Supporting information

Supported palladium catalyzed aminocarbonylation of aryl iodides employing bench-stable CO and NH\textsubscript{3} surrogates

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A. General method and materials

High quality reagents were purchased from Sigma Aldrich, Tech Chem Solutions (TCI, India), Loba Chemie, alpha aesar and Sd Fine-chem Ltd. Thin layer chromatography was performed using pre-coated silica gel plates 60F254 (Merck) in UV light detector. ESI-MS spectra were determined using a Waters micro mass Q-TOF Ultima Spectrometer. Mass spectra were recorded on electro spray ionization (ESI) quadrupole time of flight (Q-TOF) mass spectrometer. Melting points were recorded using LAB INDIA MR-VIS+. 1H and 13C NMR spectra were recorded using a Bruker Avance 600 spectrometer operating at 600 MHz (1H) and 150 MHz (13C) and Bruker Avance 300 spectrometer operating at 300 MHz (1H) and 75 MHz (13C). Spectra were recorded at 25 °C in DMSO-d6 [residual DMSO (δH 2.50 and 3.42 ppm) and DMSO (δC 39.52 ppm)] with TMS as internal standard. Chemical shifts were recorded in δ (ppm) relative to the TMS and NMR solvent signal. Coupling constants (J) are given in Hz and multiplicities of signals are reported as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; brs, broad singlet.

B. Synthesis of (1H-imidazol-1-yl) (p-tolyl)methanone (5)

The benzyol imidazole was prepared by following the literature. 1 In a 25 mL round bottom flask, imidazole (2 mmol) was dissolved in anhydrous CH2Cl2 (2 mL). Further, benzyol chloride (1mmol) was in added in solution of imidazole slowly and dropwise, resulting in precipititation of imidazolium chloride. The resultant mixture was allowed to stir for 2-3 h at room temperature. The reaction mixture was filtered, washed with cold water and extracted using dichloromethane as solvent. The organic layer was dried over sodium sulphate and solvent was evaporated under reduced pressure yielding crude benzyol imidazole (5) as viscous oil. The crude was crystallized with the help of hexanes. The formation of the product was confirmed with the help of ESI-MS [M+H]+ for C11H11N2O+ 187.1066 and found 187.0864.
C. Typical Experimental procedure

1. 4-methylbenzamide

In a double vial system (inner vial is of 2mL and another which is outer is of 5mL), 4-methyl iodobenzene (0.229 mmol, 50 mg), ammonium carbamate (0.917 mmol, 69.7mg), Pd@PS (0.0069 mmol, 70 mg), TEA (0.573 mmol, 79.7 µl), imidazole (0.172 mmol, 13 mg) and DMF (1.5 mL) were added in inner vial (2 mL) while the outer vial was charged with oxalic acid (1.37 mmol, 123.8 mg) and DMF (0.3 mL). After completion of the addition, the inner vial containing contents was placed carefully inside outer vial (5 mL) having oxalic acid. Further, the 5 mL reaction vessel tighten with the solid PTFE faced solid cap and Teflon tape. The system was further stirred in oil bath heated at 130 °C for the required time. The reaction progress was monitored by TLC and after the completion of the reaction, the inner vial was removed. The contents of the inner vial in a separatory funnel. Further, water was added in the reaction mixture and extracted with ethyl acetate. The combined organic layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude mixture was further purified by silica gel column chromatography using hexane:ethyl acetate (60:40) as elutent, afforded 3a as white solid (23 mg, 75%).

1H (600MHz, DMSO- d6), (δ ppm) 2.34(s, 3H), 2.34 (s, 3H), 7.245 (d, J = 7.98 Hz, 2H), 7.28 (brs, 1H), 7.775 (d, J = 8.1 Hz, 2H), 7.90 (brs, 1H).

13C (150 MHz, DMSO- d6), δ (ppm) 21.40, 127.90, 129.19, 131.94, 141.51, 168.25.

The expected ESI-MS, [M+H]⁺ for C₈H₁₀NO⁺ was 136.0757 and observed is 136.0755.

2. benzamide

Prepared as general procedure described for 3a from iodobenzene (0.245 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (35:65) as elutents 3b as white solid (19 mg, 65%).
$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 7.37 (brs, 1H), 7.45 (t, $J = 7.4$ Hz, 2H), 7.51-7.53 (m, 1H), 7.875 (d, $J = 15$, 7.32 Hz, 2H), 7.98 (brs, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 127.90, 128.66, 131.67, 134.71, 168.34

The calculated ESI-MS+H⁺ for C$_7$H$_8$NO⁺ is 122.06 and found 122.0602.

3-methylbenzamide

![3c](image)

Prepared as general procedure described for 3a from 3-iodotoluene (0.229 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (35:65) as elutents 3c as white solid (16 mg, 52%).

$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 2.35 (s, 3H), 7.32 (d, $J = 4.5$ Hz, 3H), 7.65-7.67 (m, 1H), 7.70 (s, 1H), 7.93 (brs, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 21.41, 125.04, 128.52, 128.54, 132.22, 134.71, 137.88, 168.48.

The expected ESI-MS, [M+H⁺]⁺ for C$_8$H$_{10}$NO⁺ was 136.0757 and observed is 136.0755

2-ethylbenzamide

![3d](image)

Prepared as general procedure described for 3a from 1-ethyl 2-iodobenzene (0.215 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (35:65) as elutents 3d as white solid (14 mg, 45%).

$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 1.14-1.16 (t, $J = 7.5$, 15Hz, 3H), 2.71-2.75 (q, $J = 7.4$ Hz, 2H), 7.21-7.22 (m, 1H), 7.25 (d, $J = 7.5$ Hz), 7.30-7.35 (m, 3H), 7.73 (brs, 1H).

The expected ESI-MS [M+H⁺]⁺ for C$_9$H$_{12}$NO⁺ is 150.0913 and observed is 150.0912.

4-(tert-butyl)benzamide

![3e](image)
Prepared as general procedure described for 3a from 4-tert-butyliodobenzene (0.192 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3e as white solid (18 mg, 53%).

$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 1.29 (s, 9H), 7.29 (brs, 1H), 7.45-7.46 (m, 2H), 7.79-7.81 (m, 2H), 7.91 (brs, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 31.42, 35.05, 125.42, 127.78, 132.00, 154.41, 168.26.

The calculated ESI-MS [M+H]$^+$ for C$_{11}$H$_{16}$NO$^+$ 178.1226 and found 178.1223.

3, 5-dimethylbenzamide$^2$

Prepared as general procedure described for 3a from 1-iodo 3, 5 dimethylbenzene (0.215 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3d as white solid (17 mg, 54%).

$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 2.30 (s, 6H), 7.14 (s, 1H), 7.23 (brs, 1H), 7.48 (s, 2H), 7.85 (brs, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 21.31, 125.71, 132.89, 134.75, 137.70, 168.59.

The calculated ESI-MS [M+H]$^+$ for C$_9$H$_{12}$NO$^+$ 150.0913 and found 150.0910

2,4-dimethylbenzamide$^2$

Prepared as general procedure described for 3a from 1-iodo 3, 5 dimethylbenzene (0.215 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3g as white solid (20 mg, 63%).
$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 2.28 (s, 3H), 2.34 (s, 3H), 7.00-7.03 (m, 2H), 7.26-7.28 (m, 2H), 7.62 (s, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 21.02, 21.20, 126.33, 127.72, 131.62, 134.43, 135.83, 139.15, 171.46.

The calculated ESI-MS [M+H]$^+$ for C$_9$H$_{12}$NO$^+$ 150.0913 and found 150.0910.

4-methoxybenzamide

Prepared as general procedure described for 3a from 4-iodoanisole (0.213 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (1:1) as elutents 3h as white solid (20 mg, 61%).

$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 3.80 (s, 3H), 6.96-6.98 (m, 2H), 7.18 (brs, 1H), 7.84-7.86 (m, 3H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 55.77, 113.83, 126.98, 129.80, 162.04, 167.88.

The expected ESI-[MS+H]$^+$ C$_8$H$_{10}$NO$_2$$^+$ for 152.0706 is and observed is 152.0706.

3-methoxybenzamide

Prepared as general procedure described for 3a from 3-iodoanisole (0.213 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (1:1) as elutents 3i as white solid (25mg, 77%).

$^1$H (600MHz, DMSO-$d_6$), (δ ppm) 3.79 (s, 3H), 7.07-7.09 (m, 1H), 7.34-7.37 (m, 2H), 7.42-7.43 (m, 1H), 7.45-7.46 (m, 1H), 7.96 (brs, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 55.68, 113.10, 117.52, 120.15, 129.78, 136.19, 159.60, 168.10.

The calculated ESI-[MS+H]$^+$ C$_8$H$_{10}$NO$_2$$^+$ for 152.0706 and found 152.0707.

2-methoxybenzamide
Prepared as general procedure described for 3a from 2-iodoanisole (0.213 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (1:1) as elutents 3j as white solid (21 mg, 65%).

$^1$H (600 MHz, DMSO-$d_6$), (δ ppm) 3.89 (s, 3H), 7.02 (t, $J = 7.6$ Hz, 1H), 7.125 (d, $J = 8.2$ Hz, 1H), 7.46-7.48 (m, 1H), 7.52 (brs, 1H), 7.64 (brs, 1H), 7.80-7.81 (m, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), δ (pm) 56.26, 112.43, 120.85, 123.17, 127.95, 131.91, 157.69, 167.77

The calculated ESI-[MS+H]$^+$ $C_8H_{10}NO_2$+ for 152.0706 and found 152.0703.

3-fluorobenzamide

Prepared as general procedure described for 3a from 3-Fluoriodobenzene (0.225 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 3k as crystalline solid (16 mg, 51%).

$^1$H (600 MHz, DMSO-$d_6$), (δ ppm) 7.37-7.38 (m, 1H), 7.49-7.52 (m, 2H), 7.64-7.67 (m, 1H), 7.72-7.73 (m, 1H), 8.05 (brs, 1H)

$^{13}$C (150 MHz, DMSO-$d_6$), δ (ppm) 119.34-119.49 (d, $J = 22.6$ Hz), 123.28 (m), 128.82, 132.56-135.61 (m), 141.92-141.96 (d, $J = 6.0$ Hz), 166.38-167.99 (d, $J = 241$ Hz), 171.71-171.72 (d, $J = 1.5$ Hz).

The calculated ESI-[M+H]$^+$ for C$_7$H$_7$FNO$^+$ is 140.0506 and found 140.0505.

2-fluorobenzamide
Prepared as general procedure described for 3a from 2-Fluoroiodobenzene (0.225 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 3l as crystalline solid (26mg, 82%).

$^1$H (600MHz, DMSO-$d_6$), ($\delta$ ppm) 7.25-7.28 (m, 2H), 7.51-7.53 (m, 1H), 7.64-7.67 (m, 2H), 7.71 (brs, 1H)

$^{13}$C (150 MHz, DMSO-$d_6$), ($\delta$ ppm) 116.55 (d, $J = 22.4$ Hz), 124.27-124.36 (d, $J = 14.2$ Hz), 124.85-124.88 (d, $J = 3.4$ Hz), 130.68-130.70 (d, $J = 2.9$ Hz), 132.90-132.96 (d, $J = 8.6$ Hz), 130.68-130.70 (d, $J = 29$ Hz), 159.75 (d, $J = 3.4$ Hz), 165.72.

The calculated ESI-[M+H]$^+$ for C$_7$H$_7$FNO$^+$ 140.0506 and found 140.0506.

3-fluoro-4-methylbenzamide

Prepared as general procedure described for 3a from 4-Fluoro-3-iodotoluene (0.212 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 3m as white solid (18mg, 56%).

$^1$H (600MHz, DMSO-$d_6$), ($\delta$ ppm) 2.27 (s, 3H), 7.18-7.21 (t, $J = 9.0$ Hz, 1H), 7.30 (brs, 1H), 7.73-7.76 (m, 1H), 7.82-7.83 (m, 1H), 7.91 (s, 1H)

$^{13}$C (150 MHz, DMSO-$d_6$), ($\delta$ ppm) 14.62, 115.10-115.25 (d, $J = 22.4$ Hz), 124.47-124.58 (d, $J = 17.5$ Hz), 127.76-127.82 (d, $J = 8.9$ Hz), 130.84-130.86 (d, $J = 3.8$ Hz), 131.75 -131.79 (d, $J=5.7$ Hz), 162.06-163.70 (d, $J = 246$ Hz), 167.40.

The calculated ESI-[MS+H]$^+$ for C$_8$H$_9$FNO$^+$ 154.0663 and found 154.0663.

4-chlorobenzamide

Prepared as general procedure described for 3a from 1-Chloro-4-iodobenzene (0.210 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 3n as white solid (25mg, 76%).
\( ^1H \) (600 MHz, DMSO-\( d_6 \)), (\( \delta \) ppm) 7.46 (brs, 1H), 7.51-7.53 (m, 2H), 7.88-7.89 (m, 2H), 8.04 (brs, 1H)

\( ^{13}C \) (150 MHz, DMSO-\( d_6 \)), \( \delta \) (ppm) 128.96, 129.87, 133.51, 136.54, 167.28.

The calculated ESI-[MS+H]\(^+\) for C\(_7\)H\(_7\)ClNO\(^+\) is 156.0211 and found 156.0210.

2-chlorobenzamide

\[
\begin{array}{c}
\text{O} \\
\text{Cl} \\
\text{3o}
\end{array}
\]

Prepared as general procedure described for 3\( a \) from 1-Chloro-2-iodobenzene (0.210 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3\( o \) as crystalline solid (24 mg, 74%).

\( ^1H \) (600 MHz, DMSO-\( d_6 \)), (\( \delta \) ppm) 7.36-7.39 (m, 1H), 7.41-7.45 (m, 2H), 7.47-7.48 (m, 1H), 7.55 (brs, 1H), 7.84 (brs, 1H)

\( ^{13}C \) (150 MHz, DMSO-\( d_6 \)), \( \delta \) (ppm) 127.48, 129.11, 130.06, 130.07, 131.02, 137.62, 168.64.

The expected ESI-[MS+H]\(^+\) for C\(_7\)H\(_7\)ClNO\(^+\) is 156.0211 and found 156.0209.

3,4-dichlorobenzamide

\[
\begin{array}{c}
\text{O} \\
\text{Cl} \\
\text{Cl} \\
\text{3p}
\end{array}
\]

Prepared as general procedure described for 3\( a \) from 3,4-Dichloroiodobenzene (0.183 mmol, 50 mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3\( p \) as white solid (17 mg, 49%).

\( ^1H \) (600 MHz, DMSO-\( d_6 \)), (\( \delta \) ppm) 7.58 (brs, 1H), 7.74-7.75 (d, J=8.3 Hz, 1H), 7.84-7.85 (d, J=8.28 Hz, 1H), 8.10-8.12 (m, 2H).

\( ^{13}C \) (150 MHz, DMSO-\( d_6 \)), \( \delta \) (ppm) 128.21, 19.93, 131.11, 131.67, 134.52, 135.18, 166.63.

The expected ESI-[MS+H]\(^+\) for C\(_7\)H\(_6\)Cl\(_2\)NO\(^+\) is 189.9821 and observed is 189.9821.

2-hydroxybenzamide

\[
\begin{array}{c}
\text{O} \\
\text{3q}
\end{array}
\]
Prepared as general procedure described for 3a from 2-iodophenol (0.227 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (3:1) as elutents 3q as brownish solid (25mg, 80%).

$^1$H (600MHz, DMSO-d$_6$), (δ ppm) 6.84-6.88 (m, 2H), 7.38-7.41 (m, 1H), 7.83-7.84 (m, 1H), 7.87 (brs, 1H), 8.38 (brs, 1H), 13.02 (brs, 1H).

$^{13}$C (150 MHz, DMSO-d$_6$), δ (ppm) 114.82, 117.87, 118.82, 128.55, 134.55, 161.38, 172.54.
The calculated ESI-MS[M+H]$^+$ for C$_7$H$_8$NO$_2$ $^+$ 138.055 and found 138.0551.

4-acetylbenzamide$^2$

Prepared as general procedure described for 3a from 4'-iodoacetophenone (0.203 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3d as white solid (21mg, 65%).

$^1$H (600MHz, DMSO-d$_6$), (δ ppm) 2.62 (s, 3H), 7.59 (brs, 1H), 7.98-7.99 (m, 2H), 8.01-8.02 (m, 2H), 8.16 (brs, 1H).

$^{13}$C (150 MHz, DMSO-d$_6$), δ (ppm) 27.45, 128.22, 128.57, 138.55, 139.09, 167.54, 198.23.
The calculated ESI-MS+H$^+$ for C$_9$H$_{10}$NO$_2$ $^+$ 164.0706 and found 164.706.

4-nitrobenzamide$^2$

Prepared as general procedure described for 3a from 1-Iodo-4-nitrobenzene (0.200 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3s as white solid (19mg, 57%).

$^1$H (600MHz, DMSO-d$_6$), (δ ppm) 7.72 (brs, 1H), 8.09-8.10 (d, $J$=8.76 Hz, 2H), 8.27 (brs, 1H), 8.30-8.31 (d, $J$=8.76 Hz, 2H)

$^{13}$C (150 MHz, DMSO-d$_6$), δ (ppm) 123.94, 129.38, 140.44, 149.52, 166.68
The calculated ESI-[MS+H]⁺ for C₇H₇N₂O₃⁺ 167.0451 and found 169.0458.

**4-cyanobenzamide**

![Image of 4-cyanobenzamide](3t)

Prepared as general procedure described for 3a from 4-Iodobenzonitrile (0.218 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3t as white solid (23mg, 72%).

\[ ^1H \quad (600\text{MHz, DMSO-}d_6, (\delta \text{ ppm}) \] 7.64 (brs, 1H), 7.94 (d, J = 8.28 Hz, 2H), 8.02 (d, J = 8.4 Hz, 2H), 8.19 (brs, 1H)

\[ ^{13}C \quad (150 \text{ MHz, DMSO-}d_6), (\delta \text{ ppm}) \] 114.11, 118.82, 128.72, 132.82, 138.79, 166.91.

The calculated ESI-[MS+H]⁺ for C₈H₇N₂O⁺ 147.0553 and found 147.0459.

**[1,1'-biphenyl]-4-carboxamide**

![Image of [1,1'-biphenyl]-4-carboxamide](3u)

Prepared as general procedure described for 3a from 4-iodobiphenyl (0.178 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 3u as white solid (21mg, 60%).

\[ ^1H \quad (600\text{MHz, DMSO-}d_6, (\delta \text{ ppm}) \] 7.38-7.42 (m, 2H), 7.48-7.51 (t, J =7.56, 15.4 Hz, 2H), 7.72-7.76 (m, 4H), 7.97 (d, J=8.3 Hz, 2H), 8.02 (brs, 1H)

\[ ^{13}C \quad (150 \text{ MHz, DMSO-}d_6), (\delta \text{ ppm}) \] 126.91, 127.34, 128.48, 128.63, 129.48, 133.57, 139.71, 143.23, 168.02

The calculated ESI-MS [M+H]⁺ for C₁₃H₁₂NO⁺ 198.0913 and found 198.0912.

**1-naphthamide**

![Image of 1-naphthamide](3v)
Prepared as general procedure described for 3a from 1-Iodonaphthalene (0.196 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3v as white solid (18mg, 55%).

\(^1\text{H} (600\text{MHz}, \text{DMSO-}d_6), (\delta \text{ ppm})\) 7.55-7.59 (m, 4H), 7.64-7.65 (m, 1H), 7.96-8.01 (m, 3H), 8.31-8.32 (m, 1H).

\(^{13}\text{C} (150 \text{ MHz, DMSO-}d_6), (\delta \text{ ppm})\) 125.42, 126.61, 127.09, 128.56, 128.75, 130.15, 130.19, 130.35, 133.68, 135.14, 171.07.

The calculated ESI-MS [M+H]⁺ for C\textsubscript{11}H\textsubscript{10}NO⁺ is 172.0757 and found 172.0754.

**2-naphthamide\(^2\)**

Prepared as general procedure described for 3a from 2-Iodonaphthalene (0.196 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3w as white solid (15mg, 45%).

\(^1\text{H} (600\text{MHz}, \text{DMSO-}d_6), (\delta \text{ ppm})\) 7.43 (brrs, 1H), 7.57-7.62 (m, 2H), 7.97-8.01 (m, 4H), 8.11 (brrs, 1H), 8.49 (s, 1H)

\(^{13}\text{C} (150 \text{ MHz, DMSO-}d_6), (\delta \text{ ppm})\) 124.86, 127.099, 128.01, 128.05, 128.21, 128.25, 129.31, 132.14, 132.63, 134.64, 168.42.

The calculated ESI-MS [M+H]⁺ for C\textsubscript{11}H\textsubscript{10}NO⁺ 172.0757 and found 172.0756.

**9H-fluorene-2-carboxamide**

Prepared as general procedure described for 3a from 2-Iodofluorene (0.171 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 3x as white solid (15mg, 42%).

\(^1\text{H} (600\text{MHz, DMSO-}d_6), (\delta \text{ ppm})\) 3.98 (s, 2H), 7.20 (brrs, 1H), 7.36-7.38(t, J = 7.3, 14.7 Hz, 1H), 7.41-7.43 (t, J = 7.38, 14.8 Hz, 1H), 7.63 (d, J = 7.38 Hz, 1H), 7.93-7.97 (m, 4H), 8.11 (s, 1H).
$^{13}$C (150 MHz, DMSO-$d_6$), $\delta$ (ppm) 120.05, 121.19, 124.82, 125.76, 126.96, 127.39, 128.01, 133.05, 140.74, 143.31, 144.33, 144.39, 168.47.

The calculated ESI-[M+H]$^+$ for $C_{14}H_{12}NO^+$ 210.0913 and found 210.0913.

thiophene-2-carboxamide$^3$

Prepared as general procedure described for 3a from 2-Iodothiophene (0.238 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 3y as white solid (20mg, 66%).

$^1$H (600MHz, DMSO-$d_6$), ($\delta$ ppm) 7.12-7.13 (t, $J = 4.0$ Hz, 8.6 Hz, 1H), 7.36 (brs, 1H), 7.73-7.74 (m, 2H), 7.95 (brs, 1H).

$^{13}$C (150 MHz, DMSO-$d_6$), $\delta$ (ppm) 128.33, 129.11, 131.42, 140.79, 163.33.

The calculated ESI-[M+H]$^+$ for $C_5H_6NOS^+$ 128.0165 and found 128.0159.

isoindoline-1,3-dione$^4$

Prepared as general procedure described for 3a from 1,2-Diiodobenzene (0.152 mmol, 50mg) or 2-Bromiiodobenzene (0.176 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 4a as white solid (20mg, 66%).

$^1$H (600MHz, DMSO-$d_6$), ($\delta$ ppm) 7.83 (s,1H), 11.35 (brs, 1H)

$^{13}$C (150 MHz, DMSO-$d_6$), $\delta$ (ppm) 123.41, 133.06, 134.80, 169.71.

Prepared as general procedure described for 3a from 2-Iodobenzoic acid (0.201 mmol, 50mg) or Methyl 2-iodobenzoate (0.190 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (60:40) as elutents 4a as white solid (20mg, 66%).

$^1$H (600MHz, DMSO-$d_6$), ($\delta$ ppm) 7.82 (s,1H), 11.35 (brs, 1H)

$^{13}$C (150 MHz, DMSO-$d_6$), $\delta$ (ppm) 123.40, 133.06, 134.79, 169.71.

1,3-diphenylurea
Prepared as general procedure described for 3a from 2-Iodoaniline (0.229 mmol, 50mg), gave after purification with silica gel column chromatography in Hexane: ethyl acetate (40:60) as elutents 3a’ as white solid (20mg, 80%).

$^1$H (600MHz, DMSO-d$_6$), (δ ppm) 6.95-6.98 (m, 2H), 7.26-7.29 (m, 4H), 7.44-7.45 (m, 4H), 8.65 (s, 2H).

$^{13}$C (150 MHz, DMSO-d$_6$), δ (ppm) 118.64, 122.27, 129.45, 140.15, 152.99.

The calculated ESI-[M+H]$^+$ for C$_5$H$_6$NOS$^+$ 213.1022 and found 213.1020.

D. References:

(E) Copies of $^1$H, $^{13}$C and ESI-MS

$3a$, $^1$H, DMSO-d$_6$, 600 MHz

$3a$, $^{13}$C, DMSO-d$_6$, 150 MHz
3a, ESI-MS
\([\text{M+H}]^+ = 136.0757\]
\([\text{M+Na}]^+ = 158.0756\)

3b, \(^1\text{H}, \text{DMSO-d6}, 600 \text{ MHz}\)
3b, $^{13}$C, DMSO-$d_6$, 150 MHz

$\text{[M+H]}^+ = 122.0602$

$\text{[M+Na]}^+ = 144.0535$
3c, DMSO-$d_6$, 
$^1$H, 600 MHz

3c, DMSO-$d_6$, 
$^{13}$C, 150 MHz
3c, ESI-MS
\[ [M+H]^+ = 136.0755 \]
\[ [M+Na]^+ = 158.0756 \]

3d, DMSO-d6,
\[^1\text{H}, 600 \text{ MHz}\]
3d, ESI-MS
[M+H]^+ = 150.0910
[M+Na]^+ = 172.0805

3e, DMSO-d6, 1H, 600 MHz
$3e$, DMSO-$d_6$, $^{13}$C, 150 MHz

$[M+H]^+=178.1223$

$[M+Na]^+=200.1353$
3f, DMSO-d$_6$, $^1$H, 600MHz

3f, DMSO-d$_6$, $^{13}$C, 150MHz
\[ 3\text{f. ESI-MS} \]
\[ [\text{M+H}^+] = 150.0910 \]
\[ [\text{M+Na}^+] = 172.0805 \]
3g, DMSO-d$_6$, $^{13}$C, 150 MHz

$\text{[M+H]}^+ = 150.0912$

$\text{[M+Na]}^+ = 172.0665$

3g, ESI-MS

$\text{[M+H]}^+ = 150.0912$

$\text{[M+Na]}^+ = 172.0665$
3h, DMSO-d$_6$, $^1$H, 600 MHz

3h, DMSO-d$_6$, $^{13}$C, 150 MHz
3h, ESI-MS
[M+H]^+=152.0706
[M+Na]^+=174.0686

3i, DMSO-d6, ^1^H, 600 MHz
**3i, DMSO-d$_6$, $^{13}$C, 150 MHz**

$[\text{M+H}]^+ = 152.0707$

$[\text{M+Na}]^+ = 174.0611$
$3j$, DMSO-d$_6$, $^1$H, 600MHz

$3j$, DMSO-d$_6$, $^{13}$C, 150MHz
$\text{3j, ESI-MS}$

$[\text{M+H}]^+=152.0703$
$[\text{M+Na}]^+=174.0544$

$\text{3k, DMSO-d}_6$

$^1\text{H}, 600 \text{ MHz}$
3k, DMSO-d$_6$, $^{13}$C, 150 MHz

3k, ESI-MS

$[M+H]^+=140.0505$
$3l$, ESI-MS

$[M+H]^+=140.0506$

$[M+Na]^+=162.0522$

$3m$, DMSO-$d_6$

$1H, 600MHz$
3m, DMSO-<sub>d6</sub>, <sup>13</sup>C, 150MHz

3m, ESI-MS
[M+H]<sup>+</sup>=154.0663
$3n$, DMSO-$d_6$, $^1H$, 600 MHz

$3n$, DMSO-$d_6$, $^{13}C$, 150 MHz
3n, ESI-MS
$[\text{M+H]}^+=156.0209$

3o, DMSO-$d_6$,
$^1\text{H}$, 600 MHz
$\text{O}$

$\text{3o, DMSO-d$_6$,}$

$\text{^{13}C, 150 MHz}$

\[\text{[M+H]}^+=156.0210\]

$\text{3o, ESI-MS}$

$\text{[M+H]}^+=156.0210$
$\text{3p}, \text{DMSO-d}_6, 1H, 600 \text{ MHz}$

$\text{3p}, \text{DMSO-d}_6, 1\text{C}, 150 \text{ MHz}$
3p, ESI-MS
[M+H]^+ = 189.9821

3q, DMSO-d6, 1H, 600MHz
$\text{3q, DMSO-d}_6$, $^{13}$C, 600 MHz
3q, ESI-MS
[M+H]^+ = 138.0551

3r, ^1^H, DMSO-d_6,
600 MHz
$3r$, $^1H$, DMSO-d$_6$, 150 MHz

$\text{ESI-MS, } [\text{M+H}]^+ = 164.0706$
$\text{NH}_2$ $\text{O}$ $\text{O}_2\text{N}$ $3s$, DMSO-$d_6$, $^{13}\text{C}$, 150 MHz

$\text{NH}_2$ $\text{O}$ $\text{O}_2\text{N}$ $3s$, DMSO-$d_6$, $^1\text{H}$, 600 MHz
3s,
ESI-MS, [M+H]^+ 
= 169.0458

3t, DMSO-d_6, ^1H, 600 MHz
3t, DMSO-d$_6$, $^{13}$C, 600 MHz

3t, ESI-MS, [M+H]$^+$

= 147.0549
3u, ESI-MS, [M+H]^+ = 198.0912

3v, DMSO-d6, ^1^H, 600MHz
$3v$, DMSO-$d_6$, $^{13}$C, 600MHz

$[M+H]^+ = 172.0754$

$3v$, ESI-MS
$3w$, DMSO-$d_6$, $^1H$, 600 MHz

$3w$, DMSO-$d_6$, $^{13}C$, 150 MHz
$\text{3w, ESI-MS}$

$[\text{M+H}^+]=172.0756$

$\text{3x, DMSO-d_6, } ^1\text{H}$,

$600 \text{ MHz}$
$3x$, DMSO-$d_6$, $^{13}C$, 150 MHz

$[M+H]^+=210.0913$

$3x$, ESI-MS
$[M+H]^+=210.0913$
3y, DMSO-d₆, ¹H, 600 MHz

3y, DMSO-d₆, ¹³C, 600 MHz
3y, ESI-MS
[M+H]$^+$=128.0159
[M+Na]$^+$=150.0874
[M+K]$^+$= 167.0270

4a, DMSO-d$_6$, $^1$H, 600 MHz
4a, DMSO-d$_6$, $^{13}$C, 150 MHz

4a, DMSO-d$_6$, $^1$H, 600 MHz
4a, DMSO-\textsubscript{d6}, $^{13}$C, 150 MHz

4a, DMSO-\textsubscript{d6}, $^1$H, 600 MHz
$NH_3$, DMSO-d$_6$, $^1H$, 600 MHz

4a, DMSO-d$_6$, $^{13}C$, 150 MHz

$3a'$, DMSO-d$_6$, $^1H$, 600 MHz
3a', DMSO-d$_6$, $^{13}$C, 600 MHz

3a',
ESI-MS [M+H$^+$] = 213.1020
[M+H$^+$+H$_2$O] = 231.0254
5, ESI-MS
=187.0864