# **Supporting Information**

### A Novel Construction of Acetamides from Rhodium-Catalyzed

### Aminocarbonylation of DMC with Nitro compounds

Zhi-Peng Bao,<sup>a</sup> Ren-Guan Miao,<sup>a</sup> Xinxin Qi,<sup>\*a</sup> Xiao-Feng Wu<sup>\*a,b</sup>

<sup>a</sup>.Department of Chemistry, Zhejiang Sci-Tech University, Hangzhou, Zhejiang 310018, People's Republic of China

<sup>b</sup>·Leibniz-Institut für Katalyse e.V. an der Institution, Universität Rostock, Albert-Einstein-Straße 29a, Rostock 18059, Germany

## **Table of Contents**

1. General Information ······S1
2. General Procedure ······S2
3. Synthesis of 1i, 1w, 4 and 6·····S2
4. Mechanistic Studies
5.1 Characterization of 1i, 1w, 4 and 6S6
5.2 Characterization of Products······S7
6. References······S21
7.1 Copy of <sup>1</sup> H and <sup>13</sup> C NMR Spectra of 1i, 1w, 4 and 6S22
7.2 Copy of <sup>1</sup> H and <sup>13</sup> C NMR Spectra of ProductsS26

#### **1. General Information**

Unless otherwise noted, all reactions were carried out under a nitrogen atmosphere. All reagents were from commercial sources, all solvents are extra dry solvents and used as received without further purification. Column chromatography was performed on silica gel (200-300 meshes) using petroleum ether (b.p. 60-90 °C) and ethyl acetate as the eluents. <sup>1</sup>H and <sup>13</sup>C NMR spectra were taken on 400 MHz instruments and spectral data were reported in ppm relative to tetramethylsilane (TMS) as the internal standard and CDCl<sub>3</sub> or DMSO-D<sub>6</sub> as solvent. All coupling constants (*J*) are reported in Hz with the following abbreviations: s = singlet, d = doublet, dd = double doublet, ddd = double doublet of doublets, t = triplet, dt = double triplet, q = quartet, m = multiplet, br = broad. Gas (GC) analyses were performed on a Shimadzu GC-2014C chromatograph equipped with FID detector. Mass spectra (MS) were measured on spectrometer by direct inlet at 70 eV.

#### 2. General Procedure



 $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. Nitrobenzenes (0.5 mmol), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via a syringe. The tube was sealed, and the mixture was stirred at 120 °C for 24 h. After the reaction was completed, the crude mixture was filtered and concentrated under vacuum. The crude product was purified by column chromatography (PE/EA=1/1) on silica gel to afford the desired acetamide products.

#### **3.** Synthesis of 1i<sup>1</sup>, 1w<sup>2</sup>, 4<sup>1</sup> and 6<sup>3</sup>



An oven-dried round-bottom flask was charged under air with 4-methylbenzenesulfonyl chloride (12 mmol, 2.28 g), DCM (40 mL), 4-nitrophenol (10 mmol, 1.39 g), and triethylamine (15 mmol, 1.52 g) was stirred at 0  $^{\circ}$ C until completion (monitored by TLC). The reaction mixture was then diluted with 40 mL of H<sub>2</sub>O and extracted three times with 25 mL of DCM. The combined organic phases were dried over magnesium sulfate, filtered through short celite pad, and concentrated under reduced pressure. Purification by column chromatography (PE/EA=10/1) afforded **1i** as a white solid (2.34g, 80%).



A mixture of indole (5 mmol, 0.59 g), 1- fluoro-2-nitrobenzene (5 mmol, 0.71 g) and NaOH (5 mmol, 0.20 g) in DMSO (5 mL) was stirred vigorously for 2 h at room temperature. After cooling, the reaction mixture was poured into water (30 mL) and extracted with EtOAc three times (3  $\times$  30 mL). The combined organic layers were dried with Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. Purification by column chromatography (PE/DCM=5/1) afforded **1w** as a light yellow solid (1.07 g, 90%).



An oven-dried round-bottom flask was charged under air with D-10-Camphorsulfonyl chloride (12 mmol, 3.00 g), DCM (40 mL), 4-nitrophenol (10 mmol, 1.39 g), and triethylamine (15 mmol, 1.52 g) was stirred at 0  $^{\circ}$ C until completion (monitored by TLC). The reaction mixture was then diluted with 40 mL of H<sub>2</sub>O and extracted three times with 25 mL of DCM. The combined organic phases were dried over magnesium sulfate, filtered through short celite pad, and concentrated under reduced pressure. Purification by column chromatography (PE/EA=10/1) afforded **4** as a white solid (2.47 g, 70%).



An oven-dried round-bottom flask was charged under air with dehydroabietylamine (10 mmol, 2.85 g), DCM (40 mL), and triethylamine (12 mmol, 1.21 g), 4-nitrobenzoyl chloride (11 mmol, 2.04 g) was add in slowly and stirred at 0 °C, then stir the reaction mixture at room temperature until completion (monitored by TLC). The reaction mixture was then diluted with 40 mL of H<sub>2</sub>O and extracted three times with 25 mL of DCM. The combined organic phases were dried over magnesium sulfate, filtered through short celite pad, and concentrated under reduced pressure. Purification by column chromatography (PE/EA=10/1 to 5/1) a fforded **6** as a light yellow solid (2.60 g, 60%).

#### 4. Mechanistic Studies



**(a)** 

i) TEMPO (1 mol, 156.1 mg),  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na\_3PO\_4 (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b** (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. The tube was sealed and the mixture was stirred at 120 °C for 24 h. After the reaction was completed, **2b** was not observed. (Yields determined by GC analysis using hexadecane as an internal standard).

ii) 1,1-DPE (1 mol, 180.1 mg),  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na\_3PO\_4 (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b** (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. The tube was sealed and the mixture was stirred at 120 °C for 24 h.After the reaction was completed, the product **2b** was observed in 73% yield. (Yields determined by GC analysis using hexa decane as an internal standard).

iii) (1) BHT (1 mol, 220.2 mg),  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na\_3PO\_4 (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b** (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. The tube was sealed and the mixture was stirred at 120 °C for 24 h. After the reaction was completed, the product **2b** was observed in 73% yield. (Yields determined by GC analysis using hexa decane as an internal standard).

iii) (2) BHT (2.5 mol, 551.5 mg), Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30

mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b** (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. The tube was sealed and the mixture was stirred at 120 °C for 24 h. After the reaction was completed, the product **2b** was observed in 72% yield. (Yields determined by GC analysis using hex a decane as an internal standard).

(b)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), W(CO)\_6 (1 mmol, 351.9 mg),  $Na_3PO_4$  (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b** (0.5 mmol, 68.5 mg), MeI (0.5 mmol, 71.0 mg),  $H_2O$  (1 mmol, 18.0 mg), and CH<sub>3</sub>CN (1.5 mL) were added into the tube via syringe. The tube was sealed and the mixture was stirred at 120 °C for 24 h. After the reaction was completed, the product **2b** was observed in 31% yield. (Yields determined by GC analysis using hexadecane as an internal standard).

(c)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b** (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and CH<sub>3</sub>OH (1.5 mL) were added into the tube via syringe. The tube was sealed and the mixture was stirred at 120 °C for 24 h. After the reaction was completed, the product **2b** was not observed. (Yields determined by GC analysis using hexadecane as an internal standard).

(d)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b** (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and anisole (1.5 mL) were added into the tube via syringe. The tube was sealed and the mixture was stirred at 120 °C for 24 h. After the reaction was completed, the product **2b** was not observed. (Yields determined by GC analysis using hexadecane as an internal standard).

(e)

(1)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na\_3PO\_4 (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1a'** (0.5 mmol, 53.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. After the reaction was completed, the product **2a** was observed in 34% yield. (Yields determined by GC analysis using hexa decane as an internal standard).

(2)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1b'** (0.5 mmol, 54.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. After the reaction was completed, the product **2a** was observed in 32% yield. (Yields determined by GC analysis using hex a decane as an internal standard).

(3)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1c'** (0.5 mmol, 91.0 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. After the reaction was

completed, the product **2a** was observed in 53% yield. (Yields determined by GC analysis using hexadecane as an internal standard).

(4)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1d'** (0.5 mmol, 92.1 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. After the reaction was completed, the product **2a** was observed in 45% yield. (Yields determined by GC analysis using hex a decane as an internal standard).

(5)  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1e'** (0.5 mmol, 46.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. After the reaction was completed, the product **2a** was observed in 33% yield. (Yields determined by GC analysis using hexa decane as an internal standard).

#### 5.1 Characterization of 1i, 1w, 4 and 6



1-methyl-4-((4-nitrophenyl)sulfonyl)benzene (1i)<sup>1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.13 (d, J = 9.1 Hz, 2H), 7.68 (d, J = 8.3 Hz, 2H), 7.32 (d, J = 8.2 Hz, 2H), 7.15 (d, J = 9.1 Hz, 2H), 2.41 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  153.6, 146.1, 145.9, 131.3, 129.9, 128.2, 125.2, 123.0, 21.5. M.p. 93.4 – 95.2 °C



1-(2-nitrophenyl)-1H-indole (1w)<sup>2</sup>

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.82 (d, J = 8.1 Hz, 1H), 7.64 – 7.56 (m, 1H), 7.44 (t, J = 7.7 Hz, 1H), 7.35 – 7.23 (m, 2H), 7.15 – 7.01 (m, 4H), 6.63 (d, J = 3.1 Hz, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 145.7, 136.3, 133.6, 132.2, 129.3, 128.7, 128.1, 127.8, 125.1, 122.6, 121.0, 120.6, 109.2, 104.6.

M.p. 59.6 – 61.5 °C

4-nitrophenyl((1S,4R)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methanesulfonate (4)<sup>1</sup>

<sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.29 (d, J = 8.7 Hz, 2H), 7.50 (d, J = 8.7 Hz, 2H), 3.86 (d, J = 15.0 Hz, 1H), 3.29 (d, J = 15.0 Hz, 1H), 2.55 - 2.38 (m, 2H), 2.22 - 1.95 (m, 3H), 1.82 - 1.73 (m, 1H), 1.58 - 1.43 (m, 1H), 1.14 (s, 3H), 0.91 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 213.6, 153.3, 145.9, 125.4, 122.6, 57.9, 48.4, 47.9, 42.6, 42.2, 26.6, 25.0, 19.5, 19.4.

HRMS (ESI): [M+Na]<sup>+</sup> calcd. for  $C_{16}H_{19}NNaO_6S^+, 376.0825; found, 376.0836.$  M.p. 103.6 – 105.4  $^{\circ}C$ 



N-(((1R,4aS,10aR)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl)methyl)-4-nitrobenzamide (**6**)<sup>3</sup>

<sup>1</sup>**H NMR (400 MHz, CDCl**<sub>3</sub>)  $\delta$  8.23 (d, J = 8.8 Hz, 2H), 7.88 (d, J = 8.8 Hz, 2H), 7.17 (d, J = 8.2 Hz, 1H), 7.01 – 6.98 (m, 1H), 6.89 (s, 1H), 6.33 (t, J = 6.1 Hz, 1H), 3.47 (dd, J = 13.7, 6.5 Hz, 1H), 3.32 (dd, J = 13.7, 6.5 Hz, 1H), 3.01 – 2.89 (m, 1H), 2.87 – 2.76 (m, 2H), 2.31 (d, J = 12.8 Hz, 1H), 2.01 – 1.90 (m, 1H), 1.84 – 1.74 (m, 2H), 1.73 – 1.65 (m, 1H), 1.56 – 1.45 (m, 2H), 1.42 – 1.33 (m, 2H), 1.25 – 1.19 (m, 9H), 1.02 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.7, 149.4, 146.8, 145.7, 140.3, 134.5, 128.0, 126.9, 124.1, 123.9, 123.7, 50.5, 45.7, 38.2, 37.7, 37.5, 36.4, 33.3, 30.3, 25.4, 23.9, 23.9, 19.0, 18.8, 18.5. HRMS (ESI): [M+H]<sup>+</sup> calcd. for C<sub>27</sub>H<sub>35</sub>N<sub>2</sub>O<sub>3</sub><sup>+</sup>, 435.2642; found, 435.2648.

M.p. 101.8 – 103.6 °C

#### **5.2 Characterization of Products**



*N*-phenylacetamide  $(2a)^4$ 

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. nitrobenzene (0.5 mmol, 61.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2a** as a **light yellow soild** (64.8 mg, 96%).

<sup>1</sup>**H NMR (400 MHz, CDCl**<sub>3</sub>) δ 8.23 (s, 1H), 7.51 (d, *J* = 7.7 Hz, 2H), 7.27 (t, *J* = 7.8 Hz, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 2.12 (s, 3H).

#### <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 169.0, 138.0, 128.8, 124.2, 120.1, 24.3.

M.p. 113.4 - 115.2 °C



N-(p-tolyl)acetamide (**2b**)<sup>4</sup>

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-methyl-4-nitrobenzene (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2b** as a **light yellow soild** (70.8 mg, 95%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)** δ 7.88 (s, 1H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.08 (d, *J* = 8.2 Hz, 2H), 2.30 (s, 3H), 2.12 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.7, 135.4, 133.8, 129.3, 120.2, 24.2, 20.8. M.p. 146.2 – 148.0 °C

*N*-(*m*-tolyl)a cetamide  $(2c)^4$ 

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-methyl-3-nitrobenzene (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2c** as a **light yellow liquid** (73.8 mg, 99%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.18 (s, 1H), 7.36 (s, 1H), 7.30 (d, *J* = 8.1 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 1H), 6.90 (d, *J* = 7.5 Hz, 1H), 2.28 (s, 3H), 2.13 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.9, 138.6, 137.9, 128.6, 124.9, 120.7, 117.2, 24.3, 21.3.

*N*-(*o*-tolyl)acetamide  $(2d)^4$ 

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-methyl-2-nitrobenzene (0.5 mmol, 68.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via

flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2d** as a **light** yellow soild (67.1 mg, 90%).

<sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.60 (d, J = 8.1 Hz, 1H), 7.42 (s, 1H), 7.15 (d, J = 4.5 Hz, 2H), 7.06 (t, J = 7.3 Hz, 1H), 2.20 (s, 3H), 2.13 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.7, 135.5, 130.4, 130.1, 126.4, 125.4, 123.9, 23.9, 17.7. M.p. 106.2 – 108.0 °C

N-(4-(dimethylamino)phenyl)acetamide (2e)<sup>5</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. *N*, *N*-dimethyl-4-nitroaniline (0.5 mmol, 83.0 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2e** as a **light yellow soild** (71.3 mg, 80%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)** δ 7.63 (s, 1H), 7.32 (d, *J* = 9.0 Hz, 2H), 6.67 (d, *J* = 9.0 Hz, 2H), 2.89 (s, 6H), 2.10 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.5, 147.8, 127.9, 122.1, 113.0, 40.9, 24.1. M.p. 102.0 – 104.0 °C

MeS

N-(4-(methylthio)phenyl)acetamide (2f)<sup>6</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. methyl(4-nitrophenyl)sulfane (0.5 mmol, 84.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2f** as a **light yellow soild** (88.7 mg, 98%).

<sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.23 (s, 1H), 7.41 (d, *J* = 8.6 Hz, 2H), 7.17 (d, *J* = 8.6 Hz, 2H), 2.43 (s, 3H), 2.11 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.9, 135.5, 133.4, 127.7, 120.8, 24.2, 16.5.

M.p. 124.8 - 126.5 °C



N-(4-methoxyphenyl)a cetamide (**2g**)<sup>4</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-methoxy-4-nitrobenzene (0.5 mmol, 76.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2g** as a **light yellow soild** (80.9 mg, 98%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.20 (s, 1H), 7.38 (d, J = 9.0 Hz, 2H), 6.80 (d, J = 9.0 Hz, 2H), 3.75 (s, 3H), 2.09 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.9, 156.2, 131.1, 122.0, 113.9, 55.3, 23.9. M.p. 133.3 – 135.1 °C

N-(4-phenoxyphenyl)acetamide (2h)<sup>7</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-nitro-4-phenoxybenzene (0.5 mmol, 107.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2h** as a **light yellow soild** (109.0 mg, 96%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.35 (s, 1H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.95 (t, *J* = 9.0 Hz, 4H), 2.15 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 169.0, 157.3, 153.3, 133.5, 129.6, 123.0, 121.9, 119.3, 118.3, 24.1. M.p. 128.2 – 129.8 °C

TsO

4-acetamidophenyl4-methylbenzenesulfonate (2i)<sup>8</sup>

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 4-nitrophenyl 4-methylbenzenesulfonate (0.5 mmol, 146.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2i** as a **light yellow soild** (143.4 mg, 94%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.41 (s, 1H), 7.67 (d, J = 8.1 Hz, 2H), 7.46 (d, J = 8.7 Hz, 2H), 7.30 (d, J = 8.1 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 2.43 (s, 3H), 2.11 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 169.0, 145.6, 145.1, 137.1, 131.8, 129.7, 128.3, 122.5, 120.6, 24.2, 21.5.

M.p. 145.1 - 147.0 °C

N-(4-(trifluoromethyl)phenyl)a cetamide (2j)<sup>4</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-nitro-4-(trifluoromethyl)benzene (0.5 mmol, 95.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **2j** as a **light yellow soild** (94.4 mg, 93%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)** δ 7.71 (s, 1H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.55 (d, *J* = 8.6 Hz, 2H), 2.20 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  168.8, 140.9, 126.2 (d, J = 3.4 Hz), 125.9, 124.1 (q, J = 271.5 Hz), 119.4, 24.6.

M.p. 149.2 – 151.0 °C



N-(4-acetylphenyl)acetamide (2k)<sup>9</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-(4-nitrophenyl)ethan-1-one (0.5 mmol, 82.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **2k** as a **light yellow soild** (83.2 mg, 94%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.44 (s, 1H), 7.89 (d, J = 8.6 Hz, 2H), 7.64 (d, J = 8.4 Hz, 2H), 2.56 (s, 3H), 2.20 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 197.4, 169.1, 142.7, 132.6, 129.6, 118.9, 26.4, 24.6.

M.p. 167.1 – 169.0 °C



N-(4-fluorophenyl)acetamide (21)<sup>4</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-fluoro-4-nitrobenzene (0.5 mmol, 70.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **21** as a **light yellow soild** (73.5 mg, 96%).

<sup>1</sup>**H NMR (400 MHz, CDCl**<sub>3</sub>)  $\delta$  7.88 (s, 1H), 7.51 – 7.39 (m, 2H), 6.97 (t, *J* = 8.6 Hz, 2H), 2.13 (s, 3H). <sup>13</sup>**C NMR (101 MHz, CDCl**<sub>3</sub>)  $\delta$  168.6, 159.3 (d, *J* = 243.3 Hz), 133.9, 121.9 (d, *J* = 7.9 Hz), 115.5 (d, *J* = 22.5 Hz), 24.3.

M.p. 151.8-153.6 °C

N-(3-fluorophenyl)acetamide (2m)<sup>10</sup>

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-fluoro-3-nitrobenzene (0.5 mmol, 70.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **2m** as a **light yellow soild** (74.2 mg, 97%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.60 (s, 1H), 7.48 (d, *J* = 11.0 Hz, 1H), 7.24 - 7.15 (m, 2H), 6.88 - 6.66 (m, 1H), 2.15 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDC<sub>3</sub>) δ 169.3, 162.8 (d, *J* = 244.3 Hz), 139.6 (d, *J* = 10.8 Hz), 129.9 (d, *J* = 9.3 Hz), 115.3 (d, *J* = 2.4 Hz), 110.8 (d, *J* = 21.3 Hz), 107.4 (d, *J* = 26.1 Hz), 24.3. M.p. 84.2 – 86.1 °C



*N*-(2-fluorophenyl)acetamide  $(2n)^{11}$ 

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-fluoro-2-nitrobenzene (0.5 mmol, 70.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash

column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **2n** as a **light** yellow soild (71.9 mg, 94%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.22 (t, J = 7.7 Hz, 1H), 7.66 (s, 1H), 7.14 – 6.96 (m, 3H), 2.19 (s, 3H). <sup>13</sup>**C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  168.5, 152.4 (d, J = 243.1 Hz), 126.2 (d, J = 10.3 Hz), 124.4 (d, J = 3.7 Hz), 124.3, 122.0, 114.7 (d, J = 19.3 Hz), 24.4. M.p. 75.0 – 76.9 °C

N-(4-chlorophenyl)acetamide (20)<sup>4</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-chloro-4-nitrobenzene (0.5 mmol, 78.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **20** as a **light yellow soild** (83.6 mg, 99%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)** δ 7.45 (d, *J* = 8.7 Hz, 2H), 7.34 (s, 1H), 7.27 (d, *J* = 7.8 Hz, 2H), 2.17 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.3, 136.4, 129.3, 129.0, 121.1, 24.6. M.p. 177.8 – 178.9 °C

N-(4-bromophenyl)acetamide (**2p**)<sup>4</sup>

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-bromo-4-nitrobenzene (0.5 mmol, 100.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **2p** as a **light yellow soild** (104.3 mg, 98%).

<sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.47 (s, 1H), 7.44 (d, J = 9.4 Hz, 4H), 2.20 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  168.4, 136.9, 131.9, 121.4, 116.8, 24.6. M p. 168.5 – 170.4 °C

M.p. 168.5 – 170.4 °C

N-([1,1'-biphenyl]-2-yl)acetamide (2q)<sup>12</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 2-nitro-1,1'-biphenyl (0.5 mmol, 99.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2q** as a **light yellow soild** (96.0 mg, 91%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.22 (d, *J* = 8.1 Hz, 1H), 7.55 – 7.32 (m, 6H), 7.14 –7.25 (m, 3H), 2.00 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.2, 138.1, 134.6, 132.3, 130.0, 129.1, 129.0, 128.3, 127.8, 124.3, 121.8, 24.4.

M.p. 112.6 - 114.6 °C



N-(naphthalen-1-yl)acetamide(2r)<sup>13</sup>

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-nitronaphthalene (0.5 mmol, 86.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2r** as a **light yellow soild** (81.4 mg, 88%).

<sup>1</sup>**H NMR (400 MHz, DMSO)** δ 9.94 (s, 1H), 8.11 (d, *J* = 7.7 Hz, 1H), 7.96 – 7.90 (m, 1H), 7.73 (t, *J* = 6.8 Hz, 2H), 7.60 – 7.43 (m, 3H), 2.21 (s, 3H).

<sup>13</sup>C NMR (101 MHz, DMSO) δ 169.0, 133.7, 128.1, 127.7, 126.0, 125.7, 125.6, 125.0, 122.7, 121.5, 39.5, 23.5.

M.p. 125.1 - 127.0 °C



*N*-(9*H*-fluoren-3-yl)acetamide(2s)

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 3-nitro-9*H*-fluorene (0.5 mmol, 105.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2s** as a **light yellow soild** (107.1 mg, 96%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.81 (s, 1H), 7.78 (s, 1H), 7.70 – 7.61 (m, 2H), 7.46 (d, J = 7.4 Hz, 1H), 7.36 – 7.30 (m, 2H), 7.24 (t, J = 7.2 Hz, 1H), 3.80 (s, 2H), 2.17 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.6, 144.2, 143.1, 141.2, 138.0, 136.7, 126.7, 126.2, 124.9, 120.0, 119.4, 118.8, 117.0, 36.9, 24.5.

HRMS (ESI):  $[M+H]^+$  calcd. for  $C_{15}H_{14}NO^+$ , 224.1070; found, 224.1085. M.p. 189.1 – 191.0 °C

N-(benzo[d][1,3]dioxol-5-yl)acetamide(2t)<sup>14</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 5-nitrobenzo[*d*][1,3]dioxole (0.5 mmol, 83.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **2t** as a **white soild** (86.0 mg, 96%).

<sup>1</sup>**H NMR (400 MHz, CDCl**<sub>3</sub>) δ 8.07 (s, 1H), 7.15 (s, 1H), 6.77 (d, *J* = 7.5 Hz, 1H), 6.68 (d, *J* = 7.9 Hz, 1H), 5.90 (s, 2H), 2.09 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.6, 147.6, 144.2, 132.1, 113.4, 107.9, 103.1, 101.2, 24.2. M.p. 134.0 – 135.8 °C

N-(1H-indol-5-yl)acetamide (2u)

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 5-nitro-1*H*-indole (0.5 mmol, 81.0 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 2/1, volume ratio) to give the titled product **2u** as a **light yellow soild** (80.1 mg, 92%).

<sup>1</sup>**H NMR (400 MHz, DMSO)** δ 10.95 (s, 1H), 9.69 (s, 1H), 7.84 (d, *J* = 1.2 Hz, 1H), 7.34 – 7.25 (m, 2H), 7.19 – 7.14 (m, 1H), 6.35 (s, 1H), 2.02 (s, 3H).

 $\label{eq:stars} \begin{array}{l} {}^{13}\text{C NMR} \mbox{ (101 MHz, DMSO) } \delta \mbox{ 168.1, 133.0, 131.8, 127.9, 126.2, 115.2, 111.5, 111.0, 101.5, 24.4. } \\ \\ \text{HRMS} \mbox{ (ESI): } [M+H]^+ \mbox{ calcd. for } C_{10} H_{11} N_2 O^+, 175.0866; \mbox{ found, 175.0882. } \\ \\ \text{M.p. 99.1} - \mbox{ 101.0 } ^\circ \mbox{C} \end{array}$ 



#### *N*-(quinolin-8-yl)acetamide $(2\mathbf{v})^{15}$

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 8-nitroquinoline (0.5 mmol, 87.0 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2v** as a **light yellow soild** (60.5 mg, 65%).

<sup>1</sup>**H NMR (400 MHz, CDCl**<sub>3</sub>) δ 9.76 (s, 1H), 8.91 – 8.63 (m, 2H), 8.13 – 8.07 (m,, 1H), 7.52 – 7.43 (m, 2H), 7.43 – 7.37 (m, 1H), 2.32 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.8, 148.1, 138.2, 136.3, 134.5, 127.9, 127.4, 121.6, 121.4, 116.4, 25.1.

 $M.p.\,99.7-101.7\,{}^{\circ}C$ 

N-(2-(1H-indol-1-yl)phenyl)a cetamide (2w)

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-(2-nitrophenyl)-1*H*-indole (0.5 mmol, 119.0 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2w** as a **light yellow soild** (113.8 mg, 91%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.38 (d, *J* = 8.1 Hz, 1H), 7.73 – 7.66 (m, 1H), 7.50 – 7.39 (m, 1H), 7.28 (d, *J* = 6.7 Hz, 1H), 7.24 – 7.14 (m, 4H), 7.09 – 7.03 (m, 1H), 6.90 (s, 1H), 6.73 (d, *J* = 3.2 Hz, 1H), 1.84 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.4, 136.5, 134.3, 128.9, 128.6, 128.5, 128.4, 127.9, 124.5, 122.8, 122.2, 121.2, 120.7, 110.3, 104.3, 24.5.

HRMS (ESI):  $[M+H]^+$  calcd. for  $C_{16}H_{15}N_2O^+, 251.1179;$  found, 251.1194. M.p. 139.6 – 141.5  $^\circ C$ 

N, N'-(1,3-phenylene)diacetamide  $(2\mathbf{x})^8$ 

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1, 3-dinitrobenzene (0.5 mmol, 84.0 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/2, volume ratio) to give the titled product **2x** as a **light yellow soild** (78.8 mg, 82%).

<sup>1</sup>**H NMR (400 MHz, DMSO)** δ 9.88 (s, 2H), 7.85 (s, 1H), 7.26 – 7.21 (m, 2H), 7.18 – 7.10 (m, 1H), 1.99 (s, 6H).

<sup>13</sup>C NMR (101 MHz, DMSO) δ 168.3, 139.6, 128.8, 113.9, 109.9, 24.0.

M.p. 178.4 - 180.4 °C

N, N'-([1,1'-biphenyl]-2,2'-diyl)diacetamide (2y)<sup>16</sup>

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 2, 2'-dinitro-1,1'-biphenyl (0.5 mmol, 122.0 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/2, volume ratio) to give the titled product **2y** as a **light yellow soild** (87.2 mg, 65%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.90 (d, J = 8.0 Hz, 2H), 7.44 – 7.33 (m, 2H), 7.30 (s, 2H), 7.20 (t, J = 7.4 Hz, 2H), 7.13 (d, J = 7.6 Hz, 2H), 1.87 (s, 6H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 169.4, 135.4, 130.5, 130.2, 129.1, 125.4, 124.0, 23.9. M.p. 154.5 – 156.5 °C

*N*-propylacetamide (**2aa**)

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-nitropropane (0.5 mmol, 44.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column

chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2aa** as a **light yellow liquid** (25.3 mg, 50%).

<sup>1</sup>**H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  6.63 (s, 1H), 3.09 – 3.03 (m, 2H), 1.86 (s, 3H), 1.47 – 1.35 (m, 2H), 0.80 (t, *J* = 7.4 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 170.3, 41.1, 22.8, 22.5, 11.1.

° ↓ ↓

N-(*tert*-butyl)a cetamide (**2ab**)<sup>17</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 2-methyl-2-nitropropane (0.5 mmol, 51.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2ab** as a **light yellow soild** (39.1 mg, 68%).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 5.72 (s, 1H), 1.84 (s, 3H), 1.27 (s, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 169.5, 50.9, 28.6, 24.3.

 $M.p. 92.8 - 94.6 \ ^{\circ}C$ 

N-cyclohexylacetamide (**2ac**)<sup>13</sup>

General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. Nitrocyclohexane (0.5 mmol, 64.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **2ac** as a **light yellow soild** (60.0 mg, 85%).

<sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  5.49 (s, 1H),3.78– 3.66 (m, 1H), 1.93 (s, 3H), 1.89 (dd, J = 12.5, 3.3 Hz, 2H), 1.73 – 1.65 (m, 2H), 1.63 – 1.56 (m, 1H), 1.40– 1.27 (m, 2H), 1.20– 1.03 (m, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  169.2, 48.2, 33.1, 25.5, 24.9, 23.5. M.p. 104.2 – 106.1 °C



4-acetamidophenyl((1*S*,4*R*)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methanesulfonate(**5**) General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1ad** (0.5 mmol, 176.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product **5** as a **light yellow liquid** (127.8 mg, 70%).

<sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.25 (s, 1H), 7.51 (d, J = 9.0 Hz, 2H), 7.17 (d, J = 9.0 Hz, 2H), 3.75 (d, J = 15.0 Hz, 1H), 3.17 (d, J = 15.0 Hz, 1H), 2.54 – 2.34 (m, 2H), 2.15 – 2.08 (m, 4H), 2.08 – 2.00 (m, 1H), 1.94 (d, J = 18.6 Hz, 1H), 1.77 – 1.65 (m, 1H), 1.48 – 1.39 (m, 1H), 1.11 (s, 3H), 0.87 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  214.1, 169.0, 144.6, 137.2, 122.4, 121.0, 58.0, 47.9, 47.3, 42.6, 42.3, 26.7, 25.0, 24.2, 19.7, 19.6.

HRMS (ESI):  $[M+Na]^+$  calcd. for  $C_{18}H_{23}NNaO_5S^+$ , 388.1189; found, 388.1203.



5-acetamido-N-(((1R,4aS,10aR)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl)methyl)benzamide (7)

General Procedure was followed with Rh<sub>2</sub>(CO)<sub>4</sub>Cl<sub>2</sub> (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)<sub>6</sub> (1 mmol, 351.9 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. **1ae** (0.5 mmol, 217.1 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe. Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/2, volume ratio) to give the titled product **7** as a **light yellow soild** (212.0 mg, 95%).

<sup>1</sup>**H NMR (400 MHz, CDCl**<sub>3</sub>)  $\delta$  9.23 (s, 1H), 7.65 (d, J = 8.3 Hz, 2H), 7.58 (d, J = 8.3 Hz, 2H), 7.19 (d, J = 8.1 Hz, 1H), 7.01 (d, J = 7.8 Hz, 1H), 6.89 (s, 1H), 6.64 (s, 1H), 3.39 (d, J = 5.5 Hz, 2H), 2.98 – 2.78 (m, 3H), 2.31 (d, J = 12.1 Hz, 1H), 2.14 (s, 3H), 1.97 (s, 1H), 1.83 – 1.65 (m, 3H), 1.53 (t, J = 14.2 Hz, 3H), 1.47 – 1.37 (m, 2H), 1.24 (s, 6H), 1.23 (s, 3H), 1.01 (s, 3H).

 $\label{eq:started_st$ 

**E**tC

N-(4-ethoxyphenyl)acetamide (3)<sup>18</sup>

(a) General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (2 mol%, 3.9 mg), DPPP (6 mol%, 12.4 mg), NaI (30 mol%, 22.5 mg), W(CO)\_6 (1 mmol, 351.9 mg), Na\_3PO\_4 (0.75 mmol, 123.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-ethoxy-4-nitrobenzene (0.5 mmol, 83.5 mg), H<sub>2</sub>O (1 mmol, 18.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product Phenacetin **3** as a **light yellow soild** (88.6 mg, 99%).

(b) General Procedure was followed with  $Rh_2(CO)_4Cl_2$  (1 mol%, 3.9 mg), DPPP (3 mol%, 12.4 mg), NaI (30 mol%, 45 mg), W(CO)<sub>6</sub> (1 mmol, 703.8 mg), Na<sub>3</sub>PO<sub>4</sub> (0.75 mmol, 246.0 mg) were added to an oven-dried tube (15 mL), which was then placed under vacuum and refilled with nitrogen for three times. 1-ethoxy-4-nitrobenzene (1.0 mmol, 167.0 mg), H<sub>2</sub>O (2 mmol, 36.0 mg), and DMC (1.5 mL) were added into the tube via syringe.Upon completion the mixture was concentrated and purified via flash column chromatography (PE/EA = 5/1 to 1/1, volume ratio) to give the titled product Phenacetin **3** as a **light yellow soild** (164.8 mg, 92%).

<sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.96 (s, 1H), 7.36 (d, J = 8.9 Hz, 2H), 6.79 (d, J = 8.9 Hz, 2H), 3.97 (q, J = 7.0 Hz, 2H), 2.09 (s, 3H), 1.37 (t, J = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.9, 155.5, 131.0, 122.0, 114.5, 63.5, 23.9, 14.7. M.p. 132.6 – 134.5 °C

#### 6. References

(1) K. A. Perez, C. R. Rogers and E. A. Weiss, Quantum dot-catalyzed photoreductive removal of sulfonyl-based protecting groups, *Angew. Chem. Int. Ed.*, 2020, **59**, 14091-14095.

(2) T. U. Thikekar and C.-M. Sun, Palladium-catalyzed regioselective synthesis of 1,2-fused indole-diazepines via [5+2] annulation of *o*-Indoloanilines with alkynes, *Adv. Synth. Catal.*, 2017, **359**, 3388-3396.

(3) Y. Gao, Y. Mao, B. Zhang, Y. zhan and Y. Huo, Regioselective nitration of anilines with  $Fe(NO_3)_3 \cdot 9H_2Oas$  promoter and nitro source, *Org. Biomol. Chem.*, 2018, **16**, 3881-3884.

(4) L. Tang, J. H. Matuska, Y.-H. Huang, Y.-H. He and Z. Guan, Amide synthesis from thiocarboxylic acids and amines by spontaneous reaction and electrosynthesis, *ChemSusChem*, 2019, **12**, 2570-2575.

(5) J. K. Augustine, R. Kumar, A. Bombrun and A. B. Mandala, An efficient catalytic method for the Beckmann rearrangement of ketoximes to amides and aldoximes to nitriles mediated by propylphosphonic anhydride, *Tetrahedron Lett.*, 2011, **52**, 1074-1077.

(6) D. Antonow, T. Marrafa, I. Dawood, T. Ahmed, M. R. Haque, D. E. Thurston and G. Zinzalla, Facile oxidation of electron-poor benzo[*b*]thiophenes to the corresponding sulfones with an aqueous solution of  $H_2O_2$  and  $P_2O_5$ , *Chem. Commun.*, 2010, **46**, 2289-2291.

(7) N. Jakilian, E. E. Ishikawa, L. F. Silva Jr. and B. Olofsson, Room temperature, metal-free synthesis of diarylethers with use of diaryliodonium salts, *Org. Lett.*, 2011, **13**, 1552-1555.

(8) H. Tsukamoto, R. Suzuki and Y. Kondo, Revisiting benzenesulfonyl linker for the deoxygenation and multifunctionalization of phenols, *J. Comb. Chem.*, 2006, **8**, 289-292.

(9) J. Ruan, J. A. Iggo, N. G. Berry and J. Xiao, Hydrogen-bonding-promoted oxidative addition and regioselective arylation of olefins with aryl chlorides, *J. Am. Chem. Soc.*, 2010, **132**, 16689-16699.

(10) J. R. Heys and C. S. Elmore, *Meta*-substituent effects on organoiridium-catalyzed *ortho*-hydrogen isotope exchange, *J. Label. Comp. Radiopharm.*, 2009, **52**, 189-200.

(11) A. Ramanathan and L. S. Jimenez, Reductive dehalogenation of aryl bromides and chlorides and their use as aryl blocking groups, *Synthesis*, 2010, 217-220.

(12) S. Yang, B. Li, X. Wan and Z. Shi, *Ortho* arylation of acetanilides via Pd(II)-catalyzed C-H functionalization, *J. Am. Chem. Soc.*, 2007, **129**, 6066-6067.

(13) C. Cazorla, E. Métay and M. Lemaire, Oxidative nucleophilic substitution: transformation of alkylboronic derivatives, *Tetrahedron*, 2011, **45**, 8615-8621.

(14) M. A. Elban, W. Sun, B. M. Eisenhauer, R. Gao and S. M. Hecht, Synthesis and biological evaluation of 10,11-methylenedioxy-14-azacamptothecin, *Org. Lett.*, 2006, **8**, 3513-3516.

(15) T. Moriuchi-Kawakami, K. kawka, S. Nakamura, Y. Koyama and Y. Shibutani, Design of bisquinolinyl malonamides as  $Zn^{2+}$  ion-selective fluoroionophores based on the substituent effect, *Tetrahedron*, 2014, **70**, 9805-9813.

(16) G. U. Chaturbhuj and K. G. Akamanchi, Copper catalyzed Gomberg-Buchmann-Hey reaction using aryldiazonium tosylate, *Tetrahedron Lett.*, 2011, **52**, 4950-4953.

(17) K. V. Katkar, P. S. Chaudhari and K. G. Akamanchi, Sulfated tungstate: an efficient catalyst for the Ritter reaction, *Green Chem.*, 2011, **13**, 835-838.

(18) H. Liu, X. Wang and Y. Gu, Direct acetoxylation and etherification of anilides using phenyliodine bis(trifluoroacetate), *Org. Biomol. Chem.*, 2011, **9**, 1614-1620.

## 7.1 Copy of <sup>1</sup>H and <sup>13</sup>C NMR Spectra of 1i, 1w, 4 and 6





## 

<sup>1</sup>H NMR 400 MHz, CDCl<sub>3</sub>









250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)







### 7.2 Copy of <sup>1</sup>H and <sup>13</sup>C NMR Spectra of Products



$$-7.38$$
  
 $-7.33$   
 $-7.37$   
 $-7.07$   
 $-2.30$   
 $-2.30$ 





---2.28 ---2.13





~2.20















<sup>13</sup>C NMR 101 MHz, CDCl<sub>3</sub>



2h



-24.1





$$-8.44$$
  
 $-8.44$   
 $-2.56$ 









---2.19











-24.6











---2.00



S42



S43



-2.17

--3.80

#### <sup>1</sup>H NMR 400 MHz, CDCl<sub>3</sub>

)0



110 100 90 80 f1 (ppm) 130 120 ( 



<sup>1</sup>H NMR 400 MHz, CDCl<sub>3</sub>



90 80 70

60

40

50

20

30

10

140 130 120 110 100 f1 (ppm)

200 190

180 170

160 150













<sup>1</sup>H NMR 400 MHz, CDCl<sub>3</sub>

--6.63





-1.27

-5.72







—169.2







$$-8.25$$
  
 $< 7.52$   
 $< 7.18$   
 $< 7.16$ 







S55