

Supporting Information

In Situ Oxidation-Imine Formation-Reduction Routes from Alcohols to Amines

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General Procedure for Imine formation

Activated manganese dioxide (Aldrich, cat no. 21,746-6) (0.435 g, 5.0 mmol) was added to a stirred solution of the specified alcohol (1 mmol), specified amine (1.0 – 3.0 mmol)^a and molecular sieves^b (0.2 g) in DCM (25 mL) and the mixture heated to reflux. The remainder of the manganese dioxide (0.435 g, 5.0 mmol) was added to the reaction after one hour and then it was stirred at reflux overnight.^c The manganese dioxide was removed by filtration through Celite, the Celite washed well with DCM, and the combined organic portions were concentrated *in vacuo* to afford crude imine.^d

a) All amines were used in 1.5 mmol except, benzylamine and (R)-1-phenylethylamine (1.3 mmol), allyl amine (3 mmol) and cyclohexyl amine (1 mmol).

b) MgSO₄ was used as the drying agent when benzylamine was the reactant amine.

c) The reaction mixture was stirred at reflux for 48 hours when allyl amine was employed.

d) The crude imine was identified by ¹H NMR spectroscopy and was used directly for the reduction.

General Procedure for Reduction of Imines to Amines

The prepared imine (1 mmol) was dissolved in methanol (5 mL) and cooled to 0 °C under N₂ (g). Sodium borohydride (1.1 mmol) was added portionwise and the reaction allowed to stir for 20 mins. The solvent was evaporated and the residue taken up in diethyl ether (15 mL) and sat. NaHCO₃ (15 mL).^a The organic layer was separated and the aqueous layer extracted with diethyl ether (2 x 15 mL). The combined organic extracts were dried (MgSO₄), filtered and concentrated *in vacuo* to afford the product amine.^b

a) When using benzylamine and (R)-1-phenylethylamine the products were slightly contaminated with benzaldehyde and acetophenone and the work up procedure was modified. The solvent was evaporated and the residue taken up in diethyl ether (15 mL) and sat. NaHCO₃ (15 mL). The organic layer was separated and the aqueous

layer extracted with diethyl ether (2 x 15 mL). The combined organic washings were extracted with 2M aq. HCl (3 x 15 mL). The acidic extracts were combined and washed with EtOAc (20 mL), then basified with NaHCO₃ and extracted with diethyl ether (4 x 20 mL). The organic layer was dried (MgSO₄), filtered and concentrated *in vacuo* to afford the product amine.^b

b) Yields of the amine refer to unpurified product. Known compounds were confirmed by comparison of ¹H NMR data to those published and novel compounds were fully characterised.

General Procedure for One Pot Oxidation-Imine Formation-Reduction

Activated manganese dioxide (0.435 g, 5.0 mmol) was added to a stirred solution of specified alcohol (1 mmol), specified amine (2.0 – 4.0 mmol),^a molecular sieves (ca. 0.200 g) and polymer supported cyanoborohydride (5 mmol) in DCM (25 mL) and the mixture heated to reflux. The remainder of the manganese dioxide (0.435 g, 5.0 mmol) was added to the reaction after one hour. AcOH (0.11 mL, 2.0 mmol) was added to the reaction mixture when TLC showed that all the alcohol had essentially reacted (approx. 3-4 hours). The reaction mixture was allowed to stir at reflux until the aldehyde/imine had reacted as shown by TLC (24 – 64 hours). It was then allowed to cool and filtered through silica washing well with EtOAc.^b The solvent was evaporated and the resulting oil dissolved in diethyl ether (15 mL). The organic layer was extracted with 2M aq. HCl (3 x 15 mL). The acidic extracts were combined and washed with EtOAc (20 mL) then basified with sat. aq. NaHCO₃ and extracted with diethyl ether (4 x 20 mL). The combined organic layers were dried (MgSO₄), filtered and concentrated *in vacuo* to afford the product amines.

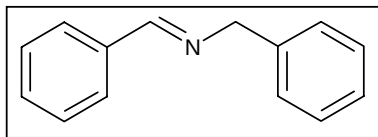
a) All amines were used in 4 mmol quantity except in the reaction of benzyl alcohol with isobutylamine when only 2 mmol was necessary.

b) The work up procedure was modified when using the following amines:

i) Di-isobutylamine – After filtration through silica, washing well with EtOAc, the solution was concentrated *in vacuo*. The crude product was then purified by column chromatography.

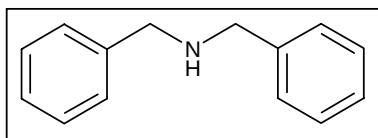
ii) Di-ethyl amine - After filtration through silica, washing well with EtOAc, the solution was concentrated *in vacuo*. The crude product then was purified by Kugerohl distillation.

iii) Pentyl- and butylamine - After filtration through silica, washing well with EtOAc, the solution was concentrated *in vacuo* and purified as the N-BOC derivative.¹



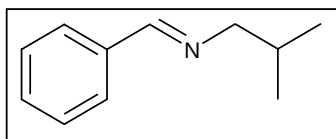
Phenyl-*N*-(phenylmethylidene)methanamine²

δ_{H} (270.01 MHz, CDCl_3) 4.84 (2 H, d, J 1.2 Hz), 7.35 - 7.81 (10 H, m), 8.37 (1 H, s).



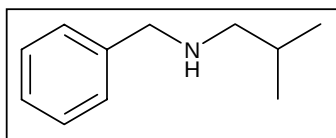
***N,N*-Dibenzylamine**³

δ_{H} (270.01 MHz, CDCl_3) 1.69 (1 H, br s), 3.83 (4 H, s), 7.25 - 7.34 (10 H, m); δ_{C} (67.9 MHz, CDCl_3) 53.2, 126.9, 128.1, 128.3, 140.2.



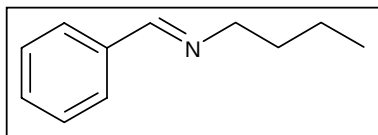
2-Methyl-*N*-(phenylmethylidene)-1-propanamine²

δ_{H} (270.01 MHz, CDCl_3) 0.97 (6 H, d, J 6.7 Hz), 2.03 (1 H, sept, J 6.7 Hz), 3.45 (2 H, dd, J 6.7, 1.2 Hz), 7.41 - 7.43 (3 H, m), 7.73 - 7.76 (2 H, m), 8.25 (1 H, t, J 1.2 Hz).



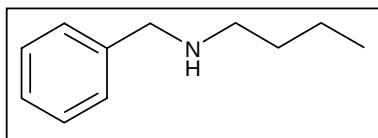
***N* - Benzyl-*N*-isobutylamine**⁴

δ_{H} (270.01 MHz, CDCl_3) 0.93 (6H, d, J 6.8 Hz), 1.43 (1H, br s), 1.79 (1H, sept, J 6.8 Hz), 2.45 (2H, d, J 6.8 Hz), 3.80 (2H, s), 7.22 - 7.35 (5H, m); δ_{C} (67.9 MHz, CDCl_3) 20.7, 28.5, 54.1, 57.5, 126.7, 127.9, 128.2, 140.7.



***N*-Phenylmethylidene-1-butanamine**⁵

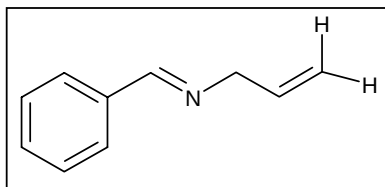
δ_{H} (270.01 MHz, CDCl_3) 0.92 (3 H, t, J 7.2 Hz), 1.36 (2 H, sex, J 7.2 Hz), 1.66 (2 H, quin, J 7.2 Hz), 3.58 (2 H, t, J 7.2 Hz), 7.36 - 7.38 (3 H, m), 7.68 - 7.70 (2 H, m), 8.24 (1 H, s).



***N* - Benzyl- *N* - butylamine**⁴

δ_{H} (270.01 MHz, CDCl_3) 0.91 (3 H, t, J 7.2 Hz), 1.31 (2 H, sex, J 7.2 Hz), 1.43 - 1.50 (2 H, m), 2.63

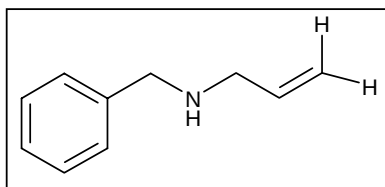
(2 H, t, J 7.2 Hz), 3.79 (2 H, s), 7.24 – 7.32 (5 H, m); δ_{C} (67.9 MHz, CDCl_3) 14.3, 20.5, 32.3, 49.2, 54.1, 126.7, 128.0, 128.2, 140.5.



***N*-(Phenylmethylidene)-2-propene-1amine⁶**

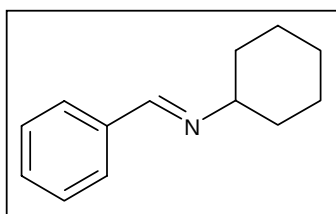
δ_{H} (270.01 MHz, CDCl_3) 4.23 (2 H, dd, J 5.6, 1.6 Hz), 5.12 (1 H, d, J 10.4 Hz), 5.20 (1 H, d, J 17.2 Hz), 6.00 – 6.07 (1 H, m), 7.37 – 7.39 (3 H, m),

7.71 – 7.73 (2 H, m), 8.27 (1 H, s).



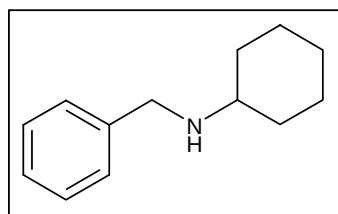
***N* – Allyl– *N* – benzylamine⁷**

δ_{H} (270.01 MHz, CDCl_3) 1.39 (1 H, br s), 3.25 (2 H, d, J 6.0 Hz), 3.76 (2 H, s), 5.08 (1 H, dd, J 10.0, 1.6 Hz), 5.17 (1 H, dd, J 17.2, 1.6 Hz), 5.91 (1 H, ddt, J 17.2, 10.0, 6.0 Hz) 7.19 – 7.34 (5 H, m); δ_{C} (67.9 MHz, CDCl_3) 51.7, 53.2, 115.8, 126.8, 128.0, 128.2, 136.6, 140.1.



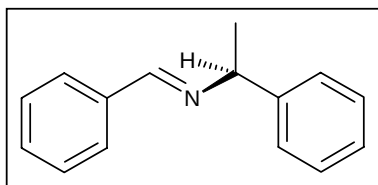
***N*-Phenyl(methylidene)cyclohexanamine⁸**

δ_{H} (270.01 MHz, CDCl_3) 1.18 – 1.82 (10 H, m), 3.12 – 3.20 (1 H, m), 7.38 – 7.41 (3 H, m), 7.72 – 7.73 (2 H, m), 8.32 (1 H, s).



***N* – Benzyl– *N* –cyclohexylamine⁹**

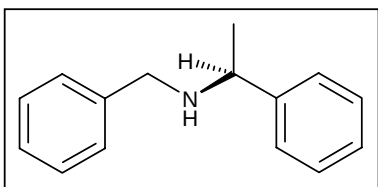
δ_{H} (270.01 MHz, CDCl_3) 1.15 – 1.28 (6 H, m), 1.56 – 1.59 (1 H, m), 1.68 – 1.72 (2 H, m), 1.86 – 1.90 (2 H, m), 2.42 – 2.48 (1 H, m), 3.78 (2 H, s), 7.19 – 7.29 (5 H, m); δ_{C} (67.9 MHz, CDCl_3) 25.0, 26.2, 33.5, 51.0, 56.2, 126.7, 128.0, 128.3, 141.0.



(1R)-1-Phenyl-*N*-(phenylmethylidene) - ethanamine

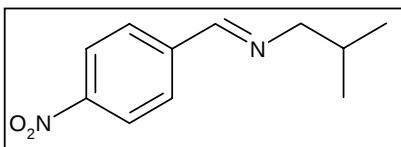
δ_{H} (270.01 MHz, CDCl_3) 1.57 (3 H, d, J 6.5 Hz), 4.52 (1 H, q, J 6.5 Hz), 7.19 – 7.95 (10 H, m), 8.35

(1H, s).



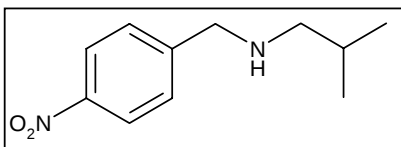
(1R)-N-Benzyl-1-phenylethanamine^{10,11}

$[\alpha]_D$ 60.1 (*c* 0.5, EtOH) lit.¹¹ 54.4 (*c* 0.5 EtOH); δ_H (270.01 MHz, CDCl₃) 1.34 (3 H, d, *J* 6.8 Hz), 1.68 (1 H, br s), 3.56 (1 H, d, *J* 13.0 Hz), 3.63 (1 H, d, *J* 13.0 Hz), 3.79 (1 H, q, *J* 6.8 Hz), 7.19 – 7.33 (10 H, m); δ_C (67.9 MHz, CDCl₃) 24.4, 51.6, 57.5, 126.7, 127.8, 126.9, 128.1, 128.3, 128.4, 145.5, 140.6.



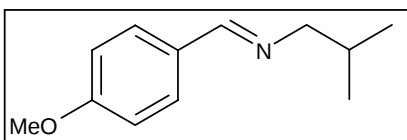
2-Methyl-N-(4-nitrophenyl)methylidene-1-propanamine

δ_H (270.01 MHz, CDCl₃) 0.94 (6 H, d, *J* 6.8 Hz), 2.00 (1 H, sept, *J* 6.8 Hz), 3.46 (2 H, d, *J* 6.8 Hz), 7.86 (2 H, d, *J* 8.8 Hz), 8.23 (2 H, d, *J* 8.8 Hz), 8.29 (1 H, s).



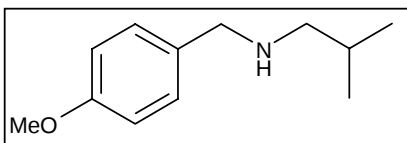
N-Isobutyl-N-(4-nitrobenzyl)amine

ν_{max} (neat)/cm⁻¹ 3329 (NH); δ_H (270.01 MHz, CDCl₃) 0.89 (6 H, d, *J* 6.8 Hz), 1.33 (1 H, br s), 1.72 (1 H, sept, *J* 6.8 Hz), 2.39 (2 H, d, *J* 6.8 Hz), 3.85 (2 H, s), 7.47 (2 H, d, *J* 8.8 Hz), 8.13 (2 H, d, *J* 8.8 Hz); δ_C (67.9 MHz, CDCl₃) 20.6, 28.5, 53.3, 57.5, 123.5, 128.4, 148.5; *m/z* (CI) 209 (M+H⁺, 100%); Found 209.1286, C₁₁H₁₇N₂O₂ requires 209.1285 (+ 0.6 ppm error).



2-Methyl-N-(4-methoxyphenyl)methylidene-1-propanamine

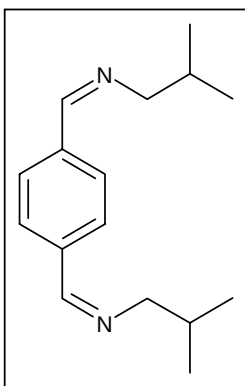
δ_H (270.01 MHz, CDCl₃) 0.92 (6 H, d, *J* 6.8 Hz), 1.96 (1 H, sept, *J* 6.8 Hz), 3.36 (2 H, d, *J* 6.8 Hz), 3.80 (3 H, s), 6.88 (2 H, d, *J* 8.8 Hz), 7.64 (2 H, d, *J* 8.8 Hz), 8.13 (1 H, s).



N-Isobutyl-N-(4-methoxybenzyl)amine

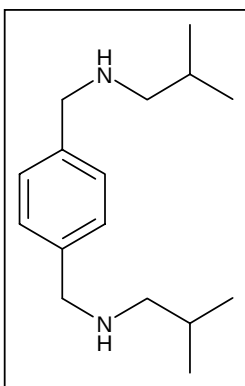
ν_{max} (neat)/cm⁻¹ 3328 (NH); δ_H (270.01 MHz, CDCl₃) 0.88 (6 H, d, *J* 6.8 Hz), 1.23 (1 H, br s), 1.73 (1 H, sept, *J* 6.8 Hz), 2.39 (2 H, d, *J* 6.8 Hz), 3.68 (2 H, s), 3.76 (3 H, s), 6.82 (2 H, d, *J* 8.8 Hz), 7.20 (2 H, d, *J* 8.8 Hz); δ_C (67.9 MHz, CDCl₃) 20.7, 28.3, 53.5, 55.2,

57.4, 113.7, 129.2, 132.9, 158.5; m/z (CI) 194 ($M+H^+$, 100 %); Found 194.1540, $C_{12}H_{20}NO$ requires 194.1540 (+ 0.12 ppm error).



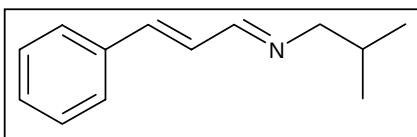
2-Methyl-N-(4-(((2-methylpropyl)imino)methyl)phenyl)methylidene-1-propanamine

δ_H (270.01 MHz, $CDCl_3$) 0.93 (12 H, d, J 6.8 Hz), 1.99 (2 H, sept, J 6.8 Hz), 3.41 (4 H, dd, J 6.8, 1.2 Hz), 7.74 (4 H, s), 8.22 (2 H, t, J 1.2 Hz).



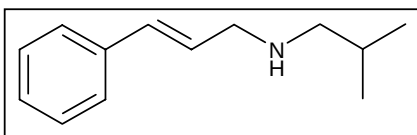
N-(4-((isobutylamine)methyl)benzyl)-2-methyl-1-propanamine

ν_{max} (neat)/ cm^{-1} 3311 (NH); δ_H (270.01 MHz, $CDCl_3$) 0.87 (12 H, d, J 6.8 Hz), 1.22 (2 H, br s), 1.73 (2 H, sept, J 6.8 Hz), 2.40 (4 H, d, J 6.8 Hz), 3.73 (4 H, s), 7.24 (4 H, s); δ_C (67.9 MHz, $CDCl_3$) 20.7, 28.4, 53.9, 57.4, 127.9, 139.2; m/z (CI) 249 ($M+H^+$, 100%); Found 249.2324, $C_{16}H_{28}N_2$ requires 249.2325 (- 0.30 ppm error).



2-Methyl-N-((2E)-3-phenyl-2-propenylidene)-1-propanamine

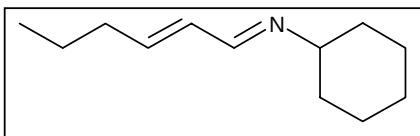
δ_H (270.01 MHz, $CDCl_3$) 0.91 (6 H, d, J 6.4 Hz), 1.94 (1 H, sept, J 6.4 Hz), 3.31 (2 H, d, J 6.4 Hz), 6.88 – 6.90 (2 H, m), 7.22 – 7.45 (5 H, m), 7.94 – 7.97 (1 H, m).



N-isobutyl-3-((2E)-3-phenyl-2-propen-1-amine)¹²

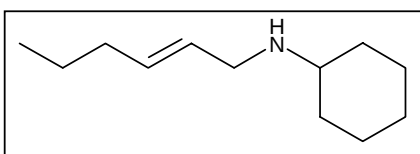
δ_H (270.01 MHz, $CDCl_3$) 0.90 (6 H, d, J 6.4 Hz), 1.22 (1 H, br s), 1.74 (1 H, sept, J 6.4 Hz), 2.44 (2 H, d, J 6.4 Hz), 3.37 (2 H, dd, J 6.4, 1.2 Hz), 6.28 (1 H, dt, J 16.0, 6.4 Hz), 6.49 (1 H, d, J 16.0 Hz), 7.16 – 7.35 (5 H, m).

m); δ_C (67.9 MHz, $CDCl_3$) 20.6, 28.3, 52.0, 57.5, 126.2, 127.2, 128.4, 128.7, 131.001, 137.2.



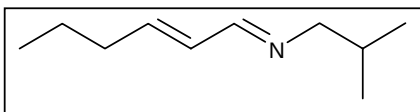
***N*-((2*E*)-2-Hexenylidene)cyclohexanamine**

δ_H (270.01 MHz, $CDCl_3$) 0.89 (3 H, t, *J* 7.5 Hz), 0.98 – 1.80 (12 H, m), 2.10 – 2.16 (2 H, m), 2.89 – 2.94 (1 H, m), 6.07 – 6.23 (2 H, m), 7.82 (1 H, d, *J* 8.0 Hz)..



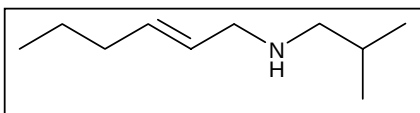
***N*-((2*E*)-2-Hexenyl)cyclohexylamine**

ν_{max} (neat)/ cm^{-1} 3222 (NH); δ_H (270.01 MHz, $CDCl_3$) 0.84 (3 H, t, *J* 7.5 Hz), 0.98 – 1.26 (6 H, m), 1.30 – 1.39 (2 H, m), 1.57 (1 H, d, *J* 12.4 Hz), 1.68 (2 H, d, *J* 13.0 Hz), 1.84 (2 H, d, *J* 13.0 Hz), 1.90 – 2.00 (2 H, m), 2.38 – 2.45 (1 H, m), 3.17 (2 H, d, *J* 5.2 Hz), 5.45 – 5.74 (2 H, m); δ_C (67.9 MHz, $CDCl_3$) 13.6, 22.0, 25.0, 26.1, 33.5, 34.4, 48.8, 56.1, 128.7, 132.2; *m/z* (ES) 182 (*M*+*H*⁺, 100%); Found 182.1905, $C_{12}H_{24}N$ requires 182.1903 (+1.23 ppm error).



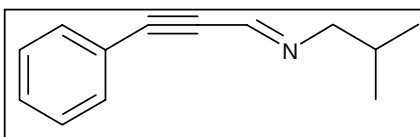
***N*-((2*E*)-2-Hexenylidene)-2-methyl-1-propanamine**

δ_H (270.01 MHz, $CDCl_3$) 0.80 - 0.90 (9 H, m), 1.44 (2 H, sex, *J* 7.5 Hz), 1.88 (1 H, sept, *J* 6.8 Hz), 2.12 – 2.17 (2 H, m), 3.20 (2 H, d, *J* 6.5 Hz), 6.10 – 6.22 (2 H, m), 7.75 (1 H, d, *J* 8.4 Hz).



***N*-((2*E*)-2-Hexenyl)isobutylamine¹²**

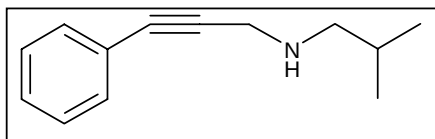
δ_H (270.01 MHz, $CDCl_3$) 0.85 – 0.90 (9 H, m), 1.37 (2 H, sex, *J* 7.5 Hz), 1.74 (1 H, sept, *J* 6.8 Hz), 1.94 – 2.02 (2 H, m), 2.39 (2 H, d, *J* 6.8 Hz), 3.15 (2 H, d, *J* 5.1 Hz), 5.45 – 5.60 (2 H, m); δ_C (67.9 MHz, $CDCl_3$) 13.6, 20.7, 22.4, 28.3, 34.4, 51.9, 57.4, 128.6, 132.3.



2-Methyl-*N*-(3-phenyl-2-propynylidene)-1-propanamine

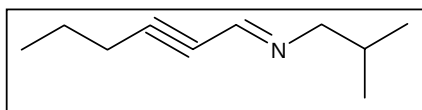
Isomer A δ_H (270.01 MHz, $CDCl_3$) 0.92 (6 H, d, *J* 6.5 Hz), 1.98 (1 H, sept, *J* 6.5 Hz), 3.36 (2 H, d, *J* 6.5 Hz), 7.28 – 7.52 (5 H, m),

7.66 (1 H, s). Isomer B 0.96 (6 H, d, J 6.8 Hz), 1.98 (1 H, sept, J 6.8 Hz), 3.54 (2 H, dt, J 6.8, 2.4 Hz), 7.28 – 7.52 (5 H, m), 7.72 (1 H, t, J 2.4 Hz).



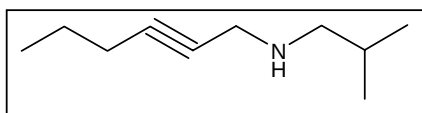
***N*-Isobutyl-3-phenyl-2-propyn-1-amine**

$\nu_{\max}$ (neat)/cm⁻¹ 3321 (NH); δ_{H} (270.01 MHz, CDCl₃) 0.91 (6 H, d, J 6.8 Hz), 1.40 (1 H, br s), 1.75 (1 H, sept, J 6.8 Hz), 2.54 (2 H, d, J 6.8 Hz), 3.61 (2 H, s), 7.23 – 7.26 (3 H, m), 7.37 – 7.40 (2 H, m); δ_{C} (67.9 MHz, CDCl₃) 20.7, 28.3, 39.3, 57.0, 83.2, 88.0, 123.3, 127.9, 128.2, 128.2, 131.6; m/z (CI) 188 (M+H⁺, 100%); Found 188.1438, C₁₃H₁₈N requires 188.1434 (+2.25 ppm error).



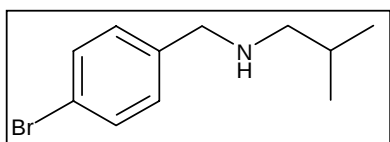
***N*-(2-Hexynylidene)-2-methyl-1-propanamine
(mixture of two isomers)**

δ_{H} (270.01 MHz, CDCl₃) 0.87 (6 H, d, J 6.8 Hz), 0.91 (6 H, d, J 6.8 Hz), 0.98 (3 H, overlapping triplets, J 7.2 Hz), 1.53 – 1.80 (2 H, m), 1.91 (1 H, sept, J 6.8 Hz), 2.27 – 2.34 (2 H, m), 3.26 (2 H, d, J 6.4 Hz), 3.40 (2 H, dd, J 6.4, 2.0 Hz), 7.42 – 7.43 (1 H, m), 7.46 (1 H, s).



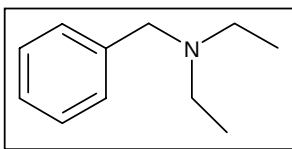
***N*-Isobutyl-2-hexyn-1-amine**

$\nu_{\max}$ (neat)/cm⁻¹ 3316 (NH); δ_{H} (270.01 MHz, CDCl₃) 0.88 (6 H, d, J 6.8 Hz), 0.94 (3 H, t, J 7.2 Hz), 1.48 (2 H, sex, J 7.2 Hz), 1.70 (1 H, sept, J 6.8 Hz), 2.10 – 2.22 (2 H, m), 2.44 (2 H, d, J 6.8 Hz), 3.34 – 3.35 (2 H, m); δ_{C} (67.9 MHz, CDCl₃) 13.4, 20.7, 20.7, 22.3, 28.3, 38.8, 56.9, 78.4, 83.3; m/z (CI) 154 (M+H⁺, 100%); Found 154.1594, C₁₀H₂₀N requires 154.1590 (+2.75 ppm error).



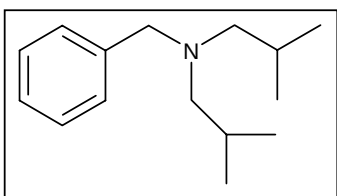
***N*-Isobutyl-*N*-(4-bromobenzyl)amine**

$\nu_{\max}$ (neat)/cm⁻¹ 2952 (CH); δ_{H} (270.01 MHz, CDCl₃) 0.92 (6 H, d, J 6.8 Hz), 1.33 (1 H, br s), 1.76 (1 H, sept, J 6.8 Hz), 2.42 (2 H, d, J 6.8 Hz), 3.74 (2 H, s), 7.22 (2 H, d, J 8.5 Hz), 7.45 (2 H, d, J 8.5 Hz); δ_{C} (67.9 MHz, CDCl₃) 20.6, 28.3, 53.4, 57.4, 120.5, 129.7, 131.4, 139.8; m/z (ES) 242 (M + 1, 100 %), 244 (M+H⁺, 95 %); Found 242.0543, C₁₁H₁₇NBr requires 242.0544 (+ 0.7 ppm error).



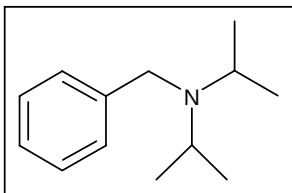
***N*-Benzyl-*N,N*-diethylamine¹³**

δ_{H} (270.01 MHz, CDCl_3) 1.07 (3 H, t, J 7.0 Hz), 2.57 (2 H, q, J 7.0 Hz), 3.63 (2 H, s) 7.21 – 7.38 (5 H, m); δ_{C} (67.9 MHz, CDCl_3) 11.3, 46.3, 57.1, 126.9, 128.1, 129.1, 138.8.



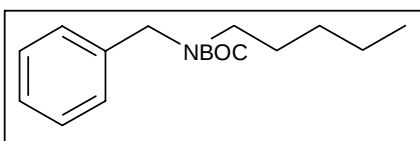
***N*-Benzyl-*N,N*-diisobutylamine**

ν_{max} (neat)/ cm^{-1} 2953 (CH); δ_{H} (270.01 MHz, CDCl_3) 0.87 (12 H, d, J 6.8 Hz), 1.78 (2 H, sept, J 6.8 Hz), 2.09 (4 H, d, J 6.8 Hz), 3.48 (2 H, s), 7.19 – 7.37 (5 H, m); δ_{C} (67.9 MHz, CDCl_3) 20.9, 26.4, 59.9, 63.4, 126.5, 127.9, 128.9, 140.6; m/z (ES) 220 ($M + 1$, 70 %), 176 ($M - 43$, 100 %); Found 220.2061, $\text{C}_{15}\text{H}_{26}\text{N}$ requires 220.2045 (+ 1.8 ppm error).



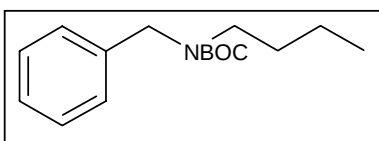
***N*-Benzyl-*N,N*-diisopropylamine**

ν_{max} (neat)/ cm^{-1} 2964 (CH); δ_{H} (270.01 MHz, CDCl_3) 1.04 (12 H, d, J 6.8 Hz), 3.04 (2 H, sept, J 6.8 Hz), 3.66 (2 H, s), 7.17 – 7.39 (5 H, m); δ_{C} (67.9 MHz, CDCl_3) 20.7, 47.8, 48.9, 126.1, 127.9, 143.; m/z (CI) 192 ($M + 1$, 100 %); Found 192.1754, $\text{C}_{13}\text{H}_{22}\text{N}$ requires 192.1752 (- 1.1 ppm error).



***Tert*-butylbenzyl(pentyl)carbamate**

ν_{max} (neat)/ cm^{-1} 2964, 1693; δ_{H} (270.01 MHz, DMSO, Temp 80°C) 0.89 (3 H, t, J 7.0 Hz), 1.17 – 1.55 (6 H, m), 1.45 (9 H, s), 3.15 – 3.20 (2 H, m), 4.42 (2 H, s), 7.26 – 7.40 (5 H, m); δ_{C} (67.9 MHz, DMSO, Temp 80°C) 13.2, 21.2, 28.0, 46.0, 49.4, 78.2, 126.4, 126.8, 127.9, 138.5, 154.6; m/z (CI) 278 ($M + 1$, 96 %); Found 278.2124, $\text{C}_{17}\text{H}_{28}\text{NO}_2$ requires 278.2120 (- 1.4 ppm error).



***Tert*-butylbenzyl(butyl)carbamate**

ν_{max} (neat)/ cm^{-1} 2962, 1695; δ_{H} (270.01 MHz, DMSO, Temp 80°C) 0.87 – 0.92 (3 H, m), 1.21 – 1.50 (4 H, m), 1.45 (9 H, s), 3.16 – 3.21 (2 H, m), 4.42 (2 H, s), 7.26 – 7.40 (5 H, m);

δ_C (67.9 MHz, DMSO, Temp 80°C) 13.1, 19.0, 27.7, 29.4, 45.7, 49.4, 78.3, 126.5, 126.8, 127.9, 138.5, 154.6; m/z (CI) 264 (M + 1, 25 %); Found 264.1972, C₁₆H₂₆NO₂ requires 264.1964 (- 3.3 ppm error).

References

1. Flynn, D. L.; Zelig, R. E.; Grieco, P. A. *J. Org. Chem.* **1983**, 48, 2424.
2. Wuts, P. G. M.; Jung, Y.-W. *J. Org. Chem.* **1991**, 56, 365.
3. Katritzky, A. R.; Zhao, X.; Hitching, G. J. *Synthesis* **1991**, 703.
4. Katritzky, A. R.; Drewniak, M. *J. Chem. Soc. Perkin. Trans. 1* **1988**, 2339.
5. Tanaka, T.; Kobayashi, T. *Synthesis* **1985**, 967.
6. Saoudi, A.; Benguedach, A.; Benhaoua, H. *Synth. Commun.* **1995**, 25, 2349.
7. Masse, G.; Sturtz, G. *Synthesis* **1988**, 907.
8. Enholm, E. J.; Farbers, D. C.; Holub, D. P. *Synth. Commun.* **1990**, 20, 981.
9. Sassaman, M. B. *Tetrahedron* **1996**, 52, 10835.
10. Tsukada, N.; Shimada, T.; Gyoung, Y. S.; Asao, N.; Yamamoto, Y. *J. Org. Chem.* **1995**, 60, 143.
11. Juaristi, E.; Murer, P.; Seebach, D. *Synthesis* **1993**, 1243.
12. Barta, N. S.; Cook, G. R.; Londis, M. S.; Stille, J. R. *J. Org. Chem.* **1992**, 57, 7188.
13. Chesta, C. A.; Avila, V.; Soltermann, A. T.; Previtali, C. M.; Cosa, I. J. *J. Chem. Soc. Perkin Trans. 2* **1994**, 2491.