Supplementary Information

Iodine Promoted Cascade Cycloisomerization of 1-En-6,11-diynes

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**General Remarks**

Column chromatography was carried out on silica gel (200-300 mesh). $^1$H NMR spectra were recorded on 400 or 600 MHz in CDCl$_3$ and chemical shifts (ppm) were recorded with tetramethylsilane (TMS) as the internal reference standard. $^{13}$C NMR spectra were recorded on 100 or 150 MHz in CDCl$_3$, $^{19}$F NMR spectra were recorded on 376 MHz in CDCl$_3$. Multiplicities are given as: s (singlet), d (doublet), t (triplet), dd (doublet of doublets), dq (doublet of quartets), q (quartet) or m (multiplet). HR-MS was obtained using a Q-TOF or Q-Orbitrap instrument equipped with ESI source. The copies of $^1$H NMR, $^{13}$C NMR, and $^{19}$F NMR spectra of all compounds are provided in the Supporting Information. Room temperature is 23–25 °C. THF was distilled immediately before use from Na/benzophenone. 3-Ethoxypropanenitrile was purchased from Shanghai Bide Pharmaceutical Technology Co., Ltd without further purification. Other commercially available reagents and solvents were used without further purification.
General Procedures

For the Preparation of Starting Materials

For the synthesis of 1a (This procedure was also used for the synthesis of substrate 1b–1j, 1m–1n, 1p–1y):

Pd(PPh$_3$)$_2$Cl$_2$ (71.9 mg, 0.1 mmol, 1 mol %) and CuI (38.1 mg, 0.2 mmol, 2 mol %) were sequentially added to a stirred solution of 2-iodobenzaldehyde A (2.32 g, 10 mmol) in triethylamine (40 mL) under argon at room temperature. The mixture was allowed to stir for 10 min. Then N-allyl-4-methyl-N-(prop-2-yn-1-yl)benzenesulfonamide (2.74 g, 11 mmol, 1.1 equiv) was added. The mixture was allowed to stir overnight. An aqueous saturated solution of NH$_4$Cl (40 mL) was poured into the resulting mixture, and the mixture was extracted with ethyl acetate (2 × 50 mL). The organic layers were combined to be washed with brine and dried over Na$_2$SO$_4$ for 20 min. Then the solution would be concentrated under reduced pressure. The obtained residue would be further purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 5:1) to give N-allyl-N-(3-(2-formylphenyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide B (98%, 3.46 g, 9.8 mmol).
"n-BuLi (2.5 M, 2.9 mL, 1.4 equiv) was added dropwise via a syringe to a stirred solution of phenylacetylene (0.61 g, 6 mmol, 1.2 equiv) in dry THF (30 mL) under argon at −78 °C. The reaction mixture was allowed to stir for 10 min. Then, the solution of N-allyl-N-(3-(2-formylphenyl) prop-2-yn-1-yl)-4-methylbenzenesulfonamide B (1.77 g, 5 mmol) in THF was added at −78 °C. The reaction mixture was allowed to stir for 10 min at room temperature. After the completion of the reaction determined by TLC, the reaction mixture was quenched by an aqueous saturated solution of NH₄Cl (30 mL) and extracted with ethyl acetate (2 × 50 mL). The organic layers were combined to be washed with brine and dried over Na₂SO₄ for 20 min. Then the solution would be concentrated under reduced pressure. The obtained residue would be further purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 3:1) to give N-allyl-N-(3-(2-(1-hydroxy-3-phenylprop-2-yn-1-yl)phenyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide 1a (95%, 2.16 g, 4.75 mmol).

For the synthesis of 1o (This procedure was also used for the synthesis of substrate 1k and 1l):

Ethynylmagnesium bromide (0.5 mol/L in THF, 24 mL, 1.2 equiv) was added dropwise into a stirred solution of N-allyl-N-(3-(2-formylphenyl) prop-2-yn-1-yl)-4-methylbenzenesulfonamide B (3.53 g, 10 mmol) in THF (35 mL) under argon. The mixture was allowed to stir for 4 h at..."
room temperature. After the completion of the reaction determined by TLC, the reaction mixture was quenched by addition of an aqueous saturated solution of NH$_4$Cl (35 mL) and extracted with ethyl acetate (2×50 mL). The combined organic layers were washed with brine, dried over Na$_2$SO$_4$, and concentrated under reduced pressure. The resulting material $N$-allyl-$N$-(3-(2-(1-hydroxyprop-2-yn-1-yl)phenyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide C (90%, 3.42 g, 9 mmol) was directly used for the next step without further purification.

\[ \text{B} \xrightarrow{\text{MgBr}} \text{C} \xrightarrow{\text{Pd(PPh$_3$)$_2$Cl$_2$, CuI}} \text{C+} \]

Pd(PPh$_3$)$_2$Cl$_2$ (56.2 mg, 0.08 mmol, 1 mol %) and CuI (30.5 mg, 0.16 mmol, 2 mol %) were sequentially added to a stirred solution of 2-iodo-1-methoxy-4-nitrobenzene (2.68 g, 9.6 mmol, 1.2 equiv) in triethylamine (40 mL) under argon at room temperature. The mixture was allowed to stir for 10 min. Then $N$-allyl-$N$-(3-(2-(1-hydroxyprop-2-yn-1-yl)phenyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide C (3.04 g, 8 mmol) was added. The mixture was allowed to stir overnight. An aqueous saturated solution of NH$_4$Cl (40 mL) was poured into the resulting mixture, and the mixture was extracted with ethyl acetate (2 × 40 mL). The organic layers were combined to be washed with brine and dried over Na$_2$SO$_4$ for 20 min. Then the solution would be concentrated under
reduced pressure. The obtained residue would be further purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 2:1) to give $N$-allyl-$N$-(3-(2-(1-hydroxy-3-(2-methoxy-5-nitrophenyl)prop-2-yn-1-yl)phenyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide 1o (96%, 4.08 g, 7.7 mmol).

For the synthesis of 1z:

Compound 3-(3-chloro-4-methylphenyl)-1-(2-iodophenyl)prop-2-yn-1-ol D (98%) was synthesized via a similar preparation procedure as substrate 1a.

Substrate dimethyl 2-allyl-2-(3-(2-(3-chloro-4-methylphenyl)-1-hydroxyprop-2-yn-1-yl)phenyl)prop-2-yn-1-yl)malonate 1z (86%) was synthesized via a similar preparation procedure as compound B.
For the Synthesis of Intermediate Int-C

Acetophenone (1.2 g, 10 equiv, 10 mmol) and NaOH (0.6 g, 1.5 equiv, 15 mmol) were sequentially added to a stirred solution of 2-iodobenzaldehyde A (2.32 g, 10 mmol) in EtOH (40 mL) at room temperature. The mixture was allowed to stir for 3 h. An aqueous saturated solution of NH₄Cl (40 mL) was poured into the resulting mixture, and the mixture was extracted with ethyl acetate (2 × 40 mL). The organic layers were combined to be washed with brine and dried over Na₂SO₄ for 20 min. Then the solution would be concentrated under reduced pressure. The obtained residue would be further purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate, 20:1) to give 3-(2-iodophenyl)-1-phenylprop-2-en-1-one E (82%, 2.74 g, 8.2 mmol).

\[ \text{A} \quad \text{NaOH} \quad \text{EtOH, rt} \quad \text{B} \quad \text{Pd(PPh₃)₂Cl₂} \quad \text{CuI} \quad \text{E} \quad \text{N₄T₅} \quad \text{Int-C} \]

Compound \( N\text{-allyl-4-methyl-N-(3-(2-(3-oxo-3-phenylprop-1-en-1-yl)phenyl)prop-2-yn-1-yl)benzenesulfonamide} \) Int-C (95%) was synthesized via a similar preparation procedure as substrate 1a.
For the Synthesis of Products

For the synthesis of 2a:

Water (7.2 µL, 0.40 mmol, 2.0 eqiv) and iodine (50.8 mg, 0.2 mmol, 1.0 equiv) were sequentially added to an oven-dried tube charged with of N-allyl-N-(3-(2-(1-hydroxy-3-phenylprop-2-yn-1-yl)phenyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide (1a; 91.0 mg, 0.2 mmol) in 3-ethoxypropanitrile. The resulting mixture was allowed to stir at 80 °C for 0.5 h. And the reaction mixture was quenched with a saturated aqueous solution of Na₂S₂O₃ (3–5 mL) and then extracted with ethyl ether (2×15 mL), washed with saturated brine, dried over Na₂SO₄, and evaporated under reduced pressure. The residue was further purified by chromatography on silica gel (petroleum ether/ethyl acetate, 10:1) to afford the product (1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)(phenyl)methanone 2a in 82% yield.
**X-ray Single Crystal Diffraction Data**

The ellipsoid contour percent probability level is 50% in the caption of the thermal ellipsoid plot. CCDC 1913985

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Npar= 326
Characterization Data

Characterization Data of 2a–2v

(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)(phenyl)methanone (2a): white solid; 95.4 mg; 82% yield; 1H NMR (400 MHz, CDCl₃) δ ppm 7.92 (d, J = 8.4 Hz, 1H), 7.84–7.77 (m, 6H), 7.73–7.69 (m, 1H), 7.65 (t, J = 7.6 Hz, 1H), 7.57–7.48 (m, 3H), 7.37 (d, J = 8.0 Hz, 2H), 4.77 (d, J = 16.8 Hz, 1H), 4.44 (d, J = 12.0 Hz, 1H), 4.12 (d, J = 16.4 Hz, 1H), 3.84 (d, J = 10.8 Hz, 1H), 3.70 (t, J = 10.8 Hz, 1H), 3.53 (d, J = 10.0 Hz, 1H), 2.75 (d, J = 11.6 Hz, 1H), 2.43 (s, 3H); 13C{¹H} NMR (100 MHz, CDCl₃) δ ppm 196.8, 143.9, 137.4, 133.4, 132.4, 132.1, 131.9, 131.0, 130.4, 129.9, 129.8, 129.3, 128.5, 128.2, 127.8, 126.7, 122.4, 47.5, 45.9, 40.1, 21.5, 7.7; HRMS (ESI/Q-TOF) m/z [M+H]⁺ calcd for C₂₉H₂₆INO₅S 582.0594, found 582.0593 (0.2 ppm).

(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)(p-tolyl)methanone (2b): light yellow solid; 89.3 mg; 75% yield; 1H NMR (400 MHz, CDCl₃) δ ppm 7.91 (d, J = 8.4 Hz, 1H), 7.81–7.68 (m, 7H), 7.54 (t, J = 7.6 Hz, 1H), 7.36 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 4.75 (d, J = 16.4 Hz, 1H), 4.43 (d, J = 12.0 Hz, 1H), 4.10 (d, J = 16.4 Hz, 1H), 3.83 (d, J = 11.2 Hz, 1H), 3.69 (t, J = 10.8 Hz, 1H), 3.53 (d, J = 10.4 Hz, 1H), 2.74 (d, J = 11.6 Hz, 1H), 2.46 (s, 3H), 2.43 (s, 3H); 13C{¹H} NMR (100 MHz, CDCl₃) δ ppm 196.5, 144.5, 143.8, 134.7, 133.8, 132.4, 132.0, 131.8, 131.0, 130.6, 130.5, 129.8, 129.8, 129.2, 129.1, 128.0, 127.8, 126.7, 122.4, 47.4, 45.9, 40.1, 21.7, 21.5, 7.8; HRMS (ESI/Q-TOF) m/z [M+Na]⁺ calcd for C₂₉H₂₆INNaO₅S 618.0570, found 618.0570 (0 ppm).

(4-ethylphenyl)(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)methanone (2c): yellow solid; 86.5 mg; 71% yield; 1H NMR (400 MHz, CDCl₃) δ ppm 7.91 (d, J = 8.4 Hz, 1H), 7.81–7.76 (m, 6H), 7.70 (t, J = 7.6 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.37–7.31 (m, 4H), 4.75 (d, J = 16.4 Hz, 1H), 4.43 (d, J = 11.6 Hz, 1H), 4.10 (d, J = 16.4 Hz, 1H), 3.83 (d, J = 10.8 Hz, 1H), 3.69 (t, J = 10.8 Hz, 1H), 3.53 (d, J = 10.0 Hz, 1H), 2.79–2.73 (m, 3H), 2.43 (s, 3H), 1.30 (t, J = 7.6 Hz, 3H); 13C{¹H} NMR (100 MHz, CDCl₃) δ ppm 196.5, 150.6,
143.8, 134.9, 133.8, 132.4, 132.0, 131.8, 131.0, 130.6, 130.5, 129.8, 129.8, 129.1, 128.0, 127.8, 126.7, 122.4, 47.4, 45.9, 40.1, 29.0, 21.5, 15.2, 7.8; HRMS (ESI/Q-TOF) m/z [M+H]⁺ calcd for C₃₀H₂₉INO₃S 610.0907, found 610.0907 (0 ppm).

(4-(tert-butyl)phenyl)(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)methanone (2d): light yellow solid; 98.2 mg; 77% yield; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.93 (d, J = 8.4 Hz, 1H), 7.83–7.78 (m, 6H), 7.71 (t, J = 7.6 Hz, 1H), 7.54 (dd, J = 18.0 Hz 7.6 Hz, 3H), 7.37 (d, J = 8.0 Hz, 2H), 4.74 (d, J = 16.8 Hz, 1H), 4.43 (d, J = 12.0 Hz, 1H), 4.11 (d, J = 16.4 Hz, 1H), 3.84 (t, J = 10.8 Hz, 1H), 3.70 (t, J = 10.8 Hz, 1H), 3.54 (d, J = 10.0 Hz, 1H), 2.77 (d, J = 12.0 Hz, 1H), 2.43 (s, 3H), 1.38 (s, 9H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ ppm 196.5, 157.5, 143.9, 134.7, 133.9, 132.7, 132.1, 131.9, 131.1, 130.5, 130.5, 129.9, 129.8, 129.1, 128.2, 127.9, 126.7, 125.6, 122.5, 47.5, 45.9, 40.2, 35.2, 31.1, 21.5, 7.8; HRMS (ESI/Q-TOF) m/z [M+Na]⁺ calcd for C₃₂H₃₂INO₃NaS 660.1040, found 660.1041 (0.2 ppm).

(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)(4-pentylphenyl)methanone (2e): light yellow solid; 75.6 mg; 58% yield; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.92 (d, J = 8.4 Hz, 1H), 7.84–7.78 (m, 6H), 7.75–7.69 (m, 1H), 7.55 (t, J = 7.6 Hz, 1H), 7.37 (d, J = 7.6 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 4.74 (d, J = 16.4 Hz, 1H), 4.43 (d, J = 11.6 Hz, 1H), 4.11 (d, J = 16.4 Hz, 1H), 3.84 (d, J = 10.8 Hz, 1H), 3.70 (t, J = 10.8 Hz, 1H), 3.54 (d, J = 10.0 Hz, 1H), 2.77–2.67 (m, 3H), 2.43 (s, 3H), 1.68 (t, J = 6.8 Hz, 2H), 1.36 (d, J = 6.8 Hz, 4H), 0.93–0.90 (m, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ ppm 196.6, 149.6, 143.9, 134.9, 133.9, 132.5, 132.1, 131.9, 131.0, 130.7, 130.5, 129.9, 129.8, 129.1, 128.6, 128.2, 127.9, 126.7, 122.5, 47.5, 45.9, 40.2, 36.0, 31.4, 30.8, 22.5, 21.5, 14.0, 7.8; HRMS (ESI/Q-TOF) m/z [M+H]⁺ calcd for C₃₃H₃₅INO₃S 652.1377, found 652.1376 (0.2 ppm).

(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)(4-methoxyphenyl)methanone (2f): light yellow solid; 75.8 mg; 62% yield; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.92 (d, J = 8.4 Hz, 1H), 7.84–7.81 (m, 3H), 7.78–7.76 (m, 3H), 7.72–7.69 (m, 1H), 7.55 (t, J = 7.8 Hz, 1H), 7.36 (d, J = 7.8 Hz, 2H), 6.97 (d, J = 9.0 Hz, 2H), 4.70 (d, J = 16.2 Hz, 1H), 4.43
(d, J = 12.0 Hz, 1H), 4.08 (d, J = 16.2 Hz, 1H), 3.91 (s, 3H), 3.84–3.82 (m, 1H), 3.71–3.67 (m, 1H), 3.54 (d, J = 10.2 Hz, 1H), 2.76 (d, J = 11.4 Hz, 1H), 2.43 (s, 3H); \(^{13}\)C\(^{1}\)H NMR (150 MHz, CDCl\(_3\)) \(\delta\) ppm 195.4, 164.1, 143.9, 134.3, 132.8, 132.6, 132.0, 131.7, 131.2, 130.1, 129.9, 129.7, 129.6, 129.0, 128.0, 127.9, 126.7, 122.5, 113.9, 55.6, 47.4, 46.0, 40.2, 21.5, 7.7; HRMS (ESI/Q-TOF) \(m/z\) [M+H]\(^+\) calcd for C\(_{29}\)H\(_{33}\)INO\(_3\)S 612.0700, found 612.0700 (0 ppm).

![Image](image-url)

\(1,1'-\text{biphenyl}-4-\text{yl}(1-\text{iodomethyl})-3-\text{tosyl}-1,2,3,4-\text{tetrahydrobenzof}[\text{f]isoquinolin-5-yl\text{meth thanone (2g)}}:\) yellow solid; 93.4 mg; 71\% yield; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.92 (t, J = 8.4 Hz, 3H), 7.87–7.83 (m, 2H), 7.78 (d, J = 7.6 Hz, 2H), 7.72 (d, J = 8.0 Hz, 3H), 7.67 (d, J = 7.2 Hz, 2H), 7.56 (t, J = 7.6 Hz, 1H), 7.50 (t, J = 7.2 Hz, 2H), 7.44–7.40 (m, 1H), 7.35 (d, J = 7.6 Hz, 2H), 4.78 (d, J = 16.4 Hz, 1H), 4.44 (d, J = 12.0 Hz, 1H), 4.12 (d, J = 16.8 Hz, 1H), 3.85 (d, J = 10.8 Hz, 1H), 3.71 (t, J = 10.4 Hz, 1H), 3.54 (d, J = 10.0 Hz, 1H), 2.75 (d, J = 11.6 Hz, 1H), 2.41 (s, 3H); \(^{13}\)C\(^{1}\)H NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 196.4, 146.2, 143.9, 139.6, 136.1, 133.7, 132.5, 132.2, 131.9, 131.1, 131.0, 130.8, 130.5, 129.9, 129.3, 129.0, 128.4, 128.1, 127.8, 127.3, 127.2, 126.8, 122.5, 47.6, 45.9, 40.2, 21.5, 7.8; HRMS (ESI/Q-TOF) \(m/z\) [M+H]\(^+\) calcd for C\(_{34}\)H\(_{35}\)INO\(_3\)S 658.0907, found 658.0907 (0 ppm).

![Image](image-url)

\((4-\text{fluorophenyl})(1-\text{iodomethyl})-3-\text{tosyl}-1,2,3,4-\text{tetrahydrobenzof}[\text{f]isoquinolin-5-yl\text{meth anone (2h)}}:\) yellow solid; 95.9 mg; 80\% yield; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.92 (d, J = 8.4 Hz, 1H), 7.89–7.59 (m, 2H), 7.80 (dd, J = 16.0 Hz 8.4 Hz, 4H), 7.73 (t, J = 8.0 Hz, 1H), 7.56 (t, J = 7.6 Hz, 1H), 7.37 (d, J = 8.0 Hz, 2H), 7.18 (t, J = 8.4 Hz, 2H), 4.73 (d, J = 16.8 Hz, 1H), 4.43 (d, J = 11.6 Hz, 1H), 4.09 (d, J = 16.4 Hz, 1H), 3.84 (d, J = 10.8 Hz, 1H), 3.69 (t, J = 10.8 Hz, 1H), 3.52 (d, J = 10.0 Hz, 1H), 2.75 (d, J = 11.6 Hz, 1H), 2.44 (s, 3H); \(^{13}\)C\(^{1}\)H NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 195.2, 166.0 (d, \(^1\)J = 254 Hz, 1C), 144.0, 133.7 (d, \(^3\)J = 3 Hz, 1C), 133.3, 133.1, 133.0, 132.4, 132.3, 132.0, 131.0, 130.7, 129.9, 129.4, 128.0, 127.8, 126.9, 122.5, 115.8 (d, \(^2\)J = 22 Hz, 1C), 47.5, 45.9, 40.1, 21.5, 7.6; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) ppm –104.27—104.34 (m, 1F); HRMS (ESI/Q-TOF) \(m/z\) [M+Na]\(^+\) calcd for C\(_{28}\)H\(_{29}\)FINNaO\(_3\)S 622.0320, found 622.0320 (0 ppm).

![Image](image-url)

\((4-\text{chlorophenyl})(1-\text{iodomethyl})-3-\text{tosyl}-1,2,3,4-\text{tetrahydrobenzof}[\text{f]isoquinolin-5-yl\text{meth}

s12
anone (2i): yellow solid; 97.3 mg; 79% yield; ¹H NMR (600 MHz, CDCl₃) δ ppm 7.93 (d, J = 8.4 Hz, 1H), 7.82 (d, J = 7.8 Hz, 1H), 7.79−7.77 (m, 5H), 7.73 (t, J = 7.8 Hz, 1H), 7.57 (t, J = 7.8 Hz, 1H), 7.48 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.4 Hz, 2H), 4.74 (d, J = 16.8 Hz, 1H), 4.43 (d, J = 12.0 Hz, 1H), 4.09 (d, J = 16.8 Hz, 1H), 3.84 (d, J = 10.8 Hz, 1H), 3.70−3.67 (m, 1H), 3.52 (d, J = 10.2 Hz, 1H), 2.76 (d, J = 11.4 Hz, 1H), 2.44 (s, 3H); ¹³C{¹H} NMR (150 MHz, CDCl₃) δ ppm 195.6, 144.0, 140.0, 135.8, 133.2, 132.5, 132.4, 132.1, 131.7, 131.1, 131.0, 129.9, 129.9, 129.5, 128.9, 128.1, 127.9, 126.9, 122.5, 47.6, 45.9, 40.2, 21.5, 7.6; HRMS (ESI/Q-TOF) m/z [M+H]⁺ calcd for C₂₈H₂₅ClINO₅S 616.0205, found 616.0205 (0 ppm).

(4-bromophenyl)(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzof[f]isoquinolin-5-yl)methanone (2j): yellow solid; 110.9 mg; 84% yield; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.98 (s, 1H), 7.83−7.76 (m, 6H), 7.71 (s, 1H), 7.66 (t, J = 7.6 Hz, 1H), 7.53−7.49 (m, 2H), 7.37 (d, J = 8.0 Hz, 2H), 4.72 (d, J = 16.8 Hz, 1H), 4.42 (d, J = 12.0 Hz, 1H), 4.07 (d, J = 16.4 Hz, 1H), 3.79 (d, J = 10.8 Hz, 1H), 3.71−3.66 (m, 1H), 3.46 (d, J = 10.0 Hz, 1H), 2.76 (d, J = 12.0 Hz, 1H), 2.44 (s, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ ppm 195.7, 144.0, 136.2, 133.0, 132.4, 132.1, 131.9, 131.8, 131.1, 131.0, 129.9, 129.9, 129.5, 128.7, 128.1, 127.8, 126.9, 122.5, 47.6, 45.9, 40.1, 21.5, 7.6; HRMS (ESI/Q-TOF) m/z [M+Na]⁺ calcd for C₂₈H₂₅BrINNaO₅S 681.9519, found 681.9520 (0.1 ppm).

1-(4-(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzof[f]isoquinoline-5-carbonyl)phenyl)et han-1-one (2k): yellow solid; 77.3 mg; 62% yield; ¹H NMR (400 MHz, CDCl₃) δ ppm 8.06 (d, J = 8.0 Hz, 2H), 7.94−7.89 (m, 3H), 7.82−7.78 (m, 4H), 7.74 (t, J = 7.6 Hz, 1H), 7.56 (t, J = 7.6 Hz, 1H), 7.38 (d, J = 7.6 Hz, 2H), 4.82 (d, J = 16.4 Hz, 1H), 4.44 (d, J = 12.0 Hz, 1H), 4.14 (d, J = 16.8 Hz, 1H), 3.84 (d, J = 10.8 Hz, 1H), 3.69 (t, J = 10.8 Hz, 1H), 3.52 (d, J = 10.0 Hz, 1H), 2.76 (d, J = 12.0 Hz, 1H), 2.68 (s, 3H), 2.44 (s, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ ppm 197.3, 196.1, 143.9, 141.0, 140.2, 132.7, 132.5, 132.2, 131.8, 130.9, 130.4, 130.0, 129.9, 129.7, 128.3, 128.3, 127.8, 126.9, 122.5, 47.6, 45.8, 40.2, 26.9, 21.5, 7.6; HRMS (ESI/Q-Orbitrap) m/z [M+H]⁺ calcd for C₃₀H₂₅INO₅S 624.0700, found 624.0703 (0.5 ppm).

513
4-(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzf[f]isoquinoline-5-carbonyl)benzonitrile (2l): white solid; 66.7 mg; 55% yield; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) ppm 7.95–7.92 (m, 3H), 7.83–7.79 (m, 6H), 7.76 (t, \(J = 7.8\) Hz, 1H), 7.59 (t, \(J = 7.8\) Hz, 1H), 7.39 (d, \(J = 8.4\) Hz, 2H), 4.80 (d, \(J = 16.8\) Hz, 1H), 4.44 (d, \(J = 12.0\) Hz, 1H), 4.13 (d, \(J = 16.8\) Hz, 1H), 3.85 (d, \(J = 10.8\) Hz, 1H), 3.69 (t, \(J = 10.8\) Hz, 1H), 3.51 (d, \(J = 10.2\) Hz, 1H), 2.77 (d, \(J = 11.4\) Hz, 1H), 2.45 (s, 3H); \(^{13}\)C\(^{\text{1}}\)H NMR (150 MHz, CDCl\(_3\)) \(\delta\) ppm 195.2, 144.1, 141.1, 132.8, 132.6, 132.4, 132.3, 132.2, 132.0, 130.9, 130.6, 130.1, 130.0, 129.9, 128.4, 127.9, 127.1, 122.6, 117.8, 116.5, 47.7, 45.9, 40.3, 21.5, 7.4; HRMS (ESI/Q-Orbitrap) \(m/z\) [M+Na]\(^+\) calcd for C\(_{28}\)H\(_{23}\)IN\(_2\)NaO\(_3\)S 629.0366, found 629.0365 (0.2 ppm).

\(\text{C NMR}\) spectrum

(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzf[f]isoquinolin-5-yl)(m-tolyl)methanone (2m): light yellow solid; 81.0 mg; 68% yield; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.91 (d, \(J = 8.4\) Hz, 1H), 7.80–7.76 (m, 4H), 7.69 (dd, \(J = 15.2\) Hz 7.6 Hz, 2H), 7.59 (d, \(J = 7.6\) Hz, 1H), 7.54 (t, \(J = 7.6\) Hz, 1H), 7.45 (d, \(J = 7.6\) Hz, 1H), 7.37 (t, \(J = 7.6\) Hz, 3H), 4.77 (d, \(J = 8.4\) Hz, 1H), 4.43 (d, \(J = 11.6\) Hz, 1H), 4.11 (d, \(J = 16.8\) Hz, 1H), 3.82 (d, \(J = 10.8\) Hz, 1H), 3.69 (t, \(J = 10.8\) Hz, 1H), 3.52 (d, \(J = 10.0\) Hz, 1H), 2.74 (d, \(J = 12.0\) Hz, 1H), 2.42 (s, 3H), 2.41 (s, 3H); \(^{13}\)C\(^{\text{1}}\)H NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 197.0, 143.8, 138.4, 137.4, 134.2, 133.6, 132.4, 132.0, 131.8, 130.9, 130.8, 130.6, 129.8, 129.8, 129.2, 128.3, 128.1, 127.7, 127.7, 126.7, 122.3, 47.5, 45.8, 40.0, 21.5, 21.2, 7.7; HRMS (ESI/Q-TOF) \(m/z\) [M+Na]\(^+\) calcd for C\(_{28}\)H\(_{23}\)IN\(_2\)NaO\(_3\)S 618.0570, found 618.0570 (0 ppm).

(3-chlorophenyl)(1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzf[f]isoquinolin-5-yl)meth anone (2n): light yellow solid; 91.2 mg; 74% yield; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) ppm 7.93 (d, \(J = 8.4\) Hz, 1H), 7.84–7.80 (m, 5H), 7.78–7.68 (m, 2H), 7.63–7.55 (m, 2H), 7.45 (t, \(J = 8.0\) Hz, 1H), 7.38 (d, \(J = 8.0\) Hz, 2H), 4.76 (d, \(J = 16.4\) Hz, 1H), 4.44 (d, \(J = 10.4\) Hz, 1H), 4.10 (d, \(J = 16.4\) Hz, 1H), 3.84 (d, \(J = 10.8\) Hz, 1H), 3.70 (t, \(J = 10.8\) Hz, 1H), 3.53 (d, \(J = 10.4\) Hz, 1H), 2.76 (d, \(J = 11.6\) Hz, 1H), 2.44 (s, 3H); \(^{13}\)C\(^{\text{1}}\)H NMR (100 MHz, CDCl\(_3\)) \(\delta\) ppm 195.4, 144.0, 139.1, 134.9, 133.3, 132.7, 132.4, 132.1, 131.3, 130.9, 130.1, 130.0, 129.9, 129.8, 129.6, 128.5, 128.2, 127.8, 126.9, 122.5, 47.5, 45.9, 40.2, 21.5, 7.6 (One of the peak of aryl group is overlapped in \(^{13}\)C NMR spectrum); HRMS (ESI/Q-TOF) \(m/z\) [M+H]\(^+\) calcd for C\(_{28}\)H\(_{23}\)ClINO\(_3\)S 616.0205, found 616.0205 (0 ppm).
(I-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)(2-methoxy-5-nitrophe nyl)methanone (2o): light yellow solid; 61.7 mg; 47% yield; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ ppm 8.42 (dd, $J = 9.6$ Hz 3.0 Hz, 1H), 8.32 (d, $J = 2.4$ Hz, 1H), 7.92 (d, $J = 8.4$ Hz, 1H), 7.86–7.82 (m, 3H), 7.78–7.73 (m, 2H), 7.54 (t, $J = 7.8$ Hz, 1H), 7.40 (d, $J = 8.4$ Hz, 2H), 7.13 (d, $J = 9.0$ Hz, 1H), 5.07 (d, $J = 17.4$ Hz, 1H), 4.46 (d, $J = 11.4$ Hz, 1H), 4.22 (d, $J = 16.8$ Hz, 1H), 3.85–3.83 (m, 4H), 3.72 (t, $J = 10.8$ Hz, 1H), 3.53 (d, $J = 10.2$ Hz, 1H), 2.78 (d, $J = 12.0$ Hz, 1H), 2.45 (s, 3H); $^{13}$C($^1$H) NMR (150 MHz, CDCl$_3$) $\delta$ ppm 194.6, 162.2, 143.9, 141.1, 133.5, 132.9, 132.7, 132.6, 132.4, 131.1, 130.3, 130.1, 129.9, 129.6, 128.8, 128.2, 127.8, 126.8, 125.8, 122.5, 111.7, 56.7, 48.1, 45.7, 40.2, 21.5, 7.7; HRMS (ESI/Q-Orbitrap) $m/z$ [M+H]$^+$ calcd for C$_{39}$H$_{26}$IN$_2$O$_5$S 657.0551, found 657.0551 (0 ppm).

(3,5-dimethylphenyl)(I-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl) methanone (2p): light yellow solid; 80.5 mg; 66% yield; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 7.92 (d, $J = 8.4$ Hz, 1H), 7.82–7.77 (m, 4H), 7.70 (t, $J = 7.6$ Hz, 1H), 7.54 (t, $J = 7.6$ Hz, 1H), 7.42 (s, 2H), 7.36 (d, $J = 7.6$ Hz, 2H), 7.28 (s, 1H), 4.75 (d, $J = 16.4$ Hz, 1H), 4.44 (d, $J = 12.0$ Hz, 1H), 4.10 (d, $J = 16.8$ Hz, 1H), 3.83 (d, $J = 11.2$ Hz, 1H), 3.70 (t, $J = 10.8$ Hz, 1H), 3.53 (d, $J = 10.0$ Hz, 1H), 2.75 (d, $J = 11.6$ Hz, 1H), 2.42 (s, 3H), 2.36 (s, 6H); $^{13}$C($^1$H) NMR (100 MHz, CDCl$_3$) $\delta$ ppm 197.2, 143.8, 138.2, 137.5, 135.2, 133.9, 132.4, 131.9, 131.8, 131.0, 130.6, 129.8, 129.1, 128.1, 127.8, 127.6, 126.6, 122.4, 47.5, 45.9, 40.0, 21.5, 21.1, 7.7; HRMS (ESI/Q-TOF) $m/z$ [M+H]$^+$ calcd for C$_{39}$H$_{26}$INO$_5$S 610.0907, found 610.0906 (0.2 ppm).

(3,5-dichlorophenyl)(I-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzo[f]isoquinolin-5-yl)m ethanone (2q): yellow solid; 97.6 mg; 75% yield; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 7.93 (d, $J = 8.4$ Hz, 1H), 7.85 (d, $J = 8.0$ Hz, 1H), 7.81–7.74 (m, 4H), 7.67 (d, $J = 0.8$ Hz, 2H), 7.60–7.57 (m, 2H), 7.39 (d, $J = 7.6$ Hz, 2H), 4.76 (d, $J = 16.4$ Hz, 1H), 4.44 (d, $J = 11.6$ Hz, 1H), 4.09 (d, $J = 16.8$ Hz, 1H), 3.84 (d, $J = 10.8$ Hz, 1H), 3.69 (t, $J = 10.8$ Hz, 1H), 3.52 (d, $J = 10.4$ Hz, 1H), 2.75 (d, $J = 11.6$ Hz, 1H), 2.44 (s, 3H); $^{13}$C($^1$H) NMR (100 MHz, CDCl$_3$) $\delta$ ppm 194.0, 144.0, 140.2, 135.5, 132.9, 132.7, 132.4, 132.3, 132.0, 131.6, 130.8, 130.1, 129.9,
128.5, 128.2, 127.8, 127.0, 122.5, 47.5, 45.8, 40.1, 21.5, 7.5; HRMS (ESI/Q-TOF) m/z [M+H]^+ calcd for C_{28}H_{23}ClINO_3S 649.9815, found 649.9815 (0 ppm).

(3,4-dimethylphenyl)(1-iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzof[f]isoquinolin-5-yl) methanone (2r): yellow solid; 95.1 mg; 78% yield; ^1H NMR (400 MHz, CDCl_3) δ ppm 7.93 (d, J = 8.8 Hz, 1H), 7.82–7.77 (m, 4H), 7.73–7.68 (m, 1H), 7.64 (s, 1H), 7.57–7.52 (m, 2H), 7.36 (d, J = 8.0 Hz, 2H), 7.25–7.23 (m, 2H), 4.73 (d, J = 16.4 Hz, 1H), 4.44 (d, J = 11.6 Hz, 1H), 4.11 (d, J = 16.4 Hz, 1H), 3.84 (d, J = 10.8 Hz, 1H), 3.71 (t, J = 10.8 Hz, 1H), 3.54 (d, J = 10.0 Hz, 1H), 2.76 (d, J = 11.6 Hz, 1H), 2.43 (s, 3H), 2.37 (s, 3H), 2.32 (s, 3H); ^13C(^1H) NMR (100 MHz, CDCl_3) δ ppm 196.9, 143.9, 143.4, 137.1, 135.2, 134.1, 132.5, 132.0, 131.8, 131.3, 131.1, 130.5, 129.9, 129.8, 129.8, 129.1, 128.4, 128.2, 127.9, 126.7, 122.5, 47.5, 46.0, 40.2, 21.5, 20.2, 19.8, 7.8; HRMS (ESI/Q-TOF) m/z [M+Na]^+ calcd for C_{30}H_{28}INaO_3S 632.0727, found 632.0727 (0 ppm).

(3-chloro-4-methylphenyl)(1-iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzof[f]isoquinolin-5-yl) methanone (2s): yellow solid; 107.1 mg; 85% yield; ^1H NMR (400 MHz, CDCl_3) δ ppm 7.91 (d, J = 8.4 Hz, 1H), 7.82–7.77 (m, 5H), 7.73–7.69 (m, 1H), 7.61–7.53 (m, 2H), 7.38–7.34 (m, 3H), 7.56 (d, J = 16.4 Hz, 1H), 4.43 (d, J = 12.0 Hz, 1H), 4.09 (d, J = 16.4 Hz, 1H), 3.82 (d, J = 10.8 Hz, 1H), 3.68 (t, J = 10.8 Hz, 1H), 3.51 (d, J = 10.8 Hz, 1H), 2.74 (d, J = 12.0 Hz, 1H), 2.47 (s, 3H), 2.43 (s, 3H); ^13C(^1H) NMR (100 MHz, CDCl_3) δ ppm 195.1, 143.9, 142.2, 136.6, 134.8, 132.9, 132.3, 132.2, 131.9, 130.9, 130.9, 130.6, 129.9, 129.8, 129.3, 128.6, 128.0, 127.7, 126.8, 122.4, 47.4, 45.8, 40.0, 21.5, 20.4, 7.6; HRMS (ESI/Q-TOF) m/z [M+Na]^+ calcd for C_{30}H_{28}INaO_3S 652.0181, found 652.0181 (0 ppm).

(1-iodomethyl)-8-methyl-3-tosyl-1,2,3,4-tetrahydrobenzof[f]isoquinolin-5-yl)(phenyl)methanone (2t): light yellow solid; 104.8 mg; 88% yield; ^1H NMR (400 MHz, CDCl_3) δ ppm 7.84–7.78 (m, 5H), 7.74 (s, 1H), 7.65 (t, J = 7.6 Hz, 1H), 7.58–7.48 (m, 4H), 7.37 (d, J = 8.0 Hz, 2H), 4.75 (d, J = 16.4 Hz, 1H), 4.42 (d, J = 12.0 Hz, 1H), 4.10 (d, J = 16.4 Hz, 1H), 3.81 (d, J = 10.8 Hz, 1H), 3.70 (t, J = 10.8 Hz, 1H), 3.52 (d, J = 9.6 Hz, 1H), 2.75 (d, J = 12.0 Hz, 1H), 2.51 (s, 3H), 2.43 (s, 3H); ^13C(^1H) NMR (100 MHz, CDCl_3) δ ppm 197.0, 143.9, 137.6,
136.7, 133.5, 133.4, 132.5, 132.0, 131.5, 131.3, 130.6, 130.4, 130.2, 129.9, 128.9, 128.5, 127.9, 127.3, 122.3, 47.6, 45.9, 40.2, 21.5, 21.3, 7.9; HRMS (ESI/Q-TOF) m/z [M+Na]^+ calcd for C_{29}H_{26}INaO_{3}S 618.0570, found 618.0569 (0.2 ppm).

(8-chloro-1-(iodomethyl)-3-tosyl-1,2,3,4-tetrahydrobenzof[f]isoquinolin-5-yl)(phenyl) methanone (2u): deep yellow solid; 76.4 mg; 62% yield; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.86–7.81 (m, 3H), 7.76 (d, J = 8.0 Hz, 3H), 7.70–7.60 (m, 3H), 7.50 (t, J = 7.6 Hz, 2H), 7.36 (d, J = 8.0 Hz, 2H), 4.73 (d, J = 16.4 Hz, 1H), 4.42 (d, J = 12.0 Hz, 1H), 4.07 (d, J = 12.4 Hz, 1H), 3.78 (d, J = 10.8 Hz, 1H), 3.68 (t, J = 10.8 Hz, 1H), 3.45 (d, J = 9.6 Hz, 1H), 2.75 (d, J = 11.6 Hz, 1H), 2.43 (s, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ ppm 196.4, 143.9, 137.0, 134.7, 133.7, 132.6, 132.4, 132.3, 131.8, 130.3, 130.1, 129.8, 129.9, 129.5, 128.6, 128.4, 128.3, 127.7, 124.2, 47.6, 45.9, 40.2, 21.5, 7.3; HRMS (ESI/Q-TOF) m/z [M+Na]^+ calcd for C_{28}H_{23}ClINaO_{3}S 638.0024, found 638.0023 (0.2 ppm).

Characterization Data of 2y and 2z

Pheny(1-(prop-1-en-2-yl)-3-tosyl-1,2,3,4-tetrahydrobenzof[f]isoquinolin-5-yl) methanone (2y): yellow solid; 67.4 mg; 70% yield; ¹H NMR (400 MHz, CDCl₃) δ ppm 7.89–7.83 (m, 3H), 7.79–7.71 (m, 4H), 7.64 (t, J = 7.6 Hz, 1H), 7.59–7.55 (m, 1H), 7.51–7.46 (m, 3H), 7.32
(d, J = 8.0 Hz, 2H), 5.01 (m, 1H), 4.78 (d, J = 16.4 Hz, 1H), 4.48 (s, 1H), 4.23 (d, J = 16.4 Hz, 1H), 4.10−4.04 (m, 2H), 2.93 (dd, J = 11.6 Hz 3.6 Hz, 1H), 2.41 (s, 3H), 1.96 (s, 3H); $^{13}$C[$^1$H] NMR (100 MHz, CDCl$_3$) $\delta$ ppm 197.2, 145.1, 143.5, 137.7, 133.5, 133.3, 133.1, 132.9, 132.4, 130.8, 130.5, 130.4, 129.7, 129.3, 128.6, 128.5, 128.3, 127.7, 126.4, 123.9, 115.9, 47.1, 47.0, 43.6, 21.8, 21.5; HRMS (ESI/Q-TOF) m/z [M+H]$^+$ calcd for C$_{30}$H$_{28}$NO$_3$S 482.1784, found 482.1784 (0 ppm).

**dimethyl 10-(3-chloro-4-methylbenzoyl)-4-(iodomethyl)-3,4-dihydrophenanthrene-2,2(1H)-dicarboxylate (2z):** yellow solid; 89.8 mg; 76% yield; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 7.92 (d, J = 8.8 Hz, 1H), 7.83 (d, J = 7.6 Hz, 2H), 7.71 (s, 1H), 7.68−7.63 (m, 2H), 7.53 (t, J = 7.6 Hz, 1H), 7.35 (d, J = 7.6 Hz, 1H), 4.15 (d, J = 7.2 Hz, 1H), 3.74 (s, 3H), 3.65 (d, J = 12.0 Hz, 1H), 3.46 (s, 3H), 3.34 (s, 2H), 2.93−2.27 (m, 1H); $^{13}$C[$^1$H] NMR (100 MHz, CDCl$_3$) $\delta$ ppm 196.2, 171.8, 170.6, 142.3, 136.9, 136.7, 134.8, 133.9, 131.7, 131.5, 131.1, 130.8, 130.3, 129.7, 128.5, 128.3, 128.2, 126.3, 122.8, 54.1, 52.9, 52.6, 35.7, 35.2, 33.8, 20.5, 14.8; HRMS (ESI/Q-TOF) m/z [M+H]$^+$ calcd for C$_{27}$H$_{25}$ClI$_2$O$_5$ 591.0430, found 591.0430 (0 ppm).

**Characterization Data of 3a, 4a−4c, 4i, 4j and Int-C**

**N-allyl-N-(3-(2,3-diiodo-1H-inden-1-yl)phenyl)prop-2-yn-1-yl)-4-methylbenzenesulfonamide (3a):** yellow solid; 88.4 mg; 64% yield; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ ppm 7.76 (d, J = 8.4 Hz, 2H), 7.34−7.30 (m, 2H), 7.22−7.21 (m, 1H), 7.18−7.15 (m, 3H), 7.13−7.09 (m, 2H), 7.01 (d, J = 7.2 Hz, 1H), 6.44 (d, J = 7.8 Hz, 1H), 5.82−5.76 (m, 1H), 5.30−5.21 (m, 2H), 4.93 (s, 1H), 4.43 (d, J = 5.4 Hz, 2H), 3.97−3.94 (m, 2H), 2.23 (m, 3H); $^{13}$C[$^1$H] NMR (150 MHz, CDCl$_3$) $\delta$ ppm 147.2, 145.0, 143.5, 139.7, 136.2, 132.7, 132.1, 129.5, 129.3, 127.8, 127.6, 127.2, 126.7, 126.6, 123.3, 123.1, 122.9, 119.9, 116.4, 110.1, 87.1, 84.0, 62.2, 49.3, 36.8, 21.5; HRMS (ESI/Q-Orbitrap) m/z [M+H]$^+$ calcd for C$_{28}$H$_{24}$I$_2$NO$_3$S 691.9612, found 691.9607 (0.7 ppm).
(1-methylbenzo[ff]isoquinolin-5-yl)(phenyl)methanone (4a): white solid; 95% yield; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ ppm 9.25 (s, 1H), 8.95 (d, $J = 8.4$ Hz, 1H), 8.62 (s, 1H), 7.94–7.91 (m, 3H), 7.88 (s, 1H), 7.79–7.72 (m, 2H), 7.46 (t, $J = 7.8$ Hz, 2H), 3.10 (s, 3H); $^{13}$C[$^1$H] NMR (150 MHz, CDCl$_3$) $\delta$ ppm 196.9, 148.9, 148.4, 137.7, 134.9, 134.7, 133.7, 132.7, 130.7, 130.3, 130.2, 129.9, 128.7, 128.6, 128.3, 128.0, 127.9, 125.3, 23.8; HRMS (ESI/Q-Orbitrap) $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{16}$NO 298.1226, found 298.1226 (0 ppm).

(1-methylbenzo[ff]isoquinolin-5-yl)(p-tolyl)methanone (4b): yellow solid; 88% yield; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 9.24 (s, 1H), 8.93 (d, $J = 8.0$ Hz, 1H), 8.60 (s, 1H), 7.92–7.69 (m, 6H), 7.25 (d, $J = 7.6$ Hz, 2H), 3.08 (s, 3H), 2.41 (s, 3H); $^{13}$C[$^1$H] NMR (100 MHz, CDCl$_3$) $\delta$ ppm 196.5, 148.7, 148.3, 144.7, 135.1, 134.6, 132.6, 130.5, 130.4, 129.8, 129.8, 129.3, 128.6, 128.3, 127.8, 127.8, 125.2, 23.8, 21.6; HRMS (ESI/Q-Orbitrap) $m/z$ [M+H]$^+$ calcd for C$_{22}$H$_{18}$NO 312.1383, found 312.1381 (0.6 ppm).

(4-ethylphenyl)(1-methylbenzo[ff]isoquinolin-5-yl)methanone (4c): yellow solid; 90% yield; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 9.25 (s, 1H), 8.89 (d, $J = 8.4$ Hz, 1H), 8.58 (s, 1H), 7.90–7.82 (m, 4H), 7.74–7.66 (m, 2H), 7.26 (d, $J = 8.0$ Hz, 2H), 3.05 (s, 3H), 2.69 (q, $J = 7.6$ Hz, 2H), 1.24 (t, $J = 7.6$ Hz, 3H); $^{13}$C[$^1$H] NMR (100 MHz, CDCl$_3$) $\delta$ ppm 196.4, 150.7, 148.7, 148.3, 135.2, 135.0, 134.5, 132.6, 130.4, 129.8, 129.7, 128.5, 128.2, 128.0, 127.8, 127.7, 125.2, 28.8, 23.7, 15.0; HRMS (ESI/Q-Orbitrap) $m/z$ [M+H]$^+$ calcd for C$_{23}$H$_{20}$NO 326.1539, found 326.1538 (0.3 ppm).

(4-chlorophenyl)(1-methylbenzo[ff]isoquinolin-5-yl)methanone (4i): yellow solid; 99% yield; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 9.22 (s, 1H), 8.95 (d, $J = 8.0$ Hz, 1H), 8.62 (s, 1H), 7.94 (dd, $J = 7.6$ Hz 1.6 Hz, 1H), 7.86 (t, $J = 8.0$ Hz, 3H), 7.81–7.73 (m, 2H), 7.44 (dd, $J = 6.8$ Hz 2.0 Hz, 2H), 3.10 (s, 3H); $^{13}$C[$^1$H] NMR (100 MHz, CDCl$_3$) $\delta$ ppm 195.6, 149.0, 148.2, 140.2, 136.0, 134.7, 134.3, 132.5, 131.6, 130.8, 130.4, 130.0, 129.0, 128.7, 128.4, 128.2, 127.9, 125.0, 23.8; HRMS (ESI/Q-Orbitrap) $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{13}$ClNO.
332.0837, found 332.0836 (0.3 ppm).

(4-bromophenyl)(1-methylbenzo[fi]soquinolin-5-yl)methanone (4j): yellow solid; 97% yield; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 9.22 (s, 1H), 8.93 (d, $J = 8.0$ Hz, 1H), 8.61 (s, 1H), 7.93 (d, $J = 7.6$ Hz, 1H), 7.86 (s, 1H), 7.80–7.71 (m, 4H), 7.60–7.58 (m, 2H), 3.09 (s, 3H); $^{13}$C($^1$H) NMR (100 MHz, CDCl$_3$) $\delta$ ppm 195.7, 149.0, 148.2, 136.4, 134.7, 134.2, 132.5, 131.9, 131.6, 130.7, 130.4, 129.9, 129.0, 128.7, 128.4, 128.2, 127.8, 125.0, 23.8; HRMS (ESI/Q-Orbitrap) $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{13}$BrNO 376.0332, found 376.0329 (0.8 ppm).

$N$-allyl-4-methyl-$N$-(3-(2-(3-oxo-3-phenylprop-1-en-1-yl)phenyl)prop-2-yn-1-yl)benzenesulfonamide (Int-C): light yellow solid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ ppm 8.02–8.00 (m, 2H), 7.88 (d, $J = 15.6$ Hz, 1H), 7.74–7.71 (m, 3H), 7.62–7.58 (m, 1H), 7.53–7.47 (m, 3H), 7.36–7.27 (m, 2H), 7.11 (d, $J = 8.0$ Hz, 3H), 5.58–5.75 (m, 1H), 5.38 (d, $J = 16.8$ Hz, 1H), 5.29–5.26 (m, 1H), 4.36 (s, 2H), 3.92 (d, $J = 6.4$ Hz, 2H), 2.18 (s, 3H); $^{13}$C($^1$H) NMR (100 MHz, CDCl$_3$) $\delta$ ppm 190.1, 143.4, 141.9, 137.9, 135.9, 135.5, 132.9, 132.9, 131.8, 129.6, 129.4, 128.6, 128.4, 127.6, 125.7, 123.5, 123.4, 120.2, 88.0, 83.2, 49.4, 36.7, 21.2; HRMS (ESI/Q-Orbitrap) $m/z$ [M+H]$^+$ calcd for C$_{26}$H$_{26}$NO$_3$S 456.1628, found 456.1628 (0 ppm).
$^1$H NMR, $^{13}$C NMR, and $^{19}$F NMR Spectra

$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^{1}H$ NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}C(H)$ NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
\[ \text{H NMR spectrum was recorded on 600 MHz in CDCl}_3. \]

\[ \text{C[H] NMR spectrum was recorded on 150 MHz in CDCl}_3. \]
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 

S24
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$.
$^{1}$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$.
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^{19}$F NMR spectrum was recorded on 376 MHz in CDCl$_3$. 
\( ^1H \) NMR spectrum was recorded on 600 MHz in CDCl\(_3\).

\( ^{13}C\{H\} \) NMR spectrum was recorded on 150 MHz in CDCl\(_3\).
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 

S32
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
$^{1}H$ NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}C$[H] NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
In

$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$.
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$.
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C($^1$H) NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^{1}$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$.
**1r**

$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C($^1$H) NMR spectrum was recorded on 100 MHz in CDCl$_3$.
$^{1}$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

540
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^{1}$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 

544
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 

S 45
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.
$^{1}H$ NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}C\{^1H\}$ NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
\[ ^1H \text{NMR spectrum was recorded on 400 MHz in CDCl}_3. \]

\[ ^{13}C\{H\} \text{NMR spectrum was recorded on 100 MHz in CDCl}_3. \]
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
\(^1\)H NMR spectrum was recorded on 400 MHz in CDCl\(_3\).

\(^{13}\)C{\(^1\)H} NMR spectrum was recorded on 100 MHz in CDCl\(_3\).
$^{1}H$ NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}C{H}$ NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$.
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$.

$^{19}$F NMR spectrum was recorded on 376 MHz in CDCl$_3$.
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 

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2i
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^{1}H$ NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}C(\text{H})$ NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

[Diagram of NMR spectra]
$2\text{n}$

$^1\text{H NMR spectrum was recorded on 400 MHz in CDCl}_3$. 

$^{13}\text{C\{H\} NMR spectrum was recorded on 100 MHz in CDCl}_3$. 

$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C($^1$H) NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^{1}H$ NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C($H$) NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

567
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$.
$^{1}$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$.
$^1$H NMR spectrum was recorded on 600 MHz in CDCl$_3$. (This by-product could not be isolated from a complex thoroughly)

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. (This by-product could not be isolated from a complex thoroughly)
$^{1}$H NMR spectrum was recorded on 600 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 150 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
\[ 4i \]

\(^1\)H NMR spectrum was recorded on 400 MHz in CDCl\(_3\).

\(^{13}\)C\{H\} NMR spectrum was recorded on 100 MHz in CDCl\(_3\).
$^1$H NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}$C{H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 
$^{1}H$ NMR spectrum was recorded on 400 MHz in CDCl$_3$.

$^{13}C${H} NMR spectrum was recorded on 100 MHz in CDCl$_3$. 

Int-C