

# Transition-metal and oxidant-free three-component reaction of quinoline *N*-oxides, sodium metabisulfite and aryldiazonium tetrafluoroborates *via* a dual radical coupling process

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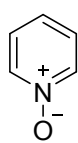
<sup>b</sup> Shandong Institute for Product Quality Inspection, Jinan 250102 (China).

## Supporting Information

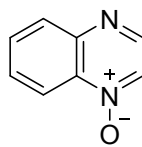
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Ineffective substrates:

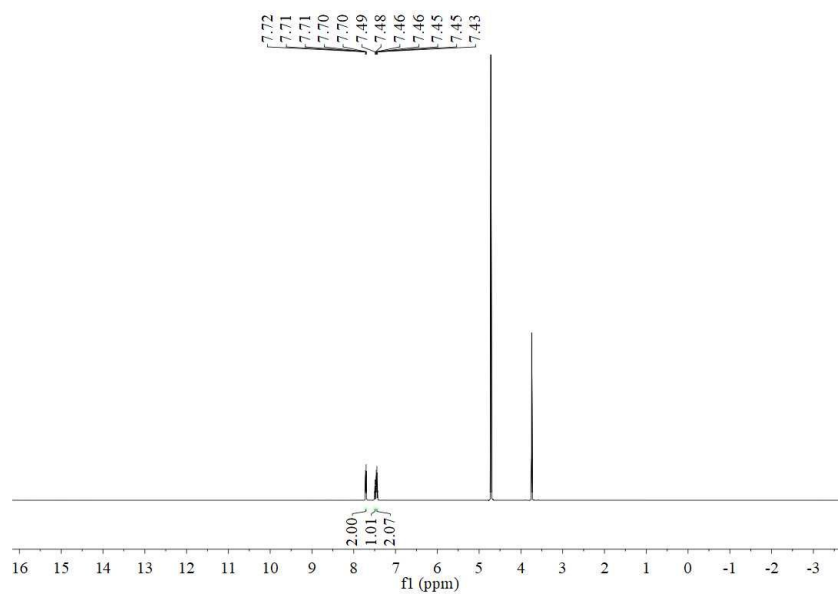


1j



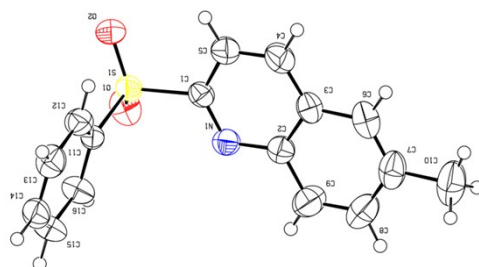
1k

Mechanism study:



The benzenesulfonic acid in the reaction mixture was detected by  $^1\text{H}$  NMR

## 1. X-ray Crystal Data for **3w**



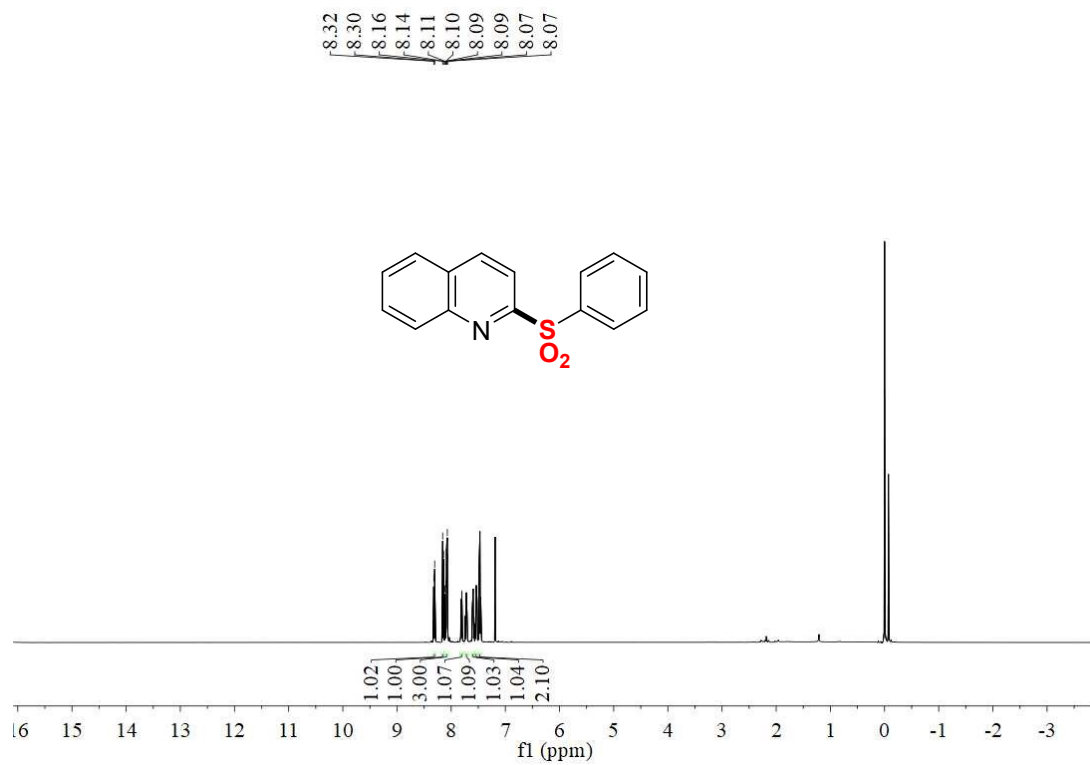
**Figure 1** Single-crystal X-ray structure of **3w**. Ellipsoids are represented at 30% probability.

**Table S1.** Crystallographic data and structure refinement for **3w**

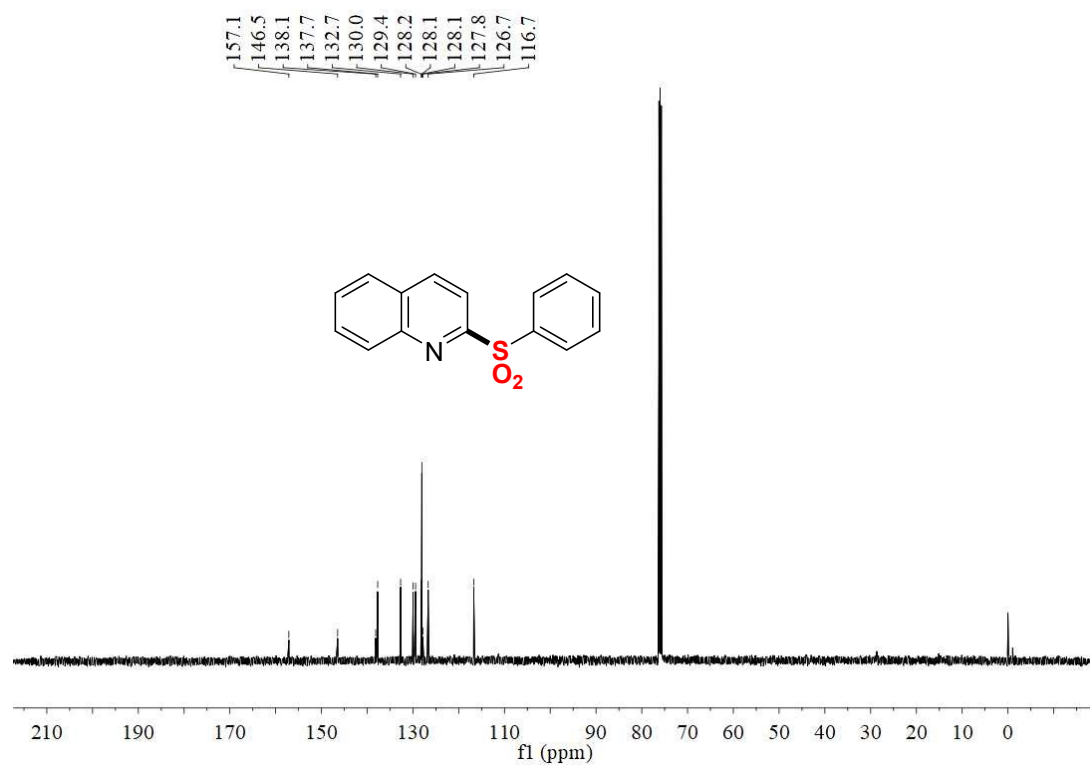
CCDC	1953274
Empirical formula	C <sub>16</sub> H <sub>13</sub> NO <sub>2</sub> S
Formula weight	283.33
Temperature, K	296.15
Wavelength, Å	0.71073
Crystal system	Triclinic
Space group	P-1
a, b, c, Å	6.4036(12), 7.8241(19), 14.538(3)
$\alpha$ , $\beta$ , $\gamma$ , °	85.370(3), 89.441(2), 70.995(2)
Volume, Å <sup>3</sup>	686.3(3)
Z	2
Calculated density, Mg/m <sup>3</sup>	1.371
Absorption coefficient, mm <sup>-1</sup>	0.236
F (000)	296
Theta range for data collection, °	2.763 to 27.582
Limiting indices	-8 ≤ h ≤ 7, -10 ≤ k ≤ 10, -18 ≤ l ≤ 18
Reflections collected / unique	5484 / 2928 [R(int) = 0.0197]
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	2928 / 0 / 182
Goodness of fit on F <sup>2</sup>	1.058
Final R indices [I > 2σ(I)]	R1 = 0.0424, wR2 = 0.1328
R indices (all data)	R1 = 0.0545, wR2 = 0.1445
Extinction coefficient	n/a
Largest diff. peak and hole e. Å <sup>-3</sup>	0.208 and -0.338

## 2. Copies of $^1\text{H}$ and $^{13}\text{C}$ NMR Spectra

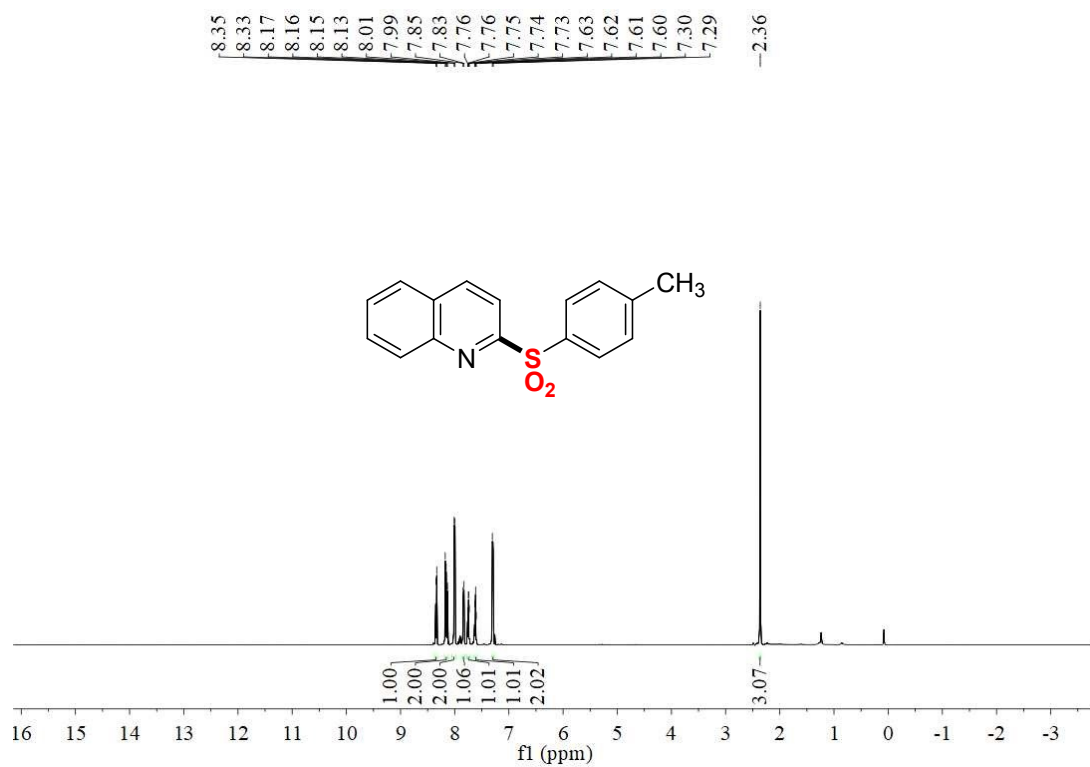
### 3a $^1\text{H}$ NMR



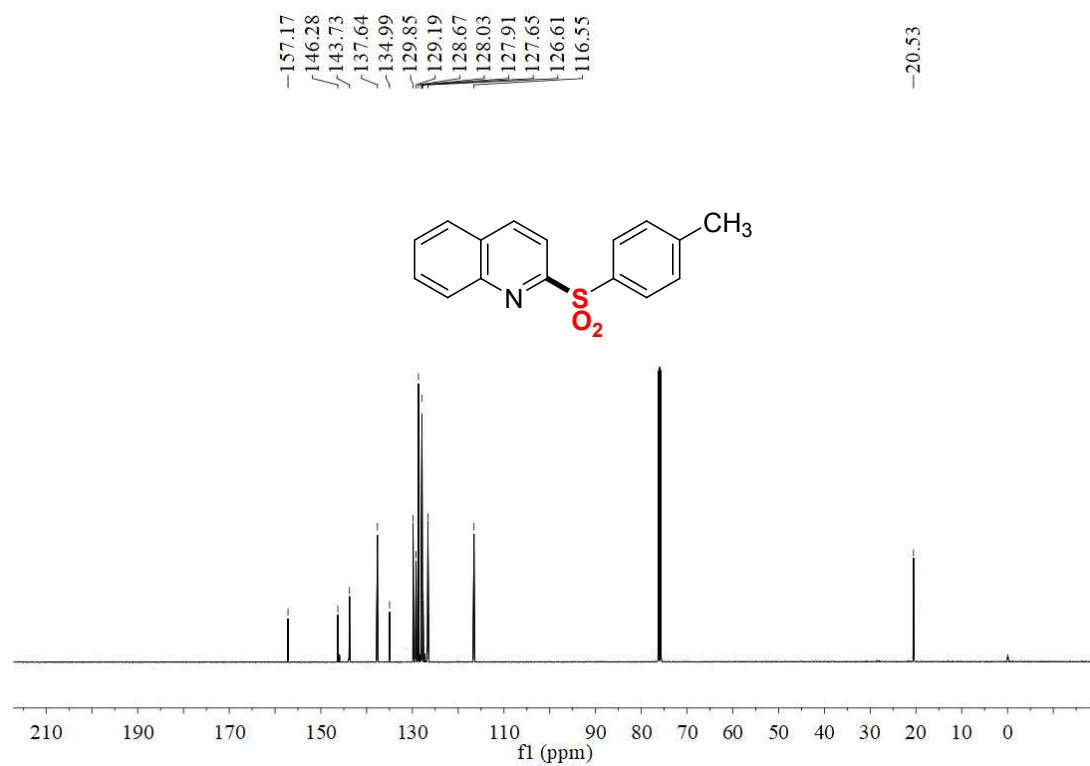
### 3a $^{13}\text{C}$ NMR



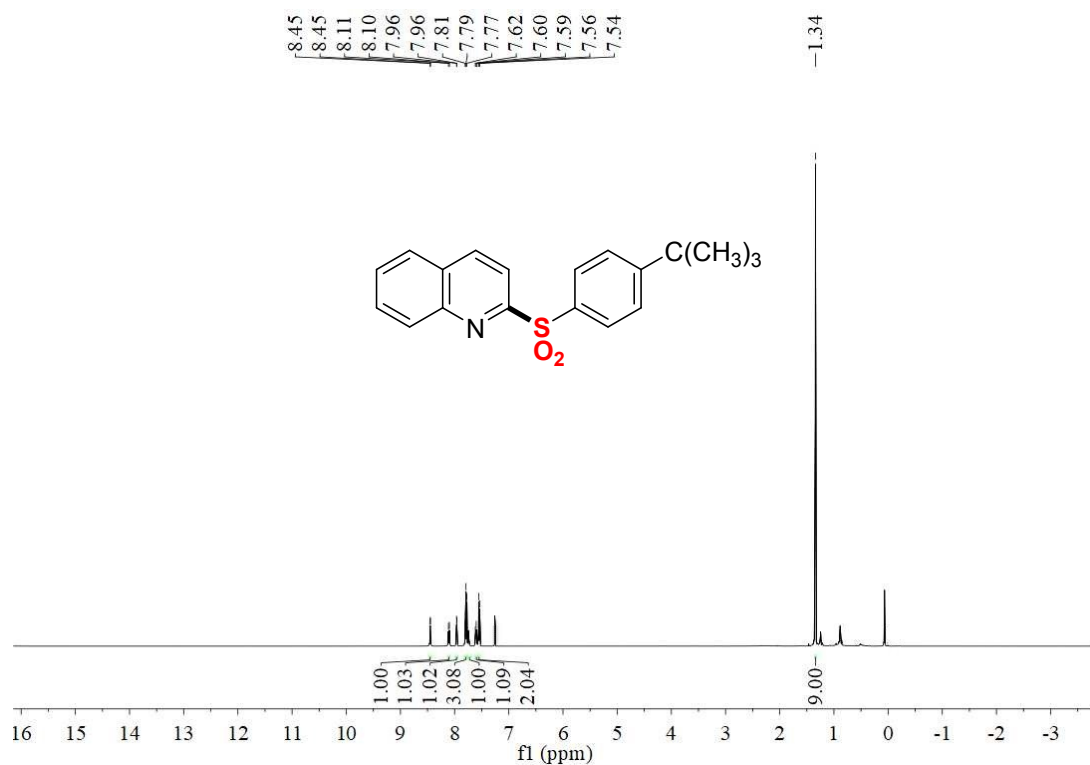
### 3b <sup>1</sup>H NMR



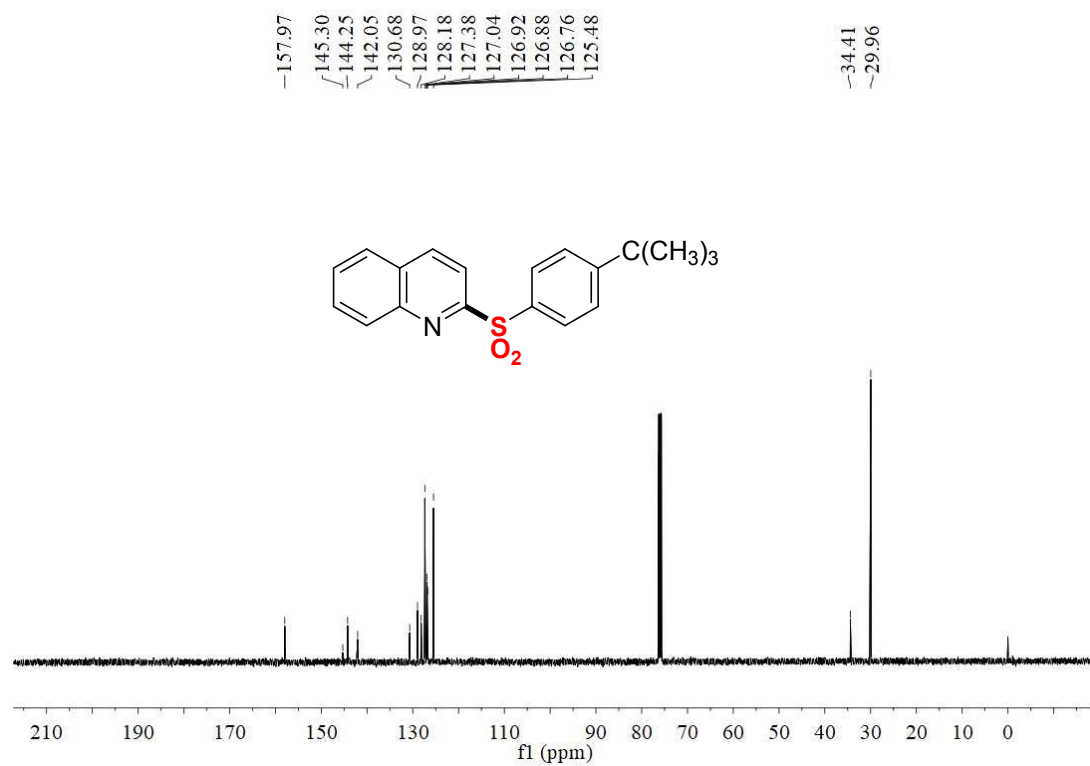
### 3b <sup>13</sup>C NMR



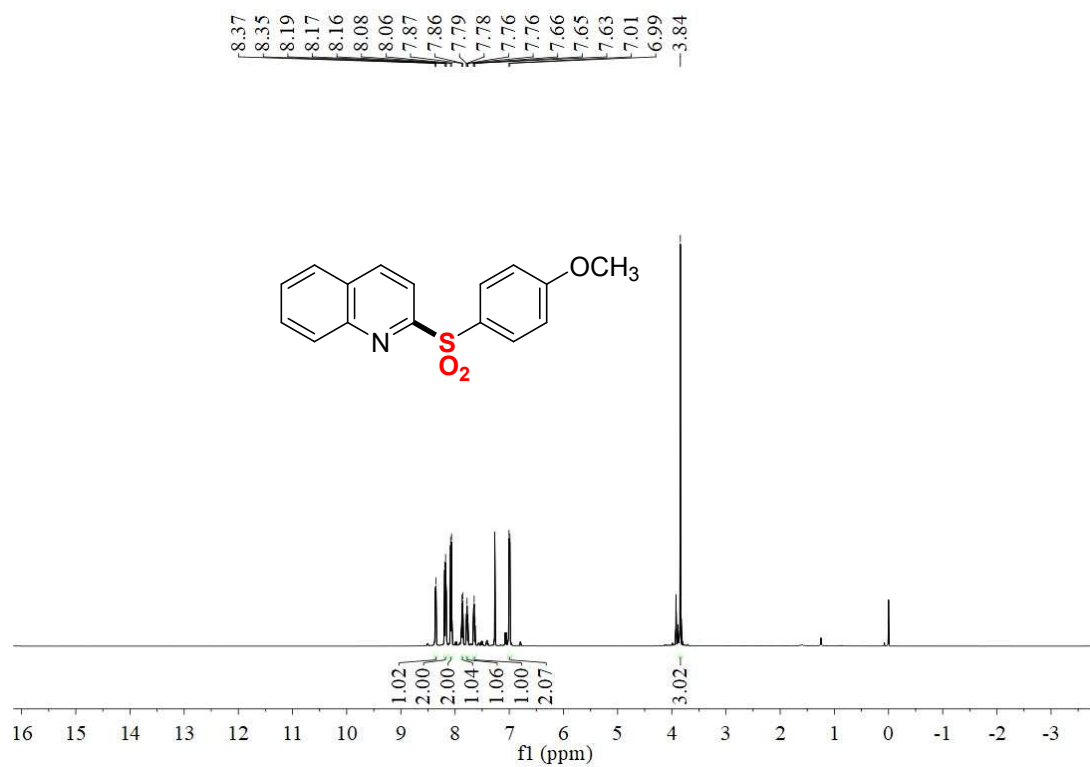
3c <sup>1</sup>H NMR



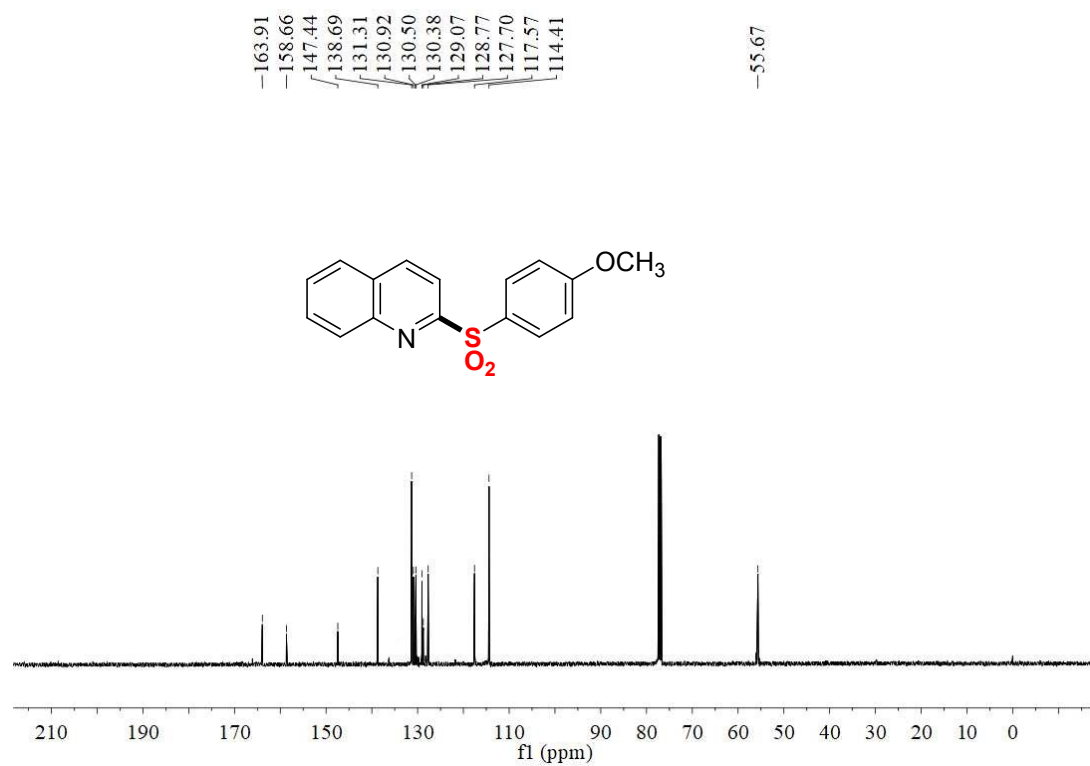
3c <sup>13</sup>C NMR



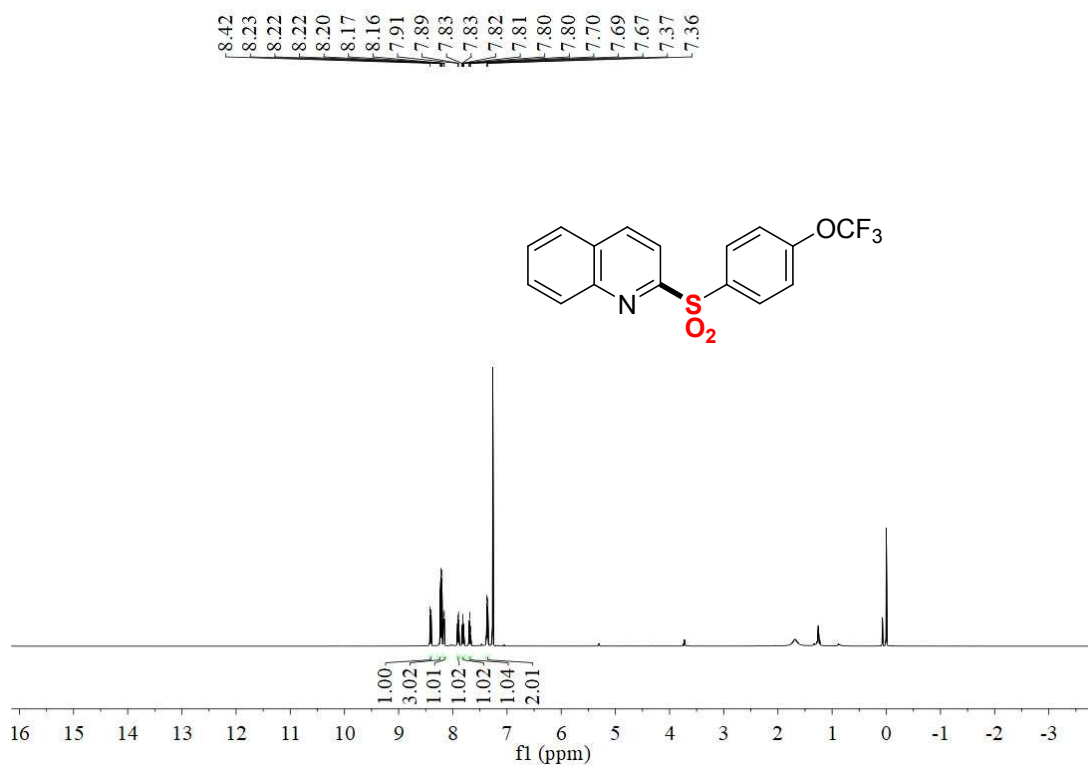
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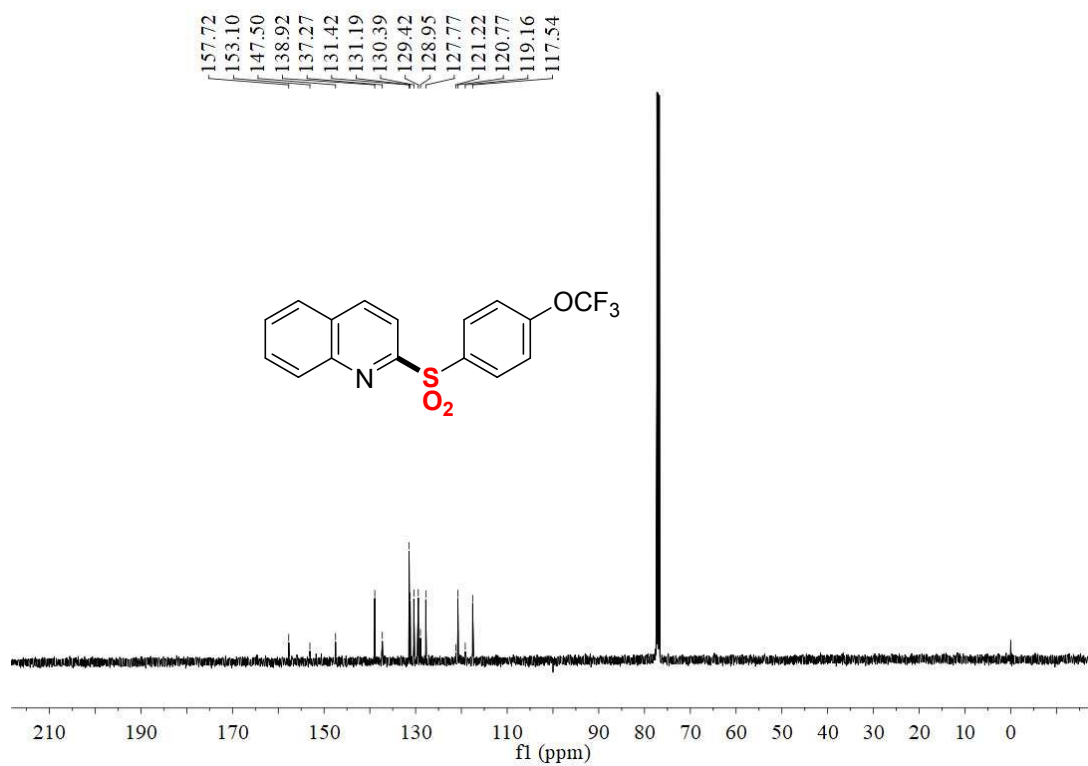
### 3d <sup>13</sup>C NMR



3e <sup>1</sup>H NMR

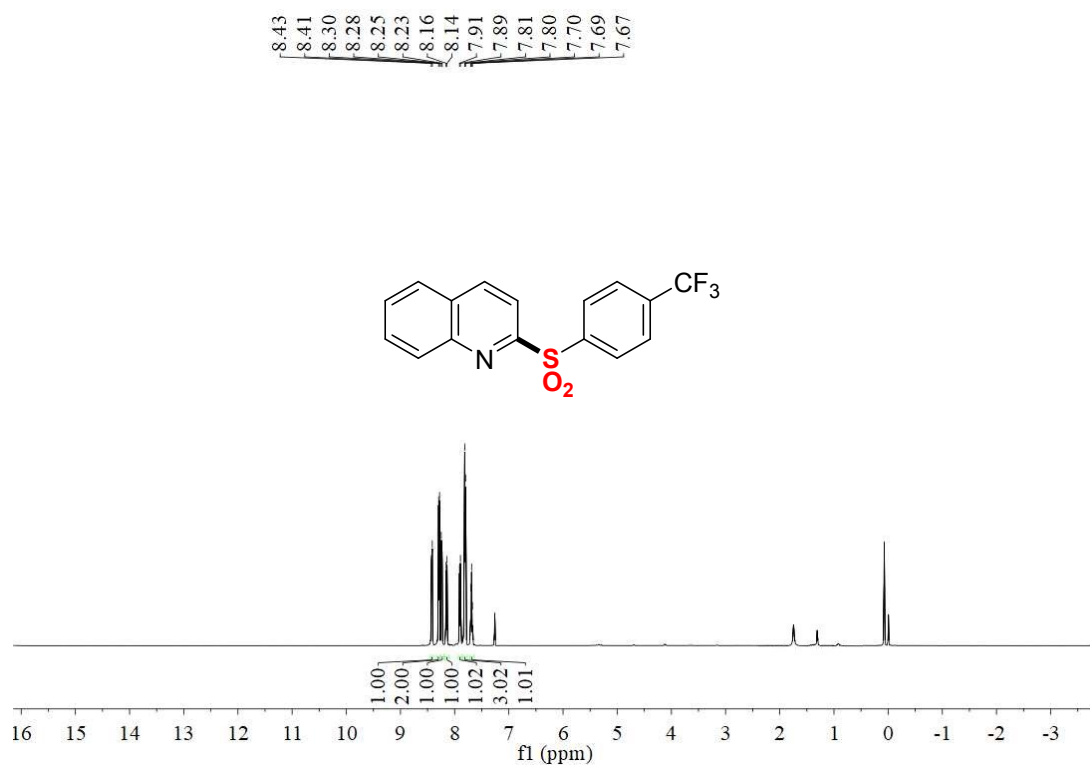


3e <sup>13</sup>C NMR

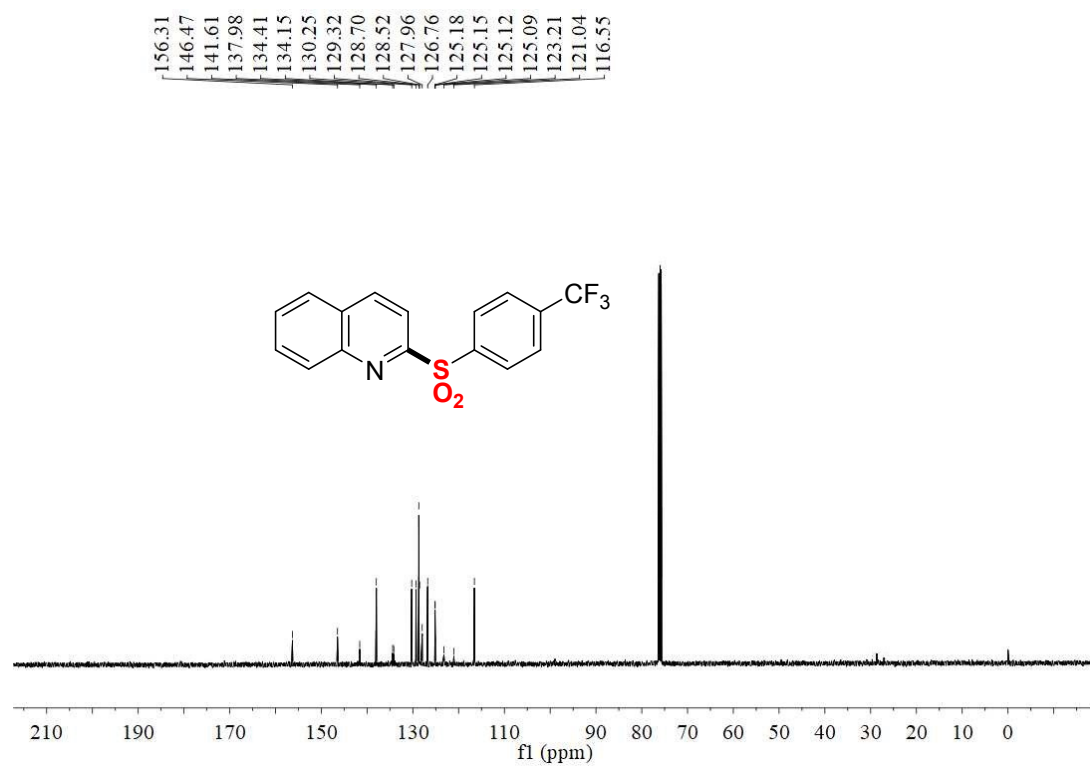




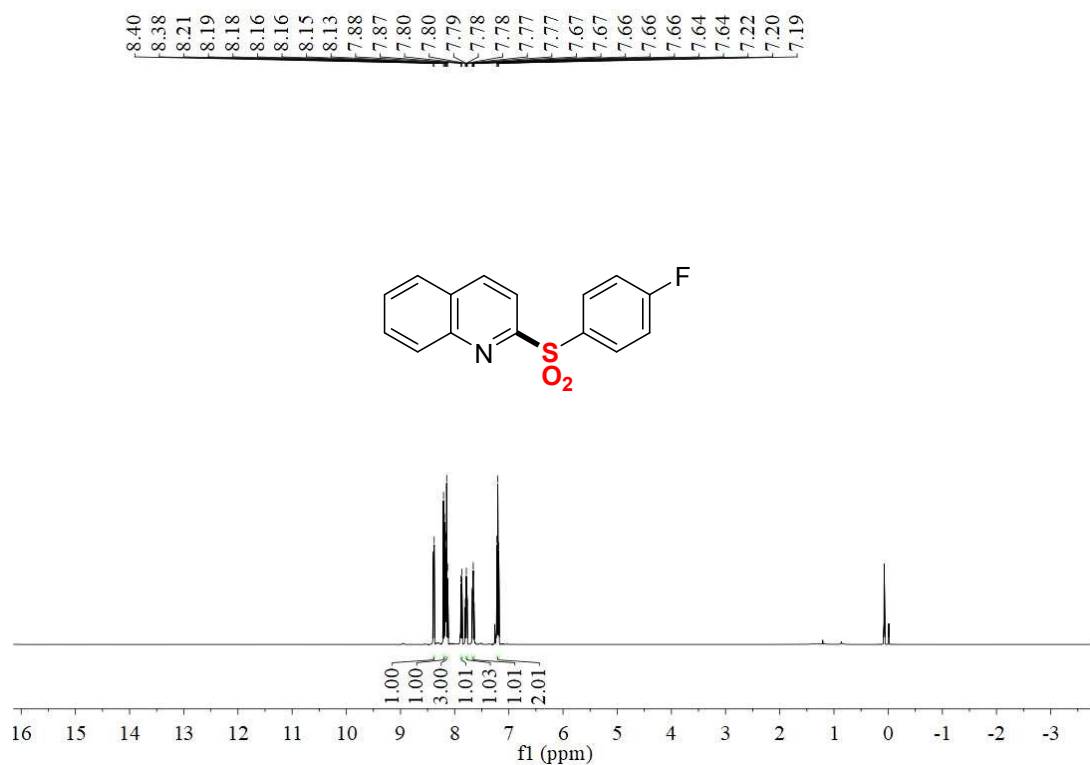
### 3f <sup>1</sup>H NMR



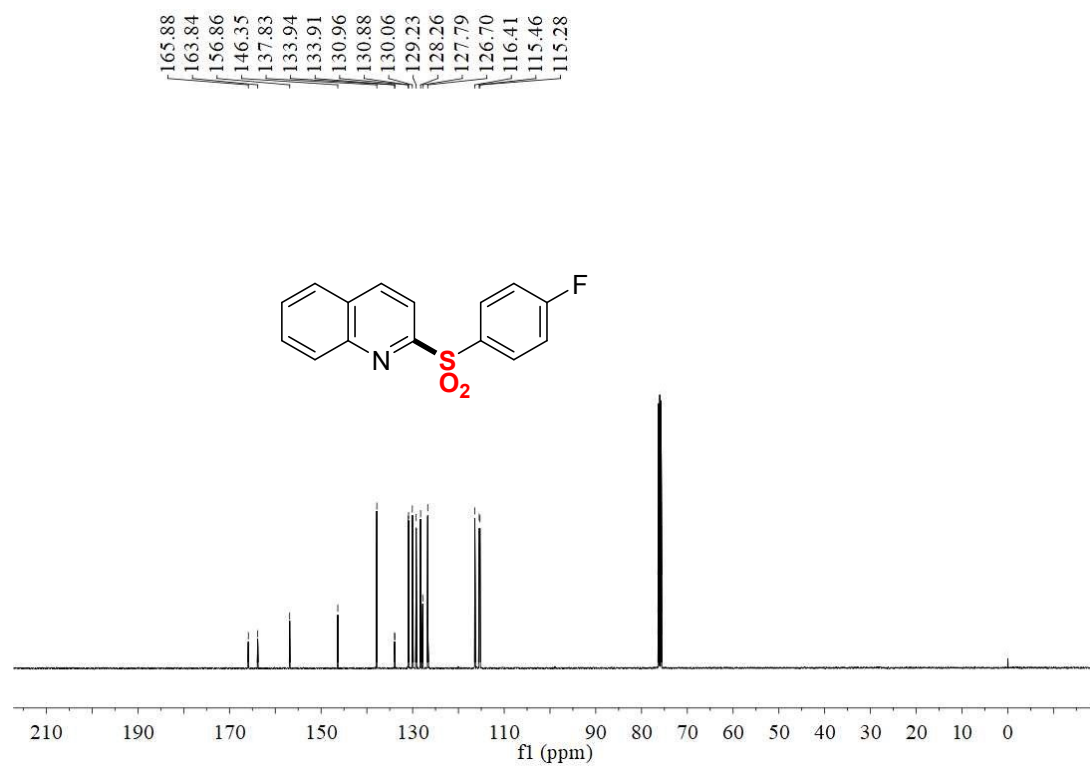
### 3f <sup>13</sup>C NMR



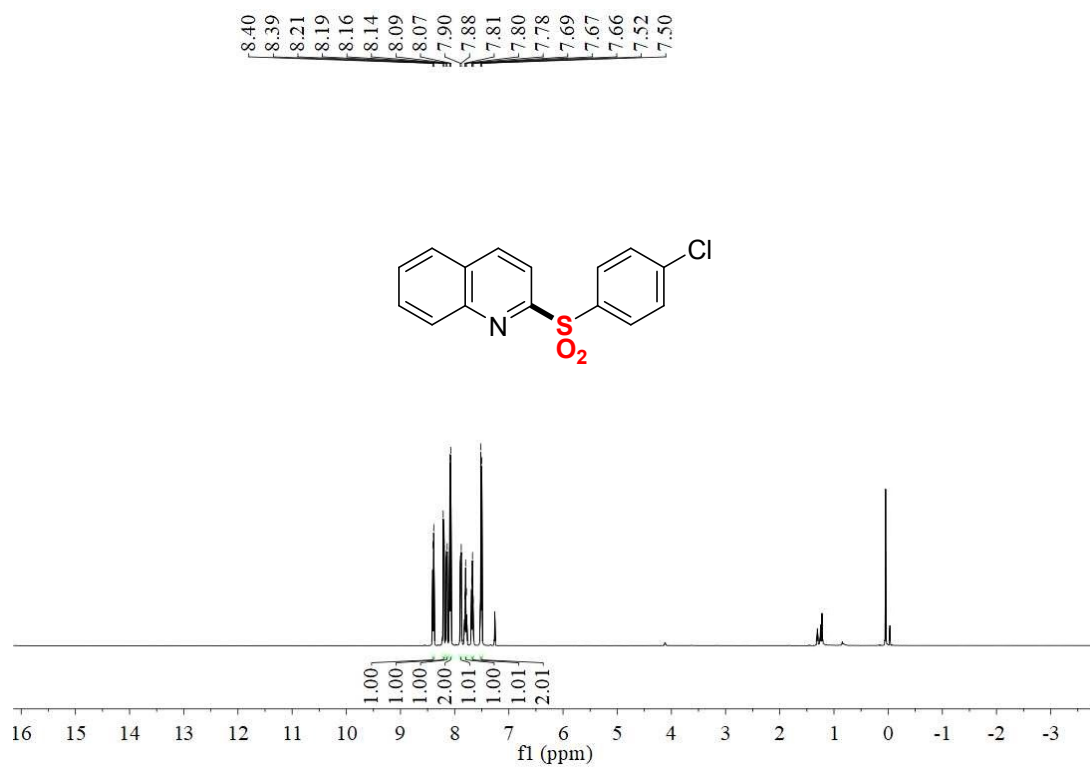
### 3g <sup>1</sup>H NMR



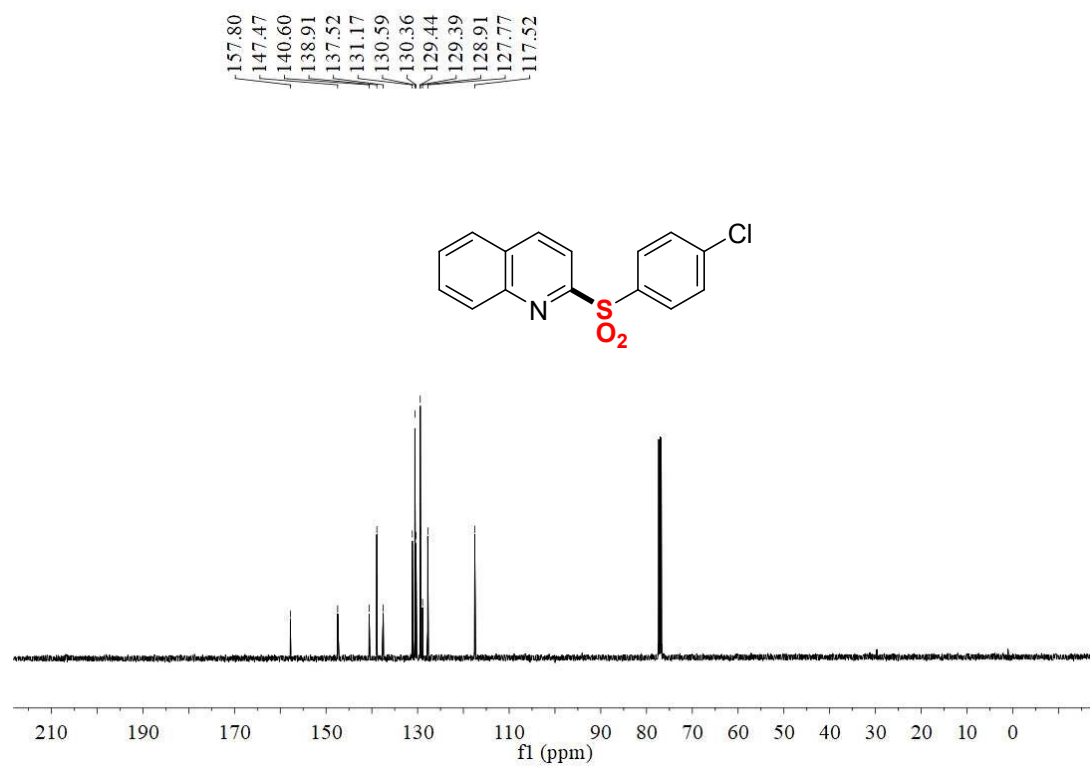
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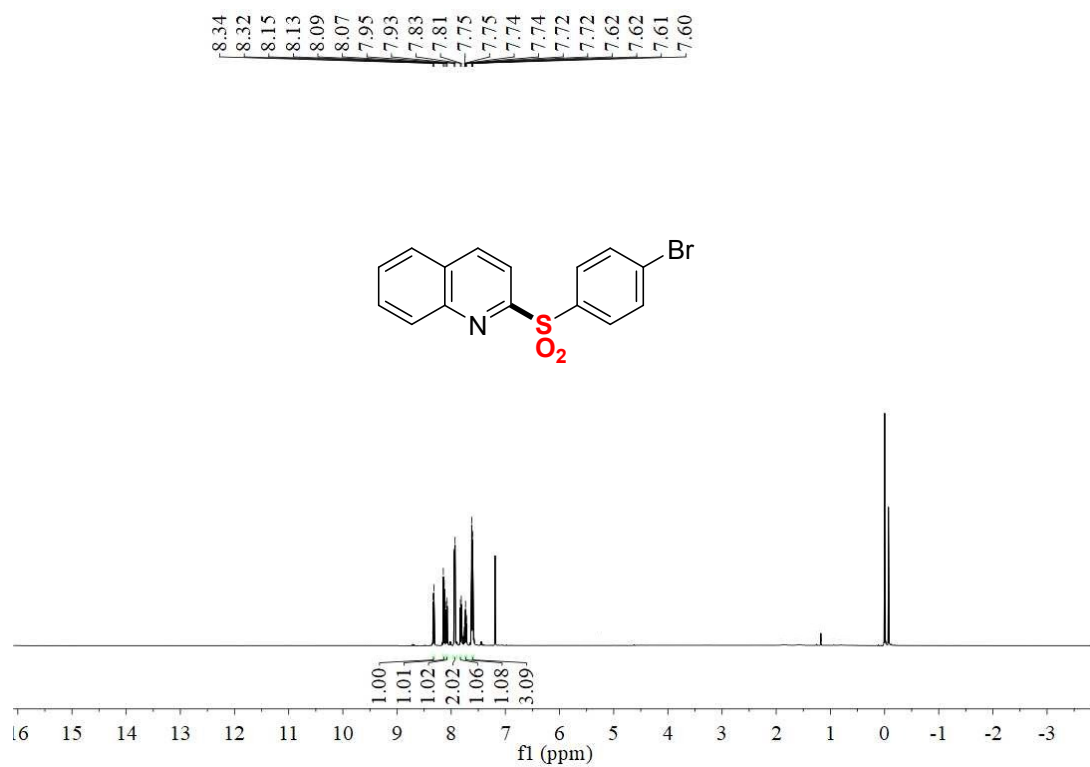
### 3h <sup>1</sup>H NMR



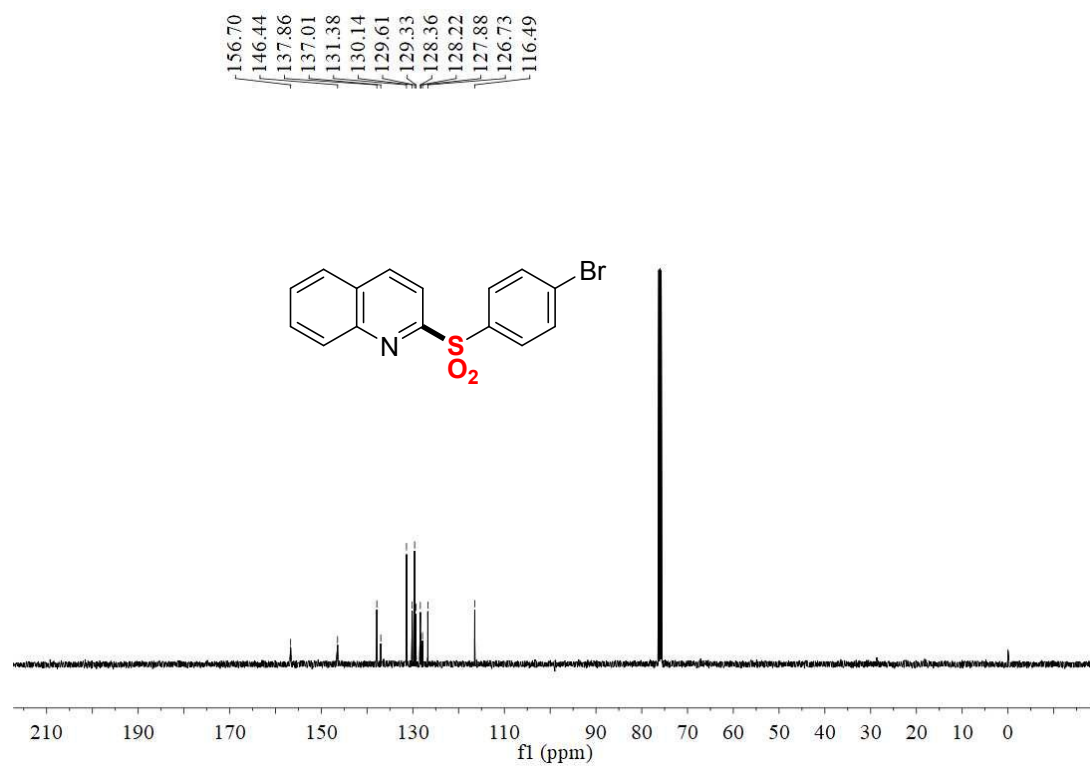
### 3h <sup>13</sup>C NMR



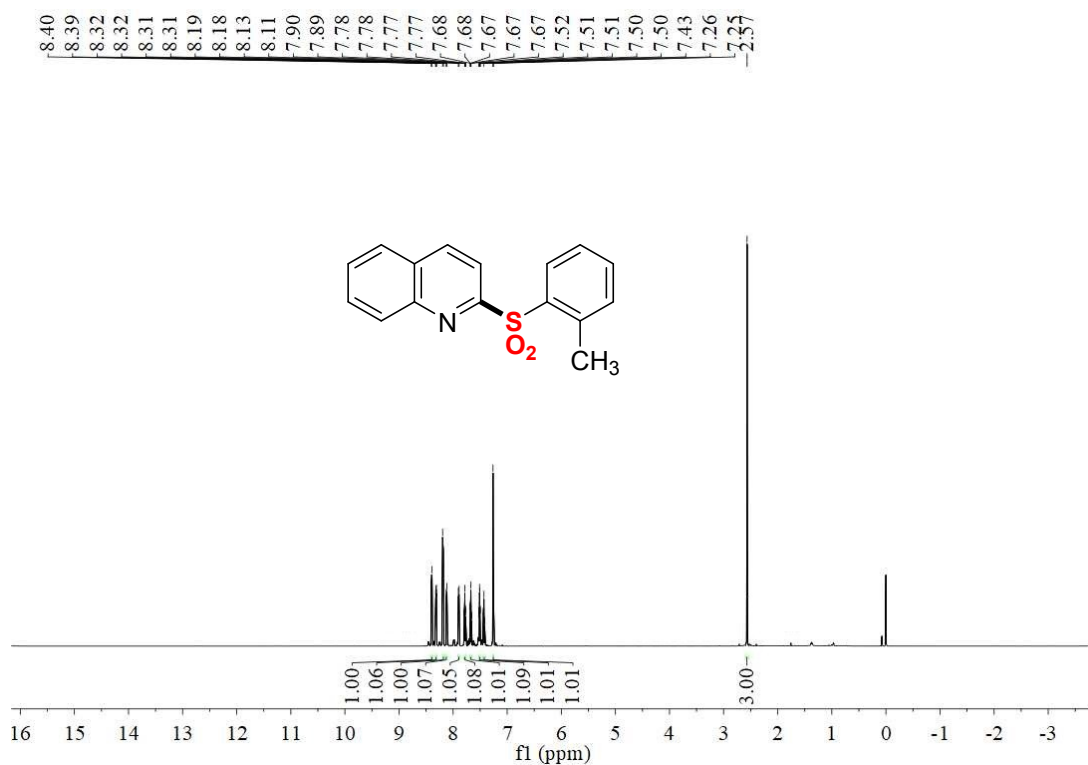
### 3i <sup>1</sup>H NMR



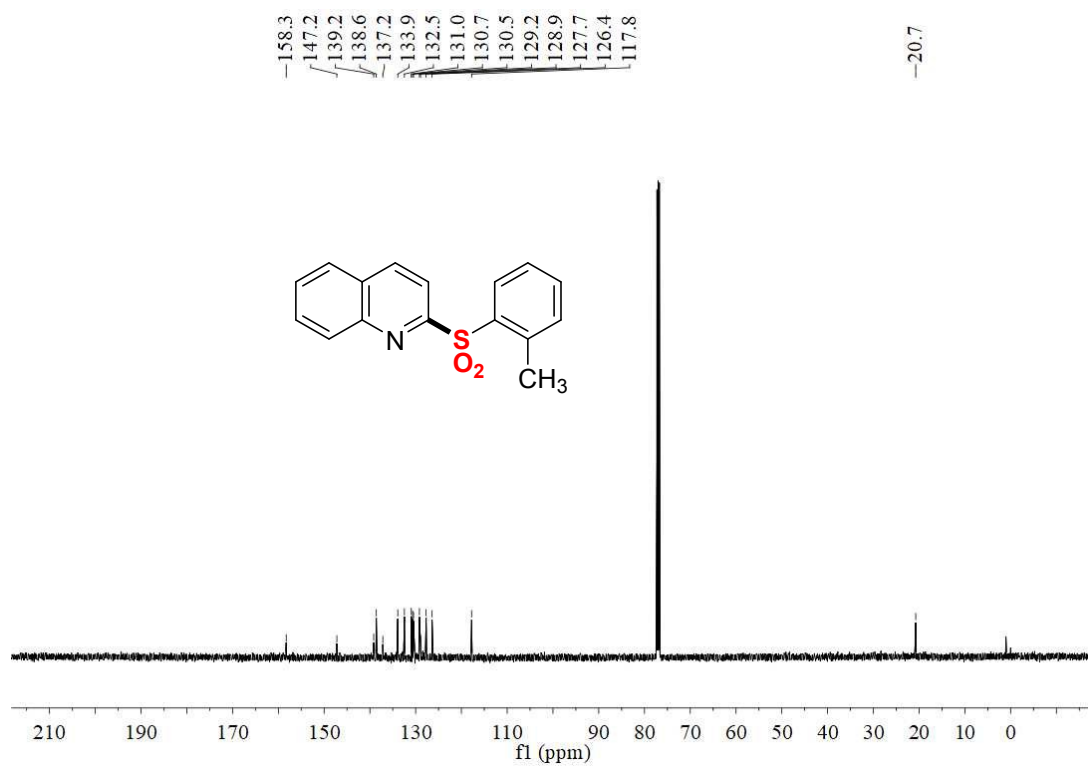
### 3i <sup>13</sup>C NMR



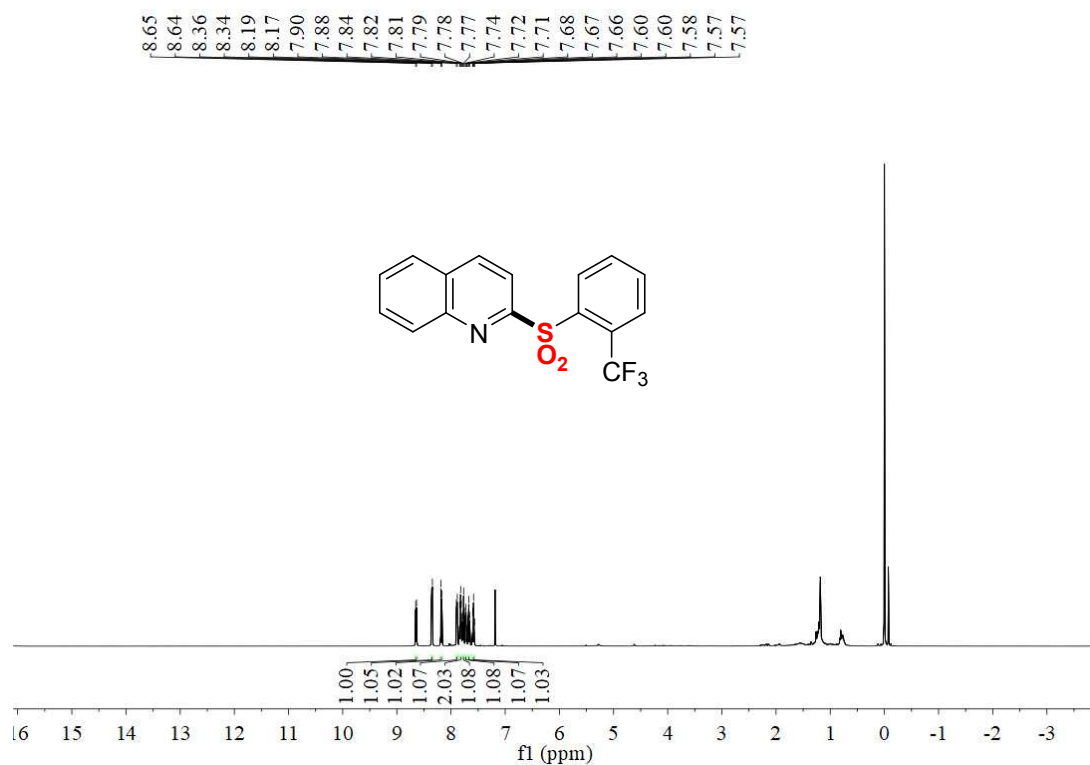
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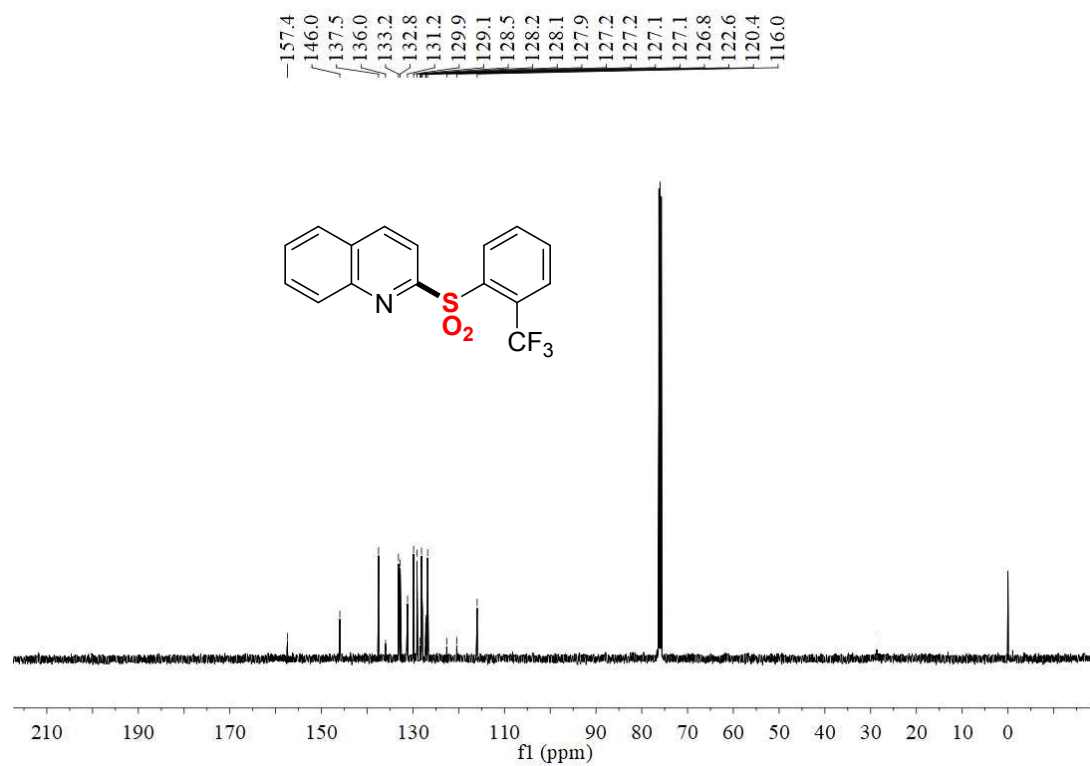
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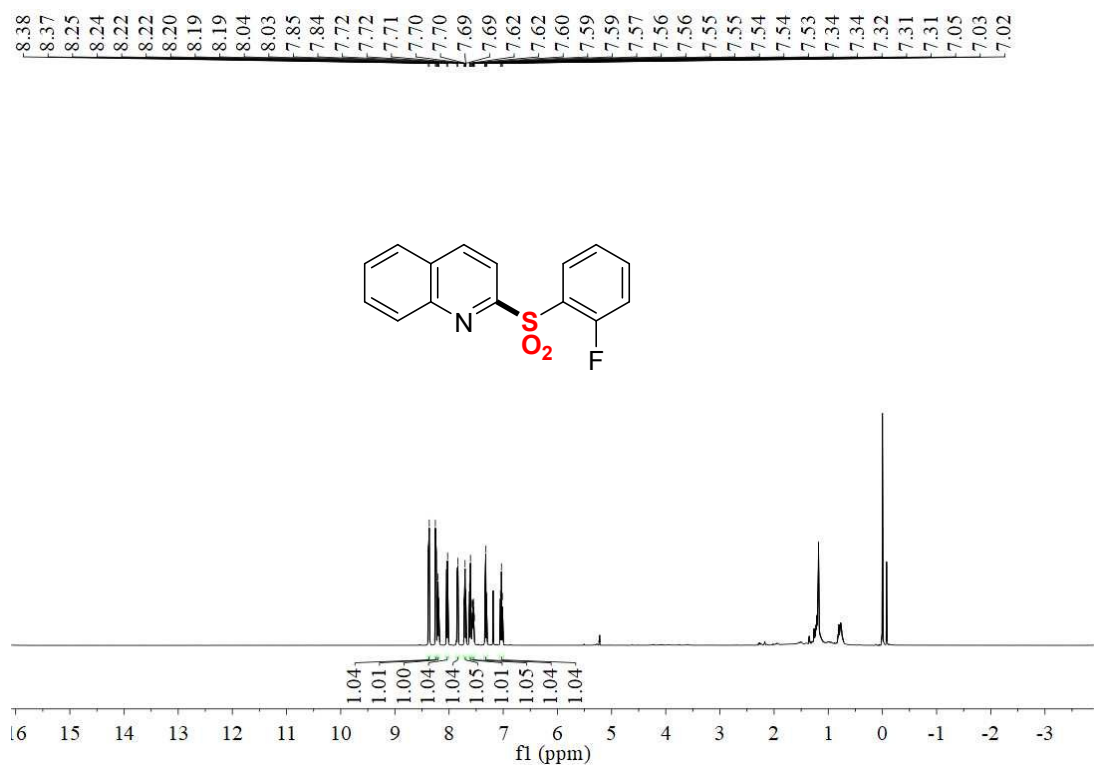
### 3k <sup>1</sup>H NMR



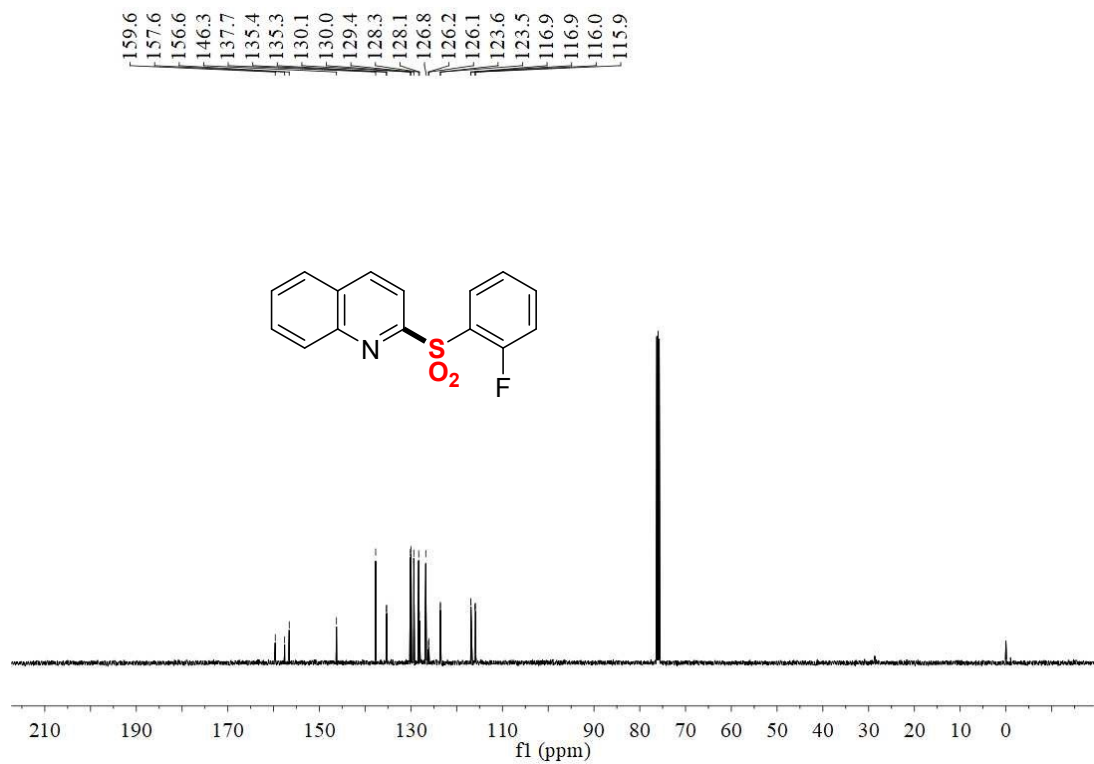
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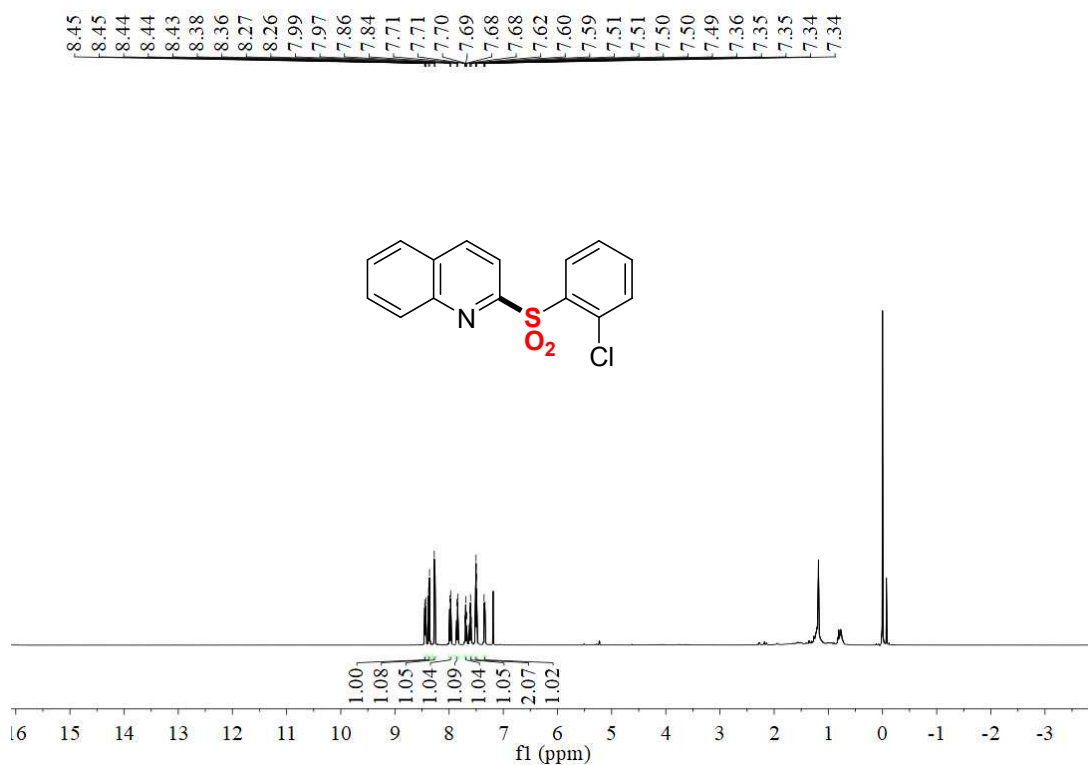
### 3I <sup>1</sup>H NMR



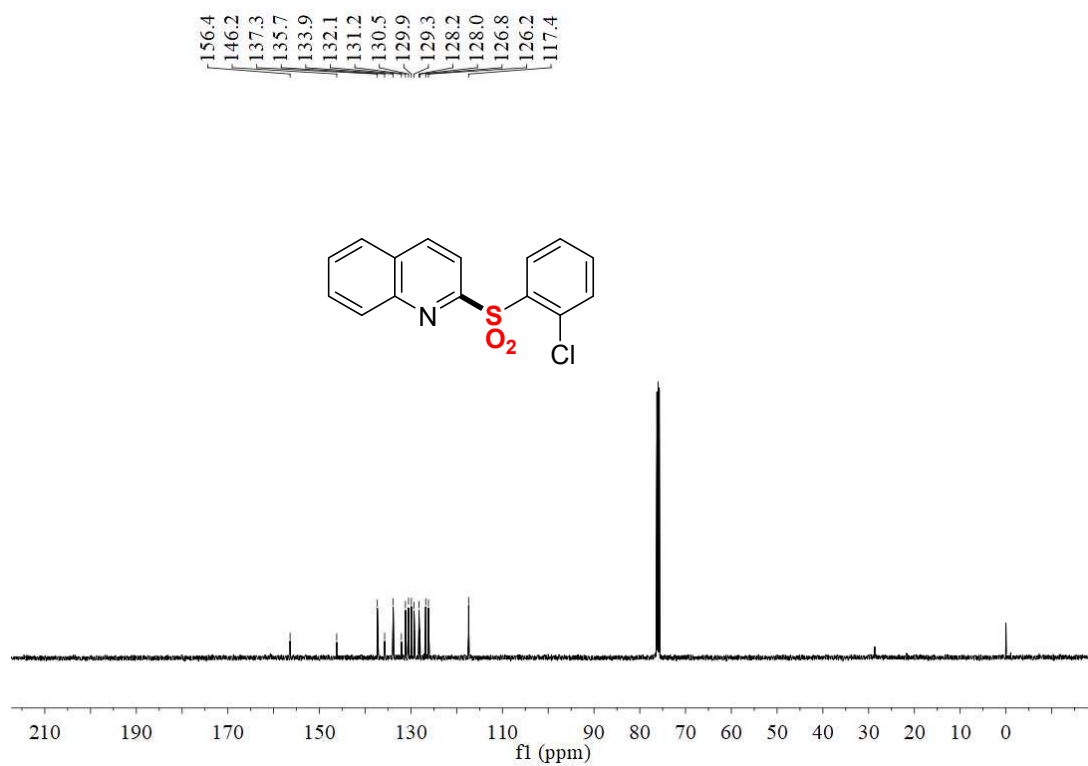
### 3I <sup>13</sup>C NMR



### 3m <sup>1</sup>H NMR

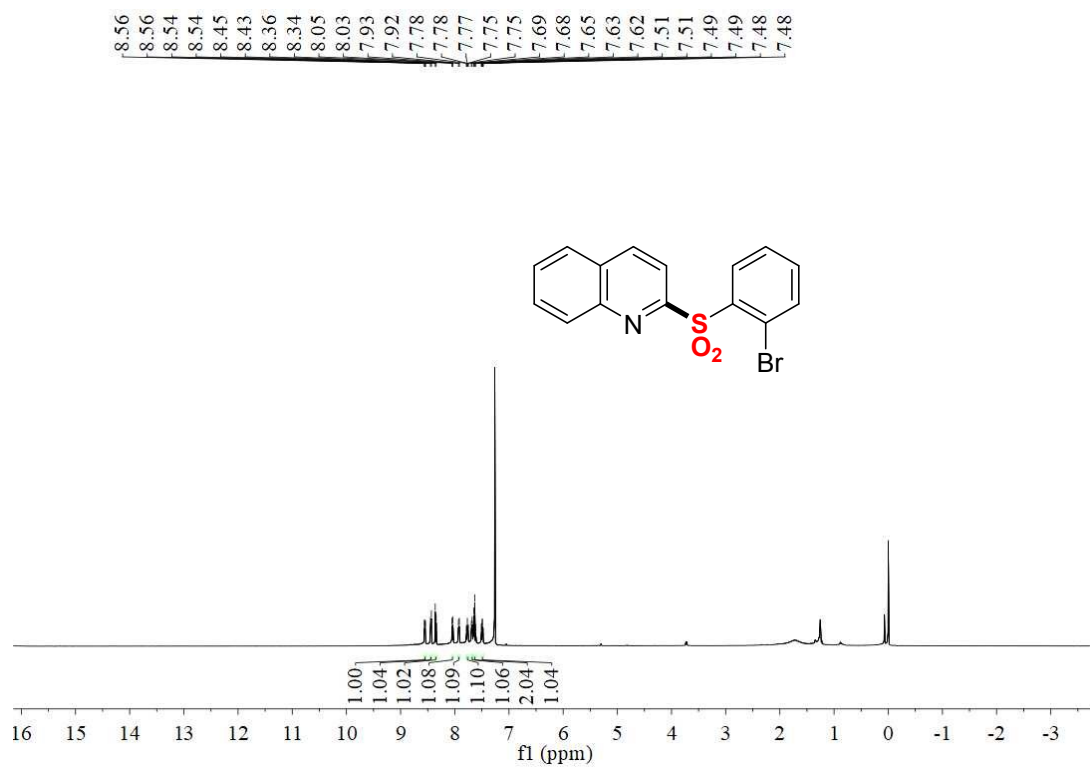


### 3m <sup>13</sup>C NMR

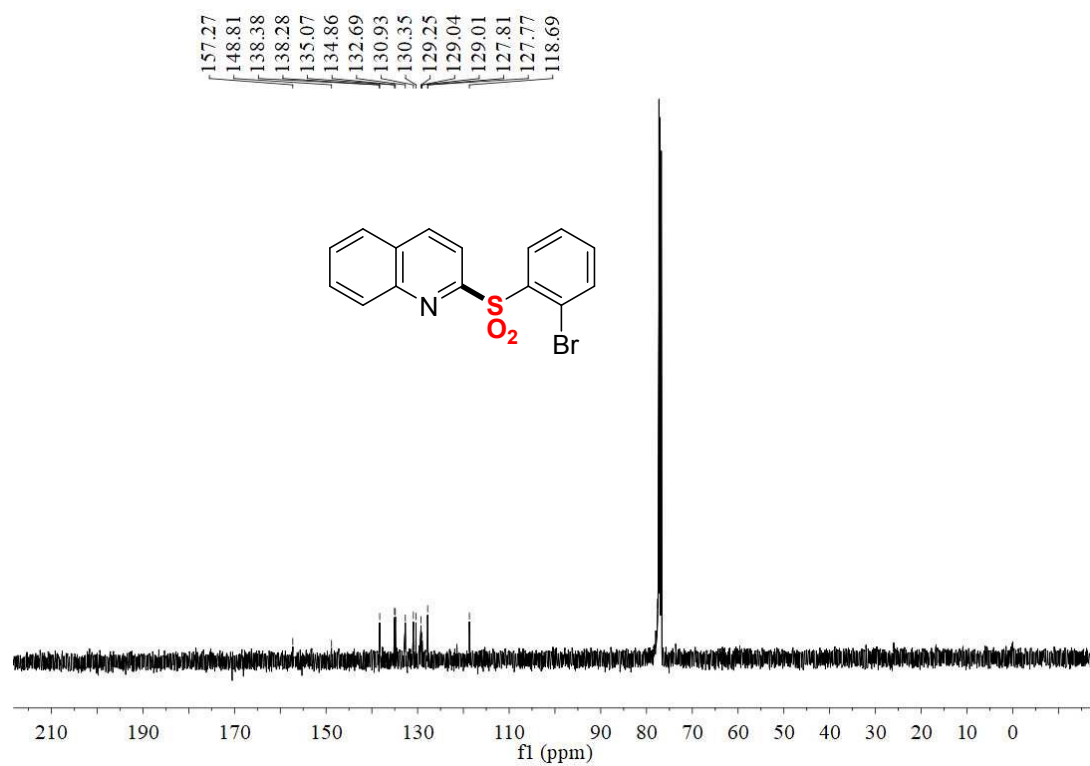




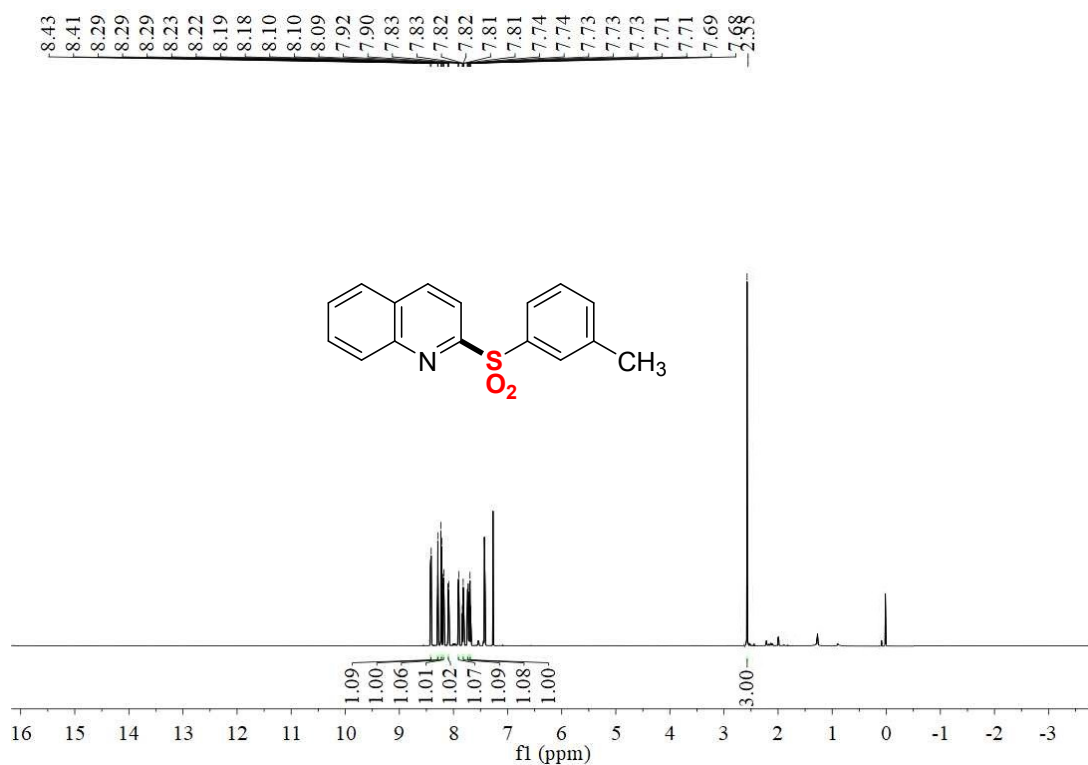
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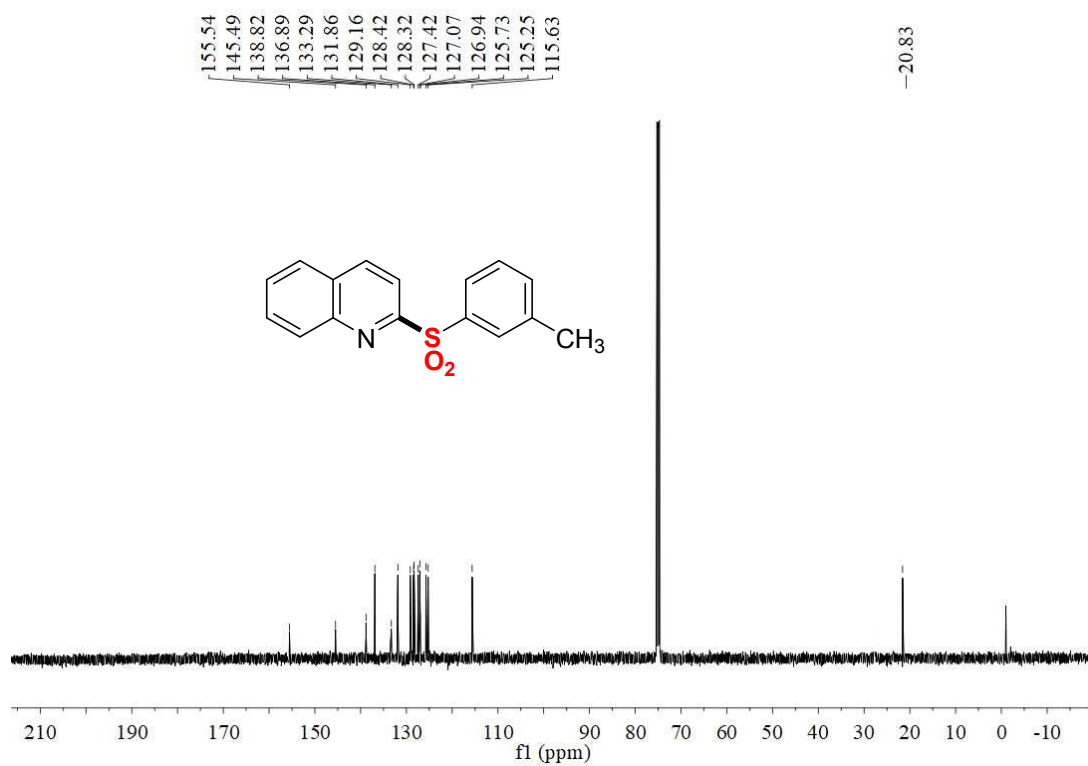
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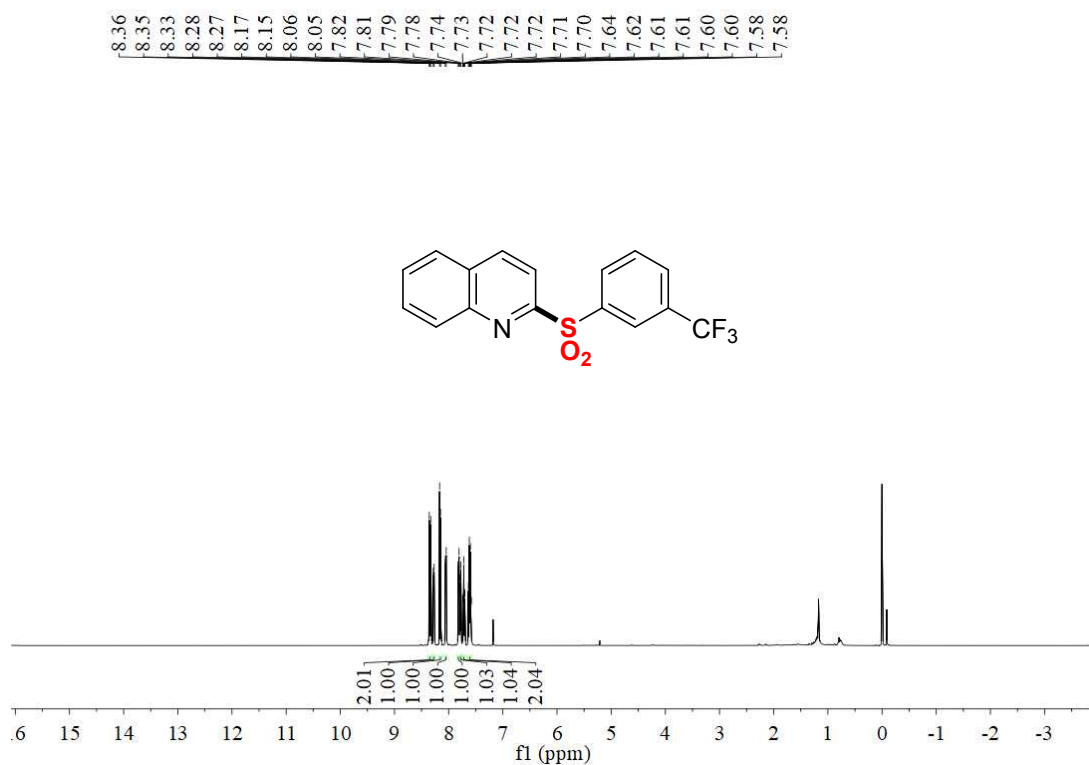
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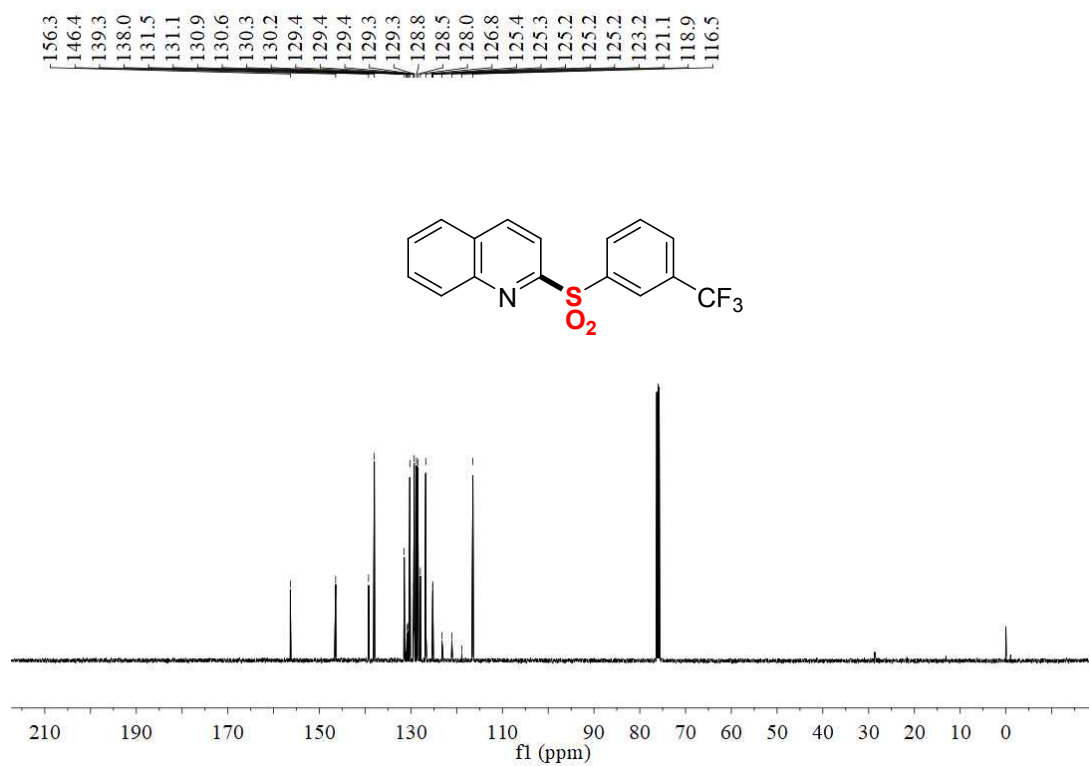
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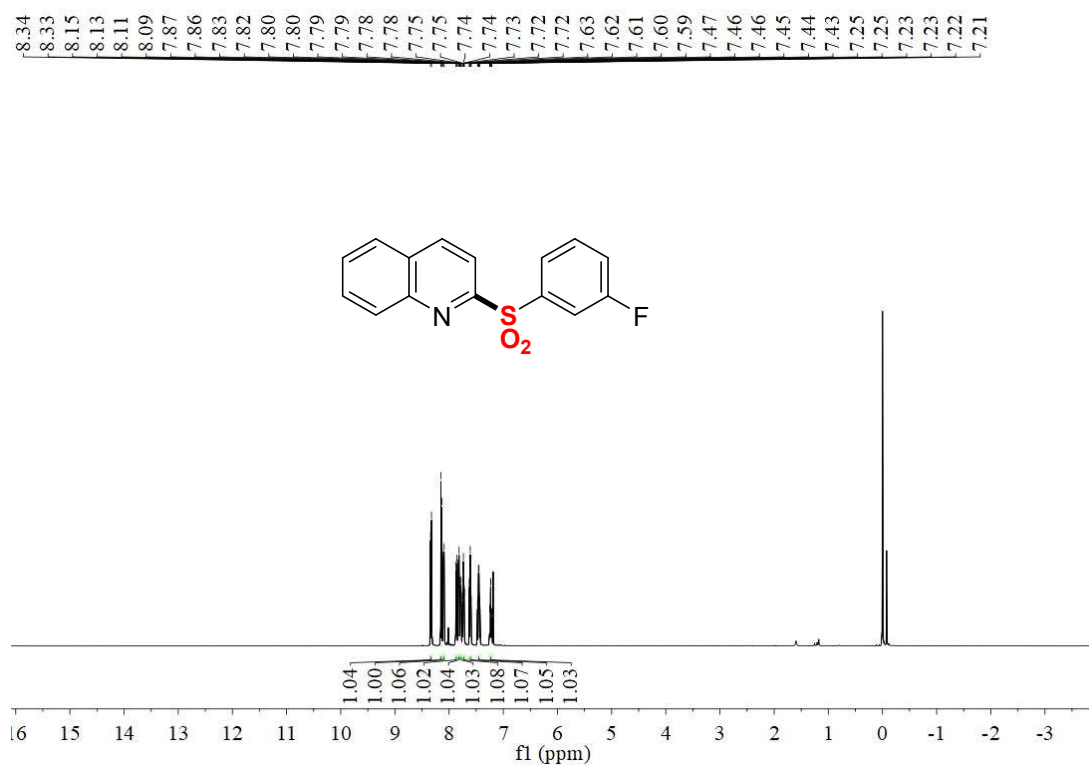
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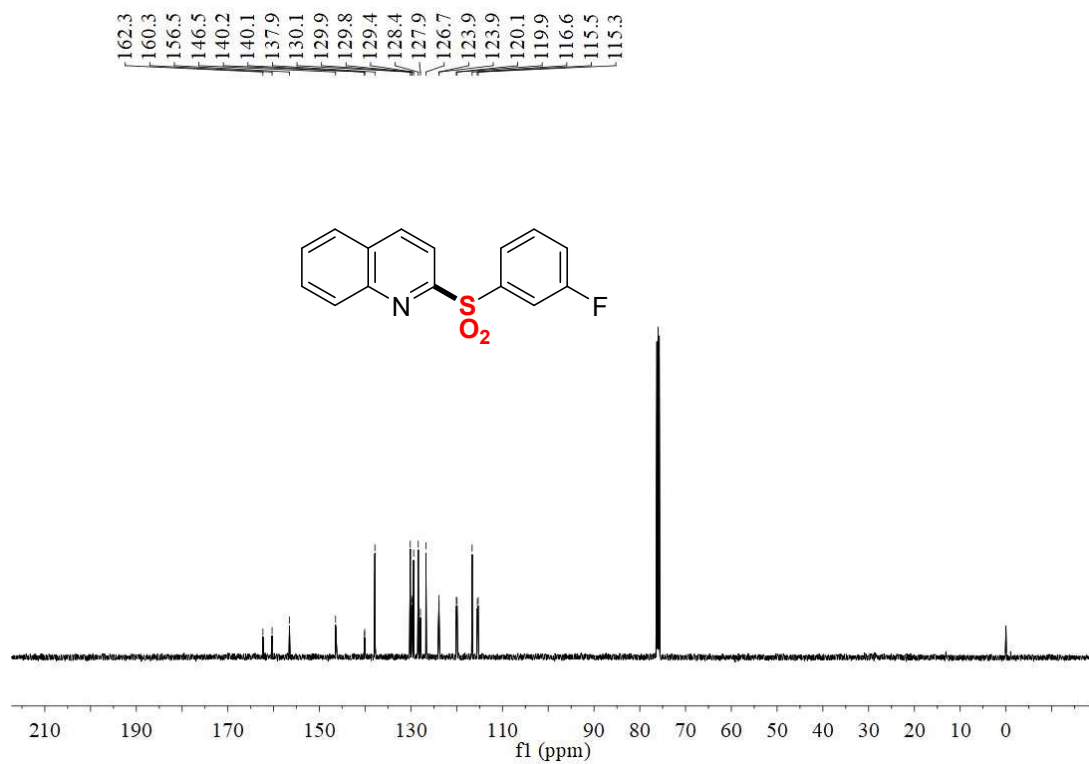
### 3p <sup>13</sup>C NMR



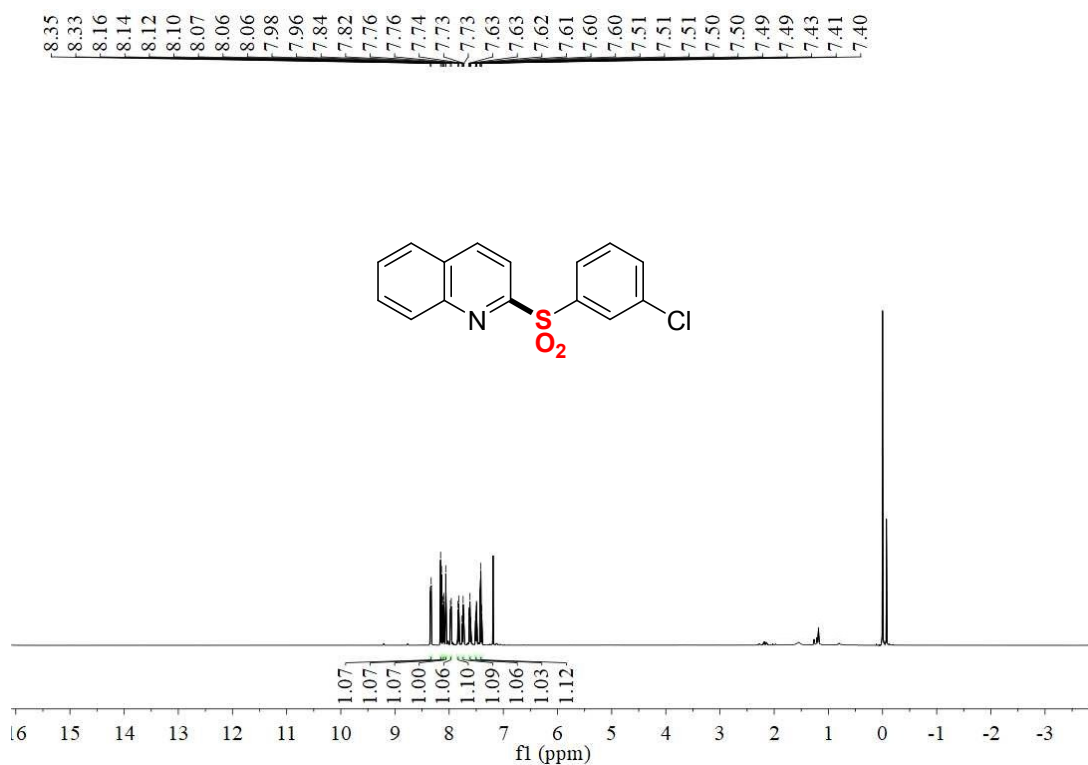
### 3q <sup>1</sup>H NMR



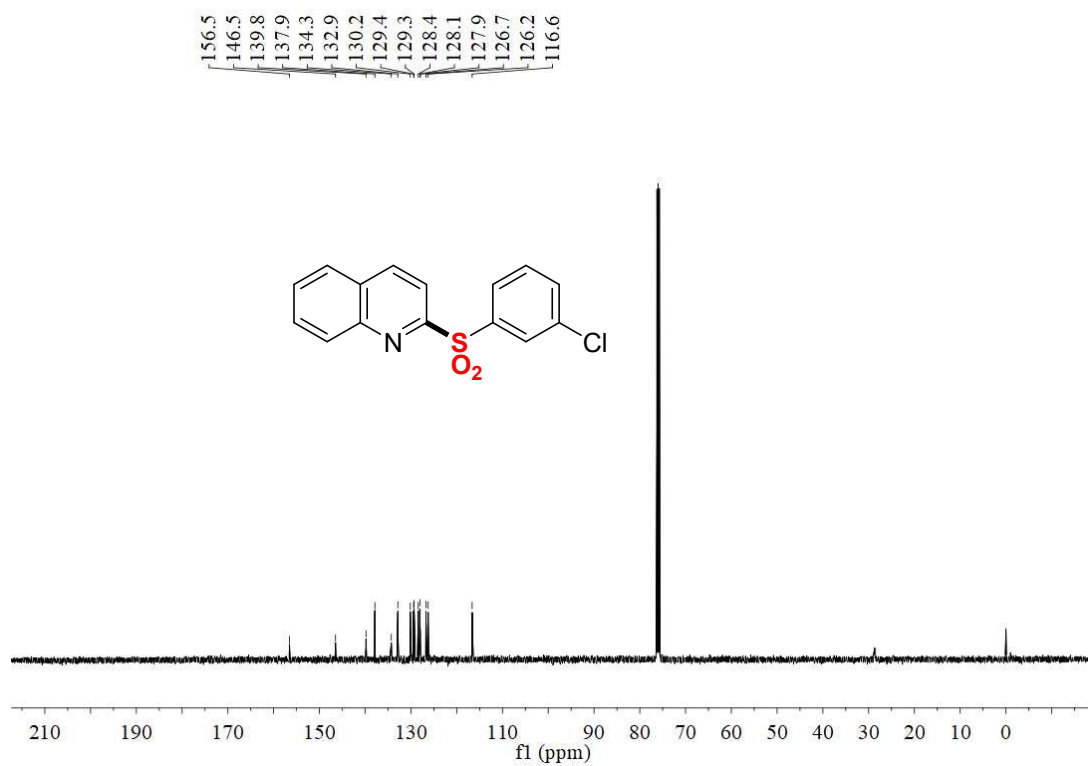
### 3q <sup>13</sup>C NMR



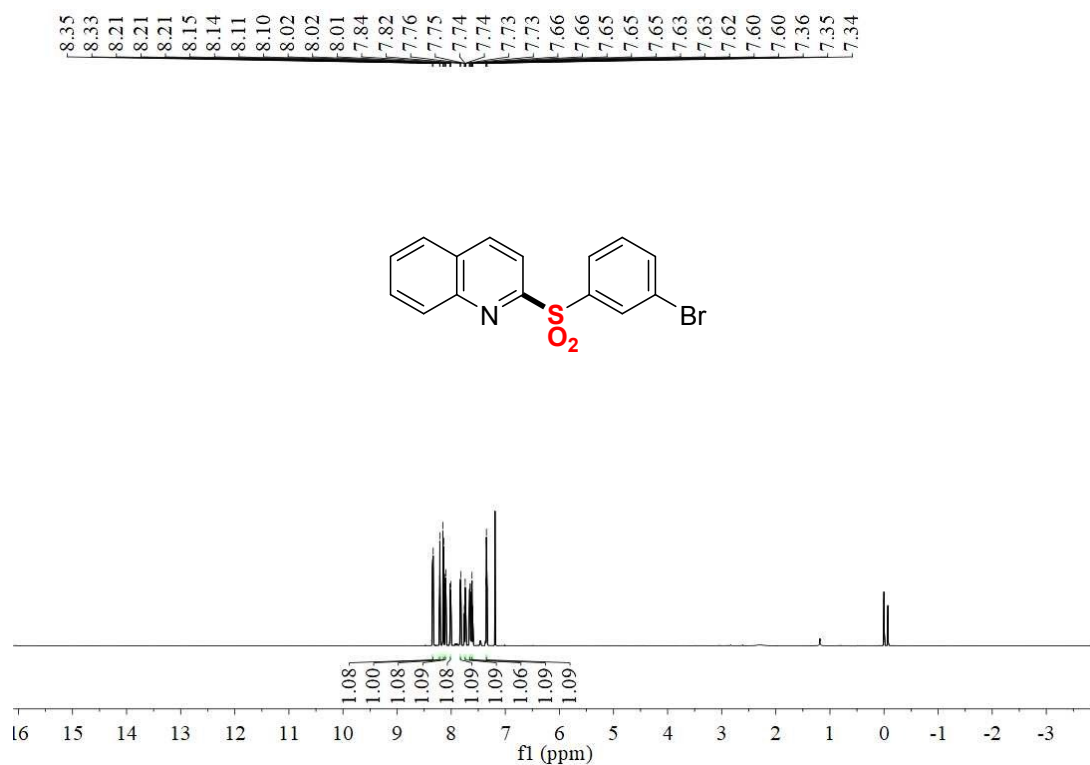
### 3r <sup>1</sup>H NMR



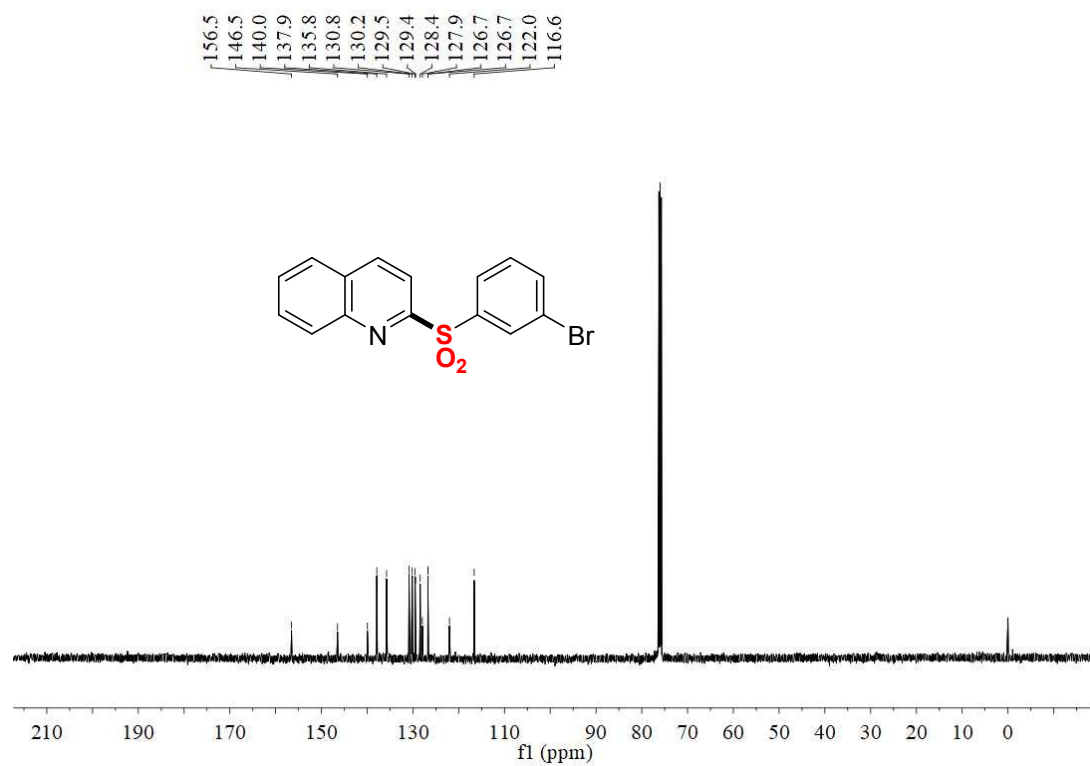
### 3r <sup>13</sup>C NMR



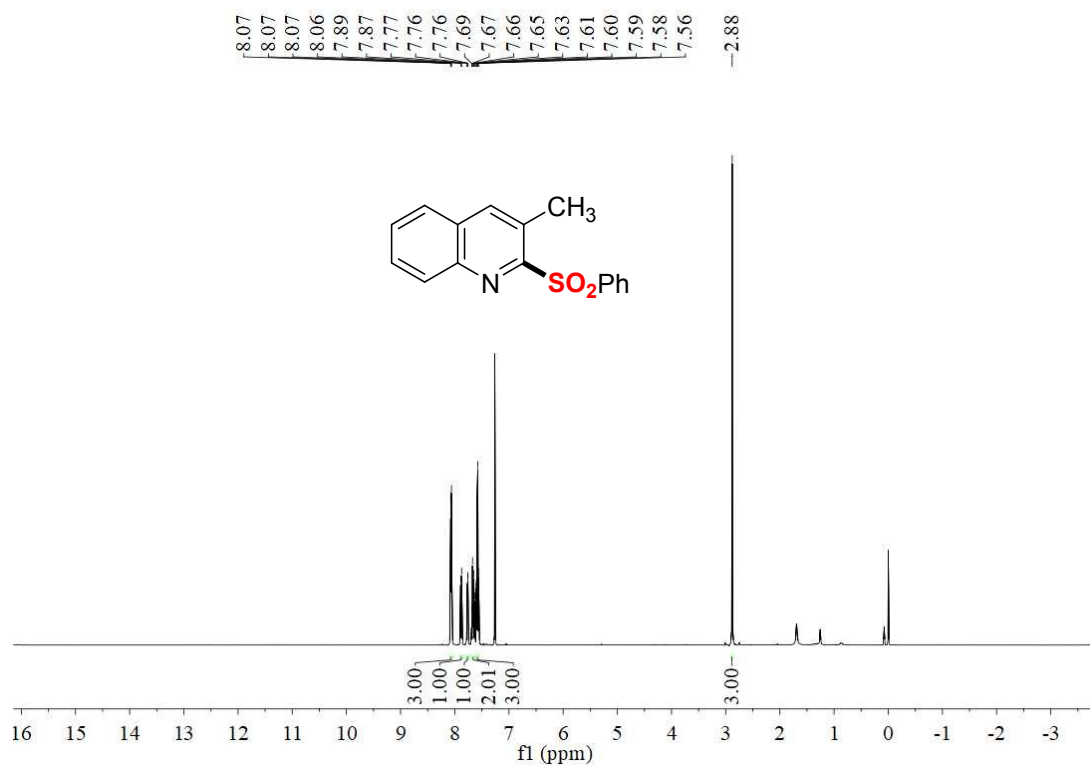
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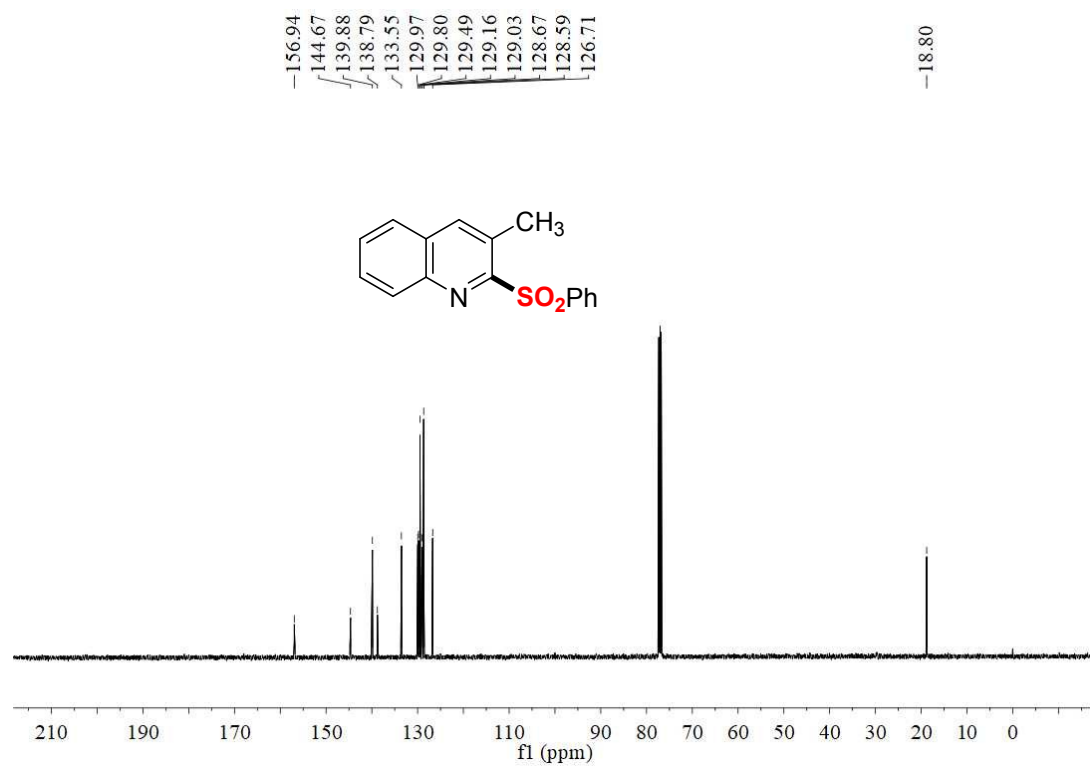
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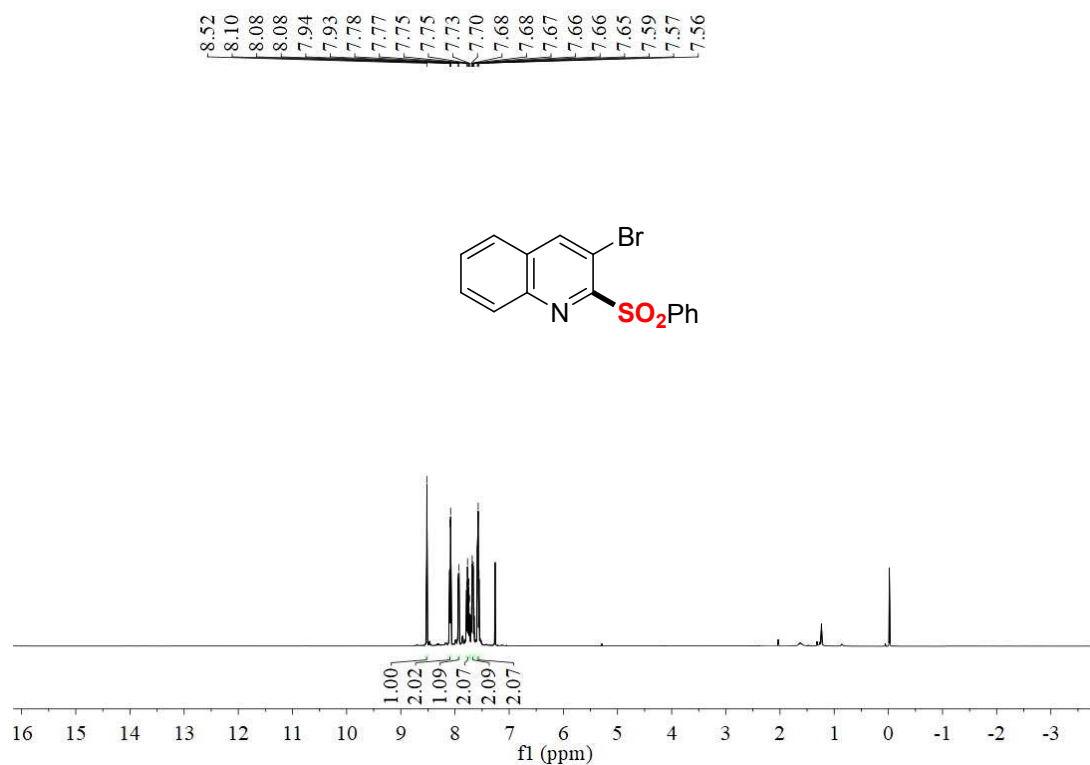
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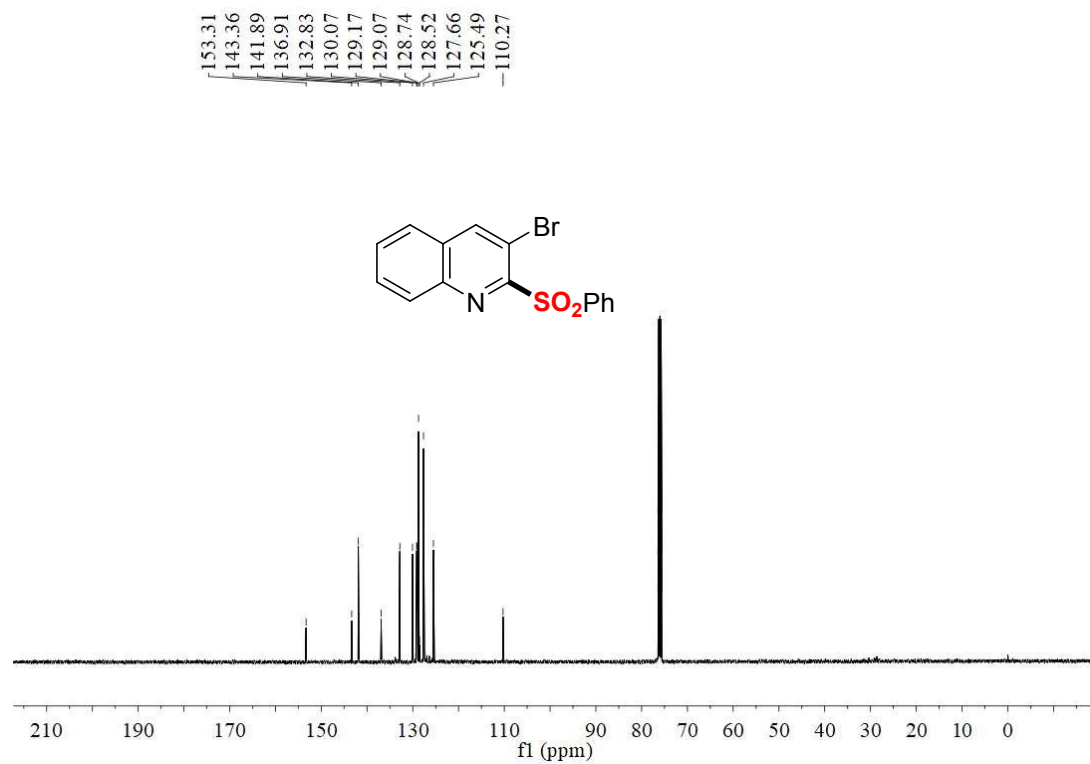
3t <sup>13</sup>C NMR



### 3u <sup>1</sup>H NMR

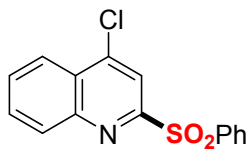
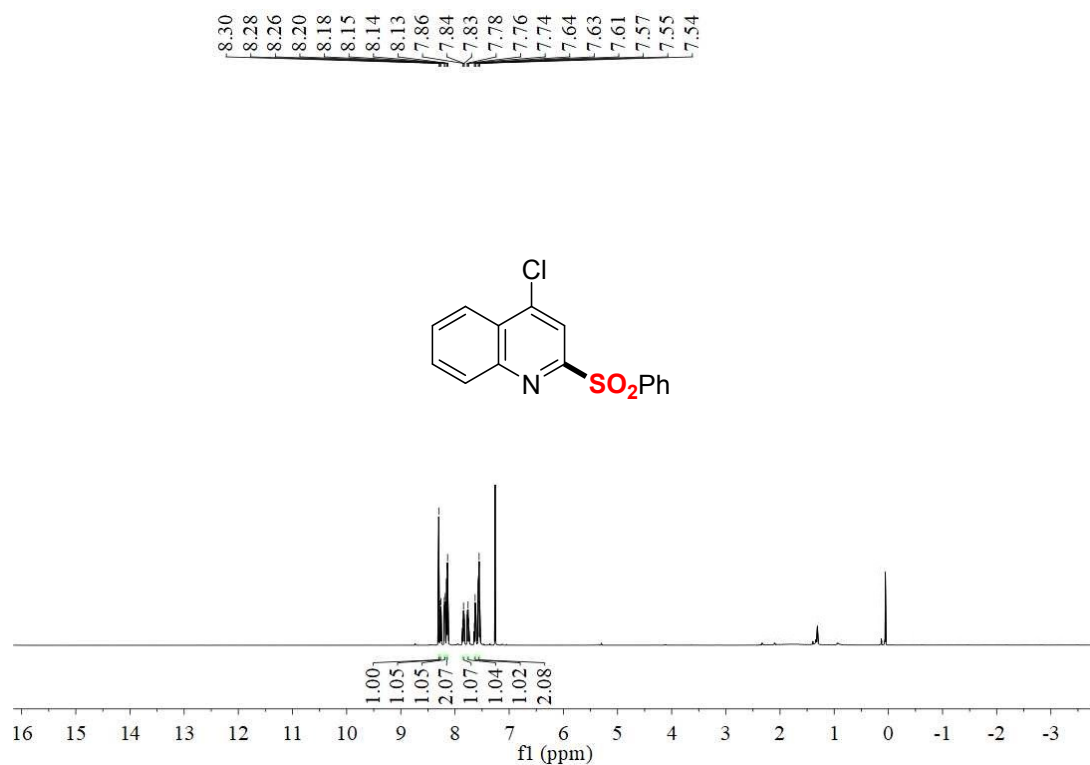


### 3u <sup>13</sup>C NMR

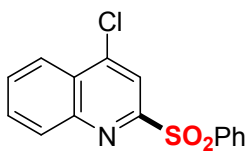
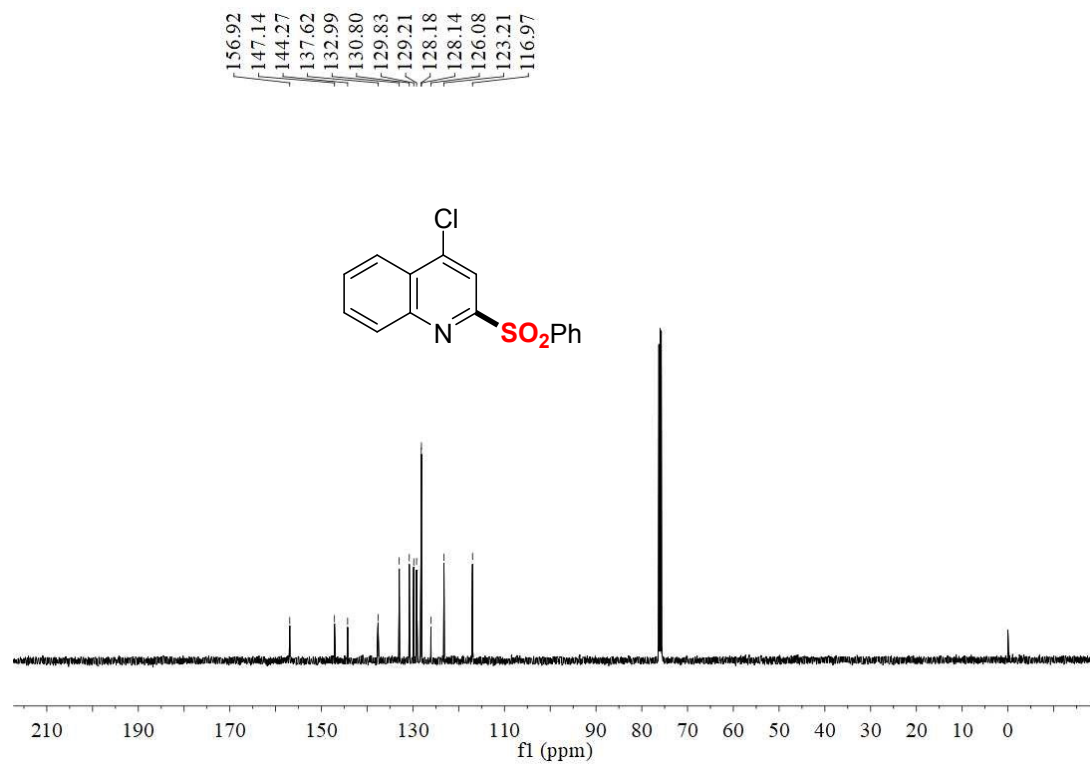




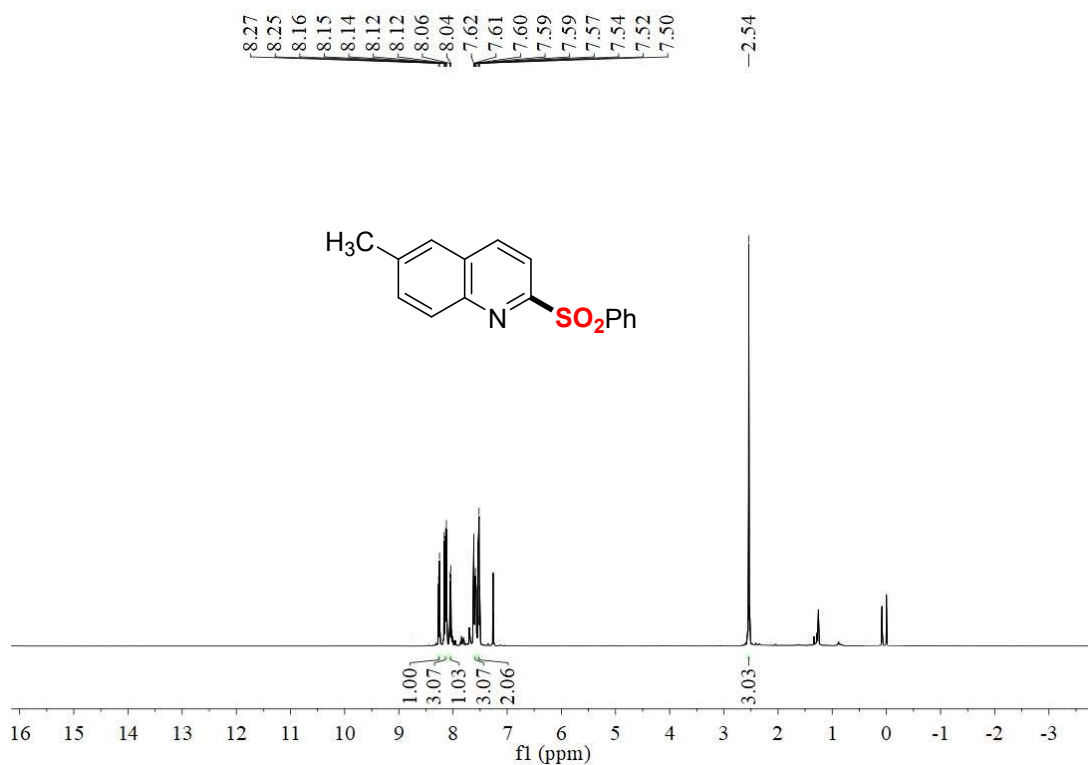
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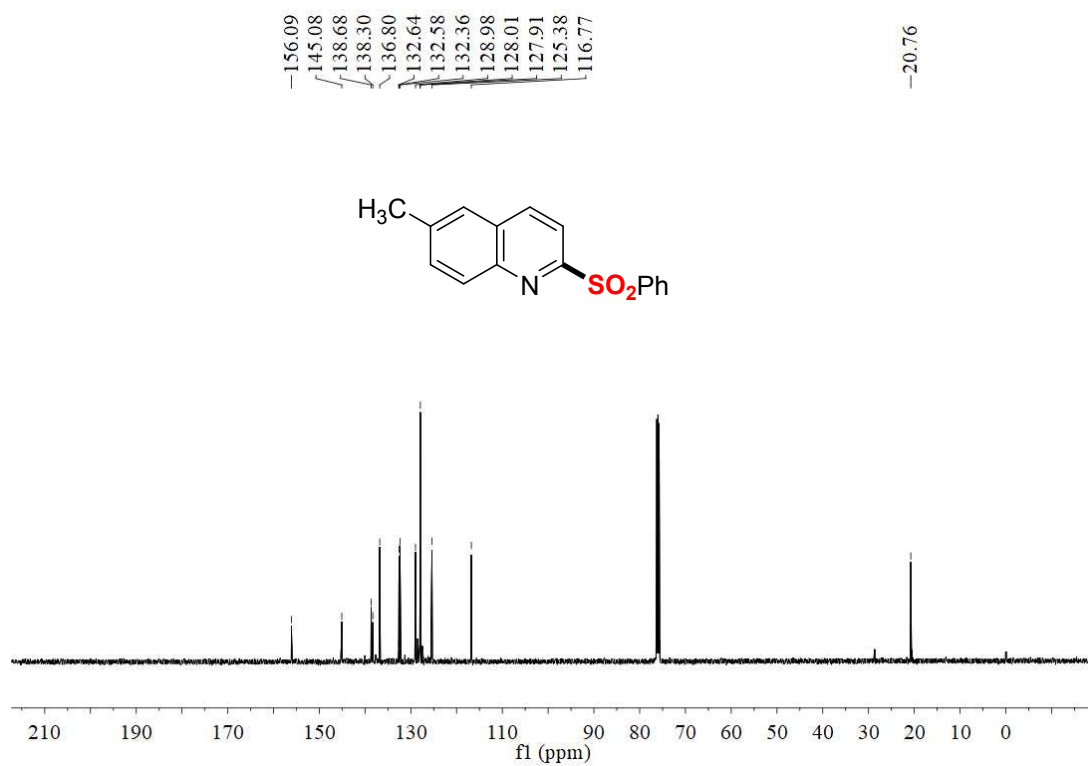
### 3v <sup>13</sup>C NMR



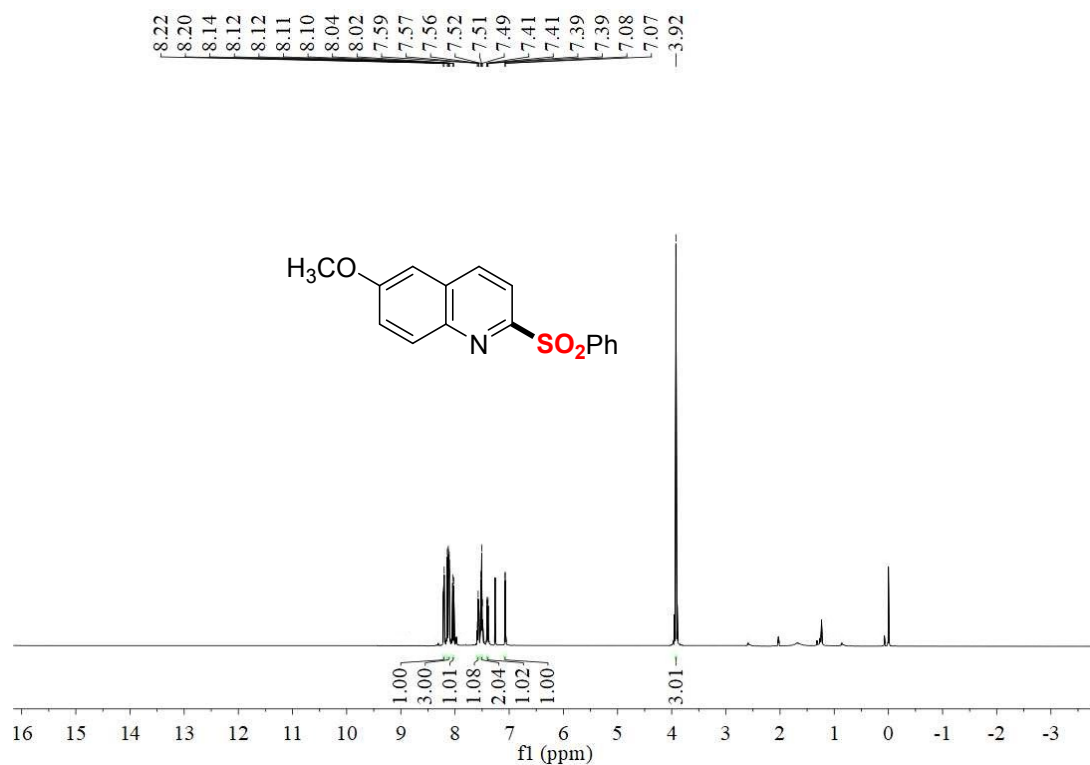
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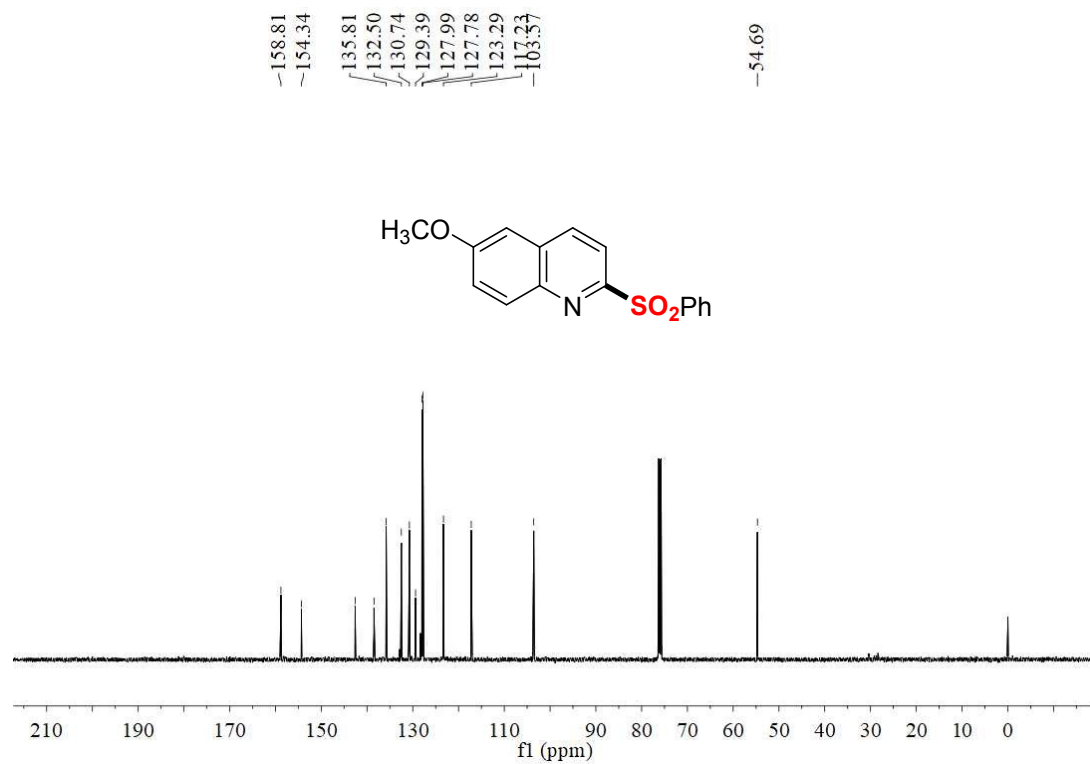
### 3w <sup>13</sup>C NMR



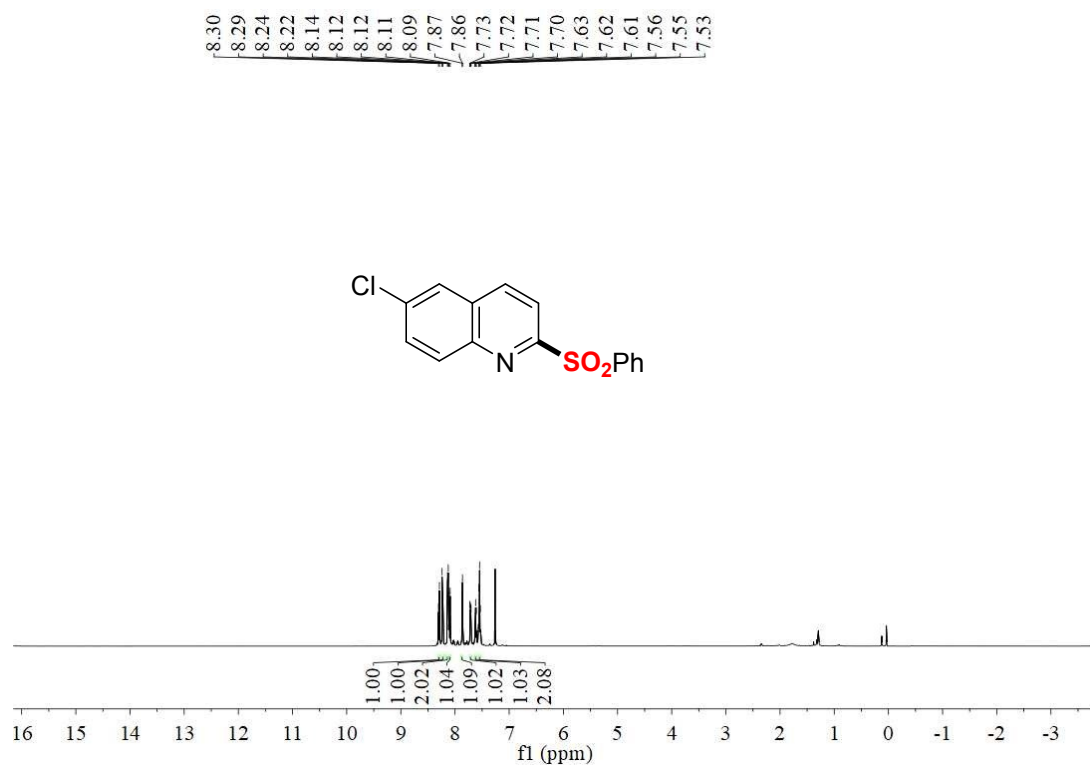
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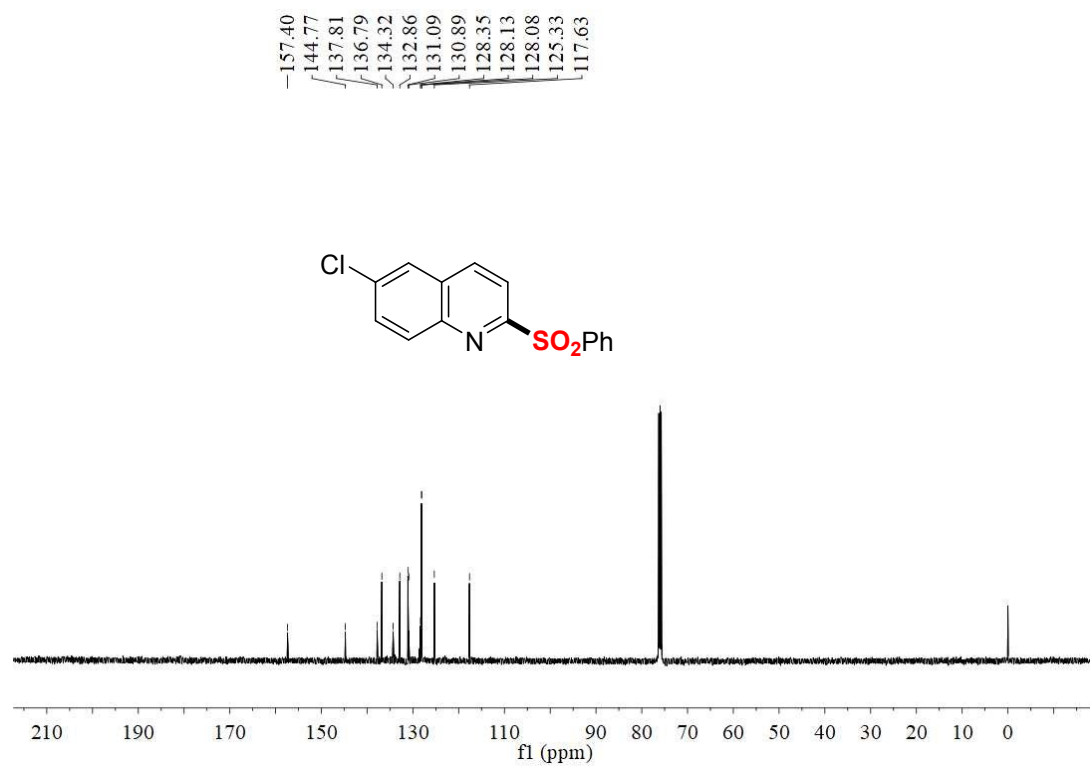
### 3x <sup>13</sup>C NMR



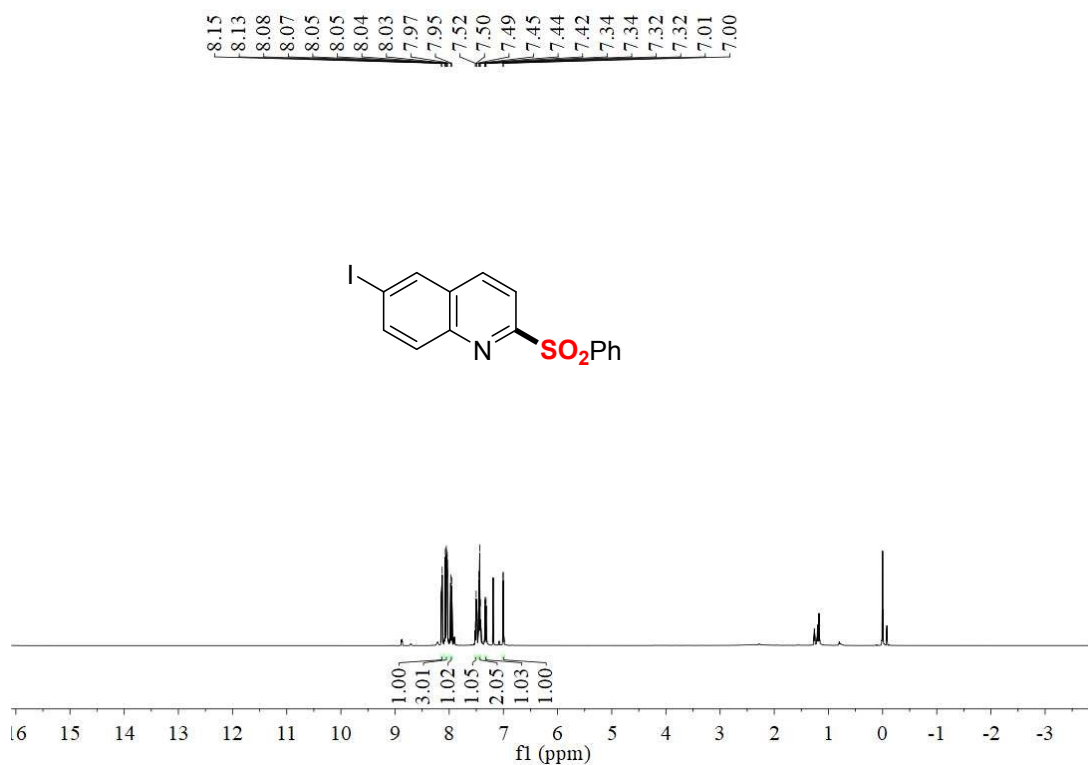
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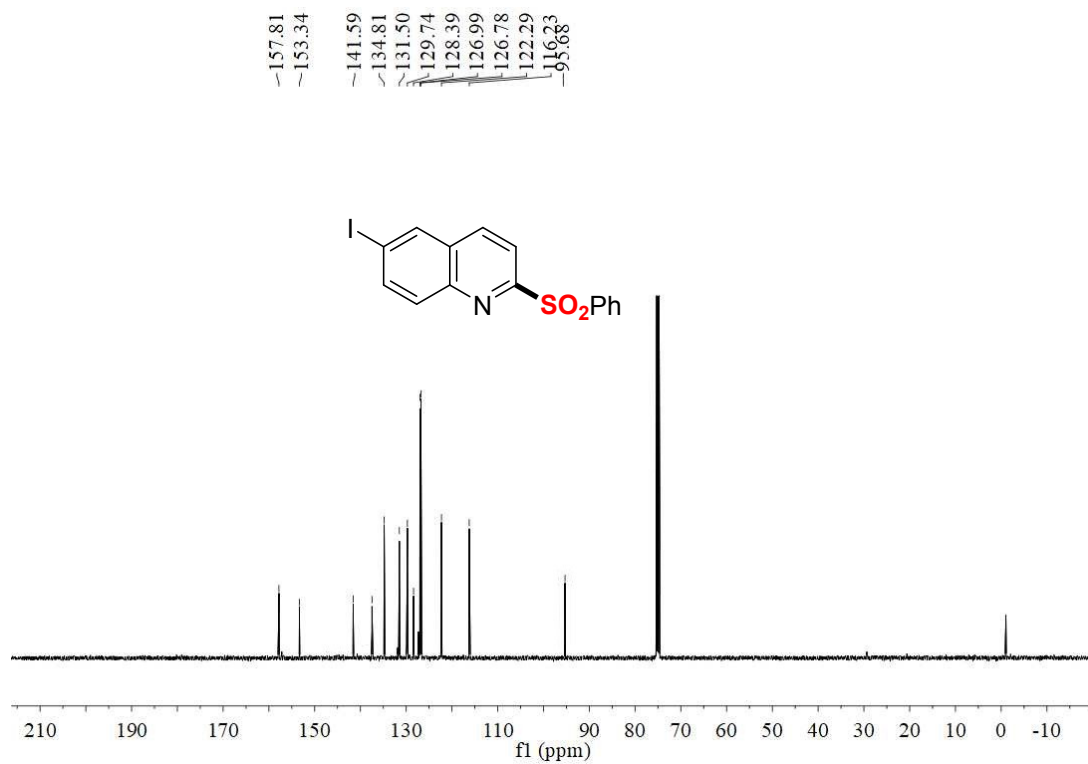
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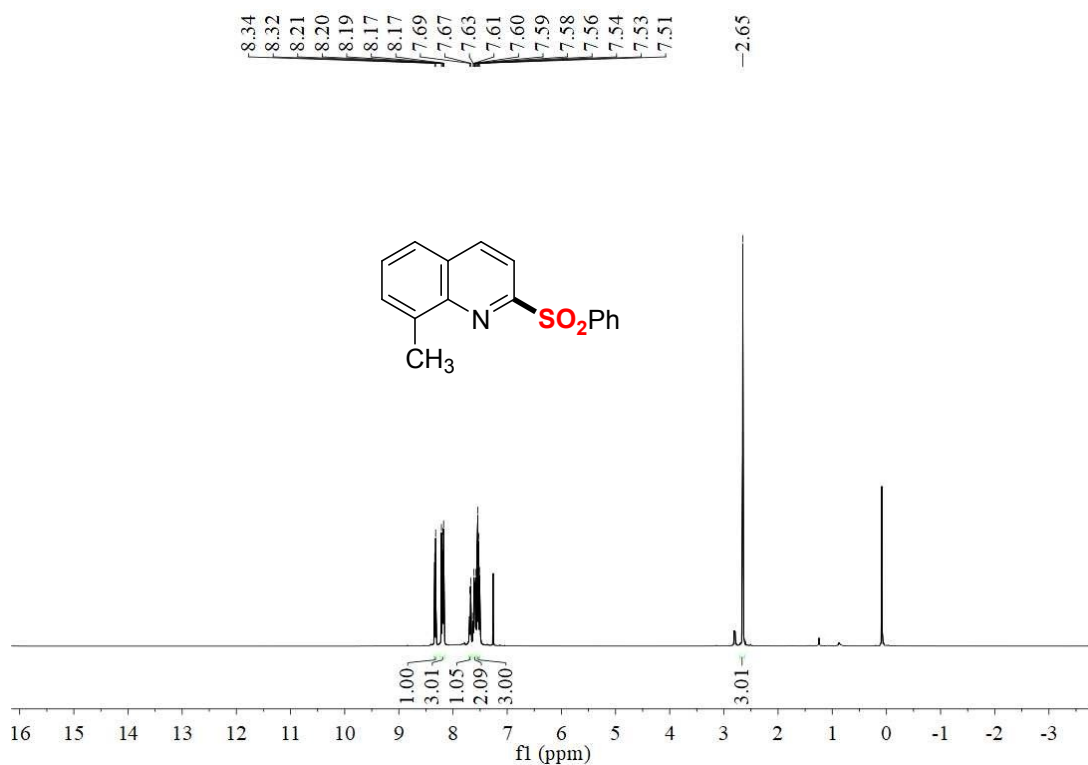
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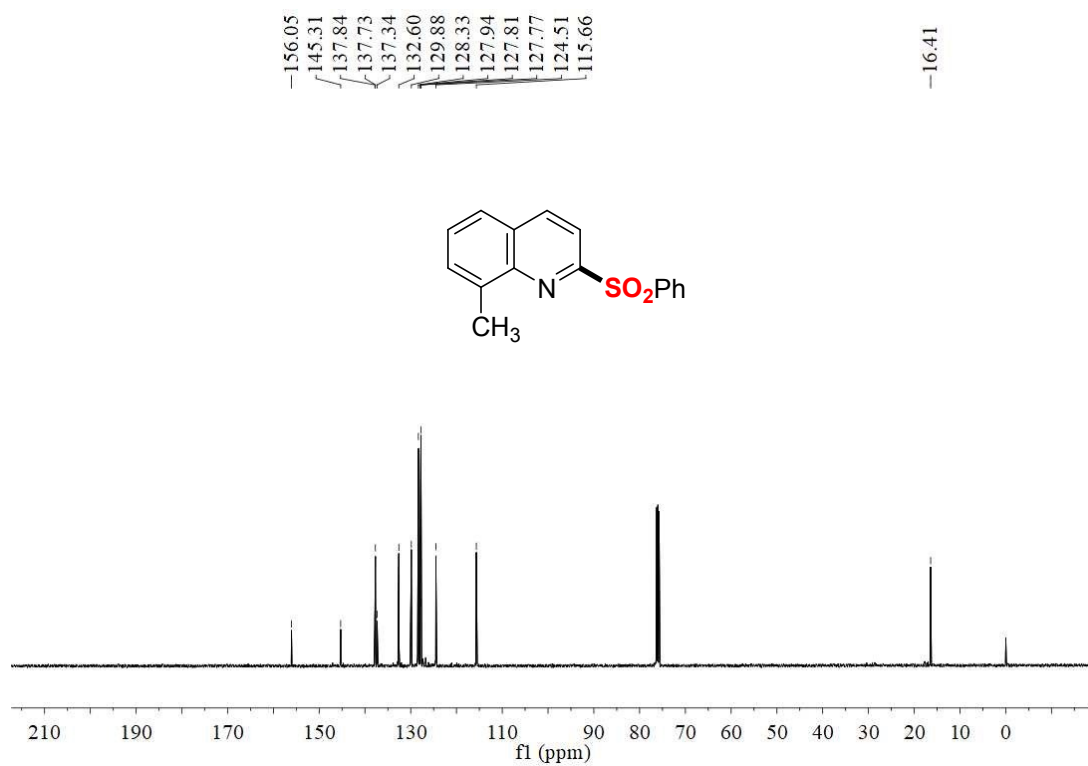
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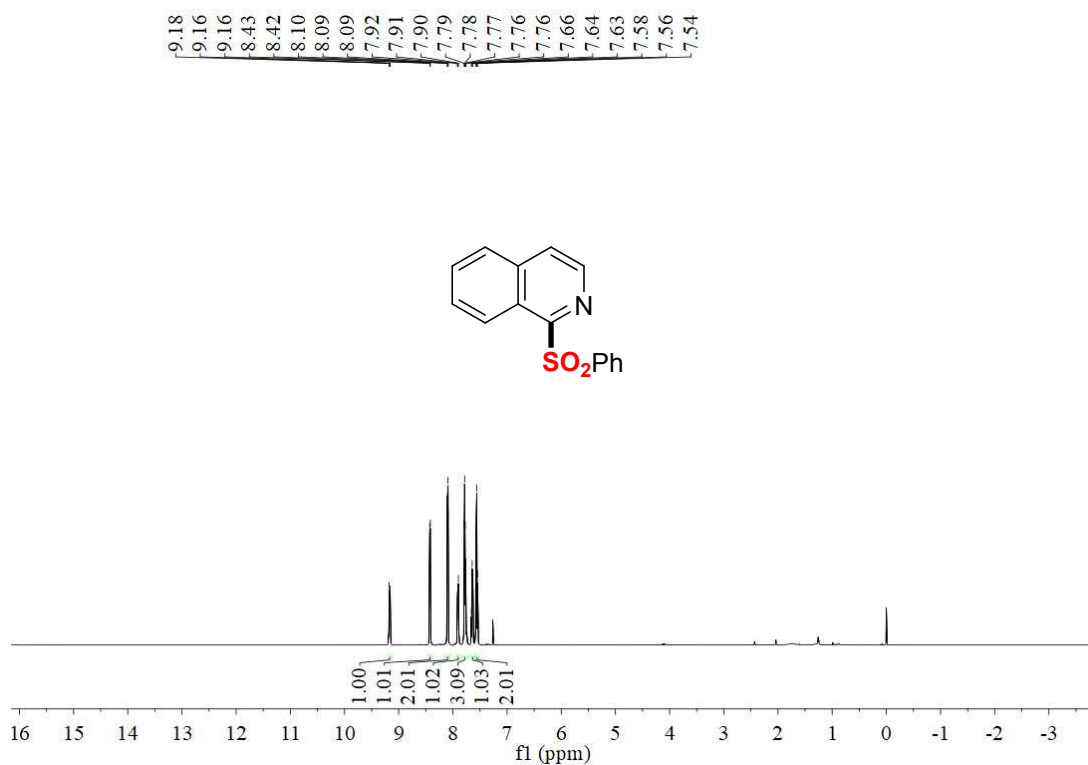
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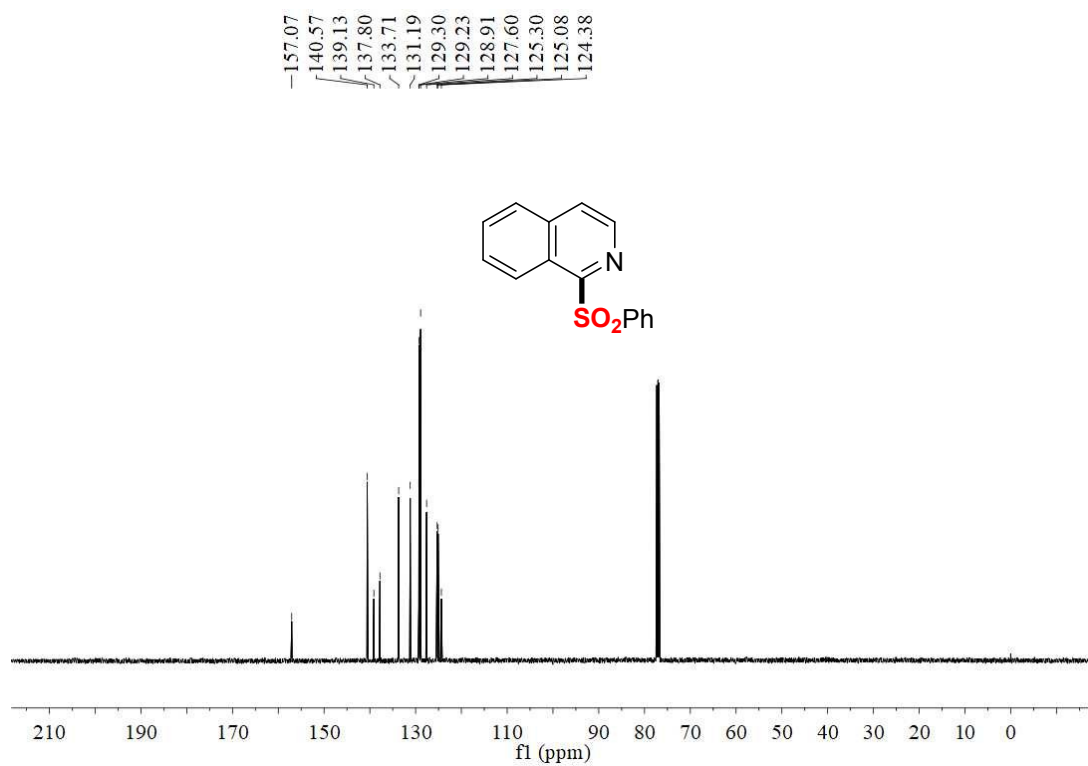
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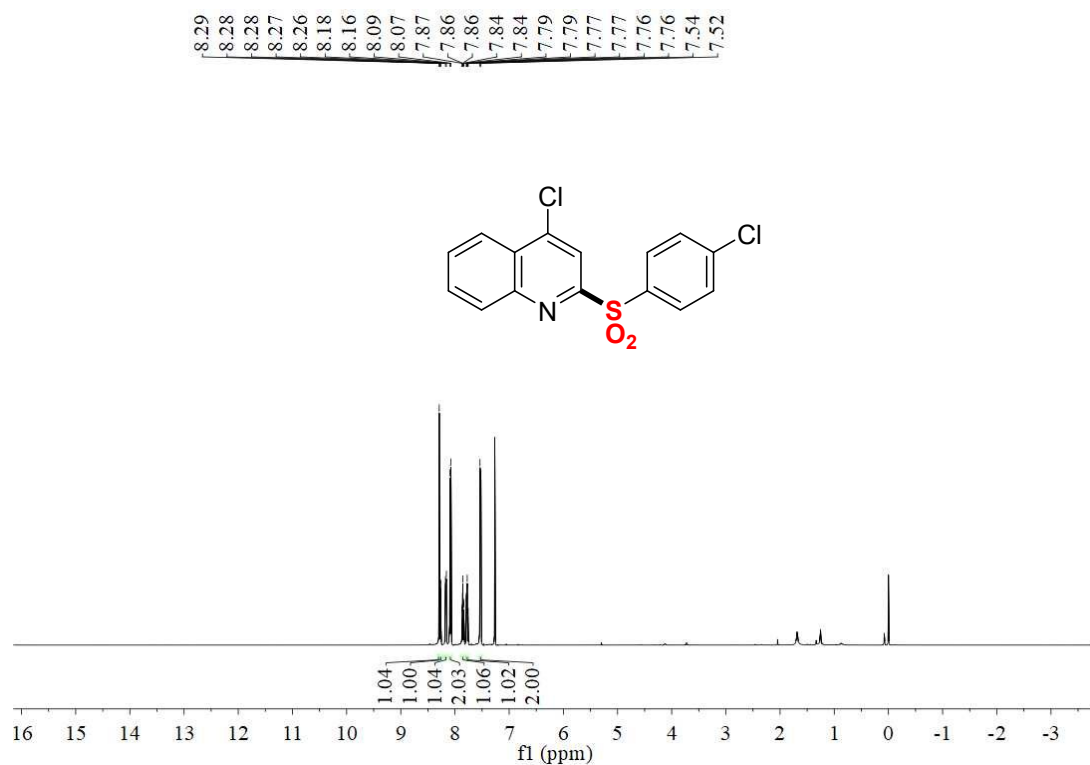
### 3ab <sup>1</sup>H NMR



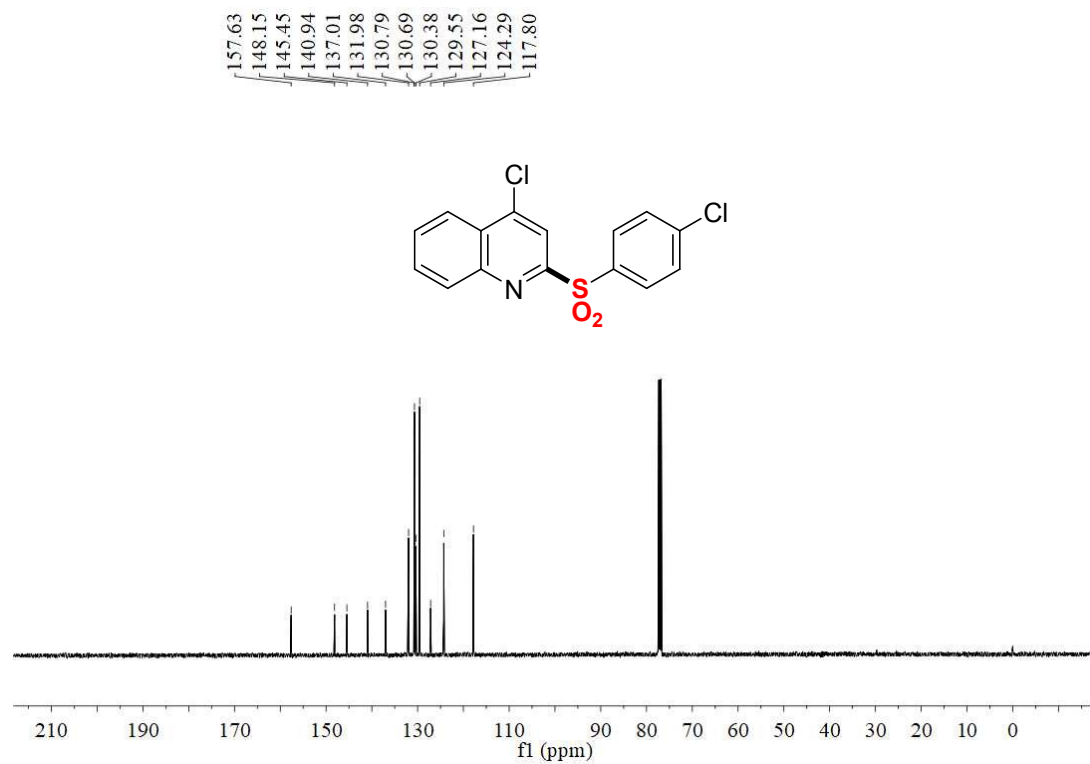
### 3ab <sup>13</sup>C NMR



### 3ac <sup>1</sup>H NMR

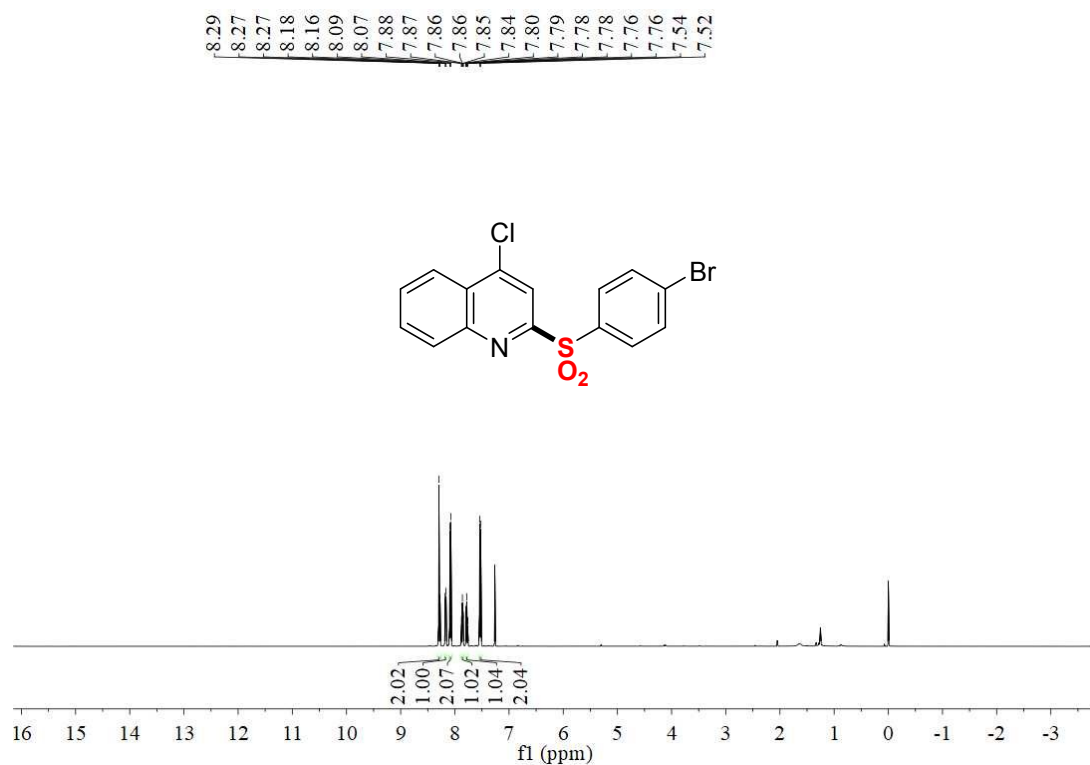


### 3ac <sup>13</sup>C NMR

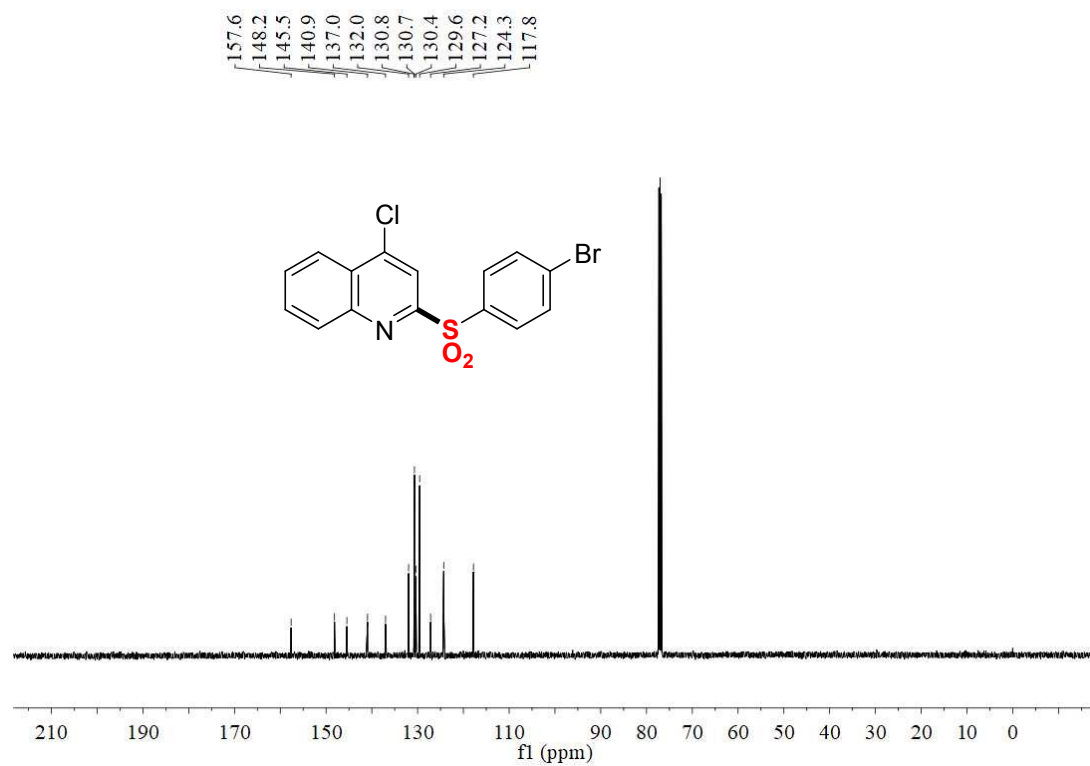




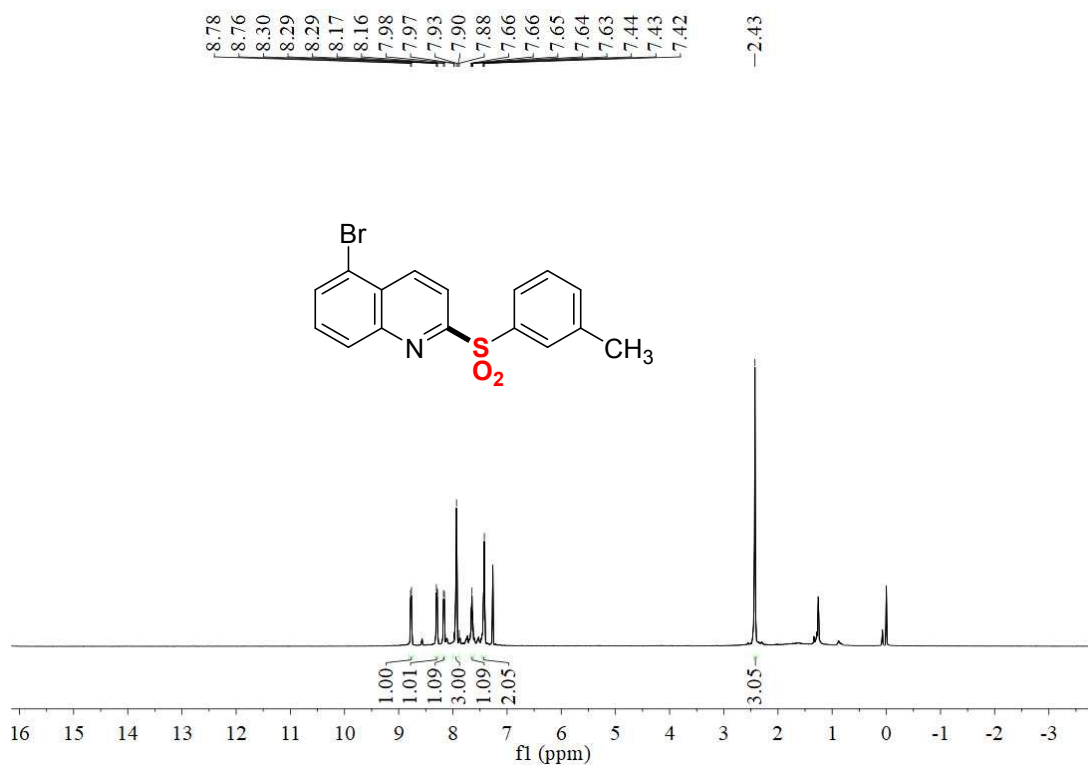
### 3ad <sup>1</sup>H NMR



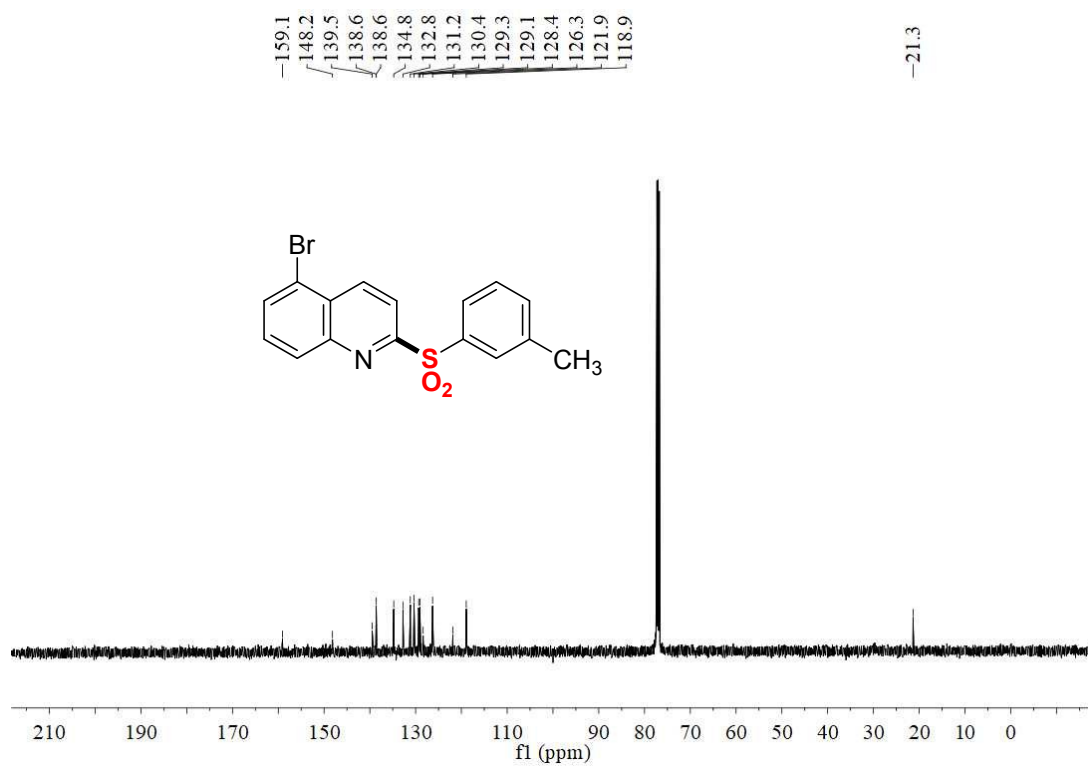
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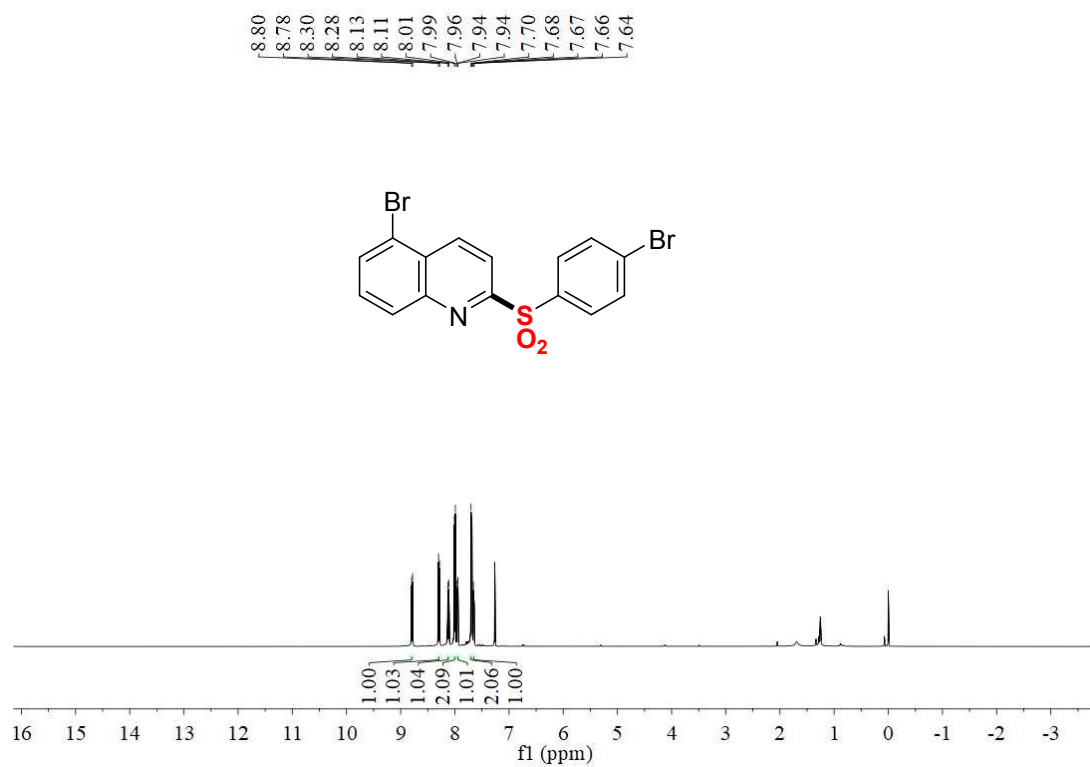
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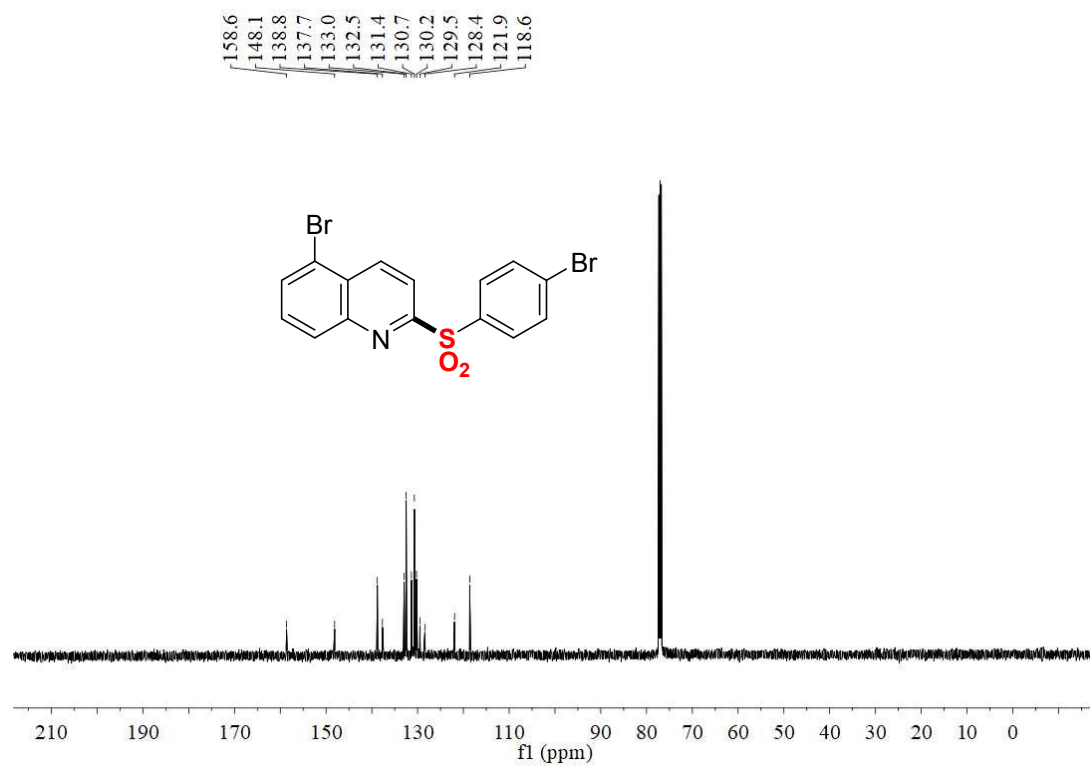
### 3ae <sup>13</sup>C NMR



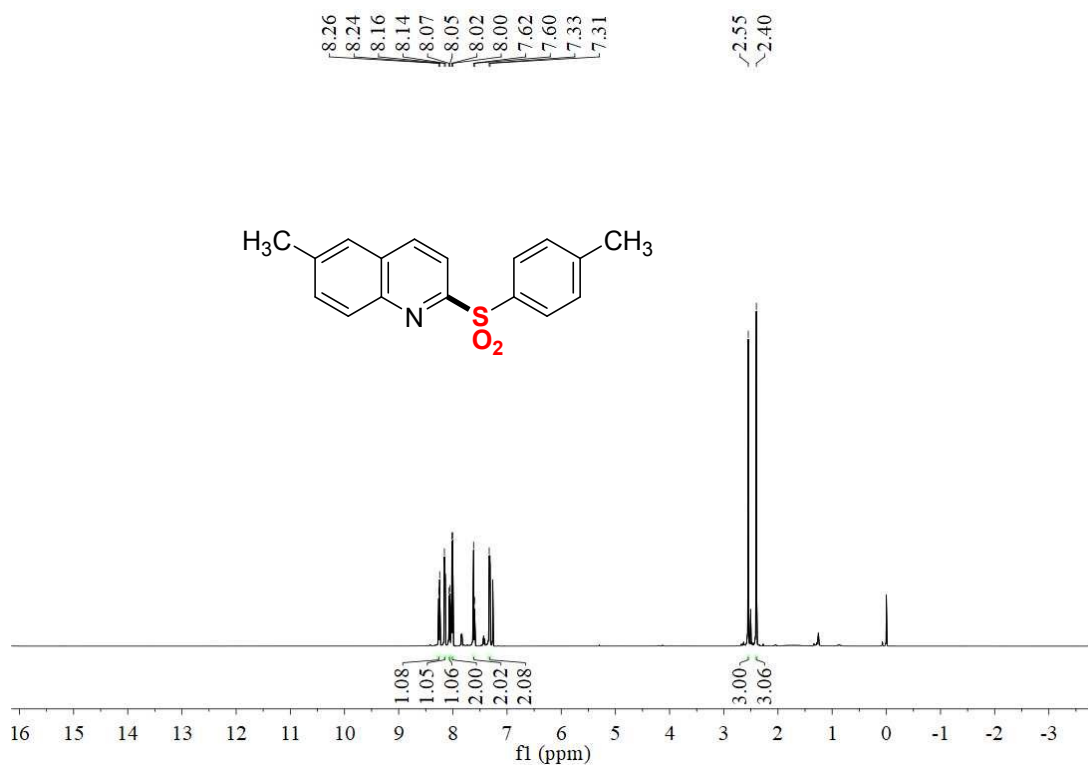
### 3af <sup>1</sup>H NMR



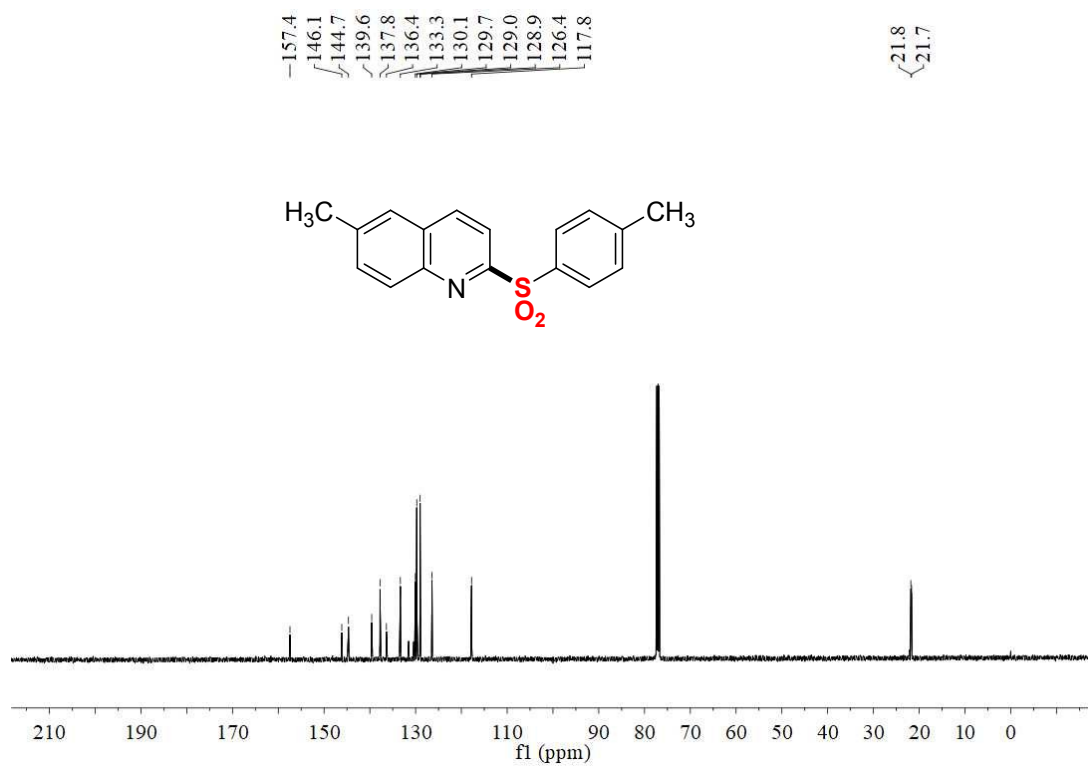
### 3af <sup>13</sup>C NMR



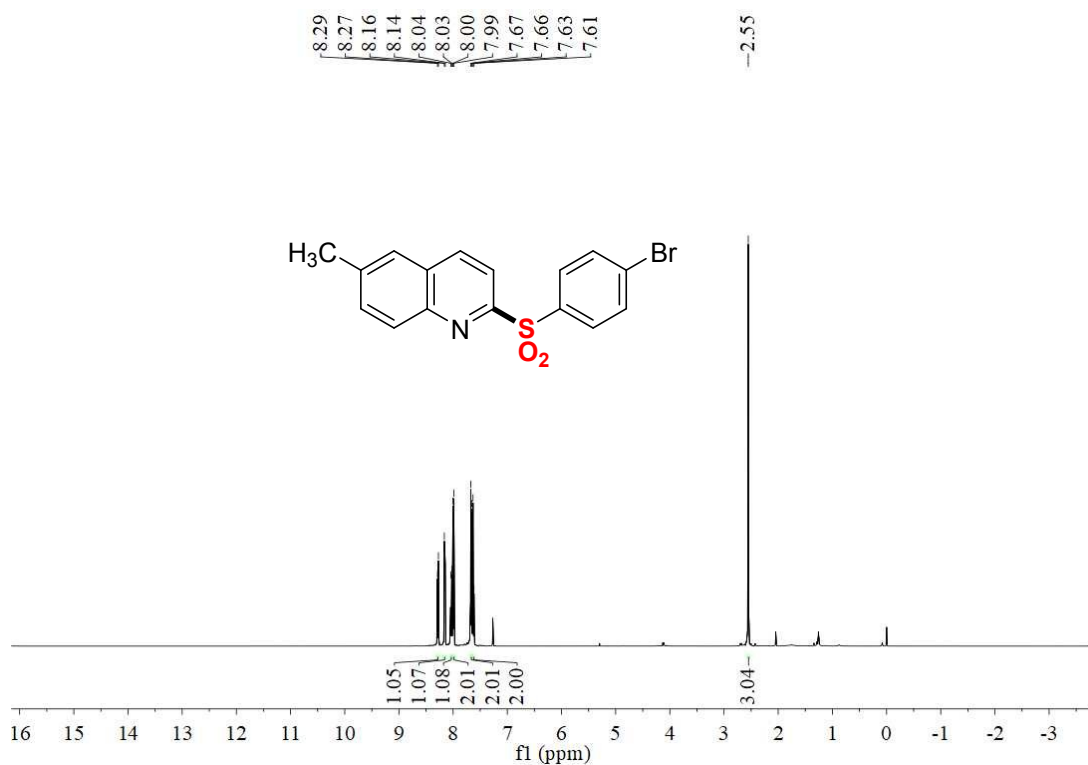
### 3ag <sup>1</sup>H NMR



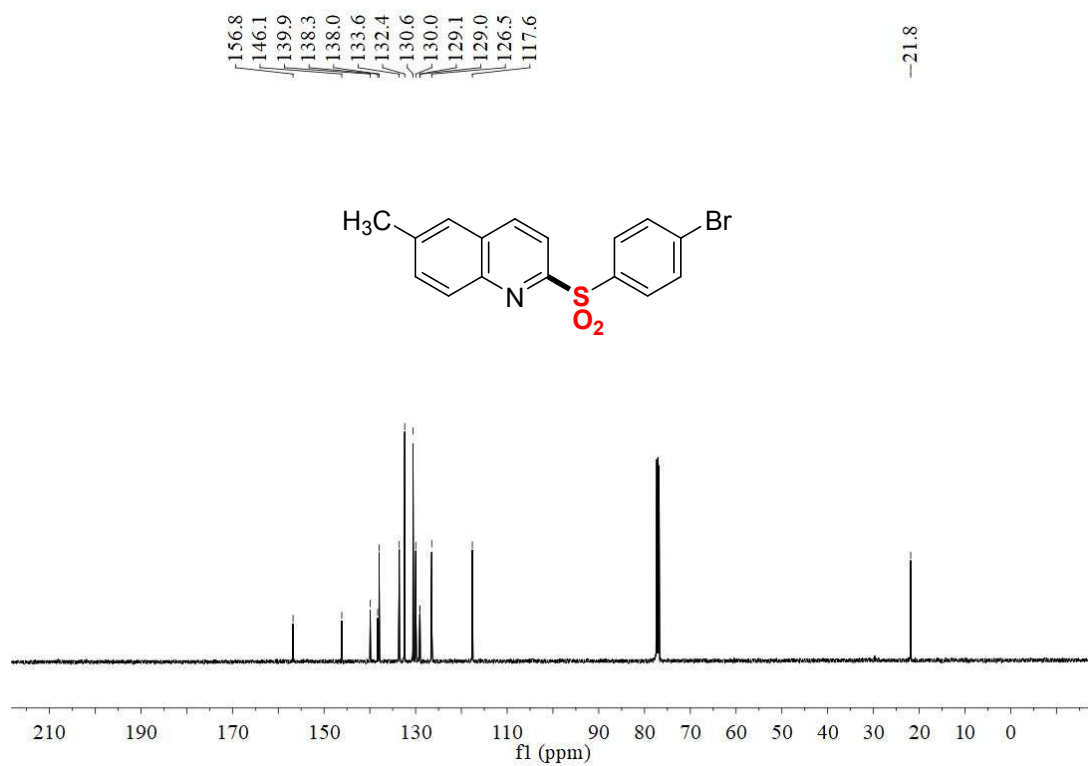
### 3ag <sup>13</sup>C NMR



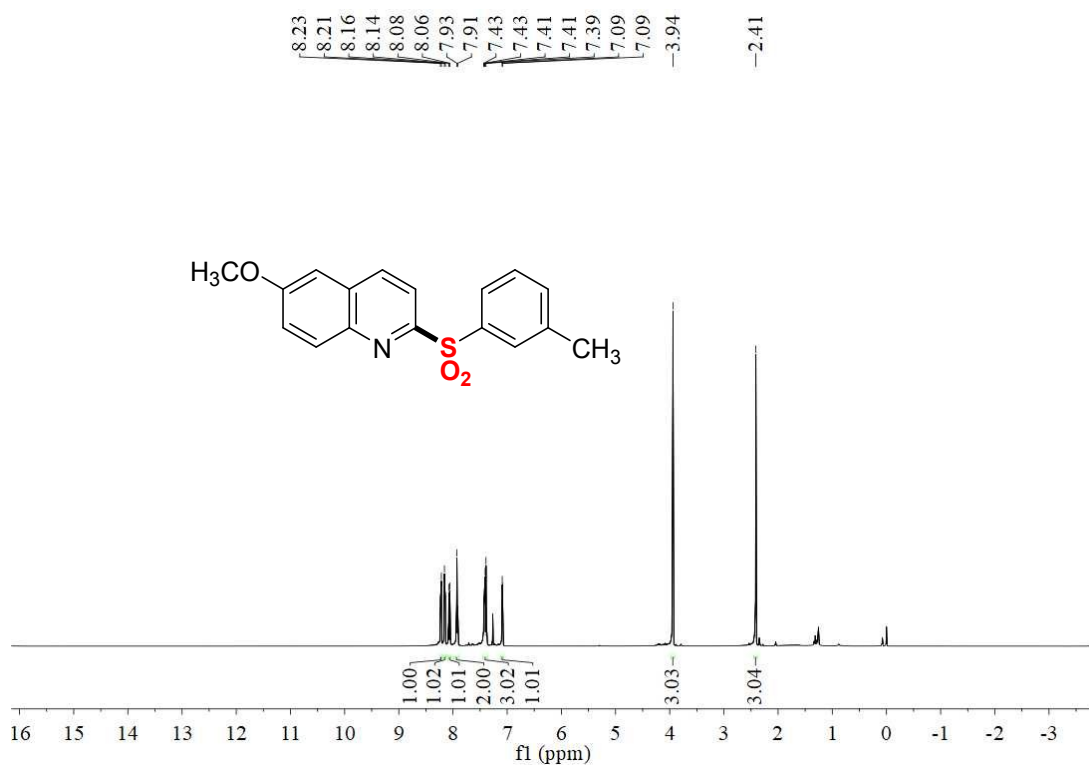
### 3ah <sup>1</sup>H NMR



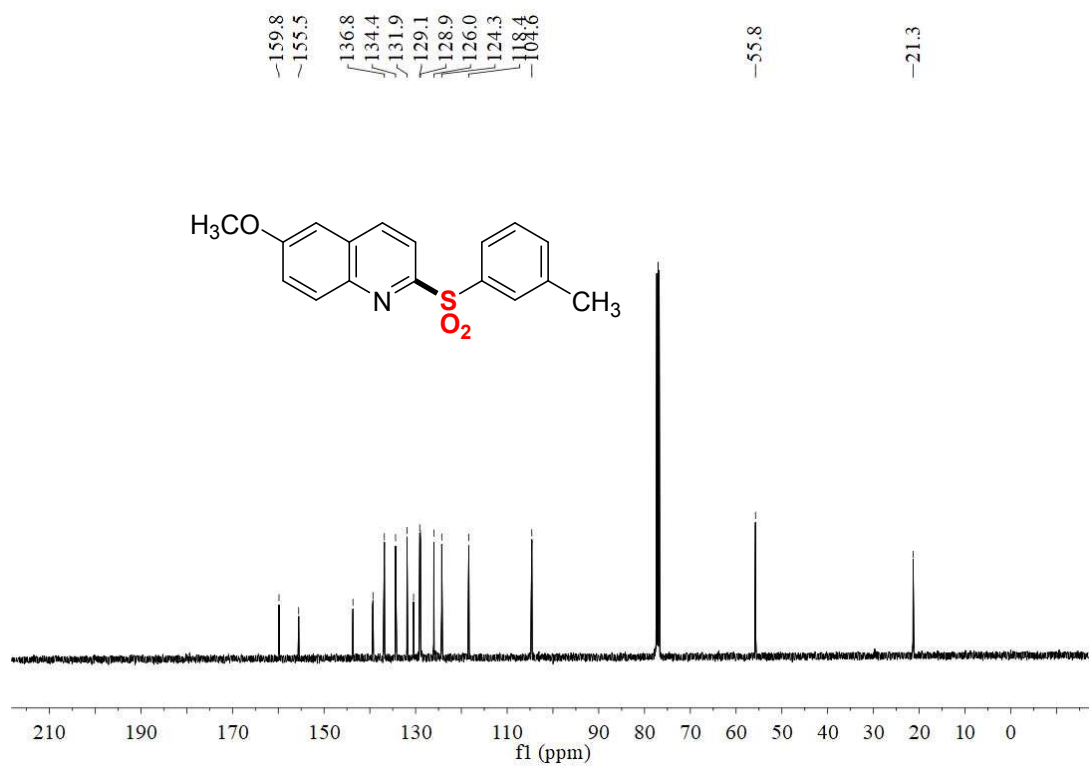
### 3ah <sup>13</sup>C NMR



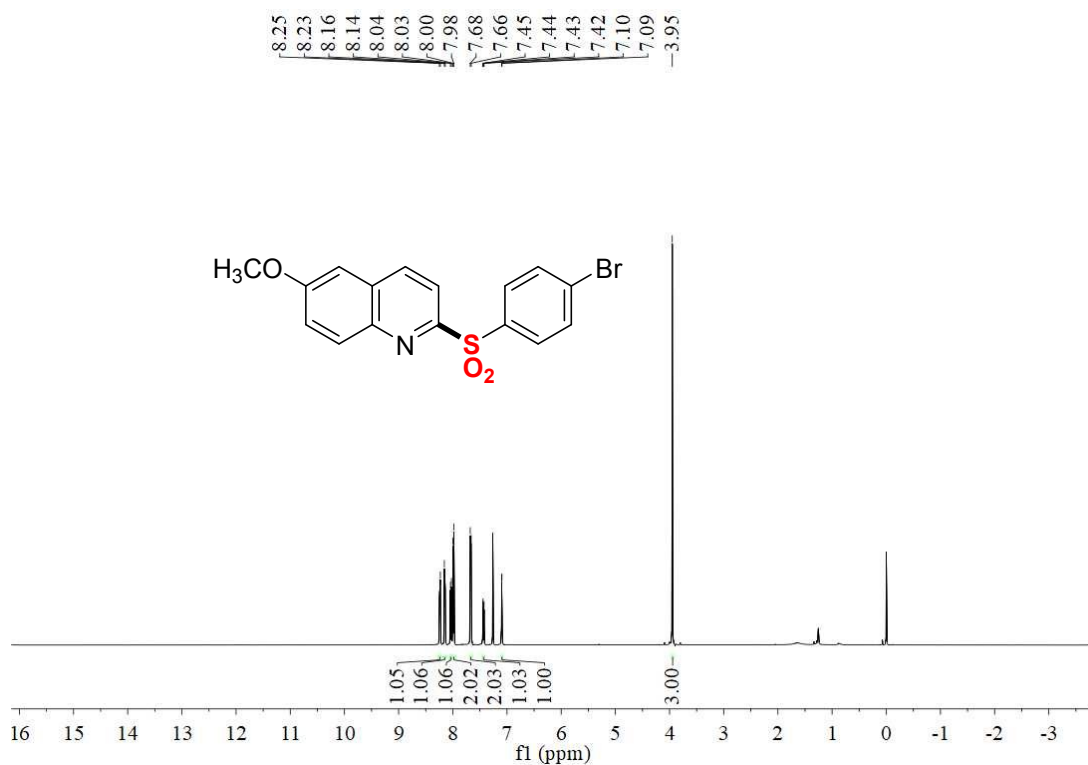
### 3ai <sup>1</sup>H NMR



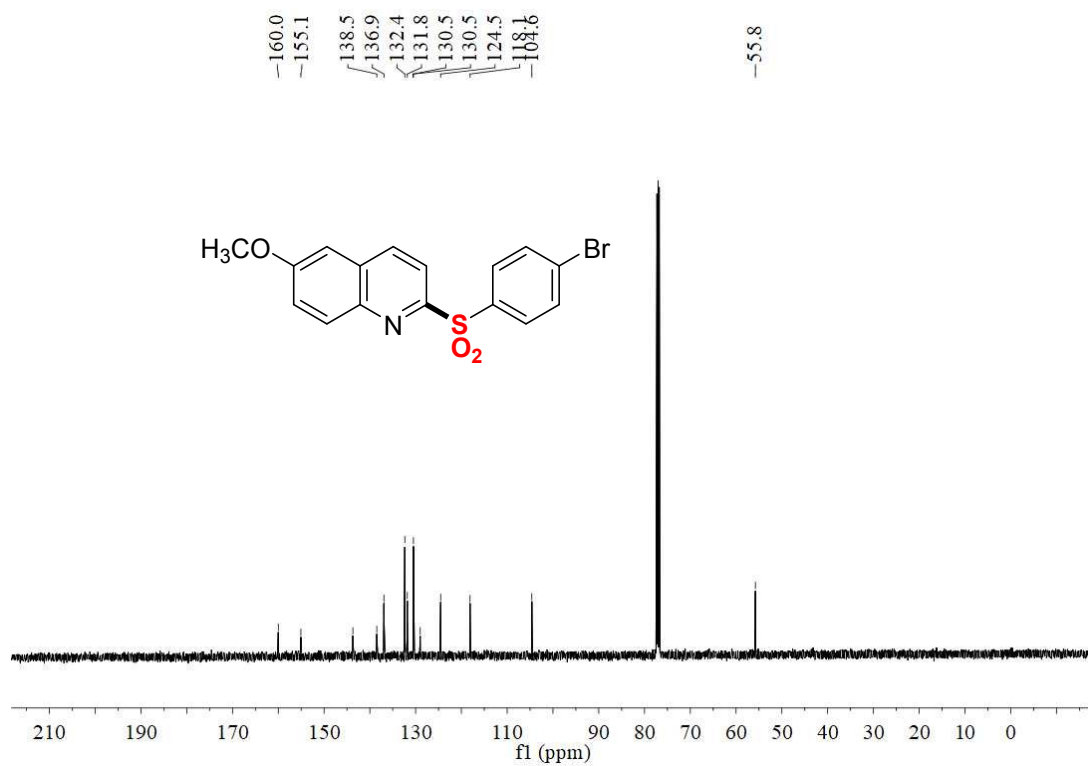
### 3ai <sup>13</sup>C NMR



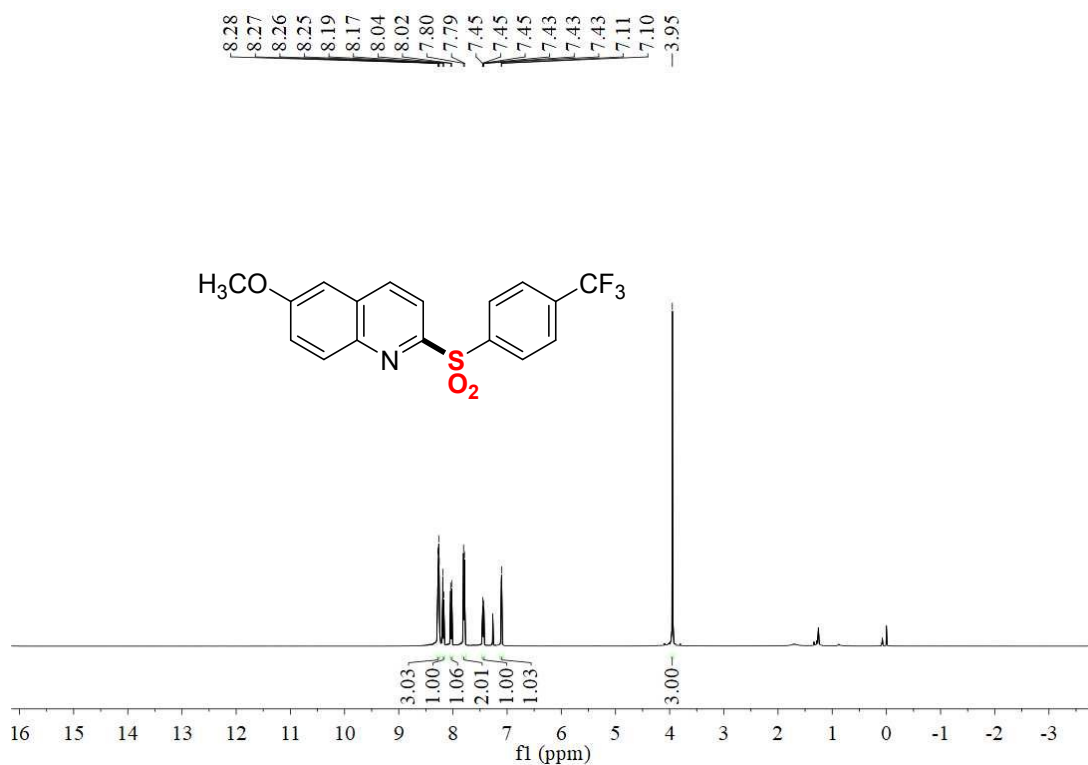
### 3aj <sup>1</sup>H NMR



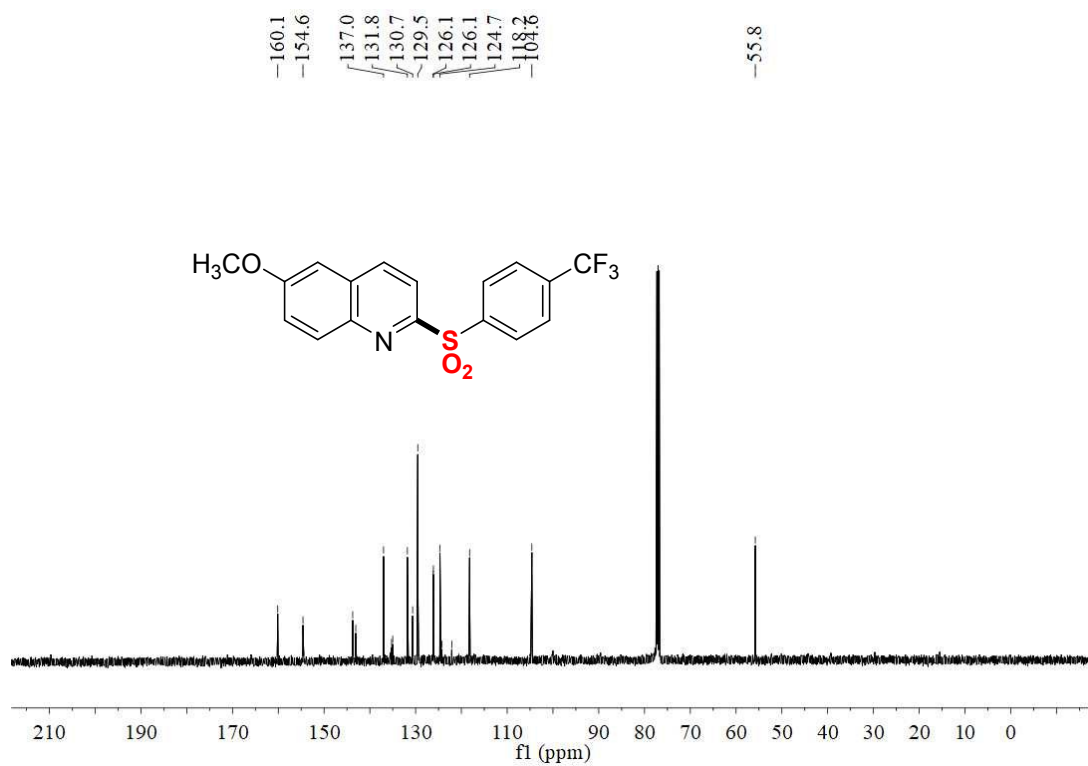
### 3aj <sup>13</sup>C NMR



### 3ak <sup>1</sup>H NMR

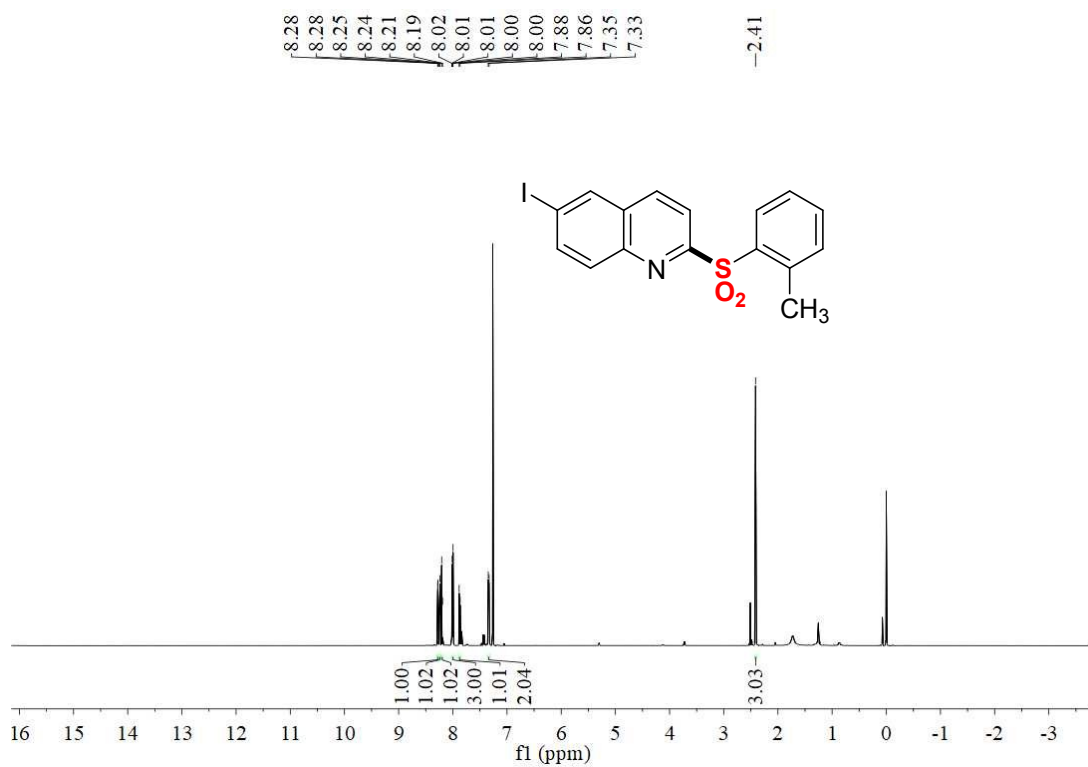


### 3ak <sup>13</sup>C NMR

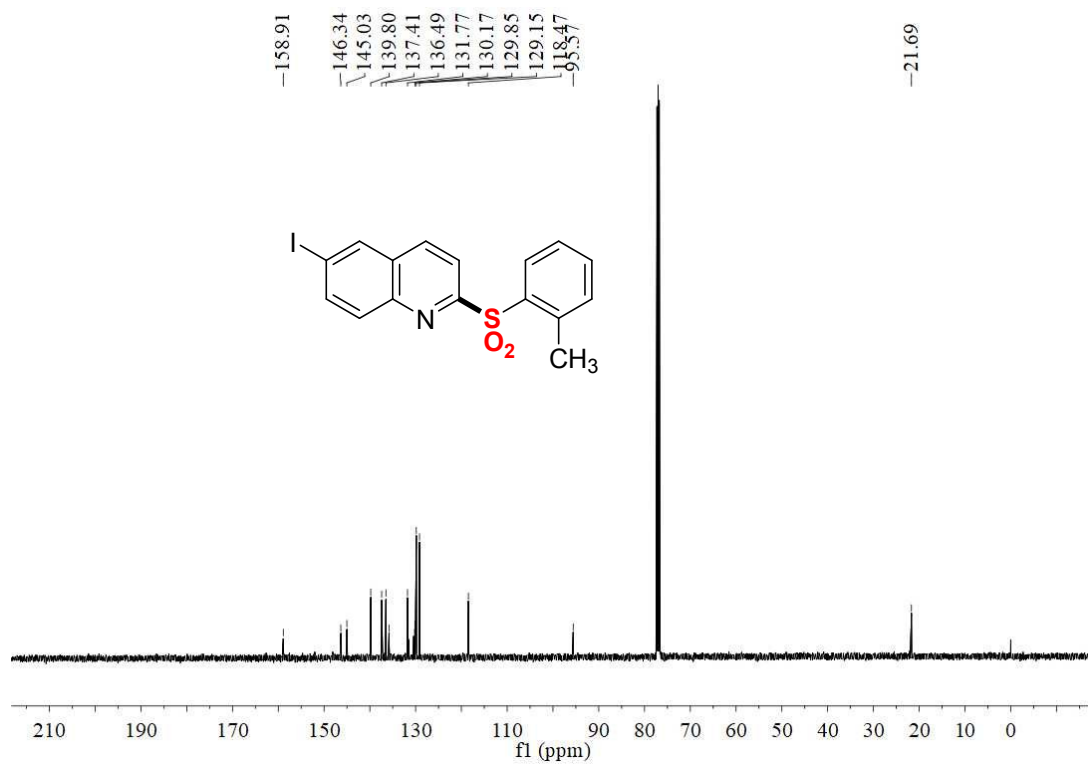




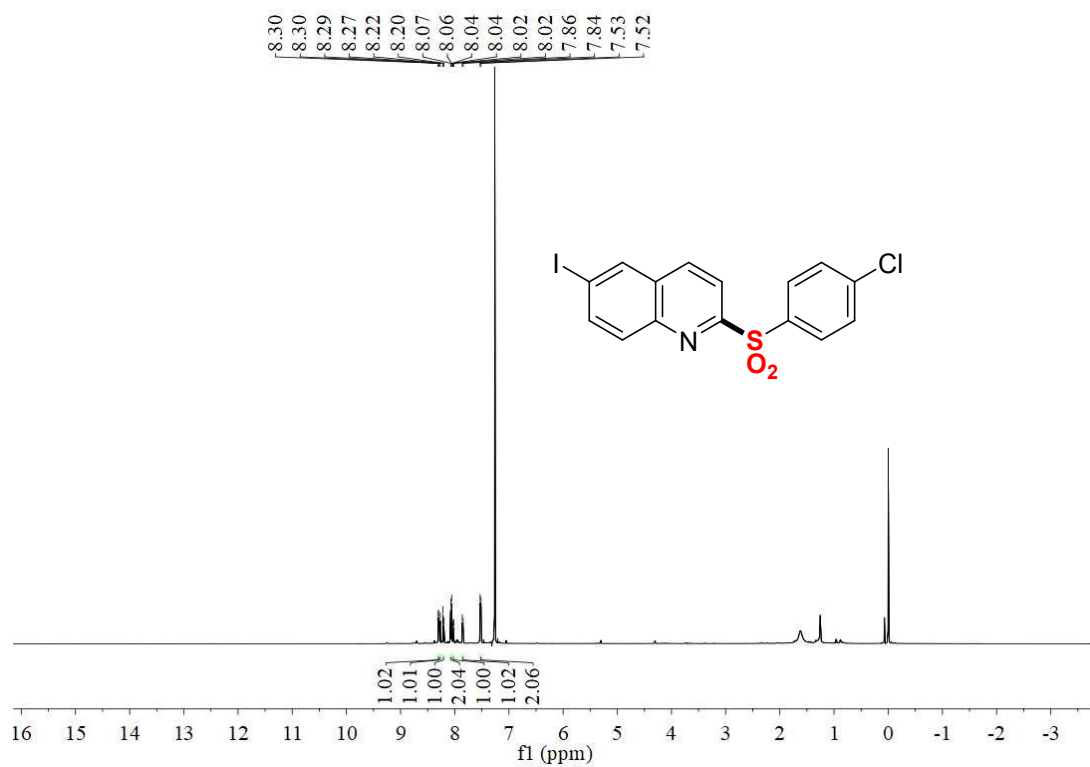
### 3al <sup>1</sup>H NMR



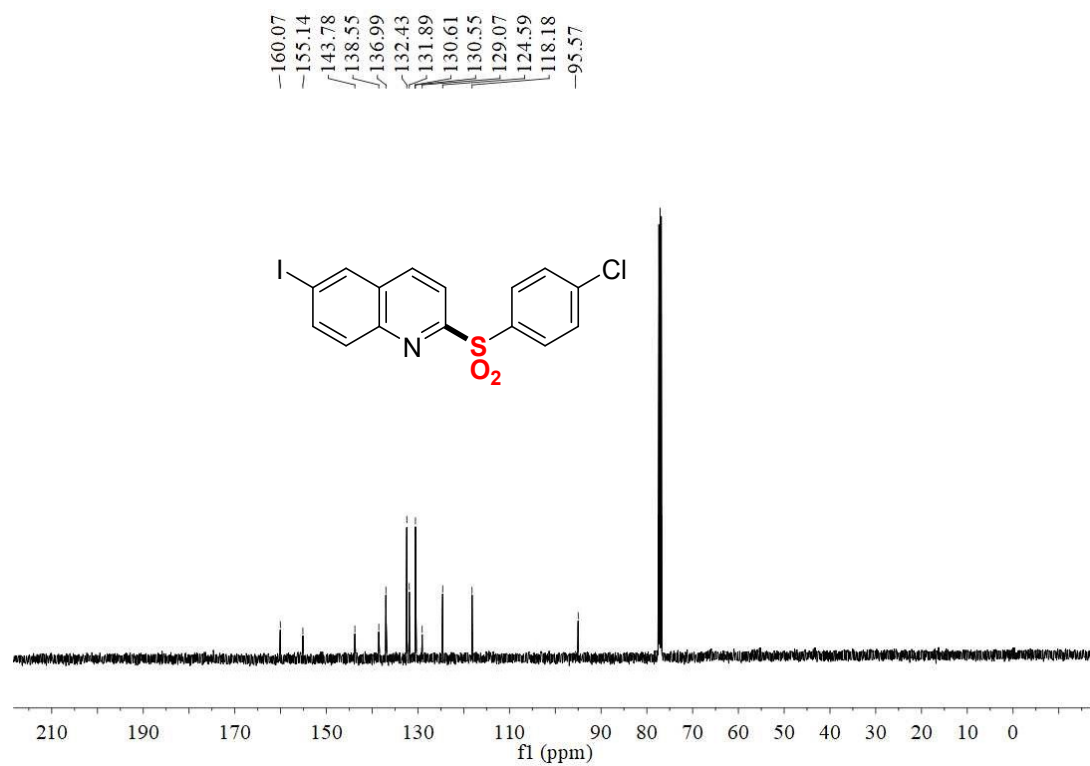
### 3al <sup>13</sup>C NMR



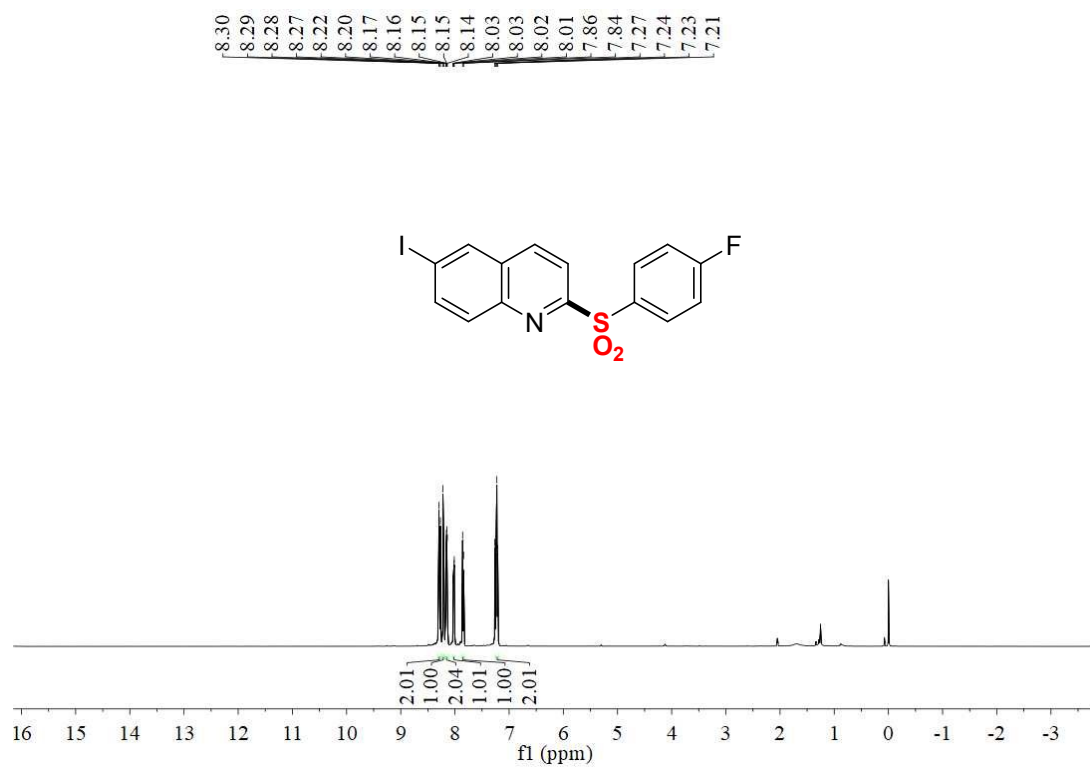
### 3am <sup>1</sup>H NMR



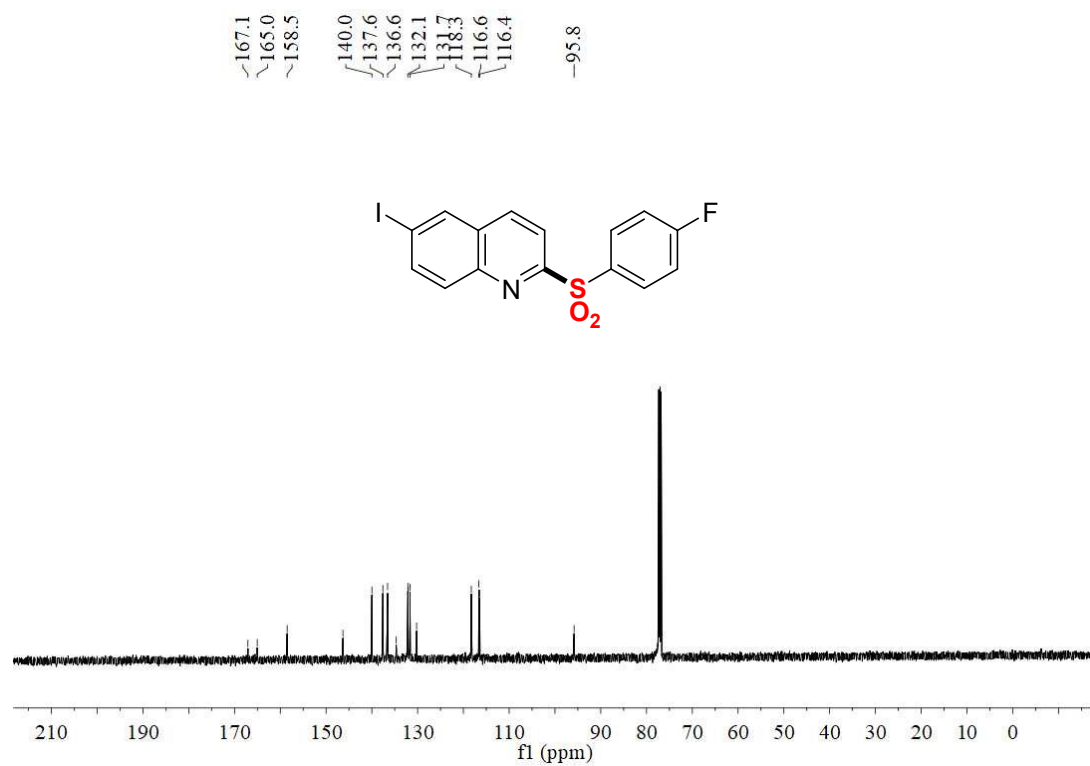
### 3am <sup>13</sup>C NMR



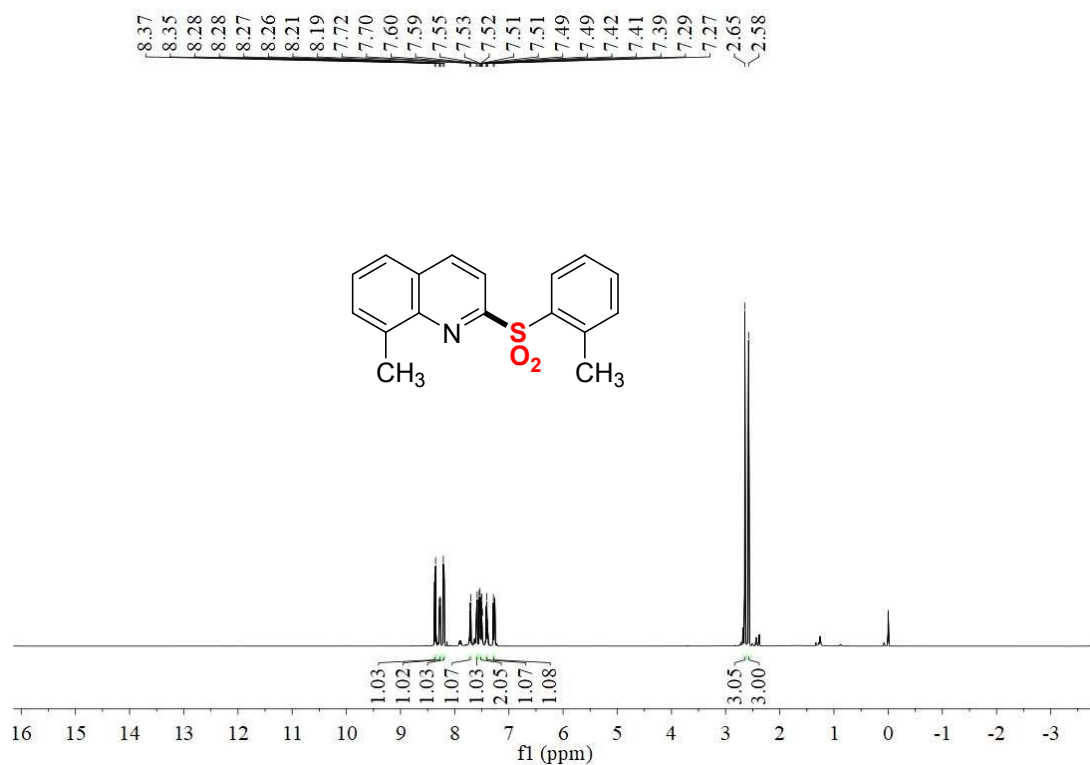
### 3an <sup>1</sup>H NMR



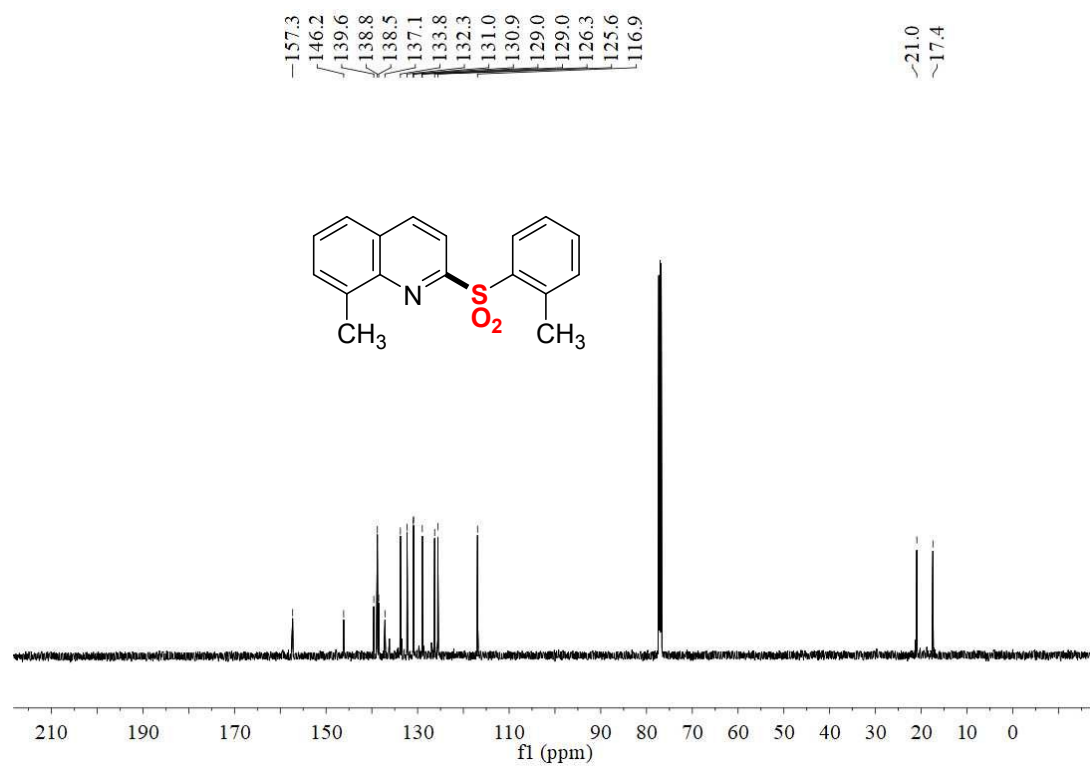
### 3an <sup>13</sup>C NMR



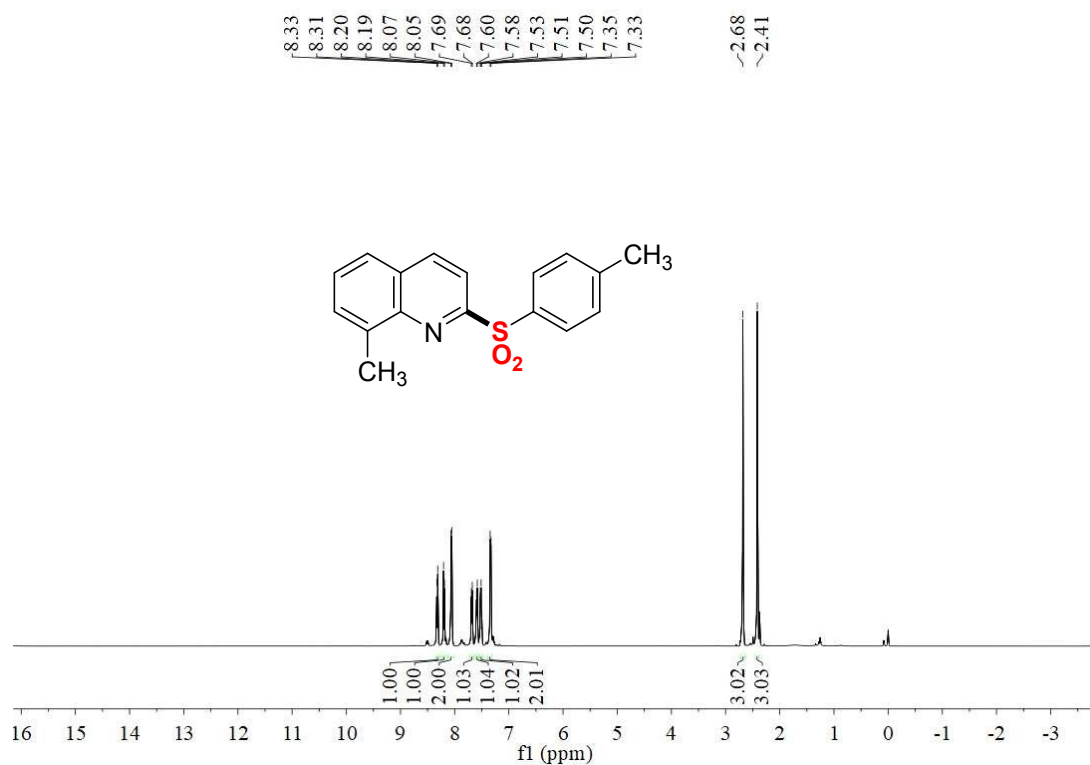
### 3ao <sup>1</sup>H NMR



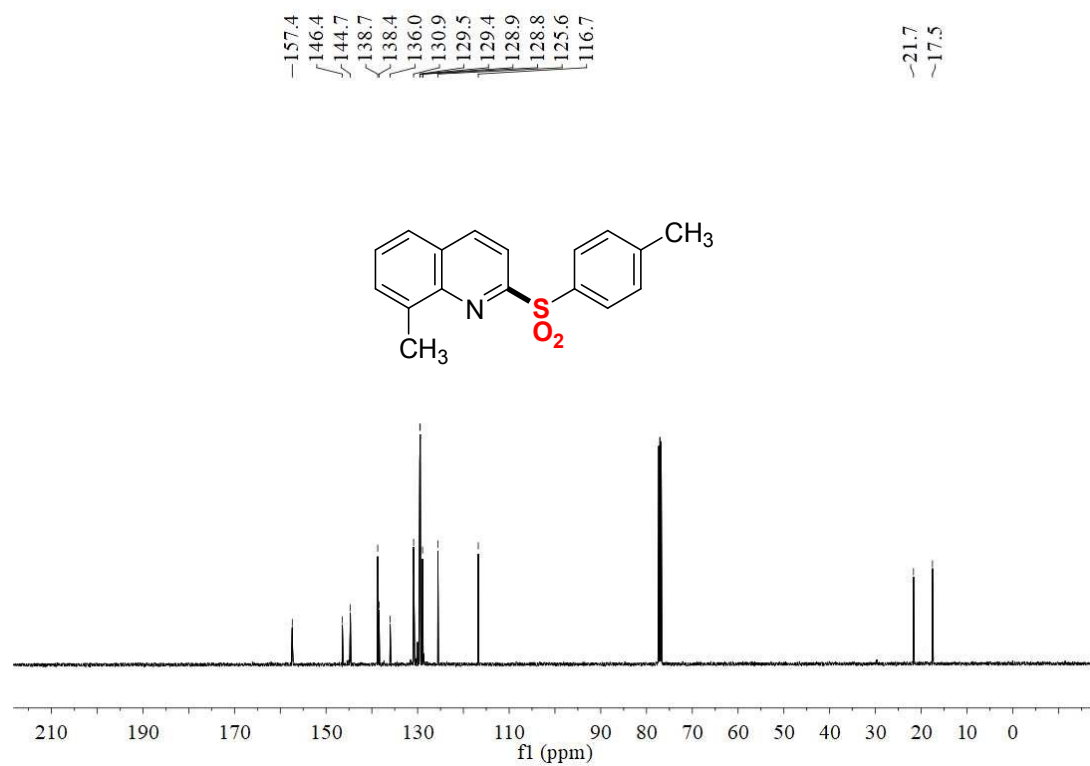
### 3ao <sup>13</sup>C NMR



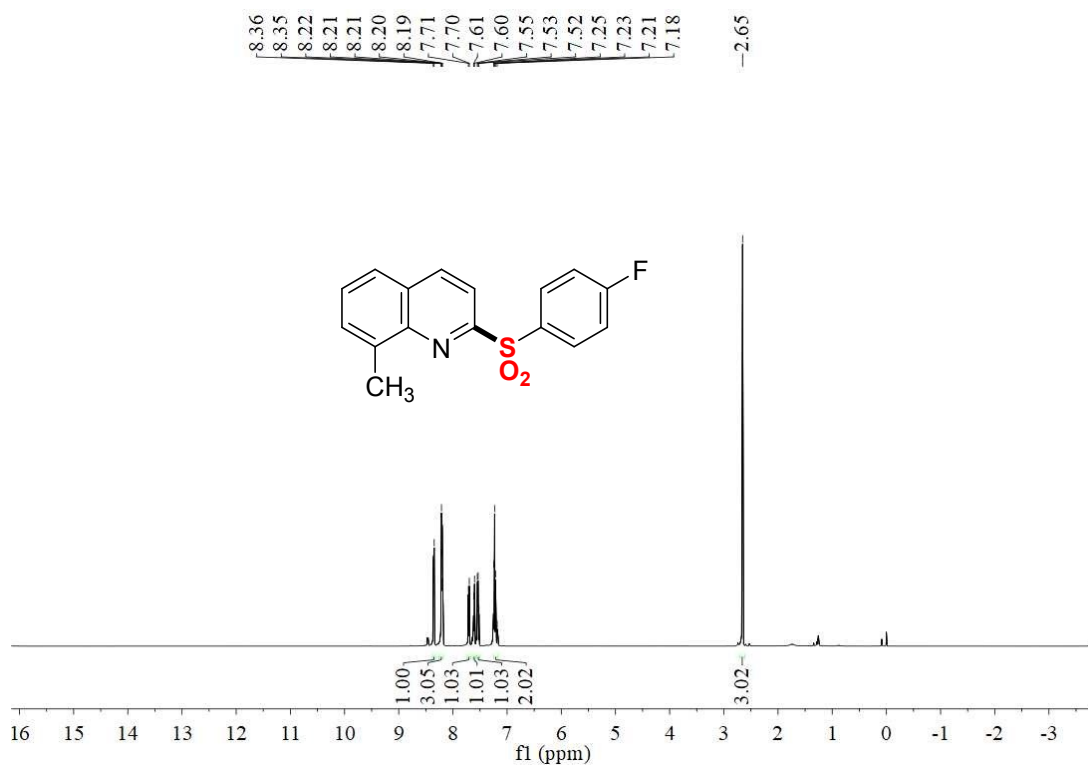
### 3ap <sup>1</sup>H NMR



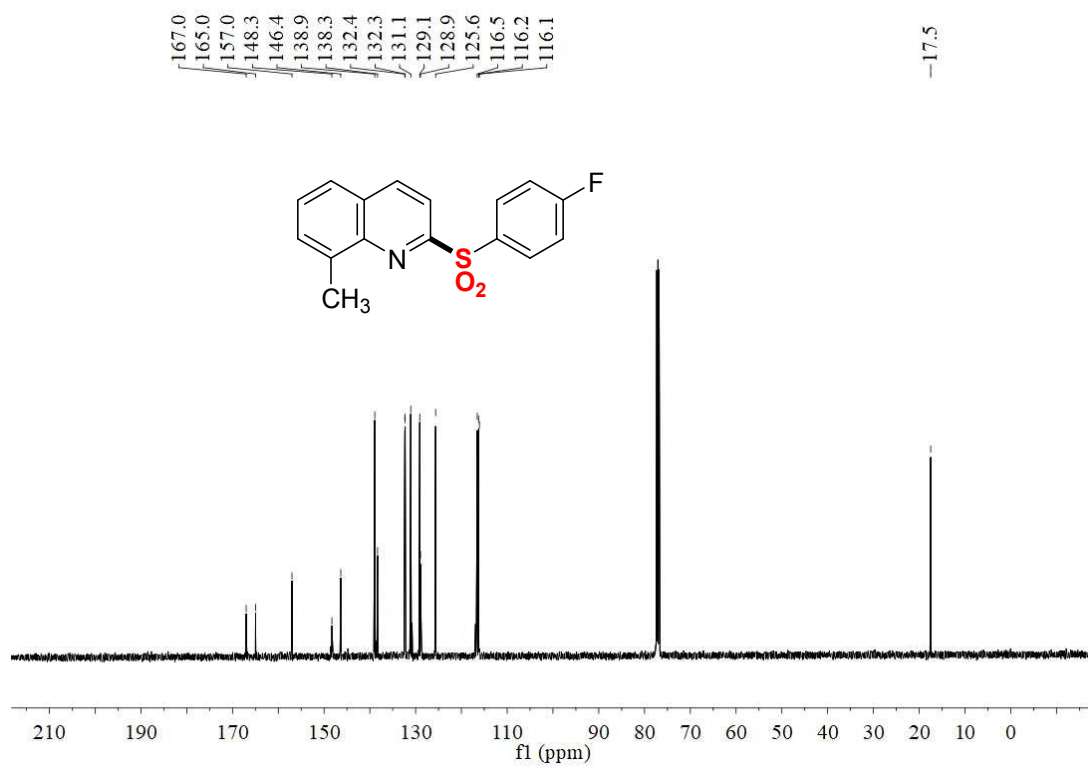
### 3ap <sup>13</sup>C NMR



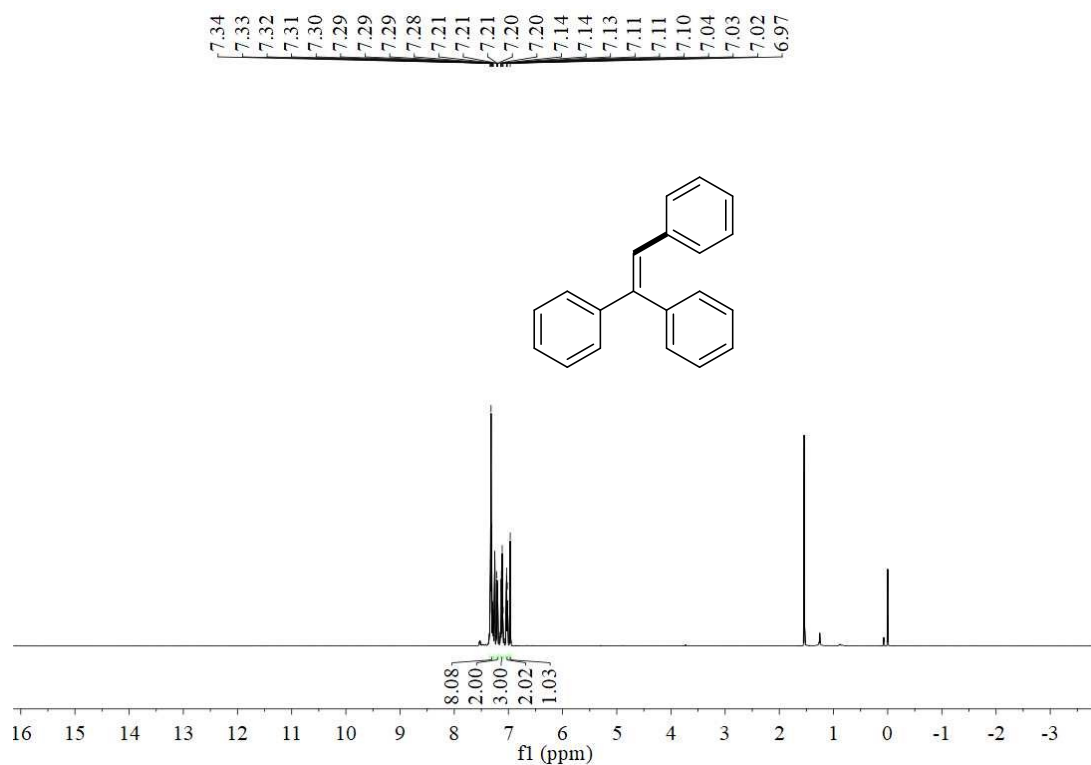
### 3aq <sup>1</sup>H NMR



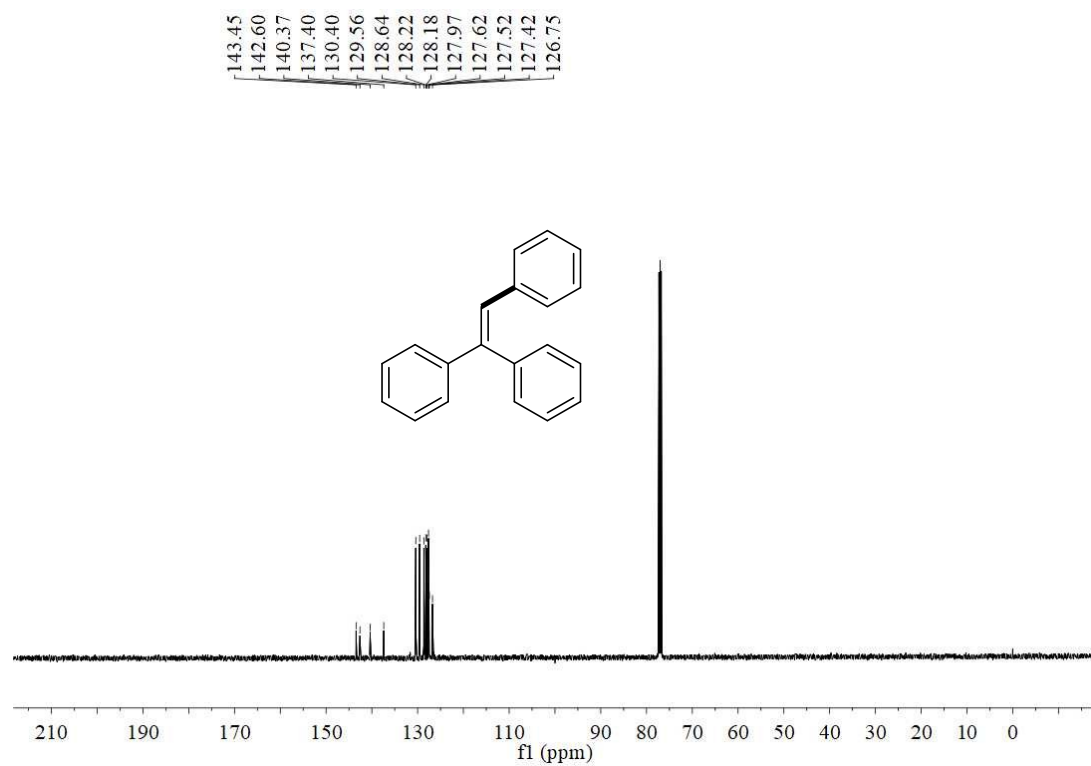
### 3aq <sup>13</sup>C NMR



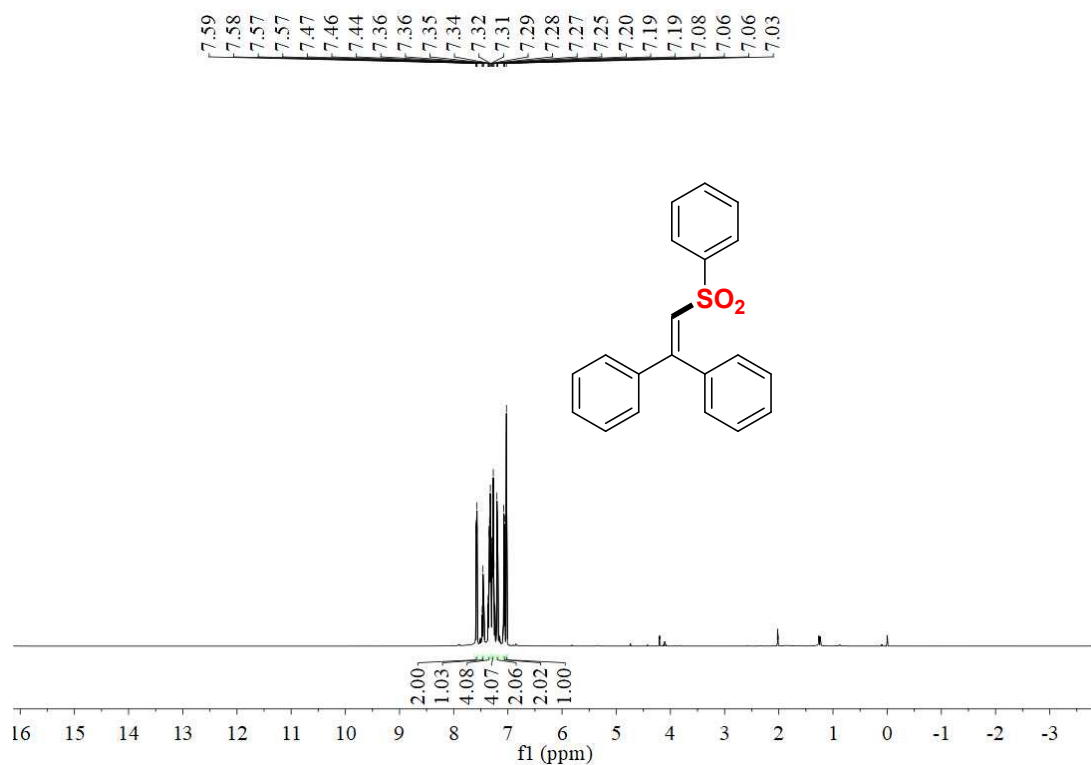
### 5 <sup>1</sup>H NMR



### 5 <sup>13</sup>C NMR



6 <sup>1</sup>H NMR



6 <sup>13</sup>C NMR

