Supporting Information

Dual NHC/Photoredox Catalytic Synthesis of 1,4-Diketones Using an MR-TADF Photocatalyst (DiKTa)

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Experimental Section

General Synthetic Procedures. The following starting materials were synthesised according to literature procedures: benzoyl fluorides 32-42,\(^1\) \(\alpha\)-keto acid 43-48,\(^2\) all other reagents and solvents were obtained from commercial sources and used as received. Photocatalysts \([\text{Ir}(\text{dF(CF}_3\text{ppy})_2(\text{dtbbpy})(\text{PF}_6)]^3, [\text{Ir}(\text{ppy})_2(\text{dtbbpy})(\text{PF}_6)]^4, 4\text{CzIPN,}^5 \) and DiK\(\text{T}a^6\) were synthesised according to literature protocols. Flash column chromatography was carried out using silica gel (Silia-P from Silicycle, 60 Å, 40-63 µm). Analytical thin-layer-chromatography (TLC) was performed with silica plates with aluminum backings (250 µm with F-254 indicator). TLC visualization was accomplished by 254/365 nm UV lamp. GCMS analysis was conducted using a Shimadzu QP2010SE GC-MS equipped with a Shimadzu SH-Rtx-1 column (30 m × 0.25 mm). \(^1\)H, \(^{13}\)C and \(^{19}\)F NMR spectra were recorded on a Bruker Advance spectrometer (500 MHz for \(^1\)H, 125 MHz for \(^{13}\)C, 471 MHz for \(^{19}\)F and 202 MHz for \(^{31}\)P). The following abbreviations have been used for multiplicity assignments: “s” for singlet, “d” for doublet, “t” for triplet, “q” for quartet, “br” for broad, “m” for multiplet. \(^1\)H and \(^{13}\)C NMR spectra were referenced to the residual solvent peaks with respect to TMS (\(\delta = 0 \) ppm). Melting points were measured using open-ended capillaries on an Electrothermal 1101D Mel-Temp apparatus and are uncorrected. High-resolution mass spectrometry (HRMS) was performed by SIRCAMS at University of Edinburgh.

Photophysical measurements. Optically dilute solutions of concentrations on the order of 10\(^{-5}\) or 10\(^{-6}\) M of the photocatalysts were prepared in spectroscopic or HPLC grade solvents for emission analysis. Steady-state emission, excitation spectra and time-resolved emission spectra were recorded at 298 K using an Edinburgh Instruments FS5. Samples were excited at 410 nm for steady-state measurements and time-resolved measurements. Fitting of time-resolved luminescence measurements: Time-resolved PL measurements were fitted to a sum of exponentials decay model, with chi-squared (\(\chi^2\)) values between 1 and 2, using the Edinburgh FS5 software.
Photocatalysis Set-up

Photocatalysis experiments were conducted using a custom-built photoreactor, as shown in Figure S1 allowing for up to 8 parallel photochemical reactions (7 mL) at a time. The reactor is placed upon a magnetic stirrer plate allowing for reactions to be completed with stirring. Reactions are irradiated using Kessil PR160 LED sources (λ_{exc} = 427 nm). Two internal fans in the photoreactor ensure the reactions are maintained at room temperature.

![Photocatalysis Set-up](image)

Figure S1. Experimental set-up for photocatalysis reactions.
Optimization

General Procedure A

To an oven dried vial was added a base, a photocatalyst, phenylglyoxylic acid and an azolium salt. The vial was then evacuated and backfilled with nitrogen three times. Benzoyl fluoride and styrene were then added. In a separate oven dried Schlenk flask anhydrous solvent was sparged for 10 minutes and then added to the reaction vial and the vial was sealed further with parafilm. The reaction was then stirred under 427 nm irradiation at rt for 16 hours. After the reactions were completed, the products were analysed by ¹H NMR spectroscopy with 1,3,5-trimethoxybenzene as an internal standard.

Variation of photocatalyst

\[
\begin{array}{ccc}
\text{PC} & \text{NMR Yield / %} \\
\hline
\text{[Ir(dF(CF}_3\text{)ppy})_2(dtbbpy)](PF}_6\text{)} & 30 \\
\text{[Ir(ppy}_2\text{(dtbbpy)](PF}_6\text{)} & 30 \\
\text{4CzIPN} & 29 \\
\text{DiKTa} & 38 \\
\text{Eosin Y (λ}_{\text{exc}} = 525 \text{ nm)} & - \\
\end{array}
\]
Figure S2. Photocatalysts used in optimization.

**Variation of reagent ratios**

\[
\text{Ir(ppy)}_2\text{(dtbbpy)}\text{][PF}_6
\]

\[
\text{4CziPN}
\]

\[
\text{DiKTa}
\]

\[
\text{[Ir(dF(CF}_3\text{ppy})_2\text{(dtbbpy)}\text{][PF}_6}
\]

\[
\text{Eosin Y}
\]

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<th>Benzoyl Fluoride equiv.</th>
<th>Styrene equiv.</th>
<th>Acid equiv.</th>
<th>NMR yield / %</th>
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</tr>
<tr>
<td>4</td>
<td>2</td>
<td>1</td>
<td>46</td>
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<td>4</td>
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<td>1</td>
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</tr>
<tr>
<td>1</td>
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<td>4</td>
<td>10</td>
</tr>
<tr>
<td>1</td>
<td>4</td>
<td>2</td>
<td>&lt; 5</td>
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Variation of NHC catalyst

\[
\begin{align*}
\text{Ph} & \equiv \text{Ph} \\
4 \text{ equiv.} & \quad 2 \text{ equiv.} \\
\text{Cs}_2\text{CO}_3 (3 \text{ equiv.}) & \\
\text{DiKTa} & \\
\text{azolium salt} (20 \text{ mol%}) & \\
427 \text{ nm LEDs} & \\
\text{CH}_2\text{Cl}_2 (0.1 \text{ M}) & \\
\text{rt, 20 h} & \\
& \text{Ph}
\end{align*}
\]

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<th>Azolium Salt</th>
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<td>54</td>
<td>17</td>
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<td>55</td>
<td>Trace</td>
</tr>
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Figure S3. Azolium salts used in optimization.

Variation of Concentration

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Variation of Reagent Ratios

![Reaction scheme]

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Variation of Solvent

![Reaction scheme]

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<tr>
<td>DMF</td>
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<tr>
<td>Toluene</td>
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<td>THF</td>
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### Variation of Base

[Chemical structure]

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<tr>
<td>NaHCO$_3$</td>
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**Variation of Acyl Leaving Group**

![Chemical reaction diagram]

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<th>NMR Yield / %</th>
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<tr>
<td>Imidazole</td>
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<td>0</td>
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<td>Cl</td>
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<td>Trace</td>
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<tr>
<td>F</td>
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<td>OOCPh (anhydride)</td>
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<tr>
<td>Imidazole</td>
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<td>39</td>
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Control Reactions

Scheme S1. Model reaction using single enantiomer of azolium salt.

Observed side products
The following products were observed and identified using GCMS of crude reaction mixtures but were not isolated (Scheme S2). A dimerization side product corresponding to dimerization of the proposed radical addition intermediate is observed (Scheme 2A). The corresponding chalcone and dihydrochalcone were also identified and are assumed to arise from the same radical addition intermediate. Benzoin ester was also observed, and is expected to form from the NHC-catalysed benzoin reaction of aldehydes generated \textit{in-situ}, followed by esterification (Scheme 2B).

Scheme S2. (A) Dimerization of radical addition products. (B) Benzoin ester formation from in-situ generated aldehydes.

Additional side products were also detected by GCMS during the synthesis of unsymmetric 1,4-diones (Scheme S3). These included the symmetrical 1,4-dione, which is assumed to form via a similar mechanism to that proposed by Wu and co-workers,\textsuperscript{7} and the unsymmetrical 1,2-dione, which likely forms via radical-radical coupling between the acyl radical generated and the NHC-stabilized ketyl radical intermediate.
Scheme S3. Observed side products during synthesis of unsymmetric 1,4-diones.

**Unsuccessful Substrates**

α and β methyl styrene gave NMR yields of < 10% product and likely did not work due to increased steric congestion. Using oct-1-ene gave a complex mixture and only traces of what we assume to be the desired product, this was expected as unactivated alkenes are known to be more challenging for the radical-radical coupling step with the NHC-stabilised radical intermediate. 2-vinylthiophene and 4-vinylpyridine, did work to some degree but with significantly reduced isolated product yields of 13% and 17%, respectively. When using 4-cyanobenzoyl fluoride only traces of the desired product were detected by GCMS and the dominant product was the dimer formation; it is not clear why this is favoured. When using cyclohexanecarbonyl fluoride only traces of the desired product were detected by GCMS, with the dominant product again being the corresponding dimeric product; this is consistent with the use of alkyl acyl fluorides that are known to be more challenging in this type of catalytic system.
General Procedures for the Synthesis of 1,4-Diketones

*General Procedure B – variation of the alkene.*

To an oven dried vial was added Cs$_2$CO$_3$ (65.2 mg, 0.20 mmol, 2.0 equiv.), DiKTa (0.6 mg, 2 µmol, 2.0 mol%), phenylglyoxylic acid (22.5 mg, 0.15 mmol, 1.5 equiv.) and azolium salt 4 (7.4 mg, 0.020 mmol, 20 mol%). The vial was then evacuated and backfilled with nitrogen three times. Benzoyl fluoride (43.5 µL, 0.40 mmol, 4.0 equiv.) and an alkene (0.10 mmol, 1.0 equiv.) were then injected. In a separate dry Schlenk flask anhydrous CH$_2$Cl$_2$ was sparged for 10 minutes and then 1.0 mL was added to the reaction vial and the vial was sealed further with parafilm. The reaction was then stirred under 427 nm irradiation at rt for 16 hours. The reaction was then evaporated to dryness and THF (4 mL) and NaOH (2 mL, 2 M) were added. The resulting solution was stirred at 70 °C for 2 h. NaOH (5 mL, 2 M) was added, and the resulting mixture was extracted with CH$_2$Cl$_2$ (3 × 5 mL). The organic phases were combined and dried (MgSO$_4$) then concentrated *in vacuo*. Purification by silica chromatography EtOAc:Hexane afforded the desired products.

*General Procedure C – variation of the alkene without hydrolysis step.*

To an oven dried vial was added Cs$_2$CO$_3$ (65.2 mg, 0.20 mmol, 2.0 equiv.), DiKTa (0.6 mg, 2 µmol, 2.0 mol%), phenylglyoxylic acid (22.5 mg, 0.15 mmol, 1.5 equiv.) and azolium salt 4 (7.4 mg, 0.020 mmol, 20 mol%). The vial was then evacuated and backfilled with nitrogen three times. Benzoyl fluoride (43.5 µL, 0.40 mmol, 4.0 equiv.) and an alkene (0.10 mmol, 1.0 equiv.) were then injected. In a separate dry Schlenk flask anhydrous CH$_2$Cl$_2$ was sparged for 10 minutes and then 1.0 mL was added to the reaction vial and the vial was sealed further with parafilm. The reaction was then stirred under 427 nm irradiation at rt for 16 hours. NaOH (5 mL, 2 M) was added, and the resulting mixture was extracted with CH$_2$Cl$_2$ (3 × 5 mL). The organic phases were combined and dried (MgSO$_4$) then concentrated *in vacuo*. Purification by silica chromatography EtOAc:Hexane afforded the desired products.
General Procedure D – variation of the aroyl fluoride.

To an oven dried vial was added Cs$_2$CO$_3$ (65.2 mg, 0.20 mmol, 2.0 equiv.), DiKTa (0.6 mg, 2 µmol, 2.0 mol%), phenylglyoxylic acid (22.5 mg, 0.15 mmol, 1.5 equiv.), azolium salt 4 (7.4 mg, 0.020 mmol, 20 mol%) and, if solid, aroyl fluoride (0.40 mmol, 4.0 equiv.). The vial was then evacuated and backfilled with nitrogen three times. Aroyl fluoride (0.40 mmol, 4.0 equiv.), if liquid, and styrene (11.5 µL, 0.10 mmol, 1.0 equiv.) were then injected. In a separate dry Schlenk flask anhydrous CH$_2$Cl$_2$ was sparged for 10 minutes and then 1.0 mL was added to the reaction vial and the vial was sealed further with parafilm. The reaction was then stirred under 427 nm irradiation at rt for 16 hours. NaOH (5 mL, 2 M) was added, and the resulting mixture was extracted with CH$_2$Cl$_2$ (3 × 5 mL). The organic phases were combined and dried (MgSO$_4$) then concentrated in vacuo. Purification by silica chromatography EtOAc:Hexane afforded the desired products.

General Procedure E – variation of the α-keto acid.

To an oven dried vial was added Cs$_2$CO$_3$ (65.2 mg, 0.20 mmol, 2.0 equiv.), DiKTa (0.6 mg, 2 µmol, 2.0 mol%), α-keto acid (0.15 mmol, 1.5 equiv.), and azolium salt 4 (7.4 mg, 0.020 mmol, 20 mol%). The vial was then evacuated and backfilled with nitrogen three times. Benzoyl fluoride (43.5 µL, 0.40 mmol, 4.0 equiv.) and styrene (11.5 µL, 0.10 mmol, 1.0 equiv.) were then injected. In a separate dry Schlenk flask anhydrous CH$_2$Cl$_2$ was sparged for 10 minutes and then 1.0 mL was added to the reaction vial and the vial was sealed further with parafilm. The reaction was then stirred under 427 nm irradiation at rt for 16 hours. NaOH (5 mL, 2 M) was added, and the resulting mixture was extracted with CH$_2$Cl$_2$ (3 × 5 mL). The organic phases were combined and dried (MgSO$_4$) then concentrated in vacuo. Purification by silica chromatography EtOAc:Hexane afforded the desired products.

General Procedure F – [Ir(ppy)$_2$(dtbbpy)](PF$_6$)$_2$ catalysed conditions for symmetrical 1,4-diones.

To an oven dried vial was added Cs$_2$CO$_3$ (130 mg, 0.40 mmol, 2.0 equiv.), [Ir(ppy)$_2$(dtbbpy)](PF$_6$)$_2$ (2.7 mg, 3 µmol, 1.5 mol%), phenylglyoxylic acid (60.0 mg, 0.40 mmol, 2.0 equiv.) and azolium salt 4 (11.1 mg, 0.030 mmol, 15 mol%). The vial was then
evacuated and backfilled with nitrogen three times. Benzoyl fluoride (43.5 µL, 0.40 mmol, 2.0 equiv.), and an alkene (0.20 mmol, 1.0 equiv.) were then injected. In a separate dry Schlenk flask anhydrous toluene was sparged for 10 minutes and then 4.0 mL was added to the reaction vial and the vial was sealed further with parafilm. The reaction was then stirred under 456 nm irradiation at rt for 16 hours. NaOH (5 mL, 2 M) was added, and the resulting mixture was extracted with CH₂Cl₂ (3 × 5 mL). The organic phases were combined and dried (MgSO₄) then concentrated in vacuo. Purification by silica chromatography EtOAc:Hexane afforded the desired products.

**General Procedure G – [Ir(ppy)₂(dtbbpy)](PF₆) catalysed conditions for unsymmetrical 1,4-diones.**

To an oven dried vial was added Cs₂CO₃ (130 mg, 0.40 mmol, 2.0 equiv.), [Ir(ppy)₂(dtbbpy)](PF₆) (2.7 mg, 3 µmol, 1.5 mol%), α-keto acid (0.60 mmol, 3.0 equiv.), azolium salt 4 (11.1 mg, 0.030 mmol, 15 mol%) and, if solid, aroyl fluoride (0.60 mmol, 3.0 equiv.). The vial was then evacuated and backfilled with nitrogen three times. Aroyl fluoride (0.60 mmol, 3.0 equiv.), if liquid, and styrene (23 µL, 0.20 mmol, 1.0 equiv.) were then injected. In a separate dry Schlenk flask anhydrous toluene was sparged for 10 minutes and then 4.0 mL was added to the reaction vial and the vial was sealed further with parafilm. The reaction was then stirred under 456 nm irradiation at rt for 16 hours. NaOH (5 mL, 2 M) was added, and the resulting mixture was extracted with CH₂Cl₂ (3 × 5 mL). The organic phases were combined and dried (MgSO₄) then concentrated in vacuo. Purification by silica chromatography EtOAc:Hexane afforded the desired products.
Figure S4. Emission quenching data of DiKTa by sequential addition of phenylglyoxylic acid in CH₂Cl₂. λ_{exc} = 410 nm.
Figure S5. Stern-Volmer plot of the quenching of the emission of DiKTa in CH$_2$Cl$_2$ by sequential addition of phenylglyoxylic acid.
Figure S6. Emission quenching data of DiKTa by sequential addition of benzoyl fluoride in CH$_2$Cl$_2$. $\lambda_{\text{exc}}$ = 410 nm.
Figure S7. Stern-Volmer plot of the quenching of the emission of DiKTa in CH₂Cl₂ by sequential addition of benzoyl fluoride.
Figure S8. Emission quenching data of DiKTa by sequential addition of styrene in CH$_2$Cl$_2$. $\lambda_{\text{exc}} = 410$ nm.
Figure S9. Stern-Volmer plot of the quenching of the emission of DiKTa in CH₂Cl₂ by sequential addition of styrene.
Figure S10. Time resolved PL decay of DiKTa recorded in CH$_2$Cl$_2$ under air in 10-5 M solutions with $\lambda_{exc} = 375$ nm.

Quenching constant:

**DiKTa** with phenylglyoxylic acid

$\tau_{PL} = 5.6 \times 10^{-9}$ s

$K_{SV} = 0.0213$ mmol$^{-1}$ dm$^3$

$k_q = K_{SV}/ \tau_{PL} = 3.8 \times 10^9$ mol$^{-1}$ dm$^3$ s$^{-1}$. 
Compound Characterization

2-Phenyl-1,4-diphenylbutane-1,4-dione (5):

![Structural formula of 2-Phenyl-1,4-diphenylbutane-1,4-dione (5)]

Synthesised using general procedure B to give 18.2 mg of 5 (58%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. Mp 121-124 °C {Lit. Mp9 124-126 °C}. $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 3.34 (1H, dd, $J$ = 18.0 Hz, 3.6 Hz, CHCH$_A$H$_B$), 4.25 (1H, dd, $J$ = 18.0 Hz, 10.1 Hz, CHCH$_A$H$_B$), 5.35 (1H, dd, $J$ = 10.1 Hz, 3.7 Hz, C$_H$CH$_A$H$_B$), 7.23 – 7.28 (1H, m, ArH), 7.34 (2H, dd, $J$ = 8.5 Hz, 6.8 Hz, ArH), 7.37 – 7.55 (7H, m, ArH), 7.56 – 7.61 (1H, m, ArH), 7.98 – 8.04 (2H, m, ArH), 8.05 – 8.09 (2H, m, ArH).

$^{13}$C {$^1$H} NMR (126 MHz, CDCl$_3$) δ (ppm): 43.9 (CHCH$_2$), 48.7 (CHCH$_2$), 127.4 (ArC), 128.2 (ArC), 128.3 (ArC), 128.5 (ArC), 128.6 (ArC), 129.0 (ArC), 129.2 (ArC), 130.0 (ArC), 132.9 (ArC), 133.3 (ArC), 136.4 (ArC), 138.6 (ArC), 198.1 (C=O), 198.9 (C=O).

Data matches that previously reported.$^{10}$

2-(4-Bromophenyl)-1,4-diphenylbutane-1,4-dione (7):

![Structural formula of 2-(4-Bromophenyl)-1,4-diphenylbutane-1,4-dione (7)]

Synthesised using general procedure B to give 17.6 mg of 7 (45%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. Mp 123-126 °C. $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 3.30 (1H, dd, $J$ = 18.0 Hz, 4.0 Hz, CHCH$_A$H$_B$), 4.16 (1H, dd, $J$ = 18.0 Hz, 9.7 Hz, CHCH$_A$H$_B$), 5.30 (1H, dd, $J$ = 9.7 Hz, 4.0 Hz, CHCH$_A$H$_B$), 7.22 – 7.26 (2H, m, ArH), 7.38 – 7.48 (6H, m, ArH), 7.49 – 7.54 (1H, m, ArH), 7.54 – 7.59 (1H, m, ArH), 7.95 – 8.04 (4H, m, ArH).
$^{13}$C-$^1$H NMR (126 MHz, CDCl$_3$) δ (ppm): 43.6 (CHCH$_2$), 48.1 (CHCH$_2$), 121.4 (ArC), 128.2 (ArC), 128.60 (ArC), 128.62 (ArC), 128.9 (ArC), 130.0 (ArC), 132.3 (ArC), 133.1 (ArC), 133.3 (ArC), 136.3 (ArC), 136.4 (ArC), 137.7 (ArC), 197.7 (C=O), 198.6 (C=O). Data matches that previously reported.$^{11}$

2-(4-Fluorophenyl)-1,4-diphenylbutane-1,4-dione (8):

![Structure of 2-(4-Fluorophenyl)-1,4-diphenylbutane-1,4-dione (8)]

Synthesised using general procedure B to give 15.3 mg of 8 (46%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. Mp 113-116 ºC. $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 3.31 (1H, dd, J = 18.0 Hz, 4.0 Hz, CHCH$_2$H$^B$), 4.17 (1H, dd, J = 18.0 Hz, 9.8 Hz, CHCH$^A$H$^B$), 5.32 (1H, dd, J = 9.8 Hz, 3.9 Hz, CHCH$^A$H$^B$), 6.97 – 7.04 (2H, m, ArH), 7.31 – 7.36 (2H, m, ArH), 7.39 – 7.48 (4H, m, ArH), 7.49 – 7.54 (1H, m, ArH), 7.94 – 8.06 (4H, m, ArH).

$^{19}$F NMR (471 MHz, CDCl$_3$) δ (ppm): -114.9

$^{13}$C-$^1$H NMR (126 MHz, CDCl$_3$) δ (ppm): 43.8 (CHCH$_2$), 47.8 (CHCH$_2$), 116.1 (d, J = 21.5 Hz, ArC), 128.2 (ArC), 128.60 (ArC), 128.64 (ArC), 128.9 (ArC), 129.8 (d, J = 8.1 Hz, ArC), 133.1 (ArC), 133.4 (ArC), 134.3 (d, J = 3.1 Hz, ArC), 136.3 (ArC), 136.4 (ArC), 162.1 (d, J = 245.8 Hz, ArC), 197.9 (C=O), 198.9 (C=O).

Data matches that previously reported.$^{11}$

1,4-Diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione (9):

![Structure of 1,4-Diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione (9)]
Synthesised using general procedure B to give 16.0 mg of 9 (42%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 3.36 (1H, dd, \(J = 18.0\) Hz, 4.0 Hz, \(\text{CHCH}^\text{A} \text{H}^\text{B}\)), 4.24 (1H, dd, \(J = 18.0\) Hz, 9.8 Hz, \(\text{CHCH}^\text{A} \text{H}^\text{B}\)), 5.44 (1H, dd, \(J = 9.7\) Hz, 4.0 Hz, \(\text{CHCH}^\text{A} \text{H}^\text{B}\)), 7.43 – 7.51 (4H, m, Ar\(\text{H}\)), 7.54 (3H, dd, \(J = 9.6\) Hz, 7.8 Hz, Ar\(\text{H}\)), 7.57 – 7.62 (3H, m, Ar\(\text{H}\)), 7.99 – 8.03 (2H, m, Ar\(\text{H}\)), 8.03 – 8.07 (2H, m, Ar\(\text{H}\)).

\(^1\)F NMR (471 MHz, CDCl\(_3\)) \(\delta\) (ppm): -62.6

\(^1\)C\{\(^1\)H\} NMR (126 MHz, CDCl\(_3\)) \(\delta\) (ppm): 43.7 (CH\(\text{CCH}_2\)), 48.3 (CH\(\text{CH}_2\)), 123.9 (q, \(J = 272.4\) Hz CF\(_3\)), 126.1 (q, \(J = 3.5\) Hz, Ar\(\text{C}\)), 128.2 (Ar\(\text{C}\)), 128.66 (Ar\(\text{C}\)), 128.71 (Ar\(\text{C}\)), 128.9 (Ar\(\text{C}\)), 129.7 (q, \(J = 32.7\) Hz, Ar\(\text{C}\)), 133.3 (Ar\(\text{C}\)), 133.5 (Ar\(\text{C}\)), 136.1 (Ar\(\text{C}\)), 136.2 (Ar\(\text{C}\)), 142.7 (Ar\(\text{C}\)), 197.7 (C=O), 198.6 (C=O).

Data matches that previously reported.\(^\text{10}\)

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1,4-Diphenyl-2-(pyridin-2-yl)butane-1,4-dione (10):

![1,4-Diphenyl-2-(pyridin-2-yl)butane-1,4-dione (10)](image)

Synthesised using general procedure B to give 17.4 mg of 10 (55%) after column chromatography using EtOAc:Hexane (30:70).

Yellow oil. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 3.54 (1H, dd, \(J = 18.0\) Hz, 4.1 Hz, \(\text{CHCH}^\text{A} \text{H}^\text{B}\)), 4.26 (1H, dd, \(J = 18.0\) Hz, 9.6 Hz, \(\text{CHCH}^\text{A} \text{H}^\text{B}\)), 5.60 (1H, dd, \(J = 9.6\) Hz, 4.1 Hz, \(\text{CHCH}^\text{A} \text{H}^\text{B}\)), 7.17 (1H, ddd, \(J = 7.6\) Hz, 4.9 Hz, 1.1 Hz, Ar\(\text{H}\)), 7.38 (1H, d, \(J = 7.7\) Hz, Ar\(\text{H}\)), 7.42 – 7.50 (4H, m, Ar\(\text{H}\)), 7.51 – 7.56 (1H, m, Ar\(\text{H}\)), 7.56 – 7.61 (1H, m, Ar\(\text{H}\)), 7.64 (1H, td, \(J = 7.7\) Hz, 1.8 Hz, Ar\(\text{H}\)), 8.00 – 8.06 (2H, m, Ar\(\text{H}\)), 8.10 – 8.14 (2H, m, Ar\(\text{H}\)), 8.59 (1H, q, \(J = 1.7\) Hz, Ar\(\text{H}\)).

\(^1\)C\{\(^1\)H\} NMR (126 MHz, CDCl\(_3\)) \(\delta\) (ppm): 42.2 (CH\(\text{CH}_2\)), 51.3 (CH\(\text{CH}_2\)), 122.2 (Ar\(\text{C}\)), 123.0 (Ar\(\text{C}\)), 128.2 (Ar\(\text{C}\)), 128.57 (Ar\(\text{C}\)), 128.58 (Ar\(\text{C}\)), 129.1 (Ar\(\text{C}\)), 133.0 (Ar\(\text{C}\)), 133.3 (Ar\(\text{C}\)), 136.4 (Ar\(\text{C}\)), 137.1 (Ar\(\text{C}\)), 150.0 (Ar\(\text{C}\)), 158.5 (Ar\(\text{C}\)), 197.8 (C=O), 198.0 (C=O).

Data matches that previously reported.\(^\text{7}\)
1,4-Diphenyl-2-(p-tolyl)butane-1,4-dione (11):

\[
\begin{array}{c}
\text{Ph} \\
\text{O} \\
\text{H} \\
\text{Me} \\
\text{Ph}
\end{array}
\]

Synthesised using general procedure B to give 18.7 mg of 11 (57%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. \(^1{\text{H}}\)NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 2.31 (3H, s, CH\(_3\)), 3.31 (1H, dd, \(J = 18.0\) Hz, 3.7 Hz, CH\(\text{CH}^\text{A}\)H\(\text{B}\)), 4.22 (1H, dd, \(J = 18.0\) Hz, 10.0 Hz, CH\(\text{CH}^\text{A}\)H\(\text{B}\)), 5.32 (1H, dd, \(J = 10.7\) Hz, 3.7 Hz, CH\(\text{CH}^\text{A}\)H\(\text{B}\)), 7.14 (2H, d, \(J = 7.8\) Hz, Ar\(H\)), 7.25 – 7.28 (2H, m, Ar\(H\)), 7.40 – 7.54 (5H, m, Ar\(H\)), 7.56 – 7.60 (1H, m, Ar\(H\)), 7.99 – 8.02 (2H, m, Ar\(H\)), 8.04 – 8.07 (2H, m, Ar\(H\)).

\(^{13}\text{C}\)\(^1{\text{H}}\) NMR (126 MHz, CDCl\(_3\)) \(\delta\) (ppm): 21.1 (CH\(_3\)), 43.9 (CHCH\(_2\)), 48.3 (CHCH\(_2\)), 128.1 (Ar\(C\)), 128.2 (Ar\(C\)), 128.5 (Ar\(C\)), 128.6 (Ar\(C\)), 128.9 (Ar\(C\)), 129.9 (Ar\(C\)), 132.9 (Ar\(C\)), 133.2 (Ar\(C\)), 135.6 (Ar\(C\)), 136.5 (Ar\(C\)), 137.1 (Ar\(C\)), 198.2 (C=O), 199.0 (C=O).

Data matches that previously reported.\(^7\)

2-(4-(Tert-butyl)phenyl)-1,4-diphenylbutane-1,4-dione (12):

\[
\begin{array}{c}
\text{Ph} \\
\text{O} \\
\text{t-Bu} \\
\text{Ph}
\end{array}
\]

Synthesised using general procedure B to give 15.9 mg of 12 (43%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. Mp 106-109 °C. \(^1{\text{H}}\)NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm): 1.30 (9H, s, C(CH\(_3\))\(_3\)), 3.33 (1H, dd, \(J = 18.1\) Hz, 3.6 Hz, CH\(\text{CH}^\text{A}\)H\(\text{B}\)), 4.24 (1H, dd, \(J = 18.1\) Hz, 10.2 Hz, CH\(\text{CH}^\text{A}\)H\(\text{B}\)), 5.33 (1H, dd, \(J = 10.3\) Hz, 3.6 Hz, CH\(\text{CH}^\text{A}\)H\(\text{B}\)), 7.29 – 7.37 (4H, m, Ar\(H\)), 7.39 – 7.61 (6H, m, Ar\(H\)), 7.97 – 8.05 (2H, m, Ar\(H\)), 8.05 – 8.11 (2H, m, Ar\(H\)).
$^{13}$C\{}^{1}H\} NMR (126 MHz, CDCl\textsubscript{3}) $\delta$ (ppm): 31.3 (CH\textsubscript{3}), 34.5 (C(CH\textsubscript{3})\textsubscript{3}), 44.0 (CHCH\textsubscript{2}), 48.1 (CHCH\textsubscript{2}), 126.1 (ArC), 127.8 (ArC), 128.2 (ArC), 128.5 (ArC), 128.6 (ArC), 129.0 (ArC), 132.8 (ArC), 133.2 (ArC), 135.4 (ArC), 136.5 (ArC), 136.6 (ArC), 150.2 (ArC) 198.2 (C=O), 199.0 (C=O).

Data matches that previously reported.\textsuperscript{7}

2-(4-(Chloromethyl)phenyl)-1,4-diphenylbutane-1,4-dione (13):

![Chemical Structure Image]

Synthesised using general procedure C to give 14.9 mg of 13 (41%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. \textbf{Mp} 155-160 °C. $^{1}$H NMR (500 MHz, CDCl\textsubscript{3}) $\delta$ (ppm): 3.33 (1H, dd, $J$ = 18.1 Hz, 3.8 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 4.23 (1H, dd, $J$ = 18.0 Hz, 10.0 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 4.55 (2H, s, CH\textsubscript{2}Cl), 5.37 (1H, dd, $J$ = 10.0 Hz, 3.8 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 7.34 – 7.42 (4H, m, ArH), 7.50 – 7.56 (1H, m, ArH), 7.56 – 7.64 (1H, m, ArH), 7.98 – 8.03 (2H, m, ArH), 8.03 – 8.07 (2H, m, ArH).

$^{13}$C\{}^{1}H\} NMR (126 MHz, CDCl\textsubscript{3}) $\delta$ (ppm): 43.8 (CHCH\textsubscript{2}), 45.8 (CH\textsubscript{2}Cl), 48.3 (CHCH\textsubscript{2}), 128.2 (ArC), 128.60 (ArC), 128.63 (ArC), 128.9 (ArC), 129.5 (ArC), 133.1 (ArC), 133.4 (ArC), 136.3 (ArC), 136.4 (ArC), 136.6 (ArC), 138.9 (ArC), 197.9 (C=O), 198.7 (C=O).

Data matches that previously reported.\textsuperscript{7}

1,4-Diphenyl-2-(o-tolyl)butane-1,4-dione (14):

![Chemical Structure Image]
Synthesised using general procedure B to give 12.8 mg of 14 (39%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 2.57 (3H, s), 3.12 (1H, dd, $J = 18.0$ Hz, 2.9 Hz, CH$^A$H$^B$), 4.18 (1H, dd, $J = 18.0$ Hz, 10.6 Hz, CH$^A$H$^B$), 5.49 (1H, dd, $J = 10.5$ Hz, 2.9 Hz, CH$^A$H$^B$), 7.12 (1H, d, $J = 2.9$ Hz, ArH), 7.17 (1H, ddd, $J = 7.5$ Hz, 5.4 Hz, 3.4 Hz, ArH), 7.27 (2H, dd, $J = 13.6$ Hz, ArH), 7.49 (3H, dt, $J = 9.2$ Hz, 7.4 Hz, ArH), 7.57-7.61 (1H, m, ArH), 7.90 – 7.95 (2H, m, ArH).

$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 19.8 (CH$_3$), 42.5 (CHCH$_2$), 45.2 (CHCH$_2$), 126.9 (ArC), 127.4 (ArC), 127.5 (ArC), 128.2 (ArC), 128.5 (ArC), 128.6 (ArC), 128.7 (ArC), 131.3 (ArC), 132.8 (ArC), 133.3 (ArC), 135.1 (ArC), 136.5 (ArC), 136.6 (ArC), 137.2 (ArC), 198.2 (C=O), 199.5 (C=O).

Data matches that previously reported.  

2-(4-Methoxyphenyl)-1,4-diphenylbutane-1,4-dione (15):

![Structure of 2-(4-Methoxyphenyl)-1,4-diphenylbutane-1,4-dione (15)](image)

Synthesised using general procedure B to give 8.9 mg of 15 (26%) and general procedure F to give 40.0 mg of 15 (58%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. Mp 134-137 °C {Lit. Mp$^{12}$ 138-139 °C}. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 3.29 (1H, dd, $J = 18.0$ Hz, 3.8 Hz, CH$^A$H$^B$), 3.76 (3H, s, OCH$_3$), 4.18 (1H, dd, $J = 18.0$ Hz, 10.0 Hz, CH$^A$H$^B$), 5.28 (1H, dd, $J = 9.9$ Hz, 3.8 Hz, CH$^A$H$^B$), 6.82 – 6.87 (2H, m, ArH), 7.26 – 7.29 (2H, m, ArH), 7.38 – 7.51 (5H, m, ArH), 7.52 – 7.58 (1H, m, ArH), 7.96 – 8.01 (2H, m, ArH), 8.01 – 8.05 (2H, m, ArH).

$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 43.9 (CH$\mathrm{CH}_2$), 47.8 (CH$\mathrm{CH}_2$), 55.3 (OCH$_3$), 114.6 (ArC), 128.2 (ArC), 128.5 (ArC), 128.6 (ArC), 128.9 (ArC), 129.3 (ArC), 130.5 (ArC), 132.9 (ArC), 133.3 (ArC), 136.48 (ArC), 136.51 (ArC), 158.8 (ArC), 198.3 (C=O), 199.1 (C=O).
Data matches that previously reported.\textsuperscript{11}

1-([1,1'-Biphenyl]-4-yl)-2,4-diphenylbutane-1,4-dione (16):

\begin{align*}
\text{Ph} & \quad \text{O} \\
\text{Ph} & \quad \text{O} \\
\text{Ph} & \quad \text{Ph}
\end{align*}

Synthesised using general procedure D to give 20.7 mg of 16 (53\%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. \textbf{Mp} 196-199 °C {Lit. Mp\textsuperscript{13} 200 °C}. \textbf{\textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) $\delta$ (ppm):} 3.33 (1H, dd, $J = 18.0$ Hz, 3.7 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 4.25 (1H, dd, $J = 18.0$ Hz, 10.1 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 5.36 (1H, dd, $J = 10.1$ Hz, 3.6 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 7.21 – 7.25 (1H, m, ArH), 7.33 (2H, dd, $J = 8.5$ Hz, 6.8 Hz, ArH), 7.37 – 7.42 (3H, m, ArH), 7.42 – 7.49 (4H, m, ArH), 7.54 – 7.61 (3H, m, ArH), 7.61 – 7.65 (2H, m, ArH), 7.97 – 8.02 (2H, m, ArH), 8.09 – 8.14 (2H, m, ArH).

\textbf{\textsuperscript{13}C\{\textsuperscript{1}H\} NMR (126 MHz, CDCl\textsubscript{3}) $\delta$ (ppm):} 43.9 (CHCH\textsubscript{2}), 48.7 (CHCH\textsubscript{2}), 122.6 (ArC), 127.3 (ArC), 127.4 (ArC), 128.1 (ArC), 128.2 (ArC), 128.3 (ArC), 128.6 (ArC), 128.9 (ArC), 129.3 (ArC), 129.5 (ArC), 133.3 (ArC), 135.1 (ArC), 136.5 (ArC), 138.7 (ArC), 140.0 (ArC), 143.3 (ArC), 145.6 (ArC), 198.1 (C=O), 198.5 (C=O).

Data matches that previously reported.\textsuperscript{14}

1-(Naphthalen-2-yl)-2,4-diphenylbutane-1,4-dione (17):

\begin{align*}
\text{Ph} & \quad \text{O} \\
\text{Ph} & \quad \text{O} \\
\text{Naphthalen-2-yl} &
\end{align*}

Synthesised using general procedure D to give 14.9 mg of 17 (41\%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. \textbf{\textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) $\delta$ (ppm):} 3.37 (1H, dd, $J = 18.1$ Hz, 3.7 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 4.28 (1H, dd, $J = 18.0$ Hz, 10.0 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 5.50 (1H, dd, $J = 10.0$ Hz, 3.7 Hz, CHCH\textsubscript{A}H\textsubscript{B}), 7.22 (1H, t, $J = 7.7$ Hz, ArH), 7.32 (2H, t, $J = 7.6$ Hz, ArH), 7.38 –
7.60 (7H, m, ArH), 7.80 – 7.87 (2H, m, ArH), 7.93 (1H, d, J = 8.0 Hz, ArH) 7.99 – 8.05 (2H, m, ArH), 8.07 (1H, dd, J = 8.7 Hz, 1.8 Hz, ArH), 8.61 (1H, d, J = 1.7 Hz, ArH).

$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) δ (ppm): 43.9 (CH$_2$C$_6$H$_4$), 48.8 (CH$_2$CH$_2$), 124.7 (ArC), 126.6 (ArC), 127.4 (ArC), 127.7 (ArC), 128.2 (ArC), 128.3 (ArC), 128.36 (ArC), 128.39 (ArC), 129.3 (ArC), 129.7 (ArC), 130.8 (ArC), 132.5 (ArC), 133.3 (ArC), 133.8 (ArC), 135.5 (ArC), 136.5 (ArC), 138.8 (ArC), 198.2 (C=O), 198.9 (C=O).

Data matches that previously reported.$^{14}$

1-(Naphthalen-1-yl)-2,4-diphenylbutane-1,4-dione (18):

![Chemical Structure](image)

Synthesised using general procedure D to give 9.8 mg of 18 (27%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. _Mp_ 129-132 °C $^1$H NMR (500 MHz, CDCl$_3$) δ (ppm): 3.38 (1H, dd, J = 18.0 Hz, 3.4 Hz, CHCH$_2$H$_B$), 4.40 (1H, dd, J = 18.0 Hz, 10.5 Hz, CHCH$_2$H$_B$), 5.35 (1H, dd, J = 10.5 Hz, 3.4 Hz, CHCH$_2$H$_B$), 7.19 – 7.25 (1H, m, ArH), 7.31 (2H, d, J = 7.6 Hz, ArH), 7.37 – 7.42 (2H, m, ArH), 7.50 (5H, dddd, J = 9.4 Hz, 7.4 Hz, 3.2 Hz, 1.6 Hz, ArH), 7.59 – 7.63 (1H, m, ArH) 7.83 (1H, dd, J = 8.3 Hz, 1.6 Hz, ArH), 7.95 (1H, d, J = 8.2 Hz, ArH), 8.03 – 8.09 (2H, m, ArH), 8.20 (1H, dd, J = 7.2 Hz, 1.2 Hz, ArH), 8.36 (1H, dd, J =8.3 Hz, 1.6 Hz, ArH).

$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) δ (ppm): 43.5 (CH$_2$C$_6$H$_4$), 52.3 (CH$_2$CH$_2$), 124.4 (ArC), 125.7 (ArC), 126.2 (ArC), 127.5 (ArC), 127.6 (ArC), 127.7 (ArC), 128.19 (ArC), 128.21 (ArC), 128.3 (ArC), 128.7 (ArC), 129.1 (ArC), 130.7 (ArC), 132.3 (ArC), 133.3 (ArC), 133.8 (ArC), 136.4 (ArC), 136.6 (ArC), 137.9 (ArC), 198.3 (C=O), 202.4 (C=O).

HRMS (ESI) C$_{26}$H$_{20}$O$_2$ [M+H]$^+$ found XX, requires XX (+XX ppm)
1-(4-Fluorophenyl)-2,4-diphenylbutane-1,4-dione (19):

Synthesised using general procedure D to give 17.0 mg of 19 (51%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. \( ^1H \text{NMR (500 MHz, CDCl}_3 \) \( \delta \) (ppm): 3.33 (1H, dd, \( J = 18.1 \text{ Hz, 3.5 Hz, CHCH}_A^A \text{H}_B^B \)), 4.24 (1H, dd, \( J = 18.1 \text{ Hz, 10.2 Hz, CHCH}_A^A \text{H}_B^B \)), 5.29 (1H, dd, \( J = 10.2 \text{ Hz, 3.5 Hz, C}_A^A \text{H}_B^B \)), 7.05 – 7.09 (2H, m, Ar\( H \)), 7.22 – 7.26 (1H, m, Ar\( H \)), 7.30 – 7.37 (4H, m, Ar\( H \)), 7.43 – 7.48 (2H, m, Ar\( H \)), 7.53 – 7.59 (1H, m, Ar\( H \)), 7.96 – 8.00 (2H, m, Ar\( H \)), 8.04 – 8.08 (2H, m, Ar\( H \)).

\( ^19F \text{NMR (471 MHz, CDCl}_3 \) \( \delta \) (ppm): -105.4.

\( ^13C\{^1H\} \text{NMR (126 MHz, CDCl}_3 \) \( \delta \) (ppm): 43.9 (CHCH\( _2 \)), 48.7 (CHCH\( _2 \)), 115.6 (d, \( J = 21.8 \text{ Hz, ArC} \)), 127.5 (Ar\( C \)), 128.2 (d, \( J = 2.6 \text{ Hz, ArC} \)), 128.6 (Ar\( C \)), 129.3 (Ar\( C \)), 131.6 (d, \( J = 9.2 \text{ Hz, ArC} \)), 133.4 (Ar\( C \)), 136.3 (Ar\( C \)), 138.4 (Ar\( C \)), 165.6 (d, \( J = 254.3 \text{ Hz, ArC} \)), 197.4 (C=O), 198.1 (C=O).

Data matches that previously reported.\(^14\)

1-(4-Chlorophenyl)-2,4-diphenylbutane-1,4-dione (20):

Synthesised using general procedure D to give 17.1 mg of 20 (49%) after column chromatography using EtOAc:Hexane (3:97).

Yellow oil. \( ^1H \text{NMR (500 MHz, CDCl}_3 \) \( \delta \) (ppm): 3.31 (1H, dd, \( J = 18.1 \text{ Hz, 3.6 Hz, CHCH}_A^A \text{H}_B^B \)), 4.21 (1H, dd, \( J = 18.1 \text{ Hz, 10.2 Hz, CHCH}_A^A \text{H}_B^B \)), 5.26 (1H, dd, \( J = 10.2 \text{ Hz, 3.6 Hz, CHCH}_A^A \text{H}_B^B \)), 7.31 – 7.35 (4H, m, Ar\( H \)), 7.35 – 7.39 (2H, m, Ar\( H \)), 7.41 – 7.48 (3H, m, Ar\( H \)), 7.53 – 7.60 (2H, m, Ar\( H \)), 7.94 – 8.00 (4H, m, Ar\( H \)).
\(^{13}\text{C}\{^1\text{H}\} \text{ NMR (126 MHz, CDCl}_3\} \delta (\text{ppm}):\) 43.9 (CH\text{CH}_2), 48.8 (CH\text{CH}_2), 127.6 (ArC), 128.2 (ArC), 128.6 (ArC), 128.9 (ArC), 129.3 (ArC), 130.4 (d, \text{J} = 9.2 \text{ Hz, ArC}), 133.4 (ArC), 134.8 (ArC), 136.3 (ArC), 138.3 (ArC), 139.3 (ArC), 197.8 (C=O), 198.0 (C=O).

Data matches that previously reported.\(^{15}\)

\textbf{1-(4-Bromophenyl)-2,4-diphenylbutane-1,4-dione (21):}

\begin{center}
\begin{tikzpicture}
\draw[thick,black] (0,0) -- (1,0) -- (1,1) -- (0,1) -- cycle;
\draw[thick,black] (1,0) -- (2,0) -- (2,1) -- (1,1) -- cycle;
\draw[thick,black] (0,1) -- (0,2) -- (1,2) -- (1,1) -- cycle;
\draw[thick,black] (2,0) -- (2,1) -- (3,1) -- (3,0) -- cycle;
\draw[thick,black] (0,2) -- (0,3) -- (1,3) -- (1,2) -- cycle;
\draw[thick,black] (2,1) -- (2,2) -- (3,2) -- (3,1) -- cycle;
\draw[thick,black] (3,0) -- (3,1) -- (4,1) -- (4,0) -- cycle;
\draw[thick,black] (3,1) -- (3,2) -- (4,2) -- (4,1) -- cycle;
\draw[thick,black] (4,0) -- (4,1) -- (5,1) -- (5,0) -- cycle;
\draw[thick,black] (4,1) -- (4,2) -- (5,2) -- (5,1) -- cycle;
\end{tikzpicture}
\end{center}

Synthesised using general procedure D to give 9.8 mg of 21 (25%) and general procedure G to give 30.7 mg of 21 (58%) after column chromatography using EtOAc:Hexane (3:97).

Colourless foam. \(^1\text{H NMR (500 MHz, CDCl}_3\} \delta (\text{ppm}):\) 3.31 (1H, dd, J = 18.1 Hz, 3.5 Hz, CH\text{CH}_A\text{H}_B), 4.21 (1H, dd, J = 18.1 Hz, 10.2 Hz, CH\text{CH}_A\text{H}_B), 5.24 (1H, dd, J = 10.2 Hz, 3.5 Hz, CH\text{CH}_A\text{H}_B), 7.24 (1H, dd, J = 5.9 Hz, 2.9 Hz, Ar\text{H}), 7.28 – 7.35 (4H, m, Ar\text{H}), 7.45 (2H, t, J = 7.7 Hz, Ar\text{H}), 7.55 (3H, dd, J = 8.5 Hz, 6.7 Hz Ar\text{H}), 7.86 – 7.92 (2H, m, Ar\text{H}), 7.94 – 8.02 (2H, m, Ar\text{H}).

\(^{13}\text{C}\{^1\text{H}\} \text{ NMR (126 MHz, CDCl}_3\} \delta (\text{ppm}):\) 43.9 (CH\text{CH}_2), 48.8 (CH\text{CH}_2), 127.6 (ArC), 128.2 (ArC), 128.6 (ArC), 129.3 (ArC), 130.5 (ArC), 131.9 (ArC), 133.4 (ArC), 135.2 (ArC), 136.3 (ArC), 138.2 (ArC), 139.3 (ArC), 197.98 (C=O), 198.02 (C=O).

Data matches that previously reported.\(^{16}\)

\textbf{1-(4-Iodophenyl)-2,4-diphenylbutane-1,4-dione (22):}

\begin{center}
\begin{tikzpicture}
\draw[thick,black] (0,0) -- (1,0) -- (1,1) -- (0,1) -- cycle;
\draw[thick,black] (1,0) -- (2,0) -- (2,1) -- (1,1) -- cycle;
\draw[thick,black] (0,1) -- (0,2) -- (1,2) -- (1,1) -- cycle;
\draw[thick,black] (2,0) -- (2,1) -- (3,1) -- (3,0) -- cycle;
\draw[thick,black] (0,2) -- (0,3) -- (1,3) -- (1,2) -- cycle;
\draw[thick,black] (2,1) -- (2,2) -- (3,2) -- (3,1) -- cycle;
\draw[thick,black] (3,0) -- (3,1) -- (4,1) -- (4,0) -- cycle;
\draw[thick,black] (3,1) -- (3,2) -- (4,2) -- (4,1) -- cycle;
\draw[thick,black] (4,0) -- (4,1) -- (5,1) -- (5,0) -- cycle;
\draw[thick,black] (4,1) -- (4,2) -- (5,2) -- (5,1) -- cycle;
\end{tikzpicture}
\end{center}

Synthesised using general procedure D to give 8.8 mg of 22 (20%) and general procedure G to give 44.0 mg of 22 (50%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. \textbf{Mp} 107-109 °C \(^1\text{H NMR (500 MHz, CDCl}_3\} \delta (\text{ppm}):\) 3.33 (1H, dd, J = 18.1 Hz, 3.6 Hz, CH\text{CH}_A\text{H}_B), 4.23 (1H, dd, J = 18.1 Hz, 10.2 Hz, CH\text{CH}_A\text{H}_B), 5.26 (1H, dd, J = 10.2 Hz, 3.6 Hz, CH\text{CH}_A\text{H}_B), 7.24 – 7.30 (3H, m, Ar\text{H}), 7.34 (2H, d, J = 1.8 Hz,
Ar$H$), 7.47 (2H, dd, $J = 8.4$ Hz, 7.1 Hz, Ar$H$), 7.55 – 7.63 (1H, m, Ar$H$), 7.71 – 7.82 (4H, m, Ar$H$) 7.96 – 8.03 (2H, m, Ar$H$).

$^{13}$C$\{^1H\}$ NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 43.9 (CH$C$H$_2$), 48.7 (CHCH$_2$), 101.0 (ArC), 127.6 (ArC), 128.2 (ArC), 128.6 (ArC), 129.3 (ArC), 130.3 (ArC), 133.4 (ArC), 135.7 (ArC), 136.3 (ArC), 137.9 (ArC), 197.8 (C=O), 198.0 (C=O).

HRMS (ESI) C$_{22}$H$_{17}$IO$_2$ [M+H]$^+$ found XX, requires XX (+XX ppm)

1-(4-Methoxyphenyl)-2,4-diphenylbutane-1,4-dione (23):

![1-Methoxyphenyl-2,4-diphenylbutane-1,4-dione](image)

Synthesised using general procedure D to give 19.6 mg of 23 (57%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 3.30 (1H, dd, $J = 18.0$ Hz, 3.8 Hz, CH$C$H$_2$A$H$B$), 3.84 (3H, s, OCH$_3$), 4.23 (1H, dd, $J = 18.0$ Hz, 10.0 Hz, CHCH$_2$A$H$B$), 5.32 (1H, dd, $J = 10.0$ Hz, 3.8 Hz, CHCH$_2$A$H$B$), 6.87 – 6.93 (2H, m, Ar$H$), 7.21 – 7.28 (1H, m, Ar$H$), 7.33 (2H, ddd, $J = 7.8$ Hz, 6.7 Hz, 1.2 Hz, Ar$H$), 7.37 – 7.42 (2H, m, Ar$H$), 7.44 – 7.50 (2H, m, Ar$H$) 7.54 – 7.62 (1H, m, Ar$H$), 7.99 – 8.03 (2H, m, Ar$H$), 8.03 – 8.08 (2H, m, Ar$H$).

$^{13}$C$\{^1H\}$ NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 43.8 (CH$C$H$_2$), 48.4 (CHCH$_2$), 55.4 (OCH$_3$), 113.7 (ArC), 127.3 (ArC), 128.2 (ArC), 128.6 (ArC), 129.2 (ArC), 129.4 (ArC), 131.3 (ArC), 133.2 (ArC), 136.6 (ArC), 139.2 (ArC), 163.4 (ArC), 197.3 (C=O), 198.2 (C=O).

Data matches that previously reported.$^{17}$

1-(4-(Tert-butyl)phenyl)-2,4-diphenylbutane-1,4-dione (24):

![1-Tert-butylphenyl-2,4-diphenylbutane-1,4-dione](image)

Synthesised using general procedure D to give 13.3 mg of 24 (36%) after column chromatography using EtOAc:Hexane (3:97).
Colourless solid. **Mp** 116-118 °C. **$^1$H NMR (500 MHz, CDCl$_3$) δ (ppm):** 1.30 (9H, s, C(CH$_3$)$_3$) 3.30 (1H, dd, J = 18.0 Hz, 3.7 Hz, CHCH$_A^A$H$_B^B$), 4.22 (1H, dd, J = 18.0 Hz, 10.1 Hz, CHCH$_A^A$H$_B^B$), 5.33 (1H, dd, J = 10.1 Hz, 3.7 Hz, CHCH$_A^A$H$_B^B$), 7.19 – 7.26 (1H, m, ArH), 7.32 (2H, ddd, J = 7.8 Hz, 6.8 Hz, 1.2 Hz, ArH), 7.36 – 7.47 (6H, m, ArH), 7.52 – 7.58 (1H, m, ArH), 7.95 – 8.01 (4H, m, ArH).

**$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) δ (ppm):** 31.1 (CH$_3$), 35.1 (C(CH$_3$)$_3$), 44.0 (CHCH$_2$), 48.5 (CHCH$_2$), 125.5 (ArC), 127.3 (ArC), 128.2 (ArC), 128.3 (ArC), 128.6 (ArC), 128.9 (ArC), 129.2 (ArC), 133.3 (ArC), 133.8 (ArC), 136.5 (ArC), 138.9 (ArC), 156.6 (ArC), 198.2 (C=O), 198.5 (C=O).

Data matches that previously reported.

**1,2-Diphenyl-4-(p-tolyl)butane-1,4-dione (25):**

![1,2-Diphenyl-4-(p-tolyl)butane-1,4-dione](image)

Synthesised using general procedure E to give 9.8 mg of **25** (30%) and general procedure G to give 32.8 mg of **25** (50%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. **$^1$H NMR (500 MHz, CDCl$_3$) δ (ppm):** 2.40 (3H, s, CH$_3$) 3.29 (1H, dd, J = 18.0 Hz, 3.6 Hz, CHCH$_A^A$H$_B^B$), 4.19 (1H, dd, J = 18.0 Hz, 10.0 Hz, CHCH$_A^A$H$_B^B$), 5.32 (1H, dd, J = 10.1 Hz, 3.7 Hz, CHCH$_A^A$H$_B^B$), 7.21 – 7.26 (3H, m, ArH), 7.28 – 7.34 (2H, m, ArH), 7.34 – 7.43 (4H, m, ArH), 7.46 – 7.52 (1H, m, ArH), 7.86 – 7.90 (2H, m, ArH), 8.02 – 8.05 (2H, m, ArH).

**$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) δ (ppm):** 21.7 (CH$_3$), 43.8 (CHCH$_2$), 48.7 (CHCH$_2$), 127.3 (ArC), 128.27 (ArC), 128.31 (ArC), 128.5 (ArC), 129.0 (ArC), 129.2 (ArC), 129.3 (ArC), 132.9 (ArC), 134.0 (ArC), 136.5 (ArC), 138.7 (ArC), 144.1 (ArC), 198.2 (C=O), 198.5 (C=O).

Data matches that previously reported.

**4-(4-Bromophenyl)-1,2-diphenylbutane-1,4-dione (26):**
Synthesised using general procedure E to give 10.6 mg of 26 (27%) and general procedure G to give 42.5 mg of 26 (54%) after column chromatography using EtOAc:Hexane (3:97). Colourless oil. \( ^1H\) NMR (500 MHz, CDCl\(_3\)) \( \delta \) (ppm): 3.24 (1H, dd, \( J = 18.0 \) Hz, 3.7 Hz, CHCH\(_A^H\)\(_B^H\)), 4.16 (1H, dd, \( J = 18.0 \) Hz, 10.0 Hz, CHCH\(_A^H\)\(_B^H\)), 5.31 (1H, dd, \( J = 10.0 \) Hz, 3.7 Hz, CHCH\(_A^H\)\(_B^H\)), 7.21 – 7.26 (1H, m, Ar\(H\)), 7.28 – 7.37 (4H, m, Ar\(H\)), 7.38 – 7.42 (2H, m, Ar\(H\)), 7.46 – 7.51 (1H, m, Ar\(H\)), 7.57 – 7.61 (2H, m, Ar\(H\)), 7.82 – 7.86 (2H, m, Ar\(H\)).
\( ^{13}C\)\{\(^1H\}\} NMR (126 MHz, CDCl\(_3\)) \( \delta \) (ppm): 43.8 (CH\(_C\)H\(_2\)), 48.8 (CHCH\(_A^H\)\(_B^H\)), 127.5 (Ar\(C\)), 128.2 (Ar\(C\)), 128.6 (Ar\(C\)), 129.0 (Ar\(C\)), 129.3 (Ar\(C\)), 129.7 (Ar\(C\)), 131.9 (Ar\(C\)), 133.0 (Ar\(C\)), 135.2 (Ar\(C\)), 136.3 (Ar\(C\)), 138.5 (Ar\(C\)), 197.2 (C=O), 198.8 (C=O).
Data matches that previously reported.\(^{18}\)

4-(4-Methoxyphenyl)-1,2-diphenylbutane-1,4-dione (27):

Synthesised using general procedure E to give 7.6 mg of 27 (22%) and general procedure G to give 30.3 mg of 27 (44%) after column chromatography using EtOAc:Hexane (3:97). Colourless foam. \( ^1H\) NMR (500 MHz, CDCl\(_3\)) \( \delta \) (ppm): 3.27 (1H, dd, \( J = 17.8 \) Hz, 3.7 Hz, CHCH\(_A^H\)\(_B^H\)), 3.86 (3H, s, OCH\(_3\)), 4.17 (1H, dd, \( J = 17.8 \) Hz, 10.1 Hz, CHCH\(_A^H\)\(_B^H\)), 5.32 (1H, dd, \( J = 10.1 \) Hz, 3.7 Hz, CHCH\(_A^H\)\(_B^H\)), 6.88 – 6.94 (2H, m, Ar\(H\)), 7.20 – 7.25 (1H, m, Ar\(H\)), 7.28 – 7.34 (2H, m, Ar\(H\)), 7.34 – 7.43 (4H, m, Ar\(H\)), 7.46 – 7.51 (1H, m, Ar\(H\)), 7.94 – 7.98 (2H, m, Ar\(H\)), 8.02 – 8.05 (2H, m, Ar\(H\)).
\( ^{13}C\)\{\(^1H\}\} NMR (126 MHz, CDCl\(_3\)) \( \delta \) (ppm): 43.6 (CH\(_C\)H\(_2\)), 48.7 (CHCH\(_2\)), 55.5 (OCH\(_3\)), 113.7 (Ar\(C\)), 127.3 (Ar\(C\)), 128.3 (Ar\(C\)), 128.5 (Ar\(C\)), 129.0 (Ar\(C\)), 129.2 (Ar\(C\)), 129.6 (Ar\(C\)), 130.5 (Ar\(C\)), 132.9 (Ar\(C\)), 136.5 (Ar\(C\)), 138.8 (Ar\(C\)), 163.6 (Ar\(C\)), 196.6 (C=O), 199.1 (C=O).
Data matches that previously reported.\(^{19}\)
4-(4-Fluorophenyl)-1,2-diphenylbutane-1,4-dione (28):

![Structure of 4-(4-Fluorophenyl)-1,2-diphenylbutane-1,4-dione (28)]

Synthesised using general procedure E to give 7.3 mg of 28 (22%) and general procedure G to give 47.2 mg of 28 (71%) after column chromatography using EtOAc:Hexane (3:97). Colourless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 3.26 (1H, dd, $J = 17.9$ Hz, 3.7 Hz, CHCH$^A$H$^B$), 4.18 (1H, dd, $J = 17.9$ Hz, 10.1 Hz, CHCH$^A$H$^B$), 5.31 (1H, dd, $J = 10.1$ Hz, 3.7 Hz, CHCH$^A$H$^B$), 7.12 (2H, t, $J = 8.6$ Hz, ArH), 7.21 – 7.25 (1H, m, ArH), 7.29 – 7.37 (4H, m, ArH), 7.38 – 7.42 (2H, m, ArH), 7.47 – 7.52 (1H, m, ArH), 7.98 – 8.05 (4H, m, ArH).

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ (ppm): -104.9.

$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 43.8 (CH$_2$), 48.8 (CH$_2$), 115.6 (d, $J = 21.9$ Hz, ArC) 127.5 (ArC), 128.2 (ArC), 128.6 (ArC), 129.0 (ArC), 129.3 (ArC), 130.8 (d, $J = 9.4$ Hz, ArC), 133.0 (ArC), 136.3 (ArC), 138.5 (ArC), 165.9 (d, $J = 254.2$ Hz, ArC), 198.2 (C=O), 198.5 (C=O).

Data matches that previously reported.$^{19}$

1,2-Diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione (29):

![Structure of 1,2-Diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione (29)]

Synthesised using general procedure E to give 6.5 mg of 29 (17%) and general procedure G to give 22.2 mg of 29 (29%) after column chromatography using EtOAc:Hexane (3:97). Yellow oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 3.31 (1H, dd, $J = 18.0$ Hz, 3.7 Hz, CHCH$^A$H$^B$), 4.25 (1H, dd, $J = 18.0$ Hz, 10.1 Hz, CHCH$^A$H$^B$), 5.35 (1H, dd, $J = 10.1$ Hz, 3.6 Hz, CHCH$^A$H$^B$), 7.24 – 7.28 (1H, m, ArH), 7.33 – 7.40 (4H, m, ArH), 7.43 (2H, t, $J = 7.7$ Hz, ArH), 7.50 – 7.55 (1H, m, ArH), 7.73 – 7.78 (2H, m, ArH), 8.03 – 8.07 (2H, m, ArH), 8.10 – 8.14 (2H, m, ArH).
$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ (ppm): -63.1

$^{13}$C($^1$H) NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 44.1 (CHCH$_2$), 48.8 (CHCH$_2$), 123.5 (q, $J = 272.4$ Hz, CF$_3$), 125.7 (q, $J = 3.9$ Hz, ArC), 127.6 (ArC), 128.2 (ArC), 128.5 (ArC), 128.6 (ArC), 129.0 (ArC), 129.3 (ArC), 133.1 (ArC), 134.6 (q, $J = 32.6$ Hz, ArC), 136.2 (ArC), 138.3 (ArC), 139.1.6 (ArC), 197.3 (C=O), 198.7 (C=O).

Data matches that previously reported.$^{14}$

1,2-Diphenylpentane-1,4-dione (30):

\[
\text{Ph} \quad \text{O} \quad \text{Me} \quad \text{Ph}
\]

Synthesised using general procedure E to give 11.4 mg of 30 (45%) after column chromatography using EtOAc:Hexane (10:90).

Yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 2.19 (3H, s, CH$_3$), 2.76 (1H, dd, $J = 18.0$ Hz, 4.0 Hz, CHCH$_A$H$_B$), 3.61 (1H, dd, $J = 18.0$ Hz, 10.1 Hz, CHCH$_A$H$_B$), 5.11 (1H, dd, $J = 10.1$ Hz, 4.0 Hz, CHCH$_A$H$_B$), 7.20 (1H, ddd, $J = 8.1$ Hz, 4.6 Hz, 2.4 Hz, ArH), 7.26 – 7.31 (4H, m, ArH), 7.37 (2H, dd, $J = 8.3$ Hz, 6.9 Hz, ArH), 7.44 – 7.50 (1H, m, ArH), 7.94 – 7.98 (2H, m, ArH).

$^{13}$C($^1$H) NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 30.0 (CH$_3$), 48.1 (CHCH$_2$), 48.8 (CHCH$_2$), 127.3 (ArC), 128.1 (ArC), 128.4 (ArC), 128.9 (ArC), 129.2 (ArC), 132.9 (ArC), 138.6 (ArC), 198.9 (C=O), 206.7 (C=O).

Data matches that previously reported.$^{20}$

4-Cyclopentyl-1-phenyl-2-(pyridin-2-yl)butane-1,4-dione (30):

\[
\text{Ph} \quad \text{O} \quad \text{Me} \quad \text{Ph}
\]

Synthesised using general procedure E to give 12.3 mg of 31 (40%) after column chromatography using EtOAc:Hexane (20:80).
Yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 1.51 – 1.89 (8H, m, CH$_2$CH$_2$CH$_2$CH$_2$), 2.87 – 3.01 (2H, m, CH$_2$CHCH$_2$ + C(3)H$^A$H$^B$), 3.66 (1H, dd, $J = 17.9$ Hz, 9.9 Hz, C(3)H$^A$H$^B$), 5.37 (1H, dd, $J = 9.8$ Hz, 4.2 Hz, C(2)H$^H$), 7.11 (1H, ddd, $J = 7.6$ Hz, 4.9 Hz, 1.2 Hz, ArH), 7.23 – 7.26 (1H, m, ArH), 7.35 – 7.41 (2H, m, ArH), 7.45 – 7.50 (1H, m, ArH), 7.58 (1H, td, $J = 7.7$ Hz, 1.8 Hz, ArH), 8.00 – 8.05 (2H, m, ArH).

$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) δ (ppm): 26.0 (CH$_2$CH$_2$CH$_2$CH$_2$), 26.1 (CH$_2$CH$_2$CH$_2$CH$_2$), 28.8 (CH$_2$CH$_2$CH$_2$CH$_2$), 44.9 (C(3)), 51.1 (C(2)), 51.2 (CH$_2$CHCH$_2$), 122.1 (ArC), 122.9 (ArC), 128.5 (ArC), 129.0 (ArC), 132.0 (ArC), 136.4 (ArC), 137.1 (ArC), 150.0 (ArC), 158.5 (ArC), 198.9 (C=O), 206.7 (C=O).

HRMS (ESI) C$_{20}$H$_{21}$O$_2$ [M+H]$^+$ found XX, requires XX (+XX ppm)

Infra-Red (ν max, cm$^{-1}$): 2955.0 (C-H), 2868.2 (C-H), 1707.0 (C=O), 1683.9 (C=O).

[1,1'-Biphenyl]-4-carbonyl fluoride (32):

 Colourless solid. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.41 – 7.54 (3H, m, ArH), 7.62 – 7.66 (2H, m, ArH), 7.72 – 7.77 (2H, m, ArH), 8.09 – 8.15 (2H, m, ArH).

$^{19}$F NMR (376 MHz, CDCl$_3$) δ (ppm): 18.1.

Data matches that previously reported.$^{21}$

2-Naphthoyl fluoride (33):

 Colourless solid. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.62 (1H, ddd, $J = 8.1$ Hz, 6.9 Hz, 1.3 Hz, ArH), 7.69 (1H, ddd, $J = 8.2$ Hz, 6.9 Hz, 1.3 Hz, ArH), 7.90 – 8.03 (4H, m, ArH), 8.63 – 8.65 (1H, m, ArH).

$^{19}$F NMR (376 MHz, CDCl$_3$) δ (ppm): 18.1.
Data matches that previously reported.\textsuperscript{21}

1-Naphthoyl fluoride (34):

\begin{center}
\includegraphics[width=1.0\textwidth]{1-naphthoyl_fluoride.png}
\end{center}

Colourless solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 7.58 (1H, dd, \(J = 8.2\) Hz, 7.4 Hz, ArH), 7.69 (1H, ddd, \(J = 8.2\) Hz, 6.9 Hz, 1.3 Hz, ArH), 7.90 – 8.03 (4H, m, ArH), 8.63 – 8.65 (1H, m, ArH).
\textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 29.9.

Data matches that previously reported.\textsuperscript{21}

4-Chlorobenzoyl fluoride (35):

\begin{center}
\includegraphics[width=1.0\textwidth]{4-chlorobenzoyl_fluoride.png}
\end{center}

Colourless solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 7.50 – 7.59 (2H, m, C(3)H), 7.97 – 8.06 (2H, m, C(2)H).
\textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 29.9.

Data matches that previously reported.\textsuperscript{21}

4-Bromobenzoyl fluoride (36):

\begin{center}
\includegraphics[width=1.0\textwidth]{4-bromobenzoyl_fluoride.png}
\end{center}

Colourless solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 7.61 – 7.81 (2H, m, C(3)H), 7.81 – 8.00 (2H, m, C(2)H).
\textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 18.4.
Data matches that previously reported.\textsuperscript{22}

4-Iodobenzoyl fluoride (37):

\[
\begin{array}{c}
\text{I} \\
\text{O} \\
\text{F} \\
\text{C} \\
\end{array}
\]

Colourless solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 7.70 – 7.78 (2H, m, C(3)\textsubscript{H}), 7.87 – 7.95 (2H, m, C(2)\textsubscript{H}).

\textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 18.4.

Data matches that previously reported.\textsuperscript{21}

4-Methoxybenzoyl fluoride (38):

\[
\begin{array}{c}
\text{MeO} \\
\text{O} \\
\text{F} \\
\text{C} \\
\end{array}
\]

Colourless oil. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 3.92 (3H, s, OCH\textsubscript{3}), 6.95 – 7.09 (2H, m, C(3)\textsubscript{H}), 7.99 – 8.06 (2H, m, C(2)\textsubscript{H}).

\textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 16.0.

Data matches that previously reported.\textsuperscript{21}

4-(\textit{tert}-butyl)benzoyl fluoride (39):

\[
\begin{array}{c}
\text{t-Bu} \\
\text{O} \\
\text{F} \\
\text{C} \\
\end{array}
\]

Colourless solid. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 1.35 (9H, s, C(CH\textsubscript{3})\textsubscript{3}), 7.54 (2H, dd, \(J = 8.6\) Hz, 1.4 Hz, C(3)\textsubscript{H}), 7.98 (2H, d, \(J = 8.5\) Hz, C(2)\textsubscript{H}).

\textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta\) (ppm): 17.7.
Data matches that previously reported.

**4-Cyanobenzoyl fluoride (40):**

![4-Cyanobenzoyl fluoride (40)](image)

Colourless solid. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.67 – 7.92 (2H, m, C(3)\(H\)), 8.17 (2H, d, \(J = 8.4\) Hz, C(2)\(H\)).

\(^{19}F\) NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): 20.2.

Data matches that previously reported.\(^{23}\)

**Cyclohexanecarbonyl fluoride (41):**

![Cyclohexanecarbonyl fluoride (41)](image)

Colourless oil. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 1.23 – 1.34 (3H, m, C\(H_2\)), 1.45 – 1.62 (2H, m, C\(H_2\)), 1.62 (1H, m, C\(H_2\)), 1.62 – 1.72 (1H, m, C\(H_2\)), 1.75 – 1.80 (2H, m, C\(H_2\)), 1.95 – 2.02 (2H, m, C\(H_2\)), 2.47 – 2.54 (1H, m, C\(H\)).

\(^{19}F\) NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): 36.7.

Data matches that previously reported.\(^{21}\)

**2-Furanoyl fluoride (42):**

![2-Furanoyl fluoride (42)](image)

Colourless oil. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 6.65 (1H, ddd, \(J = 3.6\) Hz, 1.8 Hz, 0.8 Hz, C(4)\(H\)), 7.45 (1H, dd, \(J = 3.7\) Hz, 0.8 Hz, C(3)\(H\)), 7.77 (1H, ddd, \(J = 2.5\) Hz, 1.7 Hz, 0.8 Hz, C(4)\(H\)).

\(^{19}F\) NMR (376 MHz, CDCl\(_3\)) \(\delta\) (ppm): 15.4.
Data matches that previously reported.\(^24\)

**2-Oxo-2-(p-tolyl)acetic acid (43):**

![2-Oxo-2-(p-tolyl)acetic acid](image)

Colourless solid. \textbf{Mp} 97-99 °C \{Lit. Mp\(^{25}\) 97-99 °C\}. \textbf{\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm):} 2.46 (3H, s, \(CH_3\)), 7.34 (2H, d, \(J = 8.1\) Hz, ArC(3)\(H\)), 8.28 (2H, d, \(J = 8.0\) Hz, ArC(2)\(H\))

\textbf{\(^{13}\)C\{\(^1\)H\} NMR (126 MHz, CDCl\(_3\)) \(\delta\) (ppm):} 22.1 (CH\(_3\)), 129.2 (ArC), 129.8 (ArC), 131.7 (ArC), 147.4 (ArC), 161.3 (C(O)OH), 183.6 (C=O).

Data matches that previously reported.\(^26\)

**2-(4-Bromophenyl)-2-oxoacetic acid (44):**

![2-(4-Bromophenyl)-2-oxoacetic acid](image)

Colourless solid. \textbf{Mp} 97-99 °C \{Lit. Mp\(^{27}\) 100-102 °C\}. \textbf{\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) (ppm):} 7.66 – 7.70 (2H, m, ArC(3)\(H\)), 8.20 – 8.24 (2H, m, ArC(2)\(H\))

\textbf{\(^{13}\)C\{\(^1\)H\} NMR (126 MHz, CDCl\(_3\)) \(\delta\) (ppm):} 130.5 (ArC), 131.6 (ArC), 132.4 (ArC), 132.7 (ArC), 161.1 (C(O)OH), 183.8 (C=O).

Data matches that previously reported.\(^26\)

**2-(4-Methoxyphenyl)-2-oxoacetic acid (45):**

![2-(4-Methoxyphenyl)-2-oxoacetic acid](image)
Colourless solid. Mp 74-76 °C {Lit. Mp²⁸ 79-81 °C}. ¹H NMR (500 MHz, CDCl₃) δ (ppm): 3.91 (3H, s, OCH₃), 6.95 – 7.00 (2H, m, ArC(3)H), 8.33 – 8.39 (2H, m, ArC(2)H)
¹³C{¹H} NMR (126 MHz, CDCl₃) δ (ppm): 55.7 (OCH₃), 114.4 (ArC), 124.9 (ArC), 162.6 (C(O)OH), 165.6 (ArC), 183.0 (C=O).
Data matches that previously reported.²⁶

2-(4-Fluorophenyl)-2-oxoacetic acid (46):

Colourless solid. Mp 95-97 °C {Lit. Mp²⁹ 95-96 °C}. ¹H NMR (500 MHz, CDCl₃) δ (ppm): 7.14 – 7.25 (2H, m, ArC(3)H), 8.41 – 8.49 (2H, m, ArC(2)H)
¹⁹F NMR (471 MHz, CDCl₃) δ (ppm): -99.2
¹³C{¹H} NMR (126 MHz, CDCl₃) δ (ppm): 116.5 (d, J = 22.3 Hz, ArC), 128.2 (ArC), 134.6 (d, J = 10.0 Hz, ArC), 163.8 (d, J = 662.7 Hz, ArC), 168.5 (C(O)OH), 182.4 (C=O).
Data matches that previously reported.²⁶

2-oxo-2-(4-(trifluoromethyl)phenyl)acetic acid (47):

Colourless solid. Mp 56-59 °C {Lit. Mp²⁷ 53-55 °C}. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.81 (2H, d, J = 8.3 Hz, ArC(3)H), 8.42 (2H, d, J = 8.2 Hz, ArC(2)H), 8.90 (1H, s, OH).
¹³C{¹H} NMR (126 MHz, CDCl₃) δ (ppm): 123.2 (q, J = 273.0 Hz, CF₃), 126.0 (q, J = 3.7 Hz), 131.5 (ArC), 134.4 (ArC), 136.5 (q, J = 33.0 Hz), 161.8 (C(O)OH), 183.6 (C=O).
Data matches that previously reported.³⁰
2-cyclopentyl-2-oxoacetic acid (48):

Yellow oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ (ppm): 1.65 – 1.75 (4H, m, CH$_2$), 1.77 – 1.88 (2H, m, CH$_2$), 1.95 – 2.05 (2H, m, CH$_2$), 3.69 (1H, tt, $J = 9.0$ Hz, 6.9 Hz).

$^{13}$C{$^1$H} NMR (126 MHz, CDCl$_3$) $\delta$ (ppm): 26.2 (CH$_2$) 28.8 (CH$_2$), 45.6 (CH), 159.9 (C(O)OH), 197.7 (C=O).

Data matches that previously reported.$^{31}$
References

NMR Spectra

Figure S11. $^1$H NMR spectrum of 2-Phenyl-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 
$5, {}^{13}\text{C}, \text{CDCl}_3, 126 \text{ MHz}$

Figure S12. $^{13}\text{C}$ NMR spectrum of 2-Phenyl-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S13. $^1$H NMR spectrum of 2-(4-bromophenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 

**7, $^1$H, CDCl$_3$, 500 MHz**
Figure S14. $^{13}$C NMR spectrum of 2-(4-bromophenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 

$7, ^{13}$C, CDCl$_3$, 126 MHz
Figure S15. $^1$H NMR spectrum of 2-(4-fluorophenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 

8. $^1$H, CDCl$_3$, 500 MHz
Figure S16. $^{19}$F NMR spectrum of 2-(4-fluorophenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 

8. $^{19}$F, CDCl$_3$, 471 MHz
Figure S17. $^{13}$C NMR spectrum of 2-(4-fluorophenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 

8. $^{13}$C, CDCl$_3$, 126 MHz
Figure S18. $^1$H NMR spectrum of 1,4-diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione in CDCl$_3$. 

9, $^1$H, CDCl$_3$, 500 MHz
9, $^{19}$F, CDCl$_3$, 471 MHz

Figure S19. $^{19}$F NMR spectrum of 1,4-diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione in CDCl$_3$. 
9, $^{13}$C, CDCl$_3$, 126 MHz

Figure S20. $^{13}$C NMR spectrum of 1,4-diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione in CDCl$_3$. 
Figure S21. $^1$H NMR spectrum of 1,4-diphenyl-2-(pyridin-2-yl)butane-1,4-dione in CDCl$_3$. 

$^{10}$, $^1$H, CDCl$_3$, 500 MHz
10, $^{13}\text{C}$, CDCl$_3$, 126 MHz

Figure S22. $^{13}\text{C}$ NMR spectrum of 1,4-diphenyl-2-(pyridin-2-yl)butane-1,4-dione in CDCl$_3$. 

S60
11, $^1$H, CDCl$_3$, 500 MHz

Figure S23. $^1$H NMR spectrum of 1,4-diphenyl-2-(p-tolyl)butane-1,4-dione in CDCl$_3$. 
11, $^{13}$C, CDCl$_3$, 126 MHz

Figure S24. $^{13}$C NMR spectrum of 1,4-diphenyl-2-(p-tolyl)butane-1,4-dione in CDCl$_3$.
12, $^1$H, CDCl$_3$, 500 MHz

Figure S25. $^1$H NMR spectrum of 2-(4-(tert-butyl)phenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 
12, $^{13}$C, CDCl$_3$, 126 MHz

Figure S26. $^{13}$C NMR spectrum of 2-(4-(tert-butyl)phenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 

S64
Figure S27. $^1$H NMR spectrum of 2-(4-(chloromethyl)phenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 

13, $^1$H, CDCl$_3$, 500 MHz
$^{13}$C, CDCl$_3$, 126 MHz

Figure S28. $^{13}$C NMR spectrum of 2-(4-(chloromethyl)phenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S29. $^1$H NMR spectrum of 1,4-diphenyl-2-(o-tolyl)butane-1,4-dione in CDCl$_3$. 
Figure S30. $^{13}$C NMR spectrum of 1,4-diphenyl-2-(o-tolyl)butane-1,4-dione in CDCl$_3$. 

14, $^{13}$C, CDCl$_3$, 126 MHz
Figure S31. $^1$H NMR spectrum of 2-(4-methoxyphenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S32. $^{13}$C NMR spectrum of 2-(4-methoxyphenyl)-1,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S33. $^1$H NMR spectrum of 1-([1,1'-biphenyl]-4-yl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

16, $^1$H, CDCl$_3$, 500 MHz
16, $^{13}$C, CDCl$_3$, 126 MHz

Figure S34. $^{13}$C NMR spectrum of 1-([1,1'-biphenyl]-4-yl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
17, $^1$H, CDCl$_3$, 500 MHz

Figure S35. $^1$H NMR spectrum of 1-(naphthalen-2-yl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S36. $^{13}$C NMR spectrum of 1-(naphthalen-2-yl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

$^{17}$, $^{13}$C, CDCl$_3$, 126 MHz
Figure S37. $^1$H NMR spectrum of 1-(naphthalen-1-yl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

18, $^1$H, CDCl$_3$, 500 MHz
18, $^{13}\text{C}$, CDCl$_3$, 126 MHz

Figure S38. $^{13}\text{C}$ NMR spectrum of 1-(naphthalen-1-yl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
19, $^1$H, CDCl₃, 500 MHz

Figure S39. $^1$H NMR spectrum of 1-(4-fluorophenyl)-2,4-diphenylbutane-1,4-dione in CDCl₃.
Figure S40. $^{19}$F NMR spectrum of 1-(4-fluorophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
**19, $^{13}$C, CDCl$_3$, 126 MHz**

Figure S41. $^{13}$C NMR spectrum of 1-(4-fluorophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

S79
Figure S42. $^1$H NMR spectrum of 1-(4-chlorophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

20, $^1$H, CDCl$_3$, 500 MHz
20, $^{13}$C, CDCl$_3$, 126 MHz

Figure S43. $^{13}$C NMR spectrum of 1-(4-chlorophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
21, $^1$H, CDCl$_3$, 500 MHz

Figure S44. $^1$H NMR spectrum of 1-(4-bromophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S45. $^{13}$C NMR spectrum of 1-(4-bromophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$.
Figure S46. $^1$H NMR spectrum of 1-(4-iodophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

22, $^1$H, CDCl$_3$, 500 MHz
22, $^{13}$C, CDCl$_3$, 126 MHz

Figure S47. $^{13}$C NMR spectrum of 1-(4-iodophenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S48. $^1$H NMR spectrum of 1-(4-methoxyphenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
23, $^{13}$C, CDCl$_3$, 126 MHz

Figure S49. $^{13}$C NMR spectrum of 1-(4-methoxyphenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

S87
Figure S50. $^1$H NMR spectrum of 1-(4-(tert-butyl)phenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 

$24$, $^1$H, CDCl$_3$, 500 MHz
$^{24}$, $^{13}$C, CDCl$_3$, 126 MHz

Figure S51. $^{13}$C NMR spectrum of 1-(4-(tert-butyl)phenyl)-2,4-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S52. $^1$H NMR spectrum of 1,2-diphenyl-4-(p-tolyl)butane-1,4-dione in CDCl$_3$. 

25, $^1$H, CDCl$_3$, 500 MHz
25, $^{13}$C, CDCl$_3$, 126 MHz

Figure S53. $^{13}$C NMR spectrum of 1,2-diphenyl-4-(p-toly1)butane-1,4-dione in CDCl$_3$. 
Figure S54. $^1$H NMR spectrum of 4-(4-bromophenyl)-1,2-diphenylbutane-1,4-dione in CDCl$_3$. 

26, $^1$H, CDCl$_3$, 500 MHz
Figure S55. $^{13}$C NMR spectrum of 4-(4-bromophenyl)-1,2-diphenylbutane-1,4-dione in CDCl$_3$. 

$^{26,^{13}}$C, CDCl$_3$, 126 MHz
27, $^1$H, CDCl$_3$, 500 MHz

Figure S56. $^1$H NMR spectrum of 4-(4-methoxyphenyl)-1,2-diphenylbutane-1,4-dione in CDCl$_3$. 
Figure S57. $^{13}$C NMR spectrum of 4-(4-methoxyphenyl)-1,2-diphenylbutane-1,4-dione in CDCl$_3$. 
**Figure S58.** $^1$H NMR spectrum of 4-(4-fluorophenyl)-1,2-diphenylbutane-1,4-dione in CDCl$_3$. 

28, $^1$H, CDCl$_3$, 500 MHz
28, $^{19}$F, CDCl$_3$, 471 MHz

Figure S59. $^{19}$F NMR spectrum of 4-(4-fluorophenyl)-1,2-diphenylbutane-1,4-dione in CDCl$_3$. 

S97
Figure S60. $^{13}$C NMR spectrum of 4-(4-fluorophenyl)-1,2-diphenylbutane-1,4-dione in CDCl$_3$. 

28, $^{13}$C, CDCl$_3$, 126 MHz
Figure S61. $^1$H NMR spectrum of 1,2-diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione in CDCl$_3$. 

$^{29, ~^1}$H, CDCl$_3$, 500 MHz
29, $^{19}\text{F}$, CDCl$_3$, 471 MHz

Figure S62. $^{19}\text{F}$ NMR spectrum of 1,2-diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione in CDCl$_3$. 
29, $^{13}$C, CDCl$_3$, 126 MHz

Figure S63. $^{13}$C NMR spectrum of 1,2-diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione in CDCl$_3$. 
Figure S64. $^1$H NMR spectrum of 1,2-diphenylpentane-1,4-dione in CDCl$_3$. 

30, $^1$H, CDCl$_3$, 500 MHz
**30, $^{13}$C, CDCl$_3$, 126 MHz**

Figure S65. $^{13}$C NMR spectrum of 1,2-diphenylpentane-1,4-dione in CDCl$_3$. 
Figure S66. $^1$H NMR spectrum of 4-cyclopentyl-1-phenyl-2-(pyridin-2-yl)butane-1,4-dione in CDCl$_3$. 

31, $^1$H, CDCl$_3$, 500 MHz
Figure S67. $^{13}$C NMR spectrum of 4-cyclopentyl-1-phenyl-2-(pyridin-2-yl)butane-1,4-dione in CDCl$_3$. 
32, $^1$H, CDCl$_3$, 400 MHz

Figure S68. $^1$H NMR spectrum of [1,1'-biphenyl]-4-carbonyl fluoride in CDCl$_3$. 
Figure S69. $^{19}$F NMR spectrum of [1,1'-biphenyl]-4-carbonyl fluoride in CDCl$_3$. 

32, $^{19}$F, CDCl$_3$, 376 MHz
Figure S70. $^1$H NMR spectrum of 2-naphthoyl fluoride in CDCl$_3$. 
Figure S71. $^{19}$F NMR spectrum of 2-naphthoyl fluoride in CDCl$_3$.
Figure S72. $^1$H NMR spectrum of 1-naphthoyl fluoride in CDCl$_3$. 
34, $^{19}$F, CDCl$_3$, 376 MHz

Figure S73. $^{19}$F NMR spectrum of 1-naphthoyl fluoride in CDCl$_3$. 
Figure S74. $^1$H NMR spectrum of 4-chlorobenzoyl fluoride in CDCl$_3$. 

35, $^1$H, CDCl$_3$, 400 MHz
Figure S75. $^{19}$F NMR spectrum of 4-chlorobenzoyl fluoride in CDCl$_3$. 

35, $^{19}$F, CDCl$_3$, 376 MHz
36, $^1$H, CDCl$_3$, 400 MHz

Figure S76. $^1$H NMR spectrum of 4-bromobenzoyl fluoride in CDCl$_3$. 
36, $^{19}$F, CDCl$_3$, 376 MHz

Figure S77. $^{19}$F NMR spectrum of 4-bromobenzoyl fluoride in CDCl$_3$. 
Figure S78. $^1$H NMR spectrum of 4-iodobenzoyl fluoride in CDCl$_3$. 

$^{37}, ^1$H, CDCl$_3$, 400 MHz
Figure S79. $^{19}$F NMR spectrum of 4-iodobenzoyl fluoride in CDCl$_3$. 

$^{37}$, $^{19}$F, CDCl$_3$, 376 MHz
Figure S80. $^1$H NMR spectrum of 4-methoxybenzoyl fluoride in CDCl$_3$. 
Figure S81. $^{19}$F NMR spectrum of 4-methoxybenzoyl fluoride in CDCl$_3$. 
*Figure S82.* $^1$H NMR spectrum of 4-(tert-butyl)benzoyl fluoride in CDCl$_3$. 

$39, ^1$H, CDCl$_3$, 400 MHz
Figure S83. $^{19}$F NMR spectrum of 4-(tert-butyl)benzoyl fluoride in CDCl$_3$. 

$^{39}$, $^{19}$F, CDCl$_3$, 376 MHz
Figure S84. $^1$H NMR spectrum of 4-cyanobenzoyl fluoride in CDCl$_3$. 

$\textbf{40}, ^1\text{H, CDCl}_3, 400 \text{ MHz}$
40, $^{19}\text{F}$, CDCl$_3$, 376 MHz

Figure S85. $^{19}\text{F}$ NMR spectrum of 4-cyanobenzoyl fluoride in CDCl$_3$. 

S123
41, $^1$H, CDCl$_3$, 400 MHz

Figure S86. $^1$H NMR spectrum of cyclohexanecarbonyl fluoride in CDCl$_3$. 
**41, $^{19}\text{F}$, CDCl$_3$, 376 MHz**

Figure S87. $^{19}\text{F}$ NMR spectrum of cyclohexanecarbonyl fluoride in CDCl$_3$. 
42, $^1$H, CDCl$_3$, 400 MHz

Figure S88. $^1$H NMR spectrum of 2-furanoyl fluoride in CDCl$_3$. 
Figure S89. $^{19}$F NMR spectrum of 2-furanoyl fluoride in CDCl$_3$. 

42, $^{19}$F, CDCl$_3$, 376 MHz
Figure S90. $^1$H NMR spectrum of 2-Oxo-2-(p-tolyl)acetic acid in CDCl$_3$. 

43, $^1$H, CDCl$_3$, 500 MHz
Figure S91. $^{13}$C NMR spectrum of 2-Oxo-2-(p-tolyl)acetic acid in CDCl$_3$. 
Figure S92. \(^1\)H NMR spectrum of 2-(4-bromophenyl)-2-oxoacetic acid in CDCl\(_3\).
**Figure S93.** $^{13}$C NMR spectrum of 2-(4-bromophenyl)-2-oxoacetic acid in CDCl$_3$. 

$^{13}$C, CDCl$_3$, 126 MHz
Figure S94. $^1$H NMR spectrum of 2-(4-methoxyphenyl)-2-oxoacetic acid in CDCl$_3$. 

45, $^1$H, CDCl$_3$, 500 MHz
45, $^{13}$C, CDCl$_3$, 126 MHz

Figure S95. $^{13}$C NMR spectrum of 2-(4-methoxyphenyl)-2-oxoacetic acid in CDCl$_3$. 
46, $^1\text{H}$, CDCl$_3$, 500 MHz

Figure S96. $^1\text{H}$ NMR spectrum of 2-(4-fluorophenyl)-2-oxoacetic acid in CDCl$_3$. 
46, $^{19}$F, CDCl$_3$, 471 MHz

Figure S97. $^{19}$F NMR spectrum of 2-(4-fluorophenyl)-2-oxoacetic acid in CDCl$_3$. 
Figure S98. $^{13}$C NMR spectrum of 2-(4-fluorophenyl)-2-oxoacetic acid in CDCl$_3$. 

46, $^{13}$C, CDCl$_3$, 126 MHz
Figure S99. $^1$H NMR spectrum of 2-(4-(trifluoromethyl)phenyl)-2-oxoacetic acid in CDCl$_3$. 

$^{47}, {^1}$H, CDCl$_3$, 500 MHz
47. $^{13}$C, CDCl$_3$, 126 MHz

Figure S100. $^{13}$C NMR spectrum of 2-(4-(trifluoromethyl)phenyl)-2-oxoacetic acid in CDCl$_3$. 
Figure S101. $^1$H NMR spectrum of 2-cyclopentyl-2-oxoacetic acid in CDCl$_3$. 

48, $^1$H, CDCl$_3$, 500 MHz
48, $^{13}$C, CDCl$_3$, 126 MHz

Figure S102. $^{13}$C NMR spectrum of 2-cyclopentyl-2-oxoacetic acid in CDCl$_3$.