## **Supporting Information**

# Visible-Light-Promoted Organic-Dye-Catalyzed Sulfonylation/Cyclization to Access Indolo[2,1-α]isoquinoline Derivatives

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#### **1. General Information**

All reagents and solvents were purchased from commercial suppliers and used without purifications. TLC was performed on silica gel plates (200-300mesh) using UV light (254/365 nm) for detection and column chromatography was performed on silicagel (200-300 mesh). The <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded at 25 °C in CDCl<sub>3</sub> at 400 and 100 MHz, respectively, with TMS as the internal standard. Chemical shifts ( $\delta$ ) are expressed in ppm and coupling constants *J* are given in Hz. All reactions were performed on the photoreaction instrument (WP-TEC-1020SL), which are purchased from WATTCAS, China (Figure S1).



Figure S1.Photoreactor for photoreaction

#### 2. Experimental Section

General procedures for the synthesis of ester substituted indolo[2,1- $\alpha$ ]isoquinolines and benzimidazo[2,1- $\alpha$ ]isoquinolin-6(5*H*)-ones.



To a suspension of 2-aryl-*N*-acryloyl indoles (0.2mmol) in DMSO (2mL) was added sulfonyl hydrazine(0.6 mmol, 3.0 eq.), Eosin B (5mol%) and  $(NH_4)_2S_2O_8$  (0.6 mmol, 3.0 eq.) at room temperature, and the mixture was stirred in air under a 10W blue LEDs and irradiated for 15 hours. The temperature was maintained at 20~25 °C when the LED light was on. After the reaction was complete, the reaction mixture was diluted with a brine solution (25 mL) and extracted with EtOAc (3 × 50 mL). The combined organic phase was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuo. The residue was purified by flashcolumn chromatography to afford the desired products.

#### **3.**Fluorescence quenching experiments

The fluorescence emission intensities were carried out on an F-7000 FL spectrophotometer (Hitachi Ltd, Japan) with excitation slit set at 5 nm and emission at 5 nm. The excitation wavelength was fixed at 323 nm, and the emission wavelength was measured at 350~550 nm. The samples were prepared by mixing Eosin B  $(1.0\times10^{-8}\text{mol/L})$  and different amount of  $(NH_4)_2S_2O_8$  in DMSO in a light path quartz fluorescence cuvette. The concentration of  $(NH_4)_2S_2O_8$  stock solution is  $1.3\times10^{-6}\text{mol/L}$  in DMSO. For each quenching experiment, 0.1mL of  $(NH_4)_2S_2O_8$  stock solution was titrated to a mixed solution of Eosin B (0.1mL, in a total volume = 1.0 mL). Then the emission intensity was collected and the results were presented in Figure S1.



Figure S1 Quenching of Eosin B fluorescence emission in the presence of  $(NH_4)_2S_2O_8$ .

## 4.<sup>1</sup>H and <sup>13</sup>C NMR spectra of products







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