Supporting Information

Visible Light-induced Aerobic Oxidation of Diarylalkynes to \( \alpha \)-Diketones Catalyzed by Copper-superoxo at Room Temperature

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Experimental section

General: All reactions were conducted in oven-dried glasswares. All reactions were conducted using a blue light-emitting diode (LED) array (30 lamps, power density: 40 mW/cm$^2$ at 460 nm) as the visible-light source under oxygen (O$_2$) atmosphere in all reactions. All solvents were dried according to known methods and distilled prior to use. Starting materials were commercially available (Sigma-Aldrich or Alfa-Aesar or TCI-chemicals) and used as received. $^1$H NMR and $^{13}$C NMR spectra were recorded at 600 MHz using deuterated CDCl$_3$ or CDCl$_3$-DMSO-d$_6$ mixture. Chemical shifts (δ) were reported as parts per million (ppm) and the following abbreviations were used to identify the multiplicities: s= singlet, d= doublet, t= triplet, q= quartet, m= multiplet, b= broad and all combinations thereof can be explained by their integral parts. Unless otherwise specified, the proton/carbon signal of 2 residual solvents (at δ 7.24 or 2.50 and δ 77.00 or 39.51 ppm, respectively) was used as the internal reference.

General procedure for the synthesis of α-diketones:

Scheme S1: Current photochemical process for the synthesis of α-diketones

To a dry test tube (20 mL) containing 10 mol% CuCl$_2$ and internal alkyne (diarylacetylenes) (0.5 mmol), was added 5 mL of ACN, followed by the addition of 30 μL of water. The reaction mixture was then irradiated with blue LEDs (40 mW/cm$^2$ at 460 nm) under an oxygen atmosphere at room temperature (25-28 °C) until completion of the reaction (monitored by TLC). The reaction mixture was then diluted with 40 % ethyl acetate in hexane and stirred for 10 min. The mixture was filtered through celite and silica gel pads and washed with ethyl acetate. The filtrate was concentrated and the residue was purified by column chromatography on silica gel.

General procedure for the preparation of starting materials (internal alkynes)$^{1}$:

Scheme S2: Photo induced Sonogashira coupling
A dry test tube (20 mL) with a rubber septum and a magnetic stirrer bar was charged with aryl or alkyl iodides (0.50 mmol, 1.0 equiv.), K$_2$CO$_3$ (0.52 mmol, 1.05 equiv.) and 5 mol% CuCl. The test tube was evacuated and purged with dry N$_2$ gas for three times and then dry ACN (2 mL) and MeOH (2 mL) were added via syringe, and finally the terminal acetylene (0.60 mmol, 1.2 equiv.) using syringe. The transparent suspension was irradiated with blue light output from a blue LED array (power density 40 mW/cm$^2$ at 460 nm) at room temperature for 4–12 h until completion of the cross-coupling reaction (as determined by thin layer chromatography). The reaction mixture was diluted with 40% ethyl acetate in hexane and stirred for 10 min. The mixture was filtered through celite and silica gel pads and washed with ethyl acetate. The filtrate was concentrated and the residue was purified by flash column chromatography on silica gel to afford the desired cross-coupling product.

Procedures for the further transformations from the a-diketones:

a) Formation of benzillic acid from a-diketones\textsuperscript{a2}

Scheme S3: Formation of benzillic acid from α-diketones

![Scheme S3: Formation of benzillic acid from α-diketones](image)

After cooled to room temperature, the mixture was washed with ether (50 mL x 3). The aqueous layer was cooled to 0 °C, acidified with 12 N HCl (5 mL) and extracted with DCM (50 mL x 2). The combined organic layers were washed with brine, dried over MgSO$_4$ and the filtrate was concentrated in vacuo to get benzillic acid (3) (yield 89%).

b) Synthesis of Trifenagrel drug:

Step1: Preparation of 2-(4,5-diphenyl-1H-imidazol-2-yl)phenol\textsuperscript{a3}
**Scheme S4:** Preparation of 2-(4,5-diphenyl-1H-imidazol-2-yl)phenol from α-diketones

A mixture of benzil (0.525 g; 2.5 mmol), salicylaldehyde (0.3053 g; 2.5 mmol), ammonium acetate (0.5g;6mmol) and glycine (0.05 g, 0.6 mmol) was stirred in ethanol (10ml) for 3 h at 80 °C. The completion of the reaction was monitored by TLC. Ensuring the completion of reaction, the reaction mixture was poured into crush ice: cold ethanol mixture (1:1) and filtered to afford 2-(2-Hydroxyphenyl)-4,5-diphenylimidazole (4) in 93% yield (pale yellow precipitate).

**Step2: Preparation of 2-(2-(4,5-diphenyl-1H-imidazol-2-yl)phenoxy)-N,N-dimethylethanamine (Trifenagrel)**

**Scheme S5:** Synthesis of Trifenagrel (4')

2-(2-Hydroxyphenyl)-4,5-diphenylimidazole (0.2 g, 0.6 mmol) and K₂CO₃ (1.5 equiv., 0.12 g) was dissolved in MeOH (15 mL) and stirred for 15 mins at room temperature. Then, the solution of 2-bromo-N,N-dimethylethanamine (1.5 equiv., 0.15 g, 1.0 mmol) in 3 mL of MeOH was added dropwise into the reaction mixture over the period of 10mins. The reaction mixture was then stirred for 12 h at 60 °C. After completion of reaction, the reaction mixture was then concentrated (to remove MeOH) and then diluted with ether and layers were separated. The
filtrate was then concentrated and the residue was purified by column chromatography on silica gel (yield 85%).

c) **Preparation of 2,3-diphenylquinoxaline from α-diketones**:

Scheme S6: Preparation of 2,3-diphenylquinoxaline (5) from α-diketones

Benzil (0.21 g, 1 mmol) and o-phenyldiamine (0.11 g, 1.05 equiv., 1.05 mmol) was dissolved in ethanol and stirred at 60 °C for 3 h. The reaction mixture was then concentrated and the residue was purified by column chromatography on silica gel (yield 96%).

d) **Preparation of 2,4,5-triphenyloxazole (6)**

Scheme S7: Preparation of 2,4,5-triphenyloxazole (6)

Benzil (1.0 mmol), NH4OAc (5.0 mmol) and SnCl2.2H2O (0.05 mmol) was dissolved in EtOH (4 mL). The reaction mixture was stirred and refluxed for 4 h. After completion of reaction, the reaction mixture was then concentrated (to remove EtOH) and then diluted with ether and layers were separated. The filtrate was then concentrated and the residue was purified by column chromatography on silica gel (yield 73%).

e) **Preparation of 1,2-diphenylethane-1,2-diol (7)**

Scheme S8: Preparation of 1,2-diphenylethane-1,2-diol (7)
Benzil (1.0 mmol) was dissolved in EtOH (15 mL) and stirred for 5 min at 0 °C. To this solution, NaBH₄ (2.0 equiv.) was added slowly and the reaction mixture was further stirred for 4 h at room temperature. After completion of reaction, the reaction mixture was then concentrated (to remove EtOH) and then the layers were separated. The filtrate was then concentrated and the residue was purified by column chromatography on silica gel (yield 88%).

f) Preparation of 2,2-dimethyl-4,5-diphenyl-2H-imidazole (8)

Scheme S9: Preparation of 2,2-dimethyl-4,5-diphenyl-2H-imidazole (8)

2,2-dimethyl-4,5-diphenyl-2H-imidazole

Benzil (1.0 mmol) and NH₄OAc (5.0 mmol) was dissolved in AcOH (5 mL). To this solution, acetone (4.0 equiv.) was added. The reaction mixture was stirred and refluxed (120 °C) for 4 h. After completion of reaction, the reaction mixture was then concentrated and the layers were separated. The filtrate was then concentrated and the residue was purified by column chromatography on silica gel (yield 78%).

Control Reactions:

Control reaction presented in the Scheme S10 showed that the aryl diketones are stable. In contrast, alkyl diketones are not stable and undergo over oxidation (cleavage) to form various (oxidized products) unidentified products in the presence of copper superoxide. The decomposition of 3,4-hexadione under current reaction conditions forms various products which are difficult to identify and isolate.
**Scheme S10:** Control reactions to check the stability of aryl and alkyl α-diketones in the presence of copper superoxo species.

(a) ![Scheme Image](image)

**EPR measurements:** EPR spectra were recorded at room temperature on a Bruker ESP-300E (X band, 9.8 GHz) with parameters setting as shown below: receiver gain=30n; receiver phase=0deg; receiver harmonic=1; field modulation frequency=100000 Hz; microwave frequency [Hz] = 9.660469e+09; field modulation amplitude [T]= 0.00016; receiver time constant[S] = 0.32768; microwave power= 0.015 W; receiver offset [%FS]=0; DMPO (5,5-dimethyl-1-pyrroline N-oxide) was employed as a radical trap for superoxide.

The reaction under standard condition (1a, CuCl₂, O₂) in ACN was irradiated with blue LED light for 30 min in the presence of DMPO. The EPR signals shown in Figure S1 is corresponding to DMPO-OO(Cu). This result indicates that copper superoxide free radical was formed in the reaction solution. No superoxide EPR signals were observed from the reaction solution under standard condition in absence of CuCl₂ (Figure S2).
**Figure S1:** EPR spectra of the reaction mixture: diphenylacetylene (1a) (0.5 mmol), and 10 mol% of CuCl$_2$ in ACN (5 mL), 0.5 ml of this reaction solution was taken out into a small vial, followed by the addition of 0.01 mL of DMPO ($5 \times 10^{-2}$ M). The mixture was irradiated with blue LEDs at room temperature under an oxygen atmosphere (1 atm) for 30 minutes. The reaction mixture was then analyzed by EPR spectra. There are classical 6 peaks, the signals corresponding to (DMPO-OO(Cu))
Figure S2: EPR spectra of the reaction mixture: diphenylacetylene (1a) (0.5 mmol) in ACN (5 mL), 0.5 ml of this reaction solution was taken out into a small vial, followed by the addition of 0.01 mL of DMPO (5 x 10^{-2} M). The mixture was irradiated with blue LEDs at room temperature under an oxygen atmosphere (1 atm) for 30 minutes. The reaction mixture was then analyzed by EPR spectra. No signals were detected.
Evaluation of Green metrics for the literature reported photochemical process

\[
\text{Atom economy (AE)} = \frac{\text{Molecular mass of desired product}}{\text{Molecular mass of all reactants}} \times 100
\]

\[
\text{Reaction mass efficiency (RME)} = \frac{\text{Mass of desired product}}{\text{Mass of all reactants}} \times 100
\]

<table>
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<tr>
<th>Reactant1</th>
<th>1,2-diphenylethyne</th>
<th>1g</th>
<th>5.61 mmol</th>
<th>FW 178.23</th>
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<td>Reactant2</td>
<td>4,4'-Dinitrodiphenyl disulfide</td>
<td>0.259g</td>
<td>0.84 mmol</td>
<td>FW 308.33</td>
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<tr>
<td>Solvent</td>
<td>ACN</td>
<td>15.72g</td>
<td>(20mL)</td>
<td>----</td>
</tr>
<tr>
<td>Auxiliary</td>
<td>----</td>
<td>----</td>
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<td>----</td>
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<tr>
<td>Product</td>
<td>Benzil</td>
<td>0.977g</td>
<td>4.65 mmol</td>
<td>FW 210.22</td>
</tr>
</tbody>
</table>

Product yield = 83%

\[
\text{E-factor} = \frac{1g + 15.72g + 0.259g - 0.977g}{0.977g} = 16.0 \text{ Kg waste/ 1 Kg product}
\]

\[
\text{Atom economy} = \frac{210}{210} \times 100 = 100\%
\]

\[
\text{Atom efficiency} = 83\% \times 100\% /100 = 83\%
\]

\[
\text{Carbon efficiency} = \frac{14}{14} \times 100 = 100\%
\]

\[
\text{Reaction mass efficiency} = \frac{0.977g}{1g} \times 100 = 97.7\%
\]
**H$_2^{18}$O experiment**

Scheme S11: Reaction in the presence of H$_2^{18}$O

**Procedure:** To a dry test tube (20 mL) containing 10 mol% CuCl$_2$ and diphenylacetylene (0.5 mmol), was added 5 mL of dry ACN, followed by the addition of 30 µL of H$_2^{18}$O. The reaction mixture was then irradiated with blue LEDs (40 mW/cm$^2$ at 460 nm) under an oxygen atmosphere at room temperature (25-28 °C) for 10h. The reaction mixture was then diluted with 40 % ethyl acetate in hexane and stirred for 10 min. The mixture was filtered through celite and silica gel pads and washed with ethyl acetate. The filtrate was concentrated and the residue was purified by column chromatography on silica gel.

The ESI mass of the crude reaction mixture was detected. In the ESI mass data, we have observed only (M+Na) peak of $^{16}$O$_2$ product. Thus, this experiment showed that the source of O-atom in the product is only from the molecular O$_2$. 
ESI Mass Data

Chemical Formula: C_{14}H_{10}^{16}O_{2}
Exact Mass: 210.0681
Molecular Weight: 210.2190

<table>
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<td>233.05784</td>
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<td>-0.01</td>
<td>-0.03</td>
<td>1^{12}C_{14}^{1}H_{10}^{16}O_{2}Na^{16}O_{2}</td>
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</tbody>
</table>
Spectroscopic Data

Benzil (2a)\textsuperscript{6-9}

\[
\text{\begin{array}{c}
\text{\includegraphics[width=0.2\textwidth]{benzil.png}} \\
\text{(2a)}
\end{array}}
\]

Yellow solid; \textbf{\textsuperscript{1}H NMR} (600 MHz, CDCl\textsubscript{3}): \(\delta\) 7.94 (t, \(J= 6.0\) Hz, 4H), 7.62-7.59 (m, 2H), 7.46 (t, \(J= 12.0\) Hz, 4H); \textbf{\textsuperscript{13}C NMR} (150 MHz, CDCl\textsubscript{3}): \(\delta\) 194.4, 134.7, 132.8, 129.7 and 128.8; ESI-MS calcd for C\textsubscript{14}H\textsubscript{10}O\textsubscript{2} (M+H): 210.0681, found: 211.0795.

1-(naphthalen-1-yl)-2-phenylethane-1,2-dione (2b)\textsuperscript{6,7}

\[
\text{\begin{array}{c}
\text{\includegraphics[width=0.2\textwidth]{naphthalenyl.png}} \\
\text{(2b)}
\end{array}}
\]

Yellow solid; \textbf{\textsuperscript{1}H NMR} (600 MHz, CDCl\textsubscript{3}): \(\delta\) 9.28 (d, \(J= 6.0\) Hz, 1H), 8.09 (d, \(J= 6.0\) Hz, 1H), 8.01 (t, \(J= 6.0\) Hz, 2H), 7.93-7.89 (m, 2H), 7.73 (t, \(J= 6.0\) Hz, 1H), 7.65-7.59 (m, 2H), 7.51-7.45 (m, 3H); \textbf{\textsuperscript{13}C NMR} (150 MHz, CDCl\textsubscript{3}): \(\delta\) 197.1, 194.5, 135.9, 135.0, 134.7, 134.0, 133.3, 130.9, 129.9, 129.4, 129.0, 128.7, 128.6, 127.1, 125.9 and 124.4; \textbf{ESI-MS} calcd for C\textsubscript{18}H\textsubscript{16}O\textsubscript{2} (M+H): 260.0837, found: 261.0913.

1-(3,5-dimethylphenyl)-2-phenylethane-1,2-dione (2c)\textsuperscript{7}

\[
\text{\begin{array}{c}
\text{\includegraphics[width=0.2\textwidth]{methylphenyl.png}} \\
\text{(2c)}
\end{array}}
\]

Pale yellow oil; \textbf{\textsuperscript{1}H NMR} (600 MHz, CDCl\textsubscript{3}): \(\delta\) 7.95 (d, \(J= 7.2\) Hz, 2H), 7.64-7.61 (m, 1H), 7.56 (s, 2H), 7.50-7.46 (m, 2H), 7.26 (s, 1H), 2.34 (s, 6H); \textbf{\textsuperscript{13}C NMR} (150 MHz, CDCl\textsubscript{3}): \(\delta\) 194.7,
194.4, 138.6, 136.5, 134.5, 132.8, 131.6, 129.7, 128.7, 127.3 and 21.2; ESI-MS calcd for 
C$_{16}$H$_{14}$O$_2$(M+H): 238.0994, found: 239.1075.

1-(4-tert-butylphenyl)-2-phenylethane-1,2-dione (2d)$^7$

![Structural formula of 1-(4-tert-butylphenyl)-2-phenylethane-1,2-dione (2d)]

Off white solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.95 (d, $J$= 7.2 Hz, 2H), 7.89 (d, $J$= 12.0 Hz, 2H), 7.63 (t, $J$= 6.0 Hz, 1H), 7.51-7.47 (m, 4H), 1.32 (s, 9H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 194.7, 194.2, 159.0, 134.7, 133.0, 130.4, 129.8, 128.9, 126.0, 35.3 and 30.9; ESI-MS calcd for 
C$_{18}$H$_{18}$O$_2$(M+Na): 266.1307, found: 289.1205.

1-(4-methoxyphenyl)-2-phenylethane-1,2-dione (2e)$^6$

![Structural formula of 1-(4-methoxyphenyl)-2-phenylethane-1,2-dione (2e)]

Off white solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.95-7.92 (m, 4H), 7.62 (t, $J$= 6.0 Hz, 1H), 7.48 (t, $J$= 6.0 Hz, 2H), 7.65 (d, $J$= 6.0 Hz, 2H), 3.86 (s, 3H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 194.8, 193.1, 164.9, 134.7, 133.1, 132.3, 129.8, 128.9, 126.0, 114.3 and 55.6; ESI-MS calcd for 
C$_{15}$H$_{12}$O$_3$(M+Na): 240.0786, found: 263.0677.

1-(3-methoxyphenyl)-2-phenylethane-1,2-dione (2f)$^6$

![Structural formula of 1-(3-methoxyphenyl)-2-phenylethane-1,2-dione (2f)]
Off white solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.94 (d, $J$= 12.0 Hz, 2H), 7.64-7.62 (m, 1H), 7.52-7.46 (m, 4H), 7.45-7.36 (m, 1H), 7.19-7.17 (m, 1H), 3.84 (s, 3H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 194.4, 194.4, 160.0, 134.8, 134.1, 132.9, 130.0, 129.8, 128.9, 123.2, 121.8, 112.7, and 55.5; ESI-MS calcd for C$_{15}$H$_{12}$O$_3$ (M+Na): 240.0786, found: 263.0684.

1-(4-hydroxyphenyl)-2-phenylethane-1,2-dione (2g)$^{6}$

Brown solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.92 (d, $J$= 6.0 Hz, 2H), 7.82 (d, $J$= 6.0 Hz, 2H), 7.62 (t, $J$= 6.0 Hz, 1H), 7.47 (t, $J$= 6.0 Hz, 2H), 6.87 (d, $J$= 12.0 Hz, 2H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 195.4, 193.6, 162.5, 135.0, 132.9, 132.7, 129.9, 129.0, 125.5 and 116.1; ESI-MS calcd for C$_{14}$H$_{10}$O$_3$ (M+Na): 226.0630, found: 249.0529

1-(4-fluorophenyl)-2-phenylethane-1,2-dione (2h)$^{6}$

White solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 8.01 (d, $J$= 6.0 Hz, 2H), 7.96 (d, $J$= 6.0 Hz, 2H), 7.65 (t, $J$= 6.0 Hz, 1H), 7.50 (t, $J$= 6.0 Hz, 2H), 7.17 (t, $J$= 12.0 Hz, 2H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 194.0, 192.7, 167.6 (d, $^1J_{C-F}$=258.0 Hz), 135.0, 132.7 (d, $^2J_{C-F}$=6.0 Hz), 132.6, 129.9, 129.0, and 116.4 (d, $^2J_{C-F}$=22.5 Hz); ESI-MS calcd for C$_{14}$H$_9$FO$_2$ (M+Na): 228.0587, found: 251.0479
1-(3,5-difluorophenyl)-2-phenylethane-1,2-dione (2i)

White solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.95 (d, $J$= 6.0 Hz, 2H), 7.68 (t, $J$= 6.0 Hz, 1H), 7.54-7.49 (m, 4H), 7.10 (t, $J$= 6.0 Hz, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 192.7, 191.4, 163.9 (d, $^1J_{C-F}$=252.0 Hz), 162.2 (d, $^1J_{C-F}$=252.0 Hz), 135.3, 133.0, 132.4, 130.0, 129.1, 112.7 (q, $^2J_{C-F}$=15.0, 6.0 Hz), and 110.1 (d, $^2J_{C-F}$=25.5 Hz); ESI-MS calcd for C$_{14}$H$_8$F$_2$O$_2$ (M+Na): 246.0492, found: 269.0400

1-(4-chlorophenyl)-2-phenylethane-1,2-dione (2j)\textsuperscript{46}

White solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.94 (d, $J$= 12.0 Hz, 2H), 7.90 (d, $J$= 6.0 Hz, 2H), 7.64 (t, $J$= 6.0 Hz, 1H), 7.51-7.46 (m, 4H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 193.8, 193.0, 141.5, 135.0, 132.7, 131.3, 131.2, 129.9, 129.4 and 129.0; ESI-MS calcd for C$_{14}$H$_9$ClO$_2$ (M+Na): 244.0291, found: 267.0189

1-(4-bromophenyl)-2-phenylethane-1,2-dione (2k)\textsuperscript{46}

Pale yellow solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.94 (d, $J$= 7.2 Hz, 2H), 7.83-7.81 (m, 2H), 7.65-7.63 (m, 3H), 7.51-7.49 (m, 2H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 193.8, 193.2, 135.0, 132.7, 132.4,
131.6, 131.2, 130.4, 129.9, and 129.0; **ESI-MS** calcd for C$_{14}$H$_9$BrO$_2$ (M+Na): 287.9786, found: 310.9673.

1-(4-iodophenyl)-2-phenylethane-1,2-dione (2l)

![Structure of 2l](image)

White solid; **$^1$H NMR** (600 MHz, CDCl$_3$): $\delta$ 7.93 (d, $J$= 12.0 Hz, 2H), 7.86 (d, $J$= 6.0 Hz, 2H), 7.65 (d, $J$= 12.0 Hz, 3H), 7.49 (t, $J$= 12.0 Hz, 2H); **$^{13}$C NMR** (150 MHz, CDCl$_3$): $\delta$ 193.8, 193.6, 138.3, 135.0, 132.7, 132.2, 130.9, 129.9, 129.0 and 103.6; **ESI-MS** calcd for C$_{14}$H$_9$IO$_2$ (M+Na): 335.9647, found: 358.9521

1-phenyl-2-(4-(trifluoromethyl)phenyl)ethane-1,2-dione (2m)

![Structure of 2m](image)

Pale yellow solid; **$^1$H NMR** (600 MHz, CDCl$_3$): $\delta$ 8.08 (d, $J$= 6.0 Hz, 2H), 7.95 (d, $J$= 6.0 Hz, 2H), 7.76 (d, $J$= 12.0 Hz, 2H), 7.67 (t, $J$= 6.0 Hz, 1H), 7.52 (t, $J$= 6.0 Hz, 2H); **$^{13}$C NMR** (150 MHz, CDCl$_3$): $\delta$ 193.4, 193.0, 135.6, 135.2, 132.6, 130.2, 129.9, 129.1, 128.6 and 126.0; **ESI-MS** calcd for C$_{15}$H$_9$F$_3$O$_2$ (M+Na): 278.0555, found: 301.1433
4-(2-oxo-2-phenylacetyl)benzonitrile (2n)\textsuperscript{7}

White solid; \textsuperscript{1}H NMR (600 MHz, CDCl\textsubscript{3}): \(\delta\) 8.06 (d, \(J= 12.0\) Hz, 2H), 7.95 (d, \(J= 6.0\) Hz, 2H), 7.79 (d, \(J= 12.0\) Hz, 2H), 7.67 (t, \(J= 12.0\) Hz, 1H), 7.52 (t, \(J= 6.0\) Hz, 2H); \textsuperscript{13}C NMR (150 MHz, CDCl\textsubscript{3}): \(\delta\) 192.9, 192.3, 135.8, 135.3, 132.7, 132.4, 130.1, 129.9, 129.1, 117.8 and 117.5; ESI-MS calcd for C\textsubscript{15}H\textsubscript{9}NO\textsubscript{2} (M+Na): 235.0633, found: 258.0560.

1-(3-nitrophenyl)-2-phenylethane-1,2-dione (2o)\textsuperscript{8}

Yellow solid; \textsuperscript{1}H NMR (600 MHz, CDCl\textsubscript{3}): \(\delta\) 8.79 (d, \(J= 6.0\) Hz, 1H), 8.48 (d, \(J= 6.0\) Hz, 1H), 8.29 (d, \(J= 6.0\) Hz, 1H), 7.98 (d, \(J= 6.0\) Hz, 2H), 7.73-7.67 (m, 2H), 7.53 (t, \(J= 6.0\) Hz, 2H); \textsuperscript{13}C NMR (150 MHz, CDCl\textsubscript{3}): \(\delta\) 192.6, 191.4, 148.5, 135.4, 135.2, 134.2, 132.3, 130.3, 130.0, 129.2, 128.8 and 124.5; ESI-MS calcd for C\textsubscript{14}H\textsubscript{9}NO\textsubscript{4} (M+Na): 255.0532, found: 278.0460.

1-(4-acetylphenyl)-2-phenylethane-1,2-dione (2p)\textsuperscript{7}

White solid; \textsuperscript{1}H NMR (600 MHz, CDCl\textsubscript{3}): \(\delta\) 8.13 (s, 4H), 8.04 (d, \(J= 6.0\) Hz, 2H), 7.74 (t, \(J= 6.0\) Hz, 1H), 7.60 (t, \(J= 6.0\) Hz, 2H); \textsuperscript{13}C NMR (150 MHz, CDCl\textsubscript{3}): \(\delta\) 197.2, 193.7, 193.5, 141.2,
135.9, 135.1, 132.6, 130.0, 129.9, 129.0, 128.6 and 26.8; **ESI-MS** calcd for C₁₆H₁₂O₃ (M+Na): 252.0786, found: 275.0677.

1-phenyl-2-(thiophen-2-yl)ethane-1,2-dione (2q)

![2q](image)

Yellow solid; **¹H NMR** (600 MHz, CDCl₃): δ 8.03 (d, J= 8.4 Hz, 2H), 7.80 (d, J= 6.0 Hz, 1H), 7.78 (d, J= 4.2 Hz, 1H), 7.65-7.62 (m, 1H), 7.51-7.48 (m, 2H), 7.17-7.16 (m, 1H); **¹³C NMR** (150 MHz, CDCl₃): δ 192.0, 185.5, 139.8, 136.8, 136.7, 134.8, 132.5, 130.2, 128.9 and 128.8; **ESI-MS** calcd for C₁₂H₈O₂S (M+Na): 216.0245, found: 239.0142

1-(4-ethylphenyl)-2-phenylethane-1,2-dione (2r)

![2r](image)

Yellow liquid; **¹H NMR** (600 MHz, CDCl₃): δ 7.95 (d, J= 6.0 Hz, 2H), 7.87 (t, J= 6.0 Hz, 2H), 7.63-7.60 (m, 1H), 7.49-7.46 (m, 2H), 7.31 (d, J= 6.0 Hz, 2H), 2.70 (q, 2H), 1.23 (t, J= 6.0 Hz, 3 H); **¹³C NMR** (150 MHz, CDCl₃): δ 194.7, 194.2, 152.2, 134.7, 133.0, 130.6, 130.0, 129.8, 128.9, 128.5, 29.1 and 14.9; **ESI-MS** calcd for C₁₆H₁₄O₂ (M+Na): 238.0994, found: 261.0890.

1,4-diphenylbut-3-yne-1,2-dione (2s)

![2s](image)
Yellow oil; **¹H NMR** (600 MHz, CDCl₃): δ 8.07-8.05 (m, 2H), 7.65-7.62 (m, 3 H), 7.52-7.48 (m, 3H), 7.40-7.37 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃): δ 188.4, 178.5, 134.8, 133.6, 131.6, 130.4, 128.9, 128.7, 119.1, 99.1, and 87.0; **ESI-MS** calcd for C₁₆H₁₀O₂ (M+Na): 234.0681, found: 257.0079

1-(4-tert-butylphenyl)-2-(4-methoxyphenyl)ethane-1,2-dione (2w)

Semi solid; **¹H NMR** (600 MHz, CDCl₃): δ 7.92 (d, J= 9.0 Hz, 2H), 7.88 (d, J= 8.4 Hz, 2H), 7.49 (d, J= 8.4 Hz, 2H), 6.94 (d, J= 9.0 Hz, 2H), 3.85 (s, 3H), 1.31 (s, 9H); **¹³C NMR** (150 MHz, CDCl₃): δ 194.5, 193.3, 164.8, 158.7, 132.3, 130.6, 129.8, 126.1, 125.9, 114.2, 55.6, 35.3 and 30.9; **ESI-MS** calcd for C₁₉H₂₀O₃ (M+Na): 296.1412, found: 319.1318

1-(4-butylphenyl)-2-(4-methoxyphenyl)ethane-1,2-dione (2x)

White solid; **¹H NMR** (600 MHz, CDCl₃): δ 7.88 (d, J= 12.0 Hz, 2H), 7.81 (d, J= 6.0 Hz, 2H), 7.22 (d, J= 12.0 Hz, 2H), 6.90 (d, J= 12.0 Hz, 2H), 3.82 (s, 3H), 2.61 (t, J= 6.0 Hz, 2H), 1.57-1.52 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃): δ 194.6, 193.4, 164.8, 150.8, 132.3, 130.8, 129.0, 126.1, 114.2, 55.6, 35.8, 33.1, 22.2 and 13.8; **ESI-MS** calcd for C₁₉H₂₀O₃ (M+H): 296.1412, found: 297.1490

1-(4-tert-butylphenyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-dione (2y)
White solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 8.07 (d, $J$ = 6.0 Hz, 2H), 7.89 (d, $J$ = 12.0 Hz, 2H), 7.74 (d, $J$ = 6.0 Hz, 2H), 7.52 (d, $J$ = 6.0 Hz, 2H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 193.2, 193.1, 159.4, 135.7 (t, $^2J_{C,F}$=19.3 Hz), 131.3, 131.2, 130.4, 130.1, 130.0, 129.9, 126.1, 125.9, 124.2 (q, $^1J_{C,F}$=272.2 Hz), 122.4, 35.4 and 30.9; ESI-MS calcd for C$_{19}$H$_{17}$F$_3$O$_2$ (M+Na): 334.1181, found: 357.1068

1-(4-tert-butylphenyl)-2-(4-chlorophenyl)ethane-1,2-dione (2z)

Yellow solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.91-7.88 (m, 4H), 7.51 (d, $J$ = 6.0 Hz, 2H), 7.46 (d, $J$ = 6.0 Hz, 2H), 1.32 (s, 9H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 193.5, 193.2, 159.2, 141.4, 131.4, 131.1, 130.2, 129.9, 129.3, 126.0, 35.3 and 30.9; ESI-MS calcd for C$_{18}$H$_{17}$ClO$_2$ (M+Na): 300.0917, found: 323.0805

1-(4-chlorophenyl)-2-(4-methoxyphenyl)ethane-1,2-dione (2aa)

Yellow solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 7.90 (t, $J$ = 12.0 Hz, 4H), 7.45 (d, $J$ = 6.0 Hz, 2H), 6.95 (d, $J$ = 12.0 Hz, 2H), 6.95 (d, $J$ = 6.0 Hz, 2H), 3.87 (s, 3H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 193.3, 192.4, 165.1, 141.3, 132.4, 131.5, 131.2, 129.3, 125.8, 114.4 and 55.6; ESI-MS calcd for C$_{15}$H$_{11}$ClO$_3$ (M+Na): 274.0397, found: 297.0294
1-(4-acetylphenyl)-2-(4-ethylphenyl)ethane-1,2-dione (2ab)

White solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 8.03 (s, 4H), 7.86 (d, $J$= 6.0 Hz, 2H), 7.32 (d, $J$= 12.0 Hz, 2H), 2.71 (q, 2H), 2.62 (s, 3H), 1.24 (t, $J$= 6.0 Hz, 3H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 197.2, 193.7, 193.4, 152.6, 141.2, 136.1, 130.4, 130.1, 130.0, 128.6, 128.6, 29.1, 26.9 and 15.0; ESI-MS calcd for C$_{18}$H$_{16}$O$_3$ (M+Na): 280.1099, found: 303.0982

1-(4-acetylphenyl)-2-(4-tert-butylphenyl)ethane-1,2-dione (2ac)

White solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 8.04 (s, 4H), 7.93 (d, $J$= 8.4 Hz, 2H), 7.52 (d, $J$= 9.0 Hz, 2H), 2.63 (s, 3H), 1.32 (s, 9H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 197.2, 193.8, 193.4, 159.3, 141.1, 136.0, 130.0, 129.9, 129.8, 128.6, 128.1, 126.1, 35.4, and 30.9; ESI-MS calcd for C$_{20}$H$_{20}$O$_3$ (M+Na): 308.1412, found: 331.1324

1-(4-acetylphenyl)-2-(4-methoxyphenyl)ethane-1,2-dione (2ad)

White solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 8.02 (t, $J$= 6.0 Hz, 4H), 7.92 (d, $J$= 12.0 Hz, 2H), 6.95 (d, $J$= 6.0 Hz, 2H), 3.86 (s, 3H), 2.62 (s, 3H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 197.2, 193.8,
192.2, 165.2, 141.1, 136.2, 130.0, 128.6, 125.7, 114.4, 55.6 and 26.9; **ESI-MS** calcd for C\textsubscript{17}H\textsubscript{14}O\textsubscript{4} (M+H): 282.0892, found: 283.0969

**2-hydroxy-2,2-diphenylacetic acid (Benzillic acid)** (3)

![Chemical Structure Image](image)

White solid; **\textsuperscript{1}H NMR** (600 MHz, CDCl\textsubscript{3}): \(\delta 7.33 \text{ (d, } J = 12.0 \text{ Hz, 4H), 7.16-7.11 \text{ (m, 6H), 6.19 (s, 1H);} \textsuperscript{13}C NMR** (150 MHz, CDCl\textsubscript{3}): \(\delta 175.5, 142.2, 127.4, 127.2, 127.0 \text{ and 80.1; ESI-MS calcd for C}_{14}H_{12}O_{3} (M-H): 228.0786, found: 227.0707}

**2-(4,5-diphenyl-1H-imidazol-2-yl)phenol** (4)

![Chemical Structure Image](image)

Pale yellow solid; **\textsuperscript{1}H NMR** (600 MHz, CDCl\textsubscript{3}): \(\delta 7.83 \text{ (d, } J = 6.0 \text{ Hz, 1H), 7.54 \text{ (d, } J = 6.0 \text{ Hz, 4H), 7.31 \text{ (d, } J = 12.0 \text{ Hz, 4H), 7.28 \text{ (d, } J = 12.0 \text{ Hz, 2H), 7.19 \text{ (t, } J = 6.0 \text{ Hz, 1H), 7.00 \text{ (d, } J = 6.0 \text{ Hz, 1H), 6.84 \text{ (t, } J = 6.0 \text{ Hz, 1H); \textsuperscript{13}C NMR** (150 MHz, CDCl\textsubscript{3}): \(\delta 157.0, 146.0, 132.5, 132.1, 129.7, 128.3, 127.8, 127.3, 124.2, 118.7, 117.0 \text{ and 113.0; ESI-MS calcd for C}_{21}H_{16}N_{2}O (M+H): 312.1263, found: 313.1352}
2-(2-(4,5-diphenyl-1H-imidazol-2-yl)phenoxy)-N,N-dimethylethanamine (4')

Pale yellow liquid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 12.33 (s, 1H), 8.46 (d, $J$= 6.0 Hz, 1H), 7.60-7.25 (m, 12H), 7.10 (t, $J$= 6.0 Hz, 1H), 6.98 (d, $J$= 6.0 Hz, 1H), 4.18 (t, $J$= 6.0 Hz, 2H), 2.63 (t, $J$= 6.0 Hz, 2H), 1.94 (s, 6H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 154.9, 143.6, 128.9, 128.7, 128.2, 127.4, 126.4, 122.0, 120.2, 113.5, 65.5, 57.9 and 44.3; ESI-MS calcd for C$_{25}$H$_{25}$N$_3$O (M+H): 383.1998, found: 384.2083

2,3-diphenylquinoxaline (5)

Yellow solid; $^1$H NMR (600 MHz, CDCl$_3$): $\delta$ 8.17 (t, $J$= 6.0 Hz, 2H), 7.71 (d, $J$= 6.0 Hz, 2H), 7.53 (d, $J$= 6.0 Hz, 4H), 7.34-7.30 (m, 6H); $^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 152.9, 140.8, 138.7, 129.5, 128.7, 128.4 and 127.8; ESI-MS calcd for C$_{20}$H$_{14}$N$_2$ (M+H): 282.1157, found: 283.1238

2,4,5-triphenyloxazole (6)
Pale yellow solid; \textbf{\textsuperscript{1}H NMR} (600 MHz, CDCl\textsubscript{3}): \textit{\&} 8.18 (d, \textit{J} = 6.0 Hz, 2H), 7.74 (d, \textit{J} = 6.0 Hz, 2H), 7.68 (d, \textit{J} = 6.0 Hz, 2H), 7.48-7.46 (m, 3H), 7.43-7.34 (m, 6H); \textbf{\textsuperscript{13}C NMR} (150 MHz, CDCl\textsubscript{3}): \textit{\&} 160.0, 145.4, 136.6, 134.8, 132.5, 130.2, 129.8, 128.7, 128.6, 128.5, 128.0, 126.4 and 126.3; \textbf{ESI-MS} calcd for C\textsubscript{21}H\textsubscript{15}NO (M+H): 297.1154, found: 298.1229

(1R,2R)-1,2-diphenylethane-1,2-diol (7)

\[
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\text{O} \\
\text{H}
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\text{H}
\end{array}
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\text{OH}
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\begin{array}{c}
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White solid; \textbf{\textsuperscript{1}H NMR} (600 MHz, CDCl\textsubscript{3}): \textit{\&} 7.03-7.02 (m, 6H), 6.98 (dd, \textit{J} = 6.0 Hz, 4H), 4.64 (s, 2H), 4.07 (s, 2H); \textbf{\textsuperscript{13}C NMR} (150 MHz, CDCl\textsubscript{3}): \textit{\&} 140.4, 127.1, 126.7 and 77.1; \textbf{ESI-MS} calcd for C\textsubscript{14}H\textsubscript{14}O\textsubscript{2} (M+Na): 214.0994, found: 237.0884

2,2-dimethyl-4,5-diphenyl-2H-imidazole (8)

\[
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\begin{array}{c}
\text{N}
\end{array}
\begin{array}{c}
\text{H}
\end{array}
\begin{array}{c}
\text{H}
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\begin{array}{c}
\text{H}
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\begin{array}{c}
\text{H}
\end{array}
\]

White solid; \textbf{\textsuperscript{1}H NMR} (600 MHz, CDCl\textsubscript{3}): \textit{\&} 7.43 (t, \textit{J} = 6.0 Hz, 4H), 7.33-7.30 (m, 2H), 7.25 (t, \textit{J} = 6.0 Hz, 4H), 1.58 (s, 6H); \textbf{\textsuperscript{13}C NMR} (150 MHz, CDCl\textsubscript{3}): \textit{\&} 163.8, 132.3, 129.8, 128.5, 127.9, 101.2 and 23.8; \textbf{ESI-MS} calcd for C\textsubscript{17}H\textsubscript{16}N\textsubscript{2} (M+H): 248.1313, found: 249.1389

S26
References:

Figure S3: ORTEP diagram of compound 2a (CCDC No. 1859205)

![ORTEP diagram of compound 2a](image)

Table S1. Crystal data and structure refinement for 180116LT_0M_a.

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Goodness-of-fit on $F^2$  
1.105

Final R indices [$I>2\sigma(I)$]  
$R1 = 0.0295$, $wR2 = 0.0685$

R indices (all data)  
$R1 = 0.0313$, $wR2 = 0.0695$

Absolute structure parameter  
0(2)

Extinction coefficient  
n/a

Largest diff. peak and hole  
0.144 and -0.131 e.Å$^{-3}$

Table S2. Atomic coordinates (x 10$^4$) and equivalent isotropic displacement parameters (Å$^2$ x 10$^3$) for 180116LT_0M_a. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

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Table S3. Bond lengths [Å] and angles [°] for 180116LT_0M_a.

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Table S4. Anisotropic displacement parameters (Å^2 x 10^3) for 180116LT_0M_a. The anisotropic displacement factor exponent takes the form: -2π^2 [ h^2 a^* 2 U11 + ... + 2 h k a^* b^* U12 ]
Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 180116LT_0M_a.

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<td>1096</td>
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<td>56</td>
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<tr>
<td>H(4)</td>
<td>2367</td>
<td>6409</td>
<td>3119</td>
<td>62</td>
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</tr>
<tr>
<td>H(5)</td>
<td>3255</td>
<td>4955</td>
<td>4298</td>
<td>57</td>
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<tr>
<td>H(6)</td>
<td>3236</td>
<td>2234</td>
<td>3888</td>
<td>47</td>
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Table S6. Crystal data and structure refinement for twin5.

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<td>Formula weight</td>
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<td>Temperature</td>
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</tr>
<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>C 2/c</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td>a = 23.502(2) Å, a = 90°</td>
</tr>
<tr>
<td></td>
<td>b = 3.9358(3) Å, b = 105.082(2)°</td>
</tr>
<tr>
<td></td>
<td>c = 15.1235(12) Å, g = 90°</td>
</tr>
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<td>Volume</td>
<td>1350.73(19) Å</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>Density (calculated)</td>
<td>1.388 Mg/m³</td>
</tr>
<tr>
<td>Absorption coefficient</td>
<td>0.099 mm⁻¹</td>
</tr>
<tr>
<td>F(000)</td>
<td>592</td>
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<tr>
<td>Crystal size</td>
<td>0.25 x 0.12 x 0.04 mm³</td>
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<tr>
<td>Theta range for data collection</td>
<td>1.795 to 26.552°</td>
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<tr>
<td>Index ranges</td>
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<tr>
<td>Reflections collected</td>
<td>1467</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>1467 [R(int) = 0.0370]</td>
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<tr>
<td>Completeness to theta</td>
<td>99.8 %</td>
</tr>
<tr>
<td>Absorption correction</td>
<td>Semi-empirical from equivalents</td>
</tr>
<tr>
<td>Max. and min. transmission</td>
<td>0.9485 and 0.7780</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on F²</td>
</tr>
<tr>
<td>Data / restraints / parameters</td>
<td>1467 / 52 / 122</td>
</tr>
<tr>
<td>Goodness-of-fit on F²</td>
<td>1.044</td>
</tr>
</tbody>
</table>
Final R indices [I>2sigma(I)] \( R1 = 0.0379, \text{wR}2 = 0.0865 \)
R indices (all data) \( R1 = 0.0537, \text{wR}2 = 0.0944 \)
Extinction coefficient n/a
Largest diff. peak and hole 0.208 and -0.172 e Å\(^{-3}\)

**Table S7.** Atomic coordinates (x 10\(^4\)) and equivalent isotropic displacement parameters (Å\(^2\)x 10\(^3\)) for twin5. \( U(eq) \) is defined as one third of the trace of the orthogonalized \( \text{U}ij \) tensor.

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
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<tbody>
<tr>
<td>C(1)</td>
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<td>8295</td>
<td>3835</td>
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<td>C(2)</td>
<td>3291</td>
<td>6782</td>
<td>2976</td>
<td>27</td>
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<tr>
<td>C(3)</td>
<td>3668</td>
<td>5597</td>
<td>2492</td>
<td>26</td>
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<td>C(4)</td>
<td>4279</td>
<td>5884</td>
<td>2846</td>
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<td>C(5)</td>
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<td>C(6)</td>
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<td>8593</td>
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<td>C(7)</td>
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<td>4563</td>
<td>2299</td>
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<td>3217</td>
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<td>C(8)</td>
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<td>4255</td>
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<td>9460</td>
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<td>11120</td>
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**Table S8.** Bond lengths [Å] and angles [°] for twin5.

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<th>Length</th>
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<td>1.32(2)</td>
</tr>
<tr>
<td>C(1)-C(6)</td>
<td>1.3930(19)</td>
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<tr>
<td>C(1)-C(2)</td>
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<tr>
<td>C(1)-O(3)</td>
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<tr>
<td>C(2)-C(3)</td>
<td>1.368(2)</td>
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<tr>
<td>C(2)-H(2)</td>
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</tr>
<tr>
<td>C(3)-C(4)</td>
<td>1.4002(19)</td>
</tr>
<tr>
<td>C(3)-H(3)</td>
<td>0.9500</td>
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<td>C(4)-C(5)</td>
<td>1.3950(19)</td>
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<td>C(5)-C(6)</td>
<td>1.382(2)</td>
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<td>C(5)-H(5)</td>
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<td>C(7)-O(1)</td>
<td>1.2241(17)</td>
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</tr>
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<td>1.22(2)</td>
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<tr>
<td>C(8)-C(9)</td>
<td>1.50(2)</td>
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<tr>
<td>C(9)-H(9A)</td>
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</tr>
<tr>
<td>C(9)-H(9B)</td>
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<tr>
<td>C(9)-H(9C)</td>
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<tr>
<td>O(3)-C(10)</td>
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<tr>
<td>C(10)-H(10A)</td>
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<td>C(10)-H(10B)</td>
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<td>C(10)-H(10C)</td>
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<td>C(8)-C(1)-C(6)</td>
<td>123.7(7)</td>
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<tr>
<td>C(8)-C(1)-C(2)</td>
<td>116.8(7)</td>
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<td>117.3(5)</td>
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<td>Bond/Distance</td>
<td>Angle (°)</td>
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<td>---------------</td>
<td>-----------</td>
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<td>H(10A)-C(10)-H(10C)</td>
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</table>

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y,-z+1/2
Table S9. Anisotropic displacement parameters (Å² x 10³) for twin5. The anisotropic displacement factor exponent takes the form: 
\[-2p²[ h² a*²U_{11} + ... + 2h k a*b* U_{12} ]\]

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<th>(U_{11})</th>
<th>(U_{22})</th>
<th>(U_{33})</th>
<th>(U_{23})</th>
<th>(U_{13})</th>
<th>(U_{12})</th>
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<td>26(1)</td>
<td>4(1)</td>
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<td>-2(1)</td>
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<td>-2(1)</td>
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<td>-4(1)</td>
<td>-6(1)</td>
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<td>-2(1)</td>
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<td>O(1)</td>
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<td>-10(1)</td>
<td>-2(1)</td>
<td>-3(1)</td>
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<td>C(8)</td>
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Table S10. Hydrogen coordinates (x 10⁴) and isotropic displacement parameters (Å² x 10³) for twin5.

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<th>(U(eq))</th>
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<td>H(9B)</td>
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<td>H(9C)</td>
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<td>H(10B)</td>
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<td>H(10C)</td>
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Figure S5: ORTEP diagram of compound 4 (CCDC No. 1859204)

Table S11. Crystal data and structure refinement for mo_180117lt_0m_a.

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<td>390.48</td>
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<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Triclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P -1</td>
</tr>
<tr>
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<td>a = 9.3322(5) Å, b = 10.0321(5) Å, c = 11.8425(6) Å</td>
</tr>
<tr>
<td></td>
<td>a = 95.845(2)°, b = 105.956(2)°, g = 109.513(2)°</td>
</tr>
<tr>
<td>Volume</td>
<td>981.56(9) Å³</td>
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<td>Z</td>
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<td>Density (calculated)</td>
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<tr>
<td>F(000)</td>
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<tr>
<td>Crystal size</td>
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<td>Theta range for data collection</td>
<td>1.831 to 26.568°</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-11&lt;=h&lt;=11, -12&lt;=k&lt;=12, -14&lt;=l&lt;=14</td>
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<tr>
<td>Reflections collected</td>
<td>28567</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>4075 [R(int) = 0.0443]</td>
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</table>
Completeness to theta = 25.242°  99.9 %
Absorption correction  Semi-empirical from equivalents
Max. and min. transmission  0.9485 and 0.8862
Refinement method  Full-matrix least-squares on F^2
Data / restraints / parameters  4075 / 0 / 256
Goodness-of-fit on F^2  1.139
Final R indices [I>2sigma(I)]  R1 = 0.0397, wR2 = 0.1006
R indices (all data)  R1 = 0.0566, wR2 = 0.1081
Extinction coefficient  n/a
Largest diff. peak and hole  0.296 and -0.428 e.Å^{-3}

**Table S12.** Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for mo_180117lt_0m_a. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

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Symmetry transformations used to generate equivalent atoms:

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Table S14. Anisotropic displacement parameters (Å^2 x 10^3) for mo_180117lt_0m_a. The anisotropic displacement factor exponent takes the form: -2p^2 [ h^2 a^2 U11 + ... + 2 h k a^* b^* U12 ]
Table S15. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for mo_180117lt_0m_a.

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**Table S16.** Crystal data and structure refinement for mo_170751lt_0m.

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<td>b</td>
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<tr>
<td>c</td>
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<td>Volume</td>
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Figure S6: ORTEP diagram of compound 5 (CCDC No. 1859203)
Absorption correction           Semi-empirical from equivalents
Max. and min. transmission      0.9485 and 0.6897
Refinement method               Full-matrix least-squares on F^2
Data / restraints / parameters  3101 / 0 / 199
Goodness-of-fit on F^2          1.055
Final R indices [I>2sigma(I)]   R1 = 0.0452, wR2 = 0.1197
R indices (all data)            R1 = 0.0571, wR2 = 0.1278
Extinction coefficient          n/a
Largest diff. peak and hole      0.304 and -0.275 e.Å^-3

Table S17. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for mo_170751lt_0m. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

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<th>z 10^4</th>
<th>U(eq) x 10^3</th>
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Table S18. Bond lengths [Å] and angles [°] for mo_170751lt_0m.

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C(10)-N(2)-C(11)  117.46(12)
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C(12)-C(1)-H(1)  119.7
C(2)-C(1)-H(1)  119.7
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C(3)-C(2)-H(3)  119.5
C(1)-C(2)-H(3)  119.5
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C(2)-C(3)-H(14)  120.3
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N(1)-C(5)-C(10)  121.13(12)
N(1)-C(5)-C(6)  115.66(13)
C(10)-C(5)-C(6)  123.18(12)
C(19)-C(6)-C(7)  118.90(13)
C(19)-C(6)-C(5)  118.93(12)
C(7)-C(6)-C(5)  122.10(13)
C(8)-C(7)-C(6)  120.09(14)
C(8)-C(7)-H(10)  120.0
C(6)-C(7)-H(10)  120.0
C(9)-C(8)-C(7)  120.37(14)
C(9)-C(8)-H(11)  119.8
C(7)-C(8)-H(11)  119.8
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Symmetry transformations used to generate equivalent atoms:

Table S19. Anisotropic displacement parameters (Å² x 10³) for mo_170751lt_0m. The
anisotropic displacement factor exponent takes the form: \[-2p^2\left[ h^2 a^* U_{11} + \ldots + 2hk a^* b^* U_{12} \right] \]

\[ \begin{array}{ccccccc}
 & U^{11} & U^{22} & U^{33} & U^{23} & U^{13} & U^{12} \\
N(1) & 17(1) & 20(1) & 17(1) & -1(1) & 3(1) & 0(1) \\
N(2) & 18(1) & 19(1) & 19(1) & 0(1) & 2(1) & 1(1) \\
C(1) & 20(1) & 24(1) & 24(1) & -6(1) & -2(1) & 2(1) \\
C(2) & 27(1) & 26(1) & 17(1) & -2(1) & 0(1) & 6(1) \\
C(3) & 26(1) & 22(1) & 19(1) & 1(1) & 4(1) & 1(1) \\
C(4) & 17(1) & 19(1) & 18(1) & -2(1) & 3(1) & 3(1) \\
C(5) & 16(1) & 19(1) & 17(1) & -2(1) & 5(1) & 2(1) \\
C(6) & 17(1) & 16(1) & 20(1) & 1(1) & 2(1) & 2(1) \\
C(7) & 26(1) & 24(1) & 20(1) & 1(1) & 3(1) & -3(1) \\
C(8) & 33(1) & 28(1) & 20(1) & -1(1) & -4(1) & -2(1) \\
C(9) & 22(1) & 24(1) & 33(1) & -3(1) & -5(1) & -4(1) \\
C(10) & 17(1) & 18(1) & 18(1) & -1(1) & 4(1) & 0(1) \\
C(11) & 18(1) & 18(1) & 17(1) & -2(1) & 3(1) & 3(1) \\
C(12) & 19(1) & 20(1) & 22(1) & -3(1) & 4(1) & 0(1) \\
C(13) & 19(1) & 18(1) & 18(1) & 0(1) & 0(1) & -4(1) \\
C(14) & 19(1) & 27(1) & 22(1) & 1(1) & 2(1) & -2(1) \\
C(15) & 27(1) & 33(1) & 18(1) & 3(1) & 4(1) & -6(1) \\
C(16) & 30(1) & 26(1) & 19(1) & 4(1) & -5(1) & -6(1) \\
C(17) & 21(1) & 26(1) & 27(1) & 1(1) & -4(1) & -1(1) \\
C(18) & 19(1) & 26(1) & 22(1) & 1(1) & 3(1) & -3(1) \\
C(19) & 19(1) & 18(1) & 21(1) & 1(1) & 3(1) & 3(1) \\
C(20) & 18(1) & 22(1) & 32(1) & 1(1) & 5(1) & -2(1) \\
\end{array} \]
Table S20. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for mol_170751lt_0m.

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Table S21. Crystal data and structure refinement for 190333lt.

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<tr>
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<tr>
<td></td>
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<tr>
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<tr>
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</tr>
<tr>
<td>Reflections collected</td>
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</tr>
<tr>
<td>Independent reflections</td>
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<tr>
<td>Completeness to theta = 25.242°</td>
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Absorption correction: Semi-empirical from equivalents
Max. and min. transmission: 0.7454 and 0.5925
Refinement method: Full-matrix least-squares on F^2
Data / restraints / parameters: 3037 / 0 / 209
Goodness-of-fit on F^2: 1.156
Final R indices [I>2sigma(I)]: R1 = 0.0611, wR2 = 0.1553
R indices (all data): R1 = 0.1048, wR2 = 0.2427
Extinction coefficient: 0.0030(10)
Largest diff. peak and hole: 0.459 and -0.472 e.Å^-3

**Table S22.** Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for 190333lt. U(eq) is defined as one third of the trace of the orthogonalized U_ij tensor.

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Table S23. Bond lengths [Å] and angles [°] for 190333lt.

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C(12)-C(1)-C(2)  119.5(3)
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C(2)-C(1)-H(1)  120.2
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C(1)-C(2)-H(15)  119.7
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C(3)-C(4)-C(5)  119.6(3)
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N(1)-C(5)-C(4)  126.1(3)
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C(10)-C(20)-H(12) 119.6
C(20)-C(21)-C(7) 120.2(3)
C(20)-C(21)-H(11) 119.9
C(7)-C(21)-H(11) 119.9

Symmetry transformations used to generate equivalent atoms:

Table S24. Anisotropic displacement parameters (Å² x 10³) for 190333lt. The anisotropic displacement factor exponent takes the form: 
-2\pi² \left[ h^2 a^* a U_{11} + ... + 2 h k a^* b^* U_{12} \right]

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Table S25. Hydrogen coordinates (x $10^4$) and isotropic displacement parameters ($\AA^2 \times 10^3$) for 190333lt.

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<th>x</th>
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