Iodide-catalyzed Amide Synthesis from Alcohols and

Amines

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I. General Methods

All reagents were purchased from commercial suppliers and used without further purification. ¹H-NMR, ¹³C-NMR spectra were measured on a Bruker AM 400 NMR spectrometer (400 MHz or 100MHz, respectively) with CDCl₃ as solvent and recorded in ppm relative to internal tetramethylsilane standard. The following abbreviations were used to describe splitting patterns: s = singlet, d = doublet, t = triplet, q = quartet. Coupling constants J are given in Hz. Mass spectra were recorded on a Waters Q-TOF Premier instrument by using ESI method or Agilent 6890-5973N GCMS-EI instrument. Thin layer chromatography was performed on fluorescence indicator marked precoated silca gel 60 plates and visualization was achieved by UV light (254 nm). Flash chromatography was performed on silica gel (0.040-0.063mm). Unless otherwise stated, all commercially available substances and reagents were used as received from their suppliers

General procedure for NHS active ester. (Table 2)

Alcohol 1 (0.5 mmol) and N-hydroxysuccinimide 2a (0.75 mmol) were added to a tube (10 mL) with a magnetic stirring bar and solvent (CH₃CN, 2 mL). To this mixture 10 mol% of nBu_4NI (0.05 mmol) and anhydrous TBHP (4.0 equiv) were added at room temperature. After stirring at 80 °C for 18h, 10 mL water was added and the mixture was extracted with EtOAc (3×15 mL). The combined organic phase was wished by saturated solution of NaCl (1×10 mL) and dried with Na₂SO₄ and evaporated in vacuum. The residue was purified by flash silica gel column chromatography using a mixture of petroleum ether and ethyl acetate (8:1 to 4:1) as eluent to afford the desired products.

General procedure for NHPI active ester. (Table 3)

Alcohol 1 (0.5 mmol) and N-hydroxyphthalimide **2b** (0.75 mmol) were added to a tube (10 mL) with a magnetic stirring bar and solvent (CH₃CN, 2 mL). To this mixture 10 mol% of NaI (0.05 mmol), 0.2mmol KOH and aqueous TBHP (4.0 equiv) were added at room temperature. After stirring at 80 °C for 8h, the solvent was removed under vacuum and the residue was purified by flash silica gel column chromatography using a mixture of petroleum ether and ethyl acetate (7:1) as eluent to afford the desired products.

General procedure for amide. (Table 5)

Alcohol 1 (0.5 mmol) and N-hydroxysuccinimide 2a (0.75 mmol) were added to a tube (10 mL) with a magnetic stirring bar and solvent (CH₃CN, 2 mL). To this mixture 10 mol% of nBu_4NI (0.05 mmol) and anhydrous TBHP (4.0 equiv) were added at room temperature. The mixture stir at 80 °C for 18h. After the mixture was cooled to room temperature, amine (0.75 mmol) was added to the mixture in one portion and the mixture was stirred at room temperature for 12 h. 10 mL water was added and the mixture was extracted with EtOAc (3×15 mL). The combined organic phase was wished by saturated solution of NaCl (1×10 mL) and dried with Na₂SO₄ and evaporated in vacuum. The residue was purified by flash silica gel column chromatography using a mixture of petroleum ether and ethyl acetate (10:1) as eluent to afford the desired products.

II. Added Experimental Date

OH 1a	+ HO-N O 2b	catalyst T-HYDRO additive 80 °C		O O O-N 3ab
Entry	n(1a): n(2a)	Catalyst	additive	Yield(%)
1	1.0 : 0.5	NaI	NaOH	65%
2	1.0 : 0.5	KI	NaOH	58%
3	1.0 : 0.5	I_2	NaOH	65%
4	1.0 : 0.5	<i>n</i> Bu ₄ NI	NaOH	61%
5	1.0 : 0.5	Et ₄ NI	NaOH	57%
6	1.0 : 0.5	PhI(OAc) ₂	NaOH	0%
7	1.0 : 0.5	-	NaOH	0%
8	0.5 : 1.0	NaI	Cs_2CO_3	71%
9	0.5 : 1.0	NaI	K_3PO_4	78%
10	0.5 : 1.0	NaI	КОН	95%
11	0.5 : 1.0	NaI	K_2CO_3	87%
12 ^[b]	0.5 : 0.75	NaI	КОН	34%
13 ^[c]	0.5:0.75	NaI	КОН	93%

Table S1 Optimization of the conditions for the esterification of NHPI.^[a]

^[a] Unless otherwise specified, all reactions were carried out using benzyl alcohol and **NHPI** in ethyl acetate (2.0 mL) with 10 mol% of catalyst and **aqueous TBHP** (4.0 equiv) at 80°C for 18 hours. ^[b] 4 hours. ^[c] 8 hours

III. Experimental Data of Products (Table 2, Table 3 and Table 5)

Table 2:

2,5-dioxopyrrolidin-1-yl benzoate



(**3aa**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.15 (d, J = 7.2 Hz, 2H), 7.69 (t, J = 7.5 Hz, 1H), 7.52 (t, J = 7.9 Hz, 2H), 2.91 (s, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 169.3, 161.9, 135.0, 130.6, 128.9, 125.1, 25.7.

MS (ESI) $m/z 242.01 [(M+Na)^+]$

2,5-dioxopyrrolidin-1-yl 4-methylbenzoate



(**3ba**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 2.91(s, 4H), 2.45(s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 169.4, 161.9, 146.1, 130.6, 129.6, 122.3, 25.7, 21.9. MS (ESI) m/z 256.06 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 4-methoxybenzoate



(3ca) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, J = 9.0 Hz, 2H), 6.98 (d, J = 9.0 Hz, 2H), 3.89 (s, 3H), 2.90 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 169.6, 164.9, 161.5, 132.9, 117.0, 114.2, 55.6, 25.7. MS (ESI) m/z 272.06 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 4-fluorobenzoate



(3da) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.17 (m, 2H), 7.20 (t, J = 8.4 Hz, 2H), 2.92 (s, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 169.3, 168.1, 165.6, 160.9, 133.4, 121.4, 116.3, 25.7. MS (ESI) m/z 260.04 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 4-chlorobenzoate



(**3ea**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, J = 8.6 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 2.92 (s, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 169.2, 161.1, 141.7, 131.9, 129.4, 123.5, 25.7.

MS (ESI) m/z 276.04 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 4-bromobenzoate



(**3fa**) White solid. ¹H NMR (400 MHz, CDCl₃) δ 8.00 (d, J = 8.6 Hz, 2H), 7.67 (d, J = 8.6 Hz, 2H), 2.92 (s, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 169.2, 161.3, 132.3, 131.9, 130.5, 124.0, 25.7. MS (ESI) m/z 319.77 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 3-chlorobenzoate



(**3ga**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.12 (s, 1H), 8.03 (d, J = 8.0 Hz, 1H), 7.65 (m, 1H), 7.47 (t, J = 8.0 Hz, 1H), 2.92 (s, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 169.1, 160.9, 135.1, 135.0, 130.5, 130.3, 128.7, 126.8, 25.7. MS (ESI) m/z 276.02 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 2-methylbenzoate



(**3ha**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.12 (d, J = 7,3 Hz, 1H), 7.52 (t, J=6.7 Hz, 1H), 7.32 (t, J=7.6 Hz, 2H), 2.91 (s, 4H), 2.62 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 169.5, 162.1, 142.1, 134.0, 132.0, 131.3, 126.1, 124.2, 25.7, 21.6. MS (ESI) m/z 256.10 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 2-chlorobenzoate



(**3ia**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.12 (d, J = 8.2 Hz, 1H), 7.57 (m, 2H), 7.40 (m, 1H), 2.92 (s, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 169.1, 160.2, 135.6, 134.7, 132.4, 131.7, 126.9, 124.5, 25.7. MS (ESI) m/z 276.02 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 1-naphthoate



(3ja) white solid

¹H NMR (400 MHz, CDCl₃) δ 8.81 (d, J = 8.6 Hz, 1H), 8.46 (d, J = 7,3 Hz, 1H), 8.15 (d, J = 8.0 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.66 (t, J = 7.2 Hz, 1H), 7.58 (t, J = 8.0 Hz, 2H), 2.95 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 169.6, 162.2, 135.6, 133.7, 131.9, 131.4, 128.8, 128.7, 126.9,125.2, 124.5, 121.5, 25.8. MS (ESI) m/z 292.08 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl 4-cyanobenzoate



(3ka) white solid

¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, J = 8.4 Hz, 2H), 7.83 (d, J = 8.4 Hz, 2H), 2.94 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 168.7, 160.5, 132.5, 130.9, 129.0, 126.9, 118.2, 117.3, 105.4, 25.7 MS (ESI) m/z 267.06 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl thiophene-2-carboxylate



(3la) yellow solid

¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, J = 3.7 Hz, 1H), 7.78 (d, J = 4.8 Hz, 1H), 7.21 (t, J = 4.3 Hz, 1H), 2.91 (s, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 169.2, 157.4, 136.7, 135.8, 128.4, 126.9, 25.7. MS (ESI) m/z 248.04 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl furan-2-carboxylate

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(**3ma**) yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.74 (d, J = 0.8 Hz, 1H), 7.50 (d, J = 3.6 Hz, 1H), 6.63 (q, J = 1.6 Hz, 1H), 2.91 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 169.2, 153.6, 148.9, 139.6, 122.3, 112.7, 25.6. MS (ESI) m/z 232.04 [(M+Na)⁺]

2,5-dioxopyrrolidin-1-yl cinnamate



(**3na**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, J = 16.0 Hz, 1H), 7.58 (dd, J = 7.8, 1.5 Hz, 2H), 7.43 (m, 3H), 6.60 (d, J = 16.0 Hz, 1H), 2.89 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 169.5, 162.1, 150.1, 133.4, 131.6, 129.1, 128.7, 111.5, 25.7. MS (ESI) m/z 268.08 [(M+Na)⁺]

Table 3:1,3-dioxoisoindolin-2-yl benzoate



(**3ab**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, J = 7.7 Hz, 2H), 7.93 (dd, J = 5.2, 3.4 Hz, 2H), 7.82 (dd, J = 5.2, 3.4 Hz, 2H), 7.71 (t, J = 7.4 Hz, 1H), 7.55 (t, J = 7.7, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 162.8, 162.1, 134.9, 134.8, 130.7, 128.9, 128.2, 125.3, 124.1. MS (ESI) m/z 290.01 [(M+Na)⁺]

1,3-dioxoisoindolin-2-yl 4-methylbenzoate



(**3bb**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.08(d, J = 7.2 Hz, 2H), 7.93 (t, J = 2.4 Hz, 2H), 7.81 (t, J = 2.4 Hz, 2H), 7.33 (d, J = 7.6 Hz, 2H), 2.47(s,3H).

¹³C NMR (100 MHz, CDCl₃) δ 162.8, 162.2, 146.1, 134.8, 130.7, 129.6, 129.0, 124.0, 122.4, 21.9.

MS (ESI) m/z 304.07 [(M+Na)⁺]

1,3-dioxoisoindolin-2-yl 4-fluorobenzoate



(3db) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.23(m, 2H), 7.93(d, J=2.9Hz, 2H), 7.83(d, J=1.7Hz, 2H), 7.23(t, J=8.6Hz, H=2).

¹³C NMR (100 MHz, CDCl₃) δ 168.2, 165.6, 162.0, 161.9, 134.9, 133.5, 133.4, 128.9, 124.1, 116.4, 116.2.

MS (ESI) m/z 308.06 $[(M+Na)^{+}]$

1,3-dioxoisoindolin-2-yl 4-chlorobenzoate



(**3eb**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.14 (d, J = 8.0 Hz, 2H), 7.93 (dd, J = 4.9, 3.1 Hz, 2H), 7.82 (dd, J = 4.9, 3.1 Hz, 2H), 7.53 (d, J = 8.4 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 162.1, 162.0, 141.7, 134.9, 132.0, 129.4, 128.9, 124.1, 123.7.

MS (ESI) m/z 324.01 [(M+Na)⁺].

1,3-dioxoisoindolin-2-yl 4-bromobenzoate



(**3fb**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.06(d, J = 8.3 Hz, 2H), 7.93 (dd, J = 4.8, 3.1 Hz, 2H), 7.82 (dd, J = 4.9, 3.1 Hz, 2H), 7.69 (d, J = 8.3 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 162.3, 162.0, 135.0, 132.4, 132.0, 130.5, 128.9, 124.2, 124.1. MS (ESI) m/z 367.94 [(M+Na)⁺]

1,3-dioxoisoindolin-2-yl 2-methylbenzoate



(**3hb**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.21(d, J = 7.4 Hz, 1H), 7.93 (dd, J = 5.2, 3.0 Hz, 2H), 7.82 (dd, J = 5.2, 3.0 Hz, 2H), 7.55 (t, J=7.1, 1H), 7.35 (t, J=6.4, 2H), 2.65 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 171.2, 163.1, 162.3, 142.2, 134.8, 134.0, 132.0, 131.4, 129.0, 126.1, 124.0, 21.7.

MS (ESI) m/z 304.07 $[(M+Na)^{+}]$

1,3-dioxoisoindolin-2-yl 2-chlorobenzoate



(**3ib**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.19(d, J = 7.8 Hz, 1H), 7.94 (dd, J = 5, 3.2 Hz, 2H), 7.83 (dd, J = 5, 3.2 Hz, 2H), 7.57 (s, 2H), 7.44 (m, 1H).

¹³C NMR (100 MHz, CDCl₃) δ 161.9, 161.2, 135.6, 134.9, 134.7, 132.5, 131.7, 129.0, 126.9,124.7,124.1.

MS (ESI) m/z 324.01 [(M+Na)⁺]

1,3-dioxoisoindolin-2-yl 1-naphthoate



(**3jb**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.84 (d, J =8.5 Hz, 1H), 8.54 (d, J =7.2 Hz, 1H), 8.16 (d, J =8.1 Hz, 1H), 7.93 (m, 3H), 7.81 (dd, J =5.3, 3.1 Hz, 2H), 7.66 (t, J=7.0, 1H), 7.59 (t, J=7.2, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 163.1, 162.4, 135.6, 134.8, 133.7, 132.0, 131.5, 129.1, 128.9, 128.8, 126.8, 125.3, 124.5, 124.1, 121.6. MS (ESI) m/z 340.0 [(M+Na)⁺]

1,3-dioxoisoindolin-2-yl thiophene-2-carboxylate



(**3lb**) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.09(t, J = 1.0 Hz, 1H), 7.93 (dd, J =5.4, 3.1 Hz, 2H), 7.82 (m, 3H), 7.23 (t, J=3.8, 1H).

¹³C NMR (100 MHz, CDCl₃) δ 162.0, 158.3, 136.7, 135.7, 134.9, 128.9, 128.5, 127.0, 124.1. MS (ESI) m/z 295.98 [(M+Na)⁺]

1,3-dioxoisoindolin-2-yl furan-2-carboxylate



(**3mb**) yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.93 (dd, J =5.3, 3.1 Hz, 2H), 7.82 (dd, J =5.3, 3.1 Hz, 2H), 7.77 (s, 1H), 7.55 (d, J=3.5, 1H), 6.66 (dd, J=3.4, 1.4Hz,1H). ¹³C NMR (100 MHz, CDCl₃) δ 161.9, 154.5, 148.8, 139.8, 134.9, 128.8, 124.1, 122.3, 112.7 MS (ESI) m/z 280.02 [(M+Na)⁺]

Table 5: N-benzylbenzamide



(4a) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, J = 7.2 Hz, 2H), 7.49 (m, 1H), 7.41 (t, J = 7.2 Hz, 2H), 7.32 (m, 5H), 6.5 (br, 1H), 4.63 (d, J = 6.4 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 167.5, 138.2, 134.4, 131.5, 128.8, 128.6, 127.9, 127.6, 127.0, 44.1.

MS (EI) m/z (%) 211 (M⁺, 100), 105 (86), 91 (9), 77 (42).

N-benzyl-4-methylbenzamide



(4b) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, J = 6.8 Hz, 2H), 7.33 (m, 5H), 7.22 (d, J = 6.8 Hz, 2H), 6.54 (br, 1H), 4.61 (d, J = 7.9 Hz, 2H), 2.38 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 167.4, 142.0, 138.3, 131.5, 129.2, 128.8, 127.9, 127.6, 127.0, 44.1, 21.4.

MS (EI) m/z (%) 225 (M⁺, 48), 119 (100), 106 (12), 91 (67), 77 (15).

N-benzyl-4-chlorobenzamide



(4c) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.70 (s, 2H), 6.78 (m, 7H), 6.61 (br, 1H), 4.60 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 166.4, 138.0, 137.8, 132.7, 128.8, 128.5, 127.9, 127.7, 44.2. MS (EI) m/z (%) 247 (M⁺, 20), 245 (M⁺, 62), 139 (100), 111(48), 91 (28), 77 (30).

N-benzyl-1-naphthamide



(4d) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.31 (s, 1H), 7.87 (m, 2H), 7.36 (m, 9H), 6.39 (br, 1H), 4.89 (s, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 169.5, 138.1, 134.2, 133.7, 130.7, 130.2, 128.8, 128.3, 127.9, 127.7, 127.2, 126.5, 125.4, 125.0, 124.7, 44.1.

MS (EI) m/z (%) 261 (M⁺, 58), 155 (72), 127 (100), 91 (25), 77 (28).

N-benzyl-4-methoxybenzamide



(4e) white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, J = 10.0 Hz, 1H), 7.76 (d, J = 10.0 Hz, 2H), 7.32 (m, 3H), 6.91 (t, J = 8.0 Hz, 3H), 6.46 (br, 1H), 6.62 (d, J = 4.6 Hz, 2H), 3.83 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 167.0, 162.3, 138.4, 132.3, 128.8, 127.9, 127.6, 126.6, 113.8, 55.4, 44.1.

MS (EI) m/z (%) 241 (M⁺, 48), 135 (100), 127 (6), 107 (9), 92 (20), 77 (32).

N-phenethylbenzamide



(4f) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, J = 7.2 Hz, 2H), 7.47 (t, J = 6.8 Hz, 1H), 7.39 (t, J = 6.8 Hz, 2H), 7.32 (t, J = 6.8 Hz, 2H), 7.24 (s, 3H), 6.21 (br, 1H), 3.72 (d, J = 6.0 Hz, 2H), 2.93 (t, J = 6.0 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 167.5, 138.9, 134.7, 131.4, 128.8, 128.7, 128.6, 126.8, 126.6, 41.2, 35.7.

MS (EI) m/z (%) 225 (M⁺, 26), 134 (12), 105 (100), 91 (18), 77 (59).

N-(1-phenylethyl)benzamide



(4g) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, J = 7.2 Hz, 2H), 7.26-7.48 (m, 8H), 6.43 (br, 1H), 5.33 (t, J = 6.8 Hz, 1H), 1.60 (d, J = 6.8 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 166.7, 143.1, 134.6, 131.5, 128.8, 128.7, 126.9, 126.3, 49.3, 21.7. MS (EI) m/z (%) 225 (M⁺, 39), 210 (8), 120 (7), 105 (100), 91 (2), 77 (64).

N-cyclohexylbenzamide



(4h) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, J = 7.2 Hz, 2H), 7.43 (m, 3H), 6.03 (br, 1H), 3.98 (t, J = 4.0 Hz, 1H), 2.03(d, J = 7.0 Hz, 2H), 1.75(d, J = 12.8 Hz, 2H), 1.65(d, J = 12.4 Hz, 1H), 1.43(q, J = 24.3, 12.8 Hz, 2H), 1.24(q, J = 23.2, 10.7 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 166.7, 135.1, 131.2, 128.5, 126.8, 48.7, 33.2, 25.6, 24.9. MS (EI) m/z (%) 203 (M⁺, 34), 160 (5), 122 (66), 105 (100), 92 (20), 77 (62).

N, N-diethylbenzamide

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(4i) colourless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.38 (m, 5H), 3.55 (s, 2H), 3.25 (s, 2H), 1.24 (s, 3H), 1.11 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 171.3, 137.3, 129.1, 128.4, 126.2, 43.3, 39.2, 14.2, 12.9. MS (EI) m/z (%) 177 (M⁺, 22), 176 (60), 148 (4), 134 (3), 105 (100), 77 (34).

phenyl (pyrrolidin-1-yl) methanone



(4j) colourless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.51 (m, 2H), 7.39 (m, 3H), 3.65 (t, J = 7.2 Hz, 2H), 3.42 (t, J = 7.2 Hz, 2H), 1.96 (m, 2H), 1.87 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 169.7, 137.2, 129.8, 128.2, 127.0, 49.6, 46.1, 26.4, 24.5. MS (EI) m/z (%) 175 (M⁺, 63), 146 (22), 105 (100), 77 (46).

(S)-methyl 2-benzamido-3-phenylpropanoate



(4k) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.65 (d, J = 7.2 Hz, 2H), 7.44 (t, J = 7.2 Hz, 1H), 7.35 (t, J = 7.6 Hz, 2H), 7.20 (m, 3H), 7.06 (d, J = 6.8 Hz, 2H), 6.51 (br, 1H), 5.02 (q, J = 13.2, 7.2 Hz, 1H). 3.70 (s, 3H), 3.19 (m, 2H), 1.63 (s, 1H).

¹³C NMR (100 MHz, CDCl₃) δ 172.1, 166.8, 135.9, 133.9, 131.8, 129.3, 128.6, 128.6, 127.2, 127.0, 53.5, 52.4, 37.9.

MS (EI) m/z (%) 283 (M⁺, 2), 224 (7), 207 (7), 162 (72), 131 (14), 105 (100), 91 (10), 77 (35).

N-phenylbenzamide

(41) white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.98 (br, 1H), 7.85 (d, J = 7.2 Hz, 2H), 7.63 (d, J = 7.6 Hz, 2H), 7.47 (m, 3H), 7.35 (t, J = 7.2 Hz, 2H), 7.14 (t, J = 7.2 Hz, 1H) ¹³C NMR (100 MHz, CDCl₃) δ 165.9, 138.0, 135.0, 131.8, 129.0, 128.8, 127.1, 124.6, 120.3. MS (EI) m/z (%) 197 (M⁺, 40), 105 (100), 92 (4), 77 (69).

IV. Copies of ¹H and ¹³C-NMR Spectra

2,5-dioxopyrrolidin-1-yl benzoate (3aa)



2,5-dioxopyrrolidin-1-yl 4-methylbenzoate (3ba)



2,5-dioxopyrrolidin-1-yl 4-methoxybenzoate (3ca)



2,5-dioxopyrrolidin-1-yl 4-fluorobenzoate (3da)



2,5-dioxopyrrolidin-1-yl 4-chlorobenzoate (3ea)



2,5-dioxopyrrolidin-1-yl 4-bromobenzoate (3fa)



2,5-dioxopyrrolidin-1-yl 3-chlorobenzoate (3ga)



2,5-dioxopyrrolidin-1-yl 2-methylbenzoate (3ha)



2,5-dioxopyrrolidin-1-yl 2-chlorobenzoate (3ia)



2,5-dioxopyrrolidin-1-yl 1-naphthoate (3ja)





2,5-dioxopyrrolidin-1-yl thiophene-2-carboxylate (3la)

2,5-dioxopyrrolidin-1-yl furan-2-carboxylate (3ma)



2,5-dioxopyrrolidin-1-yl cinnamate (3na)



Table 3:

1,3-dioxoisoindolin-2-yl benzoate (3ab)







180

160

140

120

100

80

60

40

20

0 ppm



P

nhpi-1

Hz Hz se

HPI-1C

Hz Hz

1,3-dioxoisoindolin-2-yl 4-fluorobenzoate (3db)

8.148 8.127 7.933 7.926 7.835 7.835 7.835 7.536 7.536 7.516 B P NHPI-2 1 20130117 18.53 spect PABBO BB/ CI 0-1 zg30 65536 CDC13 3eb 0 Hz Hz se 1.000000 ANNEL f1 9.93 usec 65536 400.1300094 MHz EM 0 0.30 Hz 0 1.00 NEL fl = NUC1 P1 SI SF WDW SSB LB GB GB PC 8 7 6 5 4 3 2 1 0 ppm 2.00 2.14 & 162.10 161.97 в R hpi-2 1 20130125 18.07 spect PABBO BB/ Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG DW DE TE D1 D11 TD0 С zgpg30 65536 CDC13 256 0 3eb 4 24038.461 0.366798 1.3631988 181.24 20.800 6.50 294.2 2.00000000 0.03000000 1 Hz Hz sec used used sec fl fl 13C 9.63 usec 32768 100.6127690 MH; EM 0 1.00 Hz 1.40 NUC1 P1 SI SF WDW SSB LB GB PC 160 140 100 80 60 40 20 0 ppm 120

1,3-dioxoisoindolin-2-yl 4-chlorobenzoate (3eb)



1,3-dioxoisoindolin-2-yl 4-bromobenzoate (3fb)

1,3-dioxoisoindolin-2-yl 2-methylbenzoate (3hb)



1,3-dioxoisoindolin-2-yl 2-chlorobenzoate (3ib)





1,3-dioxoisoindolin-2-yl 1-naphthoate (3jb)







1,3-dioxoisoindolin-2-yl furan-2-carboxylate (3mb)

Table 5: N-benzylbenzamide (4a)



N-benzyl-4-methylbenzamide(4b)



N-benzyl-4-chlorobenzamide(4c)



N-benzyl-1-naphthamide(4d)













N-cyclohexylbenzamide(4h)







phenyl (pyrrolidin-1-yl) methanone (4j)





(S)-methyl 2-benzamido-3-phenylpropanoate (4k)

N-phenylbenzamide (41)

