Electronic Supplementary Material (ESI) for ChemComm.

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] α-keto acid **43-48**,[2] all other reagents and solvents were obtained from commercial sources and used as received. Photocatalysts $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$,[3] $[Ir(ppy)_2(dtbbpy)](PF_6)$,[4] 4CzIPN,[5] and DiKTa[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 (δ = 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.

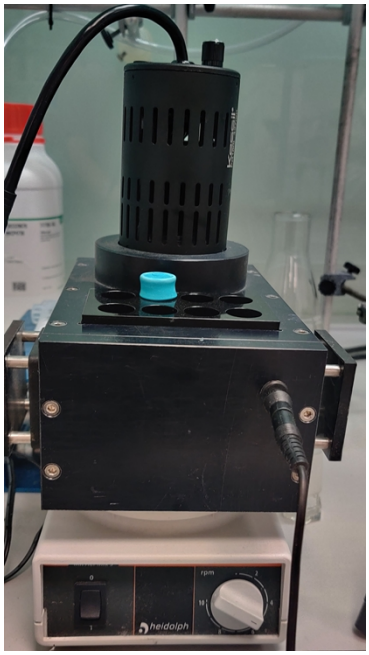


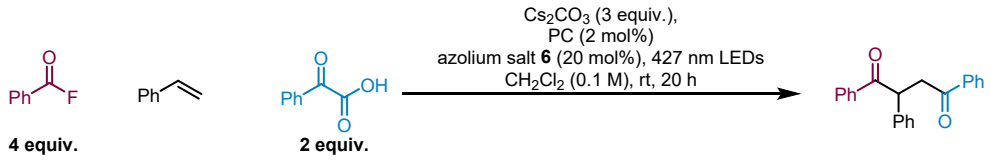
Figure **S1**. Experimental set-up for photocatalysis reactions.

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 ^{1}H NMR spectroscopy with 1,3,5-trimethoxybenzene as an internal standard.

Variation of photocatalyst



PC	***NMR Yield / %***
$[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$	30
$[Ir(ppy)_2(dtbbpy)](PF_6)$	30
4CzIPN	29
DiKTa	**38**
Eosin Y (λ_{exc} = 525 nm)	-

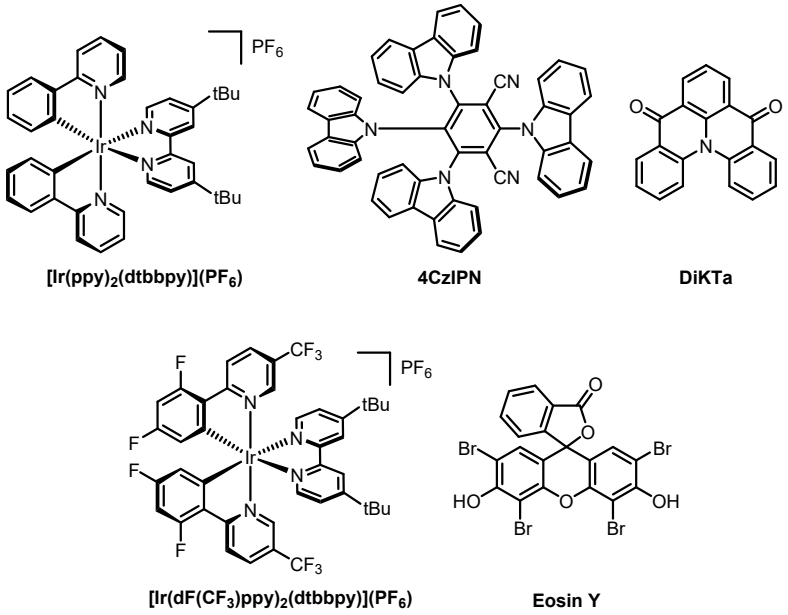
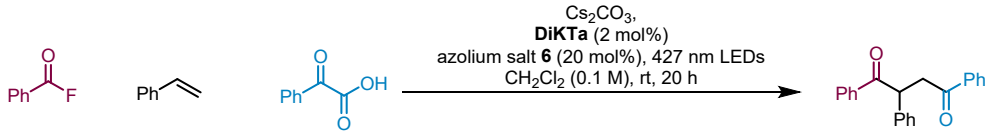


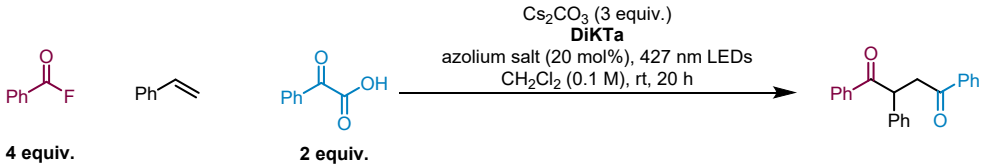
Figure S**2**. Photocatalysts used in optimization.

Variation of reagent ratios



Benzoyl Fluoride equiv.	***Styrene equiv.***	***Acid equiv.***	***NMR yield / %***
4	1	2	38
4	**2**	**1**	**46**
4	4	1	30
1	2	4	10
1	4	2	< 5

Variation of NHC catalyst



Azolium Salt	***NMR yield / %***
6	46
4	**50**
49	Trace
50	27
51	7
52	Trace
53	8
54	17
55	Trace
56	Trace

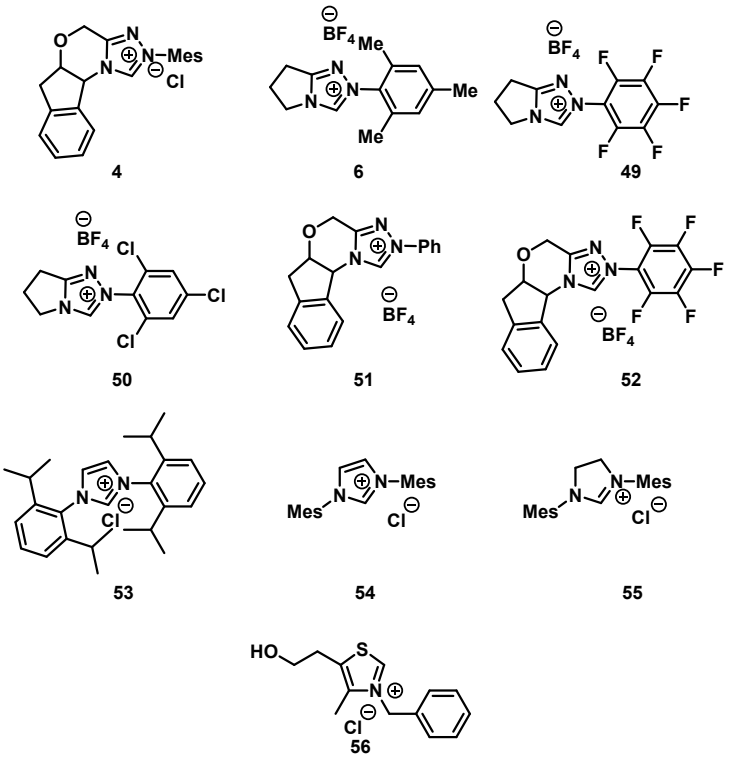
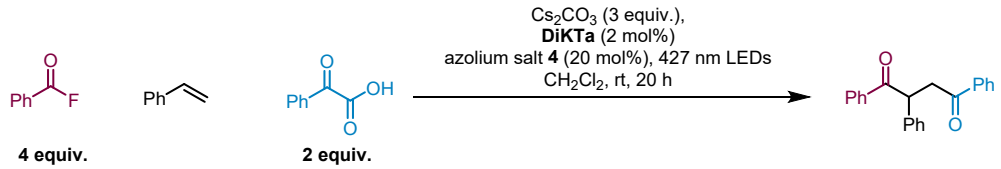


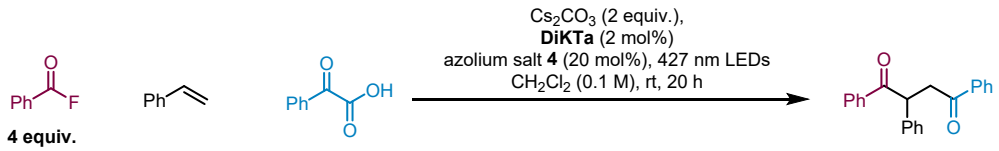
Figure S**3**. Azolium salts used in optimization.

Variation of Concentration



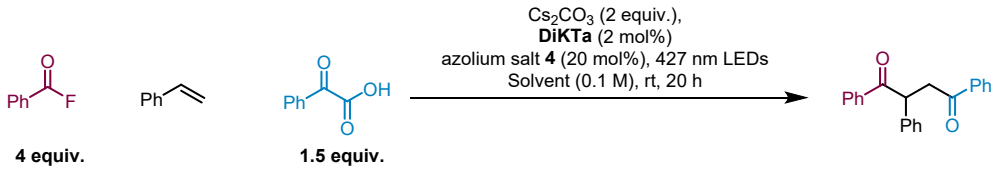
Concentration / M	***NMR Yield / %***
0.1	**50**
0.2	35
0.05	30
0.025	33

Variation of Reagent Ratios



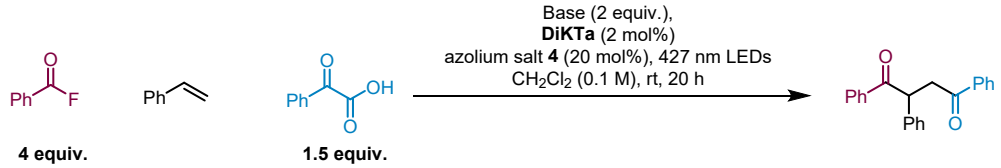
Styrene equiv.	***Acid equiv.***	***Base equiv.***	***NMR yield / %***
2	1	1.5	49
1	1	1.5	33
1.5	1	1.5	48
1	**1.5**	**2**	**61**

Variation of Solvent



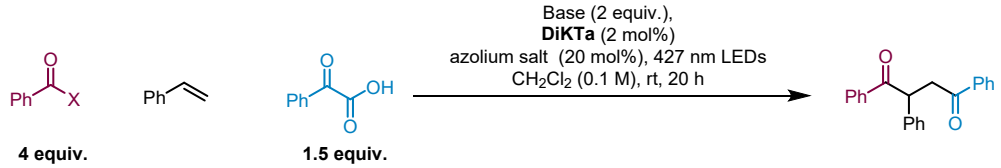
Solvent	***NMR Yield / %***
DCM	**61**
MeCN	48
DMF	51
Toluene	54
THF	33

Variation of Base



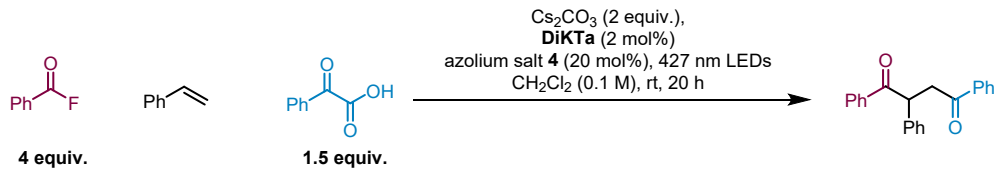
Base	***NMR Yield / %***
Cs_2CO_3	**61**
K_2CO_3	30
Na_2CO_3	16
$NaHCO_3$	8
t-BuOK	22

Variation of Acyl Leaving Group



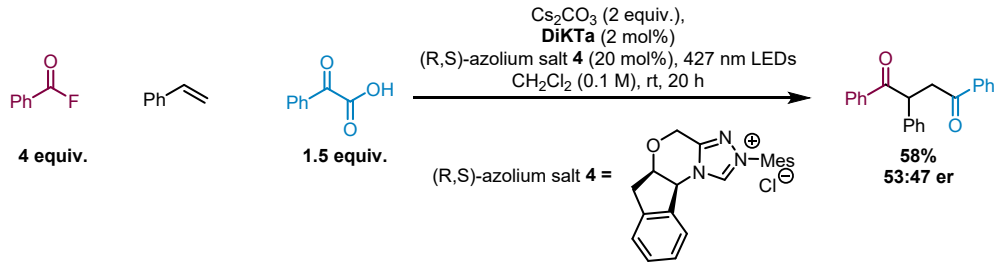
X	***Azolium Salt***	***NMR Yield / %***
F	**4**	**61**
OOCPh (anhydride)	4	26
Imidazole	4	0
Cl	4	Trace
F	6	42
OOCPh (anhydride)	6	46
Imidazole	6	39

Control Reactions



Variation	***NMR Yield / %***
No Change	61
λ_{exc} = 456 nm	48
No PC	5
No NHC	0
No benzoyl fluoride	Trace
No glyoxylic acid	0
No Light	0
No Cs_2CO_3	Trace

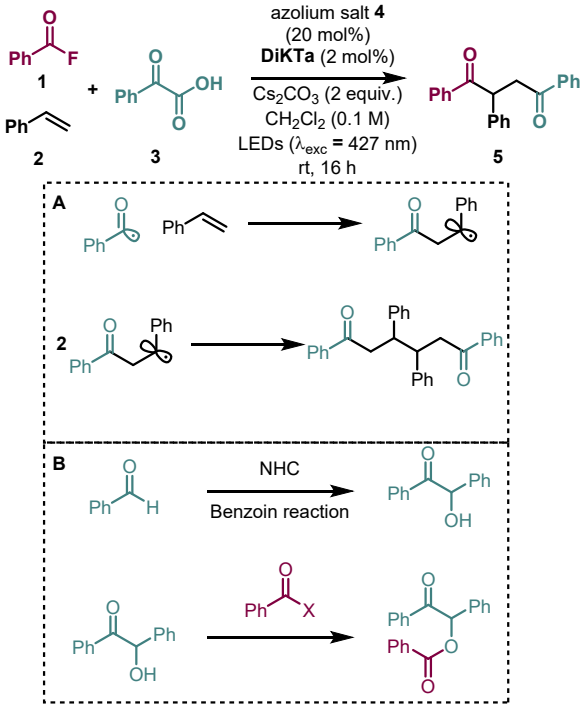
Enantioselective trial



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

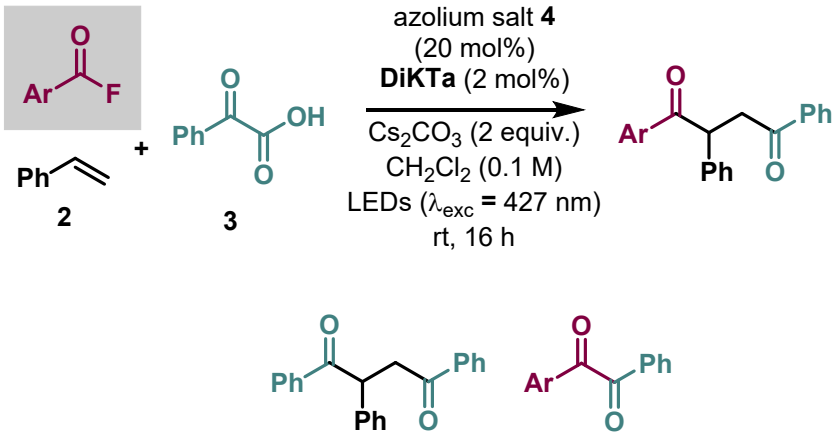
Observed side products

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 *in-situ*, followed by esterification (Scheme 2B).



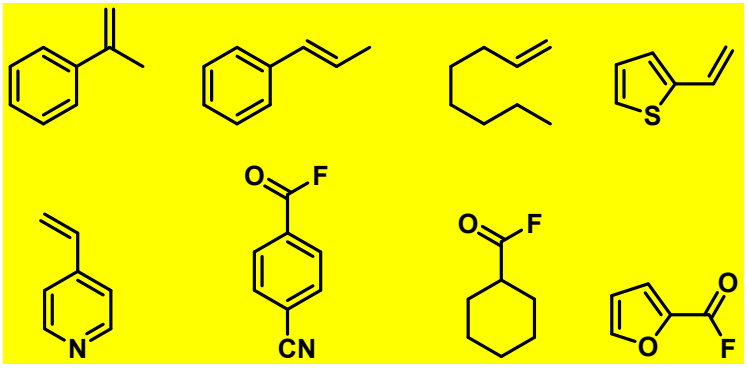
Scheme S2. (A) Dimerization of radical addition products. (B) Benzoin ester formation from in-situ generated aldehydes.

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,[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 ß 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_2CO_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_2Cl_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_2Cl_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_2CO_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_2Cl_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_2Cl_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_2CO_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_2Cl_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_2Cl_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_2CO_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_2Cl_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_2Cl_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$_6$) catalysed conditions for symmetrical 1,4-diones.[8]

To an oven dried vial was added Cs_2CO_3 (130 mg, 0.40 mmol, 2.0 equiv.), $[Ir(ppy)_2(dtbbpy)](PF_6)$ (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 CH_2Cl_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 G – [Ir(ppy)$_2$(dtbbpy)](PF$_6$) catalysed conditions for unsymmetrical 1,4-diones.[8]

To an oven dried vial was added Cs_2CO_3 (130 mg, 0.40 mmol, 2.0 equiv.), $[Ir(ppy)_2(dtbbpy)](PF_6)$ (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 CH_2Cl_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.

Stern-Volmer Quenching Experiments

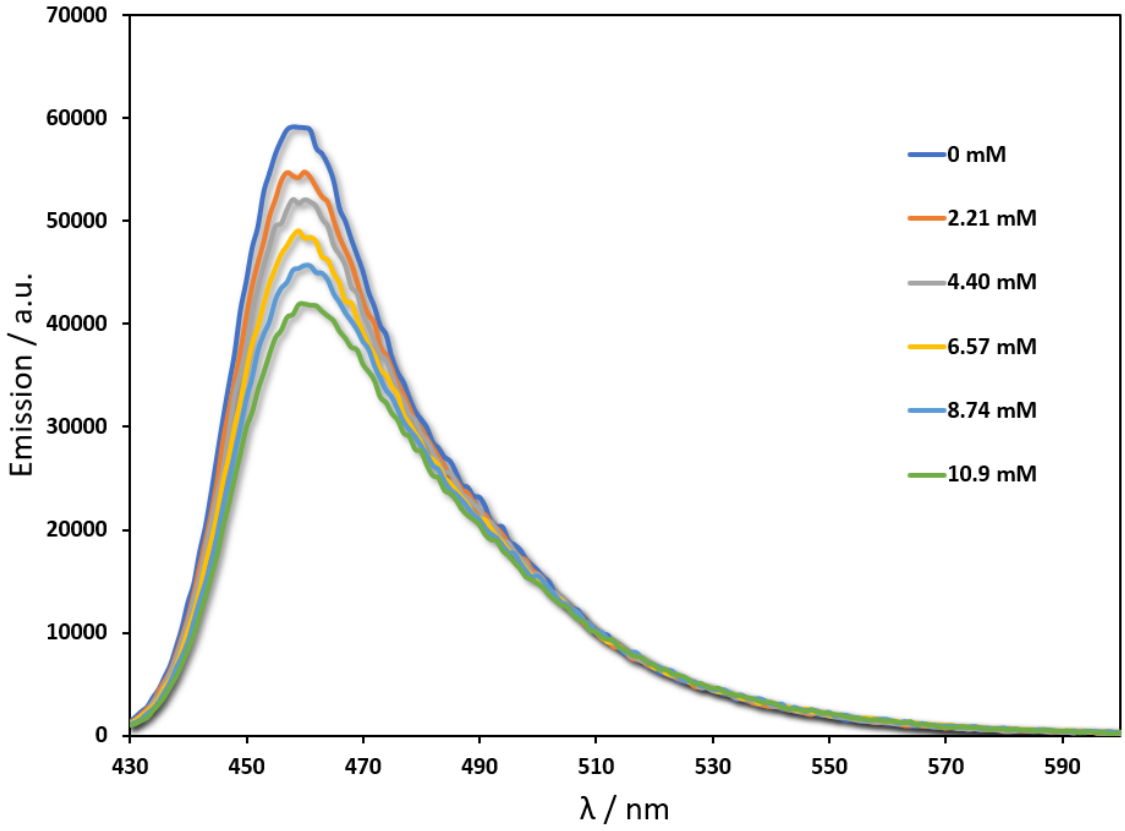


Figure **S4**. Emission quenching data of **DiKTa** by sequential addition of phenylglyoxylic acid in CH_2Cl_2. λ_{exc} = 410 nm.

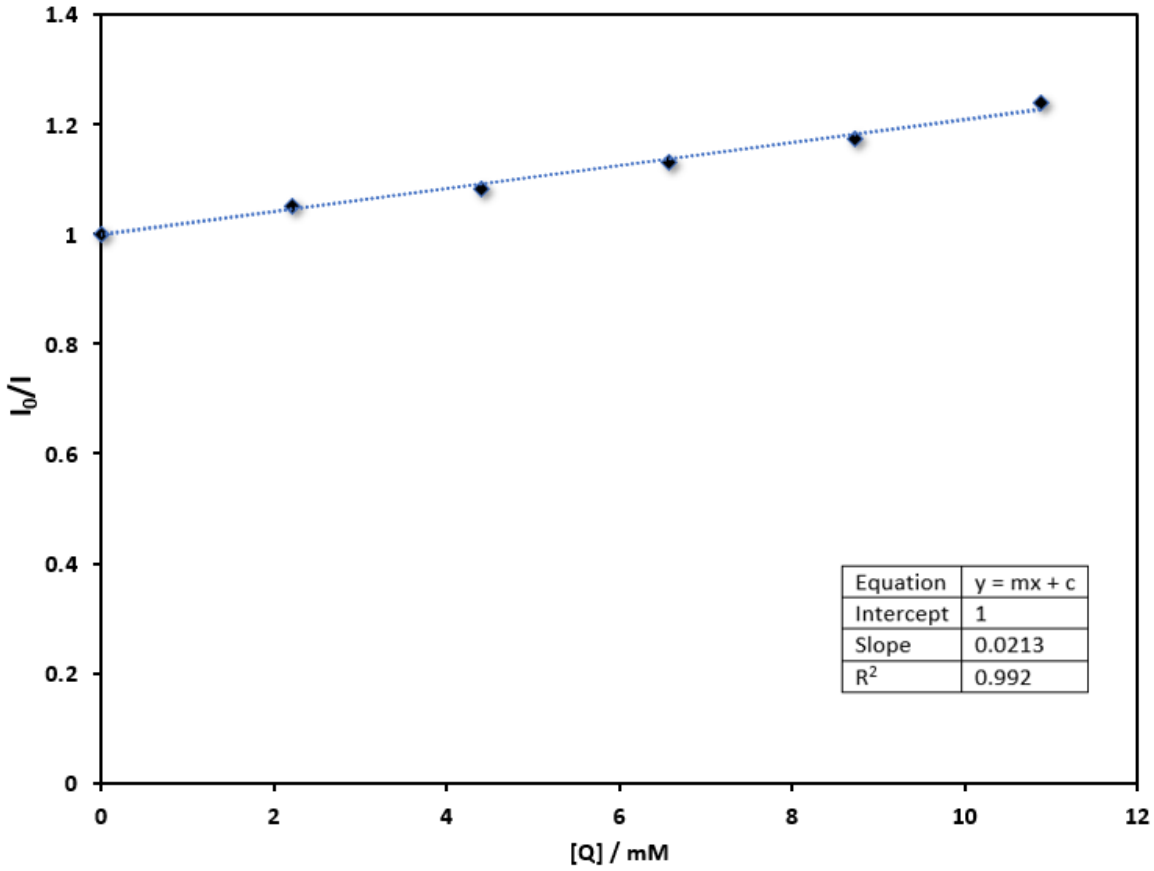


Figure **S5**. Stern-Volmer plot of the quenching of the emission of **DiKTa** in CH_2Cl_2 by sequential addition of phenylglyoxylic acid.

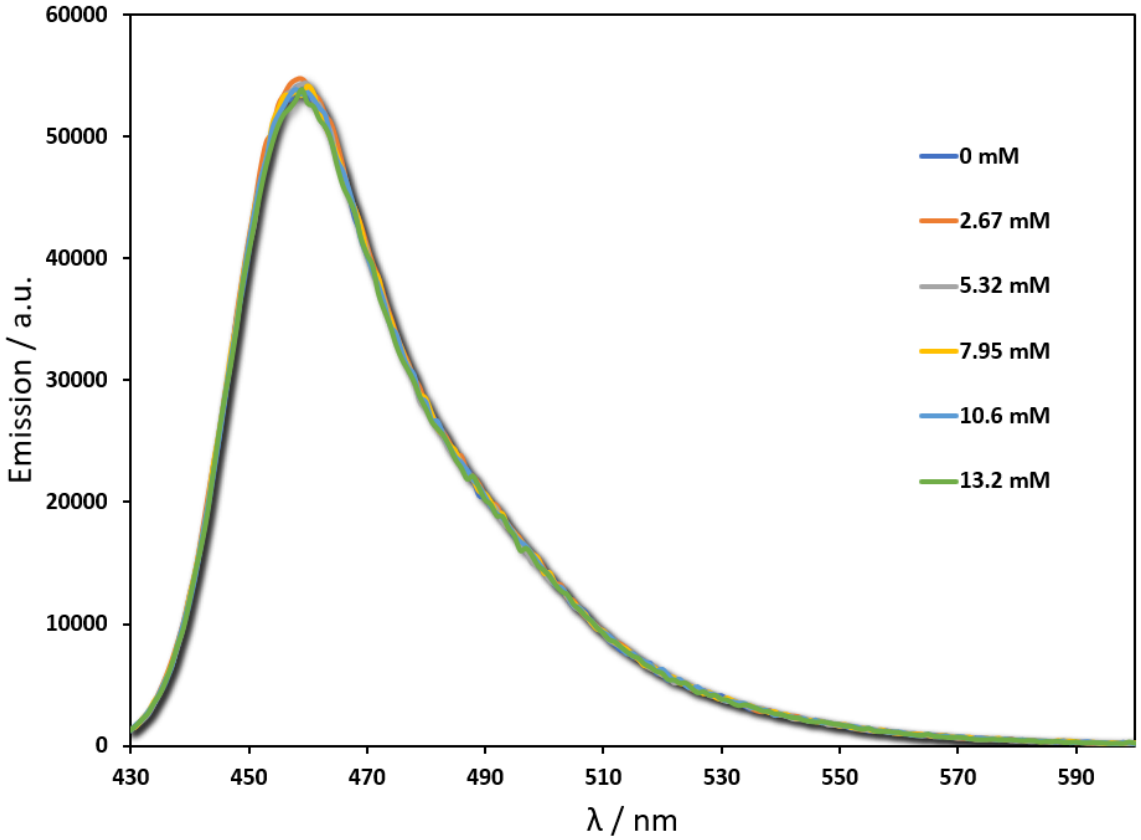


Figure **S6**. Emission quenching data of **DiKTa** by sequential addition of benzoyl fluoride in CH_2Cl_2. λ_{exc} = 410 nm.

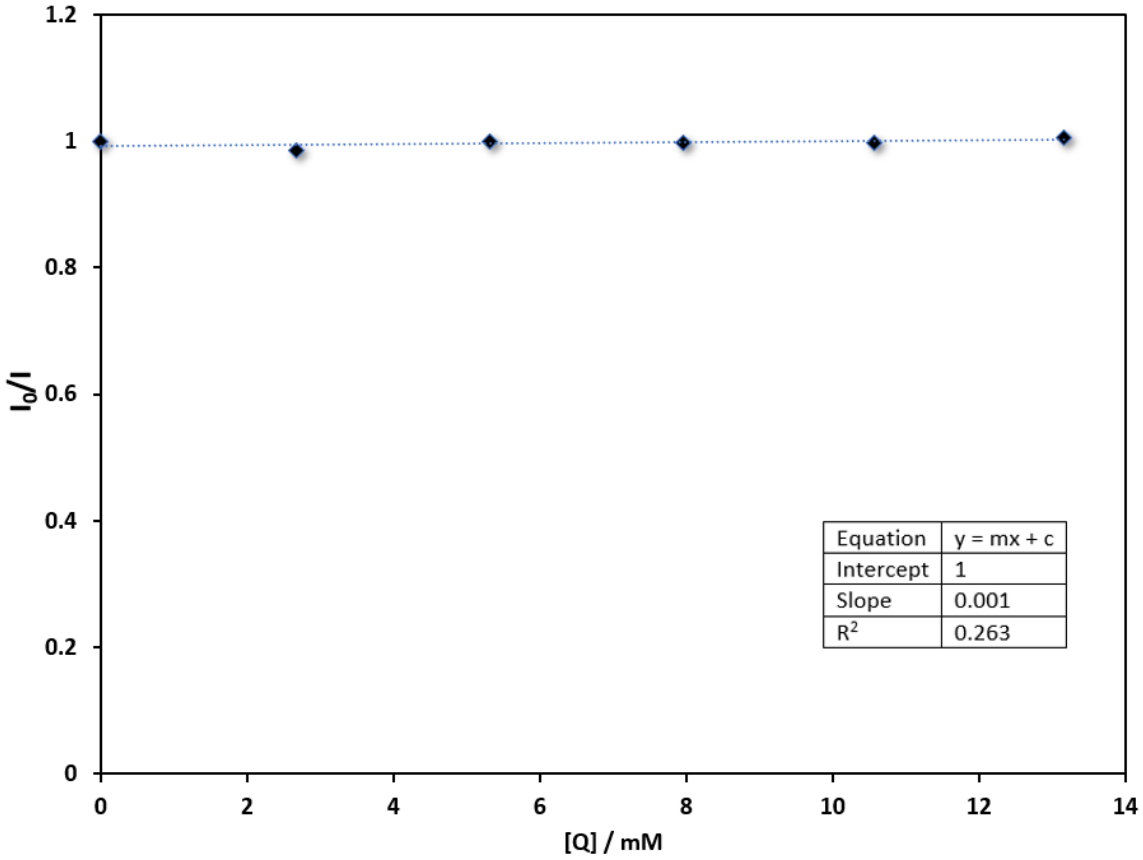


Figure **S7**. Stern-Volmer plot of the quenching of the emission of **DiKTa** in CH_2Cl_2 by sequential addition of benzoyl fluoride.

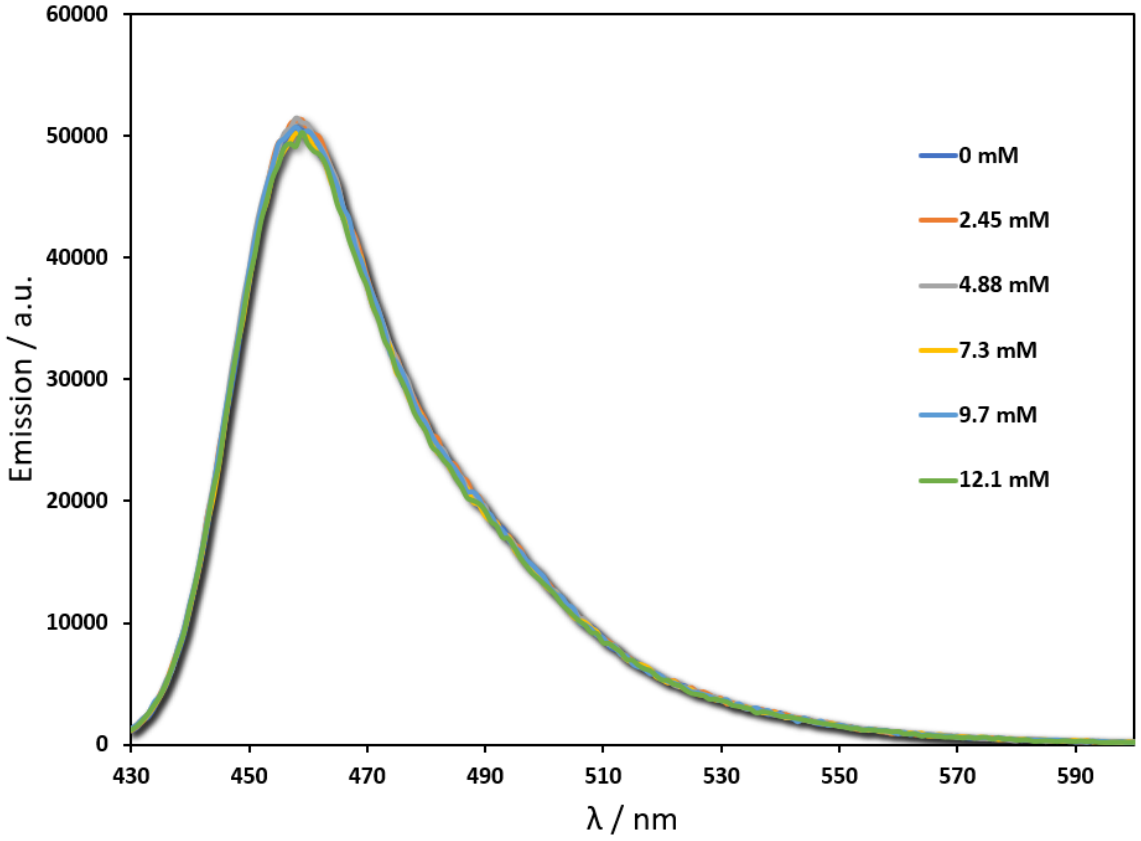


Figure **S8**. Emission quenching data of **DiKTa** by sequential addition of styrene in CH_2Cl_2. λ_{exc} = 410 nm.

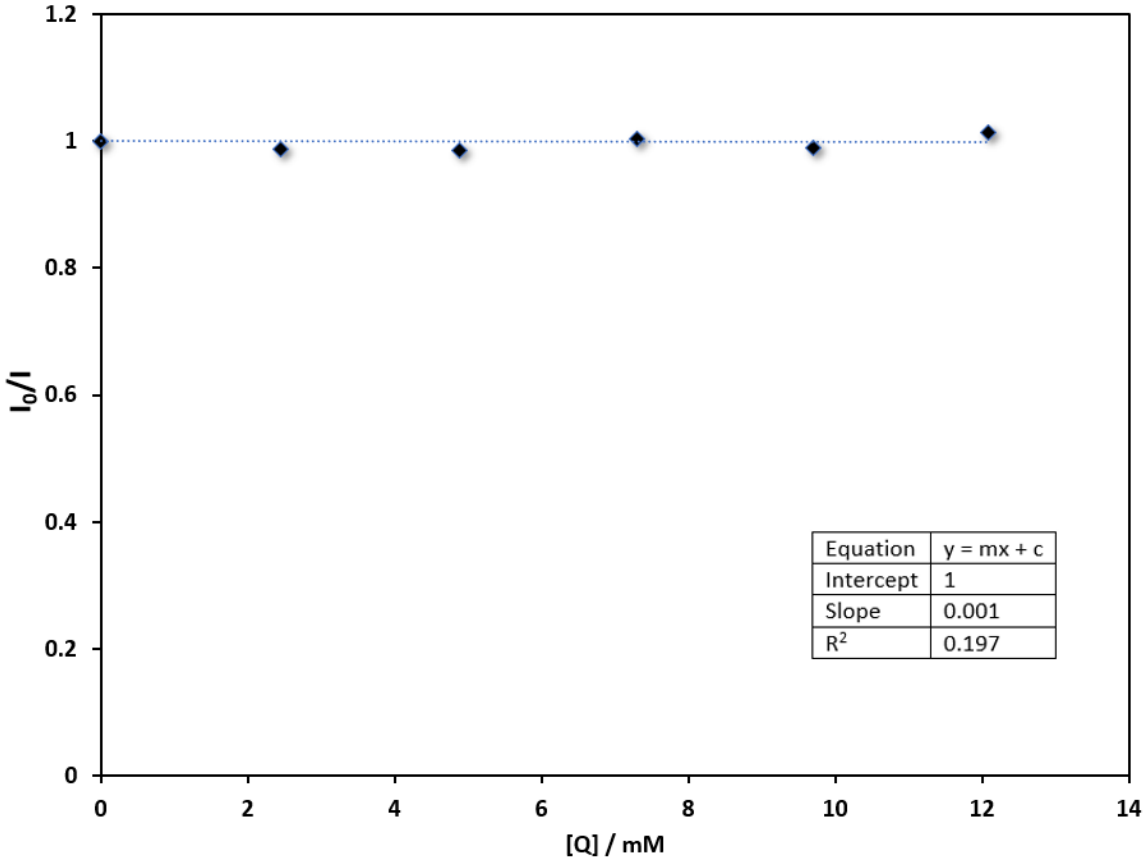


Figure **S9**. Stern-Volmer plot of the quenching of the emission of **DiKTa** in CH_2Cl_2 by sequential addition of styrene.

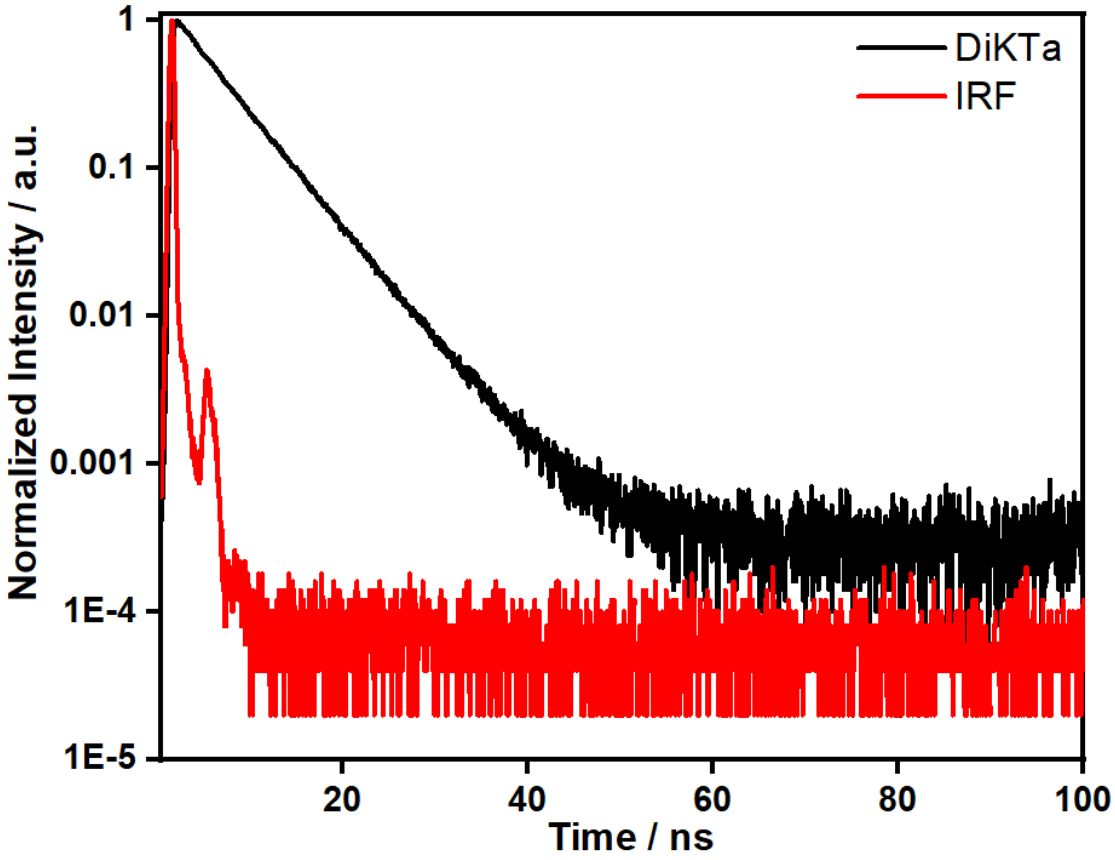


Figure S**10**. Time resolved PL decay of DikTa recorded in CH_2Cl_2 under air in 10-5 M solutions with λ_{exc} = 375 nm.

Quenching constant:

DiKTa with phenylglyoxylic acid

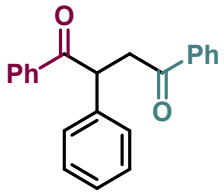
$\tau_{PL} = 5.6 \times 10^{-9}$ s

$K_{SV} = 0.0213$ 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):



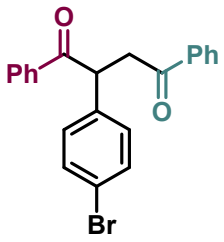
Synthesised using general procedure B to give 18.2 mg of **5** (58%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. **Mp** 121-124 °C {Lit. Mp[9] 124-126 °C}. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.34 (1H, dd, *J* = 18.0 Hz, 3.6 Hz, CHC*H*^{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, Ar*H*), 7.34 (2H, dd, *J* = 8.5 Hz, 6.8 Hz, Ar*H*), 7.37 – 7.55 (7H, m, Ar*H*), 7.56 – 7.61 (1H, m, Ar*H*), 7.98 – 8.04 (2H, m, Ar*H*), 8.05 – 8.09 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 (CH*C*H$_2$), 48.7 (*C*HCH$_2$), 127.4 (Ar*C*), 128.2 (Ar*C*), 128.3 (Ar*C*), 128.5 (Ar*C*), 128.6 (Ar*C*), 129.0 (Ar*C*), 129.2 (Ar*C*), 130.0 (Ar*C*), 132.9 (Ar*C*), 133.3 (Ar*C*), 136.4 (Ar*C*), 138.6 (Ar*C*), 198.1 (*C*=O), 198.9 (*C*=O).

Data matches that previously reported.[10]

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

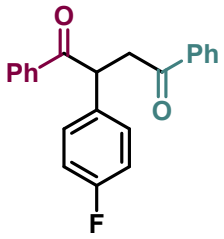


Synthesised using general procedure B to give 17.6 mg of **7** (45%) after column chromatography using EtOAc:Hexane (3:97).

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, CHC*H*^{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, C*H*CH^{A}H^{B}), 7.22 – 7.26 (2H, m, Ar*H*), 7.38 – 7.48 (6H, m, Ar*H*), 7.49 – 7.54 (1H, m, Ar*H*), 7.54 – 7.59 (1H, m, Ar*H*), 7.95 – 8.04 (4H, m, Ar*H*).

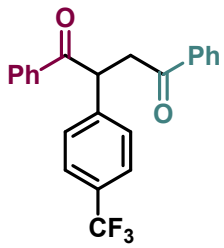
$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.6 ($CHCH_2$), 48.1 ($CHCH_2$), 121.4 (Ar*C*), 128.2 (Ar*C*), 128.60 (Ar*C*), 128.62 (Ar*C*), 128.9 (Ar*C*), 130.0 (Ar*C*), 132.3 (Ar*C*), 133.1 (Ar*C*), 133.3 (Ar*C*), 136.3 (Ar*C*), 136.4 (Ar*C*), 137.7 (Ar*C*), 197.7 (*C*=O), 198.6 (*C*=O).
Data matches that previously reported.[11]

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 °C. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.31 (1H, dd, *J* = 18.0 Hz, 4.0 Hz, $CHCH^{A}H^{B}$), 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, Ar*H*), 7.31 – 7.36 (2H, m, Ar*H*), 7.39 – 7.48 (4H, m, Ar*H*), 7.49 – 7.54 (1H, m, Ar*H*), 7.94 – 8.06 (4H, m, Ar*H*).
^{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, Ar*C*), 128.2 (Ar*C*), 128.60 (Ar*C*), 128.64 (Ar*C*), 128.9 (Ar*C*), 129.8 (d, *J* = 8.1 Hz, Ar*C*), 133.1 (Ar*C*), 133.4 (Ar*C*), 134.3 (d, *J* = 3.1 Hz, Ar*C*),136.3 (Ar*C*), 136.4 (Ar*C*), 162.1 (d, *J* = 245.8 Hz, Ar*C*), 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):



Synthesised using general procedure B to give 16.0 mg of **9** (42%) after column chromatography using EtOAc:Hexane (3:97).

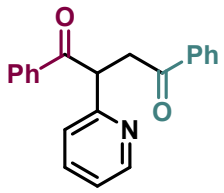
Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.36 (1H, dd, *J* = 18.0 Hz, 4.0 Hz, CHC*H*^{A}H^{B}), 4.24 (1H, dd, *J* = 18.0 Hz, 9.8 Hz, CHCH^{A}*H*^{B}), 5.44 (1H, dd, *J* = 9.7 Hz, 4.0 Hz, C*H*CH^{A}H^{B}), 7.43 – 7.51 (4H, m, Ar*H*), 7.54 (3H, dd, *J* = 9.6 Hz, 7.8 Hz, Ar*H*), 7.57 – 7.62 (3H, m, Ar*H*), 7.99 – 8.03 (2H, m, Ar*H*), 8.03 – 8.07 (2H, m, Ar*H*).

^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm): -62.6

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.7 (CH*C*H$_2$), 48.3 (*C*HCH$_2$), 123.9 (q, *J* = 272.4 Hz *C*F$_3$) 126.1 (q, *J* = 3.5 Hz, Ar*C*), 128.2 (Ar*C*), 128.66 (Ar*C*), 128.68 (Ar*C*), 128.71 (Ar*C*), 128.9 (Ar*C*), 129.7 (q, *J* = 32.7 Hz, Ar*C*) (Ar*C*), 133.3 (Ar*C*), 133.5 (Ar*C*), 136.1 (Ar*C*), 136.2 (Ar*C*), 142.7 (Ar*C*), 197.7 (*C*=O), 198.6 (*C*=O).

Data matches that previously reported.[10]

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



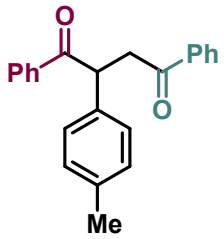
Synthesised using general procedure B to give 17.4 mg of **10** (55%) after column chromatography using EtOAc:Hexane (30:70).

Yellow oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.54 (1H, dd, *J* = 18.0 Hz, 4.1 Hz, CHC*H*^{A}H^{B}), 4.26 (1H, dd, *J* = 18.0 Hz, 9.6 Hz, CHCH^{A}*H*^{B}), 5.60 (1H, dd, *J* = 9.6 Hz, 4.1 Hz, C*H*CH^{A}H^{B}), 7.17 (1H, ddd, *J* = 7.6 Hz, 4.9 Hz, 1.1 Hz, Ar*H*), 7.38 (1H, d, *J* = 7.7 Hz, Ar*H*), 7.42 – 7.50 (4H, m, Ar*H*), 7.51 – 7.56 (1H, m, Ar*H*), 7.56 – 7.61 (1H, m, Ar*H*), 7.64 (1H, td, *J* = 7.7 Hz, 1.8 Hz, Ar*H*), 8.00 – 8.06 (2H, m, Ar*H*), 8.10 – 8.14 (2H, m, Ar*H*), 8,59 (1H, q, *J* = 1.7 Hz, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 42.2 (CH*C*H$_2$), 51.3 (*C*HCH$_2$), 122.2 (Ar*C*), 123.0 (Ar*C*), 128.2 (Ar*C*), 128.57 (Ar*C*), 128.58 (Ar*C*), 129.1 (Ar*C*), 133.0 (Ar*C*), 133.3 (Ar*C*), 136.4 (Ar*C*), 137.1 (Ar*C*), 150.0 (Ar*C*), 158.5 (Ar*C*), 197.8 (*C*=O), 198.0 (*C*=O).

Data matches that previously reported.[7]

1,4-Diphenyl-2-(p-tolyl)butane-1,4-dione (11):



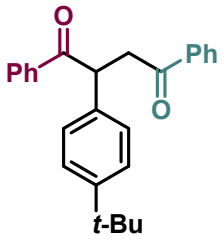
Synthesised using general procedure B to give 18.7 mg of **11** (57%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 2.31 (3H, s, CH_3), 3.31 (1H, dd, *J* = 18.0 Hz, 3.7 Hz, CHCH^AH^B), 4.22 (1H, dd, *J* = 18.0 Hz, 10.0 Hz, CHCH^AH^B), 5.32 (1H, dd, *J* = 10.7 Hz, 3.7 Hz, CHCH^{A}H^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}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 21.1 (*C*H_3), 43.9 (CH*C*H_2), 48.3 (*C*HCH$_2$), 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):



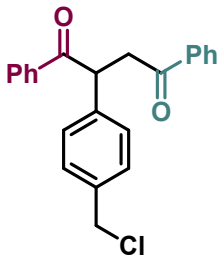
Synthesised using general procedure B to give 15.9 mg of **12** (43%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. **Mp** 106-109 °C. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 1.30 (9H, s, C(CH_3)$_3$), 3.33 (1H, dd, *J* = 18.1 Hz, 3.6 Hz, CHCH^AH^B), 4.24 (1H, dd, *J* = 18.1 Hz, 10.2 Hz, CHCH^AH^B), 5.33 (1H, dd, *J* = 10.3 Hz, 3.6 Hz, CHCH^{A}H^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, $CDCl_3$) δ (ppm): 31.3 (*C*H_3), 34.5 (*C*$(CH_3)_3$) 44.0 (CH*C*H_2), 48.1 (*C*HCH$_2$), 126.1 (Ar*C*), 127.8 (Ar*C*), 128.2 (Ar*C*), 128.5 (Ar*C*), 128.6 (Ar*C*), 129.0 (Ar*C*), 132.8 (Ar*C*), 133.2 (Ar*C*), 135.4 (Ar*C*), 136.5 (Ar*C*), 136.6 (Ar*C*), 150.2 (Ar*C*) 198.2 (*C*=O), 199.0 (*C*=O).

Data matches that previously reported.[7]

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



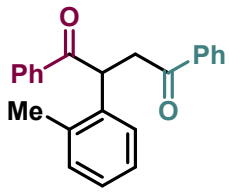
Synthesised using general procedure C to give 14.9 mg of **13** (41%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. **Mp** 155-160 °C. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.33 (1H, dd, *J* = 18.1 Hz, 3.8 Hz, CHC*H*^{A}H^{B}), 4.23 (1H, dd, *J* = 18.0 Hz, 10.0 Hz, CHCH^{A}*H*^{B}), 4.55 (2H, s, C*H*$_2$Cl), 5.37 (1H, dd, *J* = 10.0 Hz, 3.8 Hz, C*H*CH^{A}H^{B}), 7.34 – 7.42 (4H, m, Ar*H*), 7.50 – 7.56 (1H, m, Ar*H*), 7.56 – 7.64 (1H, m, Ar*H*), 7.98 – 8.03 (2H, m, Ar*H*), 8.03 – 8.07 (2H, m, Ar*H*).

$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.8 (CH*C*H$_2$), 45.8 (*C*H$_2$Cl), 48.3 (*C*HCH$_2$), 128.2 (Ar*C*), 128.60 (Ar*C*), 128.63 (Ar*C*), 128.9 (Ar*C*), 129.5 (Ar*C*), 133.1 (Ar*C*), 133.4 (Ar*C*), 136.3 (Ar*C*), 136.4 (Ar*C*), 136.6 (Ar*C*), 138.9 (Ar*C*), 197.9 (*C*=O), 198.7 (*C*=O).

Data matches that previously reported.[7]

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



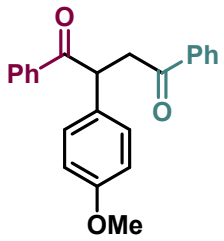
Synthesised using general procedure B to give 12.8 mg of **14** (39%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 2.57 (3H, s), 3.12 (1H, dd, *J* = 18.0 Hz, 2.9 Hz, CHC*H*^{A}H^B), 4.18 (1H, dd, *J* = 18.0 Hz, 10.6 Hz, CHCH^A*H*^{B}), 5.49 (1H, dd, *J* = 10.5 Hz, 2.9 Hz, C*H*CH^{A}H^B), 7.12 (1H, d, *J* = 2.9 Hz, Ar*H*), 7.17 (1H, ddd, *J* = 7.5 Hz, 5,4 Hz, 3.4 Hz, Ar*H*), 7.27 (2H, d, *J* = 13.6 Hz, Ar*H*), 7.40 (2H, t, *J* = 7.7 Hz, Ar*H*), 7.49 (3H, dt, *J* = 9.2 Hz, 7.4 Hz, Ar*H*), 7.57-7.61 (1H, m, Ar*H*), 7.90 – 7.95 (2H, m, Ar*H*), 8.01 – 8.05 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 19.8 (*C*H_3), 42.5 (CH*C*H_2), 45.2 (*C*HCH$_2$), 126.9 (Ar*C*), 127.4 (Ar*C*), 127.5 (Ar*C*), 128.2 (Ar*C*), 128.5 (Ar*C*), 128.6 (Ar*C*), 128.7 (Ar*C*), 131.3 (Ar*C*), 132.8 (Ar*C*), 133.3 (Ar*C*), 135.1 (Ar*C*), 136.5 (Ar*C*), 136.6 (Ar*C*), 137.2 (Ar*C*), 198.2 (*C*=O), 199.5 (*C*=O).

Data matches that previously reported.[11]

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



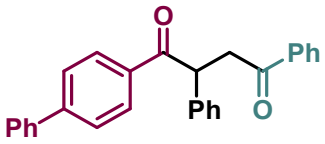
Synthesised using general procedure B to give 8.9 mg of **15** (26%) and general procedure F to give 40.0 mg of **15** (58%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. **Mp** 134-137 °C {Lit. Mp[12] 138-139 °C}. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.29 (1H, dd, *J* = 18.0 Hz, 3.8 Hz, CHC*H*^{A}H^B), 3.76 (3H, s, OC*H*$_3$), 4.18 (1H, dd, *J* = 18.0 Hz, 10.0 Hz, CHCH^A*H*^{B}), 5.28 (1H, dd, *J* = 9.9 Hz, 3.8 Hz, C*H*CH^{A}H^B), 6.82 – 6.87 (2H, m, Ar*H*), 7.26 – 7.29 (2H, m, Ar*H*), 7.38 – 7.51 (5H, m, Ar*H*), 7.52 – 7.58 (1H, m, Ar*H*), 7.96 – 8.01 (2H, m, Ar*H*), 8.01 – 8.05 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 (CH*C*H$_2$), 47.8 (*C*HCH$_2$), 55.3 (OC*H*$_3$), 114.6 (Ar*C*), 128.2 (Ar*C*), 128.5 (Ar*C*), 128.6 (Ar*C*), 128.9 (Ar*C*), 129.3 (Ar*C*), 130.5 (Ar*C*), 132.9 (Ar*C*), 133.3 (Ar*C*), 136.48 (Ar*C*), 136.51 (Ar*C*), 158.8 (Ar*C*), 198.3 (*C*=O), 199.1 (*C*=O).

Data matches that previously reported.[11]

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



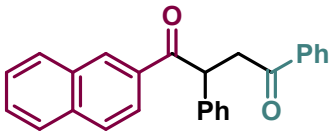
Synthesised using general procedure D to give 20.7 mg of **16** (53%) after column chromatography using EtOAc:Hexane (3:97).

Colourless solid. **Mp** 196-199 °C {Lit. Mp[13] 200 °C}. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.33 (1H, dd, *J* = 18.0 Hz, 3.7 Hz, CHC*H*^{A}H^B), 4.25 (1H, dd, *J* = 18.0 Hz, 10.1 Hz, CHCH^A*H*^{B}), 5.36 (1H, dd, *J* = 10.1 Hz, 3.6 Hz, C*H*CH^{A}H^B), 7.21 – 7.25 (1H, m, Ar*H*), 7.33 (2H, dd, *J* = 8.5 Hz, 6.8 Hz, Ar*H*), 7.37 – 7.42 (3H, m, Ar*H*), 7.42 – 7.49 (4H, m, Ar*H*), 7.54 – 7.61 (3H, m, Ar*H*), 7.61 – 7.65 (2H, m, Ar*H*), 7.97 – 8.02 (2H, m, Ar*H*), 8.09 – 8.14 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 (CH*C*H$_2$), 48.7 (*C*HCH$_2$), 122.6 (Ar*C*), 127.3 (Ar*C*), 127.4 (Ar*C*), 128.1 (Ar*C*), 128.2 (Ar*C*), 128.3 (Ar*C*), 128.6 (Ar*C*), 128.9 (Ar*C*), 129.3 (Ar*C*), 129.5 (Ar*C*), 133.3 (Ar*C*), 135.1 (Ar*C*), 136.5 (Ar*C*), 138.7 (Ar*C*), 140.0 (Ar*C*), 143.3 (Ar*C*), 145.6 (Ar*C*), 198.1 (*C*=O), 198.5 (*C*=O).

Data matches that previously reported.[14]

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



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

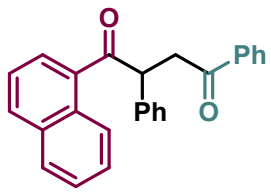
Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.37 (1H, dd, *J* = 18.1 Hz, 3.7 Hz, CHC*H*^{A}H^B), 4.28 (1H, dd, *J* = 18.0 Hz, 10.0 Hz, CHCH^A*H*^{B}), 5.50 (1H, dd, *J* = 10.0 Hz, 3.7 Hz, C*H*CH^{A}H^B), 7.22 (1H, t, *J* = 7.7 Hz, Ar*H*), 7.32 (2H, t, *J* = 7.6 Hz, Ar*H*), 7.38 –

7.60 (7H, m, Ar*H*), 7.80 – 7.87 (2H, m, Ar*H*), 7.93 (1H, d, *J* = 8.0 Hz, Ar*H*) 7.99 – 8.05 (2H, m, Ar*H*), 8.07 (1H, dd, *J* = 8.7 Hz, 1.8 Hz, Ar*H*), 8.61 (1H, d, *J* = 1.7 Hz, Ar*H*).

$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 ($CHCH_2$), 48.8 ($CHCH_2$), 124.7 (Ar*C*), 126.6 (Ar*C*), 127.4 (Ar*C*), 127.7 (Ar*C*), 128.2 (Ar*C*), 128.3 (Ar*C*), 128.36 (Ar*C*), 128.39 (Ar*C*), 128.6 (Ar*C*), 129.3 (Ar*C*), 129.7 (Ar*C*), 130.8 (Ar*C*), 132.5 (Ar*C*), 133.3 (Ar*C*), 133.8 (Ar*C*), 135.5 (Ar*C*), 136.5 (Ar*C*), 138.8 (Ar*C*), 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):



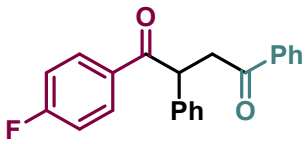
Synthesised using general procedure D to give 9.8 mg of **18** (27%) after column chromatography using EtOAc:Hexane (3:97).

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, CHC*H*^{A}H^{B}), 4.40 (1H, dd, *J* = 18.0 Hz, 10.5 Hz, CHCH^{A}*H*^{B}), 5.35 (1H, dd, *J* = 10.5 Hz, 3.4 Hz, C*H*CH^{A}H^{B}), 7.19 – 7.25 (1H, m, Ar*H*), 7.31 (2H, d, *J* = 7.6 Hz, Ar*H*), 7.37 – 7.42 (2H, m, Ar*H*), 7.50 (5H, dddd, *J* = 9.4 Hz, 7.4 Hz, 3.2 Hz, 1.6 Hz, Ar*H*), 7.59 – 7.63 (1H, m, Ar*H*) 7.83 (1H, dd, *J* = 8.3 Hz, 1.6 Hz, Ar*H*), 7.95 (1H, d, *J* = 8.2 Hz, Ar*H*), 8.03 – 8.09 (2H, m, Ar*H*), 8.20 (1H, dd, *J* = 7.2 Hz, 1.2 Hz, Ar*H*), 8.36 (1H, dd, *J* =8.3 Hz, 1.6 Hz, Ar*H*).

$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.5 ($CHCH_2$), 52.3 ($CHCH_2$), 124.4 (Ar*C*), 125.7 (Ar*C*), 126.2 (Ar*C*), 127.5 (Ar*C*), 127.6 (Ar*C*), 127.7 (Ar*C*), 128.19 (Ar*C*), 128.21 (Ar*C*), 128.3 (Ar*C*), 128.7 (Ar*C*), 129.1 (Ar*C*), 130.7 (Ar*C*), 132.3 (Ar*C*), 133.3 (Ar*C*), 133.8 (Ar*C*), 136.4 (Ar*C*), 136.6 (Ar*C*), 137.9 (Ar*C*), 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).

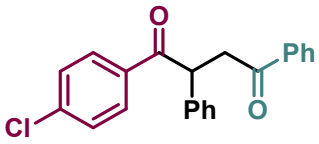
Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.33 (1H, dd, *J* = 18.1 Hz, 3.5 Hz, CHC*H*^{A}H^{B}), 4.24 (1H, dd, *J* = 18.1 Hz, 10.2 Hz, CHCH^{A}*H*^{B}), 5.29 (1H, dd, *J* = 10.2 Hz, 3.5 Hz, C*H*CH^{A}H^{B}), 7.05 – 7.09 (2H, m, Ar*H*), 7.22 – 7.26 (1H, m, Ar*H*), 7.30 – 7.37 (4H, m, 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*).

^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm): -105.4.

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 (CH*C*H$_2$), 48.7 (*C*HCH$_2$), 115.6 (d, *J* = 21.8 Hz, Ar*C*), 127.5 (Ar*C*), 128.2 (d, *J* = 2.6 Hz, Ar*C*), 128.6 (Ar*C*), 129.3 (Ar*C*), 131.6 (d, *J* = 9.2 Hz, Ar*C*), 133.4 (Ar*C*), 136.3 (Ar*C*), 138.4 (Ar*C*), 165.6 (d, *J* = 254.3 Hz, Ar*C*), 197.4 (*C*=O), 198.1 (*C*=O).

Data matches that previously reported.[14]

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

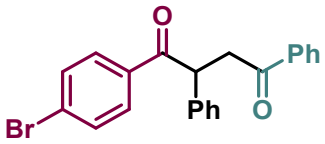


Synthesised using general procedure D to give 17.1 mg of **20** (49%) after column chromatography using EtOAc:Hexane (3:97).

Yellow oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.31 (1H, dd, *J* = 18.1 Hz, 3.6 Hz, CHC*H*^{A}H^{B}), 4.21 (1H, dd, *J* = 18.1 Hz, 10.2 Hz, CHCH^{A}*H*^{B}), 5.26 (1H, dd, *J* = 10.2 Hz, 3.6 Hz, C*H*CH^{A}H^{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*).

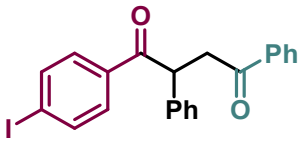
$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 (CH*C*H_2), 48.8 (*C*HCH_2), 127.6 (Ar*C*), 128.2 (Ar*C*), 128.6 (Ar*C*), 128.9 (Ar*C*), 129.3 (Ar*C*), 130.4 (d, *J* = 9.2 Hz, Ar*C*), 133.4 (Ar*C*), 134.8 (Ar*C*), 136.3 (Ar*C*), 138.3 (Ar*C*), 139.3 (Ar*C*), 197.8 (*C*=O), 198.0 (*C*=O).
Data matches that previously reported.[15]

1-(4-Bromophenyl)-2,4-diphenylbutane-1,4-dione (21):



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}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.31 (1H, dd, *J* = 18.1 Hz, 3.5 Hz, CHC*H*^{A}H^{B}), 4.21 (1H, dd, *J* = 18.1 Hz, 10.2 Hz, CHCH^{A}*H*^{B}), 5.24 (1H, dd, *J* = 10.2 Hz, 3.5 Hz, C*H*CH^{A}H^{B}), 7.24 (1H, dd, *J* = 5.9 Hz, 2.9 Hz, Ar*H*), 7.28 – 7.35 (4H, m, Ar*H*), 7.45 (2H, t, *J* = 7.7 Hz, Ar*H*), 7.55 (3H, dd, *J* = 8.5 Hz, 6.7 Hz Ar*H*), 7.86 – 7.92 (2H, m, Ar*H*), 7.94 – 8.02 (2H, m, Ar*H*).
$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 (CH*C*H_2), 48.8 (*C*HCH_2), 127.6 (Ar*C*), 128.2 (Ar*C*), 128.6 (Ar*C*), 129.3 (Ar*C*), 130.5 (Ar*C*), 131.9 (Ar*C*), 133.4 (Ar*C*), 135.2 (Ar*C*), 136.3 (Ar*C*), 138.2 (Ar*C*), 139.3 (Ar*C*), 197.98 (*C*=O), 198.02 (*C*=O).
Data matches that previously reported.[16]

1-(4-Iodophenyl)-2,4-diphenylbutane-1,4-dione (22):



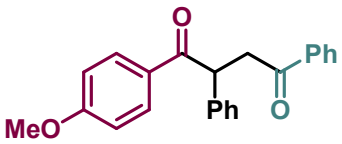
Synthesised using general procedure D to give 8.8 mg of **22** (20%) and general procedure G to give 44.0 mg of **22** (50%) after column chromatography using EtOAc:Hexane (3:97). Colourless solid. **Mp** 107-109 °C **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.33 (1H, dd, *J* = 18.1 Hz, 3.6 Hz, CHC*H*^{A}H^{B}), 4.23 (1H, dd, *J* = 18.1 Hz, 10.2 Hz, CHCH^{A}*H*^{B}), 5.26 (1H, dd, *J* = 10.2 Hz, 3.6 Hz, C*H*CH^{A}H^{B}), 7.24 – 7.30 (3H, m, Ar*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*).

$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.9 ($CHCH_2$), 48.7 ($CHCH_2$), 101.0 (Ar*C*), 127.6 (Ar*C*), 128.2 (Ar*C*), 128.6 (Ar*C*), 129.3 (Ar*C*), 130.3 (Ar*C*), 133.4 (Ar*C*), 135.7 (Ar*C*), 136.3 (Ar*C*), 137.9 (Ar*C*), 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):



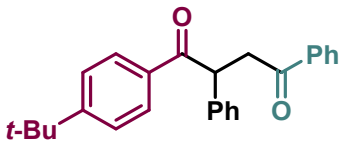
Synthesised using general procedure D to give 19.6 mg of **23** (57%) after column chromatography using EtOAc:Hexane (3:97).

Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.30 (1H, dd, *J* = 18.0 Hz, 3.8 Hz, CHC*H*^{A}H^{B}), 3.84 (3H, s, OCH_3), 4.23 (1H, dd, *J* = 18.0 Hz, 10.0 Hz, CHCH^{A}*H*^{B}), 5.32 (1H, dd, *J* = 10.0 Hz, 3.8 Hz, C*H*CH^{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*).

$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ (ppm): 43.8 ($CHCH_2$), 48.4 ($CHCH_2$), 55.4 (OCH_3), 113.7 (Ar*C*), 127.3 (Ar*C*), 128.2 (Ar*C*), 128.6 (Ar*C*), 129.2 (Ar*C*), 129.4 (Ar*C*), 131.3 (Ar*C*), 133.2 (Ar*C*), 136.6 (Ar*C*), 139.2 (Ar*C*), 163.4 (Ar*C*), 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):



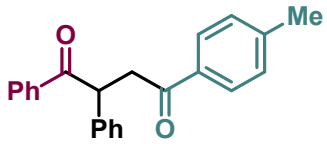
Synthesised using general procedure D to give 13.3 mg of **24** (36%) after column chromatography using EtOAc:Hexane (3:97).

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, CHC*H*^{A}H^B), 4.22 (1H, dd, *J* = 18.0 Hz, 10.1 Hz, CHCH^A*H*^{B}), 5.33 (1H, dd, *J* = 10.1 Hz, 3.7 Hz, C*H*CH^{A}H^B), 7.19 – 7.26 (1H, m, Ar*H*), 7.32 (2H, ddd, *J* = 7.8 Hz, 6.8 Hz, 1.2 Hz, Ar*H*), 7.36 – 7.47 (6H, m, Ar*H*), 7.52 – 7.58 (1H, m, Ar*H*), 7.95 – 8.01 (4H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 31.1 (*C*H_3), 35.1 (*C*(CH$_3$)$_3$), 44.0 (CH*C*H$_2$), 48.5 (*C*HCH$_2$), 125.5 (Ar*C*), 127.3 (Ar*C*), 128.2 (Ar*C*), 128.3 (Ar*C*), 128.6 (Ar*C*), 128.9 (Ar*C*), 129.2 (Ar*C*), 133.3 (Ar*C*), 133.8 (Ar*C*), 136.5 (Ar*C*), 138.9 (Ar*C*), 156.6 (Ar*C*), 198.2 (*C*=O), 198.5 (*C*=O).

Data matches that previously reported.[14]

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

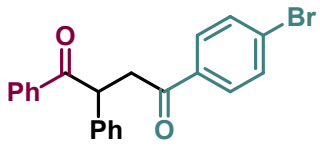


Synthesised using general procedure E to give 9.8 mg of **25** (30%) and general procedure G to give 32.8 mg of **25** (50%) after column chromatography using EtOAc:Hexane (3:97). Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 2.40 (3H, s, C*H*$_3$) 3.29 (1H, dd, *J* = 18.0 Hz, 3.6 Hz, CHC*H*^{A}H^B), 4.19 (1H, dd, *J* = 18.0 Hz, 10.0 Hz, CHCH^A*H*^{B}), 5.32 (1H, dd, *J* = 10.1 Hz, 3.7 Hz, C*H*CH^{A}H^B), 7.21 – 7.26 (3H, m, Ar*H*), 7.28 – 7.34 (2H, m, Ar*H*), 7.34 – 7.43 (4H, m, Ar*H*), 7.46 – 7.52 (1H, m, Ar*H*), 7.86 – 7.90 (2H, m, Ar*H*), 8.02 – 8.05 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 21.7 (*C*H$_3$), 43.8 (CH*C*H$_2$), 48.7 (*C*HCH$_2$), 127.3 (Ar*C*), 128.27 (Ar*C*), 128.31 (Ar*C*), 128.5 (Ar*C*), 129.0 (Ar*C*), 129.2 (Ar*C*), 129.3 (Ar*C*), 132.9 (Ar*C*), 134.0 (Ar*C*), 136.5 (Ar*C*), 138.7 (Ar*C*), 144.1 (Ar*C*), 198.2 (*C*=O), 198.5 (*C*=O).

Data matches that previously reported.[18]

4-(4-Bromophenyl)-1,2-diphenylbutane-1,4-dione (26):

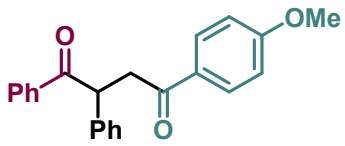


Synthesised using general procedure E to give 10.6 mg of **26** (27%) and general procedure G to give 42.5 mg of **26** (54%) after column chromatography using EtOAc:Hexane (3:97). Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.24 (1H, dd, *J* = 18.0 Hz, 3.7 Hz, CHC*H*^{A}H^B), 4.16 (1H, dd, *J* = 18.0 Hz, 10.0 Hz, CHCH^A*H*^{B}), 5.31 (1H, dd, *J* = 10.0 Hz, 3.7 Hz, C*H*CH^{A}H^B), 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*), 8.00 – 8.03 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.8 (CH*C*H$_2$), 48.8 (*C*HCH$_2$), 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):

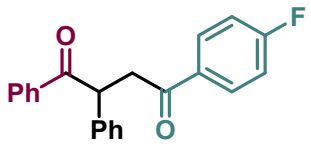


Synthesised using general procedure E to give 7.6 mg of **27** (22%) and general procedure G to give 30.3 mg of **27** (44%) after column chromatography using EtOAc:Hexane (3:97). Colourless foam. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.27 (1H, dd, *J* = 17.8 Hz, 3.7 Hz, CHC*H*^{A}H^B), 3.86 (3H, s, OC*H*$_3$), 4.17 (1H, dd, *J* = 17.8 Hz, 10.1 Hz, CHCH^A*H*^{B}), 5.32 (1H, dd, *J* = 10.1 Hz, 3.7 Hz, C*H*CH^{A}H^B), 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{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.6 (CH*C*H$_2$), 48.7 (*C*HCH$_2$), 55.5 (O*C*H$_3$), 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):



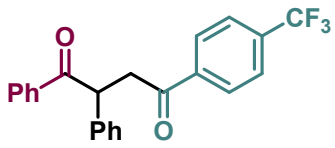
Synthesised using general procedure E to give 7.3 mg of **28** (22%) and general procedure G to give 47.2 mg of **28** (71%) after column chromatography using EtOAc:Hexane (3:97). Colourless oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.26 (1H, dd, *J* = 17.9 Hz, 3.7 Hz, CHC*H*^{A}H^{B}), 4.18 (1H, dd, *J* = 17.9 Hz, 10.1 Hz, CHCH^{A}*H*^{B}), 5.31 (1H, dd, *J* = 10.1 Hz, 3.7 Hz, C*H*CH^{A}H^{B}), 7.12 (2H, t, *J* = 8.6 Hz, Ar*H*), 7.21 – 7.25 (1H, m, Ar*H*), 7.29 – 7.37 (4H, m, Ar*H*), 7.38 – 7.42 (2H, m, Ar*H*), 7.47 – 7.52 (1H, m, Ar*H*), 7.98 – 8.05 (4H, m, Ar*H*).

^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm): -104.9.

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 43.8 (CH*C*H_2), 48.8 (*C*HCH_2), 115.6 (d, *J* = 21.9 Hz, Ar*C*) 127.5 (Ar*C*), 128.2 (Ar*C*), 128.6 (Ar*C*), 129.0 (Ar*C*), 129.3 (Ar*C*), 130.8 (d, *J* = 9.4 Hz, Ar*C*), 133.0 (Ar*C*), 136.3 (Ar*C*), 138.5 (Ar*C*), 165.9 (d, *J* = 254.2 Hz, Ar*C*), 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):



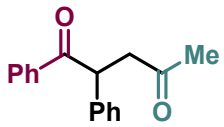
Synthesised using general procedure E to give 6.5 mg of **29** (17%) and general procedure G to give 22.2 mg of **29** (29%) after column chromatography using EtOAc:Hexane (3:97). Yellow Oil. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.31 (1H, dd, *J* = 18.0 Hz, 3.7 Hz, CHC*H*^{A}H^{B}), 4.25 (1H, dd, *J* = 18.0 Hz, 10.1 Hz, CHCH^{A}*H*^{B}), 5.35 (1H, dd, *J* = 10.1 Hz, 3.6 Hz, C*H*CH^{A}H^{B}), 7.24 – 7.28 (1H, m, Ar*H*), 7.33 – 7.40 (4H, m, Ar*H*), 7.43 (2H, t, *J* = 7.7 Hz, Ar*H*), 7.50 – 7.55 (1H, m, Ar*H*), 7.73 – 7.78 (2H, m, Ar*H*), 8.03 – 8.07 (2H, m, Ar*H*), 8.10 – 8.14 (2H, m, Ar*H*).

^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm): -63.1

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 44.1 (CH*C*H$_2$), 48.8 (*C*HCH$_2$), 123.5 (q, *J* = 272.4 Hz, *C*F$_3$), 125.7 (q, *J* = 3.9 Hz, Ar*C*), 127.6 (Ar*C*), 128.2 (Ar*C*), 128.5 (Ar*C*), 128.6 (Ar*C*), 129.0 (Ar*C*), 129.3 (Ar*C*), 133.1 (Ar*C*), 134.6 (q, *J* = 32.6 Hz, Ar*C*), 136.2 (Ar*C*), 138.3 (Ar*C*), 139.1.6 (Ar*C*), 197.3 (*C*=O), 198.7 (*C*=O).

Data matches that previously reported.[14]

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



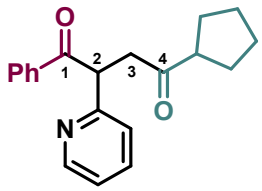
Synthesised using general procedure E to give 11.4 mg of **30** (45%) after column chromatography using EtOAc:Hexane (10:90).

Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 2.19 (3H, s, C*H*$_3$) 2.76 (1H, dd, *J* = 18.0 Hz, 4.0 Hz, CHC*H*^{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, C*H*CH^{A}H^B), 7.20 (1H, ddd, *J* = 8.1 Hz, 4.6 Hz, 2.4 Hz, Ar*H*), 7.26 – 7.31 (4H, m, Ar*H*), 7.37 (2H, dd, *J* = 8.3 Hz, 6.9 Hz, Ar*H*), 7.44 – 7.50 (1H, m, Ar*H*), 7.94 – 7.98 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 30.0 (*C*H$_3$), 48.1 (CH*C*H$_2$), 48.8 (*C*HCH$_2$), 127.3 (Ar*C*), 128.1 (Ar*C*), 128.4 (Ar*C*), 128.9 (Ar*C*), 129.2 (Ar*C*), 132.9 (Ar*C*), 138.6 (Ar*C*), 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):



Synthesised using general procedure E to give 12.3 mg of **31** (40%) after column chromatography using EtOAc:Hexane (20:80).

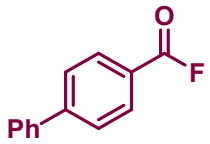
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 1.51 – 1.89 (8H, m, CH_2CH_2CH_2CH_2), 2.87 – 3.01 (2H, m, CH_2CHCH_2 + C(3)H^AH^B), 3.66 (1H, dd, *J* = 17.9 Hz, 9.9 Hz, C(3)H^AH^B), 5.37 (1H, dd, *J* = 9.8 Hz, 4.2 Hz, C(2)*H*), 7.11 (1H, ddd, *J* = 7.6 Hz, 4.9 Hz, 1.2 Hz, Ar*H*), 7.23 – 7.26 (1H, m, Ar*H*), 7.35 – 7.41 (2H, m, Ar*H*), 7.45 – 7.50 (1H, m, Ar*H*), 7.58 (1H, td, *J* = 7.7 Hz, 1.8 Hz, Ar*H*), 8.00 – 8.05 (2H, m, Ar*H*).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 26.0 ($CH_2CH_2CH_2CH_2$), 26.1 ($CH_2CH_2CH_2CH_2$), 28.8 ($CH_2CH_2CH_2CH_2$), 44.9 (*C*(3)), 51.1 (*C*(2)), 51.2 (CH_2CHCH_2), 122.1 (Ar*C*), 122.9 (Ar*C*), 128.5 (Ar*C*), 129.0 (Ar*C*), 133.0 (Ar*C*), 136.4 (Ar*C*), 137.1 (Ar*C*), 150.0 (Ar*C*), 158.5 (Ar*C*), 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):

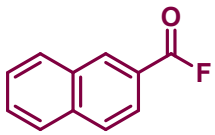


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 7.41 – 7.54 (3H, m, Ar*H*), 7.62 – 7.66 (2H, m, Ar*H*), 7.72 – 7.77 (2H, m, Ar*H*), 8.09 – 8.15 (2H, m, Ar*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 18.1.

Data matches that previously reported.[21]

2-Naphthoyl fluoride (33):

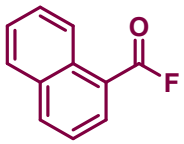


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 7.62 (1H, ddd, *J* = 8.1 Hz, 6.9 Hz, 1.3 Hz, Ar*H*), 7.69 (1H, ddd, *J* = 8.2 Hz, 6.9 Hz, 1.3 Hz, Ar*H*), 7.90 – 8.03 (4H, m, Ar*H*), 8.63 – 8.65 (1H, m, Ar*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 18.1.

Data matches that previously reported.[21]

1-Naphthoyl fluoride (34):

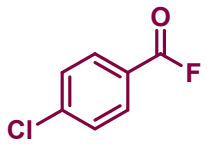


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 7.58 (1H, dd, *J* = 8.2 Hz, 7.4 Hz, Ar*H*), 7.69 (1H, ddd, *J* = 8.2 Hz, 6.9 Hz, 1.3 Hz, Ar*H*), 7.90 – 8.03 (4H, m, Ar*H*), 8.63 – 8.65 (1H, m, Ar*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 29.9.

Data matches that previously reported.[21]

4-Chlorobenzoyl fluoride (35):

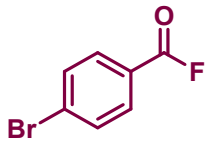


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 7.50 – 7.59 (2H, m, C(3)*H*), 7.97 – 8.06 (2H, m, C(2)*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 29.9.

Data matches that previously reported.[21]

4-Bromobenzoyl fluoride (36):

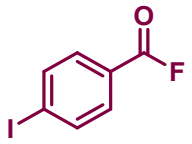


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 7.61 – 7.81 (2H, m, C(3)*H*), 7.81 – 8.00 (2H, m, C(2)*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 18.4.

Data matches that previously reported.[22]

4-Iodobenzoyl fluoride (37):

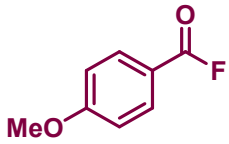


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 7.70 – 7.78 (2H, m, C(3)*H*), 7.87 – 7.95 (2H, m, C(2)*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 18.4.

Data matches that previously reported.[21]

4-Methoxybenzoyl fluoride (38):

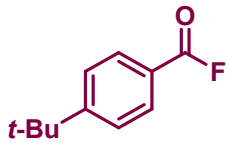


Colourless oil. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 3.92 (3H, s, OCH_3), 6.95 – 7.09 (2H, m, C(3)*H*), 7.99 – 8.06 (2H, m, C(2)*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 16.0.

Data matches that previously reported.[21]

4-(*tert*-butyl)benzoyl fluoride (39):

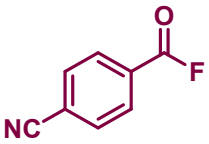


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 1.35 (9H, s, C(CH_3)$_3$), 7.54 (2H, dd, *J* = 8.6 Hz, 1.4 Hz, (C(3)*H*), 7.98 (2H, d, *J* = 8.5 Hz, C(2)*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 17.7.

Data matches that previously reported.

4-Cyanobenzoyl fluoride (40):

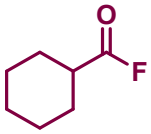


Colourless solid. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 7.67 – 7.92 (2H, m, C(3)*H*), 8.17 (2H, d, *J* = 8.4 Hz, C(2)*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 20.2.

Data matches that previously reported.[23]

Cyclohexanecarbonyl fluoride (41):

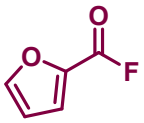


Colourless oil. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 1.23 – 1.34 (3H, m, CH_2), 1.45 – 1.62 (2H, m, CH_2), 1.62 (1H, m, CH_2), 1.62 – 1.72 (1H, m, CH_2), 1.75 – 1.80 (2H, m, CH_2), 1.95 – 2.02 (2H, m, CH_2), 2.47 – 2.54 (1H, m, C*H*).

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 36.7.

Data matches that previously reported.[21]

2-Furanoyl fluoride (42):

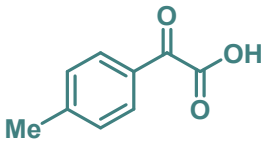


Colourless oil. **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):** 6.65 (1H, ddd, *J* = 3.6 Hz, 1.8 Hz, 0.8 Hz, C(4)*H*), 7.45 (1H, dd, *J* = 3.7 Hz, 0.8 Hz, C(3)*H*), 7.77 (1H, ddd, *J* = 2.5 Hz, 1.7 Hz, 0.8 Hz, C(4)*H*)

^{19}F NMR (376 MHz, $CDCl_3$) δ (ppm): 15.4.

Data matches that previously reported.[24]

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

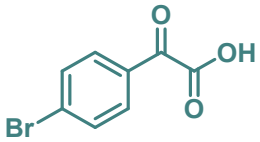


Colourless solid. **Mp** 97-99 °C {Lit. Mp[25] 97-99 °C}. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 2.46 (3H, s, CH_3), 7.34 (2H, d, *J* = 8.1 Hz, ArC(3)*H*), 8.28 (2H, d, *J* = 8.0 Hz, ArC(2)*H*)

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 22.1 (*C*H_3), 129.2 (Ar*C*), 129.8 (Ar*C*), 131.7 (Ar*C*), 147.4 (Ar*C*), 161.3 (*C*(O)OH), 183.6 (*C*=O)).

Data matches that previously reported.[26]

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

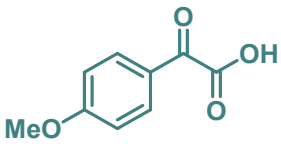


Colourless solid. **Mp** 97-99 °C {Lit. Mp[27] 100-102 °C}. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 7.66 – 7.70 (2H, m, ArC(3)*H*), 8.20 – 8.24 (2H, m, ArC(2)*H*)

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 130.5 (Ar*C*), 131.6 (Ar*C*), 132.4 (Ar*C*), 132.7 (Ar*C*), 161.1 (*C*(O)OH), 183.8 (*C*=O)).

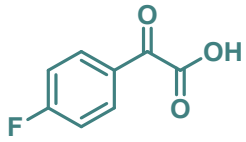
Data matches that previously reported.[26]

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



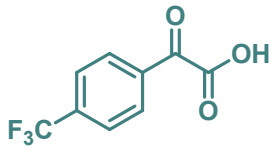
Colourless solid. **Mp** 74-76 °C {Lit. Mp[28] 79-81 °C}. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 3.91 (3H, s, OC*H*$_3$), 6.95 – 7.00 (2H, m, ArC(3)*H*), 8.33 – 8.39 (2H, m, ArC(2)*H*)
^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 55.7 (O*C*H$_3$), 114.4 (Ar*C*), 124.9 (Ar*C*), 162.6 (*C*(O)OH), 165.6 (Ar*C*), 183.0 (*C*=O).
Data matches that previously reported.[26]

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



Colourless solid. **Mp** 95-97 °C {Lit. Mp[29] 95-96 °C}. **^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm):** 7.14 – 7.25 (2H, m, ArC(3)*H*), 8.41 – 8.49 (2H, m, ArC(2)*H*)
^{19}F NMR (471 MHz, $CDCl_3$) δ (ppm): -99.2
^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 116.5 (d, *J* = 22.3 Hz, Ar*C*), 128.2 (Ar*C*), 134.6 (d, *J* = 10.0 Hz, Ar*C*), 163.8 (d, *J* = 662.7 Hz, Ar*C*), 168.5 (*C*(O)OH), 182.4 (*C*=O).
Data matches that previously reported.[26]

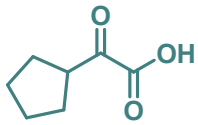
2-oxo-2-(4-(trifluoromethyl)phenyl)acetic acid (47):



Colourless solid. **Mp** 56-59 °C {Lit. Mp[27] 53-55 °C}. **^{1}H NMR (400 MHz, $CDCl_3$) 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, O*H*).
^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ (ppm): 123.2 (q, *J* = 273.0 Hz, *C*F$_3$), 126.0 (q, *J* = 3.7 Hz), 131.5 (Ar*C*), 134.4 (Ar*C*), 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, $CDCl_3$) δ (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, $CDCl_3$) δ (ppm): 26.2 (*C*H_2) 28.8 (*C*H_2), 45.6 (*C*H), 159.9 (*C*(O)OH), 197.7 (*C*=O).

Data matches that previously reported.[31]

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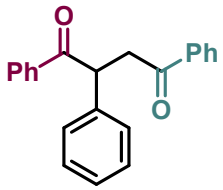
NMR Spectra



5, ^{1}H, $CDCl_3$, 500 MHz

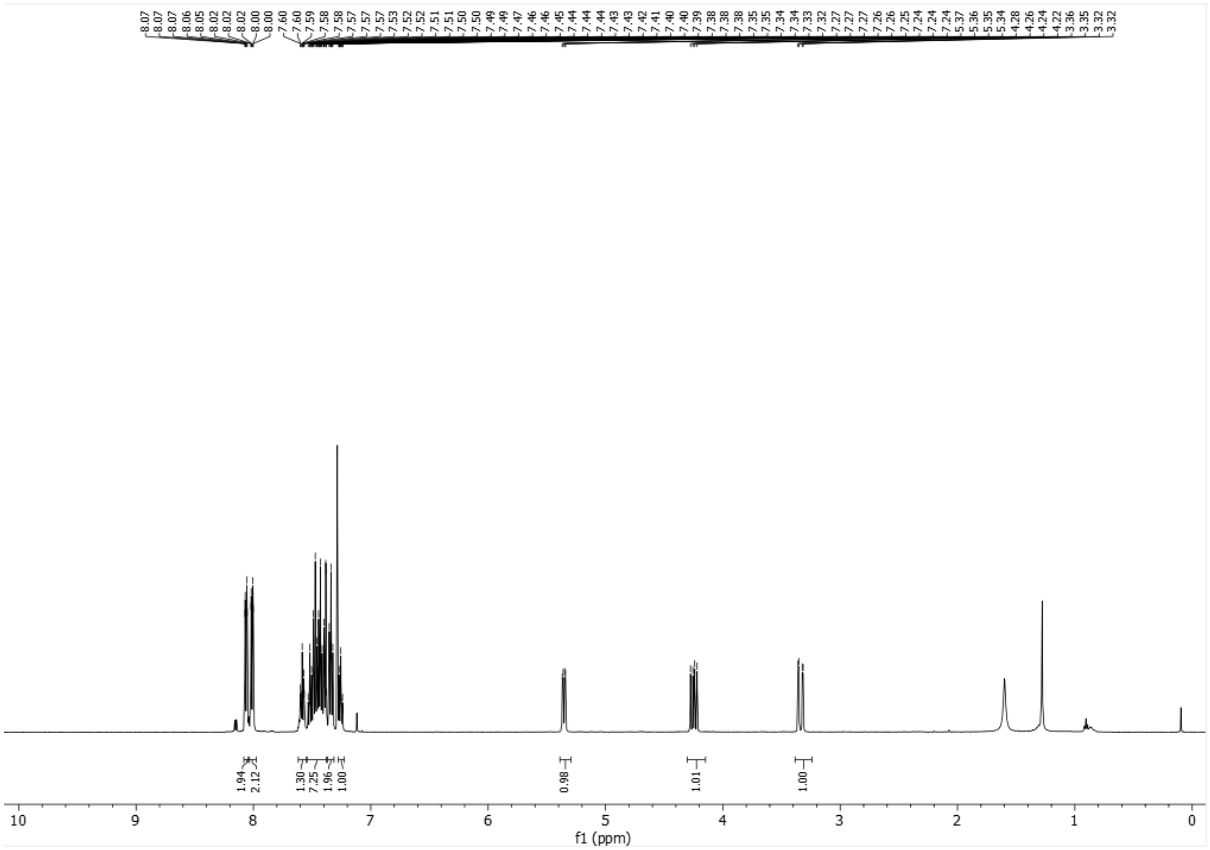


Figure **S11**. ^{1}H NMR spectrum of 2-Phenyl-1,4-diphenylbutane-1,4-dione in $CDCl_3$.

5, ^{13}C, $CDCl_3$, 126 MHz

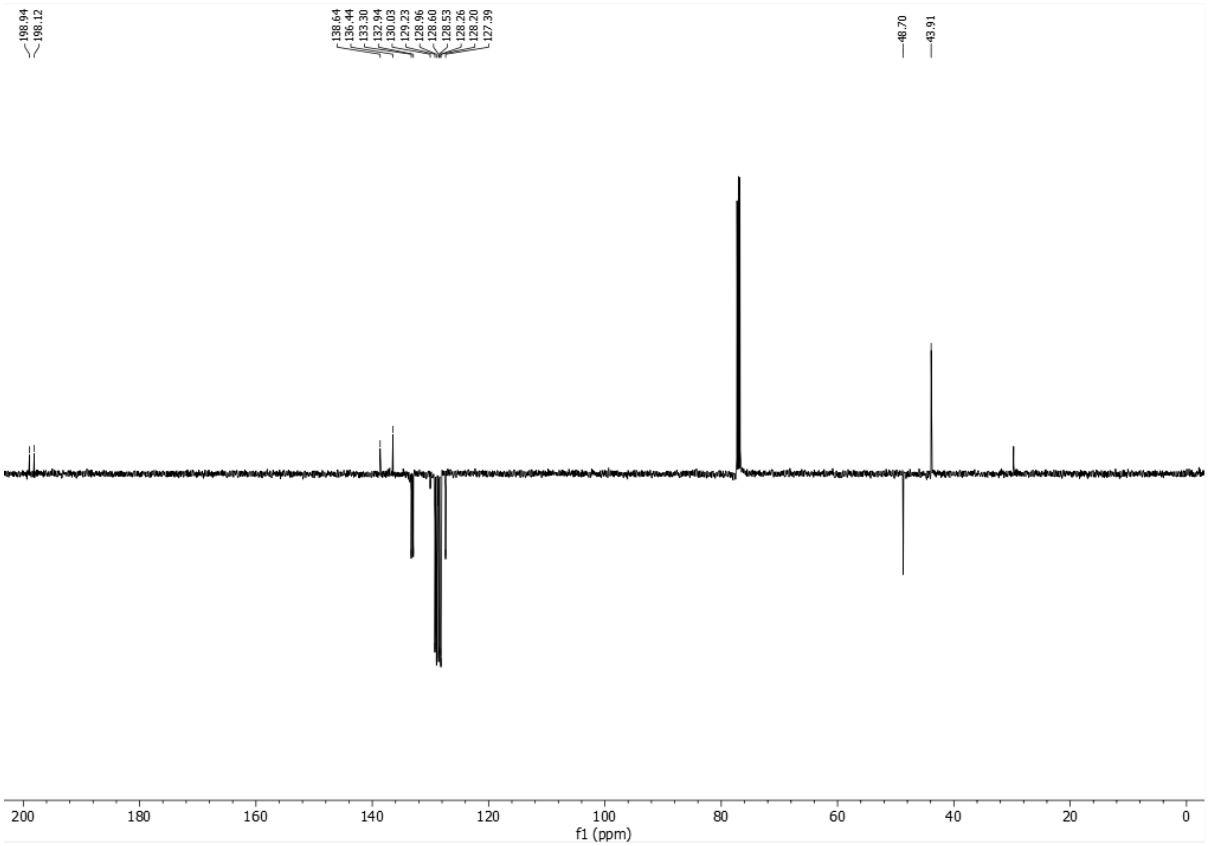
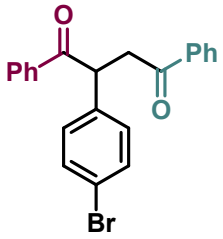


Figure **S12**. ^{13}C NMR spectrum of 2-Phenyl-1,4-diphenylbutane-1,4-dione in $CDCl_3$.



7, ^{1}H, $CDCl_3$, 500 MHz

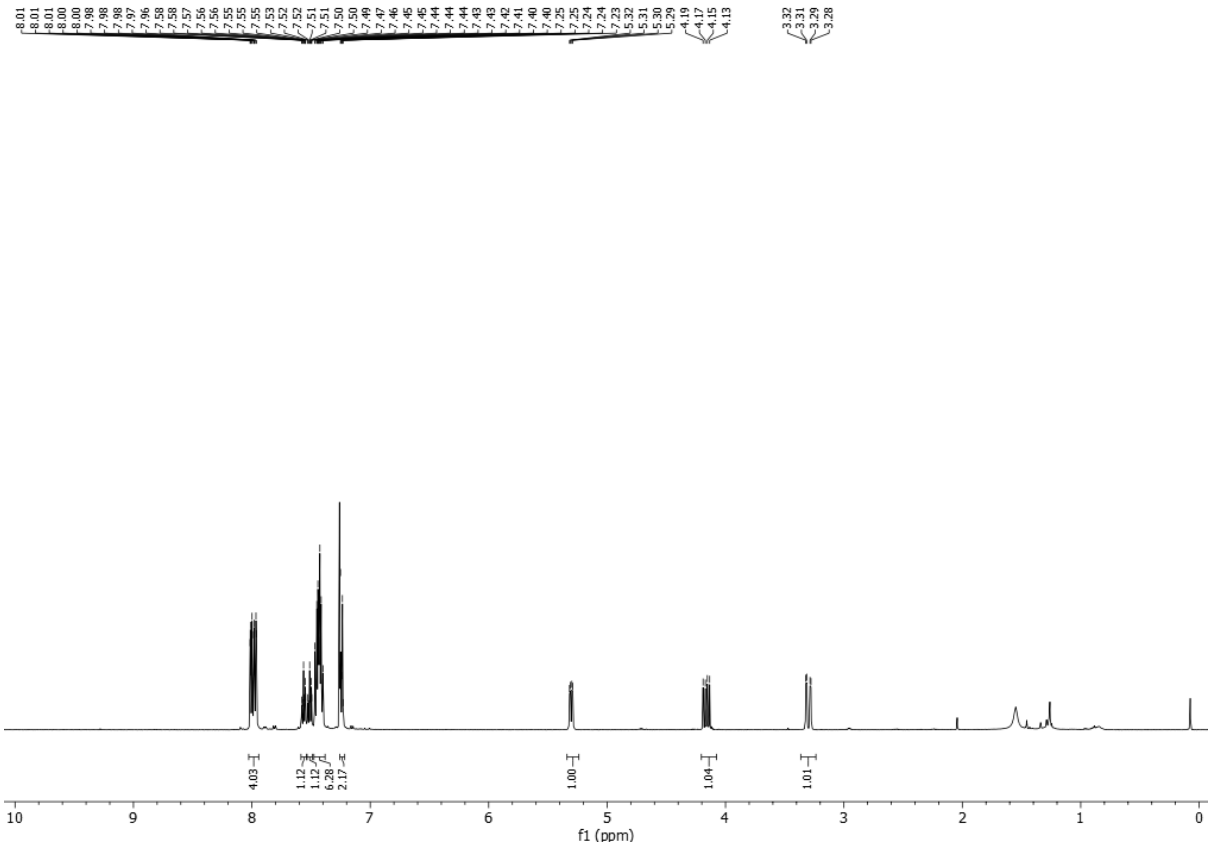


Figure **S13**. ^{1}H NMR spectrum of 2-(4-bromophenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.

7, ^{13}C, $CDCl_3$, 126 MHz

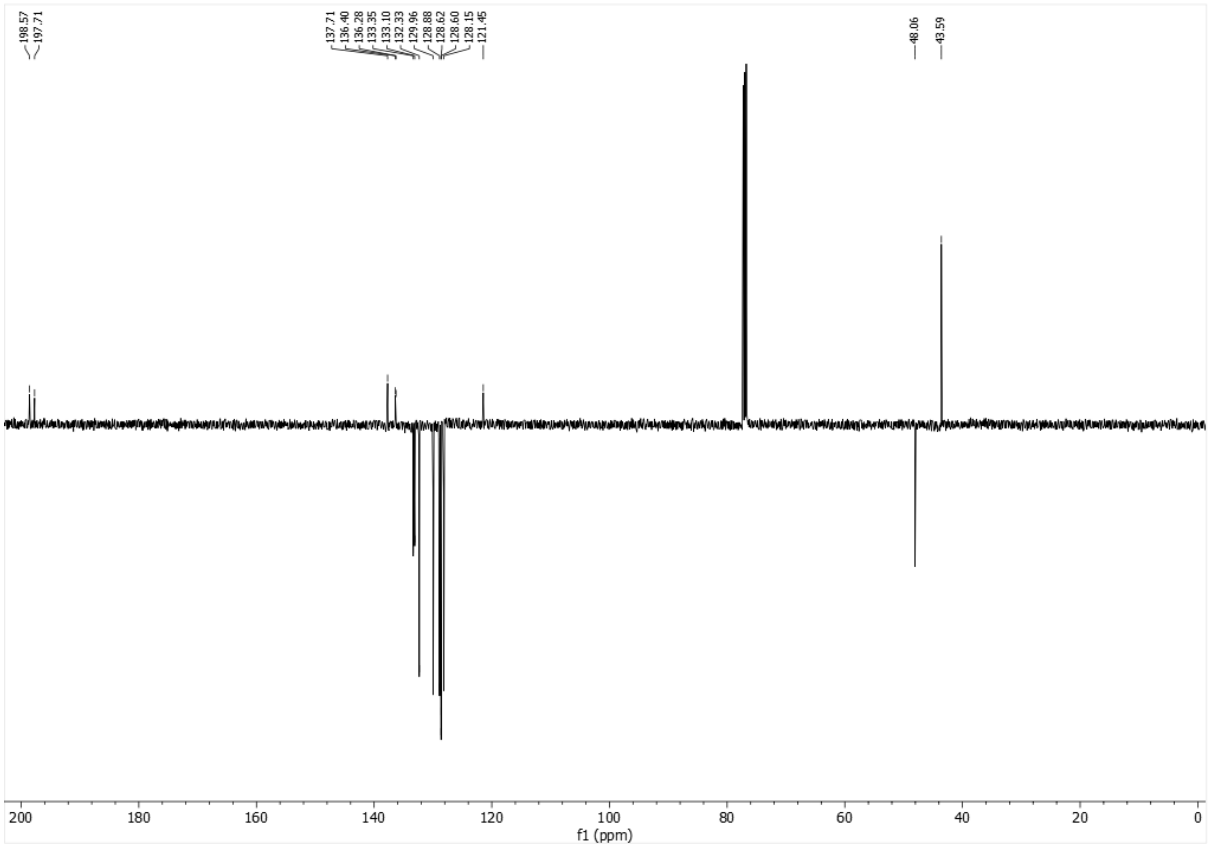
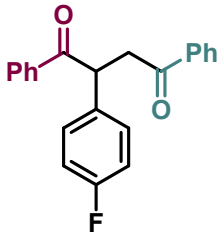


Figure **S14**. ^{13}C NMR spectrum of 2-(4-bromophenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.



8, ^{1}H, $CDCl_3$, 500 MHz

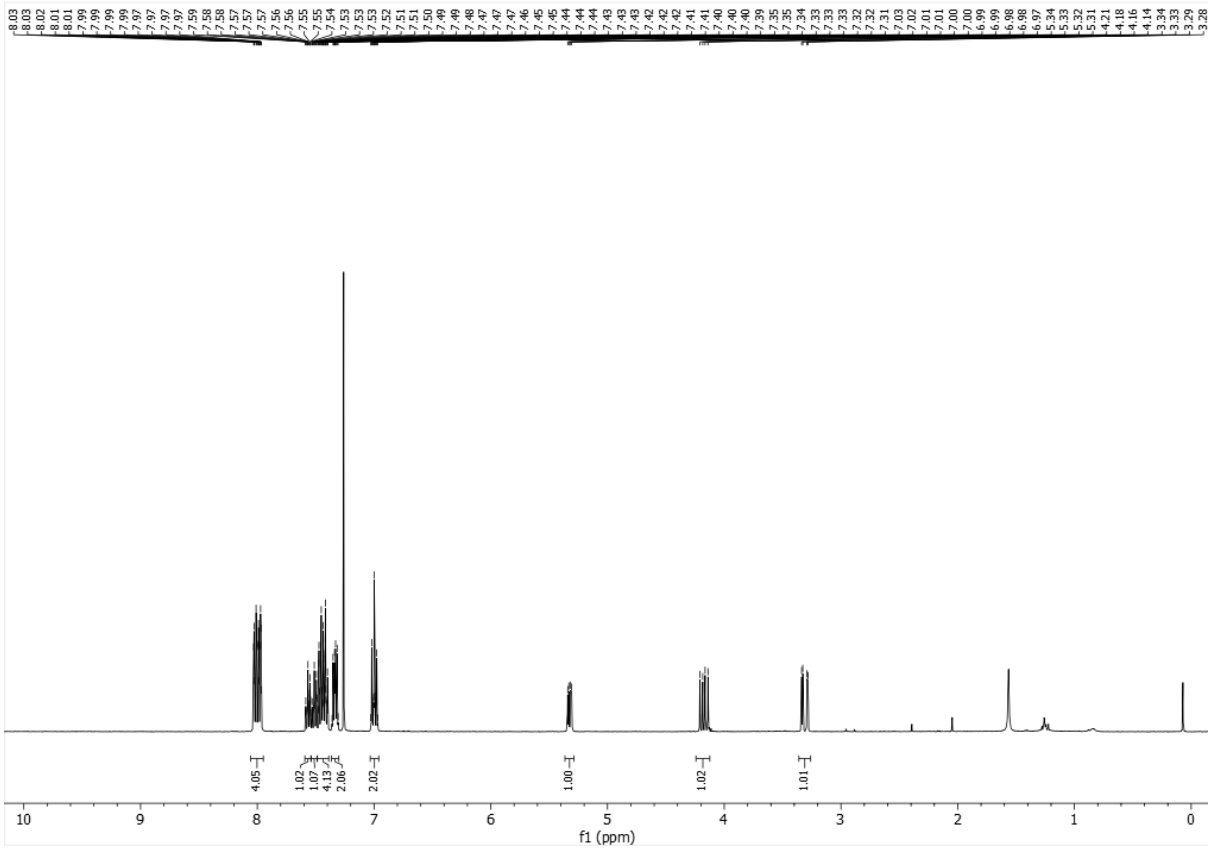


Figure **S15**. ^{1}H NMR spectrum of 2-(4-fluorophenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.

8, ^{19}F, $CDCl_3$, 471 MHz

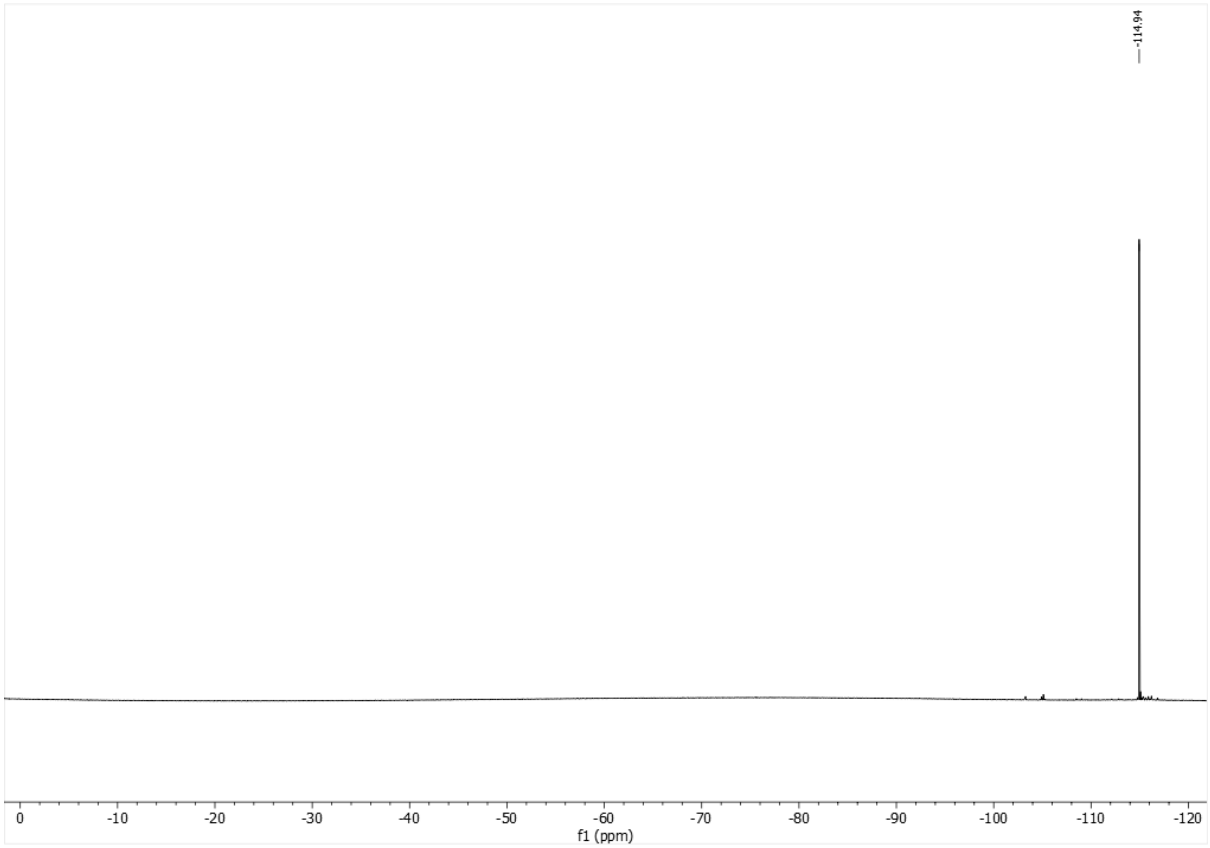


Figure **S16**. ^{19}F NMR spectrum of 2-(4-fluorophenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.

8, ^{13}C, $CDCl_3$, 126 MHz

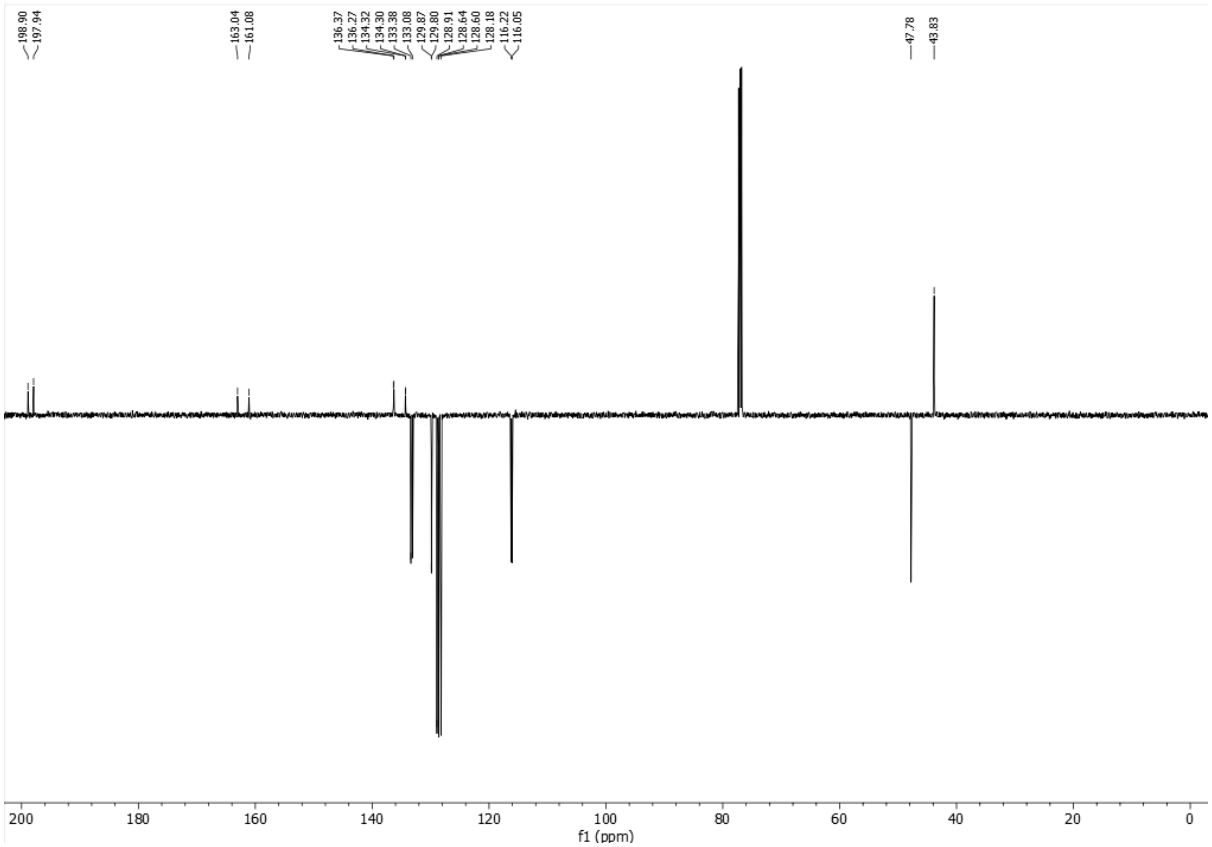
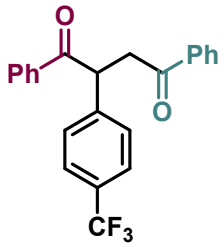


Figure **S17**. ^{13}C NMR spectrum of 2-(4-fluorophenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.



9, ^{1}H, $CDCl_3$, 500 MHz

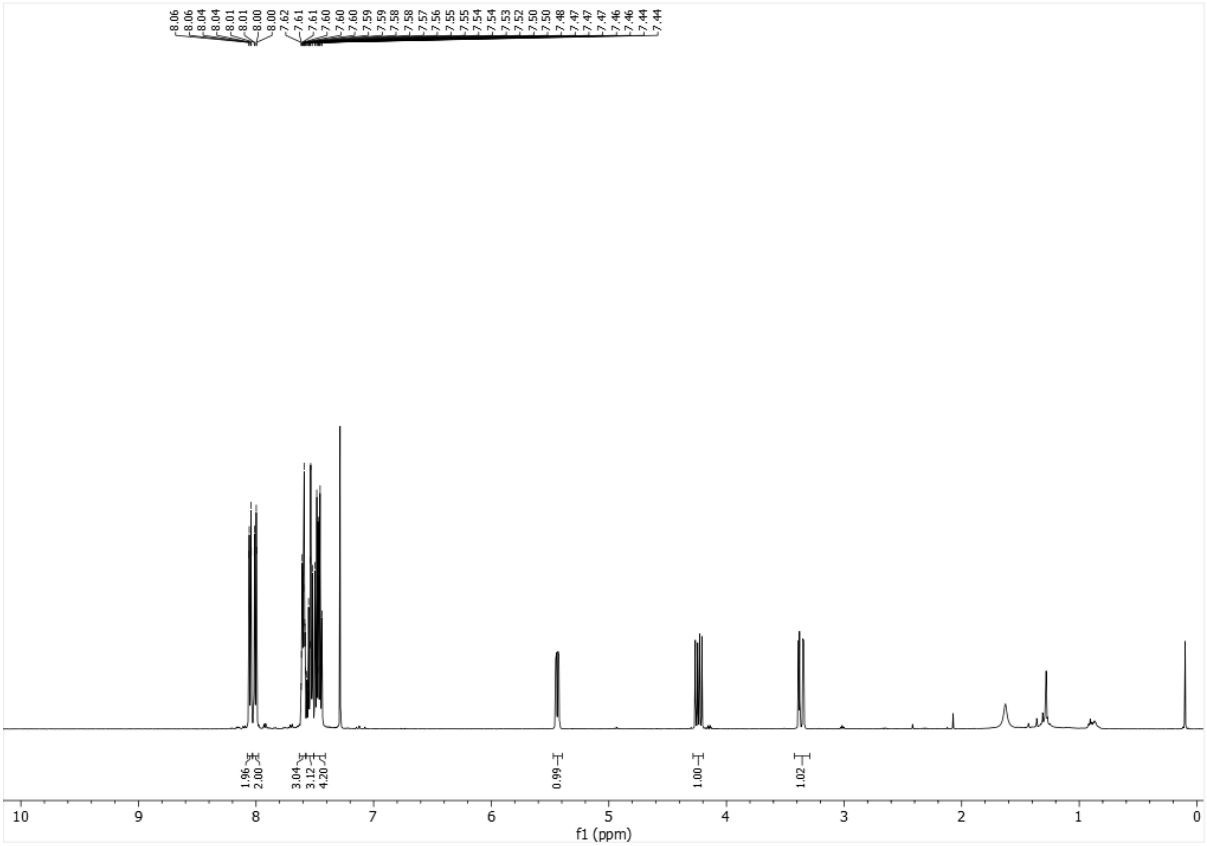


Figure **S18**. ^{1}H NMR spectrum of 1,4-diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione in $CDCl_3$.

9, ^{19}F, $CDCl_3$, 471 MHz

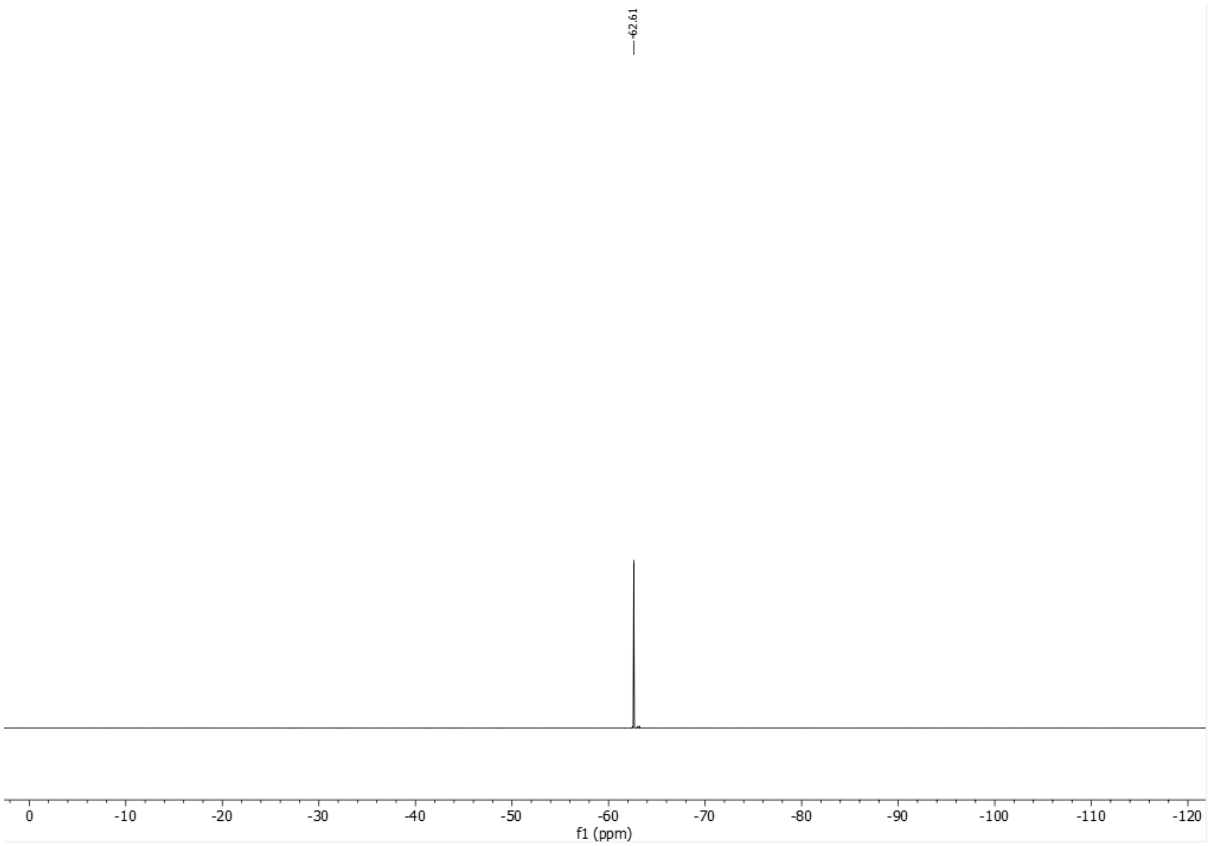


Figure **S19**. ^{19}F NMR spectrum of 1,4-diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione in $CDCl_3$.

9, ^{13}C, $CDCl_3$, 126 MHz

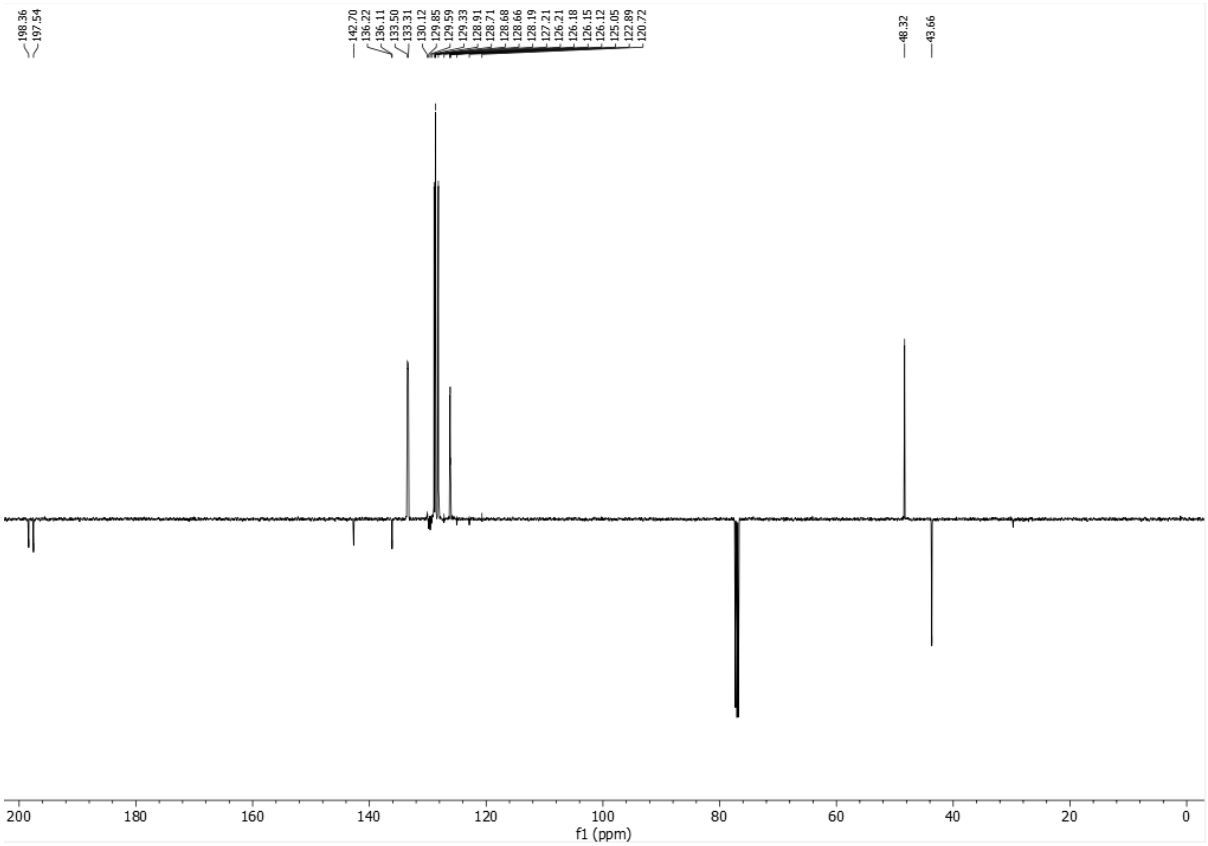
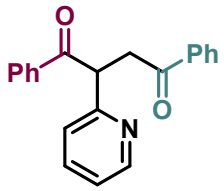


Figure **S20**. ^{13}C NMR spectrum of 1,4-diphenyl-2-(4-(trifluoromethyl)phenyl)butane-1,4-dione in $CDCl_3$.



10, ^{1}H, $CDCl_3$, 500 MHz

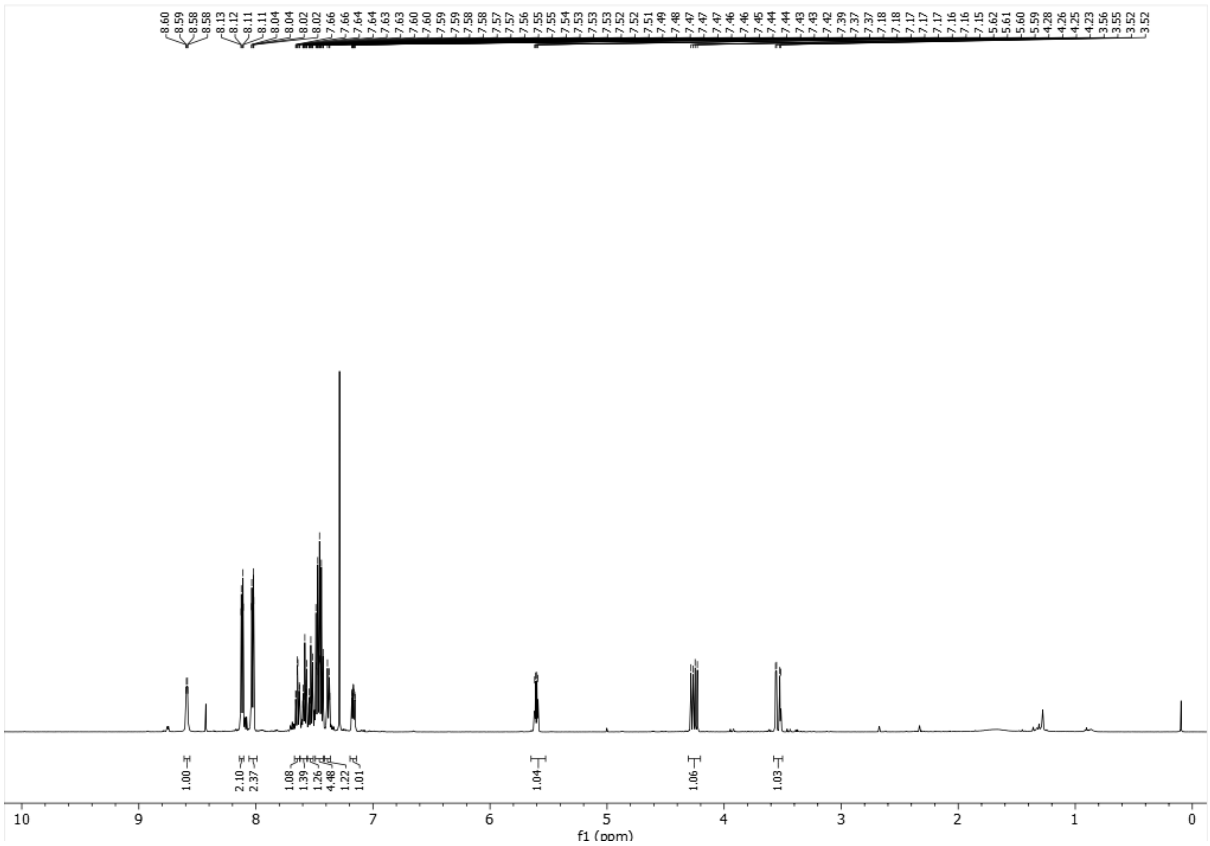


Figure **S21**. ^{1}H NMR spectrum of 1,4-diphenyl-2-(pyridin-2-yl)butane-1,4-dione in $CDCl_3$.

10, ^{13}C, $CDCl_3$, 126 MHz

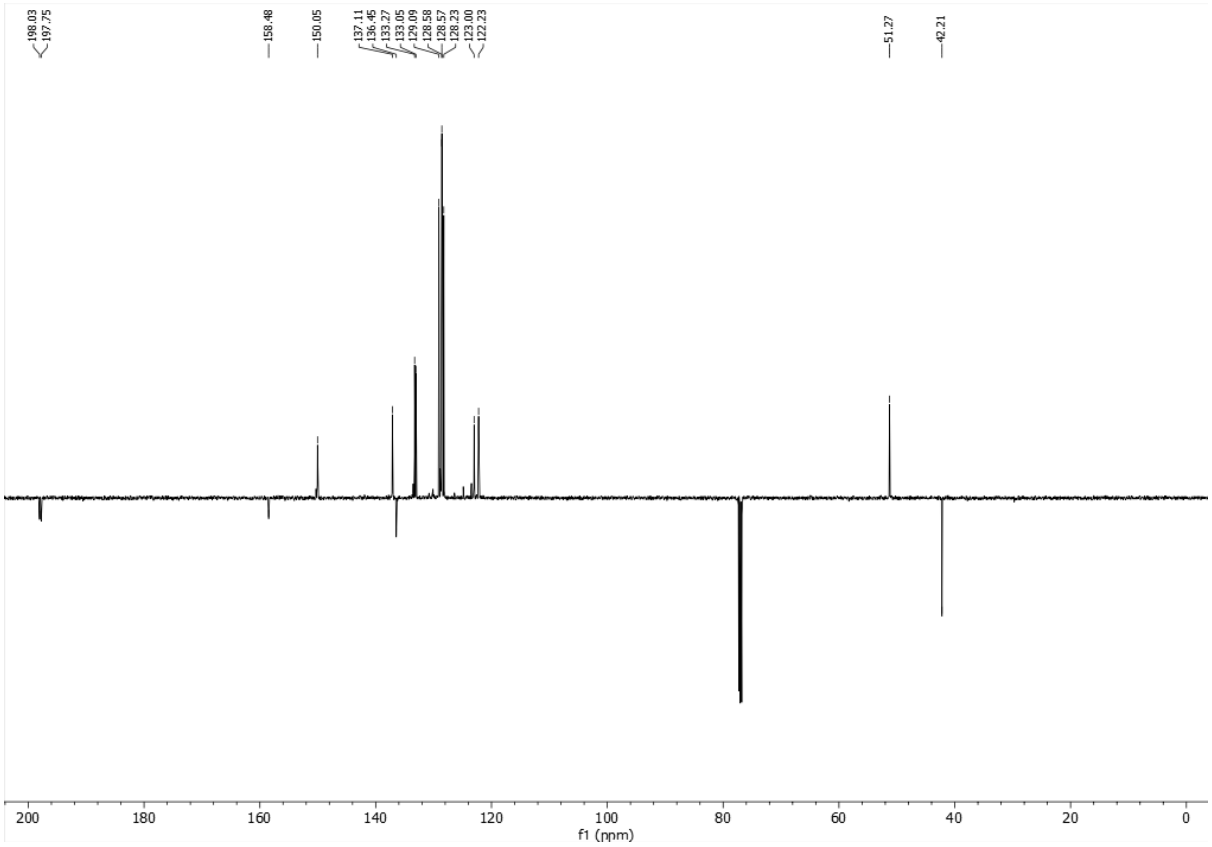
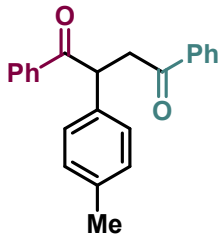


Figure **S22**. ^{13}C NMR spectrum of 1,4-diphenyl-2-(pyridin-2-yl)butane-1,4-dione in $CDCl_3$.



11, ^{1}H, $CDCl_3$, 500 MHz

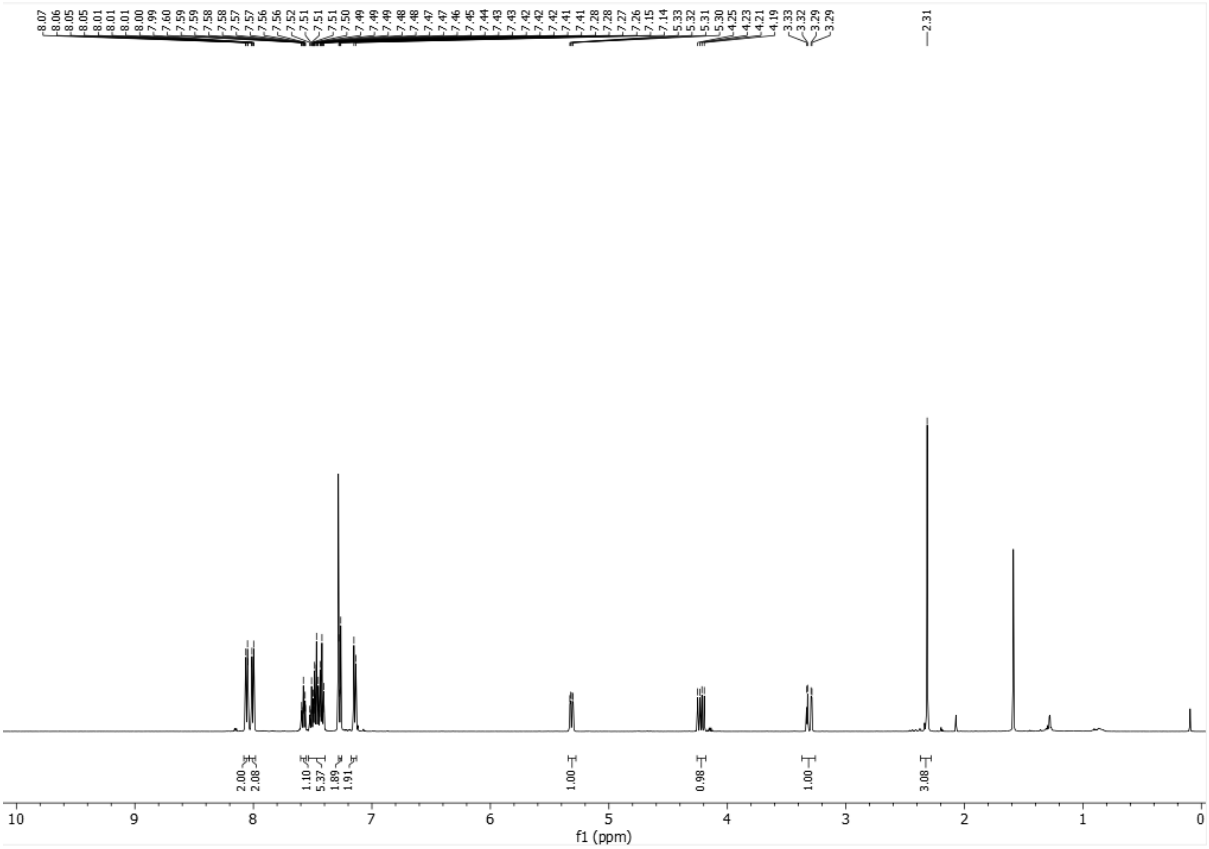


Figure **S23**. ^{1}H NMR spectrum of 1,4-diphenyl-2-(p-tolyl)butane-1,4-dione in $CDCl_3$.

11, ^{13}C, $CDCl_3$, 126 MHz

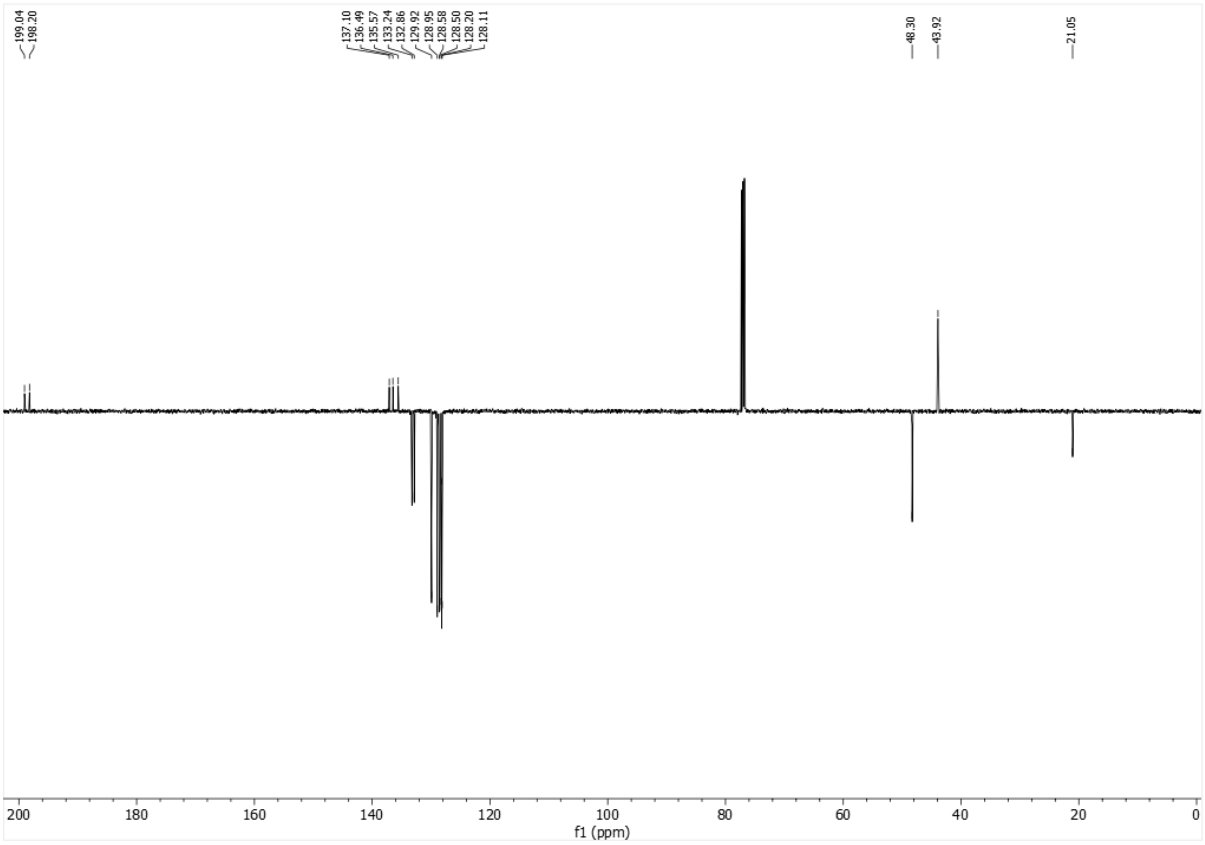
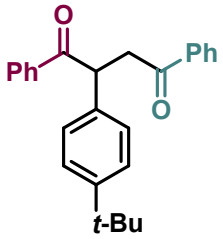


Figure **S24**. ^{13}C NMR spectrum of 1,4-diphenyl-2-(p-tolyl)butane-1,4-dione in $CDCl_3$.



12, ^{1}H, $CDCl_3$, 500 MHz

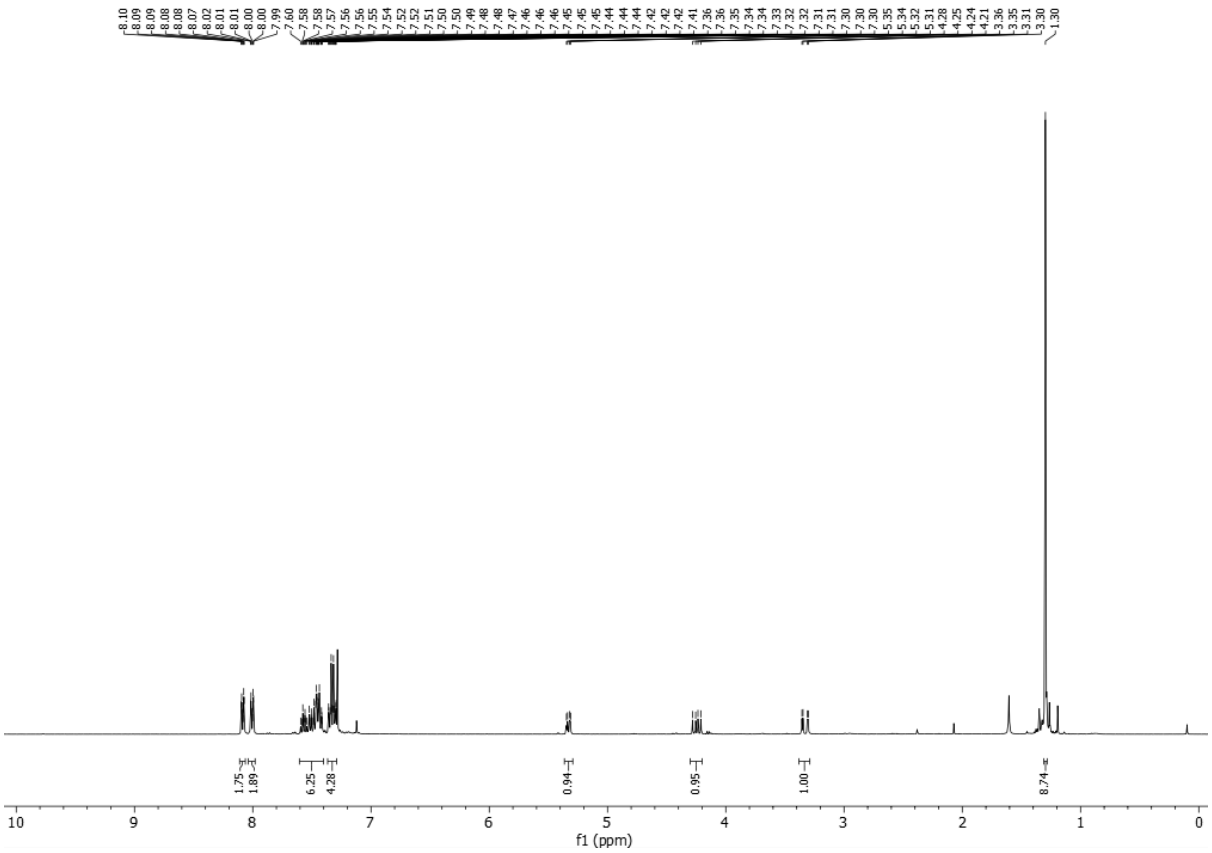


Figure **S25**. ^{1}H NMR spectrum of 2-(4-(tert-butyl)phenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.

12, ^{13}C, $CDCl_3$, 126 MHz

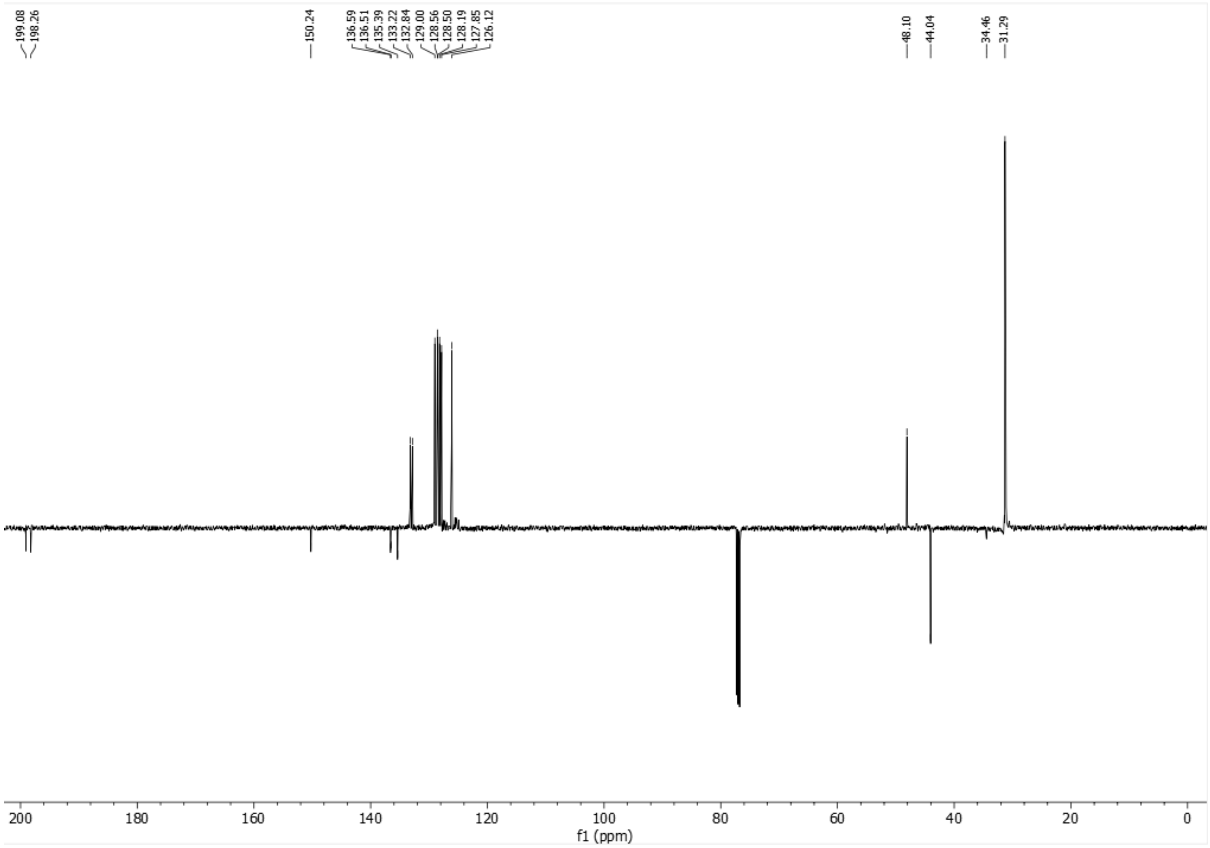
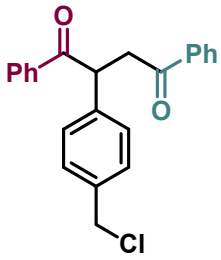


Figure **S26**. ^{13}C NMR spectrum of 2-(4-(tert-butyl)phenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.



13, ^{1}H, $CDCl_3$, 500 MHz

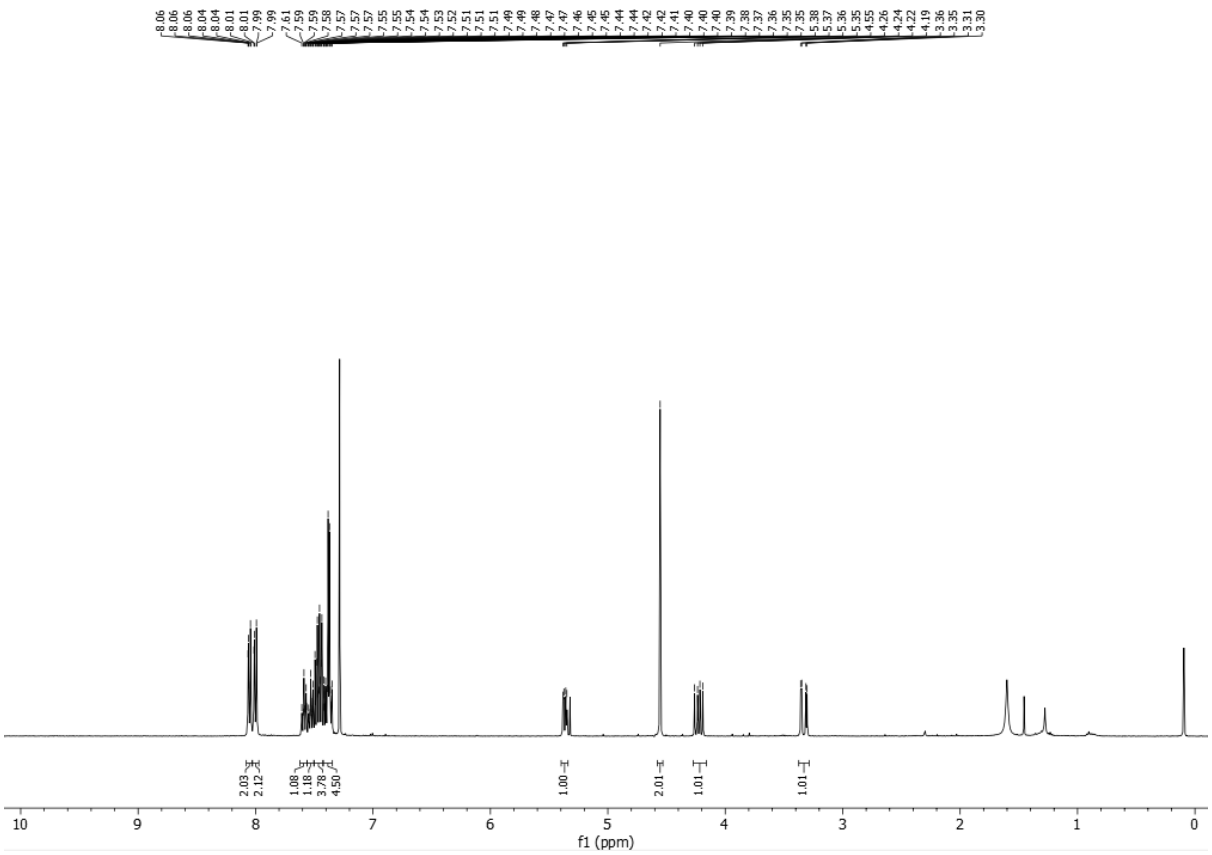


Figure **S27**. ^{1}H NMR spectrum of 2-(4-(chloromethyl)phenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.

13, ^{13}C, $CDCl_3$, 126 MHz

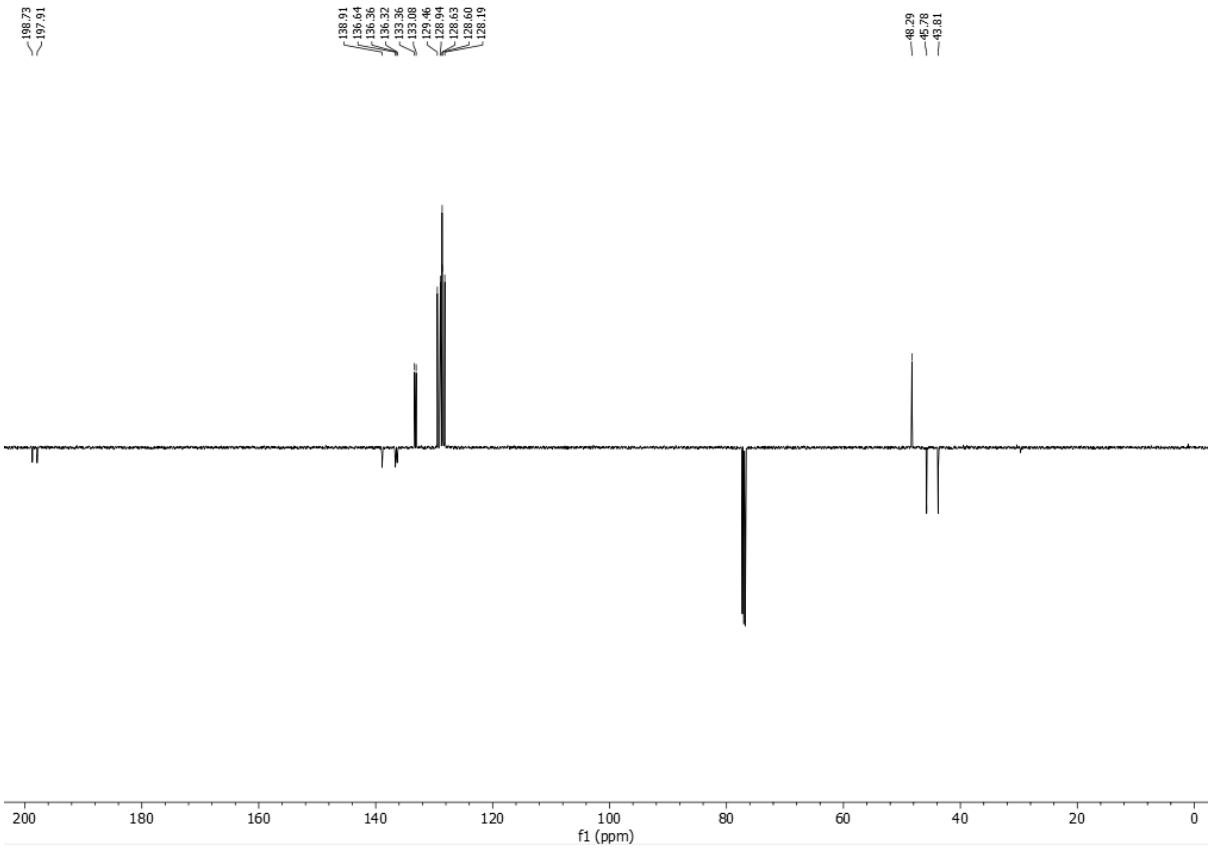
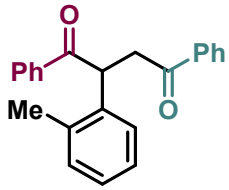


Figure **S28**. ^{13}C NMR spectrum of 2-(4-(chloromethyl)phenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.



14, ^{1}H, $CDCl_3$, 500 MHz

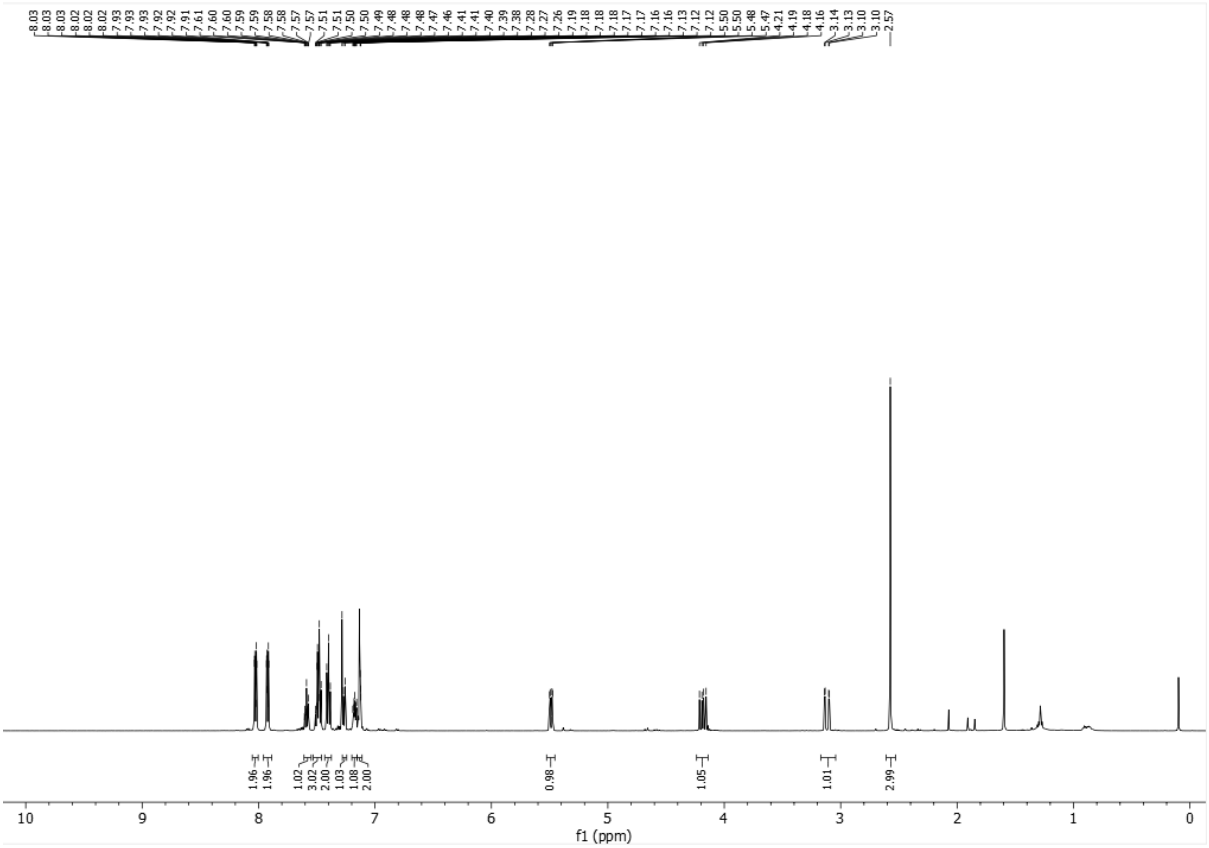


Figure **S29**. ^{1}H NMR spectrum of 1,4-diphenyl-2-(o-tolyl)butane-1,4-dione in $CDCl_3$.

14, ^{13}C, $CDCl_3$, 126 MHz

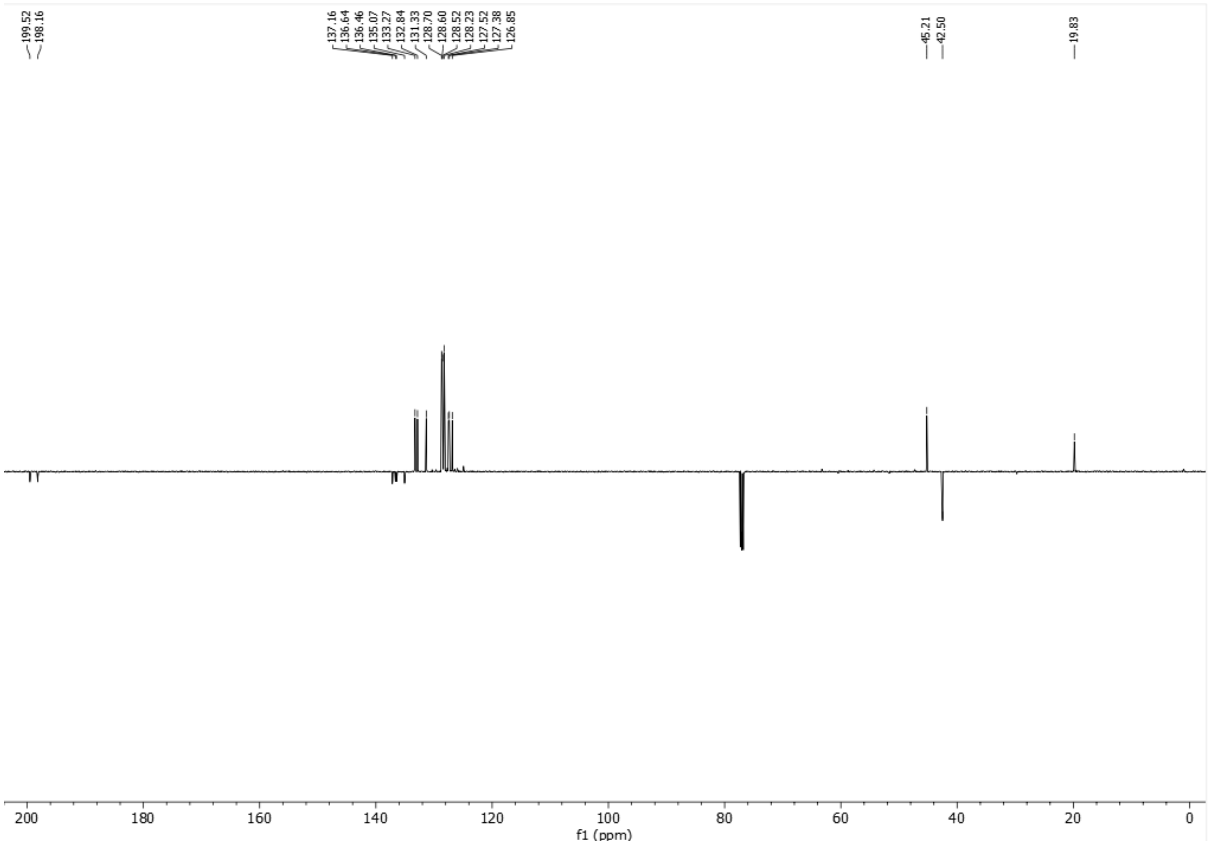
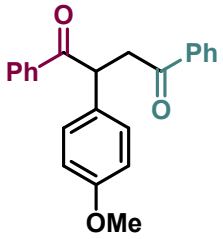


Figure **S30**. ^{13}C NMR spectrum of 1,4-diphenyl-2-(o-tolyl)butane-1,4-dione in $CDCl_3$.



15, ^{1}H, $CDCl_3$, 500 MHz

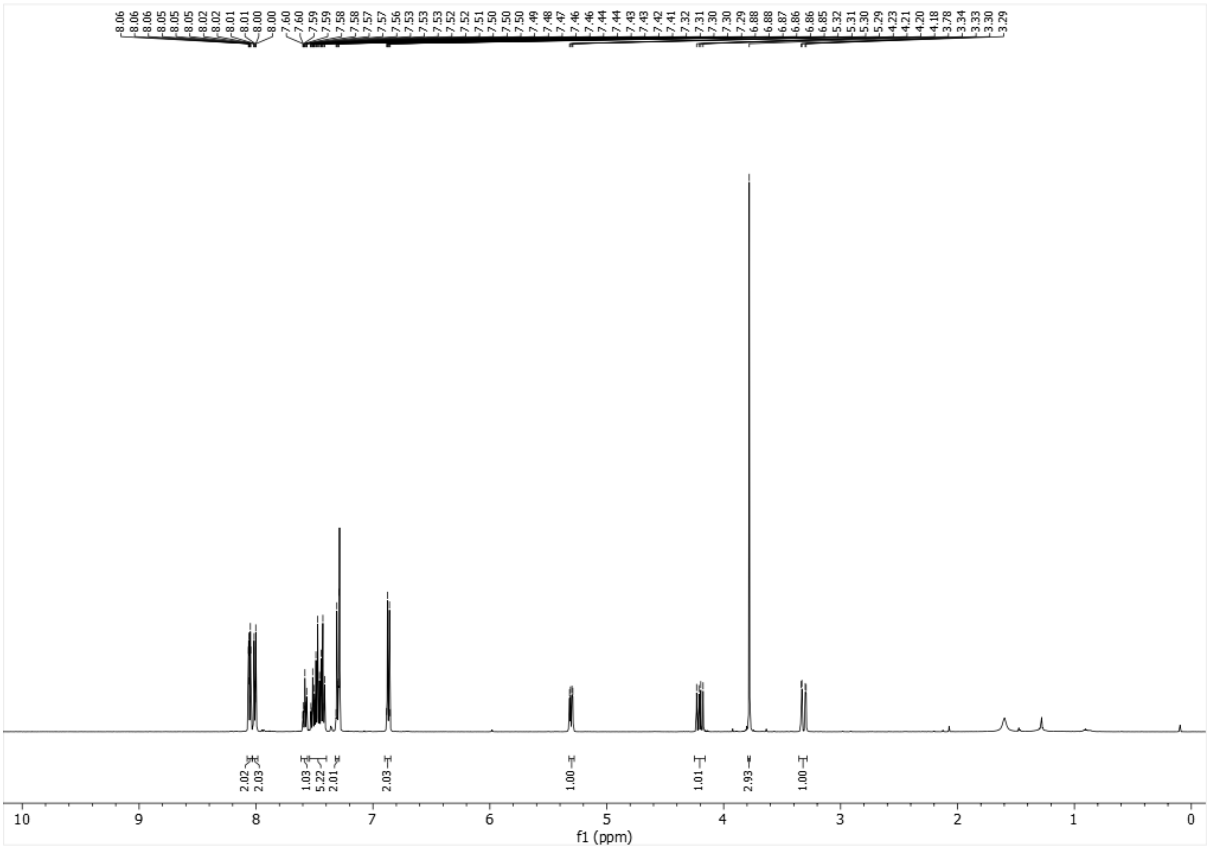


Figure **S31**. ^{1}H NMR spectrum of 2-(4-methoxyphenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.

15, ^{13}C, $CDCl_3$, 126 MHz

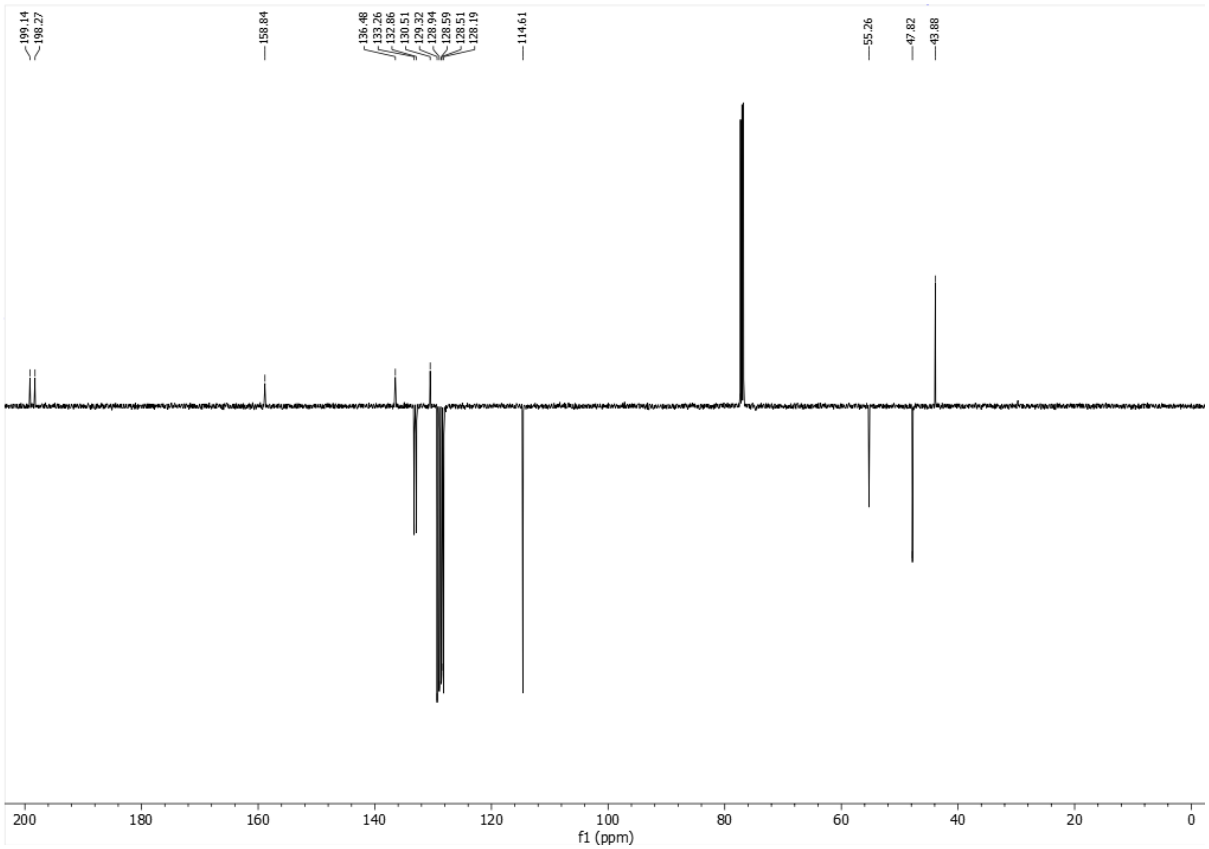
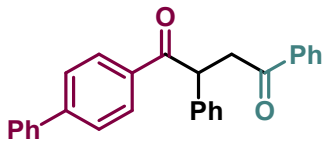


Figure **S32**. ^{13}C NMR spectrum of 2-(4-methoxyphenyl)-1,4-diphenylbutane-1,4-dione in $CDCl_3$.



16, ^{1}H, $CDCl_3$, 500 MHz

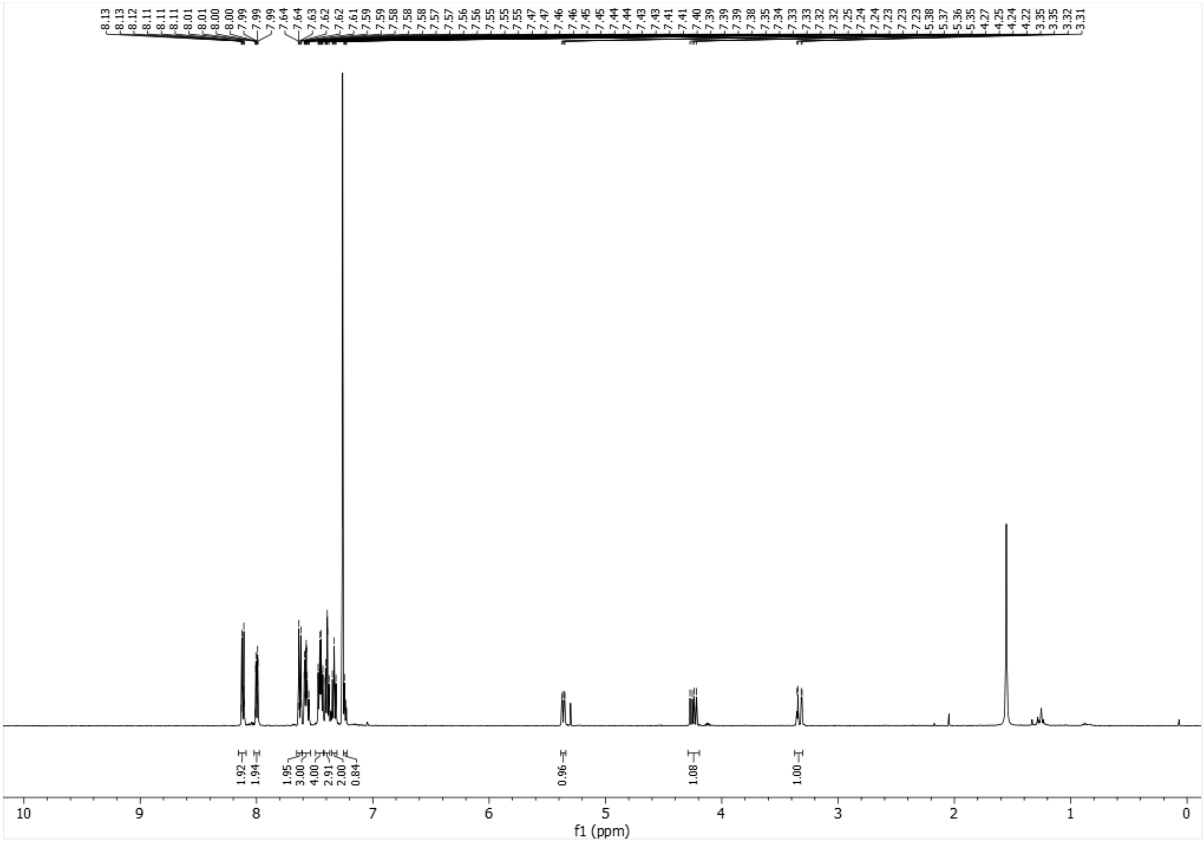


Figure **S33**. ^{1}H NMR spectrum of 1-([1,1'-biphenyl]-4-yl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

16, ^{13}C, $CDCl_3$, 126 MHz

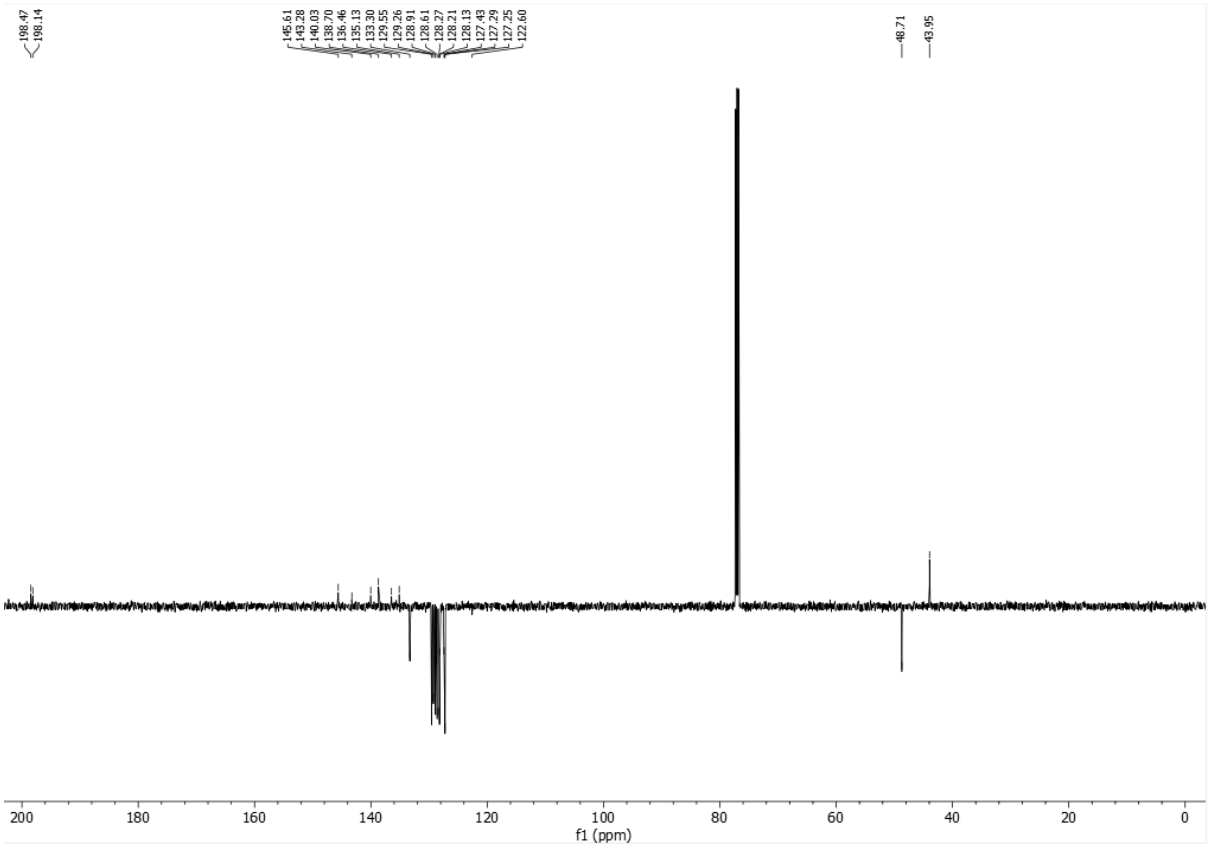
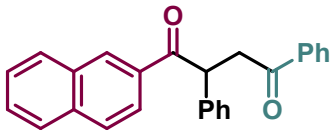
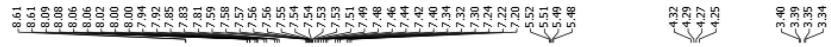


Figure **S34**. ^{13}C NMR spectrum of 1-([1,1'-biphenyl]-4-yl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



17, ^{1}H, $CDCl_3$, 500 MHz



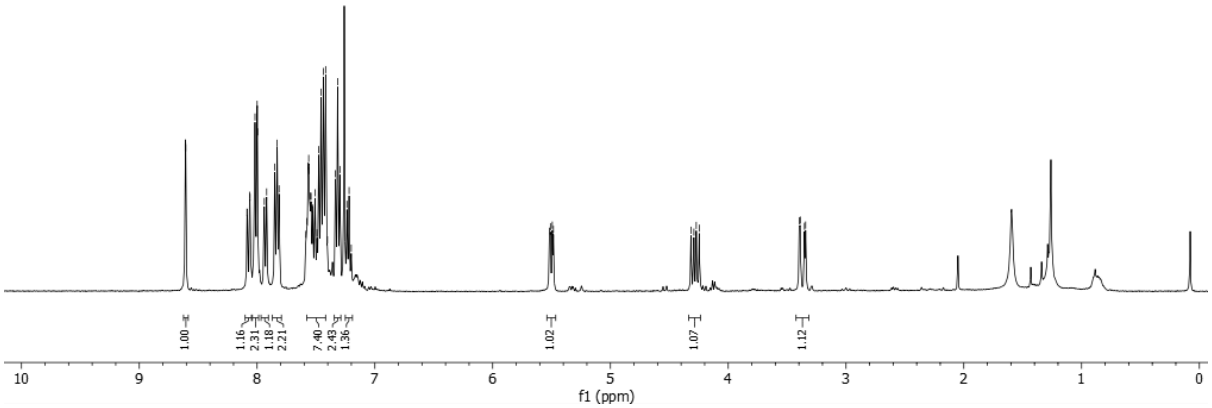


Figure **S35**. ^{1}H NMR spectrum of 1-(naphthalen-2-yl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

17, ^{13}C, $CDCl_3$, 126 MHz

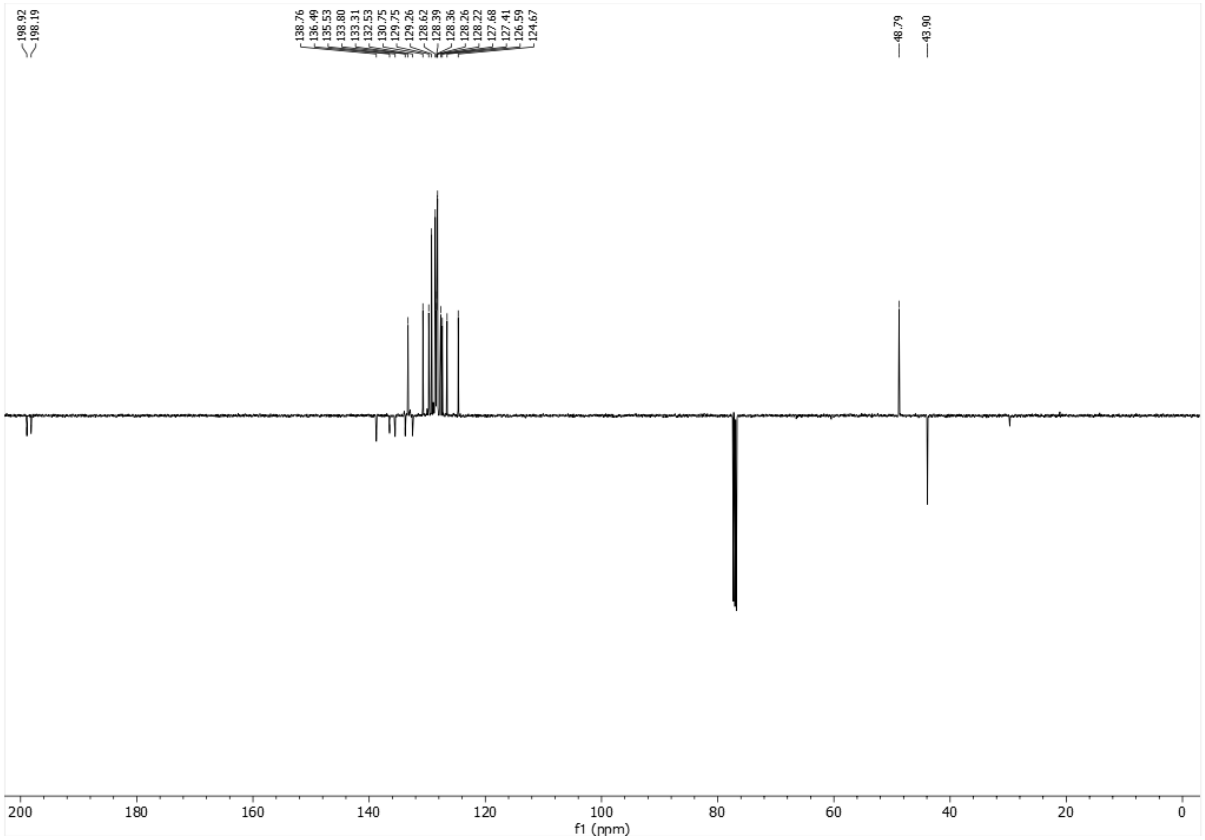
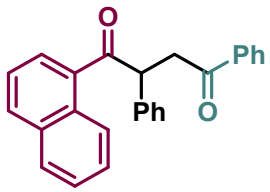


Figure **S36**. ^{13}C NMR spectrum of 1-(naphthalen-2-yl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



18, ^{1}H, $CDCl_3$, 500 MHz

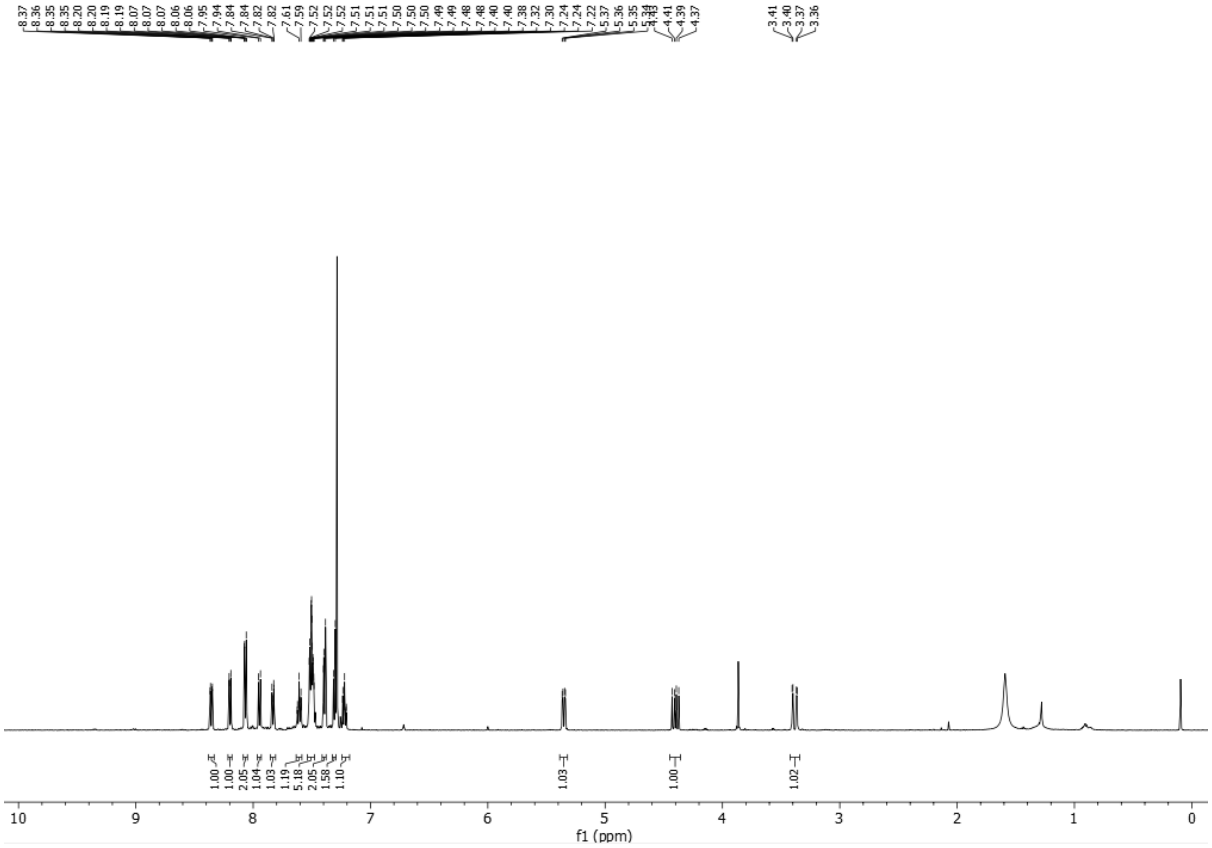


Figure **S37**. ^{1}H NMR spectrum of 1-(naphthalen-1-yl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

18, ^{13}C, $CDCl_3$, 126 MHz

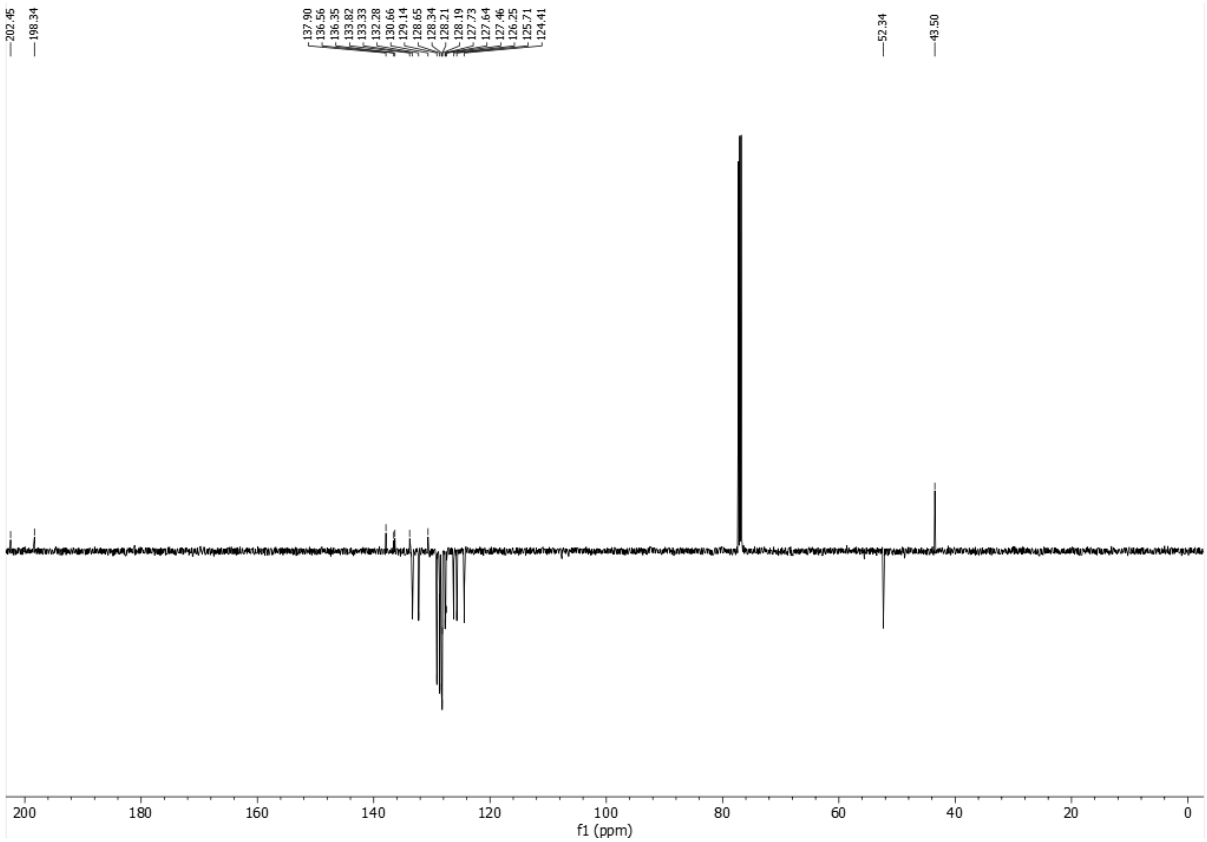
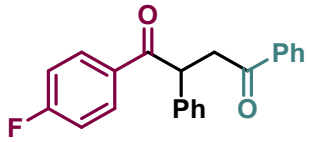


Figure **S38**. ^{13}C NMR spectrum of 1-(naphthalen-1-yl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



19, ^{1}H, $CDCl_3$, 500 MHz

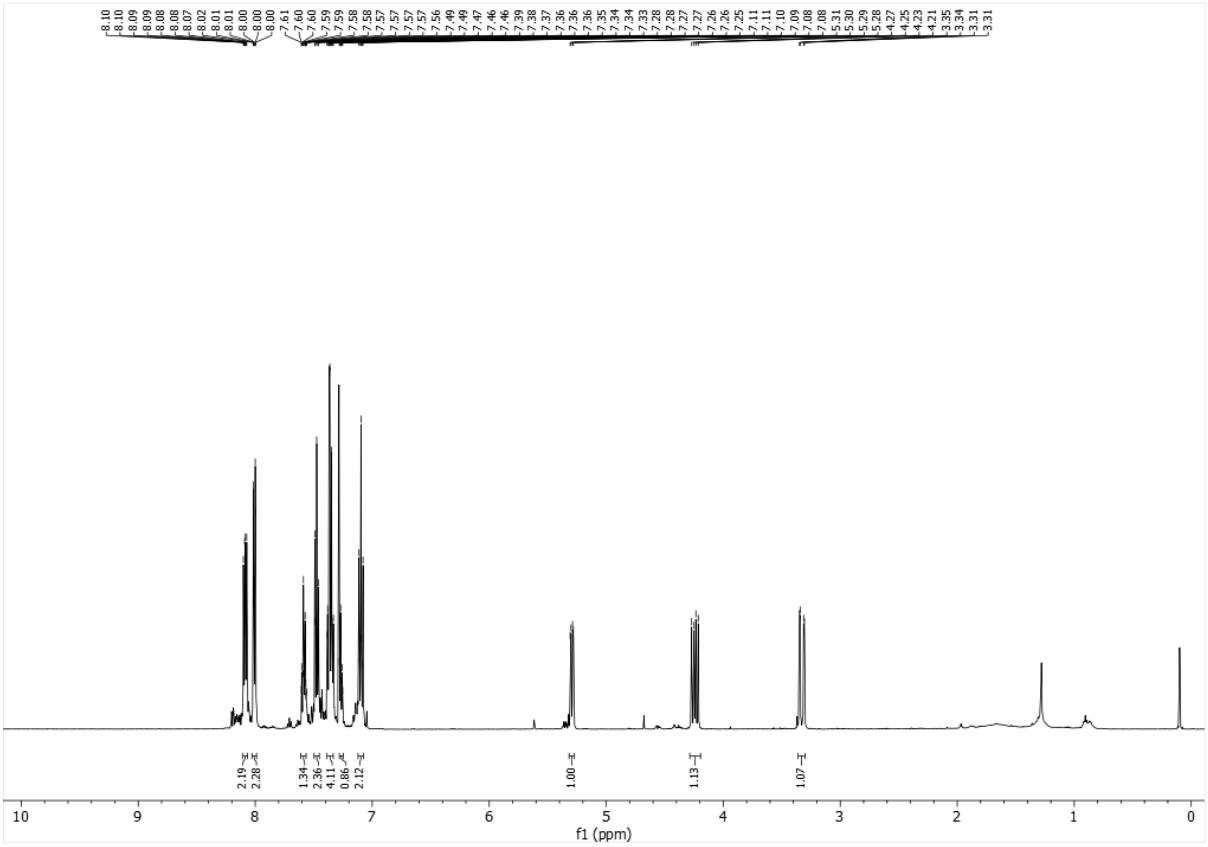


Figure **S39**. ^{1}H NMR spectrum of 1-(4-fluorophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

19, ^{19}F, $CDCl_3$, 471 MHz

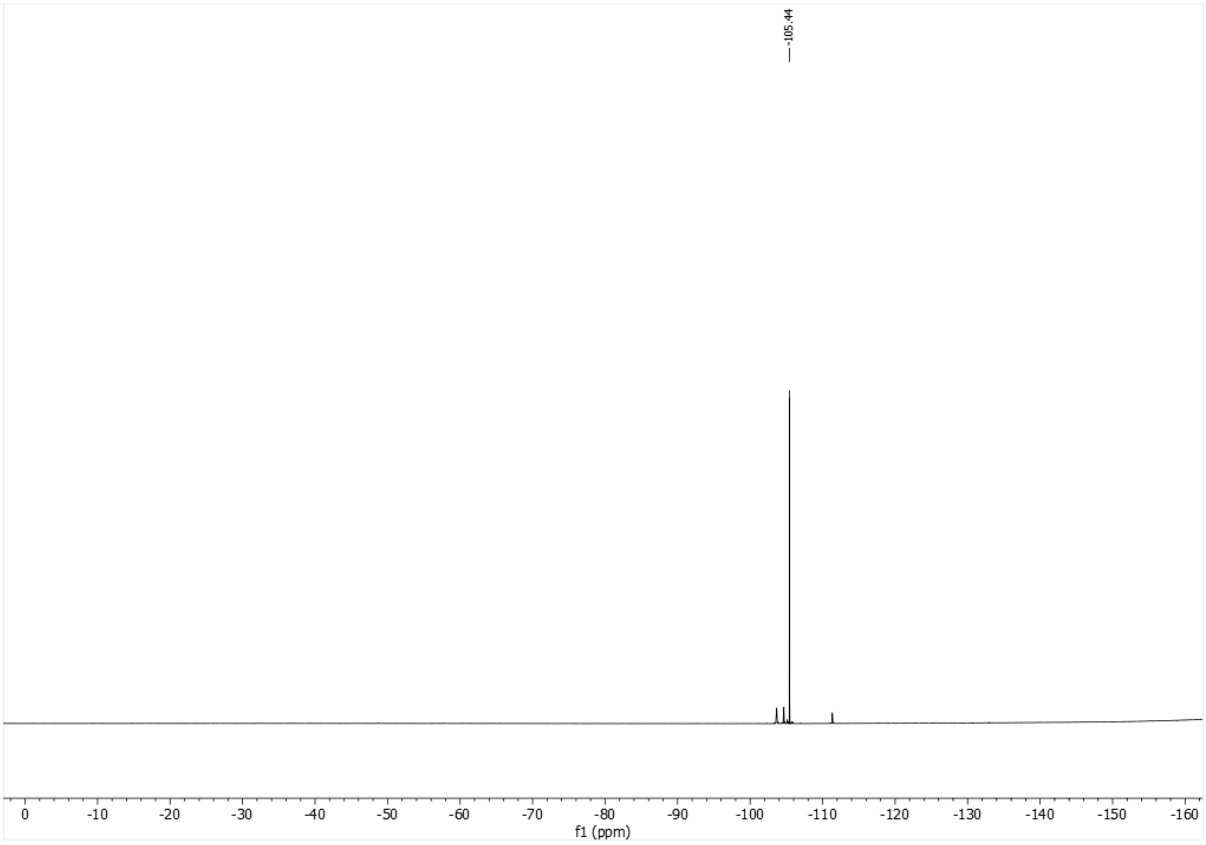


Figure **S40**. ^{19}F NMR spectrum of 1-(4-fluorophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

19, ^{13}C, $CDCl_3$, 126 MHz

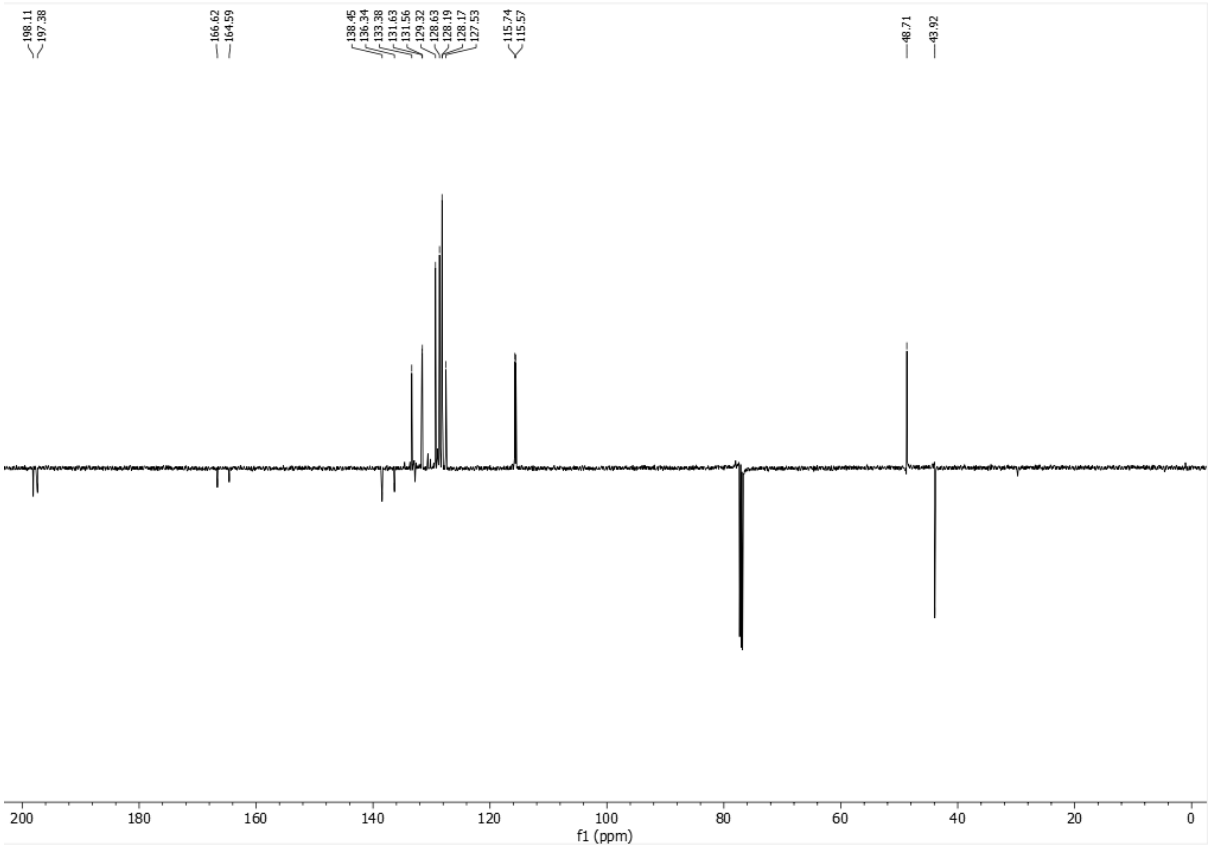
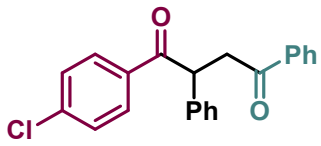


Figure **S41**. ^{13}C NMR spectrum of 1-(4-fluorophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



20, ^{1}H, $CDCl_3$, 500 MHz

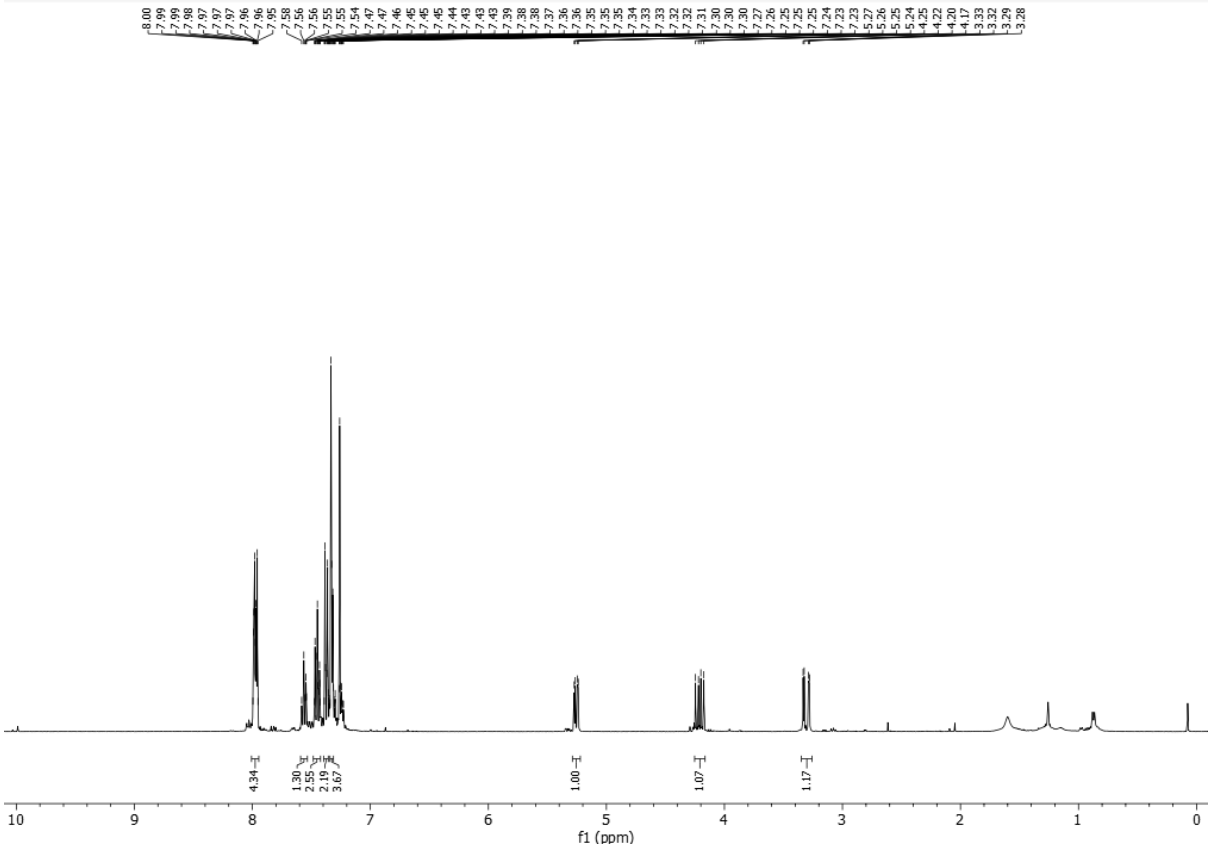


Figure **S42**. ^{1}H NMR spectrum of 1-(4-chlorophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

20, ^{13}C, $CDCl_3$, 126 MHz

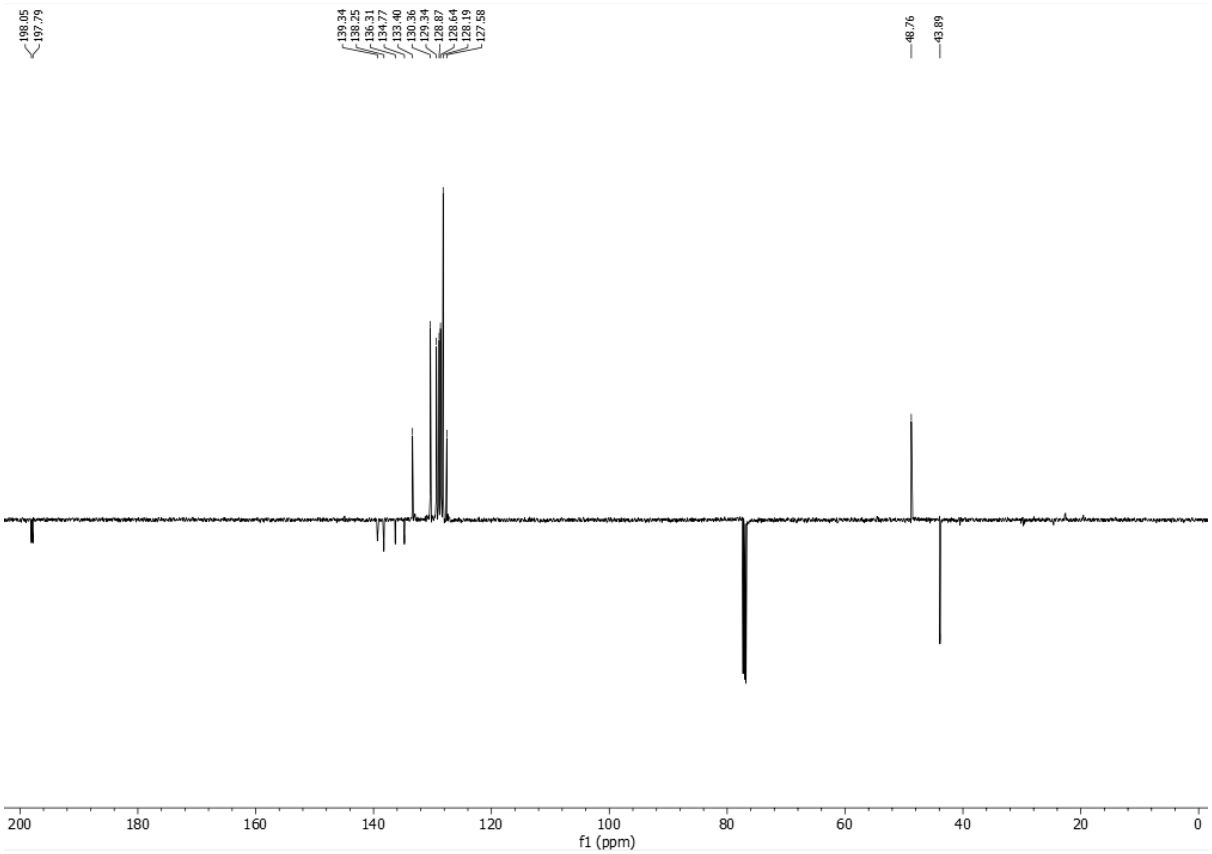
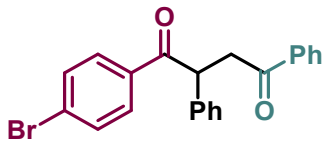


Figure **S43**. ^{13}C NMR spectrum of 1-(4-chlorophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



21, ^{1}H, $CDCl_3$, 500 MHz

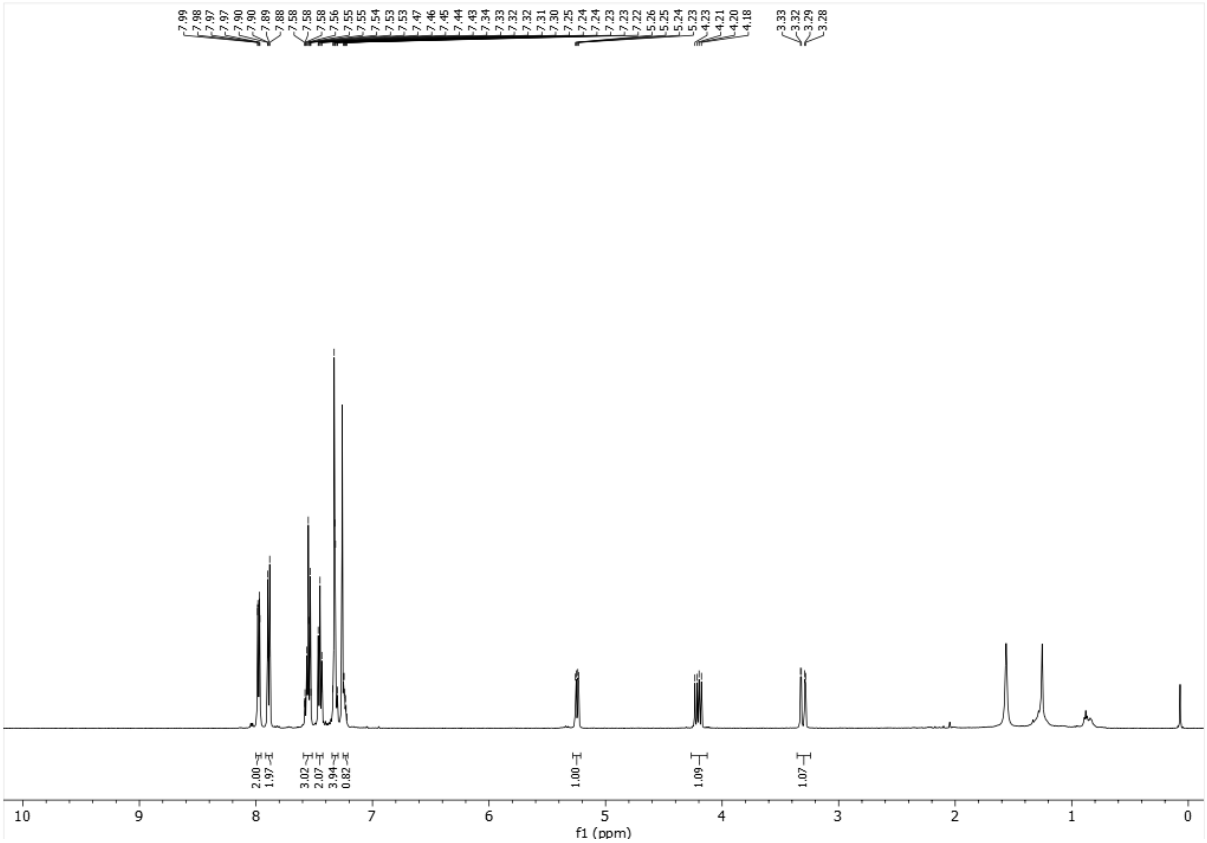


Figure **S44**. ^{1}H NMR spectrum of 1-(4-bromophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

21, ^{13}C, $CDCl_3$, 126 MHz

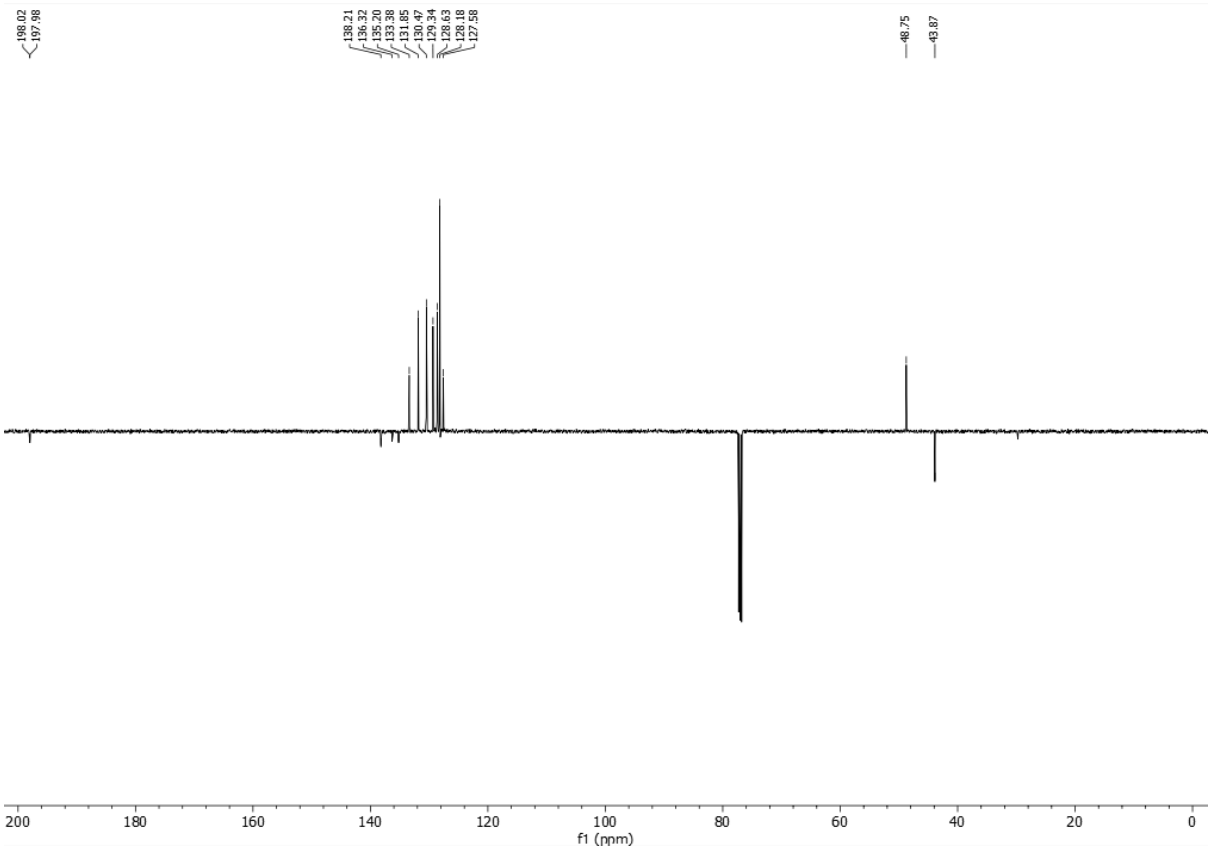
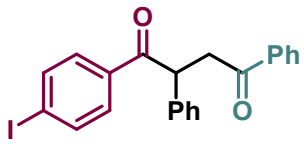


Figure **S45**. ^{13}C NMR spectrum of 1-(4-bromophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



22, ^{1}H, $CDCl_3$, 500 MHz

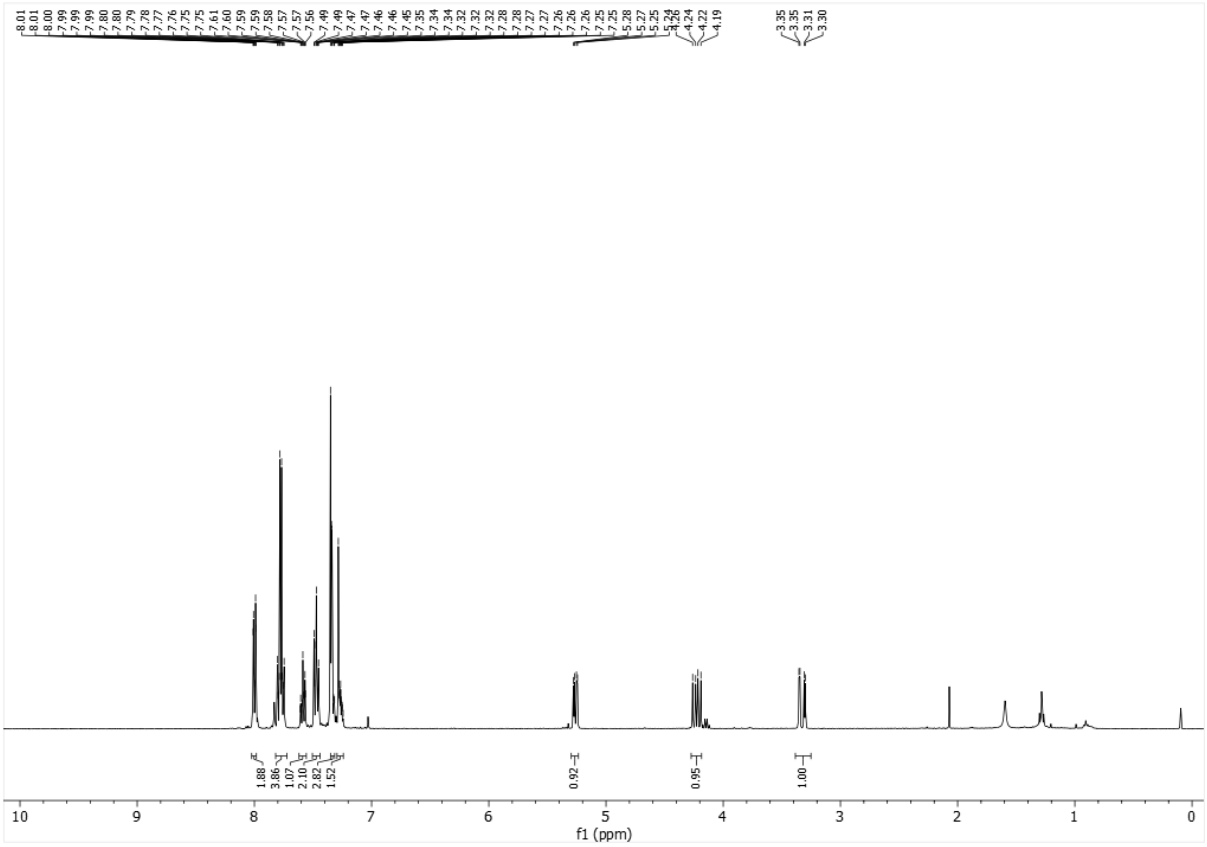


Figure **S46**. ^{1}H NMR spectrum of 1-(4-iodophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

22, ^{13}C, $CDCl_3$, 126 MHz

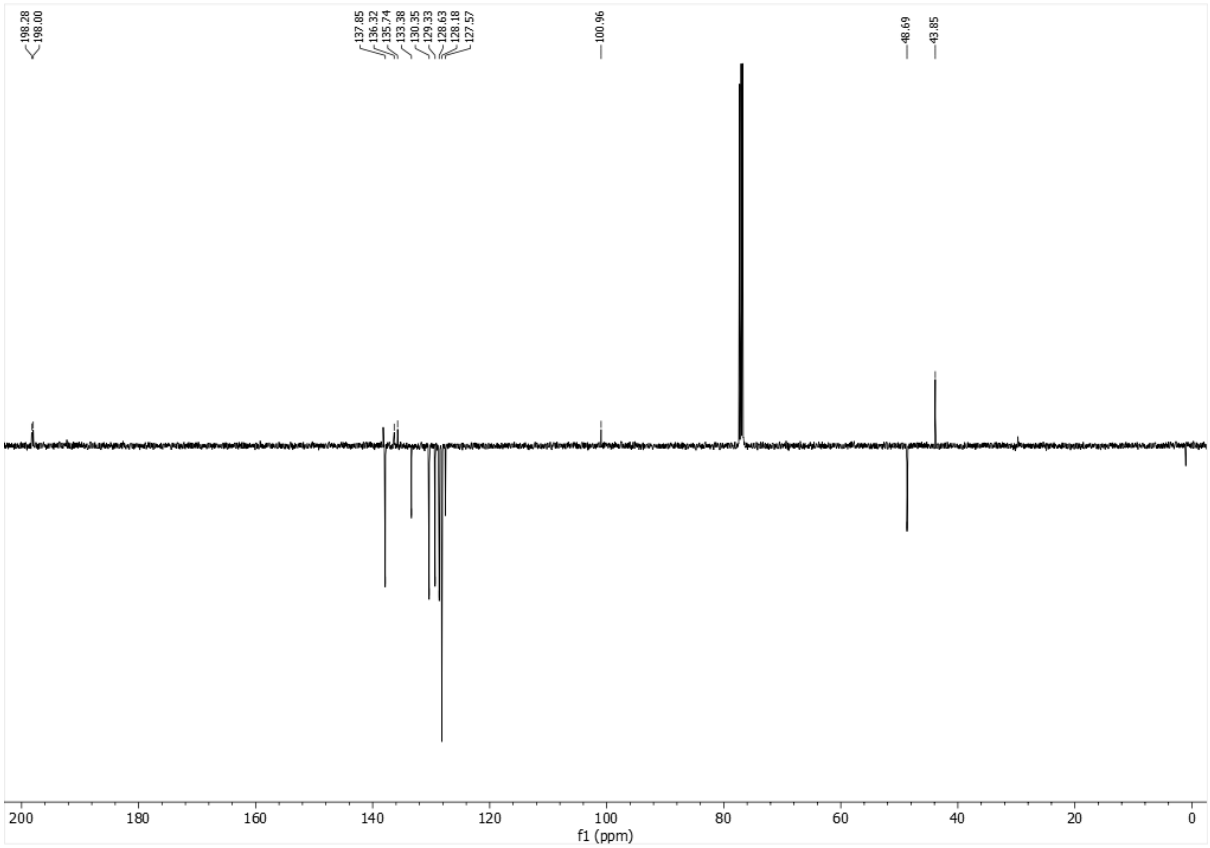
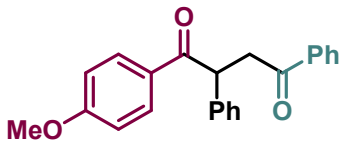


Figure **S47**. ^{13}C NMR spectrum of 1-(4-iodophenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



23, ^{1}H, $CDCl_3$, 500 MHz

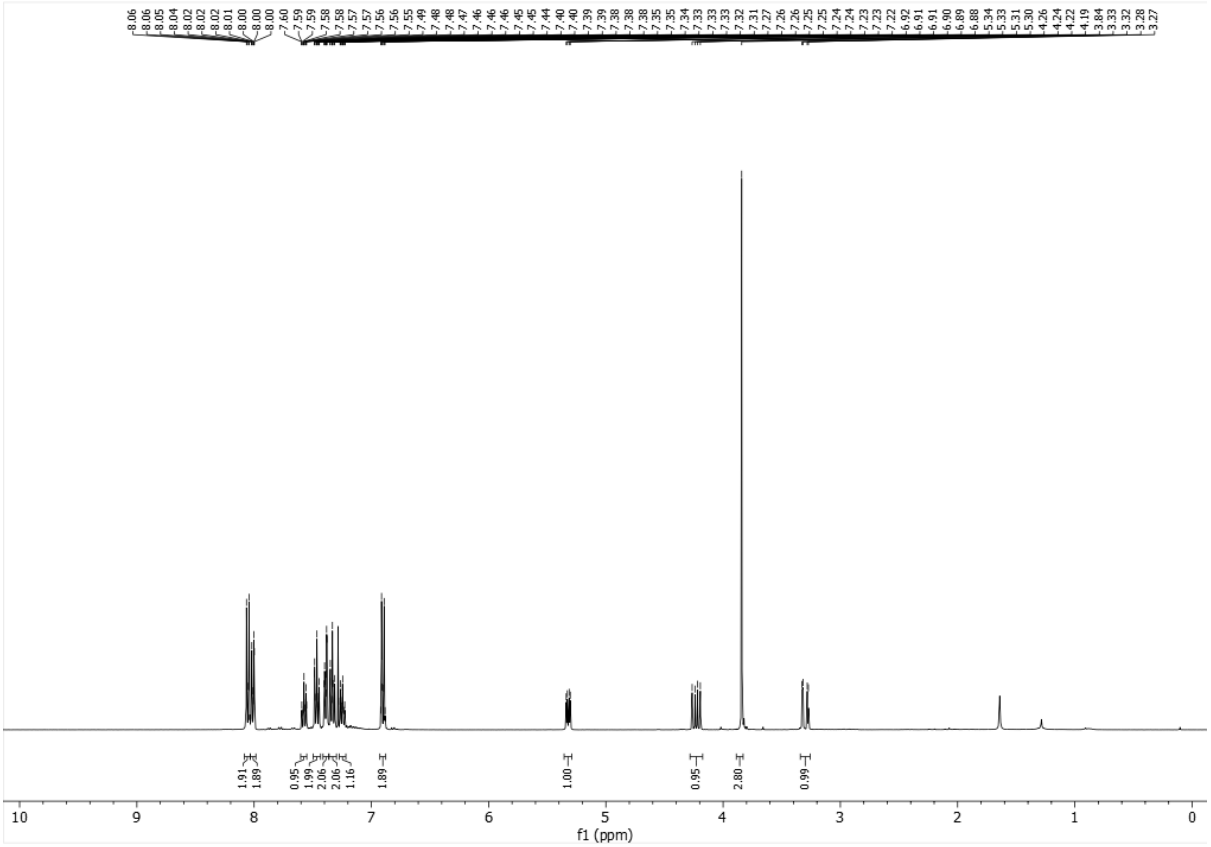


Figure **S48**. ^{1}H NMR spectrum of 1-(4-methoxyphenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

23, ^{13}C, $CDCl_3$, 126 MHz

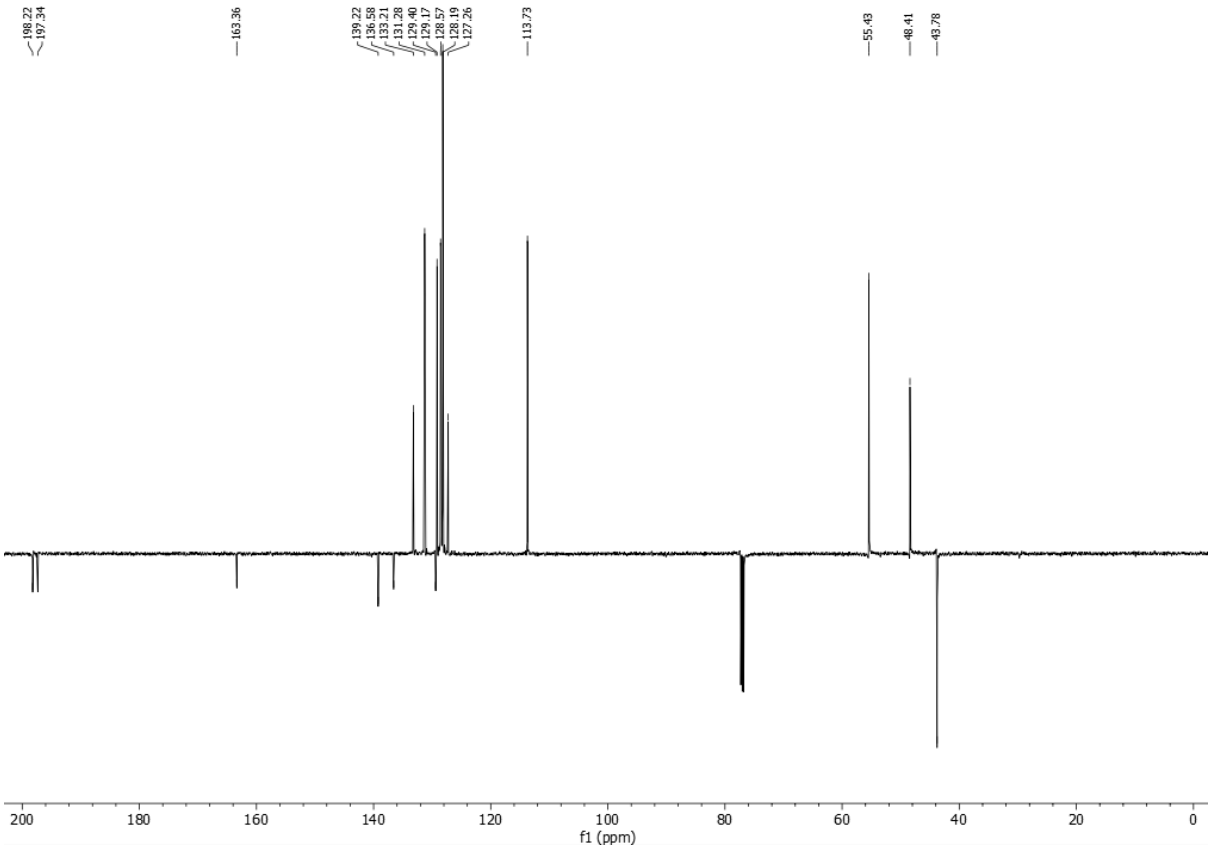
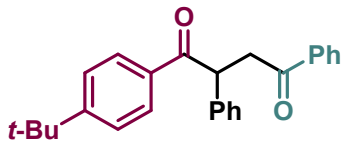


Figure **S49**. ^{13}C NMR spectrum of 1-(4-methoxyphenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



24, ^{1}H, $CDCl_3$, 500 MHz

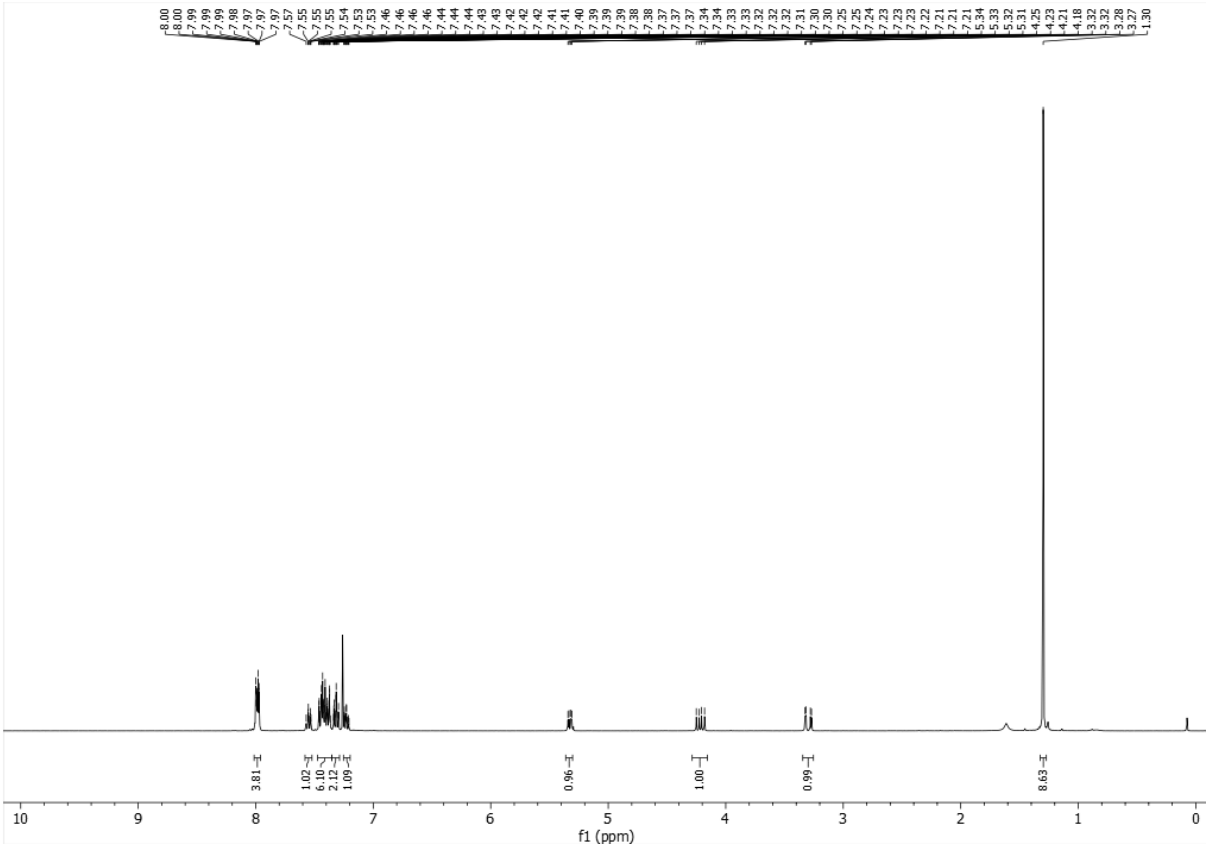


Figure **S50**. ^{1}H NMR spectrum of 1-(4-(tert-butyl)phenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.

24, ^{13}C, $CDCl_3$, 126 MHz

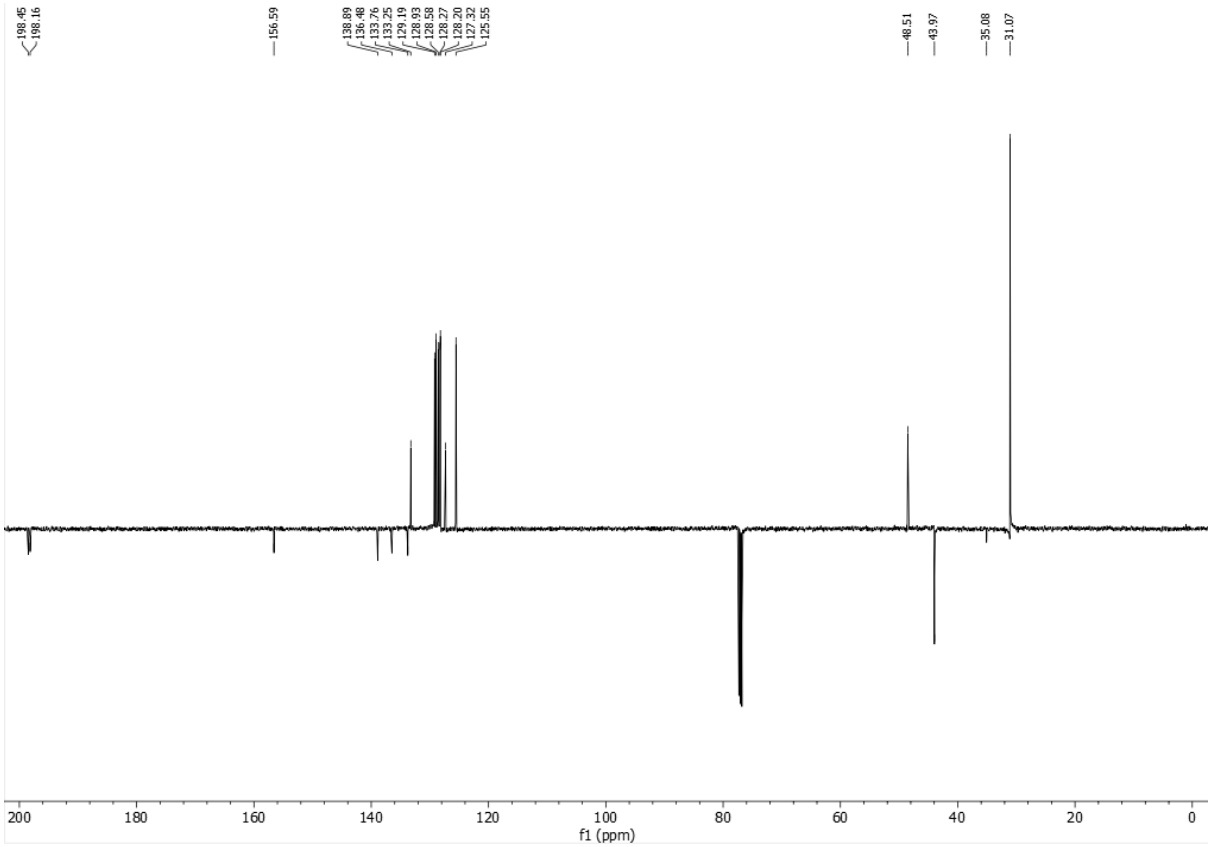
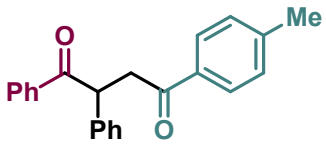


Figure **S51**. ^{13}C NMR spectrum of 1-(4-(tert-butyl)phenyl)-2,4-diphenylbutane-1,4-dione in $CDCl_3$.



25, ^{1}H, $CDCl_3$, 500 MHz

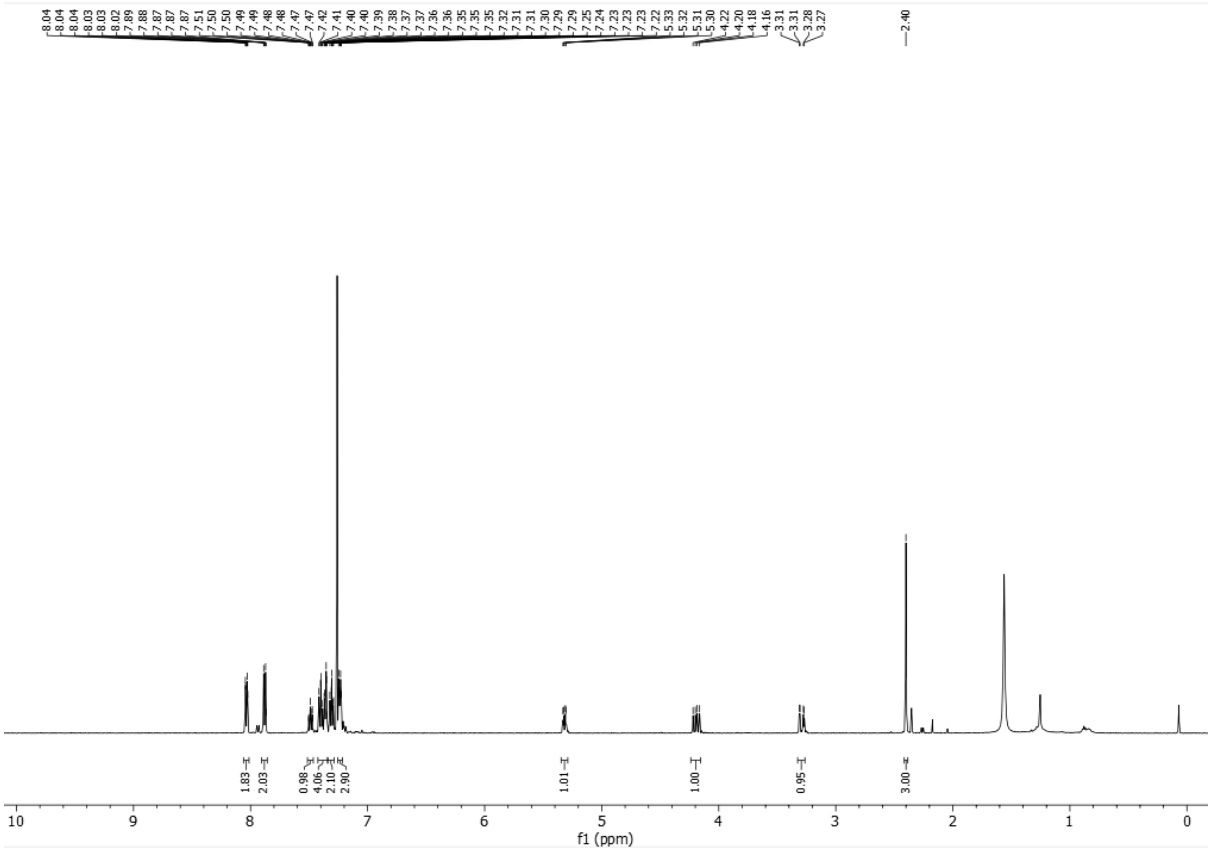


Figure **S52**. ^{1}H NMR spectrum of 1,2-diphenyl-4-(p-tolyl)butane-1,4-dione in $CDCl_3$.

25, ^{13}C, $CDCl_3$, 126 MHz

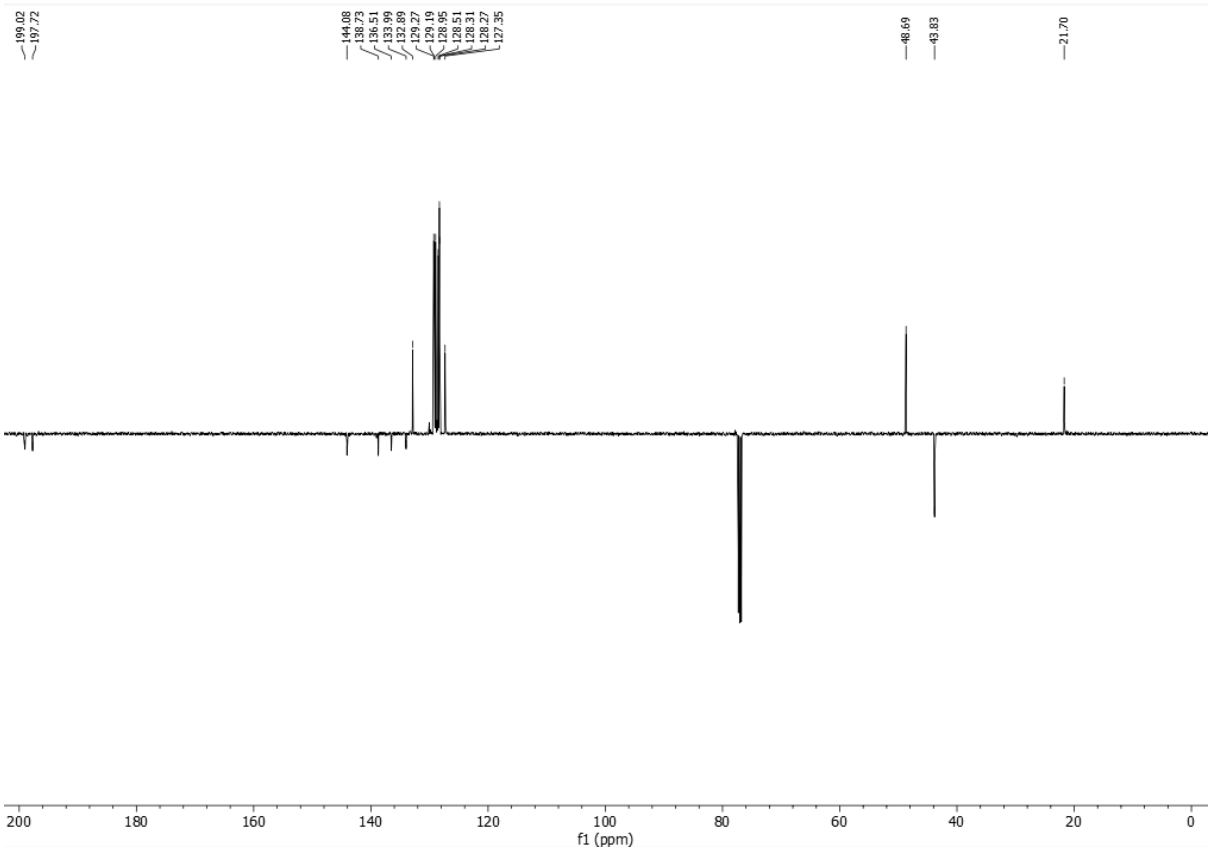
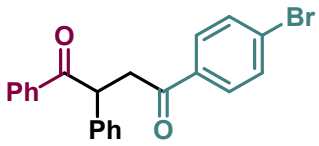


Figure **S53**. ^{13}C NMR spectrum of 1,2-diphenyl-4-(p-tolyl)butane-1,4-dione in $CDCl_3$.



26, ^{1}H, $CDCl_3$, 500 MHz

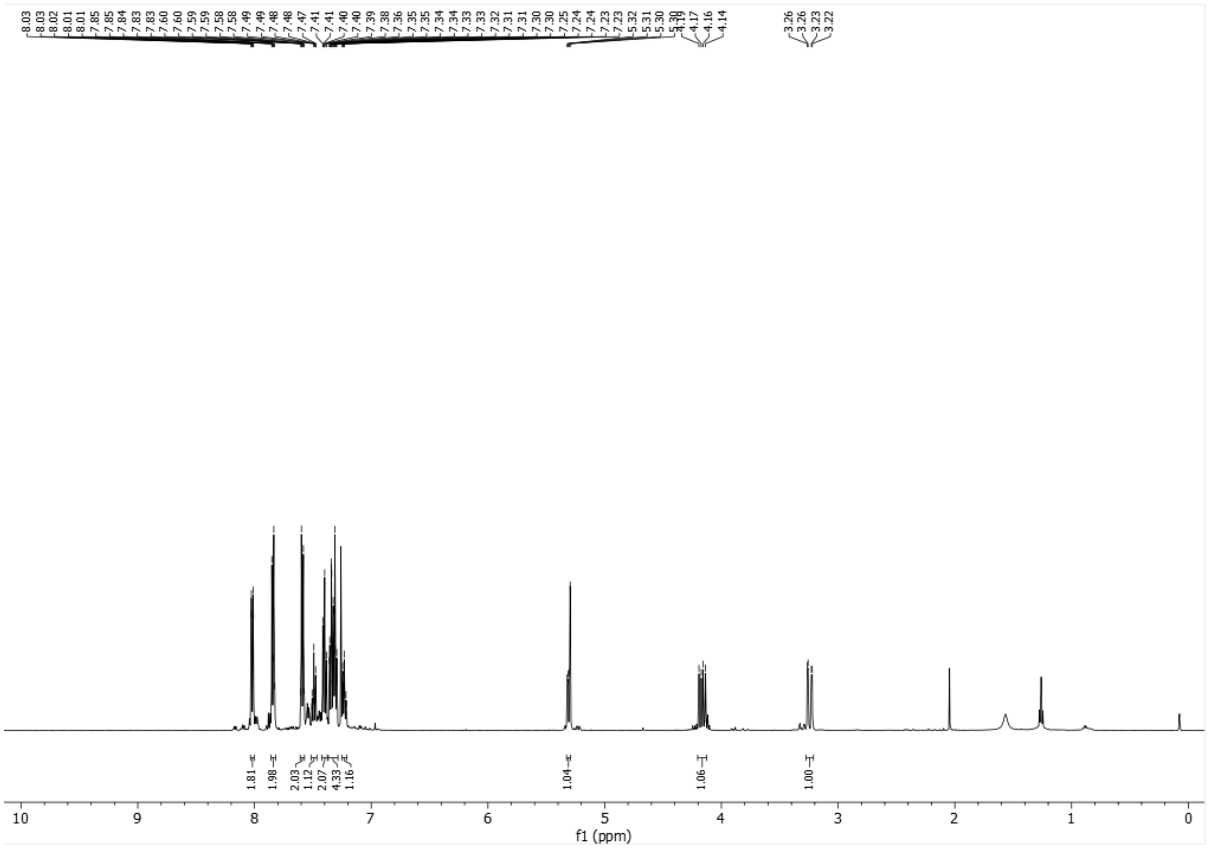


Figure **S54**. ^{1}H NMR spectrum of 4-(4-bromophenyl)-1,2-diphenylbutane-1,4-dione in $CDCl_3$.

26, ^{13}C, $CDCl_3$, 126 MHz

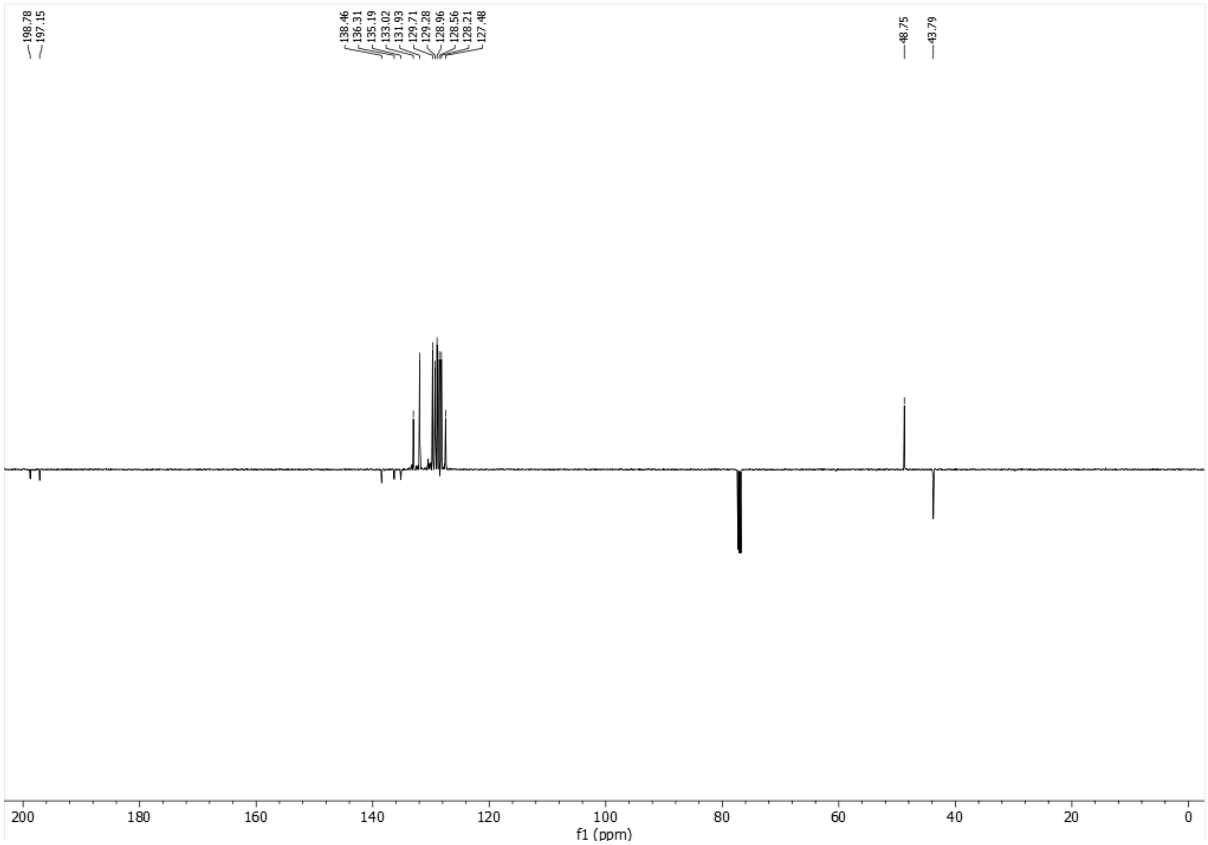
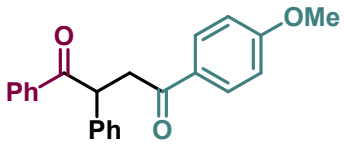


Figure **S55**. ^{13}C NMR spectrum of 4-(4-bromophenyl)-1,2-diphenylbutane-1,4-dione in $CDCl_3$.



27, ^{1}H, $CDCl_3$, 500 MHz

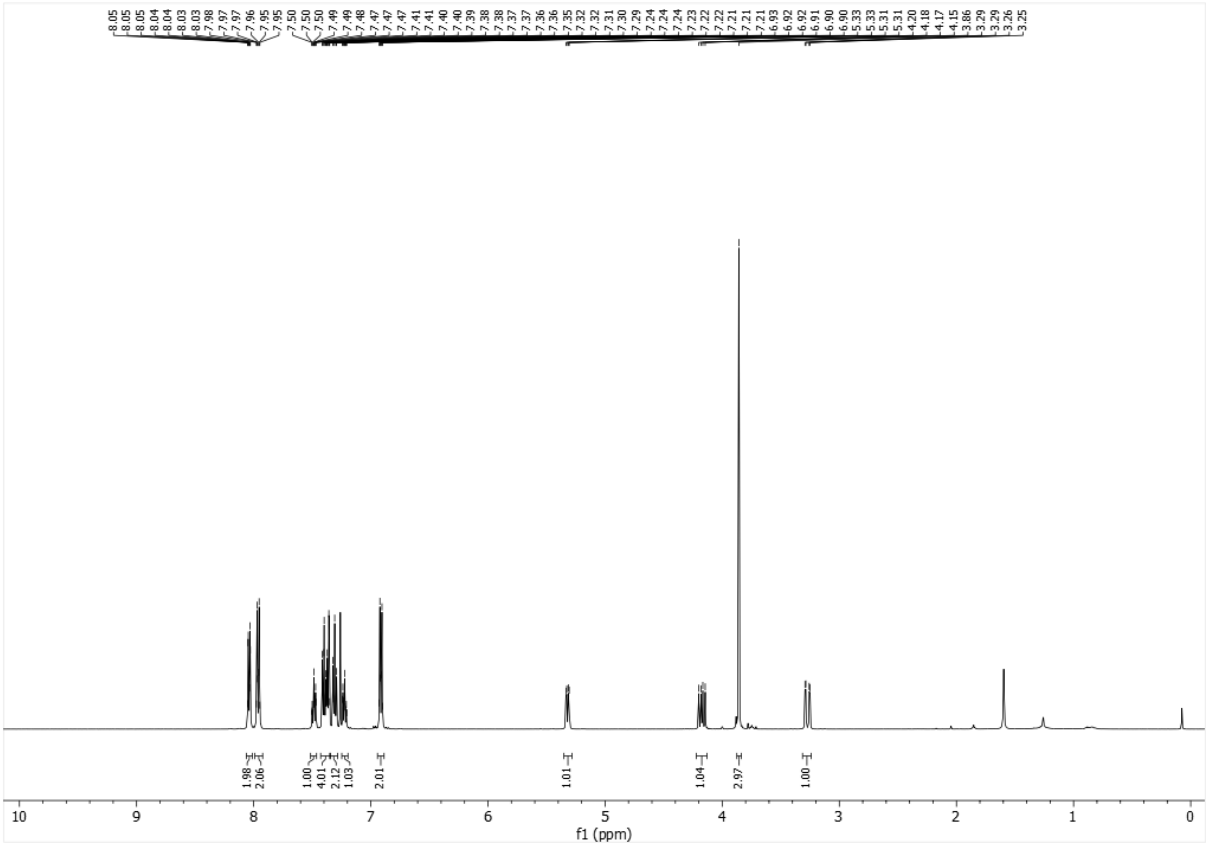


Figure **S56**. ^{1}H NMR spectrum of 4-(4-methoxyphenyl)-1,2-diphenylbutane-1,4-dione in $CDCl_3$.

27, ^{13}C, $CDCl_3$, 126 MHz

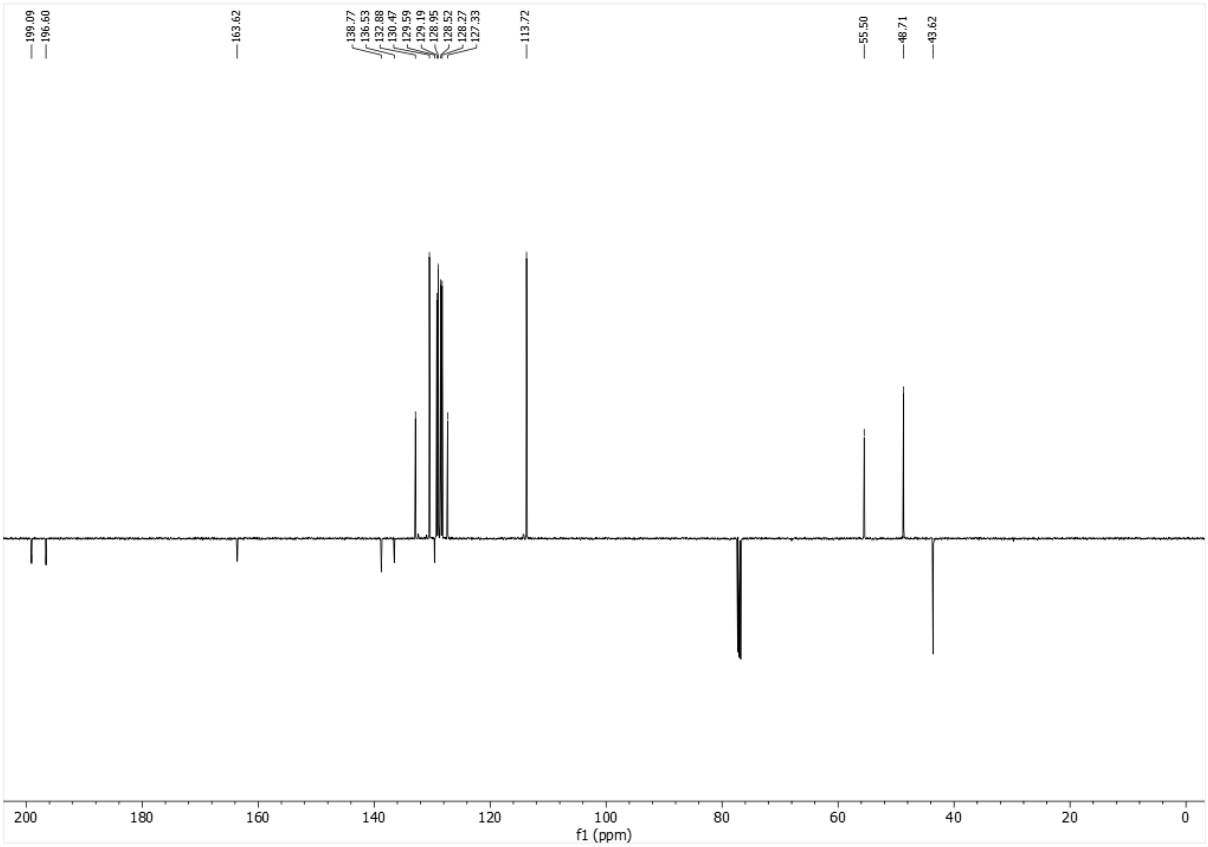
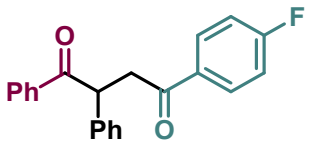


Figure **S57**. ^{13}C NMR spectrum of 4-(4-methoxyphenyl)-1,2-diphenylbutane-1,4-dione in $CDCl_3$.



28, ^{1}H, $CDCl_3$, 500 MHz

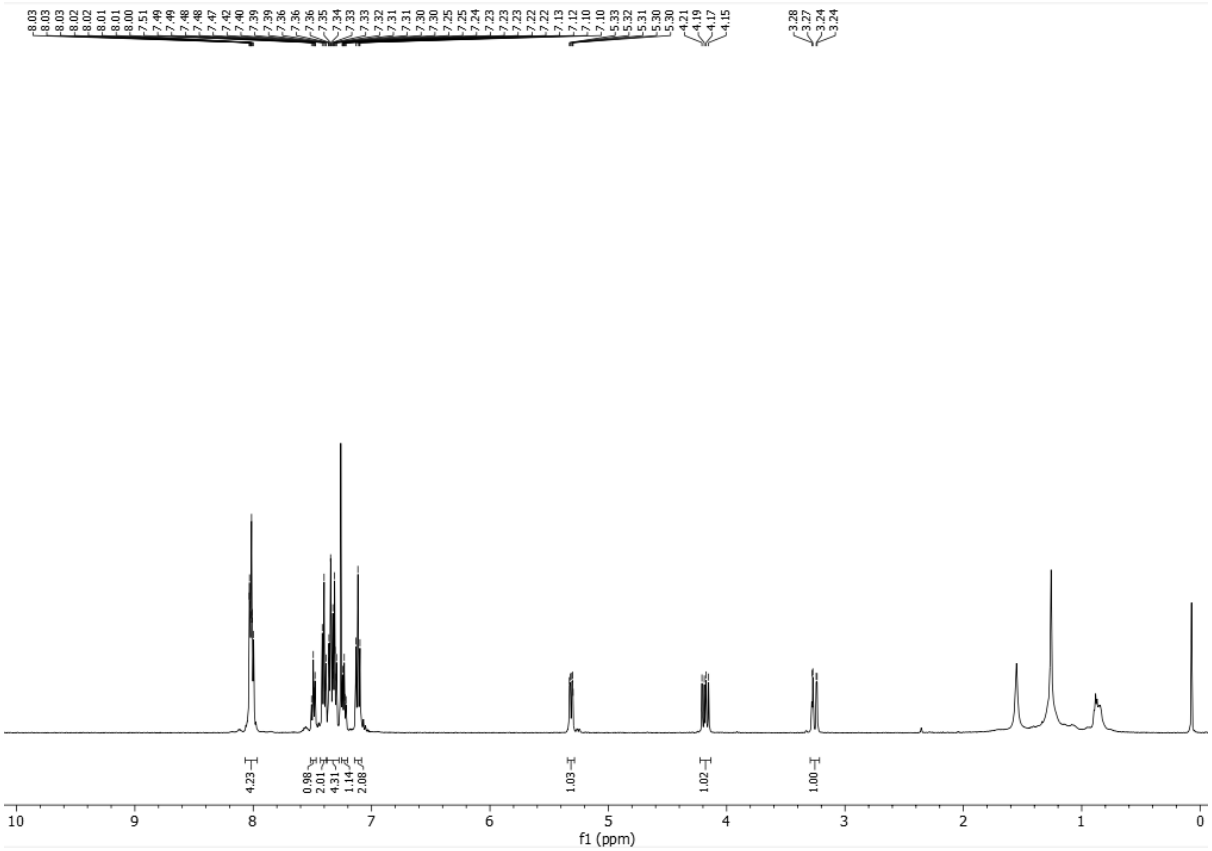


Figure **S58**. ^{1}H NMR spectrum of 4-(4-fluorophenyl)-1,2-diphenylbutane-1,4-dione in $CDCl_3$.

28, ^{19}F, $CDCl_3$, 471 MHz

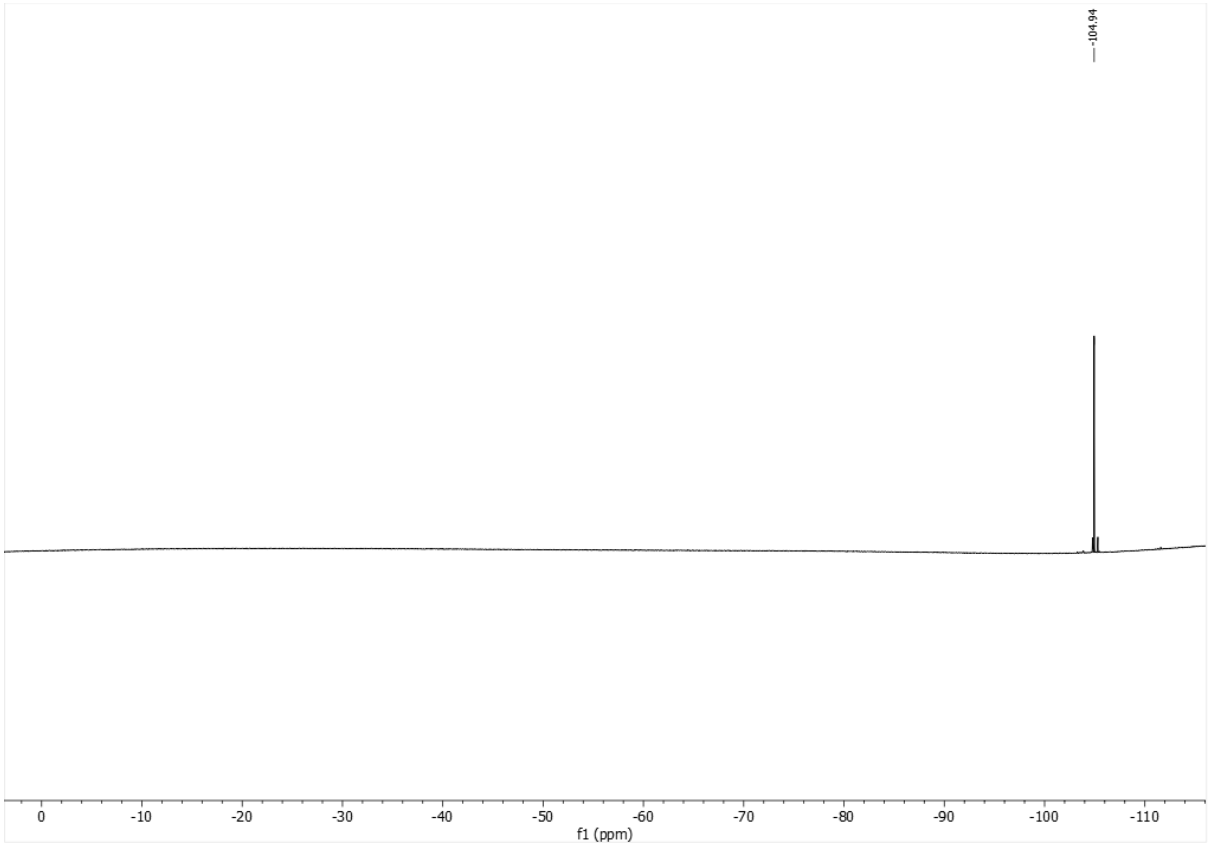


Figure **S59**. ^{19}F NMR spectrum of 4-(4-fluorophenyl)-1,2-diphenylbutane-1,4-dione in $CDCl_3$.

28, ^{13}C, $CDCl_3$, 126 MHz

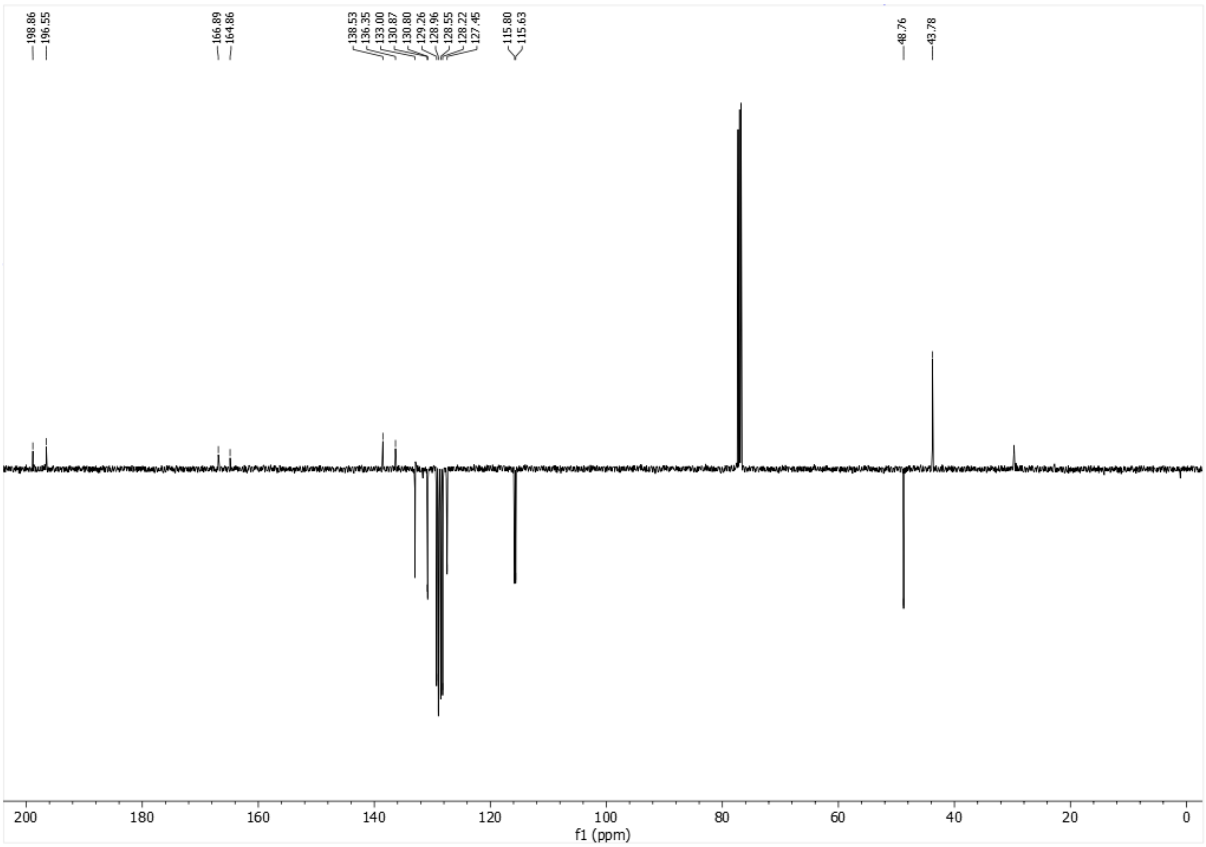
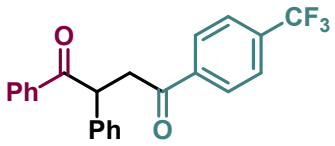


Figure **S60**. ^{13}C NMR spectrum of 4-(4-fluorophenyl)-1,2-diphenylbutane-1,4-dione in $CDCl_3$.



29, ^{1}H, $CDCl_3$, 500 MHz

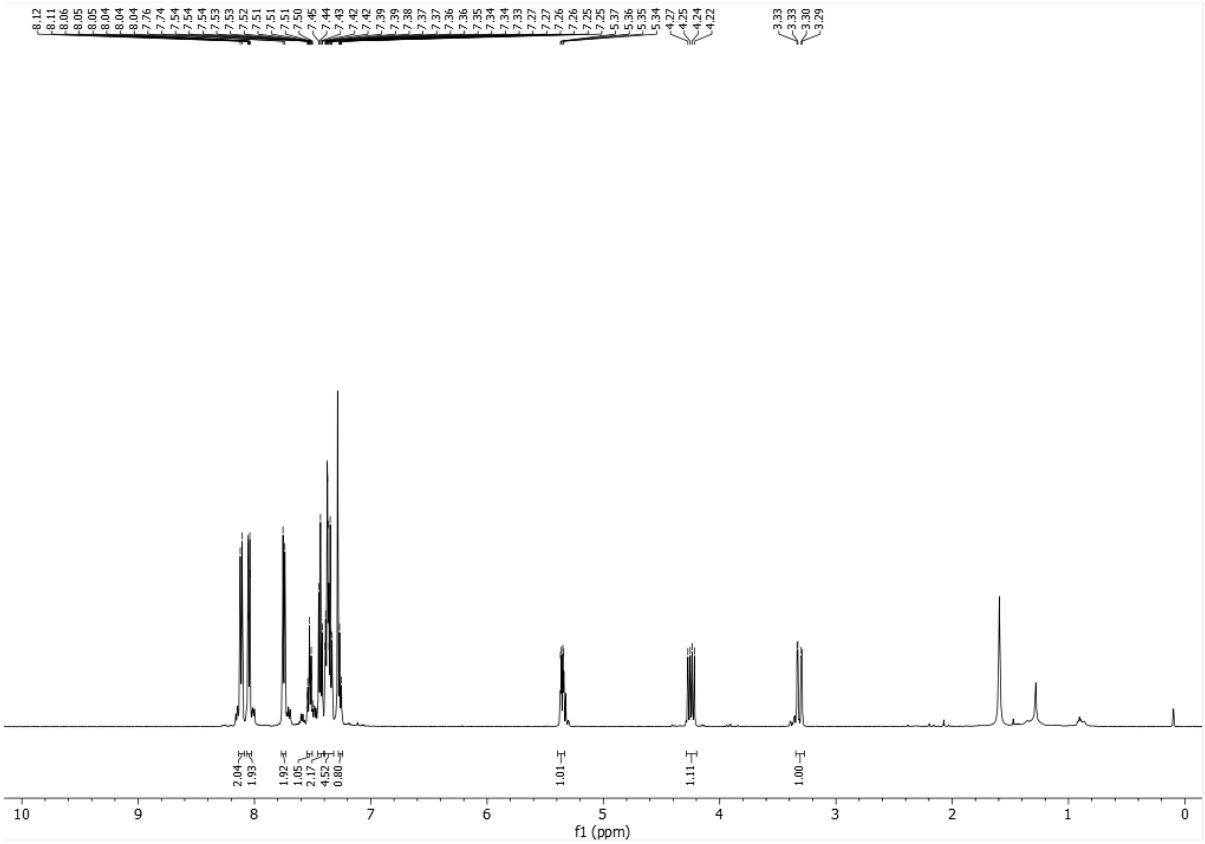


Figure **S61**. ^{1}H NMR spectrum of 1,2-diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione in $CDCl_3$.

29, ^{19}F, $CDCl_3$, 471 MHz

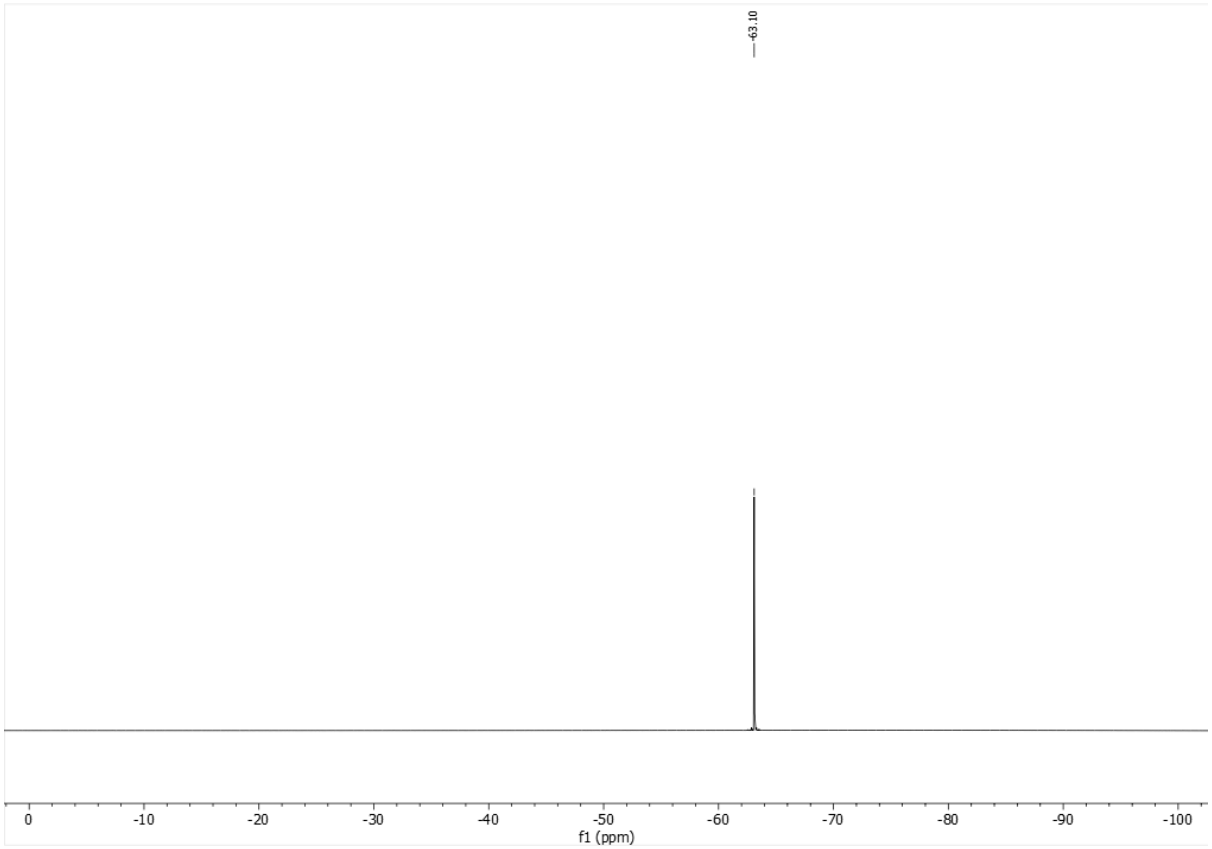


Figure **S62**. ^{19}F NMR spectrum of 1,2-diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione in $CDCl_3$.

29, ^{13}C, $CDCl_3$, 126 MHz

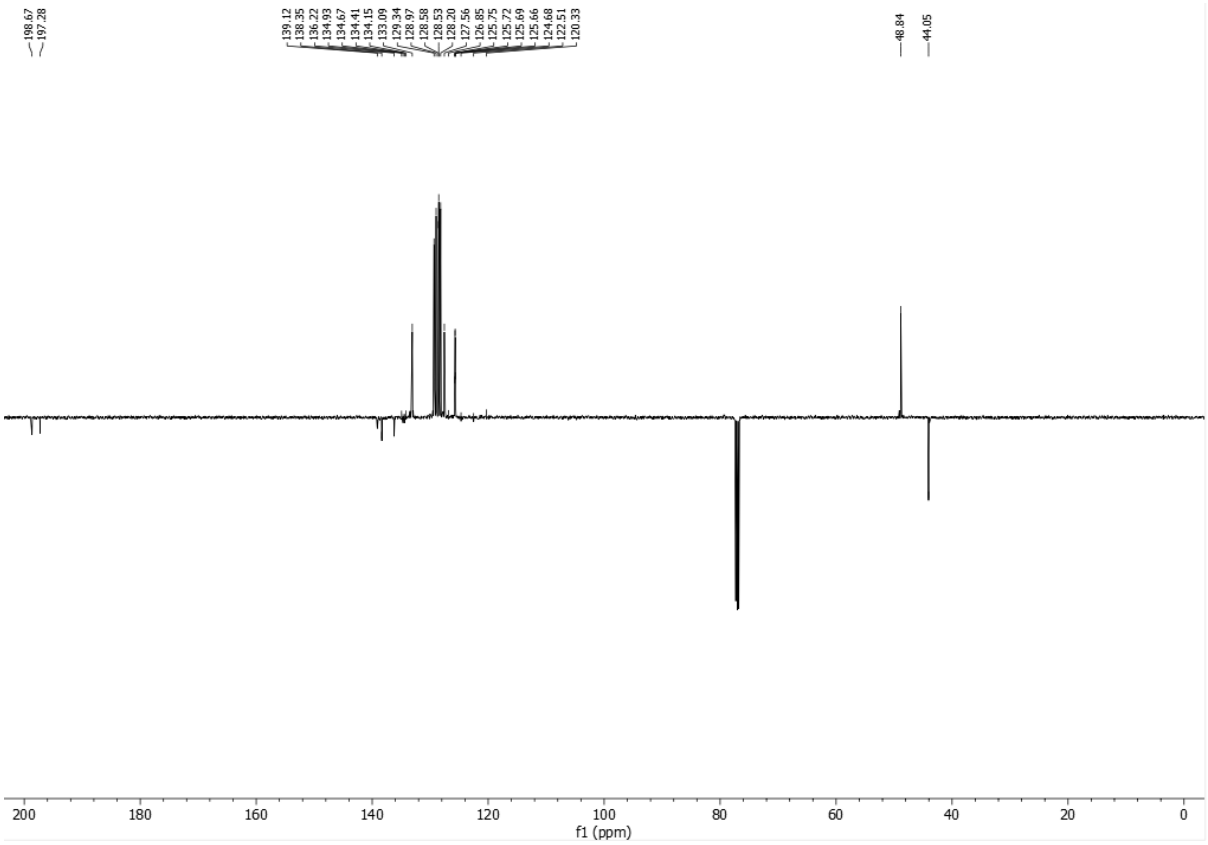
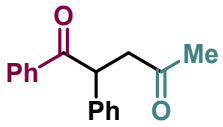


Figure **S63**. ^{13}C NMR spectrum of 1,2-diphenyl-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione in $CDCl_3$.



30, ^{1}H, $CDCl_3$, 500 MHz

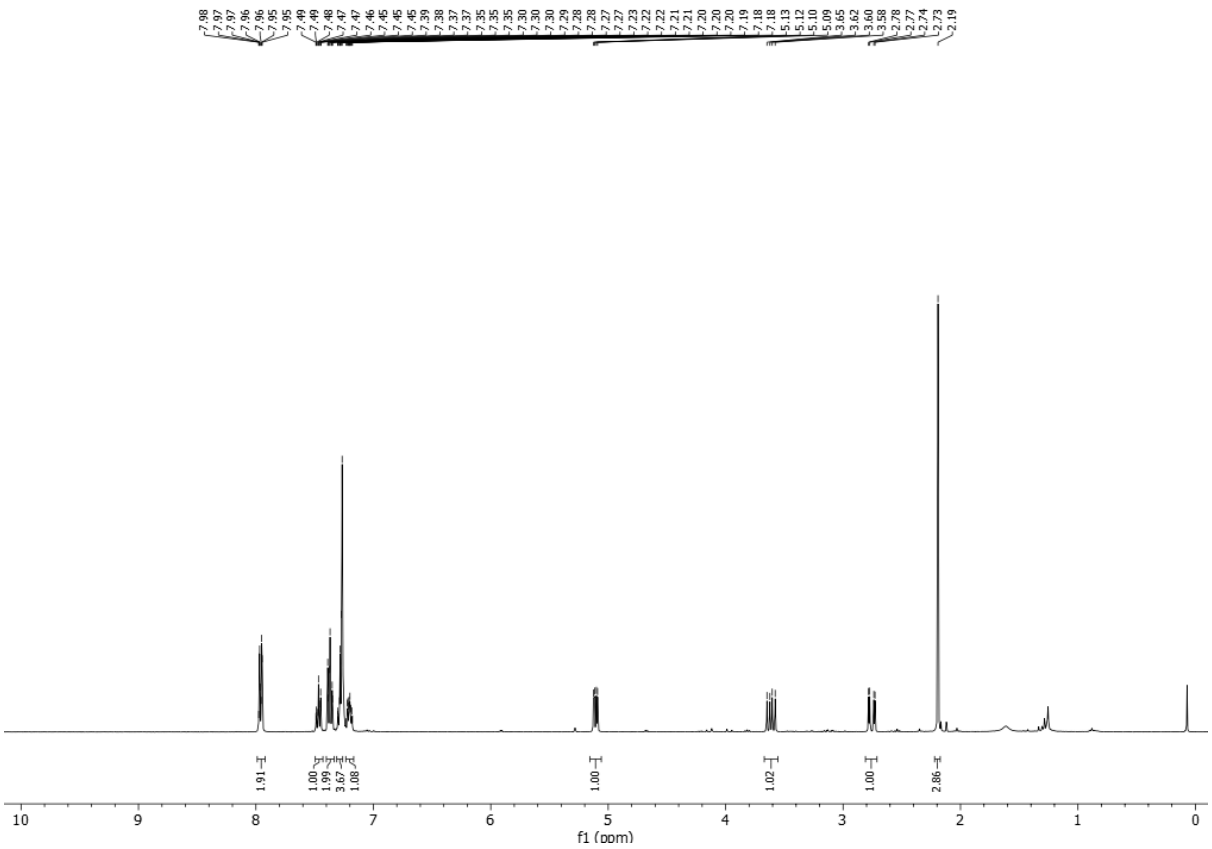


Figure **S64**. ^{1}H NMR spectrum of 1,2-diphenylpentane-1,4-dione in $CDCl_3$.

30, ^{13}C, $CDCl_3$, 126 MHz

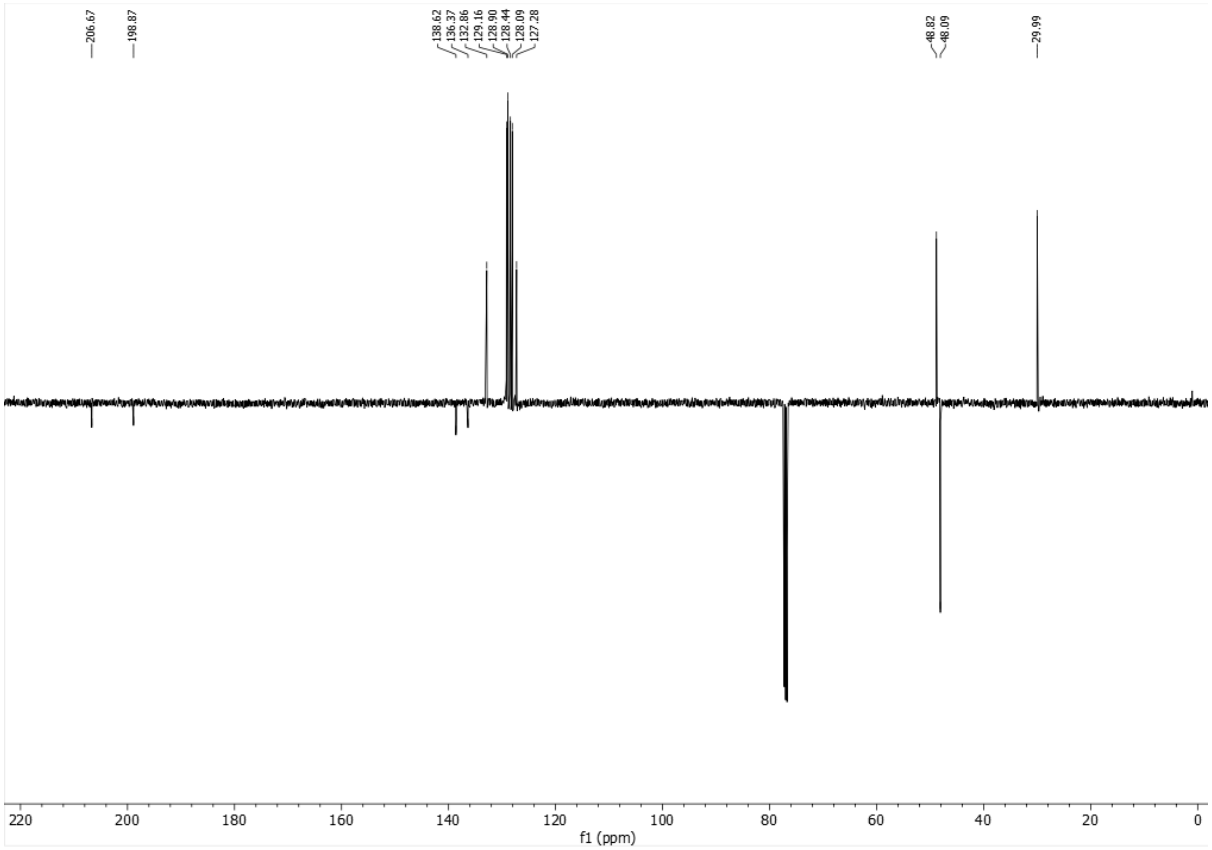
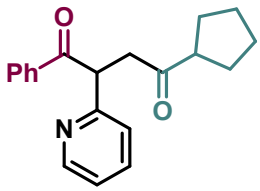


Figure **S65**. ^{13}C NMR spectrum of 1,2-diphenylpentane-1,4-dione in $CDCl_3$.



31, ^{1}H, $CDCl_3$, 500 MHz

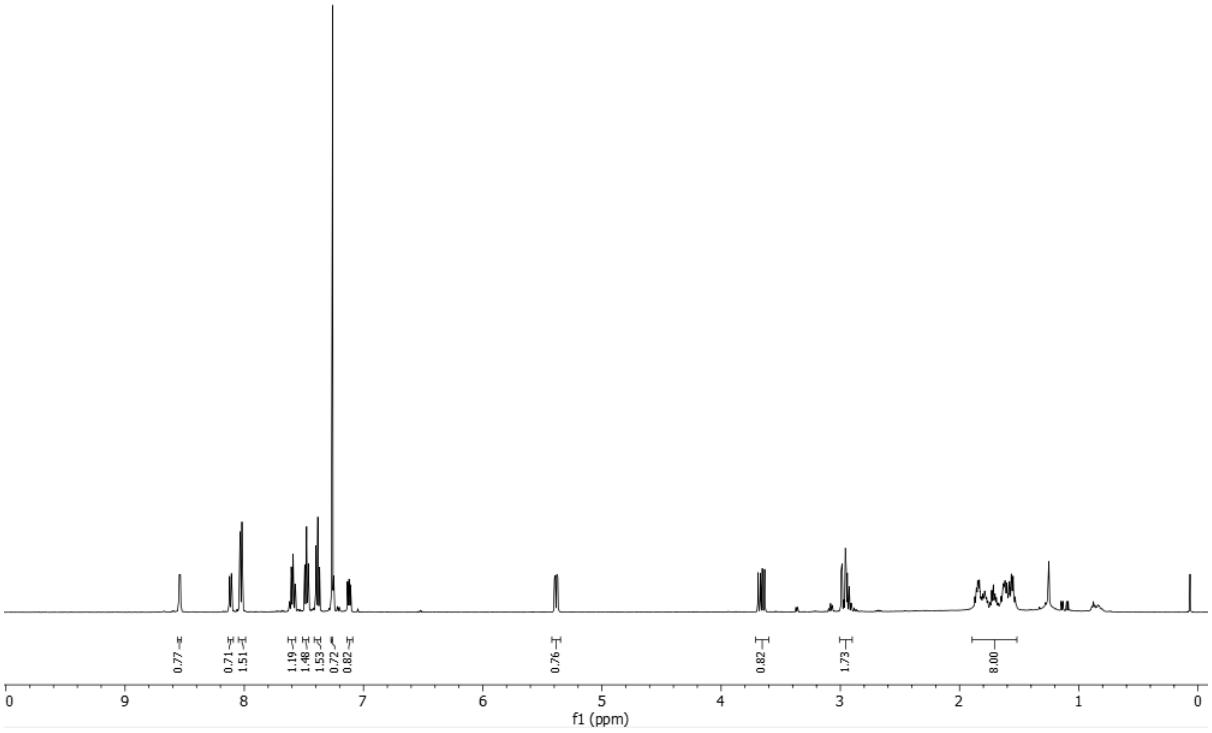


Figure **S66**. ^{1}H NMR spectrum of 4-cyclopentyl-1-phenyl-2-(pyridin-2-yl)butane-1,4-dione in $CDCl_3$.

31, ^{13}C, $CDCl_3$, 126 MHz

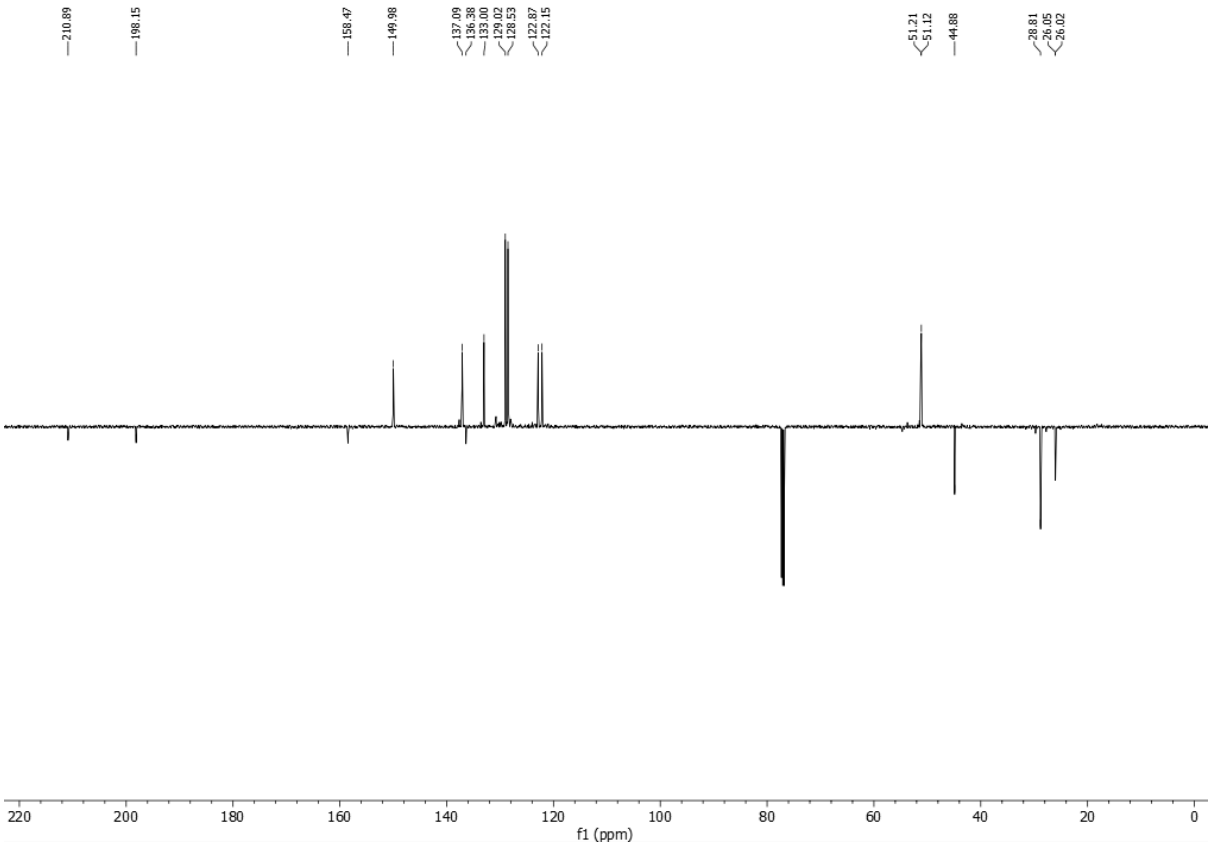
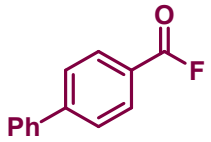


Figure **S67**. ^{13}C NMR spectrum of 4-cyclopentyl-1-phenyl-2-(pyridin-2-yl)butane-1,4-dione in $CDCl_3$.



32, ^{1}H, $CDCl_3$, 400 MHz

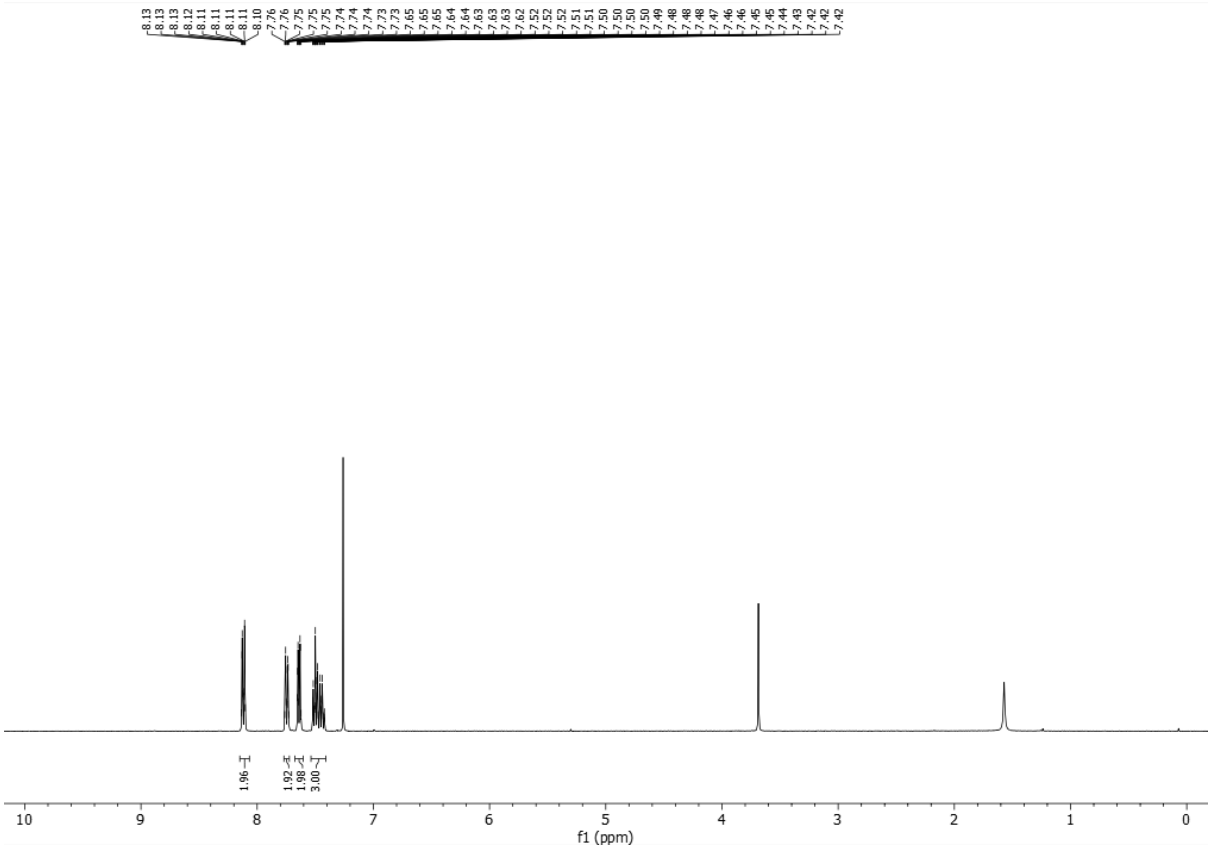


Figure **S68**. ^{1}H NMR spectrum of [1,1'-biphenyl]-4-carbonyl fluoride in $CDCl_3$.

32, ^{19}F, $CDCl_3$, 376 MHz

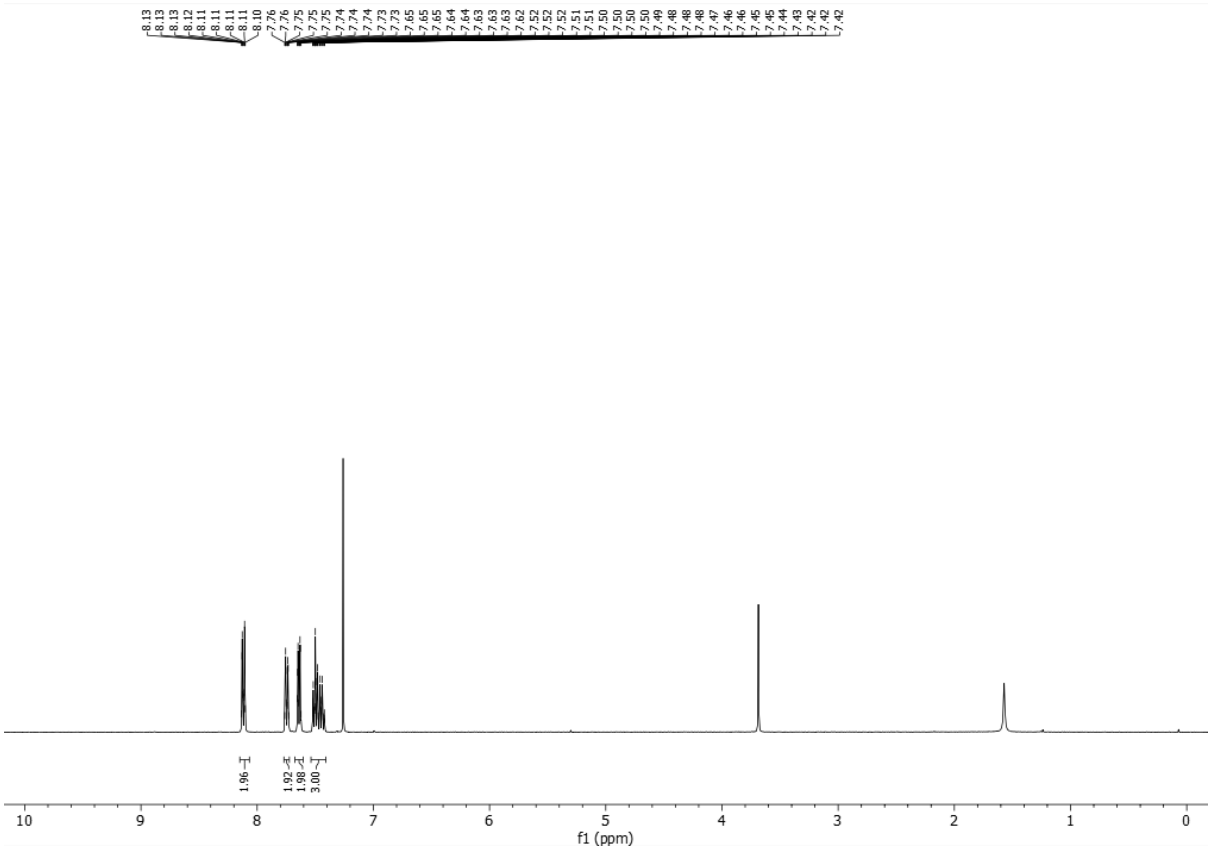
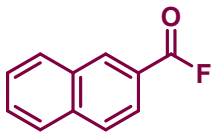


Figure **S69**. ^{19}F NMR spectrum of [1,1'-biphenyl]-4-carbonyl fluoride in $CDCl_3$.



33, ^{1}H, $CDCl_3$, 400 MHz

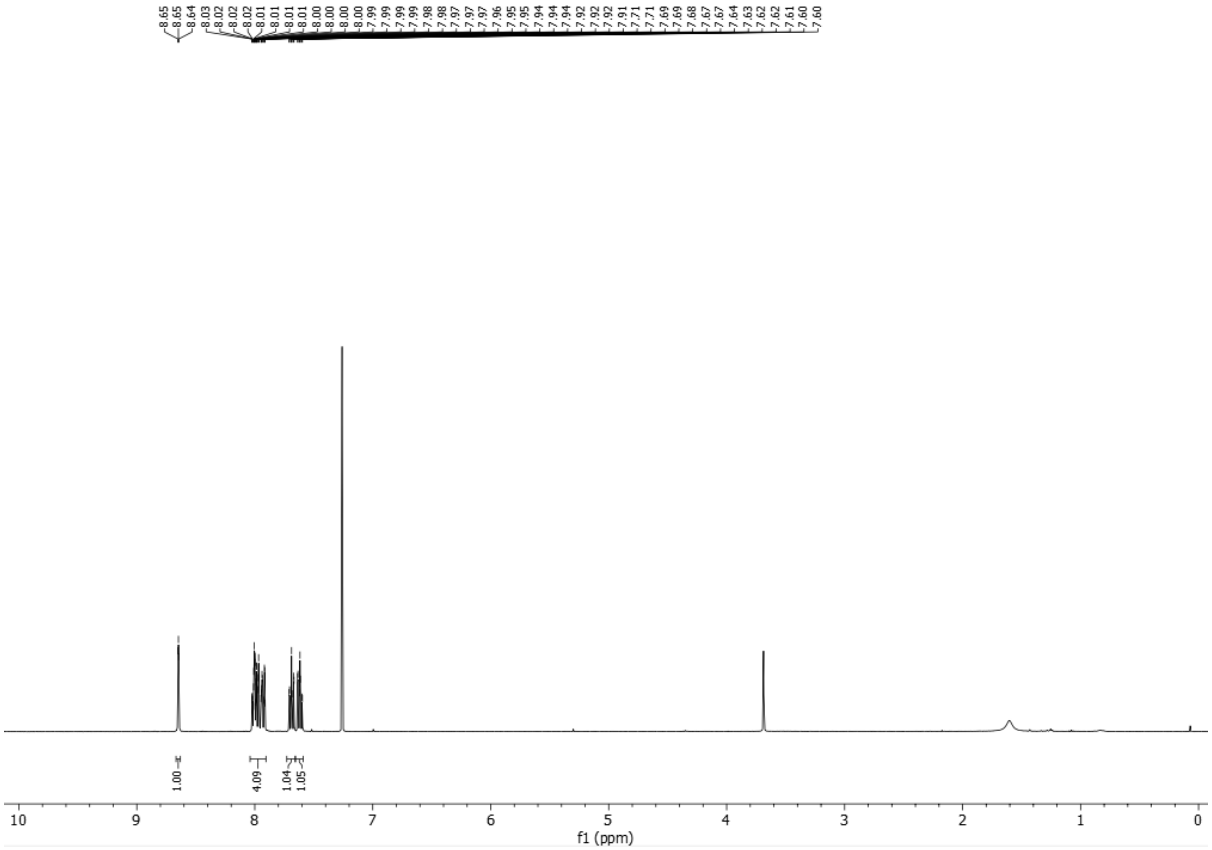


Figure **S70**. ^{1}H NMR spectrum of 2-naphthoyl fluoride in $CDCl_3$.

33, ^{19}F, $CDCl_3$, 376 MHz

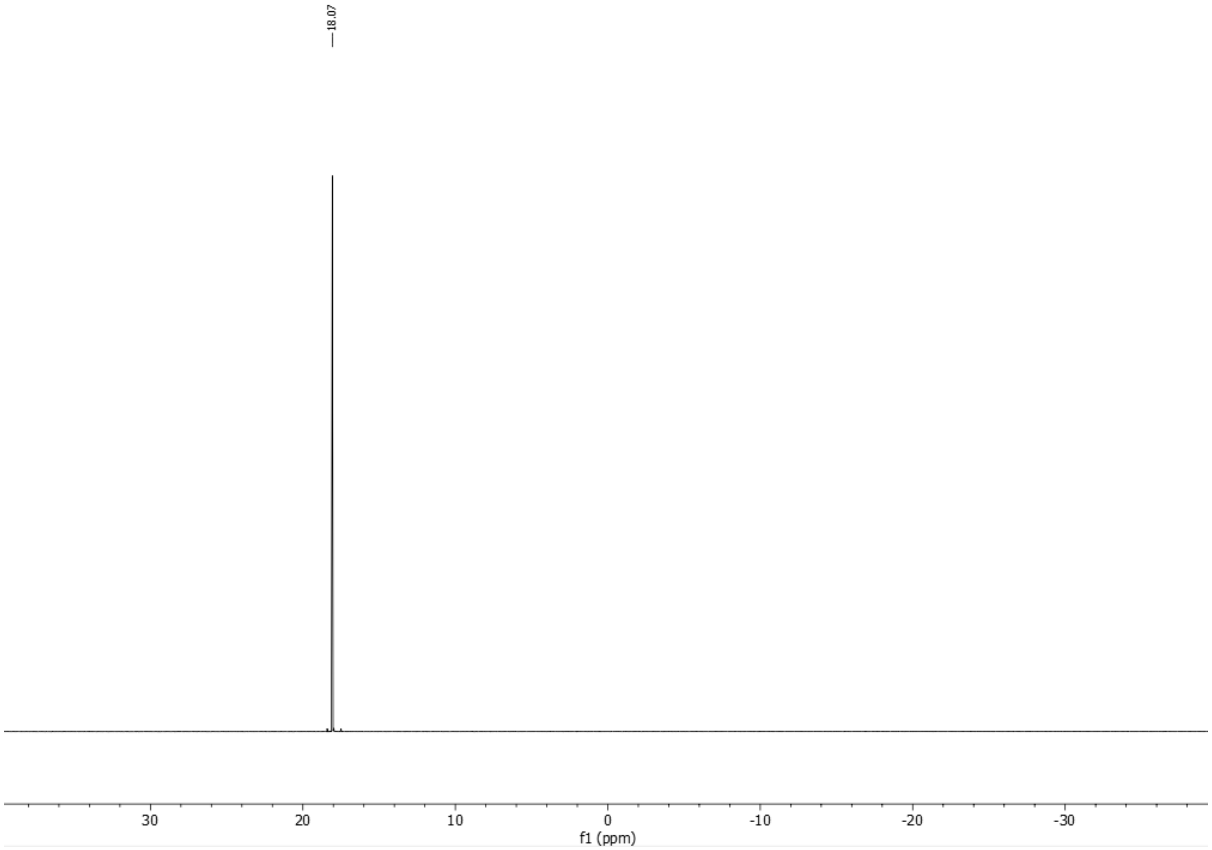
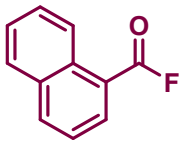


Figure **S71**. ^{19}F NMR spectrum of 2-naphthoyl fluoride in $CDCl_3$.



34, ^{1}H, $CDCl_3$, 400 MHz

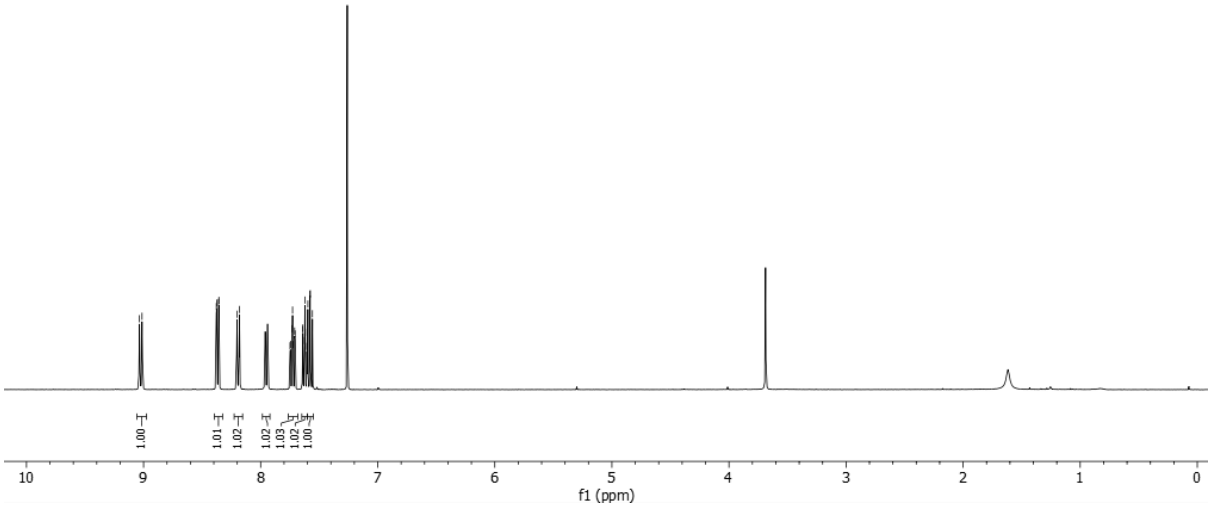


Figure **S72**. ^{1}H NMR spectrum of 1-naphthoyl fluoride in $CDCl_3$.

34, ^{19}F, $CDCl_3$, 376 MHz

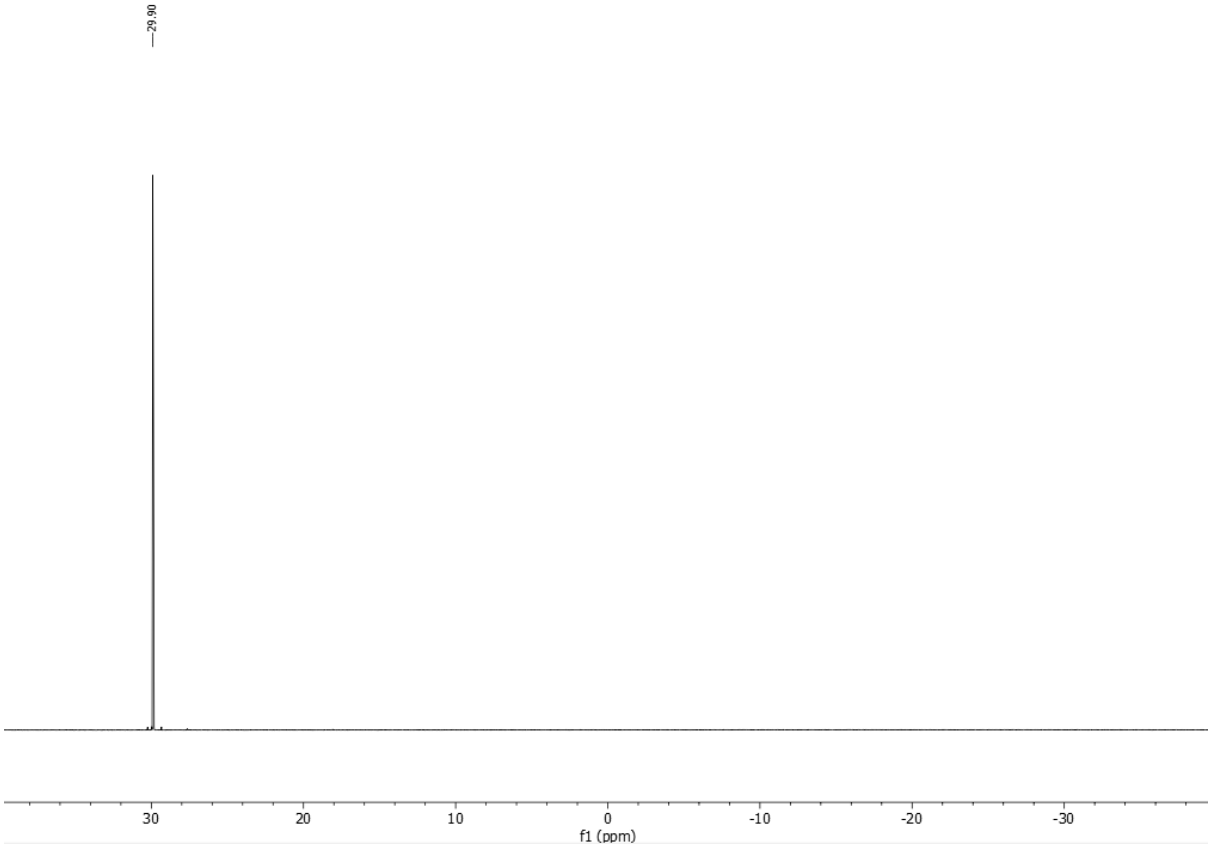
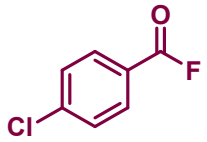


Figure **S73**. ^{19}F NMR spectrum of 1-naphthoyl fluoride in $CDCl_3$.



35, ^{1}H, $CDCl_3$, 400 MHz

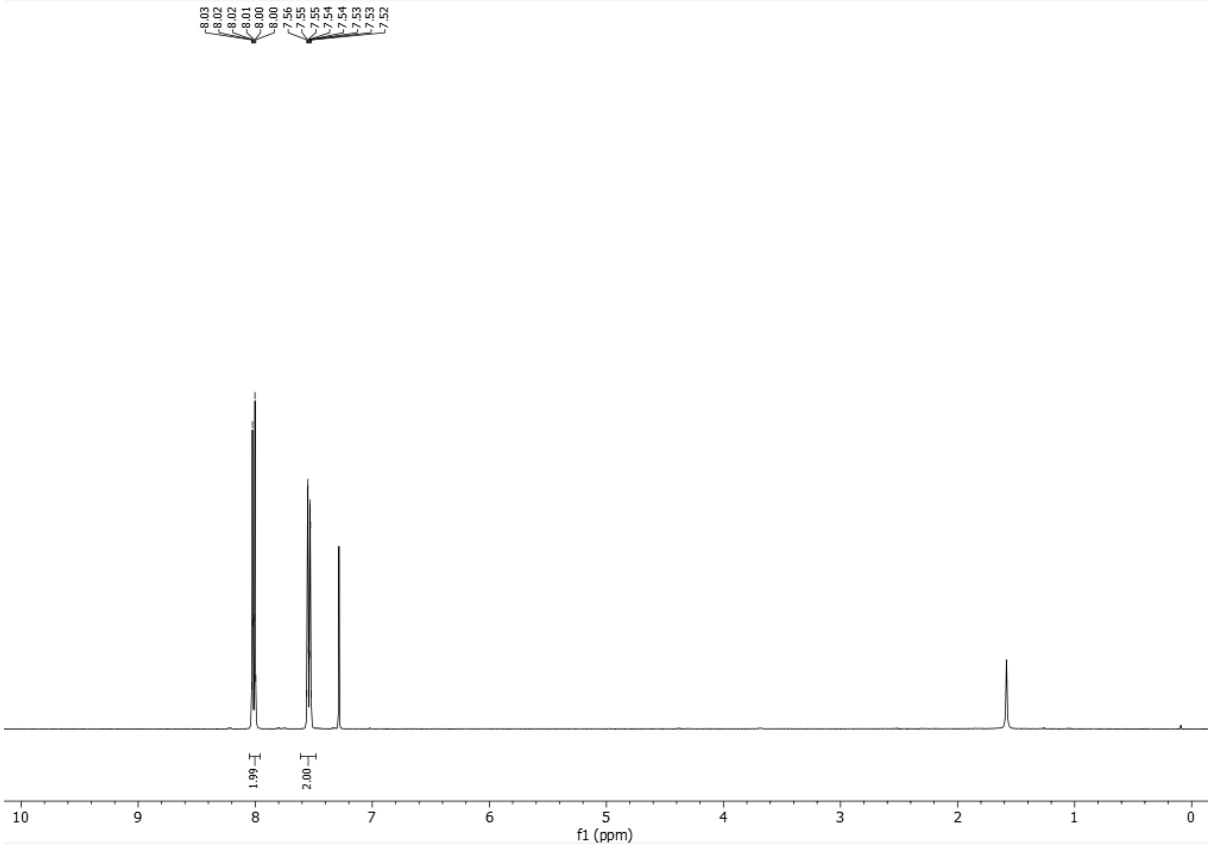


Figure **S74**. ^{1}H NMR spectrum of 4-chlorobenzoyl fluoride in $CDCl_3$.

35, ^{19}F, $CDCl_3$, 376 MHz

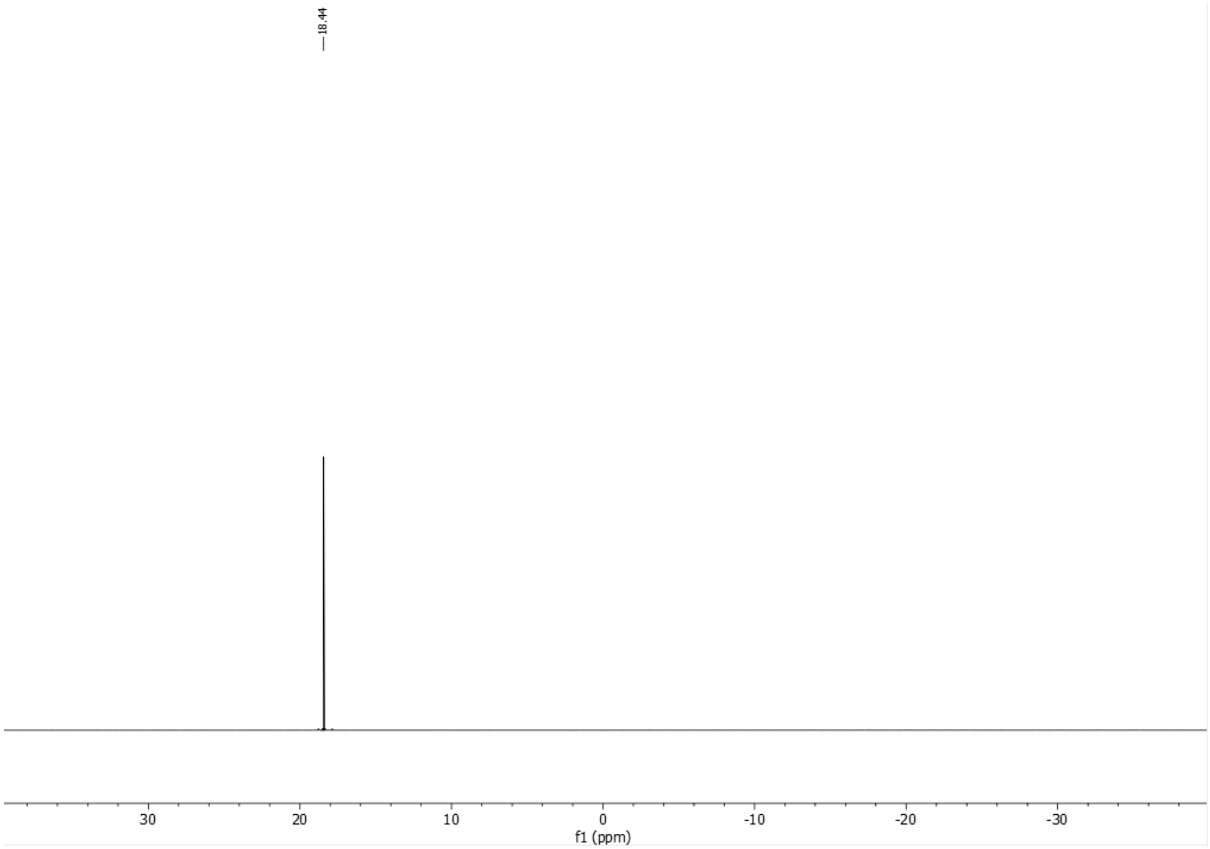
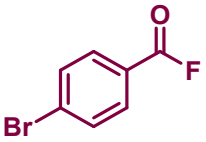


Figure **S75**. ^{19}F NMR spectrum of 4-chlorobenzoyl fluoride in $CDCl_3$.



36, ^{1}H, $CDCl_3$, 400 MHz

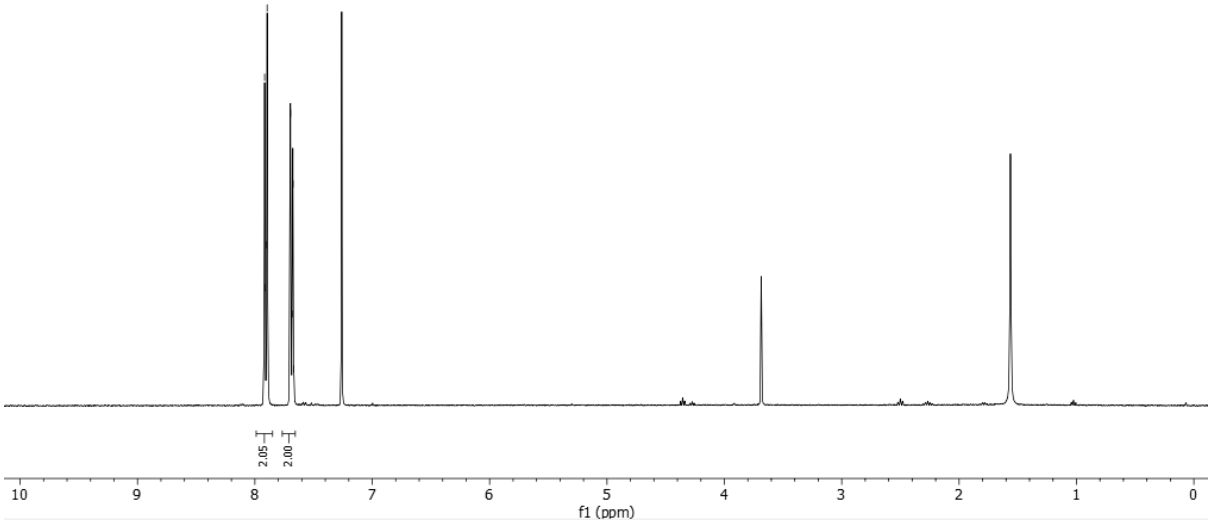


Figure **S76**. ^{1}H NMR spectrum of 4-bromobenzoyl fluoride in $CDCl_3$.

36, ^{19}F, $CDCl_3$, 376 MHz

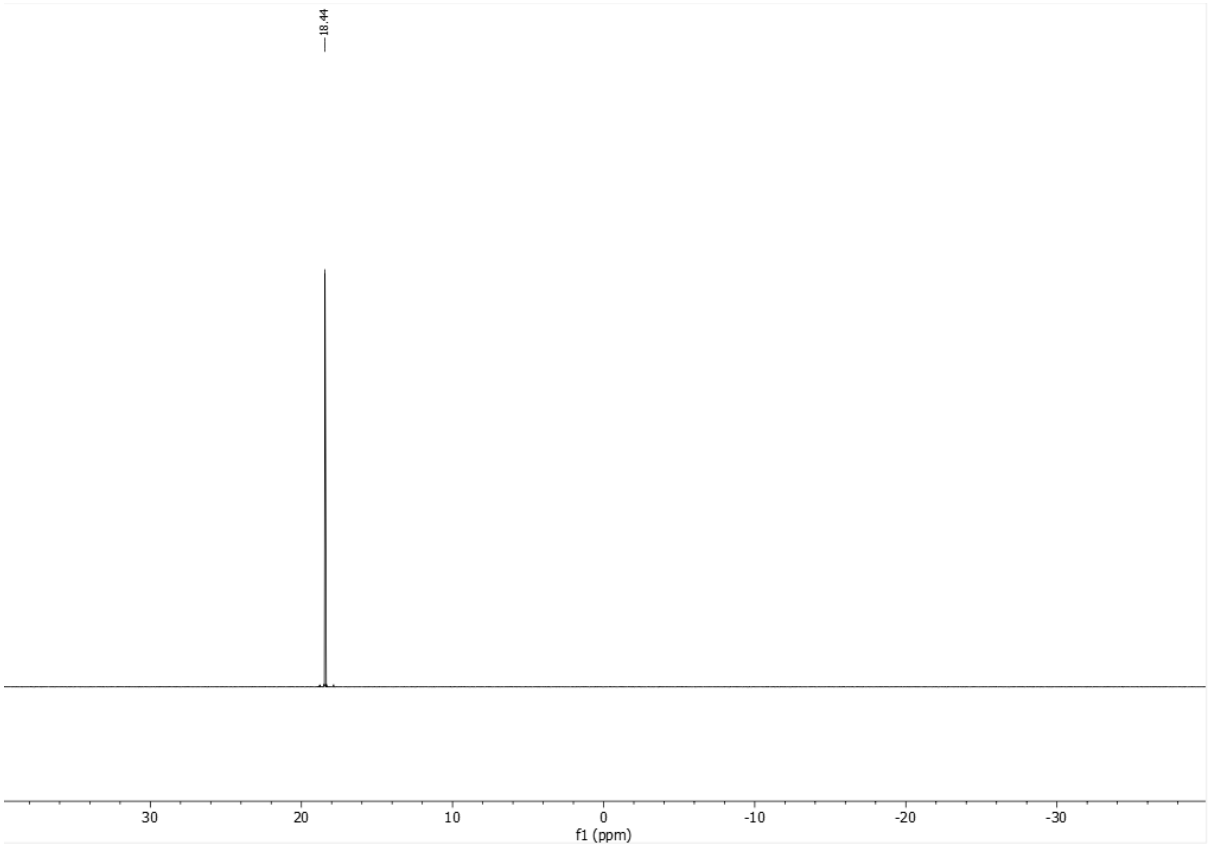
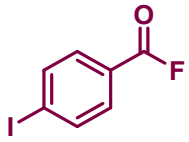


Figure **S77**. ^{19}F NMR spectrum of 4-bromobenzoyl fluoride in $CDCl_3$.



37, ^{1}H, $CDCl_3$, 400 MHz

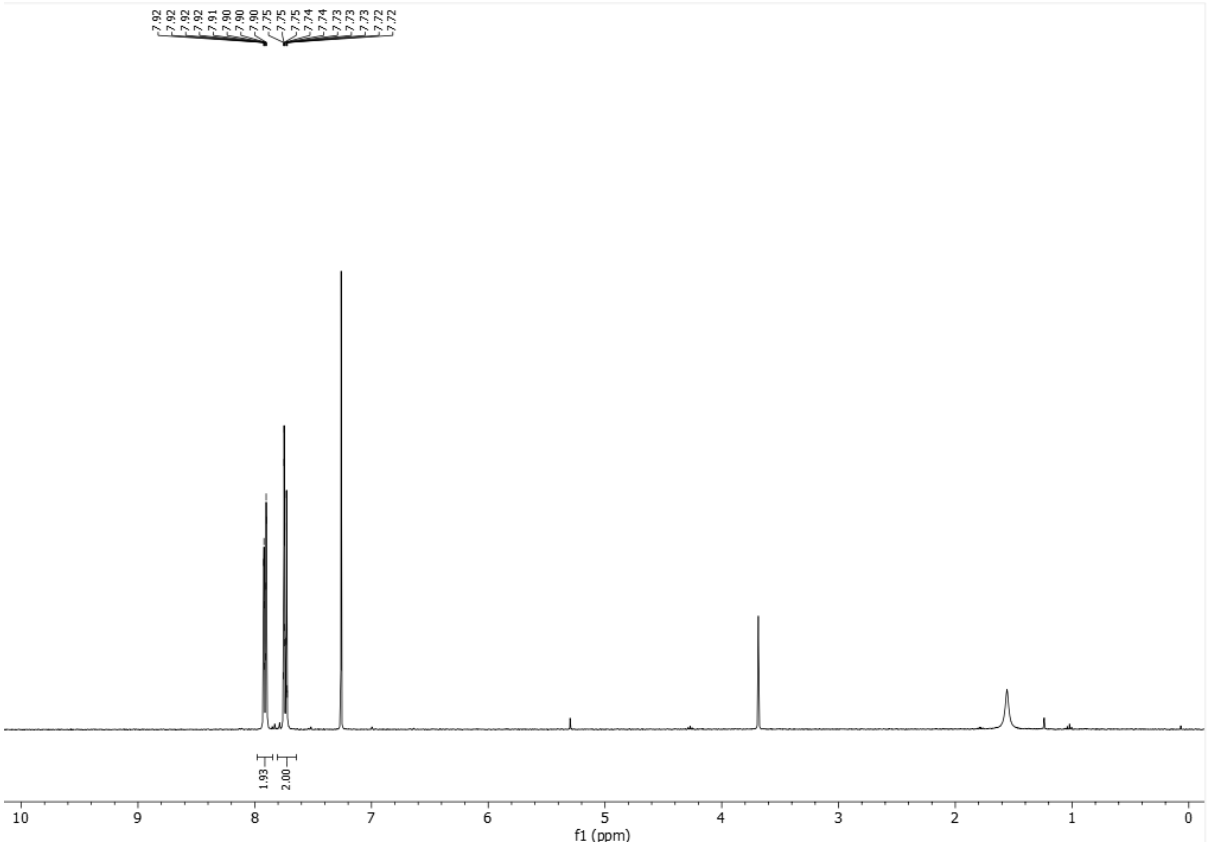


Figure **S78**. ^{1}H NMR spectrum of 4-iodobenzoyl fluoride in $CDCl_3$.

37, ^{19}F, $CDCl_3$, 376 MHz

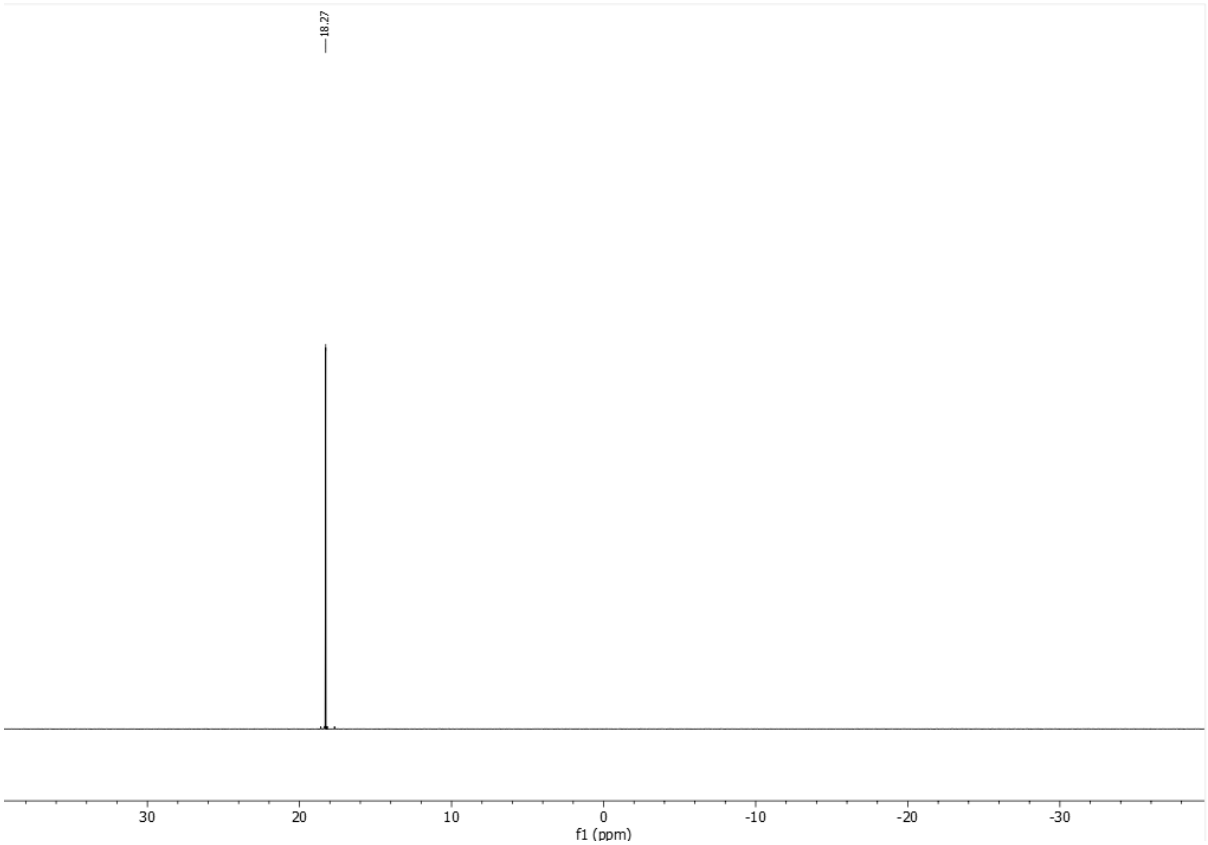
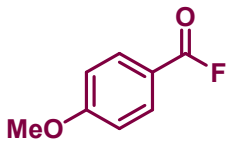


Figure **S79**. ^{19}F NMR spectrum of 4-iodobenzoyl fluoride in $CDCl_3$.



38, ^{1}H, $CDCl_3$, 400 MHz

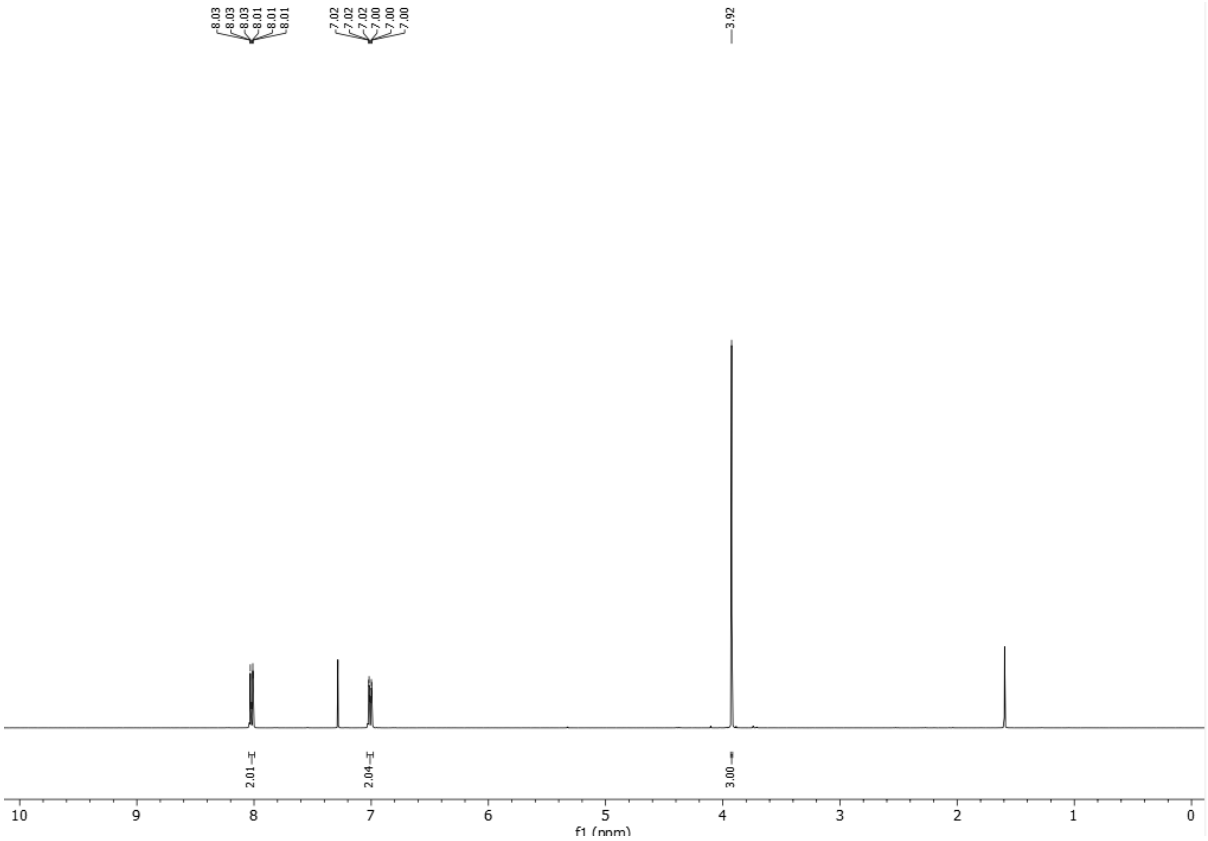


Figure **S80**. ^{1}H NMR spectrum of 4-methoxybenzoyl fluoride in $CDCl_3$.

38, ^{19}F, $CDCl_3$, 376 MHz

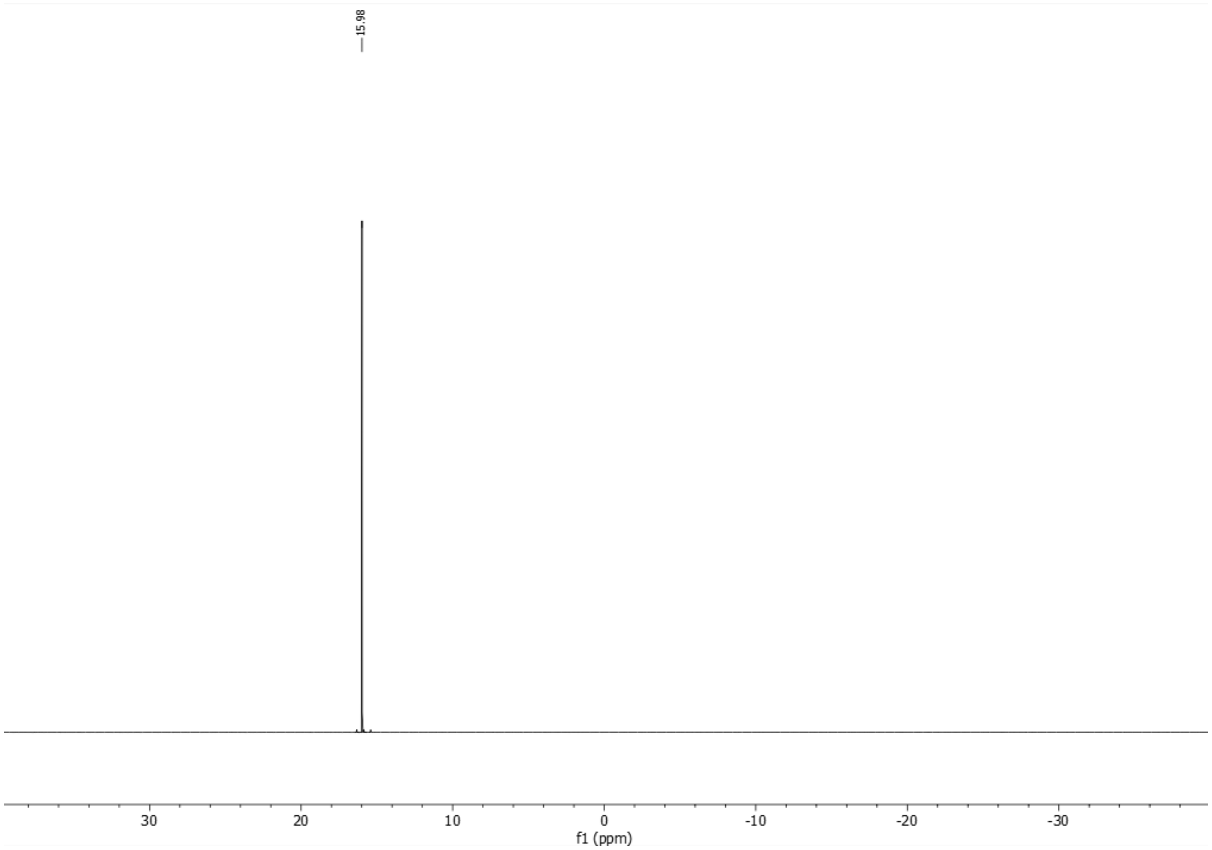
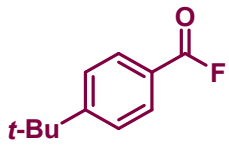


Figure **S81**. ^{19}F NMR spectrum of 4-methoxybenzoyl fluoride in $CDCl_3$.



39, ^{1}H, $CDCl_3$, 400 MHz

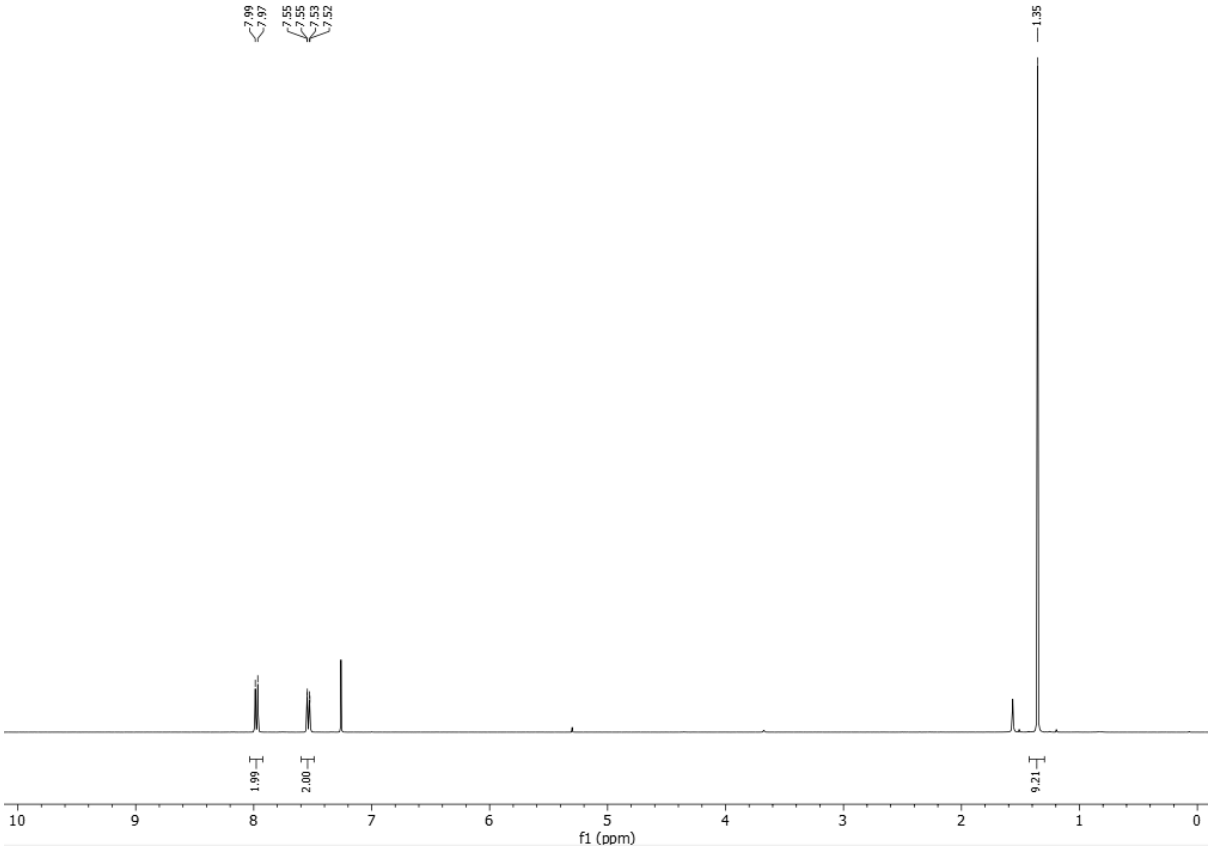


Figure **S82**. ^{1}H NMR spectrum of 4-(tert-butyl)benzoyl fluoride in $CDCl_3$.

39, ^{19}F, $CDCl_3$, 376 MHz

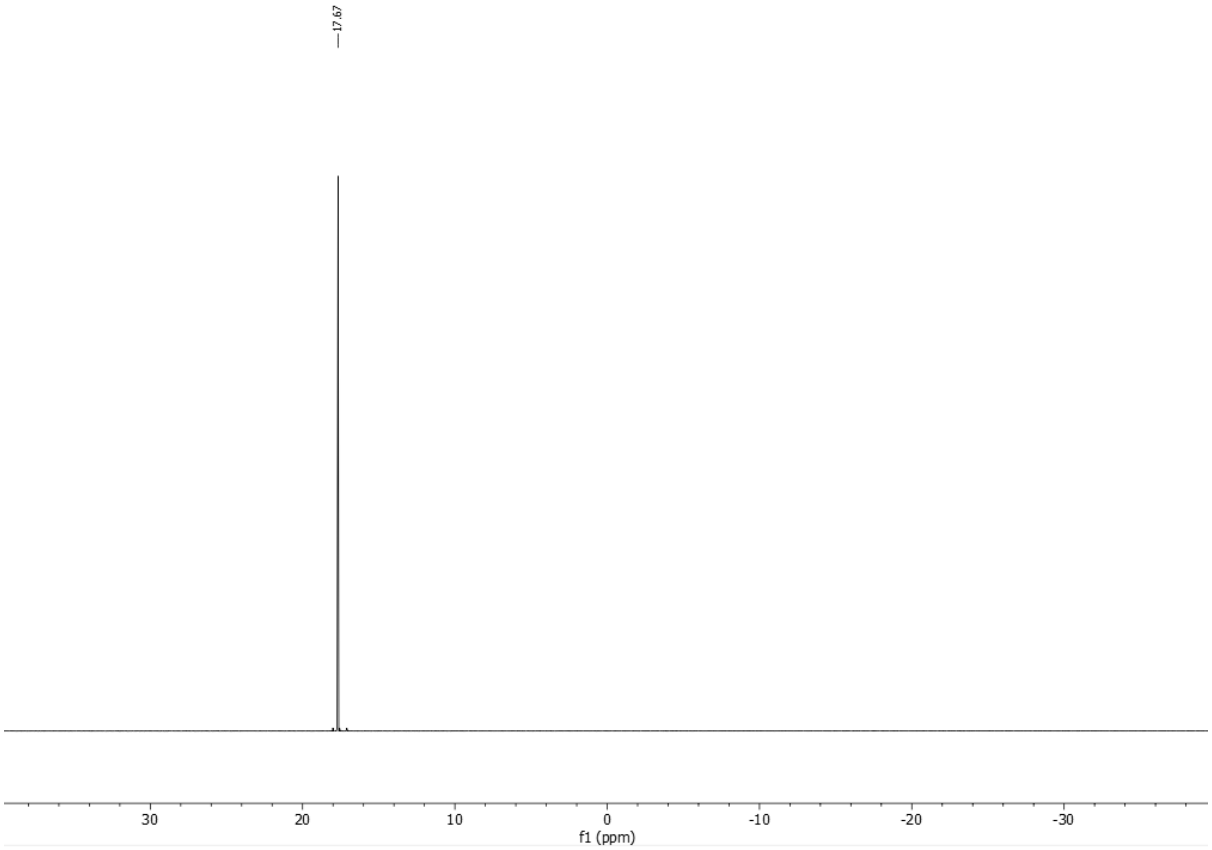
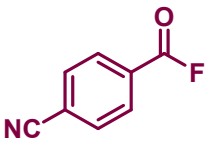


Figure **S83**. ^{19}F NMR spectrum of 4-(tert-butyl)benzoyl fluoride in $CDCl_3$.



40, ^{1}H, $CDCl_3$, 400 MHz

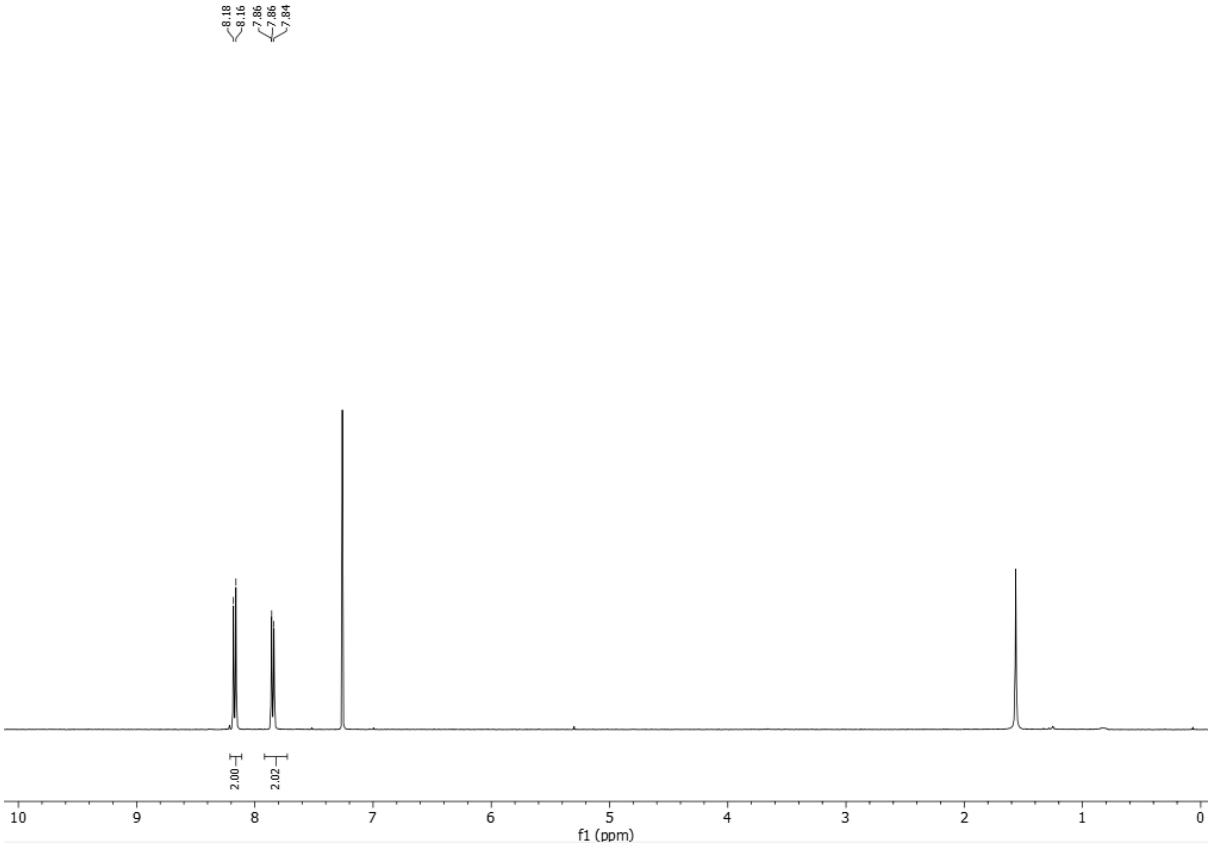


Figure **S84**. ^{1}H NMR spectrum of 4-cyanobenzoyl fluoride in $CDCl_3$.

40, ^{19}F, $CDCl_3$, 376 MHz

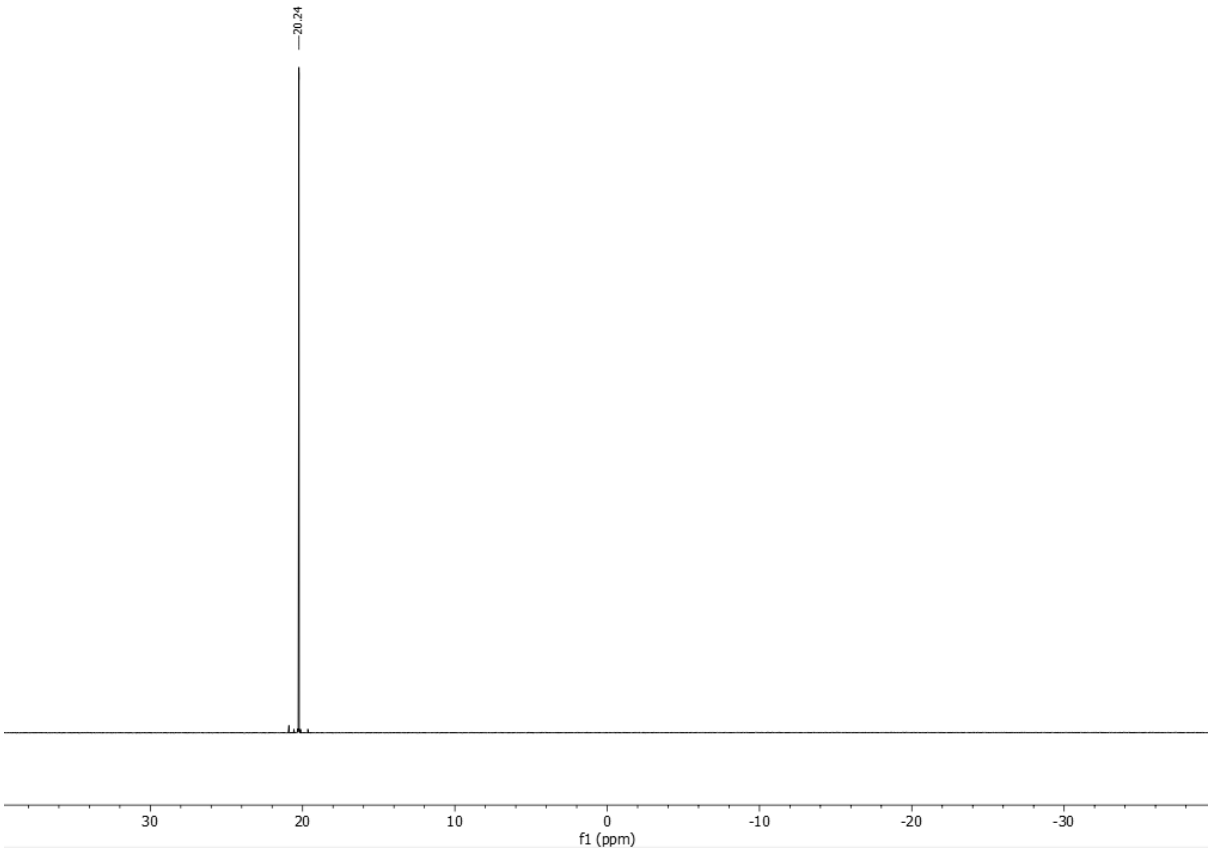
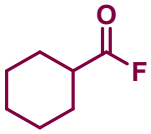


Figure **S85**. ^{19}F NMR spectrum of 4-cyanobenzoyl fluoride in $CDCl_3$.



41, ^{1}H, $CDCl_3$, 400 MHz

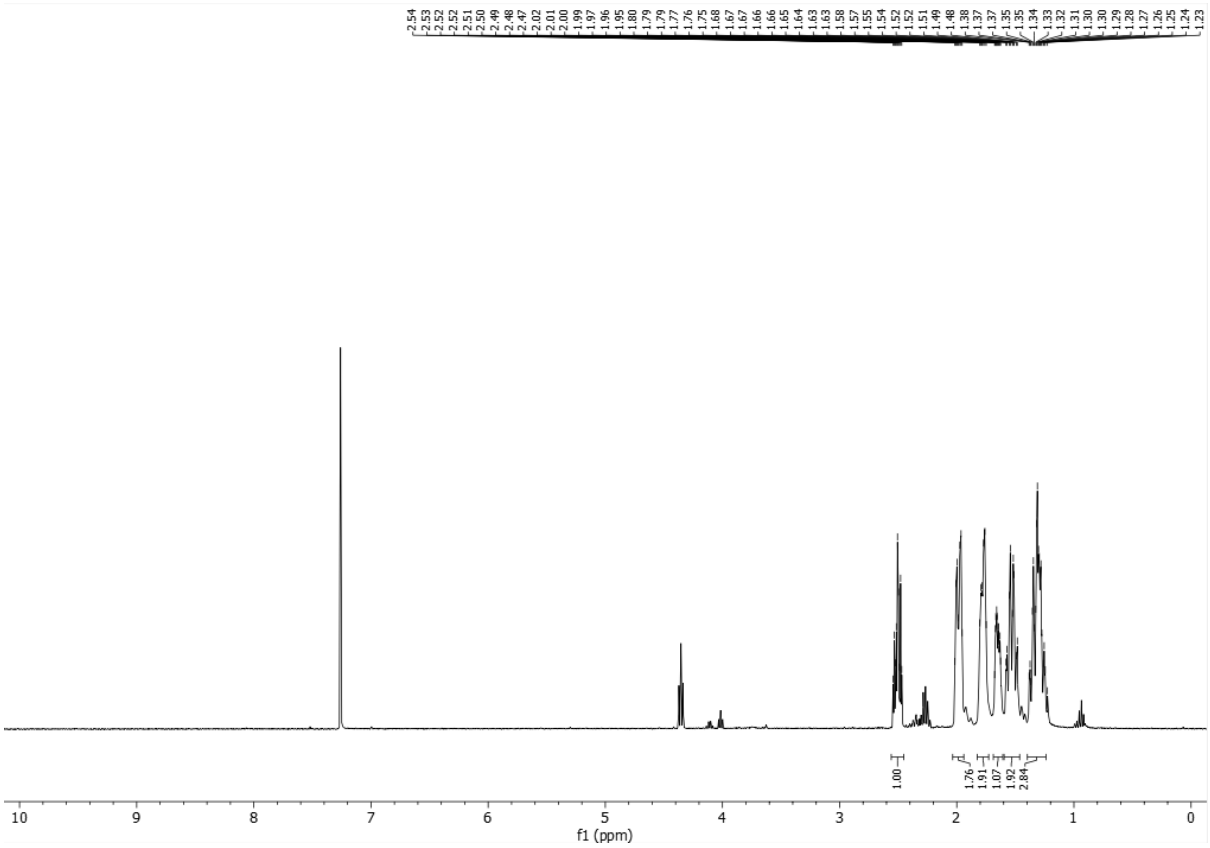


Figure **S86**. ^{1}H NMR spectrum of cyclohexanecarbonyl fluoride in $CDCl_3$.

41, ^{19}F, $CDCl_3$, 376 MHz

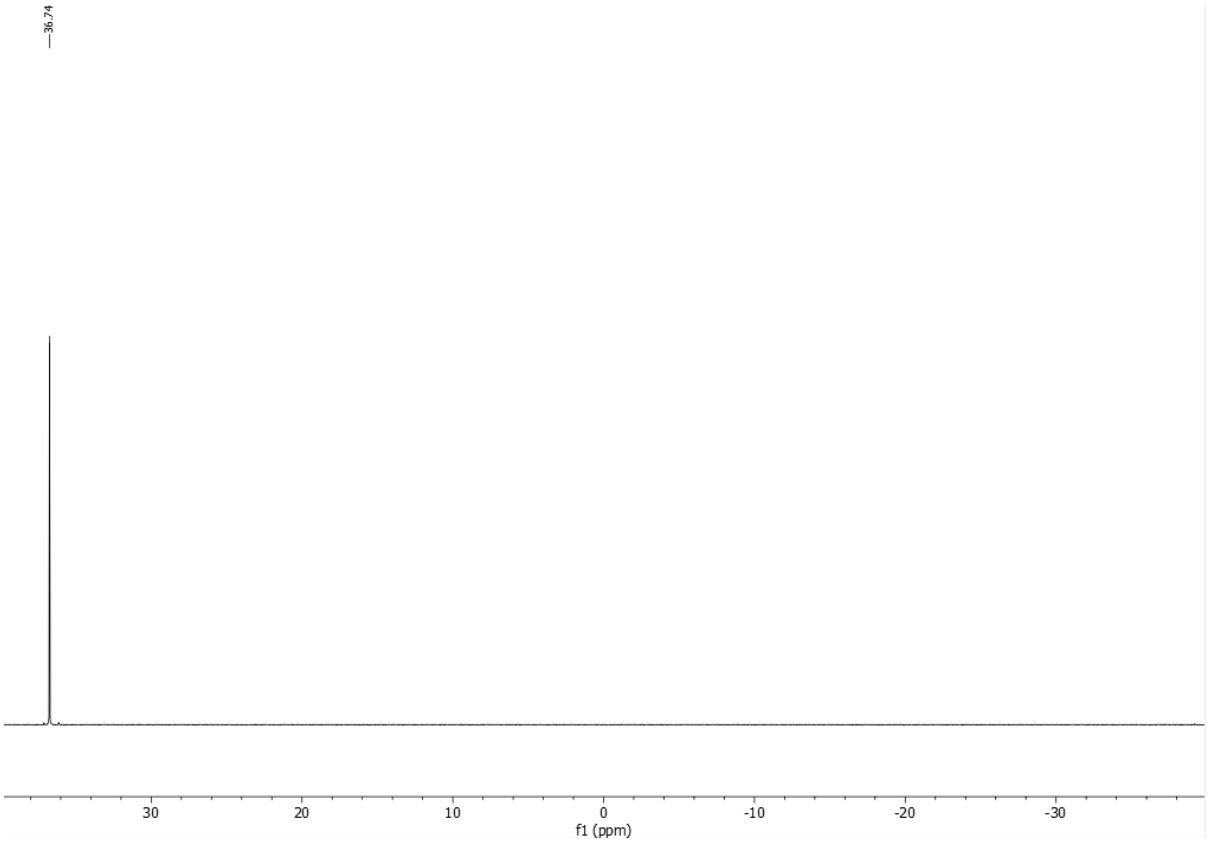
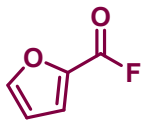


Figure **S87**. ^{19}F NMR spectrum of cyclohexanecarbonyl fluoride in $CDCl_3$.



42, ^{1}H, $CDCl_3$, 400 MHz

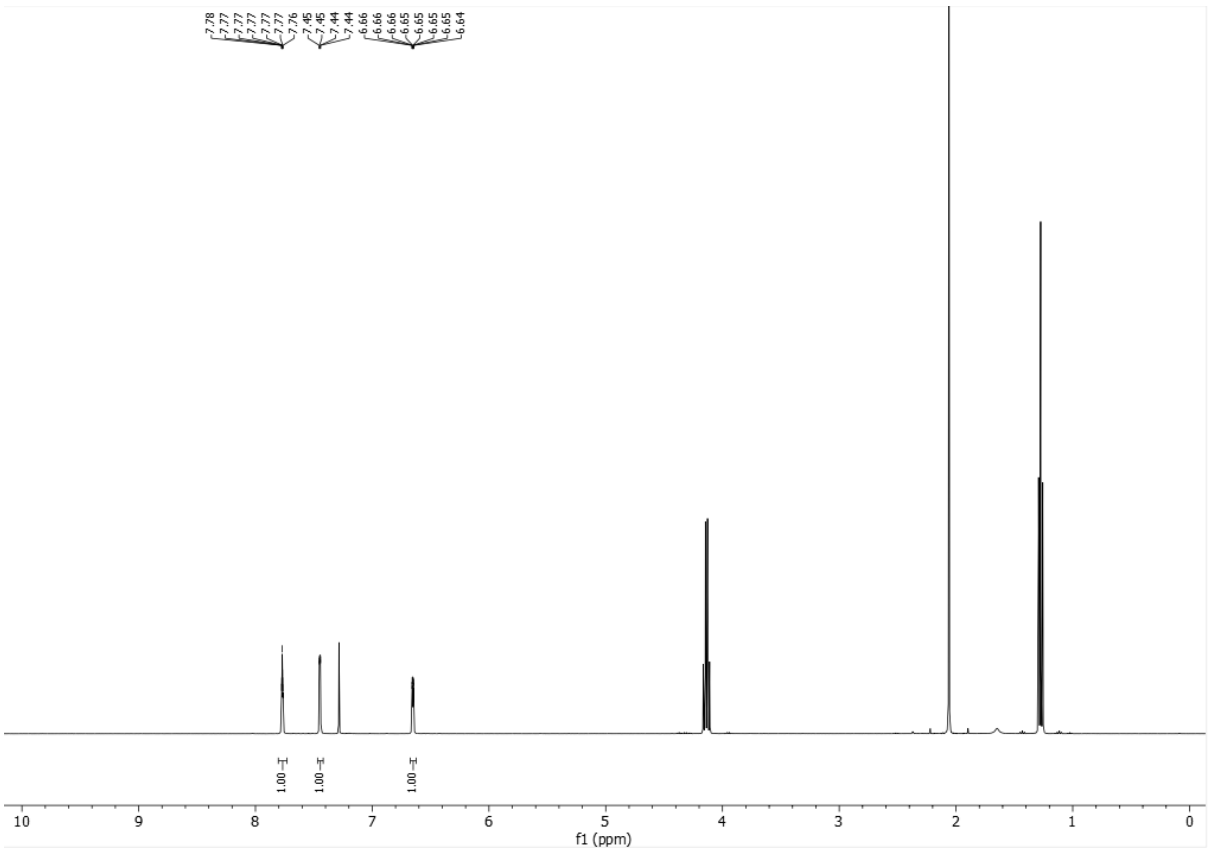


Figure **S88**. ^{1}H NMR spectrum of 2-furanoyl fluoride in $CDCl_3$.

42, ^{19}F, $CDCl_3$, 376 MHz

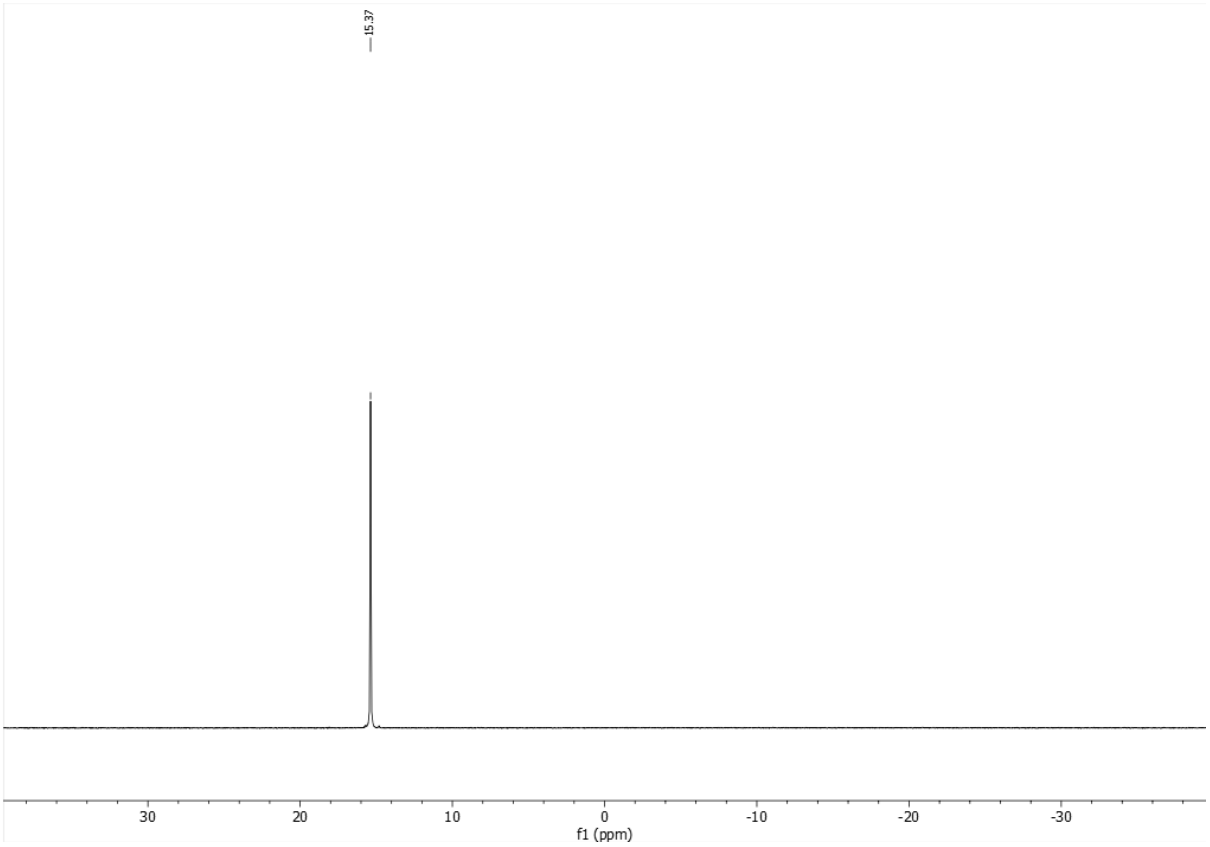
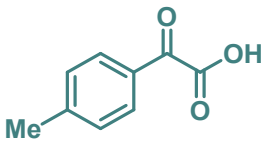


Figure **S89**. ^{19}F NMR spectrum of 2-furanoyl fluoride in $CDCl_3$.



43, ^{1}H, $CDCl_3$, 500 MHz

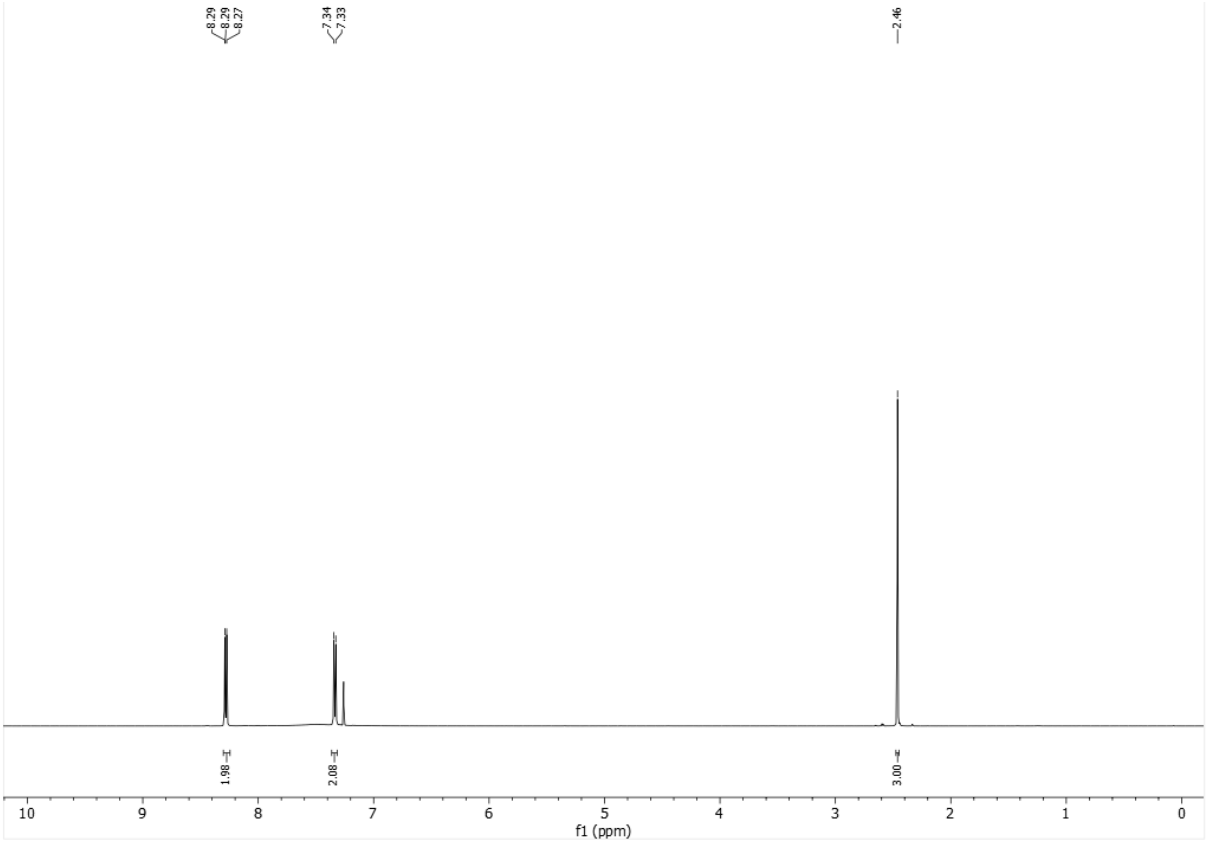


Figure **S90**. ^{1}H NMR spectrum of 2-Oxo-2-(p-tolyl)acetic acid in $CDCl_3$.

43, ^{13}C, $CDCl_3$, 126 MHz

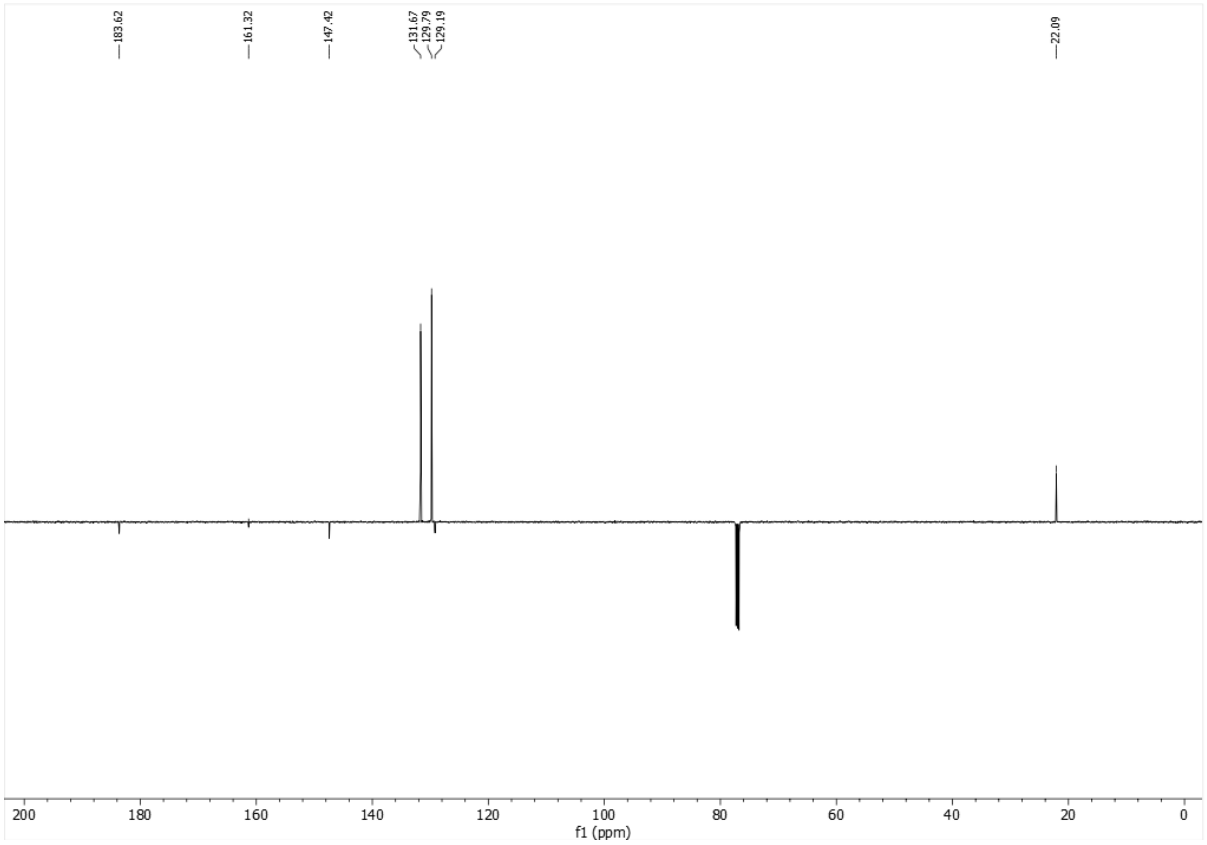
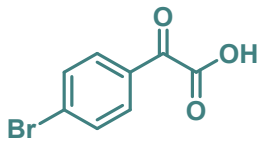


Figure **S91**. ^{13}C NMR spectrum of 2-Oxo-2-(p-tolyl)acetic acid in $CDCl_3$.



44, ^{1}H, $CDCl_3$, 500 MHz

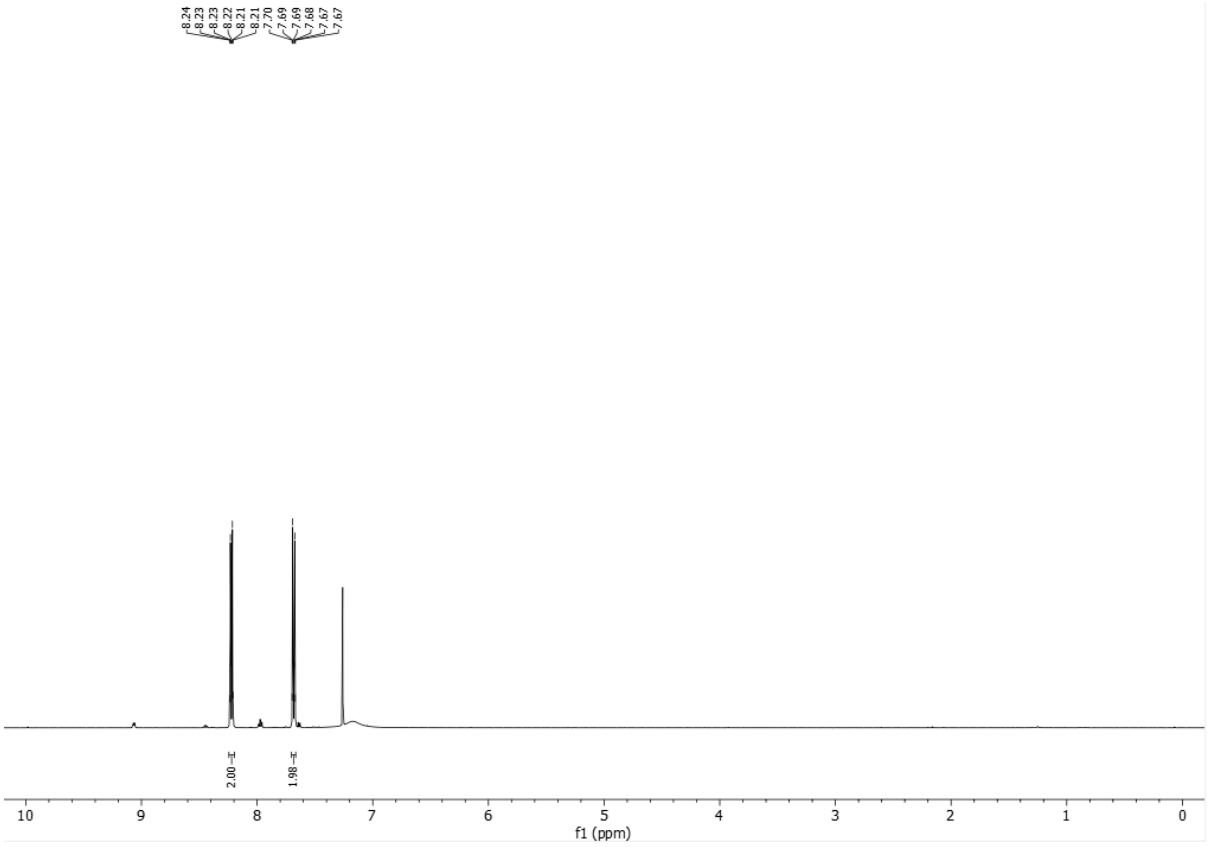


Figure **S92**. ^{1}H NMR spectrum of 2-(4-bromophenyl)-2-oxoacetic acid in $CDCl_3$.

44, ^{13}C, $CDCl_3$, 126 MHz

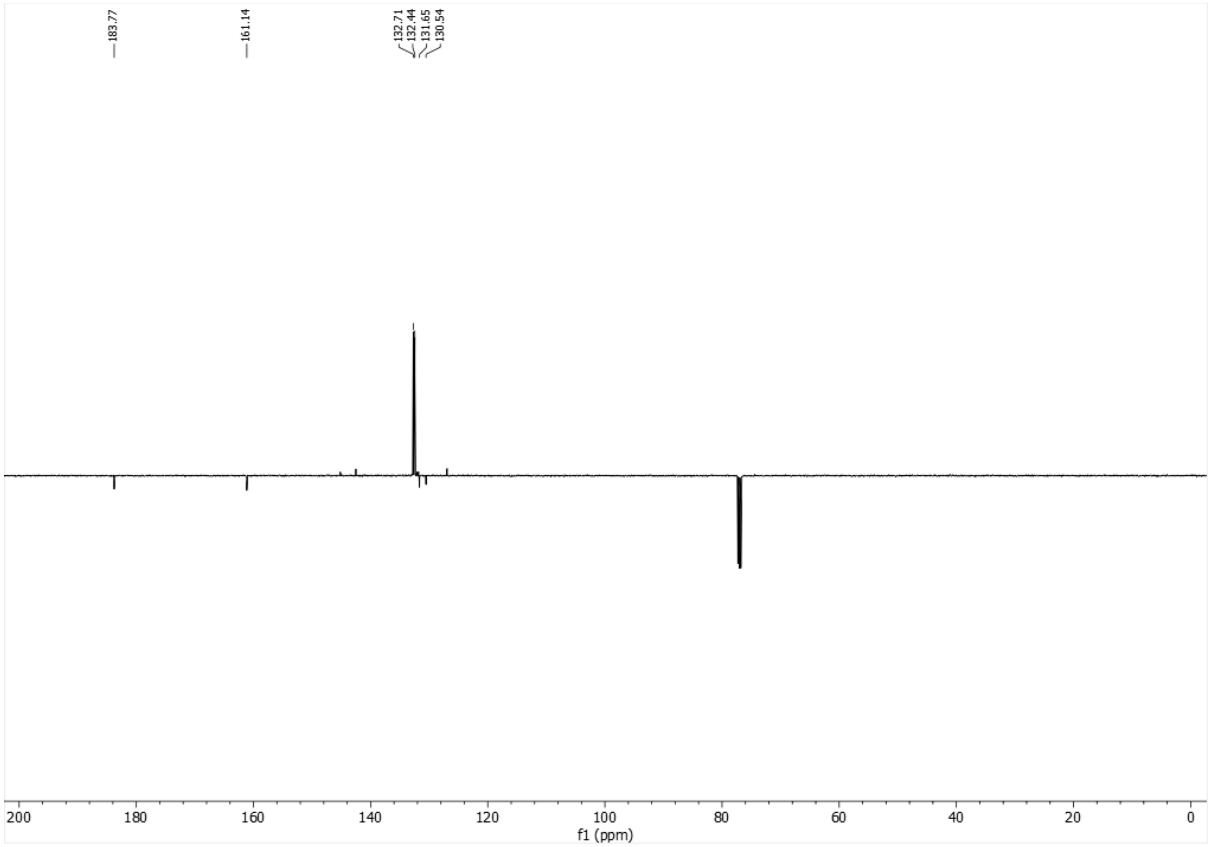
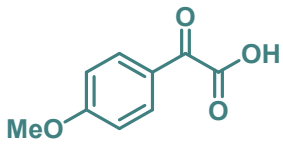


Figure **S93**. ^{13}C NMR spectrum of 2-(4-bromophenyl)-2-oxoacetic acid in $CDCl_3$.



45, ^{1}H, $CDCl_3$, 500 MHz

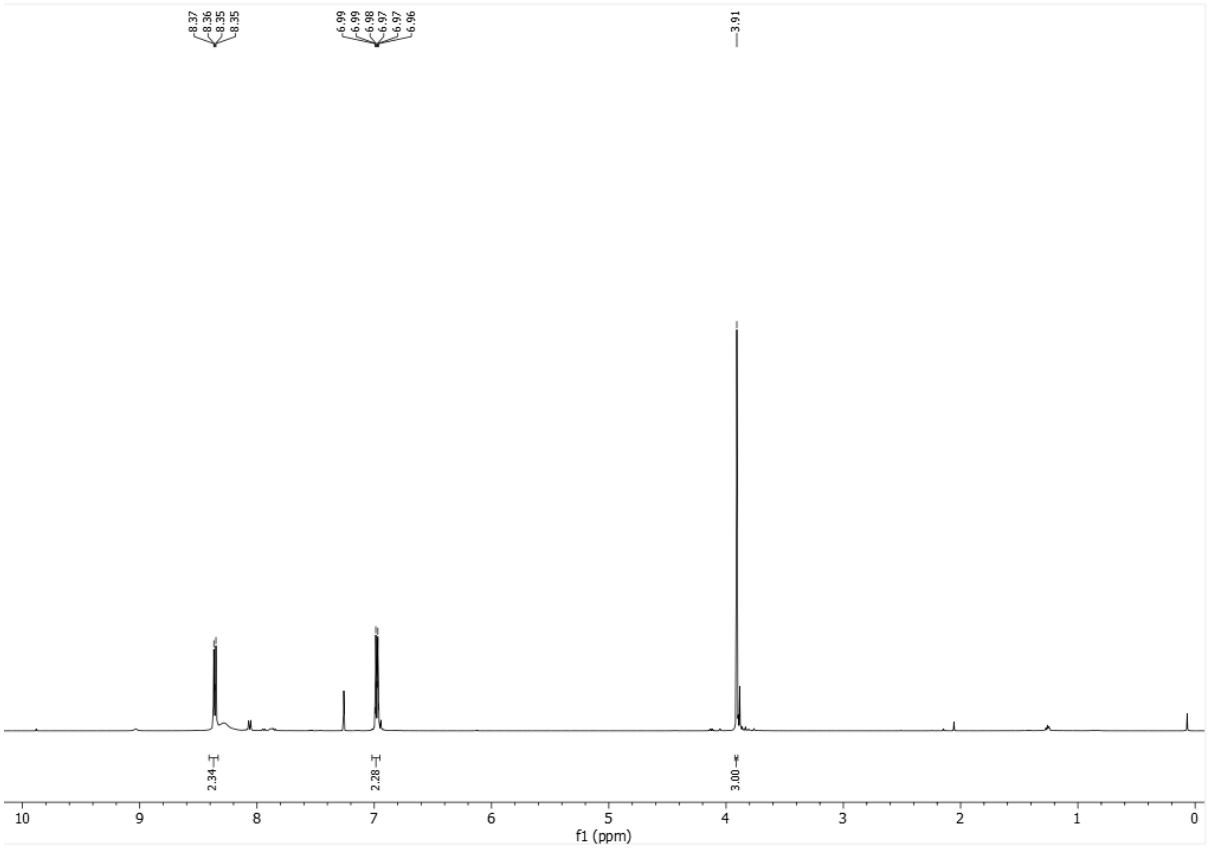


Figure **S94**. ^{1}H NMR spectrum of 2-(4-methoxyphenyl)-2-oxoacetic acid in $CDCl_3$.

45, ^{13}C, $CDCl_3$, 126 MHz

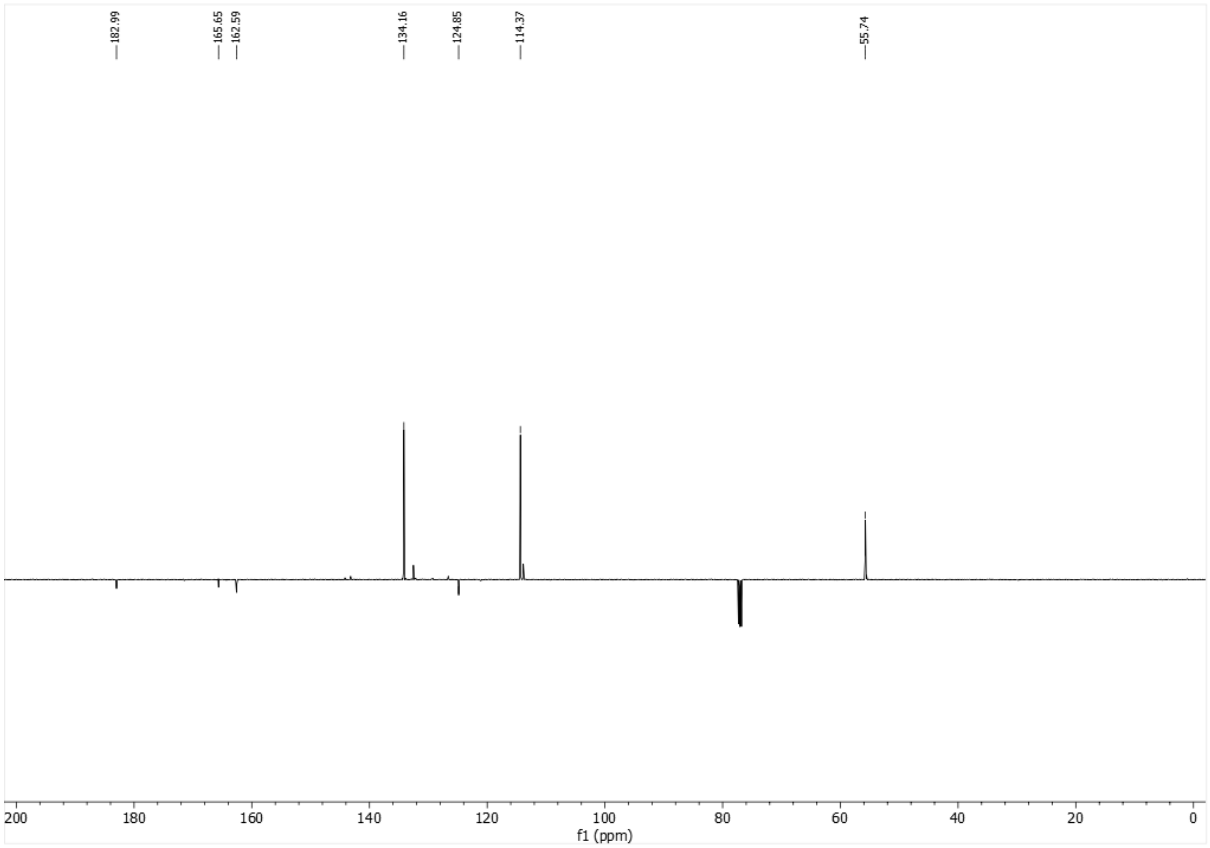
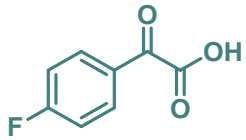
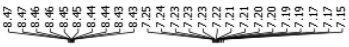


Figure **S95**. ^{13}C NMR spectrum of 2-(4-methoxyphenyl)-2-oxoacetic acid in $CDCl_3$.



46, ^{1}H, $CDCl_3$, 500 MHz



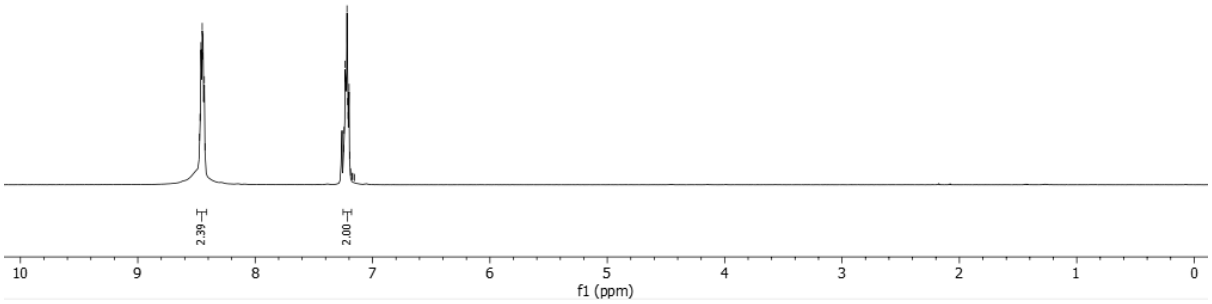


Figure **S96**. ^{1}H NMR spectrum of 2-(4-fluorophenyl)-2-oxoacetic acid in $CDCl_3$.

46, ^{19}F, $CDCl_3$, 471 MHz

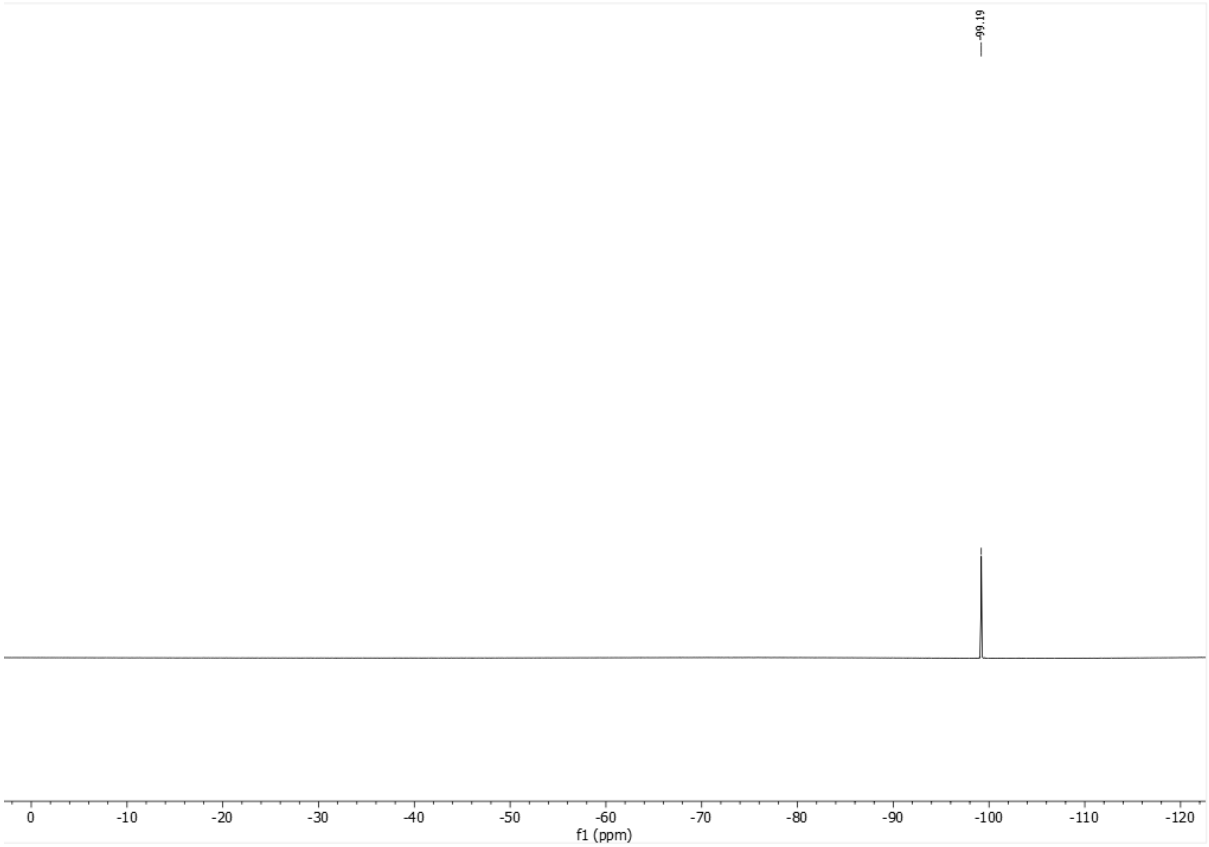


Figure **S97**. ^{19}F NMR spectrum of 2-(4-fluorophenyl)-2-oxoacetic acid in $CDCl_3$.

46, ^{13}C, $CDCl_3$, 126 MHz

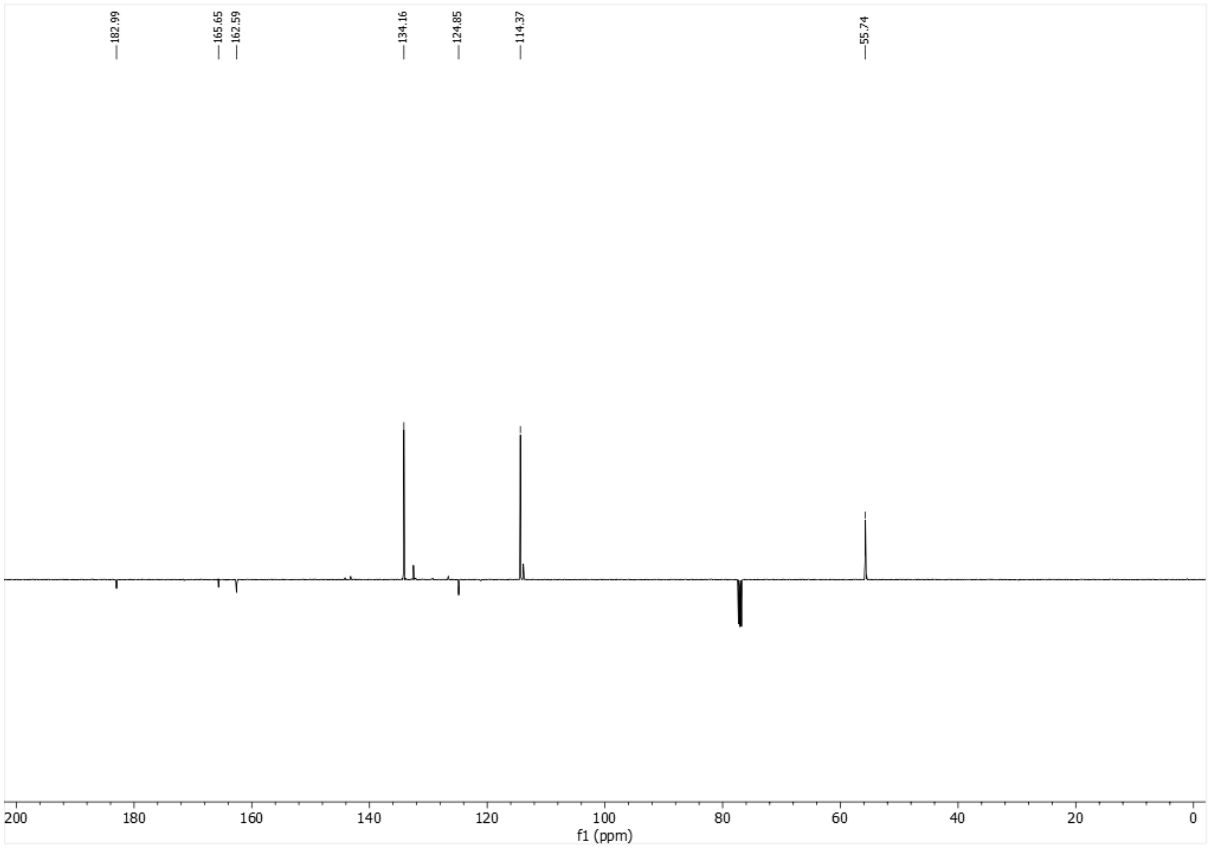
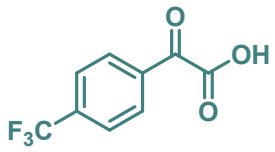


Figure **S98**. ^{13}C NMR spectrum of 2-(4-fluorophenyl)-2-oxoacetic acid in $CDCl_3$.



47, ^{1}H, $CDCl_3$, 500 MHz

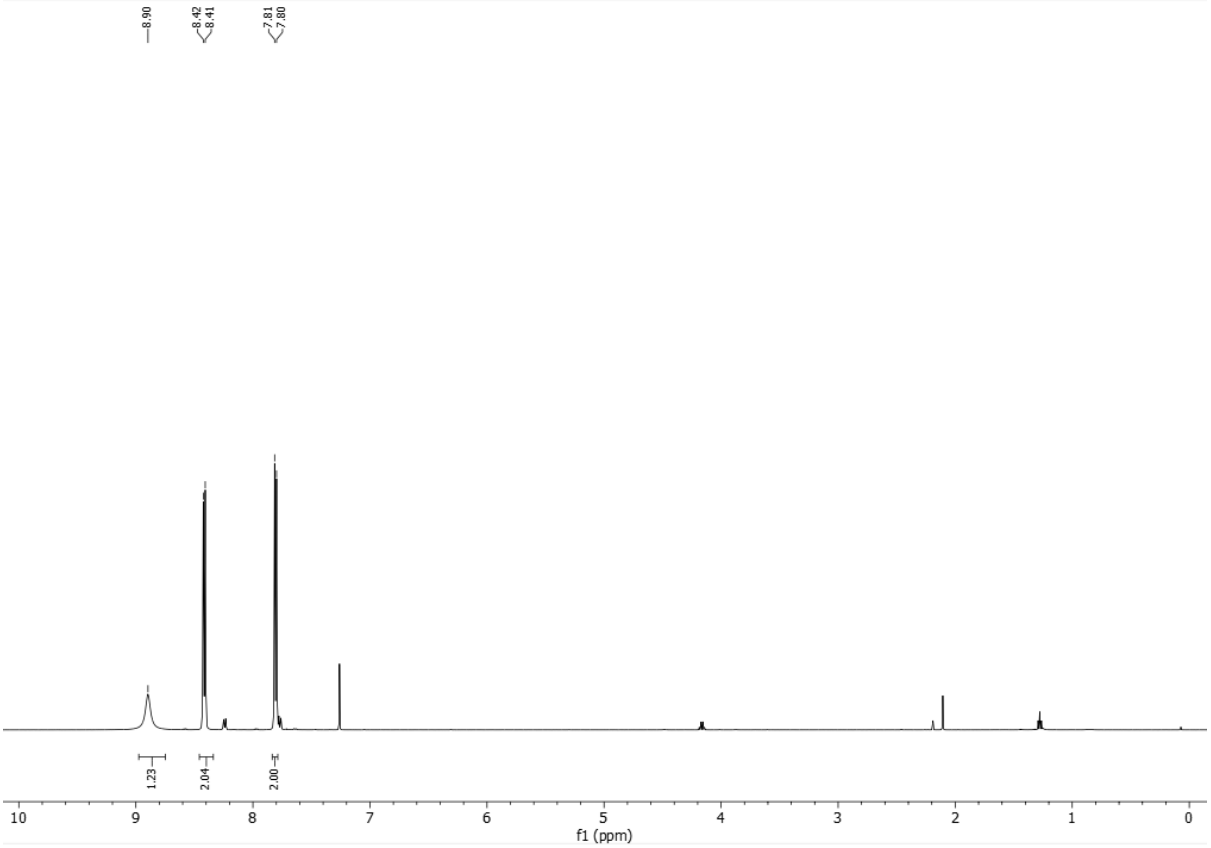


Figure **S99**. ^{1}H NMR spectrum of 2-(4-(trifluoromethyl)phenyl)-2-oxoacetic acid in $CDCl_3$.

47, ^{13}C, $CDCl_3$, 126 MHz

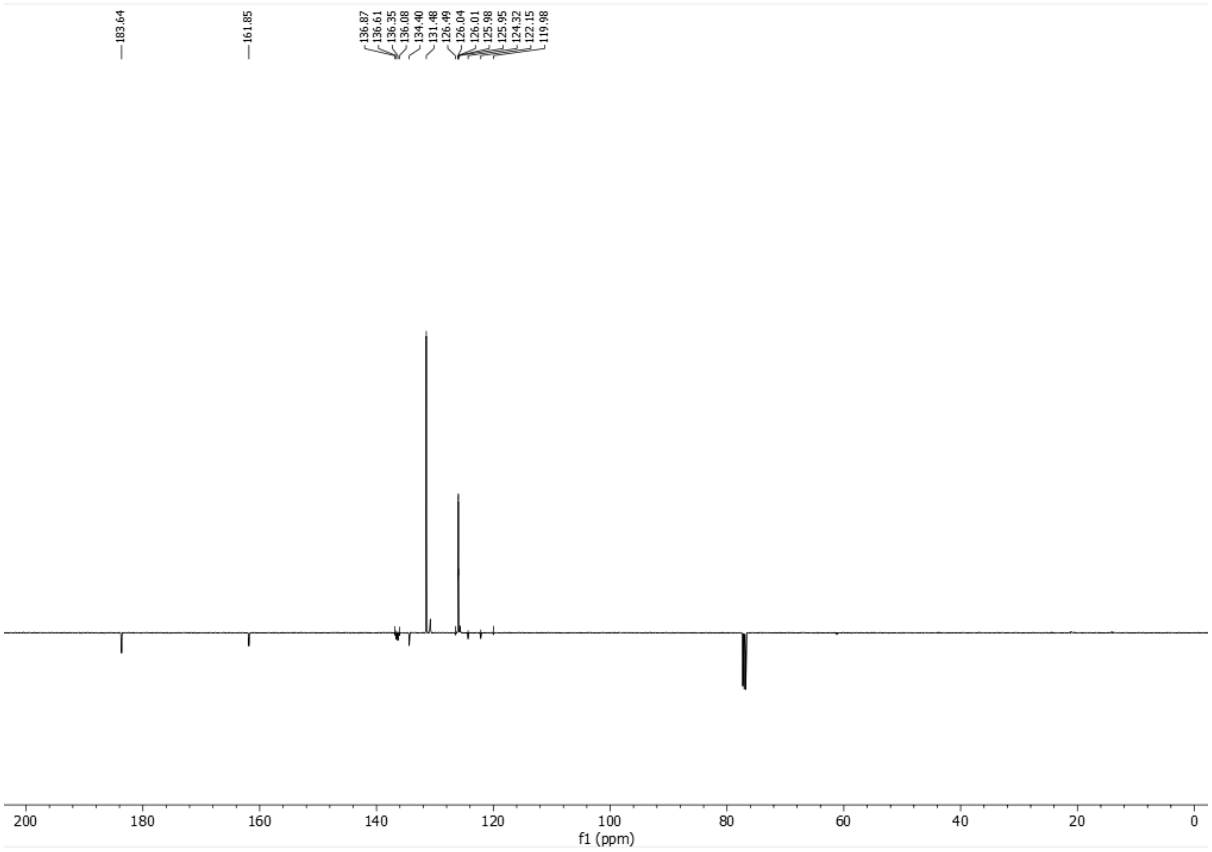
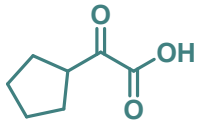


Figure **S100**. ^{13}C NMR spectrum of 2-(4-(trifluoromethyl)phenyl)-2-oxoacetic acid in $CDCl_3$.



48, ^{1}H, $CDCl_3$, 500 MHz

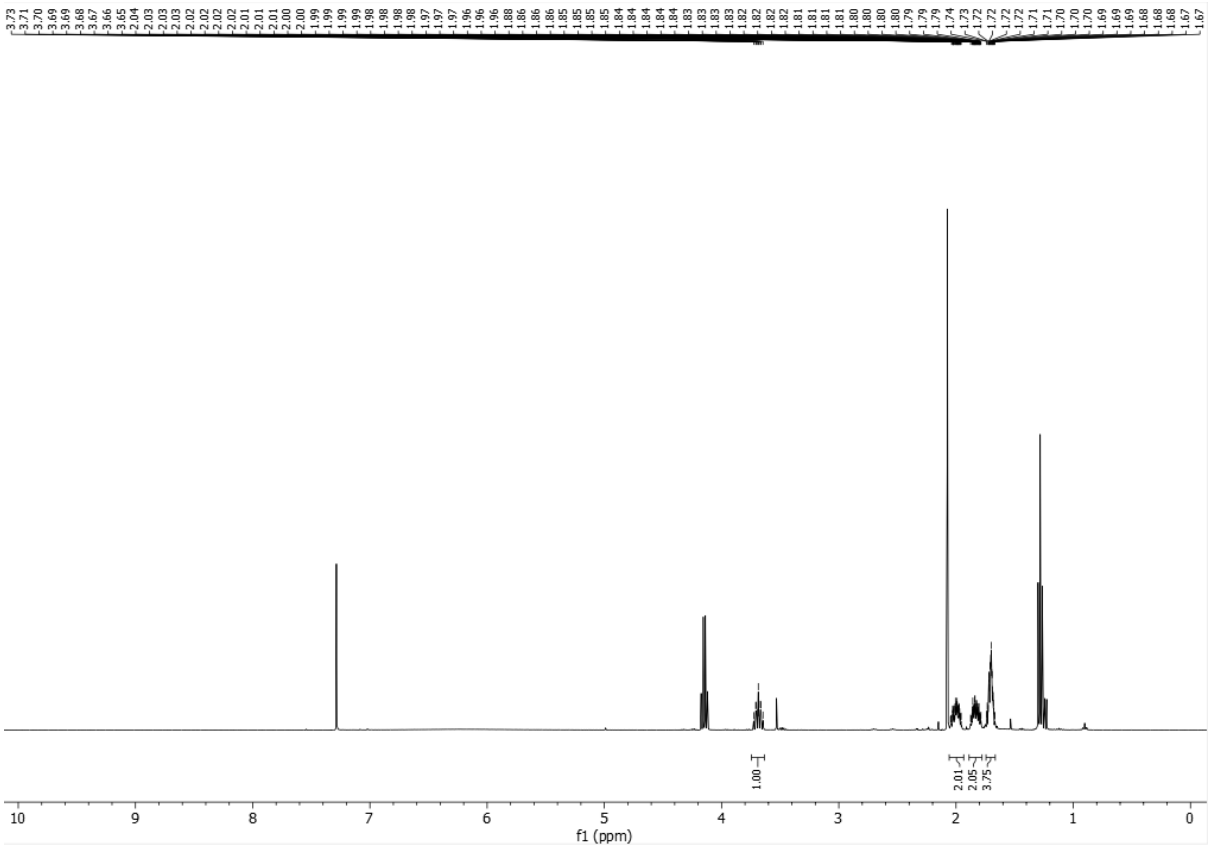


Figure **S101**. ^{1}H NMR spectrum of 2-cyclopentyl-2-oxoacetic acid in $CDCl_3$.

48, ^{13}C, $CDCl_3$, 126 MHz

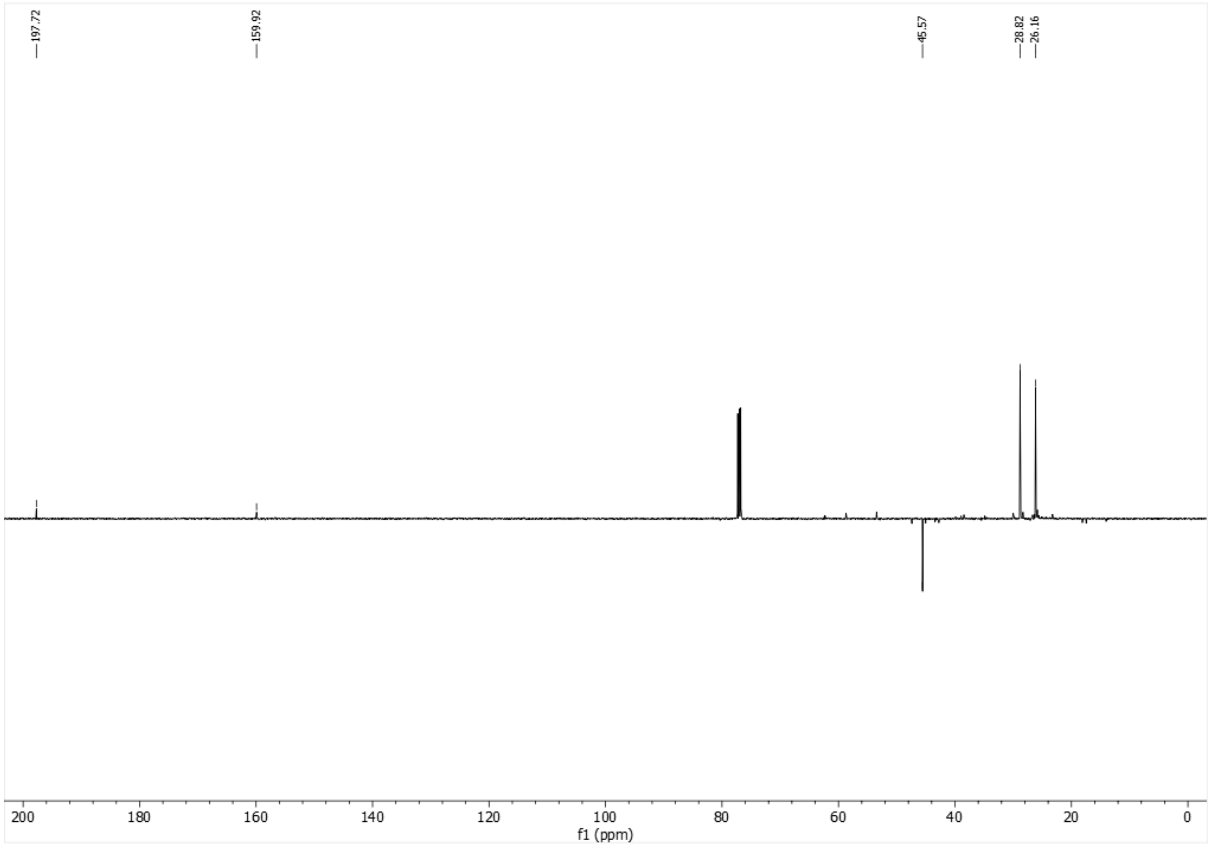


Figure **S102**. ^{13}C NMR spectrum of 2-cyclopentyl-2-oxoacetic acid in $CDCl_3$.