Hydrogenation of aldehydes and ketones to corresponding alcohols with methylamine borane in neat water

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¹H spectra of MeAB

1H NMR spectrum of 1,2,3,4-tetrahydro-1,4-benzodioxine. The spectrum shows a complex multiplet between 4.5 and 5.5 ppm, with peaks labeled 'a' through 'f'. The x-axis is labeled 'PPM' and ranges from 0 to 10.

S2. XRD pattern of MeAB

S3. Infrared spectra of MeAB

S4. Scheme 1

S5. ^1H and ^{13}C NMR characterization of products:

Phenylmethanol (entry 1, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 4.61 (s, 2H; CH_2), 7.26-7.35 ppm (m, 5H; ArH); ^{13}C NMR (100 MHz, CD_3OD , 25°C ; CD_3OD): δ = 63.89, 126.67, 126.95, 128.02, 141.24 ppm.

Cinnamyl alcohol (entry 2, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): δ = 4.24 (dd, J_{HH} = 6.0, 1.8 Hz, 2H; CH_2), 6.38 (dt, J_{HH} = 16.2, 6.0 Hz, 1H; $\text{CH}=\text{CH}$), 6.62 (d, J_{HH} = 15.6 Hz, 1H; $\text{CH}=\text{CH}$), 7.21–7.23 (t, J_{HH} = 7.8 Hz, 1H; ArH), 7.30 – 7.32 (t, J_{HH} = 7.8 Hz, 2H; ArH), 7.40 – 7.42 ppm (d, 7.8 Hz, 2H; ArH); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): δ = 63.72, 127.45, 128.53, 129.61, 130.02, 131.64, 138.48 ppm.

Furan-2-methanol (entry 3, Table 1). ^1H NMR (600 MHz, CDCl_3 , 25°C ; TMS): δ = 2.47 (br, s, 1H, OH), 4.57 (s, 2H, CH_2), 6.27 (d, J_{HH} = 3.6 Hz, 1H; $\text{CH}=\text{CH}$), 6.33 (dd, J_{HH} = 3.0, 1.2 Hz, 1H; $\text{CH}=\text{CH}$), 7.39 (d, J_{HH} = 1.8 Hz, 1H; $\text{CH}=\text{CH}$); ^{13}C NMR (150 MHz, CDCl_3 , 25°C ; CDCl_3): δ = 57.32, 107.72, 110.35, 142.53, 154.07 ppm.

Citronellol (entry 4, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): δ = 0.93 (d, J_{HH} = 6.6 Hz, 3H; CH_3), 1.15-1.22, 1.29-1.32 (m, 2H; CH_2), 1.35-1.40 (m, 2H; CH_2), 1.58-1.61 (m, 1H; CH), 1.63 (s, 3H; CH_3), 1.70 (s, 3H; CH_3), 1.98-2.07 (m, 2H; CH_2), 3.56-3.64 (m, 2H; CH_2), 5.13 ppm (t, J_{HH} = 7.2 Hz, 1H; CH); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): δ = 17.84, 20.05, 26.02, 26.55, 30.41, 38.51, 40.85, 61.16, 125.94, 131.84 ppm.

Nerol (entry 5, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): δ = 1.63 (s, 3H; CH_3), 1.70 (s, 3H; CH_3), 1.76 (s, 3H; CH_3), 2.11 (s, 4H; CH_2CH_2), 4.07 (d, J_{HH} = 7.2 Hz, 2H; CH_2), 5.14 (s, 1H; CH), 5.38 ppm (t, J_{HH} = 6.0 Hz, 1H; CH); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): δ = 17.77, 23.76, 25.84, 27.82, 33.01, 59.27,

125.08, 125.81, 132.77, 138.58 ppm.

p-tolylmethanol (entry 6, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): δ = 2.33 (s, 3H; CH_3), 4.56 (s, 2H; CH_2), 7.15 (d, $J_{\text{HH}} = 7.8\text{Hz}$, 2H; ArH), 7.24 ppm (d, $J_{\text{HH}} = 8.4\text{Hz}$, 2H; ArH); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): δ = 21.23, 65.17, 128.18, 130.00, 138.03, 139.65 ppm.

4-Methoxyphenylmethanol (entry 7, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 3.77 (s, 3H; CH_3), 4.54 (s, 2H; CH_2), 6.89 (d, $J_{\text{HH}} = 8.4\text{Hz}$, 2H; ArH), 7.27 ppm (d, $J_{\text{HH}} = 8.4\text{Hz}$, 2H; ArH); ^{13}C NMR (100 MHz, CD_3OD , 25°C ; CD_3OD): δ = 54.40, 63.59, 113.42, 128.30, 133.32, 159.10 ppm.

4-Chlorophenylmethanol (entry 8, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 4.59 (s, 2H; CH_2), 7.33-7.39 ppm (m, 4H; ArH); ^{13}C NMR (100MHz, CD_3OD , 25°C ; CD_3OD): δ = 63.02, 128.03, 129.87, 132.52, 140.16 ppm.

4-Nitrophenylmethanol (entry 9, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 4.74 (s, 2H; CH_2), 7.58 (d, $J_{\text{HH}} = 8.8\text{ Hz}$, 2H; ArH), 8.20 ppm (d, $J_{\text{HH}} = 8.4\text{ Hz}$, 2H; ArH); ^{13}C NMR (100MHz, CD_3OD , 25°C ; CD_3OD): δ = 62.65, 123.09, 126.86, 147.01, 149.45 ppm.

Methyl 4-(hydroxymethyl)benzoate (entry 10, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 3.90 (s, 3H; CH_3), 4.68 (s, 2H; CH_2), 7.46 (d, $J_{\text{HH}} = 8.0\text{ Hz}$, 2H; ArH); 7.98 ppm (d, $J_{\text{HH}} = 8.0\text{ Hz}$, 2H; ArH); ^{13}C NMR (100 MHz, CD_3OD , 25°C ; CD_3OD): δ = 51.31, 63.19, 126.26, 129.24, 147.14, 167.20 ppm.

2-Chlorophenylmethanol (entry 11, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 4.71 (s, 2H; CH_2), 7.24–7.36 (m, 3H; ArH), 7.54 ppm (d, $J_{\text{HH}} = 7.6\text{ Hz}$, 1H; ArH); ^{13}C NMR (100MHz, CD_3OD , 25°C ; CD_3OD): δ = 61.03, 126.69, 128.07, 128.22, 128.76, 132.02, 138.61 ppm.

Vanillyl alcohol (entry 12, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 3.85 (s, 3H; CH_3); 4.52 (s, 2H; CH_2), 6.79 (s, 2H; ArH), 6.95 ppm (s, 1H; ArH); ^{13}C NMR (100 MHz, CD_3OD , 25°C ; CD_3OD): δ = 55.04, 63.98, 110.79, 114.66, 119.75, 132.86, 145.47, 147.54 ppm.

3,4,5-Trimethoxyphenylmethanol (entry 13, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 3.75 (s, 3H; CH_3), 3.84 (s, 6H; CH_3), 4.56 (s, 2H; CH_2), 6.67 ppm (s, 2H; ArH); ^{13}C NMR (100 MHz, CD_3OD , 25°C ; CD_3OD): δ = 55.19, 59.79, 63.88, 103.65, 136.64, 137.59, 153.06 ppm.

2,6-Di-tert-butyl-4-(hydroxymethyl)phenol (entry 14, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): δ = 1.87 (s, 1H; O-H), 1.44 (s, 18H; CH_3), 3.73 (s, 1H; O-H), 4.50 (s, 2H; CH_2), 7.17 ppm (s, 2H; ArH); ^{13}C NMR (100 MHz, CD_3OD , 25°C ; CD_3OD): δ = 29.46, 34.13, 64.62, 123.82, 131.83, 137.66, 153.29 ppm.

Cyclohexanol (entry 15, Table 1). ^1H NMR (600 MHz, CDCl_3 , 25°C ; TMS): δ = 1.19-1.30, 1.53-1.56, 1.73-1.90 (m, 10H; CH_2), 3.60-3.62 ppm (m, 1H; CH); ^{13}C NMR (150 MHz, CDCl_3 , 25°C ; CD_3Cl_3): δ = 24.14, 25.46, 35.52, 70.27 ppm.

Cyclopentanol (entry 16, Table 1). ^1H NMR (600 MHz, CDCl_3 , 25°C ; TMS): δ = 1.58 (s, 4H; CH_2CH_2), 1.77 ppm (s, 4H; CH_2CH_2); ^{13}C NMR (150 MHz, CDCl_3 , 25°C ; CD_3Cl_3): δ = 24.22, 36.00, 74.47 ppm.

3,5,5-Trimethyl-2-cyclohexen-1-ol (entry 17, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): δ = 0.79 (s, 3H; CH_3), 0.89 (s, 3H; CH_3), 1.12 (dd, J_{HH} = 9.6, 12 Hz, 1H; $\text{CH}=\text{CH}$), 1.57 (s, 3H; CH_3), 1.45-1.52, 1.74-1.90 (m, 4H; CH_2), 3.96-4.07 (m, 1H; CH_3), 5.29 ppm (s, 1H; $\text{CH}=\text{CH}$); ^{13}C NMR (150 MHz, CDCl_3 , 25°C ; CD_3Cl_3): δ = 23.53, 26.24, 31.21, 44.14, 45.29, 66.87, 123.65, 136.05 ppm.

1-Phenylethanol (entry 18, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): δ

= 1.45 (d, $J_{\text{HH}} = 6.6$ Hz, 3H; CH_3), 4.84 (q, $J_{\text{HH}} = 6.6$ Hz, 1H; CH), 7.23–7.39 (m, 5H; ArH); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): $\delta = 25.70, 70.92, 126.53, 128.14, 129.33, 147.63$ ppm.

1-(*p*-tolyl)ethanol (entry 19, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): $\delta = 1.45$ (d, $J_{\text{HH}} = 6.4$ Hz, 3H; CH_3), 2.31 (s, 3H; CH_3), 4.81 (q, $J_{\text{HH}} = 6.4$ Hz, 1H; CH), 7.13 (d, $J_{\text{HH}} = 8.0$ Hz, 2H; ArH), 7.27 ppm (d, $^3J_{\text{HH}} = 8.0$ Hz, 2H; ArH); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): $\delta = 20.07, 24.46, 69.37, 125.20, 128.62, 136.32, 143.18$ ppm.

1-(4-Methoxyphenyl)ethanol (entry 20, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): $\delta = 1.43$ (d, $J_{\text{HH}} = 7.8$ Hz, 3H; CH_3), 3.79 (s, 3H; CH_3), 4.79 (q, $J_{\text{HH}} = 6.6$ Hz, 1H; CH), 6.89 (d, $J_{\text{HH}} = 9.0$ Hz, 2H; ArH), 7.29 ppm (d, $J_{\text{HH}} = 8.4$ Hz, 2H; ArH); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): $\delta = 25.51, 55.74, 70.50, 114.69, 127.77, 139.59, 160.34$ ppm.

1-(4-Bromophenyl)ethanol (entry 21, Table 1). ^1H NMR (400 MHz, CD_3OD , 25°C ; TMS): $\delta = 1.42$ (d, $J_{\text{HH}} = 6.4$ Hz, 3H; CH_3), 4.81 (q, $J_{\text{HH}} = 6.4$ Hz, 1H; CH), 5.50 (s, 1H; OH), 7.30 (d, $J_{\text{HH}} = 8.4$ Hz, 2H; ArH), 7.47 ppm (d, $J_{\text{HH}} = 8.4$ Hz, 2H; ArH); ^{13}C NMR (100 MHz, CD_3OD , 25°C ; CD_3OD): $\delta = 24.18, 68.74, 120.22, 127.10, 130.96, 145.58$ ppm.

Diphenylmethanol (entry 22, Table 1). ^1H NMR (600 MHz, CD_3OD , 25°C ; TMS): $\delta = 5.79$ (s, 1H; H), 7.22–7.39 (m, 10H); ^{13}C NMR (150 MHz, CD_3OD , 25°C ; CD_3OD): $\delta = 77.01, 127.78, 128.25, 129.31, 145.98$ ppm.