

# Synthesis of C4-Alkynylisoxazoles *via* a Pd-Catalyzed Sonogashira Cross-Coupling Reaction

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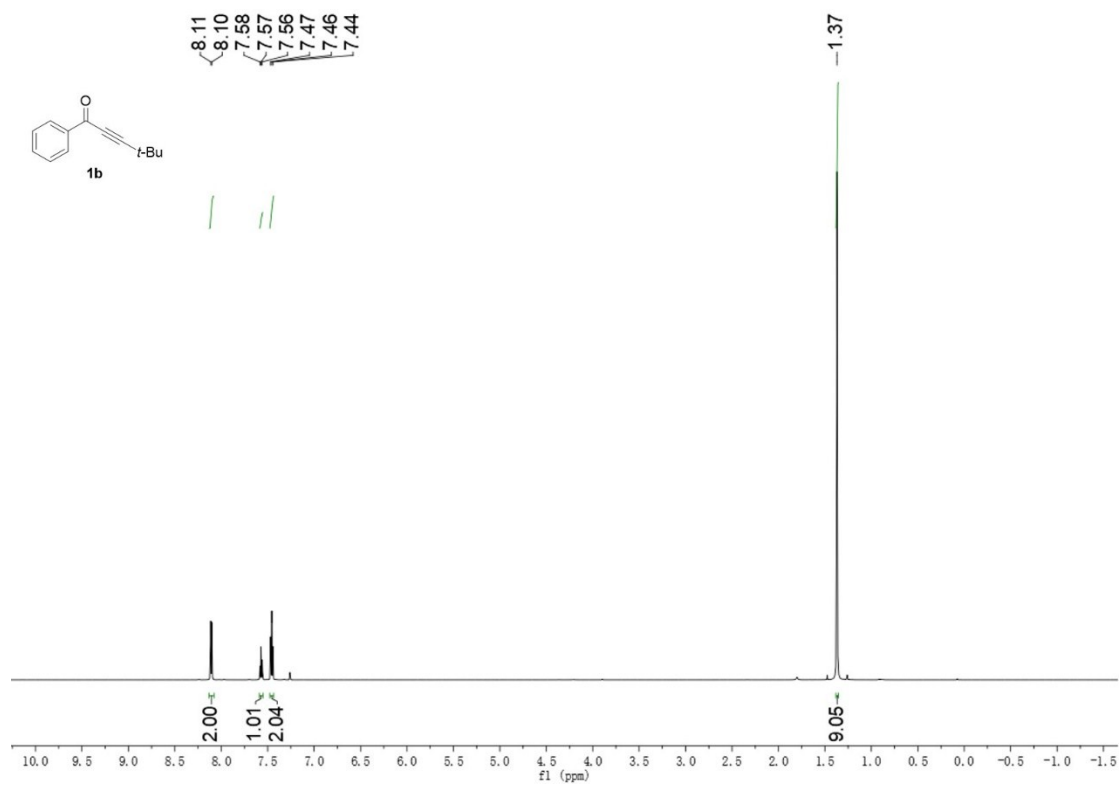
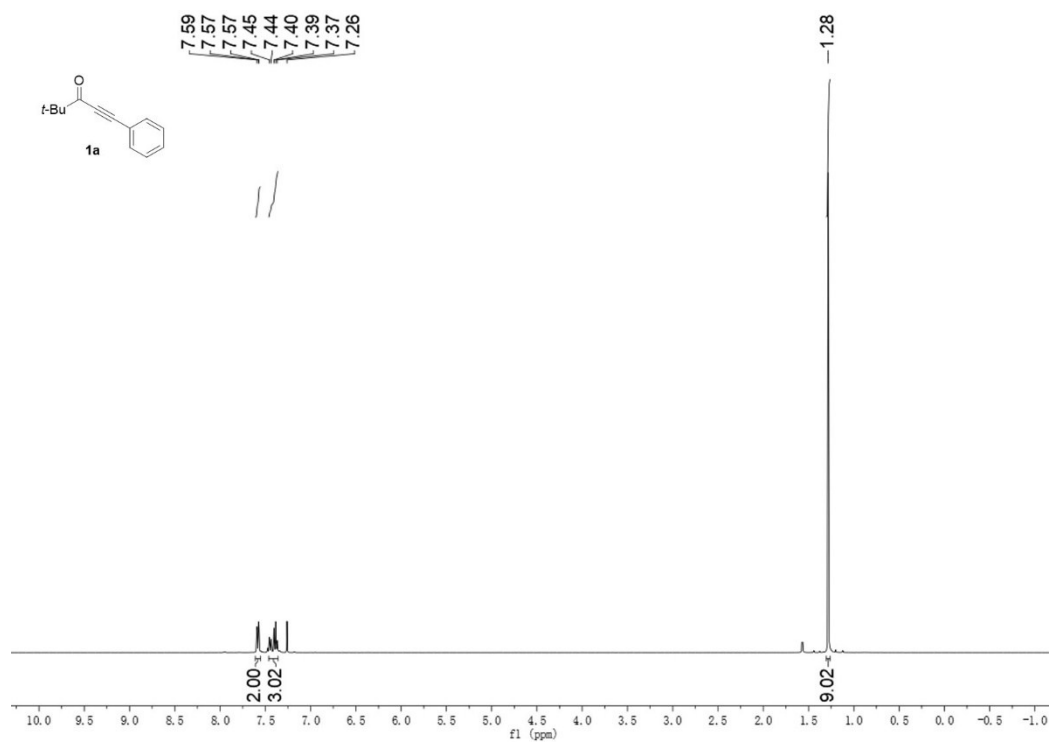
## 1. General information

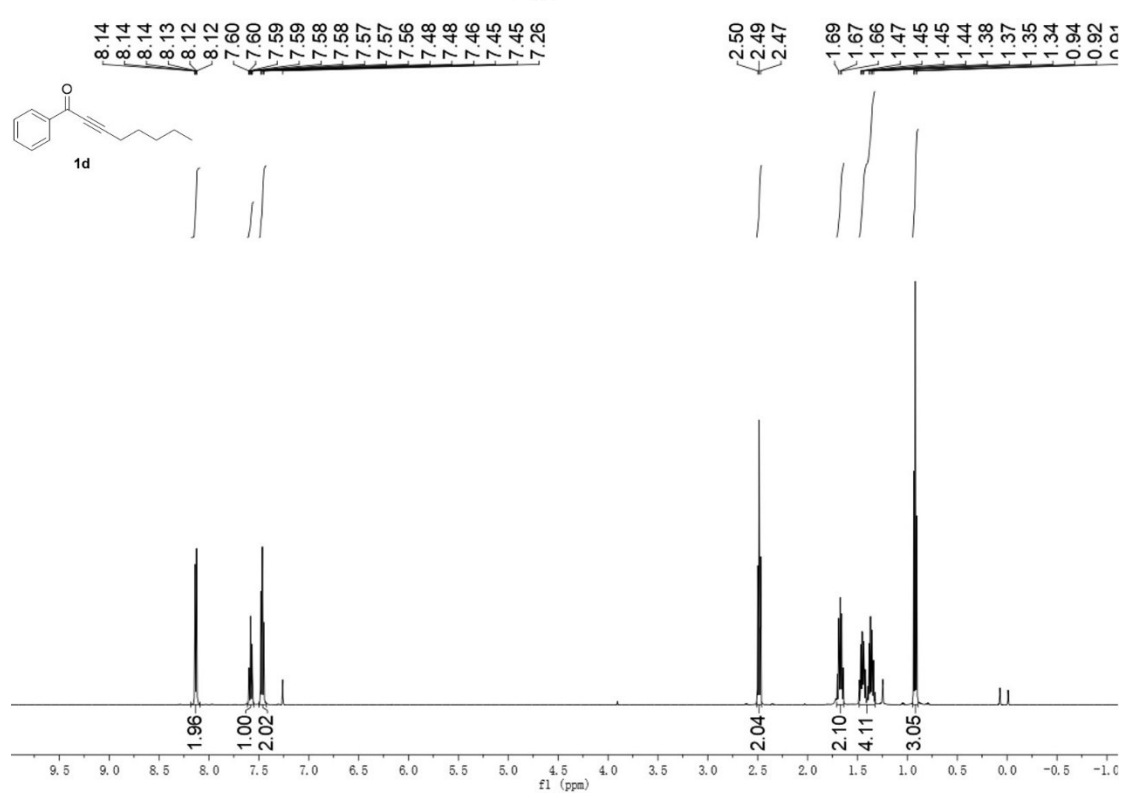
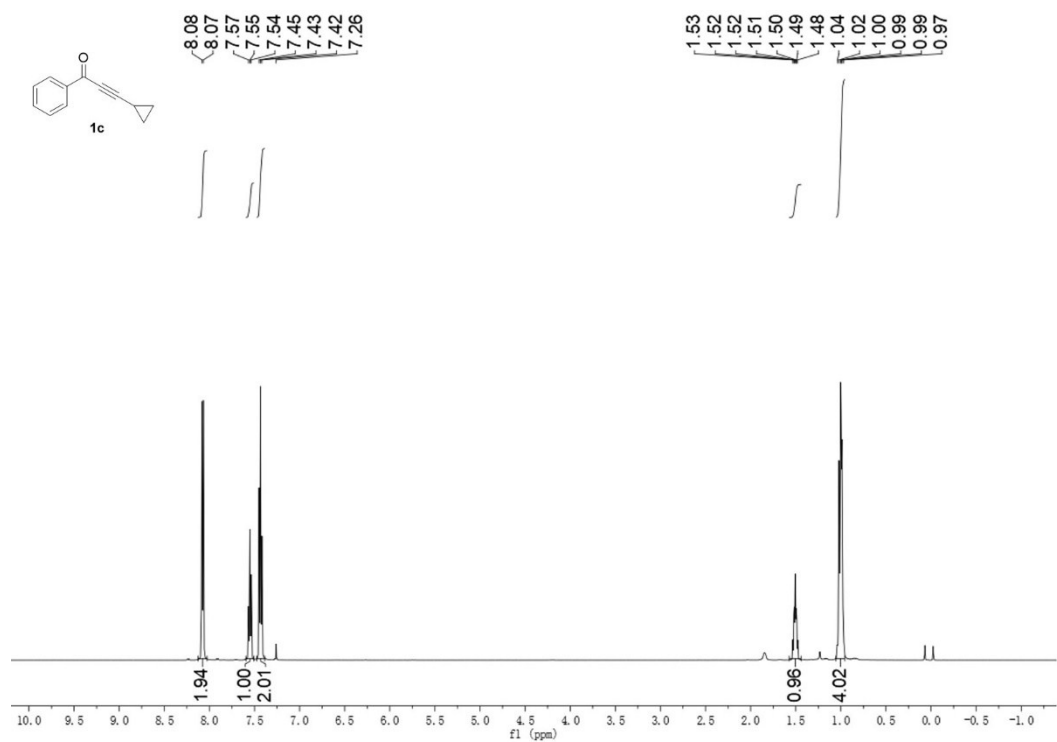
All reactions were carried out in 10.0 mL two-neck round-bottomed flask under a stream of nitrogen unless otherwise noted. Analytical thin layer chromatography (TLC) was performed with Silicycle normal phase glass plates (0.25 mm, 60A pore size, 230–400 mesh). Visualization was done under a 254 nm UV light source.

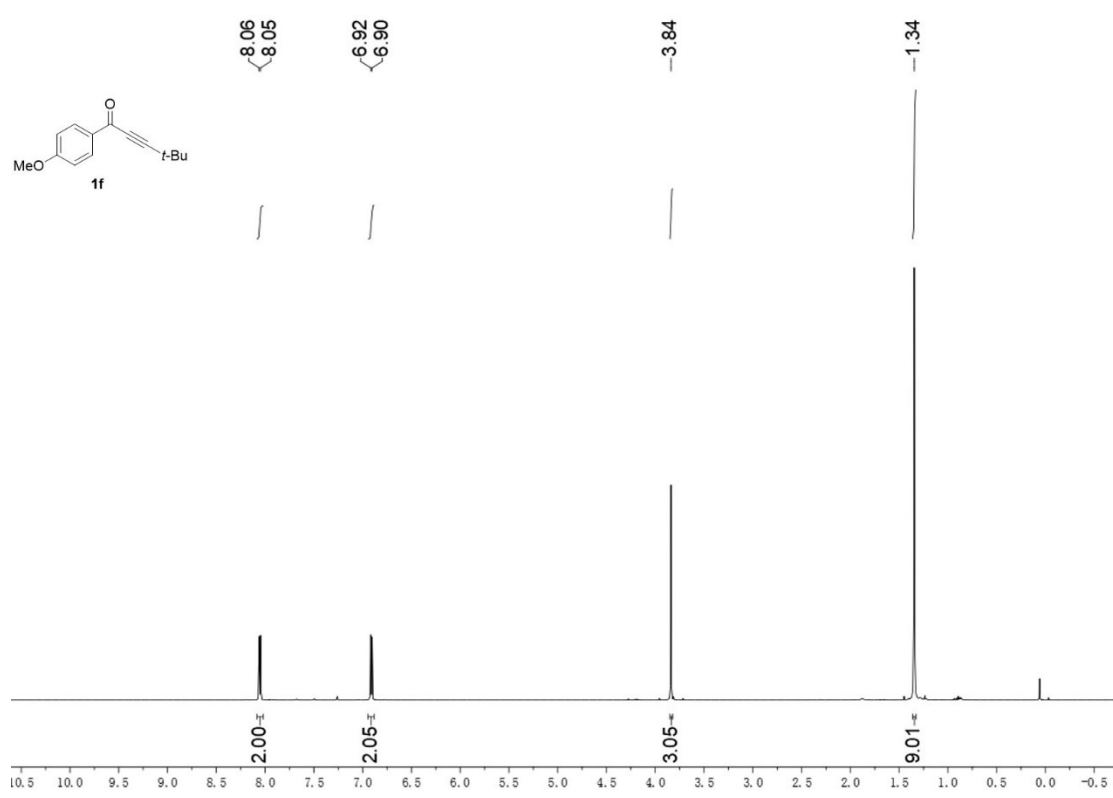
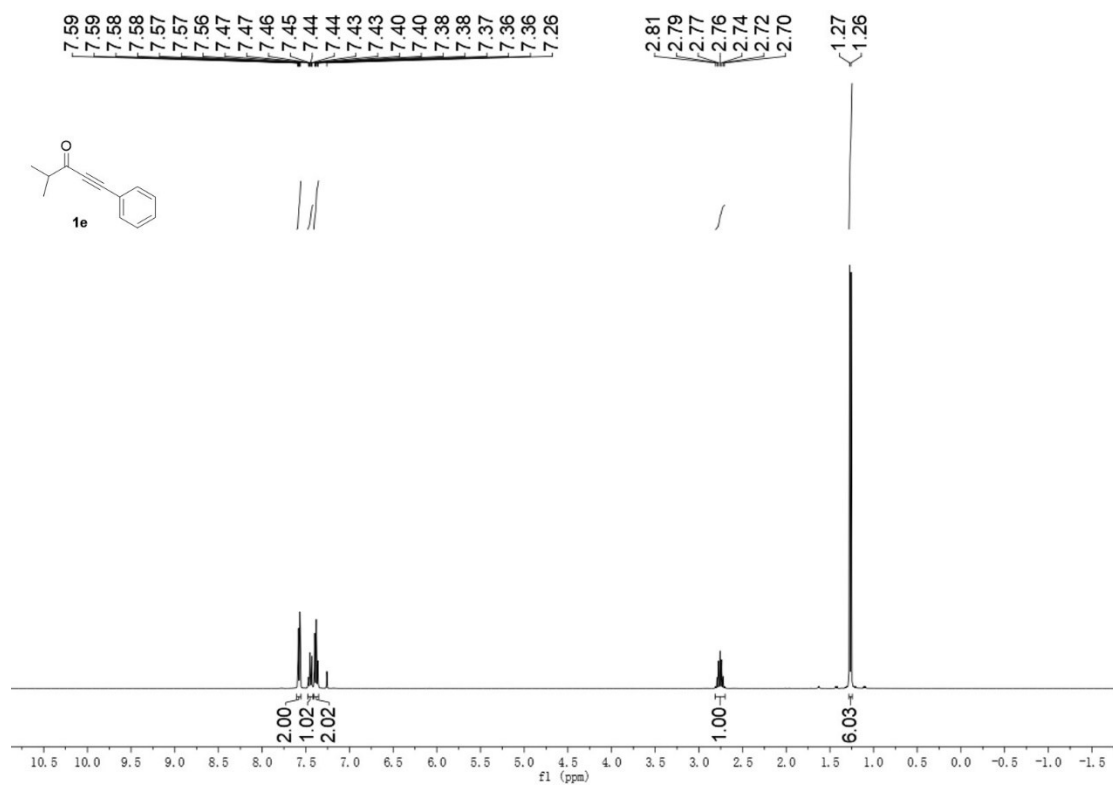
**Materials.** Unless otherwise stated, all starting materials, catalysts, and solvents were commercially available and were used as purchased without any pretreatment. Super anhydrous solvent DMF was used without any pretreatment. Flash column chromatography was performed using the indicated solvent system on Qingdao-Haiyang silica gel (200–300 mesh).

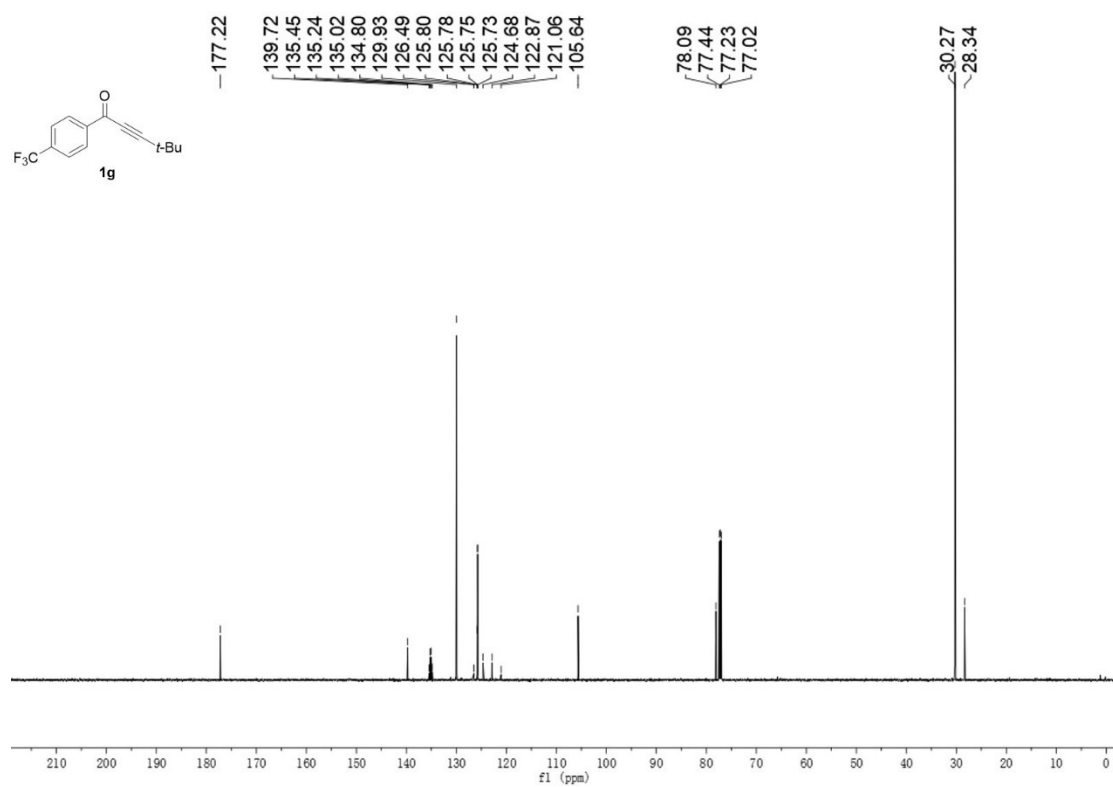
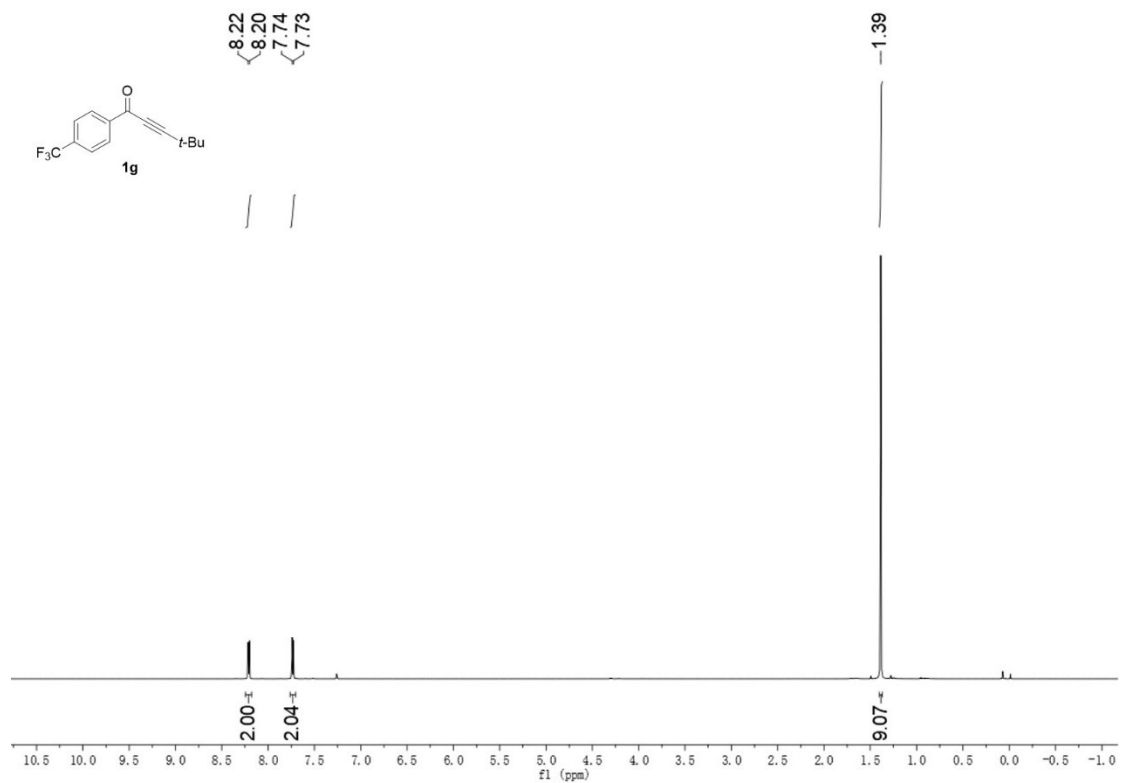
**Instrumentation.** Proton nuclear magnetic resonance spectra ( $^1\text{H}$  NMR) spectra and carbon nuclear magnetic resonance spectra ( $^{13}\text{C}$  NMR) were recorded at 23 °C with a Varian Mercury NMR spectrometer. Recorded shifts for protons are reported in parts per million ( $\delta$  scale) downfield from tetramethylsilane and are referenced to residual protium in the NMR solvents ( $\text{CDCl}_3$ :  $\delta$  7.26,  $d_6$ -DMSO:  $\delta$  2.50). Chemical shifts for carbon resonances are reported in parts per million ( $\delta$  scale) downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent ( $\text{CDCl}_3$ :  $\delta$  77.23,  $d_6$ -DMSO:  $\delta$  39.52). Data are represented as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), and coupling constant ( $J$ , Hz). HRMS (ion trap) were obtained from mass spectrometer (ESI). Melting points were recorded on an electrothermal digital melting point apparatus and were uncorrected.

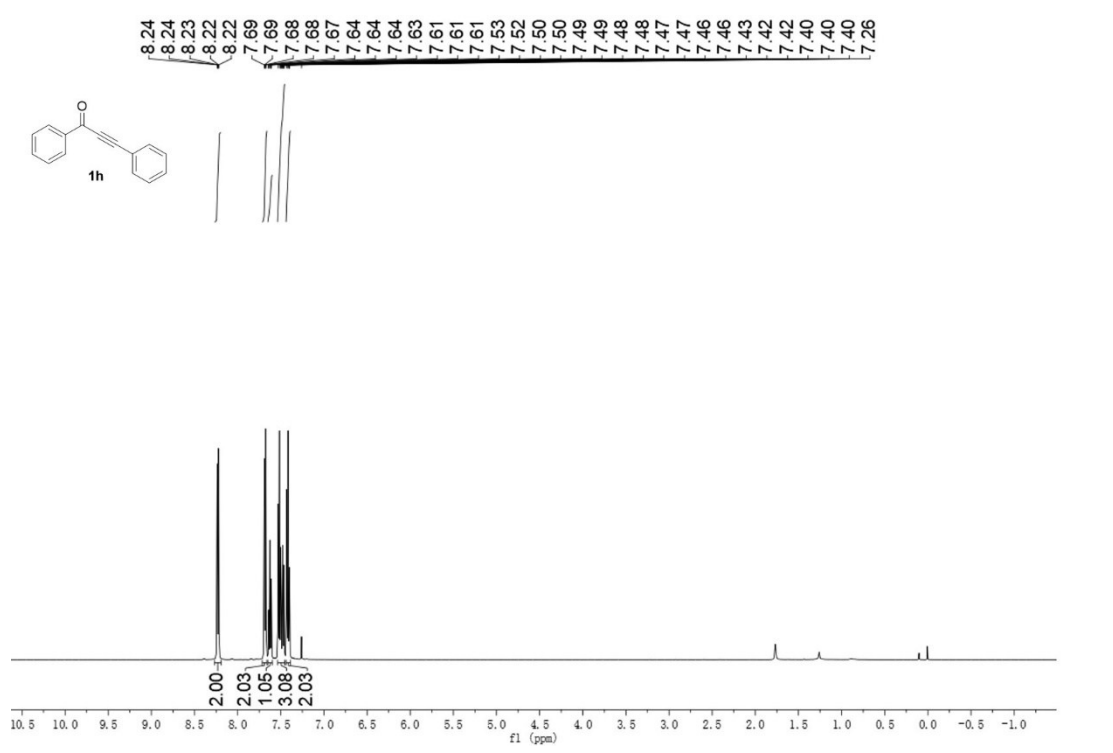
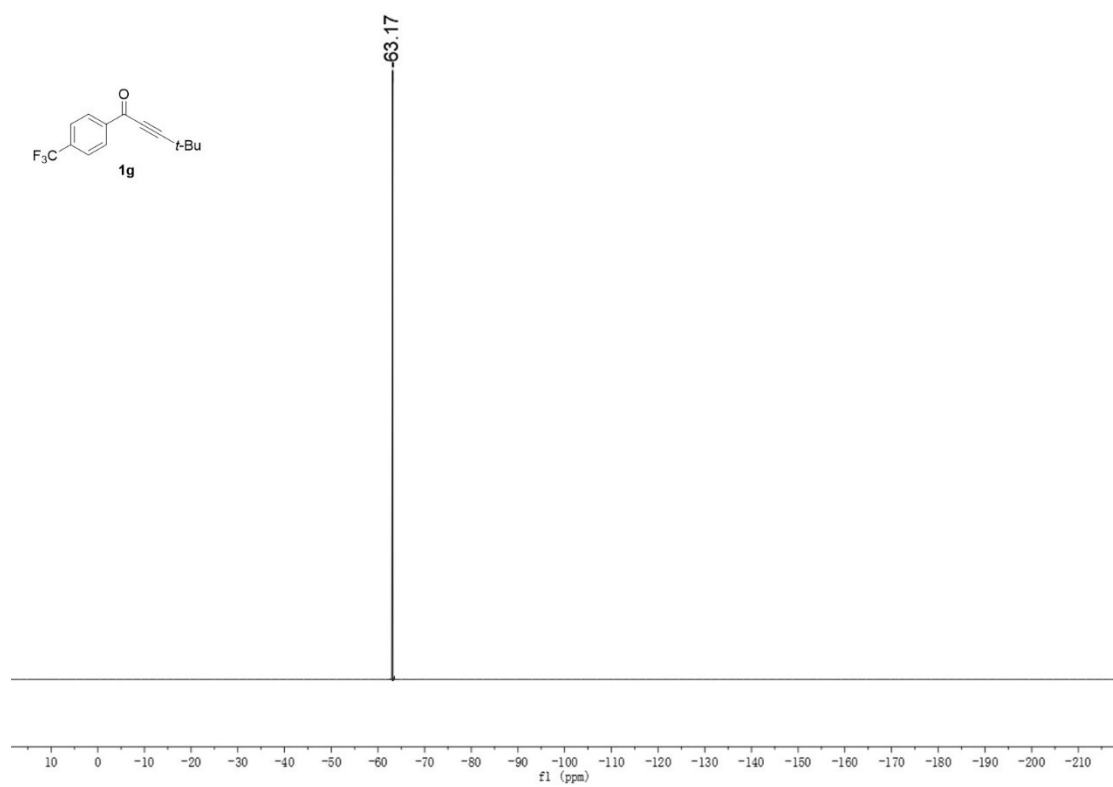
## 2. Copies of $^1\text{H}$ , $^{13}\text{C}$ and $^{19}\text{F}$ NMR spectra for intermediates 1a - 1h, 2a - 2h and 3a - 3h

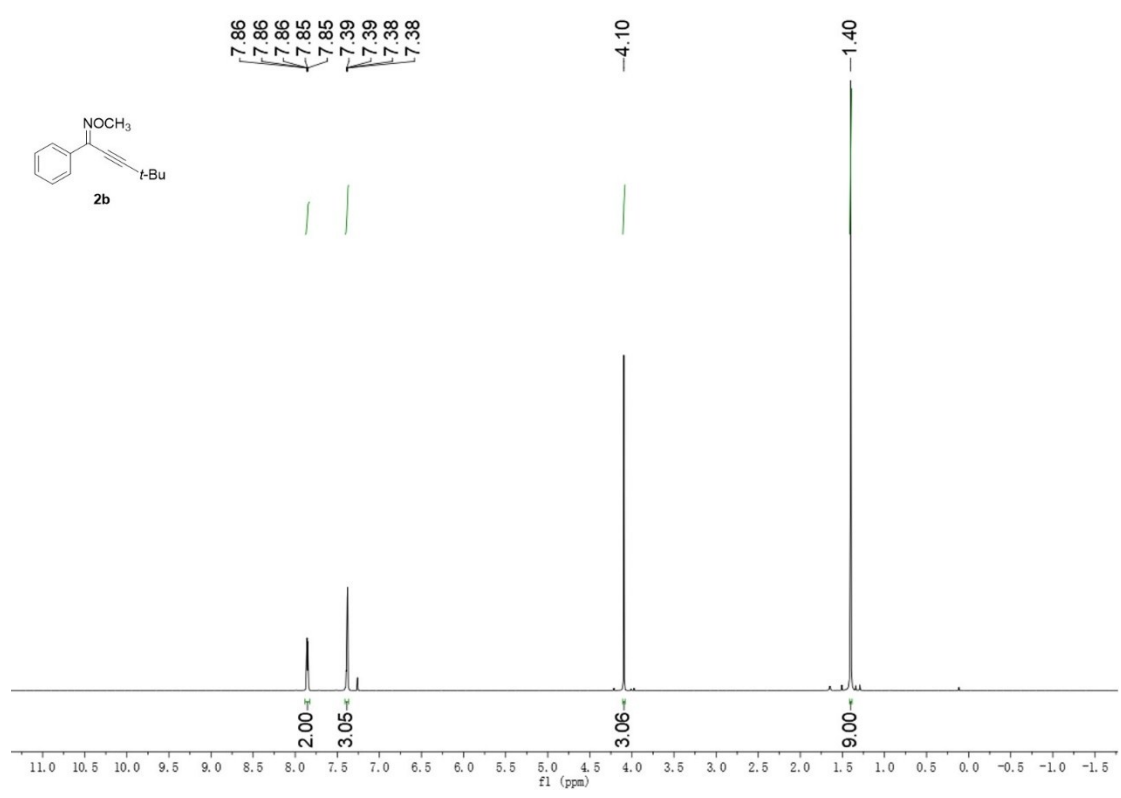
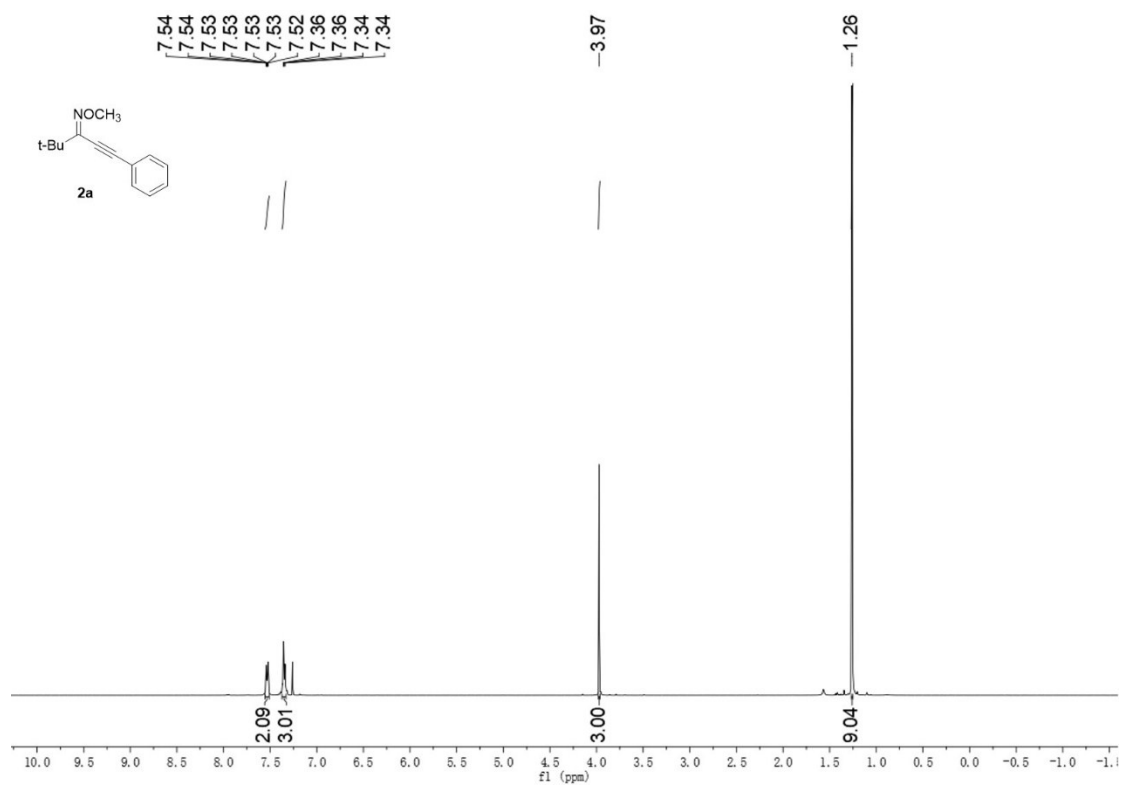




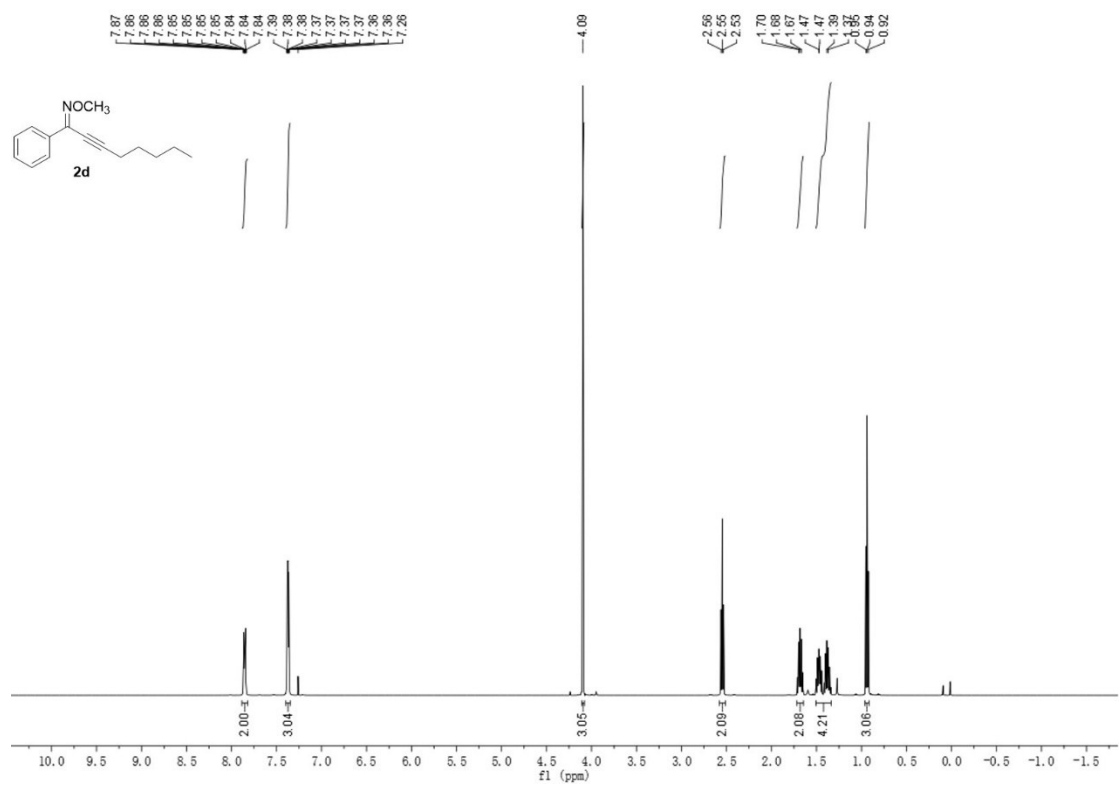
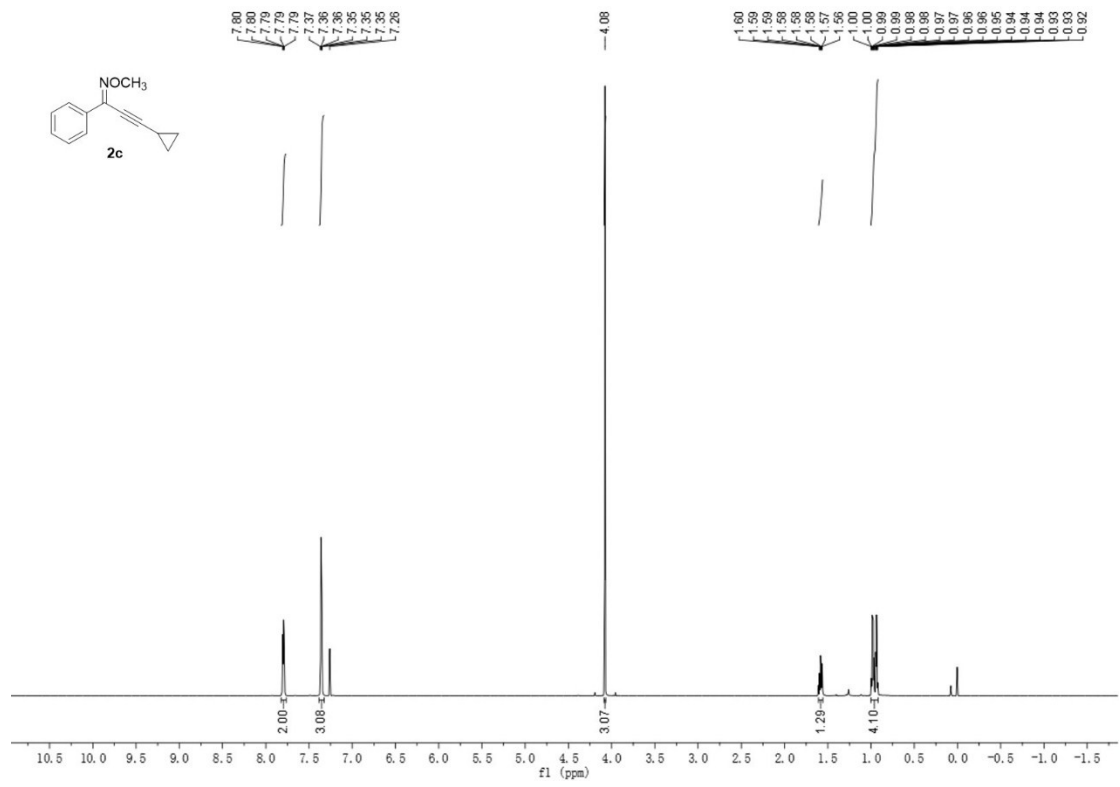


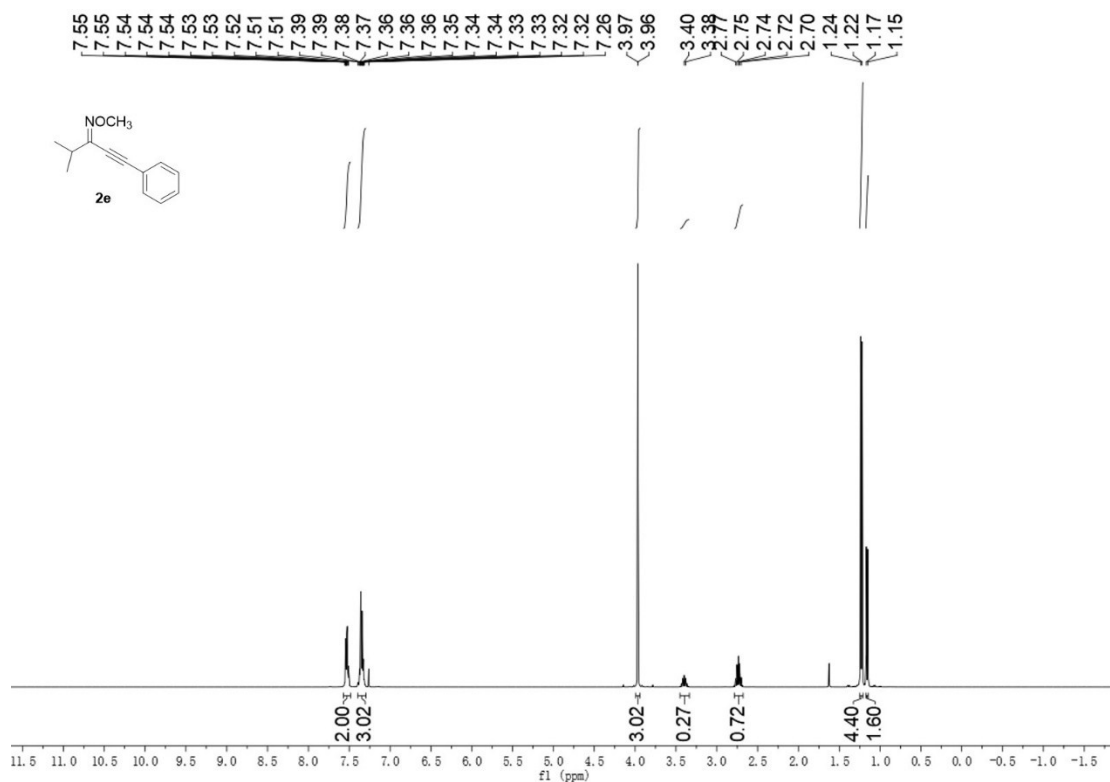
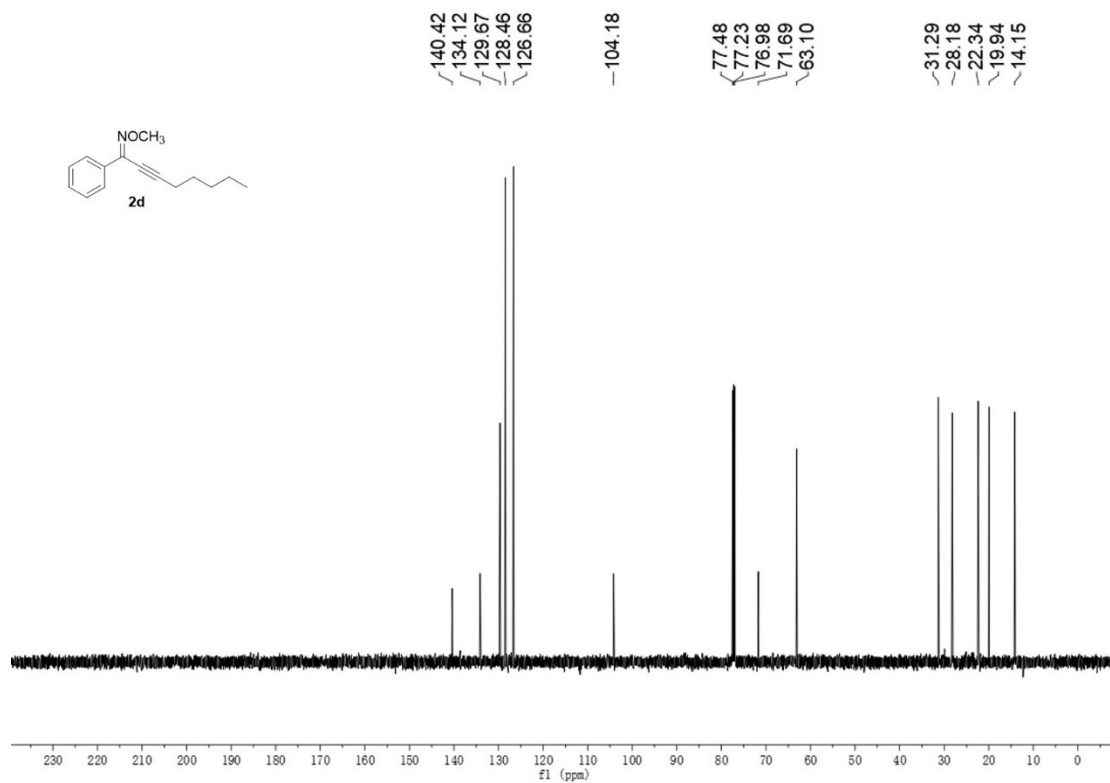


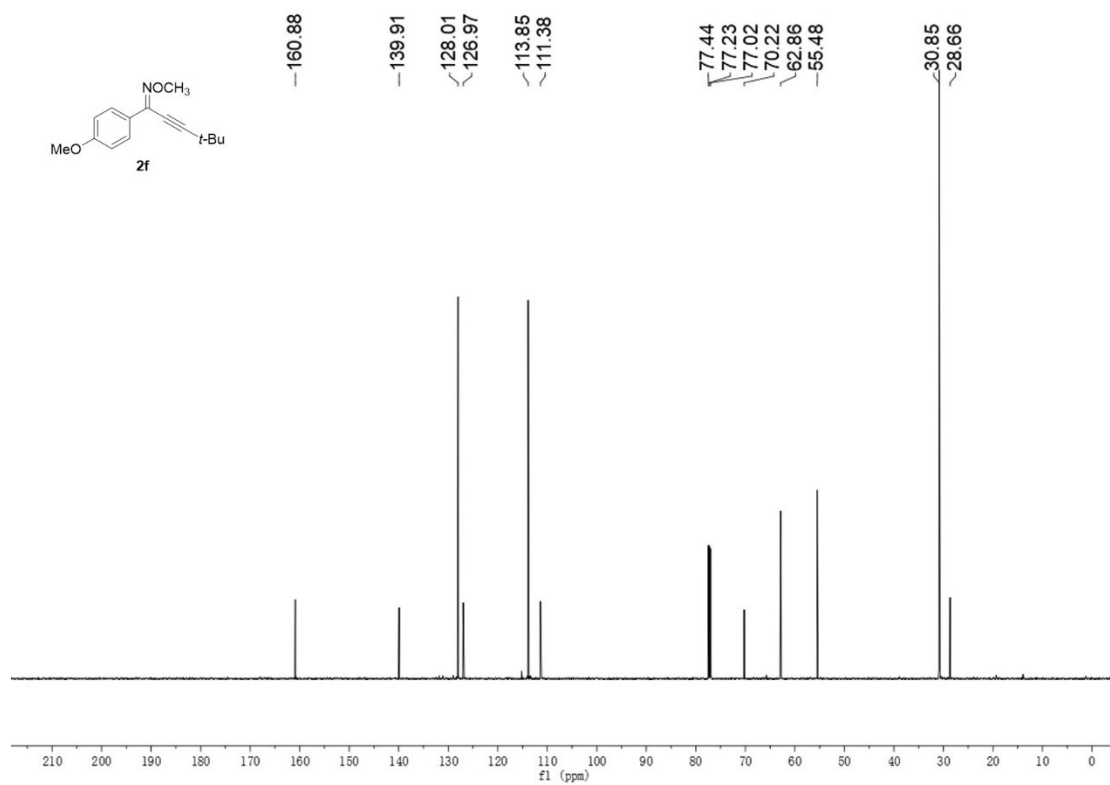
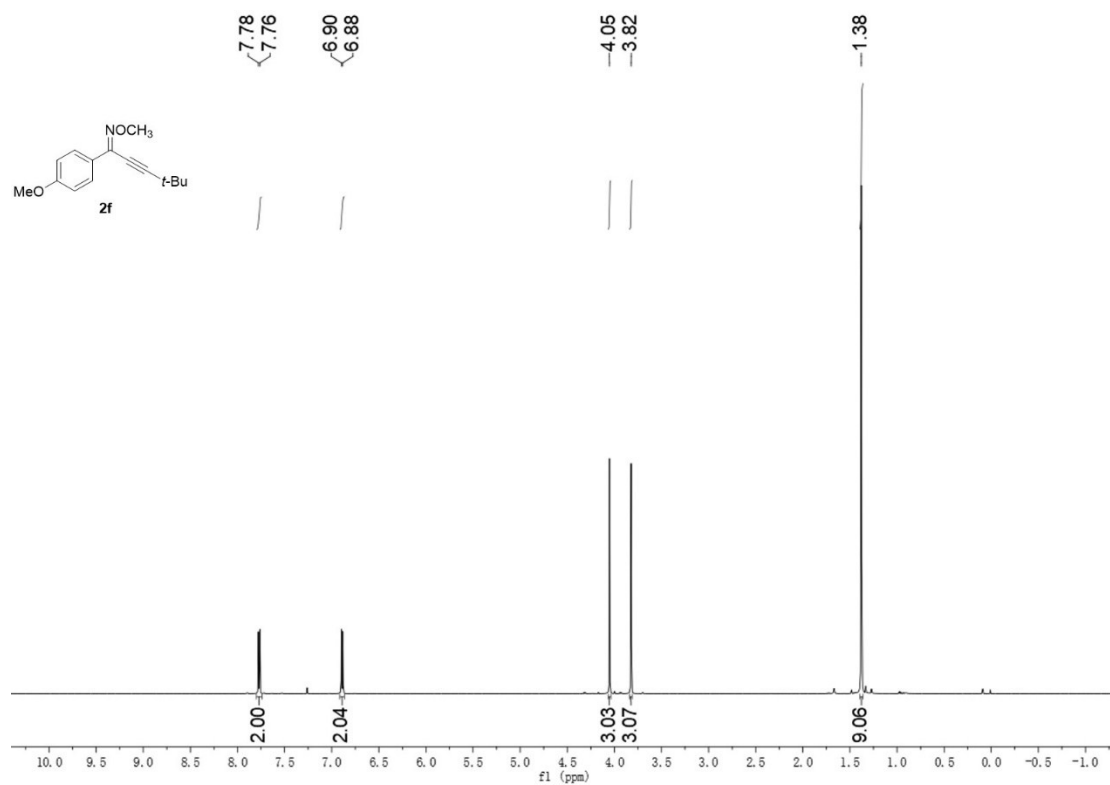


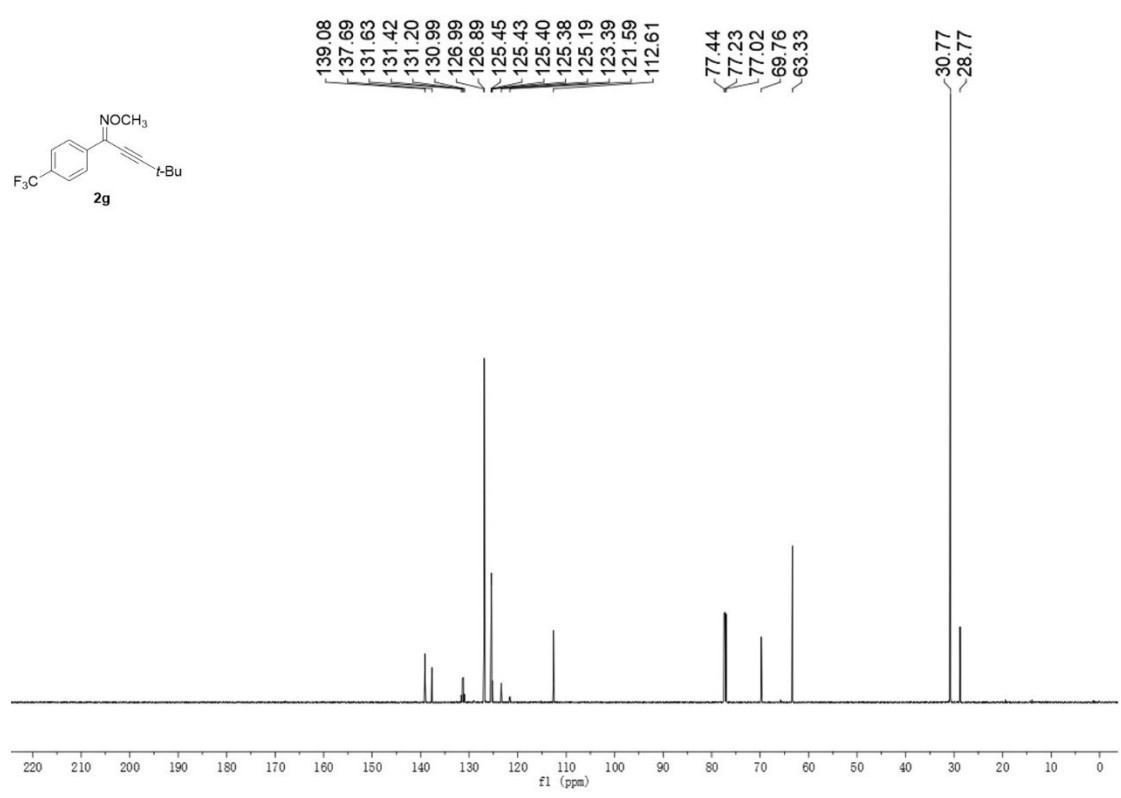
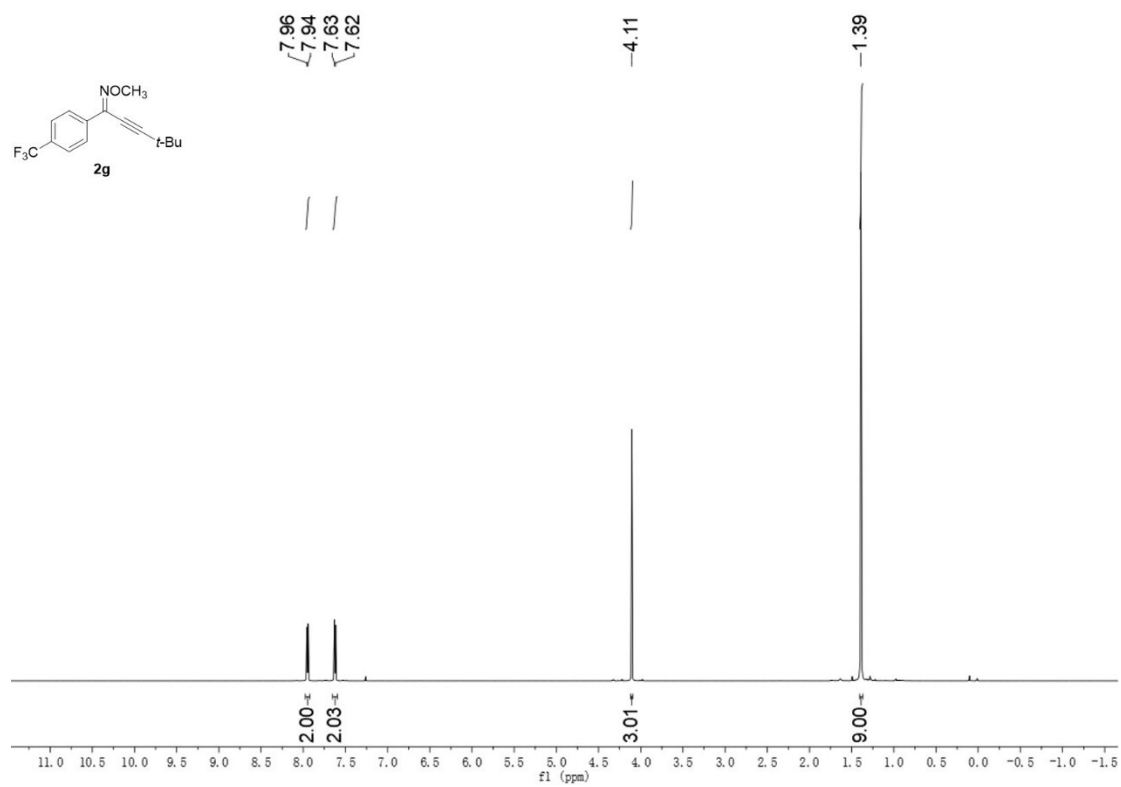


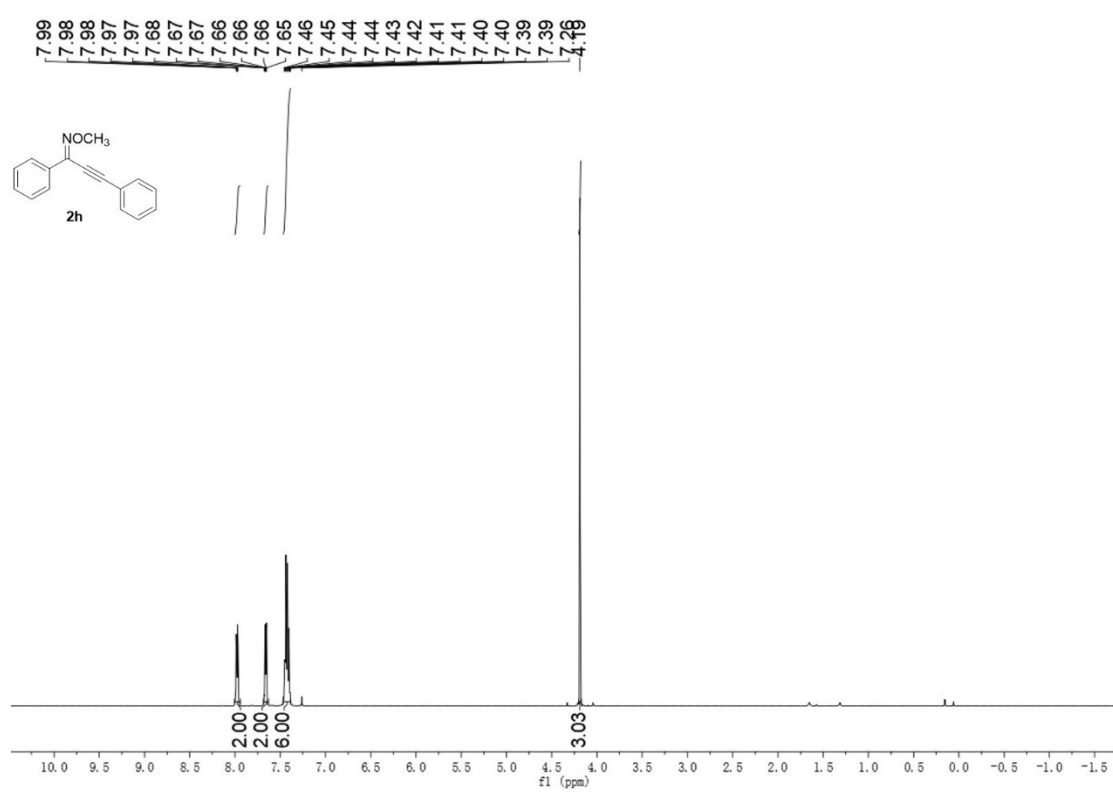
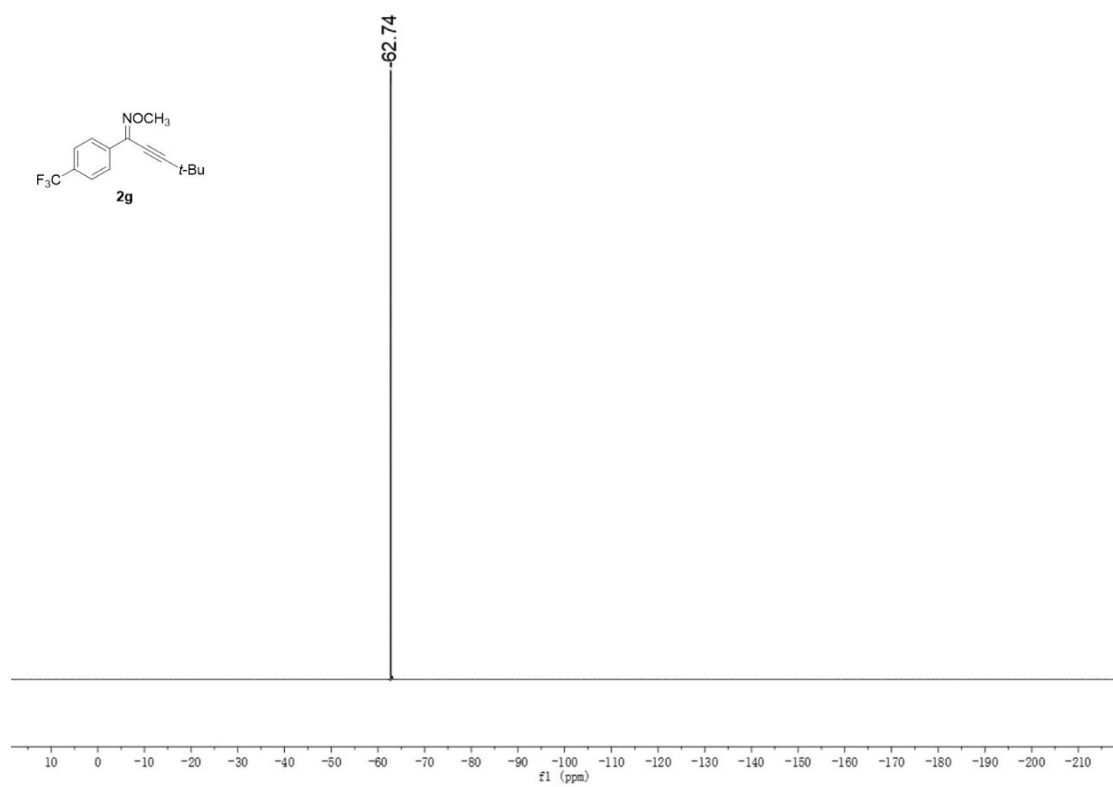


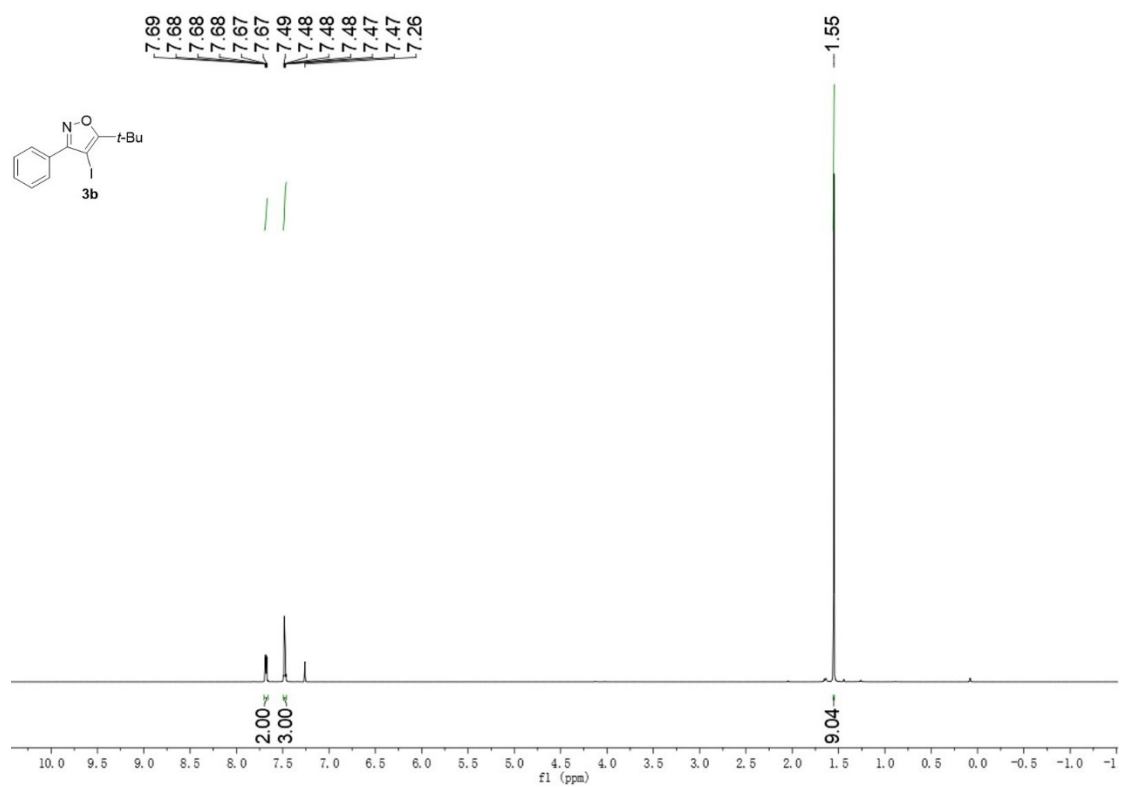
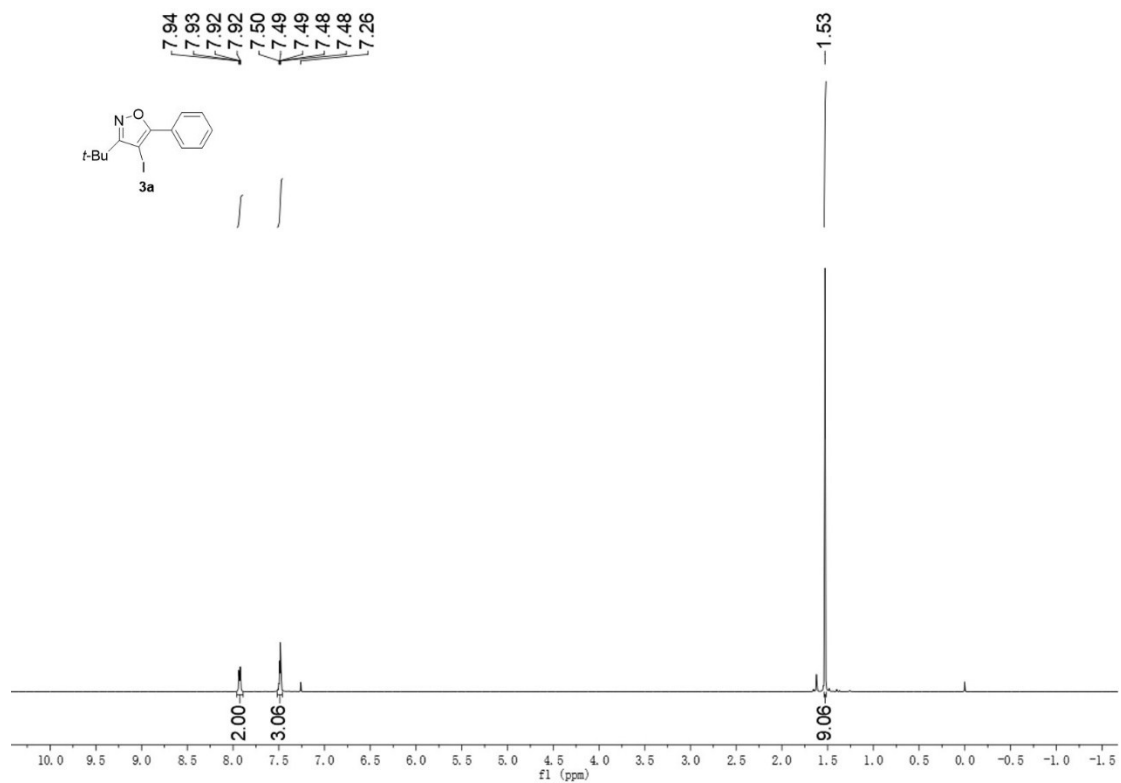


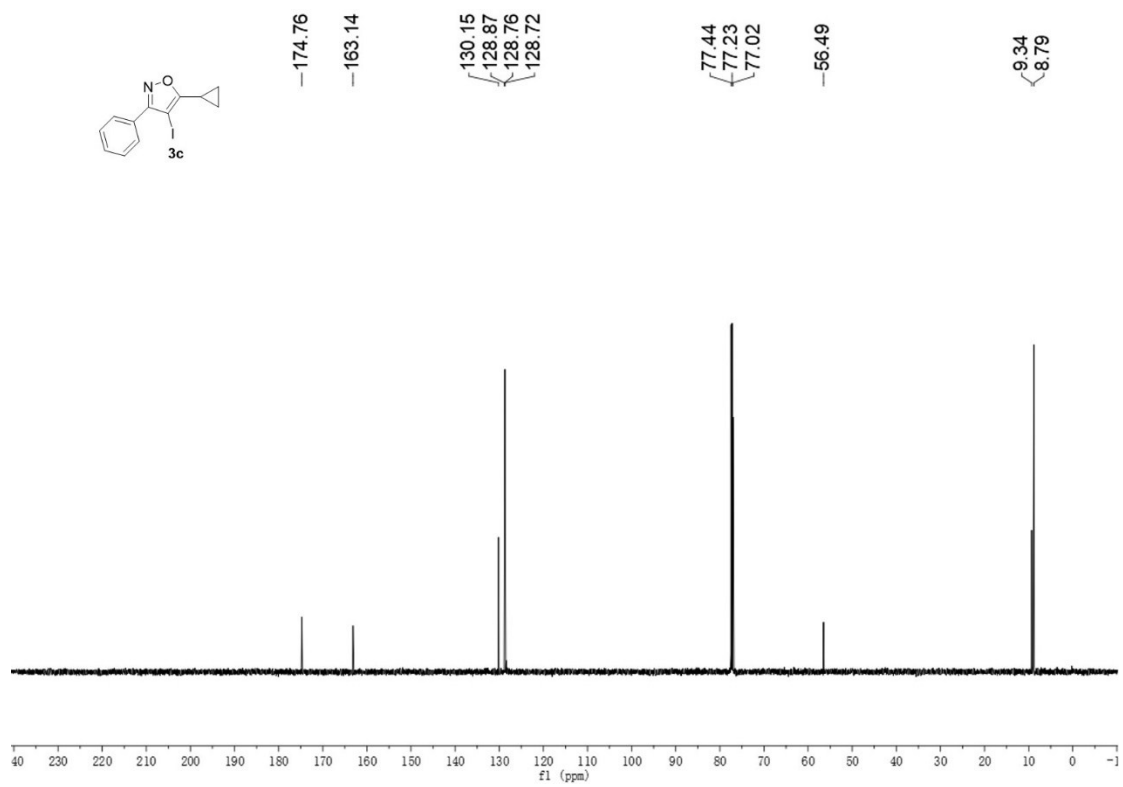
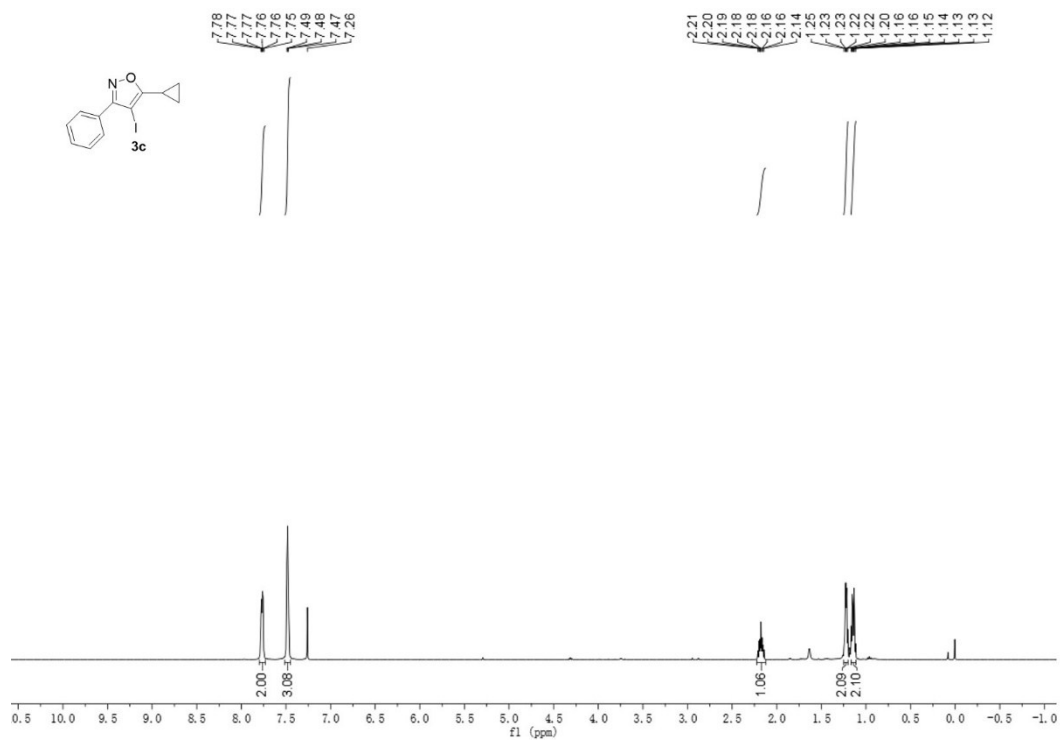


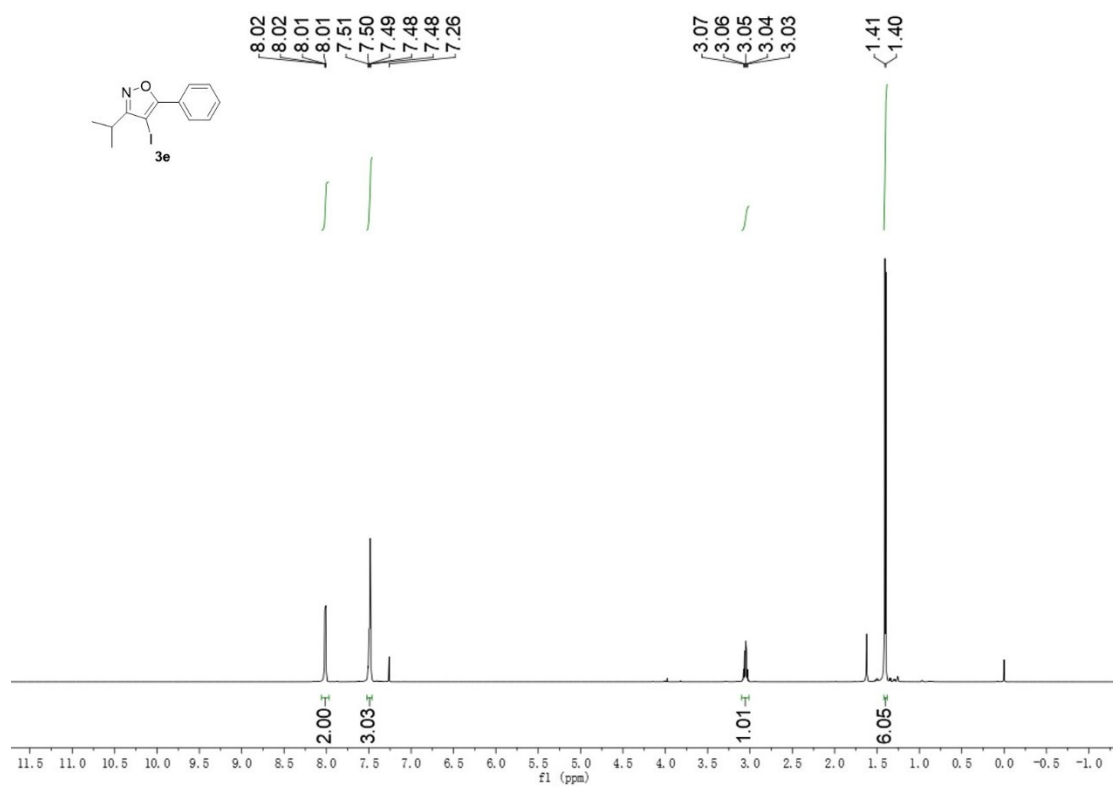
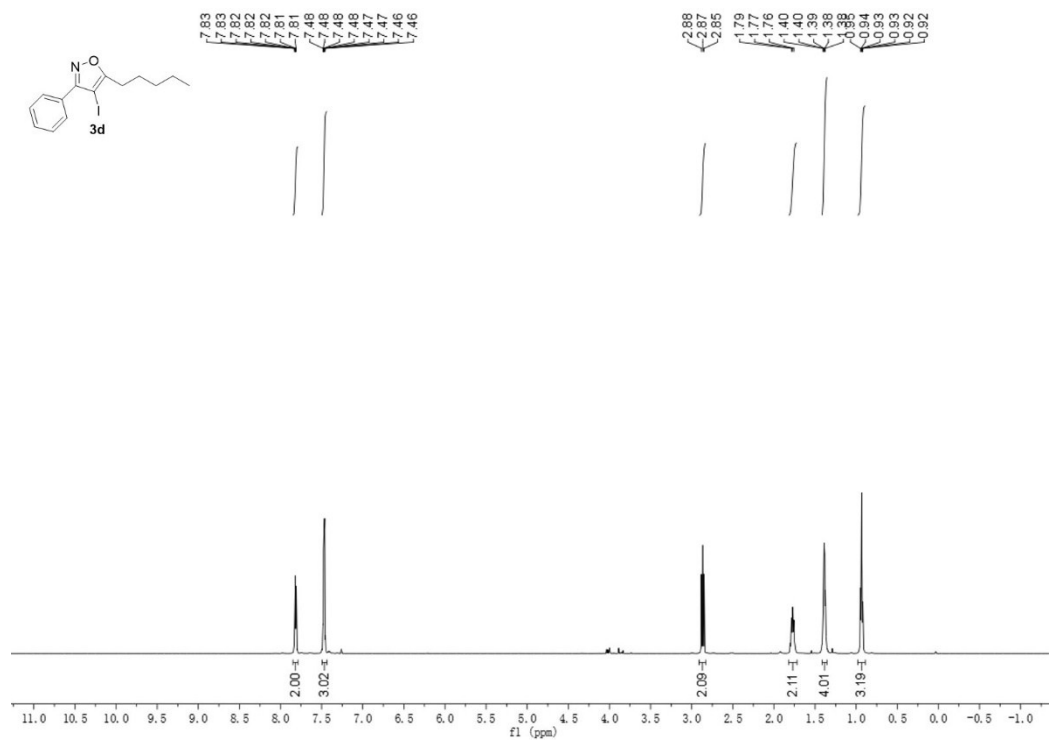




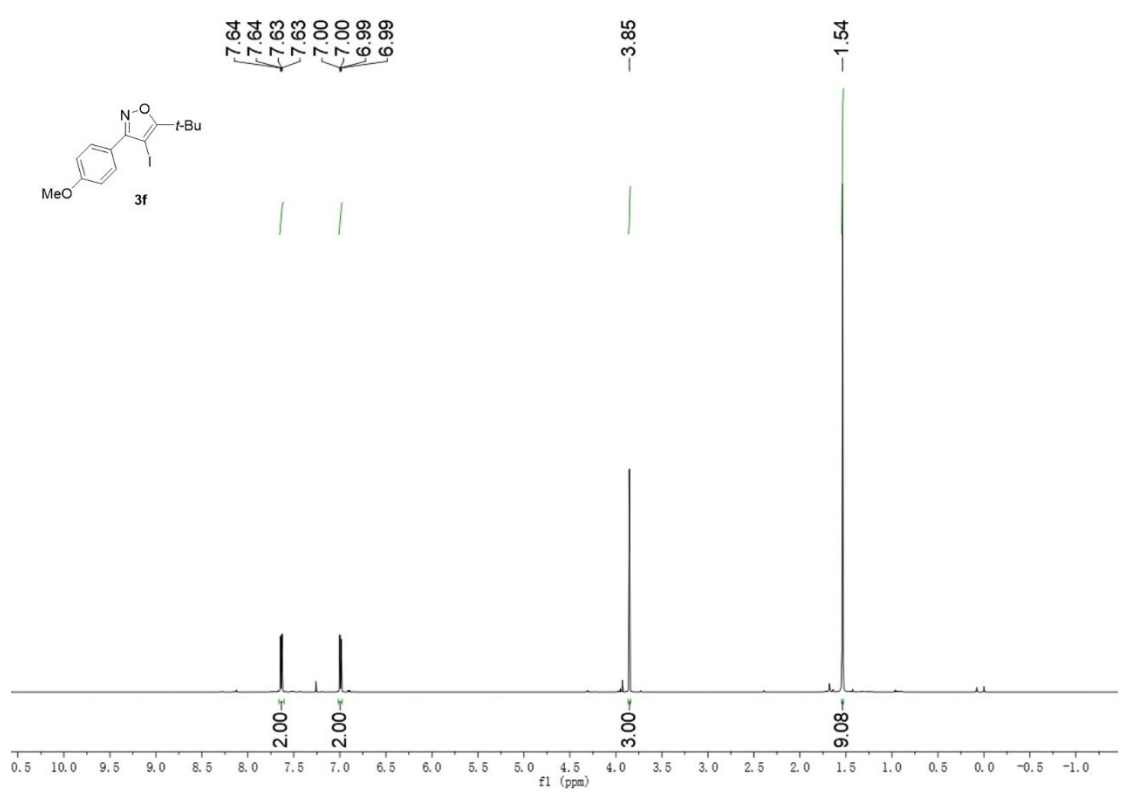
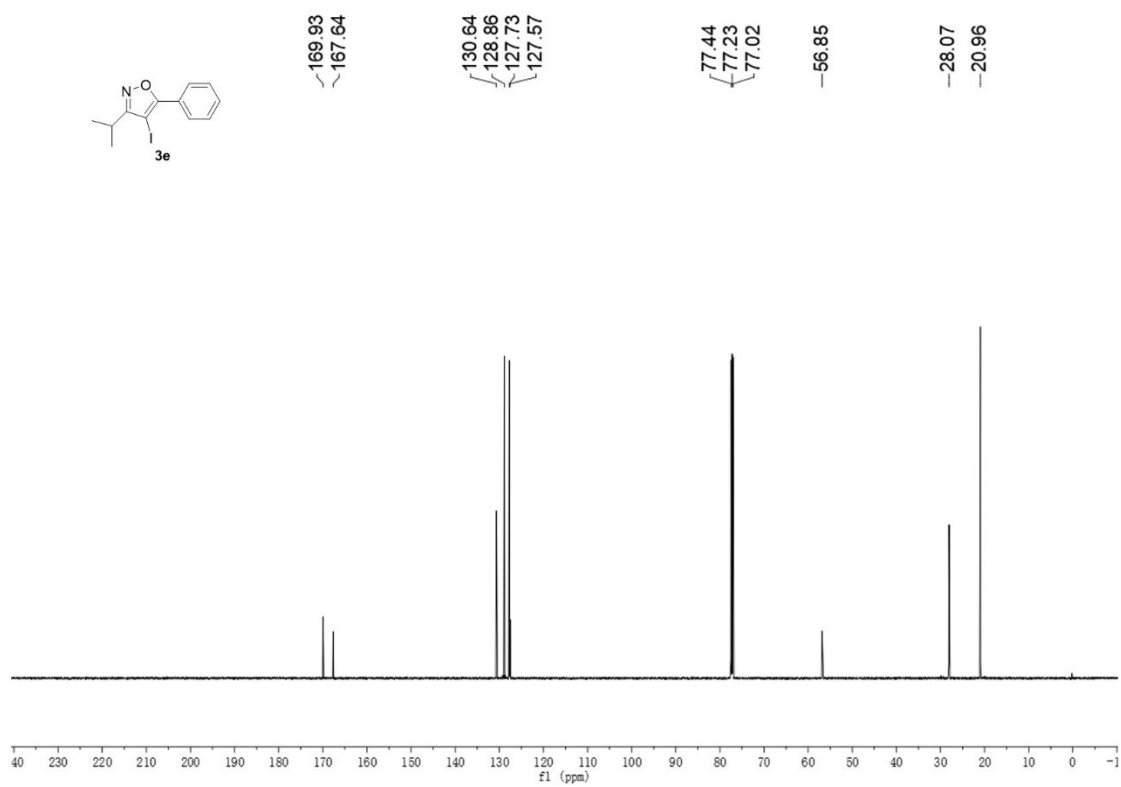


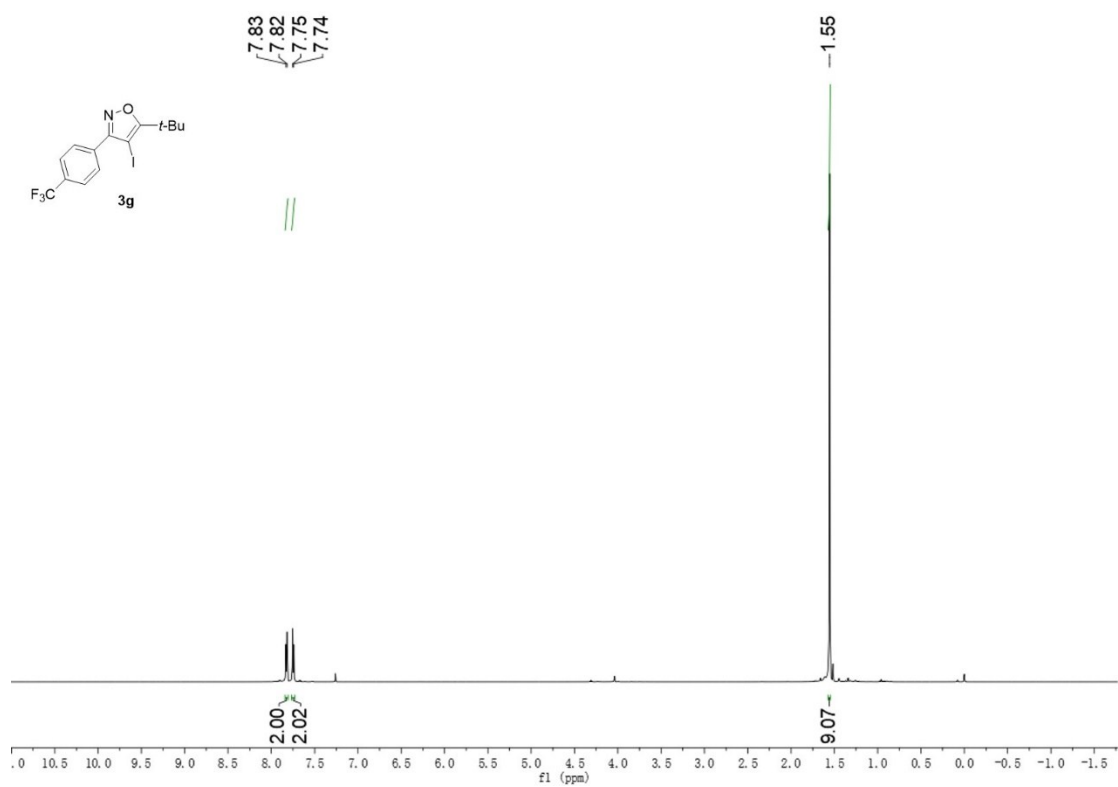
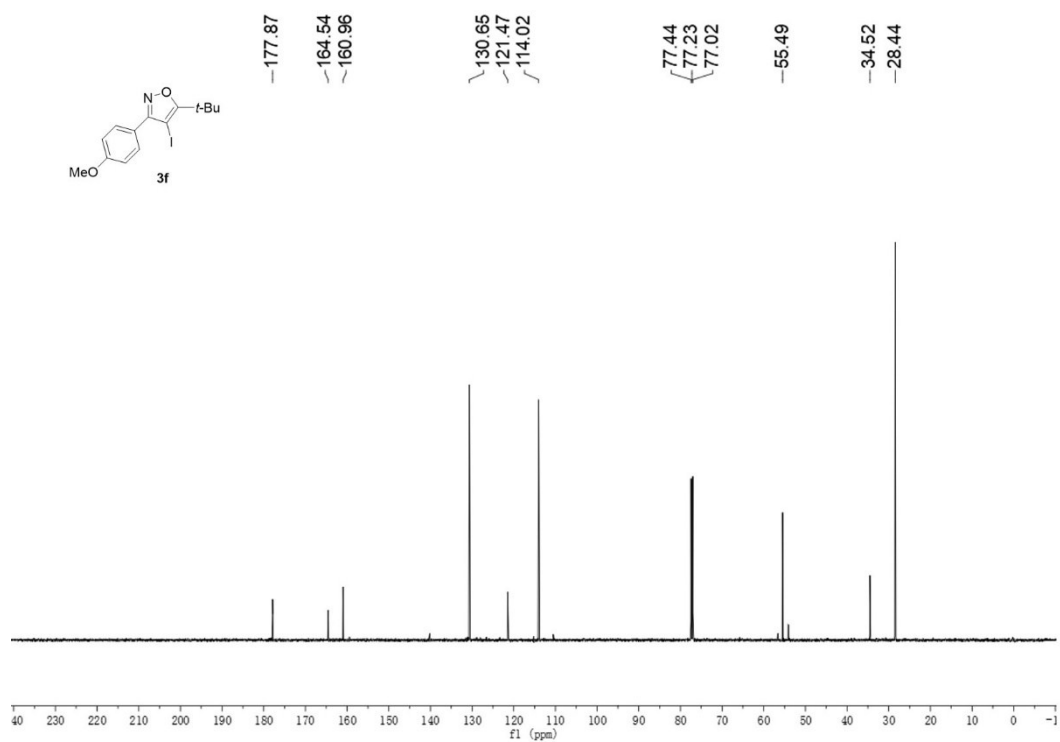


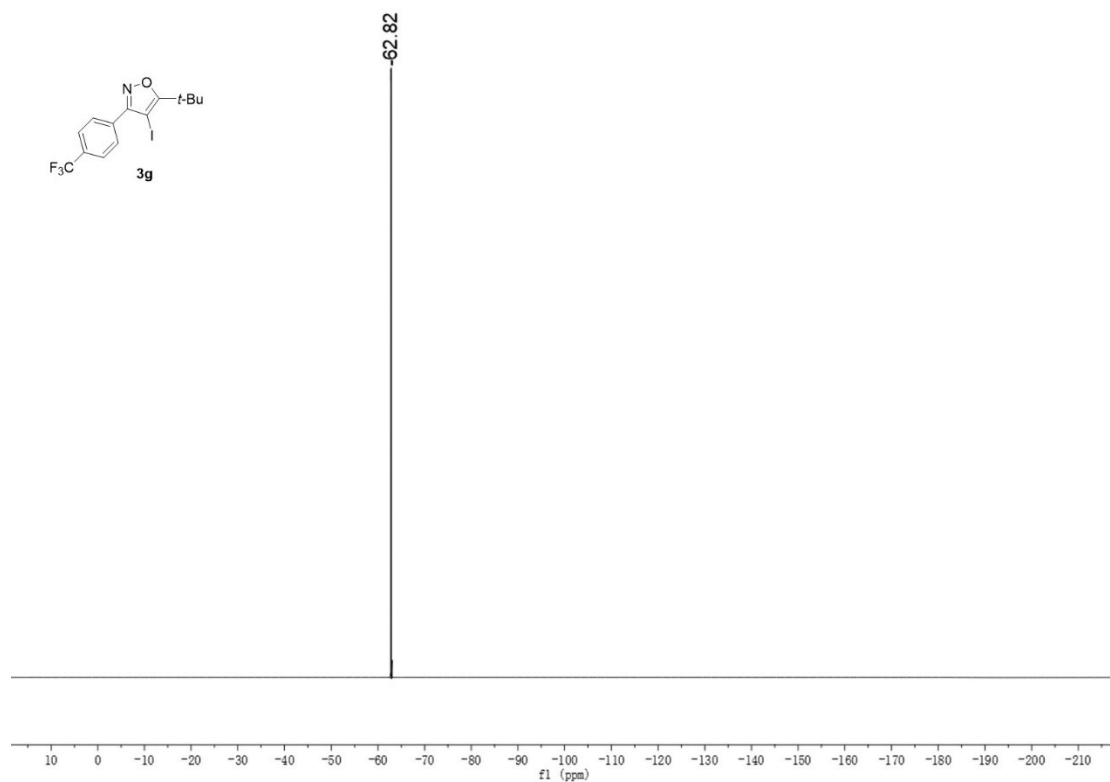
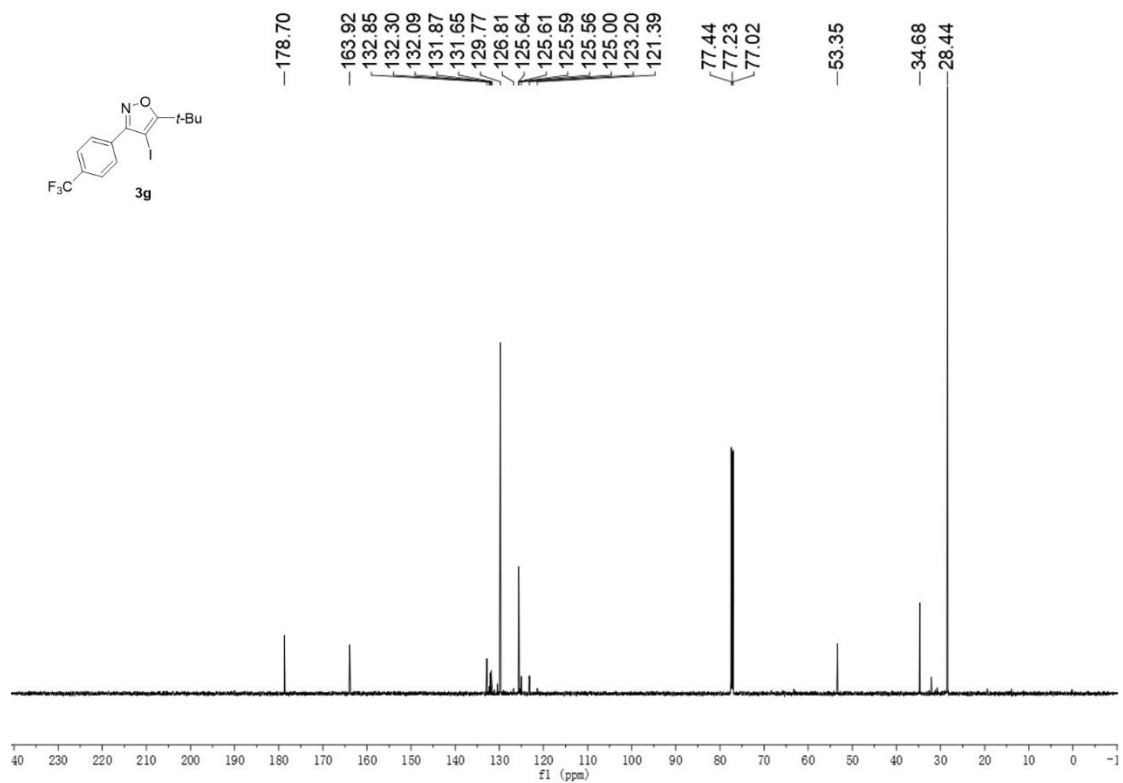


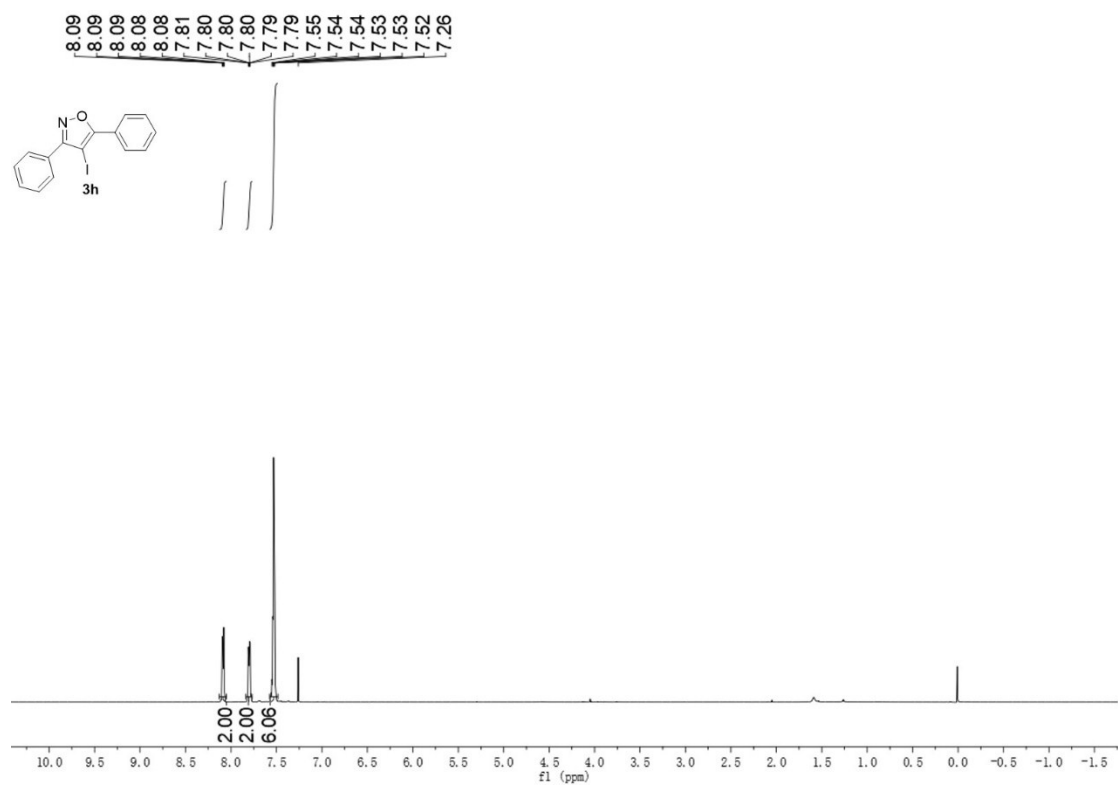




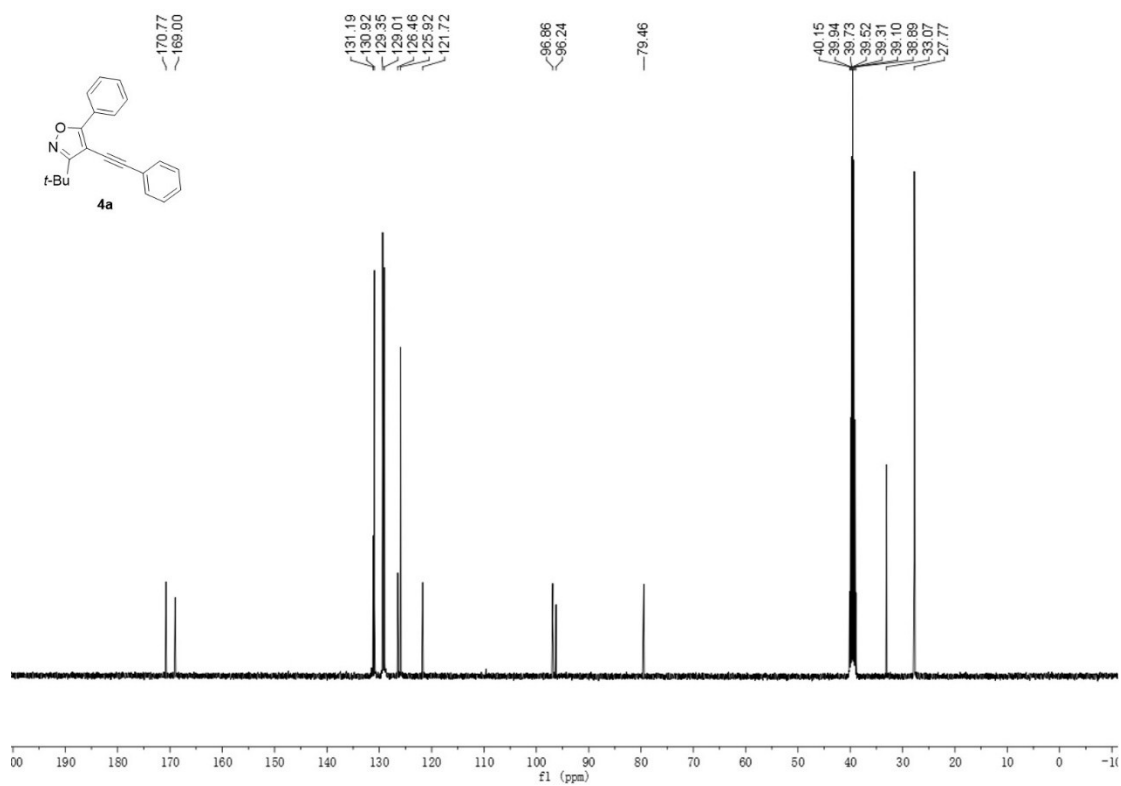
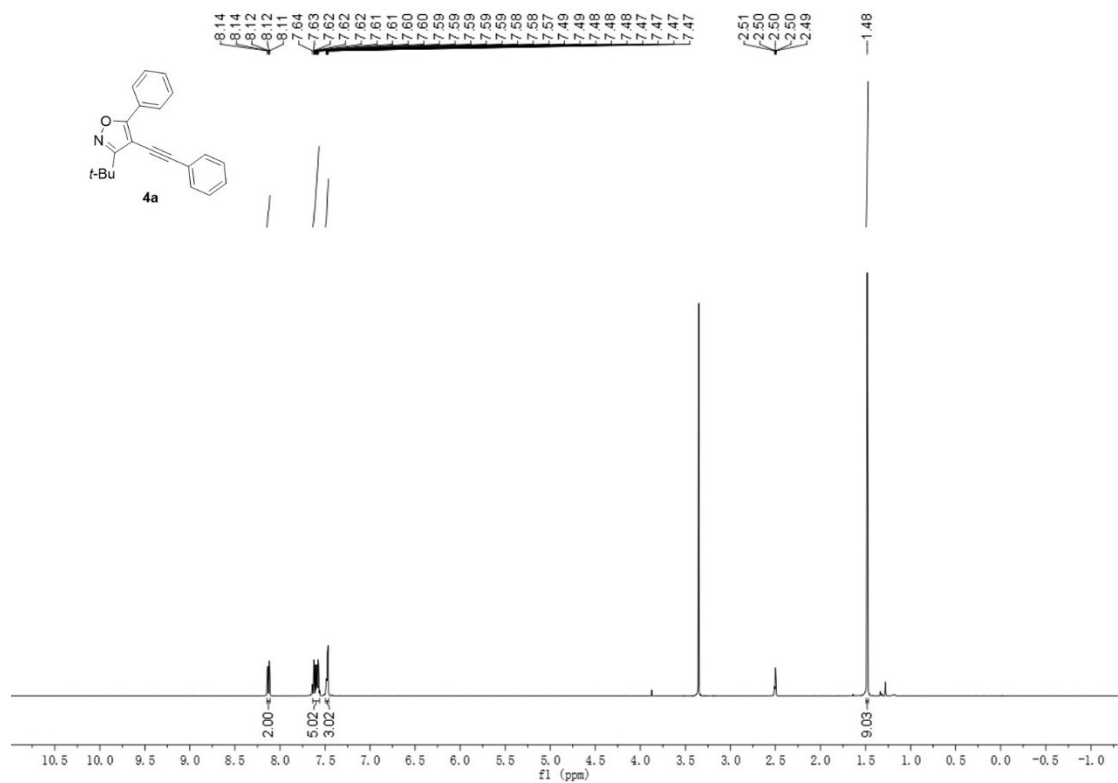


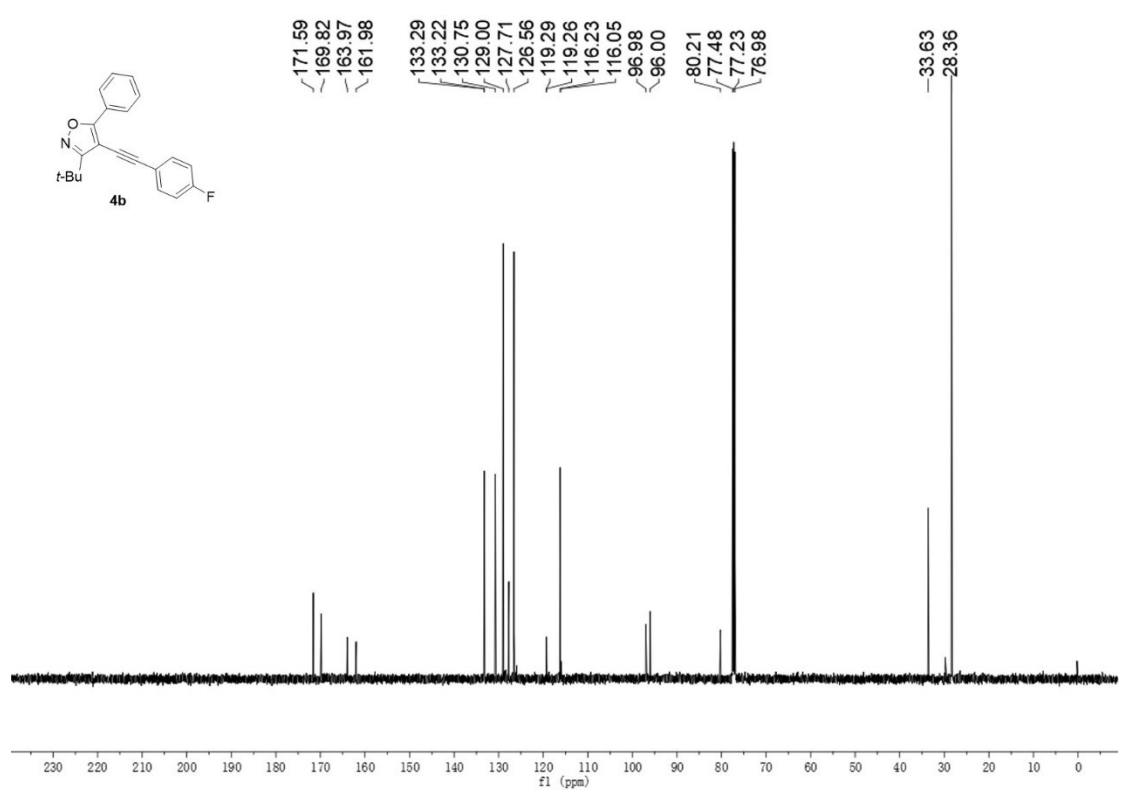
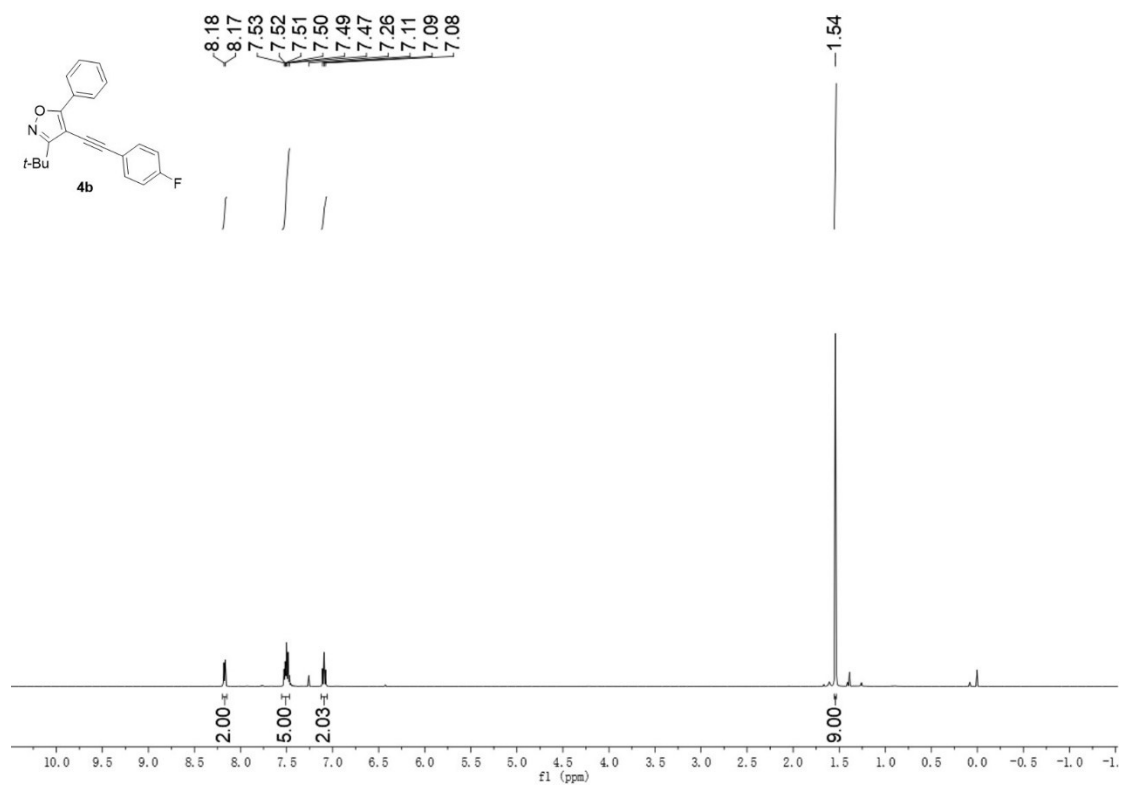


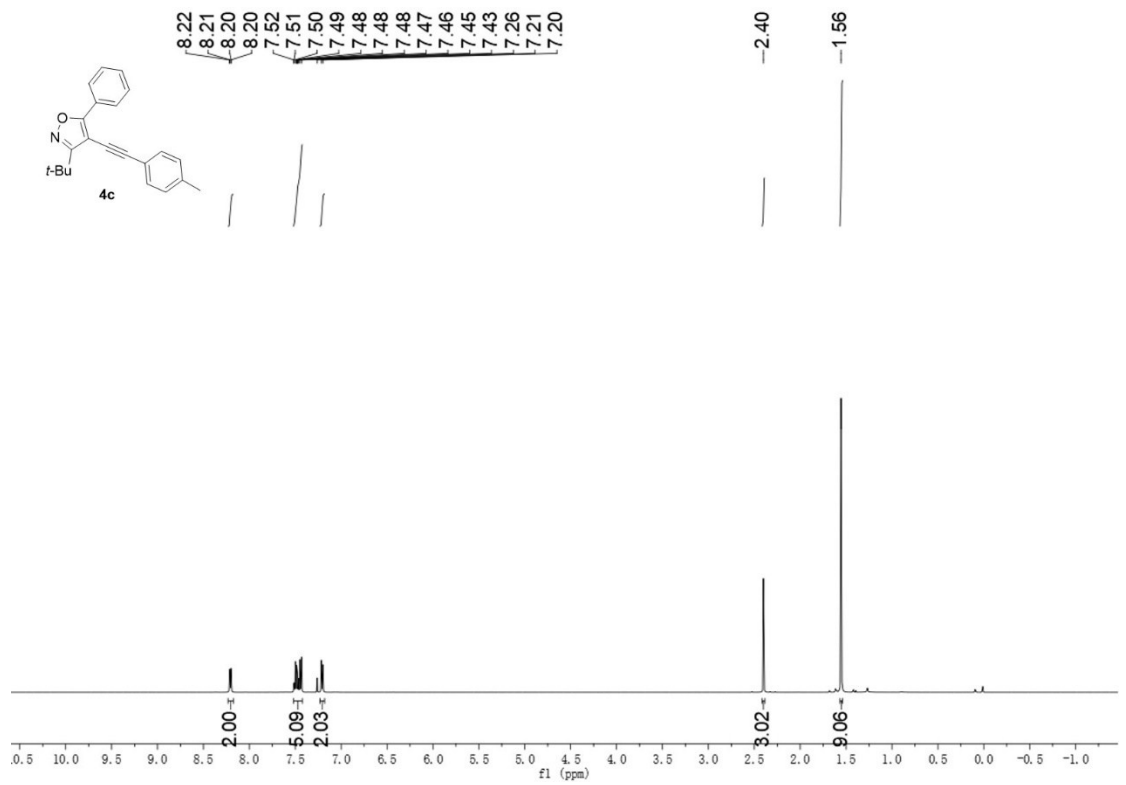
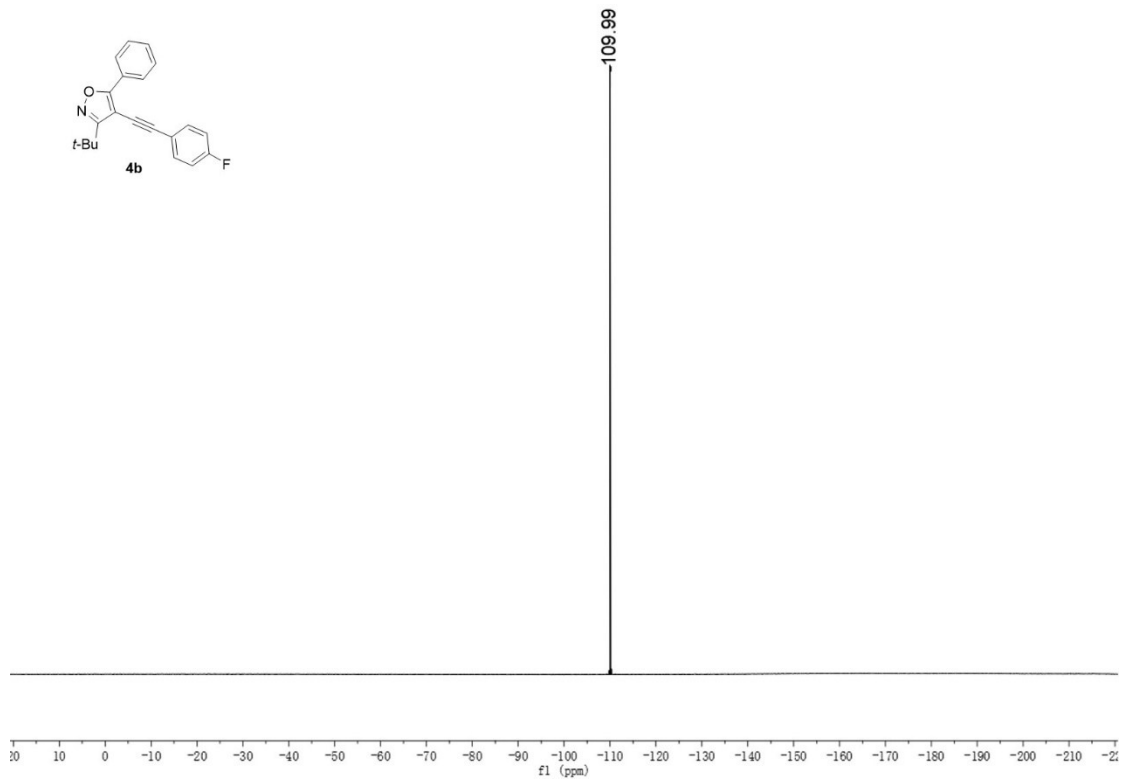


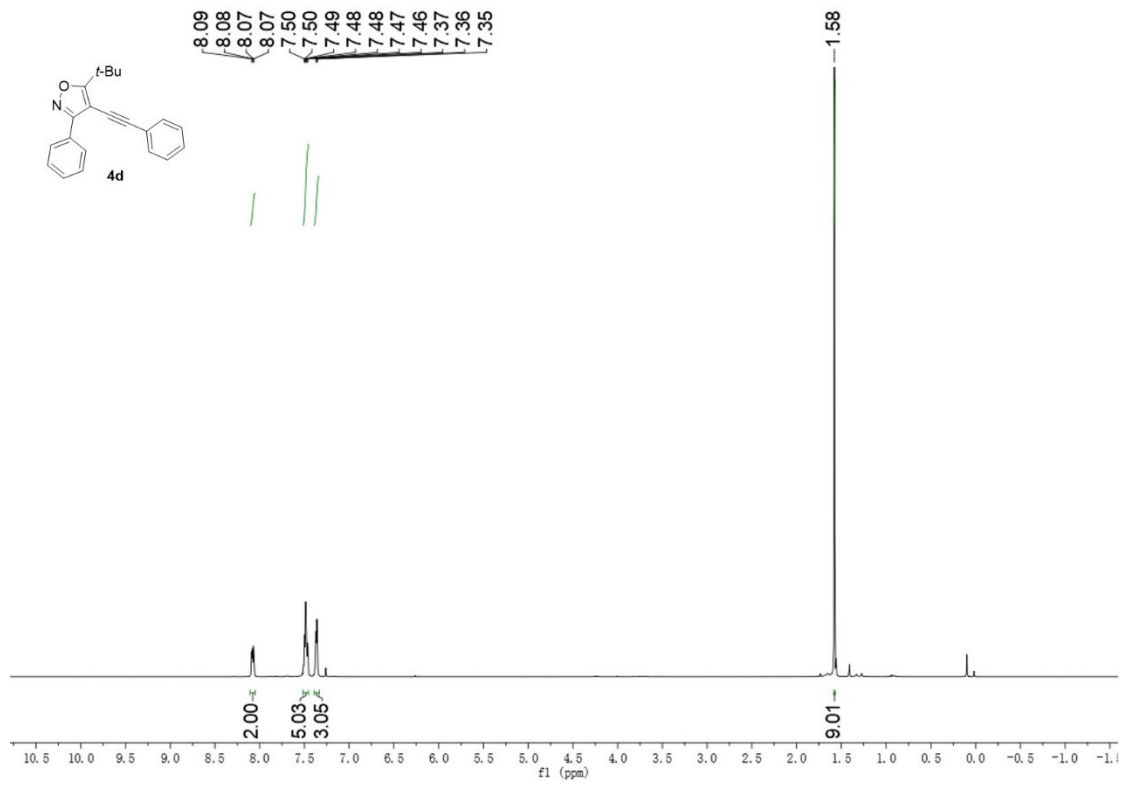
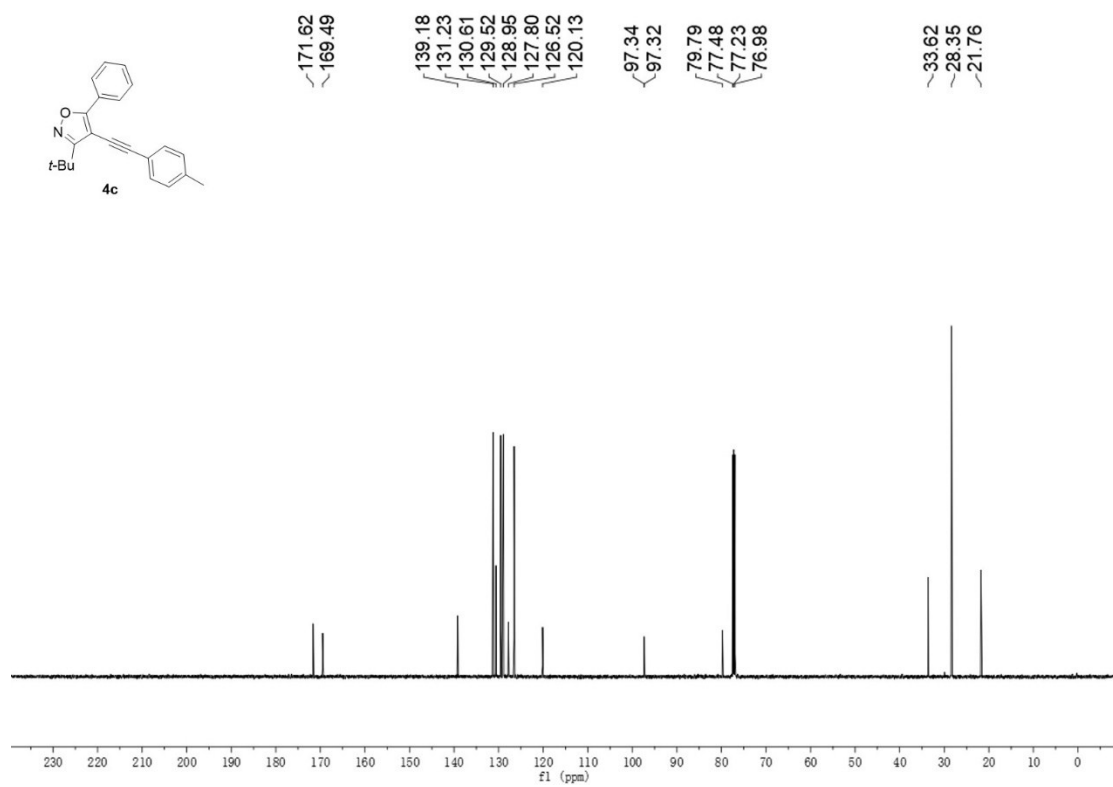


### 3. Copies of $^1\text{H}$ , $^{13}\text{C}$ and $^{19}\text{F}$ NMR spectra for targeted products 4a – 4u and 5a – 5l

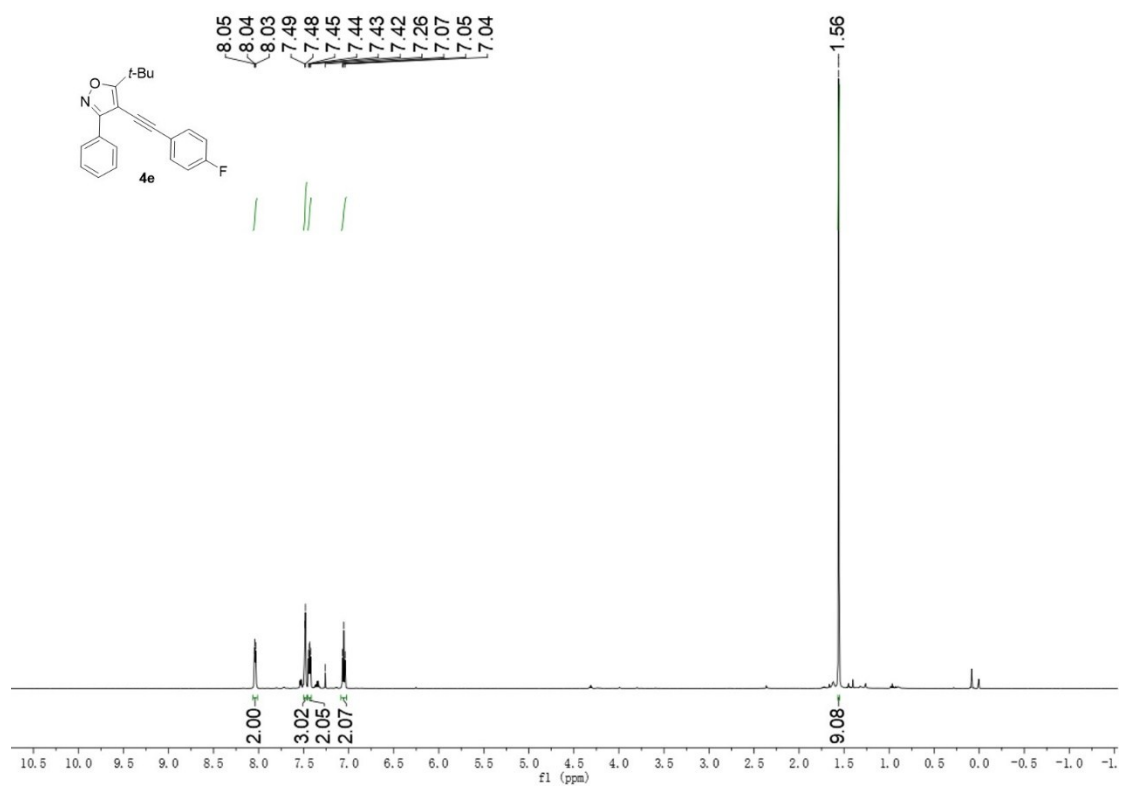
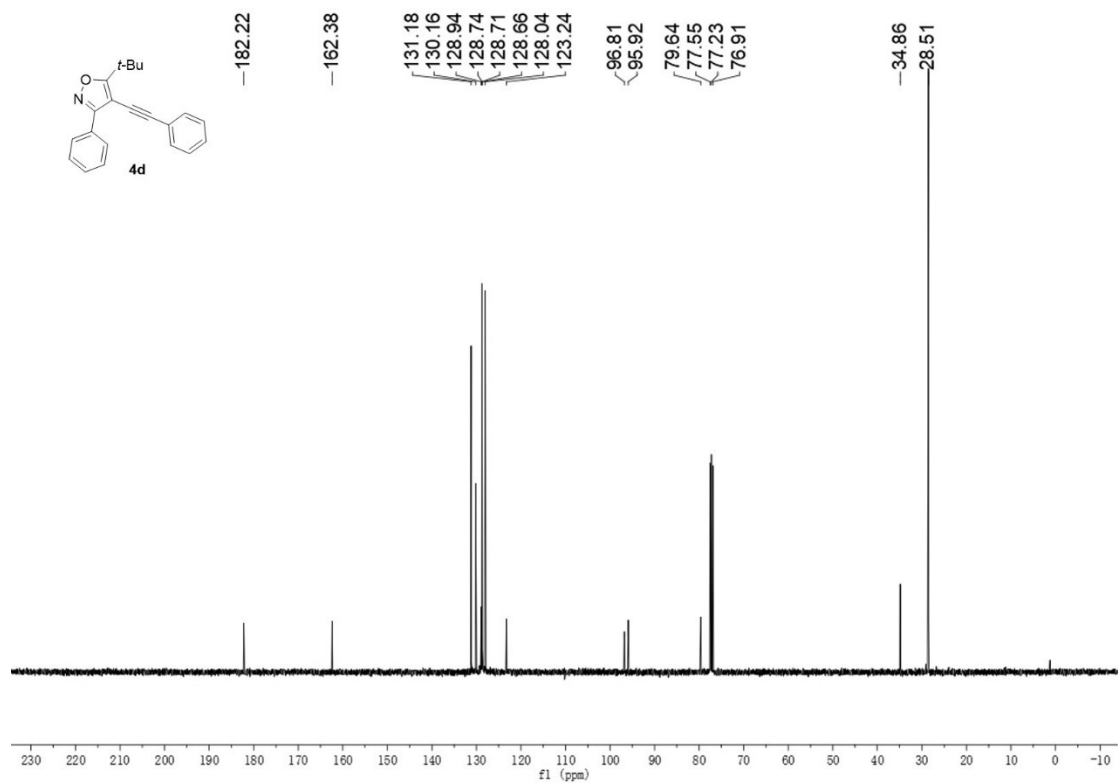


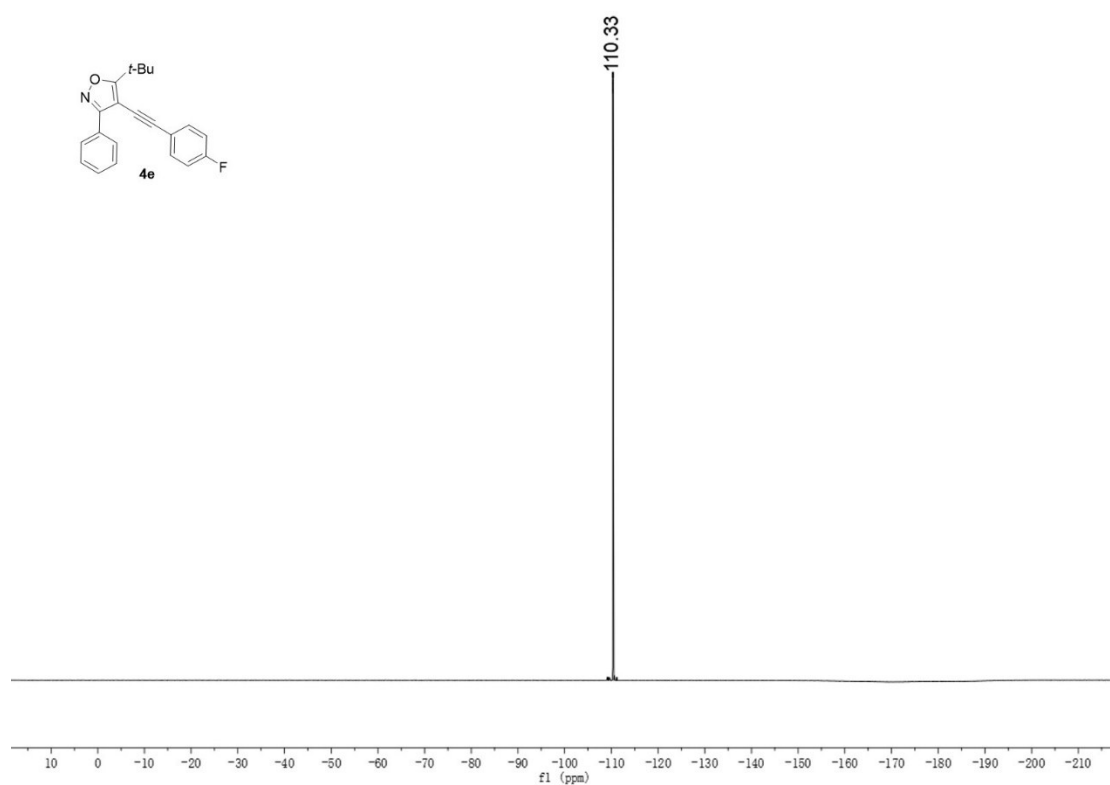
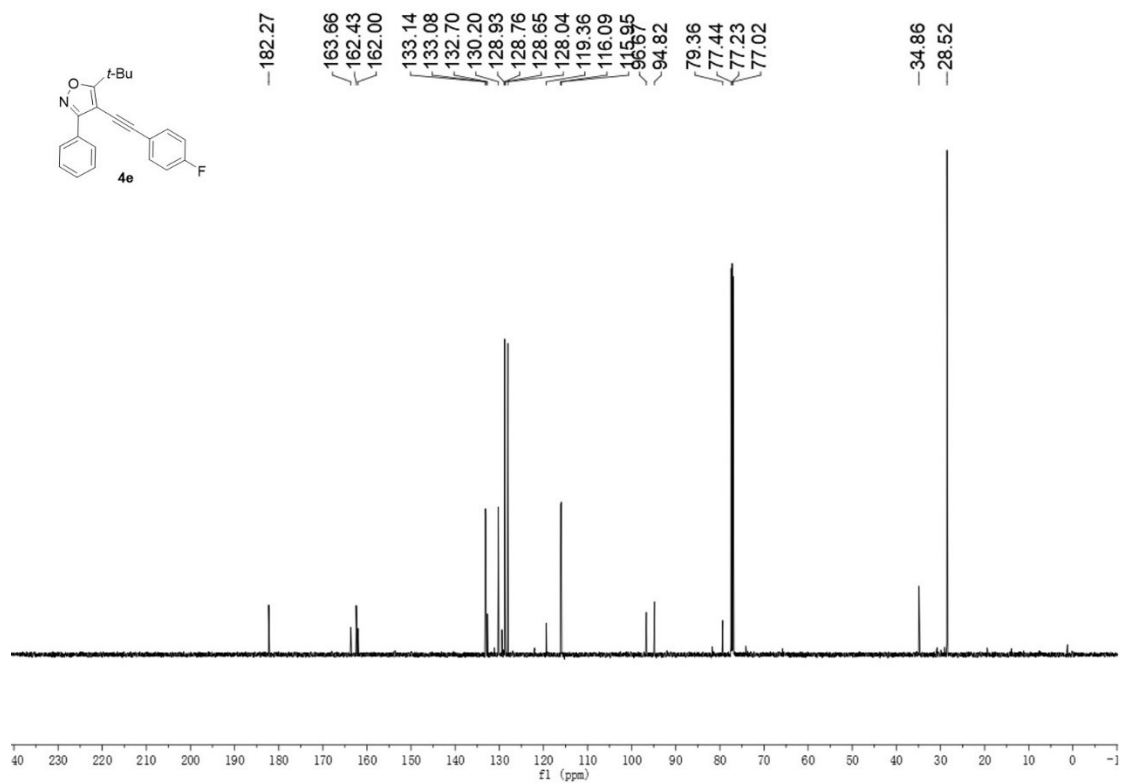


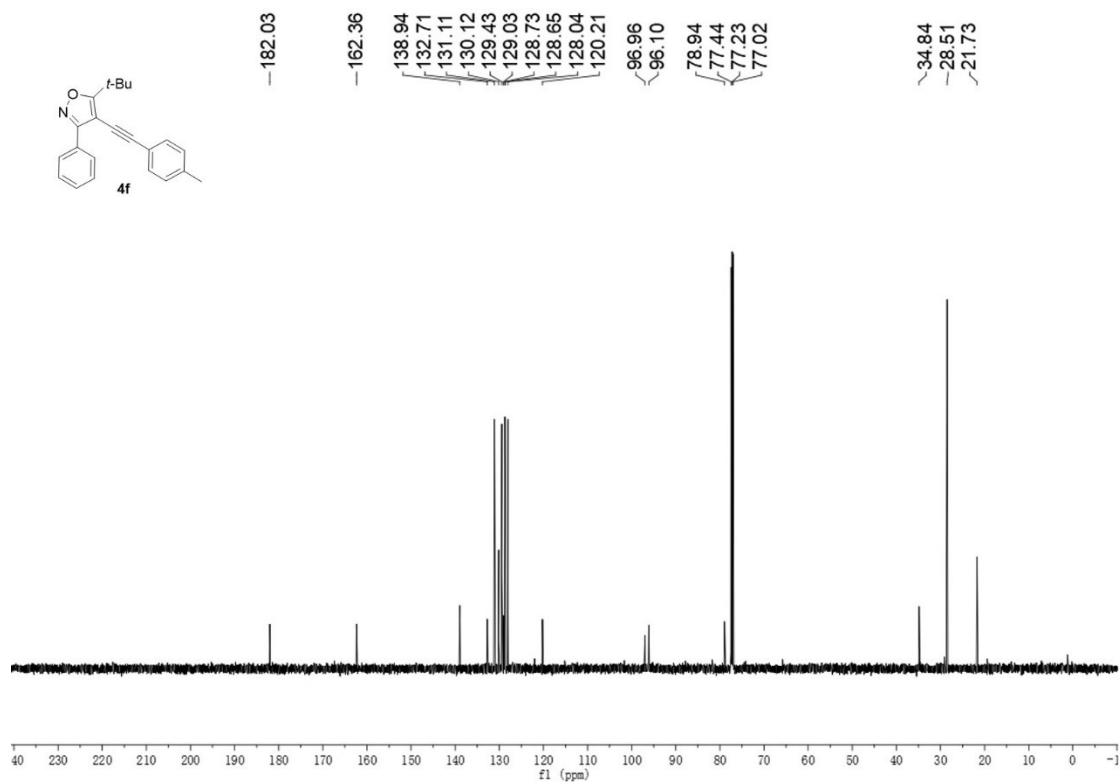
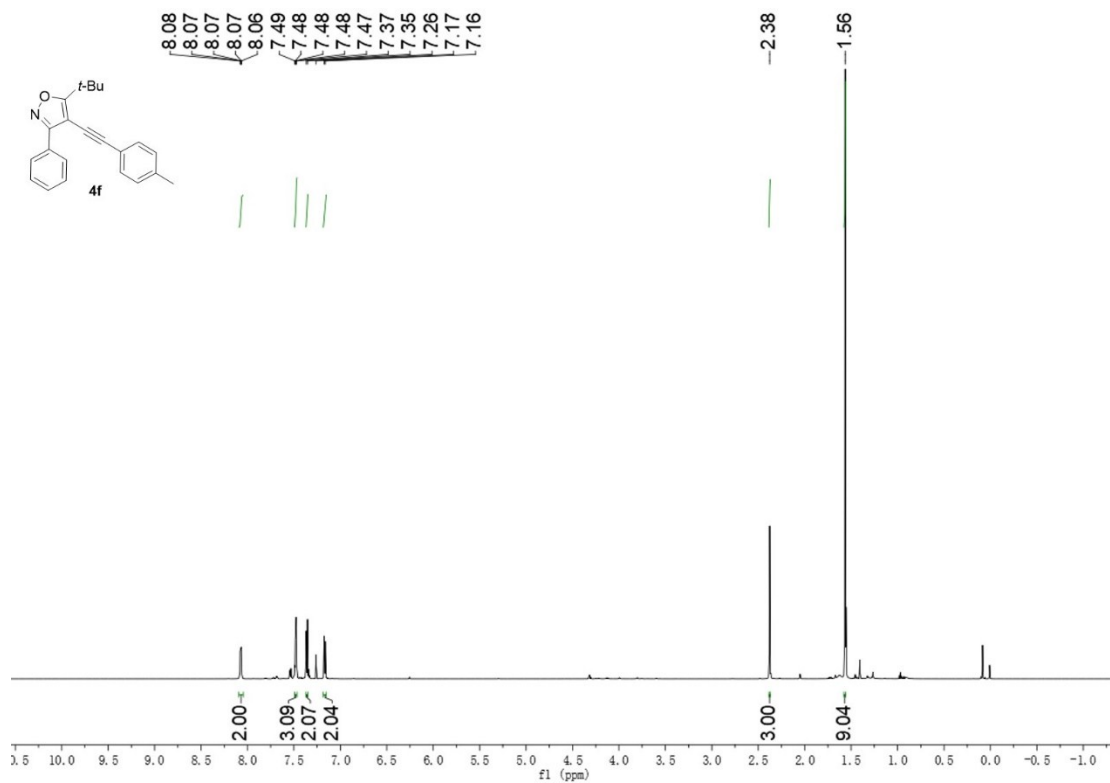


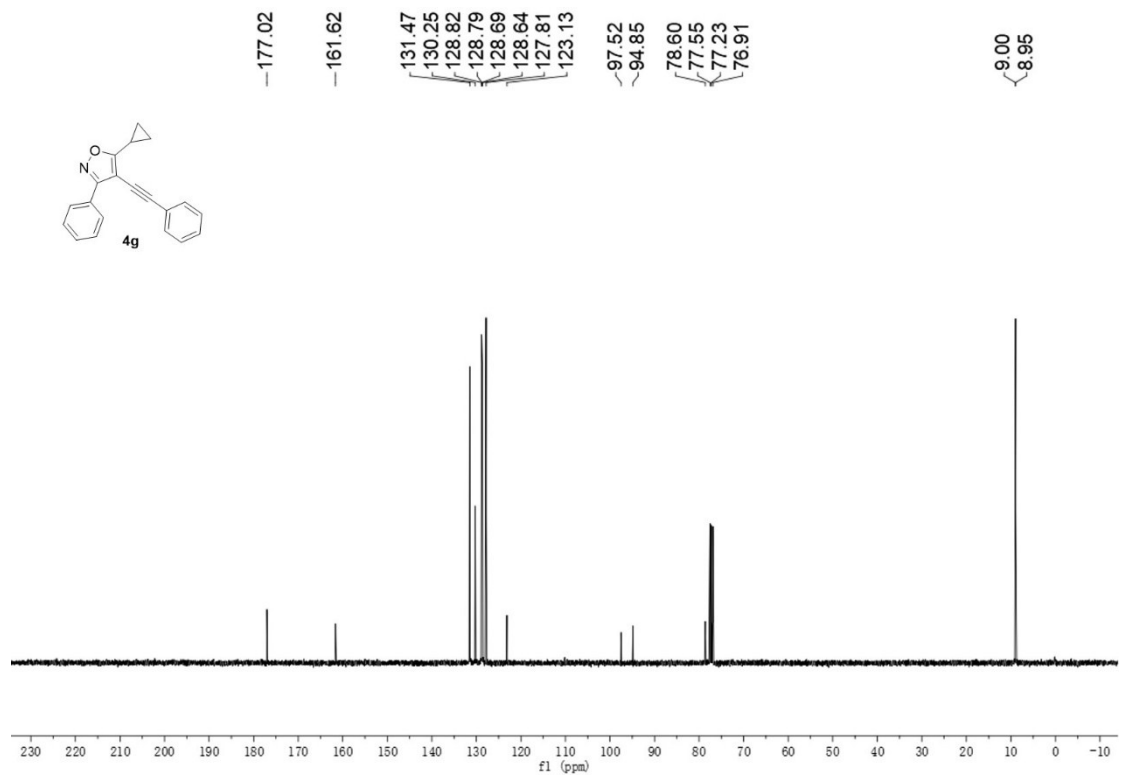
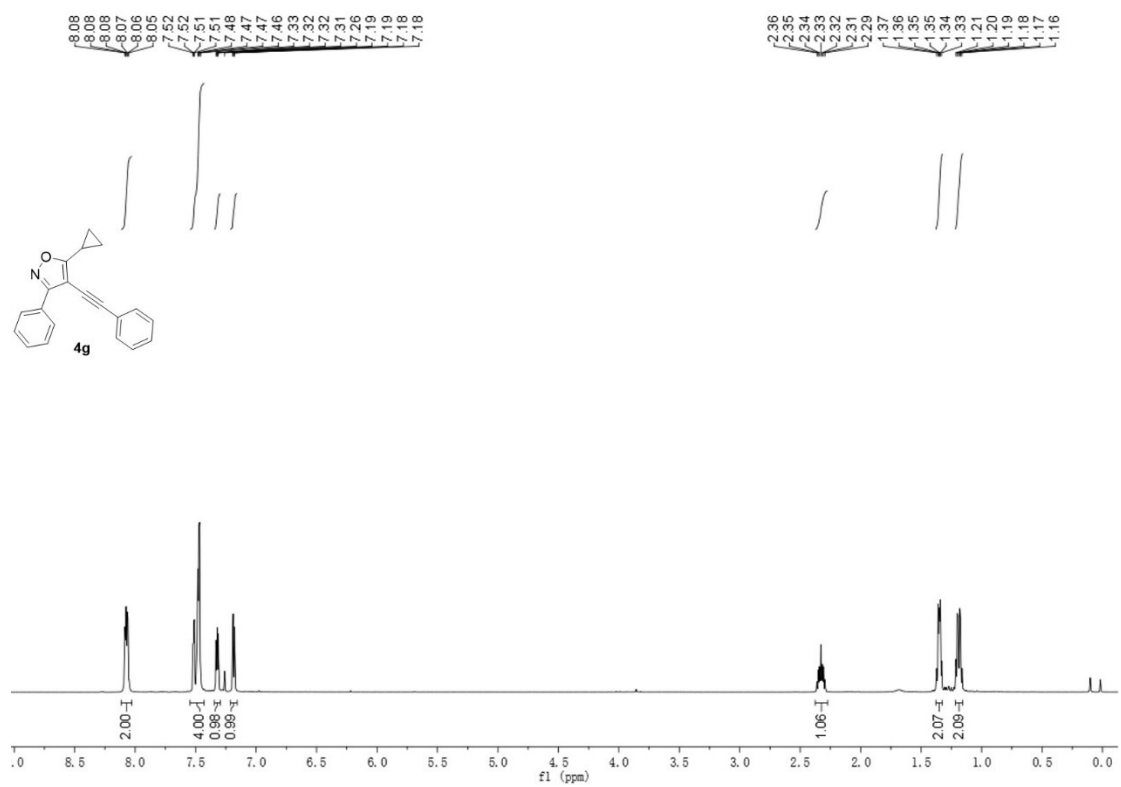


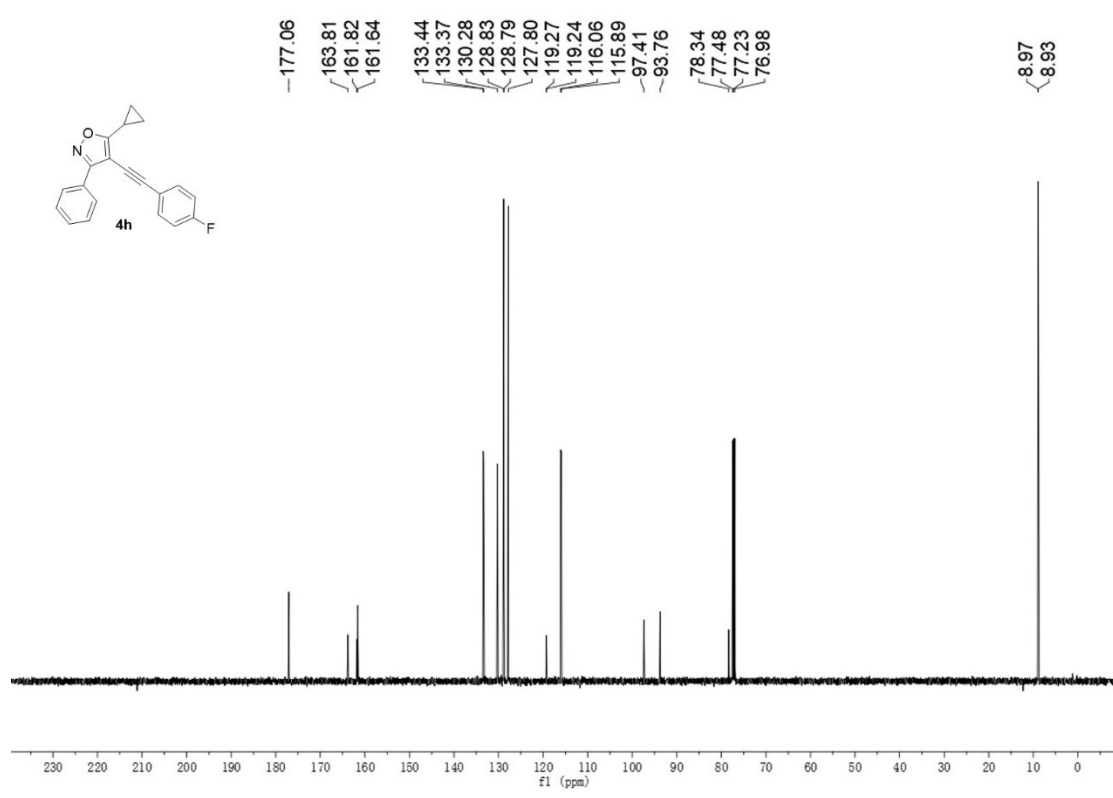
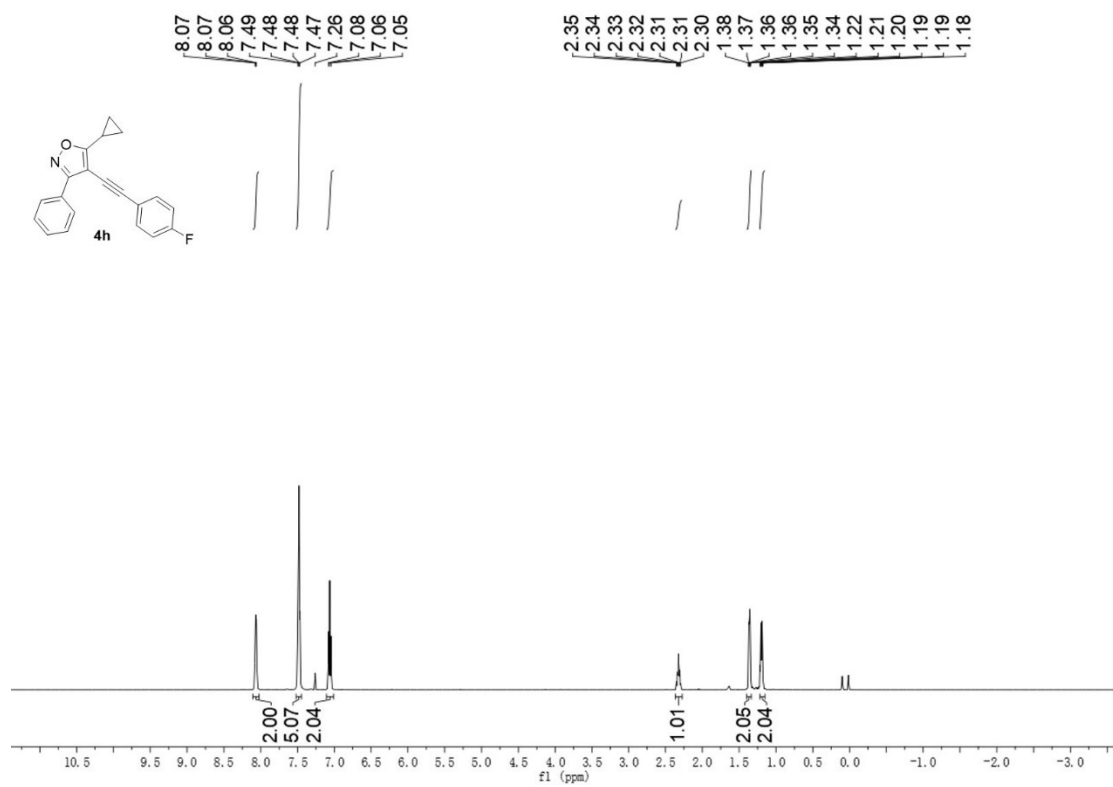


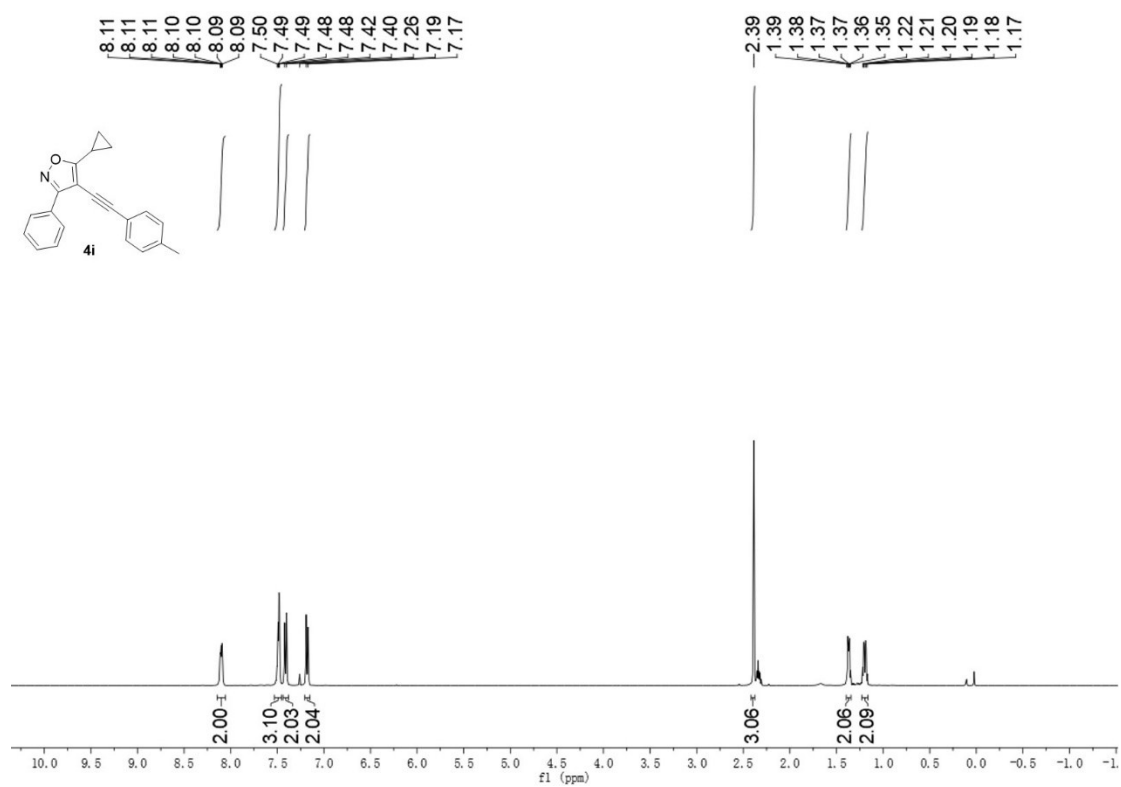
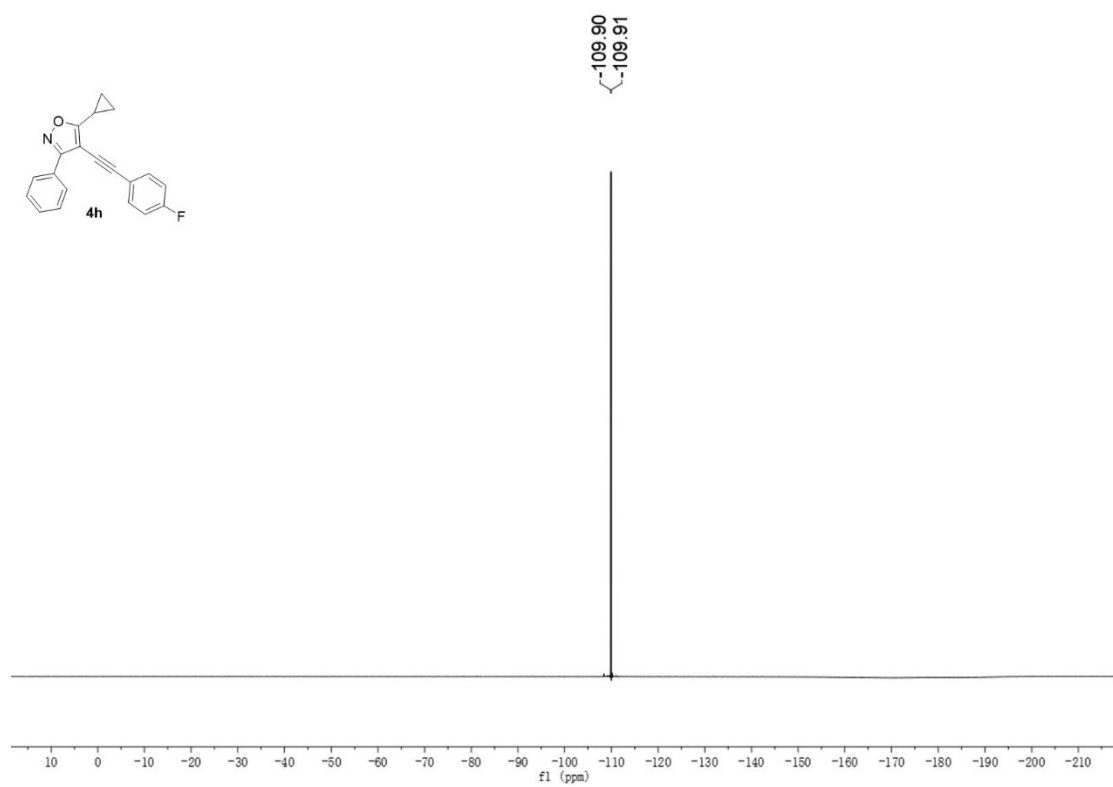


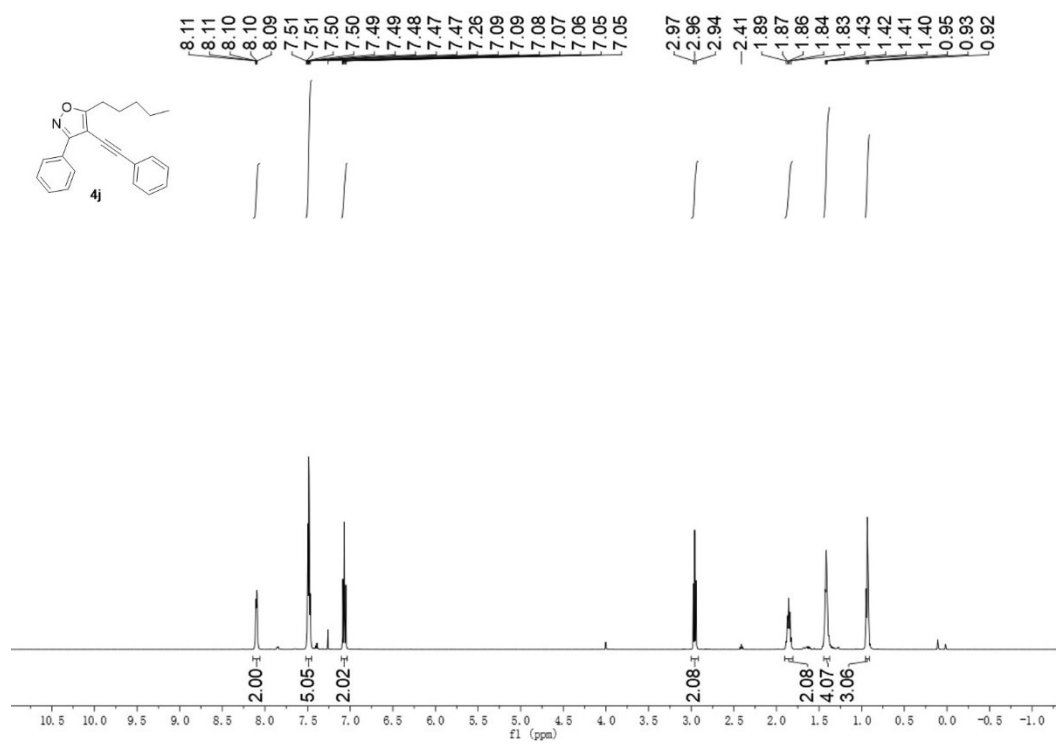
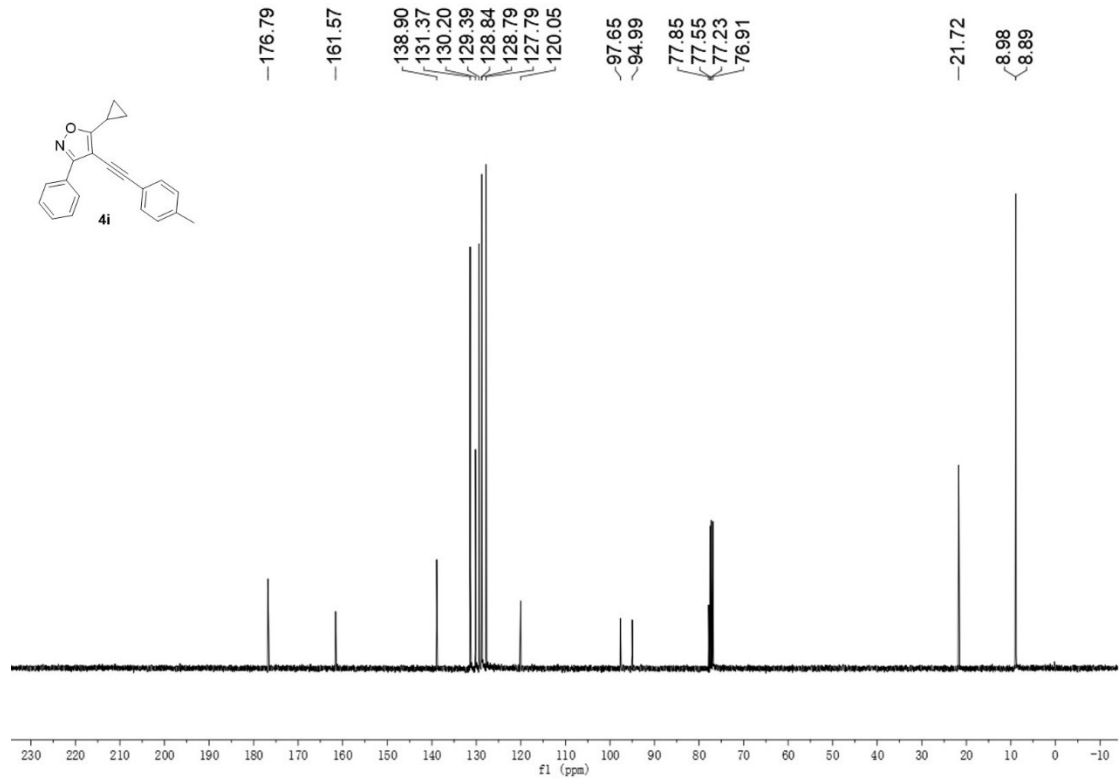


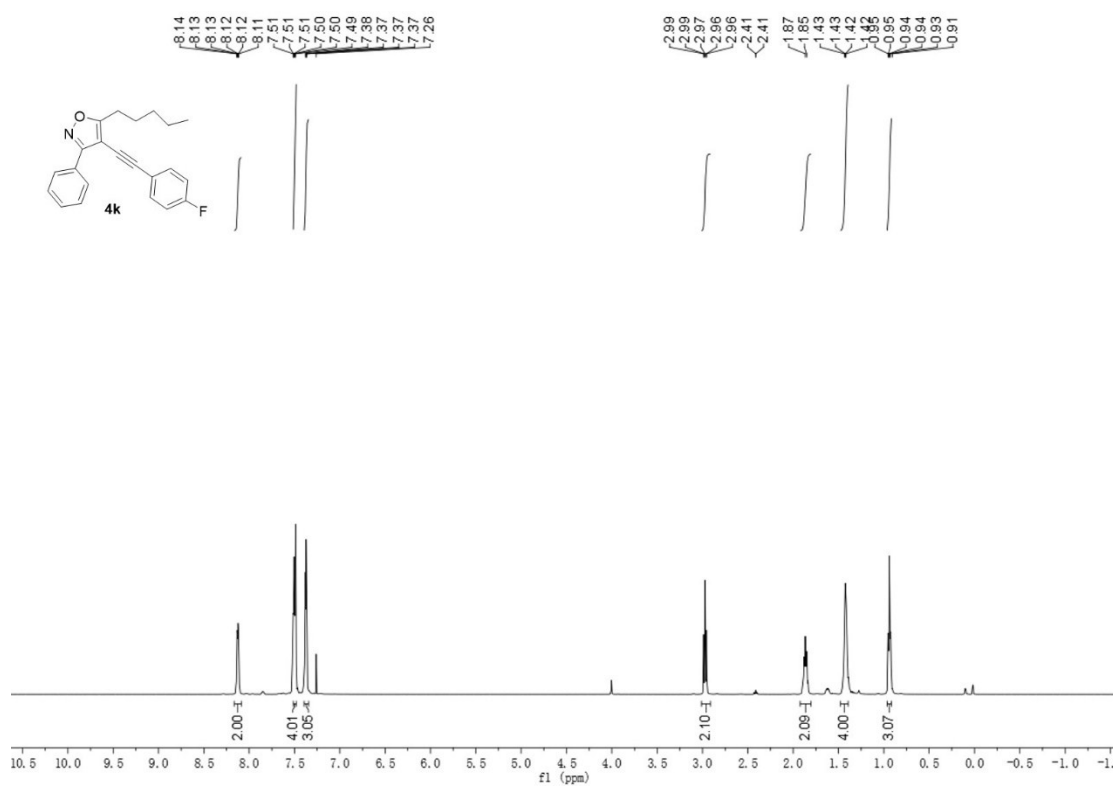
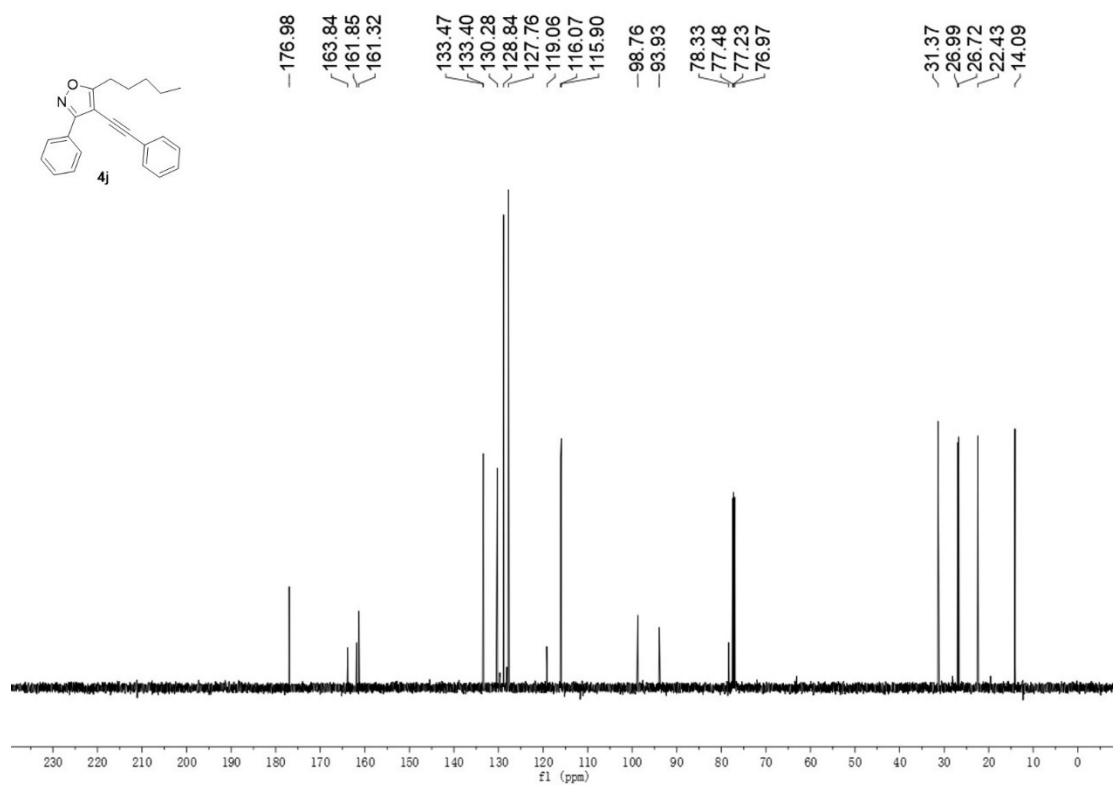




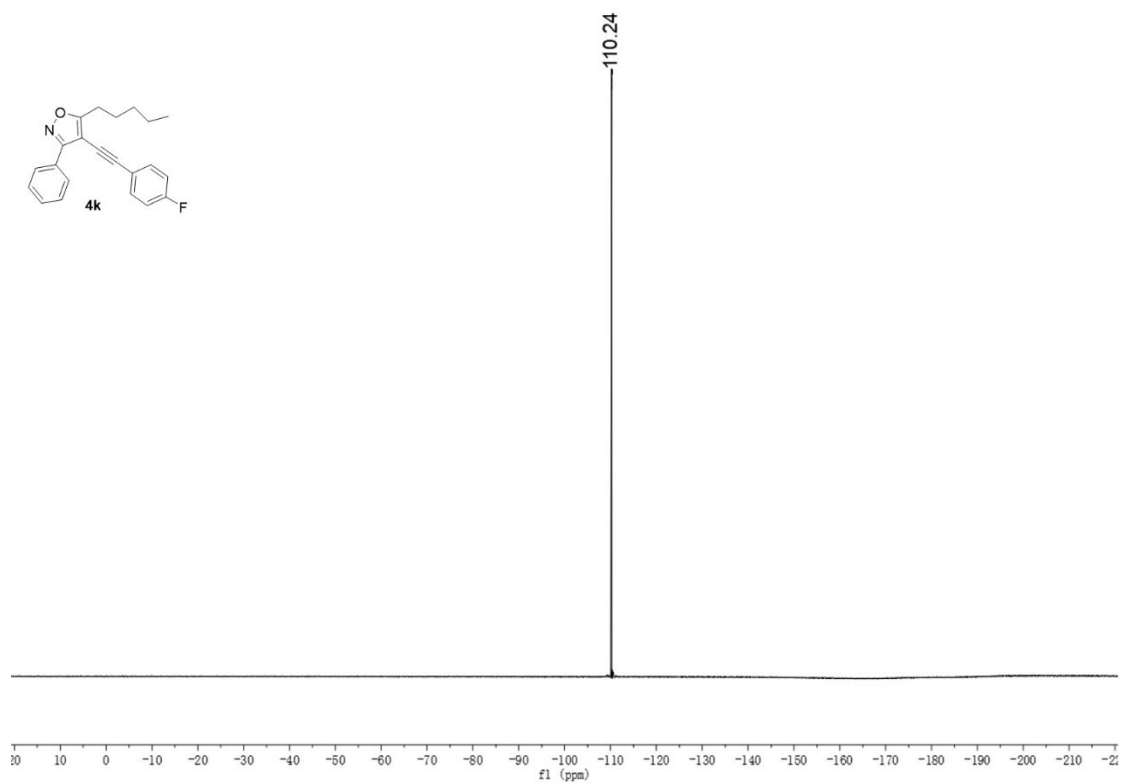
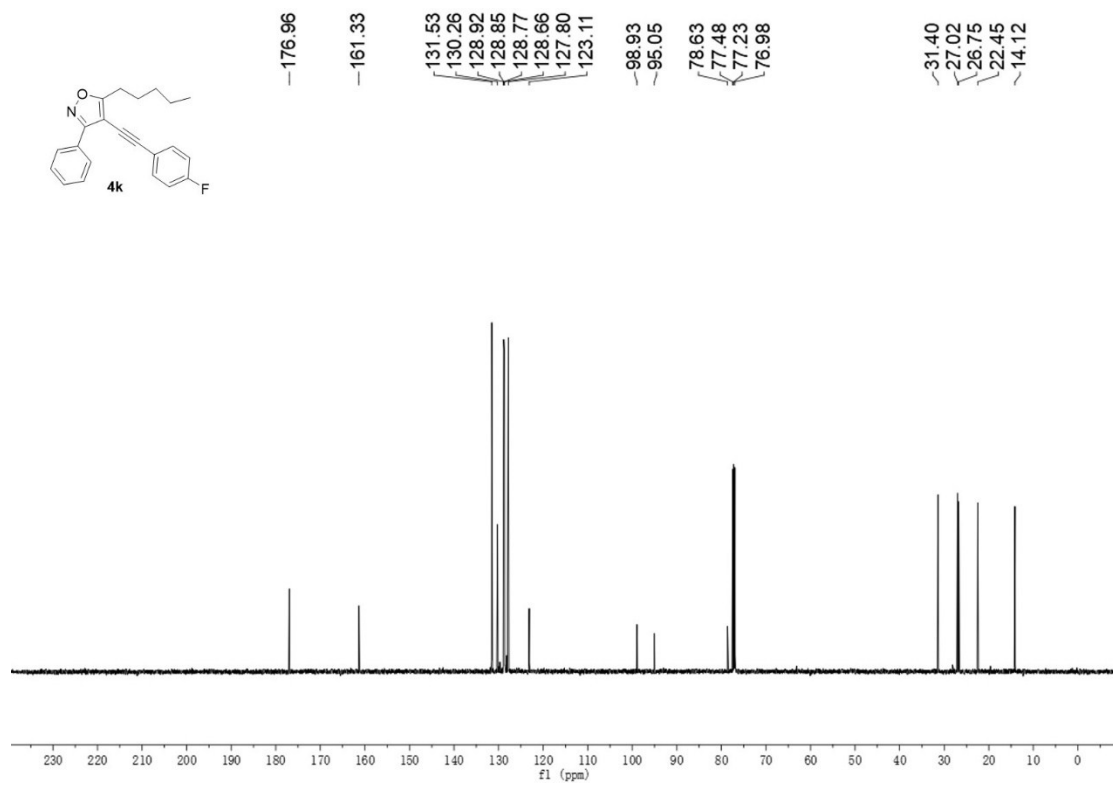


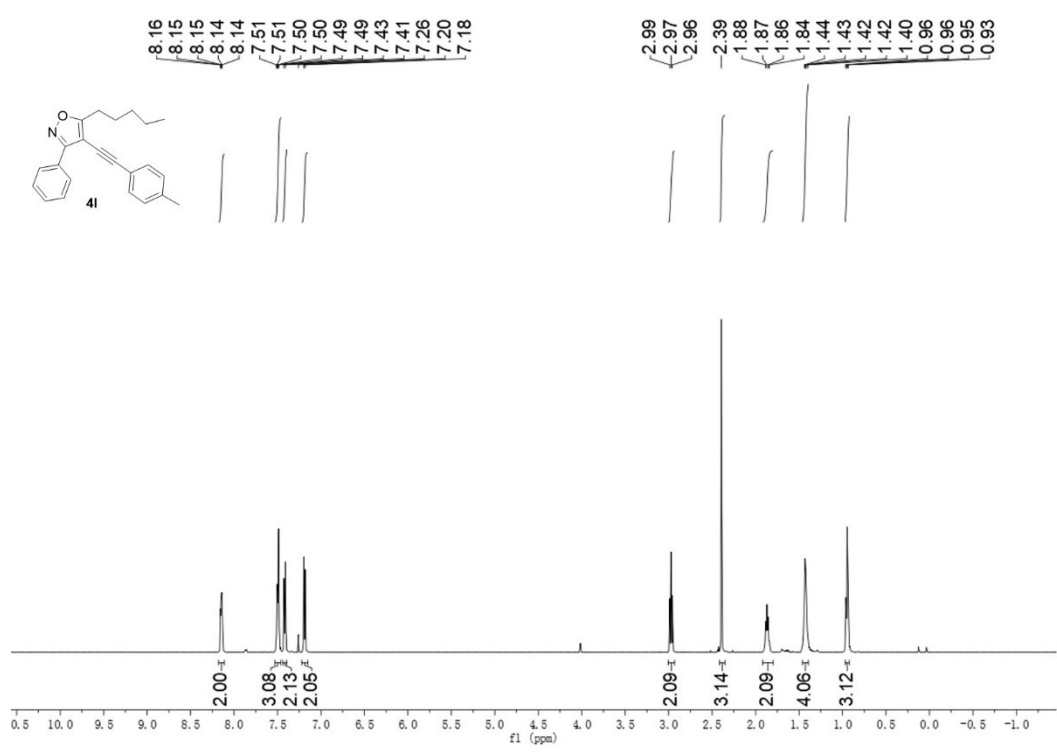
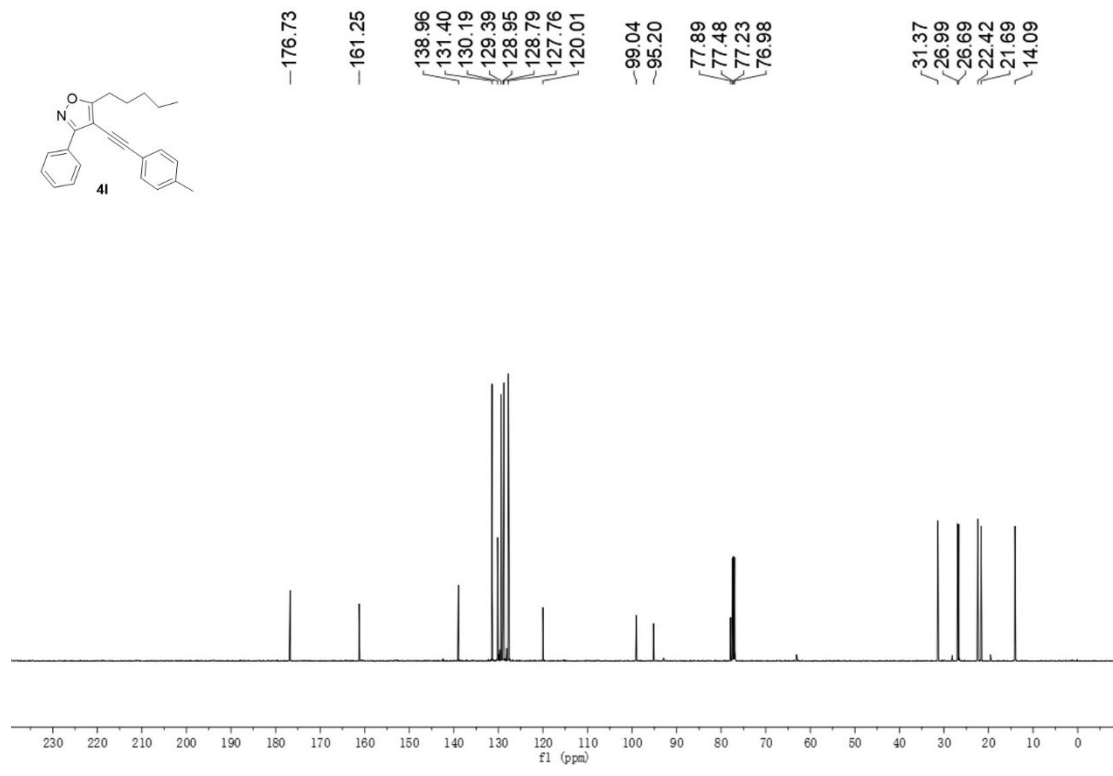


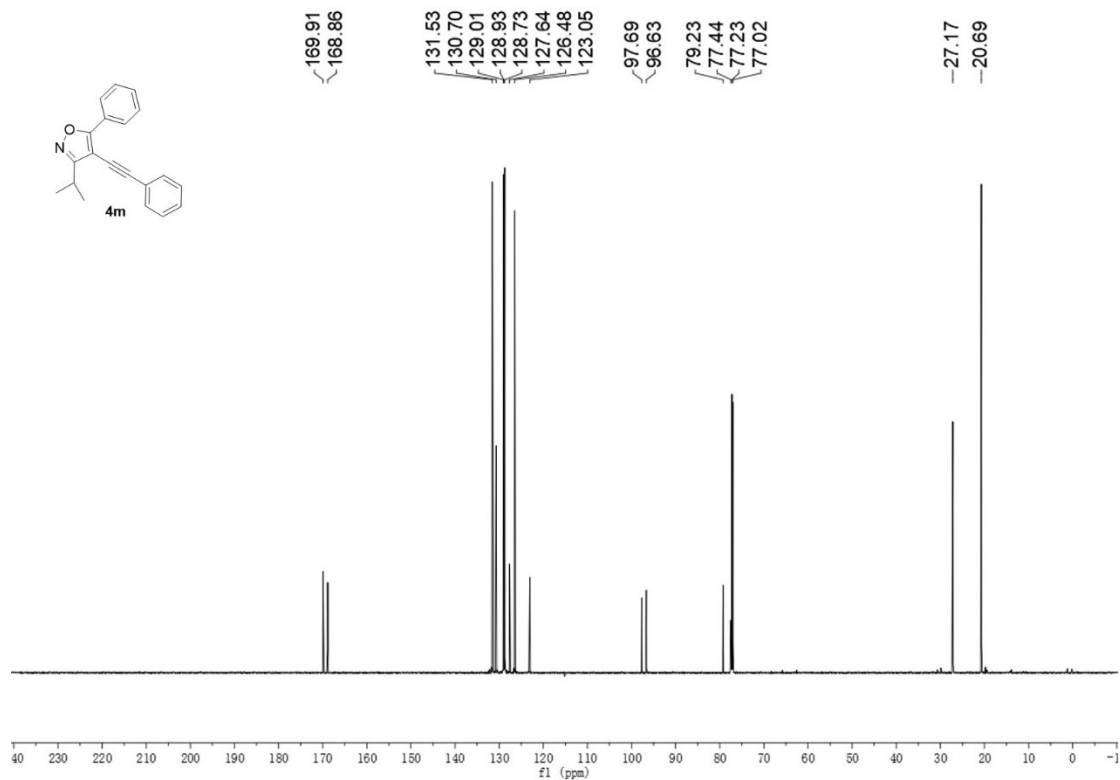
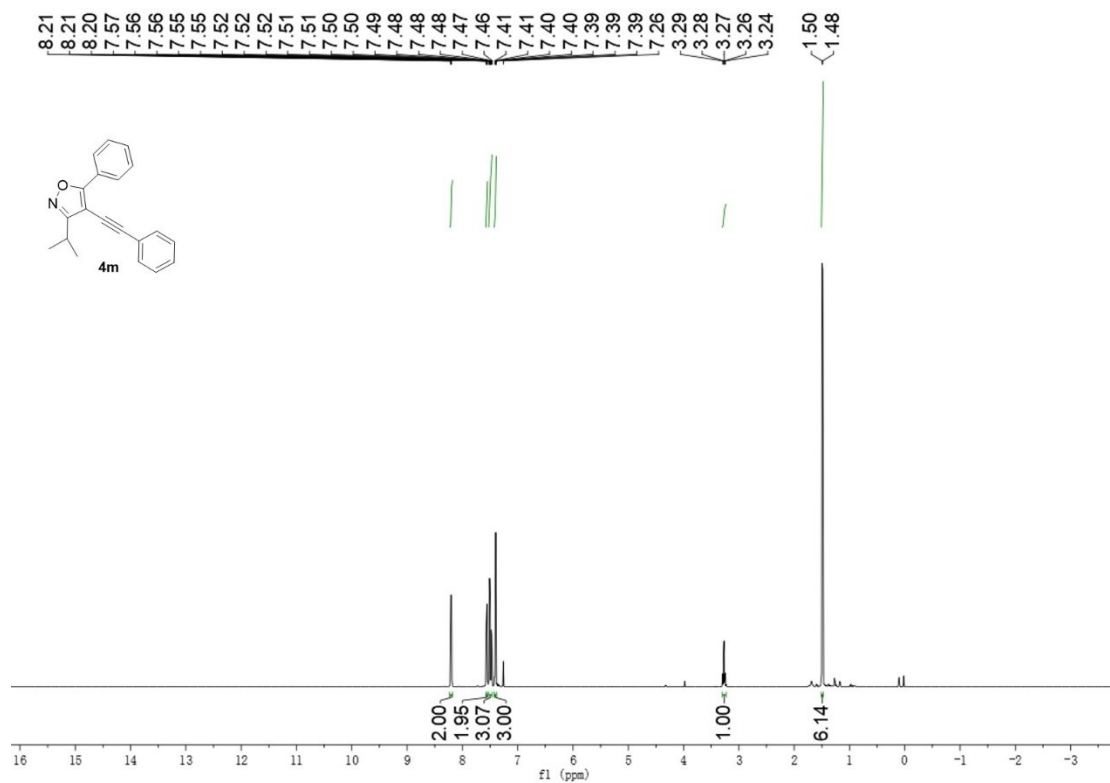


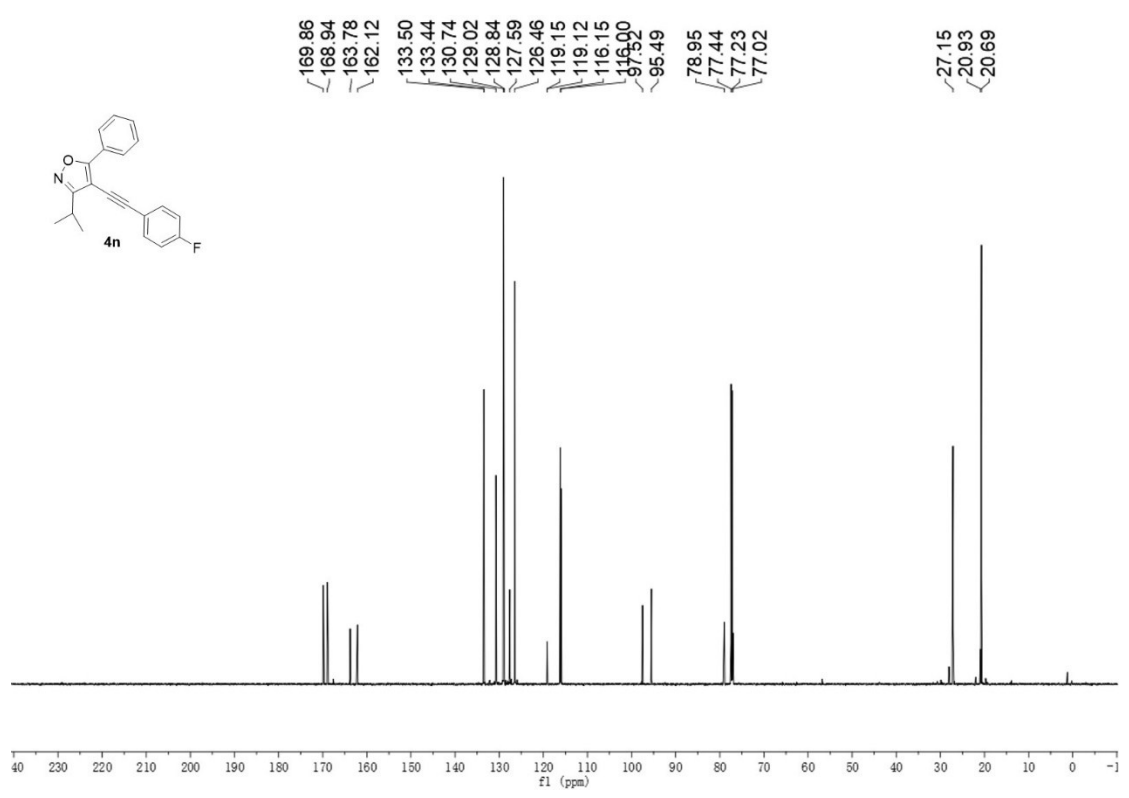
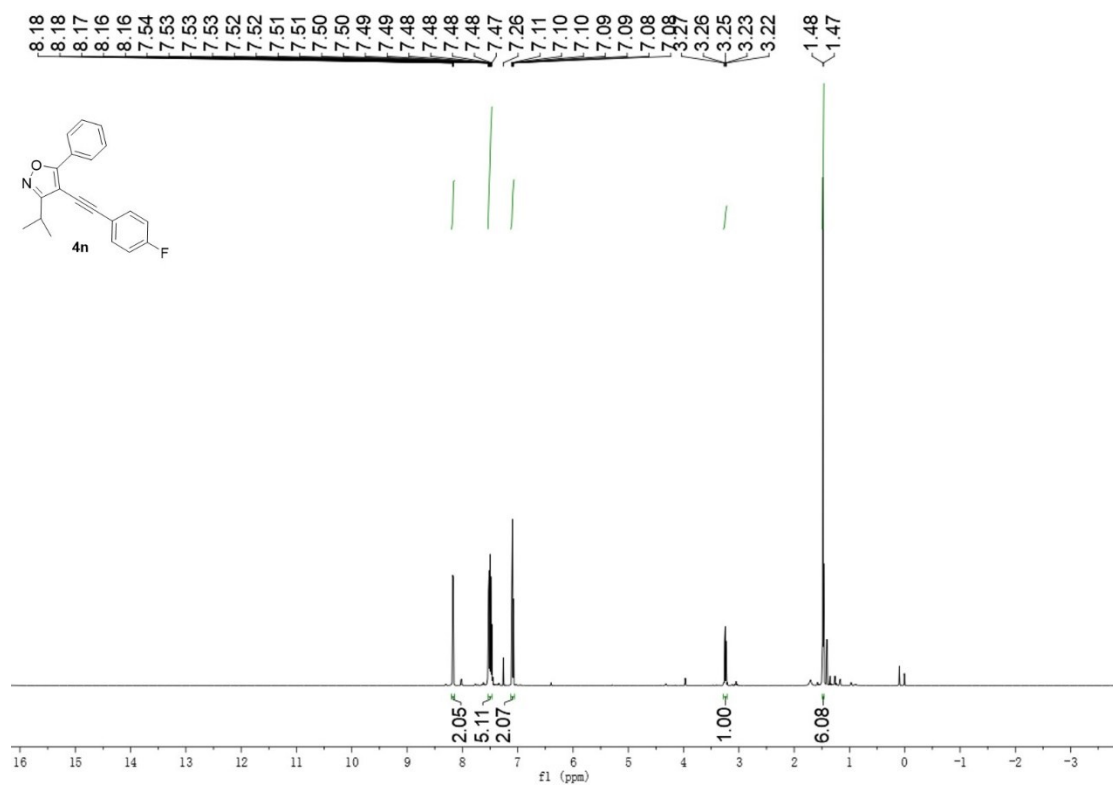


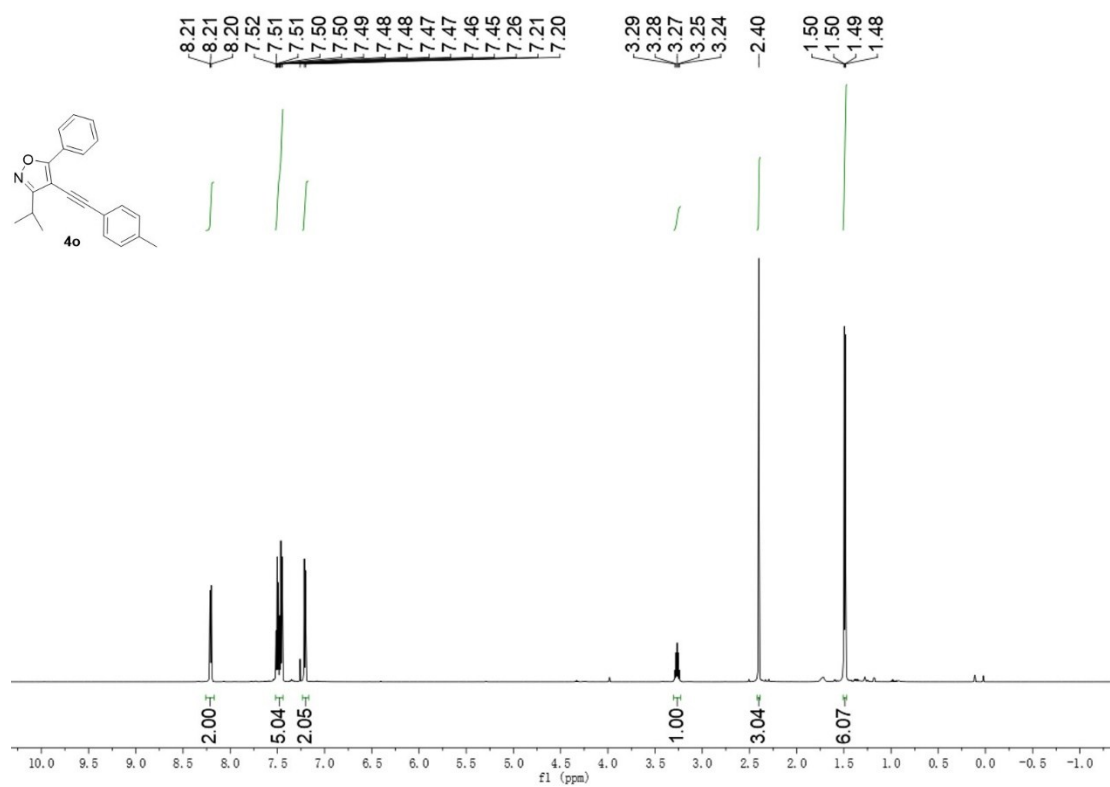
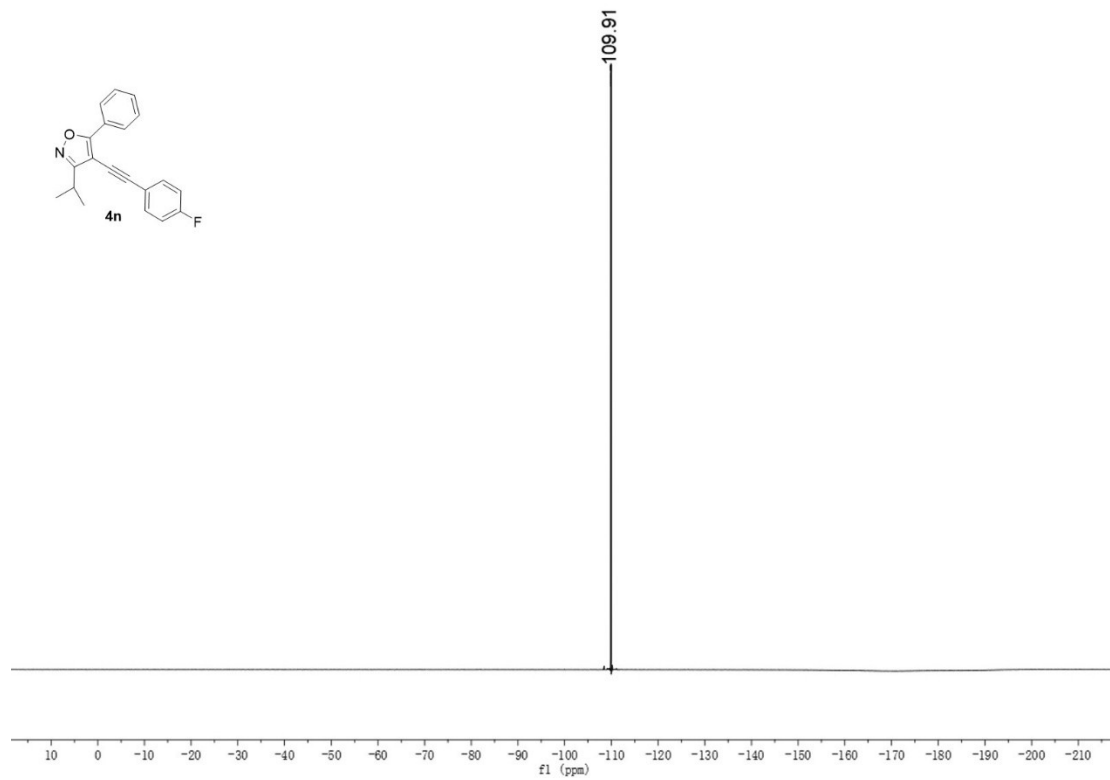


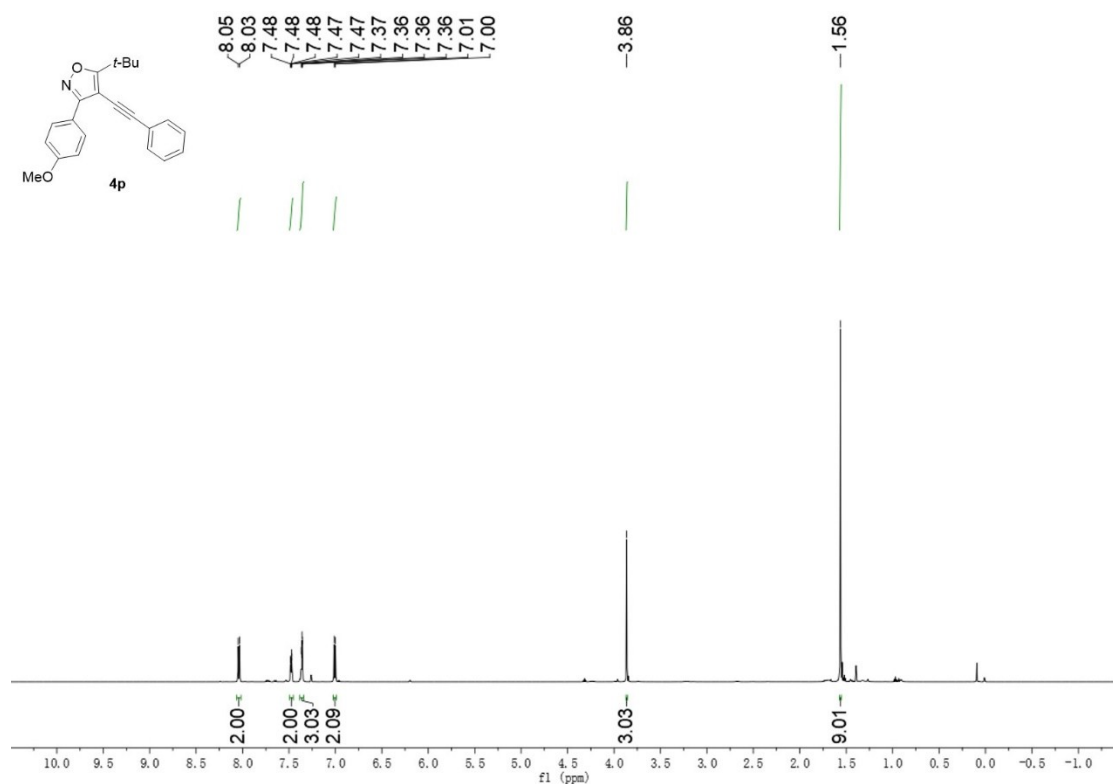
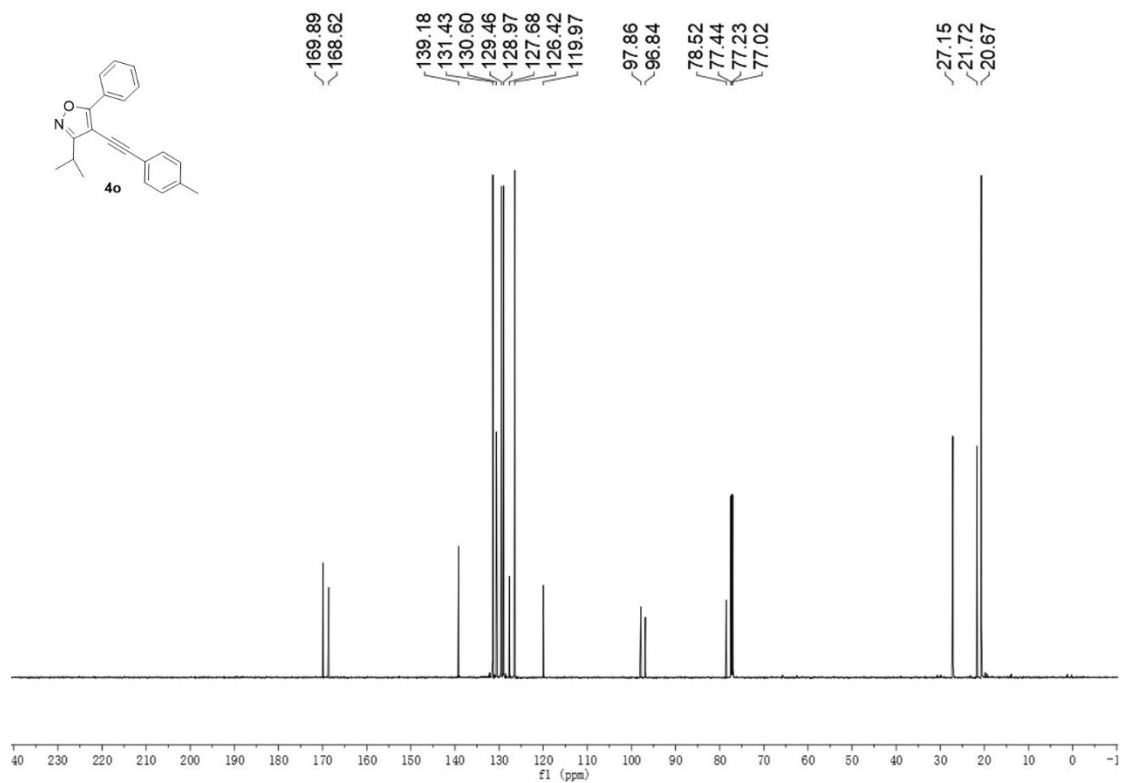


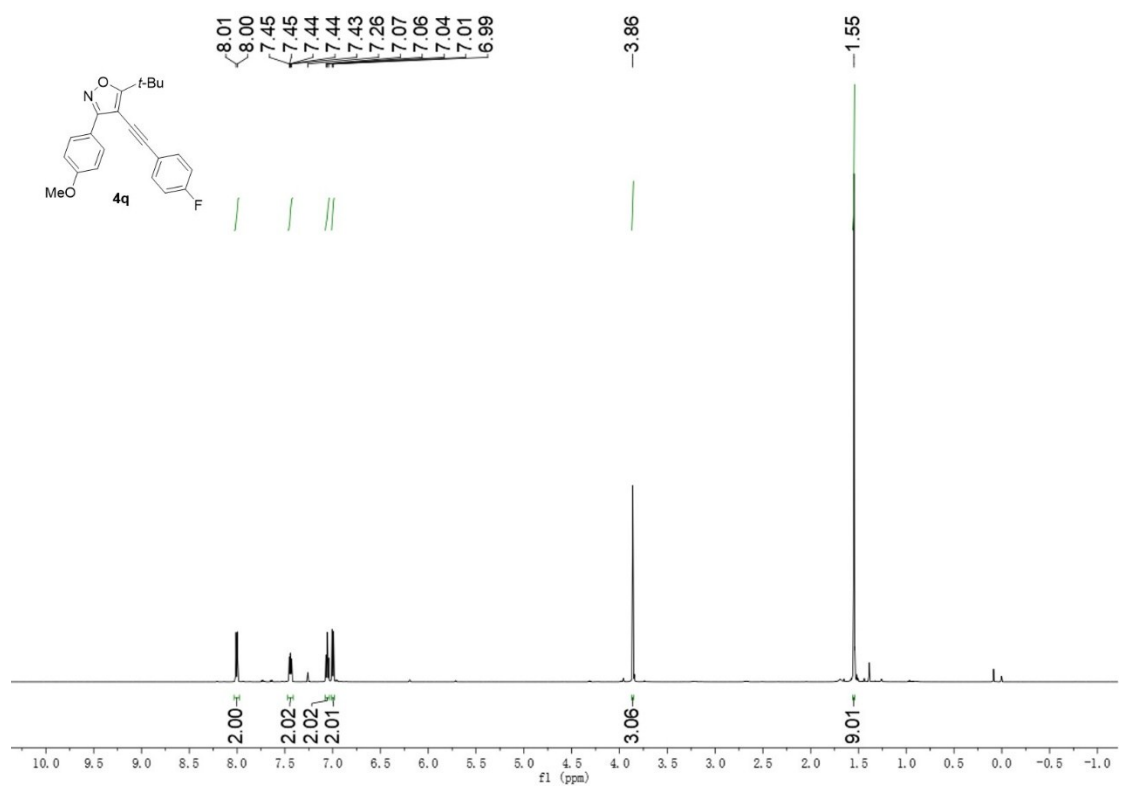
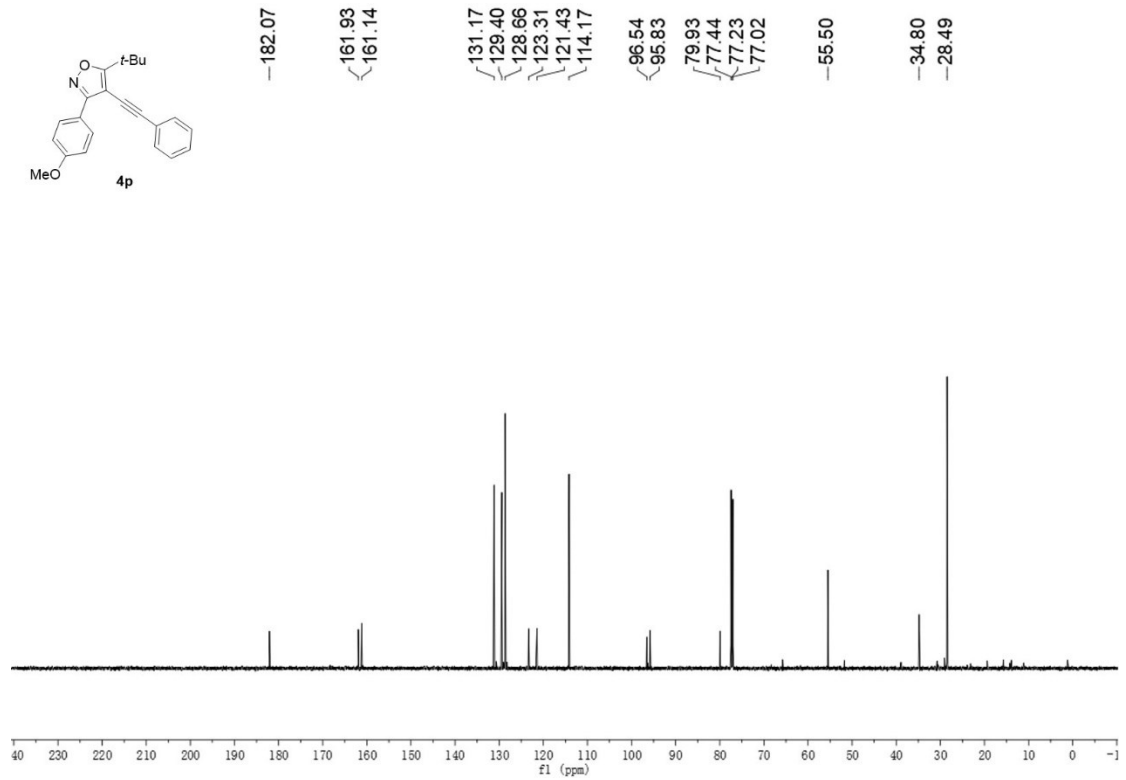


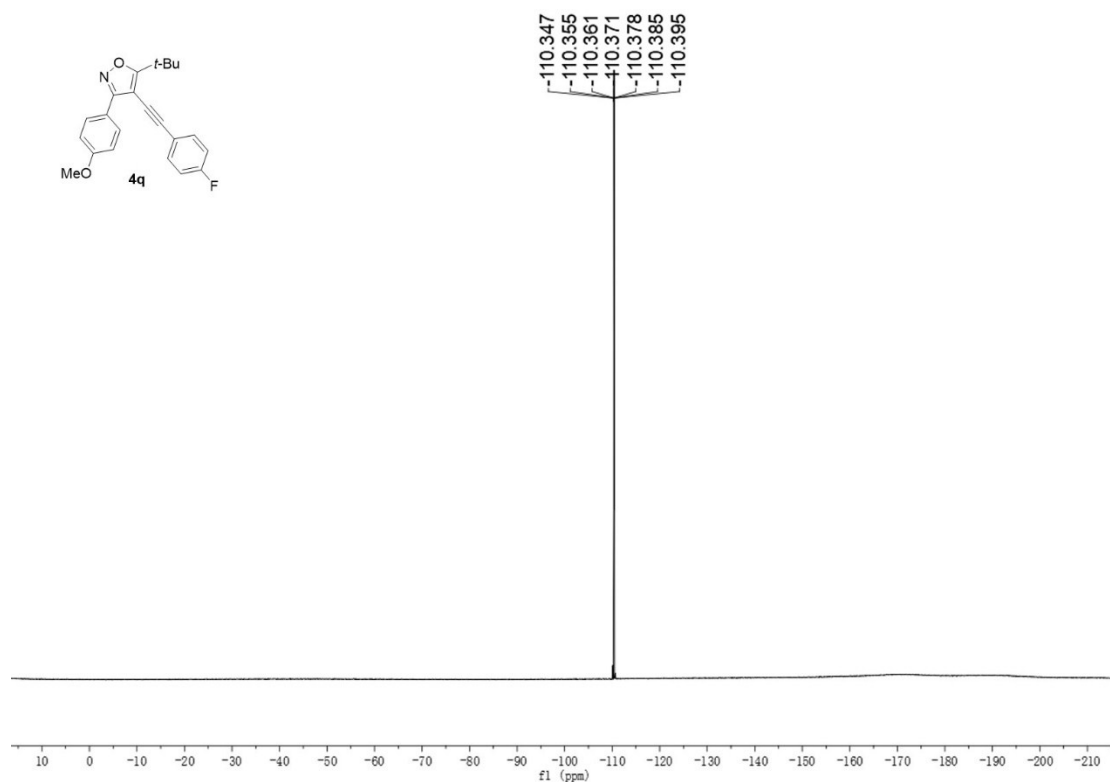
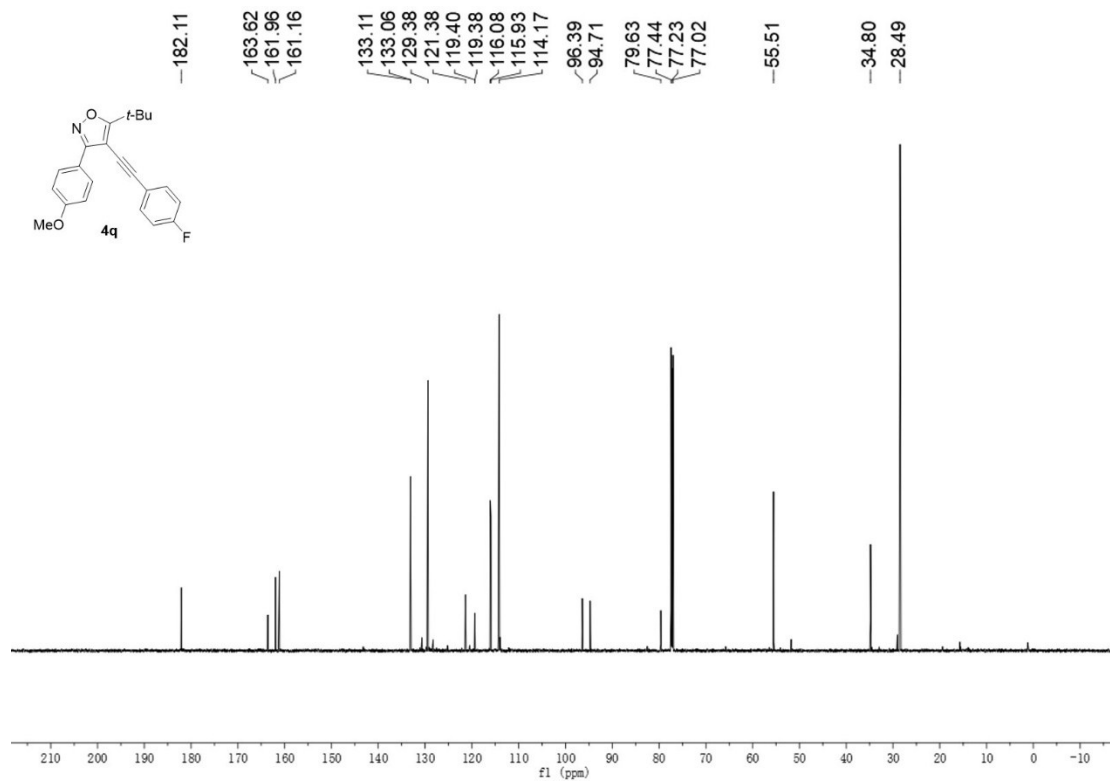




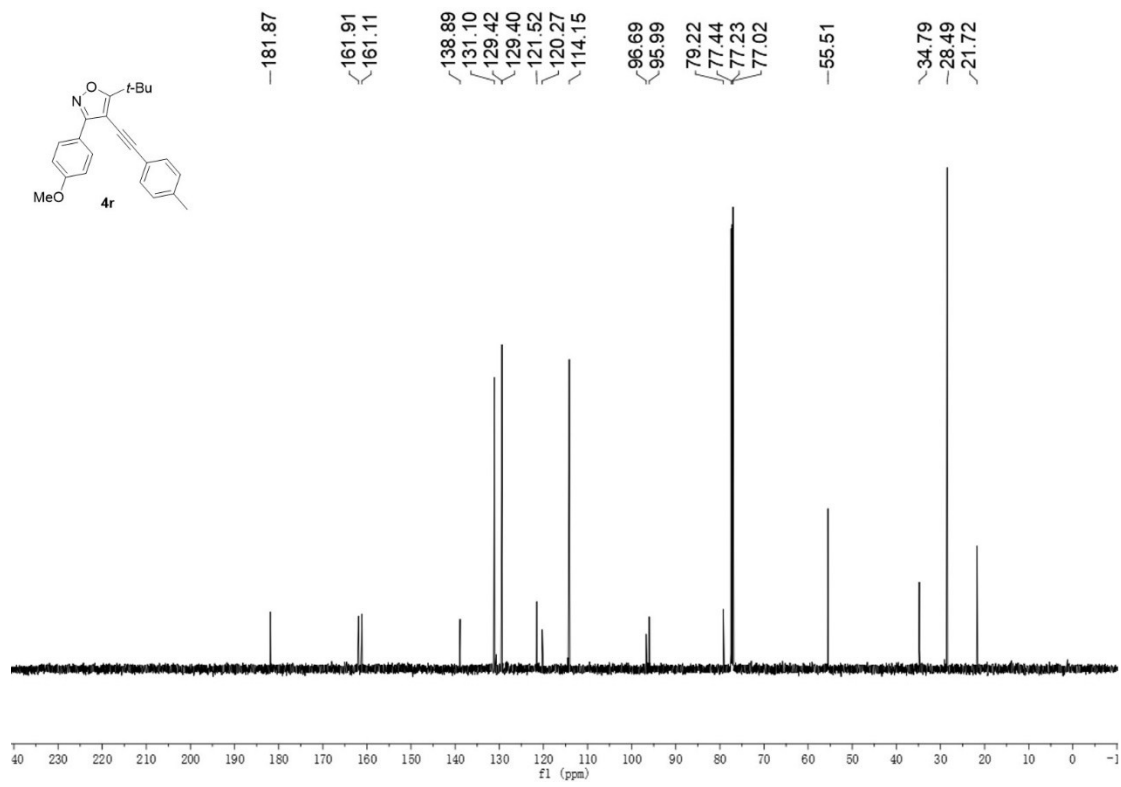
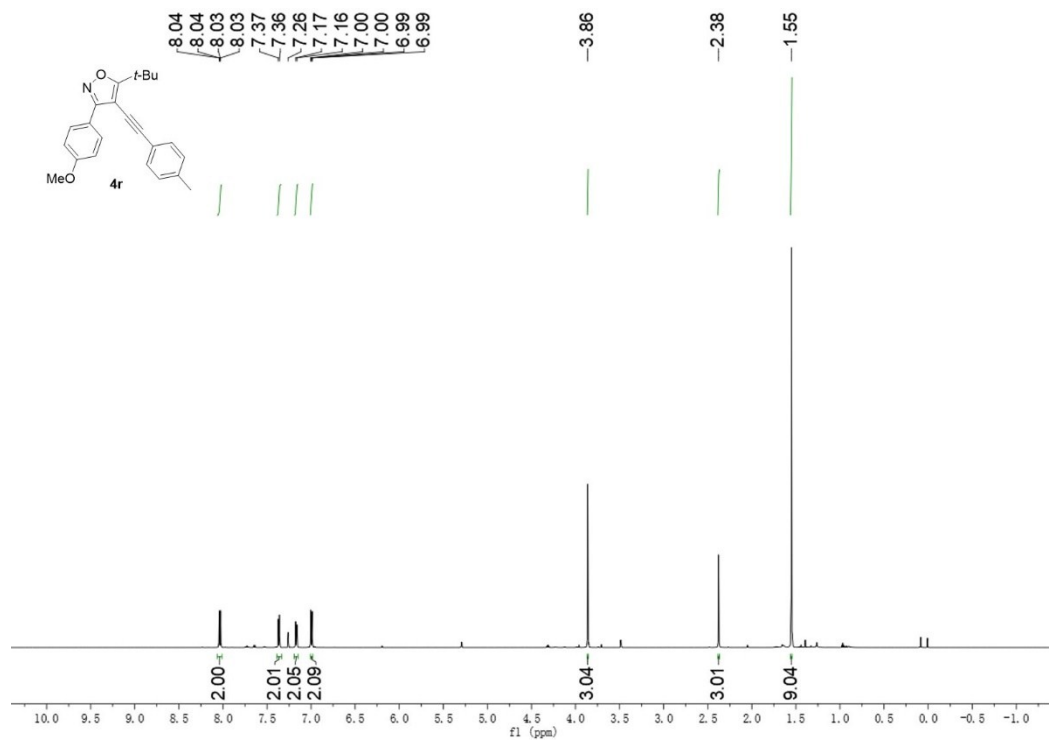


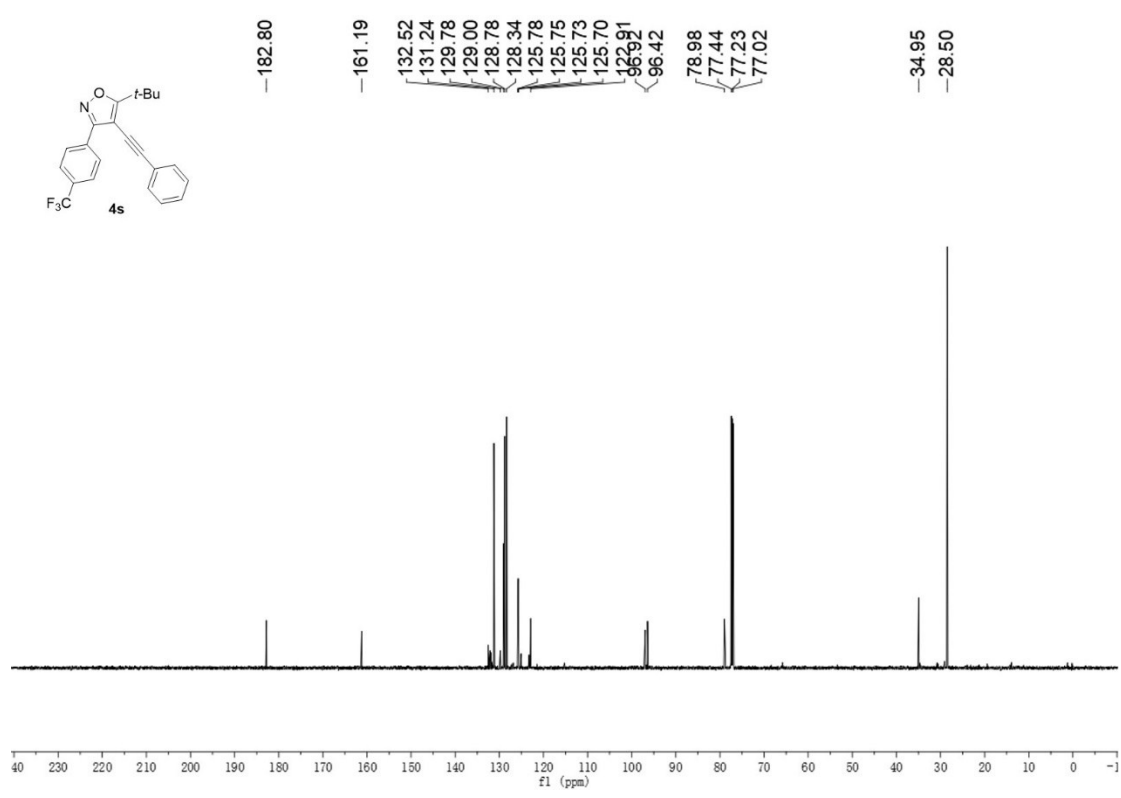
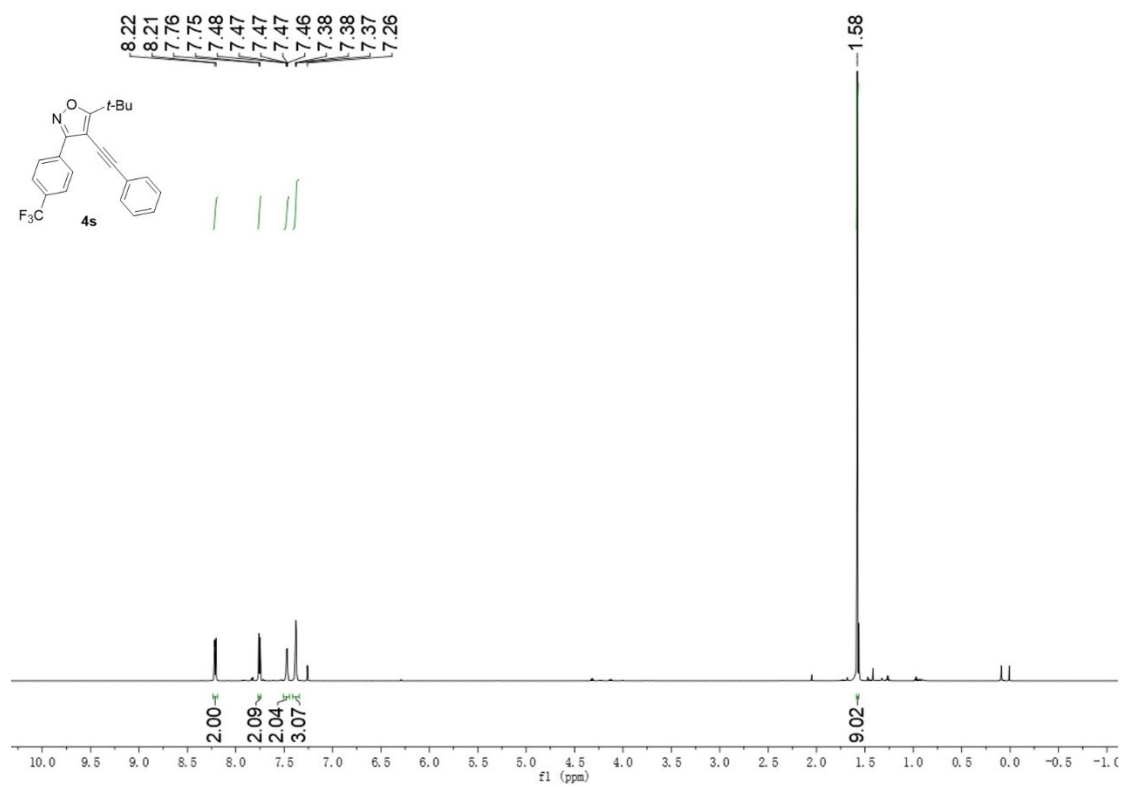


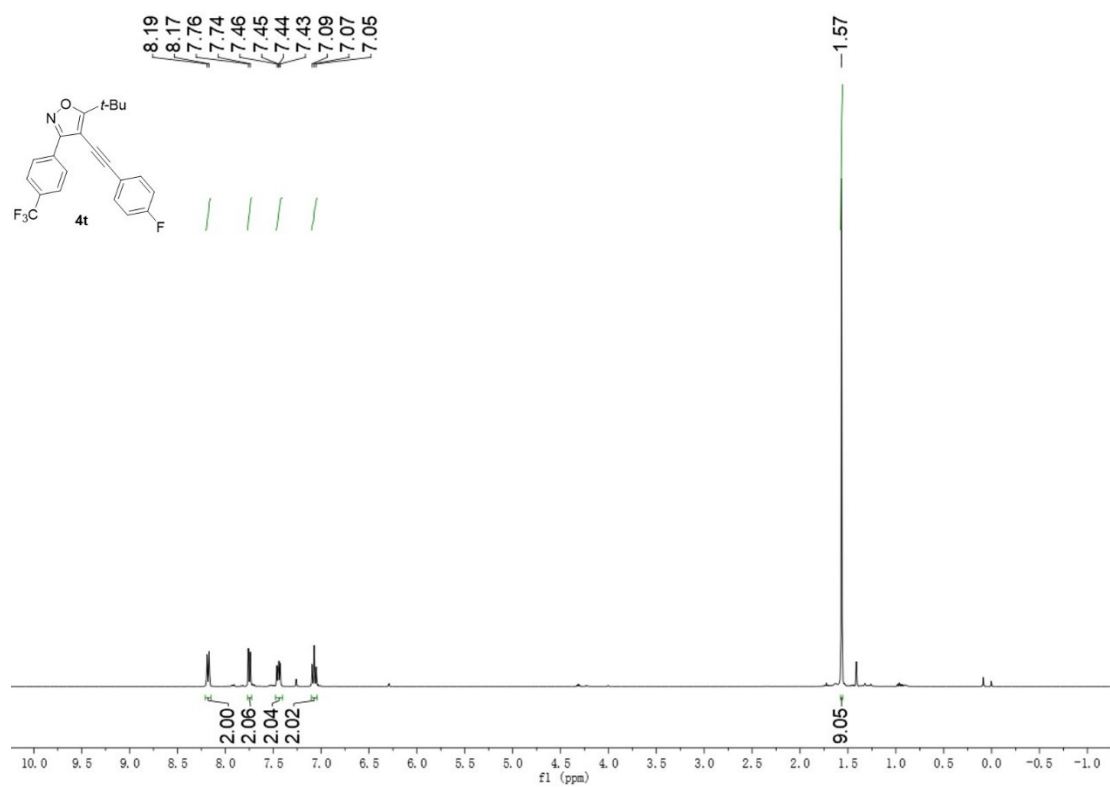
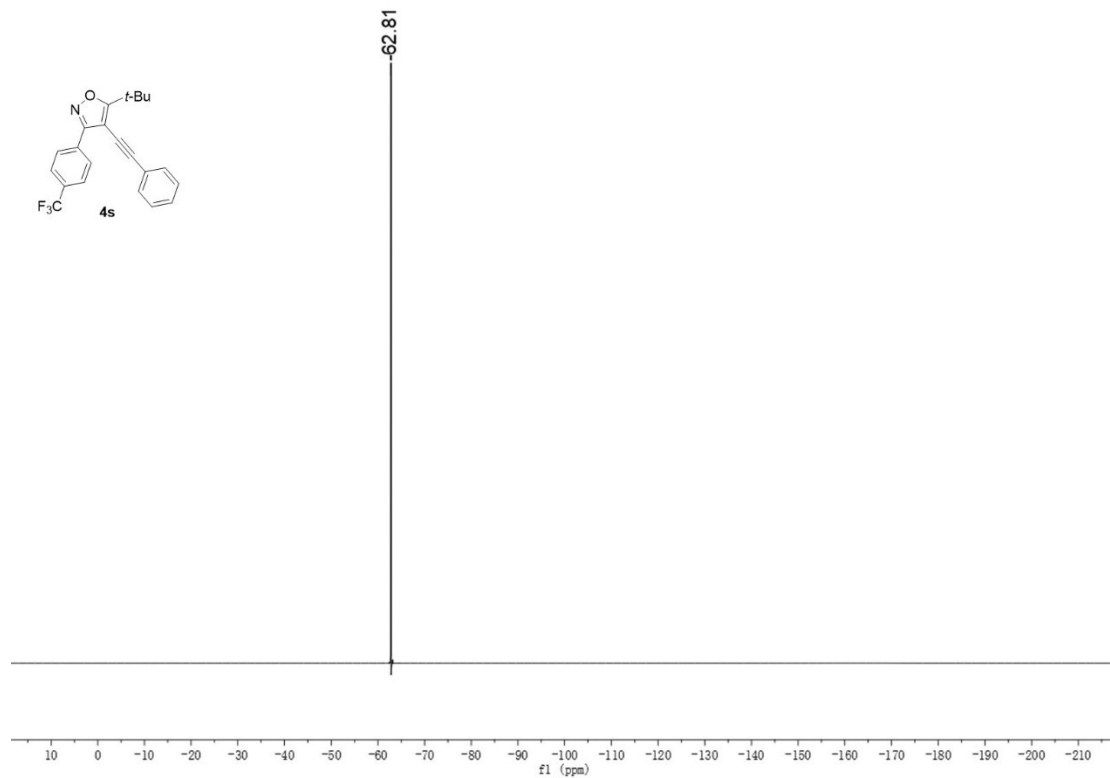


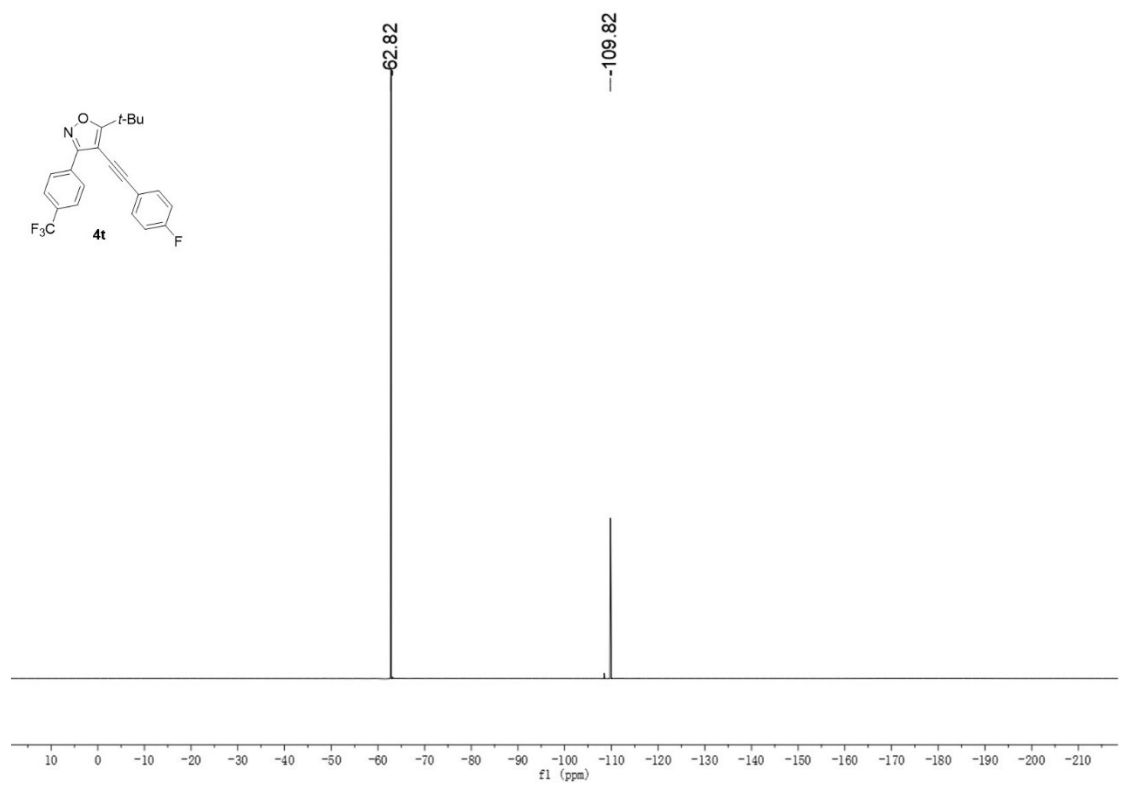
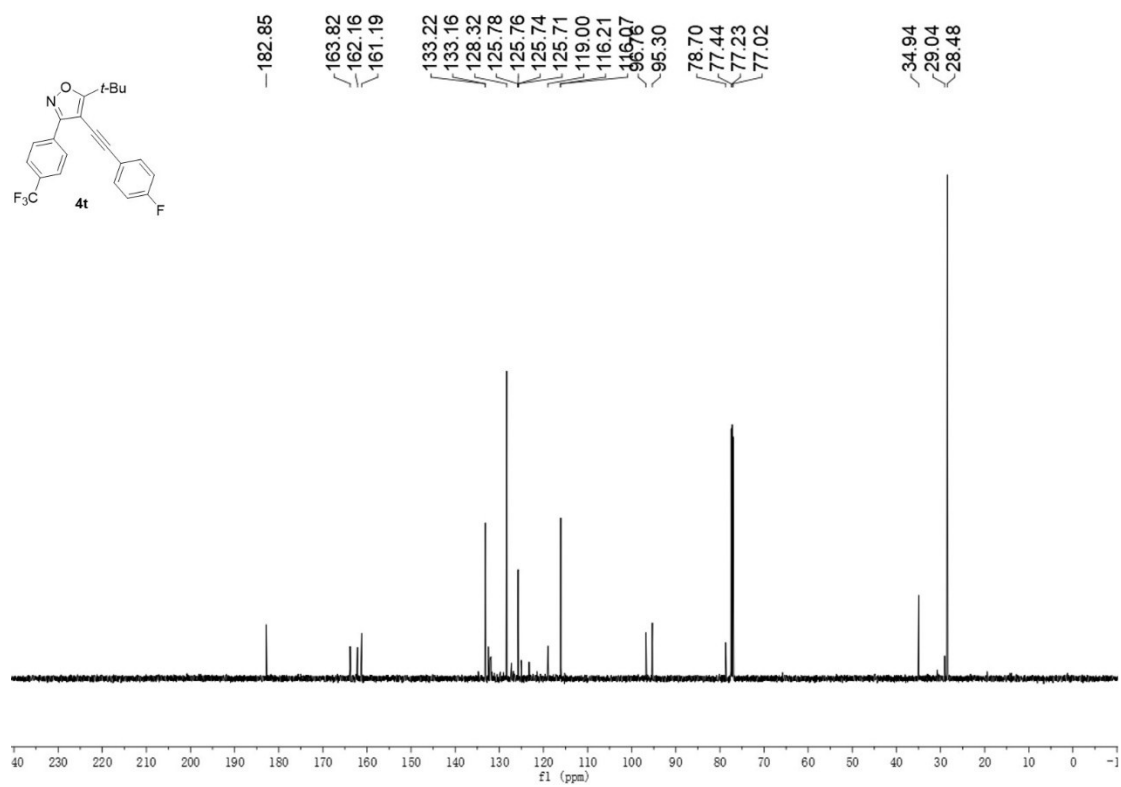


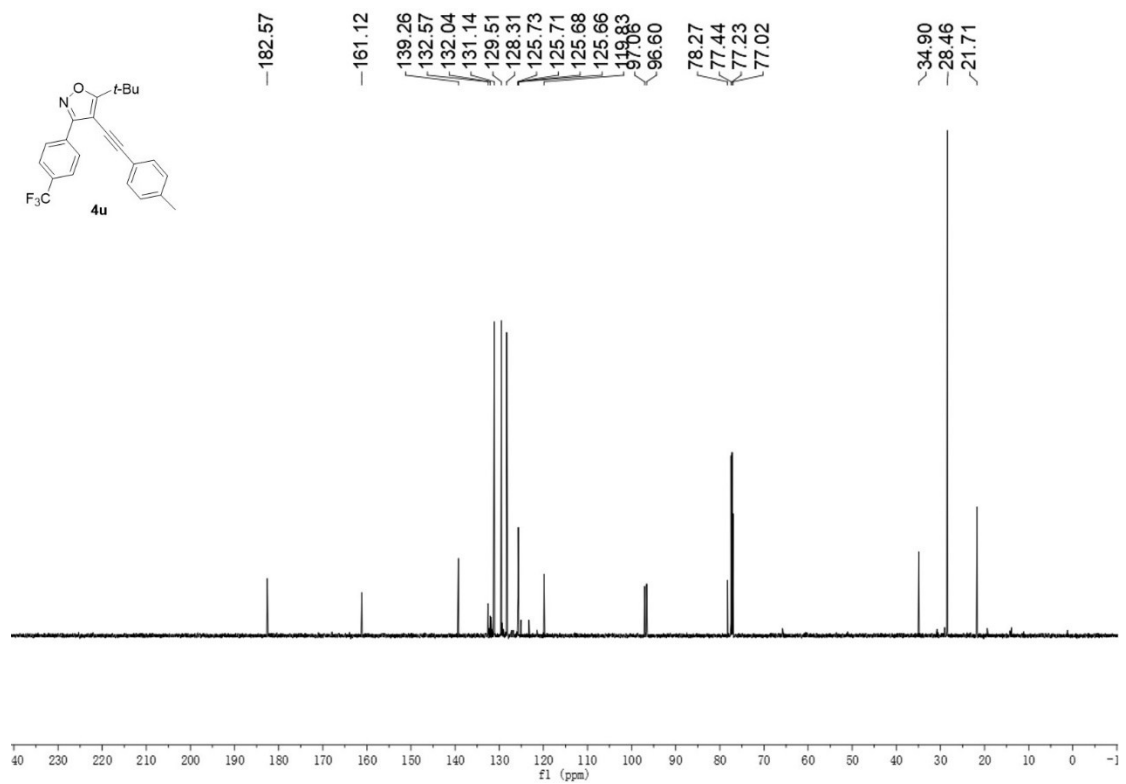
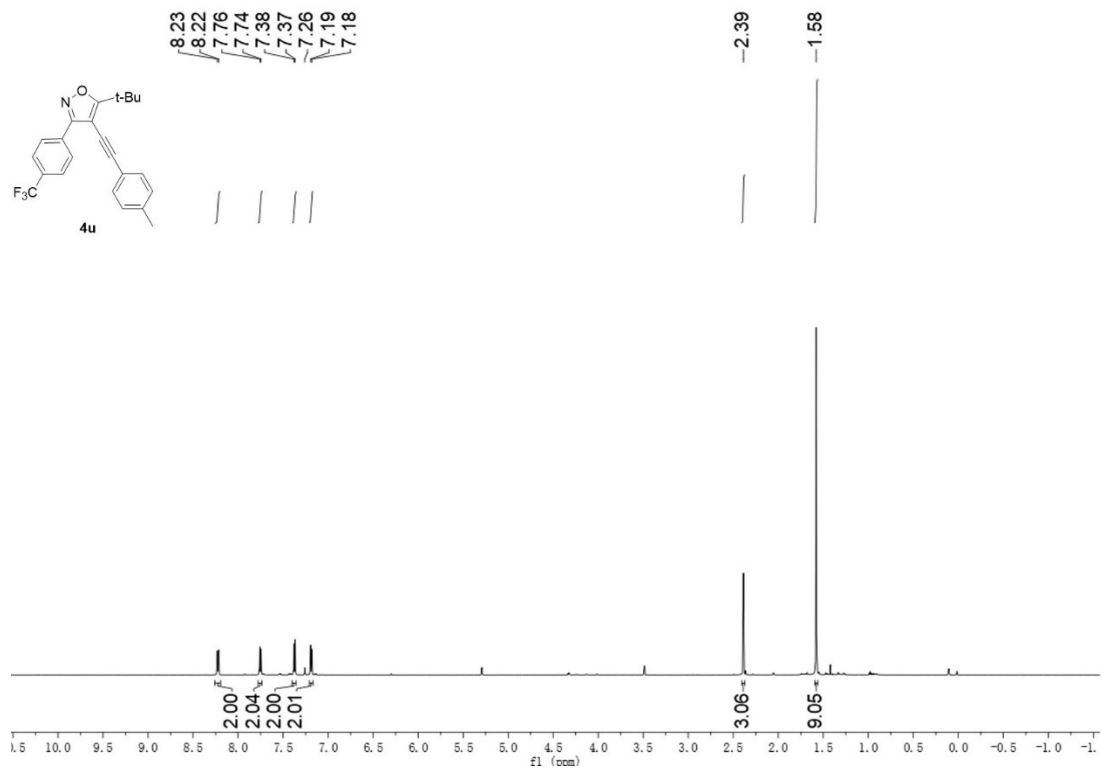


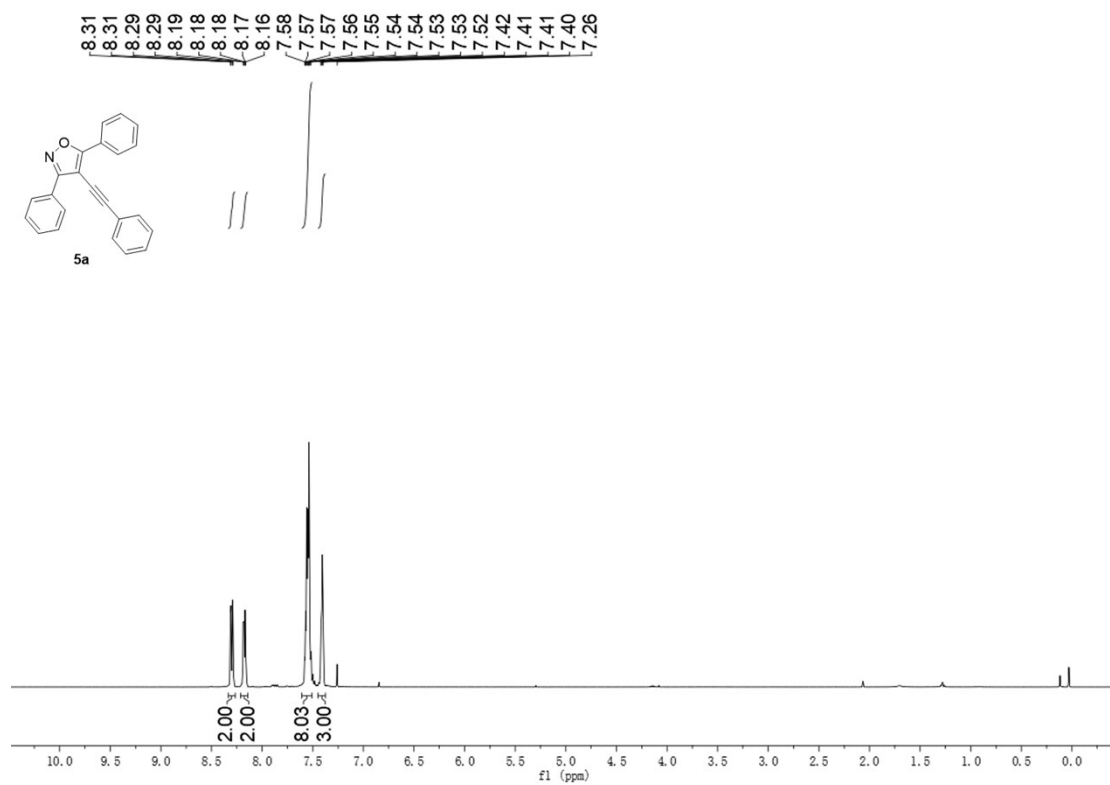
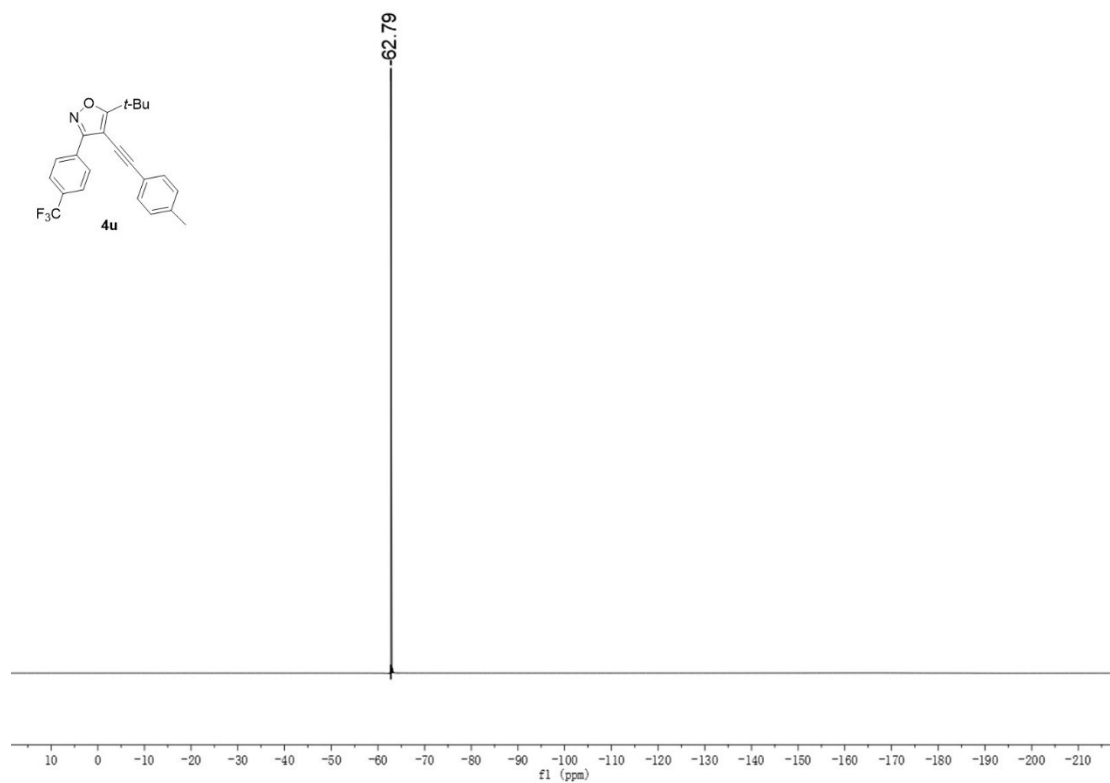


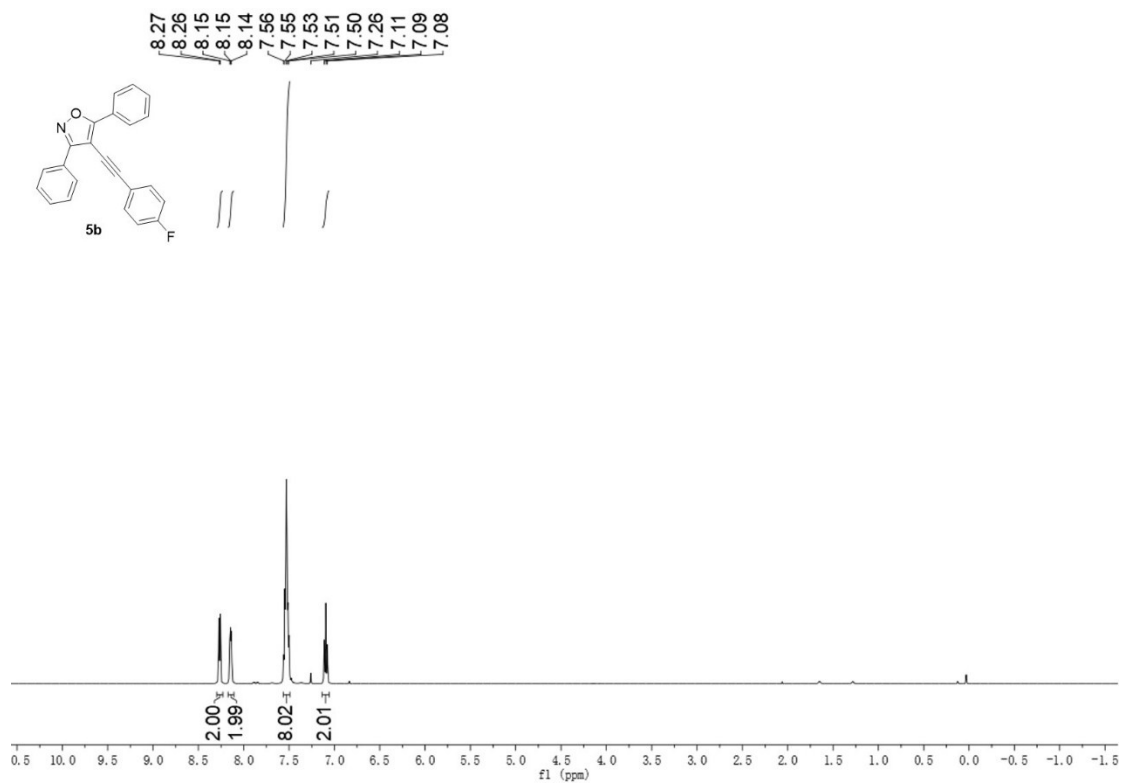
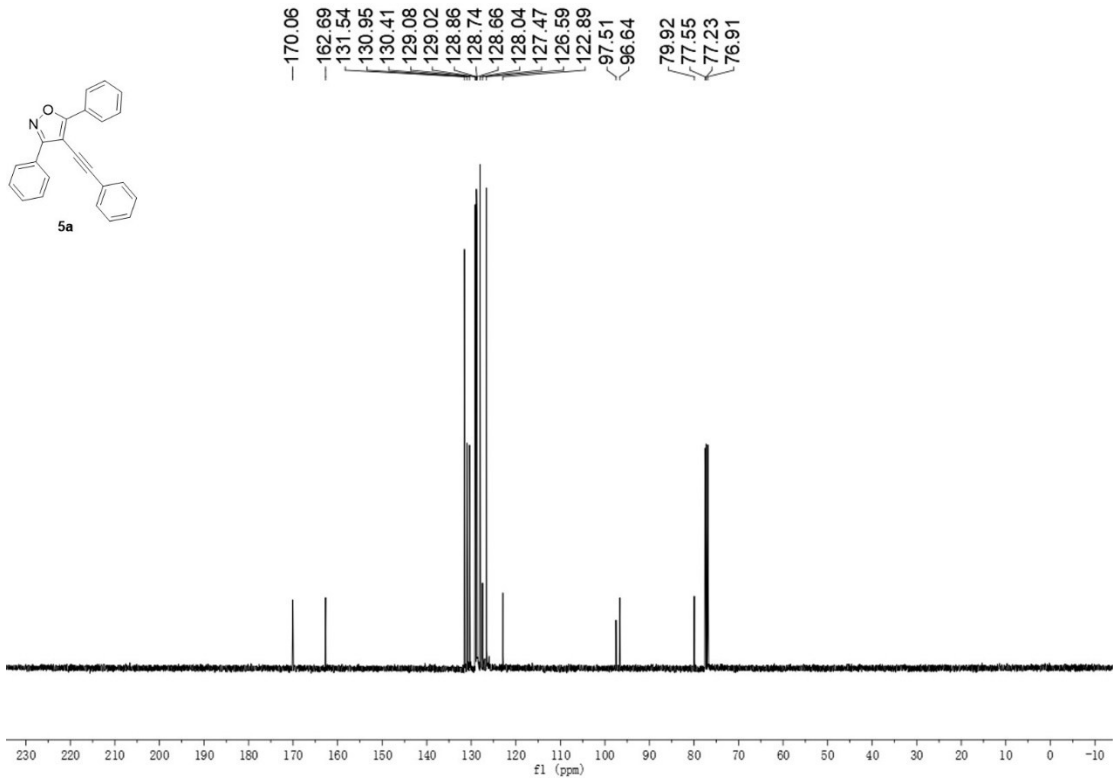


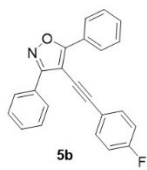




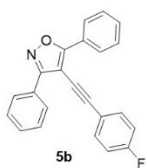
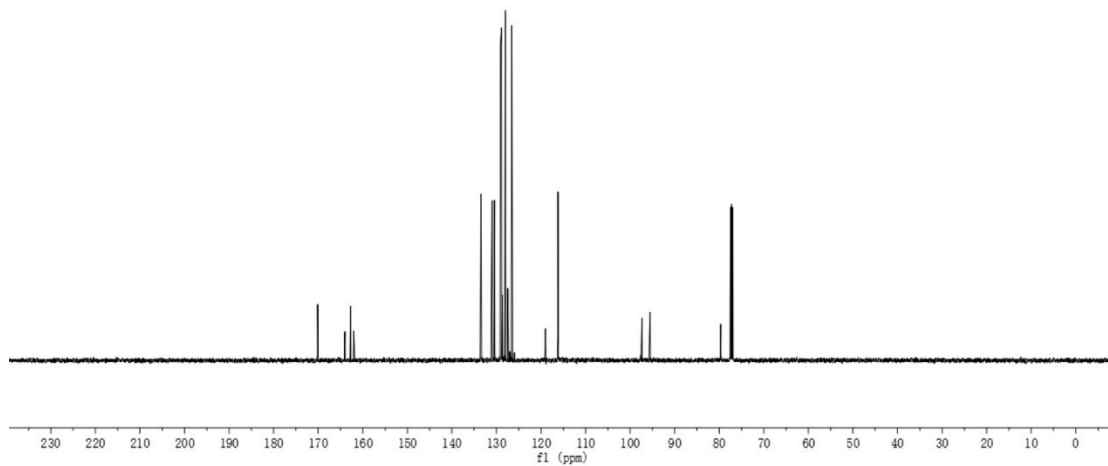








170.13  
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 162.70  
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 128.66  
 128.02  
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-109.62

