Metal-Free Oxidative Functionalization of C(sp$^3$)−H of Ketones and Alcohols for the Synthesis of Isoquinolinononediones

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**General Remarks:** Column chromatography was carried out on silica gel. Unless noted, $^1$H NMR spectra were recorded on 400 MHz in CDCl$_3$. $^{13}$C NMR spectra were recorded on 100 MHz in CDCl$_3$. IR spectra were recorded on an FT-IR spectrometer and only major peaks are reported in cm$^{-1}$. Melting points were determined on a microscopic apparatus and were uncorrected. All new products were further characterized by HRMS (high resolution mass spectra), high resolution mass spectrometry (HRMS) spectra was obtained on a Thermo Scientific LTQ Orbitrap XL instrument equipped with an ESI source; copies of their $^1$H NMR and $^{13}$C NMR spectra are provided.

**Kinetic Isotope Effect (KIE) Experiment**

![Kinetic Isotope Effect (KIE) Experiment](image)

$K_{H}/K_{D} = 1.1 : 1$
Preparation of Tetrahydrofuro[2,3-c]isoquinolin-5(2H)-one 5a from 4a.

An oven-dried Schlenk tube (10 mL) was equipped with a magnetic stir bar, 4a (0.4 mmol), dry THF (5.0 mL). The reaction mixture was then stirred at 0 °C, and 1.0 equiv. LiAlH₄ (28 mg) was slowly added. After the reaction was completed, 6 mL water was added to quench the reaction at 0 °C, and the resulting mixture was extracted twice with EtOAc (2×10 mL). The combined organic extracts were washed with brine, dried over Na₂SO₄ and concentrated. Purification of the crude product by flash column chromatography afforded the product 5a (petroleum ether/ethyl acetate as eluent (4:1)).

5a, m.p.=100-102 °C, 2,2,4,9b-tetramethyl-1,3a,4,9b-tetrahydrofuro[2,3-c]isoquinolin-5(2H)-one, ¹H NMR (400 MHz): 8.17-8.19 (m, 1 H), 7.49-7.53 (m, 1 H), 7.29-7.36 (m, 2 H), 4.94 (s, 1 H), 3.24 (s, 3 H), 2.55-2.58 (m, 1 H), 2.07-2.10 (m, 1 H), 1.40 (s, 3 H), 1.34 (s, 3 H), 0.86 (s, 3 H); ¹³C NMR (100 MHz): 163.6, 143.7, 132.2, 128.6, 126.8, 126.5, 125.5, 95.9, 78.2, 52.3, 44.9, 34.4, 30.3, 28.9, 26.9; IR (cm⁻¹): 2969, 2929, 2871, 1656, 1474, 1338, 1021, 765, 703;
3r, isomer-1, low polarity
$3\alpha$, isomer-2, high polarity