

## ***Supporting Information***

### **Preparation of OHCP nanofibers photocatalyst for cross-dehydrogenation coupling reactions**

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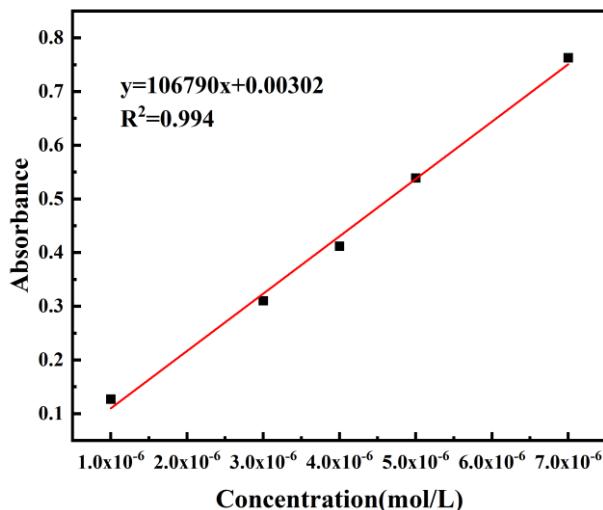
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## 1. Preparation of OHCP spinning precursors

**Table S1. Composition of different spinning precursors.**

Entry	PVA(g)	PDADMAC(g)	PHB (mg)	Deionized water(mL)
1	0.98	0.02	0	10
2	0.98	0.02	20	10
3	0.98	0.02	40	10
4	0.98	0.02	60	10
5	0.98	0.02	80	10
6	0.95	0.05	0	10
7	0.95	0.05	20	10
8	0.95	0.05	40	10
9	0.95	0.05	60	10
10	0.95	0.05	80	10
11	0.90	0.10	0	10
12	0.90	0.10	20	10
13	0.90	0.10	40	10
14	0.90	0.10	60	10
15	0.90	0.10	80	10

Taking sample 8 in Table S1 as an example, the spinning precursor was prepared as follows: PVA (0.95 g), PDADMAC (0.05 g) and deionized water were added to the sample vials and stirred magnetically for 3 hours at 95°C. The PVA solution was then allowed to cool to room temperature before being allowed to cool to room temperature. The PVA solution was then allowed to cool to room temperature. Add 2 mL of PHB-water solution (20 mg/mL) dropwise to the above PVA solution and bring the total amount of deionized water in the mixed solution to 10 ml. The OHCP spinning precursor was obtained by magnetic stirring for 6 hours at room temperature.



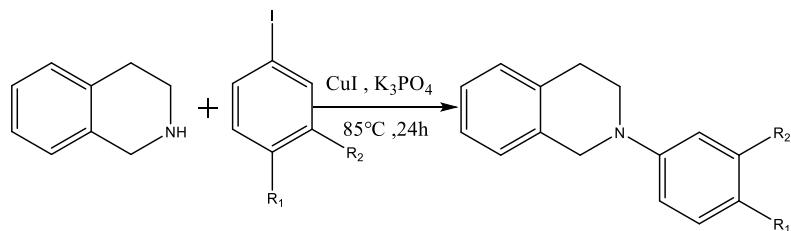
**Fig. S1** Standard curve for PHB-ethanol solution

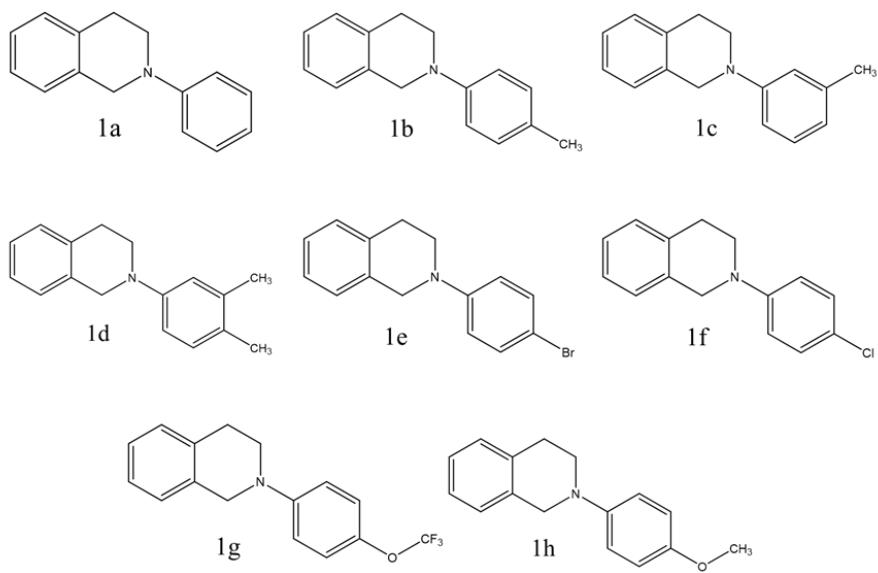
## 2. Synthesis of N-phenyl-tetrahydroisoquinoline derivatives

### 2.1 Synthesis of N-phenyl-tetrahydroisoquinoline derivatives

Copper (I) iodide (200 mg, 1.0 mmol) and potassium phosphate (4.25 g, 20.0 mmol) were put into a 50 mL three-neck flask. The three-neck flask was evacuated and back filled with argon. 2-Propanol (10.0 mL), ethylene glycol (1.11 mL, 20.0 mmol), 1,2,3,4-tetrahydroisoquinoline (2.0 mL, 15 mmol) and iodobenzene (1.12 mL, 10.0 mmol) were added successively by micro-syringe at room temperature. The reaction mixture was heated at 85-90 °C and kept for 24 h and then allowed to cool to room temperature. Ethyl acetate (20 mL) and water (20 mL) were then added to the reaction mixture. The organic layer was extracted with ethyl acetate (2\*20 mL). The combined organic phases were washed with brine and dried over anhydrous sodium sulphate. The solvent was removed by rotary evaporation and purified by column chromatography on silica gel desired product la with 60-80 % isolated yields.

#### Procedure for the synthesis



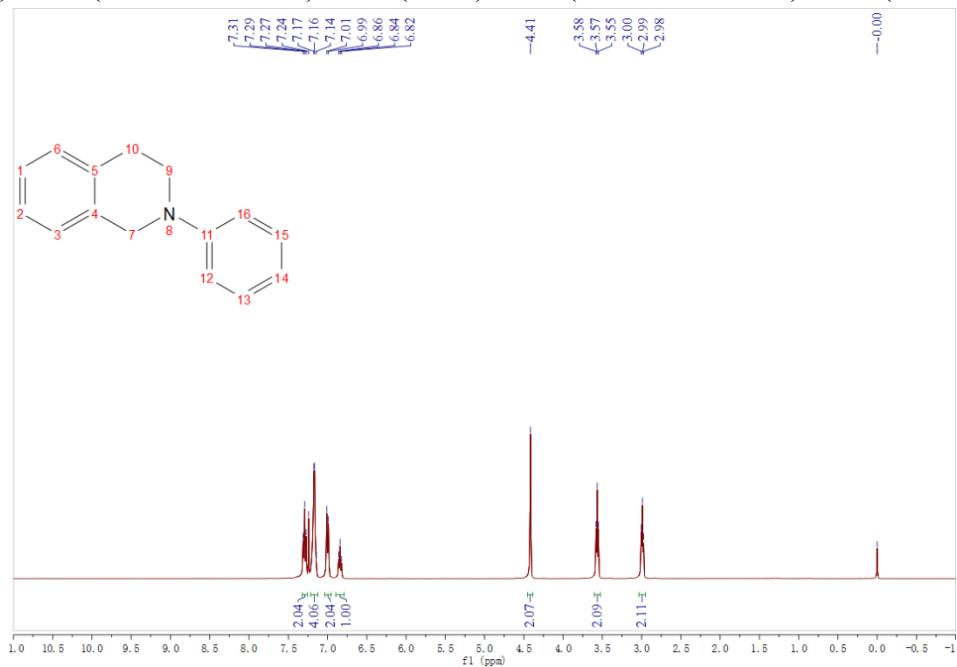


## 2.2 $^1\text{H}$ NMR spectrum of N-aryl-tetrahydroisoquinolines derivatives

Name: N-Phenyl-1,2,3,4-tetrahydroisoquinoline (1a)

Chemical Formula: C<sub>15</sub>H<sub>15</sub>N

White solid.  $^1\text{H}$  NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.29 (t,  $J = 8.0$  Hz, 2H), 7.20-7.14 (m, 4H), 7.00(d,  $J = 8.0$  Hz, 2H), 6.84 (t,  $J = 8.0$  Hz, 1H), 4.41 (s, 2H), 3.57 (t,  $J = 6.0$  Hz, 2H), 2.99 (t,  $J = 4.0$  Hz, 2H).

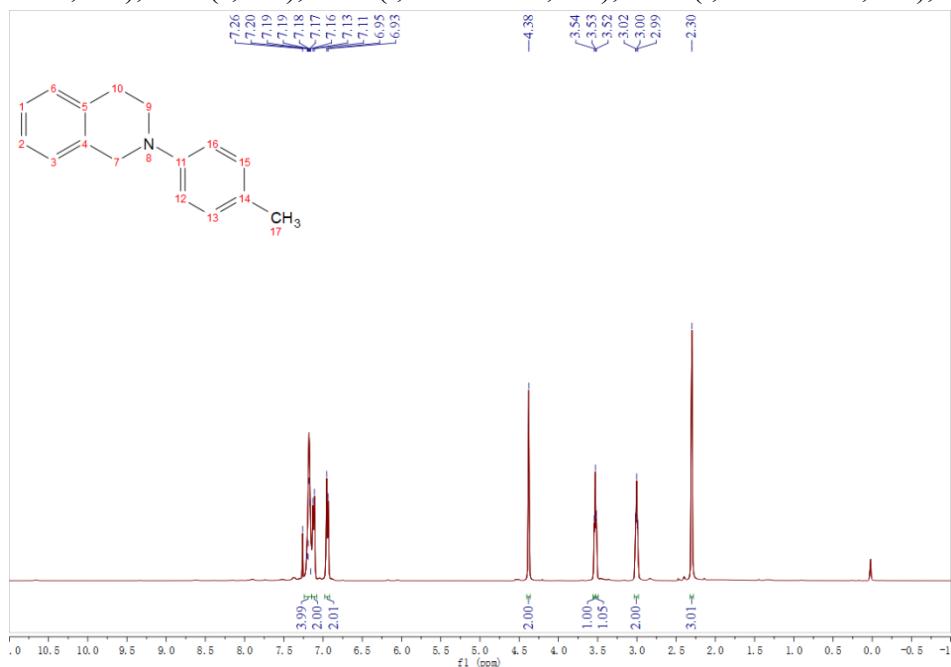


**Fig. S2**  $^1\text{H}$  NMR spectrum of 1a.

Name: 2-(4-Methylphenyl)-1,2,3,4-tetrahydroisoquinoline (1b)

Chemical Formula: C<sub>15</sub>H<sub>17</sub>N

Yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.20-7.16 (m,  $J = 4.0$  Hz, 4H), 7.12 (d,  $J = 8.0$  Hz, 2H), 6.94 (d,  $J = 8.0$  Hz, 2H), 4.38(s, 2H), 3.53 (t,  $J = 4.0$  Hz, 2H), 3.00 (t,  $J = 6.0$  Hz, 2H), 2.30 (s, 3H).

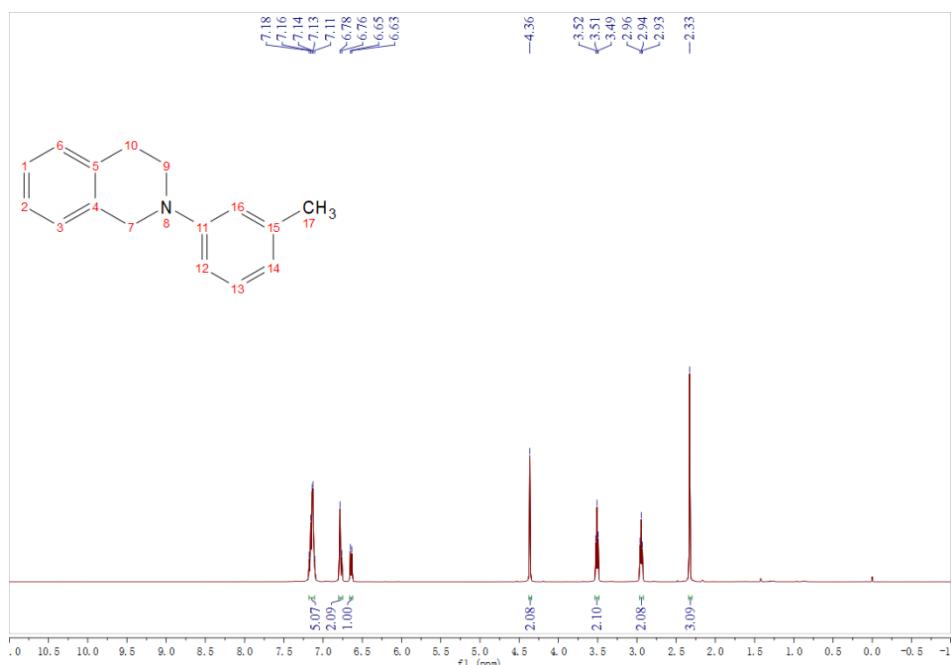


**Fig. S3**  $^1\text{H}$  NMR spectrum of 1b.

Name: 2-(3-Methylphenyl)-1,2,3,4-tetrahydroisoquinoline (1c)

Chemical Formula:  $\text{C}_{15}\text{H}_{17}\text{N}$

Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.18-7.11(m,  $J = 7.0$  Hz, 5H), 6.77 (d,  $J = 8.0$  Hz, 2H), 6.64 (d,  $J = 8.0$  Hz, 1H), 4.36 (s, 2H), 3.51 (t,  $J = 6.0$  Hz, 2H), 2.94 (t,  $J = 6.0$  Hz, 2H), 2.33 (s, 3H).

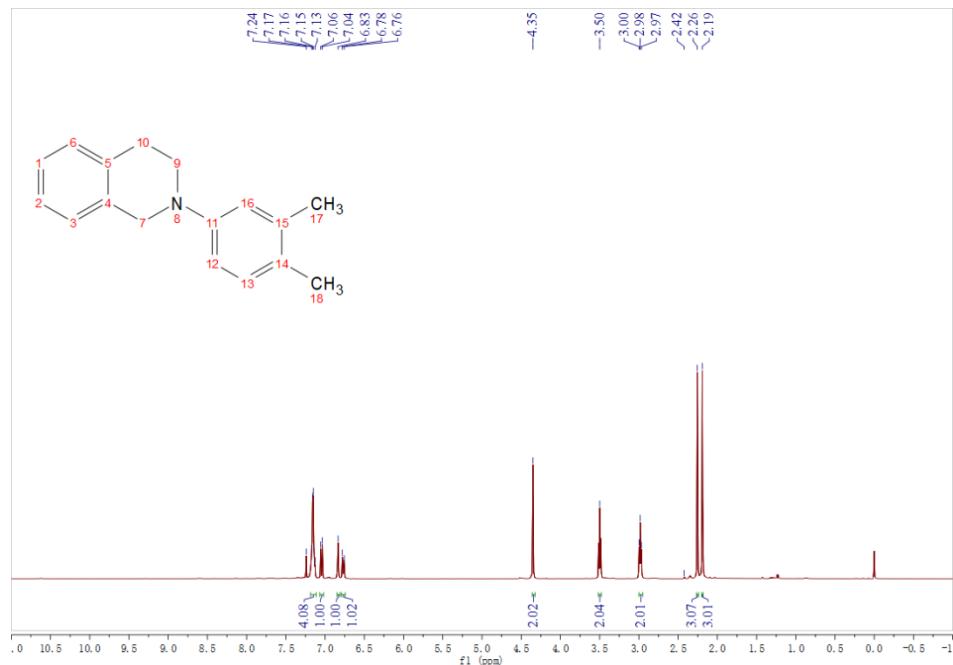


**Fig. S4**  $^1\text{H}$  NMR spectrum of 1c.

Name: 2-(3,4-Dimethylphenyl)-1,2,3,4-tetrahydroisoquinoline (1d)

Chemical Formula: C<sub>15</sub>H<sub>19</sub>N

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.18-7.13(m, *J* = 5.0 Hz, 4H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.83 (s, 1H), 6.77 (d, *J* = 8.0 Hz, 1H), 4.35 (s, 2H), 3.50 (s, 2H), 2.98 (t, *J* = 6.0 Hz, 2H), 2.26 (s, 3H), 2.19 (s, 3H).

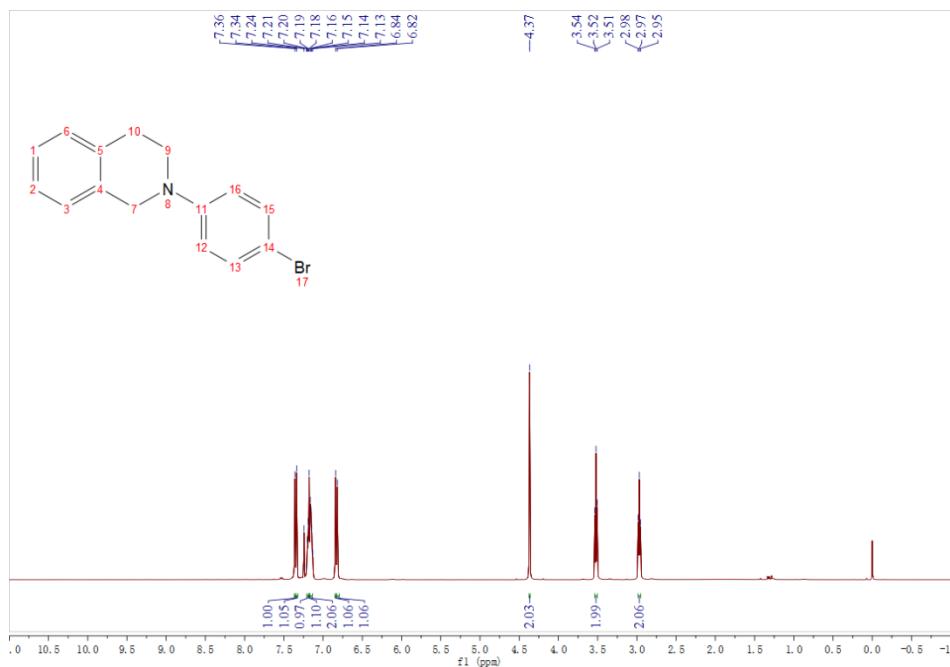


**Fig. S5** <sup>1</sup>H NMR spectrum of 1d.

Name: 2-(4-Bromophenyl)-1,2,3,4-tetrahydroisoquinoline (1e)

Chemical Formula: C<sub>15</sub>H<sub>14</sub>BrN

White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.36 (s, 1H), 7.34 (s, 1H), 7.21-7.13 (m, *J* = 4.6 Hz, 4H), 6.84 (s, 1H), 6.82 (s, 1H), 4.37 (s, 2H), 3.52 (t, *J* = 6.0 Hz, 2H), 2.97 (t, *J* = 6.0 Hz, 2H).

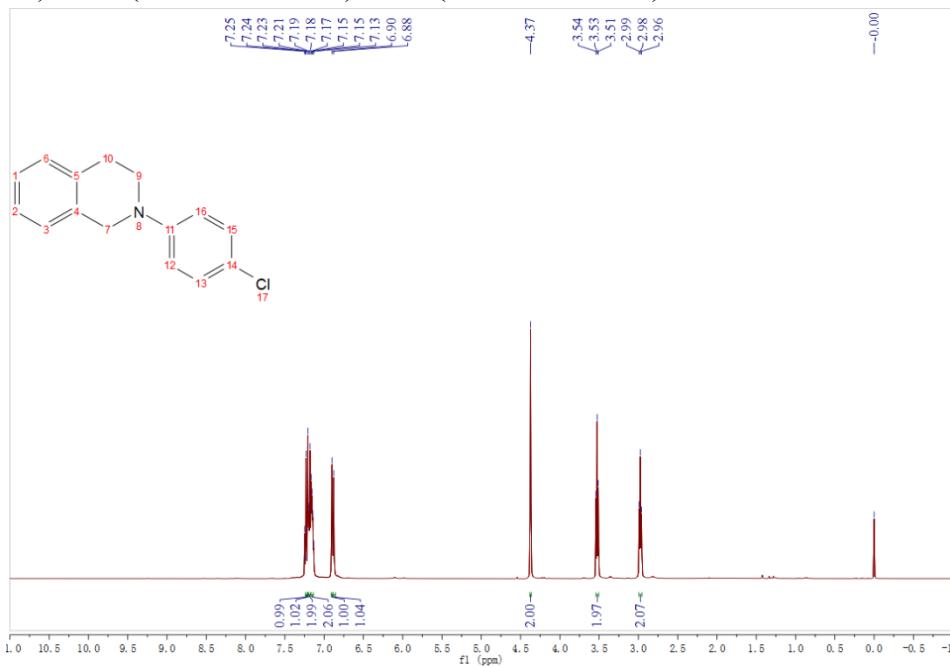


**Fig. S6**  $^1\text{H}$  NMR spectrum of 1e.

Name: 2-(4-Chlorophenyl)-1,2,3,4-tetrahydroisoquinoline (1f)

Chemical Formula: C<sub>15</sub>H<sub>14</sub>ClN

White solid.  $^1\text{H}$  NMR (400 MHz, CDCl<sub>3</sub>) δ 7.24-7.13 (m,  $J = 6.3$  Hz, 6H), 6.89 (d,  $J = 8.0$  Hz, 2H), 4.37 (s, 2H), 3.53 (t,  $J = 6.0$  Hz, 2H), 2.98 (t,  $J = 6.0$  Hz, 2H).

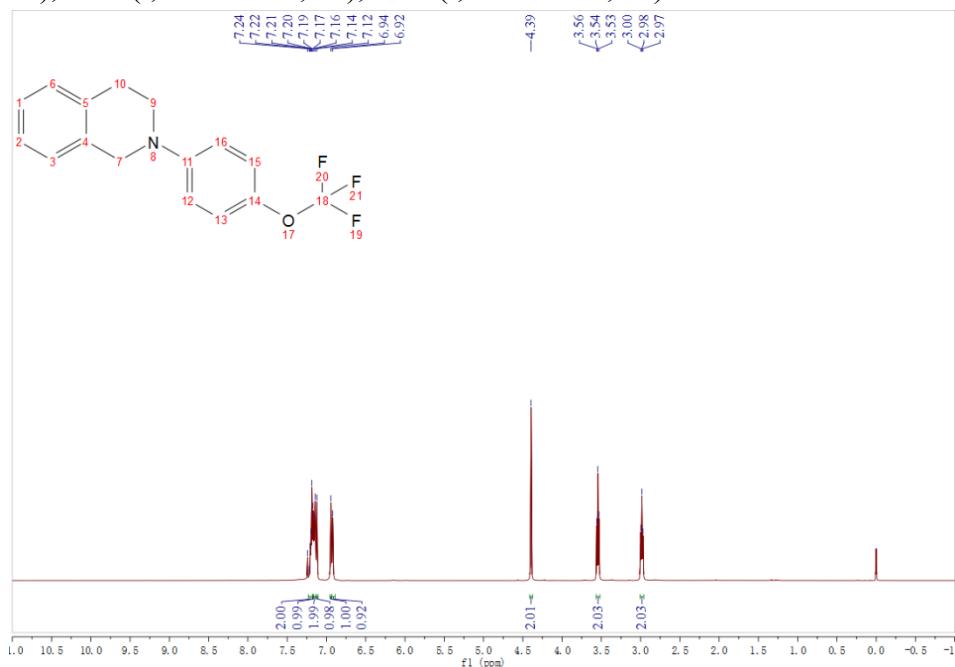


**Fig. S7**  $^1\text{H}$  NMR spectrum of 1f.

Name: 2-(4-(Trifluoromethoxy) phenyl)-1,2,3,4-tetrahydroisoquinoline (1g)

Chemical Formula: C<sub>16</sub>H<sub>14</sub>F<sub>3</sub>NO

White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.22-7.11 (m,  $J = 5.7$  Hz, 6H), 6.93 (d,  $J = 8.0$  Hz, 2H), 4.39 (s, 2H), 3.54 (t,  $J = 6.0$  Hz, 2H), 2.98 (t,  $J = 6.0$  Hz, 2H).

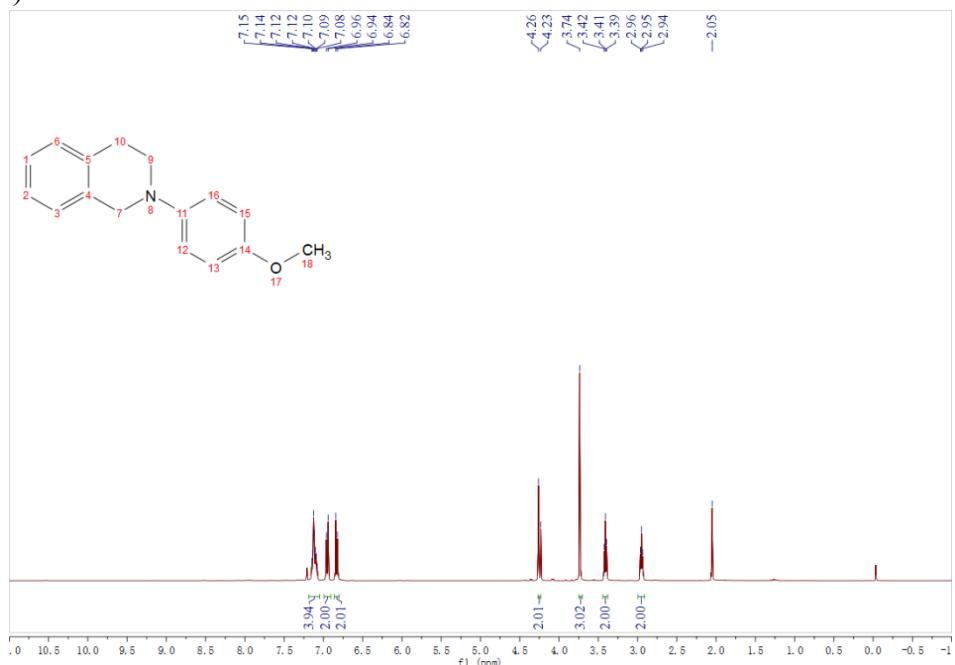


**Fig. S8**  $^1\text{H}$  NMR spectrum of 1g.

Name: 2-(4-Methoxylphenyl)-1,2,3,4-tetrahydroisoquinoline (1h)

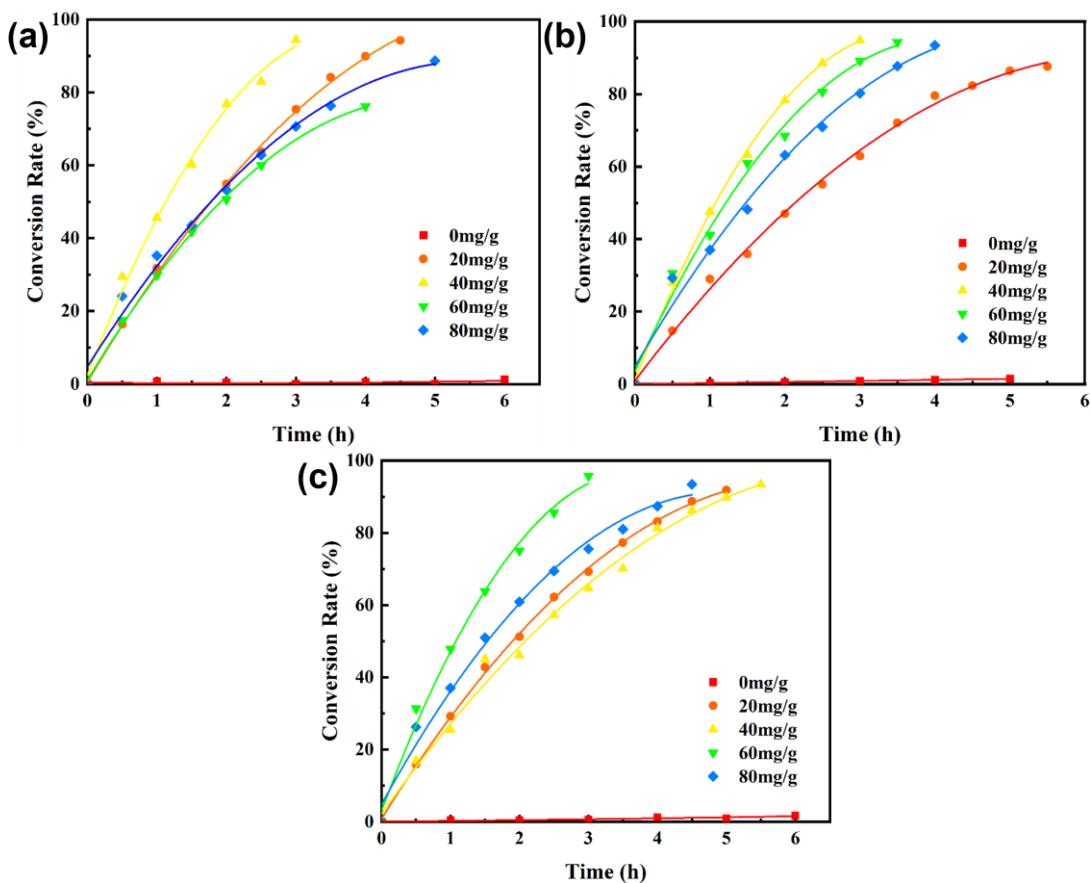
Chemical Formula:  $\text{C}_{16}\text{H}_{17}\text{NO}$

White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{C}_3\text{D}_6\text{O}$ )  $\delta$  7.15-7.08 (m,  $J = 4.7$  Hz, 4H), 6.95 (d,  $J = 8.0$  Hz, 2H), 6.83 (d,  $J = 8.0$  Hz, 2H), 4.25 (d,  $J = 12.0$  Hz, 2H), 3.74 (s, 3H), 3.41 (t,  $J = 6.0$  Hz, 2H), 2.95 (t,  $J = 4.0$  Hz, 2H).

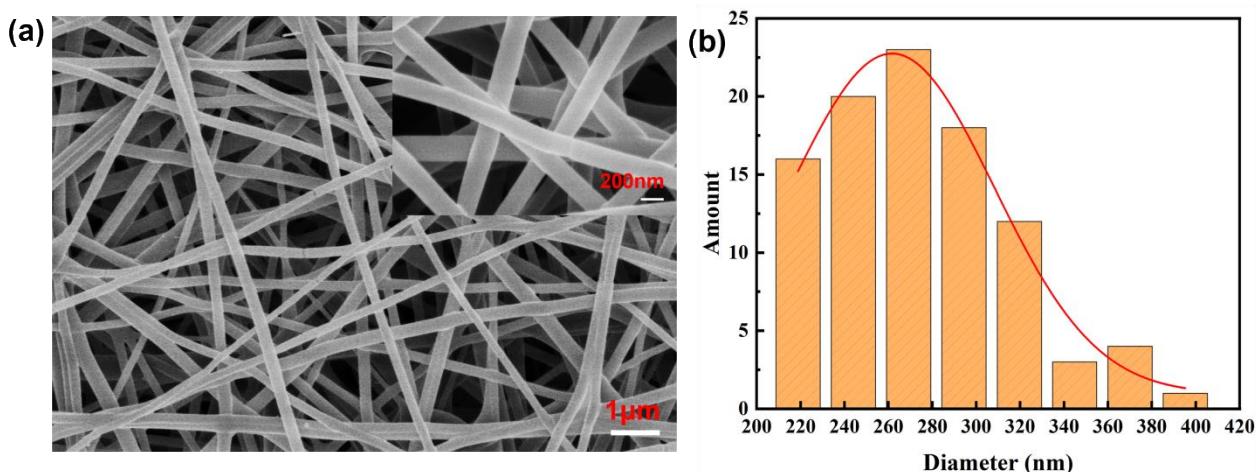


**Fig. S9**  $^1\text{H}$  NMR spectrum of 1h.

### 3. Investigation of catalytic performance



**Fig. S10** Substrate conversion rate curves with time for the oxidative coupling of N-phenyl-tetrahydroisoquinoline and dimethyl malonate catalyzed by OHCP nanofibers photocatalyst. (a) PDADMAC (Mw=10w) content of 5%. (b) PDADMAC (Mw=10w) content of 10%. (c) PDADMAC (Mw=20w) content of 5%.



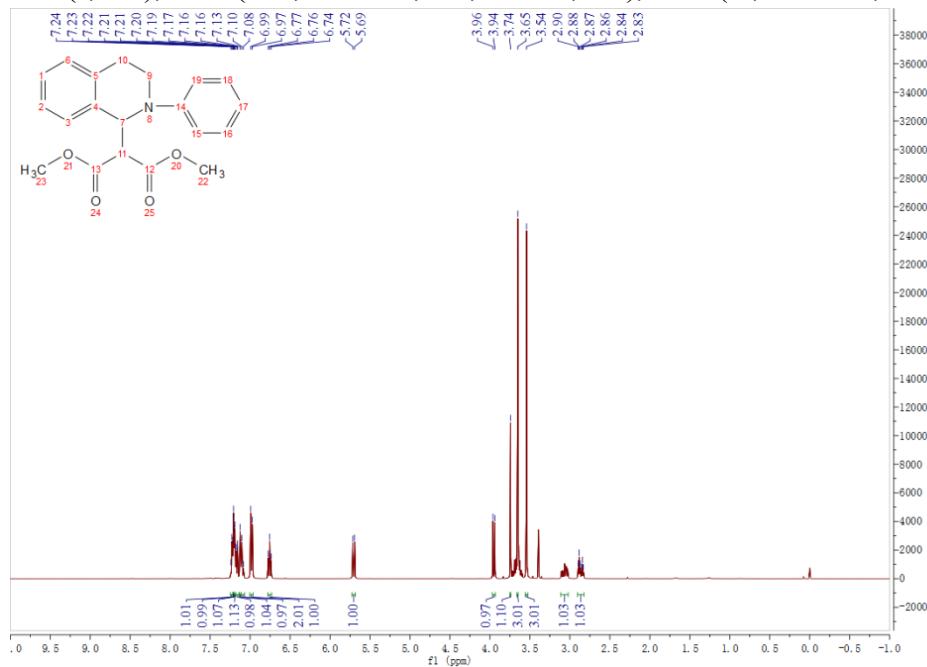
**Fig. S11** (a) SEM image of OHCP nanofibers photocatalyst with 10% of PDADMAC (Mw=100K). (b) Diameter distribution chart of OHCP nanofibers photocatalyst with 10% of PDADMAC (Mw=100K).

#### 4. $^1\text{H}$ NMR spectrum of N-phenyl-tetrahydroisoquinoline derivatives

Name: dimethyl 2-(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl) malonate(3a)

Chemical Formula:  $\text{C}_{20}\text{H}_{20}\text{NO}_4$

Yellow oil.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.23 (t,  $J = 4.3$  Hz, 1H), 7.21 (d,  $J = 1.5$  Hz, 1H), 7.19 (d,  $J = 4.5$  Hz, 1H), 7.18 – 7.14 (m, 1H), 7.13 (s, 1H), 7.09 (d,  $J = 7.6$  Hz, 1H), 6.98 (d,  $J = 8.1$  Hz, 2H), 6.76 (t,  $J = 7.3$  Hz, 1H), 5.70 (d,  $J = 9.4$  Hz, 1H), 3.95 (d,  $J = 9.4$  Hz, 1H), 3.74 (s, 1H), 3.65 (s, 3H), 3.54 (s, 3H), 3.07 (ddd,  $J = 15.7, 9.0, 6.2$  Hz, 1H), 2.86 (dt,  $J = 16.5, 5.1$  Hz, 1H).

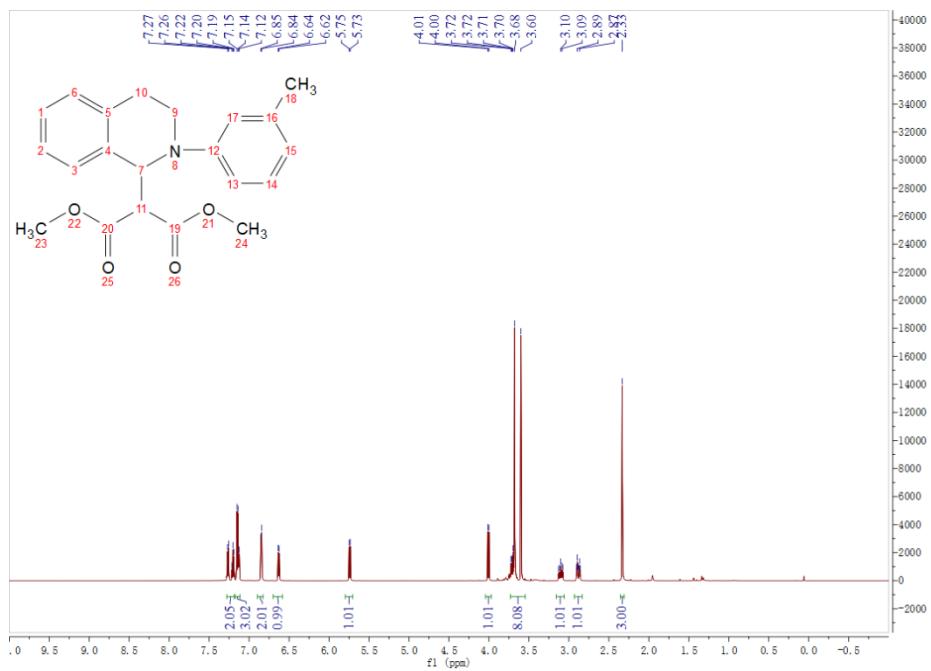


**Fig. S12**  $^1\text{H}$  NMR spectrum of 3a.

Name: dimethyl 2-(2-(m-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl) malonate (3b)

Chemical Formula:  $\text{C}_{21}\text{H}_{22}\text{NO}_4$

Yellow oil.  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 – 7.18 (m, 2H), 7.18 – 7.11 (m, 3H), 6.85 (d,  $J = 4.9$  Hz, 2H), 6.63 (d,  $J = 7.4$  Hz, 1H), 5.80 – 5.70 (m, 1H), 4.01 (d,  $J = 9.4$  Hz, 1H), 3.73 – 3.54 (m, 8H), 3.16 – 3.06 (m, 1H), 2.88 (dt,  $J = 16.5, 4.9$  Hz, 1H), 2.33 (s, 3H).

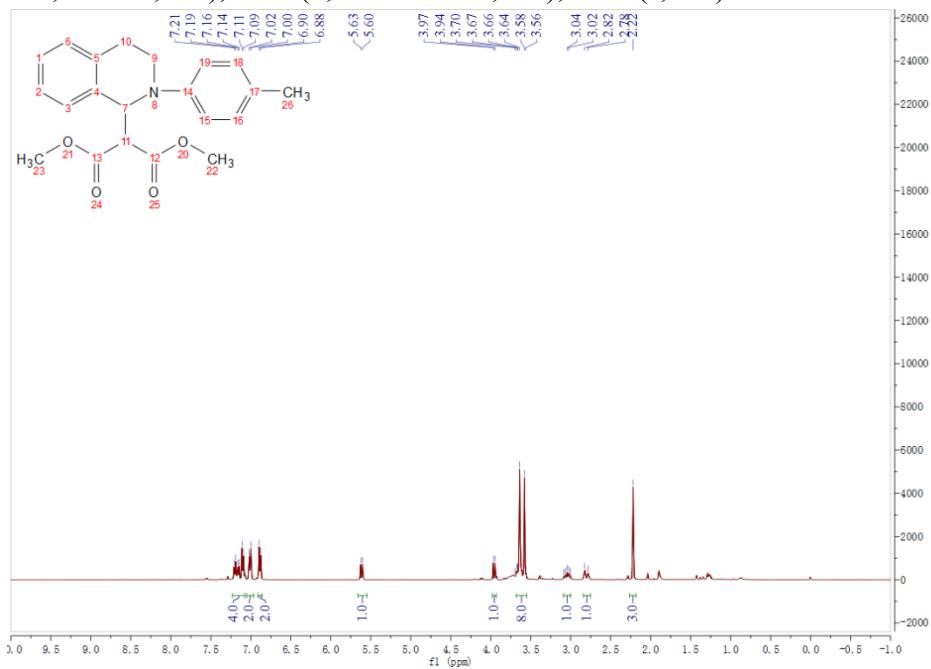


**Fig. S13**  $^1\text{H}$  NMR spectrum of 3b.

Name: dimethyl 2-(2-(p-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl) malonate(3c)

Chemical Formula:  $\text{C}_{21}\text{H}_{22}\text{NO}_4$

Yellow oil.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.23 – 7.08 (m, 4H), 7.01 (d,  $J$  = 8.0 Hz, 2H), 6.89 (d,  $J$  = 8.1 Hz, 2H), 5.61 (d,  $J$  = 9.4 Hz, 1H), 3.96 (d,  $J$  = 9.4 Hz, 1H), 3.61 (d,  $J$  = 25.0 Hz, 8H), 3.04 (dt,  $J$  = 16.1, 7.9 Hz, 1H), 2.80 (d,  $J$  = 16.5 Hz, 1H), 2.22 (s, 3H).

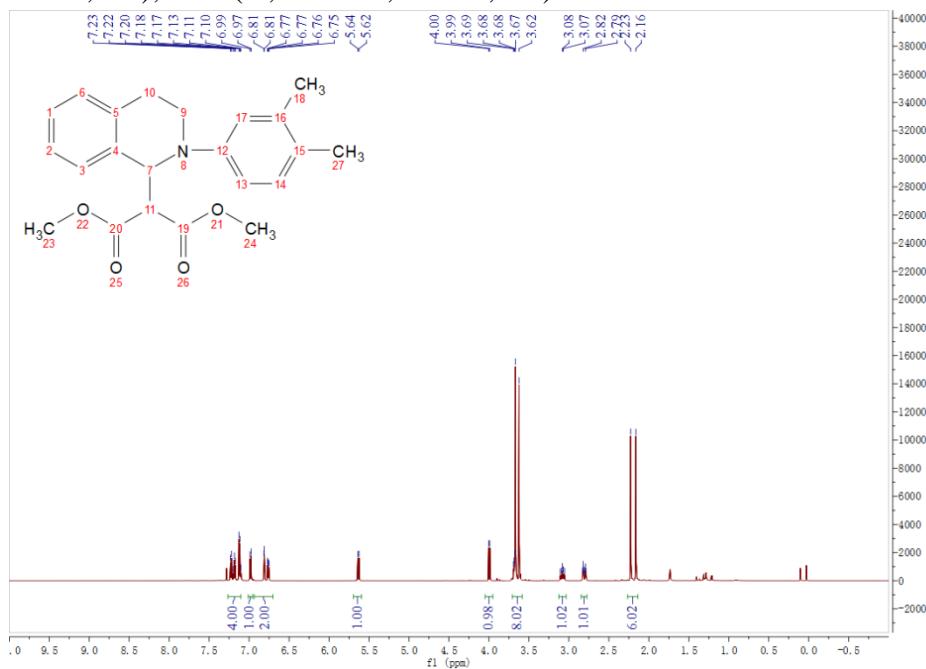


**Fig. S14**  $^1\text{H}$  NMR spectrum of 3c.

Name: dimethyl 2-(2-(3,4-dimethylphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl) malonate(3d)

Chemical Formula: C<sub>22</sub>H<sub>24</sub>NO<sub>4</sub>

Yellow oil. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 7.25 (d, J = 7.7 Hz, 1H), 7.15 (t, J = 7.3 Hz, 1H), 7.09 (d, J = 7.6 Hz, 2H), 6.95 (d, J = 8.3 Hz, 1H), 6.80 – 6.72 (m, 2H), 5.64 (d, J = 9.1 Hz, 1H), 4.22 (d, J = 7.1 Hz, 4H), 3.92 (d, J = 9.1 Hz, 1H), 3.69 – 3.62 (m, 2H), 3.09 – 3.03 (m, 1H), 2.83 – 2.77 (m, 1H), 2.17 (d, J = 39.6 Hz, 6H), 1.14 (dt, J = 14.5, 7.1 Hz, 6H).

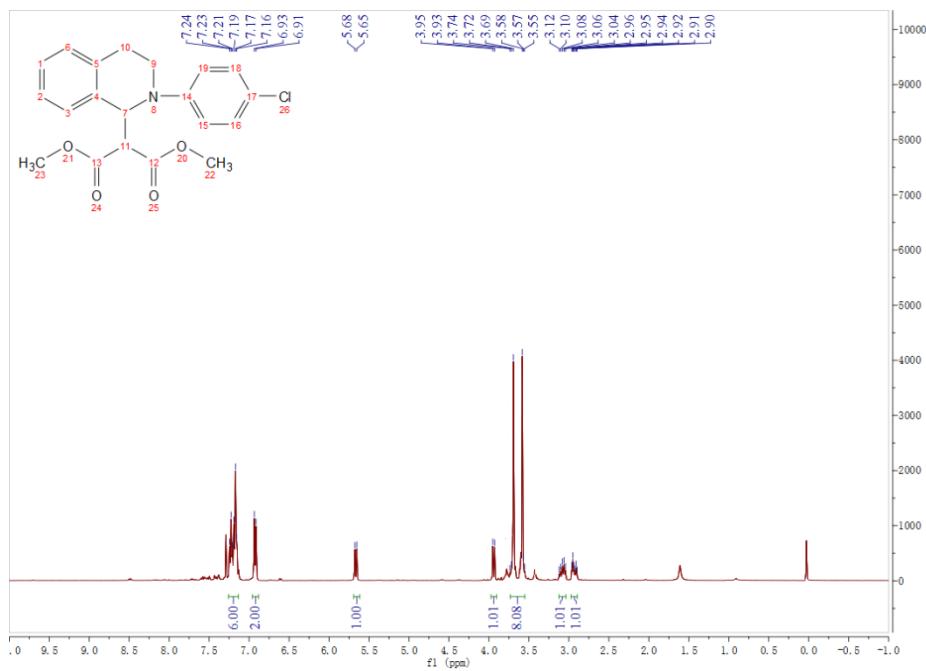


**Fig. S15** <sup>1</sup>H NMR spectrum of 3d.

Name: dimethyl 2-(2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)malonate (3e)

Chemical Formula: C<sub>20</sub>H<sub>19</sub>NO<sub>4</sub>Cl

Yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.20 (dt, *J* = 21.8, 6.5 Hz, 6H), 6.92 (d, *J* = 8.5 Hz, 2H), 5.67 (d, *J* = 9.4 Hz, 1H), 3.94 (d, *J* = 9.4 Hz, 1H), 3.64 (d, *J* = 44.2 Hz, 8H), 3.08 (dt, *J* = 15.1, 7.3 Hz, 1H), 2.93 (dt, *J* = 16.1, 5.3 Hz, 1H).

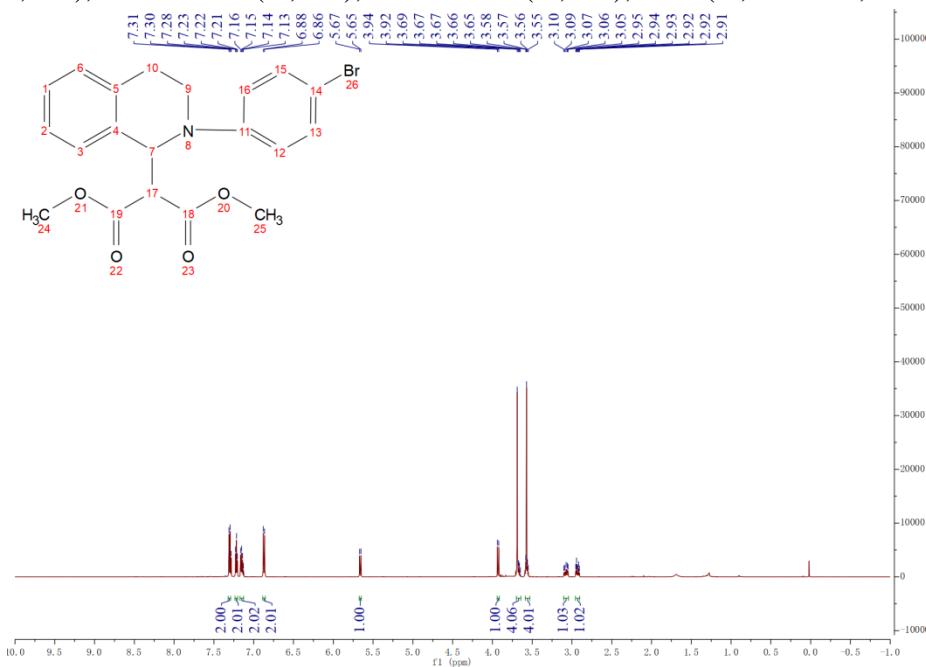


**Fig. S16**  $^1\text{H}$  NMR spectrum of 3e.

Name: dimethyl 2-(2-(4-bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)malonate (3f)

Chemical Formula:  $\text{C}_{20}\text{H}_{19}\text{NO}_4\text{Br}$

Yellow oil.  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30 (d,  $J = 9.0$  Hz, 2H), 7.22 (t,  $J = 6.8$  Hz, 2H), 7.15 (dd,  $J = 14.5, 6.8$  Hz, 2H), 6.87 (d,  $J = 9.0$  Hz, 2H), 5.66 (d,  $J = 9.5$  Hz, 1H), 3.93 (d,  $J = 9.5$  Hz, 1H), 3.70 – 3.64 (m, 4H), 3.59 – 3.53 (m, 4H), 3.10 – 3.04 (m, 1H), 2.93 (dt,  $J = 16.3, 5.4$  Hz, 1H).

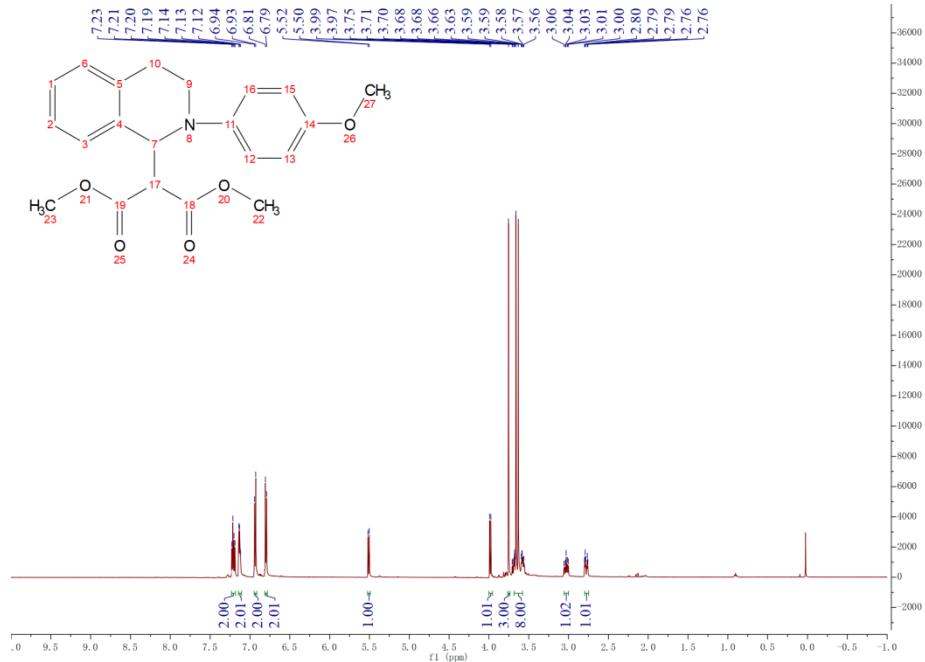


**Fig. S17**  $^1\text{H}$  NMR spectrum of 3f.

Name:dimethyl 2-(2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)malonate (3g)

Chemical Formula: C<sub>21</sub>H<sub>22</sub>NO<sub>5</sub>

Yellow oil. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 7.21 (dd, J = 15.9, 7.6 Hz, 2H), 7.14 – 7.11 (m, 2H), 6.94 (d, J = 9.0 Hz, 2H), 6.80 (d, J = 9.0 Hz, 2H), 5.51 (d, J = 9.4 Hz, 1H), 3.98 (d, J = 9.4 Hz, 1H), 3.75 (s, 3H), 3.68 – 3.58 (m, 8H), 3.02 (dd, J = 16.6, 6.3 Hz, 1H), 2.78 (dd, J = 16.6, 4.2 Hz, 1H).

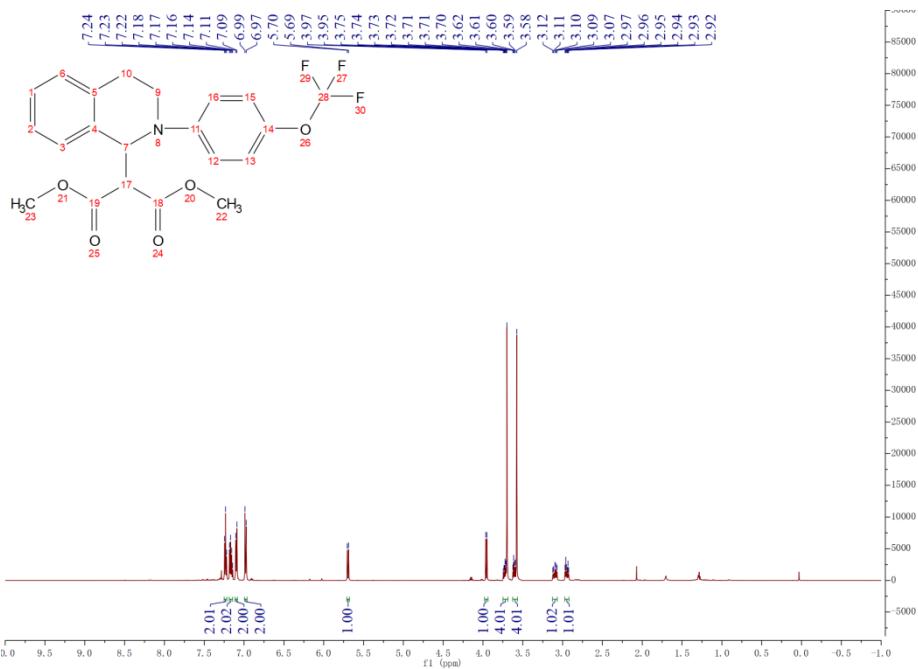


**Fig. S18** <sup>1</sup>H NMR spectrum of 3g.

Name:dimethyl 2-(2-(4-(trifluoromethoxy)phenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)malonate (3h)

Chemical Formula: C<sub>21</sub>H<sub>19</sub>NO<sub>5</sub>F<sub>3</sub>

Yellow oil. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 7.23 (t, J = 7.5 Hz, 2H), 7.16 (dd, J = 15.0, 7.2 Hz, 2H), 7.10 (d, J = 9.0 Hz, 2H), 6.98 (d, J = 9.2 Hz, 2H), 5.70 (d, J = 9.5 Hz, 1H), 3.96 (d, J = 9.5 Hz, 1H), 3.75 – 3.69 (m, 4H), 3.63 – 3.57 (m, 4H), 3.13 – 3.07 (m, 1H), 2.95 (dt, J = 16.4, 5.4 Hz, 1H).

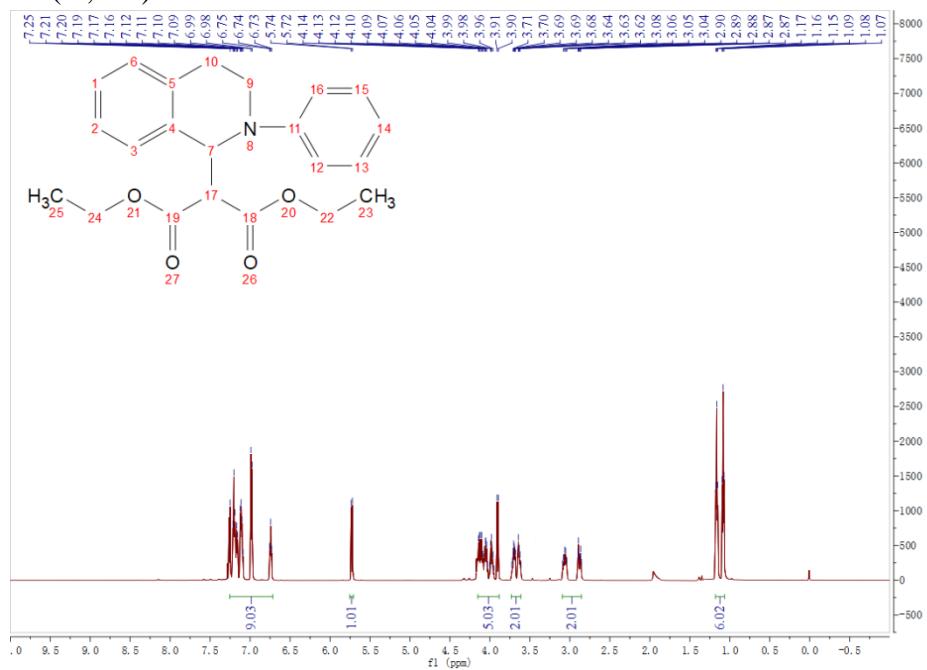


**Fig. S19**  $^1\text{H}$  NMR spectrum of 3h.

Name: diethyl 2-(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl) malonate(3i)

Chemical Formula:  $\text{C}_{22}\text{H}_{24}\text{NO}_4$

Yellow oil.  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 – 7.16 (m, 4H), 7.11 (dd,  $J = 13.8, 6.9$  Hz, 2H), 6.98 (d,  $J = 8.2$  Hz, 2H), 6.74 (t,  $J = 7.2$  Hz, 1H), 5.73 (d,  $J = 9.2$  Hz, 1H), 4.16 – 3.96 (m, 4H), 3.90 (d,  $J = 9.2$  Hz, 1H), 3.67 (dq,  $J = 12.6, 5.4$  Hz, 2H), 3.10 – 3.02 (m, 1H), 2.88 (dd,  $J = 18.0, 6.3$  Hz, 1H), 1.18 – 1.07 (m, 6H).

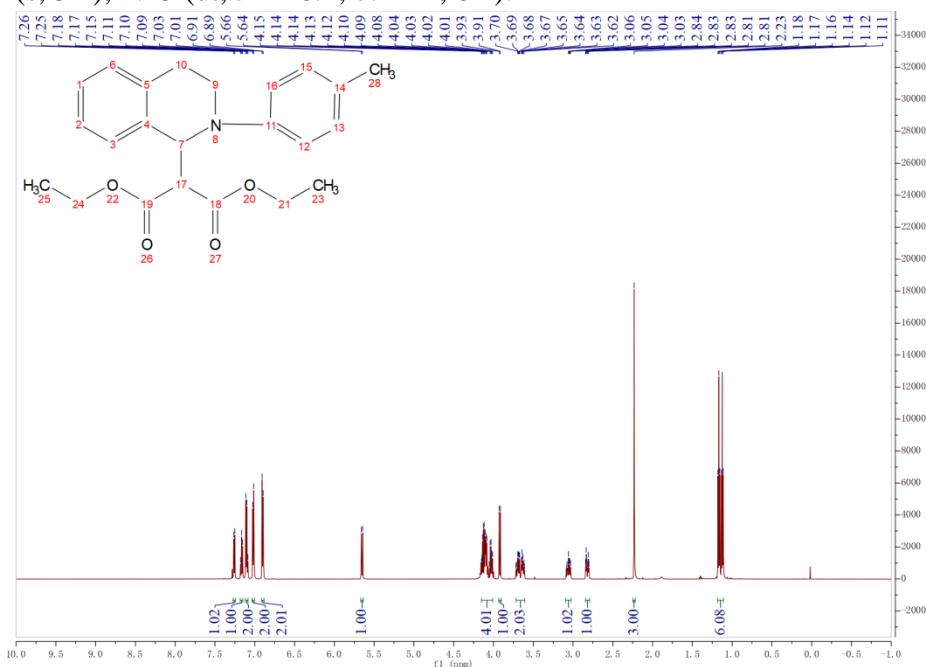


**Fig. S20**  $^1\text{H}$  NMR spectrum of 3i.

Name: diethyl 2-(2-(p-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl) malonate(3j)

Chemical Formula: C<sub>23</sub>H<sub>26</sub>NO<sub>4</sub>

Yellow oil. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 7.26 (d, J = 7.7 Hz, 1H), 7.17 (t, J = 7.1 Hz, 1H), 7.10 (t, J = 7.6 Hz, 2H), 7.02 (d, J = 8.5 Hz, 2H), 6.90 (d, J = 8.6 Hz, 2H), 5.65 (d, J = 9.2 Hz, 1H), 4.15 – 4.01 (m, 4H), 3.92 (d, J = 9.2 Hz, 1H), 3.72 – 3.61 (m, 2H), 3.09 – 3.02 (m, 1H), 2.82 (dt, J = 16.5, 4.6 Hz, 1H), 2.23 (s, 3H), 1.15 (dt, J = 26.2, 7.1 Hz, 6H).

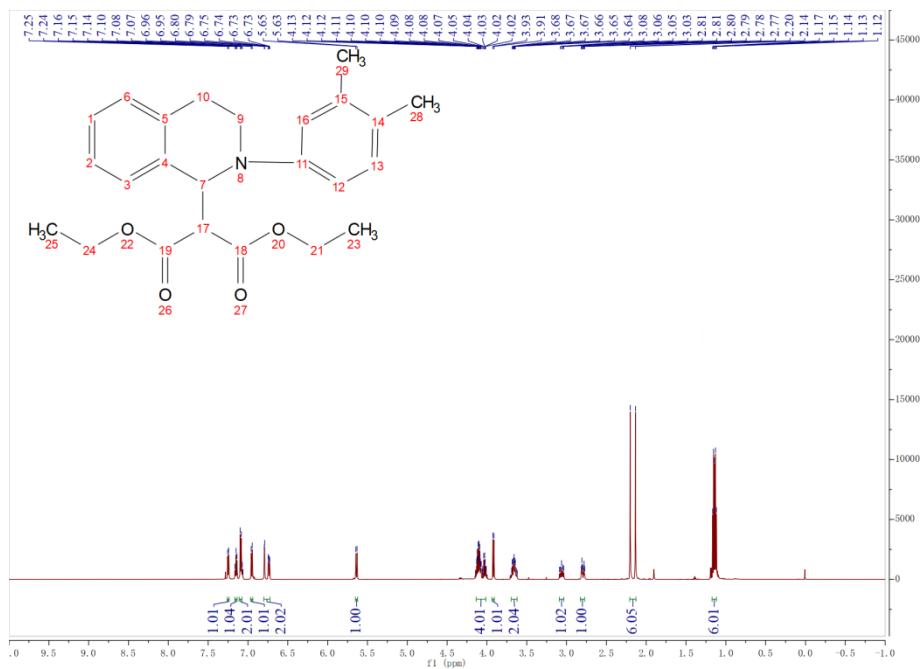


**Fig. S21** <sup>1</sup>H NMR spectrum of 3j.

Name: diethyl 2-(2-(3,4-dimethylphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl) malonate(3k)

Chemical Formula: C<sub>24</sub>H<sub>28</sub>NO<sub>4</sub>

Yellow oil. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 7.25 (d, J = 7.7 Hz, 1H), 7.15 (t, J = 7.3 Hz, 1H), 7.09 (d, J = 7.6 Hz, 2H), 6.95 (d, J = 8.3 Hz, 1H), 6.80 – 6.72 (m, 2H), 5.64 (d, J = 9.1 Hz, 1H), 4.13 – 4.01 (m, 4H), 3.92 (d, J = 9.1 Hz, 1H), 3.69 – 3.62 (m, 2H), 3.09 – 3.03 (m, 1H), 2.83 – 2.77 (m, 1H), 2.17 (d, J = 39.6 Hz, 6H), 1.14 (dt, J = 14.5, 7.1 Hz, 6H).

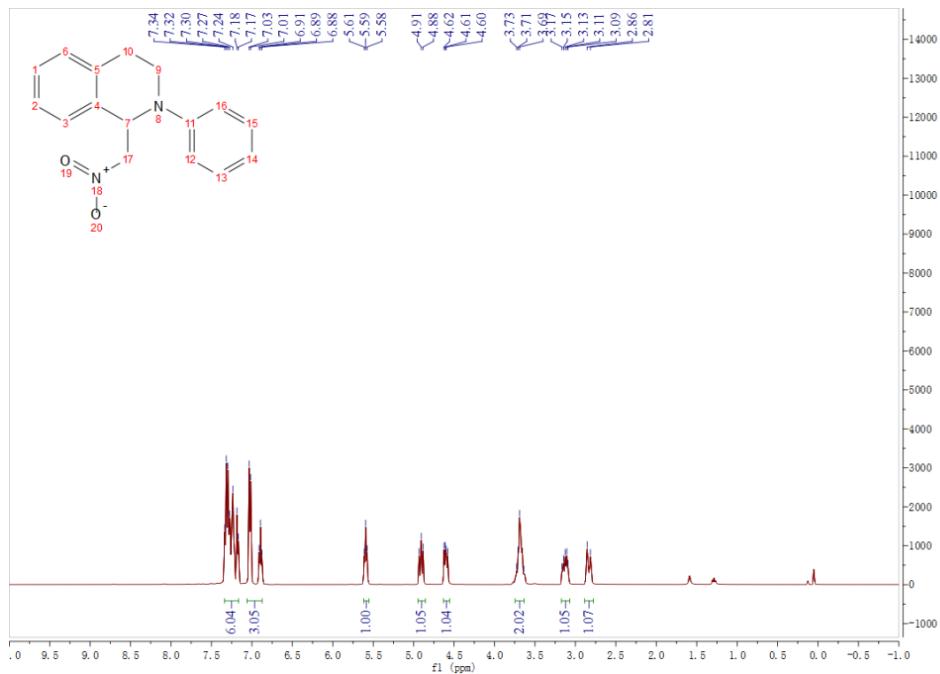


**Fig. S22** <sup>1</sup>H NMR spectrum of 3k.

Name: 1-(nitromethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline(5a)

Chemical Formula: C<sub>16</sub>H<sub>16</sub>NO<sub>2</sub>

Yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d)  $\delta$  7.29 – 7.20 (m, 3H), 7.17 (d,  $J$  = 7.4 Hz, 1H), 7.11 (d,  $J$  = 8.5 Hz, 2H), 6.93 (d,  $J$  = 8.6 Hz, 2H), 5.56 – 5.51 (m, 1H), 4.88 (dd,  $J$  = 11.9, 8.1 Hz, 1H), 4.59 (dd,  $J$  = 11.9, 6.4 Hz, 1H), 3.70 – 3.58 (m, 2H), 3.10 (ddd,  $J$  = 15.6, 9.4, 5.6 Hz, 1H), 2.79 (dt,  $J$  = 16.4, 4.5 Hz, 1H), 2.30 (s, 3H).

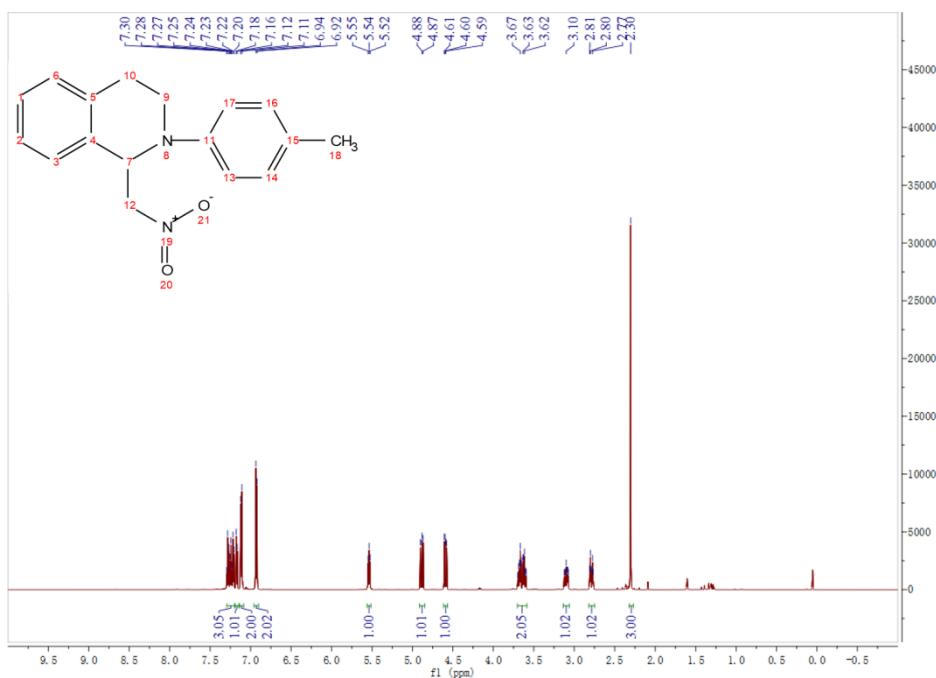


**Fig. S23** <sup>1</sup>H NMR spectrum of 5a.

Name: 1-(nitromethyl)-2-(p-tolyl)-1,2,3,4-tetrahydroisoquinoline(5b)

Chemical Formula: C<sub>17</sub>H<sub>18</sub>NO<sub>2</sub>

Yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d) δ 7.30 – 7.16 (m, 5H), 6.83 (d, J = 9.3 Hz, 2H), 6.72 (d, J = 7.4 Hz, 1H), 5.58 (t, J = 7.2 Hz, 1H), 4.90 (dd, J = 11.8, 7.8 Hz, 1H), 4.59 (dd, J = 11.8, 6.7 Hz, 1H), 3.72 – 3.61 (m, 2H), 3.12 (ddd, J = 14.9, 8.8, 5.5 Hz, 1H), 2.83 (dt, J = 16.3, 4.9 Hz, 1H), 2.37 (s, 3H).

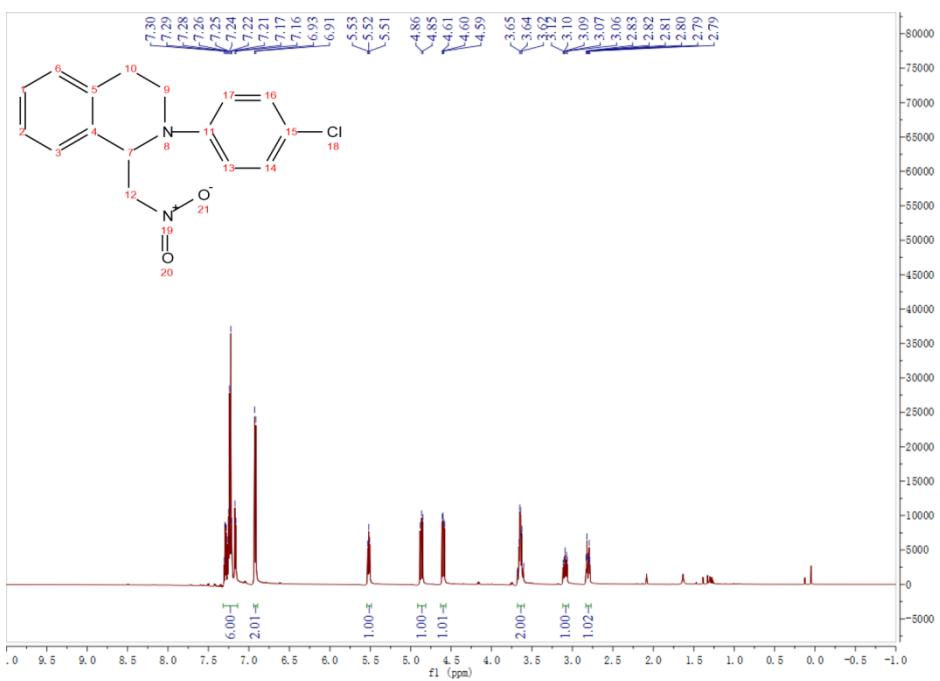


**Fig. S24** <sup>1</sup>H NMR spectrum of 5b.

Name: 2-(4-chlorophenyl)-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline(5c)

Chemical Formula:C<sub>16</sub>H<sub>15</sub>NO<sub>2</sub>Cl

Yellow oil. <sup>1</sup>H NMR (600 MHz, Chloroform-d) δ 7.37 (d, J = 9.1 Hz, 2H), 7.30 – 7.20 (m, 3H), 7.16 (d, J = 7.5 Hz, 1H), 6.87 (d, J = 9.1 Hz, 2H), 5.54 – 5.50 (m, 1H), 4.87 (dd, J = 12.0, 8.1 Hz, 1H), 4.59 (dd, J = 12.0, 6.4 Hz, 1H), 3.69 – 3.60 (m, 2H), 3.13 – 3.06 (m, 1H), 2.81 (dt, J = 16.4, 4.8 Hz, 1H).

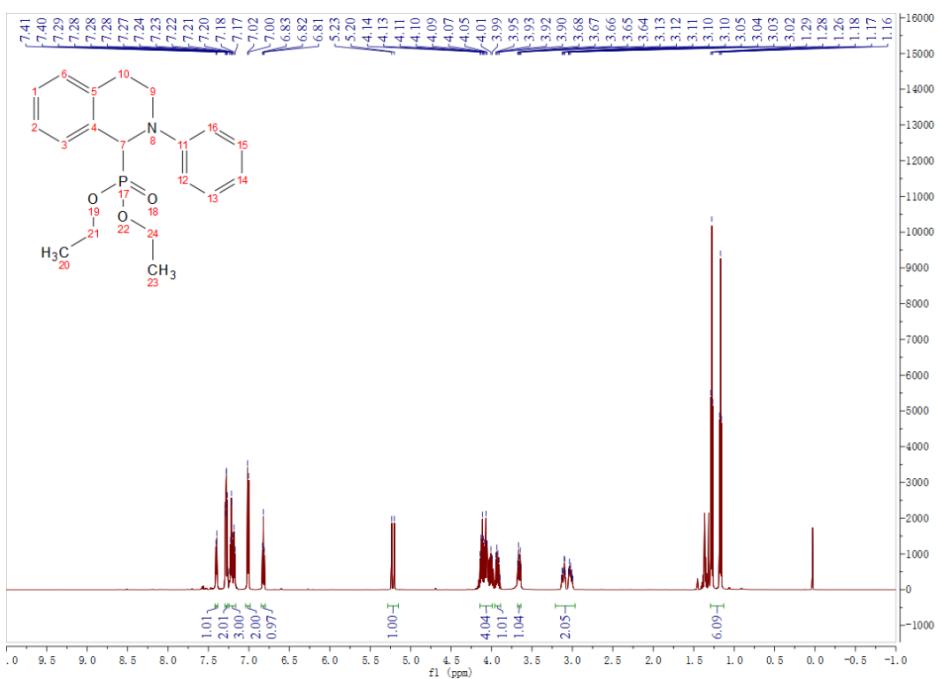


**Fig. S25**  $^1\text{H}$  NMR spectrum of 5c.

Name: diethyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (7a)

Chemical Formula:  $\text{C}_{19}\text{H}_{24}\text{NO}_3\text{P}$

Yellow oil.  $^1\text{H}$  NMR (600 MHz, Chloroform-*d*)  $\delta$  7.40 (d,  $J = 7.2$  Hz, 1H), 7.29 – 7.26 (m, 2H), 7.25 – 7.16 (m, 3H), 7.01 (d,  $J = 8.2$  Hz, 2H), 6.82 (t,  $J = 7.3$  Hz, 1H), 5.22 (d,  $J = 20.0$  Hz, 1H), 4.14 – 3.99 (m, 4H), 3.96 – 3.89 (m, 1H), 3.66 (dd,  $J = 12.0, 6.0$  Hz, 1H), 3.21 – 2.97 (m, 2H), 1.22 (dt,  $J = 64.9, 7.1$  Hz, 6H).

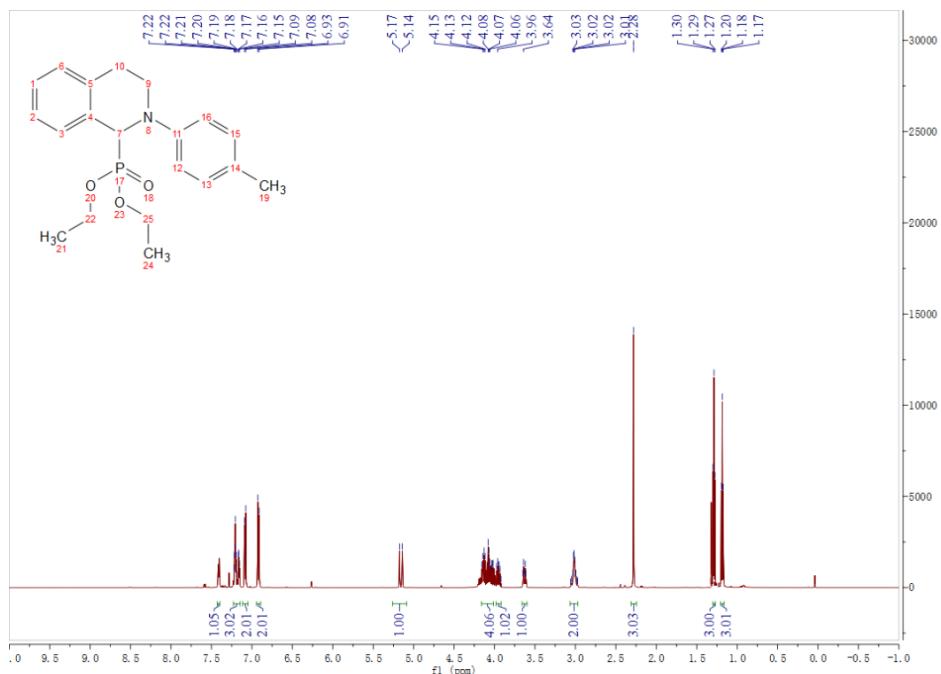


**Fig. S26**  $^1\text{H}$  NMR spectrum of 7a.

Name: diethyl (2-(p-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl) phosphonate(7b)

Chemical Formula:  $\text{C}_{20}\text{H}_{26}\text{NO}_3\text{P}$

Yellow oil.  $^1\text{H}$  NMR (600 MHz, Chloroform-*d*)  $\delta$  7.40 (s, 1H), 7.23 – 7.15 (m, 3H), 7.08 (d,  $J$  = 8.4 Hz, 2H), 6.92 (d,  $J$  = 8.6 Hz, 2H), 5.16 (d,  $J$  = 20.8 Hz, 1H), 4.16 – 4.01 (m, 4H), 3.98 – 3.92 (m, 1H), 3.66 – 3.60 (m, 1H), 3.07 – 2.97 (m, 2H), 2.28 (s, 3H), 1.29 (t,  $J$  = 7.1 Hz, 3H), 1.18 (t,  $J$  = 7.1 Hz, 3H).

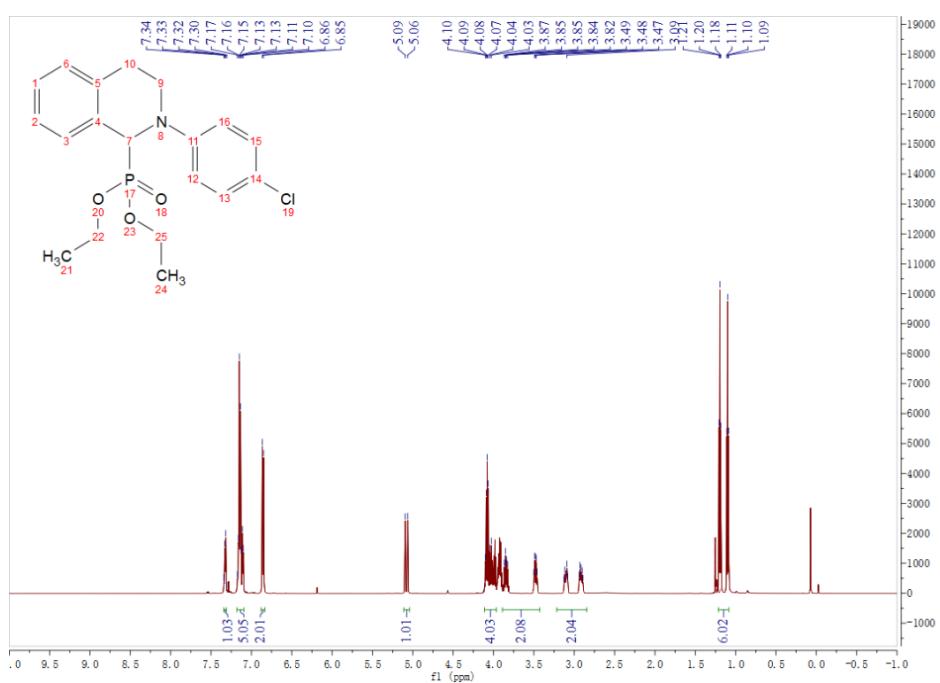


**Fig. S27**  $^1\text{H}$  NMR spectrum of 7b.

Name:diethyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (7c)

Chemical Formula:  $\text{C}_{19}\text{H}_{23}\text{NO}_3\text{PCl}$

Yellow oil.  $^1\text{H}$  NMR (600 MHz, Chloroform-*d*)  $\delta$  7.32 (d,  $J$  = 7.0 Hz, 1H), 7.14 (hept,  $J$  = 7.0 Hz, 5H), 6.86 (d,  $J$  = 9.1 Hz, 2H), 5.08 (d,  $J$  = 19.1 Hz, 1H), 4.11 – 3.96 (m, 4H), 3.89 – 3.43 (m, 2H), 3.01 (ddt,  $J$  = 112.5, 13.6, 6.1 Hz, 2H), 1.15 (dt,  $J$  = 57.8, 7.1 Hz, 6H).



**Fig. S28** <sup>1</sup>H NMR spectrum of 7c.