Supporting Information

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

Changduo Pan,* Cheng Yuan and Jin-Tao Yu*

E-mail: panchangduo@jsut.edu.cn; yujintao@cczu.edu.cn

1. Mechanistic Studies.................................................................S2

2. Copies of $^1$H NMR and $^{13}$C NMR spectra of the unknown substrate……S6

3. Copies of $^1$H NMR and $^{13}$C NMR spectra of the products.................S10
1. Mechanistic Studies

1.1 Methyl Radical Capture Experiments

![Figure S1 GC-MS spectra of the 3a](image1)

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

![Figure S2 GC-MS spectra of the methyl radical capture results by TEMPO](image2)
Standard Procedure + BHT (2.0 equiv):

![Graph 1]

![Graph 2]

![Graph 3]

![Graph 4]
1.2 The detection of acetone and *tert*-butanol

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

Figure S4 GC-MS spectra for detection of acetone
The retention time and mass spectra are consistent with pure tBuOH, see Figure S6.

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

Figure S6 GC-MS spectra of pure tert-butanol

The retention time and mass spectra are consistent with pure tBuOH, see Figure S6.
2. Copies of $^1$H NMR and $^{13}$C NMR spectra of the unknown substrate

![NMR Spectra Image]
3. Copies of $^1$H NMR and $^{13}$C NMR spectra of the products