#### Supporting Information

# Trash to Treasure: Eco-Friendly and Practical Synthesis of Amides by Nitriles Hydrolysis in WEPPA

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#### 1. General and materials

General. <sup>1</sup>H, <sup>13</sup>C and <sup>19</sup>F NMR spectra were recorded on a Varian Inova-400 (400 MHz, 100 MHz and 376 MHz, respectively) spectrometer. <sup>1</sup>H and <sup>13</sup>C NMR chemical shifts were determined relative to internal standard TMS at  $\delta$  0.0 or CDCl<sub>3</sub>  $(\delta(^{1}\text{H}), 7.26 \text{ ppm}; \delta(^{13}\text{C}), 77.16 \text{ ppm})$  or  $d_6$ -DMSO ( $\delta(^{1}\text{H}), 2.54 \text{ ppm}; \delta(^{13}\text{C}), 39.50$ ppm) and <sup>19</sup>F NMR chemical shifts were determined relative to CFCl<sub>3</sub> as internal standard. Chemical shifts ( $\delta$ ) are reported in ppm, and coupling constants (J) are in Hertz (Hz). The following abbreviations are used to explain the multiplicities: s =singlet, d = doublet, t = triplet, q = quartet, m = multiplet, bs = broad singlet. pH values were detected by PHS-3C acidometer. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) analysis was carried out on a Varian VISTA-PRO spectrometer. X-Ray photoelectron spectroscopy (XPS) was detected on a Thermo Scientific K-Alpha+X spectrometer. Energy dispersive X-ray (EDX) was recorded on the SU8010 cold field emission ultra-high resolution scanning electron microscope. The melting point was recorded on BÜCHI (M-560) and uncorrected. Analytical thin layer chromatography (TLC) was performed on 0.25 mm silica gel 60 F254 plates and viewed by UV light (254 nm). Column chromatographic purification was performed using 200-300 mesh silica gel.

**Materials.** All the chemical reagents were purchased from commercial sources and used as received unless otherwise indicated.

2. General procedure for the preparation of AWEs (taking WEPPA as an example)



The pomelo peel was obtained and dried naturally. The dried pomelo peel was burned to get its ash. Then, one gram pomelo peel ash was suspended into 10.0 mL of distilled water at room temperature for 30 min with constant stiring. The suspension was then filtered to obtain a pale yellow extract which named as WEPPA.

3. XPS spectrum of the pomelo peel ash



Figure S1 XPS spectrum of the pomelo peel ash

Element	Start	Peak	End	Height	FWHM	Area (P)		Atomio 0/
Element	BE	BE	BE	CPS	eV	CPS.eV	Area (N)	Atomic %
0	537.48	531.34	524.28	461018.18	2.4	1200937.11	6963.79	33.76
K	298.68	293.22	290.76	270622.92	2.4	704963.77	2127.97	10.32
C	290.69	284.78	277.28	342691.77	2.05	760946.07	10670.16	51.73
Ca	360.08	347.26	343.68	60779.2	2.4	158327.81	387.4	1.88

#### Table S1 XPS analysis

Р	138.08	133.08	124.88	7161.84	2.4	18656.35	176.39	0.86
S	174.68	168.95	159.88	6485.17	2.4	16893.65	117.31	0.57
Cl	210.08	198.79	188.08	5350.36	2.4	13937.51	67.6	0.33
Si	105.68	102.24	93.28	3240.34	2.4	8440.99	117.89	0.57

4. General procedure for the hydrolysis of nitriles in WEPPA (taking 1a as an example)



Under atmosphere, benzonitrile **1a** (103 mg, 1.0 mmol) and WEPPA (2.0 mL) were added into a 10 mL closed tube with a stir bar. Then the reaction was stirred in a closed vessel synthesis reactor at 150 °C for 0.5 h. After cooling to ambient temperature, the resulting precipitate was collected by filtration, washed with ice water and further dried in the vacuum drying oven. The filtrate was evaporated under reduced pressure. The resultant residue was purified by silica gel column chromatography (eluent: petroleum ether (35-60 °C)/EtOAc = 2:1 to 0:1, v/v). Finally, cmbining these two parts to afford the desired benzamide **2a** in 85% yield.

#### 5. Gram-scale experiments (taking 10 at 100 mmol as an example)



Under atmosphere, 2-aminobenzonitrile **1o** (11.8 g, 100.0 mmol) and WEPPA (150.0 mL) were added into a 300 mL closed tube with a stir bar. Then the reaction was stirred in an oil bath at 150 °C for 5 h. After cooling to ambient temperature, large amount of white solid precipitated out and was collected by filtration, washed with ice water and further dried in the vacuum drying oven. The filtrate was evaporated under reduced pressure to get the residual product. Finally, cmbining these two parts to afford the desired 2-aminobenzamide **2o** (11.6 g) in 85% yield.

6. Recycling experiments



Under atmosphere, 4-fluorobenzamide **1g** (121 mg, 1.0 mmol) and WEPPA (2.0 mL) were added into a 10 mL closed tube with a stir bar. Then the reaction was stirred in a closed vessel synthesis reactor at 150 °C for 0.5 h. After cooling to ambient temperature, the resulting precipitate was collected by filtration, washed with ice water and further dried in the vacuum drying oven. The WEPPA filtrate could be reused at least four times in good yields (89%, 88%, 84% and 75%).



#### 7. Comparative experiments

 Table S2 The conversions of 1a in the water solutions of different inorganic

 carbonates or oxides<sup>a</sup>

		$\frac{150 ^{\circ}\text{C}}{0.5 \text{h}} \qquad $	
	1a	2a	
Entry	Compound	Loading (g/10 mL)	GC yields $(\%)^b$
1	$K_2CO_3$	0.2136	17
2	Na <sub>2</sub> CO <sub>3</sub>	$1.2*10^{-3}$	N.R
3	CaCO <sub>3</sub>	0.0741	N.R
4	MgCO <sub>3</sub>	0.0192	Trace
5	CuCO <sub>3</sub>	$0.054*10^{-3}$	N.R
6	MnCO <sub>3</sub>	0.012	N.R
7	CaO	0.0741	40
8	MgO	0.0192	N.R
9	CuO	$0.054*10^{-3}$	N.R
10	$Fe_2O_3$	0.18*10 <sup>-3</sup>	N.R
11	MnO <sub>2</sub>	0.012	N.R
$12^{c}$	mixture		26

<sup>*a*</sup> Reaction conditions: **1a** (1.0 mmol), water solutions of inorganic carbonates or oxides (2.0 mL), 150 °C, 0.5 h. <sup>*b*</sup> Determined by GC analysis. <sup>*c*</sup> According to the ICP analysis, water solution of all these inorganic carbonates and oxides entries 1-11.

#### 8. Analytical data



**Benzamide** (2a)<sup>[1]</sup>: Known compound. 114.2 mg, 94% yield. White solid. m.p.: 127.3-129.1 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.83-7.80 (m, 2H ), 7.54-7.50 (m, 1H), 7.46-7.42 (m, 2H), 6.26 (bs, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 169.7, 133.5, 132.1, 128.8, 127.5.



**2-Methylbenzamide** (**2b**)<sup>[1]</sup>: Known compound. 114.1 mg, 84% yield. White solid. m.p.: 140.1-142.9 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.43 (d, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 7.6 and 1.3 Hz, 1H), 7.21 (q, *J* = 7.2 Hz, 2H), 6.28 (bs, 1H), 5.86 (bs, 1H), 2.49 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 172.4, 136.4, 135.4, 131.3, 130.4, 127.1, 125.8, 20.1.



**3-Methylbenzamide** (**2c**)<sup>[1]</sup>: Known compound. 108.2 mg, 80% yield. White solid. m.p.: 90.1-91.1 °C. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>, 400 MHz) δ 7.65 (s, 1H), 7.60-7.58 (m, 1H), 7.33-7.29 (m, 2H), 6.30 (bs, 2H), 2.39 (s, 3H); <sup>13</sup>**C NMR** (CDCl<sub>3</sub>, 100 MHz) δ 170.0, 138.6, 133.5, 132.8, 128.6, 128.2, 124.4, 21.4.



**4-Methylbenzamide** (**2d**)<sup>[1]</sup>: Known compound. 120.2 mg, 89% yield. White solid. m.p.: 148.1-148.8 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.71 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 9.0 Hz, 2H), 5.93 (bs, 2H), 2.42 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 169.5, 142.7, 130.6, 129.4, 127.5, 21.6.



**4-Ethylbenzamide** (2e)<sup>[2]</sup>: Known compound. 132.6 mg, 89% yield. White solid. m.p.: 160.2 -162.5 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.3 Hz, 2H), 6.06 (bs, 2H), 2.70 (q, *J* = 7.6 Hz, 2H), 1.25 (t, *J* = 7.6 Hz, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 169.7, 148.8, 130.9, 128.2, 127.6, 28.9, 15.4.



**4-(Chloromethyl)benzamide (2f)**<sup>[3]</sup>: Known compound. 107.2 mg, 63% yield. White solid. m.p.: 133.3 -135.1 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz)  $\delta$  7.92 (bs, 1H), 7.84 (d, J = 8.2 Hz, 2H), 7.38 (d, J = 8.0 Hz, 2H), 7.30 (bs, 1H), 4.56 (s, 2H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz)  $\delta$  167.8, 145.9, 132.6, 127.3, 125.9, 62.5.



**4-Fluorobenzamide** (**2g**)<sup>[1]</sup>: Known compound. 127.0 mg, 91% yield. White solid. m.p.: 155.3-155.5 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 8.03 (bs, 1H), 8.00-7.96 (m, 2H), 7.43 (bs, 1H), 7.34-7.28 (m, 2H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 166.8, 163.9 (d, *J* = 245.8 Hz), 130.7 (d, *J* = 11.5 Hz), 130.1 (d, *J* = 9.0 Hz), 115.1 (d, *J* = 21.6 Hz); <sup>19</sup>F NMR (*d*<sub>6</sub>-DMSO, 376 MHz) δ -109.6.



4-Chlorobenzamide (2h)<sup>[1]</sup>: Known compound. 130.1 mg, 84% yield. White solid.
m.p.: 177.4-178.8 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.77-7.74 (m, 2H), 7.45-7.42 (m, 2H), 5.85 (bs, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 168.3, 138.5, 131.8, 129.1, 128.9.



**4-Bromobenzamide** (**2i**)<sup>[1]</sup>: Known compound. 165.0 mg, 83% yield. White solid. m.p.: 188.9-191.6 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 8.08 (bs, 1H), 7.87-7.84 (m, 2H), 7.72-7.68 (m, 2H), 7.49 (bs, 1H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 166.9, 133.4, 131.2, 129.6, 125.0.



**4-Formylbenzamide** (**2j**)<sup>[4]</sup>: Known compound. 91.0 mg, 61% yield. White solid. m.p.: 178.9-182.1 °C. <sup>1</sup>**H NMR** (*d*<sub>6</sub>-DMSO, 400 MHz) δ 10.09 (bs, 1H), 8.19 (bs, 1H), 8.07 (d, *J* = 7.9 Hz, 2H), 7.99 (d, *J* = 8.0 Hz, 2H), 7.62 (bs, 1H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 192.9, 167.0, 139.3, 137.8, 129.3, 128.1.



**4-Acetylbenzamide** (**2k**)<sup>[1]</sup>: Known compound. 110.8 mg, 68% yield. Yellow solid. m.p.: 192.5-194.1 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 8.15 (bs, 1H), 8.04-7.98 (m, 4H), 7.57 (bs, 1H), 2.62 (s, 3H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 197.7, 167.1, 138.6, 138.1, 128.1, 127.7, 26.9.



[1,1'-Biphenyl]-4-carboxamide (2l)<sup>[5]</sup>: Known compound. 164.1 mg, 83% yield. White solid. m.p.: 232.1-234.5 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  8.08 (bs, 1H), 8.02 (d, J = 8.4 Hz, 2H), 7.80 (bs, 1H), 7.78-7.75 (m, 3H), 7.53 (t, J = 7.3 Hz, 2H), 7.44 (t, J = 7.2 Hz, 2H); <sup>13</sup>C NMR ( $d_6$ -DMSO, 100 MHz)  $\delta$  167.5, 142.7, 139.2, 133.1, 129.0, 128.1, 128.0, 126.8, 126.4.

H<sub>2</sub>N O

**1-Naphthamide** (**2m**)<sup>[1]</sup>: Known compound. 94.6 mg, 55% yield. White solid. m.p.: 204.8-206.2 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  8.36 (d, J = 7.3 Hz, 1H), 8.05-8.00

(m, 3H), 7.70-7.55 (m, 5H); <sup>13</sup>**C NMR** (*d*<sub>6</sub>-DMSO, 100 MHz) δ 170.5, 134.6, 133.2, 129.7 x 2, 128.1, 126.6, 126.1, 125.6, 125.1, 124.9.



Anthracene-9-carboxamide (2n)<sup>[6]</sup>: Known compound. 90.7 mg, 41% yield. Yellow solid. m.p.: 186.2-188.6 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 8.68 (bs, 1H), 8.30 (bs, 1H), 8.16 (d, *J* = 7.9 Hz, 2H), 8.08 (d, *J* = 8.8 Hz, 3H), 7.64-7.57 (m, 4H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 170.2, 133.7, 130.7, 128.3, 126.8 x 2, 126.2, 125.5, 125.4.



**2-Aminobenzamide** (**2o**)<sup>[1]</sup>: Known compound. 130.5 mg, 96% yield. Yellow solid. m.p.: 110.1-111.5 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.36 (dd, *J* = 7.9 and 1.3 Hz, 1H), 7.25-7.20 (m, 1H), 6.68 (d, *J* = 8.2 Hz, 1H), 6.66-6.62 (m, 1H), 5.90 (bs, 2H), 5.67 (bs, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 171.8, 149.6, 133.1, 128.1, 117.6, 116.5, 114.1.



**2-Amino-6-methylbenzamide** (**2p**)<sup>[7]</sup>: Known compound. 139.9 mg, 93% yield. White solid. m.p.: 143.7-144.8 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.63 (bs, 1H), 7.42 (bs, 1H), 6.92 (t, *J* = 7.7 Hz, 1H), 6.51 (d, *J* = 7.9 Hz, 1H), 6.39 (d, *J* = 7.2 Hz, 1H), 4.90 (bs, 2H), 2.21 (s, 3H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 170.5, 145.4, 134.2, 128.7, 123.0, 117.9, 112.7, 19.9.



**2-Amino-5-methylbenzamide** (**2q**)<sup>[7]</sup>: Known compound. 135.9 mg, 90% yield. Yellow solid. m.p.: 172.6-174.3 °C. <sup>1</sup>**H NMR** (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.65 (bs, 1H), 7.34 (bs, 1H), 6.95 (dd, *J* = 8.2 and 1.5 Hz, 2H), 6.59 (d, *J* = 8.2 Hz, 1H), 6.31 (bs, 2H), 2.15 (s, 3H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 171.3, 147.8, 132.7, 128.6, 122.7, 116.5, 113.7, 20.0.



2-Amino-4-methylbenzamide (2r)<sup>[7]</sup>: Known compound. 146.1 mg, 97% yield. White solid. m.p.: 148.9-149.5 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.62 (bs, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 6.92 (bs, 1H), 6.53 (bs, 2H), 6.47 (s, 1H), 6.29 (d, *J* = 8.5 Hz, 1H), 2.16 (s, 3H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 171.2, 150.3, 141.6, 128.8, 116.4, 115.6, 111.1, 21.0.



2-Amino-6-chlorobenzamide (2s)<sup>[8]</sup>: Known compound. 135.6 mg, 79% yield. White solid. m.p.: 131.6-132.3 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.81 (bs, 1H), 7.58 (bs, 1H), 7.01 (t, *J* = 8.0 Hz, 1H), 6.64 (d, *J* = 8.1 Hz, 1H), 6.58 (d, *J* = 7.8 Hz, 1H), 5.21 (bs, 2H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 167.5, 147.0, 130.0, 129.8, 121.7, 116.1, 113.6.



**2-Amino-4-chlorobenzamide** (2t)<sup>[9]</sup>: Known compound. 155.2 mg, 91% yield. White solid. m.p.: 179.7-180.6 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  7.81 (bs, 1H), 7.57 (d, J = 8.5 Hz, 1H), 7.19 (bs, 1H), 6.86 (bs, 2H), 6.77 (d, J = 2.2 Hz, 1H), 6.52 (dd, J = 8.5 and 2.2 Hz, 1H); <sup>13</sup>C NMR ( $d_6$ -DMSO, 100 MHz)  $\delta$  170.4, 151.5, 136.3, 130.6, 115.1, 114.0, 112.4.



**2-Amino-4-(trifluoromethyl)benzamide** (**2u**)<sup>[10]</sup>: Known compound. 175.8 mg, 86% yield. White solid. m.p.: 150.8-151.1 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  7.96 (bs,

1H), 7.73 (d, J = 8.2 Hz, 1H), 7.36 (s, 1H), 7.07 (d, J = 1.1 Hz, 1H), 6.91 (bs, 2H), 6.78 (dd, J = 8.2 and 1.7 Hz, 1H); <sup>13</sup>**C NMR** (*d*<sub>6</sub>-DMSO, 100 MHz)  $\delta$  170.2, 150.2, 131.8 (q, J = 31.0 Hz), 129.9, 124.0 (d, J = 271.1 Hz), 116.7, 112.5 (d, J = 4.0 Hz), 109.9 (d, J = 3.6 Hz).



**3-Aminobenzamide** (**2v**)<sup>[10]</sup>: Known compound. 125.5 mg, 92% yield. Yellow solid. m.p.: 112.1-112.7 °C. <sup>1</sup>**H NMR** (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.74 (bs, 1H), 7.15 (bs, 1H), 7.10-7.07 (m, 2H), 7.02-7.00 (m, 1H), 6.72-6.70 (m, 1H), 5.21 (bs 2H); <sup>13</sup>**C NMR** (*d*<sub>6</sub>-DMSO, 100 MHz) δ 168.7, 148.5, 135.2, 128.5, 116.5, 114.7, 113.1.



**Isophthalamide**  $(2\mathbf{w})^{[11]}$ : Known compound. 136.1 mg, 83% yield. Pale yellow solid. m.p.: > 300 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz)  $\delta$  8.42 (bs, 1H), 8.13 (bs, 2H), 8.03 (dd, J = 7.7 and 1.7 Hz, 2H), 7.57 (t, J = 7.7 Hz, 1H), 7.50 (bs, 2H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz)  $\delta$  167. 5, 134. 4, 130. 1, 128. 2, 126. 8.



**Terephthalamide**  $(2\mathbf{x})^{[6]}$ : Known compound. 143.0 mg, 87% yield. Pale yellow solid. m.p.: > 300 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz)  $\delta$  8.11 (bs, 2H), 7.97 (s, 4H), 7.52 (bs, 2H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz)  $\delta$  167.3, 136.5, 127.3.



**4-(Cyanomethyl)benzamide**  $(2y)^{[12]}$ : Known compound. 123.6 mg, 77% yield. White solid. m.p.: > 300 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  8.02 (bs, 1H), 7.94-7.92 (m, 1H), 7.92 (t, J = 1.8 Hz, 1H), 7.46 (d, J = 8.4 Hz, 2H), 7.43 (bs, 1H), 4.15 (s, 2H); <sup>13</sup>C

NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 167.3, 134.4, 133.6, 128.1, 127.9, 118.9, 22.2.



**Cinnamamide**  $(2z)^{[1]}$  Known compound. 123.0 mg, 84% yield. White solid. m.p.: 148.2-148.8 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz)  $\delta$  7.56 (d, *J* = 6.9 Hz, 3H), 7.42-7.36 (m, 4H), 7.15 (bs, 1H), 6.63 (d, *J* = 15.9 Hz 1H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz)  $\delta$  166.7, 139.1, 134.9, 129.4, 128.9, 127.5, 122.3.



**Phenyl**(*o*-tolyl)methanone (2aa)<sup>[13]</sup>: Known compound. 144.4 mg, 63% yield. Yellow solid. m.p.: 160.9-162.5 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz)  $\delta$  7.35 (bs, 1H), 6.98 (bs, 1H), 4.80 (t, *J* = 1.8 Hz, 2H), 4.36 (t, *J* = 1.8 Hz, 2H), 4.20 (s, 5H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz)  $\delta$  171.5, 76.9, 70.4, 69.8, 69.0.



**Furan-2-carboxamide** (**2a'**)<sup>[14]</sup>: Known compound. 79.0 mg, 71% yield. White solid. m.p.: 140.1-141.3 °C. **<sup>1</sup>H NMR** (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.83 (t, *J* = 0.7 Hz, 1H), 7.80 (bs, 1H), 7.41 (bs,1H), 7.14 (d, *J* = 3.4 Hz, 1H), 6.62 (q, *J* = 1.7 Hz, 1H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 159.4, 148.0, 145.0, 113.6, 111.8.



**Thiophene-2-carboxamide** (**2b'**)<sup>[1]</sup>: Known compound. 101.6 mg, 80% yield. White solid. m.p.: 178.2-179.3 °C. <sup>1</sup>**H NMR** (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.95 (bs, 1H), 7.74 (s, 2H), 7.37 (bs, 1H), 7.13 (t, *J* = 3.9 Hz, 1H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 162.8, 140.3, 130.9, 128.6, 127.8.

$$[ \searrow_N^S \bigvee_O^{NH_2} ]$$

**Thiazole-2-carboxamide** (2c')<sup>[15]</sup>: Known compound. 78.1 mg, 61% yield. White solid. m.p.: 119.0-122.1 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  8.21 (bs, 1H), 8.06 (d, J = 3.1 Hz, 1H), 8.03 (d, J = 3.1 Hz, 1H), 7.88 (bs, 1H); <sup>13</sup>C NMR ( $d_6$ -DMSO, 100 MHz)  $\delta$  164.3, 160.9, 143.9, 125.9.



**Picolinamide** (2d')<sup>[1]</sup>: Known compound. 83.3 mg, 69% yield. White solid. m.p.: 106.3-108.8 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.56 (d, *J* = 4.7 Hz, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 7.90 (bs, 1H), 7.83 (td, *J* = 7.7 and 1.0 Hz, 1H), 7.44-7.41 (m, 1H), 6.41 (bs, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 167.2, 149.7, 148.4, 137.4, 126.5, 122.5.



**Nicotinamide** (2e)'<sup>[1]</sup>: Known compound. 91.2 mg, 75% yield. White solid. m.p.: 134.4-137.5 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  9.03 (d, J = 1.4 Hz, 1H), 8.69 (dd, J = 4.7 and 1.4 Hz, 1H), 8.22-8.19 (m, 1H), 8.18 (bs, 1H), 7.63 (bs, 1H), 7.49 (dd, J = 7.8 and 4.8 Hz, 1H); <sup>13</sup>C NMR ( $d_6$ -DMSO, 100 MHz)  $\delta$  166.5, 151.9, 148.7, 135.2, 129.7, 123.4.

**Isonicotinamide**. (**2f'**)<sup>[1]</sup>: Known compound. 104.8 mg, 86% yield. White solid. m.p.: 151.1-153.9 °C. <sup>1</sup>H NMR ( $d_6$ -DMSO, 400 MHz)  $\delta$  8.64 (dd, J = 4.3 and 1.5 Hz, 2H), 7.78 (dd, J = 4.3 and 1.6 Hz, 2H); <sup>13</sup>C NMR ( $d_6$ -DMSO, 100 MHz)  $\delta$  167.3, 149.6, 144.6, 123.1.



**1***H***-Indole-4-carboxamide** (**2g'**)<sup>[6]</sup>: Known compound. 130.9 mg, 82% yield. Pale yellow solid. m.p.: 143.2-145.7 °C. <sup>1</sup>**H NMR** (*d*<sub>6</sub>-DMSO, 400 MHz) δ 11.3 (bs, 1H),

7.75 (bs, 1H), 7.58 (d, J = 8.0 Hz, 1H), 7.51 (d, J = 7.3 Hz, 1H), 7.46 (t, J = 2.8 Hz, 1H), 7.25 (bs, 1H), 7.16 (t, J = 7.7 Hz, 1H), 6.96 (t, J = 2.0 Hz, 1H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz)  $\delta$  169.8, 136.6, 126.4, 126.2, 126.1, 120.0, 118.9, 114.2, 102.0.



Phenylacetamide (2a'')<sup>[1]</sup>: Known compound. 109.4 mg, 81% yield. White solid.
m.p.: 152.6-155.1 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.38-7.34 (m, 2H), 7.32-7.27 (m, 3H), 5.82 (bs, 1H), 5.41 (bs, 1H), 3.58 (s, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 173.7, 135.0, 129.5, 129.2, 127.6, 43.5.



**1,2,3,4-Tetrahydronaphthalene-1-carboxamide** (**2b''**)<sup>[16]</sup>: Known compound. 130.3 mg, 74% yield. White solid. m.p.: > 300 °C. <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO, 400 MHz) δ 7.49 (bs, 1H), 7.15-7.09 (m, 4H), 6.99 (bs, 1H), 3.64 (t, *J* = 6.8 Hz, 1H), 2.75-2.72 (m, 2H), 1.97-1.91 (m, 3H), 1.69-1.60 (m, 1H); <sup>13</sup>C NMR (*d*<sub>6</sub>-DMSO, 100 MHz) δ 176.2, 137.0, 135.3, 128.9, 128.4, 126.0, 125.4, 45.0, 28.8, 26.9, 20.6.



**Benzothioamide** (**3a**)<sup>[17]</sup>: Known compound. 99.8 mg, 73% yield. Yellow solid. m.p.: 114.5-115.7 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.95 (bs, 1H), 7.87-7.86 (m, 1H), 7.85-7.84 (m, 1H), 7.52-7.48 (m, 1H), 7.42-7.37 (m, 2H), 7.30 (bs, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 202.9, 139.2, 132.1, 128.6, 127.0.



**2-Benzoylquinazolin-4(3***H***)-one (3c)**<sup>[18]</sup>: Known compound. 188.0 mg, 75% yield. White solid. m.p.: 182.5-183.9 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 10.5 (bs, 1H), 8.52-8.49 (m, 2H), 8.39 (dd, *J* = 7.9 and 1.4 Hz, 1H), 7.93-7.91 (m, 1H), 7.86-7.82 (m, 1H), 7.69-7.61 (m, 2H), 7.56-7.52 (m, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 185.7, 161.1, 147.6, 146.1, 134.9, 134.4, 134.1, 131.9, 129.5 x 2, 128.5, 127.0, 123.4.

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## 10. Copies of NMR spectra

## <sup>1</sup>H NMR of product 2a in CDCl<sub>3</sub> (400 MHz)



## <sup>13</sup>C NMR of product 2a in CDCl<sub>3</sub> (100 MHz)



## <sup>1</sup>H NMR of product 2b in CDCl<sub>3</sub> (400 MHz)



## <sup>1</sup>H NMR of product 2c in CDCl<sub>3</sub> (400 MHz)



## <sup>1</sup>H NMR of product 2d in CDCl<sub>3</sub> (400 MHz)





## <sup>1</sup>H NMR of product 2f in *d*<sub>6</sub>-DMSO (400 MHz)



00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)





190 180 100 90 f1 (ppm) -1



## <sup>13</sup>C NMR of product 2h in CDCl<sub>3</sub> (100 MHz)



## <sup>13</sup>C NMR of product 2i in *d*<sub>6</sub>-DMSO (100 MHz)



0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

## <sup>1</sup>H NMR of product 2j in $d_6$ -DMSO (400 MHz)

68	040040
80.	002 002 002 002 002 002 002 002 002 002
<del>1</del>	0.00 00.7
1	



## <sup>13</sup>C NMR of product 2j in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2k in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2l in *d*<sub>6</sub>-DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2m in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2n in $d_6$ -DMSO (100 MHz)



110 100 90 f1 (ppm) 190 180 150 140 130 120 -1

#### <sup>1</sup>H NMR of product 20 in CDCl<sub>3</sub> (400 MHz)





#### <sup>13</sup>C NMR of product 20 in CDCl<sub>3</sub> (100 MHz)



## <sup>13</sup>C NMR of product 2p in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2q in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2r in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2s in $d_6$ -DMSO (100 MHz)





1.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1 f1(ppm)

## <sup>13</sup>C NMR of product 2u in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2v in $d_6$ -DMSO (100 MHz)



## <sup>13</sup>C NMR of product 2w in $d_6$ -DMSO (100 MHz)





## <sup>13</sup>C NMR of product 2y in $d_6$ -DMSO (100 MHz)



00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

## <sup>1</sup>H NMR of product 2z in $d_6$ -DMSO (400 MHz)

7 5579 7 5507 7 7 4134 7 7 4079 7 7 4079 7 7 3889 7 7 3889 7 7 3639 7 7 5339 6 6470 6 6470



## <sup>13</sup>C NMR of product 2z in $d_6$ -DMSO (100 MHz)











0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1 f1(ppm)













S55





## <sup>13</sup>C NMR of product 3c in CDCl<sub>3</sub> (100 MHz)

![](_page_56_Figure_1.jpeg)

00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)