

Supporting Information

I₂-DMSO Mediated Oxidative Amidation of Methyl Ketones With Anthranils for the Synthesis of α -Ketoamides

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1. General

All of the substrates and reagents were commercially available and used without further purification unless otherwise noted. TLC analysis was performed using pre-coated glass plates. Flash column chromatography was performed on silica gel (200–300 mesh). ¹H NMR spectra was determined at 25 °C on a Varian Mercury 600 MHz spectrometer. Chemical shifts were provided in ppm relative to the internal standard of tetramethylsilane (TMS). ¹³C NMR spectra was recorded in CDCl₃ or DMSO-*d*₆ on 150 MHz NMR spectrometers and resonances (δ) in ppm. The data is being reported as s=singlet, d=doublet, t=triplet, m=multiplet or unresolved coupling constant(s) in Hz, integration. HRMS were obtained on Bruker 7-tesla FT-ICR MS equipped with an electrospray source. Melting points were determined by using an electrothermal capillary melting point apparatus and not corrected. The X-ray crystal-structures were obtained on a Bruker APEX DUO CCD system.

2. Experimental procedures

2.1 General procedure for the synthesis of 2 (2a as an example)

General procedure: A round-bottom flask equipped with a magnetic stirring bar was charged with 2-nitrobenzaldehyde (1.51g, 10 mmol) and SnCl₂ dihydrate (6.75g, 30 mmol) at room temperature, and solvent (methanol/ EtOAc=1:1, 20 mL) was added. The resulting mixture was stirred at room temperature for 24 h. After the reaction completed, the mixture was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 20:1) to give the desired product **2a** as colorless oil in quantitative yield.

2.2 General procedure for the synthesis of 3 and 4 (3a as an example)

General procedure: A sealed tube equipped with a magnetic stirring bar was charged with acetophenone (**1a**) (60 mg, 0.5 mmol), anthranil (**2a**) (59.5 mg, 0.5 mmol), iodine (2 mg, 0.8 mmol) and TfOH (37.5 mg, 0.25 mmol) at room temperature, and DMSO (2 mL) was added. The resulting mixture was stirred at 140 °C for 4h. After the reaction completed, the mixture was quenched with saturation Na₂S₂O₃ solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 20:1) to yield the desired product **3a** as a yellow solid.

2.3 Procedure for the synthesis of 6

A 25 ml Schlenk tube equipped with a magnetic stirring bar was charged with a mixture of anthranil **2a** (71.4 mg, 0.6 mmol), phenylglyoxylic acid (180 mg, 1.2 mmol), CuBr₂ (6.6 mg, 0.03 mmol) and PPh₃ (31 mg, 0.12 mmol). Under reduced pressure, the tube was filled with argon for three times. After the addition of DCE (4 mL), the reaction was stirred at 110 °C for 12 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 20:1) to yield the desired product **6** (121mg, 0.48 mmol) as a yellow solid in 80% yield.

2.4 Procedure for the synthesis of 7

A 25 ml Schlenk tube equipped with a magnetic stirring bar was charged with a mixture of **3a** (75.8 mg, 0.2 mmol), CuCN (21.6 mg, 0.24 mmol). Under reduced pressure, the tube was filled with

argon for three times. After the addition of DMF (2 mL), the reaction was stirred at 150 °C for 5 h. After the reaction completed, the mixture was quenched with saturation NaCl solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 8:1) to yield the desired product **7** (33.4 mg, 0.12 mmol) as a purple solid in 60% yield.

2.5 Procedure for the synthesis of **9**

A 25 ml Schlenk tube equipped with a magnetic stirring bar was charged with a mixture of **3a** (75.8 mg, 0.2 mmol), **8** (30.4 mg, 0.2 mmol), Pd(PPh₃)₂Cl₂ (14.0 mg, 0.02 mmol), K₂CO₃ (82.8 mg, 0.6 mmol). Under reduced pressure, the tube was filled with argon for three times. After the addition of THF (2 mL), the reaction was stirred at 60 °C for 12 h. After the reaction completed, the mixture was quenched with saturation NaCl solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 10:1) to yield the desired product **9** (66.1 mg, 0.184 mmol) as a yellow solid in 92% yield.

2.6 Procedure for the synthesis of **11**

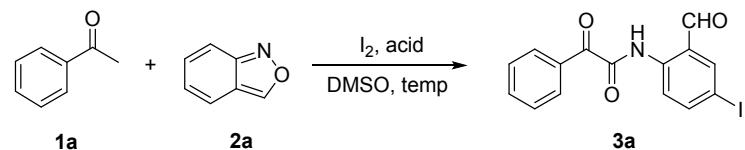
A 25 ml Schlenk tube equipped with a magnetic stirring bar was charged with a mixture of **3a** (75.8 mg, 0.2 mmol), **10** (31.7 mg, 0.24 mmol), Pd(PPh₃)₂Cl₂ (7.0 mg, 0.01 mmol), CuI (3.8 mg, 0.02 mmol). Under reduced pressure, the tube was filled with argon for three times. After the addition of DMF (1 mL) and Et₃N (2 mL), the reaction was stirred at room temperature for 12 h. After the reaction completed, the mixture was quenched with saturation NaCl solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 10:1) to yield the desired product **9** (64.3 mg, 0.168 mmol) as a yellow solid in 84% yield.

2.7 Procedure for the synthesis of **13**

A sealed tube equipped with a magnetic stirring bar was charged with **3a** (189.5 mg, 0.5 mmol), K₂CO₃ (138.2 mg, 1 mmol) at room temperature, and dry methanol (5 mL) was added. Bestmann-Ohira reagent (115.2 mg, 0.6 mmol) was added to the solution and stirred at room temperature for

4h. After the reaction completed, the mixture was quenched with an aqueous solution of NaHCO₃ (5%, 50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 10:1) to yield the desired product **13** (108.7 mg, 0.29 mmol) as a yellow solid in 58% yield.

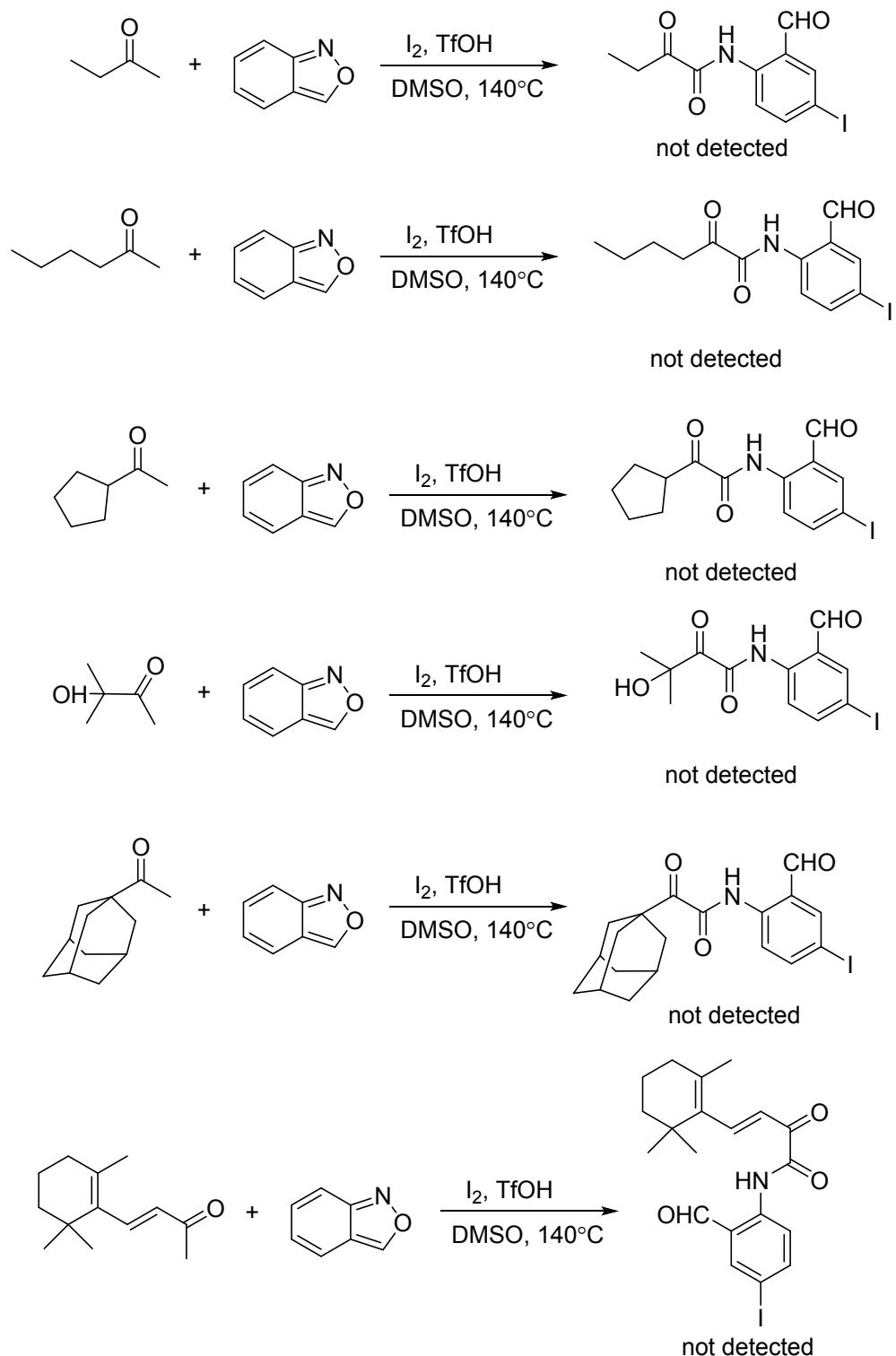
3. Optimization of the Standard Conditions



entry	acid (equiv.)	I ₂ (equiv.)	solvent	temp (°C)	yield (%)
1	TfOH (0.5)	1.6	DMF	140	ND
2	TfOH (0.5)	1.6	Toluene	110	ND
3	TfOH (0.5)	1.6	CH ₃ CN	80	ND
4	TfOH (0.5)	1.6	THF	60	ND
5	TfOH (0.5)	1.6	CH ₂ Cl ₂	rt	ND
6	TfOH (0.5)	1.6	DCE	80	ND
7	TfOH (0.5)	1.6	1,4-Dioxane	100	ND
8	TfOH (0.5)	1.6	EtOH	80	ND

4. Substrate scope of aliphatic ketones

We have used some aliphatic ketones to react with anthranils under the optimized reaction conditions. However, the aliphatic ketones were not compatible with this transformation. The obstacle to this conversion is that aliphatic ketones might not undergo Kornblum oxidation to obtain corresponding ketoaldehydes intermediates.



5. GC-MS of intermediates

5.1 GS-MS of the intermediate 5 and 5a

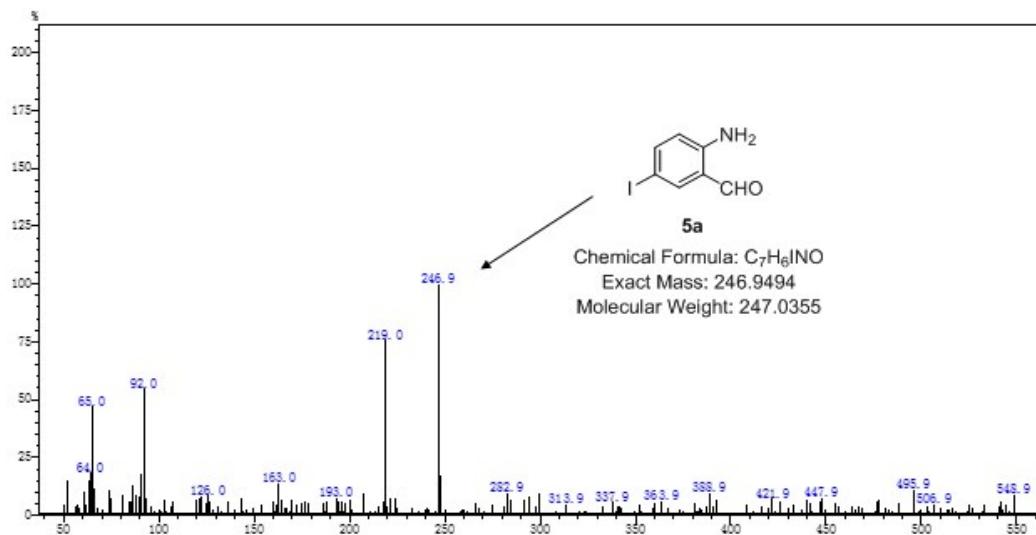
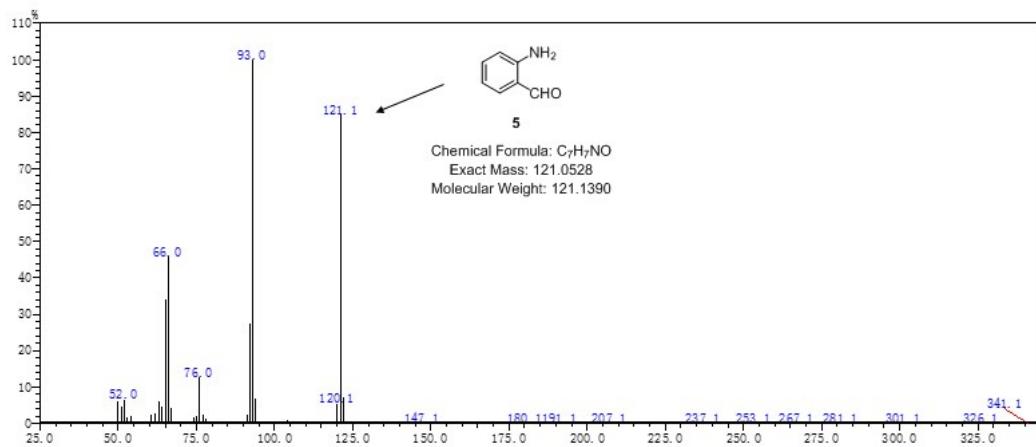
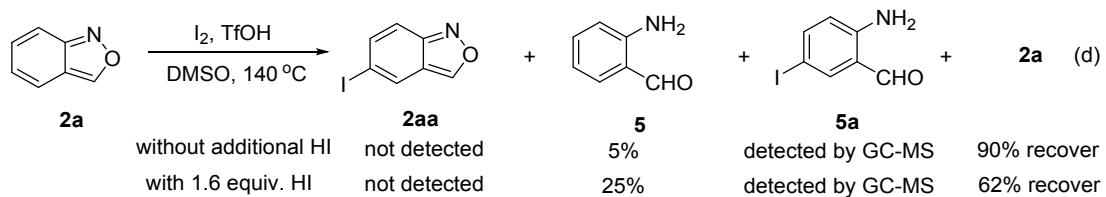


Figure S1. GS-MS of the intermediate **5** and **5a**

5.2 GS-MS of the intermediate **5a**

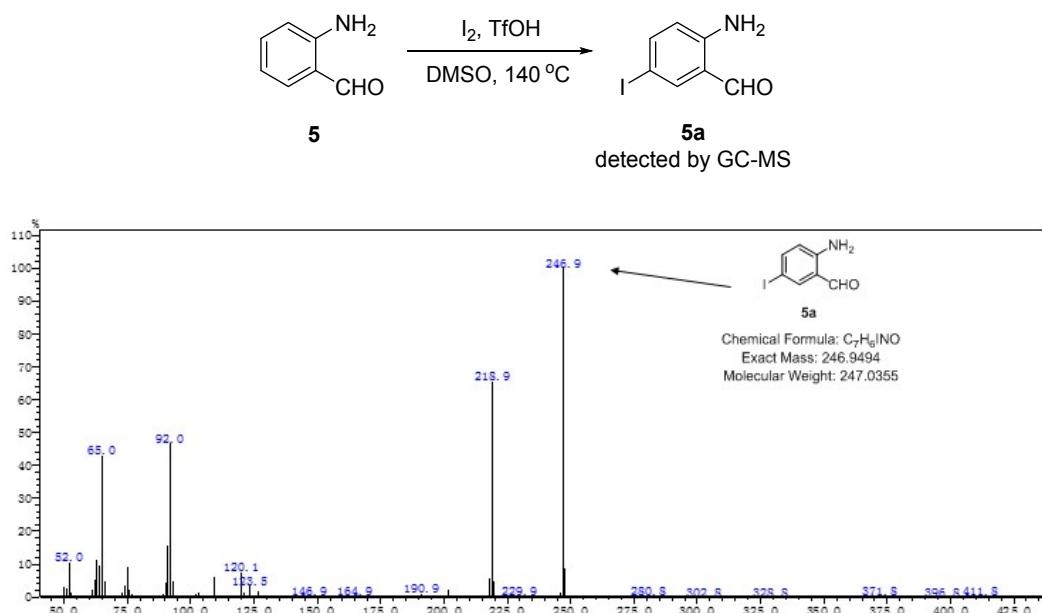
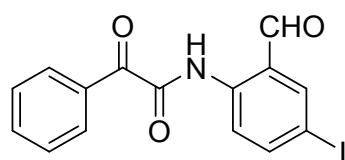
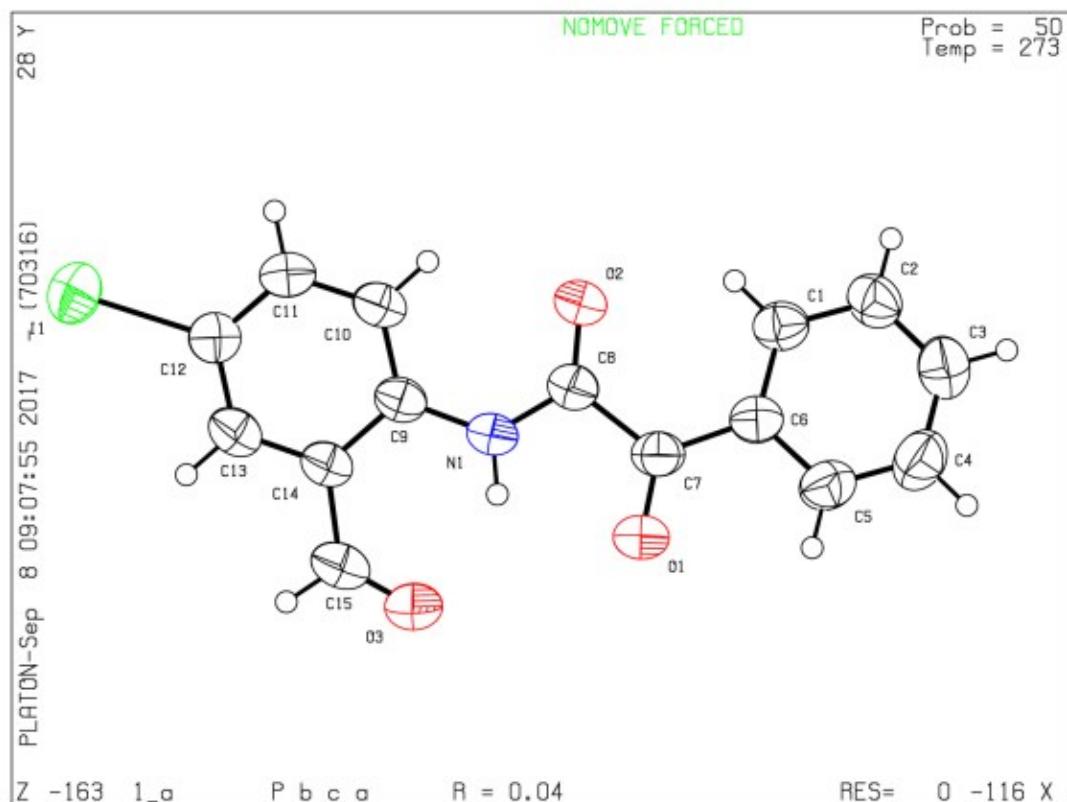


Figure S2. GS-MS of the intermediate **5a**

6. The crystallographic data



3a

Figure S2. X-ray crystal structure of **3a**

Crystal Data for Compound **3a**: CCDC 2026166 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic.

Bond precision: C-C = 0.0053 Å Wavelength=0.71073

Cell: a=7.7449(12) b=16.862(3) c=21.490(3)
alpha=90 beta=90 gamma=90

Temperature: 273 K

	Calculated	Reported
Volume	2806.5(8)	2806.3(7)
Space group	P b c a	P b c a
Hall group	-P 2ac 2ab	-P 2ac 2ab
Moiety formula	C15 H10 I N O3	?
Sum formula	C15 H10 I N O3	C15 H10 I N O3
Mr	379.14	379.14
Dx, g cm ⁻³	1.795	1.795
Z	8	8
Mu (mm ⁻¹)	2.288	2.288
F000	1472.0	1472.0
F000'	1468.83	
h, k, lmax	11,24,30	11,24,30
Nref	4335	4317
Tmin, Tmax	0.611, 0.662	0.864, 0.864
Tmin'	0.599	

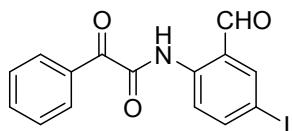
Correction method= # Reported T Limits: Tmin=0.864 Tmax=0.864
AbsCorr = MULTI-SCAN

Data completeness= 0.996 Theta(max)= 30.659

R(reflections)= 0.0398(2430) wR2(reflections)= 0.1349(4317)

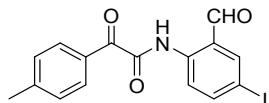
S = 0.992 Npar= 181

7. Spectroscopic data



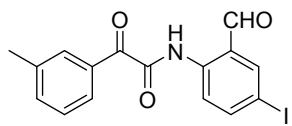
3a

N-(2-formyl-4-iodophenyl)-2-oxo-2-phenylacetamide (**3a**): Yellow solid; 147.8 mg (yield 78%); mp 139–141 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.08 (s, 1H), 9.97 (d, *J* = 7.2 Hz, 1H), 8.30 (d, *J* = 2.4 Hz, 2H), 8.20 (d, *J* = 7.8 Hz, 2H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.78–7.70 (m, 1H), 7.64–7.52 (m, 2H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.6, 186.7, 160.7, 143.7, 143.2, 137.8, 134.6, 132.8, 130.9, 128.6, 125.5, 122.2, 88.3; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₉INO₃⁻: 377.9633, found: 377.9635.



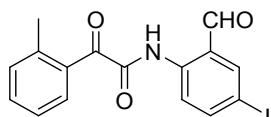
3b

N-(2-formyl-4-iodophenyl)-2-oxo-2-(p-tolyl)acetamide (**3b**): Yellow solid; 147.4 mg (yield 75%); mp 137–139 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.35 (s, 1H), 9.89 (s, 1H), 8.59 (d, *J* = 9.0 Hz, 1H), 8.28 (d, *J* = 7.2 Hz, 2H), 8.00 (s, 1H), 7.89 (d, *J* = 8.4 Hz, 1H), 7.28 (d, *J* = 7.8 Hz, 2H), 2.43 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 194.0, 193.5, 185.4, 160.5, 145.8, 144.2, 138.6, 131.4, 129.2, 124.3, 121.8, 121.7, 86.4, 21.9; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₃⁻: 391.9789, found: 391.9792.



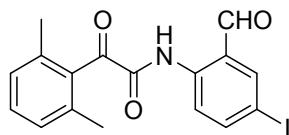
3c

N-(2-formyl-4-iodophenyl)-2-oxo-2-(m-tolyl)acetamide (**3c**): Yellow solid; 137.6 mg (yield 70%); mp 88–89 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.05 (s, 1H), 9.93 (s, 1H), 8.33–8.20 (m, 2H), 8.07–7.92 (m, 3H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 1H), 2.37 (s, 3H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.6, 186.4, 160.5, 143.6, 143.3, 137.9, 137.7, 135.2, 132.6, 131.0, 128.4, 128.1, 125.2, 121.9, 88.1, 20.9; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₃⁻: 391.9789, found: 391.9788.



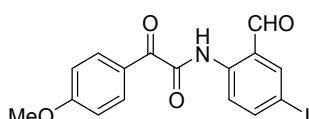
3d

N-(2-formyl-4-iodophenyl)-2-oxo-2-(o-tolyl)acetamide (**3d**): Yellow solid; 143.4 mg (yield 73%); mp 133-135 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.39 (s, 1H), 9.91 (s, 1H), 8.61 (d, *J* = 8.4 Hz, 1H), 8.02 (s, 1H), 7.98-7.84 (m, 2H), 7.47 (t, *J* = 7.2 Hz, 1H), 7.35-7.19 (m, 2H), 2.54 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 193.6, 189.5, 160.6, 144.3, 144.2, 140.5, 138.6, 132.9, 132.0, 131.9, 131.8, 125.2, 124.3, 121.7, 86.5, 21.0; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₃⁻: 391.9789, found: 391.9792.



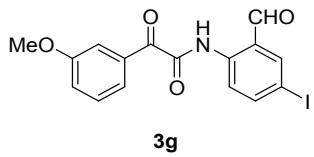
3e

2-(2,6-dimethylphenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (**3e**): Yellow solid; 132.3 mg (yield 65%); mp 184-186 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.05 (s, 1H), 9.97 (d, *J* = 9.2 Hz, 1H), 8.40-8.21 (m, 2H), 8.06 (d, *J* = 8.8 Hz, 1H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.28-7.08 (m, 2H), 2.44 (s, 3H), 2.34 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 194.5, 189.5, 161.2, 143.7, 143.6, 143.1, 139.9, 137.9, 132.6, 132.3, 129.3, 126.0, 125.6, 122.2, 88.3, 21.2, 20.5; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₇H₁₃INO₃⁻: 405.9946, found: 405.9937.

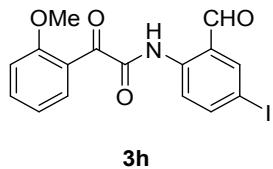


3f

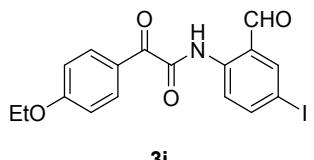
N-(2-formyl-4-iodophenyl)-2-(4-methoxyphenyl)-2-oxoacetamide (**3f**): Yellow solid; 143.2 mg (yield 70%); mp 184-186 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.40 (s, 1H), 9.93 (s, 1H), 8.64 (d, *J* = 8.4 Hz, 1H), 8.45 (d, *J* = 8.4 Hz, 2H), 8.04 (s, 1H), 7.94 (d, *J* = 9.0 Hz, 1H), 6.98 (d, *J* = 9.6 Hz, 2H), 3.91 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 194.5, 184.7, 164.4, 161.2, 143.7, 143.2, 137.8, 133.5, 125.5, 125.4, 122.2, 114.1, 88.2, 55.8; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₄⁻: 407.9738, found: 407.9741.



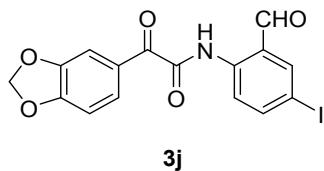
N-(2-formyl-4-iodophenyl)-2-(3-methoxyphenyl)-2-oxoacetamide (3g): Yellow solid; 141.1 mg (yield 69%); mp 113-115 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.36 (s, 1H), 9.91 (s, 1H), 8.61 (d, *J* = 8.4 Hz, 1H), 8.03 (s, 2H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.85 (s, 1H), 7.41 (t, *J* = 7.8 Hz, 1H), 7.20 (d, *J* = 6.0 Hz, 1H), 3.87 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 193.6, 185.9, 160.4, 159.5, 144.3, 144.2, 138.6, 133.9, 129.6, 124.4, 124.2, 121.8, 121.6, 114.7, 86.5, 55.4.; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₄⁻: 407.9738, found: 407.9741.



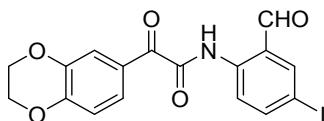
N-(2-formyl-4-iodophenyl)-2-(2-methoxyphenyl)-2-oxoacetamide (3h): Yellow solid; 132.9 mg (yield 65%); mp 184-186 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.39 (s, 1H), 9.92 (s, 1H), 8.64 (d, *J* = 8.4 Hz, 1H), 8.45 (d, *J* = 8.4 Hz, 2H), 8.03 (s, 1H), 7.94 (d, *J* = 8.4 Hz, 1H), 6.98 (d, *J* = 8.4 Hz, 2H), 3.91 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 193.5, 184.2, 164.8, 161.1, 144.3, 138.7, 134.1, 134.0, 125.8, 124.5, 122.0, 121.9, 121.8, 113.9, 86.3, 55.6; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₄⁻: 407.9738, found: 407.9741.



2-(4-ethoxyphenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3i): Yellow solid; 137.5 mg (yield 65%); mp 165-167 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.39 (s, 1H), 9.92 (s, 1H), 8.63 (d, *J* = 8.4 Hz, 1H), 8.43 (d, *J* = 7.8 Hz, 2H), 8.03 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 6.95 (d, *J* = 7.8 Hz, 2H), 4.14 (d, *J* = 6.6 Hz, 2H), 1.46 (t, *J* = 6.6 Hz, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 193.5, 184.1, 164.3, 161.1, 144.3, 138.8, 134.0, 125.6, 124.5, 121.9, 114.3, 103.8, 86.3, 63.9, 14.6; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₇H₁₃INO₄⁻: 421.9895, found: 421.9897.

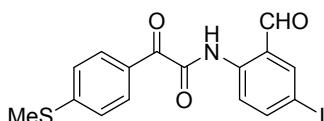


2-(benzo[d][1,3]dioxol-5-yl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3j): Yellow solid; 129.0 mg (yield 61%); mp 202-204 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.05 (s, 1H), 9.95 (s, 1H), 8.40-8.23 (m, 2H), 8.12-7.94 (m, 2H), 7.66 (s, 1H), 7.11 (d, *J*=8.4 Hz, 1H), 6.19 (s, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 194.5, 184.4, 161.1, 153.0, 147.8, 143.7, 143.1, 137.8, 128.7, 127.0, 125.6, 122.4, 109.4, 108.3, 102.5, 88.3; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₉INO₅⁻: 421.9531, found: 421.9533.



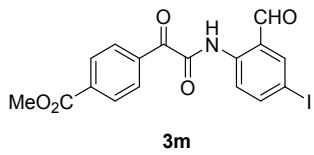
3k

2-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3k): Yellow solid; 128.9 mg (yield 59%); mp 188-190 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.05 (s, 1H), 9.93 (s, 1H), 8.27 (d, *J*=5.6 Hz, 2H), 8.03 (d, *J*=8.8 Hz, 1H), 7.86-7.69 (m, 2H), 7.00 (d, *J*=8.4 Hz, 1H), 4.33 (d, *J*=24.4 Hz, 4H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 194.6, 184.3, 160.9, 149.5, 143.7, 143.3, 143.0, 137.8, 125.9, 125.4, 122.1, 119.9, 117.3, 88.2, 64.8, 63.9; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₇H₁₁INO₅⁻: 435.9687, found: 435.9687.

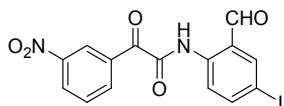


3l

N-(2-formyl-4-iodophenyl)-2-(4-(methylthio)phenyl)-2-oxoacetamide (3l): Yellow solid; 131.8 mg (yield 62%); mp 181-183 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.08 (s, 1H), 9.96 (s, 1H), 8.31 (d, *J*=11.4 Hz, 2H), 8.17 (d, *J*=7.8 Hz, 2H), 8.08 (d, *J*=8.4 Hz, 1H), 7.41 (d, *J*=7.8 Hz, 2H), 2.56 (s, 3H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.6, 185.3, 161.0, 148.0, 143.7, 143.2, 137.8, 131.3, 128.7, 125.5, 124.6, 122.3, 88.3, 13.8; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₃S⁻: 423.9510, found: 423.9506.

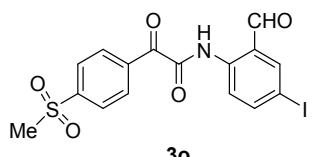


methyl 4-((2-formyl-4-iodophenyl)amino)-2-oxoacetylbenzoate (3m): Yellow solid; 142.0 mg (yield 65%); mp 207-209 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.15 (s, 1H), 9.99 (s, 1H), 8.37-8.27 (m, 4H), 8.14-8.07 (m, 3H), 3.91 (s, 3H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.8, 186.2, 165.5, 160.2, 143.8, 143.4, 137.8, 136.6, 133.9, 131.2, 129.1, 125.5, 122.2, 88.3, 52.6; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₇H₁₁INO₅⁻: 435.9687, found: 435.9690.

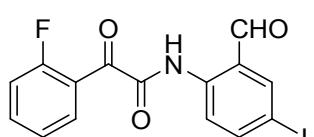


3n

N-(2-formyl-4-iodophenyl)-2-(3-nitrophenyl)-2-oxoacetamide (4n): Yellow solid; 127.2 mg (yield 60%); mp 205-207 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.22 (s, 1H), 10.00 (s, 1H), 9.04 (s, 1H), 8.55 (t, *J* = 8.4 Hz, 2H), 8.37 (d, *J* = 13.2 Hz, 2H), 8.10 (d, *J* = 8.4 Hz, 1H), 7.87 (t, *J* = 7.8 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 195.0, 184.3, 159.6, 147.3, 143.8, 143.6, 137.8, 136.8, 134.3, 130.2, 128.2, 125.7, 125.3, 122.0, 88.2; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₈IN₂O₅⁻: 422.9483, found: 422.9486.



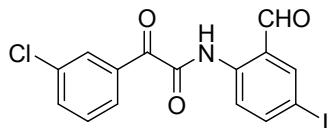
N-(2-formyl-4-iodophenyl)-2-(4-(methylsulfonyl)phenyl)-2-oxoacetamide (3o): Yellow solid; 144.0 mg (yield 63%); mp 226-228 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.39 (s, 1H), 9.94 (s, 1H), 8.65 (d, *J* = 9.0 Hz, 1H), 8.46 (d, *J* = 8.4 Hz, 2H), 8.04 (s, 1H), 7.95 (d, *J* = 7.8 Hz, 1H), 6.99 (d, *J* = 8.4 Hz, 2H), 3.92 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 194.9, 185.8, 159.8, 144.8, 143.8, 143.5, 137.8, 137.0, 131.7, 126.9, 125.4, 122.1, 88.3, 43.1; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₁INO₅S⁻: 455.9408, found: 455.9411.



3p

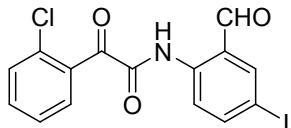
2-(2-fluorophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3p): Yellow solid; 139.0 mg

(yield 70%); mp 98-100 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.26 (s, 1H), 9.94 (s, 1H), 8.61 (d, *J* = 9.0 Hz, 1H), 8.06 (d, *J* = 1.8 Hz, 1H), 7.96 (d, *J* = 9.0 Hz, 1H), 7.90 (t, *J* = 6.6 Hz, 1H), 7.65-7.59 (m, 1H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.20 (t, *J* = 9.6 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 193.7, 186.9, 160.0, 144.5, 144.3, 138.6, 135.7, 131.9, 124.3, 122.4, 122.1, 116.7, 116.5, 108.6, 86.8; ¹⁹F NMR (376 MHz, CDCl₃) δ -109.45; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₈FINO₃⁻: 395.9538, found: 395.9541.



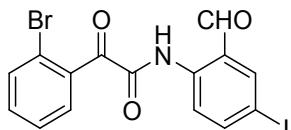
3q

2-(3-chlorophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (**3q**): Yellow solid; 144.9 mg (yield 70%); mp 165-167 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.12 (s, 1H), 9.97 (s, 1H), 8.32 (d, *J* = 8.4 Hz, 2H), 8.22 (s, 1H), 8.13 (d, *J* = 7.6 Hz, 1H), 8.07 (d, *J* = 8.4 Hz, 1H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 8.0 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 195.3, 185.6, 160.4, 144.2, 143.9, 138.2, 135.2, 134.3, 133.6, 131.0, 130.9, 129.9, 125.8, 122.6, 88.7; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₈ClINO₃⁻: 411.9243, found: 411.9237.



3r

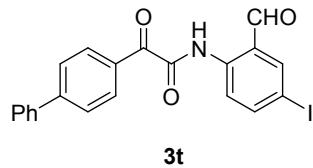
2-(2-chlorophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (**3r**): Yellow solid; 157.3 mg (yield 76%); mp 155-157 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.34 (s, 1H), 9.94 (s, 1H), 8.59 (d, *J* = 9.0 Hz, 1H), 8.06 (s, 1H), 7.94 (d, *J* = 9.0 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.54-7.44 (m, 2H), 7.41 (t, *J* = 7.2 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 193.7, 189.1, 159.5, 144.4, 144.3, 138.5, 133.3, 133.1, 131.1, 130.3, 126.7, 124.4, 122.0, 121.9, 86.8; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₈ClINO₃⁻: 411.9243, found: 411.9245.



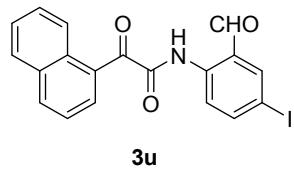
3s

2-(2-bromophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (**3s**): Yellow solid; 164.9 mg (yield 72%); mp 155-157 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.35 (s, 1H), 9.92 (s, 1H), 8.56 (d, *J*

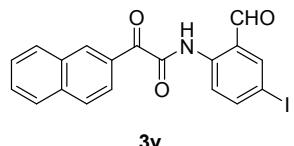
= 9.0 Hz, 1H), 8.04 (s, 1H), 7.91 (d, J = 7.2 Hz, 1H), 7.64 (t, J = 6.6, 2H), 7.48–7.35 (m, 2H); ^{13}C NMR (150 MHz, CDCl_3) δ 193.7, 189.5, 159.0, 144.3, 144.1, 138.4, 135.5, 133.3, 133.1, 131.0, 127.2, 124.3, 121.8, 120.8, 86.8; HRMS (ESI): m/z [M-H] $^-$ calcd for $\text{C}_{15}\text{H}_8\text{BrINO}_3^-$: 455.8738, found: 455.8741.



2-((1,1'-biphenyl)-4-yl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3t): Yellow solid; 125.1 mg (yield 55%); mp 124–126 °C; ^1H NMR (600 MHz, $\text{DMSO}-d_6$) δ 12.13 (s, 1H), 9.95 (s, 1H), 8.41–8.21 (m, 4H), 8.03 (d, J = 8.4 Hz, 1H), 7.84 (d, J = 7.8 Hz, 2H), 7.74 (d, J = 7.2 Hz, 2H), 7.49 (t, J = 7.2 Hz, 2H), 7.43 (t, J = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 194.7, 185.9, 160.7, 145.7, 143.8, 143.4, 138.6, 137.8, 131.7, 131.6, 129.2, 128.8, 127.1, 126.7, 125.4, 122.2, 88.3; HRMS (ESI): m/z [M-H] $^-$ calcd for $\text{C}_{21}\text{H}_{13}\text{INO}_3^-$: 453.9946, found: 453.9944.

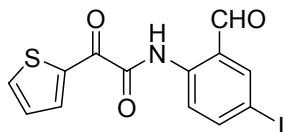


N-(2-formyl-4-iodophenyl)-2-(naphthalen-1-yl)-2-oxoacetamide (3u): Yellow solid; 139.4 mg (yield 65%); mp 148–150 °C; ^1H NMR (600 MHz, CDCl_3) δ 12.55 (s, 1H), 9.93 (s, 1H), 8.65 (t, J = 6.6 Hz, 2H), 8.32 (d, J = 7.2 Hz, 1H), 8.10 (d, J = 7.8 Hz, 1H), 8.03 (s, 1H), 7.92 (t, J = 9.6 Hz, 2H), 7.64 (t, J = 7.2 Hz, 1H), 7.60–7.52 (m, 2H); ^{13}C NMR (150 MHz, CDCl_3) δ 193.7, 188.7, 160.8, 144.3, 138.7, 134.9, 133.8, 133.2, 131.2, 129.0, 128.7, 128.6, 126.7, 125.4, 125.3, 124.4, 124.1, 121.9, 86.6; HRMS (ESI): m/z [M-H] $^-$ calcd for $\text{C}_{19}\text{H}_{11}\text{INO}_3^-$: 427.9789, found: 427.9792.



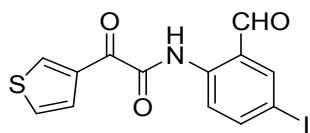
N-(2-formyl-4-iodophenyl)-2-(naphthalen-2-yl)-2-oxoacetamide (3v): Yellow solid; 141.6 mg (yield 66%); mp 148–150 °C; ^1H NMR (600 MHz, CDCl_3) δ 12.40 (s, 1H), 9.85 (s, 1H), 9.14 (s, 1H), 8.60 (d, J = 9.0 Hz, 1H), 8.20 (d, J = 7.8 Hz, 1H), 8.00–7.92 (m, 2H), 7.90–7.80 (m, 3H), 7.63–7.61 (t, J = 7.8 Hz, 1H), 7.53 (t, J = 7.2 Hz, 1H); ^{13}C NMR (150 MHz, CDCl_3) δ 193.6, 185.6, 160.5,

144.2, 138.5, 136.0, 134.9, 134.8, 132.1, 130.3, 130.0, 129.4, 128.4, 127.7, 126.8, 125.3, 124.3, 121.8, 86.5; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₉H₁₁INO₃⁻: 427.9789, found: 427.9792.



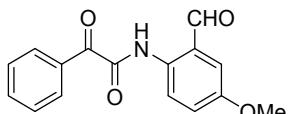
3w

N-(2-formyl-4-iodophenyl)-2-oxo-2-(thiophen-2-yl)acetamide (**3w**): Yellow solid; 115.5 mg (yield 60%); mp 149-151 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.53 (s, 1H), 9.92 (s, 1H), 8.60 (d, *J* = 9.0 Hz, 1H), 8.43 (d, *J* = 3.0 Hz, 1H), 8.03 (s, 1H), 7.92 (d, *J* = 9.0 Hz, 1H), 7.88 (d, *J* = 4.2 Hz, 1H), 7.22 (t, *J* = 4.8 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 193.6, 193.3, 177.1, 159.7, 144.2, 139.0, 138.3, 138.2, 136.0, 128.3, 124.5, 121.9, 86.8. HRMS (ESI): m/z [M-H]⁻ calcd for C₁₃H₇INO₃S⁻: 383.9197, found: 383.9199.



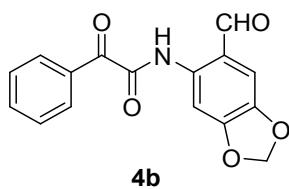
3x

N-(2-formyl-4-iodophenyl)-2-oxo-2-(thiophen-3-yl)acetamide (**3x**): Yellow solid; 121.3 mg (yield 63%); mp 139-141 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.49 (s, 1H), 9.93 (s, 1H), 9.11 (s, 1H), 8.61 (d, *J* = 9.0 Hz, 1H), 8.03 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 4.8 Hz, 1H), 7.38-7.32 (m, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 193.5, 178.8, 160.1, 144.3, 139.4, 138.5, 136.5, 128.9, 125.9, 124.5, 121.8, 86.6; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₃H₇INO₃S⁻: 383.9197, found: 383.9199.

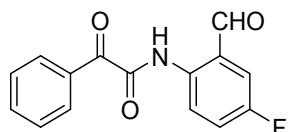


4a

N-(2-formyl-4-methoxyphenyl)-2-oxo-2-phenylacetamide (**4a**): Yellow solid; 82.1 mg (yield 58%); mp 110-112 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.16 (s, 1H), 9.91 (s, 1H), 8.73 (d, *J* = 9.6 Hz, 1H), 8.37 (d, *J* = 7.8 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 3.0 Hz, 1H), 7.18-7.14 (m, 1H), 3.83 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 194.3, 186.7, 159.9, 155.8, 134.2, 133.0, 132.3, 131.1, 128.3, 123.6, 121.5, 121.2, 120.0, 55.5. HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₂NO₄⁻: 282.0772, found: 282.0773.

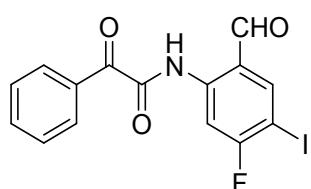


N-(6-formylbenzo[d][1,3]dioxol-5-yl)-2-oxo-2-phenylacetamide (**4b**): Yellow solid; 81.7 mg (yield 55%); mp 163-165 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.31 (s, 1H), 9.82 (s, 1H), 8.22 (d, *J* = 7.8 Hz, 2H), 8.05 (s, 1H), 7.74 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 7.2 Hz, 2H), 7.45 (s, 1H), 6.20 (s, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 193.0, 186.9, 160.7, 152.9, 144.1, 135.9, 134.5, 132.8, 130.8, 128.6, 118.0, 112.5, 102.9, 101.0; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₁₀NO₅⁻: 296.0564, found: 296.0566.



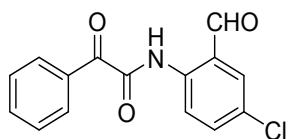
4c

N-(4-fluoro-2-formylphenyl)-2-oxo-2-phenylacetamide (**4c**): Yellow solid; 88.1 mg (yield 65%); mp 140-142 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 11.89 (s, 1H), 10.02 (s, 1H), 8.41-8.34 (m, 1H), 8.20 (d, *J* = 7.2 Hz, 2H), 7.82 (d, *J* = 7.2 Hz, 1H), 7.75 (t, *J* = 7.8 Hz, 1H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 7.8 Hz, 2H); ¹³C NMR (150 MHz, CDCl₃) δ 193.6, 186.5, 160.3, 157.7, 135.4, 134.6, 132.9, 131.2, 128.5, 123.9, 122.8, 122.2, 121.4; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₉FNO₃⁻: 270.0572, found: 270.0573.



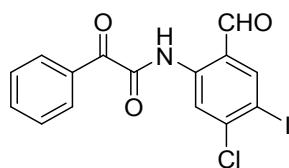
4d

N-(5-fluoro-2-formylphenyl)-2-oxo-2-phenylacetamide (**4d**): Yellow solid; 111.2 mg (yield 56%); mp 124-126 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.28 (s, 1H), 9.91 (s, 1H), 8.44 (d, *J* = 5.4 Hz, 1H), 8.32 (d, *J* = 10.2 Hz, 1H), 8.20 (d, *J* = 7.2 Hz, 2H), 7.72 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 2H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.1, 185.7, 165.6, 163.9, 160.3, 146.7, 140.2, 134.5, 132.6, 130.9, 128.5, 121.8, 106.7, 106.5, 76.0, 75.8; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₈FINO₃⁻: 395.9538, found: 395.9530.



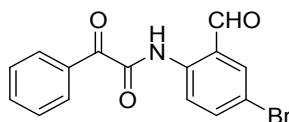
4e

N-(4-chloro-2-formylphenyl)-2-oxo-2-phenylacetamide (**4e**): Yellow solid; 97.9 mg (yield 68%); mp 138-140 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.06 (s, 1H), 9.99 (s, 1H), 8.47 (d, *J* = 8.4 Hz, 1H), 8.20 (d, *J* = 7.8 Hz, 2H), 8.03 (s, 1H), 7.79 (d, *J* = 9.0 Hz, 1H), 7.73 (t, *J* = 7.2 Hz, 1H), 7.58 (t, *J* = 7.2 Hz, 2H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.3, 186.7, 160.8, 137.0, 135.1, 134.6, 134.1, 132.8, 130.8, 128.6, 128.5, 125.3, 122.3; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₉ClNO₃⁻: 286.0276, found: 286.0278.



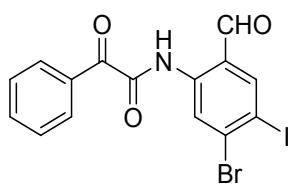
4f

N-(5-chloro-2-formylphenyl)-2-oxo-2-phenylacetamide (**4f**): Yellow solid; 132.5 mg (yield 64%); mp 148-150 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.14 (s, 1H), 9.92 (s, 1H), 8.65 (s, 1H), 8.46 (s, 1H), 8.20 (d, *J* = 7.8 Hz, 2H), 7.73 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 2H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.2, 185.7, 160.2, 146.3, 144.3, 138.5, 134.5, 132.5, 130.9, 128.4, 123.1, 119.7, 92.3; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₈ClNO₃⁻: 411.9243, found: 411.9231.



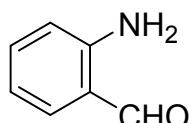
4g

N-(4-bromo-2-formylphenyl)-2-oxo-2-phenylacetamide (**4g**): Yellow solid; 106.2 mg (yield 64%); mp 120-122 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.38 (s, 1H), 9.94 (s, 1H), 8.78 (d, *J* = 9.0 Hz, 1H), 8.38 (d, *J* = 6.6 Hz, 2H), 7.87 (s, 1H), 7.77 (d, *J* = 7.2 Hz, 1H), 7.66 (t, *J* = 6.6 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 2H); ¹³C NMR (150 MHz, CDCl₃) δ 193.7, 186.3, 160.5, 138.6, 138.3, 138.1, 134.6, 132.9, 131.3, 128.6, 124.2, 121.8, 116.6; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₉BrNO₃⁻: 329.9771, found: 329.9773.



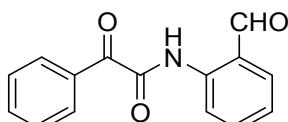
4h

N-(5-bromo-2-formylphenyl)-2-oxo-2-phenylacetamide (**4h**): Yellow solid; 135.1 mg (yield 59%); mp 142-144 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.10 (s, 1H), 9.92 (s, 1H), 8.83 (s, 1H), 8.44 (s, 1H), 8.21 (d, *J*=7.8 Hz, 2H), 7.73 (t, *J*=7.2 Hz, 1H), 7.57 (t, *J*=7.8 Hz, 2H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 194.3, 185.8, 160.2, 145.9, 138.2, 136.8, 134.5, 132.5, 130.9, 128.5, 123.5, 123.2, 95.6; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₅H₈BrINO₃⁻: 455.8738, found: 455.8721.



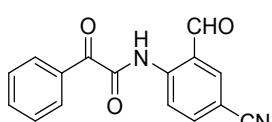
5

2-aminobenzaldehyde (**5**): Yellow solid; 30.3 mg (yield 25%); ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.81 (s, 1H), 7.51 (d, *J*=7.6 Hz, 1H), 7.29 (t, *J*=7.6 Hz, 1H), 7.16 (s, 2H), 6.77 (d, *J*=8.4 Hz, 1H), 6.62 (t, *J*=7.6 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 194.1, 150.8, 135.7, 135.1, 117.8, 115.9, 115.0; The ¹H NMR and ¹³C NMR spectra data are consistent with the reported literature.



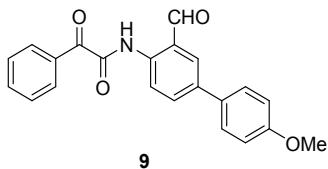
6

N-(2-formylphenyl)-2-oxo-2-phenylacetamide (**6**): Yellow solid; 121.4 mg (yield 80%); mp 110-112 °C; ¹H NMR (600 MHz, CDCl₃) δ 12.45 (s, 1H), 9.98 (s, 1H), 8.82 (d, *J*=8.4 Hz, 1H), 8.37 (d, *J*=7.8 Hz, 2H), 7.74 (d, *J*=7.2 Hz, 1H), 7.71-7.60 (m, 2H), 7.50 (t, *J*=7.8 Hz, 2H), 7.40-7.27 (m, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 194.9, 186.5, 160.4, 138.9, 136.1, 135.8, 134.4, 132.9, 131.1, 128.4, 124.1, 122.7, 119.9; The ¹H NMR and ¹³C NMR spectra data are consistent with the reported literature.

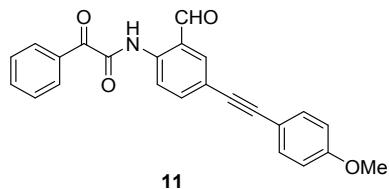


7

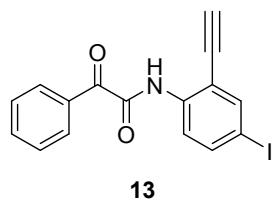
N-(4-cyano-2-formylphenyl)-2-oxo-2-phenylacetamide (**7**): Purple solid; 33.4 mg (yield 60%); mp 110-112 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.38 (s, 1H), 10.04 (s, 1H), 8.68 (d, *J* = 8.4 Hz, 1H), 8.53 (s, 1H), 8.30-8.14 (m, 3H), 7.75 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 7.2 Hz, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 193.4, 185.1, 159.8, 140.7, 138.7, 137.7, 133.6, 131.7, 129.9, 127.6, 122.6, 119.5, 116.8, 105.6; HRMS (ESI): m/z [M-H]⁻ calcd for C₁₆H₉N₂O₃⁻: 277.0619, found: 277.0609.



N-(3-formyl-4'-methoxy-[1,1'-biphenyl]-4-yl)-2-oxo-2-phenylacetamide (**9**): Yellow solid; 66.1 mg (yield 92%); mp 110-112 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.17 (s, 1H), 10.11 (s, 1H), 8.59 (d, *J* = 8.8 Hz, 1H), 8.29-8.20 (m, 3H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.77-7.67 (m, 3H), 7.59 (t, *J* = 7.6 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 2H), 3.80 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 196.0, 187.0, 160.6, 159.3, 136.7, 135.9, 134.5, 133.0, 132.9, 132.8, 130.9, 130.3, 128.6, 127.6, 124.1, 120.6, 114.5, 55.2; HRMS (ESI): m/z [M-H]⁻ calcd for C₂₂H₁₆NO₄⁻: 358.1085, found: 358.1095.



N-(2-formyl-4-((4-methoxyphenyl)ethynyl)phenyl)-2-oxo-2-phenylacetamide (**11**): Yellow solid; 64.3 mg (yield 84%); mp 110-112 °C; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.23 (s, 1H), 10.03 (s, 1H), 8.58 (d, *J* = 8.4 Hz, 1H), 8.22 (d, *J* = 7.2 Hz, 2H), 8.15 (s, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.74 (t, *J* = 7.2 Hz, 1H), 7.58 (t, *J* = 7.2 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 6.98 (d, *J* = 7.8 Hz, 2H), 3.79 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 194.4, 185.6, 159.6, 158.7, 137.3, 136.79, 136.75, 133.5, 132.0, 131.8, 129.9, 127.6, 122.7, 119.3, 117.7, 113.5, 112.8, 89.5, 85.5, 54.3; HRMS (ESI): m/z [M-H]⁻ calcd for C₂₄H₁₆NO₄⁻: 382.1085, found: 382.1090.



N-(2-ethynyl-4-iodophenyl)-2-oxo-2-phenylacetamide (**13**): Yellow solid; 108.7 mg (yield 58%); mp 200-202 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.31 (s, 1H), 9.93 (s, 1H), 8.18 (s, 1H), 8.01 (d,

J = 7.6 Hz, 1H), 7.87 (d, *J* = 6.8 Hz, 1H), 7.65 (s, 2H), 7.55 (d, *J* = 6.0 Hz, 1H), 7.50 (d, *J* = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 192.7, 150.9, 143.4, 140.8, 138.0, 132.5, 130.9, 129.1, 126.5, 124.0, 118.9, 88.9, 85.9, 83.4; HRMS (ESI): m/z [M+H]⁺ calcd for C₁₆H₁₁INO₂⁺ 375.9829, found 375.9825.

8. Copies of ^1H NMR and ^{13}C NMR spectra

