Supporting Information

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

Shi-Yi Zhuang, Yong-Xing Tang, Xiang-Long Chen, Yan-Dong Wu, and An-Xin Wu*

Key Laboratory of Pesticide & Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, P. R. China
E-mail: chwuax@mail.ccnu.edu.cn

Contents

1. General..........................................................................................................................S2
2. Experimental procedures..............................................................................................S3-S5
3. Optimization of the standard conditions.................................................................S6
4. Substrate scope of aliphatic ketones..........................................................................S7
5. GC-MS of the intermediates.......................................................................................S8-S9
6. The crystallographic data...........................................................................................S10-S11
7. Spectroscopic data.......................................................................................................S12-S24
8. Copies of ¹H NMR and ¹³C NMR spectra.................................................................S25-S61
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). $^1$H 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). $^{13}$C NMR spectra was recorded in CDCl$_3$ or DMSO-$d_6$ on 150 MHz NMR spectrometers and resonances ($\delta$) 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 (1.51 g, 10 mmol) and SnCl$_2$ dihydrate (6.75 g, 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 Na$_2$S$_2$O$_3$ solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na$_2$SO$_4$ 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), CuBr$_2$ (6.6 mg, 0.03 mmol) and PPh$_3$ (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

![Reaction](image)

<table>
<thead>
<tr>
<th>entry</th>
<th>acid (equiv.)</th>
<th>I₂ (equiv.)</th>
<th>solvent</th>
<th>temp (°C)</th>
<th>yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>DMF</td>
<td>140</td>
<td>ND</td>
</tr>
<tr>
<td>2</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>Toluene</td>
<td>110</td>
<td>ND</td>
</tr>
<tr>
<td>3</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>CH₃CN</td>
<td>80</td>
<td>ND</td>
</tr>
<tr>
<td>4</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>THF</td>
<td>60</td>
<td>ND</td>
</tr>
<tr>
<td>5</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>CH₂Cl₂</td>
<td>rt</td>
<td>ND</td>
</tr>
<tr>
<td>6</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>DCE</td>
<td>80</td>
<td>ND</td>
</tr>
<tr>
<td>7</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>1,4-Dioxane</td>
<td>100</td>
<td>ND</td>
</tr>
<tr>
<td>8</td>
<td>TfOH (0.5)</td>
<td>1.6</td>
<td>EtOH</td>
<td>80</td>
<td>ND</td>
</tr>
</tbody>
</table>
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
I$_2$, TIOH, DMSO, 140 °C

2a \rightarrow \begin{align*} \text{not detected} & \quad \text{2aa} \\ \text{without additional HI} & \quad \text{not detected} \end{align*}

with 1.6 equiv. HI

5% 25%

5 detected by GC-MS

90% recover

62% recover

5a detected by GC-MS

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

5.2 GS-MS of the intermediate 5a
NH$_2$CHO + I$_2$, TfOH, DMSO, 140 °C → NH$_2$CHO

detected by GC-MS

Figure S2. GS-MS of the intermediate 5a
6. The crystallographic data

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 Å

Cell:  
\[ a = 7.7449(12) \]  
\[ b = 16.862(3) \]  
\[ c = 21.490(3) \]  
\[ \alpha = 90^\circ \]  
\[ \beta = 90^\circ \]  
\[ \gamma = 90^\circ \]

Temperature:  
273 K

Volume  
Calculated: 2806.5(8) Å³  
Reported: 2806.3(7) Å³

Space group  
P b c a

Hall group  
-P 2ac 2ab

Moity 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

N-(2-formyl-4-iodophenyl)-2-oxo-2-phenylacetamide (3a): Yellow solid; 147.8 mg (yield 78%); mp 139-141 °C; \(^1\)H NMR (600 MHz, DMSO-\(d_6\)) \(\delta\) 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); \(^1^3\)C NMR (150 MHz, DMSO-\(d_6\)) \(\delta\) 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 C\(_{15}\)H\(_9\)INO\(_3\): 377.9633, found: 377.9635.

N-(2-formyl-4-iodophenyl)-2-oxo-2-(p-tolyl)acetamide (3b): Yellow solid; 147.4 mg (yield 75%); mp 137-139 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 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); \(^1^3\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 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 C\(_{16}\)H\(_{11}\)INO\(_3\): 391.9789, found: 391.9792.

N-(2-formyl-4-iodophenyl)-2-oxo-2-(m-tolyl)acetamide (3c): Yellow solid; 137.6 mg (yield 70%); mp 88-89 °C; \(^1\)H NMR (600 MHz, DMSO-\(d_6\)) \(\delta\) 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); \(^1^3\)C NMR (150 MHz, DMSO-\(d_6\)) \(\delta\) 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 C\(_{16}\)H\(_{11}\)INO\(_3\): 391.9789, found: 391.9788.
N-(2-formyl-4-iodophenyl)-2-oxo-2-(o-tolyl)acetamide (3d): Yellow solid; 143.4 mg (yield 73%); mp 133-135 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 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); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 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 C\(_{16}\)H\(_{11}\)INO\(_3\): 391.9789, found: 391.9792.

2-(2,6-dimethylphenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3e): Yellow solid; 132.3 mg (yield 65%); mp 184-186 °C; \(^1\)H NMR (400 MHz, DMSO-d\(_6\)) \(\delta\) 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); \(^{13}\)C NMR (100 MHz, DMSO-d\(_6\)) \(\delta\) 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 C\(_{17}\)H\(_{13}\)INO\(_3\): 405.9946, found: 405.9937.

N-(2-formyl-4-iodophenyl)-2-(4-methoxyphenyl)-2-oxoacetamide (3f): Yellow solid; 143.2 mg (yield 70%); mp 184-186 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 12.40 (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); \(^{13}\)C NMR (100 MHz, DMSO-d\(_6\)) \(\delta\) 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 C\(_{16}\)H\(_{11}\)INO\(_4\): 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; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 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); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 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, 116.4, 86.5, 55.4.; HRMS (ESI): m/z [M-H]$^-$ calcd for C$_{16}$H$_{11}$INO$_4$: 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; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 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); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 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 C$_{16}$H$_{11}$INO$_4$: 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; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 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); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 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 C$_{17}$H$_{13}$INO$_4$: 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; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 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); \(^1^3\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 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 C\(_{16}\)H\(_9\)INO\(_5\): 421.9531, found: 421.9533.

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; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 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); \(^1^3\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 194.6, 184.3, 161.1, 153.0, 147.8, 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 C\(_{17}\)H\(_{11}\)INO\(_5\): 435.9687, found: 435.9687.

N-(2-formyl-4-iodophenyl)-2-(4-(methylthio)phenyl)-2-oxoacetamide (3l): Yellow solid; 131.8 mg (yield 62%); mp 181-183 °C; \(^1\)H NMR (600 MHz, DMSO-\(d_6\)) \(\delta\) 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); \(^1^3\)C NMR (150 MHz, DMSO-\(d_6\)) \(\delta\) 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 C\(_{16}\)H\(_{11}\)INO\(_3\)S: 423.9510, found: 423.9506.
methyl 4-(2-((2-formyl-4-iodophenyl)amino)-2-oxoacetyl)benzoate (3m): Yellow solid; 142.0 mg (yield 65%); mp 207-209 °C; $^1$H NMR (600 MHz, DMSO-$d_6$) $\delta$ 12.15 (s, 1H), 9.99 (s, 1H), 8.37-8.27 (m, 4H), 8.14-8.07 (m, 3H), 3.91 (s, 3H); $^{13}$C NMR (150 MHz, DMSO-$d_6$) $\delta$ 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 C$_{17}$H$_{11}$INO$_5$: 435.9687, found: 435.9690.

N-(2-formyl-4-iodophenyl)-2-(3-nitrophenyl)-2-oxoacetamide (4n): Yellow solid; 127.2 mg (yield 60%); mp 205-207 °C; $^1$H NMR (600 MHz, DMSO-$d_6$) $\delta$ 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); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 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 C$_{15}$H$_8$IN$_2$O$_5$: 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; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 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); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$194.9, 185.8, 159.8, 144.8, 143.8, 143.5, 137.8, 137.0, 131.7, 126.9, 125.4, 122.1, 88.3, 43.1; HRMS (ESI): m/z [M-H] calcd for C$_{16}$H$_{11}$INO$_5$S: 455.9408, found: 455.9411.

2-(2-fluorophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3p): Yellow solid; 139.0 mg
(yield 70%); mp 98-100 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 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); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 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; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -109.45; HRMS (ESI): m/z [M-H]\(^-\) calcd for C\(_{15}\)H\(_9\)FINO\(_3\): 395.9538, found: 395.9541.

![3q](image)

2-(3-chlorophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3q): Yellow solid; 144.9 mg (yield 70%); mp 165-167 °C; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 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); \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 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 C\(_{15}\)H\(_8\)ClINO\(_3\): 411.9243, found: 411.9237.

![3r](image)

2-(2-chlorophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3r): Yellow solid; 157.3 mg (yield 76%); mp 155-157 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 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); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 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 C\(_{15}\)H\(_8\)ClINO\(_3\): 411.9243, found: 411.9245.

![3s](image)

2-(2-bromophenyl)-N-(2-formyl-4-iodophenyl)-2-oxoacetamide (3s): Yellow solid; 164.9 mg (yield 72%); mp 155-157 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 12.35 (s, 1H), 9.92 (s, 1H), 8.56 (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); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 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; \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -109.45; HRMS (ESI): m/z [M-H]\(^-\) calcd for C\(_{15}\)H\(_9\)BrINO\(_3\): 411.9243, found: 411.9245.
= 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\(_3\)) \( \delta \) 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 C\(_{15}\)H\(_8\)BrINO\(_3\): 455.8738, found: 455.8741.

![3t](image)

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; \(^1\)H NMR (600 MHz, DMSO-\(d_6\)) \( \delta \) 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 (150 MHz, DMSO-\(d_6\)) \( \delta \) 194.7, 185.9, 160.7, 145.7, 143.8, 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 C\(_{21}\)H\(_{13}\)INO\(_3\): 453.9946, found: 453.9944.

![3u](image)

N-(2-formyl-4-iodophenyl)-2-(naphthalen-1-yl)-2-oxoacetamide (3u): Yellow solid; 139.4 mg (yield 65%); mp 148-150 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \( \delta \) 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\(_3\)) \( \delta \) 193.7, 188.7, 160.8, 144.3, 138.7, 134.9, 133.8, 133.2, 131.2, 129.0, 128.6, 126.6, 126.7, 125.4, 125.3, 124.4, 124.1, 121.9, 86.6; HRMS (ESI): m/z [M-H]\(^{-}\) calcd for C\(_{19}\)H\(_{11}\)INO\(_3\): 427.9789, found: 427.9792.

![3v](image)

N-(2-formyl-4-iodophenyl)-2-(naphthalen-2-yl)-2-oxoacetamide (3v): Yellow solid; 141.6 mg (yield 66%); mp 148-150 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \( \delta \) 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\(_3\)) \( \delta \) 193.6, 185.6, 160.5,
N-(2-formyl-4-iodophenyl)-2-oxo-2-(thiophen-2-yl)acetamide (3w): Yellow solid; 115.5 mg (yield 60%); mp 149-151 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 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); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 193.6, 193.3, 177.1, 159.7, 144.2, 139.0, 138.3, 138.2, 136.0, 128.3, 125.9, 124.5, 121.9, 86.8. HRMS (ESI): m/z [M-H]\(^-\) calcd for C\(_{19}\)H\(_{11}\)INO\(_3\): 427.9789, found: 427.9792.

\[
\begin{array}{c}
\text{N-(2-formyl-4-iodophenyl)-2-oxo-2-(thiophen-2-yl)acetamide (3w) & }  \\
\end{array}
\]

N-(2-formyl-4-iodophenyl)-2-oxo-2-(thiophen-3-yl)acetamide (3x): Yellow solid; 121.3 mg (yield 63%); mp 139-141 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 12.49 (s, 1H), 9.93 (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); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 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 C\(_{13}\)H\(_{7}\)INO\(_3\): 383.9197, found: 383.9199.

\[
\begin{array}{c}
\text{N-(2-formyl-4-iodophenyl)-2-oxo-2-(thiophen-3-yl)acetamide (3x) & }  \\
\end{array}
\]

N-(2-formyl-4-methoxyphenyl)-2-oxo-2-phenylacetamide (4a): Yellow solid; 82.1 mg (yield 58%); mp 110-112 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 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); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 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 C\(_{16}\)H\(_{12}\)NO\(_4\): 282.0772, found: 282.0773.

\[
\begin{array}{c}
\text{N-(2-formyl-4-methoxyphenyl)-2-oxo-2-phenylacetamide (4a) & }  \\
\end{array}
\]
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 C$_{16}$H$_{10}$NO$_5$: 296.0564, found: 296.0566.

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, CDCl$_3$) 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 C$_{15}$H$_{9}$FNO$_3$: 270.0572, found: 270.0573.

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 C$_{15}$H$_{8}$FNO$_3$: 395.9538, found: 395.9530.
N-(4-chloro-2-formylphenyl)-2-oxo-2-phenylacetamide (4e): Yellow solid; 97.9 mg (yield 68%); mp 138-140 °C; $^1$H 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); $^{13}$C NMR (150 MHz, DMSO-$d_6$) δ 194.3, 186.7, 160.8, 137.0, 135.1, 134.6, 134.1, 132.8, 130.9, 128.6, 128.5, 125.3, 122.3; HRMS (ESI): m/z [M-H]· calcd for C$_{15}$H$_9$ClNO$_3$: 286.0276, found: 286.0278.

N-(5-chloro-2-formylphenyl)-2-oxo-2-phenylacetamide (4f): Yellow solid; 132.5 mg (yield 64%); mp 148-150 °C; $^1$H 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); $^{13}$C 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 C$_{15}$H$_8$ClINO$_3$: 411.9243, found: 411.9231.

N-(4-bromo-2-formylphenyl)-2-oxo-2-phenylacetamide (4g): Yellow solid; 106.2 mg (yield 64%); mp 120-122 °C; $^1$H NMR (600 MHz, CDCl$_3$) 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); $^{13}$C NMR (150 MHz, CDCl$_3$) 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 C$_{15}$H$_9$BrNO$_3$: 329.9771, found: 329.9773.
N-(5-bromo-2-formylphenyl)-2-oxo-2-phenylacetamide (4h): Yellow solid; 135.1 mg (yield 59%); mp 142-144 °C; $^1$H NMR (600 MHz, DMSO-$d_6$) $\delta$ 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); $^{13}$C NMR (150 MHz, DMSO-$d_6$) $\delta$ 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 C$_{15}$H$_8$BrINO$_3$: 455.8738, found: 455.8721.

2-aminobenzaldehyde (5): Yellow solid; 30.3 mg (yield 25%); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 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); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 194.1, 150.8, 135.7, 135.1, 117.8, 115.9, 115.0; The $^1$H NMR and $^{13}$C NMR spectra data are consistent with the reported literature.

N-(2-formylphenyl)-2-oxo-2-phenylacetamide (6): Yellow solid; 121.4 mg (yield 80%); mp 110-112 °C; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 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); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 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 $^1$H NMR and $^{13}$C NMR spectra data are consistent with the reported literature.
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-d₆) 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-d₆) 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]⁻ calc'd for C₁₆H₉N₂O₃: 277.0619, found: 277.0609.

O
H
N
O
CHO

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-d₆) 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-d₆) 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]⁻ calc'd for C₂₂H₁₆NO₄: 358.1085, found: 358.1095.

O
H
N
O
CHO

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-d₆) 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-d₆) 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]⁻ calc'd for C₂₄H₁₆NO₄: 382.1085, found: 382.1090.

O
H
N
O

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-d₆) 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-$d_6$) $\delta$ 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 C$_{16}$H$_{11}$NO$_2$ $^+$ 375.9829, found 375.9825.
8. Copies of $^1$H NMR and $^{13}$C NMR spectra
3w
600 MHz
CDCl₃

3w
150 MHz
CDCl₃
13
400 MHz
DMSO-d6

13
100 MHz
DMSO-d6