One-pot synthesis of 2,3-dihydro-pyrrolopyridinones using *in situ* generated formimines.

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SUPPORTING INFORMATION – ¹H & ¹³C NMR, MS

NMR spectra were recorded on a 300 MHz Bruker Avance. Unless otherwise indicated, all spectra were recorded at 300 MHz for ¹H and 75 MHz for ¹³C in CDCl₃. All chemical shifts are given in ppm. R_f given for TLC analyses were measured on silica gel Merck Kieselgel F₂₅₄ aluminum plates.

This supporting information will be divided in two sections:

- section 1 : preparation of compounds **3**, **4a-4c** and **16-19** (page 2)
- section 2 : preparation of nicotinamide derivatives **1**, **6-13** and cyanomethylamine derivatives **2**, **14-15** (page 18)

Section 1 : Preparation of compounds 3, 4a-4c and 16-19

N,N-diisopropyl-4-[(4-methoxybenzylamino)methyl]nicotinamide **3**

Mol. Formula : C₂₁H₂₉N₃O₂

Mol. Weight : 355.47

To a stirred solution of *n*-butyllithium (4.28 mL, 10.7 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (2.1 mL, 12.3 mmol) at -10°C . The solution was allowed to stir at 0°C for 30 minutes then cooled to -78°C . *N,N*-diisopropylnicotinamide **1** (1.02 g, 4.93 mmol) in anhydrous THF (10 mL) was added in 5 minutes immediately followed by dropwise addition of 2-(4-methoxybenzylamino)acetonitrile **2** (0.87 mL, 5.42 mmol) in anhydrous THF (7 mL) and kept 7 hours at this temperature. The reaction was quenched with aqueous ammonium chloride (689 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated *in vacuo*. The product was purified by flash chromatography through SiO₂ (dichloromethane : methanol : conc. ammonia : 99 : 1 : 0.01; *R_f* : 0.33) providing **3** (348 mg, 65 %) as a brown oil.

¹H NMR (CDCl₃, 300 MHz): 0.95 (d, *J* = 6.6 Hz, 3H), 1.03 (d, *J* = 6.6 Hz, 3H), 1.45 (m, 6H), 1.97 (bs, 1H), 3.39-3.74 (m, 9H), 6.75 (d, *J* = 8.5 Hz, 2H), 7.14 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 5.0 Hz, 1H), 8.28 (s, 1H), 8.43 (d, *J* = 5.0 Hz, 1H)

¹³C NMR (CDCl₃, 75 MHz): 20.2, 20.3, 20.5, 20.6, 45.9, 49.3, 51.0, 52.8, 55.1, 113.6, 123.7, 129.2, 131.7, 133.7, 145.5, 145.8, 149.4, 158.5, 167.5

IC(+)MS (MH⁺) *m/z* = 356, 221, 136, 121

Anal. *calcd* for C₂₁H₂₉N₃O₂: C, 70.95; H, 8.22; N, 11.82 *found* C, 70.92 ; H, 8.13 ; N, 11.97

2-(4-methoxybenzyl)-1*H*-pyrrolo[3,4-*c*]pyridin-3(2*H*)-one **4a**

Mol. Formula : C₁₅H₁₄N₂O₂

Mol. Weight : 254.28

To a stirred solution of *n*-butyllithium (3.67 mL, 9.19 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (1.61 mL, 9.46 mmol) at -10°C . The solution was allowed to stir at 0°C for 30 minutes then cooled to -78°C . Piperidine nicotinamide **8** (500 mg, 2.63 mmol) in anhydrous THF (10 mL) was added in 5 minutes immediately followed by dropwise addition of 2-(4-methoxybenzylamino)acetonitrile **2** (0.46 mL, 2.89 mmol) in anhydrous THF (7 mL) and kept 7 hours at this temperature. The reaction was quenched with aqueous ammonium chloride (530 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated *in vacuo*. The product was purified by flash chromatography through SiO₂ (ethyl acetate : isopropanol 3 : 1 *R_f* = 0.34) providing **4a** (541 mg, 81 %) as a pale brown solid.

¹H NMR (CDCl₃, 300 MHz): 3.78 (s, 3H), 4.27 (s, 2H), 4.72 (s, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.5 Hz, 2H), 7.35 (d, *J* = 5.0 Hz, 1H), 8.71 (d, *J* = 5.0 Hz, 1H), 9.10 (s, 1H)

¹³C NMR (CDCl₃, 75 MHz): 45.8, 49.1, 55.4, 114.4, 118.2, 128.6, 128.9, 129.7, 146.2, 149.7, 151.4, 159.4, 166.7

IC(+)MS (MH⁺) *m/z*= 282, 255, 179, 158, 113, 73

Anal. *calcd* for C₁₅H₁₄N₂O₂: C, 70.85; H, 5.55; N, 11.02 *found* C, 70.69; H, 5.64; N, 11.24

2-(4-methoxybenzyl)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one 4b
Mol. Formula : C ₁₅ H ₁₄ N ₂ O ₂ Mol. Weight : 254.28
<p>To a stirred solution of n-butyllithium (3.67 mL, 9.19 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (1.61 mL, 9.46 mmol) at –10 °C. The solution was allowed to stir at 0 °C for 30 minutes then cooled to –78 °C. Piperidine isonicotinamide 12 (500 mg, 2.63 mmol) in anhydrous THF (10 mL) was added in 5 minutes immediately followed by dropwise addition of 2-(4-methoxybenzylamino)acetonitrile 2 (0.46 mL, 2.89 mmol) in anhydrous THF (7 mL) and kept 7 hours at this temperature. The reaction was quenched with aqueous ammonium chloride (530 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated <i>in vacuo</i>. The product was purified by flash chromatography through SiO₂ (ethyl acetate : isopropanol 9 : 1 R_f = 0.30) providing (348 mg, 52 %) as a white solid.</p>	
¹ H NMR (CDCl ₃ , 300 MHz): 3.79 (s, 3H), 4.34 (s, 2H), 4.76 (s, 2H), 6.87 (d, <i>J</i> = 8.5, 2H), 7.24 (d, <i>J</i> = 8.5, 2H), 7.77 (d, <i>J</i> = 4.9 Hz, 1H), 8.76 (s, 1H), 8.76 (d, <i>J</i> = 4.9 Hz, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 46.2, 48.1, 55.5, 114.4, 117.9, 128.5, 129.8, 135.8, 140.5, 145.1, 149.5, 159.5, 166.7	
IC(+)MS (MH ⁺) <i>m/z</i> = 255	
Anal. <i>calcd</i> for C ₁₅ H ₁₄ N ₂ O ₂ : C, 70.85; H, 5.55; N, 11.02 <i>found</i> C, 70.63; H, 5.61; N, 11.19

cyclisation piperidineisonicotinamide

6-(4-methoxybenzyl)-5*H*-pyrrolo[3,4-*b*]pyridin-7(6*H*)-one **4c**

Mol. Formula : C₁₅H₁₄N₂O₂

Mol. Weight : 254.28

To a stirred solution of *n*-butyllithium (3.93 mL, 9.82 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (1.71 mL, 10.09 mmol) at –10 °C. The solution was allowed to stir at 0 °C for 30 minutes then cooled to –78 °C. *N,N'*-diethylpicolinamide (500 mg, 2.80 mmol) in anhydrous THF (10 mL) was added in 5 minutes followed immediately by dropwise addition of 2-(4-methoxybenzylamino)acetonitrile **2** (0.49 mL, 3.08 mmol) in anhydrous THF (7 mL) and the reaction mixture was allowed to reach –10 °C in 16 hours. The reaction was quenched with aqueous ammonium chloride (530 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated *in vacuo*. The product was purified by flash chromatography through SiO₂ (ethyl acetate : isopropanol 5 : 1 *R_f* = 0.28) providing **4c** (376 mg, 53 %) as a white solid.

¹H NMR (CDCl₃, 300 MHz): 3.77 (s, 3H), 4.23 (s, 2H), 4.79 (s, 2H), 6.84 (d, *J* = 8.2, 2H), 7.23 (d, *J* = 8.2, 2H), 7.38 (dd, *J* = 4.9 Hz, 7.7 Hz, 1H), 7.72 (d, *J* = 7.7 Hz, 1H), 8.77 (d, *J* = 4.9 Hz, 1H)

¹³C NMR (CDCl₃, 75 MHz): 46.3, 47.0, 55.4, 114.3, 125.2, 128.6, 129.8, 131.2, 135.1, 150.9, 159.4, 166.4

IC(+)MS (MH⁺) *m/z* = 255

Anal. *calcd* for C₁₅H₁₄N₂O₂: C, 70.85; H, 5.55; N, 11.02 *found* C, 70.83; H, 5.61; N, 11.09

2-allyl-1*H*-pyrrolo[3,4-*c*]pyridin-3(2*H*)-one **16**

Mol. Formula : C₁₀H₁₀N₂O

Mol. Weight : 174.20

To a stirred solution of *n*-butyllithium (3.67 mL, 9.19 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (1.61 mL, 9.46 mmol) at -10°C . The solution was allowed to stir at 0°C for 30 minutes then cooled to -78°C . Piperidine nicotinamide **8** (500 mg, 2.63 mmol) in anhydrous THF (10 mL) was added in 5 minutes immediately followed by dropwise addition of 2-(allylamino)acetonitrile **14** (274 mg, 2.89 mmol) in anhydrous THF (7 mL) and kept 7 hours at this temperature. The reaction was quenched with aqueous ammonium chloride (530 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated *in vacuo*. The product was purified by flash chromatography through SiO₂ (ethyl acetate : isopropanol 10 : 1 *R_f* = 0.23) providing **16** (307 mg, 67%) as a yellow oil.

¹H NMR (CDCl₃, 300 MHz): 4.22 (d, *J* = 6 Hz, 2H), 4.39 (s, 1H), 5.23 (dd, *J* = 1.2 Hz, 5.8 Hz, 1H), 5.27 (s, 1H), 5.83 (m, 1H), 7.42 (d, *J* = 5.09, 1H), 8.73 (d, *J* = 5 Hz, 1H), 9.09 (s, 1H)

¹³C NMR (CDCl₃, 75 MHz): 45.1, 49.4, 118.7, 118.7, 128.9, 132.6, 146.2, 146.8, 151.5, 166.6

IC(+)MS (MH⁺) *m/z* = 175

Anal. *calcd* for C₁₀H₁₀N₂O: C, 68.95; H, 5.79; N, 16.08 *found* C, 68.74; H, 5.89; N, 16.17

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2-cyclohexyl-1*H*-pyrrolo[3,4-*c*]pyridin-3(2*H*)-one **17**

Mol. Formula : C₁₃H₁₆N₂O

Mol. Weight : 216.27

To a stirred solution of *n*-butyllithium (3.67 mL, 9.19 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (1.61 mL, 9.46 mmol) at –10 °C. The solution was allowed to stir at 0 °C for 30 minutes then cooled to –78 °C. Piperidine nicotinamide (500 mg, 2.63 mmol) in anhydrous THF (10 mL) was added in 5 minutes immediately followed by dropwise addition of 2-(cyclohexylamino)acetonitrile **15** (399 mg, 2.89 mmol) in anhydrous THF (7 mL) and kept 7 hours at this temperature. The reaction was quenched with aqueous ammonium chloride (530 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated *in vacuo*. The product was purified by flash chromatography through SiO₂ (ethyl acetate : isopropanol : triethylamine 10 : 1 : 0.01 R_f = 0.51) providing **17** (397 mg, 70%) as a white solid.

¹H NMR (CDCl₃, 300 MHz): 1.14-1.23 (m, 1H), 1.39-1.51 (m, 4H), 1.71-1.75 (m, 1H), 1.85 (m, 4H), 4.27 (m, 1H), 4.38 (s, 2H), 7.42 (d, *J* = 4.8 Hz, 1H), 8.72 (d, *J* = 4.8 Hz, 1H), 9.08 (s, 1H)

¹³C NMR (CDCl₃, 75 MHz): 25.5, 25.6, 31.5, 46.0, 50.7, 118.2, 129.5, 146.1, 149.8, 151.1, 166.2

IC(+)- MS (MH⁺) *m/z* = 217

Anal. *calcd* for C₁₃H₁₆N₂O: C, 72.19; H, 7.46; N, 12.95 *found* C, 72.07; H, 7.56; N, 13.04

cyclisation nicotinamide + cyano cyclohexyl

2-(4-methoxybenzyl)-1,1-dimethyl-1*H*-pyrrolo[3,4-*c*]pyridin-3(2*H*)-one **18** Mol. Formula : C₁₇H₁₈N₂O₂

Mol. Weight : 282.34

To a stirred solution of n-butyllithium (3.67 mL, 9.19 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (1.61 mL, 9.46 mmol) at -10°C . The solution was allowed to stir at 0°C for 30 minutes then cooled to -78°C . Piperidine nicotinamide **8** (500 mg, 2.63 mmol) in anhydrous THF (10 mL) was added in 5 minutes followed immediately by dropwise addition of 2-(4-methoxybenzylamino)acetonitrile **2** (0.46 mL, 2.89 mmol) in anhydrous THF (7 mL) and kept 7 hours at this temperature. Iodomethane (0.36 mL, 5.79 mmol) in anhydrous THF (3 mL) was added at -30°C and the reaction mixture and was allowed to reach -10°C in 2 hours. The reaction was quenched with aqueous ammonium chloride (530 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated *in vacuo*. The product was purified by flash chromatography through SiO₂ (ethyl acetate : isopropanol 3 : 1 R_f = 0.66) providing **18** (237 mg, 32%) as a colorless oil.

¹H NMR (CDCl₃, 300 MHz): 1.37 (s, 6H), 3.77 (s, 3H), 4.68 (s, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 7.28 (d, *J* = 8.7 Hz, 2H), 7.33 (d, *J* = 5.0 Hz, 1H), 8.75 (d, *J* = 5.0 Hz, 1H), 9.12 (s, 1H)

¹³C NMR (CDCl₃, 75 MHz): 21.8, 25.9, 42.2, 55.4, 63.4, 114.1, 116.1, 129.3, 130.3, 146.3, 151.9, 159.1, 160.1, 166.2

IC(+)MS (MH⁺) *m/z* = 283

Anal. *calcd* for C₁₇H₁₈N₂O₂: C, 72.32; H, 6.43; N, 9.92 *found* C, 72.21; H, 6.51; N, 10.04

1,1-diallyl-2-(4-methoxybenzyl)-1*H*-pyrrolo[3,4-*c*]pyridin-3(2*H*)-one **19**

Mol. Formula : C₂₁H₂₂N₂O₂

Mol. Weight : 334.41

To a stirred solution of *n*-butyllithium (3.67 mL, 9.19 mmol) in anhydrous THF (35 mL) was added dropwise 2,2,6,6-tetramethylpiperidine (1.61 mL, 9.46 mmol) at –10 °C. The solution was allowed to stir at 0 °C for 30 minutes then cooled to –78 °C. Piperidine nicotinamide **8** (500 mg, 2.63 mmol) in anhydrous THF (10 mL) was added in 5 minutes immediately followed by dropwise addition of 2-(4-methoxybenzylamino)acetonitrile **2** (0.46 mL, 2.89 mmol) in anhydrous THF (7 mL) and kept 7 hours at this temperature. Allyl iodide (0.53 mL, 5.79 mmol) in anhydrous THF (3 mL) was added to the reaction mixture at –30 °C and was allowed to reach –10 °C in 2 hours. The reaction was quenched with aqueous ammonium chloride (530 mg in 10 mL of water) and extracted with diethyl ether (3 x 40 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated *in vacuo*. The product was purified by flash chromatography through SiO₂ (ethyl acetate : cyclohexane 1 : 1 *R_f* = 0.37) providing **19** (273 mg, 31 %) as a white solid.

¹H NMR (CDCl₃, 300 MHz): 2.60 (m, 4H), 3.71 (s, 3H), 4.61 (s, 2H), 4.61-4.88 (m, 4H), 4.90-4.99 (m, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 7.27 (d, *J* = 5.1 Hz, 1H), 7.38 (d, *J* = 8.7 Hz, 2H), 8.72 (d, *J* = 5.1 Hz, 1H), 9.08 (s, 1H)

¹³C NMR (CDCl₃, 75 MHz): 41.1, 42.9, 55.3, 69.4, 114.0, 116.9, 120.2, 128.2, 129.9, 130.1, 130.2, 146.1, 151.7, 155.9, 159.2, 167.5

IC(+)MS (MH⁺) *m/z*= 335

Anal. *calcd* for C₂₁H₂₂N₂O₂: C, 75.42; H, 6.63; N, 8.38 *found* 75.35; H, 6.69; N, 8.42

Section 2: Preparation of nicotinamides derivatives and cyanomethylamines derivatives.

Standard procedure for the preparation of nicotinamide derivatives:

Nicotinic acid (4 g, 32.5 mmol) was solubilised in thionyl chloride (30 mL) then refluxed for 2 hours. The solution was concentrated and the residue was taken up in anhydrous dichloromethane (50 mL). To this solution was added dropwise a solution of the secondary amine (129.9 mmol) in anhydrous CH₂Cl₂ (15 mL) at 0 °C and stirred overnight. The reaction mixture was washed with sodium hydroxide (4M NaOH, 3 x 30 mL). The organic layer was dried over MgSO₄ then filtered and concentrated *in vacuo* to provide the amide which was purified as described below.

<i>N,N</i>-diisopropylnicotinamide 1
Mol. Formula : C ₁₂ H ₁₈ N ₂ O Mol. Weight : 206.28
Brown solid was triturated in cyclohexane and filtrated to give a pale brown solid (5.97 g, 89% yield).	
¹ H NMR (CDCl ₃ , 300 MHz): 1.24 (m, 12H), 3.62 (m, 2H), 7.23 (dd, <i>J</i> = 4.9 Hz, 7.7 Hz, 1H), 7.55 (m, 1H), 8.52 (m, 2H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 20.9, 123.6, 133.7, 134.6, 146.8, 150.0, 168.4	
ES(+)MS (MH ⁺) <i>m/z</i> = 207.27	
Anal. <i>calcd</i> for C ₁₂ H ₁₈ N ₂ O: C, 69.87; H, 8.80; N, 13.58 <i>found</i> C, 69.75; H, 8.91; N, 13.62

<i>N,N</i> -dibenzylnicotinamide 6
Mol. Formula : C ₂₀ H ₁₈ N ₂ O Mol. Weight : 302.37
Brown solid was triturated in cyclohexane and filtrated to give a pale brown solid (6.80 g, 69% yield).	
¹ H NMR (CDCl ₃ , 300 MHz): 4.43 (s, 2H), 4.75 (s, 2H), 7.16 (m, 2H), 7.37 (m, 9H), 7.82(d, <i>J</i> = 7.7 Hz, 1H), 8.66 (d, <i>J</i> = 5Hz, 1H), 8.78 (s, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 47.5, 51.7, 123.5, 126.9, 127.9, 128.6, 128.9, 129.2, 132.1, 134.6, 147.8, 150.9, 169.8	
ES(+)MS (MH ⁺) <i>m/z</i> = 303.40	
Anal. <i>calcd</i> for C ₂₀ H ₁₈ N ₂ O : C, 79.44; H, 6.00; N, 9.26 <i>found</i> C, 79.69 ; H, 6.02 ; N, 9.58	

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bn

<i>N,N</i> -diphenylnicotinamide 7
Mol. Formula : C ₁₈ H ₁₄ N ₂ O Mol. Weight : 274.32
Brown solid was triturated in cyclohexane and filtrated to give a pale brown solid (5.79 g, 65% yield).	
¹ H NMR (CDCl ₃ , 300 MHz): 7.42-7.58 (m, 11H), 7.99 (m, 1H), 8.02 (m, 1H), 8.88 (s, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 122.9, 127.0, 127.6, 129.5, 132.2, 136.6, 143.3, 150.1, 150.9, 168.3	
ES(+)MS (MH ⁺) m/z= 275.33	
Anal. <i>calcd</i> for C ₁₈ H ₁₄ N ₂ O : C, 78.81; H, 5.14; N, 10.21 <i>found</i> C, 78.62 ; H, 4.97 ; N, 9.93

Piperidine nicotinamide 8
Mol. Formula : C ₁₁ H ₁₄ N ₂ O Mol. Weight : 190.24
Product was purified by bulb-to-bulb distillation (160 °C, 0.2 mbar) to give 8 as a yellow oil (6.78 g, 89%).	
¹ H NMR (CDCl ₃ , 300 MHz): 1.52 (m, 2H), 1.67 (m, 4H), 3.3 (m, 2H), 3.69 (m, 2H), 7.32 (m, 1H), 7.70 (td, <i>J</i> = 1.9 Hz, 7.9 Hz, 1H), 8.62 (m, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 24.5, 25.6, 26.7, 43.3, 48.9, 123.5, 132.3, 134.9, 147.8, 150.6, 167.7	
ES(+)MS (MH ⁺) <i>m/z</i> = 191.20	
Anal. <i>calcd</i> for C ₁₁ H ₁₄ N ₂ O : C, 69.45; H, 7.42; N, 14.73 <i>found</i> C, 69.39; H, 7.34; N, 14.57

Pyrrolidine nicotinamide 9
Mol. Formula : C ₁₀ H ₁₂ N ₂ O Mol. Weight : 176.22
Product was purified by bulb-to-bulb distillation (140 °C, 0.2 mbar) to give a yellow oil (3.44 g, 60%).	
¹ H NMR (CDCl ₃ , 300 MHz): 1.85 (m, 4H), 3.36 (t, <i>J</i> = 6.0 Hz, 2H), 3.58 (t, <i>J</i> = 6.0 Hz, 2H), 7.27 (m, 1H), 7.77 (dd, <i>J</i> = 2Hz , 8 Hz, 1H), 8.56 (m, 1H), 8.69 (s, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 24.2, 26.2, 46.2, 49.4, 123.2, 132.7, 134.8, 147.9, 150.6, 166.9	
ES(+)MS (MH ⁺) <i>m/z</i> = 177.20	
Anal. <i>calcd</i> for C ₁₀ H ₁₂ N ₂ O : C, 68.16; H, 6.86; N, 15.90 <i>found</i> C, 68.01; H, 6.73; N, 15.72

<i>N,N</i> -diallylnicotinamide 10
Mol. Formula : C ₁₂ H ₁₄ N ₂ O Mol. Weight : 202.25
Product was purified by bulb-to-bulb distillation (150 °C, 0.2 mbar) to give a colorless oil (3.75 g, 57%).	
¹ H NMR (CDCl ₃ , 300 MHz): 3.77 (bs, 2H), 4.07 (bs, 2H), 5.16 (m, 4H), 5.76 (m, 2H), 7.26 (m, 1H), 7.69 (d, <i>J</i> = 6.0 Hz, 1H), 8.57(m, 1H), 8.62 (s, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 47.2, 50.6, 117.8, 118.0, 123.1, 131.9, 132.2, 132.6, 134.3, 147.4, 150.6, 169.0	
ES(+)MS (MH ⁺) <i>m/z</i> = 203.20	
Anal. <i>calcd</i> for C ₁₂ H ₁₄ N ₂ O : C, 71.26; H, 6.98; N, 13.85 <i>found</i> C, 71.12; H, 7.09; N, 14.01

Morpholine nicotinamide 11
Mol. Formula : C ₁₀ H ₁₂ N ₂ O ₂ Mol. Weight : 192.21
Product was purified by bulb-to-bulb distillation (150 °C, 0.2 mbar) to give a yellow oil (5.19 g, 83%).	
¹ H NMR (CDCl ₃ , 300 MHz): 3.46-3.76 (m, 8H), 7.38 (m, 1H), 7.77 (m, 1H), 8.67 (m, 2H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 42.6, 48.2, 66.74, 123.5, 131.1, 135.1, 147.9, 150.9, 167.7	
ES(+)MS (MH ⁺) m/z= 193.33	
Anal. <i>calcd</i> for C ₁₀ H ₁₂ N ₂ O ₂ : C, 62.49; H, 6.29; N, 14.57 <i>found</i> C, 62.25; H, 6.37; N, 14.66

morpholine nico

Piperidine isonicotinamide 12
Mol. Formula : C ₁₁ H ₁₄ N ₂ O Mol. Weight : 190.24
Product was purified by bulb-to-bulb distillation (140 °C, 0.2 mbar) to give a yellow oil (4.64 g, 75%).	
¹ H NMR (CDCl ₃ , 300 MHz): 1.43 (m, 2H), 1.60 (m, 4H), 3.19 (t, <i>J</i> = 5.3 Hz, 2H), 3.62 (m, 2H), 7.22 (d, <i>J</i> = 4.7 Hz, 2H), 8.60 (d, <i>J</i> = 4.7 Hz, 2H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 24.2, 25.3, 26.3, 42.8, 48.3, 121.0, 144.2, 149.8, 167.3	
ES(+)MS (MH ⁺) <i>m/z</i> = 191.40	
Anal. <i>calcd</i> for C ₁₁ H ₁₄ N ₂ O : C, 69.45; H, 7.42; N, 14.73 <i>found</i> C, 69.31; H, 7.47; N, 14.79

<i>N,N</i> -diethylpicolinamide 13
Mol. Formula : C ₁₀ H ₁₄ N ₂ O Mol. Weight : 178.23
Product was purified by bulb-to-bulb distillation (100 °C, 0.2 mbar) to give a colorless oil (5.10 g, 88 %).	
¹ H NMR (CDCl ₃ , 300 MHz): 1.14 (t, <i>J</i> = 7.2 Hz, 3H), 1.25 (t, <i>J</i> = 7.2 Hz, 3H), 3.36 (q, <i>J</i> = 7.16 Hz, 2H), 3.57 (q, <i>J</i> = 7.2 Hz, 2H), 7.30 (ddd, <i>J</i> = 1.3 Hz, 4.9 Hz, 17.7 Hz, 1H), 7.55 (m, 1H), 7.79 (td, <i>J</i> = 1.3 Hz, 7.7 Hz, 1H), 8.57 (m, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 12.2, 13.6, 39.4, 42.5, 122.2, 123.4, 136.2, 147.7, 154.5, 167.8	
ES(+)MS (MH ⁺) <i>m/z</i> = 179.27	
Anal. <i>calcd</i> for C ₁₀ H ₁₄ N ₂ O: C, 67.39; H, 7.92; N, 15.72 <i>found</i> C, 67.31; H, 7.96; N, 15.80

2-(4-methoxybenzylamino)acetonitrile 2
Mol. Formula : C ₁₀ H ₁₂ N ₂ O
	Mol. Weight : 176.21
	d:1.1 (20 °C)
<p>To a stirred solution of <i>p</i>-methoxybenzylamine (10 mL, 76.5 mmol) in acetonitrile (100 mL), were added chloroacetonitrile (5.35 mL, 84.2 mmol), and potassium carbonate (21.2 g, 153 mmol). The reaction mixture was refluxed overnight. After filtration through celite, solvents were evaporated to give the desired compound. The product was purified by bulb-to-bulb distillation (125 °C, 0.2 mbar) to give compound 2 as a colorless oil (11.3 g, 84%).</p>	
¹ H NMR (CDCl ₃ , 300 MHz): 1.80 (bs, 1H), 3.67 (s, 2H), 3.94 (s, 3H), 3.99 (s, 2H), 7.01 (d, <i>J</i> = 8.2 Hz, 2H), 7.41 (d, <i>J</i> = 8.2 Hz, 2H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 36.1, 51.7, 55.3, 114.0, 117.7, 129.7, 129.9, 159.1	
ES(+)MS (MH ⁺) <i>m/z</i> = no MS. (degradation occurred)	
Anal. <i>calcd</i> for C ₁₀ H ₁₂ N ₂ O: C, 68.16; H, 6.86; N, 15.90 <i>found</i> C, 67.98; H, 6.97; N, 16.03

2-(allylamino)acetonitrile 14
Mol. Formula : C ₅ H ₈ N ₂ Mol. Weight : 96.13
<p>To a stirred solution of acetonitrile (100 mL), was added allylamine (5.72 mL, 76.5 mmol), chloroacetonitrile (5.35 mL, 84.2 mmol), and potassium carbonate (21.2g, 153 mmol). The reaction mixture was heated at 80 °C overnight. After filtration through celite, solvents were evaporated to give the desired compound. The product was purified by bulb-to-bulb distillation (130 °C, 0.2 mbar) to give compound 14 as a colorless liquid (4.79 g, 65%)</p>	
¹ H NMR (CDCl ₃ , 300 MHz): 1.49 (bs, 1H), 3.30 (m, 2H), 3.51 (m, 2H), 5.17 (m, 2H), 5.77 (m, 1H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 36.1, 50.7, 117.7, 117.8, 134.5	
ES(+)MS (MH ⁺) m/z= no MS. degradation occurred	
Anal. <i>calcd</i> for C ₅ H ₈ N ₂ : C, 62.47; H, 8.39; N, 29.14 <i>found</i> C, 62.33; H, 8.46; N, 29.23

2-(cyclohexylamino)acetonitrile 15
Mol. Formula : C ₈ H ₁₄ N ₂ Mol. Weight : 138.21
<p>To a stirred solution of acetonitrile (100 mL), was added cyclohexylamine (8.72 mL, 76.5 mmol), chloroacetonitrile (5.35 mL, 84.2 mmol), and potassium carbonate (21.2 g, 153 mmol). The reaction mixture was heated at 80 °C overnight. After filtration through celite, solvents were evaporated to give the desired compound. The product was purified by bulb-to-bulb distillation (135 °C, 0.2 mbar) to give compound 15 as a white solid (8.89 g, 84%)</p>	
¹ H NMR (CDCl ₃ , 300 MHz): 1.00-1.36 (m, 6H), 1.59 (m, 1H), 1.60-1.85 (m, 4H), 2.65 (m, 1H), 3.61 (s, 2H)	
¹³ C NMR (CDCl ₃ , 75 MHz): 24.5, 25.9, 32.6, 34.4, 55.2, 118.2	
ES(+)MS (MH ⁺) m/z= no MS. degradation occurred	
Anal. <i>calcd</i> for C ₈ H ₁₄ N ₂ : C, 69.52; H, 10.21; N, 20.27 <i>found</i> C, 69.41; H, 10.35; N, 20.40