

# Supporting Information

**Peroxide-mediated synthesis of  
benzimidazo[2,1-a]isoquinoline-6(5H)-ones via cascade  
methylation/ethylation and intramolecular cyclization**

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## 1. Mechanistic Studies

### 1.1 Methyl Radical Capture Experiments

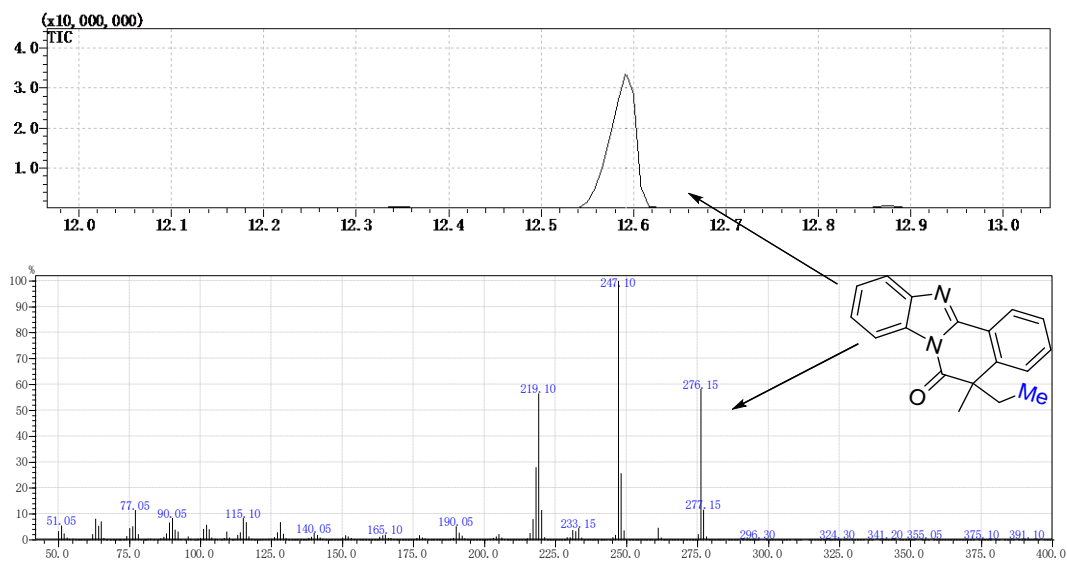


Figure S1 GC-MS spectra of the 3a

### Standard Procedure + TEMPO (2.0 equiv):

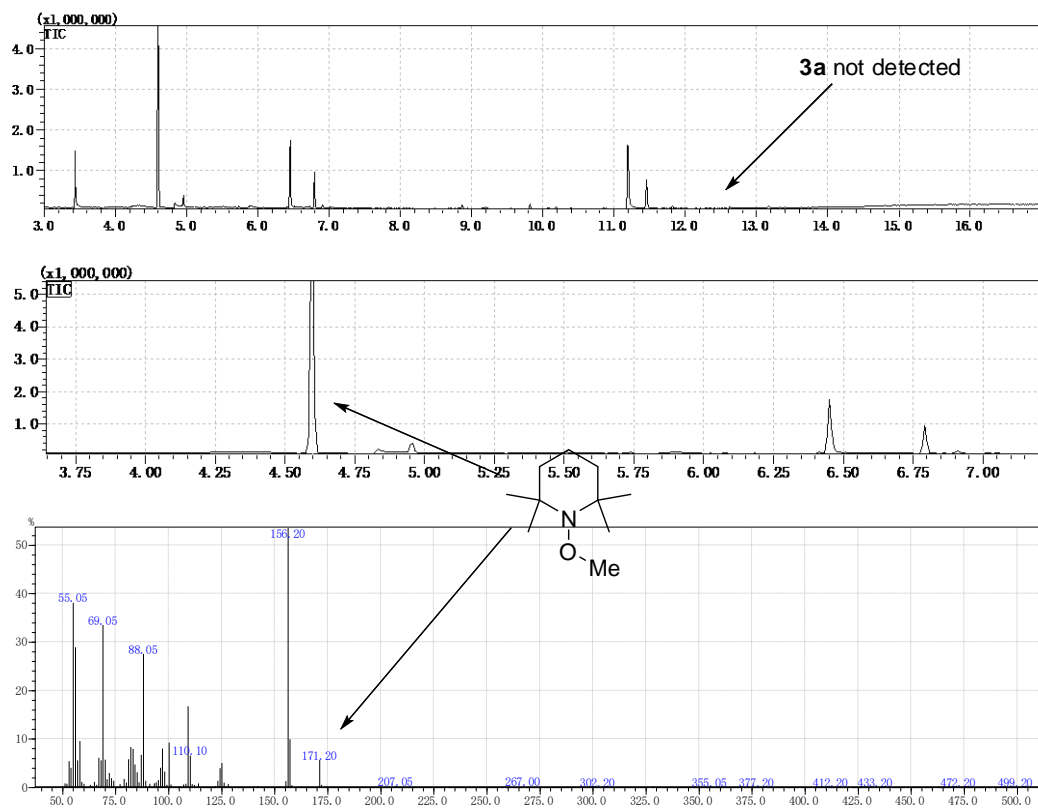
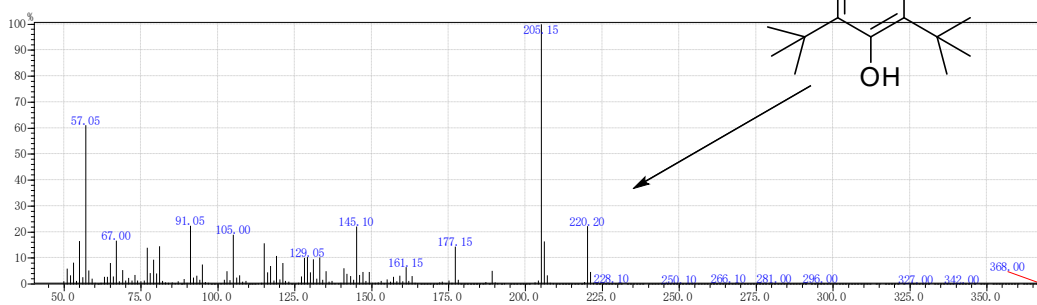
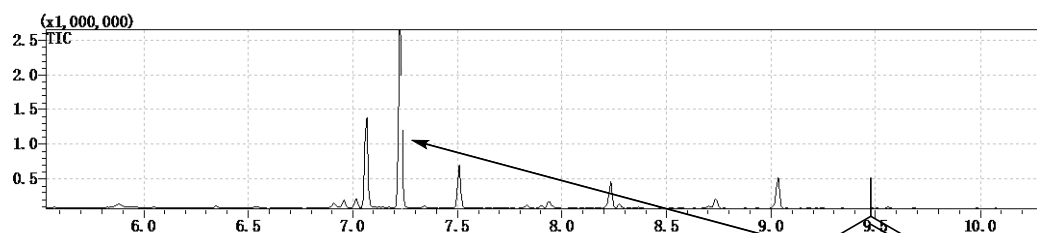
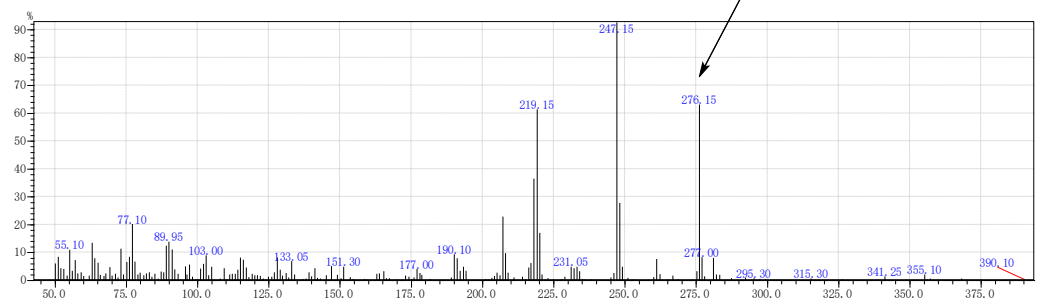
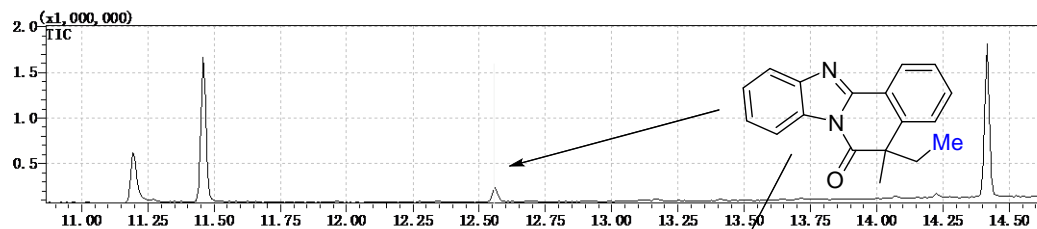
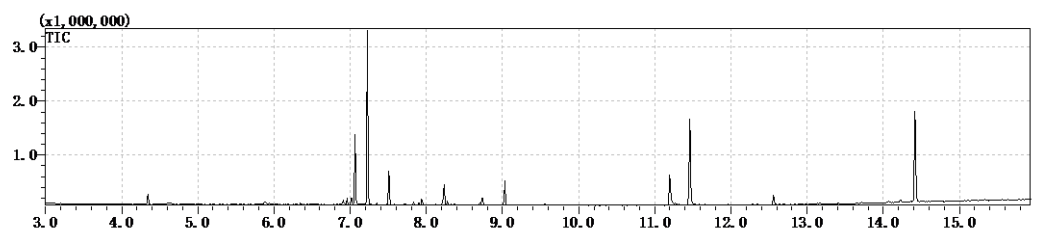


Figure S2 GC-MS spectra of the methyl radical capture results by TEMPO

Standard Procedure + BHT (2.0 equiv):



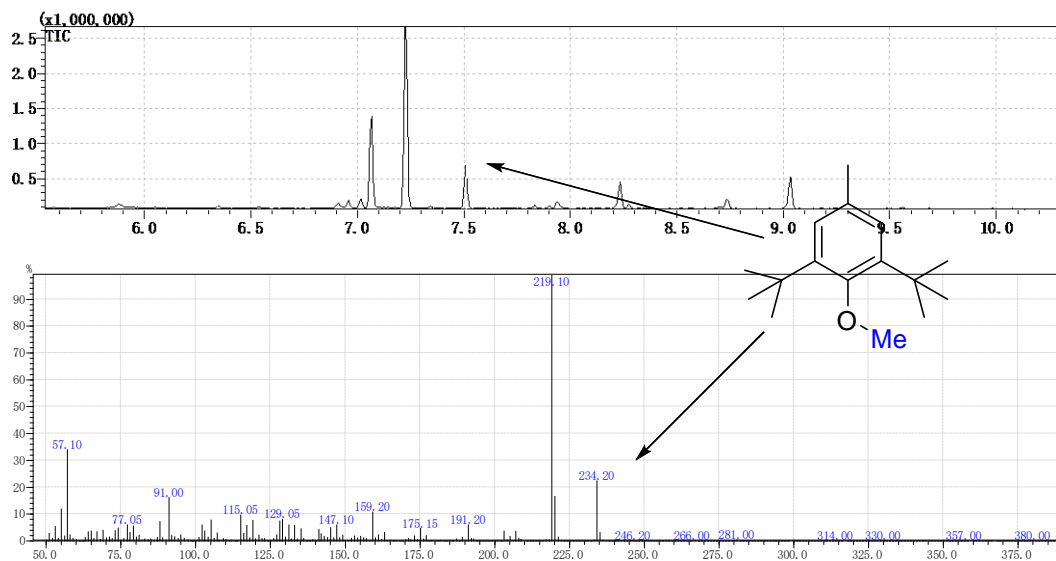


Figure S3 GC-MS spectra of the methyl radical capture results by BHT

## 1.2 The detection of acetone and *tert*-butanol

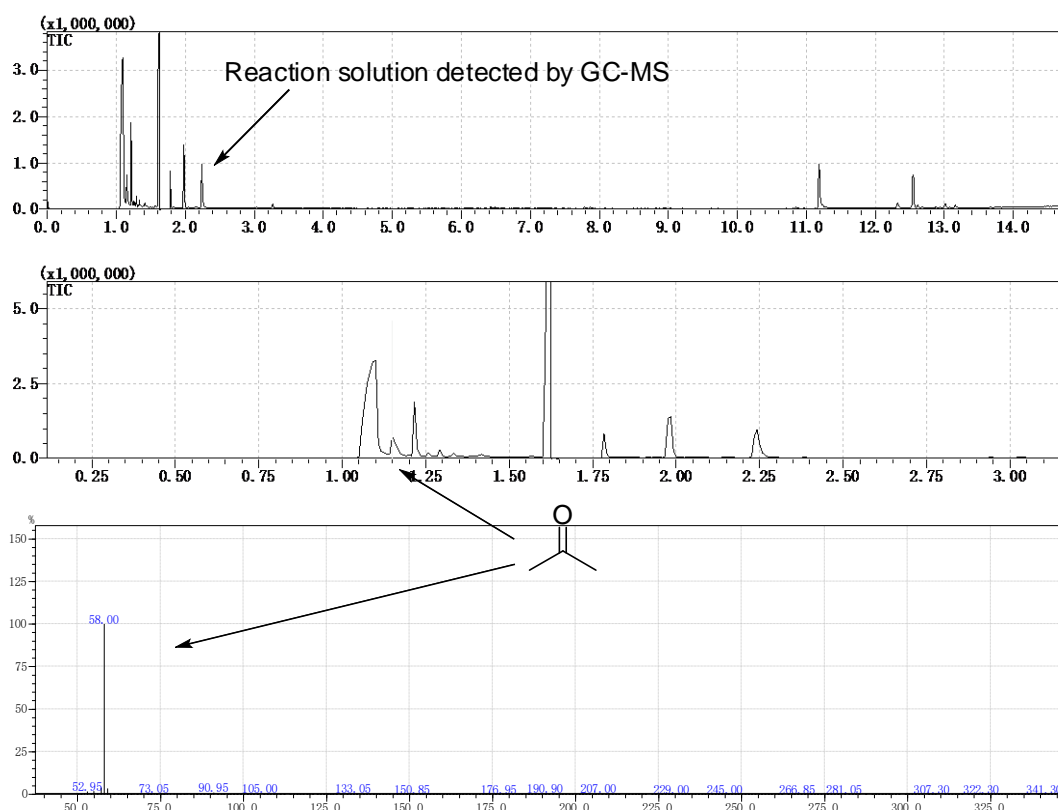


Figure S4 GC-MS spectra for detection of acetone

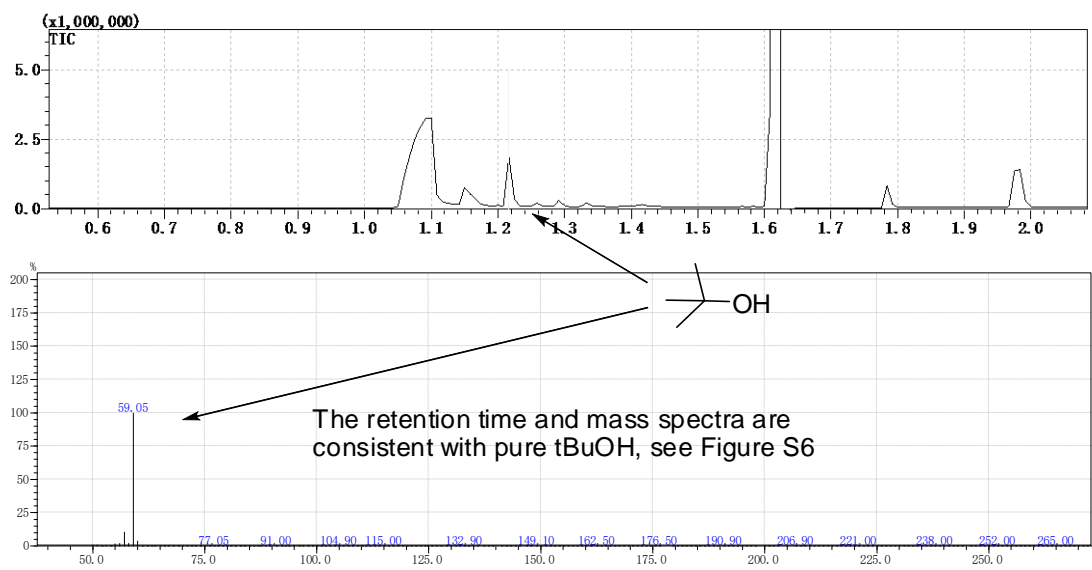


Figure S5 GC-MS spectra for detection of tert-butanol

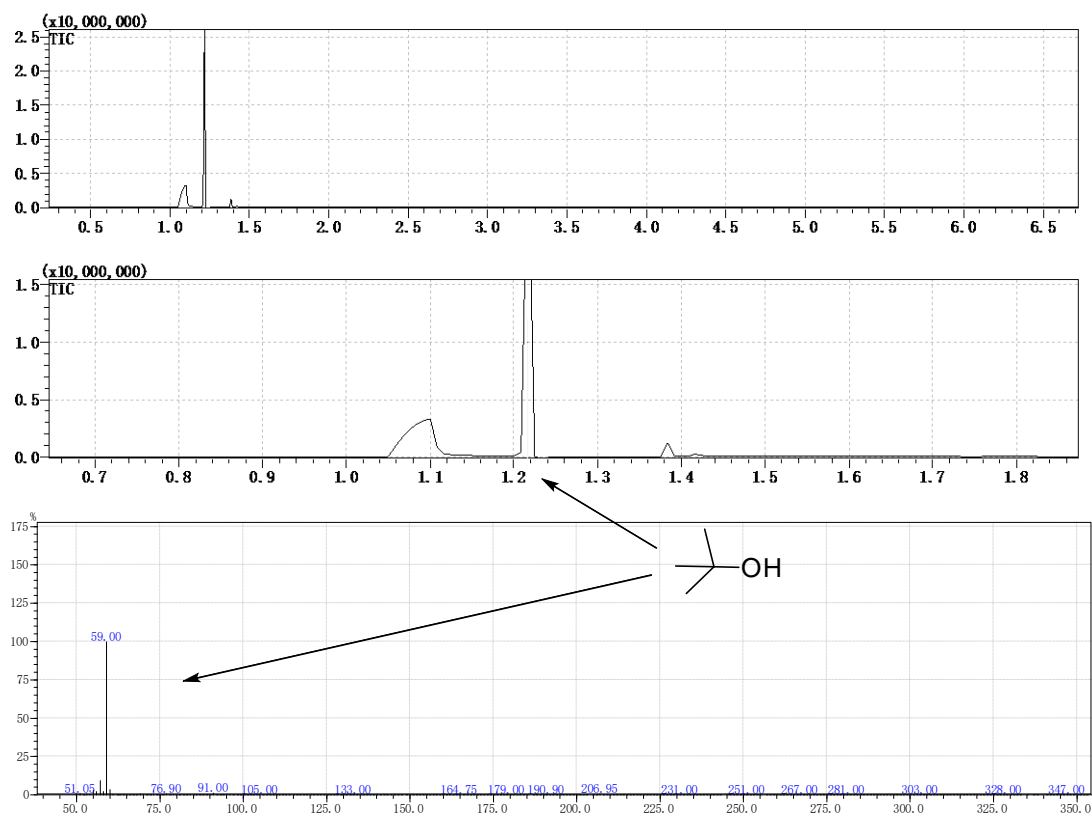
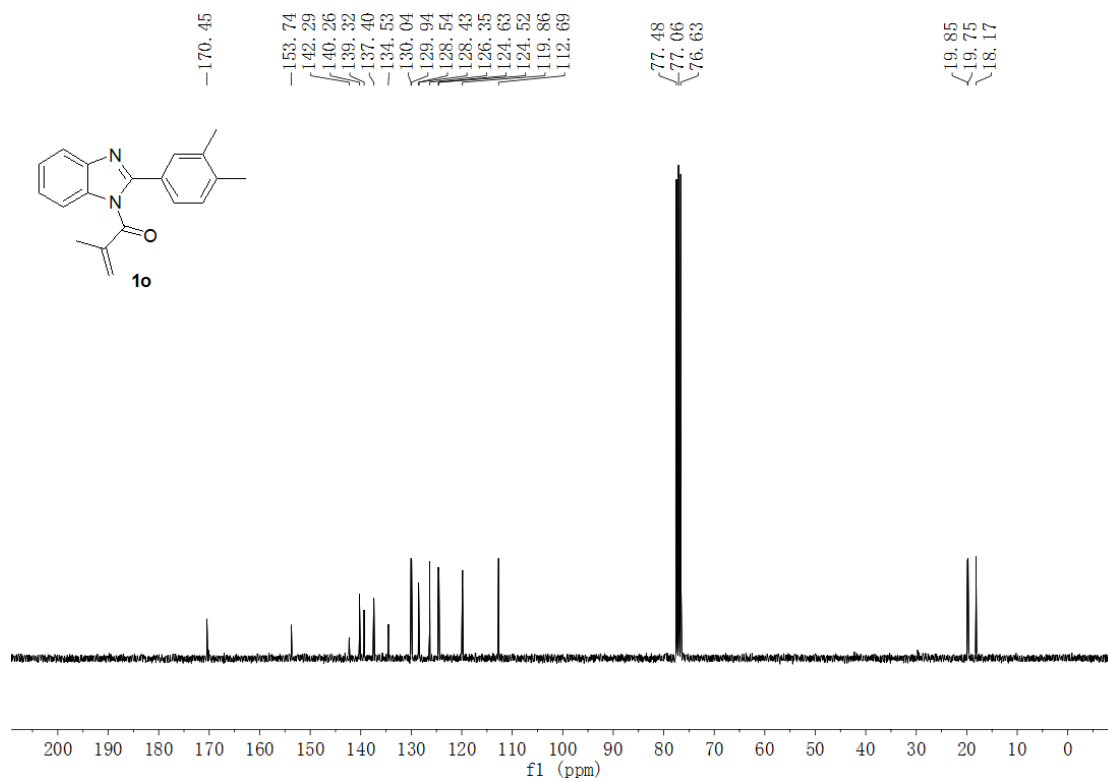
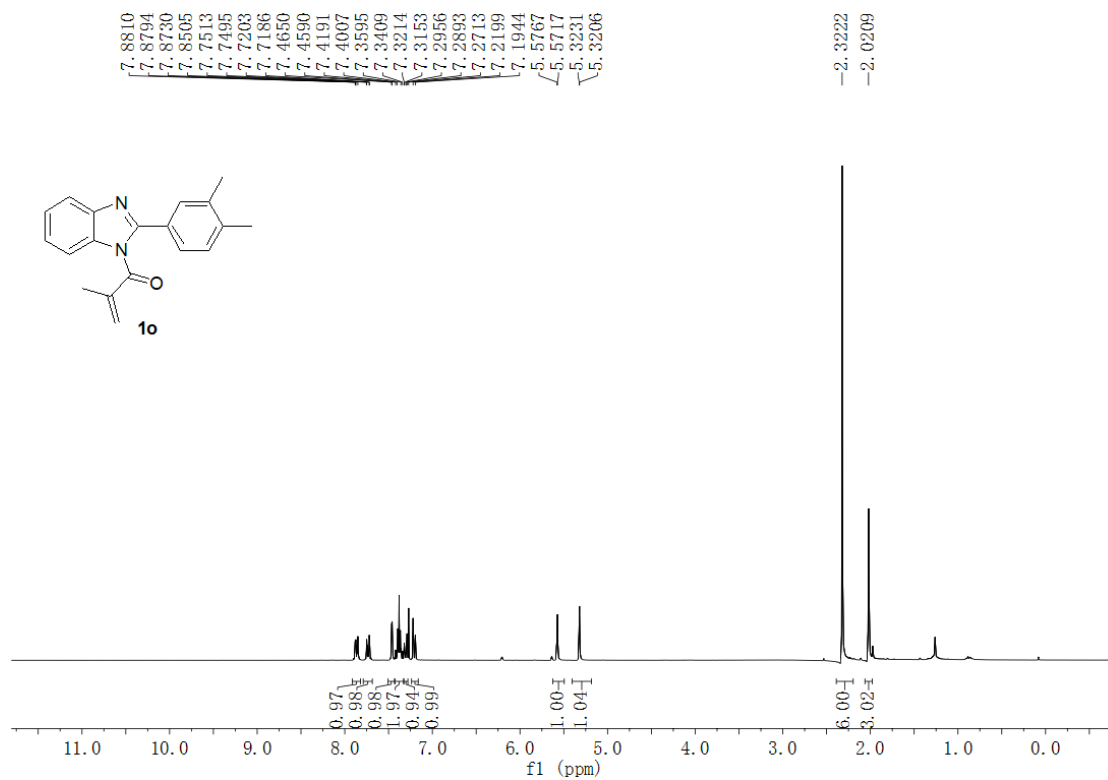
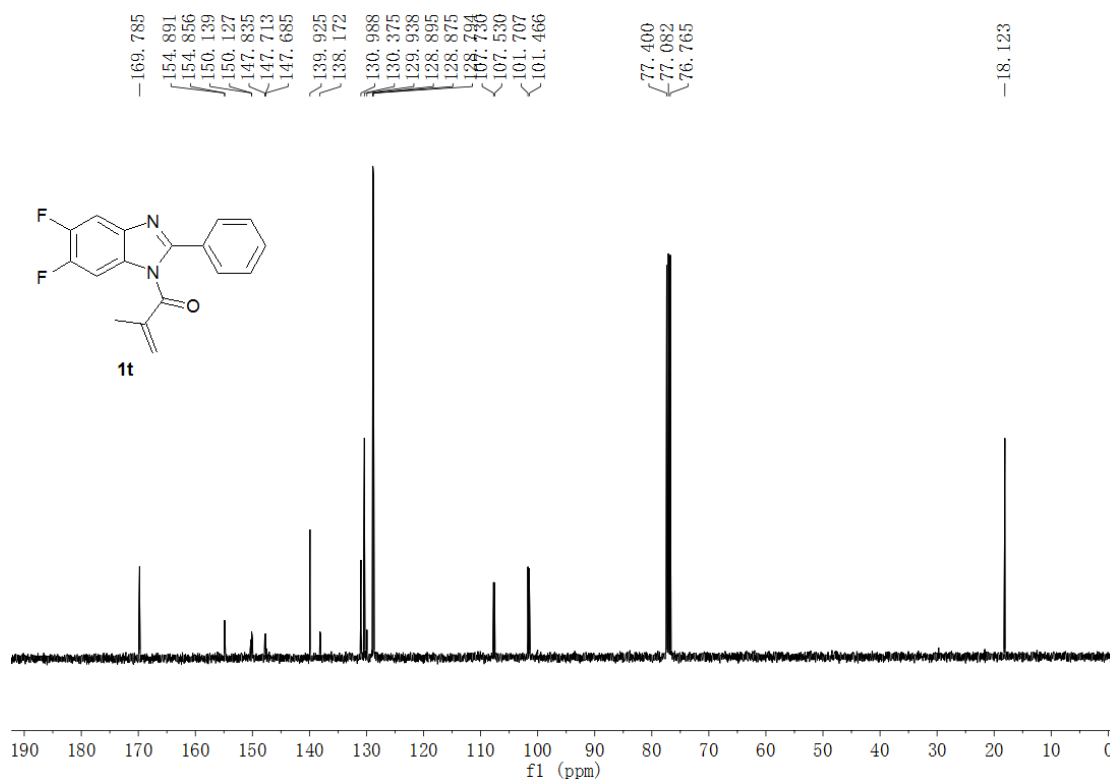
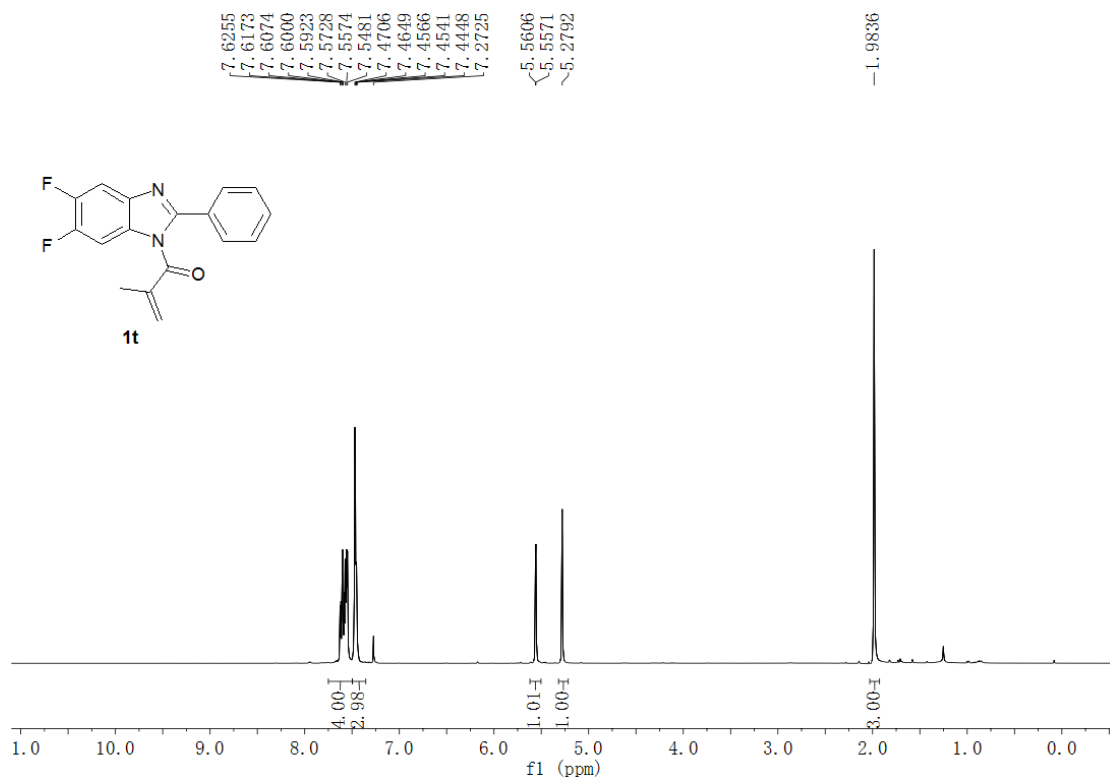
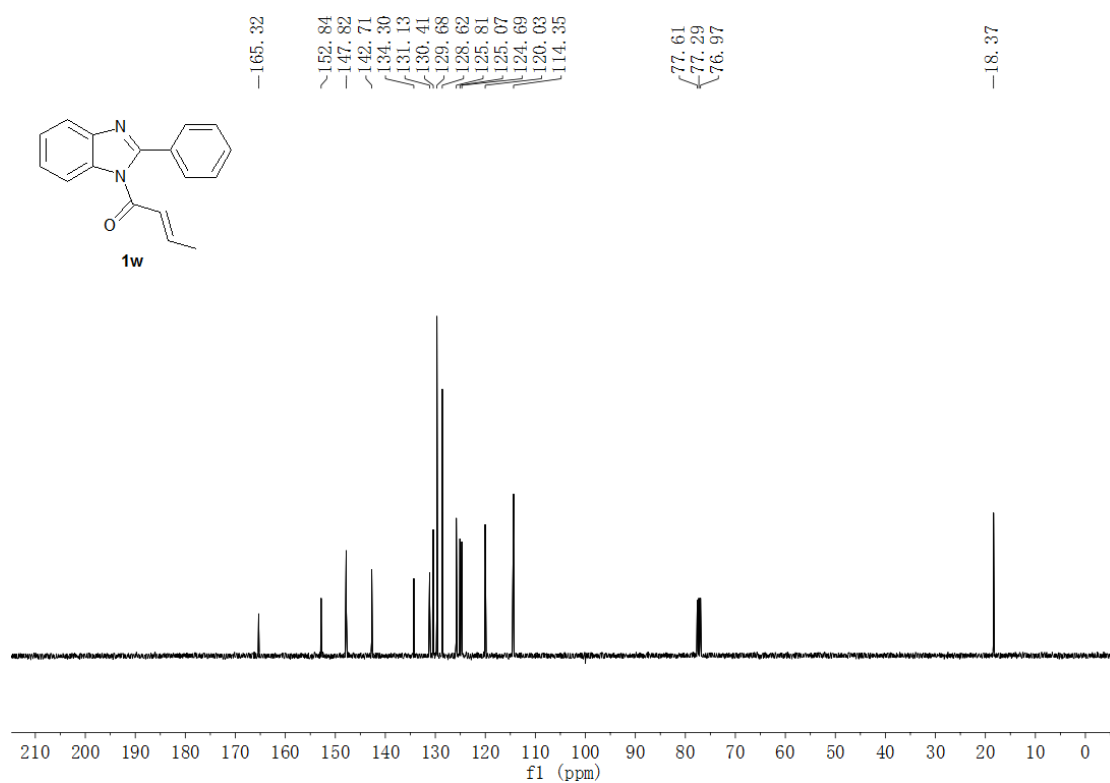
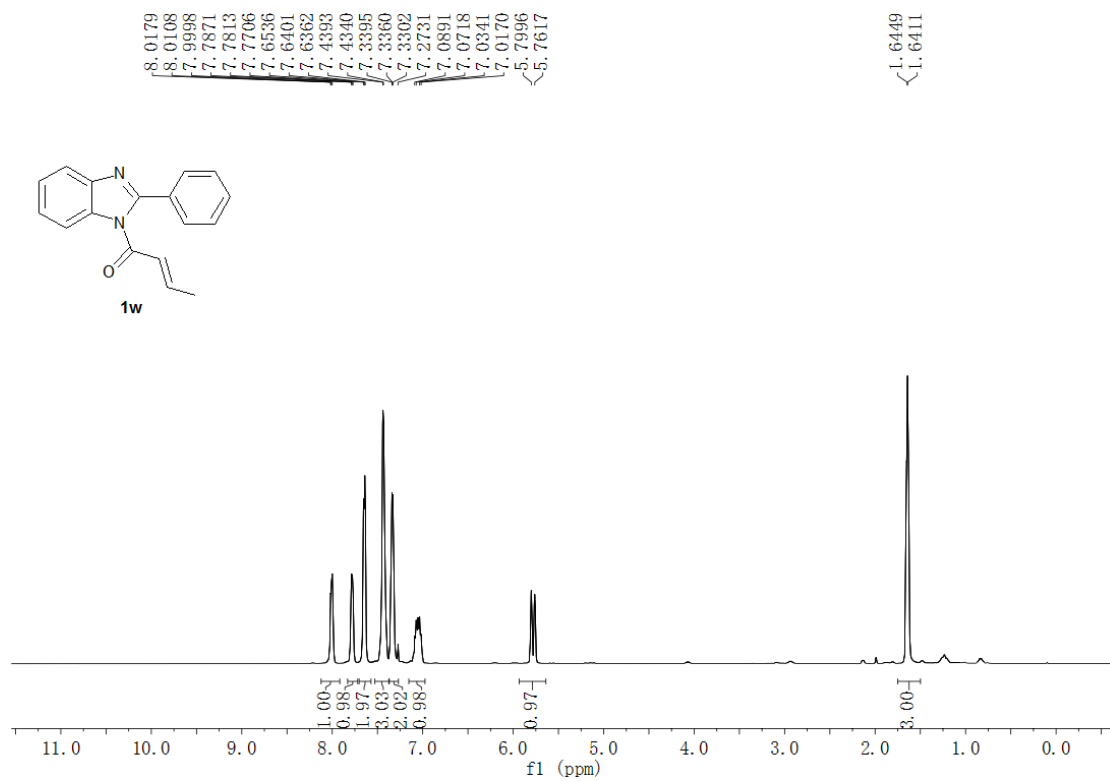


Figure S6 GC-MS spectra of pure *tert*-butanol

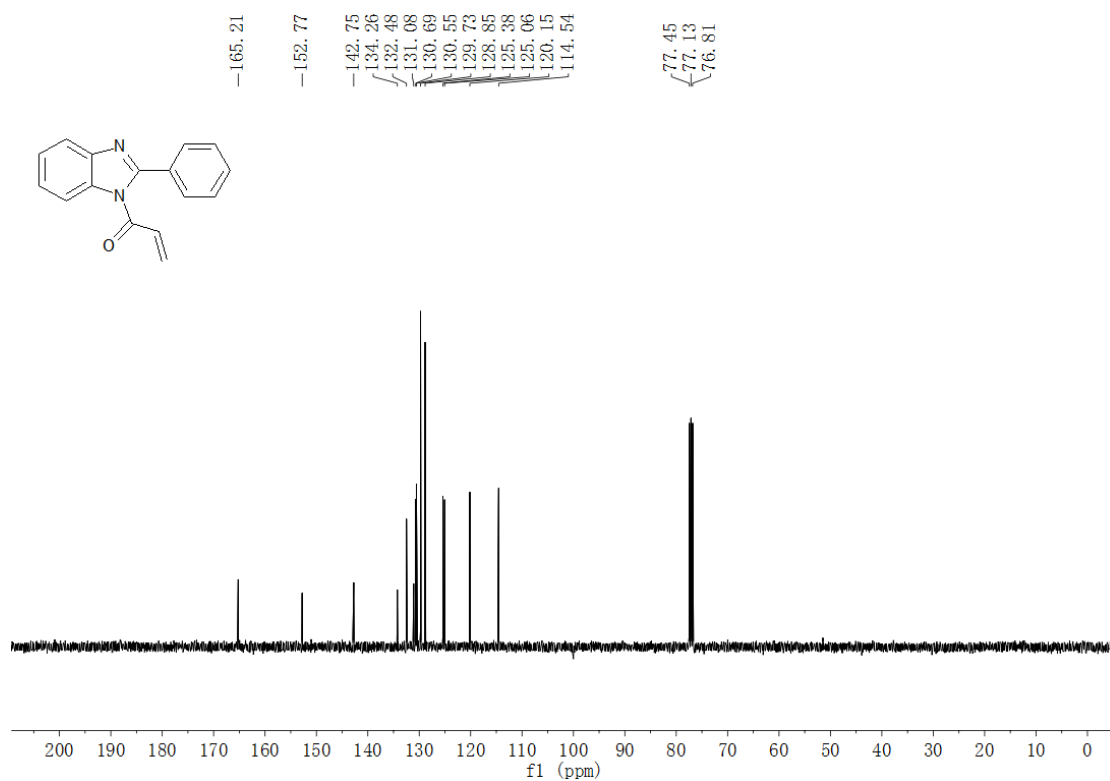
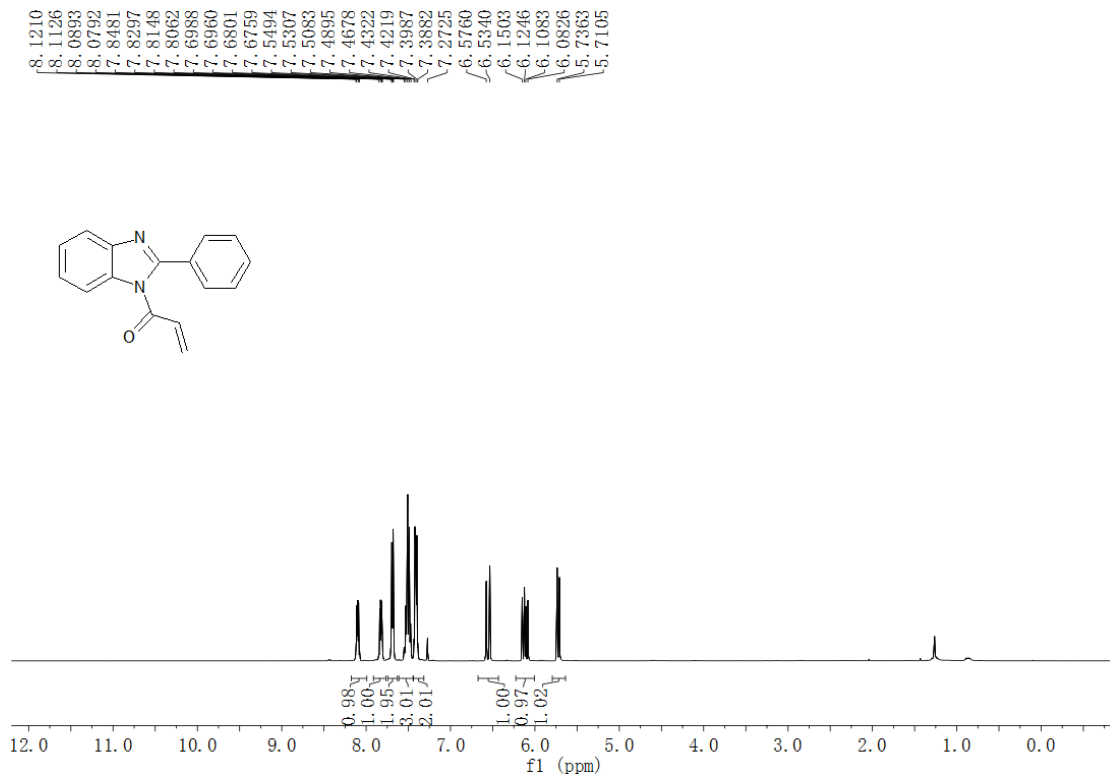
2. Copies of  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra of the unknown substrate











### 3. Copies of $^1\text{H}$ NMR and $^{13}\text{C}$ NMR spectra of the products

