

Supporting Information  
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# Supporting Information

## **Redox-Neutral 1,2-Dicarbofunctionalization of Buta-1,3-diene by Merging Decatungstate and Chromium Catalysis**

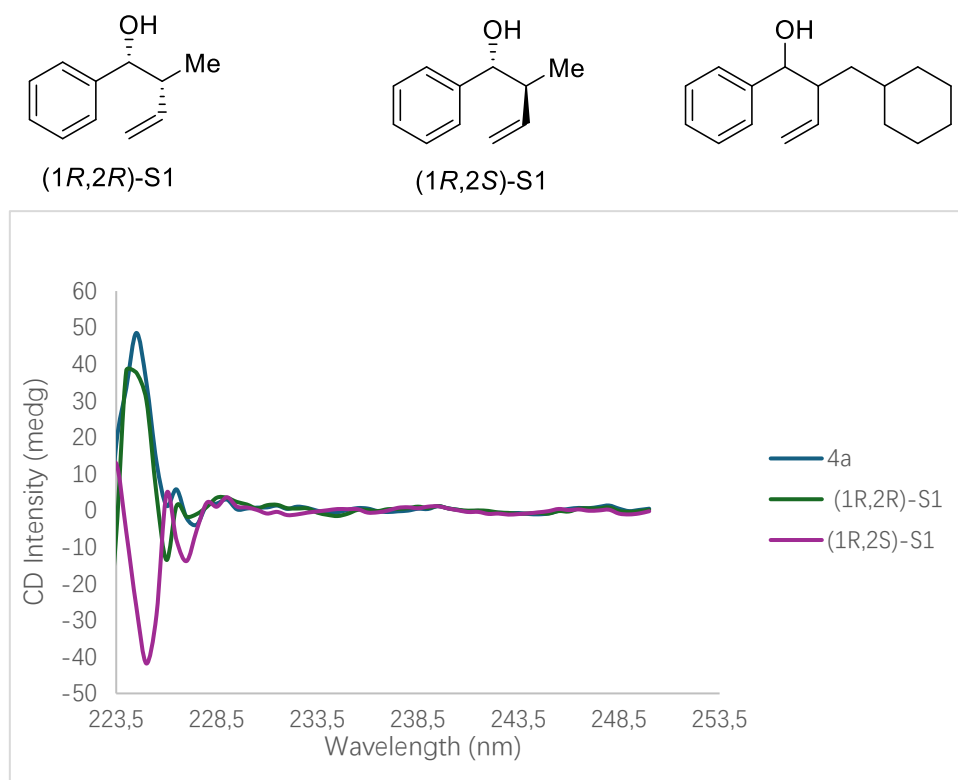
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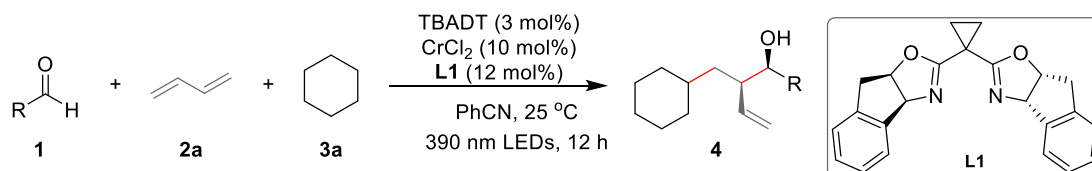
## Circular Dichroic Spectra of (1*R*,2*R*)-**S1**, (1*R*,2*S*)-**S1** and (1*R*,2*R*)-**4a**

The absolute configuration of **4a** and **4r** is assigned by a CD spectra analysis. According to the literature (*J. Am. Chem. Soc.* **2010**, *132*, 11884),<sup>1</sup> chiral homoallylic alcohols (1*R*,2*R*)-**S1** and (1*R*,2*S*)-**S1** are synthesized with high ee. By comparing the CD spectrum, the CD intensity of **4a** closely resembles that of (1*R*,2*R*)-**S1**. Based on this similarity in CD profiles, the absolute configuration of **4a** is assigned as (1*R*,2*R*). The absolute configuration of **4r** is assigned by analogy.



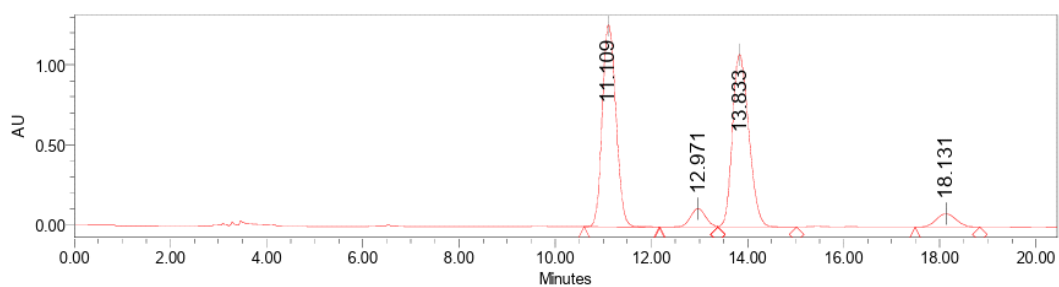
CD spectra of (1*R*,2*R*)-**S1**, (1*R*,2*S*)-**S1** and **4a**

## Asymmetric Synthesis of **4a** and **4r**



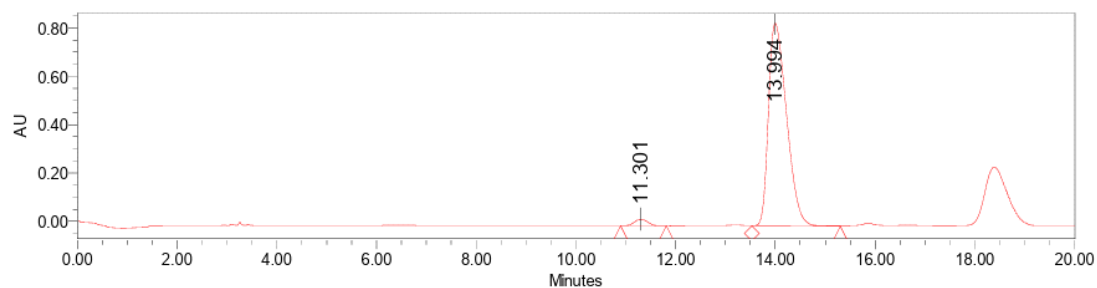
To a N<sub>2</sub>-purged 10 mL Schlenk tube were added **2a** (2.1 M in hexane, 3 equiv, 0.60 mmol), **3a** (8 mmol), aromatic aldehyde **1** (0.2 mmol), TBADT (0.006 mmol), CrCl<sub>2</sub> (0.020 mmol), **L1** (0.024 mmol), PhCN (2.0 mL) and a stir bar. The mixture was stirred at 25 °C for 24 h under 10 W 390 nm LEDs. The diastereoselectivity were determined by <sup>1</sup>H NMR analysis of the crude reaction mixture. The solvent was removed under vacuum and the residue was purified by flash chromatography (SiO<sub>2</sub>, petroleum ether/ethyl acetate/ = 10/1) to provide the desired product **4**.

### HPLC data for compound 4a



Peak Results

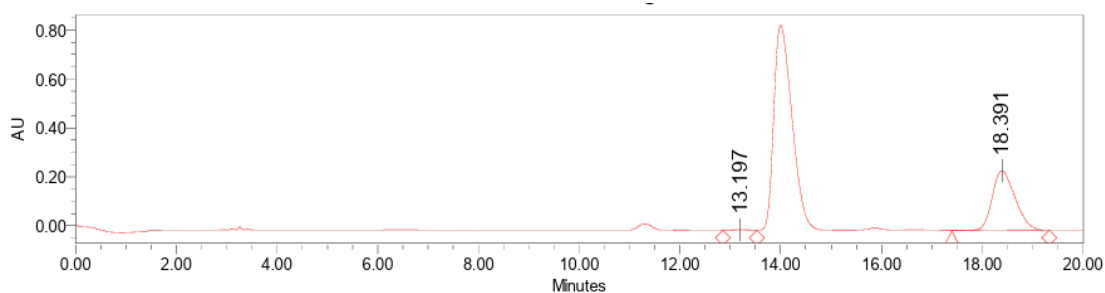
Name	RT	Area	Height	% Area
1	11.109	24903382	1261867	44.95
2	12.971	2518327	115432	4.55
3	13.833	25488056	1079717	46.01
4	18.131	2489239	84585	4.49



Peak Results

Name	RT	Area	Height	% Area
1	11.301	533676	27866	2.46
2	13.994	21174628	838218	97.54

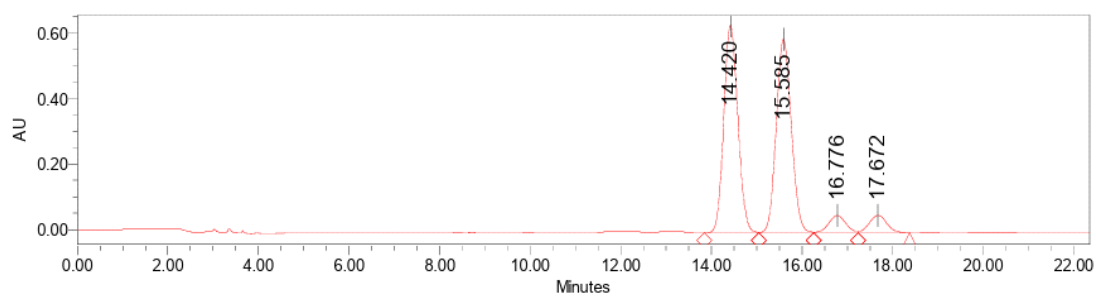
### HPLC data for another diastereomer of 4a



Peak Results

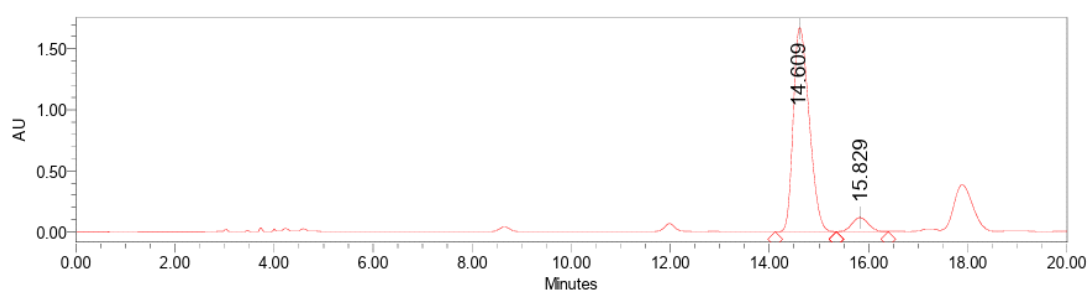
Name	RT	Area	Height	% Area
1	13.197	101227	4792	1.29
2	18.391	7726292	244230	98.71

### HPLC data for compound 4r



**Peak Results**

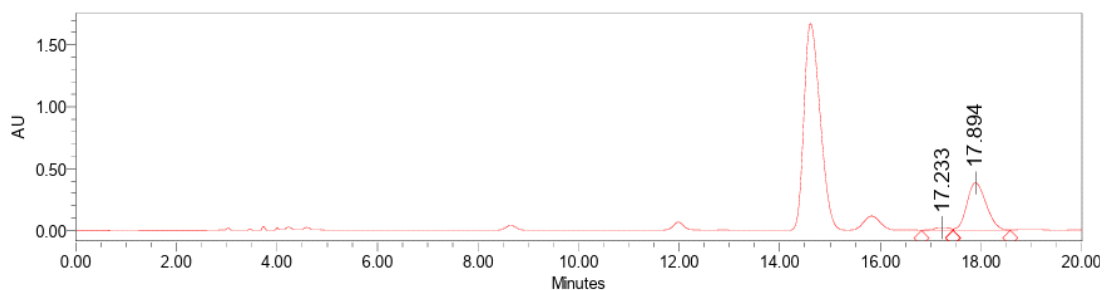
Name	RT	Area	Height	% Area
1	14.420	13630546	632148	45.44
2	15.585	13654075	589657	45.52
3	16.776	1339826	51905	4.47
4	17.672	1373977	52434	4.58



**Peak Results**

Name	RT	Area	Height	% Area
1	14.609	37548496	1671472	93.21
2	15.829	2735547	116327	6.79

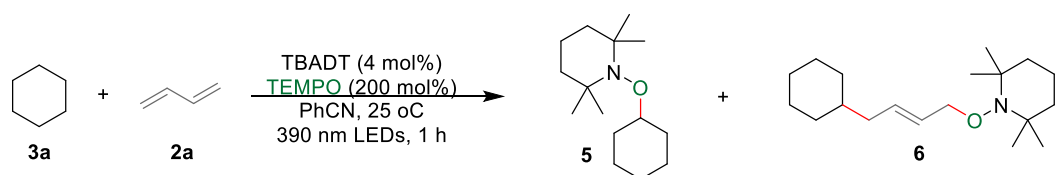
### HPLC data for another diastereomer of 4r



**Peak Results**

Name	RT	Area	Height	% Area
1	17.233	499313	21794	4.61
2	17.894	10327712	385124	95.39

### Radical trapping experiment



HRMS(ESI,m/z) calculated for C<sub>15</sub>H<sub>29</sub>NO (M+H<sup>+</sup>): 240.2332  
found:240.2330

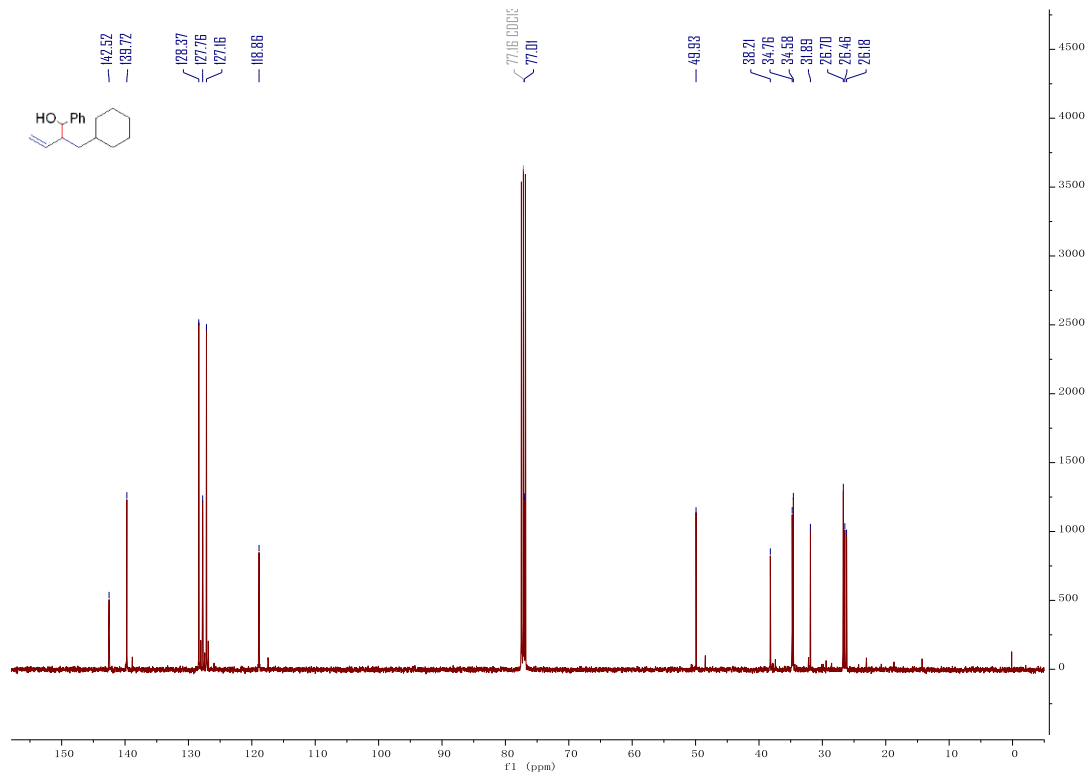
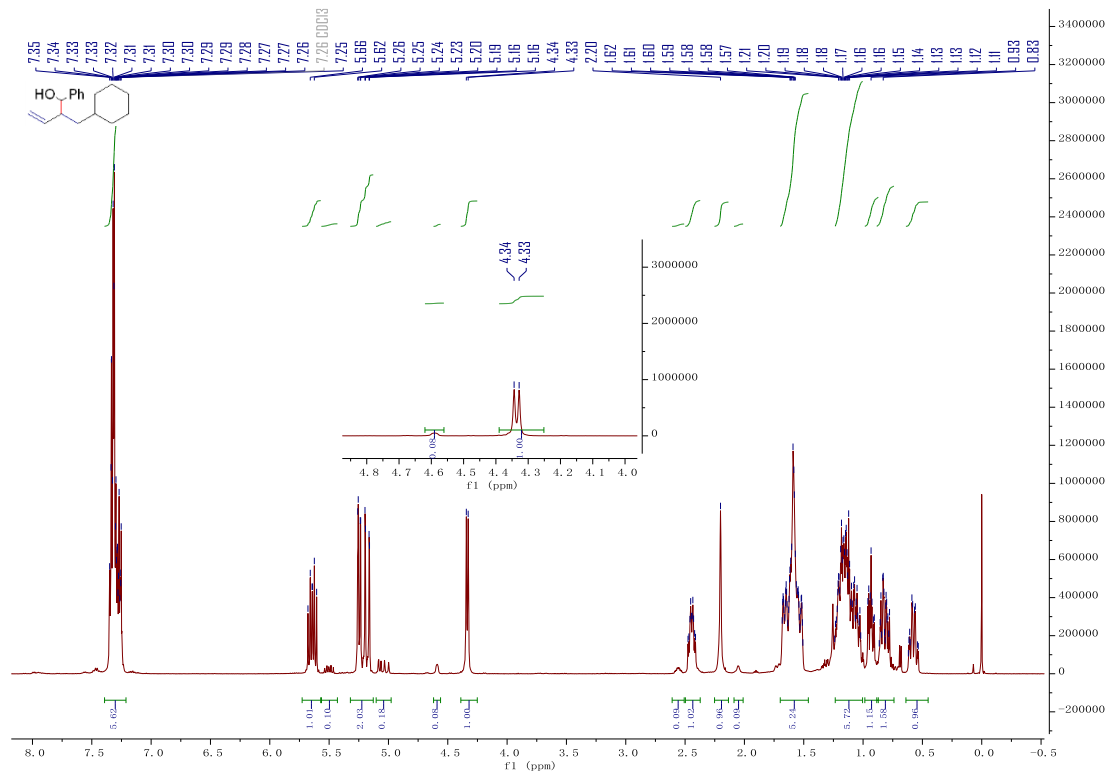
HRMS(ESI,m/z) calculated for C<sub>19</sub>H<sub>35</sub>NO (M+H<sup>+</sup>): 294.2791  
found:294.2788

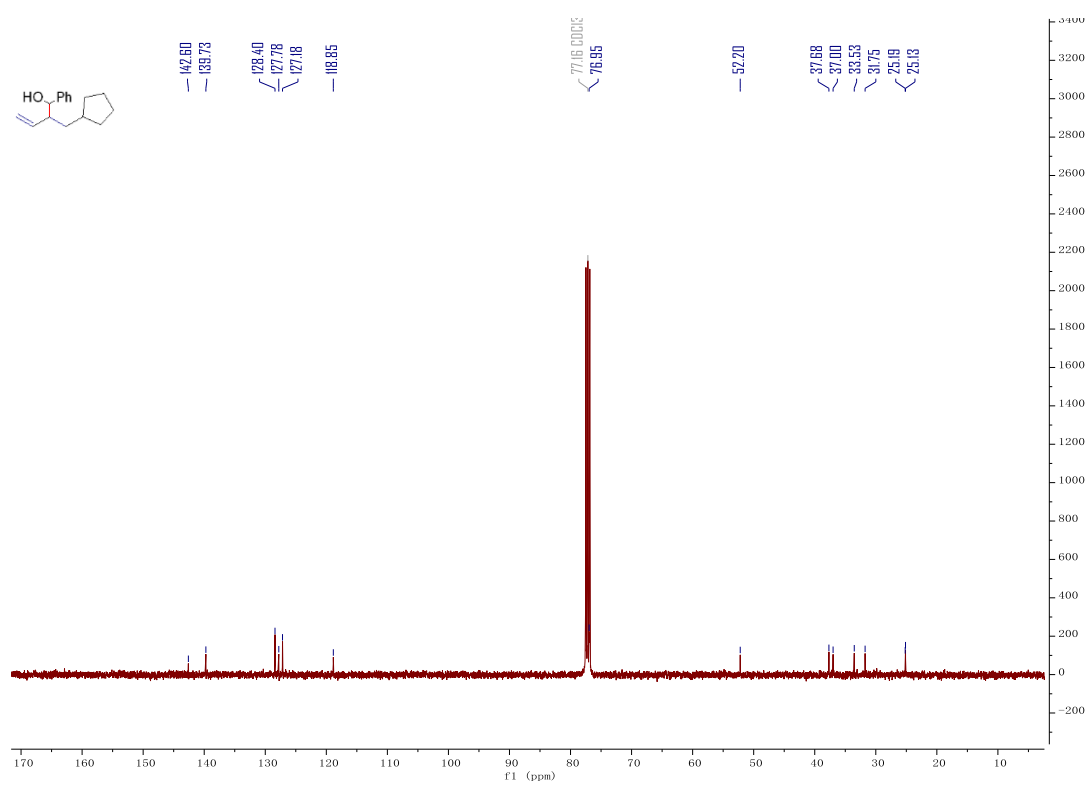
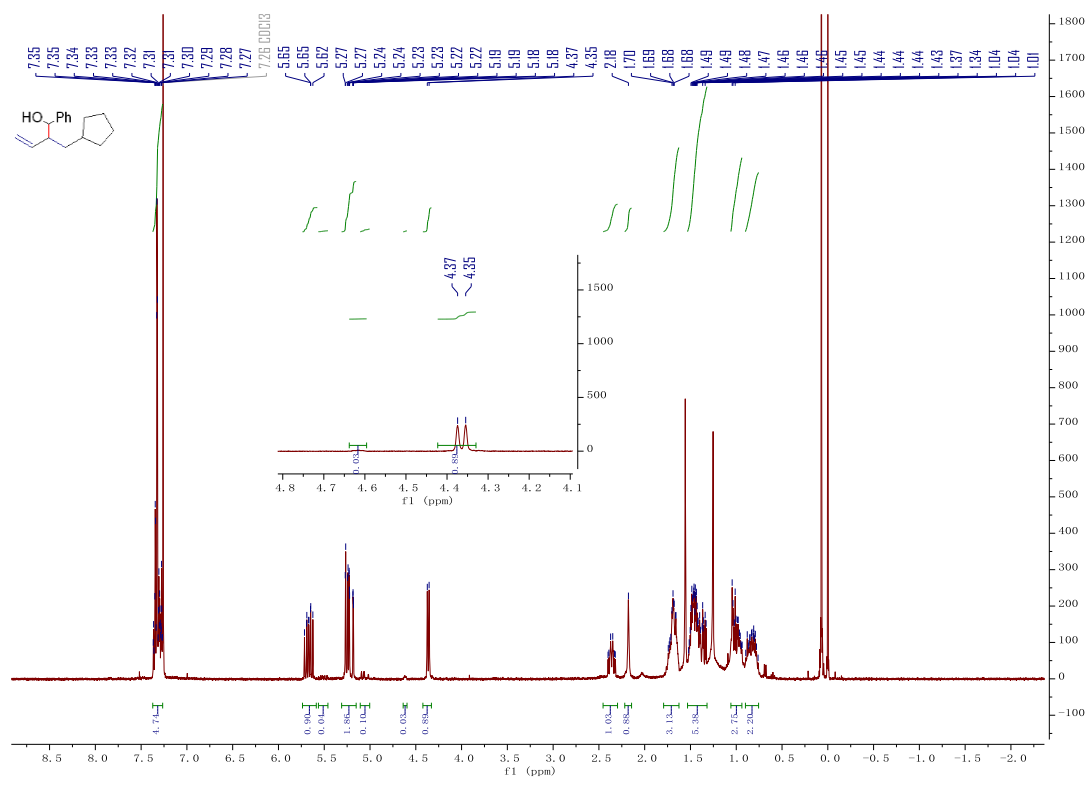
To a N<sub>2</sub>-purged 10 mL Schlenk tube were added TBADT (26.4 mg, 4 mol%), **3a** (0.85 mL, 40 equiv), **2a** (0.1 mL (2.1 M in hexane), 0.2 mmol), TEMPO (62.4 mg, 2 equiv), PhCN (2.0 mL) and a stir bar. The mixture was stirred at 25 °C for 1 h under 10 W 390 nm LEDs. Then the mixture was analyzed by high-resolution mass spectrometry (HRMS).

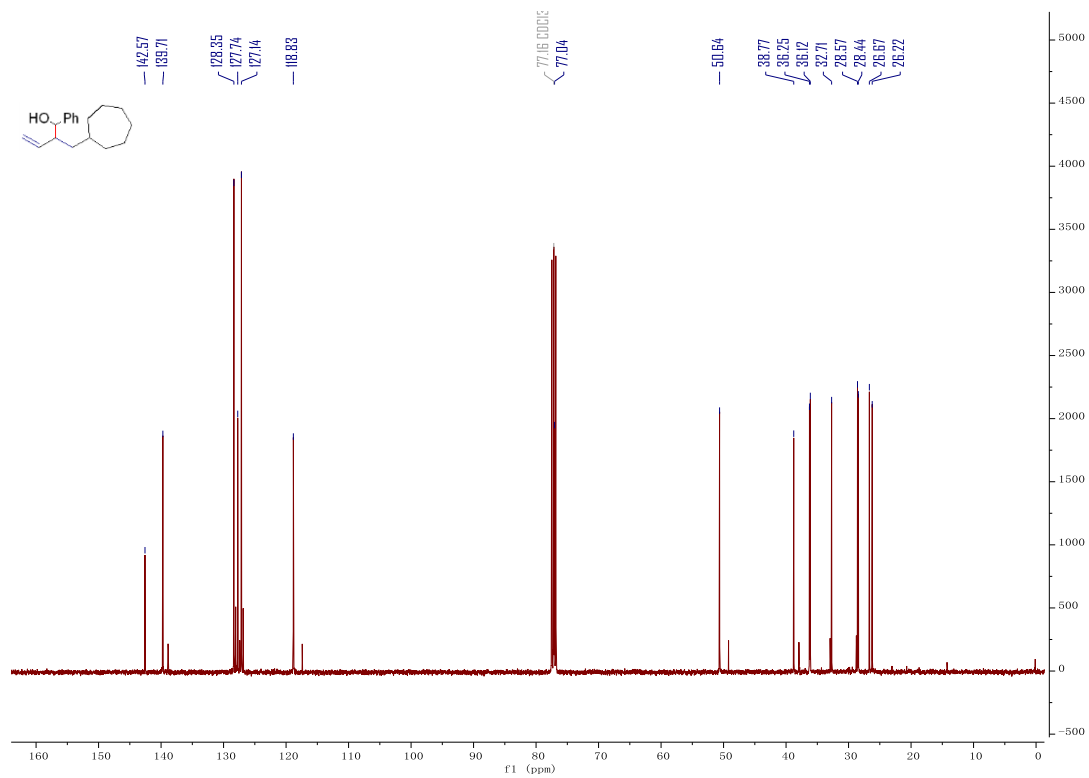
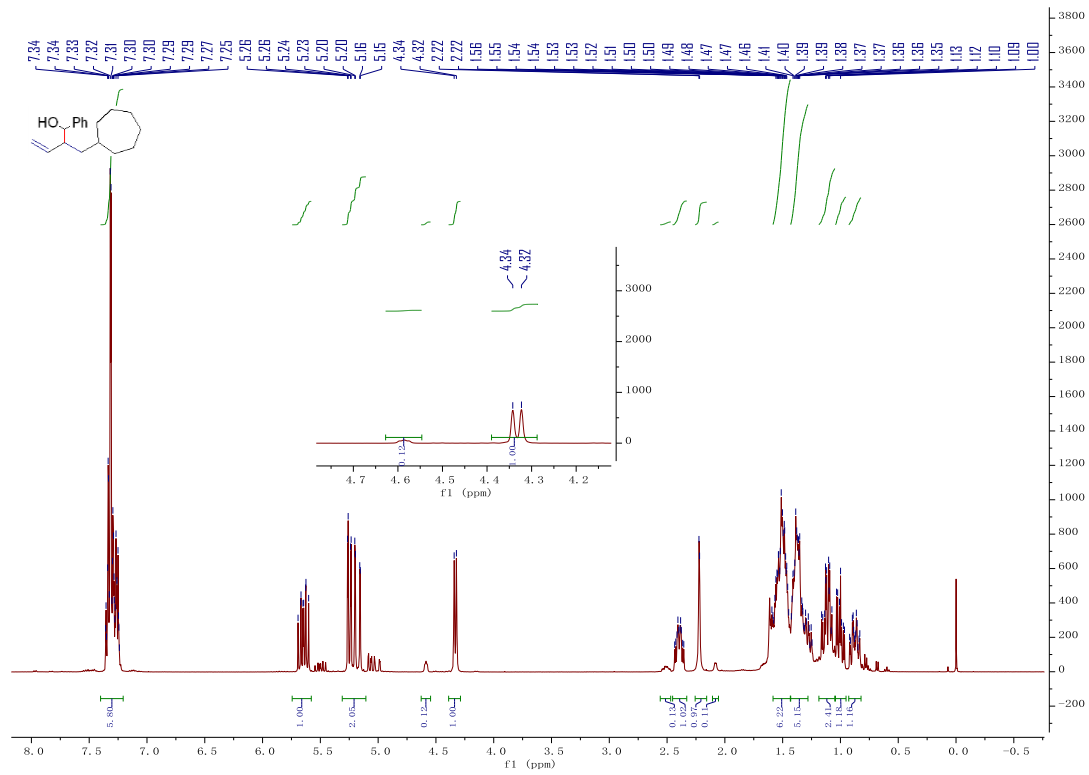
#### References

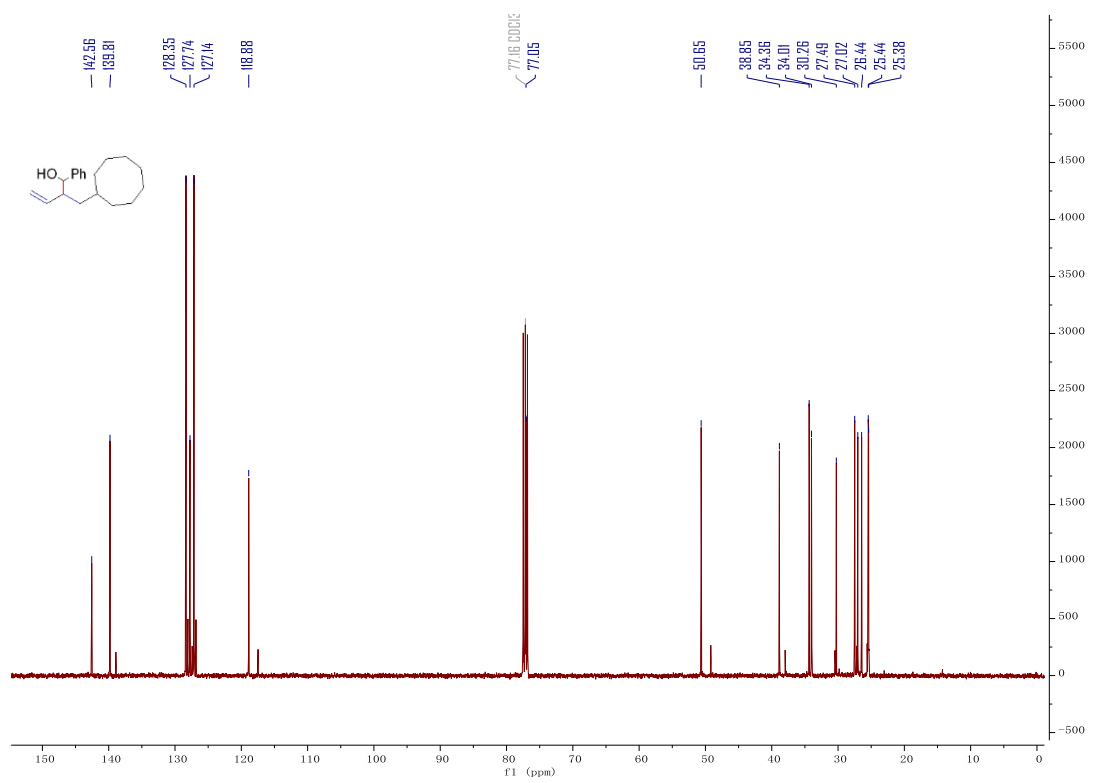
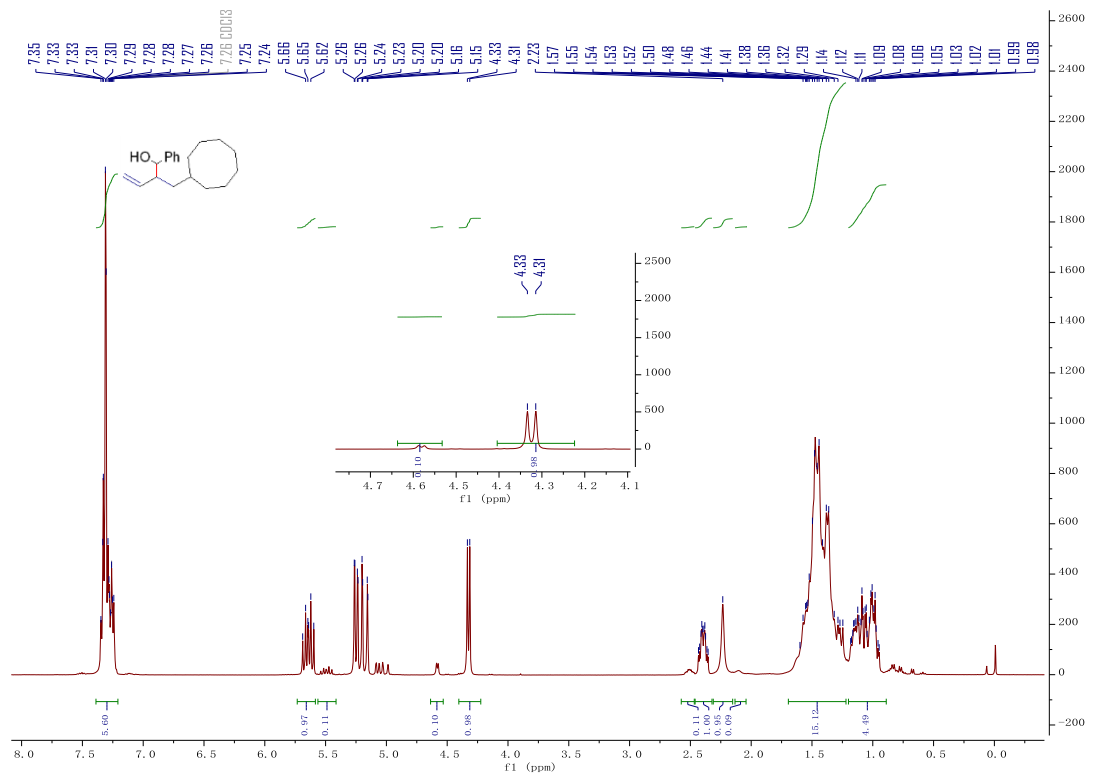
1. Jain, P.; Antilla, J. C. *J. Am. Chem. Soc.* **2010**, 132, 11884-11886.

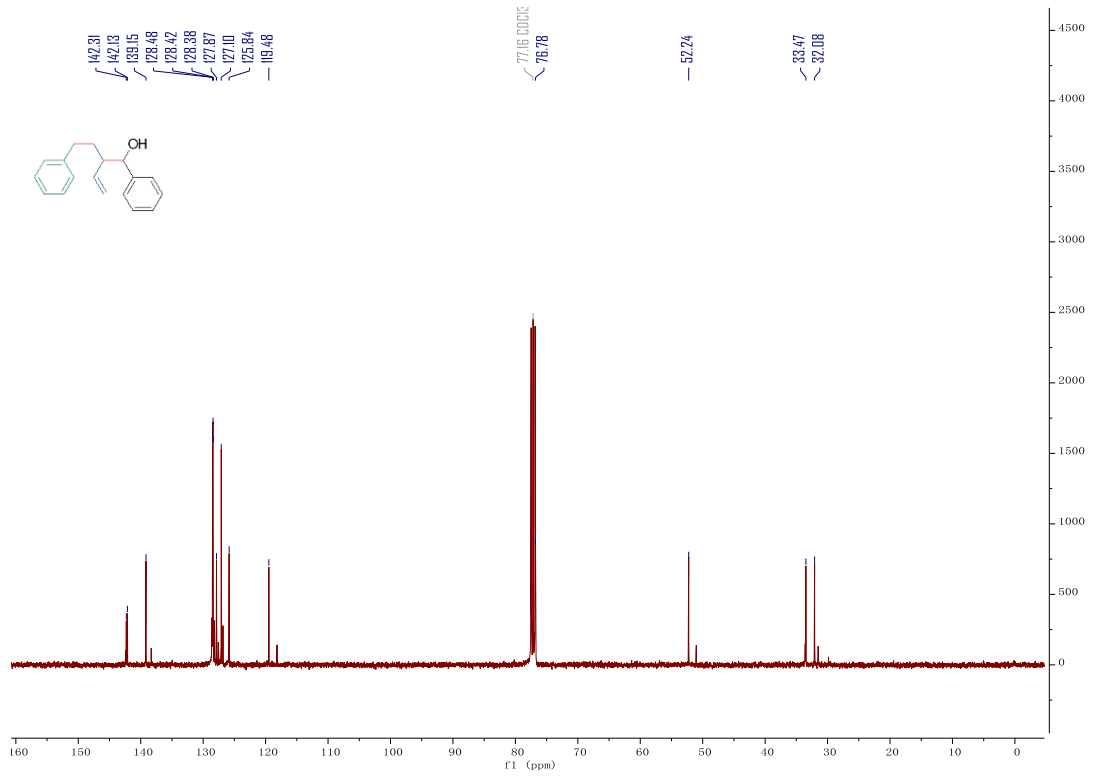
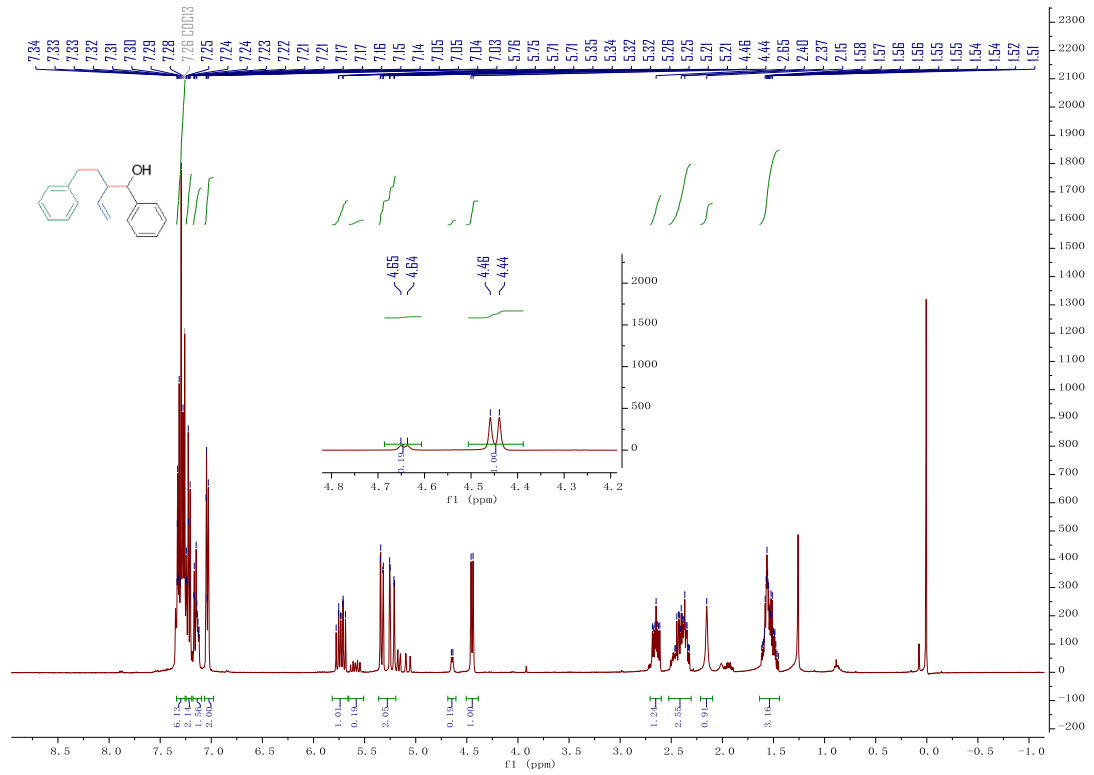
# Spectra



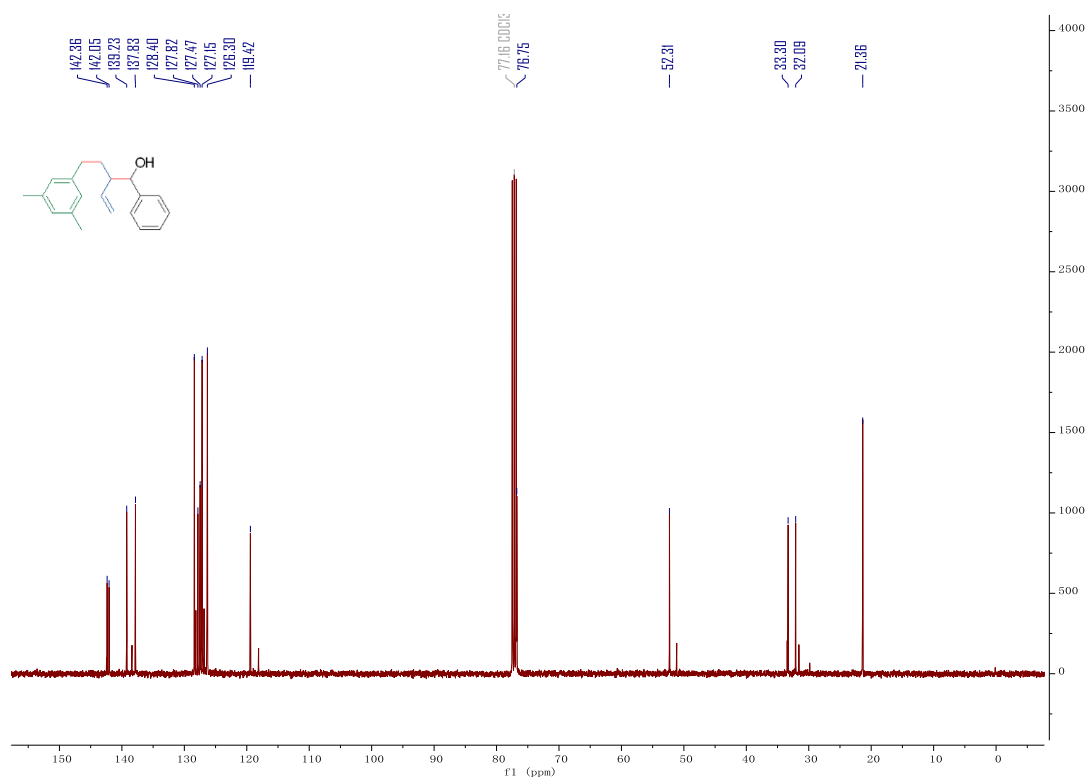
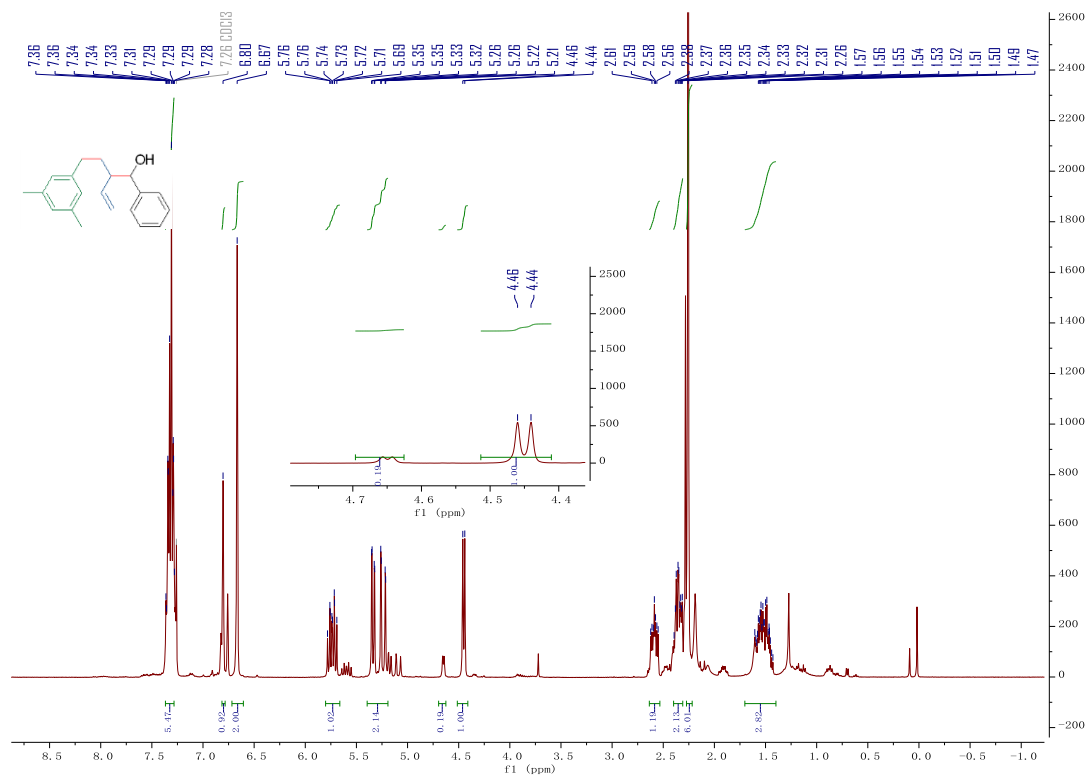


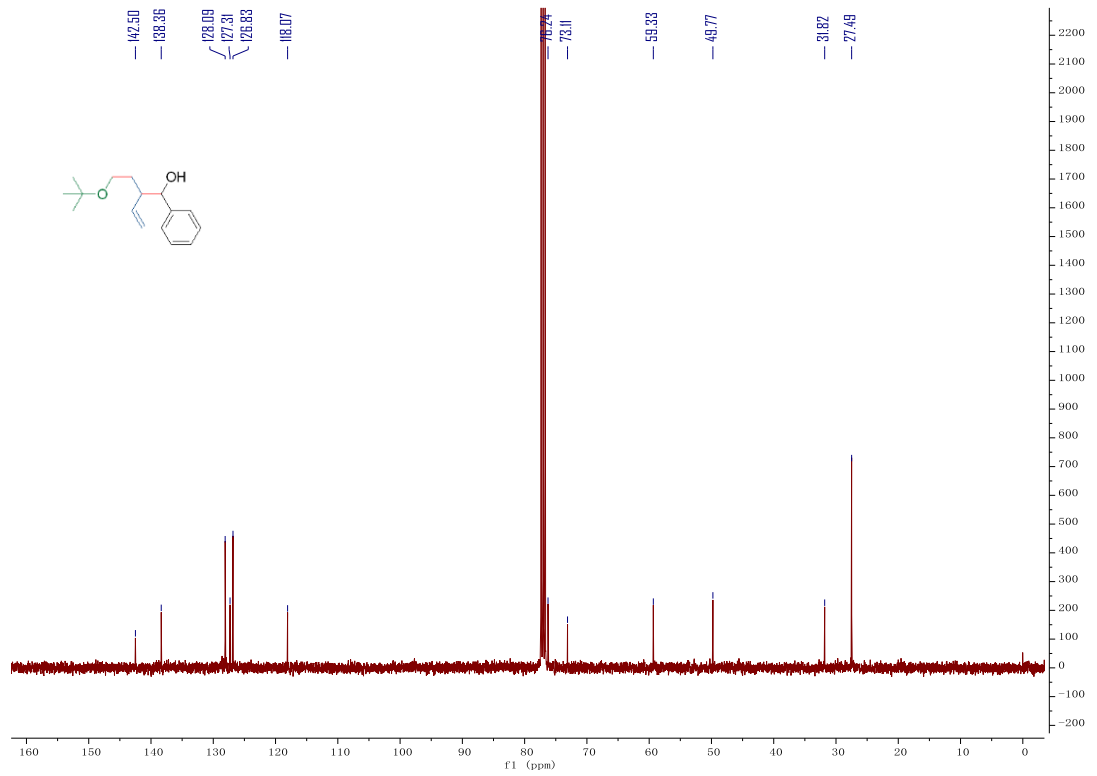
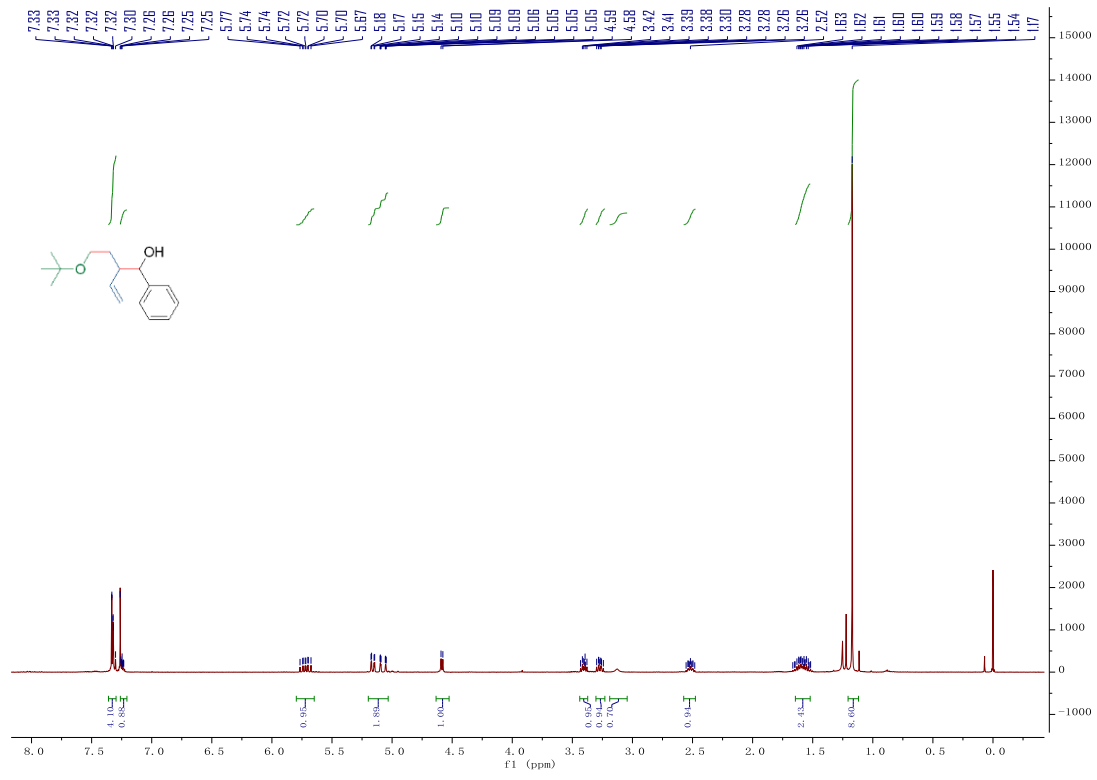


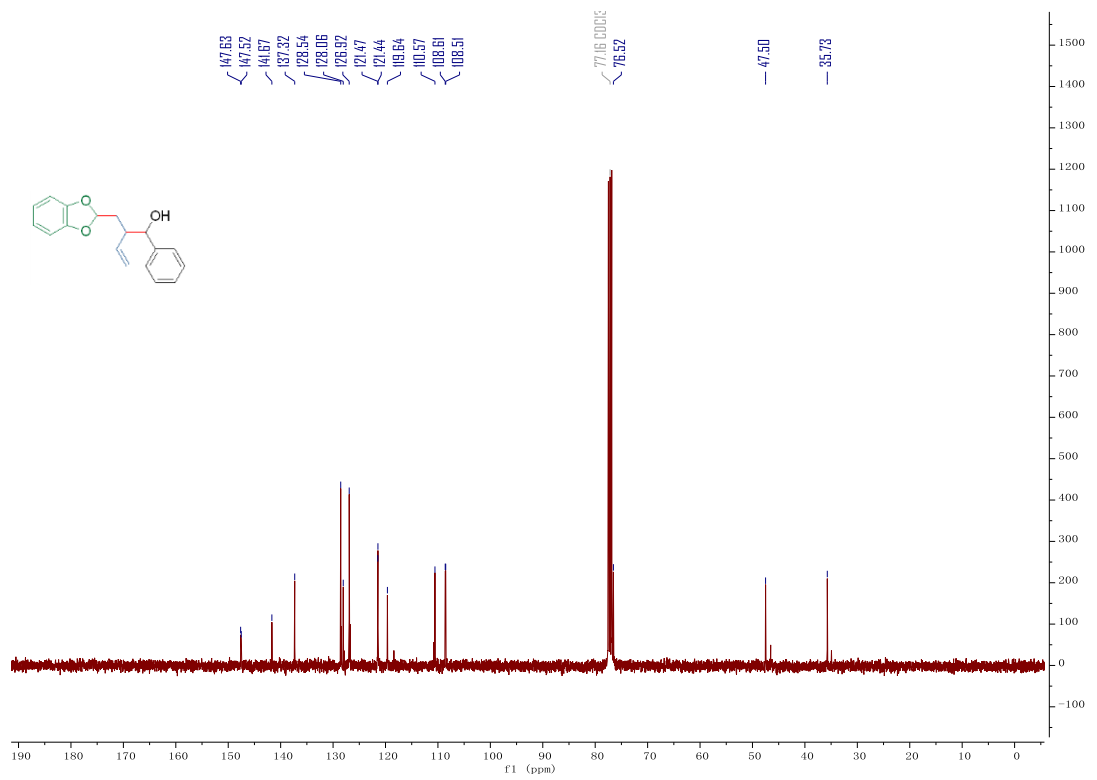
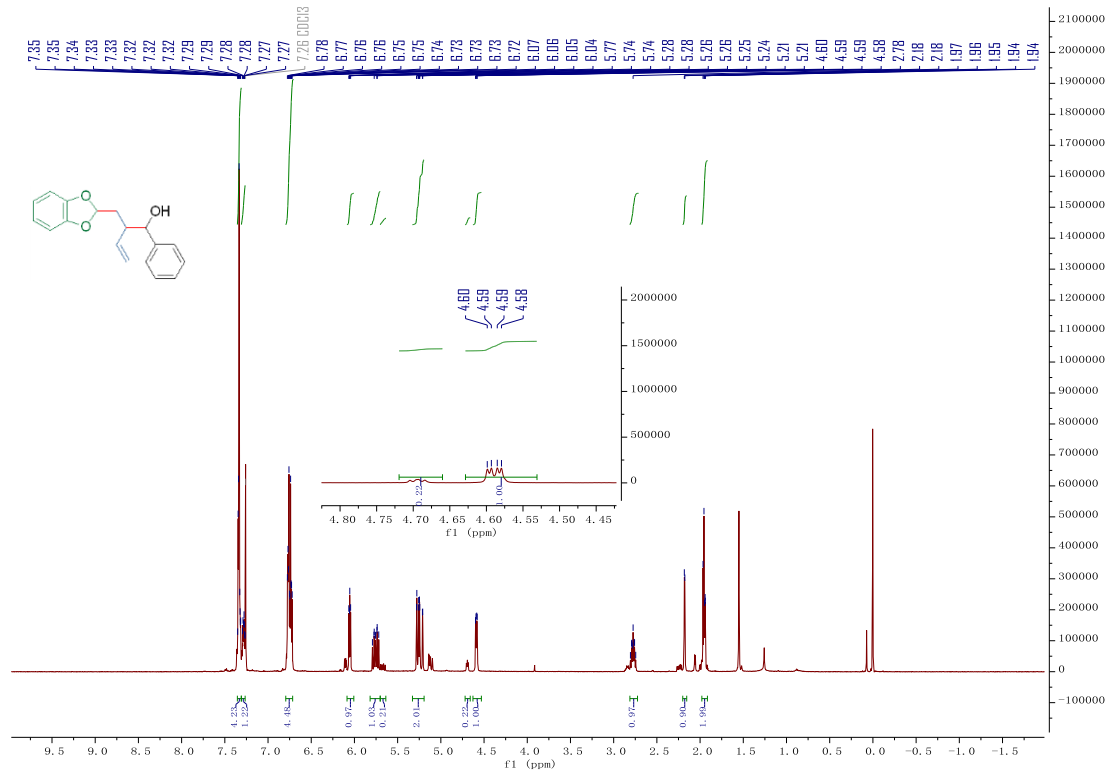


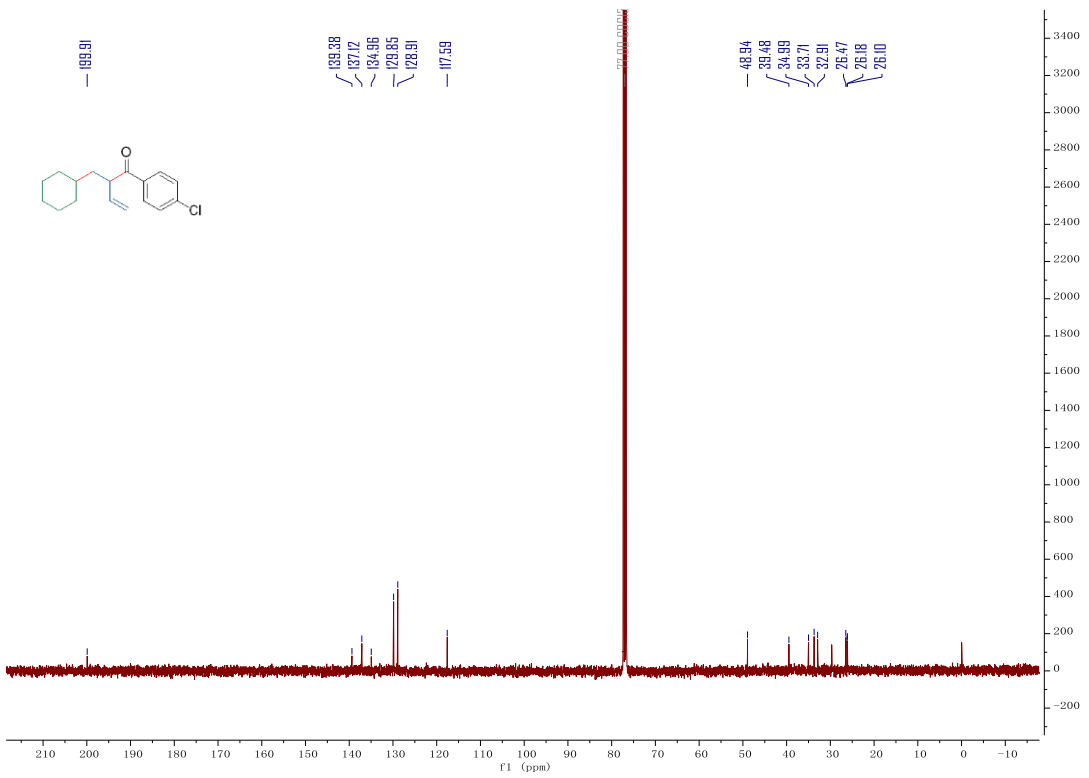
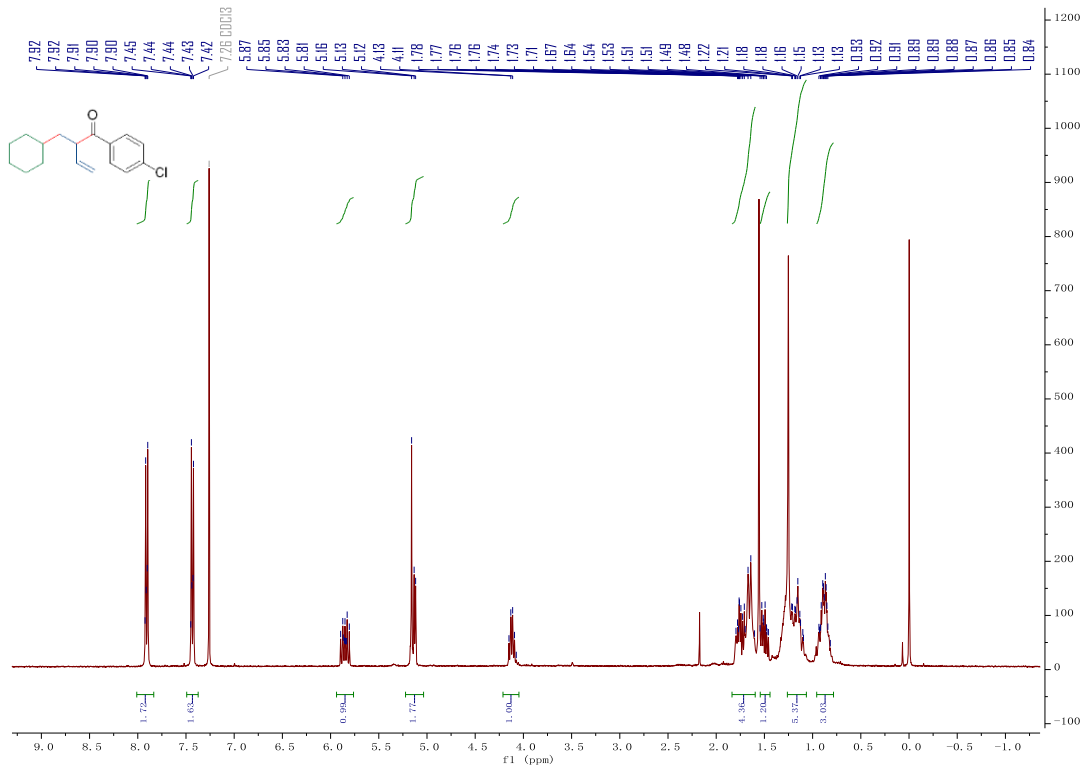


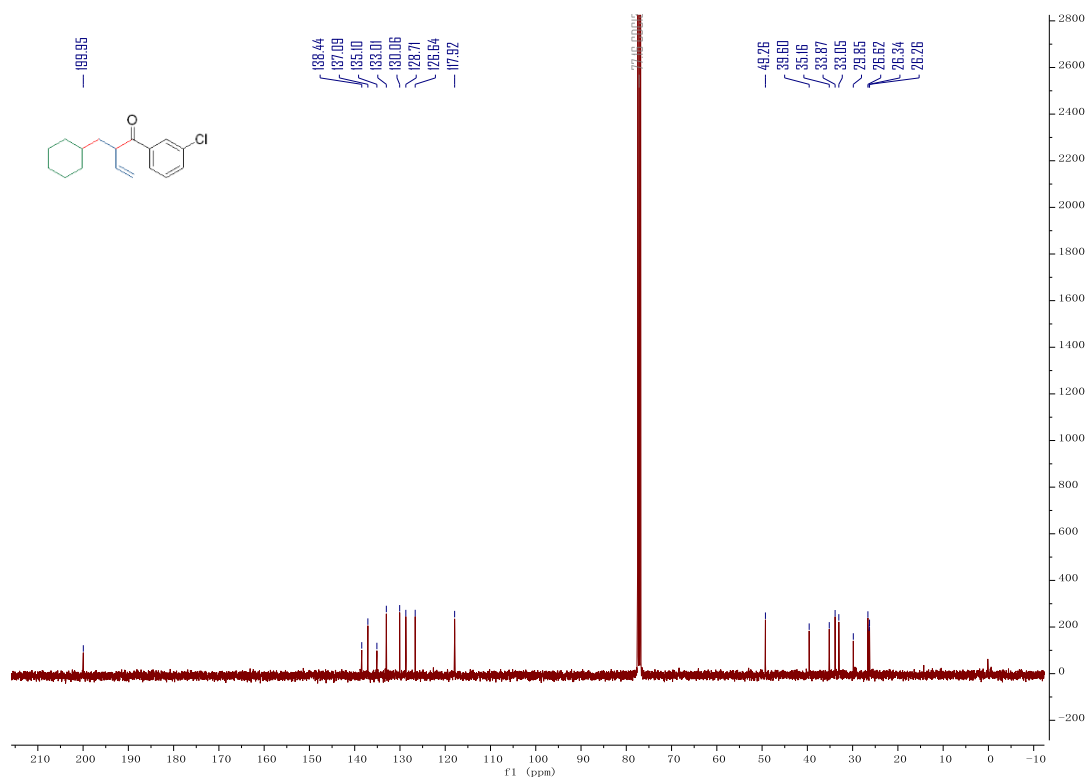
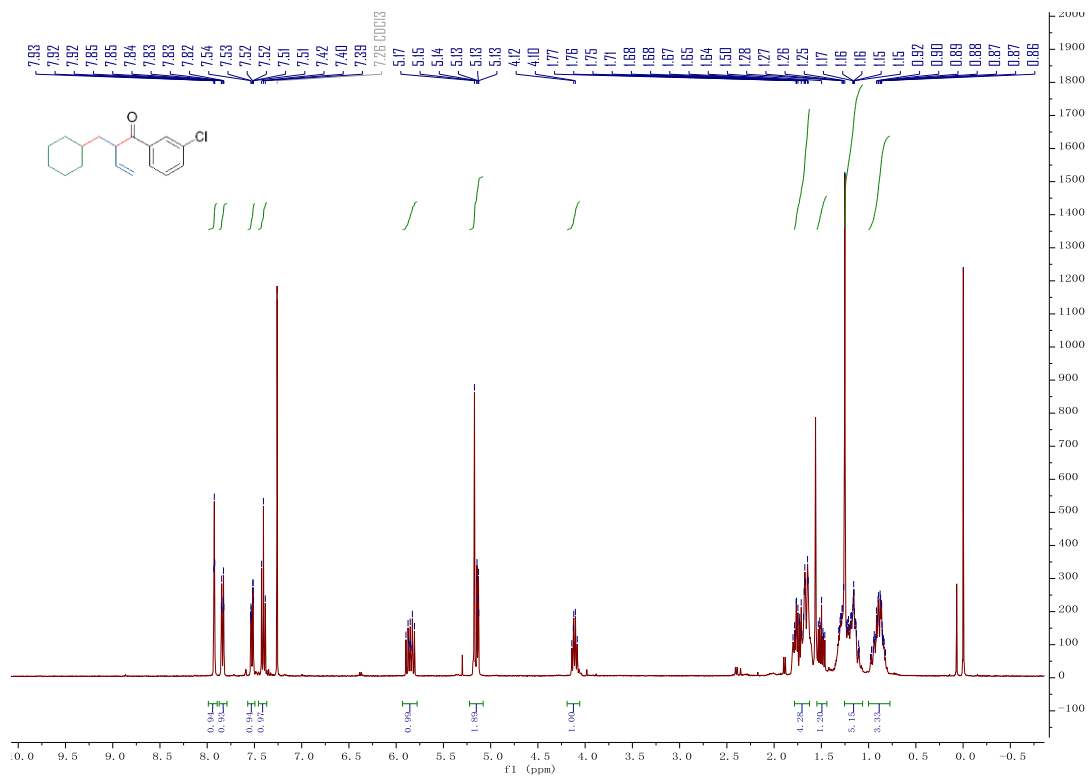


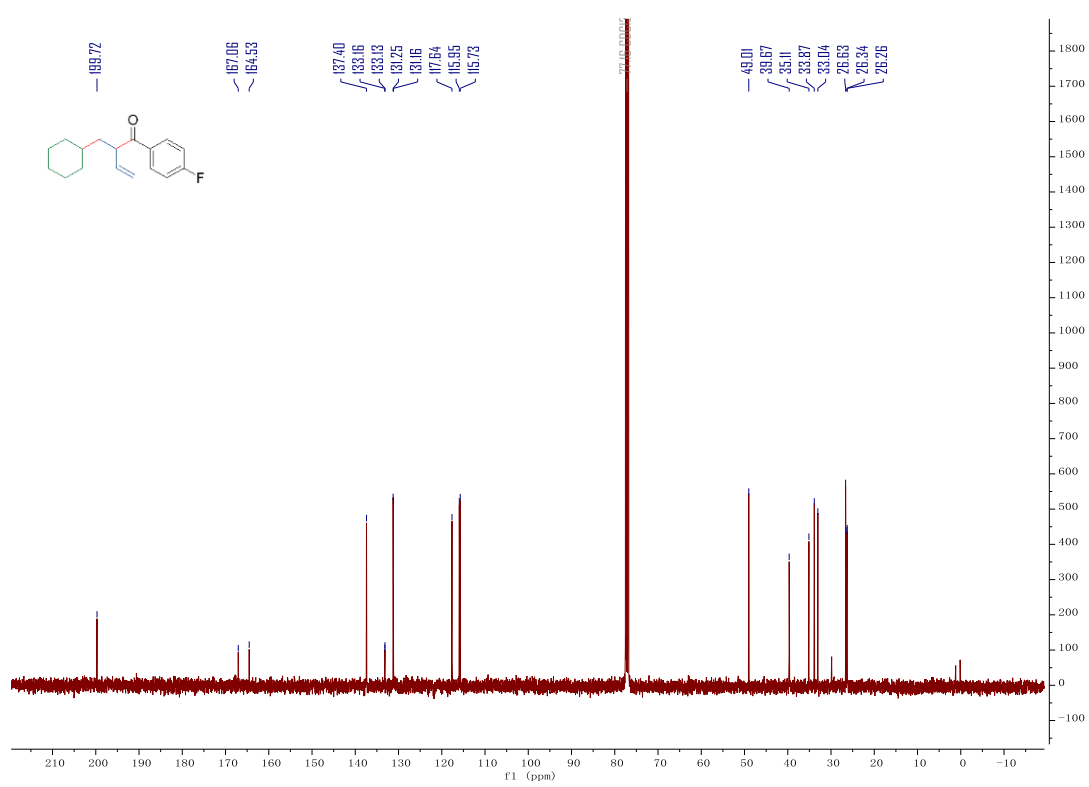
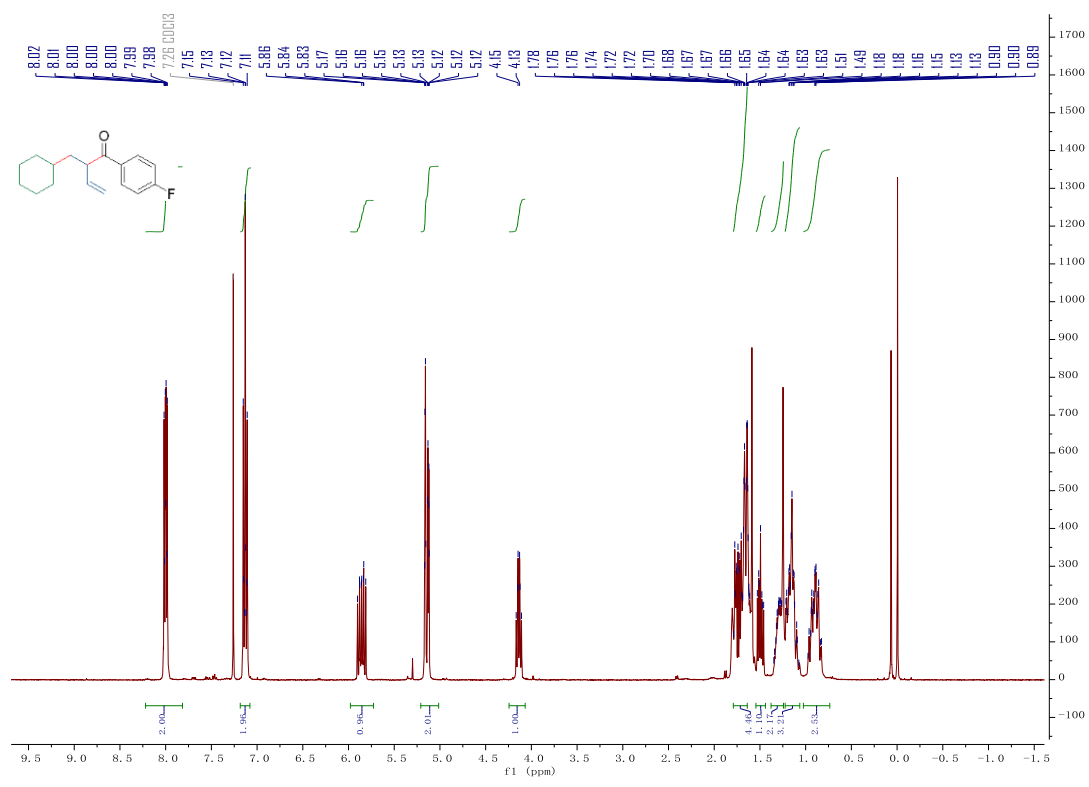




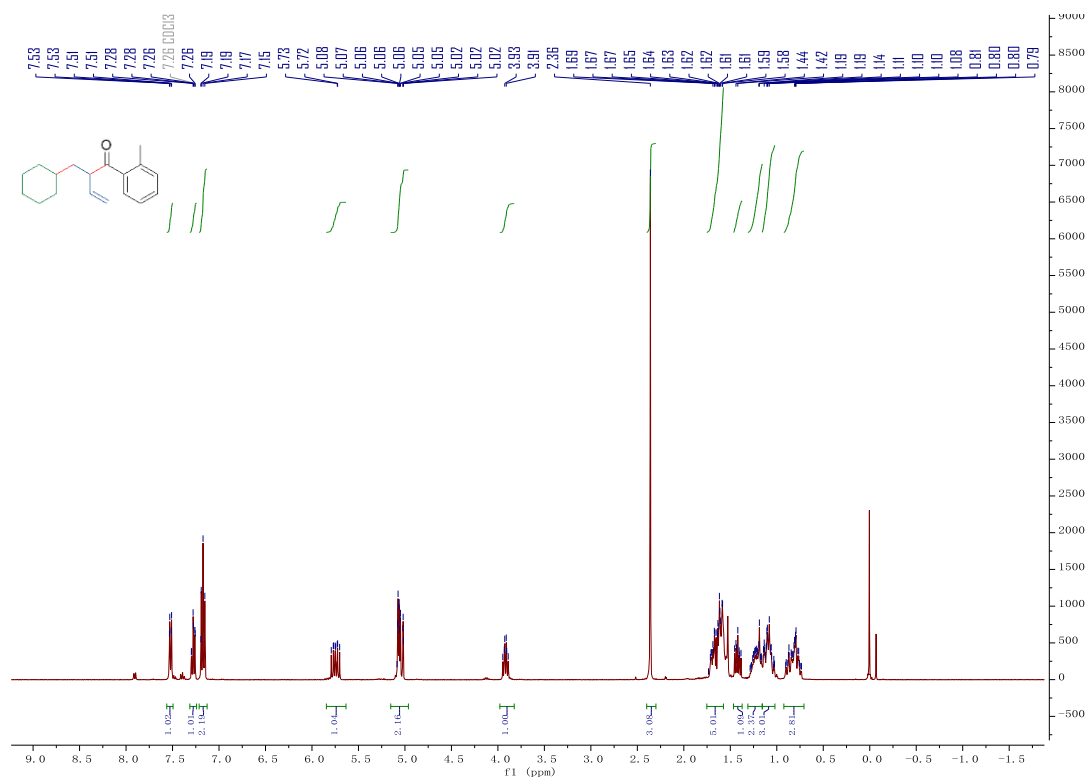
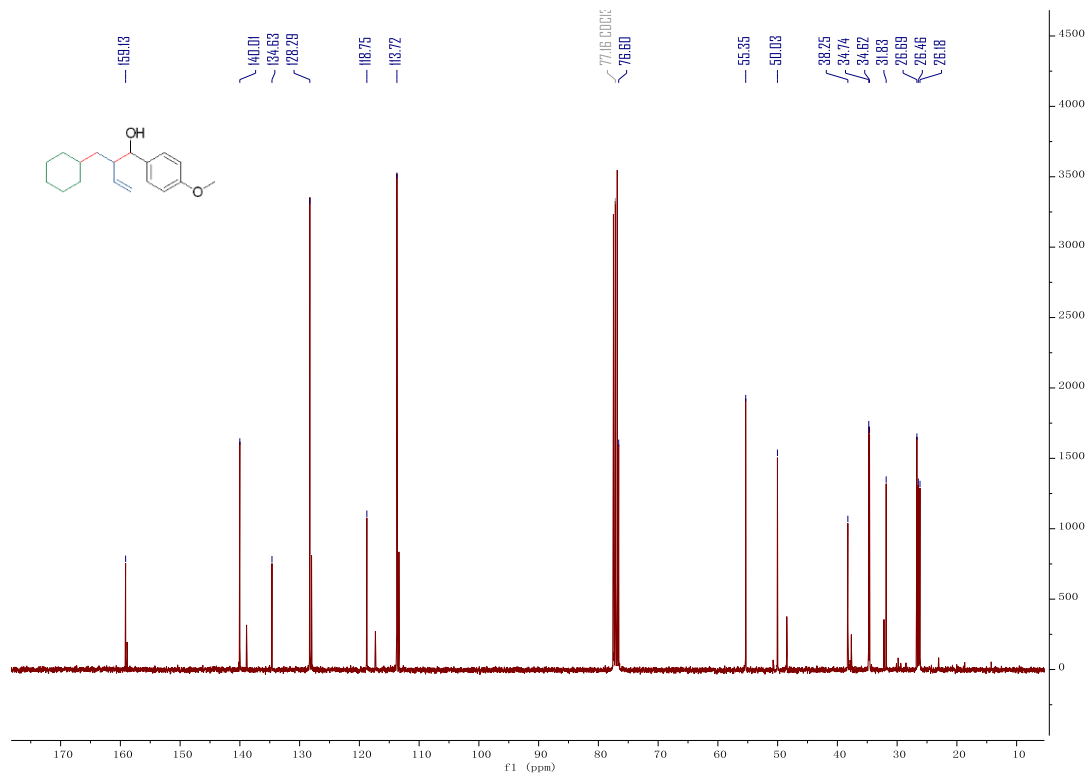


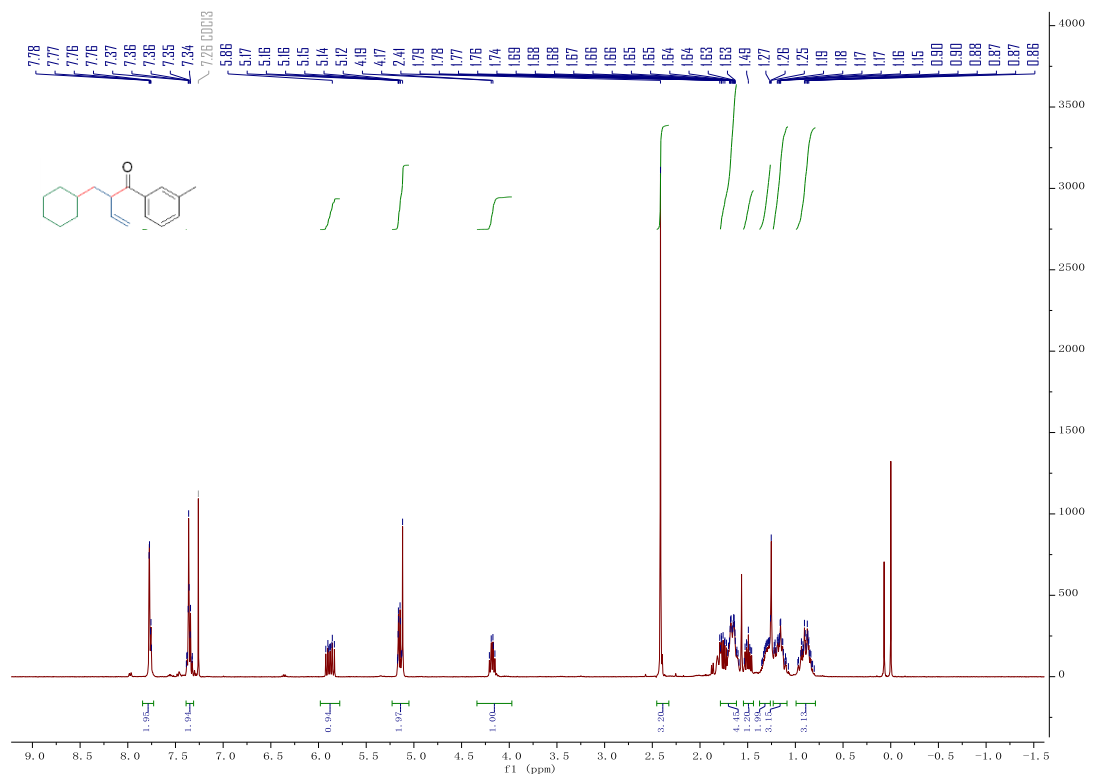
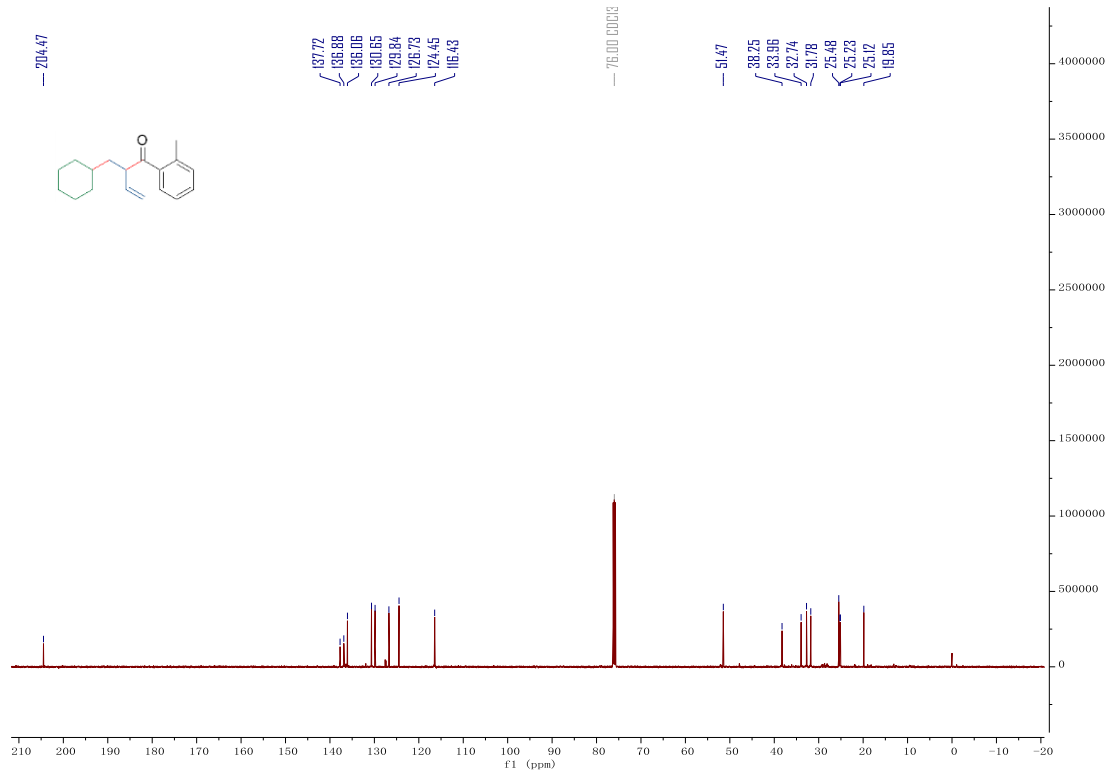






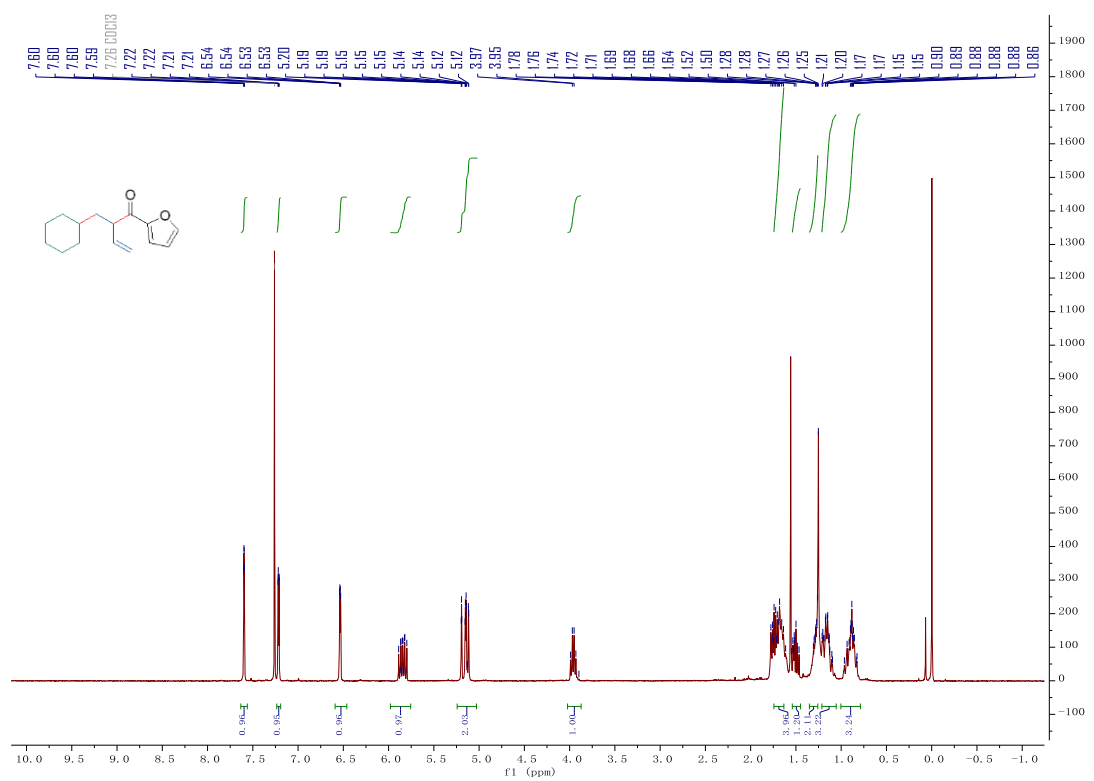
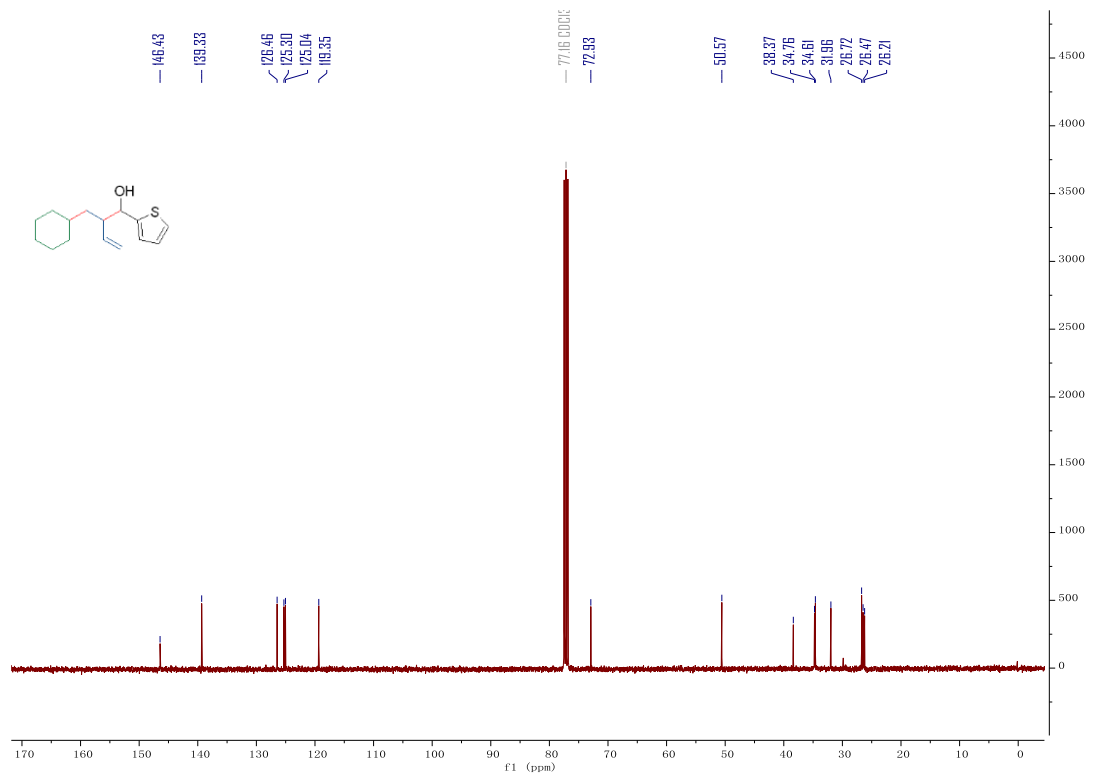


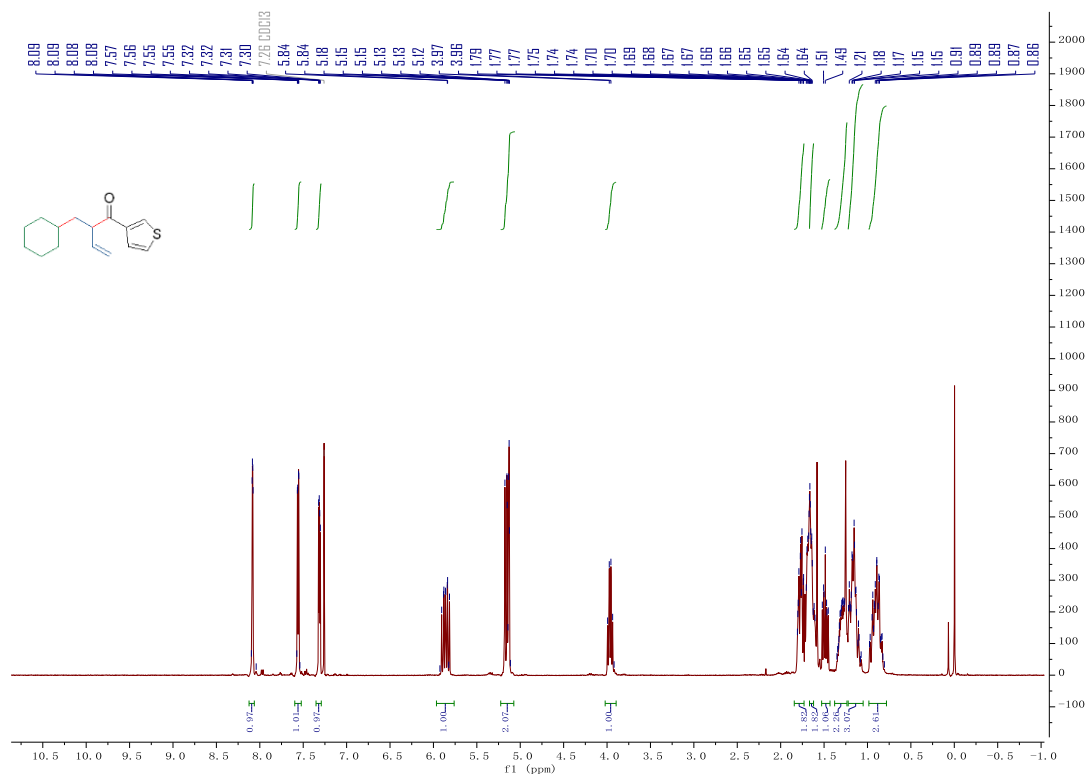
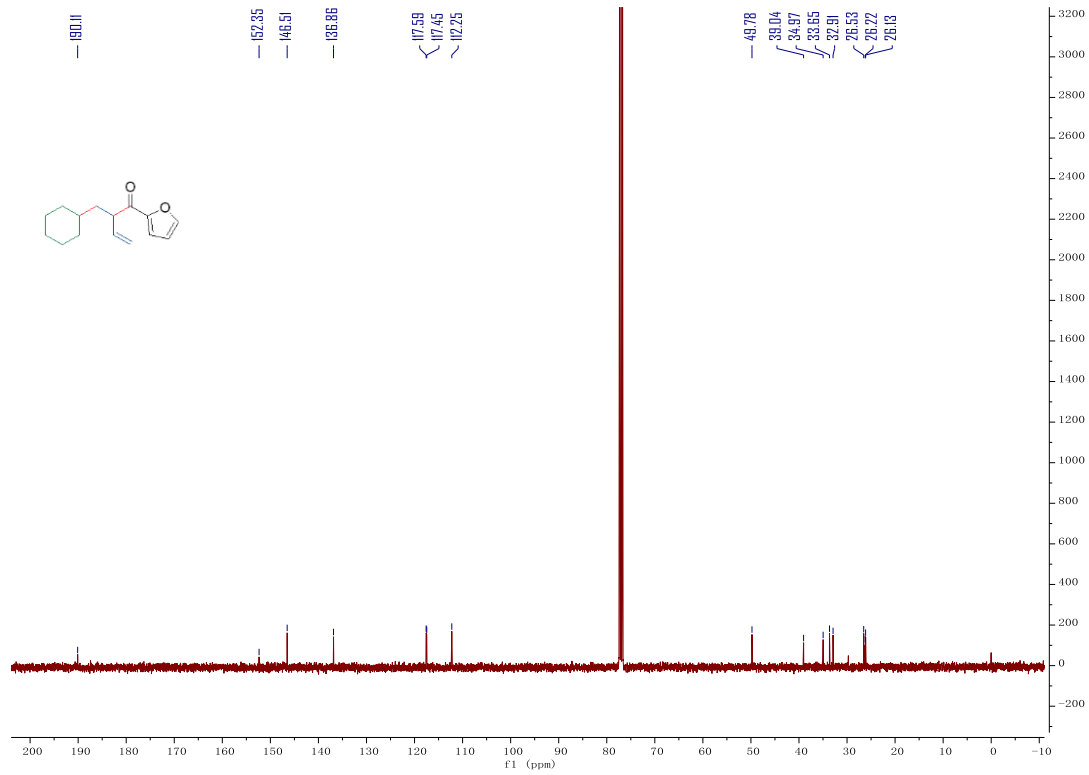


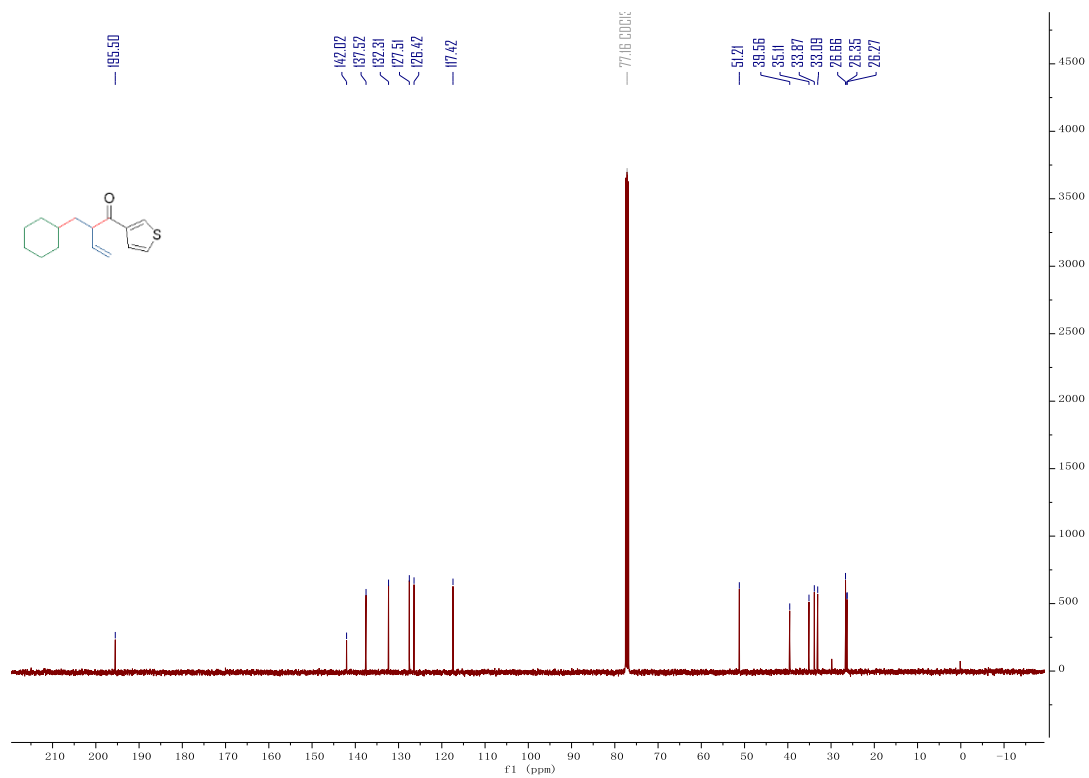












1. Jain, P.; Antilla, J. C. *J. Am. Chem. Soc.* **2010**, 132, 11884-11886.