Supporting Information

Photocatalytic hydrogen-evolution of 1-tetralones to \( \alpha \)-naphthols by continuous-flow technology

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1. General information

All reagents and solvents were commercial available unless individually noted. Co(dmgH)$_2$PyCl (III) was purchased from Acros. Quinolinium photocatalysts$^1$ and other cobaloxime catalysts$^2$ were prepared as previously reported. 500 W medium pressure Hanovia mercury lamp with a glass water cooling jacket (λ > 300 nm) was used as light source (please pay attention to the eye protection). The products were isolated by silica column chromatograph (200-300 meshes silica gel). $^1$H NMR, $^{13}$C NMR and $^{19}$F NMR spectra were recorded using a Bruker Advance DPX instrument (400, 101 and 377 MHz, respectively) with tetramethylsilane (TMS) as an internal standard. Data for $^1$H NMR are presented as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet, br = broad), coupling constant $J$ (Hz) and integration. Data for $^{13}$C NMR and $^{19}$F NMR was reported in term of chemical shift relative to different deuterium reagents. Mass spectra were recorded using a Trio-2000 GC-MS spectrometer. The generated photoproduct of H$_2$ was characterized and measured by GC analysis (14B Shimadzu) using argon as the carrier gas with a molecular sieve column (5 Å; 30 m × 0.53 mm) and a thermal conductivity detector (TCD). Methane was used as internal standard for the measurement of the yield of H$_2$. GC analysis was performed using N$_2$ as the carrier gas with capillary column (30 m × 0.25 mm × 0.33 µm) and a flame ionization detector (FID) using n-tetradecane as internal standard. Conversions and yields that were determined by $^1$H NMR were obtained from the crude reaction mixture by using n-tetradecane as an internal standard. Yields of products were based on the consumed substrates. UV-Vis absorption and luminescent quenching was performed on U-3900, F-4600 and LP-920. Cyclic voltammetry was performed on a CHI660E.
2. Experimental procedures

2.1 The lab-scale experiment conditions

QuCN⁺ (1×10⁻² mmol, 2.69 mg) and Co(dmgBF₂)₃(CH₃CN)₂ (II) (6×10⁻³ mmol, 2.8 mg) were dissolved in different solvents and the reaction tube was sealed. After degassing by the argon, 1-tetralone (0.2 mmol, 27 µL) and BF₃·Et₂O (4×10⁻² mmol, 5 µL) were added. Methane (1 mL) was then injected as the internal standard for analysis of generated H₂, and the pinholes were sealed by paraffin. The reaction mixture was irradiation at room temperature using a 500 W medium pressure Hanovia mercury lamp with a glass water cooling jacket (λ > 300 nm). After 5 h irradiation, yield of H₂ was detected by GC-TCD. The conversion of 1-tetralone and yield of 1-naphathol (based on consumed 1-tetralone) were obtained from the crude reaction mixture by GC-FID using n-tetradecane as an internal standard.

Table S1. Optimization of solvents for the reaction condition

<table>
<thead>
<tr>
<th>entry</th>
<th>solvent (5 mL)</th>
<th>conv. 1a (%)</th>
<th>Yield 2a (%)(\text{a})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DCE</td>
<td>54</td>
<td>70</td>
</tr>
<tr>
<td>2</td>
<td>DCM</td>
<td>43</td>
<td>46</td>
</tr>
<tr>
<td>3</td>
<td>EtOH</td>
<td>n.d.</td>
<td>—</td>
</tr>
<tr>
<td>4</td>
<td>Acetone</td>
<td>66</td>
<td>49</td>
</tr>
<tr>
<td>5</td>
<td>DMF</td>
<td>n.d.</td>
<td>—</td>
</tr>
<tr>
<td>6</td>
<td>DMSO</td>
<td>n.d.</td>
<td>—</td>
</tr>
<tr>
<td>7</td>
<td>H₂O</td>
<td>n.d.</td>
<td>—</td>
</tr>
<tr>
<td>8</td>
<td>CH₃CN</td>
<td>63</td>
<td>70</td>
</tr>
</tbody>
</table>

\(\text{a}\) Yields of 2a were based on the consumed 1a.

Photocatalysts (1×10⁻² mmol) and cobaloxime complexes (6×10⁻³ mmol, 2.8 mg) were dissolved in CH₃CN and the reaction tube was sealed. After degassing by the argon, 1-tetralone (1a, 0.2 mmol, 27 µL) was added. Methane (1 mL) was then injected as the internal standard for analysis of generated H₂, and the pinholes were sealed by paraffin. The reaction mixture was irradiation at room temperature using a 500 W medium pressure Hanovia mercury lamp with a glass water cooling jacket (λ > 300 nm). After 5 h irradiation, yield of H₂ was detected by GC-TCD. The conversion of 1a and yield of 1-naphathol 2a (based on consumed 1a) were obtained from the crude reaction mixture by GC-FID using n-tetradecane as an internal standard.

Table S2. Optimization of the reaction condition
<table>
<thead>
<tr>
<th>entry</th>
<th>PC</th>
<th>Co</th>
<th>Additives</th>
<th>Conv. 1a (%)</th>
<th>Yield 2a. (%)&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Yield H&lt;sub&gt;2&lt;/sub&gt; (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>QuH&lt;sup&gt;+&lt;/sup&gt;</td>
<td>I</td>
<td>—</td>
<td>48</td>
<td>48</td>
<td>31</td>
</tr>
<tr>
<td>2</td>
<td>QuH&lt;sup&gt;+&lt;/sup&gt;</td>
<td>II</td>
<td>—</td>
<td>40</td>
<td>52</td>
<td>25</td>
</tr>
<tr>
<td>3</td>
<td>QuH&lt;sup&gt;+&lt;/sup&gt;</td>
<td>III</td>
<td>—</td>
<td>45</td>
<td>62</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>QuH&lt;sup&gt;+&lt;/sup&gt;</td>
<td>IV</td>
<td>—</td>
<td>25</td>
<td>60</td>
<td>22</td>
</tr>
<tr>
<td>5</td>
<td>QuCN&lt;sup&gt;+&lt;/sup&gt;</td>
<td>I</td>
<td>—</td>
<td>30</td>
<td>53</td>
<td>20</td>
</tr>
<tr>
<td>6</td>
<td>QuCN&lt;sup&gt;+&lt;/sup&gt;</td>
<td>II</td>
<td>—</td>
<td>63</td>
<td>70</td>
<td>50</td>
</tr>
<tr>
<td>7</td>
<td>QuCN&lt;sup&gt;+&lt;/sup&gt;</td>
<td>II</td>
<td>BF&lt;sub&gt;3&lt;/sub&gt;·Et&lt;sub&gt;2&lt;/sub&gt;O&lt;sup&gt;b&lt;/sup&gt;</td>
<td>78</td>
<td>77</td>
<td>70</td>
</tr>
<tr>
<td>8&lt;sup&gt;c&lt;/sup&gt;</td>
<td>QuCN&lt;sup&gt;+&lt;/sup&gt;</td>
<td>II</td>
<td>BF&lt;sub&gt;3&lt;/sub&gt;·Et&lt;sub&gt;2&lt;/sub&gt;O&lt;sup&gt;b&lt;/sup&gt;</td>
<td>73</td>
<td>76</td>
<td>65</td>
</tr>
<tr>
<td>9&lt;sup&gt;d&lt;/sup&gt;</td>
<td>QuCN&lt;sup&gt;+&lt;/sup&gt;</td>
<td>II</td>
<td>BF&lt;sub&gt;3&lt;/sub&gt;·Et&lt;sub&gt;2&lt;/sub&gt;O&lt;sup&gt;b&lt;/sup&gt;</td>
<td>36</td>
<td>53</td>
<td>0</td>
</tr>
<tr>
<td>10&lt;sup&gt;d&lt;/sup&gt;</td>
<td>QuCN&lt;sup&gt;+&lt;/sup&gt;</td>
<td>—</td>
<td>BF&lt;sub&gt;3&lt;/sub&gt;·Et&lt;sub&gt;2&lt;/sub&gt;O&lt;sup&gt;b&lt;/sup&gt;</td>
<td>19</td>
<td>34</td>
<td>0</td>
</tr>
</tbody>
</table>

<sup>a</sup> Yields of 2a were based on the consumed 1a.  
<sup>b</sup> 20 mol% BF<sub>3</sub>·Et<sub>2</sub>O was added.  
<sup>c</sup> Under N<sub>2</sub> atmosphere.  
<sup>d</sup> Under 1 atm O<sub>2</sub>.  

(4)
2.2 The attempt to hydroxylation of naphthalene with H$_2$O

The reaction was carried out according the previous report.$^{1b}$ After 10 hour’s irradiation, H$_2$ was detected by GC-TCD. The conversion of naphthalene and yield of 1-naphthol and 2-naphthol were detected from the crude reaction mixture by GC-FID using n-tetradecane as an internal standard.

\[
\text{Naphthalene} + \text{H}_2\text{O} \xrightarrow{\text{QuCN}^+ (5 \text{ mol} \%) \text{ II} (3 \text{ mol} \%)} \lambda > 300 \text{ nm, CH}_3\text{CN} (5 \text{ mL}) \xrightarrow{\text{Ar, rt, 10 h}} \text{1-naphthol} + \text{H}_2
\]

2.3 The continuous-flow reactor

The self-built continuous-flow reactor: a PTFE tube (ID = 612 µm, L = 9 m, V_r = 2.65 mL) is coiled around a glass cooling jacket, and a 500 W medium pressure mercury lamp is put into the jacket. The jacket system is immersed in a cooling water bath. The two ends of the tube are respectively connected with a peristaltic pump to push the solution through the PTFE tube and a conical flask for receiving the reaction mixture (Fig. S1).

2.4 General Procedure for Continuous-Flow Experiments

1-tetralones (0.2 mmol, liquid 1-tetralone derivatives were added after degassing), QuCN$^+$ (1×10$^{-2}$ mmol, 2.69 mg) and Co(dmgBF$_2$)$_2$(CH$_3$CN)$_2$ (II) (6×10$^{-3}$ mmol, 2.8 mg), were dissolved in CH$_3$CN (5 mL) and the reaction tube was sealed. After degassing by argon, a 10 mL syringe was used to extract the liquid from the reaction and connected to PTEF tube (ID = 612 µm, L = 9 m, V_r = 2.65 mL) of continuous flow reactor. The solution was irradiation at room temperature using a 500 W high pressure Hanovia mercury lamp with a glass water cooling jacket (λ > 300 nm). The conversion and yields were determined by $^1$H NMR using n-tetradecane as an internal standard. All the reactions were performed at least twice. The reaction mixture of parallel reactions were combined and isolated by flash column chromatography on silica gel using a mixture of petroleum ether-EtOAc as eluent. The isolated yields were the average yields of parallel reactions.
Table S3. Optimization of the continuous-flow reaction condition

<table>
<thead>
<tr>
<th>entry</th>
<th>reaction time</th>
<th>BF₃·Et₂O</th>
<th>Conv. 1a (%)</th>
<th>Yield 2a (%) (^a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30 min</td>
<td>10 mol%</td>
<td>60</td>
<td>97</td>
</tr>
<tr>
<td>2</td>
<td>1 h</td>
<td>10 mol%</td>
<td>78</td>
<td>77</td>
</tr>
<tr>
<td>3</td>
<td>30 min</td>
<td>20 mol%</td>
<td>66</td>
<td>100</td>
</tr>
<tr>
<td>4</td>
<td>1 h</td>
<td>20 mol%</td>
<td>83</td>
<td>48</td>
</tr>
</tbody>
</table>

\(^a\) Yields of 2a were based on the consumed 1a.

2.5 The scaled-up experiment in continuous-flow conditions

QuCN\(^\circ\) (5×10⁻² mmol, 13.4 mg), Co(dmgBF₂)₂(II) (3×10⁻² mmol, 14 mg) and 1n (1 mmol, 158.2 mg,) were dissolved in CH₃CN (25 mL) and the reactions tube was sealed. After degassing by the argon, BF₃·Et₂O (0.2 mmol, 25 µL) was added. After that, the reaction mixture was pumped into a 50 mL syringe under argon atmosphere; the syringe was then attached to the peristaltic pump. Continuous flow reactor: PTEF tube (ID = 612 µm, L = 9 m, \(V_R = 2.65 \text{ mL}\)) was set as 2.65 mL/h and the reaction time was determined to be 1 hour. 500 W medium pressure Hanovia mercury lamp with a glass water cooling jacket (\(\lambda > 300 \text{ nm}\)) was used as light source. After irradiation for a while, the conversion of 4-methyl-3,4-dihyronaphthalen-1(2H)-one and yield of 4-methylnaphthalen-1-ol were determined by \(^1\text{H NMR}\) from the crude reaction mixture by using \(n\)-tetradecane as an internal standard. The isolated yield of 1n and the yield of 2n (based on consumed 1n) were given under the product 2n.

2.6 Preparation of D₂-1a

\(D_2-1a\) was synthesized according the previous report.\(^4\) The \(D_2-1a\) was obtained in 85% yield with 98% D incorporation at C-2 according to spectroscopic analysis. \(^1\text{H NMR}\) (400 MHz, CDCl₃) \(\delta\): 8.03 (d, \(J = 7.8 \text{ Hz, 1H}\)), 7.47 (t, \(J = 7.4 \text{ Hz, 1H}\)), 7.38 – 7.20 (m, 2H), 2.97 (t, \(J = 6.0 \text{ Hz, 2H}\)), 2.63 (s, 0.04H), 2.13 (t, \(J = 5.9 \text{ Hz, 2H}\)) (Fig. S2).
2.7 Photocatalysts and cobaloxime complexes:

**Fig. S2.** $^1$H NMR of a mixture of D$_2$-1a and 1a

**Fig. S3.** Photocatalysts and cobaloxime catalysts
3. Mechanism studies

3.1 Cyclic voltammetry (CV) experiments:
A CH$_3$CN solution (3×10$^{-4}$ M) of 1a and a CH$_3$CN solution (3×10$^{-4}$ M) of 1-tetralone 1a with BF$_3$•Et$_2$O (3×10$^{-4}$ M) was prepared with NBu$_4$PF$_6$ (0.1 M) as the supporting electrolyte respectively. The cyclic voltammogram was obtained using a glassy carbon as working electrode, a Pt strip as counter electrode, and a saturated calomel electrode as reference electrode. Scan rate = 0.1 V/s, range from 0 V to 3.0 V.

![Cyclic voltammetry spectra of 1a](image1)

**Fig. S4.** Cyclic voltammetry spectra of 1a shows an oxidative potential at 2.51 V vs. SCE in anhydrous CH$_3$CN.

![Cyclic voltammetry spectra of 1a with BF$_3$•Et$_2$O](image2)

**Fig. S5.** Cyclic voltammetry spectra of 1a with BF$_3$•Et$_2$O shows an oxidative potential at 2.44 V vs. SCE in anhydrous CH$_3$CN.
3.2 Spectroscopy experiments

Fig. S6. Fluorescence quenching spectra of QuCN⁺ (2×10⁻⁵ M) with different concentrations of 1a in degassed CH₃CN with excitation at 340 nm.

Fig. S7. Fluorescence quenching spectra of QuCN⁺ (2×10⁻⁵ M) with different concentrations of Co(dmgBF₂)(MeCN)₂ (II) in degassed CH₃CN with excitation at 340 nm.
Fig. S8. UV-Vis absorption spectra of QuCN$^+$ (2×10$^{-5}$ M), Co(dmgBF$_2$)$_2$(MeCN)$_2$ (II) (1.2 × 10$^{-5}$ M) and 1a (4×10$^{-4}$ M), respectively in degassed CH$_3$CN; QuCN$^+$ (2×10$^{-5}$ M) in the presence of Co(dmgBF$_2$)$_2$(MeCN)$_2$ (II) (1.2 × 10$^{-5}$ M) or 1a (4×10$^{-4}$ M), respectively in degassed CH$_3$CN; QuCN$^+$ (2×10$^{-5}$ M) in conjugation with Co(dmgBF$_2$)$_2$(MeCN)$_2$ (II) (1.2 × 10$^{-5}$ M) and 1a (4×10$^{-4}$ M) in degassed CH$_3$CN.

Fig. S9. UV-Vis absorption spectra of 1a (4×10$^{-4}$ M) and 2a (2×10$^{-4}$ M) in anhydrous CH$_3$CN.
Fig. S10. UV-Vis absorption spectra of 1a (4×10^{-4} M), 1a (4×10^{-4} M) with BF$_3$•Et$_2$O (8×10^{-5} M), 2a (2×10^{-4} M) and 2a (2×10^{-4} M) with BF$_3$•Et$_2$O (4×10^{-5} M) in anhydrous CH$_3$CN.

Fig. S11. UV-Vis absorption spectra of QuCN$^+$ (2×10^{-4} M) and 2a (2×10^{-3} M) in anhydrous CH$_3$CN.
3.3 Kinetics isotopic effect experiment

A Pyrex tube equipped with a stir-bar was charged with QuCN+ (1×10^{-2} mmol, 2.69 mg), Co(dmgBF$_2$)$_2$(CH$_3$CN)$_2$ (II) (6×10^{-3} mmol. 2.8 mg) and CH$_3$CN (5 mL). After degassing by the argon, 1a (0.10 mmol, 13.5 µL) and D$_2$-1a (0.1 mmol, 13.5 µL) were added and the pinholes were sealed by paraffin. The reaction mixture was irradiation at room temperature using a 500 W medium pressure Hanovia mercury lamp with a glass water cooling jacket (λ > 300 nm) for 1 h. And then the solvent of the reaction mixture was removed, the pure product was obtained by flash column chromatography on silica gel (eluent: petroleum ether/EtOAc = 20:1) to afford 17% combined products. Comparing the $^1$H NMR spectra of 2a and D$_2$-2a, we found the ratio of 2a: D$_2$-2a was 0.55:0.45. So the KIE value ($k_H/k_D = 1.2$) was determined to be 1.2 (Fig. S12-S13).

Fig. S12. $^1$H NMR of 2a
Fig. S13. $^1$H NMR of a mixture of D$_2$-2a and 2a
4. **Substrate synthesis**

**General procedures (1)**

![Reaction Scheme]

**Methyl 5-oxo-5,6,7,8-tetrahydronaphthalene-2-carboxylate (1h)**

Methyl 5-oxo-5,6,7,8-tetrahydronaphthalene-2-carboxylate was synthesized according the previous report. A 10 mL round-bottom flask was charged with 5-oxo-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid (190.2 mg, 1 mmol), oxone (0.3 mmol) and methanol (3 mL). The mixture was stirred at 65 °C. After completion of the reaction that was confirmed by thin layer chromatography the crude mixture was cooled to room temperature, filtered and purified by column chromatography using silica gel (200-300 meshes silica gel) with ethyl acetate and petroleum ether as an eluent to afford the title compound as a white solid (150.2 mg, 73% yield).

**1H NMR (400 MHz, CDCl₃, ppm)** δ: 8.06 (d, J = 8.0 Hz, 1H), 7.92 (m, 2H), 3.92 (s, 3H), 3.01 (t, J = 6.0 Hz, 2H), 2.72 – 2.64 (t, J = 6.4 Hz, 2H), 2.19 – 2.11 (m, 2H).

**13C NMR (101 MHz, CDCl₃, ppm)** δ: 197.66, 166.32, 144.35, 135.53, 133.93, 130.19, 127.39, 127.25, 52.41, 39.12, 29.60, 23.07. HRMS (EI): m/z calculated for C₁₂H₁₂O₃ [M]+ 204.0786, found 204.0788.

**Ethyl 5-oxo-5,6,7,8-tetrahydronaphthalene-2-carboxylate (1i)**

Ethyl 5-oxo-5,6,7,8-tetrahydronaphthalene-2-carboxylate was synthesized according the previous report. A 10 mL round-bottom flask was charged with 5-oxo-5,6,7,8-tetrahydronaphthalene-2-carboxylic acid (285.3 mg, 1.5 mmol), oxone (0.45 mmol) and alcohol (5 mL). The mixture was stirred at 65 °C. After completion of the reaction that was confirmed by thin layer chromatography the crude mixture was cooled to room temperature, filtered and purified by column chromatography using silica gel (200-300 meshes silica gel) with ethyl acetate and petroleum ether as an eluent to afford the title compound as a colorless oil (192.3 mg, 59%).

**1H NMR (400 MHz, CDCl₃, ppm)** δ: 8.07 (d, J = 8.0 Hz, 1H), 7.93 (m, 2H), 4.40 (q, J = 7.1 Hz, 2H), 3.02 (t, J = 6.1 Hz, 2H), 2.75 – 2.61 (t, J = 6.4 Hz, 2H), 2.22 – 2.10 (m, 2H), 1.40 (t, J = 7.1 Hz, 3H).

Butyl 5-oxo-6,7,8-tetrahydronaphthalene-2-carboxylate (1j)

Butyl 5-oxo-6,7,8-tetrahydronaphthalene-2-carboxylate was synthesized according to the previous report. A 10 mL round-bottom flask was charged with 5-oxo-6,7,8-tetrahydronaphthalene-2-carboxylic acid (285.3 mg, 1.5 mmol), oxone (0.45 mmol) and n-butyl alcohol (5 mL). The mixture was stirred at 65 °C. After completion of the reaction that was confirmed by thin layer chromatography the crude mixture was cooled to room temperature, filtered and purified by column chromatography using silica gel (200-300 meshes silica gel) with ethyl acetate and petroleum ether as an eluent to afford the title compound as a colorless oil (142.6 mg, 38%).

\[ \text{1H NMR (400 MHz, CDCl}_3, \text{ppm)} \delta: 8.07 (d, J = 8.5 Hz, 1H), 7.92 (m, 2H), 4.33 (t, J = 6.6 Hz, 2H), 3.02 (t, J = 6.0 Hz, 2H), 2.73 – 2.64 (m, 2H), 2.21 – 2.09 (m, 2H), 1.80 – 1.69 (m, 2H), 1.55 – 1.41 (m, 2H), 0.98 (t, J = 7.4 Hz, 3H). \]

\[ \text{13C NMR (101 MHz, CDCl}_3, \text{ppm)} \delta: 197.8, 166.0, 144.4, 135.56, 134.45, 130.21, 127.47, 127.35, 65.38, 39.24, 30.82, 29.73, 23.17, 19.35, 18.38. \]

HRMS (EI): m/z calculated for C_{15}H_{18}O_3 [M + H]^+ 247.1329, found 247.1322.

Isopropyl 5-oxo-6,7,8-tetrahydronaphthalene-2-carboxylate (1k)

Isopropyl 5-oxo-6,7,8-tetrahydronaphthalene-2-carboxylate was synthesized according to the previous report. To a 10 mL round-bottom flask, sulfuric acid (98%, 0.94 mmol, 50 µL) was added to a well-stirred mixture of 5-oxo-6,7,8-tetrahydronaphthalene-2-carboxylic acid (0.8 mmol, 152.16 mg), isopropanol (137 µL), and acetonitrile (7 mL) at room temperature, and the temperature was maintained at 80–85°C for 18 h. The reaction mixture was concentrated under reduced pressure and purified over silica gel using ethyl acetate–petroleum ether to afford the title compound as a colorless oil (142 mg, 62%).

\[ \text{1H NMR (400 MHz, CDCl}_3, \text{ppm)} \delta: 8.07 (d, J = 8.5 Hz, 1H), 7.93 (m, 2H), 5.38 – 5.15 (m, 1H), 3.02 (t, J = 6.0 Hz, 2H), 2.69 (t, J = 6.5 Hz, 2H), 2.23 – 2.07 (m, 2H), 1.38 (d, J = 6.2 Hz, 6H). \]

\[ \text{13C NMR (101 MHz, CDCl}_3, \text{ppm)} \delta: 197.95, 165.52, 144.40, 135.52, 134.90, 130.20, 127.51, 127.35, 69.09, 39.28, 29.76, 23.22, 22.02. \]

HRMS (EI): m/z calculated for C_{14}H_{16}O_3 [M + H]^+ 233.1172, found 233.1167.

General procedures (2)
4-Phenyl-3,4-dihydronaphthalen-1(2H)-one (1p)

4-phenyl-3,4-dihydronaphthalen-1(2H)-one was synthesized according the previous report.\textsuperscript{7} \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}, ppm) $\delta$: 8.12 (d, $J = 7.7$ Hz, 1H), 7.53 – 7.21 (m, 5H), 7.12 (d, $J = 7.3$ Hz, 2H), 6.99 (d, $J = 7.7$ Hz, 1H), 4.31 (dd, $J = 7.7$, 4.6 Hz, 1H), 2.69 (m, 2H), 2.48 (dd, $J = 8.6$, 4.5 Hz, 1H), 2.31 (m, $J = 13.3$, 8.7, 4.5 Hz, 1H).

6-Fluoro-4-(4-fluorophenyl)-3,4-dihydronaphthalen-1(2H)-one (1q)

6-fluoro-4-(4-fluorophenyl)-3,4-dihydronaphthalen-1(2H)-one was synthesized according the previous report.\textsuperscript{7} \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}, ppm) $\delta$: 8.14 (dd, $J = 8.7$, 6.1 Hz, 1H), 7.20 – 6.95 (m, 5H), 6.60 (dd, $J = 9.6$, 2.1 Hz, 1H), 4.25 (dd, $J = 8.6$, 4.4 Hz, 1H), 2.85 – 2.57 (m, 2H), 2.44 (m, 1H), 2.34 – 2.19 (m, 1H).

7-Fluoro-4-(3-fluorophenyl)-3,4-dihydronaphthalen-1(2H)-one (1r)

7-fluoro-4-(3-fluorophenyl)-3,4-dihydronaphthalen-1(2H)-one was synthesized according the previous report.\textsuperscript{7} \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}, ppm) $\delta$: 7.70 (dd, $J = 9.0$, 2.6 Hz, 1H), 7.39 – 7.04 (m, 5H), 6.66 (d, $J = 9.9$ Hz, 1H), 4.21 (dd, $J = 7.4$, 4.8 Hz, 1H), 2.62 (m, 2H), 2.40 (m, 1H), 2.21 (m, 1H).

6-Chloro-4-(4-chlorophenyl)-3,4-dihydronaphthalen-1(2H)-one (1s)

6-chloro-4-(4-chlorophenyl)-3,4-dihydronaphthalen-1(2H)-one was synthesized according the
previous report.\textsuperscript{7} \textsuperscript{1}H NMR (400 MHz, Acetone, ppm) \( \delta \): 8.02 (d, \( J = 8.3 \) Hz, 1H), 7.53 – 7.35 (m, 3H), 7.28 – 7.20 (d, \( J = 8.0 \) Hz, 2H), 6.98 (s, 1H), 4.46 (dd, \( J = 7.8, 4.3 \) Hz, 1H), 2.75 – 2.57 (m, 2H), 2.46 (m, 1H), 2.34 (m, 1H).

6-Bromo-4-(4-bromophenyl)-3,4-dihyronaphthalen-1(2H)-one (1t)

![Chemical Structure]

6-bromo-4-(4-bromophenyl)-3,4-dihydronaphthalen-1(2H)-one was synthesized according to the previous report.\textsuperscript{7} \textsuperscript{1}H NMR (400 MHz, CDCl\(_3\), ppm) \( \delta \): 7.96 (d, \( J = 8.4 \) Hz, 1H), 7.53 – 7.41 (m, 3H), 7.10 (s, 1H), 6.99 (d, \( J = 8.2 \) Hz, 2H), 4.22 (dd, \( J = 8.1, 4.5 \) Hz, 1H), 2.75 – 2.55 (m, 2H), 2.43 (m, 1H), 2.29 – 2.19 (m, 1H).
5. Characterization of products (2a – 2u)

Naphthalen-1-ol (2a)

White solid. Isolated yield: 37.5 mg, 65%. Petroleum ether/EtOAc=20/1-10/1.

\[^1\text{H} \text{NMR} \ (400 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: 8.19 \ (m, 1H), 7.89 \text{ –} 7.73 \ (m, 1H), 7.57 \text{ –} 7.38 \ (m, 3H), 7.32 \ (t, J = 7.8 \text{ Hz, 1H}), 6.83 \ (d, J = 7.4 \text{ Hz, 1H}), 5.26 \ (br, 1H).\]

\[^{13}\text{C} \text{NMR} \ (101 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: 151.4, 134.9, 127.8, 126.6, 126.0, 125.4, 124.4, 121.6, 120.9, 108.8.\]

HRMS (ESI): m/z calculated for C\(_{10}\)H\(_8\)O [M - H] 143.0502 found 143.0498.

6-Fluoronaphthalen-1-ol (2b)

White solid. Isolated yield: 32.3 mg, 50%. Petroleum ether/EtOAc=20/1-15/1.

\[^1\text{H} \text{NMR} \ (400 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: 7.79 \ (m, 2H), 7.43 \ (d, J = 8.3 \text{ Hz, 1H}), 7.36 \text{ –} 7.17 \ (m, 2H), 6.83 \ (d, J = 7.4 \text{ Hz, 1H}), 5.31 \ (br, 1H).\]

\[^{13}\text{C} \text{NMR} \ (101 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: 160.6 \ (d, J = 245.1 \text{ Hz}), 151.1 \ (d, J = 5.4 \text{ Hz}), 131.9 \ (s), 130.2 \ (d, J = 8.8 \text{ Hz}), 125.3 \ (d, J = 8.9 \text{ Hz}), 125.1 \ (d, J = 2.5 \text{ Hz}), 120.7 \ (s), 116.9 \ (d, J = 25.5 \text{ Hz}), 109.5 \ (s), 105.7 \ (d, J = 22.3 \text{ Hz}).\]

\[^{19}\text{F} \text{NMR} \ (377 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: -114.69.\]

HRMS (ESI): m/z calculated for C\(_{10}\)H\(_7\)FO [M - H] 161.0408 found 161.0396.

7-Fluoronaphthalen-1-ol (2c)

White solid. Isolated yield: 29.0 mg, 45%. Petroleum ether/EtOAc=15/1-10/1.

\[^1\text{H} \text{NMR} \ (400 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: 7.84 \text{ –} 7.74 \ (m, 2H), 7.43 \ (d, J = 8.3 \text{ Hz, 1H}), 7.31 \text{ –} 7.20 \ (m, 2H), 6.83 \ (d, J = 7.4 \text{ Hz, 1H}), 5.25 \ (br, 1H).\]

\[^{13}\text{C} \text{NMR} \ (101 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: 160.6 \ (d, J = 245.1 \text{ Hz}), 151.1 \ (d, J = 5.4 \text{ Hz}), 131.9 \ (s), 130.2 \ (d, J = 8.8 \text{ Hz}), 125.3 \ (d, J = 8.9 \text{ Hz}), 125.1 \ (d, J = 2.5 \text{ Hz}), 120.7 \ (d, J = 1.0 \text{ Hz}), 117.0 \ (d, J = 25.4 \text{ Hz}), 109.5 \ (s), 105.7 \ (d, J = 22.3 \text{ Hz}).\]

\[^{19}\text{F} \text{NMR} \ (377 \text{ MHz, CDCl}_3, \text{ ppm}) \delta: -114.68.\]

HRMS (ESI): m/z calculated for C\(_{10}\)H\(_7\)FO [M - H] 161.0481, found 161.0395.
6,7-Difluoronaphthalen-1-ol (2d)

White solid. Isolated yield: 31.1 mg, 43%. Petroleum ether/EtOAc=20/1-15/1.
$^1$H NMR (400 MHz, CDCl$_3$, ppm) $\delta$: 7.92 (dd, $J = 11.5, 8.2$ Hz, 1H), 7.51 (dd, $J = 11.0, 7.9$ Hz, 1H), 7.37 – 7.21 (m, 2H), 6.78 (d, $J = 7.3$ Hz, 1H), 5.36 (br, 1H).
$^{13}$C NMR (101 MHz, CDCl$_3$, ppm) $\delta$: 151.2 (dd, $J = 5.2, 1.9$ Hz), 150.7 (dd, $J = 250.2, 14.8$ Hz), 149.7 (dd, $J = 249.4, 15.2$ Hz) 131.9 (d, $J = 7.6$ Hz), 126.5 (d, $J = 2.4$ Hz), 121.3 (d, $J = 7.1$ Hz), 120.0 (dd, $J = 4.9, 1.9$ Hz), 113.5 (d, $J = 16.7$ Hz), 108.8 (d, $J = 18.3$ Hz), 108.8 (d, $J = 2.2$ Hz).
$^{19}$F NMR (377 MHz, CDCl$_3$, ppm) $\delta$: -136.91 (d, $J = 20.7$ Hz), -137.51 (d, $J = 20.7$ Hz).
HRMS (ESI): m/z calculated for C$_{10}$H$_6$F$_2$O [M -H] $^{+}$ 179.0314 found 179.0305.

5-Chloronaphthalen-1-ol (2e)

White solid. Isolated yield: 20.3 mg, 28%. Petroleum ether/EtOAc=15/1-10/1.
$^1$H NMR (400 MHz, CDCl$_3$, ppm) $\delta$: 8.15 (d, $J = 8.5$ Hz, 1H), 7.87 (d, $J = 8.6$ Hz, 1H), 7.59 (d, $J = 7.4$ Hz, 1H), 7.47 – 7.33 (m, 2H), 6.88 (d, $J = 7.5$ Hz, 1H), 5.35 (br, 1H).
$^{13}$C NMR (101 MHz, CDCl$_3$, ppm) $\delta$: 151.8, 132.4, 132.1, 127.1, 127.0, 125.9, 125.1, 121.1, 117.4, 109.7.
HRMS (ESI): m/z calculated for C$_{10}$H$_7$ClO [M -H] $^{+}$ 177.0113, found 177.0103.

6-Chloronaphthalen-1-ol (2f)

White solid. Isolated yield: 23.9 mg, 34%. Petroleum ether/EtOAc=20/1-10/1.
$^1$H NMR (400 MHz, CDCl$_3$, ppm) $\delta$: 8.14 (d, $J = 9.0$ Hz, 1H), 7.79 (s, 1H), 7.42 (d, $J = 9.0$ Hz, 1H), 7.37 – 7.29 (m, 2H), 6.80 (d, $J = 6.5$ Hz, 1H), 5.27 (br, 1H).
$^{13}$C NMR (101 MHz, CDCl$_3$, ppm) $\delta$: 151.6, 135.6, 132.6, 127.3, 126.4, 126.2, 123.8, 122.8, 120.0, 109.0.
HRMS (ESI): m/z calculated for C$_{10}$H$_7$ClO [M -H] $^{+}$ 177.0113, found 177.0101.

7-Chloronaphthalen-1-ol (2g)

White solid. Isolated yield: 16.5 mg, 22%. Petroleum ether/EtOAc=15/1-10/1.
Methyl-5-hydroxy-2-naphthoate (2h)

![Methyl-5-hydroxy-2-naphthoate](image)

White solid. Isolated yield: 38.5 mg, 48%. Petroleum ether/EtOAc=20/1-10/1.

$^1$H NMR (400 MHz, CDCl$_3$, ppm) $\delta$: 8.17 (s, 1H), 7.72 (d, $J = 8.8$ Hz, 1H), 7.41 (m, 2H), 7.35 – 7.19 (m, 1H), 6.81 (d, $J = 7.4$ Hz, 1H), 5.23 (br, 1H).

$^{13}$C NMR (101 MHz, CDCl$_3$, ppm) $\delta$: 150.7, 133.1, 131.3, 129.4, 127.5, 126.2, 125.2, 121.2, 120.7, 109.6.

HRMS (ESI): m/z calculated for C$_{10}$H$_7$ClO [M -H] $^{177.0113}$, found $^{177.0099}$

Ethyl 5-hydroxy-2-naphthoate (2i)

![Ethyl-5-hydroxy-2-naphthoate](image)

White solid. Isolated yield: 32.2 mg, 37%. Petroleum ether/EtOAc=20/1-15/1.

$^1$H NMR (400 MHz, CD$_3$COCD$_3$, ppm) $\delta$: 9.26 (br, 1H), 8.55 (s, 1H), 8.32 (d, $J = 8.8$ Hz, 1H), 8.00 (d, $J = 8.8$ Hz, 1H), 7.53 (d, $J = 8.2$ Hz, 1H), 7.37 (t, $J = 7.9$ Hz, 1H), 7.25 (d, $J = 7.7$ Hz, 1H), 6.96 (d, $J = 7.5$ Hz, 1H), 6.04 (br, 1H), 4.00 (s, 3H).

$^{13}$C NMR (101 MHz, CD$_3$COCD$_3$, ppm) $\delta$: 167.0, 154.1, 135.0, 131.1, 129.1, 128.2, 127.8, 124.7, 122.4, 121.4, 111.4, 61.6, 14.7.

HRMS (ESI): m/z calculated for C$_{12}$H$_{10}$O$_3$ [M -H] $^{201.0557}$, found $^{201.0548}$.

Butyl 5-hydroxy-2-naphthoate (2j)

![Butyl-5-hydroxy-2-naphthoate](image)

White solid. Isolated yield: 49.1 mg, 50%. Petroleum ether/EtOAc=20/1-10/1.

$^1$H NMR (400 MHz, CDCl$_3$, ppm) $\delta$: 8.58 (s, 1H), 8.28 (d, $J = 8.8$ Hz, 1H), 8.07 (d, $J = 8.8$ Hz, 1H), 7.53 (d, $J = 8.2$ Hz, 1H), 7.36 (t, $J = 7.8$ Hz, 1H), 6.98 (d, $J = 7.5$ Hz, 1H), 6.47 (br, 1H), 4.42 (t, $J = 6.6$ Hz, 2H), 1.90 – 1.72 (m, 2H), 1.62 – 1.42 (m, 2H), 1.01 (t, $J = 7.4$ Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$, ppm) $\delta$: 167.5, 151.8, 134.0, 130.8, 128.2, 126.9, 126.7, 124.4, 122.4, 121.9, 111.0, 65.4, 30.9, 19.4, 13.9.

HRMS (ESI): m/z calculated for C$_{15}$H$_{16}$O$_3$ [M -H] $^{243.1022}$, found $^{243.1022}$. 
Isopropyl 5-hydroxy-2-naphthoate (2k)

White solid. Isolated yield: 36.5 mg, 40%. Petroleum ether/EtOAc=20/1-10/1.

\( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): 8.57 (s, 1H), 8.28 (d, \( J = 8.8 \) Hz, 1H), 8.07 (d, \( J = 8.8 \) Hz, 1H), 7.53 (d, \( J = 8.2 \) Hz, 1H), 7.36 (t, \( J = 7.9 \) Hz, 1H), 6.98 (d, \( J = 7.5 \) Hz, 1H), 6.47 (br, 1H), 5.45 – 5.27 (m, 1H), 1.44 (d, \( J = 6.3 \) Hz, 6H).

\( ^{13}C \) NMR (101 MHz, CDCl\(_3\), ppm): 167.1, 151.9, 134.0, 130.8, 128.5, 126.9, 126.7, 124.5, 122.4, 121.8, 111.0, 69.1, 22.1.

HRMS (ESI): m/z calculated for C\(_{14}\)H\(_{14}\)O\(_3\) [M -H] 229.0870 found 229.0864.

2-Methylnaphthalen-1-ol (2m)

Colorless oil. Isolated yield: 30.5 mg, 48%. Petroleum ether/EtOAc=20/1-10/1.

\( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): 8.13 (d, \( J = 8.2 \) Hz, 1H), 7.78 (d, \( J = 8.0 \) Hz, 1H), 7.52 – 7.35 (m, 3H), 7.25 (d, \( J = 8.1 \) Hz, 1H), 5.10 (br, 1H), 2.42 (s, 3H).

\( ^{13}C \) NMR (101 MHz, CDCl\(_3\), ppm): 148.7, 133.5, 129.1, 127.8, 125.5, 125.4, 124.4, 121.0, 120.3, 116.4, 15.8.

HRMS (ESI): m/z calculated for C\(_{11}\)H\(_{10}\)O [M -H] 157.0659, found 157.0648.

4-Methylnaphthalen-1-ol (2n)

White solid. Isolated yield: 45.0 mg, 71%. Petroleum ether/EtOAc=20/1-10/1.

\( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): 8.23 (d, \( J = 8.0 \) Hz, 1H), 7.96 (d, \( J = 8.5 \) Hz, 1H), 7.60 – 7.44 (m, 2H), 7.14 (d, \( J = 7.5 \) Hz, 1H), 6.73 (d, \( J = 7.5 \) Hz, 1H), 5.16 (br, 1H), 2.63 (s, 3H).

\( ^{13}C \) NMR (101 MHz, CDCl\(_3\), ppm): 150.0, 133.7, 126.8, 126.4, 126.2, 125.1, 124.8, 124.4, 122.2, 18.9.


4-Phenylnaphthalen-1-ol (2p)

White solid. Isolated yield: 51.0 mg, 58%. Petroleum ether/EtOAc=50/1-20/1.
$^1$H NMR (400 MHz, CDCl$_3$, ppm) $\delta$: $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.26 (d, $J$ = 8.3 Hz, 1H), 7.87 (d, $J$ = 8.4 Hz, 1H), 7.57 – 7.35 (m, 7H), 7.25 (d, $J$ = 7.7 Hz, 1H), 6.85 (d, $J$ = 7.7 Hz, 1H), 5.32 (br, 1H).

$^{13}$C NMR (101 MHz, CDCl$_3$, ppm) $\delta$: 151.1, 140.9, 133.4, 132.8, 130.4, 128.4, 127.1, 127.0, 126.7, 126.1, 125.3, 124.0, 122.0, 108.3.

HRMS (ESI): m/z calculated for C$_{16}$H$_{12}$O [M - H] 219.0815, found 219.0807.

6-Fluoro-4-(4-fluorophenyl)naphthalen-1-ol (2q)

White solid. Isolated yield: 20.5 mg, 40%. Petroleum ether/EtOAc=100/1-70/1.

$^1$H NMR (400 MHz, CD$_2$COCD$_3$, ppm) $\delta$: 9.35 (br, 1H), 8.39 (dd, $J$ = 9.1, 6.2 Hz, 1H), 7.48 (dd, $J$ = 8.1, 5.8 Hz, 2H), 7.41 – 7.23 (m, 5H), 6.98 (d, $J$ = 7.8 Hz, 1H).

$^{13}$C NMR (101 MHz, CD$_2$COCD$_3$, ppm) $\delta$: 162.9 (d, $J$ = 244.9 Hz), 162.2 (d, $J$ = 243.9 Hz), 154.0, 137.6 (d, $J$ = 3.3 Hz), 134.7 (d, $J$ = 8.7 Hz), 132.6 (d, $J$ = 8.0 Hz), 130.7 (d, $J$ = 5.3 Hz), 129.7, 126.5 (d, $J$ = 9.3 Hz), 123.0, 116.0 (d, $J$ = 21.5 Hz), 115.3 (d, $J$ = 25.3 Hz), 109.2 (d, $J$ = 22.1 Hz), 108.1 (d, $J$ = 2.1 Hz).

$^{19}$F NMR (377 MHz, CD$_2$COCD$_3$, ppm) $\delta$: -115.10, -117.29.

HRMS (ESI): m/z calculated for C$_{16}$H$_{10}$F$_2$O [M - H] 255.0627, found 255.0622.

7-Fluoro-4-(3-fluorophenyl)naphthalen-1-ol (2r)

White solid. Isolated yield: 12.8 mg, 25%. Petroleum ether/EtOAc=100/1-50/1.

$^1$H NMR (400 MHz, CDCl$_3$, ppm) $\delta$: 7.89 (dd, $J$ = 10.3, 2.4 Hz, 1H), 7.84 (dd, $J$ = 9.3, 5.5 Hz, 1H), 7.43 (dd, $J$ = 14.3, 7.5 Hz, 1H), 7.30 – 7.04 (m, 5H), 6.89 (d, $J$ = 7.7 Hz, 1H), 5.52 (br, 1H).

$^{13}$C NMR (101 MHz, CDCl$_3$, ppm) $\delta$: 162.9 (d, $J$ = 236.1 Hz), 160.5 (d, $J$ = 235.9 Hz), 151.0 (d, $J$ = 5.3 Hz), 142.9 (d, $J$ = 7.8 Hz), 132.2, 129.9 (d, $J$ = 8.5 Hz), 129.7, 128.5 (d, $J$ = 8.7 Hz), 126.2 (d, $J$ = 2.4 Hz), 126.1 (d, $J$ = 2.9 Hz), 125.7 (d, $J$ = 8.9 Hz), 117.3 (d, $J$ = 12.4 Hz), 117.0 (d, $J$ = 16.2 Hz), 114.2 (d, $J$ = 21.0 Hz), 109.1, 106.2 (d, $J$ = 22.2 Hz).

$^{19}$F NMR (377 MHz, CD$_2$COCD$_3$, ppm) $\delta$: -114.89, -117.16.

HRMS (ESI): m/z calculated for C$_{16}$H$_{10}$F$_2$O [M - H] 255.0627, found 255.0618.
6-Chloro-4-(4-chlorophenyl)naphthalen-1-ol (2s)

White solid. Isolated yield: 25.9 mg, 45%. Petroleum ether/EtOAc=100/1-80/1.
$^1$H NMR (400 MHz, CD$_3$COCD$_3$, ppm) $\delta$: 9.45 (br, 1H), 8.34 (d, $J$ = 9.0 Hz, 1H), 7.74 (s, 1H), 7.51 (m, 5H), 7.33 (d, $J$ = 7.8 Hz, 1H), 7.03 (d, $J$ = 7.8 Hz, 1H).
$^{13}$C NMR (101 MHz, CD$_3$COCD$_3$, ppm) $\delta$: 154.3, 140.1, 134.3, 133.7, 133.3, 132.7, 130.4, 130.0, 129.6, 126.3, 125.9, 124.8, 124.4, 109.3.
HRMS (ESI): m/z calculated for C$_{16}$H$_{10}$Cl$_2$O [M -H] $^-$ 287.0036, found 287.0035.

6-Bromo-4-(4-bromophenyl)naphthalen-1-ol (2t)

White solid. Isolated yield: 26.5 mg, 35%. Petroleum ether/EtOAc=100/1-80/1.
$^1$H NMR (400 MHz, CD$_3$COCD$_3$, ppm) $\delta$: 9.45 (br, 1H), 8.27 (d, $J$ = 9.0 Hz, 1H), 7.91 (s, 1H), 7.70 (d, $J$ = 8.2 Hz, 2H), 7.61 (d, $J$ = 9.0 Hz, 1H), 7.41 (d, $J$ = 8.2 Hz, 2H), 7.32 (d, $J$ = 7.8 Hz, 1H), 7.05 (d, $J$ = 7.8 Hz, 1H).
$^{13}$C NMR (101 MHz, CD$_3$COCD$_3$, ppm) $\delta$: 154.3, 140.5, 134.6, 133.0, 132.6, 130.4, 129.9, 128.9, 128.1, 126.0, 124.6, 121.8, 121.8, 109.5.
HRMS (ESI): m/z calculated for C$_{16}$H$_{10}$Br$_2$O [M -H] $^-$ 376.9005, found 376.9004.
6. Reference


7. NMR spectra for substrates
8. NMR spectra for products

[Diagram of NMR spectra for product 2a]

[Diagram of NMR spectra for product 2a]