Electronic Supplementary Information

**Iron-surfactant nanocomposite catalyzed benzylic oxidation in water**

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General

Unless otherwise indicated, all starting materials were obtained from commercial suppliers, and were used without further purification. Analytical thin-layer chromatography (TLC) was performed on Merck DC pre coated TLC plates with 0.25 mm Kieselgel 60 F_{254}. Visualization was performed with a 254 nm UV lamp. The $^1$H and $^{13}$C NMR spectra were recorded on a Bruker Avance-250 spectrometer in CDCl$_3$. Chemical shifts are expressed in parts per million ($\delta$) using residual solvent protons as internal standards ($\delta$ 7.26 for $^1$H, $\delta$ 77.0 for $^{13}$C). Coupling constants ($J$) are reported in Hertz (Hz). Splitting patterns are designated as s (singlet), d (doublet), t (triplet), m (multiplet). Combination gas chromatography and low resolution mass spectrometry was obtained on an Agilent 6890N Gas Chromatograph (30 m x 0.25 mm column with 0.25 $\mu$m HP-5MS coating, He carrier gas) and Agilent 5973 Mass Spectrometer (Ion source: EI+, 70eV, 230°C; interface: 300°C). IR spectra were obtained on a Bruker Alpha spectrometer on a single-reflection diamond ATR unit. All melting points were measured on Büchi 501 apparatus and are uncorrected. High-resolution mass spectra were recorded on an Agilent Technologies 6210 Time of Flight mass spectrometer.
Effect of TBHP loading and the role of SDS in different solvents.

Fluorene (1 mmol) TBHP (70% aq, 5 mmol); FeCl₃•6H₂O (0.02 mmol, 2%) 5 w/w% SDS solution in water or tBuOH (1 mL), at 50°C, % composition of the product determined by GC.

Fluorene (1 mmol) TBHP (70% aq, 5 mmol); FeCl₃•6H₂O (0.02 mmol, 2%) 5 w/w% SDS solution in water or tBuOH (1 mL), at 50°C. % composition of the product determined by GC.
The influence of the carbon chain length on the conversion

A screw capped vial with stir bar was charged with the 2 mol% FeCl₃, 2 mol% SDS, and water. Fluorene (1 eq.) was added followed by 5 eq. TBHP (70% aq.). The reaction vessel was closed, and stirred for 24 hours at 50°C. Samples were taken and analyzed by GC.
The effect of SDS amount on the conversion

A screw capped vial with stir bar was charged with the 2 mol% FeCl₃, x mol% SDS and water. Fluorene (1 eq.) was added followed by 5 eq. TBHP (70% aq.). The reaction vessel was closed, and stirred for 4 hours at 50°C. Samples were taken and analyzed by GC.
Addition of excess TBHP, FeCl$_3$/Fe$_2$(SO$_4$)$_3$, SDS after 2 hours

However, addition of 2 mol% iron salt was sufficient to reach full conversion of fluorene to fluorenone in short reaction time, the oxidation of ethylbenzene to acetophenone was not completed in the presence of either 2 mol% FeCl$_3$ or 2 mol% Fe$_2$(SO$_4$)$_3$ (60% and 80% conversions were obtained after 24 hours respectively). In order to reach full conversion of this less reactive substrate we added a second portion of additives after 2 hours into the reaction mixture. Addition of TBHP ensures the completion of the reaction in 24 hours, and joint addition of extra TBHP/FeCl$_3$ or TBHP/FeCl$_3$/SDS did not cause better acceleration.$^{[12]}$ Interestingly, reactivation of the catalyst system in case of Fe$_2$(SO$_4$)$_3$ did not occurred as significantly as in the case of FeCl$_3$. 

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A screw capped vial with stir bar was charged with the 2 mol% catalyst, 2 mol% SDS and water. Ethyl benzene or fluorene (1 eq.) was added followed by 5 eq. TBHP (70% aq.). The reaction vessel was closed, and stirred for 2 hours at 50°C. Then second portion of iron salt, SDS, TBHP was added, and stirring was continued for further 22 hours. Samples were taken and analyzed by GC.

**Oxygen evolution measurement**

When ethylbenzene substrate was in the reaction mixture of FeCl₃, SDS and TBHP in water the oxygen slowly absorbed back into the solution. Oxygen volume was measured with gas burette:

![Graph showing oxygen evolution experiments](image)

Oxygen evolution experiments. TBHP (70% aq, 5 mmol, 5 eq.); FeCl₃*6 H₂O (0.05 mmol, 5%), SDS (0.05 mmol, 5%), H₂O (1 ml) at 50°C. % composition of the product determined by GC. ■: FeCl₃+ SDS. ●: FeCl₃ ▲ FeCl₃+ SDS + ethylbenzene (1 mmol, 1 eq.), ● FeCl₃+ SDS + Fluorene (1 mmol, 1eq.).
The influence of sulfate, chloride ions

The effect of Na$_2$SO$_4$ using Fe$_2$(SO$_4$)$_3$ catalyst

The effect of NaCl using Fe$_2$(SO$_4$)$_3$ catalyst
A screw capped vial with stir bar was charged with 83 mg (0.5 mmol, 1 eq.) fluorene, the 2 mol% catalyst, 2.9 mg (0.01 mmol, 2 mol%) SDS and the appropriate salt (Na₂SO₄ or NaCl). 0.5 mL water was added followed by 323 µL TBHP (2.5 mmol, 5 eq., 70% aq.). The reaction vessel was closed, and stirred for 2 hours at 50°C. Samples were taken and analyzed with GC.
Preparation of starting materials

2-(phenylethynyl)-9H-fluorene¹

A round-bottom flask was charged with 292 mg (1 mmol, 1 eq.) 2-iodo-9H-fluorene, 21 mg (0.03 mmol, 3 mol%) bis(triphenylphosphine)palladium(II) dichloride, and 5.7 mg (0.03 mmol, 3 mol%) copper(I) iodide. The flask was fit with a rubber septum, and purged with argon. DIPA (4 mL) and 165 µL phenylacetylene (1.5 mmol, 1.5 eq.) was added via syringe and the reaction was stirred at 50 °C for 24 hours. The reaction mixture was filtered, than diethyl ether was added. The solution washed with 10 % HCl (aq.) and water, dried with MgSO₄, and concentrated under vacuo. The crude product was purified with column chromatography (in hexane-EtOAc).

White solid. 113 mg (0.42 mmol, yield: 42%) Rf: 0.65 (in hexane:EtOAc = 5:1) ¹H NMR (250 MHz, CDCl₃): δ = 7.82-7.76 (m, 3H), 7.64-7.57 (m, 4H), 7.46-7.33 (m, 5H), 3.92 (s, 2H) ppm. ¹³C NMR (62.5 MHz, CDCl₃): δ = 143.4, 143.1, 141.7, 141, 131.5, 130.4, 128.30, 128.08, 127.07, 126.83, 125.00, 123.39, 121.16, 120.12, 119.7, 90.2, 89.3, 36.7 ppm. $\nu_{\text{max}}$ 2919, 1451, 832, 753, 730, 670 cm⁻¹ MS (EI, 70 eV): m/z (%): 266 (100, [M⁺]), 189 (10), 133 (15). m.p.: 164-168°C

(E)-methyl 3-(9H-fluoren-2-yl)acrylate²

A round-bottom flask was charged with 584 mg (2 mmol, 1 eq.) 2-iodo-9H-fluorene, and 22.4 mg (0.1 mmol, 5 mol%) palladium(II) acetate. The flask was fitted with a rubber septum, and purged with argon. DMF (2mL), 418 µL TEA (3 mmol, 1.5 eq.) and 182 µL (3 mmol, 1.5 eq.) methyl acrylate was added via syringe and the reaction was stirred at 80 °C for 24 hours. After completion, the reaction mixture was filtered, and poured onto ice. The precipitated crude product was filtered, and purified by column chromatography (in hexane-EtOAc).
White solid. 309 mg (1.2 mmol, yield: 62%) Rf: 0.48 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta$ = 7.68-7.61 (m, 3H), 7.55 (s, 1H), 7.42 (t, 2H $J$ = 6.16 Hz), 7.30-7.19 (m, 2H), 6.36 (d, 1H $J$ = 15.95 Hz), 3.76 (s, 2H), 3.71 (s, 3H) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta$ = 168.1, 145.7, 144.4, 144.3, 144.2, 141.3, 133.3, 127.9, 127.9, 127.4, 125.6, 124.9, 120.8, 120.6, 117.1, 52.1, 37.2 ppm. $\nu_{max}$ 2946, 2928, 1709, 1636, 1322, 1169, 977, 836, 741 cm$^{-1}$

MS (EI, 70 eV): $m/z$ (%): 250 (100, [M$^+$]), 218 (75), 189 (86), 165 (42), 94 (45). m.p.: 134-137°C
**General procedure for Fe catalyzed benzylic oxidation:**

A test tube with a septum cap and a stir bar was charged with the catalyst, SDS and water. The starting material and TBHP was added with stirring. The reaction vessel was closed and the reaction was stirred for x hours at 50°C. After cooling to ambient temperature, the solution was washed with EtOAc, dried over magnesium sulfate and concentrated under vacuo. The crude product was purified with chromatography (in hexane/EtOAc).

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Catalyst</th>
<th>SDS/mol%</th>
<th>TBHP/ eq.</th>
<th>Time/h</th>
<th>Conversion in 5 h</th>
</tr>
</thead>
<tbody>
<tr>
<td>fluorene</td>
<td>2 mol% FeCl₃</td>
<td>2</td>
<td>5</td>
<td>2h</td>
<td>100 without SDS</td>
</tr>
<tr>
<td>2-ethyl tiophene</td>
<td>2 mol% Fe₂(SO₄)₃</td>
<td>2</td>
<td>5 + 5 after 24h</td>
<td>48h</td>
<td>25</td>
</tr>
<tr>
<td>9H-xanthene</td>
<td>2 mol% FeCl₃</td>
<td>2</td>
<td>5</td>
<td>24h</td>
<td>100 without SDS</td>
</tr>
<tr>
<td>9,10-dihydroanthracene</td>
<td>2 mol% Fe₂(SO₄)₃</td>
<td>2</td>
<td>5</td>
<td>24h</td>
<td>100 without SDS</td>
</tr>
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<td>2-(phenylethynyl)-9H-fluorene</td>
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<td>2</td>
<td>5</td>
<td>24h</td>
<td>50</td>
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<tr>
<td>(E)-methyl 3-(9H-fluoren-2-yl)acrylate</td>
<td>2 mol% Fe₂(SO₄)₃</td>
<td>2</td>
<td>5</td>
<td>24h</td>
<td>40</td>
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<tr>
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<td>2 mol% Fe₂(SO₄)₃</td>
<td>2</td>
<td>5</td>
<td>24h</td>
<td>100 without SDS</td>
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<tr>
<td>methyl 7-iodo-9H-fluorene-4-carboxylate</td>
<td>2 mol% Fe₂(SO₄)₃</td>
<td>2</td>
<td>5</td>
<td>24h</td>
<td>100 without SDS</td>
</tr>
<tr>
<td>diphenylmethane</td>
<td>2 mol% Fe₂(SO₄)₃</td>
<td>2</td>
<td>5</td>
<td>24h</td>
<td>93 without SDS</td>
</tr>
<tr>
<td>2-bromo-9H-fluorene</td>
<td>2 mol% Fe₂(SO₄)₃</td>
<td>2</td>
<td>5 + 4 after 5h</td>
<td>24h</td>
<td>90 without SDS</td>
</tr>
<tr>
<td>2,7-dibromo-9H-fluorene</td>
<td>2 mol% Fe₂(SO₄)₃</td>
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<td>5</td>
<td>24h</td>
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<tr>
<td>(methoxymethyl)benzene</td>
<td>2.5 mol% Fe₂(SO₄)₃</td>
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<td>24h</td>
<td>49 without SDS</td>
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<td>5</td>
<td>24h</td>
<td>58 without SDS</td>
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<td>N-(2-benzylphenyl)acetamide</td>
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<td>5</td>
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<td>5</td>
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<td>24h</td>
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<tr>
<td>butylbenzene</td>
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<td>24h</td>
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<td>24h</td>
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<td>1-ethyl-4-methoxybenzene</td>
<td>5 mol% FeCl₃</td>
<td>5</td>
<td>5 + 5 after 1.5h</td>
<td>2.5h</td>
<td>73 without SDS</td>
</tr>
<tr>
<td>isochroman</td>
<td>5 mol% FeCl₃</td>
<td>5</td>
<td>5</td>
<td>10 min</td>
<td>92 without SDS</td>
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<tr>
<td>1,3-dihydroisobenzofuran</td>
<td>5 mol% FeCl₃</td>
<td>5</td>
<td>5</td>
<td>10 min</td>
<td>100 without SDS</td>
</tr>
<tr>
<td>2-phenylacetonitrile</td>
<td>5 mol% FeCl₃</td>
<td>5</td>
<td>5</td>
<td>24h</td>
<td>100 without SDS</td>
</tr>
<tr>
<td>1-tosyl-1,2,3,4-tetrahydroquinoline</td>
<td>5 mol% Fe₂(SO₄)₃</td>
<td>5</td>
<td>5 + 1 after 8h</td>
<td>24h</td>
<td>22 without SDS</td>
</tr>
<tr>
<td>1-benzylindole</td>
<td>5 mol% Fe₂(SO₄)₃</td>
<td>5</td>
<td>5</td>
<td>24h</td>
<td>8 without SDS</td>
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<tr>
<td>2,3-cyclopentenopyridine</td>
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<td>5</td>
<td>5</td>
<td>24h</td>
<td>3 without SDS</td>
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<tr>
<td>1,2,3,4-tetrahydronaphthalene</td>
<td>5 mol% Fe₂(SO₄)₃</td>
<td>5</td>
<td>5</td>
<td>24h</td>
<td>67 without SDS</td>
</tr>
<tr>
<td>benzyl alcohol</td>
<td>5 mol% Fe₂(SO₄)₃</td>
<td>5</td>
<td>5</td>
<td>24h</td>
<td>93 without SDS</td>
</tr>
</tbody>
</table>

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Products

**Fluorene-9-one (2a)**

A conical test tube was charged with FeCl$_3$ (5.4 mg, 0.02 mmol, 2 mol%), SDS (5.8 mg, 0.02 mmol, 2 mol%), and 1 mL of water. Then fluorene (166 mg, 1 mmol) was added followed by TBHP (0.65 mL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 2 hours at 50 °C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Yellow solid 180 mg (1 mmol, quantitative yield). Rf: 0.50 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta$ = 7.45 (t, 8H, $J = 46.0$ Hz) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta$ = 193.8, 144.3, 134.6, 128.9, 124.1, 120.2 ppm. $\nu_{\text{max}}$ 1710, 1597, 1449, 1295, 1149, 916, 731 440 cm$^{-1}$ MS (EI, 70 eV): $m/z$ (%): 180 (100, [M$^+$]), 152 (50), 126 (15), 76 (25), 63 (10). m.p. 79.5-80 °C (lit.): 80-83 °C

**2-iodo-9H-fluoren-9-one (2b)**

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (1.6 mg, 0.006 mmol, 2 mol%), SDS (1.7 mg, 0.006 mmol, 2 mol%), and 0.34 mL of water. Then 2-iodo-9H-fluorene (100 mg, 0.3 mmol) was added followed by TBHP (0.19 mL, 1.5 mmol). The reaction vessel was closed with rubber septa, and stirred for 5 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Yellow solid 83 mg (0.27 mmol, yield: 79%). Rf: 0.51 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta$ = 7.90 (s, 1H), 7.76 (dd, 1H $J_1 = 1.58$ Hz, $J_2 = 7.74$ Hz), 7.61 (d, 1H $J = 7.42$ Hz) 7.48-7.43 (m, 2H), 7.34-7.28 (m, 1H), 7.21 (d, 1H $J = 7.74$ Hz) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta$ = 192.3, 143.6, 143.5, 143.0, 135.6, 134.9, 133.1, 129.5, 124.4, 121.9, 120.4, 93.9 ppm. $\nu_{\text{max}}$ 2921, 2851, 1713, 1587, 1437, 1403, 1254, 1184, 1106, 816, 758, 731, 654,
453 cm\(^{-1}\) MS (EI, 70 eV): \(m/z\) (%): 306 (75, [M\(^+\)]), 151 (100), 139 (25), 75 (30). m.p. 141-143°C (lit.): 142-144 °C

\[
\text{2-bromo-9H-fluoren-9-one (2d)}
\]

A conical test tube was charged with Fe\(_2\)(SO\(_4\))\(_3\) (16 mg, 0.04 mmol, 2 mol%), SDS (23 mg, 0.08 mmol, 4 mol%), and 2 mL of water. Then 2-bromo-9H-fluorene (488 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 4 hours at 50°C, then TBHP (1.0 ml, 8 mmol, 4 eq.) was added and the reaction mixture was stirred for further 20 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

Yellow solid 445 mg (1.72 mmol, yield: 86%). Rf: 0.77 (in hexane:EtOAc = 5:1) \(^1\)H NMR (250 MHz, CDCl\(_3\)): \(\delta = 7.59\) (d, 1H \(J = 1.74\)), 7.51 (d, 1H \(J = 7.42\)), 7.46 (dd, 1H \(J_1 = 1.90\) \(J_2 = 7.90\)), 7.38-7.35 (m, 2H), 7.24-7.16 (m, 2H) ppm. \(^{13}\)C NMR (62.5 MHz, CDCl\(_3\)): \(\delta = 192.7, 144.0, 143.3, 137.4, 136.1, 135.4, 134.0, 129.8, 127.8, 124.9, 123.3, 122.1, 120.8\) ppm. \(\nu_{\text{max}}\) 1714, 1592, 1441, 1185, 818, 733, 658, 456 cm\(^{-1}\) MS (EI, 70 eV): \(m/z\) (%): 258(80, [M\(^+\)]), 151(100), 75(20). m.p. 139-141 °C (lit.): 146-148 °C

\[
\text{2,7-dibromo-9H-fluoren-9-one (2e)}
\]

A conical test tube was charged with Fe\(_2\)(SO\(_4\))\(_3\) (16 mg, 0.04 mmol, 2 mol%), SDS (23 mg, 0.08 mmol, 4 mol%), and 2 mL of water. Then 2,7-dibromo-9H-fluorene (648 mg, 2 mmol) was added followed by TBHP (1.3 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 4 hours at 50°C, then TBHP (1 ml, mmol, 4 eq.) was added and the reaction mixture was stirred for further 20 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).
Yellow solid 645 mg (1.9 mmol, yield: 95%) Rf: 0.63 (in hexane:EtOAc = 5:1) 1H NMR (250 MHz, CDCl3): δ = 7.70 (s, 2H), 7.58 (dd, 2H, J1 = 1.74 Hz, J2 = 8 Hz), 7.34 (d, 2H J = 8.10 Hz) ppm. 13C NMR (62.5 MHz, CDCl3): δ = 190.8, 142.2, 137.4, 135.2, 127.7, 123.2, 121.8 ppm. νmax 3080, 1719, 1590, 1443, 1242, 1178, 1052, 902, 821, 680, 589, 470 cm⁻¹. MS (EI, 70 eV): m/z (%): 338 (75, [M⁺]), 231 (20), 150 (100), 98 (23), 75 (42). m.p.: 200-201°C (lit.): 203-205°C

**Methyl 7-iodo-9-oxo-9H-fluorene-4-carboxylate (2f)**

A conical test tube was charged with Fe2(SO4)3 (1.4 mg, 0.0034 mmol, 2 mol%), SDS (1 mg, 0.0034 mmol, 2 mol%), and 0.17 mL of water. Then methyl 7-iodo-9H-fluorene-4-carboxylate (60.6 mg, 0.17 mmol) was added followed by TBHP (0.11 mL, 0.85 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

Yellow solid 50.6 mg (0.14 mmol, yield: 80%). Rf: 0.50 (in hexane:EtOAc = 5:1) 1H NMR (250 MHz, CDCl3): δ = 8.07-7.78 (m, 5H), 7.37 (t, 1H J = 7.50 Hz), 3.99 (s, 3H) ppm. 13C NMR (62.5 MHz, CDCl3): δ = 191.5, 166.7, 143.5, 142.3, 136.5, 135.8, 134.7, 132.9, 129.0, 128.0, 127.5, 126.8, 95.4, 52.6 ppm. νmax 2923, 2851, 1717, 1579, 1433, 1382, 1356, 1305, 1242, 1193, 1175, 1137, 989, 833, 754, 740 cm⁻¹. MS (EI, 70 eV): m/z (%): 364 (98, [M⁺]), 333(42), 305 (40), 207 (60), 150 (100), 138 (25), 75 (27). m.p. 180°C HRMS calcd. for C15H9IO3 [M+H]+ 364.9669 found 364.9676

![2-(phenylethynyl)-9H-fluoren-9-one (2g)](image)

Yellow solid. 36 mg (0.13 mmol, yield: 64%) Rf: 0.51 (in hexane:EtOAc = 5:1) 1H NMR (250 MHz, CDCl3): δ = 7.69 (d, 1H J = 0.95 Hz), 7.58-7.51 (m, 2H), 7.47-7.37 (m, 5H), 7.30-7.17 (m, 4H) ppm. 13C NMR (62.5 MHz, CDCl3): δ = 193.0, 143.9, 143.6, 137.6, 134.8, 134.2, 130.5, 129.0, 128.0, 127.5, 126.8, 95.4, 52.6 ppm. νmax 3080, 1719, 1590, 1443, 1242, 1178, 1052, 902, 821, 680, 589, 470 cm⁻¹. MS (EI, 70 eV): m/z (%): 338 (75, [M⁺]), 231 (20), 150 (100), 98 (23), 75 (42). m.p.: 200-201°C (lit.): 203-205°C

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134.1, 131.6, 129.3, 128.5, 128.4, 127.2, 124.4, 124.1, 122.7, 120.6, 120.2, 91.0, 88.6 ppm. 
$\nu_{\text{max}}$ 2917, 2850, 1714, 1600, 755, 733, 688 cm$^{-1}$ MS (EI, 70 eV): $m/z$ (%): 280 (100, [M$^+$]), 250 (30), 207 (40), 140 (15). m.p.: 140-143 °C

(E)-methyl 3-(9-oxo-9H-fluoren-2-yl)acrylate (2h)

Yellow solid. 140 mg (0.53 mmol, yield: 53%) Rf: 0.29 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta$ = 7.74 (s, 1H), 7.64 – 7.47 8m, 6H), 7.31-7.28 (m, 1H), 6.42 (d, 1H), 3.79 (s, 3H) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta$ = 192.9, 167.0, 145.6, 143.6, 143.2, 135.3, 134.9, 134.8, 134.6, 129.6, 124.4, 122.7, 120.7, 118.5, 51.8 ppm. $\nu_{\text{max}}$ 3059, 2917, 1706, 1580, 1437, 1317, 1169, 1004, 840, 719 cm$^{-1}$ MS (EI, 70 eV): $m/z$ (%): 264 (100, [M$^+$]), 233 (98), 205 (40), 176 (75), 151 (42), 88 (45). m.p.: 169-171 °C

Acetophenone (2i)$^3$

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (20 mg, 0.05 mmol, 2.5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and 2 mL of water. Then ethylbenzene (245 µl, 212 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50 °C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (40 mg, 0.1 mmol, 5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and 2 mL of water. Then benzyl-alcohol (207.2 µl, 216.3 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).
Colorless oil 180 mg (1.5 mmol, yield: 75%, from ethylbenzene), 235 mg (1.96 mmol, yield: 98% from 1-phenyl-1-ethanol) Rf: 0.49 (in hexane:EtOAc = 5:1) ¹H NMR (250 MHz, CDCl₃): δ = 7.89-7.86 (m, 2H), 7.51-7.34 (m, 3H), 2.52 (s, 3H) ppm. ¹³C NMR (62.5 MHz, CDCl₃): δ = 198.2, 137.0, 133.0, 128.5, 26.5 ppm. νmax 2926, 2855, 1684, 1599, 1467, 1358, 1262, 954, 758, 688, 587 cm⁻¹ MS (EI, 70 eV): m/z (%): 120 (32, [M⁺]), 105 (100), 77 (45).

Propiophenone (2j)⁸
A conical test tube was charged with Fe₂(SO₄)₃ (5 mg, 0.0125 mmol, 2.5 mol%), SDS (14.4 mg, mmol, 5 mol%), and 355 µl of water. Then propylbenzene (139.4 µl, 120.18 mg, 1 mmol) was added followed by TBHP (645 µl, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50 °C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Colorless oil 56 mg (0.42 mmol, yield: 42%), Rf: 0.65 (in hexane:EtOAc = 5:1) ¹H NMR (250 MHz, CDCl₃): δ = 7.87 (d, 2H, J = 6.95 Hz), 7.48-7.34 (m, 3H, J = 7.42 Hz), 2.92 (q, 2H, J = 14.44 Hz), 1.14 (t, 3H, J = 7.20 Hz) ppm. ¹³C NMR (62.5 MHz, CDCl₃): δ = 200.2, 137.3, 133.3, 128.9, 128.3, 32.2, 8.6 ppm. νmax 2978, 2938, 1685, 1449, 1319, 1218, 1014, 950, 743, 689 cm⁻¹ MS (EI, 70 eV): m/z (%): 134 (15, [M⁺]), 105 (100), 77 (50), 51 (15).

Butyrophenone (2k)⁹
A conical test tube was charged with Fe₂(SO₄)₃ (5 mg, 0.0125 mmol, 2.5 mol%), SDS (14.4 mg, mmol, 5 mol%), and 355 µl of water. Then butylbenzene (156.1 µl, 134.2 mg, 1 mmol) was added followed by TBHP (645 µl, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).
Colorless oil 63.8 mg (0.43 mmol, yield: 43%), Rf: 0.67 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta = 7.86$ (d, 2H, $J = 8.53$ Hz), 8.16 (m, 3H), 2.85 (t, 2H, $J = 7.27$ Hz), 1.68 (d, 2H, $J = 22.00$ Hz), 0.92 (t, 3H, $J = 7.42$ Hz) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta =$ 200.8, 137.5, 133.2, 128.9, 18.4, 40.9, 18.1, 14.3 ppm. $\nu_{\text{max}}$ 2962, 2933, 2874, 1683, 1448, 1315, 1272, 1212, 1001, 753, 745, 689 cm$^{-1}$ MS (EI, 70 eV); $m/z$ (%): 148 (15, [M$^+$]), 120 (10), 105 (100), 77 (45), 51 (15).

![Methyl 5-oxo-5-phenylpentanoate (2l)](image)

**Methyl 5-oxo-5-phenylpentanoate (2l)**

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (5 mg, 0.0125 mmol, 2.5 mol%), SDS (14.4 mg, mmol, 5 mol%), and 355 $\mu$l of water. Then 5-phenylvaleric acid (192.2 mg, 1 mmol) was added followed by TBHP (645 $\mu$l, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 48 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Colorless oil 56 mg (0.42 mmol, yield: 42%), Rf: 0.36 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta = 7.88$ (d, 2H, $J = 7.11$ Hz), 7.52-7.35 (m, 3H), 3.61 (s, 3H), 2.98 (t, 2H, $J = 7.19$ Hz), 2.38 (t, 2H, $J = 7.19$ Hz), 2.00 (quint., 2H, $J = 7.30$) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta =$ 200.0, 174.1, 137.2, 133.5, 129.0, 128.4, 52.0, 37.8, 33.5, 19.7 ppm. $\nu_{\text{max}}$ 2951, 1732, 1682, 1448, 1437, 1366, 1209, 1175, 1149, 1015, 742, 690 cm$^{-1}$ MS (EI, 70 eV); $m/z$ (%): 206 (2, [M$^+$]), 175 (5), 147 (6), 133 (5), 120 (12), 105 (100), 77 (40).

![Methyl 4-heptanoylbenzoate (2m)](image)

**Methyl 4-heptanoylbenzoate (2m)**

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (5 mg, 0.0125 mmol, 2.5 mol%), SDS (14.4 mg, mmol, 5 mol%), and 355 $\mu$l of water. Then 4-heptylbenzoic acid (234.3 mg, 1 mmol) was added followed by TBHP (645 $\mu$l, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 48 hours at 50°C. After cooling to room temperature, the solution was washed
with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

White solid 100.8 mg (0.40 mmol, yield: 40%), Rf: 0.53 (in hexane:EtoAc = 5:1) \(^1\)H NMR (250 MHz, CDCl\(_3\)): \(\delta = 8.08\) (2H, J = 8.37 Hz), 7.97 (d, 2H, J = 8.53 Hz), 3.91 (s, 3H), 2.95 (t, 2H, J = 7.35 Hz), 1.70 (quint., 2H, J = 14.45 Hz), 1.36-1.25 (m, 6H), 0.85 (t, 3H, J = 6.64 Hz) ppm. \(^{13}\)C NMR (62.5 MHz, CDCl\(_3\)): \(\delta = 200.3, 166.6, 140.7, 134.0, 1301, 128.3, 52.8, 39.3, 32.0, 29.3, 24.5, 22.8, 14.4\) ppm. \(\nu_{\text{max}}\) 2957, 2929, 2871, 1720, 1675, 1435, 1278, 1239, 1194, 1108, 958, 764, 687 cm\(^{-1}\) MS (EI, 70 eV): \(m/z\) (%): 248 (3, [M\(^+\)]), 217 (5), 189 (15), 178 (80), 163 (100), 147 (20), 135 (25), 120 (10), 104 (15), 76 (10). m.p.: 73-75°C

\[\text{1-(4-methoxyphenyl)ethanone (2n)\(^{12}\)}\]

A conical test tube was charged with FeCl\(_3\) (27 mg, 0.1 mmol, 5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and mL of water. Then 1-ethyl-4-methoxybenzene (283 \(\mu\)l, 272 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 90 min at 50°C, then TBHP (1.29 ml, 10 mmol, 5 eq) was added and the reaction mixture was stirred for further 1 hour (2.5 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

White solid 115 mg (0.77 mmol, yield: 40%) Rf: 0.21 (in hexane:EtoAc = 5:1) \(^1\)H NMR (250 MHz, CDCl\(_3\)): \(\delta = 7.82\) (d, 2H J = 8.85 Hz), 6.81 (d, 2H, J = 8.85 Hz), 3.74 (s, 3H), 2.43 (s, 3H) ppm. \(^{13}\)C NMR (62.5 MHz, CDCl\(_3\)): \(\delta = 196.5, 163.3, 130.3, 113.4, 55.2, 26.1\) ppm. \(\nu_{\text{max}}\) 2965, 2842, 1666, 1597, 1356, 1247, 1019, 830, 575 cm\(^{-1}\) MS (EI, 70 eV): \(m/z\) (%): 150 (40, [M\(^+\)]), 135 (100), 107 (22), 92 (30), 77 (40). m.p. 32-35°C (lit.): 37-39°C

\[\text{1-(thiophen-2-yl)ethanone (2p)\(^{13}\)}\]

A conical test tube was charged with FeCl\(_3\) (16.2 mg, 0.06 mmol, 2 mol%), SDS (17.4 mg, 0.06 mmol, 2 mol%), and 3 mL of water. Then 2-ethylthiophene (340 \(\mu\)l, 224.3 mg, 3 mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 3 hours at 50°C. After cooling to room temperature, the solution
was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Brown oil 123 mg (0.98 mmol, yield: 33%) Rf: 0.58 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta = 7.66$ (dd, 1H $J_1 = 1.11$ Hz, $J_2 = 3.79$ Hz), 7.60 (dd, 1H $J_1 = 1.11$ Hz, $J_2 = 5.06$ Hz), 7.09 (dd, 1H $J_1 = 4.90$ Hz, $J_2 = 3.79$ Hz), 2.52 (s, 3H) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta = 191.1$, 144.9, 134.2, 132.9, 128.5 ppm. $\nu_{\max}$ 2978, 2930, 1660, 1413, 1357, 1271, 857, 721, 590 cm$^{-1}$ MS (EI, 70 eV): m/z (%): 126 (40, [M$^+$]), 111 (100), 83 (12).

Benzophenone (2q)$^3$

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (16 mg, 0.04 mmol, 2 mol%), SDS (11.6 mg, 0.04 mmol, 2 mol%), and 3 mL of water. Then diphenylmethane (333 µl, 336.5 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Colorless oil 326 mg (1.79 mmol yield: 90 %) Rf: 0.53 (in hexane:EtOAc = 3:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta = 7.71$ (d, 4H $J = 6.79$ Hz), 7.50 (t, 2H $J = 7.35$ Hz), 7.39 (t, 4H, $J = 7.27$ Hz) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta = 196.6$, 137.4, 132.3, 129.9, 128.2 cm$^{-1}$ $\nu_{\max}$ 3060, 1655, 1446, 1274, 940, 918, 694, 637, 437 cm$^{-1}$ MS (EI, 70 eV): m/z (%) : 182 (75, [M$^+$]), 105 (100), 77 (72), 51 (30).

2-benzoylephnol acetate (2r)$^{14}$

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (10 mg, 0.025 mmol, 2.5 mol%), SDS (14.4 mg, 0.05 mmol, 5 mol%), and 1 mL of water. Then 2-benzylphenyl acetate (226 mg, 1 mmol) was added followed by TBHP (645 µL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was...
washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Yellow oil 68 mg (0.28 mmol, yield: 28% Rf: 0.72 (in hexane:EtOAc = 2:1))

1H NMR (250 MHz, CDCl3): δ = 7.77 (d, 2H J = 7.27 Hz), 7.55 (t, 2H J = 6.48 Hz), 7.45 (t, 2H J = 7.35 Hz), 7.33 (t, 1H J = 7.11 Hz), 7.20 (d, 1H J = 7.90 Hz), 1.94 (s, 3H) ppm.

13C NMR (62.5 MHz, CDCl3): δ = 194.8, 169.1, 148.7, 137.5, 133.0, 132.2, 131.4, 130.4, 129.7, 128.3, 125.6, 123.2, 20.5 ppm. νmax 1765, 1662, 1448, 1184, 1102, 761, 698, 633 cm⁻¹ MS (EI, 70 eV): m/z (%): 240 (5, [M⁺]), 197 (100), 121 (45), 105 (30), 77 (43).

N-(2-benzoylphenyl)acetamide (2s)

A conical test tube was charged with Fe₂(SO₄)₃ (10 mg, 0.025 mmol, 2.5 mol%), SDS (14.4 mg, 0.05 mmol, 5 mol%), and 1 mL of water. Then N-(2-benzylphenyl)acetamide (225 mg, 1 mmol) was added followed by TBHP (645 μL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Off-white solid 148 mg (0.62 mmol, yield: 62% Rf: 0.51 (in hexane:EtOAc = 2:1))

1H NMR (250 MHz, CDCl3): δ = 10.84 (s, 1H), 8.64 (d, 1H J = 8.21 Hz), 7.71 (d, 2H J = 7.11 Hz), 7.63-7.46 (m, 5H), 7.09 (t, 1H J = 7.58 Hz), 2.23 (s, 3H) ppm. 13C NMR (62.5 MHz, CDCl3): δ = 199.5, 169.0, 140.3, 138.4, 134.1, 133.3, 132.4, 129.7, 128.2, 123.1, 121.9, 121.3, 25.0 ppm. νmax 1698, 1580, 1518, 1444, 1289, 1256, 1156, 919, 751, 698, 607 cm⁻¹ MS (EI, 70 eV): m/z (%): 239 (12, [M⁺]), 196 (100), 167 (15), 134 (17), 120 (40), 105 (12), 92 (15), 77 (35), m.p.: 75-77°C (lit.: 81°C)

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

A conical test tube was charged with Fe₂(SO₄)₃ (60 mg, 0.15 mmol, 5 mol%), SDS (43.3 mg, 0.15 mmol, 5 mol%), and 3 mL of water. Then 1,2,3,4-tetrahyronaphthalene (408.6 μl, 396.3
mg, 3 mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Yellow oil 114 mg (0.78 mmol, yield: 26%) Rf: 0.52 (in hexane:EtOAc = 4:1) \(^1\)H NMR (250 MHz, CDCl\(_3\)): \(\delta = 7.22\) (d, 1H, \(J = 8.06\) Hz), 7.38-7.32 (m, 1H), 7.21-7.12 (m, 2H), 2.85 (t, 2H, \(J = 6.08\) Hz), 2.54 (t, 2H, \(J = 6.55\) Hz), 2.02 (q, 2H, \(J = 12.64\) Hz) ppm. \(^{13}\)C NMR (62.5 MHz, CDCl\(_3\)): \(\delta = 198.7, 144.9, 133.8, 133.0, 129.1, 127.5, 127.0, 39.6, 30.1, 23.7\) ppm. \(\nu_{\text{max}}\) 2976, 2933, 1681, 1599, 1299, 1284, 762, 734, 553 cm\(^{-1}\) MS (EI, 70 eV): \(m/z\) (%): 146 (70, [M\(^+\)]), 131 (15), 118 (100), 103 (2), 90 (70), 77 (5)

**Anthracene-9,10-dione (2v)\(^6\)**

A conical test tube was charged with FeCl\(_3\) (10.8 mg, 0.04 mmol, 2 mol%), SDS (11.5 mg, 0.04 mmol, 2 mol%), and 2 mL of water. Then 9,10-dihydroanthracene (360 mg, 2 mmol) was added followed by TBHP (2.58 mL, 20 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Off-white solid 445 mg (2.1 mmol, yield: 88%) Rf: 0.39 (in hexane:EtOAc = 3:1) \(^1\)H NMR (250 MHz, CDCl\(_3\)): \(\delta = 8.32\) (s, 4H), 7.81 (s, 4H) ppm. \(^{13}\)C NMR (62.5 MHz, CDCl\(_3\)): \(\delta = 183.1, 134.1, 133.5, 127.2\) ppm. \(\nu_{\text{max}}\) 1673, 1573, 1281, 1168, 935, 808, 691, 620 cm\(^{-1}\) MS (EI, 70 eV): \(m/z\) (%): 208 (100, [M\(^+\)]), 180 (100), 152 (100), 126 (15), 76 (42). m.p. 279-281°C

**9H-xanthen-9-one (2w)\(^3\)**

A conical test tube was charged with FeCl\(_3\) (10.8 mg, 0.04 mmol, 2 mol%), SDS (11.5 mg, 0.04 mmol, 2 mol%), and 2 mL of water. Then 9H-xanthene (364 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa,
and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

Off-white solid 380 mg (1.94 mmol, yield: 97%) Rf: 0.39 (in hexane:EtOAc = 3:1) ¹H NMR (250 MHz, CDCl₃): δ = 8.32 (d, 2H, J = 7.74 Hz), 7.70 (t, 2H, J = 7.5 Hz), 7.46 (d, 2H, J = 8.06 Hz), 7.35 (t, 2H, J = 7.19 Hz) ppm. ¹³C NMR (62.5 MHz, CDCl₃): δ = 177.1, 156.0, 134.7, 126.6, 123.8, 121.7, 117.9 ppm. ν max 1653, 1615, 1478, 1454, 1329, 1144, 755, 670, 625 cm⁻¹ MS (EI, 70 eV): m/z (%): m.p. 174 °C (lit.): 172-174 °C

Isochroman-1-one (2x)³
A conical test tube was charged with FeCl₃ (40.5 mg, 0.15 mmol, 5 mol%), SDS (43.3 mg, 0.15 mmol, 5 mol%), and 3 mL of water. Then isochroman (377.3 µl, 402.5 mg, 3 mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 10 min at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

Colorless oil 123 mg (0.83 mmol, yield: 28%) Rf: 0.16 (in hexane:EtOAc = 3:1) ¹H NMR (250 MHz, DMSO-d₆): δ = 8.00 (d, 1H, J = 7.58 Hz), 7.46 (t, 1H, J = 7.50 Hz), 7.30 (t, 1H, J = 7.42 Hz), 7.19 (d, 1H, J = 7.58 Hz), 4.44 (t, 2H, J = 6.00 Hz), 2.98 (t, 2H, J = 6.00 Hz) ppm. ¹³C NMR (62.5 MHz, DMSO-d₆): δ = 163.4, 140.2, 133.6, 129.4, 127.6, 124.9, 67.1, 27.0 ppm. ν max 2900, 1715, 1392, 1292, 1240, 1118, 1089, 1027, 743, 693, 491. MS (EI, 70 eV): m/z (%): 148 (50, [M⁺]), 118 (100), 90 (85), 63 (20).

Isobenzofuran-1(3H)-one (2y)³
A conical test tube was charged with FeCl₃ (40.5 mg, 0.15 mmol, 5 mol%), SDS (43.3 mg, mmol, 5 mol%), and 3 mL of water. Then 1,3-dihydroisobenzofuran (330.7 µl, 360.5 mg, mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 10 min at 50°C. After cooling to room temperature, the
solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

White solid 133 mg (0.99 mmol, yield: 33%) Rf: 0.53 (in hexane:EtOAc = 3:1) $^1$H NMR (250 MHz, DMSO-$d_6$): $\delta = 7.83-7.72$ (m, 2H), 7.66-7.52 (m, 2H), 5.39 (s, 2H) ppm. $^{13}$C NMR (62.5 MHz, DMSO-$d_6$): $\delta = 170.64, 147.2, 134.1, 128.8, 124.8, 124.8, 122.9, 69.8$ ppm. $\nu_{\text{max}}$ 2916, 1745, 1708, 1466, 1437, 1366, 1216, 1048, 999, 737, 679, 472 cm$^{-1}$. MS (EI, 70 eV): $m/z$ (%): 134 (50, [M$^+$]), 105 (100), 77 (45), 51 (15). m.p. 72°C (lit.): 71-74°C

1-tosyl-2,3-dihydroquinolin-4(1H)-one (2z)$^{17}$

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (20 mg, 0.05 mmol, 5 mol%), SDS (14.4 mg, 0.05 mmol, 5 mol%), and 1 mL of water. Then 1-tosyl-1,2,3,4-tetrahydroquinoline (287.1 mg, 1 mmol) was added followed by TBHP (0.65 mL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 8 hours at 50°C, then TBHP (0.13 ml, 1 mmol, 1 eq.) was added and the reaction mixture was stirred for further 16 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

White solid 90.1 mg (0.3 mmol, yield: 30%) Rf.: 0.72 (in hexane:EtOAc = 3:1) $^1$H NMR (250 MHz, CDCl$_3$): $\delta = 7.93$ (d, 1H, $J = 7.74$ Hz), 7.85 (d, 1H, $J = 8.37$ Hz), 7.59-7.53 (m, 3H), 7.29-7.20 (m, 3H), 4.22 (t, 2H, $J = 6.40$ Hz), 2.37 (t, 2H, $J = 6.24$ Hz), 2.37 (s, 3H) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): $\delta = 193.1, 145.0, 142.8, 137.2, 135.1, 130.5, 128.2, 127.3, 126.1, 126.0, 125.0, 46.6, 36.9, 22.0$ ppm. $\nu_{\text{max}}$ 3064, 2923, 1680, 1595, 1349, 1294, 1162, 1076, 922, 772, 728, 682, 575, 550, 539 cm$^{-1}$. MS (EI, 70 eV): $m/z$ (%): 301 (10, [M$^+$]), 155 (20), 146 (75), 128 (4), 117 (15), 104 (4), 91 (100), 77 (10), 65 (20). m.p.: 138-140°C
Methyl 2-oxo-2-phenylacetate (2aa)\textsuperscript{18}

A conical test tube was charged with Fe\textsubscript{2}(SO\textsubscript{4})\textsubscript{3} (20 mg, 0.1 mmol, 2.5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and 2 mL of water. Then methyl 2-phenylacetate (285 µl, 300.3 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 8 hours at 50°C, then TBHP (1.29 ml, 10 mmol, 5 eq.) was added and the reaction mixture was stirred for further 16 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Colorless oil 264 mg (1.6 mmol, yield: 80%) Rf: 0.41 (in hexane:EtOAc = 5:1) \textsuperscript{1}H NMR (250 MHz, CDCl\textsubscript{3}): δ = 8.05 (dd, 2H, J\textsubscript{1} = 7.11 Hz, J\textsubscript{2} = 1.42 Hz), 7.65 (t, 1H, J = 7.5 Hz), 7.53-7.45 (m, 2H), 3.97 (s, 3H) ppm. \textsuperscript{13}C NMR (62.5 MHz, CDCl\textsubscript{3}): δ = 186.5, 164.0, 135.0, 132.2, 130.1, 128.8, 52.7 ppm. ν\textsubscript{max} 2956, 1737, 1689, 1204, 1172, 1003, 677 cm\textsuperscript{-1} MS (EI, 70 eV): m/z (%): 164(2, [M\textsuperscript{+}]), 105(100), 77(52), 51(20).

\begin{center}
\includegraphics[width=0.5\textwidth]{methyl_2-oxo-2-phenylacetate.png}
\end{center}

1-benzyl isatin (2bb)\textsuperscript{19}

A conical test tube was charged with Fe\textsubscript{2}(SO\textsubscript{4})\textsubscript{3} (5mg, 0.025 mmol, 5 mol%), SDS (1.8 mg, mmol, 5 mol%), and 0.266 mL of water. Then 1-benzylindole (52 mg, 0.25 mmol) was added followed by TBHP (0.484mL, 0.75mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

Orange solid. 12.3 mg (0.05 mmol, yield: 21%, Purity 92%) Rf: 0.40 (in hexane:EtOAc = 3:1) \textsuperscript{1}H NMR (250 MHz, CDCl\textsubscript{3}): δ = 7.55 (d, 1H, J = 6.63 Hz), 7.41 (t, 1H, J = 7.82 Hz), 7.55 (d, 1H, J = 6.63 Hz), 7.28-7.19 (m, 5H), 7.02 (t, 1H, J = 7.66 Hz), 6.01 (d, 1H, J = 8.20 Hz), 4.86 (s, 2H) ppm. \textsuperscript{13}C NMR (62.5 MHz, CDCl\textsubscript{3}): δ = 183.6, 158.7, 151.1, 138.7, 134.9, 129.4, 127.8, 124.3, 118.1, 111.4, 44.4 ppm. ν\textsubscript{max} 2925, 1737, 1689, 1467, 1345, 1174, 1091, 1001, 852, 751, 691, 469 cm\textsuperscript{-1} MS (EI, 70 eV): m/z (%): 237 (35, [M\textsuperscript{+}]), 207 (5), 180 (40), 146 (100), 104 (10), 91 (50), 77 (10). m.p.: 110-120°C (lit.): 133-135°C
Benzoic acid (2cc)

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (30mg, 0.075mmol, 5 mol%), SDS (43.3mg, mmol, 5 mol%), and 1.1 mL of water. Then 2-phenylacetonitrile (345 μl, 351 mg, 3 mmol) was added followed by TBHP (1.9 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

A conical test tube was charged with Fe$_2$(SO$_4$)$_3$ (20 mg, mmol, 5 mol%), SDS (28.8 mg, mmol, 5 mol%), and 0.710 mL of water. Then benzylalcohol (207 μl, 216 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

White solid 168 mg (1.38 mmol, yield: 46% from 2-phenylacetonitrile), 99.7 mg (0.82 mmol, yield: 41% from benzylalcohol) Rf: 0.32 (in hexane:EtOAc = 5:1) $^1$H NMR (250 MHz, CDCl$_3$): δ = 12.71 (s, 1H), 8.16 (d, 1H, $J$ = 7.42 Hz), 7.64 (t, 1H, $J$ = 7.35 Hz), 7.50 (t, 1H, $J$ = 7.50 Hz) ppm. $^{13}$C NMR (62.5 MHz, CDCl$_3$): δ = 173.1, 134.3, 130.7, 129.8, 128.9 ppm. ν$_{max}$ 3070, 2603, 2551, 1678, 1581, 1419, 1288, 1180, 930, 804, 704, 545 cm$^{-1}$ MS (EI, 70 eV): m/z (%): 122 (90, [M$^+$]), 105 (100), 94 (5), 77 (80), 51 (40). m.p.: 121-123°C (lit.): 121-125°C
NMR spectrum

Fluorene-9-one (2a)
2-iodo-9H-fluoren-9-one (2b)
2-bromo-9H-fluoren-9-one (2d)
2,7-dibromo-9H-fluoren-9-one (2e)

Electronic Supplementary Material (ESI) for RSC Advances
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Methyl 7-iodo-9-oxo-9H-fluorene-4-carboxylate (2f)
2-(phenylethynyl)-9H-fluoren-9-one (2g)
(E)-methyl 3-(9-oxo-9H-fluoren-2-yl)acrylate (2h)
Acetophenone (2i)³
Propiophenone (2j)
Butyrophenone (2k)⁹
Methyl 5-oxo-5-phenylpentanoate (2I)
Methyl 4-heptanoylbenzoate (2m)$^{11}$
1-(4-methoxyphenyl)ethanone (2n)\textsuperscript{12}
1-(thiophen-2-yl)ethanone (2p)\textsuperscript{13}
Benzophenone (2q)³
2-benzoylphenyl acetate (2r)
N-(2-benzoylphenyl)acetamide (2s) \textsuperscript{15}
3,4-dihyronaphthalen-1(2H)-one (2t)³
Anthracene-9,10-dione (2v)
9H-xanthen-9-one (2w)
Isochroman-1-one (2x)^3
**Isobenzofuran-1(3H)-one (2y)**

![COSY Spectrum](image)
1-tosyl-2,3-dihydroquinolin-4(1H)-one (2z)\textsuperscript{17}
Methyl 2-oxo-2-phenylacetate (2aa)\textsuperscript{18}
1-benzyl isatin (2bb)\textsuperscript{19}
Benzoic acid (2cc)\textsuperscript{20}

\[\text{COOH} \quad \text{COOH}\]
MS Spectras

Fluorene-9-one (2a)³

A b u n d a n c e

Scan 987 (6.675 min): SZF357-4.D data.mzs

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2-iodo-9H-fluoren-9-one (2b)
2-bromo-9H-fluoren-9-one (2d)³

Abundance

Scan 1132 (7.350 min): Bromfluorene.Da.ta.m.s

![Chemical Structure](image)
2,7-dibromo-9H-fluoren-9-one (2e)
Methyl 7-iodo-9-oxo-9H-fluorene-4-carboxylate (2f)
2-(phenylethynyl)-9H-fluoren-9-one (2g)\(^7\)

Abundance

Scan 1788 (10.406 min): SZF1067T.D\data.ms
(E)-methyl 3-(9-oxo-9H-fluoren-2-yl)acrylate (2h)
Acetophenone (2i)

**Abundance**

Scan 348 (3.700 min): SZF4885.D data.ms

Electronic Supplementary Material (ESI) for RSC Advances
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Propiophenone (2j)
Butyrophenone (2k)
Methyl 5-oxo-5-phenylpentanoate (2l)\textsuperscript{10}

Abundance

Scan 1116 (7.275 min): GZS7481.0

Electronic Supplementary Material (ESI) for RSC Advances
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Methyl 4-heptanoylbenzoate (2m)
1-(4-methoxyphenyl)ethanone (2n)
1-(thiophen-2-yl)ethanone (2p)
Benzophenone (2q)³

Abundance

Scan 826 (5.925 min): IZE.D.data.ms

Electronic Supplementary Material (ESI) for RSC Advances
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2-benzoylphenyl acetate (2r)
N-(2-benzoylphenyl)acetamide (2s)\textsuperscript{15}

Abundance

Scan 1219 (7.755 min): SZF822BT_D\_data.ms

m / z -->
3,4-dihyronaphthalen-1(2H)-one (2t)^3

Abundance

Scan 617 (4.952 min): SZF594.D\data.ms
Anthracene-9,10-dione (2v) \(^{16}\)

Abundance

Scan 1172 (7.536 min): PB171.Data.ms

![Anthracene-9,10-dione (2v)](image)
9H-xanthen-9-one (2w)
Isochroman-1-one (2x)

Scan 706 (5.367 min): SZF658.D data.m
Isobenzofuran-1(3H)-one (2y)³

Scan 587 (4.813 min): SZF748FS.D data.ms

Electronic Supplementary Material (ESI) for RSC Advances
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1-tosyl-2,3-dihydroquinolin-4(1H)-one (2x)\textsuperscript{17}

Abundance

Scan 1426 (8.718 min): szf774.dat
Methyl 2-oxo-2-phenylacetate (2aa)\textsuperscript{18}

\begin{center}
\textbf{Abundance}
\end{center}

\textit{Scan 524 (4.519 min): szf775.D data.ms}

\begin{center}
\includegraphics[width=\textwidth]{methyl_2-oxo-2-phenylacetate.png}
\end{center}
1-benzyl isatin (2bb)\(^1\)

A b u n d a n c e

Scan 1266 (7.974 min): GZS6871.Data.ms
Benzoic acid (2cc)"^20"

Scan 390 (3.895 min): GZS6762.D data.m

Electronic Supplementary Material (ESI) for RSC Advances
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