

Supplementary Information

TBHP-promoted sequential carboxamidation and aromatisation of aryl isonitriles with formamides

Xiaomei Feng, Hui Zhu, Lei Wang, Yan Jiang, Jiang Cheng and Jin-Tao Yu*

*School of Petrochemical Engineering, Jiangsu Key Laboratory of Advanced Catalytic Materials &
Technology, Jiangsu Province Key Laboratory of Fine Petrochemical Engineering, Changzhou
University, Changzhou 213164, P. R. China*

Email: yujintao@cczu.edu.cn

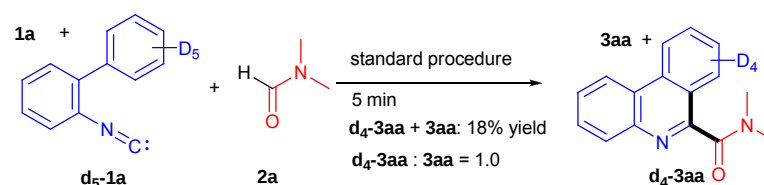
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1 Intermolecular KIE experiments

KIE experiment of **1a** and **d₅-1a**

In a sealed tube, the mixture of **1a** (0.1 mmol) and **d₅-1a** (0.1 mmol) was treated under standard procedures and heated for 5 min. After that, the mixture was concentrated in vacuum and the residue was purified by flash column chromatography on silica gel with petroleum ether- ethyl acetate as eluent to give **3aa** and **d₄-3aa** in 18% total yield. The mixture of **3aa** and **d₄-3aa** was analyzed using ¹H NMR spectrometer. As shown in Figure S1, the ratio of **3aa** and **d₄-3aa** is nearly 1: 1.



Scheme S1 KIE experiment of **1a** and **d₅-1a**

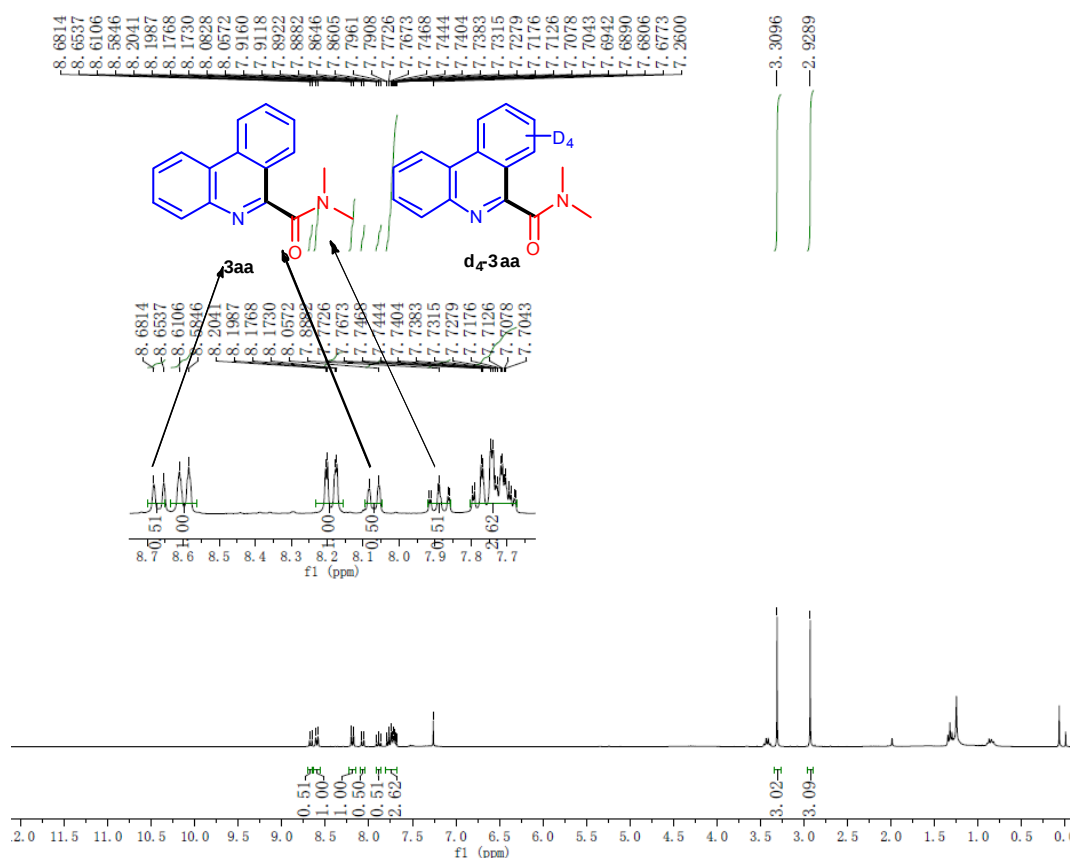
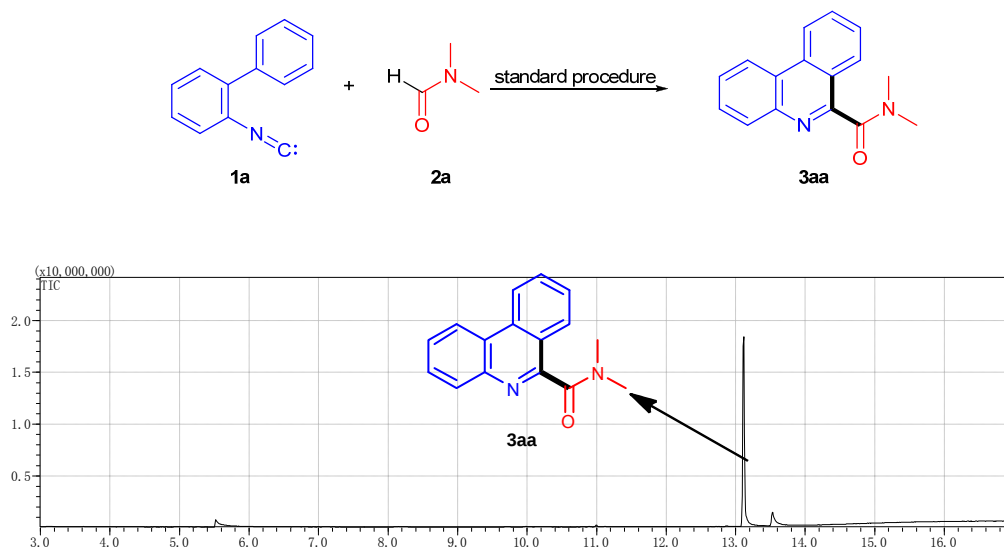


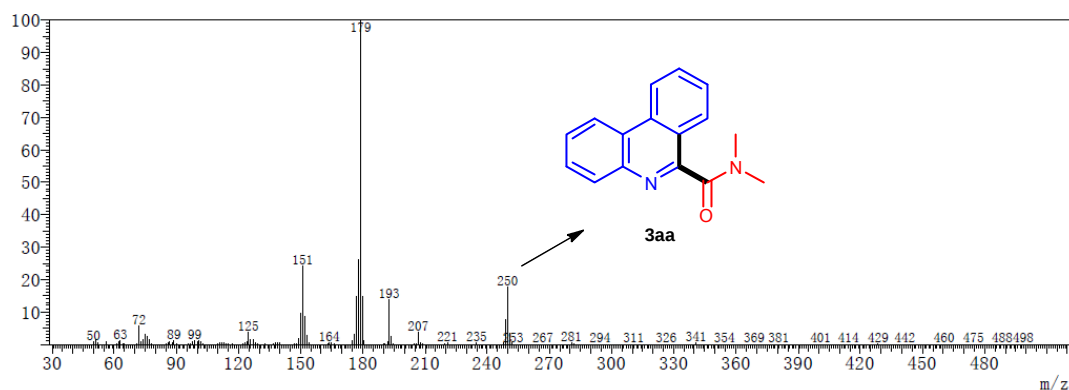
Figure S1 The ¹H NMR spectrum of the KIE results.

2 Free radical capture experiments

2.1 Standard procedure

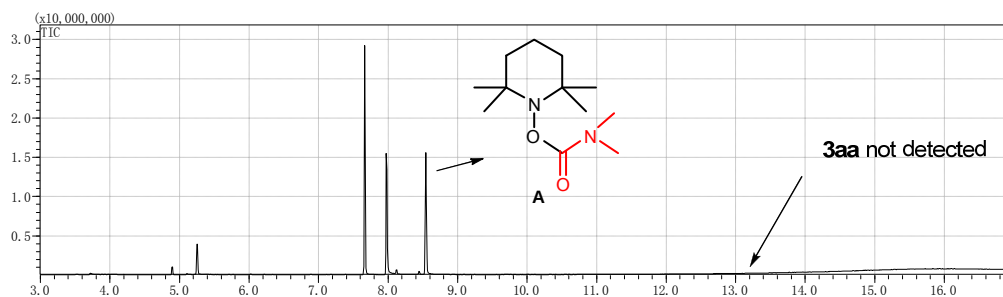
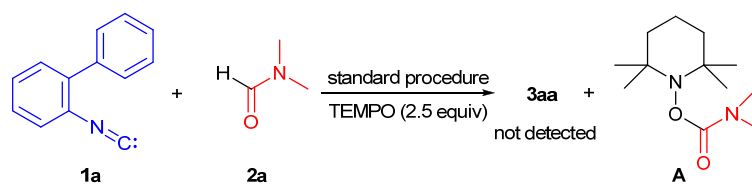


Residual time of 3aa: 13.117 min.



2.2 Adding 2.5 equiv of TEMPO

Under standard procedure, a sealed tube was charged with **1a** (0.2 mmol), TBHP (0.4 mmol) (70% in water), NaOAc (0.1 mmol), TEMPO (0.5 mmol) in DMF (2.0 mL). The reaction was conducted under air at 130 °C for 5 h. The reaction mixture was analyzed using GCMS spectrometer, as shown in Figure S2, no product **3aa** was detected, while the TEMPO-DMF adduct **A** was detected by GCMS.



Residual time of adduct A: 8.541 min

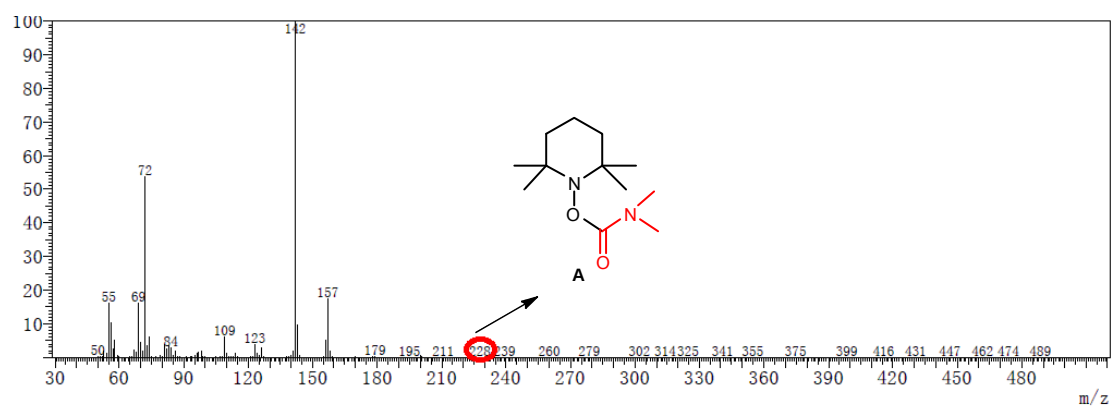


Figure S2 GC-MS spectra of the free radical capture results

3 Copies of ^1H NMR and ^{13}C NMR spectra

