

A three-component reaction of cyclobutanone oxime esters, sulfur dioxide and *N*-alkyl-*N*-methacryloyl benzamides

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Supporting Information

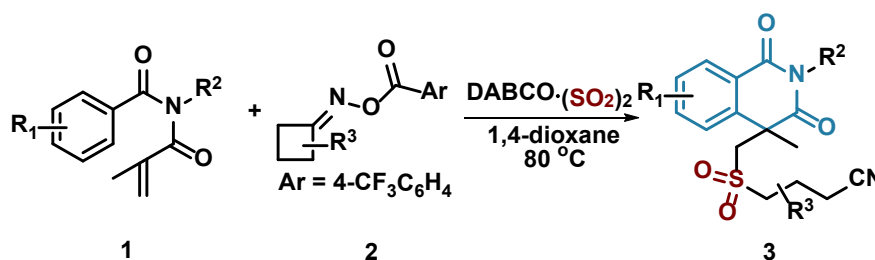
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1. General experimental methods:

Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32-63 μm , standard grade). Analytical thin-layer chromatography was performed using glass plates pre-coated with 0.25 mm 230-400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~ 20 Torr at 25-35 $^{\circ}\text{C}$. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^1H , and ^{13}C NMR spectra were recorded in CDCl_3 on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values were quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

2. General experimental procedure



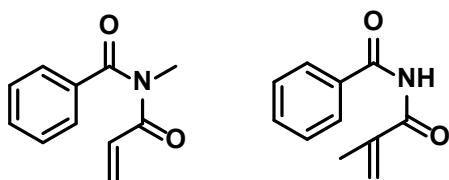
To an oven-dried tube (10 mL) equipped with a magnetic stir bar, *N*-alkyl-*N*-methacryloyl benzamide **1** (0.2 mmol), cycloketone oxime ester **2** (0.3 mmol), $\text{DABCO}\cdot(\text{SO}_2)_2$ (0.2 mmol), and 1,4-dioxane (2.0 mL) were added under N_2 atmosphere. The mixture was stirred at $80\text{ }^{\circ}\text{C}$ for 48 hours. After completion of the reaction as monitored by TLC analysis, The mixture was evaporated, and the residue was washed with saturated NaHCO_3 aqueous solution (2.0 mL). The aqueous phase was extracted with EtOAc (2.0mL X 3). The combined organic phase was evaporated, and the residue was purified directly by flash column chromatography (PE/EtOAc (v/v): 1/1) to provide the desired product **3**.

General procedure for scale-up reaction of N-alkyl-N-methacryloyl benzamide **1**, cycloketone oxime ester **2a** DABCO·(SO₂)₂

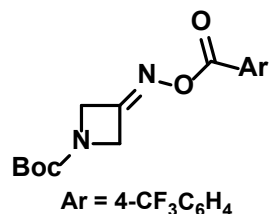
To an oven-dried sealed tube (100 mL) equipped with a magnetic stir bar, N-alkyl-N-methacryloyl benzamide **1a** (2.0 mmol), cycloketone oxime ester **2** (3.0 mmol), DABCO·(SO₂)₂ (2.0 mmol), and 1,4-dioxane (10 mL) were added under N₂ atmosphere. The mixture was stirred at 80 °C for 48 hours. After completion of the reaction as monitored by TLC analysis, The mixture was evaporated, and the residue was washed with saturated NaHCO₃ aqueous solution (20 mL). The aqueous phase was extracted with EtOAc (10 mL X 3). The combined organic phase was evaporated, and the residue was purified directly by flash column chromatography (PE/EtOAc (v/v): 1/1) to provide the desired product **3a** in 66% yield (440.9 mg).

3. Failed substrates

Failed acryloyl benzamides:



Failed cycloketone oxime esters:



4. ¹H and ¹³C NMR spectra of compounds 3

