Supplementary Material (ESI) for Green Chemistry

Green alcohol couplings without transition metal catalysts: base-mediated β-alkylation of alcohols in aerobic conditions

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Supplementary Data

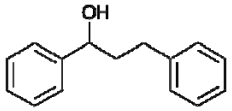
Experimental: All reagents were received from commercial sources and used without further purification. Toluene and dichloromethane were dried with a solvent purification system using 1 m column containing activated alumina. Proton NMR spectra were obtained at room temperature using $CDCl_3$ as solvent on 400 and 500 MHz Bruker spectrometers.

General Procedure: Secondary alcohol (2.0 mmol), primary alcohol (2.0 mmol), and base (2.0 mmol) were combined in a 5-mL roundbottom flask equipped with a micro stir bar, toluene (0.75 mL) and a reflux condenser. The reaction was refluxed for the appropriate amount of time and then cooled to room temperature. The reaction mixture was then quenched with 0.5 M citric acid, diluted with ethyl acetate and washed with distilled water and brine, dried over Na_2SO_4, and concentrated *in vacuo* to give the crude product, which was further purified using flash column chromatography.

^{1}H NMR and references:

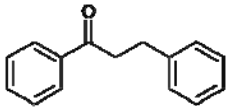
All products are known and their identity was established by comparison of their ^{1}H NMR spectral data with the data given in the references cited.

1,3-diphenylpropan-1-ol[1]



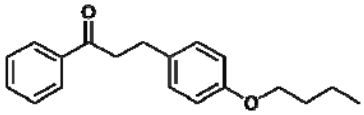
^{1}H NMR: (400 MHz, $CDCl_3$) 7.37 – 7.21 (10 H, m), 4.70 (1 H, m), 2.83 – 2.61 (2 H, m), 2.22 – 1.97 (2 H, m).

1,3-diphenylpropan-1-one[2]



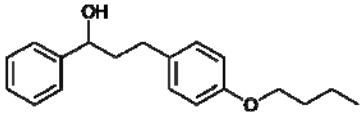
^{1}H NMR: (400 MHz, $CDCl_3$) 7.99 – 7.92 (2 H, m), 7.58 – 7.52 (1 H, m), 7.48 – 7.41 (2 H, m), 7.34 – 7.23 (4 H, m), 7.23 – 7.17 (1 H, m), 3.31 (2 H, m), 3.07 (2 H, t *J* = 7.7 Hz). mp 69-70 ^{o}C (from EtOAc) [lit.70-72 ^{o}C (ethanol)]

3-(4-butoxyphenyl)-1-phenylpropan-1-one[3]



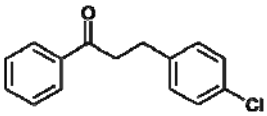
^{1}H NMR: (400 MHz, $CDCl_3$) 7.99 – 7.94 (2 H, d, *J* 8.0), 7.56 (1 H, t, *J* 7.4), 7.46 (2 H, t, *J* 7.6), 7.16 (2 H, d, *J* 8.4), 6.84 (2 H, d, *J* 8.4), 3.94 (2 H, td, *J* 3.0, 6.5), 3.27 (2 H, t, *J* 7.7) 3.02 (2 H, t, *J* 7.6), 1.80 – 1.72 (2 H, m), 1.49 (2 H, m), 0.98 (3 H, t, *J* 7.4).

3-(4-butoxyphenyl)-1-phenylpropan-1-ol[3]



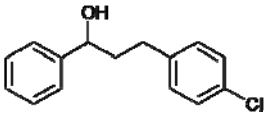
^{1}H NMR: (400 MHz, $CDCl_3$) 7.38 – 7.34 (4 H, m), 7.30 (1 H, d, *J* 2.1), 7.10 (2 H, d, *J* 8.6), 6.85 – 6.80 (2 H, m), 4.67 (1 H, dd, *J* 5.4, 7.8), 3.94 (2 H, t, *J* 6.5), 2.74 – 2.56 (2 H, m), 2.16 – 1.94 (2 H, m), 1.80 – 1.71 (2 H, m), 1.55 – 1.44 (2 H, m), 0.98 (3 H, m).

3-(4-chlorophenyl)-1-phenylpropan-1-one[4]



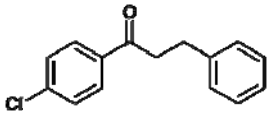
^{1}H NMR: (400 MHz, $CDCl_3$) 7.96 – 7.90 (2 H, m), 7.55 (1 H, m), 7.44 (2 H, m), 7.27 – 7.21 (2 H, m), 7.17 (2 H, m), 3.27 (2 H, t, *J* 7.5), 3.03 (2 H, t, *J* 7.5).

3-(4-chlorophenyl)-1-phenylpropan-1-ol[5]



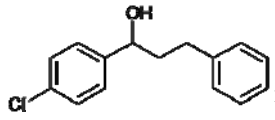
^{1}H NMR: (400 MHz, $CDCl_3$) 7.31 – 7.18 (5 H, m), 7.18 – 7.13 (2 H, m), 7.03 (2 H, d, *J* 8.3), 4.57 (1 H, m), 2.69 – 2.48 (2 H, m), 2.09 – 1.84 (3 H, m). mp 70-71 ^{o}C (from EtOAc) [lit.71-72 ^{o}C]

1-(4-chlorophenyl)-3-phenylpropan-1-one[6]



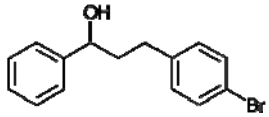
^{1}H NMR: (400 MHz, $CDCl_3$) 7.89 (2 H, d, *J* 8.8), 7.43 (2 H, d, *J* 8.8), 7.36 – 7.18 (5 H, m), 3.27 (2 H, t, *J* 7.6), 3.06 (2 H, t, *J* 7.6). mp 74-75 ^{o}C (from EtOAc) [lit.78-80 ^{o}C]

1-(4-chlorophenyl)-3-phenylpropan-1-ol[7]



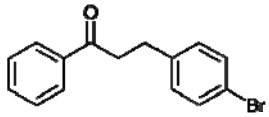
^{1}H NMR (400 MHz, $CDCl_3$) 7.26 – 7.15 (6 H, m), 7.15 – 7.07 (3 H, m), 4.61 – 4.53 (1 H, m), 2.72 – 2.50 (2 H, m), 2.08 – 1.87 (2 H, m).

3-(4-bromophenyl)-1-phenylpropan-1-ol[7]



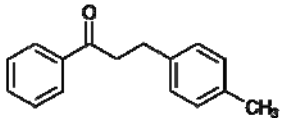
^{1}H NMR: (400 MHz, $CDCl_3$) 7.43 – 7.27 (7 H, m), 7.07 (2 H, d, *J* 8.3), 4.69 – 4.63 (1 H, m), 2.76 – 2.57 (2 H, m), 2.17 – 1.93 (3 H, m). mp 69-71 ^{o}C

3-(4-bromophenyl)-1-phenylpropan-1-one[4]



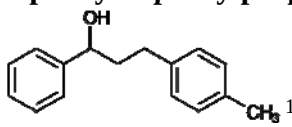
^{1}H NMR: (500 MHz, $CDCl_3$) 7.95 (2 H, d, *J* 7.9), 7.59 – 7.54 (1 H, m), 7.46 (2 H, t, *J* 6.8), 7.41 (2 H, d, *J* 7.3), 7.13 (2 H, d, *J* 8.2), 3.28 (2 H, t, *J* 7.5), 3.03 (2 H, t, *J* 7.5).

1-phenyl-3-*p*-tolylpropan-1-one[8]



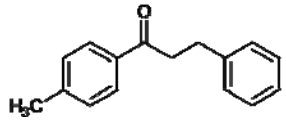
^{1}H NMR: (400 MHz, $CDCl_3$) 7.99 – 7.92 (2 H, m), 7.57 – 7.51 (1 H, m), 7.48 – 7.40 (2 H, m), 7.18 – 7.08 (4 H, m), 3.31 – 3.23 (2 H, m), 3.03 (2 H, t *J* 7.7), 2.32 (3 H, s).

1-phenyl-3-*p*-tolylpropan-1-ol[7]



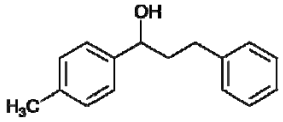
^{1}H NMR: (400 MHz, $CDCl_3$) 7.36 (4 H, d, *J* 4.3), 7.30 (1 H, dt, *J* 4.4, 9.0), 7.10 (4 H, s), 4.69 (1 H, m), 2.77 – 2.59 (2 H, m), 2.33 (3 H, s), 2.19 – 1.96 (2 H, m). mp 56-58 ^{o}C (from EtOAc) [lit.54-56 ^{o}C]

3-phenyl-1-*p*-tolylpropan-1-one[8]



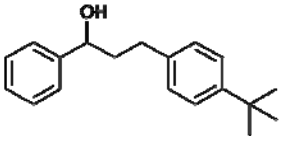
^{1}H NMR: (500 MHz, $CDCl_3$) 7.86 (2 H, m), 7.31 – 7.27 (2 H, m), 7.24 (4 H, dd, *J* 4.7, 11.1), 7.22 – 7.17 (1 H, m), 3.28 – 3.23 (2 H, m), 3.06 (2 H, m), 2.39 (3 H, s).

3-phenyl-1-*p*-tolylpropan-1-ol[5]



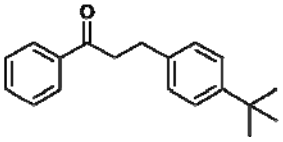
^{1}H NMR: (500 MHz, $CDCl_3$) 7.29 – 7.24 (2 H, m), 7.24 – 7.20 (2 H, m), 7.17 (5 H, dd, *J* 7.5, 15.2), 4.65 – 4.60 (1 H, m), 2.76 – 2.68 (1 H, m), 2.68 – 2.60 (1 H, m), 2.34 (3 H, s), 2.11 – 1.93 (2 H, m).

3-(4-*tert*-butylphenyl)-1-phenylpropan-1-ol[3]



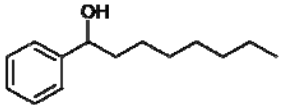
^{1}H NMR: (400 MHz, $CDCl_3$) 7.37 (4 H, d, *J* 4.2), 7.30 (3 H, m), 7.15 (2 H, d, *J* 8.1), 4.71 (1 H, m), 2.83 – 2.57 (2 H, m), 2.20 – 1.98 (2 H, m), 1.32 (9 H, s).

3-(4-*tert*-butylphenyl)-1-phenylpropan-1-one[3]



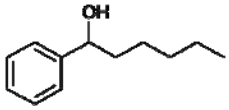
^{1}H NMR: (400 MHz, $CDCl_3$) 7.95 (2 H, dd, *J* 1.2, 8.4), 7.57 (1 H, dd, *J* 4.2 10.5), 7.47 (2 H, t, *J* 7.6), 7.36 (2 H, d, *J* 8.3), 7.22 (2 H, d, *J* 8.1), 3.38 – 3.26 (2 H, m), 3.13 – 2.99 (2 H, m), 1.33 (9 H, s).

1-phenyloctan-1-ol[9]



^{1}H NMR: (400 MHz, $CDCl_3$) 7.34 (4 H, m), 7.27 (1 H, m), 4.65 (1 H, t, *J* 6.5), 1.76 (1 H, m), 1.74 (1 H, s), 1.26 (10 H, d, J 4.2), 0.87 (3 H, m).

1-phenylhexan-1-ol[10]



^{1}H NMR: (400 MHz, $CDCl_3$) 7.35 (3 H, m), 7.28 (2 H, m), 4.66 (1 H, m), 1.84 (1 H, m), 1.71 (1 H, m), 1.40 (1 H, s), 1.27 (5 H, m), 0.88 (3 H, s).

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