Electronic Supplementary Information

An ultrastable olefin-linked covalent organic framework for photocatalytic
decarboxylative alkylation under highly acidic conditions

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I. General information

$^1$H NMR spectra were recorded on Bruker 400 or 500 MHz spectrometer with CDCl$_3$ and DMSO-$d_6$ as the solvent; $^{13}$C NMR spectra were recorded on Bruker 101 or 126 MHz spectrometer with CDCl$_3$ as the solvent. Chemical shifts were reported in parts per million (δ) with TMS (0 ppm) as the internal standard. The peak patterns are indicated as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br s = broad singlet. The coupling constants (J) are reported in Hertz (Hz). The unknown products were additionally characterized by HRMS. Powder X-ray diffraction (PXRD) patterns of the as-prepared samples were obtained on a powder X-ray diffractometer (Cu Kα radiation source, Ultima IV, Rigaku). UV-Vis diffuse reflectance spectra were performed using a Hitachi UH4150 spectrophotometer in the wavelength range of 300-800 nm. FT-IR spectra were collected in the range of 400-4000 cm$^{-1}$ on Bruker IFS 66 v/s Fourier transform infrared spectrometer. Brunauer-Emmett-Teller (BET) surface area analysis was carried out using a Micromeritics TriStar II 3020 instrument at 77 K. Scanning electron microscopy (SEM) was performed using a ZEISS Gemini 300. The solid phases $^{13}$C CP/MAS NMR spectra were obtained on a Bruker 400 MHz or Agilent 600 MHz solid state NMR spectrometer. Electron spin resonance spectra (ESR) were collected on a JES-FA200 (JEOL) electron paramagnetic resonance spectrometer under visible-light irradiation.

Electrochemical measurements were carried out on a three-electrode system with CHI660E electrochemical workstation. Indium-tin oxide (ITO) glasses were cleaned by sonication in acetone for 15 min and dried under UV lamp. 5 mg of 2D-COFs powder was mixed with 0.2 mL of DMF and 0.2 mL 5 wt % Nafion to get slurry, which was spreading on the surface of ITO glass and the boundary were protected by Scotch tape. Then put it in the vacuum oven at 100 °C for 2 h. After to the room temperature and removed the Scotch tape. The measurements were carried out in a 0.1 mol L$^{-1}$ Sodium sulfate, Ag/AgCl electrode (saturated KCl) as reference electrode, a platinum wire as the counter electrode for photocurrent responses, electrochemical impedance spectra and Mott-Schottky (M-S) experiments. Visible-light-irradiation was provided by a 300 W xenon lamp with a λ > 400 nm cut-off filter. The potential was measured by using a glassy carbon working electrode, the electrolyte was a 0.1 mol L$^{-1}$ solution of tetrabutylammonium hexafluorophosphate in acetonitrile. A Pt electrode, and a calomel electrode (SCE) as counter and reference electrode. Scan rate: 100 mV/s.

All starting materials (from energy chemical or bidepharm) and solvents were used as received and
without further purification, unless otherwise specified.

II. Preparation of 2D-COFs and substrates

i. Preparation of 2D-COFs

2D-COF-1, 2D-COF-2, 2D-COF-3, 2D-COF-4, and 2D-COF-5 were synthesized according to the literature and our previously reported methods.

![Figure S1. Synthesis of 2D-COF-1.](image)

![Figure S2. Synthesis of 2D-COF-2.](image)
Figure S3. Synthesis of 2D-COF-3.

Figure S4. Synthesis of 2D-COF-4.

Figure S5. Synthesis of 2D-COF-5.
ii. Preparation of alkyl NHPI esters

According to literature reports, the alkyl NHPI esters can be synthesized by the condensation of \( N \)-hydroxyphthalimide with the corresponding carboxylic acids.

\[
\text{C}^\text{1.2equiv.} \quad \text{C}^\text{1.0equiv.} \quad \text{DMAP (5 mol%), DCC (1.2 equiv.)} \quad \text{CH}_2\text{Cl}_2, \text{r.t.} \quad \text{C}
\]

\( N \)-hydroxyphthalimide (5 mmol, 1.0 equiv.) and the corresponding alkyl carboxylic acids (6 mmol, 1.2 equiv.) and 4-dimethylaminopyridine (2.5 mol%) were mixed in a round-bottomed flask with a magnetic stirring bar, then 10 mL CH\(_2\)Cl\(_2\) was added. A solution of \( N, N' \)-dicyclohexylcarbodiimide (6 mmol, 1.2 equiv.) in CH\(_2\)Cl\(_2\) was added slowly. The reaction mixture was allowed to stir at room temperature for 0.5-3 hours. The white solid was removed by filtration and the filtrate was concentrated under reduced pressure. The crude product was purified by chromatography on a silica gel column.

iii. Preparation of quinoxalin-2(1H)-ones

\[
\text{a} \quad \text{b} \quad \text{c}
\]

O-arylenediamine a (10 mmol, 1 equiv.) and ethanol (10 mL) were mixed in a round-bottomed flask with a magnetic stirring bar, and ethyl 2-oxoacetate (11 mmol, 1.1 equiv.) was added. The reaction mixture was stirred at reflux for 1 h, then maintained overnight at room temperature. The solid was filtered and washed with ethanol, then dried to give quinoxalinone b. Quinoxalinone b (1 equiv.), DMF (5 mL), and potassium carbonate (1.2 equiv.) were mixed in another round-bottomed flask with a magnetic stirring bar, then the corresponding halogenoalcane (1.6 equiv.) was added slowly. The reaction mixture was stirred overnight at room temperature. The liquid was extracted with ethyl acetate. The organic layers were washed with a saturated solution of NH\(_4\)Cl then brine, dried over MgSO\(_4\), filtered and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel to afford the desired product c.
III. Structure characterizations and photocatalytic properties of 2D-COFs

In our previous\textsuperscript{1,16} and newly published online work\textsuperscript{8}, some chemical structure characterizations and photocatalytic property measurements of 2D-COF-1, 2D-COF-3, and 2D-COF-5 have been already performed and reported, so they are not repeated here.

![13C CP/MAS NMR spectra of 2D-COF-2.](image)

*Figure S6. 13C CP/MAS NMR spectra of 2D-COF-2.*

![13C CP/MAS NMR spectra of 2D-COF-4.](image)

*Figure S7. 13C CP/MAS NMR spectra of 2D-COF-4.*
Figure S8. IR spectra of 2D-COF-2 and 2D-COF-4.

Figure S9. Powder X-ray diffraction of 2D-COFs.
Figure S10. Cyclic voltammetry of 2D-COF-2 at a scan rate of 100 mV/s.

Figure S11. Cyclic voltammetry of 2D-COF-4 at a scan rate of 100 mV/s.

Figure S12. Direct Kubelka-Munk plot for 2D-COF-2 and 2D-COF-4.
**Figure S13.** Mott-Schottky (M-S) plot for 2D-COFs measured in 0.1 mol L⁻¹ Na₂SO₄ (pH 7.1) with Ag/AgCl (+0.199 V vs. NHE) as the reference electrode under dark.

**Figure S14.** (a) N₂ adsorption and desorption isotherms of 2D-COF-2 measured at 77 K. (b) Pore size distributions of 2D-COF-2 derived from N₂ sorption isotherm measured at 77 K.
Figure S15. (a) N$_2$ adsorption and desorption isotherms of 2D-COF-3 measured at 77 K. (b) Pore size distributions of 2D-COF-3 derived from N$_2$ sorption isotherm measured at 77 K.

Figure S16. (a) N$_2$ adsorption and desorption isotherms of 2D-COF-4 measured at 77 K. (b) Pore size distributions of 2D-COF-4 derived from N$_2$ sorption isotherm measured at 77 K.

Figure S17. (a) N$_2$ adsorption and desorption isotherms of 2D-COF-5 measured at 77 K. (b) Pore size distributions of 2D-COF-5 derived from N$_2$ sorption isotherm measured at 77 K.
IV. Optimization of the reaction conditions

Table S1. Screening of photocatalysts\textsuperscript{a}.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Change from the standard reaction conditions</th>
<th>Yields (%)\textsuperscript{b}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2D-COF-1</td>
<td>69</td>
</tr>
<tr>
<td>2</td>
<td>2D-COF-2</td>
<td>85</td>
</tr>
<tr>
<td>3</td>
<td>2D-COF-3</td>
<td>78</td>
</tr>
<tr>
<td>4</td>
<td>2D-COF-4</td>
<td>59</td>
</tr>
<tr>
<td>5</td>
<td>2D-COF-5</td>
<td>50</td>
</tr>
</tbody>
</table>

\textsuperscript{a} Standard reaction conditions: 1 (0.1 mmol, 12.9 mg), 2a (0.2 mmol, 54.6 mg), 2D-COFs (2D-COF-1 (4 mg), 2D-COF-2 (2 mg), 2D-COF-3 (3 mg), 2D-COF-4 (4 mg), 2D-COF-5 (4 mg)), TFA (0.2 mmol, 22.8 mg) in 2 mL DMA, 2* 456 nm blue LED, Ar, r.t., 24 h. \textsuperscript{b} Isolated yield.

Figure S18. PXRD patterns of newly synthesized 2D-COFs (red), PXRD patterns of recycled 2D-COFs (30 mg) after the treatment with TFA (3 mmol) in DMA (5 mL) for 24 h (blue), PXRD patterns of recycled 2D-COFs (30 mg) after the treatment with blue LED irradiation (2*40 W blue LEDs 456 nm) in DMA (5 mL) for 24 h (green).
Figure S19. The recovery rate of 2D-COFs (30 mg) under standard reaction conditions.

Table S2. Control experiments\textsuperscript{a}.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Change from standard conditions</th>
<th>Yields (%)\textsuperscript{b}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>under oxygen atmosphere</td>
<td>N.R.</td>
</tr>
<tr>
<td>2</td>
<td>under air atmosphere</td>
<td>N.R.</td>
</tr>
<tr>
<td>3</td>
<td>without light</td>
<td>N.R.</td>
</tr>
<tr>
<td>4</td>
<td>390 nm blue LED, 40 W</td>
<td>64%</td>
</tr>
<tr>
<td>5</td>
<td>427 nm blue LED, 40 W</td>
<td>49%</td>
</tr>
<tr>
<td>6</td>
<td>467 nm blue LED, 40 W</td>
<td>trace</td>
</tr>
</tbody>
</table>

\textsuperscript{a} Standard reaction conditions: 1 (0.1 mmol, 12.9 mg), 2a (0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), TFA (0.2 mmol, 22.8 mg) in 2 mL DMA, 2* 456 nm blue LED, 40 W, Ar, r.t., 24 h. \textsuperscript{b} Isolated yield.
Table S3. Screening of different additives and solvents$^a$.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Additive</th>
<th>Solvent</th>
<th>Yields (%)$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TFA</td>
<td>DMA</td>
<td>85</td>
</tr>
<tr>
<td>2</td>
<td>TFA</td>
<td>DMF</td>
<td>43</td>
</tr>
<tr>
<td>3</td>
<td>TFA</td>
<td>DMSO</td>
<td>N.R.</td>
</tr>
<tr>
<td>4</td>
<td>TFA</td>
<td>Acetone</td>
<td>N.R.</td>
</tr>
<tr>
<td>5</td>
<td>TFA</td>
<td>EtOH</td>
<td>76</td>
</tr>
<tr>
<td>6</td>
<td>TFA</td>
<td>DIPE</td>
<td>N.R.</td>
</tr>
<tr>
<td>7</td>
<td>TFA</td>
<td>EA</td>
<td>N.R.</td>
</tr>
<tr>
<td>8</td>
<td>TFA</td>
<td>DCM</td>
<td>N.R.</td>
</tr>
<tr>
<td>9</td>
<td>TFA</td>
<td>CHCl$_3$</td>
<td>N.R.</td>
</tr>
<tr>
<td>10</td>
<td>TFA</td>
<td>CH$_3$OH</td>
<td>63</td>
</tr>
<tr>
<td>11</td>
<td>TFA</td>
<td>H$_2$O</td>
<td>Trace</td>
</tr>
<tr>
<td>12</td>
<td>TFA</td>
<td>MeCN</td>
<td>N.R.</td>
</tr>
<tr>
<td>13</td>
<td>TfOH</td>
<td>DMA</td>
<td>62</td>
</tr>
<tr>
<td>14</td>
<td>CH$_3$COOH</td>
<td>DMA</td>
<td>5</td>
</tr>
<tr>
<td>15</td>
<td>Adamantoic acid</td>
<td>DMA</td>
<td>69</td>
</tr>
<tr>
<td>16</td>
<td>-</td>
<td>DMA</td>
<td>44</td>
</tr>
</tbody>
</table>

$^a$ Reaction conditions: 1 (0.1 mmol, 12.9 mg), 2a (0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), additive (0.2 mmol, 2 equiv.), solvent 2 mL, Ar, r.t., 24 h, 2*40 W blue LEDs (456 nm). $^b$ Isolated yield.
V. General procedures for visible-light-driven decarboxylative alkylation of heterocycles

i. General procedure A

\[
\text{Het} + \underset{0.1 \text{ mmol}}{\text{alkyl NHPI ester}} \xrightarrow{2 \text{ equiv.}} \text{2D-COF-2 (2 mg)} \xrightarrow{\text{TFA 2 equiv., DMA 2 mL, Ar, r.t., 24 h, 2" 456 nm blue LEDs}} \text{Het}_\text{alkyl}
\]

To a 10 mL glass tube was charged with heterocycles (0.1 mmol, 1 equiv.), alkyl NHPI ester (0.2 mmol, 2 equiv.), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMA (2 mL) and TFA (0.2 mmol, 2 equiv.) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, the remaining mixture was slowly added to a saturated aq solution of Na₂CO₃ (10 mL). The aqueous layer was extracted with ethyl acetate (5 mL × 3). The organic layers were washed with saturated brine then dried (MgSO₄), filtered, and concentrated under reduced pressure. The remaining mixture was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product.

ii. General procedure B

\[
\text{R₁=}[\text{N}=\text{O}] + \underset{0.1 \text{ mmol}}{\text{alkyl NHPI ester}} \xrightarrow{2 \text{ equiv.}} \text{2D-COF-2 (2 mg)} \xrightarrow{\text{Et₃N 3 equiv., DMA 2 mL, Ar, r.t., 24 h, 2" 456 nm blue LEDs}} \text{R₂}
\]

To a 10 mL glass tube was charged with quinoxalin-2(1H)-ones (0.1 mmol, 1 equiv.), alkyl NHPI ester (0.2 mmol, 2 equiv.), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMA (2 mL) and Et₃N (0.3 mmol, 3 equiv.) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, the remaining mixture was quenched with water and extracted with ethyl acetate (5 mL × 3). The organic layers were washed with saturated
brine then dried (MgSO₄), filtered, and concentrated under reduced pressure. The remaining mixture was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product.

**iii. General procedure C**

![Chemical structure](image)

To a 10 mL glass tube was charged with N-oxides (0.1 mmol, 1 equiv.), alkyl NHPI ester (0.15 mmol, 1.5 equiv.), Cs₂CO₃ (2.5 mol%), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMF (2 mL) was added via a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 12 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, the remaining mixture was quenched with water and extracted with ethyl acetate (5 mL x 3). The organic layers were washed with saturated brine then dried (MgSO₄), filtered, and concentrated under reduced pressure. The residue was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product.

**VI. Recycling experiments**

![Chemical structure](image)

To a 10 mL glass tube was charged with Isoquinoline (1, 0.1 mmol, 12.9 mg), cyclohexyl NHPI ester (2a, 0.2 mmol, 54.6 mg), and 2D-COF-2 (2 mg). The tube was evacuated and filled with argon for three cycles. Then, DMA (2 mL) and TFA (0.2 mmol, 22.8 mg) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/ cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, and washed with plenty of ethyl acetate,
ethanol and water. Then the powder was dried at 120 °C under vacuum for 6 h to yield the recovered 2D-COF-2. In addition, the remaining mixture was slowly added to a saturated aq solution of Na₂CO₃ (10 mL). The aqueous layer was extracted with ethyl acetate (5 mL × 3). The organic layers were washed with saturated brine then dried (MgSO₄), filtered, and concentrated under reduced pressure. The residue was purified on silica gel (petroleum ether and ethyl acetate) to afford the desired product 3.

Figure S20. (a) N₂ sorption isotherm of 2D-COF-2. (b) Pore size distributions of 2D-COF-2.

Figure S21. (a) N₂ sorption isotherm of 2D-COF-2 after recycling. (b) Pore size distributions of 2D-COF-2 after recycling.
Figure S22. Multi-point BET plot derived from the N$_2$ sorption isotherm of 2D-COF-2 before and after recycling measured at 77 K. $S_{\text{BET}}$.

$2D$-COF-2 = 225 m$^2$ g$^{-1}$; $r = 0.99815$; $S_{\text{BET-Recycled 2D-COF-2}} = 266 m^2 g^{-1}$; $r = 0.99800$.

Figure S23. $^{13}$C CP/MAS NMR spectra of 2D-COF-2 before and after recycling.

Figure S24. SEM images of 2D-COF-2 before and after recycling.
VII. Application and scale up experiment

Isoquinoline (1, 0.5 g, 3.88 mmol), alkyl NHPI ester (2q, 2 equiv., 7.76 mmol), 2D-COF-2 (30 mg) were added into a reaction flask (50 mL) equipped with a magnetic stir bar. Then, the flask was evacuated and filled with argon for three cycles. TFA (7.76 mmol, 2 equiv.) and EtOH (30 mL) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred (1500 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 72 h. Upon completion, the photocatalyst 2D-COF-2 was removed by centrifugation, then Potassium carbonate (3.88 mmol, 0.535 g) was added into the remaining mixture for neutralization, the solvent was removed under reduced pressure and the residue was on silica gel (petroleum ether and ethyl acetate) to afford the desired product 58 (0.31 g, 68 %).

VIII. Radical intermediate capture experiment

Isoquinoline (1, 0.1 mmol, 12.9 mg), cyclohexyl NHPI ester (2a, 0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), and 2,2,6,6-tetramethyl-1-piperidinoxyloxyl (TEMPO, 0.2 mmol, 31.2 mg) were placed in a glass tube equipped with a stirring bar, the tube was evacuated and filled with argon for three cycles. TFA (0.2 mmol, 22.8 mg) and DMA (2 mL) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred (1000 rpm) with the irradiation of two 40 W blue LED (456 nm, the power density of the incident light near the reactor is 0.003 W/cm², and the distance between reactor and lamp is approximately 5 cm.) at room temperature for 24 h. The reaction was greatly suppressed. The photocatalyst 2D-COF-2 was removed by centrifugation, the above solution was detected by GC-MS. The cyclohexyl radical adduct 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine was observed in GC-MS spectra. The desired product 3 was isolated in 14 % yield.
Figure S25. GC-MS spectra of the model reaction in the presence of 2 equiv. TEMPO

IX. Calculation of Apparent Quantum Efficiency (A.Q.E.)

In principle, it takes one photon to generate one radical to form one target product. The energy of one photon ($E_{\text{photon}}$) with wavelength of $\lambda_{\text{inc}}$ (nm) is calculated using eq.1.

$$E_{\text{photon}} = \frac{hc}{\lambda_{\text{inc}}} = \frac{6.63 \times 10^{-34} \text{ J} \cdot \text{s} \times 3 \times 10^8 \text{ m/s}}{456 \times 10^{-9} \text{ m}} = 4.36 \times 10^{-19} \text{ J} \quad \text{(eq.1)}$$

Where $h$ (J·s) is Planck’s constant, $c$ (m·s$^{-1}$) is the speed of light and $\lambda_{\text{inc}}$ (m) is the wavelength of the incident light, 456 nm. And the total energy of the incident monochromatic light ($E_{\text{total}}$) is calculated using eq.2.

$$E_{\text{total}} = PSt \quad \text{(eq.2)}$$
Where $P$ (W·cm$^{-2}$) is the power density of the incident light, $S$ (cm$^2$) is the irradiation area, $t$ (s) is the photoreaction time. The total number of incident photons can be obtained through eq.3.

$$\text{Number of incident photons} = \frac{E_{\text{tot}}}{E_{\text{photon}}} \quad \text{(eq.3)}$$

The apparent quantum efficiency (A.Q.E.) is defined as eq.4.

$$\text{A.Q.E.} \% = \frac{\text{Number of product}}{\text{Number of incident photons}} \times 100\% \quad \text{(eq.4)}$$

A.Q.E% of the reaction between 1 and 2a.

$$E_{\text{tot}} = PST = 0.003 \text{ W} / \text{cm}^2 \times 2 \text{ cm}^2 \times 24 \times 3600 \text{ s} = 518.4 \text{ J}$$

$$\text{Number of incident photons} = \frac{E_{\text{tot}}}{E_{\text{photon}}} = \frac{518.4 \text{ J}}{4.36 \times 10^{19} \text{ J}} = 1.19 \times 10^{21} = 1.97 \text{ mmol}$$

$$\text{A.Q.E.} \% = \frac{\text{Number of product}}{\text{Number of incident photons}} \times 100 \% = \frac{0.085 \text{ mmol}}{1.97 \text{ mmol}} \times 100 \% = 4.3 \%$$

**X. In situ ESR spectra**

A glass tube was charged with cyclohexyl NHPI ester 2a (0.2 mmol, 54.6 mg), 2D-COF-2 (2 mg), TFA (0.2 mmol, 22.8 mg) and 2 mL DMA in the dark room. The mixture was degassed by bubbling with N$_2$ for 5 minutes. Under nitrogen atmosphere, 50 μL of DMPO was added. The formed mixture (50-100 μL) was transferred into the ESR capillary and sealed quickly and subsequently. The ESR spectra was firstly recorded under dark. Secondly, it was recorded with the light irradiation from a 300 W Xenon lamp (λ >400 nm) for 2 minutes.

Finally, another glass tube was charged with cyclohexyl NHPI ester 2a (0.2 mmol, 54.6 mg), isoquinoline 1 (0.1 mmol, 12.9 mg), 2D-COF-2 (2 mg), TFA (0.2 mmol, 22.8 mg) and 2 mL DMA in the dark room. The mixture was degassed by bubbling with N$_2$ for 5 minutes. Under nitrogen atmosphere, 50 μL of DMPO was added. The formed mixture (50-100 μL) was transferred into the ESR capillary and sealed quickly and subsequently. The third ESR spectra was then recorded with the light irradiation from a 300 W Xenon lamp (λ >400 nm) for 2 minutes.
ESR conditions: frequency (9.057 GHz), power (0.998 mW), modulation width (0.1 mT), center field (322.500 mT), sweep width (5 mT), sweep time (1 min), time constant (0.03 s).

XI. Characterization data of compounds 3-58.

1-cyclohexylisoquinoline (3)\(^9\)

Following the general procedure A, 3 was obtained in 85% yield (18 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.44 (d, \(J = 5.7\) Hz, 1H), 8.13 (d, \(J = 8.4\) Hz, 1H), 7.67 (d, \(J = 7.7\) Hz, 1H), 7.55-7.43 (m, 2H), 7.36 (d, \(J = 5.7\) Hz, 1H), 3.50 (tt, \(J = 11.5, 3.2\) Hz, 1H), 1.97-1.73 (m, 7H), 1.54-1.29 (m, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 165.45, 141.78, 136.19, 129.31, 127.35, 126.61, 126.09, 124.50, 118.70, 41.39, 32.47, 26.76, 26.16.

1-cyclobutylisoquinoline (4)\(^10\)

Following the general procedure A, 4 was obtained in 82% yield (15 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.49 (d, \(J = 5.7\) Hz, 1H), 8.08-8.04 (m, 1H), 7.80 (d, \(J = 8.1\) Hz, 1H), 7.64 (ddd, \(J = 8.1, 6.9, 1.2\) Hz, 1H), 7.56 (ddd, \(J = 8.3, 6.9, 1.3\) Hz, 1H), 7.49 (d, \(J = 5.7\) Hz, 1H), 4.37 (p, \(J = 8.7\) Hz, 1H), 2.69-2.58 (m, 2H), 2.50 (m, 2H), 2.25-2.12 (m, 1H), 2.01-1.92 (m, 1H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 163.60, 141.92, 136.24, 129.77, 127.42, 126.87, 126.47, 125.34, 119.13, 39.46, 27.85, 18.69.

5-bromo-1-cyclobutylisoquinoline (5)\(^11\)

Following the general procedure A, 5 was obtained in 52% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.63-8.52 (m, 1H), 8.04 (d, \(J = 8.4\) Hz, 1H), 7.97-7.83 (m, 2H), 7.60-7.36 (m, 1H), 4.35 (p, \(J = 8.6\) Hz, 1H), 2.67-2.57 (m, 2H), 2.50 (m, 2H), 2.24-2.14 (m, 1H), 1.96 (m, 1H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 163.99, 143.29, 135.42, 133.57, 127.60, 127.20, 125.05, 122.49, 117.96, 39.55, 27.93, 18.64.
1-cyclohexyl-6-methylisoquinoline (6)$^{10}$

Following the general procedure A, 6 was obtained in 80% yield (18 mg). Eluent (petroleum ether: ethyl acetate, 10:1). White solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.43 (d, $J = 5.7$ Hz, 1H), 8.11 (d, $J = 8.7$ Hz, 1H), 7.57 (s, 1H), 7.42-7.37 (m, 2H), 3.52 (tt, $J = 11.7$, 3.2 Hz, 1H), 2.53 (s, 3H), 1.99-1.90 (m, 4H), 1.86-1.76 (m, 3H), 1.53 (qt, $J = 12.8$, 3.7 Hz, 2H), 1.40 (m, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 165.51, 142.17, 139.83, 136.83, 129.13, 126.59, 124.78, 124.71, 118.56, 41.63, 32.70, 27.03, 26.39, 21.91.

6-bromo-1-cyclohexylisoquinoline (7)$^{10}$

Following the general procedure A, 7 was obtained in 73% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 10:1). White solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.49 (d, $J = 5.7$ Hz, 1H), 8.08 (d, $J = 9.1$ Hz, 1H), 7.97 (d, $J = 2.0$ Hz, 1H), 7.64 (dd, $J = 9.0$, 2.0 Hz, 1H), 7.38 (d, $J = 5.7$ Hz, 1H), 3.49 (tt, $J = 11.7$, 3.2 Hz, 1H), 1.98-1.89 (m, 4H), 1.86-1.75 (m, 3H), 1.51 (m, 2H), 1.39 (m, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 166.11, 143.18, 137.72, 130.41, 129.76, 126.72, 124.81, 124.37, 117.95, 41.77, 32.71, 26.94, 26.31.

1-octylisoquinoline (8)$^{12}$

Following the general procedure A, 8 was obtained in 54% yield (13 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.43 (d, $J = 5.7$ Hz, 1H), 8.18-8.13 (m, 1H), 7.80 (d, $J = 8.1$ Hz, 1H), 7.65 (ddd, $J = 8.1$, 6.9, 1.2 Hz, 1H), 7.58 (ddd, $J = 8.2$, 6.9, 1.3 Hz, 1H), 7.49 (d, $J = 5.7$ Hz, 1H), 3.31-3.26 (m, 2H), 1.89-1.82 (m, 2H), 1.52-1.24 (m, 10H), 0.90-0.85 (m, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 162.60, 142.05, 136.38, 129.85, 127.50, 127.04, 127.03, 125.51, 119.23, 35.76, 31.95, 30.03, 29.99, 29.36, 22.80, 14.24.

1-(1-phenylcyclopropyl) isoquinoline (9)$^{10}$

Following the general procedure A, 9 was obtained in 71% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.54 (d, $J = 5.7$ Hz, 1H), 8.31 (d, $J = 8.5$ Hz, 1H), 7.81 (d, $J = 8.2$ Hz, 1H), 7.63-7.58 (m, 2H), 7.47 (ddd, $J = 8.2$, 7.0, 1.1 Hz, 1H), 7.21-7.17 (m, 2H), 7.10 (m, 3H), 1.60
(m, 4H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 162.44, 144.68, 142.00, 136.85, 129.84, 128.39, 127.99, 127.36, 127.06, 127.03, 126.09, 125.72, 120.33, 30.09, 16.75.

**tert-butyl 2-(isoquinolin-1-yl)pyrrolidine-1-carboxylate (10)$^{13}$**

Following the general procedure A, 10 was obtained in 67% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.44 (m, 1H), 8.23-8.15 (m, 1H), 7.81 (m, 1H), 7.68-7.48 (m, 3H), 5.88-5.58 (m, 1H), 3.86 (m, 1H), 3.76-3.57 (m, 1H), 2.48 (m, 1H), 2.11-1.92 (m, 3H), 1.44 (s, 3H), 0.91 (s, 6H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 162.23, 161.85, 154.48, 154.46, 141.91, 136.33, 129.75, 127.60, 127.03, 124.39, 124.15, 119.84, 119.63, 79.28, 78.84, 59.59, 58.75, 47.45, 47.18, 34.02, 33.01, 28.64, 28.04, 24.17, 23.89.

**4-cyclobutylquinoline:2-cyclobutylquinoline (11, 11')$^{11}$**

Following the general procedure A, 11 and 11' was obtained in 71% yield (13 mg, 11:11'=7:4). Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 8.07 (dd, $J = 8.2$, 5.8 Hz, 2H), 7.86 (d, $J = 8.3$ Hz, 1H), 7.77 (d, $J = 8.0$ Hz, 1H), 7.70-7.62 (m, 2H), 7.49-7.43 (m, 2H), 7.35 (d, $J = 8.5$ Hz, 1H), 7.20 (s, 1H), 4.12 (p, $J = 8.8$ Hz, 1H), 3.87 (h, $J = 8.6$ Hz, 2H), 2.58-2.52 (m, 2H), 2.51-2.44 (m, 6H), 2.32 (pd, $J = 9.2$, 2.4 Hz, 2H), 2.24-2.17 (m, 1H), 2.17-2.08 (m, 2H), 2.00-1.91 (m, 3H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 165.21, 164.90, 151.17, 148.01, 147.94, 136.28, 129.83, 129.37, 129.19, 128.84, 127.57, 126.93, 125.75, 125.72, 125.29, 123.93, 119.72, 116.08, 43.11, 42.92, 37.49, 28.93, 28.41, 18.81, 18.52.

**4-cyclobutyl-3-methylquinoline:2,4-dicyclobutyl-3-methylquinoline (12, 12')**

Following the general procedure A, 12 and 12' was obtained in 60% yield (14 mg, 12:12'=2:5). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.09-8.03 (m, 1H), 8.00-7.96 (m, 1H), 7.58 (m, 1H), 7.41 (m, 1H), 4.26 (p, $J = 9.2$ Hz, 1H), 3.98-3.86 (m, 1H), 2.72 (m, 2H), 2.61 (m, 3H), 2.22-2.08 (m, 1H), 2.08-1.91 (m, 3H), 1.91-1.78 (m, 2H), 1.78-1.64 (m, 2H), 1.54-1.41 (m, 2H), 1.41-1.26 (m, 2H), 1.26-1.13 (m, 2H), 1.13-0.99 (m, 2H), 0.99-0.86 (m, 2H), 0.86-0.73 (m, 2H), 0.73-0.59 (m, 2H), 0.59-0.46 (m, 2H), 0.46-0.33 (m, 2H), 0.33-0.19 (m, 2H), 0.19-0.06 (m, 2H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 165.21, 164.90, 151.17, 148.01, 147.94, 136.28, 129.83, 129.37, 129.19, 128.84, 127.57, 126.93, 125.75, 125.72, 125.29, 123.93, 119.72, 116.08, 43.11, 42.92, 37.49, 28.93, 28.41, 18.81, 18.52.
2.52-2.45 (m, 2H), 2.41 (qd, J = 5.9, 3.6 Hz, 4H), 2.28 (s, 3H), 2.18-2.05 (m, 3H), 1.96-1.87 (m, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 163.38, 147.99, 146.56, 135.43, 129.95, 129.02, 128.19, 127.29, 126.91, 126.69, 126.30, 125.67, 124.49, 124.45, 41.04, 40.52, 38.76, 31.98, 27.14, 26.86, 19.30, 18.99, 18.22, 18.15, 16.33.

6-bromo-4-cyclobutylquinoline: 6-bromo-2-cyclobutylquinoline (13, 13’)

Following the general procedure A, 13 and 13’ was obtained in 68% yield (18 mg, 13:13’=1.78:1). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.99-7.96 (m, 2H), 7.94-7.90 (m, 2H), 7.71 (m, 2H), 7.35 (d, $J = 8.5$ Hz, 1H), 7.19 (s, 1H), 4.04 (p, $J = 9.1$ Hz, 1H), 3.83 (h, $J = 8.4$ Hz, 2H), 2.54 (m, 2H), 2.49-2.42 (m, 7H), 2.30 (pd, $J = 8.8$, 8.3, 2.1 Hz, 2H), 2.25-2.07 (m, 3H), 1.95 (m, 3H), 1.37-1.22 (m, 1H), 0.87 (m, 1H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 165.69, 165.39, 150.41, 146.66, 146.55, 135.25, 132.76, 132.19, 131.57, 130.97, 129.60, 128.06, 127.00, 126.39, 120.68, 119.42, 119.22, 116.98, 43.01, 42.83, 37.23, 28.90, 28.30, 28.29, 18.77, 18.48, 18.47.

4-cyclobutyl-8-methylquinoline: 2-cyclobutyl-8-methylquinoline (14, 14’)

Following the general procedure A, 14 and 14’ was obtained in 66% yield (13 mg, 14:14’=1.85:1). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 8.00 (d, $J = 8.4$ Hz, 1H), 7.70 (d, $J = 8.3$ Hz, 1H), 7.59 (d, $J = 8.1$ Hz, 1H), 7.50 (dd, $J = 14.0$, 7.0 Hz, 2H), 7.37-7.30 (m, 2H), 7.26 (d, $J = 15.5$ Hz, 1H), 7.12 (d, $J = 1.0$ Hz, 1H), 4.10 (p, $J = 8.8$ Hz, 1H), 3.84 (h, $J = 8.4$ Hz, 2H), 2.83 (s, 5H), 2.57-2.48 (m, 6H), 2.42 (m, 4H), 2.29 (pd, $J = 9.1$, 2.4 Hz, 2H), 2.21-2.05 (m, 3H), 2.02-1.88 (m, 3H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 163.78, 163.37, 150.97, 146.93, 146.85, 137.71, 137.16, 136.26, 129.37, 128.95, 126.74, 125.49, 125.36, 124.80, 121.78, 119.70, 116.10, 43.25, 43.06, 37.66, 29.01, 28.37, 18.78, 18.54, 18.53, 17.92.
4-cyclobutyl-6-methoxyquinoline:2,4-dicyclobutyl-6-methoxyquinoline (15, 15’)

Following the general procedure A, 15 and 15’ was obtained in 43% yield (11 mg, 15:15’=1:5). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. 

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.97 (m, 1H), 7.34-7.28 (m, 1H), 7.16 (s, 1H), 7.11 (d, $J = 2.7$ Hz, 1H), 4.05 (p, $J = 8.7$ Hz, 1H), 3.92 (s, 3H), 3.83 (m 1H), 2.55 (m, 2H), 2.49-2.40 (m, 4H), 2.32 (pd, $J = 9.2$, 2.3 Hz, 2H), 2.24-2.15 (m, 1H), 2.14-2.06 (m, 1H), 1.99-1.90 (m, 2H).$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 162.75, 162.46, 157.29, 156.92, 149.78, 143.84, 135.15, 131.19, 130.58, 127.73, 126.41, 121.81, 120.57, 119.93, 116.28, 105.33, 102.77, 55.57, 42.95, 42.75, 37.62, 28.71, 28.51, 18.78, 18.50, 18.48.

2-cyclohexyl-4-methylquinoline (16)

Following the general procedure A, 16 was obtained in 89% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 20:1). Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.05 (d, $J = 8.4$ Hz, 1H), 7.94 (d, $J = 8.3$ Hz, 1H), 7.68-7.64 (m, 1H), 7.49 (t, $J = 7.6$ Hz, 1H), 7.16 (s, 1H), 2.87 (tt, $J = 12.1$, 3.4 Hz, 1H), 2.68 (s, 3H), 2.04-1.99 (m, 2H), 1.89 (dt, $J = 12.9$, 3.0 Hz, 2H), 1.79 (d, $J = 14.0$ Hz, 1H), 1.63 (qd, $J = 12.5$, 3.1 Hz, 2H), 1.47 (m, 2H), 1.35 (m, $J = 12.8$, 3.5 Hz, 1H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 166.65, 147.79, 144.30, 129.66, 129.03, 127.17, 125.47, 123.68, 120.37, 47.76, 32.97, 26.71, 26.28, 18.97.

tert-butyl (1-(4-methylquinolin-2-yl)-2-phenylethyl)carbamate (17)

Following the general procedure A, 17 was obtained in 94% yield (34 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.06 (d, $J = 8.0$ Hz, 1H), 7.94 (d, $J = 8.1$ Hz, 1H), 7.72-7.66 (m, 1H), 7.56-7.51 (m, 1H), 7.17 (s, 3H), 7.01 (d, $J = 4.6$ Hz, 2H), 6.81 (s, 1H), 6.16 (d, $J = 7.2$ Hz, 1H), 5.11 (q, $J = 7.2$ Hz, 1H), 3.31 (dd, $J = 13.2$, 5.6 Hz, 1H), 3.17 (dd, $J = 13.2$, 7.7 Hz, 1H), 2.58 (s, 3H), 1.46 (s, 9H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 159.47, 155.44, 147.43, 144.30, 137.65, 129.79, 129.20, 128.20, 127.45, 126.41, 126.08, 123.80, 121.38, 79.34, 57.06, 42.83, 28.56, 18.73.
4-methyl-2-(tert-pentyl)quinoline (18)\(^\text{10}\)

Following the general procedure A, 18 was obtained in 80% yield (17 mg).

Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.07 (d, \(J = 8.3\) Hz, 1H), 7.96-7.92 (m, 1H), 7.69-7.63 (m, 1H), 7.53-7.46 (m, 1H), 7.30 (s, 1H), 2.69 (s, 3H), 1.85 (q, \(J = 7.5\) Hz, 2H), 1.43 (s, 6H), 0.74 (t, \(J = 7.5\) Hz, 3H). \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 168.18, 147.49, 143.48, 130.10, 128.74, 126.61, 125.48, 123.53, 119.53, 41.32, 35.98, 27.48, 19.13, 9.39.

tert-butyl 2-(4-methylquinolin-2-yl)pyrrolidine-1-carboxylate (19)\(^\text{10}\)

Following the general procedure A, 19 was obtained in 80% yield (28 mg).

Eluent (petroleum ether: ethyl acetate, 10:1). Colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.02 (d, \(J = 8.4\) Hz, 1H), 7.93 (dd, \(J = 16.9, 8.3\) Hz, 1H), 7.65 (m, 1H), 7.49 (m, 1H), 7.15-7.12 (m, 1H), 5.14-4.95 (m, 1H), 3.71 (t, \(J = 6.7\) Hz, 2H), 2.67 (s, 3H), 2.43 (m, 1H), 2.05-1.87 (m, 3H), 1.46 (s, 3H), 1.10 (s, 6H).

\(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 163.98, 154.80, 147.43, 144.66, 134.21, 129.53, 129.25, 127.14, 125.78, 123.71, 118.32, 79.46, 63.67, 47.37, 34.69, 28.21, 23.71, 18.93.

8-chloro-3-cyclohexylquinoxalin-2(1H)-one (20)

Following the general procedure A, 20 was obtained in 88% yield (23 mg).

Eluent (petroleum ether: ethyl acetate, 5:1). White solid. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 11.91 (s, 1H), 7.69 (d, \(J = 7.9\) Hz, 1H), 7.61 (d, \(J = 7.8\) Hz, 1H), 7.28 (t, \(J = 8.0\) Hz, 1H), 3.22-3.14 (m, 1H), 1.84 (dd, \(J = 28.2, 11.8\) Hz, 4H), 1.71 (d, \(J = 12.5\) Hz, 1H), 1.50-1.31 (m, 4H), 1.27-1.19 (m, 1H). \(^13\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 166.64, 154.18, 133.53, 129.04, 127.94, 127.86, 123.71, 118.50, 40.39, 30.57, 26.36, 26.23. HR-MS (ESI)[M+H]\(^+\) m/z calcd for C\(_{14}\)H\(_{16}\)N\(_2\)OCl 263.0946, found 263.0933.

1-methyl-3-octylquinoxalin-2(1H)-one (21)

Following the general procedure B, 21 was obtained in 92% yield (25 mg).

Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.83 (dd, \(J = 8.0, 1.4\) Hz, 1H), 7.52 (ddd, \(J = 8.6, 7.4, 1.5\) Hz, 1H),
7.36-7.28 (m, 2H), 3.70 (s, 3H), 2.96-2.91 (m, 2H), 1.82-1.74 (m, 2H), 1.48-1.21 (m, 10H), 0.89-0.86 (m, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 161.56, 155.06, 133.23, 132.86, 129.73, 129.62, 123.66, 113.68, 34.56, 31.92, 29.72, 29.30, 29.17, 27.03, 22.79, 14.24. HR-MS (ESI)[M+H]$^+$ m/z calcd for C$_{17}$H$_{25}$N$_2$O 273.1961, found 273.1954.

3-heptadecyl-1-methylquinoxalin-2(1H)-one (22)

Following the general procedure B, 22 was obtained in 82% yield (33 mg). Eluent (petroleum ether: ethyl acetate, 3:1). Pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.82 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.53-7.47 (m, 1H), 7.34-7.29 (m, 1H), 7.29-7.26 (m, 1H), 3.69 (s, 3H), 2.96-2.89 (m, 2H), 1.81-1.73 (m, 2H), 1.24 (s, 30H), 0.87 (t, $J = 6.7$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 161.51, 154.99, 142.25, 134.39, 133.19, 132.82, 129.75, 128.66, 128.37, 125.83, 123.66, 113.66, 35.82, 33.99, 29.15, 28.41.

1-methyl-3-(3-phenylpropyl)quinoxalin-2(1H)-one (23)$^{14}$

Following the general procedure B, 23 was obtained in 73% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 5:1). White solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.83 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.52 (ddd, $J = 8.6, 7.5, 1.5$ Hz, 1H), 7.36-7.31 (m, 1H), 7.29-7.24 (m, 5H), 7.16 (dt, $J = 6.9, 2.5$ Hz, 1H), 3.68 (s, 3H), 3.03-2.98 (m, 2H), 2.80-2.75 (m, 2H), 2.19-2.11 (m, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 160.91, 154.99, 142.25, 134.39, 133.19, 132.82, 129.75, 128.66, 128.37, 125.83, 123.66, 113.66, 35.82, 33.99, 29.15, 28.41.

3-(4-methoxyphenethyl)-1-methylquinoxalin-2(1H)-one (24)

Following the general procedure B, 24 was obtained in 93% yield (27 mg). Eluent (petroleum ether: ethyl acetate, 5:1). White solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.92 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.62-7.57 (m, 1H), 7.44-7.39 (m, 1H), 7.38-7.34 (m, 1H), 7.30 (s, 2H), 6.92-6.88 (m, 2H), 3.85 (s, 3H), 3.77 (s, 3H), 3.33-3.28 (m, 2H), 3.14 (m, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$)
δ 160.25, 157.95, 154.95, 133.82, 133.23, 132.81, 129.79, 129.77, 129.62, 123.68, 113.87, 113.69, 55.36, 36.39, 31.81, 29.15. HR-MS (ESI)[M+H]+ m/z calcd for C_{18}H_{19}N_{2}O_{2} 295.1441, found 295.1437.

3-cyclohexyl-1-methylquinoxalin-2(1H)-one (25)\textsuperscript{16}

Following the general procedure A, 25 was obtained in 91% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.79 (dd, \textit{J} = 8.0, 1.2 Hz, 1H), 7.49-7.43 (m, 1H), 7.31-7.26 (m, 1H), 7.25-7.22 (m, 1H), 3.65 (s, 3H), 3.30 (tt, \textit{J} = 11.5, 3.2 Hz, 1H), 1.95-1.80 (m, 4H), 1.73 (d, \textit{J} = 12.6 Hz, 1H), 1.55 (m, 2H), 1.42 (tt, \textit{J} = 12.7, 3.0 Hz, 2H), 1.27 (qt, \textit{J} = 12.1, 3.2 Hz, 1H). \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}) δ 164.36, 154.63, 132.99, 132.95, 129.86, 129.46, 123.47, 113.54, 40.87, 30.62, 29.14, 26.42, 26.26.

1-benzyl-3-cyclohexylquinoxalin-2(1H)-one (26)\textsuperscript{16}

Following the general procedure A, 26 was obtained in 92% yield (29 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.83 (dd, \textit{J} = 7.9, 1.5 Hz, 1H), 7.38-7.34 (m, 1H), 7.32-7.26 (m, 3H), 7.25-7.20 (m, 4H), 5.48 (s, 2H), 3.39 (t, \textit{J} = 11.5 Hz, 1H), 2.03-1.76 (m, 5H), 1.63-1.44 (m, 4H), 1.33 (m, 1H). \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}) δ 164.52, 154.71, 135.62, 133.29, 132.31, 130.01, 129.48, 129.01, 127.74, 127.05, 123.56, 114.36, 46.05, 40.95, 30.71, 26.47, 26.30.

1-allyl-3-cyclohexylquinoxalin-2(1H)-one (27)\textsuperscript{17}

Following the general procedure A, 27 was obtained in 75% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.82 (dd, \textit{J} = 8.0, 1.5 Hz, 1H), 7.44 (ddd, \textit{J} = 8.6, 7.4, 1.5 Hz, 1H), 7.30-7.22 (m, 2H), 5.91 (ddt, \textit{J} = 17.3, 10.4, 5.2 Hz, 1H), 5.26-5.11 (m, 2H), 4.87 (dt, \textit{J} = 5.2, 1.7 Hz, 2H), 3.33 (tt, \textit{J} = 11.6, 3.3 Hz, 1H), 1.98-1.91 (m, 2H), 1.84 (dt, \textit{J} = 12.6, 3.0 Hz, 2H), 1.78-1.71 (m, 1H), 1.55 (qd, \textit{J} = 12.2, 2.5 Hz, 2H), 1.44 (qt, \textit{J} = 12.8, 2.9 Hz, 2H), 1.29 (qt, \textit{J} = 12.4, 3.3 Hz, 1H). \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}) δ 164.45, 154.19, 133.20, 132.19, 130.95, 130.00, 129.40, 123.49, 118.11, 114.11, 44.66, 40.87, 30.67, 26.45, 26.29.
3-cyclohexyl-1-(prop-2-yn-1-yl)quinoxalin-2(1H)-one (28)\textsuperscript{17}

Following the general procedure A, 28 was obtained in 86\% yield (23 mg).
Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.84 (dd, \( J = 8.1, 1.2 \) Hz, 1H), 7.53 (td, \( J = 7.9, 7.2, 1.4 \) Hz, 1H), 7.45-7.41 (m, 1H), 7.37-7.32 (m, 1H), 5.04 (d, \( J = 2.5 \) Hz, 2H), 3.33 (tt, \( J = 11.5, 3.2 \) Hz, 1H), 2.28 (t, \( J = 2.5 \) Hz, 1H), 1.96 (d, \( J = 11.1 \) Hz, 2H), 1.86 (dt, \( J = 12.6, 2.9 \) Hz, 2H), 1.79-1.73 (m, 1H), 1.57 (m, 2H), 1.45 (m, 2H), 1.31 (m, 1H). \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}) \( \delta \) 164.30, 153.62, 133.21, 131.47, 130.05, 129.60, 123.90, 114.07, 73.15, 40.96, 31.58, 30.66, 26.42, 26.26.

3-cyclohexyl-1-propylquinoxalin-2(1H)-one (29)\textsuperscript{17}

Following the general procedure A, 29 was obtained in 63\% yield (17 mg).
Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.81 (dd, \( J = 8.0, 1.1 \) Hz, 1H), 7.49-7.42 (m, 1H), 7.30-7.23 (m, 2H), 4.21-4.15 (m, 2H), 3.31 (tt, \( J = 11.5, 3.2 \) Hz, 1H), 1.93 (d, \( J = 11.6 \) Hz, 2H), 1.88-1.81 (m, 2H), 1.76 (m, 3H), 1.55 (m, 2H), 1.49-1.38 (m, 2H), 1.28 (m, 1H), 1.02 (t, \( J = 7.4 \) Hz, 3H). \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}) \( \delta \) 164.40, 154.37, 133.28, 132.16, 130.13, 129.37, 123.27, 113.60, 43.91, 40.86, 30.66, 26.47, 26.30, 20.78, 11.54.

1-butyl-3-cyclohexylquinoxalin-2(1H)-one (30)

Following the general procedure A, 30 was obtained in 85\% yield (24 mg).
Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \( \delta \) 7.83-7.79 (m, 1H), 7.49-7.43 (m, 1H), 7.29-7.23 (m, 2H), 4.24-4.18 (m, 2H), 3.31 (tt, \( J = 11.5, 3.1 \) Hz, 1H), 1.93 (d, \( J = 12.7 \) Hz, 2H), 1.87-1.81 (m, 2H), 1.77-1.68 (m, 3H), 1.60-1.47 (m, 3H), 1.44 (m, 3H), 1.33-1.23 (m, 1H), 0.98 (m, 3H). \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}) \( \delta \) 164.35, 154.33, 133.29, 132.13, 130.11, 129.37, 123.25, 113.56, 42.22, 40.84, 30.65, 29.45, 26.46, 26.29, 20.43, 13.89. HR-MS (ESI)[M+H]\textsuperscript{+} m/z calcd for C\textsubscript{18}H\textsubscript{25}N 285.1691, found 285.1953.
3-cyclohexyl-1-pentylquinoxalin-2(1H)-one (31)

Following the general procedure A, 31 was obtained in 81% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.81 (dd, $J = 7.9, 1.2$ Hz, 1H), 7.49-7.43 (m, 1H), 7.30-7.23 (m, 2H), 4.23-4.17 (m, 2H), 3.32 (tt, $J = 11.5, 3.2$ Hz, 1H), 1.94 (d, $J = 11.3$ Hz, 2H), 1.88-1.81 (m, 2H), 1.73 (m, 3H), 1.56 (m, 2H), 1.51-1.43 (m, 2H), 1.40 (m, 4H), 1.30 (m, 1H), 0.90 (t, $J = 6.9$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 164.35, 154.29, 133.28, 132.13, 130.10, 129.36, 123.22, 113.54, 42.43, 40.83, 30.64, 29.25, 27.07, 26.45, 26.28, 22.49, 14.08. HR-MS (ESI)[M+H]$^+$ m/z calcd for C$_{19}$H$_{27}$N$_2$O 299.2118, found 299.2114.

3-cyclohexyl-1-(3-hydroxypropyl)quinoxalin-2(1H)-one (32)

Following the general procedure A, 32 was obtained in 80% yield (23 mg). Eluent (petroleum ether: ethyl acetate, 5:1). White solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.87 (d, $J = 8.0$ Hz, 1H), 7.52 (t, $J = 7.8$ Hz, 1H), 7.36 (d, $J = 8.0$ Hz, 2H), 4.44 (t, $J = 6.1$ Hz, 2H), 3.68 (t, $J = 6.8$ Hz, 1H), 3.52 (q, $J = 5.9$ Hz, 2H), 3.32 (m, 1H), 2.05-1.99 (m, 2H), 1.98-1.92 (m, 2H), 1.86 (d, $J = 12.6$ Hz, 2H), 1.76 (m, 1H), 1.56 (m, 2H), 1.51-1.40 (m, 2H), 1.35-1.27 (m, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 163.89, 155.35, 134.39, 133.28, 131.64, 130.32, 129.77, 123.95, 113.69, 58.23, 50.10, 40.88, 39.00, 30.61, 30.16, 26.41, 26.25. HR-MS (ESI)[M+H]$^+$ m/z calcd for C$_{17}$H$_{23}$N$_2$O$_2$ 287.1754, found 287.1757.

1-methyl-3-(pentan-3-yl)quinoxalin-2(1H)-one (33)

Following the general procedure B, 33 was obtained in 61% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.87-7.83 (m, 1H), 7.53-7.49 (m, 1H), 7.35-7.28 (m, 2H), 3.70 (s, 3H), 3.35 (m, 1H), 1.87 (m, 2H), 1.73-1.67 (m, 2H), 0.88 (t, $J = 7.4$ Hz, 6H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 163.99, 155.23, 132.98, 132.97, 129.98, 129.56, 123.49, 113.59, 44.79, 29.26, 25.89, 12.12.
3-cyclobutyl-1-methylquinoxalin-2(1H)-one (34)\(^{18}\)

Following the general procedure B, 34 was obtained in 81% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.86 (dd, \(J = 8.0, 1.4\) Hz, 1H), 7.50 (m, 1H), 7.34-7.30 (m, 1H), 7.28-7.25 (m, 1H), 4.10-4.01 (m, 1H), 3.67 (s, 3H), 2.47-2.40 (m, 2H), 2.37 (m, 2H), 2.10 (m, 1H), 1.89 (m, 1H). \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 162.38, 154.71, 133.11, 132.98, 129.88, 129.50, 123.55, 113.59, 38.48, 28.97, 26.45, 18.37.

3-cyclopentyl-1-methylquinoxalin-2(1H)-one (35)\(^{16}\)

Following the general procedure B, 35 was obtained in 92% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.81 (dd, \(J = 8.0, 1.3\) Hz, 1H), 7.51-7.46 (m, 1H), 7.33-7.26 (m, 2H), 3.74-3.69 (m, 1H), 3.69 (s, 3H), 2.06 (m, 2H), 1.96-1.88 (m, 2H), 1.84-1.78 (m, 2H). \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 163.85, 155.11, 133.10, 132.85, 129.87, 129.41, 123.48, 113.53, 42.85, 30.97, 29.14, 26.07.

1-methyl-3-(tetrahydrofuran-2-yl)quinoxalin-2(1H)-one (36)\(^{19}\)

Following the general procedure B, 36 was obtained in 91% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.94 (dd, \(J = 8.0, 1.4\) Hz, 1H), 7.53 (ddd, \(J = 8.6, 7.4, 1.5\) Hz, 1H), 7.35-7.28 (m, 2H), 5.37 (m, 1H), 4.25-4.19 (m, 1H), 4.03-3.97 (m, 1H), 3.68 (s, 3H), 2.53-2.45 (m, 1H), 2.06-2.00 (m, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 159.55, 154.17, 133.26, 132.60, 130.55, 130.26, 123.76, 113.64, 77.71, 69.25, 30.59, 28.93, 25.74.

3-(cyclohex-3-en-1-yl)-1-methylquinoxalin-2(1H)-one (37)\(^{20}\)

Following the general procedure B, 37 was obtained in 91% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \(^1\)H NMR (400 MHz, Chloroform-d) \(\delta\) 7.83 (dd, \(J = 8.0, 1.4\) Hz, 1H), 7.51 (ddd, \(J = 8.6, 7.4, 1.5\) Hz, 1H), 7.35-7.27 (m, 2H), 5.83-5.73 (m, 2H), 3.70 (s, 3H), 3.58 (m, 1H), 2.10 (m, 1H), 1.89 (m, 1H).
2.46 (m, 1H), 2.34-2.16 (m, 3H), 2.08-2.01 (m, 1H), 1.78 (m, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 163.80, 154.72, 133.00, 132.94, 129.95, 129.65, 126.65, 126.43, 123.57, 113.61, 37.00, 29.19, 28.98, 26.98, 25.72.

3-cyclohexyl-1,6,7-trimethylquinoxalin-2(1H)-one (38)$^{16}$

Following the general procedure A, 38 was obtained in 79% yield (21 mg).

Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.59 (s, 1H), 7.03 (s, 1H), 3.66 (s, 3H), 3.31 (tt, $J$ = 11.5, 3.2 Hz, 1H), 2.39 (s, 3H), 2.33 (s, 3H), 1.93-1.70 (m, 5H), 1.57-1.43 (m, 4H), 1.29 (m, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 163.14, 154.74, 139.09, 132.31, 131.40, 130.93, 130.01, 114.18, 40.76, 30.70, 29.08, 26.48, 26.31, 20.57, 19.21.

6-bromo-3-cyclohexyl-1-methylquinoxalin-2(1H)-one (39)

Following the general procedure A, 39 was obtained in 75% yield (24 mg).

Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.66 (d, $J$ = 8.7 Hz, 1H), 7.41 (m, 2H), 3.64 (s, 3H), 3.30 (tt, $J$ = 11.5, 3.2 Hz, 1H), 1.95-1.83 (m, 4H), 1.76 (d, $J$ = 12.9 Hz, 1H), 1.53 (m, 2H), 1.44 (m, 2H), 1.29 (m, 1H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 164.79, 154.29, 134.06, 131.87, 131.12, 126.70, 123.38, 116.61, 40.98, 30.58, 29.30, 26.39, 26.26. HR-MS (ESI)[M+H]$^+$ m/z calcd for C$_{15}$H$_{18}$N$_2$OBr 321.0597, found 321.0589.

3-cyclohexyl-7-fluoro-1-methylquinoxalin-2(1H)-one (40)

Following the general procedure A, 40 was obtained in 92% yield (24 mg).

Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.51 (d, $J$ = 8.9 Hz, 1H), 7.22 (m, 2H), 3.67 (d, $J$ = 3.0 Hz, 3H), 3.32 (m, 1H), 1.93 (d, $J$ = 12.0 Hz, 2H), 1.88-1.81 (m, 2H), 1.78-1.72 (m, 1H), 1.55-1.41 (m, 4H), 1.29 (m, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 165.95, 158.74 (d, $J$ = 242.9 Hz), 154.28, 133.61 (d, $J$ = 11.3 Hz), 129.63, 117.09 (d, $J$ = 23.8 Hz), 115.33 (d, $J$ = 22.3 Hz), 114.58 (d, $J$ = 8.8 Hz), 40.98, 30.60, 29.42, 26.37, 26.24. HR-MS (ESI)[M+H]$^+$ m/z calcd for C$_{15}$H$_{18}$N$_2$OF 261.1398, found 261.1390.
7-chloro-3-cyclohexyl-1-methylquinoxalin-2(1H)-one (41)\textsuperscript{17}

Following the general procedure A, 41 was obtained in 87% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl\textsubscript{3}) $\delta$ 7.82 (s, 1H), 7.44 (dd, $J = 8.9$, 2.4 Hz, 1H), 7.19 (d, $J = 8.9$ Hz, 1H), 3.67 (s, 3H), 3.32 (m, 1H), 1.96-1.90 (m, 2H), 1.89-1.82 (m, 2H), 1.76 (m, 1H), 1.50 (d, $J = 8.9$ Hz, 2H), 1.48-1.39 (m, 2H), 1.30 (m, 1H). $^{13}$C NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 165.84, 154.32, 133.55, 131.69, 129.43, 129.28, 128.80, 114.70, 40.95, 30.60, 29.36, 26.37, 26.24.

7-bromo-3-cyclohexyl-1-methylquinoxalin-2(1H)-one (42)

Following the general procedure A, 42 was obtained in 75% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl\textsubscript{3}) $\delta$ 7.99 (m, 1H), 7.63-7.53 (m, 1H), 7.17-7.12 (m, 1H), 3.66 (s, 3H), 3.36-3.31 (m, 1H), 1.93 (d, $J = 11.9$ Hz, 2H), 1.86 (d, $J = 12.3$ Hz, 2H), 1.76 (d, $J = 12.9$ Hz, 1H), 1.50 (m, 4H), 1.32-1.26 (m, 1H). $^{13}$C NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 165.84, 154.34, 133.89, 132.36, 132.21, 132.16, 116.08, 115.03, 40.96, 30.64, 29.35, 26.39, 26.26. HR-MS (ESI)[M+H]$^+$ m/z calcd for C\textsubscript{15}H\textsubscript{18}N\textsubscript{2}OBr 321.0597, found 321.0592.

3-\textit{(tert-butyl)-1-methylquinoxalin-2(1H)-one} (43)\textsuperscript{16}

Following the general procedure A, 43 was obtained in 65% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl\textsubscript{3}) $\delta$ 7.83 (dd, $J = 8.0$, 1.3 Hz, 1H), 7.52-7.47 (m, 1H), 7.31 (m, 1H), 7.26 (dd, $J = 8.4$, 1.0 Hz, 1H), 3.67 (s, 3H), 1.49 (s, 9H). $^{13}$C NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 165.41, 153.86, 133.45, 132.31, 130.24, 129.63, 123.29, 113.37, 39.60, 28.88, 28.01.

1-methyl-3-\textit{(tert-pentyl)quinoxalin-2(1H)-one} (44)\textsuperscript{21}

Following the general procedure A, 44 was obtained in 97% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. $^1$H NMR (400 MHz, CDCl\textsubscript{3}) $\delta$ 7.84 (dd, $J = 8.0$, 1.4 Hz, 1H), 7.50 (m, 1H), 7.33-7.29 (m, 1H), 7.28-7.25 (m, 1H), 3.67 (s, 3H), 2.04 (q, $J = 7.5$ Hz, 2H), 1.43 (s, 6H), 0.75 (t, $J =$
7.5 Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 164.88, 153.89, 133.36, 132.36, 130.27, 129.59, 123.26, 113.38, 43.21, 32.47, 28.89, 25.98, 9.66.

\(3-((3r,5r,7r)-adamantan-1-yl)-1\text{-}methyl\text{-}1\text{-}H\text{-}quinoxalin\text{-}2\text{-}one\) (45)

Following the general procedure A, 45 was obtained in 71% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.82 (dd, \(J = 8.0, 1.3\) Hz, 1H), 7.52-7.46 (m, 1H), 7.33-7.28 (m, 1H), 7.26 (m, 1H), 3.66 (s, 3H), 2.24 (d, \(J = 2.7\) Hz, 6H), 2.11 (s, 3H), 1.82 (m, 6H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 164.88, 153.76, 133.16, 132.54, 130.21, 129.59, 123.09, 42.09, 38.95, 37.14, 28.78, 28.69.

\(1\text{-}methyl\text{-}3\text{-}(1\text{-}phenylcyclopropyl)quinoxalin\text{-}2\text{-}one\) (46)

Following the general procedure A, 46 was obtained in 73% yield (20 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.89 (dd, \(J = 8.0, 1.3\) Hz, 1H), 7.51 (m, 1H), 7.48-7.45 (m, 2H), 7.35-7.31 (m, 1H), 7.28-7.24 (m, 3H), 7.20-7.15 (m, 1H), 3.61 (s, 3H), 1.50-1.47 (m, 2H), 1.38-1.35 (m, 2H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 160.49, 154.55, 141.98, 133.72, 132.59, 130.21, 130.08, 128.66, 128.26, 126.56, 123.55, 113.58, 30.83, 29.10, 13.81. HR-MS (ESI)[M+H]\(^+\) m/z calcd for C\(_{18}\)H\(_{17}\)N\(_2\) 277.1335, found 277.1330.

\(6\text{-}octylphenanthridine\) (47)

Following the general procedure A, 47 was obtained in 72% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.63 (d, \(J = 8.2\) Hz, 1H), 8.55-8.52 (m, 1H), 8.25 (d, \(J = 8.1\) Hz, 1H), 8.13 (dd, \(J = 8.2, 1.1\) Hz, 1H), 7.82 (m, 1H), 7.73-7.66 (m, 2H), 7.61 (m, 1H), 3.39-3.34 (m, 2H), 1.92 (m, 2H), 1.58-1.36 (m, 5H), 1.31 (m, 5H), 0.89 (t, \(J = 6.9\) Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 162.63, 143.88, 133.05, 130.35, 129.68, 128.67, 127.31, 126.48, 126.35, 125.34, 123.75, 122.58, 122.01, 36.67, 31.96, 30.13, 29.84, 29.38, 22.80, 14.24. HR-MS (ESI)[M+H]\(^+\) m/z calcd for C\(_{21}\)H\(_{26}\)N 292.2060, found 292.2043.
6-cyclohexylphenanthridine (48)\(^9\)

Following the general procedure A, 48 was obtained in 93% yield (24 mg). Eluent (petroleum ether: ethyl acetate, 20:1). Colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.66-8.63 (m, 1H), 8.54 (dd, \(J = 8.2, 1.3\) Hz, 1H), 8.34-8.30 (m, 1H), 8.16 (dd, \(J = 8.2, 1.0\) Hz, 1H), 7.81 (m, 1H), 7.70 (m, 2H), 7.61 (m, 1H), 3.63 (tt, \(J = 11.2, 3.2\) Hz, 1H), 2.13-2.07 (m, 2H), 2.02-1.94 (m, 4H), 1.90-1.83 (m, 1H), 1.59 (m, 2H), 1.45 (qt, \(J = 12.9, 3.3\) Hz, 1H). \(^1\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 165.38, 144.00, 133.11, 130.05, 129.48, 127.16, 126.22, 125.71, 124.83, 123.45, 122.68, 121.92, 42.11, 32.42, 27.01, 26.45.

6-(\textit{tert}-pentyl)phenanthridine (49)\(^{22}\)

Following the general procedure A, 49 was obtained in 68% yield (17 mg). Eluent (petroleum ether: ethyl acetate, 20:1). Pale yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.70 (d, \(J = 8.0\) Hz, 1H), 8.64 (d, \(J = 8.4\) Hz, 1H), 8.55-8.51 (m, 1H), 8.14 (dd, \(J = 8.0, 1.2\) Hz, 1H), 7.79 (m, 1H), 7.73-7.68 (m, 1H), 7.66-7.59 (m, 2H), 2.23 (m), 1.70 (d, \(J = 3.6\) Hz, 6H), 0.75 (m, 3H). \(^1\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 165.82, 143.10, 133.92, 130.48, 129.37, 128.42, 127.77, 126.52, 126.16, 124.92, 123.44, 123.08, 121.72, 44.14, 35.65, 29.37, 9.69.

3-cyclohexyl-2\(\textit{H}\)-chromen-2-one (50)\(^{10}\)

Following the general procedure A, 50 was obtained in 61% yield (14 mg). Eluent (petroleum ether: ethyl acetate, 5:1). Pale yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.48-7.42 (m, 3H), 7.30 (d, \(J = 8.1\) Hz, 1H), 7.24 (m, 1H), 2.78 (m, 1H), 2.03-1.93 (m, 2H), 1.90-1.74 (m, 3H), 1.45 (qt, \(J = 12.7, 3.2\) Hz, 2H), 1.28 (m, 3H). \(^1\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 161.66, 152.83, 136.42, 135.00, 130.53, 127.41, 124.27, 119.79, 116.41, 38.31, 32.24, 26.63, 26.29.

2-(\textit{tert}-butyl)quinoxaline (51)\(^{23}\)

Following the general procedure A, 51 was obtained in 65% yield (12 mg). Eluent (petroleum ether: ethyl acetate, 10:1). Pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.99 (s, 1H), 8.09-8.03 (m, 2H), 7.75-7.67 (m, 2H), 1.51 (s, 9H). \(^1\)C NMR
(101 MHz, CDCl₃) δ 163.83, 143.57, 141.75, 140.90, 129.78, 129.42, 129.02, 37.39, 29.89.

**4-methyl-2-(3-phenylpropyl)quinoline 1-oxide (52)**

Following the general procedure C, 52 was obtained in 76% yield (21 mg). Eluent (petroleum ether: ethyl acetate, 2:1). Pale yellow solid.

$^1$H NMR (400 MHz, CDCl₃) δ 8.81 (d, J = 8.7, 1.2 Hz, 1H), 7.90 (d, J = 8.3, 1.3 Hz, 1H), 7.73 (m, 1H), 7.60 (m, 1H), 7.27 (d, J = 7.7 Hz, 1H), 7.25-7.20 (m, 3H), 7.18-7.13 (m, 1H), 7.13 (d, J = 8.7 Hz, 1H), 7.06 (s, 1H), 3.16-3.10 (m, 2H), 2.78 (t, J = 7.7 Hz, 2H), 2.60 (s, 3H), 2.15 (m, 2H). $^{13}$C NMR (101 MHz, CDCl₃) δ 148.24, 141.85, 141.31, 133.56, 130.12, 128.69, 128.62, 128.50, 127.65, 126.05, 124.67, 122.65, 120.39, 35.97, 31.29, 27.89, 18.43. HR-MS (ESI)[M+H]+ m/z calcd for C₁₉H₂₀NO 278.1539, found 278.1544.

**2-cyclohexyl-4-methylquinoline 1-oxide (53)**

Following the general procedure C, 53 was obtained in 80% yield (19 mg). Eluent (petroleum ether: ethyl acetate, 4:1). Colorless oil. $^1$H NMR (400 MHz, CDCl₃) δ 8.84 (d, J = 8.7 Hz, 1H), 7.90 (d, J = 8.3 Hz, 1H), 7.73 (m, 1H), 7.61-7.56 (m, 1H), 7.13 (s, 1H), 3.83 (m, 1H), 2.63 (s, 3H), 2.08 (d, J = 11.7 Hz, 2H), 1.84 (m, 3H), 1.55 (m, 2H), 1.46-1.29 (m, 3H). $^{13}$C NMR (101 MHz, CDCl₃) δ 152.37, 141.19, 133.79, 130.06, 128.27, 127.53, 124.53, 120.64, 120.06, 37.85, 30.71, 26.51, 26.40, 18.61.

**4-methyl-2-(tert-pentyl)quinoline 1-oxide (54)**

Following the general procedure C, 54 was obtained in 85% yield (19 mg). Eluent (petroleum ether: ethyl acetate, 4:1). Colorless oil. $^1$H NMR (400 MHz, CDCl₃) δ 8.84 (d, J = 8.8 Hz, 1H), 7.91 (d, J = 8.3 Hz, 1H), 7.72 (t, J = 7.8 Hz, 1H), 7.60 (m, 1H), 7.22 (s, 1H), 2.64 (s, 3H), 2.31-2.21 (m, 2H), 1.59-1.52 (m, 6H), 0.70-0.63 (m, 3H). $^{13}$C NMR (101 MHz, CDCl₃) δ 152.53, 142.55, 132.73, 129.99, 128.68, 127.70, 124.47, 121.92, 120.55, 40.62, 30.48, 25.96, 18.75, 9.89.
2-((3r,5r,7r)-adamantan-1-yl)-4-methylquinoline 1-oxide (55)²⁴

Following the general procedure C, 55 was obtained in 76% yield (22 mg). Eluent (petroleum ether: ethyl acetate, 2:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 8.85 (d, J = 8.8 Hz, 1H), 7.91 (d, J = 8.2 Hz, 1H), 7.74 (t, J = 7.8 Hz, 1H), 7.61 (t, J = 7.5 Hz, 1H), 7.22 (s, 1H), 2.64 (m, 6H), 2.44 (m, 6H), 2.15 (s, 3H), 1.90-1.78 (m, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 153.12, 142.84, 133.27, 130.08, 128.45, 127.67, 124.46, 120.79, 120.52, 39.02, 37.27, 37.06, 28.70, 18.79.

3-(3-cyclohexyl-2-oxoquinoxalin-1(2H)-yl)propyl 2-(4-isobutylphenyl)propanoate (56)

Following the general procedure A, 56 was obtained in 71% yield (34 mg). Eluent (petroleum ether: ethyl acetate, 3:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (dd, J = 7.9, 1.4 Hz, 1H), 7.39-7.34 (m, 1H), 7.30-7.23 (m, 3H), 7.10 (d, J = 8.0 Hz, 2H), 7.01 (d, J = 8.0 Hz, 1H), 4.23 (m, 1H), 4.19-4.15 (m, 2H), 3.73 (q, J = 7.1 Hz, 1H), 3.31 (t, J = 11.5, 3.1 Hz, 1H), 2.41 (d, J = 7.2 Hz, 2H), 2.04 (m, 2H), 1.93 (d, J = 12.6 Hz, 2H), 1.89-1.83 (m, 2H), 1.80-1.74 (m, 2H), 1.58 (d, J = 12.3 Hz, 1H), 1.51 (d, J = 7.2 Hz, 3H), 1.43 (m, 2H), 1.36-1.20 (m, 2H), 0.83 (dd, J = 6.6, 1.5 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 174.65, 164.24, 154.27, 140.81, 137.80, 133.17, 131.94, 130.12, 129.57, 129.52, 127.27, 123.40, 113.25, 62.29, 45.26, 45.10, 40.75, 39.47, 30.63, 30.26, 26.54, 26.42, 26.24, 22.43, 18.43. HR-MS(ESI)[M+H]^+ m/z calcd for C₃₀H₃₉N₂O₃ 475.2955, found 475.2930.

3-((3R)-3-((3R,5R,8R,9S,10S,13R,14S)-3-hydroxy-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)butyl)-1-methylquinoxalin-2(1H)-one (57)

Following the general procedure B, 57 was obtained in 78% yield (38 mg). Eluent (petroleum ether: ethyl acetate, 2:1). White solid. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (dd, J = 8.0, 1.3 Hz, 1H), 7.53-7.47 (m, 1H), 7.34-7.30 (m, 1H), 7.28 (d, J = 8.4 Hz, 1H), 3.69 (s, 3H), 3.61 (m, 1H), 3.00 (m, 1H), 2.80 (m, 1H), 2.02-1.96 (m, 1H), 1.94-1.83
(m, 3H), 1.75 (m, 3H), 1.69-1.63 (m, 2H), 1.58-1.53 (m, 2H), 1.52-1.45 (m, 2H), 1.36 (m, 7H), 1.25-1.15 (m, 4H), 1.04 (d, J = 6.3 Hz, 6H), 0.91 (s, 3H), 0.65 (s, 3H). ^13^C NMR (101 MHz, CDCl₃) δ 162.03, 155.01, 133.19, 132.88, 129.68, 129.53, 123.60, 113.64, 71.97, 56.61, 42.89, 42.24, 40.56, 40.30, 36.59, 36.04, 35.99, 35.49, 34.70, 33.02, 31.37, 30.69, 29.15, 28.38, 27.34, 26.56, 24.38, 23.52, 20.96, 18.75, 12.20. HR-MS (ESI)[M+H]^+ m/z calcd for C₃₂H₄₇N₂O₂ 491.3632, found 491.3622.

(3^R,5^R,8^R,9^S,10^S,13^R,14^S)-17-((3^R)-4-(isoquinolin-1-yl)butan-2-yl)-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-3-ol (58)²⁵

Following the general procedure A, 58 was obtained in 74% yield (34 mg). Eluent (petroleum ether: ethyl acetate, 2:1). White crystal. ^1^H NMR (400 MHz, CDCl₃) δ 8.41 (d, J = 5.7 Hz, 1H), 8.13 (d, J = 8.3 Hz, 1H), 7.80 (d, J = 8.1 Hz, 1H), 7.62 (m, 2H), 7.48 (d, J = 5.7 Hz, 1H), 3.62 (m, 1H), 3.38 (td, J = 12.5, 11.7, 3.8 Hz, 1H), 3.19-3.09 (m, 1H), 2.01 (d, J = 10.6 Hz, 1H), 1.98-1.82 (m, 4H), 1.77 (s, 2H), 1.59 (m, 6H), 1.38 (m, 6H), 1.29-1.19 (m, 5H), 1.13 (s, 3H), 1.08-0.97 (m, 3H), 0.91 (s, 3H), 0.67 (s, 3H). ^13^C NMR (101 MHz, CDCl₃) δ 163.13, 141.99, 136.42, 129.89, 127.53, 127.08, 126.97, 125.46, 119.23, 71.93, 56.61, 56.16, 42.91, 42.23, 40.55, 40.33, 36.59, 36.56, 36.33, 35.97, 35.49, 34.70, 32.72, 30.69, 28.50, 27.33, 26.55, 24.38, 23.52, 20.97, 18.93, 12.19.
XII. References

XIII. The $^1$H and $^{13}$C NMR spectra of compounds 3-58.

$^1$H NMR for 3 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 3 (101 MHz, CDCl$_3$)
$^1$H NMR for 4 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 4 (101 MHz, CDCl$_3$)
$^1$H NMR for 5 (400 MHz, CDCl₃)

$^{13}$C NMR for 5 (101 MHz, CDCl₃)
$^1$H NMR for 6 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 6 (101 MHz, CDCl$_3$)
$^1$H NMR for 7 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 7 (101 MHz, CDCl$_3$)
$^1$H NMR for 8 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 8 (101 MHz, CDCl$_3$)
$^1$H NMR for 9 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 9 (101 MHz, CDCl$_3$)
$^1$H NMR for 10 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 10 (101 MHz, CDCl$_3$)
$^1$H NMR for 11 (500 MHz, CDCl$_3$)

$^{13}$C NMR for 11 (126 MHz, CDCl$_3$)
$^1$H NMR for 12 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 12 (101 MHz, CDCl$_3$)
$^1$H NMR for 13 (500 MHz, CDCl$_3$)

$^{13}$C NMR for 13 (126 MHz, CDCl$_3$)

550
$^1$H NMR for 15 (500 MHz, CDCl$_3$)

$^{13}$C NMR for 15 (126 MHz, CDCl$_3$)
$^1$H NMR for 16 (500 MHz, CDCl$_3$)

$^{13}$C NMR for 16 (126 MHz, CDCl$_3$)
$^1$H NMR for 17 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 17 (101 MHz, CDCl$_3$)
$^1$H NMR for 18 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 18 (101 MHz, CDCl$_3$)

S55
$^1$H NMR for 19 (400 MHz, CDCl₃)

$^{13}$C NMR for 19 (101 MHz, CDCl₃)
$^1$H NMR for 20 (400 MHz, DMSO-$d_6$)

$^{13}$C NMR for 20 (126 MHz, CDCl$_3$)
$^1$H NMR for 21 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 21 (101 MHz, CDCl$_3$)
$^1$H NMR for 22 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 22 (101 MHz, CDCl$_3$)
$^1$H NMR for 23 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 23 (101 MHz, CDCl$_3$)
$^1$H NMR for 24 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 24 (101 MHz, CDCl$_3$)
$^1$H NMR for 25 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 25 (101 MHz, CDCl$_3$)
$^1$H NMR for 26 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 26 (101 MHz, CDCl$_3$)

563
$^1$H NMR for 27 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 27 (101 MHz, CDCl$_3$)
$^1$H NMR for 28 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 28 (101 MHz, CDCl$_3$)
$^1$H NMR for 29 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 29 (101 MHz, CDCl$_3$)
$^1$H NMR for 30 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 30 (101 MHz, CDCl$_3$)
$^1$H NMR for 31 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 31 (101 MHz, CDCl$_3$)
$^1$H NMR for 32 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 32 (101 MHz, CDCl$_3$)
$^1$H NMR for 33 (500 MHz, CDCl$_3$)

$^{13}$C NMR for 33 (126 MHz, CDCl$_3$)
\(^1\)H NMR for 34 (500 MHz, CDCl\(_3\))

\(^{13}\)C NMR for 34 (126 MHz, CDCl\(_3\))
$^1$H NMR for 35 (500 MHz, CDCl$_3$)

$^{13}$C NMR for 35 (126 MHz, CDCl$_3$)
$^1$H NMR for 36 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 36 (101 MHz, CDCl$_3$)
$^1$H NMR for 37 (400 MHz, CDCl$_3$)

![NMR Spectrogram](image)

$^{13}$C NMR for 37 (101 MHz, CDCl$_3$)

![NMR Spectrogram](image)
$^1$H NMR for 38 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 38 (101 MHz, CDCl$_3$)
$^1$H NMR for 39 (500 MHz, CDCl$_3$)

$^{13}$C NMR for 39 (126 MHz, CDCl$_3$)
$^1$H NMR for 40 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 40 (101 MHz, CDCl$_3$)
$^1$H NMR for 41 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 41 (101 MHz, CDCl$_3$)
$^1$H NMR for 42 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 42 (101 MHz, CDCl$_3$)
$^1$H NMR for 44 (400 MHz, CDCl₃)

$^{13}$C NMR for 44 (101 MHz, CDCl₃)
$^1$H NMR for 45 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 45 (101 MHz, CDCl$_3$)
$^{1}H$ NMR for 46 (400 MHz, CDCl$_3$)

$^{13}C$ NMR for 46 (101 MHz, CDCl$_3$)
$^1$H NMR for 47 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 47 (101 MHz, CDCl$_3$)
$^1$H NMR for 48 (400 MHz, CDCl₃)

13C NMR for 48 (101 MHz, CDCl₃)
$^1$H NMR for 49 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 49 (101 MHz, CDCl$_3$)

49
$^1$H NMR for 50 (400 MHz, CDCl$_3$)

$^1$C NMR for 50 (101 MHz, CDCl$_3$)
$^1$H NMR for 51 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 51 (101 MHz, CDCl$_3$)
$^1$H NMR for 52 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 52 (101 MHz, CDCl$_3$)
$^1$H NMR for 53 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 53 (101 MHz, CDCl$_3$)

53
$^1$H NMR for 54 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 54 (101 MHz, CDCl$_3$)
**$^1$H NMR for 55 (400 MHz, CDCl$_3$)**

![NMR Spectrum](image1)

**$^{13}$C NMR for 55 (101 MHz, CDCl$_3$)**

![NMR Spectrum](image2)
$^1$H NMR for 56 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 56 (101 MHz, CDCl$_3$)
$^1$H NMR for 57 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 57 (101 MHz, CDCl$_3$)
$^1$H NMR for 58 (400 MHz, CDCl$_3$)

$^{13}$C NMR for 58 (101 MHz, CDCl$_3$)