Supporting Information

DDQ-Promoted direct Transformation of Benzyl Hydrocarbons to Amides under Transition Metal-Free Conditions

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1. General Methods

Unless stated otherwise, all reagents were purchased commercially without further purification. All reagents were weighed and handled in air at room temperature. All glassware was oven or flame dried immediately prior to use.

$^1$H NMR and $^{13}$C NMR were obtained at 400 MHz and recorded relative to the tetramethylsilane signal (0 ppm) or residual protio-solvent. Chemical shifts are expressed in parts per million values (δ, ppm). $^1$H NMR spectra were calibrated with DMSO-$d_6$ (δ = 2.50 ppm) and CDCl$_3$ (δ = 7.26 ppm). $^{13}$C-NMR spectra were obtained at 100 MHz and were calibrated with DMSO-$d_6$ (δ = 39.50 ppm) and CDCl$_3$ (δ = 77.00 ppm). Data for $^1$H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s= singlet, d= doublet, t= triplet, q= quartet, m= multiplet or unresolved, br= broad singlet, coupling constant(s) in Hz, integration). Mass spectra were recorded using a PE SCLEX QSTAR spectrometer. Purification was done by column chromatography on silica gel (200–300 mesh) with petroleum ether and ethyl acetate as the eluent to give the pure product.

2. General procedure

2.1 Synthesis of amides from 1, 3-diarylpropene and diarylmethane:

**Condition A**: An oven-dried Schlenk tube was charge with $1a$-o (0.5 mmol), NH$_2$-OH.HCl (0.108g, 1.5 mmol), DDQ (0.34 g, 1.5mmol), PPA (0.052g, 0.15mmol), HCOOH (1.5 mL) and CH$_3$CN (1.5 mL). The reaction mixture was stirred at 80 ºC for 12 h monitored by TLC. The mixture was allowed to cool to room temperature and was quenched with H$_2$O (10 mL). The mixture was extracted with DCM (3 × 10 mL), and the organic layer was washed with brine (10mL). The combined organic layers were dried with Na$_2$SO$_4$, concentrated under reduced pressure, and dried under high vacuum. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate, 15:1 to 8:1) to obtain the desired products $2a$-o.

2.2 Synthesis of amides from diarylmethane:

**Condition B**: An oven-dried Schlenk tube was charge with $3a$-h (1.5 mmol), NH$_2$-OH.HCl (0.035g, 0.5 mmol), DDQ (0.34 g, 1.5mmmol), AlCl$_3$ (0.052g, 0.15mmol), HCOOH (1.5 mL) and CH$_3$CN (1.5 mL). The reaction mixture was stirred at 80ºC for 12 h monitored by TLC. The mixture was allowed to cool to room temperature and was quenched with H$_2$O (10 mL). The mixture was extracted with DCM (3 × 10 mL), and the organic layer was washed with brine (10mL). The combined organic layers were dried with Na$_2$SO$_4$, concentrated under reduced pressure, and dried under high vacuum. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate, 15:1 to 8:1) to obtain the desired products $4a$-h.

2.3 Investigation of the mechanism and possible key intermediates:

An oven-dried Schlenk tube was charge with $1a$ (0.5 mmol), NH$_2$-OH.HCl (0.108g, 1.5 mmol), DDQ (0.114 g, 0.5mmmol), HCOOH (1.5 mL) and CH$_3$CN (1.5 mL). The reaction mixture was stirred at room temperature for 12h monitored by TLC. The mixture was quenched with H$_2$O (10 mL). The mixture was extracted with DCM (3 × 10 mL), and the organic layer was washed with brine (10 mL). The combined organic layers were dried with Na$_2$SO$_4$, concentrated under reduced pressure, and dried under high vacuum. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate, 4:1) to obtain the desired products $5a$ (97% yield).

N-[1, 3-Diphenyl-(2E)-propenyl] hydroxylamine (5a)

$^1$H NMR (400 MHz, CDCl$_3$, ppm): δ 7.43-7.31 (m, 9H), 7.26-7.19 (m, 1H), 6.64(d, $J = 15.6$Hz, 1H), 6.39 (dd, $J = 7.6$, 12.0 Hz, 1H), 5.43 (br, 1H), 4.74(d, $J = 7.6$Hz, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$, ppm): δ 139.9, 136.6, 132.8, 156.1, 128.7, 128.6, 128.5, 127.9, 127.8, 127.9, 126.5, 69.2.
An oven-dried Schlenk tube was charge with 5a (0.5 mmol), DDQ (0.227 g, 1 mmol), CH3CN (2 mL). The reaction mixture was stirred at 80 °C for 4 h monitored by TLC. The mixture was allowed to cool to room temperature and was quenched with H2O (10 mL). The mixture was extracted with DCM (3 × 10 mL), and the organic layer was washed with brine (10 mL). The combined organic layers were dried with Na2SO4, concentrated under reduced pressure, and dried under high vacuum. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate, 8:1) to obtain the desired products 6a (98% yield).

\[(1E,2E)-\text{Chalcone O-acetyloxime (6a)}\]

\[\text{1H NMR (400 MHz, CDCl}_3\text{, ppm): } \delta 8.93 \text{ (br, 1H), 7.71 (d, } J = 16.4 \text{ Hz, 1H), 7.57-7.47 (m, 7H), 7.40-7.34 (m, 3H), 6.84 (d, } J = 16.4 \text{ Hz, 1H).} \]

\[\text{13C NMR (101 MHz, CDCl}_3\text{, ppm): } \delta 157.8, 139.7, 136.2, 134.8, 129.3, 129.2, 128.8, 128.5, 127.5, 117.5.\]

An oven-dried Schlenk tube was charge with 6a (0.5 mmol), PPA (0.052 g, 0.15 mmol), HCOOH (1.5 mL) and CH3CN (1.5 mL). The reaction mixture was stirred at room temperature for 12 h monitored by TLC. The mixture was quenched with H2O (10 mL). The mixture was extracted with DCM (3 × 10 mL), and the organic layer was washed with brine (10 mL). The combined organic layers were dried with Na2SO4, concentrated under reduced pressure, and dried under high vacuum. The residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate, 15:1) to obtain the desired products 2a (91% yield).

### 3. Characterization of the Compounds

**N-Phenylcinnamamide (2a)**

Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (83% yield); 2a \[\text{1H NMR (400 MHz, CDCl}_3\text{, ppm): } \delta 7.79 \text{ (d, } J = 15.6 \text{ Hz, 1H), 7.66-7.64 (m, 2H), 7.57-7.55 (m, 2H), 7.44-7.36 (m, 5H), 7.16 \text{ (t, } J = 7.2 \text{ Hz, 1H), 6.59 (d, } J = 15.6 \text{ Hz, 1H).} \]

\[\text{13C NMR (101 MHz, CDCl}_3\text{, ppm): } \delta 164.4, 142.3, 138.1, 134.6, 129.9, 129.1, 128.9, 128.0, 124.5, 121.0, 120.2. \]

HRMS m/z (ESI) calcd. for C15H14NO (M+Na)+: 246.0895, found 246.0893.

**3. Di-p-tolylacrylamide (2b)**

Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (89% yield); 2b \[\text{1H NMR (400 MHz, CDCl}_3\text{, ppm): } \delta 7.72 \text{ (d, } J = 15.6 \text{ Hz, 1H), 7.50 (d, } J = 6.8 \text{ Hz, 2H), 7.42 (d, } J = 7.6 \text{ Hz, 2H), 7.33 (s, 1H), 7.19-7.14 (m, 4H), 6.49 (d, } J = 15.6 \text{ Hz, 1H), 2.37 (s, 3H), 2.33 (s, 3H).} \]

\[\text{13C NMR (101 MHz, CDCl}_3\text{, ppm): } \delta 164.1, 142.1, 140.2, 135.5, 134.0, 132.0, 129.6, 127.9, 120.0, 21.4, 20.9. \]

HRMS m/z (ESI) calcd. for C17H17NO (M+Na)+: 274.1208, found 274.1190.
(E)-N, 3-Bis(4-fluorophenyl)acrylamide (2d)
Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (78% yield); 2d \( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): \( \delta 7.73 \) (d, \( J = 15.6 \) Hz, 1H), 7.60-7.50 (m, 5H), 7.07 (dd, \( J = 18.4, 9.2 \) Hz, 4H), 6.49 (d, \( J = 15.6 \) Hz, 1H). \( ^{13}C \) NMR (101 MHz, CDCl\(_3\), ppm): \( \delta 165.0, 163.9, 162.5, 160.7, 141.3, 133.9, 130.8, 130.7, 129.8, 129.7, 121.8, 120.3, 116.2, 115.9, 115.7 \). HRMS m/z (ESI) calcd. for C\(_{15}H_{11}F_2NO (M+Na)^+\): 282.0706, found 282.0701.

(2d)

(E)-N, 3-Bis(4-chlorophenyl)acrylamide (2e)
Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (85% yield); 2e \( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): \( \delta 7.73 \) (d, \( J = 15.6 \) Hz, 1H), 7.59 (d, \( J = 8.0 \) Hz, 2H), 7.49-7.33 (m, 7H), 6.52 (d, \( J = 15.6 \) Hz, 1H). \( ^{13}C \) NMR (101 MHz, DMSO-\( d_6 \), ppm): \( \delta 163.9, 139.6, 138.6, 134.8, 134.1, 129.9, 129.5, 127.5, 123.3, 121.2 \). HRMS m/z (ESI) calcd. for C\(_{15}H_{11}Cl_2NO (M+Na)^+\): 314.0115, found 314.0113.

(2e)

(E)-N, 3-Bis(4-bromophenyl)acrylamide (2f)
Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (82% yield); 2f \( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): \( \delta 7.72 \) (d, \( J = 15.6 \) Hz, 1H), 7.56-7.47 (m, 6H), 7.41 (d, \( J = 8.4 \) Hz, 1H), 7.36 (br, 1H), 6.53 (d, \( J = 15.2 \) Hz, 1H), 7.36 (br, 1H). \( ^{13}C \) NMR (101 MHz, DMSO-\( d_6 \), ppm): \( \delta 163.9, 139.7, 139.0, 134.4, 132.5, 130.2, 123.5, 123.3, 121.6, 115.5 \). HRMS m/z (ESI) calcd. for C\(_{15}H_{11}Br_2NO (M+Na)^+\): 403.9085, found 403.9095.

(2f)

N-\( p \)-Tolylcinnamamide (2g)
(2g)

(2g)

(E)-3-(\( p \)-Tolyl)-N-phenylacrylamide (2g')
Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (86% yield); 2g \( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): \( \delta 7.77 \) (d, \( J = 15.6 \) Hz, 1H), 7.64 (d, \( J = 7.6 \) Hz, 1H), 7.54 (br, 1H), 7.46-7.35 (m, 5H), 7.22-7.14 (m, 3H), 6.58 (d, \( J = 15.6 \) Hz, 1H), 2.35 (s, 3H). \( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): \( \delta 7.76 \) (d, \( J = 15.6 \) Hz, 1H), 7.64 (d, \( J = 6.4 \) Hz, 1H), 7.45 (d, \( J = 7.6 \) Hz, 2H), 7.40 (br, 1H), 7.37 (t, \( J = 8.0 \) Hz, 2H), 7.21 (d, \( J = 7.6 \) Hz, 2H), 7.15 (t, \( J = 7.2 \) Hz, 1H), 6.53 (d, \( J = 15.6 \) Hz, 1H), 2.40 (s, 1H). \( ^{13}C \) NMR (101 MHz, CDCl\(_3\), ppm): \( \delta 164.1, 163.7, 140.4, 140.6, 140.1, 139.8, 132.5, 130.2, 129.6, 129.5, 130.1, 129.2, 128.2, 123.7, 123.5, 121.7, 119.7, 21.4, 21.0 \). HRMS m/z (ESI) calcd. for C\(_{16}H_{15}NO (M+Na)^+\): 260.1051, found 260.1048.

(2g')

N-\( o \)-Tolylecinnamamide (2h)

(E)-3-(\( o \)-Tolyl)-N-phenylacrylamide (2h')
Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (64% yield); 2h \( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): \( \delta 7.72 \) (d, \( J = 15.6 \) Hz, 1H), 7.64 (d, \( J = 7.6 \) Hz, 1H), 7.54 (br, 1H), 7.46-7.35 (m, 5H), 7.22-7.14 (m, 3H), 6.58 (d, \( J = 15.6 \) Hz, 1H), 2.35 (s, 3H). \( ^1H \) NMR (400 MHz, CDCl\(_3\), ppm): \( \delta 7.76 \) (d, \( J = 15.6 \) Hz, 1H), 7.64 (d, \( J = 6.4 \) Hz, 1H), 7.45 (d, \( J = 7.6 \) Hz, 2H), 7.40 (br, 1H), 7.37 (t, \( J = 8.0 \) Hz, 2H), 7.21 (d, \( J = 7.6 \) Hz, 2H), 7.15 (t, \( J = 7.2 \) Hz, 1H), 6.53 (d, \( J = 15.6 \) Hz, 1H), 2.40 (s, 1H). \( ^{13}C \) NMR (101 MHz, CDCl\(_3\), ppm): \( \delta 164.1, 163.7, 140.4, 140.6, 140.1, 139.8, 132.5, 130.2, 129.6, 129.5, 130.1, 129.2, 128.2, 123.7, 123.5, 121.7, 119.7, 21.4, 21.0 \). HRMS m/z (ESI) calcd. for C\(_{16}H_{15}NO (M+Na)^+\): 260.1051, found 260.1048.

(2h')
δ 8.08 (d, J = 15.2 Hz, 1H), 7.67 (m, 3H), 7.55 (d, J = 15.6 Hz, 1H), 7.37 (t, J = 8.0 Hz, 2H), 7.23-7.14 (m, 3H), 6.51 (d, J = 15.6 Hz, 1H), 2.45 (s, 3H).

2h': 1H NMR (400 MHz, CDCl₃, ppm): δ 8.00 (br, 1H), 7.79 (d, J = 15.6 Hz, 1H), 7.57 (s, 2H), 7.40 (s, 3H), 7.27-7.14 (m, 4H), 6.62 (d, J = 15.2 Hz, 1H), 2.34 (s, 1H).

13C NMR (101 MHz, CDCl₃, ppm): δ 164.2, 163.9, 142.4, 140.2, 138.1, 137.8, 136.0, 135.8, 134.7, 133.7, 130.8, 130.5, 129.9, 129.7, 129.1, 128.9, 128.0, 126.9, 126.2, 124.4, 122.0, 120.0, 100.0, 19.8, 17.8. HRMS m/z (ESI) calcd. for C₁₆H₁₅NO (M+Na)⁺: 260.1051, found 260.1036.

N-(3-(Trifluoromethyl) phenyl)cinnamamide (2j)

(E)-3-(3- (Trifluoromethyl) phenyl)-N-phenylacrylamide (2j')

Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (75% yield); 2j: 1H NMR (400 MHz, CDCl₃, ppm): δ 8.38 (br, 1H), 7.93 (s, 1H), 7.86 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 15.6 Hz, 1H), 7.43-7.30 (m, 7H), 6.65 (d, J = 15.6 Hz, 1H).

2j': 1H NMR (400 MHz, CDCl₃, ppm): δ 8.25 (br, 1H), 7.93 (s, 1H), 7.86 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 15.6 Hz, 1H), 7.43-7.30 (m, 7H), 6.64 (d, J = 15.6 Hz, 1H). 13C NMR (101 MHz, CDCl₃, ppm): δ 164.7, 143.2, 138.6, 134.3, 131.9, 131.6, 131.3, 130.9, 130.2, 129.6, 128.9, 128.0, 125.2, 122.3, 122.5, 121.0, 121.0, 120.3, 116.9. HRMS m/z (ESI) calcd. for C₁₆H₁₂F₃NO (M+Na)⁺: 314.0769, found 324.0747.

N-(3-Bromophenyl)cinnamamide (2k)

(E)-3-(3-Bromophenyl)-N-phenylacrylamide (2k')

Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (68% yield); 2k: 1H NMR (400 MHz, CDCl₃, ppm): δ 7.93 (d, J = 16.0 Hz, 1H), 7.65 (d, J = 6.0 Hz, 1H), 7.49 (d, J = 7.6 Hz, 2H), 7.39-7.34 (m, 3H), 7.27-7.14 (m, 3H), 6.57 (d, J = 15.6 Hz, 1H).

2k': 1H NMR (400 MHz, CDCl₃, ppm): δ 7.85 (br, 1H), 7.77 (d, J = 15.6 Hz, 1H), 7.47 (d, J = 7.2 Hz, 1H), 7.40-7.35 (m, 3H), 7.27-7.14 (m, 3H), 6.57 (d, J = 15.6 Hz, 1H). 13C NMR (101 MHz, CDCl₃, ppm): δ 164.4, 164.0, 142.9, 140.7, 139.5, 136.7, 134.4, 132.7, 130.5, 130.4, 130.3, 129.1, 128.9, 128.0, 127.4, 124.8, 123.2, 123.0, 122.7, 122.4, 120.6, 120.3, 118.7. HRMS m/z (ESI) calcd. for C₁₅H₁₂BrNO (M+Na)⁺: 325.9979, found 325.9969.

N-(4-Bromophenyl)cinnamamide (2l)

(E)-3-(4-Bromophenyl)-N-phenylacrylamide (2l')

Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (83% yield); 2l: 1H NMR (400 MHz, CDCl₃, ppm): δ 7.77 (d, J = 15.2 Hz, 1H), 7.64 (d, J = 7.2 Hz, 1H), 7.56 (br, 1H), 7.54-7.51 (m, 3H), 7.47 (d, J = 7.2 Hz, 1H), 7.40-7.35 (m, 3H), 6.57 (d, J = 15.6 Hz, 1H).

2l': 1H NMR (400 MHz, CDCl₃, ppm): δ 7.70 (d, J = 15.2 Hz, 1H), 7.56 (br, 1H), 7.54-7.51 (m, 3H), 7.47 (d, J = 7.2 Hz, 1H), 7.40-7.35 (m, 2H), 7.16 (d, J = 7.2 Hz, 1H), 6.57 (d, J = 15.2 Hz, 1H). 13C NMR (101 MHz, DMSO-d₆, ppm): δ 164.1, 163.8, 141.0, 139.7, 139.3, 139.1, 135.1, 134.5, 132.5, 132.1, 130.4, 130.1, 129.5, 129.3, 128.2, 123.4, 123.9, 123.7, 123.4, 122.4, 119.7, 115.4. HRMS m/z (ESI) calcd. for C₁₅H₁₂BrNO (M+Na)⁺: 324.0000, found 324.0022.
\[ \text{N-(4-Fluorophenyl)cinnamamide (2n')} \]

\[(E)-3-(4-Fluorophenyl)-N-phenylacrylamide (2n)\]

Condition A. Eluent: petroleum ether/ethyl acetate (15:1). White solid (76% yield); \(2n\): \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.78 (d, \(J = 15.6\) Hz, 1H), 7.65-7.50 (m, 5H), 7.40-7.36 (m, 2H), 7.16 (t, \(J = 7.2\) Hz, 1H), 7.10-7.04 (m, 2H), 6.58 (d, \(J = 15.6\) Hz, 1H). \(2n'\): \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.74 (d, \(J = 15.2\) Hz, 1H), 7.65-7.50 (m, 5H), 7.40-7.36 (m, 3H), 7.10-7.04 (m, 2H), 6.52 (d, \(J = 15.6\) Hz, 1H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\), ppm): \(\delta\) 165.0, 164.1, 162.5, 142.5, 141.2, 141.0, 139.3, 137.9, 134.5, 134.1, 132.5, 132.1, 130.9, 130.1, 130.0, 129.8, 129.7, 129.5, 129.3, 129.1, 128.9, 128.8, 128.0, 124.6, 123.4, 122.4, 121.8, 120.6, 120.0, 119.7, 116.1, 115.9, 115.8, 115.6. HRMS m/z (ESI) calcd. for \(C_{15}H_{12}FNO\) (M+Na)\(^{+}\): 264.0801, found 264.0791.

\[4a\]

4-Methoxy-\(N\)-(4-Methoxyphenyl)benzamide (4a)

Condition B. Eluent: petroleum ether/ethyl acetate (8:1). White solid (96% yield); \(4a\): \(^1\)H NMR (400 MHz, DMSO-\(d_6\), ppm): \(\delta\) 9.96 (s, 1H), 7.94 (d, \(J = 8.8\) Hz, 2H), 7.65 (d, \(J = 9.2\) Hz, 2H), 7.05 (d, \(J = 8.8\) Hz, 2H), 6.91 (d, \(J = 8.8\) Hz, 2H), 3.83 (s, 3H), 3.74 (s, 3H). \(^{13}\)C NMR (101 MHz, DMSO-\(d_6\), ppm): \(\delta\) 164.9, 162.2, 155.9, 132.9, 129.9, 127.5, 122.4, 114.2, 114.0, 55.9, 55.0. HRMS m/z (ESI) calcd. for \(C_{15}H_{15}NO_3\) (M+Na)\(^{+}\): 280.0950, found 280.0948.

\[4b\]

\(p\)-Tolyl-\(N\)-(\(p\)-tolyl)benzamide (4b)

Condition B. Eluent: petroleum ether/ethyl acetate (15:1). White solid (89% yield); \(4b\): \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.79 (d, \(J = 8.4\) Hz, 3H), 7.54 (d, \(J = 7.6\) Hz, 2H), 7.30 (d, \(J = 8.0\) Hz, 2H), 7.19 (d, \(J = 8.4\) Hz, 2H), 2.44 (s, 3H), 2.36 (s, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\), ppm): \(\delta\) 165.6, 165.2, 162.4, 156.5, 142.2, 135.5, 134.1, 132.2, 129.6, 129.4, 127.0, 120.2, 21.5, 20.9. HRMS m/z (ESI) calcd. for \(C_{15}H_{15}NO_3\) (M+Na)\(^{+}\): 248.1051, found 248.1024.

\[4c\]

\(N\)-(4-Methoxyphenyl)-\(p\)-toluamide (4c)

\[4c'\]

4-Methoxy-\(N\)-(\(p\)-methoxyphenyl)benzamide (4c')

Condition B. Eluent: petroleum ether/ethyl acetate (15:1). White solid (93% yield); \(4c\): \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.86 (s, 1H), 7.85 (d, \(J = 8.0\) Hz, 2H), 7.54 (d, \(J = 8.8\) Hz, 2H), 7.17 (d, \(J = 8.0\) Hz, 2H), 6.91 (d, \(J = 8.4\) Hz, 2H), 3.83 (s, 3H), 2.43 (s, 3H). \(4c'\): \(^1\)H NMR (400 MHz, CDCl\(_3\), ppm): \(\delta\) 7.86 (s, 1H), 7.85 (d, \(J = 8.4\) Hz, 2H), 7.53 (d, \(J = 8.8\) Hz, 2H), 7.27 (d, \(J = 8.0\) Hz, 2H), 6.96 (d, \(J = 8.8\) Hz, 2H), 3.88 (s, 3H), 2.35 (s, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\), ppm): \(\delta\) 165.6, 165.2, 162.4, 156.5, 142.2, 135.6, 134.0, 132.2, 131.2, 129.5, 129.4, 128.9, 127.3, 127.0, 122.1, 120.3, 114.2, 113.9, 55.5, 21.5, 20.9. HRMS m/z (ESI) calcd. for \(C_{15}H_{15}NO_2\) (M+Na)\(^{+}\): 264.1000, found 264.1028.
N-(4-Methoxyphenyl)benzamide (4d)

4-Methoxy-N-phenylbenzamide (4d')

Condition B. Eluent: petroleum ether/ethyl acetate (15:1). White solid (87% yield); 4d: 1H NMR (400 MHz, CDCl3, ppm): δ 7.88 (d, J = 7.2 Hz, 2H), 7.83 (br, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 8.8 Hz, 2H), 7.50 (d, J = 8.8 Hz, 1H), 7.38 (t, J = 7.6 Hz, 2H), 6.93 (d, J = 8.8 Hz, 1H), 3.84 (s, 3H). 13C NMR (101 MHz, CDCl3, ppm): δ 162.5, 156.7, 138.1, 135.1, 131.7, 131.0, 129.1, 128.9, 128.8, 127.0, 124.3, 122.1, 120.2, 114.3, 114.0, 55.5. HRMS m/z (ESI) calcd. for C14H13NO2 (M+Na)+: 250.0844, found 250.0828.

4-Fluoro-N-(4-Methoxyphenyl)benzamide (4e)

4-Methoxy-N-(4-fluorophenyl)benzamide (4e')

Condition B. Eluent: petroleum ether/ethyl acetate (15:1). White solid (61% yield); 4e: 1H NMR (400 MHz, CDCl3, ppm): δ 7.86 (br, 1H), 7.85 (d, J = 7.6 Hz, 2H), 7.60 (d, J = 7.6 Hz, 2H), 7.06 (t, J = 8.0 Hz, 2H), 6.98 (d, J = 8.0 Hz, 2H), 3.89 (s, 3H). 13C NMR (101 MHz, CDCl3, ppm): δ 165.2, 162.6, 160.7, 158.1, 131.2, 130.8, 129.4, 129.3, 128.9, 126.9, 122.2, 122.1, 122.0, 115.9, 115.8, 115.7, 115.6, 114.3, 114.0, 55.5. HRMS m/z (ESI) calcd. for C14H12FNO2 (M+Na)+: 268.0750, found 268.0731.

4-Chloro-N-(4-Methoxyphenyl)benzamide (4f)

4-Methoxy-N-(4-chlorophenyl)benzamide (4f')

Condition B. Eluent: petroleum ether/ethyl acetate (15:1). White solid (73% yield); 4f: 1H NMR (400 MHz, CDCl3, ppm): δ 7.83 (d, J = 9.2 Hz, 2H), 7.76 (br, 1H), 7.61 (d, J = 7.6 Hz, 2H), 7.54 (d, J = 8.0 Hz, 1H), 7.47 (d, J = 7.6 Hz, 1H), 7.34 (d, J = 8.0 Hz, 2H), 6.93 (d, J = 8.0 Hz, 1H), 3.84 (s, 3H). 13C NMR (101 MHz, CDCl3, ppm): δ 165.4, 164.4, 162.5, 156.1, 138.8, 136.7, 134.2, 132.5, 130.1, 130.0, 128.9, 127.5, 127.2, 122.5, 122.3, 114.2, 114.1, 55.9, 55.7. HRMS m/z (ESI) calcd. for C14H12ClNO2 (M+Na)+: 284.0405, found 250.0448.

3-Trifluoromethyl-N-(4-Methoxyphenyl)benzamide (4g)

4-Methoxy-N-(3-(trifluoromethyl)phenyl)benzamide (4g')

Condition B. Eluent: petroleum ether/ethyl acetate (15:1). White solid (38% yield); 4g: 1H NMR (400 MHz, CDCl3, ppm): δ 8.14 (br, 1H), 8.08 (d, J = 7.6 Hz, 1H), 7.83 (d, J = 7.6 Hz, 2H), 7.65 (t, J = 8.0 Hz, 1H), 7.57 (d, J = 8.8 Hz, 2H), 6.95 (d, J = 8.8 Hz, 2H), 3.85 (s, 3H). 13C NMR (101 MHz, CDCl3, ppm): δ 7.95 (br, 1H), 7.91-7.86 (m, 3H), 7.83 (d, J = 7.6 Hz, 1H), 7.51 (t, J = 8.0 Hz, 1H), 7.41 (d, J = 7.6 Hz, 1H), 7.01 (d, J = 8.8 Hz, 2H), 3.85 (s, 3H).

4. References


5. NMR Charts

![NMR Chart Image]