Electronic Supplementary Information

Visible light-enabled aerobic oxidative C_{sp3}−H functionalization of glycine derivatives using an organic photocatalyst: access to substituted quinoline-2-carboxylates

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1. Optimization of Reaction Conditions

Description of the blue LEDs used in this reaction: The blue LED strip was purchased from supplier (BeiLiang, Type and Specification: QDD5050K60C, 11.5 W/m, wavelengths: 450-470nm). The blue LED strip (2 m) was coiled around a transparent glass culture vessel (diameter: 14 cm), and the reaction flask was placed into the center of the culture vessel. The blue LED bulb was purchased from supplier (QianFang, 3 W, wavelengths: 450-470nm).

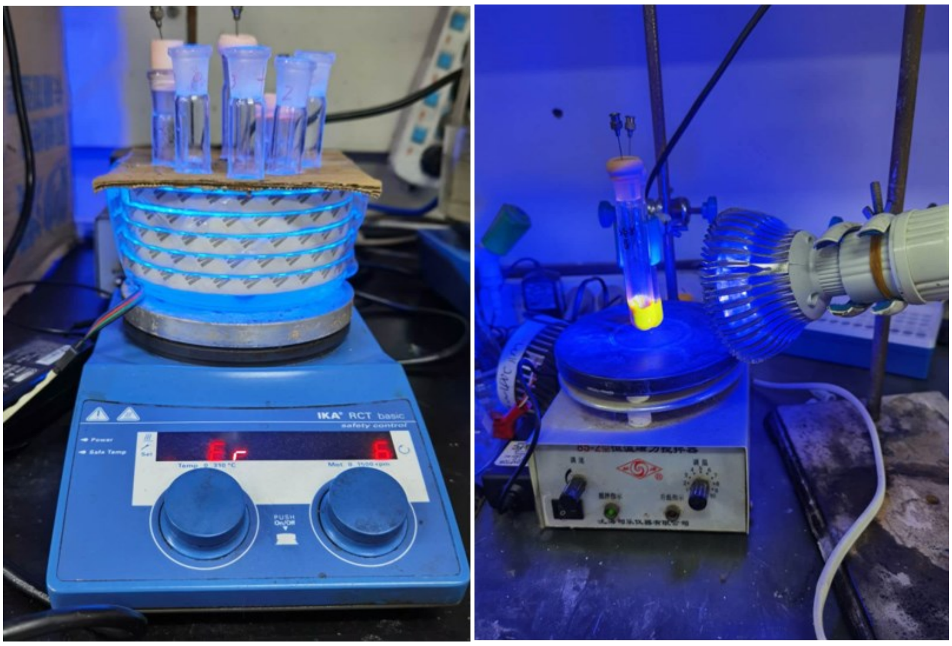
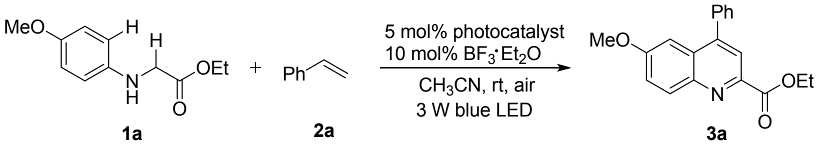


Figure S1. The reaction set-up of the visible light-induced reaction.

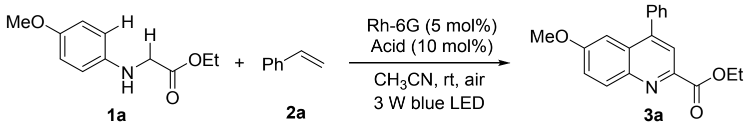
Table S1. Screening of Photocatalysts[a]



entry	photocatalyst	yield (%)[b]
1	Rhodamine 6G	11
2	Rose Bengal	10
3	$Acr^{+}MesClO_4^{-}$	-
4	Methylene blue	-

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.5 mmol), photocatalyst (5 mol%), $BF_3 \cdot Et_2O$ (10 mol%), CH_3CN (1.0 mL), 3 W blue LED light irradiation under air for 30 h. [b]Isolated yields.

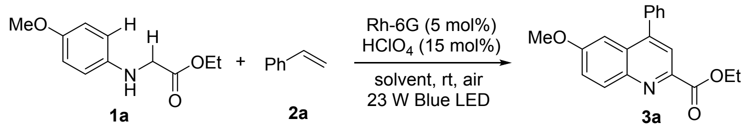
Table S2. Screening of Acids[a]



entry	acid	yield (%)[b]
1	$BF_3{\cdot}Et_2O$	11
2	TFA	20
3	Citric acid	21
4	TsOH	33
5	H_2SO_4	58
6[c]	TfOH	53
7	-	ND
8[c]	$HClO_4$	60
9[c, d]	$HClO_4$	68
10[d]	$HClO_4$	55
11[d, e]	$HClO_4$	64
12[d, f]	$HClO_4$	62

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.5 mmol), Rhodamine 6G (5 mol%), additive (10 mol%), CH_3CN (1.0 mL), 3 W blue LED light irradiation under air for 24 h. [b]Isolated yields. [c]15 mol% of additive was used. [d] Under the irradiation of 23 W blue LED strip. [e] 20 mol% $HClO_4$ was used. [f]30 mol% $HClO_4$ was used.

Table S3. Screening of Solvents[a]



entry	solvent	yield (%)[b]
1	CH_3CN	68
2	DCE	54
3	DCM	40
4[c]	**CH_3CN**	**75**
5[d]	CH_3CN	71
6[c,e]	CH_3CN	51

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.5 mmol), Rhodamine 6G (5 mol %), $HClO_4$ (15 mol%), solvent (1.0 mL), 23 W blue LED light irradiation under air for 24 h. [b]Isolated yields. [c]2.0 mL CH_3CN was used. [d]3.0 mL CH_3CN was used. [e]A 5 W green LED bulb was used as the light source.

2. Experiment Information

2.1 General Information.

Unless otherwise noted, all reagents were purchased from commercial sources and used as received without further purification. *N*-arylglycine derivatives[1,2] were prepared according to literature procedures. Unless otherwise indicated, all experiments were carried out under air atmosphere. Irradiation of photochemical reactions was carried out using a 3 W blue LED bulb or a 23 W blue LED strip. The silica gel (200−300 meshes) was used for column chromatography and TLC inspections were taken on silica gel GF254 plates. Liquid ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance III 400 MHz spectrometer. High resolution mass spectra (HRMS) were obtained on a mass spectrometer by using electrospray ionization (ESI) analyzed by quadrupole time-of-flight (QTof). Luminescence spectra were surveyed on a PerkinElmer LS 55 spectrophotometer. EPR spectra were recorded at room temperature using an EPR spectrometer at 9.448 GHz.

2.2 General Procedure for the Metal-Free Visible Light-Induced Oxidative Dehydrogenative Coupling/Aromatization Tandem Reaction of Glycine Esters and Alkenes.

To a solution of *N*-arylglycine ester **1a** (20.9 mg, 0.1 mmol, 1 eq) and Rhodamine 6G (2.4 mg, 0.005 mmol, 5 mol%) in dry CH_3CN (2.0 mL, 0.05 M) were added $HClO_4$ (the $HClO_4$ used in this study was purchased as neat; 1.5 mg, 0.015 mmol, 15 mol%) and styrene **2a** (52.1 mg, 57.3 μL, 0.5 mmol, 5 eq). The mixed solution was irradiated with blue LED ((3 W in optimization, 23 W in scope) under air atmosphere at room temperature. After completion of the reaction as monitored by TLC, the solvent was removed under vacuo, and the residue was separated by silica gel column chromatography (with petroleum ether/EtOAc = 8/1 as eluent) to afford the product **3a**.

3. Luminescence Quenching Experiments

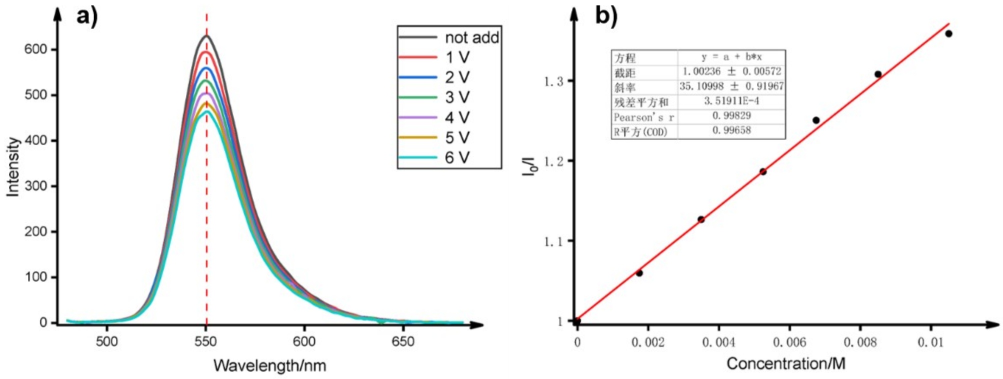


Figure S2. a) Changes in the fluorescence spectra of Rh-6G (1.0×10^{-6} M) upon the progressive addition of **1a** (1.5×10^{-3} M) in CH_3CN; b) Stern–Volmer quenching plot of Rh-6G in the presence of **1a**.

4. EPR Spectra

General Information.

EPR spectra were recorded at room temperature using an EPR spectrometer at 9.445 GHz. Typical spectrometer parameters are shown as follows, sweep width: 100.0 G; center field set: 3362.0 G; time constant: 81.920 ms; sweep time:75.0 s; modulation amplitude: 0.8 G; modulation frequency: 100.0 kHz; receiver gain: 1.00×10^3; microwave power: 24.640 mW.

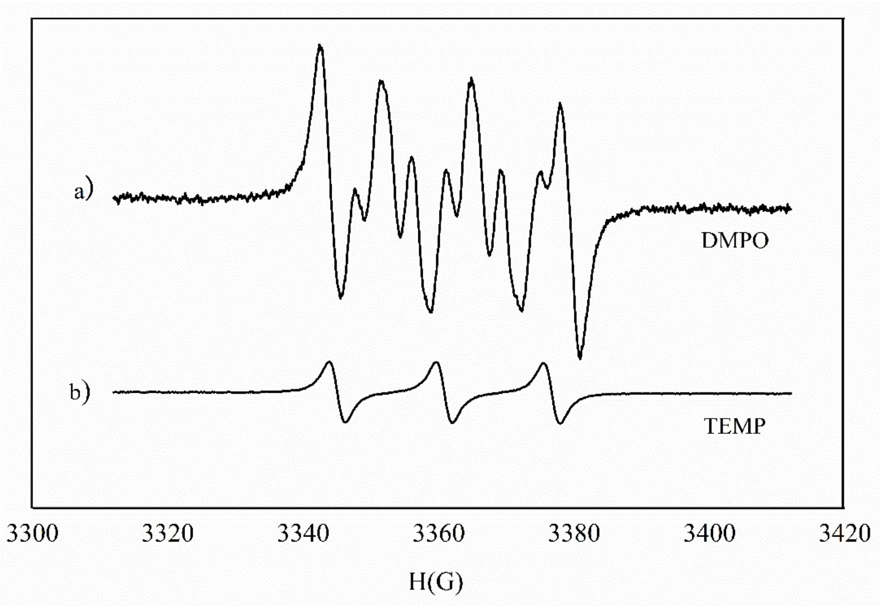


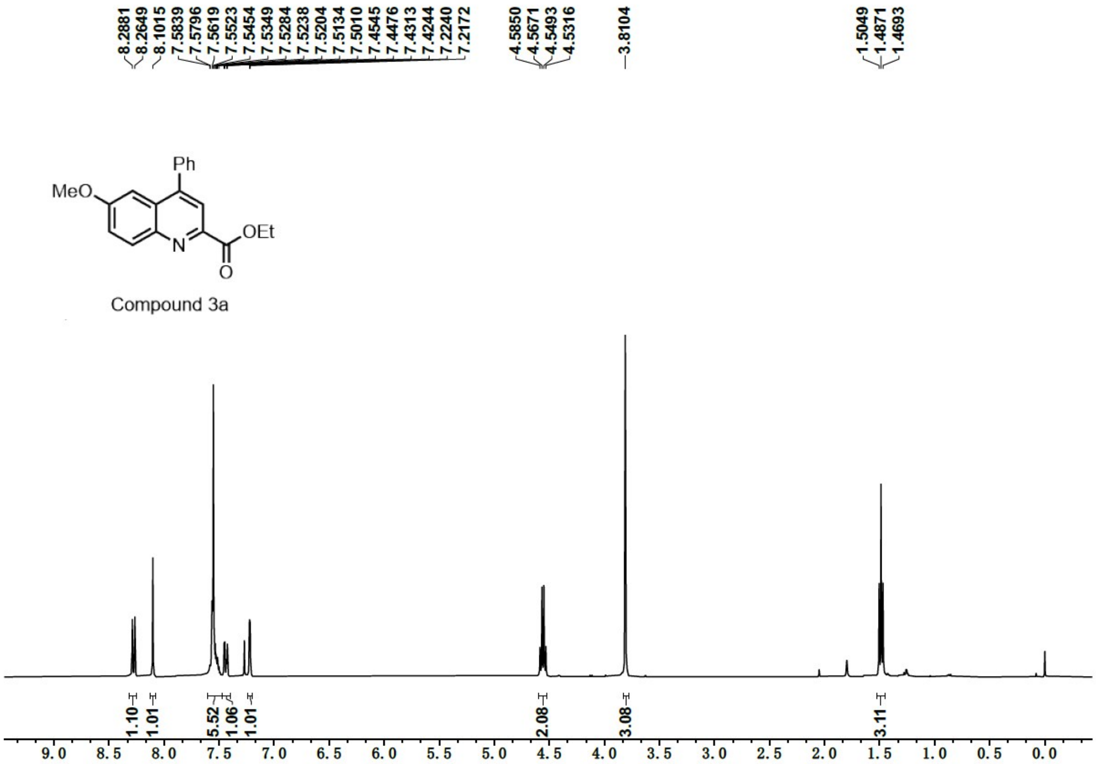
Figure S3. EPR spectra of a solution of a) **1a** (0.01 $molL^{-1}$), Rhodamine 6G (5×10^{-4} $molL^{-1}$), and DMPO (0.1 $molL^{-1}$) in air-saturated CH_3CN upon irradiation with blue LED for 100 s; b) **1a** (0.01 $molL^{-1}$), Rhodamine 6G (5×10^{-4} $molL^{-1}$), and TEMP (0.12 $molL^{-1}$) in air-saturated CH_3CN upon irradiation with blue LED for 100 s.

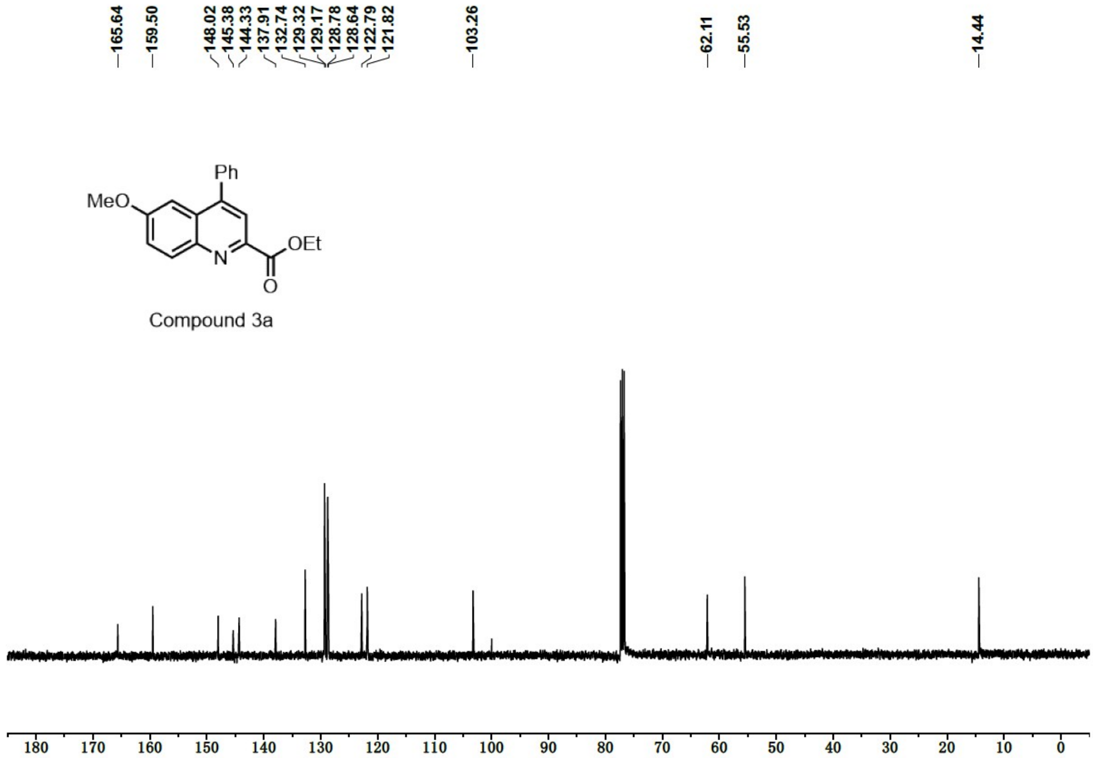
5. References

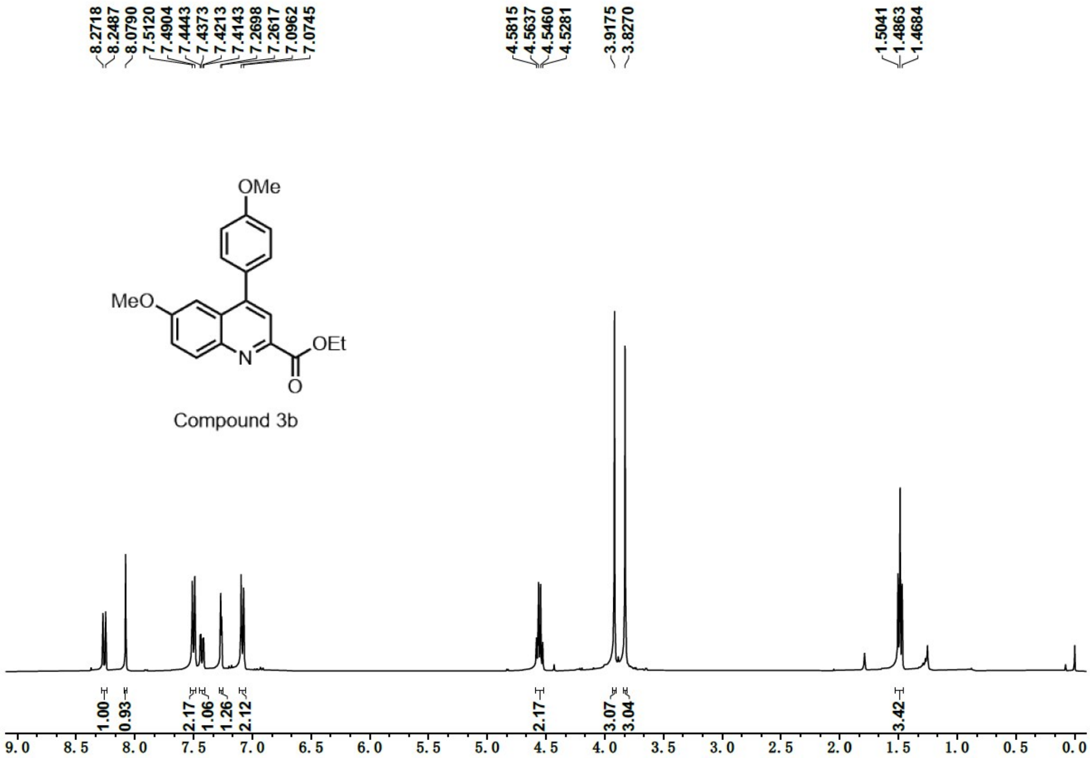
[1] Xie, J.; Huang, Z.-Z. *Angew. Chem., Int. Ed.* **2010**, *49*, 10181.

[2] Zhao, L.; Basle, O.; Li, C.-J. *Proc. Natl. Acad. Sci. U. S. A.* **2009**, *106*, 4106.

6. Copies of ^{1}H and ^{13}C NMR Spectra



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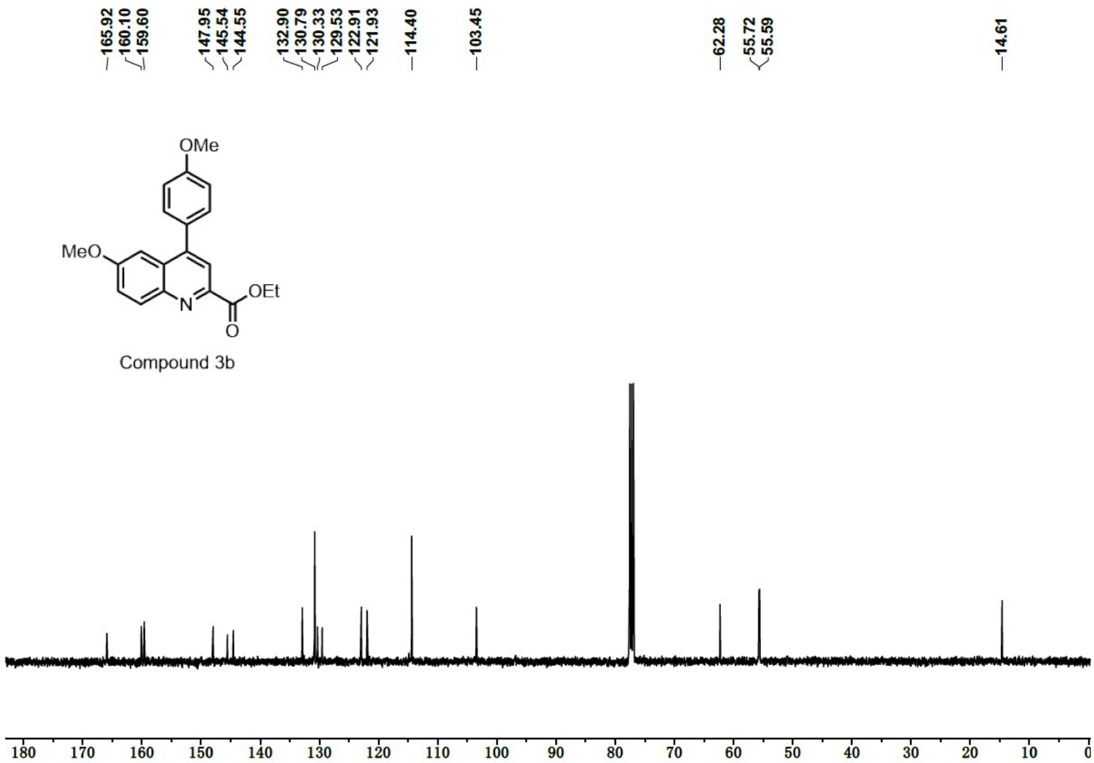
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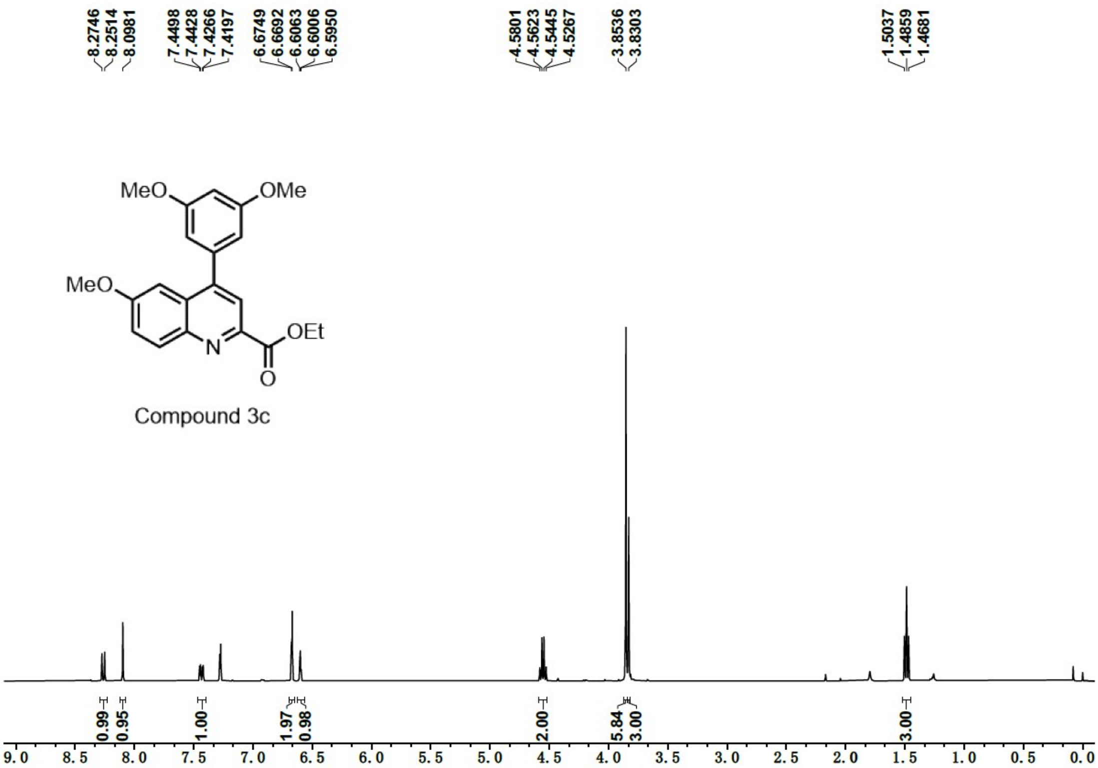
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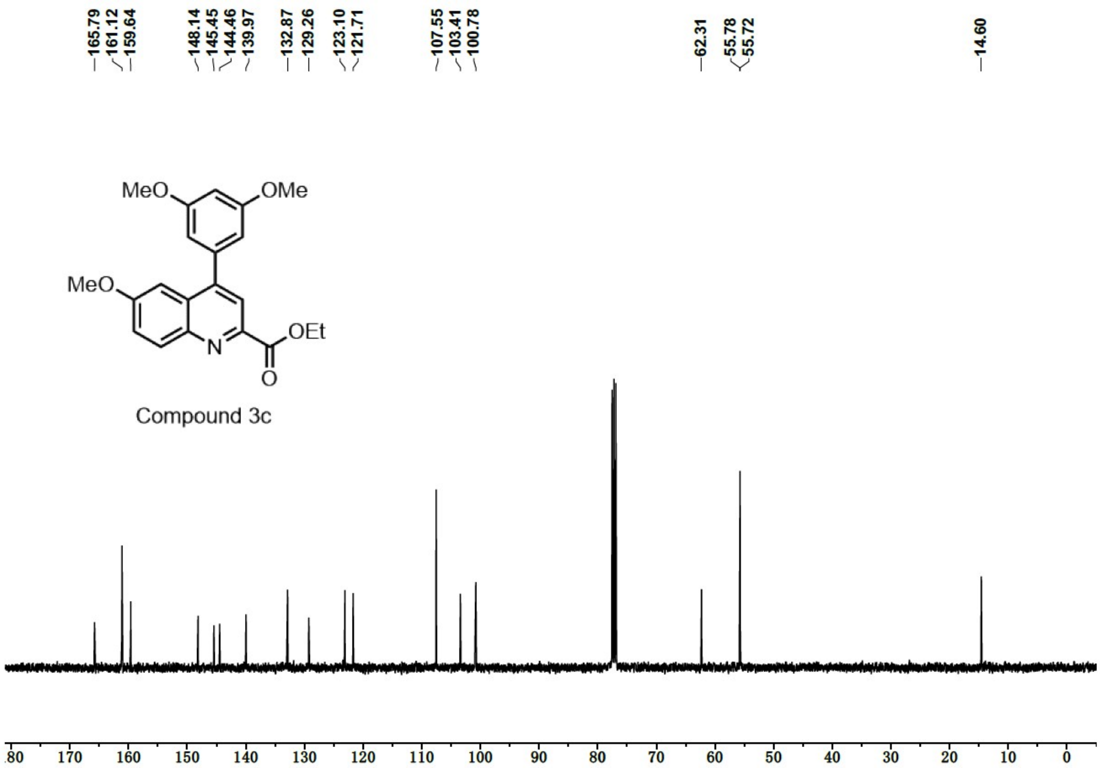
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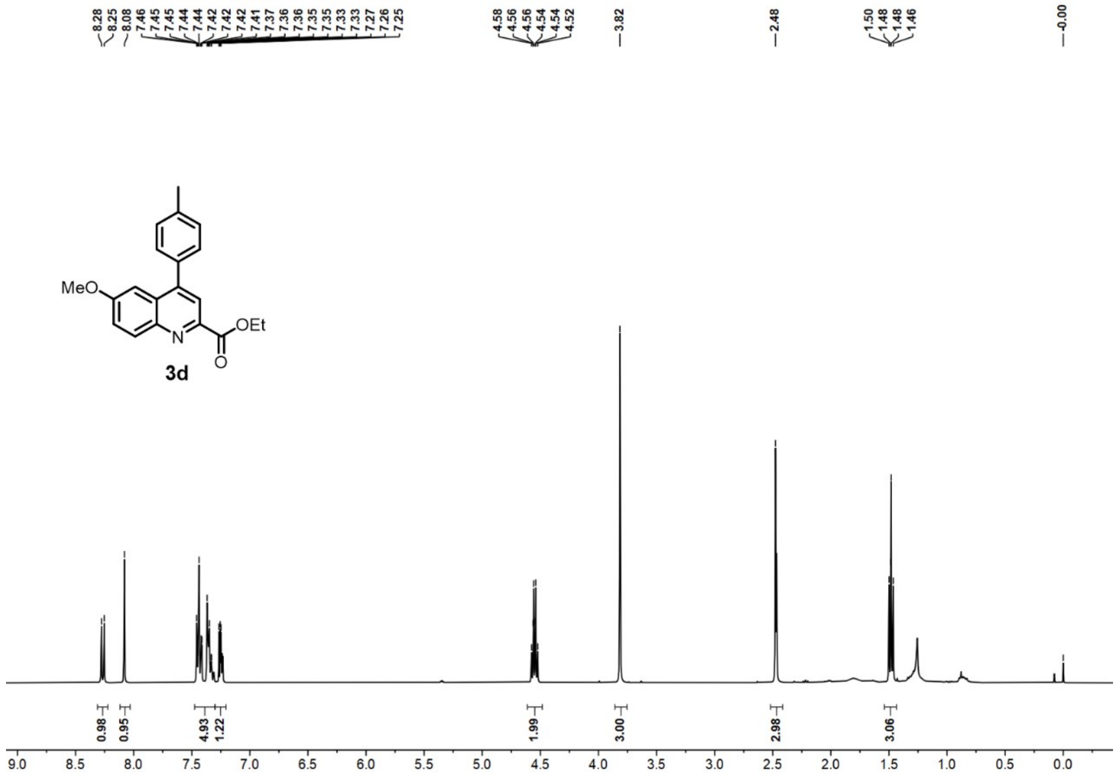
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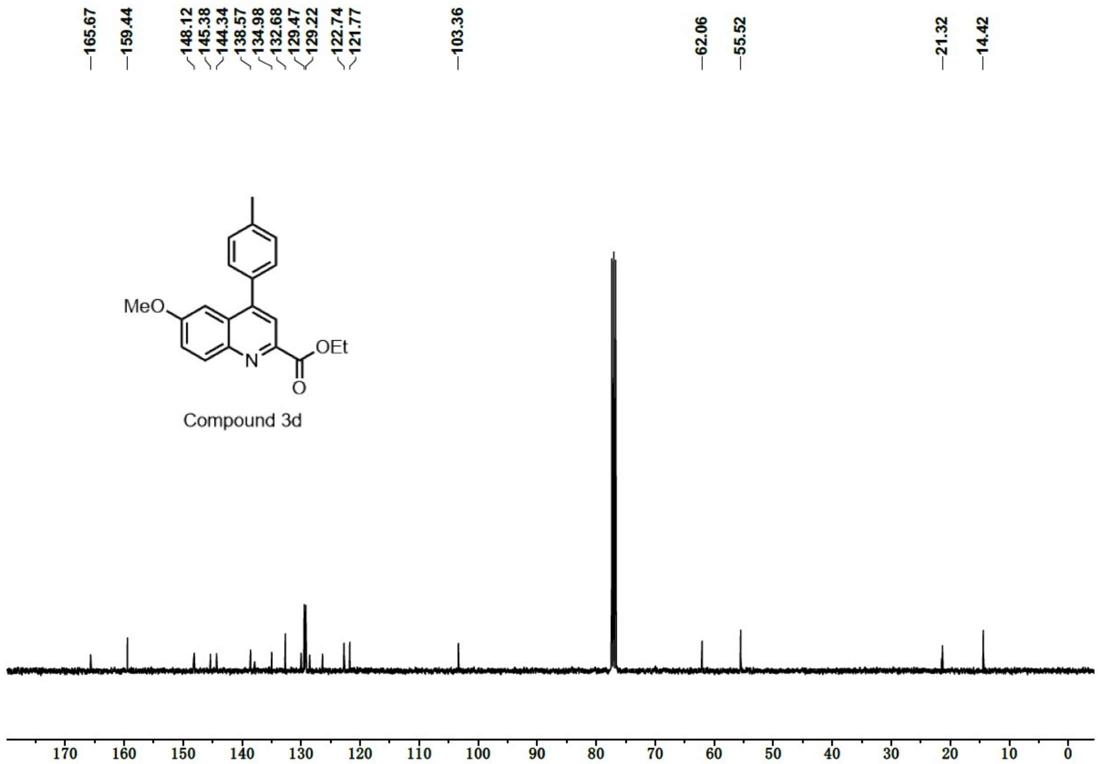
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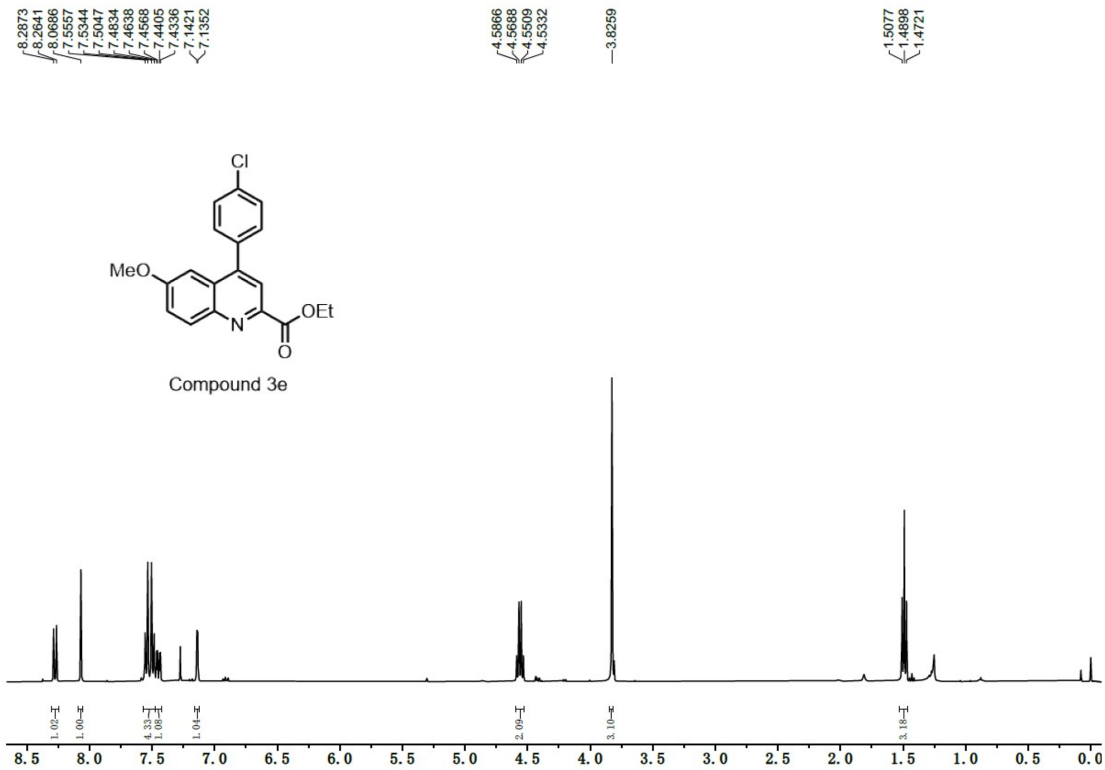
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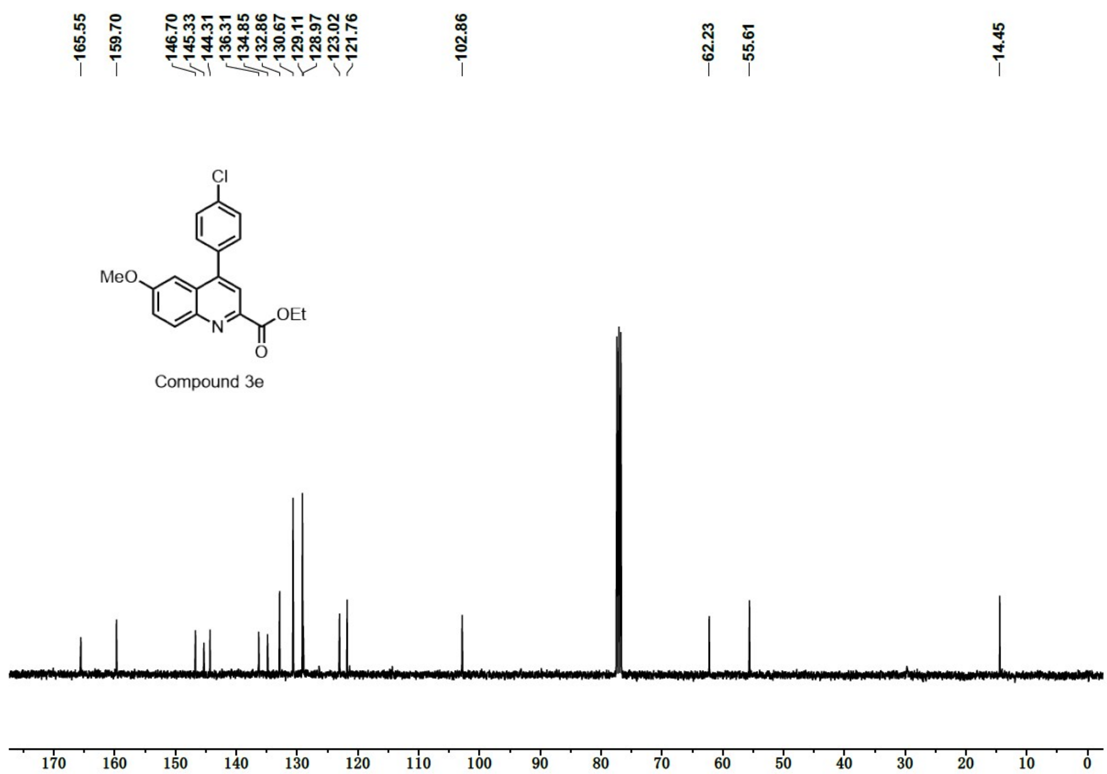
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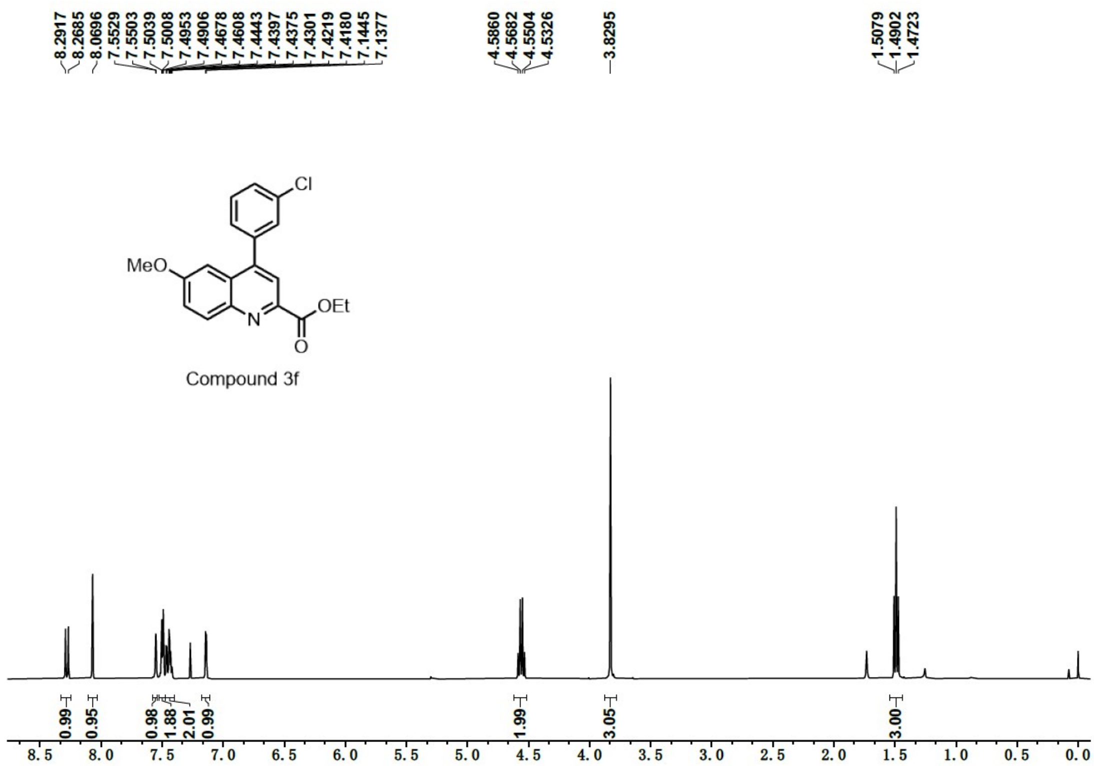

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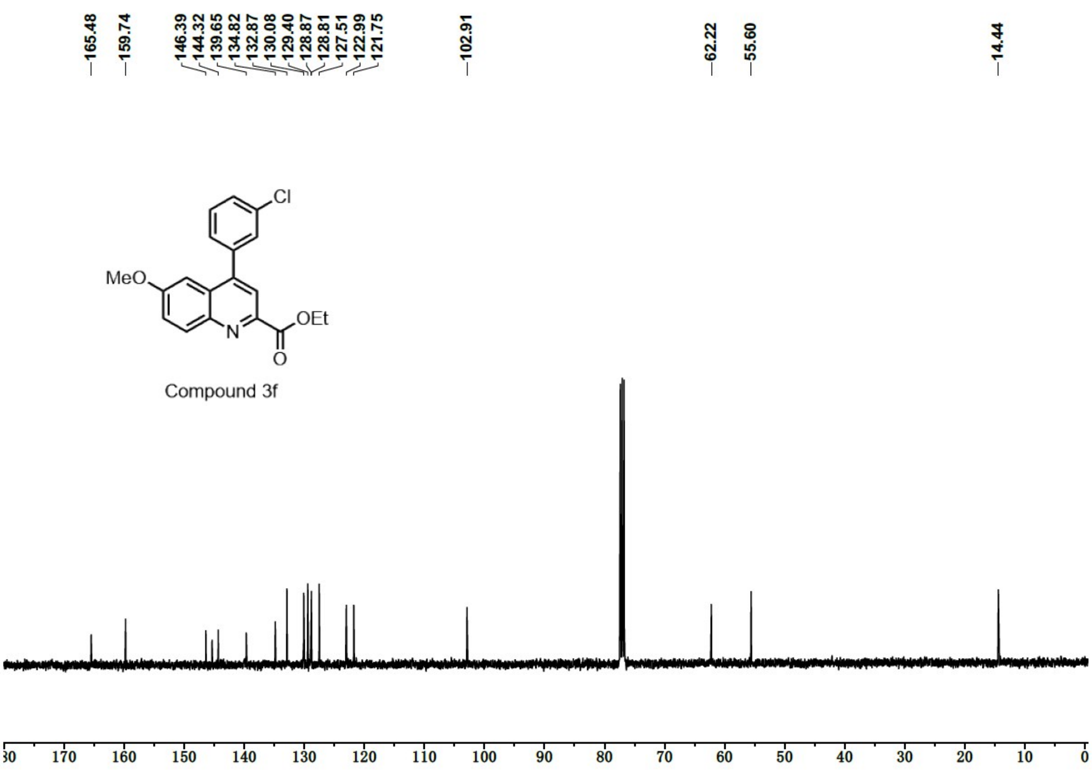

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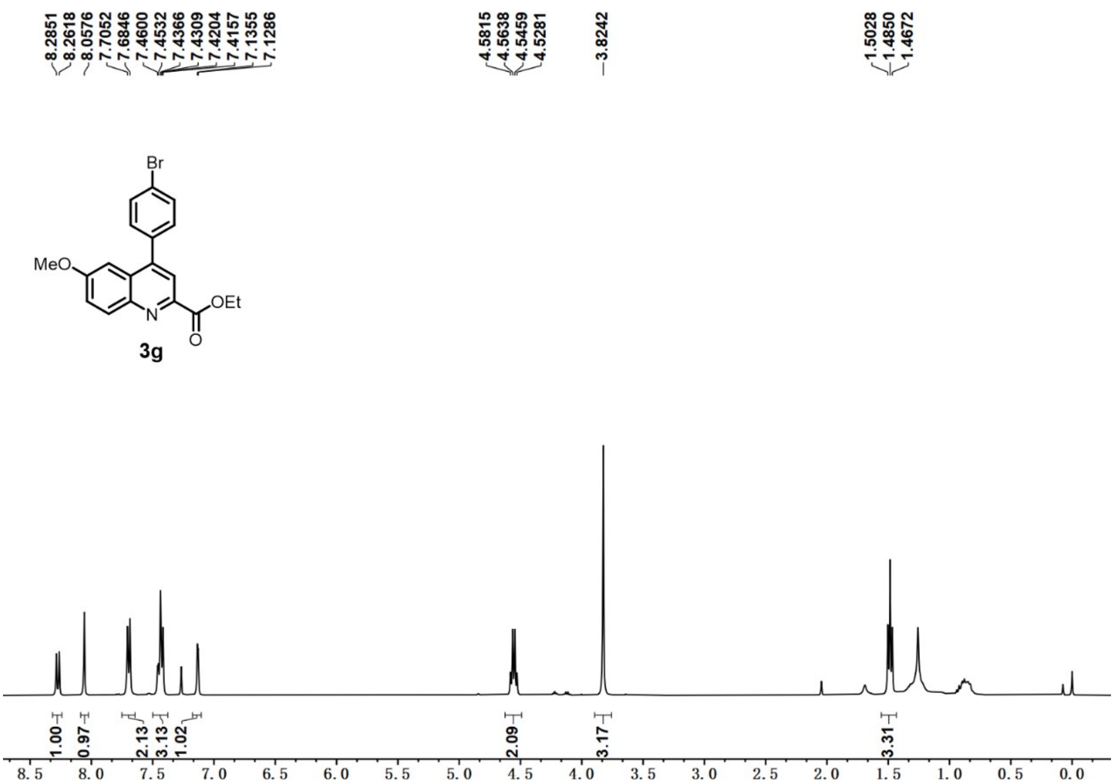
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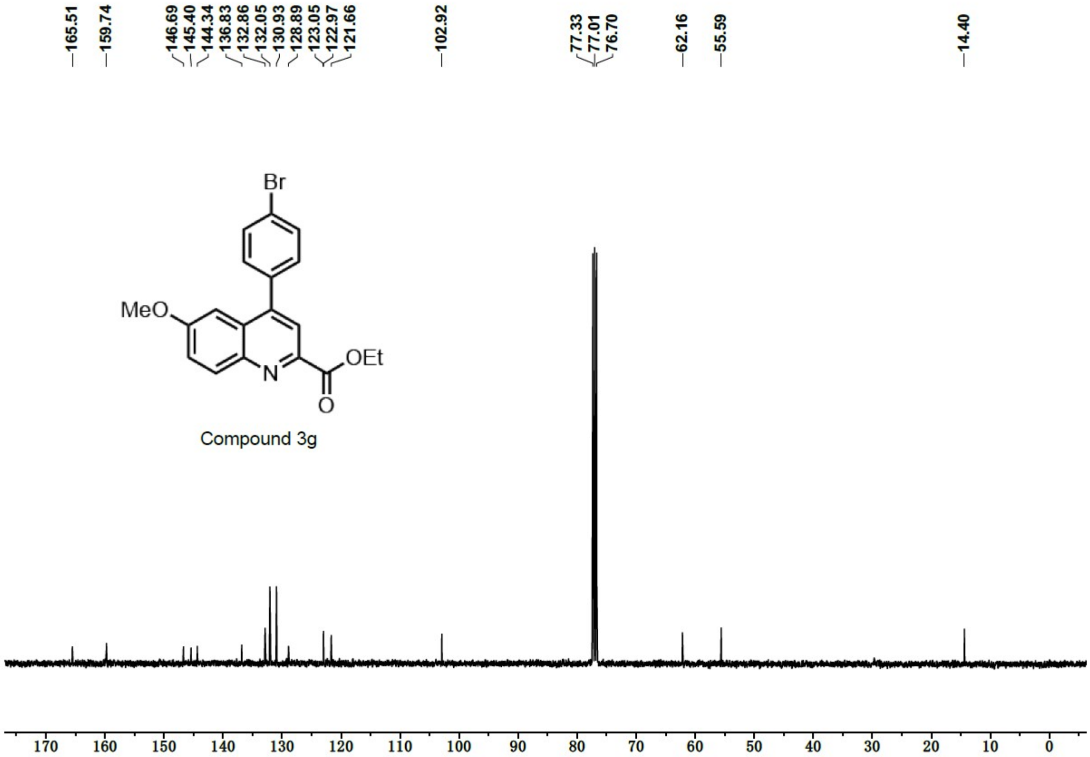
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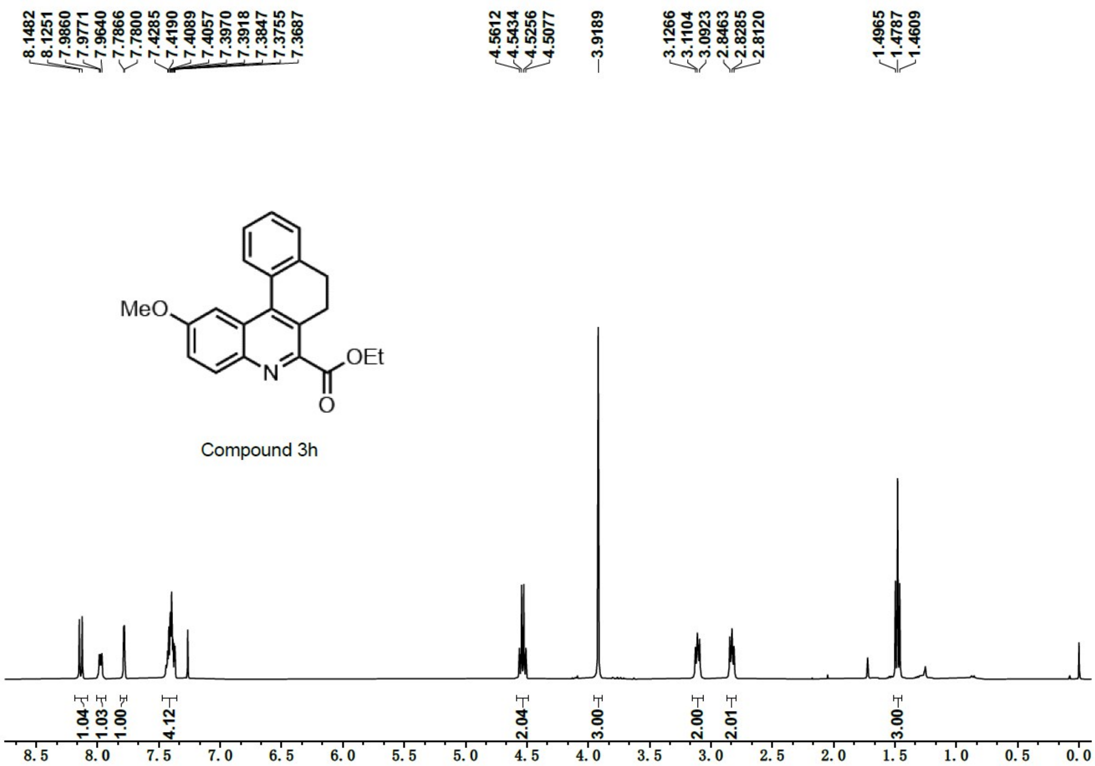
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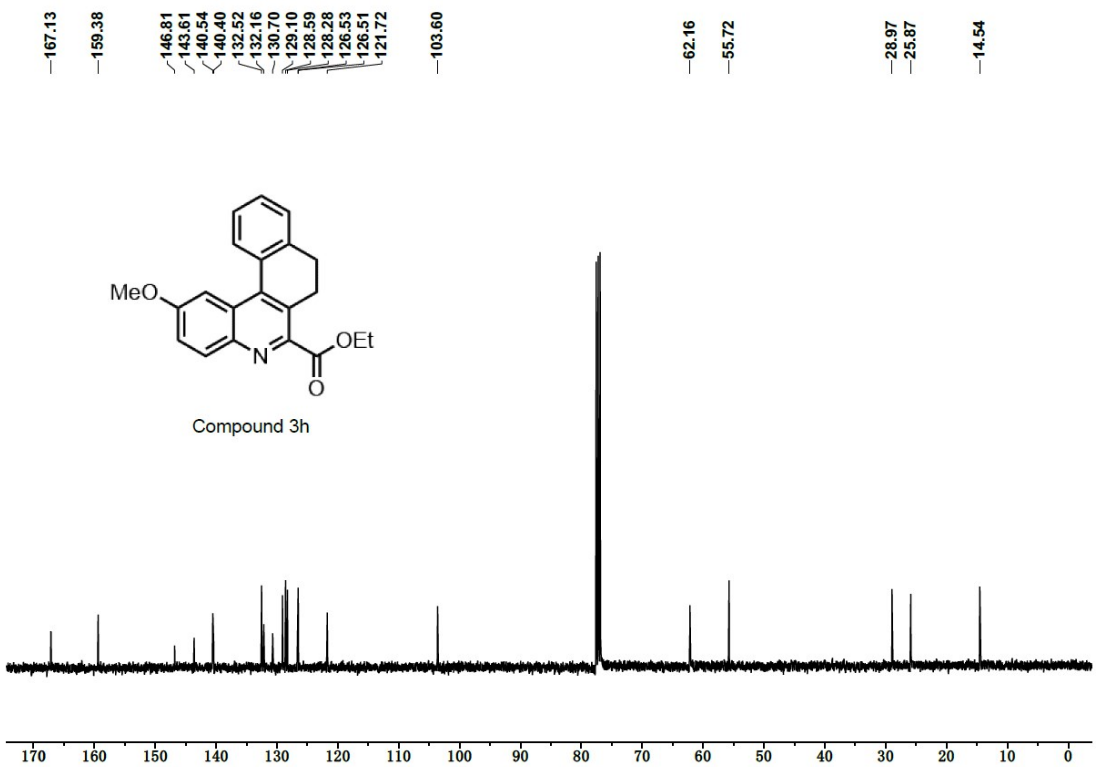
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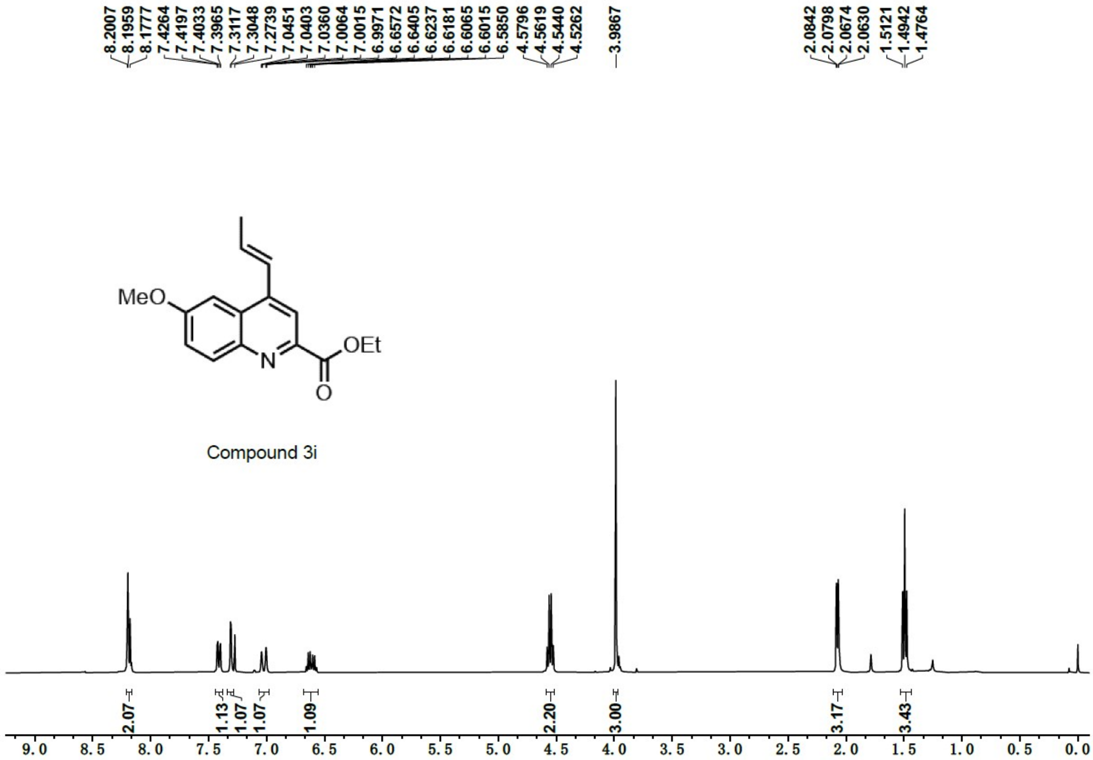
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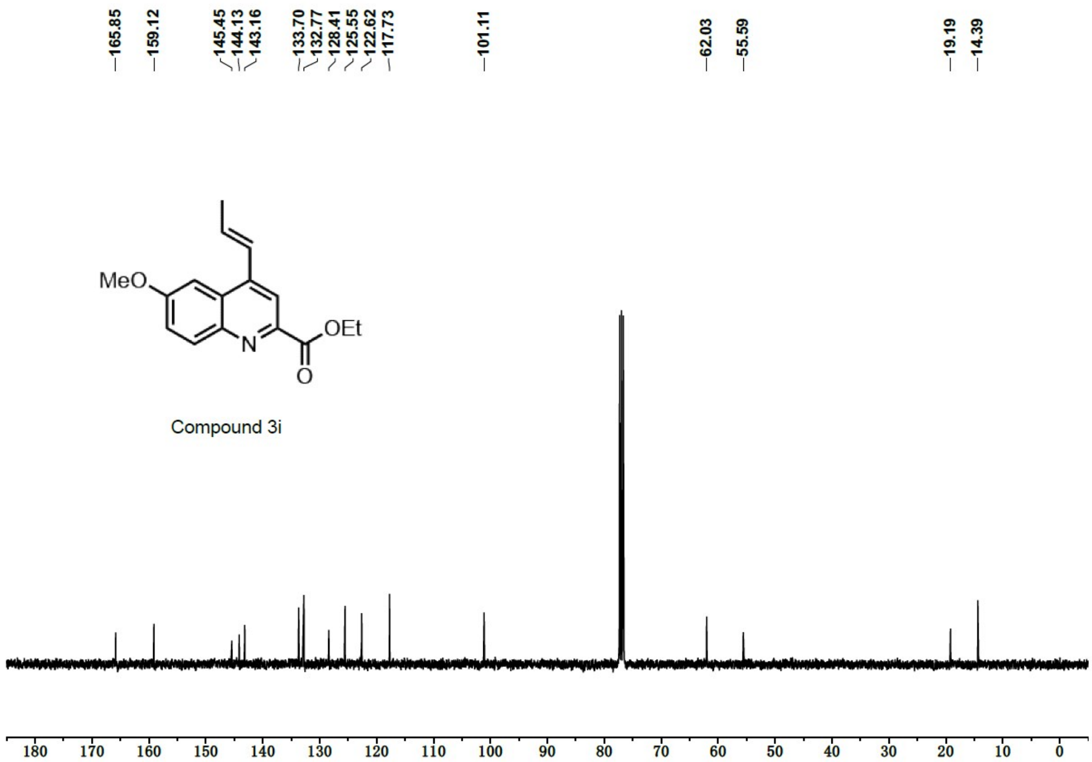
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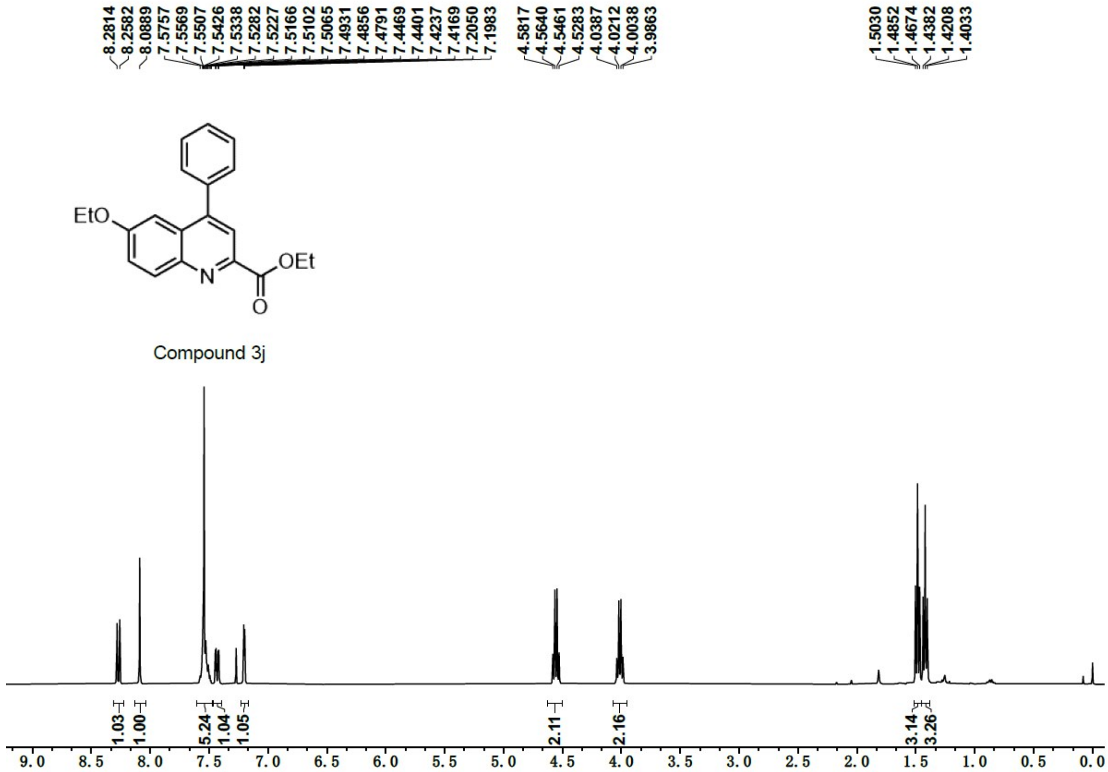
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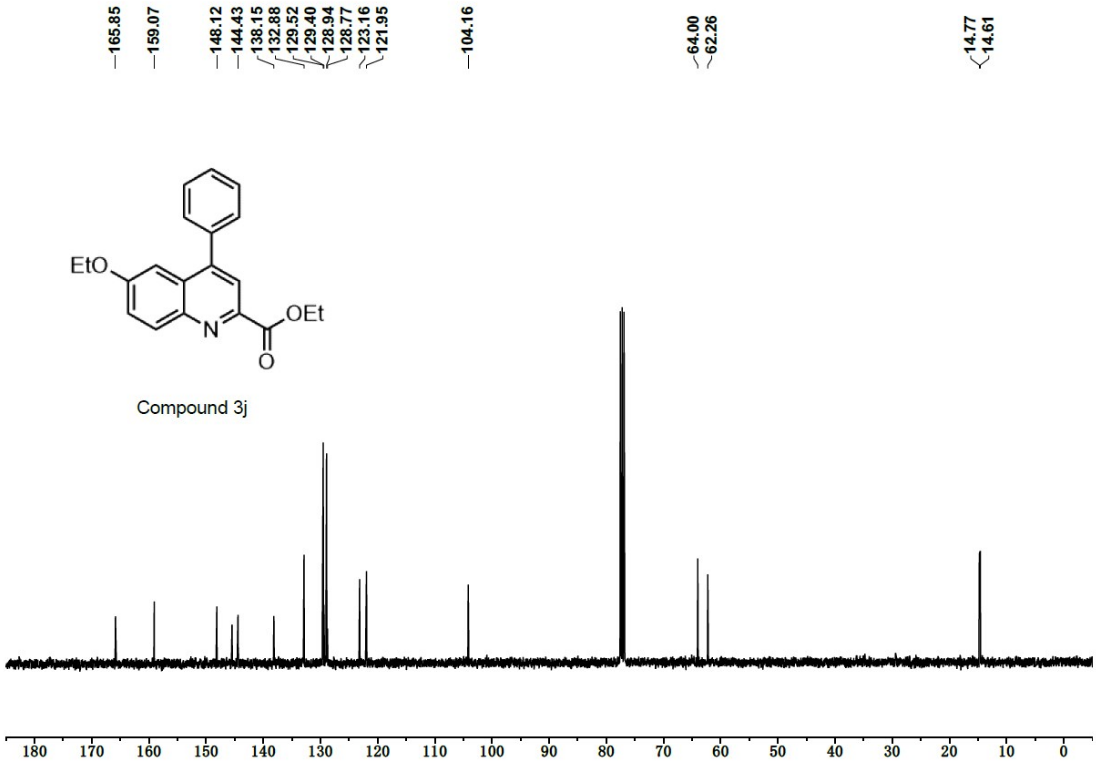
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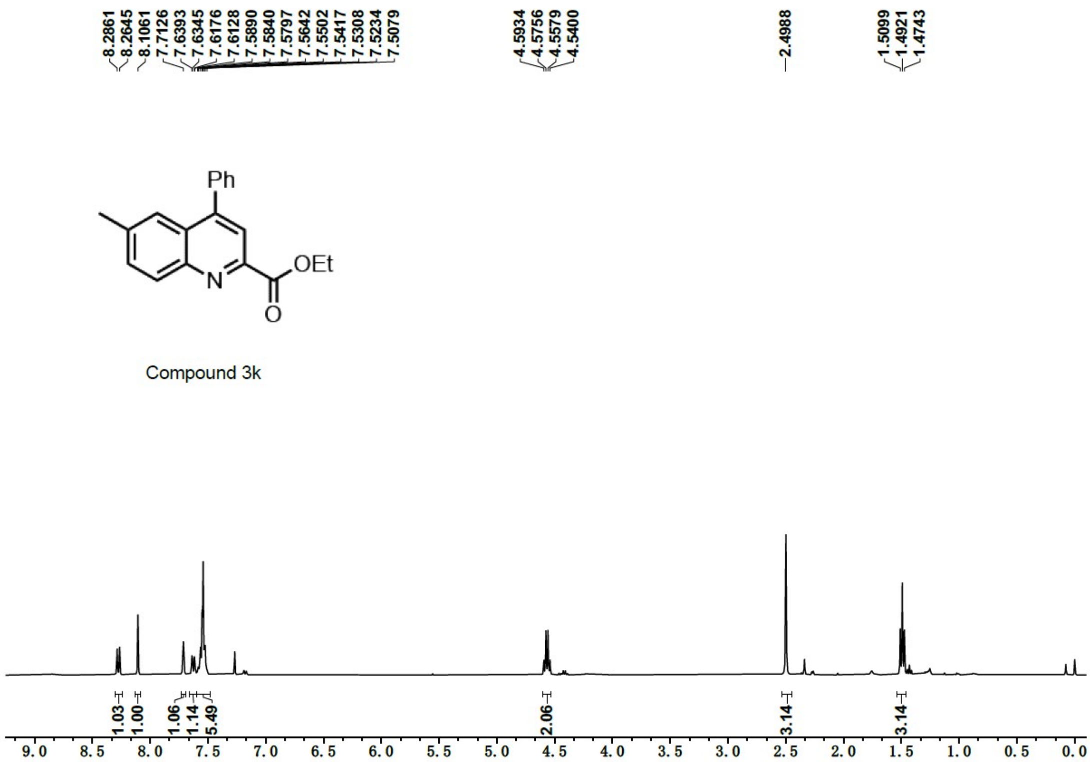
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O
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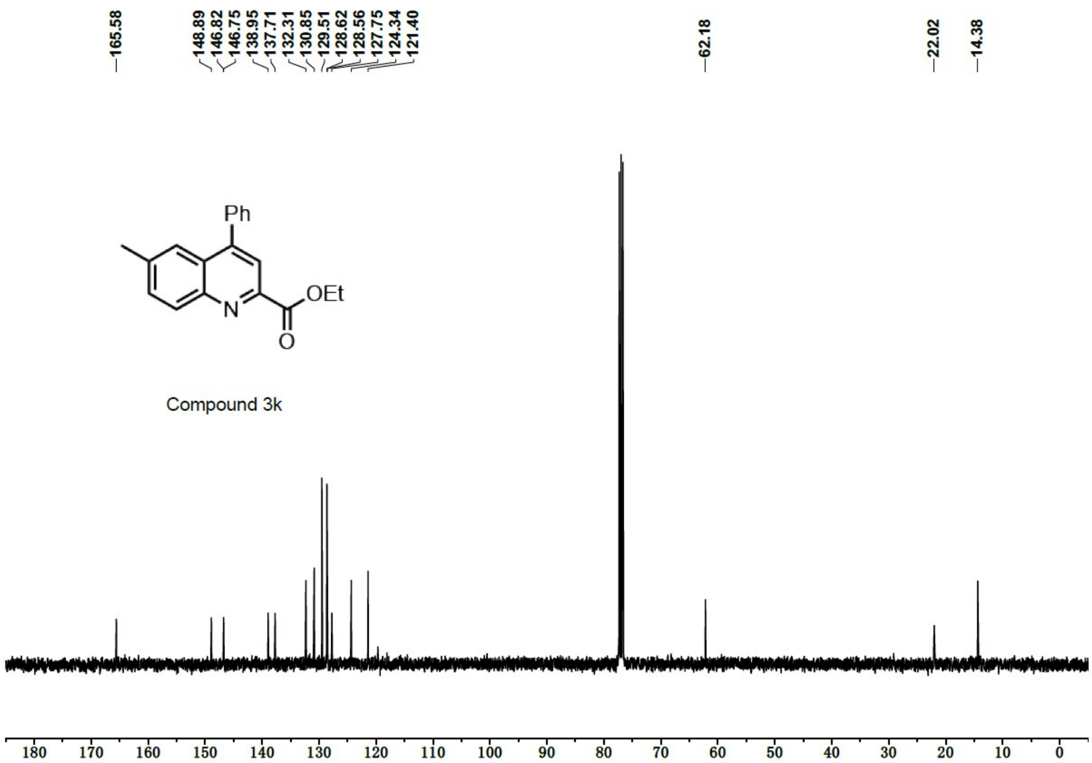
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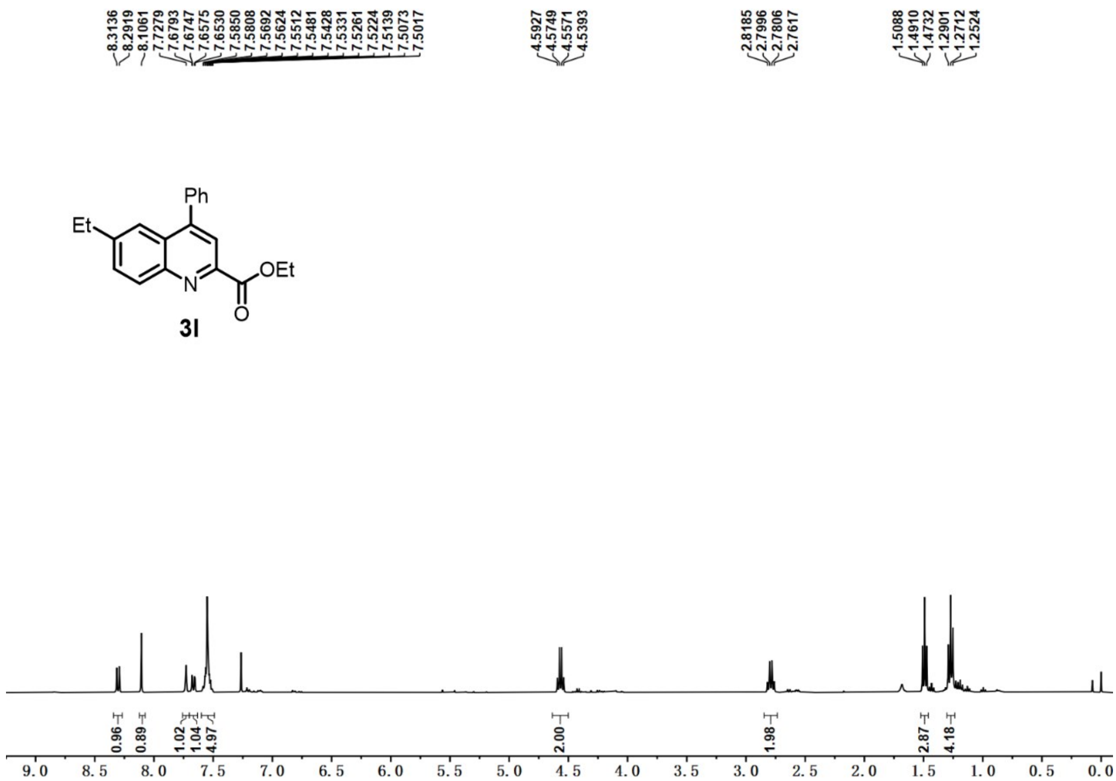

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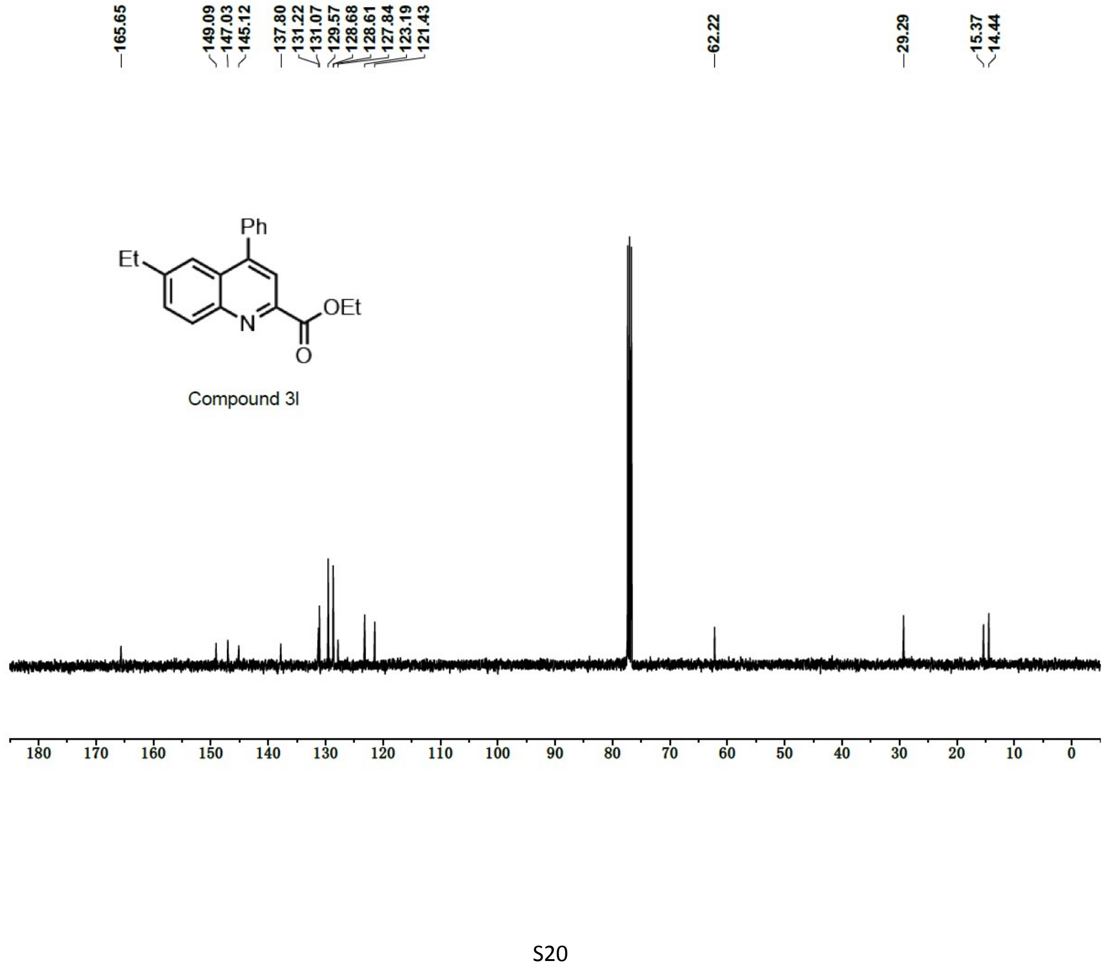

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N
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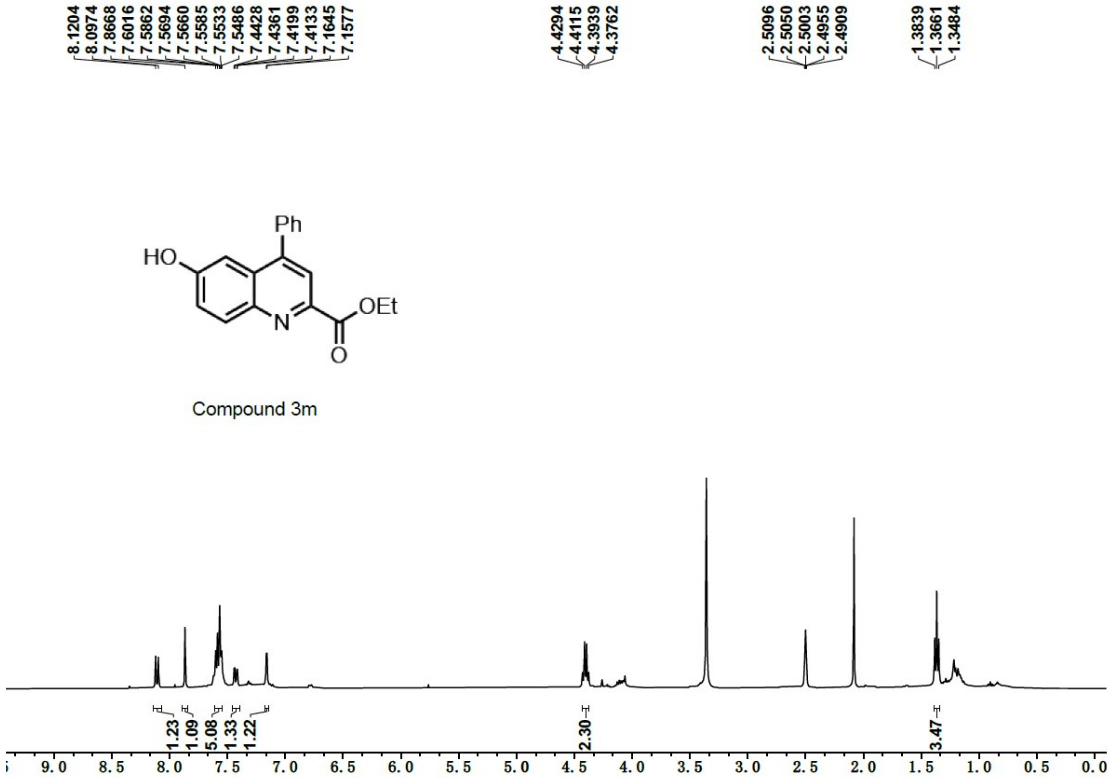
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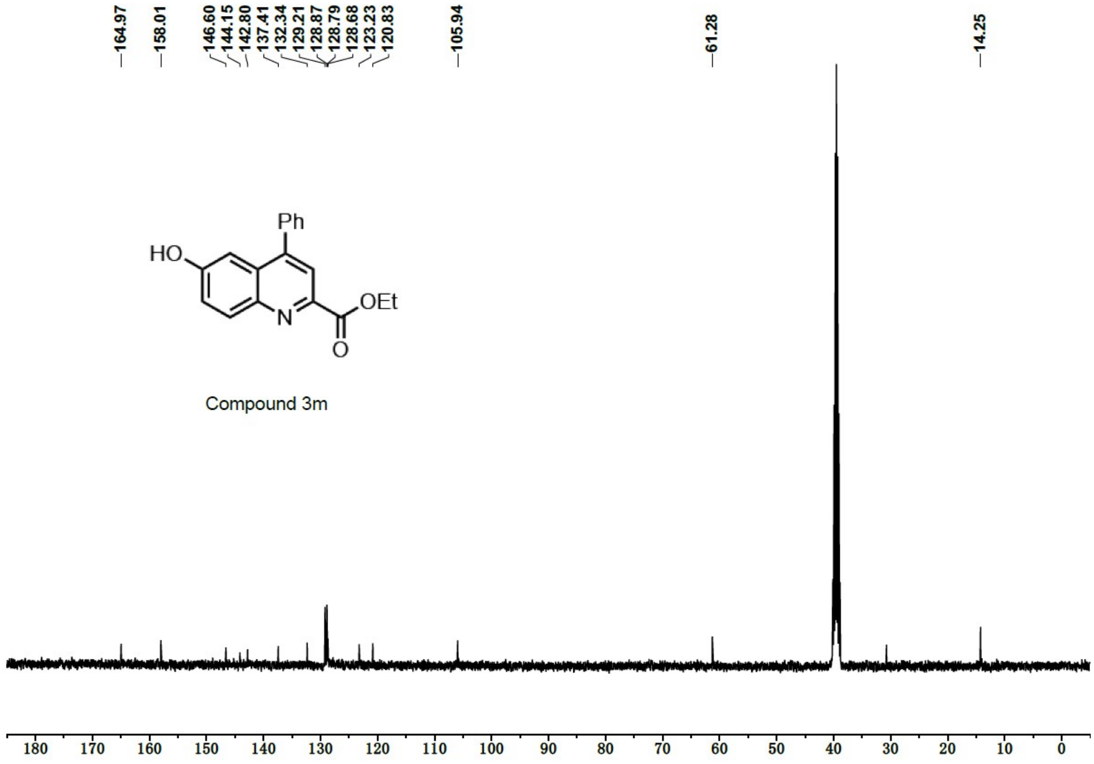
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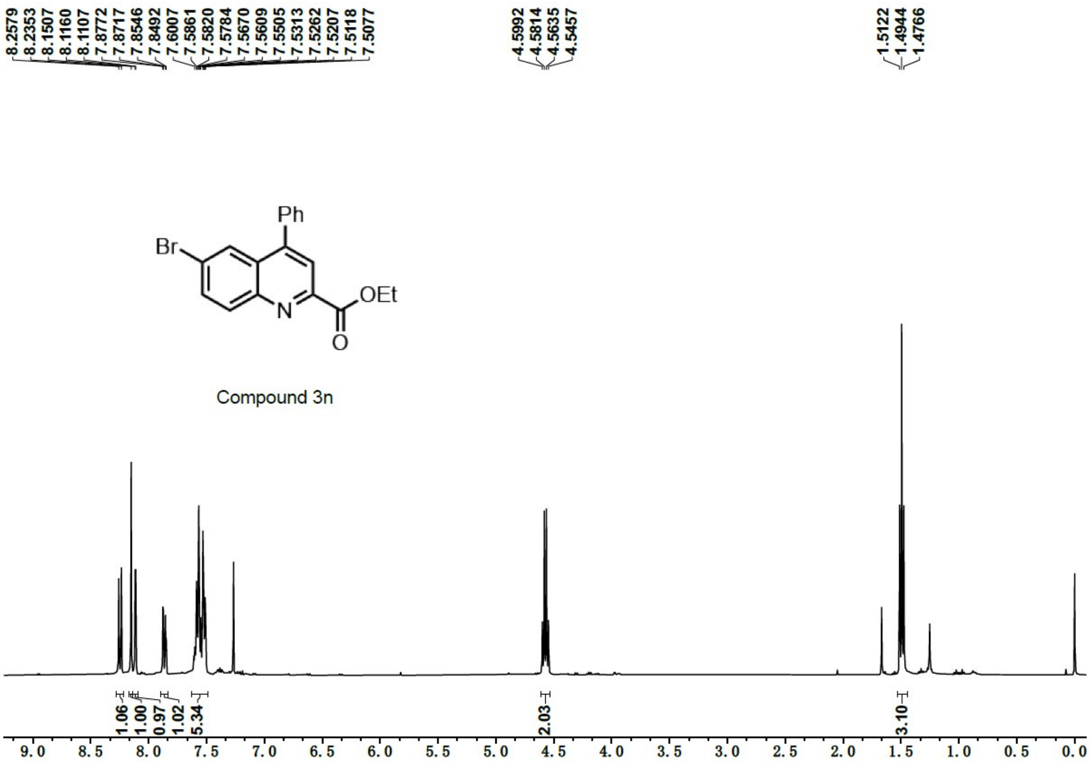
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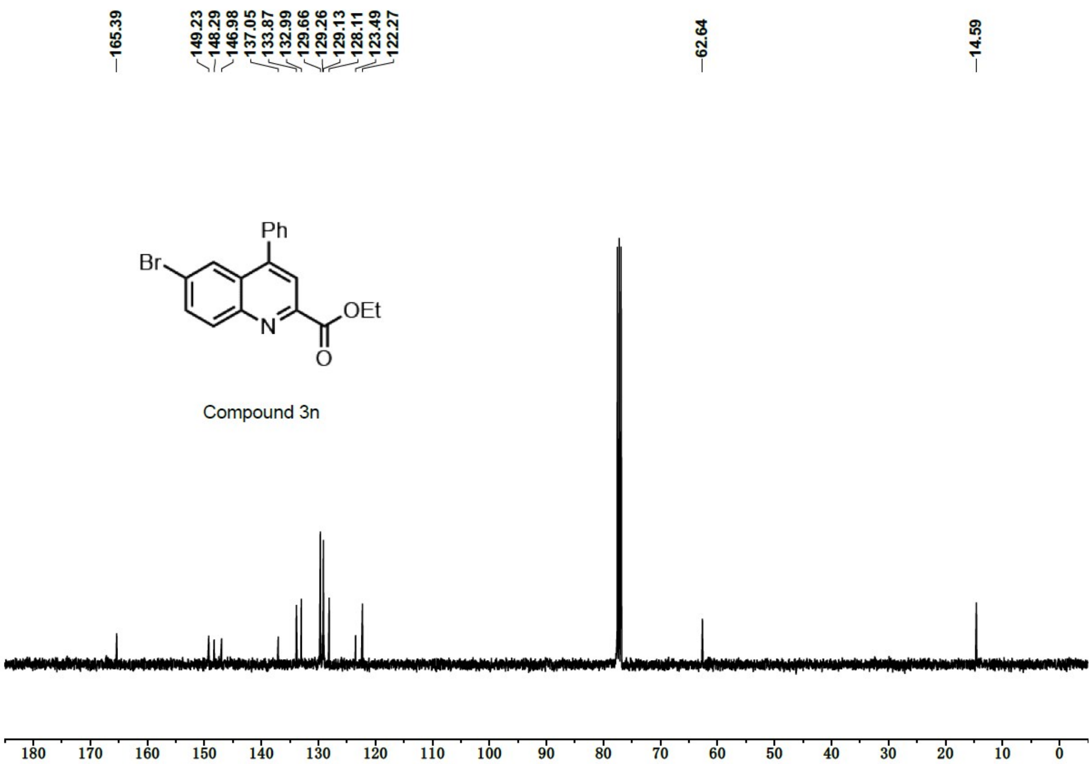
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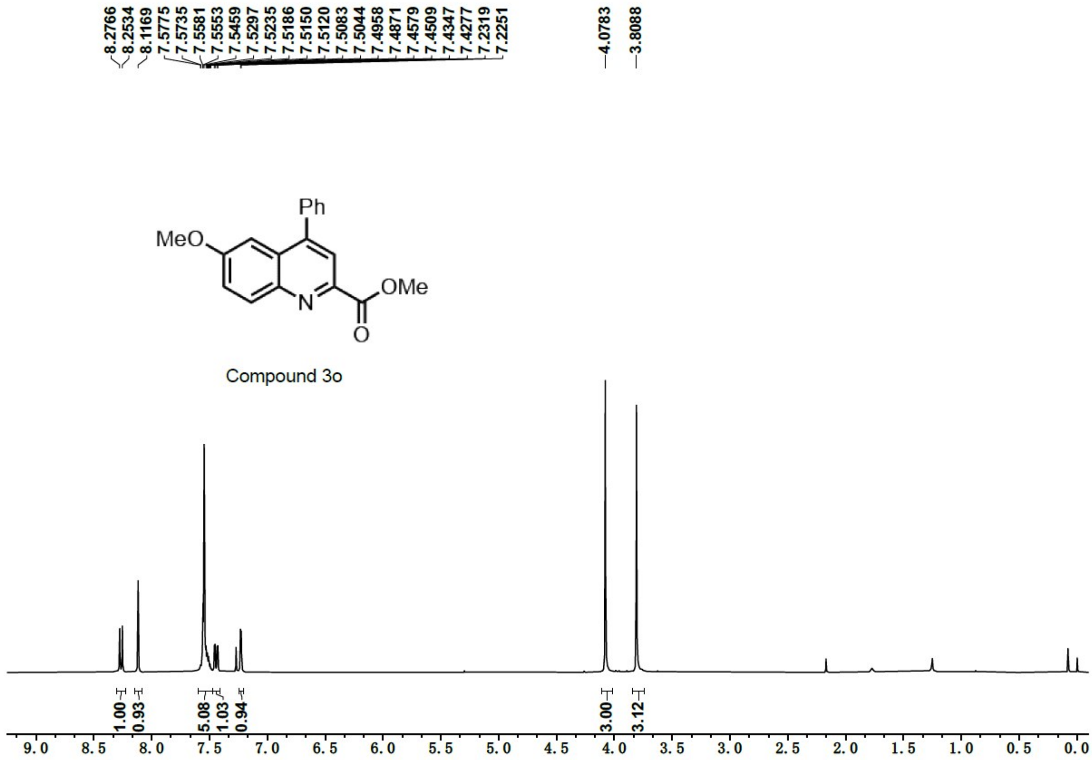
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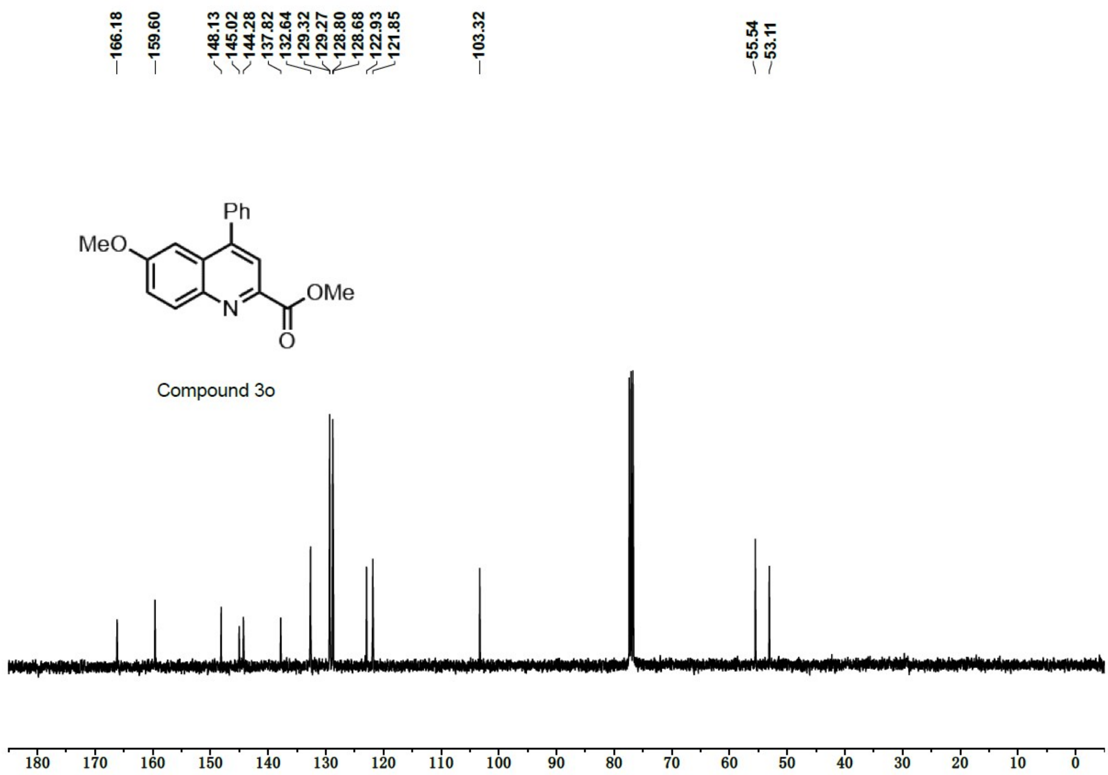
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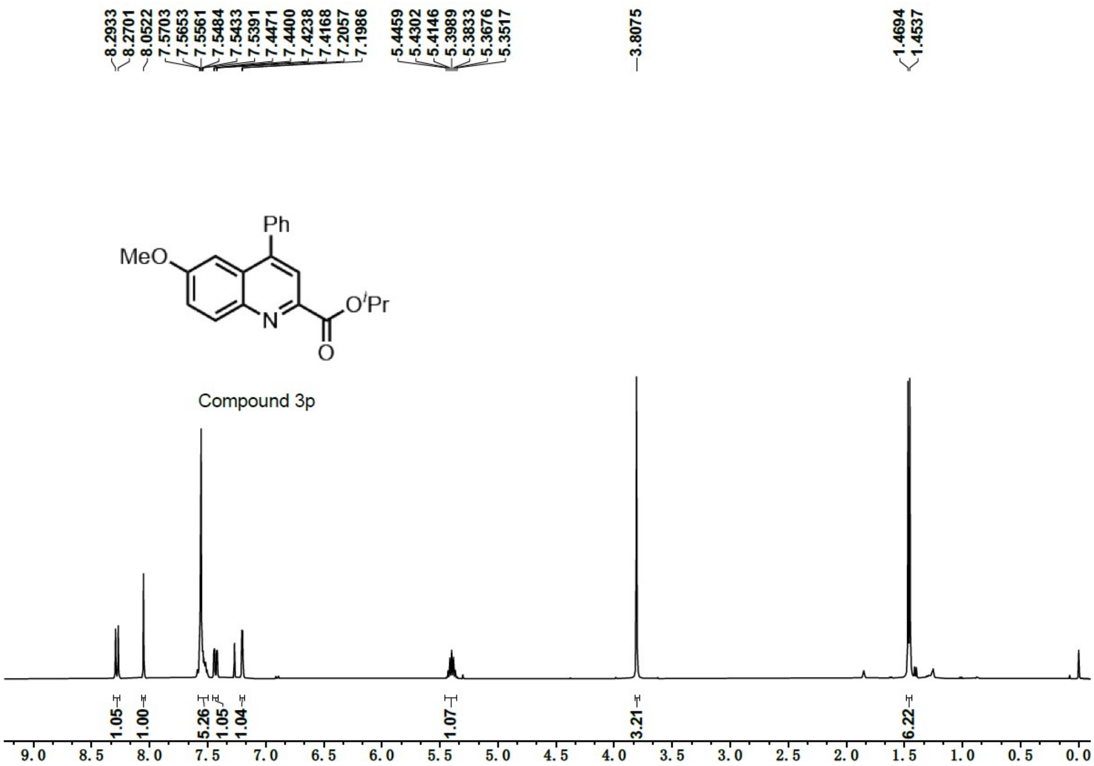

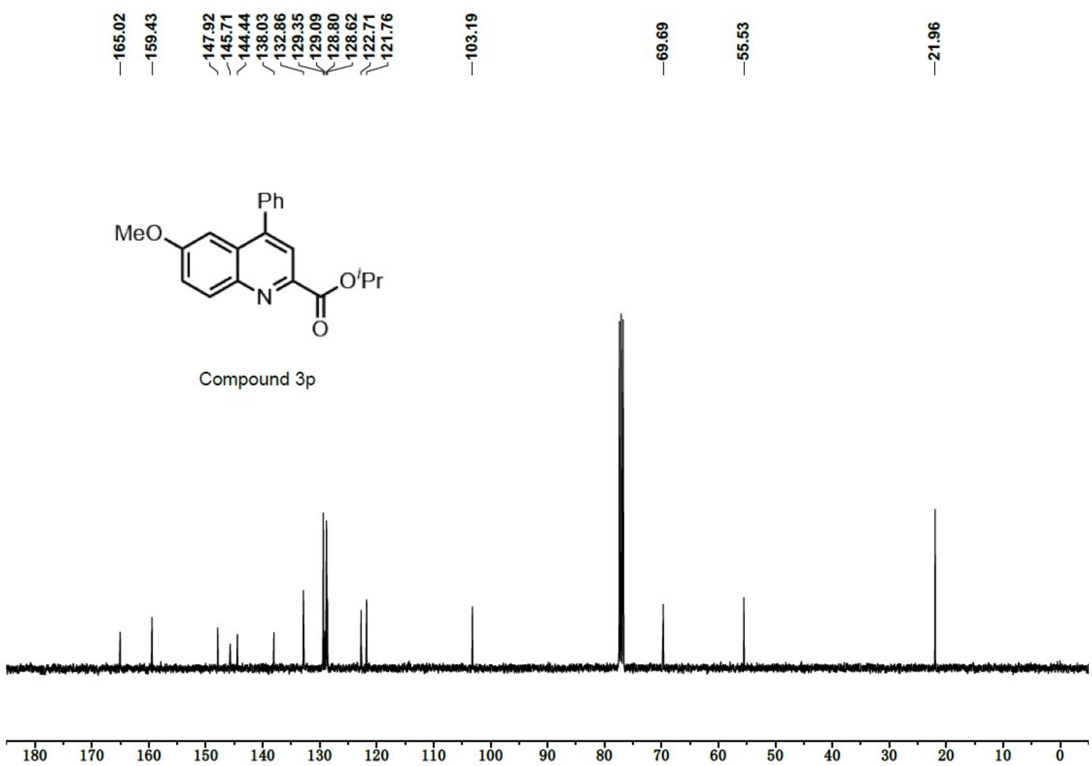

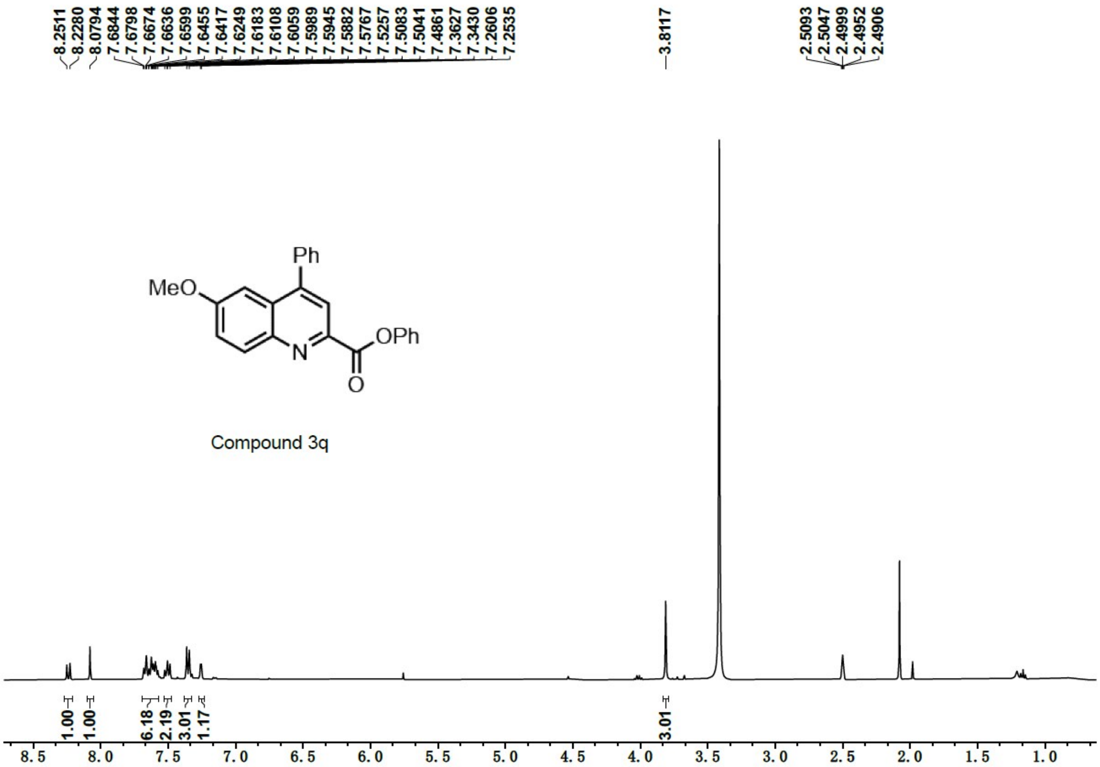
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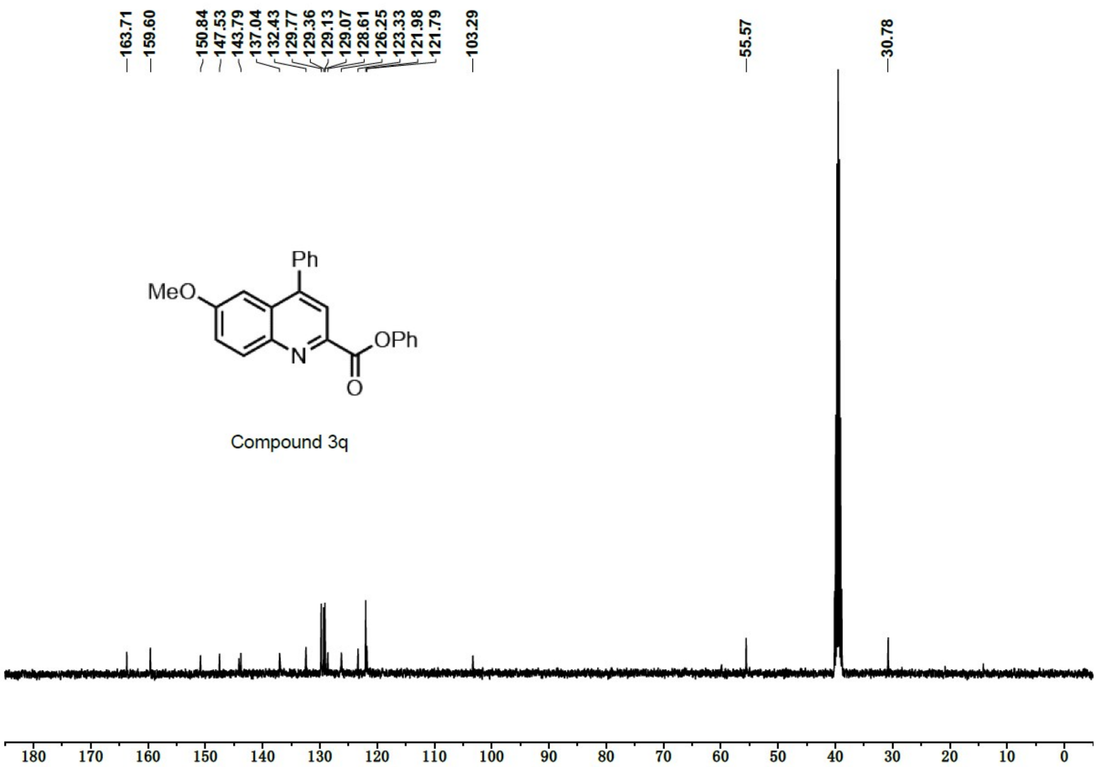
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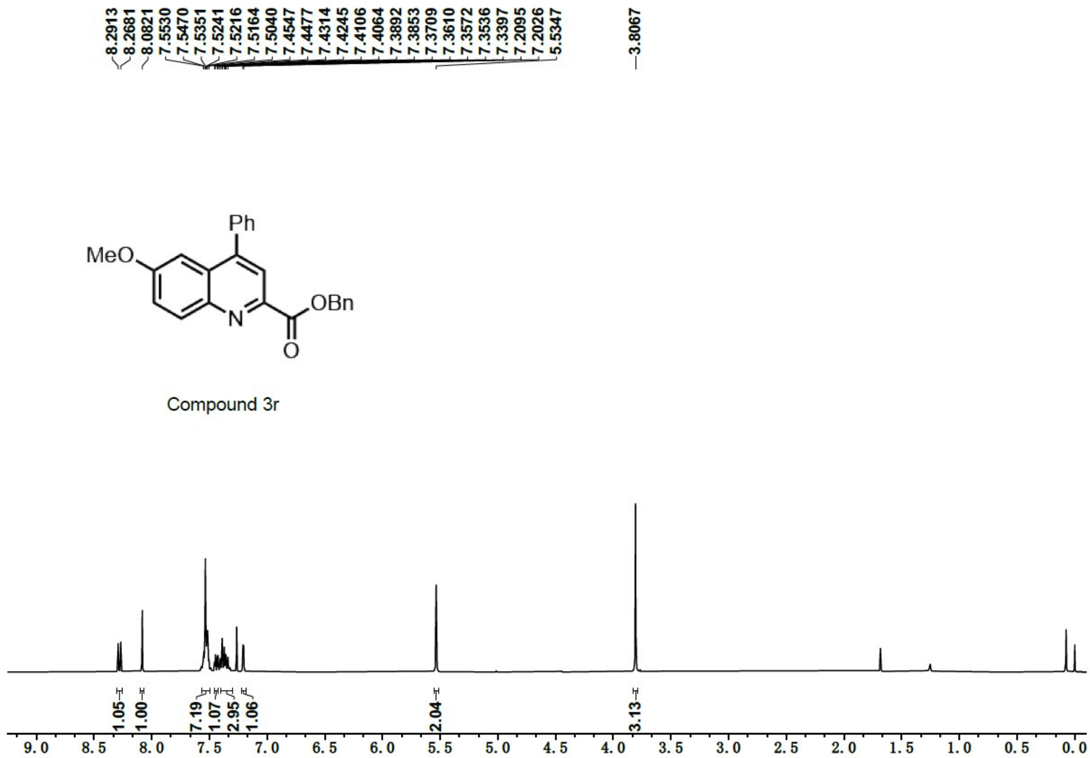
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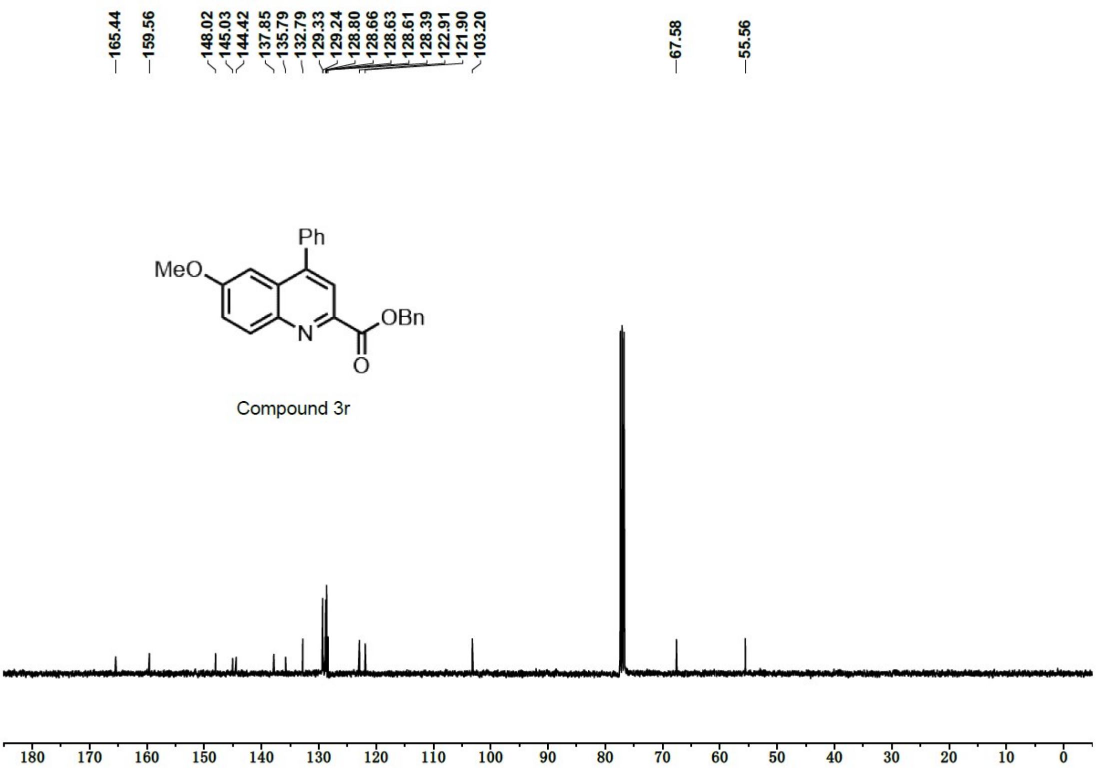
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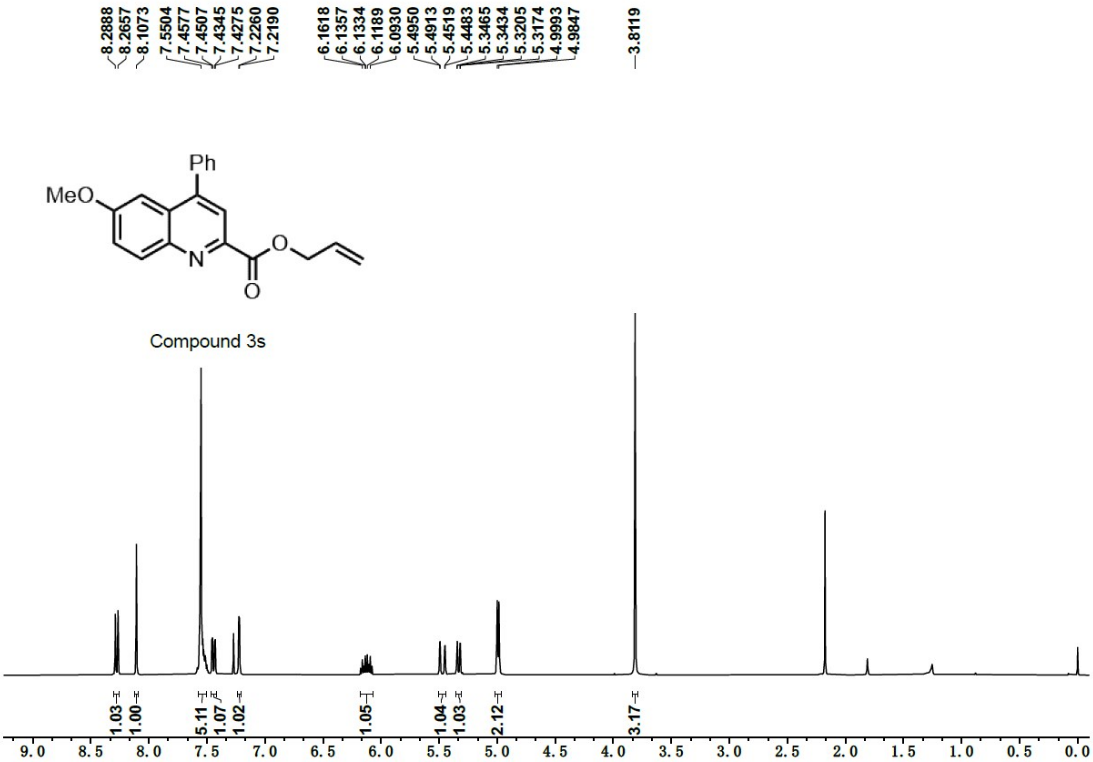
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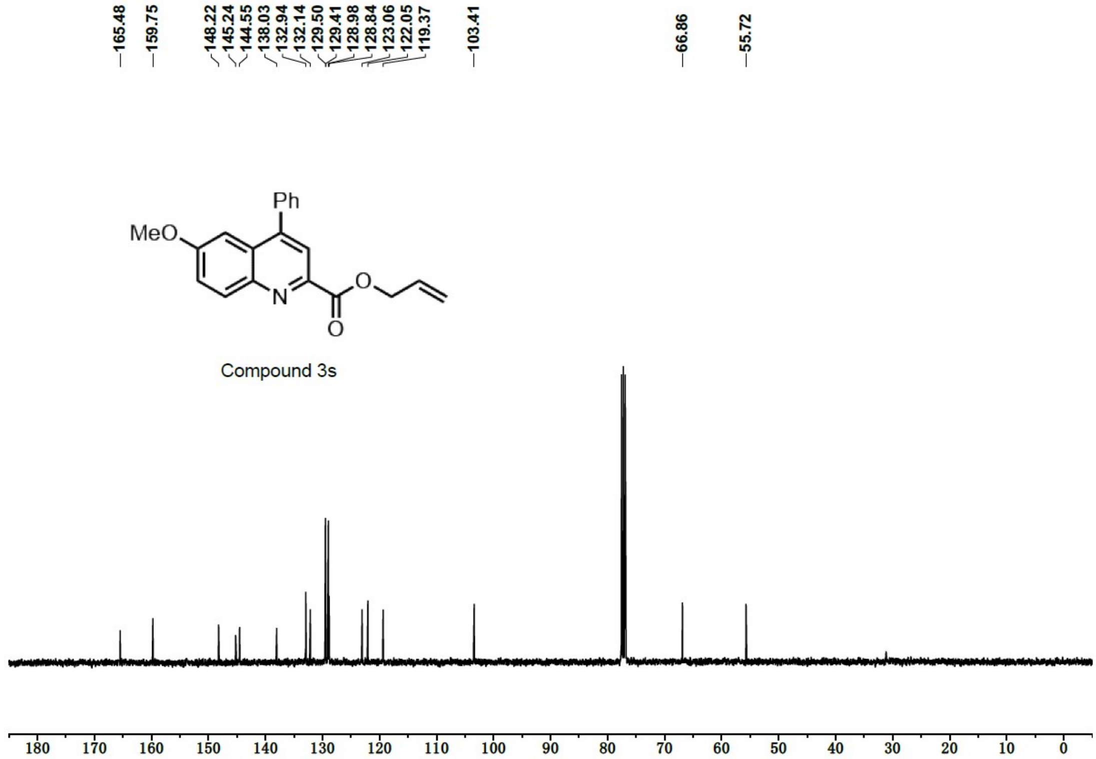
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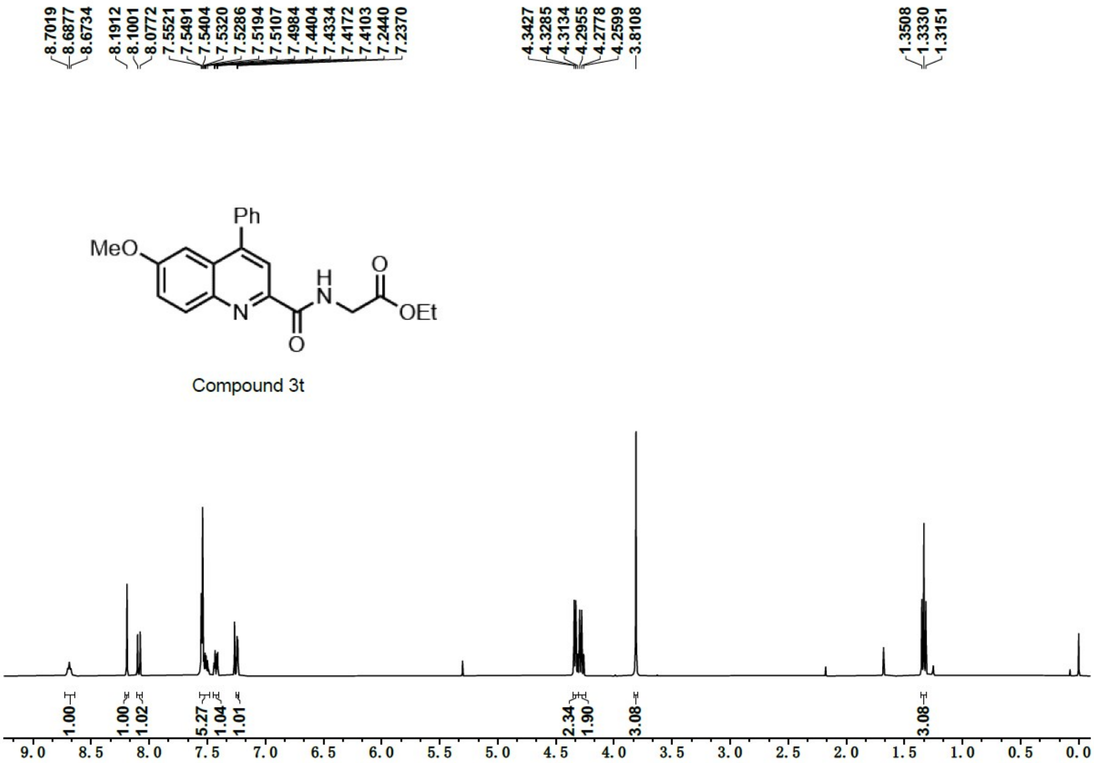

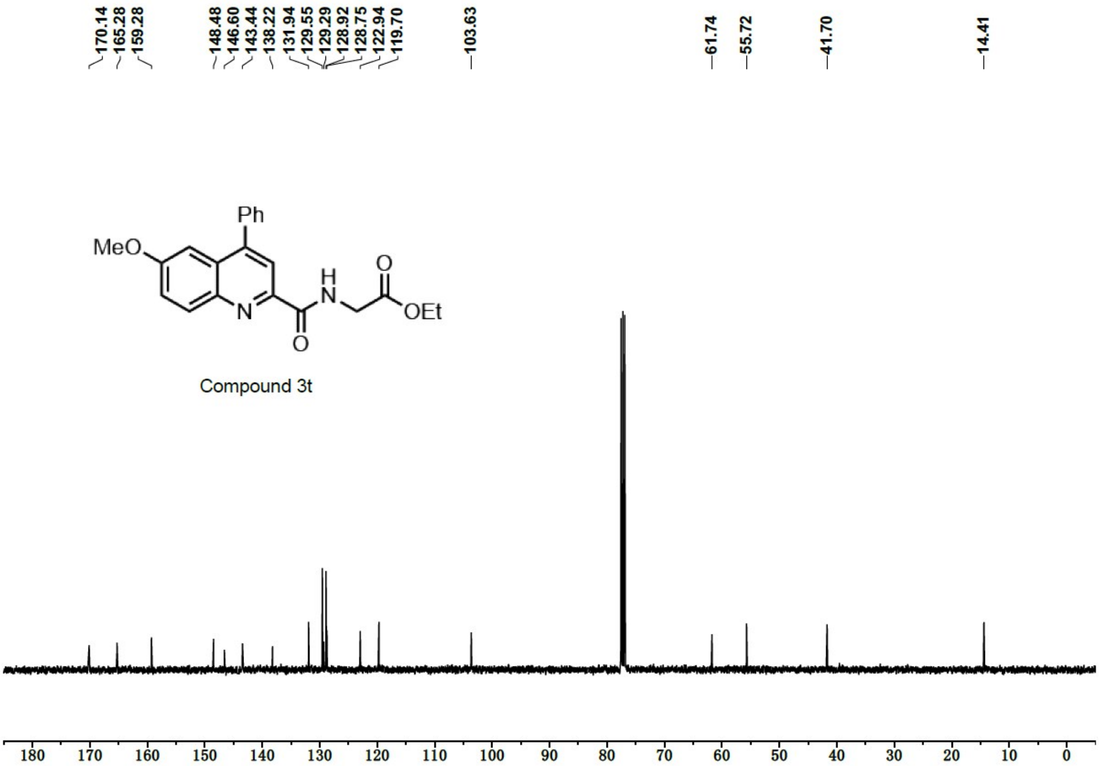

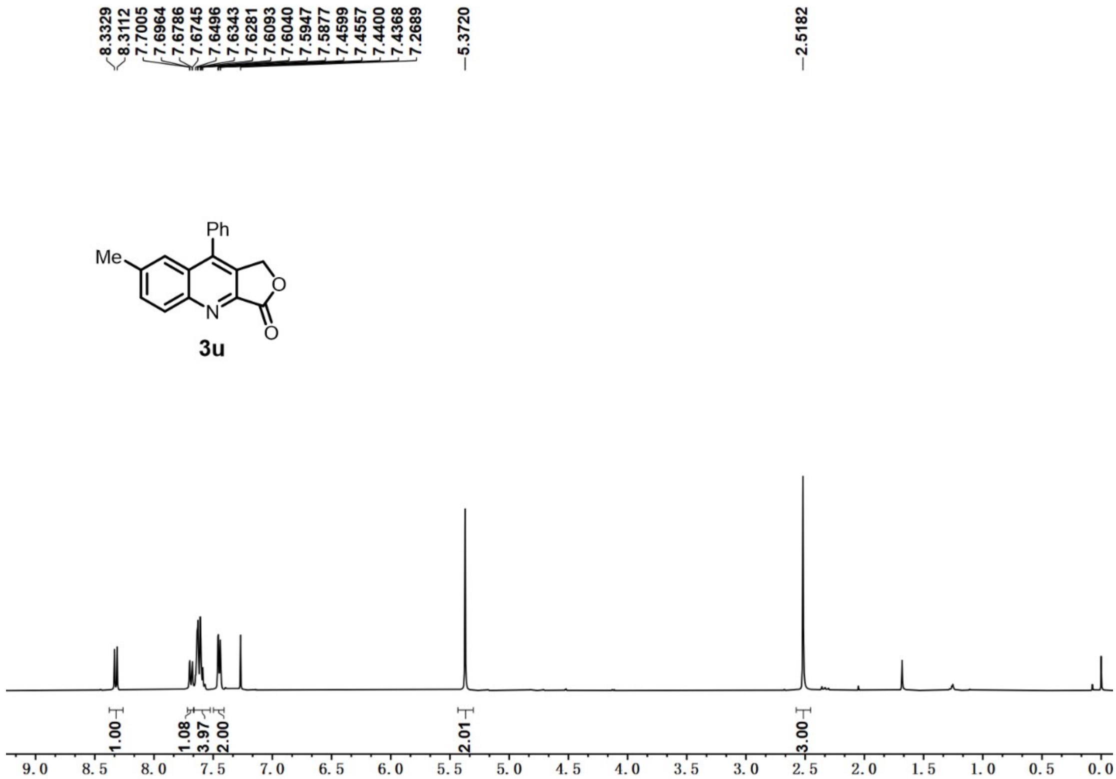
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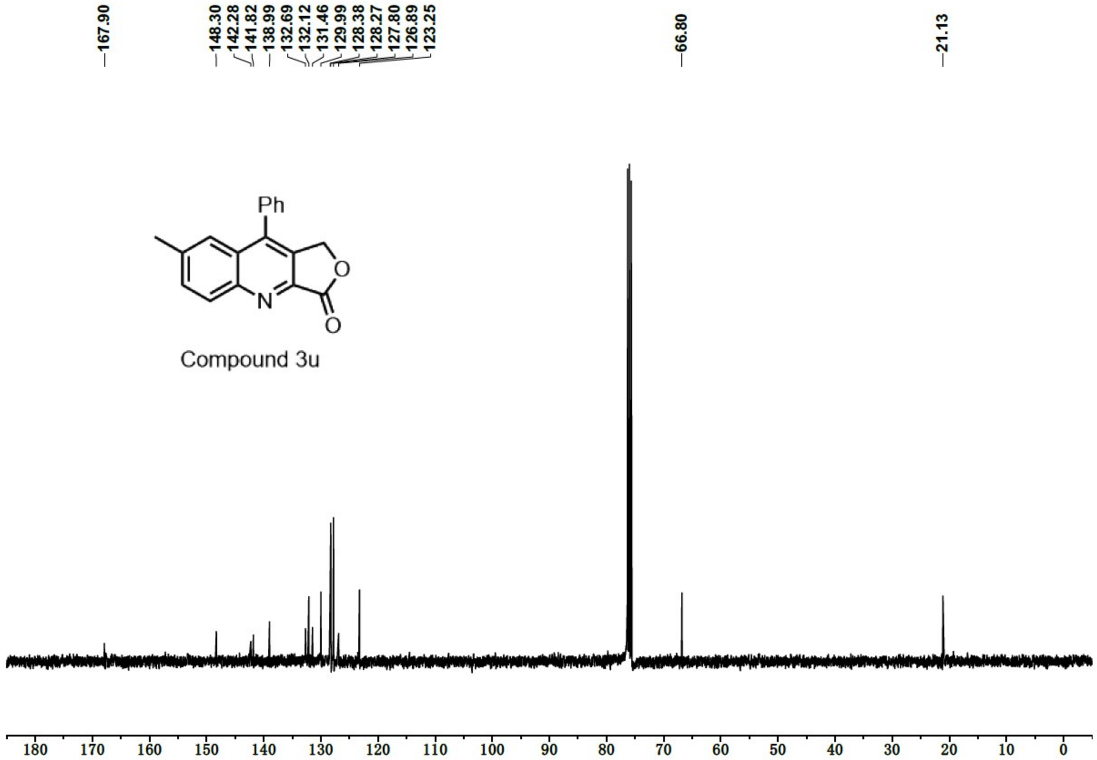
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