

## Electronic Supporting Information

### Guanidine Catalyzed Aerobic Reduction: A Selective Reduction of Olefins using Aqueous Hydrazine

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#### General experimental

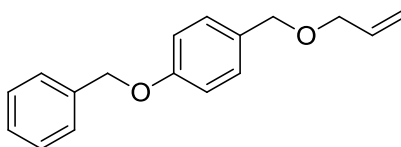
NMR spectra were recorded on a JEOL LA-400, BRUKER-AV400 spectrometer in CDCl<sub>3</sub>, Tetramethylsilane (TMS;  $\delta = 0.00$  ppm) served as internal standards for <sup>1</sup>H NMR. The corresponding residual non-deuterated solvent signal (CDCl<sub>3</sub>:  $\delta = 77.00$  ppm) was used as internal standards for <sup>13</sup>C NMR. IR spectra were measured using a JASCO FT/IR-410 spectrometer, and Perkin-Elmer FT/IR Spectrum BX, GX. Mass spectra were measured with Micromass Q-ToF (ESI-HRMS). Column chromatography was conducted on Silica gel 230-400 mesh (Merck) and preparative thin-layer chromatography was carried out using SILICA GEL GF-254. Aq hydrazine (hydrazine monohydrate) was purchased from sigma Aldrich composition: N<sub>2</sub>H<sub>4</sub>, 64-65 %, reagent grade, assay: 98%, Batch # 24696JK) and guanidine nitrate was is obtained from SD Fine-Chem Ltd, Mumbai, India, (assay: 98%, Batch no: BO8X/0708/0502/13).

#### Typical experimental procedure

To a stirred solution of guanidine nitrate (12 mg, 0.1 mmol) and olefin (**3a**, 148 mg, 1 mmol) in ethanol (2 mL) was added NH<sub>2</sub>NH<sub>2</sub>·H<sub>2</sub>O (100 mg, 2 mmol). The reaction was stirred at room temperature in the presence of oxygen (1atm). Ethanol was removed under reduced pressure and extracted with dichloromethane (20 mL x 3) and dried over Na<sub>2</sub>SO<sub>4</sub>, solvent was removed under reduced pressure to afford the reduced product **3b** (135 mg, 90%).

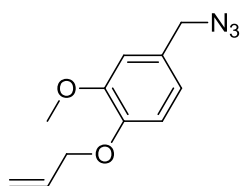
## Characterization data for selected olefins

### 1-((allyloxy)methyl)-4-(benzyloxy)benzene (4a)<sup>1</sup>:



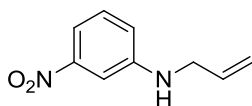
Pale yellow liquid; yield- 80%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3066, 2857, 1612, 1512, 1242, 1080, 738; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.42-7.25 (m, 7H), 6.94 (d,  $J = 8.8$  Hz, 2H), 5.97-5.90 (m, 1H), 5.31-5.26 (m, 1H), 5.20-5.17 (m, 1H), 5.04 (s, 2H), 4.44 (s, 2H), 4.0-3.98 (m, 2H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  158.3, 137.0, 134.8, 130.6, 129.3, 128.5, 127.9, 127.4, 117.0, 114.7, 71.7, 70.8, 69.9; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{17}\text{H}_{18}\text{O}_2$  ( $M + \text{Na}$ ): 277.1204, found ( $M + \text{Na}$ ): 277.1201.

### 1-(allyloxy)-4-(azidomethyl)-2-methoxybenzene (8a)<sup>2</sup>:



Colorless liquid; Yield – 92%; **IR** (Neat,  $\text{cm}^{-1}$ ): 2099; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400MHz):  $\delta$  6.81-6.88 (m, 3H), 6.03-6.13 (m, 1H), 5.41 (dd,  $J_1 = 13.8$  Hz,  $J_2 = 1.2$  Hz, 1H), 5.29 (dd,  $J_1 = 13.8$  Hz,  $J_2 = 1.2$  Hz, 1H), 4.62 (d,  $J = 5.2$  Hz, 2H), 4.27 (s, 2H), 3.89 (s, 3H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$  149.6, 148.1, 133.1, 128.2, 120.6, 118.1, 113.3, 111.7, 69.8, 55.9, 54.7; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{11}\text{H}_{13}\text{N}_3\text{O}_2$  ( $M + \text{Na}$ ): 242.0905, found ( $M + \text{Na}$ ): 242.0902.

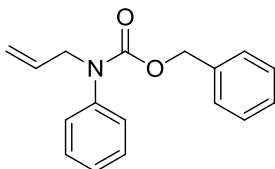
### N-allyl-3-nitroaniline (9a)<sup>3</sup>:



Pale yellow liquid; yield - 96%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3408, 2922, 1619, 1542, 1340, 733; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400MHz):  $\delta$  7.53-7.50 (m, 1H), 7.40 (s, 1H), 7.29-7.24 (m, 1H), 6.87 (dd,  $J_1 = 8.1$ Hz,  $J_2 = 1.8$ Hz, 1H), 5.97- 5.88 (m, 1H), 5.32-5.20(m, 2H), 4.19(s, br, 1H), 3.84(s, 2H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$

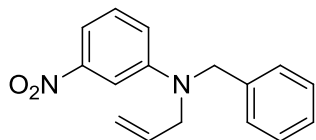
149.4, 148.7, 134.04, 118.82, 116.93, 112.01, 106.52, 46.14, 30.89; **HRESI-MS** ( $m/z$ ): Calculated for  $C_9H_{10}N_2O_2$  ( $M + H$ ): 179.0821, found ( $M + H$ ): 179.0831.

**benzyl allyl(phenyl)carbamate (10a)<sup>4</sup>:**



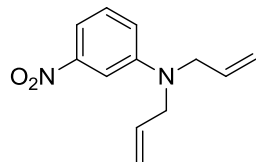
Brown liquid, Yield-96%; **IR** (Neat,  $cm^{-1}$ ): 1705, 1496, 1398, 697; **<sup>1</sup>H NMR** ( $CDCl_3$ , 400 MHz):  $\delta$  7.51-7.00 (m, 10H), 5.94-5.85 (m, 1H), 5.15-5.10 (m, 4H), 4.26 (d,  $J = 5.6$  Hz, 2H); **<sup>13</sup>C NMR** ( $CDCl_3$ , 100 MHz): 155.1, 136.5, 133.6, 128.7, 128.3, 127.8, 127.5, 126.4, 67.1, 53.2; **HRESI-MS** ( $m/z$ ): Calculated for  $C_{17}H_{17}NO_2$  ( $M + Na$ ): 290.1157, found ( $M + Na$ ): 290.1157.

**N-allyl-N-benzyl-3-nitroaniline (25a):**



Brown liquid; yield-55%; **IR** (Neat,  $cm^{-1}$ ): 3086, 2865, 1526, 1347, 1237, 733; **<sup>1</sup>H NMR** ( $CDCl_3$ , 400 MHz):  $\delta$  7.53-7.48 (m, 2H), 7.35-7.31 (m, 2H), 7.27-7.20 (m, 4H), 6.93 (d,  $J = 8.4$  Hz, 1H), 5.91-5.82 (m, 1H), 5.25-5.18 (m, 2H), 4.60 (s, 2H), 4.07 (d,  $J = 3.6$  Hz, 2H); **<sup>13</sup>C NMR** ( $CDCl_3$ , 100 MHz):  $\delta$  149.5, 149.4, 137.4, 132.2, 129.6, 128.8, 127.3, 126.4, 117.9, 117.0, 111.0, 106.5, 54.1, 53.3; **HRESI-MS** ( $m/z$ ): Calculated for  $C_{16}H_{16}N_2O_2$  ( $M + H$ ): 269.1290, found ( $M + H$ ): 269.1284.

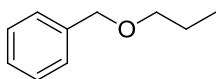
**N,N-diallyl-3-nitroaniline (26a)<sup>5</sup>**



Pale yellow liquid; yield - 60%; **IR** (Neat,  $cm^{-1}$ ): 3085, 2982, 2867, 1619, 1526, 1495, 1391, 1347, 1238, 1181, 922, 734, 669; **<sup>1</sup>H NMR** ( $CDCl_3$ , 400 MHz):  $\delta$  7.49-7.47 (m, 2H), 7.30-7.26 (m, 1H), 6.94-6.91 (m, 1H), 5.89-5.80 (m, 2H), 5.22-5.15 (m, 4H), 3.99-3.98 (m, 4H); **<sup>13</sup>C NMR** ( $CDCl_3$ , 100 MHz):  $\delta$  149.42, 149.16, 132.56, 129.54, 117.72, 116.63, 110.79, 106.36, 52.92; **HRESI-MS** ( $m/z$ ): Calculated for  $C_{12}H_{14}N_2O_2$  ( $M + Na$ ): 241.0953, found ( $M + Na$ ): 241.0947.

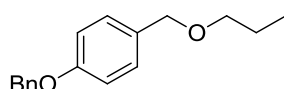
### Characterization data for reduced products:

#### (propoxymethyl)benzene (3b)<sup>6</sup>:



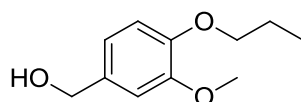
Colorless liquid; Yield - 90%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3065, 2965, 2937, 2877, 1720, 713; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400MHz):  $\delta$  7.35- 7.34(m, 4H), 7.32-7.26 (m, 1H), 4.51 (s, 2H), 3.44 (t,  $J = 6.7\text{Hz}$ , 2H), 1.69-1.60 (m, 2H), 0.94 (t,  $J = 7.6\text{Hz}$ , 3H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100MHz)  $\delta$ :138.69, 128.33, 127.61, 127.45, 72.88, 72.11, 22.95, 10.62; **MS** ( $m/e$ ) 150.

#### 1-(benzyloxy)-4-(propoxymethyl)benzene (4b):



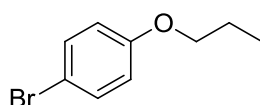
Pale yellow liquid; yield- 92%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3065, 2962, 2859, 1612, 1242, 1096, 820, 696; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.4-7.25 (m, 7H), 6.92(d,  $J = 8.4\text{Hz}$ , 2H), 5.01 (s, 2H), 4.41 (s, 2H), 3.38 (t,  $J = 6.7\text{ Hz}$ , 2H), 1.65-1.56 (m, 2H), 0.91 (t,  $J = 7.4\text{ Hz}$ , 3H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  158.2, 137.0, 131.0, 129.1, 128.4, 127.8, 127.3, 114.6, 72.4, 71.9, 70.0, 22.9, 10.6; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{17}\text{H}_{20}\text{O}_2$  (M + Na): 279.1361, found (M + Na): 279.1357.

#### (3-methoxy-4-propoxyphenyl)methanol (5b):



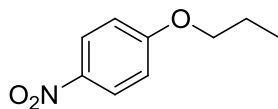
Colorless liquid; yield-85%; **IR** (Neat,  $\text{cm}^{-1}$ ): 2964, 2937, 2877, 1510, 1263, 1234, 1159, 1037, 978, 807; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400MHz ):  $\delta$  6.92(s, 1H), 6.858-6.852 ( m, 2H), 4.60(s, 2H), 3.97(t, 2H,  $J = 7.2\text{Hz}$ ), 3.87(s, 3H), 1.88-1.79(m, 2H), 1.03(t, 3H,  $J = 8\text{Hz}$ ). **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$  149.46, 148.06, 133.47, 119.39, 112.75, 110.86, 70.54, 65.29, 55.90, 22.40, 10.36; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{11}\text{H}_{16}\text{O}_3$  (M + Na): 219.0997, found (M + Na): 219.0995.

#### 1-Bromo-4-propoxybenzene (6b)<sup>7</sup>:



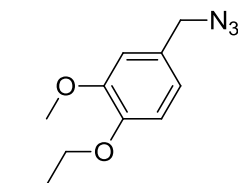
Pale yellow liquid; Yield - 91%; **IR** (Neat,  $\text{cm}^{-1}$ ): 2965, 1489, 1243, 1071, 977, 821;  **$^1\text{H NMR}$**  ( $\text{CDCl}_3$ , 400MHz):  $\delta$  7.34 (d,  $J = 8.9$  Hz, 2H), 6.76 (d,  $J = 8.9$  Hz, 2H), 3.86 (t,  $J = 6.5$ Hz, 2H), 1.83-1.74 (m, 2H), 1.02 (t,  $J = 7.3$ Hz, 3H);  **$^{13}\text{C NMR}$**  ( $\text{CDCl}_3$ , 100MHz):  $\delta$  158.21, 132.14, 116.27, 112.52, 69.7, 22.46, 10.43; Anal.Calcd for  $\text{C}_9\text{H}_{12}\text{O}$ ; C, 50.26; H, 5.15; Found: C, 50.61; H, 5.47.

**1-Nitro-4-propoxybenzene (7b)<sup>8</sup>:**



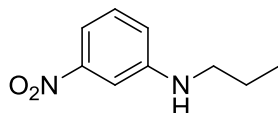
Colorless liquid; Yield - 92%; **IR** (Neat,  $\text{cm}^{-1}$ ): 2970, 1489, 1505, 1338, ;  **$^1\text{H NMR}$**  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$ : 8.19 (d,  $J = 9.2$  Hz, 2H), 6.94 (d,  $J = 9.2$  Hz, 2H), 4.01 (t,  $J = 6.5$ Hz, 2H), 1.90-1.81 (m, 2H), 1.06 (t,  $J = 7.4$ Hz, 3H);  **$^{13}\text{C NMR}$**  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  164.2, 141.2, 125.8, 114.3, 70.3, 22.3, 10.3; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_9\text{H}_{11}\text{NO}_3$  (M + Na): 204.0637, found (M + Na): 204.0632.

**4-(azidomethyl)-2-methoxy-1-propoxybenzene (8b):**



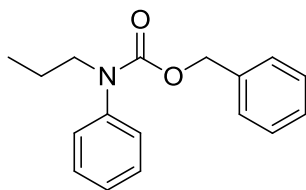
Colorless liquid; Yield - 85%; **IR** (Neat,  $\text{cm}^{-1}$ ):2938, 2100,1515, 1264;  **$^1\text{H NMR}$**  ( $\text{CDCl}_3$ , 400MHz):  $\delta$  6.84-6.83 (m, 3H), 4.25 (s, 2H), 3.97(t,  $J = 6.8$  Hz, 2H ), 3.88 (s, 2H), 1.90-1.81(m, 2H ), 1.03(t,  $J = 7.4$  Hz, 2H );  **$^{13}\text{C NMR}$**  ( $\text{CDCl}_3$ , 100MHz):  $\delta$  149.54, 148.61, 127.64, 120.75, 112.70, 111.78, 70.44, 55.93, 54.71, 22.37, 10.33; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{11}\text{H}_{15}\text{N}_3\text{O}_2$  (M + Na): 244.1062, found (M + Na): 244.1066.

**3-Nitro-N-propylaniline (9b)<sup>9</sup>:**



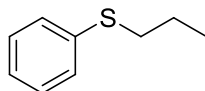
Pale yellow liquid; yield - 92%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3415, 2964, 2876, 1623, 1526, 1350, 1260, 1142, 991, 734;  **$^1\text{H NMR}$**  ( $\text{CDCl}_3$ , 400MHz):  $\delta$  7.748-7.46 (m, 1H), 7.36 (t,  $J = 2.4$  Hz, 1H), 7.26-7.22 (m, 1H), 6.86-6.83(m, 1H), 4.04 (s, br, 1H), 3.12(t,  $J = 6.8$  Hz, 2H), 1.71-1.62(m, 2H), 1.01(t,  $J = 7.2$  Hz, 3H);  **$^{13}\text{C NMR}$**  ( $\text{CDCl}_3$ , 100MHz):  $\delta$  149.38,149.17, 129.55, 118.56, 111.43, 105.97, 45.47, 22.34, 11.45; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_9\text{H}_{12}\text{N}_2\text{O}_2$  (M + Na): 203.0796, found (M + Na): 203.0795.

**Benzyl phenyl(propyl)carbamate (10b)<sup>10</sup>:**



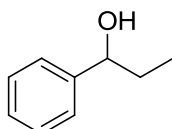
Pale yellow liquid, Yield-99%; **IR** (Neat,  $\text{cm}^{-1}$ ): 2963, 2934, 1704, 1149, 696; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.30-7.11 (m, 10H), 5.06 (s, 2H), 3.56 (t,  $J = 7.5$  Hz, 2H), 1.53-1.43 (m, 2H), 0.86 (t,  $J = 7.6$  Hz, 3H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100 MHz): 155.4, 141.8, 136.8, 128.9, 128.3, 127.7, 127.4, 127.3, 126.6, 66.9, 52.0, 21.5, 11.1; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{17}\text{H}_{19}\text{NO}_2$  (M + H): 270.1494 found (M + H): 270.1494.

**Phenyl 1-propyl sulfide (11b)<sup>11</sup>:**



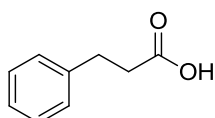
Colorless liquid; Yield - 88%; **IR** (Neat,  $\text{cm}^{-1}$ ): 2962, 1585, 1480, 1091, 737, 690; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.33-7.24 (m, 4H), 7.17-7.13 (m, 1H), 2.89 (t,  $J = 7.3$  Hz, 2H), 1.71-1.62 (m, 2H), 1.02 (t,  $J = 7.3$  Hz, 3H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  136.9, 129.0, 128.9, 125.6, 35.6, 22.5, 13.4; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_9\text{H}_{12}\text{S}$  (M + K): 191.0297, found (M + K): 191.0300.

**1-Phenyl-1-propanol (12b)<sup>12</sup>:**



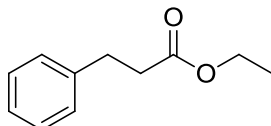
Colorless liquid; Yield - 80%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3453, 3078, 3035, 2978, 2910, 1591, 1455, 917, 753; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400MHz):  $\delta$  7.33-7.24 (m, 5H), 4.56 (t,  $J = 6.4$  Hz, 1H), 2.12 (b, 1H), 1.94-1.67 (m, 2H), 0.89(t,  $J = 7.6$  Hz, 3H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$  144.58, 128.34, 127.42, 125.94, 75.95, 31.82, 10.07; Anal.Calcd for  $\text{C}_9\text{H}_{12}\text{O}$ ; C, 79.37; H, 8.88; Found: C, 79.22; H, 8.90.

**3-phenylpropanoic acid (14b)<sup>13</sup>:**



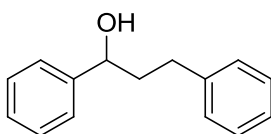
White solid; yield - 97%; mp:44-46 °C ; **IR** (Neat,  $\text{cm}^{-1}$ ): 3030, 2980, 2938, 1704, 1599, 1498, 1412, 1377, 1286, 1183, 1066, 937, 859, 761;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400MHz):  $\delta$  9.23(br, 1H), 7.31-7.20(m, 5H), 2.95(t, 2H,  $J = 7.6\text{Hz}$ ), 2.68(t, 2H,  $J = 8\text{Hz}$ );  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$  178.91, 140.15, 128.52, 128.23, 126.32, 35.61, 35.6; Anal.Calcd for  $\text{C}_9\text{H}_{10}\text{O}_2$ ; C, 71.98; H, 6.71; Found: C, 71.85; H, 6.75.

**Ethyl 3-phenylpropanoate (15b)**<sup>14</sup>:



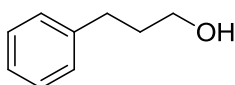
Colorless liquid; Yield – 55%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3086, 2975, 2938, 1739, 1496, 1452, 1373, 1296, 1165, 1041, 748;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.30-7.19 (m, 5H), 4.11 (q,  $J = 7\text{ Hz}$ , 2H), 2.94 (t,  $J = 7.7\text{Hz}$ , 2H), 2.61(t,  $J = 8\text{ Hz}$ , 2H), 1.22 (t,  $J = 7.1\text{ Hz}$ , 3H);  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  172.9, 140.5, 128.5, 128.3, 126.2, 60.4, 35.9, 30.9, 14.2; **MS** (EI)  $m/z$  178 ( $\text{M}^+$ ); Anal.Calcd for  $\text{C}_9\text{H}_{12}\text{O}$ ; C, 74.13; H, 7.92; Found: C, 73.74; H, 7.74.

**1,3-diphenylpropan-1-ol (16b)**<sup>15</sup>:



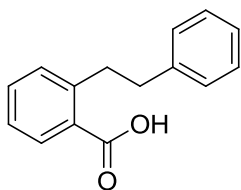
**Colorless liquid; Yield- 90%; IR** (Neat,  $\text{cm}^{-1}$ ): 3367, 3028, 1495, 1455, 1057, 698;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400 MHz ):  $\delta$  7.35-7.18(m, 10H), 4.69-4.66 (m, 1H) 2.77-2.62 (m, 2H), 2.15-1.99 (m, 3H) ,1.87(br, 1H);  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  144.5, 141.7, 128.5, 128.4, 128.3, 127.6, 125.9, 125.8, 73.8, 40.4, 32.0; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{15}\text{H}_{16}\text{O}$  ( $\text{M} + \text{Na}$ ): 235.1099 found ( $\text{M} + \text{Na}$ ): 235.1096.

**3-Phenyl-1-propanol (17b)**<sup>16</sup>:



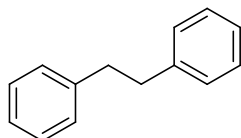
Colorless liquid; Yield - 90%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3338, 2939,1058, 1031;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.29-7.16 (m, 5H), 3.65 (t,  $J = 6.4\text{ Hz}$ , 2H), 2.69 (t,  $J = 7.6\text{ Hz}$ , 2H), 1.91-1.84 (m, 2H), 1.78 (s, 1H);  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  141.8, 128.4, 128.3, 125.8, 62.2, 34.1, 32.0; Anal.Calcd for  $\text{C}_9\text{H}_{12}\text{O}$ ; C, 79.37; H, 8.88; Found: C, 79.47; H, 8.59.

**2-phenethylbenzoic acid (18b)**<sup>17</sup>:



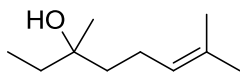
Colorless solid, Yield-95%; mp:130-132 °C ; **IR** (Neat,  $\text{cm}^{-1}$ ): 2926, 2863, 1690, 1475, 1273, 758, 699;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400 MHz) :  $\delta$  8.10 (d,  $J = 7.8$  Hz, 1H), 7.47 (t,  $J = 7.4$  Hz, 1H), 7.33-7.17 (m, 7H), 3.36-3.32 (m, 2H), 2.97-2.93 (m, 2H);  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  172.9, 144.8, 141.9, 133.0, 131.8, 131.5, 128.5, 128.3, 128.0, 126.2, 125.9; Anal.Calcd for  $\text{C}_{15}\text{H}_{14}\text{O}_2$ ; C, 79.62; H, 6.24; Found: C, 79.59; H, 6.35.

**1,2-diphenylethane (19b)<sup>15</sup>:**



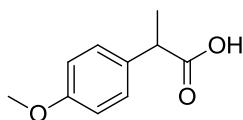
White solid; yield- 98%; mp: 48-51 °C; **IR** (KBr,  $\text{cm}^{-1}$ ): 3082, 3026, 2943, 2918, 1599, 1490, 1450;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.29-7.16 (m, 10H), 2.91 (s, 4H);  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$  141.8, 128.4, 128.3, 125.9, 37.9; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{14}\text{H}_{14}$  (M + K): 221.0733, found (M + K): 221.080.

**3,7-dimethyloct-6-en-4-ol (20b)<sup>18</sup>:**



**Colorless liquid; Yield- 90%; IR** (Neat,  $\text{cm}^{-1}$ ): 3754, 3401, 2971, 2928, 1638, 1378, 1116, 836;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400MHz) :  $\delta$  5.15-5.11(m,1H), 2.06-2.00(m, 2H), 1.68(s, 3H), 1.62(s, 3H), 1.52-1.45(m, 4H), 1.15(s,3H),0.90(t, 3H, 8Hz).  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$  131.49, 124.49, 72.83, 41.00, 34.21, 26.15,25.61, 22.55, 17.53,8.15; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{10}\text{H}_{20}\text{O}$  (M + K): 195.1151, found (M + K): 195.1300.

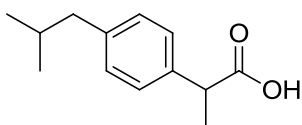
**2-(4-methoxyphenyl)propanoic acid (23b)<sup>19</sup>:**



White solid, Yield- 98%; mp:56-58 °C; **IR** (KBr,  $\text{cm}^{-1}$ ): 1710, 1605, 1515, 1465, 1305, 1185, 950, 795;  **$^1\text{H}$  NMR** ( $\text{CDCl}_3$ , 400MHz):  $\delta$  8.7(br, 1H), 7.23(d, 2H,  $J = 8$ Hz), 6.85(d, 2H,  $J = 8$ Hz), 3.77(s, 3H), 3.67(q,  $J = 7$ Hz,1H), 1.47(d, 3H,  $J = 8$ Hz)  **$^{13}\text{C}$  NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$ 180.29, 158.74,132.11, 128.57, 113.98, 55.21, 44.52, 18.19; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{14}\text{H}_{14}$  (M + Na): 203.0684, found (M + Na): 203.0695.

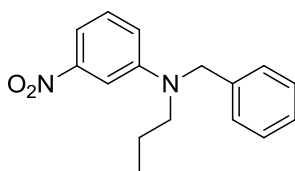


**2-(4-isobutylphenyl)propanoic acid (24b)<sup>20</sup>:**



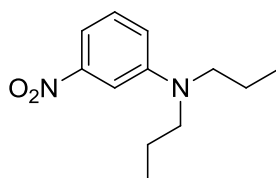
**White solid;** Yield 80%; mp:74-76 °C ; **IR** (Neat,  $\text{cm}^{-1}$ ): 3461, 2956, 1716, 1722, 1231,936; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.21(d,  $J = 8\text{Hz}$ , 2H), 7.09 (d,  $J = 8\text{Hz}$ , 2H), 3.69 (q,  $J = 7.1\text{Hz}$ , 1H), 2.43 (d,  $J = 7.1\text{ Hz}$ , 2H), 1.87-1.80 (m, 1H), 1.48 (d,  $J = 7.1\text{Hz}$ , 3H), 0.89 (d,  $J = 6.6\text{Hz}$ , 6H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  181.1, 140.8, 136.9, 129.4, 127.3, 45.0, 45.0, 30.1, 22.4, 18.0; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{13}\text{H}_{18}\text{O}_2$  (M + Na): 229.1204, found (M + Na): 229.1209.

**N-benzyl-3-nitro-N-propylaniline (25b):**



Brown liquid; yield-94%; **IR** (Neat,  $\text{cm}^{-1}$ ): 2962, 2874, 1621, 1525, 1347, 1239, 733; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.49-7.42 (m, 2H), 7.32-7.28 (m, 2H), 7.21-7.17 (m, 4H), 6.87 (dd,  $J_1 = 2.24$ ,  $J_2 = 8.36$ ), 4.58 (s, 2H), 3.43 (t,  $J = 7.7\text{ Hz}$ , 2H), 1.76-1.66 (m, 2H), 0.97 (t,  $J = 7.4\text{ Hz}$ , 3H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  149.4, 149.0, 137.5, 129.6, 128.7, 127.0, 126.2, 117.5, 110.3, 105.9, 54.3, 53.2, 20.1, 11.3; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2$  (M + Na): 293.1266, found (M + Na): 293.1265.

**3-nitro-N-dipropylaniline (26b)<sup>21</sup>:**



Pale yellow liquid; yield - 98%; **IR** (Neat,  $\text{cm}^{-1}$ ): 3834, 3732, 2918, 1697, 1521, 734; **<sup>1</sup>H NMR** ( $\text{CDCl}_3$ , 400MHz):  $\delta$  7.42-7.41 (m, 2H), 7.29-7.25 (m, 1H), 6.88-6.85(m, 1H), 3.29 (t,  $J = 7.5\text{ Hz}$ , 4H), 1.67-1.58 (m, 4H), 0.95 (t,  $J = 7.4\text{ Hz}$ , 6H); **<sup>13</sup>C NMR** ( $\text{CDCl}_3$ , 100MHz):  $\delta$  149.60, 148.72, 129.57, 117.04, 109.58, 105.60, 52.84, 20.16, 11.34; **HRESI-MS** ( $m/z$ ): Calculated for  $\text{C}_{12}\text{H}_{18}\text{N}_2\text{O}_2$  (M + Na): 245.1266, found (M + Na): 245.1261.

References:

- 1) M. Ohkubo, S. Mochizuki, T. Sano, Y. Kawaguchi and O. Sentaro, *org. Lett.*, 2007, **9**, 773.
- 2) M. Maddani and K. R. Prabhu, *Tetrahedron Lett.*, 2008, **49**, 4526.
- 3) L. Adak, K. Chattopadhyay and B. C. Ranu, *J. Org. Chem.*, 2009, **74**, 3982.
- 4) A. Mori, Y. Miyakawa, E. Ohashi, T. Haga, T. Maegawa and H. Sajiki, *Org. Lett.*, 2006, **8**, 3279.
- 5) M. Lee, M. S. Tremblay, S. Jockusch, N. J. Turro and D. Sames, *Org. Lett.*, 2011, **13**, 2802.
- 6) Y. Imada, T. Kitagawa, T. Ohno, H. Iida and T. Naota, *Org. Lett.*, 2010, **12**, 32.
- 7) M. Belloni, M. Manickam, P. Ashton, B. M. Kariuki, J. A. Preece, N. Spencer and Wilkie, *J. Mol. Cryst. Liq. Cryst.*, 2001, **369**, 17.
- 8) I-T. Ho, J-H. Chu and W-S. Chung, *Eur. J. Org. Chem.*, 2011, 1472.
- 9) K. R. Buszek and N. Brown, *J. Org. Chem.*, 2007, **72**, 3125.
- 10) A. Mori, Y. Miyakawa, E. Ohashi, T. Haga, T. Maegawa and H. Sajiki, *Org. Lett.*, 2006, **8**, 3279.
- 11) Y. Imada, T. Kitagawa, T. Ohno, H. Iida and T. Naota, *Org. Lett.*, 2010, **12**, 32.
- 12) H. Miyabe, A. Matsumura, K. Moriyama and Y. Takemoto, *Org. Lett.*, 2004, **6**, 4631.
- 13) H. Ohmiya, M. Tanabe and M. Sawamura, *Org. Lett.*, 2011, **13**, 1086.
- 14) Y. Jang, S. Kim, S. W. Jun, B. H. Kim, S. Hwang, I. K. Song, B. M. Kim and T. Hyeon, *Chem. Commun.*, 2011, **47**, 3601.
- 15) W. M. Czaplik, J-M. Neudorfl and A. J. V. Wangelin, *Green Chem.*, 2007, **9**, 1163.
- 16) K. R. Buszek and N. Brown, *J. Org. Chem.*, 2007, **72**, 3125.
- 17) A.G. Schultz and N. J. Green, *J. Am. Chem. Soc.*, 1991, **113**, 13.
- 18) C. Smit, M. W. Fraaije and A. J. Minnaard, *J. Org. Chem.*, 2008, **73**, 9482.
- 19) M. Allegretti, R. Bertini, M. C. Cesta, Bizzarri. C, R. Di. Bitondo, V. Di. Cioccio, E. Galliera, V. Berdini, A. Topai, G. Zampella, V. Russo, N. Di. Bello, G. Nano, L. Nicolini, M. Locati, P. Fantucci, S. Florio and F. Colotta, *J. Med. Chem.*, 2005, **48**, 4312.
- 20) I. Shiina, K. Nakata, K. Ono, Y-S. Onda and M. Itagaki, *J. Am. Chem. Soc.*, 2010, **132**, 11629.
- 21) G. Verardo, A. G. Giumanini and P. Strazzolini, *Synthetic Communications*, 1994, **24**, 609.

