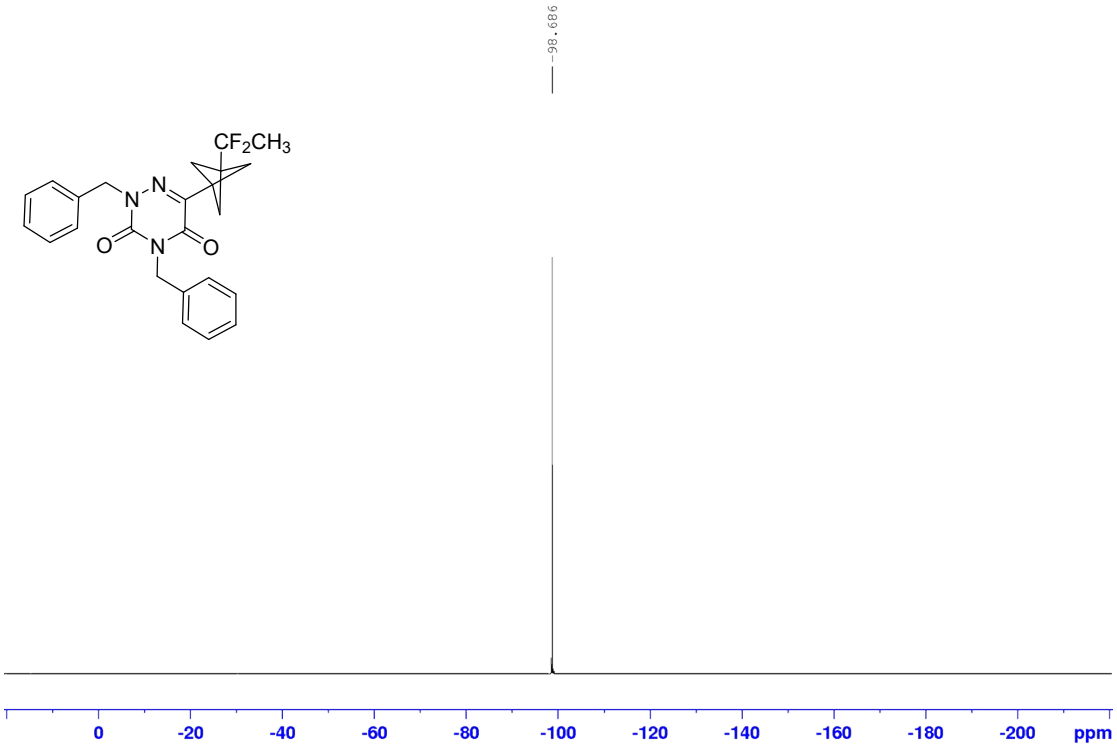

**4ah $^{1}$H NMR (400 MHz, $CDCl_3$)**



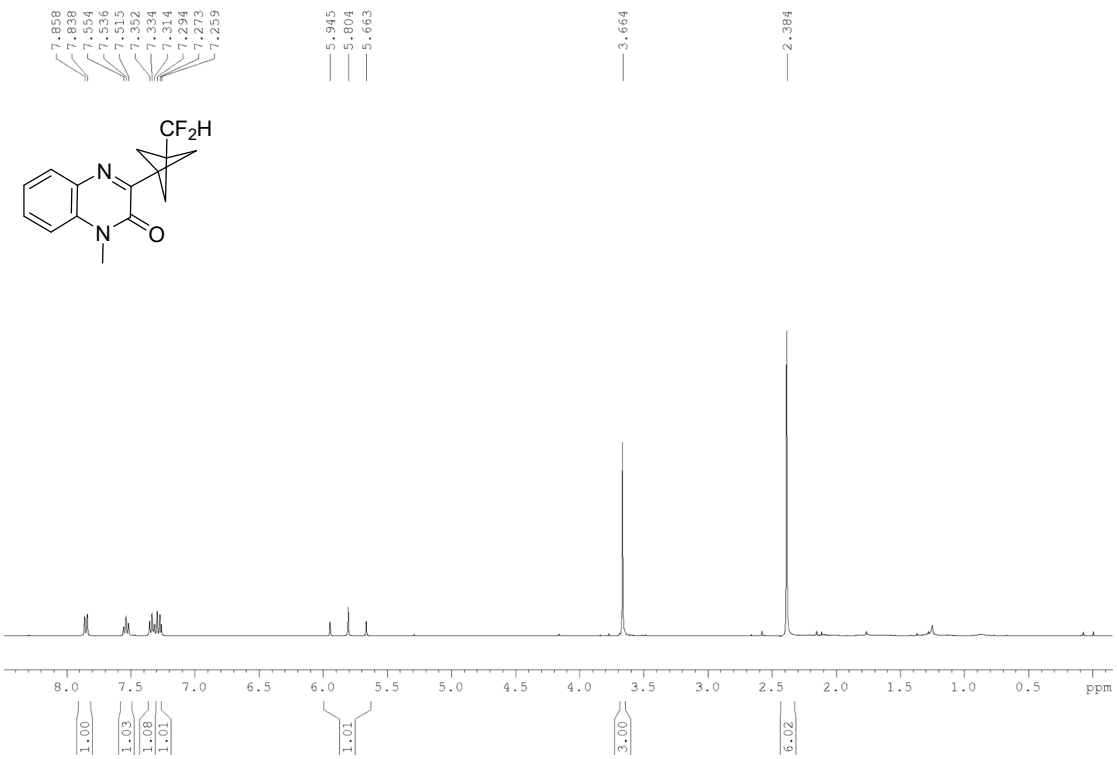

**4ah $^{13}$C NMR (100 MHz, $CDCl_3$)**



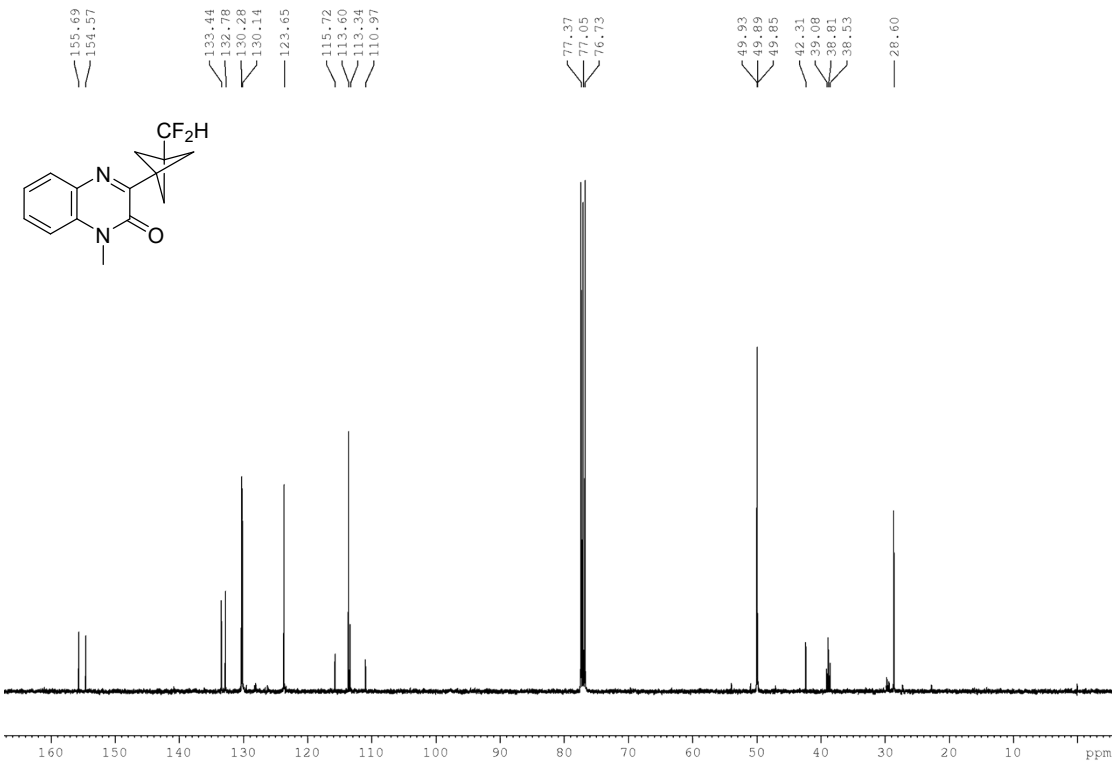

**4ah $^{19}$F NMR (376 MHz, $CDCl_3$)**



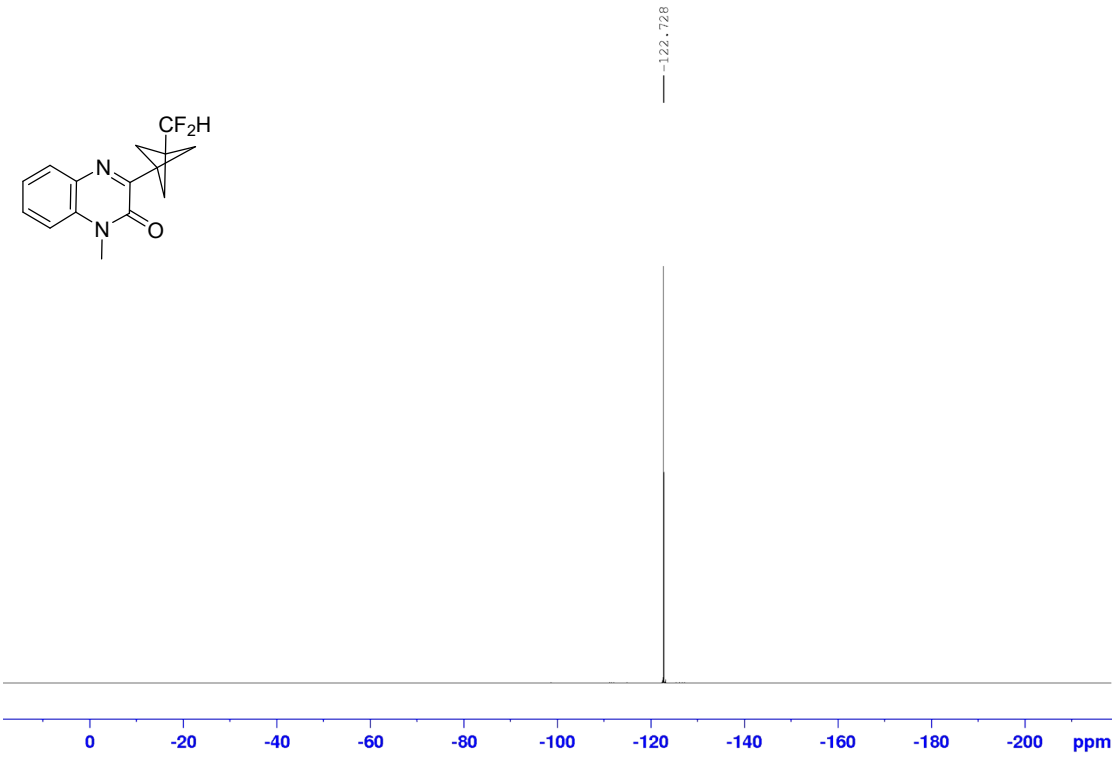

**4ai $^{1}$H NMR (400 MHz, $CDCl_3$)**



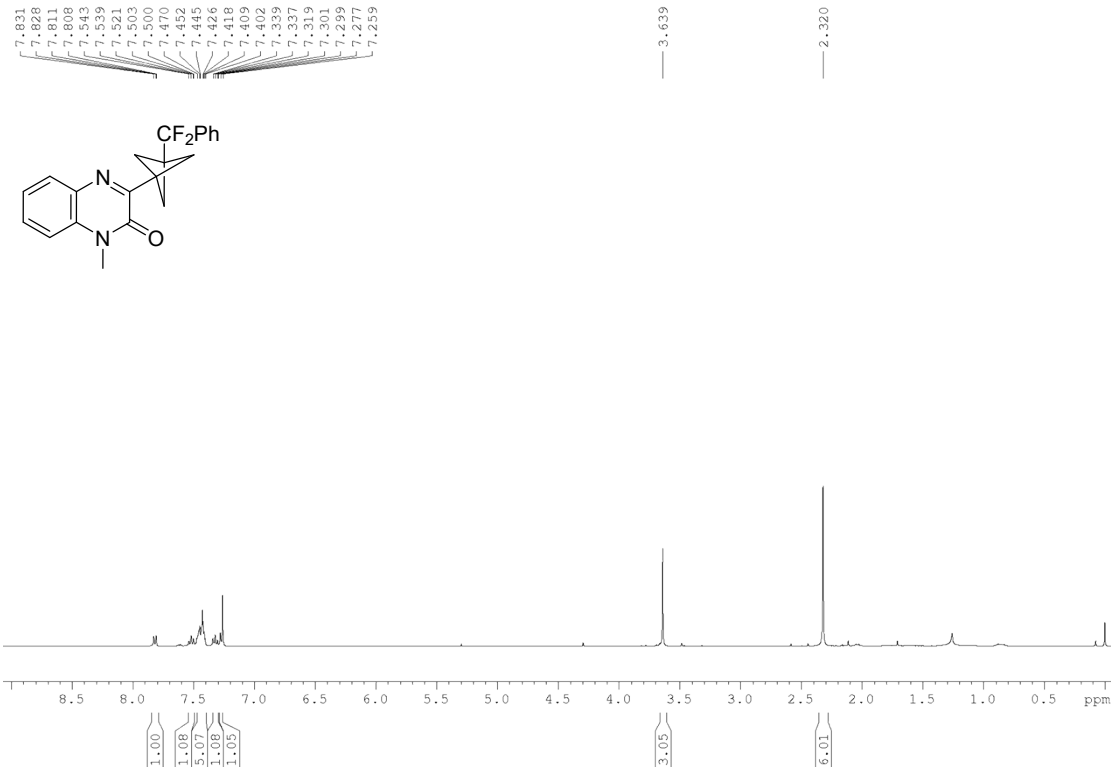

**4ai $^{13}$C NMR (100 MHz, $CDCl_3$)**



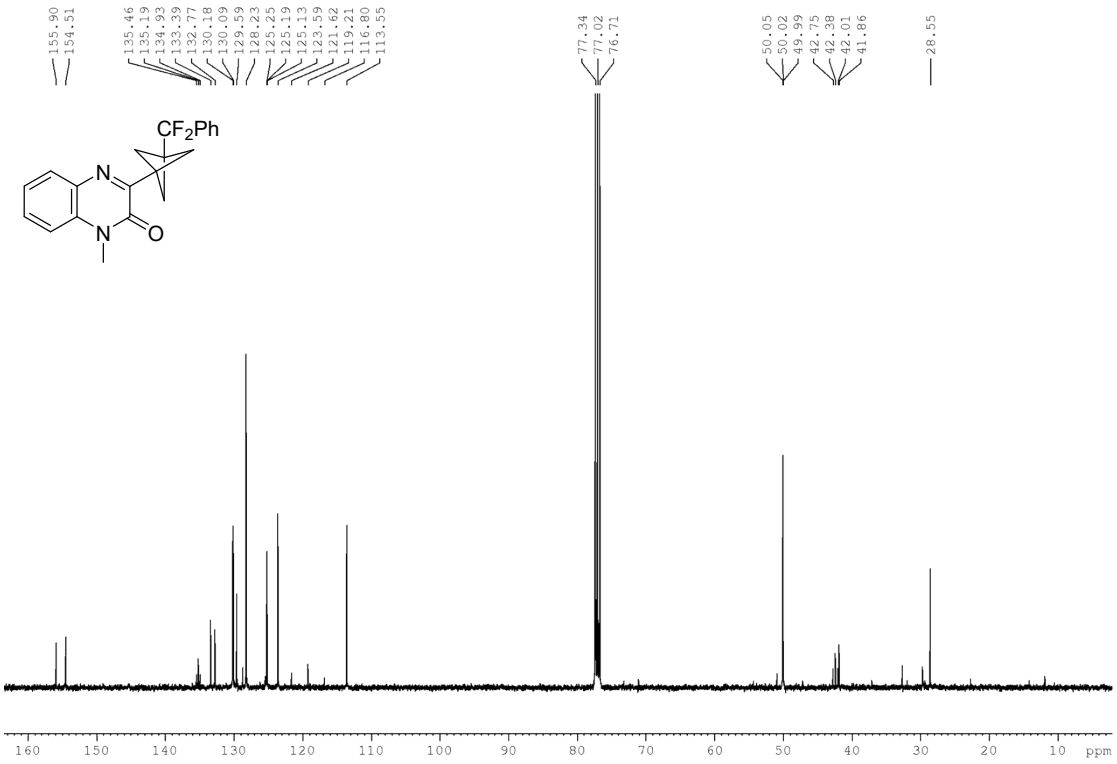

**4ai $^{19}$F NMR (376 MHz, $CDCl_3$)**



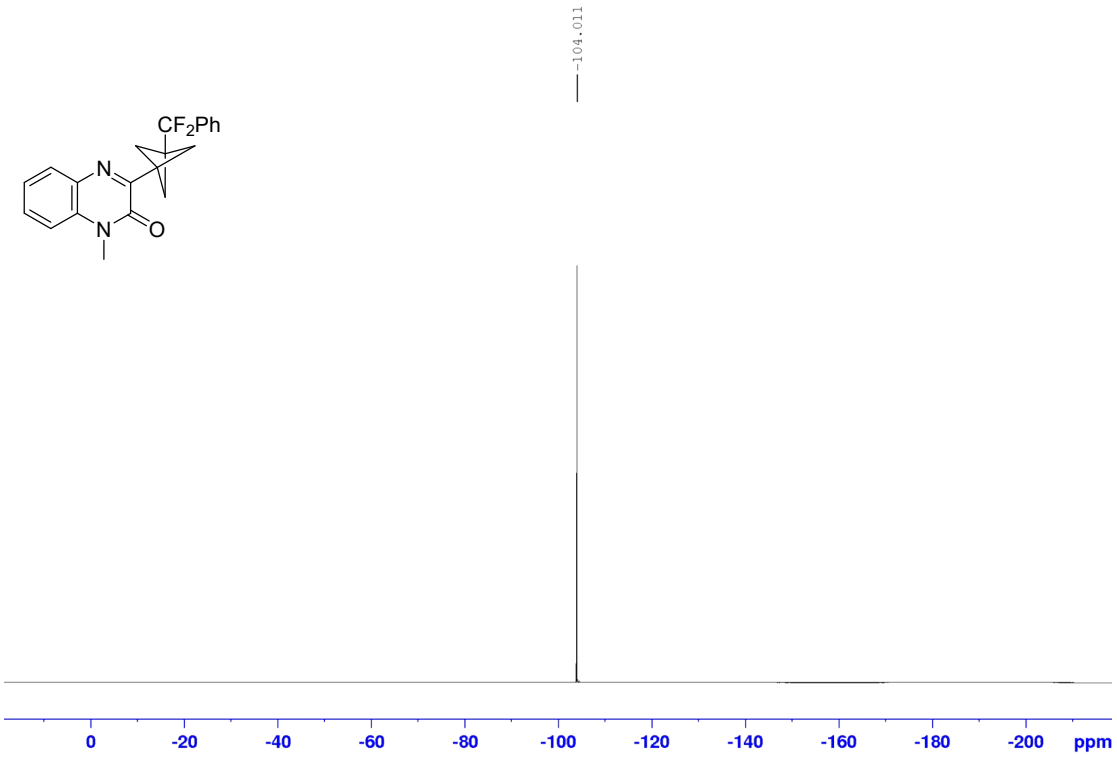

**4aj $^{1}$H NMR (400 MHz, $CDCl_3$)**



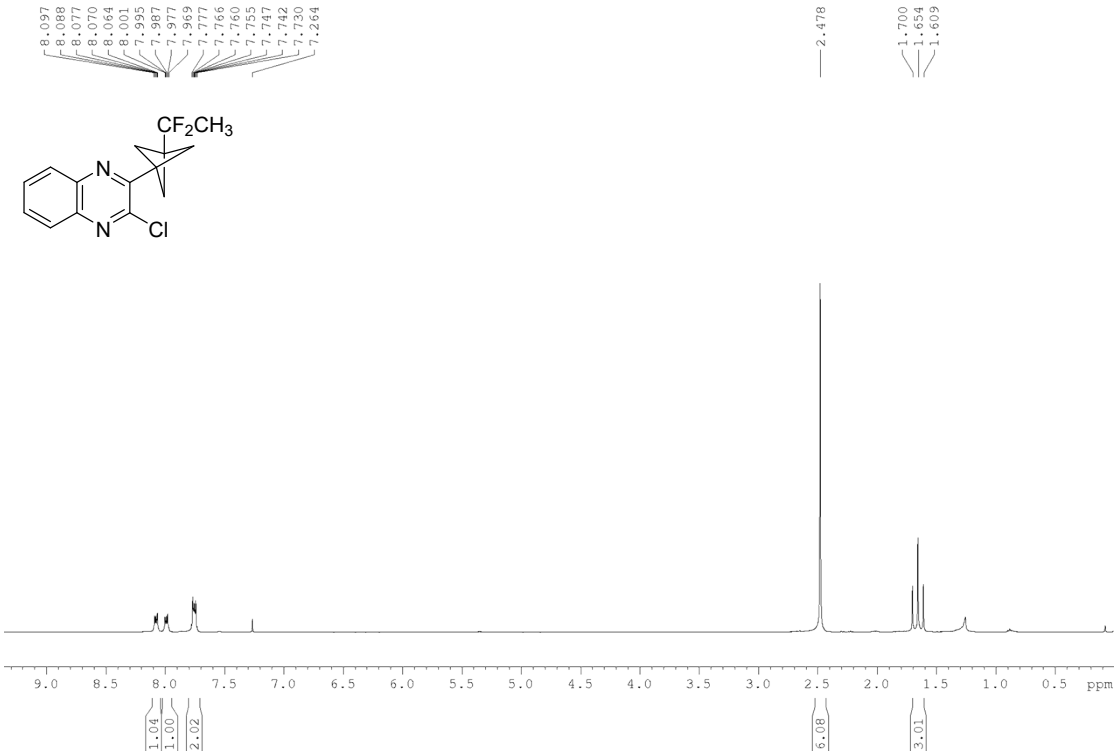

**4aj $^{13}$C NMR (100 MHz, $CDCl_3$)**



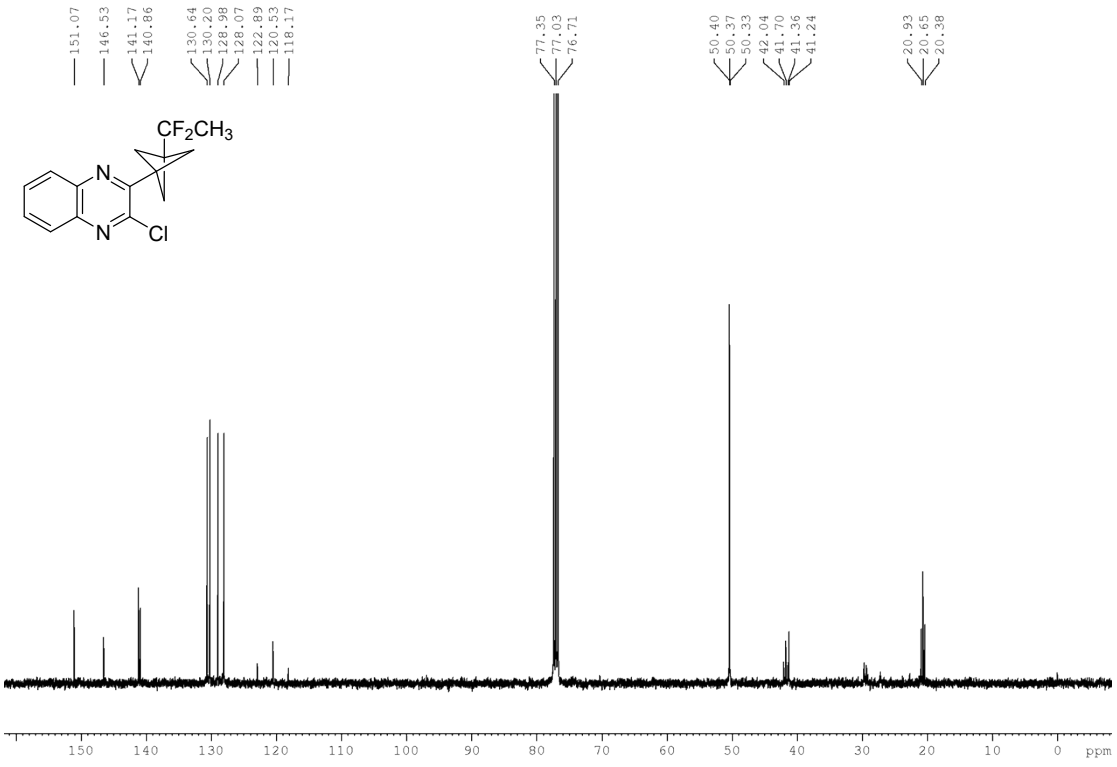

**4aj $^{19}$F NMR (376 MHz, $CDCl_3$)**



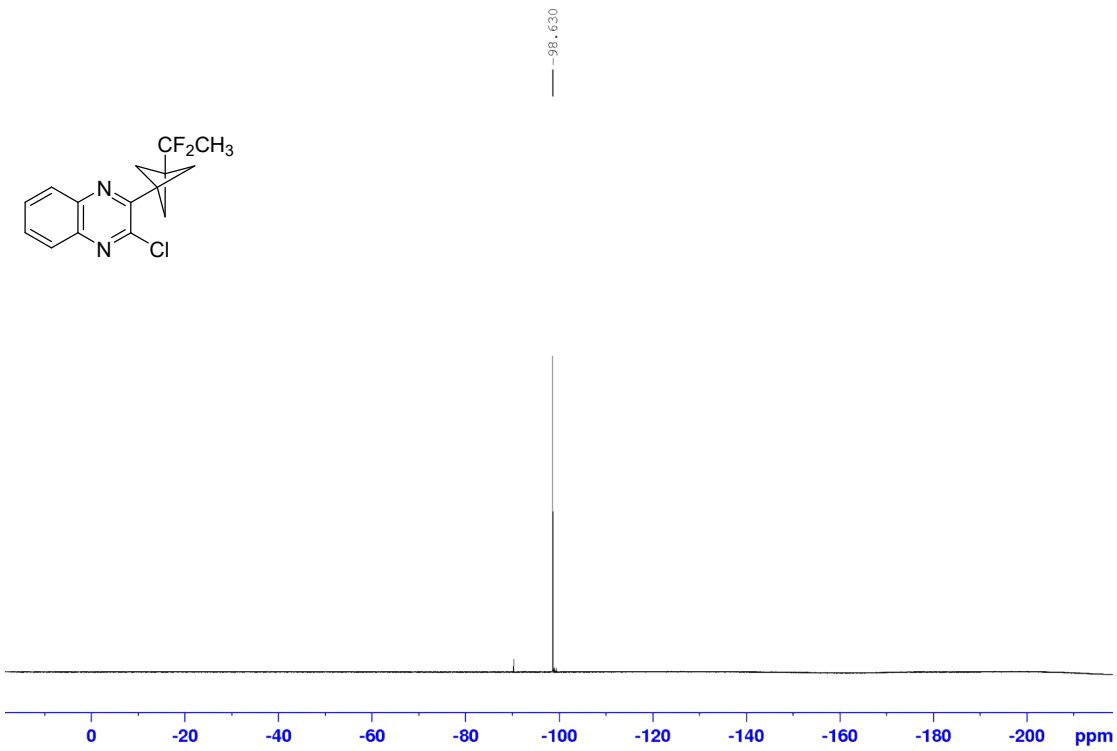

**7 $^{1}$H NMR (400 MHz, $CDCl_3$)**



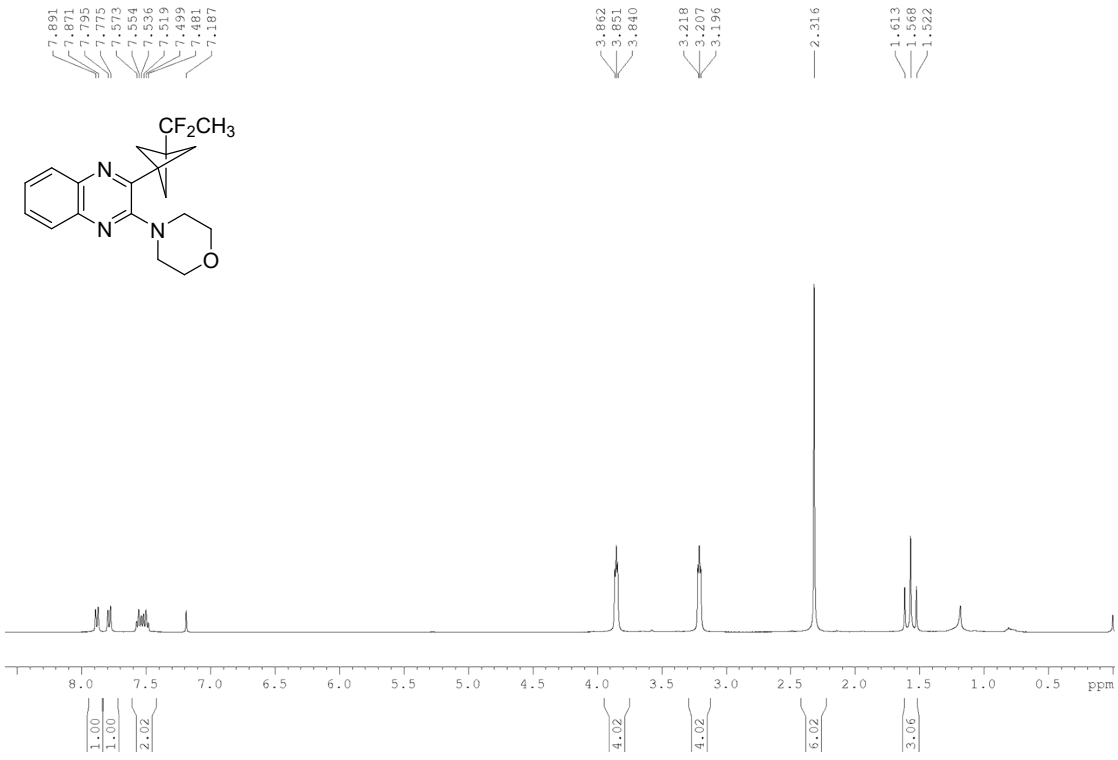

## 7 $^{13}$C NMR (100 MHz, $CDCl_3$)



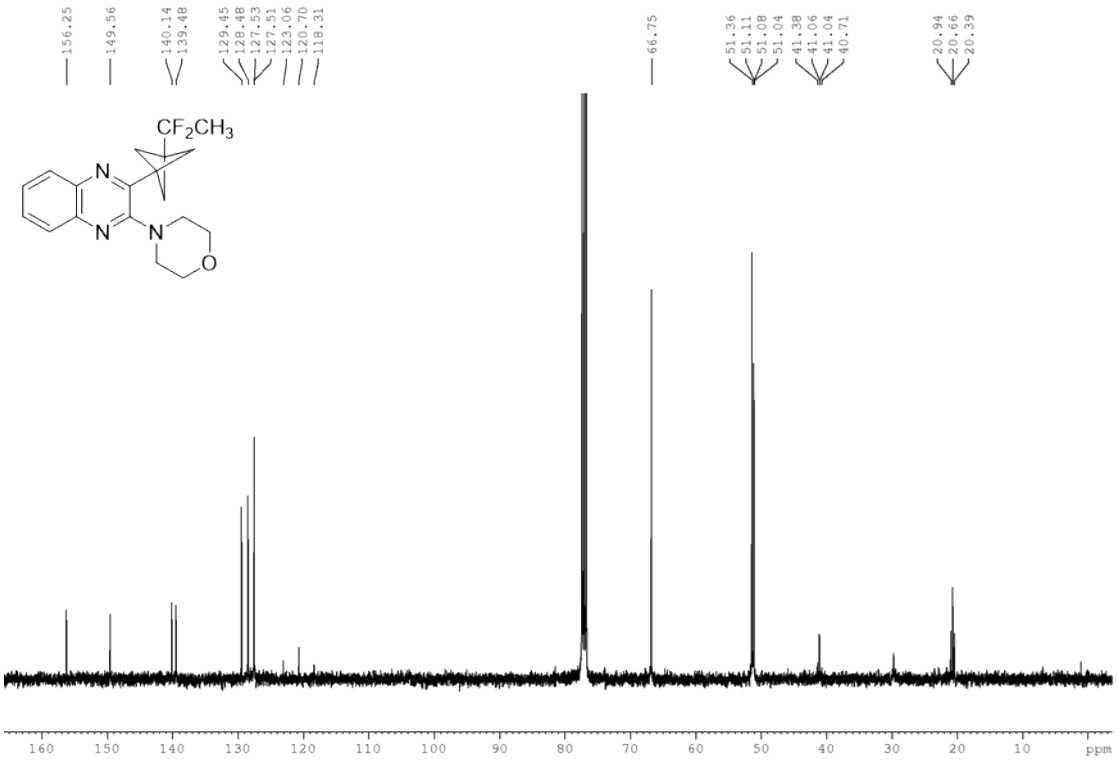

## 7 $^{19}$F NMR (376 MHz, $CDCl_3$)




## 8 $^{1}$H NMR (400 MHz, $CDCl_3$)




## 8 $^{13}$C NMR (100 MHz, $CDCl_3$)




## 8 $^{19}$F NMR (376 MHz, $CDCl_3$)