Supporting Information
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Supporting Information

Domino Access to Yne-functionalized Benzoisoindolines from Triynes

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1. General experimental procedures

All the catalytic reactions were performed under an argon atmosphere using the over-dried Schlenk flask. The chemicals were purchased from Alfa Aesar and Acros Chemicals. All solvents and materials were pre-dried, redistilled or recrystallized before use. $^1$H NMR (300 MHz) and $^{13}$C NMR (75 MHz) spectra were recorded on a Bruker Avance 300 spectrometer with CDCl$_3$ as the solvent. Chemical shifts are reported in ppm by assigning TMS resonance in the $^1$H NMR spectra as 0.00 ppm and CDCl$_3$ resonance in the $^{13}$C spectra as 77.0 ppm. All coupling constants ($J$ values) were reported in Hertz (Hz). Column chromatography was performed on silica gel 300–400 mesh. Melting points were determined using a Gallenkamp melting point apparatus and are uncorrected. The FT-IR spectra were recorded from KBr pellets or thin film from CHCl$_3$ on the NaCl window in the 4000–400 cm$^{-1}$ ranges on a Nicolet 5DX spectrometer. All HRMS spectra were recorded using EI or APCI at 70 eV. X-ray Crystallography diffraction data of 2h was collected at room temperature with a Bruker SMART Apex CCD diffractometer with Mo-Kα radiation ($\lambda = 0.71073$ Å) with a graphite monochromator using the $\omega$-scan mode. Data reductions and absorption corrections were performed with SAINT and SADABS software, respectively. The structure was solved by direct methods and refined on $F^2$ by full-matrix least squares using SHELXTL. All non-hydrogen atoms were treated anisotropically. The positions of hydrogen atoms were generated geometrically.

**General procedures:**

**Preparation of Triynes:**

![Triyne Preparation Diagram]

Triyne substrates were prepared in one step using the Cadiot-Chodkiewicz coupling reaction. To a 30% $n$-BuNH$_2$ (3 mL/1 mmol of substrate) aqueous solution containing CuCl (1 equiv), and NH$_2$OH·HCl (0.1 equiv) was added diyne at 0 °C. Bromoalkyne (1.5 equiv) was then added dropwise over 5 min and the reaction mixture was stirred at 0 °C for additional 5 min. After aqueous work up, the crude product was purified by column chromatography on silica gel to afford triynes in moderate to good yields.

**Preparation of aryl halides:** Typical procedure: Triyne 1a-1w (1.0 equiv.) and CuI (10 mol %) were added to a degassed solution of CH$_3$CN (5 mL) and the mixture was stirred at room temperature for half an hour and then heated at 90 °C for 60 h. The reaction mixture was then cooled, quenched with water, and extracted with ethyl acetate (30 mL). The combined organic layers were washed with hydrochloric acid (5 %), sodium carbonate (5 %), and saturated sodium chloride solution, dried over MgSO$_4$, and...
concentrated. The residue was purified by flash column chromatography (eluent: petroleum ether/ethyl acetate = 6:1) to give 2a-2w.

2. X-Ray Structure for 2h
3. $^1$H NMR & $^{13}$C NMR Spectra for New Compounds