Supporting Information

Synthesis of Carbonylated Heteroaromatic Compounds via Visible-Light-Driven Intramolecular Decarboxylative Cyclization of \( o \)-Alkynylated Carboxylic Acids

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1. General Information

All starting materials were purchased from commercial sources without further purification. Glassware was dried in oven and cooled before use. All reactions were monitored by TLC and visualized by UV lamp (254nm). The solvents were distilled from the appropriate drying reagents. Yields generally referred to chromatographically isolated yields, unless otherwise noted.

$^1$H NMR (400 MHz) and $^{13}$C NMR (100 MHz) spectra were obtained on Bruker AV-400 instrument in CDCl$_3$ or DMSO-d$_6$. For $^1$H NMR (400MHz), CDCl$_3$ ($\delta = 7.26$ ppm) and DMSO-d$_6$ ($\delta = 2.5$ ppm) served as internal standard and data are reported as follows: chemical shift (in ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad singlet), coupling constant (in Hz), and integration. HRMS (ESI) spectra were recorded on a Bruker Esquire LC mass spectrometer using electrospray ionization. GC-MS analysis was performed on a 7890A-5975C/Agilent. Flash column chromatography was performed using 200-300 mesh silica gel.

2. Typical Procedures for Preparation of Substrates

2.1 Synthesis of o-alkynylated $\alpha$-amino acids 1

For substrates 1a-1c, 1e-1r were synthesized according to the procedure described in the literature. $^1$Synthesis of 1a is representative.

**Synthesis of S1**

To a stirred solution of 2-iodoaniline (1.0 equiv), Pd(PPh$_3$)$_2$Cl$_2$ (0.02 equiv), and Cul (0.04 equiv) at room temperature in degassed Et$_3$N (0.2 M) was added dropwise phenylacetylene (1.5 equiv). The mixture was heated to 50 °C for 5-8 h. When the consumption of the corresponding 2-iodoaniline completed (monitored by TLC), the reaction was cooled to room temperature. Subsequent filtration through a pad of celite rinsing with ethyl acetate, followed by purification of the remaining crude material via flash chromatography afforded the compound S1.

**Synthesis of S2**

S2
Compound S1 (1.0 equiv) was dissolved in THF (0.2 M) under N₂ atmosphere and cooled to –78 °C. nBuLi solution (2.5 M in hexanes, 1.1 equiv) was added dropwise and the mixture was stirred for 1 h. CH₃I (1.5 equiv) was added and the mixture was stirred at room temperature for 2 h. Upon reaction completion, the reaction was carefully quenched by dropwise addition of NH₄Cl aq. solution at 0 °C. Then the solution was added to saturated NaHCO₃ aq. solution and extracted with EtOAc (3 x). The combined organic phases were dried with MgSO₄, filtered, and concentrated in vacuo. The crude material was purified by flash chromatography using the indicated eluent.

**Synthesis of S3**

To a solution of S2 (1.0 equiv) in ethanol (0.5 M) was added Na₂CO₃ (1.5 equiv) and ethyl bromoacetate (1.5 equiv). Then the suspension was stirred at reflux for 14 h. Upon reaction completion, the reaction was quenched with water and extracted with ethyl acetate (4 x). The combined organics were washed with water (1 x), dried over Na₂SO₄, filtered and concentrated under vacuum. The crude material was purified by flash chromatography using the indicated eluent.

**Synthesis of 1a**

To a solution of S3 (1.0 equiv) in EtOH (0.2 M) at 0°C was slowly added a 15% m/v NaOH aq. solution (5.0 equiv). The reaction mixture was allowed to room temperature overnight with stirring. Ethanol and water was removed in vacuo and the residue redissolved in the minimal amount of H₂O possible. Concentrated hydrochloric acid was added dropwise until the pH was acidic. The solid formed was filtered off and washed with cold H₂O (5 mL). The solid was dried under high vacuum and pure 1a was obtained.

2.2 Synthesis of N-acetyl-N-(2-(phenylethynyl)phenyl)glycine 1d
Step 1: To a stirred solution of 2-iodoaniline (1.0 equiv), Pd(PPh₃)₂Cl₂ (0.02 equiv), and CuI (0.04 equiv) at room temperature in degassed Et₃N (0.2 M) was added dropwise phenylacetylene (1.5 equiv). The mixture was heated to 50 °C for 5-8 h. When the consumption of the corresponding 2-iodoaniline completed (monitored by TLC), the reaction was cooled to room temperature. Subsequent filtration through a pad of celite rinsing with ethyl acetate, followed by purification of the remaining crude material via flash chromatography afforded the compound S1.

Step 2: To a solution of S1 (1.0 equiv) in ethanol (0.5 M) was added Na₂CO₃ (1.5 equiv) and ethyl bromoacetate (1.5 equiv). Then the suspension was stirred at reflux for 14 h. The reaction was quenched with water and extracted with ethyl acetate (4 x). The combined organics were washed with water (1 x), dried over Na₂SO₄, filtered and concentrated under vacuum. The crude material was purified by flash chromatography using the indicated eluent.

Step 3: To a solution of S4 (1.0 equiv) in anhydrous dichloromethane, were added Na₂CO₃ (1.5 equiv.) and acetyl chloride (2.5 equiv.). The mixture was refluxed overnight and cooled to rt. The reaction was quenched with water and extracted with dichloromethane (3 x). The organic phase was dried over Na₂SO₄, filtered and the solvent was removed in vacuo. The crude material was purified by flash chromatography using the indicated eluent.

Step 4: To a solution of S5 (1.0 equiv) in EtOH (0.2 M) at 0°C was slowly added a 15% m/v NaOH (aq) solution (5.0 equiv). The reaction mixture was allowed to room temperature overnight with stirring. Ethanol and water was removed in vacuo and the residue redissolved in the minimal amount of H₂O possible. Concentrated hydrochloric acid was added dropwise until the pH was acidic. The solid formed was filtered off and washed with cold H₂O (5 mL). The solid was dried under high vacuum and pure 1d was obtained.

2.3 Synthesis of o-alkynylated phenoxyacetic acids 3

Phenoxyacetic acids 3a-3q were synthesized according to the procedure described in the literature.² Synthesis of 3b is representative.

Synthesis of S6

To a 0.3 M solution of 2-iodophenol (1.0 equiv.) in N, N-dimethylformamide (DMF), was added K₂CO₃ (1.5 equiv.), and the resulting solution was stirred at ambient temperature for 30 min. Then was added ethyl a-bromophenylacetate (1.5 equiv) in one portion and the resulting solution was stirred at ambient temperature for 18 h. The solvent was added H₂O (a volume equivalent to the amount of DMF initially used), and dichloromethane was used to extract (3 x half the volume of DMF initially used). The combined organics were washed with brine (1 x), dried over Na₂SO₄, filtered and concentrated in vacuo. The crude material was purified by flash chromatography using the indicated eluent.
**Synthesis of S7**

\[
\begin{array}{c}
\text{S6} \\
\text{COOEt} \\
\text{Ph} \\
\text{O} \\
\text{S7} \\
\text{COOEt} \\
\text{Ph} \\
\end{array}
\begin{array}{c}
\text{PdCl}_2(PPh_3)_2 \quad (2.0 \text{ mol\%}) \\
\text{CuI} \quad (4.0 \text{ mol\%}), \text{Et}_3N
\end{array}
\]

To solution of S6 (1.0 equiv), PdCl\(_2\)(PPh\(_3\))\(_2\) (0.02 equiv) and CuI (0.04 equiv) in anhydrous NEt\(_3\) (0.2 M) was added phenylacetylene (1.5 equiv) under N\(_2\). The reaction mixture was stirred at ambient temperature. Upon full consumption of the starting material, the reaction mixture was filtered through a pad of celite, eluting with EtOAc (3 x). The combined organics were concentrated in vacuo. The resulting crude mixture was purified by flash chromatography using the indicated eluent.

**Synthesis of 3b**

\[
\begin{array}{c}
\text{S7} \\
\text{COOEt} \\
\text{Ph} \\
\text{O} \\
\text{S8} \\
\text{COOEt} \\
\text{Ph} \\
\end{array}
\begin{array}{c}
\text{NaOH} \\
\text{MeOH}
\end{array}
\]

The corresponding ester S7 (1.0 equiv) was dissolved in methanol. To the resulting 0.5 M solution was added a 15% m/v NaOH\(_{\text{aq}}\) solution (5.0 equiv). The reaction mixture was stirred at ambient temperature for 18 h. The solvent was removed in vacuo and the residue redissolved in the minimal amount of H\(_2\)O possible. Concentrated hydrochloric acid was added drop wise until the pH was acidic. The white solid formed was filtered off and washed with cold H\(_2\)O (5 mL). The solid was dried under high vacuum and pure o-alkynylated phenoxyacetic acids 3b was obtained.

**2.4 Synthesis of o-alkynylated carboxylic acids 5**

\[
\begin{array}{c}
\text{Br} \\
\text{R}_2 \quad \text{COOEt} \\
\text{NaOH, EtOH}
\end{array}\quad \begin{array}{c}
\text{Br} \\
\text{R}_2 \quad \text{COOEt} \\
\text{NaOH, EtOH}
\end{array}\quad \begin{array}{c}
\text{Br} \\
\text{R}_3 \\
\text{S9}
\end{array}
\]

Carboxylic acids 5a-5h were synthesized according to the procedure described in the literature. Synthesis of 5a is representative.

**Synthesis of S8**

\[
\begin{array}{c}
\text{Br} \\
\text{S8} \\
\text{Br} \\
\text{NaOH (1.5 eq.), EtOH}
\end{array}
\begin{array}{c}
\text{Ph} \quad \text{COOEt} \\
\text{NaOH (1.5 eq.), EtOH}
\end{array}
\]

To a 0.2 M solution of 2-bromothiophenol (1.0 equiv) in EtOH was added NaOH (1.5 equiv), and the resulting solution was stirred at ambient temperature for 30 min. Then was added ethyl o-bromophenylacetate (2.0 equiv) in one portion and the resulting solution was stirred overnight at room temperature. The reaction mixture was filtered through a pad of celite, eluting with EtOAc (3 x). The combined organics were concentrated in vacuo. The resulting crude mixture was
purified by flash chromatography using the indicated eluent.

**Synthesis of S9**

![Chemical structure of S9]

To a solution of S8 (1.0 equiv.), 1,4-diazabicyclo[2.2.2]octane (2.0 equiv), [Pd(allyl)Cl]_2 (0.025 equiv), t-Bu_3P (0.1 equiv) in acetonitrile (1.0 M) was added phenylacetylene (1.6 equiv) under N_2 atmosphere. The reaction mixture was stirred overnight at room temperature. The mixture was diluted with ether, filtered celite pad and concentrated *in vacuo*. The resulting crude mixture was purified by flash chromatography using the indicated eluent.

**Synthesis of 5a**

![Chemical structure of 5a]

The corresponding ester S9 (1.0 equiv.) was dissolved in methanol. To the resulting 0.5 M solution was added a 15% m/v NaOH (aq) solution (5.0 equiv). The reaction mixture was stirred overnight at ambient temperature. The solvent was removed *in vacuo* and the residue redissolved in the minimal amount of H_2O possible. Concentrated hydrochloric acid was added dropwise until the pH was acidic. The pale yellow solid formed was filtered off and washed with cold H_2O (5 mL). The solid was dried under high vacuum and pure o-alkynylated carboxylic acids 5a was obtained.

**Reference:**


### 3. Study of Mechanism

#### 3.1 Controlled Experiment

**Typical procedure for ^18_O_2 labeling experiments:** We have performed an ^18_O_2 labeling experiment under the standard conditions (^18_O_2 gas instead of ^16_O_2 air, was filled in the reaction system). Isolated yield: 54%. From GC-MS (EI) the final product which was determined to contain an ^18_O was obtained exclusively, indicating that the oxygen atom of the ketonic carbonyl group in the 3-Acylindoles products is originated from O_2 in the air.
Typical procedure for H_{18}O labeling experiment: To a 10 mL bottom flask were added compound 1a (0.1 mmol), Ir[dF(CF_{3}ppy)]_{2}(dtbbpy)PF_{6} (5 mol %, 5.6 mg), Cs_{2}CO_{3} (1.5 equiv, 48.8 mg), anhydrous DMF (4 mL). Then, H_{2}^{18}O (5.0 equiv) was added into the system. The reaction mixture was stirred under air atmosphere with the irradiation of blue LEDs (30 W) at room temperature for about 24 h. After the reaction was completed, as monitored by TLC, water
(12 mL) was added, the resulting mixture was extracted with ethyl acetate (15 mL × 5). The organic layer was combined, dried over Na₂SO₄. The filtrate was concentrated for purification by chromatography on silica gel with petroleum ether/EtOAc to afford the desired products 2a. Isolated yield: 52%.

The GC-MS spectra
3.2 Cyclic voltammogram experiment

Cyclic voltammogram 1a, 3b and 5a in 0.1 M TBAP/MeCN at a Pt working electrode with a Pt counter electrode and saturated calomel electrode (SCE). Potential sweep rate was 100 mV/s.

4. General Procedures for Decarboxylative Cyclization

![Diagram of general procedures for decarboxylative cyclization]
**General procedure 1:** To a 10 mL bottom flask were added substrate 1 (0.1 mmol), Cr₂CO₃ (0.15 mmol), DMF (anhydrous, 4.0 mL), [Ir{dF(CF₃ppy)}₂(dtbppy)]PF₆ (0.005 mmol). The reaction mixture was stirred under air atmosphere with the irradiation of blue LEDs (30 W) at room temperature. After the reaction was completed, as monitored by TLC, water (12 mL) was added, the resulting mixture was extracted with ethyl acetate (15 mL × 5). The organic layer was combined and dried over Na₂SO₄. Then the filtrate was concentrated for purification by chromatography on silica gel with petroleum ether/EtOAc to afford the desired products 2.

![Reaction Scheme 1](image1)

**General procedure 2:** To a 10 mL bottom flask were added substrate 3 (0.1 mmol), Cr₂CO₃ (0.15 mmol), DMSO (anhydrous, 4.0 mL), [Ir{dF(CF₃ppy)}₂(dtbppy)]PF₆ (0.005 mmol). The mixture was carried out under air atmosphere with blue LEDs (30 W) irradiation at room temperature. After the substrate was consumed (monitored by TLC), the reaction mixture was quenched with water (12 mL) and extracted with EtOAc (15 mL × 5). The organic layer was combined and dried over Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by silica gel flash column chromatography (petroleum ether/EtOAc) to afford the desired product 4.

![Reaction Scheme 2](image2)

**General procedure 3:** To a 10 mL bottom flask were added substrate 5 (0.1 mmol), Cr₂CO₃ (0.15 mmol), DMSO (anhydrous, 4.0 mL), [Ir{dF(CF₃ppy)}₂(dtbppy)]PF₆ (0.005 mmol). The mixture was carried out under air atmosphere with blue LEDs (30 W) irradiation at room temperature. After the substrate was consumed (monitored by TLC), the reaction mixture was quenched with water (12 mL) and extracted with EtOAc (15 mL × 5). The organic layer was combined, dried (Na₂SO₄), filtered, and concentrated in vacuo. The residue was purified by silica gel flash column chromatography (petroleum ether/EtOAc) to afford the desired product 6.

**Gram Scale Reaction**

To a 250 mL flask unsealed under air atmosphere was charged with compound 1j (0.885 g, 3 mmol), Ir[dF(CF₃ppy)]₂(dtbppy)PF₆ (5 mol %, 168 mg), Cs₂CO₃ (1.5 equiv, 1.46 g), anhydrous DMF (120 mL). The reaction mixture was stirred at room temperature with the irradiation of a 30 W blue LEDs for about 36 h. After the reaction was completed, as monitored by TLC, water (150 mL) was added. The resulting mixture was extracted with ethyl acetate (70 mL × 5). The combined organic layers were washed with brine and dried over Na₂SO₄. Then the filtrate was concentrated for purification by chromatography on silica gel with petroleum ether/ethylacetate to afford the desired products 2j (574 mg, 72%).
5. Characterization of new substrates

N-methyl-N-(2-(phenylethynyl)phenyl)glycine (1a)

\[
\text{\includegraphics[width=1cm]{image1.png}}
\]

White solid, mp = 103.7-104.8 °C. \( ^1H \text{NMR} \) (400 MHz, DMSO) \( \delta \) 12.54 (s, 1H), 7.55-7.50 (m, 2H), 7.45-7.39 (m, 4H), 7.28 (t, \( J = 7.8 \) Hz, 1H), 6.97 (d, \( J = 8.3 \) Hz, 1H), 6.87 (t, \( J = 7.4 \) Hz, 1H), 4.27 (s, 2H), 2.98 (s, 3H). \( ^{13}C \text{NMR} \) (100 MHz, DMSO) \( \delta \) 171.9, 152.4, 134.2, 130.9, 129.6, 128.7, 128.4, 122.8, 119.7, 117.5, 111.8, 94.3, 88.9, 55.7, 40.0. \( \text{IR} \) (cm\(^{-1}\)): 754, 948, 1198, 1496, 1592, 1719. \( \text{HRMS} \) m/z (ESI) calcd for C\(_{17}\)H\(_{15}\)NNaO\(_2^+\) [M+Na]\(^+\): 288.1000, found: 288.1003.

N-ethyl-N-(2-(phenylethynyl)phenyl)glycine (1b)

\[
\text{\includegraphics[width=1cm]{image2.png}}
\]

Yellow oil. \( ^1H \text{NMR} \) (600 MHz, CDCl\(_3\)) \( \delta \) 10.89 (s, 1H), 7.62-7.47 (m, 3H), 7.40-7.29 (m, 4H), 7.13 (t, \( J = 7.5 \) Hz, 2H), 3.92 (s, 2H), 3.26 (t, \( J = 6.8 \) Hz, 2H), 1.15 (t, \( J = 7.0 \) Hz, 3H). \( ^{13}C \text{NMR} \) (151 MHz, CDCl\(_3\)) \( \delta \) 172.5, 150.7, 134.0, 131.6, 129.5, 128.7, 128.5, 124.7, 122.7, 122.3, 119.7, 95.2, 86.8, 56.9, 49.5, 12.6. \( \text{IR} \) (cm\(^{-1}\)): 755, 1222, 1386, 1495, 1593, 1722, 2975, 3059. \( \text{HRMS} \) m/z (ESI) calcd for C\(_{18}\)H\(_{17}\)NNaO\(_2^+\) [M+Na]\(^+\): 302.1157, found: 302.1163.

N-methyl-N-(2-(phenylethynyl)phenyl)glycine (1c)

\[
\text{\includegraphics[width=1cm]{image3.png}}
\]

White solid, mp = 124.6-125.3 °C. \( ^1H \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.62-7.57 (m, 2H), 7.45 (d, \( J = 7.5 \) Hz, 1H), 7.41-7.35 (m, 3H), 7.29-7.23 (m, 1H), 6.78 (t, \( J = 7.5 \) Hz, 1H), 6.55 (d, \( J = 8.2 \) Hz, 1H), 4.11 (s, 2H). \( ^{13}C \text{NMR} \) (151 MHz, CDCl\(_3\)) \( \delta \) 176.5, 147.4, 132.2, 131.5, 130.1, 128.5, 128.3, 123.2, 117.8, 109.7, 108.5, 95.7, 85.5, 45.3. \( \text{IR} \) (cm\(^{-1}\)): 744, 895, 1257, 1284, 1435, 1511, 1595, 1726, 2922, 2953, 3393. \( \text{HRMS} \) m/z (ESI) calcd for C\(_{18}\)H\(_{17}\)NNaO\(_2^+\) [M+Na]\(^+\): 274.0844, found: 274.0846.

N-acetyl-N-(2-(phenylethynyl)phenyl)glycine (1d)
N-methyl-N-(2-(phenylethynyl)phenyl)alanine (1e)

White solid, mp = 122.3-123.1 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 10.99 (s, 1H), 7.62-7.55 (m, 3H), 7.41-7.32 (m, 4H), 7.18-7.10 (m, 2H), 4.57 (q, $J = 7.1$ Hz, 1H), 2.81 (s, 3H), 1.43 (d, $J = 7.1$ Hz, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$C 175.1, 152.0, 134.3, 131.6, 129.4, 128.6, 128.5, 122.7, 120.6, 117.8, 95.9, 86.9, 62.4, 35.0, 11.5. IR (cm$^{-1}$): 755, 1097, 1205, 1374, 1445, 1495, 1591, 1710, 2986, 3061. HRMS m/z (ESI) calcd for C$_{18}$H$_{17}$NNaO$_2$ $[M+Na]^{+}$: 302.1157, found: 302.1154.

N-(2-((4-fluorophenyl)ethynyl)phenyl)-N-methylglycine (1f)

White solid, mp = 113.0-113.8 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 9.57 (s, 1H), 7.53-7.47 (m, 3H), 7.06 (d, $J = 8.2$ Hz, 1H), 7.04-6.99 (m, 3H), 4.11 (s, 2H), 2.95 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$C 174.7, 162.6 (d, $J = 249.9$ Hz), 152.2, 134.3, 133.4 (d, $J = 8.1$ Hz), 129.5, 122.5, 119.1 (d, $J = 3.4$ Hz), 118.5, 115.7 (d, $J = 22.0$ Hz), 115.5, 94.5, 87.0, 58.4, 40.9. IR (cm$^{-1}$): 758, 836, 948, 1231, 1356, 1508, 1599, 1724, 2882. HRMS m/z (ESI) calcd for C$_{17}$H$_{15}$FNNaO$_2$ $[M+Na]^{+}$: 306.0906, found: 306.0903.

N-(2-((4-chlorophenyl)ethynyl)phenyl)-N-methylglycine (1g)
White solid, mp = 128.7-129.4 °C. \(^1\)H NMR (400 MHz, DCCl\(_3\)) \(\delta_H\) 10.49 (s, 1H), 7.54 (dd, \(J = 7.7, 1.6\) Hz, 1H), 7.47 (d, \(J = 8.5\) Hz, 2H), 7.36-7.30 (m, 3H), 7.10 (d, \(J = 8.2\) Hz, 1H), 7.05 (td, \(J = 7.5, 1.1\) Hz, 1H), 4.11 (s, 2H), 2.98 (s, 3H).
\(^{13}\)C NMR (151 MHz, DCCl\(_3\)) \(\delta_C\) 174.0, 152.3, 134.5, 134.4, 132.7, 129.7, 128.8, 122.7, 121.5, 118.6, 115.5, 94.5, 88.2, 58.7, 41.0. \(\text{IR (cm}^{-1}\): 760, 830, 949, 1093, 1495, 1592, 1726, 2962. HRMS m/z (ESI) calcd for C\(_{17}\)H\(_{14}\)ClNNaO\(_2\)\(^{+}\) [M+Na]: 322.0611, found: 322.0608.

N-(2-((4-ethylphenyl)ethynyl)phenyl)-N-methylglycine (1h)

Yellow oil. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta_H\) 9.22 (s, 1H), 7.51 (d, \(J = 7.6\) Hz, 1H), 7.44 (d, \(J = 7.4\) Hz, 2H), 7.28 (t, \(J = 7.7\) Hz, 1H), 7.16 (d, \(J = 7.5\) Hz, 2H), 7.07 (d, \(J = 8.2\) Hz, 1H), 7.02 (t, \(J = 7.4\) Hz, 1H), 4.10 (s, 2H), 2.93 (s, 3H), 2.64 (q, \(J = 7.5\) Hz, 2H), 1.22 (t, \(J = 7.6\) Hz, 3H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta_C\) 173.7, 152.1, 145.0, 134.2, 131.5, 129.2, 128.0, 122.8, 120.1, 118.9, 116.4, 96.0, 86.5, 58.7, 41.1, 28.9, 15.4. \(\text{IR (cm}^{-1}\): 750, 833, 948, 1198, 1370, 1487, 1593, 1720, 2931, 2965, 3027. HRMS m/z (ESI) calcd for C\(_{19}\)H\(_{19}\)NNaO\(_2\)\(^{+}\) [M+Na]: 316.1313, found: 316.1311.

N-(2-((4-(tert-butyl)phenyl)ethynyl)phenyl)-N-methylglycine (1i)

Brown oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta_H\) 9.42 (s, 1H), 7.52 (d, \(J = 7.6\) Hz, 1H), 7.47 (d, \(J = 7.5\) Hz, 2H), 7.36 (d, \(J = 7.7\) Hz, 2H), 7.29 (t, \(J = 7.7\) Hz, 1H), 7.08 (d, \(J = 8.2\) Hz, 1H), 7.03 (t, \(J = 7.5\) Hz, 1H), 4.08 (s, 2H), 2.94 (s, 3H), 1.31 (s, 9H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta_C\) 173.3, 152.0, 151.8, 134.3, 131.3, 129.3, 125.5, 123.0, 119.8, 119.0, 116.6, 96.1, 86.4, 58.9, 41.2, 34.8, 31.2. \(\text{IR (cm}^{-1}\): 754, 948, 1199, 1268, 1363, 1487, 1593, 1721, 2962, 3034. HRMS m/z (ESI) calcd for C\(_{21}\)H\(_{23}\)NNaO\(_2\)\(^{+}\) [M+Na]: 344.1626, found: 344.1629.

N-(2-((4-methoxyphenyl)ethynyl)phenyl)-N-methylglycine (1j)
Yellow solid, mp = 103.2-104.4 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 9.53 (s, 1H), 7.54 (dd, $J = 7.6, 1.5$ Hz, 1H), 7.51-7.47 (m, 2H), 7.34-7.29 (m, 1H), 7.10 (d, $J = 8.2$ Hz, 1H), 7.06 (td, $J = 7.5, 1.0$ Hz, 1H), 6.91-6.87 (m, 2H), 4.09 (s, 2H), 3.84 (s, 3H), 2.96 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$C 173.4, 159.8, 152.0, 134.1, 133.0, 129.1, 123.0, 118.8, 116.7, 115.0, 114.1, 95.9, 85.7, 58.9, 55.3, 41.1. IR (cm$^{-1}$): 754, 948, 1029, 1176, 1248, 1487, 1512, 1605, 1721, 2837, 2957. HRMS m/z (ESI) calcd for C$_{18}$H$_{17}$NNaO$_3$ $^{[M+Na]^{+}}$: 318.1106, found: 318.1100.

N-(2-((3-fluorophenyl)ethynyl)phenyl)-N-methylglycine (1k)

Yellow solid, mp = 82.3-87.6 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$H 10.45 (s, 1H), 7.50 (d, $J = 7.5$ Hz, 1H), 7.32-7.24 (m, 3H), 7.20 (d, $J = 9.4$ Hz, 1H), 7.04 (d, $J = 8.3$ Hz, 1H), 7.02-6.97 (m, 2H), 4.14 (s, 2H), 2.97 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$C 175.3, 162.4 ($J = 246.4$ Hz), 152.5, 134.4, 130.0 (d, $J = 8.7$ Hz), 129.8, 127.4 (d, $J = 2.8$ Hz), 124.9 (d, $J = 9.2$ Hz), 122.1, 118.4, 111.7 (d, $J = 22.5$ Hz), 115.7 (d, $J = 21.1$ Hz), 114.8, 94.1 (d, $J = 3.2$ Hz), 88.4, 58.1, 40.7. IR (cm$^{-1}$): 752, 784, 948, 1202, 1231, 1494, 1578, 1721, 2884, 3068. HRMS m/z (ESI) calcd for C$_{17}$H$_{14}$FNNaO$_2$$^{[M+Na]^{+}}$: 306.0906, found: 306.0903.

N-methyl-N-(2-(m-tolylethynyl)phenyl)glycine (1l)

Brown oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 10.36 (s, 1H), 7.55 (dd, $J = 7.6, 1.7$ Hz, 1H), 7.39-7.29 (m, 3H), 7.24 (t, $J = 7.5$ Hz, 1H), 7.16 (d, $J = 7.6$ Hz, 1H), 7.09 (d, $J = 8.1$ Hz, 1H), 7.05 (t, $J = 7.5$ Hz, 1H), 4.15 (s, 2H), 2.99 (s, 3H), 2.37 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$C 174.4, 152.2, 138.1, 134.3, 132.0, 129.4, 129.3, 128.6, 128.3, 122.8, 122.5, 118.8, 116.0, 95.9, 87.0, 58.3, 41.0, 21.3. IR (cm$^{-1}$): 750, 948, 1047, 1358, 1493, 1593, 1715, 2920. HRMS m/z (ESI) calcd for C$_{18}$H$_{18}$NNaO$_2^{+}$ $^{[M+Na]^{+}}$: 302.1157, found: 302.1157.

N-(4-fluoro-2-(phenylethynyl)phenyl)-N-methylglycine (1m)
Yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)H 10.04 (s, 1H), 7.55 (dd, \(J = 6.5, 2.9\) Hz, 2H), 7.38-7.33 (m, 3H), 7.24 (dd, \(J = 8.7, 2.7\) Hz, 1H), 7.10-6.98 (m, 2H), 4.10 (s, 2H), 2.96 (s, 3H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta\)C 173.5, 158.2 (d, \(J = 242.9\) Hz), 148.6 (d, \(J = 2.5\) Hz), 131.6, 128.9, 128.5, 122.4, 120.7 (d, \(J = 8.6\) Hz), 120.2 (d, \(J = 23.6\) Hz), 118.2 (d, \(J = 9.3\) Hz), 116.3 (d, \(J = 22.5\) Hz), 96.4, 85.9, 58.7, 41.6. IR (cm\(^{-1}\)): 756, 871, 944, 1120, 1195, 1413, 1488, 1723, 2886, 3059. HRMS m/z (ESI) calcd for C\(_{17}\)H\(_{14}\)FNNaO\(_2^+\) [M+Na\(^+\): 306.0906, found: 306.0907.

N-methyl-N-(4-methyl-2-(phenylethynyl)phenyl)glycine (1n)

Yellow solid, mp = 97.0-98.3 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)H 9.30 (s, 1H), 7.55 (dd, \(J = 6.5, 3.1\) Hz, 2H), 7.41-7.33 (m, 4H), 7.14 (dd, \(J = 8.3, 1.7\) Hz, 1H), 7.02 (d, \(J = 8.3\) Hz, 1H), 4.04 (s, 2H), 2.94 (s, 3H), 2.33 (s, 3H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta\)C 173.5, 149.9, 134.5, 132.8, 131.5, 130.2, 128.5, 128.4, 122.9, 119.1, 116.5, 95.4, 87.1, 59.1, 41.4, 20.5. IR (cm\(^{-1}\)): 756, 950, 1094, 1192, 1353, 1503, 1722, 2920. HRMS m/z (ESI) calcd for C\(_{18}\)H\(_{17}\)NNaO\(_2^+\) [M+Na\(^+\): 302.1157, found: 302.1159.

N-(5-chloro-2-(phenylethynyl)phenyl)-N-methylglycine (1o)

Purple solid, mp = 111.4-112.3 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)H 9.56 (s, 1H), 7.51-7.47 (m, 2H), 7.42 (d, \(J = 8.2\) Hz, 1H), 7.33-7.29 (m, 3H), 7.00 (d, \(J = 1.9\) Hz, 1H), 6.95 (dd, \(J = 8.2, 2.0\) Hz, 1H), 4.22 (s, 2H), 2.97 (s, 3H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta\)C 174.8, 153.1, 135.2, 131.4, 128.6, 128.4, 122.8, 122.0, 118.7, 113.3, 96.3, 86.6, 57.4, 40.6. IR (cm\(^{-1}\)): 755, 953, 1201, 1236, 1406, 1496, 1585, 1721, 2961, 3061. HRMS m/z (ESI) calcd for C\(_{18}\)H\(_{16}\)ClNNaO\(_2^+\) [M+Na\(^+\): 322.0611, found: 322.0610.

N-methyl-N-(2-(thiophen-3-ylethynyl)phenyl)glycine (1p)
Yellow oil. **^1H NMR** (600 MHz, CDCl\(_3\)) \(\delta\) H 10.84 (s, 1H), 7.55 (d, \(J = 2.1\) Hz, 1H), 7.51 (d, \(J = 6.9\) Hz, 1H), 7.29 (t, \(J = 7.3\) Hz, 1H), 7.27-7.25 (m, 1H), 7.19 (d, \(J = 4.8\) Hz, 1H), 7.07 (d, \(J = 8.2\) Hz, 1H), 7.02 (t, \(J = 7.4\) Hz, 1H), 4.07 (s, 2H), 2.94 (s, 3H). **^13C NMR** (151 MHz, CDCl\(_3\)) \(\delta\) C 174.0, 152.2, 134.1, 129.8, 129.3, 129.0, 125.5, 122.8, 121.9, 118.6, 116.0, 91.0, 86.6, 58.9, 41.0. **IR** (cm\(^{-1}\)): 747, 841, 1076, 1238, 1373, 1465, 1524, 1609, 1720, 2931, 3105. **HRMS** m/z (ESI) calcd for C\(_{15}\)H\(_{13}\)NNaO\(_2\)S\(^+\) [M+Na\(^+\)]: 294.0565, found: 294.0561.

N-methyl-N-(2-(thiophen-2-ylethynyl)phenyl)glycine (1q)

![Structure of N-methyl-N-(2-(thiophen-2-ylethynyl)phenyl)glycine (1q)](image)

Brown solid, mp = 110.4-111.2 °C. **^1H NMR** (600 MHz, CDCl\(_3\)) \(\delta\) H 10.51 (s, 1H), 7.49 (d, \(J = 7.6\) Hz, 1H), 7.05 (d, \(J = 8.2\) Hz, 1H), 7.02-6.94 (m, 2H), 4.11 (s, 2H), 2.96 (s, 3H). **^13C NMR** (151 MHz, CDCl\(_3\)) \(\delta\) C 174.6, 152.2, 134.0, 132.2, 129.6, 127.6, 127.2, 122.9, 122.4, 118.6, 115.3, 90.9, 88.9, 58.3, 40.8. **IR** (cm\(^{-1}\)): 749, 853, 951, 1039, 1119, 1246, 1405, 1489, 1593, 1694, 2883. **HRMS** m/z (ESI) calcd for C\(_{15}\)H\(_{13}\)NNaO\(_2\)S\(^+\) [M+Na\(^+\)]: 294.0565, found: 294.0559.

N-(2-(3,3-dimethylbut-1-yn-1-yl)phenyl)-N-methylglycine (1r)

Yellow oil. **^1H NMR** (600 MHz, CDCl\(_3\)) \(\delta\) H 10.91 (s, 1H), 7.37 (dd, \(J = 7.6, 1.5\) Hz, 1H), 7.21 (d, \(J = 8.2\) Hz, 1H), 7.00 (t, \(J = 7.5\) Hz, 1H), 6.94 (t, \(J = 7.5\) Hz, 1H), 4.07 (s, 2H), 2.89 (s, 3H), 1.31 (s, 9H). **^13C NMR** (151 MHz, CDCl\(_3\)) \(\delta\) C 174.4, 151.7, 134.4, 128.6, 122.6, 118.8, 116.8, 105.0, 77.0, 58.2, 41.0, 30.8, 28.3. **IR** (cm\(^{-1}\)): 750, 954, 1097, 1198, 1283, 1361, 1490, 1594, 1723, 2868, 2967. **HRMS** m/z (ESI) calcd for C\(_{15}\)H\(_{19}\)NNaO\(_2\)S\(^+\) [M+Na\(^+\)]: 268.1313, found: 268.1311.

2-(2-(phenylethynyl)phenoxy)acetic acid (3a)

Pale yellow solid, mp = 94.2-97.0 °C. **^1H NMR** (400 MHz, DMSO) \(\delta\) H 13.13 (s, 1H), 7.59-7.53 (m, 2H), 7.51 (d, \(J = 7.6, 1.3\) Hz, 1H), 7.47-7.40 (m, 3H), 7.36 (t, \(J = 8.6\) Hz, 1H), 7.00 (t, \(J = 7.5\) Hz, 1H), 6.96 (d, \(J = 8.4\) Hz, 1H), 4.82 (s, 2H). **^13C NMR** (100 MHz, DMSO) \(\delta\) C 167.0, 158.2, 133.1, 131.2, 130.1, 128.7, 128.6, 122.7, 121.0, 112.2, 111.5, 93.1, 86.1, 64.8. **IR** (cm\(^{-1}\)): 1705, 1593, 1574, 1497, 1482, 1447, 1218, 1163, 1114, 1071, 920, 783, 755, 689. **HRMS** m/z (ESI) calcd for C\(_{16}\)H\(_{13}\)O\(_3\) [M+H\(^+\)]: 253.0865, found: 253.0860.

2-phenyl-2-(2-(phenylethynyl)phenoxy)acetic acid (3b)
Pale yellow solid, mp = 162.3-164.3 °C. $^1$H NMR (400 MHz, DMSO) $\delta_{H}$ 13.33 (s, 1H), 7.68 (d, $J = 7.1$ Hz, 2H), 7.55-7.49 (m, 3H), 7.47-7.35 (m, 7H), 7.05-6.98 (m, 2H), 5.97 (s, 1H). $^1$C NMR (100 MHz, DMSO) $\delta_{C}$ 170.6, 157.7, 136.1, 132.8, 131.1, 130.1, 128.8, 128.7, 128.6, 128.5, 127.0, 122.7, 121.4, 113.2, 113.2, 93.5, 86.0, 77.4. IR (cm$^{-1}$): 1697, 1593, 1496, 1241, 1227, 955, 752, 718, 688. HRMS m/z (ESI) calcd for C$_{23}$H$_{16}$NaO$_3^+$ [M+Na]$^+$: 351.0997, found: 351.0994.

2-(2-(phenylethynyl)phenoxy)propanoic acid (3c)

Pale yellow solid, mp = 107.2-110.7 °C. $^1$H NMR (400 MHz, DMSO) $\delta_{H}$ 13.23 (s, 1H), 7.56-7.47 (m, 3H), 7.47-7.39 (m, 3H), 7.34 (t, $J = 7.9$ Hz, 1H), 6.99 (t, $J = 7.5$ Hz, 1H), 6.90 (d, $J = 8.4$ Hz, 1H), 4.90 (q, $J = 6.7$ Hz, 1H), 1.58 (d, $J = 6.7$ Hz, 3H). $^1$C NMR (100 MHz, DMSO) $\delta_{C}$ 173.4, 158.6, 133.5, 131.7, 130.6, 129.2, 129.0, 123.3, 121.5, 113.7, 112.5, 93.6, 86.7, 73.0, 18.8. IR (cm$^{-1}$): 1699, 1498, 1482, 1446, 1285, 1275, 1240, 1134, 1044, 757, 692. HRMS m/z (ESI) calcd for C$_{17}$H$_{14}$NaO$_3^+$ [M+Na]$^+$: 289.0841, found: 289.0841.

2-(2-chlorophenyl)-2-(2-(phenylethynyl)phenoxy)acetic acid (3d)

Pale yellow solid, mp = 107.0-110.2 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta_{H}$ 11.01 (s, 1H), 7.82-7.74 (m, 1H), 7.53-7.47 (m, 3H), 7.41-7.36 (m, 1H), 7.33- 7.25 (m, 5H), 7.19 (t, $J = 7.7$ Hz, 1H), 6.99 (t, $J = 7.5$ Hz, 1H), 6.84 (d, $J = 8.3$ Hz, 1H), 6.24 (s, 1H). $^1$C NMR (100 MHz, CDCl$_3$) $\delta_{C}$ 173.5, 157.2, 133.5, 133.4, 133.1, 131.6, 130.5, 129.9, 129.7, 129.0, 128.4, 127.5, 123.3, 122.7, 114.7, 114.4, 94.7, 85.2, 75.9. IR (cm$^{-1}$): 1720, 1594, 1574, 1498, 1484, 1447, 1233, 1198, 1109, 931, 746, 723, 687. HRMS m/z (ESI) calcd for C$_{22}$H$_{16}$ClO$_3^+$ [M+H]$^+$: 363.0788, found: 363.0781.

2-(3-chlorophenyl)-2-(2-(phenylethynyl)phenoxy)acetic acid (3e)

White solid, mp = 169.3-170.6 °C. $^1$H NMR (400 MHz, DMSO) $\delta_{H}$ 13.54 (s, 1H), 7.78 (s, 1H), 7.66-7.60 (m, 1H), 7.59-7.52 (m, 3H), 7.51-7.42 (m, 5H), 7.39 (t, $J = 7.8$ Hz, 1H), 7.04 (t, $J = 8.3$ Hz, 2H), 6.07 (s, 1H). $^1$C NMR (100 MHz, DMSO) $\delta_{C}$ 170.2, 157.4, 138.4, 133.2, 132.8, 131.1, 130.5, 130.2, 128.8, 128.7, 128.6, 126.4, 125.7, 122.6, 121.5, 113.0,
112.3, 93.6, 85.9, 76.5. IR (cm⁻¹): 1698, 1593, 1575, 1497, 1481, 1286, 1277, 1237, 1226, 1115, 1070, 766, 751, 715, 688. HRMS m/z (ESI) calcd for C_{22}H_{15}ClNaO_3^+ [M+Na]^+: 385.0607, found: 385.0603.

2-(4-chlorophenyl)-2-(2-(phenylethynyl)phenoxy)acetic acid (3f)

\[
\begin{align*}
\text{Cl} & \quad \text{Ph} \\
\text{Ph} & \quad \text{O} \\
\text{O} & \quad \text{COOH}
\end{align*}
\]

White soild, mp = 150.6-153.1 °C. ¹H NMR (400 MHz, DMSO) δ_H 13.43 (s, 1H), 7.69 (d, J = 8.5 Hz, 2H), 7.58-7.50 (m, 5H), 7.49-7.41 (m, 3H), 7.37 (td, J = 8.3, 1.6 Hz, 1H), 7.07-6.97 (m, 2H), 6.03 (s, 1H). ¹³C NMR (100 MHz, DMSO) δ_C 170.3, 157.4, 135.1, 133.4, 132.9, 131.1, 130.1, 128.9, 128.7, 128.6, 122.7, 121.5, 113.3, 112.4, 93.6, 85.9, 76.7. IR (cm⁻¹): 1703, 1595, 1574, 1498, 1451, 1287, 1243, 1093, 1016, 934, 753, 686. HRMS m/z (ESI) calcd for C_{22}H_{15}ClNaO_3^+ [M+Na]^+: 385.0607, found: 385.0607.

2-(2-((4-fluorophenyl)ethynyl)phenoxy)-2-phenylacetic acid (3g)

\[
\begin{align*}
\text{F} & \quad \text{Ph} \\
\text{Ph} & \quad \text{O} \\
\text{O} & \quad \text{COOH}
\end{align*}
\]

Pale yellow soild, mp = 177.4-181.6 °C. ¹H NMR (400 MHz, DMSO) δ_H 13.34 (s, 1H), 7.68 (d, J = 7.2 Hz, 2H), 7.57 (dd, J = 8.6, 5.6 Hz, 2H), 7.52 (dd, J = 7.7, 1.3 Hz, 1H), 7.49-7.43 (m, 2H), 7.42-7.34 (m, 2H), 7.31 (t, J = 8.8 Hz, 2H), 7.07-6.96 (m, 2H), 5.97 (s, 1H). ¹³C NMR (100 MHz, DMSO) δ_C 170.6, 161.9 (d, J = 247.6 Hz), 157.7, 136.1, 133.3 (d, J = 8.6 Hz), 132.7, 130.2, 128.7, 128.5, 127.0, 121.4, 119.2 (d, J = 3.2 Hz), 116.1 (d, J = 22.1 Hz), 113.2, 112.2, 92.4, 85.8, 77.4. IR (cm⁻¹): 1698, 1593, 1509, 1485, 1447, 1285, 1228, 1115, 831, 752, 718, 689. HRMS m/z (ESI) calcd for C_{22}H_{15}FNaO_3^+ [M+Na]^+: 369.0903, found: 369.0901.

2-(2-((4-chlorophenyl)ethynyl)phenoxy)-2-phenylacetic acid (3h)

\[
\begin{align*}
\text{Cl} & \quad \text{Ph} \\
\text{Ph} & \quad \text{O} \\
\text{O} & \quad \text{COOH}
\end{align*}
\]

White soild, mp = 180.2-185.9 °C. ¹H NMR (400 MHz, DMSO) δ_H 13.35 (s, 1H), 7.68 (d, J = 7.6 Hz, 2H), 7.56-7.51 (m, 5H), 7.46 (t, J = 7.3 Hz, 2H), 7.41 (d, J = 6.9, 7.4 Hz, 1H), 7.37 (d, J = 7.8 Hz, 1H), 7.07-7.00 (m, 2H), 5.98 (s, 1H). ¹³C NMR (100 MHz, DMSO) δ_C 171.1, 158.3, 136.6, 133.8, 133.3, 133.2, 130.8, 129.5, 129.2, 129.0, 127.5, 122.1, 121.9, 113.7, 112.6, 92.8, 87.7, 78.0. IR (cm⁻¹): 1699, 1495, 1480, 1447, 1278, 1088, 823, 752, 717, 688. HRMS m/z (ESI) calcd for C_{22}H_{15}ClNaO_3^+ [M+Na]^+: 385.0607, found: 385.0597.

2-phenyl-2-(2-(p-tolylethynyl)phenoxy)acetic acid (3i)
Pale yellow solid, mp = 175.9-178.0 °C. **^1^H NMR** (400 MHz, DMSO) δ H 13.34 (s, 1H), 7.70 (d, J = 7.1 Hz, 2H), 7.51 (dd, J = 7.6, 1.2 Hz, 1H), 7.48-7.33 (m, 6H), 7.26 (d, J = 8.0 Hz, 2H), 7.02 (dd, J = 7.9, 4.9 Hz, 2H), 5.96 (s, 1H), 2.35 (s, 3H). **^13^C NMR** (100 MHz, DMSO) δ C 171.1, 158.1, 138.9, 136.6, 133.2, 131.5, 130.4, 129.9, 129.2, 129.0, 127.5, 121.9, 120.3, 113.7, 113.1, 94.2, 85.9, 78.0, 21.5. **IR** (cm⁻¹): 1699, 1596, 1517, 1497, 1485, 1450, 1277, 1243, 1190, 1114, 1065, 951, 812, 750, 717, 688. **HRMS** m/z (ESI) calcd for C23H18NaO3+ [M+Na]^+: 365.1154, found: 365.1150.

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2-(2-((4-ethylphenyl)ethynyl)phenoxy)-2-phenylacetic acid (3j)

White solid, mp = 153.6-156.6 °C. **^1^H NMR** (400 MHz, DMSO) δ H 13.34 (s, 1H), 7.69 (d, J = 7.0 Hz, 2H), 7.50 (dd, J = 7.8, 1.5 Hz, 1H), 7.48-7.33 (m, 6H), 7.29 (d, J = 8.1 Hz, 2H), 7.05-6.96 (m, 2H), 5.96 (s, 1H), 2.64 (q, J = 7.6 Hz, 2H), 1.20 (t, J = 7.6 Hz, 3H). **^13^C NMR** (100 MHz, DMSO) δ C 170.7, 157.6, 144.6, 136.1, 132.7, 131.1, 129.9, 128.7, 128.5, 128.2, 127.0, 121.3, 120.0, 113.1, 112.5, 93.7, 85.4, 77.4, 28.1, 15.3. **IR** (cm⁻¹): 1699, 1595, 1514, 1485, 1448, 1276, 1240, 1224, 1114, 1063, 828, 752, 717, 688. **HRMS** m/z (ESI) calcd for C24H21O3+ [M+H]^+: 357.1491, found: 357.1496.

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2-(2-((4-methoxyphenyl)ethynyl)phenoxy)-2-phenylacetic acid (3k)

Pale yellow solid, mp = 157.6-158.0 °C. **^1^H NMR** (400 MHz, DMSO) δ H 13.33 (s, 1H), 7.70 (d, J = 7.2 Hz, 2H), 7.51-7.38 (m, 6H), 7.34 (t, J = 7.9 Hz, 1H), 7.01 (t, J = 7.8 Hz, 4H), 5.96 (s, 1H), 3.81 (s, 3H). **^13^C NMR** (100 MHz, DMSO) δ C 171.2, 159.9, 158.0, 136.6, 133.1, 133.0, 130.1, 129.2, 129.0, 127.5, 121.8, 115.2, 115.0, 113.7, 113.3, 94.2, 85.1, 78.0, 55.8. **IR** (cm⁻¹): 1698, 1608, 1517, 1485, 1289, 1247, 1222, 752, 717, 692. **HRMS** m/z (ESI) calcd for C23H19O4+ [M+H]^+: 359.1283, found: 359.1286.

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2-(2-((3-fluorophenyl)ethynyl)phenoxy)-2-phenylacetic acid (3l)

Pale yellow solid, mp = 168.7-172.0 °C. **^1^H NMR** (400 MHz, DMSO) δ H 13.35 (s, 1H), 7.67 (d, J = 6.9 Hz, 2H), 7.55-7.26 (m, 9H), 7.03 (dd, J = 7.8, 5.6 Hz, 2H), 5.98 (s, 1H). **^13^C NMR** (100 MHz, DMSO) δ C 170.6, 161.9 (d, J = 244.8 Hz), 157.8, 136.1, 132.8, 131.0 (d, J = 8.9 Hz), 130.5, 128.7, 128.5, 127.4 (d, J = 2.3 Hz), 127.0, 124.7 (d, J = 9.7 Hz),
121.4, 117.6 (d, \( J = 22.9 \) Hz), 115.9 (d, \( J = 21.0 \) Hz), 113.3, 111.9, 92.2 (d, \( J = 2.8 \) Hz), 87.1, 77.4. IR (cm\(^{-1}\)): 1698, 1577, 1495, 1448, 1279, 1227, 1229, 943, 788, 751, 718, 680. HRMS m/z (ESI) calcd for C\(_{22}\)H\(_{16}\)F\(_3\)O\(^+\) [M+H]\(^+\): 347.1083, found: 347.1082.

2-phenyl-2-(2-(m-tolylethynyl)phenoxy)acetic acid (3m)

\[
\text{Pale yellow solid, mp} = 168.7-172.6 \, ^{\circ}\text{C}. \quad \text{\( ^1H \) NMR} (400 MHz, DMSO) \delta 13.34 (s, 1H), 7.70 (d, \( J = 7.0 \) Hz, 2H), 7.51 (d, \( J = 6.6 \) Hz, 1H), 7.49-7.29 (m, 7H), 7.24 (d, \( J = 6.5 \) Hz, 1H), 7.02 (t, \( J = 7.2 \) Hz, 2H), 5.97 (s, 1H), 2.34 (s, 3H).
\]

\[
\text{\( ^{13}C \) NMR} (100 MHz, DMSO) \delta 170.7, 157.7, 138.1, 136.1, 132.7, 131.6, 130.0, 129.4, 128.7, 128.6, 128.5, 128.1, 127.0, 122.6, 121.4, 113.1, 112.4, 93.7, 85.7, 77.4, 20.8. \quad \text{IR (cm\(^{-1}\))}: 1698, 1593, 1574, 1494, 1446, 1277, 1228, 1115, 1065, 955, 768, 752, 716, 689. \quad \text{HRMS} \quad \text{m/z (ESI) calcd for C\(_{23}\)H\(_{18}\)NaO\(_3\) [M+Na]\(^+\): 365.1154, found: 365.1152.}
\]

2-phenyl-2-(2-(thiophen-3-ylethynyl)phenoxy)acetic acid (3n)

Yellow solid, mp = 177.9-182.8 \(^{\circ}\)C. \( ^1H \) NMR (400 MHz, DMSO) \( \delta \) 13.33 (s, 1H), 7.82 (d, \( J = 2.0 \) Hz, 1H), 7.74-7.66 (m, 3H), 7.52-7.34 (m, 5H), 7.52 -7.34 (m, 5H), 7.22 (d, \( J = 4.8 \) Hz, 1H), 7.05-6.98 (m, 2H), 5.96 (s, 1H). \( ^{13}C \) NMR (100 MHz, DMSO) \( \delta \)c 171.1, 158.1, 136.6, 133.2, 130.4, 129.9, 129.8, 129.2, 129.0, 127.5, 127.4, 122.1, 121.9, 113.7, 113.0, 89.5, 85.8, 78.0. IR (cm\(^{-1}\)): 1699, 1597, 1485, 1448, 1273, 1239, 1223, 1190, 1113, 773, 750, 717, 689, 623. HRMS m/z (ESI) calcd for C\(_{20}\)H\(_{15}\)O\(_3\)S\(^+\) [M+H]\(^+\): 335.0742, found: 335.0739.

2-phenyl-2-(2-(thiophen-2-ylethynyl)phenoxy)acetic acid (3o)

Yellow solid, mp = 168.5-174.0 \(^{\circ}\)C. \( ^1H \) NMR (400 MHz, DMSO) \( \delta \) 13.37 (s, 1H), 7.74-7.66 (m, 3H), 7.52 (d, \( J = 6.5 \) Hz, 1H), 7.46 (t, \( J = 7.3 \) Hz, 2H), 7.44-7.36 (m, 3H), 7.15 (dd, \( J = 5.0, 3.8 \) Hz, 1H), 7.06-6.99 (m, 2H), 5.98 (s, 1H). \( ^{13}C \) NMR (100 MHz, DMSO) \( \delta \)c 171.1, 158.0, 136.5, 132.9, 132.6, 130.7, 129.3, 129.1, 129.0, 128.3, 127.4, 122.9, 121.9, 113.5, 112.5, 90.2, 87.2, 77.9. IR (cm\(^{-1}\)): 1699, 1484, 1450, 1274, 1241, 1224, 1214, 952, 751, 719, 690. HRMS m/z (ESI) calcd for C\(_{20}\)H\(_{15}\)O\(_3\)S\(^+\) [M+H]\(^+\): 335.0742, found: 335.0741.

2-(2-(3,3-dimethylbut-1-yn-1-yl)phenoxy)-2-phenylacetic acid (3p)
White solid, mp = 90.1-93.5 °C. $^1$H NMR (400 MHz, DMSO) $\delta$H 13.27 (s, 1H), 7.66 (d, $J = 6.6$ Hz, 2H), 7.44-7.37 (m, 3H), 7.32 (dd, $J = 7.5, 1.4$ Hz, 1H), 7.27 (t, $J = 7.9$ Hz, 1H), 6.95-6.88 (m, 2H), 5.84 (s, 1H), 1.29 (s, 9H). $^{13}$C NMR (100 MHz, DMSO) $\delta$C 170.8, 157.5, 136.2, 132.6, 129.0, 128.5, 126.8, 121.1, 113.2, 113.0, 102.6, 77.3, 75.2, 30.8, 27.8. IR (cm$^{-1}$): 2968, 1703, 1490, 1450, 1276, 1240, 1222, 1122, 752, 719, 692. HRMS m/z (ESI) calcd for C$_{20}$H$_{20}$NaO$_3^+$ [M+Na]$^+$: 331.1310, found: 331.1308.

2-phenyl-2-((1-(phenylethynyl)naphthalen-2-yl)oxy)acetic acid (3q)

Pale yellow solid, mp = 151.9-154.7 °C. $^1$H NMR (400 MHz, DMSO) $\delta$H 8.30 (d, $J = 8.4$ Hz, 1H), 7.99 (d, $J = 9.1$ Hz, 1H), 7.93 (d, $J = 8.1$ Hz, 1H), 7.75 (d, $J = 7.2$ Hz, 2H), 7.70-7.62 (m, 3H), 7.54-7.40 (m, 8H), 6.21 (s, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$C 170.7, 156.9, 156.2, 133.4, 131.1, 130.4, 128.9, 128.7, 128.6, 128.4, 128.3, 127.8, 127.2, 124.7, 124.6, 123.0, 115.0, 106.4, 98.9, 84.2, 78.0. IR (cm$^{-1}$): 1702, 1589, 1509, 1491, 1267, 1227, 803, 754, 719, 690. HRMS m/z (ESI) calcd for C$_{26}$H$_{18}$O$_2$Na$^+$ [M+Na]$^+$: 401.1154, found: 401.1150.

2-phenyl-2-((2-(phenylethynyl)phenyl)thio)acetic acid (5a)

Yellow solid, mp = 90.8-93.6 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 9.88 (s, 1H), 7.57-7.50 (m, 3H), 7.46 (dd, $J = 6.4, 2.8$ Hz, 2H), 7.38 (d, $J = 7.8$ Hz, 1H), 7.35-7.32 (m, 3H), 7.31-7.27 (m, 3H), 7.25-7.20 (m, 8H), 5.19 (s, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$C 175.9, 135.7, 134.8, 133.0, 132.4, 131.8, 128.9, 128.8, 128.7, 128.6, 128.4, 127.9, 126.2, 122.9, 95.5, 87.3, 54.5. IR (cm$^{-1}$): 1710, 1598, 1492, 756, 720, 691. HRMS m/z (ESI) calcd for C$_{22}$H$_{17}$O$_2$S$^+$ [M+H]$^+$: 345.0949, found: 345.0953.

2-((2-(phenylethynyl)phenyl)thio)acetic acid (5b)

Pale yellow solid, mp = 117.7-120.2 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 10.78 (s, 1H), 7.60 (dd, $J = 6.5, 3.0$ Hz, 2H), 7.56 (dd, $J = 7.6, 1.2$ Hz, 1H), 7.43 (d, $J = 7.9$ Hz, 1H), 7.41-7.35 (m, 3H), 7.32 (td, $J = 7.7, 1.3$ Hz, 1H), 7.25 (t, $J = 7.2$
Hz, 1H), 3.80 (s, 2H). \(^{13}\text{C}\) NMR (100 MHz, CDCl\(_3\)) \(\delta\)C 175.6, 137.2, 132.8, 131.7, 129.0, 128.7, 128.5, 128.4, 126.6, 123.8, 122.9, 96.0, 86.8, 35.1. IR (cm\(^{-1}\)): 1710, 1598, 1491, 1295, 755, 719, 690. HRMS m/z (ESI) calcd for C\(_{16}\)H\(_{13}\)O\(_2\)S\(^{+}\) [M+H]\(^{+}\): 269.0636, found: 269.0635.

2-((2-(phenylethynyl)phenyl)thio)propanoic acid (5c)

![2-((2-(phenylethynyl)phenyl)thio)propanoic acid (5c)](image)

Yellow oil. \(^1\text{H}\) NMR (400 MHz, CDCl\(_3\)) \(\delta\)H 10.65 (s, 1H), 7.62-7.57 (m, 3H), 7.54 (dd, \(J\) = 5.8, 3.3 Hz, 1H), 7.41-7.36 (m, 3H), 7.31-7.26 (m, 2H), 4.05 (q, \(J\) = 7.2 Hz, 1H), 1.58 (d, \(J\) = 7.2 Hz, 3H). \(^{13}\text{C}\) NMR (100 MHz, CDCl\(_3\)) \(\delta\)C 178.8, 135.8, 133.0, 132.3, 131.7, 128.8, 128.6, 128.4, 127.7, 126.1, 123.0, 95.3, 87.4, 44.1, 17.1. IR (cm\(^{-1}\)): 3449, 1726, 1598, 1492, 1014, 757, 720, 691. HRMS m/z (ESI) calcd for C\(_{17}\)H\(_{13}\)O\(_2\)S\(^{+}\) [M+H]\(^{+}\): 283.0793, found: 283.0794.

2-phenyl-2-((2-(p-tolylethynyl)phenyl)thio)acetic acid (5d)

![2-phenyl-2-((2-(p-tolylethynyl)phenyl)thio)acetic acid (5d)](image)

Pale yellow solid, mp = 113.5-115.3 °C. \(^1\text{H}\) NMR (400 MHz, DMSO) \(\delta\)H 13.24 (s, 1H), 7.56-7.49 (m, 3H), 7.44 (d, \(J\) = 8.0 Hz, 2H), 7.40-7.30 (m, 5H), 5.41 (s, 1H), 2.35 (s, 3H). \(^{13}\text{C}\) NMR (100 MHz, DMSO) \(\delta\)C 171.1, 138.9, 137.4, 135.9, 132.4, 131.2, 129.4, 129.1, 128.7, 128.5, 128.4, 128.2, 126.3, 122.3, 119.0, 95.5, 86.4, 53.0, 21.1. IR (cm\(^{-1}\)): 1708, 1599, 1495, 816, 749, 700. HRMS m/z (ESI) calcd for C\(_{23}\)H\(_{19}\)O\(_2\)S\(^{+}\) [M+H]\(^{+}\): 359.1106, found: 359.1103.

2-((2-((4-chlorophenyl)ethynyl)phenyl)thio)-2-phenylacetic acid (5e)

![2-((2-((4-chlorophenyl)ethynyl)phenyl)thio)-2-phenylacetic acid (5e)](image)

Yellow solid, mp = 124.7-128.9 °C. \(^1\text{H}\) NMR (400 MHz, CDCl\(_3\)) \(\delta\)H 10.38 (s, 1H), 7.52 (d, \(J\) = 7.3 Hz, 1H), 7.48-7.40 (m, 4H), 7.37 (d, \(J\) = 7.6 Hz, 1H), 7.33-7.27 (m, 5H), 7.25-7.18 (m, 2H), 5.13 (s, 1H). \(^{13}\text{C}\) NMR (100 MHz, CDCl\(_3\)) \(\delta\)C 176.3, 135.8, 134.7, 134.6, 133.0, 132.9, 132.4, 129.0, 128.9, 128.8, 128.7, 128.6, 127.9, 125.9, 121.4, 94.3, 88.3, 54.7. IR (cm\(^{-1}\)): 1709, 1583, 1491, 1092, 1014, 828, 755, 720, 696. HRMS m/z (ESI) calcd for C\(_{22}\)H\(_{16}\)ClO\(_2\)S\(^{+}\) [M+H]\(^{+}\): 379.0560, found: 379.0560.

2-((2-((3-fluorophenyl)ethynyl)phenyl)thio)-2-phenylacetic acid (5f)

![2-((2-((3-fluorophenyl)ethynyl)phenyl)thio)-2-phenylacetic acid (5f)](image)
Yellow oil. **1H NMR** (400 MHz, CDCl$_3$) $\delta$H 11.80 (s, 1H), 7.61 (dd, $J = 7.3, 1.5$ Hz, 1H), 7.55 (dd, $J = 6.5, 2.8$ Hz, 2H), 7.47 (d, $J = 8.6$ Hz, 1H), 7.40-7.32 (m, 5H), 7.10 (t, $J = 8.0$ Hz, 1H), 5.23 (s, 1H). **13C NMR** (100 MHz, CDCl$_3$) $\delta$C 177.0, 162.4 (d, $J = 246.9$ Hz), 136.1, 134.8, 133.2, 132.3, 130.1 (d, $J = 8.6$ Hz), 129.2, 128.9, 128.8, 128.7, 128.0, 127.7 (d, $J = 2.5$ Hz), 125.7, 124.8 (d, $J = 9.6$ Hz), 118.5 (d, $J = 22.6$ Hz), 116.0 (d, $J = 21.1$ Hz), 94.2, 88.3, 54.8. **IR** (cm$^{-1}$): 1709, 1608, 1579, 1489, 1455, 1435, 1287, 1208, 943, 872, 785, 755, 681. **HRMS** m/z (ESI) calcd for C$_{22}$H$_{16}$FO$_2$S$^+$ [M+H]$^+$: 363.0855, found: 363.0850.

2-phenyl-2-((2-(thiophen-3-ylethynyl)phenyl)thio)acetic acid (5g)

Pale yellow solid, mp = 126.3-128.6°C. **1H NMR** (400 MHz, CDCl$_3$) $\delta$H 9.78 (s, 1H), 7.56-7.49 (m, 2H), 7.45 (d, $J = 6.1, 2.4$ Hz, 2H), 7.37 (d, $J = 7.7$ Hz, 1H), 7.32-7.27 (m, 4H), 7.22 (d, $J = 7.4$ Hz, 1H), 7.20-7.15 (m, 2H), 5.18 (s, 1H). **13C NMR** (100 MHz, CDCl$_3$) $\delta$C 176.1, 135.5, 134.8, 132.9, 132.5, 129.9, 129.3, 128.7, 128.6, 128.5, 127.9, 126.3, 125.5, 122.0, 90.7, 86.9, 54.5. **IR** (cm$^{-1}$): 3434, 1708, 1583, 1495, 756, 736, 697. **HRMS** m/z (ESI) calcd for C$_{20}$H$_{15}$O$_2$S$^+$ [M+H]$^+$: 351.0513, found: 351.0511.

2-((2-(3,3-dimethylbut-1-yn-1-yl)phenyl)thio)-2-phenylacetic acid (5h)

White oil. **1H NMR** (400 MHz, CDCl$_3$) $\delta$H 10.35 (s, 1H), 7.44 (d, $J = 6.5$ Hz, 2H), 7.37 (d, $J = 7.5$ Hz, 1H), 7.31-7.22 (m, 4H), 7.12 (t, $J = 7.4$ Hz, 1H), 7.06 (t, $J = 7.4$ Hz, 1H), 5.16 (s, 1H), 1.33 (s, 9H). **13C NMR** (100 MHz, CDCl$_3$) $\delta$C 176.5, 135.3, 135.1, 132.9, 131.9, 128.7, 128.6, 128.4, 128.0, 127.5, 126.6, 105.0, 54.3, 30.9, 28.3. **IR** (cm$^{-1}$): 2969, 1708, 1599, 1583, 1462, 1385, 1292, 751, 736, 697. **HRMS** m/z (ESI) calcd for C$_{20}$H$_{21}$O$_2$S$^+$ [M+H]$^+$: 325.1262, found: 325.1259.

6. Characterization of products

(1-methyl-1H-indol-3-yl)(phenyl)methanone (2a)
Yellow solid (13.7 mg, 58%), mp = 110.5-111.6 °C. ¹H NMR (400 MHz, CDCl₃) δ_H 8.46-8.39 (m, 1H), 7.81 (d, J = 7.4 Hz, 2H), 7.56-7.45 (m, 4H), 7.39-7.32 (m, 3H), 3.83 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ_C 190.9, 141.0, 138.0, 137.6, 131.1, 128.7, 128.3, 127.2, 122.8, 122.7, 115.6, 109.7, 33.6. IR (cm⁻¹): 746, 874, 1073, 1125, 1232, 1368, 1465, 1524, 1616, 2933, 3054, 3110. GC-MS (EI): 235.2, 158.1, 130.1, 103.1, 77.1. HRMS m/z (ESI) calcd for C₁₆H₁₃NNaO⁺ [M+Na]⁺: 258.0895, found: 258.0887.

(1-ethyl-1H-indol-3-yl)(phenyl)methanone (2b)

Yellow oil (14.0 mg, 56%). ¹H NMR (600 MHz, CDCl₃) δ_H 8.40-8.31 (m, 1H), 7.74 (d, J = 7.4 Hz, 2H), 7.51 (s, 1H), 7.47 (t, J = 7.3 Hz, 1H), 7.41 (t, J = 7.4 Hz, 2H), 7.36-7.31 (m, 1H), 7.30-7.25 (m, 2H), 4.14 (q, J = 7.2 Hz, 2H), 1.43 (t, J = 7.3 Hz, 3H). ¹³C NMR (151 MHz, CDCl₃) δ_C 190.9, 141.0, 136.6, 136.3, 131.1, 128.7, 128.3, 127.4, 123.6, 122.9, 122.7, 115.7, 109.8, 41.8, 15.3. IR (cm⁻¹): 716, 746, 874, 1221, 1384, 1520, 1622, 2933, 2978. GC-MS (EI): 249.1, 234.1, 220.1, 192.0, 172.1, 144.0, 116.1, 105.0, 89.0, 77.1. HRMS m/z (ESI) calcd for C₁₇H₁₅NNaO⁺ [M+Na]⁺: 272.1051, found: 272.1048.

(1,2-dimethyl-1H-indol-3-yl)(phenyl)methanone (2e)

Yellow solid (14.3 mg, 57%), mp = 134.3-135.8 °C. ¹H NMR (400 MHz, CDCl₃) δ_H 7.79 (d, J = 7.3 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 7.34 (dd, J = 7.9, 4.7 Hz, 2H), 7.24 (t, J = 7.6 Hz, 1H), 7.10 (t, J = 7.6 Hz, 1H), 3.76 (s, 3H), 2.62 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ_C 192.9, 144.8, 141.5, 136.6, 131.5, 129.1, 128.3, 127.1, 122.1, 121.4, 121.0, 113.6, 109.2, 29.7, 12.6. IR (cm⁻¹): 728, 754, 1057, 1227, 1382, 1403, 1446, 1471, 1520, 1614, 2938, 3051. GC-MS (EI): 249.2, 248.2, 232.2, 172.1, 143.1, 115.1, 77.1. HRMS m/z (ESI) calcd for C₁₇H₁₅NNaO⁺ [M+Na]⁺: 272.1051, found: 272.1048.

(4-fluorophenyl)(1-methyl-1H-indol-3-yl)methanone (2f)

Yellow solid (15.2 mg, 60%), mp = 138.1-138.6 °C. ¹H NMR (600 MHz, CDCl₃) δ_H 8.33-8.28 (m, 1H), 7.79-7.71 (m, 2H), 7.43 (s, 1H), 7.32-7.25 (m, 3H), 7.08 (t, J = 8.6 Hz, 2H), 3.77 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ_C 189.4, 164.6 (d, J = 251.6 Hz), 137.6, 137.5, 137.1 (d, J = 2.5 Hz), 131.0 (d, J = 8.8 Hz), 127.2, 123.8, 122.8, 122.7, 115.5, 115.3 (d, J = 21.4 Hz), 109.7, 33.6. IR (cm⁻¹): 750, 773, 844, 881, 1079, 1128, 1237, 1372, 1523, 1619, 2927, 3046. GC-MS (EI): 253.1, 183.1, 158.1, 130.1, 112.6, 95.1, 77.1. HRMS m/z (ESI) calcd for C₁₀H₁₂FNNaO⁺ [M+Na]⁺: 272.0851, found: 272.0847.
(4-chlorophenyl)(1-methyl-1H-indol-3-yl)methanone (2g)

White solid (16.9 mg, 63%), mp = 144.0-144.7 °C. \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta_{H} \) 8.49-8.35 (m, 1H), 7.78 (d, \( J = 8.5 \) Hz, 2H), 7.53 (s, 1H), 7.48 (d, \( J = 8.4 \) Hz, 2H), 7.42-7.34 (m, 3H), 3.88 (s, 3H). \( ^13C \) NMR (100 MHz, CDCl\(_3\)) \( \delta_{C} \) 189.4, 139.2, 137.7, 137.6, 137.3, 130.1, 128.6, 127.1, 123.8, 122.9, 122.7, 115.4, 109.7, 33.7. IR (cm\(^{-1}\)): 754, 879, 1086, 1234, 1371, 1389, 1470, 1522, 1589, 1624, 2934, 3053, 3086, 3112. GC-MS (EI): 269.1, 234.1, 158.1, 130.1, 103.0, 77.1. HRMS m/z (ESI) calcd for C\(_{16}\)H\(_{12}\)ClNNaO\(^+\) [M+Na\(^+\)]: 292.0505, found: 292.0504.

(4-ethylphenyl)(1-methyl-1H-indol-3-yl)methanone (2h)

Yellow oil (12.6 mg, 48%). \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta_{H} \) 8.50-8.43 (m, 1H), 7.79 (d, \( J = 8.1 \) Hz, 2H), 7.57 (s, 1H), 7.41-7.30 (m, 5H), 3.86 (s, 3H), 2.77 (q, \( J = 7.6 \) Hz, 2H), 1.32 (t, \( J = 7.6 \) Hz, 3H). \( ^13C \) NMR (151 MHz, CDCl\(_3\)) \( \delta_{C} \) 190.7, 147.8, 138.4, 137.7, 129.0, 127.8, 127.3, 123.6, 122.8, 122.6, 115.7, 109.6, 33.6, 28.9, 15.5. IR (cm\(^{-1}\)): 748, 879, 1234, 1369, 1464, 1524, 1620, 1695, 2874, 2932, 2965, 3051. GC-MS (EI): 263.1, 246.1, 234.1, 158.1, 130.1, 103.1, 77.1. HRMS m/z (ESI) calcd for C\(_{18}\)H\(_{17}\)NNaO\(^+\) [M+Na\(^+\)]: 286.1208, found: 286.1207.

(4-(tert-butyl)phenyl)(1-methyl-1H-indol-3-yl)methanone (2i)

Yellow oil (15.9 mg, 55%). \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta_{H} \) 8.51-8.46 (m, 1H), 7.80 (d, \( J = 8.3 \) Hz, 2H), 7.59 (s, 1H), 7.53 (d, \( J = 8.3 \) Hz, 2H), 7.40-7.35 (m, 3H), 3.86 (s, 3H), 1.41 (s, 9H). \( ^13C \) NMR (151 MHz, CDCl\(_3\)) \( \delta_{C} \) 189.6, 153.5, 137.1, 136.7, 136.4, 127.6, 126.2, 124.2, 122.5, 121.7, 121.6, 114.6, 108.5, 33.9, 32.5, 30.2. IR (cm\(^{-1}\)): 745, 836, 881, 1235, 1464, 1523, 1614, 2962. GC-MS (EI): 291.1, 276.1, 248.1, 234.1, 220.1, 158.0, 145.1, 130.1, 115.1, 103.0, 77.1. HRMS m/z (ESI) calcd for C\(_{20}\)H\(_{21}\)NNaO\(^+\) [M+Na\(^+\)]: 314.1521, found: 314.1517.

(4-methoxyphenyl)(1-methyl-1H-indol-3-yl)methanone (2j)
Yellow solid (20.6 mg, 78%), mp = 135.7-137.3 °C. ¹H NMR (400 MHz, CDCl₃) δ_H 8.42-8.38 (m, 1H), 7.87 (d, J = 8.8 Hz, 2H), 7.57 (s, 1H), 7.42-7.32 (m, 3H), 7.01 (d, J = 8.8 Hz, 2H), 3.91 (s, 3H), 3.87 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ_C 189.8, 162.2, 137.5, 137.1, 133.5, 130.9, 127.3, 123.5, 122.7, 122.5, 115.7, 113.5, 109.6, 55.5, 33.6. IR (cm⁻¹): 754, 832, 1029, 1176, 1248, 1487, 1512, 1605, 1721, 2837, 2957. GC-MS (EI): 265.1, 250.1, 234.1, 222.1, 158.0 130.1, 118.6, 103.1, 77.1. HRMS m/z (ESI) calcd for C₁₇H₁₅NNaO₂⁺ [M+Na⁺]: 288.1000, found: 288.1002.

(3-fluorophenyl)(1-methyl-1H-indol-3-yl)methanone (2k)

Yellow solid (17.0 mg, 67%), mp = 121.2-122.0 °C. ¹H NMR (400 MHz, CDCl₃) δ_H 8.50-8.40 (m, 1H), 7.61 (d, J = 7.7 Hz, 1H), 7.56-7.44 (m, 3H), 7.42-7.35 (m, 3H), 7.29-7.23 (m, 1H), 3.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ_C 189.1 (d, J = 1.5 Hz), 162.5 (d, J = 247.4 Hz), 143.0 (d, J = 6.0 Hz), 138.0, 137.6, 130.0 (d, J = 7.9 Hz), 127.1, 124.4 (d, J = 2.4 Hz), 123.9, 122.9, 122.7, 118.0 (d, J = 21.5 Hz), 115.6 (d, J = 22.2 Hz), 115.2, 109.8, 33.7. IR (cm⁻¹): 748, 769, 829, 1208, 1245, 1371, 1525, 1578, 1623, 2936, 3066. GC-MS (EI): 253.1, 158.1, 130.1, 103.1, 77.1. HRMS m/z (ESI) calcd for C₁₆H₁₂FNNaO⁺ [M+Na⁺]: 276.0801, found: 276.0798.

(1-methyl-1H-indol-3-yl)(m-tolyl)methanone (2l)

Yellow oil (14.1 mg, 57%). ¹H NMR (600 MHz, CDCl₃) δ_H 8.49-8.43 (m, 1H), 7.65 (s, 1H), 7.64-7.59 (m, 1H), 7.54 (s, 1H), 7.41-7.35 (m, 5H), 3.86 (s, 3H), 2.46 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ_C 190.0, 139.9, 137.1, 136.9, 136.5, 130.8, 128.1, 127.0, 126.2, 124.8, 122.6, 121.7, 121.6, 114.6, 108.6, 32.5, 20.4. IR (cm⁻¹): 747, 1243, 1368, 1464, 1522, 1621, 2917. GC-MS (EI): 249.1, 234.1, 158.0, 130.1, 103.1, 91.1, 77.1. HRMS m/z (ESI) calcd for C₁₇H₁₅NNaO⁺ [M+Na⁺]: 272.1051, found: 272.1041.

(5-fluoro-1-methyl-1H-indol-3-yl)(phenyl)methanone (2m)

Yellow oil (15.1 mg, 60%). ¹H NMR (400 MHz, CDCl₃) δ_H 8.13 (dd, J = 9.7, 2.5 Hz, 1H), 7.80 (d, J = 7.0 Hz, 2H), 7.60-7.54 (m, 2H), 7.49 (t, J = 7.4 Hz, 2H), 7.28 (dd, J = 9.0, 4.1 Hz 1H), 7.08 (td, J = 9.0, 2.5 Hz 1H), 3.83 (s, 3H). ¹³C
NMR (151 MHz, CDCl$_3$) $\delta$ C 189.5, 158.8 (d, $J = 237.9$ Hz), 139.5, 137.9, 133.0, 131.2, 127.5, 127.3, 126.8 (d, $J = 11.1$ Hz), 114.3 (d, $J = 4.4$ Hz), 110.9 (d, $J = 26.5$ Hz), 109.5 (d, $J = 9.8$ Hz), 106.9 (d, $J = 25.0$ Hz), 32.8. IR (cm$^{-1}$): 717, 842, 1111, 1197, 1368, 1479, 1524, 1615, 1681, 2926, 3060, 3112. GC-MS (EI): 253.1, 224.1, 176.0, 148.1, 101.0, 77.1. HRMS m/z (ESI) calcd for C$_{16}$H$_{12}$NFNaO$^+$ [M+Na]$^+$: 276.0801, found: 276.0793.

(1,5-dimethyl-1H-indol-3-yl)(phenyl)methanone (2n)

Yellow oil (14.0 mg, 56%). $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ H 8.27 (s, 1H), 7.79 (d, $J = 7.5$ Hz, 2H), 7.53 (t, $J = 7.3$ Hz, 1H), 7.48-7.44 (m, 3H), 7.25 (d, $J = 8.1$ Hz, 1H), 7.17 (d, $J = 8.3$ Hz, 1H), 3.80 (s, 3H), 2.51 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ C 190.9, 141.1, 138.1, 136.0, 132.5, 131.0, 128.7, 128.3, 127.4, 125.2, 122.5, 115.1, 109.3, 33.6, 21.6. IR (cm$^{-1}$): 718, 837, 1237, 1364, 1524, 1616, 2917, 3056. GC-MS (EI): 249.2, 234.1, 220.2, 204.1, 190.1, 172.2, 143.2, 115.1, 77.2. HRMS m/z (ESI) calcd for C$_{17}$H$_{15}$NNaO$^+$ [M+Na]$^+$: 272.1051, found: 272.1053.

(6-chloro-1-methyl-1H-indol-3-yl)(phenyl)methanone (2o)

Yellow solid (13.8 mg, 51%), mp = 126.6-127.5 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ H 8.36 (d, $J = 8.5$ Hz, 1H), 7.82 (d, $J = 7.0$ Hz, 2H), 7.58 (t, $J = 7.3$ Hz, 1H), 7.54-7.48 (m, 3H), 7.38 (d, $J = 1.6$ Hz, 1H), 7.32 (dd, $J = 8.5$, 1.7 Hz, 1H), 3.83 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ C 190.7, 140.5, 138.2, 138.0, 131.3, 129.6, 128.7, 128.4, 125.7, 123.7, 123.3, 115.7, 109.8, 33.7. IR (cm$^{-1}$): 719, 814, 886, 1072, 1227, 1365, 1464, 1525, 1624, 2933, 3054. GC-MS (EI): 269.1, 252.1, 192.1, 164.1, 128.1, 102.1, 77.1. HRMS m/z (ESI) calcd for C$_{16}$H$_{12}$NClNaO$^+$ [M+Na]$^+$: 292.0505, found: 292.0494.

(1-methyl-1H-indol-3-yl)(thiophen-3-yl)methanone (2p)

Yellow oil (13.3 mg, 55%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ H 8.48-8.42 (m, 1H), 7.91 (d, $J = 2.7$ Hz, 1H), 7.69 (s, 1H), 7.61 (d, $J = 5.0$ Hz, 1H), 7.42-7.33 (m, 4H), 3.87 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ C 184.2, 143.7, 137.5, 136.8, 129.7, 128.2, 127.1, 126.0, 123.7, 122.7, 122.6, 116.3, 109.7, 33.6. IR (cm$^{-1}$): 747, 768, 841, 1238, 1465, 1524, 1609, 2931, 3105. GC-MS (EI): 241.0, 213.1, 198.0, 171.0, 158.0, 130.1, 103.1, 77.1. HRMS m/z (ESI) calcd for C$_{14}$H$_{11}$NNaOS$^+$ [M+Na]$^+$: 292.0459, found: 292.0451.

(1-methyl-1H-indol-3-yl)(thiophen-2-yl)methanone (2q)
Brown solid (15.1 mg, 63%), mp = 136.1-137.3 °C. \textbf{1H NMR (400 MHz, CDCl}_3\text{)} \delta_{H} 8.49-8.42 (m, 1H), 7.82 (s, 1H), 7.76 (dd, \textit{J} = 3.6, 0.8 Hz, 1H), 7.63 (d, \textit{J} = 4.9 Hz, 1H), 7.40-7.34 (m, 3H), 7.18 (dd, \textit{J} = 4.8, 3.8 Hz, 1H), 3.89 (s, 3H).

\textbf{13C NMR (151 MHz, CDCl}_3\text{)} \delta_{C} 181.4, 145.4, 137.4, 136.1, 131.3, 131.0, 127.6, 127.3, 123.7, 122.6, 115.4, 109.7, 33.6.

\textbf{IR (cm}^{-1}\text{): 734, 747, 811, 1237, 1370, 1464, 1521, 1572, 1586, 2931, 3046, 3077, 3109. \textbf{GC-MS (EI): 241.1, 224.1, 213.1, 158.1, 130.1, 103.1, 77.1. \textbf{HRMS m/z (ESI) calcd for C}_{14}H_{11}NNaOS [M+Na]^{+}: 264.0459, found:264.0459 .}

\textbf{2,2-dimethyl-1-(1-methyl-1H-indol-3-yl)propan-1-one (2r)}

Yellow solid (6.2 mg, 29%), mp = 126.0-127.2 °C. \textbf{1H NMR (600 MHz, CDCl}_3\text{)} \delta_{H} 8.52 (d, \textit{J} = 6.9 Hz, 1H), 7.79 (s, 1H), 7.34-7.28 (m, 3H), 3.85 (s, 3H), 1.42 (s, 9H).

\textbf{13C NMR (151 MHz, CDCl}_3\text{)} \delta_{C} 202.1, 136.5, 134.4, 128.3, 123.4, 123.2, 122.5, 112.7, 109.2, 44.1, 33.5, 29.0. \textbf{IR (cm}^{-1}\text{): 749, 899, 1083, 1360, 1469, 1523, 1624, 2967. \textbf{GC-MS (EI): 215.1, 172.1, 158.1, 130.1, 103.1, 77.1. \textbf{HRMS m/z (ESI) calcd for C}_{14}H_{17}NNaO [M+Na]^{+}: 238.1208, found:238.1207 .}

\textbf{Benzofuran-3-yl(phenyl)methanone (4a)}

Pale yellow soild (5.8 mg, 26%), mp = 58.2-60.9 °C. \textbf{1H NMR (400 MHz, CDCl}_3\text{)} \delta_{H} 8.27-8.22 (m, 1H), 8.09 (s, 1H), 7.89 (d, \textit{J} = 7.0 Hz, 2H), 7.64-7.50 (m, 4H), 7.44-7.38 (m, 2H). \textbf{13C NMR (100 MHz, CDCl}_3\text{)} \delta_{C} 190.3, 155.6, 152.3, 139.3, 132.5, 128.8, 128.7, 125.9, 125.2, 124.6, 122.9, 121.3, 111.6. \textbf{IR (cm}^{-1}\text{): 1646, 1547, 1479, 1449, 1286, 1132, 886, 749, 715, 698. \textbf{GC-MS (EI): 77.1, 89.1, 145.1, 165.1, 194.1, 222.1. \textbf{HRMS m/z (ESI) calcd for C}_{15}H_{11}O_{2} [M+H]^{+}: 223.0759, found:223.0760.}

\textbf{Phenyl(2-phenylbenzofuran-3-yl)methanone (4b)}

Pale yellow soild (16.3 mg, 55%), mp = 90.5-93.9 °C. \textbf{1H NMR (400 MHz, CDCl}_3\text{)} \delta_{H} 7.88 (d, \textit{J} = 7.1 Hz, 2H), 7.76-7.67 (m, 2H), 7.61 (t, \textit{J} = 9.0 Hz, 2H), 7.52 (t, \textit{J} = 7.4 Hz, 1H), 7.43-7.28 (m, 7H). \textbf{13C NMR (100 MHz, CDCl}_3\text{)} \delta_{C} 192.4, 157.7, 153.9, 137.8, 133.2, 129.9, 129.7, 129.5, 128.5, 128.4, 125.4, 123.9, 121.5, 116.2, 111.3. \textbf{IR (cm}^{-1}\text{): 1658,
1454, 1235, 884, 747, 718, 693. GC-MS (EI): 51.2, 77.2, 105.1, 165.1, 221.1, 298.1. HRMS m/z (ESI) calcd for $\text{C}_{13}\text{H}_{12}\text{O}^+ [\text{M+H}]^+$: 299.1072, found: 299.1073.

**(2-methylbenzofuran-3-yl)(phenyl)methanone (4c)**

![2-methylbenzofuran-3-yl](image)

Yellow oil (2.3 mg, 10%). $^1\text{H NMR}$ (400 MHz, CDCl$_3$) $\delta$ 7.86-7.79 (m, 2H), 7.62-7.57 (m, 1H), 7.52-7.45 (m, 3H), 7.41 (d, $J = 7.9$ Hz, 1H), 7.30-7.26 (m, 1H), 7.19 (td, $J = 7.8$, 1.0 Hz, 1H), 2.54 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl$_3$) $\delta$C 192.0, 162.0, 153.7, 139.4, 132.7, 129.1, 128.5, 124.1, 123.6, 121.4, 117.0, 110.9, 14.7. IR (cm$^{-1}$): 3462, 1740, 1648, 1578, 1454, 1386, 1242, 1179, 899, 749, 699. GC-MS (EI): 77.1, 105.1, 159.1, 236.1. HRMS m/z (ESI) calcd for $\text{C}_{16}\text{H}_{13}\text{O}^+ [\text{M+H}]^+$: 237.0916, found: 237.0915.

**(2-(2-chlorophenyl)benzofuran-3-yl)(phenyl)methanone (4d)**

![2-(2-chlorophenyl)](image)

Pale yellow solid (14.9 mg, 45%), mp = 104.9-107.1 °C.$^1\text{H NMR}$ (400 MHz, CDCl$_3$) $\delta$ 7.84 (d, $J = 7.7$ Hz, 1H), 7.72 (d, $J = 7.1$ Hz, 2H), 7.60 (d, $J = 8.2$ Hz, 1H), 7.44-7.29 (m, 5H), 7.26-7.21 (m, 3H), 7.17 (td, $J = 7.5$, 1.3 Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl$_3$) $\delta$C 191.7, 156.9, 154.4, 138.1, 133.8, 132.6, 132.4, 131.1, 129.9, 129.5, 129.4, 128.0, 127.2, 126.6, 125.7, 124.2, 122.1, 118.9, 111.5. IR (cm$^{-1}$): 1647, 1574, 1449, 1378, 1043, 884, 750, 697. GC-MS (EI): 77.1, 105.2, 148.7, 163.1, 220.1, 297.1, 332.1. HRMS m/z (ESI) calcd for $\text{C}_{16}\text{H}_{14}\text{ClO}^+ [\text{M+H}]^+$: 333.0682, found: 333.0686.

**(2-(3-chlorophenyl)benzofuran-3-yl)(phenyl)methanone (4e)**

![2-(3-chlorophenyl)](image)

Pale yellow solid (17.9 mg, 54%), mp = 90.2-91.4 °C.$^1\text{H NMR}$ (400 MHz, CDCl$_3$) $\delta$ 7.84 (d, $J = 7.7$ Hz, 2H), 7.74 (s, 1H), 7.60-7.50 (m, 4H), 7.37 (q, $J = 7.6$ Hz, 3H), 7.30-7.24 (m, 2H), 7.20 (t, $J = 7.9$ Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl$_3$) $\delta$C 192.1, 155.7, 153.8, 137.7, 134.5, 133.5, 131.1, 129.8, 129.7, 129.6, 128.6, 128.2, 128.1, 126.5, 125.8, 124.0, 121.7, 117.1, 111.4. IR (cm$^{-1}$): 3437, 1656, 1450, 1234, 888, 748, 696. GC-MS (EI): 77.2, 105.2, 163.1, 199.1, 200.1, 255.1, 332.1. HRMS m/z (ESI) calcd for $\text{C}_{16}\text{H}_{14}\text{ClO}^+ [\text{M+H}]^+$: 333.0682, found: 333.0687.

**(2-(4-chlorophenyl)benzofuran-3-yl)(phenyl)methanone (4f)**

![2-(4-chlorophenyl)](image)
Pale yellow solid (18.4 mg, 55%), mp = 105.0-110.3 °C. 1H NMR (400 MHz, CDCl3) δH 7.84 (d, J = 7.1 Hz, 2H), 7.6-7.64 (m, 2H), 7.58 (d, J = 8.3 Hz, 1H), 7.53 (t, J = 7.4 Hz, 1H), 7.48 (d, J = 7.9 Hz, 1H), 7.40-7.34 (m, 3H), 7.30-7.23 (m, 3H). 13C NMR (100 MHz, CDCl3) δC 192.2, 156.2, 153.8, 137.7, 135.9, 129.8, 129.5, 128.8, 128.6, 128.3, 127.9, 125.6, 124.0, 121.6, 116.6, 111.3. IR (cm⁻¹): 1656, 1598, 1578, 1488, 1450, 1409, 884, 748, 695. GC-MS (EI): 77.2, 105.1, 139.1, 255.1, 329.1. HRMS m/z (ESI) calcd for C21H14ClO2+ [M+H]+: 333.0682, found: 333.0690.

(4-fluorophenyl)(2-phenylbenzofuran-3-yl)methanone (4g)

Pale yellow solid (15.8 mg, 50%), mp = 132.0-136.8 °C (uncorrected). 1H NMR (400 MHz, CDCl3) δH 7.86 (dd, J = 8.8, 5.5 Hz, 2H), 7.66 (dd, J = 7.6, 1.9 Hz, 2H), 7.58 (t, J = 7.2 Hz, 2H), 7.40-7.35 (m, 1H), 7.34-7.26 (m, 4H), 6.99 (t, J = 8.6 Hz, 2H). 13C NMR (100 MHz, CDCl3) δC 190.7, 165.8 (d, J = 255.3 Hz), 157.7, 153.9, 134.1 (d, J = 2.5 Hz), 132.5 (d, J = 9.5 Hz), 129.9, 129.3, 128.5, 128.4, 128.3, 125.5, 124.0, 121.4, 115.9, 115.6 (d, J = 22.0 Hz), 111.3. IR (cm⁻¹): 3467, 1646, 1598, 1236, 1154, 888, 764, 749, 693, 603. GC-MS (EI): 95.1, 123.1, 165.1, 221.1, 316.1. HRMS m/z (ESI) calcd for C21H14FO2+ [M+H]+: 317.0978, found: 317.0980.

(4-chlorophenyl)(2-phenylbenzofuran-3-yl)methanone (4h)

White solid (18.1 mg, 55%), mp = 115.3-117.9 °C. 1H NMR (400 MHz, CDCl3) δH 7.80 (d, J = 8.5 Hz, 2H), 7.69 (dd, J = 7.8, 1.6 Hz, 2H), 7.61 (t, J = 8.1 Hz, 2H), 7.43-7.29 (m, 7H). 13C NMR (100 MHz, CDCl3) δC 191.0, 157.9, 153.9, 139.6, 136.2, 131.2, 130.0, 129.3, 128.8, 128.6, 128.5, 128.3, 125.5, 124.0, 121.4, 115.8, 111.4. IR (cm⁻¹): 1654, 1588, 1454, 1372, 1239, 1088, 885, 749, 694. GC-MS (EI): 111.1, 139.1, 165.1, 221.1, 332.1. HRMS m/z (ESI) calcd for C21H14ClO2+ [M+H]+: 333.0682, found: 333.0675.

(2-phenylbenzofuran-3-yl)(p-tolyl)methanone (4i)

Pale yellow solid (16.5 mg, 55%), mp = 98.9-102.1 °C. 1H NMR (400 MHz, CDCl3) δH 7.77 (d, J = 8.2 Hz, 2H), 7.73-7.67 (m, 2H), 7.57 (d, J = 8.2 Hz, 1H), 7.49 (d, J = 8.3 Hz, 1H), 7.37-7.29 (m, 4H), 7.24 (t, J = 8.0 Hz, 1H), 7.14 (d, J = 8.0 Hz, 2H), 2.35 (s, 3H). 13C NMR (100 MHz, CDCl3) δC 192.1, 156.9, 153.8, 144.3, 135.2, 130.1, 129.7, 129.5, 129.2, 128.6, 128.5, 128.2, 125.3, 123.8, 121.4, 116.4, 111.3, 21.8. IR (cm⁻¹): 1655, 1605, 1454, 887, 747, 693. GC-MS (EI): 91.2, 119.2, 221.1, 297.1, 312.2. HRMS m/z (ESI) calcd for C22H17O2+ [M+H]+: 313.1229, found: 313.1231.

(4-ethylphenyl)(2-phenylbenzofuran-3-yl)methanone (4j)
Pale yellow oil (16.0 mg, 49%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 7.82 (d, $J$ = 8.2 Hz, 2H), 7.74 (dd, $J$ = 6.7, 3.0 Hz, 2H), 7.61 (d, $J$ = 8.2 Hz, 1H), 7.57 (d, $J$ = 7.6 Hz, 1H), 7.41-7.36 (m, 1H), 7.36-7.31 (m, 3H), 7.31-7.26 (m, 1H), 7.20 (d, $J$ = 8.2 Hz, 2H), 2.69 (q, $J$ = 7.6 Hz, 2H), 1.24 (t, $J$ = 7.6 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$C 192.1, 157.0, 153.8, 150.4, 135.4, 130.2, 129.6, 129.5, 128.6, 128.4, 128.2, 128.0, 125.3, 123.7, 121.5, 116.4, 111.2, 29.0, 15.2.

IR (cm$^{-1}$): 3467, 1655, 1605, 1454, 1257, 1237, 888, 748, 707, 693.

GC-MS (EI): 133.2, 165.1, 222.1, 297.1, 326.2.

HRMS m/z (ESI) calcd for C$_{23}$H$_{19}$O$_2$+ [M+H]$^+$: 327.1385, found: 327.1389.

(4-methoxyphenyl)(2-phenylbenzofuran-3-yl)methanone (4k)

Pale yellow oil (16.7 mg, 51%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 7.87 (d, $J$ = 8.9 Hz, 2H), 7.75-7.70 (m, 2H), 7.57 (d, $J$ = 7.4 Hz, 1H), 7.49 (d, $J$ = 6.8 Hz, 1H), 7.37-7.30 (m, 4H), 7.26-7.22 (m, 1H), 6.82 (d, $J$ = 8.9 Hz, 2H), 3.81 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$C 190.9, 163.9, 156.3, 153.8, 132.4, 130.6, 129.6, 128.7, 128.5, 128.1, 125.3, 123.7, 121.3, 116.4, 113.8, 111.2, 55.5.

IR (cm$^{-1}$): 3462, 1650, 1599, 1258, 1200, 1110, 1070, 816, 748, 693.

GC-MS (EI): 77.2, 135.2, 222.1, 328.2.

HRMS m/z (ESI) calcd for C$_{22}$H$_{17}$O$_3$+ [M+H]$^+$: 329.1178, found: 329.1172.

(3-fluorophenyl)(2-phenylbenzofuran-3-yl)methanone (4l)

Pale yellow oil (16.7 mg, 53%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 7.69-7.50 (m, 6H), 7.41-7.36 (m, 1H), 7.34-7.23 (m, 5H), 7.16 (ddd, $J$ = 8.2, 2.5, 0.8 Hz, 1H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$C 191.0, 162.7, 158.4, 153.9, 140.0 (d, $J$ = 6.5 Hz), 130.0 (d, $J$ = 9 Hz), 129.9, 129.3, 128.6, 128.5, 128.2, 125.7 (d, $J$ = 2.5 Hz), 125.6, 124.1, 121.5, 120.0 (d, $J$ = 21.7 Hz), 116.3 (d, $J$ = 22.7 Hz), 115.8, 111.3.

IR (cm$^{-1}$): 3444, 1649, 1587, 1442, 1376, 1258, 1200, 1110, 1070, 816, 748, 693.

GC-MS (EI): 95.2, 123.1, 165.1, 222.1, 316.2.

HRMS m/z (ESI) calcd for C$_{21}$H$_{14}$FO$_2$+ [M+H]$^+$: 317.0978, found: 317.0981.

(2-phenylbenzofuran-3-yl)(m-tolyl)methanone (4m)

Pale yellow oil (15.2 mg, 49%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$H 7.71-7.65 (m, 3H), 7.62 (d, $J$ = 7.8 Hz, 1H), 7.57 (t, $J$ =
8.1 Hz, 2H), 7.38-7.33 (m, 1H), 7.32-7.24 (m, 5H), 7.20 (t, \(J = 7.6\) Hz, 1H), 2.27 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta_c\) 192.6, 157.6, 153.8, 138.3, 134.0, 130.3, 129.7, 129.6, 128.5, 128.4, 128.3, 127.2, 125.3, 123.8, 121.5, 116.3, 111.2, 21.2. IR (cm\(^{-1}\))): 3061, 1658, 1584, 1560, 1454, 1374, 1257, 748, 693. GC-MS (EI): 65.2, 91.2, 119.2, 165.1, 221.1, 312.2. HRMS m/z (ESI) calcd for C\(_{22}\)H\(_{17}\)O\(_2\)\(^+\) [M+H\(^+\)]: 313.1229, found: 313.1227.

(2-phenylbenzofuran-3-yl)(thiophen-3-yl)methanone (4n)

Pale yellow oil (14.5 mg, 48%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta_h\) 7.83 (dd, \(J = 2.9, 1.2\) Hz, 1H), 7.74-7.69 (m, 2H), 7.65 (d, \(J = 7.8\) Hz, 1H), 7.57 (d, \(J = 8.2\) Hz, 1H), 7.52 (dd, \(J = 5.1, 1.1\) Hz, 1H), 7.38-7.24 (m, 5H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta_c\) 185.6, 156.9, 153.8, 142.3, 135.0, 129.8, 129.5, 128.6, 128.4, 128.3, 127.8, 126.3, 125.5, 123.9, 121.4, 117.1, 111.3. IR (cm\(^{-1}\))): 3104, 1641, 1561, 1510, 1454, 1413, 1242, 1070, 848, 748, 694. GC-MS (EI): 39.2, 83.1, 111.1, 165.1, 221.1, 275.1, 304.2. HRMS m/z (ESI) calcd for C\(_{19}\)H\(_{13}\)O\(_2\)S\(^+\) [M+H\(^+\)]: 305.0636, found: 305.0633.

(2-phenylbenzofuran-3-yl)(thiophen-2-yl)methanone (4o)

Pale yellow solid (16.4 mg, 54%), mp = 79.4-82.9 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta_h\) 7.76 (dd, \(J = 6.5, 3.2\) Hz, 2H), 7.66-7.61 (m, 2H), 7.53 (d, \(J = 8.2\) Hz, 1H), 7.45 (dd, \(J = 3.8, 1.0\) Hz, 1H), 7.39-7.32 (m, 4H), 7.28 (t, \(J = 7.9\) Hz, 1H), 6.93 (dd, \(J = 4.8, 3.9\) Hz, 1H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta_c\) 184.0, 156.4, 153.8, 144.4, 135.2, 134.8, 129.8, 129.5, 128.6, 128.3, 128.2, 128.1, 125.5, 123.9, 121.4, 117.1, 111.3. IR (cm\(^{-1}\))): 1630, 1454, 1411, 1244, 815, 747, 728, 690. GC-MS (EI): 39.2, 83.1, 111.1, 165.1, 221.1, 271.1, 304.1. HRMS m/z (ESI) calcd for C\(_{19}\)H\(_{13}\)O\(_2\)S\(^+\) [M+H\(^+\)]: 305.0636, found: 305.0634.

2,2-dimethyl-1-(2-phenylbenzofuran-3-yl)propan-1-one (4p)

White solid (6.2 mg, 22%), mp = 93.9-96.8 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta_h\) 7.68 (dd, \(J = 8.2, 1.5\) Hz, 2H), 7.53 (d, \(J = 8.2\) Hz, 1H), 7.46-7.39 (m, 4H), 7.35-7.30 (m, 1H), 7.28-7.23 (m, 1H), 1.17 (s, 9H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta_c\) 211.2, 153.7, 151.2, 130.5, 129.4, 128.8, 128.6, 127.1, 125.1, 123.4, 120.5, 116.9, 111.3, 46.5, 26.9. IR (cm\(^{-1}\))): 3437, 2969, 1687, 1589, 1455, 1259, 1065, 922, 746, 696. GC-MS (EI): 28.2, 165.1, 221.1, 278.1. HRMS m/z (ESI) calcd for C\(_{19}\)H\(_{19}\)O\(_2\)\(^+\) [M+H\(^+\)]: 279.1385, found: 279.1378.

Phenyl(2-phenylnaphtho[2,1-b]furan-1-yl)methanone (4q)
Yellow oil (11.1 mg, 32%). \[^{1}H\text{ NMR}\ (400 \text{ MHz, } \text{CDCl}_3) \delta_H 8.04 (d, J = 7.3 \text{ Hz, } 2\text{H}), 7.93 (d, J = 8.0 \text{ Hz, } 1\text{H}), 7.84-7.79 (m, 2\text{H}), 7.75 (d, J = 9.0 \text{ Hz, } 1\text{H}), 7.70 (d, J = 7.2 \text{ Hz, } 2\text{H}), 7.53 (t, J = 7.4 \text{ Hz, } 1\text{H}), 7.44-7.41 (m, 1\text{H}), 7.41-7.35 (m, 3\text{H}), 7.34-7.26 (m, 3\text{H}). \[^{13}C\text{ NMR} (100 \text{ MHz, } \text{CDCl}_3) \delta_C 195.2, 152.9, 151.7, 137.3, 134.2, 131.0, 130.1, 129.6, 129.1, 129.0, 128.9, 128.7, 127.3, 127.0, 126.9, 126.7, 124.8, 123.9, 122.6, 117.5, 112.1. \text{ IR (cm}^{-1}\text{): 2968, 1724, 1594, 1491, 1362, 1284, 1198, 950, 750. \text{ HRMS } m/z (\text{ESI}) \text{ calcd for C}_{25}H_{16}NaO}_2^+ [\text{M+Na}]^+ : 371.1048, \text{ found: 371.1050.}

\text{Phenyl(2-phenylbenzo[b]thiophen-3-yl)methanone (6a)}

Pale yellow solid (15.4 mg, 49%), mp = 110.8-113.3\text{oC}. \[^{1}H\text{ NMR}\ (400 \text{ MHz, } \text{CDCl}_3) \delta_H 7.95-7.89 (m, 1\text{H}), 7.83-7.74 (m, 3\text{H}), 7.48-7.38 (m, 5\text{H}), 7.32-7.21 (m, 5\text{H}). \[^{13}C\text{ NMR} (100 \text{ MHz, } \text{CDCl}_3) \delta_C 194.3, 146.5, 139.7, 138.9, 137.5, 133.3, 131.5, 129.9, 129.4, 128.8, 128.6, 128.3, 125.2, 125.1, 123.7, 122.1. \text{ IR (cm}^{-1}\text{): 3455, 1650, 1597, 1494, 1348, 1232, 753, 727, 694. \text{ GC-MS (EI): 77.1, 105.1, 165.1, 208.1, 237.1, 314.1. HRMS } m/z (\text{ESI}) \text{ calcd for C}_{21}H_{15}OS^+ [\text{M+H}]^+ : 315.0844, \text{ found: 315.0840.}

\text{Benzo[b]thiophen-3-yl(phenyl)methanone (6b)}

Yellow oil (12.4 mg, 52%). \[^{1}H\text{ NMR}\ (400 \text{ MHz, } \text{CDCl}_3) \delta_H 8.57 (d, J = 7.9 \text{ Hz, } 1\text{H}), 8.00 (s, 1\text{H}), 7.91 (d, J = 8.0 \text{ Hz, } 1\text{H}), 7.89-7.85 (m, 2\text{H}), 7.63-7.57 (m, 1\text{H}), 7.54-7.43 (m, 4\text{H}). \[^{13}C\text{ NMR} (100 \text{ MHz, } \text{CDCl}_3) \delta_C 190.9, 140.1, 139.3, 138.3, 137.5, 134.8, 132.4, 129.5, 128.5, 125.7, 125.6, 125.2, 122.4. \text{ IR (cm}^{-1}\text{): 1644, 1597, 1494, 1459, 1364, 1237, 1050, 841, 765, 713, 665. \text{ GC-MS (EI): 77.1, 89.1, 105.1, 133.0, 161.1, 238.1. HRMS } m/z (\text{ESI}) \text{ calcd for C}_{15}H_{11}OS^+ [\text{M+H}]^+: 239.0531, \text{ found: 239.0532.}

\text{(2-methylbenzo[b]thiophen-3-yl)(phenyl)methanone (6c)}

Pale yellow oil (12.6 mg, 50%). \[^{1}H\text{ NMR}\ (400 \text{ MHz, } \text{CDCl}_3) \delta_H 8.57 (d, J = 8.3, 1.3 \text{ Hz, } 2\text{H}), 8.00 (s, 1\text{H}), 7.91 (d, J = 8.0 \text{ Hz, } 1\text{H}), 7.89-7.85 (m, 2\text{H}), 7.63-7.57 (m, 1\text{H}), 7.54-7.43 (m, 4\text{H}). \[^{13}C\text{ NMR} (100 \text{ MHz, } \text{CDCl}_3) \delta_C 193.5, 145.9, 139.1, 138.7, 138.0, 133.3, 132.3, 129.7, 128.7, 124.8, 124.3, 123.3, 121.8, 15.8. \text{ IR (cm}^{-1}\text{): 3444, 1651, 1433,
1353, 1274, 1231, 754, 726, 694. GC-MS (EI): 77.1, 105.1, 147.1, 175.1, 235.1, 251.1. HRMS m/z (ESI) calcd for C$_{16}$H$_{13}$OS$^+$ [M+H]$^+$: 253.0687, found: 253.0686.

(2-phenylbenzo[b]thiophen-3-yl)(p-tolyl)methanone (6d)

![Chemical structure](image)

Pale yellow oil (14.4 mg, 44%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta_{H}$ 7.88 (dd, $J = 6.6$, 2.0 Hz, 1H), 7.72-7.65 (m, 3H), 7.47-7.42 (m, 2H), 7.26-7.21 (m, 3H), 7.07 (d, $J = 8.0$ Hz, 2H), 2.31 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta_{C}$ 194.1, 145.5, 144.4, 139.8, 138.9, 134.9, 133.3, 131.8, 130.1, 129.2, 129.1, 128.7, 128.6, 125.1, 125.0, 123.6, 122.0, 21.7. IR (cm$^{-1}$): 1651, 1604, 1457, 1433, 1346, 1277, 1235, 1176, 755, 728, 695. GC-MS (EI): 91.1, 119.1, 165.1, 179.1, 208.1, 237.1, 328.1.

HRMS m/z (ESI) calcd for C$_{22}$H$_{17}$OS$^+$ [M+H]$^+$: 329.1000, found: 329.1001.

(4-chlorophenyl)(2-phenylbenzo[b]thiophen-3-yl)methanone (6e)

![Chemical structure](image)

Pale yellow oil (17.7 mg, 51%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta_{H}$ 7.92-7.86 (m, 1H), 7.80-7.73 (m, 1H), 7.69 (d, $J = 8.4$ Hz, 2H), 7.42-7.36 (m, 4H), 7.26 -7.19 (m, 5H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta_{C}$ 192.9, 146.9, 139.6, 139.5, 139.0, 135.8, 133.1, 131.3, 131.0, 129.4, 129.1, 128.7, 128.6, 125.4, 125.3, 123.6, 122.1. IR (cm$^{-1}$): 3467, 1651, 1586, 1347, 1232, 1089, 1013, 845, 755, 734, 695. GC-MS (EI): 75.1, 111.1, 139.1, 165.1, 208.1, 237.1, 348.1. HRMS m/z (ESI) calcd for C$_{21}$H$_{14}$ClOS$^+$ [M+H]$^+$: 349.0454, found: 349.0448.

(3-fluorophenyl)(2-phenylbenzo[b]thiophen-3-yl)methanone (6f)

![Chemical structure](image)

Pale yellow oil (13.3 mg, 40%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta_{H}$ 7.89 (dd, $J = 6.3$, 2.7 Hz, 1H), 7.80 (dd, $J = 6.4$, 2.8 Hz, 1H), 7.50-7.43 (m, 2H), 7.43-7.36 (m, 4H), 7.25-7.16 (m, 4H), 7.08 (td, $J = 8.2$, 1.8 Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta_{C}$ 192.8, 162.5 (d, $J = 248.2$ Hz), 147.5, 139.6 (d, $J = 6.5$ Hz), 139.5, 139.0, 133.1, 130.9, 129.9 (d, $J = 7.5$ Hz), 129.4, 129.0, 128.7, 125.8 (d, $J = 2.9$ Hz), 125.4, 125.3, 123.6, 122.1, 120.1 (d, $J = 21.7$ Hz), 116.3 (d, $J = 22.4$ Hz). IR (cm$^{-1}$): 3449, 1655, 1587, 1442, 1349, 1250, 755, 724, 696. GC-MS (EI): 95.1, 165.1, 208.1, 237.1, 332.1. HRMS m/z (ESI) calcd for C$_{21}$H$_{14}$FOS$^+$ [M+H]$^+$: 333.0749, found: 333.0757.

(2-phenylbenzo[b]thiophen-3-yl)(thiophen-3-yl)methanone (6g)

Pale yellow oil (13.3 mg, 40%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta_{H}$ 7.89 (dd, $J = 6.3$, 2.7 Hz, 1H), 7.80 (dd, $J = 6.4$, 2.8 Hz, 1H), 7.50-7.43 (m, 2H), 7.43-7.36 (m, 4H), 7.25-7.16 (m, 4H), 7.08 (td, $J = 8.2$, 1.8 Hz, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta_{C}$ 192.8, 162.5 (d, $J = 248.2$ Hz), 147.5, 139.6 (d, $J = 6.5$ Hz), 139.5, 139.0, 133.1, 130.9, 129.9 (d, $J = 7.5$ Hz), 129.4, 129.0, 128.7, 125.8 (d, $J = 2.9$ Hz), 125.4, 125.3, 123.6, 122.1, 120.1 (d, $J = 21.7$ Hz), 116.3 (d, $J = 22.4$ Hz). IR (cm$^{-1}$): 3449, 1655, 1587, 1442, 1349, 1250, 755, 724, 696. GC-MS (EI): 95.1, 165.1, 208.1, 237.1, 332.1. HRMS m/z (ESI) calcd for C$_{21}$H$_{14}$FOS$^+$ [M+H]$^+$: 333.0749, found: 333.0757.
Pale yellow oil (10.1 mg, 32%). ¹H NMR (400 MHz, CDCl₃) δH 7.90-7.85 (m, 1H), 7.83-7.78 (m, 1H), 7.70 (d, J = 1.9 Hz, 1H), 7.4-7.43 (m, 3H), 7.42-7.36 (m, 2H), 7.30-7.24 (m, 3H), 7.14 (dd, J = 5.0, 3.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δC 187.6, 146.0, 142.5, 139.5, 138.9, 135.5, 133.4, 132.4, 129.3, 128.9, 128.7, 127.6, 126.1, 125.2, 125.1, 123.6, 122.0. IR (cm⁻¹): 3457, 1646, 1509, 1238, 756, 723, 695. GC-MS (EI): 39.2, 83.1, 111.1, 165.1, 208.1, 237.1, 291.1, 303.1, 320.1. HRMS m/z (ESI) calcd for C₁₉H₁₃OS⁺ [M+H]+: 321.0408, found: 321.0411.

2,2-dimethyl-1-(2-phenylbenzo[b]thiophen-3-yl)propan-1-one (6h)

Pale yellow oil (7.3 mg, 25%). ¹H NMR (400 MHz, CDCl₃) δH 7.76 (d, J = 6.8 Hz, 1H), 7.6-7.49 (m, 3H), 7.37-7.25 (m, 5H), 0.92 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δC 212.6, 138.8, 138.3, 138.2, 133.5, 132.4, 128.0, 127.9, 127.8, 123.8, 123.7, 122.0, 121.1, 45.0, 26.0. IR (cm⁻¹): 1681, 1261, 1099, 755, 696. GC-MS (EI): 165.1, 208.1, 237.1, 294.1. HRMS m/z (ESI) calcd for C₁₉H₁₉OS⁺ [M+H]+: 295.1157, found: 295.1156.
7. $^1$H NMR and $^{13}$C NMR spectra for new substrates 1a
If
1h
1k
S62
3k
3q
5a
8. $^1$H NMR and $^{13}$C NMR spectra for products 2a
2b

S80
2r
4b