Supporting Information for:
Controllable synthesis of 3-iodo-2H-quinolizin-2-ones and 1,3-diiodo-2H-quinolizin-2-ones via electrophilic cyclization of azacyclic yrones

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1. General information

NMR spectra were recorded with tetramethylsilane as the internal standard. NMR spectra were recorded on a BrukerAvanceII400M type (\textsuperscript{1}H NMR, 400 MHz; \textsuperscript{13}C NMR, 100 MHz) spectrometer. High resolution mass spectra (HRMS) were recorded on a Q-TOF mass spectrometry (Micromass, Wythenshawe, UK) equipped with Z-spray ionization source. Infrared spectra (IR) was measured using a Nicolet NEXUS FT-IR spectrophotometer. Melting points were measured on a RY-I apparatus and are reported uncorrected. Substrates \textbf{1a-1b}, \textbf{1e-1g} and \textbf{1i-1w} were prepared according to the previous report.\textsuperscript{1} Substrates \textbf{1c-1d} and \textbf{1h} were synthesized by a similar procedure.

2. Optimization of reaction conditions

Table S1 Optimization of reaction conditions

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<th>entry</th>
<th>electrophile (x eq)</th>
<th>additive</th>
<th>solvent</th>
<th>yield (%)</th>
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$^a$ Reaction conditions: 1a (0.20 mmol, 1.0 equiv), electrophile, additive (2.5 eq), solvent (2.0 mL), rt, 0.5 h. $^b$ Isolated yields. $^c$ 1 h. $^d$ 2 h.
3. General experimental procedures

3.1 General procedure for the synthesis of products 2

A 10 mL reaction tube was successively charged with 0.2 mmol azacyclic ynones 1, 0.5 mmol I$_2$, 0.5 mmol NaHCO$_3$ and 2.0 mL 1,4-dioxane. The tube was sealed and the reaction mixture was stirred at room temperature for 0.5 h. After completion of this reaction, the resulting mixture was quenched with saturated solution of NaS$_2$O$_3$ (15 ml) and then extracted with dichloromethane (15 mL×3). Then the combined organic layers were washed with brine, dried over anhydrous Na$_2$SO$_4$, filtered and concentrated in-vacuo. The crude reaction mixture was purified by column chromatography on silica gel (dichloromethane/methanol) to give products 2.

3.2 General procedure for the synthesis of products 3

A 10 mL reaction tube was successively charged with 0.20 mmol azacyclic ynones 1, 0.5 mmol NIS and 2.0 mL DCE. The tube was sealed and the reaction mixture was stirred at room temperature for 2 h. After completion of this reaction, the resulting mixture was quenched with saturated solution of NaHCO$_3$ (15 ml) and then extracted with dichloromethane (15 mL×3). Then the combined organic layers were washed with brine,
dried over anhydrous Na$_2$SO$_4$, filtered and concentrated in-vacuo. The crude reaction mixture was purified by column chromatography on silica gel (dichloromethane/methanol) to give products 3.

3.3 General procedure for the synthesis of product 4

![Chemical structure of 4](image)

The 3-iodo-4-phenyl-2H-quinolizin-2-one 2a (0.3 mmol), phenylboronic acid (0.6 mmol), palladium acetate (0.03 mmol), triphenylphosphine (0.06 mmol) and potassium carbonate (0.6 mmol) were dissolved in dioxane/water 4:1 (3 ml). The solution was degassed and flushed with nitrogen and subjected to stirring at 60°C for 15 h. Then the reaction mixture was diluted with ethyl acetate (50 ml) and washed with water (50 ml) and brine (50 ml). Organic layer was dried under Na$_2$SO$_4$ and concentrated in vacuo. Purification was performed by column chromatography (dichloromethane/methanol = 25:1) affording 4 (74.6 mg, 84% yield).

3.4 General procedure for the synthesis of product 5

![Chemical structure of 5](image)

The 1,3-diiodo-4-phenyl-2H-quinolizin-2-one 3a (0.3 mmol), phenylboronic acid (1.2 mmol), palladium acetate (0.03 mmol), triphenylphosphine (0.06 mmol) and potassium
carbonate (1.2 mmol) were dissolved in dioxane/water 4:1 (4 ml). The solution was degassed and flushed with nitrogen and subjected to stirring at 80°C for 20 h. Then the reaction mixture was diluted with ethyl acetate (50 ml) and washed with water (50 ml) and brine (50 ml). Organic layer was dried under Na₂SO₄ and concentrated in vacuo. Purification was performed by column chromatography (dichloromethane/methanol =100:1) affording 5 (80.7 mg, 72% yield).

3.5 General procedure for the synthesis of product 6

A 10 mL reaction tube was successively charged with 0.20 mmol 2a, 0.3 mmol NBS and 2.0 mL DCE. The tube was sealed and the reaction mixture was stirred at room temperature for 2 h. After completion of this reaction, the resulting mixture was quenched with saturated solution of NaHCO₃ (15 ml) and then extracted with dichloromethane (15 mL×3). Then the combined organic layers were washed with brine, dried over anhydrous Na₂SO₄, filtered and concentrated in-vacuo. The crude reaction mixture was purified by column chromatography on silica gel (dichloromethane/methanol) to give product 6.
4. Characterization data

4-(4-Ethylphenyl)-1-(pyridin-2-yl)but-3-yn-2-one (1c):

Purified by column chromatography (petroleum ether/ethyl acetate = 10:1); yellow solid (544.6 mg, 71% yield for 3mmol 2-methylpyridine as material; mp 52.8–54.0 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 14.96 (s, 1H, enol), 8.61 (d, $J$ = 4.2 Hz, 1H, keto), 8.27 (d, $J$ = 4.5 Hz, 1H, enol), 7.69 (td, $J$ = 7.7, 1.8 Hz, 1H, keto), 7.62 (td, $J$ = 7.9, 1.7 Hz, 1H, enol), 7.46 (d, $J$ = 8.2 Hz, 2H, enol), 7.39 (d, $J$ = 8.2 Hz, 2H, keto), 7.32 (d, $J$ = 7.8 Hz, 1H, keto), 7.22 (dd, $J$ = 6.8, 5.0 Hz, 1H, keto), 7.18 (d, $J$ = 8.1 Hz, 3H, enol), 7.03–6.93 (m, 3H, keto+enol), 5.87 (s, 1H, enol), 4.16 (s, 2H, keto), 2.66 (q, $J$ = 7.6 Hz, 4H, keto+enol), 1.23 (td, $J$ = 7.6, 5.3 Hz, 6H, keto+enol), (enol:keto = 76:24); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 184.0, 157.7, 154.0, 149.7, 149.7, 147.9, 145.6, 144.1, 137.5, 136.7, 133.3, 132.0, 128.2, 128.0, 124.5, 122.3, 121.4, 119.2, 119.1, 116.8, 103.6, 100.0, 93.7, 90.1, 87.8, 86.0, 77.6, 77.2, 76.8, 54.4, 29.0, 28.9, 15.3, 15.1; IR (KBr, cm$^{-1}$): 3434, 3076, 2966, 2877, 2197, 2877, 2877, 2193, 1607, 1542, 1460; HRMS (ESI-TOF) calcd for C$_{17}$H$_{16}$NO$^+$ ([M+H]$^+$): 250.1226, found: 250.1227.

4-(4-Tert-butylphenyl)-1-(pyridin-2-yl)but-3-yn-2-one (1d):

Purified by column chromatography (petroleum ether/ethyl acetate = 10:1); yellow solid (657.3 mg, 79% yield for 3mmol 2-methylpyridine as material); mp 89.2–90.0 °C; $^1$H
NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 14.95 (s, 1H, enol), 8.61 (d, \(J = 4.7\) Hz, 1H, keto), 8.28–8.24 (m, 1H, enol), 7.69 (td, \(J = 7.7, 1.7\) Hz, 1H, keto), 7.64–7.58 (m, 1H, enol), 7.50–7.46 (m, 4H, enol), 7.41–7.30 (m, 5H, keto+enol), 7.22 (dd, \(J = 7.4, 5.1\) Hz, 1H, keto), 7.03–6.96 (m, 4H, keto+enol), 5.87 (s, 1H, enol), 4.16 (s, 2H, keto), 1.31 (d, \(J = 6.1\) Hz, 18H, keto+enol), \((\text{enol}:\text{keto} = 80:20)\); \(^{13}\)C NMR (75 MHz, CDCl\textsubscript{3}) \(\delta\) 184.0, 157.7, 154.7, 154.0, 152.4, 149.7, 149.7, 144.1, 137.5, 136.7, 133.1, 131.8, 125.7, 125.4, 124.5, 122.3, 121.4, 119.1, 119.0, 116.7, 103.7, 93.7, 90.0, 87.8, 85.9, 54.4, 35.1, 34.9, 31.2, 31.0; IR (KBr, cm\textsuperscript{-1}): 3431, 3082, 3046, 2961, 2866, 2194, 1607, 1545, 1509, 1464; HRMS (ESI-TOF) calcd for C\textsubscript{19}H\textsubscript{20}NO\textsuperscript{+} (M+H)\textsuperscript{+} 278.1539, found: 278.1540.

4-(4-Chlorophenyl)-1-(pyridin-2-yl)but-3-yn-2-one (1h):

![Chemical structure](image)

Purified by column chromatography (petroleum ether/ethyl acetate = 10:1); yellow solid (475.6 mg, 62% yield for 3mmol 2-methylpyridine as material); mp 85.6–87.4 °C; \(^1\)H NMR (300 MHz, CDCl\textsubscript{3}) \(\delta\) 14.97 (s, 1H, enol), 8.63 (s, 1H, keto), 8.29 (s, 1H, enol), 7.78–7.58 (m, 2H, keto+enol), 7.54–7.22 (m, 10H, keto+enol), 7.10–6.97 (m, 2H, enol), 5.89 (d, \(J = 8.1\) Hz, 1H, enol), 4.20 (s, 2H, keto), \((\text{enol}:\text{keto} = 86:14)\); \(^{13}\)C NMR (75 MHz, CDCl\textsubscript{3}) \(\delta\) 157.5, 153.7, 149.6, 149.3, 144.0, 137.5, 136.8, 135.1, 134.3, 133.1, 129.1, 128.8, 124.6, 122.4, 121.5, 120.6, 119.2, 118.2, 103.9, 91.4, 88.4, 87.4, 54.2; IR (KBr, cm\textsuperscript{-1}): 3401, 2961, 1617, 1595, 1551, 1490, 1474; HRMS (ESI-TOF) calcd for C\textsubscript{15}H\textsubscript{11}ClNO\textsuperscript{+} ([M+H]\textsuperscript{+}): 256.0524, found: 256.0525.

3-Iodo-4-phenyl-2\(H\)-quinolin-2-one (2a):
Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (60.3 mg, 87% yield); mp 257.9–259.6 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.68–7.57 (m, 3H), 7.36 (d, \(J = 7.5\) Hz, 1H), 7.30 (dd, \(J = 7.9, 1.4\) Hz, 2H), 7.21 (d, \(J = 9.0\) Hz, 1H), 7.08 (dd, \(J = 8.8, 6.4\) Hz, 1H), 6.69 (s, 1H), 6.39–6.32 (m, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.8, 146.9, 144.3, 136.9, 130.6, 130.4, 130.2, 129.1, 128.5, 124.4, 112.3, 109.3, 106.9; IR (KBr, cm\(^{-1}\)): 3439, 3090, 2961, 1643, 1592, 1535, 1503; HRMS (ESI-TOF) calcd for C\(_{15}\)H\(_{11}\)INO\(^+\) ([M+H\(^+\)]: 347.9884, found: 347.9884.

3-Iodo-4-(p-tolyl)-2\(H\)-quinolizin-2-one (2b):

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (68.1 mg, 94% yield); mp 200.0–201.7 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.44 (d, \(J = 7.9\) Hz, 2H), 7.40–7.35 (m, 1H), 7.18 (dd, \(J = 8.3, 6.6\) Hz, 3H), 7.10–7.03 (m, 1H), 6.67 (s, 1H), 6.38–6.32 (m, 1H), 2.50 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.9, 147.1, 144.3, 140.6, 134.0, 130.8, 130.7, 129.0, 128.6, 124.4, 112.3, 109.3, 106.8, 21.6; IR (KBr, cm\(^{-1}\)): 3439, 3132, 3050, 2920, 1645, 1589, 1526, 1482, 1407; HRMS (ESI-TOF) calcd for C\(_{16}\)H\(_{13}\)INO\(^+\) ([M+H\(^+\)]: 362.0036, found: 362.0037.

4-(4-Ethylphenyl)-3-iodo-2\(H\)-quinolizin-2-one (2c):
Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (70.7 mg, 94% yield); mp 164.1–165.8 °C; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.46 (d, \(J = 8.1\) Hz, 2H), 7.40 (d, \(J = 7.5\) Hz, 1H), 7.20 (d, \(J = 8.2\) Hz, 3H), 7.10–7.04 (m, 1H), 6.68 (s, 1H), 6.38–6.33 (m, 1H), 2.80 (q, \(J = 7.6\) Hz, 2H), 1.34 (t, \(J = 7.6\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.8, 147.2, 146.7, 144.3, 134.2, 130.7, 129.6, 129.0, 128.7, 124.4, 112.4, 109.2, 106.8, 28.8, 15.2; IR (KBr, cm\(^{-1}\)): 3431, 3128, 3048, 2962, 2926, 2869, 1643, 1591, 1523, 1408; HRMS (ESI-TOF) calcd for C\(_{17}\)H\(_{15}\)INO\(^+\) ([M+H\(^+\))]: 376.0193, found: 376.0194.

4-(4-(Tert-butyl)phenyl)-3-iodo-2\(H\)-quinolizin-2-one (2d):

\[
\begin{array}{c}
\text{N} \\
\text{I} \\
\text{Bu}
\end{array}
\]

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (68.8 mg, 85% yield); mp 285.6–287.4 °C; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.63 (d, \(J = 8.3\) Hz, 2H), 7.39 (d, \(J = 7.5\) Hz, 1H), 7.20 (t, \(J = 8.1\) Hz, 3H), 7.07 (dd, \(J = 8.7, 6.7\) Hz, 1H), 6.68 (s, 1H), 6.40–6.32 (m, 1H), 1.42 (s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.9, 153.7, 147.2, 144.3, 134.0, 130.8, 128.7, 128.5, 127.0, 124.4, 112.2, 109.3, 106.8, 35.0, 31.3; IR (KBr, cm\(^{-1}\)): 3431, 2957, 2865, 1644, 1599, 1527, 1478, 1454, 1401; HRMS (ESI-TOF) calcd for C\(_{19}\)H\(_{19}\)INO\(^+\) ([M+H\(^+\))]: 404.0506, found: 404.0509.

3-Iodo-4-(4-methoxyphenyl)-2\(H\)-quinolizin-2-one (2e):

\[
\begin{array}{c}
\text{N} \\
\text{I} \\
\text{MeO}
\end{array}
\]

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (72.4 mg, 96% yield); mp 231.6–233.0 °C; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.46–7.41 (m, 1H), 7.23–7.11 (m, 5H), 7.10–7.04 (m, 1H), 6.66 (s, 1H), 6.39–6.34 (m, 1H),
3.92 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 171.9, 160.8, 146.9, 144.3, 130.7, 130.6, 129.1, 128.6, 124.3, 115.5, 112.3, 109.9, 106.8, 55.5; IR (KBr, cm$^{-1}$): 3436, 3114, 3084, 2959, 2836, 1642, 1596, 1522, 1482, 1407; HRMS (ESI-TOF) calcd for C$_{16}$H$_{13}$INO$_2^+$ ([M+H]$^+$): 377.9986, found: 377.9993.

3-Iodo-4-(m-tolyl)-2$H$-quinolizin-2-one (2f):

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (62.1 mg, 86% yield); mp 101.7–103.2 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.52 (t, $J$ = 7.6 Hz, 1H), 7.39 (t, $J$ = 7.4 Hz, 2H), 7.21 (d, $J$ = 9.0 Hz, 1H), 7.12–7.04 (m, 3H), 6.69 (s, 1H), 6.40–6.34 (m, 1H), 2.46 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 171.8, 147.2, 144.3, 140.2, 136.8, 131.1, 130.8, 130.0, 129.4, 128.8, 126.1, 124.4, 112.6, 108.7, 106.8, 21.5; IR (KBr, cm$^{-1}$): 3426, 3054, 2964, 2216, 1646, 1590, 1518, 1466; HRMS (ESI-TOF) calcd for C$_{16}$H$_{13}$INO$_2^+$ ([M+H]$^+$): 362.0036, found: 362.0039.

3-Iodo-4-(3-methoxyphenyl)-2$H$-quinolizin-2-one (2g):

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (70.4 mg, 93% yield); mp 127.3–129.0 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.55 (t, $J$ = 8.0 Hz, 1H), 7.39 (d, $J$ = 7.5 Hz, 1H), 7.20 (d, $J$ = 9.0 Hz, 1H), 7.15–7.04 (m, 2H), 6.87 (d, $J$ = 7.5 Hz, 1H), 6.82 (s, 1H), 6.68 (s, 1H), 6.37 (t, $J$ = 6.9 Hz, 1H), 3.87 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 171.8, 160.8, 146.7, 144.3, 137.9, 131.4, 130.7, 128.7, 124.4, 121.0, 116.0, 114.5, 112.4, 108.7, 106.8, 55.6; IR (KBr, cm$^{-1}$): 3421, 3136, 3052, 2964, 2846, 2217, 1647, 1590, 1518, 1466; HRMS (ESI-TOF) calcd for C$_{16}$H$_{13}$INO$_2^+$ ([M+H]$^+$): 377.9986, found: 377.9984.

4-(4-Chlorophenyl)-3-iodo-2$H$-quinolizin-2-one (2h):
Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid
(56.0 mg, 73% yield); mp 227.1 – 229.0 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.64 (d, \(J = 8.4\) Hz, 2H), 7.34 (d, \(J = 7.5\) Hz, 1H), 7.27 (d, \(J = 8.3\) Hz, 2H), 7.22 (d, \(J = 9.1\) Hz, 1H), 7.13 – 7.05 (m, 1H), 6.68 (s, 1H), 6.40 (t, \(J = 6.9\) Hz, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.6, 145.7, 144.3, 136.7, 135.2, 130.7, 130.6, 128.7, 124.5, 112.6, 109.5, 107.0; IR (KBr, cm\(^{-1}\)): 3434, 3116, 3088, 3042, 1646, 1594, 1534, 1497; HRMS (ESI-TOF) calcld for C\(_{15}\)H\(_{10}\)IClNO\(^+\) ([M+H]\(^+\)): 381.9490, found: 381.9493.

4-(4-Fluorophenyl)-3-iodo-2\(\text{H}\)-quinolizin-2-one (2i):

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid
(67.2 mg, 92% yield); mp 273.0 – 274.5 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.43 – 7.28 (m, 5H), 7.20 (d, \(J = 9.1\) Hz, 1H), 7.07 (ddd, \(J = 9.1, 6.5, 0.8\) Hz, 1H), 6.67 (s, 1H), 6.40 – 6.33 (m, 1H); \(^13\)C NMR (75 MHz, DMSO) \(\delta\) (ppm): 170.8, 163.2 (d, \(J_{\text{C-F}} = 245.9\) Hz), 146.6, 144.6, 133.9 (d, \(J_{\text{C-F}} = 3.4\) Hz), 132.5 (d, \(J_{\text{C-F}} = 8.6\) Hz), 131.4, 129.8, 124.2, 117.5 (d, \(J_{\text{C-F}} = 21.8\) Hz), 113.1, 110.4, 106.0; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): -108.8; IR (KBr, cm\(^{-1}\)): 3435, 3117, 3044, 1645, 1594, 1521, 1483, 1405; HRMS (ESI-TOF) calcld for C\(_{15}\)H\(_{10}\)IFNO\(^+\) ([M+H]\(^+\)): 365.9786, found: 365.9786.

3-Iodo-4-(naphthalen-1-yl)-2\(\text{H}\)-quinolizin-2-one (2j):
Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (77.4 mg, 97% yield); mp 272.0–274.0 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 8.10 (d, $J$ = 8.3 Hz, 1H), 8.01 (d, $J$ = 8.2 Hz, 1H), 7.68 (dd, $J$ = 20.3, 12.2 Hz, 1H), 7.59 (t, $J$ = 7.4 Hz, 1H), 7.47 (t, $J$ = 7.5 Hz, 2H), 7.27 (dd, $J$ = 8.5, 6.3 Hz, 2H), 7.13 (d, $J$ = 7.4 Hz, 1H), 7.05 (dd, $J$ = 25.9, 17.1 Hz, 1H), 6.78 (s, 1H), 6.25 (dd, $J$ = 10.2, 3.7 Hz, 1H); $^{13}$C NMR (75 MHz, DMSO) δ (ppm): 170.9, 145.9, 144.8, 134.3, 134.1, 131.1, 131.0, 129.8, 129.8, 129.4, 129.0, 128.4, 127.5, 126.9, 124.5, 123.8, 113.4, 111.1, 106.3; IR (KBr, cm$^{-1}$): 3431, 3043, 2928, 1643, 1591, 1526, 1494, 1449, 1403; HRMS (ESI-TOF) calcd for C$_{19}$H$_{13}$INO$^+$ ([M+H$^+$]): 398.0036, found: 398.0039.

3-Iodo-4-(thiophen-2-yl)-2H-quinolizin-2-one (2k):

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (45.4 mg, 64% yield); mp 278.0–280.0 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.68 (dd, $J$ = 5.1, 0.9 Hz, 1H), 7.53 (d, $J$ = 7.5 Hz, 1H), 7.28 (dd, $J$ = 5.1, 3.6 Hz, 1H), 7.18 (t, $J$ = 5.1 Hz, 2H), 7.12–7.06 (m, 1H), 6.67 (s, 1H), 6.45–6.39 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 171.5, 144.6, 140.3, 136.7, 130.8, 130.4, 129.7, 129.0, 128.4, 124.2, 113.2, 112.7, 107.2; IR (KBr, cm$^{-1}$): 3437, 3126, 3068, 2925, 1640, 1591, 1530, 1481, 1421; HRMS (ESI-TOF) calcd for C$_{13}$H$_9$INO$^+$ ([M+H$^+$]): 353.9444, found: 353.9443.

4-Butyl-3-iodo-2H-quinolizin-2-one (2m):

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid
(29.2 mg, 45% yield); mp 111.6–112.9 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.89 (d, \(J = 7.5\) Hz, 1H), 7.19 (d, \(J = 8.8\) Hz, 1H), 7.11 (dd, \(J = 8.8, 6.5\) Hz, 1H), 6.70–6.62 (m, 1H), 6.59 (s, 1H), 3.48–3.38 (m, 2H), 1.73–1.53 (m, 4H), 1.04 (t, \(J = 7.2\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.7, 146.3, 144.2, 128.2, 128.1, 125.2, 113.1, 109.9, 106.8, 37.9, 27.7, 22.6, 13.7; IR (KBr, cm\(^{-1}\)): 3359, 3157, 3095, 2925, 2863, 1648, 1587, 1515, 1474; HRMS (ESI-TOF) calcd for C\(_{13}\)H\(_{15}\)INO\(^+\) ([M+H]\(^+\)): 328.0193, found: 328.0198.

3-Iodo-4-phenethyl-2H-quinolizin-2-one (2n):

\[
\begin{align*}
\text{Purified by column chromatography (dichloromethane/methanol = 25:1); brown solid (56.6 mg, 67\% yield); mp 205.2–207.0 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.88 (d, \(J = 7.5\) Hz, 1H), 7.38–7.23 (m, 5H), 7.17 (d, \(J = 8.7\) Hz, 1H), 7.07 (dd, \(J = 8.8, 6.3\) Hz, 1H), 6.61–6.55 (m, 2H), 3.75–3.66 (m, 2H), 3.06–2.95 (m, 2H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.5, 145.2, 144.2, 139.0, 128.9, 128.3, 128.1, 127.0, 125.2, 113.3, 110.4, 106.9, 39.9, 31.8; IR (KBr, cm\(^{-1}\)): 3440, 3151, 2941, 2878, 1645, 1594, 1534, 1476; HRMS (ESI-TOF) calcd for C\(_{17}\)H\(_{15}\)INO\(^+\) ([M+H]\(^+\)): 376.0193, found 376.0190.
\end{align*}
\]

4-Cyclopentyl-3-iodo-2H-quinolizin-2-one (2o):

\[
\begin{align*}
\text{Purified by column chromatography (dichloromethane/methanol = 25:1); brown solid (38.7 mg, 57\% yield); mp 192.5–194.0 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.71 (d, \(J = 7.6\) Hz, 1H), 7.20 (d, \(J = 8.9\) Hz, 1H), 7.07 (dd, \(J = 8.8, 6.6\) Hz, 1H), 6.57 (s, 1H), 6.56–6.48 (m, 1H), 4.78–4.64 (m, 1H), 2.24–2.08 (m, 2H), 1.99 (ddd, \(J = 12.2, 10.2, 7.5\) Hz, 6H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.9, 147.5, 144.3, 129.2, 127.8, 125.7, 112.9, 111.5, 106.8, 50.4, 28.0, 27.0; IR (KBr, cm\(^{-1}\)): 3434, 2941, 2878, 1645, 1596, 1528, 1460, 1405; HRMS (ESI-TOF) calcd for C\(_{14}\)H\(_{15}\)INO\(^+\) ([M+H]\(^+\)): 340.0193, found 340.0191.
\end{align*}
\]
3-Iodo-7-methyl-4-phenyl-2H-quinolizin-2-one (2p):

![Chemical Structure]

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (57.6 mg, 80% yield); mp 242.3–244.2 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.69–7.54 (m, 3H), 7.29–7.26 (m, 2H), 7.14 (d, $J = 9.2$ Hz, 1H), 7.09 (s, 1H), 6.94 (d, $J = 9.1$ Hz, 1H), 6.66 (s, 1H), 2.01 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 171.7, 146.5, 143.2, 137.1, 131.9, 130.3, 130.1, 129.1, 127.5, 124.0, 122.1, 108.9, 106.7, 18.2; IR (KBr, cm$^{-1}$): 3432, 3057, 2857, 1599, 1532, 1506, 1447; HRMS (ESI-TOF) calcd for C$_{16}$H$_{13}$INO$^+$ ([M+H]$^+$): 362.0036, found: 362.0036.

8-Bromo-3-iodo-4-phenyl-2H-quinolizin-2-one (2q):

![Chemical Structure]

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (57.5 mg, 68% yield); mp 277.2–279.0 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.69–7.58 (m, 3H), 7.37 (d, $J = 2.1$ Hz, 1H), 7.30–7.26 (m, 2H), 7.20 (d, $J = 7.9$ Hz, 1H), 6.57 (s, 1H), 6.38 (dd, $J = 7.9$, 2.2 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 171.9, 146.9, 144.1, 136.5, 131.3, 130.6, 130.3, 129.1, 125.4, 123.4, 116.0, 109.2, 106.2; IR (KBr, cm$^{-1}$): 3430, 3123, 3051, 2922, 1680, 1626, 1596, 1514, 1442; HRMS (ESI-TOF) calcd for C$_{15}$H$_{10}$BrINO$^+$ ([M+H]$^+$): 425.8985, found: 425.8988.

3-Iodo-9-methyl-4-phenyl-2H-quinolizin-2-one (2r):
Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (61.5 mg, 85% yield); mp 212.2–214.1 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.72–7.55 (m, 3H), 7.28 (dd, \(J = 14.1, 5.9\) Hz, 3H), 6.96 (d, \(J = 6.6\) Hz, 1H), 6.80 (s, 1H), 6.29 (t, \(J = 7.1\) Hz, 1H), 2.37 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 172.0, 147.4, 144.7, 137.5, 130.8, 130.3, 130.1, 129.3, 129.1, 127.9, 111.8, 108.3, 104.4, 77.4, 77.1, 76.8, 19.6; IR (KBr, cm\(^{-1}\)): 3435, 3047, 1906, 1638, 1587, 1540, 1447; HRMS (ESI-TOF) calcd for C\(_{16}\)H\(_{13}\)INO\(^+\) ([M+H]\(^+\)): 362.0036, found: 362.0034.

**3-Iodo-4,9-diphenyl-2\(H\)-quinolizin-2-one (2s):**

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (76.4 mg, 90% yield); mp 224.5–225.8 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.70–7.59 (m, 3H), 7.50–7.41 (m, 4H), 7.38 (dd, \(J = 7.5, 1.7\) Hz, 2H), 7.36–7.31 (m, 2H), 6.99 (d, \(J = 6.6\) Hz, 1H), 6.74 (s, 1H), 6.44 (t, \(J = 7.1\) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.9, 147.3, 144.5, 137.4, 136.8, 136.6, 130.4, 130.2, 130.2, 129.1, 129.0, 128.8, 128.7, 112.0, 108.5, 106.9; IR (KBr, cm\(^{-1}\)): 3440, 3122, 3048, 2924, 1766, 1593, 1541, 1506, 1443; HRMS (ESI-TOF) calcd for C\(_{21}\)H\(_{15}\)INO\(^+\) ([M+H]\(^+\)): 424.0193, found: 424.0189.

**3-Iodo-4-phenyl-2\(H\)-pyrido[2,1-\(a\)]isoquinolin-2-one (2t):**
Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (49.7 mg, 63% yield); mp 281.9–282.8 °C; ¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.29 (d, J = 7.7 Hz, 1H), 7.72–7.58 (m, 5H), 7.56 (s, 1H), 7.53–7.48 (m, 1H), 7.33 (dd, J = 7.8, 1.4 Hz, 2H), 7.20 (d, J = 7.8 Hz, 1H), 6.55 (d, J = 7.9 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 173.0, 148.7, 143.0, 137.5, 131.4, 130.3, 129.5, 129.2, 129.2, 127.1, 127.0, 125.0, 124.2, 112.7, 105.3, 105.3; IR (KBr, cm⁻¹): 3441, 3023, 1648, 1589, 1481, 1452, 1433; HRMS (ESI-TOF) calcd for C₁₉H₁₃INO⁺ ([M+H]⁺): 398.0036, found: 398.0036.

7-Iodo-6-phenyl-8H-pyrido[1,2-a]pyrazin-8-one (2u):

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (44.9 mg, 65% yield); mp 247.9–259.6 °C; ¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.68 (s, 1H), 7.69–7.61 (m, 3H), 7.36–7.29 (m, 3H), 7.06 (d, J = 5.2 Hz, 1H), 6.84 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ (ppm): 172.8, 152.3, 147.1, 137.2, 135.3, 130.9, 130.2, 128.9, 127.9, 120.6, 111.2, 108.8; IR (KBr, cm⁻¹): 3439, 3132, 2922, 1592, 1495, 1404; HRMS (ESI-TOF) calcd for C₁₄H₁₀IN₂O⁺ ([M+H]⁺): 348.9832, found: 348.9831.

3-Iodo-1-methyl-4-phenyl-2H-quinolizin-2-one (2w):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (37.1 mg, 51% yield for I₂ as electrophile; 44.8 mg, 62% yield for NIS as electrophile); mp
247.2–248.4 °C; \( ^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) (ppm): 7.67–7.55 (m, 3H), 7.47 (d, \( J = 9.4 \) Hz, 1H), 7.40 (t, \( J = 12.1 \) Hz, 1H), 7.31–7.24 (m, 2H), 7.11 (ddd, \( J = 9.4, 6.4, 1.0 \) Hz, 1H), 6.32 (ddd, \( J = 7.7, 6.6, 1.4 \) Hz, 1H), 2.45–2.37 (m, 3H); \( ^{13}\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 171.0, 145.5, 141.0, 137.5, 131.3, 130.2, 130.1, 129.3, 127.8, 121.9, 114.0, 111.4, 107.2, 11.9; IR (KBr, cm\(^{-1}\))): 3434, 3143, 3046, 2915, 1640, 1559, 1503, 1449, 1413; HRMS (ESI-TOF) calcd for C\(_{16}\)H\(_{13}\)INO\(^+\) ([M+H\(^+\)]: 362.0036, found: 362.0038.

1,3-Diiodo-4-phenyl-2\(H\)-quinolizin-2-one (3a):

![Structure of 3a](image)

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (85.7 mg, 90% yield); mp 287.9–290.3 °C; \( ^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) (ppm): 8.00 (d, \( J = 9.3 \) Hz, 1H), 7.72–7.58 (m, 3H), 7.43 (d, \( J = 7.4 \) Hz, 1H), 7.32–7.28 (m, 2H), 7.23–7.15 (m, 1H), 6.44–6.38 (m, 1H); \( ^{13}\)C NMR (100 MHz, DMSO) \( \delta \) 168.4, 147.3, 143.4, 137.5, 133.1, 133.1, 130.6, 130.4 129.8, 128.3, 113.4, 103.3, 83.1; IR (KBr, cm\(^{-1}\))): 3439, 3084, 2925, 1639, 1573, 1477; HRMS (ESI-TOF) calcd for C\(_{15}\)H\(_{10}\)I\(_2\)NO\(^+\) ([M+H\(^+\)]: 473.8846, found: 473.8847.

1,3-Diiodo-4-(p-tolyl)-2\(H\)-quinolizin-2-one (3b):

![Structure of 3b](image)

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (93.5 mg, 96% yield); mp 229.0–231.0 °C; \( ^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \)(ppm): 7.99 (d, \( J = 9.3 \) Hz, 1H), 7.46 (dd, \( J = 10.4, 7.8 \) Hz, 3H), 7.24–7.13 (m, 3H), 6.45–6.37 (m, 1H), 2.50 (s, 3H); \( ^{13}\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) (ppm): 168.7, 146.7, 143.2, 140.8, 133.9, 132.2, 131.3, 130.9, 129.3, 129.0, 112.6, 102.9, 83.2, 21.6; IR (KBr, cm\(^{-1}\))): 3437, 3043, 2921,
1638, 1573, 1519, 1469; HRMS (ESI-TOF) calcd for C_{16}H_{12}I_{2}NO^{+} ([M+H]^{+}): 487.9003, found: 487.9000.

**4-(4-Ethylphenyl)-1,3-diiodo-2H-quinolizin-2-one (3c):**

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (95.9 mg, 96% yield); mp 238.9–240.4 °C; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.99 (d, \(J = 9.3\) Hz, 1H), 7.50–7.43 (m, 3H), 7.24–7.15 (m, 3H), 6.45–6.38 (m, 1H), 2.88–2.74 (m, 2H), 1.34 (t, \(J = 7.6\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 168.7, 146.9, 146.7, 143.2, 134.1, 132.2, 131.24, 129.7, 129.3, 129.0, 112.5, 102.9, 83.2, 28.8, 15.2; IR (KBr, cm\(^{-1}\)): 3444, 2963, 2926, 1638, 1572, 1517, 1469; HRMS (ESI-TOF) calcd for C_{17}H_{14}I_{2}NO^{+} ([M+H]^{+}): 501.9159, found: 501.9158.

**4-(4-(Tert-butyl)phenyl)-1,3-diiodo-2H-quinolizin-2-one (3d):**

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (97.3 mg, 92% yield); mp 301.8–303.6 °C; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.98 (t, \(J = 9.5\) Hz, 1H), 7.67–7.60 (m, 2H), 7.48 (d, \(J = 7.4\) Hz, 1H), 7.24–7.16 (m, 3H), 6.45–6.39 (m, 1H), 1.42 (s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 168.8, 153.9, 146.8, 143.2, 133.9, 132.3, 131.2, 129.3, 128.8, 127.1, 112.5, 102.9, 83.1, 35.1, 31.3; IR (KBr, cm\(^{-1}\)): 3437, 3134, 3057, 2959, 2867, 1933, 1641, 1573, 1517, 1469; HRMS (ESI-TOF) calcd for C_{19}H_{18}I_{2}NO^{+} ([M+H]^{+}): 529.9472, found: 529.9466.

**1,3-Diiodo-4-(4-methoxyphenyl)-2H-quinolizin-2-one (3e):**
Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (95.9 mg, 95% yield); mp 272.5–274.2 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.98 (d, $J = 9.4$ Hz, 1H), 7.52 (d, $J = 7.4$ Hz, 1H), 7.24–7.17 (m, 3H), 7.17–7.11 (m, 2H), 6.45–6.40 (m, 1H), 3.92 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 168.8, 160.9, 146.5, 143.2, 132.2, 131.2, 130.6, 129.3, 129.0, 115.6, 112.5, 103.5, 83.2, 55.5; IR (KBr, cm$^{-1}$): 3437, 3130, 2943, 2832, 1572, 1468; HRMS (ESI-TOF) calcd for C$_{16}$H$_{12}$I$_2$NO$_2^+$ ([M+H]$^+$): 503.8952, found: 503.8947.

1,3-Diiodo-4-(m-tolyl)-2H-quinoliniz-2-one (3f):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (93.6 mg, 96% yield); mp 220.8–222.2 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.99 (d, $J = 9.3$ Hz, 1H), 7.53 (t, $J = 8.0$ Hz, 1H), 7.45 (d, $J = 7.4$ Hz, 1H), 7.41 (d, $J = 7.8$ Hz, 1H), 7.20 (ddd, $J = 9.4$, 6.5, 1.0 Hz, 1H), 7.08 (d, $J = 7.1$ Hz, 2H), 6.44–6.38 (m, 1H), 2.46 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 168.7, 146.7, 143.2, 140.3, 136.8, 132.2, 131.3, 131.2, 130.1, 129.5, 129.3, 126.1, 112.5, 102.6, 83.2, 21.6; IR (KBr, cm$^{-1}$): 3437, 3130, 3045, 2918, 2860, 1638, 1572, 1475; HRMS (ESI-TOF) calcd for C$_{16}$H$_{12}$I$_2$NO$_2^+$ ([M+H]$^+$): 487.9003, found: 487.9009.

1,3-Diiodo-4-(3-methoxyphenyl)-2H-quinoliniz-2-one (3g):

- S 20 -
Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (95.2 mg, 95% yield); mp 235.5–236.8 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.99 (d, \(J = 9.4\) Hz, 1H), 7.56 (t, \(J = 8.0\) Hz, 1H), 7.47 (d, \(J = 7.4\) Hz, 1H), 7.25–7.18 (m, 1H), 7.12 (dd, \(J = 8.4, 2.4\) Hz, 1H), 6.85 (t, \(J = 7.4\) Hz, 1H), 6.81 (d, \(J = 2.0\) Hz, 1H), 6.46–6.40 (m, 1H), 3.87 (s, 3H); \(^{13}\)C NMR (75 MHz, DMSO) \(\delta\) (ppm): 168.3, 160.7, 147.1, 143.3, 138.6, 133.3, 133.1, 131.7, 128.2, 121.7, 116.2, 115.3, 113.4, 103.1, 83.0, 55.9; IR (KBr, cm\(^{-1}\)): 3439, 3140, 3056, 2928, 2842, 1638, 1575, 1467, 1429; HRMS (ESI-TOF) calcd for C\(_{16}\)H\(_{12}\)I\(_2\)NO\(_2\)\(^+\) ([M+H\(^+\)]\(^+\)): 503.8952, found: 503.8958.

**4-(4-Chlorophenyl)-1,3-diiodo-2H-quinolin-2-one (3h):**

\[
\begin{align*}
\text{Cl} & \quad \text{I} \\
\text{N} & \quad \text{I} \\
\text{C} & \quad \text{C} \\
& \quad \text{C}
\end{align*}
\]

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (78.4 mg, 77% yield); mp 237.5–239.4 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 8.00 (d, \(J = 9.4\) Hz, 1H), 7.67–7.60 (m, 2H), 7.41 (d, \(J = 7.4\) Hz, 1H), 7.30–7.24 (m, 2H), 7.21 (ddd, \(J = 9.3, 6.6, 0.9\) Hz, 1H), 6.48–6.41 (m, 1H); \(^{13}\)C NMR (75 MHz, DMSO) \(\delta\) (ppm): 168.3, 146.3, 143.4, 136.4, 135.5, 133.5, 133.2, 132.0, 130.6, 128.3, 113.5, 103.6, 83.2; IR (KBr, cm\(^{-1}\)): 3437, 3131, 3062, 1638, 1579, 1482; HRMS (ESI-TOF) calcd for C\(_{15}\)H\(_9\)Cl\(_2\)NO\(_2\)\(^+\) ([M+H\(^+\)]\(^+\)): 507.8457, found: 507.8456.

**4-(4-Fluorophenyl)-1,3-diiodo-2H-quinolin-2-one (3i):**

\[
\begin{align*}
\text{F} & \quad \text{I} \\
\text{N} & \quad \text{I} \\
\text{C} & \quad \text{C} \\
& \quad \text{C}
\end{align*}
\]

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (96.2 mg, 98% yield); mp 264.7–266.2 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 8.00 (d, \(J = 9.3\) Hz, 1H), 7.43 (d, \(J = 7.4\) Hz, 1H), 7.38–7.28 (m, 4H), 7.22 (ddd, \(J = 9.4, 6.6, 1.0\) Hz,
1H), 6.49–6.41 (m, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 168.7, 163.7 (d, \(J_{CF} = 250.8\) Hz), 145.5, 143.3, 132.9 (d, \(J_{CF} = 3.3\) Hz), 131.9, 131.5 (d, \(J_{CF} = 8.6\) Hz), 131.2, 129.5, 117.6 (d, \(J_{CF} = 21.9\) Hz), 112.8, 103.3, 83.5; IR (KBr, cm\(^{-1}\)): 3438, 3068, 3046, 2030, 1927, 1799, 1639, 1571, 1512, 1468; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): -108.4; HRMS (ESI-TOF) calcd for C\(_{15}\)H\(_9\)F\(_2\)NO\(^+\) ([M+H]\(^+\)): 491.8752, found: 491.8759.

1,3-Diiodo-4-(naphthalen-1-yl)-2H-quinolizin-2-one (3j):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (94.2 mg, 90% yield); mp 271.0–272.9 \(^\circ\)C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 8.11 (d, \(J = 8.3\) Hz, 1H), 8.04 (dd, \(J = 15.8, 8.8\) Hz, 2H), 7.70 (t, \(J = 7.7\) Hz, 1H), 7.60 (t, \(J = 7.5\) Hz, 1H), 7.47 (t, \(J = 6.6\) Hz, 2H), 7.26–7.14 (m, 3H), 6.31 (t, \(J = 6.8\) Hz, 1H); \(^{13}\)C NMR (100 MHz, DMSO) \(\delta\) 168.4, 145.7, 143.7, 134.4, 134.1, 133.1, 133.0, 131.1, 129.9, 129.4, 129.1, 128.6, 128.5, 127.5 126.9, 124.1, 113.8, 104.7, 83.6; IR (KBr, cm\(^{-1}\)): 3442, 3132, 3053, 1639, 1574,1474; HRMS (ESI-TOF) calcd for C\(_{19}\)H\(_{12}\)I\(_2\)NO\(^+\) ([M+H]\(^+\)): 523.9003, found: 523.9006.

1,3-Diiodo-4-(thiophen-2-yl)-2H-quinolizin-2-one (3k):

Purified by column chromatography (dichloromethane/methanol =100:1); yellow solid (64.0 mg, 67% yield); mp 258.0–260.0 \(^\circ\)C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 8.00 (d, \(J = 9.3\) Hz, 1H), 7.70 (d, \(J = 4.9\) Hz, 1H), 7.63 (d, \(J = 7.3\) Hz, 1H), 7.33–7.28 (m, 1H), 7.21 (dd, \(J = 13.2, 6.0\) Hz, 2H), 6.50 (t, \(J = 6.7\) Hz, 1H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 168.5, 143.5, 139.8, 136.7, 132.2, 131.6, 130.7, 129.9, 129.3, 128.5, 112.8, 106.5, 83.8; IR (KBr, cm\(^{-1}\)): 3439, 3138, 3073, 1638, 1574, 1530, 1469, 1425; HRMS (ESI-TOF) calcd for C\(_{13}\)H\(_8\)I\(_2\)NOS\(^+\) ([M+H]\(^+\)): 479.8411, found: 479.8414.
1,3-Diiodo-4-(pyridin-3-yl)-2H-quinolin-2-one (3l):

\[
\begin{align*}
 & \text{Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (35.1 mg, 37\% yield); mp 274.0–276.0 °C; } \\
 & \text{\(^1H\) NMR (400 MHz, CDCl}_3) \delta \text{(ppm): 8.88 (d, } J = 3.5 \text{ Hz, 1H), 8.60 (d, } J = 1.3 \text{ Hz, 1H), 8.02 (d, } J = 9.3 \text{ Hz, 1H), 7.71 (dt, } J = 7.8 \text{, 1.8 Hz, 1H), 7.62 (dd, } J = 7.7 \text{, 4.9 Hz, 1H), 7.39 (d, } J = 7.4 \text{ Hz, 1H), 7.24 (dd, } J = 8.9 \text{, 7.0 Hz, 1H), 6.54–6.41 \text{ (m, 1H); } \\
 & \text{\(^{13}C\) NMR (75 MHz, CDCl}_3/CD_3OD = 20:1) \delta \text{(ppm): 168.8, 151.4, 149.7, 143.5, 143.3, 137.7, 133.3, 131.7, 131.6, 129.7, 124.9, 113.6, 103.6, 83.6; IR (KBr, cm}^{-1}): 3452, 1638, 1569, 1475, 1408; HRMS (ESI-TOF) calcd for C_{14}H_9I_2N_2O^+ ([M+H]^+): 474.8799, found 474.8792.
\end{align*}
\]

4-Butyl-1,3-diiodo-2H-quinolin-2-one (3m):

\[
\begin{align*}
 & \text{Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (45.8 mg, 51\% yield); mp 175.6–177.2 °C; } \\
 & \text{\(^1H\) NMR (400 MHz, CDCl}_3) \delta \text{(ppm): 7.99 (t, } J = 10.2 \text{ Hz, 1H), 7.92 (t, } J = 9.1 \text{ Hz, 1H), 7.22 (dd, } J = 9.3 \text{, 6.5 Hz, 1H), 6.71–6.65 \text{ (m, 1H), 3.50–3.42 \text{ (m, 2H), 1.67 (dt, } J = 15.9 \text{, 8.0 Hz, 2H), 1.57 (dt, } J = 14.2 \text{, 7.1 Hz, 2H), 1.04 (t, } J = 7.2 \text{ Hz, 3H); } \\
 & \text{\(^{13}C\) NMR (100 MHz, CDCl}_3) \delta \text{(ppm): 168.6, 146.0, 143.0, 130.9, 130.0, 129.7, 113.5, 103.5, 83.0, 38.3, 27.8, 22.6, 13.8; IR (KBr, cm}^{-1}): 3433, 3135, 2953, 2861, 1640, 1577, 1466; HRMS (ESI-TOF) calcd for C_{13}H_{14}I_2NO^+ ([M+H]^+): 453.9159, found: 453.9161.
\end{align*}
\]

1,3-Diiodo-4-phenethyl-2H-quinolin-2-one (3n):
Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (72.2 mg, 72% yield); mp 222.8–224.4 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 8.01 (d, $J = 9.6$ Hz, 1H), 7.94 (d, $J = 7.5$ Hz, 1H), 7.38–7.26 (m, 5H), 7.21 (dd, $J = 9.3$, 6.5 Hz, 1H), 6.68–6.61 (m, 1H), 3.79–3.72 (m, 2H), 3.05–2.95 (m, 2H); $^{13}$C NMR (75 MHz, CDCl$_3$/CD$_3$OD = 20:1) δ (ppm): 168.8, 145.0, 143.1, 138.8, 131.2, 130.1, 129.5, 128.9, 128.2, 127.0, 113.8, 103.7, 83.2, 40.3, 31.8; IR (KBr, cm$^{-1}$): 3435, 1643, 1564, 1463; HRMS (ESI-TOF) caleld for C$_{17}$H$_{14}$I$_2$NO$^+$ ([M+H]$^+$): 501.9159, found 501.9151.

4-Cyclopentyl-1,3-diiodo-2H-quinolizin-2-one (3o):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (67.0 mg, 72% yield); mp 231.9–233.4 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 8.02 (t, $J = 9.4$ Hz, 1H), 7.74 (d, $J = 7.5$ Hz, 1H), 7.24–7.16 (m, 1H), 6.65–6.57 (m, 1H), 4.76–4.62 (m, 1H), 2.22–2.10 (m, 2H), 1.99 (ddd, $J = 14.7$, 13.8, 7.6 Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 168.8, 147.2, 143.0, 130.6, 130.5, 130.4, 111.7, 106.4, 83.0, 50.5, 28.3, 27.1; IR (KBr, cm$^{-1}$): 3440, 3182, 2947, 1636, 1573, 1460; HRMS (ESI-TOF) caleld for C$_{14}$H$_{14}$I$_2$NO$^+$ ([M+H]$^+$): 465.9159, found 465.9155.

1,3-Diiodo-7-methyl-4-phenyl-2H-quinolizin-2-one (3p):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid
(89.2 mg, 92% yield); mp 250.7–252.7 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.71–7.59 (m, 3H), 7.28 (d, $J$ = 6.9 Hz, 2H), 7.19 (s, 1H), 7.07 (d, $J$ = 9.5 Hz, 1H), 2.08 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 168.5, 146.0, 142.2, 137.1, 134.4, 130.5, 130.2, 129.2, 129.0, 128.9, 122.4, 102.6, 83.0, 17.9; IR (KBr, cm$^{-1}$): 3432, 3041, 2973, 2918, 1650, 1575, 1489, 1436, 1417; HRMS (ESI-TOF) calcd for C$_{16}$H$_{12}$I$_2$NO$^+$ ([M+H]$^+$): 487.9003, found: 487.9006.

8-Bromo-1,3-diiodo-4-phenyl-2$H$-quinolizin-2-one (3q):

![8-Bromo-1,3-diiodo-4-phenyl-2$H$-quinolizin-2-one (3q)](image)

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (80.8 mg, 73% yield); mp 250.0–252.0 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 8.20 (d, $J$ = 2.0 Hz, 1H), 7.70–7.57 (m, 3H), 7.31–7.27 (m, 3H), 6.44 (dd, $J$ = 7.8, 2.2 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 167.8, 145.5, 142.2, 135.4, 131.7, 129.8, 129.6, 129.4, 128.1, 125.7, 115.4, 101.6, 81.4; IR (KBr, cm$^{-1}$): 3434, 3067, 2923, 1709, 1626, 1577, 1522, 1503, 1462; HRMS (ESI-TOF) calcd for C$_{15}$H$_9$BrI$_2$NO$^+$ ([M+H]$^+$): 551.7951, found: 551.7947.

1,3-Diiodo-9-methyl-4-phenyl-2$H$-quinolizin-2-one (3r):

![1,3-Diiodo-9-methyl-4-phenyl-2$H$-quinolizin-2-one (3r)](image)

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (77.2 mg, 79% yield); mp 207.2–209.0 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ (ppm): 7.67–7.55 (m, 3H), 7.48 (d, $J$ = 7.4 Hz, 1H), 7.31–7.25 (m, 2H), 7.15 (d, $J$ = 6.8 Hz, 2H), 6.33 (t, $J$ = 7.1 Hz, 1H), 3.06 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ (ppm): 169.6, 147.4, 145.2, 137.8, 132.5, 131.6, 131.3, 130.4, 130.2, 129.2, 111.8, 99.6, 80.2, 82.3; IR (KBr,
cm$^{-1}$): 3436, 3065, 1634, 1492, 1450; HRMS (ESI-TOF) calcd for C$_{16}$H$_{12}$I$_2$NO$^+$ ([M+H]$^+$): 487.9003, found: 487.9009.

1,3-Diiodo-4,9-diphenyl-$2H$-quinolizin-2-one (3s):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (34.9 mg, 32% yield); mp 190.0–192.0 $^\circ$C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.70–7.58 (m, 3H), 7.51 (d, $J = 7.2$ Hz, 1H), 7.42 (t, $J = 7.2$ Hz, 3H), 7.36 (d, $J = 7.0$ Hz, 2H), 7.32 (d, $J = 7.3$ Hz, 2H), 7.23 (d, $J = 6.6$ Hz, 1H), 6.46 (t, $J = 7.0$ Hz, 1H); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 170.0, 147.1, 143.3, 139.7, 137.4, 133.2, 131.4, 130.6, 130.2, 129.8, 129.4, 128.6, 128.2, 111.4, 100.3, 82.7; IR (KBr, cm$^{-1}$): 3737, 3125, 3063, 2923, 1631, 1574, 1489, 1451; HRMS (ESI-TOF) calcd for C$_{21}$H$_{14}$I$_2$NO$^+$ ([M+H]$^+$): 549.9159, found: 549.9161.

1,3-Diiodo-4-phenyl-$2H$-pyrido[2,1-$a$]isoquinolin-2-one (3t):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (74.4 mg, 71% yield); mp 239.0–241.0 $^\circ$C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 9.44 (d, $J = 8.3$ Hz, 1H), 7.69–7.53 (m, 5H), 7.45 (d, $J = 7.5$ Hz, 1H), 7.34 (dd, $J = 7.2$, 1.9 Hz, 2H), 7.04 (d, $J = 7.6$ Hz, 1H), 6.54 (d, $J = 7.7$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.6, 148.5, 144.6, 137.0, 131.9, 131.3, 131.1, 130.6, 129.9, 129.6, 127.4, 126.6, 125.8, 125.3, 112.5, 95.6, 84.7; IR (KBr, cm$^{-1}$): 3747, 3440, 3124, 3042, 2924, 1718, 1647, 1577, 1496, 1459; HRMS (ESI-TOF) calcd for C$_{19}$H$_{12}$I$_2$NO$^+$ ([M+H]$^+$): 523.9003, found: 523.9005.

7,9-Diiodo-6-phenyl-$8H$-pyrido[1,2-$a$]pyrazin-8-one (3u):

- S 26 -
Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (51.1 mg, 54% yield); mp 258.6–259.9 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 9.35 (s, 1H), 7.70–7.61 (m, 3H), 7.43 (d, \(J = 5.2\) Hz, 1H), 7.30 (dd, \(J = 7.5, 1.6\) Hz, 2H), 7.07 (d, \(J = 5.1\) Hz, 1H); \(^{13}\)C NMR (100 MHz, DMSO) \(\delta\) 169.8, 155.6, 147.5, 136.2, 135.6, 130.9, 130.3, 129.7, 128.5, 122.4, 105.1, 86.0; IR (KBr, cm\(^{-1}\)): 3449, 2384, 2309, 1632, 1575, 1499, 1431; HRMS (ESI-TOF) calcd for \(\text{C}_{14}\text{H}_{9}\text{I}_2\text{N}_2\text{O}^+\) ([M+H]\(^+\)): 474.8799, found: 474.8803.

**6,8-Diiodo-5-phenyl-2,3-dihydroindolizin-7(1H)-one (3v):**

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (84.1 mg, 91% yield); mp 253.6–255.2 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.59–7.47 (m, 3H), 7.27 (dd, \(J = 7.2, 2.1\) Hz, 2H), 3.94 (t, \(J = 7.4\) Hz, 2H), 3.23 (t, \(J = 7.8\) Hz, 2H), 2.20 (p, \(J = 7.6\) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.0, 154.0, 149.2, 136.0, 130.3, 129.7, 128.2, 127.1, 90.7, 81.4, 56.2, 36.2, 20.2; IR (KBr, cm\(^{-1}\)): 3442, 2958, 1642, 1580, 1480, 1434; HRMS (ESI-TOF) calcd for \(\text{C}_{14}\text{H}_{12}\text{I}_2\text{NO}^+\) ([M+H]\(^+\)): 463.90028, found: 463.90051.

**1,3-Dibromo-4-phenyl-2\(H\)-quinolizin-2-one (3x):**

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid (71.3 mg, 94% yield); mp 264.2–266.1 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.98 (d, \(J = 5.1\) Hz, 1H), 7.70–7.61 (m, 3H), 7.43 (d, \(J = 5.2\) Hz, 1H), 7.30 (dd, \(J = 7.5, 1.6\) Hz, 2H), 7.07 (d, \(J = 5.1\) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.8, 155.6, 147.5, 136.2, 135.6, 130.9, 130.3, 129.7, 128.5, 122.4, 105.1, 86.0; IR (KBr, cm\(^{-1}\)): 3449, 2384, 2309, 1632, 1575, 1499, 1431; HRMS (ESI-TOF) calcd for \(\text{C}_{14}\text{H}_{9}\text{I}_2\text{N}_2\text{O}^+\) ([M+H]\(^+\)): 474.8799, found: 474.8803.
= 9.3 Hz, 1H), 7.68–7.57 (m, 3H), 7.47 (d, \( J = 7.4 \) Hz, 1H), 7.38–7.31 (m, 2H), 7.31–7.26 (m, 1H), 6.57–6.48 (m, 1H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)/CD\(_3\)OD = 20:1) \( \delta \) (ppm): 165.8, 143.4, 141.4, 133.2, 131.0, 130.9, 130.6, 130.2, 129.0, 124.3, 122.4, 113.1, 106.3; IR (KBr, cm\(^{-1}\)): 3474, 2925, 1640, 1589, 1537, 1486; HRMS (ESI-TOF) calcd for C\(_{15}\)H\(_{10}\)Br\(_2\)NO\(^+\) ([M+H]\(^+\)): 377.9124, found 377.9123.

3,4-Diphenyl-2\(H\)-quinoliniz-2-one (4):

![3,4-Diphenyl-2\(H\)-quinoliniz-2-one (4)](image)

Purified by column chromatography (dichloromethane/methanol = 25:1); yellow solid (74.6 mg, 84% yield); mp 307.0–309.0 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) (ppm): 7.41 (d, \( J = 7.4 \) Hz, 1H), 7.36–7.29 (m, 3H), 7.20–7.11 (m, 5H), 7.10–6.96 (m, 4H), 6.76 (s, 1H), 6.36–6.29 (m, 1H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \( \delta \) 174.4, 144.2, 143.4, 135.9, 134.9, 132.5, 130.4, 130.3, 130.0, 129.3, 129.1, 128.1, 127.6, 126.9, 124.3, 111.5; IR (KBr, cm\(^{-1}\)): 3432, 3088, 3061, 2963, 2924, 2857, 1969, 1612, 1541; HRMS (ESI-TOF) calcd for C\(_{21}\)H\(_{16}\)NO\(^+\) ([M+H]\(^+\)): 298.1226, found: 298.1229.

1,3,4-Triphenyl-2\(H\)-quinoliniz-2-one (5):

![1,3,4-Triphenyl-2\(H\)-quinoliniz-2-one (5)](image)

Purified by column chromatography (dichloromethane/methanol =100:1); green solid (80.7 mg, 72% yield); mp 318.0–319.0 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.52 (d, \( J = 7.5 \) Hz, 1H), 7.46 (d, \( J = 4.4 \) Hz, 4H), 7.40–7.31 (m, 4H), 7.26 (t, \( J = 8.7 \) Hz, 3H), 7.15–7.08 (m, 4H), 7.08–7.01 (m, 1H), 6.90 (dd, \( J = 9.2, 6.4 \) Hz, 1H), 6.33 (t, \( J = 6.4 \) Hz, 1H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \( \delta \) 172.2, 142.8, 142.0, 135.3, 135.0, 133.5, 132.9, 131.3, 130.8, 130.4, 130.3, 129.2, 129.2, 128.4, 127.5, 127.3, 127.3, 126.6, 123.3, 123.0, 111.1; IR (KBr, cm\(^{-1}\)):
3388, 3062, 3028, 2928, 1726, 1607, 1563, 1519, 1481, 1438; HRMS (ESI-TOF) calcd for 
C_{27}H_{20}NO^+ ([M+H]^+): 374.1539, found: 374.1543.

1-Bromo-3-iodo-4-phenyl-2H-quinolinizin-2-one (6):

Purified by column chromatography (dichloromethane/methanol = 100:1); yellow solid 
(80.4 mg, 94% yield); mp 227.4–229.2 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.97 (d, J = 9.3 Hz, 1H), 7.71–7.57 (m, 3H), 7.47 (d, J = 7.4 Hz, 1H), 7.27 (dd, J = 14.7, 7.1 Hz, 3H), 
6.48–6.39 (m, 1H); ^13C NMR (75 MHz, CDCl_3/CD_3OD = 12:1) δ (ppm): 167.2, 146.3, 
141.7, 136.6, 131.8, 131.0, 130.6, 130.3, 129.1, 124.3, 113.1, 105.6, 103.4; IR (KBr, cm^{-1}): 
3441, 3062, 1637, 1577, 1485; HRMS (ESI-TOF) calcd for C_{15}H_{9}BrINO C_{15}H_{10}BrINO^+ 
([M+H]^+): 425.8985, found 425.8984.
5. $^1$H NMR and $^{13}$C NMR Spectra
6. Crystallography
Diffraction data were collected at 293 K on a Bruker SMART-CCD diffractometer using graphite-monochromated Mo Kα radiation. The structure was solved by direct methods and refined by full-matrix least squares on \( F^2 \). All nonhydrogen atoms were refined anisotropically, and the hydrogen atoms were included in idealized positions. All calculations were performed using the SHELXTL crystallographic software packages. CCDC-1940902 (2a) and CCDC-1940903 (3a) contain supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

![Molecular structures of 2a and 3a](image)

Figure S1 Molecular structures of 2a and 3a with thermal ellipsoids drawn at 30% probability. Hydrogen atoms have been omitted for clarity.
7. Mechanism Studies
To prove the alpha-iodination of substrate 1a with NIS, we try our best to isolate the alpha-iodination product S1. Unfortunately, the compound S1 can’t be isolated possibly due to its instability.

\[
\text{1a} \rightarrow \text{NIS (0.5 eq)} \rightarrow \text{S1}
\]

Therefore, we attempt to capture the information of compound S1 by monitoring the reaction course by \(^1\)H NMR spectroscopy (Fig. S2). When substrate 1a mixed with 0.5 eq NIS for 1 min, the characteristic peak of methyne (H5) in compound S1 were observed at 6.13 ppm. Next, 2 eq NIS was added into the reaction mixture, and substrate 2a and compound S1 quickly disappeared. Meanwhile, the products 2a, 3a and succinimide were also observed. Expectedly, the diiodination product 3a was almost only observed after 2 h. Base on the control experiments and \(^1\)H NMR monitoring, two competitive reaction procedure for preparation of diiodination products 3 may occur during the cyclization process.

**Fig. S2** Monitoring the reaction by \(^1\)H NMR.