Supporting Information

Nickel-catalyzed Electrochemical Reductive Decarboxylative Coupling of

N-Hydroxyphthalimide Esters with Quinoxalinones

Fei Lian,^a Kun Xu,^{a,b*} Wei Meng,^a Haonan Zhang,^a Zhoumei Tan,^a and Chengchu Zeng^{a,*}

^{a.}Beijing Key Laboratory of Environmental and Viral Oncology, College of Life Science &

Bioengineering, Beijing University of Technology, Beijing 100124, China. ^{b.}College of Chemistry

and Pharmaceutical Engineering, Nanyang Normal University, Henan 473061, China.

kunxu@bjut.edu.cn; zengcc@bjut.edu.cn

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1. General information

Unless otherwise special indicated, all the reagents were purchased from commercial supplies unless otherwise stated. And all the solvents were used without any purification. Thin-layer chromatography (TLC) was performed on plastic plates coated with silica gel GF254 with 0.2 mm thickness (Yantai Yuanbo Biological Technology Co., Ltd.) and all compounds were visualized with a UV light at 254 nm. Flash column chromatography was performed using silica gel (200-300 mesh, Yantai Yuanbo Biological Technology Co., Ltd.). NMR spectra were recorded on a Bruker Avance III spectrometer operating at 400 MHz (¹H NMR) and 100 MHz (¹³C NMR). Chemical shifts were reported in ppm downfield and referenced as follows: ¹H: residual internal CHCl₃ (δ 7.26 ppm); ¹³C: internal CDCl₃ (δ 77.2 ppm). Coupling constants were quoted in Hz(*J*). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet).

2. General procedure for the synthesis of starting materials (1b-1m)¹



To a stirred solution of 2-quinoxalinone (5 mmol) in DMF (20 mL) was added the corresponding halide (1.6 equiv.) potassium and carbonate (1.2 equiv.) at room temperature overnight. Then resulting mixture was transferred to a separatory funnel. Ethanol and water were added to the reaction mixture, and the aqueous layer was extracted twice with ethyl acetate. The combined organic layers were washed with a saturated solution of NH₄Cl and then with brine, dried over Na₂SO₄, filtered and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel to obtain product (**1b-1j, 1m**).



To ethanol (20 ml) suspension solution of *o*-arylenediamine (5 mmol) was added Ethyl 2-oxoacetate (1.1 equiv.). The reaction system was stirred and heated to reflux at 85 °C for 1 h, then stirred at room temperature for 16 h. After the reaction was completed (as monitored by TLC), the precipitate was filtered and washed with ethanol (5 ml*3), and finally dried to give quinoxalinone (**1d-1n**).

3. General procedure for the synthesis of starting materials $(2a-2q)^2$

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The corresponding carboxylic acids or N-protected amino acids (12 mmol, 1.2 *N*-hydroxyphthalimide equiv), (1.63 g,10 mmol, 1.0 equiv.), and 4dimethylaminopyridine (61 mg, 0.5 mmol, 5 mol %) were mixed in a flask, 30 mL CH₂Cl₂ was added. Then a solution of N, N'-dicyclohexylcarbodiimide (2.47 g, 12mmol, 1.2 equiv) in CH₂Cl₂ (10 mL) was added slowly at room temperature. The reaction mixture was maintained at room temperature with stirring for 1-3 h, during which time the reaction mixture became cloudy and a white solid precipitated from the solution. The white solid was removed via vacuum filtration and the filtrate was removed under reduced pressure. The residue was purified by flash column chromatography to give corresponding redox active esters.

4. General procedure for the synthesis of 3ae



An undivided cell was equipped with a graphite felt anode (10 mm × 10 mm × 1 mm) and a Ni foam cathode (10 mm × 10 mm × 1 mm) and connected to a DC regulated power supply. To the cell was added 2-quinoxalinone **1a** (1.0 equiv., 0.3 mmol), NHP ester **2e** (2.0 equiv., 0.6 mmol), LiClO₄ (1.0 mmol), NiCl₂·6H₂O (0.2equiv., 0.06 mmol), 4,4'-di-*tert*-butyl-2,2'-bipyridine (0.2equiv., 0.06 mmol). Seal the device tightly and

introduce argon into the tube (three times). Then, anhydrous *N*, *N*-dimethylacetamide (DMA, 4.0 mL) and triethylamine (0.25 mL) was added via a syringe under argon atmosphere, and insert the argon-filled balloon into the bottle with a rubber stopper. The mixture was first reacted at 60 °C for 30 minutes under magnetic stirring, then the mixture was electrolyzed under constant current conditions (8 mA) for 3 hours. After the reaction was completed, the mixture was quenched with water and extracted with ethyl acetate (3 x 10 mL). The organic layers were combined and concentrated under vacuo. The desired product **3ae** was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate).

5. Optimization of the reaction conditions

Table S1. Optimization of Reaction Conditions ^a

		1a 2	2e		3ae		
Entry	Electrolyte	Catalyst	Ligand	I/mA	Anode/Cathode	Yield(%) ^b	
1 ^c	LiClO ₄	NiBr ₂ •glyme	dtbpy	4	C felt/Ni foam	26	
2	LiCIO ₄	NiBr ₂ •glyme	dtbpy	4	C felt/Ni foam	38	
3	LiClO ₄	NiBr ₂ •glyme	dtbpy	6	C felt/Ni foam	82	
4	LiClO ₄	NiBr ₂ •glyme	dtbpy	8	C felt/Ni foam	85	
5	LiClO ₄	NiBr ₂ •.glyme	dtbpy	8	C felt/ C felt	82	
6	LiClO ₄	NiBr ₂ •glyme	dtbpy	8	C felt/Pt	13	
7	LiClO ₄	NiBr ₂ •glyme	dtbpy	8	Mg/Ni foam	14	
8	LiClO ₄	NiBr ₂	dtbpy	8	C felt/Ni foam	72	
9	LiClO ₄	Ni(acac) ₂	dtbpy	8	C felt/Ni foam	84	
10	LiClO ₄	NiCl ₂ •glyme	dtbpy	8	C felt/Ni foam	37	
11	LiClO ₄	$NiCl_2(pph_3)_2$	dtbpy	8	C felt/Ni foam	73	
12	LiClO ₄	NiCl ₂ (dppe)	dtbpy	8	C felt/Ni foam	73	
13	LiClO ₄	NiCl ₂ (Cy) ₂	dtbpy	8	C felt/Ni foam	80	
14	LiClO ₄	NiCl ₂ ·6H ₂ O	dtbpy	8	C felt/Ni foam	87	
15	LiClO ₄	NiCl ₂ •6H ₂ O	dtbpy	8	C felt/Ni plate	48	
16	Bu4NBF4	NiCl ₂ •6H ₂ O	dtbpy	8	C felt/Ni foam	58	
17	Bu_4NPF_6	NiCl ₂ •6H ₂ O	dtbpy	8	C felt/Ni foam	49	
18	Bu ₄ NClO ₄	NiCl ₂ •6H ₂ O	dtbpy	8	C felt/Ni foam	67	



19 ^d	LiClO ₄	NiCl ₂ •6H ₂ O	1,10-phen	8	C felt/Ni foam	70
20 ^e	LiClO ₄	NiCl ₂ •6H ₂ O	dmbpy	8	C felt/Ni foam	71
21	LiClO ₄	-	dtbpy	8	C felt/Ni foam	27
22 ^f	LiClO ₄	NiCl ₂ •6H ₂ O	dtbpy	0	-	21

^a Reaction conditions: 1a (0.3 mmol), 2e (0.6 mmol), LiClO₄ (1.0 mmol), NiCl₂·6H₂O (0.06 mmol), 4,4'-di-*tert*-butyl-2,2'-bipyridine (dtbpy, 0.06 mmol), triethylamine (0.25 mL), anhydrous DMA (4 mL), graphite felt (10 mm × 10 mm × 1 mm) as the anode, nickel foam (10 mm × 10 mm × 1 mm) as the cathode, undivided cell, 8 mA, 60 °C, 3 h, Ar. ^b Isolated yield. ^c CH₃CN as the solvent. ^d 1,10-phen = 1,10-phenanthrolinium. ^e dmbpy = 4,4'-dimethyl-2,2'-bipyridyl.
 ^f The reaction was carried out at 120 °C for 6h without electrolysis.

6. Gram-Scale Experiment:



Reaction conditions: **1a** (3 mmol), **2e** (6 mmol), LiClO₄ (2.0 mmol), NiCl₂·6H₂O (0.6 mmol), 4,4'-di*tert*-butyl-2,2'-bipyridine (dtbpy, 0.6 mmol), triethylamine (2.5 mL), anhydrous DMA (20 mL), graphite felt (20 mm × 40 mm × 1 mm) as the anode, nickel foam (20 mm × 40 mm × 1 mm) as the cathode, undivided cell, 40 mA, 60 °C, 10 h, Ar. ^b Isolated yield.

An undivided cell was equipped with a graphite felt anode (20 mm × 40 mm × 1 mm) and a Ni foam cathode (20 mm × 40 mm × 1 mm) and connected to a DC regulated power supply. To the cell was added 2-quinoxalinone **1a** (1.0 equiv., 3 mmol), NHP ester **2e** (2.0 equiv., 6 mmol), LiClO₄ (2.0 mmol), NiCl₂·6H₂O (0.2 equiv., 0. 6 mmol), 4,4'-di-*tert*-butyl-2,2'-bipyridine (0.2 equiv., 0. 6 mmol). Seal the device tightly and introduce argon into the tube (three times). Then, anhydrous *N*, *N*-dimethylacetamide (DMA, 20.0 mL) and triethylamine (2.5 mL) was added via a syringe under argon atmosphere, and insert the argon-filled balloon into the bottle with a rubber stopper. The mixture was first reacted at 60 °C for 30 minutes under magnetic stirring, then the mixture was completed, the mixture was quenched with water and extracted with ethyl acetate (3 x 50 mL). The organic layers were combined and concentrated under vacuo. The desired product **3ae** was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate) to give 68% yield.

7. Cyclic voltammograms



Figure 1. Cyclic voltammograms of 0.1 M LiClO₄ and related compounds in anhydrous DMA (5 mL) using glassy carbon working electrode, Pt wire, and Ag/AgNO₃ as counter and reference electrode at 100 mV/s scan rate, (a) 0.1 M LiClO₄ in DMA (b) NiCl₂· $6H_2O$ (5.0 mmol/L) + dtbpy (5.0 mmol/L), (c) **2e** (5.0 mmol/L).

7. Characterization data of 3aa-3aq

3-(4-methylbenzyl)quinoxalin-2(1H)-one (3aa)



Following the general procedure, the desired compound was obtained as white solid, mp 195-196 °C, 26.1 mg, 35% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.37 (s, 1H), 7.73 (d, J = 7.8 Hz, 1H), 7.48 (t, J = 7.6 Hz, 1H), 7.30-7.27 (m, 2H), 7.25-7.22 (m, 2H), 7.09 (d, J = 7.8 Hz, 2H), 4.09 (s, 2H), 2.25 (s, 3H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 160.9, 155.0, 135.8, 134.8, 132.4, 132.1, 130.1, 129.5, 129.3, 128.7, 123.6, 115.7, 39.0, 21.1.

HRMS (ESI) m/z calculated for $C_{16}H_{15}N_2O^+$ 251.1179, Found 251.1171.

3-phenethylquinoxalin-2(1H)-one (3ab)



Following the general procedure, the desired compound was obtained as white solid, mp 210-211 °C, 53.2 mg, 71% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 7.73 (d, J = 7.9 Hz, 1H), 7.47 (t,

J = 7.6 Hz, 1H), 7.30-7.25 (m, 6H), 7.19-7.16 (m, 1H), 3.09-3.07 (m, 2H), 3.05-3.03 (m, 2H). ¹³C NMR (100 MHz, *d*₆-DMSO) δ 166.1, 159.8, 146.7, 137.0, 136.8, 134.7, 133.6, 133.5, 133.3, 131.1, 128.3, 120.5, 39.71, 36.93.

HRMS (ESI) m/z calculated for $C_{16}H_{15}N_2O^+$ 251.1179, Found 251.1170.

3-(3-phenylpropyl)quinoxalin-2(1H)-one (3ac)



Following the general procedure, the title compound was obtained as white solid, mp 166-167 °C, 70.7 mg, 89% yield. ¹H NMR (400 MHz, CDCl₃) δ 12.44 (s, 1H), 7.82 (d, J = 7.9 Hz,

1H), 7.48 (t, J = 7.2 Hz, 1H), 7.33 (t, J = 8.0 Hz, 2H), 7.29-7.25 (m, 4H), 7.19-7.16 (m, 1H), 3.06-3.02 (t, J = 8.0 Hz, 2H), 2.83-2.79 (t, J = 8.0 Hz, 2H), 2.24-2.16 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 161.3, 156.7, 142.1, 132.9, 131.0, 129.7, 128.8, 128.6, 128.3, 125.8, 124.1, 115.7, 35.7, 33.0, 28.3.

HRMS (ESI) m/z calculated for $C_{17}H_{17}N_2O^+$ 265.1335, Found 265.1329.

3-*iso*pentylquinoxalin-2(1*H*)-one (3ad)



Following the general procedure, the desired compound was obtained as white solid, mp 165-167 °C, 40.6 mg, 63% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.30 (s, 1H), 7.71 (d, J = 7.9 Hz, 1H),

7.47 (t, *J* = 7.5 Hz, 1H), 7.29-7.25 (m, 2H), 2.81-2.77 (m, 3H), 1.65-1.58 (m, 3H), 0.94 (d, *J* = 6.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 162.6, 155.06, 132.2, 132.1, 129.8, 128.5, 123.5, 115.6, 35.6, 31.2, 28.0, 22.9.

HRMS (ESI) m/z calculated for $C_{13}H_{17}N_2O^+$ 217.1335, Found 217.1330.

3-cyclohexylquinoxalin-2(1*H*)-one (3ae)



Following the general procedure, the desired compound was obtained as white solid, mp 177-178 °C, 59.6 mg, 87% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.24 (s, 1H), 7.69 (d, J = 7.7 Hz, 1H), 7.45 (t, J = 7.2 Hz, 1H), 7.28-7.23 (m, 2H), 3.17 (t, J = 10.3 Hz, 1H),

1.93-1.65 (m, 5H), 1.55-1.29 (m, 4H), 1.29-1.16 (m, 1H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 165.2, 154.6, 132.1, 132.0, 129.8, 128.6, 123.4, 115.6, 39.9, 30.5, 26.3, 26.2.

HRMS (ESI) m/z calculated for $C_{14}H_{17}N_2O^+$ 229.1335, Found 229.1326.

3-(4-*iso*propylcyclohexyl)quinoxalin-2(1H)-one (3af)



Following the general procedure, the desired compound was obtained as white solid, mp 220-221 °C, 68.1 mg, 84% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.28 (d, J = 8.3 Hz, 1H), 7.71 (t, J = 7.2 Hz, 1H), 7.46 (t, J = 7.5 Hz, 1H), 7.28-

7.24 (m, 2H), 3.11 (t, J = 12.0 Hz, 1H), 1.93 (d, J = 12.7 Hz,

2H), 1.91-1.73 (m, 3H), 1.67-1.63 (m, 2H), 1.51-1.46 (m, 3H), 1.23-1.12 (m, 1H), 0.89-0.87 (m, 6H). ¹³C NMR (100 MHz, *d*₆-DMSO) δ 163.4, 154.6, 132.1, 132.0, 130.1, 128.8, 123.6, 115.7, 37.43, 33.42, 33.17, 32.95, 26.72, 26.62 (d, *J* = 7.9 Hz).

HRMS (ESI) m/z calculated for $C_{17}H_{23}N_2O^+ 271.1805$, Found 271.1802.

3-(4,4-difluorocyclohexyl)quinoxalin-2(1*H*)-one (3ag)



Following the general procedure, the desired compound was obtained as White solid, mp 214-215 °C, 58.6 mg, 74% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.39 (s, 1H), 7.73 (d, J = 7.6 Hz, 1H), 7.49 (t, J = 7.4 Hz, 1H), 7.30-7.27 (m, 2H), 2.15-2.06

(m, 2H), 2.01-1.91 (m, 2H), 1.80-1.71 (m, 2H).¹³C NMR (100 MHz, *d*₆-DMSO) δ 163.4, 154.6, 132.1, 132.0, 130.1, 128.8, 123.6, 115.7, 37.43, 33.42, 33.17, 32.95, 26.72, 26.62 (d, *J* = 7.9 Hz).

HRMS (ESI) m/z calculated for $C_{14}H_{15}N_2F_2O^+$ 265.1147, Found 265.1141.

3-(tetrahydro-2*H*-pyran-4-yl)quinoxalin-2(1*H*)-one (3ah)



Following the general procedure, the desired compound was obtained as white solid, mp 215-216 °C, 47.0mg, 68% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.36 (s, 1H), 7.73 (d, J = 7.8 Hz, 1H), 7.49 (t, J = 7.4 Hz, 1H), 7.33-7.26 (m, 2H), 3.98-3.95 (m, 2H),

3.50-3.45 (m, 2H), 1.82-1.71 (m, 4H),1.30-1.16 (m, 1H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 163.1, 154.1, 131.5, 129.5, 128.2, 123.0, 115.1, 66.9, 37.0, 29.7.

HRMS (ESI) m/z calculated for $C_{13}H_{15}N_2O^+ 231.1128$, Found 231.1117.

3-cyclopentylquinoxalin-2(1*H*)-one (3ai)



Following the general procedure, the desired compound was obtained as white solid, mp 202-203 °C, 58.4 mg, 91% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.29 (s, 1H), 7.70 (d, J = 7.9 Hz, 1H), 7.46 (m, 1H), 7.27 (m, 2H), 3.60-3.56 (m, 1H), 2.00-1.92

(m, 2H), 1.88-1.79 (m, 2H), 1.77-1.68 (m, 2H), 1.66-1.62 (m, 2H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 164.8, 155.1, 132.1, 131.9, 129.7, 128.6, 123.4, 115.6, 41.8, 30.7, 25.9. HRMS (ESI) m/z calculated for C₁₃H₁₅N₂O⁺ 215.1179, Found 215.1175.

3-cyclobutylquinoxalin-2(1H)-one (3aj)



Following the general procedure, the desired compound was obtained as white solid, mp 194-196 °C, 54.0 mg, 90% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.25 (s, 1H), 7.75 (d, J = 7.9 Hz, 1H), 7.49-7.45 (m, 1H), 7.29-7.26 (m, 2H), 3.96-3.88 (m, 1H), 2.36-2.23

(m, 4H), 2.09-2.00 (m, 1H), 1.88-1.79 (m, 1H). 13 C NMR (100 MHz, d_6 -DMSO) δ 163.4, 154.7, 132.2, 132.1, 129.8, 128.6, 123.5, 115.6, 37.5, 26.2, 18.1.

HRMS (ESI) m/z calculated for $C_{12}H_{13}N_2O^+$ 201.1022, Found 201.1018.

3-(*tert*-pentyl)quinoxalin-2(1*H*)-one (3ak)



Following the general procedure, the desired compound was obtained as white solid, mp 137-138 °C, 47.7 mg, 74% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.19 (s, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 7.7 Hz, 1H), 7.26 (m, 2H), 1.97 (d, J = 7.5 Hz, 2H),

1.35 (s, 6H), 0.67 (t, J = 7.5 Hz, 3H). ¹³C NMR (100 MHz, d₆-DMSO) δ 165.7, 154.2, 132.4, 131.5, 120.0, 128.9, 123.4, 115.3, 42.7, 32.0, 26.0, 9.8.

HRMS (ESI) m/z calculated for $C_{13}H_{17}N_2O^+ 217.1335$, Found 217.1334.

3-(*tert*-butyl)quinoxalin-2(1*H*)-one (3al)



Following the general procedure, the desired compound was obtained as white solid, mp 172-173 °C, 24.5 mg, 40% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.22 (s, 1H), 7.72 (d, J = 7.5 Hz, 1H), 7.50-7.42 (m, 1H), 7.30-7.25 (m, 2H), 1.42 (s, 8H). ¹³C NMR (100

MHz, d_6 -DMSO) δ 166.2, 154.1, 132.5, 131.4, 130.0, 128.9, 123.4, 115.3, 39.2, 28.0. HRMS (ESI) m/z calculated for C₁₂H₁₅N₂O⁺ 203.1179, Found 203.1170.

3-((3r,5r,7r)-adamantan-1-yl)quinoxalin-2(1H)-one (3am)



Following the general procedure, the desired compound was obtained as white solid, mp 237-238 °C, 45.5mg, 54% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.18 (s, 1H), 7.70 (d, J = 7.9 Hz, 1H),

7.46 (t, J = 7.6 Hz, 1H), 7.27-7.24 (m, 2H), 2.16 (s, 6H), 2.06 (s, 3H), 1.75 (s, 6H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 165.7, 154.1, 132.2, 131.6, 130.0, 128.9, 123.4, 115.2, 41.5, 38.7, 37.0, 28.3.

HRMS (ESI) m/z calculated for $C_{18}H_{21}N_2O^+$ 281.1648, Found 281.1640.

3-(1-tosylpiperidin-4-yl)quinoxalin-2(1H)-one (3an)



Following the general procedure, the desired compound was obtained as white solid, mp 280-281 °C, 80.1 mg, 70% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.34 (s, 1H), 7.66 (t, J = 8.0 Hz, 4H), 7.46 (d, J = 7.5 Hz, 3H), 7.27 (d, J = 7.8 Hz,

2H), 3.74 (d, *J* = 10.9 Hz, 3H), 3.07 (d, *J* = 10.6 Hz, 1H), 2.41-

2.35 (m, 5H), 1.96 (d, J = 12.3 Hz, 2H), 1.75-1.69 (m, 2H). ¹³C NMR (100 MHz, d₆-DMSO)
δ 163.2, 154.5, 143.9, 133.2, 132.1, 131.9, 130.3, 130.1, 128.7, 127.9, 123.6, 115.68,
46.4, 37.3, 28.8, 21.5.

HRMS (ESI) m/z calculated for C₂₀H₂₂N₃O₃S⁺ 384.1376, Found 384.1383.

(3ao)



Following the general procedure, the desired compound was obtained as white solid, mp 216-217 °C, 52.5 mg, 51% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.30 (s, 1H), 7.73 (d, J = 7.9 Hz, 1H), 7.50 (t, J = 7.6 Hz, 1H), 7.31-7.27 (m,

2H), 3.53-3.50 (m, 2H), 3.24 (s, 2H), 2.48-2.44 (m, 2H), 1.67-1.62 (m, 2H), 1.40-1.23 (m, 11H).¹³C NMR (100 MHz, *d*₆-DMSO) δ 164.1, 154.5, 154.2, 132.3, 131.5, 130.3, 129.0, 123.5, 115.3, 78.8, 40.9, 34.7, 28.6, 24.1.

HRMS (ESI) m/z calculated for $C_{19}H_{26}N_3O_3Na^+366.1788$, Found 366.1785.

tert-butyl 2-((tert-butoxycarbonyl)amino)-4-(3-oxo-3,4-dihydroquinoxalin-2-

yl)butanoate (3ap)



Following the general procedure, the desired compound was obtained as white solid, mp 128-129 °C, 84.0 mg, 70% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.59 (s, 1H), 7.82 (d, J = 8.0 Hz, 1H), 7.49 (t, J = 7.5 Hz, 1H), 7.38 (d, J = 7.8 Hz, 1H), 7.33 (t, J = 7.6 Hz, 1H),

5.54 (d, J = 7.8 Hz, 1H), 4.38 (d, J = 4.6 Hz, 1H), 3.08 (s, 2H), 2.33 (t, J = 21.2 Hz, 1H), 2.25-2.18 (m, 1H), 1.49-1.43 (m, 18H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 171.9, 160.2,

156.5, 155.6, 132.7, 131.0, 129.8, 128.7, 124.1, 115.7, 81.9, 79.5, 53.9, 29.2, 28.3, 28.0. HRMS (ESI) m/z calculated for $C_{21}H_{30}N_3O_5^+$ 404.2180, Found 404.2175.

tert-butyl (1-(3-oxo-3,4-dihydroquinoxalin-2-yl)ethyl)carbamate (3aq)



Following the general procedure, the desired compound was obtained as white solid, mp 194-196 °C, 78.1mg, 90% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.44 (s, 1H), 7.74 (d, J = 7.9 Hz, 1H), 7.52 (t, J = 7.6 Hz, 1H), 7.33- 7.28 (m,

2H), 5.00-4.97 (m, 1H), 1.38-1.33 (m, 12H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 161.9, 155.4, 154.3, 132.4, 131.8, 130.3, 128.6, 123.7, 115.8, 78.3, 47.7, 28. 7, 19.0. HRMS (ESI) m/z calculated for C₁₅H₂₀N₃O₃⁺ 290.1499, Found 290.1495.

8. Characterization data of 3ba-3ma

6-bromo-3-cyclohexylquinoxalin-2(1H)-one (3ba)



Following the general procedure, the desired compound was obtained as white solid, mp 224-225 °C, 47.3 mg, 51% yield. ¹H NMR (400 MHz, d_6 -DMSO) 12.42 (s, 1H), 7.88 (d, J = 2.0 Hz, 1H), 7.63 (dd, J = 8.7, 2.0 Hz, 1H), 7.22 (d, J = 8.7)

Hz, 1H), 3.17 (t, J = 11.0 Hz, 1H), 1.83 (dd, J = 21.0, 11.7 Hz, 3H), 1.72 (d, J = 12.6 Hz, 1H), 1.50-1.26 (m, 5H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 166.8, 154.4, 133.1, 132.4, 131.3, 130.6, 117.6, 114.8, 30.4, 26.23, 26.18.

HRMS (ESI) m/z calculated for $C_{14}H_{16}BrN_2O^+$ 307.0441, Found 307.0439.

3-cyclohexyl-2-oxo-1,2-dihydroquinoxaline-6-carbonitrile (3ca)



Following the general procedure, the desired compound was obtained as white solid, mp 225-226 °C, 47.0 mg, 62% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.55 (s, 1H), 7.85 (d, J = 8.3 Hz, 1H), 7.64 (dd, J = 8.3, 1.4 Hz, 1H), 7.59 (s, 1H),

3.22-3.16 (m, 1H), 1.88 (d, J = 11.5 Hz, 2H), 1.81 (d, J = 12.1 Hz, 2H), 1.72 (d, J = 12.6 Hz, 1H), 1.55-1.32 (m, 5H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 168.9, 154.2, 134.4, 132.4, 129.8, 126.3, 119.7, 118.8, 111.4, 30.3, 26.8, 26.1.

HRMS (ESI) m/z calculated for $C_{15}H_{16}N_3O^+254.1288$, Found 254.1284.

3-cyclohexyl-5-methylquinoxalin-2(1H)-one (3da)



Following the general procedure, the desired compound was obtained as white solid, mp 221-223 °C, 46.8 mg, 65% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.21 (s, 1H), 7.34 (t, J = 7.8 Hz, 1H), 7.11 (dd, J = 12.9, 7.7 Hz, 2H), 3.16 (t, J = 10.8 Hz, 1H), 1.90

(d, *J* = 11.9 Hz, 2H), 1.82 (d, *J* = 12.7 Hz, 2H), 1.72 (d, *J* = 12.3 Hz, 1H), 1.53-1.34 (m, 5H). ¹³C NMR (100 MHz, *d*₆-DMSO) δ 163.5, 154.6, 136.9, 132.0, 130.6, 129.6, 124.4, 113.4, 30.6, 26.3, 17.2.

HRMS (ESI) m/z calculated for $C_{15}H_{19}N_2O^+$ 243.1492, Found 243.1486.

3-cyclohexyl-6-methylquinoxalin-2(1H)-one (3ea) and 3-cyclohexyl-7-

methylquinoxalin-2(1H)-one (3ea')



Following the general procedure, the desired compound was obtained as white solid, mp 212-213 °C, 66.6 mg, 92% yield. ¹H NMR (400 MHz, d_6 -DMSO)

δ 12.21 (s, 1H), 7.56 (d, J = 8.0 Hz, 0.5H), 7.50 (s, 0.5H), 7.26 (d, J = 8.2 Hz, 0.5H), 7.15 (d, J = 8.2 Hz, 0.5H), 7.05 (d, J = 9.2 Hz, 1H), 3.14 (dd, J = 7.0, 3.2 Hz, 1H), 2.37 (s, 1.5H), 2.34 (s, 1.5H), 1.86-1.77 (m, 4H), 1.69 (d, J = 12.2 Hz, 1H), 1.47-1.28 (m, 4H), 1.26-1.18 (m, 1H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 165.1, 163.9, 154.8, 154.6, 139.8, 132.7, 132.1, 131.9, 130.8, 130.4, 129.7, 128.4, 124.7, 115.3, 115.2, 39.9, 30.5, 26.3, 26.2, 21.6, 20.8. HRMS (ESI) m/z calculated for C₁₅H₁₉N₂O⁺ 243.1492, Found 243.1484.

6-benzoyl-3-cyclohexylquinoxalin-2(1*H*)-one (3fa)



Following the general procedure, the desired compound was obtained as white solid, mp 243-244 °C, 30.8 mg, 32% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.41 (s, 1H), 7.85 (d, J = 8.1 Hz, 1H), 7.77 (d, J = 7.4 Hz, 2H), 7.73-7.69 (m,

1H), 7.66 (s, 1H), 7.59 (t, *J* = 7.0 Hz, 3H), 3.21 (t, *J* = 10.5 Hz, 1H), 1.89 (d, *J* = 11.4 Hz, 2H), 1.82 (d, *J* = 11.1 Hz, 2H), 1.72 (d, *J* = 11.7 Hz, 1H), 1.41 (m, 5H).

¹³C NMR (100 MHz, *d*₆-DMSO) δ 195.4, 168.0, 154.5, 137.4, 137.3, 134.4, 133.4, 131.8, 130.1, 129.1, 128.8, 124.3, 117.5, 30.5, 26.23.

HRMS (ESI) m/z calculated for $C_{21}H_{21}N_2O_2^+$ 333.1598, Found 333.1602.

7-benzoyl-3-cyclohexylquinoxalin-2(1H)-one (3fa')



Following the general procedure, the desired compound was obtained as white solid, mp 267-268 °C, 28.0 mg, 29% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.64 (s, 1H), 7.96 (s, 1H), 7.92 (d, J = 8.5 Hz, 1H), 7.75 (d, J = 7.6 Hz, 2H), 7.69 (d, J = 7.3 Hz, 1H), 7.59 (t, J = 7.6 Hz, 2H), 7.41 (d, J =

8.5 Hz, 1H), 3.17 (t, J = 11.0 Hz, 1H), 1.88 (d, J = 11.8 Hz, 2H), 1.80 (d, J = 12.1 Hz, 2H), 1.71 (d, J = 12.9 Hz, 1H), 1.46-1.30 (m, 6H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 194.9, 166.7, 154.7, 137.8, 135.6, 132.9, 132.0, 131.14, 131.01, 130.7, 129.8, 129.1, 116.1, 30.4, 26.24.

HRMS (ESI) m/z calculated for $C_{21}H_{21}N_2O_2^+$ 333.1598, Found 333.1598.

3-cyclohexyl-6,7-dimethylquinoxalin-2(1H)-one (3ga)



Following the general procedure, the desired compound was obtained as white solid, mp 233-234 °C, 70.8 mg, 92% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.03 (s, 1H), 7.41 (s, 1H), 6.96 (s, 1H), 3.07 (t, J = 10.5 Hz, 2H), 2.22 (s, 2H), 2.20 (s, 2H), 1.76

(t, *J* = 14.7 Hz, 4H), 1.64 (d, *J* = 12.1 Hz, 1H), 1.42-1.16 (m, 5H). ¹³C NMR (100 MHz, *d*₆-DMSO) δ 163.9, 154.7, 139.1, 132.0, 130.6, 129.9, 128.6, 115.6, 30.6, 26.3, 26.3, 20.1, 19.3.

HRMS (ESI) m/z calculated for $C_{16}H_{21}N_2O^+$ 257.1648, Found 257.1648.

3-cyclohexyl-6,7-difluoroquinoxalin-2(1H)-one (3ha)



Following the general procedure, the desired compound was obtained as white solid, mp 220-221 °C,48.0 mg, 61% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.42 (s, 1H), 7.79 (dd, J = 11.0, 8.3 Hz, 1H), 7.18 (dd, J = 11.0, 7.7 Hz, 1H), 3.15 (dd,

 $J = 15.0, 6.8 \text{ Hz}, 1\text{H}, 1.83 \text{ (dd}, J = 20.9, 11.3 \text{ Hz}, 4\text{H}), 1.72 \text{ (d}, J = 12.4 \text{ Hz}, 1\text{H}), 1.47-1.30 \text{ (m, 4H)}, 1.27-1.22 \text{ (m, 1H)}. {}^{13}\text{C} \text{ NMR} (100 \text{ MHz}, d_6-\text{DMSO}) \delta 166.0, 154.3, 150.3 \text{ (q}, J = 245.0 \text{ Hz}), 146.0 \text{ (q}, J = 241.0 \text{ Hz}, 14.0 \text{ Hz}), 129.3 \text{ (d}, J = 9.9 \text{ Hz}), 128.5 \text{ (d}, J = 7.1 \text{ Hz}), 116.4 \text{ (d}, J = 17.9 \text{ Hz}), 103.4 \text{ (d}, J = 21.8 \text{ Hz}), 40.0, 30.4, 26.22, 26.18. \text{HRMS} (ESI) m/z calculated for <math>C_{14}H_{15}F_2N_2O^+265.1147$, Found 265.1141.

6,7-dichloro-3-cyclohexylquinoxalin-2(1H)-one (3ia)



Following the general procedure, the desired compound was obtained as white solid, mp 263-264 °C, 58.8 mg, 66% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.49 (s, 1H), 7.91 (s, 1H), 7.41 (s, 1H), 3.15 (s, 1H), 1.85-1.80 (m, 4H), 1.73 (d, J =

11.3 Hz, 1H), 1.44-1.35 (m, 3H), 1.30-1.22 (m, 2H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 167.2, 154.2, 131.9, 131.8, 131.6, 129.6, 125.2, 116.7, 40.1, 30.4, 26.2.

HRMS (ESI) m/z calculated for $C_{14}H_{14}Cl_2N_2O^+$ 297.0556, Found 297.0553.

3-cyclohexyl-1-methylquinoxalin-2(1H)-one (3ja)



Following the general procedure, the desired compound was obtained as white solid, mp 99-100 °C, 48.0 mg, 66% yield. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 7.9 Hz, 1H), 7.50 (t, *J* = 7.8 Hz, 1H), 7.33-7.26 (m, 2H), 3.37-3.31 (m, 1H), 1.96 (d, *J* = 12.2

Hz, 2H), 1.87 (d, J = 12.1 Hz, 2H), 1.77 (d, J = 11.5 Hz, 1H), 1.62-1.42 (m, 5H), 1.35-126 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 164.3, 154.5, 132.9, 132.9, 129.8, 129.4, 123.4, 113.4, 40.8, 30.5, 29.0, 26.3, 26.2.

HRMS (ESI) m/z calculated for $C_{15}H_{19}N_2O^+$ 243.1492, Found 243.1489.

1-benzyl-3-cyclohexylquinoxalin-2(1H)-one (3ka)



Following the general procedure, the desired compound was obtained as white solid, mp 134-136 °C, 70.6 mg, 74% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 7.32 (d, J = 7.7 Hz, 1H), 7.01 (t, J = 7.6 Hz, 1H), 6.95 (d, J = 8.1 Hz, 1H), 6.87-6.84 (m, 3H), 6.81-6.75 (m, 3H), 5.05 (s, 2H), 2.81-2.80 (m, 1H), 1.50 (t, J = 13.0 Hz, 2H),

1.37 (d, J = 12.5 Hz, 2H), 1.27 (d, J = 12.2 Hz, 1H), 1.10-0.88 (m, 4H), 0.84-0.78 (m, 1H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 163.5, 153.8, 136.0, 132.3, 131.9, 129.7, 129.2, 128.7, 127.3, 126.8, 123.4, 114.9, 44.9, 30.2, 25.9, 25.8.

HRMS (ESI) m/z calculated for C₂₁H₂₃N₂O⁺ 319.1805, Found 319.1804.

Methyl 2-(3-cyclohexyl-2-oxoquinoxalin-1(2H)-yl)acetate (3la)



Following the general procedure, the desired compound was obtained as white solid, mp 110-112 °C, 51.3 mg, 57% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 7.80 (d, J = 7.8 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.49 (d, J = 8.2 Hz, 1H), 7.36 (t, J = 7.4 Hz, 1H), 5.11 (s, 2H), 3.72 (s, 3H), 3.19 (t, J = 11.1 Hz, 1H), 1.88 (d, J = 11.7 Hz,

2H), 1.81 (d, *J* = 12.0 Hz, 2H), 1.71 (d, *J* = 11.9 Hz, 1H), 1.52-1.33 (m, 4H), 1.28-1.12 (m, 1H). ¹³C NMR (100 MHz, *d*₆-DMSO) *δ* 168.5, 163.5, 153.9, 132.5, 130.3, 129.7, 124.1, 114.8, 52.9, 44.1, 30.5, 26.3, 26.2.

HRMS (ESI) m/z calculated for $C_{17}H_{21}N_2O_3^+$ 301.1547, Found 301.1541.

3-cyclohexylpyrido[3,4-b]pyrazin-2(1H)-one (3ma)



Following the general procedure, the desired compound was obtained as white solid, mp 240-241 °C, 20.9 mg, 30% yield. ¹H NMR (400 MHz, d_6 -DMSO) δ 12.54 (s, 1H), 8.85 (s, 1H), 8.44 (d, J = 5.3 Hz, 1H), 7.18 (d, J = 5.3 Hz, 1H), 3.16 (t, J = 11.0 Hz, 1H),

1.87 (d, J = 11.7 Hz, 2H), 1.81 (d, J = 11.7 Hz, 2H), 1.71 (d, J = 11.7 Hz, 1H), 1.40 (m, 5H). ¹³C NMR (100 MHz, d_6 -DMSO) δ 167.3, 154.9, 150.1, 148.8, 137.5, 128.6, 110.0, 40.1, 30.4, 26.21, 26.17.

HRMS (ESI) m/z calculated for $C_{13}H_{16}N_3O^+ 230.1288$, Found 230.1285.

9. Reference

- 1 G.-Y. Dou, Y.-Y. Jiang, K. Xu and C.-C. Zeng, *Org. Chem. Front.*, **2019**, *6*, 2392-2397.
- C. Zheng, Y.-T. Wang, Y.-R. Xu, Z. Chen, G.-Y.Chen, and S.-H. Liang, Org. Lett. 2018, 20, 4824-4827.

10. Copies of NMR spectra for prepared compounds





¹H NMR of 3-phenethylquinoxalin-2(1*H*)-one (3ab)

D0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppa)



¹H NMR of 3-(3-phenylpropyl)quinoxalin-2(1*H*)-one (3ac)



S24

¹H NMR of 3-cyclohexylquinoxalin-2(1*H*)-one (3ae) -12.24











¹H NMR of 3-(4,4-difluorocyclohexyl)quinoxalin-2(1*H*)-one (3ag)



¹H NMR of 3-(tetrahydro-2*H*-pyran-4-yl)quinoxalin-2(1H)-one (3ah)











¹H NMR of 3-(*tert*-butyl)quinoxalin-2(1*H*)-one (3al)



S33



S34

¹H NMR of *tert*-butyl 4-methyl-4-(3-oxo-3,4-dihydroquinoxalin-2-yl)piperidine-1carboxylate (3ao)



¹³C NMR of *tert*-butyl 4-methyl-4-(3-oxo-3,4-dihydroquinoxalin-2-yl)piperidine-1carboxylate (3ao)



¹H NMR of *t*ert-butyl 2-((tert-butoxycarbonyl)amino)-4-(3-oxo-3,4dihydroquinoxalin-2-yl)butanoate (3ap)









-])0 100 90 fl (ppm)



¹H NMR of 3-cyclohexyl-2-oxo-1,2-dihydroquinoxaline-6-carbonitrile (3ca)

¹H NMR of 3-cyclohexyl-5-methylquinoxalin-2(1*H*)-one (3da)



¹H NMR of 3-cyclohexyl-6-methylquinoxalin-2(1*H*)-one (3ea) and 3-cyclohexyl-7methylquinoxalin-2(1*H*)-one (3ea')



¹³C NMR of 3-cyclohexyl-6-methylquinoxalin-2(1*H*)-one(3ea) and 3-cyclohexyl-7-

methylquinoxalin-2(1H)-one (3ea')





¹H NMR of 6-benzoyl-3-cyclohexylquinoxalin-2(1*H*)-one (3fa)









¹³C NMR of 3-cyclohexyl-6,7-dimethylquinoxalin-2(1*H*)-one (3ga)





¹H NMR of 3-cyclohexyl-6,7-difluoroquinoxalin-2(1*H*)-one (3ha)

100 90 f1 (ppm) 70 60 50

80

110

130 120

40

20

10 0

30

-]

00

190

180 170 160 150 140





¹³C NMR of 6,7-dichloro-3-cyclohexylquinoxalin-2(1*H*)-one (3ia)





¹H NMR of 3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one (3ja)



¹H NMR of 1-benzyl-3-cyclohexylquinoxalin-2(1*H*)-one (3ka)



¹H NMR of methyl 2-(3-cyclohexyl-2-oxoquinoxalin-1(2*H*)-yl)acetate (3la)



S50